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Investigation of Cadmium Combustion Potential

by J. D. Gabor, R. T. Purviance,
J. C. Cassulo, I. Charak, and
V. J. Novick

Argonne National Laboratory, Argonne, Illinois 60439
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INVESTIGATION OF CADMIUM COMBUSTION POTENTIAL

by

J. D. Gabor, R. T. Purviance, J. C. Cassulo, I. Charak, and V. J. Novick*

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IFR TECHNICAL MEMORANDUM NO. 133

Results reported in the IFR-TM series of memoranda
frequently are preliminary and subject to revision.
Consequently they should not be quoted or referenced.

APPLIED TECHNOLOGY

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*Engineering Physics Division.

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INVESTIGATION OF CADMIUM COMBUSTION POTENTIAL

by

J. D. Gabor, R. T. Purviance, J. C. Cassulo,
I. Charak, and V. J. Novick

ABSTRACT

The key step in the Integral Fast Reactor (IFR) fuel re-
processing is electrorefining. The demonstration electrorefiner
design specifies 1000 kg of cadmium in the anode. A scenario
involving an earthquake, in which the Argon Cell containing the
pyrometallurgical facility is breached and the electrorefiner
containment fails, must be considered in terms of the combustion
potential of the cadmium and smoke loading on the facility fil-
ters. Two types of experiments were conducted to address these
issues: one which simulated a breach in the electrorefiner by
pouring molten cadmium at ~500C onto a 5/8-in. thick steel plate
(representative of the cell liner) and the other in which
stagnant molten cadmium was exposed to air. It was concluded
that cadmium at the electrorefiner operating temperature of 500C
poses no combustion hazard either when spilled or as a stagnant
pool when exposed to air. Only 0.0316 g of CdO smoke was
collected on filters for a cadmium spill of 1.71 kg leading to
an inferred Cd release fraction of 1.6×10^{-5}.

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I. INTRODUCTION

The Integral Fast Reactor (IFR) concept is a sodium-cooled pool reactor incorporating passive safety features. The reactor uses metallic fuel, and an on-site pyrometallurgical fuel cycle facility provides proper control of radioactive materials and economical production. The objectives of the IFR fuel reprocessing program at Argonne National Laboratory (ANL) include the demonstration of the operational safety of the fuel reprocessing facility as well as the development of operations and equipment. The program is being carried out at ANL's Idaho National Engineering Laboratory site in the existing Hot Fuel Examination Facility/South (HFEF/S).

The key step in the pyrometallurgical process is the electrorefining in which the fission products are removed and the uranium and plutonium recovered.[1] The demonstration electrorefiner is designed to have a 40-in. diameter with a 6-in. layer of cadmium as the anode. A 12-in. deep pool of 58 mol % LiCl - 42 mol % KCl eutectic salt above the cadmium serves as the electrolyte. For a complete safety analysis there is a need to determine the combustion potential of cadmium released from the electrorefiner in case of a seismic event. The scenario for this event envisions not only failure of the electrorefiner vessel but also the HFEF/S Argon Cell housing the fuel reprocessing equipment. A breach in the Argon Cell would permit air to enter into the cell atmosphere and create the potential for metal fires. Cadmium vapor is toxic, and smoke from a cadmium fire would add to the burden of the filters needed for environmental protection. Therefore, testing of the potential for cadmium combustion and determining the amount of smoke emission generated by cadmium spills at electrorefiner operating conditions are needed for definitive safety assessments.

Laboratory experience at ANL[2] and literature indicate that molten cadmium is not readily combustible. The work by Gruhl and Wasserman[3] has shown that an oxide layer on the surface of molten cadmium pools inhibits the diffusion of oxygen and hence oxidation rates. Grosse and Conway[4] determined that cadmium would ignite at 760C in pure oxygen. This, coincidentally is the boiling point of cadmium at 1 atm[5] (Figure 1). However, it is conceivable that molten cadmium released from a failed vessel could spread rapidly enough
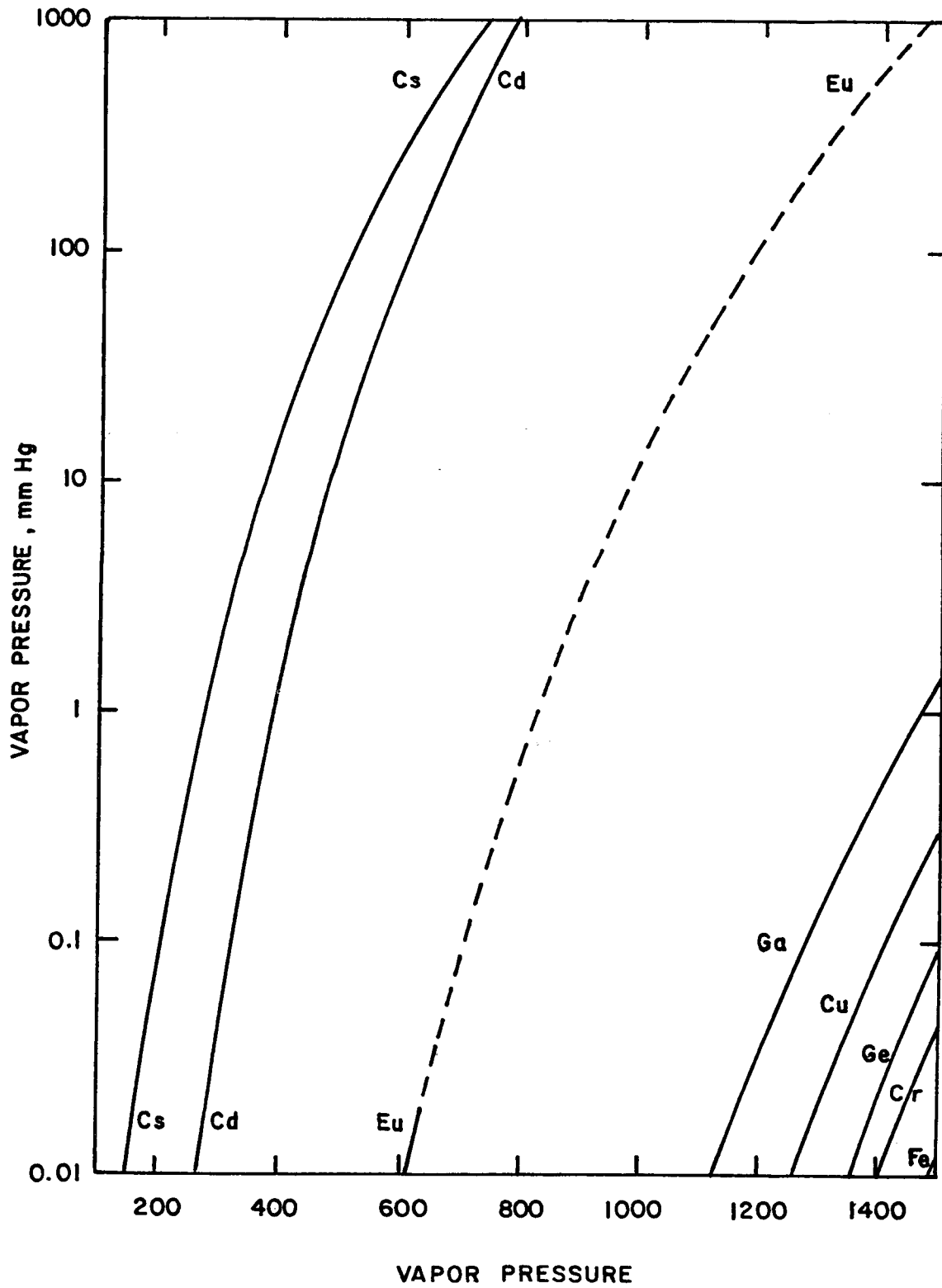
Figure 1. Vapor Pressures of Various Metals

to form a large surface area without a protective oxide layer which could make it susceptible to ignition. On the other hand, cadmium spreading on the cell steel liner would rapidly transfer its heat to the steel and drop in temperature. The steel liner is 1/2-in. thick [(ASTM A-283-54, grade C with a zinc coating (A-93-58T)].

An investigation was therefore undertaken to determine if cadmium combustion is a potential hazard in case of a seismic event. There were two primary goals of this investigation: (1) assessment of the possibility for the cadmium burning under anticipated conditions of a spill and (2) establishment of criteria for the ignition of a molten pool of cadmium, which would be of value in analyzing other conceivable scenarios.

II. EXPERIMENTAL

Two types of experiments were conducted: one which simulated a breach in the electrorefiner vessel that results in spillage of the cadmium onto the steel-lined floor and the other in which a pool of cadmium was heated to different temperatures under an inert gas blanket and then exposed to air. The second type of experiment was conducted to determine if conditions exist for combustion of cadmium confined in the electrorefiner but exposed to air.

A. Test Apparatus

The spill tests were conducted in the vessel (41-mm ID by 203-mm deep) shown on Figure 2 which was located inside a laboratory hood. It had two zones wrapped with 0.09 in. coaxial heating wire and was enclosed in thermal insulation. The lower and upper flanges were wrapped with 0.0625 in. coaxial heating wire. The lower flange had a 25-mm diameter opening sealed by a 0.05-mm stainless steel diaphragm. A pneumatically actuated cutter cut the diaphragm to release the molten cadmium onto the steel plate. The cadmium was heated under an argon atmosphere. Figure 3 is a sketch of the hood and the assembled apparatus. There were four Group II prefilters (15 x 19 x 2 in.) in the back wall of the hood. The exhaust then went through a bank of two HEPA filters (24 x 24 x 12 in.). The gas flow was measured to be 136 ft/min for a 16-in. open window.
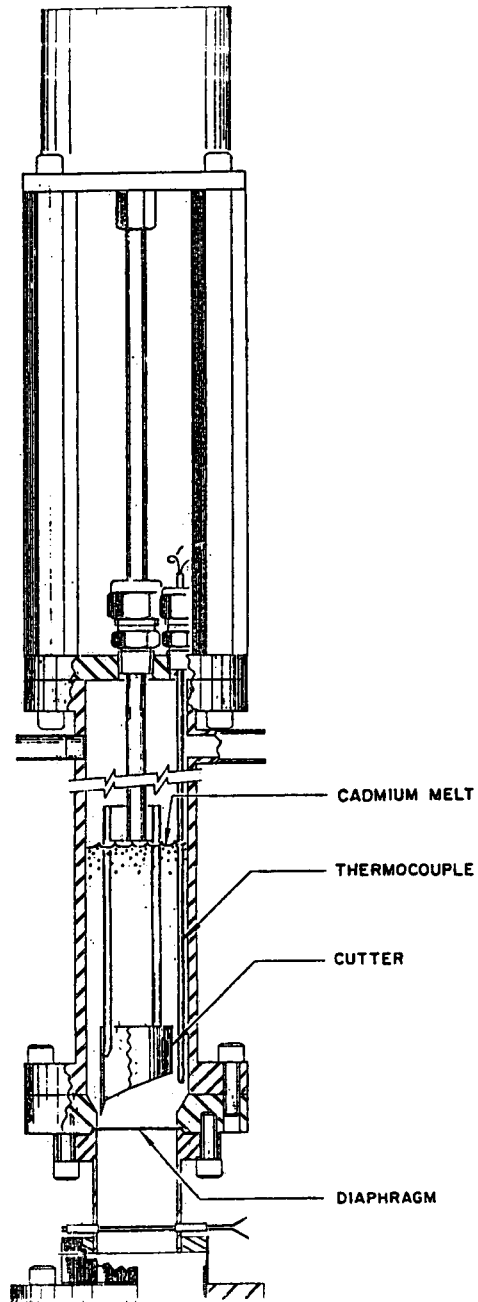
CADMIUM MELT

