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Evaluation of Hydroxamic Acid in Uranium Extraction Process: Literature Review

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ABSTRACT

The ***Ur***anium ***Ex***traction (UREX) process is being developed for the spent oxide fuel from light water reactors as part of the Accelerator Transmutation of Waste Project. The UREX process is similar to the ***P***lutonium and ***Ur***anium ***Ex***traction (PUREX) process in that it involves solvent extraction based on a tributyl phosphate extractant. To assure that actinide reduction and complexation are effective, the reductant/complexant aceto-hydroxamic acid is being considered for use in the UREX process. The following literature review describes the solvent extraction, kinetics, oxidation-reduction, and complexation properties of aceto-hydroxamic acid and its derivatives.

I. INTRODUCTION

The Accelerator Transmutation of Waste (ATW) Process is being developed to address disposal of commercial nuclear fuel and improve the performance of a long-term geologic repository [LAIDLER-1999]. The ATW flowsheet separates the dissolved commercial fuel into three streams: (1) a transuranic product stream that will be further processed, converted to ATW fuel, and transmutated by fissioning to generate electrical power, (2) a ^{99}Tc and ^{129}I stream, which will be fabricated into targets for transmutation to short-lived nuclides that significantly reduce the dose from the parent isotopes, and (3) a uranium product stream that will be disposed of as low-level waste. Solvent extraction processes can provide the large-scale selective and efficient separations needed to obtain the above-mentioned product streams.

The ***P***lutonium and ***Ur***anium ***Ex***traction (PUREX) process is a mature solvent extraction technology for the reprocessing of nuclear fuel [NAVRATIL-1984]. The PUREX process was designed to recover plutonium and uranium, while the goal of the ***Ur***anium ***Ex***traction (UREX) process is that plutonium will not be extracted and will remain in the raffinate with the fission products. Further goals are (1) to produce a separate technetium product stream and (2) to collect neptunium in the raffinate and/or a separate product stream. The objectives of the UREX process are summarized as follows:

- >99.9 % of the uranium in the feed would be recovered in a separate stream.
- The uranium product would qualify as a less-than-class-C low-level waste. (This requirement calls for decontamination of the uranium.)

- Plutonium would report to the raffinate, thus lessening proliferation concerns.
- Technetium would be recovered in a separate stream.
- Neptunium will be collected in the raffinate or in a separate stream.

In addition to the uranium product, the UREX process will product the feed streams for (1) PYRO-A, a pyrochemical process that partitions the TRU elements and fission products, and (2) the transmutation target containing the Tc and I radioisotopes. To be designated low-level waste (LLW),[1] the UO_3 product from the UREX process must meet several criteria. The UO_3 product must have less than 100 nCi/g alpha-emitting transuranic isotopes with half-life greater than 5 years.[2] To meet the Class-C LLW limit, fission-product content also is important. The PUREX literature indicates that the only isotopes of concern for the raffinate of a UREX process are ^{99}Tc, plutonium, and neptunium. The UREX solvent is ineffective in extracting strontium, cesium, americium, and curium isotopes; therefore, the potential for high concentrations of these radionuclides in the uranium waste form is not a serious concern. Minimum decontamination factors desired for ^{99}Tc, ^{237}Np, and plutonium are >60, >7, and >7000, respectively, to assure that the uranium can be classified as LLW. Further, the total uranium content in the raffinate, Np product, and Tc product must be less than 0.1%. This decontamination is required for preparing a ^{99}Tc-bearing target and for treating the solidified raffinate in the PYRO-A process to render the transuranic elements suitable for transmutation [LAIDLER-1999].

The baseline UREX flowsheet is shown in Fig. 1 and excludes the iodine separation scheme.[3] This proposed UREX flowsheet was designed to be conservative and is based on past PUREX experience at the Savannah River Site. Note in Fig. 1 the presence of aceto-hydroxamic acid (AHA) in the dissolved-fuel feed in the extraction section and in the aqueous feeds to the scrub and Np-scrub sections. The AHA (Fig. 2) is crucial in achieving the technical objectives of the UREX process by: (1) reducing the important actinides, (2) complexing some of the important actinides, and (3) not affecting the uranium extraction behavior. The AHA is added to reduce Np(VI) and/or Np(V) to Np(IV) and Pu(IV) to Pu(III). Further, AHA forms strong complexes with Np(IV) and Pu(IV), greatly reducing their extractability by the UREX solvent. In addition, the AHA does not reduce the U(VI), thus not affecting its extractability in the UREX solvent. Thus, AHA addition appears to be the key to success of the process.

