

**URANIUM SOLUBILITY STUDIES DURING WASTE  
EVAPORATION (U)**

by

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## Uranium Solubility Studies During Waste Evaporation (U)

August 16, 1993

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Uranium Solubility Studies During Waste Evaporation (U)

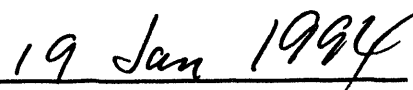
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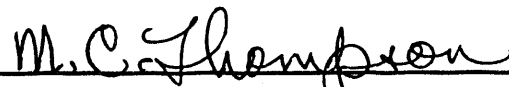
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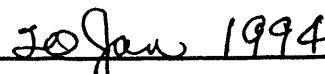




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## 1.0 Introduction

The liquid waste streams from chemical processing of reactor-irradiated targets and fuel are neutralized with excess NaOH and discharged to mild steel waste tanks for interim storage. In the waste tanks, the basic waste separates into two phases, a solid phase containing base-insoluble cations--Fe, Mn, Pu, U, etc.--and a liquid supernate that contains NaOH, NaNO<sub>3</sub>, NaNO<sub>2</sub> (formed by the radiolysis of NaNO<sub>3</sub>), <sup>137</sup>Cs, etc. The supernate also contains very low concentrations of the precipitated cations--U, Pu, Am, etc. To reduce the number of tanks required, and thus the cost of waste storage, the supernate is evaporated to about 70% solids, discharged while hot into "clean" waste tanks. As the solution cools, solids (NaNO<sub>3</sub>, NaNO<sub>2</sub>, etc.) crystallize from the saturated solution and form a solid layer on the bottom of the tank. The supernate is re-evaporated to concentrate the volume further. Evaporation and crystallization are continued until, for tank 41, the tank is almost filled with crystallized salts. During sludge processing the supernate is drawn off the tank, the sludge layer is washed with either water and/or NaOH solution to reduce the Al and sulfate content. These sludge-washing solutions are also evaporated to solids like the supernate.

In the DWPF processing scheme, these salts will be redissolved in water and <sup>137</sup>Cs precipitated with sodium tetraphenylborate in the in-tank precipitation facility. The decontaminated supernate is now mixed with cement and stored as a solid monolith; the precipitated Cs and the base-insoluble solids are encapsulated in glass for permanent storage.

Questions have been raised about the nuclear safety of these operations, particularly for tank 41, where the waste source was waste from the H-Area fuel processing. The uranium in H-Area is mainly enriched U, rather than the depleted U from F-Area Purex processing. One scenario for a potential nuclear accident considers that the salts in tank 41 would dissolve in water, but the enriched uranium solids would not dissolve. The uranium is hypothesized to settle to the bottom of the tank and become concentrated enough to reach a critical mass. A second scenario, promulgated by West Valley, is that uranium would precipitate in the evaporator and form a critical mass in the evaporator. To shed some light on the probable behavior of U in the waste system, the solubility of U in synthetic waste was studied. The results are reported here.

## 2.0 Summary

Evaporation of synthetic waste supernate solutions found that the uranium solubility decreases from 30-40 ppm to 10 ppm as the NaOH concentration increases from 1 to 7M. After U has been precipitated with salts, the solubility of U toward redissolution is 2-5 ppm, demonstrating that the system is not reversible. Attempts to increase the solubility by sparging with CO<sub>2</sub> were unsuccessful. As applied to plant waste evaporation, both supernate and sludge wash solutions will precipitate U on evaporation.

### 3.0 Data and Results

#### Experimental Section

##### Solutions

The starting point for all studies has been the average waste supernate, whose composition is shown in Table I.

Table I. Average Waste Tank Supernate<sup>1</sup>

Compound	Concentration, M
NaOH	1.2, 1.7
NaNO <sub>3</sub>	2.4
NaNO <sub>2</sub>	0.9
NaAlO <sub>2</sub> *	0.5
Na <sub>2</sub> CO <sub>3</sub>	0.2
Na <sub>2</sub> SO <sub>4</sub>	0.2
NaCl	0.03
KF	0.02
Na <sub>2</sub> C <sub>2</sub> O <sub>4</sub>	0.02
Na <sub>3</sub> PO <sub>4</sub>	0.01

\* Al added as Al(NO<sub>3</sub>)<sub>3</sub>•9H<sub>2</sub>O; NaOH and NaNO<sub>3</sub> adjusted to compensate.  
U spike added as UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub> solution with Al(NO<sub>3</sub>)<sub>3</sub>.

In the solution make-up, Al is dissolved separately in about 1/4 the final volume and the U spike added to this solution. This solution is slightly acid--pH 1 or so--so the U spike is easily soluble. Other compounds are dissolved in a second solution that is strongly basic; the Al-U solution is poured slowly into the basic solution with rapid stirring. Immediately after mixing, the U is in solution, but precipitates in the next 2-8 hours as Na<sub>2</sub>U<sub>2</sub>O<sub>7</sub>. The uranium precipitate is the yellow form of the mixed sodium uranates, formed by adding dilute uranium solution to strong NaOH. (The addition of a concentrated UNH solution to strong NaOH precipitates an orange solid that has essentially an identical x-ray diffraction pattern with the yellow solid.) In the interest of brevity, the mixed uranates will be referred to as Na<sub>2</sub>U<sub>2</sub>O<sub>7</sub> until the Discussion section.

Samples from the solutions normally included solids--Na<sub>2</sub>U<sub>2</sub>O<sub>7</sub> and salt crystals. The solutions were filtered before analysis, originally with a fine-frit Buchner funnel, and later, by Millipore syringe filters. Use of the Buchner funnels has been abandoned because filtration of 1 ml solution by this method is slow--one hour to filter 1 ml is typical. A solution can be filtered with syringe filters, either the 0.45μ or the 0.20μ, in a few minutes.

##### Analytical Methods

Uranium was analyzed by laser-activated fluorescence ("Chemchek") and x-ray fluorescence. Chemchek analyses encountered interferences among the species in the solution that were largely eliminated by fuming the samples in 85% H<sub>3</sub>PO<sub>4</sub>, then diluting before submission to Analytical Development Section. The standard procedure for the fuming involved adding 1 ml sample to 1

until no more bubbles escaped, and then diluting to 10 ml with H<sub>2</sub>O. This procedure eliminates NO<sub>2</sub><sup>-</sup>, Cl<sup>-</sup>, and CO<sub>3</sub><sup>2-</sup>, all potential quenching agents to the U emission fluorescence.