THERMOCOUPLE

CUTTER

DIAPHRAGM

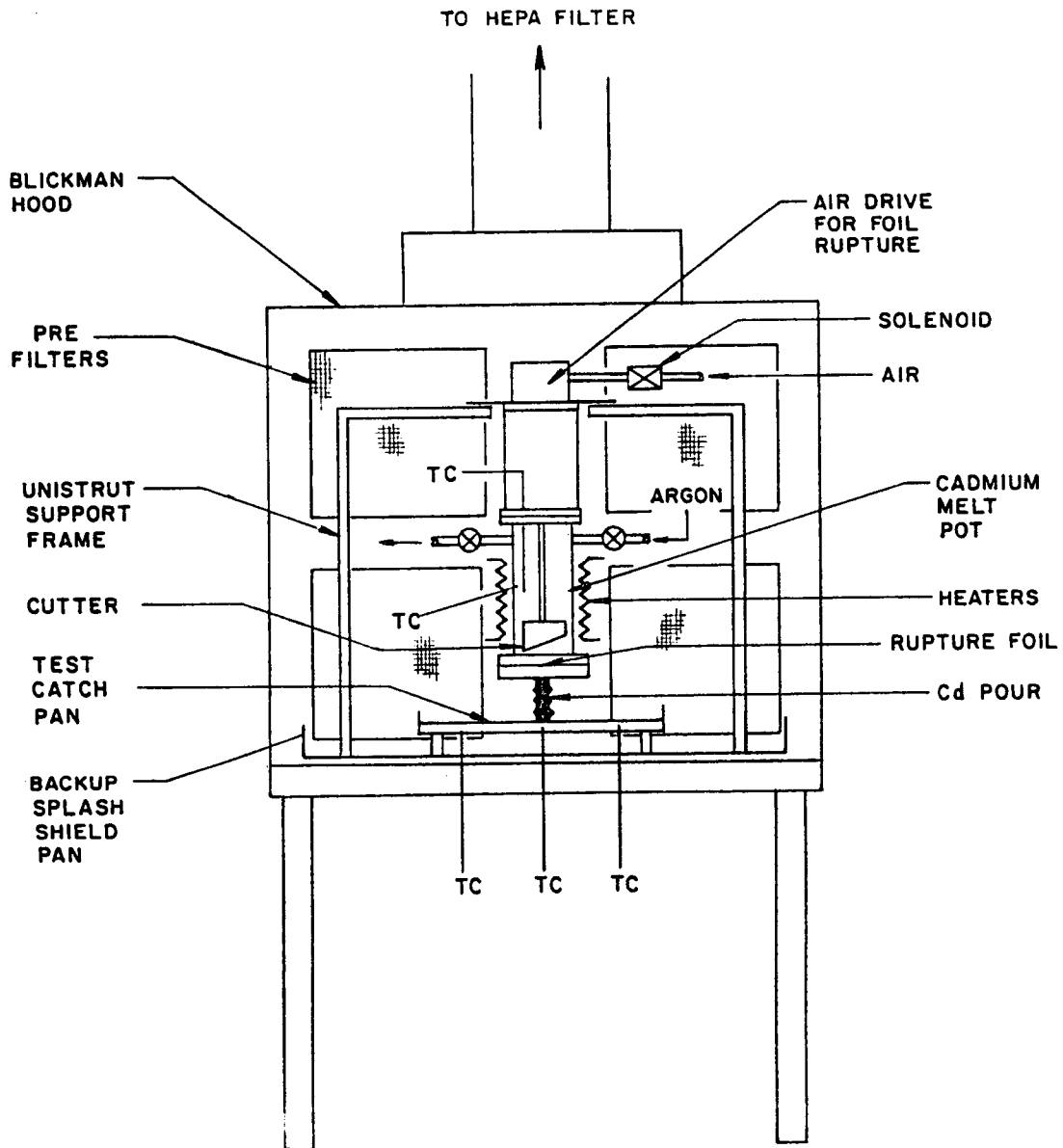Figure 2. Cadmium Melt Vessel for Spill Tests

Figure 3. Apparatus for Cadmium Combustion Experiments

The catch pan had a steel base 5/8-in. thick and an 11-in. by 12-in. surface. Side walls 1/8-in. thick and 1-in. high were welded to the base. The steel for the base was ASTM-36 (0.26C-0.04P-0.05S) which is commercial grade construction material. This is comparable to the 1/2-in. thick steel liner in the Argon Cell which is ASTM A-283-54, grade C with a zinc coating (A-93-58T).

The other test vessel, which was used for simply exposing a pool of molten cadmium to air, was a stainless steel beaker wrapped with coaxial heating wire and insulation. This stainless steel container was 60 mm in diameter and 83 mm deep. The top rim of the beaker was machined off to provide a flat surface for sealing with a flat lid. The lid was pneumatically operated.

B. Instrumentation

Type K thermocouples were used to monitor and control the cadmium melt temperature and to record the temperature response of the steel base plate. Two thermocouples with a stainless steel sheath were in the melt and three were attached to the outside wall of the vessel used for the spill tests. Six thermocouples were located in the steel base plate just below the surface.

One Hewlett-Packard 7132A two-pen strip chart recorder and three Hewlett-Packard 7100B two-pen strip chart recorders were used to record the termperatures of the cadmium melt and the base plate temperature response. Weather Measure TPC-1 Type K temperature controllers actuated the heaters.

A LOCAM (Red Lake Corp., Santa Clara, CA) motion picture camera (10 to 500 pps) and a NAC High Speed Video System (HSV-200) consisting of a Model V-10 color camera and a Model V-30 video tape recorder were used to photograph the experimental events.

III. RESULTS

The experimental procedures and results are described in the order in which they were conducted.

CADD-1

The first of the cadmium drop tests (CADD-1) was a spill of molten cadmium at ~505C onto the 5/8-in. thick steel plate located 6 in. below the vessel. The video tape indicated that there was no ignition at the initial inpact on the base plate by the molten cadmium flow. However, when the cadmium flow splashed against the side walls of the catch pan ignition occurred. Apparently the splashing produced small droplets having a high surface area which rapidly oxidized producing a flare. However, the burning lasted for only 3 sec. The quenching of the cadmium fire is attributed primarily to the rapid heat transfer to the steel base plate, whose temperature did not exceed 107C (see Figure 4). Surface oxide formation also impeded oxygen diffusion to the metal. Flaming of a stalactite of frozen cadmium hanging from the opening of the bottom flange of the melt vessel persisted somewhat longer (1.5 min) after the burning of the main bulk of cadmium ceased. The motion picture camera unfortunately was started after the spill and therefore gave no record at this event.

The temperature response 1/8-in. below the surface of the steel plate after the drop of the 505C cadmium is shown on Figure 4. The maximum temperature of 107C was experienced at the center of the pour impact. The plate eventually equilibrated in the range of 74 to 79C about 100 sec after the pour. At 60 sec the thermocouple 5 in. from the center indicated a temperature rise from 78 to 85C. This late temperature rise is attributed to the heat of combustion of the abbreviated burning.

