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SURVEY OF SOLVENTS FOR URANIUM EXTRACTION WITHOUT SALTING AGENTS

C. N. Stover, Jr., H. W. Crandall,

D. C. Stewart and P. C. Mayer

January 13, 1950

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SURVEY OF SOLVENTS FOR URANIUM EXTRACTION WITHOUT SALTING AGENTS

C. N. Stover, Jr., H. W. Crandall, D. C. Stewart, and P. C. Mayer
Radiation Laboratory, University of California, Berkeley, California

ABSTRACT

The uranium extraction characteristics of over fifty organic solvents and solvent mixtures have been investigated in a "shotgun survey" in the absence of non-volatile salting agents. Generalizations of the extraction characteristics of the various families of solvents are made, and trends are established within the families. The extraction power by families is in the order ethers < alcohols < ketones. An increase in the number of oxygen atoms per molecule favors the extraction of uranium while an increase in the molecular weight per oxygen atom decreases the extraction.

Certain of the more promising solvents are considered in slightly more detail.

The most suitable solvent located thus far is penta ether (dibutoxytetraethyleneglycol) and cyclohexanone is second-best. No solvent mixtures were found which are any better than the pure solvents.

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INTRODUCTION

Numerous surveys have been made of the solvent extraction of uranium in the presence of salting agents, and continuous extraction processes have been developed using certain solvents.(1-10) However, the fission product storage problem resulting from such processes has made it desirable to find a process which eliminates the non-volatile salting agent.

A study has been initiated in this laboratory to develop such a process. A portion of this study has consisted of a "shotgun survey" of over fifty solvents and solvent mixtures. This survey was designed primarily to locate the most promising solvents as quickly as possible and secondly to reach some understanding of the mechanism of the extraction in order to locate other solvents which should give favorable uranium extraction. Extraction characteristics of certain of the more promising solvents were investigated in slightly more detail. The results of this survey are given in this report in the form of tables, graphs, and text discussion.

EXPERIMENTAL

In all cases the solvents used were the best grades obtainable from the manufacturers and no attempt was made to further purify the solvents for this survey.

The stock solution was prepared from the commercially pure uranium nitrate hexahydrate (Mallinckrodt) and concentrated nitric acid, C. P. (Merck). The solution was 0.50M U, 0.34M HNO_3 to correspond to column conditions in the critical region where uranium extraction becomes more difficult in the absence of a salting agent such as $Al(NO_3)_3$ or HNO_3, and thereby enable the most promising solvents to be located as quickly as possible. The low uranium concentration, however, limited the number of solvents which could be studied

since many solvents showing essentially no extraction at this point are known to give fairly good extraction at higher uranium concentrations.[1,2]

Equal aliquots of stock solution and solvent were equilibrated for two hours at 25°C. After the volume changes were recorded, aliquots of each phase were evaporated, ignited, and weighed as U_3O_8. Material balances were calculated in each case and found to check within ± 3%.

DISCUSSION OF RESULTS

The following values were calculated for each solvent:

$$\text{Distribution Ratio} = K = \frac{\underline{M}\ U\ (\text{organic phase})}{\underline{M}\ U\ (\text{aqueous phase})}$$

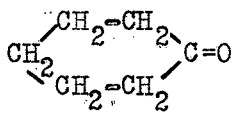
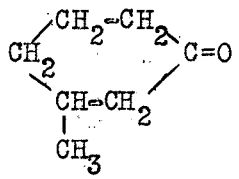
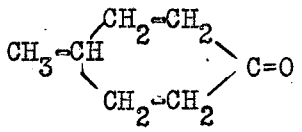
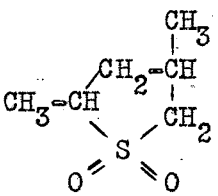
$$\text{Mole Ratio} = \frac{\text{Moles U/cc} \cdot (\text{organic phase})}{\text{Moles pure solvent/cc}}$$

While the distribution ratio is a satisfactory means of describing the extraction characteristics of each individual solvent, it is also advantageous for comparison with other solvents to use the mole ratio which gives a relative measure of the extractability per molecule of solvent. Except for steric or activity effects, the mole ratio should be roughly constant for a homologous series of solvents, and, therefore, gives a more direct comparison. These values for the pure solvents included in this survey are listed in Table I.