Analysis for U by x-ray fluorescence (xrf) was less sensitive, but did not require a solution adjustment. Normally, xrf gave good results in the range 10 ppm and above, and Chemchek could analyze solutions as low as 50 ppb.

Al was measured by inductively coupled plasma emission spectroscopy (icp), and NO<sub>3</sub><sup>-</sup>, NO<sub>2</sub><sup>-</sup>, Cl<sup>-</sup>, SO<sub>4</sub><sup>2-</sup>, C<sub>2</sub>O<sub>4</sub><sup>2-</sup>, and PO<sub>4</sub><sup>3-</sup> by ion chromatography. Hydroxide and carbonate values were obtained from titration with standard HCl. This titration is influenced by AlO<sub>2</sub><sup>-</sup> and CO<sub>3</sub><sup>2-</sup>, so the following procedure was adapted from E.W. Baumann.<sup>2</sup> An aliquot was mixed with excess 1M BaCl<sub>2</sub> solution to precipitate BaCO<sub>3</sub>, then titrated with HCl to pH 9-10 (thymolphthalein indicator). Bromocresol purple indicator was now added and the titration continued to pH 6-7. A second aliquot (no BaCl<sub>2</sub> addition) was titrated to pH 6-7. The titration to pH 9 determines OH<sup>-</sup>, the titration from pH 9-10 to pH 6-7 determines AlO<sub>2</sub><sup>-</sup>, and the difference between the titres for the two aliquots determines CO<sub>3</sub><sup>2-</sup>.

The acid titration to pH 9-10 was tested against titration by phenolphthalein and found to be about 5% low. The measurement of Al by titration was compared with 14 Al determinations by icp and found to average +19%. The carbonate determination depends on the level of carbonate--with relatively high carbonate--0.3M and up, results appeared fairly good. Low values of carbonate obviously are subject to considerable error, as the determination involves the difference between two large numbers. However, these approximate values are sufficient to determine the major changes in solution during evaporation or dissolution of solids. The results presented here were not corrected for the analytical bias in the determination.

Particle size and x-ray diffraction determinations were determined by ADS in the normal manner.

### Experimental Approach

Experimental work was aimed at three areas pertinent to the Tank 41 problem: determination of U solubility as waste and wash solutions were evaporated, determination of U solubility as salt cakes were dissolved, and attempts to increase U solubility during salt dissolution by adding carbonate to the dissolving solution.

### EVAPORATOR SOLUBILITY EXPERIMENTS

These experiments involved evaporation of the synthetic waste solution, sampling the solution as the volume decreased. During evaporation, the composition of the solution changes as different salts are precipitated and water is evaporated. The changes in solution are shown graphically for major species--NaOH, NaAlO<sub>2</sub>, NaNO<sub>2</sub>, NaNO<sub>3</sub>--(Figure 1) and minor species--NaCl, Na<sub>2</sub>SO<sub>4</sub>, and Na<sub>2</sub>CO<sub>3</sub>-- (Figure 2). Sulfate and carbonate were precipitated in part as the mineral burkite, Na<sub>2</sub>CO<sub>3</sub>•2Na<sub>2</sub>SO<sub>4</sub>. Na<sub>3</sub>PO<sub>4</sub> and Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub> precipitated early in the evaporation, and F<sup>-</sup> could not be determined because of nitrate interference with the ic method.

The solubility of U as the solution was evaporated is shown in Figure 3, where the initial solubility of 50-70 ppm decreased to 7.6 ppm at 9.4M NaOH. (The results shown in Figure 3 are the combined values from two separate evaporation runs.)



The solid phase that precipitated during evaporation included sodium salts of most of the anions with trace amount of U. The U in the solid phase was identified by x-ray diffraction as  $\text{Na}_2\text{U}_2\text{O}_7$ . The solids in the slurry are generally small crystals that suspend easily in the liquid; a settling rate to obtain a clear solution above the slurry layer was measured as 2 mm/hr. A particle size measurement on a sample precipitated from dilute solution found a rather symmetric distribution between 2 and  $18\mu$ , centered at  $5\mu$ . Particle sizes from 0.8 to  $105\mu$  were found for a sample precipitated from a concentrated solution. The distribution was "double-peaked" with maxima at 3 and  $37\mu$ . The settling rate of precipitated  $\text{Na}_2\text{U}_2\text{O}_7$  will be reported separately.<sup>3</sup>

#### SLUDGE WASH EVAPORATIONS

Waste tank sludge is washed with 1M NaOH to remove soluble salts, Al,  $\text{SO}_4^{2-}$ , etc. but since the separation of supernate from sludge is not complete, the wash solution contains some waste supernate as well. The first wash will contain a relatively high supernate-to-wash mixture; successive washes will have progressively less supernate. These solutions are later evaporated and concentrated to salt-supernate for minimum volume storage. Again, there is concern over the behavior of U during this processing. The experimental simulation of this process was to evaporate 1M NaOH solutions containing 1 vol% waste supernate (Table I), 17 vol% waste supernate, and 30 vol% waste supernate.

##### One Percent Wash

The U spike was added to 300 ml of  $\text{H}_2\text{O}$ , 4 ml of waste supernate added and mixed, then 100 ml 4M NaOH mixed. Uranium precipitated after several hours; the solution was evaporated to about 25 ml, and sampled at intervals during the evaporation. NaOH was the major component of the solution; only trace levels of  $\text{AlO}_2^-$  and  $\text{CO}_3^{2-}$  were present. The U solubility (shown graphically in Figure 4) was found to be relatively high--70-45 ppm at 1-3M NaOH, decreasing to 10 ppm at 12-15M NaOH.

##### Seventeen Percent Wash

The procedure for this experiment was the same as for the 1% wash, except for volume adjustments to make the 17% wash. As before, the major species was NaOH, but  $\text{AlO}_2^-$  and  $\text{CO}_3^{2-}$  both reached concentrations of about 0.25M at the final concentration of 6.4M NaOH. The solution sp.g. at this point was 1.322; further sampling was unsuccessful because the hot samples became solid at room temperature. The U solubility (shown in Figure 5) was about 30 ppm until 6.4M NaOH, where a value of 11 ppm was measured.

##### Thirty Percent Wash

The solution make-up was similar to the two previous experiments, and was intended to be representative of the first wash-supernate mixture. The data are shown in Figure 6; the measured solubility decreases gradually from 96 ppm at 1.4M NaOH to 9 ppm at 6.9M.