The hood atmosphere was sampled by cascade impactors and by filters. The cumulative weight per cent of the particles collected in the impactors determined by weighing is given on Figure 5. Histograms of the weight distributions are given in the Appendix. The median particle size was 1.2 µm. Two glass fiber filters in an air sampler in the hood collected
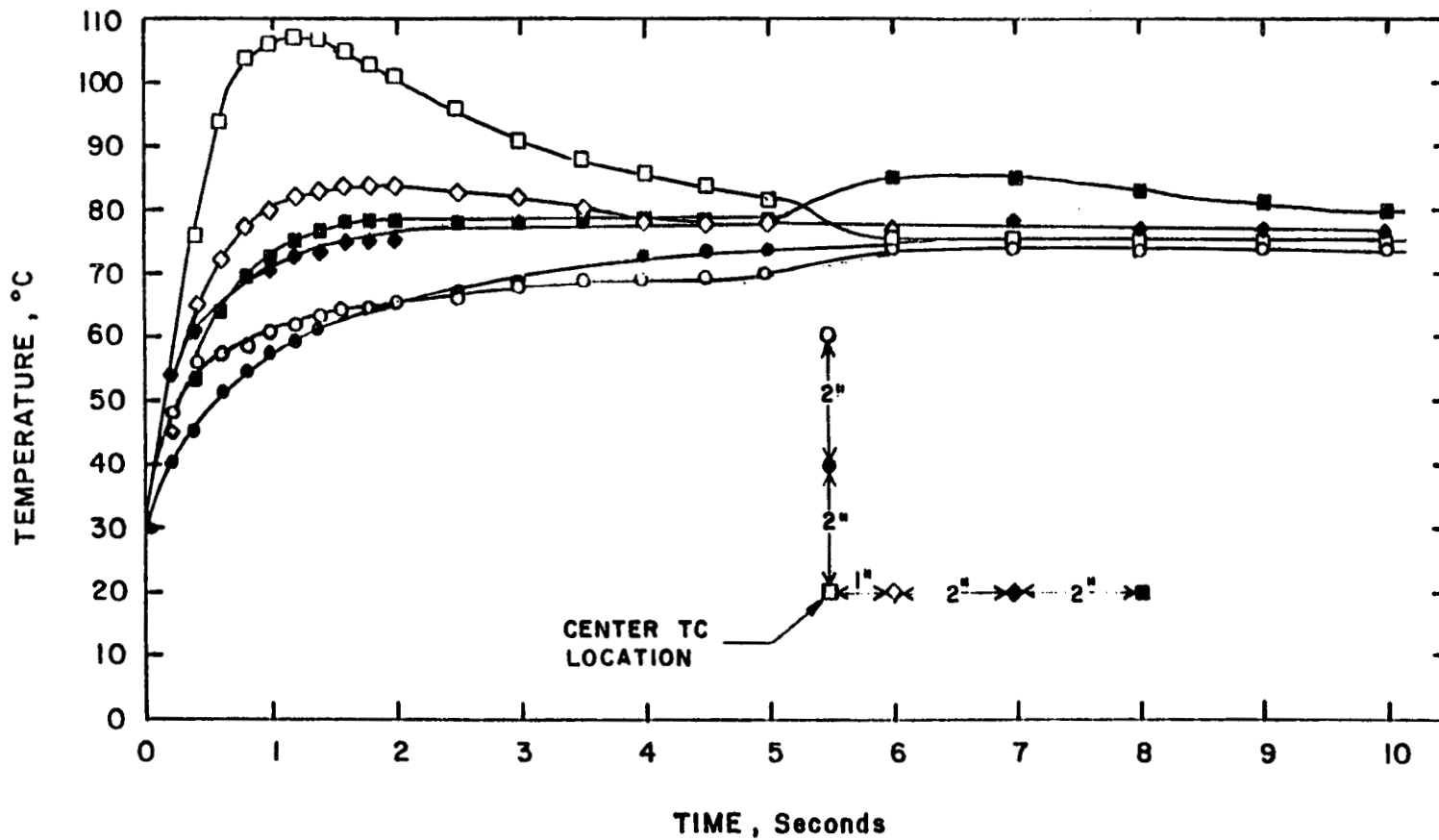
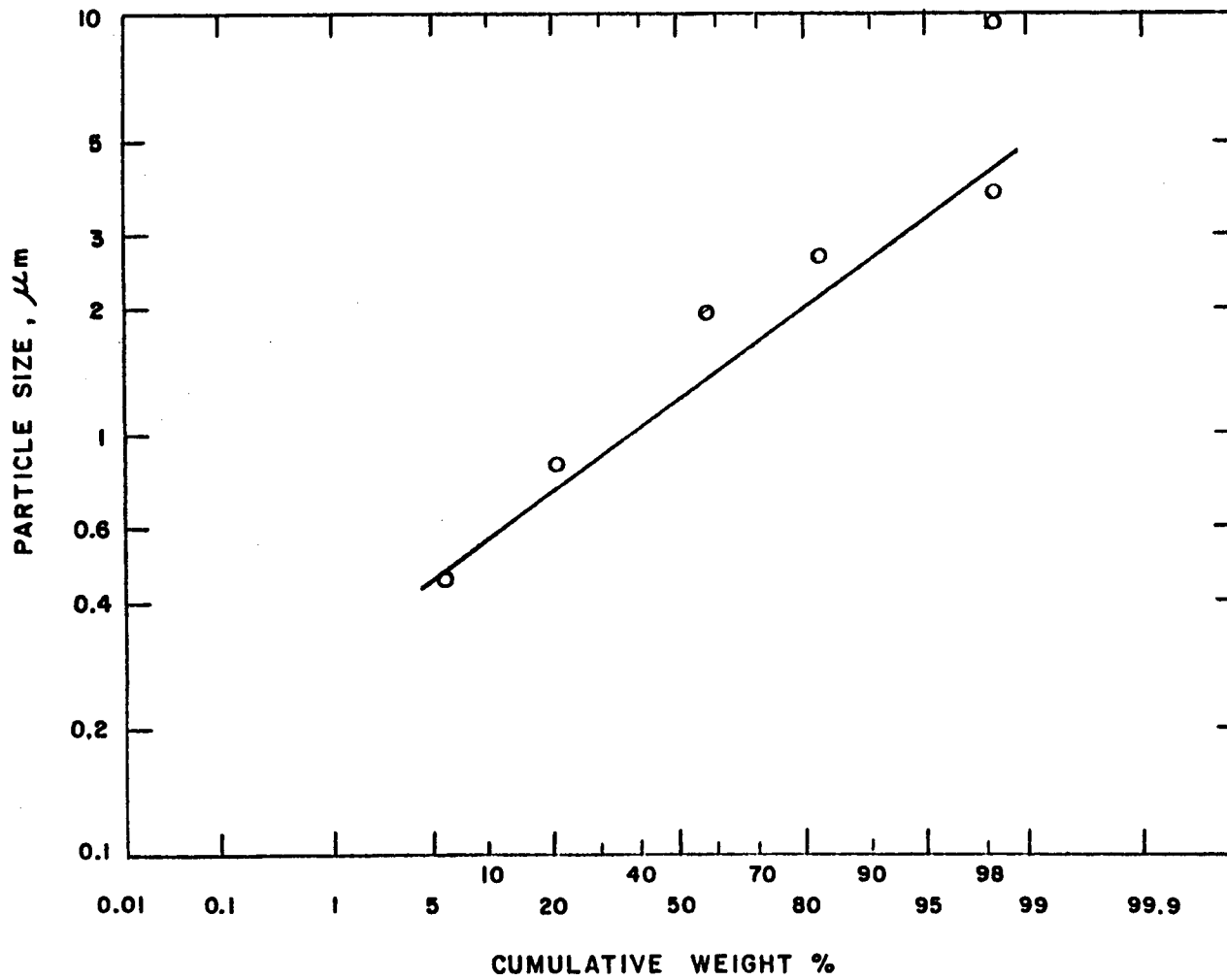Figure 4. Temperature Response of Steel Plate to Cadmium Drop in Test CADD-1

Figure 5. CADD-1 Particle Size Cumulative Distribution

0.7 mg and 1.1 mg of CdO as determined by weighing. Chemical analysis for cadmium confirmed the weights by measuring respectively 0.64 mg Cd (equivalent to 0.731 mg CdO) and 0.923 mg Cd (1.054 mg CdO).

CADD-2

This experiment was conducted to determine the potential for combustion of a stagnant pool of molten cadmium exposed to air. The cadmium was heated to 520C in the 60-mm diameter stainless steel vessel covered with a pneumatically operated lid and purged with argon. The cadmium was 50 mm below the top edge of the container. After the lid was raised and the hot argon cover gas was displaced by the hood air, combustion did not occur. An electric spark subsequently generated by a Tesla coil over the cadmium surface also did not produce ignition. A surface layer of cadmium oxide scum soon formed over the cadmium pool creating a barrier to further oxidation.

CADD-3

A second test was conducted in which a stagnant pool of molten cadmium was exposed to air. In this test the cadmium temperature was increased to 611C for a 100 mm Hg v.p. (see Figure 1) and its level was raised to within 32 mm below the top edge of the container. There was some fuming caused by oxidation of the cadmium vapor after the cover was removed. However, no ignition occurred even when a spark was generated over the cadmium. These tests demonstrated that contained cadmium at the electrorefiner operating temperature of 500C has no potential for spontaneous combustion.

CADD-4

In this test the cadmium was pressurized to 3 psi with argon to simulate the liquid head in the electrorefiner. The pressure was based on a 6-in. depth of cadmium overlaid with 12 in. of LiCl-KCl salt eutectic. The catch pan with the 5/8-in. steel base and 1-in. high side walls was placed upside down to prevent the cadmium flow from splashing against the side walls and forming a spray which would be susceptible to ignition. The gadmium flow ran off over the edge of the steel plate into a secondary container (2-ft by 4-ft pan) on the bottom of the hood.

The cadmium was heated to 521C before the pneumatically operated cutter opened the stainless steel diaphragm. The 5/8-in. thick steel plate was 7-3/4-in. below the cadmium melt vessel to simulate the actual HFEF/S installation. There was no sustained ignition of the cadmium that poured onto the steel plate. With the absence of the side walls the cadmium flowed over the edge without further splashing and quenched. However, after the pour, argon from the cylinder used to pressurize the melt container continued to flow out through the opening in the bottom flange. This flowing gas conducted cadmium vapor from the cadmium still remaining on the hot walls of the melt vessel into the atmosphere. This cadmium vapor oxidized forming a smoke. The smoking persisted for 2.0 min until the argon cylinder was valved off. Aerosols are typically generated by devices using this principle.

The cascade impactor indicated a bimodal distribution of collected particles. Aerosols generated by vapor condensation are typically very small. It is seen from Figure 6 that 62 wt% of the particles were less than 0.84 μm. Larger particles are produced by burning and agglomeration.

CADD-5

Because the aerosol generation in CADD-4 was not the result of combustion or conditions representative of an electrorefiner breach, this test was repeated with the melt vessel pressurized from a 1 liter reservoir to limit the subsequent gas flow after the melt is discharged. The use of the argon reservoir limited the amount of gas flow through the opening in the flange to about 1 sec after the cadmium was discharged. This was evidenced by cessation of smoke generation emanating from the melt vessel.

