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TECHNICAL PROGRESS REPORT**

Part 3 - Metal Processing (U)

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HIGHLIGHTS

URANIUM EXTRACTION SOLVENTS

Various organic ethers, hydroxy ethers, ether ketones, acids, amides, and diketones were studied for their ability to extract uranyl nitrate from aqueous solutions. (Page 6)

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METAL PROCESSING

URANIUM EXTRACTION SOLVENTS

R. F. Simandl, J. M. Googin, G. F. Dorsey, J. D. Brown, J. D. Hasse

Summary

Various organic ethers, hydroxy ethers, ether ketones, acids, amides, and diketones were studied for their ability to extract uranyl nitrate from aqueous solutions. Although many of these solvents were obtained commercially, others had to be synthesized in-house. The authors found a large range of extraction coefficients for these solvents. Because of steric hindrance or some other factor, certain ethers performed poorly. On the other hand, various mono- and diethers of tetrahydrofurfuryl alcohol exhibited excellent extraction and stripping coefficients for uranyl nitrate, justifying purchase of a "pilot plant" batch of one of this family of solvents. Likewise, the authors determined the extraction coefficient for one of the two amides synthesized in-house to be quite high.

Introduction

As part of the effort to downsize its uranium processing facilities, the Y-12 Plant has supported an investigation to identify extraction solvents that would both work efficiently in centrifugal contactors and be disposed of easily. Although both dibutyl carbitol and tributyl phosphate were used successfully in pulsed extraction columns, both solvents exhibit inherent problems. Contact with the nitric acid aqueous phase tends to oxidize and degrade the dibutyl carbitol and forms an intractable uranium oxalate precipitate that fouls the extraction columns. Moreover, the ethylene glycol ethers have recently come under increasing scrutiny for their chronic effects on health. The tributyl phosphate is quite toxic and presents a disposal problem in that it cannot be completely disposed of by combustion because it leaves toxic, reactive, corrosive oxides of phosphorous.

The senior staff member defined desirable extraction solvents as having the following properties:

- extraction coefficients approximating or exceeding those of dibutyl carbitol (i.e., >55 from half-saturated solutions of calcium nitrate having a pH = 1, >1 from 30% nitric acid solutions),
- a stripping coefficient into 0.1 N nitric acid of <0.05 ,
- fast extraction kinetics,
- very low water solubility,
- low break times for aqueous nitric acid/solvent emulsions,
- low density ($\sim 0.8 \text{ g/cm}^3$),
- stability in the presence of 30 wt % nitric acid,
- non-Resource Conservation and Recovery Act (RCRA),

- have low toxicity, and
- 100% combustible with negligible ash content.

Various organic phosphates, phosphonates, phosphinates, and phosphine oxides were studied,¹ and some of them complexed aggressively with actinide nitrates. However, because these solvents leave an undesirable residue upon ignition, they were not chosen for the current investigation. Various amides exhibited excellent extraction properties for actinide nitrates.² Because amides tend to form stable emulsions with an aqueous phase, they appeared initially to be unsuitable for use in centrifugal contactors. Moreover, amides tend to hydrolyze in the presence of nitric acid. Nevertheless, the authors chose two of these amides for study as part of this investigation. Ethers and ketones also were studied extensively as extraction solvents.^{1,3} Ethers such as dibutyl carbitol were used extensively for extractions because of their low density, low viscosity, and stability in an acid environment. Because of recent concern about both the chronic effects on health and the undesirable degradation products of the ethylene glycol ethers, new families of ethers using propylene glycol, tetrahydrofurfuryl alcohol, and glycerin were prepared and investigated as part of this study. Griffith and Compere recently investigated several of these ethers and reported their results in internal correspondence.⁴

Prior Work

Although a considerable amount of work on uranium extraction solvents (particularly dibutyl carbitol) has been reported in earlier Y-12 Plant Development Organization technical progress reports, this is the first quarterly report by these particular authors.

Presentation of Experimental Work

Six families of organic solvents were chosen for testing and were either purchased from some outside source or, if they were commercially unavailable, synthesized in-house. These solvents—each of which is discussed in the following paragraphs—included ethers, hydroxy ethers, ether ketones, organic acids, amides, and diketones.

Ethers. The following ethers were tested as uranium extraction solvents:

- dibutyl carbitol,
- 1,8-bis-tetrahydrofurfuryl (THFA)-n-octyl ether,
- di-n-propyl ether of 2,5-bis(hydroxymethyl)THF,
- THFA-n-butyl ether,
- THFA-n-hexyl ether,
- 1,5-di-n-propoxy pentane,
- di-n-propyl ether of di-propylene glycol,
- tri-n-propyl ether of glycerin, and

- di-n-butyl ether of 2-methyl-1,3-propanediol.

All of the THFA ethers were obtained from QO Chemicals in Memphis, Tennessee, a supplier that is very familiar with the chemistry of tetrahydrofurfuryl alcohol. The dibutyl carbitol and the di-n-hexyl ether were also purchased from outside sources. All of the other ethers were synthesized in-house using a Williamson synthesis.

Table 1 summarizes various physical characteristics of these ethers. The di-n-propyl ether of 1,2-dipropylene glycol consisted of mixed isomers. To test the performance of a given solvent for extracting uranyl nitrate, the following standard solutions were prepared:

1. stripping solution consisting of 10 g/L uranium (21 g/L uranyl nitrate) in 0.1 N nitric acid,
2. extraction phase consisting of 10 g/L uranium (21 g uranyl nitrate) half-saturated with calcium nitrate tetrahydrate (300 g/100 g water), and
3. a third aqueous phase consisting of 10 g/L uranium (21 g uranyl nitrate) in 30 wt % nitric acid in water.

