Low- Enriched Uranium High-Density Target Project: Compendium Report

Nuclear Engineering Division

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by

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ACRONYMS

Activities, components, descriptive terms

ANOVA	analysis of variance
BOC	beginning of cycle (reactor cycle)
CRP	Coordinated Research Project
DU	depleted uranium
EABD	Experiment Authorization Basis Document
EBW	electron beam welding
EDS	energy dispersive X-ray spectroscopy
FEA	finite element analysis
GTAW	gas tungsten arc welding
HDT	high density target
HEU	highly enriched uranium
HT	hydraulic tube
ICTL	internal compression test loop
LEU	low enriched uranium
LOOP	loss of off-site power
MCNP	Monte Carlo N-Particle
MRA	manufacturing readiness assessment
MRL	manufacturing readiness level
NA	natural abundance
NCR	non-conformance report
NQA	nuclear quality assurance
NU	natural uranium
PIE	post-irradiation examination
PVD	physical vapor deposition
R&D	research and development
RMS	root mean square
RSS	Research Safety Summary
RUS	resonant ultrasound spectroscopy
SBLOCA	small-break loss of coolant accident

SBS	safety basis summary
S/N	signal-to-noise ratio
SS	stainless steel
TIG	tungsten inert gas
TRA	Technology Readiness Assessment
TRL	Technology Readiness Level
TSD	Transportation Safety Document
VXF	Vertical Experiment Facility
XRD	X-ray diffraction

Research laboratories or institutes, companies, organizations, programs, facility names

AECL	Atomic Energy of Canada Limited; <i>Ontario, Canada</i>
ANL	Argonne National Laboratory; <i>Lemont, Illinois, USA</i>
ASME	American Society for Mechanical Engineers
ANSTO	Australian Nuclear Science and Technology Organization; <i>Lucas Heights, Australia</i>
BATAN	Badan Tenaga Nuklir Nasional, Puspipstek Complex; <i>Serpong, Indonesia</i>
CEA	Commissariat à l'Energie Atomique; <i>Paris and Gif-sur-Yvette, France</i>
CNEA	Comisión Nacional de Energia Atómica; <i>Buenos Aires, Argentina</i>
DOD	U.S. Department of Defense; <i>Washington DC, USA</i>
DOE	U.S. Department of Energy; <i>Washington DC, USA</i>
DOE-NNSA	U.S. Department of Energy-National Nuclear Security Administration; <i>Washington DC, USA</i>
FDA	Food and Drug Administration; <i>USA</i>
GTIR	Global Threat Reduction Initiative
HFIR	High Flux Isotope Reactor; <i>Oak Ridge, Tennessee, USA</i>
IAEA	International Atomic Energy Agency; <i>Vienna, Austria</i>
ICN	Institutul de Cercetari Nucleare; <i>Romania</i>
INR	Institute of Nuclear Research - Pitesti reactor facility; <i>Mioveni, Romania</i>
IRE	Institut National des Radioéléments; <i>Fleurus, Belgium</i>
KAERI	Korean Atomic Energy Research Institute; <i>Daejeon, Republic of Korea</i>
LANL	Los Alamos National Laboratory; <i>Los Alamos, New Mexico</i>

MU	University of Missouri; <i>Columbia, Missouri, USA</i>
MURR	University of Missouri Research Reactor; <i>Columbia, Missouri, USA</i>
NSITD	Nuclear Security and Isotope Technology Division (ORNL)
ORNL	Oak Ridge National Laboratory, <i>Oak Ridge, Tennessee, USA</i>
RERTR	Reduced Enrichment for Research and Test Reactors program
SNL	Sandia National Laboratories; <i>Albuquerque, New Mexico, USA</i>
Y-12	National Security Complex; <i>Oak Ridge, Tennessee, USA</i>

LOW-ENRICHED URANIUM HIGH-DENSITY TARGET PROJECT: COMPENDIUM REPORT

ABSTRACT

At present, most ^{99}Mo is produced in research, test, or isotope production reactors by irradiation of highly enriched uranium targets. To achieve the denser form of uranium needed for switching from high to low enriched uranium (LEU), targets in the form of a metal foil ($\sim 125\text{-}150\text{ }\mu\text{m}$ thick) are being developed. The LEU High Density Target Project successfully demonstrated several iterations of an LEU-fission-based Mo-99 technology that has the potential to provide the world's supply of Mo-99, should major producers choose to utilize the technology. Over 50 annular high density targets have been successfully tested, and the assembly and disassembly of targets have been improved and optimized. Two target front-end processes (acidic and electrochemical) have been scaled up and demonstrated to allow for the high-density target technology to mate up to the existing producer technology for target processing. In the event that a new target processing line is started, the chemical processing of the targets is greatly simplified. Extensive modeling and safety analysis has been conducted, and the target has been qualified to be inserted into the High Flux Isotope Reactor, which is considered above and beyond the requirements for the typical use of this target due to high fluence and irradiation duration.

1 INTRODUCTION

Technetium-99m (^{99m}Tc), the daughter (decay) product of molybdenum-99 (^{99}Mo), is the most commonly used medical radioisotope in the world. Annually, it is used for approximately 25 million medical diagnostic procedures, comprising some 80% of all nuclear medicine procedures [IAEA-1995]. Other important medical isotopes such as ^{131}I (iodine-131) and ^{133}Xe (xenon-133) can be recovered during ^{99}Mo processing. These radioactive isotopes are being used in therapeutic and diagnostic applications, and have promising applications in the area of radio-immunotherapy. Technetium-99m is recovered from ^{99}Mo generators by clinics and treated chemically using “kits” supplied by radiopharmaceutical suppliers that allow the ^{99m}Tc tagged protein to bind to specific organs, which are then gamma scanned to look at the organ, blood flow through the organ, and other effects. Technetium-99m decays with a half-life of 6.5 hours.

Today, most ^{99}Mo is produced in research, test, or isotope production reactors by irradiation of highly enriched uranium (HEU) targets, which are subsequently processed primarily to recover ^{99}Mo . To yield equivalent amounts of ^{99}Mo , an LEU target must contain approximately five times the uranium of an HEU target. Consequently, substituting LEU for HEU requires changes in both target design and chemical processing. Three major challenges are evident for the substitution of low enriched uranium (LEU) for HEU: (1) modifying the targets and purification processes as little as possible, (2) assuring continued high yield and purity of the ^{99}Mo product, and (3) limiting economic disadvantages. Keeping the target geometry the same between HEU and LEU targets requires using a denser form of uranium in order to increase the amount of uranium per target. To yield equivalent amounts of ^{99}Mo , an LEU target must contain approximately five times as much uranium as an HEU target. Consequently, substituting LEU for HEU will require changes in both target design and chemical processing. Three major challenges have been identified with substituting LEU for HEU: (1) modify the targets and purification processes as little as possible, (2) assure continued high yield and purity of the ^{99}Mo product, and (3) limit economic disadvantages.

To achieve the denser form of uranium needed for changing from HEU to LEU, targets in the form of a metal foil (~125-150 μm thick) are being developed. During irradiation, a large quantity of heat is generated in the uranium foil, up to 200 W/cm^2 in some reactors. In order for the heat to be dissipated to the coolant, the uranium foil must be in good thermal contact with the target structural materials (i.e., the target tubes). If there is poor thermal contact between the uranium foil and the target tubes, the temperature in the uranium foil will increase until the point where uranium will melt and/or react with the fission-recoil barrier or the target structural materials, a condition that must be avoided.

The design of the annular target was conceived to address the above challenges, and its design should significantly reduce the cost of target fabrication by using aluminum target tubes that are extruded to size. The annular design will allow coolant to flow over the outside and through the inside of the target, effectively doubling the heat transfer surface. A schematic of the annular target is shown in Figure 1.1, and a photograph of a series of test targets fabricated for irradiation in Indonesia is shown in Figure 1.2; the two darker targets were clad in zirconium.

FIGURE 1.1 Schematic of Annular Target

FIGURE 1.2 Five Al-Clad and Two Zr-Clad Annular LEU-Foil Targets before Irradiation in the Indonesian RSG-GAS Reactor

The target fabrication procedure is as follows:

- A piece of uranium foil first wrapped in a fission recoil barrier (Ni or Al) is wrapped around the inner tube. A small “undercut” on the inner tube ensures that the uranium foil is positioned correctly within the target.
- The inner tube with the uranium foil wrapped around it is slid into the outer tube.
- The inner and outer tubes with the wrapped uranium foil sandwiched between them are slid into a draw die.

- The inner tube is then expanded by pulling a draw plug through the inside of the inner tube. The diameters of the inner and outer tubes and of the draw plug are chosen such that the inner tube is expanded plastically, and the outer tube is expanded elastically. This creates a target in which the uranium foil is held tightly between the two tubes, effecting good heat transfer within and outside the target.
- After removal from the draw die, the two ends of the target are welded shut.

The target can be inserted into the reactor by using an insertion rig that balances the flow of coolant between the inside and outside of the target. A sketch of a typical target insertion rig is shown in Figure 1.3.

FIGURE 1.3 (A) Target Insertion Rig and (B) Target Insertion Rig with Target Showing Cooling Channel on Inside of Target. Up to Three Targets Can Be Placed on Each Rig.

Until the end of 2012, all LEU-target and -process development was sited at Argonne National Laboratory. At the end of 2012, the Global Threat Reduction Initiative (GTRI) formulated a new strategy for increasing the technical readiness of the annular LEU-foil target and front-end processing to allow its use by current Mo-99 producers. This effort was led by John Creasy, Y-12 National Security Complex (Y-12) in partnership with Argonne National Laboratory (Argonne), Oak Ridge National Laboratory (ORNL), the University of Missouri (MU) Mechanical Engineering Department, and the University of Missouri Research Reactor (MURR) [CREASY-2013, CREASY-2014]. Figure 1.4 describes the individual roles of the partners in this new plan supported by the National Nuclear Security Administration (NNSA).

FIGURE 1.4 Structure for the GTRI High-Density Target Team

The technology was developed in three distinct areas: (1) fabrication of the target, (2) its qualification for irradiation in a high-flux reactor, and (3) frontend processing of the irradiated target to allow the Mo-99 to be purified by the current schemes employed with HEU-dispersion targets. Figure 1.5 is a schematic describing the first two areas. The high density target (HDT) is designed to be compatible with the irradiation sites in reactors utilized by the current Mo-99 producers. The target must meet safety and security requirements before it is approved for irradiation. These requirements carry through into the target specification, which is the first step in the target lifecycle depicted in Figure 1.5. Components of the target are fabricated to the specification. Uranium foils are cast and rolled to the specified thickness, the aluminum cladding tubes are machined with the appropriate geometry to capture the foil, and all components are thoroughly inspected.

The targets are assembled, consolidated, and welded to prevent escape of fission products. The targets are inspected again to ensure that the specification is met. Targets are then loaded into the reactor and irradiated. A typical cycle is 120 to 200 hours (approximately 1 week). The targets are then allowed to cool for a short period following end of irradiation, and they are typically moved to a hot cell in a shielded container. Inside the hot cell the targets are cut open, and the uranium foil is removed to be dissolved. Since the foil is removed from the cladding, waste is minimized for the target process. Following target disassembly and removal of the LEU foil from the cladding, the frontend processing will dissolve the foil, perform an initial separation from other components, and put the Mo into a media that is an acceptable feed to the current purification processes.

FIGURE 1.5 Target Lifecycle through Disassembly (Frontend Processing follows Disassembly)

This report is a compendium of (1) background activities performed at Argonne and (2) the progress made in this project through September 2014. Numerous appendices provide detailed procedures, and QA/QC documents written for this program; they will be listed in the last section of the report.

2 HISTORICAL ACTIVITIES LEADING TO THE 2012 GTRI PROJECT TO IMPROVE TRL

By 1986, the success of the Reduced Enrichment for Research and Test Reactor (RERTR) program in developing U_3Si_2 fuel and codes and analyses for proving the efficacy of converting many reactors made the hitherto minor amount of HEU exported for Mo-99 production become a more important fraction of the HEU consumption. It had become approximately 20% of exported HEU. With the Mo-99 market continuing to grow by ~5%/year, and continuing success in reactor conversion, that fraction would continue to grow. Therefore, a new task to develop means to convert HEU to LEU targets for the production of Mo-99 was begun.

The major obstacles to converting HEU to LEU targets with which to produce fission-product Mo-99 are (1) five-times more uranium is required for irradiation to provide an equivalent Mo-99 yield, (2) approximately fifty times more Pu-239 is formed by irradiating LEU compared to HEU, (3) targets, processes, and waste treatment have to be modified to accommodate the conversion. The goal of the RERTR and subsequent programs has been to find means to make this conversion possible both technically and economically.

Early work at Argonne National Laboratory (Argonne) was directed toward the Union Carbide process (which would soon become the Cintichem process). Union Carbide had testified to a Congressional committee on the concerns and obstacles for conversion to LEU. At the 1986 RERTR meeting, a paper was presented addressing the Union Carbide (Cintichem) technical concerns showing that they were solvable [VANDEGRIFT-1986]. The remaining concern was finding means to make conversion economical.

During 1987, the RERTR program focus was to (1) expand the R&D on Cintichem processing and (2) develop a substitute LEU target by electroplating uranium metal on the inside of a standard Cintichem target [VANDEGRIFT-1987] (such a target would keep target geometry and the dissolution procedure exactly the same for LEU and HEU.) All work was based on open-literature publications and patents.

Argonne also began to look at means to convert targets and processes for those, like IRE (Institut National des Radioéléments—Belgium) and CNEA (Comisión Nacional de Energía Atómica—Argentina), that digested plates of dispersed HEU aluminide or aluminum-alloy in alkaline solution to recover Mo-99. Substitution of LEU silicide plates for the HEU plates would allow targets of the same geometry and Mo yield/target. However, digestion of U_3Si or U_3Si_2 is far more difficult than uranium aluminide and would require two steps—dilution of the cladding by base alone followed by dissolution of the meat by alkaline peroxide.

During the 1987 RERTR meeting, high-level discussions were held with CNEA. A verbal agreement was made to cooperate on converting the Argentine process to LEU. Argonne presented a paper discussing its efforts on (1) electroplating uranium metal onto the inner surface of a Cintichem target, (2) conducting experiments designed to understand the effects of LEU targets on the Cintichem process, and (3) processing LEU U_3Si_2 targets.

Following the 1987 RERTR meeting, Argonne participated in an International Atomic Energy Agency's (IAEA's) Technical Committee Meeting that assessed current targets and technologies for production of Mo-99, problems associated with radioactive waste disposal, economic factors, and conversion to LEU [IAEA-1987]. Argonne was given an opportunity to present its advances in conversion technologies. Atomic Energy Canada Limited (AECL) presented work they had performed on converting their process to LEU targets using LEU silicide targets. Dissolving uranium silicide in nitric acid forms a silica gel that makes processing difficult and reduces Mo yield. Due to these problems, AECL was pursuing other target designs, including uranium oxide and uranium metal. Ali Sameh (then at the Nuclear Research Centre, Karlsruhe) described the process he developed that would become the Mallinckrodt process. Representatives from IRE, AECL, CNEA (using the Sameh process), and the German Democratic Republic described their processes. Following the meeting, Argonne staff members were invited to visit the IRE facilities, where they were given a full tour of the Mo-99 production facility, and open conversations were held with great promise of future cooperation.

In 1988 and 1989, Argonne continued work on electroplating uranium metal [MARSHALL-1988] and HIPping (hot isostatic pressing) uranium foils onto a Zircaloy tube [VANDEGRIFT-1989]. Argonne also pursued R&D to allow conversion of the IRE and CNEA process to LEU [KWOK-1988]. Progress was made on developing a digestion method for U₃Si₂ targets, and experiments were run to understand the effects of LEU conversion on the initial IRE Mo-recovery step using an alumina column. Acid dissolution of U₃Si₂ targets was also pursued as a means to mitigate the negative effects of silica-gel formation.

Development of technologies for conversion of Mo-99 targets was inactive from FY 1990 to late FY 1993. However, in October 1992, the U.S. Congress passed an amendment to the Atomic Energy Act of 1954, often referred to as the Schumer Amendment [H12103]. This amendment prohibited export of HEU for use as a fuel or target in a research or test reactor unless several conditions are met: (1) no alternative LEU fuel or target can be used, (2) the U.S. is actively developing an LEU fuel or target for that reactor, and (3) the proposed recipient of the HEU provides assurances that, whenever possible, an LEU fuel or target will be used in that reactor. In 1993, the RERTR program efforts entailed renewing interactions with ⁹⁹Mo producers, defining the means to test the Argonne process for recovering Mo-99 from irradiated LEU-silicide targets, and beginning testing a process on spent LEU-silicide mini-plates stored at Argonne from past fuel development studies. Argonne also began pursuing processing of LEU metal targets. The thrust of Mo-99 papers presented at the 1993 RERTR meeting indicated that research efforts in the Mo-target conversion program was active again, and its research efforts would be expanded. Proposals for active cooperation with the Mo-99 production community were emphasized [VANDEGRIFT-1993, WIENCEK-1993].

In 1994, active cooperation began with both the Indonesian Badan Tenaga Atom Nasional (BATAN) and the Argentine CNEA. The concept of the LEU-foil annular thermal-expansion target was developed for processes that used acid dissolution as the first step in Mo recovery [SRINIVASAN-1994]. The idea for this target was to have a uranium foil held between two concentric cylinders of different material. The material used in the inner cylinder would have a higher thermal expansion than the outer-cylinder material, assuring good contact between the

uranium foil and the target walls. Further, to aid in assembly and disassembly of the target, the walls would have a slight taper [WIENCEK-1994].

Digestion of a high-burnup U_3Si_2 miniplate was demonstrated at ANL to test the efficacy of the ANL-designed process [HUTTER-1994]. Argonne also began to pursue the dissolution of uranium foil under conditions of the Cintichem process (as provided to Argonne by its Indonesian partner) [SRINIVASAN-1994].

The 1995 RERTR meeting was the first one to have a separate Mo-99 production session. Seven papers were presented by ANL and its BATAN and CNEA partners. BATAN described bench-scale demonstration of the ANL-modified Cintichem process for LEU-foil targets [ALILUDIN-1995]. CNEA presented their own studies on digesting LEU silicide; Cols et al. [COLS-1995] and ANL described the R&D progress pursuing conversion of the Cintichem and CNEA processes [SNELGROVE-1995, WU-1995, SRINIVASAN-1995, BUCHHOLZ-1995]. A process for digestion of uranium-metal foil by alkaline peroxide was also described at the 1995 RERTR meeting [DONG-1995].

In 1996, the ANL/BATAN cooperation was in full gear. Targets were irradiated in Indonesia, and early tests led to the requirement for a fission-recoil barrier between the uranium foil and the target wall. Without this barrier, fission product tracks acted to bond the foil to the target walls and made extraction difficult [HOFMAN-1996]. Based on material, nuclear, and chemical properties, nickel was chosen as the best fission recoil for acid-dissolution and zinc alkaline-digestion processes. At Argonne and the BATAN PUSPITEK facility, experiments set conditions for dissolution of the fission-barrier clad foils [LEONARD-1996]. At the University of Illinois and Argonne, tracer-level experiments led to greater understanding of the Cintichem process chemistry and led to means to modify the process for using LEU targets [WU-1996A, WU-1996B]. In PUSPITEK, Argonne was able to observe target processing using the Cintichem process and was given documents that describe the process by BATAN. Three papers on progress for the BATAN cooperation were presented at the 1996 RERTR meeting.

Also at that meeting, Argonne presented a paper describing detailed kinetic models for digestion of uranium silicide and uranium metal by alkaline peroxide [CHEN-1996, DONG-1996]. This paper was essentially a conclusion of the uranium silicide effort. The RERTR program had decided by that meeting that the use of the LEU-foil target was the best alternative to converting both acid and basic dissolution processes. Although Argonne had developed and demonstrated means to dissolve and recover Mo-99 from irradiated uranium-silicide plates, the method was cumbersome.

In 1997, Argonne cooperation on modifying the Cintichem process with BATAN was extended to include Los Alamos National Laboratory (LANL) and Sandia National Laboratories (SNL). SNL and LANL were preparing to produce commercial Mo-99 at SNL using the Cintichem process. The LEU-modified Cintichem process continued to be developed [VANDEGRIFT-1997A], and Argonne developed means to electroplate thin layers of nickel and zinc onto LEU foils for fission-recoil barriers [SMAGA-1997]. A full-scale demonstration of the LEU-modified Cintichem process was being planned for 1998 in Indonesia. Use of LEU UO_2 -dispersion targets was studied as an alternative to uranium-silicide targets for producers who did

not want to use metal foil targets. Compacts with 40 vol% UO₂ were fabricated and dissolved in alkaline peroxide [CONNER-1997].

George Vandegrift attended an IAEA consultants' meetings at the National Accelerator Centre of South Africa, Faure, from April 10-12, 1997, to discuss (1) progress in converting HEU targets to LEU and (2) suggestions on how conversion could take place for every manufacturer. A 50-page chapter in the IAEA technical document that was generated from this meeting documented his presentation [VANDEGRIFT-1997B].

In 1998, irradiation of eight targets and processing of two irradiated LEU-foil targets were demonstrated in Indonesia using the LEU-modified Cintichem process [MUTALIB-1998]. Further, the LEU-foil target was improved by heat treating the foil before assembly. This heat treatment decreased the grain size in the foil and improved its performance during irradiation. Concerns about the zinc fission-recoil barrier reacting with the uranium or target materials after irradiation and before processing were addressed and found to be inconsequential [CONNER-1998].

During this year, key international collaborations were growing. Argonne's collaboration with the Indonesian BATAN continued to move forward. A cooperative agreement between the Argentine CNEA and Argonne was completed. Argonne was also in the planning stage of cooperation with the Australian National Science and Technology Organization (ANSTO), which had provided the opportunity to develop optimized target designs. The IRE in Belgium volunteered to cooperate with Argonne in future activities to test the irradiation and processing of an LEU metal target. The Mo-99 session had a record number of presentations at the 1998 RERTR meeting, nine presentations from five countries.

In 1999, the RERTR program's Mo-99 activity had become a significant strength in the HEU minimization efforts, as seen from the Mo-99 overview paper presented by Jim Snelgrove at the 1999 RERTR Meeting [SNELGROVE-1999]:

Over this past year, extraordinary progress has been made in executing our charter to assist in converting Mo-99 production worldwide from HEU to LEU. Building on the successful development of the experimental LEU-foil target, we have designed a new, economical irradiation target. We have also successfully demonstrated, in collaboration with BATAN in Indonesia, that LEU can be substituted for HEU in the Cintichem target without loss of product yield or purity; in fact, conversion may make economic sense. We are interacting with a number of commercial producers--we have begun active collaborations with the CNEA and ANSTO; we are working to define the scope of collaborations with MDS Nordion and Mallinckrodt; and IRE has offered its services to irradiate and test a target at the appropriate time. Conversion of the CNEA process is on schedule.

The key to our meeting the RERTR program's goal of complete conversion to LEU is tied to open dialog and active cooperation with all producers. BATAN's willingness to allow us to test new target concepts and processing has led to our

success in developing the annular, metal-foil targets. CNEA's open sharing of information and active development activities will make their successful conversion to LEU a reality. ANSTO's openness and willingness will also lead to their success. IRE commitment to irradiate and test LEU targets when we are ready for them is gratifying. The open technical sharing over the years and recent overtures for active cooperation from Mallinckrodt bodes well.

An activity that started five years ago against heavy opposition now appears to be on a solid road to success. We will continue to cooperate with producers as our resources allow. Our goal continues to be to provide a benefit to humankind by ending the use of HEU for ⁹⁹Mo production, and to do it in a way that provides an economic benefit to the producer as well.

In 1999, the current annular target design was developed and tested. Targets with depleted uranium (DU)-foils were tested by thermal cycling and measuring its effect on swelling and residual hoop stress. During this year, seven targets with a variety of fission-recoil barriers were successfully irradiated in Indonesia [CONNER-1999].

Two demonstrations of processing irradiated targets were performed in Indonesia in March and September of 1999 using the LEU modified Cintichem process [VANDEGRIFT-1999A]. Additionally, dispersion plates were fabricated with 20-25 vol% uranium powder in an aluminum matrix to produce high-density dispersion plates [VANDEGRIFT-1999B]. The same paper looked at means to introduce LEU foils into the CNEA alkaline process.

At the 2000 RERTR program, momentum from the 1999 successes continued. Argonne's ongoing collaborations continued to flourish, and all producers were becoming active in looking at conversion. Armando Travelli summarized the situation in his RERTR overview paper [TRAVELLI-2000]:

Significant progress was achieved during the past year on several aspects of producing ⁹⁹Mo from fission targets utilizing LEU instead of HEU. The goal is to develop and demonstrate during the next few years one or more viable technologies compatible with the processes currently in use with HEU at various production sites throughout the world. This activity is conducted in cooperation with several other laboratories including the Indonesian National Nuclear Energy Agency (BATAN), the Australian Nuclear Science and Technology Organization (ANSTO), and the Argentina Comisión Nacional de Energía Atómica (CNEA). Cooperation in this area is also beginning with MDS Nordion and AECL (Canada), Mallinckrodt Medical (The Netherlands), IRE (Belgium), NECSA (South Africa), and KAERI (Republic of Korea). During the past year, four targets of the new annular design reported last year were irradiated at BATAN, showing that (1) aluminum can be used for the target tubes and (2) uranium foils with nickel, zinc, or aluminum fission-recoil barriers can be removed from the target. The acidic chemical process to be used in combination with this target was demonstrated, and the feasibility of

using LEU metal foil targets in combination with the basic process was assessed.

Two papers were presented by Argonne at the 2000 RERTR meeting. One described irradiation of another series of eight targets in Indonesia and processing an irradiated foil for the LEU modified Cintichem process [CONNER-2000]. The other described plans for irradiating four targets (two foils each) in Argentina and progress in developing alkaline digestion of the irradiated foil [VANDEGRIFT-2000A]. Argentina presented their progress is developing the UAl₂ high-density dispersion target [KOHUT-2000] and testing it and a high-density uranium-silicide dispersion target [COLS-2000]. Australia discussed their cooperation with Argonne, their LEU-foil test target, and their plans to irradiate it in December 2000 [DONLEVY-2000].

The final demonstration of the LEU modified Cintichem process in Indonesia was scheduled for right before the 2001 RERTR meeting. This demonstration was planned to include the irradiation of an Indonesian-fabricated LEU-foil target and show that Mo-99 produced from LEU would act the same as HEU-produced Mo-99 on the Indonesian generator. Past tests had already proven equivalent purity, higher Mo-99 yield, shorter processing time, and smaller waste volumes through conversion to LEU. The events and aftermath of September 11, 2001, prevented the completion of the demonstration. It also led to the cancellation of the 2001 RERTR meeting. Because of that cancellation, much of the RERTR progress was presented at the 6th International Meeting on Research Reactor Fuel Management (RRFM) held in the spring of 2002; Argonne progress on Mo-99 target conversion was presented at that meeting [VANDEGRIFT-2002A]. The presentation highlighted (1) developments in dissolving irradiated foils in alkaline solutions, (2) modifications to the anion-exchange step possible due to the conversion to LEU foils and not needing to dissolve the entire target for Argonne's CNEA cooperation, and (3) demonstration of how use of titania-based sorbents for molybdenum recovery and improvements to the waste-calcination process could allow facile conversion of the AECL/Nordion process to LEU targets.

A 2001 demonstration using an irradiated full-size LEU-foil target in Argentina was a partial success. The target irradiation and disassembly were flawless; however, processing was not fully developed. Lessons learned from the demonstration charted a path forward.

At that time, interactions with MDS Nordion had increased, and a substantial R&D program was underway with its partners AECL and SGN. MDS Nordion's stated main concern regarding conversion was the effect of five times more uranium on its waste-calcining operations. Argonne provided data and technical solutions to address the main concerns and enable conversion.

In 2002, Argonne efforts were directed toward supporting conversion technologies for three producers (CNEA, MDS Nordion, and ANSTO). CNEA converted to LEU targets using a dispersion target suggested by Argonne [CRISTINI-2002]. This target allowed CNEA to continue production at their current rate but allowed no opportunity for increasing production. Conversion to an LEU foil target would allow a tripling of their Mo-99 production in the same irradiation position, and it would lower the liquid waste from processing by 6-10 times and processing time by at least 4 hours. Major development challenges for the CNEA conversion using LEU-foil targets were (1) designing a prototype production vessel for digesting irradiated

LEU foils in alkaline solutions, (2) developing means to improve digestion efficiency, and (3) modifying ion-exchange processes used in the CNEA recovery and purification of Mo-99 to deal with the lower volumes generated from LEU-foil digestion [VANDEGRIFT-2002B].

Argonne continued to collect data for allowing economic conversion of the AECL calcining method to LEU. Results showed that using the in-place equipment LEU conversion would produce twice the waste cans as HEU—not the five-times more cans that concerned MDS Nordion [BAKEL-2002]. Argonne also showed that changing the sorbent for Mo recovery could have the effect of lowering the waste solution volume.

Interactions with ANSTO continued, especially dealing with target fabrication, which was their current technical driver. Argonne provided technical assistance on processing and waste treatment and disposal as requested [DONLEVY-2002].

A member of the Korean Atomic Energy Research Institute (KAERI) presented an analysis of irradiating an annular target containing uranium foil or uranium oxide at the 2002 RERTR meeting [LEE-2002].

The technical cooperation on target conversion between the RERTR program and MDS Nordion and the other major producers slowed down significantly towards the end of 2002. Cooperation still continued with Argentina during 2003, and an improved digester and digestion process were demonstrated in spring 2004 [GELIS-2003]. Development of processes to convert the Canadian process to LEU continued into 2003 [BAKEL-2003], but progress on the technical cooperation with MDS Nordion stalled.

According to a presentation at the 2003 RERTR meeting, KAERI had chosen the annular LEU-foil target for production of Mo-99 at the HANARO reactor [LEE-2003].

In 2004, the U.S. Department of Energy established the Global Threat Reduction Initiative (GTRI) program under the National Nuclear Security Administration (NNSA) that incorporated HEU minimization programs, such as RERTR, including the reactor fuel conversion program as well as the Mo-99 target conversion program. GTRI continued the annual international conversion meeting, maintaining “RERTR” as a well-established and recognizable acronym for the meeting. Under GTRI, Argonne began to look at digestion of LEU foils using IRE conditions as well as conditions used by CNEA [GELIS-2004]. The IRE dissolver solution contains sodium nitrate as well as sodium hydroxide, which should improve the oxidation of uranium metal to U(VI). This work was performed with no direct interaction with current producers. Further development on titania-based sorbents for Mo-99 also continued, but for a more generic process rather than specifically intended for the Canadian process [BAKEL-2004].

A significant technical innovation was presented by KAERI at the 2004 RERTR meeting announcing a method to produce large quantities of uranium foil quickly by cooling roll casting [KIM-2004].

It was also at the 2004 RERTR meeting that the idea that the IAEA initiate a Coordinated Research Project (CRP) was introduced to assist development of small indigenous production of Mo-99 without the use of HEU [IAEA-2015].

On August 8, 2005, the U.S. energy bill, which included the Burr amendment, was signed into law. This amendment modified the restrictions of HEU exports to five countries (Canada, Belgium, France, Germany, and the Netherlands) for use as targets to produce medical isotopes [KUPERMAN-2005, -2006].

At the 2005 RERTR meeting, Argonne presented two papers describing progress on conversion technology. The first was a paper describing the technical “facts and myths” surrounding conversion to LEU targets [VANDEGRIFT-2005]. The second was a summary of the progress in Indonesia and Argentina, and in development of titania-based-sorbent recovery of Mo from concentrated LEU solutions [BAKEL-2005].

At this same meeting, Argentina provided data showing that the Mo-99 product from processing its LEU targets was of even greater purity than what it had produced from HEU [DURAN-2005], and the IAEA announced the start of its coordinated research project for the production of Mo-99 using LEU fission or neutron activation [GOLDMAN-2005]. Much of the technology that the RERTR program had developed for conversion of the Indonesian process would be shared with a group of potential small-scale producers.

In 2006, Argonne GTRI work was focused on both acid and alkaline processes in cooperation with Indonesia and Argentina, respectively [BAKEL-2006A]. There was also a very close cooperation with members of the IAEA CRP on Molybdenum-99 Production Using LEU or Neutron Activation [GOLDMAN-2006, MEDEL-2006, TOMA-2006].

In 2007, all of Argonne’s Mo-99 conversion activities were focused on the IAEA CRP on Molybdenum-99 Production Using LEU or Neutron Activation. Argonne continued conversion activities with both Indonesia and Argentina [VANDEGRIFT-2007A, VANDEGRIFT-2007B, BAKEL-2007]. Indonesia presented an update on their conversion using LEU-foil annular targets [BRIYATMOKO-2007]. Several members of the CRP presented their progress at the 2007 RERTR Meeting [GOLDMAN-2007, BSEBSU-2007, SCHRAEDER-2007]. A paper by Charles Allen, University of Missouri Research Reactor (MURR), discussed the possibility of an LEU-foil plate-type target [ALLEN-2007]. Argentina presented updated results following their conversion to high-density LEU targets [CESTAU-2007].

In 2008, GTRI was overseeing both conversion activities and means to secure a stable U.S. domestic supply of Mo-99. Conversion activities were almost entirely centered on the IAEA CRP on Molybdenum-99 Production Using LEU or Neutron Activation. Argonne was also providing technical assistance to two potential domestic producers of fission-product Mo-99—MURR (annular targets) and Babcock and Wilcox Technical Services Group (solution reactor). Argonne continued to assist CRP members in fabricating LEU-foil annular targets and using the LEU-modified Cintichem process for producing small-scale indigenous production of fission-product Mo-99 [SCHRAEDER-2008, BAKEL-2008]. The IAEA presented an overview paper at the 2008 RERTR meeting. MURR was studying the possibility of producing 50% of the U.S. domestic supply of Mo-99 using LEU-foil targets and provided several papers on engineering studies on assembling annular targets and predicting their thermal behavior during irradiation [SOLBREKKEN-2008A, SOLBREKKEN-2008B, EL-GIZAWY-2008].

Argonne also presented a paper at the 2008 RERTR meeting on (1) a study performed on KAERI cold-rolled foils, which included activities to prepare foils for irradiation at MURR and ingots for rolling in Indonesia, and (2) initial results for a pre-conceptual design of a facility to produce LEU foils and annular targets [WIENCEK-2008]. The major finding for the KAERI foil is that the roughness of the foil could not be improved by cold rolling, and the overall thickness was reduced, but the difference between low and high spots was not lessened. The pre-conceptual design study for a facility to fabricate LEU-foil targets was ultimately published as an Argonne topical report [WIENCEK-2009].

The most significant event in 2009 was the release of the U.S. National Academies Report on Medical Isotope Production without Highly Enriched Uranium in January [NAP-2009]. This report addressed five topics mandated by the U.S. Congress; these topics and short summaries of their findings follow [VANDEGRIFT-2009A, VANDEGRIFT-2009B]:

- Feasibility of procuring supplies of medical isotopes from commercial sources that do not use HEU

Production of ^{99}Mo on a large scale using LEU targets is technically feasible. Converting current producers to LEU would require a significant cost and time. Not enough ^{99}Mo is currently produced from LEU to meet U.S. demand, but no technical reasons exist for that not being true in the future.

- Current and projected demand and availability of medical isotopes in regular current domestic use

The current U.S. demand for ^{99}Mo is 5000-7000 6-day-Ci/week. Future demand growth is likely 3-5%/year. Reliability of supply is a current problem that should continue with or without conversion. New foreign or domestic sources are several years away.

- Progress being made by the Department of Energy and others to eliminate all use of HEU in reactor fuel, reactor targets, and medical isotope production facilities

The GTRI program has made substantial progress in converting reactor fuels and targets, but much is still left to do. It was recommended that the GTRI focus on removing HEU waste from ^{99}Mo production by either down blending or returning US-origin material to the US.

- Potential cost differential in medical isotope production in the reactors and target processing facilities if the products were derived from production systems that do not involve fuels and targets with HEU

The cost of conversion to LEU targets would be less than 10% of the current cost of HEU-produced ^{99}Mo . While determining the absolute cost of

conversion is difficult to quantify in such a complex production/distribution system, the report concluded that conversion cost would have a negligible impact to the cost to the patient.

- Identify additional steps that could be taken by the Department of Energy (DOE) and medical isotope producers to improve the feasibility of such conversions

Mo-99 producers should commit to conversion and take steps to move forward. The Department of Energy should (1) assist producers with the technical expertise of its laboratory system, (2) examine options to share R&D costs with existing and new producers, and (3) remove disincentives to conversion by maintaining the cost of LEU so it is competitive with HEU. The Department of State should intensify diplomatic pressure on countries that still use HEU targets and fuel. The U.S. Food and Drug Administration (FDA) should work with industry and DOE technical experts to (1) ensure a common understanding of LEU-based production from a regulatory perspective and (2) promote a good understanding of likely FDA requirements for obtaining approval.

Argonne continued to be very active in the IAEA-CRP for Small-Scale Indigenous Production of Mo-99 Using LEU Targets or Neutron Activation, assisting Chile, Libya, Poland, and Romania to implement the LEU-modified Cintichem process. Supplies and equipment were sent to partners, and visits were undertaken or planned [VANDEGRIFT-2009B]. A redesigned pressurized dissolver for up to 120 g-U was fabricated and sent to two CRP partners [JERDEN-2009].

The electrochemical dissolution of U foils was introduced for the first time at the 2009 RERTR meeting [GELIS-2009]. Until this development, the only means to digest uranium metal in alkaline solution was at high temperatures (275°C, therefore high pressure) with the addition of an oxidizing agent.

Solbrekken et al. presented a paper reviewing analytic, numeric, and experimental activities performed at the University of Missouri for annular and plate geometries for LEU-foil targets [SOLBRECKEN-2009].

A collaboration between the GTRI and the IAEA had been developing and in 2010 became paramount to GTRI's Mo-99 conversion efforts. In a 2010 RERTR paper, Bradley et al. documented this collaboration [BRADLEY-2010]. Along with the still active CRP on Developing Techniques for Small Scale Indigenous Mo-99 Production Using LEU Fission or Neutron Activation, an International Working Group was launched by the IAEA in August 2010 to provide support for HEU-to-LEU conversion projects underway or being considered by major Mo-99 producers, and work was begun to develop a comprehensive document on the status and prospects of various non-HEU Mo-99 production technologies.

The latter activity ended with the publication of a report entitled *Non-HEU Production for Molybdenum-99 and Technetium-99m* [IAEA-2013]. This report reviews all current and potential means of producing Mo-99 and Tc-99m and attempts to gauge their current maturity.

The IAEA maintains a website for documenting the progress of the International Working Group for Conversion Planning [IAEA-2010].

Another significant paper at the 2010 RERTR meeting was by Gavin Ball, who presented the status of the South-African conversion to high-density uranium-aluminide LEU targets [BALL-2010]. Kim et al. presented an update on KAERI efforts to improve cast-rolled foils (C. Kim, Y. Woo, K. Kim, J. Oh, M. Sim, “An Effort to Improve U Foil Fabrication Technology of Roll-casting for Fission Mo Target,” but did not provide a paper to the proceeding). A poster presentation updated Argonne progress in developing electrochemical dissolution of LEU foils [GELIS-2010]. A second poster presented experimental data for a production-scale (~250 g-U) dissolver designed by Argonne for acidic dissolution of LEU foils [JERDEN-2010].

A poster presentation summarized Brazilian activities in dissolving high-density $\text{UAl}_2\text{-Al}$ dispersion targets in alkaline solution [CAMILO-2010].

In 2011, a major change occurred in the GTRI program with respect to the Mo-99 activity. Because of (1) the growing worldwide interest in converting Mo-99 production to LEU targets and (2) the growing U.S. program for developing domestic production of Mo-99, the RERTR meeting was felt to be no longer the proper venue for hosting Mo-99 sections, so a separate Mo-99 Topical Meeting was held in December, 2011, in Santa Fe, New Mexico. Los Alamos National Laboratory hosted the meeting. Papers from that meeting are hosted on an Argonne website [MO-99-2011].

Because of the limiting venue for the Mo-99 Topical Meeting and the U.S. emphasis on domestic production at the Mo-99 topical meeting, Vandegrift did present a paper at the 2011 RERTR meeting on GTRI/Argonne conversion activities [VANDEGRIFT-2011]. For conversion activities, the GTRI continued to make technical expertise available, on a non-proprietary basis, to global medical-isotope producers to assist with converting their Mo-99 processes to use LEU and to provide technical support in a number of areas, including foil rolling, target fabrication, target irradiation, target disassembly, target dissolution, product recovery and purification, and waste treatment. Argonne continued to support the conversion of international ^{99}Mo production by (1) developing front-end processes that will allow high-density-LEU-foil targets to be used in the alkaline processes for the production of ^{99}Mo using HEU targets by the current producers, (2) cooperating with the Institutul de Cercetari Nucleare (also known as ICN and the Institute of Nuclear Research, Subsidiary of the Romanian Authority for Nuclear Activities, Mioveni, Romania (INR)) and Y-12 to perform LEU-foil-target qualification experiments in Pitesti, Romania, and (3) working with the IAEA Working Group for Conversion Planning for ^{99}Mo Production Facilities from HEU to LEU. In this paper, Vandegrift et al. updated progress on the electrochemical dissolution of LEU foils. Based on comments from current producers, the development of the pressurized acidic dissolver was abandoned, and the design was shifted to an ambient pressure dissolver; this design and preliminary experimental data were discussed. Further, a chromatographic method for recovering Mo from the acidic dissolver solution using a titania column and purifying it from uranium and many other fission products was presented, as were experimental data demonstrating its usefulness.

A new activity in 2011 was cooperating with Y-12 and ICN to perform a series of irradiations and post-irradiation examination (PIE) of LEU-foil targets in Pitesti, Romania, to collect data necessary to qualify the LEU-foil target for the producers to irradiate these targets in reactors they currently use for their HEU targets. In this collaboration, Y-12 was to fabricate the annular LEU-foil targets and ICN to irradiate them in their 14-MW TRIGA reactor and performed PIE in their hot-cell facility over a twelve-month period. Argonne coordinated activities and provided technical support. This project was started in the fall of 2011.

A third Argonne activity at the end of 2011 was participation in the IAEA Working Group for Conversion Planning for Molybdenum-99 (Mo-99) Production Facilities from HEU to LEU. On December 1-2, 2011, Argonne hosted the working group and gave them an update on progress, including laboratory tours. Y-12 was also present at the meeting and informally updated the group on their activities. Argonne presentations at that meeting are on the Working Group's website [IAEA-2010].

A third meeting of the working group was held in November 2012. A meeting report and presentations made at the meeting are available online [IAEA-2010].

Argonne contracted ICN to prepare for irradiating and performing PIE of annular LEU-foil targets. The INR successfully completed the first work order, which was designing experiments and equipment for the irradiations and the PIE of a series of targets in October 2012 [CIOCANESCU-2013]. The INR performed exceptionally, and their products were of the highest quality; however, the project was not extended due to significant increases in the cost estimates and prioritization of program resources.

Due to this change in events, at the end of 2012, GTRI formulated a new strategy for increasing the technical readiness of the annular LEU-foil target and front-end processing to allow its use by current Mo-99 producers. This effort was led by John Creasy, Y-12 National Security Complex in partnership with Argonne, ORNL, the University of Missouri Mechanical Engineering Department, and MURR.

3 TARGET FABRICATION

3.1 CLADDING TUBE MACHINING

The primary cladding used in this project was aluminum (several alloys), although instances utilizing Zircaloy were successfully demonstrated as well. The geometry of the target is customizable for the irradiation geometry and location, although most targets were approximately 1.2 in. (3.0 cm) in diameter and 6.5 in. (16.5 cm) in length.

The cladding inner and outer tubes were typically rough cut to length from tubing stock, then finish machined to the specified diameters. The pocket (or relief), which is used to retain the uranium foil in the target, is machined into the inner tube to a specified depth, which is calculated using formula defined later in this document, with the objective of constraining the foil during assembly and ensuring adequate thermal contact.

3.2 FISSION RECOIL BARRIERS

Fission recoil barriers are an important feature of the high-density metal target. Fission recoil barrier application may be performed by a large variety of methods, ranging from manual application of foils or wraps to various plating techniques, including physical vapor deposition (PVD).

3.2.1 Background Information (Early Argonne R&D)

3.2.1.1 The Need for a Fission-Recoil Barriers

In 1995, the first annular LEU-foil targets irradiated in Indonesia could not be disassembled due to the uranium foils bonding to the target walls. This bonding was due to intimate contact and an intense flux of fission recoil atoms at the uranium surface, apparently resulting in sufficient atomic mixing at the interface to effect a strong metallurgical bond. A fission-recoil barrier needed to be placed between the uranium foil and the target walls. Several metals were evaluated for fission recoil barriers. The principal requirement for these metals is primarily of a chemical nature. Because the barrier metal will bond to the uranium foil, it should not interfere with the dissolution and purification parts of the ^{99}Mo process. Four metals were thus selected as barrier candidates: copper, nickel, iron, and zinc. The first three are suitable only for acid dissolution processes, while zinc can be used for acid or base processes. At this time, copper and nickel were selected. Since the recoil range in Cu and Ni is approximately 7 mm, a 10-mm-thick barrier should stop all recoil atoms from reaching the target tube surfaces [HOFMAN-1996].

3.2.1.2 Nuclear and Chemical Effects of Using a Fission-Recoil Barrier

In a parallel paper presented at the 1996 RERTR meeting, the effects on dissolution of the uranium foils by the addition of a 10- μ m metal fission-recoil barrier between the U foil and the target walls was considered [LEONARD-1996]. The overall dissolution reactions for metal barriers of Cu/Ni and Fe using nitric acid alone are:

Dissolution rates for these three metals were measured over a variety of conditions in a covered, but unsealed, centrifuge tube in a constant-temperature bath. The results indicated that all three metals dissolve faster than U: Ni is five times faster, Cu is 190 times faster, and Fe is 560 times faster. Equations 3.1 and 3.2 and the ideal gas law were used in calculating the dissolution pressure for a two-sided barrier on a typical 18-g U-foil target with barrier dimensions of $76 \times 102 \times 0.010$ mm ($3 \times 4 \times 0.00039$ in.), a dissolver temperature of 103°C, and a gas volume of 282 mL. The results indicated an increase in the final dissolver pressure of 23, 35, and 25 psig for Cu, Fe, and Ni, respectively, over that for U alone. Thus, the dissolver pressure will be increased about 10% by the presence of a two-sided barrier. This pressure increase due to the dissolution of these three barrier materials could be used without affecting the operation of the U-foil dissolver.

In two tests, Ni foil was used in the closed (sealed) stainless-steel dissolver. The results of these tests were very similar. Their pressure-time plots indicated that the Ni dissolved easily, as expected. The final gas pressure for this piece of Ni foil was expected to be 159 psia. Instead, a much lower pressure, about 40 psia, was actually realized. The difference in these two pressures may be due to NO gas solubility in the dissolver solution.

A third paper at the 1996 RERTR meeting looked at the nuclear properties of fission-recoil barriers and their effects on the recovery and purity of ^{99}Mo [WU-1996A]. Based on their physical, chemical, and nuclear properties, three metals (Cu, Fe, and Ni) were selected as primary candidates for the fission-barrier material. Physical characteristics are important to target fabrication and are discussed in [BUSEV-1969]. Important chemical characteristics are foil dissolution (reported in [LEONARD-1996]) and the effect of barrier materials on the recovery and purity of ^{99}Mo (discussed below). The nuclear properties of interest are the radioisotopes generated in the barrier during target irradiation and their activity levels, which must be removed from molybdenum during processing, as discussed below.

Table 3.1 shows ORIGEN2 calculations for the radioisotopes generated in Fe, Ni, and Cu barriers during LEU target irradiations in the RGS-GAS reactor. The target contains an 18-g uranium-metal foil with a 10- μ m metal barrier on each side of the foil. The results of these calculations show that only a copper barrier would generate enough radioactivity to be of concern. For ^{64}Cu to be less than 0.1 $\mu\text{Ci/mCi-}^{99}\text{Mo}$ in the molybdenum product, its overall decontamination factor must be $>3,100$.

TABLE 3.1 Radioisotopes Generated from Barrier Metals during Irradiation of an LEU Metal Target^a

Metal	Isotope	Half-Life	Activity, mCi
Fe	Fe-55	2.73 y	37
	Fe-59	44.5 d	26
	Mn-54	312 d	1.3
	Mn-56	2.56 h	1.0
Ni	Ni-65	2.52 h	53
	Co-58	70.92 d	97
Cu	Cu-64	12.7 h	153,000
	Cu-67	61.9 h	0.6

^a Barrier material (10 μ m) on both sides of an 18-g metal foil irradiated in the RGS-GAS reactor at full power for 120 hours.

Neither the barrier materials nor their neutron-activation products are reported to interfere with the precipitation of molybdenum by alpha-benzoin-oxime (also known as α BO or ABO) [BUSEV-1969, ELWELL-1977, PARKER-1983], which is the major purification step in the LEU-modified Cintichem process. Experiments were run to verify the noninterference of these metal ions by using solutions prepared to simulate dissolving the fission-barrier-clad uranium foil in nitric acid. In the same experiments, we measured the amount of each barrier metal that carried with the molybdenum precipitate. Table 3.2 shows the results of these experiments. The molybdenum recovery was high for all experiments, as were the measured decontamination factors. It is likely that the differences in the decontamination factors are more an indication of how well the precipitate was washed in each experiment than chemical differences in the barrier-metal ions.

The decontamination factors measured for iron and nickel are more than high enough to meet impurity requirements for the molybdenum product. However, the removal of ⁶⁴Cu may require additional decontamination. The use of copper foil as a fission-recoil barrier was dropped based on this result.

TABLE 3.2 Effects of Barrier Materials on α -BO Precipitation: Molybdenum Yield and Barrier-Metal Decontamination Factors^a

	Cu	Fe	Ni
Molybdenum recovery (%)	99±3	96 ± 3	96 ± 3
Decontamination factors	1680	258	660

^a Solution contained 0.75M HNO₃, 1.5M UO₂(NO₃)₂, and the concentration of Cu, Ni, or Fe corresponding to a 10- μ m barrier on either side of the uranium foil.

3.2.1.3 Zinc Fission-Recoil Barriers

Further results of the fission recoil barriers were presented at the 1997 RERTR meeting, where the use of zinc was considered [VANDEGRIFT-1997A]. Because the uranium foil target was now being developed as the primary LEU target for ⁹⁹Mo-production processes that begin by dissolving HEU-aluminide-dispersion targets in base, we needed to select and test the use of a base-soluble metal as a fission-recoil barrier. A literature survey found the following elements that dissolve in alkaline solutions: aluminum, zinc, beryllium, gallium, tin, arsenic, niobium, and tantalum. Aluminum dissolves at about the same rate as uranium metal in 5.0M H₂O₂/1.5M NaOH solution at 70°C. However, there was a strong concern that uranium would react with aluminum during the target irradiation. Germanium and rhenium, although not amphoteric, are reported to dissolve readily in dilute hydrogen peroxide. The toxicity of beryllium metal and the low melting point of gallium (30°C) preclude their use. Arsenic is classified as a non-metal and may not have sufficient metallic properties to be made into a foil. Zinc is an active electropositive element and forms a strong anion with oxygen. It also dissolves readily in sodium-hydroxide/nitrate solution. Work on barrier materials for targets to be processed by dissolution in base had, therefore, focused on zinc. Because of the appeal of developing just one target for all processes, a zinc fission barrier was also being considered for the target in acid-side processing.

Three methods of forming the zinc barrier were considered: (1) pressing together zinc and uranium foils, (2) hot dipping the uranium target in molten zinc, and (3) electroplating the zinc onto the uranium foil. One of the most important constraints on the barrier is that it should be of relatively uniform thickness and not greater than 15 μ m to minimize the material to be dissolved after irradiation. Use of pre-formed zinc foil pressed onto the uranium target was eliminated because of the high cost and porous nature of the zinc foils thinner than 20 μ m. Hot dipping provides a coating of rather poorly controlled thickness, and thicknesses less than 20 μ m are unlikely. Electroplating of zinc, on the other hand, seemed to be a relatively simple and inexpensive process with good control over the thickness. Another paper presented at the 1987 RERTR meeting discussed Argonne progress in electrodepositing zinc fission-recoil barriers [SMAGA-1997].

The ORIGEN2 code was used to calculate the neutron activation of zinc being used as a fission-recoil barrier for irradiation of an LEU-foil target in the BATAN RGS-GAS reactor. Table 3.3 presents the activity generated after 12 hr of cooling for one gram of the 99.95% pure zinc. An 18-g LEU-foil target would require about 1.7 g of zinc for a double-sided 15- μ m barrier. This target would produce about 700 Ci of ^{99}Mo for a 120-h irradiation. The documentation accompanying the zinc foil we purchased for use in earlier test targets listed the impurity content. The neutron activation products formed from the impurities were also examined. It was found that Zn has a lower neutron absorption than either Cu, Fe, or Ni. As expected, only a small amount of radioactivity is produced from the impurities, and they can be ignored for the zinc foil we purchased. However, the results in Table 3.3 show that the impurity levels of cadmium and copper should be carefully scrutinized in selecting zinc for barrier materials.

Three types of solutions for dissolving zinc were studied: NaOH, NaOH/H₂O₂, and NaOH/NaNO₃. A variety of solutions containing NaOH or NaOH/H₂O₂ showed dissolution rates less than the desired 2 mg/(cm²•min). On the other hand, a variety of solutions containing NaOH and NaNO₃ gave dissolution rates well above that. A solution of 2.5M NaOH/1M NaNO₃ at 70°C is our standard for rapid dissolution of pure zinc metal. Experiments have confirmed that a zinc coating electroplated onto the uranium-foil surface is also dissolved rapidly by this solution. Analysis by energy dispersive spectroscopy (EDS) of the foils left after dissolution showed that all of the zinc plate was dissolved from the uranium. Liquid scintillation counting of the dissolution solution showed that virtually none of the uranium foil was dissolved with the zinc.

TABLE 3.3 The Activity Generated per Gram of Zinc Barrier Material during Target Irradiation

Element	Content, g	Product	Half-Life	Activity
Zn	0.9995	Zn-65	243.9 d	0.43 Ci
		Zn-69m	13.76 h	0.79 Ci
		Cu-64	12.7 h	0.13 Ci
Ca	0.000001	Ca-44	165 d	0.1 μ Ci
Cd	0.000020	Cd-115	53.46 d	0.41 Ci
Cu	0.000015	Cu-64	12.7 h	1.65 Ci
Fe	0.000010	Fe-55	2.73 y	0.31 μ Ci
		Fe-59	44.5 d	0.21 μ Ci
In	0.000010	In-114m	49.51 d	79 μ Ci
Mg	0.000001	Na-24	14.96 h	0.03 μ Ci
Na	0.000002	Na-24	14.96 h	98 μ Ci
Ni	0.000001	Co-59	70.92 h	0.07 μ Ci
Pb	0.000100	Pb-208	3.25 h	0.26 μ Ci
Si	0.000002	Si-31	2.62 h	0.04 μ Ci
Sn	0.000008	Sn-119m	245 d	2.1 μ Ci

(Irradiation for 120 hours with a thermal flux of $1.9\text{E}+14$ n/cm²-s and an epithermal flux of $6.90\text{E}+13$ n/cm²-s, followed by 12 hours cooling at the end of irradiation.)

Because radioactive-decay heat is generated within the irradiated LEU, Argonne was concerned that a zinc barrier might melt during transport of the target from the reactor pool to the processing hot cell. The biggest hindrance to the transfer of heat from the target during transport is the relatively small surface area from which heat can be transferred. Simple calculations were made for natural convection in the air immediately after the target leaves the cooling pool and for the possibility that the target will be placed in a cask and shipped over a 24-h time frame. Although exact temperatures to be experienced by the zinc barriers cannot be predicted, calculations show that temperatures in the range of 300-400°C are likely. Although this is very close to zinc's melting point (420°C), it is expected that the zinc will not melt. However, when a zinc-plated uranium foil was heat treated at 375°C overnight in an evacuated glass tube, an intermetallic U/Zn compound was formed. The intermetallic U/Zn compound is visually distinct from the unheated zinc-plated uranium foil when viewed through a microscope with a magnification of about 440X. Figures 3.1(A) and (B) are micrographs of a zinc-plated DU foil after heat treatment. Figure 3.2 is a micrograph of another section of the foil before this heat treatment. With this intermetallic compound, the interface between the zinc and the uranium is almost indistinguishable (Figure 3.1 (A)), whereas it had been pronounced before (Figure 3.2). Notice the dendrite formations along the edge of the foil in Figure 3.1(A). Even if the target is held at low overall temperatures during transport, ion mixing resulting from stopping the fission fragments during irradiation will likely cause the formation of this intermetallic compound at the U/Zn interface.

We were unsuccessful in trying to duplicate the effect of this heat treatment. In the sample shown in Figure 3.2, the uranium was severely over etched during surface preparation for electrodeposition; however, the adhesion of the zinc to the uranium was excellent. (Details on surface preparation for electrodeposition can be found in [SMAGA-1997].) The obvious difference between the “successfully” heat-treated foil and the “unsuccessful” attempts is the quality of the zinc plate. In the successful foil, the zinc plate was even, continuous, and non-nodular. In the unsuccessful foils, the zinc plate was nodular and porous. The unsuccessful foils show severe oxidation and separation of the zinc plate from the foil in a scaly pattern. In fact, it appears that holes have been “blown” all the way through the uranium foil. One possible explanation is that the zinc plate on the unsuccessful foils retains water because of its nodular, porous nature. This water expands tremendously when it is converted to steam and becomes extremely corrosive to uranium. The successful foil was not porous and should not have retained much water in its matrix. Future experiments were planned to test this hypothesis.

Solutions suitable for dissolving the intermetallic U/Zn compound were investigated. Attempts to dissolve the zinc from the heated foil showed that this intermetallic would not dissolve as the zinc plate had from the unheated foils (at 70°C with a solution of 2.5 M NaOH and 1 M NaNO₃). On the other hand, a solution of 1.5 M NaOH/5 M H₂O₂ appeared to dissolve the intermetallic compound faster than it did pure zinc or pure uranium. Additional experimental work is necessary to clarify the rate of dissolution.

(A)

(B)

FIGURE 3.1 Two Sections of Zn-plated Uranium Foil after Heating at 375°C Overnight (Compare to Figure 3.2)

FIGURE 3.2 Early Zn-Plated Uranium Foil Showing Considerable Loss of Uranium during Etching

Researchers at PUSPIPTEK attempted to dissolve a Zn-barrier LEU foil that was successfully removed from an irradiated target. They used conditions that should have completely dissolved the foil in 30 min. The gas pressure generated during dissolution and the radioactivity levels in the spent dissolver solution both were far lower than expected. This may be due to (1) the formation of the U/Zn compound at the Zn-foil/U-foil interface during irradiation and (2) this compound dissolving at a lower rate than either uranium or zinc. To test this hypothesis, a piece of the zinc-plated uranium foil that formed the U/Zn intermetallic was dissolved in 8 M HNO₃ at 80°C. It did dissolve at a rate substantially lower than either metal alone. (Zinc dissolves almost instantaneously in 8 M HNO₃ at 80°C.)

3.2.1.4 Electroplating Zn and Ni Fission-Recoil Barriers

In a second Argonne paper at the 1997 RERTR meeting, progress in electroplating both zinc and nickel fission-recoil barriers was presented [SMAGA-1997]. This paper presented the work done to date on electroplating a metal barrier onto the uranium foil. In terms of cost and performance, electroplating is considered the best fabrication approach. The literature contains a modest number of publications on electroplating nickel on uranium [DINI-1967, DINI-1969, DINI-1974, WEIRICK-1981, HENRI-1997, PETIT-1969, O'LEARY-1956, [LUNDQUIST-1967]. No publications on electroplating uranium with zinc were found.

The principal difficulty in electroplating uranium is that it oxidizes readily in air and water, and the resulting oxide layer makes it impossible to obtain a metal-to-metal bond. A mechanical bond has been obtained by cleaning the surface of most of the uranium oxide, etching the uranium to obtain a rough surface, and electroplating. Uranium has been successfully electroplated in this way with nickel to prevent corrosion. Electroless nickel plating of uranium has also been demonstrated [PETIT-1969]. The etchants reported in the literature, thus far, include concentrated solutions of nickel, ferric, stannous, cupric, or lithium chloride. Mixtures of hydrochloric and sulfuric acids have also been tried, as well as anodic etching procedures [O'LEARY-1956, LUNDQUIST-1967]. The most frequently used etchants have been the nickel- and ferric-chloride solutions. Surface preparation methods used prior to etching were buffing with pumice or calcium oxide, sand blasting, and immersion in 8 M HNO₃.

3.2.1.4.1 Experimental

Depleted uranium (DU) foil was used for the bulk of the plating experiments performed because of the limited availability of LEU foil. The primary difference between the DU and LEU foils was not the isotopic content. It was that the DU foil was standard-purity uranium metal. The LEU foils contained alloy additions of iron (450 ppm) and aluminum (1000 ppm) [VANDEGRIFT-1997A]. These additives refine the grain size of the uranium [MCDONELL-1968]. Indicative of the smaller grain size for the LEU foil with this adjusted composition is that it dissolved at twice the rate of the pure uranium (DU) foil in 8M HNO₃ at 80°C.

3.2.1.4.1.1 Pre-Plating Preparation

The procedure for preparing these uranium foils prior to actual plating proved to have a major impact on the quality of the plating. A descaling step was necessary to remove the pre-existing oxide scale. Then, an etching step was needed to roughen the surface and promote coating adherence. Without an irregular surface to mechanically key the plating, the substrate and plating readily separated. The etching agents investigated included chloride salts of iron, nickel, and zinc in concentrations that ranged from 0.45 to 9.0 M. At times, the preparation procedure was augmented with immersions of the foil into strongly alkaline as well as strongly acidic solutions. Ultimately, the procedure we employed contained the following essential steps:

1. A sample of foil was cut, its dimensions measured, and its weight recorded. These samples were typically 15 to 50% of the full target size to provide a reasonably representative geometry; however, the first LEU samples were < 10% of full target size due to limited availability.
2. To degrease the foil, it was immersed in xylene or tetrachloroethylene for one minute, then immersed for one minute in methyl alcohol, ending with a one-minute rinse in deionized water.
3. The foil was pickled in an 8 M nitric-acid solution to completely remove the thick oxide scale. This step took usually 5 to 10 minutes, depending on the thickness of the scale, and was followed by multiple immersions in deionized water for total time periods of 6 to 15 minutes.
4. The sample was etched in a stirred 5.33 M ferric-chloride solution for 2 to 5 minutes at 40°C to roughen the surface. For this step only, a nylon strap replaced the Pt hanger securing the sample. Multiple rinses in deionized water again removed the etching solution. The total rinse time varied from 6 to 30 minutes, depending on factors like continued gas evolution in the rinse.
5. The sample was returned to the 8 M HNO₃ solution for 2 to 7 minutes to remove the black coating left by the etching treatment. Once again, multiple rinses in deionized water completed this step.

Weight and thickness were often measured after each rinsing operation to assess the amount of uranium removal. When these measurements were made, the fifth step was often repeated, but the immersion in the HNO₃ solution was limited to less than one minute. The purpose of the repetition was to remove any oxide film caused by air exposure and to activate the surface for electroplating.

Copper foils were also plated to evaluate the operation and performance of the electroplating baths. A different and much simpler preparation procedure was used for the copper samples. Samples were cut to size, gently abraded with powdered calcium oxide, immersed in a 1 M hydrochloric acid solution, and thoroughly rinsed in running deionized water. This treatment

did not provide the same level of surface roughness as the treated uranium samples. Prior to placement in a plating bath, the weight and dimensions were measured for these samples.

3.2.1.4.1.2 Electroplating Solutions and Conditions

Pre-mixed, commercially obtained, electrolytes¹ were used for both the zinc and nickel plating experiments. The zinc electrolyte was an all-alkaline solution containing sodium zincate, sodium hydroxide, and an additive, STARZINC 595, to promote smooth, uniform, and adherent deposits. The polyethylene plating tank had interior measurements of 30 cm long by 16 cm wide and was filled to a depth of ~25 cm. The samples were hung from a central copper rod that was secured to the top of the tank at mid-length. The two zinc anodes (99.99% purity) were suspended from two parallel copper rods that flanked the central rod at a spacing of 11.5 cm each. These anodes were removed from the bath during periods of nonuse to minimize dissolution of zinc into the bath. This plating bath was not agitated or filtrated, and plating was performed at ambient temperature.

The nickel electrolyte was a nickel sulfamate solution to which nickel bromide was added to improve anode efficiency and reduce stress in the deposited nickel. Boric acid was also added to stabilize the pH at 4, which also lowered stress in the deposits. This polyethylene plating tank had interior measurements of 30 cm long by 30 cm wide and was filled to a depth of 30 cm. The spacing between adjacent copper rods for electrode connections was 12.5 cm. The two nickel anodes were believed to be 99.99% in purity. This bath was filtered continuously, via connections to an external pump, to remove any suspended solids. The pumping action caused a steady circulating motion in the electrolyte. The bath temperature was maintained at ~40°C through the use of an immersion heater. Control of temperature provided another means of minimizing stress in the deposited nickel. Deionized water was added periodically to make up evaporative losses from both baths.

For both types of plating experiments, the sample was secured by means of a clipped hook. The length of the clipped hook was sized to provide complete immersion of the sample in the bath with minimal exposure of the clip to the electrolyte, once the hook was suspended from the energized central Cu rod. For some tests, a direct lead from the power supply was also connected to the clipped hook. The second connection made the plating circuit immediately active once any part of the sample contacted the electrolyte. The power supplies provided constant-current output at pre-set levels. Current levels were calculated on the basis of the sample geometry and the desired current density.

Bath temperature, current, and voltage were recorded at the beginning and the end of an experiment. The current density and the time of plating were the two main variables. The plating duration was predetermined based on the intended current density and the expected plating efficiency. Upon completion of a plating experiment, the sample was rinsed, dried, and weighed.

¹ Starlite Chemicals, Inc., 1319 West North Avenue, Chicago, IL 60622.

3.2.1.4.1.3 Assessment of Plated Samples

The quality of the plating was assessed by three methods. First, the weight gain was used to calculate the average plating thickness based on a fully dense coating. Second, micrometer measurements were taken to quickly gauge the uniformity of the plating thickness. Third, the sample was sectioned and examined microscopically. Sectioning was done in a manner that provided a fully representative cross section, generally along a diagonal of these rectangular samples.

Standard preparation procedures for metallographic samples were followed, with 0.3 μm alumina powder and MetadiTM fluid as the lubricant for the final polishing. The local coating thickness was then determined from photomicrographs taken at multiple locations on the sample. In addition, optical microscopy provided estimates of coating coverage, indications of coating adherence, and the degree of substrate roughening developed during the pre-plating steps.

3.2.1.4.2 Zinc-Plating Results

Chronologically, the first plating experiments were conducted using the zinc bath, and many issues regarding proper preparation of the foil surface were resolved during the early plating experiments. For instance, we found the simplest and most consistent method of descaling the foil surface was immersion in 8 M HNO_3 . Thus, this solution became the preferred method of removing the oxide. In trials of various etchants, nickel chloride (containing nitric acid) proved too vigorous for 130- μm -thick foils, and its usage was discontinued. The ferric-chloride etchant was ultimately chosen, but not before a number of concentrations were tried. The 5.33 M FeCl_3 became the standard based on its ability to dissolve a predictable quantity of uranium in a reasonable time span.

As time progressed, the zinc-plating bath proved to be an ideal medium for determining the influence of the etching on plating quality. As shown in Table 3.4, plating coverage tended to decrease as the amount of foil dissolution increased. This inverse relationship was not exact because other factors, such as coating thickness, also influenced coverage. Nevertheless, this correlation stressed the need to counterbalance the need for maximum surface roughness to obtain maximum adherence. Microscopy revealed that the source of this relationship was non-uniform dissolution of the foil surface. Numerous deep crevices and/or irregular pits were present in the substrate, where essentially no deposition occurred during electroplating. Over-etching increased the number of deep depressions, and the limited throwing power of the zinc bath prevented plating in these areas.

TABLE 3.4 Influence of Pre-plating Weight Loss on Plating Coverage for Zinc-Plated Samples

Number of Samples	Range in Pre-plating Weight Loss (%)	Range in Plating Coverage (%)
6	6 to 15	100
3	10 to 30	90 to 99
3	36 to 43	<90

The results of plating-thickness measurements from later zinc-plating experiments are summarized in Table 3.5. The samples whose designations begin with “ZnDU” had DU foil substrates, whereas those beginning with “ZnLEU” had LEU foil substrates. Thickness determinations by three different methods are given, with the exception of three samples (ZnDU17-19) for which the preparation and plating steps intentionally were not interrupted for intermediate weight and thickness measurements. The micrometer measurements provided a fast assessment of plating uniformity but overstated the plating thickness due to overemphasis of the thicker edge and corner values. Through Faraday’s law, the weight measurements gave the most accurate indication of the amount of deposited metal, but no insight into the uniformity of the deposition. The comparatively lengthy process of optical microscopy gave the best indication of the range in coating thickness. The reported value typifies plating thickness over most of the sample but specifically excludes measurements taken at the edge.

TABLE 3.5 Plating Thickness (in μm) for Zinc-Plated Substrates by Three Different Methods

Sample Identification	Thickness via Micrometer	Thickness via Weight Gain	Thickness via Microscopy
ZnDU17	N.M. ^a	N.M.	12
ZnDU18	N.M.	N.M.	17
ZnDU19	N.M.	N.M.	7
ZnDU20	25	10	18
ZnDU21	80	41	57
ZnLEU22	112	52	58
ZnDU23	34	14	7
ZnLEU24	27	16	27

^a N.M. = not measured.

Samples ZnDU17-20 were highly and deeply etched by the ferric chloride. The resultant zinc coatings were discontinuous and coverage was incomplete. In extreme cases, such as that shown in Figure 3.3, the plating consisted of discrete and isolated nodules. Where deposition occurred, it tended to be thick. Thus, microscopy measurements of thickness, which were not

adjusted for lack of coverage, tended to overstate the thickness in comparison to the calculations based on weight change. The estimated plating coverage ranged from 74% for ZnDU20 to 93% for ZnDU17. As shown in Figure 3.4, the morphology of the deposited zinc on ZnDU17 still retained nodular characteristics, even though the general coating appearance was much improved.

FIGURE 3.3 Discontinuous Coverage and Nodular Zinc Deposits on a Severely Over-etched DU Substrate (ZnDU20, 350X)

FIGURE 3.4 Improved Coverage and Semi-nodular Zinc Plating on an Over-etched DU Substrate (ZnDU17, 350X)

The behavior of ZnDU21 was unusual. A 2.54 cm × 5.08 cm piece of foil did not react in 8 M HNO₃ at room temperature in over 20 minutes, although other pieces of the same foil (6.5 cm × 8.0 cm) were freed of the oxide in <10 min. The oxide scale was finally removed in 3 minutes when the temperature of the descaling solution was raised to 50°C. There was also a problem with the electroplating bath due to corrosion scales on the clipped hook and copper rods. The current did not reach the desired value until the scales were removed, and consequently, the current varied widely during the electroplating. The current (coulombs) that was passed was not known. In spite of these difficulties, zinc coverage was complete. As shown in Figure 3.5, the plated layer was overly thick but uniform. The substrate showed no evidence of being deeply etched, a finding that was consistent with the measured pre-plating weight loss of 7.8%. The improvement in coating coverage again correlated with minimization of etching treatment.

Samples ZnLEU22 and ZnLEU24 were the first adjusted uranium foils electroplated. The surface of these foils was only slightly oxidized, as opposed to the pure uranium foils, and the surfaces cleaned up well in 8 M HNO₃. Sample ZnLEU22 was tested in the as-received state, while sample ZnLEU24 was heat-treated in a vacuum for 10 minutes at 720°C before testing. The zinc plating on ZnLEU22 was about four times the desired thickness. In large part, the thick

plating was due to an atypical increase in coulombic efficiency to 80%, twice the value of the expected efficiency used in calculating the plating time. The plating on ZnLEU24 was about one-third greater than the desired 15 μm . Once again, the coulombic efficiency of 78% was greater than the 60% assumed in calculating the plating time. (The unpredictable variation in efficiency continued to be a major challenge for zinc plating. In early experiments, plating efficiencies were as low as 20%. The trend has been to better efficiencies as our plating experience increases.) Both of these samples had coatings that were smooth and relatively uniform. The thick plating on ZnLEU22 separated from most of one side, but the ZnLEU24 plating generally exhibited good adhesion, as shown in Figure 3.6. These results indicate that there is no inherent problem in plating adjusted-uranium foils as opposed to pure-uranium foils; in fact, our limited experience shows superior results for plating of the adjusted uranium.

FIGURE 3.5 Uniform and Continuous Zinc Plating on a Properly Etched Pure-Uranium Substrate (ZnDU17, 180X)

FIGURE 3.6 Uniform and Continuous Zinc Plating on a Properly Etched Adjusted-Uranium-Foil Substrate (ZnLEU24, 220X)

Sample ZnDU23 was 7.02 cm $\times$ 5.08 cm in size. It was the largest foil plated thus far, and demonstrated the ability to plate larger foils. The average thickness based on weight gain was 14 μm , essentially the desired thickness. Microscopy found that the trends observed previously for smaller samples were qualitatively true for the larger sample. In a few areas of localized over-etching, coverage was discontinuous. The bulk of the surface, however, had an adherent plating that was free of nodular characteristics. Variations in thickness were clearly

related to sample geometry. At corner and edge locations, the thickness was two to three times the thickness in the central areas of the surfaces.

The dependence of plating thickness on sample geometry is presented more rigorously in Figure 3.7 for ZnDU23 and other samples from the ZnDU series. To normalize for the difference in sample size, the abscissa is expressed as the distance from the center divided by one-half of the diagonal distance. To adjust for sample-to-sample variations in thickness, the ordinate is the thickness at a given location divided by the thickness found at the center of that particular sample. Scaling up the sample size apparently exacerbated the center-to-edge variation in coating thickness because the ZnDU23 data are the uppermost points in Figure 3.7.

FIGURE 3.7 Relative Thickness of Zn Plating as a Function of Distance from the Sample Center

3.2.1.4.3 Nickel-Plating Results

The initial tests in the nickel-plating bath used copper rather than uranium as the substrate. The four samples in the NiCu series were plated at different current densities to determine the range in coulombic efficiency for this bath. The target current densities were 10, 20, 40, and 80 mA/cm², and the plating time was adjusted between 0.25 and 2.0 hr to produce a nominally constant plating thickness of 25 μ m. The only serious difficulty encountered in this series of experiments involved the sample plated at the lowest current density. The power supply failed to maintain the preset current setting and drifted up with time. The total charge passed was estimated as the average of the initial and final currents, which did not vary appreciably for the other three experiments. The net weight gains were used to calculate the quantity of charge that went into plating. The coulombic efficiencies ranged from 85% to 98%. The greatest uncertainty was associated with the 98% value because this value was obtained from the compromised test. Figure 3.8 illustrates the inverse relationship between current density and coulombic efficiency found for these data. The decline in efficiency at higher current densities was relatively modest over this range.

FIGURE 3.8 Effect of Current Density on Plating Efficiency for the Nickel Sulfamate Bath at 40°C

The nickel-plated copper samples possessed the following characteristics. In all cases, coverage was 100% except for small areas ($\sim 2 \text{ mm}^2$) covered by the electrical connection. The surface of the plating was smooth except for the edges of the sample. At the edges, excess localized plating took the form of large rounded deposits with a characteristic diameter of $\sim 20 \text{ }\mu\text{m}$. The plating thickness was very close to the intended thickness of $25 \text{ }\mu\text{m}$ with the exception of the sample plated at the lowest current density. As shown in Table 3.6, the three different methods used to determine thickness gave values that differed, but the variation tended to be consistent.

TABLE 3.6 Plating Thickness (in μm) for Nickel-Plated Substrates by Three Different Methods

Sample Identification	Thickness via Micrometer	Thickness via Weight Gain	Thickness via Microscopy
NiCu10	47	40	30
NiCu20	36	25	21
NiCu40	37	24	19
NiCu80	32	23	18
NiDU1	14	18	14
NiDU2	30	N.M. ^a	16
NiDU3	19	15	15
NiDU4	19	15	10

^a N.M. = not measured.

The intent of the early nickel-plated uranium samples (NiDU series) was to establish which steps in the intricate sample preparation procedure have the greatest impact on plating quality. Thus, plating parameters were not intentionally altered for these four samples. Based on the results for the copper substrates, the current density was fixed at a nominal value of 32 mA/cm^2 , the plating time was set at 25 minutes, and the bath temperature was held at $40 \mu\text{m} \pm 2^\circ\text{C}$. Under these conditions, the nickel-plated uranium samples were expected to have an average thickness of $15 \mu\text{m}$.

Pretreatment of sample NiDU1 included all of the previously described degreasing, descaling, etching, and pickling steps, whereas sample NiDU2 was only descaled in 8 M HNO_3 , followed by four 2-minute rinses in deionized water. Sample NiDU1 had a 34.8% metal weight loss beyond the weight loss associated with descaling. The resulting roughened surface, however, greatly improved the adherence of the nickel coating. As shown in Figure 3.9, nickel deposited on all exposed substrate surfaces, even those pitted areas well below the original surface. As a consequence, the nickel coating was able to mechanically lock into the uranium substrate. In contrast, the plating on sample NiDU2 failed to adhere well to its smooth substrate, as Figure 3.10 illustrates. Plating coverage was 100% for both samples, and, as summarized in Table 3.6, the typical coating thickness was essentially the same. The poor adherence for the NiDU2 sample was due to the smooth substrate. These experiments confirmed that some degree of surface roughening is a prerequisite for well-bonded coatings, as was previously found for the zinc-plated samples.

FIGURE 3.9 Complete Coverage and Adherence for Nickel Plating on a Severely Overetched DU Substrate (NiDU1, 350X).

FIGURE 3.10 Delaminated Nickel Plating on an Un-etched DU Substrate (NiDU2, 180X).

Two experiments performed subsequently sought to reduce the quantity of uranium lost during pretreatment yet still provide sufficient bonding. One key factor observed in most of the tests with uranium substrates was that most of the weight loss did not occur during the actual FeCl_3 etching step but during the subsequent pickling and rinsing operations. These simple immersions were not removing entrapped etchant completely, and often vigorous reaction continued to occur. Magnetic stirring was introduced to improve the effectiveness of these removal operations. Samples NiDU3 and NiDU4 also served a dual purpose in that they came from different sheets of foil. Standard sample preparation and plating were performed on the two samples concurrently, thus eliminating potential procedural differences as a source of differences in plating response.

The weight losses of these two samples at intermediate processing stages were comparable with the exception of the descaling step. During descaling, sample NiDU3 (foil A) lost 1.5% of its weight, whereas sample NiDU4 (foil B) lost 1.1%. After etching and pickling, the respective losses were virtually identical: 5.5% and 4.4% for NiDU3 and 5.6% and 4.5% for NiDU4. The smaller weight losses after etching were attributed to the effectiveness of magnetic stirring. The post-plating weight gains were identical at 12.6%, which met the thickness goal on a gravimetric basis. Subsequent studies judged the plating quality to be good. Coverage was 100% and, as shown in Figure 3.11, a relatively smooth coating maintained good contact to a substrate that was sufficiently roughened. As was noted for the Cu-substrate samples, the edges of the uranium samples also over-plated. The ball-shaped edge deposit shown in Figure 3.12 was typical of this condition. Two key findings from these concurrent experiments were (1) variations in plating results are not attributable to differences in uranium foils, and (2) the quantity of uranium lost in pretreatment can be controlled at acceptable levels (~10%) without losing plating adherence if adequate agitation is used for subsequent aqueous processing steps.

The microscopic studies of the nickel-plated samples showed a greater proclivity toward over-plating at the edges than was noted for zinc-plated samples. Systematic analysis of the photomicrographs revealed that the effect was relatively independent of the substrate. The results of this analysis are presented in Figure 3.13, a dimensionless plot similar to Figure 1.7. In Figure 3.13 the abscissa is unchanged from Figure 1.7, but the ordinate is now the ratio of the measured plating thickness at a location divided by the gravimetrically determined average thickness. At center and intermediate positions, the data overlapped for the eight datasets. At edge positions, the data again overlapped, but the Cu samples tended to have greater values. The measured thickness was relatively constant for the central region and was approximately 80% of the gravimetric thickness. The change in thickness increased at an accelerated rate as the corners were neared. Typically, the measured thickness was three times greater than the gravimetric thickness, but the spread in the data was much greater. The severity of over-plated edges was clearly more pronounced with nickel plating than with zinc plating.

FIGURE 3.11 Uniform and Continuous Nickel Plating on a Properly Etched DU Substrate (NiDU4, 350X).

FIGURE 3.12 Rounded Nickel Deposits and Localized Loss of Adherence at an Over-plated Edge (NiDU3, 180X).

FIGURE 3.13 Relative Thickness of Ni Plating as a Function of Distance from the Sample Center

3.2.1.4.4 Conclusions of 1997 Plating Studies

The feasibility of electroplating zinc or nickel barrier layers on uranium foils has been demonstrated. The plating conditions can be controlled to provide a barrier layer with a nominal (gravimetric) thickness of 15 μm that also provides complete coverage of the substrate. Although these techniques were developed through work on pure-uranium foils, our limited work with adjusted uranium has indicated that the transition to this material will proceed smoothly.

Work thus far also revealed the importance of the surface preparation prior to plating. A roughened surface is essential to bind the plating to the substrate. Excessive dissolution of the foil during etching, however, creates localized crevices that fail to plate in the zinc bath and result in discontinuous coverage. Over-etched foils plated in the nickel bath did not exhibit a loss in coverage; however, the deposits in the crevices often fell below the minimum requirement of 7 μm . The preparation procedure outlined in this paper provides reasonable control of total uranium dissolution. By limiting this loss to <10%, the subsequent plating can be expected to combine full coverage with sufficient adherence. Better control of the etching process is expected in the near future as improvements, such as ultrasonic cleaning, are incorporated into the preparation procedure.

These experiments have also highlighted a concern about adequate control of the as-plated coating thickness over the full foil. Over-plating at the edges, especially in the nickel bath, may exceed the tolerances needed to maintain the desired fit within the target, and our preliminary indication with larger foils was that the degree of over-plating increases with foil size. Future work addressed changes in the design of the plating bath (i.e., auxiliary anodes, robber cathodes, and perimeter shielding of the cathode) that could potentially reduce the center-to-edge variation in thickness for full-sized foils.

3.2.1.5 1998 Progress in Electroplating of Fission Recoil Barriers

An update on Argonne progress in fission-recoil development was presented at the 1998 RERTR Meeting [CONNER-1998, VANDEGRIFT-2000B]. These results are discussed below.

3.2.1.5.1 Plated-Foils for Irradiation in Indonesia

After heat treatment (beta quenching), two foils were plated for the demonstration in Indonesia. One was plated with nickel and the other with zinc; the plated thickness targets were 10 μm and 15 μm , respectively. Plating was done at a current density of 20 mA/cm^2 , and the electroplating time was adjusted to obtain the desired plated thickness. The thicknesses of the electroplated foils were measured by using a micrometer and weighing before and after etching and after plating. The micrometer-caliper measurements were made at 10–12 locations around the foil and varied by $\pm 8\%$ at all stages of plating. The uranium mass losses from etching were 4.9% and 5.8% for the two foils. The final thicknesses of the fission fragment barriers used for the demonstration in Indonesia are shown in Table 3.7.

**TABLE 3.7 Fission Fragment Barrier
Thicknesses of Foils Used for Demonstration
in Indonesia**

Plate	Expected Thickness, μm	Thickness by Micrometer, μm	Thickness by Mass, μm
Zn	15	21	17.1
Ni	10	9	10.9

3.2.1.5.2 Fission-Recoil Barrier Reaction with Uranium

Rough calculations showed that it may be possible for the target to reach a temperature of 375°C during transport. We were concerned that this high temperature may cause problems in target disassembly, as we had previously found that a temperature of 375°C allowed an intermetallic U/Zn compound to form from a reaction between the barrier and the uranium. Samples used to investigate the reaction of fission fragment barrier materials were prepared by compressing metal powder (either aluminum or zinc containing less than 1 wt% oxide) around a piece of DU foil that was wrapped with nichrome wire to mark the interface. The samples were compressed at ~60,000 psi for thirty seconds in a die. Figure 3.14 (A) shows a sample compact.

Interaction with nickel fission-recoil barriers was studied using samples that had been electroplated with nickel. The samples were then sealed under rough vacuum in aluminum tubing. Reaction of the barriers with the uranium was then simulated by heating the sample.

FIGURE 3.14 Sample Zn/U Compact with (A) No Heat-Treatment and Following Reaction of Zinc with Uranium at 450°C for (B) 16 and (C) 24 Hours

The following observations were made. Nickel and aluminum did not react with uranium foil at temperatures up to 450°C. Zinc-uranium reactions at temperatures below 375°C are minimal. At a temperature of 400°C, the zinc and uranium begin to react. The uranium in these samples is completely consumed at temperatures near or above the melting point of zinc (419.6°C).

Figure 3.14 (B) shows the extensive reaction of zinc with uranium; this sample was heated at 450°C for 16 hours. Figure 3.14 (C) shows a section of the reaction product of zinc with uranium; this sample was heated at 450°C for 24 hours. Samples of the reaction product were analyzed by inductively coupled plasma/atomic emission spectroscopy. The product typically contained 8-10 wt% with the remainder zinc. The zinc-uranium-reaction products were analyzed by electron dispersive spectroscopy, which indicated that some previously unpublished phases may have been formed. However, reaction of the fission-recoil barriers with uranium should not be a problem under normal irradiation and transport conditions.

3.2.1.5.3 Conclusions on Electroplating Ni and Zn Fission Recoil Barriers

The feasibility of electroplating zinc or nickel barrier layers on uranium foils has been demonstrated. The plating conditions can be controlled to provide a barrier layer with a nominal (gravimetric) thickness of 15 μm that also provides complete coverage of the substrate. Although these techniques were developed through work on pure-uranium foils, Argonne's limited work with adjusted uranium has indicated that the transition to this material will proceed smoothly.

Argonne work has also revealed the importance of the surface preparation prior to plating. A roughened surface is essential to bind the plating to the substrate. Excessive dissolution of the foil during etching, however, creates localized crevices that fail to plate in the zinc bath and result in discontinuous coverage. Over-etched foils plated in the nickel bath did not exhibit a loss in coverage; however, the deposits in the crevices often fell below the minimum requirement of 7 μm . The preparation procedure outlined above provides reasonable control of total uranium dissolution. By limiting this loss to <10%, the subsequent plating can be expected to combine full coverage with sufficient adherence.

These experiments have also highlighted a concern about adequate control of the as-plated coating thickness over the full foil. Over-plating at the edges, especially in the nickel bath, may exceed the tolerances needed to maintain the desired fit within the target, and our preliminary indication with larger foils was that the degree of over-plating increases with foil size. Future work addressed changes in the design of the plating bath (i.e., auxiliary anodes, robber cathodes, and perimeter shielding of the cathode) that reduced the center-to-edge variation in thickness for full-sized foils. Figure 3.15 shows a Cu foil placed in a robber-cathode configuration; this configuration minimized over-plating at the edges.

FIGURE 3.15 Copper Foil in a Robber-Cathode Configuration

3.2.1.6 Summary of Argonne Fission-Recoil Work

One of the benefits of the Argonne-designed annular target is the ability to remove the uranium foil from the target cladding, thus lowering liquid-waste volumes by not having to dissolve the entire target and lowering processing times. Fission-recoil barriers are a necessary ingredient to the annular LEU-foil target. If not present, the uranium foil will bind to the target walls and be impossible to remove mechanically.

Electroplating of Ni and Zn fission-recoil barriers was successfully developed to the point that smooth, uniform, well-bonded layers could be consistently plated onto the uranium foils. However, due to the uranium losses during the uranium-foil preparation steps (~5%) and the time required for plating the layers compared to wrapping them in foils, electroplating was abandoned in favor of wrapping the uranium foils in metal foils.

Based on their nuclear, mechanical, and chemical properties, nickel and aluminum were deemed the best materials for fission-recoil barriers. Based on their abilities to stop fission tracks, Ni barriers must be at least 10- μm thick, and Al barriers at least 15- μm thick. Nickel barriers would be used by acidic dissolution and Al barriers for alkaline digestion.

3.2.2 Fission-Recoil Barrier Development at Y-12

3.2.2.1 Electroplating

Work performed at Y-12 on electroplating of uranium foils was published earlier [Holland-2010]. By the end of FY2010, we were able to successfully electroplate a depleted uranium foil with a 10-20 micron layer of nickel. Future work was planned, but we were not able to continue due to infrastructure problems.

3.2.2.2 Physical Vapor Disposition

The PVD system (“ion plater”) consists of a vacuum chamber into which the workpiece (item to be plated) and a target disk of the plating material are loaded. The chamber is evacuated and backfilled with inert gas (typically argon, neon, etc.) to the desired operating pressure. Processing voltages are applied to the workpiece and target. The target is bombarded with ionized (electrically charged) argon, which removes atoms from the target. These atoms are attracted to the workpiece and are deposited on its surface.

The PVD system at the Y-12 Development Facility has been used in the past on various shapes and coating thicknesses. There were no specific development activities related to foil coating. Past processing knowledge was used to PVD the foils.

3.3 FOIL FABRICATION

3.3.1 Background of Argonne Foil Preparation

Argonne has been preparing foils for LEU high-density targets since 1993 [Wiencek-1993]. In a 1994 paper, Wiencek described step-by-step the Argonne process for rolling foils [Wiencek-1994].

3.3.1.1 Foil Rolling

A schematic of this operation is duplicated in Figure 3.16.

FIGURE 3.16 Schematic of Argonne Uranium-Foil Rolling Scheme [WIENCEK-1994]

A typical batch size at Argonne National Laboratory is a 250-300 g ($5\text{ cm} \times 5\text{ cm} \times 0.63\text{ cm}$) ingot made by arc melting. After coating with Y_2O_3 as a parting agent, the ingot is placed in a “picture frame” ($15\text{ cm} \times 15\text{ cm} \times 0.63\text{ cm}$) piece of plain carbon steel with a hole matching the ingot outline in the center. Two $15\text{ cm} \times 15\text{ cm} \times 0.63\text{ cm}$ pieces of plain carbon steel without the center hole are used to clad the picture frame, which contains the ingot. The

three plates are then welded 100% on the edges and hot rolled at 625°C from 1.9 cm to 0.08 cm. After hot rolling and removal of the cladding, the foil is about 0.025-cm thick. The foil is then cold rolled to the final 125- μ m thickness and is sheared to final size. Yields of acceptable foils are typically 50–70% after shearing. The foils are individually vacuum sealed in 125- μ m thick stainless steel bags for beta quenching. The heat treatment includes heating the foils to 700–720°C for 20 min and water quenching. The same heat treatment is then repeated. After removal of the bag, the foil is ready to be assembled into a target.

A later report describes the foil-rolling procedure used at Argonne in detail. It also provides alternative methods that could be used in a large-scale, production operation [WIENCEK-2009].

3.3.1.2 Beta Treatment

Performing β -treatment of the uranium foil after rolling is essential to eliminate the foil texture. A highly textured structure is inevitably formed after the rolling process, which causes undesirable anisotropic growth that leads to undesirable surface effects, such as swelling during irradiation. In general, randomly oriented and moderately fine-grained uranium is necessary for successful irradiation behavior. Cold working of uranium tends to produce a textured preferential grain orientation with the (100) plane parallel to the rolling plane [KAUFMANN-1962]. It has also been shown that highly textured pure uranium will exhibit severe antistrophic growth when irradiated [KAUFMANN-1962]. Growth of this type might cause the foil to bond to the cladding, which would make removal of the foil after irradiation difficult. In addition, the target might warp under irradiation, making its removal from the irradiation position complicated. In the worst case, the anisotropic growth could cause the target cladding to fail during irradiation. A heat treatment has been developed to produce small, randomly oriented grains to provide no texture in the depleted uranium foil [CONNER-1998]. The foil is then heated to 720°C in a vacuum-sealed metal bag, forming the beta uranium phase of uranium metal, and then cooled by dropping the sealed pouch into water. The vacuum is needed to prevent oxidation of the foil and to allow for good contact between the pouch and the foil, so the heat can be rapidly removed. The rapid cooling through the beta-to-alpha crystal structure results in the formation of small and randomly oriented grains.

3.3.2 Foil Preparation at Y-12

Y-12 also has a long history of rolling uranium and alloy foils. The process flow for the Y-12 method is shown in Figure 3.17 and summarized below.

FIGURE 3.17 Y-12 Fabrication Process Flow for Foil Manufacture

Determine Enrichment-Value Target

- Weigh feed materials as needed to achieve the desired ^{235}U enrichment of the uranium foil (see Table 3.8).

TABLE 3.8 Example Feed of Uranium Enrichment

	Enrichment (%)	Mass (g)
HEU	93.155	15.1
DU	0.192	184.9
Target LEU	7.21	200

Casting

- Cast billet of uranium with VIM furnace or arc melter.
- Record casting technique (VIM or arc).
- Record casting ID/name.
- Hot roll using salt bath between 600°C and 700°C until thickness of between 0.20 cm and 0.30 cm.

Cold Rolling

- Cold roll using Fenn or Cauffiell rolling mill.
- Vacuum anneal foils at 600°C and 800°C for 20 minutes to one hour.
- Cold roll until material hardens and resists and achieves further reduction.
- Measure thickness and repeat cold roll and anneal steps until target thickness ($120\text{ }\mu\text{m} \pm 10$) is achieved.
- Cut sample of foil for chemical analysis.

Rough Cutting

- Cut foils to nominal target size plus additional minimum 1/4-inch boundary to allow for later shearing.
- Measure and record foil dimensions.

Quenching

- Wrap foil in tantalum sleeve.
- Load sample into quench furnace.
- Evacuate to operating pressure (10^{-4} torr).
- Heat furnace to 760°C ($\pm 15^\circ\text{C}$); hold for 5 to 30 min.
- Backfill with argon to atmospheric pressure.
- Using trap door, drop quench foils into water.

Annealing

- Place foils in vacuum-annealing furnace for 20 minutes at temperature.
- Repeat the quenching and annealing steps for a total of two cycles.

Shearing (terminal dimension)

- Shear to final size.
- Measure and record foil dimensions.

3.4 ANALYSIS OF FOIL PROPERTIES

For good behavior under irradiation, the uranium grains in the foils must be randomly oriented and small. If not, uranium may grow in one direction and cause a breach of the target. Methods to measure the grain structure are described below.

Three depleted uranium foils were produced as described in Section 3.3.2—cast in the VIM furnace, hot rolled, cold rolled, and heat treated—with the final reductions coming from cold cross-rolling. Foils were turned 90° with each pass from 0.02 cm to 0.01 cm thickness. The foils were investigated by both X-ray diffraction (XRD) and resonant ultrasound spectroscopy (RUS) to gain insight on the mechanical properties of these foils. In addition to XRD and RUS, the foil microstructure was examined via optical microscopy that utilized polarized light. The foil dimensions included a depth of 125 μm and areas as seen in Figure 3.18

FIGURE 3.18 Uranium Foil Cross Sections

3.4.1 X-Ray Diffraction

The XRD utilized a Miniflex II Instrument with measurement conditions of a step at 0.020 degrees at a rate of 0.25-degrees/min and a wavelength of 1.540 angstroms. The measurements were conducted in the x- and y-directions, as seen in Figure 3.19, to provide a diffraction pattern that was plotted as intensity versus d-spacing. Each measurement allowed for two major phases to be identified and indexed, the uranium-metal phase (orange) and the UO_2 phase (green).

The first foil provided three important peaks in the x-direction of the diffraction pattern of uranium phases: $\text{U}(020)$, $\text{U}(002)$, and $\text{U}(200)$, which can be distinguished from their spectrum by yellow, red, and blue circles, respectively. These peaks are representative of the b , c , and a axes, respectively.

Figure 3.20 provides a large graphical representation in the x-direction of the diffraction pattern while Figure 3.21 is a closer look at the features seen in Figure 3.20 with special attention to the absence of $\text{U}(020)$ and $\text{U}(040)$ from the same family. This absence is typical for a cross-rolled microstructure, and the absences represent the b -axis. Both a and c axes are represented by $\text{U}(200)$ and $\text{U}(002)$ when the peaks are present. It is important to note that the same features are displayed on the graphical representation of the y-direction of the diffraction pattern, as seen in Figures 3.22 and 3.23. Similar graphs were obtained for the first and second foils (Figures 3.24–3.27), which yielded a the comparison graph seen in Figure 3.28. This graph illustrates that the spectra were quite similar in that both foils provided similar peaks for $\text{U}(020)$, $\text{U}(002)$, $\text{U}(200)$, and $\text{U}(040)$, and the only difference was the amount of counts for each spectrum. As foils one and two had similar results, XRD was not completed for the third foil.

The XRD results detailed above lend support to the need for a beta quench in the foil fabrication process. This will randomize the grains (make them more isotropically oriented).

FIGURE 3.19 Example of a Diffraction Pattern for a Uranium Foil

FIGURE 3.21 Close Look at the X-Direction Diffraction Spectra of the First Foil

FIGURE 3.22 Second Foil's Overview X-Direction Diffraction Pattern

FIGURE 3.23 Close Look at X-Direction Diffraction Spectra of the Second Foil

FIGURE 3.24 Overview of Y-Direction of First Foil's Diffraction Spectrum

FIGURE 3.25 Close Look at Y-Direction Diffraction Spectra of the First Foil

FIGURE 3.26 Second Foil Overview of Y-Direction Diffraction Pattern

FIGURE 3.27 Second Foil Close Look of Y-Direction Diffraction Spectra

FIGURE 3.28 Comparison of XRD Spectra for Depleted Uranium Foils

3.4.2 Resonant Ultrasound Spectroscopy

The elastic properties of materials are extremely important for many design and engineering applications. Therefore, there have been many experimental methods, both static and dynamic, developed to determine them. Static methods include tensile, compressive, flexural, and torsional tests. Direct measurements of stresses and strains are made, and the elastic moduli determined from the slope of the linear region of the stress-strain curve. Dynamic methods include resonance and pulse methods, and provide advantages over the static techniques due to ease of specimen preparation, a wide variety of specimen shapes and sizes, greater precision, and measurement over a wider temperature range.

Resonant ultrasound spectroscopy, also known as RUS, is a unique and sophisticated non-destructive technique for determining the complete elastic tensor of a solid by measuring the resonant spectrum of mechanical resonance for a sample of known geometry, dimensions, and mass [MIGLIORI-1993, -1997]. The mechanical resonance frequencies of a freely suspended solid object are the special solutions to the equation of motion, which depend on density, elastic moduli, and shape of the object.

One of the fundamental principles behind RUS is to drive the material at well-defined frequencies and to extract precise locations for the resonant frequencies [RADOVIC-2004]. Three piezoelectric transducers support the sample, in the form of a disc or parallelepiped. One of the transducers (transmitting transducer) converts an applied electrical signal to a mechanical stress at the surface of the material, generating an elastic wave of constant amplitude but varying frequency, covering a large number of vibrational eigenmodes of the sample. The resonance response of the excited sample is detected by the other two transducers (receiving transducers). These transducers are responsible for picking up the physical response of the material and converting it back into an electrical signal for data acquisition and subsequent analysis.

From the known sample dimensions, mass, and a set of assumed elastic constants, an approximate spectrum is calculated. The measured spectra of mechanical resonances are compared with the theoretically calculated spectrum. The true set of elastic constants is then calculated by a recursive regression method that varies the assumed elastic constants until the calculated resonant spectrum is matched with the experimental spectrum. A multidimensional algorithm minimizes the root-mean-square (RMS) error between the measured and calculated resonant peaks and enables the determination of the elastic constants from a single frequency scan.

The RUS technique provided an insight into the elastic mechanical properties for both foils one and two. The gathered values of the mechanical properties can be found in Table 3.9.

**TABLE 3.9 Elastic Mechanical Properties
for Depleted Uranium Foils**

Mechanical Properties	Foil #1	Foil #2
Bulk Modulus (GPa)	102.2	144.4
Shear Modulus (GPa)	70.6	71.6
Young's Modulus	172.3	181.3
Poisson's Ratio	0.219	0.266
RMS Error %	7.93	8.92

3.4.3 Optical Microscopy

Optical metallography was performed on the third cross-rolled depleted uranium foil to examine the as-rolled condition of the microstructure. The four images provided in Figure 3.29 examine the uranium foil cross section. The top two images show the microstructure of uranium plus an unknown second phase with an unusual matrix indicative of an intermediate cooling rate, and the bottom two images are the same regions in polarized light showing grain-like colonies of fine cellular decomposed martensite. The images in Figure 3.29 were taken of the as-polished microstructure at magnifications of 200x and 500x in bright-field and polarized light illuminations. The image in Figure 3.30 is a micrograph in the as-rolled direction showing uranium carbide inclusions dispersed throughout the microstructure taken at 1000x magnification.

FIGURE 3.29 Optical Microscopy of the Third Depleted Uranium Foil

FIGURE 3.30 Micrograph at 1000X of Uranium Carbide Inclusions in the “As-rolled” Direction

3.5 ANNULAR TARGET ASSEMBLY

High-density annular target assemblies consist of two concentric annular aluminum tubes drawn together with a fission-recoil-wrapped uranium foil embedded within a pocket on the outer surface of the inner tube. The function of the target is to contain fission products during irradiation and during transport to a processing facility (hot cell). Since mechanical properties of target materials change with temperature, most times weakening the material structure as the temperature increases, it is necessary to ensure that there is low thermal contact resistance between the heat-generating LEU foil and the cladding material. The challenge is to maintain a target structure that will allow the foil to be removed from the target after irradiation for processing, and still have the very intimate mechanical contact consistent with low thermal contact resistance. Further, to make the target easier to assemble, the tube dimensions and tolerances should allow the tubes to easily slide past each other while keeping the LEU foil properly positioned. Still, the fit needs to be snug to facilitate end sealing.

The general process taken to prepare the target for consolidation is illustrated in the steps shown in Figure 3.31. The first step is to machine the inner and outer tubes to the proper dimensions, trim the LEU foil to the desired size/mass, and prepare the fission-recoil barrier. In this series of steps, it is assumed that the fission-recoil barrier is provided by a foil that is wrapped around the LEU, as illustrated in steps 2–4. It is possible to have the fission-recoil barrier applied directly to the LEU foil by electroplating or PVD or any other appropriate coating process. After the fission-recoil barrier has been applied to the LEU foil, the prepared foil is wrapped around the inner tube in the recess that has been machined. The outer tube needs to slide past the inner tube, as illustrated in steps 5 and 6. Care needs to be taken to ensure that the fission-recoil barrier does not get torn. It is also necessary to ensure that the foil twists between the inner and outer tubes as the positioning of the foil and the location of the gap between the ends of the foil need to be controlled. After the tubes have slid past one another with the LEU and fission-recoil barrier properly positioned, the location of the gap between the ends of the LEU is marked on the outer tube for future reference. The target is now ready for consolidation and end sealing. Figure 3.32 is a larger image of the target during insertion.