This report includes a review of hydroxamic-acid chemistry and encompasses the following topics:

- Chemistry of aceto- and formo-hydroxamic acid
- General hydroxamic acid chemistry
- Hydrolysis of FHA and AHA in nitric acid
- Actinide complexation by hydroxamic acid
- Reduction of Np(VI) and Pu(IV) by FHA and AHA
- Distribution ratios for U, Np, and Pu

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[1] That is, not transuranic (TRU) or high level waste.

[2] The sum-of-fractions rule applies. The sum of the ratios of isotope-concentration/isotope-limit must be less than one.

[3] Scrubbers will recover iodine in the off-gas system for the dissolver. The spent nuclear fuel will be chopped and then leached in the dissolver by hot nitric acid.

Unfortunately, one of the major species of concern in the UREX process is technetium, and our literature review did not uncover any technetium hydroxamic acid complexation/separation data with hydroxamic acid.

II. CHEMISTRY OF ACETO- AND FORMO-HYDROXAMIC ACIDS

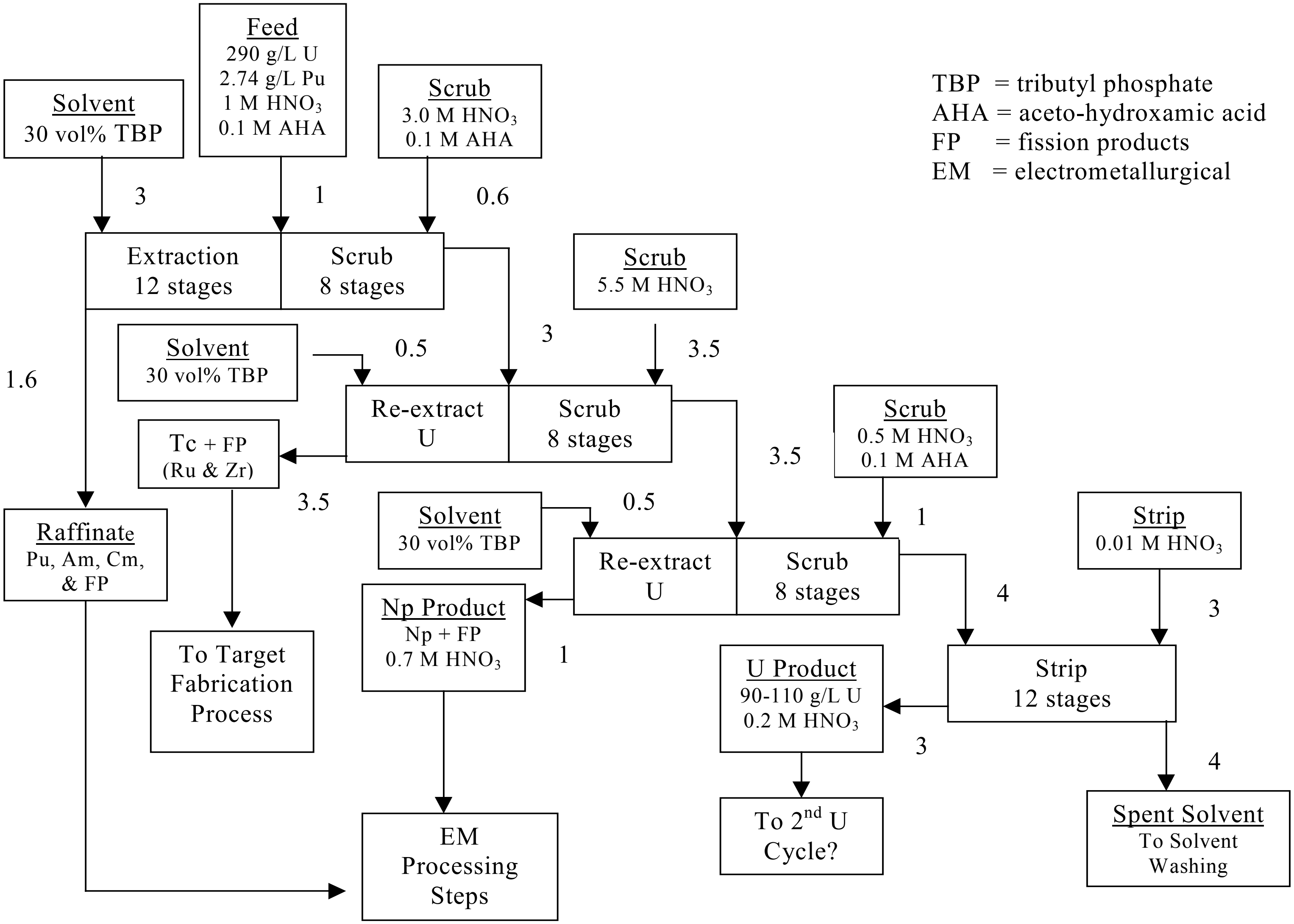
The development of a one-cycle "advanced PUREX process" has focused on formo-hydroxamic acid (FHA) and aceto-hydroxamic acid (AHA) as the reducing agent [TAYLOR-1998A]. The unique chemical characteristics of these organic acids include:

- The oxygen donor ligands have shown a strong affinity for hard metal ions such as Fe^{3+}, Al^{3+}, Np^{4+}, and Pu^{4+}.
- The small organic backbone ensures low solubility in TBP solvents.
- The hydroxamic acids decompose into NO_2 and CO_2. For example, in 6 M HNO_3, FHA will decompose into diatomic gases [MAY-1998].

However, more importantly, the hydroxamic acids are rapid reducing agents for Np(VI) without the reduction of U(VI) [TAYLOR-1999]. Hydroxamic acids with a low carbon backbone, such as FHA and AHA, have been shown to aid in the separation of Np(IV) from U(VI) by selectively forming a Np(IV) hydrophilic complex [TAYLOR-1998A, -1998B, -1999; MAY-1998]. For both FHA and AHA, Taylor has shown that between 0.1 and 1 M HNO_3, Np(IV) complexation was preferred over U(VI), but above 1.8 M HNO_3, some U(VI) complexation was observed. Furthermore, both FHA and AHA have been reported to form a red complex with Pu(IV) ions, which transform into the blue Pu(III) complex after standing for several hours [TAYLOR 1998A]. The best conditions for using the hydroxamic acids are in <3 M HNO_3, where the hydroxamic-acid hydrolysis to give hydroxylamine and the parent carboxylic acid (RCOOH) is minimized:

$$RCONHOH + H_2O + H^+ \leftrightarrow RCOOH + H_3NOH^+ \qquad \text{(Eq. 1)}$$

In this equation, R represents methyl for the aceto and hydrogen for the formo moieties. The hydroxamic acid instability in nitric acid should not affect planned flowsheet operations due to the use of centrifugal contactors, where the processing time is expected to be short compared to the measured destruction half-life in nitric acid [TAYLOR-1999].



TBP = tributyl phosphate
AHA = aceto-hydroxamic acid
FP = fission products
EM = electrometallurgical

Fig. 1. Schematic of UREX Flowsheet. The numbers next to the arrows are the relative flowrates. The high uranium extraction needed was verified by batch-contact test in August 2000.

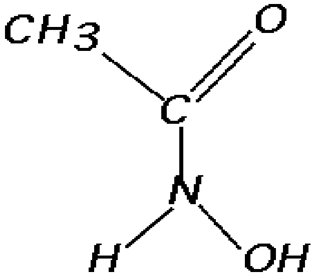


Fig. 2. Chemical Structure of Aceto-Hydroxamic Acid

III. GENERAL HYDROXAMIC ACID CHEMISTRY

For many years, hydroxamic acids were used as colorimetric and gravimetric reagents [ALIMARIN-1962], and current interest focuses on the reduction/complexation chemistry with metals. Some of the general chemical properties of the hydroxamic acids are described below.

Hydroxamic acids exist in two tautomeric forms (keto and enol). The keto-enol isomerism provides a number of sites for chelation. The keto form is predominant in acid medium, and the enol form in alkaline medium [HERVEY-1950].

In general, the hydroxamic acids are weak donors. The pK values for many hydroxamic acids have been determined. The pK values for the acids vary from 7.05 (nitrobenzohydroxamic acid) to 11.33 (N-phenyl-n-butyrodhyroxamic acid) [CHATTERJEE-1978]. The AHA has a pK value of 7.397 [WISE-1955].

