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STUDIES IN THE CARBONATE-URANIUM SYSTEM

Part II. The Solubility of Sodium Uranyl Tricarbonate in Solutions of Certain Sodium Salts

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Abstract

Determinations have been made of the solubility of sodium uranyl tricarbonate in water and in solutions of sodium carbonate, sodium sulfate, sodium chloride and sodium perchlorate. The solubility is presented in its relation to the molarity of the co-solute and the solubility product is presented in its relation to the total ionic strength. The data are also considered as to their bearing on plant processes such as those employed in the carnotite mill at Monticello, Utah.

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THE SOLUBILITY OF SODIUM URANYL TRICARBONATE IN SOLUTIONS OF CERTAIN SODIUM SALTS

INTRODUCTION AND SUMMARY

The solubility of sodium uranyl tricarbonate in solutions of sodium carbonate and sodium bicarbonate has been determined by workers at Princeton University.[1] Some additional measurements have been made in these systems and the studies have been extended to include solutions of sodium chloride, sodium sulfate, sodium perchlorate and mixtures. Excellent correlation with the Princeton results has been obtained.

In examining the composited data it may be noted that the solubility of the tricarbonate is nearly the same in solutions of each co-solute at the same molarity -- regardless of whether the co-solute is a di- or mono-sodium salt. Obviously this is not the relationship expected from application of the mass action law, without correction for activity coefficients. At least a partial explanation of the data is given, however, from consideration of the solubility product, as determined for each experiment, in conjunction with the ionic strength. A plot of the logarithm of the solubility product versus the square root of the ionic strength is expressed approximately by a straight line according to the following equation:

$$\log S.P. = 1.25\sqrt{\mu} - 2.2 \qquad \text{temp.} = 26\text{-}30^{\circ}C$$

The uniformity and generality of the variation of solubility with molarity of the co-solutes suggest a use for the data in predicting the

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limiting concentrations of uranium that may be attained in plants using a sodium carbonate leach. Reconsideration of previous data on the build-up of uranium in the salt roast, sodium carbonate leach process at Monticello, Utah shows a reasonable agreement between the measured and predicted results. It is believed that the solubility of sodium uranyl tricarbonate is a factor to be considered in this plant.

EXPERIMENTAL METHODS

Preparation of Sodium Uranyl Tricarbonate

Sodium uranyl tricarbonate ($2Na_2CO_3 \cdot UO_2CO_3$) has been prepared according to the general procedure first suggested by Ebelman[2] and later used by workers at Princeton, New Jersey.[1]

Sodium diuranate was prepared by precipitating from uranyl nitrate solution with sodium hydroxide. This precipitate was washed by repeated centrifuging and decantation to remove most of the sodium hydroxide, and was then dissolved in a solution containing a slight excess of sodium bicarbonate. The solid tricarbonate was obtained by evaporating the solution to a low volume while passing carbon dioxide to prevent decomposition. A recrystallization was carried out for further purification.

The bright yellow sodium uranyl tricarbonate prepared in this manner is quite pure. Chemical analyses for sodium, uranium and carbonate give values very close to the calculated stoichiometric quantities (Table 1). The compound is stable at temperatures as high as 200°C.[3]

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Table 1

Analyses of Sodium Uranyl Tricarbonate*

Property	Theory For $2Na_2CO_3 \cdot UO_2CO_3$	Batch V	Batch XVI	Batch VI
CO_3(%)	33.22	33.4	32.9	32.3
Na (%)	16.96	16.5	16.8	16.3
U (%)	43.92	43.0	43.4	42.5
CO_3/U (Mole Ratio)	3.00	3.07	3.00	3.03
Na/U (Mole Ratio)	4.00	3.96	4.00	3.98

* Several other batches of this compound have been prepared and the analyses presented are typical for these preparations. The tendency for the analyses to be somewhat lower than theoretical is accounted for by incomplete drying of the samples.

Measurement of Solubility

Measured quantities of solid sodium uranyl tricarbonate and the solution under consideration were placed in either pyrex erlenmeyer flasks or plastic (polyfluorethylene) bottles and all vessels were sealed with a rubber stopper. To speed operations a constant temperature bath was not used. All samples were placed in a constant temperature room and vigorously agitated with an International bottle shaker. Room temperature was 26 ± 2°C.

In a number of the first experiments, the times necessary for the systems to attain equilibrium were measured by sampling periodically until constant uranium analyses were obtained. It was found that virtual equilibrium had been reached in all cases after two weeks. Further tests were then run without periodic sampling, but the time was extended to four weeks

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as an added safeguard. The consistency of the data and the agreement between like experiments support the conclusion that the systems were at, or essentially at, equilibrium.

In terminating each test the solid and solution were separated by centrifuging, using a tared centrifuge tube. The solutions were decanted, filtered through fine sintered glass, and analyzed for sodium, uranium, carbonate and whatever anion was contributed by the co-solute. The density and pH were also usually measured. The wet residues were weighed and the total quantities from each test were submitted for the same analytical treatment as given the solutions. From knowledge of the total weight of residue and the total quantities of solid constituents present, a correction was made for adhering solution and the composition of the dry solid was thus obtained.

In each case the solid had remained unaltered from its original form. To avoid repetition of identical data for the numerous experiments throughout the report, representative analyses of solids from typical experiments are presented in Table 2.