In succeeding generalizations involving the mole ratio, only those solvents whose solubility in H_2O is low are considered because the mole ratio cannot be calculated precisely in systems where an appreciable quantity of solvent goes into the aqueous phase since the amount of solvent in the aqueous phase was not determined. When K is high a correction is made in the mole ratio for the dilution of solvent by uranyl nitrate by noting the volume change.

Table I. Pure Solvents*

Solvent	Formula	Distribution Ratio = K	Mole Ratio ($X10^3$)	Solubility in H_2O (Wt. %)	Solubility of H_2O in Solvent (Wt. %)
Ethers					
Diethyl Ether	$C_2H_5OC_2H_5$	0.09		6.9	1.3
Isopropyl ether	$(CH_3)_2CHOCH(CH_3)_2$	0.00	0.00	0.9	0.57
Dibutyl ether	$C_4H_9OC_4H_9$	0.00	0.00	0.03	0.19
Diethyl "Cellosolve"	$C_2H_5OC_2H_4OC_2H_5$	0.29		21.0	3.4
Dibutyl "Cellosolve"	$C_4H_9OC_2H_4OC_4H_9$	0.01	0.96	0.2	0.6
"Phenylcellosolve"	$C_6H_5OC_2H_4OH$	0.00	0.00	2.7	10.8
Dibutyl "Carbitol"	$C_4H_9O(C_2H_4O)_2C_4H_9$	0.09	9.31	0.3	1.4
"Penta Ether" (Dibutoxytetraethyleneglycol)	$C_4H_9O(C_2H_4O)_4C_4H_9$	0.54	57.4	1.3	4.8
Alcohols					
Methyl isobutyl carbinol	$CH_3CH_2CH(CH_3)CH_2OH$	0.01_3	2.62		
2-Ethyl butanol	$CH_3CH_2CH(C_2H_5)CH_2OH$	0.01	0.61	0.43	4.6
2-Ethylhexanediol-1,3	$C_3H_7CH(OH)CH(C_2H_5)CH_2OH$	0.09	6.74	4.2	11.7
Ketones					
Diethyl ketone	$(C_2H_5)_2CO$	0.08			
Hexone	$(CH_3)_2CHCH_2COCH_3$	0.00_4	2.58	2.0	2.2
Methyl n-amyl ketone	$CH_3CO(CH_2)_4CH_3$	0.02	1.10	0.43	1.5
Methyl n-hexyl ketone	$CH_3CO(CH_2)_5CH_3$	0.01	1.07		
Acetophenone	$CH_3COC_6H_5$	0.05	2.66	0.55	1.67
Cyclopentanone	$\begin{matrix} CH_2-CH_2 \\ \vert \quad\quad C=O \\ CH_2-CH_2 \end{matrix}$	0.80	18.7		

Solvent	Formula	Distribution Ratio = K	Mole Ratio ($X10^3$)	Solubility in H_2O (Wt.%)	Solubility of H_2O in Solvent (Wt. %)
Cyclohexanone		0.53	18.4	2.4	
m-methylcyclohexanone		0.24	11.9		
p-methylcyclohexanone		0.30	14.3		
Mesityloxide	$(CH_3)_2C{:}CHCOCH_3$	0.24	10.3	2.8	3.4
Esters					
Ethylacetoacetate	$CH_3COCH_2COOC_2H_5$	0.11		11.6	4.9
Glycol diacetate	$CH_3COOCH_2CH_2OCOCH_3$	0.25		16.4	7.0
Diethyl Maleate	$HCCOOC_2H_5$ ‖ $HCCOOC_2H_5$	0.07	4.82	1.3	2.1
Others					
Dimethylsulfolane		1.68			13.3-15.6
Nitropropane	$CH_3(CH_2)_2NO_2$	0.00	0.00		

*For physical properties of solvents see appendix.