Figure 7 is a sequence of photographs of the cadmium spill. The photographs were taken from motion picture frames at a film speed of 100 frames per second. Figure 7a shows the apparatus before the spill was initiated by the diaphragm cutter. The catchpan with the 5/8-in. steel base was placed upside down as in CADD-4. The three legs of the pan are seen protruding from the surface. These legs presented negligible interference to the cadmium flow as can be seen in the subsequent photographs. Figure 7b was taken 0.04 sec after the diaphragm was cut. The front of the cadmium pour stream is seen near the top center of the photo. Initial impact of the pour stream is shown on Figure
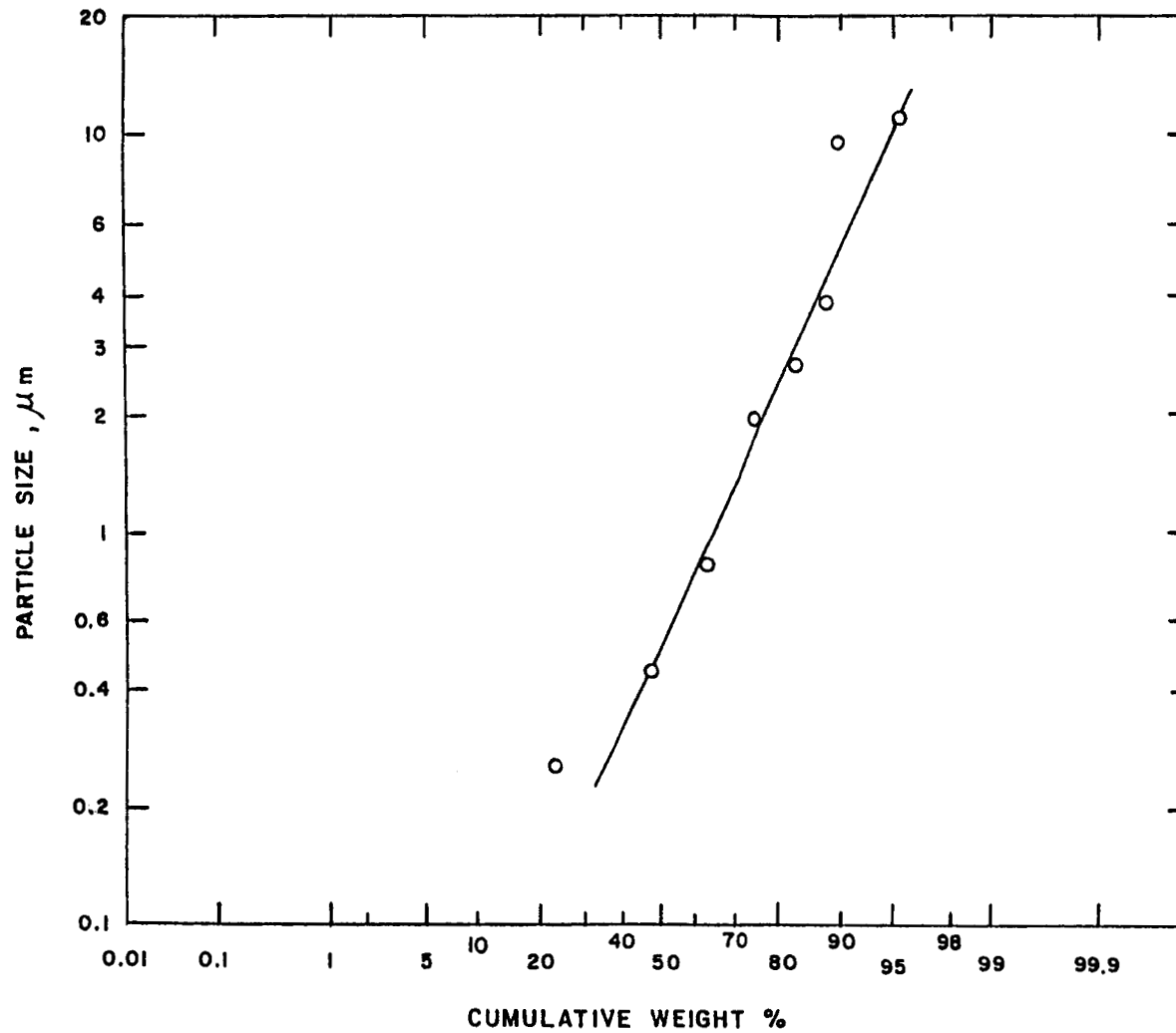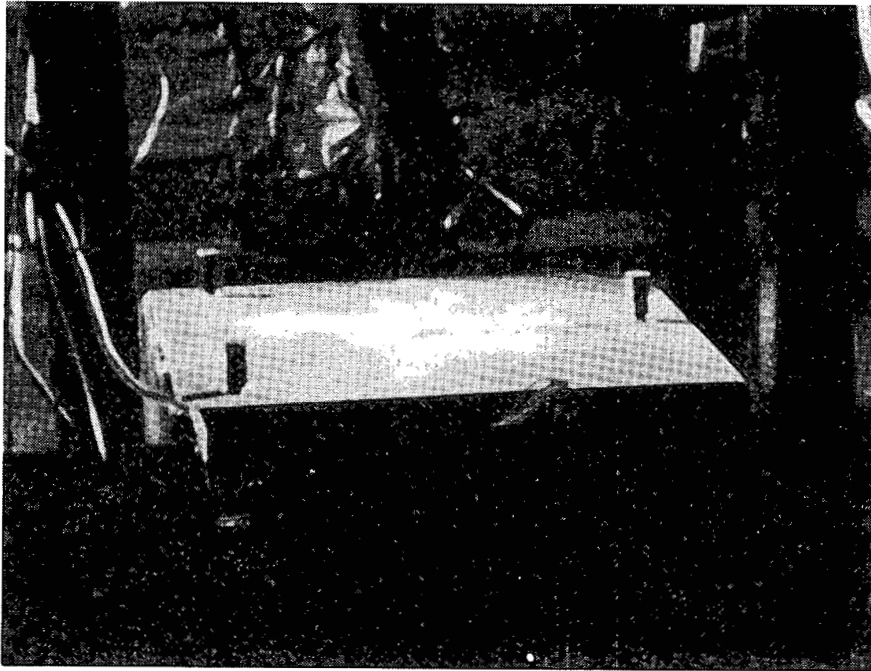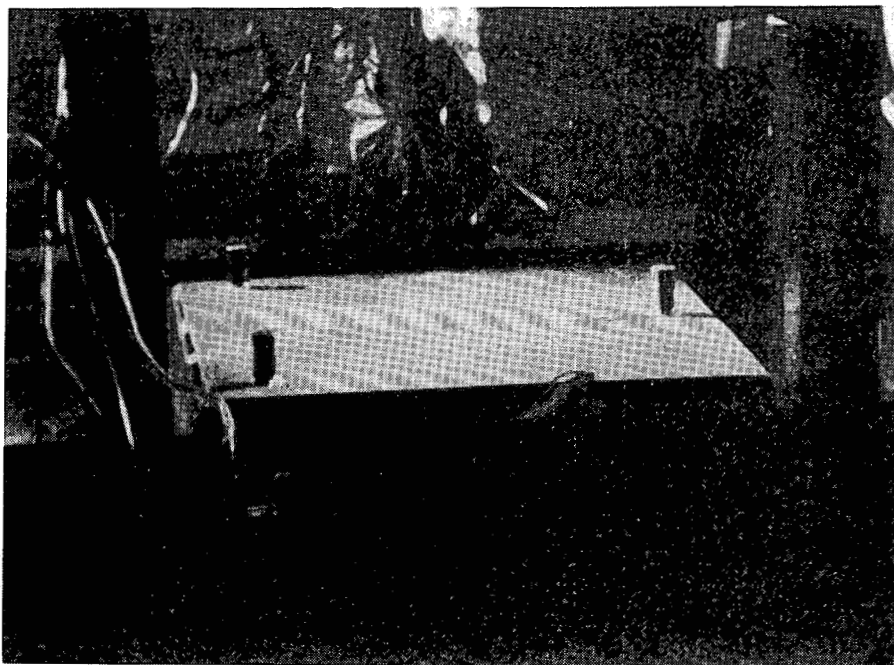
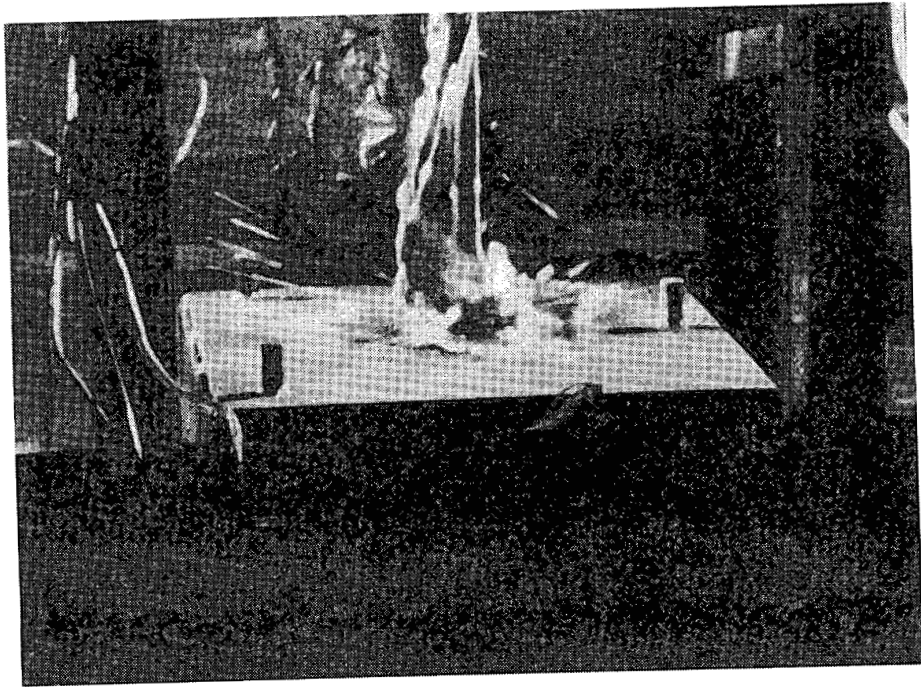Figure 6. CADD-4 Particle Size Cumulative Distribution

a. 0.1 sec before Cd spill (ANL Neg. 7952, Frame 14A)
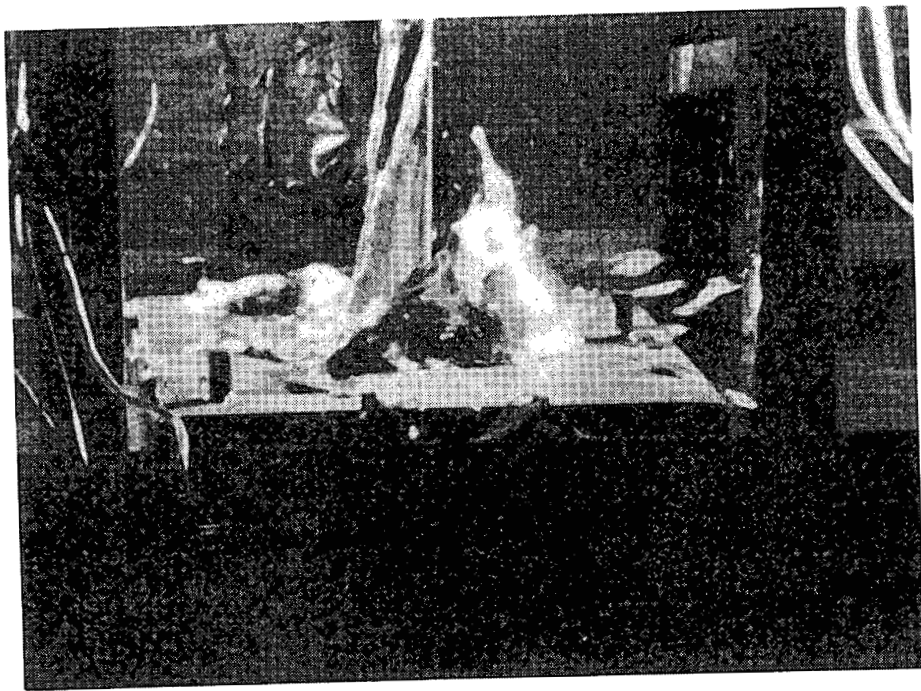

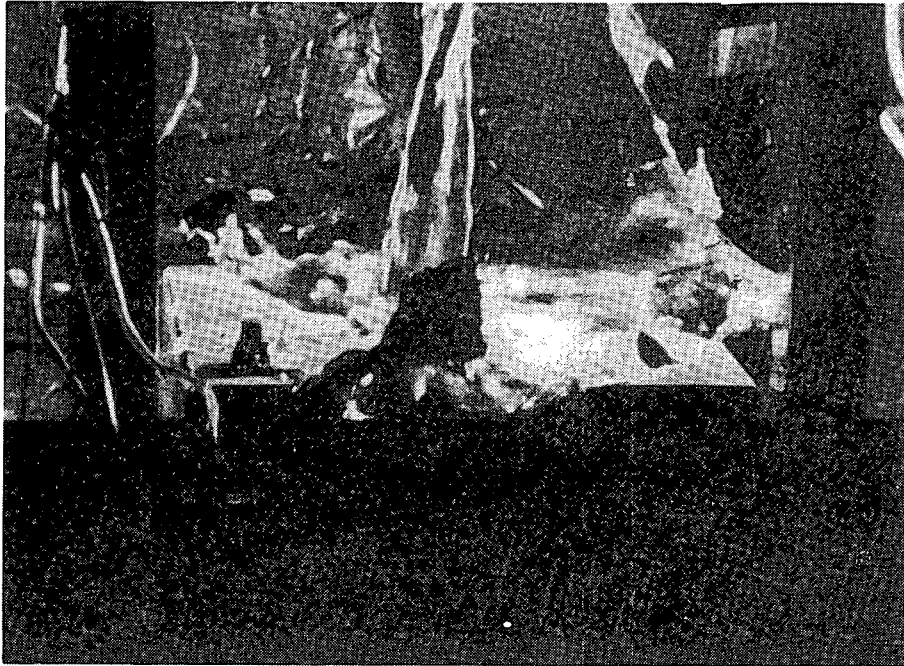b. 0.04 sec (ANL Neg. 7951, Frame 5A)