FIGURE 3.31 Target Assembly Steps with Foil

FIGURE 3.32 Target Assembly Showing Scribing Mark on Tube

3.6 TARGET CONSOLIDATION

Once the inner tube is inserted into the outer tube, it must be drawn into the outer tube such that the inner tube is expanded plastically, and the outer tube is expanded elastically. This creates a target in which the uranium foil is held tightly between the two tubes, effecting good heat transfer within and out of the target.

The assumed initial state of the target components before consolidation is shown on the left-hand side of Figure 3.33. The gap between the inner and outer tubes is to allow the inner tube to easily slide inside the outer tube. One option to consolidate the target components is to expand the inner tube into the LEU foil and the outer tube. The inner tube must be expanded plastically while the outer tube allowed to deform only elastically. This will allow the inner tube to remain in contact with the outer tube and LEU after the expanding force is removed.

FIGURE 3.33 Target Consolidation Using an Expansion Process

3.6.1 Simple Draw Die Consolidation

An example of a simple draw die assembly is shown in Figures 3.34–3.36. The interior of the draw die is cleaned using a wipe and isopropyl alcohol. Both the draw plug and interior of the target are lubricated with a graphite/alcohol lubricant without coating the ends of the tube. Tubes are then pulled through the draw die assembly with a draw plug optimized for target geometry.

FIGURE 3.34 Draw Die Hardware

FIGURE 3.35 Assembled Draw Die with Target and Draw Plug Exposed

FIGURE 3.36 Cut-Away Sketch of Draw Die Assembly

3.6.2 Alternative Assembly Methods

To ensure sufficient heat transfer from the heat-generating LEU foil and the target cladding material, interfaces between the LEU foil and the tube cladding need to be managed. In particular, the thermal contact resistance needs to be sufficiently low to ensure that the LEU foil does not exceed reactor safety limits. The gaps between the tube and LEU/recoil barrier foils that are necessary to facilitate assembly need to be removed through a target consolidation process. Two consolidation techniques were explored as part of the program—an advanced draw-plug expansion and hydroforming. These techniques and others are discussed below.

3.6.2.1 Draw Plug

One strategy to expand the inner tube into the outer tube is to use a draw-plug process. The draw-plug process uses a solid draw plug made from hardened tool steel that has a cone-shaped geometry with a base diameter slightly larger than the inside diameter of the inner tube. Examples of such plugs are shown in Figure 3.37. The plug is driven through the interior tube of the target as indicated in Figure 3.38. Mechanical support around the outer tube is provided by a hardened tool steel die. While the sketch in Figure 3.38 exaggerates the amount of deformation in the outer tube caused by the draw plug, the draw plug needs to be designed to make sure that no plastic deformation occurs in the outer surface of the outer tube. Elastic deformation is acceptable as long as the target can be easily removed from the die after the draw plug has been completely drawn through the target.

FIGURE 3.37 Examples of Draw Plugs Used for Target Drawing Process

FIGURE 3.38 Draw Plug Being Drawn through Target to Consolidate the Tubes

The means used to drive the draw plug through the target in the current study is with the aid of a hand-driven hydraulic pump/cylinder assembly. As shown in Figure 3.39, the hand pump and hydraulic cylinder are mounted onto a steel plate. The hydraulic cylinder was aligned with the draw die as shown in the figure. The drive rod connecting the hydraulic cylinder with the draw plug is held in alignment with a sleeve bearing. A back plate is mounted at the end of the draw die to prevent the target from being pushed out of the die by the draw plug. After the plug has been drawn through the target and the drive rod withdrawn back, the draw die can be rotated out of alignment using the hinged cradle, and the target removed from the die.

In the study presented in Section 3.7.1.1, the target geometry and draw plug size were explored in depth. Using a statistical experimental design approach, we estimated the geometric tolerances. Some model matching studies were also conducted. Aluminum 6061-T6 was used as the prototypical material with some extrapolations to other aluminum grades made. Further analysis for other materials or target geometries should be possible by using scale modeling and the experimental measurements presented.

FIGURE 3.39 Schematic of Draw-Plug Consolidation System Used for Current Study

3.6.2.2 Hydro-Forming

A second expansion process that can be used to consolidate that target is hydro-forming. Hydro-forming applies a high-pressure fluid to the inside of the inner tube, which causes the expansion of the inner tube. The primary conceptual difference between the draw-plug and the hydro-forming methods for target consolidation is the manner in which the radial force is developed. For the draw-plug method, linear motion of the draw plug through the target is converted into a radial force through the geometry of the draw plug. A necessary artifact of the draw-plug method is frictional heat generation between the draw plug and inner tube, as well as inner tube elongation. The hydro-forming method applies radial force directly, and thus does not generate any frictional heating, nor does the inner tube elongate. Preliminary analysis of hydro-forming has been completed as part of this study using the plug-and-die fixture shown in Figure 3.40. It is still of interest to have the inner tube deformed plastically and the outer tube deformed only elastically to facilitate disassembly.

FIGURE 3.40 Photograph of Hydro-Forming Die and End Plugs Used for Current Study

3.6.2.3 Tube-Compression Technology

Figure 3.41 illustrates a second manner in which the target tubes can be consolidated by a radial compression process. In this case, the outer tube is compressed into the foil and inner tube. One challenge with this approach is having the outer tube develop wrinkles as the outer tube is compressed.

FIGURE 3.41 Method of Target Consolidation Using a Compression Process

3.6.2.4 Swaging

Rotary swaging is one technique that could be used to compress the outer tube. Many times a mandrel is placed inside the inner tube to provide structural support so that the inner tube does not collapse. The general approach uses a rotary fixture that has wheels which make contact with the outside surface of the outer tube. There are usually at least four wheels, located uniformly around the outer tube. As the wheels roll around the circumference of the outer tube, hammers pound the wheels into the outer tube. The continual pounding as the wheels rotate around the outer tube compresses the tubing. The target is then slowly drawn through the swaging wheel until the entire length of the target has been compressed. This technique has not been explored in this project.

3.6.3 End Sealing Technology

The second part of the assembly process is end sealing. Sealing the ends of the two tubes prevents any fission products from leaking out of the target during irradiation and during transport to the hot cell for processing. Welding is typically the method of choice for sealing containers to be irradiated, although cold pressure forming could also be considered.

3.6.3.1 Welding

Two different welding strategies were evaluated as part of this study. Aluminum is a notoriously difficult material to weld due to the rapid formation of an oxide layer on its surface. Cleaning is very important to obtain quality welds.

Tungsten-inert-gas (TIG) welding is a common welding approach for aluminum. It is the lowest cost and fastest welding technique examined in this research. The process uses a torch that is surrounded by a layer of an inert gas to help reduce the formation of oxides on the heated surface. A filler material is commonly used. The process also happens to be dependent on the skill of the welder. For the results presented in Section 6.3, all samples were welded by a master welder at the University of Missouri.

A second welding type that was explored as part of this study is electron beam welding (EBW), a fusion welding process in which a beam of high-velocity electrons is directed toward the two materials to be joined. The EBW method is done in a vacuum environment, and thus it needs to be done remotely and generally requires a significant capital investment to purchase the equipment. Electron beam welding can be used to make a very high quality weld and, if batched correctly, could be scaled up to production levels of target production. A technician at ORNL was able to successfully prepare EBW samples.

The type of aluminum alloy utilized (1000, 3003, and 6061 for this project) greatly affects the welding methods to be used. For the targets to be irradiated in the High Flux Isotope Reactor (HFIR), an autogenous weld was initially specified with 6061 aluminum, and this resulted in a very difficult weld qualification process. In general, the 1000 and 3000 types of aluminum were found to be straightforward to weld and are recommended for use.

3.6.3.2 Preparation of Targets for Irradiation in the High Flux Isotope Reactor

While all of the annular LEU-foil targets irradiated in Indonesia, Argentina, Australia, and MURR since 1994 either had dry air or vacuum environments inside the cladding, the HFIR targets were required to have helium inside to enhance thermal conductivity. This resulted in additional steps required to encapsulate and weld the targets that are typically not necessary for the standard target.

To ensure the presence of helium gas in LEU targets manufactured at Y-12 for the HFIR targets, a combination of EBW and gas tungsten arc welding (GTAW, aka TIG) was developed. After assembly, annular targets were welded using flat edge joint seal welds performed with EBW and GTAW. Welds were qualified by virtue of macro sections of each weld and hydrostatic performance testing.

The drawing process of the concentric annular tubes causes a slight unevenness in the lengths of the two tubes. After assembly, each end is machined so that the ends are flush to the specified length. The exterior of the target was cleaned with acetone or a lint-free cloth dampened with alcohol. Careful attention was given to keeping the cleaning fluids from the

interface between the inner and outer tubes, since wicking of these fluids may result in unintended liquids surrounding the foil, which may be difficult to remove, even in a vacuum chamber.

Electron beam welding is accomplished in a vacuum chamber. The entire circumference of the target is welded on one end, the target is rotated, and the other end is welded to create, theoretically, a vacuum in the target's interior. The welded target is transferred to a glovebox filled with high purity helium. Using a rotary tool with 1/8-in. diameter carbide end mill, a small notch (approximately 1/8-in. deep $\times$ 3/16-in. wide) is made through the weld on the end with the machined gas channel. The notched target is then moved from the loading/unloading vestibule of the chamber, where it will be evacuated to 2×10^{-2} torr and pressurized to +15 psi (with helium) for three cycles, each with a 30-minute hold. This step ensures helium is present inside the target and can be credited in thermal-hydraulic safety calculations for determining the heat removal capabilities of the target.

After three cycles of backfilling helium, the target is returned into the main chamber for GTAW. The seal weld occurs under high-purity helium shielding gas, while argon shielding gas is supplied through the welding torch. The notch is welded, sealing the gas channel, and removed from the chamber for leak testing. Weld filler for this GTAW is typically 4047 or 4043 aluminum for 6061 aluminum target tubes, but for 3003 aluminum, no filler may be required, as it is more amenable to autogenous welding.

Figures 3.42–3.44 depict in various ways the chill used for GTAW. Figure 3.45 is a diagram showing the notch cut into the target, and Figures 3.46 and 3.47 are photographs of the GTAW of the notches.

FIGURE 3.42 Copper Chill Diagram

FIGURE 3.43 Assembled Target in Copper Chill

FIGURE 3.44 Outside Diameter Chill of Target

FIGURE 3.45 Scribe line, GTAW Notch, Groove, and Electron Beam Weld

FIGURE 3.46 Target with Three GTAW Notch Welds

FIGURE 3.47 Metallographic Examination of GTAW Notch Welds

3.7 FABRICATION ANALYSIS

During the target irradiation, the fission process generates heat that needs to be dissipated to the reactor coolant pool. The generated heat increases the target temperature, and the increase in temperature causes thermal expansion of the target materials. Additionally, during irradiation, the materials in the target thermally expand in proportion to their change in temperature. Since the LEU and the aluminum have different thermal expansion coefficients, the magnitude of their thermal expansion will differ. The thermal expansion mismatch changes the internal stress distribution of the target and has the potential to lower the contact pressure between the LEU and cladding, or even allow open gaps between the aluminum cladding and the uranium foil and the fission recoil barrier to develop.

Lower uranium-cladding contact pressure or gaps that open up at an interface between the two materials will increase thermal contact resistance and thereby increase the temperature of the LEU foil. Since reactor technical specifications include a limit on maximum temperature, having an increased thermal contact resistance will increase the risk of the LEU temperature exceeding reactor operating limits. Understanding the temperature of the LEU foil during normal irradiation and during off-normal conditions is important for qualifying the target for reactor irradiations. In particular, LEU foils have exhibited uncontrolled growth at certain irradiation temperatures and for LEU materials that are fabricated in certain ways [HOFFMAN-1996, SNELGROVE-1999]. It is, therefore, necessary to fully understand the mechanical state/geometry of the target during irradiation to ensure compliance with reactor safety

specifications and to better understand foil performance during irradiation. This section will provide an overview of the mechanical state at various points in the life of a target, ranging from the fabrication process steps discussed in Section 3.5 to the irradiation effects, including heating, fission gas generation, and LEU swelling.

3.7.1 Target Consolidation

The target consolidation step of the target fabrication process mechanically deforms the aluminum tubing to establish good contact between the target components. It is an important part of the irradiation modeling as the initial stress state of the aluminum tubes and LEU foil for the model is established by the residual stresses that remain after target consolidation. This subsection will describe the experiments carried out to study both the draw plug and hydroforming methods of target consolidation.

3.7.1.1 Draw Plug Consolidation

The draw plug consolidation method used at the University of Missouri has been described in previous publications [EL-GIZAWY-2011A]. A sketch of the overall configuration of the consolidation rig was shown in Figure 3.39. It comprises a hydraulic pump, a hydraulically driven jack, a ram, the draw plug, and the draw die. The draw die is attached to a hinge such that it can be swung into and out of alignment with the draw plug ram to facilitate placing and removing the target into and out of the die.

The consolidation approach uses a sub-assembly of the LEU and fission-recoil barrier foils and aluminum-tube cladding, as described previously in this document. The inner surface of the target is sprayed with graphite for lubrication. The lubricated sub-assembly is placed into the draw die, and the draw die swung into alignment with the draw plug/ram assembly. The hydraulically driven draw plug is pushed through the target. After the plug is driven entirely through the target, the draw plug is removed from the ram, the ram is withdrawn back through the target, the draw die is swung out of alignment with the ram, and the consolidated target is removed from the die. The ends of the consolidated target are then sealed by a separate welding process. Figure 3.48 shows photographs of various steps throughout the consolidation process:

- Top left shows the entire rig.
- Top center shows the target already in the draw die and the draw plug being guided into the target.
- Top right shows the hand-driven hydraulic pump being used to drive the draw plug through the target.
- Bottom left shows the consolidated target being withdrawn from the die, after the draw die has been swung out of alignment.

- Bottom right photo shows the consolidated target with a mark drawn to indicate where the disassembly cutter is to open the target.

FIGURE 3.48 Target Consolidation Steps Using University of Missouri Draw Plug Rig

A study was carried out to systematically explore and optimize the target assembly process with respect to certain target design parameters [EL-GIZAWY-2011B, EL-GIZAWY-2012, HOYER-2013A, MONTGOMERY-1996]. A robust experimental design, also known as the Taguchi method, is a cost effective means to quickly explore a parametric design space. This factorial approach that uses statistical data analysis gives a functional expression that relates metrics that describe target behavior, like residual stress, to the input parameters [MONTGOMERY-1996].

The experiment was designed to have four factors with three levels each. The factors include the amount of deformation (i.e., size of the draw plug diameter and deformation angle), relief area depth, disassembly cutting gap (distance between foil ends after wrapped), and distance from target ends to the relief area. The levels included low, medium, and high. The importance of testing at these levels was to find the factor that is statistically most significant for target consolidation, and to eliminate factors that are not statistically significant. Table 3.10 lists the factors with their different levels. Figure 3.49 is a sketch illustrating the factors and other geometric design parameters. The experimental matrix combinations are shown in Table 3.11. The matrix was replicated five times for a total of 40 targets built and evaluated.

TABLE 3.10 Robust Design Experimental Factors and Levels

Factors	Number of Levels		
	1	2	3
Draw plug diameter, mm	26.57	26.59	26.62
Depth of relief area (t_R), mm	0.15	0.18	0.20
Cutting gap (g), mm	12.70	7.62	3.81
Distance from target end to relief area (W_e), mm	34.85	19.05	12.70

FIGURE 3.49 Sketch of Target with Some Design Parameters Identified

While the factors in Table 3.10 were varied for this study, all other parameters that can impact the target state were held constant. Other than modifying the relief area, tube geometry was not changed. For the inner tube, the inside and outside diameters were kept at 25.27 mm and 27.77 mm, respectively. For the outer tube, the inside and outside diameters were kept at 27.90 mm and 29.76 mm, respectively. The cladding tubes were made from 6061-T6, since this is the material HFIR requested for its target irradiations. Estimates for target behavior of other materials can be made by using numeric models that are calibrated from the experimental data shared here.

The nickel fission-recoil barrier used for all the experiments was 0.015-mm thick, and the LEU surrogate foil used was stainless steel with 0.150-mm thickness. Because uranium foils were not practically available for these experiments, stainless steel was used as a surrogate. A study at Pacific Northwest National Laboratory found that stainless steel was the second most prototypic material as a surrogate for LEU based on mechanical properties such as hardness and Young's modulus [DELEGARD-2004]. The best material from their study was tungsten, which was also not practically available for our experiments.

The output metrics tracked in this analysis were the forming pressure, residual stresses, roundness, and presence of defects, such as cracks, unnecessary gaps, and heat effects from welding. Table 3.11 describes the conditions used to draw the nine samples that were submitted for residual stress analysis reported below. For a full description of the experimental program and full results of this study, the reader is referred to the MS thesis of Annemarie Hoyer [HOYER-2013B].

Residual stresses are those that remain in the target cladding after consolidation. This post-forming state is important as it can either enhance or detract from the state of the interface during irradiation and the disassembly process. Because the target is cut longitudinally for disassembly, a tensile residual stress state in the outer tube is desired, such that the outer tube “springs” open instead of collapsing closed after being cut. A tensile hoop stress will also help maintain contact between the LEU and the cladding. It should be noted that the targets undergo some stress recovery during welding due to accompanying welding-induced heat; however, since heat is focused on the target ends, and welding occurs relatively quickly, the foil region of the target does not generally experience annealing.

TABLE 3.11 Experimental Log for Parametric Analysis

Exp. No.	Draw Plug Diameter (mm)	Relief Depth (mm)	Gap (mm)	Distance to Relief Area (mm)
1	26.5684	0.15	12.70	34.85
2	26.5684	0.18	7.62	19.05
3	26.5684	0.20	3.81	12.70
4	26.5938	0.15	7.62	12.70
5	26.5938	0.18	3.81	34.85
6	26.5938	0.20	12.70	19.05
7	26.6192	0.15	3.81	19.05
8	26.6192	0.18	12.70	12.70
9	26.6192	0.20	7.62	34.85

Residual stresses are those that remain in the target cladding after consolidation. This post-forming state is important as it can either enhance or detract from the state of the interface during irradiation and the disassembly process. Because the target is cut longitudinally for disassembly, a tensile residual stress state in the outer tube is desired, such that the outer tube “springs” open instead of collapsing after being cut. A tensile hoop stress will also help maintain contact between the LEU and the cladding. It should be noted that the targets undergo some stress recovery during welding due to accompanying heat.

X-ray diffraction was used to evaluate the stress in the target's outer surface. The measurements were taken at seven locations, axially on the outer surface of the target as suggested in Figure 3.50, where each of the black arrows represents a location for a measurement. The azimuthal location was directly opposite the gap in the LEU foil.

FIGURE 3.50 Measurement Locations for XRD

Table 3.12 identifies the specifications for the XRD system used at ORNL. Briefly, a single-axis (β) goniometer was employed for the stress measurements using the “ Ω -goniometer geometry” [NOYAN-1987]. A goniometer is an instrument that either measures an angle or allows an object to be rotated to a precise angular position. The (311) crystal lattice structure reflection from the aluminum phase was used for the strain measurements. During scanning, the incident beam and detectors were oscillated $\pm 3^\circ$ about the β axis to improve particle statistics. Specimen alignment was accomplished with a contact probe. Goniometer alignment was ensured by similarly examining an aluminum powder puck (low stress). The maximum observed peak shift for the (331) reflection of Al ($2\theta = \sim 150^\circ$) was less than $2\theta = 0.04^\circ$ for Ω tilting, as described in Table 3.12. (The XRD measurements were done by Dr. Thomas Watkins at ORNL.)

TABLE 3.12 Specifications for the PROTO iXRD System at ORNL

Parameter	Condition
Equipment	PROTO iXRD Portable Residual Stress Measurement System MGR40 goniometer head. Two position sensitive scintillation detectors, $2\theta = 18.5^\circ$ each.
Power	80 W, 20 kV, 4 mA
Radiation	Co K α , $\lambda = 1.788965 \text{ \AA}$
Aperture size	0.5, 1.0, 2.0, 3.0, 4.0 mm round, 0.5×3 , 0.5×5 , 1×5 , 1.5×5 , 2×5 mm
Source to specimen distance	96 mm
Specimen to detector distance	40 mm
Tilt axis/axes	Ω , a max of 11 ψ tilt/detector: -41.8 to $18.2^\circ \psi$ and -18.2 to $41.8^\circ \psi$
Oscillation axis/axes X, Y, Z, azimuthal ranges	Each with 132 mm, $\pm 180^\circ \phi$
2θ scans	$0.036^\circ 2\theta$ step/channel, range 149.8 to $168.3^\circ 2\theta$, 10 scans at 0.5 sec/scan

The data were analyzed with the XrdWin software. The peaks were fit with a Pearson VII function to the upper 85% of the peak to determine their location. The fully expanded equation relating strain to stress in terms of the goniometer tilt and axes was derived with XrdWin (PROTO Manufacturing, Ltd., Oldcastle, Ontario).

The measurements were analyzed with statistical response tables and plots of the mean effects for each factor as they influence the residual stresses of the assembled target. The response analysis helps in identifying those factors that have the greatest impact on process variability and the level of performance. In determining this, we used the signal-to-noise (S/N) ratio analysis. The transformation method was used to convert the measured response into an S/N ratio. The S/N ratio serves as a performance measure to optimize a process. It not only describes the magnitude of a change in output metric for a given change in input factor, but also accounts for the variability of experiments. In this way, any optimization is the most robust, meaning least sensitive to changes in factors.

Calculation with a two-dimensional ABAQUS model indicated that hoop stresses should be 43 MPa in order to ensure no air gaps and good foil contact. This was the value used for the “nominal-is-best” analysis. The transformation method to convert the measured response into an S/N ratio for this criterion is given by:

$$S/N = \eta = -10 \log (MSD) \quad (\text{Eq. 3.3})$$

where the *MSD* term is the mean square deviation, given by

$$MSD = (\mu - t)^2 + \sigma^2 \quad (\text{Eq. 3.4})$$

Therefore,

$$S/N = 10 \log \left(\frac{\mu^2}{\sigma^2} \right) \quad (\text{Eq. 3.5})$$

where $\mu = \text{mean} = \frac{1}{n} \sum_{i=1}^n y_i$, and $\sigma^2 = \text{variance} = \frac{1}{n-1} \sum_{i=1}^n (y_i - \mu)^2$. Additionally, the mean effect of each factor may be calculated from the following equation:

$$m = \frac{1}{n} \sum_{i=1}^n \eta_{xi} \quad (\text{Eq. 3.6})$$

Table 3.13 displays the three factors measured at the seven locations in Figure 3.50 and S/N calculations for the hoop stress. This represents one complete set of nine targets, or one experiment log.

The mean effect of each factor was derived from the data in Table 3.13 and is shown in Table 3.14. The highlighted value indicates the best result. The analysis of variance (ANOVA) technique [SCHEAFFER-2007] was used to predict the relative significance of the four design factors. It gives the percentage of contribution of each factor to the metric of residual stress, thus providing a quantitative measure of the effect of various factors on design performance. The ANOVA results are shown in Table 3.15, where the best condition is also highlighted. Here, the

column headers are the degrees of freedom (DF), total sum of squares of the deviation from the mean values for all levels of the factor (S_x), the mean square due to the factor (V_x), variance ratio used to measure the effect of the factor compared with the random effect due to error (F), and percent contribution to the assembly process (ρ).

Of the four design factors examined in this study, the draw plug diameter was found to be the most important for controlling the amount of hoop stress, followed by gap size and the relief depth. Distance to the relief zone was found to have the least effect.

This assessment process was repeated for every output metric measured to fully describe the most significant factor for the cases studied. Considering all of the output metrics, and placing the most weight on what the residual stress is and whether or not any gaps remained after cross-sectional analysis, we determined a “best” target geometry. The drawings for the inner and outer tubes of the “best” target (Figures 3.51 and 3.52) required the use of a 26.5938 mm diameter draw plug. It should be re-iterated that this design was derived only considering the factors varied in this study. Further analysis will be needed if other geometric configurations might be of interest (inner or outer tube diameter, for example).

A more in-depth description of the experiment that was just reviewed, along with other studies including a Monte Carlo analysis used to estimate tolerances, can be found in the MS thesis of Annemarie Hoyer [HOYER-2013B].

TABLE 3.13 Factor Measurements and S/N Calculations for Hoop Stress

Trial #	Factors Tested at Level 1, 2, and 3				Meas. 1 (MPa)	Meas. 2 (MPa)	Meas. 3 (MPa)	Meas. 4 (MPa)	Meas. 5 (MPa)	Meas. 6 (MPa)	Meas. 7 (MPa)	S/N (no units)
	Draw Plug Diameter	Relief Depth	Gap	Distance to Relief								
1	26.5684 mm	1	1	1	-56.43	-43.53	-30.34	-30.69	-11.42	-0.14	-34.3	-42.8955
2	26.5684 mm	2	2	2	-31.49	2.51	33.97	-23.61	-43.29	-22.66	-72.16	-44.8422
3	26.5684 mm	3	3	3	-55.5	-21.91	-9.81	-20.7	-41.53	6.64	-73.1	-44.5317
4	26.5938 mm	1	2	3	14.06	-19.95	9.83	-28.12	-33.58	61.67	-31.09	-42.4560
5	26.5938 mm	2	3	1	-43.63	-7.29	-3.29	1.23	2.99	-6.26	8.28	-39.4037
6	26.5938 mm	3	1	2	11.56	22.72	-37.58	-11.59	8.54	38.62	22.32	-37.5906
7	26.6192 mm	1	3	2	57.48	51.17	6.71	-14.44	16.92	-7.9	-119.34	-45.2051
8	26.6192 mm	2	1	3	48.82	12.04	11.18	20.29	1.37	12.3	-19.11	-38.6542
9	26.6192 mm	3	2	1	-42.5	-59.69	0.73	16.67	39.53	-16.06	12.64	-38.9879
Average												-41.6186

TABLE 3.14 Mean Effect of Each Factor for Residual Hoop Stress (no units – S/N)

	Draw Plug Diameter	Relief Depth	Gap	Distance to Relief
1	-44.0898	-43.5189	-39.7135	-40.4290
2	-39.8168	-40.9667	-42.0954	-42.5460
3	-40.9491	-40.3701	-43.0469	-41.8806
Diff.	4.2731	3.1488	3.3334	2.1169

TABLE 3.15 ANOVA Results for Residual Hoop Stress

Source	DF	Sx	Vx	F	p
Draw Plug Diameter	2	88.22	44.11	1.09	41.47%
Relief Depth	2	50.35	25.18	0.62	23.67%
Gap	2	53.07	26.54	0.65	24.95%
Distance to Relief	2	21.09	10.55	0.26	9.92%
TOTAL	8	212.74	26.59	—	100.00%
ERROR	4	162.38	40.60	—	—

FIGURE 3.51 Part Drawing for Recommended Inner Tube

FIGURE 3.52 Part Drawing for Recommended Outer Tube

Major conclusions are as follows:

- The roundness tolerance for inner tube internal diameter is recommended to be 0.002 in. (0.051 mm); inner tube outer diameter, 0.001 in. (0.025 mm); outer tube internal diameter, 0.001 in. (0.025 mm); and outer tube outer diameter, 0.002 in. (0.051 mm).
- The deformation size that gave the best results was 1.047 in. (26.59 mm), the medium draw plug.
- As a result of the medium plug size, the hoop residual stresses were nominalized.
- Based on the recommended draw-plug size, there should be very few air gaps at the interfaces between the LEU foil and cladding.
- GTAW can be effective if a well-trained welder is available. For high volume, repeatable welds, however, EBW is recommended if the equipment is available, and if the reactor can accept vacuum conditions in the gas gaps between the LEU foil and cladding.

3.7.1.2 Hydroforming

An alternative approach to expand the inner tube into the outer tube is through a hydroforming process. Hydroforming introduces high-pressure fluid into the interior of the inner tube and allows plastic deformation of the inner tube to take place. The outer tube is held in position by using a restraining die, as shown in Figure 3.53. After the target has been placed in the die, end plugs are introduced into the ends of the target. One of the plugs is the end plug, which is just a solid piece of aluminum that seals into the interior of the target with the use of O-rings. The other plug is the fill plug, which also seals into the interior of the inner tube with the use of O-rings. However, there is a hole drilled through the center of the plug that allows the hydraulic fluid to be introduced into the target. A threaded rod connects the two plugs and allows the user to control how much pressure is needed to hold the plugs into the target and to seal the O-rings. A threaded fitting from the hydraulic pump is introduced into the fill plug.

FIGURE 3.53 Sketch of Hydroforming System Built at the University of Missouri

A photograph of the prototype hydroforming system is shown in Figure 3.54. The restraining die is built in two pieces such that it can be bolted together to hold the outer tube of the target. A photo showing the target placed in the die, before the end plug is inserted, is provided in Figure 3.55. After the end plug was tightened on the threaded rod, hydraulic oil was introduced into the inner tube, and the hydroforming system was pressurized by using a hand pump.

FIGURE 3.54 Target Placed on Hydroforming Die with Filler Plug Inserted

FIGURE 3.55 Photograph of Target Placed in the Die before Insertion of End Plug

The set-point pressure for the hydraulic fluid was determined from the results from a simple numeric model of two concentric aluminum tubes for which plastic material properties were assumed. With this model, a uniform pressure load was applied to the inner wall of the inner tube, and the resulting states of the inner and outer tubes were observed. The pressure load increased until the inner tube finally plastically deformed in the model, and closed the gap with the outer tube. The lowest pressure that satisfied that criterion is 36 MPa (see Figure 3.56). This value was used for the actual prototype assembly.

FIGURE 3.56 Numeric Simulation using ABAQUS to Determine the Pressure Load That Plastically Deforms the Inner Tube and Closes the Interfacial Gap

Figure 3.57 presents an example of one of the targets consolidated by using hydraulic oil-based hydroforming and a fluid pressure of 36 MPa. The photograph of the interior of the inner tube clearly shows the outline of the stainless steel surrogate foil. That qualitative observation suggests good mechanical contact between the cladding and the foil. One drawback of the current technique is that some of the hydraulic oil wicks onto the ends of the tubes and, presumably, into the interface between the two tubes. Clearly, this wicking is an issue when considering that the target will eventually be irradiated. Alternative fluids and fluid-containing strategies are topics for further analysis

FIGURE 3.57 Photograph of Hydro-formed Target Showing Outline of LEU-Foil Surrogate

3.7.2 End Sealing

Sealing the target ends is crucial so that fission products remain contained until the target is opened for processing. Both TIG welding and EBW were examined as part of this study. All TIG-welded samples were done by a former technician at the University of Missouri (Brian Samuels), while EBW samples were successfully done without filler by Alan Fredericks, a technician at ORNL in the Materials Division.

For EBW samples, the weld is created under vacuum conditions to prevent dissipation of the electron beam. With the target under vacuum, dust particles and other potential contaminants can lead to weld defects. The weld is typically clean and precise, and does not leave a mound of welded material around the edges, known as a “hump,” that is visible with the TIG-welded targets. All samples were examined with a Nikon SMZ-10 stereomicroscope with a 115 V AC Fiber Optic Light source. The sample photos shown in Figure 3.58 were taken with a Polaroid digital microscope camera having a HRD-100-CMT lens.

FIGURE 3.58 Comparison of TIG-Welded Sample (left) to EBW Sample (right)

Both TIG-welded and EBW targets were difficult to weld due to contaminants left between the tubes. Contaminants included, but were not limited to, paper particles, dust, oxide layers, leftover lubricant, and leftover cleaning substances. A neutral pH fluid can be used to clean the targets, followed by a volatile liquid to effectively evaporate and leave a dry clean surface. The TIG-welded surfaces exhibited some gaps and other “blow out” evidence, as can be seen in Figure 3.58. Repairs could be made to the TIG welds with additional passes. The TIG-welded samples tended to show some cracks and significant mounding when cross-sectioned.

Each EBW sample was prepared by retracing the weld three times. The linear welding rate and beam energy could be controlled such that less energy could be used to minimize the heat-affected zone. The cross sections of the EBW samples show relatively little mounding, and the ends generally did not show significant blow out. For these reasons, we recommend EBW be used if the equipment is available. While TIG welding has been demonstrated to be effective at sealing the targets such that they can pass leak-tests and successfully contain fission products during irradiation, it poses difficulty in obtaining quality welds in high volume production, although that problem could be overcome by good quality control.

3.8 FABRICATION OF CAPSULES AND TARGETS FOR HFIR IRRADIATIONS

To support chemical processing trials using irradiated materials, we launched two separate efforts to irradiate LEU foils. One method would support nitric acid dissolution testing using equipment and processes developed at ANL in a hot cell located at ORNL. The second method would support electrochemical dissolution in another ORNL hot cell, but also be performed by ANL. Each of these efforts had different requirements, from target design to shipping.

3.8.1 Small Capsule Targets for HFIR and Nitric Acid Dissolution Testing

To perform hot demonstration of the nitric-acid dissolution system (Section 9) in an ORNL hot cell, we needed a pre-determined quantity of unirradiated depleted uranium to be added to the dissolver to simulate full-batch production. To create a spiking sample, we also needed that an irradiated target be quickly removed from the reactor and moved from the reactor pool to the hot cell facility. Ideally, full-size annular targets would be used; however, the irradiation locations in HFIR make it impossible to quickly remove a full-length target. Additionally, the irradiation location makes it impossible to remove the target prior to completion of a full 25-day HFIR cycle.

To provide Mo-99 to the hot cell for dissolution testing, we devised a new capsule that would allow at-will insertion and removal from the HFIR by means of the hydraulic tube. This “rabbit” capsule is based on an existing housing design, with one key difference being that the housing material is fabricated from high purity (99.995%) nickel, and there is an internal spring-sheet, also made of nickel, that applies pressure to the inside of the LEU foil. The high purity nickel allows the capsule to be directly dropped into the nitric acid bath without removing the LEU foil first, negating the need for capsule-opening equipment in the hot cell.

The requirements for the nickel included purity as well as hardness. The nickel used for the capsule housing and cap were required to be fabricated from half-hard nickel, which is achieved via a rolling or forging process, but greatly increases the mechanical properties of the nickel. Half-hard, high-purity nickel is easily purchased, and the additional hardness makes machining simple.

One advantage of nickel is that it is readily autogenously welded, but in case of a need for filler material, high purity nickel wire is also readily available. Our capsules did not require filler wire.

For this irradiation, LEU at 19.5% enrichment would generate entirely too much heat in the hydraulic tube facility of the HFIR (thermal flux of 1.5×10^{15} neutrons/cm²-sec in the uppermost position). To accommodate the heating that occurs in this facility, the enrichment of the U foil was reduced to that of natural uranium (0.7142 wt% ²³⁵U).

The natural uranium foil was to be treated with a pure nickel by using traditional electroplating techniques. While this foil was not intended to be removed (declad) from the capsule, the plating offered a method for elimination of one gas gap. This simplified the calculations and allowed for better cooling of the foil through the single gas gap and out to the primary coolant.

Figure 3.59 is a drawing of the capsule as fabricated, and Figure 3.60 is a photograph of the capsule.

FIGURE 3.59 Nickel “Rabbit” Capsule Drawing

FIGURE 3.60 Nickel Capsule with LEU Foil (surrogate), Internal Nickel Spring, and Nickel End Cap

4 REACTOR QUALIFICATION OF PRODUCTION-STYLE ANNULAR TARGETS

The LEU foils used in this project include the following:

- 2 in.² (2 in. × 1 in.) – HFIR nickel capsule containing NU foil
- 3 in.² (1 in. × 3 in.) – MURR annular LEU target
- 21 in.² (3 in. × 7 in.) – HFIR annular LEU target

4.1 GENERAL REACTOR CONCERNS WITH LEU METAL ANNULAR TARGETS

Every research reactor has different requirements for operation and for target configuration. Some of these requirements can drive target design to be different for each facility. Finding a common design that works for all reactors may be feasible but was beyond the scope of this project. The two reactors participating in this project had unique enough requirements and characteristics that they could not accept identical targets; however, the targets prepared for each reactor have many common attributes.

The following highlights some of the concerns and limitations for the LEU metal targets in the reactors utilized for this project.

4.1.1 Limit for Reactor Fission Product Release

One constraint that limits the size of an LEU foil that can be irradiated in a reactor is the amount of fission products that will be released in the case of failure of a target cladding. A common conservative assumption is that 100% of the fission products generated will be released into containment. However, it is unlikely that all of the gases generated will be released, rather, they will be contained within the LEU foil itself. Testing performed at the University of Missouri and described in this document shows release rates lower than 1% for LEU metals under typical Mo-99 production irradiation.

Additionally, each reactor has unique systems for handling such events. For example, in the case of the Missouri reactor, where the targets are irradiated in an open pool, there is risk of releasing fission gases into the reactor bay. Despite systems that may be in place for handling such releases, personnel hazards alone would require strict limits as to the allowable quantity of such a potential release. The HFIR, for example, is completely contained in a pressure vessel, so breaches could more easily be handled by the existing primary coolant cleanup systems. While HFIR may physically be capable of handling such a release, a target failure would be viewed unfavorably by DOE; hence, careful planning and attention will ensure target integrity and quality to minimize the possibility of such a release.

4.1.2 Target Material Temperature Limits and Unique Behavior of Uranium Metal

The temperature of target components needs to be kept below specified thresholds as determined by the reactor. For most components, this corresponds to the melting temperature of the material. For HFIR, the melting point of the U foil does not dictate the maximum allowable temperature. A lower limit has been established based on earlier work showing uranium metal swelling reactions to increased temperature and neutron irradiation (Figure 4.1). The HFIR bounding temperature has been determined to be 375°C, which corresponds to a region where uranium has been shown to exhibit high growth [LEGGETT-1963]. This is to ensure that the temperature limit will still be sufficient to avoid this swelling phenomenon. At this time, there is no experimental technique that is capable of measuring the internal temperature of components like the LEU foil. Therefore, modeling must be used to estimate the target behavior during irradiation conditions. The computer simulation that was used is summarized later in this document (Section 5).

FIGURE 4.1 High-Purity Uranium Swelling at Various Temperatures [LEGGETT-1963]

4.1.3 Target Overpressure (Internal Pressure) Conditions

In rare cases, impurities will be sealed into a capsule, and when irradiation begins or when the capsule is being shipped in an evacuated cask and the temperature rises, the impurities

could come to a boil, causing a buildup of internal pressure. This pressure on an annular target can cause deformation of the capsule and subsequent flow-blockage of the capsule-coolant gap in the reactor, as well as potentially result in capsule breach and release of fission gases and/or particulates from the base uranium material. The preferred failure mode of a breach is to fail at the weld without significant bulging of the cladding walls.

To identify the performance of this newly developed capsule design, destructive tests are necessary to provide mechanical boundary conditions for capsule containment. This report documents the destructive test results for various failure limits of the housing material.

The internal compression test loop (ICTL) was developed to hydrostatically load test capsules to various internal pressures to set bounding pressures for capsule failure, plastic deformation, operating pressure, etc. This loop includes a manually operated pump, pressure relief valve, calibrated gauge, bleeder valve, and required plumbing. See the piping schematic in Figure 4.2 for more detail.

The ICTL requires an outside water source to operate. Once a capsule is properly installed and the water source is established, the loop must be purged of all air. This purging is accomplished by opening the bleeder valve and running water through the loop until air is no longer ejected from the valve outlet. Once this task is complete, the bleeder valve should be closed, and the loop is ready for operation.

FIGURE 4.2 Schematic of Internal Compression Test Loop Piping

The ICTL was designed to accept various test section geometries. A cast iron vice was modified to receive the test geometries and mount plumbing. For the annular targets, a modified swage joint was made at one end of the capsule and was fitted to the ICTL. The opposite end of the fitting mated with the outer shell of the annular target and sealed to the target surface with an O-ring. See Figure 4.3 for the initial ICTL configuration. Figure 4.4 shows the modified Swagelok fitting that interfaces with the ICTL and the target.

FIGURE 4.3 Test Capsule Fitted to the ICTL

FIGURE 4.4 Swagelok Fitting Connection with ICTL

Once the test capsule was successfully coupled to the ICTL and the system was leak tight, hydrostatic pressure was slowly applied to the internal volume of the capsule by pulling down on the pump lever. Pressure was slowly added until the capsule was forced to fail. The inner diameter of capsule 1 was constrained to test the annular welds. This capsule containment was taken to failure at a pressure of 1800 psig. Figure 4.5 shows the weld failure of capsule 1. For target capsule 2, the failure location occurred on the inner wall of the annular target. The failure pressure for capsule 2 was 800 psig. It should be noted the term “failure” for this case

does not imply the containment was breached, but describes where the containment yielded due to pressure. See Figure 4.6 for the failure location of the capsule 2. The summary of this test's results can be found in Table 4.1.

FIGURE 4.5 Test Capsule 1 Showing Failure Location

FIGURE 4.6 Test Capsule 2 Showing Failure Location

TABLE 4.1 Summary of Capsule Failure Results

• Test Capsule 1 (constrained inner diameter)	Failure pressure: 1800 psig	Failure at annular weld
• Test Capsule 2 (left unconstrained)	Failure pressure: 800 psig	Failure at inner wall

Destructive tests were performed to quantify the containment failure pressure for the ^{99}Mo annular target capsules. The minimum reported failure pressure for a collapsed target is 800 psig. This pressure was observed for the unconstrained target, and the failure mode was a bulged inner wall. A second (constrained inner wall) target was taken to 1800 psig, where it failed in the weld.

4.2 IRRADIATION MODELING—THERMAL PERFORMANCE, NEUTRONICS, BURNUP, AND FP INVENTORIES

As a pre-requisite for irradiation, an understanding of how the irradiation capsule and its contents perform is required. This includes a set of computer calculations that starts with a neutron-photon-electron transport simulation using Monte Carlo theory.

Separately, a structural analysis is performed to simulate the forming process during target assembly. This provides a pre-stressed condition of the target before the irradiation begins. The results of the neutronics and structural analysis feed steady-state thermal-structural-hydraulic simulations (using finite element methods) to understand heat transfer of the irradiation target interacting with the primary coolant of the reactor. Transient thermal-hydraulic calculations, typically performed with 1-D software such as RELAP, are used to understand the target's performance during reactor accident conditions. All three of these types of analyses are used to simulate nominal conditions of the irradiation as well as off-normal conditions postulated by the reactor's Safety Analysis Report (SAR). Figure 4.7 schematically defines these relationships.

All analyses include definition of the target and holder in the reactor core. Figure 4.8 was used as a reference for locating and holding the target in the irradiation positions of the large vertical experimental facility of HFIR.

FIGURE 4.7 Analysis Relationships

FIGURE 4.8 Vertical Experiment Facility (VXF) Target Holders for Annular Target in HFIR

4.2.1 Neutronics Analysis of Annular Targets Irradiated in the HFIR Reflector

To determine the heat deposition into and from the target as well as estimate Mo-99 production and other fission products for shipping purposes, neutronics and depletion code calculations were required.

4.2.1.1 Model Setup and Assumptions

For HFIR, neutronics calculations were performed using the Monte Carlo N-Particle (MCNP) code with the purpose of supporting the irradiation of an LEU foil contained within an aluminum annular target assembly in one of the HFIR's VXF. Depletion analysis was conducted using the SCALE code with COUPLE and ORIGEN-S modules. Additionally, VESTA Monte-Carlo based depletion software was used as a comparison of the depletion calculated using the COUPLE/ORIGEN modules.

Key assumptions for the MCNP calculations were:

- The fresh (pre-irradiated) LEU-foil isotopic composition is 0.00200 wt% U-233, 0.06675 wt% U-234, 7.50000 wt% U-235, 0.03450 wt% U-236, and 92.39675 wt% U-238.
- The LEU-foil density is 19.10 g/cm³.
- The fission products deposit their energy locally.
- For the aluminum components, the beta particles deposit their energy locally.

To support the safety basis evaluation of these targets, neutronics calculations were performed to determine:

- Nuclear heat-generation rates in the LEU foil and assembly components,
- The LEU foil's fission gas inventory,
- Transient decay heat for the LEU foil and assembly components,
- Post-discharge radionuclide inventories in the LEU foil and the target tubes, and
- The LEU foil's accumulated fission density and burn-up.

4.2.1.2 Results

Neutronics calculations were performed to support the irradiation of a LEU foil contained within an annular aluminum target tube assembly in a HFIR VXF position for a single cycle (26 days). The MCNP transport code was used for neutron, photon, coupled neutron-photon, and electron transport calculations to estimate energy-dependent neutron fluxes and heat generation rates. The SCALE code, including the COUPLE and ORIGEN-S modules, was used to perform depletion/activation, decay, and source term calculations.

The target was to be loaded in large vertical experiment number 16 (Figure 4.9), and the uranium foil analyzed in this set of calculations was 7.500 wt. % $^{235}\text{U}/\text{U}$, 7.454 cm in width, 8.992 cm in length, 125 microns in thickness, 0.838 cm^3 in volume, and 16.001 g in mass. The neutronics calculation was performed by breaking the foil into six sections with a 0.5 cm gap facing away from the core (Figure 4.10).

FIGURE 4.9 Cross Section of MCNP Model at Core Horizontal Midplane

FIGURE 4.10 LEU Foil Sector Description (sector 1 facing core)

The calculated fission and burn-up over time are shown in Table 4.2. The foil's maximum local and volume-averaged heat generation rates were determined to be 825 and 729 W/g-U, respectively. The maximum total power, accumulated fission density, and burn-up were calculated to be 11.67 kW, 8.92×10^{20} fissions/cm³, and 0.01845 fissions per initial heavy metal atom, respectively.

TABLE 4.2 Fission and Burn-up Results as a Function of Time into the Cycle

Day into Cycle	FR ^(a) (fissions/s)	AF ^(b) (fissions)	AFD ^(c) (fissions/cm ³)	U-235 (grams)	U-235(t) / U-235(t=0)	AF/IHM ^(d)
0	3.689E+14	0.000E+00	0.000E+00	1.200	1.000	0.000%
1	3.645E+14	3.168E+19	3.782E+19	1.186	0.988	0.078%
2	3.616E+14	6.305E+19	7.526E+19	1.171	0.976	0.156%
3	3.588E+14	9.417E+19	1.124E+20	1.157	0.964	0.232%
4	3.560E+14	1.251E+20	1.493E+20	1.143	0.953	0.309%
5	3.533E+14	1.557E+20	1.858E+20	1.129	0.941	0.384%
6	3.506E+14	1.861E+20	2.221E+20	1.115	0.929	0.459%
7	3.480E+14	2.163E+20	2.582E+20	1.101	0.918	0.534%
8	3.454E+14	2.462E+20	2.939E+20	1.088	0.907	0.608%
9	3.428E+14	2.760E+20	3.294E+20	1.074	0.895	0.681%
10	3.402E+14	3.055E+20	3.646E+20	1.061	0.884	0.754%
11	3.376E+14	3.348E+20	3.996E+20	1.048	0.873	0.826%

TABLE 4.2 (Cont.)

Day into Cycle	FR ^(a) (fissions/s)	AF ^(b) (fissions)	AFD ^(c) (fissions/cm ³)	U-235 (grams)	U-235(t) / U-235(t=0)	AF/IHM ^(d)
12	3.350E+14	3.638E+20	4.343E+20	1.034	0.862	0.898%
13	3.324E+14	3.926E+20	4.687E+20	1.021	0.851	0.969%
14	3.298E+14	4.212E+20	5.028E+20	1.009	0.841	1.040%
15	3.273E+14	4.496E+20	5.367E+20	0.996	0.830	1.110%
16	3.247E+14	4.778E+20	5.703E+20	0.983	0.819	1.179%
17	3.222E+14	5.057E+20	6.037E+20	0.971	0.809	1.248%
18	3.196E+14	5.335E+20	6.368E+20	0.958	0.798	1.317%
19	3.171E+14	5.610E+20	6.696E+20	0.946	0.788	1.384%
20	3.146E+14	5.883E+20	7.022E+20	0.934	0.778	1.452%
21	3.121E+14	6.153E+20	7.345E+20	0.922	0.768	1.519%
22	3.096E+14	6.422E+20	7.666E+20	0.910	0.758	1.585%
23	3.070E+14	6.688E+20	7.984E+20	0.898	0.748	1.651%
24	3.045E+14	6.953E+20	8.299E+20	0.886	0.738	1.716%
25	3.021E+14	7.215E+20	8.612E+20	0.874	0.729	1.781%
26	2.996E+14	7.475E+20	8.922E+20	0.863	0.719	1.845%

^(a) FR(t) = fission rate at t days into the cycle.

^(b) AF(t) = accumulated number of fissions at t days into the cycle.

^(c) AFD(t) = accumulated fission density at t days into the cycle.

^(d) AF/IHM = accumulated number of fissions at t days into the cycle per initial heavy metal atom (x100).

The annular target's decay heat (Al + LEU) and ¹³¹I activity following a 26-day irradiation cycle and a 2-day decay period were determined to be 23.1 W and 192.5 Ci, respectively. Figure 4.11 shows the decay heat vs. days after irradiation. Based on 23–26 day cycle lengths, the foil's ⁹⁹Mo content was estimated to be 308.4–317.7 Ci following 2 days of decay, 144.7–149.0 Ci following 5 days of decay, and 41.0–42.2 Ci following 10 days of decay.

The post-irradiation radionuclide inventory, similar to the post-irradiation decay heat, is dependent on the irradiation time. Thus, the four ORIGEN calculations described in the previous section were also used to estimate the post-irradiation radionuclide inventories for cycle lengths of 23, 24, 25, and 26 days. Again, the Al annular target assembly was only activated assuming a 26-day cycle length. The post-irradiation radionuclide inventories for the LEU foil following 23-, 24-, 25-, and 26-day irradiations are listed in Table 4.2, and the radionuclide inventory for the Al annular target assembly is listed in Table 4.3. The inventories are listed at several time steps following the end of the cycle out to 50 days of decay and are sorted according to their activity (Ci) at 2 days of decay. Only those nuclides whose activity is greater than 1 µCi following 2 days of decay are listed. The nuclides were sorted at day 2 because the targets are to be transported as soon as reasonably possible, which is expected to be in the 2-3 day range.

The dominant fission products in terms of curies following 2 days of decay include, but are not limited to, ^{239}Np , ^{133}Xe , ^{140}La , and ^{140}Ba . The ^{131}I activity following 26 days of irradiation and 2 days of decay is 190.2, 191.2, 192.0, and 192.5 Ci, respectively.

FIGURE 4.11 Fission Decay Heat Following Irradiation

TABLE 4.3 LEU Foil Post-26 Day Irradiation Radionuclide Inventory (Ci) as a Function of Decay Time (days) for Top 50 Isotopes

26 day	1.0	2.0	3.0	4.0	5.0	7.5	10.0	15.0	20.0	30.0	40.0	50.0
total	6.589E+03	5.086E+03	4.278E+03	3.732E+03	3.324E+03	2.627E+03	2.185E+03	1.656E+03	1.345E+03	9.817E+02	7.695E+02	6.301E+02
Np-239	6.795E+02	5.063E+02	3.773E+02	2.811E+02	2.095E+02	1.004E+02	4.811E+01	1.105E+01	2.538E+00	1.339E-01	7.065E-03	3.727E-04
Xe-133	5.233E+02	4.801E+02	4.306E+02	3.819E+02	3.368E+02	2.435E+02	1.753E+02	9.063E+01	4.682E+01	1.248E+01	3.328E+00	8.872E-01
La-140	4.007E+02	3.936E+02	3.821E+02	3.681E+02	3.527E+02	3.128E+02	2.748E+02	2.102E+02	1.602E+02	9.306E+01	5.404E+01	3.138E+01
Ba-140	3.909E+02	3.702E+02	3.506E+02	3.321E+02	3.145E+02	2.746E+02	2.397E+02	1.826E+02	1.392E+02	8.081E+01	4.693E+01	2.725E+01
Pr-143	3.668E+02	3.601E+02	3.491E+02	3.360E+02	3.218E+02	2.859E+02	2.523E+02	1.957E+02	1.516E+02	9.098E+01	5.459E+01	3.275E+01
Mo-99	3.969E+02	3.084E+02	2.396E+02	1.862E+02	1.447E+02	7.700E+01	4.098E+01	1.161E+01	3.288E+00	2.638E-01	2.116E-02	1.698E-03
Tc-99m	3.812E+02	2.982E+02	2.318E+02	1.801E+02	1.400E+02	7.450E+01	3.965E+01	1.123E+01	3.181E+00	2.552E-01	2.048E-02	1.643E-03
I-132	3.012E+02	2.426E+02	1.954E+02	1.574E+02	1.268E+02	7.374E+01	4.294E+01	1.456E+01	4.935E+00	5.672E-01	6.519E-02	7.492E-03
Te-132	2.919E+02	2.351E+02	1.894E+02	1.525E+02	1.229E+02	7.154E+01	4.165E+01	1.412E+01	4.787E+00	5.502E-01	6.324E-02	7.269E-03
Ce-141	2.167E+02	2.122E+02	2.077E+02	2.033E+02	1.990E+02	1.887E+02	1.789E+02	1.608E+02	1.446E+02	1.168E+02	9.437E+01	7.625E+01
I-131	2.090E+02	1.925E+02	1.770E+02	1.626E+02	1.493E+02	1.204E+02	9.703E+01	6.300E+01	4.090E+01	1.723E+01	7.262E+00	3.060E+00
Ce-143	2.958E+02	1.788E+02	1.081E+02	6.531E+01	3.947E+01	1.121E+01	3.184E+00	2.568E-01	2.071E-02	1.347E-04	8.761E-07	5.699E-09
Zr-95	1.409E+02	1.394E+02	1.379E+02	1.364E+02	1.350E+02	1.314E+02	1.279E+02	1.211E+02	1.147E+02	1.030E+02	9.241E+01	8.293E+01
Y-91	1.382E+02	1.370E+02	1.355E+02	1.339E+02	1.323E+02	1.285E+02	1.247E+02	1.176E+02	1.108E+02	9.842E+01	8.743E+01	7.766E+01
Nd-147	1.371E+02	1.287E+02	1.208E+02	1.134E+02	1.065E+02	9.093E+01	7.766E+01	5.664E+01	4.131E+01	2.197E+01	1.169E+01	6.216E+00
Sr-89	1.244E+02	1.227E+02	1.210E+02	1.193E+02	1.177E+02	1.138E+02	1.099E+02	1.026E+02	9.583E+01	8.354E+01	7.283E+01	6.350E+01
I-133	2.511E+02	1.128E+02	5.071E+01	2.279E+01	1.024E+01	1.387E+00	1.878E-01	3.444E-03	6.314E-05	2.123E-08	7.137E-12	2.399E-15

TABLE 4.3 (Cont.)

26 day	1.0	2.0	3.0	4.0	5.0	7.5	10.0	15.0	20.0	30.0	40.0	50.0
Ru-103	9.957E+01	9.782E+01	9.611E+01	9.443E+01	9.278E+01	8.877E+01	8.494E+01	7.776E+01	7.119E+01	5.967E+01	5.001E+01	4.192E+01
Rh-103m	9.840E+01	9.667E+01	9.498E+01	9.332E+01	9.169E+01	8.773E+01	8.394E+01	7.685E+01	7.035E+01	5.897E+01	4.942E+01	4.142E+01
Nb-97	1.959E+02	7.254E+01	2.686E+01	9.945E+00	3.682E+00	3.072E-01	2.563E-02	1.784E-04	1.242E-06	6.014E-11	2.913E-15	1.411E-19
Zr-97	1.818E+02	6.733E+01	2.493E+01	9.232E+00	3.418E+00	2.852E-01	2.379E-02	1.656E-04	1.152E-06	5.582E-11	2.704E-15	1.310E-19
Pm-149	6.295E+01	4.602E+01	3.364E+01	2.459E+01	1.797E+01	8.210E+00	3.750E+00	7.825E-01	1.633E-01	7.110E-03	3.096E-04	1.348E-05
Nb-95	3.555E+01	3.760E+01	3.957E+01	4.149E+01	4.333E+01	4.767E+01	5.162E+01	5.848E+01	6.408E+01	7.207E+01	7.663E+01	7.857E+01
Pr-144	3.013E+01	3.006E+01	2.998E+01	2.991E+01	2.984E+01	2.966E+01	2.948E+01	2.912E+01	2.877E+01	2.808E+01	2.740E+01	2.674E+01
Ce-144	3.013E+01	3.006E+01	2.998E+01	2.991E+01	2.984E+01	2.966E+01	2.948E+01	2.912E+01	2.877E+01	2.808E+01	2.740E+01	2.674E+01
Rh-105	4.578E+01	2.879E+01	1.799E+01	1.124E+01	7.021E+00	2.166E+00	6.680E-01	6.355E-02	6.046E-03	5.473E-05	4.954E-07	4.485E-09
Xe-135	1.107E+02	2.654E+01	4.984E+00	8.619E-01	1.440E-01	1.558E-03	1.652E-05	1.846E-09	2.060E-13	2.567E-21	3.199E-29	0.000E+00
y-93	1.003E+02	1.957E+01	3.819E+00	7.452E-01	1.454E-01	2.445E-03	4.113E-05	1.163E-08	3.290E-12	2.632E-19	2.105E-26	0.000E+00
Sr-91	8.284E+01	1.472E+01	2.617E+00	4.651E-01	8.266E-02	1.101E-03	1.466E-05	2.600E-09	4.612E-13	1.451E-20	4.564E-28	0.000E+00
Pm-151	1.967E+01	1.095E+01	6.096E+00	3.393E+00	1.889E+00	4.367E-01	1.010E-01	5.398E-03	2.885E-04	8.245E-07	2.356E-09	6.733E-12
Te-127	1.210E+01	1.038E+01	8.724E+00	7.305E+00	6.114E+00	3.922E+00	2.525E+00	1.064E+00	4.695E-01	1.265E-01	6.676E-02	5.408E-02
Sb-127	1.177E+01	9.830E+00	8.210E+00	6.858E+00	5.728E+00	3.652E+00	2.328E+00	9.464E-01	3.847E-01	6.357E-02	1.050E-02	1.735E-03
Sm-153	1.355E+01	9.458E+00	6.603E+00	4.609E+00	3.218E+00	1.310E+00	5.334E-01	8.842E-02	1.466E-02	4.028E-04	1.107E-05	3.042E-07
Y-91m	5.187E+01	9.220E+00	1.639E+00	2.912E-01	5.177E-02	6.894E-04	9.181E-06	1.628E-09	2.888E-13	9.086E-21	0.000E+00	0.000E+00
Te-131m	1.189E+01	6.828E+00	3.922E+00	2.252E+00	1.294E+00	3.234E-01	8.085E-02	5.053E-03	3.158E-04	1.234E-06	4.819E-09	1.882E-11
u-237	6.198E+00	5.593E+00	5.047E+00	4.554E+00	4.110E+00	3.179E+00	2.460E+00	1.472E+00	8.808E-01	3.154E-01	1.130E-01	4.045E-02
Xe-133m	5.448E+00	4.447E+00	3.455E+00	2.614E+00	1.948E+00	9.053E-01	4.134E-01	8.517E-02	1.750E-02	7.388E-04	3.118E-05	1.316E-06
I-135	4.070E+01	3.236E+00	2.572E-01	2.045E-02	1.626E-03	2.897E-06	5.162E-09	1.639E-14	5.204E-20	0.000E+00	0.000E+00	0.000E+00
Pm-148	2.823E+00	2.482E+00	2.182E+00	1.919E+00	1.687E+00	1.224E+00	8.879E-01	4.687E-01	2.486E-01	7.210E-02	2.301E-02	9.019E-03
Eu-156	2.231E+00	2.136E+00	2.042E+00	1.951E+00	1.864E+00	1.663E+00	1.484E+00	1.181E+00	9.401E-01	5.957E-01	3.774E-01	2.391E-01
Pm-147	1.890E+00	1.985E+00	2.074E+00	2.157E+00	2.235E+00	2.409E+00	2.556E+00	2.788E+00	2.953E+00	3.152E+00	3.247E+00	3.286E+00
Ru-106	1.937E+00	1.933E+00	1.930E+00	1.926E+00	1.922E+00	1.914E+00	1.905E+00	1.887E+00	1.870E+00	1.835E+00	1.802E+00	1.768E+00
Rh-106	1.937E+00	1.933E+00	1.930E+00	1.926E+00	1.922E+00	1.914E+00	1.905E+00	1.887E+00	1.870E+00	1.835E+00	1.802E+00	1.768E+00
Xe-131m	1.713E+00	1.750E+00	1.774E+00	1.787E+00	1.790E+00	1.761E+00	1.694E+00	1.497E+00	1.269E+00	8.435E-01	5.274E-01	3.180E-01
Te-131	2.676E+00	1.537E+00	8.829E-01	5.071E-01	2.912E-01	7.281E-02	1.820E-02	1.138E-03	7.110E-05	2.777E-07	1.085E-09	4.237E-12
Ag-111	1.659E+00	1.512E+00	1.377E+00	1.255E+00	1.144E+00	9.062E-01	7.182E-01	4.510E-01	2.832E-01	1.117E-01	4.406E-02	1.738E-02
Nb-95m	1.326E+00	1.359E+00	1.383E+00	1.400E+00	1.411E+00	1.421E+00	1.412E+00	1.367E+00	1.306E+00	1.177E+00	1.057E+00	9.491E-01
Pr-145	1.985E+01	1.231E+00	7.638E-02	4.739E-03	2.940E-04	2.818E-07	2.702E-10	2.483E-16	2.282E-22	0.000E+00	0.000E+00	0.000E+00
Sn-125	1.215E+00	1.131E+00	1.052E+00	9.793E-01	9.114E-01	7.614E-01	6.361E-01	4.440E-01	3.099E-01	1.510E-01	7.357E-02	3.585E-02
Cs-137	9.146E-01	9.146E-01	9.145E-01	9.144E-01	9.144E-01	9.142E-01	9.141E-01	9.138E-01	9.135E-01	9.129E-01	9.124E-01	9.118E-01

4.2.2 Structural/Thermal Calculations Supporting HFIR Irradiations

Structural and thermal calculations were performed using input from the neutronics heat generation results. These calculations were developed to address a specific target geometry based on the inspected dimensions of individual target components, and not based on a generic target design. Completion of this calculation does not guarantee that a target fabricated to the drawing dimensions will be approved for irradiation. A separate set of repeat calculations may be required to qualify each unique target until further testing of irradiated foils confirms the assumptions regarding swelling of the uranium foil. (Detailed information about this calculation can be found in Appendix C.)

4.2.2.1 Method

Due to the difficulty associated with physically measuring certain dimensions of the annular target assembly, we decided to simulate the assembly process by means of an axisymmetric stress analysis, using the commercial finite element analysis (FEA) code ABAQUS FEA. The dimensions of the annular target obtained from the assembly were used to build an irradiation model by including the residual stresses developed during assembly as an initial input. The purpose of the irradiation model is to simulate in-vessel irradiation by accounting for the volumetric heat generation in the LEU foil, gamma heating in the cladding, and fission gas release. The melting point of the cladding was taken to be the thermal safety limit while the von Mises yield criteria for ductile materials as applied to the cladding were used as the structural safety limit.

4.2.2.2 Results

The safety-basis calculations indicate that the annular target temperature will remain below the cladding melting temperature of 923 K for all power levels between 11 and 17 kW. This range includes the 130% power level at 14.3 kW.

The structural results indicate that the von Mises stresses in the cladding are lower than the yield strength of the Al 6061-T4 cladding for all power levels between 11 and 17 kW for the inner (Figure 4.12) and outer (Figure 4.13) cladding. Figure 4.14 shows the temperature profile of the target at three locations.

FIGURE 4.12 Von Mises Stresses in the Inner Tube Outer Surface

FIGURE 4.13 Von Mises Stresses in the Outer Tube Inner Surface

FIGURE 4.14 Temperature Distribution Contour of the Annular Target at 17 kW Power

4.2.2.3 Conclusions

The commercial FEA code ABAQUS was used to develop simulations to assess the thermal and structural integrity of an LEU-foil-based annular target and support its safe irradiation at HFIR. A model to simulate the assembly process of the annular target was first developed. The output of this simulation provided the dimensions of the assembled annular target and the residual stresses. These were used as inputs to build an irradiation model to simulate the in-vessel irradiation of the annular target. An interfacial conductance model that accounts for the conduction through the solid spots and gas gaps was developed. A parametric study was performed on the target power between 11 kW and 17 kW. This range included the 130% power level required for the safety calculations. The results from the analysis indicate that the maximum temperature in the cladding is within its melting temperature of 923 K, and the von Mises stress in the cladding is within its yield strength of 145 MPa, thus satisfying the thermal and structural safety criteria.

4.2.2.4 Irradiation Modeling—Stress/Thermal Analysis

The goal of the irradiation modeling is to determine the thermal/mechanical state of the target, in particular, the state of the interface (e.g., the interfacial pressure) between the LEU foil and the aluminum-tube cladding. The target-consolidation processes described above needed to be evaluated during a preliminary mechanical step in order to determine the initial state of the target before irradiation. This section explores those models, beginning with an analysis that evaluates a baseline case when no residual stresses are considered to elucidate what the natural tendencies of the target are during irradiation.

4.2.2.4.1 Irradiation Analysis—Without the Assembly Residual Stresses

Initial analysis [GOVINDARAJAN-2012] of the annular target involved development of a 2D uniform-heating numerical model in the commercial code ABAQUS. No gap between the edges of the LEU foil is assumed, as illustrated in Figure 4.15. To validate this model, an analytical model of a triple-layer annular cylinder was constructed.

FIGURE 4.15 Uniform and Nonuniform Heating Models for the Initial Analysis without Residual Stresses

The validated uniform-heating model was then used to validate the nonuniform heating model (annular target with the recess) by using an order-of-magnitude comparison approach. Based on the analysis of the nonuniform heating model, we found that the inner tube was at a greater risk of failing first (if the target were to fail) because the magnitude of hoop stresses in the inner tube is greater than that on the outer tube. The uniform heating model was able to match the inner-tube hoop stresses in the nonuniform heating model to within 20%. Parametric studies were performed with the uniform heating analytical model to assess the sensitivity of the target to varying LEU heat generation rates, heat transfer coefficient ratios, and tube thickness ratios. The analysis results indicate that the temperatures and stresses are unlikely to exceed the melting point and yield strength of the Al 6061-T6 cladding. It was also observed that the magnitude of the interfacial separation can be controlled by adjusting the heat transfer coefficient split between the inner and outer tube surfaces.

4.2.2.4.2 Integrating the Assembly and Irradiation Modeling—Inclusion of Residual Stresses

The initial annular-target analysis provided an insight into the behavior of the target under various operating conditions, without the effect of the assembly residual stresses. However, the analysis of the target assembly is important before performing an irradiation analysis as it provides a way to include the residual stresses due to assembly. Hence, the second part of the annular-target analysis focused on integrating the assembly and the irradiation model.

This integration was done by using a three-step modeling approach, where the first step simulates the hydroforming or the draw-plug assembly process, the second step serves as the pressure relaxation step in the hydroforming process and the plug removal step in the draw-plug process, and the third step simulates the irradiation of the annular target. As the analysis proceeds in a step-wise fashion, the output from an analysis step is automatically used as an initial input in the subsequent step. In doing so, the assembly residual stresses are automatically applied as initial inputs to the irradiation model. The integrated assembly and irradiation modeling approach is illustrated in Figure 4.16.

FIGURE 4.16 The Integrated Assembly and Irradiation Model

In the planar hydroforming analysis, the internal pressure was found to be 36.40 MPa [GOVINDARAJAN-2014A, -2016], and in the axisymmetric draw-plug analysis, a velocity of 0.16 m/s was found to be adequate to move the plug through the inner tube without causing any wrinkling [GOVINDARAJAN-2015, -2016]. The draw-plug required to assemble the Al 6061-T6 tubes, categorized based on the largest diameter of the plug, was found to be 26.62 mm (1.048 in.) [GOVINDARAJAN-2015]. The thermal and mechanical constraints are as provided in Figure 4.16

The results from the hydroforming and the draw-plug analysis procedures indicate that the residual stresses from the assembly process tend to negate and decrease the stresses at the end of the irradiation step, as illustrated in Figure 4.17. The temperature variation across the thickness of the assembly at half the target length has been compared for the two modeling approaches in Figure 4.18. In both cases, the maximum temperature is lesser than the melting temperature of the Al 6061-T6 (855 K).

FIGURE 4.17 Hoop Stresses at Half the Target Length after Assembly and Irradiation Modeling

FIGURE 4.18 Radial Temperature Distribution in the Annular Target

4.2.2.4.3 Integrated Draw-Plug and Irradiation Model—Effects Study

The effects of fission-gas release, uranium swelling, thermal contact, heat generation in the cladding, and the pressure exerted by the coolant flow were studied for the draw-plug based irradiation model [GOVINDARAJAN-2013, -2014A, -2014B, -2015]. The goal was to assess the effects of each of these parameters on the temperatures and stresses in the cladding and establish whether these values are within the prescribed thermal and structural safety limits. In addition to the loads and boundary conditions in Figure 4.16 for the axisymmetric draw-plug model, the constraints presented in Figure 4.19 were added to the model for the effects study. Uranium swelling was modeled by providing a volumetric swelling strain rate of $2.48 \times 10^{-8} \text{ s}^{-1}$. A detailed description of the calculation procedure used to determine each of the parameters used in the effects study can be found in [GOVINDARAJAN-2015].

FIGURE 4.19 Additional Constraints Applied to the Axisymmetric Integrated Draw-Plug Model for the Effects Study

The analysis results indicate very small changes in temperatures and von Mises stresses for each of the cases considered in the analysis. The parameters that provide the worst-case scenarios for the temperatures and von Mises stresses were found to be different. The combination of thermal contact conductance, uranium swelling, fission-gas pressure, and the cladding heat generation represents the worst-case scenario for the temperatures, while this combination along with the pressure exerted by the coolant represents the worst-case scenario for the von Mises stresses. The swelling of the uranium was found to have no significant impact on the temperatures and von Mises stresses, while the fission-gas pressure based on fractional release was found to cause only very small changes on the temperatures and the stresses. The radial temperature distribution across the assembly thickness is illustrated in Figure 4.20.

FIGURE 4.20 Radial Temperature Distribution in the Annular Target at Half the Target Length Illustrating the Interfacial Temperature Drop

A parametric study on the target heat-generation rate showed that the maximum temperature and von Mises stress (Figure 4.21), even at the highest heat generation rate (four times the baseline heat-generation rate), are within the melting temperature and yield strength of the Al 6061-T6 cladding. Therefore, it can be concluded that the annular target is thermally and structurally safe and is unlikely to cause a safety concern during irradiation.

FIGURE 4.21 Variation of Maximum Temperature and Maximum von Mises Stress with the Heat Generation Rate. (Note: The yield point of the Al 6061-T6 is based on the plasticity model used in the analysis.)

5 REACTOR QUALIFICATION OF SMALL TEST CAPSULES FOR IRRADIATION IN HFIR

To demonstrate the acid frontend process (Section 9) in a hot cell at ORNL, a uranium target needed to be irradiated. Due to the size of production-style annular targets and the high flux of the HFIR, a reduced enrichment capsule that could be irradiated in a hydraulic tube system had to be developed. This new capsule (Figure 5.1) allowed irradiation to occur in the HFIR hydraulic tube, which can be ejected at any time during a HFIR cycle and shipped to the hot cell soon after ejection. The nickel content allowed for insertion into the target dissolution vessel for dissolving the entire capsule, eliminating the need to open the capsule and retrieve the NU foil. This would provide between 25 and 100 Ci of Mo-99 materials for the test.

FIGURE 5.1 CAD Model Cutaway of Nickel Capsule with Natural Uranium Foil and Internal Pressure Spring

5.1 DESIGN OVERVIEW OF THE NICKEL–NATURAL URANIUM TEST CAPSULES

The capsule assembly containing the Ni housing and components is described in Section 3.8.2.

5.2 IRRADIATION MODELING—NEUTRONICS AND THERMAL PERFORMANCE

5.2.1 Neutronics Calculation Supporting HFIR Irradiation of Nickel Capsule Containing Natural Uranium Foil in the Hydraulic Tube

The purpose of this set of calculations was to support the irradiation of a high-density NU capsule in the HFIR hydraulic tube (HT) facility. To support the safety basis evaluation of these targets, neutronics calculations were performed to determine:

- the nuclear heat generation rates for the NU foil and associated nickel-capsule housing components,
- the decay heat for the NU foil and associated nickel-capsule housing components,
- the compiled transient heat generation rates of the NU foil and associated nickel-capsule housing components,
- post-discharge radionuclide inventories of the NU foil and associated nickel-capsule housing components,
- the produced fission gases of the NU foil, and
- the potential impact of a HT capsule stack insertion/ejection in terms of its reactivity.

The following assumptions were made in this set of calculations.

- The reactor will operate for a 26-day cycle, and the capsule will remain for that full period of time.
- The naturally enriched U foil is a perfectly homogeneous solid composed of similar isotopics and impurities measured by the Y-12 National Security Complex. Specifically, a limit of 0.7142 wt% U-235 and 0.002 wt% U-233 is used.
- The beta particles in the nickel capsule components and fission fragments in the NU foil deposit their energy locally.
- The variance for the total heating can be estimated by using a propagation of error formula.

5.2.1.1 Design of NU Irradiation Capsule

The ID of the Ni housing is nominally 0.374 in. (0.94996 cm) with a nominal closed end thickness of 0.07 in. (0.18 cm). The end cap is welded within the housing and has a nominal length of 0.125 in. (3.18 cm). The maximum length of the NU irradiation capsule is 2.625 in. (6.68 cm), which is used for this calculation. The NU irradiation capsule as modeled in MCNP can be seen in Figure 5.2. The NU irradiation capsule parts list is shown in Table 5.1.

TABLE 5.1 Irradiation Capsule Parts List

p/n	Description	Material
DD-M802243-0003-5	inner sleeve	4N5 Nickel
DD-M802243-0002-3/4	nickel	Y-12*
DD-M802243-0002-2	U foil	Y-12**
DD-M802243-0002-2	end cap	4N5 Nickel
DD-M802243-0002-1	housing	4N5 Nickel

* Provided by Y-12; hardened nickel 4N5 material is used.

** Provided by Y-12; uranium material properties as detailed in Section 4.3.2.1 are used.

The NU foil is fabricated at the Y-12 National Security Complex using available natural uranium feedstock. Two sample measurements taken at Y-12 of natural uranium isotopics and impurities are shown in Appendices D-1 and -2. Per Justification 3, the impurities of the natural uranium are distributed to the ^{235}U mass, and the maximum measured weight percentages of ^{235}U and ^{233}U are used to maximize fissionable isotopes. The remaining nominal non- ^{238}U mass is distributed to ^{234}U , and the remainder is distributed to the fertile isotope ^{238}U . A high uranium density of 19.1 g/cm^3 was used [JONES-2002]. The as-modeled NU foil atomic composition (in atoms/barn-cm) at the beginning of the cycle (BOC) is shown below:

```
c  NU foil, 0.7142 wt.% U-235/U, 19.10 g/cc (4.83231E-02 at/b-cm)
m78      92233.70c  9.871532E-07   92234.70c  1.474394E-06
          92235.70c  3.495064E-04   92238.70c  4.797113E-02
```

The hardened nickel 4N5 impurities were measured at Goodfellow Corp. and are shown in Appendix D-3. Since there are no Ta-180 cross sections in the ENDF/B-VII neutron cross-section library and its abundance is so small (0.00012 at.% $^{180}\text{Ta}/\text{Ta}$), the Ta impurity was modeled as 100% ^{181}Ta . Osmium was removed from the initial composition and distributed to the neighboring element Re because there are no Os isotope cross sections in the ENDF/B-VII

neutron cross-section library. However, the impurity is present at a quantity of only 0.215 ppm and, therefore, has no impact on the results.

5.2.1.2 MCNP Input Modifications to HFIR Model

The MCNP inputs for the BOC and end of the cycle (EOC) were modified to make small corrections in the HFIR flux trap region and add the eight-capsule stack to the hydraulic tube (six HFIR dummy capsules, one capsule pusher, and one LEU irradiation capsule) for each case. The BOC base input used was the HFIR Cycle 400 model [PRIMM-2005], which is used as the basis for most HFIR-related calculations requiring neutron and photon transport. The EOC input is based on the generated VESTA depletion calculation [ILAS-2011] result for the revised model for HFIR HEU cycle 400.

5.2.1.3 Geometry

The capsule stack was modeled in flux trap position B-3 (hydraulic tube). The nominal tube ID and OD were modified from their as-modeled cycle 400 values to reflect the current drawings at 0.56 in. (1.42 cm) and 0.657 in. (1.67 cm), respectively. The capsule stop (or bottom of the capsule stack) with respect to the reactor centerline is located at an elevation of 11.596 in. (29.45 cm) below the reactor centerline.

The HFIR dummy capsule has a minimum capsule height of 2.53 in. (6.43 cm). The HFIR capsule pusher capsules are placed at the top and bottom (positions 9 and 1, respectively, in the stack of nine capsules) of capsule stacks as large OD capsules that help push/pull the capsule stack into/out of the hydraulic tube. In this case, since the irradiation capsule is heavy compared to dummy capsules, only a bottom capsule pusher is needed, whose nominal height and diameter are 2.50 in. (6.35 cm) and 0.469 in. (1.19 cm).

The design geometry for the NU foil irradiation capsule is described in Section 4.2. The diameters of the NU foil, nickel coating, and inner sleeve are determined relative to the ID of the nickel housing. The as-modeled thickness of the inner sleeve is slightly greater than the actual thickness to conserve the total mass of the nickel sleeve since it is modeled as a hollow cylinder. The NU foil was modeled with a 342 degree sector angle. The 17.61 degree gap is directed away from the core centerline, since the thermal flux is greatest in the center of the core. A foil volume and mass of 0.178 cm³ and 3.39 g, respectively, are modeled.

FIGURE 5.2 As-modeled X-Y Cross Section of HT and NU Irradiation Capsule at Core Horizontal Midplane

For the nominal case considered, the capsule pusher is placed at position 1, HFIR dummy capsules at positions 2 through 7, and the U foil irradiation capsule placed at position 8. For the loading error case, the capsule pusher is placed at position 1, HFIR dummy capsules at positions 2 through 4 and 6 through 8, and the NU foil irradiation capsule at position 5. In the position 5 case, the height of the capsule pusher is increased to 2.93 in. (7.45 cm) in order to place the LEU foil at the reactor core horizontal midplane (the location of highest flux and thus highest heat generation). The capsule stack in the HT for the position 5 case is shown in Figure 5.3 as modeled in MCNP.

FIGURE 5.3 Nickel Capsule Axial Location in HFIR Core

5.2.1.4 Activation, Depletion, and Decay Calculations using SCALE

The SCALE code (including the COUPLE, ORIGEN-S, and OPUS modules) was used to perform activation, depletion, and decay calculations on the NU foil material and nickel assembly component material, including the housing capsule, the inner sleeve, the foil's nickel coating, and the end cap. The COUPLE code, utilizing the BOC 238-group neutron flux spectrum as cross-section weighting data, was used to collapse the 238-group AMPX neutron activation cross sections into one-group format as required by ORIGEN-S, unless user-supplied cross sections were made available. A Python script was generated and utilized to execute a Fortran program to extract the fluxes and actinide cross sections from the MCNP output, write an ORIGEN-S calculation, and execute it. The module COUPLE provided actinide fission and radiative capture cross sections calculated in the BOC MCNP input, as described later, to account for self-shielding effects in the NU foil material cross sections. Since self-shielding effects are not significant for the activation calculations of the nickel components, the infinite dilute cross sections are utilized. Then, the ORIGEN-S code was used to perform the activation and depletion calculations, and OPUS was used to post-process the results into easy-to-read tables.

For the ORIGEN-S calculations, one 26-day fuel cycle was modeled, as was a 10-day irradiation for shipping calculations. Both cases utilized a constant total neutron flux to activate the foil and assembly components. Transport calculations were performed on day 0 (BOC) to calculate the spatially dependent neutron fluxes (among other parameters wanting to be studied, but not documented here). The BOC was chosen as it is representative of the neutron flux in the target region over the course of the cycle. The day-26 fluxes were calculated in the EOC transport calculations, and the thermal neutron fluxes were calculated to be within ± 1 % of the BOC while the fast neutron fluxes decreased from their BOC values.

ORIGEN calculations for a 10-day irradiation were performed on the NU foil and nickel component materials to calculate their post-irradiation radionuclide inventories and decay heats for transportation and post-irradiation experiment (PIE) requirements. Also, the ORIGEN calculations were used to determine the EOC-delayed photon sources (photons/second) resolved in a 57-energy group structure for each of the NU foil and nickel component materials. Based on these results, fixed-source MCNP photon-type calculations were constructed to determine the photon heating rates due to delayed photons. The heat generation rates of the NU foil due to delayed photons from the core and NU foil can reach up to 47.6 and 2.1 W/g, respectively, whereas the delayed photons of the nickel components contribute considerably less (below 0.01 W/g).

5.2.1.4.1 Neutron Flux Calculations

The neutron fluxes resolved in a 238-energy group structure were calculated for the irradiation capsule materials with track length estimate of flux tallies (F4:N) in MCNP. The fluxes were resolved into 238-group structures because, in this format, they can be used to define a source weighting spectrum for the SCALE activation calculations, which was described in more detail in a previous section. The MCNP tallies were normalized per starting particle (i.e., per source fission neutron), and therefore, the tally results must be multiplied by the fission neutron source strength. The fission neutron source strength is defined by

$$S_n \left(\frac{\text{fission neutrons}}{\text{second}} \right) = \frac{P(W) \times \bar{\nu}(n/\text{fission})}{e(J/\text{MeV}) \times Q(\text{MeV}/\text{fission}) \times k_{\text{eff}}} \quad (\text{Eq. 5.1})$$

where P is the reactor power in watts (85×10^6), $\bar{\nu}$ is the average number of neutrons produced per fission reaction, e is a unit conversion factor (1.60218×10^{-13} J/MeV), Q is the average recoverable energy released per fission reaction, and k_{eff} is the effective multiplication factor. For these calculations, we used a fixed average recoverable energy of 200.7 MeV/fission, which is slightly less than the EOC Q -value of 201.3 MeV/fission and slightly greater than the BOC Q -value of 200.2 MeV/fission that was calculated in [PETERSON-2012]. The average number of neutrons produced per fission is given in the MCNP output and typically varies between 2.439 and 2.441 for HFIR calculations. The MCNP also outputs the multiplication factor, which should be close to unity for these calculations. Thus, a total fission neutron source of approximately 6.4 to 6.5×10^{18} fission neutrons per second is used for the 85 MW calculations.

5.2.1.4.2 Neutron and Photon Heating

The most conservative heat generation rates in the NU foil occur at the EOC; however, the beginning of the cycle heating rates was also calculated and included. Heating due to fission dominates other heating mechanisms in the NU foil, and the greatest amounts of fissile material or energy release (primarily due to ^{239}Pu , ^{235}U , and ^{241}Pu) are present in the foil at the EOC in comparison to any other point in time due to the buildup of ^{239}Pu from the fertile isotope ^{238}U through neutron absorption and subsequent β decays. Thus, the greatest fission rates and heat generation rates at the NU foil occur at EOC. Also, at the EOC, the delayed photon sources from the HFIR fuel and NU foil are greatest due to the buildup of fission products, resulting in the peak delayed photon source heating at the EOC.

Track length estimates of energy deposition tallies (type 6) were used to calculate heating due to neutrons and fission products (F6:N) and prompt and capture photons (F6:P). The units corresponding to the F6 tally are MeV/g-fission neutron and the desired units are W/g, so the tally results are multiplied by the total fission neutron source (fission neutrons/second) and the unit conversion factor (1.60218×10^{-13} W-s/MeV).

5.2.1.4.3 Delayed Photon Heating

Delayed photons are not included in the MCNP photon-production cross sections, so the delayed photon sources were determined from ORIGEN calculations as previously described. Then, fixed-source MCNP calculations in the photon mode were run with the BOC and EOC inputs and the photon sources previously described. The delayed photon source heating at the BOC is conservatively determined using the EOC delayed photon source, where the delayed photon source is largest. The photon heating tallies previously described were used to calculate the photon heating rates due to the delayed photons. Multipliers equal to the product of the total source strength (photons/second) and 1.60218×10^{-13} W-s/MeV were used to convert the photon heating results in terms of MeV/g-source photons to units of W/g.

5.2.1.4.4 Beta Plus Alpha Heating

Beta plus alpha heating in the NU foil material can be calculated by two methods. The ORIGEN-S calculations described previously can be used to determine the total heat (watts) due to photons, beta particles, and alpha particles, as well as the heat due to photons only. Assuming the betas and alphas are locally deposited, the heat due to betas and alphas can be calculated by subtracting the heat due to photons only from the total heat. Heating due to beta and alpha radiation is, however, dominated by the beta radiation.

5.2.1.4.5 Actinide Capture and Fission Cross Sections

The radiative and reaction rates for 30 important actinides were calculated in the NU foil, in addition to the total neutron flux, in MCNP, and the results used in the SCALE calculations as previously described.

The one-group cross sections (barns) were calculated for each actinide, as defined by

$$\bar{\sigma}_x = \frac{\int \sigma_x(E) \varphi(E) dE}{\int \varphi(E) dE} \quad (\text{Eq. 5.2})$$

where $\bar{\sigma}$ is the one-group actinide cross section in barns, σ_x is the energy-dependent microscopic cross section for reaction type x in barns, E is energy, and φ is the energy-dependent neutron flux in neutrons/cm²-sec per fission neutron. The total neutron source is applied to both the denominator and numerator and thus cancels itself out. The resulting actinide cross sections are then used in the NU foil ORIGEN depletion calculations previously described.

5.2.1.4.6 Transient Heat Generation

Three components contribute to the total transient decay heating of the irradiation capsule in this analysis: prompt particle heating, reactor-delayed photon heating, and target decay heating. The first is due to “prompt” effects, i.e., the capsule heating due to reactions from prompt neutrons and photons in the reactor (primarily neutron-induced fission in the NU foil). This includes all contributions from prompt neutrons and photons produced in the reactor, including those from the irradiation capsule itself. The initial prompt heat generation after reactor scram is determined from MCNP calculations, as previously described. This effect is the primary contributor over short time periods (less than 1 second) due to its large value at time zero after reactor shutdown. However, its time-dependent power curve, developed by the HFIR point-kinetics reactivity model, falls off rapidly due to the rapid insertion of the safety plates.

The second heating component is “core decay” due to the delayed photons emitted by the reactor fuel and structural materials after shutdown from fission and activation products.

The third heating component is the local decay components from the irradiation capsule itself. The radioactive decay of the capsule produces beta (electron or positron), alpha (⁴He), and gamma (photon) particles due to its fission and activation products. All radiation released is conservatively assumed to deposit its energy locally (in the decay material). However, the fixed source calculations transport these delayed photons, and most of the energy is deposited elsewhere in the system. To be conservative, delayed photon heating between the capsule materials (the NU foil and the nickel components) is counted twice according to the delayed-photon EOC fixed-source calculations. This additional heating contribution decays according to a conservative decay power fraction, which bounds the local decay fraction of the contributing source (the NU foil or nickel components).

The total steady-state heat generation rate is used as the initial total heat generation rate at reactor scram. The contributions from the core decay photons and local target decay at time zero are subtracted from this value to determine the initial prompt heat generation rate. Since the local target decay is conservatively overestimated to be locally deposited, this results in a corresponding underestimation of the prompt contribution. However, as the “prompt” power fraction is bounded by the local target decay power fractions (see Figure 5.4), this represents a conservative total transient heat generation over all time steps. The time-dependent curves of the HFIR prompt and delayed photon heating are developed with the HFIR RELAP model (Figure 5.5).

FIGURE 5.4 Transient Power Fractions for Different Heat Sources from Shutdown to 0.6 Seconds Decay

FIGURE 5.5 Transient Power Fractions for Different Heat Sources from Shutdown to 10 Hours Decay

5.2.1.5 NU Foil Post-irradiation Radionuclide Inventory

Neptunium-239 is the dominant radionuclide in terms of curies for the first 10 to 15 days after shutdown. Neptunium-239 has a half-life of about 2.356 days and decays via β^- transitions to ^{239}Pu (with a mean β^- energy of 111 keV), which alpha decays with a half-life of about 24,000 years. The radioactivity of the NU foil reduces quickly over the first 10 days after shutdown due to the radioactive decay of ^{239}Np . The dominant fission products include, but are not limited to, ^{140}La , ^{140}Ba , Xe, I, and ^{99}Mo .

The maximum nickel heat generation rates were determined to be 27.19 W/g for position 8 and 39.85 W/g at position 5.

5.2.1.6 Summary of Results and Conclusions of Nickel Capsule Neutronics Calculations

Neutronics calculations were performed to support the irradiation of a high-density NU foil and nickel irradiation capsules in the HFIR HT for up to 1 full cycle or 10 days (for shipping requirements). The SCALE code (including the COUPLE, ORIGEN-S, and OPUS modules) was used to perform depletion, activation, decay, and source term calculations on the NU foil and nickel capsule assembly. The MCNP code was utilized for neutron, photon, and neutron-photon

transport calculations to estimate energy-dependent neutron fluxes, actinide cross sections, and heat generation rates.

Nuclear heat generation rates, nuclide inventories, and decay heating rates for the NU foil and its associated nickel capsule components (including the housing capsule), the inner sleeve, the foil's nickel coating, and the end cap were calculated. The maximum heat generation rates for the NU foil at positions 8 and 5 were determined to be 559.33 and 816.51 W/g, respectively. The maximum heat generation rates for the nickel capsule components at positions 8 and 5 were determined to be 27.19 and 39.85 W/g, respectively.

5.2.2 Thermal-Hydraulic Calculation Supporting Irradiating Nickel Capsules with NU Foils in the HFIR Hydraulic Tube

Seven calculations were performed with four capsules loaded in the hydraulic tube of the HFIR. The capsules are: one NU capsule and three generic capsules. The seven calculations are:

- (1) Steady-state normal operation at full power;
- (2) Steady state with flow blockage at full power and 50% of the full flow;
- (3) Steady state at 130% power and 100% flow;
- (4) Loss of offsite power (LOOP) with two pony motors;
- (5) Small break loss-of-coolant accident (SBLOCA);
- (6) Steady state at full power with upward flow of 0.5 gpm (valve error); and
- (7) Steady state with capsules loaded in the worst configuration (loading error).

The 130% power case was run at 110.5 MW (130% of 85 MW); the remaining cases were run at a full power of 87.6 MW (85 MW is the nominal value).

The NU capsule is loaded in position 8 (top position) and the generic capsules in positions 4, 5, and 6 of the hydraulic tube for the first six calculations (normal configuration). The first position (bottom position) is a capsule pusher, and the other positions (2, 3, and 7) are three dummy capsules. For Calculation (7), the worst configuration, the NU capsule is loaded in position 5, and the generic capsules are in positions 4, 6, and 7. This configuration results in the largest amount of heat generation. Dummy capsules are in positions 2, 3, and 8.

The calculations were performed with the transient thermal-hydraulics code RELAP5 Mod3.3, and confirmatory runs were made using ANSYS 14.5.7.

Different acceptance criteria apply to each calculation. For the steady-state normal operation case, the criterion is that the surface temperature of the capsules is below the saturation temperature of the coolant. Also, internal temperatures should be under 350°C in the NU capsule because of swelling. For the flow blockage case, the 130% power case, and the flow reverse case, the acceptance criterion is no onset of net vapor generation and no melting. For the LOOP transient and the SBLOCA, the acceptance criteria are no surface burnout, which includes heat fluxes below the CHF, and no onset of net vapor generation (i.e., no flow excursion). The temperature of the coolant must be below the saturation temperature at each elevation of the

capsule and at all times. Prevention of flow excursion can be conservatively satisfied by not reaching the onset of net vapor generation point, as calculated by using a well-known correlation like the Saha-Zuber correlation [SAHA-1974]. For this correlation the onset of net vapor generation is prevented when the Nusselt number in a flow channel is under 455 with the Peclet number under 70,000. The Nusselt number is defined as:

$$\text{Nu} = q'' D_H / (k \Delta T_{\text{sub}}) \quad (\text{Eq. 5.3})$$

with q'' being the heat flux, D_H , the hydraulic diameter; k , the thermal conductivity of the coolant (water in this case); and ΔT_{sub} , the subcooling of the coolant ($T_{\text{sat}} - T_{\text{coolant}}$).

5.2.2.1 Results and Conclusions

RELAP5 calculations of a high density NU capsule (in position 8) together with three generic capsules (in Positions 4, 5 and 6) have been completed for seven cases, as described above. The ANSYS mesh is shown in Figure 5.6. These calculations have demonstrated that: (1) the coolant temperature is below saturation at all times, (2) no net vapor generation in the coolant occurs, (3) no flow instabilities in the coolant are created, and (4) none of the materials approaches melting temperatures. There is a large margin to vapor generation or boiling since the minimum subcooling calculated is 13 K (for the SBLOCA transient) and the maximum Nu is 236.7 for the 0.5 gpm reverse flow case.

FIGURE 5.6 Mesh Used in the ANSYS 2-D Model

The maximum temperature calculated by RELAP5 inside the NU capsule is 670 K for the 0.5 gpm reverse flow case. This temperature is for the nickel spring inside the capsule. The maximum surface NU capsule temperature calculated by RELAP5 is 507 K, for the same case. Several conservative assumptions have been considered in these calculations – conservative heat generation rates, fission gas releases, dimensions, and radial gaps. Because of these conservatisms, all of the calculated temperatures in these transients are higher than the expected actual values.

The maximum temperature calculated by ANSYS for the NU capsule is 629 K for the spring in the 130% power case. The ANSYS and RELAP5 results agree well. The ANSYS calculations are also conservative as thermal expansions are not considered. Figure 5.7 shows the ANSYS calculated temperatures through the cross section of the rabbit target.

FIGURE 5.7 Radial Temperature (°C) for Normal Steady-State Operation

Thermal expansions (longitudinal, radial and axial) can be safely accommodated for the maximum temperatures calculated in any case with the available gaps. Also, NU swelling can be controlled as the NU maximum calculated temperatures are under 350°C for most cases (with the exception of the 0.5 gpm reverse flow case and the 130% power case, which would both result in reactor shutdown and would not have time to initiate growth in the uranium).

Steady-state (normal operations) temperatures are shown in Figures 5.8 and 5.9 for the ANSYS model for the entire capsule and only the uranium foil.

1

FIGURE 5.8 Calculated Temperatures (°C) for Normal Steady-State Operation

1

FIGURE 5.9 Calculated Peak Temperatures in the NU Foil during Normal Steady-State Operation

6 TARGET QUALITY

All research reactors conform to rules regarding the quality of components that are installed in the reactor facility. Many research reactors use a graded approach to quality levels based on probabilistic risk assessment methods. This also applies to irradiation targets. In the United States, Nuclear Quality Assurance-1 (NQA-1) is a regulatory standard created and maintained by the American Society of Mechanical Engineers (ASME). The NQA-1 program offers a rigorous and complete set of requirements and controls for assuring quality in nuclear reactor environments. At the HFIR, NQA-1 standards are applied to all activities considered safety related, including in-vessel irradiation targets.

6.1 NQA-1 PROCESS FOR INSERTING AN EXPERIMENT INTO HFIR

NQA-1 standards are implemented by means of procedures at ORNL. Specific irradiation experiment procedures control the experiment process that ultimately results in a document called an Experiment Authorization Basis Document (EABD). This document is reviewed, including all related calculations and assumptions, and an Un-reviewed Safety Question Determination is performed on the proposed experiment. The EABD also specifies the quality requirements necessary to meet the criteria for safety. Typically, the EABD preparation and actual target fabrication are occurring at the same time, so iteration may be necessary to bring both into sync.

The EABD includes a section describing the “as-built” or “as-fabricated” experiment, and demonstrates that what was fabricated matches the EABD quality requirements previously defined. This set of documentation fully describes the composition of the experiment capsule as well as the processes used to fabricate and assemble the capsule. The requirement is simply that all materials are fully documented and traceable back to raw material or stock, including such details as elemental composition for all stages of fabrication and assembly. This also includes process-related materials such as weld wire, welding cover gases, and assembly sweep gases. As-built fabrication information can be significant, the paperwork usually weighing many times more than the actual irradiation capsule.

Simply, the EABD documents that an approved plan has been prepared for an experiment AND that the experiment was actually executed according to the plan. Figure 6.1 shows the flowsheet.

For the annular targets, quality measures revolve primarily around the uranium-foil properties and the success of the assembly method. The aluminum 6061 and 3003 have full traceability, including the heat number at the manufacturer, and all other aspects such as weld wire and cover gases. Several of the most important quality measures include the following:

1. Verification of the enrichment and the physical size of the LEU foil.
2. Evidence of the contact between the uranium foil and the capsule cladding.

3. Demonstration that the welds are of high quality, meeting either code requirements or requirements defined in the EABD.

Of course, many fabrications do not go as planned. Sometimes tolerances are not maintained, or assembly procedures do not go as anticipated. This does not mean that the irradiation will not occur, but instead that there will be a non-conformance report (NCR) that describes what is different than planned. This NCR will be reviewed by a safety analyst to assess whether the bounding conditions and assumptions made in the safety calculations still apply to the target with this non-conformance. If there is no un-reviewed safety question created by the NCR, then the experiment can proceed.

FIGURE 6.1 Quality Flowsheet for an Experiment Authorization Basis Document

6.2 RESONANT ULTRASOUND SPECTROSCOPY AS A POTENTIAL QUALITY CONTROL TOOL

6.2.1 Introduction

Resonant ultrasound spectroscopy (RUS) is a non-destructive technique that can be used to evaluate the elastic properties of materials when the samples have been cut into simple geometric configurations. Such configurations include cubes and cylinders, for which exact analytic solutions or numeric solutions for resonant vibration modes can be derived. An example of a finite element analysis of a cylindrical structure is shown in Figure 6.2, where the bending positions of the first vibration mode are shown.

FIGURE 6.2 First-Vibration Resonance Mode for a Short Cylinder

While obtaining fundamental resonant frequencies for a given sample in a non-destructive manner may be useful, it is not practical for complex geometries and structures. However, we hypothesized that a spectral scan of a target will provide information about the integrity of the interface between the two cylinders. In particular, we hypothesized that a stronger resonant frequency signal will be present when the thermal interface is relatively good. A poor interface will result in a weak resonant signal. The purpose of the current study was to explore this hypothesis.

To obtain the free resonant vibration modes, it is necessary to mechanically stimulate the sample. At the same time, it is necessary to not dampen the vibration modes, so the coupling

between the sample and the vibration source needs to be made as little as possible. Clearly this is a challenge, and chattering is virtually impossible to eliminate. Further, for measurements to be taken, a detector needs to be put in contact with the sample to determine the sample response to the vibration input. A conventional configuration for an RUS system is shown in Figure 6.3. In that example, a cubic sample is placed between two piezo-electric transducers (one sender and one receiver) at opposite corners. The sender is stimulated by a function generator over a broad range of frequencies while the receiver is attached to a lock-in amplifier to filter out all received signals at frequencies different from the driving frequency. The resulting signal spectrum allows the user to determine which frequencies result in large signals that can be interpreted as the resonant or harmonic frequencies.

FIGURE 6.3 Representative Experimental Configuration for a RUS System

6.2.2 Experimental Procedure

To carry out a RUS analysis, it is necessary to excite a sample with constant amplitude over a range of frequencies, and then record the responses. The range of frequencies usually starts from 1 kHz or more and increases to around 3 MHz. The response is recorded with the use of a lock-in amplifier. Ultrasonic transducers made from PZT (piezo-electric material) were used to stimulate and sense the vibrations in the sample.

There is no adhesive between the transducers and the target, or mechanical support for the sample as it is necessary for the sample to vibrate in a free environment. Although the transducers touch the sample, it is assumed that the vibration happens freely since the inducer's cross-sectional area is small. The actual contact pressure between the transducers is important because it might affect the natural frequency. An AHF2LI (Zurich Instrument) lock-in amplifier was used to generate and record the vibration data. LabVIEW (National Instrument) was used as the data acquisition and control software package.

A cylindrical tube was used as a reference sample before taking measurements on a target structure. Since the sample is larger than typical RUS samples, two measurement configurations were used: one with two and the other with three transducers. For the two-transducer configuration, it is necessary to provide additional mechanical fixtures to hold the sample in position as the transducers are not able to mechanically hold the sample in place (see Figure 6.4). The three-transducer configuration allows for the transducers to be oriented in such a way that they hold the sample without any external fixture. In the three-transducer approach, one of the transducers is used to vibrate the cylinder, and the other two transducers are used to receive the signal (see Figure 6.5).

(A)

(B)

FIGURE 6.4 Photographs of Two Transducer Configuration

(A)

(B)

FIGURE 6.5 Three Transducer Configuration: (A) Transducers without the Sample and (B) Transducers with the Cylindrical Sample

6.2.3 Results and Conclusions

The spectral scan for a cylinder made from 6061 aluminum that is 6.313 in. (16.03 cm) long with inner diameter of 0.992 in. (2.520 cm) and outer diameter of 1.131 in. (2.873 cm) is shown in Figure 6.6. It shows a peak at a frequency of around 750 kHz. This is in contrast with the three-transducer configuration profiles shown in Figure 6.7, where there are peaks around 1.5 MHz for both receiving transducers, which is roughly twice the frequency peak for the two-transducer configuration. The explanation for the difference in peak frequency between the two configurations is unclear at present.

FIGURE 6.6 Spectral Response of Single Cylinder Using Two-Transducer Configuration

FIGURE 6.7 Spectral Responses of the Two Receiving Transducers (A and B) for a Single Cylinder Using the Three-Transducer Configuration

A sample target was also experimentally evaluated using both the two- and three-transducer configurations. The geometry of the target tubes is shown in Figure 6.8. The tubes were drawn together before testing, but the ends were not welded.

FIGURE 6.8 Geometry of Target Tubes Used for RUS Experiment

The spectral response for the two-transducer configuration is provided in Figure 6.9, while the response for the three-transducer configuration is given in Figure 6.10. From Figure 6.9, there does not appear to be any particular frequency for which there is a dominant peak. However, a dominant peak for the three-transducer configuration appears at around 1.5 MHz. A similar frequency is seen for the single-cylinder response shown in Figure 6.7. However, the magnitude of the peak for the target is about two orders of magnitude smaller than the peak magnitude for the cylinder. Again, at this time, there is no explanation for this behavior. At this point, we cannot determine any benefit to using RUS as a tool to interrogate the interface areas of annular targets.

FIGURE 6.9 Spectral Response of Target Using Two-Transducer Configuration

FIGURE 6.10 Spectral Response of Target Using Three-Transducer Configuration

7 TARGET IRRADIATION

Unfortunately, due to fabrication delays at Y-12, no irradiations of either the capsule or the annular target could be performed in HFIR.

8 TARGET DISASSEMBLY

A major benefit of using the LEU-foil based target is that it can be disassembled to extract the LEU for dissolution. This reduces the amount of high activity liquid waste compared to dissolving the entire target, cladding included. The challenge here is to cut open the target cladding so that the LEU foil can be removed in a hot cell environment.