Table 2

Analyses of Solid Phases From Typical Equilibrium Experiments

		Analyses of Solids After Correcting For Adhering Solution				
Exp. No.	Exp. Type	U (mg)	Na (mg)	$CO_3^{=}$ (mg)	CO_3/U	Na/U
183	1.35 M $NaClO_4$	935	351	710	3.00	3.9
184	1.8 M $NaClO_4$	1057	404	815	3.05	3.96
194	2.0 M NaCl	1100	423	852	3.07	4.0
420	1.5 M Na_2SO_4	327	131	257	3.1	4.15
421	2.0 M Na_2SO_4	480	200	356	2.9	4.3

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Analyses of Experimental Samples

Most of the analyses were made by the ORNL Analytical Chemistry Division, Y-12. High level uranium samples were handled by reducing the uranium and titrating potentiometrically with ferric sulfate solution. On low level samples the ascorbic acid, colorimetric procedure was used. Sodium was determined with the flame photometer and the usual adsorption, gravimetric method was used for carbonate.

SOLUBILITY DATA

Effect of Sodium Carbonate and Sodium Bicarbonate (Princeton, 30°C)

The solubility of sodium uranyl tricarbonate in solutions of sodium carbonate and sodium bicarbonate has been measured by workers at Princeton University. For convenient reference these data are presented in Figure 1. Although the Princeton measurements were made at 30°C and measurements at this laboratory have been made at 26°C, the results are not measurably affected so as to disturb comparison.

Effect of Sodium Chloride and Sodium Sulfate (26°C)

Data showing the influence of sodium chloride and sodium sulfate on the solubility of sodium uranyl tricarbonate are tabulated in Tables 3 and 4. Graphical presentations of the solubility versus molarity of the co-solutes are given in Figure 2. (Compare Figures 1 and 2). The pH values listed in these and subsequent tables are not necessarily representative of solutions of pure sodium uranyl tricarbonate since some of the preparations were contaminated by small quantities of excess sodium carbonate.

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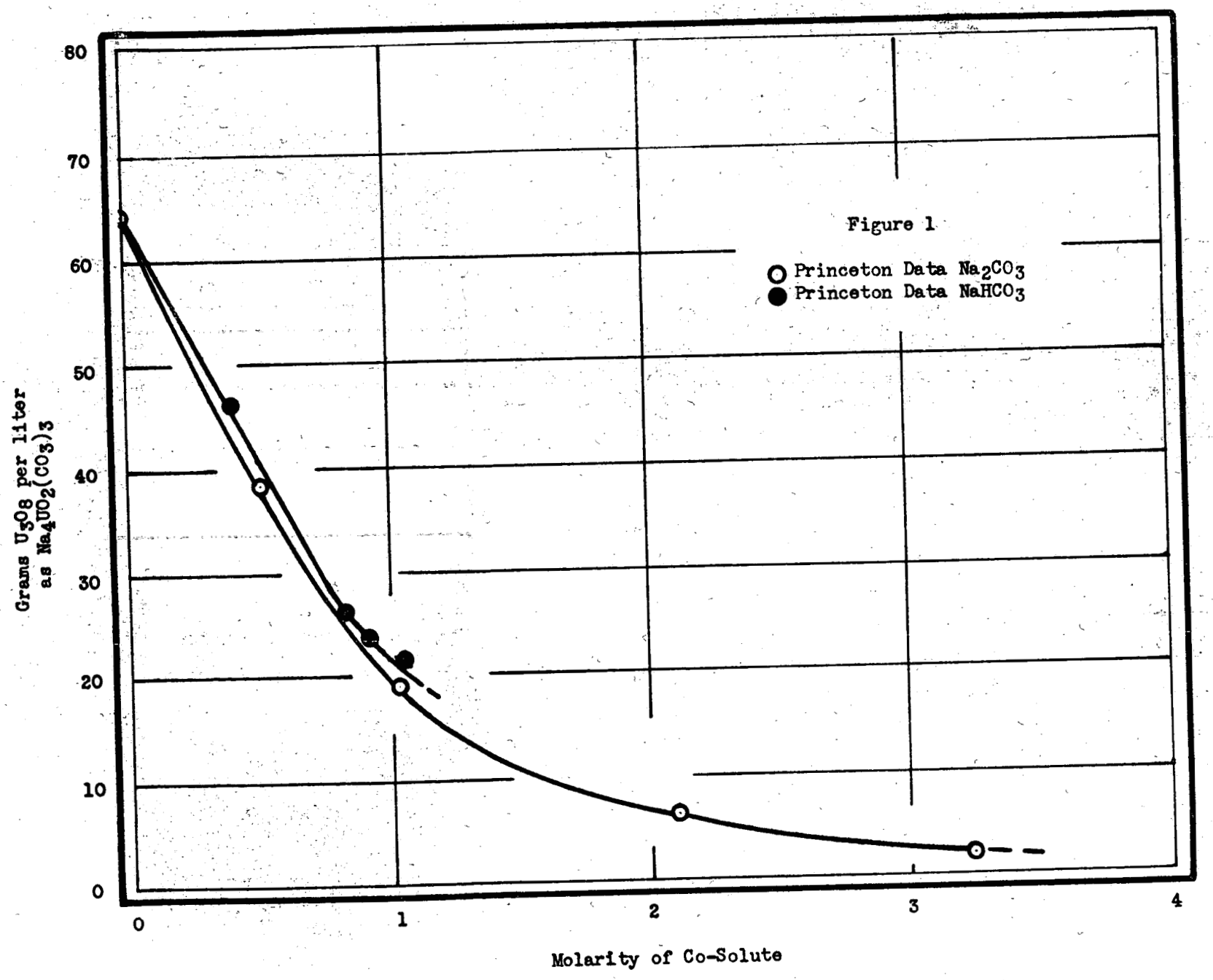
Figure 1
Princeton Data Na_2CO_3
Princeton Data $NaHCO_3$
Grams U_3O_8 per liter as $Na_4UO_2(CO_3)_3$
Molarity of Co-Solute
0
10
20
30
40
50
60
70
80
0
1
2
3
4