Figure 7. CADD-5 Cadmium Spill

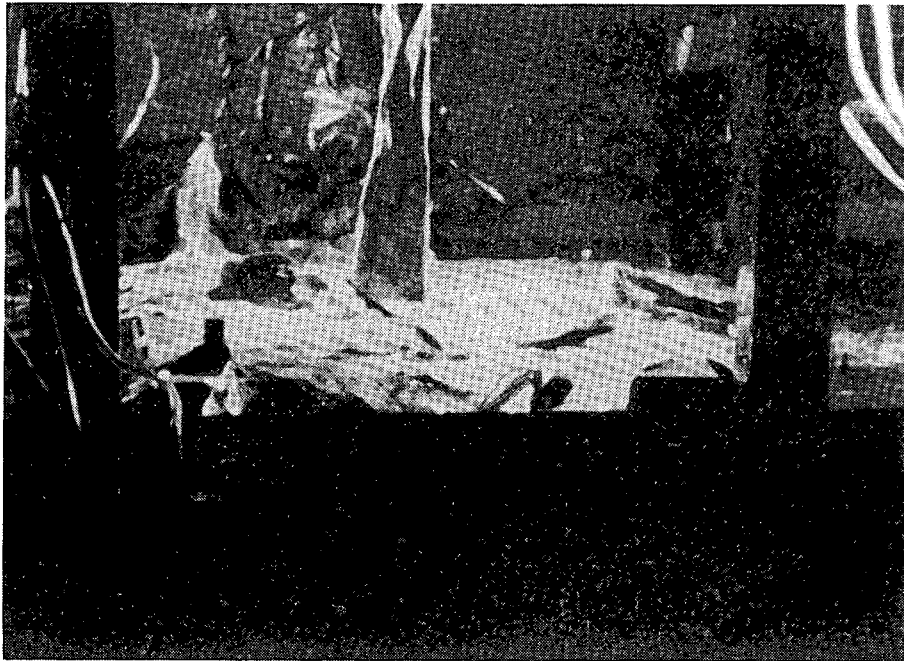c. 0.12 sec (ANL Neg 7951, Frame 8A)
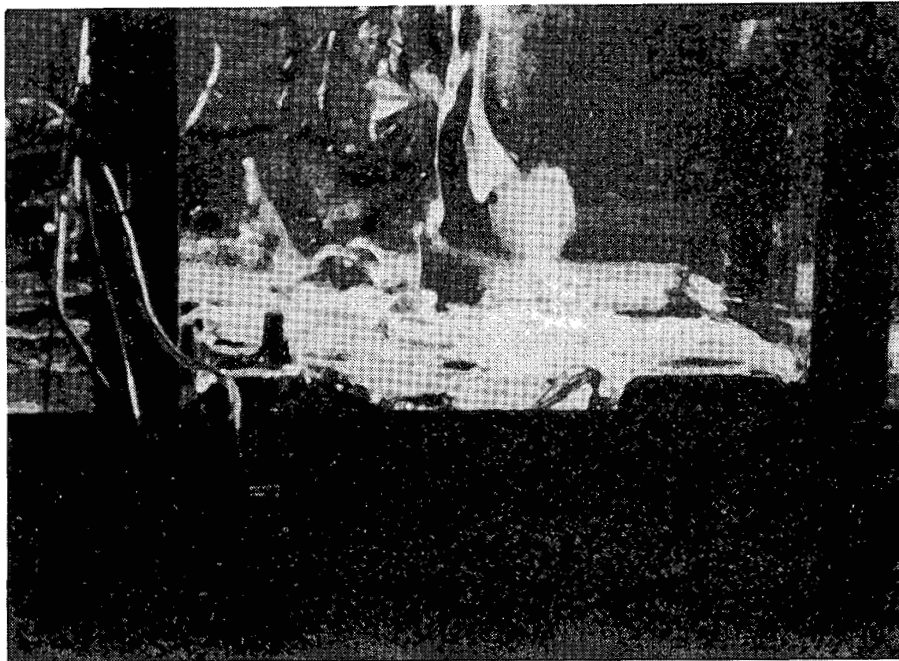

d. 0.15 sec (ANL Neg. 7951, Frame 11A)
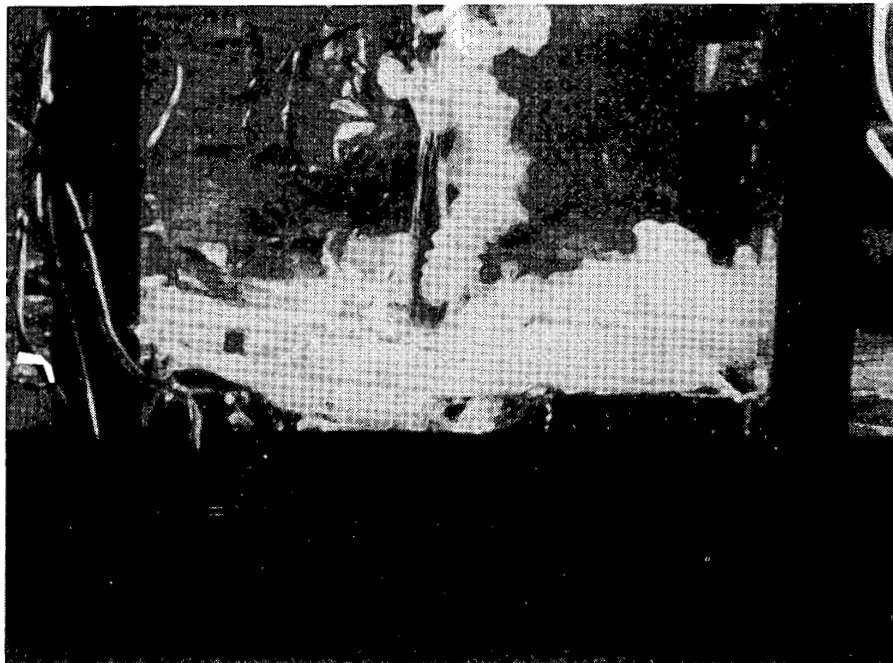
e. 0.22 sec (ANL Neg. 7951, Frame 14A)

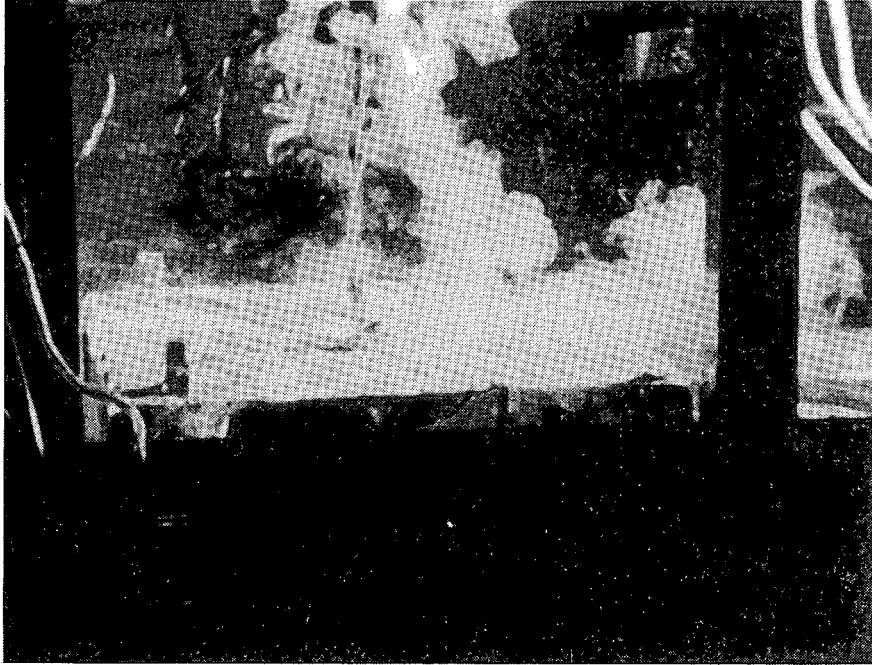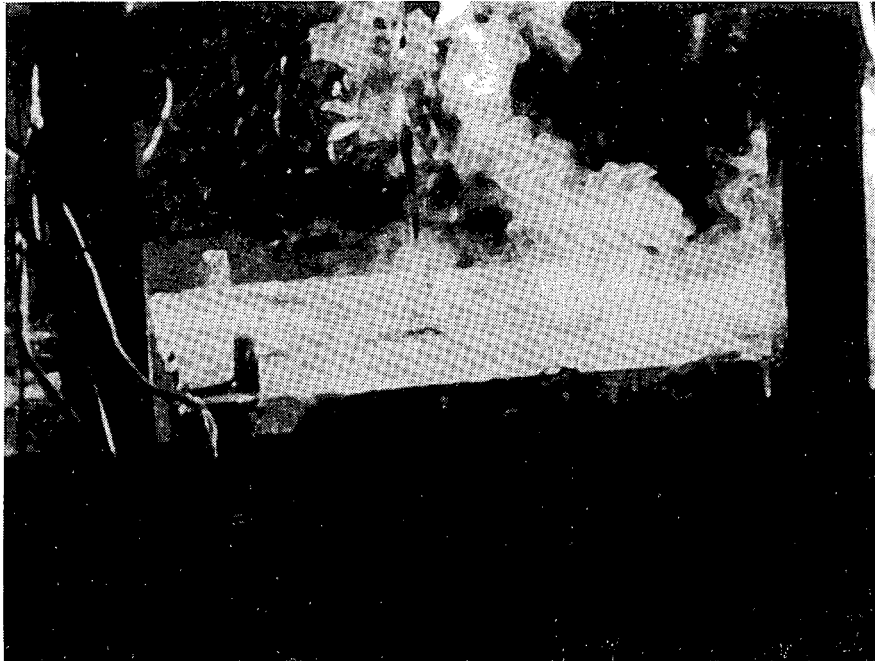f. 0.32 sec (ANL Neg. 7951, Frame 17A