The criterion chosen for an acceptable solvent in this survey was based on the fact that the stock solution corresponds to a point in a column where the aqueous phase is approximately 20% saturated or 25% of a 2.0M U feed. Therefore, if the flow ratio (organic/aqueous) is restricted to less than 3 or 4, to achieve greater than 90% recovery of uranium, K must be greater than 0.2.

A. Pure Solvents

The absence of salting agents increases the water solubility of many solvents making it impossible to study them under the conditions of this survey, e.g., the lower molecular weight cellosolves and carbitols. However, within these solubility limits the simpler members of the various families of compounds were studied, and several general comparisons may be made. The simple, or single, ethers were poor. The polyethers will be discussed separately. The simpler alcohols were somewhat better and the ketones were still better. No comparison may yet be made between primary, secondary, and tertiary alcohols. The esters which were investigated gave still more favorable extraction but were not considered further due to probable acid instability.

Although these comparisons are based on comparatively few solvents, they correspond in many cases to the comparisons made by other investigators using salting agents.[1,2,4,7] It will also be noted that the dipole moments of these families of compounds vary in the same order as their extraction power, i.e., ethers < alcohols < ketones.[11]

From a study of the extraction characteristics within each family of solvents more significant comparisons may be made. The mole ratio values in Table I clearly show that the extraction power is increased by an increase in the number of oxygen atoms per solvent molecule. For example, penta ether

>dibutyl carbitol >dibutyl cellosolve > dibutyl ether.

The K values for ketones, alcohols, and ethers is decreased by an increase in molecular weight per oxygen. For example, in the ketones, diethyl ketone (mol. wt. = 86) > methyl n-amyl ketone (114) > methyl n-hexyl ketone (128); in the alcohols, methyl isobutyl carbinol (88) > 2-ethyl butanol (102); and in the ethers, ethyl ether (74) > dibutyl ether (130).

Inductive effects of substituents within a homologous series probably would be too small to be noted in most cases. However, steric effects are indicated, for example, in the mole ratios of cyclohexanone, p-methylcyclohexanone, m-methylcyclohexanone, and hexone since the mole ratios decrease in the order of increasing steric hindrance. Possibly another example of this may be seen in comparing the mole ratios of methyl isobutyl carbinol and 2-ethyl butanol.

It may be seen that the functional group is the oxygen atom, especially when it is in the ketone form or in a series of ether linkages, and that its ability to extract uranium is a function of its electronegativity.

Only one nitro-compound, 1-nitropropane, was investigated as a pure solvent and gave no indication of extraction which is in accordance with observations in the presence of salting agents.[1,2,4,7]

B. Mixtures

To further investigate the mechanism of uranium extraction, various solvent mixtures were studied. The distribution ratios and mole ratios* of the mixtures studied are listed in Table II.

---

*Mole Ratio (mixtures) = $\frac{\text{moles U/c.c. (organic phase)}}{\text{moles total solvents/cc.}}$

Table II. Solvent Mixtures

Composition*	Distribution Ratio = K	Mole Ratio** ($X10^3$)
Similar		
20% Hexone, 80% Cyclohexanone	0.31	12.6
50% Dibutyl "Carbitol", 50% Cyclohexanone	0.07	2.63
20% Penta Ether, 80% Cyclohexanone	0.58	21.6
50% Penta Ether, 50% Cyclohexanone	0.57	27.8
80% Penta Ether, 20% Cyclohexanone	0.58	42.9
50% Polypropylene Glycol 425, 50% Cyclohexanone	0.73	32.8
50% Polypropylene Glycol 1025, 50% Cyclohexanone	0.54	32.2
50% Butyl "Cellosolve", 50% Penta Ether	0.47	32.1
20% Dimethylsulfolane, 80% Cyclohexanone	0.50	17.3
50% Dimethylsulfolane, 50% Cyclohexanone	0.46	17.3
50% Dimethylsulfolane, 50% Penta Ether	0.52	30.2
Different		
20% Xylene, 80% Cyclohexanone	0.23	9.71
20% Aniline, 80% Cyclohexanone	0.19	5.74
20% Diethyl aniline, 80% Cyclohexanone	0.20	7.54
20% Dimethylsulfolane, 80% Benzene	0.00	0.00