The strategy typically employed for opening the target is to first cut off the welded ends of the tubes and then make a longitudinal cut along the length of the outer tube. The longitudinal cut is located in the gap between the ends of the LEU to avoid cutting through the fission-recoil barrier (see Figure 3.33 for a photograph of the foil gap). The cut is typically made through the outer tube wall only as the residual hoop stress from the target fabrication process facilitates removal of the outer tube. Figure 8.1 shows a photograph of a target with the outer tube slit open and the tendency for the outer tube to separate a little from the inner tube. It should be noted that this extent of opening after irradiation is not always observed.

8.1 PREVIOUS WORK

A brief overview of previous cutters is presented here [IAEA-2015].

FIGURE 8.1 Target with Outer Tube Slit Showing Opening That Results from Residual Stress [HARRIS-2013]

Two abrasive-cutting-wheel concepts were developed and built by Argonne and Comisi3n Chilena de Energia Nuclear. Photos of these designs are shown in Figures 8.2–8.4. In both cases, the target was rigidly held stationary while the cutting wheel made the cuts. After each of the welded ends was removed, the target was rotated and held in place to make the longitudinal cut. The Chilean device improved on the Argonne cutter by adding a second cutting, which allowed both ends of the target to be removed simultaneously. A significant disadvantage of these designs for use in a hot cell is the cutting debris that is generated.

FIGURE 8.2 Argonne National Lab Target Cutting Design [BAKEL-2006B]

FIGURE 8.3 Chilean Target Cutting Device in Position to Remove Both Welded Ends

FIGURE 8.4 Chilean Target Cutting Device in Position to Make Longitudinal Cut

An improvement over using an abrasive cutting wheel is to use a tubing cutter. A new disassembly device based on a conventional tube cutter that can be purchased from a hardware store was designed and built at the University of Missouri. A photo of the device that was actually used to remove the LEU foil from an irradiated target at MURR is shown in Figure 8.5. This device is manually driven using a manipulator arm. A tube cutter is mounted onto a crank fixture, as shown on the right-hand side of the photo. The target is set into a cradle and held in place with an arm. There is only one tube cutter so the target needs to be flipped on end in order to cut the second welded end off. The cradle assembly is then translated along the guides in the base of the device to make the longitudinal cut using the wheel mounted on the retaining arm. Multiple passes are necessary to complete the cut through the outer tube.

FIGURE 8.5 Target Cutter Used Previously at MURR for LEU Processing

8.2 CURRENT PROJECT

The University of Missouri design eliminated dust and debris in the hot cell caused by abrasive grinding approaches. The device is also very compact, taking up very little space inside the constrained hot cell. However, the manual operation is slow, and the use of a hand crank would make it difficult to use for the fission-gas-collection experiment to be described in Section 6.3. To overcome those drawbacks, alternative disassembly devices have been designed, prototyped, and used.

8.2.1 ICN Target Cutter Design

One design concept was developed by ICN in Pitesti, Romania. A photo of their prototype is shown in Figure 8.6. The device was designed to extract the LEU foil from the target for later post-irradiation examination (PIE). While the photo shows hand-cranks that are used for driving the tube rollers and cutters for prototype experimentation, electric motors and pneumatic cylinders can be used when the device is implemented in a hot cell. A gas-trapping cover can also be integrated with the device to allow a fission-gas release experiment to be conducted before other PIE work on the LEU foil. The device initially makes a cut through the outer tube only near the welded ends. This is in contrast to the way in which all other devices work, where they cut off both of the welded ends. The cut is made using cutters that are held stationary and then driving the target to rotate by using knurled rollers. A cut-depth limiting gauge is placed around the cutters to ensure that only the outer tube gets cut.

After the end cuts are made, a pair of cutting wheels set at the depth of the outer tube thickness is driven along the length of the target in the gap between the ends of the LEU foil. This removes a longitudinal strip of the outer tube. Through a series of drivers and a knife edge, the outer tube is peeled away from the target. A second knife edge then scrapes the surface of the intact inner tube to collect the LEU foil. The foil is guided into a collection box where it is coiled. The box is then removed from the disassembly device for further PIE work after the fission gas trapping cover is removed from the device. To our knowledge, a fully working prototype has not been demonstrated or used as part of an LEU-foil based target study.

FIGURE 8.6 Target Disassembly Device to Facilitate PIE at ICN

8.2.2 University of Missouri Target Cutter Design

The disassembly approach taken by the University of Missouri to complete the fission-gas-release experiment (discussed in Section 8.3) was to combine features from the ICN design with the manual device used previously at MURR. It was designed to be compact and electric motor driven so that it could be driven with an electronic control panel once the target was placed on the device in the hot cell. A general concept of the device is sketched in Figure 8.7.

FIGURE 8.7 General Sketch of Automated Disassembly Device Developed at the University of Missouri [HARRIS-2013]

The extensive design analysis that was conducted during the development of the device is described in the two undergraduate honors theses written by Harris [HARRIS-2013] and Gunn [GUNN-2013]. An overview will be given here.

8.2.2.1 Design Details

A photo of the original prototype is shown in Figure 8.8. In this design, the tube is rotated by knurled rollers that are on an axle driven by an electric motor. In the photo, the motor is configured to directly drive the axle with a worm gear (not shown). In the final implemented design, the motor was rotated 90°, and the worm gear was replaced with a chain drive linking the motor and the roller drive axle. A closer view of the target sitting on roller drive axels and the cutter wheels is shown in Figure 8.9. The cutting wheels are adjusted into position and held in contact with the target using the vertical positioning motor shown in Figure 8.8. The motor is connected to the fixture holding the cutting wheels with a ball screw.

The longitudinal cut along the length of the target is made with a cutting wheel mounted onto a block that is driven by a ball screw. The cutting wheel can be seen in contact with the bottom of the target in Figure 8.9, between the two bottom roller axels. A photo taken from underneath the cutter is shown in Figure 8.10 and shows the block that the cutting wheel is attached to, as well as the drive screw that moves the cutter longitudinally. While the longitudinal cut is being made, resisting force is applied to the top of the target with the spring-loaded pressure plate shown in Figure 8.11.

FIGURE 8.8 Photograph of University of Missouri Automated Disassembly Device Prototype

FIGURE 8.9 Target Sitting on Rollers with Cutting Wheels in Contact

FIGURE 8.10 Longitudinal Cutting Wheel

FIGURE 8.11 CAD Drawing of Disassembly Device Used for Fission-Gas-Collection Experiments

Small modifications were made to the disassembly device based on preliminary target-cutting experiments. Those modifications were made before the final design was eventually used in the fission-gas-collection experiments (Section 8.3). Those modifications are pointed out in the CAD drawing shown in Figure 8.11. The biggest change was to chain-drive motion control with stepper motors. The use of chain drives allowed the tolerances and positioning of the axels and motors to be looser, and potentially easier, to maintain in a hot cell environment, where any repairs are difficult to carry out. A second change was to add in a spring-loaded pressure plate that holds the target into contact with the bottom rollers (the drive roller and the other idler). The pressure plate position is controlled by the vertical stepper motor and allows the downward force to be controlled during the longitudinal cut. It also allows for more robust alignment as the springs can adjust for any irregularities in the geometry of the target after irradiation. For components that are likely to fail or wear out, pins such as the one shown in Figure 8.12 are used to hold those components in place. It should be noted that all components are machined from 306 stainless steel.

FIGURE 8.12 Pin Used to Hold Components That Will Likely Need Replacing

8.2.2.2 Mechanical Analysis

Mechanical analysis of each of the load-bearing components was completed to determine the potential for any component failure and to help select motor control parameters. The structural analysis was conducted using the commercial FEA code ABAQUS. Some of the key areas analyzed are identified in Figure 8.13. Two examples of the analysis will be presented here, with the interested reader referred to the undergraduate theses by Harris [HARRIS-2013] and Gunn [GUNN-2013] for the analysis of other components.

FIGURE 8.13 Locations of Key Components Where Structural Failure Is Likely

8.2.2.2.1 Load-Bearing Components

The pins that hold the cutting wheels in position, locations C and E in Figure 8.13, are likely to experience a significant shearing load that could lead to the failure. The pin holding the wheel at location E can experience forces due to the movement during the longitudinal cutting process that depends on the loading from the spring-loaded pressure plate. Assuming a cutting force of 100 pounds (445 N) as experienced by the longitudinal ball screw, the resulting stress distribution on the pin is as shown in Figure 8.14. The maximum stress levels occur where the pin meets the hole edges. With a maximum stress of just over 7100 psi (49 MPa) and the yield stress of stainless steel as about 25,000 psi (172 MPa), the factor of safety for the pin is over 3.5. So as long as the cutting force is kept at or below 100 pounds-force (445 N), there should be no risk in the pins failing due to excessive loading, at least before any radiation damage is incurred.

FIGURE 8.14 Finite Element Analysis Stress Field for the Longitudinal Cutting Wheel Retaining Pin [GUNN-2013]

8.2.2.2.2 Mechanical Analysis of Target Deformation

A deformation analysis was carried out to determine how the target will flatten out on the bottom during the longitudinal cutting. An analysis was conducted where the target was constrained at four locations indicated by green arrows in Figure 8.15. These locations represent

the positions of the roller supports on the bottom and the upper spring-loaded plate. A 60 pound (270 N) force is applied from the bottom to represent the longitudinal cutting wheel making a cutting pass. The deformation plot suggests that the bottom of the target will indeed flatten out by around 10 mil (0.25 mm) during a cutting pass. A 60 pound (270 N) force is consistent with about 1/5 of the full spring compression from the upper spring-loaded plate. The amount of deformation is approximately 10% of the thickness of the outer tube. By applying a lighter pressure, less flattening will take place, but the depth of cut during a longitudinal pass will also be less, and additional passes will be necessary.

FIGURE 8.15 Finite Element Analysis of Target Deformation during Longitudinal Cut [GUNN-2013]

The ABAQUS calculated results for deflection analysis suggest that the largest risk is for the target to deform during cutting. We determined how the cutting force applied relates to cutting depth, number of cutting passes, and applied shaft torque.

Figure 8.16 provides an illustration of the cutting wheel as it is cutting the outer tube and the assumed area for the shear forces. On the basis of an analytic study, described in [GUNN-2013], the relationship between the shear force and the cut depth is given in Figure 8.17.

The curves indicate that deeper cuts generate a larger shear force on the cutting wheel and will apply a larger load to the cutting wheel pins and cause more deformation of the target. Minimizing the target deformation to less than 10% of the outer tube thickness will keep the forces below 50 pounds (220 N), which means the cutting depth must be about 15 mil (0.4 mm) for each cutting pass.

It is difficult to control the cut depth from a remote location, as will be needed when cutting open targets in a hot cell. What can be detected from a remote location is the applied torque from the stepper motors that drive the longitudinal cut-ball screw. Therefore, the relationship between the applied force at the cutting wheel and the motor torque needs to be developed. The relationship can be developed with the aid of the sketch shown in Figure 8.18. It can be seen that the cutting force is transferred to the ball screw via a bearing embedded in the mounting block. For the diameter of the ball screw used in the present design (1/3 in., 8.4 mm) and gear/screw analysis described in [GUNN-2013], the required motor torque corresponding to a given cutting depth can be found. For a targeted cutting depth of 15 mil (0.4 mm), the required motor torque is estimated to be about 0.2 lb-in. (23 mN-m). This value provides the needed specifications when sizing electric motors and a means to control the cutting depth through electronic control of the stepper motors.

All of the stepper motors are controlled using an Arduino-based microcontroller. The Arduino platform provides a simple means to control the electric loading to the stepper motors, as motor shields are widely available and only require the user to provide an external power supply. While the analysis just presented the initial starting constants for the control program, the constants were adjusted through experimentation to the point where between 5 and 10 cutter passes completely cut through the outer tube.

FIGURE 8.16 Schematic Showing Depth of Cut into Outer Tube with Assumed Area for Shear Forces [GUNN-2013]

FIGURE 8.17 Relationship between the Cutting Depth and Cutting Force at the Cutting Wheel and the Corresponding Motor Torque

FIGURE 8.18 Structural Relationship between Shear Force Caused by Target Cutting and Torque Required at the Ball Screw Shaft

8.3 FISSION GAS COLLECTION FROM AN IRRADIATED LEU TARGET

Before irradiating a target, reactor operators need to conduct a safety case analysis for the purpose of establishing that operating technical limits will not be violated. In the case of a fission-based target, like the high density target (HDT), material temperature and fission product release criteria are generally limiting. While there is evidence that all fission gases will not be released in a fissioning sample if there is a mechanical breach [VISWANATHAN-2009] in MURR and other reactors, we assume that ALL of the gas produced in the target gets released into the facility. The theoretical isotopic release for an LEU foil (Appendix B-1) limits the mass that can be irradiated to 5 g, significantly smaller than what is expected for a production target.

A concern regarding fission gases is the potential release inside the frontend processing hot cell when the target is cut open, before the LEU dissolution. The release of fission products will need to be trapped by the ventilation system filters, and some of the fission products will likely be released through the stack to the environment. While the dose rates may be small, the release can confound nuclear-weapon-testing monitor readings [BOWYER-2014].

It is hypothesized that only a fraction of the total fission-product gas produced in the LEU foil will be released if the target fails mechanically. Some of the produced gas will be trapped inside the foil matrix and not be able to escape. The balance of the fission gas would be trapped within the fission-recoil barrier that is wrapped around the LEU foil. Experiments executed to test the hypothesis are described here.

The experimental approach was to assemble two annular targets using 5 g of LEU foil fabricated at Argonne. The target cladding was 3003 series aluminum, and target dimensions were based on the Argonne design for targets irradiated in Indonesia [CONNER-2000]. The 3003 aluminum and the Argonne design were chosen because they both have been used as part of an irradiation at MURR in the past, and did not require any additional safety case analysis. The 5-g LEU foil was manually wrapped with a 15- μm -thick nickel foil as a fission recoil barrier for the first irradiation and a 10- μm -thick aluminum foil as the fission recoil barrier for the second irradiation. The targets were opened in a fission gas collection system, where the activity of the released off-gas was counted. The count value was related to the trapped gas through a geometry calibration obtained from previous tracer runs. Details of the fission-gas collection system and tracer run calibration follow.

8.3.1 Design of Fission-Gas Collection System

The LEU-foil targets were irradiated at MURR in a neutron flux of 1.5×10^{13} n/s/cm² for 150 hours. At the end of the irradiation, the targets were pulled from the irradiation position and allowed to decay in the pool for at least 20 hours before moving to the hot cell for processing. Within the hot cell, the targets were opened, and the fission gas was counted in the fission-gas collection system.

The design of the fission-gas collection system followed the design described in previous studies. Specifically, studies used a non-condensable sweep gas flowing over an irradiated sample to entrain released gases [PARSLY-1968, MERRIMAN-1970, MELEHAN-1961, SLUTSKY-2009, HOFMAN-1996]. The sweep gas exiting the confinement was chilled by using refrigerants or liquid nitrogen to condense or dissolve the gases of interest (krypton, xenon, and iodine). Merriman et al. [MERRIMAN-1970] attempted to dissolve the entrained gases in liquid refrigerants and then separated the gases by using the difference in dissolution coefficients for each gas as the means to separate the krypton from the xenon. A similar strategy was used by Parsly and Franzreb [PARSLY-1968] to remove iodine from a potential accidental release. Counting was done after the sweep gas was captured in a container and then moved elsewhere for gamma counting. For all cases, the amount of fission gas observed was relatively small. For this experiment, we decided to design a condenser that contained activated charcoal and was chilled by liquid nitrogen. The intent of adding the activated carbon was to increase the surface area over which the fission gases may condense and/or offer the potential for the fission gases to adsorb within the carbon structure [ROSE-1969, MYERS-1965, BAZAN-2008, BAZAN-2011, DO-2003, KITANI-1965, LEVENSON-1971]. The sweep gas was selected to be helium. Instead of attempting to trap the gases for counting at another location, we decided to take the gamma-count measurement in real time at the condenser.

A description of the data-collection process is provided by [SOLBREKKEN-2014]. A sketch of the overall layout is shown in Figure 8.19. It can be seen that the automated target cutter placed in a collection box was mounted into the main chamber of the hot cell. The collection box is shown in Figure 8.20 and can be noted to have a gasket around the latch-able doors to create a seal. Helium gas is introduced into the collection box through the top and drained through the bottom. The down-flow over the target is intended to drain fission gases released as the target is cut open. The drained gas mixture is ducted to the condenser (Figure 8.21) that is held in the Dewar (Figure 8.22) that is located in the counting well (a separate shielded cavity that is mounted below the hot cell). Liquid nitrogen is flowed into the Dewar continuously to maintain the condenser at cryogenic temperatures. The sweep gas is then vented from the condenser back into the hot cell, where exhaust filters can trap any un-condensed fission gases. A high-purity germanium gamma detector is positioned outside the counting well, as seen in Figure 8.23, to take gamma-count measurements from trapped fission gases while the sweep gas is still flowing.

FIGURE 8.19 Layout of Fission-Gas Collection System

FIGURE 8.20 Target Cutter inside the Fission-Gas Collection Box

FIGURE 8.21 Fission-Gas Condenser

FIGURE 8.22 Custom-Built Dewar to Hold Fission-Gas Condenser

FIGURE 8.23 Gamma Counter Directly In-line with the Dewar in the Counting Well

8.3.2 Fission-Gas Collection System Calibration

The overall objective for the fission-gas counting experiment was to estimate the release when the target is cut open. It was, therefore, necessary to relate the number of gamma counts observed by the detector coming from the condenser to the activity that exists at the target. Vials with known doses of Xe-133 were used as the basis for conducting the calibration study.

One 10 mCi and two 20 mCi Xe-133 sources were tested on three days to complete the calibration process. The calibrated sources were from Lantheus Medical Imaging. The vials were taped to the front of the condenser and counts taken. After scaling based on area and decay, the relationship between gamma counts and known activity at the location of the condenser was determined.

The second part of the calibration was to determine how the activity trapped in the condenser relates to a release in the fission collection box. The vials were placed in a specially designed fixture that was placed in the target cutter. The fixture held the vial in such a way that one of the cutters crushed the vial when lowered. The box door was closed, and the helium sweep gas flowed through the box to purge the air out of the box. After the air was purged, liquid nitrogen was added to the Dewar that held the activated charcoal-containing condenser. While the gas was purging the collection box, counts were taken with the gamma detector.

After a baseline gamma-count reading was taken to obtain the background gamma release, the glass vial was crushed in the target cutter. Gamma counts were taken every 5 minutes until steady results were obtained. At that point, it was assumed that all of the tracer gas released in the box had been swept away by the helium. Counting continued for at least two additional hours to make sure that steady state had been achieved. The calculated activity in the condenser was then compared to the activity measured in the original vials taped to the front of the Dewar to determine the fraction of the released gas that was captured. The detector is positioned vertically to obtain the largest signal, even though radiation counts existed over the vertical range of the 6-in. tall condenser. Further, all of the gas was likely not captured within the condenser, but rather vented into the hot cell. That amount was not quantified.

8.3.3 Results and Discussion

The background gamma count rate for the 81 keV signal corresponding to the presence of Xe-133 was less than 1 count/s. This value is less than 1% of the final steady count rate for all of the tracer and LEU target experiments that were carried out. The calibration curve relating the measured, steady count rate to the released dose of Xe-133 in the fission collection box for the tracer runs is shown in Figure 8.24. Since only two nominal tracer doses were used for the calibration samples (around 20 mCi and 5 mCi after time de-rating), a straight-line profile was assumed. The equation of the best-fit line through those points while forcing the intercept to be 0 (corresponding to a nominally 0 background noise) is shown in the plot. Further analysis/calibration experiments would be needed to confirm if the relationship is indeed linear.

FIGURE 8.24 Calibration Curve Relating the Steady-State Count Rate to the Released Dose in the Fission Collection Box

The LEU-foil targets were irradiated at MURR on two occasions. Table 8.1 describes the different fission-recoil barriers, along with the mass of each LEU foil. Again, the irradiations took place in an irradiation position at MURR (hole K-2), where the neutron flux is about 1.5×10^{13} n/s/cm² for about 150 hours. After a cooling period of about 20 hours in the pool, the targets were moved in a transport cask and put into a hot cell. The targets then were placed onto the target cutter located in the fission collection box (Figure 8.25).

TABLE 8.1 Recoil Barrier and LEU Mass for Two Irradiated Targets

Recoil Barrier	LEU Mass
15- μ m thick Ni foil	4.1846 g
10- μ m thick Al foil	4.7536 g

FIGURE 8.25 Placement of Irradiated Target onto the Cutter

The transient count rate observed for both of the targets can be seen in Figures 8.26 and 8.27. It can be seen that immediately after the targets were cut open, the gamma counter began to show evidence of Xe-133 being trapped in the condenser. The count rate continued to increase until about 2.5 hours after the target was opened. At that point, the count rate leveled off. While not shown explicitly in the plots, counting continued for another 2 hours, where no count-rate changes were observed. It was interpreted that all of the released fission gases had been purged from the fission gas collection box 2.5 hours after opening the target.

FIGURE 8.26 Transient Response of 81 keV Gamma-Ray Count Rate from the Nickel-Wrapped LEU Foil Target

FIGURE 8.27 Transient Response of 81 keV Gamma-Ray Count Rate from the Aluminum-Wrapped LEU Foil Target

The calibration curve shown in Figure 8.25 was used to convert the gamma-ray count rate observed at the condenser to the amount of Xe-133 that was released by the targets during opening. That activity was scaled to a reference time of 20 hours after the irradiation was completed. Twenty hours was chosen as a reference time since that is the amount of time required by MURR to allow short-lived isotopes to decay and represents a typical time when a target might be processed by a producer. Thus, the gas release represents what would be expected to be given off in a processing hot cell. The time-scaled activity on a per-mass-LEU basis is summarized in Table 8.2.

Measuring the release fraction of the total inventory of Xe-133 that was produced during the irradiation of an LEU target was the objective of this study. Therefore, it was necessary to know the total amount of Xe-133 that is produced from fissioning in the foil. The difference between the amount of released gas and the total gas production is assumed to be trapped within the foil matrix itself. The fraction release can then be used by irradiators to help estimate the amount of release in the event of a target cladding failure for any size LEU foil.

The total amount of Xe-133 generated during the irradiation of an LEU foil target was estimated by using ORIGEN 2.2, as outlined in Appendix B-1. The results from that study have been scaled for neutron flux, LEU mass, and time after irradiation. That scaled activity is shown in Table 8.2.

It is known that the output from the ORIGEN code does not necessarily represent the actual isotope production. A study was carried out at MURR in 2010 that evaluated the actual isotopic content in an LEU foil wrapped with a nickel recoil barrier that was irradiated in 2009, similar to the targets that are irradiated as part of this study. In that study, the long-lived isotopes Ru-103, Ru-106, Cs-137, Ba-140, and Ce-144 were gamma counted and compared with the

ORIGEN predictions. It was found that the actual activity of each of those isotopes was about 54.2% of the ORIGEN calculation. Therefore, it is assumed that for the current irradiations and Xe-133, the same fraction applies. That corrected activity for the ORIGEN calculation of Xe-133 is also shown in Table 8.2.

Finally, the fraction of Xe-133 that was counted and assumed to be released during the opening of the targets is shown in the last column of Table 8.2, based on the experimentally corrected activity calculation from ORIGEN. It can be seen that for both targets evaluated, the fraction released is less than 0.04% of the total gas inventory, suggesting that most all of the Xe-133 remains trapped inside the LEU foil or within the fission-recoil barrier wrap. In terms of a comparison between the nickel and aluminum recoil barriers, the relatively small magnitude in fractional release (0.01%) and the uncertainties associated with this experiment make it difficult to make a conclusive statement of one being better at trapping fission gases than the other.

TABLE 8.2 Summary of Xe-133 Release for LEU Target Experiments

Target	20-hr Activity (mCi/g) Experimental	20-hr Activity (mCi/g) ORIGEN	Corrected Activity (mCi/g) ORIGEN	Fraction of Xe-133 released (%)
Ni Recoil Barrier	0.80	3338	1809	0.04
Al Recoil Barrier	0.63			0.03

9 FRONT-END PROCESSING

9.1 DEVELOPMENT OF THE FRONT-END PROCESSES

9.1.1 Introduction

The goal of this part of the project was to develop means for the annular LEU-foil target to be used by current producers with only minimal changes to their Mo-purification processes. The output to the front-end process must be an alkaline solution of Mo-99 with an impurity content similar to that generated by dissolving the HEU-dispersion target in a basic solution.

A proceedings paper [BROWN-2014A] presents the Argonne work to develop and demonstrate front-end processes that will allow Mo solutions produced by dissolving LEU foils to be fed into current producers' purification processes. The proceedings paper is the basis for the expanded description below.

The two front-end processes developed as part of this project are (1) the dissolution of irradiated LEU foil (up to 250 g in a single batch) and nickel fission-recoil barrier in nitric acid at ambient pressure followed by separation of Mo on a titania column and (2) the electrochemical dissolution of LEU foil followed by actinide and fission product precipitation. Both these front-end processes produce an alkaline (basic) solution feed for Mo purification. The flow diagrams for these processes are compared in Figure 9.1.

FIGURE 9.1 Flow Diagram of Two Front-end Processes for LEU-Foil Dissolution and Mo-99 Recovery

9.1.2 Nitric Acid Dissolution

A nitric-acid-dissolver system designed for the dissolution of up to 250 g of irradiated LEU foil and associated fission recoil barrier metal (e.g., Ni) at ambient pressure has been tested with irradiated and non-irradiated foils (Figure 9.2). The design, fabrication, and previous performance tests for this system are described in more detail in previous reports [JERDEN-2009, JERDEN-2010, JERDEN-2012A, JERDEN-2012B, JERDEN-2012C, JERDEN-2013, JERDEN-2014]. A second series of reports discusses in more detail the chromatographic separation of Mo from the acid solution and its recovery in alkaline solution [STEPINSKI-2011, STEPINSKI-2012, STEPINSKI-2014, and JERDEN-2013]. A report was also written that estimates of compositions and volumes of all waste streams generated from this front-end process [JERDEN-2011]. The process was tested for operability in an Argonne hot-cell-mockup facility before being sent to ORNL for a demonstration using a target to be irradiated in HFIR [VANDEGRIFT-2013].

9.1.2.1 Design and Testing of Foil Dissolution

The dissolver system components were tested at full scale. The key design criteria of the acid dissolver system are summarized below:

- All water vapor, reaction products, and fission gases must be contained within the dissolver system at a maximum temperature of 125°C and 2 atm of pressure (absolute) under both normal and off-normal (loss of cooling during reaction) conditions.
- The acid-feed system must be designed so that the thermally hot LEU foil (hot from decay heat) can be immersed in nitric acid without losing solution due to instantaneous boiling.
- Gas-trap components must be designed to trap/neutralize all nitrogen oxide and acid gases (NO, NO₂, HNO₂, HNO₃) as well as trap iodine gas.
- Noble fission gases are passively contained within the system.
- All dissolver system components must be designed for remote operation in a hot-cell facility.

FIGURE 9.2 Dissolver System Design Drawings and a Photograph of the Layout

The volume and concentration of nitric acid used for a given dissolution run depend on the mass of the metal being dissolved as well as the desired final acid concentration of the “product” solution (i.e., the solution produced by dissolution experiment). Controlling the final acid concentration is important for optimizing the Mo-99 extraction step that follows dissolution. The volumes and concentrations of acid as well as the amount of nitrogen oxide gas (NO , NO_2 , and N_2O_4) that will be produced are determined by the following general reactions:

In the presence of oxygen, the $\text{NO}(\text{g})$ produced in these dissolution reactions is rapidly converted to $\text{NO}_2(\text{g})$:

When water vapor and oxygen are present, NO_2 is readily converted to both nitrous and nitric acid vapors [$\text{HNO}_2(\text{g})$ and $\text{HNO}_3(\text{g})$], which will dissolve in condensed water and flow back down into the dissolver. One of the main purposes of the experiments discussed below was to determine how much acidity is lost from the dissolver solution as a result of the loss of NO_x and acid gases from the condenser section of the dissolver.

Three experiments were performed as part of this study: First, 197.26 g depleted uranium was dissolved discontinuously, that is, the test was interrupted to determine the extent of dissolution after 20, 40, 80, and 90 min. The acid dissolver solution was not changed out during

the interruptions of this test. Second, 202.02 g depleted uranium was dissolved with minimal heat input to measure the temperature profile produced by the heat of dissolution of the foil. Third, 242.4 g depleted uranium foil was combined with 6.84 g irradiated LEU foil and dissolved with the full heat load. Nickel metal was added to each test in an amount consistent with its presence as a fission recoil barrier (7–10 μm thick) in the actual target.

The starting acid concentrations for these experiments were 9.7 molar for the 197 and 202 g U tests and 11.5 molar for the 242 g U test. These concentrations are in excess of the starting concentrations required to end up with 1.0 molar nitric acid if the dissolution follows the stoichiometry described in reactions (9.1) and (9.2) above. The higher-than-stoichiometric starting acid concentrations were used to counter for the loss of acid through the loss of NO_x gas during the tests. Using the potentiometric titration method of Motojima and Izawa [MOTOJIMA-1964], we found that for all three tests there was only minor (if any) decrease in acidity due to the loss of NO_x gas during the dissolution. The acidities of the final solutions were as follows: 1.8 M for the 197 g U test, 2.1 M for the 202 g U test, and 2.1 M for the 242 g U test. These results indicate that the dissolution is close to following the stoichiometry shown in reactions (9.1) and (9.2). The final solution from the 242 g U test was adjusted, by using NaOH, to 1.0 M acidity prior to transferring it for the Mo-99 extraction step.

9.1.2.2 Mo Column Recovery and Strip

The dissolved irradiated target was followed by Mo recovery on a titania column. In this step, ~ 650 mL (899 g of solution, ~ 350 g-U/L) of irradiated target solution was loaded on a 3.5×5 cm column containing 63 g of Sachtopore[®] S40 sorbent at 25 mL/min and 60°C. To mimic the molybdenum concentration in a batch of fully irradiated targets, stable Mo in the form of Na_2MoO_4 was added to the feed to increase the concentration of Mo to ~ 5.4 mM. The loading was followed by ~ 260 mL 1 M HNO_3 wash and 75 mL H_2O wash. The Mo was stripped from the column with 0.1 M NaOH at 5 cm/min and 25°C. The strip was divided into four fractions containing 224, 292, 293, and 120 mL of solution.

The Mo recovery process was designed by using VERSE simulation program (Purdue University, IN) to determine intraparticle diffusivity and column process designs [STEPINSKI-2011]. Isotherm parameters, as well as solution, sorbent, and mass transfer parameters, were determined for column designs using Sachtopore sorbent S40 (40- μm particle size). Column designs were calculated for loading 500 mL LEU target solution containing ~ 7 mM Mo, ~ 450 g-U/L, and the nitric acid after dissolution (~ 1 M). The loading time of 20 min was chosen to limit the total column process time to 1 hr. Column length was limited by a ΔP constraint of 0.8 atm based on use of a vacuum/gravity fluid-transfer system. Two column designs were determined for loading LEU target solution at 25 mL/min; 3.5×5 cm with superficial velocity of 2.60 cm/min and 5×2.5 cm with superficial velocity of 1.27 cm/min. The former column design was selected due to its slightly more efficient sorbent utilization and was the basis for the Mo recovery process (Table 9.1). The column design was tested by loading tracer Mo-99 in uranyl nitrate solution containing 7 mM Mo, 450 g-U/L, and 1 M H^+ on the column, followed by recovery of Mo with 0.1 M NaOH by counter-current stripping at 3 cm/min and 25°C. Then,

99.4 ± 3% of Mo was loaded on the column, and 99.9 ± 3% was recovered in strip fractions #2 and #3 (6 BVs each).

TABLE 9.1 Process for Recovery of Mo from LEU Target Solution Using 3.5 × 5 cm Column and S40 Sorbent

Step	Loading Time (min)	Volume (mL)	Flow Rate (mL/min)	Velocity (cm/min)	Solution Volume (BV)
Load	20	500	25	2.6	
Wash, 1 M HNO ₃	8	385	48.1	5	8
Wash, H ₂ O	3	144	48.1	5	3
Strip, 0.1 M NaOH	26.7	770	28.9	3	16

The results in Table 9.2 show partitioning of molybdenum, uranium, and fission products between process streams during column processing of the irradiated foil solution. All activities were decay corrected to the start of the Mo recovery process (5/1/2014 at 8 am). The results show percent partitioning of irradiated solution components in the process streams, normalized to 100%. The actual mass balance is also indicated in Table 9.2. The results of Mo partitioning indicate that 99.3% of Mo was loaded on the column. Subsequently, 28.3% of Mo was in strip fraction 2, and 70.1% of Mo was in strip fraction 3, for a total of 98.4% Mo recovery (product). Strip fractions 1 and 4 contained 1% of Mo. The Mo activities in the nitric acid and water washes were below detection limits. Furthermore, the results in Table 9.2 indicate that the majority of radionuclides, including uranium, iodine, alkaline earth metals (Ba and Sr), transition metals (Cd, Rh, and Ru), and lanthanides (Ce and Nd), partition into the eluent and nitric acid wash streams. Only Sb (~60%) was found in the strip fraction 1. Other irradiated target components such as Ru, I, Te, and Zr partitioned <1% into the Mo strip fractions 1–3. Fractions of Sb (1.6%), Te (7.1%), and Zr (1.4%) remained on the sorbent. The activities of I-131 ($t^{1/2} = 8.02$ days) and La-140 ($t^{1/2} = 1.68$ days), which are growing in from their respective parents Te-131m ($t^{1/2} = 1.25$ days) and Ba-140 ($t^{1/2} = 12.79$ days), were determined ~24 hr after the column experiment. As with other irradiated solution components, I-131 and La-140 and their parent isotopes mainly partition in the eluent and acid-wash process streams.

TABLE 9.2 Percent Partitioning of Irradiated Target Solution Components Normalized

Isotope	Half-life (days)	Eluent (%)	1 M HNO ₃ Wash (%)	H ₂ O Wash (%)	Strip fraction 1 (%)	Strip fraction 2 (%)	Strip fraction 3 (%)	Strip fraction 4 (%)	Sorbent (%)	Mass Balance (%)
Ba-140	12.79	88.2	11.8	-	-	-	-	-	-	85.1
Ce-141	32.28	87.9	12.1	-	-	-	-	-	-	91.4
Ce-143	1.404	88.8	11.2	-	-	-	-	-	-	86.9
I-133	0.867083	89.0	10.1	0.0	0.0	0.4	0.5	-	-	87.3
I-135	0.274167	92.2	7.8	-	-	-	-	-	-	116.0
Mo-99	2.743056	0.7	-	-	0.2	28.3	70.1	0.8	-	91.3
Nd-147	11.06	82.4	17.5	-	-	0.2	-	-	-	106.3
Pm-151	1.18	88.2	11.8	-	-	-	-	-	-	91.3
Rh-105	1.473333	90.2	9.8	-	-	-	-	-	-	95.1
Ru-103	39.35	88.0	11.4	0.0	-	0.3	0.3	0.1	-	84.1
Sb-127	3.85	16.3	22.5	-	59.5	-	-	-	1.6	79.7
Sm-153	1.9285	89.2	10.7	0.0	0.0	-	-	-	-	94.4
Sr-91	0.400417	89.2	10.8	0.0	-	-	-	-	-	103.0
Te-132	3.246	79.5	11.9	0.0	0.4	0.3	0.3	0.5	7.1	12.8
U-237	6.75	86.2	13.8	-	-	-	-	-	-	69.4
Y-93	0.455417	100.0	0.0	-	-	-	-	-	-	110.0
Zr-95	64.4	98.1	-	-	0.3	0.2	0.1	-	1.4	91.8

9.1.3 Electrochemical Dissolution

A series of reports documenting our progress in developing the electrochemical dissolution method is available [GELIS-2009, JERDEN-2011, GELIS-2013, BROWN-2014A, BROWN-2014B]. A report was also written that estimates compositions and volumes of all waste streams generated from this front-end process [JERDEN-2011]. The process was tested for operability in an Argonne hot-cell-mockup facility before being demonstrated at ORNL using a target to be irradiated in HFIR [VANDEGRIFT-2013].

9.1.3.1 Process and Equipment Design

Caustic digestion of irradiated aluminum-clad HEU uranium-aluminide targets is employed for Mo-99 production [VANDEGRIFT-2002B]. However, under normal conditions, uranium metal is not digested in NaOH due to the formation of a passive uranium dioxide layer. Using alkali solutions for short-cooled processing of the irradiated target has several advantages over the more common acidic nitrate approach. The digestion of uranium in nitric acid produces substantial amounts of undesirable NO_x gases that demand some degree of off-gas treatment. The chemical speciation of iodine – a major fission product – exists as predominantly I₂ under acidic conditions [GOTTARDI-2000]. In alkali solutions, iodine is in the reduced anionic forms I⁻ or I₃⁻,

which are not volatile. The remainder of the fission products, such as zirconium, lanthanides, barium, and strontium, can be co-precipitated with uranium and calcium carbonate in basic solutions.

The electrochemical oxidation of uranium metal is assumed to proceed quickly through U(III), producing U(IV) oxide. Then, according to literature data, the oxidation of UO_2 proceeds through the formation of UO_{2+x} species on the surface until $\text{UO}_3 \cdot 2\text{H}_2\text{O}$ is formed [SUNDER-1996]. Hexavalent uranium has rather high solubility in the presence of carbonate/bicarbonate ions [MCLAREN-1950]. The dissolution occurs when these negative ions reach the positively charged surface of the uranium anode. A schematic of the basket and the dissolver is shown in Figure 9.3. The basket's vertical bolt pushes the outer frame with the welded screen toward the inner frame with the attached screen, thus providing the necessary contact between the anode lead and the foil. The authors have found from previous experiments that, using a basket, it takes about 2.5 hr to dissolve nearly 99% of the full-size foil due to the partial reduction of the free foil surface area, caused by the mesh interference [GELIS-2009]. The material used for the prototype is Type 304 stainless steel (SS). Nitrogen gas was used to sweep the dissolver. Fission gases will be collected and hydrogen gas, which is generated during the electrolysis, will need to be oxidized for commercial-scale processing, but both were diluted and released in this experiment.

FIGURE 9.3 Dissolver Tank and Electrochemical Basket

Figure 9.4 is a photograph of the entire apparatus in a vac-frame hood. After mechanical decladding of the irradiated foil, the next step is the dissolution of Al barrier in 1 M NaOH as NaAl(OH)_4 (Figure 9.1). This dissolution is performed in the electrochemical dissolver.

FIGURE 9.4 Full Prototype Process Setup Used for Low-Burnup LEU Experiment inside Vac-Frame Hood

Following dissolution of the cladding, the solution is drained through a ball valve on the side of the dissolver. After a wash step, a sodium bicarbonate solution is added in the dissolver through one of the ports on the lid. The uranium foil electrolysis is conducted at elevated temperature (85–95°C) with intense stirring. Heating is provided by an external heat gun (not

shown). The approximate volume of the solution required to cover a 24 g uranium foil ($10\text{ cm} \times 8\text{ cm}$) is about 1.2 L. However, the solution/U-metal ratio will be significantly smaller if a batch of several foils is dissolved in a vessel modified for the full-scale process. The solubility of uranium in bicarbonate medium is 80 g-U/L (it is soluble as both the neutral and anionic complexes of uranyl carbonate) [MCLAREN-1950]; thus for a 225 g batch of U foils, at least 3 L of the solution is required. For the full-size dissolver, the anode basket(s) will be fabricated from nickel or nickel alloys, which are resistant to oxidation in alkaline media. Use of 304 SS is acceptable for performing several experiments, so it was chosen to minimize the cost of the prototype. The cathode and the dissolver body of the production dissolver can be made from stainless steel, as there should be no significant corrosion during the processing. A $4 \times 4\text{ cm}$ SS plate, welded to a SS rod, served as a cathode.

The *U precipitation* (Figure 9.1) is the critical step to remove actinides and the majority of the fission products from the product solution. Another very important role of this step is to remove the carbonate from solution as it interferes with the anion exchange step for the molybdate recovery. The mixing vessel was built with 304 SS and designed to hold 2 L of solution. The base of the vessel was built with an incline to assist the flow of the uranium-carbonate slurry and to avoid clogging. Five ports were drilled into the head of the vessel: a funnel to introduce the calcium oxide powder, a mixing port to hold the stir shaft, and connections for the vacuum, solution entrance, and N_2 gas. The exit valve diameter was $\frac{1}{2}$ -in. (12.3 mm) to allow the slurry to move freely to the filter. An in-line 10- μm Whatman filter with $\frac{1}{2}$ -in. barbed connections was used to capture the slurry.

9.1.3.2 Process Demonstration with Irradiated Foil

A $7.6\text{ cm} \times 2.5\text{ cm}$ LEU foil (0.32-mm thick, 15.3 g) was obtained from laboratory stocks. The uranium foil was wrapped in aluminum foil and placed into an irradiation vessel that had been purged with helium. The vessel was then irradiated for ten minutes at the Argonne low energy accelerator facility (LINAC) using a neutron converter; the thermal neutron flux was of the order of $10^{11}\text{ n-cm}^{-2}\text{-s}^{-1}$. The target was allowed to cool for eight hours to minimize the dose rate from short-lived fission products.

The target was removed from the irradiation vessel, secured in the dissolver basket, and placed into the dissolver. The basket was then submerged in 1.5 L of 1 M NaOH under a continuous N_2 sweep gas with intense mixing at 70°C . It took approximately 30 min to completely dissolve the aluminum and leave the uranium metal exposed and in contact with the basket. The NaOH solution was drained from the dissolver into a collection flask for analysis, and the dissolver was filled with 1.2 L of 0.9 M NaHCO_3 . Anode and cathode leads were connected to the basket and the cathode, respectively. At a constant voltage of 9 V, the current was maintained at $41 \pm 2\text{ A}$ using a Magna Power supply. The temperature of the solution was $92 \pm 2^\circ\text{C}$. Nitrogen gas was continuously flushed through the dissolver and into a condensing coil and liquid trap. After four hours, the electrolysis was stopped, and the foil was examined. Over 98% of the uranium foil was dissolved in approximately 650 mL of carbonate solution (pH 10.0). An aliquot of the solution was taken for analysis. The solution was then fed into the mixing vessel, where it was contacted with 89 g of calcium oxide. The solution was mixed for

approximately 15 min and rinsed with de-ionized water to flush precipitates from the mixing vessel. The slurry then exited the vessel through the in-line Whatman filter and was weighed in a collection flask (1.16 L total, pH 13.0). The product solution was clear, indicating that most of the uranium had been removed. An aliquot of the product solution was taken for analysis. The uranium precipitate that was captured on the filter was eluted with concentrated nitric acid. A small volume of acidic 0.5 M stable-Mo carrier was added to the eluted solution with 2% alpha-benzoin-oxime (α BO) [WU-1995]. The precipitate was filtered, dried, and then digested in sodium hydroxide and hydrogen peroxide to a final volume of 0.215 L. An aliquot of this solution (labelled “Mo Separated Filter” in Table 9.3) was taken for analysis.

9.1.3.3 Results and Discussion

The samples were gamma-counted on a high-purity germanium well detector (calibrated with Eckert & Ziegler mixed isotope standard). The activities of each nuclide were decay-corrected to the time of irradiation. The dead time in each case was less than 5%. The Mo-99 concentrations were determined from the gamma peak at 778 keV. Table 9.3 lists the results of the analysis. The NaOH-aluminum solution showed only trace amounts of technetium and iodine. The activity from this solution was considered negligible in the recovery calculations. The dissolved LEU solution (listed as “Dissolver Solution” in Table 9.3) was heavily loaded with fission product and actinide gamma peaks; thus the results may not be strictly quantitative due to interference from the multitude gamma peaks in the spectra. There is also some uncertainty about the solution volume because of evaporation. Gamma analysis of this solution indicated approximately 32 μ Ci of Mo-99, assuming a final volume of 700 mL. The Mo-separated filter contained approximately 1.9 μ Ci Mo-99. This activity was determined by dissolution of the precipitate in acid, followed by the quantitative precipitation of Mo with α BO. This loss in product can be attributed to Mo-99 being trapped within the precipitate.

Gamma analysis of the product solution showed strong signals from technetium, molybdenum, iodine, and several xenon isotopes as a result of iodine decay. We assume the concentrations of Mo-99 in the product solution to be quantitative due to the chemical treatment and hindered gamma interference. The Mo-99 recovery was calculated from the sum of the Mo-99 captured on the filter (loss) and the product solution, which results in over 92% recovery ($\pm$ one-sigma notation from counting statistics). From Table 9.3 we can approximate that 30–50% of the iodine was recovered based on the I-133 and I-131 isotopes, though it was anticipated that more I-131 should be present. The U-237 tracer (induced by photon reactions with U-238) confirms that over 95% of the uranium was removed with the precipitation step and by the single in-line filter. This removal can be improved with additional filtration and Mo purification.

TABLE 9.3 Gamma Analysis Results of the Electrochemical Dissolver Process^a

Isotope	μCi			% Mo Recovered [†]
	Dissolver Solution*	Mo Separated Filter	Product Solution	
Mo-99	32.1	1.9	25.6	92 $\pm$ 3
U-237	16.7		0.7	
La-140	5.6		0.7	
Zr-95	1.4		0.1	
Nb-95	0.2		ND	
Nd-147	0.8		0.1	
Np-239	10.2		0.3	
Te-132	0.7		0.1	
I-133	32.3		11.1	
I-131	1.6		0.9	
Ru-103	0.3		< 0.1	
Rh-105	1.4		0.1	

* These values may not represent the true values on account of the uncertainty in solution volume and gamma-interference.

[†] Calculated from the sum of the Mo Separated Filter and the Product Solution

ND = below detection limit or complications with the peak shape.

Note: These activities have been decay-corrected to the time of irradiation.

To demonstrate the compatibility of this technology with back-end Mo purification procedures, an aliquot of the product solution was mixed with AG-MP-1(BioRad) anion exchange resin. Gamma analysis of the product solution afterward showed that technetium, molybdenum, and iodine were strongly retained on the resin.

9.1.4 Conclusion

Two front-end processes for Mo-99 recovery by dissolution of irradiated LEU foil were demonstrated at Argonne. Both processes are compatible with Mo-purification procedures. A full-scale prototype of the nitric-acid LEU-foil dissolver system at ambient pressure (capable of dissolving 250 g irradiated LEU) was designed and tested. Tests results showed that the cooling system of the dissolver (reflux condenser) is sufficient to remove all heat produced by the exothermic dissolution reaction and continuous decay heat of the irradiated foils. The uranyl-nitrate solution (895 g, ~350 g U/L) was loaded onto a S40 column (3.5 $\times$ 5 cm) at 25 mL/min and 60°C. The majority of Mo, 98.4%, was recovered in strip fractions 2 and 3 (product). Partitioning of actinides, lanthanides, and fission products between different process streams was determined.

The second process to separate Mo-99 from irradiated uranium targets electrochemically dissolved LEU foil in an alkali carbonate media. The actinides and the majority of the fission products were precipitated with calcium oxide, which left technetium, molybdenum, and iodine in solution. This process demonstrated over 92% Mo-99 recovery with appreciable separation from the actinides and fission products.

9.2 PLANNING FOR HOT-CELL DEMONSTRATION OF FRONT-END PROCESSES

9.2.1 Transportation of Irradiated Targets

In a production operation, the front-end processing would be performed in close proximity to the nuclear reactor to minimize the delay between the end of irradiation and chemical processing of the target. Ideally, targets would be moved within the same facility to reduce the transportation time to less than 12 hr, which reduces the loss of Mo-99 due to radioactive decay. At ORNL, the hot cells are not in the same facility as the nuclear reactor, and there is no mechanical connection between the hot-cell facility and HFIR. Irradiated targets removed from HFIR must be moved overland in a shielded shipping container to reach the closest hot-cell facility, a process that takes at least a half a day to complete. Fire and radiation safety limits affect shipping times too because of the overland route, and it was determined that the soonest an irradiated Mo-99 production-style target could be shipped is about two days after irradiation. This shipping time is adequate for a process development effort, but is too long for a commercial operation as the half-life of Mo-99 is approximately 66 hours.

Oak Ridge has three shipping casks that could be used to ship the Mo-99 targets—the Q-Ball Cask, the Sugarman Cask, and the LOOP Cask. The Q-Ball Cask is a large (25-ton) shielded shipping cask that is used at ORNL to ship irradiated targets between HFIR and the hot cells in Building 7920 (see Figure 9.5). It has top and bottom-loading capabilities. A crane, rigging crew, and a flatbed truck are required to move it.

FIGURE 9.5 Q-Ball Cask, Also Known as the Lawrence Radiation Laboratory (LRL) – 25T Cask

The Sugarman Cask is a smaller (1200 lb) cask that is used to ship rabbits and smaller irradiated targets to the hot cell facilities in Buildings 3525 and 4501. It is a top-loaded vessel (see Figure 9.6).

FIGURE 9.6 Sugarman Cask

The LOOP Cask, shown in Figure 9.7, is a steel-and-lead-lined (16,000 lb) transportation vessel that is used to ship irradiated targets and other materials on-site at ORNL. It has top- and bottom-loading capabilities.

The LOOP Cask was selected for use with the Mo-99 project because it can be loaded and unloaded from either end, it is smaller, and it weighs less than the Q-Ball cask. Bottom-loading capabilities are important because the Building 7920 hot cells, the hot cells closest to the nuclear reactor, only allow for bottom loading and unloading. The cylindrical cavity is lined with a 0.125-in. (0.318-cm) thick 304 stainless steel liner, which has an inside diameter of 5.75 in. (14.6 cm) and is 69 in. (175 cm) long. Physically, the cask is greatly oversized for use with the project's Mo-99 targets, but the cask was available, and qualification of the cask for transport of Mo-99 targets would allow it to be used to transport targets to both facilities.

Transport of the Mo-99 targets in the cask requires use of a custom target holder that slides into the larger cavity and holds the target tightly in place to prevent it from moving during transport. The target holder also provides a thermal conductive pathway to help remove decay heat from the target. Figure 9.8 shows a sketch of the LOOP cask target holder.

FIGURE 9.8 Drawing of LOOP Cask Target Holder (The cavity in the holder is large enough to hold two production-style targets or multiple rabbit targets)

Initially, the DOE Transportation Safety Document (TSD) for the LOOP cask did not include Mo-99 on the list of authorized materials that could be shipped. The list is changeable, and new items can be added to the list if the items are supported by favorable radiation and thermal safety analyses.

Using ORIGEN, we determined the radioactive content of the target designs as a function of irradiation time, decay time, and position in the nuclear reactor. The results were then compared to the radiation and material limits of the LOOP cask. The radiation emitted by an irradiated target of either design was well below the limits of the LOOP cask, and it was determined that the targets could be moved in the LOOP cask without causing excess exposure to workers or the environment.

Then, the decay heat of the targets was calculated to determine the temperature of the targets during transport (see Appendix C-2). The annular target contains more uranium than the

rabbit targets, so the cylindrical target was used as the bounding case in the calculations supporting the transportation document. The maximum thermal loading of the LOOP cask with the target holder is 24 W, as determined from prior analyses. The transport of a single target emitting 24 W of thermal power was analyzed to determine the steady-state temperature profile and the transient response during an unlikely fire event during cask transport. The analysis concluded that a single cylindrical target could be transported safely in the LOOP cask. In practice, the cylindrical targets would generate between 10 and 15 W of thermal power, so two cylindrical targets could not be transported in the LOOP cask without potentially exceeding the 24 W limit.

The rabbit targets contain less uranium than the cylindrical targets, so presumably more than one rabbit target could be transported in the LOOP cask. This scenario was not investigated because the rabbit target design was only planned to be used for one set of experiments, and it was assumed that the cylindrical target design bounded the rabbit target design. Applying the same quantity limit to the rabbit target as to the production-style target, the LOOP cask could be used to transport only one rabbit target at a time.

The facility safety officer for Building 3525 modified the TSD for the LOOP cask to include Mo-99 on the authorized transport list, and the DOE Oak Ridge Site Office approved the modification.

9.2.2 Selection of Hot Cell Testing Locations

At the start of the work, it was accepted that testing of the target dissolution systems would be performed at ORNL, which routinely performs hot-cell operations with irradiated materials from the HFIR, and has decades of experience performing experiments with radioactive solid and liquid materials.

Ideally, both target dissolution methods (acidic and electrochemical) would have been tested in the hot-cell facility located closest to HFIR, Building 7920. Unfortunately, hot cells in Building 7920 lacked 220 VAC power connections, which made it very difficult to operate the electrochemical dissolution equipment in that facility. Additional power lines would need to be installed to allow operation of the electrochemical equipment in that facility, and the expense of this work was beyond the allowable budget for the project.

Instead, testing of the electrochemical dissolution method was moved to another hot-cell facility at ORNL, Building 3525. The hot cells in Building 3525 are equipped with shielded plugs that allow additional cables and instrument lines to be run into the hot cell through the walls at the experimenter's discretion. The facility has 220 VAC power outlets outside of the hot cell, which could be used to power a large DC power supply, and the leads for the power supply could be fed through the shielded plug to drive the electrochemical dissolution equipment.

Building 3525's capacity to handle liquid waste is limited, and only the electrochemical dissolution testing could be performed there. Unlike Building 7920, Building 3525 lacks a radioactive liquid waste storage and treatment system, and all radioactive liquid waste must be

solidified before it can be removed from the hot cells for disposal. This requirement eliminated the possibility of testing both the acid and the electrochemical dissolution systems in Building 3525.

9.2.3 Radioactive Iodine as a Safety Driver

Prior to work in the hot-cell facilities, the safety aspects of the experiments needed to be analyzed to ensure that the planned work fell within the approved safety envelope. While nearly all aspects of the work fell within the standard operating envelope of the hot cell facilities, one item emerged as a driver in influencing the design of the test targets and the conduct of the experiments—radioactive iodine. When the uranium foil in the test targets is irradiated, radioactive iodine species are generated. Radioactive iodine is readily absorbed into the biosphere if it leaks into the environment, so limits on total iodine in the irradiated test targets were imposed. In Building 7920, the total amount of radioactive iodine in the test target was limited to no more than 25 Ci, and this limit caused the target designers to use depleted uranium in the rabbit target since enriched uranium would have caused excessive radioactive iodine levels in the test target. In Building 3525, a less conservative limit of about 50 Ci was imposed because the facility has fewer other sources of radioactive iodine, and this allowed for a greater uranium enrichment in the production-style targets.

Use of these iodine limits was contingent upon the application of additional iodine controls in the hot cells. Each test system had to be equipped with iodine capture traps, and each facility had to deploy activated-charcoal HEPA filters on the hot cell exhaust in order to prevent the release of iodine to the environment through the hot-cell exhaust stacks. For the acid dissolution system, a liquid NaOH trap was to be used to capture radioactive iodine from the equipment when uranium foils are dissolved. For the electrochemical dissolution system, a filter loaded with NUCON International Nusorb Kiteg II activated charcoal (4-in. long bed, 1-in. diameter) was installed to capture radioactive iodine. The safety engineer for Building 3525 imposed an additional requirement: the activated charcoal filter on the electrochemical equipment must be capable of capturing 99.9% of the radioactive iodine to prevent too much iodine build-up on the downstream activated-charcoal HEPA filters, which would have made them more difficult to remove and dispose after completion of the experimental work. No such separation requirement was imposed by the safety engineer for Building 7920, but the liquid NaOH iodine trap was still perceived as an essential component in the defense-in-depth approach for controlling radioactive iodine during the experiment.

9.2.4 Additional Safety Analyses

The acid dissolution system that was to be tested in Building 7920 required the development of a Research Safety Summary (RSS). Work performed in this facility is governed by the research and development policies of the ORNL's Nuclear Security and Isotope Technology Division (NSITD). Division policies dictate that all experimental work be performed under the guidance of RSSs, which address specific safety hazards and controls. The acid

dissolution system had unique safety aspects that were not covered under general RSSs, and a specific RSS had to be developed and approved for this work (see Appendix C-1).

The electrochemical system that was to be tested in Building 3525 did not require an RSS because work in that facility is governed by ORNL's Nonreactor Nuclear Facilities Division. Instead, the facility's Safety Basis Supplement (SBS) and Technical Safety Requirements (TSR) had to be revised to include Mo-99 processing as an allowed activity. The SBS (Appendix C-1) was approved by the DOE Oak Ridge Site Office on September 3, 2013.

9.2.5 Mock-up Testing at ORNL

Oak Ridge staff took part in the mock-up operations at Argonne [VANDEGRIFT-2013] and made suggestions for improving operations in the ORNL hot cells. The experimental systems were then shipped to ORNL and set up in cold (radiation-free) areas where they could be uncrated, assembled, and examined by facility personnel. At this time, Argonne and ORNL personnel worked together to refine the operating instructions for each piece of equipment and to make adjustments to the experimental equipment if changes were needed to make the equipment easier to operate in a hot cell.

The acid dissolution equipment needed some modification to fit it into the Building 7920 hot cell. The downstream Mo-99 extraction column and valve structure were attached to a vertical frame that could be rotated on a hinge, which would allow the extraction column to be pushed out of the way and against the cell wall when the acid dissolution vessel and gas capture tank were in use. With these changes, the equipment was authorized by the Building 7920 facility manager to go into the hot cell as soon as delivery of an irradiated target was imminent.

The electrochemical dissolution equipment also required some changes. An activated charcoal iodine filter was installed in the inlet line for the vacuum pump. The metal grid plate to which the electrochemical equipment was attached was replaced with a more open metal structure, which could more easily be disassembled and decontaminated after completion of the experimental work. A photograph of the electrochemical dissolution apparatus in the mock-up area of Building 3525 is shown in Figure 9.9.

FIGURE 9.9 Electrochemical Dissolution Apparatus in Mock-up Area (Old Grid Plate Shown)

9.2.6 Hot Cell Testing Cancelled

Hot cell testing of the equipment was cancelled after there were repeated delays in receiving and qualifying the manufactured rabbit and production-style target from Y-12. Although there could have been some utility in testing the equipment in the hot cells without dissolving irradiated targets, the equipment would have to be disposed after testing due to radioactive contamination from the hot cell environments. Instead, the test equipment was packed back into shipping crates and was shipped back to Argonne for additional cold testing and some limited hot tests with uranium foils that were irradiated by the Argonne linac.

The safety documentation developed to support the hot cell tests remains in place. If there is a desire to perform hot cell testing of the equipment in the future, the safety analyses are still available to support that work.

10 TECHNOLOGY READINESS OF THE LEU TARGET

10.1 INTRODUCTION

Technology Readiness Levels (TRLs) are used in private industry and in many government agencies to guide technology investment, development, and deployment. Examples of several TRL systems used in various applications are provided in [USDOE-2009, USDOD-2009]. The TRL definitions used in this report were tailored from [USDOE-2009B, USDOD-2009].

Both TRLs and Manufacturing Readiness Levels (MRLs) were first developed by the U.S. Department of Defense (DOD) and later adopted by the DOE. The DOE-published TRLs, and MRLs were modified by an IAEA-sponsored working-group so as to be directly applicable to LEU ⁹⁹Mo production, including the development of HDT technology. The definition of the TRLs and MRLs developed by the working group are published in an IAEA report [IAEA-2013].

The following sections define TRL and MRL, and discuss the generally accepted correspondence of TRLs and MRLs to a process. Also discussed are the consensus TRL rankings the R&D team gave the LEU-target fabrication, irradiation, and processing steps leading to providing a feed to current Mo-99 purification processes.

10.2 TECHNOLOGY READINESS LEVELS AND THEIR DEFINITIONS

10.2.1 Introduction

Industry-recognized product development methodologies are used to some extent in all major industries. A Technology Readiness Assessment (TRA) is used to ensure that the technology required to deploy a product to market is consistent with the product's business development model. These methodologies provide a common language to describe the extent to which the product design is complete. They also provide a guide for the designers of a manufacturing process to follow, allowing them to focus only on the tasks defined for a given development level. This prevents superfluous studies that do not contribute to the advancement of the product's technology. Furthermore, it provides assurance that the product's business development model is both practical and economically viable. The rationale for TRLs is that they provide a metric for measuring technical maturity and communicating readiness of the product for each phase of a project. In summary, a TRA is performed to establish and document the "maturity" of a specific technology.

Many studies performed in the U.S. for both private industry and the federal government have concluded that insertion of immature technologies and/or immature manufacturing systems increase risk and cost, and significantly decrease the success of manufacturing implementation.

For the purpose of this report, a TRA is performed by using established TRL requirements (i.e., milestones). The established TRL scale ranges from one (1) through nine (9) and is presented in Figure 10.1. Note the color scheme, which shows that TRLs 1 and 2 are considered research levels; TRLs 3-5 define development levels; and TRLs 6-9 define manufacturing/industrial-type activities. The TRLs progress from research-and-development activities to design validation in a production environment. TRLs 4-6 represent the bridge from scientific research to process design development.

The TRL assignment is a snapshot of where the maturity of the technology is at a given point in time. It should represent the highest level of TRL for which all exit criteria are satisfied.

There is an associated methodology that define MRLs. A Manufacturing Readiness Assessment (MRA) is performed in conjunction with a TRA to make a final determination of the overall readiness level of a production technology.

FIGURE 10.1 TRL Element Identification and Activity Type

10.2.2 TRL Definitions and Exit Criteria

TRL 1. Lowest level of technology readiness. Scientific research begins to be translated into a concept for a technology.

Exit Criteria. A fundamental concept, innovation, or scientific principle has been identified.

TRL 2. Practical applications are beginning to be identified or developed. There is no proof or detailed analysis to support assumptions. The step up from TRL 1 to TRL 2 moves the concept from pure to applied research. Experimental work is designed to corroborate the basic scientific observations made during TRL 1 work. The filing of a patent is under consideration.

Exit Criteria. Potential practical applications for this research and/or innovation are identified. A research and development (R&D) plan has been developed.

TRL 3. Active research and development (experiments, simulations) is initiated to validate key elements of the technology. The results of this work should constitute “proof-of-concept” validation of the applications/concepts formulated at TRL 2. Examples include the study of separate elements of the technology that are not yet integrated or representative. At TRL 3 the work has moved beyond the paper phase to experimental work that verifies that the concept works as anticipated. Components of the technology are validated, but there is no attempt to integrate the components into a complete system. Modeling and simulation may be used to complement physical experiments.

Exit Criteria. Key elements of the technology have been identified and proof-of-concept has been demonstrated for each. Individual elements are not necessarily integrated at this technology readiness level.

TRL 4. The key elements of the technology are integrated (i.e., elements will function together) in the laboratory. TRL 4 is the first step in determining whether the individual components will work together as a system.

Exit Criteria. 1) A laboratory validation establishes that key elements function together, 2) validation in a laboratory environment can be made with mock material, and 3) performance metrics for the laboratory prototype have been defined and met.

TRL 5. Key elements of the technology are integrated with applicable supporting elements so that the system can be tested in a simulated or somewhat realistic environment. The major difference between TRL 4 and 5 is the increase in the fidelity of the system and environment to the actual application. The system tested is almost prototypical.

Exit Criteria. 1) Validation in a simulated or somewhat realistic environment establishes that the key elements function together, and 2) performance metrics have been defined and met.

TRL 6. A system or subsystem prototype is demonstrated in a high-fidelity, simulated, or actual environment. This represents a major step in a technology's demonstrated readiness. TRL 6 begins true engineering development of the technology as an operational system. The major difference between TRL 5 and 6 is the step up from laboratory scale to engineering scale and the determination of scaling factors that will enable design of the operating system.

Exit Criteria. 1) A prototype has been created and successfully demonstrated in a high-fidelity, simulated, or actual environment, and 2) the metrics for successful performance in an actual environment have been refined, met, and documented.

TRL 7. An actual system prototype is demonstrated in an operational environment. In almost all cases, TRL 7 coincides with the end of development.

Exit Criteria. 1) An actual system prototype has been demonstrated in an operational environment, and 2) the metrics for successful performance in operational environments have been refined, met, and documented.

TRL 8. The actual system has been proven to work in its final form under expected conditions based on certification and qualification activities to validate that it meets design requirements, product definition, and quality requirements.

Exit Criteria. A manufacture system has been certified/qualified.

TRL 9. Application of the technology is in its final form. The system is fully developed and is now operational. TRL 9 does not include ongoing or planned product improvement, or scale-up to larger production volumes.

10.3 MANUFACTURING READINESS LEVELS AND THEIR DEFINITIONS

10.3.1 Introduction

There is an associated methodology that defines manufacturing readiness levels [MANKINS-1995]. An MRA and MRLs are designed to manage manufacturing risk in conjunction with the development of new technologies. The MRL definitions create a measurement scale and vocabulary for assessing and discussing manufacturing maturity and risk. Using the MRL definitions, an assessment of manufacturing readiness is a structured evaluation of a technology's manufacturing process. An MRA is performed to 1) define the current level of manufacturing maturity, 2) identify maturity shortfalls and risks, and 3) provide the basis for manufacturing risk management.

For the purpose of this document, an MRA is performed with established MRL requirements or milestones. The established MRL scale ranges from one (1) through nine (9) and is presented in Figure 10.2. The MRL assignment is a snapshot of where the maturity of the manufacturing system is at a given point of time. It should represent the highest level of MRL for which all the exit criteria are satisfied. The MRLs range from a "Manufacturing Feasibility Assessment" to the "Repeatable Quality Production on a Routine Basis" The MRL definitions are more stringent at the higher levels, reflecting the increased complexity of the manufacturing requirements during the scale-up of production level.

There are nine risk areas that should be assessed as the manufacturing process matures. The risk areas are consistent for all MRLs, but the assessment questions of the risk areas change as a more sophisticated manufacturing capability, and higher MRL ranking, are achieved. In general terms, the risk areas are:

1. Technology & Customer Base
2. Design
3. Materials
4. Cost & Funding
5. Process Capability and Control
6. Quality Management (**Note:** This risk area is applicable to MRL 5 through MRL 8.)
7. Manufacturing Personnel
8. Facilities
9. Manufacturing Planning, Scheduling, and Control

FIGURE 10.2 MRL Element Identification and Activity Type

A description of the MRLs has been included to help the reader understand the relationship between TRLs and MRLs. A more comprehensive explanation of MRLs 1-9 is presented below.

10.3.2 MRL Definitions and Exit Criteria

MRL 1. A multidisciplinary team identifies a conceptual design; process; materials; and manufacturing options, needs, and risks.

Exit Criteria. 1) A rough estimate of production quantities is defined, 2) production capacity/capability evaluations are initiated, 3) production strategy is formulated, and 4) initial risks (product quality, business, safety, and security) are identified and documented.

MRL 2. Manufacturing options, gaps, and risks are identified and evaluated. Preliminary cost estimates are prepared.

Exit Criteria. 1) Qualification strategy is developed, 2) manufacturing technology options are identified and documented, 3) facilities and equipment gaps/budget needs are documented, 4) capacity/capability assessments are updated, 5) rough order of magnitude cost estimates are available, 6) manufacturing options are identified and assessed, 7) production strategy is updated (including waste stream disposition paths and material reprocessing concepts identified), and 8) risks are updated.

MRL 3. Component and subsystem conceptual design review is completed. Initial manufacturing options are down-selected. Manufacturing development needs and strategies are identified.

Exit Criteria. 1) Manufacturing technology down-select decisions are documented, 2) conceptual design review is completed (components and subsystems) and verified to meet customer requirements, 3) draft qualification plan is documented, 4) capacity/capability evaluations are updated, 5) high-level process maps are completed, and 6) product quality determination strategy is formulated.

MRL 4. Initiating manufacturing system capability is developed, and production component/subsystem proof-of-concept is demonstrated by bench-top testing.

Exit Criteria. 1) Development/draft product definition is prepared (drawings, models, product specification, etc.), 2) critical process parameters are identified and documented, 3) training requirements are identified, 4) facility and equipment upgrades are implemented, 5) key process areas are identified, and 6) production strategy is finalized and risk assessment is updated.

MRL 5. Development builds successfully meet requirements, and the prototype design review is complete.

Exit Criteria. 1) Final design review is complete, 2) a prototype build successfully meets definition requirements, 3) detailed qualification plan is documented, 4) initial producibility assessment is completed, 5) draft product specification review is completed, and 6) process engineering specifications are documented.

MRL 6. Materials, process hardware, personnel skills, and quality control test equipment are demonstrated and approved. Formal procedures are completed to ensure quality.

Exit Criteria. 1) Equipment calibration, preventive maintenance, and house-keeping procedures are completed, 2) product is determined to meet quality requirements using expected production processes by manufacturing personnel, 3) equipment is qualified for production use by validation protocols, 4) facilities and equipment upgrades are completed, and 5) finished product packaging requirements are established.

MRL 7. Successful producibility, readiness review, and production proof-run are achieved. All suppliers/materials are available and qualified.

Exit Criteria. 1) Training/qualification activities are complete for processing technician and quality control personnel, 2) vendor qualification program is documented and implemented, and 3) production “batch” record is approved by the Quality Control Unit.

MRL 8. Manufacturing process is qualified, and initial production metrics are achieved. The First Production Unit is delivered to the customer.

Exit Criteria. 1) The First Production Unit meets the customer’s quality control receipt inspection criteria.

MRL 9. The ability to consistently meet cost and product quality goals and customer’s delivery schedule is demonstrated.

Exit Criteria. Not applicable. MRL 9 does not include ongoing or planned manufacturing improvement, or scale-up to larger production volumes.

10.4 SYNCHRONIZATION OF TRLs AND MRLs

The MRLs synchronize with the TRLs in a manner illustrated in Figure 10.3. It can be seen that during the first three (3) technology development levels, limited manufacturing capability development takes place. At TRL 4, manufacturing capability development must commence in order to move the level of technology development forward.

TRL	1	2	3	4	5		6			7	8	9
MRL				1	2	3	4	5	6	7	8	9

FIGURE 10.3 Synchronization of TRLs and MRLs

10.5 TRLs AT PROJECT COMPLETION

At the completion of this program, the R&D team met to reach a consensus on the R&D levels for target manufacturing, irradiation performance, and target front-end processing. That consensus is recorded below. A discussion of how the TRL was assessed follows each category.

10.5.1 Target Manufacturing TRLs

Target manufacturing TRL/MRL levels are broken down into the categories below.

10.5.2 Foil Manufacturing

10.5.2.1 Foil Production Technique

- **Roll Casting (TRL 4/MRL 2)**
Roll casting development is led by KAERI and is characterized by rapid foil production and noticeable surface texture on one of the two foil faces. The technology has been demonstrated and refined to reduce the surface and thickness inconsistencies. The TRL level 4 was determined because the technologies used are integrated and have been used to demonstrate several prototypes. TRL 5 will be achieved when the system is scaled and validated in a relevant environment and meets key performance objectives. MRL level 2 was determined because the manufacturing options have been analyzed. MRL level 3 will be reached when a requirements-driven manufacturing method has been determined, and a qualification plan has been drafted.
- **Cold Rolling (TRL 6/MRL 4)**
Cold rolling is used as a production method for numerous uranium alloys, and is usually preceded by hot forming or hot rolling to reduce the material in size. Cold rolling of thin LEU metal foils is at TRL 6 because the system has been demonstrated in a relevant environment with applicable materials and throughputs. MRL 4 has been achieved as several thin LEU metal foils have been successfully rolled to the required thickness.
 - **Beta Quench (TRL 7/MRL 7)**
Beta-quench/alpha anneal methods for grain refinement have been utilized for a variety of applications over the past 50 years. This technology is at TRL 9 for most cases, and is reduced to a TRL 6 due to the need for full qualification for this application.
 - **Cross Roll (TRL 7/MRL 7)**
Cross rolling is an experimental technique that may assist in preventing a uniaxial preferred grain orientation that occurs when foils are rolled. It is

expected to assist in reducing the swelling effect, but has not been demonstrated in a reactor. The effect of the cross rolling in mitigating swelling effects has had minimal study.

10.5.2.2 Quality Control Procedure

- **Dimensional inspection (TRL 6/MRL 5)**
Dimensional inspection techniques within the HDT team are at a stand-alone TRL of 8 and 9, depending on the techniques and throughputs. The TRL of 6 is chosen because the optimal suite of dimensional inspection techniques will be determined by the experiments conducted in the reactor.
- **Sampling (TRL 9/MRL 9)**
This technique is considered to be at TRL 9 for all applications, and is utilized on a daily basis to determine the chemical and isotopic properties of the materials produced.
- **Grain validation (Micro/XRD) (TRL 8/MRL NA)**
X-ray diffraction and microscopy are used extensively on a daily basis for sample analysis and regularly for production verification. TRL 9 will be achieved when the techniques are used regularly for this application.

10.5.2.3 Recoil Barrier Technology

- **Foil barrier wrap (TRL 7/MRL 7)**
Foil-barrier wrap techniques involve wrapping the U foil with a layer of Al or Ni foil to prevent fission recoil products from fixing the foil to the cladding. This technique has been demonstrated on targets irradiated in reactors many times.
- **E-plating (TRL 6/MRL 4)**
Electroplating Ni onto uranium is at a TRL 8 in the DOE complex, and was used for decades to coat and protect some uranium products.
- **Aluminum PVD (TRL 2/MRL 3)**
The physical vapor deposition (PVD) system (“ion plater”) consists of a vacuum chamber into which the workpiece (item to be plated) and a target disk of the plating material are loaded. The chamber is evacuated and backfilled with inert gas (typically argon, neon, etc.) to the desired operating pressure. Processing voltages are applied to the workpiece and target. The target is bombarded with ionized (electrically charged) argon, which removes atoms from the target. These atoms are attracted to the workpiece and are deposited on its surface. The PVD system at the Y-12 Development Facility has been used in the past on various shapes and coating thicknesses. There

were no specific development activities related to foil coating. Past processing knowledge was used to PVD the foils. For an aluminum fission-recoil barrier, the PVD layer needs to be 20- μm thick. Attempts to deposit this thick a layer failed; any layer over 10 μm flaked off the uranium foil.

10.5.2.4 Target Assembly

- **Tube Machining (TRL 9/MRL 8)**
This is an industry standard, and MRL will be at TRL 9 when the technique is regularly used for the end customer.
- **Assembly Technique**
 - **Drawing (TRL 7/MRL 6)**
Drawing the tubes together to sandwich the foil is an extensively used technique for the targets that have been tested. This technique has evolved and been analyzed to perfect the final product and reduce defects. This technique does impart a residual stress on the target, which has some positive side effects. This technique is at TRL 7 because of the extensive testing and actual demonstration of the system in a relevant environment. TRL 8 will be achieved when a final product design is determined and shown to work in a relevant quality controlled setup.
 - **Hydroforming (TRL 3)**
Hydroforming is a common industrially used process, which is at a TRL 9 even for nuclear applications. For this application it is at TRL 3, and will reach TRL 4 when the system has been integrated and tested to make surrogate targets.

10.5.2.5 Welding

- **E-beam (TRL 6/MRL 5)**
E-beam welding is commonly used for many nuclear applications, and is at a TRL 5-9 depending on the application. Targets irradiated in Indonesia were beam welded at Argonne.
- **Tungsten Inert Gas (TIG) (TRL 6/MRL 5)**
TIG welding is used as a less expensive weld than E-beam welding. It also allows for an inert gas to be put into the target, which may be beneficial for heat transfer and leak testing. Targets irradiated in Indonesia were TIG welded at Argonne and in Indonesia, Argonne TIG-welded targets were also irradiated in Argentina, and Australian TIG-welded targets were irradiated in Australia. TIG is at a TRL 9 for most industry applications. TRL 6 is chosen because it has not been demonstrated for large production runs of targets.

- **Deformation Forming (TRL 2/MRL 1)**
Deformation forming is common in industry (TRL 9), and essentially would weld the tubes by a swaging/canning process similar to what is used to make soda/coke/pop cans. This technique is at early stages of development, and remains to be demonstrated for this application.

10.5.2.6 Quality Control

- **X-ray (TRL 7/MRL 6)**
Quality control TRLs are difficult to analyze in the technology readiness level frame of reference. X-ray of targets can provide useful data, but the annular geometry does present some challenges.
- **Resonant Ultrasound Spectroscopy (TRL 3/MRL 1)**
- **Thermal Contact Resistance (LFA – diffusivity) (TRL 2/MRL 1)**
For other geometries up to TRL4 has been achieved.
- **Helium Leak Testing (TRL 6/MRL 6)**
This technique is used routinely for testing research reactor plates following fabrication and has been used at Argonne for annular targets.

10.5.2.7 Target Performance

- **Thermal-Hydraulic Mechanical Analysis (TRL 6)**
Thermal-hydraulic mechanical analysis is at a TRL level 9 for industry, and is considered to be at TRL 6 for this project.
- **Irradiation Performance (TRL5/6)**
TRLs are difficult to assign to the sub parts of the target, but the successful irradiation history of the previous targets in relevant sub-environments defines a TRL5/6 assignment.
- **Fission-Gas Collection**
Post-irradiation examination techniques for this application are at TRL 5, and sub technologies range from TRL 3 to TRL 9 for hot cell operations.

10.5.2.8 Target Disassembly

- **Manual (TRL 7/MRL 6)**
Manual disassembly methods have been demonstrated and refined to a TRL 7 due to the previous target demonstrations.

- **Semi-automated (TRL 7/MRL 5)**
Semi-automated disassembly techniques are at TRL level 6 for this project.

10.5.2.9 Front-end Processing

- **Acid Process**
 - **Acid Dissolution**
 - The chemistry of the dissolution is well understood and practiced (TRL 9).
 - The equipment has been tested at lab-scale with low-burnup foils and in a hot-cell mock-up but not tested in a hot cell or with fully irradiated foils (TRL 3-4).
 - **Mo Recovery**
 - The chemistry is well characterized, but not demonstrated with fully irradiated foils (TRL 3-4).
 - The equipment has been demonstrated at full scale but not in a hot cell or with fully irradiated foils (TRL 2-3).
- **Electrochemical Process**
The chemistry is fairly well understood but not optimized, and the equipment is not full scale and not optimized (TRL 3).

11 PROJECT TIMELINE

1995

- Development of the differential thermal-expansion LEU-foil target

1999

- Development of the annular LEU foil target using two drawn Al tubes

1999-2009

- Irradiation of annular LEU-foil targets in Indonesia, Argentina, and Australia

2009

- MURR and ANL demonstrate the high-density annular target technology in a low power test and subsequent dissolution.

2010

- Collaboration with INR (Romania) begins.

2011

- Test plans are developed to utilize INR's TRIGA reactor and hot cells for full-scale target demonstration and test matrix.

2012

- Project is redirected to focus on a reduced test matrix to accelerate schedule.
- ORNL is brought on board as a partner to utilize the Radiochemical Engineering Development Center (REDC) hot cells and HFIR reactor.
- ANL begins scale up to production-scale dissolvers.

2013

- Dissolution equipment is designed and fabricated by ANL to function in the REDC hot cells at ORNL.
- ANL equipment is sent to ORNL for test runs.
- Specification for targets for ORNL tests are derived from ORNL modeling of the previously tested targets.
- Y-12 begins fabrication, demonstration, and testing and procedure writing for target fabrication.
- Argonne rolls LEU foils for use at MURR and HFIR.

- Due to welding issues, the 2013 produced targets are delayed for the ORNL tests.

2014

- ANL conducts “warm” tests with DU targets.
- MU irradiates two targets and performs post-irradiation disassembly and fission gas collection testing.
- The NU “capsule” type target is complete and stored for later use by the project.
- The HDT project is completed and closeout documentation is compiled.

12 PROJECT CONCLUSIONS

The HDT Project successfully demonstrated several iterations of an LEU-fission-based Mo-99 technology that has the potential to provide the world's supply of Mo-99, should major producers choose to utilize the technology.

Over 50 annular high density targets have been successfully tested, and the assembly and disassembly of targets have been improved and optimized.

Two target front-end processes (acidic and electrochemical) have been scaled up and demonstrated to allow for the HDT technology to mate up to the existing producer technology for target processing. In the event that a new target processing line is started, the chemical processing of the targets is greatly simplified.

Extensive modeling and safety analysis of the HDT target type have been conducted, and the target has been qualified to be inserted into the HFIR reactor, which is considered above and beyond the requirements for the typical use of this target due to high fluence and irradiation duration.

Producers of Mo-99 have interests and considerations that this project attempted to address where applicable. It is understood that Mo-99 producers have many factors to consider when deciding an LEU-target implementation plan, and thus the HDT Project technology is made available freely to producers of Mo-99 for independent analysis and determination of feasibility.

13 RECOMMENDED NEXT STEPS FOR USERS OF THE TECHNOLOGY

- Due to a variety of unforeseen circumstances, this program failed to irradiate annular LEU-foil targets at a high power density for a prolonged period. It was, therefore, not possible to process high-burnup targets using the two front-end processes developed for allowing use of foil targets in the purification schemes used by current producers. Both of these items must be successfully completed to fully demonstrate the processes.
- A safety analysis must be performed for irradiating LEU-foil targets in each reactor utilized by the producer considering the use of this target.
- A firm cost for producing foils and/or targets must be obtained from a commercial entity with proven ability to fabricate and deliver hundreds to thousands of foils and/or targets per year.
- An economic analysis must be performed based on user-specific information to establish the efficacy of using this target.
- The geometry of an annular target must be designed that will work in all reactors that irradiate targets for a given producers. If the annular LEU foil is the same geometry as the IRE current HEU dispersion target, this may be a simple task.
- Radiation rigs must be designed and tested for irradiating the annular LEU-foil targets. If the annular LEU foil is the same geometry as the IRE current HEU dispersion target, this may be a simple task.
- A safety analysis must be performed to assess using one of the front-end processes developed (acidic or electrochemical) in the producers' hot cell facility. Possible modifications to the hot-cell processing line to accommodate the front-end processing must be assessed. Design changes to the experimental designs of front-end equipment will be required to meet local infrastructural and regulatory requirements.
- Hot pilot demonstrations of the combined process with initial low-burnup and then high-burnup targets must be performed to assess scale-up issues and assure Mo-99 product yield and purity.
- The current target-transportation casks and procedures must be modified. If the targets are the same geometry of the current IRE HEU targets, this could be a simpler process.
- Analysis of changes that would need to be made to current regulatory documentation and approvals should be performed.

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APPENDIX A:
ARGONNE NATIONAL LABORATORY

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A-1 ARGONNE U-FOIL ROLLING PROCEDURE

Revision No.: _____
Revision Date: _____
Written By: _____
Approved By: _____

RERTR ⁹⁹Mo URANIUM FOIL ROLLING

Shearing and Machining of COVERS and FRAMES

I. Purpose

Rolling of a Clad Uranium INGOT into foil must be done in isolation from air to avoid the buildup of oxide. In order to do that, the ingot will be rolled inside of low carbon steel cladding for pure uranium or 304 Stainless Steel cladding for adjusted uranium. The cladding is an assembly made up of one FRAME and two COVERS that will be welded to avoid any air contact. The carbon steel stock is purchased in several thicknesses and is sheared into pieces suitable for the machining operation. The following procedure describes the shearing and machining procedure developed at Argonne National Laboratory (ANL) to produce the COVER plates and FRAMES.

II Materials and Equipment:

Latex gloves
Safety Glasses
Approved radiation safety clothing
3 – 6 mm thick low carbon steel plate stock
3 – 6 mm thick stainless steel plate stock
Shear with 6 mm low carbon steel capacity
Milling machine

III Procedure:

1. Pick a uranium ingot to be rolled and measure its dimensions and mass. Write them down in a log book or recording sheet.
2. Pick a carbon-steel stock if pure uranium is being rolled or pick a stainless steel stock if adjusted uranium is being rolled that has the thickness of the uranium INGOT to be rolled, expected to be between 3 and 6 mm. The thickness of the FRAME should be slightly (0.02-0.1 mm) larger than the ingot thickness. The COVER plate thickness is the same as the FRAME thickness.
3. Shear three pieces of low-carbon or stainless steel plate stock to 13 cm x 13 cm. squares.

4. All three pieces are machined into 12.7 cm x 12.7 cm. Two of these pieces are the COVER plates depicted in Figure 1 (pieces to the left and to the right). The edges are machined to 45° across half the thickness on one side to facilitate a stronger weld.
5. The third piece will be the FRAME, and it is produced by machining a cavity into the center of the remaining piece (Figure 1, piece in the center). This hole is slightly (0.05 mm) larger than the uranium ingot dimensions (see ingot dimensions in log book or on recording sheet). The edges are machined to 45° across half the thickness on both sides to facilitate a stronger weld
6. Specifications for the frames and cover plates are shown in the attached figures (ANL drawings 99MoTCW.Frame and 99MoTCW.Cover). The thickness dimensions are not indicated in these drawings. These dimensions will vary according to the thickness of the ingot to be rolled. The plate thickness range will typically be from 3 to 6 mm.

Figure 1. Stainless-Steel COVERS and FRAME for Uranium Rolling

Uranium Cladding

I. Purpose

Uranium must be protected from oxidation during hot rolling, and methods commonly used are (1) canning and (2) coating with a molten salt. The following procedure describes the canning procedure developed at ANL to protect uranium during the hot rolling operation.

- II. Materials and Equipment:
- Latex gloves
 - Approved radiation safety clothing
 - Yttrium-oxide powder
 - Two low-carbon or stainless-steel cover plates
 - One low-carbon or stainless steel frame
 - Two Copper chill blocks (10.2 x 10.2 x 0.6 cm)
 - Four C-clamps (10.2 cm size)
 - 300-amp AC/DC welder
 - Ethyl alcohol
 - Abrasive cleaning equipment
 - Arc-melted Low Enriched Uranium (LEU) ingot
- III. Procedure:
1. Put on required radiation safety clothing (latex or equivalent gloves and lab coat).
 2. Prepare an yttrium-oxide/alcohol slurry having the consistency of a thin paste.
 3. The arc-melted LEU ingots are stored in a vacuum or inert atmosphere container until needed for assembly. An LEU ingot of 100–300g mass is removed from vacuum storage. Coat the ingot with the yttrium-oxide/alcohol slurry and allow it to air dry.
 4. Check the two steel cover plates for oxide. If oxidation is significant, abrasively clean until shiny.
 5. Choose a frame with the appropriate thickness. The frame thickness is slightly greater (0.02-0.1 mm) than the ingot thickness. A complete assembly requires three pieces--a top and bottom COVER plate and a FRAME which sits between the cover plates.
 6. The assembly area should be covered with plastic, and paper towels may be used as the primary barrier for any loose contamination.
 7. Place one copper chill block on a cube (3–5 cm on a side) of aluminum. The purpose of the cube is to raise the assembly so that the C-clamps can be attached without lifting the assembly.
 8. Place the bottom coverplate on top of chill block. The chill block should be centered such that the overhang of the bottom cover plate is equal on all four sides.

9. Place frame on top of bottom coverplate.
10. Coat the cavity in the frame and the matching surface of the bottom cover plate with the yttrium-oxide slurry and allow to air dry.
11. The coated ingot is then placed inside the carbon steel frame. Previous ingots were smeared and showed no loose activity (before slurry coating). Even so, all the loading is done on a plastic and paper covered surface.
12. Coat the inside surface of the top cover plate with the yttrium-oxide slurry and allow to air dry. Do not coat within 2 cm of the edges.
13. Place the top cover plate over the ingot.
14. Place the second copper chill block on top of the top coverplate and make sure it is properly aligned. The chill block should be centered such that the overhang of the top cover plate is equal on all four sides.
15. C-clamps are placed and tightened on each end directly on the chill blocks (four total). The assembly is now ready for welding.
16. The steel plates are tack welded on opposite sides, and then a light (50–100 amp) weld is run around the entire assembly. This will prevent oxidation of the uranium during the final welding pass. Welding filler rod is optional, depending on the quality of the steel.
17. A heavy (200–300 amp) weld is then run around the assembly.
18. The weld should be examined for surface cracks and gas holes. Any flaws detected should be cleaned and rewelded.
19. When a good weld is produced, the copper chills are removed and the assembly is allowed to air cool. Figure 2 shows the assembly after welding.

Figure 2. Uranium Ingot assembled inside the SS frame after welding.

20. The assembly is wrapped in stainless-steel heat-treating foil and heated at 625°C for 24 h to anneal the cast structure of the uranium ingot.
21. After air cooling and removal of the heat-treating foil, the assembly is ready for hot rolling.

Uranium Hot Rolling

I. Purpose

The basic rolling mill unit, or stand, consists of rotating rolls mounted in a framework. They are used to reduce the cross-sectional area of metal stock and to produce certain desired shapes. Metals may be hot or cold rolled, depending the specific material to be rolled. The final product may be flats, rounds, squares, or other structural shapes. The following procedure describes the method developed at ANL to produced uranium foil by hot rolling a clad uranium assembly.

II. Materials and Equipment:

- Stanat two-high rolling mill with (22.9 cm. dia. x 40.6 cm width rolls.)
- Welded LEU assembly
- Ethyl alcohol
- 50% nitric/50% water solution
- Abrasive paper
- Acetone or equivalent solvent
- 300-amp AC/DC welder
- Two sets of tongs
- Box furnace ~30 x 30 x 100 cm
- Cloth or paper towels

Personal Protective Equipment

III. Procedure

1. Examine the mill rolls for adhering foreign material before each rolling operation. If necessary, clean the rolls with a solvent such as acetone and a cloth or paper towel. For very heavy deposits, 600-grit abrasive paper may be used. Figure 3 shows the rolling mill used to roll the LEU assembly.
2. Set the furnace at 625°C and allow temperature to stabilize. Handling the welded LEU assembly with suitable tongs, load it into the furnace and pre-heat at 625°C for a one hour. Figure 4 shows the furnace used to heat treat the assembly.
3. Check that the rolls are parallel and adjust if necessary. The rolls are rough set by lowering the rolls until the minimum amount of light will pass across the entire width. The rolls are adjusted to give a uniform gap judged by eye. The rolls are then raised to the first pass setting. Two test pieces 2-3 cm x 10-15 cm x 5-10% thicker than the first pass setting cut from the same stock are rolled simultaneously 15-20 cm apart and measured for thickness. The rolls should be adjusted until the difference in thickness between the pieces is 0- 0.005mm. The rolls are now parallel. If during rolling the piece begins to curve or one side becomes longer than the other the rolls should be immediately checked for parallelism.

Figure 3. Rolling Mill to roll LEU

4. The first few (1-5) passes may be limited to 5% or less reduction per pass, depending upon the roll size. The assembly should be perpendicular to the rolls when it enters the mill. The initial thickness of the ANL assembly is typically 2 cm. The assembly is rotated 180° along the rolling axis after every pass (assembly is turned top to bottom such that the front of the assembly on the first pass is always the first to enter the rolls for all passes). A table on the inlet side to aid with piece alignment has worked well for ANL. In addition, a second person to catch the assembly on the exit side of the mill (with or without the table) reduces the amount of time per pass, thereby minimizing cooling of the assembly. Figure 5 shows the assembly going into the rolls.
5. After each pass, reheat the assembly for 10 min.
6. Gradually increase the reduction per pass to 10%, with the rate of increase being determined by the ease with which the assembly is rolled.
7. Twenty passes with reduction to 3 mm were completed before the can (assembly) became too long to fit in the ANL furnace, Figure 6. Allow can to air cool.

Figure 4. Furnace to heat LEU assembly

Figure 5. LEU assembly going into rolling mill

Figure 6. Rolled LEU assembly

8. To reduce the size on the can so that it will fit inside the furnace for reheating, the excess canning material at the front and back of the can is cut off to within 3 cm of the foil. The can is then sealed by welding the cut surfaces at the front and back shut.
9. After a 20 min. reheat, the assembly is hot rolled for 10 passes with an average percent reduction of 10%.
10. After the last pass, the assembly is annealed for 30 minute at 625°C.
11. The welded material is cut off of all four sides of the can. The can is then placed on a plastic sheet and disassembled. If the weld seam is not completely cut off, it may be necessary to pry the can open with a screwdriver.
12. The resulting foil target thickness should be 0.5 – 0.75 mm.
13. The foil is then etched in a 50% concentrated nitric acid/50% water solution to remove any oxide. Typically etching times are 2 - 10 min. After an acceptable etch, the metal will have a dull shine.
14. The foil then is cut into square or rectangular pieces of suitable size for cold rolling, depending on the method (bare or open can rolling) to be used.
15. Any gross surface defects should be removed by wet sanding with abrasive paper and ethyl alcohol. Extra attention should be given to the surface

condition of the foil, since surface defects and uneven thickness material may lead to pinholes or tears during subsequent cold rolling.

16. The material is now ready for cold rolling.

Uranium Cold Rolling

I. Purpose

Uranium sheet < 0.5-mm thick that has been hot rolled from pure or adjusted uranium has sufficient ductility to be cold rolled to foil (0.125 mm or less). Cold rolling also generally improves the surface finish of the material and increases its stiffness, making it easier to shear. The following procedure describes the method developed at ANL to cold roll uranium.

II. Materials and Equipment:

Stanat two-high rolling mill (12.7 cm. dia. x 20.8 cm width rolls.)
LEU foil 0.5 – 0.75 mm thick
Ethyl alcohol
0.38 –0.75 mm 304 stainless steel sheet
Acetone or equivalent solvent
Abrasive paper
Two sets of tongs
Box furnace ~30 x 30 x 100 cm
Bending brake

III. Procedure:

1. Cut the stainless-steel sheet into suitable size pieces, based upon the size of the uranium pieces to be cold rolled. To determine the required width of the stainless steel sheet, add a minimum of 6 cm to the width of the hot rolled uranium foil. The required length of the stainless steel sheet is two times the length of the hot rolled uranium foil plus a minimum of 6 cm.
2. Degrease the stainless-steel sheet and abrasively clean one side.
3. Using a bending brake, fold the stainless steel sheet at the midpoint of its length, with the cleaned surface on the inside. The resulting can should accept the uranium foil and have a minimum of 3 cm of excess material on the width and the trailing end. It is critical that the top and bottom are parallel after bending. Any misalignment will cause the top and bottom

sheets to cross roll and may result in reject foils because part of the foil extends beyond the top or bottom sheet of the can.

4. Before inserting the uranium foil, close the cans by cold rolling with a minimum of deformation to flatten the radius of the bend.
5. Open the can and insert the foil as far into the can as possible. The foil should be square to the sides of the can and centered in the can.
6. Cold roll the can containing the uranium foil with a maximum reduction in thickness of 0.127 mm per pass. Final passes may be even less. The folded edge of the can is always fed into the rollers first, and the can should be rotated 180° around the rolling axis after every pass.
7. After 50–70% reduction, a new can (fabricated as described in steps 1-5) may be required. Before inserting the foil into the new can, the foil should be trimmed square, edge cracks should be trimmed off and rotated from front to back to minimize the natural tapering effect that occurs from rolling in one direction only. Continue cold rolling for 15 – 40 passes, depending on the original thickness of the material, to reduce the foil to 0.125 ± 0.013 mm. The as-rolled length is at least 15% greater than the final foil length of the trimmed foil.
8. After rolling, the foils should be stored under a vacuum or an inert atmosphere to minimize oxidation before heat treating and assembly into targets.

Special notes for crossrolling

1. Depending upon the size of the original cast uranium ingot and the desired dimensions of the final foil, some cross rolling may be required. This may be done during the first hot passes if the rolls are sufficiently wider than the piece or during the first cold rolling passes. The foil should not be cross rolled for the final cold rolling passes to avoid buckling.
2. Before the cross rolling operation during cold rolling, the stainless steel sheet must be cut into appropriate size pieces. The dimensions are determined by the size of the hot rolled uranium pieces. To determine the required *width* of the stainless steel sheet, add a minimum of 6 cm to the *length* of the hot rolled uranium foil. The required *length* of the stainless steel sheet is two times the *width* of the hot rolled uranium foil plus a minimum of 6 cm. Except for the dimensions, the remaining can forming and rolling procedures are the same as above.

3. After cross rolling, the as-rolled width is at least 15% greater than the final foil width of the trimmed foil.
4. After rolling, the foils should be stored under a vacuum or an inert atmosphere to minimize oxidation before heat treating and assembly into targets.

Heat Treating (Beta-Quench)

I. Purpose

Hot and cold rolling of the uranium can produce large grains in the uranium foil, oriented in the direction of rolling. For safe irradiation, uranium grains must be fine and randomly oriented. To produce fine grains in random orientation, the foils must be heat treated. In the heat treatment, uranium (which is in the alpha phase at room temperature) is heated into the range where the beta phase is stable (above 658°C). This change in crystal structure breaks up the grains and their orientation. Heating to 705-720°C can be done by using (1) a furnace, (2) a molten lead bath, or (3) a molten salt bath like the eutectic mixture of 65 wt.% K_2CO_3 and 35 wt.% Li_2CO_3 . The uranium foil must then be quickly cooled as it moves back into the alpha phase. It is quenched by dropping it into a bath of water. When using molten salts or lead great care must be taken to minimize the amount of lead or salt on the sample before quenching. Molten lead or molten salts and water can produce an explosion!. The sample must also be completely dry before reheating in the molten lead or salt because explosions can also occur when the water reacts with the salt or lead! To assure random, small grains, the procedure is repeated again. To keep the uranium foil from reacting, it is sealed in a stainless-steel envelope. Once the foil(s) are inside, a vacuum is pulled on the envelopes as they are welded closed.

II Materials and Equipment:

- Stainless steel envelopes
- 300-amp AC/DC welder
- Thermal-protection gloves
- Tongs to hold the envelopes
- Furnace, molten lead, or molten carbonate bath
- Bucket of water
- Safety face shield
- Approved radiation safety clothing
- Metal Hand Shears
- Standard Bolt with 3 mm hole drilled down center
- Two nuts and washers to fit above bolt
- O-Ring (3 mm dia. Thickness) to fit above bolt
- Foil sealing (roller) tool

Copper Chills
Vacuum grease
6 mm hole punch

III Procedure:

1. Measure thickness and mass of foil(s). Record in log book or record sheets.
2. Place sample in commercial pre-welded (three sides) heat treating envelope which has had a 6 mm hole pre-punched in one side approximately 5 cm from the open end.
3. Insert washer on standard bolt with hole, put assembly into envelope and push bolt through hole in envelope.
4. Place vacuum greased O-ring on bolt
5. Place washer on bolt.
6. Put nut on bolt and tighten.
7. Fold (on open end) over 5 mm wide strip of heat treating bag and compress with sealing tool.
8. Repeat step 7.
9. Attach vacuum pump to bolt and evacuate envelope.
10. Fold over envelope 5 cm from the bolt on the side with the foil.
11. Fold over 5 mm wide strip of heat treating envelope next to the fold made in step 10.
12. Place copper chills across fold and seal envelope by welding under dynamic vacuum.
13. Shut off vacuum.
14. Take shears and cut envelope near bolt side to separate sealed foil from the bolt end of the envelope. The sealed foil is now ready for heat treatment.
15. Heat envelope between 705 and 720°C for twenty minutes.
16. Remove from heating unit and immediately quench into water bath stirring it for one minute to maximize cooling rate. Let it remain in bath for 3 minutes.
17. Remove from water bath, dry the envelope, and heat envelope between 705 and 720°C for twenty minutes.
18. Remove from heating unit and immediately quench into water bath stirring it for one minute to maximize cooling rate. Let it remain in bath for 3 minutes.

19. Remove from water bath, dry the envelope, and remove foil(s) from assembly.
20. Store foil(s) in a vacuum or inert atmosphere container until ready for their insertion into targets.

99Mo Cover Plates
Material - Plain Carbon Steel
Thickness as Provided
Drawing - 99MoTCW.Cover
4/17/95

99Mo Frame Plates
Material - Plain Carbon Steel
Thickness as Provided
Drawing - 99MoTCW.Frame
4/17/95

LEU Target Background Information

Cliff Conner

LEU Conversion Goals

- Assist current and future producers to convert to LEU
- Make conversion to LEU as painless as possible
 - Attain same ^{99}Mo yield for LEU target and process
 - Attain same product purity
 - Minimize changes in target geometry
 - Minimize modification to target processing
 - Minimize economic costs of conversion

Development Activities in CMT

- Develop LEU Target Technology
 - Develop heat treatment (β -quench) of U foils
 - Add fission recoil barriers to U foils
- Develop chemistry/equipment to dissolve U foils
 - Modify recovery and purification steps to obtain same yield and purity of ^{99}Mo
 - Determine effects of more U and Pu on current purification processes

Heat Treatment

- Heat Treatment
 - Heat foil to Beta Region ($T > 668^{\circ}\text{C}$); Quench
 - Removes preferred orientation
 - Obtains small grain size

Preferred Orientation

- Preferred orientation induced by foil manufacturing process.
 - All grains are aligned and elongated in a common direction
 - Rolling destroys crystal structure in uranium

Arc Melting

Arc Melting

Hot Rolling

Hot Rolling

Hot Rolling

Hot Rolling

Hot Rolling

Etching

Etching

Etching

Cold Rolling

Cold Rolling

Shearing

Beta Quench Heat Treatment

- Seal sample in stainless steel “can”
- Immerse in molten salt (700 - 720°C)
- Water Quench

Heat Treatment

Heat Treatment Test Matrix

- Samples Cold Worked to 30, 40, 50, and 60%
- Heat treat once or twice
- Analyzed using XRD for preferred orientation
- Grain size determined by microscopy

Preferred Orientation (XRD Results)

Ratio of 111/113 Reflections							
XRD Position							
60% CW	0°	30°	60°	90°	180°	270°	Average
No HT	1.52	1.21	1.10	0.80	0.67	0.92	1.04±30%
HT(x1)	6.20	5.84	5.93	6.74	5.98	5.57	6.04±7%
HT(x2)	5.61			5.90			5.76±4%
50% CW	0°	30°	60°	90°	180°	270°	Average
No HT	0.43			0.46			0.44±5%
HT(x1)	5.52			6.15			5.84±8%
HT(x2)	6.93			5.57			6.25±15%
CW=Cold Work							
HT=Heat Treatment							

Preferred Orientation (XRD Results)

Ratio of 111/113 Reflections						
XRD Position						
40% CW	0°	30°	60°	90°	180°	270°
No HT	1.73			1.57		Average 1.65±7%
HT(x1)	4.92			3.74		4.33±19%
HT(x2)	6.44			5.48		5.96±11%

30% CW	0°	30°	60°	90°	180°	270°
No HT	3.75			4.13		Average 3.94±7%
HT(x1)	4.53			4.29		4.41±4%
HT(x2)	5.83			6.33		6.08±6%

CW=Cold Work
HT=Heat Treatment

Grain Size of 30% Cold Worked Sample

No Heat Treatment

Double Heat Treatment

Grain Size of 60% Cold Worked Sample

No Heat Treatment

Double Heat Treatment

Heat Treatment

- Double heat treatment effective regardless of starting amount of cold work.
 - removing preferred orientation
 - providing small grain size

Fission Recoil Barriers

- Prevent bonding of uranium foil to target structural materials
 - Catch fission recoils
 - Increases post irradiation durability
- Fission recoil barriers
 - Electroplated
 - Wrapped foils

Fission Recoil Barriers

■ Foil Wrapped Barriers

- Ni
- Zn
- Al
- Cu
- Fe

■ Electroplated Barriers

- Ni
- Zn

Foil Barriers

- Grain structure of foil fission recoil barriers better than electroplated barriers
- Barriers bond to uranium foil during irradiation
- For annular targets assembly is easy with foil fission recoil barriers
 - For tapered style target was difficult

Electroplating

- Fission-fragment barriers can be electroplated onto uranium to prevent bonding of the uranium to the target tubes.
- Nickel or zinc fission-fragment barriers have been plated onto uranium foils.
- For tapered style target assembly is easier with electroplated barriers

Plating of Fission Recoil Barriers

- Surface preparation of uranium foil is key to successful electrodeposition
 - Too little surface etching and barrier does not adhere
 - Too much surface etching and foil can be destroyed
 - We have set conditions so that good plating can be achieved with <5% loss of uranium

Nickel Plating Bath

Heater

Cathode Bar

Power Supply Filter/Pump Bath

Ni Anodes

Plating of Fission Recoil Barriers

Robber Cathode Reduces Ni Overplating at Edges

Plated Uranium Foil

- 130- μm uranium foil with 10- μm fission-recoil barrier of nickel

Nickel-Plated Foil

Instructions for Assembling Ni-Foil-Wrapped LEU-Metal-Foil Annular Targets

- I. Prepare the Target Parts
 - A. Machine the inner and outer tubes per drawing CMT-C1518-1/-2 Rev. 01.
 - B. Clean-off machining fluids from tubes using standard machine shop method. They will be thoroughly cleaned in later steps.
- II. Prepare the Uranium Foil for Insertion into Target.
 - A. Size the uranium foil
 1. Cut 125 μ m [0.005"] thick uranium foil stock material to 44 $\pm$ 2 x 76 $\pm$ 2 mm [1.75" x 3"] for an 8 g target or 76 $\pm$ 2 x 88 $\pm$ 2 mm [3" x 3.5"] for a 16 g target.
 2. Place foil on top of a piece of glass previously cleaned with ethanol
 3. Clean the Uranium foil with a lint free cloth moist with ethanol. Use latex gloves to handle the foil
 4. Weigh the cut uranium foil. Use a scale that has at least two decimal points.
 5. Record the weight of the uranium foil in the recording sheet provided (attached).
 6. Make several thickness measurements on the uranium foil using the digital micrometer and record them in the recording sheet provided
 - B. Envelope the uranium foil with nickel-foil fission-recoil barrier
 1. Cut a piece of 15 μ m [0.0006"] nickel foil slightly larger than necessary to fully cover both sides of the uranium foil.
 2. Place Ni foil on the piece of glass for cleaning.
 3. Clean the foil with a lint free cloth moist with ethanol.
 4. Fold the nickel foil in half to create an envelope.
 5. Slide the uranium foil into the nickel-foil envelope, Figure 1.
 6. Use a plastic ruler (clean with ethanol first) to smooth out the foils on both sides, especially in the fold area. Use a diluted rubber-cement mixture to keep the foil in place during assembly.
 - a. Dilute the rubber cement approximately one part cement to nine parts hexane.
 - b. Open the envelope and place two drops of the rubber-cement mixture onto the uranium foil.
 - c. Smooth the nickel foil out over the uranium foil using a paper towel.
 - d. Turn envelope over, open the other side of the envelope, and place two drops of the rubber-cement mixture onto this side of the uranium foil.
 - e. Smooth the nickel foil out over the uranium foil with a paper towel until all the excess hexane/rubber-cement mixture has been expelled from the edges of the envelope.