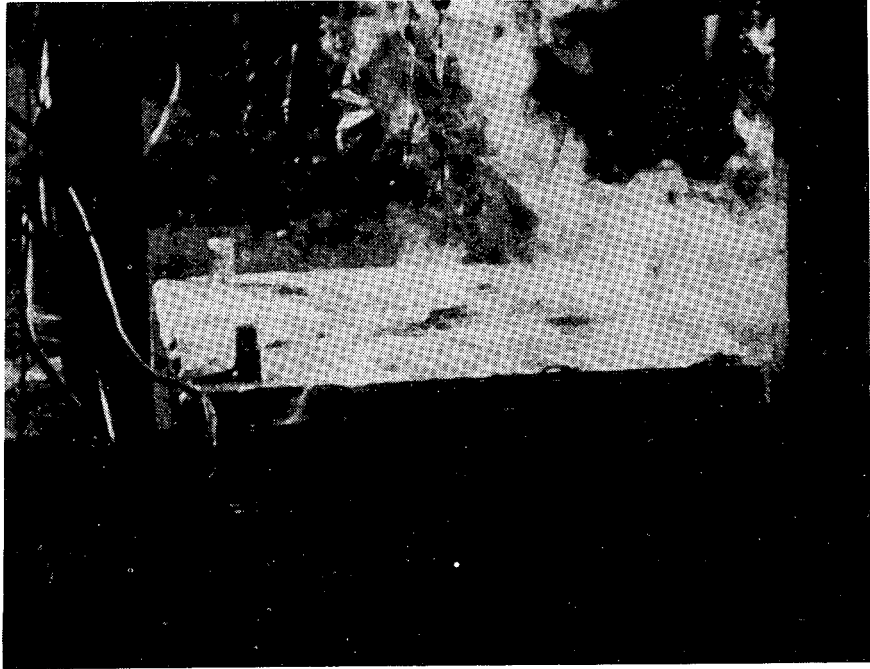g. 0.40 sec (ANL Neg. 7951, Frame 20A)

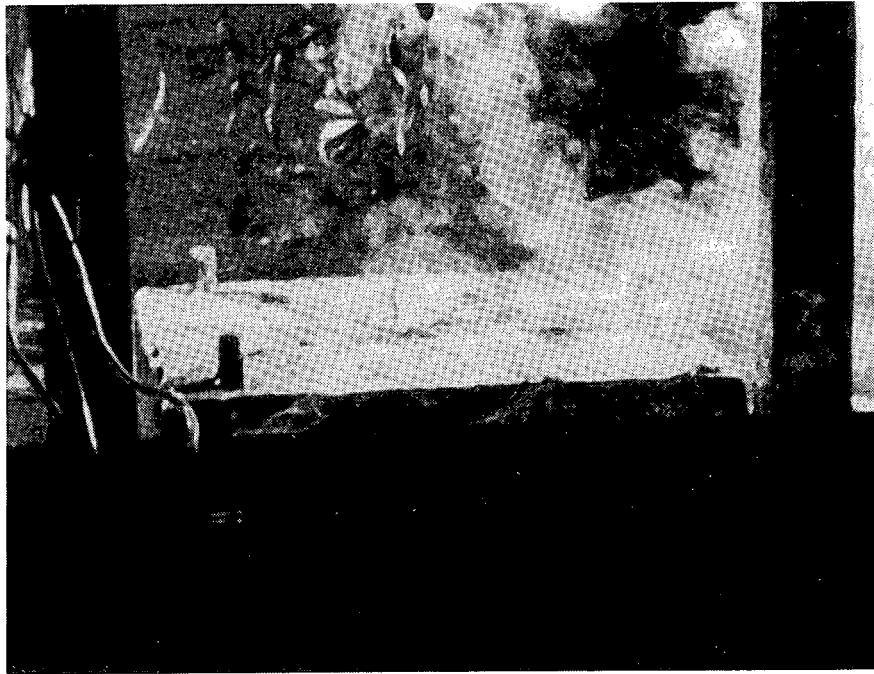h. 0.50 sec (ANL Neg. 7951, Frame 23A)

i.   0.60 sec (ANL Neg. 7952, Frame 2A)

j.   0.70 sec (ANL Neg. 7952, Frame 5A)

k. 0.79 sec (ANL Neg. 7952, Frame 8A)

ℓ. 0.90 sec (ANL Neg. 7952, Frame 11A)

7c. The streaks off to the left are drop trajectories resulting from the splash at impact. The progression of the cadmium spread is shown in Figures 7d, 7e, and 7f. There is no evidence of ignition or smoking up to this point. A puff of smoke is seen emanating from the pour stream 0.4 sec after the diaphragm was cut (Figure 7g). This smoke is attributed to aerosol formation caused by the release of entrained argon, used to pressurize the cadmium, from the pour stream. The pour stream was no longer solid cadmium at this point but a two-phase mixture of argon and liquid cadmium. This com-bination of smoke and liquid cadmium is seen in the remainder of the figures (7g through 7ℓ). After 0.7 sec the pour stream was dissipated and the remaining cadmium was draining out in dribbles with the escaping argon gas generating the smoke (Figures 7i, 7j, 7k and 7ℓ). No smoke was seen coming from the cadmium spill on the steel plate, even during the initial impact when drops were splashed through the air. As in CADD-4 the cadmium flow was quickly quenched with no ignition occurring.

The back panel of the hood (Figure 2) was fitted with four 8 x 10-in. sheets of Gelman Type A/E fiber filters through which all the air flowing through the hood passed. The air flow rate with these filters was 26.6 cfm or a velocity of 12 ft/min through the filters. These four filters in-creased a total of 0.78 g in weight after the test. A chemical analysis measured a total of 0.0277 g of Cd, which is equivalent to 0.03160 g of CdO. This is considerably less than the total weight of particles collected. This difference is attributed to smoke emanating from the binders in the insulation surrounding the ejector heaters, a plastic thermocouple connector which touched the ejector flange resulting in melting and smoke, and room dust down into the hood. An ICP-Spectrochemical analysis of the material collected on the filters (see Appendix) indicated no discernible collection of other metals in addition to the cadmium. It was not feasible with this type of analysis to determine carbon which was the most likely element in the insulation binder and plastic connector smoke. The Cd release fraction was 1.6×10^{-5} inferred from the chemical analysis of the filters and a cadmium spill of 1.71 kg.

It was not possible to estimate the amount of cadmium oxidized from a weight balance between that recovered afterward and that charged to the in-jector. There was a 54.5 g (3 wt %) loss of material which is accounted for by losses from splashing, posttest scraping from surfaces, and equipment

dissassembly. However, the air flow (26.6 cfm) through the hood was sufficient to prevent smoke from escaping into the room atmosphere and to ensure capture of the bulk of the smoke generated and any other fine particulate by the filters on the back panel before settling.

The median particle diameter as measured by the cascade impactor was 2.0 μm indicating very little contribution of aerosol generation by condensing cadmium vapor (see Figure 8). However, as in the case of the large filters placed in the back panel of the hood, most of the particle collection was not CdO. The weight difference for impactor Stage 5 indicated a collection of 1.2 mg of particles whereas the chemical analysis yielded only 1.0 μg of Cd or 1.14 μg of CdO.

The temperature response of the steel plate to the cadmium spill is shown in Figure 9. The temperatures indicated for this test are similar to that for CADD-1 in that a peak temperature of 110C compared to 107C was measured and the plate temperature approached equilbrium in the range of 55 to 75C after 100 sec. Thermocouples 3, 4, and 5 were located near the plate center. The response of thermocouple 4 which was 1/4 in. below the surface is not shown on Figure 9 because the recorder pen skipped and gave an incomplete trace. Where the pen marked, it was within a few degrees of Thermocouple 3. There is no satisfactory explanation of why Thermocouple 5 located 1/8 in. below the surface at the plate center indicated lower temperatures than Thermocouple 3. Most likely the band did not contact the plate when positioned in the hole. Thermocouple 8 was at the bottom surface of the 5/8-in. thick plate near the center of the plate.
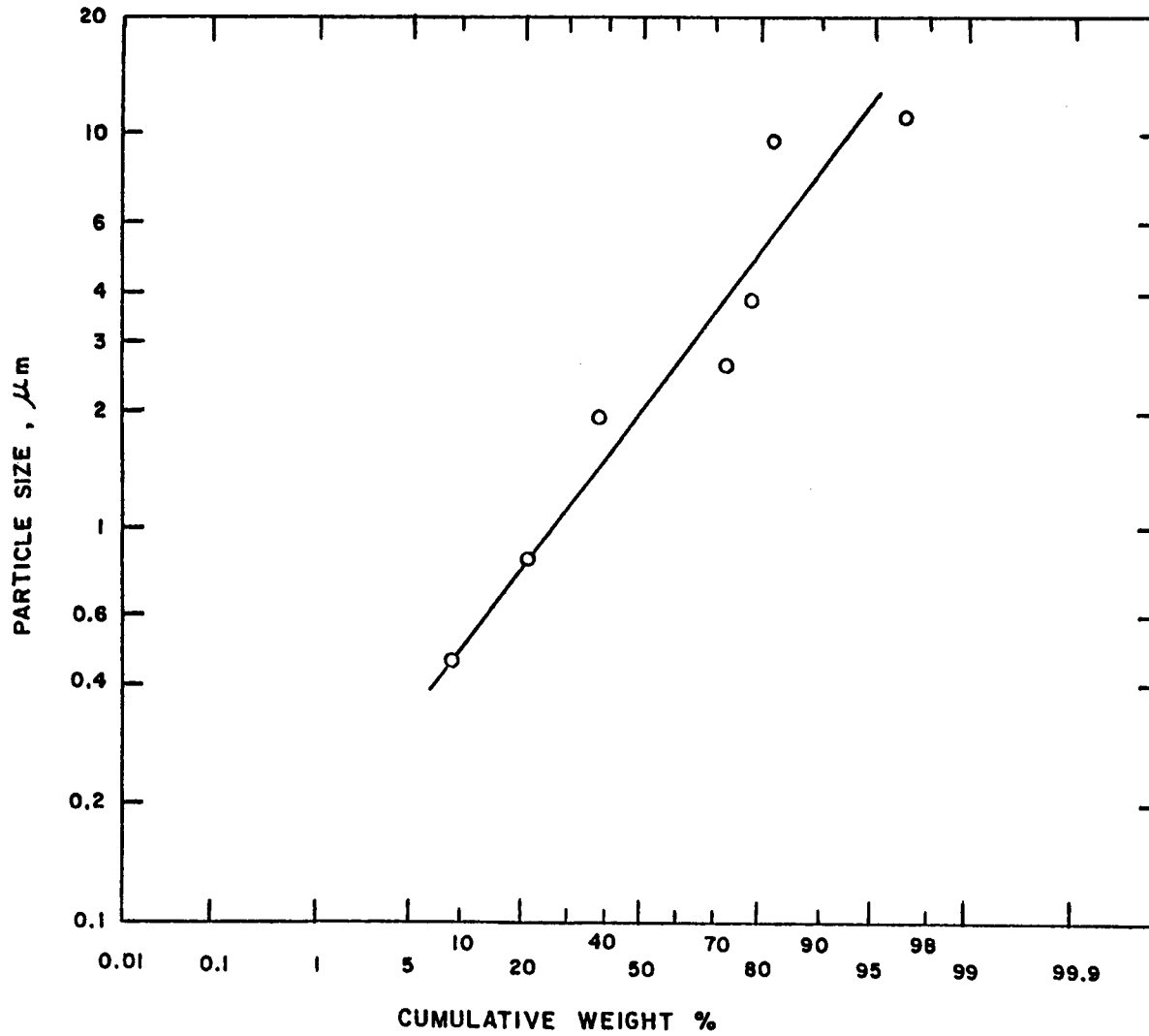
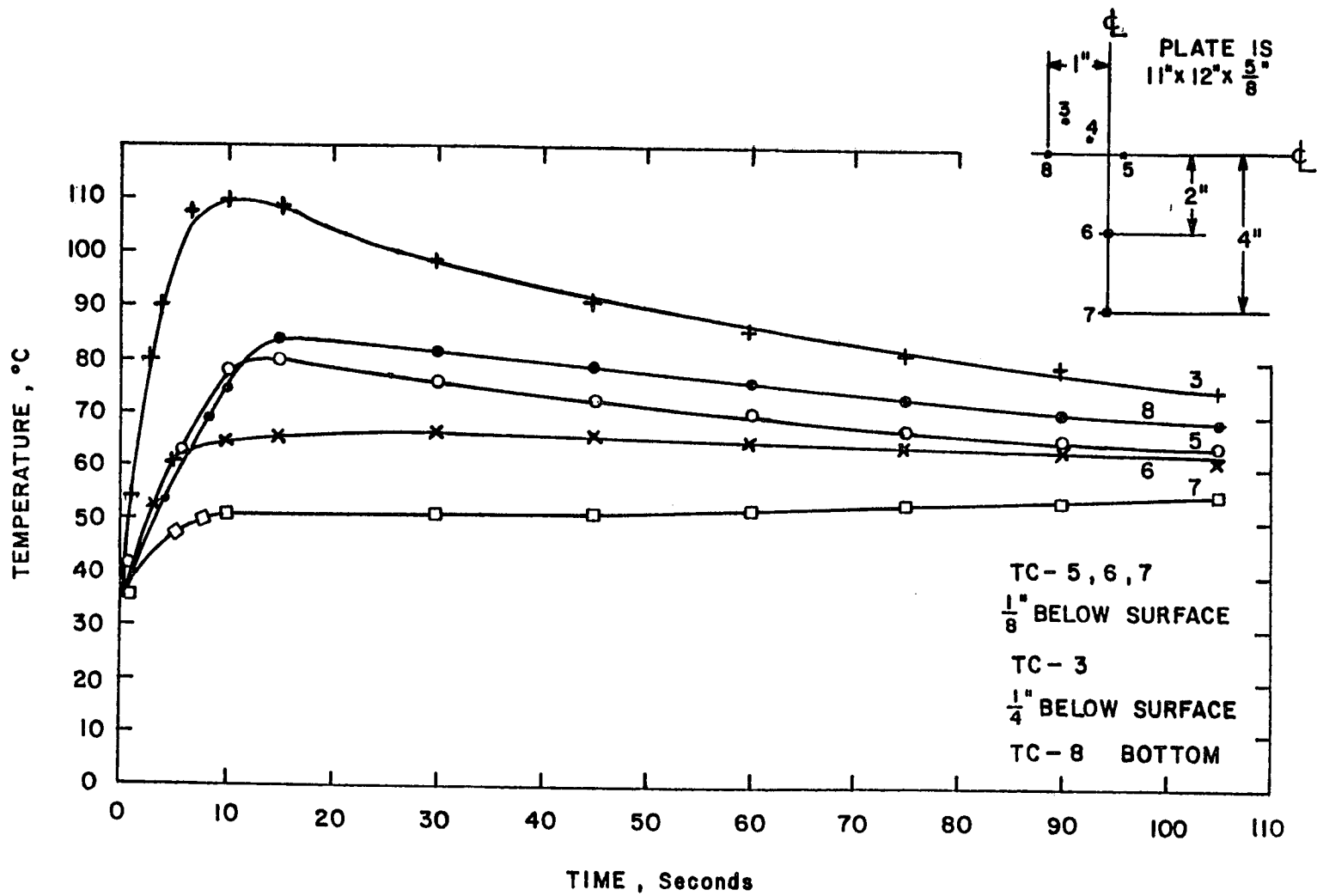Figure 8. CADD-5 Particle Size Cumulative Distribution