The third aqueous phase was included because the senior staff member was interested in determining whether uranyl nitrate could be extracted from nitric acid alone without using a salting agent. The densities of the preceding solutions at 22°C were measured 1.005 g/cm³ ($\pm 0.68\%$) for the first solution, 1.498 g/cm³ ($\pm 1.47\%$) for the second solution, and 1.159 g/cm³ ($\pm 0.517\%$) for the third solution. Uranium extractions were studied by shaking 10 cm³ of each solvent with each of the preceding three aqueous uranium solutions. At first, the aqueous/organic pairs were not separated and sent to the Plant Laboratory for uranium assay. As the study progressed, however, increased accuracy was desired; hence, both phases were separated, and both the volumes and the masses were determined before analysis at the Plant Laboratory.

Table 2 lists the results of the extraction phase of this study. It is organized into "groups" of individual solvents, each tested against three aqueous uranium phases. Ten groups of ether experiments were tested to this point, the first nine of which are listed in the first part of Table 2. The tenth ether, di-n-hexyl ether, is listed as Group 16 at the beginning of the section labeled "Diketones in Dihexyl Ether." It was included at this point for comparison with the extraction results using diketones in this particular ether.

Column 5 of Table 2 lists the actual concentration of uranium in the aqueous phase (in grams of uranium per gram of solution) as determined by the Plant Laboratory. Column 7 lists the corresponding concentration for the organic phase. Columns 6 and 8 list the total uranium content in the aqueous and organic layers, respectively. These values were calculated for Groups 1 through 17, 19, and 21 using approximate solution densities. The corresponding values for Groups 18, 20, and 22 were determined using more careful measurements. Column 9 provides a quick check on the total amount of uranium as determined experimentally. Values in this column were determined by adding Columns 6 and 8 and should be close to 0.1. In certain cases, samples were lost, and data had to be back-calculated using the assumption that the total uranium was 0.1 g/cm³. Column 10 lists the values calculated for the uranium distribution coefficients. These values were determined by dividing the total uranium in the organic layer by that in the aqueous layer (Column 8/Column 6).

Table 1. Physical properties of various ethers

Solvent	Purity	Density (g/cc)	Solubility in water starting with 4 cc each (vol. org/vol aq. phase)
Dibutylcarbitol		0.8797	95% aq./100% org.
1,8-Bis-tetrahydrofurfuryl)-n-octyl ether		0.960 $\pm$ 1.33%	
Di-n-propyl ether of 2,5-bis (hydroxymethyl)THF		0.92	4.0/3.9
THFA-n-butyl ether		0.92	<0.3%
THFA-n-hexyl ether	>95%	0.89	170 ppm
1,4-Bis(tetrahydrofurfuryl) butyl ether			30%-40%
1,5-di-n-propoxypentane			
Di-n-propyl ether of 1,2- dipropylene glycol (mixed isomers)	>96% diether, (>99% propylated)	0.857 $\pm$ 0.95%	4.0/3.9
1,2,3-tri-n-propyl ether of glycerine	>98%	0.8567 $\pm$ 0.52%	3.8 org/4.1 aq.
Di-n-butyl ether of 2-methyl-1,3- propanediol	97.5 mol % diether, 2.5% mono	0.817 $\pm$ 1.04%	
Di-n-hexyl ether	98%	0.793	4.0/4.0

Table 1. Physical properties of various ethers

Density (g/cc)	Solubility in water starting with 4 cc each (vol. org/vol aq. phase)	Boiling point (°C)	Flash point (°F)	Refractive index at temperature (°C)	Reaction with 30 wt % nitric acid
8797	95% aq./100% org.	254.6	245	1.4308 (25°C)	
960 ± 1.33%					Orange fumes at 100°C
92	4.0/3.9	>10 mm at 125°C		1.4330 (25°C)	
92	<0.3%	195	156	1.4315 (25°C)	Orange fumes at 100°C
89	170 ppm	231	196	1.4359 (25°C)	
	30%-40%	>150			Orange fumes at 100°C
857 ± 0.95%	4.0/3.9				Orange fumes at 100°C
8567 ± 0.52%	3.8 org/4.1 aq.	<120°C 1 mm vacuum			
817 ± 1.04%					
793	4.0/4.0	228-229	173	1.4204 (20°C)	

Table 2. Uranium extraction data for various organic solv

1	2	3	4	5	6	7
Group no.	Organic layer	Aqueous layer (21 g/L uranium nitrate)	Req. no.	Concentration uranium in aqueous layer (g/g)	Total uranium in 10 cc aqueous layer (g)	Concentration uranium in organic layer (g/g)
Ethers						
1	Dibutyl carbitol	0.1 N nitric acid	241184 241185	0.009764	0.0981282	0.000038
	Dibutyl carbitol	0.1 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	241186 241187	0.000093	0.00139314	0.010545
	Dibutyl carbitol	30% nitric acid	241188 241189	0.003305	0.03830495	0.006432
2	1,8-bis-(THFA)-n-octyl ether	0.1 N nitric acid	660739	0.009973	0.10022865	0.00024
	1,8-bis-(THFA)-n-octyl ether	0.1 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	660740	0.000042	0.00062916	0.008387
	1,8-bis-(THFA)-n-octyl ether	30% nitric acid	660741	0.002631	0.03049329	0.00532
	1,8-bis-(THFA)-n-octyl ether	30% nitric acid repeat	241242 calculated 241243	0.0026 lost to salvage	0.030134	calculated 0.0072777083
3	Di-n-propyl ether of 2,5-bis(hydroxymethyl)THF	0.1 N nitric acid	241211 241212	0.009562	0.0960981	0.000048
	Di-n-propyl ether of 2,5-bis(hydroxymethyl)THF	0.1 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	241213 calculated 241214	0.000058 lost to salvage	0.00086884	calculated 0.0107751261
	Di-n-propyl ether of 2,5-bis(hydroxymethyl)THF	30% nitric acid	241215 241216	0.0032	0.037088	0.006588