- f. Continue smoothing the nickel foil out until the hexane has evaporated. (This takes about a minute).

Figure 1. Rubber cement applied on U foil inside Ni foil. Note: the foil in this photograph is copper.

7. Wipe the surface with a lint-free cloth dampened with ethyl alcohol
 8. Trim the nickel foil to ~2 mm larger than the uranium foil and fold the ends over to facilitate assembly in the tubes. Use a clean ruler or smooth object like a Sharpie® marker to flatten out the folds. The flatter the folds, the easier assembly will be. Do this flattening on top of the piece of glass.
 - C. Weigh the uranium foil with the nickel barrier to at least two decimal points.
 - D. Record the weight of the foil and barrier. Subtract the foil weight from the weight of the foil plus the barrier to obtain the weight of the nickel foil barrier. Record weights on the log sheets.
 - E. Assemble uranium foil into target within a few hours of gluing the nickel barrier onto the target.
- III. Assemble the Target
- A. Prepare the tubes prior to assembly
1. Measure the tubes at several places both ID and OD.
 2. Mark the tubes by scribing with a target identification code.
 3. Make a longitudinal scribe on the outer tube for the foil gap position, Figure 2.

Figure 2. Target assembly showing scribing mark on tube

- B. Clean the tubes (use powder-free clean-room gloves to handle tubes to prevent contamination of the weld faying surfaces with particulates).
1. Place in ultrasonic cleaner for ~10 minutes at ~90°C in Contrex™ or the equivalent.
 2. Rinse the tubes thoroughly in running water.
 3. Bright dip¹ the weld faying surfaces for 30-60 seconds at 90±10°C. Do this procedure inside a hood since harmful vapors will be produced, Figure 3.

Figure 3. Tube in Bright Dip.

4. Rinse the tubes thoroughly in running water.
5. Place tubes in the tube holder and hot air dry for 10 minutes at ~125°C using a heat gun. Figure 4.

¹ Bright dip consists of 90 vol% concentrated H_3PO_4 / 5 vol% H_2SO_4 / 5vol% HNO_3 .

² This scribe mark is critical to making a longitudinal cut along the target after irradiation for disassembly

6. Assemble targets within 3 hours after cleaning target tubes or store in He atmosphere.

Figure 4. Drying of tubes after bright dip.

- C. Assemble the tubes (use powder free clean room gloves to handle tubes to prevent contamination of the weld faying surfaces with particulates).
 1. Wrap the uranium/nickel foil around the inner tube.
 2. Slide the inner tube into the outer tube (alcohol may be used as a lubricant), Figure 2.
 3. Ensure that the foil gap is lined up with the scribed mark on the outer tube²
 4. When tubes are nearly slid together, wipe the exposed weld faying surfaces with a lint free cloth dampened with ethyl alcohol.
 5. Push the inner tube slightly past flush and wipe the other exposed weld faying surfaces with a lint free cloth dampened with ethyl alcohol.
 6. Push the inner tube back flush.
 7. Place in a vacuum chamber and evacuate for ~10 minutes to ensure that the alcohol is completely evaporated, Figure 5. Clean the interior of the draw die (Figure 6) with a paper towel moist with ethanol.
 8. Remove from vacuum chamber and wipe a small amount of Joy™ detergent (liquid dish washer detergent) or equivalent on the inside of the inner tube. This will act as a lubricant for the draw plug.

Figure 5. Vacuum Chamber.

9. Pull an appropriately sized draw plug through the inner tube. The size of the draw plug required is calculated by subtracting the OD of the inner tube (~28.2 mm [1.11"]) then adding the Id of the inner tube (~26.2 mm [1.03"]), adding 0.01 mm [0.0005"], and finally selecting the draw plug closest in size to the one required. Draw plug sizes are 26.42 mm, 26.44 mm, 26.47 mm, 26.49 mm and 26.52 mm [1.040", 1.041", 1.042", 1.043" and 1.044"]. An example calculation is given below:

ID of Outer Tube	28.22 mm [1.111"]
OD of Inner Tube	-27.99 mm [1.102"]
Difference	0.23 mm [0.09"]
ID of Inner Tube	+26.21 mm [1.032"]
Draw Plug Size	26.44 mm [1.0415"]

- a. Figure 6 shows the draw die hardware.
- b. Figure 7 shows the assembled die with a target and draw plug exposed.
- c. A cut-away sketch of the draw die assembly is shown in Fig. 8.
- d. Drawings of the draw die and hardware are given in CMT-D1562 Rev. 01.

Figure 6. Draw Die Hardware

Figure 7. Assembled Draw Die with Target and Draw Plug Exposed

Figure 8. Cut Away Sketch of Draw Die Assembly

10. Wipe off any excess detergent and clean target lightly with lint-free cloth dampened with ethyl alcohol.
11. Put targets into vacuum chamber, evacuate for at least 15 minutes before welding (this evaporates the ethanol). If welding is not done immediately, back fill the chamber with He and allow tubes to stand overnight in He atmosphere.

III. Weld the Ends of the Target

- A. Remove the targets from He atmosphere
- B. Prepare targets for welding
 1. Machine target to correct length facing off equal amounts of material from both ends to create flat parallel surfaces, Figure 9.

Figure 9. Machining the end of the tubes.

2. Cut $\sim 0.5 \text{ mm} \times 45^\circ$ [$\sim 0.020'' \times 45^\circ$] chamfer on the OD and ID surfaces after facing.
 3. After machining, avoid touching the ends of the target. Use gloves to handle them or wrap them in a paper towel. Any dirt on the welding surface might create pinholes during welding.
 4. Weld target within 2 hours of removing from He atmosphere and machining ends (or store in He atmosphere).
 5. Place the chills that go on the OD approximately 1 wall thickness below end of target, Figure 10. A sketch of the chill is given in Fig. 11.
 6. Place the chills that go in the ID approximately 1 wall thickness below end of target, Figure 10. A sketch of the chill is given in Fig. 12.
- C. TIG weld the target closed using a single fusion pass with the following parameters (Figures 13 and 14):
- DC current full amperage $\sim 40\text{A}$
 - 90% He/10% Ar cover gas at $\sim 20 \text{ scfm}$.
 - #4 gas cup
 - $1/16''$ thoriated-tungsten electrode ground to a sharp point
 - Target rotated at $\sim 3 \text{ rpm}$ during welding
 - Strike the arc on the OD chill and move to the aluminum tubes while bringing the weld arc up to full amperage
1. Electrode to work gap $1/32'' - 1/16''$
- D. Remove weld chills immediately and allow them to cool.
- E. Weld the other end closed using the same method.

Figure 10. Target with chills assembled

Figure 11. OD chill of the target

Figure 12. ID chill of the Target

Figure 13. Target Welding.

Figure 14. Welded Target.

IV. Initial Leak Test Target

- A. Put targets into vacuum chamber evacuate for >10 minutes and back fill with ~70 kPa [10 psi] He for >5 minutes.
- B. Wipe outside of targets with alcohol to remove any adsorbed He.
- C. Allow targets to stand ~2 hours to allow any residual adsorbed He to evaporate.
- D. Put target in He leak detector and verify that the leak rate is $<5 \times 10^{-7} \text{ cm}^3/\text{s}$. Repair welds if leak rate exceeds specified value.

V. Repair Welds as Necessary

- A. Place OD and ID weld chills approximately 3 wall thickness below the end of the targets.
- B. TIG weld any holes closed with the following parameters:
 - AC current with superimposed high frequency full amperage ~60A
 - Ar cover-gas at ~20 scfm.
 - #4 gas cup
 - 3/32" zirconated-tungsten electrode with a ball tip
 - Strike the arc on the aluminum tubes over spot needing repair. Repair hole using skill of craft.
- C. Remove weld chills immediately and allow them to cool.
- D. Make any other necessary repairs and He leak check target again.

VI. Final Sizing of Diameters

- A. Welding the ends of the target may cause a small amount of roll-over on the ID of the target. This can be corrected using a slip-roller with 25 mm [1"] diameter rolls (Figure 15).
- B. Determine how much of the weld roll-over must be taken out by attempting to slide the target onto a target insertion rig (drawing CMT-D1519).
- C. Slide the target over the roller of the slip-roller. Adjust the roller to "squeeze" the weld down.
- D. Roll the target in the slip-roller, Figure 15.
- E. Remove from slip roller. Repeat B-E until the target easily slides onto the target insertion rig, Figure 16.
- F. Verify that the target will slide through the gauge shown in Fig. 17 to ensure that the OD of the target is the correct size, Figure 18.

Figure 15. Resizing of target in slip-roller.

Figure 16. Target fitted in insertion rig.

Figure 17. OD Diameter Gauge

Figure 18. Target inserted in Diameter Gauge.

VII. Initial Cleaning

- A. Rinse target thoroughly in running water.
- B. Buff all surfaces with Scotchbrite™ pad or equivalent using soap and water.
- C. Rinse target thoroughly in running water.
- D. Wipe target thoroughly with alcohol and lint-free cloth.

VIII. Final Leak Test

- A. Put targets into vacuum chamber evacuate for >10 minutes and back fill with ~70 kPa [10 psi] He for >5 minutes.
- B. Wipe outside of targets with alcohol to remove any adsorbed helium.
- C. Allow targets to stand ~2 hours to allow any residual adsorbed He to evaporate.
- D. Put target in He leak detector and verify that the leak rate is $<5 \times 10^{-7} \text{ cm}^3/\text{s}$.

IX. Final Cleaning

- A. Rinse target thoroughly in running water.
- B. Wipe target thoroughly with alcohol and lint-free cloth.
- C. Wrap target in a paper towel and store until irradiation

Person responsible _____

Name of Target _____

Date Assembled _____

Weight of LEU Foil _____ g

Weight of LEU + Ni Foil _____ g
after trimming

Weight of Ni Foil _____ g

CALCULATION ON DRAW DIE SIZE

ID of Outer Tube	
OD of Inner Tube	
Difference	
ID of Inner Tube	
Sum	
Additional 0.01 mm (0.0005")	
Draw Plug Size	
Selected Draw Size	

Person responsible

Name of Target

Date Assembled

Inner Tube	

Average

Outer Tube

Average

A-4 ARGONNE TARGET DISASSEMBLY PROCEDURE

Target Disassembly Procedure

Description of the target cutter

The target cutter (Figure 1) is essentially a modified precision tube cutoff machine that is designed for making smooth, perfectly square cuts. The manufacturer's specifications are given below. The cutoff machine can be powered with either 110 VAC or 220 VAC.

Cutter type	Dry abrasive tool
Dressing tool	Precision ground and aligned hard tool steel
Dimensions	8" x 6.25" x 4.25" (20.3 cm x 15.8 cm x 10.8 cm)
Weight	11 lbs (4.9 kg)
Power	55 W

Figure 1. Photograph of the target cutter before final assembly. In the back is the tube cutoff machine (Model TC-20 from LabAlliance Inc.). The slide mechanism is shown with the target holder and a mock target. In the front are the precision positioner and an abrasive wheel.

The target is clamped into an aluminum holder that was machined to hold the target. The holder is attached to a bracket that is, in turn, attached to an extended slide mechanism. The long axis of the slide is perpendicular to the motor shaft (parallel to the cutting surface of the abrasive wheel). The slide is attached to a positioner that allows one to precisely control the depth of the cut in the target. It is essential that the outside tube of the target be cut through, but the inside tube of the target be left intact. The reinforced abrasive wheel is 4.5" in diameter and is 1/32" thick. The target cutter is optimized for making the longitudinal cut on the target. However, the holder is made so that the target can be clamped so that its long axis is parallel to the motor shaft (perpendicular to the cutting surface of the abrasive wheel). This method of cutting the ends from the targets requires that several cuts be made in each end requiring several repositionings. In addition, we have modified a 1/4 -1 5/8" manual tubing cutter for remote use to cut the welded ends off of the targets. The better alternative method for cutting off the ends depends on the preference of the hot-cell technician.

Cutting targets Open

The following is a step-by-step procedure for target disassembly:

Cutting off the Ends

1. _____ Place target into holder with the long axis of the target perpendicular to the slide and the cutting surface of the wheel (Figure 2) and tighten set screw.

Figure 2. The target is secured in the holder, and ready for cutting by the cutting machine. This operation is repeated on the other end of the target.

2. _____ Place the tube end directly over the cutting blade.
3. _____ Turn on cutter.
4. _____ Push the block fully down.
5. _____ Turn off the cutter.
6. _____ Loosen set screw and rotate the target about 120°
7. _____ Repeat steps 3 through 6 until the target is cut through.
8. _____ Repeat steps 1-4 on the other end of the target

Alternate Method for Cutting Ends Off of Target

1. _____ Place manual tube cutter on target and cut end of target about ¼" from the end of the target as shown in Figure 3.

Figure 3. The target is secured in the holder, and the end is cut using a manual tubing cutter. This operation is repeated on the other end of the target.

2. _____ Rotate manual tube cutter, and tighten periodically until target is cut through.

Cutting the Outer Tube Longitudinally

1. _____ Make sure the cutting machine is off.
2. _____ Place the target into holder with the long axis of the target parallel to the slide and the cutting surface of the wheel (Figure 4) and tighten set screw.

Figure 4. Photograph of the assembled target cutter. The abrasive wheel is located under the target. A block has been added to the positioner to make it easier to use with a manipulator.

3. _____ Position the target in the holder so that the scribe mark (gap in the foil) is pointing directly up. *Note: Targets should be scribed along the gap in the foil. Alternatively, the position of the foil in an irradiated target can be seen based on the dark oxidation on the outside of the target.*
4. _____ Position the target over the cutting wheel and move it down by turning the positioner clockwise until the target just touches the cutting wheel. See Figure 5.
5. _____ Slide the target to the left side of the slide
6. _____ Turn the positioner 1.25 turns down (clockwise).
7. _____ Turn on cutter and slowly push target into the rotating wheel and through to the other side of the slide so that the cutting wheel has cut a gap through the entire length of the target. See Figure 6.
8. _____ Turn cutter off and slide the target forward.
9. _____ Move the positioner up ~1.5 counterclockwise turns.

Figure 5. The target can be precisely positioned relative to the cutting wheel by means of the micrometer. This positioning is used to control the depth of the cut and to compensate for wear of the cutting wheel.

Figure 6. Photograph of the target cutter. The tube on the right is a mock target, containing no uranium foil that is clamped into the holder. The target and holder move along the slide (into and out of the plane of the picture). The cutting wheel is below the target.

10. _____ Turn the target in the holder 180° so that the cut just made is pointing up. *This will position the gap in the foil precisely over the cutting wheel.*
11. _____ Set target directly over the cutting wheel and move the positioner so that it just touches the outer tube.
12. _____ Slide target forward.
13. _____ Turn positioner 1 clockwise turns down.
14. _____ Turn on the cutter and slowly push target into the rotating wheel until the target is completely cut from end to end. **DO NOT PUSH DOWN ON THE BLOCK.**
15. _____ Turn cutter off and slide the target forward
16. _____ Turn positioner 1 clockwise turns down.
17. _____ Turn on the cutter and slowly push target into the rotating wheel until the target is completely cut from end to end. **DO NOT PUSH DOWN ON THE BLOCK.**
18. _____ Turn cutter off and slide the target forward
19. _____ Turn positioner 0.5 clockwise turns down.
20. _____ Turn on the cutter and slowly push target into the rotating wheel until the target is completely cut from end to end. **DO NOT PUSH DOWN ON THE BLOCK.**
21. _____ Turn cutter off and slide the target forward
22. _____ Remove target from holder. *Note: if the outer tube is cut through, then it will probably spring open and wedge itself in the holder. If the target is difficult to remove from the holder, then it is probably cut correctly, if it is loose in the holder it probably needs to be cut deeper. (Figure 6)*

Figure 6. Photograph of target after it has been cut.

Removing the Foil from the Cut Tube

1. _____ Once the outer tube has been cut through, then the target can be placed on the cone tool (Figure 7a).
2. _____ Push the target down, forcing it over the cone tool. This should allow the outer tube to slide to the bottom of the tool, and leave the inner tube nearer the top. (Figure 7b).
3. _____ Remove the foil(s) and place in transfer container. *Note: The foil usually adheres weakly to the inner tube and can be removed with tweezers.*

Figure 7 Photograph showing the cone tool and a cut target. In 7a the target is sitting to the left of the tool. In 7b, the separation of the target has been accomplished. Near the bottom is the outer tube; the long cut can be seen. Near the top is the inner tube. The foil is near the middle.

Replacement of Cutting Wheel

As the blade wears, at some point (determined by operator) the abrasive cutting wheel will need to be replaced. Figure 8 shows a blade change operation. The steps for replacement are:

1. _____ Raise the cover of the target cutter.
2. _____ Place a pin through the motor shaft between the motor and the cutting wheel to lock the motor into position.

3. _____ Use a nut driver to loosen the outer nut that secures the cutting wheel to the motor shaft.
4. _____ When the nut is removed, the wheel can be removed and a new one put in its place.
5. _____ Retighten nut.
6. _____ Remove pin from motor shaft.
7. _____ Close cover of the target cutter.

Figure 8. Photograph of remote operation for changing the cutting wheel. The left manipulator is holding a pin that goes through the motor shaft to lock the shaft in place. The right manipulator is unscrewing the nut that secures the wheel to the shaft.

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APPENDIX B:
MISSOURI UNIVERSITY RESEARCH REACTOR

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B-1 CALCULATION OF FISSION GAS INVENTORY PRODUCED DURING U-FOIL IRRADIATION

TDR-0111

***Production of Fission Product Mo-99
Using the LEU-Modified Cintichem Process***

***Calculation of Fission Gas Inventory
Produced During U-Foil Irradiation***

University of Missouri Research Reactor

August 2007

Title: Production of Fission Product Mo-99 Using the LEU-Modified Cintichem Process -
Calculation of Fission Gas Inventory Produced During U-Foil Irradiation

Originator(s) Signature	Date	Approval(s) Signature	Date
<i>Charles W. Allen</i>	<i>8/13/07</i>	<i>[Signature]</i>	<i>10/24/07</i>
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Electronic Distribution	Abstract:
D. Robertson A. Ketring L. Foyto R. Butler W. Miller W. Meyer C. Allen M. Flagg	<p>This calculation was performed to determine the total inventory (moles gas) of all fission gases generated during the irradiation of an LEU-Foil target for an irradiation time of 150 hours (6.25 days) at a neutron flux of $8.0E13 \text{ n/cm}^2\text{-s}$.</p> <p>The results of this calculation identify that approximately $1.44E-04$ moles (mol) of fission gas will be generated during the irradiation of a 20 gram LEU-Foil target. The results also indicate that xenon is the predominant fission gas. Approximately six times more xenon is generated than krypton. Approximately six times more xenon is generated than iodine.</p> <p>The results of this calculation will be used to establish a maximum conservatively bounding internal pressure of an annular or plate type LEU-foil target. During irradiation, the walls of a target will deform and thus create a volume in which the fission gases can accumulate. The maximum pressure will be used as an input parameter to the finite element analysis of the target. The finite element analysis will be performed to evaluate the structural stability and integrity of the target during both irradiation and post-irradiation.</p>

TDR No.: 0111 Revision No.: 0

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Introduction

The purpose of this calculation is determine the total inventory (moles gas) of all fission gases generated during the irradiation of an LEU-foil target for an irradiation time of 150 hours (6.25 days). The definition of a “mol” is presented in Attachment 1. The results of this calculation will be used to establish the maximum conservatively bounding internal pressure of an annular or plate type LEU-Foil target. During irradiation, the walls of a target will deform and thus create a volume in which the fission gases can accumulate. The maximum pressure will be used as an input parameter to the finite element analysis of the target. The finite element analysis will be performed to evaluate the structural stability and integrity of the target during both irradiation and post-irradiation.

Calculation

The ORIGEN 2.0 neutronics code is used to determine the mass of the fission gases produced during target irradiation. The ORIGEN 2.0 input parameters are:

Irradiation Time = 150 hours LEU-Foil Enrichment = 19.75% U-235 Neutron Flux = $8.0\text{E}13 \text{ n/cm}^2\text{-s}$

The moles of gaseous actinide decay daughters are intentionally omitted as their ORIGEN 2.0 calculated mass is considered to be negligible. The moles of tritium are also omitted.

${}^4_2\text{He} \rightarrow 2.65\text{E-}12 \text{ g per gram of LEU-Foil irradiated}$

${}^{219}_{86}\text{Rn}$
 ${}^{220}_{86}\text{Rn}$
 ${}^{222}_{86}\text{Rn}$

Total $\rightarrow 2.54\text{E-}23 \text{ g per gram of LEU-Foil irradiated}$

Tritium, ${}^3_1\text{H} \rightarrow 6.87\text{E-}09 \text{ g per gram of LEU-Foil irradiated}$

Element: ${}^{84}_{36}\text{Kr}$ (Noble Gas)

Isotope	Half-Life	ORIGEN 2.0 Isotope Mass (g) per gram of U-Foil irradiated	[ORIGEN 2.0 Mass ÷ Atomic Mass] yields moles of gas generated	Moles of krypton gas per gram of U-Foil irradiated
Kr^{80}	Stable	1.11E-11	1.11E-11 / 79.91 amu	1.40E-13
Kr^{81}	2.1E05 yrs	3.43E-14	3.43E-14 / 80.91 amu	4.24E-16
Kr^{82}	Stable	1.58E-09	1.58E-09 / 81.91 amu	1.93E-11
Kr^{83}	Stable	8.79E-06	8.79E-06 / 82.91 amu	1.06E-07
$\text{Kr}^{83\text{m}}$	1.86 hrs	1.60E-07	1.60E-07 / 82.91 amu	1.93E-09
Kr^{84}	Stable	1.79E-05	1.79E-05 / 83.91 amu	2.13E-07
Kr^{85}	10.73 yrs	4.60E-06	4.60E-06 / 84.91 amu	5.42E-08
$\text{Kr}^{85\text{m}}$	4.48 hrs	9.59E-07	9.59E-07 / 84.91 amu	1.13E-08
Kr^{86}	Stable	3.62E-05	3.62E-05 / 85.91 amu	4.21E-07
Kr^{87}	1.27 hrs	5.66E-07	5.66E-07 / 86.91 amu	6.51E-09
Kr^{88}	2.84 hrs	1.80E-06	1.80E-06 / 87.91 amu	2.05E-08
Total moles of krypton per gram of U-Foil irradiated = 8.34E-07				

Calculation (Cont'd)

Element: $_{54}\text{Xe}^{131}$ (Noble Gas)

Isotope	Half-Life	ORIGEN 2.0 Isotope Mass (g) per gram of U-Foil irradiated	[ORIGEN 2.0 Mass + Atomic Mass] yields moles of gas generated	Moles of xenon gas per gram of U-Foil irradiated
Xe^{128}	Stable	4.62E-10	4.62E-10 / 127.9 amu	3.61E-12
Xe^{129}	Stable	2.33E-13	2.33E-13 / 128.9 amu	1.81E-15
$\text{Xe}^{129\text{m}}$	8.9 days	1.10E-13	1.10E-13 / 128.9 amu	8.53E-16
Xe^{130}	Stable	1.36E-08	1.36E-08 / 129.9 amu	1.05E-10
Xe^{131}	Stable	1.64E-05	1.64E-05 / 130.9 amu	1.25E-07
$\text{Xe}^{131\text{m}}$	4.9 days	1.63E-07	1.63E-07 / 130.9 amu	1.25E-09
Xe^{132}	Stable	5.21E-05	5.21E-05 / 131.9 amu	3.95E-07
Xe^{133}	5.24 days	1.06E-04	1.06E-04 / 132.9 amu	7.98E-07
$\text{Xe}^{133\text{m}}$	2.19 days	2.13E-06	2.13E-06 / 132.9 amu	1.60E-08
Xe^{134}	Stable	2.12E-04	2.12E-04 / 133.9 amu	1.58E-06
Xe^{135}	9.1 hrs	1.45E-06	1.45E-06 / 134.9 amu	1.07E-08
$\text{Xe}^{135\text{m}}$	15.4 min	7.74E-08	7.74E-08 / 134.9 amu	5.74E-10
Xe^{136}	Stable	3.41E-04	3.41E-04 / 135.9 amu	2.51E-06
Xe^{138}	14.13 min	4.05E-07	4.05E-07 / 137.9 amu	2.94E-09
Total moles of xenon per gram of U-Foil irradiated = 5.44E-06				

Calculation (Cont'd)

Element: $_{53}\text{I}^{127}$ (Sublimes @ Room Temperature)

Isotope	Half-Life	ORIGEN 2.0 Isotope Mass (g) per gram of U-Foil irradiated	[ORIGEN 2.0 Mass + Atomic Mass] yields moles of gas generated	Moles of iodine gas per gram of U-Foil irradiated
I^{127}	Stable	1.12E-06	1.12E-06 / 126.9 amu	8.83E-09
I^{128}	25 min	2.60E-12	2.60E-12 / 127.9 amu	2.03E-14
I^{129}	1.57E07 y	1.46E-05	1.46E-05 / 128.9 amu	1.13E-07
I^{130}	12.4 hrs	2.56E-09	2.56E-09 / 129.9 amu	1.97E-11
$\text{I}^{130\text{m}}$	9 min	1.94E-11	1.94E-11 / 129.9 amu	1.49E-13
I^{131}	8 days	5.74E-05	5.74E-05 / 130.9 amu	4.39E-07
I^{132}	2.3 hrs	1.90E-06	1.90E-06 / 131.9 amu	1.44E-08
I^{133}	20.8 hrs	3.68E-05	3.68E-05 / 132.9 amu	2.77E-07
I^{134}	52.6 min	1.77E-06	1.77E-06 / 133.9 amu	1.32E-08
I^{135}	6.6 hrs	1.12E-05	1.12E-05 / 134.9 amu	8.30E-08
Total moles of iodine per gram of U-Foil irradiated = 9.48E-07				

Total moles of Kr, Xe, and I per gram of U-Foil irradiated = 7.22E-06 mol

Total moles of fission gas for a 20g U-Foil = 7.22E-06 mol/g of U-Foil x 20g = 1.44E-04 mol

Conclusions

- Xenon is the predominant fission gas. Approximately six times more xenon is generated than krypton. Approximately six times more xenon is generated than iodine.
 - Xenon represents approximately 75% of the total fission gas generated.
 - Krypton represents approximately 12% of the total fission gas generated.
 - Iodine represents approximately 13% of the total fission gas generated.
- The “stable” xenon isotopes represent approximately 64% of the total fission gas generated.
- The “stable” krypton isotopes represent approximately 10% of the total fission gas generated.
- The “stable” iodine isotope (I-127) represents only about 0.12% of the total fission gas generated.

Attachment 1

TDR-0111
Revision 0

Conversion factor between atomic mass units and grams

The standard scientific unit for dealing with atoms in macroscopic quantities is the mole (mol), which is defined arbitrarily as the amount of a substance with as many atoms or other units as there are in 12 grams of the carbon isotope C-12.

The number of atoms in a mole is called Avogadro's number, the value of which is approximately 6.02×10^{23} . One mole of a substance always contains almost exactly the relative atomic or molar mass of that substance (which is the concept of molar mass), expressed in grams.

For example, the relative atomic mass of iron is 55.847, and therefore one mole of iron has a mass of 55.847 grams. The formulaic conversion between atomic mass and SI mass in grams for a single atom is

$$m_{\text{grams}} = \frac{m_u}{N_A}$$

where u is the atomic mass unit and N_A is Avogadro's number.

Credit / Source: <http://en.wikipedia.org>

B-2 MURR ROLLED FOIL TARGET ASSEMBLY INSTRUCTIONS

Revision 1

Annular Target Assembly Instructions

ANL Rolled LEU-Foil

- 1.0 Prepare the Target Tubes
 - 1.1 Machine the inner and outer tubes to the dimensions identified in Attachment 4.
 - 1.2 Clean-off machining fluids from tubes using standard machine shop method (i.e., detergent soap & water). The tubes will be thoroughly cleaned in later steps.
- 2.0 Prepare the Uranium Foil for Annular Target Assembly
 - 2.1 Size the uranium foil as follows:
 - a. Cut 135 μm [0.0053"] thick low enriched uranium foil stock material to $25.4 \pm 2 \times 76 \pm 2 \text{ mm}$ [1.0" x 3.0"] for a 5-g target.
 - b. Place foil on top of a piece of glass previously cleaned with ethanol.
 - c. Clean the uranium foil with a lint free cloth moist with ethanol. Use powder free latex gloves to handle the foil.
 - 2.2 Weigh the cut uranium foil to at least two (2) decimal places.
 - a. Record the weight of the uranium foil on the data sheet provided (Attachment 1).
 - b. Take thickness measurements of the uranium foil using a digital micrometer and record them on Attachment 1.
 - 2.3 Envelope the uranium foil with the nickel-foil fission recoil barrier.
 - a. Cut a piece of 15 μm [0.0006"] thick nickel foil slightly larger than necessary to fully cover both sides of the uranium foil.
 - b. Place Ni-foil on the piece of glass for cleaning.
 - c. Clean the Ni-foil with a lint free cloth moist with ethanol.
 - d. Fold the Ni-foil in half to create an envelope.
 - e. Slide the uranium foil into the nickel-foil envelope as shown in Figure 1a.
 - f. Use a plastic ruler (clean with ethanol first) to smooth out the foils on both sides, especially in the fold area.

Figure 1a

Figure 1b

- g. Trim the Ni-foil to ~6 mm larger than the uranium foil on each of the three open sides. Fold the ends over to facilitate placement in the aluminum tubes. Fold the short-ends of the Ni-foil over itself only, not back over the uranium foil (Figure 1b). After all folds are complete, use a smooth object like a Sharpie® marker to flatten out the folds. The flatter the folds, the easier assembly will be. Flatten on top of a glass surface.

- h. Weigh the Ni-foil wrapped uranium foil in grams to at least two decimal places and record the weight on Attachment 1.
- i. Subtract the uranium foil weight from the weight of the uranium foil wrapped in its Ni fission recoil barrier to obtain the weight of the Ni-foil. Record weight of the Ni-foil on Attachment 1.

3.0 Annular Target Assembly

- 3.1 Prepare the inner and outer tubes prior to assembly.
 - a. Measure and record the I.D. and O.D. of the inner tube on Attachment 2.
 - b. Measure and record the I.D. of the outer tube on Attachment 2.
 - c. Make a longitudinal scribe mark on the outer tube to identify foil gap position (Figure 2).

Figure 2

- 3.2 Clean the tubes. Use powder-free latex gloves to handle the tubes to prevent particulate contamination of the weld faying surfaces.
 - 3.2.1 Place in ultrasonic cleaner containing a solution of Citranox (Attachment 3) for ~10 minutes at ~ 90°C.
 - 3.2.2 Thoroughly rinse tubes in running water and shake to remove excess water.
 - 3.2.3 Bright Dip¹ (Figure 3) the weld faying surfaces for 30 – 60 seconds at 90 ± 10°C. Perform this step inside a hood as harmful vapors will be produced.

Figure 3

- 3.2.4 Thoroughly rinse tubes in running water.
- 3.2.5 Hot-air dry tubes for at least ten minutes at ~ 125°C using a heat gun (Figure 4).

¹ Bright Dip consists of 90 vol% concentrated H₃PO₄ / 5 vol% H₂SO₄ / 5vol% HNO₃.

Figure 4

3.2.6 Assemble the target within 3 hours after cleaning or store in a helium atmosphere, or under vacuum.

3.3 Target Assembly

3.3.1 Wrap the uranium/nickel foil around the inner tube. Note: Wear powder-free latex gloves when assembling the target to prevent particulate contamination of the weld faying surfaces.

3.3.2 Slide the inner tube into the outer tube (Figure 2).

3.3.3 Ensure that the foil gap is lined up with the scribed mark on the outer tube.

3.3.4 When the tubes are nearly slid completely together, wipe the exposed weld faying surfaces with a lint-free cloth dampened with acetone.

3.3.5 Push the inner tube slightly past flush and wipe the other exposed weld faying surfaces with a lint-free cloth dampened with acetone.

3.3.6 Push the inner tube back flush.

3.3.7 Place in a vacuum chamber (Figure 5) and evacuate for ~10 minutes to ensure that the acetone is completely evaporated. A vacuum oven can be used instead of vacuum chamber. Clean the interior of the draw die (Figure 6) with a paper towel moist with acetone.

3.3.8 Coat the inside of the inner tube with "dry graphite" (Neolube or equivalent, Attachment 5). This will act as a lubricant for the draw plug and prevent the inner tube from galling.

Figure 5

Figure 6

3.3.9 Pull the draw plug through the inner tube. The size of the draw plug to be used is calculated by subtracting the O.D. of the inner tube from the I.D. of the outer tube, then adding the I.D. of the inner tube plus an additional 0.0125 mm (0.0005"). The draw plug to be used is the one closest in size to the calculated value. Draw plug sizes are 26.47 mm, 26.49 mm, 26.52 mm, 26.54 mm, and 26.57 mm [1.042", 1.043", 1.044", 1.045", and 1.046"]. An example calculation is provided below:

I.D. of Outer Tube	28.22 mm [1.111"]
O.D. of Inner Tube	- 27.99 mm [1.102"]
Difference	0.23 mm [0.009"]
I.D. of Inner Tube	+ 26.21 mm [1.032"]
Addition of 0.0125 mm	+0.0125 mm [0.0005"]
Draw Plug Size	26.45 mm [1.041"]

3.3.10 The draw die hardware is shown in Figure 6.

3.3.11 The assembled draw die with the target and draw plug exposed is shown in Figure 7.

3.3.12 A cut-away sketch of the draw die assembly is shown in Figure 8.

Figure 7

Figure 8

3.3.13 Wipe off "dry graphite" and clean the inner and outer surfaces of the target with a lint-free cloth dampened with acetone.

3.3.14 Place target in vacuum chamber, evacuate for at least 15 minutes before welding (this evaporates the ethanol). If welding is not done immediately, back-fill the chamber with He for storage or store under vacuum.

3.4 Welding Target Ends

- 3.4.1 Remove target from helium atmosphere.
- 3.4.2 Prepare target for welding. Weld target ends within 2 hours of removing from helium atmosphere and machining ends. If welding cannot be performed within 2 hours, store the target in a helium atmosphere or vacuum chamber.
 - a. Oil-free machine target to correct length facing off equal amounts of material from both ends to create flat parallel surfaces (Figure 9). Clean cutting tool face with a lint-free cloth dampened in acetone prior to machining.

Figure 9

- b. Cut ~ 0.5 mm x 45° [~ 0.020 " x 45°] chamfer on the O.D. and I.D. surfaces after facing.
 - c. After machining, avoid touching the ends of the target. Use lint-free gloves when handling or wrap in a paper towel. Wipe ends with a lint free cloth dampened with ethanol. Any dirt on the welding surface has the potential to create pinholes during welding.
 - d. Place the set of outer chills approximately 1 wall thickness below end of target (Figure 10).

Figure 10

- e. Place the set of inner chills approximately 1 wall thickness below the end of the target (Figure 10). The inner/outer chill set is shown in Figure 11

Figure 11

- 3.4.3 TIG weld the target ends closed (Figures 12 & 13) with a single fusion pass using the following parameters:
- AC current full amperage ~50 A.
 - 100% argon cover gas at ~25 scfm.
 - #4 gas cup
 - 1/16" pure tungsten electrode ground to a sharp point
 - Target rotated at ~ 3 rpm during welding
 - Strike the arc on the O.D. chill and move to the aluminum tubes while bringing the weld arc up to full amperage
 - Electrode to work gap 1/32" – 1/16"
- 3.4.4 Remove weld chills immediately and allow them to cool.
- 3.4.5 Weld the opposite end of the target closed using the same method.
- 3.4.6 Verify that the target fits on its irradiation holder.

Figure 12

Figure 13

- 3.5 Target Leak Test
- 3.5.1 Leak test assembled target in accordance with MURR Irradiations Procedure, IRR-PSO-105, "Encapsulation Leak Check."
- 3.6 Weld Repair
- 3.6.1 Thoroughly dry the target by vacuum and/or heating prior repair.
- 3.6.2 Place OD and ID weld chills approximately 3 wall thickness below the end of the targets.
TIG weld any holes closed by repeating Step 3.4.3.
- 3.6.3 Remove weld chills immediately and allow them to cool.
- 3.6.4 Repeat helium leak test.
- 3.7 Cleaning Assembled Target
- 3.7.1 Thoroughly rinse in running water.
- 3.7.2 Thoroughly wipe using a lint-free cloth dampened with acetone.
- 3.7.3 Wrap in a clean paper towel and store.

Attachment 1

Data Sheet

ANL Rolled 5-g LEU-Foil Thickness Measurements

Parent LEU-Foil I.D.: 08-3-2

Calculated Mass of 5-g Piece: $2.54 \text{ cm} \times 7.62 \text{ cm} \times 0.0135 \text{ cm (ave.)} \times 19.0 \text{ g/cm}^3 = 5.0 \text{ g}$

Thickness of 5-g Piece (Arithmetic Ave.): 0.0054"

<div> <div>1.0 in</div> <div>2.54 cm</div> </div>	4.70 mils	4.95 mils	5.15 mils	5.45 mils	5.45 mils	5.50 mils
	5.15 mils	5.30 mils	5.40 mils	5.55 mils	5.70 mils	5.80 mils
	5.10 mils	5.30 mils	5.40 mils	5.55 mils	5.80 mils	5.80 mils
<div> <div>3.0 in</div> <div>7.62 cm</div> </div>

Actual Mass: 4.59 g Balance I.D.: S/N 1126282009
(Step 2.2.a) (ANL Balance)

Weight of Ni-foil Wrapped U-Foil: 5.36 g
(Step 2.3.h)

Weight of Ni-Foil: 0.77 g
(Step 2.3.i)

Attachment 2

Determination of Draw Die Plug Size

I.D. of Outer Tube (Average of 3 Measurements)	1.1130
O.D. of Inner Tube Ends (Average of 2 Measurements)	1.1012
Difference	0.0118
I.D. of Inner Tube (Average of 3 Measurements)	1.0318
Sum (I.D. of Inner Tube + Difference)	1.0436
Addition of 0.0125 mm (0.0005")	0.0005
Calculated Draw Plug Size	1.0441
Draw Plug Size to be Used	1.044

Attachment 3

Critical-cleaning detergents for laboratory, healthcare and industrial applications

30 Glenn Street
White Plains NY 10603 USA
Tel. 914.948.4040
Fax. 914.948.4088

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Acid Cleaner and Detergent

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- Use to pass your cleaning validation tests for lab accreditation and plant inspection approval
- Contains acid ingredients for high performance removal of metal oxides, salts and inorganic residues combined with full detergent capability

Used to clean: Dairy equipment, laboratory ware, sampling apparatus, lab stills, clean rooms, optical parts, electronic components, pharmaceutical apparatus, cosmetics manufacturing equipment, metal forgings, stampings and castings, industrial parts, pipes, tanks, boilers and reactors. Authorized by USDA for use in federally inspected meat and poultry plants. Passes inhibitory residue test for water analysis. FDA certified. Recommended for laser chiller cleaning and maintenance to remove scale.

Used to remove: Scale, metal oxides, metal complexes, trace inorganics, milkstone, soil, grit, buffing compound, grime, grease, fats, oils, particulates, deposits, chemical and solvents.

Surfaces cleaned: Corrosion inhibited formulation recommended for glass, metal, stainless steel, porcelain, ceramic, plastic, rubber and fiberglass. Can be used on soft metals such as copper, bronze, aluminum, zinc and magnesium if rinsed promptly. Corrosion testing may be advisable.

Cleaning method: Soak, brush, sponge, cloth, ultrasonic, flow through clean-in-place. Will foam—not for spray or machine use.

Directions: Make a fresh 1 - 2% solution (3 - 5 Tbsp. per gal., 1 1/4 - 2 1/2 oz. per gal. or 10 - 20 ml per liter) in cold, warm or hot water. If available, use hot water. For difficult soils, raise water temperature and use more detergent. Clean by soak, circulate, wipe or ultrasonic method. Wear protective gloves and eyewear. Not for spray machines, will foam. RINSE THOROUGHLY—preferably with running water. For critical cleaning, do final or all rinsing in distilled, deionized or purified water. For food contact surfaces, rinse with potable water. Used on a wide range of glass, ceramic, plastic and metal surfaces. Corrosion testing may be advisable.

Available in Convenient Sizes:

	Alconox Cat. #
Case 4 x 1 Gallons	1801
15 Gal. Drum	1815
30 Gal. Drum	1830
55 Gal. Drum	1855
1 gal. makes 50 gal. cleaning solution

Citranox is available from leading laboratory, hospital, clinical and industrial suppliers. To find a distributor for Alconox, Inc. detergents, visit "Find Dealer" at the website. To request FREE samples, visit Sample Request at www.alconox.com, write or call Alconox, Inc. today.

Attachment 3

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(e-mail) cleaning@alconox.com

(URL) <http://www.alconox.com>

- distributors
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- free samples
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Fax Document #0418

Citranox Technical Bulletin

PHYSICAL DATA

	TYPICAL VALUE
pH of 1% solution	2.5
Specific gravity (g/ml)	1.10
Density (lbs./gal.)	9.15
Flash Point (degrees F)	None
Phosphate Content (as Phosphorus)	0%
Organic Carbon (% calculated w/w)	17%
Fragrance Content	0%
Surface Tension 1% Sol'n (Dyne/cm)	32
Color: Pale Yellow	
Form: Liquid	
Solubility in Water: Completely soluble in all proportions	
Hard Water Effectiveness: Highly Effective	
Biodegradability: Biodegradable	
Foam Tendency: High Foaming	
Shelf Life: Two years from the date of manufacture	

Chemical Description

Citranox contains a blend of organic acids, anionic and non-ionic surfactants and alkanolamines. Citranox is phosphate free.

Cleaning Validation Methods:

Test a parameter of rinse water before and after rinsing the cleaned surface, or test the clean surface. No significant change in the parameter indicates no detectable residue. Parameters measured include: pH, conductivity, UV, TOC, HPLC, anionic surfactant concentration using inexpensive detergent water testing kits, surface tension, and surface analysis. For details see the Pharmaceutical Cleaning Validation Reference at www.alconox.com

Health Safety Information:

OSHA Hazardous Ingredients: None

RCRA Hazard Class: Non-hazardous

EPA Priority Pollutants: None

DOT Hazard Class: Non-hazardous

Flammability: Non-flammable

Latex Content: None in detergent, packaging materials or adhesives.

Oral Toxicity: LD₅₀ > 5000 mg/kg oral rat

No ingredient defined as an oral toxicant by OSHA

Eye Irritation: Serious potential for eye irritation if not rinsed

Inhalation Toxicity: Non-irritating

VOC Content: 0%

Carcinogenicity: NTP = No IARC = No OSHA = No

All ingredients in Citranox are listed in TSCA inventory. USDA NSF cat A3

Precautions:

No special precautions other than good industrial hygiene and safety practices employed with any industrial chemical (see Directions). A Material Safety Data Sheet is available at www.alconox.com or by calling fax-on-demand at 914-948-4040 and following the prompts. Use fax document #0318.

Contact Alconox, Inc. for purchase specifications. Information presented is typical. Typical data is not a specification.

While the information in this report should not be considered to be a product warranty, we urge you to investigate, test and verify the suitability of Alconox detergents for your specific application. We, of course, can not give permission to use, or recommend the use of, our detergents where they infringe patents. No representation or warranty is made as to the safety of products or materials mentioned under the Federal Food Additives Amendment of 1958.

Attachment 4

ANL Lab Notebook

Attachment 4

ANL Lab Notebook

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2139

MURR LEU FOIL SPECIFICATIONS

6/12/08

Attachment 1 Rolled LEU Data Sheet

5-g ANL Rolled LEU-Foil Thickness Measurements

Parent LEU-Foil I.D.: 08-3-2

Parent LEU-Foil Mass: 4.59 gr

Parent LEU-Foil Length: 2.99 in

Parent LEU-Foil Width: 0.99 in

Calculated Mass of 5-g Piece: 2.54 cm x 7.62 cm x 0.0135 cm (ave.) x 19.0 g/cm³ = 5.0 g

Thickness of 5-g Piece (Arithmetic Ave.): 0.005392 in

Actual Mass:

Balance I.D.:

Insert a photo of the rolled foil.

Weight of Ni-foil Wrapped U-Foil: _____ g
(Step 2.3.f)

Weight of Ni-Foil: _____ g
(Step 2.3.g)

LEU Avg thickness 0.005392
 2X Ni foil thickness 0.0012
 0.006592
 0.0005 -
 → 0.00609 ± 0.0003"
 Groove Depth

Attachment 4

ANL Lab Notebook

59

2139
MURR TUBE MEASUREMENTS
6/13/08

ROLLED LEU

CALCULATION ON DRAW DIE SIZE

ID of Outer Tube	1.1130
OD of Inner Tube	1.1012
Difference	0.0118
ID of Inner Tube	1.0318
Sum	1.0436
Additional 0.01 mm (0.0005")	0.0005
Draw Plug Size	1.0441
Selected Draw Size	1.044

Name of Target

08-3-2

Date Assembled

6/16/08

1.0316	1.1012
Inner Tube	
1.0320	1.0897
1.0319	1.10125
1.0318	1.1012
Average	

1.1131
Outer Tube
1.1131
1.1129
1.1130
Average

Inner Tube After Drawing
Average

Attachment 5

Technical Information for Neolube

Neolube No.1 Lubricant - Colloidal Graphite in ISOPROPANOL manufactured by Huron Industries, Inc Page 1 of 4

[link to Home Page](#)

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Neolube No.1 Lubricant

[MSDS](#)

TECHNICAL INFORMATION SHEET

NEOLUBE NO. 1 LUBRICANT - COLLOIDAL GRAPHITE IN ISOPROPANOL

APPLICATION: NUCLEAR POWER GENERATING PLANTS, NUCLEAR REACTORS, COMMERCIAL AND NAVAL

Neolube No. 1 is a dry film, conductive lubricant, used extensively at nuclear power generating plants and other nuclear facilities as an anti seize compound, thread lubricant and for lubricating moving parts and rubbing surfaces.

The composition of this material is 99% pure furnace graphite particles, a thermoplastic resin and isopropanol. The material has excellent radiation resistance and high chemical purity. The thin, non corrosive film of **Neolube No. 1** prevents seizing, fretting, galling and resists abrasion. It is easy to apply by spray, dip or brush and has excellent adhesion after a fast air dry. The dry film of **Neolube No. 1** will not migrate and it is non freezable.

Neolube No. 1 has satisfied the stringent requirements for lubricating the internal and auxiliary equipment mechanisms of commercial and naval nuclear reactor systems. **Neolube No. 1** allows easy assembly, trouble-free operation and non destructive disassembly. **Neolube No. 1** provides non corrosive, dry adherent lubrication for metal parts with limited clearances in applications where control of impurities is required.

Neolube No. 1 is **NOT RECOMMENDED FOR LUBRICATING THREADS IN THE REACTOR PRIMARY CONTAINMENT AREAS**, where operating temperatures for the fittings are greater than 400°F. **Neolube No. 650** is recommended for use in containment and/or secondary side in nuclear applications.

A Certificate of Quality Conformance and Analysis is available for each lot upon request.

Neolube No. 1 has been used successfully in applications where it has withstood radiation levels of 1×10^9 rads.

Physical and Chemical Requirements	MIL-L-24131C
Total Solids Content, weight percent	3.3% " 0.50%
Graphite Content, percent of total solids	75% " 5%
Particle size, microns	
Maximum dimensions of 90% of the particles	4 Microns
Maximum dimensions of any particle	10 Microns
Ash, weight percent, maximum on total solids	0.75%
Fluorine, parts per million, maximum on total solids	20 PPM
Chlorine, parts per million, maximum on total solids	200 PPM
Sulfur, parts per million, maximum on total solids	200 PPM
Lead, parts per million, maximum on total solids	150 PPM
Film Properties (regular & irregular surfaces):	
Adherence	The coated surface will be dry and will not become exposed when subjected to light abrasion.

<http://www.newmantools.com/chemicals/neolube1pds.htm>

10/17/2008

Attachment 5

Technical Information for Neolube

Neolube No.1 Lubricant - Colloidal Graphite in ISOPROPANOL manufactured by Huron Industries, Inc Page 2 of 4

Spalling	Film continuity will not be broken, and metal surfaces will not be exposed.
Appearance	Dry, nonoily.
Odor	Characteristic of isopropanol, no odor of halogenated solvents will be detected.

Mercury: Instruments and equipment containing mercury or compounds of mercury were not used in the manufacture and packaging of the lubricant, nor in testing and inspection, unless samples were discarded after the test. During the manufacturing processes, tests, and inspection, **Neolube No. 1** did not come in contact with mercury or any of its compounds nor any mercury-containing devices employing a single boundary of containment. Compounds containing boron were not used in cleaning, processing equipment, or containers.

There is no intentional addition of low melting point metals such as lead, bismuth, zinc, mercury antimony, cadmium, tin, silicon, gallium, indium or arsenic to this product; nor of copper or silver.

Physical Properties:

Lubricant	Processed micro graphite	Service Temperature	400°F (204°C)	
Binder	Thermoplastic Resin	Intermittent Temperature	850°F (454°C)	
Diluent	Isopropanol	Color	Black	
Consistency	Liquid	Solids Content	3.30% " 0.50%	
Density	6.6 lb./gal (0.791 kg/l)	Flash Point	52°F (11°C)	
Shelf Life	No limit in a closed container	Coefficient of Friction	0.15 (Static)	

Dilution:

Neolube No. 1, is supplied in a ready-for-use form conforming to the requirements of MIL-L-24131C. If further dilution is required by the application, add isopropanol while stirring thoroughly.

Usage:

Substrates should be clean and dry before application. A solvent wipe and air dry is usually sufficient. For critical applications requiring maximum adhesion, the following pretreatment is recommended:

Steel	Degrease, sand or vapor blast and/or phosphate coat.
Aluminum	Degrease, sand or vapor blast, degrease, Meta Etch or anodize.
Stainless Steel	Degrease, sand or vapor blast and/or Granodine 16 (Amchem)
Copper Alloys	Degrease, sand or vapor blast, degrease, dilute nitric acid dip and water rinse.
Rubber	Clean with appropriate solvent.
Plastic	Sand lightly, wipe with appropriate solvent.

NOT RECOMMENDED FOR USE IN OXYGEN SYSTEMS

Application:

Shake or agitate well before using. Apply with the brush in cap applicator or by conventional spray, brush or dip methods.

Cure Time:

Material dries in approximately five minutes to the touch depending on temperature and humidity. Full cure properties up to two hours depending on thickness, temperature and humidity.

Packaging:

Neolube No. 1 is packaged in 2 ounce and 8 ounce non halogenated plastic bottles with a brush in cap applicator.

Qualification Approval: Approving organization is the Naval Ship Engineering Center, Hyattsville, Maryland 20782. The date of the approval letter is 21 June 1974, Test Report Number (QPL) 10744.

PLEASE NOTE: ANEOLUBE PRODUCTS ARE NOT CONSIDERED SAFETY-RELATED GOODS. AS SUCH, THEY ARE NOT DESIGNED, FABRICATED, HANDLED, SHIPPED, STORED, ETC., UNDER A QUALITY ASSURANCE PROGRAM THAT COMPLIES WITH THE REQUIREMENTS OF 10CFR50, APPENDIX B, 10CFR21, OR ANSI STANDARDS.@

The data contained herein are furnished for information only and are believed to be reliable. We cannot assume responsibility for the results obtained by others over whose methods we have no control. It is the user's responsibility to decide suitability for the user's purchase of any production methods mentioned herein and to adopt such precautions as may be advisable for the protection of property and of persons against any hazards that may be involved in the handling and use thereof. Considering the above, Huron Industries, Incorporated specifically disclaims all warranties of merchantability or fitness for a particular purpose arising from sale or use of Huron's products. Huron Industries, Inc., specifically disclaims any liability for consequential or incidental damages of any kind, including lost profits. The discussion herein of various processes or compositions is not being interpreted as representation that they are free from domination of patents owned by others or as a license under any Huron Industries' patents that may cover such processes or compositions. We recommend that each prospective user test the proposed application to determine its suitability for the purposes intended before incorporating any product or application in its manufacturing process using this data as a guide. This product may be covered by one or more United States or foreign patents or patent applications. The recording of false, fictitious or fraudulent statements or entries on this document may be punished as a felony under Federal Statutes including Federal Law, Title 18, Chapter 47.

<http://www.newmantools.com/chemicals/neolube1nds.htm>

10/17/2008

B-3 ANNULAR TARGET NUMERICAL MODELING UPDATE

Annular Target Numerical Modeling Update

By Srisharan Garg Govindarajan

Until now all the finite element modeling was performed on the 2-D geometry of the annular target. This helped to understand the behavior of the target during irradiation and establish that the use of the low enriched uranium foil (LEU) based annular target is unlikely to compromise on the reactor safety. Though the 2-D models are helpful in providing a basic idea of the target behavior, they cannot be used to explore the fission gas release, uranium swelling and the stresses induced in the welds due to radiative heating. For this purpose it was required to build a 3-D finite element model of the annular target in Abaqus FEA [1].

3-D Modeling in Abaqus

The 3-D annular target model was built in Abaqus FEA [1] as illustrated in Fig 1. The inner tube has a recess cut on its surface along a length of 100 mm while the overall length of the inner tube is 152 mm. The foil is of open cross section (Fig 1) and its length is equivalent to the length of the recess. The length of the outer tube is 152 mm and equivalent to that of the inner tube. The assembled annular target with the LEU foil is illustrated in Fig 2. It was assumed that perfect contact exists at the interface of the parts.

Figure 1. The inner tube, foil and the outer tube before assembly.

Figure 2. Assembled annular target with the LEU foil sandwiched between the inner and outer aluminum cladding.

Boundary Conditions and Constraints

To numerically simulate the welded ends of the annular target a 'tie' constraint was applied in Abaqus FEA [1]. The tie constraint allows 2 regions to be fused irrespective of their meshes. Figure 3 illustrates the regions of the target to which the tie constraint has been applied. The mechanical boundary condition was applied to the model by fixing one end of the target as illustrated in Fig 4.

To simulate water cooling in the reactor, a heat transfer coefficient of $19000 \text{ W/m}^2 \text{ K}$ was used on the inner and outer regions of the inner and outer tubes respectively. The cooling water temperature was taken to be 323 K .

Figure 3. Tie constraint applied to the edges to simulate the welded regions.

Figure 4. Mechanical boundary condition applied to one end of the annular target.

Thermal Load

An heat generation rate of $1.6 \times 10^{10} \text{ W/m}^3$ was applied to the LEU foil to help simulate radiative heating in the target. This thermal load is roughly equivalent to a heat flux of 100 W/cm^2 from each side of the foil.

Finite Element Mesh

The assembled annular target consisted of 614,010 elements and 667,416 nodes with 15 elements through the thickness of each part. The finite element mesh is illustrated in Fig 5. Once the load, boundary conditions, constraints and contact interactions were specified the model was run. It took approximately 4 weeks to converge. The temperature distribution in the annular target is as illustrated in Fig 6. The portion of the tubes in contact with the region occupied by the foil has the maximum temperature.

Figure 5. Finite element mesh of the annular target assembly.

Figure 6. Temperature distribution contour of the annular target.

Since the 2D modeling results have already been verified with the help of analytical models [2], it was decided to compare the 3D modeling numerical results against the 2D modeling results. To do this a cut was made perpendicular to the longitudinal direction, right in the middle of the target. The cross sectional radial results were obtained and compared with the 2D models as illustrated in Fig 7. The results suggest that the 3D radial stress results are within 6% of the radial stress results from the 2D model while the radial temperatures are within 0.5 %.

Figure 7. Percentage comparison between 2D and 3D models.

In the absence of a 3D analytical model to validate the numerical results, Fig 7 illustrates that the current model is likely to provide accurate results. With a working 3D numerical model in place, the fission gas pressure and uranium swelling parameters can be added to the model.

Fission Gas Released from an Irradiated U-Foil

Reference [3] provides experimental data for the emission of rare gases from Uranium during neutron irradiation. The paper provided data for Xe^{135} , Kr^{35m} , Xe^{133} and the data showed Xe^{133} to be the largest contributor. These experiments were conducted using a 123 μm natural uranium foil, with an enrichment of 0.72% at a neutron flux in the range (1- 5) $\text{n/cm}^2\text{-s}$. The emission rate for these gases can be obtained from Table 3 in [3]. These emission rates can be used to perform the calculations to determine the volume of fission gas released as illustrated in Fig 8.

<p>Amount of substance for each gas can be found, (mol/s), $n = \frac{\text{EmissionRate}}{\text{Avogadro'sNumber}}$</p> <p>Volume flow rate of fission gas ($\frac{\text{m}^3}{\text{s}}$), $v = \frac{n * R * T}{P}$</p> <p>Total volume flow rate of fission gas ($\frac{\text{m}^3}{\text{hour}}$), $V_{\text{rate}} = [\sum (v_{\text{Xe}^{135}} + v_{\text{Kr}^{35m}} + v_{\text{Xe}^{133}})] * 3600$</p> <p>Total irradiation time (t) = 200 hours</p> <p>Total volume over the entire irradiation time (μl), $V_{\text{irradiation}} = (V_{\text{rate}} * t) * 10^9$</p> <p>Total volume of fission gas released (μl), $V_{\text{total}} = V_{\text{irradiation}} * \text{neutron flux ratio} * \text{ratio enrichment}$</p> <p>For a thermal neutron flux of 1E 12 $\text{n/cm}^2\text{-s}$ (lower bound), neutron flux ratio of 300, temperature of 573 K, pressure of 101325 Pa and an enrichment of 0.72 %, </p>	<p>$V_{\text{total}} = 0.0030 \mu\text{l}$</p>	<p>← This lower bound estimate for natural uranium is taken as the base case</p>
---	--	--

Figure 8. Steps to determine the volume of fission gas released.

The base case calculations can be applied to the LEU which has an enrichment of 19.75 %. By using this method it is easy to determine the volume of Kr^{35m} , Xe^{133} and Xe^{135} released for any magnitude of neutron flux, irradiation time and enrichment. The experiments that were conducted only measured the release of

Kr-85m, Xe-133, and Xe-135. However, other Kr and Xe isotopes are released as well [4]. Hence a method to estimate the total volume of gas released while taking all the isotopes into consideration needs to be developed.

Future Work

- Continue working with the 3D numerical model, add the fission gas and uranium swelling pieces.
- Simultaneously work on the fission gas release hand calculations to develop a method that predicts the volume of fission gas released by taking into account all the isotopes of the gases that are released.

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- [1] "Abaqus FEA", Version 6.10, D S Simulia. © Dassault Systemes 2002-2012.
- [2] Govindarajan, S.G, Solbrekken, G.L., Allen , C.W., " Thermal-Mechanical Analysis of a Low-Enriched Uranium Foil Based Annular Target for the Production of Molybdenum-99",*Proceedings of the ASME International Mechanical Engineering Congress and Exposition ,IMECE 2012 , Houston , Texas , Nov 09-15 , 2012 (To be presented)*.
- [3] Proceedings of the Second United Nations International Conference on the Peaceful Uses of Atomic Energy, Vol. 5, UNITED NATIONS, Geneva, September 1958.
- [4] Allen C.W, " Production of fission product Mo-99 using the LEU-modified Cintichem process- Calculation of fission gas inventory produced during U-foil irradiation ", TDR-0111, University of Missouri Research Reactor, August, 2007.

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APPENDIX C:
OAK RIDGE NATIONAL LABORATORY

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C-1 COMBINED ORNL SAFETY DOCUMENTS

Department of Energy

ORNL Site Office
P.O. Box 2008
Oak Ridge, Tennessee 37831-6269

September 3, 2013

Dr. Thomas E. Mason, Director
Oak Ridge National Laboratory
UT-Battelle, LLC
Post Office Box 2008
Oak Ridge, Tennessee 37831-6255

Dear Dr. Mason:

**DEPARTMENT OF ENERGY (DOE) APPROVAL OF SAFETY BASIS
SUPPLEMENT (SBS) SBS/3525/2013-01 REVISION 0 AND TECHNICAL
SAFETY REQUIREMENTS (TSR) REVISION (ORNL/3525/TSR REVISION 6)**

- References:
1. 08/19/2013 letter, Joseph N. Herndon to Johnny O. Moore, subject, "Contract DE-AC05-00OR22725, Resubmittal of Safety Basis Supplement for Molybdenum-99 Recovery at Building 3525 and TSR Revisions"
 2. 05/31/2013 letter, Joseph N. Herndon to Johnny O. Moore, subject, "Contract DE-AC05-00OR22725, Safety Basis Supplement for Molybdenum-99 Recovery at Building 3525 and TSR Revisions"

This letter transmits my approval of SBS/3525/2013-01 Revision 0, *Electro-Dissolution of Uranium Metal for Molybdenum-99 Recovery Project at Building 3525*, and associated TSR (ORNL/3525/TSR Revision 6) Revision. A Safety Evaluation Report (SER) Supplement documenting my basis for approval is included with this letter. The SER Supplement is to be considered a part of the safety basis for the facility.

There is one condition of approval. The Nuclear Criticality Safety Analysis (NCSA) for the facility needs to be revised to protect the assumption regarding criticality in the hazards analysis of the SBS. You have committed to revising the NCSA to limit the East Hot Cell to less than 700 grams uranium-235 fissile equivalent material (FEM) with no restrictions on liquids being in the hot cell and notifying me of its revision prior to implementation of the SBS. In addition to being notified when the NCSA has been revised, a copy of the NCSA revision is to be provided to me prior to implementation of this SBS.

You requested that SBS/3525/2013-01 Revision 0 and ORNL/3525/TSR Revision 6 be implemented prior to accepting irradiated uranium metal for processing to recover the Molybdenum-99 or accepting unirradiated uranium metal in the facility. I concur with that request.

Dr. Thomas E. Mason

-2-

September 3, 2013

If there are any questions or additional information is required, please contact Martha Kass at 576-0717 or Randy Persinger at 241-6588.

Sincerely,

Johnny D. Moore, Manager
ORNL Site Office

Enclosure

cc w/enclosure:
Carol L. Sohn, SC-3
Robert S. McKeehan, ORNL
John H. Platfoot, ORNL

September 3, 2013

Dr. Thomas E. Mason, Director
Oak Ridge National Laboratory
UT-Battelle, LLC
Post Office Box 2008
Oak Ridge, Tennessee 37831-6255

Dear Dr. Mason:

**DEPARTMENT OF ENERGY (DOE) APPROVAL OF SAFETY BASIS
SUPPLEMENT (SBS) SBS/3525/2013-01 REVISION 0 AND TECHNICAL
SAFETY REQUIREMENTS (TSR) REVISION (ORNL/3525/TSR REVISION 6)**

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Molybdenum-99 Recovery at Building 3525 and TSR Revisions"

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Dr. Thomas E. Mason

-2-

September 3, 2013

If there are any questions or additional information is required, please contact Martha Kass at 576-0717 or Randy Persinger at 241-6588.

Sincerely,

Johnny O. Moore, Manager
ORNL Site Office

Enclosure

cc w/enclosure:
Carol L. Sohn, SC-3
Robert S. McKeehan, ORNL
John H. Platfoot, ORNL

New Commitment: Yes ☒ No ☐
Commitment to Action: # _____
Approved for Release: _____ (initiator initial)

R. Persinger:1-6588:AM:4-6363:8/26/13:8313 1.AM

SC-OSO
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Initials/Sig.
HRP <i>HRP</i>
Date: <i>8/27/13</i>
Rtg. Symbo
SC-OSO
Initials/Sig.
<i>MR</i>
Date: <i>9/3/2013</i>
Rtg. Symbo
SC-OSO
Initials/Sig.
<i>MAW</i>
Date: <i>9/3/13</i>
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**SAFETY EVALUATION REPORT FOR THE
SAFETY BASIS SUPPLEMENT
FOR THE
IRRADIATED FUELS EXAMINATION LABORATORY
ELECTRO-DISSOLUTION OF URANIUM METAL FOR MOLYBDENUM-99
RECOVERY PROJECT AT BUILDING 3525**

SAFETY BASIS SUPPLEMENT, SBS/3525/2013-01 R0

**Supplement 7 to the SAFETY EVALUATION REPORT FOR THE
IRRADIATED FUELS EXAMINATION LABORATORY,
BUILDING 3525
AT THE
OAK RIDGE NATIONAL LABORATORY
SAFETY ANALYSIS REPORT, ORNL/3525/SAR R1C
AND
TECHNICAL SAFETY REQUIREMENTS, ORNL/3525/TSR R2C**

REVISION 0

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ACRONYMS

COA	Conditions of Approval
DOE	Department of Energy
DSA	Documented Safety Analysis
FEM	Fissile Equivalent Material
HEGA	High Efficiency Gas Absorber
HEPA	High Efficiency Particulate Air
LCO	Limiting Condition for Operation
LEU	Low Enriched Uranium
NCSA	Nuclear Criticality Safety Analysis
OOD	Operations and Oversight Division
ORNL	Oak Ridge National Laboratory
OSO	ORNL Site Office
SAC	Specific Administrative Control
SAR	Safety Analysis Report
SBS	Safety Basis Supplement
SER	Safety Evaluation Report
SME	Subject Matter Expert
SSCs	Structures, Systems, and Components
TSR	Technical Safety Requirements
UT-Battelle	UT-Battelle, LLC

1.0 EXECUTIVE SUMMARY

Introduction

This Safety Evaluation Report (SER) Supplement has been prepared to document the review and the basis for the Department of Energy's (DOE) approval of the Oak Ridge National Laboratory's (ORNL) Safety Basis Supplement (SBS) for Electro-Dissolution of Uranium Metal for Molybdenum-99 Recovery Project at Building 3525 including Documented Safety Analysis (DSA) and Technical Safety Requirements (TSRs) revisions as required by 10 CFR 830, Subpart B. SBS/3525/ORNL/2013-01 Rev. 0 documents the hazard and accident analysis for this modification and determines preventive/mitigative controls in accordance with the safe harbor methodology (DOE-STD-3009) of 10 CFR 830. This SBS was submitted by UT-Battelle, LLC (UT-Battelle), by letter dated May 31, 2013, from Joseph N. Herndon to Johnny O. Moore, subject, "Contract DE-AC05-00OR22725, Safety Basis Supplement for Molybdenum-99 Recovery at Building 3525 and TSR Revisions" (Reference 6.1) to obtain DOE's approval of the hazards and accident analysis and the controls. A revised submittal (Reference 6.2) was submitted by letter dated August 19, 2013, from Joseph N. Herndon to Johnny O. Moore, subject, "Contract DE-AC05-00OR22725, Resubmittal of Safety Basis Supplement for Molybdenum-99 Recovery at Building 3525 and TSR Revisions," ORNL/3525/TSR Rev. 6 and Rev. 7, incorporating the controls associated with this SBS, were also submitted for DOE approval. ORNL/3525/TSR Rev. 6 incorporates the required changes resulting from SBS/3525/2013-01 Rev. 0. ORNL/3525/TSR Rev. 7 includes the controls from SBS/3525/2013-01 Rev. 0 in addition to changes associated with the Building 3525 DSA annual update submitted by letter dated March 21, 2013, from Joseph N. Herndon to Johnny O. Moore, subject, "Contract DE-AC05-00OR22725, Annual Update to the SAR, Annual USQD Report, and Update of Authorization Agreement for Building 3525" (Reference 6.3). Review and basis for approval of ORNL/3525/TSR Rev. 7 will be included with the SER Supplement for the Building 3525 DSA Annual update.

This SBS provides the safety analysis and controls for recovery of molybdenum-99 from small quantities of irradiated uranium metal by means of an electrolytic dissolution process that will take place in the facility's East Hot Cell. Modification of the radioactive materials allowed in the facility to include new material forms, irradiated, and unirradiated uranium metal, is required. The activity is a proof of concept activity. As such, this SBS will have a limited duration. However, the material limits for irradiated and unirradiated uranium, incorporated into the Specific Administrative Control (SAC) for radioactive material, and the safety analysis will be incorporated into the DSA for Building 3525 (ORNL/3525/SAR). The TSR Revision incorporates the required controls specified in the SBS, and made editorial and other changes for clarity.

Building 3525 is a DOE Hazard Category 2 non-reactor nuclear facility whose current mission involves the receipt and handling of irradiated materials, transfer of materials, packaging and shipment of materials, waste packaging, maintenance of remote equipment, and decontamination of the facility and equipment. Examination and testing

activities that occur within the facility hot cells include metrology, metallographic sample preparation, optical microscopy, scanning electron microscopy/microprobe, gamma spectrometry, and other physical properties evaluations.

Building 3525 is a two-story brick building with a partial basement with a gross floor area of about 27,000 square feet. The operating and service areas occupy about 220,000 cubic feet. The primary contained complex is an additional 29,300 cubic feet. The building is divided into two functional sections. The front section is a single-story office area. The two-story area to the immediate rear houses the cell complex, the operating areas, and other supporting activities. The identified hazards are radioactive materials (including fissionable material), radioactive surface contamination, and radioactive waste.

DSAs are reports which document the safety analysis of a nuclear facility to ensure that the facility can be constructed, operated, maintained, shut down, and decommissioned safely and in compliance with applicable laws and regulations. It is a report that summarizes the systematic identification of hazards within a facility or operation; describes and reports the analyses of the adequacy of measures taken to eliminate, control, or mitigate identified hazards; and reports the evaluation and analysis of potential accidents and their associated risks. DSAs are required by 10 CFR 830, Subpart B. A TSR is a document containing that set of limits, controls, and related actions that establish the specific parameters and requisite actions for the operation of a nuclear facility and include, as appropriate for the work and the hazards identified in the DSA: safety limits, operating limits, surveillance requirements, administrative and management controls, use and application provisions, and design features, as well as a bases appendix. TSRs are required by 10 CFR 830, Subpart B.

A review team of ORNL Site Office (OSO) personnel was formed under the lead of the OSO's Operations and Oversight Division (OOD) Director. A complete review of the SBS was performed to ascertain if the document was consistent with the requirements of DOE-STD-3009, DOE Guide 421.1-2, *Implementation Guide for Use in Developing Documented Safety Analyses to Meet Subpart B of 10 CFR 830*, and 10 CFR 830.204, and that the revision did not substantially alter the conclusions of the base SER for the Building 3525 DSA and TSR. The TSR Revision was reviewed to ensure that it captured the controls delineated in the SBS hazards and accident analysis, met the requirements of 10 CFR 830.205, and remained consistent with the Building 3525 DSA.

Conclusions

The review team has concluded the following regarding SBS/3525/2013-01 Rev. 0 and ORNL/3525/TSR Rev. 6:

- The base sections provide a basic understanding of the new activity processes and new material characteristics;
- The SBS provided an adequate hazards analysis for the activity and processes submitted for approval;

- No new safety class or safety-significant structures, systems, or components (SSCs) were determined to be needed to prevent/mitigate the accident scenarios postulated for the new radioactive material forms;
- Revision of the TSR SAC on radioactive material limits to protect the assumptions of the hazards analysis for the activity is acceptable;
- Other changes to the TSR are editorial or enhancements for clarity and are considered acceptable;
- It is recommended that a copy of the Nuclear Criticality Safety Analysis (NCSA) revision limiting the East Hot Cell to less than 700 grams uranium-235 fissile equivalent material (FEM) with no restrictions on liquids being in the hot cell be provided to the approval authority prior to implementation of this SBS; and
- The SBS and TSR Rev. 6 meet the requirements of 10 CFR 830.204 and .205.

SBS/3525/2013-01 Rev. 0 and associated TSR Revision, ORNL/3525/TSR Rev. 6, are considered acceptable with the following condition of approval (COA):

- It is recommended that a copy of the NCSA revision limiting the East Hot Cell to less than 700 grams uranium-235 FEM with no restrictions on liquids being in the hot cell be provided to the approval authority prior to implementation of this SBS.

The contractor has stated that SBS/3525/2013-01 Rev. 0 and ORNL/3525/TSR Rev. 6 will be implemented prior to accepting irradiated uranium metal for recovery of molybdenum-99 or unirradiated uranium metal in the facility for this activity. The review team concurs with this implementation timeframe.

2.0 INTRODUCTION, REVIEW PROCESS, STANDARDS, AND ACCEPTANCE CRITERIA

Introduction

UT-Battelle has submitted SBS/3525/2013-01 Rev. 0 and associated TSR Revision, ORNL/3525/TSR Rev. 6 for Building 3525 by References 6.1 and 6.2.

This SBS modifies the radioactive materials allowed to be in the facility to include new material forms, irradiated and unirradiated uranium metal, and provides limits for this material. It also analyzes the electro-dissolution process for recovery of molybdenum-99 from irradiated uranium metal. The electro-dissolution process for recovery of molybdenum-99 will be a temporary process, not to be incorporated into the DSA. However, the safety analyzes for the new material forms will be incorporated into the DSA. The TSR Revision:

1. Incorporate the required controls specified in the SBS;
2. Made miscellaneous changes, mainly editorial, to improve clarity; and
3. Limiting Condition for Operation (LCO) 3/4.3.1, Action A.2.1.1 was revised to remove a redundant action.

Safety Analysis Reports (SARs) are DSAs, which document the safety analysis of a nuclear facility to ensure that the facility can be constructed, operated, maintained, shut down, and decommissioned safely and in compliance with applicable laws and regulations. It is a report that summarizes the systematic identification of hazards within a facility or operation; describes and reports the analyses of the adequacy of measures taken to eliminate, control, or mitigate identified hazards; and reports the evaluation and analysis of potential accidents and their associated risks. DSAs are required by 10 CFR 830, Subpart B. A TSR is a document containing that set of limits, controls, and related actions that establish the specific parameters and requisite actions for the operation of a nuclear facility and include, as appropriate for the work and the hazards identified in the SAR: safety limits, operating limits, surveillance requirements, administrative and management controls, use and application provisions, and design features, as well as a bases appendix. TSRs are required by 10 CFR 830, Subpart B. This SER supplement documents the results and conclusions of the review of the submittals and provides the basis for DOE approval.

Review Team

A review team of OSO personnel was formed under the lead of the OSO's OOD Director. Team members were subject matter experts (SMEs) experienced in the review of safety basis documentation, the facility representative, and the program coordinator.

Review Process and Acceptance Criteria

Each principal review team member was to review the SBS against DOE-STD-3009-94, *Change Notice 3, Preparation Guide for U.S. Department of Energy Nonreactor Nuclear Facility Documented Safety Analysis*, the existing DSA (ORNL/3525/SAR Rev. 3a), any outstanding approved SBSs and associated SER and SER Supplements. This SER Supplement organization is based on the guidelines provided in DOE-STD-1104-2009, *Review and Approval of Nuclear Facility Safety Basis and Safety Design Basis Documents*. Coordination of comments and/or consultation with other SMEs as necessary was done by individual reviewers and the comments forwarded to the team lead.

Section 3.0 of this SER Supplement provides an evaluation of the SBS and the TSR Revision. The review and approval focuses on the adequacy of the following approval basis (DOE-STD-1104-2009) as it affects the currently approved Building 3525 DSA:

- Base Information;
- Hazard and accident analyses;
- Safety SSCs;
 - SACs;
- Derivation of TSRs; and
- Safety management program characteristics.

Office of Science Management System: *Environment, Safety, and Health*; Subject Area: *Facility Safety*; Procedure 1, *Reviewing and Approving Nuclear Facility Safety Basis Documentation*, requires the SER/SER Supplement to document (as applicable): (1) the sufficiency of the DSA for the DOE Hazard Category 1, 2, or 3 nuclear facility; (2) the extent to which a contractor has satisfied the requirements of 10 CFR 830.204 and .205; and (3) the basis for approval by DOE of the safety basis for the facility including any COAs. The base SER contains statements regarding the sufficiency of the DSA for Building 3525. As long as the SBS does not significantly change the conclusions of the base SER, this criteria will be assumed to have been met. Item Number 2 will be specifically addressed in Section 3.0 of this SER Supplement. Item Number 3 will be addressed by DOE-STD-1104-2009 approval bases listed above.

TSR approval basis are the TSR provisions themselves, i.e., adequate incorporation of the DSA/SBS hazard and accident controls.

Conclusions, recommendations, and references are provided in Sections 4.0, 5.0, and 6.0 respectively.

No changes were made to the Safety Management Program chapters by this SBS. Therefore, this area of the approval basis has been excluded, as it is not applicable.

3.0 EVALUATION

3.1 Base Information

The Base Information consists:

- Executive Summary;
- Purpose of SBS; and
- Background, consisting of Facility Overview, Facility Description, and New Activity Description.

The *Executive Summary* provides a synopsis of the SBS package including a description of the activity and the results of the hazards analysis. The *Executive Summary* is consistent with the remaining sections of the SBS and is considered acceptable.

The *Purpose of the SBS* section states that a new activity has been proposed for Building 3525 that involves receipt and processing of approximately 20-gram Low Enriched Uranium (LEU) metal foil. The section states that the SBS evaluates the radioactive material release characteristics to add new controls or confirm the existing controls to ensure the activity can be safely conducted within the facility. The *Purpose* also states that a revision was made to TSR section 5.11.1. *Radioactive Material Limit*, SAC by identifying a revised SAR Table 3-5 provided in the SBS. The *Purpose of the SBS* section is consistent with the remaining portions of the SBS and is considered acceptable.

The *Background* section provides a synopsis of the activities that take place in Building 3525. The synopsis is consistent with the approved Building 3525 DSA and is considered acceptable. The *Background* section also states that the activities addressed in the SBS are limited and will not become a permanent part of the facility with the exception of the evaluation/analysis of the uranium metal, which will be retained and incorporated into a future update of the facility DSA.

The *Facility Overview* subsection provides a brief description of ORNL and Building 3525. The descriptions are consistent with the Building 3525 DSA and are considered acceptable.

The *Facility Description* subsection describes the facility structure, provides a general description of the hot cells, and specific descriptions of the East Hot Cell and the Hot Cell Ventilation System. The facility structure description and general description of the hot cells are consistent with the approved Building 3525 DSA and are considered acceptable.

A more detailed description of the East Hot Cell is provided because the electro-dissolution equipment for the new activity will be located at Workstation 9 in this cell. The detailed description of the East Hot Cell is consistent with the approved DSA and is considered acceptable.

The Hot Cell Ventilation System is described. The majority of this system is designated as safety-significant in the approved DSA. The salient features germane to this SBS are the two High Efficiency Particulate Air (HEPA) filter banks. Each HEPA filter bank consists of two HIEPA filters. Only one HIEPA filter is required to be operational for the HEPA filter bank to perform its safety-significant function in accordance with the approved DSA and the Building 3525 TSRs (LCO 3.4.1). Either or both of the HEPA filter banks can be online for the system to be considered operational. For this new activity, one of the HEPA filters in each bank will be temporarily replaced with a High Efficiency Gas Absorber (HEGA) filter (charcoal filter) to provide defense-in-depth protection for control/mitigation of the potential release of radioactive iodine in the East Hot Cell.

The *New Activity Description* subsection provides a description of the new process and the equipment involved. The new activity is to electro-dissolve and separate Mo-99 from an approximate 20-gram LEU uranium metal foil. The following skid mounted equipment will be located in the East Hot Cell to perform this activity:

- Dissolution tank;
- Waste tank;
- Precipitation tank;
- Filtrate tank;
- Vacuum pump; and

- Transfer lines and inline components such as inline filters, valves, pressure indicators, flow indicators, and flow controllers.

A direct current power supply control unit will be located outside of the East Hot Cell with output lines passing through hot cell penetrations to the appropriate equipment. An iodine trap will be installed in the dissolution tank vent line, which will use activated carbon to trap the iodine. No credit is taken for the iodine trap in the hazards analysis.

Nitrogen gas will be supplied to the dissolution tank and precipitate tank to provide a gas purge which will sweep any hydrogen generated into the hot cell and any iodine and noble gases that may be released into the gas space through the iodine filter in the vent line.

As mentioned above, one of the HEPA filters in each HEPA filter bank will be replaced with a HEGA filter for the capture of any radioactive iodine potentially released to the hot cell. The HEGA filter has been designated as a defense-in-depth SSC. No credit has been taken in the hazards analysis for the HEGA filter to reduce the consequences of a radioactive material release. This will not affect the Building 3525 TSR LCO (3.4.1) as only one HEPA filter in each bank is required for Operability.

This activity constitutes a temporary change to the facility. At the completion of the activity, the equipment will be declared inoperable/removed from the hot cell. The HEGA filters installed in the HEPA filter banks will be removed and replaced with HEPA filters consistent with the required functional requirements of the approved DSA.

The review team has determined that the Base Information adequately describes the activities/operations for which safety basis approval is being sought. The information provides a knowledgeable reviewer sufficient background material to understand the revisions/changes to the facility safety analysis. The requirement of 10 CFR 830.204(b)(1) [Describe the facility (including the design of safety SSCs) and the work to be performed] is considered met. The Base Information is considered acceptable.

3.2 Hazard and Accident Analysis

A comprehensive listing of potential hazards associated with the electro-dissolution and separation of Mo-99 from an approximate 20-gram LEU uranium metal foil was provided. The majority of hazards were either evaluated to be not applicable or screened out as being below established levels of concern.

The hazards kept for evaluation were radiological hazards (inhalation and direct dose hazard) and fissionable material hazards. Note that radioactive iodine, due to the irradiated uranium, is included in the radiological hazards. The review

team concurs with this determination.

The facility's unmitigated hazards analysis dose consequences calculation (ORNL/3525/DAC/2007-01, "IFEL Building 3525 Unmitigated Hazard Evaluation Threshold Consequence Calculations") was revised to reflect the radioactive material forms, irradiated and unirradiated uranium metal, including release of radioactive iodine. One hundred percent of the radioactive iodine from the irradiated uranium metal was assumed to be released in the gaseous radioactive material form. Airborne Release Fractions and respirable fractions for unirradiated and irradiated uranium metal were determined and appear reasonable, conservative, and appropriate. The calculation results indicate that including the unirradiated uranium under the existing "powders and solutions" radioactive material form will bound the release characteristics. One hundred percent of the radioactive iodine (a gas) is assumed to be released. The calculation was revised to include this conservative assumption. Results of the revised calculation are reflected in the Unmitigated Hazard Evaluation discussed below.

An Unmitigated Hazard Evaluation was performed for this activity. Estimated dose consequences for off-site maximally exposed individuals, on-site worker, and facility worker were determined to be "low" for all applicable scenarios based on the calculation above. No new safety class or safety-significant SSCs were determined necessary based on the results of the unmitigated hazard evaluation. Credited control for the protection of the facility worker is the Radiological Protection Program, the Procedures and Training Program, and the Emergency Management Program. These safety management programs have been previously credited with protection of the facility worker therefore; this does not result in any new safety management programs requiring TSR coverage. The review team concurs that the controls taken credit for are appropriate and sufficient.

However, one of the unmitigated hazards analysis assumptions is there is less than 700 grams uranium-235 FEM in the East Hot Cell with no restrictions on liquids being in the hot cell. The East Hot Cell NCSA currently allows for up to 1,400 grams uranium-235 with restrictions on the amount of liquids. The NCSA has not been revised to reflect this change. The SBS states that the NCSA will be revised and the revision verified during the implementation of the SBS to ensure it reflects the assumptions of the SBS hazards analysis. However, the NCSA should have been revised to support the SBS hazards analysis prior to submission. The SBS commits to notifying the DOE Site Office when the NCSA has been revised. However, the NCSA should be reviewed by the approval authority to ensure that it supports the unmitigated hazards analysis. Therefore, it is recommended that the approval authority be provided a copy of the NCSA revision prior to implementation of this SBS. This will be recommended as a COA.

Required revisions to the DSA were included in the SBS. The changes reflected the discussion provided in the SBS and are considered acceptable.

The requirement of 10 CFR 830.204(b)(2) [Provide a systematic identification of both natural and man-made hazards associated with the facility] and (3) [Evaluate normal, abnormal, and accident conditions, including consideration of natural and man-made external events, identification of energy sources, or processes that might contribute to the generation or uncontrolled release of radioactive and other hazardous materials, and consideration of the need for analysis of accidents which may be beyond the design basis of the facility] is considered met.

The requirement of 10 CFR 830.204(b)(4) [Derive the hazard controls necessary to ensure adequate protection of workers, the public, and the environment, demonstrate the adequacy of these controls to eliminate, limit, or mitigate identified hazards, and define the process for maintaining the hazard controls current at all times and controlling their use] is considered met.

The review team considers the hazards analysis for this activity presented in the SBS acceptable.

3.3 Safety Structures, Systems, and Components

No SSCs has been designated as safety class or safety-significant as a result of the hazards analysis presented in the SBS. The review team concurs with this determination.

3.4 Specific Administrative Controls

Based on calculation ORNL/3525/DAC/2007-01, the currently approved DSA Table 3-5 (3a-²³⁸Pu-GB), which lists the radioactive material forms and their limits, needs to be revised to:

- Add irradiated uranium metal to the "contaminated combustibles or HEPA filter" radioactive material form with a note that states that any radioactive iodine needs to be considered a gaseous radioactive material form, and
- Add unirradiated uranium metal to the "powders or solution" radioactive material form.

A revised Table 3-5 was included in the SBS incorporating the required changes and is considered acceptable by the review team.

3.5 Derivation of Technical Safety Requirements

The *Hazards Analysis* section of this SBS took credit for the following items for prevention/mitigation of the evaluated scenarios:

- Revision to the SAC for limiting the radioactive materials inventory;
- Radiological Protection Program;
- Procedures and Training Program; and

- Emergency Management Program.

As stated in Section 3.4 of this SER Supplement, a revised DSA Table 3-5 has been included in the SBS. The review team has determined that the revised DSA Table 3-5 reflects the appropriate changes and is considered acceptable.

The currently approved 3525 DSA contains the Radiological Protection Program, Procedures and Training Program, and the Emergency Management Program as safety management programs needing TSR coverage. No change to the DSA is required to designate these safety management programs as needing TSR coverage.

The review team considers the *Derivation of TSRs* section of the SBS as acceptable.

3.6 Technical Safety Requirements

The following evaluation is for ORNL/3525/TSR Rev. 6. Note that Rev. 6 contains the changes required for the SBS and numerous “editorial” changes. The changes specific to the SBS will be discussed first and the editorial changes second.

Section 1.1, *Definitions*, the term “Significant Quantities of Radioactive Material,” has been revised to change the amounts for:

- Powders, solutions, or unirradiated uranium metals; and
- Contaminated combustibles, HEPA filters, or irradiated uranium metals.

The bases for the amounts are provided in calculation ORNL/3525/DAC/2007-01 and are considered acceptable.

Section 5.11.1, *Specific Administrative Controls, Radioactive Material Limit*, has been revised to reference SBS/3525/2013-01 Rev. 0 and Table 3-5 (3a-²³⁸Pu-GB-⁹⁵Mo), “Hazardous Material Listing for SAR Rev 3a and SBS/3525/2010-02, SBS/3525/2012-01, and SBS/3525/2013-01.” The *Critical Safety Function, SAC Description*, and *Basis for the SAC* were adequately revised to reflect the required items from SBS/3525/2013-01 for the material limits.

These changes meet the requirements of 10 CFR 830.205(a)(1) [Develop TSRs that are derived from the DSA] and (2) [Prior to use, obtain DOE approval of TSRs and any change to TSRs]. The revised SAC is considered acceptable by the review team.

An editorial change was made to the definition of “Calibrate/Calibration” and is considered acceptable.

LCO 3.0.3 Bases had the phrase "STANDBY" changed to "WARM STANDBY"
LCO 3/4.3.1 Bases "MODE APPLICABILITY" had the word "only" added; LCO
3/4.3.1 Bases for "ACTION STATEMENT" A.1 had an "if" corrected to "of."
These are considered editorial changes and are acceptable.

LCO 3/4.3.1, ACTION A.2.1.1 was revised to remove a statement requiring a fire watch. This is acceptable because ACTION A.2.1.3 requires establishing a fire watch. Thus a duplicate action/requirement was removed. This change is acceptable to the review team.

The above editorial changes and the change to remove the duplicate fire watch action meets the requirements of 10 CFR 830.205(a)(2).

The review team considers ORNL/3525/TSR Rev. 6 to be consistent with the SBS/3525/2013-01 Rev. 0 and considers the changes acceptable.

4.0 CONCLUSION(S)

The review team has concluded the following regarding SBS/3525/2013-01 Rev. 0 and ORNL/3525/TSR Rev. 6:

- The base sections provide a basic understanding of the new activity processes and new material characteristics;
- The SBS provided an adequate hazards analysis for the activity and processes submitted for approval;
- No new safety class or safety-significant SSCs were determined to be needed to prevent/mitigate the accident scenarios postulated for the new radioactive material forms;
- Revision of the TSR SAC on radioactive material limits to protect the assumptions of the hazards analysis for the activity is acceptable;
- Other changes to the TSR are editorial or enhancements for clarity and are considered acceptable;
- The SBS and TSR Rev. 6 meet the requirements of 10 CFR 830.204 and .205; and
- It is recommended that a copy of the NCSA revision limiting the East Hot Cell to less than 700 grams uranium-235 FEM with no restrictions on liquids being in the hot cell be provided to the approval authority prior to implementation of this SBS.

SBS/3525/2013-01 Rev. 0 does not significantly change the approval basis provided in the base SER. SBS/3525/2013-01 Rev. 0 and ORNL/3525/TSR Rev. 6 meet the requirements of 10 CFR 830.204 and .205 as appropriate.

SBS/3525/2013-01 Rev. 0 and ORNL/3525/TSR Rev. 6 are considered acceptable with the COA noted above.

5.0 RECOMMENDATION(S)

Given:

- a. SBS/3525/2013-01 Rev. 0 and ORNL/3525/TSR Rev. 6 have undergone an appropriate review, and
- b. SBS/3525/2013-01 Rev. 0 and ORNL/3525/TSR Rev. 6 establish the safety bases that are acceptable and meet the requirements of 10 CFR 830 Subpart B/DOE-STD-3009 for the activity/processes requesting approval for. There is one COA as noted in Section 4.0.

The review team recommends approval of SBS/3525/2013-01 Rev. 0 and ORNL/3525/TSR Rev. 6 with the COA described in Section 4.0.

The contractor has stated that SBS/3525/2013-01 Rev. 0 and ORNL/3525/TSR Rev. 6 will be implemented prior to receipt to accepting irradiated or unirradiated uranium metal in the facility. The review team recommends approval of this implementation scheme.

6.0 REFERENCES

- 6.1 05/31/2013 letter, Joseph N. Herndon to Johnny O. Moore, subject, "Contract DE-AC05-00OR22725, Safety Basis Supplement for Molybdenum-99 Recovery at Building 3525 and TSR Revisions"
- 6.2 08/19/2013 letter, Joseph N. Herndon to Johnny O. Moore, subject, "Contract DE-AC05-00OR22725, Resubmittal of Safety Basis Supplement for Molybdenum-99 Recovery at Building 3525 and TSR Revisions"
- 6.3 03/21/2013 letter, Joseph N. Herndon to Johnny O. Moore, subject, "Contract DE-AC05-00OR22725, Annual Update to the SAR, Annual USQD Report, and Update of Authorization Agreement for Building 3525."

SIGNATURE PAGE

This SER Supplement was principally prepared by H. Randall Persinger with input from the review team members.

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August 19, 2013

Mr. Johnny O. Moore
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Dear Mr. Moore:

Contract DE-AC05-00OR22725, Resubmittal of Safety Basis Supplement for Molybdenum-99 Recovery at Building 3525 and TSR Revisions

Reference: Letter, J. N. Herndon to J. O. Moore, "Safety Basis Supplement for Molybdenum-99 Recovery at Building 3525 and TSR Revisions," May 31, 2013

The enclosed *Safety Basis Supplement for the Electro-Dissolution of Uranium Metal for Molybdenum-99 Recovery Project at Building 3525*, (SBS/3525/2013-01, R0) is resubmitted for Department of Energy (DOE) approval (Enclosure 1). This Safety Basis Supplement (SBS) provides the safety analysis and controls for recovery of molybdenum-99 from small quantities of irradiated uranium metal by means of an electrolytic dissolution process. This SBS was initially submitted to DOE by the referenced letter. The SBS in this submittal has been revised to address informal DOE comments on the initial submittal. Those DOE comments and their resolutions are provided in Enclosure 2. Additionally, Revisions 6 and 7 of the Building 3525 Technical Safety Requirements (TSR) document were submitted with the referenced letter. Those documents are also resubmitted (Enclosures 3 and 4, respectively) with changes made to address two of the DOE comments.

The SBS and the two revisions of the TSR will be implemented as described in the referenced letter. This submittal has been discussed with Randy Persinger. If you have any comments or questions concerning the enclosed documents, please contact John Platfoot at 576-9571.

Sincerely,

Joseph N. Herndon, Director
Environment, Safety and Health

JNH:lah

Enclosures (4)

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Page 2
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ENCLOSURE 1
BUILDING 3525
SAFETY BASIS SUPPLEMENT
SBS/3525/2013-01

SAFETY BASIS SUPPLEMENT
FOR THE
ELECTRO-DISSOLUTION OF URANIUM METAL FOR MOLYBDENUM-99
RECOVERY PROJECT AT BUILDING 3525

Date Approved: .

Date Effective:

Prepared by the
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SAFETY BASIS SUPPLEMENT

ELECTRO-DISSOLUTION OF URANIUM METAL FOR MOLYBDENUM-99

RECOVERY PROJECT AT BUILDING 3525

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DEPARTMENT OF ENERGY APPROVALS
SAFETY BASIS SUPPLEMENT
ELECTRO-DISSOLUTION OF URANIUM METAL FOR MOLYBDENUM-99
RECOVERY PROJECT AT BUILDING 3525

Approved by:

Department of Energy

9/3/13
Date

REVISION LOG		
REVISION	DESCRIPTION	DATE APPROVED
0	Initial issue.	

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LIST OF ACRONYMS AND ABBREVIATIONS

<u>Term</u>	<u>Definition</u>
ALARA	As Low As Reasonably Achievable
ANSI	American National Standards Institute
CFM	Cubic feet per minute
CFR	Code of Federal Regulations
DAC	Design Analysis Calculation
DOE	Department of Energy
PEM	Fissionable Equivalent Mass
GMR	Glove Maintenance Room
HEGA	High efficiency gas adsorber
HEPA	High efficiency particulate air
HESA	Hot Equipment Storage Area
HFIR	High Flux Isotope Reactor
Hz	Hertz
IFEL	Irradiated Fuels Examination Laboratory
IMGA	Irradiated Microsphere Gamma Analyzer
LEL	Lower Explosive Limit
ICO	Limiting Condition for Operation
LCS	Limiting Control Setting
LLC	Limited Liability Company
LLV	Low Level Ventilation
MAR	Material at Risk
MCC	Motor Control Center
MHE	Mitigated Hazard Evaluation
MTS	Methyltrichlorosilane
NCSA	Nuclear Criticality Safety Approval
OSHA	Occupational Safety and Health Administration
ORNL	Oak Ridge National Laboratory
PC	Performance Category
RCT	Radiological control technician
REM	Roentgen-equivalent-man
RQ	Reportable Quantity
SAC	Specific Administrative Control
SAR	Safety Analysis Report
SBMS	Standards-Based Management System
SBS	Safety Basis Supplement
SEM	Scanning electron microscope
SIH	Standard Industrial Hazard
SL	Safety Limit
SMP	Safety Management Program
SNF	Spent Nuclear Fuel
SSCs	Structures, systems, and components

TEM	Transmission Electron Microscope
TNT	Trinitrotoluene
TSR	Technical Safety Requirement
UHE	Unmitigated Hazard Evaluation
USQ	Unreviewed Safety Question
UT	University of Tennessee
V	Volt

1. EXECUTIVE SUMMARY

A short-term project will be conducted within the Irradiated Fuels Examination Laboratory (IFEL), Building 3525, east hot cell to recover the molybdenum-99 (Mo-99) from an approximately 17-gram low enriched uranium (LEU) foil (i.e., approximately 7% enrichment) that has been irradiated in the High Flux Isotope Reactor (HFIR). The recovery process will use several liters of a sodium hydroxide solution to dissolve the aluminum cladding and then a low temperature electrolytic dissolution of the uranium foil and its fission products and actinides in a sodium bicarbonate solution. Solid calcium oxide (CaO) will be added to the sodium bicarbonate solution to precipitate out most of the dissolved uranium, fission products, actinides, and carbonate ions while leaving mostly molybdate (MoO_4) in solution. The molybdate solution will be filtered to remove any suspended solids and the filtrate will be sampled and analyzed to determine the Mo-99 content to evaluate the success of the process. The process liquids and precipitates will be disposed. Several irradiated targets will be processed then the equipment disposed.

Although the equipment will be located in the east hot cell for shielding of the irradiated LEU foil, the process equipment is bench-top sized involving several liters of sodium hydroxide and sodium bicarbonate solutions. Several hot cell penetrations will be modified to provide the utilities and control for the equipment operation such as a nitrogen supply for a cover gas and electric supply for the electrolytic dissolution process. The small quantities of inert gas fission products and hydrogen gas generated will be discharged to the hot cell; however, the radioactive iodine gases possibly released during dissolution will be trapped and held for decay. One of the two in-series hot cell ventilation high efficiency particulate air (HEPA) filters in the exhaust header will be replaced with a charcoal filter to provide defense-in-depth protection from an inadvertent radioactive iodine release.

Uranium metal is not a radioactive material form currently addressed in the Safety Analysis Report (SAR) and approved Safety Basis Supplements (SBSs). The release characteristics of the irradiated uranium metal were evaluated and determined to be bounded by the current radioactive material grouping for "contaminated combustibles or HEPA filters" with the added requirement that all radioiodine must be tracked in the radioactive material grouping for "gaseous materials." Although not directly related to the Mo-99 project, the unirradiated uranium metal material form was also added to the safety analysis. The release characteristics for the unirradiated uranium metal were evaluated and determined to be bounded by the current radioactive material grouping for "powders and solutions." The SAR Table 3-5, which lists the radioactive material limits, requires updating to address the new material forms for the metallic uranium. The Technical Safety Requirements (TSR) changes for the proposed activities are limited to modification of the radioactive material inventory limit reference provided in TSR Section 5.11.1 to update it to reference the revised SAR Table 3-5 provided in this SBS.

2. PURPOSE OF THE SBS

A new activity has been proposed for Building 3525 that involves the receipt and processing of approximately 20-gram LEU metal foil. The SBS will evaluate the radioactive material release characteristics to add new controls or confirm the existing controls to ensure the activity can be

safely conducted within the facility. The radioactive material limits for the unirradiated and irradiated uranium metal will be specified in a revision to the TSR Section 5.11.1 "Radioactive Material Limit," Specific Administrative Control (SAC) by identifying a revised SAR Table 3-5 provided in this SBS. The SBS also provides the supporting safety analysis documentation for changes to the Building 3525 TSR.

3. BACKGROUND

Building 3525 is currently used to receive and handle irradiated/radioactive materials, transfer materials, package and ship materials, package waste, maintain remote equipment, and decontaminate the facility and equipment. Examination and testing activities that occur within the facility hot cells include metrology, metallographic sample preparation, optical microscopy, scanning electron microscopy/microprobe, gamma spectrometry, and other physical properties evaluations as appropriate to the experimental objectives of the particular program.

Building 3525 is an operating facility with a planned long remaining operational lifetime. The activities addressed in this SBS are limited and will not become a permanent part of the facility; however, the new radioactive material form for uranium metal will be retained within the safety analysis. Therefore, the results of this SBS related to the uranium metal material form will be incorporated into a future update of the facility SAR.

3.1 FACILITY OVERVIEW

The Oak Ridge National Laboratory (ORNL) is managed by UT-Battelle, LLC for the Department of Energy (DOE). ORNL is located in Bethel and Melton Valleys approximately eight miles southwest of the population center of the city of Oak Ridge, Tennessee.

Building 3525 is situated on a relatively flat elevation in the central portion of the main ORNL campus. The building is located at the corner of Central Avenue and Fourth Street.