Figure 9.   Temperature Response of Steel Plate to Cadmium
Drop in Test CADD-5

IV. CONCLUSIONS

1. Cadmium at the electrorefiner operating temperature of 500C poses no combustion hazard either when spilled or as a stagnant pool when exposed to air.

2. Only 0.0316 g of CdO smoke was collected on filters for a spill of 1.71 kg, implying a Cd release fraction of 1.6×10^{-5}.

3. The median particle size of the aerosol collected in the cascade impactor was 1.2 μm to 2.0 μm, although only a small fraction of particles collected can be attributed to cadmium oxidation.

V. ACKNOWLEDGMENTS

VI. REFERENCES

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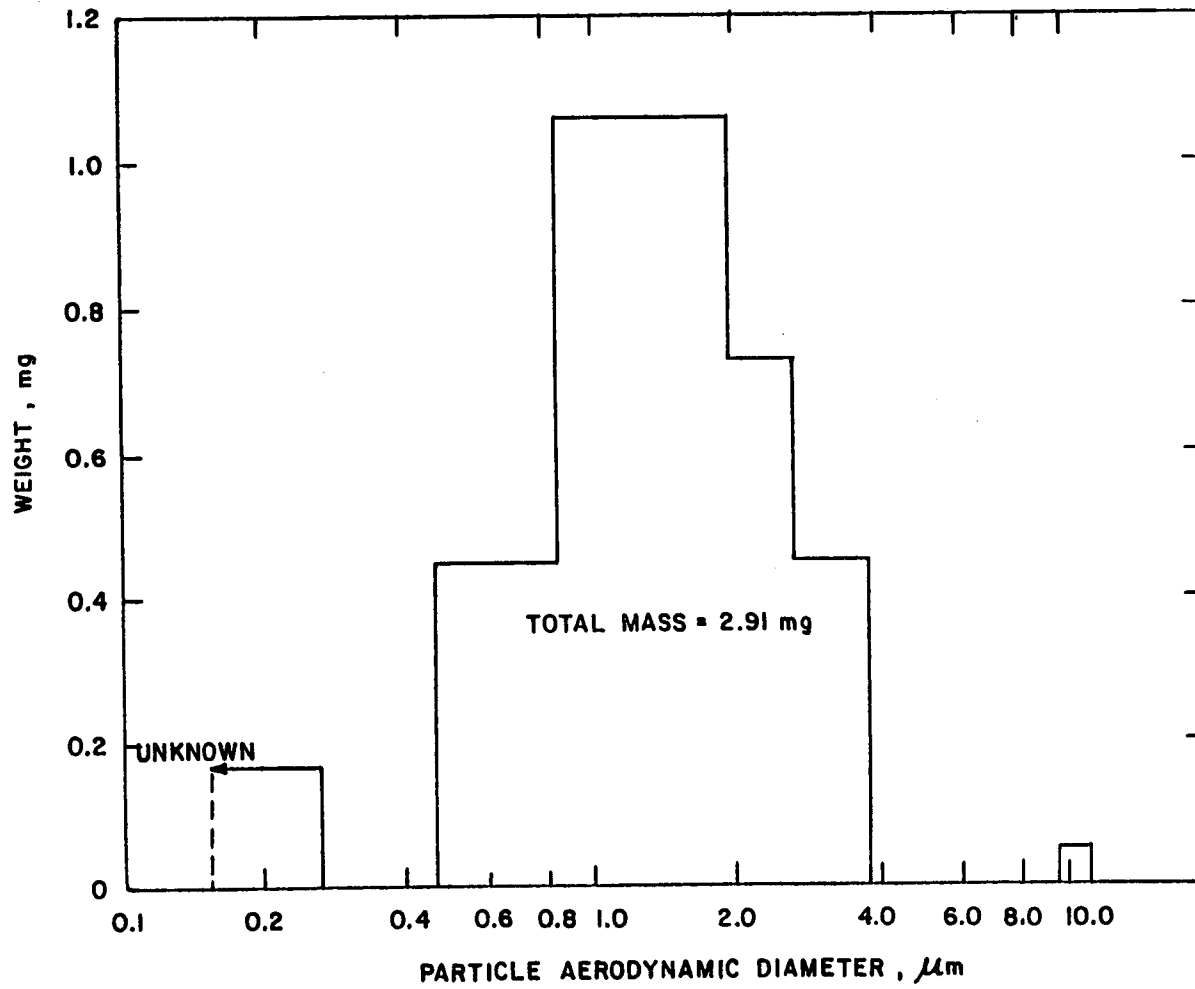
APPENDIX

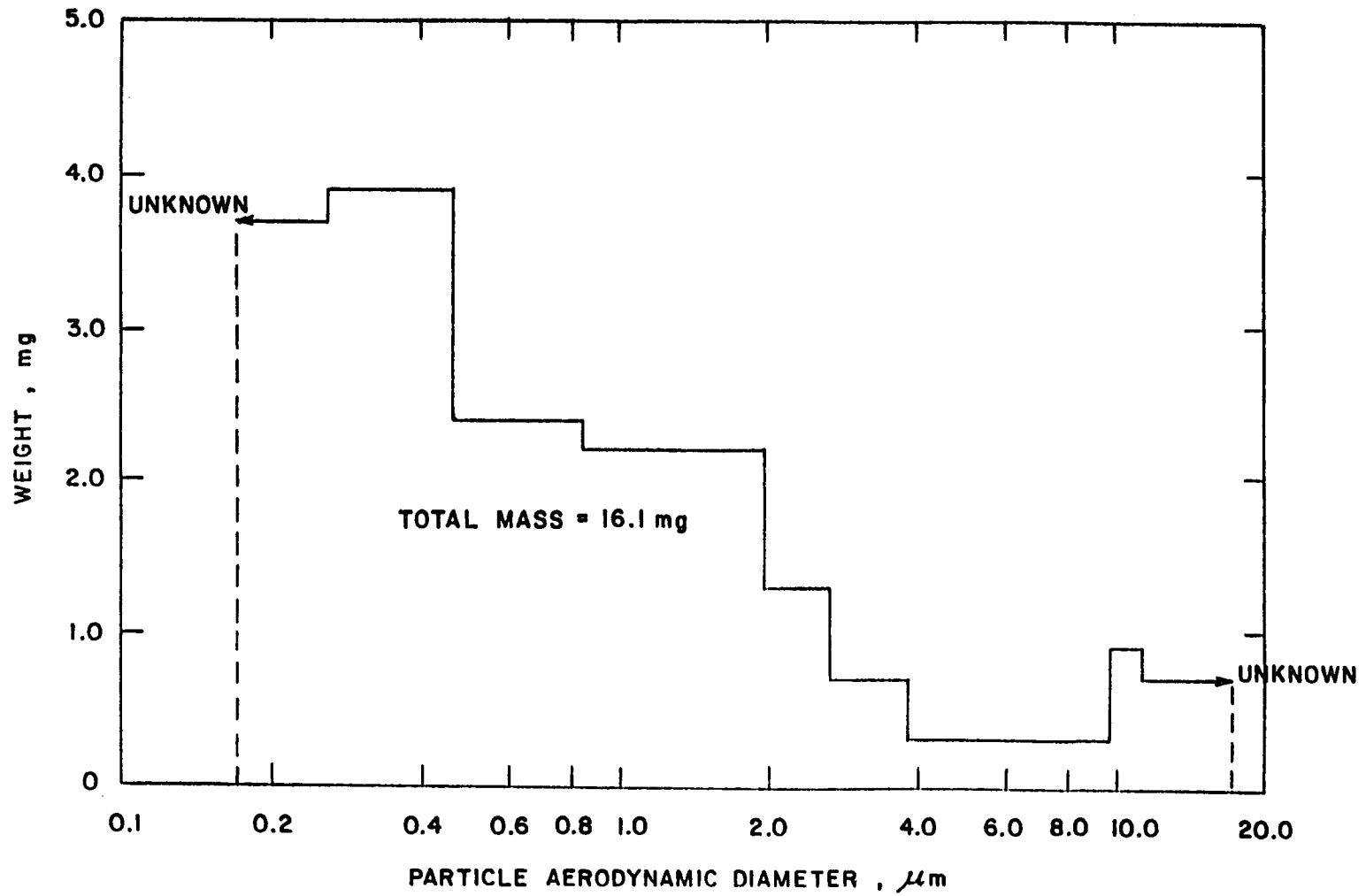Figure A1. CADD-1 Aerosol Weight Distribution