Table 2. Uranium extraction data for various organic solvents

5	6	7	8	9	10	11
Uranium in is layer /g)	Total uranium in 10 cc aqueous layer (g)	Concentration uranium in organic layer (g/g)	Total uranium in 10 cc organic layer (g)	Check sum: total uranium (aqueous + organic)	Distribution ratio: total uranium in 10 cc organic/total uranium in 10 cc aqueous	Comments
9764	0.0981282	0.000038	0.0003249	0.0984531	0.00331	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0093	0.00139314	0.010545	0.09015975	0.09155289	64.71693	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
3305	0.03830495	0.006432	0.0549936	0.09329855	1.43568	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
9973	0.10022865	0.00024	0.002304	0.10253265	0.02299	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0042	0.00062916	0.008387	0.0805152	0.08114436	127.97253	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
2631	0.03049329	0.00532	0.051072	0.08156529	1.67486	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
026 salvage	0.030134	calculated 0.0072777083	calculated 0.069886	Sample lost to salvage...calculated 0.1	2.31851 calculated	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
9562	0.0960981	0.000048	0.0004416	0.0965397	0.00460	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0058 salvage	0.00086884	calculated 0.0107751261	calculated 0.09913116	Sample lost to salvage...calculated 0.1	114.09599 calculated	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
032	0.037088	0.006588	0.0606096	0.0976976	1.63421	Col. 10 calculated using Columns 6 and 8 and approximate solution densities

Table 2 (continued)

1	2	3	4	5	6	7
Group no.	Organic layer	Aqueous layer (21 g/L uranium nitrate)	Req. no.	Concentration uranium in aqueous layer (g/g)	Total uranium in 10 cc aqueous layer (g)	Concentration uranium in organic layer (g/g)
4	THFA-n-butyl ether	0.1 N nitric acid	660743	0.009696	0.0974448	0.00013
	THFA-n-butyl ether	0.1 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	660743	0.000106	0.00158788	0.010388
	THFA-n-butyl ether	30% nitric acid	660744	0.003556	0.04121404	0.006107
5	THFA-n-hexyl ether	0.1 N nitric acid	241197 241198	0.009444	0.0949122	
	THFA-n-hexyl ether	0.1 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	241199 241200 calculated	0.00008	0.0011984	Error...too low!! 0.0026 0.0111013034
	THFA-n-hexyl ether repeat	0.1 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	241236 241237	0.0000368	0.000551264	0.010794
	THFA-n-hexyl ether	30% nitric acid	241201 241202	0.005261	0.06097499	Radio chem: $\pm 20-30\%$ 0.0031
	THFA-n-hexyl ether repeat	30% nitric acid repeat	241238 calculated 241239	0.004 lost to salvage	0.04636	calculated 0.0060269663

Table 2 (continued)

5	6	7	8	9	10	11
Concentration uranium in aqueous layer (g/g)	Total uranium in 10 cc aqueous layer (g)	Concentration uranium in organic layer (g/g)	Total uranium in 10 cc organic layer (g)	Check sum: total uranium (aqueous + organic)	Distribution ratio: total uranium in 10 cc organic/total uranium in 10 cc aqueous	Comments
009696	0.0974448	0.00013	0.001196	0.0986408	0.01227	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
00106	0.00158788	0.010388	0.0955696	0.09715748	60.18692	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
03556	0.04121404	0.006107	0.0561844	0.09739844	1.36323	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
09444	0.0949122		0	0.0949122		Col. 10 calculated using Columns 6 and 8 and approximate solution densities
00008	0.0011984	Error...too low!! 0.0026 0.0111013034	0.02314 0.0988016	0.0243384 0.1 calculated	19.30908 error 82.44459 calculated	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
000368	0.000551264	0.010794	0.0960666	0.096617864	174.26605	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
05261	0.06097499	Radio chem: ± 20-30% 0.0031	0.02759	0.08856499	0.45248	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
004	0.04636	calculated 0.0060269663	calculated 0.05364	Sample lost to salvage...calculated 0.1	1.15703 calculated	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
salvage						

Table 2 (continued)