Building 3525 is a two-story brick building with a partial basement. The facility has a gross floor area of about 27,000 ft². The operating and service areas occupy about 220,000 ft³. The primary-contained complex is an additional 29,300 ft³. The building is divided into two functional sections. The front section is a single-story office area. The back section is a two-story area south of the office area that houses the hot cell complex, the operating areas, and other supporting activities. The Building 3525 SAR provides a complete description of the facility areas.

3.2 FACILITY DESCRIPTION

This SBS addresses a proposed new activity for the IFEL Building 3525 located in the east hot cell. This chapter provides a description of the facility areas, equipment, structures, and processes related to the new activity. This chapter also describes facilities and processes needed to support assumptions used in the hazard and accident analyses for the new activity. The Building 3525 SAR provides a complete facility description.

3.2.1 Facility Structure

The two-story part of the Building 3525 structure has three major operating areas: (1) a shielded hot cell complex having three primary hot cells, two shielded areas to service the hot cells, three auxiliary hot cells for research, and a charging area; (2) maintenance and service areas (including the partial basement) surrounding the cell complex; and (3) an operating area. The single story front office area is adjacent to, but isolated from, the operating areas. The building is constructed of structural steel, reinforced concrete and masonry. Perimeter walls are constructed of reinforced concrete block with an outer brick veneer. Floors are reinforced concrete slabs that are either poured on compacted aggregate or supported on structural steel. The roof, which was installed above the existing roof in 1999, is a sprayed-on silicone coating over polyurethane foam insulation. In 2009, parts of this roof layer were sprayed with a high-performance polyurea material to seal and maintain the integrity of the roof surface.

The general layout of the first floor of Building 3525 is shown in Fig. 3-1. The first floor provides space for personnel offices, hot cell operations, maintenance and utility areas, the charging area and truck unloading area, and the Core Conduction Cooldown Test Facility (CCCTF). Radioactive material shipments are received on the south side of the facility. The transport vehicle is moved into the truck bay (Room 127) where an overhead crane removes the shipping cask from the vehicle and transports it into the charging area (Room 130). The charging area has a dedicated ventilation system and the area is used to insert and remove materials and components into or from the hot cells. The radioactive materials are removed from the shipping cask and moved into the north hot cell using one of several loading ports or an airlock in the south wall of the north hot cell. The radioactive material is then moved within the hot cells using overhead cranes and manipulators. Small openings, approximately 18-in by 18-in, between each of the hot cells allows small items to be moved as needed from hot cell to hot cell.

3.2.2 Hot Cells

The main shielded hot cell complex is centrally located on the first floor. The main cells, which are designed to handle a variety of radioactive materials, are in the shape of a "U." The first floor plan view of the hot cell complex is shown in Fig. 3-1. The cell structure is divided into three separate cells that are known as the north cell, east cell and west cell. The face of the north cell is 53 ft long and the face of the east and west cells are 43 ft long. Each cell has an exterior depth of 16.5 ft and an interior depth of 10 ft. The cell walls are constructed of barytes concrete and are 3-ft. thick. Each cell is 15 ft. from cell floor to ceiling. The cells are separated by permanent 18-in. thick concrete walls with an approximately 18-in-by-18-in opening. Each cell is further subdivided into workstations. Workstations 1 through 4 are in the west cell, workstations 5 through 8 are in the north cell and workstations 9 through 13 are in the east cell. The 13 workstations are served by 15 oil-filled, lead glass viewing windows (the corner workstations 5 and 9 each have two windows) and a pair of master-slave manipulators located at each window.

Fig. 3-1. First Floor Plan of 3525

The inside surfaces of the cells are lined with stainless steel to provide confinement of particulate matter and aid in cell decontamination. Penetrations are provided for stereomicroscopes and periscopes and the sealed entry of services such as instrument lines, lights, water and gas.

Heavy objects within each cell are moved by electromechanical manipulators and a three-ton overhead bridge crane. Radioactive materials primarily enter and leave the cells through three shielded transfer stations provided at the rear face of the north cell. Two small diameter horizontal transfer stations are used for small objects while larger items are transferred through the shielded air-lock door system. Additional shielded ports are in each cell for introduction and removal of supplies or low activity radioactive materials not posing a personnel dose problem. A shielded port also may be opened to measure the dose rate from an item located inside the cell. Installed equipment in the cells include metallography equipment such as metal cutting saws, guides, polishers and micro-hardness tester; gamma scanning equipment; and related items for remote mechanical disassembly of experiment capsules and physical examination of fuel elements and irradiated materials.

3.2.3 East Hot Cell

The east cell (Fig 3-2) measures 36 ft long by 10 ft deep by 14 ft high internally and is divided into workstations 9 through 13 numbered from north to south. Workstations 10 through 13 are roughly 6 ft by 10 ft and work station 9 (a corner station) is 12 ft by 10 ft. At the northwest corner of work station 9 is a pass-through port (approximately 18-in by 18-in) with a cart that allows the transfer of materials from the adjacent north cell (work station 8). Material or equipment can be moved between workstations in the east cell using a three-ton crane, electro-mechanical manipulator, or master-slave manipulators. Typically, any materials or equipment that is brought into the east cell is transferred from the north cell. However, equipment larger than the pass-through port opening can be moved into the east cell via a set of three removable plugs in the ceiling of the east cell directly above workstation 9 creating an approximately 4-ft by 11-ft opening. The decontamination area, which is also a shielded area, is located immediately above the north and east hot cells. Equipment can be raised or lowered from the east cell (work station 9) using a 3-ton crane located in the decontamination area. The east cell also has a 4-in service sleeve or "throw-in" port located in the back wall of workstation 10. On the south wall of the east cell are located two sleeves/ports that are normally closed with shield plugs. The 10-in port through the south wall can be used for the transfer of materials from carriers directly into the east cell. In the southeast corner of the cell at station 13 in the roof is a sleeve that connects to the irradiated microsphere gamma analyzer (IMGA) cell on the second floor. This sleeve permits the passage of an elevator for material transfer between the IMGA cell and the east cell.

The electro-dissolution processing equipment will be located at the north end of the east hot cell at work station 9 which allows manipulator access from the manipulators at both windows 9N and 9E. The support equipment for utilities and control can be located in either Room 131 or Room 130 (Fig 3-1) depending on the potential for the equipment to contain radioactive materials.

Figure 3-2 Hot Cell Complex

3.2.4 Hot Cell Ventilation System

The Hot Cell Ventilation (HCV) System (Fig. 3-3) maintains a negative air pressure within the facility primary confinement areas including the three hot cell banks, hot cell equipment storage area (HESA), decontamination area, glove maintenance room (GMR), scanning electron microscope (SEM) cell, IMGA cell and core conduction cooldown test facility (CCCTF) cell. The negative pressure for the K-15 cell exhaust system is provided by either the ORNL 3039 Stack fan(s) or the building's exhaust (booster) fans (K-15.8-1 and K-15.8-2) arranged in parallel (one operating with the other fan in standby or, if required, both fans running together). The booster fans do not run under normal conditions. The 3039 Stack fans, which have backup capabilities, are shared with other facilities at ORNL. A local building stack is available. The booster fans are used to boost the exhaust suction differential pressure of the K-15 cell exhaust system in the event that the differential pressure between the exhaust duct and operating area is not sufficiently maintained. When the differential pressure decreases below a set point, fan K-15.8-1 will automatically start to increase the negative pressure in the hot cells. If that fan fails to maintain sufficient negative pressure, fan K-15.8-2 will be automatically started. Whenever the booster fans are automatically started, the

Figure 3-3 Hot Cell Ventilation (HCV) System

last fan started will continue to operate until it is manually shutdown at the operations area console. The fans are located in the cell exhaust fan room in the basement of Building 3525 and normally discharge via underground ducting to the 3039 Stack. In the event the 3039 Stack differential pressure drops below a set point, (differential pressure between the exhaust duct and static atmospheric pressure) the exhaust is automatically switched to the local building stack that is located at the south side of the building. Whenever the local building stack is switched into operation, the booster fan automatic controls described above will sense the loss of the 3039 Stack vacuum supply and start (and control) the building booster fans to maintain negative air pressure within the facility primary confinement areas.

The HCV System is provided with multiple sets of HEPA filtration. Each of the three hot cell banks has a set of three parallel roughing filters in series with three parallel HEPA filters. These filters are located in the west hot cell at workstation 4 (K-15.4-2), in the north hot cell at workstation 5 (K-15.4-1), and in the east hot cell at workstation 9 (K-15.4-3). The HCV System exhaust lines are ducted to the basement and into the HCV System cell exhaust filter room where the four, 24-in-by-24-in, primary HEPA filters are located. There are two filter banks containing four filters (i.e., two filters per bank) identified as (1) Filter Bank #1 and (2) Filter Bank #2 that filter the exhaust air. The four primary filters are identified as K-15.12-1 through K-15.12-4 with filters K-15.12-1 and K-15.12-3 being the upstream filters in the two banks and K-15.12-2 and K-15.12-4 being the downstream filters. These filters banks are configured so that either or both of the filters banks can be online and provide the back-up for the in-cell filters. These four HEPA filters have been designated as safety-significant equipment to ensure hot cell exhaust air filtration prior to discharging. The TSRs require that at least one of the HEPA filters in an online HEPA bank must be declared as Operable prior to allowing hot cell operations.

Since only one of the exhaust header HEPA filters in each of the online HEPA banks must be operable, the two downstream HEPA filters (i.e., K-15.12-2 and K-15.12-4) will each be replaced with a charcoal filter to provide defense-in-depth protection if the systems radioactive iodine traps fail and release radioactive iodine to the hot cells.

3.3 New Activity Description

The new activity to electro-dissolve and separate the Mo-99 from an approximately 20-gram LEC uranium metal foil involves the installation and operation of the following tanks (see Figure 3-4) that have volumes of several liters and the control and supporting equipment:

- 1) dissolution tank (T-1),
- 2) waste tank (T-2),
- 3) precipitation tank (T-3), and
- 4) filtrate tank (T-4)

The in-cell equipment will be located on a small skid that will be moved into the hot cell as a unit. Service and control connections will be made with the supporting equipment located in the Room 131 operating area or the Room 130 charging area.

Figure 3-4. Mo-99 Process Diagram

3.3.1 Uranium Metal Foil Target

The Mo-99 target will be an approximately 6.0-inch long hollow outer aluminum cylinder with an outer diameter of approximately 1.4 inches and a 6.0-inch long inner cylinder with an inner diameter of approximately 1.2 inches with a compressed uranium metal foil between the inner and outer cylinders. The target construction (Figure 3-5), which is taken from Y-12 drawing M802224-0001, is two concentric aluminum tubes with the uranium foil and fission-recoil barriers sandwiched between the tubes. The uranium metal foil has an aluminum fission-recoil barrier on both sides. The fission-recoil barrier for the annular target consists of two layers of aluminum foil with the uranium foil pressed between them. The uranium metal foil is

approximately 3.4-in long by 3.2-in wide and 125 microns thick between two aluminum fission barriers that are 15-20 micron thick. The uranium foil (with fission-recoil barriers) is wrapped around the inner tube, which has a small "undercut" to ensure that the uranium foil is positioned correctly within the target, and the inner tube with the uranium foil wrapped around it is slid into the outer tube. The inner tube is then expanded to create a target in which the uranium foil is held tightly between the two tubes, providing good heat transfer within and out of the target and essentially eliminating any air gap around the foil. After the expansion of the inner cylinder, the two ends of the target are welded shut. For processing within the hot cell, the inner and outer tubes will be mechanically removed and the uranium metal foil with its aluminum fission-recoil barriers will be dissolved and the Mo-99 separated.

Figure 3-5. Mo-99 Target

3.3.2 Process Tanks

Dissolution Tank (T-1)

The dissolution tank completes two dissolving processes. The aluminum clad uranium metal foil target is placed into a mesh basket that is suspended in the dissolution tank. First it dissolves the aluminum cladding from the target leaving the irradiated uranium metal in the mesh basket. This process is completed using several liters of approximately 1 molar (1M) sodium hydroxide (NaOH) solution and is a routine process at facilities like Building 7920 for dissolving aluminum cladding. No electrical voltage or current is used for this dissolution. The chemical process is:

A nitrogen sweep on the dissolution tank will sweep the several liters of hydrogen that will be generated into the hot cell. An iodine trap will also be installed on the vent line; however, no significant quantities of radioactive iodine will be released. Since the aluminum cladding is in direct contact with the uranium metal, some of the fission products near the aluminum-uranium interface may recoil into the aluminum.

After the aluminum has been dissolved from the target into the sodium hydroxide solution, the dissolved aluminum solution will be transfer to the waste tank (T-2). A small quantity of water (~0.5 liter) will be used to rinse the tank and uranium foil and then be transferred to the waste tank (T-2). Approximately 1M sodium bicarbonate solution is added to the dissolution tank. A low voltage (i.e., approximately 10 volts) direct current (DC) will be used to pass approximately 20 amps of current through the solution while the solution is stirred with a mechanical stirrer that is also in the tank. The solution will be at approximately 90°C from the current passing through the solution. Numerous chemical processes are required to dissolve the uranium metal, the actinides, and fission products requiring several hours of operation. Several liters of hydrogen and oxygen gases will be generated by the chemical processes and the electrolysis of the water from the electrical current. The hydrogen and oxygen will be swept from the tank by the nitrogen sweep and discharged to the hot cell. Preliminary estimates indicate that less than 1 cubic foot of hydrogen and oxygen are generated in the dissolution process.

All iodine releases to the process nitrogen gas sweep will be elemental iodine and no significant quantities of organic iodine forms are expected such as methyl iodine, which is the most difficult to trap. In the irradiated uranium metal foil, the iodine will exist primarily as cesium iodide and elemental iodine. During the caustic dissolution, the iodine will be primarily iodine ions in the caustic solution. Since the dissolution process is not an organic process and no carbon is used, no significant quantities of methyl iodine will be generated. The iodine is released from the caustic solution when the iodine ions combine to form elemental iodine, escape from the liquid surface, and are swept out by the nitrogen cover gas. Similarly in accidental releases, the predominant form of the released iodine will be elemental iodine.

Most of the fission products will be dissolved in the sodium bicarbonate solution although some of the noble gases and iodine will be released into the nitrogen sweep. The noble gases and iodine will pass through the vent line iodine trap which will capture the iodine and pass the noble gases to the hot cell. The iodine trap uses activated carbon as the trapping material. The iodine will be held by the trap and allowed to remain on the trap until it has sufficiently decayed.

The sodium bicarbonate solution with the dissolved irradiated uranium target will be vacuum-transferred to the precipitation tank (T-3). A small filter will be in the line to filter any unexpected solids that may be in the solution.

Waste Tank (T-2)

The waste tank collects the sodium hydroxide solution with the dissolved aluminum and the rinse water. The solution is vacuum pumped to the waste tank by drawing a vacuum on the waste tank. A small filter will be in the transfer line to collect any solids although no solids are expected. After the process is completed, the waste tank will be disconnected and the solution disposed by solidification, evaporation or other acceptable method addressed in the current safety analysis.

Precipitation Tank (T-3)

Pellets of calcium oxide (CaO) will be added to the precipitation tank containing the sodium bicarbonate solution with the dissolved target and the solution will be stirred. The CaO pellets will react with most of the dissolved actinides, fission products, and uranium metal leaving a relatively pure solution containing the dissolved Mo-99. The solution containing the dissolved Mo-99 will be vacuum transferred to the filtrate tank (T-4). The line to the filtrate tank will contain a small filter to remove any suspended precipitates. After the process is completed, the precipitation tank will be disconnected and the wet precipitate disposed by solidification, drying or other acceptable method addressed in the current safety analysis.

Filtrate Tank (T-4)

The solution containing the dissolved Mo-99 in the filtrate tank will be sampled to determine the success of the dissolution process. After the solution sampling has been completed, the solution will be disposed by solidification, evaporation, or other acceptable method addressed in the current safety analysis.

3.3.3 Support Equipment

The tanks are used to complete all major processes for the dissolution but supporting equipment and services are needed to support the processes. The major supporting equipment and services are:

1. bottled nitrogen gas supply,
2. iodine trap,
3. vacuum pump,
4. transfer lines and inline components (e.g., isolation valves, filters, roto-meters, vacuum regulator, pressure controller, pressure indicator, quick-connections, etc.), and
5. DC power supply

In addition to the equipment and services directly supporting the operation, one of the HEPA filters in each of the HCV System exhaust header Filter Bank #1 and Filter Bank #2 will be removed and replaced with a charcoal filter to provide a defense-in-depth filter for any iodine that may be released to the hot cell by the process.

Bottled Nitrogen Gas Supply

A standard nitrogen gas cylinder will be located in Room 131 and connected via tubing equipped with a regulator to provide a gas sweep to the dissolution tank and precipitate tank. The tubing will pass through a service penetration. The gas purge will sweep (1) any hydrogen generated into the hot cell and (2) any iodine and noble gases that may be released into the gas space through the iodine filter in the vent line.

Iodine Trap

The iodine trap will be installed in the dissolution tank vent line. The trapping material will use activated carbon as the trapping material. The estimated quantity of iodine in the irradiated uranium metal foil is approximately 0.1 mg.

Vacuum Pump

A small electrical vacuum pump will be located on the process skid that will be used to draw a vacuum on the tanks to allow (1) the nitrogen sweep to occur under a vacuum, and (2) a vacuum transfer between the tanks. The transfers will be controlled by manipulating the valves and connections to the tanks and the operation of the pump. The vacuum pump exhaust will be discharged directly to the hot cell.

Transfer Lines and Inline Components

The tanks will be connected by transfer lines to support the solution transfers. Small inline filters will be installed in the lines to filter out any solids. The used filters will be evaluated to determine the types and quantities of solids captured to provide a better understanding of the process. Various other inline components such as valves, disconnects, pressure indicators, flow indicators, flow controllers, etc. are required to operate the system.

DC Power Supply

A DC power supply control unit will be located in Room 131 and the output lines will pass through a hot cell penetration and connect to the dissolution tank. The input to the power supply will be 220 volts from a convenience outlet in Room 131. The power supply will be capable of supplying outputs of approximately 0-32 volts and 0-64 amps and is expected to operate at approximately 10 volts and 20 amps for the electro-dissolution.

HCV Charcoal Filters

As discussed later in Section 4, the presence of the radioiodine that is evaluated as a gaseous radioactive material form has resulted in a facility decision to add charcoal filters to the HCV System exhaust path to filter possible radioiodine from the exhaust flow stream. The radioiodine limits provided by the radioactive material control program will restrict the quantities to levels below that which would require the designation of new safety-related features. The charcoal filters will be new defense-in-depth components in the safety-significant HCV System.

In 2012, the HEPA filter banks were replaced with new Flanders HEPA filter banks BG-series units containing two in-series 24-in by 24-in by 11.5 HEPA filters in each unit. Flanders also provides high efficiency gas adsorber (HEGA) filters that remove gaseous contamination in the flow stream and fit into the HEPA filter slot in the HEPA filter unit. Under the scope of a temporary modification, a Flanders HEGA filter model AG-GG12-101-NS (or equivalent) will be installed in Filter Banks #1 and #2 that replaces one of the two currently installed HEPA filters. This temporary modification is expected to be in place for several months to support

preparation activities for the project. The HEPA filters are rated for 1000 cfm with an approximately 0.9 in water column (WC) pressure drop. The SBMS criteria for changed out HEPA filters is a differential pressure of 4.0 in WC so the HEPA filter differential pressure is within the system requirements. At the rated flow rate, the filter will provide a radioiodine decontamination factor (DF) for elemental radioiodine of at least 100. Since the HCV System is designed for up to 2000 cfm with only one HEPA bank online, both HEPA banks would need to be placed online to protect the HEPA filters if the HCV System is operated at flow rates of over 1000 cfm. A facility standing order or other appropriate control method will be applied to ensure the rated HEPA filter flow is not exceeded.

One of the two HEPA filters in the exhaust header HEPA Filter Bank #1 (see Figure 3-3) and one filter in HEPA Filter Bank #2 will be replaced with charcoal filters. Filter Bank #1 and Filter Bank #2 are safety-significant components that provide part of the hot cell confinement boundary. Since the filter banks are safety-significant components, only one of the filters must be operable for TSR compliance allowing the second HEPA filter to be replaced with a charcoal filter to provide additional protection for the potential radioiodine releases.

The HCV System has an overall designation of a safety-significant system because (1) the exhaust header HEPA filters form part of the confinement boundary for the safety-significant hot cells, and (2) the maintenance of a negative pressure on the hot cells during accident conditions provides a significant defense-in-depth function which requires upgrading the system categorization to a safety-significant system. The parts of the HCV System that do not directly support either of these two safety functions are designated as defense-in-depth components. Since the charcoal filters do not support either of these functions, they are not required to be designated as safety-significant components even though they are installed in the safety-significant HEPA filter banks.

The irradiated uranium foil is expected to contain up to approximately 150 Ci of I-131 and 50 Ci of I-133. The safety analysis conservatively assumes all of the radioiodine as a gaseous material which results in the radioiodine contributing approximately 7% to the total hot cell inventory. Since (1) the 100% release assumption is a very conservative assumption and the process accidents are not likely to release a large percentage of the radioiodine inventory, (2) the radioiodine nuclides have relatively short half-lives and quickly decays after being received in the facility, and (3) the process will only be conducted several times, the defense-in-depth function of the charcoal filters do not provide a significant defense-in-depth function which would require upgrading their categorization to a safety-significant categorization. The charcoal filters perform a defense-in-depth function.

4. IMPACT ON HAZARDS AND RISK

4.1 HAZARD IDENTIFICATION AND SCREENING

The hazards associated with the existing operations at Building 3525 are identified and screened in ORNL/3525/SAR, *Safety Analysis Report – Irradiated Fuels Examination Laboratory, Building 3525*, (Ref. 1); SBS/3525/2010-02, *Safety Basis Supplement for the Installation and*

Operation of New Glove Boxes at Building 3525, (Ref. 2); and SBS/3525/2012-01, *Safety Basis Supplement for the Receipt of Cermet Targets as New Radioactive Material Form for the Irradiated Fuels Examination Laboratory, Building 3525*, (Ref. 3). The hazards associated with the Mo-99 dissolution activities are identified in Appendix A and discussed below. The hazards identified and evaluated in Appendix A consist of the specific hazards associated with the chemical separation of the Mo-99 from the irradiated LEU metal foil. The evaluation does not include (1) the installation of the equipment in the hot cell, (2) the receipt of the irradiated target and movement into the hot cells, or (3) the mechanical removal of the irradiated LEU foil from the inner and outer aluminum tubes. These are routine activities for the facility already addressed in the current safety analysis.

4.1.1 Radiological Hazards

The existing SAR limits for radioactive materials for the facility are provided in SBS/3525/2012-01, *Safety Basis Supplement for the Receipt of Cermet Targets as New Radioactive Material Form for the Irradiated Fuels Examination Laboratory, Building 3525*, Table 3-5 (3a-²³⁸Pu-GB), "Hazardous Material Listing for SAR Rev 3a, SBS/3525/2010-02 and SBS/3525/2012-01." Section 3.3.1 describes the physical characteristics of the Mo-99 target. The radiological hazards are an inhalation hazard from accidental releases, a direct dose hazard, and a fissionable material hazard.

Inhalation Hazard

The irradiation of aluminum and uranium produce well known radionuclides. Irradiated aluminum metal has been received and processed many times at the facility. The aluminum metal inner and outer tubes will be mechanically removed from the foil and added to the metal discard area located within the hot cells. The aluminum metal inner and outer tubes are routine hazards for the facility and do not require any further analysis in the SBS.

Irradiated uranium is routinely received by the facility primarily in the physical form of irradiated uranium oxide reactor fuel rods. The radionuclides produced by the irradiated uranium are well known and routinely calculated. The physical form of the uranium metal foil is different than the currently evaluated physical forms for irradiated uranium. SBS/3525/2012-01 Table 3-5 (3a-²³⁸Pu-GB) indicates that irradiated uranium has been evaluated for the physical forms of spent reactor fuel, cermet targets, and alloy fuels. Irradiated uranium metal will have different release characteristics for the accidents evaluated in the safety analysis. The evaluation of the release characteristics of the radioactive material forms is completed in calculation ORNL/3525/DAC/2007-01, *IFEL Building 3525 Unmitigated Hazard Evaluation Threshold Consequence Calculations*. Calculation ORNL/3525/DAC/2007-01 Rev. 8 (Ref. 4) evaluates the release characteristics of the irradiated uranium metal and facility radioactive material limits established for the accidents currently identified in the safety analysis. This SBS analysis will evaluate and confirm that the new activity does not create any new accidents. The evaluation will determine if the release characteristics for the current facility accidents will bound those of the irradiated uranium metal if the uranium metal were added to the "contaminated combustible & HEPA filter" radioactive material form. The Mo-99 project personnel indicate that the Mo-99 target will be processed before the short-lived radioiodine will decay to insignificant quantities.

The radioactive material form was also modified to address the potential presence of radioiodine from the Mo-99 target in a similar approach as that used for the presence of radioiodine in the spent nuclear fuel and cermet radioactive material forms. The evaluation for the irradiated uranium metal conservatively assumed that 100% of the radioiodine may be released in the gaseous radioactive material form. This conservative assumption is not based on documented test data but rather a lack of test data so 100% of the radioiodine was conservatively assumed to be releasable in the gaseous material form. The quantity of radioiodine in the Mo-99 target is sufficiently low such that the Mo-99 activity can proceed without an undue impact to the facility operations.

Calculation ORNL/3525/DAC/2007-01 Rev. 6 (Ref. 5) identified the release characteristics of unirradiated uranium metal but the SAR Table 3-5 has not been updated to include unirradiated uranium metal. The calculation results indicated that including the unirradiated uranium under the "powders and solutions" radioactive material form will ensure that the release characteristics are bounded.

Direct Dose Hazard

The presence of irradiated aluminum and uranium are common within the facility; however, the uranium is typically uranium oxide instead of uranium metal. The direct dose hazard primarily comes from the fission products. These are typical hazards that are routinely present within the facility and already evaluated in the currently approved SAR.

Fissionable Material Hazard

The uranium, which is LEU, is the fissionable material that presents the hazard. The facility contains significant quantities of fissionable materials particularly in the form of uranium oxide. The physical form of the uranium as metal and then later as a solution when dissolved has no impact on the fissionable material controls. Nuclear Criticality Safety Approval (NCSA) NCSA-121 Rev. 5 (Ref. 6) provides the current nuclear criticality safety controls for the facility areas impacted by this SBS including the east hot cell. The NCSA allows up to 1400 g of dry ^{235}U fissionable equivalent material (FEM) in the east hot cell with limits on the quantities of liquids permitted in the hot cell. Prior to implementation of this SBS, the NCSA will be revised as a permanent revision to reduce the east hot cell limits to less than 700 g ^{235}U FEM and remove the restrictions on the quantities of liquids permitted in the east hot cell. The DOE site office will be notified when the NCSA has been revised for this project.

4.1.2 Non-radiological Hazards

Typical standard industrial electrical, mechanical, chemical, and thermal hazards are associated with the Mo-99 recovery process. These hazards include: standard electrical power supply to mixers, vacuum pump, and the dissolution tank; kinetic energy hazards associated with tank mixers and vacuum pump; potential energy hazards associated with pressurized gas cylinders; chemical hazards associated with laboratory quantities of chemicals for processing and hydrogen produced by the process.

Potential health effects resulting from exposure to hazardous materials used for processing are controlled through the Hazardous Material Protection provisions as described in Chapter 8 of the Building 3525 SAR and are not further evaluated. The non-radiological hazards do not impact the current safety analysis evaluations.

4.2 HAZARD EVALUATION

The Mo-99 recovery process will occur in the east hot cell. The Unmitigated Hazard Evaluation (UHE) for the hot cells is provided in the SAR Appendix B, "Unmitigated Hazard Evaluation -- Hot Cells." The impacts from the Mo-99 recovery process on the hot cells UHE are evaluated in Appendix B, "Mo-99 Recovery Impacts on Hot Cells UHE." A review of the evaluation indicates that the process will not impact the probability or consequences of any of the events for the few dissolutions that will be completed. The identified process controls ensure the radioiodine is not released during the process. No new safety-related controls are required to support the Mo-99 recovery process.

The radioactive material limits for the facility are initial underlying assumptions for the hazard evaluation. The existing Specific Administrative Control (SAC) identified in the SAR Section 4.5.1, "Radioactive Material Limit," must be expanded to include the limits on the irradiated uranium metal that includes radioiodine. When the irradiated uranium metal is dissolved in the sodium bicarbonate solution, the radioiodine will also be dissolved in the solution. The radioactive material inventory control program will be revised to assume that 100% of this radioiodine may be releasable in the gaseous radioactive material form.

4.3 DEFENSE-IN-DEPTH FEATURES AND WORKER SAFETY

The Mo-99 recovery process will take place in the east hot cell which provides worker safety similar to all radioactive operations in the hot cell. The HCV System provides a negative pressure on the hot cell and filters the exhaust air stream to capture the airborne particulate material onto the HEPA filters. Although the presence of radioactive iodine is permitted in the metal/spent nuclear fuel and the cermet targets/alloy fuel radioactive material forms, the radioiodine is primarily contained in the fuel and cermet structure and is relatively unreleasable unless a fire event occurs. The Mo-99 recovery process will dissolve the irradiated uranium metal in the sodium bicarbonate solution making for a more releasable form than previously analyzed for the facility. The dissolver tank includes a radioiodine trap on the vent line that uses activated charcoal as the trapping material. Most of the radioiodine remains in the solution but some will off-gas and be swept out by the cover gas into the vent line containing the radioiodine trap. The radioiodine will remain in the solution unless an acid is added to change the pH of the solution. No acid solutions are in the east hot cell and the recovery process will occur over a very short period of time. If the radioiodine trap fails or radioiodine is accidentally released from the solution, the current HCV System does not have any method to capture the radioiodine other than to rely on its plate-out characteristics. To rectify this situation, as discussed in Section 3.3.3, the two downstream HEPA filters of the HCV System exhaust header HEPA filters in each operating bank will be removed and replaced with a charcoal IIEGA filter to provide a defense-in-depth feature to prevent an accidental release of the radioiodine. The radioactive material inventory limit restricts the quantities of radioiodine so that the accident consequence limits

would not be exceeded if an accident occurred during the recovery process. As a result, the HEGA filters do not require a safety-related designation.

The installation of the two HEGA filters in the downstream filter slots of the HEPA filter banks ensures that the HEGA filters do not impact the existing smoke safety evaluation of the HCV System provided in SAR Section 4.4.3, "Hot Cell Ventilation System." Since the HEGA filter is located downstream of the credited HEPA filter evaluated, smoke plugging characteristics of the HEGA filter will not impact the HEPA filter smoke plugging analysis provided in SAR Section 4.4.2.4. Also, the exhaust air flow through the HEGA filters will have been filtered by the HEPA filters so any entrained smoke would be filtered out before passing through the HEGA filters. Although there are no ignition sources near the HEGA filter, any smoke produced by the HEGA filter charcoal would be discharged through the exhaust stack without impacting the filtration function of the two credited upstream HEPA filters.

5. ADEQUACY OF COMPENSATORY MEASURES

5.1 SAFETY-CLASS STRUCTURES, SYSTEMS, AND COMPONENTS

Safety class structures, systems, and components (SSCs) are those SSCs whose preventive or mitigative function is necessary to keep radiological material exposure to the public below the off-site evaluation guideline. Based on the results of the evaluation in Section 4, no SSCs associated with the proposed changes have been designated as safety class.

5.2 SAFETY-SIGNIFICANT STRUCTURES, SYSTEMS, AND COMPONENTS

Safety-significant SSCs are those SSCs that are (1) credited in the hazard evaluation to prevent or mitigate the consequences that can result in unacceptable risks to facility workers and co-located workers, and provides defense-in-depth protection for the public, and (2) major contributors to defense-in-depth or worker safety. Based on the results of the evaluation in Section 4, no SSCs associated with the proposed changes have been designated as safety-significant SSCs.

5.3 SPECIFIC ADMINISTRATIVE CONTROLS

Administrative controls that provide a specific preventive or mitigative function for accident scenarios identified in the hazard evaluation where the safety function has importance similar to, or the same as, the safety function of a safety SSC are considered to be Specific Administrative Controls (SACs). A SAC for limiting the radioactive material inventory of irradiated uranium metal has been identified to limit consequences to the public and workers from potential accidents. Additionally, the radioactive material inventory limits for unirradiated uranium metal has been determined and will also be added to the SAC.

5.4 TSR COVERAGE

TSR coverage is required for features that (1) maintain the consequences of facility operations below the off-site evaluation guideline or are relied upon in the hazard evaluation to maintain the risk of facility operations at an acceptable level, (2) provide significant defense-in-depth, and (3) provide for significant worker safety. The features may be SSCs or administrative controls (operator actions or responses, operating restrictions or limits, and safety management programs). The SSCs required to maintain the consequences of potential accident events associated with facility operations below the off-site evaluation guideline are determined in the accident analysis and are designated as safety class SSCs. Those SSCs that are not designated as safety class, but are required by the hazard evaluation to maintain the off-site, on-site, or facility worker risk associated with potential accident events at an acceptable level, are designated as safety-significant SSCs. Any additional SSCs determined to provide significant defense-in-depth or worker safety are also designated as safety-significant SSCs. No new safety class or safety-significant SSCs were identified in Section 4. The existing hot cells safety-significant confinement safety function is not impacted by the process. Section 4.2 identified required changes to the current administrative control for radioactive material inventory. The administrative controls that were identified in Section 4 are summarized in Table 5-1. This section identifies the required changes to the TSR programs and controls to ensure the TSR provides the additional controls for the Mo-99 recovery process.

Table 5-1. Features Requiring TSR Coverage

Feature	Type	Applicable Hazards	Applicable Facility Operations	Type of TSR Coverage Required/Comments
Irradiated uranium metal	SAC	Irradiated uranium metal	All	SAC
Unirradiated uranium metal	SAC	Unirradiated uranium metal	All	SAC

5.5 TSR DERIVATION

There are no safety limits (SLs) or limiting control settings (LCSs) as defined in 10 CFR 830 Subpart B required for safe operation of the activities within the scope of this SBS. No new limiting condition of operations (LCOs) or changes to existing LCOs is required for the Mo-99 process operations. A revision to the existing radioactive material inventory control SAC is required to establish radioactive material inventory limits irradiated and unirradiated uranium metal and to incorporate the new radioactive materials into the existing SAC.

5.5.1 Radioactive Material Inventory Control SAC

The irradiated and unirradiated uranium metal radioactive material inventory limits are specified to ensure that radioactive material releases from these materials are properly characterized to reflect their potential to create airborne hazards for the various accident scenarios. Since the

radioactive material inventory control already identifies several radioactive material forms, the irradiated and unirradiated uranium metal forms were added to existing radioactive material forms that provided bounding release characteristics. The potential presence of radioiodine in the irradiated uranium metal requires a change to its bounding radioactive material form of "contaminated combustibles or HEPA filters" to be revised to address the potential presence of the radioiodine which is not bounding by the release characteristics of this radioactive material form. The currently approved SAR Table 3-5 (3a-²³⁸Pu-GB), "Hazardous Material Listing for SAR Rev 3a, SBS/3525/2010-02 and SBS/3525/2012-01," will be revised to add (1) irradiated uranium metal to the "contaminated combustibles or HEPA filter" radioactive material form with a note that states that any radioiodine needs to be considered a gaseous radioactive material form and (2) unirradiated uranium metal to the "powders or solution" radioactive material form.

These limits will be added to the limits in SAR Table 3-5(3a-²³⁸Pu-GB) which are referenced in the current TSR Specific Administrative Control (SAC). The revised table will be designated as Table 3-5(3a-²³⁸Pu-GB-⁹⁹Mo) "Hazardous Material Listing for SAR Rev 3a, SBS/3525/2010-02, SBS/3525/2012-01, and SBS/3525/2013-01" and is provided below.

Table 3-5(3a-²³⁸Pu-GB-⁹⁹Mo). Hazardous Material Listing for SAR Rev 3a, SBS/3525/2010-02, SBS/3525/2012-01, and SBS/3525/2013-01

(Note: Yellow highlighting indicates table changes for this SBS)

Hazardous Material Type	Location in Facility	Quantity	Physical and Chemical Form
Radioactive materials (including fissionable materials)	Main Hot Cells	<p><u>Inhalation dose equivalent:</u></p> <ul style="list-style-type: none"> • Metal/Encased Spent Nuclear Fuel (SNF): < 6.16E+12 rem (Note 3) • Intact Cermet Targets/Alloy Fuel: < 5.28E+12 rem (Note 4) • Segmented SNF: < 2.98E+12 rem (Note 3) • Segmented Cermet Targets/Alloy Fuel: < 2.89E+12 rem (Note 4) • Powders, solutions, or unirradiated uranium metals: < 1.83E+11 rem (Notes 5 and 6) • Contaminated combustibles, TOLPA filters, or irradiated uranium metals: < 2.64E+10 rem (Note 6) • Gaseous materials: < 6.86E+07 rem (Note 2) <p>When multiple material forms are present, the sum of the ratios of each material form to its limit shall not exceed 1.0</p> <p>Total radioactive material at risk (MAR) that may be present in the main hot cells is limited to the MARs associated with "Areas Outside Hot Cells" in this table if:</p> <ul style="list-style-type: none"> • processes in the main hot cells present a credible means of creating an explosive atmosphere <p>OR</p> <ul style="list-style-type: none"> • heavy loads capable of breaching the main hot cells if dropped are lifted over the building roof above the main hot cells <p><u>Direct dose source: (Note 1)</u></p>	<ul style="list-style-type: none"> • Large pieces of highly irradiated material fuels are received in the form of intact fuel elements or rods or segments. • Destructive examination processes (e.g., grinding, sawing, machining) generate fine metal powders of dispersible fuel and fission products. • Oxidation of fuel or specimens forms metal oxides. • Solutions • Encapsulated fission products or actinides • Sample particles, compacts of particles, capsules • Fission products contain some gases that are released when heating • Equipment that has become contaminated as a result of being used in the hot cells.

Table 3-5(3a-²³⁸Pu-GB-⁹⁹Mo). Hazardous Material Listing for SAR Rev 3a, SBS/3525/2010-02, SBS/3525/2012-01, and SBS/3525/2013-01
(Note: Yellow highlighting indicates table changes for this SBS)

Hazardous Material Type	Location in Facility	Quantity	Physical and Chemical Form
	Areas Outside Main Hot Cells	<p><u>Inhalation dose equivalent:</u></p> <ul style="list-style-type: none"> • Metal/Encased SNF: < 1.54E+12 rem (Note 3) • Intact Cermet Targets/Alloy Fuel: <1.32E+12 rem (Note 4) • Segmented SNF: < 7.46E+11 rem (Note 3) • Segmented Cermet Targets/Alloy Fuel: <7.23E+11 rem (Note 4) • Powders, solutions, or unirradiated uranium metals: < 4.57E+10 rem (Notes 5 and 6) • Contaminated combustibles, HEPA filters, or irradiated uranium metals: < 6.61E+09 rem (Note 6) • Gaseous materials: <6.86E+07 rem (Note 2) <p>The radioactive material quantities in each of the glove boxes in Rooms 221 and 222 are additionally limited to the inhalation dose equivalent:</p> <ul style="list-style-type: none"> • Metal/Encased SNF: < 7.94E+08 rem (Note 3) • Intact Cermet Targets/Alloy Fuel: <6.80E+08 rem (Note 4) • Segmented SNF: < 4.76E+08 rem (Note 3) • Segmented Cermet Targets/Alloy Fuel: <4.76E+08 rem (Note 4) • Powders, solutions, or unirradiated uranium metals: < 2.38E+07 rem (Notes 5 and 6) • Contaminated combustibles, HEPA filters, or irradiated uranium metals: < 4.76E+06 rem (Note 6) • Gaseous materials: <4.76E+04 rem (Note 2) <p>When multiple material forms are present, the sum of the ratios of each material form to its limit shall not exceed 1.0</p> <p><u>Direct dose source: (Note 1)</u></p>	

Table 3-5(3a-²³⁸Pu-GB-⁹⁹Mo). Hazardous Material Listing for SAR Rev 3a, SBS/3525/2010-02, SBS/3525/2012-01, and SBS/3525/2013-01

(Note: Yellow highlighting indicates table changes for this SDS)

Hazardous Material Type	Location in Facility	Quantity	Physical and Chemical Form
	Total in Facility	<p><u>Inhalation dose equivalent:</u></p> <p>The sum of the ratio values of the multiple material forms inside the hot main hot cells and the areas outside the main hot cells shall not exceed 1.0</p> <p>Direct dose source: (Note 1)</p>	

Note 1: Because Building 3525 is a hot cell facility, it is recognized that materials handled in shielded structures or containers present a potentially severe external radiation hazard. The shielded structures and containers are necessary during normal operations to maintain worker doses within acceptable ranges. It is difficult to compare external exposure MARs for different materials because of the varying energy levels of the radiation being emitted and the corresponding effectiveness of various shield materials. Therefore, facility and facility area limits are not set based on external dose potential. As long as the MARs in the shielded structures and containers are controlled such that operating area dose rates are maintained within as low as reasonably achievable (ALARA) guidelines, the results of the external exposure hazard evaluation presented in this SAR are valid.

Note 2: Since the determination of the gaseous radioactive material threshold quantities assumed (1) 100% of the material is released, (2) 100% of the released material is in a respirable form, and (3) no particle settling, confinement or filtration in the facility or airborne plume; the gaseous material form can conservatively represent any radionuclide's physical form and accident release fraction.

Note 3: If the encased or segmented SNFs contain radioactive iodine, then 10% of each radioiodine quantities must be entered and tracked in the radioactive material inventory control program as a gaseous radioactive material for its location within the facility.

Note 4: If the intact or segmented cermet targets or alloy fuels contain radioactive iodine, then 50% of each radioiodine quantities must be entered and tracked in the radioactive material inventory control program as a gaseous radioactive material for its location within the facility. Additionally, the hazard level of the radioactive cesium radionuclides must be increased by a factor of 14.3 for intact cermet targets and alloy fuels and 5.9 for segmented cermet targets and alloy fuels to reflect their higher airborne respirable release fraction (ARRF) than the other non-iodine radionuclides as discussed in ORNL/3525/DAC/2007-01.

Note 5: The solutions must be non-organic forms that cannot burn and generate a very high release rate.

Note 6: If the radioactive material contains radioactive iodine, then 100% of each radioiodine quantities must be entered and tracked in the radioactive material inventory control program as a gaseous radioactive material for its location within the facility.

5.5.2 Hot Cell Ventilation System

SAR Section 5.5.1.1, states, "The safety function of the HCV system can be accomplished if (1) the pressures in the main hot cells are at least 0.3 in. of water lower than the pressures in the operating and charging areas, (2) a stage of online HEPA filters in the HCV exhaust flow path is capable of removing at least 99.95% of a test aerosol, and (3) the 3039 stack is providing sufficient vacuum for the operation of the HCV system." The installation of the HEPA filter in the HEPA bank downstream filter slot ensures no HCV System changes between the hot cells and the credited HEPA filter for this configuration. Additionally, the smoke plugging characteristics of the HEPA filter from a fire inside the hot cell do not require an evaluation because the credited HEPA filters will filter the smoke from the airstream and the HEPA filter will not be impacted. The installation of the HEPA filter in the HEPA bank downstream slot will not impact the HCV System TSR derivation.

5.6 INTERFACE WITH TSRS FROM OTHER FACILITIES

There are no TSRS from other ORNL facilities that affect the safety basis of Building 3525.

6. CONCLUSION

The proposed activities associated with the Mo-99 recovery do not involve any new types of accidents. The new radioactive materials forms for irradiated uranium metal and unirradiated uranium metal have been identified and added to the radioactive materials inventory limits. The radioactive material limits are specified such that no new safety class SSCs are necessary for protection of off-site receptors and no new safety-significant SSCs are necessary for protection of co-located and on-site receptors. The revised controls will be implemented through a revised SAC as discussed in Section 7 below. With this control in place, the activities for the Mo-99 recovery may be conducted safely and the radioactive material forms will be bounded by the safety analysis. One of the HCV System exhaust header HEPA filters in an online bank will be removed and replaced with a charcoal filter to provide defense-in-depth protection from an inadvertent radioiodine release.

7. REVISED CONTROL SET

The TSRS for Building 3525 will require revision to incorporate new inventory controls for the presence of irradiated and unirradiated uranium metal radioactive material forms identified in this document. The existing inventory control SAC in TSRS Section 5.11 will be revised to reference the revised limits. The TSR Section 1.1 definition of "significant quantities of radioactive material" requires revision to include the new radioactive material forms. "Significant quantities of radioactive materials" is defined in SAR Section 5.4, "Derivation of Facility Modes."

TSR Rev. 5d (Ref. 7) is currently approved and implemented for the facility. The TSR Section 5.11 radioactive material inventory control program identifies SAR Table 3-5(3a-²³³Pu-GB) for the specific listing of the material limits. The TSR Rev. 6 identifies the SAR Table 3-5(3a-²³⁸Pu-GB-⁹⁹Mo) as the source of the radioactive material inventory control limits.

TSR Rev. 6 has Section 1.2 definition of “significant quantities of radioactive material” updated to include the new radioactive material forms.

8. TERMINATION OF THE SBS

The activities described in this SBS are considered a short duration project within the facility. The only changes needed to support the project were the changes to the SAC for the new forms of radioactive materials and the use of the charcoal filters when processing materials with radioiodine. Even though the project is of a short duration, the changes needed to support the receipt of the new forms of radioactive materials will be retained by the safety basis. The use of the HEGA filters will not be retained beyond their support of this project. Therefore, parts of this SBS related to the new radioactive material forms will be incorporated into a future revision of the facility SAR. After the revised SAR, which includes those parts of the scope of this SBS, has been approved by DOE and implemented by the facility, this SBS may be terminated. Until that time, this SBS will be a part of the facility safety basis.

9. REFERENCES

1. *Safety Analysis Report – Irradiated Fuels Examination Laboratory – Building 3525*, ORNL/3525/SAR, Revision 3a, September 21, 2011.
2. *Safety Basis Supplement for the Installation and Operation of New Glove Boxes at Building 3525*, SBS/3525/2010-02, Revision 0, August 31, 2012.
3. *Safety Basis Supplement for the Receipt of Cermet Targets as New Radioactive Material Form for the Irradiated Fuels Examination Laboratory, Building 3525*, SBS/3525/2012-01, Revision 0, July 30, 2012.
4. *IFEL Building 3525 Unmitigated Hazard Evaluation Threshold Consequence*, ORNL/3525/DAC/2007-01, Revision 8, February 14, 2013.
5. *IFEL Building 3525 Unmitigated Hazard Evaluation Threshold Consequence*, ORNL/3525/DAC/2007-01, Revision 6, June 5, 2012.
6. *Fissionable Material handling and Storage at Building 3525*, NCSA-121, Revision 5, August 28, 2012.
7. *Technical Safety Requirements – Irradiated Fuels Examination Laboratory – Building 3525*, Revision 5d, approved August 31, 2012.

APPENDIX A
HAZARDS IDENTIFICATION

A-1

Type hazard	Keep if criteria are exceeded	Action (keep or screen out)	Action basis (includes inventory and form of all materials)
Radioactive material	Any radioisotope meeting or exceeding the Table A1, DOE-STD-1027-92 threshold quantity (TQ) criteria; or exceeding the Appendix B, 40 CFR 302 reportable quantity (RQ) criteria. The inventory/RQ or inventory/TQ ratios should be added when making this evaluation	Keep	<p>Approximately 6-in long, 1.2-in outer diameter and 1.0-in inner diameter irradiated aluminum cylinder containing aluminum-clad LEU uranium metal foil pressured between inner and outer parts of the cylinder. The target will be irradiated for one IIFIR cycle such that radionuclides will consist of fission products, activation products, actinides, and uranium metal.</p> <p><u>Inhalation dose equivalent:</u></p> <ul style="list-style-type: none"> The irradiated aluminum target with LEU uranium metal foil will have the typical fission products, activation products, actinides, and uranium metal. The target will be received only days after being removed from IIFIR so it will contain significant quantities of radioiodine. <p><u>Direct dose source:</u></p> <ul style="list-style-type: none"> < 1.8E+5 R/hr unshielded at 1 ft <p><u>Fissionable material:</u></p> <ul style="list-style-type: none"> < 700 g U²³⁵FEM
Toxic, corrosive, or reactive material	<p>Material available to the general public at retail outlets without permit and is present in end-user quantities</p> <p>OR</p> <p><RQ value in Title 40 CFR Part 302.4</p> <p>OR</p> <p>Laboratory scale quantities subjected to requirements of 29CFR1910.1450</p>	Screen out	Several liters of 1M sodium hydroxide solution, several liters of 1M sodium bicarbonate solution, several kg of calcium oxide pellets, water, and aluminum hydroxide solution.
Biohazard	Any known biohazard where special controls are required	N/A	None identified.

Type hazard	Keep if criteria are exceeded	Action (keep or screen out)	Action basis (includes inventory and form of all materials)
Asphyxiant	Any asphyxiant (i.e., gas at ambient temperatures that is denser than air) that could either affect a large number of people or any unsuspecting people	Screen out	Nitrogen from compressed gas cylinder supplies.
Flammable material	> 5000 lb of a liquid with a flash point < 100°F or > 3000 standard ft ³ of a gas with an established lower explosive or flammability limit (LEL)	Screen out	Several liters of hydrogen generated by the dissolution processes and electrolysis of water.
Explosive and pyrophoric material	Any 49 CFR 173 Division 1.1, 1.2, or 1.3; or >10 oz of Division 1.4	N/A	None identified.
Electrical energy	Unusual application not adequately controlled by OSHA (e.g., soil vitrification); ≥ 800 volts and 24 ma output; or stored energy ≥ 50 joules at 600 volts	Screen out	220 V, 3-phase, 60 Hz normal power is supplied to DC voltage supply unit and up to 60 volts DC output from the supply unit. Standard electrical power for vacuum pump operation, etc.
Kinetic energy	High energy (e.g., flywheel or centrifuge type equipment)	Screen out	Operating electrical motors for stirring, pump operation, etc.
Thermal energy	Not unusual or unique with only significant consequence contact burns (high temperature) or frostbite (low temperature).	Screen out	Hot surfaces from operating motors and approximately 90°C solution in dissolution tank from exothermic reaction and electric current passing through the solution.
High pressure	> 3,000 psig or ≥ 0.1 lb TNT (1.4 x 10 ⁵ ft-lb _f) equivalent energy	Screen out	Standard compressed gas cylinders < 3000 psig.

Type hazard	Keep if criteria are exceeded	Action (keep or screen out)	Action basis (includes inventory and form of all materials)
Lasers	Any Class IV, any Class IIIb with non-enclosed beam per American National Standards Institute Z-136.1	Screen out	None.
Potential energy	Not unusual or unique high potential energy.	Screen out	Elevated materials and equipment common to routine handling operations in light industry. No unusual or high potential energy hazards.

APPENDIX B
MO-99 RECOVERY IMPACTS ON
HOT CELLS UHE

B-1

APPENDIX B

MO-99 RECOVERY IMPACTS ON HOT CELLS UHE

AREA OF OPERATIONS: East Hot Cell

PROCESS/OPERATION: Recovery of Mo-99 from irradiated uranium metal target

EQUIPMENT/SYSTEMS:

1. Recovery skid
2. Nitrogen cover gas compressed cylinder and regulator
3. DC voltage supply
4. Hot cell ventilation exhaust system

OPERATIONS/PROCESSES/ACTIVITIES:

1. Loading/unloading materials into/from the recovery skid.
2. Dissolve aluminum fission-recoil barrier.
3. Rinse exposed uranium metal foil.
4. Dissolve uranium metal foil.
5. Precipitate fission products, actinides and uranium metal.
6. Vacuum transfer solutions between tanks.
7. Equipment maintenance, modification, and/or repair.

RELEASE PATHS:

1. Through the hot cell ventilation system to the outside environment.
2. Through cracks/penetrations/openings from the primary confinement to the building secondary confinement boundary and to the outside environment through the ventilation systems.
3. Through cracks/penetrations/openings from the primary confinement to the building secondary confinement boundary and to the outside environment through cracks/penetrations/openings in the secondary confinement boundary.

ASSUMPTIONS:

1. Facility workers are assumed to evacuate the immediate area for personal protection for events that are readily detectable and pose an obvious hazard (e.g., fire, explosions). This assumption is reasonable based on provisions of the Radiological Protection Program (radiological workers), Emergency Management Program (all workers), and Procedures and Training Program (e.g., General Employee Training for all workers) that ensure workers are aware of accidents/hazardous conditions that require evacuation.

INITIAL CONDITIONS:

1. Based on project need, the total amount of radioactive material that may be present in irradiated uranium target is limited to the following:

Inhalation dose equivalent:

One irradiated uranium metal target whose radioactive material inventory will be limited by the SAR Table 3-5 ($3a\text{-}^{238}\text{Pu-GB-}^{99}\text{Mo}$) limits.

Direct dose source:

It is difficult to compare external exposure MARs for different materials because of the varying energy levels of the radiation being emitted and the corresponding effectiveness of various shield materials. Therefore, facility and facility area limits are not set based on external dose potential.

Fissionable material:

< 700 g U^{235}FEM

2. The Radiological Protection Program ensures that training of workers that access radiological areas is provided including techniques to reduce exposure to existing or potential radiological conditions.
3. The Emergency Management Program determines the actions to take (e.g., evacuate, shelter in place) in response to an emergency including the required notifications.
4. The Procedures and Training Program ensures workers are trained to take the appropriate actions in response to an emergency.

Table B-1. Mo-99 Recovery Impacts on Hot Cells UHE

Event No.	Event	Causes	Freq	Consequence	Controls/Comments
Dissolution Tank and Precipitate Tank					
1. Aluminum Fission-Barrier Dissolution, (2) Uranium Metal Dissolution, and (3) Precipitation Process					
B-1	Cover gas flow stops	Operator error Equipment failure Supply tank empty	A	<p>Vacuum pump will continue to maintain vacuum and dissolution off-gases will flow through radioiodine filter and exhaust to hot cell without being diluted by the cover gas flow. The dissolution process would not have started unless the cover gas had been established so the air and its oxygen would have been evacuated. The hydrogen concentration in the tank would increase but the lack of oxygen would prevent an explosion although oxygen from inleakage would occur. The uranium metal dissolution also causes the hydrolysis of water which will produce both hydrogen and oxygen. If an explosion did occur it would only cause local damage to the Mo-99 recovery equipment because of the low volumes of hydrogen being produced. The radioactive material inventory maintains the process inventory to levels such that accidents do not exceed the evaluation guidelines. If an explosion occurred, the consequences would be in the low category.</p> <p>Off-site: Low On-site: Low Facility Worker: Low</p>	<p><i>The project operating instructions will ensure that the tank has been closed and that the cover gas flow from the tank through the radioiodine trap is established before the dissolution or precipitation starts.</i></p> <p><i>The project operating instructions for the uranium metal dissolution process will ensure the electric current is stopped until the cover gas flow is restored.</i></p> <p><i>One of the HEPA filters in an online HCV System exhaust header Filter Bank #1 and/or Filter Bank #2 will be replaced with a high efficiency gas absorber (HGA) filter containing activated charcoal as a defense-in-depth measure. The HEGA filter will be tested to ensure that its decontamination factor for elemental radioiodine is at</i></p>

Table B-1. Mo-99 Recovery Impacts on Hot Cells UHF

Event No.	Event	Causes	Freq	Consequence	Controls/Comments
B-2	Vacuum pump stops	Operator error Equipment failure Loss of electrical power	A	<p>The cover gas supply would continue to supply the cover gas and would cause the dissolution off-gases to flow out of the tank through the radiiodine trap. The process would start to operate under a positive pressure so the tank penetration leakages would allow some of the hydrogen and other gases to be released to the hot cell before passing through the radiiodine filter. The uranium metal dissolution also causes the hydrolysis of water which will produce both hydrogen and oxygen. The radioactive material inventory maintains the process inventory to levels such that accidents do not exceed the evaluation guideline. If increased leakage or an explosion occurred during uranium metal dissolution, the consequences would be in the low category. The radioactive material limits and the defense-in-depth HCV HEPA filters will limit the potential consequences.</p> <p>Off-site: Low On-site: Low Facility Worker: Low</p>	<p><i>The project operating instructions will ensure that the dissolution tank has a low leak rate before the dissolution starts.</i></p> <p><i>The project operating instructions for the uranium metal dissolution process will ensure the electric current until the vacuum pump is restored</i></p> <p><i>The HEPA filter will be tested to ensure that its decontamination factor for elemental radiiodine is at least 100.</i></p>
B-3	Tank stirring mechanism stops (or electric current stopped for uranium metal dissolution)	Operator error Equipment failure Loss of electrical power	A	<p>The failure of the stirring would make the dissolution process take a much longer time to complete.</p> <p>Off-site: Low On-site: Low Facility Worker: Low</p>	

Table B-1. Mo-99 Recovery Impacts on Hot Cells UHE

Event No.	Event	Causes	Freq	Consequence	Controls/Comments
B-4	Electrical current inadvertently applied during process (except for uranium metal dissolution)	Operator error Equipment failure	A	The electric current would not affect the dissolution process. The electric current would cause the dissolution of some water. The cover gas flow would prevent a buildup of oxygen and hydrogen. Off-site: Low On-site: Low Facility Worker: Low	
B-5	Tank leaks	Operator error Equipment failure	E (short duration process)	The solution would leak to the system catch pan and further dissolution would stop. The solution may contain up to all of the uranium metal, actinides and fission products. The radioactive material limits and the defense-in-depth HCV HEGA filters will limit the potential consequences. Off-site: Low On-site: Low Facility Worker: Low	<i>The Mo-99 recovery process skid has a catch pan that can contain the leaked contents of the system.</i> <i>The HEGA filter will be tested to ensure that its decontamination factor for elemental radioiodine is at least 100.</i>
B-6	Incorrect chemicals added to the tank for dissolution and precipitation	Operator error	A	The facility does not normally have the types of chemicals that would result in catastrophic consequences that would fill the tank. The chemicals for the process will be supplied from outside the facility so incorrect chemicals are possible. If incorrect chemicals were added and a highly reactive process occurred, a release of the tank inventory may happen. The radioactive material limits and the defense-in-depth HCV HEGA filters will limit the potential consequences. Off-site: Low On-site: Low Facility Worker: Low	<i>The operation procedure will have a specific step to confirm that the correct chemicals are being added.</i> <i>The HEGA filter will be tested to ensure that its decontamination factor for elemental radioiodine is at least 100.</i>

Table B-1. Mo-99 Recovery Impacts on Hot Cells UHE

Event No.	Event	Causes	Freq	Consequence	Controls/Comments
B-7	Iodine retention material fails to trap radioiodine	Wrong material installed or filters clog	A	<p>The radioactive iodine trap and HEGA filter will be tested but it is conservatively assumed that the filters may fail to trap the iodine. If the trap and filter fail, the radioiodine will be released to the 3039 Stack; however much of the radioiodine will remain in solution and the elemental iodine has a strong tendency to plate out on cool surfaces. The safety analysis assumes that the radioiodine is released at ground level; however, the radioiodine will most likely be released from the 250-ft high 3039 Stack.</p> <p>Off-site: Low On-site: Low Facility Worker: Low</p>	<p><i>The radioiodine trap will be tested prior to use to confirm that it has a decontamination factor for elemental radioiodine is at least 1000.</i></p> <p><i>The HEGA filter will be tested to ensure that its decontamination factor for elemental radioiodine is at least 100.</i></p>
B-8	Heavy load drops onto recover skid	Operator error Equipment failure	A	<p>The tank would fail and off-gas sweep would stop. The off-gases would be released to the hot cell. Any potential explosion from the hydrogen generation would be very localized and not damage the hot cell or any other safety-related equipment. The radioactive material limits and the defense-in-depth HCV HEGA filters will limit the potential consequences.</p> <p>Off-site: Low On-site: Low Facility Worker: Low</p>	
B-9	Earthquake, hot cell fire, etc.	As discussed in the SAR UHE tables.	See SAR UHE tables	The presence and operation of the Mo-99 recovery process does not impact the consequences of these events since the equipment does not have new release mechanisms or energy sources that would impact the evaluation of the existing events.	

Filtrate Tank and Waste Tank Liquid Retention					
B-10	Tank leaks	Equipment failure	A	<p>The solution would leak to the system catch pan. The solution will contain some of the aluminium, uranium, actinides, and fission products from the uranium metal. The radioactive material limits and the defense-in-depth HCV HEPA filters will limit the potential consequences.</p> <p>Off-site: Low On-site: Low Facility Worker: Low</p>	<p><i>The tank connection to the vacuum pump with the inline radioiodine filter is only method to provide a motive force (i.e., vacuum) to transfer the liquid between tanks.</i></p> <p><i>The HEPA filter will be tested to ensure that its decontamination factor for elemental radioiodine is at least 100.</i></p>

APPENDIX C
SAR Text Changes

C-1

The following changes need to be made to the SAR Revision 3a text to interface with and support the SBS:

1. (Section 3.3.2.1.1) The description of the radioactive materials in the facility needs to be updated to include the unirradiated uranium metal and the irradiated uranium metal containing radioiodine. The description of the powders and solutions radioactive materials needs to be updated to address the potential presence of radioiodine.
2. (Table 3-5) Table 3-5 needs to be updated to the Table 3-5 (3a-²³⁸Pu-GB-⁹⁹Mo) provided in this SBS.
3. (Section 3.3.2.3.4) Update the defense-in-depth discussion to address the allowed replacement of a HEPA filter in the K-15 exhaust header HEPA bank with a HEGA (i.e., charcoal) filter for the Mo-99 project.
4. (Section 4.4.3) Update the discussion of the IICV System to discuss that one of the HEPA filters in an online Filter Bank #1 or #2 may be replaced by a HEGA filter for the Mo-99 project.
5. (Section 5.5.1) Update the development of the Limiting Conditions for Operation (LCO) to discuss the allowable presence and impacts of the HEGA filter in the HCV System exhaust header HEPA bank downstream filter position.
6. (Section 5.4) Update the description of the listing for significant quantities of radioactive materials to include unirradiated uranium metal and irradiated uranium metals with the radioiodine.
7. (Appendices) Update the appendices to reflect changes to the radioactive material inventory that allow presence of unirradiated uranium metal and irradiated uranium metal with radioiodine.

ENCLOSURE 2

DOE COMMENTS AND RESOLUTIONS

Mo-99 SBS DOE Informal Comments and Resolutions

No	Section/pg	Comment	Discussion/Resolution
1	SAR Comments List of Acronyms and Abbreviations/ pg viii	HEGA is not included in list.	The list will be revised to include HEGA.
2	3.3.2/ pg 11	In last sentence of the first paragraph on the page, oxygen should be included with hydrogen as a gas swept from the dissolver tank since the electrolysis process will produce both hydrogen and oxygen.	The last sentence will be revised to identify both hydrogen and oxygen as being swept out of the tank by the gas sweep. Preliminary estimates indicate that approximately 0.3 ft ³ of hydrogen is generated from the dissolution and water radiolysis.
3	3.3.2/ pg 11	In the paragraph discussing the Waste Tank (T-2), the last sentence does not explicitly state that the solidification, evaporation or other acceptable method is one that is already approved in the current safety analysis.	The last sentence will be revised to state that the methods to disposition the liquid wastes are addressed in the current safety analysis.
4	3.3.3/ pg 13	In the last paragraph on the page, what method will be used to ensure that the flow rate through a HEGA filter does not exceed its 1000 cfm design limit?	Since the intended duration of the HEGA filters is for several weeks or months, a Standing Order will be issued addressing the system lineup.
5	4.1/ pg 14	The last sentence on the page does not explicitly state that the installation of the Mo-99 equipment, receipt of the irradiated target and movement into the hot cell, and the mechanical removal of the irradiate LEU foil from the target are routine activities that have been previously approved in the SAR.	The phrase "addressed in the current safety analysis" will be added to the subject paragraph.
6	4.4.1/ pg 16	The NCS revision is not identified as a permanent change to the NCS limits.	The NCSA revision will be identified as a permanent change and a statement will be added to provide DOE site office notification after completion of revision.

7	4.3/ pg 17	What impact will the installation of the charcoal filter have on the SAR HEPA filter smoke loading analysis?	The HEPA filters will be installed in the downstream HEPA filter bank filter slot. The HEPA filter, which will be in the upstream filter slot, will be the operable HEPA filter to meet the LCO Operability requirements. The HEPA filter in the upstream filter slot will not be affected by the installation of the HEPA filter in the downstream filter slot. Since the airflow through the HEPA filter will have been filtered by the HEPA filter, the smoke will have been removed and there will be no smoke impacts to the HEPA filter.
8	5.5/ pg 19	Does the HEPA filter impact SAR Section 5.5 for the HCV HEPA filter LCO?	SBS Section 5.5 will be revised to address the impacts of the temporary HEPA filter on the LCO development for the Hot Cell Ventilation System.
9	Table B-1/ multiple pgs	Are the consequences in the Unmitigated Hazards Evaluation (UHE) actually mitigated or unmitigated consequences?	The SBS Appendix B UHE consequences are unmitigated consequences. The expected Mo-99 annual target radioactive inventory was entered into the facility's Radioactive Material Inventory Control (RMIC) database to confirm that the radioactive materials are less than a Significant Quantity of Radioactive Material (SQRM). As discussed in SAR Rev 3a Section 5.4, the definition of SQRM is that quantity above which result in consequences greater than "Low" consequences per SAR Table 3-2. Since the Mo-99 annual target is less than a SQRM, the UHE consequences are in the "Low" consequence bin.
10	Appendix C/ pg C-2	Should SAR Section 5.5.1 for the HEPA temporary changes and the fire impact on the HEPA filters be included in the list of SAR changes?	Appendix C will be revised to include the SAR Section 5.5.1 to address the impacts from the HEPA filter and the HEPA filters' impact on the HEPA filter fire analysis.

TSR Comments			
1	1.1/ pg 1-5	How are the "Significant Quantities of Radioactive Materials" values derived?	<p>SAR Rev 3a Section 5.4 defines SQRM as that quantity above which result in consequences greater than "Low" consequences per SAR Table 3-2. SAR Table 3-2 defines "Low" consequences for radiological impacts as less than 1 rem TLDE for offsite receptors and less than 100 rem TEDE for onsite and facility receptors. The specific values are determined by reviewing calculation</p> <p>ORNL/3525/DAC/2007-01, <i>IEEE Building 3525 Unmitigated Hazard Evaluations Threshold Consequence Calculations</i>. For each radioactive material form, the limits provided in Table 2 column "Inhalation dose equivalent MAR (rem) for 100 rem onsite dose (LPF- 100%)" and Table 3 column "Inhalation dose equivalent MAR (rem) for 1 rem off-site dose (LPF - 100%)" are compared and the lowest value is the SQRM for that radioactive material form.</p> <p>Added the SAR location of the definition of the SQRM to the SBS Section 7.</p>

2	1.1/ pg 1-5	The TSR Rev 6 SQRM for powder, solution or unirradiated uranium metals is 4.44E+10 rem and the 2013 SAR Annual Update Rev 4d Section 5.4 SQRM is 4.53E+10 rem	<p>The SAR Section 5.4 lists the incorrect value of 4.53E+10 rem as the SQRM for the powder and solutions radioactive material form for both the currently implemented SAR Rev 3a and the annual update Rev 4d. The currently implemented TSR Rev 5d and the two draft TSRs Rev 6 and 7 correctly list the SQRM as 4.44E+10 rem. The facility RMIC also uses the correct SQRM of 4.44E+10 rem for the powders and solutions form. Since the correct value is listed in the TSRs and used in RMIC, the SAR values will be corrected.</p> <p>No changes to SBS or TSRs. The SAR will be revised for the next annual update.</p>
3	5.11.1.3/ pg 5-9	The TSR Basis states that if material capable of forming an explosive atmosphere or heavy loads capable of breaching the hot cell if dropped. The Table 3-5 states that if material can create a credible explosive atmosphere or if heavy loads capable of breaching hot cells if dropped.	The text will be revised to be consistent with the SAR Table 3-5 and SBS Table 3-5(3a- ²³⁸ Pu-GB- ⁹⁶ Mo).
4	Basis LCO 3.3.3/ pg Basis 3/4-4	Carol Solin required that HFIR justify the 12 time frame to reach Warm Standby for the LCO based on a risk justification.	The LCO basis will be revised to provide a risk based justification for the time frame. An ACTS Action will be initiated to review the other TSRs with the LCOs to review and update as necessary at the next annual update requiring a TSR revision.

ENCLOSURE 3
BUILDING 3525
TECHNICAL SAFETY REQUIREMENTS
ORNL/3525/TSR
REVISION 6

ORNL/3525/TSR
Revision 6

**TECHNICAL SAFETY REQUIREMENTS
IRRADIATED FUELS EXAMINATION LABORATORY
BUILDING 3525**

Approval Date: _____

Effective Date: _____

Prepared by the
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831
managed by
UT-BATTELLE, LLC
for the
U.S. DEPARTMENT OF ENERGY
under contract No. DE-AC05-00OR22725

ORNL/3525/TSR
Revision 6

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DOE APPROVALS

**TECHNICAL SAFETY REQUIREMENTS
IRRADIATED FUELS EXAMINATION LABORATORY
BUILDING 3525**

Approved by:

Johnny O. Moore Date 7/3/12
Manager, ORNL Site Office

ORNL/3525/TSR
Revision 6

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UT-BATTELLE, LLC, APPROVALS

TECHNICAL SAFETY REQUIREMENTS
IRRADIATED FUELS EXAMINATION LABORATORY
BUILDING 3525

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ORNL/3525/TSR
Revision 6

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REVISION LOG		
REVISION	DESCRIPTION	DATE APPROVED
0	Initial Issue	2-24-04
1	<ul style="list-style-type: none"> Section 1, Use and Application – Revised to indicate that the configuration management program applies to safety-class in addition to safety-significant and defense-in-depth systems, structures, and components (SSCs). Section 6, Design Features – Revised to indicate that hot cell structures are credited as safety-class design features for providing passive mitigation of criticality accident. 	6-30-04
2	<ul style="list-style-type: none"> Section 1.1 Definitions – Added definitions for Action(s), Calibrate/Calibration, Immediately, Mode(s), and Operable/Operability; Section 1.2 Operational Modes – Defined modes for operation of Building 3525; Section 1.3 Frequency Notation – Added section to define frequencies for surveillance requirements; Section 1.4 Operable/Operability Implementation Principles – Added section with standard general principles to define system operability; Section 3/4.0 General Application – Added section for clarification and additional guidance for LCOs and SRs; Section 3/4.1 Ventilation Systems – Added LCO for Hot Cell Ventilation (HCV) system; Section 5.4.4 Fire Protection – Revised to add standard wording as used in other ORNL non-reactor nuclear facility TSR administrative controls; Section 5.4.6 Configuration Management – Revised to add standard wording as used in other ORNL non-reactor nuclear facility TSR administrative controls; Section 5.3.9 Maintenance – Added Maintenance as an administrative control as a best management practice using standard wording used in other ORNL non-reactor nuclear facility TSR administrative controls; Section 5.10 Facility Inventory Control – Added restrictions for dispersible material operations; Section 5.11 Deviation from TSRs – Revised to indicate the applicable DOE order for occurrence reporting; and Appendix A Technical Safety Requirement Bases – Added bases for each Building 3525 LCO and SR. 	Rev 2A submitted without Rev 2 approval
2A	<ul style="list-style-type: none"> Section 1.1, Definitions – Added definitions for Dispersible Material and Specific Administrative Control. Revised text throughout to capitalize these terms at each use. Section 1.2, Operation Modes – Deleted reference to SAR as source of definition for Dispersible Material because it is now included in the TSR. Deleted operational status of HCV system from operational mode. Section 5.10, Facility Inventory Control – Deleted entire section and renumbered following sections accordingly. This section is no longer needed because, except for the fissionable material controls, the requirements are captured in LCOs or Specific Administrative Controls. The specific fissionable material limits that were deleted were not identified as requiring TSR coverage (other than application of the NCS program) in Chapter 5 of the SAR. Section 5.12, Specific Administrative Controls – Added new section to address Specific Administrative Controls. The controls addressed include those facility inventory controls that were previously addressed in Section 5.10. 	Rev 2B submitted without Rev 2A approval

REVISION LOG		
REVISION	DESCRIPTION	DATE APPROVED
2B	<ul style="list-style-type: none"> Acronyms - Added CCCTF, GMR, HESA, HFIR, IMGA, MAR, SEM. Section 1.1, Definitions - Deleted "Dispersible Materials" and "Calibrate/Calibration." Revised definition for "Immediately" to note action is as soon as safely possible. Added definition for "Significant Quantities of Radioactive Material." Section 1.2, Operational Modes - Revised definition of Operation and Standby modes to incorporate significant quantities of radioactive material. Section 1.3, Frequency Notations - Deleted "Immediately" because it is not a surveillance frequency. LCO 3.1.1 - Incorporated revised operational modes. Revised process area applicability to be consistent with revised analysis in the safety analysis report (SAR). Corrected Condition A. Deleted Condition B, associated surveillance requirement, and associated basis (renumbered steps as applicable). Section 5.3, Procedures - Deleted requirement for specific transport carrier handling procedures. Section 5.4.4, Fire Protection - Added requirement for facility-specific combustible control program credited in the SAR. Section 5.4.9, Initial Testing, In-service Surveillance, and Maintenance - Revised title of program to match the description in the SAR. Section 5.4.10, Hoisting and Rigging - Added program that was credited in the SAR. Deleted Section 5.11, Transport Carrier Handling Program - This program is no longer credited in the SAR. Deleted Section 5.12.2, Dispersible Material Protection - This administrative control is no longer credited in the SAR. Section 6.0, Design Features - Reformatted section. Deleted function of the hot cells to provide shielding following a nuclear criticality accident because this function is no longer credited in the SAR. Added SEM Cell, HESA and Decontamination Area structures, Shielded Metallograph, and Shielded Hardness Tester as credited design features. Deleted Dispersible Material Containers because they are no longer credited in the SAR. Bases - Updated consistent with changes to LCO 3.1.1. <p>Because of the extent of revisions accumulated in Revisions 2, 2A, and 2B, revision bars are not used.</p>	Rev 2C submitted without Rev 2B approval
2C	<ul style="list-style-type: none"> Section 1, Use and Application - Added spent fuel oxidation and processing to activities conducted at the HFIR. Section 1.2, Operational Modes - Added a warm standby and cold standby mode and definitions of each. Clarified operations mode to agree with new modes. Added a note that the HESA and Decontamination cells are considered part of the main hot cells for purposes of the TSRs if the main hot cell roof plugs are removed. Section 1.3, Frequency Notation - Added Daily, Weekly, and Quarterly frequencies to support new LCO surveillances. 	10-18-07

REVISION LOG		
REVISION	DESCRIPTION	DATE APPROVED
2C (cont'd)	<ul style="list-style-type: none"> LCO 3.1.1 – Revised LCO to require operability of all HEPA filters. Deleted note allowing delta-p to fall below surveillance requirement for up to 30 minutes without entry into LCO. Increased time for action in response to Condition A to 4 h to be consistent with Condition C. Revised action C.1 to indicate inoperable HEPA filter must be isolated. Revised mode to be entered on failure to complete actions to warm standby. Updated associated bases to reflect LCO changes. Added LCO 3.1.2, associated surveillances and bases to address oxidation furnace off-gas line HEPA filter. Added LCO 3.1.3, associated surveillances and bases to address oxidation furnace gas-supply-line and gas-recirculation-line check valves. Section 5.1, Contractor Responsibility – Revised to indicate facility manager designee is a written delegation. Section 5.3, Procedures – Added that procedures are necessary to address the safety management programs described in the TSR. Section 5.4.1, Nuclear Criticality Safety – included element to review NCS controls for identification as safety SSCs or SACKs. Section 5.4.4, Fire Protection – Deleted requirements for combustible control program to limit flammable materials. This requirement is implemented via a SAC. Section 5.4.11 – Added new section to address Conduct of Operations. Section 5.5, Minimum Operations Shift Complement – expanded justification of required staffing. Section 5.8, Record Keeping – Added list of records to be retained at a minimum. Section 5.9, Reviews and Audits – Added requirements for self-assessment program, independent reviews and audits, and safety system assessments. 5.11.1, Radioactive Material Limit – Revised title of SAC and wording within it to avoid confusion regarding inhalation dose limits and fissionable material mass limits. Added discussion of material limits during selected activities. Section 6.1, Main Hot Cell Structure – Deleted reference to full building height. Deleted reference to HFIR Core. Added description of east hot cell pass-through port. Section 6.5, HESA and Decontamination Area – Added note that the HESA and Decontamination Area are considered part of the main hot cells if the main hot cell roof plugs are removed. Section 6.9, Oxidation Furnace Gas Supply Line Flow Orifice – Added new design feature for safety as described in revisions to the SAR. Bases, 3.1.1, HCV System – Added a "Process Area Applicability" discussion. Bases, 3.1.1, HCV System – Clarified in the "Background" Section that the in-cell and in-line HEPA pre-filters are not safety significant. Bases, 3.1.1, HCV System – "Application to Safety Analysis" section rewritten to more adequately describe system function. Bases, 3.1.1, HCV System – Deleted justification for allowing delta-p to fall below surveillance requirement for up to 30 minutes without entry into LCO. 	

REVISION LOG		
REVISION	DESCRIPTION	DATE APPROVED
2C (cont'd)	<ul style="list-style-type: none"> Bases, 3.1.1, HCV System – Action statement A.1 – Revised to reflect change to 4-h action time and add justification for action time. Bases, 3.1.1, HCV System – Action statements B.1, C.1, D.1 – Revised to add more justification for action times. Bases, 3.1.1, HCV System – Revised “Surveillance Requirements” to add more justification for surveillance intervals. Throughout – Miscellaneous editorial changes <p>Because of the extent of revisions accumulated in Revisions 2, 2A, 2B and 2C, revision bars are not used.</p>	
3	<ul style="list-style-type: none"> LCO 3.1.2 Process Area Applicability – Added a phrase to indicate the LCO does not apply if the oxidation furnace is not connected to the off-gas line. LCO 3.1.2 Action Statement B.1 – Identified the valves that should be used to isolate the HEPA filter. LCO 3.1.2 Action Statement B.2 – Added an optional action for the inoperable HEPA to disconnect it from the off-gas line. SR 4.1.2.1 – Revised the surveillance frequency to quarterly. SR 4.1.2.2 – Replaced the “TBD” with “≤18 psig.” SR 4.1.2.5 – Replaced the entire surveillance requirement with a new surveillance requirement to annually test PRV-101.” LCO 3.2.1 Process Area Applicability – Added a phrase to indicate that LCO does not apply if the oxidation furnace is not connected to the gas supply/recirculation line. LCO 3.2.1 Action Statement B.1 – Identified the valves that should be used to isolate the check valves. LCO 3.2.1 Action Statement B.2 – Added an optional action for an inoperable check valve to disconnect the oxidation furnace from the off-gas line. Section 6.1 – Revised to correct cell dimensions. Section 6.2 – Revised to correct cell dimensions and the identification of a second window. Section 6.3 – Revised to correct the stainless steel liner thickness. Section 6.9 – Revised to identify the use of multiple orifices. Bases, LCO 3.1.2 Process Area Applicability – Included a statement that the LCO was applicable only if the oxidation furnace is connected to the off-gas line. Bases, LCO 3.1.2 Action Statement B.1 – Provided the bases for the valves identified to isolate the HEPA. Bases, LCO 3.1.2 Action Statement B.2 – Provided the bases for disconnecting the oxidation furnace from the off-gas line as an acceptable action for an inoperable HEPA. 	5-15-08

REVISION LOG		
REVISION	DESCRIPTION	DATE APPROVED
3 (cont'd)	<ul style="list-style-type: none"> Bases SR 4.1.2.1 – Revised the frequency from annually to quarterly and provide the reason for the changed frequency. Bases, SR 4.1.2.2 – Replaced the “TBD” with “≤18 psig.” Revised text to indicate that PRV-01 in conjunction with FE-01 and FE-02 limit the gas supply flow rate. Bases, SR 4.1.2.3 – Added a statement that inspection of no visible damage to the HEPA filter external cover meets the intent of the surveillance requirement. Bases SR 4.1.2.5 – Completely revised the bases to address the revised SR 4.1.2.5. LCO 3.2.1 Process Area Applicability – Added a phrase to indicate that LCO does not apply if the oxidation furnace is not connected to the gas supply/recirculation line. Bases, LCO 3.2.1 Action Statement B.1 – Provided the bases for the valves identified to isolate the check valves. Bases, LCO 3.2.1 Action Statement B.2 – Added a phrase to indicate that an optional action for an inoperable check valve is to disconnect the oxidation furnace from the off-gas line. 	
3a	<ul style="list-style-type: none"> SR 4.1.2.4 – Revised the SR to indicate it applied to the gas recirculation line relief valve (PRV-101). SR 4.1.2.5 – Revised the SR to indicate it applied to the gas recirculation line relief valve (PRV-101). Bases, SR 4.1.2.3 – Revised the discussion to provide the reason the visual inspection is required. Bases, SR 4.1.2.4 – Revised the discussion to state it applied to the gas recirculation line relief valve (PRV-101) and to provide the reason the visual inspection is required. Bases, LCO 3/4.2.1 Background – Changed the size of the lines from 1/4-in or 3/8-in to 1/2-in. 	Rev 4a submitted before Rev 3a approved

REVISION LOG		
REVISION	DESCRIPTION	DATE APPROVED
4	<ul style="list-style-type: none"> Table of Contents – Revised to add LCO 3/4.3, removed Section 5.11, and added Bases for LCO 3/4.3. Acronyms – Added Fissionable Mass Equivalent, Nuclear Criticality Safety Approval, and Nuclear Material Control and Accountability. Section 1.1, Definitions – Added definition for IMMEDIATELY and revised the definition of SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIAL to be consistent with the new radioactive material limits. Section 1.2 – Revised the definitions of OPERATION MODE and WARM STANDBY MODE such that the quantities of radioactive materials are “allowed to be present” rather than “are” present in the main hot cells and that active operations may be conducted rather than are being conducted. Section 1.3, Frequency – Added definitions for MONTHLY and BIENNIALY. LCO 3.0.3 – Clarified that statement to indicate that the STANDBY MODE addressed in the LCO statement is the WARM STANDBY MODE. SR 4.1.2.5 – Corrected description of PRV-101. LCO 3.3.1 – Added LCO for Radioactive Material Limit. Section 5.11 – Deleted because the requirement was replaced with an LCO. Bases – (Several locations) changed “OPERATIONS” to “OPERATION.” Bases, LCO 3.0.3 – Clarified that statement to indicate that the STANDBY MODE addressed in the LCO statement is the WARM STANDBY MODE. Bases, LCO 3.1.1 – Added a justification for the use of the engineering judgment of the minimum hot cell pressure to confirm hot cell ventilation system operation. Bases, LCO 3.3.1 – Added bases for LCO 3.3.1. 	Rev 4a submitted before Rev 4 approved
4a	<ul style="list-style-type: none"> Rev 4a combines most of the changes for Rev 3a and Rev 4 in a single Rev 4a with the primary exception of not adding an LCO for radioactive material inventory control. Several other new changes were added. Section 1.1, Definitions – Added definition for IMMEDIATELY and revised the definition of SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIAL to be consistent with the new radioactive material limits and to remove the phrase “in the main hot cells.” Section 1.2 – Revised the definitions of OPERATION MODE and WARM STANDBY MODE such that the quantities of radioactive materials are “allowed to be present” rather than “are” present in the main hot cells and that active operations may be conducted rather than are being conducted. LCO 3.0.3 – Clarified that statement to indicate that the STANDBY MODE addressed in the LCO statement is the WARM STANDBY MODE. LCO 3/4.4.1 – Changed the names of the filter banks and stated that the LCO only applied to online filter banks. SR 4-1-1-2 – Revised “filters” to “filter.” SR 4.1.2.4 – Revised the SR to indicate it applied to the gas recirculation line relief valve (PRV-101). SR 4.1.2.5 – Revised the SR to indicate it applied to the gas recirculation 	Rev 4b submitted before Rev 4a approved

REVISION LOG		
REVISION	DESCRIPTION	DATE APPROVED
	<p>line relief valve (PRV-101).</p> <ul style="list-style-type: none"> Section 6.9- Changed the size of the line from 1/4-in or 3/8-in to 1/2-in. Bases - (Several locations) changed "OPERATIONS" to "OPERATION." Bases, LCO 3.0.3 - Clarified that statement to indicate that the STANDBY MODE addressed in the LCO statement is the WARM STANDBY MODE. Bases, LCO 3/4.4.1.1 - Corrected the last sentence in the first paragraph to state the filters are in series and not in parallel. Revised the sentence to delete the phrase "and the Hood Exhaust System" from the sentence identifying non-hot cell inputs into the ventilation system since this system is inactive. Bases, LCO 3/4.4.1 LCO - Changed the names of the filter banks and stated that the LCO only applied to online filter banks. Defined the requirements for a filter bank to not be considered online. Bases, LCO 3/4.2.1 Background - Changed the size of the lines from 1/4-in or 3/8-in to 1/2-in. Bases, SR 4.1.2.3 - Revised the discussion to provide the reason the visual inspection is required. Bases, SR 4.1.2.4 - Revised the discussion to state it applied to the gas recirculation line relief valve (PRV-101) and to provide the reason the visual inspection is required. 	
4b	<p>Rev 4b scope of change removes all of the scope identified in Rev 3a, Rev 4, and Rev 4a except for (1) changes to the LCO 3/4.4.1 to resolve Occurrence Report SC-ORO-ORNL-X10NUCLEAR-2010-002, "PISA Related to Bldg 3525 Hot Cell Ventilation System," and (2) a change to LCO 3/4.4.1 to permit only one bank of HCV HEPA filters to be online and operable. The specific changes to the TSR are:</p> <ul style="list-style-type: none"> Section 1.2 - Deleted Note (1). LCO 3/4.4.1 - Changed the names of the filter banks and stated that the LCO only applied to online filter banks. Changed the hot cell pressure requirement from 0.1 in. of water to 0.6 in. of water. Added a requirement that the charging area be no more than 0.3 in. of water. Revised the Process Area Applicability to be consistent with Section 1.2 with Note (1) deleted. Revised the Actions to be consistent with the LCO revision. SR 4.1.1.1 - Revised 0.1 in. of water to 0.6 in. of water. SR 4.1.1.2 - Revised "filters" to "filter." SR 4.1.1.3 - Added the new surveillance requirement to address the added requirement associated with the charging area pressure. Bases, LCO 3/4.4.1 LCO - Revised the LCO bases to be consistent with the revisions to the LCO statement and requirements. Changed the names of the filter banks and stated that the LCO only applied to online filter banks. Defined the requirements for a filter bank to not be considered online. 	11-10-10
4c	<p>Rev 4c scope of change is the scope of Rev 4a that was added to the changes made in Rev 4b with the addition of LCO 3/4.3.1 "Hot Cell Roof Plugs," and other changes. Removed the Rev 4a changes related to LCO 3/4.4.1 since those changes were made in Rev 4b. Added LCO 3/4.3.1, revised Section 5.10, and added the roll-around shield as a design feature. The specific changes to the TSR are:</p>	9/21/11

REVISION LOG		
REVISION	DESCRIPTION	DATE APPROVED
	<ul style="list-style-type: none"> • TOC – Updated for revisions. • Acronyms – Added acronyms for personal protection equipment and Radiological Control Technician. • Section 1.1, Definitions – Added definition for IMMEDIATELY, CALIBRATE, and FUNCTIONAL TEST and revised the definition of SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIAL to be consistent with the new radioactive material limits and to remove the phrase “in the main hot cells.” • Section 1.2 – Revised the definitions of OPERATION MODE and WARM STANDBY MODE such that the quantities of radioactive materials are “allowed to be present” rather than “are” present in the main hot cells and that active operations may be conducted rather than are being conducted. • LCO 3.0.3 – Clarified the statement to indicate that the STANDBY MODE addressed in the LCO statement is the WARM STANDBY MODE. • LCO 3/4.1.1 – Revised to add 3039 Stack pressure, 3039 Stack pressure alarm, and several dampers position to the OPERABILITY requirements. • SR 4.1.1.4 – 4.1.1.6 – Added SRs. • SR 4.1.2.1 and SR 4.1.2.3 – Revised the SRs to indicate they applied to the HEPA filter HF-102. • SR 4.1.2.4 – Revised the SR to indicate it applied to the gas recirculation line relief valve (PRV-101). • SR 4.1.2.5 – Revised the SR to indicate it applied to the gas recirculation line relief valve (PRV-101). • LCO 3/4.3.1 – Added new LCO for the hot cell roof plugs. • Section 5.10 – Deleted the first sentence of the section. • Section 6.9 – Changed the size of the line from 1/4-in or 3/8-in to 1/2-in. • Section 6.10 – Added a new section to discuss the roll-around shield. • Bases – (Several locations) changed “OPERATIONS” to “OPERATION.” • Bases, LCO 3.0.3 – Clarified that statement to indicate that the STANDBY MODE addressed in the LCO statement is the WARM STANDBY MODE. • Bases, LCO 3/4.1.1 – Revised the bases to reflect the additions made to the LCO statement. • Bases, SR 4.1.2.3 – Revised the discussion to provide the reason the visual inspection is required. • Bases, SR 4.1.2.4 – Revised the discussion to state it applied to the gas recirculation line relief valve (PRV-101) and to provide the reason the visual inspection is required. • Bases, SR 4.1.2.5 – Revised the discussion to identify the gas recirculation line relief valve as PRV-101. • Bases, LCO 3/4.2.1 Background – Changed the size of the lines from 1/4-in or 3/8-in to 1/2-in. • Bases, LCO 3/4.3.1 – Added bases section for the LCO. 	

REVISION LOG		
REVISION	DESCRIPTION	DATE APPROVED
5	<p>(2009 Annual Update) The revision bars for Rev 5 of the TSR assumes that TSR Rev 4a is approved by DOE and that TSR Rev 3a and Rev 4 have not been approved by DOE. The specific changes to the TSR are:</p> <ul style="list-style-type: none"> • Table of Contents – Revised to add LCO 3/4.3, removed Section 5.11, and added Bases for LCO 3/4.3, • Acronyms – Added Fissionable Equivalent Mass, Nuclear Criticality Safety Approval, and Nuclear Material Control and Accountability, • Editorial correction to the definition of IMMEDIATELY, • Section 1.3, Frequency – Added definitions for MONTHLY and BIENNIALY, • LCO 3/4.1.1, Action Condition A, and SR 4.1.1.1 – Revised the hot cell minimum vacuum from -0.1 in. WG to -0.3 in. WG and added text to clarify that the -0.3 in WG is an indicated pressure, • LCO 3/4.3.1 – Added LCO for Radioactive Material Limit, • Section 5.11 – Deleted because the requirement was replaced with LCO 3/4.3.1, • Bases, LCO 3/4.4.1 LCO and Surveillance Requirements – Revised the hot cell minimum vacuum from -0.1 in. WG to -0.3 in. WG and added text to clarify that the -0.3 in WG is an indicated pressure, • Bases, LCO 3/4.3.1 – Added bases for LCO 3/4.3.1 	<p>Rev 5a submitted without Rev 5 approval</p> <p>Rev 4b and Rev 4c were submitted after this annual update and included the needed changes from this draft</p>
5a	<p>(2010 Annual Update) The revision bars for Rev 5a were added to those from Rev 5. The specific changes to the TSR are:</p> <ul style="list-style-type: none"> • Acronyms – Deleted FEM from the listing, • LCO 3/4.3.1 – Removed all requirements associated with tracking fissionable material from the LCO. • Section 5.3 – Deleted the phrase “and fissionable” from the first bullet. <p>Bases, LCO 3/4.4.1 LCO and Surveillance Requirements – Removed all discussions associated with tracking fissionable material from the bases.</p>	<p>Rev 5b submitted without Rev 5a approval</p> <p>Rev 4b and Rev 4c were submitted after this annual update and included the needed changes from this draft</p>
5b	<p>(2011 Annual Update) This revision is submitted as the subsequent revision to Rev 4c and assumes that Rev 4c is approved. Revised the LCO Statement for LCO 3/4.1.2 to state that the gas-supply-line relief valve and gas-recirculation-line relief valve must be operable in addition to the HEPA filter. Also updated the associated ACTION A to include the two relief valves. Updated the LCO 3/4.1.2 Bases to address the relief valve operability along with several other changes to update the text and add clarifying information.</p>	<p>Rev 5c submitted without Rev 5b approval</p>

REVISION LOG		
REVISION	DESCRIPTION	DATE APPROVED
5c	<p>(2012 Annual Update and SBS/3525/2012-01) This revision is submitted as the 2012 annual update and to make changes necessary to support SBS/3525/2012-01 and to correct the definition of SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIALS for powders or solutions. The TSR revision is based on the approved TSR Rev 4c and includes the changes from TSR draft Rev 5b. The specific changes are:</p> <ul style="list-style-type: none"> • Table of Contents updated to reflect changes, • Gaseous materials added to the definition of SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIALS and replaced 4.53E+10 rem for powders and solutions with 4.44E+10 rem., • Updated the definition of TSR VIOLATION, • MODE Definition Note 1 added to state that STANDBY MODE designates either WARM STANDBY MODE or COLD STANDBY MODE, • LCO 3.1.1 updated ACTION I to also permit the placing the affected PROCESS AREA in the STANDBY MODE. • LCO 3.1.2 updated to address relief valve OPERABILITY • Section 5.4.11 updated to reflect new DOE Order on Conduct of Operations, • Section 5.7 updated to reflect new DOE Order on Training, • Section 5.11.1 updated to reflect the radioactive material limits provided in SBS/3525/2012-01, • Section 6.11 added to address Rooms 127, 130, 208 and 209 as design features for safety, • Bases for LCO-3.1.1 and LCO-3.1.2 updated to reflect LCO changes, • Bases for LCO 3.3.1 updated to correct descriptive text for SRs. 	Rev 5d submitted without Rev 5c approval
5d	<p>This revision is submitted to make changes necessary to support SBS/3525/2010-02. The specific changes are:</p> <ul style="list-style-type: none"> • Section 5.11.1 updated to reflect the glove box radioactive material limits provided in SBS/3525/2010-02. 	8-13-12 (Implemented 12-3-12)
6	<p>This revision is submitted to make changes necessary to support SBS/3525/2013-01 and make several editorial corrections identified during Rev 4c implementation. The specific changes are:</p> <ul style="list-style-type: none"> • Definition of SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIAL updated to include unirradiated uranium metal and irradiated uranium metals. • Definition of CALIBRATE/CALIBRATION revised to change the word "responses" to "responds." • Section 5.11.1 updated to reflect the addition of unirradiated uranium metal and irradiated metal to the radioactive material limits by SBS/3525/2013-01. • Section 5.11.1.3 updated to change "capable" to "credible" for explosive gases. • LCO 3/4.3.1 ACTION A.2.1.1 revised to remove statement related to the fire watch. • LCO 3.0.3 Bases (1) had the phrase "STANDBY" changed to "WARM STANDBY" in two locations to make them consistent with the LCO statement and (2) provided justification for action times. • LCO 3/4.3.1 Bases MODE APPLICABILITY had word "only" added to make it consistent with the other similar statements. • LCO 3/4.3.1 Bases For ACTION STATEMENT A.1 had word "if" corrected to "or" to make the sentence grammatically correct. 	

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ACRONYMS

AC	Administrative Control
CCCTF	Core Conduction Cooldown Test Facility
COO	Conduct of Operations
CFR	Code of Federal Regulation
DOE	Department of Energy
GMR	Glove Maintenance Room
HCV	Hot Cell Ventilation
HESA	Hot Equipment Storage Area
IFEL	Irradiated Fuels Examination Laboratory
IMGA	Irradiated Microsphere Gamma Analyzer
LCO	Limiting Conditions for Operation
LCS	Limiting Control Setting
MAR	Material at Risk
NCS	Nuclear Criticality Safety
NNFD	Non-Reactor Nuclear Facilities Division
ORNL	Oak Ridge National Laboratory
PPE	Personal Protection Equipment
RCT	Radiological Control Technician
SL	Safety Limit
SAC	Specific Administrative Control
SAR	Safety Analysis Report
SEM	Scanning Electron Microscope/Microprobe
SR	Surveillance Requirements
SSC	Structure, System or Component
TSR	Technical Safety Requirement
USQ	Unreviewed Safety Question
USQD	Unreviewed Safety Question Determination

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SECTION 1

USE AND APPLICATION

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1.0 USE AND APPLICATION

The Irradiated Fuels Examination Laboratory (IFEL), Building 3525, at Oak Ridge National Laboratory (ORNL) is operated by the Nonreactor Nuclear Facilities Division (NNFD). The IFEL was initially designed and constructed to be used for metallurgical and metallographic studies, primarily in connection with the development of more economic fuels and blankets for power and high flux test reactors. Also, it was planned that the facility would be used for similar examinations of test loops and other reactor components which were sufficiently radioactive to require handling under shielded conditions and in radiological hot cells. Additionally, the facility was designed to be used to study the changes in and the composition of materials associated with the production of plutonium at high burn-up and to study the various phase and microstructural changes induced by thermal effects. Operations have included a number of routine examinations of a wide variety of highly radioactive fuel elements including the High Flux Isotope Reactor, the Oak Ridge Research Reactor, Peach Bottom Reactor, various light water reactor fuels, and prototypes for the high temperature gas-cooled reactor and liquid-metal fast breeder reactor fuels, and most recently, Keuring van Electrotechnische materialen (KEMA) Suspension Test Reactor fuel.

The current mission of the IFEL involves the receipt and handling of irradiated materials, processing of radioactive materials, transfer of materials, packaging and shipment of materials, waste packaging, maintenance of remote equipment, and decontamination of the facility and equipment. Examination and testing activities that occur within the facility hot cells include metrology, metallographic sample preparation, optical microscopy, scanning electron microscopy/microprobe, gamma spectrometry, and other physical properties evaluations as appropriate to the experimental objectives of the particular program. Processing activities include oxidation of spent nuclear fuel and dissolution of materials for further analysis and examination.

These Technical Safety Requirements (TSRs) represent a commitment by UT-Battelle, LLC, the managing contractor for ORNL, to the U. S. Department of Energy (DOE) regarding the safe operation of the IFEL. This document is prepared and formatted using the requirements and guidelines from Title 10 of the Code of Federal Regulations Part 830 (10 CFR 830) Subpart B, "Safety Basis Requirements" and DOE G 423.1-1, "Implementation Guide for Use in Developing Technical Safety Requirements." These TSRs were derived in the latest revision of the IFEL Safety Analysis Report (SAR), ORNL/3525/SAR, "Safety Analysis Report Irradiated Fuels Examination Laboratory, Building 3525."

Various programs have been established at ORNL to maintain facility safety basis documents (i.e., SARs and TSRs) current and to ensure their proper use and control. A facility safety analysis program is established for the control of the safety of activities and operations involving hazards that have the potential for significant impact to workers and the public. The program includes the objectives of ensuring that appropriate safety analysis documentation is prepared, reviewed, approved, implemented, and maintained and that new activities, changes, and modifications are evaluated using the unreviewed safety question (USQ) process. A configuration management program is established at IFEL to ensure that safety-class, safety-significant, and defense-in-depth structures, systems, and components (SSCs) are identified, documented, and maintained. Facility personnel who use IFEL safety documents are trained in their use and to identify when changes need to be made to these documents. Facility safety documents are maintained as controlled documents, and those personnel who are authorized to use these documents are identified.

1.1 DEFINITIONS

Note: Defined terms in this list appear in uppercase throughout these Technical Safety Requirements

TERM	DEFINITION
ACTION(S)	The steps listed in each SAFETY LIMIT, LIMITING CONTROL SETTING, and LIMITING CONDITION FOR OPERATION that are required to be performed within specific completion times when the specified SAFETY LIMIT, LIMITING CONTROL SETTING, or LIMITING CONDITION FOR OPERATION is not met.
ADMINISTRATIVE CONTROL (AC)	The provisions relating to organization, management, procedures, record keeping, assessments, and reporting necessary to ensure safe operation of the facility.
CALIBRATE/ CALIBRATION	A system or component calibration is an adjustment of that system or component such that its output responds, within acceptable range and accuracy, to known values of the parameter which the system or component measures.
DESIGN FEATURES	Systems, structures, and components that are taken credit for in the accident analyses. They are normally passive characteristics of the facility not subject to change by operations personnel (e.g., shielding, structural walls, relative locations of major components, installed poisons, or special materials). DESIGN FEATURES are those permanently built-in features critical to safety that do not require, or infrequently require, maintenance or surveillance.
FISSIONABLE MATERIAL	Material that contains any combination of fissionable nuclides (e.g., ^{233}U , ^{238}Pu , or ^{239}Pu) and other isotopes of the same chemical elements in which a fission chain reaction can be self-sustaining.
FUNCTIONAL (FUNCTIONALLY) TEST	A test of a component or a system of components to assure that the component or system of components performs its required safety function(s). At a minimum the testing shall include, where applicable, failure mode testing (which include loss of utility services, e.g., air, electrical).
IMMEDIATELY	When the term IMMEDIATELY is used as a special completion time, the required ACTION is to be commenced without delay and continuously pursued in a controlled manner until complete. The use of IMMEDIATELY implies the highest sense of urgency.
INSPECTION	Examination of equipment and/or devices to ensure operability.
LIMITING CONDITIONS FOR OPERATION (LCO)	The lowest functional capability or performance level of safety-related structures, systems, components, and their support systems required for normal safe operation of the facility.
LIMITING	The settings on safety-related structures, systems, and components that control

Note: Defined terms in this list appear in uppercase throughout these Technical Safety Requirements

TERM	DEFINITION
CONTROL SETTING (LCS)	process variables to prevent exceeding SAFETY LIMITS.
MODE(S)	Any of the facility conditions specified in Section 1.2.
OPERABLE/ OPERABILITY	A system or component is considered OPERABLE when it and its support systems and components are capable of performing their intended function or intended support function, respectively. A system or component is considered OPERABLE when the required SURVEILLANCE has been accomplished in accordance with the required frequency, and there is no indication of inoperability.
PROCESS AREA	A designated area within the facility (e.g., East Cell, North Cell, West Cell).
SAFETY LIMIT (SL)	Limits on process variables associated with those physical barriers, generally passive, that are necessary for the intended facility function and which are found to be required to guard against the uncontrolled release of radioactivity and other hazardous materials. Process variables subject to SLs are measurable parameters that, individually or in combination, reflect the basic hazard for which controls or limits are specified.
SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIAL	<p>Total inventory (inhalation dose equivalent) of more than the following:</p> <ul style="list-style-type: none"> • Metal/Encased SNF: $1.54\text{E}-12$ rem • Intact metal alloy fuel and cermet targets: $1.32\text{E}+12$ • Segmented SNF: $5.83\text{E}+11$ rem • Segmented metal alloy fuel and cermet target: $5.49\text{E}+11$ • Powders, solutions or unirradiated uranium metals: $4.44\text{E}+10$ rem • Contaminated combustibles, HEPA filters or irradiated uranium metals: $4.66\text{E}+09$ rem • Gaseous materials: $7.62\text{E}+06$ rem <p>When multiple material forms are present, the sum of the ratios of each material form to its limit shall not exceed 1.0.</p>
SPECIFIC ADMINISTRATIVE CONTROL (SAC)	ADMINISTRATIVE CONTROLS that provide a specific preventive or mitigative function for accident scenarios identified in the SAR where the safety function has importance similar to, or the same as, the safety function of a safety SSC.
SURVEILLANCE REQUIREMENT (SR)	Requirements relating to testing, calibration, or INSPECTION to ensure that the necessary operability of systems and components is maintained or that operations are within the specified SLs, LCSs, and LCOs.