*Percent by volume

**Mole Ratio (mixtures) = $\frac{\text{moles U/cc (organic phase)}}{\text{moles total solvents/cc}}$

From the study of pure solvents it is possible to make certain predictions concerning the behavior of solvents in mixture. Two solvents having measurable extraction characteristics by themselves and having no mutual interaction involving the active oxygen atom should give a mole ratio corresponding approximately to the geometric mean of the individual values. That this is verified may be seen by studying the "similar" mixtures in the first portion of Table II. For example, the mole ratios of the three penta ether-cyclohexanone mixtures vary between 18.4 for pure cyclohexanone and 57.4 for pure penta ether.

The polypropylene glycols, which are too viscious to be studied by themselves, and butyl "cellosolve", which is too water soluble to be studied by itself, were studied in mixtures of cyclohexanone or penta ether and indicate acceptable extraction characteristics.

Correspondingly, a mixture of two solvents having any interaction which would decrease the strength of the functional group would give a mole ratio considerably less than the geometric mean of their individual values. This is illustrated in the second portion of Table II, for example, in the appreciable lowering produced by the addition of 20% xylene to cyclohexanone. This indicates an effect other than the dilution of the cyclohexanone. In the case of dimethylsulfolane, which has a high mole ratio by itself, the addition of benzene strongly inhibits the extraction. While the mechanism of this interaction is not yet clearly understood, it may be that the benzene prevents interaction between the dimethylsulfolane molecules, with the result that the dimethylsulfolane is largely in the aqueous phase.

Presumably, two compounds could interact to increase the strength of the functional group and correspondingly enhance the extraction characteristics, but no such combination has yet been found.

It is possible to form a compound of uranium with one of the organic compounds present in a mixture of solvents which is soluble in the second organic compound contained in the solvent mixture. This may be visualized as "coating" the uranium atom with an organic layer which then is soluble in solvents having little or no interaction with uranyl nitrate. Examples of this are tributylphosphate in hexane,[12,13] butyl phosphoric acid in dibutyl ether,[14] and TTA (thenoyltrifluoracetone) in hexone.[15] Some attention has been given to systems of this type which will be discussed in a separate report.

DISTRIBUTION STUDIES USING LABELED $UO_2(NO_3)_2$

Two of the more promising solvents and one solvent mixture were studied in somewhat more detail using uranium stock solutions containing a measured quantity of U^{233} tracer. The equilibration period for these studies was thirty minutes at 25°C. Aliquots of each phase were then plated on stainless steel plates with a little tetraethylene glycol added to produce a spreading film.[16] The plates were then dried, ignited, and counted in the usual manner.

The results of these studies on cyclohexanone, penta ether, and on the mixture 50%* polypropylene glycol 425, 50% cyclohexanone are presented in Figures 1 and 2.

MECHANISM OF EXTRACTION

Katzin and Sullivan[17] have demonstrated that four molecules of water per uranyl nitrate transfer into the organic phase in diethyl ether and methyl isobutyl ketone, showing that the mechanism of uranium extraction into the organic phase involves a competition between water and the solvent for the uranium molecule.

---

*Percent by volume.

The oxygen atom of the solvent molecule may be involved in an ion dipole bond with the uranium atom, R_2O---$\overset{O}{U}$---, or in a hydrogen bond with the water molecules, R_2O---H-$\underset{H}{\overset{O}{O}}$---$\overset{O}{U}$---, where R_2O represents a solvent molecule. Thus, the extractability of a given solvent for uranium should be related to the electronegativity of its oxygen atoms or its dipole moment.