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Table 3

Solubility of Sodium Uranyl Tricarbonate in Solutions of Sodium Chloride

Exp. A	NaCl M	Solubility as gms U_3O_8/l	Solution Density	Solution pH
145	0.0(water)	77.4	1.128	9.25
192	1.0	21.1	1.070	9.02
193	1.5	9.45	1.071	8.4
194	2.0	5.55	1.085	8.58
195	2.5	3.26	1.100	8.2
196	3.0	1.69	1.115	8.82
197	3.44	0.885	1.130	8.40
198	4.0	0.720	1.150	8.0

Table 4

Solubility of Sodium Uranyl Tricarbonate in Solutions of Sodium Sulfate

Exp. A	Na_2SO_4 M	Solubility as gms U_3O_8/l	Solution Density	Solution pH
146	0.0(water)	80.0	1.1280	9.8
419	0.30	47.2	1.110	8.8
420	1.5	12.6	1.197	8.65
421	2.0	9.9	1.244	8.65
422	3.0	3.2	1.334	8.75

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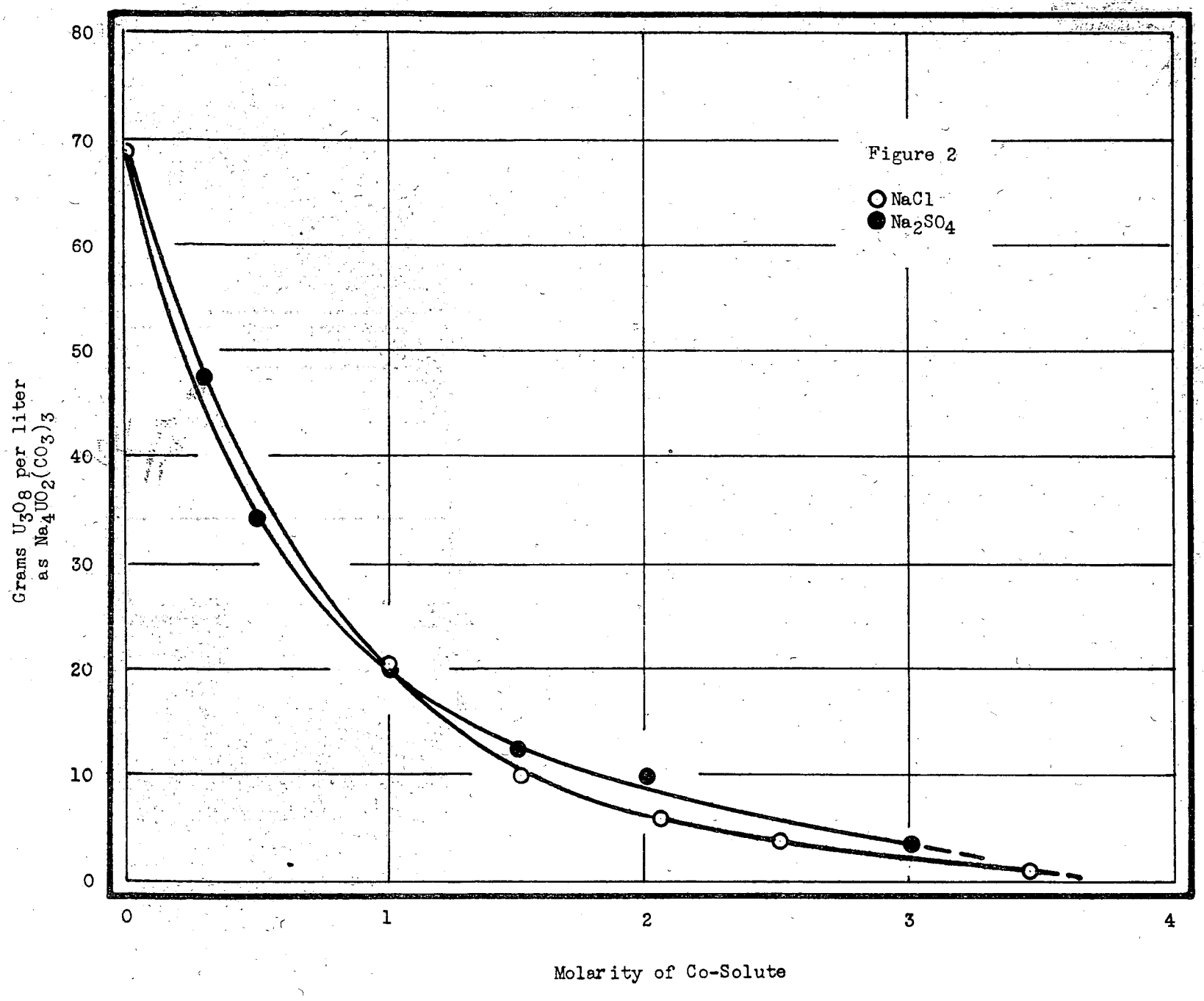
Figure 2
NaCl
Na_2SO_4
Grams U_3O_8 per liter as $Na_4UO_2(CO_3)_3$
Molarity of Co-Solute
0
10
20
30
40
50
60
70
80
0
1
2
3
4

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Effect of Sodium Perchlorate and Mixtures of Sodium Chloride and Sodium Carbonate (26°C)

Experimental results showing the influence of sodium perchlorate and sodium chloride, sodium carbonate mixtures are given in Tables 5 and 6. Consistent with previous data these results are depicted graphically in Figure 3. (Compare Figures 1, 2 and 3.)