### SALT DISSOLVING EXPERIMENTS

A more important factor to the tank 41 problem than U solubility during evaporation is U solubility during salt dissolving. The plan for salt dissolution from tank 41 is to add water, agitate until a pre-determined specific gravity of the solution is reached, transfer the solution to tank 48, and then repeat this process as long as necessary to remove the salt. The time for dissolving and transfer of a batch of re-dissolved salt has been estimated as 10-14 days. The ideal would be for U to dissolve at the same rate as the salt cake, since this would prevent an accumulation of U in the tank residue.

Experimental tests of the solubility of U during dissolution of the salt cake were simulated with the salt cakes produced during evaporation experiments. Typically, 5-10 ml of the dissolving solution to be tested was added to the salt cake, and the mixture stirred for a period of 1 to 7 days, then the supernate decanted, sampled and analyzed. This procedure was repeated until the salt was essentially all dissolved. Excess salt remained after all dissolvings. EDTA and carbonate both complex U(VI) and were added in an attempt to increase U solubility. Results of a typical experiment are shown in Table II.

Table II. Salt Dissolution Results

Solution (5 mL)	Mixing Time, Days	Final Concentration			
		NaOH, M	NaAlO <sub>2</sub> , M	Na <sub>2</sub> CO <sub>3</sub> , M	U, ppm
H <sub>2</sub> O	1	1.65	1.44	0.26	3.8
H <sub>2</sub> O	2	0.55	0.47	1.59	1.6
0.2M Na <sub>2</sub> CO <sub>3</sub>	1	0.32	0.73	0.79	1.5
0.2M Na <sub>2</sub> CO <sub>3</sub>	6	0.16	0.37	0.90	1.8
0.5M NaHCO <sub>3</sub> (10 mL)	5	10 <sup>-5</sup> (pH 9)	0.58	0.85	34
10 <sup>-2</sup> M EDTA	1	3.16	1.40	0.23	3.2
10 <sup>-3</sup> M EDTA	7	2.38	1.39	0.67	2.7

These data show a low solubility for precipitated Na<sub>2</sub>U<sub>2</sub>O<sub>7</sub> for solutions that are not too much different from the solutions measured in evaporation experiments. The only high solubility found was at pH 9, far lower in base strength than corrosion considerations would allow.

Measurements of the solution concentrations during salt dissolving generally show that the last compounds to precipitate during evaporation are the first to redissolve. Representative data are shown in Table III; compare with Figures 1 and 2.

Table III. Concentrations (M/L) in Salt Dissolving Fractions

Fraction (10 mL)	NaOH	Na <sub>2</sub> CO <sub>3</sub>	NaCl	NaNO <sub>2</sub>	NaNO <sub>3</sub>	Na <sub>3</sub> PO <sub>4</sub>	Na <sub>2</sub> SO <sub>4</sub>	NaAlO <sub>2</sub>
1	5.26	0.10	0.20	3.62	2.35	0.0066	0.021	2.27
2	3.92	0.39	0.19	3.05	2.83	0.0066	0.035	2.09
3	1.65	0.26	0.12	2.43	3.43	0.013	0.079	1.28

## CARBONATE COMPLEXING DISSOLUTION

There remains the possibility of aiding the redissolution of  $\text{Na}_2\text{U}_2\text{O}_7$  by adding a complexing agent, such as  $\text{CO}_3^{2-}$ , to the dissolving solution. Uranium (VI) forms a carbonate complex that might increase its solubility. The acceptable choices are limited by the insolubility of most of complexing anions in strong NaOH. Available data show that  $\text{CO}_3^{2-}$ ,  $\text{PO}_4^{3-}$ ,  $\text{C}_2\text{O}_4^{2-}$  and  $\text{SO}_4^{2-}$  are essentially gone from solution at 6M NaOH. The addition of most complexing agents to the dissolving solution also presents a practical problem for the plant; tankage to make up a dissolving solution almost certainly is not available. However,  $\text{CO}_2$  could be sparged into the tank to both bring down the NaOH concentration and increase the  $\text{CO}_3^{2-}$  concentration. Some data that bear on this proposal are shown in Table IV.

Table IV. Bicarbonate Salt Dissolving

10 mL fractions	0.5M $\text{NaHCO}_3$	NaOH*	$\text{NaAlO}_2^*$	$\text{Na}_2\text{CO}_3^*$	sp. g	U, ppm
1		4.48	1.55	0.05		5.2
2		2.21	1.21	0.68	1.38	4.2
3		0.48	0.85	1.10	1.379	4.1
4		0.11	0.46	1.00	1.380	4.7

\* Concentration in M/L.

Note in Table IV that the  $\text{CO}_3^{2-}$  added with the dissolving solution precipitated while  $\text{NaNO}_2$ ,  $\text{NaNO}_3$ , etc. redissolved.

The possible application of  $\text{CO}_3^{2-}$  complexing to dissolving  $\text{Na}_2\text{U}_2\text{O}_7$  in tank 41 would involve adding water, mixing until the sp. g. is about 1.4, then reducing the  $\text{OH}^-$  concentration by sparging with  $\text{CO}_2$  gas. This procedure requires only easily available equipment, but has the disadvantage of requiring rather sensitive control to avoid precipitation of  $\text{Al}(\text{OH})_3 \cdot x\text{H}_2\text{O}$ .

A test of this procedure was successful in reducing the NaOH concentration, but did not increase the solubility of U. Repeated sparging of a solution initially 6.89M NaOH-0.10M  $\text{Na}_2\text{CO}_3$ -9.17 ppm U with  $\text{CO}_2$  resulted in changing the composition to 3.73M NaOH-0.26M  $\text{Na}_2\text{CO}_3$ -7.8 ppm U. This change in U concentration was not deemed significant. An estimated 175 g/L of  $\text{Na}_2\text{CO}_3$  was precipitated by the  $\text{CO}_2$  sparging. The prospect of increasing the volume of dissolving solution for no gain in U solubility suggests that this approach has little value.

### 4.0 Discussion

Comparison of the U solubility in boiled-down solutions (ca. 10-30 ppm) with the solubility found on redissolving crystallized salts (2-5 ppm) demonstrates that the system is not at equilibrium. In principle, there is a dynamic equilibrium between U in solution and U in the solid phase. Thus, if the system was at equilibrium, the solubilities measured in the solution and those measured during redissolution should be the same; moreover, at equilibrium, only one solid phase will be present. Equilibrium presumably would be established eventually, but not on the time scale of these experiments.