1	2	3	4	5	6	7
Group no.	Organic layer	Aqueous layer (21 g/L uranium nitrate)	Req. no.	Concentration uranium in aqueous layer (g/g)	Total uranium in 10 cc aqueous layer (g)	Concentration uranium in organic layer (g/g)
6	1,5-di-n-propoxy pentane	0.1 N nitric acid	660745	0.010048	0.1009824	less than 0.0000
	1,5-di-n-propoxy pentane	0.1 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	660746	0.000733	0.01098034	0.010129
	1,5-di-n-propoxy pentane	30% nitric acid	660747	0.007424	0.08604416	0.001861
7	Di-n-propyl ether of di-propylene glycol	0.1 N nitric acid	660749	0.009878	0.0992739	0.000003
	Di-n-propyl ether of di-propylene glycol	0.1 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	660750	0.000311	0.00465878	0.009477
	Di-n-propyl ether of di-propylene glycol	30% nitric acid	241176	0.00536	0.0621224	0.00457
8	Tri-n-propyl ether of glycerine	0.1 N nitric acid	241191 241192	0.0095 4	0.095475	0.00000088
	Tri-n-propyl ether of glycerine	0.1 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	241193 241194	0.00064	0.0095872	0.009336
	Tri-n-propyl ether of glycerine	30% nitric acid	241195 241196	0.0074	0.085766	0.001648

Table 2 (continued)

	6	7	8	9	10	11
Concentration in organic layer (g)	Total uranium in 10 cc aqueous layer (g)	Concentration uranium in organic layer (g/g)	Total uranium in 10 cc organic layer (g)	Check sum: total uranium (aqueous + organic)	Distribution ratio: total uranium in 10 cc organic/total uranium in 10 cc aqueous	Comments
0048	0.1009824	less than 0.000001	0.0000089	0.1009913	0.00009	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0733	0.01098034	0.010129	0.0901481	0.10112844	8.20996	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
07424	0.08604416	0.001861	0.0165629	0.10260706	0.19249	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
9878	0.0992739	0.000003	0.00002571	0.09929961	0.00026	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0311	0.00465878	0.009477	0.08121789	0.08587667	17.43330	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0536	0.0621224	0.00457	0.0391649	0.1012873	0.63045	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
095	0.095475	0.00000088	0.000007541	0.0954825416	0.00008	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0064	0.0095872	0.009336	0.08000952	0.08959672	8.34545	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
074	0.085766	0.001648	0.01412336	0.09988936	0.16467	Col. 10 calculated using Columns 6 and 8 and approximate solution densities

Table 2 (continued)

1	2	3	4	5	6	7
Group no.	Organic layer	Aqueous layer (21 g/L uranium nitrate)	Req. no.	Concentration uranium in aqueous layer (g/g)	Total uranium in 10 cc aqueous layer (g)	Concentration uranium in organic layer (g/g)
9	Di-n-butyl ether of 2-methyl-1,3-propanediol	0.1 N nitric acid	241244 241245	0.010239	0.10290195	
	Di-n-butyl ether of 2-methyl-1,3-propanediol	0.1 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	241246 calculated 241247	0.0025	0.03745	calculated 0.0076560588
	Di-n-butyl ether of 2-methyl-1,3-propanediol	30% nitric acid	241248 241249	0.008724	0.10111116	0.000359
Hydroxy ethers						
10	2-n-propoxy-4-methyl-4-hydroxy pentane	0.1 N nitric acid	241223 241224	0.00965	0.0969825	0.00017
	2-n-propoxy-4-methyl-4-hydroxy pentane	0.1 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	241225 calculated 241226	0.00039 lost to salvage	0.0058422	calculated 0.0110643713
	2-n-propoxy-4-methyl-4-hydroxy pentane	30% nitric acid	241227 calculated 241228	0.0044 lost to salvage	0.050996	calculated 0.0057584019
Ether ketones						
11	Pentoxone (4-methoxy-4-methyl-2-pentanone)	0.1 N nitric acid	241753 241754	0.00717	0.0720585	
	Pentoxone	0.1 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	241755 calculated 241756	0.000071 lost to salvage	0.00106358	calculated 0.0109929356
	Pentoxone	30% nitric acid	241757 241758	0.005098	0.05908582	0.0049

Table 2 (continued)

5	6	7	8	9	10	11
Concentration of uranium in organic layer (g/g)	Total uranium in 10 cc aqueous layer (g)	Concentration of uranium in organic layer (g/g)	Total uranium in 10 cc organic layer (g)	Check sum: total uranium (aqueous + organic)	Distribution ratio: total uranium in 10 cc organic/total uranium in 10 cc aqueous	Comments
0239	0.10290195		0	0.10290195	0.00000	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
025	0.03745	calculated 0.0076560588	calculated 0.06255	Sample lost to salvage...calculated 0.1	1.67023 calculated	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
08724	0.10111116	0.000359	0.00293303	0.10404419	0.02901	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0965	0.0969825	0.00017	0.0014467	0.0984292	0.01492	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0039 salvage	0.0058422	calculated 0.0110643713	calculated 0.0941578	sample lost to salvage...calculated 0.1	16.11684 calculated	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0044 salvage	0.050996	calculated 0.0057584019	calculated 0.049004	sample lost to salvage...calculated 0.1	0.96094 calculated	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0717	0.0720585		0	0.0720585	0.00000	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0071 salvage	0.00106358	calculated 0.0109929356	calculated 0.09893642	sample lost to salvage...calculated 0.1	93.02208 calculated	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
05098	0.05908582	0.0049	0.0441	0.10318582	0.74637	Col. 10 calculated using Columns 6 and 8 and approximate solution densities

Table 2 (continued)