Effect of Sodium Carbonate, Sodium Bicarbonate and Sodium Sulfate at Higher Temperatures (Princeton, 60 and 90°C)

High temperature tests have not been made as yet at this laboratory. Some work from Princeton University is available, however, and since this information is important to subsequent discussions, the tests are represented in Figure 4 in a manner comparable to the foregoing data.

Solubility versus Molarity of the Co-solute

Close observation of Figures 1, 2 and 3 reveals a remarkable consistency in the variation of sodium uranyl tricarbonate solubility with changes in molar concentrations of any of the added sodium salts. The same effect is noted in the Princeton experiments at higher temperatures (Figure 4), although the information is somewhat limited. To emphasize this point, the results previously presented in Figures 1-3 have been combined in Figure 5 where the solubility has been plotted against the molar concentration of the co-solute, or mixtures thereof, regardless of their identification. All points can be represented rather closely by a single curve. This is not the relationship expected from application of the mass action law, without correction for activity coefficients, since different effects would be required for mono and di-sodium salts.

Table 5

Solubility of Sodium Uranyl Tricarbonate

in Solutions of Sodium Perchlorate

Exp. A	$NaClO_4$ M	Solubility as gms U_3O_8/l	Solution Density	Solution pH
181	0.46	40.7	1.105	8.8
182	0.90	18.6	1.099	8.7
183	1.33	9.6	1.117	8.3
184	1.80	5.85	1.149	8.1
185	2.25	3.13	1.178	7.5
186	2.70	1.74	1.208	7.2
187	3.11	1.10	1.239	-
188	3.40	0.68	1.264	-

Table 6

Solubility of Sodium Uranyl Tricarbonate in Mixed Solutions of

Sodium Carbonate and Sodium Chloride*

Exp. A	Na_2CO_3 M	NaCl M	Solubility as gms U_3O_8/l
V-782	0.28	0.0	49.1
V-783	0.57	0.0	31.5
V-813	0.28	0.32	30.4
V-814	0.28	0.67	19.0
V-815	0.28	1.35	8.4
V-816	0.28	2.6	2.4

* This table was compiled from previous data in report Y-571 (see References).

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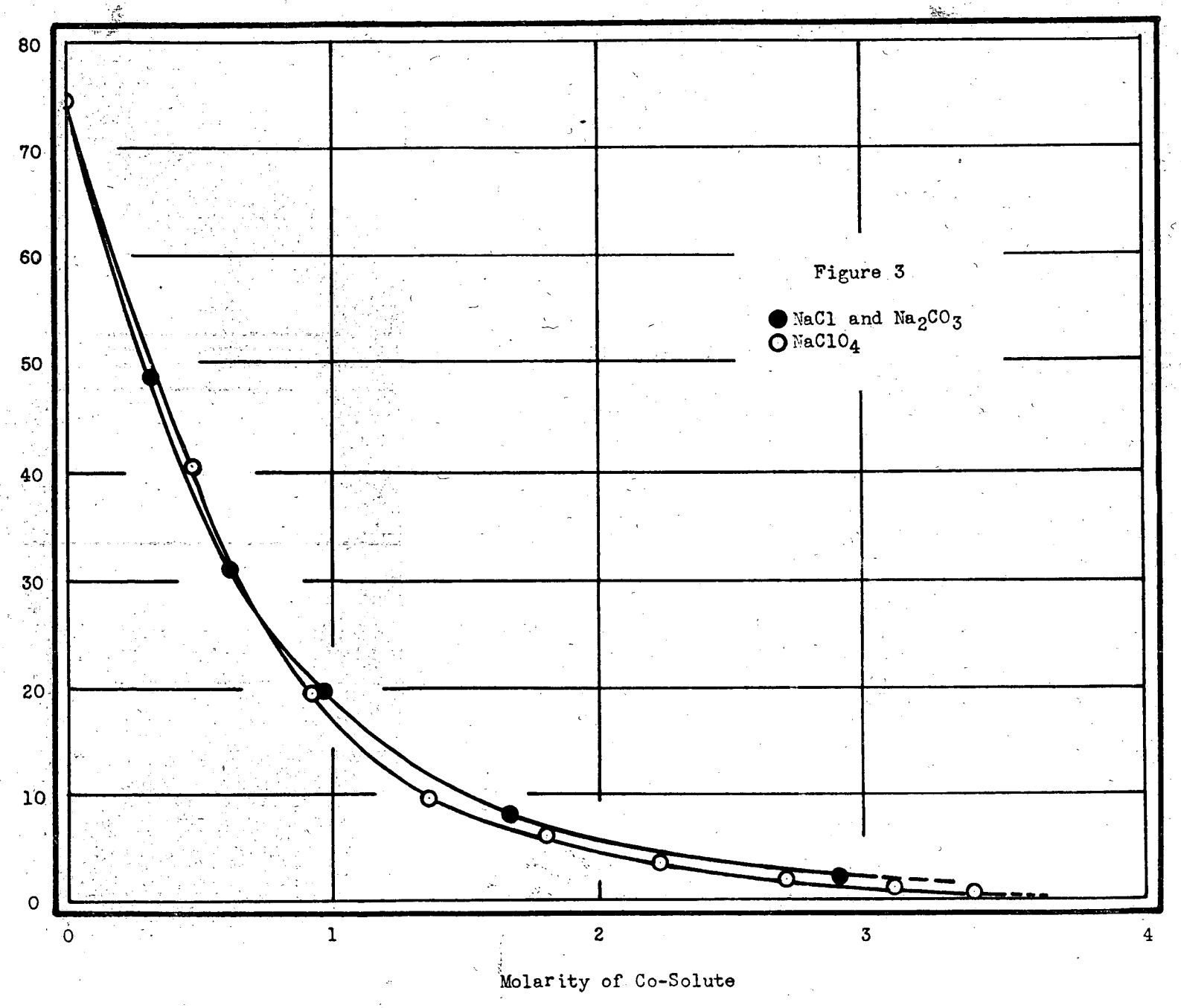
Figure 3
NaCl and Na_2CO_3
$NaClO_4$
Grams U_3O_8 per liter as $Na_4UO_2(CO_3)_3$
Molarity of Co-Solute
0
10
20
30
40
50
60
70
80
0
1
2
3
4