There are extensive reviews on the stability constants and metal complexation properties of hydroxamic acids, but many focus on the benzohydroxamic acid complexes [CHATTERJEE-1978]. The metals of interest to UREX processing that have been studied with benzohydroxamic acid are Fe(III), Ru(III), U(VI), Zr(VI), Th(VI), Pu(VI), rare earths, and Al(III) [CHATTERJEE-1978, BARONCELLI-1965, BAROCAS-1966]. Stability constants for AHA and benzohydroxamic acid are shown in Table 1.

IV. HYDROLYSIS OF FHA AND AHA IN NITRIC ACID

The degradation of hydroxamic acids produces a carboxylic acid and hydroxylamine, as shown in Eq. 1.

Both FHA and AHA reaction kinetics are described by Eq. 2, where X= aceto or formo:

$$-d\frac{[XHA]}{dt} = k[XHA][H^+] \qquad \text{(Eq. 2)}$$

The rates and activation energies for both FHA and AHA at 25°C are listed in Table 2. For AHA, the decomposition rate is 0.00205 mol/(L•min) at 25°C, with an activation energy of 79.9 ± 2.9 kJ/mol [GLENNOR-1989]. Table 3 shows the half-life of degradation of AHA vs. the hydrogen ion activity. As can be seen from these results, half the AHA will be destroyed in eleven hours when the hydrogen ion activity is 0.5 M; however, the time decreases to about an hour at activities greater than 5 M.

Table 1. Stability Constants for Select Cations with Hydroxamic Acids

Ions	**Logβ_1**	**Logβ_2**	**Logβ_3**	**Logβ_4**	**Hydroxamic Acid**	**Reference**
U(VI)	8.72	16.77	-	-	Benzo-	BARONCELLI-1965
U(IV)	9.89	18.0	26.32	32.94	Benzo-	BAROCAS-1966
Pu(IV)	12.73	-	-	-	Benzo-	BAROCAS-1966
Th(IV)	9.60	19.81	28.76	-	Benzo-	BAROCAS-1966
Zr(IV)	12.43	24.08	-	-	Benzo-	BARONCELLI-1965
Fe(III)	11.42	21.10	28.33	-	Aceto-	ANDEREGG-1963
Fe(III)	12.18	-	-	-	Benzo-	BARONCELLI-1965
Al(III)	7.95	15.29	21.47	-	Aceto-	ANDEREGG-1963
Ce(III)	5.45	9.79	12.8	-	Aceto-	ANDEREGG-1963
La(III)	5.16	9.33	11.88	-	Aceto-	ANDEREGG-1963
Ca(II)	2.4	-	-	-	Aceto-	ANDEREGG-1963

Table 2. Kinetic Rate and Activation Energy for Hydroxamic Acids at 25°C [TAYLOR-1998A, TAYLOR-1999, MAY-1998]

Acid	**Rate Constant mol/(L•min)**	**Activation Energy (kJ/mol)**
FHA	0.016	77.3
AHA	0.00205	79.9

Table 3. Effects of Hydrogen Ion Activity on the Destruction of AHA at 25°C

$\{H^+\}$, M	**Destruction Half-Life, min**
0.5	676
1	338
2	169
3	113
5	68
10	34

The rapid acid degradation is a concern in prolonged processing. This degradation effect can be addressed by using the centrifugal contactor and is part of a pilot demonstration planned at ANL. However, one of the major advantages of the hydroxamic acids is their actinide complex stability.

V. ACTINIDE COMPLEXATION BY HYDROXAMIC ACID

The stability constants for the actinide-hydroxamate complexes increase in the order:

$$\beta_{U(VI)} < \beta_{H+} < \beta_{U(IV)} < \beta_{Np(IV)} < \beta_{Pu(IV)}$$

For benzo-hydroxamic acid, the ratio of $\beta_{Pu(IV)}/\ \beta_{U(VI)}$ equals 10^4 [CHATTERJEE-1978, BARONCELLI-1965, BAROCAS-1966]. Thus, tetravalent actinides should be preferentially complexed in the presence of U(VI). The complexation chemistry of hydroxamic acids with U, Np, and Pu is described.

The complexation of UO_2^{2+} in FHA and AHA occurs at near neutral pH, producing a red/orange solution (U = 50 g/L and ligand = 2 M). Sometimes, a red precipitate forms on standing for long periods. Increasing the HNO_3 concentration inhibits the precipitation reaction, and in 1M HNO_3, no precipitate is formed. May et al. [MAY-1998] showed that for the FHA-U(VI) complex, >95% complexation is only observed for >40 times excess ligand, and this is greatly inhibited in nitric acid solutions.