1	2	3	4	5	6	7
Group no.	Organic layer	Aqueous layer (21 g/L uranium nitrate)	Req. no.	Concentration uranium in aqueous layer (g/g)	Total uranium in 10 cc aqueous layer (g)	Concentration uranium in organic layer (g/g)
12	Pentoxone (4-methoxy-4-methyl-2-pentanone) 4 mol/mol of uranium in dihexyl ether	0.1 N nitric acid	241779 241780	0.009772	0.0982086	0.0000017
	Pentoxone (4 mol/mol uranium in dihexyl ether)	0.1 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	241781 241782	0.003146	0.04712708	0.005678
	Pentoxone (4 mol/mol uranium in 21.84 g/L of dihexyl ether)	30% nitric acid	241783 241784	0.008571	0.09933789	0.00016
13	Pentoxone (4-methoxy-4-methyl-2-pentanone) 51 mol/mol uranium in dihexyl ether (20.1 g + 39.5 g ether)	0.01 N nitric acid pH = 2.44	241793 241794	0.00884	0.077269556 (in 8.7409 g)	0.00056
	Pentoxone (51 mol/mol uranium in dihexyl ether)	0.01 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	241795 241796	0.000526	0.0075385794 (in 14.3319 g)	0.006533
Organic acids						
14	2-ethylhexanoic acid (acid hydrolysis product)	0.1 N nitric acid	241217 241218	0.009755	0.09803775	0.000036
	2-ethylhexanoic acid	0.1 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	241219 241220	0.006104	0.09143792	0.0011
	2-ethylhexanoic acid	30% nitric acid	241221 241222	0.008489	0.09838751	0.0000065

Table 2 (continued)

6	7	8	9	10	11
Total uranium in 10 cc aqueous layer (g)	Concentration uranium in organic layer (g/g)	Total uranium in 10 cc organic layer (g)	Check sum: total uranium (aqueous + organic)	Distribution ratio: total uranium in 10 cc organic/total uranium in 10 cc aqueous	Comments
0.0982086	0.0000017	0.000013804	0.098222404	0.00014	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0.04712708	0.005678	0.04610536	0.09323244	0.97832	Col. 10 calculated using Column 6 and 8 and approximate solution densities
0.09933789	0.00016	0.0012992	0.10063709	0.01308	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0.077269556 (in 8.7409 g)	0.00056	(in 7.5437 g) 0.004224472	0.081494028	0.05467	Aqueous layer: 10.448 g/10.6 cc Organic: 7.5437 g/9.3 cc
0.0075385794 (in 14.3319 g)	0.006533	(in 8.741 g) 0.057104953	0.0646435324	7.57503	Aqueous layer: 14.3319 g/9.9 cc Organic: 8.7409 g/10.2 cc
0.09803775	0.000036	0.00032508	0.09836283	0.00332	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0.09143792	0.0011	0.009933	0.10137092	0.10863	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0.09838751	0.0000065	0.000058695	0.098446205	0.00060	Col. 10 calculated using Columns 6 and 8 and approximate solution densities

Table 2 (continued)

1	2	3	4	5	6	7
Group no.	Organic layer	Aqueous layer (21 g/L uranium nitrate)	Req. no.	Concentration uranium in aqueous layer (g/g)	Total uranium in 10 cc aqueous layer (g)	Concentration uranium in organic layer (g/g)
Amides						
15	Isobutyryl-di-(2-ethylhexyl) amide	0.1 N nitric acid	241177	0.009152	0.0919776	0.001129
	Isobutyryl-di-(2-ethylhexyl) amide	0.1 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	241178	0.000036	0.00053928	0.011833
	Isobutyryl-di-(2-ethylhexyl) amide	30% nitric acid	241179	0.00057	0.0066063	0.010256
Diketones in dihexyl ether						
16	Dihexyl ether	0.1 N nitric acid	241761 241762	0.009836	0.0988518	0.000001
	Dihexyl ether	0.1 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	241763 calculated 241764	0.003543 0.0039371235	0.05307414 0.05897811	0.005173
	Dihexyl ether	30% nitric acid	241765 241766	0.008525	0.09880475	0.000092
17	Dibenzoylmethane, 4 mol/mol of uranium, or 0.168 mol/L of dihexyl ether	0.1 N nitric acid	241767 241768	0.009816	0.0986508	0.0000052
	Dibenzoylmethane, 4 mol/mol of uranium, or 0.168 mol/L of dihexyl ether	0.1 N nitric acid + 1/2 sat. $\text{Ca}(\text{NO}_3)_2$	241769 241770	0.003506	0.05251988	0.004983
	Dibenzoylmethane, 4 mol/mol of uranium, or 0.168 mol/L of dihexyl ether	30% nitric acid	241771 241772	0.008625	0.09996375	0.00015

Table 2 (continued)

5	6	7	8	9	10	11
Concentration um in as layer (g)	Total uranium in 10 cc aqueous layer (g)	Concentration uranium in organic layer (g/g)	Total uranium in 10 cc organic layer (g)	Check sum: total uranium (aqueous + organic)	Distribution ratio: total uranium in 10 cc organic/total uranium in 10 cc aqueous	Comments
9152	0.0919776	0.001129	0.00965295	0.10163055	0.10495	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0036	0.00053928	0.011833	0.10117215	0.10171143	187.60597	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
057	0.0066063	0.010256	0.0876888	0.0942951	13.27351	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
836	0.0988518	0.000001	0.00000793	0.09885973	0.00008	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
543	0.05307414			0.1	0.77292	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
71235	0.05897811	0.005173	0.04102189		0.69554 calculated	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
525	0.09880475	0.000092	0.00072956	0.09953431	0.00738	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
816	0.0986508	0.0000052	0.000041756	0.098692556	0.00042	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
506	0.05251988	0.004983	0.04001349	0.09253337	0.76187	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
625	0.09996375	0.00015	0.0012045	0.10116825	0.01205	Col. 10 calculated using Columns 6 and 8 and approximate solution densities