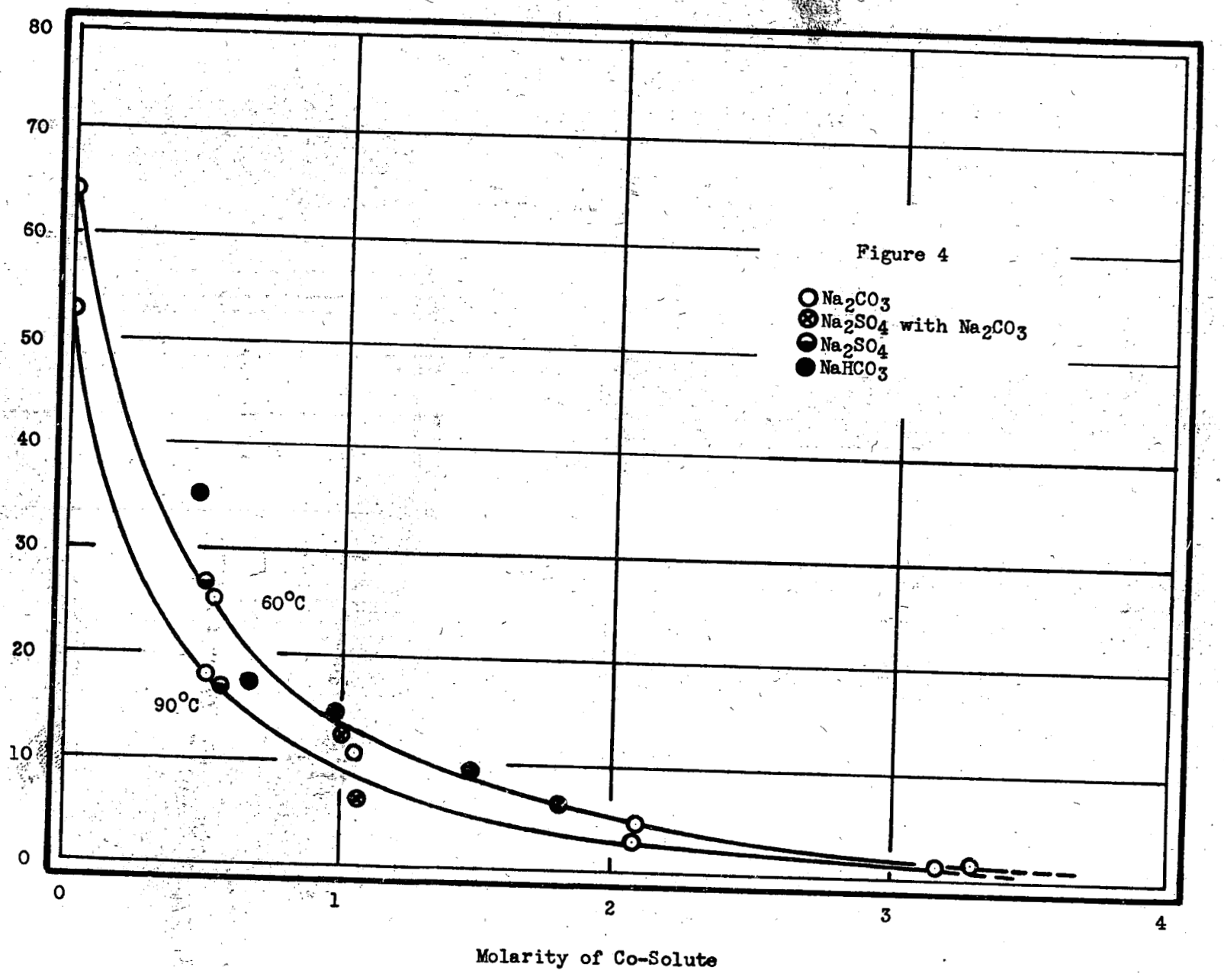
Figure 4
Na_2CO_3
Na_2SO_4 with Na_2CO_3
Na_2SO_4
$NaHCO_3$
60°C
90°C
Grams U_3O_8 per liter as $Na_4UO_2(CO_3)_3$
Molarity of Co-Solute
0
10
20
30
40
50
60
70
80
0
1
2
3
4