Note: Defined terms in this list appear in uppercase throughout these Technical Safety Requirements

TERM	DEFINITION
TSR VIOLATION	<p>A TSR VIOLATION is defined as including any one or combination of the following:</p> <ul style="list-style-type: none"> • Exceeding a SAFETY LIMIT; • Failure to complete an ACTION within the required time limit following exceeding an LIMITING CONTROL SETTING or failing to comply with an LIMITING CONDITION FOR OPERATION; • Failure to perform a surveillance within the required time limit; and • Failure to comply with an ADMINISTRATIVE CONTROL statement, including a directive action SPECIFIC ADMINISTRATIVE CONTROL. <p>Failure to comply with an ADMINISTRATIVE CONTROL statement is a TSR VIOLATION when either the ADMINISTRATIVE CONTROL is directly violated, as would be the case with not meeting minimum staffing requirements or not satisfying the control description of a directive action SPECIFIC ADMINISTRATIVE CONTROL, for example, or the intent of a referenced program is not fulfilled. To qualify as a TSR VIOLATION, the failure to meet the intent of the referenced program would need to be significant enough to render the documented safety analysis summary invalid.</p>

1.2 OPERATIONAL MODES

DOE G 423.1-1 defines preferred operational modes for nonreactor nuclear facilities. Modes should be established based on the minimum number required to distinguish between different facility conditions as dictated by required equipment operability and needed parameter limits. There are no SLs or LCSs as defined in 10 CFR 830 Subpart B required for safe operation of the IFEL. LCOs are only necessary when facility operations involve SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIALS.

Therefore, the following modes are defined for operations in Building 3525.

OPERATION –	SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIAL are allowed to be present in the main hot cells and operations other than passive storage of materials and equipment may be conducted in the main hot cells.
WARM STANDBY –	SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIAL are allowed to be present in the main hot cells and operations conducted in the main hot cells are limited to passive storage of materials and equipment.
COLD STANDBY –	SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIAL are not present in the main hot cells.

- Notes:
- (1) STANDBY MODE designates either WARM STANDBY or COLD STANDBY.
 - (2) “Passive storage of materials and equipment” is intended to exclude movement or handling of radioactive materials or equipment and/or application of external energy sources or

chemical agents to radioactive materials or equipment. It is not intended to exclude operation of normally energized monitoring, sampling, or ventilation systems (e.g., radiation monitors or detectors, thermocouples, pressure measurement instruments, HCV System) or require depressurization or shutdown of normally pressurized or energized utilities (e.g., process water, process air, electrical receptacles, lights).

1.3 FREQUENCY NOTATION

The frequency notations, as used in the SURVEILLANCE REQUIREMENTS and elsewhere, shall be defined as follows when included in this TSR:

<u>Notation</u>	<u>Frequency</u>
DAILY	At least once every 24 h.
WEEKLY	At least once every 7 days.
QUARTERLY	At least once every 90 days.
ANNUALLY	At least once every 365 days.

Note: SR 4.0.2 permits an extension of 25% to the specified frequency intervals as measured from the previous performance or as measured from the time a specified condition of the frequency is met. This extension is permitted to facilitate scheduling of surveillances and to allow surveillances to be performed at the best time for the operation of the facility. The extension is not intended to be used routinely to extend the specified frequency interval.

1.4 OPERABLE/OPERABILITY IMPLEMENTATION PRINCIPLES

The definition of OPERABLE/OPERABILITY is contained in Section 1.1. The general principles of OPERABLE/OPERABILITY are as follows:

- (1) A system is considered OPERABLE as long as there is an assurance that it is capable of performing its specified safety function(s).
- (2) A system can perform its specified safety function(s) only when all of its necessary support systems are capable of performing their related support functions.
- (3) Ensuring the capability of a system to perform a safety function is an ongoing and continuous process.
- (4) When a system designed to perform a certain safety function is not capable of performing that safety function, a loss of function condition exists. Facility operation shall be controlled through specific ACTIONS and completion times detailed in the LCO.
- (5) When a system is determined to be incapable of performing its intended safety function(s), the declaration of inoperability shall be performed immediately.
- (6) Any exception to an immediate declaration of inoperability shall be justified.

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SECTION 2

SAFETY LIMITS

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2.0 SAFETY LIMITS

There are no SLs for the IFEL.

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SECTION 3/4

**LIMITING CONTROL SETTINGS,
LIMITING CONDITIONS FOR OPERATION
AND SURVEILLANCE REQUIREMENTS**

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3/4.0 GENERAL APPLICATION

LIMITING CONDITIONS FOR OPERATION

Clarification and additional guidance for the use of these LCOs and SRs are contained in Appendix A.

- LCO 3.0.1** LCOs shall be met during all MODES or other specified conditions in the Applicability, except as provided in LCO 3.0.2.
- LCO 3.0.2** Upon discovery of failure to meet an LCO, the associated ACTIONS shall be met. If the LCO is restored before the specified completion time(s) expires, completion of the ACTION is not required, unless otherwise stated.
- LCO 3.0.3** When an LCO statement is not met and the associated ACTIONS are not met, or an associated ACTION is not provided, the affected facility areas shall be placed in a MODE or other specified condition in which the LCO is not applicable. If the LCO is applicable in all MODES, the affected facility areas shall be placed in the safest MODE. ACTIONS shall be initiated within 1 h to place the affected facility areas in a condition in which the LCO is not applicable or WARM STANDBY MODE. The affected facility areas shall be in a condition in which the LCO is not applicable or WARM STANDBY MODE within 12 h. Where corrective measures are completed that permit operation in accordance with the LCO or ACTIONS, completion of the ACTIONS required by LCO 3.0.3 is not required. LCO 3.0.3 is applicable to all MODES. Exceptions to LCO 3.0.3 may be stated in the individual LCOs.
- LCO 3.0.4** When an LCO is not met, a MODE or other specified condition in the Applicability shall not be entered, except when the associated ACTIONS to be entered permit continued operation in that MODE or other specified condition in the Applicability for an unlimited period of time. LCO 3.0.4 shall not prevent changes in MODES or other specified conditions in the Applicability that are required to comply with ACTIONS. Exceptions to LCO 3.0.4 are stated in the individual LCOs. When an individual LCO states that LCO 3.0.4 does not apply, it allows entry into MODES or other specified conditions in the Applicability when the associated ACTIONS to be entered permit operation in the MODE or other specified condition for only a limited time.
- LCO 3.0.5** Equipment removed from service or declared inoperable to comply with ACTIONS may be returned to service under administrative control solely to perform testing required to demonstrate its OPERABILITY or the OPERABILITY of other equipment. This is an exception to LCO 3.0.2 for the system returned to service under administrative control to perform the testing required to demonstrate OPERABILITY.
- LCO 3.0.6** When a support system is inoperable and an LCO for that support system is specified in the TSRs, the supported system is not required to be declared inoperable due solely to support system inoperability. Only the support system's ACTIONS are required to be entered. This is a clarification of the definition of OPERABILITY. LCO 3.0.2 is still applicable to the supported system.
-

SURVEILLANCE REQUIREMENTS

- SR 4.0.1** SRs shall be met during the MODES or other specified conditions in the Applicability for individual LCOs unless otherwise stated in the SR. Failure to meet a SR (whether such failure is experienced during the performance of the surveillance or between performances of the surveillance) shall constitute failure to meet the LCO. Failure to perform a surveillance within the specified frequency shall constitute failure to meet the LCO, except as provided in SR 4.0.3. Surveillances do not have to be performed on inoperable equipment or variables outside specified limits.
- SR 4.0.2** The specified frequency for each SR is met if the surveillance is performed within 1.25 times the interval specified in the frequency, as measured from the previous performance or as measured from the time a specified condition of the frequency is met. For frequencies specified as "once," the above interval extension does not apply. If a completion time requires periodic performance of "once every ...," the above frequency extension applies to each performance after the initial performance.
- SR 4.0.3** If it is discovered that a surveillance was not performed within its specified frequency, compliance with the requirement to declare the LCO not met may be delayed from the time of discovery up to 24 h or up to the limit of the specified frequency, whichever is less. This delay period is permitted to allow performance of the surveillance. If the surveillance is not performed within the delay period, the LCO shall IMMEDIATELY be declared not met, and the applicable ACTIONS shall be taken. The completion times of the ACTIONS begins IMMEDIATELY on the expiration of the delay period. When the surveillance is performed within the delay period and the surveillance is not met, the LCO shall IMMEDIATELY be declared not met, and the applicable ACTIONS shall be taken. The completion times of the ACTIONS begins IMMEDIATELY on failure to meet the surveillance.
- SR 4.0.4** Entry into a MODE or other specified condition in the Applicability of an LCO shall not be made unless the LCO's surveillances have been met within their specified frequency. This provision shall not prevent passage through or to MODES or other specified conditions in compliance with ACTIONS.
-

3/4.1 VENTILATION/FILTRATION SYSTEMS

3/4.1.1 Hot Cell Ventilation (HCV) System

LCO: The HCV System shall be OPERABLE with

1. At least one HEPA filter in each online HEPA filter bank OPERABLE (i.e., one of K-15.12-1 or K-15.12-2 in Filter Bank #1 and one of K-15.12-3 or K-15.12-4 in Filter Bank #2),
2. hot cell pressures at least 0.6 in. of water lower than the pressure in the operating area,
3. charging area pressure no more than 0.3 in. of water lower than the pressure in the operating area,
4. 3039 Stack vacuum supply header at least 3.8 in. of water lower than atmospheric pressure and has an OPERABLE alarm system that alarms before the supply header is less than 3.8 in. of water lower than atmospheric pressure, and
5. damper K-15-V-10 is closed and damper K-15-V-11 is open.

MODE APPLICABILITY: OPERATION

PROCESS AREA APPLICABILITY: North, East, and West hot cells.

ACTIONS

CONDITIONS	REQUIRED ACTION	COMPLETION TIME
A. Pressure in a hot cell is not at least 0.6 in. of water lower than pressure in operating areas	A.1 Restore negative pressure in hot cell	4 h
B. The ACTION and associated completion time of Condition A is not met	B.1 Place affected PROCESS AREAS in WARM STANDBY	8 h
C. Both HEPA filters in an online HEPA filter bank are inoperable	C.1 Restore at least one HEPA filter in the online HEPA filter bank to OPERABLE condition	4 h
D. The ACTION and associated completion time of Condition C is not met	D.1 Place affected PROCESS AREAS in WARM STANDBY	8 h
E. Pressure in charging area is more than 0.3 in. of water lower than pressure in operating areas	E.1 Reduce negative pressure in charging area	4 h

CONDITIONS	REQUIRED ACTION	COMPLETION TIME
F. The ACTION and associated completion time of Condition E is not met	F.1 Place affected PROCESS AREAS in WARM STANDBY	8 h
G. Pressure in 3039 Stack vacuum header is not at least 3.8 in. of water below atmospheric pressure, damper K-15-V-10 is not closed, or K-15-V-11 is not open.	G.1 Increase negative pressure in 3039 Stack vacuum header and/or return the dampers to the correct open/closed positions	4 h
H. The ACTION and associated completion time of Condition G is not met	H.1 Place affected PROCESS AREAS in WARM STANDBY	8 h
I. The 3039 Stack vacuum header alarm is INOPERABLE	I.1 Perform SR 4.1.1.4 HOURLY OR I.2 Place affected PROCESS AREAS in STANDBY MODE	IMMEDIATELY

SURVEILLANCE REQUIREMENTS

SURVEILLANCE REQUIREMENT	FREQUENCY
SR 4.1.1.1 Verify the hot cells pressures at least 0.6 in. of water lower than the pressure in the operating area	DAILY
SR 4.1.1.2 Verify at least one HEPA filter in each online HEPA filter bank to be at least 99.95% efficient for removal of test aerosol	ANNUALLY <u>AND</u> after replacement and/or maintenance that could affect HEPA filter OPERABILITY
SR 4.1.1.3 Verify the charging area pressure not more than 0.3 in. of water lower than the pressure in the operating area	DAILY
SR 4.1.1.4 3039 Stack vacuum header is at least 3.8 in. of water lower than atmospheric pressure	DAILY
SR 4.1.1.5 CALIBRATE and FUNCTIONALLY TEST 3039 Stack vacuum header alarm instrument loop	ANNUALLY <u>AND</u> after replacement and/or maintenance that could affect alarm OPERABILITY
SR 4.1.1.6 Verify that damper K-15-V-10 is closed and damper K-15-V-11 is open	DAILY

3/4.1 VENTILATION/FILTRATION SYSTEMS

3/4.1.2 Oxidation Furnace Off-Gas Line HEPA Filter

LCO: The oxidation furnace off-gas line HEPA filter (HF-102), the gas-supply-line relief valve (PRV-01), and the gas-recirculation-line relief valve (PRV-101) shall be OPERABLE.

MODE APPLICABILITY: OPERATION

PROCESS AREA APPLICABILITY: North hot cell with SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIAL in the oxidation furnace and it is connected to the off-gas line.

ACTIONS

CONDITIONS	REQUIRED ACTION	COMPLETION TIME
A. Inoperable HEPA filter in oxidation furnace off-gas line, gas-supply-line relief valve, or gas-recirculation-line relief valve	A.1 Restore HEPA filter, gas-supply-line relief valve, and gas-recirculation-line relief valve to an OPERABLE condition	4 h
B. The ACTION and associated completion time of Condition A is not met	B.1 De-energize oxidation furnace and isolate the HEPA filter downstream of the oxidation furnace by closing valve HV-105 or HV-201 OR B.2 Disconnect the oxidation furnace from the off-gas line	4 h

SURVEILLANCE REQUIREMENTS

SURVEILLANCE REQUIREMENT		FREQUENCY
SR 4.1.2.1	Verify HEPA filter (HF-102) to be at least 99.95% efficient for removal of a test aerosol	QUARTERLY <u>AND</u> after maintenance that could affect HEPA filter OPERABILITY
SR 4.1.2.2	Verify gas-supply-line relief valve (PRV-01) opens at ≤ 18 psig.	ANNUALLY <u>AND</u> after maintenance that could affect relief valve OPERABILITY
SR 4.1.2.3	Visually inspect the HEPA filter (HF-102) housing/assembly and verify that no visible damage exists that would indicate potential damage to filtering capabilities	WEEKLY
SR 4.1.2.4	Visually inspect the gas-recirculation-line relief valve (PRV-101) and verify that no visible damage exists that would indicate potential damage to relief capabilities	WEEKLY
SR 4.1.2.5	Verify gas-recirculation-line relief valve (PRV-101) opens at ≤ 15 psig	ANNUALLY <u>AND</u> after maintenance that could affect relief valve OPERABILITY

3/4.2 CHECK VALVES

3/4.2.1 Oxidation Furnace Gas-Supply-Line and Gas-Recirculation-Line Check Valves

LCO: The oxidation furnace gas-supply-line check valve (CV-101) and gas-recirculation-line check valve (CV-102) shall be OPERABLE.

MODE APPLICABILITY: OPERATION

PROCESS AREA APPLICABILITY: North hot cell with SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIAL in the oxidation furnace and it is connected to the gas supply/recirculation line.

ACTIONS

CONDITIONS	REQUIRED ACTION	COMPLETION TIME
A. Inoperable check valve in oxidation furnace gas supply line or gas recirculation line	A.1 Restore check valve to OPERABLE condition	4 h
B. The ACTION and associated completion time of Condition A is not met	B.1 De-energize oxidation furnace and isolate affected gas supply line by closing valve HV-19 and/or gas recirculation line with valve HV-101, or HV-102, or HV-107 and HV-104, or HV-247 and HV-201 OR B.2 Disconnect the oxidation furnace from the gas supply/recirculation line	4 h

SURVEILLANCE REQUIREMENTS

SURVEILLANCE REQUIREMENT		FREQUENCY
SR 4.2.1.1	Perform a direct-pressure bubble test verifying the check valves close on reverse flow and there is no continuous formation of bubbles	Annually <u>AND</u> after maintenance that could affect check valve OPERABILITY
SR 4.2.1.2	Visually inspect the check valves and verify that no visible damage exists that would indicate potential damage to check valve capabilities	WEEKLY

3/4.3 HOT CELL ROOF PLUGS

3/4.3.1 Hot Cell Roof Plugs

LCO: The hot cell roof plug or a HESA/Decontamination Cell shield door must be installed/shut to form a closed boundary for each of the hot cells.

MODE APPLICABILITY: OPERATION

PROCESS AREA APPLICABILITY: North, East, and West hot cells.

ACTIONS

CONDITIONS	REQUIRED ACTION	COMPLETION TIME
A. Roof plug and shield door(s) for a hot cell not installed or closed while in the OPERATION MODE	A.1.1 (If The Ships Door Is Closed) Confirm GMR differential pressure at least 0.3 in. of water below the GMR airlock	2 h
	OR	
	A.1.2 (If The Ships Door Is Open) Confirm GMR airlock differential pressure at least 0.3 in. of water below the operating area	8 h
	AND	
	A.2. 1.1 Confirm Fire Alarm Control Panel has no alarms and no inputs bypassed.	
	AND	
	A.2. 1.2 Confirm sprinkler system operational by completing the monthly fire department inspection which has no failures recorded	
	AND	
	A.2. 1.3 Establish a fire watch for hourly inspections of the building second floor area	
	OR	
	A.2.2 Place the facility in the STANDBY Mode	

SURVEILLANCE REQUIREMENTS

SURVEILLANCE REQUIREMENT		FREQUENCY
SR 4.3.1.1	(West hot cell) the west hot cell roof plugs must be installed or the HESA shield door closed or the decontamination cell shield door closed.	DAILY
SR 4.3.1.2	(North hot cell) the north hot cell roof plugs must be installed or the HESA shield door closed or the decontamination cell shield door closed.	DAILY
SR 4.3.1.3	(East hot cell) the east hot cell roof plugs must be installed or the decontamination cell shield door closed.	DAILY

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SECTION 5

ADMINISTRATIVE CONTROLS

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5.0 ADMINISTRATIVE CONTROLS

5.1 CONTRACTOR RESPONSIBILITY

The NNFD shall be responsible for overall safe operation and maintenance of the Irradiated Fuels Examination Laboratory (IFEL) Facility. The IFEL facility manager and/or his written designee shall be directly responsible for safe operation within the facility. Safe operation shall include, as necessary, review and approval of material transfers to/from the IFEL Facility, daily operation of the facility, and interface requirements with other organizations and facilities to ensure availability of essential services.

5.2 CONTRACTOR ORGANIZATION

Lines of authority, responsibility, and communication shall be established and defined for the highest management levels, through intermediate levels, to and including line management and supervisory positions involved in IFEL daily operations. These relationships shall be documented and updated, as appropriate, in the form of organizational charts, job descriptions and responsibilities for key personnel positions, or in equivalent forms of documentation.

5.3 PROCEDURES

Procedures shall be established, implemented, and maintained to address the following items:

- (1) Management and control of the inventory of radioactive and fissionable materials to ensure that limitations and assumptions established in the Safety Analysis Report (SAR) are not exceeded;
- (2) Management and control of maintenance, repair, and modification work activities;
- (3) Periodic in-service INSPECTION of selected elements of the safety-class and safety-significant design features for safety to ensure that the safety functions specified in Chapter 4 are maintained; and
- (4) The programs identified in Section 5.4.

The procedures for the above items and changes to these procedures shall be approved by appropriate management levels in accordance with an approved documented procedure review, approval, and revision control process.

5.4 PROGRAMS

5.4.1 Nuclear Criticality Safety

A nuclear criticality safety (NCS) program shall be established, implemented, and maintained. The program shall be a formal, documented system for control of NCS parameters and their bases, identification, and verification that includes the following objectives:

- (1) The NCS of operations involving fissionable materials is addressed and reviewed if there are fissionable nuclide quantities exceeding a threshold quantity;
- (2) The need for NCS controls is identified and the controls are implemented when required, including evaluation of the need to identify NCS controls as safety-significant, safety-class, or Specific Administrative Controls;
- (3) Operations involving fissionable materials are performed by trained personnel as specified in the applicable NCS approval;

- (4) NCS INSPECTIONS/audits are performed; and
- (5) NCS infractions are reported and corrected.

5.4.2 Radiological Protection

A radiological protection program shall be established, implemented, and maintained for control of and protection of personnel from sources of ionizing radiation. The program shall be based on 10 CFR 835, *Occupational Radiation Protection*, and include the following objectives:

- (1) Radiation exposures are maintained at levels that are as low as reasonably achievable;
- (2) Radiation exposure for occupational workers is limited, monitored, and recorded;
- (3) Radioactive materials are identified;
- (4) Entry control programs are established for areas where elevated radiation fields or radioactive surface contamination may be present, and the areas are posted; and
- (5) Periodic radiation safety training is provided.

5.4.3 Public and Environment Radiological Protection

Public and environment radiological protection programs shall be established, implemented, and maintained that includes the following objectives:

- (1) Airborne and liquid releases are monitored in accordance with the applicable regulatory requirements;
- (2) Regulatory limits on routine discharges of radionuclides are met;
- (3) Doses to members of the public are periodically assessed; and
- (4) Protection of the environment from contamination is provided to the degree practical.

5.4.4 Fire Protection

A fire protection program shall be established, implemented, and maintained that includes the following objectives:

- (1) A trained emergency fire fighting and emergency medical response crew is available on a 24-h basis;
- (2) Periodic INSPECTION, maintenance, and testing of fire protection equipment are performed;
- (3) Periodic facility fire safety/prevention INSPECTIONS and fire protection engineering facility assessments are performed; and
- (4) Combustible materials and ignition sources are controlled.

5.4.5 Emergency Preparedness

An emergency preparedness program shall be established, implemented, and maintained. The program shall ensure effective planning for and implementation and control of the response to emergency conditions and include the following objectives:

- (1) Emergency planning, preparedness, response, mitigation, and recovery capabilities are developed and maintained, as well as effective public and interagency communications; and
- (2) Emergencies are responded to in an effective and timely manner to mitigate the consequences and bring the emergency situation under control.

5.4.6 Configuration Management

A configuration management program shall be established, implemented, and maintained that includes the following objectives:

- (1) Safety-class, safety-significant and defense-in-depth structures, systems, and components (SSCs) and the requirements for each safety-class, safety-significant, and defense-in-depth SSC are identified, documented, and maintained;
- (2) Activities and changes are controlled to ensure that safety-class, safety significant and defense-in-depth SSC requirements are met;
- (3) A storage and retrieval system for safety-class, safety-significant, and defense-in-depth SSC documents and records is implemented; and
- (4) A process for maintaining designated aging safety-class, safety-significant, and defense-in-depth SSCs is implemented.

5.4.7 Quality Assurance

A graded quality assurance program shall be established, implemented, and maintained for control of the quality of operations and activities and their results. The program shall be based on 10 CFR 830, Subpart A, *Quality Assurance Requirements*, and include the following objectives:

- (1) Work is performed to established technical standards and administrative controls;
- (2) Design changes are controlled commensurate with those controls applied to the original design;
- (3) Personnel are trained and qualified to perform their assigned work;
- (4) Procured items and services must meet established requirements;
- (5) Identified records are maintained; and
- (6) Periodic assessments of the program and its performance are conducted.

5.4.8 Facility Safety Analysis

A facility safety analysis program shall be established, implemented, and maintained for the control of the safety of activities and operations involving hazards which have the potential for significant impact to workers and the public. The program shall ensure that potential hazards are systematically identified; potential impacts and risks are analyzed and evaluated; reasonable measures have been taken to eliminate, control, or mitigate the hazards; and formal management authorization has been received for the conduct of hazardous facility operations or activities. The program shall be based on 10 CFR 830, Subpart B, *Safety Basis Requirements*, and include the following objectives:

- (1) Appropriate safety analysis documentation is prepared, reviewed, approved, implemented, and maintained; and
- (2) New activities, changes, and modifications are evaluated using the Unreviewed Safety Question (USQ) process.

5.4.9 Initial Testing, In-service Surveillance and Maintenance

An initial testing, in-service surveillance and maintenance program shall be established, implemented, and maintained. The program shall ensure the accomplishment of inspections, adjustments, lubrication, reconditioning, and other repetitive maintenance activities designed to reduce failures and increase the reliability of equipment (preventive maintenance) and include the following objectives:

- (1) Safety-class and safety-significant SSCs requiring preventive maintenance are identified;
- (2) Preventive maintenance is planned, scheduled, and accomplished on the identified safety-class and safety-significant SSCs; and
- (3) Post-maintenance testing is performed to ensure that safety-class and safety-significant SSCs are operable following the completion of maintenance activities.

5.4.10 Hoisting and Rigging Program

A Hoisting and Rigging Program applicable to Building 3525 shall be established, implemented and maintained that establishes requirements for safely performing hoisting and rigging activities, including the use of cranes, hoists and forklift trucks. The Hoisting and Rigging Program shall address the following:

- (1) Preparing for hoisting and rigging activities, including preparation of lift plans, as applicable;
- (2) Performing critical lifts, pre-engineered production lifts, and ordinary lifts;
- (3) Required operator qualifications; and
- (4) Inspection and maintenance of hoisting and rigging equipment.

5.4.11 Conduct of Operations Program

A Conduct of Operations (COO) Program applicable to Building 3525 shall be established, implemented and maintained that includes the following objectives:

- (1) Conduct of Operations shall be based on well-developed industrial operations practices;
- (2) Procedures for conformance to the COO requirements for Building 3525 shall define the purpose, scope and requirements for implementation; and
- (3) Matrices shall be prepared that address the conformance of the COO Program applicable to Building 3525 with each chapter of Attachment 2 of DOE Order 422.1, *Conduct of Operations Requirements*.

5.5 MINIMUM OPERATIONS SHIFT COMPLEMENT

Other than evacuation for personal protection, the hazards and accident analysis in Chapter 3 did not take credit for any personnel actions to prevent, detect, or mitigate an accident. Per DOE G 423.1-1, "The required staffing of operation shifts for nonreactor nuclear facilities and the members of the shift staff required to be present in the control room or control area for different operating conditions should be specified in the AC section on the basis of relevant safety analyses." Because the safety analyses did not identify any required operator actions to be taken, there are no minimum staff requirements for IFEL.

5.6 OPERATING SUPPORT

Support personnel requirements for facility operations shall be designated in the instructions, work package, or procedure, when required for the specific operation. Support personnel or organizations for IFEL associated with facility management, technical support, and radiation protection shall be at the facility or designated for contact.

5.7 FACILITY STAFF QUALIFICATIONS AND TRAINING

The IFEL operator/supervisor minimum qualifications shall be in accordance with Attachment 1, "Contractor Requirements Document," Chapter II, "Hazard Category 1, 2, and 3 Nuclear Facility Personnel," Section 2 Table 1, "Hazard Category 2 and 3 Nonreactor Nuclear Facility Personnel Education and Experience Requirements," and Section 6, "Non-Reactor Nuclear Facility Specific Requirements," paragraphs a and c of DOE Order 426.2, "Personnel Selection, Training, Qualification, and Certification Requirements for DOE Nuclear Facilities," (as amended). A retraining and replacement training program for IFEL operators/supervisors shall be maintained under the direction of the training department of the operating organization(s) responsible for IFEL and shall meet the requirements of Attachment 1, "Contractor Requirements Document," Chapter I, "General Requirements," Section 8, "Requalification," of DOE Order 426.2 and, for appropriate designated positions, shall include familiarization with relevant industry operational experience.

5.8 RECORD KEEPING

An ORNL Records Management system is in place that establishes requirements for the identification, distribution, maintenance, storage, retrieval, inventory, and disposition of ORNL records. Facility management will interface with the ORNL program office to ensure appropriate implementation.

In addition to the requirements of applicable regulations, and in no way substituting therefore, records and logs shall be prepared and retained for the IFEL, Building 3525, for at least the following items:

- (1) Facility operating logs
- (2) Principal maintenance activities
- (3) Training records
- (4) Unreviewed Safety Question Determinations, and
- (5) Occurrence reports applicable to the IFEL facility.

5.9 REVIEWS AND AUDITS

Unreviewed safety question determinations, Technical Safety Requirement (TSR) changes, facility safety basis documentation changes, proposed tests and experiments, and facility changes and modifications shall be reviewed by appropriate individuals of the IFEL operating organization.

TSR changes and facility safety basis documentation changes shall be reviewed by individuals of a facility safety committee, group, or organization that is independent of the IFEL operating organization.

Management assessments or readiness reviews shall be performed as applicable for new facility operations or facility modifications.

A self-assessment program applicable to the IFEL shall be established, implemented, and maintained that includes line management assessments, independent assessments, issue management and reporting, and

employee involvement and improvement.

Independent audits, surveillances, and other assessments to measure item and service quality, determine adequacy of work performance, and promote improvement shall be performed periodically.

In addition to the surveillances required by the TSRs, safety system assessments of active safety-class, safety-significant, and defense-in-depth systems shall be performed periodically to ensure they are capable of performing their safety/design function.

5.10 DEVIATIONS FROM TSRs

IFEL personnel may take action that departs from the requirements of this TSR provided (a) an emergency situation exists; (b) these actions are needed immediately to protect workers, the public, or the environment from imminent and significant harm; and (c) no action consistent with the TSR is immediately apparent. Such action must be approved by the facility manager or his designee. If emergency action is taken, both a verbal notification should be made to the DOE Site Office and a written report made to the Cognizant Secretarial Officer (CSO) within 24 h. Reporting of all TSR VIOLATIONS or deviations from the TSR should be made in accordance with the provisions of DOE O 231.1A, *Environment, Safety and Health Reporting* (or successor).

5.11 SPECIFIC ADMINISTRATIVE CONTROLS

5.11.1 Radioactive Material Limit

5.11.1.1 Critical Safety Function

The radioactive material limit is an initial underlying assumption for the hazard evaluation and accident analysis performed in Chapter 3 of the SAR, SBS/3525/2010-02, SBS/3525/2012-01, and SBS/3525/2013-01. The limit protects this assumption and ensures that facility operations are not conducted in formally unanalyzed space.

5.11.1.2 SAC Description

The inventory of radioactive materials within Building 3525 shall be maintained below the limits in SBS/3525/2013-01 Table 3-5(3a-²³⁸Pu-GB-⁹⁹Mo) *Hazardous Material Listing For SAR Rev 3a*, SBS/3525/2010-02, SBS/3525/2012-01 and SBS/3525/2013-01. The SBS Table 3-5(3a-²³⁸Pu-GB-⁹⁹Mo) provides the same radioactive material limits as those in SAR Rev 3a Table 3-5 with the addition of the cermet targets/alloy fuels, SNF with radioactive iodine, gaseous radioactive materials limits, glove box limits, irradiated uranium metals, and unirradiated uranium metals determined by the three SBSs.

5.11.1.3 Basis

The hazard evaluation and accident analysis for Building 3525 did not evaluate accidents that involve radioactive materials in amounts greater than those listed in Table 3-5(3a-²³⁸Pu-GB-⁹⁹Mo) of SBS/3525/2012-01. The radioactive material limits in Table 3-5(3a-²³⁸Pu-GB-⁹⁹Mo) are based on the type/form of the radioactive materials and, in some cases, on the work activities that are to be conducted when the radioactive materials are present. As noted in Table 3-5(3a-²³⁸Pu-GB-⁹⁹Mo), the radioactive material limits in the hot cells are decreased when (1) materials are being used that create a credible explosive atmosphere in the hot cells, and (2) heavy loads capable of breaching the hot cells if dropped are being handled above the hot cells. This SAC is needed for worker safety because the hazard evaluation and accident analysis did not evaluate any accidents that involve quantities of radioactive materials in excess of the limits in Table 3-5(3a-²³⁸Pu-GB-⁹⁹Mo). Therefore, this limit is required to ensure that the bounding consequences are not exceeded as analyzed in the SAR, SBS/3525/2010-02, SBS/3525/2012-01, and SBS/3525/2013-01.

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SECTION 6
DESIGN FEATURES FOR SAFETY

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6.0 DESIGN FEATURES

Design features for safety are systems, structures, and components that are credited in the accident analyses. They are normally passive characteristics of the facility not subject to change by operations personnel (e.g., shielding, structural walls, relative locations of major components, installed poisons, or special materials). Design features are those permanently built-in features critical to safety that do not require, or infrequently require, maintenance or surveillance. The shielded structures and transport carriers provide significant shielding protection to facility personnel from external radiation exposure during normal operations and accidents and, therefore, are considered safety-significant design features. The main hot cells remain intact and provide limited confinement of airborne radioactive materials following accidents to protect on-site personnel. The confinement function is considered a safety-significant design feature. The integrity of the design features is periodically checked through routine radiological surveys and/or scheduled in-service inspections.

6.1 MAIN HOT CELL STRUCTURE

The hot cell structure (defined as the cell walls, floor, and ceiling along with the viewing windows, doors, and penetrations) is centrally located. The cells, which are designed to handle a variety of radioactive materials, are in the shape of a "U." The cell structure is divided into three separate cells that are known as the north cell, east cell, and west cell. The face of the north cell is 53 ft, and the face of the east and west cells are 43 ft. Each cell has an exterior depth of 16.5 ft. The cell walls are constructed of barytes concrete and are 3 ft. thick. Each cell is 15 ft. from cell floor to ceiling. The cells are separated by permanent 18-in-thick concrete walls with an approximately 18-in-wide and 18-in-high opening that may be closed for isolation. Each cell is further subdivided into work stations. The work stations are served by oil-filled, lead-glass viewing windows and a pair of master-slave manipulators located at each window. The inside surfaces of the cell are lined with stainless steel to provide confinement of particulate matter and aid in cell decontamination. Penetrations provide for stereomicroscopes and periscopes and the sealed entry of services such as instrument lines, lights, water, and gas.

Heavy objects within each cell are moved by electromechanical manipulators and a 3-ton crane. Radioactive materials primarily enter and leave the cells through three shielded transfer stations provided at the rear face of the north cell. Two small-diameter horizontal transfer stations are used for small objects, while larger items are transferred through the shielded air-lock door system. A 10-in. shielded port on the south wall of the east hot cell allows for introduction of materials directly into the east hot cell. Additional shielded ports are in each cell for introduction and removal of supplies or low-activity radioactive materials not posing a personnel dose problem.

6.2 CCCTF CELL

The Core Conduction Cooldown Test Facility (CCCTF) cell is constructed of 4-in-thick chevron interlocking lead bricks and sheathed in steel plate and is 61 in. wide by 84 in. deep by 104 in. high. Two lead-glass viewing windows are located on the south end of the cell along with a set of manipulators. Materials are loaded into the CCCTF hot cell via a pass-through port located on the east end of the cell or the main cell door on the west end.

6.3 IMGA CELL

The Irradiated Microsphere Gamma Analyzer (IMGA) shielded cell is constructed of two layers of 4-in. steel plates. The external dimensions of the cell are 80 in. wide by 60 in. deep by 60 in. high. On the front of the cubicle is a shield door with a 3-ft by 2-ft by 10-in.-thick lead glass window. The cell is lined with 3/16-in.-thick stainless steel plate.

6.4 SEM CELL

The Scanning Electron Microscope/Microprobe (SEM) sample preparation cell is located on the second floor between room 224 and the hot equipment storage area (HESA). It is essentially a sealed stainless steel box with double 4-in.-thick carbon steel shielding plates on the front, back, right side, and bottom constructed similar to the IMGA cell. The left side of the cell is shielded by the 2-ft-thick concrete wall of the HESA, and the top is shielded by a single 4-in. thick carbon steel plate and various amounts of stacked lead bricks. The cell is equipped with a pair of master-slave manipulators, and the door/window is composed of 10-in.-thick leaded glass.

6.5 HESA AND DECONTAMINATION AREA

The decontamination area and the HESA are shielded by 2-foot-thick walls constructed of regular concrete. There are two shield doors, one between the decontamination area and the HESA and one between the decontamination area and the glove maintenance room (GMR). There are three removable shield plugs in the floor between first and second floor in the decontamination area that open to work station 9 in the east cell. There are three removable shield plugs in the north end of the HESA that open to work station 5 of the north cell and there are three removable shield plugs in the middle of the HESA that open to work station 4 of the west cell. If the shield plugs are removed, the HESA and decontamination cells are considered to be part of the main hot cells for application of Technical Safety Requirements (TSRs).

6.6 SHIELDED METALLOGRAPH AND SHIELDED HARDNESS TESTER

The shielded remote control metallograph is installed in Room 122 adjacent to the West Hot Cell. The mechanical stage, microscope, and associated components are contained in a shielded box constructed of 8-in.-thick steel plate. A small, lead glass window is provided in the steel cover plate for access viewing. The shielded hardness tester is installed on the mezzanine in Room 122 adjacent to the West Hot Cell. The hardness tester is contained in a shielded box constructed of 8-in.-thick steel plate. Two small, lead glass windows are provided for access viewing.

6.7 CHARGING AREA STORAGE WELLS

The floor of the charging area is concrete covered with stainless steel for ease of decontamination and contains 24 storage wells for temporary storage of sources and radioactive materials. These wells are arranged in four rows of six wells, two rows on the east side and two on the west side of the charging area. Six of the wells (three on each side at the south end of the matrix) are 8 in. in diameter; the remainder are all 6-in.-diameter wells. All 24 wells are nominally 7 ft deep and are covered with a 3-ft gasketed cast-iron shielding plug.

6.8 SHIELDED TRANSPORT CARRIERS

Radioactive materials are transported into and out of Building 3525 via an assorted number of shielded transport carriers, depending on the size, shape and radioactivity of the material being transported. Requirements for these transport carriers are specified in separate documentation for the transportation of hazardous materials. The requirements established by these documents along with radiation protection requirements establish the necessary shielding required to protect workers from potentially significant direct radiation exposure.

6.9 OXIDATION FURNACE GAS SUPPLY LINE FLOW ORIFICES

Cover gases (e.g., nitrogen, oxygen, ozone) are supplied to the oxidation furnace in the north hot cell through a small (e.g., 1/2-in.) supply line. The gases may be supplied from standard gas cylinders/generators in the operating area or the plant air system. When pressurized gases are used to supply the cover gases, two flow orifices as described in SAR Section 4.4.6 are installed in the supply line to limit gas flow to the furnace and the downstream HEPA filter. The gas flow must be limited to within the design capacity of the HEPA filter to ensure that the air is filtered prior to discharge outside the hot cells.

6.10 ROLL-AROUND SHIELD

The roll-around shield is a moveable shield that can be used to move radioactive materials between compatible top loading casks and the north hot cell 6.5-in or 14.5-in transfer ports. The roll-around shield has five of its six sides shielded with either 5.5 or 6.5 inches of lead that is clad in 0.5 inches of steel on the inside and outside. The sixth side is a steel plate with a circular opening that matches the hot cell transfer port opening. The bottom of the shield has an approximately 12-in. opening that interfaces with the top of the top-loading cask. The face away from the hot cell is sloped and has a shielded viewing window and simple manipulator that can be used to move material between the cask and the hot cell transfer port. The manipulator penetrates the front shield through a rotating lead ball in a socket.

6.11 Rooms 127, 130, 208, and 209

Rooms 127, 130, 208 and 209 must maintain their structural integrity during PC-2 NPH earthquake or high wind events so that any water that may be released in the area of the stored fuel assemblies can drain to the main second floor level. No piping systems are credited as seismically qualified but the rooms must retain their ability to maintain the location of the fuel assemblies and to drain away any water that may be released into the rooms. The bottom of the double doors between Room 208 and Room 209 has been replaced with expanded metal with openings (i.e., similar to grating) to allow any water released into Room 209 to drain to Room 208 and then to the main second floor elevation.

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APPENDIX A
TECHNICAL SAFETY REQUIREMENT BASES

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Bases 3/4-2

3/4.0 GENERAL APPLICATION

BASES

GENERAL APPLICATION LCOs AND SRs

LCOs 3.0.1 through 3.0.6 and SRs 4.0.1 through 4.0.4 establish the general requirements applicable to all LCOs and SRs at all times, unless otherwise stated. The general requirements have two principal functions:

- (1) To provide the basic application rules for use of the LCOs and SRs; and
- (2) To ensure that there is always a corrective action for all LCO statements that are not met.

Since the general application LCOs and SRs are standard requirements, except for a few facility-specific changes in LCO 3.0.3 involving facility process area and MODE names, no justification is given in this Bases section for the individual requirements. This Bases section only provides clarification and additional guidance for the use of the general requirements.

LCO 3.0.1

LCO 3.0.1 establishes the requirement that an LCO's statement (the requirements or specifications) be met during the MODES or other specified conditions in the LCO's Applicability statement, except when undertaking and operating in the LCO's ACTION statement(s). The LCO's ACTION statements are the measures that must be taken with specified completion times when the LCO statement is not being met. The LCO is considered to be met (not violated) as long as the LCO's ACTION statements are undertaken and completed within their specified completion times.

LCO 3.0.2

LCO 3.0.2 establishes the requirement that when it is discovered (found out) that an LCO statement is not being met, the associated ACTION statement(s) for the applicable condition(s) specified in the LCO's ACTION section must be taken. If none of the conditions specified in the LCO's ACTION section are applicable to the problem, the ACTION statements in LCO 3.0.3 must be taken. The completion time for an ACTION statement(s) starts at the time it is discovered that the degraded condition exists and the LCO statement is not being met. The term discovery is used because in many cases it may not be found out that an LCO statement is not being met until a SR is performed or until a structure, system, or component fails to work. The actual determination of whether the LCO statement is or is not being met must be made as soon as possible upon discovery of a problem. Time is allowed to perform a good-faith effort to determine if the LCO statement is being met; however, an unreasonable postponement in making the met/not met decision after the discovery of a problem is unacceptable. If the LCO is restored while undertaking an ACTION statement, the ACTION statement in progress does not have to be completed, unless otherwise stated in the ACTION statement. Restored means the equipment addressed by the LCO is returned to an OPERABLE condition meeting the requirements of the LCO statement and having passed the applicable SRs. If the LCO is no longer applicable (i.e., the facility has been placed in a MODE in which the LCO statement is not applicable), any ACTION(s) in progress also does not have to be completed.

There are three types of basic ACTION statements specified in LCOs. The first type of ACTION statement specifies a completion time to restore inoperable equipment to an OPERABLE condition (restoring compliance with the LCO statement).

(LCO 3.0.2 continued on next page)

3/4.0 GENERAL APPLICATION

BASES

LCO 3.0.2 (continued)

Whether stated as a specific ACTION or not, restoration of inoperable equipment to an OPERABLE condition in compliance with the LCO statement may always be considered on entering any other type of ACTION statement. The second type of ACTION statement specifies a completion time for placing the facility in a MODE in which the LCO is not applicable. The third type of ACTION statement specifies remedial or compensatory measures that are taken to permit continued operation of the facility with the inoperable equipment, not further restricted by the completion time. Compliance with the measures provides an acceptable level of safety for continued operation.

LCO 3.0.3

LCO 3.0.3 establishes the requirements for the ACTIONs to take when an LCO's statement is not met and

- (1) an LCO's associated ACTION statement(s) is not met (completed) within its completion time (unless otherwise specified in the LCO's ACTION statements) or
- (2) there are no ACTION statements in the LCO's ACTION section applicable to the specific problem (condition not specifically addressed).

When an LCO statement is not met AND the associated ACTION statement(s) cannot be met (completed) or if there is no ACTION statement provided that is applicable to the problem, the affected facility area(s) must be placed in a MODE or other specified condition in which the LCO is not applicable. If the LCO is applicable in all MODES, then the affected facility area(s) must be placed in the safest MODE. For Building 3525 the safest MODE (without removing all of the radioactive materials from an area or the facility) is the WARM STANDBY MODE. Activities must be initiated within 1 h to place the affected facility area(s) in a condition in which the LCO is not applicable or in the WARM STANDBY MODE. The affected facility area(s) must be in a condition in which the LCO is not applicable or in the WARM STANDBY MODE within 12 h. The completion time of 1 h to start the shutdown process; which allows time to assess the condition, correct very minor problems, and confirm the restoration; is acceptable because of the low probability of an accident in this short period of time and a safety system not being OPERABLE will not impact the probability of an accident. The completion time of 12 h, which allows sufficient time for the affected facility area(s) to be placed in the WARM STANDBY MODE in a controlled and orderly manner, is also acceptable because of the low probability of an accident in this short period of time and a safety system not being OPERABLE will not impact the probability of an accident.

As with LCO 3.0.2, if the LCO is restored while undertaking the actions to make the MODE change (or change to the condition in which the LCO is not applicable), the change to the WARM STANDBY MODE (or change to the condition in which the LCO is not applicable) does not have to be completed.

Exceptions to LCO 3.0.3 can be stated in the individual LCOs. Justification for the exception must be placed in the Bases for the individual LCO. Exceptions are provided in instances where requiring a MODE change of the affected facility area(s) would not provide the appropriate remedial measures for the associated condition of the inoperable equipment.

3/4.0 GENERAL APPLICATION

BASES

LCO 3.0.4

LCO 3.0.4 establishes the requirements for limitations on changes in MODES or other specified conditions in the LCO Applicability statements. For a facility area(s) to change to a new MODE, all of the LCOs required for that MODE must be met without reliance on their ACTION statements. This means that no known problem that will require or has required entry into an ACTION statement exists with any of the equipment required by the LCOs for that MODE. An exception is made if the LCO ACTION statement(s) for the applicable degraded equipment condition allows the affected facility area(s) to stay in the MODE for an unlimited time. This case is generally when the required ACTION statements are remedial or compensatory measures. An additional exception is made if entry into the MODE is required to comply with an LCO's ACTION statement(s). Additional exceptions to LCO 3.0.4 can be made in individual LCOs. In general these exceptions are made for equipment that does not significantly impact facility safety if it is inoperable for a limited time or the equipment is not required for every phase of a given MODE.

LCO 3.0.5

LCO 3.0.5 establishes the requirement allowing equipment to be returned to service under administrative control only to perform testing to demonstrate its OPERABILITY or the OPERABILITY of other equipment, after it has been removed from service or declared inoperable to comply with the ACTION statements of an LCO. The administrative control is to ensure that the length of time the equipment is returned to service is limited to the maximum time absolutely necessary to perform the allowed SR. This LCO does not provide an exception to perform any other preventive or corrective maintenance. LCO 3.0.2 requires that an LCO's ACTION statement(s) be taken when the LCO statement is not met. LCO 3.0.5 is an exception to LCO 3.0.2 when the ACTION statement(s) required that the equipment be taken out of service. Placing equipment back into service under this LCO does not mean that the equipment is OPERABLE. OPERABILITY of the equipment is only determined by successfully completing all applicable SRs and any other tests as required.

LCO 3.0.6

LCO 3.0.6 establishes an exception to the requirements of LCO 3.0.2 for a supported system whose support system(s) has an individual LCO. When a support system is inoperable, the system that is supported is also inoperable. If the support system has an LCO, then only the support system's ACTION statements must be taken. Implicitly understood here is that the ACTION statements for the support system's LCO provide all of the requirements needed to address the inoperability of the supported system as well as the support system. Surveillance testing is not required to demonstrate that a supported system is OPERABLE on the return to service of the support system. However, LCO 3.0.6 is not to be interpreted to say that the ACTION statements of the supported system's LCO can be ignored; they are still fully active for other concurrent problems.

3/4.0 GENERAL APPLICATION

BASES

SR 4.0.1

SR 4.0.1 establishes the requirement that SRs must be performed within their required frequency intervals (the specified frequency plus the 25% extension of SR 4.0.2) during the MODES or other specified conditions in the LCO's Applicability statement, unless otherwise specified in the individual SR. When a surveillance is performed and the results of the surveillance indicate that the equipment is inoperable, the LCO statement is not met and the applicable ACTION statements must be entered. This applies to anytime a surveillance is performed, whether to meet the required frequency interval or as part of a special test. If a surveillance is not performed within the required frequency interval, the equipment must be considered inoperable and the LCO statement not met, and the applicable ACTION statements entered, except as provided by SR 4.0.3. A delay period of up to 24 h is allowed under the provisions of SR 4.0.3 to perform the surveillance and avoid having to declare the equipment inoperable and entering the applicable ACTION statements. Any missed surveillance is a violation of the TSR, even if the surveillance completed under the provisions of SR 4.0.3 indicates that the equipment was OPERABLE.

SR 4.0.2

SR 4.0.2 establishes the requirements for meeting the surveillance frequency intervals specified in the individual SRs and ACTION statements. An extension of the specified frequency interval by 25% is permitted to facilitate scheduling of surveillances and to allow for the surveillance to be performed at the best time for operation of the facility. The 25% extension does not apply to one-time or single-event surveillances or the initial performance of a periodic surveillance. Exceptions to SR4.0.2 maybe stated in individual SRs. The 25% extension is not intended to be used routinely to extend the specified frequency interval.

SR 4.0.3

SR4.0.3 establishes the flexibility to defer declaring affected equipment inoperable when a surveillance has not been performed within the required frequency interval (the specified frequency interval plus the 25% extension). Upon discovery that a surveillance has not been performed within its required frequency interval, a delay period of up to 24 h is allowed to perform the surveillance and avoid declaring the equipment inoperable and entering the LCO ACTION statements. If the surveillance is not completed within the delay period, the equipment must be considered inoperable and the LCO ACTION statements must be entered. The completion time of the ACTION statements begins IMMEDIATELY on the expiration of the delay period. As with any surveillance, if a surveillance performed during the delay period does not meet its specifications, the equipment must be declared inoperable and the LCO ACTION statements entered. Successful performance of the surveillance during the delay period does not remove the TSR violation associated with not performing the surveillance within its required frequency interval.

SR 4.0.4

SR 4.0.4 establishes the requirement that all of the SRs for all LCOs required for a MODE must have been successfully performed within their required frequency (specified frequency plus the 25% extension) before the MODE is entered. When going from a MODE that does not require a particular surveillance to a MODE that does, the surveillance must be completed within its specified frequency without reliance on the 25% extension. Entry into a MODE cannot be made with a missed surveillance currently being performed under the provisions of SR 4.0.3. An exception is made for entry into MODES required to comply with ACTION statements.

3/4.1 VENTILATION/FILTRATION SYSTEMS

3/4.1.1 HCV System

BASES

BACKGROUND	<p>The HCV System provides the primary containment of the high-activity radioactive material particulates generated during examination and processing activities. The negative pressure provided to the HCV System is provided by either the 3039 stack or facility exhaust (booster) fans and a local building stack. The 3039 stack, which has backup capability, is not only shared with other facilities at the Oak Ridge National Laboratory (ORNL) but is operated by another contractor. The facility exhaust (booster) fans and local stack are available in the unlikely event the 3039 stack is inoperable. Regardless of which system provides the negative pressure, the negative pressure requirement in the cells is the important attribute of the HCV System. The LCO OPERABILITY statement will require that the 3039 stack is providing sufficient negative pressure. The exhaust that flows from the cells is filtered in the cell, controlled by a valve, and filtered again before it passes through the booster fans to the stack. The booster fans do not run under normal conditions. The filtration prior to the fans is through one or two parallel HEPA filter banks.</p> <p>The North, East, and West cells have dedicated in-cell filters and control valves. After passage through the control valves, the exhaust joins into a common exhaust header. Also joining this header are ducts from the Glove Maintenance Room and the Hood Exhaust System. Connected to the Glove Maintenance Room exhaust is the Core Conduction Cool-down Test Facility Cell with its dedicated in-line filter. The in-cell filters and in-line filters are not considered to be safety significant and are not subject to TSR SURVEILLANCE REQUIREMENTS.</p>
APPLICATION TO SAFETY ANALYSIS	<p>This LCO is applied to ensure that the HCV System is OPERABLE. The safety function of the HCV System, if available, is to provide enhanced mitigation of the effects of an uncontrolled release involving a SIGNIFICANT QUANTITY OF RADIOACTIVE MATERIAL due to accidents that may make radioactive materials in the hot cells airborne where they could leak to the outside environment. The HCV System performs this function by ensuring that air flow is into the hot cells and filtering particulates passing through the ventilation system prior to their discharge outside the IFEL.</p>
LCO	<p>The HCV System consists of the cell differential pressure provided by the 3039 or local stack, a cell filter, and parallel HEPA filter banks. The system is determined to be OPERABLE when the required negative pressure is obtained in the cells, the negative pressure is provided by the 3039 stack, and each ventilation flow path has filtration which has successfully passed an in-place leak test. The basis for the safety goal of 0.3 in. of water differential pressure across the hot cell walls is engineering judgment. The safety goal is achieved by ensuring that the hot cells actual pressures are at least 0.6 in. of water less than the operating area pressure and by ensuring that the charging area actual pressure is no more than 0.3 in. of water below that of the operating area pressure. The differential pressure control excludes the wall between the east hot cell and the GMR as discussed in SAR Section 4.4.3.4. The 3039 stack vacuum supply is achieved by ensuring that the actual supply header duct is at least 3.8 in. of water less than atmospheric pressure</p>

Bases 3/4-7

3/4.1 VENTILATION/FILTRATION SYSTEMS

3/4.1.1 HCV System (continued)

BASES (continued)

	and by ensuring dampers K-15-V-10 and K-15-V-11 are positioned to align the vacuum supply from the 3039 stack header. An alarm senses the 3039 stack pressure and alerts the operators if the 3039 stack header pressure increases above 3.8 in. of water less than atmospheric pressure. Each of the online parallel HEPA filter banks (Filter Bank #1 and Filter Bank #2) is required to have at least one OPERABLE HEPA filter in place to ensure that any exhaust flow is filtered prior to discharge from the IFEL. A filter bank is considered to be online unless the exhaust flow through the filter bank is isolated by a blank flange installed in the inlet or discharge line to block flow through the filter bank. Therefore, one of the filters in the Filter Bank #1 (K-15.12-1 or K-15.12-2) and one of the filters in the Filter Bank #2 (K-15.12-3 or K-15.12-4) are required to be OPERABLE if that bank is online. The basis for one OPERABLE HEPA filter in each online filter bank is to provide filtration of the exhaust from the cells. Some documentation may identify Filter Bank #1 as the North Primary Filters and Filter Bank #2 as the South Primary Filters.
MODE	This LCO is applicable during the OPERATION MODE. In-cell operations with
APPLICABILITY	SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIALS are only performed during the OPERATION MODE.
PROCESS AREA	The LCO is applicable to the North, East, and West Hot cells.
APPLICABILITY	
ACTION	
STATEMENTS	
A.1	If the required differential pressure in a hot cell is not at least 0.6 in. of water below the operating area, actions shall be taken to restore the required differential pressure within 4 h. The completion time is based on allowing sufficient time to correct minor problems and perform verification of required differential pressure, the low probability of an accident occurring during this interval, and continued workplace monitoring in accordance with the Radiological Protection Program, a credited TSR control.
B.1	If unable to restore the required pressure differential within 4 h (ACTION A.1), operations in the hot cells are to be terminated and the cells placed in the WARM STANDBY MODE. This is based on there being no OPERABLE ventilation confinement for the hot cells. The completion times are based on the maximum time required to safely terminate operations inside the hot cells, the low probability of an accident occurring during this interval, and continued workplace monitoring in accordance with the Radiological Protection Program, a credited TSR control. Placing the process area in WARM STANDBY reduces the probability that an accident will occur that affects a SIGNIFICANT QUANTITY OF RADIOACTIVE MATERIAL by terminating those active operations.

Bases 3/4-8

3/4.1 VENTILATION/FILTRATION SYSTEMS

3/4.1.1 HCV System (continued)

BASES (continued)

C.1	The completion time of 4 h for restoring at least one HEPA filter in an online HEPA filter bank to an OPERABLE condition is based on allowing sufficient time to correct minor problems and perform retesting of a HEPA filter, the low probability of an accident occurring during this interval, and continued workplace monitoring in accordance with the Radiological Protection Program, a credited TSR control. In most cases the HEPA filter bank would likely still be providing a high degree of filtration. The in-cell roughing and HEPA filters also provide exhaust filtration.
D.1	If unable to restore at least one HEPA filter in the online HEPA filter bank to OPERABLE condition within 4 h, operations in the hot cells are to be terminated and the cells placed in the WARM STANDBY MODE. This is based on there being an inoperable HEPA filter bank in the exhaust flow path from the hot cells. The completion times are based on the maximum time required to safely terminate operations inside the hot cells, the low probability of an accident occurring during this interval, and continued workplace monitoring in accordance with the Radiological Protection Program, a credited TSR control. Placing the process area in WARM STANDBY reduces the probability that an accident will occur that affects a SIGNIFICANT QUANTITY OF RADIOACTIVE MATERIAL by terminating those active operations.
E.1	If the differential pressure in the charging area is excessive, actions shall be taken to reduce the required differential pressure within 4 h. The charging area ventilation system has system transients from activities such as closing a charging area door that cause the charging area ventilation system to momentarily lower the charging area pressure to more than 0.3 in. of water below the operating area. These are very short duration transients and do not require entry into this ACTION. The completion time is based on allowing sufficient time to correct minor problems and perform verification of the differential pressure, the low probability of an accident occurring during this interval, and continued workplace monitoring in accordance with the Radiological Protection Program, a credited TSR control.

3/4.1 VENTILATION/FILTRATION SYSTEMS

3/4.1.1 HCV System (continued)

BASES (continued)

F.1	If unable to restore the required pressure differential within 4 h (ACTION A.1), operations in the hot cells are to be terminated and the cells placed in the WARM STANDBY MODE. This is based on there being no OPERABLE ventilation confinement for the hot cells. The completion times are based on the maximum time required to safely terminate operations inside the hot cells, the low probability of an accident occurring during this interval, and continued workplace monitoring in accordance with the Radiological Protection Program, a credited TSR control. Placing the process area in WARM STANDBY reduces the probability that an accident will occur that affects a SIGNIFICANT QUANTITY OF RADIOACTIVE MATERIAL by terminating those active operations.
G.1	If the required 3039 stack vacuum header is not at least 3.8 in. of water below atmospheric pressure or the dampers are not in the correct positions, actions shall be taken to restore the required differential pressure and damper positions within 4 h. The completion time is based on allowing sufficient time to correct minor problems and perform verification of required differential pressure and dampers positions, the low probability of an accident occurring during this interval, and continued workplace monitoring in accordance with the Radiological Protection Program, a credited TSR control.
H.1	If unable to restore the required pressure differential or damper positions within 4 h (ACTION G.1), operations in the hot cells are to be terminated and the cells placed in the WARM STANDBY MODE. This is based on there being no OPERABLE ventilation confinement for the hot cells. The completion times are based on the maximum time required to safely terminate operations inside the hot cells, the low probability of an accident occurring during this interval, and continued workplace monitoring in accordance with the Radiological Protection Program, a credited TSR control. Placing the process area in WARM STANDBY reduces the probability that an accident will occur that affects a SIGNIFICANT QUANTITY OF RADIOACTIVE MATERIAL by terminating those active operations.
I.1	If the 3039 stack vacuum header alarm system is INOPERABLE, the SR 4.1.1.4 pressure differential check is to be performed HOURLY instead of DAILY. The increased surveillance frequency is to compensate for the loss of the alarm system that is relied upon to provide immediate indication of the possible degradation or loss of the 3039 stack vacuum header and possible low differential pressure between the hot cells and the operating area. The completion time is based on starting the HOURLY checks as soon as possible after it is discovered that the alarm system is inoperable.
I.2	The PROCESS AREA may also be placed in the STANDBY Mode which does not require the availability of the 3039 stack vacuum.

Bascs 3/4-10

3/4.1 VENTILATION/FILTRATION SYSTEMS

3/4.1.1 HCV System (continued)

BASES (continued)

SURVEILLANCE REQUIREMENTS	4.1.1.1	The pressures in the hot cells shall be verified to be at least 0.6 in. of water lower than the pressure in the Operating Area on a DAILY basis when in the OPERATIONS MODE. This is performed to ensure that the negative pressure is provided as required by the LCO. Loss of hot cell differential pressure does not immediately create an unsafe condition but over time could allow radiological contamination to migrate to the operating areas and indicates that the HCV system may not be able to perform its safety function. Contamination is controlled in accordance with the Radiological Protection Program, a credited TSR control. Operating experience with the HCV system is that it is highly reliable, and a DAILY verification is acceptable to ensure continued reliability. Because the loss of hot cell differential pressure does not immediately create an unsafe condition and the probability of an accident occurring in this interval is low, a DAILY surveillance is acceptable and differential pressure alarms are not required to perform the surveillance. The 0.6 in. of water value represents the actual value and the procedural implementation of this SR must consider the accuracy of the instruments used to monitor the parameter. The procedurally required reading must ensure that the actual parameter is at least 0.6 in of water lower than the pressure in the Operating Area.
	4.1.1.2	The in-place HEPA filter bank leak tests are to be performed ANNUALLY and after maintenance or modification that could affect HEPA filter operation to ensure adequate filtration of the exhaust stream prior to release to the environment. The efficiency requirement of $\geq 99.95\%$ is the ORNL standard for filter in-place efficiency. DOE-HDBK-1169-2003, <i>Nuclear Air Cleaning Handbook</i> , indicates ANNUAL testing and testing after maintenance or modification that could affect HEPA filter operation is acceptable to ensure OPERABILITY.
	4.1.1.3	The pressure in the charging area shall be verified to be no more than 0.3 in. of water lower than the pressure in the operating area on a DAILY basis when in the OPERATIONS MODE. This ensures negative pressure is provided as required by the LCO. Loss of hot cell differential pressure does not immediately create an unsafe condition but over time could allow radiological contamination to migrate to the charging area and indicates that the HCV system may not be able to perform its safety function. Contamination is controlled in accordance with the Radiological Protection Program, a credited TSR control. Operating experience with the HCV system indicates it is highly reliable and a DAILY verification is acceptable to ensure continued reliability. Because the loss of hot cell differential pressure caused by excessive charging area pressure does not immediately create an unsafe condition and the probability of an accident occurring in this interval is low, a DAILY surveillance is acceptable and a differential pressure alarm is not required to perform the surveillance. The 0.3 in. of water value represents the actual value and the procedural implementation of this SR must consider the accuracy of the instruments used to monitor the parameter. The procedurally required reading must ensure that the actual parameter is no more than 0.3 in of water lower than the pressure in the Operating Area.

Bases 3/4-11

3/4.1 VENTILATION/FILTRATION SYSTEMS

3/4.1.1 HCV System (continued)

BASES (continued)

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- 4.1.1.4 The pressures in the 3039 stack vacuum header shall be verified to be at least 3.8 in. of water lower than atmospheric pressure on a DAILY basis when in the OPERATIONS MODE. This is performed to ensure that the negative pressure is provided as required by the LCO. Loss of 3039 stack vacuum header differential pressure does not immediately create an unsafe condition because of the booster fan backup capability but the backup fans may fail and the HCV system may not be able to perform its safety function. Contamination is controlled in accordance with the Radiological Protection Program, a credited TSR control. Operating experience with the HCV system suggests it is highly reliable, and a DAILY verification is acceptable to ensure continued reliability. Because the loss of 3039 stack vacuum header differential pressure does not immediately create an unsafe condition and the probability of an accident occurring in this interval is low, a DAILY surveillance is acceptable and is supplemented by the 3039 stack vacuum header differential pressure alarms which is determined to OPERABLE by SR 4.1.1.5. The 3.8 in. of water value represents the actual value and the procedural implementation of this SR must consider the accuracy of the instruments used to monitor the parameter. The procedurally required reading must ensure that the actual parameter is at least 3.8 in of water lower than the atmospheric pressure.
- 4.1.1.5 The 3039 stack vacuum header pressure alarm is CALIBRATED and FUNCTIONALLY TESTED to ensure that the alarm system functions and annunciates at the required differential pressure. The CALIBRATION and FUNCTIONAL TEST, as a minimum, is performed ANNUALLY, and after replacement and/or maintenance that could affect the alarm OPERABILITY. Operating experience with instrumentation system and pressure instruments at ORNL has shown that annual FUNCTIONAL TESTING and CALIBRATION is acceptable to ensure reliability.
- 4.1.1.6 Dampers K-15-V-10 must be closed and K-15-V-11 must be open to properly align the 3039 stack vacuum header to the HCV system to the hot cells. The required positions for these dampers are the fail-position on loss of control air pressure. The control system consists of pneumatic controllers and pneumatic solenoid valves and does not rely on electrical power for operation. A pneumatic gauge on the Room 131 control board indicates the control air pressure to the damper operators provides indication of the damper position. If the dampers are not properly positioned numerous other system instrumentation such as booster fan operation, exhaust headers flow rates, and hot cell pressures will provided backup indication of the system alignment. The repositioning of the dampers rarely occurs and a DAILY verification of the positions ensures that the dampers are positioned to have the 3039 stack provide the HCV system vacuum to the hot cells.
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3/4.1 VENTILATION/FILTRATION SYSTEMS

3/4.1.2 Oxidation Furnace Off-Gas Line HEPA Filter

BASES

BACKGROUND	Off-gases generated during heating of materials in the oxidation furnace in the North hot cell are collected and routed through a line to traps in the Charging Area. The off-gas line is equipped with a sintered metal HEPA filter inside the hot cell to prevent discharge of radioactive particulates outside the hot cell. The gas-supply-line relief valve and gas-recirculation-line relief valve provide supporting functions to prevent excessive flow rates through the HEPA filter and to prevent over pressurizations of the HEPA filter body.
APPLICATION TO SAFETY ANALYSIS	This LCO is applied to ensure that the oxidation furnace off-gas line HEPA filter, the gas-supply-line relief valve, and the gas-recirculation-valve are OPERABLE. The safety function of the HEPA filter in the oxidation furnace off-gas line is to mitigate the effects of accidents that impact a SIGNIFICANT QUANTITY OF RADIOACTIVE MATERIAL in the oxidation furnace and make the radioactive materials in the furnace airborne where they could be transported through the off-gas line to the outside environment. The HEPA filter performs this function by filtering particulates passing through the off-gas line prior to their discharge outside the hot cells. The pressure relief valves in the gas supply line and gas recirculation line are required to ensure that operating conditions at the HEPA filter remain within design flow rate and pressure for anticipated operational transients.
LCO	The oxidation furnace off-gas-line HEPA filter consists of the sintered metal HEPA filter in the off-gas line. The HEPA filter is determined to be OPERABLE when it is in place, has been demonstrated capable of removing > 99.95 % of a test aerosol, and relief valves are installed in the gas supply-line and gas recirculation-line that have been demonstrated capable of opening at the values specified in SAR Section 4.4.5 and Section 4.4.8.
MODE APPLICABILITY	This LCO is applicable during the OPERATION MODE. In-cell operations with SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIALS are only performed during the OPERATION MODE.
PROCESS AREA APPLICABILITY	The LCO is applicable to the north hot cell with SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIAL in the oxidation furnace. Components related to the confinement of materials in the oxidation furnace are only applicable when the materials are present in the oxidation furnace and it is connected to the off-gas line.
ACTION STATEMENTS	A.1 The completion time of 4 h for restoring the HEPA filter or relief valves to an OPERABLE condition is based on allowing sufficient time to correct minor problems, the low probability of an accident occurring during this interval, and continued workplace monitoring in accordance with the Radiological Protection Program, a credited TSR control. The relief valves ensure that the HEPA filter operates within its design flow rates and pressures. If either relief valve is not OPERABLE, then the HEPA filter is not OPERABLE.

3/4.1 VENTILATION/FILTRATION SYSTEMS

3/4.1.2 Oxidation Furnace Off-Gas Line HEPA Filter (continued)

BASES (continued)

	B.1	If unable to restore the HEPA filter and relief valves to an OPERABLE condition, the furnace is de-energized and the HEPA filter is isolated downstream of the oxidation furnace. This is based on there being no OPERABLE HEPA filter bank in the oxidation furnace off-gas line. The completion times are based on the maximum time required to safely terminate furnace operations, the low probability of an accident occurring during this interval, and continued workplace monitoring in accordance with the Radiological Protection Program, a credited TSR control. De-energizing the furnace and isolating the HEPA filter downstream of the oxidation furnace reduces the probability that an accident will occur that affects a SIGNIFICANT QUANTITY OF RADIOACTIVE MATERIAL by terminating those active operations. The valves identified to isolate the filter are either located inside the hot cell or the first valve located outside the hot cell.
	B.2	If unable to restore the HEPA filter and relief valves to an OPERABLE condition, the oxidation furnace may be disconnected from the off-gas line. The HEPA filter no longer is required to be OPERABLE because a direct release path through the hot cell wall does not exist.
SURVEILLANCE REQUIREMENTS	4.1.2.1	<p>The HEPA filter (HF-102) leak test is to be performed QUARTERLY and after maintenance or modification that could affect HEPA filter operation to ensure adequate filtration of the exhaust stream prior to release to the environment. The efficiency requirement of $\geq 99.95\%$ is the ORNL standard for filter in-place efficiency. DOE-HDBK-1169-2003, <i>Nuclear Air Cleaning Handbook</i>, indicates ANNUAL testing and testing after maintenance or modification that could affect HEPA filter operation is acceptable to ensure OPERABILITY. A QUARTERLY frequency was chosen to provide enhanced testing to ensure that the process conditions do not induce a HEPA failure.</p> <p>Bench tests prior to HEPA filter installation are acceptable to demonstrate OPERABILITY. The sintered metal HEPA filter is a simple, rugged component, and no assembly/disassembly or adjustment is required during installation that would affect OPERABILITY. The HEPA filter is not subject to deformation during normal handling and installation. Therefore, post-installation testing is not required.</p>

Basics 3/4-14

3/4.1 VENTILATION/FILTRATION SYSTEMS

3/4.1.2 Oxidation Furnace Off-Gas Line HEPA Filter (continued)

BASES (continued)

- 4.1.2.2 The gas-supply-line relief-valve (PRV-01) functional test is to be performed ANNUALLY and after maintenance or modification that could affect relief valve operation. Demonstrating that the relief valve opens at ≤ 18 psig in conjunction with the flow elements FE-01 and FE-02 ensures that gas flow rate at the downstream HEPA filter will not exceed HEPA filter design flow conditions for anticipated operational transients. Operating experience indicates that relief valves are highly reliable and ANNUAL testing is sufficient to demonstrate their OPERABILITY.
- Bench tests prior to relief valve installation are acceptable to demonstrate OPERABILITY. The relief valve is a simple, rugged component, and no assembly/disassembly or adjustment is required during installation that would affect OPERABILITY. The relief valve is not subject to deformation during normal handling and installation. Therefore, post-installation testing is not required.
- 4.1.2.3 A visual inspection of the HEPA filter (HF-102) is performed WEEKLY to verify that there is no visible damage that would indicate potential external impact damage to HEPA filter capability. The HEPA filter is held in place with an external cover and an inspection that includes verification of no visible damage to the cover meets the intent of the inspection since the cover encloses the HEPA filter. The HEPA filter is not normally subject to impacts or degradation during normal operations, and a WEEKLY inspection is considered adequate to confirm its integrity given the low probability of damage and the low probability of an accident occurring in the interval between the inspections. Since the HEPA filter is located inside the hot cell and in an area under the 3-ton overhead crane and the remote manipulator, which can lift up to 700 pounds, periodic inspections are necessary to ensure no inadvertent damage has occurred.
- 4.1.2.4 A visual inspection of the gas recirculation line relief valve (PRV-101) is performed WEEKLY to verify that there is no visible damage that would indicate potential damage to relief valve capabilities. The relief valve is not normally subject to impacts or degradation during normal operations, and a WEEKLY inspection is considered adequate to confirm its integrity given the low probability of damage and the low probability of an accident occurring in the interval between the inspections. Since the relief valve is located inside the hot cell and located in an area under the 3-ton overhead crane and the remote manipulator, which can lift up to 700 pounds periodic inspections are necessary to ensure no inadvertent damage has occurred.

3/4.1 VENTILATION/FILTRATION SYSTEMS

3/4.1.2 Oxidation Furnace Off-Gas Line HEPA Filter (continued)

BASES (continued)

4.1.2.5 The gas recirculation line relief valve (PRV-101) functional test is to be performed ANNUALLY and after maintenance or modification that could affect relief valve operation. Demonstrating the relief valve opens at ≤ 15 psig ensures that system pressure at the downstream HEPA filter will not exceed HEPA design conditions for anticipated operational transients. Operating experience indicates that relief valves are highly reliable and ANNUAL testing is sufficient to demonstrate their OPERABILITY.

Bench tests prior to relief valve installation are acceptable to demonstrate OPERABILITY. The relief valve is a simple, rugged component, and no assembly/disassembly or adjustment is required during installation that would affect OPERABILITY. The relief valve is not subject to deformation during normal handling and installation. Therefore, post-installation testing is not required.

3/4.2 CHECK VALVES

3/4.2.1 Oxidation Furnace Gas-Supply-Line and Gas-Recirculation-Line Check Valves

BASES

BACKGROUND	Cover gases (e.g., nitrogen, oxygen, ozone) are supplied to the oxidation furnace in the North hot cell through a small (e.g., 1/2-in.) supply line. The gases may be supplied from standard gas cylinders/generators in the hot cell operating area or the plant air system. The supply line has a check valve installed in it to prevent the backflow of radioactive materials through the supply line. Process gases may be recirculated to the oxidation furnace through a small (e.g., 1/2-in.) recirculation line. The recirculation line has a check valve installed in it to prevent the backflow of radioactive materials through the recirculation line.
APPLICATION TO SAFETY ANALYSIS	This LCO is applied to ensure that the check valves in the oxidation furnace gas supply line and gas recirculation line are OPERABLE. The safety function of the check valves is to mitigate the effects of accidents that impact a SIGNIFICANT QUANTITY OF RADIOACTIVE MATERIAL in the oxidation furnace and make the radioactive materials in the furnace airborne where they could be transported through the supply line or recirculation line to the outside environment. The check valves perform this function by limiting leakage of radioactive particulates through the supply line and recirculation line.
LCO	The check valves in the oxidation furnace gas supply line and gas recirculation line are determined to be OPERABLE when they are in place, capable of closing on reverse flow, and capable of limiting leakage of radioactive materials through the supply line.
MODE APPLICABILITY	This LCO is applicable during the OPERATION MODE. In-cell operations with SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIALS are only performed during the OPERATION MODE.
PROCESS AREA APPLICABILITY	The LCO is applicable to the north hot cell with SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIAL in the oxidation furnace. Components related to the confinement of materials in the oxidation furnace are only applicable when the materials are present in the oxidation furnace and it is connected to the gas supply/recirculation line.
ACTION STATEMENTS	A.1 The completion time of 4 h for restoring the check valve to an OPERABLE condition is based on allowing sufficient time to correct minor problems, the low probability of an accident occurring during this interval, and continued workplace monitoring in accordance with the Radiological Protection Program, a credited TSR control

3/4.2 CHECK VALVES

3/4.2.1 Oxidation Furnace Gas-Supply-Line and Gas-Recirculation-Line Check Valves (continued)

BASES (continued)

	B.1	If unable to restore the check valve to an OPERABLE condition, the oxidation furnace is de-energized and the affected gas supply lines and/or recirculation line are isolated. This is based on there being no OPERABLE check valves in the oxidation furnace gas supply line and/or gas recirculation line. The completion time is based on the maximum time required to safely de-energize the furnace and isolate the affected gas supply lines and/or gas recirculation lines, the low probability of an accident occurring during this interval, and continued workplace monitoring in accordance with the Radiological Protection Program, a credited TSR control. De-energizing the furnace and isolating the affected gas supply lines and/or gas recirculation line reduces the probability that an accident will occur that affects a SIGNIFICANT QUANTITY OF RADIOACTIVE MATERIAL by terminating those active operations. The valves identified to isolate the check valves are either located inside the hot cell or the first valves located outside the hot cell.
	B.2	If unable to restore the check valve to an OPERABLE condition, the oxidation furnace may be disconnected from the gas supply/recirculation line. The check valves are not required to be OPERABLE because a direct release path through the hot cell does not exist.
SURVEILLANCE REQUIREMENTS	4.2.1.1	<p>The check valves in the oxidation furnace gas supply line and gas recirculation line are demonstrated to be OPERABLE by performing an ANNUAL direct pressure bubble test verifying the check valve closes on reverse flow and there is no continuous formation of bubbles. Operating experience indicates that check valves are highly reliable and ANNUAL testing is sufficient to demonstrate their OPERABILITY. Direct pressure bubble testing is considered acceptable per ASME Code, Section V.</p> <p>Bench tests prior to check valve installation are acceptable to demonstrate OPERABILITY. The check valves are simple, rugged components, and no assembly, disassembly, or adjustment is required during installation that would affect OPERABILITY. If a check valve is installed backwards, the system will not operate (normal flow will be obstructed) and the error will be corrected. The check valves are spring actuated and vertical or horizontal orientation will not affect their function to close on reverse flow. In addition, the check valves are only credited with providing limited leakage protection (SAR Section 4.4.7). Therefore, post-installation testing is not required.</p>

3/4.2 CHECK VALVES

3/4.2.1 Oxidation Furnace Gas-Supply-Line and Gas-Recirculation-Line Check Valves (continued)

BASES (continued)

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4.2.1.2	A visual inspection of the check valves is performed WEEKLY to verify that there is no visible damage that would indicate potential damage to check valve capability. The check valves are not normally subject to impacts or degradation during normal operations and a WEEKLY inspection is considered adequate to confirm their integrity given the low probability of damage and the low probability of an accident occurring in the interval between the inspections.
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3/4.3 HOT CELL ROOF PLUGS

3/4.3.1 Hot Cell Roof Plugs

BASES

BACKGROUND	The hot cells are passive design features for safety that provide both a shielding safety function and a confinement safety function as discussed in SAR Section 4.4.1, "Shielded Structures and Transport Carriers," and Section 4.4.2, "Main Hot Cell Confinement Structures." Each of the hot cells contains three roof plugs that can be removed to insert or removal large items into or out of the hot cells. The removal of the three roof plugs creates an opening of approximate 4 ft by 11 ft that opens into either the HESA or Decontamination Cell. Since the HESA and Decontamination Cell are also credited shielded structures and the removal of the roof plugs will result in the radiation shine that hits the roofs of these structures, the removal does not degrade the overall shielding safety function. The removal of the roof plugs does degrade the confinement boundary. SAR Section 4.4.2 discusses the extension of the hot cell confinement boundary into the HESA and Decontamination Cell but these also contain shielding doors (openings of approximately 6 ft by 13 ft) that can be opened for the insertion and removal of large components. The HESA and Decontamination Cell will be used as airlocks for the insertion and removal of large components. Since the hot cell roof plugs may be removed several times a month, an LCO is necessary to ensure that either the roof plugs are installed or a shield door is closed when the hot cells are placed in the OPERATION MODE.
APPLICATION TO SAFETY ANALYSIS	The LCO ensures that the confinement capability of the hot cells is available for all the accidents which credit the confinement capability of the hot cells which are (1) radioactive material dropped, knocked over or hit by falling equipment/debris, (2) pressurized release of radioactive material in hot cell, (3) fire in hot cell, (4) earthquake, and (5) high winds. The HESA and Decontamination Cell are used as airlocks to insert and remove components and materials from the hot cells. The LCO ensures that either the roof plugs are installed or one of the appropriate shield doors is closed to provide the confinement boundary.
LCO	The east hot cell open roof plugs create an opening into the Decontamination Cell so either the east hot cell roof plugs must be installed or the shield door between the Decontamination Cell and the GMR (Room 207) must be closed. The north hot cell and west hot cell roof plugs create an opening into the HESA. If the roof plugs to these hot cells are not installed, then either the shield door between the HESA and Decontamination Cell or the shield door between the Decontamination Cell and the GMR must be closed.
MODE APPLICABILITY	The LCO is applicable during the OPERATIONS MODE. In-cell operations with SIGNIFICANT QUANTITIES OF RADIOACTIVE MATERIALS are only performed during the OPERATIONS MODE.
PROCESS AREA APPLICABILITY	The LCO is applicable to the North, East and West Hot Cell, the HESA, and the Decontamination Cell.

3/4.3 HOT CELL ROOF PLUGS

3/4.3.1 Hot Cell Roof Plugs

BASES (continued)

ACTION STATEMENTS	A.1	<p>If neither the roof plugs are installed nor a shield door closed to establish a confinement boundary, then significant quantities of the airborne radioactive materials released during an accident can escape outside the credited confinement boundary. The airborne radioactive materials will be released to the GMR (Room 207) (see SAR Figure 2-3). The GMR is also ventilated by the HCV system which is the same safety-significant ventilation system that ventilates the hot cells, HESA, and Decontamination Room. The ACTION requires that the differential pressure of the GMR relative to either the GMR airlock or the operating area is confirmed to be less than 0.3 in of WC within 2 hours of discovery. A DAILY SURVEILLANCE REQUIREMENT for LCO 3/4.1.1 for the HCV system confirms that the HCV system is operating when in the OPERATIONS MODE. If the roof plugs are removed and the shield door(s) are open, then the airflow is typically from the GMR into the Decontamination Cell, HESA, and hot cells since they have a lower operating setpoints than the GMR. If the GMR airlock pressure were to be at a lower pressure than the Decontamination Cell, HESA, and hot cells then the airflow would be into the safety-significant HCV system. The GMR has a door between the GMR and GMR airlock (Room 206) that is called the ships door because of its construction style. If the door to the GMR airlock is open, then the airflow from the GMR airlock is into the GMR. Since the airflow is always into the hot cells or HCV system when the roof plugs, shield doors and GMR airlock doors, are open and the HCV system is verified DAILY to be operating when in the OPERATIONS MODE, a period of 2 hours to confirm that the GMR pressures are less than that specified in the hot cell SURVEILLANCE REQUIREMENT, the probability that an accident would occur and the airborne radioactive materials released is extremely low.</p>
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3/4.3 HOT CELL ROOF PLUGS

3/4.3.1 Hot Cell Roof Plugs

BASES (continued)

- A.2 If the confinement boundary cannot be re-established within 2 hours, the ACTION requires that additional measures be taken to protect against a fire event or place the hot cells in the STANDBY MODE. The GMR has also been evaluated to be able to withstand the NPH events that can challenge its integrity (Reference 1). Although not a credited characteristic, the GMR can withstand a PC-2 earthquake without a failure and the building is capable of withstanding PC-2 high winds without a failure. The roof of the GMR is will not fail from roof water loading since its construction does not allow water accumulation. The GMR is capable of withstanding all of the events that the hot cells must withstand except for the fire event. The GMR has a glove port wall and glove box that are not able to withstand a fire event. The ACTIONS ensure that a fire event will not initiate a failure of the GMR glove wall and glove box. The ACTIONS require confirming that (1) the Fire Alarm Control Panel (FACP) has no alarms or inputs bypassed, (2) the sprinklers are operational by completing the monthly fire department inspection with no recorded failures, and (3) the establishment of a fire watch on the building second floor area to provide early detection of fire events. These steps ensure the fire vulnerability of the GMR glove port wall and glove box will not result in a loss of confinement capability from a fire event. The probability that a fire occurs and the fire detection and suppression systems are not operating in 8 hours is extremely low. If the ACTIONS are not completed within the 8 hour period, then the hot cells must be placed in the STANDBY MODE to stop the in-cell operations that could initiate a release of airborne radioactive materials.

Completion of the ACTIONS A.2.1.1, A.2.1.2 and A.2.1.3 allow the facility to remain the OPERATIONS MODE indefinitely since the fire vulnerability of the GMR glove port wall and glove box are prevented from fire damage. LCO 3.0.4 permits the entry into the OPERATION MODE while meeting these ACTIONS since there is no time limit to go to the STANDBY MODE. This possibility is specifically added to permit the repair of the overhead cranes and shield doors if a failure were to occur so that neither the shield doors could be closed nor the roof plugs re-installed within the specified time periods.

3/4.3 HOT CELL ROOF PLUGS

3/4.3.1 Hot Cell Roof Plugs

BASES (continued)

SURVEILLANCE REQUIREMENTS	4.3.1.1	The west hot cell opens into the HESA, so the verification that either the west hot cell roof plugs are installed or a shield door between (1) the Decontamination Cell and the GMR or (2) the HESA and the Decontamination Cell is closed ensures that no large opening exists. The HESA and Decontamination Cell have been credited with a confinement function so the closure of one of the shield doors is sufficient to provide a confinement boundary for the west hot cell. Since the roof plugs and both shield doors must be open before the confinement boundary is compromised and the opening of a shield door or the removal of the roof plugs will be completed by qualified operator who is knowledgeable of the TSR requirements, a DAILY verification of the shield door and roof plugs status provides sufficient assurance that the confinement boundary will be maintained.
	4.3.1.2	The north hot cell opens into the HESA, so the verification that either the north hot cell roof plugs are installed or a shield door between (1) the Decontamination Cell and the GMR or (2) the HESA and the Decontamination Cell is closed ensures that no large opening exists. The HESA and Decontamination Cell have been credited with a confinement function so the closure of one of the shield doors is sufficient to provide a confinement boundary for the north hot cell. Since the roof plugs and both shield doors must be open before the confinement boundary is compromised and the opening of a shield door or the removal of the roof plugs will be completed by qualified operator who is knowledgeable of the TSR requirements, a DAILY verification of the shield doors and roof plugs status provides sufficient assurance that the confinement boundary will be maintained.
	4.3.1.3	The east hot cell opens into the Decontamination Cell, so the verification that either the east hot roof plugs are installed or the shield door between the Decontamination Cell and the GMR is closed ensures that no large opening exists. The Decontamination Cell and HESA have been credited with a confinement function so closure of the shield door between the Decontamination Cell and the GMR also is sufficient to provide a confinement boundary for the hot cell. Since the roof plugs and the shield door between the Decontamination Cell and the GMR must be open before the confinement boundary is compromised and the opening of a shield door or the removal of the roof plugs will be completed by qualified operator who is knowledgeable of the TSR requirements, a DAILY verification of the shield doors and roof plugs status provides sufficient assurance that the confinement boundary will be maintained.
REFERENCES	1	NNFD-3525-DAC-2010-002, 2010 NPH Analysis & SSC Checks, Rev. 1, November 2010.

Bases 3/4-23

RSS No	11716.0															
Status	Inactive															
Minor Revisions	None.															
Last Modified by	Maples, Curtis A (24087) on 10/14/2014 3:40:55 PM															
Notice	This Research Safety Summary is not currently valid , only <i>Authorized</i> Research Safety Summaries are considered to be complete/valid.															
Title	Chemical Processing of Mo-99 Targets															
Approvals/Authorizations	<table><tr><th>Date/Time</th><th>Name</th><th>Role</th></tr><tr><td>11/21/2013 8:09:14 AM</td><td>Busby, Jeremy T (742645)</td><td>Group Leader</td></tr><tr><td>11/21/2013 8:14:26 AM</td><td>Lewis Jr, Benjamin E (16548)</td><td>Group Leader</td></tr><tr><td>11/22/2013 9:48:11 AM</td><td>Caquelin, Dale A (28652)</td><td>Group Leader</td></tr><tr><td>11/22/2013 1:09:06 PM</td><td>Icenhour, Alan S (31487)</td><td>Division Work Authority</td></tr></table>	Date/Time	Name	Role	11/21/2013 8:09:14 AM	Busby, Jeremy T (742645)	Group Leader	11/21/2013 8:14:26 AM	Lewis Jr, Benjamin E (16548)	Group Leader	11/22/2013 9:48:11 AM	Caquelin, Dale A (28652)	Group Leader	11/22/2013 1:09:06 PM	Icenhour, Alan S (31487)	Division Work Authority
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11/21/2013 8:09:14 AM	Busby, Jeremy T (742645)	Group Leader														
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11/22/2013 9:48:11 AM	Caquelin, Dale A (28652)	Group Leader														
11/22/2013 1:09:06 PM	Icenhour, Alan S (31487)	Division Work Authority														
Description Text	<p>This RSS will be utilized to perform experiments related to recovery of Molybdenum-99 (Mo-99) from irradiated targets. This work will be performed in the Building 3525 hot cells. This RSS applies only to the work performed in the east hot cells, and does not include work associated with out-of-cell testing of the laboratory equipment, or to initial installation of the equipment into the hot cells, as these tasks will be covered under other procedures.</p> <p>The following work tasks will be carried out under this RSS.</p> <ul style="list-style-type: none">• Facility and operations personnel will do all of the hands-on work for this experiment. However, researchers are expected to provide technical direction.• The reactor target used in this experiment is a unique design. It is made of an inner aluminum cylinder, a thin uranium metal foil, and an outer aluminum cylinder. The target is formed by wrapping the uranium foil around the outside of the inner cylinder, sliding the inner cylinder and foil inside the outer cylinder, and pressing the inner cylinder against the outer cylinder by pulling or pushing a die through the hollow center of the target (i.e., swaging). The ends of the target are then welded closed. The uranium metal foil weighs approximately 17 grams, and for this experiment, is enriched to between 7 and 7.5% U-235. The target will be irradiated for one cycle in HFIR prior to processing in 3525.• The irradiated target is placed into a custom tube cutting device, and the target is cut so that the uranium foil can be separated from the inner and outer aluminum cylinders. The device is used to cut the target. First, the welded target ends are cut around the circumference of each end of the target. Then, the target is cut lengthwise at a marked radial position. The lengthwise cut allows the outer cylinder to spring open and away from the inner cylinder and allows the two cylinders to be separated. Once the outer and inner cylinders are separated, the uranium foil may be freed from the target. The cladding materials are discarded, and the uranium foil is used for the remainder of the experiment.• The uranium foil is loaded into a metal basket, and the metal basket is loaded into an electrochemical dissolution vessel.• A strong sodium hydroxide (NaOH) solution is added to the															

	<p>electrochemical dissolution vessel. The sodium hydroxide solution reacts with and dissolves the thin aluminum surface layer on the uranium foil. The uranium foil is unaffected by the sodium hydroxide.</p> <ul style="list-style-type: none"> • The NaOH solution is drained from the vessel, and the vessel is loaded with a sodium carbonate solution. • The DC power supply to the electrochemical dissolution vessel is activated, and the uranium foil is dissolved electrochemically into solution. Dissolution of the uranium foil releases Mo-99 into solution. • Once the uranium foil is completely dissolved, the solution is transferred to another vessel called the mixing vessel. • The sodium carbonate solution is neutralized with nitric acid while being mixed in the mixing vessel. This causes the dissolved uranium and fission products to precipitate out of solution, while Mo-99 remains in solution. • The mixture is filtered to separate the solids from the liquid, and the liquid is recovered as the product of the experiment. • Samples of liquids and solids will be recovered at various points in the process, and the samples will be analyzed in an analytical laboratory by alpha and/or gamma spectroscopy to track the movement of Mo-99 throughout the experiment. • The experimental vessels will be operated at sub-atmospheric pressure, and exhaust gases will be filtered through activated carbon absorption columns and activated carbon filters to capture radioactive iodine. Radioactive iodine is a by-product of the target irradiation process, and radioactive iodine released from the target during the experiment must be captured to the extent possible to limit its migration to the outside atmosphere through the facility exhaust stack. <p>Once the experimental work is concluded, waste liquids will be disposed, and the hardware will be dismantled.</p> <p>These activities will be conducted as permitted by the Facility Safety Basis documentation and the Facility Use Agreement, and as determined within the scope of work authorization documentation as determined by the NSITD Research Operations Manager and the NNFD Facility Manager or their designated alternate(s). Additional detail regarding description of work, equipment and work scope limitations beyond those described in this document can be found in the Facility Safety Basis documentation.</p> <p>A draft researcher procedure is attached to this RSS. This RSS is provided for informational purposes only, and should not be used by operators to perform the actual experiment. A final researcher instruction set will be provided to the operators prior to performing the actual experiment.</p>
Description File	Researcher Operating Instructions, Rev 4..docx
Division	X130: Nuclear Security & Isotope Technology
Start Date	6/28/2013
End Date	3/31/2014
Account	ZZAC9239

General Notes	<p>The experiment will be performed in a hot cell. The hot cell walls will protect the worker against exposure to radiation, radioactive contamination, and chemical hazards while the experiment is underway.</p> <p>Worker exposure to chemical or radiation hazards may occur when samples are removed from the hot cell and transported to Analytical Chemistry for analysis. Appropriate PPE should be worn when handling samples, as defined in the Radiological Work Permit (RWP) for this task.</p> <p><u>Safety Glasses</u></p> <ul style="list-style-type: none"> Safety glasses will be worn when handling samples outside of the hot cell environment. <p><u>Standard Operating Guidelines</u></p> <ul style="list-style-type: none"> Specific procedures for operation of the equipment, and performance of the experiment, will be developed for this experiment. Use of equipment is limited to authorized personnel. <p><u>Radiological Work</u></p> <ul style="list-style-type: none"> Work in Radiological Areas requires an RWP. Notify facility RCTs prior to performing radiological work. <p><u>Integrated Safety Management</u></p> <ul style="list-style-type: none"> Use the Integrated Safety Management (ISM) approach when developing the specific work procedures for this experiment. Keep chemical containers and equipment properly labeled. Line Management is responsible for ensuring that personnel use the designated PPE, required for each task. These requirements may be passed down to staff by the use of such documentation as pre-job briefings, RWPs, special Work Plans, MSDS Sheets and operating procedures or guidelines.
General Attachments	None.
Point of Contact	Maples, Curtis A (24087)
Division Work Authority	Parks, Cecil V (19993)
Principal Investigator	Sherman, Steven R (922849);
PI Delegates	Sherman, Steven R (922849);
Group Leaders	Busby, Jeremy T (742645); Caquelin, Dale A (28652); Lewis Jr, Benjamin E (16548);
Participants	Caquelin, Dale A (28652); Chemical Operator, Group (113); Sherman, Steven R (922849); Woody, Bryan L (947945);
Required Reviewers	Baldwin, Mark E (19003) - ES&H; Drake, David D (21091) - ES&H; Dunsmore, Mary Ruth (676970) - ES&H; Maples, Curtis A (24087) - ES&H; Morris, Robert Noel (26219) - Lab Space

	<p><i>Manager</i>; Murphy, Ann M (30519); Owen, Bruce Allen (22949) - <i>ES&H</i>; Perkins, Dale E (34225) - <i>Rad Protection</i>; Platfoot, John H (32377); Vrooman, Steve (707113) - <i>Rad Protection</i>;</p>
Optional Reviewers	<p>Busby, Jeremy T (742645) - <i>GL</i>; Caquelin, Dale A (28652) - <i>CFM</i>; Lewis Jr, Benjamin E (16548) - <i>GL</i>; Lewis-Lambert, Sandra (32020) - <i>DTO</i>;</p>
Lab Space Managers	<p>None.</p>
Locations	<p>🚩 Building 3525, Room 130; 🚩 Building 3525, Room 131; 🚩 Building 3525, Room EASTCELL</p>
Nepa Documentation	<p>Work in an authorized lab is covered under NEPA Categorical Exclusion Determination for Indoor, Small- and Pilot-Scale Research and Development (3767X), and specifically, B3.6 - Small-scale research and development, laboratory operations, and pilot projects.</p>
Hazards	<p>2.1 This operation involves the generation, handling, processing, use, or storage of radioactive materials (in</p> <p>Potential Training: Please refer to the SBMS Radiation Safety Training exhibit for training requirements. Source Control Custodian - Radioactive Source users shall receive source-specific training provided by the Source Custodian.</p> <p>Requirements: Facility Hazard Categorization Facility Use Agreements Radiological Area Controls Radiological Design Requirements Radiological Dosimetry Radiological Work Safety and Security Regulatory (SSR) Program Sealed Radioactive Source Control</p> <p>Hazard Notes:</p> <ul style="list-style-type: none"> • Hot Cells and some adjacent areas contain radioactive materials. • Storage Wells in the Charging Area may contain radioactive materials. • Manipulations of radioactive materials and samples will be performed in the exposure to radiation, radioactive contamination, and chemical exposure. <p>Control Notes:</p> <ul style="list-style-type: none"> • No nuclear or radiological material may be transferred into the facility or have inventory checked by the NNFD Facility Manager or his designated alternate • Staff must notify the NNFD Facility Manager or his designated alternate prior • The Facility Manager is responsible for ensuring requirements of the FSB and • Work will be done in accordance with established Radiological Work Permits required. • Rad waste will be disposed of in accordance with applicable NNFD and ORNL <p>Locations: Building 3525, Room 130 Building 3525, Room 131 Building 3525, Room EASTCELL</p> <p>Attachments:</p>

None.

2.3 This operation will include the use of enriched uranium, uranium-233, plutonium, neptunium, americium

Potential Training:

See [Meeting NCS Training Requirements](#).

Requirements:

[Facility Hazard Categorization](#)

[Facility Use Agreements](#)

[Nuclear Criticality Safety](#)

[Obtain, Implement, and Comply with Nuclear Criticality Safety \(NCS\) Approvals \(NCSAs\)](#)

[Safety and Security Regulatory \(SSR\) Program](#)

Hazard Notes:

- Enriched uranium, transuranics, and other fission products will be present when the

Control Notes:

- Prior to transferring nuclear material into the IFEL, the proposed transfer must follow established procedures.
- Before transferring fissionable material into the IFEL, staff must check with a supervisor in accordance with ORNL NMC&A procedures prior to initiating the transfer.
- Staff who work with fissionable materials or have other responsibilities for controlling access to Laboratory-level NCS Fundamentals training module ("ORNL NCS Fundamentals") must complete the training to which they are assigned.
- NCSA Facility Specific, NCS Fundamentals, and 3525 Building Access training

Locations:

Building 3525, Room 130

Building 3525, Room 131

Building 3525, Room EASTCELL

Attachments:

None.

2.4 This operation involves work conducted in a [Category 1, 2, or 3 Nuclear Facility](#).

Potential Training:

Facility-Specific Training is required.

Requirements:

[Facility Hazard Categorization](#)

[Facility Use Agreements](#)

[Safety and Security Regulatory \(SSR\) Program](#)

[Safety Basis](#)

[Unreviewed Safety Question \(USQ\) Process for Nuclear and Facility Safety](#)

Hazard Notes:

- Building 3525 is a Cat 2 Nuclear Facility per DOE Standard 1027-92, as amended.
- Work is performed on radiological and irradiated materials.

Control Notes:

- All work must be conducted in compliance with the Facility Safety Basis document and other applicable documents.
- The Unreviewed Safety Question Determination (USQD) process is the means by which physical changes to the Operating Procedures or Standard Operating Guidelines, all of which must be evaluated

Question (USQ) and would require DOE approval prior to implementation. If in doubt, consult the Safety Documentation SME, or the IFEL Facility Manager.

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

Approved SBS and USQD as Built.pdf
USQD SCR 3525 - RSS 11716.0.pdf

5.1 This operation involves work on or near equipment with unguarded, energized electrical components operating.

Potential Training:

DISCONTINUED CERT: Electrical Worker Safety Trng for R & D
DISCONTINUED CERT: High Voltage Safety Awareness Training

Requirements:

Electrical Safety
Lockout/Tagout

Hazard Notes:

- The power supply that will be used with this work is an XR32-62 Series II DC power supply at 62 amps, and a total power output of 2 kW. Under normal operating conditions, the power supply provides power to the electrochemical dissolver through the DC power supply.
- The DC power supply provides power to the electrochemical dissolver through the DC power supply.

Control Notes:

- The power supply cannot exceed 32 VDC due to its design.
- The power supply is equipped with circuits that sense when a short circuit has occurred and eliminate the power to the dissolver.
- Workers operating the electrochemical dissolver shall have the appropriate training and experience.
- The combined electrochemical dissolver/DC power supply system will be inspected and tested. If necessary, the system will be modified (e.g., install covers on the dissolver) to align with ORNL practices for electrical equipment prior to performing work.

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

5.3 This operation involves one or more of the following:

- Electrical equipment or components labeled and listed by a Nationally Recognized Testing Laboratory for the purpose for which they are used;
- Labeled and listed electrical equipment used for something other than its listed purpose;
- Electrical equipment that is labeled by a testing laboratory that is not nationally recognized.

Potential Training:

Electrical Safety Outside the Office

Requirements:

Electrical Safety

Hazard Notes:

- The electrochemical dissolver is not "UL" listed.

Control Notes:

- Request a review and approval from the Authority Having Jurisdiction (AHJ) equipment. If major modifications are made to the Inspection/Examination equipment are resumed.
- Verify approval prior to energizing equipment for the first time or after major

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

7.1 This operation involves work conducted under the OSHA Laboratory Standard.

Potential Training:

General Hazard Communication
ORNL Chemical Hygiene Plan
ORNL Hazard Communication Job Specific Training
OSHA Lab Standard Training - Includes site-specific

Requirements:

Chemical Safety

Hazard Notes:

- Chemical manipulations and equipment utilized meet the OSHA definition of must be conducted in compliance with the ORNL SBMS Chemical Hygiene Safety Requirements (TSR) documentation. Elements of the Chemical Hygiene designated work locations are within HMMIS Control Area XU062.
- Specific chemical hazards include the use of aqueous nitric acid, aqueous sodium

Control Notes:

- Work with chemicals is conducted under the OSHA Laboratory Standard and
- Always review MSDSs before handling or using chemicals.
- The Facility SAR requires that toxic chemical inventories must be maintained
- If you are unsure of the RQ values for a given chemical, consult with your direct Manager as needed to determine the appropriate work control requirements facility.
- Prior to exceeding an RQ for a chemical, a USQD must be completed and approved

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:
None.

7.7 This operation involves chemicals or wastes that are flammable or combustible.

Requirements:
Chemical Safety
Exposure Assessments
Personal Protective Equipment

Hazard Notes:

- During the electrochemical dissolution step, flammable hydrogen gas is generated.

Control Notes:

- Physically, the amount of hydrogen generated is bounded. During dissolution, the amount of hydrogen generated will be less than 0.2 L by volume, or less than 0.02 g of hydrogen by mass. During the uranium foil dissolution step, the amount of hydrogen generated will be about 8 L of hydrogen by volume per period at constant current.
- The rate of hydrogen generation during the uranium foil dissolution step is controlled by the current. When the current is stopped, the generation of hydrogen stops.
- A flow of nitrogen gas will be maintained to the dissolution vessel during electrochemical dissolution. The partial pressure of the hydrogen gas will be suppressed.
- A vacuum pump will be used to draw gas out of the headspace of the dissolution vessel. This will dilute the hydrogen gas in the head space, and drive the concentration of hydrogen down. Preliminary calculations show that maintaining a volumetric flow rate of 5 liters/minute during dissolution of the foil is sufficient to keep the concentration of hydrogen below the lower flammability limit of hydrogen in air (4 vol%). So, even if there is a leak, the concentration of hydrogen inside the dissolution vessel is avoided.
- **The DC power supply must be turned off, and the flow of nitrogen stopped, if the vacuum header reads less than 10 L/min (<20 scfh).** The atmosphere inside the dissolution vessel will be replaced through the mixer stirrer shaft and other leak points in the dissolver cover. This will prevent the formation of a combustible atmosphere inside the dissolver. The gas flow rate must be maintained at 5 liters/minute (>20 scfh) to ensure that the maximum hydrogen concentration is maintained, and so the DC power supply must be turned off and the DC power supply turned back on once the problem has been resolved.
- **The DC power must be turned off if the nitrogen flow stops.** The DC power supply must be turned off if the nitrogen flow stops.