Table 2 (continued)

1	2	3	4	5	6	7
Group no.	Organic layer	Aqueous layer (21 g/L uranium nitrate)	Req. no.	Concentration in aqueous layer (g/g)	Total uranium in 10 cc aqueous layer (g)	Concentration in organic layer (g/g)
18	Dibenzoylmethane, 7.8 mol/mol of uranium, or 0.33 mol/L of dihexyl ether (saturated)	0.01 N nitric acid 247197 pH = 2.44	0.009188	0.0861724144	(in 9.3788 g) 0.00071	
	Dibenzoylmethane, 7.8 mol/mol of uranium, or 0.33 mol/L of dihexyl ether (saturated)	0.01 N nitric acid 241799 + 1/2 sat. Ca(NO ₃) ₂	0.002694	0.0374121168	(in 13.8872 g) 0.0026	
19	Benzoylacetone, 4 mol/mol of uranium, or 0.168 mol/L of dihexyl ether	0.1 N nitric acid 241773 + 1/2 sat. Ca(NO ₃) ₂	0.00985	0.0989925		0.00000012
	Benzoylacetone, 4 mol/mol of uranium, or 0.168 mol/L of dihexyl ether	0.1 N nitric acid 241775 + 1/2 sat. Ca(NO ₃) ₂	0.003256	0.04877468		0.005394
	Benzoylacetone, 4 mol/mol of uranium, or 0.168 mol/L of dihexyl ether	30% nitric acid 241777	0.008556	0.09916404		0.00018
20	Benzoylacetone, 18.7 mol/mol of uranium, or 0.786 mol/L of dihexyl ether (saturated)	0.01 N nitric acid 241801 pH = 2.44	0.06251693	(calculated)	0.0019	
	Benzoylacetone, 18.7 mol/mol of uranium, or 0.786 mol/L of dihexyl ether (saturated)	0.01 N nitric acid 241803 + 1/2 sat. Ca(NO ₃) ₂	0.002268	0.0315850752	(in 13.9264 g) 0.0041	
21	Thenoylfluoroacetone, 4 mol/mol of uranium, or 0.168 mol/L of dihexyl ether	0.1 N nitric acid 241785	0.008092	0.0813246		0.002922
	Thenoylfluoroacetone, 4 mol/mol of uranium, or 0.168 mol/L of dihexyl ether	0.1 N nitric acid 241787 + 1/2 sat. Ca(NO ₃) ₂	0.001391	0.02083718		0.009849
	Thenoylfluoroacetone, 4 mol/mol of uranium, or 0.168 mol/L of dihexyl ether	30% nitric acid 241789	0.008642	0.10016078		0.00021
22	Thenoylfluoroacetone, 43.2 mol/mol of uranium, or 1.81 mol/L of dihexyl ether	0.01 N nitric acid 241805 pH = 2.44	0.0025	0.0232335	(in 9.2934 g) 0.00807	
	Thenoylfluoroacetone, 43.2 mol/mol of uranium, or 1.81 mol/L of dihexyl ether	0.01 N nitric acid 241807 + 1/2 sat. Ca(NO ₃) ₂	0.00044	0.00605858	(in 13.7695 g) 0.009277	

Table 2 (continued)

6	7	8	9	10	11
Total uranium in 10 cc aqueous layer (g)	Concentration uranium in organic layer (g/g)	Total uranium in 10 cc organic layer (g)	Check sum: total uranium (aqueous + organic)	Distribution ratio: total uranium in 10 cc organic/total uranium in 10 cc aqueous	Comments
0.0861724144 (in 9.3788 g)	0.00071	(in 8.4806 g) 0.006021226	0.0921936404	0.06987	Aqueous layer: 9.3788 g/9.6 cc Organic: 8.4806 g/10.4 cc
0.0374121168 (in 13.8872 g)	0.0026	(in 8.9203 g) 0.02319278	0.0606048968	0.61993	Aqueous layer: 13.8872 g/9.5 cc Organic: 8.9203 g/10.6 cc
0.0989925	0.00000012	0.000000958	0.0989934588	0.00001	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0.04877488	0.005394	0.04309806	0.09187294	0.88361	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0.09916404	0.00018	0.0014382	0.10060224	0.01450	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0.08251693 (calculated)	0.0019	0.01648307	0.099	0.19975 (calculated)	Aqueous layer: 9.4181 g/9.4 cc Organic: 8.6753 g/10.6 cc
0.0315850752 (in 13.9264 g)	0.0041	(calculated) 0.067414924 0.0356905	(calculated) 0.099 0.0673	(calculated) 2.13439 1.12998	Aqueous layer: 13.9264 g/9.4 cc Organic: 8.705 g/10.3 cc
0.0813246	0.002922	0.02349288	0.10481748	0.28888	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0.02083718	0.009849	0.07918596	0.10002314	3.80022	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0.10016078	0.00021	0.0016884	0.10184918	0.01686	Col. 10 calculated using Columns 6 and 8 and approximate solution densities
0.0232335 (in 9.2934 g)	0.00807	(in 9.616 g) 0.07760112	0.10083462	3.34005	Aqueous layer: 9.2934 g/9.4 cc Organic: 9.616 g/10.4 cc
0.00605858 (in 13.7695 g)	0.009277	(in 9.9892 g) 0.092669808	0.0987283884	15.29563	Aqueous layer: 13.7695 g/9.4 cc Organic: 9.9892 g/10.4 cc