The solubility of water in a solvent may bear, within steric limitations, a relationship to the extractability of the solvent for uranium. Insufficient data are available to draw any conclusions at this time. However, trends favoring this theory may be seen in the polyether series: dibutyl cellosolve, dibutyl carbitol and penta ether.

SUMMARY

The uranium extraction characteristics of over fifty organic solvents and solvent mixtures have been investigated in a "shotgun survey" in the absence of non-volatile salting agents. Generalizations of the extraction characteristics of the various families of solvents have been made, and trends have been established within the families. The extraction power by families is in the order ethers < alcohols < ketones. An increase in the number of oxygen atoms per molecule favored the extraction of uranium while an increase in the molecular weight per oxygen atom decreased the extraction.

Hence, the most favorable solvents for uranium extraction in the ether family would involve numerous oxygen atoms and the lowest molecular weight per oxygen atom within the limits of mutual solubility with water for practical solvent extraction. Many of the solvents investigated gave favorable extraction but were discarded on the basis of mutual solubility with water, acid instability, or unfavorable physical characteristics,* e.g., dimethylsulfolane, cyclopentanone, polypropylene glycols, butyl cellosolve, and glycol diacetate. The most suitable solvent located thus far is penta ether and cyclohexanone is second-best.

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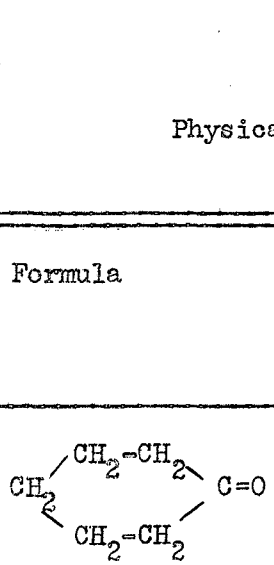
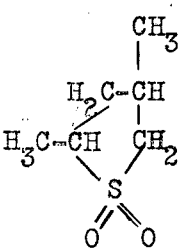
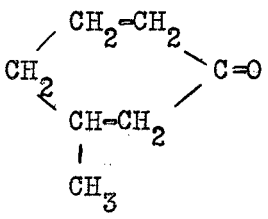
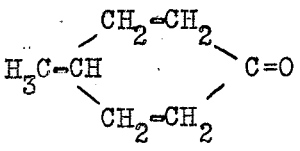
*For physical properties of solvents see appendix.

APPENDIX

Physical Properties of Solvents[21-25]

Solvent	Formula	Mol. Wt.	Sp.Gr. 20/20°C	B.P. °C., 760mm	Flash Pt. (a)	Solubility in H_2O (Wt. %)	Solubility in H_2O (Wt.%)	Viscosity Centipoise 20°C
Cyclohexanone		98.14	0.9478	156.7	117			
1-Nitropropane	$CH_3(CH_2)_2NO_2$	89.09	1.003	132	93			
Penta Ether (Dibutoxytetraethyleneglycol)	$C_4H_9O(C_2H_4O)_4C_4H_9$	306.43	0.9436	237 (50mm)	355	1.3	4.8	5.7
Dibutyl "Carbitol"	$C_4H_9O(C_2H_4O)_2C_4H_9$	218.33	0.8853	254.6	260	0.3	1.4	2.39
Dibutyl "Cellosolve"	$C_4H_9OC_2H_4OC_4H_9$	174.28	0.8374	203.3	185	0.2	0.6	1.34
Diethyl "Cellosolve"	$C_2H_5OC_2H_4OC_2H_5$	118.17	0.8417	121.4	95	21.0	3.4	0.65
Diethyl ketone	$(C_2H_5)_2CO$	86.13	0.8159 (19/4°C)	102.7				
Dimethylsulfolane		148.16	1.1362	(b)	146(°C)		13.3-15.6	
Aniline[c]	$C_6H_5NH_2$	93.12	1.022	184.4		3.4		
Diethyl aniline[c]	$C_6H_5N(C_2H_5)_2$	149.23	0.9351	215.5		1.44 (12°C)		
Methyl n-hexyl ketone	$CH_3CO(CH_2)_5CH_3$	128.21	0.818	170-172				