A possible explanation for the non-reversibility of this system lies in the nature of the aqueous species and the solid phase. Previous investigations<sup>4</sup> of  $\text{UO}_2^{2+}$  in basic perchlorate solutions were interpreted as showing a series of basic polymeric species, as  $\text{U}_2\text{O}_5^{2-}$ ,  $\text{U}_3\text{O}_8^{2+}$  and  $\text{U}_3\text{O}_8(\text{OH})^+$ . (These may also be written as  $\text{UO}_3 \cdot \text{UO}_2^{2+}$ ,  $2\text{UO}_3 \cdot \text{UO}_2^{2+}$ , etc.) Several solid phases have been deduced from x-ray and solution studies for the solid normally referred to as "Na<sub>2</sub>U<sub>2</sub>O<sub>7</sub>". Wamser, et al.<sup>5</sup> identified two solid phases, Na<sub>2</sub>O·7UO<sub>3</sub> and 3Na<sub>2</sub>O·7UO<sub>3</sub> at pH 6 and 10, respectively. Ricci and Loprest<sup>6</sup> deduced the compound Na<sub>2</sub>O·6UO<sub>3</sub> and solid solutions of Na<sub>2</sub>O in UO<sub>3</sub> with Na/U ratios from 8:11 to 1:3 and 1:12 to 1:18. The kinetic path between the polymeric solution species and the polymerized solid may depend upon the transfer of U via one or more species of very low concentration. The attainment of equilibrium in this situation might then be a very lengthy process. Thus, measurements of the solubility of U in strongly basic solutions can depend to a major extent on the method used for solution make-up.

When all the data from this study are plotted against NaOH concentration on one graph (Figure 7), the average demonstrates a decreasing solubility with decreasing NaOH concentration, regardless of the concentration of other salts present. The graph shows considerable scatter at lower NaOH concentrations, but the bulk of the data is consistent. Some of the data scatter might be caused by solid particles small enough to pass through the 0.2 $\mu$  filter, but most is probably due to the non-equilibrium system.

As applied to plant waste evaporation, this work predicts that U will precipitate as solutions are evaporated. Whether this U precipitate will accumulate in the evaporator, as at West Valley<sup>7</sup>, or be carried through as a slurry depends on physical factors.

The data also indicate that the major component affecting the solubility is NaOH. The solubility of U in the 1% waste-supernate is essentially the same as the solubility in 100% supernate, demonstrating that the complexing anions have little effect on the solubility. As the concentration of NaOH increases, most of the potential anionic complexants--CO<sub>3</sub><sup>2-</sup>, C<sub>2</sub>O<sub>4</sub><sup>2-</sup>, PO<sub>4</sub><sup>3-</sup>--crystallize as sodium salts. Chloride is only potential complexant that can be positively identified as increasing as the solution is concentrated. (It is suspected that fluoride precipitates as Na<sub>3</sub>AlF<sub>6</sub>, since this compound was found in one x-ray pattern.) Sludge wash solutions can be expected to precipitate U during evaporation, just like the supernate.

## 5.0 References

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7. Acid washes of the West Valley Waste Evaporator recovered 10 kg U, 255 g Pu, Garrett Smith (WVNS Co.) to T. Hsu, WSRC Fax, August 1991

Figure 1. Synthetic Waste Supernate  
 $\text{AlO}_2^-$ ,  $\text{NO}_2^-$ ,  $\text{NO}_3^-$  Conc during Evaporation