Locations:
Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:
None.

7.8 This operation involves chemicals or wastes that are caustic or corrosive (e.g. acids or bases).

Requirements:
Chemical Safety
Personal Protective Equipment

Hazard Notes:

- Aqueous sodium hydroxide and sodium carbonate will be used to dissolve the
- Aqueous nitric acid will be used to neutralize solutions and to cause precipitation of the acid.

Control Notes:

- Proper controls will be used for all work with these chemicals outside of the installation of the equipment in the hot cell).
- The hot cell will protect workers against exposure when work is performed in
- A catch pan will be placed beneath the equipment to capture spilled liquids.
- If there is potential for splashing, goggles and a face shield must be worn.

Locations:

Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

7.9 This operation involves chemicals or wastes that are toxic.

Requirements:

Chemical Safety
Exposure Assessments
Personal Protective Equipment

Hazard Notes:

- The acids and bases used in this work are toxic if ingested.

Control Notes:

- All work with acids and bases will be performed in the hot cell to prevent acc

Low Risk Exposure Justification:

All chemicals will be handled in a hot cell. Participants are unlikely to exceed any occupational exposure limits (OELs) or recommended maximum exposure limits (RMS).

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

8.3 This operation generates radioactive waste (including mixed hazardous, solid low level, liquid low level, c

Potential Training:

Liquid Waste Generator at ORNL
RCRA 90 Day or 180 Day Accumulation Area
RCRA HAZARDOUS WASTE CHARACTERIZATION AT ORNL
RCRA AND NRC REGULATORY RESTRICTIONS FOR GENERATORS

RCRA SATELLITE ACCUMULATION AREA AT ORNL
Waste Generator Awareness Training

Requirements:

Comply with Clean Water Act
Environmental Management
Manage Waste and Excess Materials

Hazard Notes:

- Radioactive waste (solid low-level, liquid low-level and/or mixed waste) may be released from the equipment, experiment disassembly, sample preparation, and testing or e
- The experimental equipment will need to be dried, dismantled (as needed), and be saved and stored for future work.

Control Notes:

- NNFD personnel are responsible for disposal of radioactive waste from this fa

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

8.8 This operation generates air emissions or uses Class I or II ozone depleting substances.

Requirements:

Comply with Clean Air Requirements
Environmental Management

Hazard Notes:

- Radioactive Kr and Xe will be released from the irradiated target when it is c
- Radioactive iodine will be released from the irradiated target when it is cut, a

Control Notes:

- No capture of Kr and Xe will be performed, and it is assumed that all Kr and Xe will be released. The total amount of Kr and Xe, and the isotopic composition of these gases will be reported to NNFD Management for submittal to the NESHAP annual inventory report.
- The size, enrichment, and position of the target will be adjusted to limit the release of radioactive iodine. However, capture of radioactive iodine is planned in order to limit or eliminate the release of iodine.
- The experimental apparatus will be equipped with an activated carbon iodine trap. The trap will be filled with nitrogen, and the sweep gas will be channeled through the activated carbon trap.
- One of the hot cell HEPA filters be replaced with an activated carbon filter. The filter will be installed into the cell atmosphere from the equipment, or that makes it past the iodine trap.
- No safety credit will be given to the iodine trap installed on the apparatus, and the HEPA filter in the HEPA bank must capture all emitted iodine. Iodine capture data produced by the activated carbon is capable of capturing greater than 99.9% of the molecular iodine. The data was tested by facility personnel to show that flow through the filter does not bypass the filter.
- The activated carbon filter used on the apparatus itself will use activated carbon from a reputable manufacturer. The test data will show that the in-line activated carbon filter

	<p>exhaust gas of the equipment.</p> <ul style="list-style-type: none"> • Isotopic calculations for an irradiated target are provided in the attached file foil that has been irradiated for 25 days in the reactor, and that has been • After the experiment, the in-line activated carbon filter on the apparatus will to decay before the filter is disposed. The amount of I-131 in the filter and attached isotopic calculation table (LEU foil post-25 day irradiation radionuclides) in the target foil as a function of time. Facility personnel will take into account disposing of the filter. <p>Locations: Building 3525, Room 130 Building 3525, Room 131 Building 3525, Room EASTCELL</p> <p>Attachments: Isotopic Calculations for Target.xlsx</p>
8.9	<p>This operation generates wastewater.</p> <p>Potential Training: Liquid Waste Generator at ORNL RCRA HAZARDOUS WASTE CHARACTERIZATION AT ORNL Waste Generator Awareness Training</p> <p>Requirements: Comply with Clean Water Act Environmental Management</p> <p>Hazard Notes: The experimental equipment will contain aqueous radioactive materials at the end of the experiment in the hot cell once the experimental work is completed.</p> <p>Control Notes: Liquid waste materials generated by the experiment will be treated and/or disposed of as liquids. The waste liquids will then be stored in bottles in the east cell until they can be handled on a case-by-case basis. In such cases, bottles may be set aside for a long time. Those bottles will be combined with other compatible LLLW so that the combined material can be handled.</p> <p>Locations: Building 3525, Room 130 Building 3525, Room 131 Building 3525, Room EASTCELL</p> <p>Attachments: None.</p>
9.1	<p>This operation involves activities requiring the use of mechanically-assisted lifting, rigging or hoisting.</p> <p>Potential Training: Basic Rigger</p> <p>Requirements: Material Handling</p> <p>Hazard Notes: The equipment will need to be dismantled and removed from the hot cell at the end of the experiment. The equipment will be stored in the east cell until it can be removed from the hot cell.</p>

Control Notes:

All work of this nature will be performed inside the hot cell, and must be performed by qualified hot cell

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

11.5 This operation involves compressed gases at greater than 100 psi.

Requirements:

Compressed Gas Cylinders and Related Systems

Hazard Notes:

- Compressed nitrogen will be used to provide a sweep gas for the equipment.

Control Notes:

- Compressed gas cylinders will be handled by facility personnel.
- Facility personnel will follow work rules and procedures for handling compressed g

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

14.2 This operation involves work performed outside normal working hours (6am to 7pm).

Hazard Notes:

- The radioisotope under study, Mo-99, has a 2.748d halflife. The experimental work experiment is completed to minimize losses of Mo-99 due to radioactive decay. This (6am to 7pm during weekdays). The work will be planned so that no work is perform

Control Notes:

- NNFD building management will be informed if after-hours operations (after 5:00 areas).
- Some contingent of NNFD personnel must be present when in-cell manipulator work in the Charging Area, Room 130. Support staff includes the technicians perform sample handling.
- NNFD operations staff must be consulted prior to performing any in-cell work to e cell radiological inventory and the type of work being performed. This is a TSR requirement.
- If an emergency occurs while performing work outside normal working hours, eit

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

15.4 This operation involves packaging, transporting, or shipping of samples containing hazardous, radioactive

Hazard Notes:

- Staff may remove analytical samples from the hot cell and transport them to
- Lower activity samples will be transported by hand to 4501 for analysis.

Control Notes:

- Samples will be carried by hand to 4501 by someone with rad worker training, and
- When hand-carrying radiological materials, use shielded containers as appropriate permitted for less than Hazard Category 3 quantities).

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

17.2 This work requires facility modifications or changes to the Facility Use Agreement (e.g. changes in chemical services.)

Requirements:

Facility Use Agreements

Hazard Notes:

There are potentials for modification or changes to research equipment or procedures

Control Notes:

Changes or modifications are screened following SBMS Subject Area - Unreviewed Safety that may affect the authorization basis of a nuclear facility require completion of a safety

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

17.3 There are critical engineering controls or monitoring equipment that must meet an operating standard with exposures, an oxygen deficiency alarm, a cooling water loop for a high temperature apparatus).

Hazard Notes:

Hydrogen gas is generated during dissolution of the aluminum surface layer on the iodine will be released into the gas phase when the uranium foil is dissolved.

Control Notes:

A flow of nitrogen through the apparatus (i.e., gas flush) must be maintained during equipment. The flush gas must be filtered through an iodine trap to capture radioactive iodine in protected by a back-up activated carbon filter that will capture radioactive iodine in nitrogen is lost during performance of the electrochemical dissolution step, then the until the flow of nitrogen through the equipment can be restored.

Locations:

Building 3525, Room 130
Building 3525, Room 131

Building 3525, Room EASTCELL

Attachments:

None.

18.5 This operation requires special controls on procurement, such as PAAA or critical item review.

Requirements:

Purchasing Supplies and Services

Safety and Security Regulatory (SSR) Program

Hazard Notes:

Building 3525 is a Cat 2 Nuclear Facility per DOE Standard 1027-92, as amended. Work is performed on

Control Notes:

Procurement of materials that may affect operation of facility operating systems must be screened to en

Locations:

Building 3525, Room 130

Building 3525, Room 131

Building 3525, Room EASTCELL

Attachments:

None

**Mo-99 Electrochemical Dissolution Experiment
Researcher Operational Procedure, Rev. 0
Extraction of Metal Foil from Cylindrical Target**

Supplies

Target Cutting Device

Cylindrical Target (1.04" outer diameter, 6.5" length)

Open-top liquid nitrogen flask (1 liter capacity, deep enough to hold 1 target)

Liquid nitrogen

Long tongs or tweezers

Procedure

Remove target ends:

1. Place the target in the target cutting device. Align one end of target to overhang the circumferential cutting element (right side of device in the figures) by approximately 3/16" to 1/4".
2. Cut off end of target by turning the crank and adjusting the depth of the cutter.
3. Repeat Steps 1 and 2 to remove the other end of the target.
4. Discard the cut pieces of the target.

Cut the target lengthwise:

5. Place the target in the target cutting device, and align the lengthwise score mark on the target with the lengthwise cutting blade (top of the device).
6. Cut the target lengthwise along the score mark.
 - a. Do not penetrate all of the way through the target. Use the depth gauge to determine the cutting depth, and stop cutting when the bottom of the prongs on the depth gauge are no longer visible when inserted into the cut.

Foil Removal:

7. Ideally, the outer cylinder will spring open and away from the inner cylinder once the longitudinal cut is made. Mechanically separate the inner cylinder from the outer cylinder to free the uranium foil sandwiched in between.

What to do if the foil sticks to the inner tube, the outer tube, or both tubes, and cannot be easily separated:

8. Place the tube in the liquid nitrogen dewar to freeze the tube. Then remove the tube from the dewar and allow it to warm to room temperature.
9. Attempt to remove the foil from the tube using light mechanical force. Be careful not to tear or break the foil, as this will make it more difficult to handle and dissolve in future processing steps.
 - a. If the foil fell off of the tube when the tube was in the dewar, remove the foil from the dewar, and the separation task is complete.

10. If the foil cannot be removed from the tube, repeat the freeze/thaw cycle and try removing it again.
11. If the foil still can't be removed from the tube, then ask the principal investigator for further instructions. Options include:
 - a. Repeating the freeze/thaw cycle, and attempt foil removal again by the same method.
 - b. Attempt to remove the foil by using stronger force and/or alternate tooling.
 - c. Stop the experiment.

Approval of Researcher Instructional Set – Extraction of Metal Foil

Name, Principal Investigator

Date

Name, 3525 Facility Manager

Date

**Mo-99 Electrochemical Dissolution Experiment
Researcher Operational Procedure, Rev. 2
Uranium Foil Processing**

Figure 1. As-built Electrochemical Dissolution Equipment Schematic (two feed bottles shown).

Supplies

- 1.3 L NaOH solution (1M, reagent grade) in graduated 2 L Erlenmeyer flask with threaded cap
- 1.3 L NaHCO₃ solution (1M, ACS grade, prepared from distilled water and bicarbonate powder) in graduated 2 L Erlenmeyer flask with threaded cap
- 500 ml NaHCO₃ solution (1M, ACS grade, prepared from distilled water and bicarbonate powder) in graduated 2 L Erlenmeyer flask with threaded cap
- 100 g CaO powder (pre-weighed, reagent grade) in closed sample container.
- Pressurized nitrogen tank

Pre-Operation Checks

- Confirm that the downstream HEPA filter (K-15-12.2 and K-15-12-4) in each of the online hot cell ventilation system HEPA banks has been replaced with a high efficiency gas absorber filter containing activated charcoal.

Initials

Date

- Confirm that there is documentation available on the HEPA filter that states that the decontamination factor for elemental radioiodine is at least 100.

Initials

Date

- Confirm that the activated carbon trap is installed in-line with the vacuum pump.

Initials

Date

- Confirm that the activated carbon in the in-line trap is identified with a label, that the label matches the supporting documentation on the activated carbon, and that the documentation states that the activated carbon in the in-line trap has an elemental radioiodine decontamination factor of at least 1000.

Initials

Date

- Confirm that a catch pan has been placed beneath the electrochemical platform to capture spills.

Initials

Date

- Confirm that liquids may only be moved from one vessel to the next by deploying the vacuum pump (i.e., no pumping of liquid will occur by pressurizing a tank or vessel above atmospheric pressure and opening an outlet flow path).

Initials

Date

- Confirm that the necessary chemical reagents have been placed in the cell prior to starting the experimental work.

Initials

Date

- Confirm that the nitrogen tank is installed, the tank has nitrogen in it, and the nitrogen line to the apparatus is operational.

Initials

Date

- Confirm that the electrically powered devices for the experiment are operational.

Initials

Date

Operation of the Electrochemical Dissolver Platform

Load the foil into the dissolver

1. Flatten irradiated foil using the manipulators, and place it in the cage. Attempt to flatten the foil as much as practical to ensure good electrical contact with the cage grid. Then, close the cage and tighten the wing nut on the top of the cage to lock the cage.
2. Place basket into the dissolution tank, T-1. Seal the tank using the red lever.

Dissolve the outer aluminum layer

3. Turn on the vacuum pump.
4. Perform dissolution vessel excessive leak check: Open V-2 and look at the pressure gauge on the vacuum header. Does the gauge show a vacuum greater than 1 in Hg? If not, then do the following:
 - a. Close V-2.
 - b. Turn off the vacuum pump.
 - c. Unseal the tank by lifting the red lever.
 - d. Reseat the basket in the tank.
 - e. Lock down the red lever.
 - f. Repeat the vessel leak check.
 - g. If the leak check fails again, increase the draw of the vacuum pump until the vacuum gauge reads greater than 1 in Hg.
 - h. If adjustment of the vacuum draw fails to correct the problem at this point, then there is a large air leak somewhere in the flow path. Stop the experiment, and find and fix the leak before proceeding with the experiment.
5. Activate the mixer motor for the electrochemical dissolver tank, T-1, and adjust the motor control to set it at full speed.
6. Connect M-1 (quick disconnect) to the NaOH feed bottle. Have a second person confirm that the correct feed bottle is attached.

Operator, initials

Observer, initials

Date

7. Open valve V-2 on the vacuum header to pull the liquid from the NaOH feed bottle into the electrochemical dissolution vessel T-1.
8. Close valve V-2 when the feed bottle is empty. An empty feed bottle will be indicated by lack of liquid in the translucent feed line and an increase in the flow rate of gas through the rotameter on the vacuum header.
9. Disconnect M-1 quick disconnect from the NaOH feed bottle. Note that the absolute pressure in the vacuum header will decrease (vacuum gauge pressure will increase), and gas flow through the vacuum header will also decrease (rotameter indicator will show a lower flow rate).
10. Connect M-2 quick disconnect to the electrochemical dissolver – this is the nitrogen feed. Adjust the vacuum pump controller to set the flow rate through the vacuum header at 20-25 scfh.
11. Plug in heat gun and monitor the temperature of the electrochemical dissolver using the external temperature display. Unplug the heat gun when the thermocouples read 60°C or greater.

During Dissolution of Outer Aluminum Layer

- NOTE: The vacuum pump is expected to be operating at all times during the aluminum dissolution step to help dissipate hydrogen and capture radioiodine. If the vacuum pump stops operating during the dissolution, then turn off the flow of nitrogen gas to the dissolution vessel and turn off the heat gun if it is on. Leave the gas flow paths open. When the problem with the vacuum pump is resolved, re-activate the vacuum pump first, and then restore the flow of nitrogen to the tank. If the vessel was being heated at the time
12. Allow 20 minutes to pass after turning off the heat gun.
 13. Close V-2 on the vacuum header.
 14. Open V-3 on the vacuum header.
 15. Open V-11 to drain the liquid from the electrochemical dissolver into the waste collection tank, T-3.
 16. Close V-3 when electrochemical dissolver has been emptied. This is indicated by lack of flow in the drainage line, increase in flow rate in the flow meter on the vacuum header.
 17. Close V-11.

Flush out residual NaOH using NaHCO₃ solution

18. Connect M-1 quick disconnect to the 2 L flask containing 500 ml NaHCO₃ solution. Have a second person confirm that the correct feed bottle is attached.

Operator, initials

Observer, initials

Date

19. Open V-2. This will reduce the pressure in the electrochemical dissolver and cause the contents of the flask to flow into the dissolver.

20. Close V-2 when the flask has been fully drained. This is indicated by lack of flow in the line, and by an increase in the flow rate of gas through the vacuum header.
21. Disconnect M-1 from the flask.
22. Connect M-4 quick disconnect to F-5 on the mixing tank (T-3).
23. Open V-11.
24. Open V-4. Liquid will move from the electrochemical dissolver into the mixing tank.
25. Close V-4 when liquid flow has stopped. This is indicated by a lack of flow in the line, and by an increase in the flow rate of gas through the vacuum header.
26. Connect F-7 quick disconnect fitting to T-2 (liquid waste collection tank).
27. Open V-3 on the vacuum header.
28. Open V-6 on the mixing tank outlet (T-3). This will cause the liquid to drain from the mixing tank (T-3) into the waste collection tank (T-2).
29. Close V-6 when all of the liquid has been drained from the mixing tank into the waste collection tank. This is indicated by no flow in the line, and an increase in the gas flow rate through the vacuum header.
30. Close V-3 on the vacuum header.
31. Close V-11 (inlet to the liquid feed port for the dissolver, T-1).

Dissolve the foil

32. Connect the electrodes to the electrochemical dissolver (positive electrode connects to the basket, negative electrode connects to the body). Cover the terminals with the temporary insulating cover.
33. Connect M-1 quick disconnect to the flask containing 1.3 L NaHCO_3 solution. Have a second person confirm that the correct feed bottle is attached.

Operator, initials

Observer, initials

Date

34. Open V-2.
35. When liquid transfer is complete, disconnect M-1 from the feed flask. This is indicated by an empty flask, lack of liquid flow, and an increase in the gas flow rate through the vacuum header.
36. Activate the DC power supply. Set the power supply at 10 V.
37. Turn on the heat gun, and heat the electrochemical dissolver to about 80°C, then turn off the heat gun.

During Electrochemical Dissolution

- If the temperature of the electrochemical dissolver falls below 75°C, turn on the heat gun, and heat the dissolution vessel until it reaches 80°C.
- If the temperature of the electrochemical dissolver rises above 85°C, turn on the heat gun in “air” mode to cool the vessel. Cool the vessel until the temperature falls to 80°C.
- If the vacuum pump stops operating during the dissolution, turn off the DC power supply and turn off the flow of nitrogen gas to the electrochemical dissolution vessel. Leave the gas flow paths open. When the vacuum pump is restored to operation, first turn back on the flow of nitrogen, and then reactivate the DC power supply.
- If the flow of gas through the vacuum header falls below 20 scfm, then turn off the DC power supply. After the problem is resolved and the proper flow rate is restored, the DC power supply can be reactivated.
 - First try increasing the vacuum draw.
 - If increasing the vacuum draw fails to increase the flow rate or if the vacuum draw is at maximum, try slightly increasing the flow of nitrogen into the vessel and watch for a response in the vacuum header flow meter.
 - If neither of these responses fixes the problem, maintain the hold point and consult with the principal investigator for alternative approaches.

After Electrochemical Dissolution

38. After about 2 hours, turn off the DC power supply to stop the dissolution. Activate the heat gun in “air” mode to cool the vessel. Turn off the heat gun when the temperature of the vessel has cooled below 35°C.
39. Connect F-7 to T-4 (product tank).
40. Connect M-4 to T-3 (mixing tank).
41. Close V-2.
42. Activate the mixer motor for T-3 (mixing tank).
43. Open V-11.
44. Open V-4 on the vacuum header to move the liquid from the electrochemical dissolver into the mixing tank (T-3).
45. Close V-4 when all of the liquid has been drained from the electrochemical dissolver. This is indicated by lack of flow in the liquid line and an increase in the gas flow rate through the vacuum header.
46. Select the CaO bottle. Have a second person confirm that the correct chemical bottle has been selected.

Operator, initials

Observer, initials

Date

47. Load the CaO powder into the feed funnel for the mixing tank. Then, open the valve funnel to drop the CaO into the mixing vessel.
48. After 10 minutes of stirring, open V-5 on the vacuum header.
49. Open V-6 to transport the liquid from the mixing tank (T-3) into the product tank (T-4). The filter in between the two vessels will capture the solids in the line, and the liquid will pass on through to the product tank.
50. Close V-5 when all of the liquid has been transported to the product tank. This is indicated by lack of liquid flow, and by an increase in the flow rate of gas through the vacuum header.
51. Turn off the vacuum pump.
52. Disconnect the nitrogen supply line, M-2, from the electrochemical dissolver, and close the nitrogen regulator.
53. Close V-7 and V-8 on either side of the line filter.

Sample Collection and Analysis

54. Remove in-line filter by disconnecting the lines outside of V-7 and V-8. Bag it out of the hot cell and scan it using gamma spectroscopy.
55. Drain the waste tank (T-2) into a waste liquid container, and obtain a sample of the material (1 ml) using a pipette. Scan the sample using gamma spectroscopy.
56. Drain the product tank (T-4) into another waste liquid container, and obtain a sample of the material (1 ml) using a pipette. Scan the sample using gamma spectroscopy.

Approval of Researcher Instructional Set – Dissolution of Metal Foil

Name, Principal Investigator

Date

Name, 3525 Facility Manager

Date

C-2 SAFETY ANALYSIS FOR TRANSPORT OF HFIR TARGETS: LOOP CASK THERMAL ANALYSIS FOR MO-99 ANNULAR HFIR TARGETS

UT-BATTELLE, LLC DESIGN ANALYSIS CALCULATION - (DAC)

TITLE APPROVAL

PROJECT TITLE Safety Analysis for Transport of HFIR Targets	ACCOUNT NO.	
CALCULATION TITLE: Loop Cask Thermal Analysis for Mo99 Annular HFIR Targets	DAC Number: NNFD-3525-DAC-2013-001-R0	Revision: 0

Signatures

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R0		

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15	6. Results and Discussion
18	7. Conclusions
18	8. References
19	ATTACHMENT 1: Sample output from steady state HEATING 7.3 calculation

REVISION LOG

<u>Revision No.</u>	<u>Issue Date</u>	<u>Description</u>
0		Initial Issue

GENERAL DESIGN AND COMPUTATION SHEET		
PROJECT TITLE Safety Analysis for Transport of HFIR Targets		ACCOUNT NO.
CALCULATION TITLE: Loop Cask Thermal Analysis for Mo99 Annular HFIR Targets Page 1 of 48	DAC Number: NNFD-3525-DAC-2013-001-R0	Revision: 0

1. OBJECTIVES

The purpose of this calculation is to determine the maximum temperature to which HFIR-irradiated Mo99 annular target is exposed during transportation to an onsite facility following HFIR irradiation. Thermal analysis is provided for steady state and transient conditions to which the target might be exposed. The target will be loaded into a holder and transported within the ORNL Loop Cask between nuclear facilities on the ORNL campus. The steady state calculation analyzed is an anticipated condition to which the targets will be subjected after they are loaded into the LOOP cask for transportation. The transient scenario presented is for an unlikely fire event during transport.

The specific objective of this analysis is to determine the maximum temperature that would be reached in the annular target if the heat load is 24 W.

2. METHOD OF ANALYSIS

Axisymmetric (r - z) thermal calculations are performed using HEATING7 (Ref. 1). The objective is to provide the maximum temperature reached by the target during a steady state and hypothesized 10-minute fire.

For the steady state, the target temperature must rise until the decay heat generated in the targets is removed to the ambient air outside of the cask (Fig. 1).

3. GEOMETRY

3.1 Target capsule assembly

The target is shown in Fig. 1. HEATING7 geometry is defined by "regions." The heated annular region which represents the irradiated target is number 50. The outside dimension is chosen as a minimum so that the maximum air gap between the target and holder is modeled, thus providing a conservative estimate of the temperature.

3.2 Target Holder

The aluminum target holder is shown in Fig. 2. To keep the geometry axisymmetric, the target is modeled on the centerline instead of the off-axis target position shown in Fig. 3. This assumption has little affect on the result because the aluminum holder has such a high thermal conductivity that it cannot sustain a significant temperature gradient.

3.3 ORNL LOOP Cask

The target will be inserted into the holder and then shipped within the ORNL LOOP Cask which is shown in Fig. 3. The cylindrical inside cavity is lined with 0.125-in (3-mm-thick) 304 stainless steel which has an inside diameter of 5.75 inches (14.6 cm) and is 69 inches (175.3 cm) long. The top and bottom plugs are also lined with stainless steel. Most of the cask weight is due to the lead shielding that surrounds the cavity. This cask has common usage at ORNL and has been analyzed previously (Ref. 2). There is a concern that during a fire transient, some of the lead shielding could melt. Because only 10-minute fires are analyzed here, this concern is not applicable, since the maximum temperature reached in the lead during the fire is 307°C, which is less than the melting temperature.

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Figure 1. Geometry of annular target design for analysis.

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Figure 2. Geometry of target capsule basket used for transporting up to 3 targets.

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Figure 3. ORNL LOOP Cask from Drawing N3E020566A086, Chemical Technology Division, Oak Ridge National Laboratory.

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4. PHYSICAL ASSUMPTIONS

4.1 Boundary conditions

4.1.1 External boundary conditions (Surface-to-boundary)

Boundary conditions are listed in Table 4. BC number 4 is used to represent the heat exchange to/from the cask exterior to the outside environment. The temperature of the environment is assumed to be 100°F (37.8°C) during the steady state and post-fire transients. For the standard fire transient the environmental temperature is 800°C (1475°F) (per DOE 10CFR71 page 352). This temperature was used to analyze a fire duration of 10 minutes.

The heat sink for the steady state simulation is the outside air, and heat transfer to this heat sink occurs from the cask by radiation and natural convection.

Natural convection from the cask exterior was modeled in Ref. 3 (Based on Ref. 4) as;

$$h_{\text{naturalconvection}} = 1.24 \times 10^{-4} (\Delta T)^{1/3} \text{ W}/(\text{cm}^2 \cdot ^\circ\text{C}) .$$

This expression will lead to an under-prediction of natural convection by about 6-32% as quantified in Ref. 3. The software in Ref. 3 has been written specifically for analyzing these types of fire transients with HEATING7. The coefficient used as the default in HTAS1 ($1.10 \times 10^{-4} \text{ W}/(\text{cm}^2 \cdot ^\circ\text{C}^{4/3})$) is used here as well to account for the effect of natural convection to the ambient.

The external shell of the Loop cask is made from mild (carbon) steel. According to Ref. 5, the emissivity of mild steel that is not polished is between 0.66 and 0.81. In Ref. 6, the emissivity of 0.62 was used for the Loop cask exterior. This lower value of 0.62 is more conservative for the steady state and transient, so it is assumed here for the cask liner.

In the fire transient, there is no heat sink. Instead the environment is a heat source, and as long as the fire transient lasts, heat flows into the cask and its internals. In keeping with the specifications in Part 71 of Title 10, Chapter 1, Code of Federal Regulations-Energy, U.S. Nuclear Regulatory Commission, the value for the emissivity on the surfaces of the package during the fire is 0.8. A conservative value of 1.0 is used for the environment. Natural convection is also allowed on the external boundary during the fire using the same coefficient (per Ref. 3), but it makes very little difference in the temperature solution.

4.1.2 Internal boundary radiation (Surface-to-surface)

Boundary condition input (2, 3, 5, 6, 7 and 8) are used in HEATING7.3 to define one-dimensional connections between internal surfaces for radiation across air spaces. These internal connections are made between the target and the holder and between the holder and the cask liner.

A reliable value for a polished aluminum emissivity would be 0.05 (varies very little with temperature) which is used for the target capsule in all simulations. The stainless steel varies from 0.16 at room temperature to about 0.3 at 1000°C, but with a rough oxide layer (Ref. 7), it could as high as 0.73 at 827 °C. In Ref. 2, values of 0.62 @ 216 °C and 0.73 @ 527°C are given which is consistent with the highest values found in the texts. Since the lower emissivities have been found (Ref. 2) to lead to highest

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predicted target assembly temperature for both the steady state and the transient fire, the 0.16 value is assumed.

Based on these emissivities, the exchange factors for radiation heat transfer between the internal surface pairs are provided in Table 1.

Table 1. Surface-to-surface radiation exchange factors supplied to HEATING7 for cask simulation.

BC	Region	Component	Orientation	Component	H7 multiplier
2	41	Holder	Radial	Cask Liner	2.319×10^{-13}
3	42, 43	Cask Liner	Axial	top cask liner	4.930×10^{-13}
4	1, 13, 25, 36, 37	cask	Axial & Radial	Ambient	3.515×10^{-12}
5	51	Target	radial	holder	1.499×10^{-13}
6	44	Picker Shaft	Radial	Liner-corner	7.785×10^{-13}
7	54	Holder	Axial	shutter	1.454×10^{-13}
8	40	Picker shaft	Radial	Cask Liner	8.136×10^{-13}

Corresponding HEATING7 input for Table 1 is shown below. Note that BC6 is unused.

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4.2 Heat generation

The volumetric heat generation applied in this problem represents the total decay heat of the assembly. A constant value is used for the annulus which represents the target. The value is set as to yield a total decay heat of either 24 W or 48 W, the two cases that were simulated for this analysis.

4.3 Material Properties

Conductivity, specific heat, density

In Table 2 the properties of the materials that were used in the simulation are shown. If the property was treated as a variable function of temperature, then the figure number which shows the temperature dependence of the property is indicated.

The conductivity of the air is taken from Ref. 8 (p. 503).

Lead thermal conductivity is taken from Ref. 9. The lead values span the phase change temperature, and the thermal conductivity is less for liquid than for solid. As noted previously, during the 10-minute fire, there is no melting.

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Table 2. Thermal properties of materials used for the Loop cask containing Mo99 targets inside target holder.

Material	Thermal Conductivity (W/cm-°C)	Density (kg/cm ³)	Specific Heat (J/kg-°C)	Total Hemispherical Emissivity	
				Minimum	Maximum
Aluminum	1.72 @ 100°C Figure 4	2.7×10 ⁻³	850 @ 100°C Figure 5	0.05	0.05
Air	0.32×10 ⁻³ @ 100°C Figure 4	0.9458×10 ⁻⁶ Figure 6	1009 @ 100°C Figure 5	NA	NA
Stainless Steel 304	0.154 @ 100°C Figure 4	7.978×10 ⁻³	504 @ 100°C Figure 5	0.16	0.73
Lead	0.343 @ 100°C Figure 4	0.01134	131 @ 100°C Figure 5	NA	NA
Mild (Carbon) Steel	0.567 @ 100°C Figure 4	7.854×10 ⁻³	471 @ 100°C Figure 5	0.62	0.8

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Figure 4. Thermal conductivities of materials used in thermal analysis.

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Figure 5. Specific heat capacity of materials used in thermal analysis.

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Figure 6. Density of air used in thermal analysis.

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5. CALCULATION INPUT

HEATING7.3 has been used to perform all of the thermal simulations presented here. The materials by color are shown over the axisymmetric computational domain at the top of the cask in Fig. 7. Figure 8 shows a close-up on the top part of the LOOP cask cavity around annular target.

There are three parts to each of the two simulations: an initial steady state, a 10-minute fire transient (where the boundary emissivity and environment temperature are suddenly increased), and a post-fire transient (where the environment temperature is decreased back to 37.8°C).

Case 1 is for the expected full-loaded target case at 24 W decay power. Case 2, at 48 W was included to show the power dependence of the maximum temperatures computed by the model.

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Figure 7. Materials in the axisymmetric cask domain near the top end of the LOOP cask.

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Figure 8. Materials in the axisymmetric cask domain around annular Mo99 target..

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6. RESULTS AND DISCUSSION

Table 3 presents the maximum temperatures obtained for the 10-minute fire transient simulations. The pre-fire steady state temperature distribution is shown in Fig. 9 for the 24 W case.

Figure 10 shows the time-dependent maximum temperature for the 10-minute fire transient and post-fire cool-down all the way to 50,000 s (13.9 hours).

Table 3. Summary of results for 10-minute fire cases that were simulated for thermal analysis of Mo99 targets shipped inside of Loop cask

Case	Decay Heat Load (W)	Maximum Capsule Temperature in the Initial Steady State (°C)	Maximum Capsule Temperature in the Transient (°C)	Time During Transient that Capsule Max Temperature is Reached (min)
1	24	122	172	283
2	48	194	237	270

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Figure 9. Steady-state temperature distribution for 24 W .

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Figure 10. Maximum temperature in the target for the fire transient and cool-down period.

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7. CONCLUSIONS

An axisymmetric thermal model has been built and used to simulate the heat transfer within the LOOP cask during a steady state and fire transient. The contents of the cask consist of an aluminum holder with a single annular Mo99 target.

For a 24 W decay heat load, the maximum temperature in the pellets will not exceed 172°C due to a 10-minute fire at 800°C.

For a 48 W decay heat load, the maximum temperature in the pellets will not exceed 237°C due to a 10-minute fire at 800°C.

8. REFERENCES

1. ORNL/TM-12262, HEATING 7.2 User's Manual, February 1993, K. W. Childs.
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4. Heat Transfer, J. P. Holman, McGraw-Hill Book Company Inc, Fourth Edition, 1976, pp 244-253, p263 – horizontal cylinder natural convection.
5. Thermal Radiation Heat Transfer, R. Siegel, and J. Howell, Second Edition, Hemisphere Publishing Corporation, 1981, Appendix D, "Radiative Properties."
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ATTACHMENTS

ATTACHMENT 1: Output from steady state HEATING 7.3 calculation

Current Time: 20 Feb 2013 14:13:44

```
H H EEEEE AAA TTTT III N N GGG
H H E A A T I N N G G
H H E A A T I NN N G
HHHHH EEE AAAAA T I N N N G
H H E A A T I N NN G GG
H H E A A T I N N G G
H H EEEEE A A T III N N GGG
```

Version : HEATING 7.3
Last modified: March 10, 2005

Contact : Kenneth W. Childs
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Address : Modeling and Simulation Group
Computational Sciences and Engineering Division
Oak Ridge National Laboratory
Post Office Box 2008
Oak Ridge, Tennessee 37831-6085

***** ECHO OF INPUT DATA *****

```
Record
1 cask
2 1000 3 0 1
3 regions
4 * Stainless
5 1 1 0.0 14.6 0 0 0.0 2.54
6 0 0 0 4 0 0 4 0
7 2 1 0.0 9.8 0 0 2.54 8.9
8 0 0 0 0 0 0 0 0
9 3 1 0.0 7.3 0 0 23.6 26.4
10 0 0 0 0 0 0 0 0
11 4 1 7.3 9.8 0 0 8.9 26.4
12 0 0 0 0 0 0 0 0
13 5 1 9.8 10.8 0 0 2.54 26.4
14 0 0 0 0 0 0 0 0
15 6 1 10.8 14.6 0 0 2.54 6.0
16 0 0 0 0 0 0 0 0
17 7 1 5.08 7.3 0 0 26.4 28.3
18 0 0 0 0 0 0 0 0
19 8 1 0 1.0 0 0 189.5 195.3
20 0 0 0 0 0 0 0 0
21 9 1 0.0 1.0 0 0 195.3 226.4
22 0 0 0 0 0 0 0 0
```


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```

23 10 1 0.0 1.0 0 0 226.4 231.4
24 0 0 0 0 0 0 0 0
25 11 1 7.3 7.62 0 0 26.4 201.6
26 1 0 0 0 0 0 0 0
27 12 1 7.62 10.8 0 0 26.4 27.0
28 1 0 0 0 0 0 0 0
29 13 1 10.8 11.1 0 0 226.4 235.0
30 1 0 0 4 0 0 0 4
31 14 1 0.0 10.8 0 0 234.4 235.0
32 1 0 0 0 0 0 0 4
33 15 1 5.08 7.3 0 0 195.9 201.6
34 0 0 0 0 0 0 0 0
35 16 1 1.0 2.0 0 0 216.9 226.4
36 0 0 0 0 0 0 0 4
37 17 1 1.0 1.3 0 0 201.6 216.9
38 0 0 0 0 0 0 0 0
39 18 1 2.0 2.4 0 0 216.9 225.1
40 0 0 0 0 0 0 0 0
41 19 1 1.3 9.4 0 0 201.6 202.4
42 0 0 0 0 0 0 0 0
43 20 1 7.62 9.8 0 0 201.0 201.6
44 0 0 0 0 0 0 0 0
45 21 1 9.4 9.8 0 0 201.6 225.1
46 0 0 0 0 0 0 0 0
47 22 1 9.8 10.5 0 0 201.0 225.1
48 0 0 0 0 0 0 0 0
49 23 1 10.5 14.3 0 0 221.9 225.1
50 0 0 0 0 0 0 0 0
51 24 1 2.0 11.1 0 0 225.1 226.4
52 0 0 0 0 0 0 0 0
53 25 1 11.1 14.3 0 0 225.1 226.4
54 0 0 0 4 0 0 0 4
55 * Lead Regions
56 27 2 0.0 7.3 0 0 8.9 23.6
57 0 0 0 0 0 0 0 0
58 28 2 14.6 29.0 0 0 3.5 6.0
59 0 0 0 0 0 0 0 0
60 29 2 10.8 29.0 0 0 6.0 27.0
61 0 0 0 0 0 0 0 0
62 30 2 7.62 29.0 0 0 27.0 201.0
63 1 0 0 0 0 0 0 0
64 31 2 1.3 9.4 0 0 202.4 216.9
65 0 0 0 0 0 0 0 0
66 32 2 2.4 9.4 0 0 216.9 225.1
67 0 0 0 0 0 0 0 0
68 33 2 14.3 29.0 0 0 221.9 224.1
69 0 0 0 0 0 0 0 0
70 34 2 10.5 29.0 0 0 201.0 221.9
71 0 0 0 0 0 0 0 0
72 * Carbon Steel
73 35 5 14.3 29.0 0 0 224.1 225.1
74 0 0 0 0 0 0 0 4
75 36 5 29.0 30.48 0 0 2.54 225.1
76 0 0 0 4 0 0 4 4
77 37 5 14.6 29.0 0 0 2.54 3.5
78 0 0 0 0 0 0 4 0
79 * Air inside cask

```

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```

80 38 3 0.0 5.08 0 0 26.4 28.3
81 0 0 0 0 0 0 0 0
82 39 3 0.0 7.3 0 0 28.3 156.91
83 0 0 0 0 0 0 0 0
84 40 3 1.0 7.3 0 0 189.5 195.3
85 0 0 8 8 0 0 0 0
86 41 3 6.19 7.3 0 0 156.91 189.5
87 0 0 2 2 0 0 0 0
88 42 3 1.0 10.8 0 0 226.4 234.4
89 0 0 0 0 0 0 3 3
90 43 3 0.0 1.0 0 0 231.4 234.4
91 0 0 0 0 0 0 3 3
92 44 3 1.0 5.08 0 0 195.3 201.6
93 0 0 6 6 0 0 0 0
94 45 3 5.08 7.3 0 0 195.3 195.9
95 0 0 0 0 0 0 0 0
96 *
97 * Plug in bottom of holder
98 46 4 0.0 1.588 0 0 156.91 160.72
99 0 0 0 0 0 0 0 0
100 * Heated annular region
101 * ID = 1.043 inches = 2*1.32 cm
102 * OD = 1.043 inches = 2*1.49 cm
103 50 4 1.32 1.49 0 0 160.72 176.92
104 0 1 0 0 0 0 0 0
105 * critical air gap
106 51 3 1.49 1.588 0 0 160.72 176.92
107 0 0 5 5 0 0 0 0
108 * Air-filled space above inside of target
109 52 3 0.0 1.32 0 0 160.72 176.92
110 0 0 0 0 0 0 0 0
111 * Air-filled space above target
112 53 3 0.0 1.588 0 0 176.92 187.39
113 0 0 0 0 0 0 0 0
114 * 12-inch-high Holder
115 * with 1.25-in-diam hole (radius = 1.588 cm)
116 54 7 1.588 6.19 0 0 156.91 187.39
117 0 0 0 0 0 0 0 0
118 * air gap between holder lid and holder
119 55 3 1.0 6.19 0 0 187.39 187.79
120 0 0 0 0 0 0 7 7
121 * steel nub between holder lid and holder
122 56 7 0.0 1.0 0 0 187.39 187.79
123 0 0 0 0 0 0 0 0
124 * holder lid
125 57 7 0.0 1.0 0 0 187.79 189.5
126 0 0 0 0 0 0 0 0
127 58 7 1.0 6.19 0 0 187.79 189.5
128 0 0 0 0 0 0 0 0
129 *
130 materials
131 1 Ssteel 1.0 0.00798 1.0 -1 0 -7 0
132 2 lead 1.0 0.010768 1.0 -2 0 -8 1
133 327.6 26000.
134 3 Air 1.0 1.0 1.0 -3 -6 -9 0
135 4 Al6061T6 1.0 0.0027 1.0 -4 0 -10 0
136 5 Csteel 1.0 0.00798 1.0 -5 0 -11 0

```

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```

137 *Basket stainless with reduced conductivity and density
138 7 AlHolder 1.0 0.0027 1.0 -4 0 -10 0
139 9 Al-axial 0.79 0.0021 1.0 -4 0 -10 0
140 *
141 initial temperatures
142 1 100
143 *
144 * CL 3/8-inch-long (0.9525-cm-long) capsule has total heat of 1.2 W
145 * apply heat load to Al core region, so
146 * CL heat generation = 1.2/0.9525/(pi*0.417^2) = 2.3 W/cm^3
147 heat generations
148 1 0.9872 0 0 0 0 0
149 *
150 boundary conditions
151 * from holder to cask liner
152 2 3
153 0 2.319E-13
154 * from Basket rod to cask liner axial
155 3 3
156 0 4.93043E-13
157 * From cask to ambient Tambient = 37.7 C
158 4 1 37.7
159 0 3.5154E-12 1.022e-4 0.333
160 * from target to holder
161 5 3
162 0 1.499E-13
163 * from picker shaft, radial to cask liner corner
164 6 3
165 0 7.785E-13
166 * from holder to holder lid
167 7 3
168 0 1.454E-13
169 * from picker shaft, radial to cask liner
170 8 3
171 0 8.136E-13
172 xgrid
173 * 0.306 ID on Mo specimen, inside of 0.5-inch tube
174 * 2.2 inches long
175 0 1.0 1.3 1.32 1.49 1.588 2.0
    @2.4 4.44 4.76 5.08 6.19 7.3 7.62 9.4 9.8
    @10.5 10.8 11.1 14.3 14.6 29.0 30.48
176 2 2 1 1 1 2 2 1
    @ 3 2 1 5 2 4 1 1
    @ 5 1 3 2 12 2
177 *
178 zgrid
179 0.0 2.54 3.5 6.0 8.9 23.6 26.4 27.0
    @28.3 53.1 156.91 160.72 176.92
    @187.39 187.79 189.5 195.3 195.9
    @201.0 201.6 202.4 216.9 221.9
    @224.1 225.1 226.4 231.4 234.4 235.0
180 2 2 1 1 6 2 1 1
    @ 10 34 5 12 10
    @ 1 2 4 1 5
    @ 1 1 5 1 2
    @ 1 1 3 2 1
181 *

```

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```

182 tabular functions
183 *
184 * conductivity table for aluminum (deg C W/cm-K)
185 *
186 4 9
187 -223.,1.6855,20.,1.6686,50.,1.692,75.,1.7088,
    @100.,1.7233,125.0,1.7358,150.,1.7483,175.,1.7593,
    @200.,1.7689
188 *
189 * Specific Heat table for aluminum (J/kg.C)
190 *
191 10 6
192 -173.15,482,-73.15,798.,27.15,903.,126.85,949.,
    @326.85,1033.,526.85,1146.
193 *
194 *
195 * conductivity table for air (deg C W/cmK)
196 *
197 3 34
198 -173.15,0.00009246,-123.15,0.00013735,-73.15,0.0001809,
    @-23.15,0.0002227,26.85,0.0002624,76.85,0.0003003,
    @126.85,0.0003365,176.85,0.0003707,226.85,0.0004038,
    @276.85,0.0004360,326.85,0.0004659,376.85,0.0004953,
    @426.85,0.0005230,476.85,0.0005509,526.85,0.0005779,
    @576.85,0.0006028,626.85,0.0006279,676.85,0.0006525,
    @726.85,0.0006752,826.85,0.000732,926.85,0.000782,
    @1026.85,0.000837,1126.85,0.000891,1226.85,0.000946,
    @1326.85,0.00100,1426.85,0.00105,1526.85,0.00111,
    @1626.85,0.00117,1726.85,0.00124,1826.85,0.00131,
    @1926.85,0.00139,2026.85,0.00149,2126.85,0.00161,
    @2226.85,0.00175
199 *
200 * Air density ( kg/cc)
201 6 19
202 25.,1.184e-6,40.,1.127e-6,50.,1.092e-6,70.0,1.028e-6,
    @100.,0.9458e-6,120.,0.8977e-6,160.,0.8148e-6,
    @200.,0.7459e-6,300.,0.6158e-6,350.,0.5664e-6,
    @400.,0.5243e-6,450.,0.488e-6,500.,0.4565e-6,
    @600.,0.4042e-6,700.,0.3627e-6,800.,0.3289e-6,
    @900.,0.3008e-6,1000.,0.2772e-6,2000.,0.1553e-6
203 *
204 * Air Specific Heat (J/kg.C)
205 9
206 25.,1007.,100.,1009,140.,1013.,200.,1023.,
    @400.,1069.,600.1115.,800.,1153.,
    @1000.,1184.,2000.,1264.
207 * conductivity table for 347 stainless (deg C W/cm-K)
208 *
209 1
210 27.,0.142,126.85,0.158,326.85,0.189,
    @526.85,0.219,726.85,0.247
211 *
212 * Specific heat table for 347 stainless (J/kg.C)
213 *
214 7 5
215 27.,480.,126.85,513.,326.85,559.,
    @526.85,585.,726.85,606.

```

GENERAL DESIGN AND COMPUTATION SHEET

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```

216 *
217 * conductivity table for lead (deg C W/cm-K)
218 *
219 2 23
220 27,0.4584101,50,0.462672,100,0.471937,150,0.481202,
    0200,0.490467,250,0.499732,300,0.508997,320,0.512703,
    0328,0.1569118,350,0.1595342,400,0.1654942,
    0450,0.1714542,500,0.1774142,550,0.1833742,
    0600,0.1893342,650,0.1952942,700,0.2012542,
    0750,0.2072142,800,0.2131742,850,0.2191342,
    0900,0.2250942,950,0.2310542,1000,0.2370142
221 *
222 *
223 * Specific Heat table for lead (J/kg.C)
224 8 5
225 -173.15,118.,-73.15,125.,27.0,129.,126.85,132.,
    0326.85,142.
226 *
227 *
228 * conductivity table for carbon steel (deg C W/cm-K)
229 *
230 5 4
231 126.85,0.567,326.85,0.480,526.85,0.392,726.85,0.300
232 * Specific Heat table for carbon steel (J/kg.C)
233 *
234 11 5
235 27.15,434.,126.85,487.,326.85,559.,
    0526.85,685.,726.85,1169
236 * decay heat generation distribution = 1.0
237 13
238 0 0
    0159.01      0.0
    0159.02      1.0
    0189.5       1.0
    0189.51      0.0
    0300.00      0
239 *
240 *Printout Times
241 *2000 4000 6000 8000 10000 12000 14000 16000
242 *Nodes monitored
243 *1 3159
244 steady state
245 2 20000
246 %

```

***** CASE DESCRIPTION *****

cask

***** SUMMARY OF PARAMETER CARD DATA *****

```

Maximum cpu time - 1000.00 seconds
Geometry type number - 3 (rz )
Initial time - 0.0000000E+00

```

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This is a restart of previous case - No
 Read node-to-node connector data file - No
 Redirect or suppress convergence information - Yes (Suppress)

Output selected information during calculations - No
 Write surface heat fluxes to plot file - No

Units used in problem solution and in material properties library

	Problem	Library
Time	- s	s
Length	- m	cm
Mass	- kg	g
Energy	- J	cal
Temperature	- C	C

***** SUMMARY OF REGION DATA *****

Region Number	Material Number	Initial Temp. No.	Heat Gen. Number
1	1	0	0
2	1	0	0
3	1	0	0
4	1	0	0
5	1	0	0
6	1	0	0
7	1	0	0
8	1	0	0
9	1	0	0
10	1	0	0
11	1	1	0
12	1	1	0
13	1	1	0
14	1	1	0
15	1	0	0
16	1	0	0
17	1	0	0
18	1	0	0
19	1	0	0
20	1	0	0
21	1	0	0
22	1	0	0
23	1	0	0
24	1	0	0
25	1	0	0
27	2	0	0
28	2	0	0
29	2	0	0
30	2	1	0
31	2	0	0
32	2	0	0
33	2	0	0
34	2	0	0
35	5	0	0

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36	5	0	0
37	5	0	0
38	3	0	0
39	3	0	0
40	3	0	0
41	3	0	0
42	3	0	0
43	3	0	0
44	3	0	0
45	3	0	0
46	4	0	0
50	4	0	1
51	3	0	0
52	3	0	0
53	3	0	0
54	7	0	0
55	3	0	0
56	7	0	0
57	7	0	0
58	7	0	0

Region Number	----- Dimensions / Boundary Numbers -----					
	First Axis		Second Axis		Third Axis	
	Smaller	Larger	Smaller	Larger	Smaller	Larger
1	0.0000E+00	1.4600E+01	0.0000E+00	0.0000E+00	0.0000E+00	2.5400E+00
	0	4	0	0	4	0
2	0.0000E+00	9.8000E+00	0.0000E+00	0.0000E+00	2.5400E+00	8.9000E+00
	0	0	0	0	0	0
3	0.0000E+00	7.3000E+00	0.0000E+00	0.0000E+00	2.3600E+01	2.6400E+01
	0	0	0	0	0	0
4	7.3000E+00	9.8000E+00	0.0000E+00	0.0000E+00	8.9000E+00	2.6400E+01
	0	0	0	0	0	0
5	9.8000E+00	1.0800E+01	0.0000E+00	0.0000E+00	2.5400E+00	2.6400E+01
	0	0	0	0	0	0
6	1.0800E+01	1.4600E+01	0.0000E+00	0.0000E+00	2.5400E+00	6.0000E+00
	0	0	0	0	0	0
7	5.0800E+00	7.3000E+00	0.0000E+00	0.0000E+00	2.6400E+01	2.8300E+01
	0	0	0	0	0	0
8	0.0000E+00	1.0000E+00	0.0000E+00	0.0000E+00	1.8950E+02	1.9530E+02
	0	0	0	0	0	0
9	0.0000E+00	1.0000E+00	0.0000E+00	0.0000E+00	1.9530E+02	2.2640E+02
	0	0	0	0	0	0
10	0.0000E+00	1.0000E+00	0.0000E+00	0.0000E+00	2.2640E+02	2.3140E+02
	0	0	0	0	0	0
11	7.3000E+00	7.6200E+00	0.0000E+00	0.0000E+00	2.6400E+01	2.0160E+02
	0	0	0	0	0	0
12	7.6200E+00	1.0800E+01	0.0000E+00	0.0000E+00	2.6400E+01	2.7000E+01
	0	0	0	0	0	0
13	1.0800E+01	1.1100E+01	0.0000E+00	0.0000E+00	2.2640E+02	2.3500E+02
	0	4	0	0	0	4
14	0.0000E+00	1.0800E+01	0.0000E+00	0.0000E+00	2.3440E+02	2.3500E+02
	0	0	0	0	0	4
15	5.0800E+00	7.3000E+00	0.0000E+00	0.0000E+00	1.9590E+02	2.0160E+02
	0	0	0	0	0	0
16	1.0000E+00	2.0000E+00	0.0000E+00	0.0000E+00	2.1690E+02	2.2640E+02
	0	0	0	0	0	4

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17	1.0000E+00	1.3000E+00	0.0000E+00	0.0000E+00	2.0160E+02	2.1690E+02
	0	0	0	0	0	0
18	2.0000E+00	2.4000E+00	0.0000E+00	0.0000E+00	2.1690E+02	2.2510E+02
	0	0	0	0	0	0
19	1.3000E+00	9.4000E+00	0.0000E+00	0.0000E+00	2.0160E+02	2.0240E+02
	0	0	0	0	0	0
20	7.6200E+00	9.8000E+00	0.0000E+00	0.0000E+00	2.0100E+02	2.0160E+02
	0	0	0	0	0	0
21	9.4000E+00	9.8000E+00	0.0000E+00	0.0000E+00	2.0160E+02	2.2510E+02
	0	0	0	0	0	0
22	9.8000E+00	1.0500E+01	0.0000E+00	0.0000E+00	2.0100E+02	2.2510E+02
	0	0	0	0	0	0
23	1.0500E+01	1.4300E+01	0.0000E+00	0.0000E+00	2.2190E+02	2.2510E+02
	0	0	0	0	0	0
24	2.0000E+00	1.1100E+01	0.0000E+00	0.0000E+00	2.2510E+02	2.2640E+02
	0	0	0	0	0	0
25	1.1100E+01	1.4300E+01	0.0000E+00	0.0000E+00	2.2510E+02	2.2640E+02
	0	4	0	0	0	4
27	0.0000E+00	7.3000E+00	0.0000E+00	0.0000E+00	8.9000E+00	2.3600E+01
	0	0	0	0	0	0
28	1.4600E+01	2.9000E+01	0.0000E+00	0.0000E+00	3.5000E+00	6.0000E+00
	0	0	0	0	0	0
29	1.0800E+01	2.9000E+01	0.0000E+00	0.0000E+00	6.0000E+00	2.7000E+01
	0	0	0	0	0	0
30	7.6200E+00	2.9000E+01	0.0000E+00	0.0000E+00	2.7000E+01	2.0100E+02
	0	0	0	0	0	0
31	1.3000E+00	9.4000E+00	0.0000E+00	0.0000E+00	2.0240E+02	2.1690E+02
	0	0	0	0	0	0
32	2.4000E+00	9.4000E+00	0.0000E+00	0.0000E+00	2.1690E+02	2.2510E+02
	0	0	0	0	0	0
33	1.4300E+01	2.9000E+01	0.0000E+00	0.0000E+00	2.2190E+02	2.2410E+02
	0	0	0	0	0	0
34	1.0500E+01	2.9000E+01	0.0000E+00	0.0000E+00	2.0100E+02	2.2190E+02
	0	0	0	0	0	0
35	1.4300E+01	2.9000E+01	0.0000E+00	0.0000E+00	2.2410E+02	2.2510E+02
	0	0	0	0	0	4
36	2.9000E+01	3.0480E+01	0.0000E+00	0.0000E+00	2.5400E+00	2.2510E+02
	0	4	0	0	4	4
37	1.4600E+01	2.9000E+01	0.0000E+00	0.0000E+00	2.5400E+00	3.5000E+00
	0	0	0	0	4	0
38	0.0000E+00	5.0800E+00	0.0000E+00	0.0000E+00	2.6400E+01	2.8300E+01
	0	0	0	0	0	0
39	0.0000E+00	7.3000E+00	0.0000E+00	0.0000E+00	2.8300E+01	1.5691E+02
	0	0	0	0	0	0
40	1.0000E+00	7.3000E+00	0.0000E+00	0.0000E+00	1.8950E+02	1.9530E+02
	8	8	0	0	0	0
41	6.1900E+00	7.3000E+00	0.0000E+00	0.0000E+00	1.5691E+02	1.8950E+02
	2	2	0	0	0	0
42	1.0000E+00	1.0800E+01	0.0000E+00	0.0000E+00	2.2640E+02	2.3440E+02
	0	0	0	0	3	3
43	0.0000E+00	1.0000E+00	0.0000E+00	0.0000E+00	2.3140E+02	2.3440E+02
	0	0	0	0	3	3
44	1.0000E+00	5.0800E+00	0.0000E+00	0.0000E+00	1.9530E+02	2.0160E+02
	6	6	0	0	0	0
45	5.0800E+00	7.3000E+00	0.0000E+00	0.0000E+00	1.9530E+02	1.9590E+02
	0	0	0	0	0	0
46	0.0000E+00	1.5880E+00	0.0000E+00	0.0000E+00	1.5691E+02	1.6072E+02

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	0	0	0	0	0	0
50	1.3200E+00	1.4900E+00	0.0000E+00	0.0000E+00	1.6072E+02	1.7692E+02
	0	0	0	0	0	0
51	1.4900E+00	1.5880E+00	0.0000E+00	0.0000E+00	1.6072E+02	1.7692E+02
	5	5	0	0	0	0
52	0.0000E+00	1.3200E+00	0.0000E+00	0.0000E+00	1.6072E+02	1.7692E+02
	0	0	0	0	0	0
53	0.0000E+00	1.5880E+00	0.0000E+00	0.0000E+00	1.7692E+02	1.8739E+02
	0	0	0	0	0	0
54	1.5880E+00	6.1900E+00	0.0000E+00	0.0000E+00	1.5691E+02	1.8739E+02
	0	0	0	0	0	0
55	1.0000E+00	6.1900E+00	0.0000E+00	0.0000E+00	1.8739E+02	1.8779E+02
	0	0	0	0	7	7
56	0.0000E+00	1.0000E+00	0.0000E+00	0.0000E+00	1.8739E+02	1.8779E+02
	0	0	0	0	0	0
57	0.0000E+00	1.0000E+00	0.0000E+00	0.0000E+00	1.8779E+02	1.8950E+02
	0	0	0	0	0	0
58	1.0000E+00	6.1900E+00	0.0000E+00	0.0000E+00	1.8779E+02	1.8950E+02
	0	0	0	0	0	0

***** SUMMARY OF MATERIAL DATA *****

Material Number	Material Name	----- Thermal Parameters ----- -- Temperature-Dependent Function Numbers --			Phase Change
		Conductivity	Density	Specific Heat	
1	ssteel	1.000000E+00 -1	7.980000E-03 0	1.000000E+00 -7	No
2	lead	1.000000E+00 -2	1.076800E-02 0	1.000000E+00 -8	Yes
3	air	1.000000E+00 -3	1.000000E+00 -6	1.000000E+00 -9	No
4	al6061t6	1.000000E+00 -4	2.700000E-03 0	1.000000E+00 -10	No
5	csteel	1.000000E+00 -5	7.980000E-03 0	1.000000E+00 -11	No
7	alholder	1.000000E+00 -4	2.700000E-03 0	1.000000E+00 -10	No
9	al-axial	7.900000E-01 -4	2.100000E-03 0	1.000000E+00 -10	No

***** SUMMARY OF PHASE CHANGES *****

Phase Change	Material Number	Transition Temperature	Latent Heat
1	2	3.276000E+02	2.600000E+04

***** SUMMARY OF INITIAL TEMPERATURE DATA *****

Number	Initial Temperature	Position-Dependent Function Numbers		
		x or r	y or th	z or p
1	1.00000E+02	0	0	0

***** SUMMARY OF HEAT GENERATION RATE DATA *****

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Number	Power	Time-, Temperature-, and Position-Dependent Function Numbers
	Density	Time Temperature X or R Y or Theta Z or Phi
1	9.87200E-01	0 0 0 0 0

***** SUMMARY OF BOUNDARY CONDITION DATA *****

Number: 2 Type: Surface-to-Surface
Heat Transfer Coefficients and Any Functions Used to Define Dependence:
Radiation : 2.319000E-13

Number: 3 Type: Surface-to-Surface
Heat Transfer Coefficients and Any Functions Used to Define Dependence:
Radiation : 4.930430E-13

Number: 4 Type: Surface-to-Environment
Temperature and Any Functions Used to Define Dependence:
Temperature : 3.770000E+01
Heat Transfer Coefficients and Any Functions Used to Define Dependence:
Radiation : 3.515400E-12
Natural Convection : 1.022000E-04 (Multiplier)
Natural Convection : 3.330000E-01 (Exponent)

Number: 5 Type: Surface-to-Surface
Heat Transfer Coefficients and Any Functions Used to Define Dependence:
Radiation : 1.499000E-13

Number: 6 Type: Surface-to-Surface
Heat Transfer Coefficients and Any Functions Used to Define Dependence:
Radiation : 7.785000E-13

Number: 7 Type: Surface-to-Surface
Heat Transfer Coefficients and Any Functions Used to Define Dependence:
Radiation : 1.454000E-13

Number: 8 Type: Surface-to-Surface
Heat Transfer Coefficients and Any Functions Used to Define Dependence:
Radiation : 8.136000E-13

***** SUMMARY OF GRID STRUCTURE *****

X (or R) Gross Grid Lines and Number of Divisions

0.000000E+00	1.000000E+00	1.300000E+00	1.320000E+00	1.490000E+00
1.588000E+00	2.000000E+00	2.400000E+00	4.440000E+00	4.760000E+00
5.080000E+00	6.190000E+00	7.300000E+00	7.620000E+00	9.400000E+00
9.800000E+00	1.050000E+01	1.080000E+01	1.110000E+01	1.430000E+01
1.460000E+01	2.900000E+01	3.048000E+01		
2	2	1	1	1
2	2	1	3	2
1	5	2	4	1
1	5	1	3	2
12	2			

X (or R) Fine Grid Lines Generated by HEATING

1	0.00000E+00	2	5.00000E-01	3	1.00000E+00	4	1.15000E+00
---	-------------	---	-------------	---	-------------	---	-------------

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5	1.30000E+00	6	1.32000E+00	7	1.49000E+00	8	1.58800E+00
9	1.79400E+00	10	2.00000E+00	11	2.20000E+00	12	2.40000E+00
13	4.44000E+00	14	4.54667E+00	15	4.65333E+00	16	4.76000E+00
17	4.92000E+00	18	5.08000E+00	19	6.19000E+00	20	6.41200E+00
21	6.63400E+00	22	6.85600E+00	23	7.07800E+00	24	7.30000E+00
25	7.46000E+00	26	7.62000E+00	27	8.06500E+00	28	8.51000E+00
29	8.95500E+00	30	9.40000E+00	31	9.80000E+00	32	1.05000E+01
33	1.05600E+01	34	1.06200E+01	35	1.06800E+01	36	1.07400E+01
37	1.08000E+01	38	1.11000E+01	39	1.21667E+01	40	1.32333E+01
41	1.43000E+01	42	1.44500E+01	43	1.46000E+01	44	1.58000E+01
45	1.70000E+01	46	1.82000E+01	47	1.94000E+01	48	2.06000E+01
49	2.18000E+01	50	2.30000E+01	51	2.42000E+01	52	2.54000E+01
53	2.66000E+01	54	2.78000E+01	55	2.90000E+01	56	2.97400E+01
57	3.04800E+01						

Z (or Phi) Gross Grid Lines and Number of Divisions

0.000000E+00	2.540000E+00	3.500000E+00	6.000000E+00	8.900000E+00
2.360000E+01	2.640000E+01	2.700000E+01	2.830000E+01	5.310000E+01
1.569100E+02	1.607200E+02	1.769200E+02	1.873900E+02	1.877900E+02
1.895000E+02	1.953000E+02	1.959000E+02	2.010000E+02	2.016000E+02
2.024000E+02	2.169000E+02	2.219000E+02	2.241000E+02	2.251000E+02
2.264000E+02	2.314000E+02	2.344000E+02	2.350000E+02	
2	2	1	1	6
2	1	1	10	34
5	12	10	1	2
4	1	5	1	1
5	1	2	1	1
3	2	1		

Z (or Phi) Fine Grid Lines Generated by HEATING

1	0.00000E+00	2	1.27000E+00	3	2.54000E+00	4	3.02000E+00
5	3.50000E+00	6	6.00000E+00	7	8.90000E+00	8	1.13500E+01
9	1.38000E+01	10	1.62500E+01	11	1.87000E+01	12	2.11500E+01
13	2.36000E+01	14	2.50000E+01	15	2.64000E+01	16	2.70000E+01
17	2.83000E+01	18	3.07800E+01	19	3.32600E+01	20	3.57400E+01
21	3.82200E+01	22	4.07000E+01	23	4.31800E+01	24	4.56600E+01
25	4.81400E+01	26	5.06200E+01	27	5.31000E+01	28	5.61532E+01
29	5.92065E+01	30	6.22597E+01	31	6.53129E+01	32	6.83662E+01
33	7.14194E+01	34	7.44726E+01	35	7.75259E+01	36	8.05791E+01
37	8.36324E+01	38	8.66856E+01	39	8.97388E+01	40	9.27921E+01
41	9.58453E+01	42	9.88985E+01	43	1.01952E+02	44	1.05005E+02
45	1.08058E+02	46	1.11111E+02	47	1.14165E+02	48	1.17218E+02
49	1.20271E+02	50	1.23324E+02	51	1.26378E+02	52	1.29431E+02
53	1.32484E+02	54	1.35537E+02	55	1.38591E+02	56	1.41644E+02
57	1.44697E+02	58	1.47750E+02	59	1.50804E+02	60	1.53857E+02
61	1.56910E+02	62	1.57672E+02	63	1.58434E+02	64	1.59196E+02
65	1.59958E+02	66	1.60720E+02	67	1.62070E+02	68	1.63420E+02
69	1.64770E+02	70	1.66120E+02	71	1.67470E+02	72	1.68820E+02
73	1.70170E+02	74	1.71520E+02	75	1.72870E+02	76	1.74220E+02
77	1.75570E+02	78	1.76920E+02	79	1.77967E+02	80	1.79014E+02
81	1.80061E+02	82	1.81108E+02	83	1.82155E+02	84	1.83202E+02
85	1.84249E+02	86	1.85296E+02	87	1.86343E+02	88	1.87390E+02
89	1.87790E+02	90	1.88645E+02	91	1.89500E+02	92	1.90950E+02
93	1.92400E+02	94	1.93850E+02	95	1.95300E+02	96	1.95900E+02
97	1.96920E+02	98	1.97940E+02	99	1.98960E+02	100	1.99980E+02
101	2.01000E+02	102	2.01600E+02	103	2.02400E+02	104	2.05300E+02
105	2.08200E+02	106	2.11100E+02	107	2.14000E+02	108	2.16900E+02

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109	2.21900E+02	110	2.23000E+02	111	2.24100E+02	112	2.25100E+02
113	2.26400E+02	114	2.28067E+02	115	2.29733E+02	116	2.31400E+02
117	2.32900E+02	118	2.34400E+02	119	2.35000E+02		

***** LISTING OF TABULAR FUNCTIONS *****

Table number -	4	Number of pairs -	9
Argument	Value	(Min) <- Relative Value -> (Max)	
-2.23000000E+02	1.68550000E+00	*****	
2.00000000E+01	1.66860000E+00	*	
5.00000000E+01	1.69200000E+00	*****	
7.50000000E+01	1.70880000E+00	*****	
1.00000000E+02	1.72330000E+00	*****	
1.25000000E+02	1.73580000E+00	*****	
1.50000000E+02	1.74830000E+00	*****	
1.75000000E+02	1.75930000E+00	*****	
2.00000000E+02	1.76890000E+00	*****	

Table number -	10	Number of pairs -	6
Argument	Value	(Min) <- Relative Value -> (Max)	
-1.73150000E+02	4.82000000E+02	*	
-7.31500000E+01	7.98000000E+02	*****	
2.71500000E+01	9.03000000E+02	*****	
1.26850000E+02	9.49000000E+02	*****	
3.26850000E+02	1.03300000E+03	*****	
5.26850000E+02	1.14600000E+03	*****	

Table number -	3	Number of pairs -	34
Argument	Value	(Min) <- Relative Value -> (Max)	
-1.73150000E+02	9.24600000E-05	*	
-1.23150000E+02	1.37350000E-04	*	
-7.31500000E+01	1.80900000E-04	**	
-2.31500000E+01	2.22700000E-04	**	
2.68500000E+01	2.62400000E-04	***	
7.68500000E+01	3.00300000E-04	****	
1.26850000E+02	3.36500000E-04	****	
1.76850000E+02	3.70700000E-04	*****	
2.26850000E+02	4.03800000E-04	*****	
2.76850000E+02	4.36000000E-04	*****	
3.26850000E+02	4.65900000E-04	*****	
3.76850000E+02	4.95300000E-04	*****	
4.26850000E+02	5.23000000E-04	*****	
4.76850000E+02	5.50900000E-04	*****	
5.26850000E+02	5.77900000E-04	*****	
5.76850000E+02	6.02800000E-04	*****	
6.26850000E+02	6.27900000E-04	*****	
6.76850000E+02	6.52500000E-04	*****	
7.26850000E+02	6.75200000E-04	*****	
7.76850000E+02	7.32000000E-04	*****	
8.26850000E+02	7.82000000E-04	*****	
1.02685000E+03	8.37000000E-04	*****	
1.12685000E+03	8.91000000E-04	*****	
1.22685000E+03	9.46000000E-04	*****	

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1.32685000E+03	1.00000000E-03	*****
1.42685000E+03	1.05000000E-03	*****
1.52685000E+03	1.11000000E-03	*****
1.62685000E+03	1.17000000E-03	*****
1.72685000E+03	1.24000000E-03	*****
1.82685000E+03	1.31000000E-03	*****
1.92685000E+03	1.39000000E-03	*****
2.02685000E+03	1.49000000E-03	*****
2.12685000E+03	1.61000000E-03	*****
2.22685000E+03	1.75000000E-03	*****

Table number -	6	Number of pairs -	19
Argument	Value	(Min) <- Relative Value -> (Max)	
2.50000000E+01	1.18400000E-06	*****	
4.00000000E+01	1.12700000E-06	*****	
5.00000000E+01	1.09200000E-06	*****	
7.00000000E+01	1.02800000E-06	*****	
1.00000000E+02	9.45800000E-07	*****	
1.20000000E+02	8.97700000E-07	*****	
1.60000000E+02	8.14800000E-07	*****	
2.00000000E+02	7.45900000E-07	*****	
3.00000000E+02	6.15800000E-07	*****	
3.50000000E+02	5.66400000E-07	*****	
4.00000000E+02	5.24300000E-07	*****	
4.50000000E+02	4.88000000E-07	*****	
5.00000000E+02	4.56500000E-07	*****	
6.00000000E+02	4.04200000E-07	*****	
7.00000000E+02	3.62700000E-07	*****	
8.00000000E+02	3.28900000E-07	*****	
9.00000000E+02	3.00800000E-07	****	
1.00000000E+03	2.77200000E-07	***	
2.00000000E+03	1.55300000E-07	*	

Table number -	9	Number of pairs -	9
Argument	Value	(Min) <- Relative Value -> (Max)	
2.50000000E+01	1.00700000E+03	*****	
1.00000000E+02	1.00900000E+03	*****	
1.40000000E+02	1.01300000E+03	*****	
2.00000000E+02	1.02300000E+03	*****	
4.00000000E+02	1.06900000E+03	*****	
6.00111500E+02	8.00000000E+02	*****	
1.15300000E+03	1.00000000E+03	*****	
1.18400000E+03	2.00000000E+03	*****	
1.26400000E+03	0.00000000E+00	*	

Table number -	1	Number of pairs -	5
Argument	Value	(Min) <- Relative Value -> (Max)	
2.70000000E+01	1.42000000E-01	*	
1.26850000E+02	1.58000000E-01	****	
3.26850000E+02	1.89000000E-01	*****	
5.26850000E+02	2.19000000E-01	*****	
7.26850000E+02	2.47000000E-01	*****	

Table number -	7	Number of pairs -	5
----------------	---	-------------------	---

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Argument	Value	(Min) <- Relative Value -> (Max)
2.70000000E+01	4.80000000E+02	*
1.26850000E+02	5.13000000E+02	*****
3.26850000E+02	5.59000000E+02	*****
5.26850000E+02	5.85000000E+02	*****
7.26850000E+02	6.06000000E+02	*****

Table number - 2 Number of pairs - 23

Argument	Value	(Min) <- Relative Value -> (Max)
2.70000000E+01	4.58410100E-01	*****
5.00000000E+01	4.62672000E-01	*****
1.00000000E+02	4.71937000E-01	*****
1.50000000E+02	4.81202000E-01	*****
2.00000000E+02	4.90467000E-01	*****
2.50000000E+02	4.99732000E-01	*****
3.00000000E+02	5.08997000E-01	*****
3.20000000E+02	5.12703000E-01	*****
3.28000000E+02	1.56911800E-01	*
3.50000000E+02	1.59534200E-01	*
4.00000000E+02	1.65494200E-01	*
4.50000000E+02	1.71454200E-01	**
5.00000000E+02	1.77414200E-01	**
5.50000000E+02	1.83374200E-01	**
6.00000000E+02	1.89334200E-01	***
6.50000000E+02	1.95294200E-01	***
7.00000000E+02	2.01254200E-01	****
7.50000000E+02	2.07214200E-01	****
8.00000000E+02	2.13174200E-01	****
8.50000000E+02	2.19134200E-01	****
9.00000000E+02	2.25094200E-01	****
9.50000000E+02	2.31054200E-01	*****
1.00000000E+03	2.37014200E-01	*****

Table number - 8 Number of pairs - 5

Argument	Value	(Min) <- Relative Value -> (Max)
-1.73150000E+02	1.18000000E+02	*
-7.31500000E+01	1.25000000E+02	*****
2.70000000E+01	1.29000000E+02	*****
1.26850000E+02	1.32000000E+02	*****
3.26850000E+02	1.42000000E+02	*****

Table number - 5 Number of pairs - 4

Argument	Value	(Min) <- Relative Value -> (Max)
1.26850000E+02	5.67000000E-01	*****
3.26850000E+02	4.80000000E-01	*****
5.26850000E+02	3.92000000E-01	*****
7.26850000E+02	3.00000000E-01	*

Table number - 11 Number of pairs - 5

Argument	Value	(Min) <- Relative Value -> (Max)
2.71500000E+01	4.34000000E+02	*
1.26850000E+02	4.87000000E+02	**

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```

3.26850000E+02      5.59000000E+02      *****
5.26850000E+02      6.85000000E+02      *****
7.26850000E+02      1.16900000E+03      *****

```

```

Table number -      13      Number of pairs -      6

Argument      Value      (Min) <- Relative Value -> (Max)
0.00000000E+00      0.00000000E+00      *
1.59010000E+02      0.00000000E+00      *
1.59020000E+02      1.00000000E+00      *****
1.89500000E+02      1.00000000E+00      *****
1.89510000E+02      0.00000000E+00      *
3.00000000E+02      0.00000000E+00      *

```

***** SOURCES OF NON-LINEARITY IN THE MODEL *****

Radiation (in calculations 273.15 will be added to temperatures to
convert them to absolute)
Natural convection
Temperature dependent conductivity
Temperature dependent density or specific heat (transient calculations)

```

***WARNING*** Table      7 must be evaluated for 0.00000000E+00. The
***WARNING*** value of the function will be 4.80000000E+02 for all
***WARNING*** arguments less than 2.70000000E+01

```

```

***WARNING*** Table     11 must be evaluated for 0.00000000E+00. The
***WARNING*** value of the function will be 4.34000000E+02 for all
***WARNING*** arguments less than 2.71500000E+01

```

```

***WARNING*** Table      9 must be evaluated for 0.00000000E+00. The
***WARNING*** value of the function will be 1.00700000E+03 for all
***WARNING*** arguments less than 2.50000000E+01

```

```

***WARNING*** Table      6 must be evaluated for 0.00000000E+00. The
***WARNING*** value of the function will be 1.18400000E-06 for all
***WARNING*** arguments less than 2.50000000E+01

```

```

***WARNING*** Table      1 must be evaluated for 0.00000000E+00. The
***WARNING*** value of the function will be 1.42000000E-01 for all
***WARNING*** arguments less than 2.70000000E+01

```

```

***WARNING*** Table      5 must be evaluated for 0.00000000E+00. The
***WARNING*** value of the function will be 5.67000000E-01 for all
***WARNING*** arguments less than 1.26850000E+02

```

```

***WARNING*** Table      2 must be evaluated for 0.00000000E+00. The
***WARNING*** value of the function will be 4.58410100E-01 for all
***WARNING*** arguments less than 2.70000000E+01

```

***** SUMMARY OF AREAS AND VOLUMES *****

These values are for model checking only and are not used in
any calculations. Some roundoff errors may occur in cases
with a large number of small nodes. User-defined node-to-node

GENERAL DESIGN AND COMPUTATION SHEET

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connectors may cause incorrect surface areas to be displayed.
To check areas, run the case without node-to-node connectors.

Boundary Condition	Surface Area
2	1.267520E+03
3	3.664354E+02
4	4.940967E+04
5	1.516635E+02
6	3.958407E+01
7	1.172320E+02
8	3.644247E+01

Material	Volume
1	1.682226E+04
2	5.416180E+05
3	2.786364E+04
4	5.449590E+01
5	6.544340E+04
7	3.634611E+03
9	0.000000E+00

Heat Gen.	Volume
1	2.431197E+01

***** NUMBER OF PARAMETERS SPECIFIED BY THE INPUT DATA *****

Regions	54
Materials	7
Phase changes	1
Initial temperatures	1
Heat generations	1
Boundary conditions	7
Gross grid lines along x or r axis	23
Fine grid lines along x or r axis	57
Gross grid lines along y or theta axis	1
Fine grid lines along y or theta axis	1
Gross grid lines along z or phi axis	29
Fine grid lines along z or phi axis	119
Analytic functions	0
Tabular functions	12
Node-to-node connectors	0
Transient printout times	0
Nodes for monitoring of temperatures	0
Number of nodes	6625
Number of specified-temperature nodes	0
Position-dependent boundary temperature nodes	0
Bandwidth for direct solution	387

***** INITIAL CONDITIONS *****

Number of iterations completed	=	0
Current problem time	=	0.00000000E+00
Elapsed wallclock time (h:m:s)	=	00:00:00.21

GENERAL DESIGN AND COMPUTATION SHEET

PROJECT TITLE

Safety Analysis for Transport of HFIR Targets

ACCOUNT NO.

CALCULATION TITLE:

Loop Cask Thermal Analysis for Mo99 Annular HFIR
Targets Page 36 of 37

DAC Number:

NNFD-3525-DAC-2013-001-R0

Revision:

0

Minimum Temperature = 0.00000E+00 at node 1
Maximum Temperature = 1.00000E+02 at node 853

MATERIAL TEMPERATURES

Number	Minimum	Average	Maximum
1	0.00000E+00	1.87665E+01	1.00000E+02
2	0.00000E+00	7.69081E+01	1.00000E+02
3	0.00000E+00	4.06411E+00	9.99960E+01
4	0.00000E+00	0.00000E+00	0.00000E+00
5	0.00000E+00	7.10973E+00	3.94111E+01
7	0.00000E+00	0.00000E+00	0.00000E+00

HEAT GENERATION

Number	Current Rate (energy/time) (Modeled)	(Neglected)
1	2.40008E+01	0.00000E+00

BOUNDARY HEAT FLOW

Number	Environment Temperature	Current Rate (energy/time) (Modeled)	(Neglected)
2	-----	0.00000E+00	0.00000E+00
3	-----	0.00000E+00	0.00000E+00
4	3.77000E+01	1.21033E+03	0.00000E+00
5	-----	0.00000E+00	0.00000E+00
6	-----	0.00000E+00	0.00000E+00
7	-----	0.00000E+00	0.00000E+00
8	-----	0.00000E+00	0.00000E+00
Sum		1.21033E+03	0.00000E+00

BEGIN STEADY STATE CALCULATION - DIRECT SOLUTION TECHNIQUE

Maximum number of iterations = 20000
Convergence criterion = 1.0000000E-05

***** STEADY-STATE SOLUTION OUTPUT *****

Number of iterations completed = 5
Current problem time = 0.00000000E+00
Elapsed wallclock time (h:m:s) = 00:00:01.10

Minimum Temperature = 3.82406E+01 at node 43
Maximum Temperature = 1.21993E+02 at node 4310

MATERIAL TEMPERATURES

Number	Minimum	Average	Maximum
1	3.82406E+01	3.85564E+01	6.78714E+01
2	3.82491E+01	3.86545E+01	3.92392E+01

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3	3.82507E+01	4.17580E+01	1.21993E+02
4	9.24670E+01	1.02005E+02	1.21993E+02
5	3.82480E+01	3.86345E+01	3.90180E+01
7	6.78131E+01	9.06096E+01	9.27622E+01

HEAT GENERATION			
Number		Current Rate (energy/time)	
		(Modeled)	(Neglected)
1		2.40008E+01	0.00000E+00

BOUNDARY HEAT FLOW			
Number	Environment Temperature	Current Rate (energy/time)	
		(Modeled)	(Neglected)
2	-----	0.00000E+00	0.00000E+00
3	-----	0.00000E+00	0.00000E+00
4	3.77000E+01	-2.40006E+01	0.00000E+00
5	-----	0.00000E+00	0.00000E+00
6	-----	0.00000E+00	0.00000E+00
7	-----	0.00000E+00	0.00000E+00
8	-----	0.00000E+00	0.00000E+00
---		-----	-----
Sum		-2.40006E+01	0.00000E+00

***** END OF HEATING EXECUTION *****

cask

***** Number of warnings -- 7
***** Number of errors -- 0

C-3 RSS FOR CHEMICAL PROCESSING OF MO-99 TARGETS

Mo-99 Electrochemical Dissolution Experiment Researcher Operational Procedure, Rev. 0 Extraction of Metal Foil from Cylindrical Target

Supplies

Target Cutting Device

Cylindrical Target (1.04" outer diameter, 6.5" length)

Open-top liquid nitrogen flask (1 liter capacity, deep enough to hold 1 target)

Liquid nitrogen

Long tongs or tweezers

Procedure

Remove target ends:

1. Place the target in the target cutting device. Align one end of target to overhang the circumferential cutting element (right side of device in the figures) by approximately 3/16" to 1/4".
2. Cut off end of target by turning the crank and adjusting the depth of the cutter.
3. Repeat Steps 1 and 2 to remove the other end of the target.
4. Discard the cut pieces of the target.

Cut the target lengthwise:

5. Place the target in the target cutting device, and align the lengthwise score mark on the target with the lengthwise cutting blade (top of the device).
6. Cut the target lengthwise along the score mark.
 - a. Do not penetrate all of the way through the target. Use the depth gauge to determine the cutting depth, and stop cutting when the bottom of the prongs on the depth gauge are no longer visible when inserted into the cut.

Foil Removal:

7. Ideally, the outer cylinder will spring open and away from the inner cylinder once the longitudinal cut is made. Mechanically separate the inner cylinder from the outer cylinder to free the uranium foil sandwiched in between.

What to do if the foil sticks to the inner tube, the outer tube, or both tubes, and cannot be easily separated:

8. Place the tube in the liquid nitrogen dewar to freeze the tube. Then remove the tube from the dewar and allow it to warm to room temperature.
9. Attempt to remove the foil from the tube using light mechanical force. Be careful not to tear or break the foil, as this will make it more difficult to handle and dissolve in future processing steps.
 - a. If the foil fell off of the tube when the tube was in the dewar, remove the foil from the dewar, and the separation task is complete.

10. If the foil cannot be removed from the tube, repeat the freeze/thaw cycle and try removing it again.
11. If the foil still can't be removed from the tube, then ask the principal investigator for further instructions. Options include:
 - a. Repeating the freeze/thaw cycle, and attempt foil removal again by the same method.
 - b. Attempt to remove the foil by using stronger force and/or alternate tooling.
 - c. Stop the experiment.

Approval of Researcher Instructional Set – Extraction of Metal Foil

Name, Principal Investigator

Date

Name, 3525 Facility Manager

Date

**Mo-99 Electrochemical Dissolution Experiment
Researcher Operational Procedure, Rev. 2
Uranium Foil Processing**

Figure 1. As-built Electrochemical Dissolution Equipment Schematic (two feed bottles shown).

Supplies

- 1.3 L NaOH solution (1M, reagent grade) in graduated 2 L Erlenmeyer flask with threaded cap
- 1.3 L NaHCO₃ solution (1M, ACS grade, prepared from distilled water and bicarbonate powder) in graduated 2 L Erlenmeyer flask with threaded cap
- 500 ml NaHCO₃ solution (1M, ACS grade, prepared from distilled water and bicarbonate powder) in graduated 2 L Erlenmeyer flask with threaded cap
- 100 g CaO powder (pre-weighed, reagent grade) in closed sample container.
- Pressurized nitrogen tank

Pre-Operation Checks

- Confirm that the downstream HEPA filter (K-15-12.2 and K-15-12-4) in each of the online hot cell ventilation system HEPA banks has been replaced with a high efficiency gas absorber filter containing activated charcoal.

Initials Date

- Confirm that there is documentation available on the HEPA filter that states that the decontamination factor for elemental radioiodine is at least 100.

Initials Date

- Confirm that the activated carbon trap is installed in-line with the vacuum pump.

Initials Date

- Confirm that the activated carbon in the in-line trap is identified with a label, that the label matches the supporting documentation on the activated carbon, and that the documentation states that the activated carbon in the in-line trap has an elemental radioiodine decontamination factor of at least 1000.

Initials Date

- Confirm that a catch pan has been placed beneath the electrochemical platform to capture spills.

Initials Date

- Confirm that liquids may only be moved from one vessel to the next by deploying the vacuum pump (i.e., no pumping of liquid will occur by pressurizing a tank or vessel above atmospheric pressure and opening an outlet flow path).

Initials Date

- Confirm that the necessary chemical reagents have been placed in the cell prior to starting the experimental work.

Initials Date

- Confirm that the nitrogen tank is installed, the tank has nitrogen in it, and the nitrogen line to the apparatus is operational.

Initials

Date

- Confirm that the electrically powered devices for the experiment are operational.

Initials

Date

Operation of the Electrochemical Dissolver Platform

Load the foil into the dissolver

1. Flatten irradiated foil using the manipulators, and place it in the cage. Attempt to flatten the foil as much as practical to ensure good electrical contact with the cage grid. Then, close the cage and tighten the wing nut on the top of the cage to lock the cage.
2. Place basket into the dissolution tank, T-1. Seal the tank using the red lever.

Dissolve the outer aluminum layer

3. Turn on the vacuum pump.
4. Perform dissolution vessel excessive leak check: Open V-2 and look at the pressure gauge on the vacuum header. Does the gauge show a vacuum greater than 1 in Hg? If not, then do the following:
 - a. Close V-2.
 - b. Turn off the vacuum pump.
 - c. Unseal the tank by lifting the red lever.
 - d. Reseat the basket in the tank.
 - e. Lock down the red lever.
 - f. Repeat the vessel leak check.
 - g. If the leak check fails again, increase the draw of the vacuum pump until the vacuum gauge reads greater than 1 in Hg.
 - h. If adjustment of the vacuum draw fails to correct the problem at this point, then there is a large air leak somewhere in the flow path. Stop the experiment, and find and fix the leak before proceeding with the experiment.
5. Activate the mixer motor for the electrochemical dissolver tank, T-1, and adjust the motor control to set it at full speed.
6. Connect M-1 (quick disconnect) to the NaOH feed bottle. Have a second person confirm that the correct feed bottle is attached.

Operator, initials

Observer, initials

Date

7. Open valve V-2 on the vacuum header to pull the liquid from the NaOH feed bottle into the electrochemical dissolution vessel T-1.
8. Close valve V-2 when the feed bottle is empty. An empty feed bottle will be indicated by lack of liquid in the translucent feed line and an increase in the flow rate of gas through the rotameter on the vacuum header.
9. Disconnect M-1 quick disconnect from the NaOH feed bottle. Note that the absolute pressure in the vacuum header will decrease (vacuum gauge pressure will increase), and gas flow through the vacuum header will also decrease (rotameter indicator will show a lower flow rate).
10. Connect M-2 quick disconnect to the electrochemical dissolver – this is the nitrogen feed. Adjust the vacuum pump controller to set the flow rate through the vacuum header at 20-25 scfh.
11. Plug in heat gun and monitor the temperature of the electrochemical dissolver using the external temperature display. Unplug the heat gun when the thermocouples read 60°C or greater.

During Dissolution of Outer Aluminum Layer

- NOTE: The vacuum pump is expected to be operating at all times during the aluminum dissolution step to help dissipate hydrogen and capture radioiodine. If the vacuum pump stops operating during the dissolution, then turn off the flow of nitrogen gas to the dissolution vessel and turn off the heat gun if it is on. Leave the gas flow paths open. When the problem with the vacuum pump is resolved, re-activate the vacuum pump first, and then restore the flow of nitrogen to the tank. If the vessel was being heated at the time
12. Allow 20 minutes to pass after turning off the heat gun.
 13. Close V-2 on the vacuum header.
 14. Open V-3 on the vacuum header.
 15. Open V-11 to drain the liquid from the electrochemical dissolver into the waste collection tank, T-3.
 16. Close V-3 when electrochemical dissolver has been emptied. This is indicated by lack of flow in the drainage line, increase in flow rate in the flow meter on the vacuum header.
 17. Close V-11.

Flush out residual NaOH using NaHCO₃ solution

18. Connect M-1 quick disconnect to the 2 L flask containing 500 ml NaHCO₃ solution. Have a second person confirm that the correct feed bottle is attached.

Operator, initials

Observer, initials

Date

19. Open V-2. This will reduce the pressure in the electrochemical dissolver and cause the contents of the flask to flow into the dissolver.

20. Close V-2 when the flask has been fully drained. This is indicated by lack of flow in the line, and by an increase in the flow rate of gas through the vacuum header.
21. Disconnect M-1 from the flask.
22. Connect M-4 quick disconnect to F-5 on the mixing tank (T-3).
23. Open V-11.
24. Open V-4. Liquid will move from the electrochemical dissolver into the mixing tank.
25. Close V-4 when liquid flow has stopped. This is indicated by a lack of flow in the line, and by an increase in the flow rate of gas through the vacuum header.
26. Connect F-7 quick disconnect fitting to T-2 (liquid waste collection tank).
27. Open V-3 on the vacuum header.
28. Open V-6 on the mixing tank outlet (T-3). This will cause the liquid to drain from the mixing tank (T-3) into the waste collection tank (T-2).
29. Close V-6 when the all of the liquid has been drained from the mixing tank into the waste collection tank. This is indicated by no flow in the line, and an increase in the gas flow rate through the vacuum header.
30. Close V-3 on the vacuum header.
31. Close V-11 (inlet to the liquid feed port for the dissolver, T-1).

Dissolve the foil

32. Connect the electrodes to the electrochemical dissolver (positive electrode connects to the basket, negative electrode connects to the body). Cover the terminals with the temporary insulating cover.
33. Connect M-1 quick disconnect to the flask containing 1.3 L NaHCO_3 solution. Have a second person confirm that the correct feed bottle is attached.

Operator, initials

Observer, initials

Date

34. Open V-2.
35. When liquid transfer is complete, disconnect M-1 from the feed flask. This is indicated by an empty flask, lack of liquid flow, and an increase in the gas flow rate through the vacuum header.
36. Activate the DC power supply. Set the power supply at 10 V.
37. Turn on the heat gun, and heat the electrochemical dissolver to about 80°C, then turn off the heat gun.

During Electrochemical Dissolution

- If the temperature of the electrochemical dissolver falls below 75°C, turn on the heat gun, and heat the dissolution vessel until it reaches 80°C.
- If the temperature of the electrochemical dissolver rises above 85°C, turn on the heat gun in “air” mode to cool the vessel. Cool the vessel until the temperature falls to 80°C.
- If the vacuum pump stops operating during the dissolution, turn off the DC power supply and turn off the flow of nitrogen gas to the electrochemical dissolution vessel. Leave the gas flow paths open. When the vacuum pump is restored to operation, first turn back on the flow of nitrogen, and then reactivate the DC power supply.
- If the flow of gas through the vacuum header falls below 20 scfm, then turn off the DC power supply. After the problem is resolved and the proper flow rate is restored, the DC power supply can be reactivated.
 - First try increasing the vacuum draw.
 - If increasing the vacuum draw fails to increase the flow rate or if the vacuum draw is at maximum, try slightly increasing the flow of nitrogen into the vessel and watch for a response in the vacuum header flow meter.
 - If neither of these responses fixes the problem, maintain the hold point and consult with the principal investigator for alternative approaches.

After Electrochemical Dissolution

38. After about 2 hours, turn off the DC power supply to stop the dissolution. Activate the heat gun in “air” mode to cool the vessel. Turn off the heat gun when the temperature of the vessel has cooled below 35°C.
39. Connect F-7 to T-4 (product tank).
40. Connect M-4 to T-3 (mixing tank).
41. Close V-2.
42. Activate the mixer motor for T-3 (mixing tank).
43. Open V-11.
44. Open V-4 on the vacuum header to move the liquid from the electrochemical dissolver into the mixing tank (T-3).
45. Close V-4 when all of the liquid has been drained from the electrochemical dissolver. This is indicated by lack of flow in the liquid line and an increase in the gas flow rate through the vacuum header.
46. Select the CaO bottle. Have a second person confirm that the correct chemical bottle has been selected.

Operator, initials

Observer, initials

Date

47. Load the CaO powder into the feed funnel for the mixing tank. Then, open the valve funnel to drop the CaO into the mixing vessel.
48. After 10 minutes of stirring, open V-5 on the vacuum header.
49. Open V-6 to transport the liquid from the mixing tank (T-3) into the product tank (T-4). The filter in between the two vessels will capture the solids in the line, and the liquid will pass on through to the product tank.
50. Close V-5 when all of the liquid has been transported to the product tank. This is indicated by lack of liquid flow, and by an increase in the flow rate of gas through the vacuum header.
51. Turn off the vacuum pump.
52. Disconnect the nitrogen supply line, M-2, from the electrochemical dissolver, and close the nitrogen regulator.
53. Close V-7 and V-8 on either side of the line filter.

Sample Collection and Analysis

54. Remove in-line filter by disconnecting the lines outside of V-7 and V-8. Bag it out of the hot cell and scan it using gamma spectroscopy.
55. Drain the waste tank (T-2) into a waste liquid container, and obtain a sample of the material (1 ml) using a pipette. Scan the sample using gamma spectroscopy.
56. Drain the product tank (T-4) into another waste liquid container, and obtain a sample of the material (1 ml) using a pipette. Scan the sample using gamma spectroscopy.

Approval of Researcher Instructional Set – Dissolution of Metal Foil

Name, Principal Investigator

Date

Name, 3525 Facility Manager

Date

C-4 RHACS FOR MO-99 ELECTROCHEMICAL DISSOLUTION EXPERIMENTS

RSS No	11716.0																	
Status	Inactive																	
Minor Revisions	None.																	
Last Modified by	Maples, Curtis A (24087) on 10/14/2014 3:40:55 PM																	
Notice	This Research Safety Summary is not currently valid , only <i>Authorized</i> Research Safety Summaries are considered to be complete/valid.																	
Title	Chemical Processing of Mo-99 Targets																	
Approvals/Authorizations	<table><thead><tr><th>Date/Time</th><th>Name</th><th>Role</th></tr></thead><tbody><tr><td>11/21/2013 8:09:14 AM</td><td>Busby, Jeremy T (742645)</td><td>Group Leader</td></tr><tr><td>11/21/2013 8:14:26 AM</td><td>Lewis Jr, Benjamin E (16548)</td><td>Group Leader</td></tr><tr><td>11/22/2013 9:48:11 AM</td><td>Caquelin, Dale A (28652)</td><td>Group Leader</td></tr><tr><td>11/22/2013 1:09:06 PM</td><td>Icenhour, Alan S (31487)</td><td>Division Work Authority</td></tr></tbody></table>	Date/Time	Name	Role	11/21/2013 8:09:14 AM	Busby, Jeremy T (742645)	Group Leader	11/21/2013 8:14:26 AM	Lewis Jr, Benjamin E (16548)	Group Leader	11/22/2013 9:48:11 AM	Caquelin, Dale A (28652)	Group Leader	11/22/2013 1:09:06 PM	Icenhour, Alan S (31487)	Division Work Authority		
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Description Text	<p>This RSS will be utilized to perform experiments related to recovery of Molybdenum-99 (Mo-99) from irradiated targets. This work will be performed in the Building 3525 hot cells. This RSS applies only to the work performed in the east hot cells, and does not include work associated with out-of-cell testing of the laboratory equipment, or to initial installation of the equipment into the hot cells, as these tasks will be covered under other procedures.</p> <p>The following work tasks will be carried out under this RSS.</p> <ul style="list-style-type: none">• Facility and operations personnel will do all of the hands-on work for this experiment. However, researchers are expected to provide technical direction.• The reactor target used in this experiment is a unique design. It is made of an inner aluminum cylinder, a thin uranium metal foil, and an outer aluminum cylinder. The target is formed by wrapping the uranium foil around the outside of the inner cylinder, sliding the inner cylinder and foil inside the outer cylinder, and pressing the inner cylinder against the outer cylinder by pulling or pushing a die through the hollow center of the target (i.e., swaging). The ends of the target are then welded closed. The uranium metal foil weighs approximately 17 grams, and for this experiment, is enriched to between 7 and 7.5% U-235. The target will be irradiated for one cycle in HFIR prior to processing in 3525.• The irradiated target is placed into a custom tube cutting device, and the target is cut so that the uranium foil can be separated from the inner and outer aluminum cylinders. The device is used to cut the target. First, the welded target ends are cut around the circumference of each end of the target. Then, the target is cut lengthwise at a marked radial position. The lengthwise cut allows the outer cylinder to spring open and away from the inner cylinder and allows the two cylinders to be separated. Once the outer and inner cylinders are separated, the uranium foil may be freed from the target. The cladding materials are discarded, and the uranium foil is used for the remainder of the experiment.• The uranium foil is loaded into a metal basket, and the metal basket is loaded into an electrochemical dissolution vessel.• A strong sodium hydroxide (NaOH) solution is added to the																	

electrochemical dissolution vessel. The sodium hydroxide solution reacts with and dissolves the thin aluminum surface layer on the uranium foil. The uranium foil is unaffected by the sodium hydroxide.

- The NaOH solution is drained from the vessel, and the vessel is loaded with a sodium carbonate solution.
- The DC power supply to the electrochemical dissolution vessel is activated, and the uranium foil is dissolved electrochemically into solution. Dissolution of the uranium foil releases Mo-99 into solution.
- Once the uranium foil is completely dissolved, the solution is transferred to another vessel called the mixing vessel.
- The sodium carbonate solution is neutralized with nitric acid while being mixed in the mixing vessel. This causes the dissolved uranium and fission products to precipitate out of solution, while Mo-99 remains in solution.
- The mixture is filtered to separate the solids from the liquid, and the liquid is recovered as the product of the experiment.
- Samples of liquids and solids will be recovered at various points in the process, and the samples will be analyzed in an analytical laboratory by alpha and/or gamma spectroscopy to track the movement of Mo-99 throughout the experiment.
- The experimental vessels will be operated at sub-atmospheric pressure, and exhaust gases will be filtered through activated carbon absorption columns and activated carbon filters to capture radioactive iodine. Radioactive iodine is a by-product of the target irradiation process, and radioactive iodine released from the target during the experiment must be captured to the extent possible to limit its migration to the outside atmosphere through the facility exhaust stack.

Once the experimental work is concluded, waste liquids will be disposed, and the hardware will be dismantled.

These activities will be conducted as permitted by the Facility Safety Basis documentation and the Facility Use Agreement, and as determined within the scope of work authorization documentation as determined by the NSITD Research Operations Manager and the NNFD Facility Manager or their designated alternate(s). Additional detail regarding description of work, equipment and work scope limitations beyond those described in this document can be found in the Facility Safety Basis documentation.

A draft researcher procedure is attached to this RSS. This RSS is provided for informational purposes only, and should not be used by operators to perform the actual experiment. A final researcher instruction set will be provided to the operators prior to performing the actual experiment.

Description File	Researcher Operating Instructions, Rev 4..docx
Division	X130: Nuclear Security & Isotope Technology
Start Date	6/28/2013
End Date	3/31/2014
Account	ZZAC9239

General Notes	<p>The experiment will be performed in a hot cell. The hot cell walls will protect the worker against exposure to radiation, radioactive contamination, and chemical hazards while the experiment is underway.</p> <p>Worker exposure to chemical or radiation hazards may occur when samples are removed from the hot cell and transported to Analytical Chemistry for analysis. Appropriate PPE should be worn when handling samples, as defined in the Radiological Work Permit (RWP) for this task.</p> <p><u>Safety Glasses</u></p> <ul style="list-style-type: none"> Safety glasses will be worn when handling samples outside of the hot cell environment. <p><u>Standard Operating Guidelines</u></p> <ul style="list-style-type: none"> Specific procedures for operation of the equipment, and performance of the experiment, will be developed for this experiment. Use of equipment is limited to authorized personnel. <p><u>Radiological Work</u></p> <ul style="list-style-type: none"> Work in Radiological Areas requires an RWP. Notify facility RCTs prior to performing radiological work. <p><u>Integrated Safety Management</u></p> <ul style="list-style-type: none"> Use the Integrated Safety Management (ISM) approach when developing the specific work procedures for this experiment. Keep chemical containers and equipment properly labeled. Line Management is responsible for ensuring that personnel use the designated PPE, required for each task. These requirements may be passed down to staff by the use of such documentation as pre-job briefings, RWPs, special Work Plans, MSDS Sheets and operating procedures or guidelines.
General Attachments	None.
Point of Contact	Maples, Curtis A (24087)
Division Work Authority	Parks, Cecil V (19993)
Principal Investigator	Sherman, Steven R (922849);
PI Delegates	Sherman, Steven R (922849);
Group Leaders	Busby, Jeremy T (742645); Caquelin, Dale A (28652); Lewis Jr, Benjamin E (16548);
Participants	Caquelin, Dale A (28652); Chemical Operator, Group (113); Sherman, Steven R (922849); Woody, Bryan L (947945);
Required Reviewers	Baldwin, Mark E (19003) - ES&H; Drake, David D (21091) - ES&H; Dunsmore, Mary Ruth (676970) - ES&H; Maples, Curtis A (24087) - ES&H; Morris, Robert Noel (26219) - Lab Space

	<p><i>Manager; Murphy, Ann M (30519); Owen, Bruce Allen (22949) - ES&H; Perkins, Dale E (34225) - Rad Protection; Platfoot, John H (32377); Vrooman, Steve (707113) - Rad Protection;</i></p>
Optional Reviewers	<p>Busby, Jeremy T (742645) - GL; Caquelin, Dale A (28652) - CFM; Lewis Jr, Benjamin E (16548) - GL; Lewis-Lambert, Sandra (32020) - DTO;</p>
Lab Space Managers	<p>None.</p>
Locations	<p>🚧 Building 3525, Room 130; 🚧 Building 3525, Room 131; 🚧 Building 3525, Room EASTCELL</p>
Nepa Documentation	<p>Work in an authorized lab is covered under NEPA Categorical Exclusion Determination for Indoor, Small- and Pilot-Scale Research and Development (3767X), and specifically, B3.6 - Small-scale research and development, laboratory operations, and pilot projects.</p>
Hazards	<p>2.1 This operation involves the generation, handling, processing, use, or storage of radioactive materials (inc</p> <p>Potential Training: Please refer to the SBMS Radiation Safety Training exhibit for training requirements. Source Control Custodian - Radioactive Source users shall receive source-specific training provided by the Source Custodian.</p> <p>Requirements: Facility Hazard Categorization Facility Use Agreements Radiological Area Controls Radiological Design Requirements Radiological Dosimetry Radiological Work Safety and Security Regulatory (SSR) Program Sealed Radioactive Source Control</p> <p>Hazard Notes:</p> <ul style="list-style-type: none"> • Hot Cells and some adjacent areas contain radioactive materials. • Storage Wells in the Charging Area may contain radioactive materials. • Manipulations of radioactive materials and samples will be performed in the exposure to radiation, radioactive contamination, and chemical exposure. <p>Control Notes:</p> <ul style="list-style-type: none"> • No nuclear or radiological material may be transferred into the facility or have inventory checked by the NNFD Facility Manager or his designated alternate • Staff must notify the NNFD Facility Manager or his designated alternate prior • The Facility Manager is responsible for ensuring requirements of the FSB and • Work will be done in accordance with established Radiological Work Permits required. • Rad waste will be disposed of in accordance with applicable NNFD and ORNL <p>Locations: Building 3525, Room 130 Building 3525, Room 131 Building 3525, Room EASTCELL</p> <p>Attachments:</p>

None.