Solvent	Formula	Mol. Wt.	Sp.Gr. 20/20°C	B.P. °C., 760mm	Flash Pt. (a)	Solubility in H_2O (Wt.%)	Solubility in H_2O (Wt.%)	Viscosity Centipoise 20°C
Dibutyl ether	$C_4H_9OC_4H_9$	130.22	0.7694	142.2	100	0.03	0.19	0.69
Polypropylene glycol 425 (c)		400-450	1.0103		390	completely miscible		30 (d)
Polypropylene glycol 1025 (c)		975-1025	1.0072		440	1.5		77.1 (d)
Butyl "Cellosolve" (c)	$C_4H_9OC_2H_4OH$	118.17	0.9019	171.2	165	completely miscible		6.42
"Phenylcellosolve"	$C_6H_5OC_2H_4OH$	146.22	0.8894	208.3	195	0.99	18.78	5.15
Methyl n-amyl ketone	$CH_3CO(CH_2)_4CH_3$	114.18	0.8166	150.6	120	0.43	1.5	0.65
Acetophenone	$CH_3COC_6H_5$	120.14	1.030	201.7	175	0.55	1.65	1.84
Ethylacetoacetate	$CH_3COCH_2COOC_2H_5$	130.14	1.0261	180.7	185	11.6	4.9	1.64
Mesityloxide	$(CH_3)_2C{:}CHCOCH_3$	98.14	0.8569	128.0	90	2.8	3.4	0.60
Diethyl maleate	$HCCOOC_2H_5$ ‖ $HCCOOC_2H_5$	172.18 (e)	1.0687	225.3	250	1.3	2.1	
m-methylcyclohexanone		112.17	0.915	169				
p-methylcyclohexanone		112.17	0.912	169				

Solvent	Formula	Mol. Wt.	Sp.Gr. 20/20°C	B.P. °C., 760mm	Flash Pt. (a)	Solubility in H_2O (Wt.%)	Solubility in H_2O (Wt.%)	Viscosity Centipoise 20°C
Cyclopentanone	H_2C-CH_2 / H_2C-CH_2 > C=O	84.11	0.9480	130.6				
Sec-butyl alcohol	$CH_3CH_2CHOHCH_3$	74.12	0.808	99.5-100		12.5		
2-Ethyl butanol	$(C_2H_5)_2CHCH_2OH$	102.17	0.8328	148.9	135	0.43	4.6	5.63
2-Ethylhexane-diol-1,3	$C_3H_7CH(OH)CH(C_2H_5)CH_2OH$	146.22	0.9422	244.2	260	4.2	11.7	323
Methyl isobutyl carbinol	$CH_3CH_2CH(CH_3)CH_2OH$	88.15	0.816	128				
Glycol di-acetate	$CH_3COOCH_2CH_2OCOCH_3$	146.14	1.1063	190.5	220	16.4	7.0	2.86
Diethyl ether	$C_2H_5OC_2H_5$	74.12	0.7146	34.5	-40	6.9	1.3	0.25
Hexone (methyl isobutyl ketone)	$(CH_3)_2CHCH_2COCH_3$	100.16	0.8042	115.8	75	2.0	2.2	0.59
Isopropyl ether	$(CH_3)_2CHOCH(CH_3)_2$	102.17	0.7244	68.5	15	0.90	0.57	0.34

(a) Open cup

(b) Decomposes above 220°C.

(c) Investigated in mixtures only

(d) At 100°F.