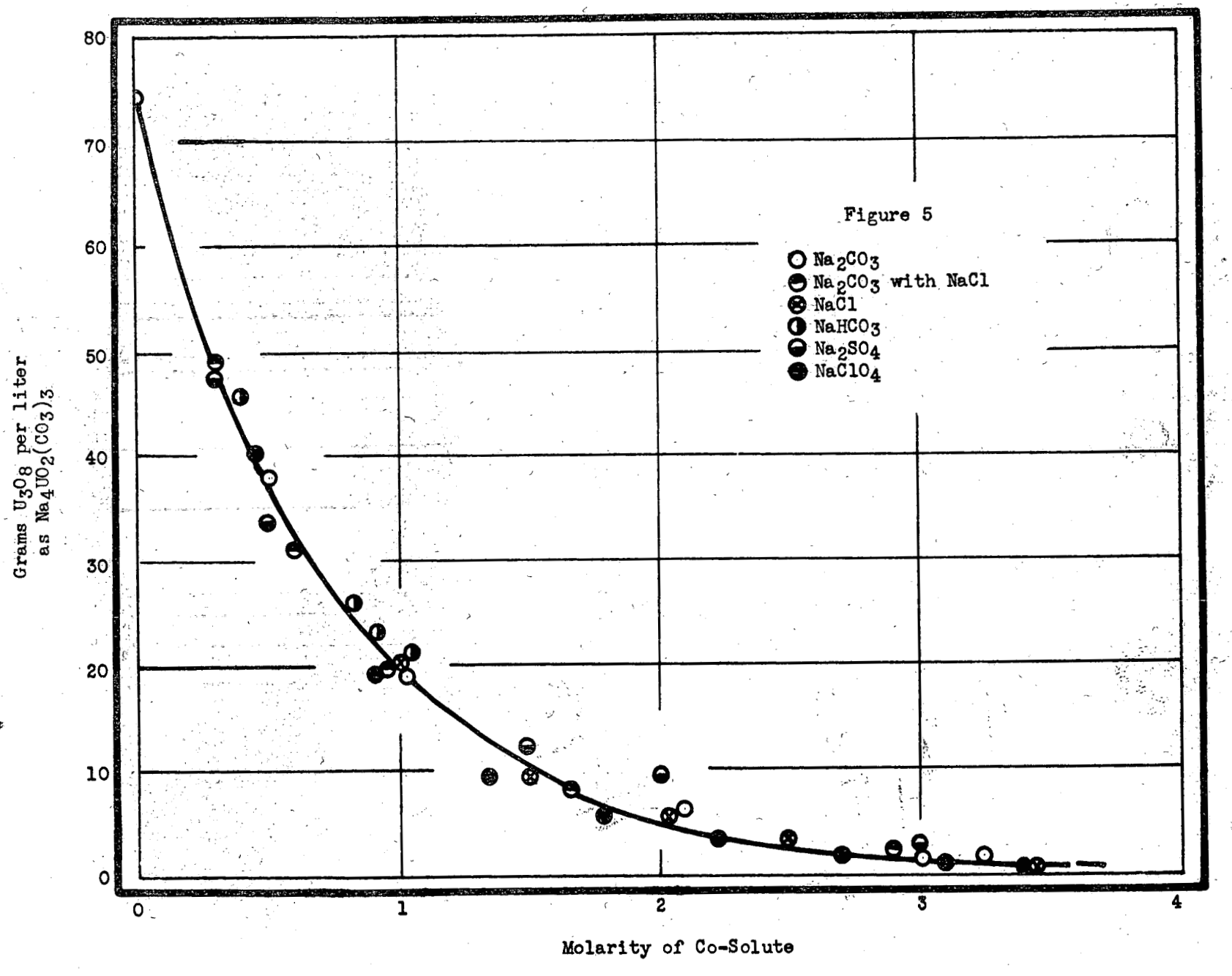
Figure 5
Na_2CO_3
Na_2CO_3 with NaCl
NaCl
$NaHCO_3$
Na_2SO_4
$NaClO_4$
Grams U_3O_8 per liter as $Na_4UO_2(CO_3)_3$
Molarity of Co-Solute
0
10
20
30
40
50
60
70
80
0
1
2
3
4

A brief discussion of this point is presented later.

For practical application the primary value of the data arises from the uniformity of the empirical relationship between solubility and molar concentration of the sodium salts. In systems analogous to those studied the equilibrium solubility of sodium uranyl tricarbonate can be closely predicted once the total molar concentration of sodium salts is known.

Solubility versus Ionic Strength

Although the results presented above are sufficient for any practical application to plant processing problems, their somewhat surprising pattern led to a further analysis of the data in which activity coefficients for the various salts in solution were considered.

If the solubility product for sodium uranyl tricarbonate $[Na_4UO_2(CO_3)_3]$ is expressed as follows:

$$S.P. = [Na^+]^4 [UO_2(CO_3)_3^{\equiv}] \qquad (1)$$

where $[x]$ = molality

values for S. P. can be determined readily for each experiment from a knowledge of the composition of the solutions at equilibrium. These values, along with the calculated ionic strengths of the corresponding solutions are given in Table 7. The variation of log S. P. with the square root of the ionic strength ($\sqrt{\mu}$) is depicted by Figure 6. To serve all of the experimental points, this variation is best expressed by a straight line, defined by the following equation:

$$\log S.P. = 1.25\sqrt{\mu} - 2.2 \qquad (2)$$

where temp. = 26-30°C

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Table 7

The Molal Solubility Product Versus Ionic Strength*

Exp. A.	Solution Type	Molal Solubility Product	Log S.P.	$\sqrt{\mu}$
145	Water	0.58	- 0.2369	1.7
419	0.3 M Na_2SO_4	0.44	- 0.3569	1.63
420	1.5 M "	6.06	+ 0.7826	2.29
421	2.0 M "	15.0	+ 1.1764	2.62
422	3.0 M "	28.3	+ 1.4524	3.18
192	1.0 M NaCl	0.288	- 0.5415	1.35
193	1.5 M "	0.278	- 0.5559	1.37
194	2.0 M "	0.483	- 0.3164	1.53
195	2.5 M "	0.630	- 0.2011	1.66
196	3.0 M "	0.723	- 0.1409	1.81
197	3.44 M "	0.651	- 0.1864	1.93
198	4.0 M "	0.801	- 0.0957	2.09
181	0.46 M $NaClO_4$	0.194	- 0.7111	1.40
182	0.90 M "	0.148	- 0.8300	1.28
183	1.33 M "	0.220	- 0.6553	1.34
184	1.80 M "	0.376	- 0.4245	1.47
185	2.25 M "	0.512	- 0.2908	1.62
186	2.70 M "	0.622	- 0.2064	1.76
187	3.11 M "	0.918	- 0.0376	1.92
188	3.40 M "	0.780	- 0.0974	2.02
Princeton	0.51 M Na_2CO_3	0.91	- 0.04051	1.72
"	1.03 M "	2.21	+ 0.3441	1.96
"	2.11 M "	8.61	+ 0.9355	2.49
"	3.24 M "	14.3	+ 1.1556	3.24

* Y-12 tests were at 26°C.
Princeton tests were at 30°C.