Comparing the values in Column 10, notice that the THFA-ethers (Groups 2 through 5) were either equivalent to or better than dibutyl carbitol at extracting uranyl nitrate. Di-n-propyl ether of dipropylene glycol (Group 7) was ~27% as efficient as dibutyl carbitol. Di-propoxy pentane (Group 6) was essentially equivalent to the tri-propyl ether of glycerin (Group 8), both being ~12% as efficient as dibutyl carbitol as an extractant. Both the di-n-butyl ether of 2-methyl-1,3-propanediol (Group 9) and the di-n-hexyl ether (Group 16) were comparatively poor extractants of uranyl nitrate, exhibiting extraction coefficients of 0.7 to 1.7 for half-saturated solutions of calcium nitrate. Of course, these solvents could be used if a sufficient number of contactors were employed.

Hydroxy Ethers. An attempt to make a di-n-propyl ether of hexylene glycol or 4-methyl-2,4-pentanediol resulted in the formation of only the monoether or 2-n-propoxy-4-methyl-4-hydroxy pentane. The sodium would not attack the proton at the tertiary 4-hydroxy position. Nevertheless, this ether proved to be quite insoluble in water (see Table 3); hence, it was tested for its affinity for uranyl nitrate. The extraction data are summarized under Group 10, Table 2. This solvent exhibited good distribution coefficients for uranyl nitrate in half-saturated calcium nitrate in 0.1 N nitric acid. In fact, its distribution coefficients were comparable to those of di-n-propylether of di-propylene glycol (Group 7).

Table 3. Physical properties of hydroxy ether

Solvent	Density (g/cc)	Solubility in water starting with 4 cc each (vol. org/vol.aq. phase)
2-n-propoxy-4-methyl-4-hydroxypentane (hexylene glycol derivative)	0.851 $\pm$ 0.76%	4.0/3.9

Ether Ketones. Pentoxone® (a registered trademark of Shell Chemical Co.) or 4-methoxy-4-methyl-2-pentanone reportedly was used in the past as a solvent for resin coating systems.⁵ Recently, however, this solvent proved difficult to obtain. Small 100-g quantities were finally obtained from ICN Chemicals of Costa Mesa, California, at the high cost of ~\$1.00/g. Selected physical data for this solvent are listed in Table 4. This solvent has high water solubility; hence, it would not be an attractive extraction solvent. However, because of its favorable molecular geometry, extraction experiments were completed. These data are summarized in Table 2 under Group 11. One can see that Pentoxone® has good extraction coefficients.

Table 4. Physical properties of an ether ketone

Solvent	Density (g/cc)	Solubility in water starting with 4 cc each (vol. org/vol ag. phase)	Boiling point (°C)	Flash point (°F)	Comments
Pentoxone (4-methoxy-4-methyl-2-pentanone)	0.90	2.6/5.2	157-162	141	Single phase; very water soluble

To take advantage of its affinity for uranyl nitrate and to reduce losses of inventory to water, the authors dissolved Pentoxone® in di-n-hexyl ether in two different concentrations: 21.8 g/L, representing 4 mol for every mole of uranyl nitrate (Group 12, Table 2), and 20.1 g

Pentoxone®/39.5 g hexyl ether or 51 mol Pentoxone® per mole of uranyl nitrate (Group 13, Table 2). Table 2 indicates that the very dilute Pentoxone® in hexyl ether extracted no better than hexyl ether alone. The more concentrated Pentoxone® in Group 13 extracted slightly better and behaved similarly to di-propoxy pentane and tri-n-propyl ether of glycerine.

The excellent extraction behavior of the neat Pentoxone® still makes this ether ketone an interesting solvent. Replacement of the methoxy with a longer chain, less water soluble group (such as butoxy) has been attempted. Although initial attempts at synthesis were unsuccessful, additional attempts will be made.

Organic Acids. Because all oxygen-bearing organic solvents degrade to some extent in contact with nitric acid, and because oxalic acid creates problems in the current extraction stream, a concern arose over the affinity of various degradation or hydrolysis products for uranyl nitrate. Because of its low water solubility and low odor (compared with shorter chain acids), 2-ethyl hexanoic acid was chosen to represent monofunctional organic acids. Table 5 lists various physical properties of this acid. The extraction results listed in Table 2 under Group 14 indicate that 2-ethyl hexanoic acid has very little affinity for uranyl nitrate. Although other acids, particularly the difunctional acids, need to be tested, these results give the authors a feeling that certain monoacids might not present problems during extraction.

Table 5. Physical properties of an organic acid

Solvent	Purity	Density (g/cc)	Boiling point (°C)	Flash point (°F)	Refractive index at temperature (°C)
2-Ethylhexanoic acid	99+	0.903	228	>230	1.4250 (20°C)

Amides. Amides are powerful extraction solvents.² Although amides were expected to produce intractable, viscous emulsions with water, and although they were expected to suffer degradation by nitric acid, two amides were synthesized for direct observations. Both the isobutyryl-di(2-ethylhexyl)amide and the lauroyl-di(2-ethylhexyl)amide were synthesized using di-(2-ethylhexyl)amine and the corresponding acid chlorides. Table 6 lists certain physical properties of these amides. Because the lauryl amide was purified with difficulty to 76 mol % amide and 20 mol % water, work was temporarily postponed to work on more forgiving extractants. Uranium extraction data can be found in Table 2, Group 15. The data indicate very high extraction coefficients for both calcium nitrate and 30 wt % nitric acid solutions, which is consistent with values found in the literature.² The stripping coefficient was somewhat high at 0.105.