(e) Calculated[23]

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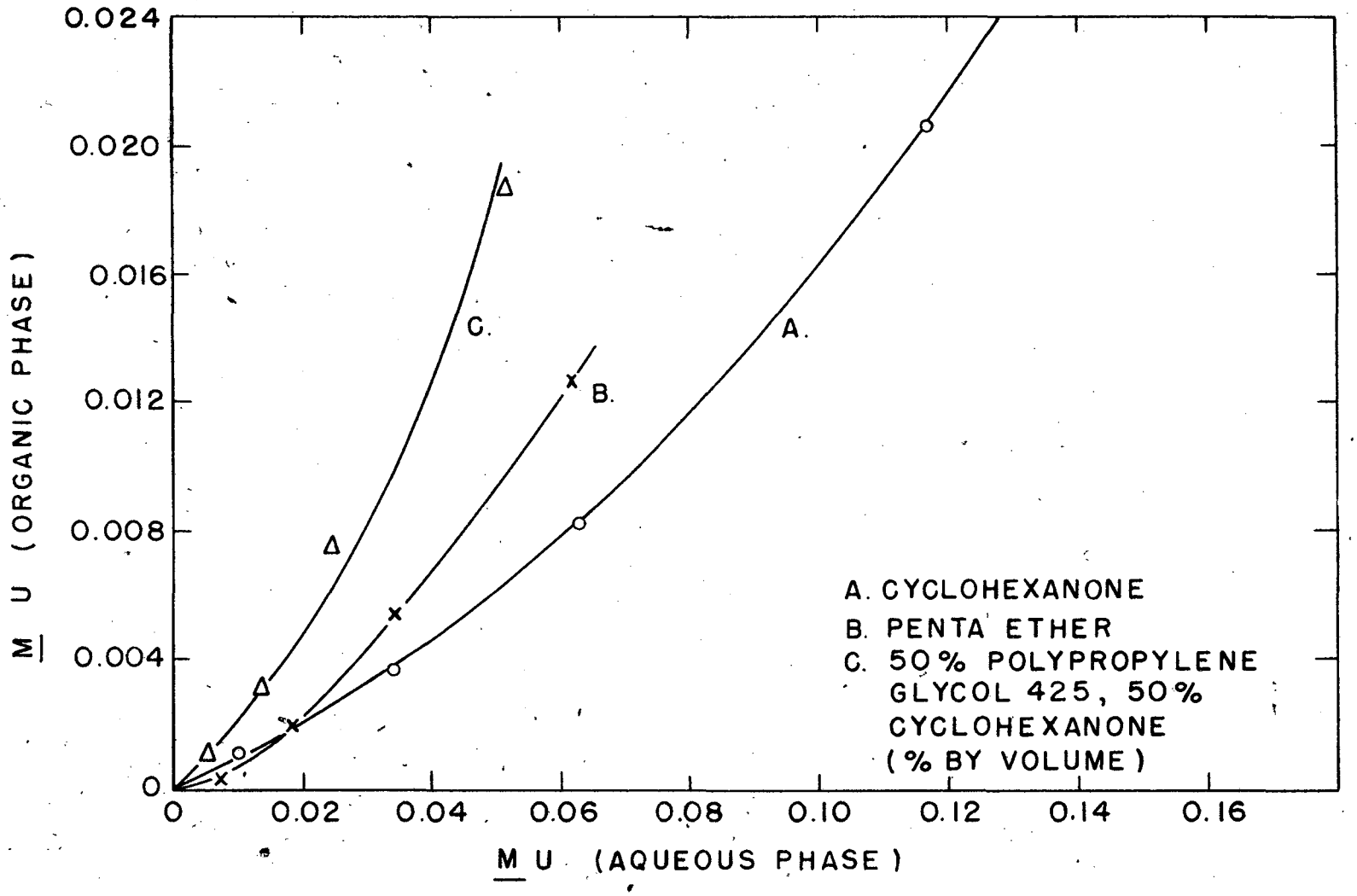

FIG. 1

DISTRIBUTION OF LABELED $UO_2(NO_3)_2$ (0.34 M HNO_3)