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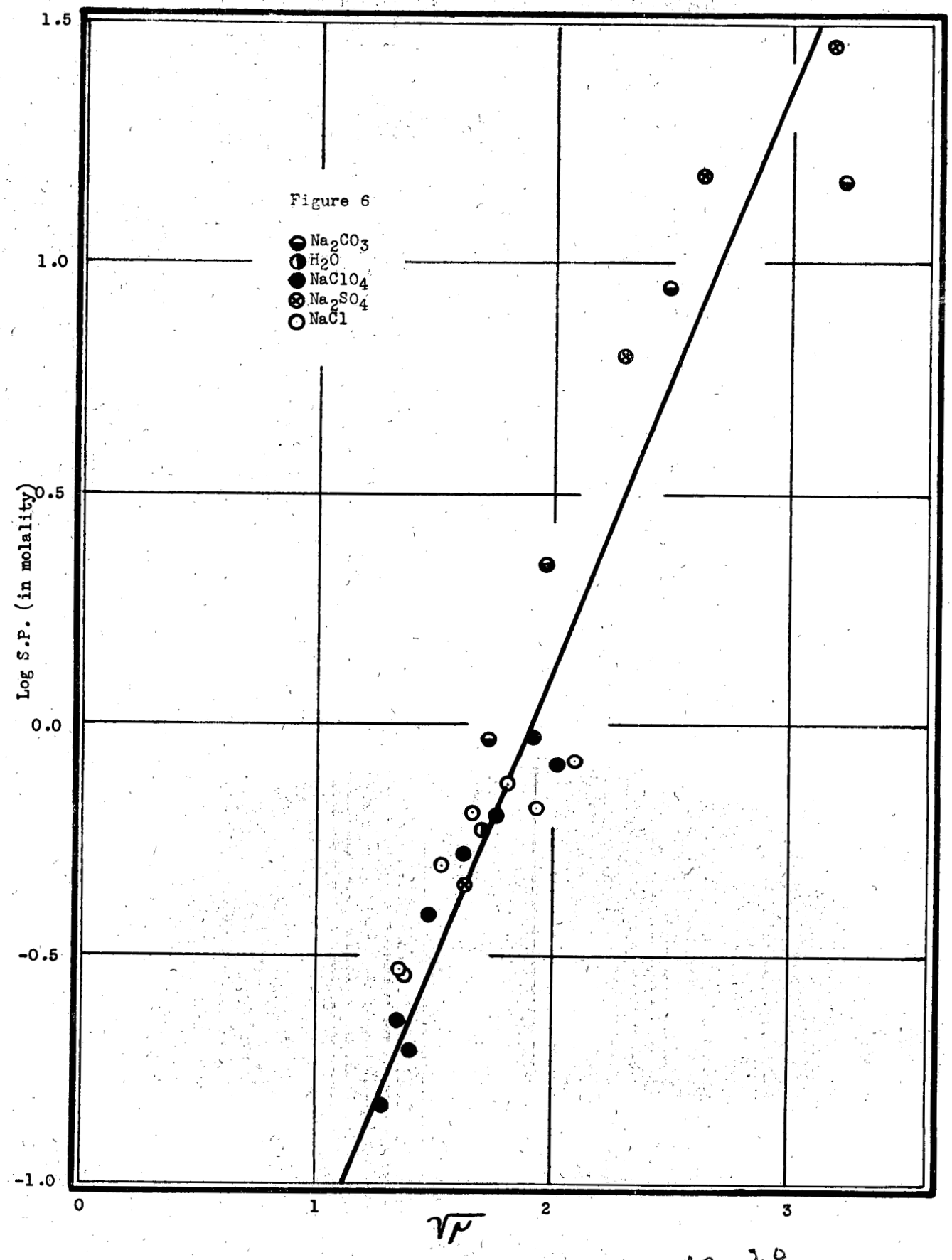

Figure 6

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Hence remembering that the activity coefficients for the salts are strongly dependent upon the ionic strength, the surprising observations described earlier are qualitatively accounted for. Also the empirical relationship given by equation (2) should have a practical use in predicting the results for other similar systems.

An extended treatment of the data is hardly justified in view of the high solubility of sodium uranyl tricarbonate and the inclusion of a fourth power term in the expression for the solubility product. It is interesting to note, however, that from equation (2) log $K_{s.p.}$ is equal to -2.2 at infinite dilution or $K_{s.p.}$ (the activity solubility product) is equal to 0.006. From the solubility measurements in sodium perchlorate solutions an independent calculation has been made according to the method proposed by Harned and Owen for highly soluble salts.[4] Using hydrochloric acid as the reference electrolyte, the calculated value for $K_{s.p.}$ is 0.01. Upon taking $K_{s.p.}$ = 0.006 and substituting into standard equations the mean ionic activity coefficient for sodium uranyl tricarbonate may be derived. For a saturated solution of the salt in water the calculated coefficient is about 0.4 -- a value somewhat greater than might be expected for a 4-1 sodium salt at this concentration.