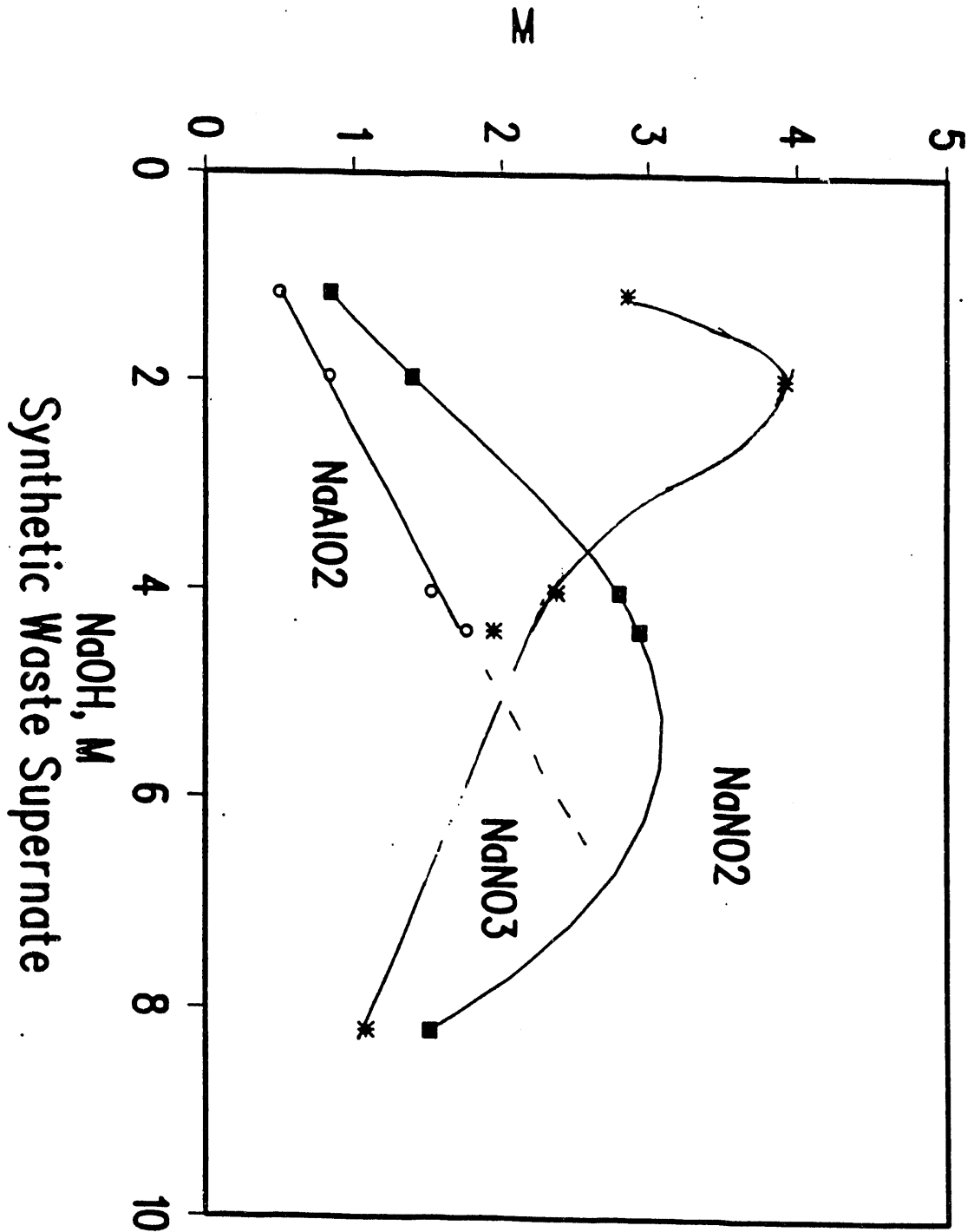


Figure 2. Synthetic Waste Supernate  
Cl<sup>-</sup>, SO<sub>4</sub><sup>2-</sup>, CO<sub>3</sub><sup>2-</sup> Conc in Evaporation

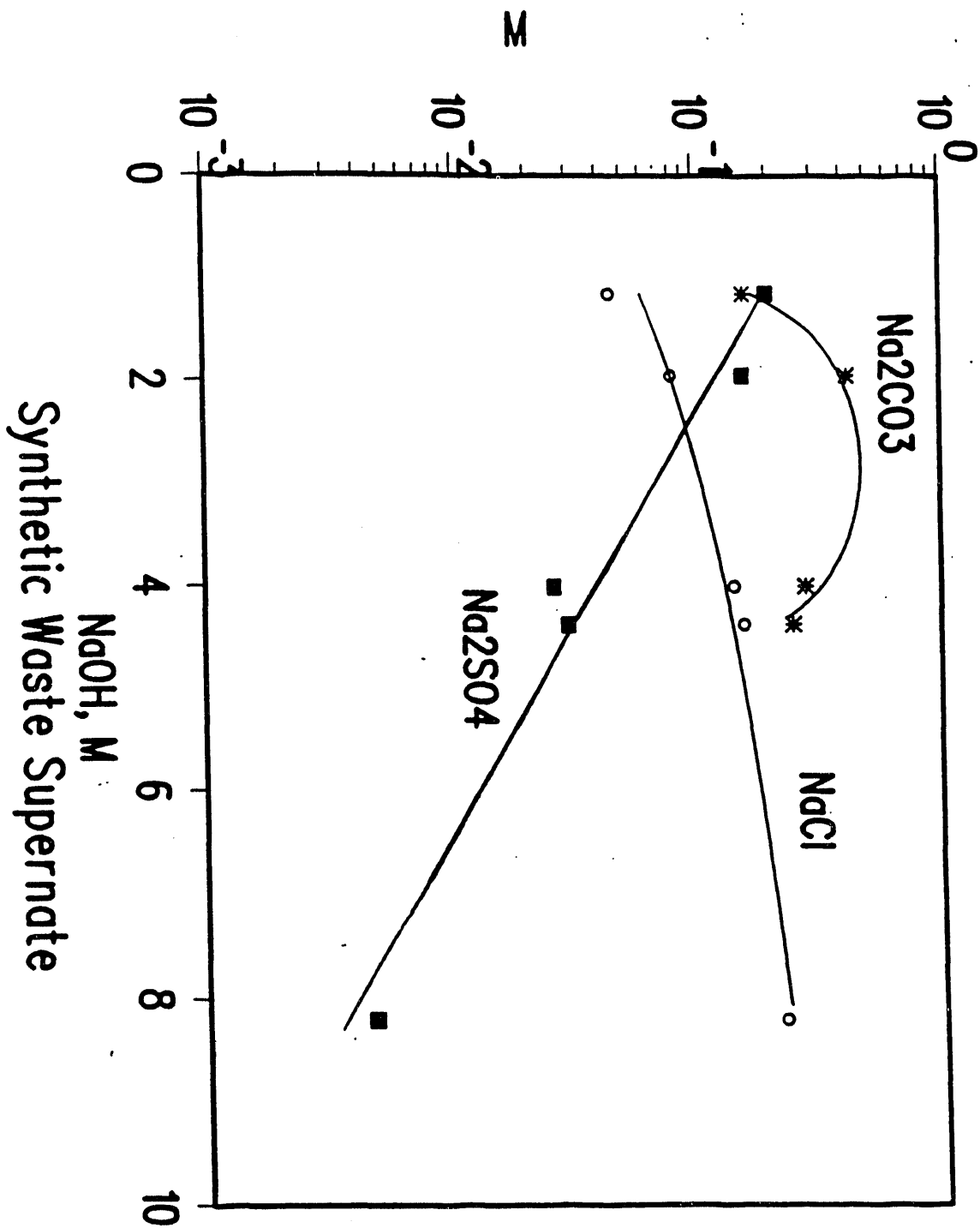


Figure 3.  
Na<sub>2</sub>U<sub>2</sub>O<sub>7</sub> Solubility in Tank 41 Synthetic