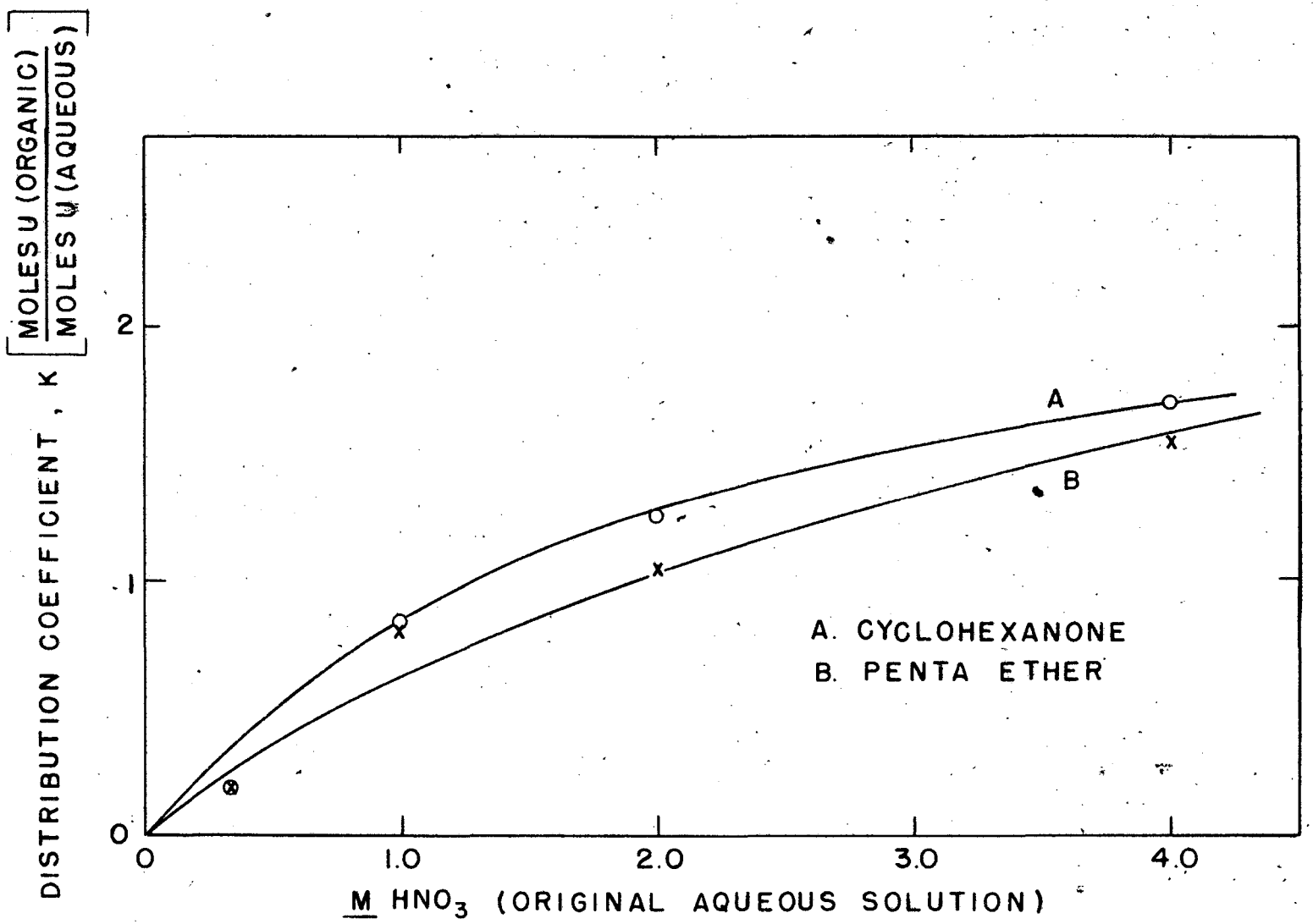

FIG. 2

EFFECT OF INITIAL HNO_3 CONCENTRATION ON EXTRACTION OF LABELED $UO_2(NO_3)_2$

DECLASSIFIED