Neptunium(IV) exhibits characteristic absorption bands at 723 and 960 nm in 0.2 M HNO_3 (25°C) and at 706 and 729 nm in 30% TBP/odorless kerosene [TAYLOR-1998A]. On the addition of hydroxamic acid to a Np(IV) solution, distinct peaks occur at 714, 732, 851, and 975 nm. The peak height is maximized at 2-2.5 times excess ligand (FHA or AHA). For neptunium, 1:1 and 2:1 complexes are formed, with complex formation being inhibited at higher HNO_3 concentrations. The characteristic absorption wavelengths for the actinide complexes are shown in Table 4.

Table 4. Characteristic Absorption Wavelengths for Metal-Hydroxamate Complexes [TAYLOR-1998A, MAY-1998].

Actinide	Complexant	Wavelengths (nm)
Fe(III)	AHA	499
Fe(III)	FHA	489
U(VI)	FHA	373, 473, 480
Np(IV)	FHA	714, 734, 851, 975
Pu(IV)	AHA	900
Pu (IV)	FHA[a]	660
Pu (III)	FHA[a]	570, 607

[a] Estimated from absorption spectrum.

Both AHA and FHA form a deep red complex with Pu(IV), which is inextractable in TBP and has a distinct absorption spectrum. Furthermore, this complex slowly changes to a blue Pu(III) complex when standing for several hours. The reduction was performed at 22°C under solution conditions of 1.0 g/L Pu(IV), 0.5 M HNO_3, and 0.1 M AHA. The change of the Pu(IV) complex to Pu(III) was monitored for 5.5 hours. The results suggest that even with favorable thermodynamics, Pu(IV) to Pu(III) kinetics are slow. With the addition of U(VI) at 50 g/L, an increase in the rate of Pu(IV) reduction was also observed. Although the actinide complexation stability is important, the ability of the hydroxamic acids to reduce both Pu(IV) and Np(VI) selectively is crucial in the UREX flowsheet development.

VI. REDUCTION OF Np(VI) AND Pu(IV) BY FHA AND AHA

The FHA and AHA onset potential was determined by cyclic voltammetry as +0.55 V vs. a saturated calomel electrode in 0.1 M HNO_3 and +0.58 V in neutral 0.1 M KNO_3 [TAYLOR-1998B]. The onset potential indicates that the hydroxamic acids can thermodynamically reduce neptunium and plutonium, but not uranium. However, the reaction kinetics for Pu reduction is slow in the above-mentioned complexation. The reduction of Np(VI) to Np(V) occurs over a short time. In 1.8 M HNO_3 at 25°C, the reduction requires only a few seconds. Furthermore, according to Taylor et al., the reduction of Np(V) to Np(IV) by FHA in HNO_3 essentially does not occur due to the slower Np(V) to Np(IV) kinetics expected and the competition with the hydrolysis of FHA [TAYLOR-1998A].

VII. DISTRIBUTION RATIOS OF U, Np, AND Pu WITH HYDROXAMIC ACIDS

Reported actinide distribution values with AHA and FHA in 30% TBP systems are sparse in the literature. The Np(IV) distribution ratios in 30% TBP are decreased by an increase in FHA concentration and an increase in nitric acid concentration. For a neptunium concentration of 0.005 M and an equilibrium-HNO_3 concentration of 1.2 M, the distribution ratio is less than 0.15. The neptunium distribution ratio is unaffected for an AHA/Np concentration ratio of 25, and higher with an organic phase of 30% TBP.

At 0.4 M HNO_3 and 0.1 M FHA with neptunium equal to 0.005 M, the Np(VI) distribution ratio in 30% TBP decreases from ~0.3 to ~0.05 due to the Np reduction/complexation. For 0.1 M FHA, 0.005% M Np(VI), and 2 M HNO_3, the neptunium distribution ratio decreases from ~1.05 to 0.4 due to the Np reduction/complexation. Distribution ratios for uranium were within 10% of those obtained without a reducing agent.

Specific data on the distribution ratios of Pu were not given in the reviewed papers.

VIII. CONCLUSIONS

The information gathered from the literature is encouraging for addition of AHA or FHA to achieve the actinide reduction/complexation needed to meet UREX process objectives. However, considerable R&D will be required to understand the chemistry well enough to (1) use AHA or FHA in a plant and (2) model the chemistry for flowsheet design. Work being undertaken in FY 2001 is designed to meet both objectives.

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