2.3 This operation will include the use of enriched uranium, uranium-233, plutonium, neptunium, americium

Potential Training:

See [Meeting NCS Training Requirements](#).

Requirements:

[Facility Hazard Categorization](#)

[Facility Use Agreements](#)

[Nuclear Criticality Safety](#)

[Obtain, Implement, and Comply with Nuclear Criticality Safety \(NCS\) Approvals \(NCSAs\)](#)

[Safety and Security Regulatory \(SSR\) Program](#)

Hazard Notes:

- Enriched uranium, transuranics, and other fission products will be present when the

Control Notes:

- Prior to transferring nuclear material into the IFEL, the proposed transfer must follow the established procedures.
- Before transferring fissionable material into the IFEL, staff must check with a supervisor in accordance with ORNL NMC&A procedures prior to initiating the transfer.
- Staff who work with fissionable materials or have other responsibilities for controlling access to Laboratory-level NCS Fundamentals training module ("ORNL NCS Fundamentals") must complete the training to which they are assigned.
- NCSA Facility Specific, NCS Fundamentals, and 3525 Building Access training

Locations:

Building 3525, Room 130

Building 3525, Room 131

Building 3525, Room EASTCELL

Attachments:

None.

2.4 This operation involves work conducted in a [Category 1, 2, or 3 Nuclear Facility](#).

Potential Training:

Facility-Specific Training is required.

Requirements:

[Facility Hazard Categorization](#)

[Facility Use Agreements](#)

[Safety and Security Regulatory \(SSR\) Program](#)

[Safety Basis](#)

[Unreviewed Safety Question \(USQ\) Process for Nuclear and Facility Safety](#)

Hazard Notes:

- Building 3525 is a Cat 2 Nuclear Facility per DOE Standard 1027-92, as amended.
- Work is performed on radiological and irradiated materials.

Control Notes:

- All work must be conducted in compliance with the Facility Safety Basis document
- The Unreviewed Safety Question Determination (USQD) process is the means by which changes to the documents. This not only applies to new work, or proposed physical changes to the Operating Procedures, or Standard Operating Guidelines, all of which must be evaluated

Question (USQ) and would require DOE approval prior to implementation. If in doubt, consult the Safety Documentation SME, or the IFEL Facility Manager.

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

Approved SBS and USQD as Built.pdf
USQD SCR 3525 - RSS 11716.0.pdf

5.1 This operation involves work on or near equipment with unguarded, energized electrical components open.

Potential Training:

DISCONTINUED CERT: Electrical Worker Safety Trng for R & D
DISCONTINUED CERT: High Voltage Safety Awareness Training

Requirements:

Electrical Safety
Lockout/Tagout

Hazard Notes:

- The power supply that will be used with this work is an XR32-62 Series II DC power supply at 62 amps, and a total power output of 2 kW. Under normal operating conditions, the power supply provides power to the electrochemical dissolver through the DC power supply.
- The DC power supply provides power to the electrochemical dissolver through the DC power supply.

Control Notes:

- The power supply cannot exceed 32 VDC due to its design.
- The power supply is equipped with circuits that sense when a short circuit has occurred and eliminate the power supply.
- Workers operating the electrochemical dissolver shall have the appropriate level of training and experience.
- The combined electrochemical dissolver/DC power supply system will be inspected and tested. If necessary, the system will be modified (e.g., install covers on the power supply) to align with ORNL practices for electrical equipment prior to performing work.

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

5.3 This operation involves one or more of the following:

- Electrical equipment or components labeled and listed by a Nationally Recognized Testing Laboratory
- Labeled and listed electrical equipment used for something other than its listed purpose;
- Electrical equipment that is labeled by a testing laboratory that is not nationally recognized.

Potential Training:

Electrical Safety Outside the Office

Requirements:

Electrical Safety

Hazard Notes:

- The electrochemical dissolver is not "UL" listed.

Control Notes:

- Request a review and approval from the Authority Having Jurisdiction (AHJ) equipment. If major modifications are made to the Inspection/Examination equipment are resumed.
- Verify approval prior to energizing equipment for the first time or after major

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

7.1 This operation involves work conducted under the **OSHA Laboratory Standard**.

Potential Training:

General Hazard Communication
ORNL Chemical Hygiene Plan
ORNL Hazard Communication Job Specific Training
OSHA Lab Standard Training - Includes site-specific

Requirements:**Chemical Safety****Hazard Notes:**

- Chemical manipulations and equipment utilized meet the OSHA definition of must be conducted in compliance with the ORNL SBMS Chemical Hygiene Safety Requirements (TSR) documentation. Elements of the Chemical Hygiene designated work locations are within HMMIS Control Area XU062.
- Specific chemical hazards include the use of aqueous nitric acid, aqueous sodium

Control Notes:

- Work with chemicals is conducted under the OSHA Laboratory Standard and
- Always review MSDSs before handling or using chemicals.
- The Facility SAR requires that toxic chemical inventories must be maintained
- If you are unsure of the RQ values for a given chemical, consult with your direct Manager as needed to determine the appropriate work control requirements facility.
- Prior to exceeding an RQ for a chemical, a USQD must be completed and approved

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

7.7 This operation involves chemicals or wastes that are flammable or combustible.

Requirements:

Chemical Safety

Exposure Assessments

Personal Protective Equipment

Hazard Notes:

- During the electrochemical dissolution step, flammable hydrogen gas is generated.

Control Notes:

- Physically, the amount of hydrogen generated is bounded. During dissolution, the amount of hydrogen generated will be less than 0.2 L by volume, or less than 0.02 g of hydrogen. If the foil is dissolved, the amount of hydrogen generated will be about 8 L of hydrogen by volume over a 10-minute period at constant current.
- The rate of hydrogen generation during the uranium foil dissolution step is controlled by the current. When the current generation of hydrogen stops.
- A flow of nitrogen gas will be maintained to the dissolution vessel during electrochemical dissolution. The potential of the hydrogen gas will be suppressed.
- A vacuum pump will be used to draw gas out of the headspace of the dissolution vessel. This will dilute the hydrogen gas in the headspace, and drive the concentration of hydrogen down. Preliminary calculations show that maintaining a volumetric flow rate of 5 liters/minute during dissolution of the foil is sufficient to keep the concentration of hydrogen below the lower flammability limit of hydrogen in air (4 vol%). So, even if there is a leak, the condition inside the dissolution vessel is avoided.
- The DC power supply must be turned off, and the flow of nitrogen gas must be maintained until the vacuum header reads less than 10 L/min (<20 scfh).** The atmosphere inside the dissolution vessel through the mixer stirrer shaft and other leak points in the dissolver cover must be maintained. The formation of a combustible atmosphere inside the dissolver. The gas flow must be maintained at 5 liters/minute (>20 scfh) to ensure that the maximum hydrogen concentration is maintained, and so the DC power supply must be turned off and the DC power supply turned back on once the problem has been resolved.
- The DC power must be turned off if the nitrogen flow stops.** The DC power must be turned off if the nitrogen flow stops.

Locations:

Building 3525, Room 130

Building 3525, Room 131

Building 3525, Room EASTCELL

Attachments:

None.

7.8 This operation involves chemicals or wastes that are caustic or corrosive (e.g. acids or bases).

Requirements:

Chemical Safety

Personal Protective Equipment

Hazard Notes:

- Aqueous sodium hydroxide and sodium carbonate will be used to dissolve the
- Aqueous nitric acid will be used to neutralize solutions and to cause precipitation.

Control Notes:

- Proper controls will be used for all work with these chemicals outside of the hot cell (installation of the equipment in the hot cell).
- The hot cell will protect workers against exposure when work is performed in the hot cell.
- A catch pan will be placed beneath the equipment to capture spilled liquids.
- If there is potential for splashing, goggles and a face shield must be worn.

Locations:

Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

7.9 This operation involves chemicals or wastes that are **toxic**.

Requirements:

Chemical Safety
Exposure Assessments
Personal Protective Equipment

Hazard Notes:

- The acids and bases used in this work are toxic if ingested.

Control Notes:

- All work with acids and bases will be performed in the hot cell to prevent accidental exposure.

Low Risk Exposure Justification:

All chemicals will be handled in a hot cell. Participants are unlikely to exceed any occupational exposure limits (OELs).

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

8.3 This operation generates **radioactive waste** (including mixed hazardous, solid low level, liquid low level, c

Potential Training:

Liquid Waste Generator at ORNL
RCRA 90 Day or 180 Day Accumulation Area
RCRA HAZARDOUS WASTE CHARACTERIZATION AT ORNL
RCRA LAND DISPOSAL RESTRICTIONS FOR GENERATORS

RCRA SATELLITE ACCUMULATION AREA AT ORNL
Waste Generator Awareness Training

Requirements:

Comply with Clean Water Act
Environmental Management
Manage Waste and Excess Materials

Hazard Notes:

- Radioactive waste (solid low-level, liquid low-level and/or mixed waste) may be released from the equipment, experiment disassembly, sample preparation, and testing or e
- The experimental equipment will need to be dried, dismantled (as needed), i
- be saved and stored for future work.

Control Notes:

- NNFD personnel are responsible for disposal of radioactive waste from this fa

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

8.8 This operation generates air emissions or uses Class I or II ozone depleting substances.

Requirements:

Comply with Clean Air Requirements
Environmental Management

Hazard Notes:

- Radioactive Kr and Xe will be released from the irradiated target when it is c
- Radioactive iodine will be released from the irradiated target when it is cut, a

Control Notes:

- No capture of Kr and Xe will be performed, and it is assumed that all Kr and Xe will be released. The total amount of Kr and Xe, and the isotopic composition of these gases will be reported to NNFD Management for submittal to the NESHAP annual inventory report.
- The size, enrichment, and position of the target will be adjusted to limit the release of radioactive iodine. However, capture of radioactive iodine is planned in order to limit or eliminate its release.
- The experimental apparatus will be equipped with an activated carbon iodine trap. The trap will be purged with nitrogen, and the sweep gas will be channeled through the activated carbon trap.
- One of the hot cell HEPA filters will be replaced with an activated carbon filter. This filter will prevent iodine from entering the cell atmosphere from the equipment, or that makes it past the iodine trap.
- No safety credit will be given to the iodine trap installed on the apparatus, as the iodine trap in the HEPA bank must capture all emitted iodine. Iodine capture data from the iodine trap and activated carbon is capable of capturing greater than 99.9% of the molecular iodine. The iodine trap will be tested by facility personnel to show that flow through the filter does not bypass the filter.
- The activated carbon filter used on the apparatus itself will use activated carbon from a reputable manufacturer. The test data will show that the in-line activated carbon filter is capable of capturing greater than 99.9% of the molecular iodine.

exhaust gas of the equipment.

- Isotopic calculations for an irradiated target are provided in the attached file foil that has been irradiated for 25 days in the reactor, and that has been
- After the experiment, the in-line activated carbon filter on the apparatus will to decay before the filter is disposed. The amount of I-131 in the filter and attached isotopic calculation table (LEU foil post-25 day irradiation radionuclide) in the target foil as a function of time. Facility personnel will take into account disposing of the filter.

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

[Isotopic Calculations for Target.xlsx](#)

8.9 This operation generates wastewater.

Potential Training:

Liquid Waste Generator at ORNL
RCRA HAZARDOUS WASTE CHARACTERIZATION AT ORNL
Waste Generator Awareness Training

Requirements:

[Comply with Clean Water Act](#)
[Environmental Management](#)

Hazard Notes:

The experimental equipment will contain aqueous radioactive materials at the end of the experiment in the hot cell once the experimental work is completed.

Control Notes:

Liquid waste materials generated by the experiment will be treated and/or disposed of as liquids. The waste liquids will then be stored in bottles in the east cell until they can be handled on a case-by-case basis. In such cases, bottles may be set aside for a long time; those bottles will be combined with other compatible LLLW so that the combined material can be disposed of.

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

9.1 This operation involves activities requiring the use of mechanically-assisted lifting, rigging or hoisting.

Potential Training:

Basic Rigger

Requirements:

[Material Handling](#)

Hazard Notes:

The equipment will need to be dismantled and removed from the hot cell at the end of the experiment. The equipment will be stored in the east cell until it can be removed from the hot cell.

Control Notes:

All work of this nature will be performed inside the hot cell, and must be performed by qualified hot cell

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

11.5 This operation involves compressed gases at greater than 100 psi.

Requirements:

Compressed Gas Cylinders and Related Systems

Hazard Notes:

- Compressed nitrogen will be used to provide a sweep gas for the equipment

Control Notes:

- Compressed gas cylinders will be handled by facility personnel.
- Facility personnel will follow work rules and procedures for handling compressed g

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

14.2 This operation involves work performed outside normal working hours (6am to 7pm).

Hazard Notes:

- The radioisotope under study, Mo-99, has a 2.748d half-life. The experimental work experiment is completed to minimize losses of Mo-99 due to radioactive decay. This (6am to 7pm during weekdays). The work will be planned so that no work is performed

Control Notes:

- NNFD building management will be informed if after-hours operations (after 5:00 areas).
- Some contingent of NNFD personnel must be present when in-cell manipulator work in the Charging Area, Room 130. Support staff includes the technicians performing sample handling.
- NNFD operations staff must be consulted prior to performing any in-cell work to ensure cell radiological inventory and the type of work being performed. This is a TSR requirement.
- If an emergency occurs while performing work outside normal working hours, either

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

15.4 This operation involves packaging, transporting, or shipping of samples containing **hazardous, radioactive**

Hazard Notes:

- Staff may remove analytical samples from the hot cell and transport them to
- Lower activity samples will be transported by hand to 4501 for analysis.

Control Notes:

- Samples will be carried by hand to 4501 by someone with rad worker training, and
- When hand-carrying radiological materials, use shielded containers as appropriate permitted for less than Hazard Category 3 quantities).

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

17.2 This work requires facility **modifications** or changes to the **Facility Use Agreement** (e.g. changes in chemical services.)

Requirements:

Facility Use Agreements

Hazard Notes:

There are potentials for modification or changes to research equipment or procedures.

Control Notes:

Changes or modifications are screened following SBMS Subject Area - **Unreviewed S** that may affect the authorization basis of a nuclear facility require completion of a safety

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None.

17.3 There are critical engineering controls or monitoring equipment that must meet an operating standard with respect to radiation exposures, an oxygen deficiency alarm, a cooling water loop for a high temperature apparatus).

Hazard Notes:

Hydrogen gas is generated during dissolution of the aluminum surface layer on the uranium foil. Iodine will be released into the gas phase when the uranium foil is dissolved.

Control Notes:

A flow of nitrogen through the apparatus (i.e., gas flush) must be maintained during dissolution. The flush gas must be filtered through an iodine trap to capture radioactive iodine in the gas phase. The nitrogen must be protected by a back-up activated carbon filter that will capture radioactive iodine in the gas phase. If nitrogen is lost during performance of the electrochemical dissolution step, then the flow of nitrogen through the equipment can be restored.

Locations:

Building 3525, Room 130
Building 3525, Room 131

Building 3525, Room EASTCELL

Attachments:

None.

18.5 This operation requires special controls on procurement, such as PAAA or critical item review.

Requirements:

Purchasing Supplies and Services
Safety and Security Regulatory (SSR) Program

Hazard Notes:

Building 3525 is a Cat 2 Nuclear Facility per DOE Standard 1027-92, as amended. Work is performed on

Control Notes:

Procurement of materials that may affect operation of facility operating systems must be screened to en

Locations:

Building 3525, Room 130
Building 3525, Room 131
Building 3525, Room EASTCELL

Attachments:

None

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APPENDIX D:
Y12 NATIONAL SECURITY COMPLEX

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D-1 NU SAMPLE RESULTS 1

Report for Sample Y121310054 (U)

Y12 National Security Laboratory

09/29/2012 09:10

Distributed To:

Name:	Badge:	Media Type:
BURNETT, LP	027645	Electronic
PIERCE, DA	033241	Electronic

Classified By:

Derived From:

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Y12 National Security Laboratory
PO Box 2009 Oak Ridge, TN 37831 (865) 574-2284
Correction Report

Page 1 of 5
Lab Smp1 Id: Y121310054
Cust Smp1 Id: *3G607DV1KX

Project: Y SF C SFC MD0720

Proj Mgr: M C TUCKER-WATSON (865) 576-5216

Customer: D A PIERCE Bldg 9720-82 MS-8202 (865) 574-2644

Matrix	Chain of Custody	Work Agreement Number	Charge Number	Date Sampled	Date Received	Date Needed	Date Approved
920720	PIV539	-----	PD10670A	05/03/2012 00:00	05/10/2012 14:30	06/09/2012 23:59	09/29/2012 09:10

Sample Description: 920720
Location: 321061
Sampler(s):

Sample Status: APPROVED
Sample Approver: M J SAND

Customer Comments: Parent Lot ID: 3G607CV1KW, Sample Lot ID: 3G607DV1KX.
Lab Comments: Results changed due to chloride contamination and calculation error

Comment Objects:

<<<< Inorganic >>>>

Test: ICPMS6503 (Trace Elements by ICPMS)
Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-6503 R 0.0
Approver: V F BELT/Chemist
QC Batch/File: QCL2650002/12145A / 12145A ALL
Prep Method/Date:
Test Status: APPROVED
MT Deadline:
Date Analyzed: 05/24/2012 10:27
Lab Group: ATIA

Analyte Id	Analyte Name	Customer MT L1M FN	Dilution Factor	Lower Limit	Result	EPA Qual	Confidence	Unit
7440360	Antimony		5	0.24	<1			ug/g U
7440382	Arsenic		5	0.38	<1			ug/g U
7440393	Barium		5	0.24	1.9			ug/g U
7440699	Bismuth		5	0.24	<1			ug/g U
7440428	Boron		5	0.28	<0.5			ug/g U
7440439	Cadmium		5	0.095	<0.3			ug/g U
7440451	Cerium		5	0.24	<1			ug/g U
7440462	Cesium		5	0.24	<1			ug/g U
7429916	Dysprosium		5	0.095	<0.3			ug/g U
7440520	Erbium		500	0.97	<2			ug/g U
7440531	Europium		5	0.095	<0.3			ug/g U
7440542	Gadolinium		5	0.095	<0.3			ug/g U
7440553	Gallium		5	0.047	<1			ug/g U
7440564	Germanium		5	0.38	<1			ug/g U
7440575	Gold		500	1.9	<5			ug/g U
7440586	Hafnium		5	0.24	<1			ug/g U
7440600	Holmium		500	0.93	<1			ug/g U
7440746	Indium		5	0.24	<1			ug/g U
7439885	Iridium		5	0.24	<1			ug/g U
7439910	Lanthanum		5	0.24	<1			ug/g U
7439921	Lead		5	0.24	<1			ug/g U
7439932	Lithium		5	0.24	<1			ug/g U
7439943	Lutetium		500	0.93	<1			ug/g U
7439976	Mercury		5	0.47	<1			ug/g U
7439987	Molybdenum		5	0.24	<1			ug/g U
7440008	Neodymium		5	0.24	<1			ug/g U
7440031	Niobium		5	0.24	<1			ug/g U
7440042	Osmium		5	0.24	<1			ug/g U
7440053	Palladium		5	0.24	<1			ug/g U
7723140	Phosphorus		500	19	<20			ug/g U
7440064	Platinum		5	0.24	<1			ug/g U
7440097	Potassium		5	14	<15			ug/g U
7440100	Praseodymium		5	0.24	<1			ug/g U
7440155	Rhenium		5	0.24	<1			ug/g U
7440166	Rhodium		5	0.24	<1			ug/g U
7440177	Rubidium		5	0.26	<1			ug/g U
7440188	Ruthenium		5	0.24	<1			ug/g U
7440199	Samarium		5	0.24	<0.3			ug/g U
7440202	Scandium		500	1.9	<2			ug/g U
7782492	Selenium		5	0.47	<1			ug/g U
7440224	Silver		5	0.24	<1			ug/g U
7440246	Strontium		5	0.24	<1			ug/g U
7440257	Tantalum		5	0.28	<1			ug/g U
13494809	Tellurium		5	0.24	<1			ug/g U
7440280	Thallium		500	1.9	<2			ug/g U
7440291	Thorium		500	1.9	<2			ug/g U
7440304	Thulium		500	0.93	<1			ug/g U
7440315	Tin		5	0.24	<1			ug/g U
7440337	Tungsten		5	0.24	5.3			ug/g U
7440644	Ytterbium		500	0.93	<1			ug/g U

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Lab Smp] Id: Y121310054
Cust Smp] Id: *3G607DV1KX

7440577 Zirconium

Correction Report
5 0.24 1.4

ug/g u

Comment Objects:

Test: ICPOES6504 (Trace Elements by ICPOES)

Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y12 Y50-AC-65-6504
Approver: V F BELT/chemist
QC Batch/File: QC12650003/121458 / 121458

Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 05/24/2012 14:00
Lab Group: ATIA

Analyte Id	Analyte Name	Customer HT Lim Fn	Dilution Factor	Lower Limit	Result	EPA Qual	Confidence	Unit
7429905	Aluminum	-- -- --	5	0.47	<10			ug/g U
7440417	Beryllium	-- -- --	5	0.18	<0.2			ug/g U
7440702	Calcium	-- -- --	5	0.47	<5			ug/g U
7440473	Chromium	-- -- --	5	0.24	<5			ug/g U
7440484	Cobalt	-- -- --	5	0.24	<1			ug/g U
7440508	Copper	-- -- --	5	1.3	16			ug/g U
7439896	Iron	-- -- --	5	1.7	15			ug/g U
7439954	Magnesium	-- -- --	5	0.0024	5.6			ug/g U
7439965	Manganese	-- -- --	5	0.0024	9.3			ug/g U
7440020	Nickel	-- -- --	5	0.47	15			ug/g U
7440213	Silicon	-- -- --	5	2.4	<15			ug/g U
7440235	Sodium	-- -- --	5	2.2	<5			ug/g U
7440279	Terbium	-- -- --	5	1.8	<2			ug/g U
7440326	Titanium	-- -- --	5	1.7	6.8			ug/g U
7440622	Vanadium	-- -- --	5	0.55	<1			ug/g U
7440655	Yttrium	-- -- --	5	1.8	<5			ug/g U
7440666	Zinc	-- -- --	5	1.4	2.1			ug/g U

Comment Objects:

<<<< None >>>>

Test: 100D (Uranium By Difference)

Rpt Basis: M
Test Req Cnt: 01
Analysis Method: Y12 Y50-AC-65-9067 (ASO-COMPCALC)
Approver: A C COMPUTER/Computer
QC Batch/File:

Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 05/31/2012 15:36
Lab Group: --CALC

Analyte Id	Analyte Name	Customer HT Lim Fn	Dilution Factor	Lower Limit	Result	EPA Qual	Confidence	Unit
7440611	Uranium	-- -- --			0.999821			g/g

Comment Objects:

Test: 330 (Carbon Leco)

Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-3520 R 0.1
Approver: H M PARIKH/
QC Batch/File: QC12652027

Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 05/30/2012 12:58
Lab Group: ATIA

Analyte Id	Analyte Name	Customer HT Lim Fn	Dilution Factor	Lower Limit	Result	EPA Qual	Confidence	Unit
7440440	Carbon	-- -- --	1		16			ug/g
	Aliquot	-- -- --	1		16	1		ug/g
	Aliquot	-- -- --	1		17	1		ug/g

Comment Objects:

Test: 804 (Isotopic Uranium)

Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-8052 R 0.0
Approver: G P WATSON/chemist
QC Batch/File: QC12638005/12943

Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 05/16/2012 00:00
Lab Group: NYIS01

Analyte Id	Analyte Name	Customer HT Lim Fn	Dilution Factor	Lower Limit	Result	EPA Qual	Confidence	Unit
13968553	Uranium-233	-- -- --	1		<0.002			wt %
13966295	Uranium-234	-- -- --	1		0.005			wt %
15117961	Uranium-235	-- -- --	1		0.712			wt %

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Correction Report

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Lab Smp1 Id: Y121310054
Cust Smp1 Id: *3G607DV1KX

13982702 Uranium-236
24678828 Uranium-238

1
1
<0.002
99.282
wt %
wt %

Sample Test Comments: The precision of analytical sample results will vary with the nature and the enrichment of the sample.
For pure and homogeneous HEU samples, the 2-sigma relative uncertainty for the U235 wt% is typically within 0.025%.
At lower enrichments, the uncertainty in the relative precision for the U235 wt% is larger.

Comment Objects:

Test: 807B (Isotopic Dilution)
Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-8053 R 0.0
Approver: G P WATSON/Chemist
QC Batch/File: QC12638005/12943
Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 05/16/2012 00:00
Lab Group: MY1501

Analyte Id	Analyte Name	Customer HT Lim Fn	Dilution Factor	Lower Limit	Result	EPA Qual	Confidence	Unit
#7440611	Uranium		1		0.9928			g/g
	Aliquot		1		0.9924			g/g
	Aliquot		1		0.9932			g/g
#13966295	Uranium-234		1		0.005			wt %
	Aliquot		1		0.005			wt %
	Aliquot		1		0.005			wt %
#15117961	Uranium-235		1		0.712			wt %
	Aliquot		1		0.712			wt %
	Aliquot		1		0.712			wt %
#13982702	Uranium-236		1		<0.002			wt %
	Aliquot		1		<0.002			wt %
	Aliquot		1		<0.002			wt %
#24678828	Uranium-238		1		99.280			wt %
	Aliquot		1		99.283			wt %
	Aliquot		1		99.282			wt %

Sample Test Comments: The precision of analytical sample results will vary with the nature and the enrichment of the sample.
For pure and homogeneous samples, the g/g 2-sigma relative uncertainty is typically within 0.2% and the absolute difference for the U235 wt% is typically within 0.025% for HEU.
At lower enrichments and total g/g values, the uncertainty in the relative precision for the U235 wt% and g/g value is larger.
Sample could not be cleaned as required by the method due to the turnings being so thin. The cleaning procedure would have dissolved the sample. [030294 05/17/2012 11:00:51]

Comment Objects:

Test: *UHALIDE (Determination of Halides by IC)
Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-2732 R 0.2
Approver: V F BELT/Chemist
QC Batch/File: QC12663001/1024
Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 06/11/2012 15:17
Lab Group: AT1A

Analyte Id	Analyte Name	Customer HT Lim Fn	Dilution Factor	Lower Limit	Result	EPA Qual	Confidence	Unit
*24959679	Bromide		1		<1.00			ug/g
*16887006	Chloride		1		<1.00			ug/g
*16984488	Fluoride		1		2.961			ug/g
*20461545	Iodide (I-)		1		<1.00			ug/g

Sample Test Comments: Results changed due to chloride contamination and calculation error

Comment Objects:

<<<< Radiological >>>>

Test: 763A (U-232 Activity (dpm/ug U))
Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-7080 R 0.0
Approver: D K MANN/Chemist
QC Batch/File: QC12666009
Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 06/14/2012 07:51
Lab Group: RYRAD1

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09/29/2012 09:10

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Lab Smp1 Id: Y121310054

Correction Report

Cust Smp1 Id: *3G607DV1KX

Analyte Id	Analyte Name	Customer		MDA	Lower Limit	Result	EPA Qual	Uncert	Sig	Unit
		HT	Lim Fn							
12587461	Alpha activity					1.46E+00			2	dpm/ug U
14158293	Uranium-232			1.3E-02		-3.13E-04		2.0E-03	2	dpm/ug U

Comment Objects:

Test: ALPBET (Analysis of Gross Alpha/Beta Activity)

Rpt Basis: AR	Prep Method/Date:
Test Req Cnt: 01	Test Status: APPROVED
Analysis Method: Y50 AC-65-7081 R 0.0	HT Deadline:
Approver: D K MANN/Chemist	Date Analyzed: 06/07/2012 12:43
QC Batch/File: QC12665003	Lab Group: RYRAD1

Analyte Id	Analyte Name	Customer		MDA	Lower Limit	Result	EPA Qual	Uncert	Sig	Unit
		HT	Lim Fn							
#12587461	Alpha activity			3.00E+03		5.2E+5		0.7E+5	2	pCi/g
12587472	Beta activity			5.70E+03		4.9E+5		0.7E+5	2	pCi/g

Comment Objects:

Test: CT-AM-5 (Analysis for Am and Cm)

Rpt Basis: AR	Prep Method/Date:
Test Req Cnt: 01	Test Status: APPROVED
Analysis Method: Y50 AC-65-7227 R 0.1	HT Deadline:
Approver: D K MANN/Chemist	Date Analyzed: 06/12/2012 13:21
QC Batch/File: QC12665000/RAD-024795	Lab Group: RYRAD1

Analyte Id	Analyte Name	Customer		MDA	Lower Limit	Result	EPA Qual	Uncert	Sig	Unit
		HT	Lim Fn							
14596102	Americium-241			7.4E+01		-7.2E-01		3.4E+01	2	pCi/g
N191	Cm-243/244			4.8E+01		1.3E+01		2.6E+01	2	pCi/g

Comment Objects:

Test: CT-NP-5 (Neptunium 237)

Rpt Basis: AR	Prep Method/Date:
Test Req Cnt: 01	Test Status: APPROVED
Analysis Method: Y50 AC-65-7082 R 0.0	HT Deadline:
Approver: D K MANN/Chemist	Date Analyzed: 06/13/2012 05:38
QC Batch/File: QC12666004/RAD-024797	Lab Group: RYRAD1

Analyte Id	Analyte Name	Customer		MDA	Lower Limit	Result	EPA Qual	Uncert	Sig	Unit
		HT	Lim Fn							
13994202	Neptunium-237			8.9E+01		8.0E+00		4.4E+01	2	pCi/g

Comment Objects:

Test: CT-PU-5 (Plutonium by Alpha Spectrometry)

Rpt Basis: AR	Prep Method/Date:
Test Req Cnt: 01	Test Status: APPROVED
Analysis Method: Y50 AC-65-7225 R 0.1	HT Deadline:
Approver: D K MANN/Chemist	Date Analyzed: 06/12/2012 13:22
QC Batch/File: QC12665001/RAD-024799	Lab Group: RYRAD1

Analyte Id	Analyte Name	Customer		MDA	Lower Limit	Result	EPA Qual	Uncert	Sig	Unit
		HT	Lim Fn							
13981163	Plutonium-238			6.7E+01		-6.1E+00		2.9E+01	2	pCi/g
N760	Plutonium-239/240			5.9E+01		-6.0E+00		2.4E+01	2	pCi/g

Comment Objects:

Test: ND-GAM (Analysis of Gamma-Emitting Isotopes by Gamma Spec.)

Rpt Basis: AR	Prep Method/Date:
Test Req Cnt: 01	Test Status: APPROVED
Analysis Method: Y50 AC-65-7039 R 0.1	HT Deadline:
Approver: D K MANN/Chemist	Date Analyzed: 05/30/2012 06:29
QC Batch/File: QC12665002	Lab Group: RYRAD1

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Lab Smp1 Id: V121310054

Correction Report

Cust Smp1 Id: *3G607DV1KX

Analyte Id	Analyte Name	Customer			MDA	Lower Limit	Result	EPA Qual	Uncert	Sig	Unit
		HT	Lim	Fn							
14234356	Antimony-125			j	4.4E+02		0.0E+00		2.7E+02	2	pci/g
14913496	Bismuth-212			j	1.6E+03		4.9E+02		9.8E+02	2	pci/g
13967743	Cerium-141			y	3.7E+02		2.1E+03		2.0E+02	2	pci/g
14762788	Cerium-144			j	9.1E+02		3.4E+02		6.0E+02	2	pci/g
13967709	Cesium-134			j	1.8E+02		1.0E+01		1.1E+02	2	pci/g
10045973	Cesium-137			j	2.1E+02		8.0E+01		1.2E+02	2	pci/g
14392020	Chromium-51			j	1.0E+03		4.0E+02		5.9E+02	2	pci/g
13981505	Cobalt-57			y	1.2E+02		1.3E+02		8.0E+01	2	pci/g
13981389	Cobalt-58			j	1.5E+02		-1.3E+02		1.1E+02	2	pci/g
10198400	Cobalt-60			j	1.7E+02		0.0E+00		1.1E+02	2	pci/g
14391163	Europium-155			y	5.5E+02		9.5E+02		3.8E+02	2	pci/g
15092941	Lead-212			j	1.8E+02		0.0E+00		1.2E+02	2	pci/g
13967765	Niobium-95			y	2.0E+02		1.0E+03		3.0E+02	2	pci/g
N2006	Protactinium-234m				2.3E+04		3.5E+05		6.0E+04	2	pci/g
13233324	Radium-224			i	2.2E+03		0.0E+00		1.4E+03	2	pci/g
13968531	Ruthenium-103			j	1.7E+02		8.0E+01		1.0E+02	2	pci/g
13967481	Ruthenium-106			j	1.7E+03		0.0E+00		1.1E+03	2	pci/g
14913509	Thallium-208			j	1.7E+02		0.0E+00		1.3E+02	2	pci/g
14932402	Thorium-231			i	2.0E+03		1.4E+04		2.0E+03	2	pci/g
15065108	Thorium-234				4.0E+03		3.1E+05		1.0E+04	2	pci/g
#13966295	Uranium-234				1.4E+05		6.5E+05		1.4E+05	2	pci/g
#15117961	Uranium-235				1.8E+02		1.7E+04		1.0E+03	2	pci/g
#24678828	Uranium-238				4.0E+03		3.1E+05		1.0E+04	2	pci/g
13967710	Zirconium-95			j	3.9E+02		0.0E+00		2.5E+02	2	pci/g

Comment Objects:

Test: TC99-95 (Determination of Technetium-99)

Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-7166 R 0.2
Approver: D K MANN/Chemist
QC Batch/File: QC12665004

Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 06/07/2012 00:00
Lab Group: RVRAD1

Analyte Id	Analyte Name	Customer			MDA	Lower Limit	Result	EPA Qual	Uncert	Sig	Unit
		HT	Lim	Fn							
14133767	Technetium-99				57		2.8E+1		3.5E+1	2	pci/g

Comment Objects:

Footnotes:

- i - Result is biased due to incomplete peak separation.
- j - Isotope analyzed for at customer request, but not detected
- y - False positive due to Gamma photopeak interference

An asterisk (*) beside a Customer Sample, Test, Analyte, Aliquot, or TIC indicates a modification after re-open.
A pound sign (#) beside a Analyte id indicates another Lab Test may have a more accurate result.

***** The results relate only to the items tested. *****
***** Results contained within this report are not Blank Corrected unless specifically noted *****
***** END OF REPORT *****

D-2 NU SAMPLE RESULTS 2

Report for Sample Y120100009 (U)

Y12 National Security Laboratory

09/29/2012 09:22

Distributed To:

Name:	Badge:	Media Type:
BURNETT, LP	027645	Electronic
PIERCE, DA	033241	Electronic

Classified By:

Derived From:

LIMS 3.00.037
09/29/2012 09:22

Y12 National Security Laboratory
PO Box 2009 Oak Ridge, TN 37831 (865) 574-2284
Correction Report

Page 1 of 6
Lab Smp1 Id: Y120100009
Cust Smp1 Id: *3G607DTFTE

Project: Y SF C SFC MD0720

Proj Mgr: M C TUCKER-WATSON (865) 576-5216

Customer: D A PIERCE Bldg 9720-82 MS-8202 (865) 574-2644

Matrix	Chain of Custody	Work Agreement Number	Charge Number	Date Sampled	Date Received	Date Needed	Date Approved
920720	PJT790	-----	PD10670A	01/10/2012 00:00	01/10/2012 10:10	02/09/2012 23:59	09/29/2012 09:22

Sample Description: 920720

Location:

Sampler(s):

Sample Status: APPROVED

Sample Approver: M J SAND

Customer Comments: Parent Lot ID: 3G607CTFTE, Sample Lot ID: 3G607DTFTE.

Lab Comments: Results changed due to chloride contamination and calculation error

Comment Objects:

<<<< Inorganic >>>>

Test: *ICPMS6503 (Trace Elements by ICPMS)

Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-6503 R 0.0
Approver: V F BELT/Chemist
QC Batch/File: QC12524007/12019A / 12019A ALL

Prep Method/Date:
Test Status: APPROVED
MT Deadline:
Date Analyzed: 01/19/2012 10:28
Lab Group: ATIA

Analyte Id	Analyte Name	Customer MT Lm Fn	Dilution Factor	Lower Limit	Result	EPA Qual	Confidence	Unit
*7440360	Antimony	5	0.24	<1				ug/g U
*7440382	Arsenic	5	0.38	<1				ug/g U
*7440393	Barium	5	0.24	<1				ug/g U
*7440699	Bismuth	5	0.24	<1				ug/g U
*7440428	Boron	5	0.28	<0.5				ug/g U
*7440439	Cadmium	5	0.095	<0.3				ug/g U
*7440451	Cerium	5	0.24	<1				ug/g U
*7440462	Cesium	5	0.24	<1				ug/g U
*7429916	Dysprosium	5	0.095	<0.3				ug/g U
*7440520	Erbium	500	0.97	<2				ug/g U
*7440531	Europium	5	0.095	<0.3				ug/g U
*7440542	Gadolinium	5	0.095	<0.3				ug/g U
*7440553	Gallium	5	0.047	<1				ug/g U
*7440564	Germanium	5	0.38	<1				ug/g U
*7440575	Gold	500	1.9	<5				ug/g U
*7440586	Hafnium	5	0.24	<1				ug/g U
*7440600	Holmium	500	0.93	<1				ug/g U
*7440746	Indium	5	0.24	<1				ug/g U
*7439885	Iridium	5	0.24	<1				ug/g U
*7439910	Lanthanum	5	0.24	<1				ug/g U
*7439921	Lead	5	0.24	<1				ug/g U
*7439932	Lithium	5	0.24	<1				ug/g U
*7439943	Lutetium	500	0.93	<1				ug/g U
*7439976	Mercury	5	0.47	<1				ug/g U
*7439987	Molybdenum	5	0.24	<1				ug/g U
*7440008	Neodymium	5	0.24	<1				ug/g U
*7440031	Niobium	5	0.24	<1				ug/g U
*7440042	Osmium	5	0.24	<1				ug/g U
*7440053	Palladium	5	0.24	<1				ug/g U
*7223140	Phosphorus	500	19	<20				ug/g U
*7440064	Platinum	5	0.24	<1				ug/g U
*7440097	Potassium	5	14	<15				ug/g U
*7440100	Praseodymium	5	0.24	<1				ug/g U
*7440155	Rhenium	5	0.24	<1				ug/g U
*7440166	Rhodium	5	0.24	<1				ug/g U
*7440177	Rubidium	5	0.28	<1				ug/g U
*7440188	Ruthenium	5	0.24	<1				ug/g U
*7440199	Samarium	5	0.24	<0.3				ug/g U
*7440202	Scandium	500	1.9	<2				ug/g U
*7782492	Selenium	5	0.47	<1				ug/g U
*7440224	Silver	5	0.24	<1				ug/g U
*7440246	Strontium	5	0.24	<1				ug/g U
*7440257	Tantalum	5	0.28	<1				ug/g U
*13494809	Tellurium	5	0.24	<1				ug/g U
*7440280	Thallium	500	1.9	<2				ug/g U
*7440291	Thorium	500	1.9	<2				ug/g U
*7440304	Thulium	500	0.93	<1				ug/g U
*7440315	Tin	5	0.24	<1				ug/g U
*7440337	Tungsten	5	0.24	<1				ug/g U
*7440644	Ytterbium	500	0.93	<1				ug/g U

Sample Test Comments:
customer request [027645 01/10/2012 12:40:18]
add tests per J Cantrell [033950 01/18/2012 10:15:05]

Comment Objects:

Test: *ICP0ES6504 (Trace Elements by ICP0ES)
Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y12 Y50-AC-65-6504
Approver: V F BELT/chemist
QC Batch/File: QC12524008/120198 / 120198

Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 01/24/2012 10:40
Lab Group: ATIA

Analyte Id	Analyte Name	Customer HT Lim Fn	Dilution Factor	Lower Limit	Result	EPA Qual	Confidence	Unit
*7429905	Aluminum	5	0.47	<10				ug/g u
*7440417	Beryllium	5	0.18	<0.2				ug/g u
*7440702	Calcium	5	0.47	<5				ug/g u
*7440473	Chromium	5	0.24	<5				ug/g u
*7440484	Cobalt	5	0.24	<1				ug/g u
*7440508	Copper	5	1.3	18				ug/g u
*7439896	Iron	5	1.7	20				ug/g u
*7439954	Magnesium	5	0.0024	3.4				ug/g u
*7439965	Manganese	5	0.0024	8.9				ug/g u
*7440020	Nickel	5	0.47	13				ug/g u
*7440213	Silicon	5	2.4	<15				ug/g u
*7440235	Sodium	5	2.2	<5				ug/g u
*7440279	Terbium	5	1.8	<2				ug/g u
*7440326	Titanium	5	1.7	7.1				ug/g u
*7440622	Vanadium	5	0.55	<1				ug/g u
*7440655	Yttrium	5	1.8	<5				ug/g u
*7440666	Zinc	5	1.4	<2				ug/g u

Sample Test Comments:
customer request [027645 01/10/2012 12:40:29]
add tests per J Cantrell [033950 01/18/2012 10:15:20]

Comment Objects:

<<<< None >>>>

Test: *100D (Uranium By Difference)
Rpt Basis: M
Test Req Cnt: 01
Analysis Method: Y12 Y50-AC-65-8067 (ASO-COMPCALC)
Approver: A C COMPUTER/Computer
QC Batch/File:

Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 01/24/2012 14:24
Lab Group: --CALC

Analyte Id	Analyte Name	Customer HT Lim Fn	Dilution Factor	Lower Limit	Result	EPA Qual	Confidence	Unit
7440611	uranium				0.999825			g/g

Sample Test Comments:
Analyze by TIMS [027645 01/10/2012 12:38:33]
add tests per J Cantrell [033950 01/18/2012 10:13:33]

Comment Objects:

Test: *330 (Carbon Leco)
Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-3520 R 0.1
Approver: M M PARIKH/
QC Batch/File: QC12524017

Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 01/23/2012 14:11
Lab Group: ATIA

Analyte Id	Analyte Name	Customer HT Lim Fn	Dilution Factor	Lower Limit	Result	EPA Qual	Confidence	Unit
*7440440	Carbon		1		15			ug/g
	Aliquot		1		15		1	ug/g
	Aliquot		1		14		1	ug/g

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Correction Report

Page 3 of 6
Lab Smp] Id: Y120100009
Cust Smp] Id: *3G607DTFTE

Sample Test Comments:
customer request [027645 01/10/2012 12:39:01]
add tests per J Cantrell [033950 01/18/2012 10:13:47]

Comment Objects:

Test: 804 (Isotopic Uranium)
Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-8052 R 0.0
Approver: G P WATSON/Chemist
QC Batch/File: QC12517001/12598

Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 01/12/2012 23:11
Lab Group: MYISOL

Analyte Id	Analyte Name	Customer HT Lim Fn	Dilution Factor	Lower Limit	Result	EPA Qual	Confidence	Unit
13968553	Uranium-233		1		<0.002			wt %
13966295	Uranium-234		1		0.006			wt %
15117961	Uranium-235		1		0.712			wt %
13982702	Uranium-236		1		<0.002			wt %
24678828	Uranium-238		1		99.282			wt %

Sample Test Comments: The precision of analytical sample results will vary with the nature and the enrichment of the sample.
For pure and homogeneous HEU samples, the 2-sigma relative uncertainty for the U235 wt% is typically within 0.025%.
At lower enrichments, the uncertainty in the relative precision for the U235 wt% is larger.

Comment Objects:

Test: 807 (Isotopic Dilution)
Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y12 Y50-AC-65-8044
Approver: G P WATSON/Chemist
QC Batch/File: QC12517000/12597

Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 01/12/2012 16:19
Lab Group: MYISOL

Analyte Id	Analyte Name	Customer HT Lim Fn	Dilution Factor	Lower Limit	Result	EPA Qual	Confidence	Unit
#7440611	Uranium		1		998800			ug/g
	Aliquot		1		999000			ug/g
	Aliquot		1		998000			ug/g
#13966295	Uranium-234		1		0.005			wt %
	Aliquot		1		0.005			wt %
	Aliquot		1		0.005			wt %
#15117961	Uranium-235		1		0.713			wt %
	Aliquot		1		0.712			wt %
	Aliquot		1		0.714			wt %
#13982702	Uranium-236		1		<0.002			wt %
	Aliquot		1		<0.002			wt %
	Aliquot		1		<0.002			wt %
#24678828	Uranium-238		1		99.280			wt %
	Aliquot		1		99.283			wt %
	Aliquot		1		99.280			wt %

Sample Test Comments: For pure and homogeneous samples, the g/g 2-sigma relative uncertainty is typically within 0.2% and the absolute difference for the U235 wt% is typically within 0.025% for HEU.

Comment Objects:

Test: 807B (Isotopic Dilution)
Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-8053 R 0.0
Approver:
QC Batch/File:

Prep Method/Date:
Test Status: CANCELLED
HT Deadline:
Date Analyzed:
Lab Group: MYISOL

Sample Test Comments:
do 807 [027645 01/11/2012 10:59:01]

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09/29/2012 09:22

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Correction Report

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Lab Smp1 Id: Y120100009
Cust Smp1 Id: *3G607DTFTE

Test: *UHALIDE (Determination of Halides by IC)
Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-2732 R 0.2
Approver: D K MANN/Chemist
QC Batch/File: QC12533004/1006
Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 01/26/2012 08:41
Lab Group: ATIA

Analyte Id	Analyte Name	Customer HT Lim Fn	Dilution Factor	Lower Limit	Result	EPA Qual	Confidence	Unit
*24959679	Bromide		1		<1.00			ug/g
*16887006	Chloride		1		33.636			ug/g
*16984488	Fluoride		1		3.1585			ug/g
*20461545	Iodide (I-)		1		<1.00			ug/g

Sample Test Comments: Results changed due to chloride contamination and calculation error

Comment Objects:

<<<< Radiological >>>>

Test: *763A (U-232 Activity (dpm/ug U))
Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-7080 R 0.0
Approver: E S CARLTON/
QC Batch/File: QC12547001
Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 02/08/2012 05:37
Lab Group: RYRAD1

Analyte Id	Analyte Name	Customer HT Lim Fn	MDA	Lower Limit	Result	EPA Qual	UnCert	Sig	Unit
*12587461	Alpha activity				1.60E+00			2	dpm/ug U
*14158293	Uranium-232		1.3E-04		9.66E-09		1.9E-06	2	dpm/ug U

Sample Test Comments:

customer request [027645 01/10/2012 12:39:17]
add tests per J Cantrell [033950 01/18/2012 10:13:58]

Comment Objects:

Test: *ALPBET (Analysis of Gross Alpha/Beta Activity)
Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-7081 R 0.0
Approver: E S CARLTON/
QC Batch/File: QC12540027
Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 02/09/2012 13:51
Lab Group: RYRAD1

Analyte Id	Analyte Name	Customer HT Lim Fn	MDA	Lower Limit	Result	EPA Qual	UnCert	Sig	Unit
*#12587461	Alpha activity		1.70E+04		7.1E+5		0.8E+5	2	pCi/g
*12587472	Beta activity		2.40E+04		5.5E+5		0.5E+5	2	pCi/g

Sample Test Comments:

customer request [027645 01/10/2012 12:39:30]
add tests per J Cantrell [033950 01/18/2012 10:14:12]

Comment Objects:

Test: *CT-AM-5 (Analysis for Am and Cm)
Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-7227 R 0.1
Approver: E S CARLTON/
QC Batch/File: QC12532023/RAD-023874
Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 02/01/2012 06:19
Lab Group: RYRAD1

Analyte Id	Analyte Name	Customer HT Lim Fn	MDA	Lower Limit	Result	EPA Qual	UnCert	Sig	Unit
*14596102	Americium-241		1.2E+02		-1.1E+01		5.3E+01	2	pCi/g

Sample Test Comments:

customer request [027645 01/10/2012 12:39:44]
add tests per J Cantrell [033950 01/18/2012 10:14:25]

LIMS 3.00.037
09/29/2012 09:22

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Correction Report

Page 5 of 6
Lab Smp1 Id: Y120100009
Cust Smp1 Id: *3G607DTFTE

Comment Objects:

Test: *CT-NP-5 (Neptunium 237)
Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-7082 R 0.0
Approver: E S CARLTON/
QC Batch/File: QC12540024/RAD-023876

Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 02/06/2012 08:08
Lab Group: RYRAD1

Analyte Id	Analyte Name	Customer HT Lim Fn	MDA	Lower Limit	Result	EPA Qual	UnCert	Sig	Unit
*13994202	Neptunium-237	-- -- --	1.2E+02	-----	1.0E+01	-----	5.8E+01	2	pCi/g

Sample Test Comments:
customer request [027645 01/10/2012 12:39:57]
add tests per J Cantrell [033950 01/18/2012 10:14:39]

Comment Objects:

Test: *CT-PU-5 (Plutonium by Alpha Spectrometry)
Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-7225 R 0.1
Approver: E S CARLTON/
QC Batch/File: QC12540025/RAD-023878

Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 02/07/2012 09:32
Lab Group: RYRAD1

Analyte Id	Analyte Name	Customer HT Lim Fn	MDA	Lower Limit	Result	EPA Qual	UnCert	Sig	Unit
*13981163	Plutonium-238	-- -- --	6.8E+01	-----	-6.0E+00	-----	2.8E+01	2	pCi/g
*N760	Plutonium-239/240	-- -- --	4.7E+01	-----	-6.4E+00	-----	1.3E+01	2	pCi/g

Sample Test Comments:
customer request [027645 01/10/2012 12:40:07]
add tests per J Cantrell [033950 01/18/2012 10:14:52]

Comment Objects:

Test: *ND-GAM (Analysis of Gamma-Emitting Isotopes by Gamma Spec.)
Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-7039 R 0.1
Approver: E S CARLTON/
QC Batch/File: QC12540026

Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 02/02/2012 08:30
Lab Group: RYRAD1

Analyte Id	Analyte Name	Customer HT Lim Fn	MDA	Lower Limit	Result	EPA Qual	UnCert	Sig	Unit
*14234356	Antimony-125	j	3.1E+02	-----	0.0E+00	-----	1.9E+02	2	pCi/g
*14913496	Bismuth-212	j	1.2E+03	-----	2.0E+01	-----	7.4E+02	2	pCi/g
*13967743	Cerium-141	y	2.4E+02	-----	1.5E+03	-----	2.0E+02	2	pCi/g
*14762788	Cerium-144	j	6.3E+02	-----	-1.0E+01	-----	4.3E+02	2	pCi/g
*13967709	Cesium-134	j	1.2E+02	-----	6.0E+00	-----	7.4E+01	2	pCi/g
*10045973	Cesium-137	j	1.4E+02	-----	2.9E+01	-----	8.5E+01	2	pCi/g
*14392020	Chromium-51	j	7.0E+02	-----	0.0E+00	-----	4.2E+02	2	pCi/g
*13981505	Cobalt-57	y	9.2E+01	-----	1.7E+02	-----	6.0E+01	2	pCi/g
*13981389	Cobalt-58	j	1.5E+02	-----	-2.2E+01	-----	9.4E+01	2	pCi/g
*10198400	Cobalt-60	j	1.2E+02	-----	1.6E+01	-----	7.8E+01	2	pCi/g
*14391163	Europtium-155	y	4.1E+02	-----	1.2E+03	-----	3.0E+02	2	pCi/g
*15092941	Lead-212	j	1.4E+02	-----	-3.0E+01	-----	9.2E+01	2	pCi/g
*13967765	Niobium-95	y	1.5E+02	-----	1.2E+03	-----	2.0E+02	2	pCi/g
*N2006	Protactinium-234m	y	1.7E+04	-----	3.6E+05	-----	5.0E+04	2	pCi/g
*13233324	Radium-224	i	1.6E+03	-----	4.0E+02	-----	1.0E+03	2	pCi/g
*13968531	Ruthenium-103	j	1.2E+02	-----	-1.3E+01	-----	7.3E+01	2	pCi/g
*13967481	Ruthenium-106	j	1.2E+03	-----	-4.6E+02	-----	7.9E+02	2	pCi/g
*14913509	Thallium-208	j	1.4E+02	-----	-3.7E+01	-----	9.2E+01	2	pCi/g
*14932402	Thorium-231	i	1.4E+03	-----	1.2E+04	-----	2.0E+03	2	pCi/g
*15065108	Thorium-234	j	2.8E+03	-----	3.4E+05	-----	1.0E+04	2	pCi/g
*13966295	uranium-234	j	9.5E+04	-----	7.4E+05	-----	1.0E+04	2	pCi/g
*15117961	uranium-235	j	1.3E+02	-----	1.6E+04	-----	1.0E+03	2	pCi/g
*24678828	uranium-238	j	2.8E+03	-----	3.4E+05	-----	1.0E+04	2	pCi/g
*13967710	Zirconium-95	j	3.4E+02	-----	9.0E+01	-----	2.0E+02	2	pCi/g

LIMS 3.00.037
09/29/2012 09:22

Y12 National Security Laboratory
PO Box 2009 Oak Ridge, TN 37831 (865) 574-2284
Correction Report

Page 6 of 6
Lab Smp1 Id: Y120100009
Cust Smp1 Id: *3G607DTFTE

Sample Test Comments:

customer request [027645 01/10/2012 12:40:39]
add tests per J Cantrell [033950 01/18/2012 10:15:32]

Comment Objects:

Test: *TC99-95 (Determination of Technetium-99)
Rpt Basis: AR
Test Req Cnt: 01
Analysis Method: Y50 AC-65-7166 R 0.2
Approver: E S CARLTON/
QC Batch/File: QC12540028
Prep Method/Date:
Test Status: APPROVED
HT Deadline:
Date Analyzed: 02/01/2012 00:00
Lab Group: RYRAD1

Analyte Id	Analyte Name	Customer HT Lim Fn	MDA	Lower Limit	Result	EPA Qual	UnCert	Sig	Unit
*14133767	Technetium-99	-- -- --	31	-----	-0.9E+1	-----	1.9E+1	2	pci/g

Sample Test Comments:

customer request [027645 01/10/2012 12:40:49]
add tests per J Cantrell [033950 01/18/2012 10:15:45]

Comment Objects:

Footnotes:

- i - Result is biased due to incomplete peak separation.
- j - Isotope analyzed for at customer request, but not detected
- y - False positive due to Gamma photopeak interference

An asterisk (*) beside a Customer Sample, Test, Analyte, Aliquot, or TIC indicates a modification after re-open.
A pound sign (#) beside a Analyte id indicates another Lab Test may have a more accurate result.

***** The results relate only to the items tested. *****
***** Results contained within this report are not Blank Corrected unless specifically noted *****
***** END OF REPORT *****

D-3 GOODFELLOW NICKEL CHEMICAL ANALYSIS

CERTIFICATE OF CONFORMITY

Ermine Business Park
Huntingdon PE29 6WR England
Telephone 011 44 1480 424 800
Fax : 011 44 1480 424 900

Page 1 of 1

We certify that these goods are in accordance with the specification given below.
Goodfellow is registered to ISO 9001 / 2008

U.S. Department of Energy
Oak Ridge National Laboratory
Building 7910, Room 38C
Bethel Valley Road
OAK RIDGE
TN 37830
USA

For the attention of:

Your order number 11819
Date of your order May 16 2013
Our order reference LS407972/R R L
Customer reference no. 12054-44
Invoice number U76371
Order complete
Date of shipment May 22 2013
5216 431 079 80
2 May 22 2013

Item	Description	Quantity ordered	Quantity	HS Tariff number	Unit price
1	NI007968 Nickel rod Ni 99.99% Diameter : 16.0mm +/-0.5mm Length : 100mm +/-1mm	8 pcs	8 pcs	7505113000	
<p>Supplied for Research, Development or Industrial purposes only. I certify that all the chemical substances in this shipment are not subject to the TSCA. D. Bush (Shipping Manager) Goodfellow Corporation is classified by U.S. Government regulations as a Small Business for purposes of Federal procurement. No returns will be accepted after 14 days from receipt of material.</p> <p>Consignee EIN : 52-1946 236</p>					

Ship to:
as shown above

Method of shipment
FedEx Air Express : 5216 4310 7980

Mark on parcel

Additional notes
Order being paid for by Credit Card

Parcel type
1 Polythene Transit Bag

Net weight
1.07 kg

Gross weight
1.39 kg

Size
16 x 13 x 11 cm

Contents

WARNING: our products are of a highly specialised nature and are packaged with the utmost care. The goods delivered are as detailed above. It is essential that all items are checked correctly against this advice note and that they correspond in all respects with your requirements. Any discrepancies should be notified to us immediately. We would advise that all goods are unpacked and handled only by a person qualified to deal with such specialised products to safeguard against injury to the product itself or the purchaser.

Goodfellow Corporation
125 Hookstown Grade Road, Coraopolis, PA 15108-9302. USA
E-mail: info@goodfellow.com
Web: www.goodfellow.com
Tel: 1-800-821-2870
Fax: 1-800-283-2020

U.S. Department of Energy
Oak Ridge National Laboratory
Bethel Valley Road
OAK RIDGE
TN 37831-8094

Your reference
Dated
Our reference LS407972/L O
NI007968/1
Date 22-May-2013

CERTIFICATE OF ANALYSIS

Our reference : LS407972
Your order number : SBR5556
Item number : NI007968/1

Nickel	: Matrix
Antimony	: 0.74
Chromium	: 2.05
Cobalt	: 1
Copper	: 0.115
Iridium	: 0.245
Iron	: 12.25
Molybdenum	: 0.32
Osmium	: 0.215
Rhenium	: 0.135
Silicon	: 0.145
Tantalum	: <1
Titanium	: 0.185

All impurities in ppm unless otherwise stated.
The analysis was performed on this batch either in the final form or at an earlier stage of manufacture.

D-4 Y-12 ASSEMBLY OF NICKEL IRRADIATION CAPSULE

Y/??-???

**Y-12
NATIONAL
SECURITY
COMPLEX**

MANAGED BY
B&W Y-12, LLC
FOR THE UNITED STATES
DEPARTMENT OF ENERGY
UCN-13672 (12-09)

ASSEMBLY OF NICKEL IRRADIATION CAPSULE

**Barak Tjader
John Creasy
Jack Gooch
Amy Demint
J. M. Holland**

July 2013

Prepared by
Y-12 National Security Complex
Babcock & Wilcox Technical Services Y-12, LLC
P. O. Box 2009
Oak Ridge, TN 37831-8169

This document has been reviewed by an Authorized
Derivative Classifier/UCNI RO and has been determined
to be UNCLASSIFIED and contains NO UCNI. This
review does not constitute clearance for public release.
Name: Steve McClanahan Date: Nov 3, 2011

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ASSEMBLY OF NICKEL IRRADIATION CAPSULE

Barak Tjader
John Creasy

Nuclear Nonproliferation and Global Security Programs

Jack Gooch
Amy Demint
J. M. Holland

Y-12 Development

July 2013

Prepared by
Babcock & Wilcox Technical Services Y-12, LLC
Managing & Operating Contractor
for the
Y-12 National Security Complex
under Contract No. DE-AC05-00OR22800
with the
U.S. Department of Energy
National Nuclear Security Administration

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1. INTRODUCTION

The Global Threat Reduction Initiative (GTRI) Mo-99 High Density Target (HDT) project objective is to demonstrate a LEU target which is able to provide similar or greater Mo-99 yields per target than the existing dispersion HEU based targets currently used to provide over 90% of the world's Mo-99. A key aspect of this technology is the ability to roll thin uranium bare metal foils accurately and consistently, in addition, improved chemical dissolution methods are under development to enable the chemical extraction of the Mo-99 from the irradiated targets. To allow for the testing of the chemical processes in an economical fashion, a rabbit based capsule has been developed for irradiation in the High Flux Isotope Reactor (HFIR) that will allow for foils to be irradiated by HFIR and subsequently dissolved in the chemical processing equipment installed in the Oak Ridge National Lab (ORNL) hot cells. This document serves to describe the nickel capsule assembly procedure.

2. Bill of Materials

Receive nickel capsules, springs, and mandrel from Oak Ridge National Laboratory (ORNL). Natural uranium foils shall be produced per Y/??-???, "Preparation of Uranium Foils for Ni Capsule Assembly."

Uranium Foil Lot ID: _____

Net weight: _____

Uranium weight: _____

U-235 weight: _____

Sign: _____ Date: _____

3. Foil Preparation

Induce curving in uranium foil by wrapping the foil around mandrel provided by ORNL. How do they hold the foil in place???

Sign: _____ Date: _____

4. Assembly Loading

Use forceps to compress nickel spring and wrap uranium foil around the spring. Insert into nickel capsule, aligning the gap in the spring with the gap in the capsule.

Alternatively, pre curve uranium foil and insert into capsule. Use forceps to compress nickel spring from center of tabs and insert into capsule, aligning the gap in the spring with the gap in the foil.

Sign: _____ Date: _____

5. Welding

Weld capsule per Y/DV-2294, "Welding Procedure for HFIR Target Irradiation Nickel Capsules."

Sign: _____ Date: _____

Weld Inspection:

Sign: _____ Date: _____

D-5 Y-12 FOIL-PREPARATION GUIDE

Document No. : Y/PM-166
Date: September 20, 2013
Page 1 of 12

STANDARD OPERATING GUIDELINE

Foil Preparation

TITLE: Foil Preparation

9/24/2013 4:57:55 PM

This document has been reviewed by a CNS Dual
Authority DC/RO and confirmed to be
UNCLASSIFIED.

Name: Amanda Crumby

Date: 07/26/2016

A handwritten signature in black ink that reads 'A. Crumby'.

Digitally signed by Amanda B (46V) Crumby
DN: cn=US, o=U.S. Government,
ou=Department of Energy, ou=Y-12 National
Security Complex, ou=CAs, ou=people,
cn=Amanda B (46V) Crumby
Date: 2016.07.26 16:54:59 -0400

Document No. : Y/PM-166
Date: September 20, 2013
Page 2 of 12

STANDARD OPERATING GUIDELINE

Foil Preparation

RECORD OF REVISION

Revision	Date	Description
0 -NEW	9/20/2013	New operating guideline detailing instructions for foil preparation.

9/24/2013 4:57:55 PM

STANDARD OPERATING GUIDELINE

Foil Preparation

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STANDARD OPERATING GUIDELINE

Foil Preparation

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STANDARD OPERATING GUIDELINE

Foil Preparation

NOMENCLATURE

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STANDARD OPERATING GUIDELINE

Foil Preparation

1. PURPOSE AND SCOPE

This procedure describes the process for Uranium thin foil fabrication for Mo-99 Targets.

2. ENVIRONMENT, SAFETY, AND HEALTH CONCERNS

Follow appropriate RWP. This procedure involves handling of RAD material.

3. PREPARATION: Foil

3.1. Determine enrichment value target

3.1.1. Weigh feed materials as needed to achieve enrichment target (see Figure 1: Enrichment Calculations).

	Enrichment	Mass (g)
HEU	93.155%	15.1
DU	0.192%	184.9
Target LEU	7.21%	200

Figure 1: Enrichment Calculations

3.2. Casting

3.2.1. Cast billet of uranium with VIM or Arc melter.

3.2.2. Record casting technique (VIM or Arc).

3.2.3. Record casting ID/Name.

3.2.4. Hot roll using salt bath between 600° C and 700° C until thickness of between 0.0080" and 0.00120".

3.3. Cold Roll

NOTE Cold rolling is achieved using Fenn or Cauffiell rolling mill.

3.3.1. Vacuum anneal foils at 600° C and 800° C for 20 minutes to one hour.

3.3.2. Cold roll until material hardens and resists and achieves reduction of between 5% and 40%.

3.3.3. Measure thickness and repeat Steps 3.3.1 through 3.3.3 until target thickness ($120\mu\text{m} \pm 10$) is achieved.

3.3.4. Cut sample of foil for chemical analysis.

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STANDARD OPERATING GUIDELINE

Foil Preparation

4. Rough cut

- 4.1. Cut foils to nominal target size PLUS additional minimum ¼-inch boundary to allow for later shearing.
- 4.2. Measure and record foil dimension in Appendix A.

5. Quench and Anneal

5.1. Quench

- 5.1.1. Wrap foil in tantalum sleeve.
- 5.1.2. Load sample into quench furnace.
- 5.1.3. Evacuate to operating pressure (E-4 torr).
- 5.1.4. Heat furnace to 760° C ($\pm 15^\circ\text{C}$); HOLD for five to 30 minutes.
- 5.1.5. Backfill with Argon to atmospheric pressure.
- 5.1.6. Using trap door, drop quench foils into water.

5.2. Anneal

- 5.2.1. Place foils in vacuum-annealing furnace for 20 minutes at temperature.
- 5.3. Repeat Steps 5.1 through 5.2 for a total of two cycles.

6. Shear (terminal dimension)

- 6.1. Shear to final size as defined in drawing M802224.
- 6.2. Measure and record foil dimensions in Appendix A.

7. Final Cleaning

- 7.1. Clean foils with detergent (e.g., Formula 409®).
- 7.2. Clean with isopropyl alcohol and lint-free cloth.

8. Bag and Transfer

- 8.1. Place foils in clean plastic bag.
- 8.2. Transfer foils to assembly area.

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STANDARD OPERATING GUIDELINE

Foil Preparation

9. PVD Coatings/ PVD/Plate CALPVD OR Nickel Plate

9.1. Cleaning

- 9.1.1. Process plates in electropolisher using phosphoric acid bath.
- 9.1.2. Clean plates using isopropyl alcohol and lint-free cloth.
- 9.1.3. Place plates in Ultrasonic bath of deionized water and Micro™ detergent.
- 9.1.4. Clean with isopropyl alcohol and a lint-free cloth.
- 9.1.5. Measure and record foil dimensions in Appendix B.

9.2. Coatings (Aluminum via PVD or Ni via plating)

- 9.2.1. Load in PVD chambers.
- 9.2.2. Evacuate chamber to < E-05 torr.
- 9.2.3. Backfill chamber with Argon to operating pressure.
- 9.2.4. Run aluminum PVD coating cycle.
- 9.2.5. Backfill with air to atmospheric pressure.
- 9.2.6. Open chamber and flip targets.
- 9.2.7. Repeat Steps 8.2.1 through 8.2.7 once to coat other side of samples.

9.3. Move target to assembly area.

9.4. Per procedure Y/PM-170, Nickel plate assembly an alternative.

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STANDARD OPERATING GUIDELINE

Foil Preparation

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STANDARD OPERATING GUIDELINE

APPENDIX A: Foil Dimensions

APPENDIX A
FOIL DIMENSIONS

Rough Cut Foil Dimensions

				Measured Value			
				T1	_____	in	
				T2	_____	in	
				T3	_____	in	
				T4	_____	in	
				T5	_____	in	
				T6	_____	in	
				T7	_____	in	
				T8	_____	in	
				T9	_____	in	
				W1	_____	in	
				W2	_____	in	
				W3	_____	in	
				W4	_____	in	
				L1	_____	in	
				L2	_____	in	
				L3	_____	in	
				L4	_____	in	

Instrument(s) Used _____

SAP # _____

Cal Date _____

Personnel Measuring

Initials _____

Badge # _____

Date _____

Personnel Verifying

Initials _____

Badge # _____

Date _____

Foil ID _____

Mass _____ g

STANDARD OPERATING GUIDELINE

APPENDIX A: Foil Dimensions

APPENDIX B (cont.)

Final Cut Foil Dimensions

Measured Value

T1 _____ in
T2 _____ in
T3 _____ in
T4 _____ in
T5 _____ in
T6 _____ in
T7 _____ in
T8 _____ in
T9 _____ in

W1 _____ in
W2 _____ in
W3 _____ in
W4 _____ in

L1 _____ in
L2 _____ in
L3 _____ in
L4 _____ in

Instrument(s) Used

SAP # _____

Cal Date _____

Personnel Measuring

Initials _____

Badge # _____

Date _____

Personnel Verifying

Initials _____

Badge # _____

Date _____

Foil ID _____

Mass _____ g

STANDARD OPERATING GUIDELINE

APPENDIX A: Foil Dimensions

APPENDIX B (cont.)

Post PVD Foil Dimensions

The diagram shows a 3x3 grid of squares. Each square contains a circular measurement point labeled T1 through T9. T1 is at the top-left, T2 is top-middle, T3 is top-right, T4 is middle-left, T5 is center, T6 is middle-right, T7 is bottom-left, T8 is bottom-middle, and T9 is bottom-right. To the right of the grid, four circular measurement points are labeled W1, W2, W3, and W4, each connected by a line to the right edge of the grid. Below the grid, four circular measurement points are labeled L1, L2, L3, and L4, each connected by a line to the bottom edge of the grid.

Instrument(s) Used _____
SAP # _____
Cal Date _____

Measured Value

T1 _____ in
T2 _____ in
T3 _____ in
T4 _____ in
T5 _____ in
T6 _____ in
T7 _____ in
T8 _____ in
T9 _____ in

W1 _____ in
W2 _____ in
W3 _____ in
W4 _____ in

L1 _____ in
L2 _____ in
L3 _____ in
L4 _____ in

Personnel Measuring

Initials _____
Badge # _____
Date _____

Personnel Verifying

Initials _____
Badge # _____
Date _____
Foil ID _____

Mass _____ g

D-6 Y-12 TARGET-FABRICATION GUIDE

Babcock & Wilcox Technical Services Y-12, LLC Management Requirements	System Operating Procedure Title: Assembling LEU-Metal-Foil Annular Targets	Number: Y/PM-167 Revision: 0 Supersedes: NEW Page: 1 of 11 Use Category: REFERENCE
---	---	--

Responsible Organization/Department		
Responsible Manager		
_____ Signature		_____ Date
Approved for Practice		
_____ Signature		_____ Date
Verified to be the latest revision		Effective Date
_____ Initials		_____ Date
Concurrence by the following is documented in the Procedure History File:		
Program Manager	Procedures	Development logistics, packaging,
Project Manager	Foundry operations	measurement evaluator
Schedule and logistics manager	Foundry operations	Leak test designer and operator

Revision Number	Modification Description	Affected Page(s)
0 – NEW	New operating guideline detailing instructions for assembly.	ALL

This procedure has been reviewed by a Derivative Classifier and an UCNI Reviewing Official and has been determined to be UNCLASSIFIED and contains no UCNI. This review does not constitute clearance for public release.

Reviewed by: _____

Date: _____

This document has been reviewed by a CNS Dual Authority DC/RO and confirmed to be UNCLASSIFIED.

Name: Amanda Crumby

Date: 07/26/2016

A. Crumby

Digitally signed by Amanda B (46V) Crumby
DN: cn=US, o=U.S. Government,
ou=Department of Energy, ou=Y-12 National
Security Complex, ou=C&E, ou=people,
cn=Amanda B (46V) Crumby
Date: 2016.07.26 16:11:22 -0400

STANDARD OPERATING GUIDELINE

Assembling LEU-Metal-Foil Annular Targets

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STANDARD OPERATING GUIDELINE

Assembling LEU-Metal-Foil Annular Targets

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STANDARD OPERATING GUIDELINE

Assembling LEU-Metal-Foil Annular Targets

1. PURPOSE AND SCOPE

This procedure describes the process for assembly of Aluminum Annular Mo-99 Targets.

2. ENVIRONMENT, SAFETY, AND HEALTH CONCERNS

Follow appropriate RWP. This procedure involves handling of RAD material.

3. PREPARATION:ANNULAR TARGET ASSEMBLY (LEU-Metal-Foil)

NOTE Use of Isopropyl alcohol is required throughout this operating guideline.

3.1. Prepare the Target Parts

3.1.1.Machine the inner and outer tubes per drawing M802224.

3.1.2.Clean-off machining fluids from tubes using standard machine shop method. They will be thoroughly cleaned in later steps.

3.2. Prepare the Uranium Foil for Insertion into Target.

3.2.1.Size the uranium foil

3.2.1.1. Verify the uranium foil meets dimensions in the drawing M802224, or as requested by customer.

3.2.1.2. Place foil on top of a clean, flat surface previously cleaned with isopropyl alcohol.

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STANDARD OPERATING GUIDELINE

Assembling LEU-Metal-Foil Annular Targets

4. Assembly

4.1. Assemble the Target.

4.1.1. Prepare the tubes prior to assembly.

NOTE Previous inspection report measurement may be credited for results as long as the identity of the component is confirmed.

4.1.1.1. Measure the tubes at several places both ID and OD; alternately, validate tube ID numbers and measurement data.

4.1.1.2. Mark the tubes with a target identification code.

4.1.1.3. Verify a longitudinal scribe on the outer tube for the foil gap position, Figure 1.

Figure 1: Target assembly showing scribing mark on tube.

4.1.2. Clean the tubes (use powder-free clean-room gloves to handle tubes to prevent contamination of the weld faying surfaces with particulates).

4.1.2.1. Clean tubes in Potassium Hydroxide (KOH) solution.

4.1.2.2. Rinse in deionized water; wipe with a lint-free cloth and then dry in oven for one to five minutes between 80°C and 120°C.

4.1.2.3. Rinse in Isopropyl alcohol and dry in flowing Argon.

4.1.2.4. Assemble targets within 3 hours after cleaning target tubes or store in vacuum or inert environment.

4.1.3. Assemble the tubes (use powder free clean room gloves to handle tubes to prevent contamination of the weld faying surfaces with particulates).

4.1.3.1. Wrap the uranium foil around the inner tube.

4.1.3.2. Slide the inner tube into the outer tube.

4.1.3.3. Ensure that the foil gap is lined up with the scribed mark on the outer tube.

4.1.3.4. When tubes are approximately 1cm of assembled configuration, wipe the exposed weld faying surfaces with a lint free cloth.

4.1.3.5. Push the inner tube back flush.

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STANDARD OPERATING GUIDELINE

Assembling LEU-Metal-Foil Annular Targets

- 4.1.3.6. Clean the interior of the draw die (Figure 2) with a paper towel or wipe with isopropyl alcohol.
- 4.1.3.7. Coat draw plug and interior of target with graphite (neolube). Take care to avoid coating the ends of the tube.
- 4.1.3.8. Pull a 1.046 inch draw plug through the inner tube, or customer-requested draw plug.
 - a. Figure 2 shows the draw die hardware.
 - b. Figure 3 shows the assembled die with a target and draw plug exposed.
 - c. A cut-away sketch of the draw die assembly is shown in Fig. 4.
 - d. Drawings of the draw die and hardware are given in CMT-D1562 Rev. 01.

Figure 2: Draw Die Hardware

- 4.1.3.9. Clean target lightly with lint-free cloth using no liquids and ensuring that all remaining lubricant within 2cm of each and is removed.

4/5/2014 9:30:18 AM

STANDARD OPERATING GUIDELINE

Assembling LEU-Metal-Foil Annular Targets

Figure 3: Assembled Draw Die with Target and Draw Plug Exposed

Figure 4: Cut Away Sketch of Draw Die Assembly

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STANDARD OPERATING GUIDELINE

Assembling LEU-Metal-Foil Annular Targets

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STANDARD OPERATING GUIDELINE

Assembling LEU-Metal-Foil Annular Targets

APPENDIX A
Diameter and Validation Certifications

ID of Outer Tube	
OD of Inner Tube	
Difference	
ID of Inner Tube	
Sum	
Record ID's	
Draw Plug Size	

RECORD ID'S AND VALIDATE
CERTIFICATIONS

Person responsible

Name of Target

Date Assembled _____

4/5/2014 9:30:18 AM

Document No. Y/PM-167
Date: September, 20 2013
10 of 11

STANDARD OPERATING GUIDELINE

Assembling LEU-Metal-Foil Annular Targets

APPENDIX B
Cleanliness Report for Materials and Components

Job No. _____ Description: _____

Drawing No. _____ Part No. _____

Cleaned in accordance with paragraph(s) _____

Component meets the cleanliness requirements stated above.

Final Acceptance: _____ Date: _____

Y-12 Quality Department

Date:

4/5/2014 9:30:18 AM

STANDARD OPERATING GUIDELINE

Assembling LEU-Metal-Foil Annular Targets

APPENDIX C
Checklist

3.1.1	Machine the inner and outer tubes per drawing M802224		4.1.2.4	Assemble targets within 3 hours after cleaning target tubes or store in vacuum or inert environment.	
3.1.2	Clean-off machining fluids from tubes using standard machine shop method.		4.1.3.1	Wrap the uranium foil around the inner tube.	
3.2.1.1	Verify the uranium foil meets dimensions in the drawing M802224 or customer spec.		4.1.3.2	Slide the inner tube into the outer tube.	
4.1.1.1	Measure the tubes at several places both ID and OD; alternately, validate tube ID numbers and measurement data.		4.1.3.3	Ensure that the foil gap is lined up with the scribed mark on the outer tube.	
4.1.1.2	Mark the tubes with a target identification code.		4.1.3.4	When tubes are approximately 1cm of assembled configuration, wipe the exposed weld faying surfaces with a lint free cloth.	
4.1.1.3	Verify a longitudinal scribe on the outer tube for the foil gap position, Figure 1.		4.1.3.5	Push the inner tube back flush.	
4.1.2.1	Clean tubes in Potassium Hydroxide (KOH) solution.		4.1.3.6	Clean the interior of the draw die (Figure 2) with a paper towel or wipe with isopropyl alcohol.	
4.1.2.2	Rinse in deionized water; wipe with a lint-free cloth and then dry in oven for one to five minutes between 80°C and 120°C.		4.1.3.7	Coat draw plug and interior of target with graphite (neolube). Take care to avoid coating the ends of the tube.	
4.1.2.3	Rinse in Isopropyl alcohol and dry in flowing Argon.		4.1.3.8	Pull a 1.046 inch draw plug through the inner tube, or customer-requested draw plug.	
			4.1.3.9	Clean target lightly with lint-free cloth using no liquids and ensuring that all remaining lubricant within 2cm of each end is removed.	

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D-7 Y-12 TARGET-ASSEMBLY GUIDE

Babcock & Wilcox Technical Services Y-12, LLC Management Requirements	System Operating Procedure Title: Nickel Capsule Assembly	Number: Y/PM-170 Revision: 0 Supersedes: NEW Page: 1 of 7 Use Category: REFERENCE
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Responsible Organization/Department		
	Responsible Manager	
	_____ Signature Date	
	Approved for Practice	
	_____ Signature Date	
Verified to be the latest revision		
_____ Initials	_____ Date	_____ Effective Date
Concurrence by the following is documented in the Procedure History File:		
Program Manager	Procedures	Development logistics, packaging,
Project Manager	Foundry operations	measurement evaluator
Schedule and logistics manager	Foundry operations	Leak test designer and operator

Revision Number	Modification Description	Affected Page(s)
0 – NEW	New operating guideline detailing instructions for assembly for Mo-99 Foil Targets.	ALL

This procedure has been reviewed by a Derivative Classifier and an UCNI Reviewing Official and has been determined to be UNCLASSIFIED and contains no UCNI. This review does not constitute clearance for public release.

Reviewed by: _____

Date: _____

6/27/2016 3:04:58 PM

This document has been reviewed by a CNS Dual Authority DC/RO and confirmed to be UNCLASSIFIED.
Name: Amanda Crumby
Date: 07/26/2016

A. Crumby

Digitally signed by Amanda B (46V) Crumby
DN: cn=US, o=U.S. Government,
ou=Department of Energy, ou=Y-12 National
Security Complex, ou=C&E, ou=people,
cn=Amanda B (46V) Crumby
Date: 2016.07.26 15:55:04 -0400

STANDARD OPERATING GUIDELINE

Nickel Capsule Assembly

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7. Assembly Loading.....	4
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STANDARD OPERATING GUIDELINE

Nickel Capsule Assembly

1. PURPOSE AND SCOPE

This procedure describes the process for assembling the Mo-99 Capsule Assembly.

2. ENVIRONMENT, SAFETY, AND HEALTH CONCERNS

Follow appropriate RWP. This procedure involves handling of RAD material.

3. INTRODUCTION

The Global Threat Reduction Initiative (GTRI) Mo-99 High Density Target (HDT) project objective is to demonstrate a LEU target which is able to provide similar or greater Mo-99 yields per target than the existing dispersion HEU based targets currently used to provide over 90% of the world's Mo-99. A key aspect of this technology is the ability to roll thin uranium bare metal foils accurately and consistently, in addition, improved chemical dissolution methods are under development to enable the chemical extraction of the Mo-99 from the irradiated targets. To allow for the testing of the chemical processes in an economical fashion, a rabbit based capsule has been developed for irradiation in the High Flux Isotope Reactor (HFIR) that will allow for foils to be irradiated by HFIR and subsequently dissolved in the chemical processing equipment installed in the Oak Ridge National Lab (ORNL) hot cells. This document serves to describe the nickel capsule assembly procedure.

4. Bill of Materials

Receive nickel capsules, springs, and mandrel from Oak Ridge National Laboratory (ORNL). Natural uranium foils shall be produced per Y/PM-166 "Preparation of Uranium Foils for Ni Capsule Assembly."

Uranium Foil Lot ID: _____

Net weight: _____

Uranium weight: _____

U-235 weight: _____

Sign: _____ Date: _____

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STANDARD OPERATING GUIDELINE

Nickel Capsule Assembly

5. Cleaning

5.1.1. Clean the tubes (use powder-free clean-room gloves to handle tubes).

5.1.1.1. Clean ultrasonically with isopropyl alcohol and dry in flowing Argon.

5.1.1.2. Place tubes in clean plastic bag until assembly.

6. Foil Preparation

Induce curving in uranium foil by wrapping the foil around mandrel provided by ORNL.

7. Assembly Loading

Use forceps to compress nickel spring and wrap uranium foil around the spring. Insert into nickel capsule, aligning the gap in the spring with the gap in the capsule.

Alternatively, pre curve uranium foil and insert into capsule. Use forceps to compress nickel spring from center of tabs and insert into capsule, aligning the gap in the spring with the gap in the foil.

Sign: _____ Date: _____

8. Welding

Weld capsule per Y/DV-2294, "Welding Procedure for HFIR Target Irradiation Nickel Capsules."

Sign: _____ Date: _____

Weld Inspection:

Sign: _____ Date: _____

6/27/2016 3:04:58 PM

STANDARD OPERATING GUIDELINE

Nickel Capsule Assembly

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Document No. : Y/PM-170
Date: June 27, 2016
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STANDARD OPERATING GUIDELINE

Nickel Capsule Assembly

APPENDIX A
ASSEMBLY LOG

Person responsible _____

Name of Target _____

Date Assembled _____

Weight of LEU Foil _____ g

Weight of LEU +Coating _____ g

after trimming

Weight of Coating _____ g

Comment [GSD1]: Move to foil procedure.
Moved from Annular Target procedure per roundtable markup.

6/27/2016 3:04:58 PM

STANDARD OPERATING GUIDELINE

Nickel Capsule Assembly

APPENDIX B
Checklist

5.1.1.1	Clean ultrasonically with isopropyl alcohol and dry in flowing Argon.	<div></div>	7.0	Assembly Loading (Complete per procedure)	<div></div>
5.1.1.2	Place tubes in clean plastic bag until assembly.	<div></div>	8.0	Weld capsule per Y/DV-2294, "Welding Procedure for HFIR Target Irradiation Nickel Capsules. (Complete per procedure)	<div></div>

6/27/2016 3:04:58 PM

D-8 Y-12 TARGET-WELDING GUIDE

Babcock & Wilcox Technical Services Y-12, LLC Management Requirements	System Operating Procedure Title: Weld: Aluminum Annular Mo-99 Target	Number: Y/PM-168 Revision: 0 DRAFT 1/8/14 Supersedes: NEW Page: 1 of 15 Use Category: REFERENCE
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Responsible Organization/Department																	
Responsible Manager																	
_____ Signature		_____ Date															
Approved for Practice																	
_____ Signature		_____ Date															
Verified to be the latest revision		Effective Date															
_____ Initials	_____ Date																
Concurrence by the following is documented in the Procedure History File: <table> <tr> <td>Program Manager</td> <td>Procedures</td> <td>Development logistics, packaging,</td> </tr> <tr> <td>Project Manager</td> <td>Foundry operations</td> <td>measurement evaluator</td> </tr> <tr> <td>Schedule and logistics manager</td> <td>Foundry operations</td> <td>Leak test designer and operator</td> </tr> <tr> <td></td> <td></td> <td>Welder</td> </tr> <tr> <td></td> <td></td> <td>Weld Inspector</td> </tr> </table>			Program Manager	Procedures	Development logistics, packaging,	Project Manager	Foundry operations	measurement evaluator	Schedule and logistics manager	Foundry operations	Leak test designer and operator			Welder			Weld Inspector
Program Manager	Procedures	Development logistics, packaging,															
Project Manager	Foundry operations	measurement evaluator															
Schedule and logistics manager	Foundry operations	Leak test designer and operator															
		Welder															
		Weld Inspector															

Revision Number	Modification Description	Affected Page(s)
0 – NEW	New operating guideline detailing instructions for welding.	ALL

This procedure has been reviewed by a Derivative Classifier and an UCNI Reviewing Official and has been determined to be UNCLASSIFIED and contains no UCNI. This review does not constitute clearance for public release.

Reviewed by: _____

Date: _____

This document has been reviewed by a CNS Dual Authority DC/RO and confirmed to be UNCLASSIFIED.

Name: Amanda Crumby

Date: 07/26/2016

A. Crumby

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DN: cn=US, o=U.S. Government,
ou=Department of Energy, ou=Y-12 National
Security Complex, ou=C&E, ou=people,
cn=Amanda B (46V) Crumby
Date: 2016.07.26 16:57:40 -0400

Title: Weld: Aluminum Annular Mo-99 Target	Number: Y/PM-168 Revision: 0 DRAFT A Supersedes: NEW Page: 2 of 15 Use Category: REFERENCE
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1. PURPOSE AND SCOPE

This procedure describes the process for welding of Aluminum Annular Mo-99 Targets.

By virtue of macro sections of each weld and hydrostatic performance testing by the customer, the welding procedure is considered qualified. This also qualifies the welding operator/welder accordingly.

2. ENVIRONMENT, SAFETY, AND HEALTH CONCERNS

Follow appropriate RWP. This procedure involves handling of RAD material.

3. PREPARATION:WELD

3.1. Dress both ends of tubes to flush and machine to specifications in drawing M802224.

3.2. Prepare targets for welding.

3.2.1.Machine target to correct length (per drawing M802224 and the weld procedure)
facing off equal amounts of material from both ends to create flat parallel surfaces.

Figure 1: Machining the end of the tubes.

3.3. Verify that tube ends are flush.

3.4. Avoid fee liquids at ends AND clean exterior of target with a damp (acetone or alcohol) lint-free cloth.

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4. Electron Beam Welding (EBW) Technique

4.1. Preweld Checks and Set-up

- 4.1.1. Check joint runout per Y/DV-2316, Section 5.B while adjusting travel speed.
- 4.1.2. Set welding parameters per Y/DV-2316 on target block (see Table 1).
- 4.1.3. Set timer to make a short pulsed spot weld.
- 4.1.4. Perform final beam alignment check using spot weld on target.

4.2. Alignment Tack

- 4.2.1. Optically align beam on the joint and check using a short pulsed spot weld.
- 4.2.2. Spot tack as necessary to align beam on joint.

4.3. Welding

- 4.3.1. After aligning is completed, perform the following:
 - 4.3.1.1. Set Rise and Fall times.
 - 4.3.1.2. Start rotation.
- 4.3.2. After approximately one revolution, start weld.
- 4.3.3. Manually guide weld on joint, as necessary.
- 4.3.4. After approximately 2.2 revolutions, stop weld.
- 4.3.5. After welding, visually examine through optics.

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5. Notch, Helium (He) Purge, Gas Tungsten Arc Welding (GTAW)

- 5.1. Move fully welded targets to high purity helium glovebox.
- 5.2. Inside the glove box, machine/Dremel (1/8" carbide mill) a small notch at the scribe line.
Target vacuum draws in helium.

Figure 2: Scribe line, GTAW Notch, groove and EB-Weld

- 5.3. Move notch target back to glovebox vestibule to perform backfill.
 - 5.3.1. Vacuum to 2×10^{-2} TORR, refill with +15psi helium.
 - 5.3.1.1. Perform 5.3.1 three times.
 - 5.3.2. Move target back to glovebox.

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Title: Weld: Aluminum Annular Mo-99 Target	Number: Y/PM-168 Revision: 0 DRAFT A Supersedes: NEW Page: 7 of 15 Use Category: REFERENCE
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5. Notch, Helium (He) Purge, Gas Tungsten Arc Welding (GTAW) (cont.)

- 5.4. GTAW weld the notch closed per Procedure Qualification Record (PQR) for annular (Y/DV-2285).
 - 5.4.1. Rollover at notch is permissible to 0.030 inches.
 - 5.4.2. Filing of excess rollover is permissible.
 - 5.4.3. Weld will be inspected by independent inspector.
 - 5.4.4. Welds to be qualified by metallurgical section and ORNL-performed burst test.
 - 5.4.5. Acceptable results automatically qualify the welding operator.
 - 5.4.6. ORNL has the option to witness the weld, inspect, and review results and sign off.
- 5.5. Place and secure target in small volume leak-test canister and remove from glovebox.

6. Perform Leak Test per Y/PM 169, *Leak Detection for Mo-99 Foil Capsule Target*.

7. Cleaning

- 7.1. Wearing powder-free gloves, wipe using lint-free cloth soaked in isopropyl alcohol.
- 7.2. Place in plastic bag.

8. Pack

- 8.1. Contact RADCON to check tube for removable contamination.
- 8.2. Weigh welded tube and record value.
- 8.3. Place tube inside labeled (part number and gross, net and tare weight) plastic zip-top bag.

9. REFERENCE DOCUMENTS

- Y/DV-2285, *Welding Procedure for HFIR Annular Aluminum Irradiation Targets*.

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10. Specifications

PROCESS	Electron Beam
MATERIAL	
Base Metal:	6061 Aluminum
Filler Metal:	None
EQUIPMENT	
Power Supply:	Leybold-Heraeus 7.5 kW Electron Beam Welder
Gun Type:	CL-R167-R (P/N 12G0758588 or 12G080022)
Filament:	Ribbon filament (P/N 12G0749750)
Fixtures:	
Outer Chill Ring:	Copper
Stainless steel or copper target holder.	
Tungsten target material (Stores catalog # 07-589-7855) or equivalent.	
Welding Positioner	
Rotary-type positioner with 3-jaw chuck	
Rotation 0-15 rpm speed range	
POSITION	
Electron Beam Position:	Aligned with the center of weld joint.
Alignment:	maximum. Horizontal Runout: 8 optical divisions
Target Height:	Set at the same height as the part surface within 1/16-in.
Reference Position at Weld Start:	None
JOINT DESCRIPTION	
Joint Geometry:	Edge-type joint (Fig. 1)
Joint Design:	Two autogenous passes
JOINING DETAILS	
Welding Parameters:	Table I
End Load:	N/A

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Title: Weld: Aluminum Annular Mo-99 Target	Number:	Y/PM-168
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Table 1

Welding Parameters for Annular Target

(11-in. Work Distance)

Kilovolts	90 ± 1
Milliamperes	3.5 ± 0.5
Travel Speed (sec/rev)	10 ± 0.5
Travel Speed (rpm)	6 ± 0.3
Sharp Focus Setting (mA)	614 (Ref.) at 3 mA
Weld Focus Setting	Sharp + 10 (624)
Chamber pressure (mbar)	$<8 \times 10^{-5}$
Filament Current	Peak
Deflection Mode	None (Spot)
Rise Time (sec)	0
Fall Time (sec)	2
Overlap Time (sec)	2

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Title: Weld: Aluminum Annular Mo-99 Target	Number: Y/PM-168
	Revision: 0 DRAFT A
	Supersedes: NEW
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Figure 3: Joint Geometry for Annular Target

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Title: Weld: Aluminum Annular Mo-99 Target	Number: Y/PM-168 Revision: 0 DRAFT A Supersedes: NEW Page: 11 of 15 Use Category: REFERENCE
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APPENDIX A
Assembled Annular Target Measurements

Assembled Annular Target Measurements

End A

End B

Overall length
Measure 3 plcs
both ends

OD
Measure 3 plcs

ID
Measure 3 plcs
both ends

Thickness
Measure 3 plcs
both ends

Overall Length
Before Welding

L1 _____ in

L2 _____ in

L3 _____ in

After Welding

L1 _____ in

L2 _____ in

L3 _____ in

Measure OD

OD1 _____ in

OD2 _____ in

OD3 _____ in

Pre/Post Weld End Thickness

A-T1 _____ / _____ in

A-T2 _____ / _____ in

A-T3 _____ / _____ in

B-T1 _____ / _____ in

B-T2 _____ / _____ in

B-T3 _____ / _____ in

Measure ID Post Weld

A-ID1 _____ in

A-ID2 _____ in

A-ID3 _____ in

B-ID1 _____ in

B-ID2 _____ in

B-ID3 _____ in

Final Target Mass _____ g

Instrument(s) Used _____

SAP # _____

Cal Date _____

Personnel Measuring

Initials _____

Badge # _____

Date _____

Personnel Verifying

Initials _____

Badge # _____

Date _____

Target ID _____

Title: Weld: Aluminum Annular Mo-99 Target	Number: Y/PM-168
	Revision: 0 DRAFT A
	Supersedes: NEW
	Page: 14 of 15
	Use Category: REFERENCE

APPENDIX C
Checklist

3.1 Dress both ends of tubes to flush and machine to specifications in drawing M802224.			4.3 Welding	
3.2.1 Machine target to correct length (per drawing M802224 and the weld procedure) facing off equal amounts of material from both ends to create flat parallel surfaces.			5.1 Move fully welded targets to high purity helium glovebox.	
3.3 Verify that tube ends are flush.			7.1 Wearing powder-free gloves, wipe using lint-free cloth soaked in isopropyl alcohol.	
3.4 Avoid fee liquids at ends AND clean exterior of target with a damp (acetone or alcohol) lint-free cloth.			7.2 Place in plastic bag.	
4.1 Preweld Checks and Set-up.			8.2 Weigh welded tube and record value.	
4.2 Alignment Tack			8.3 Place tube inside labeled (part number and gross, net and tare weight) plastic zip-top bag.	

Title: Weld: Aluminum Annular Mo-99 Target	Number:	Y/PM-168
	Revision:	0 DRAFT A
	Supersedes:	NEW
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Appendix D
GTAW Seal Weld Procedure Qualification Record

GTAW Seal Weld Procedure Qualification Record		POR#: CAH-2014-1
Reference Document: Y/PM-168 Applicable Code: AWS B2.1		
Date: <u>1/29/2014</u>		
Program: <u>Annular Mo-99 Target</u>		
Work Request: <u>N/A</u>		
PIN: <u>M802224-0001 & 0002</u>		
Name: <u>C. A. Hayes</u>		
Equipment S/N:	<u>n/a</u>	Machine Type: <u>Miller Synchrowave</u>
Joint Geometry: <u>Edge Joint Seal Weld (notch re-seal)</u>		
Base Material: <u>Aluminum 6061 -T4</u>		
Electrode Material: <u>0.040" Pure Tungsten (Ball End)</u>		
Shielding Gas:	<u>Helium w/Argon torch</u>	Flow Rate: <u>15 CFH</u>
Filler Metal: <u>ER-4043 Al, 3/64" dia.</u>		
Preheat: <u>68°F</u>		
Process: <u>Manual GTAW, Single Pass, Stringer</u>		
Position: <u>Flat</u>		
Thickness: <u>5/32" max.</u>		
COMMENTS: <u>Cleaning with alcohol or acetone is permitted. Qualified by visual and macro-sections</u> <u>Hydrostatic testing to be performed by ORNL</u>		
WELDING CHARACTERISTICS		
	Initial Pass	Remainder
Voltage	<u>n/a</u>	<u>n/a</u>
Current Type	<u>AC</u>	<u>n/a</u>
Polarity	<u>n/a</u>	<u>n/a</u>
Amps (Range)	<u>15-20</u>	<u>n/a</u>
High Frequency	<u>continuous</u>	<u>n/a</u>
Preheat	<u>70°</u>	<u>n/a</u>
Nozzle Size	<u>0.320"</u>	<u>n/a</u>
Electrode Extension	<u>-0.30"</u>	<u>n/a</u>
Pulsing	<u>NO</u>	<u>n/a</u>
TECHNIQUE: <u>With target clamped lightly in vise (approximately 1.5" extending beyond) start arc on Electron</u> <u>beam weld slightly away from notch. Allow puddle to form. Feed filler perpendicular to puddle from</u> <u>slightly below joint. Move to close and fill notch.</u> <u>Visual inspection for cracks, undercut, overlap, voids or porosity.</u>		
Welded by: <u>C. A. Hayes</u> Inspected by: <u>K. D. Nicklas</u>		
		Approved by: <u>[Signature]</u> Section Manager Date: <u>2/29/14</u>

D-9 Y-12 TARGET LEAK-DETECTION GUIDE

Babcock & Wilcox Technical Services Y-12, LLC Management Requirements	System Operating Procedure Title: Leak Detection for Mo-99 Foil Capsule Target	Number: Y/PM-169 Revision: 0 Supersedes: NEW Page: 1 of 4 Use Category: REFERENCE
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Responsible Organization/Department		
	Responsible Manager	
	_____ Signature Date	
	Approved for Practice	
	_____ Signature Date	
Verified to be the latest revision		
_____ Initials	_____ Date	_____ Effective Date
Concurrence by the following is documented in the Procedure History File: Subject Matter Expert		

Revision Number	Modification Description	Affected Page(s)
0 – NEW	New operating guideline detailing instructions for leak detection for Mo-99 Foil.	ALL

This procedure has been reviewed by a Derivative Classifier and an UCNI Reviewing Official and has been determined to be UNCLASSIFIED and contains no UCNI. This review does not constitute clearance for public release.

Reviewed by: _____

Date: _____

10/17/2013 1:48:49 PM

This document has been reviewed by a CNS Dual Authority DC/RO and confirmed to be UNCLASSIFIED.
Name: Amanda Crumby
Date: 07/26/2016

A. Crumby

Digitally signed by Amanda B (46V) Crumby
DN: cn=US, o=U.S. Government,
ou=Department of Energy, ou=Y-12 National
Security Complex, ou=C&E, ou=people,
cn=Amanda B (46V) Crumby
Date: 2016.07.26 15:59:42 -0400

STANDARD OPERATING GUIDELINE

Leak Detection for Mo-99 Foil Capsule Target

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STANDARD OPERATING GUIDELINE

Leak Detection for Mo-99 Foil Capsule Target

1. PURPOSE AND SCOPE

This procedure details the steps necessary to ensure a qualified weld for the Capsule Target.

2. ENVIRONMENT, SAFETY, AND HEALTH CONCERNS

RADCON participation is necessary for this procedure. Follow appropriate RWP.

3. PREPARATION:LEAK DETECTION FOR Mo-99 FOIL

NOTE The Leak Detection system will be calibrated using a certified E-07 std cc/sec helium standard prior to the start of step 1.

3.1. Evacuation

3.1.1. Evacuate the carrier vessel and the foil capsule to a pressure of E-04 torr. One hour of pumping is required to remove air gases and water.

3.2. RADCON Check

3.2.1. After the foil capsule is welded in the helium glove box, it will be removed from the glove box and checked for contamination by Radcon. At this point it will be loaded into the carrier vessel.

3.3. Attaching vessel to leak detection system

3.3.1. The vessel will be attached to the Leak Detection system and pumped down to E-02 torr. This should take only 2 minutes. At this point, the gas flow will be sent to the leak detector for verification that there are no helium leaks greater than E-02 std cc/sec.

3.4. Leak Determination and High Vacuum

3.4.1. IF the carrier vessel foil capsule shows no helium leak greater than E-02 std cc/sec, THEN the gas flow will be pumped by a high vacuum system down to E-06 torr.

3.4.2. After E-06 torr is achieved, the gas flow will be sent to the Leak Detection system. The helium leak rate will be followed until a stable leak rate is established. At this point, the leak rate will be measured for 2 hours.

3.4.3. After the 2 hour measurement, the leak rate will be documented and reported.

4. Equipment-specific Order of Operations

- 4.1. All valves closed except **V6**, connect vessel to leak detection system
- 4.2. Continue to open **V1**, proceed to open **V3, V4, V5** to achieve UHV & pump out all gasses and H₂O from vessel for 1hr. Pressure must pump down to 10^{-4} before removal on gauge#2.
- 4.3. Record vacuum pressure from gauge#2 ____ millitorr & make certain it's 10^{-4}
- 4.4. Close **V1, V3, V4, V5** then remove vessel off vacuum station. Give to weld lab. RADCON to check sample and get approval prior before loading sample into vessel.

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STANDARD OPERATING GUIDELINE

Leak Detection for Mo-99 Foil Capsule Target

- 4.5. Make certain all valves are closed except **V6** (pass through valve)
- 4.6. Attach vessel to leak detection system
- 4.7. Open **V1, V3** to rough out vessel & pump down to 10^{-2} and observe reading on gauge#2- don't exceed 2 minutes
- 4.8. Close **V3**
- 4.9. Open **V7, V4**, observe leak rate on detector for no more than 2 minutes- should not have any leaks greater than 10^{-2}
- 4.10. If no leaks, close **V7**, open **V3, V5**, allow pumping down to 10^{-6} on gauge#1
- 4.11. When 10^{-6} is achieved, immediately close **V5, V3** & open **V7** and let establish a stable leak rate for 2hrs- Record ev. ____ minute(s).
- 4.12. -After 2 hrs, record and document the leak rate on Varian detector. ____

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