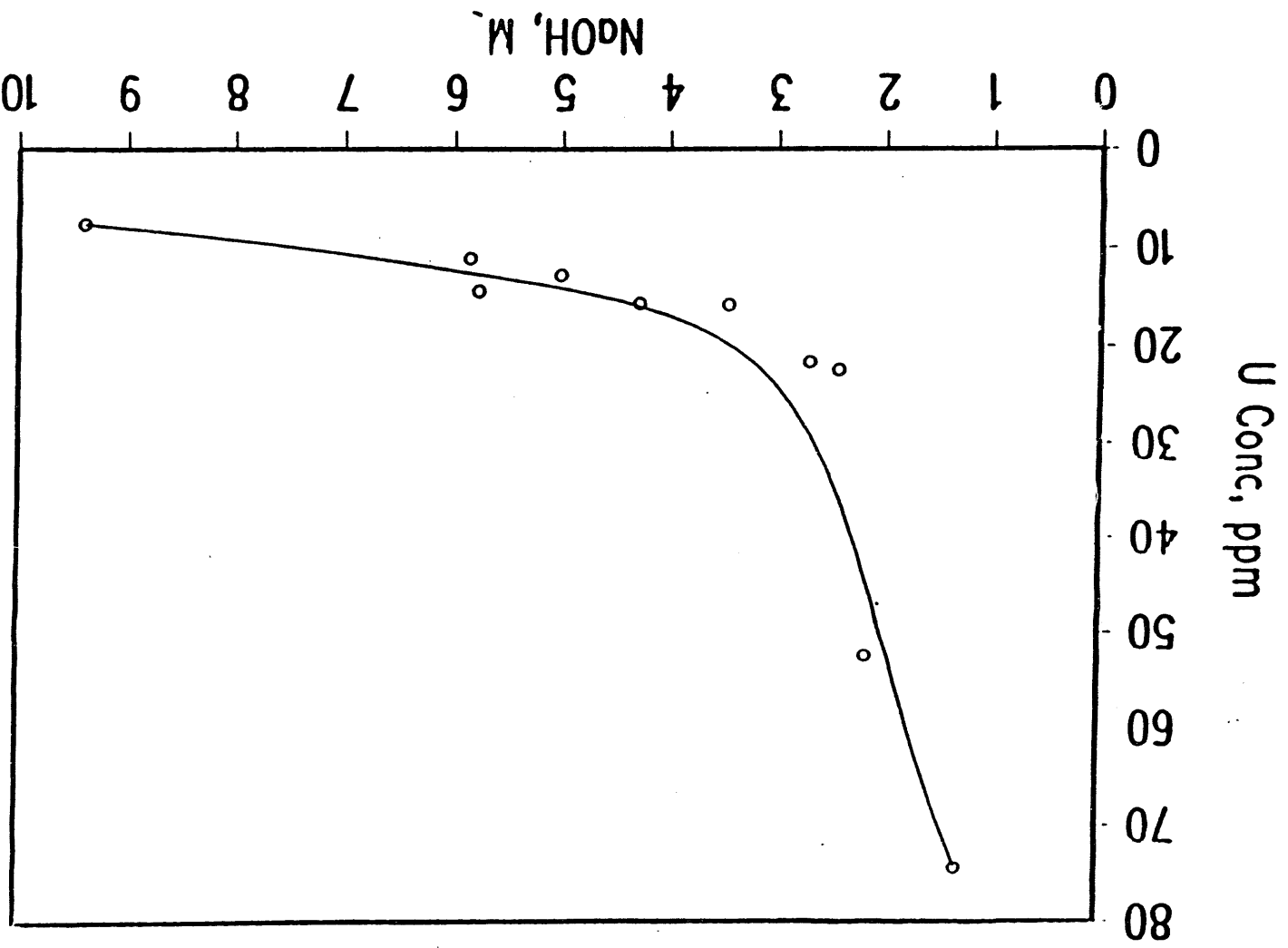




Figure 4.  
U Solubility in NaOH Solution

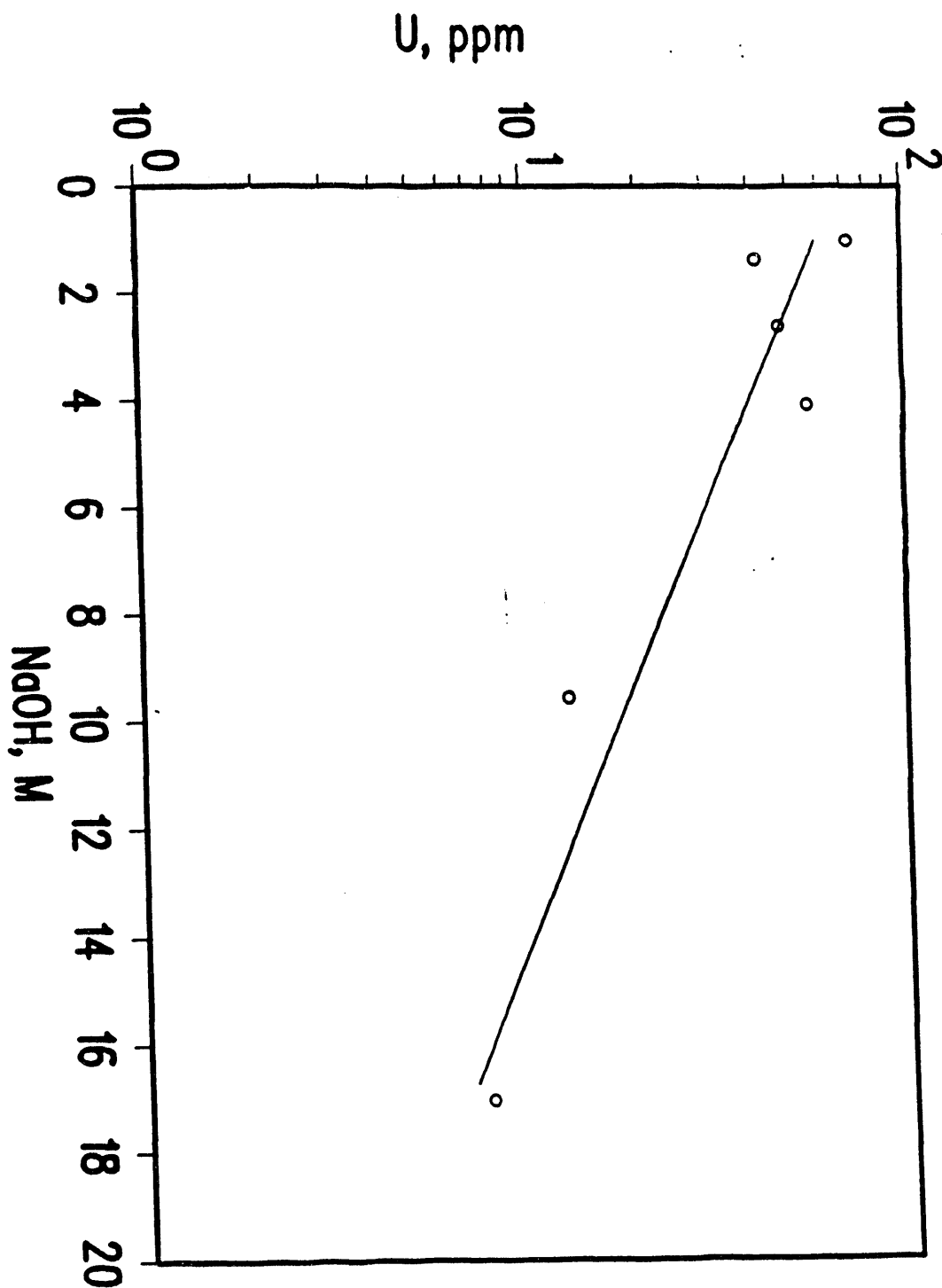


Figure 5.  
U Solubility during 17% Supernate Evaporation

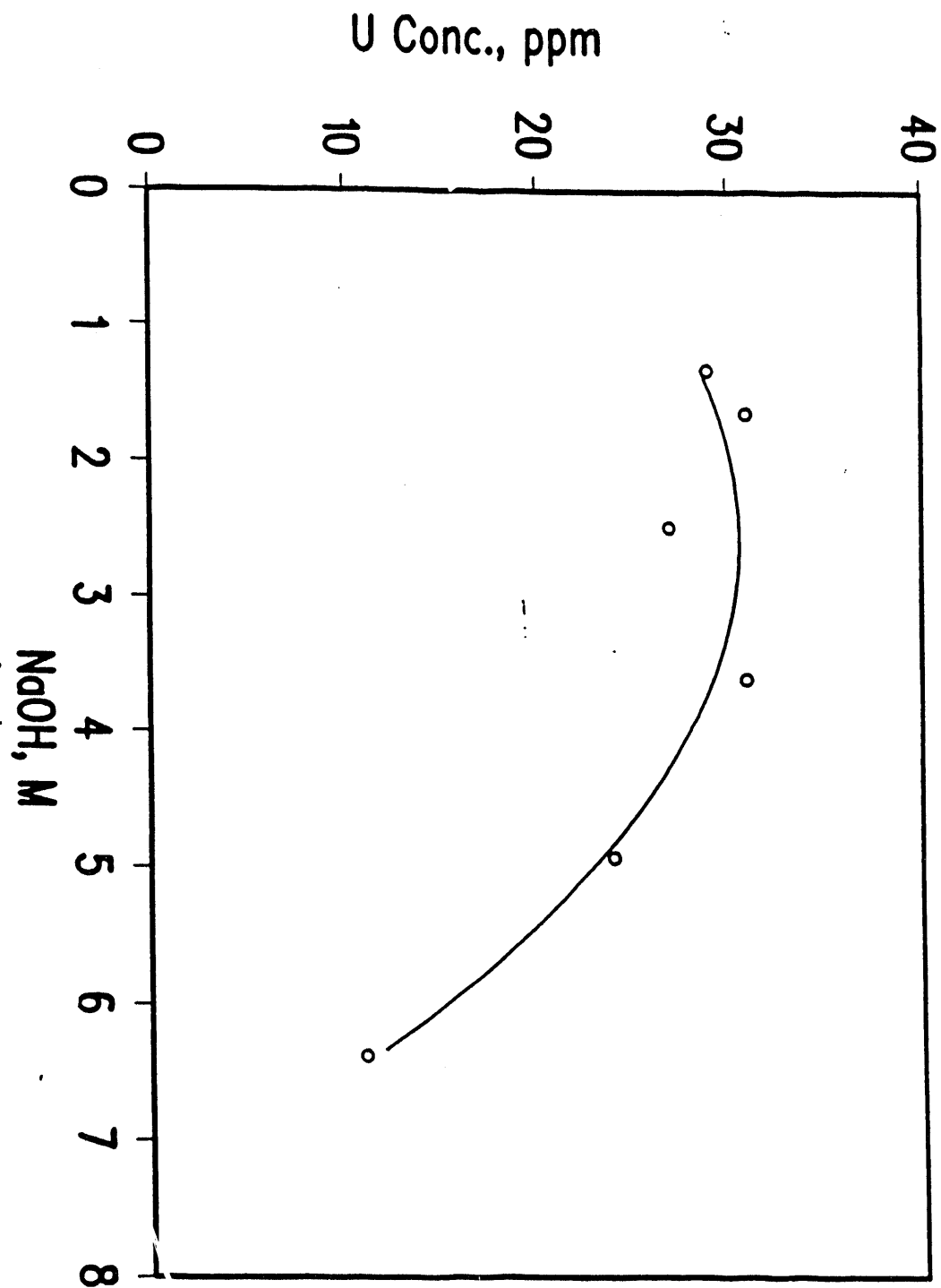


Figure 6. NaOH, M  
U Solubility in 30% Supernate Evaporation

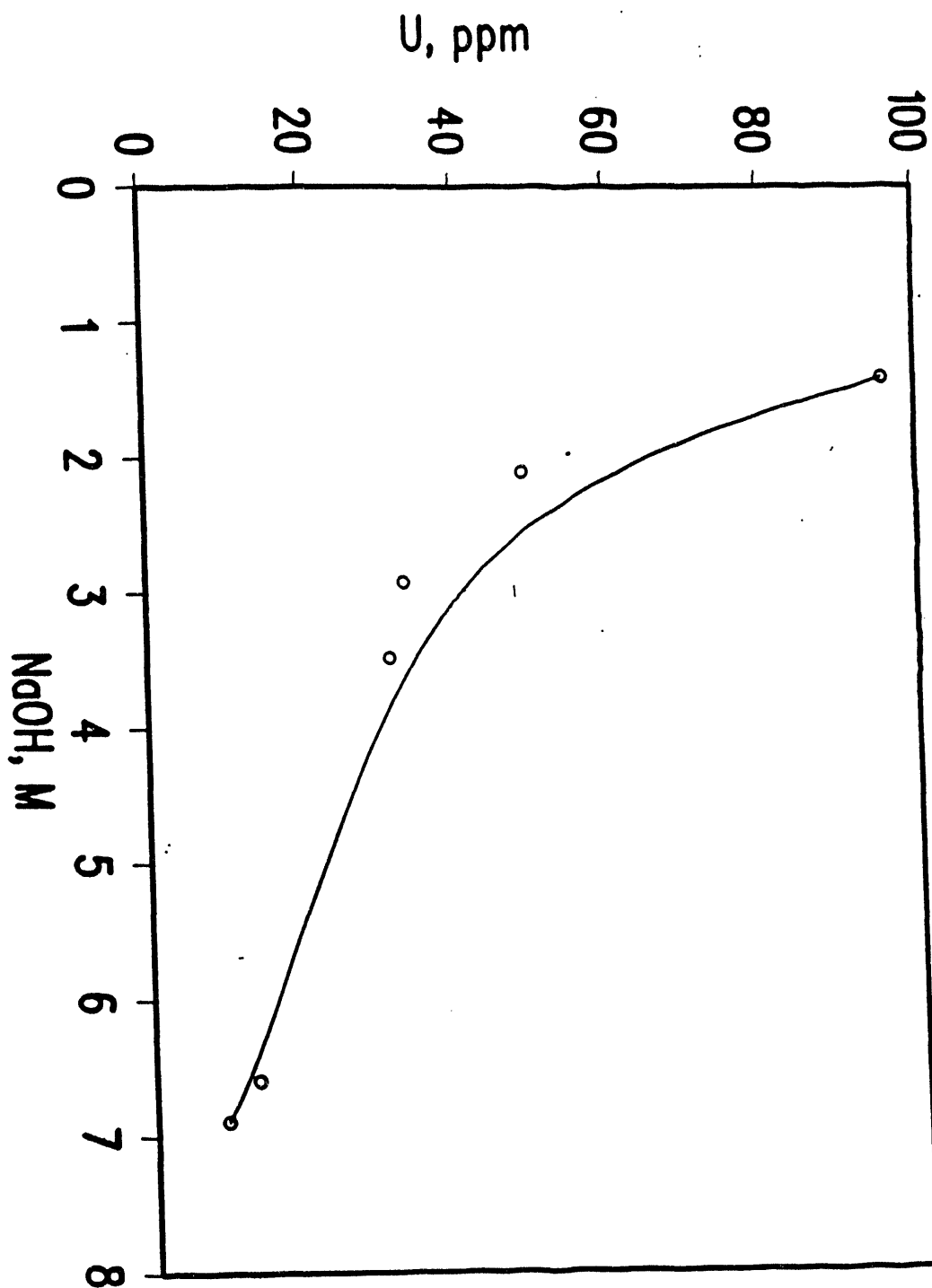