Precipitation of Sodium Uranyl Tricarbonate

Only a few tests were made in which solid sodium uranyl tricarbonate was precipitated from the solution phase. In one experiment, solid sodium chloride was added to a saturated solution of the tricarbonate and precipitation was initiated by gentle warming. In another case precipitation

from a saturated solution was forced by the addition of methyl alcohol. Both solids contained sodium, uranium and carbonate in the correct proportion for sodium uranyl tricarbonate.

At room temperature the precipitation of tricarbonate from solution by addition of sodium salts takes place very slowly. Tests are currently underway in which solid sodium chloride and sodium carbonate have been added to saturate solutions of the tricarbonate. Only a slight precipitate has been evidenced after two weeks of standing.

APPLICATION OF DATA TO THE MONTICELLO PROCESS

For processes in which analogous solutions are encountered, the data described herein might be useful in predicting the limiting levels of uranium concentration. Except for a rather high vanadium content, the liquors obtained from the salt roast, carbonate leach process at Monticello, Utah fall into this category. If one can make a reasonable assumption that the presence of vanadium does nothing to enhance the solubility of uranium, the data should be applicable.

Actually it is believed that evidence for the effect of sodium uranyl tricarbonate solubility on the Monticello process was given by experiments conducted sometime ago at this laboratory. In report Y-571,[5] tests were described in which batches of salt roasted, carnotite ore were treated countercurrently with sodium carbonate solution to determine directly the maximum build-up of uranium that might be attained in the Monticello plant. During the course of these treatments, uranium was precipitated

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as a greenish-yellow compound which was subsequently dissolved by water. Chemical analyses of the precipitates showed the presence of sodium, uranium and carbonate with little, if any, vanadium. It seems likely that the material was sodium uranyl tricarbonate. A reconsideration of these experiments is now in order to determine whether the solubility limits as defined by the foregoing data had been exceeded.

In Table 8, the approximate NaCl, Na_2CO_3 and U_3O_8 concentrations are given for several leach liquors in which the yellow uranium precipitate had been observed. Uranium concentrations as predicted from the solubility studies are given in the right hand column. These values were derived by assuming that the consistency of the relationship between molarity of the co-solute and uranium concentrations at 90°C was comparable to that at 26°C, and reading the uranium concentration corresponding to the appropriate molarity from the plot in Figure 4. The vanadium contents of the solutions were ignored. Also it should be noted that although the leachings were conducted at 90°C, appreciably cooling may have occurred during filtration. Sodium uranyl tricarbonate has a somewhat greater solubility at lower temperatures.

Though no attempt was made at the time to obtain precise compositions of the leach solutions, the agreement between the measured results and those predicted is quite reasonable. From consideration of this data and from consideration of the fact that sodium uranyl tricarbonate is the only water soluble compound likely to be formed in these liquors, it is believed that the solubility of sodium uranyl tricarbonate is a factor to be considered at the Monticello plant.

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Table 8

Uranium Build-up in Sodium Carbonate Leaching of Salt Roasted Carnotite Ores

Test No.	Leach Solns in which a U Ppt. has Formed: NaCl (approx. M)	Na_2CO_3 (approx. M)	U_3O_8 (g/l)	Predicted Result U_3O_8 (approx. g/l)
L-560-569	1.7	0.3	3.6	3.5
L-671-675	2.0	0.3	3.8	3
	2.2	0.6	5.0	3
L-711-714	1.2	0.6	4.8	4
L-568	2.8	0.6	1.4	1
L-569	3.0	0.6	0.9	< 1
L-673	3.0	0.3	1.4	1
L-675	3.8	0.3	0.8	< 1

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DISCUSSION OF DATA

Stability of Solutions

The reproducibility of the foregoing solubility results shows that the water, sodium carbonate, and sodium bicarbonate solutions of sodium uranyl tricarbonate lie in a well defined region of the UO_3 - Na_2O - CO_2 - H_2O system. This is better demonstrated perhaps by other studies in the system which have been described in report Y-673 (part I of the series)[6]. Also from report Y-673 it may be noted that different and probably erratic solubilities would be expected in solutions containing mixtures of sodium hydroxide and sodium carbonate because of the formation of complex hydrolysis products. The leach liquors at Monticello apparently contain mixtures of sodium carbonate and sodium bicarbonate since the uranium solubilities predicted on this basis are in reasonable agreement with those measured.

Rate of Sodium Uranyl Tricarbonate Precipitation

It has been mentioned previously that the precipitation of sodium uranyl tricarbonate from supersaturated solutions is very slow at room temperature, although it may be hastened somewhat by heating. Hence, in leaching of ores, considerable time might elapse between the dissolution of the uranium mineral and the precipitation of the tricarbonate, even though the concentration of sodium salts be so high that the tricarbonate solubility is exceeded. Apparently, equilibrium was rapidly attained in the high temperature leaching of the salt-roasted Monticello ores (see above), but this does not eliminate the possibility that the equilibrium

solubility limit might be exceeded in actual plant practice.

The Uranyl Tricarbonate Anion

The existence of a uranyl tricarbonate ion in solution has been postulated by several workers, but to our knowledge it has never been proved. The conformation of the measured solubility to a mass action expression based on the tetrasodium uranyl-tricarbonate formula provides indirect evidence for the existence and stability of the tricarbonate ion, $UO_2(CO_3)_3^{-4}$. Direct evidence of the existence of this ion in analogous systems has been obtained at this laboratory by colorimetric studies,[7] which have also shown the existence in somewhat different systems of other complex ions, e.g., a uranyl dicarbonate complex and a mixed complex with carbonate and hydroxyl ions.

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Several important parts of this investigation were carried out by Dr. C. A. Blake and Mr. R. S. Lowrie.

Most of the analyses were performed by the Y-12 section of the ORNL Analytical Chemistry Division under the direction of Mr. C. D. Susano.

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