Table 6. Physical properties of various amides

Solvent	Purity	Density (g/cc)	Solubility in water starting with 4 cc each (vol. org/vol aq. phase)
Isobutyryl-di(2-ethyl hexyl)amide	99+%	0.855 ± 2%	4.1/3.9
Lauroyl-di(2-ethyl hexyl)amide	76% amide, 20% water		

Diketones in Di-n-hexyl ether. The senior staff member recommended that the authors look at three diketones to which the literature for uranium extraction alluded. More specifically, these diketones included dibenzoylmethane, benzoylacetone, and thenoyltrifluoroacetone. Because all three of these diketones are solids at room temperature, a suitable solvent with some polarity was needed. Dihexyl ether was again chosen because of its high boiling point, slight polarity, and modest affinity for uranyl nitrate. At first, solutions were made using enough of each diketone in dihexyl ether to provide 4 mol of diketone for each mole of uranyl nitrate in the aqueous solutions (see Groups 17, 19, and 21 in Table 2).

For the second series, higher concentrations of the diketones were prepared. Two of the ketones had limited solubility in hexyl ether. Three and seven-tenths grams of dibenzoylmethane saturated 50 mL di-n-hexyl ether with heating, which was equivalent to 0.33 mol/L or 7.8 mol of dibenzoylmethane per mole of uranyl nitrate in each aqueous phase (see Group 18, Table 2). Likewise, 6.38 g of benzoylacetone saturated 50 mL of di-n-hexyl ether, which was equivalent to 0.79 mol/L or 18.7 mol of benzoylacetone per mole of uranyl nitrate in each aqueous phase (see Group 20 in Table 2). Thenoyltrifluoroacetone was much more soluble in di-n-hexyl ether than either of the two preceding diketones. The authors dissolved 20 g of this diketone in 50 mL of di-n-hexyl ether, which was equivalent to 1.81 mol/L or 43.2 mol diketone per mole of uranyl nitrate dissolved in each aqueous phase (see Group 22 in Table 2).

Although more thenoyltrifluoroacetone could have dissolved, the density of this last solution exceeded 0.92 g/cm^3 , and the authors decided to use this concentration. The densities for all of the preceding solutions are listed in Table 7. The extraction data in Table 2, particularly the distribution coefficient or ratio in Column 10, indicate that the dibenzoylmethane (Groups 17 and 18) was no better at extracting uranyl nitrate than was the di-n-hexyl ether carrier alone. The same can be said for low concentrations of benzoylacetone (Group 19). A higher concentration of benzoylacetone (Group 20) was somewhat better at extracting uranyl nitrate than the di-n-hexyl ether carrier alone, but the distribution coefficient still was not attractive. The low concentration of thenoyltrifluoroacetone (Group 21) showed modest affinity for extracting uranyl nitrate, with a distribution coefficient of 3.8. The more concentrated solution (Group 22) showed strong affinity for extracting uranium. Unfortunately, the stripping coefficient of 3.34 was much too high to make this an attractive candidate. [Notice that the pH for the higher concentrations of diketones was higher ($\text{pH} = 2.4$) than for the other extractants ($\text{pH} = 1$) tested in this study.] The senior staff member thought that the extraction behavior would be better in a slightly higher pH.]

Table 7. Physical properties of various diketones in dihexyl ether

Solvent	Density (g/cc)
Pentoxone, 0.168 M in dihexyl ether, or 4 mol/mol uranium	$0.812 \pm 4.00\%$ (17°C)
Pentoxone, 20.1 g in 39.5 g hexyl ether, or 51 mol/mol uranium	$0.825 \pm 1.07\%$ (22°C)
Dibenzoylmethane, 0.168 M in dihexyl ether, or 4 mol/mol uranium	$0.803 \pm 1.53\%$ (17°C)
Dibenzoylmethane, 0.33 M in hexyl ether (saturated), or 7.8 mol/mol uranyl nitrate	$0.817 \pm 0.83\%$ (20°C)
Benzoylacetone, 0.168 M in dihexyl ether, or 4 mol/mol uranium	$0.799 \pm 1.18\%$ (17°C)
Benzoylacetone, 0.786 M in hexyl ether (saturated), or 18.7 mol/mol uranyl nitrate	$0.822 \pm 1.01\%$ (22°C)
Thenoyltrifluoroacetone, 0.168 M in dihexyl ether, or 4 mol/mol uranium	$0.804 \pm 1.72\%$ (17°C)
Thenoyltrifluoroacetone, 1.81 M in dihexyl ether, or 43.2 mol/mol uranium	$0.929 \pm 0.70\%$ (22°C)

Future Work

Other ethers are being synthesized and will be tested for affinity for uranyl nitrate. Because the THFA ethers exhibited good extraction parameters, the senior staff member recommended that a larger batch of one representative of this solvent family be obtained to test it in actual centrifugal contactors. Consequently, the authors are negotiating with a manufacturer for a 10-gal batch of THFA-n-hexyl ether. If tests are successful, chronic toxicity, stability, and other issues will need to be addressed. Additional physical measurements are planned on all of the solvents studied so far to complete the physical properties tables.

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