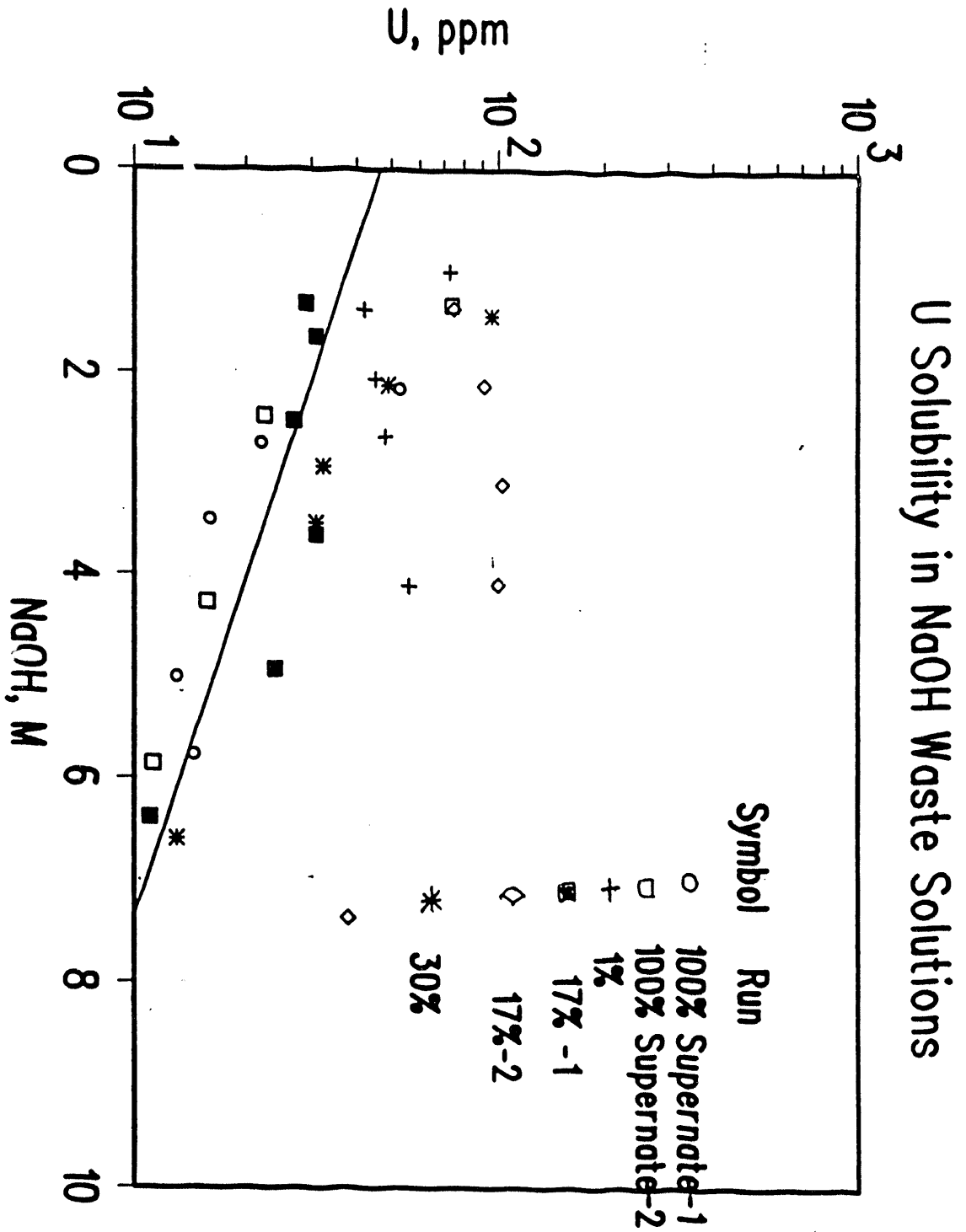


Figure 7.  
 U Solubility in NaOH Waste Solutions



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F. R. Graham, 773-A  
M. S. Hay, 773-A  
D. T. Hobbs, 773-A  
E. W. Holtzscheiter, 773-A  
D. G. Karraker, 773-A  
L. M. Papouchado, 773-A  
C. P. Reeve, 773-42A  
W. L. Tamosaitis, 773-A  
M. C. Thompson, 773-A  
R. C. Tuckfield, 773-42A  
D. D. Walker, 773-A  
E. L. Wilhite, 773-43A  
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M. C. Chandler, 703-H  
J. S. Clemmons, 703-H  
P. D. d'Entremont, 703-H  
G. K. Georgeton, 703-H  
B. L. Lewis, 703-H  
J. E. Marra, 703-H  
T. M. Monahon, 703-H  
R. A. Scaggs, 703-H

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