Figure A2.   CADD-4 Aerosol Weight Distribution
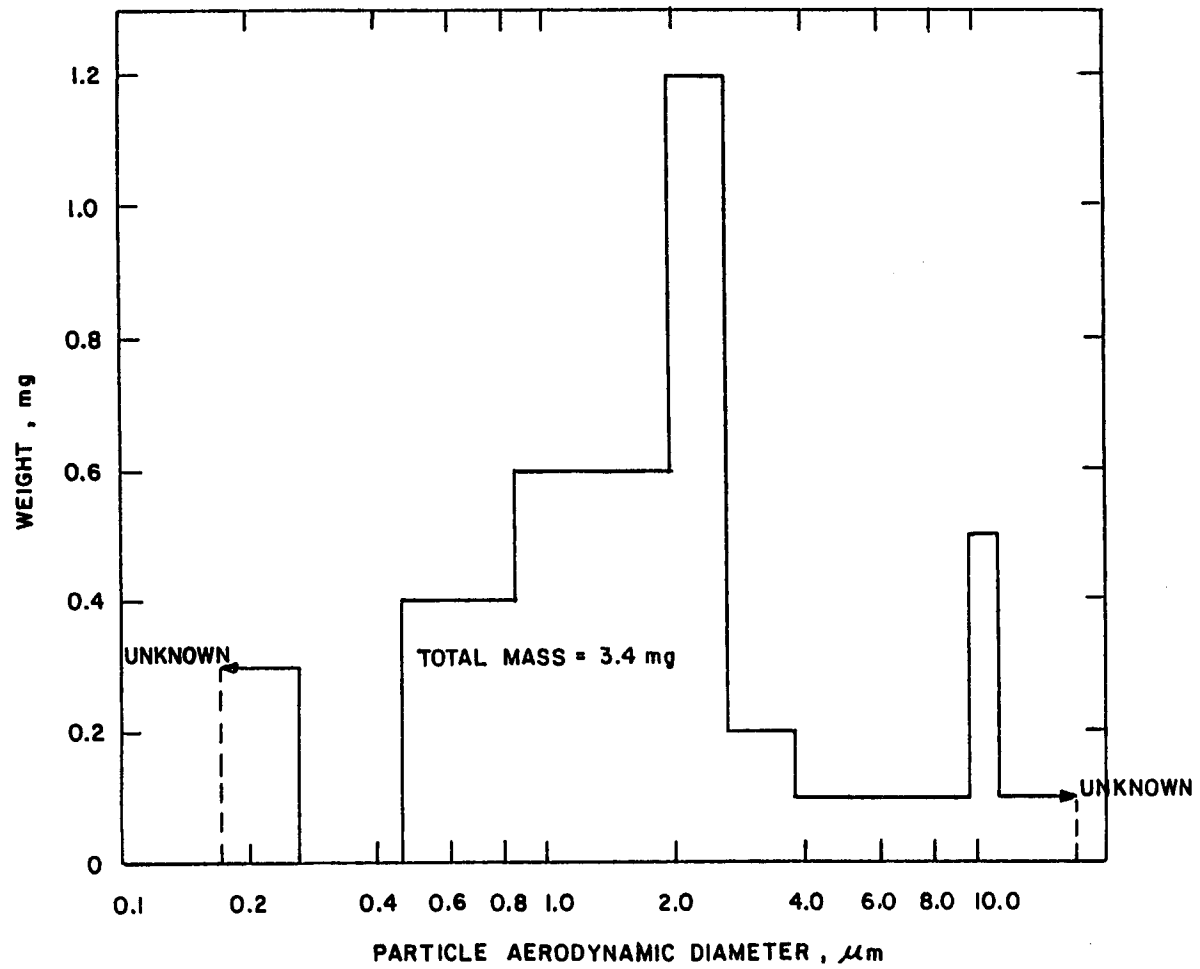
Figure A3.   CADD-5 Aerosol Weight Distribution

Analytical Report for CADD-1

ANALYTICAL CHEMISTRY LABORATORY
Argonne National Laboratory
Argonne, IL 60439

REPORT OF ANALYTICAL RESULTS

Sample Material: Cd and CdO on Filters   Date Received: 5/26/89

Submitted by: J. Cassulo   Date Reported: 6/1/89

Your Number	Our Number	µg Cd (Total on Filter)
CADD Filter A	89-0453-01	650
CADD Filter B	-02	<1
CADD FF	-03	146
CADD 5	-04	640
CADD 6	-05	923
Blank #1 (Impactor)	-06	<1
Blank #2 (Filter)	-07	<1

Estimated accuracy is ±5% of amount present.

Detection limit is 0.5 µg in 50 mL dilution.

NOTE: Samples will be discarded one (1) month after date of report unless otherwise arranged. When making future inquiries regarding this work, you must reference *OUR* number(s) above. For further information about the results reported here, please call ___I Fox_____ at 2-_7398_____.

Copies To: J. Cassulo   Analyst(s): I. Fox
            J. Gabor
            V. Novick
            D. Green
/amb   D. Graczyk
CMT-84 (10-84)   I. Fox
            File

Analytical Report for CADD-5

ANALYTICAL CHEMISTRY LABORATORY
Argonne National Laboratory
Argonne, IL 60439

REPORT OF ANALYTICAL RESULTS

Sample Material: Filters   Date Received: 8/21/89

Submitted by: J. Cassulo   Date Reported: 10/12/89

Your Number	Our Number	Cadmium
CADD5 filter 1	89-0691-01	10.7 ± 0.5 mg
Filter Blank	-02	<5 µg

NOTE: Samples will be discarded one (1) month after date of report unless otherwise arranged. When making future inquiries regarding this work, you must reference **OUR** number(s) above. For further information about the results reported here, please call _____I. Fox_____ at 2-___7398___.

Copies To: J. Cassulo   Analyst(s):   I. Fox
             D. Green   J. Gabor
             D. Graczyk   V. Novick
             I. Fox   File

/mje

CMT-84 (10-84)

Analytical Report for CADD-5

ANALYTICAL CHEMISTRY LABORATORY
Argonne National Laboratory
Argonne, IL 60439

REPORT OF ANALYTICAL RESULTS

Sample Material: Filters   Date Received: 10/30/89

Submitted by: J. Cassulo   Date Reported: 11/20/89

Your Number	Our Number	Cadmium (total µg)
CADD5 Filter 2	90-0067-01	8400.
CADD5 Filter 3	-02	7000.
CADD5 Filter 4	-03	1600.
CADD5 Impactor	-04	1.0
Gelman Filter Blank	-05	2.8
		Estimated accuracy is ±5%.

NOTE: Samples will be discarded one (1) month after date of report unless otherwise arranged. When making future inquiries regarding this work, you must reference **OUR** number(s) above. For further information about the results reported here, please call _I. Fox_____ at 2-_7398____.

Copies To: J. Cassulo   I. Fox   Analyst(s): I. Fox
            J. Gabor   F. Smith   F. Smith
            V. Novick   File
            D. Green
/vts   D. Graczyk
CMT-84 (10-84)

ICP-SPECTROCHEMICAL ANALYSIS REPORT

SPEC NO. 56385

Our No.	Your No.	Material	Ag	Al	As	B	Ba	Be	Bl	Ca	Co	Cr	Cu	Fe	Hg	K	Li
90- 0067-01	Capo5	Filter 2		14.5		4.00	0.48	<.01		24.5		<.01	0.05	0.49			
02	"	" 3		6.9		1.49	0.21	"		13.3		"	0.06	0.30			
03	"	" 4		9.4		3.02	0.27	"		15.0		"	0.06	0.52			
04	"	Impactor		1.4		0.68	0.03	"		0.26		"	<.01	0.20			
05	Graham	Filter Blank		8.7		2.46	0.26	"		14.0		"	0.03	0.41			

Our No.	Your No.	Material	Mg	Mn	Mo	Na	Ni	P	Pb	Sb	Si	Sn	Sr	Ti	V	Zn	Zr
90- 0067-01	see	above	9.42	0.02			<.01				0.90		0.02	0.01	<.01	0.35	<.01
02			4.92	0.01			"				0.51		0.01	<.01	"	0.18	"
03			5.62	0.03			0.01				0.57		0.01	0.01	"	0.20	"
04			0.06	0.02			<.01				0.26		<.01	<.01	"	<.01	"
05			5.25	0.02			"				0.52		0.01	0.01	"	0.16	"

RESULTS REPORTED IN FOLLOWING UNITS

- [ ] parts per million
- [ ] per cent
- [ ] micrograms per ml.
- [✓] micrograms mg in total sample
- [ ]

ESTIMATED ACCURACY OF RESULTS

- [ ] order of magnitude
- [ ] factor of two
- [✓] 10 % of amount present

Plate No. _____

By: E. Huff, F. Smith

Date: 12-15-89

CHM-61 (7-78)

SYMBOL	MEANING
L	less than
G	greater than
	element not investigated
—	element not detected
•	interference
( )	accuracy uncertain
?	presence uncertain

SYMBOL	MEANING	APPROX. CONC.
VS	very strong	100% to 10%
S	strong	10% to 1%
M	moderate	1% to 0.1%
W	weak	0.1% to 0.01%
T	trace	0.01% to 0.001%
FT	faint trace	0.001% to 0.0001%

REMARKS:

Submitted By: J. Caosulo   Location: 206   Date Received: 10-30-89

Reports To: _____, J. Gabor, V. Novich, F. Smith, ACL Office