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VAPOR-LIQUID-SOLID PHASE EQUILIBRIA OF RADIOACTIVE SODIUM SALT WASTES AT HANFORD

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January 1976

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ABSTRACT

Phase equilibria of the seven-component system, $NaOH-NaNO_3-NaNO_2-NaAlO_2-Na_2SO_4-Na_2CO_3-H_2O$, *were studied. This system simulates radioactive sodium salt wastes stored in underground tanks at Hanford. Equilibrium water vapor pressure of the aqueous phase and solubility of the salts were measured as a function of temperature and NaOH concentration. These data are used to predict deliquescence of stored salt wastes and also to predict the chemical identity of solid residues from evaporation of liquid wastes.*

VAPOR-LIQUID-SOLID PHASE EQULIBRIA OF RADIOACTIVE SODIUM SALT WASTES AT HANFORD

INTRODUCTION

Radioactive sodium salt wastes have been accumulated at Hanford as a result of reprocessing spent fuel elements from nuclear reactors. Sodium nitrate, the principal salt, was formed by neutralization of a nitric acid waste stream (containing fission products) with sodium hydroxide. Other sodium salts were added to the waste from various processing steps. The salts present in the highest concentrations are: $NaNO_3$, $NaNO_2$, NaOH, $NaAlO_2$, Na_2SO_4, and Na_2CO_3. Radioactive salts comprise only a very small fraction of the waste volume.

Aqueous solutions of these salts are presently being evaporated in order to immobilize the radionuclides in crystalline salt cakes. Phase equilibrium data are necessary to predict the identity of salts precipitated by evaporation and to predict whether or not a given salt waste composition will deliquesce under storage conditions. This information will allow improved control of evaporation-crystallization processes for solidification of the radioactive waste and help determine the safety of salt cake waste for long-term storage.

As expected, very few equilibrium data are available in the literature on systems having compositions similar to that of Hanford sodium salt wastes. The systems which come closest are: $NaOH-NaAlO_2-H_2O$,[1,2] $NaOH-NaNO_3-H_2O$,[3] and $NaOH-NaNO_2-H_2O$.[3] Vapor pressure and solubility data are also available for the two component systems, Salt-H_2O, where Salt = NaOH, $NaNO_3$, $NaNO_2$, Na_2SO_4, or Na_2CO_3. Mendel[4] has measured water vapor pressure for several different synthetic alkaline waste compositions. However, these measurements were limited to temperatures above 75° C.

The seven-component system, $NaOH-NaNO_3-NaNO_2-NaAlO_2-Na_2SO_4-Na_2CO_3-H_2O$, was chosen for study since these salts represent the major components of Hanford wastes. It is assumed that minor components have insignificant effects on the phase data. No attempt was made to obtain complete phase diagrams. Only water vapor pressures and salt solubilities were measured as a function of temperature and NaOH concentration. In each experiment the aqueous phase was saturated with $NaNO_3$, $NaNO_2$, $NaAlO_2$, Na_2SO_4, and Na_2CO_3. The condition of saturation was imposed because it closely simulates waste solution conditions during the evaporation-crystallization process and during storage of salt cake-interstitial liquor mixtures. In addition, the concentration variable for each salt (except NaOH) was thus eliminated, substantially reducing the number of measurements necessary. Sodium hydroxide was considered a variable because a wide range of concentrations has been found in the wastes. Also, NaOH concentration is known to strongly influence the vapor pressure of aqueous solutions and solubility of other salts.

SUMMARY AND CONCLUSIONS

Water vapor pressure of the seven-component system, $NaOH-NaNO_3-NaNO_2-NaAlO_2-Na_2SO_4-Na_2CO_3-H_2O$, was measured as a function of temperature and NaOH concentration. This system simulates actual Hanford high-salt, radioactive wastes (crystalline salts containing interstitial liquor). The aqueous phase was saturated with each component salt, except for NaOH. The vapor pressure data show that the waste will not deliquesce in Hanford air as long as the NaOH concentration in the interstitial liquor is below $\sim$ 6.4*M* (at 17° C). However, if the waste is exposed to surrounding soil through cracks in tank walls, it will deliquesce as it approaches soil temperature. The rate of water diffusion into the tanks is calculated to be very slow--only a few liters per year.

Solubilities of the salts in the seven-component system were also measured as a function of temperature and NaOH concentration. The solubility curves for $Al(OH)_3$ and $NaAlO_2$ in this system are significantly different from the $NaOH$-$NaAlO_2$-H_2O system. The maximum aluminum solubility occurs at $\sim$2*M* NaOH for the system studied, compared with $\sim$6*M* NaOH in the three-component system. In general, the solubility of the salts decreased in the order: $NaNO_2 > NaNO_3 >> Na_2CO_3 > Na_2SO_4$. These results indicate that Hanford concentrated waste liquor is saturated with each of these salts except for $NaNO_2$.

EXPERIMENTAL

SYNTHETIC WASTE MIXTURES

The compositions of synthetic waste mixtures used for making vapor pressure and solubility measurements are given in Table I. Reagent-grade chemicals and distilled water were used in each mixture.

VAPOR PRESSURE MEASUREMENTS

Vapor pressures of the synthetic waste solutions were measured by the "gas saturation method." The design of the gas saturators was the same as that given by Thomson.[5] To

TABLE I

COMPOSITION OF SYNTHETIC WASTE MIXTURES

Waste Mixture	Weight of Component, g						
	H_2O	NaOH	$Al(OH)_3$	$NaNO_3$	$NaNO_2$	Na_2SO_4	Na_2CO_3
1	250	375	10	20	30	5	5
2A	300	200	200	150	150	10	10
3A	350	150	200	150	150	20	20
3.5A	375	125	200	250	250	20	20
4A	400	100	150	250	250	50	50
5A	450	50	100	300	300	100	100
6A	300	300	250	150	150	10	10

make a measurement, a sample of the waste mixture was placed in the saturator which was fitted with a gas drying tube filled with Drierite® (Baker and Adamson). The saturator was also fitted with a manometer to measure pressure above the sample and thermostated by placing it in a constant temperature (± 0.5° C) oven. After equilibration for several days at the specified temperature, nitrogen gas was bubbled through the sample in the saturator at a constant rate and then passed into the drying tube, a second saturator filled with distilled water, and then into a second drying tube. After several hours the gas flow was stopped and the drying tubes weighed to measure the amount of water absorbed. The vapor pressure of the sample was calculated from the equation

$$P_w = P_w' \left(\frac{g_w}{g_w'}\right) \left(\frac{P_t - P_w}{P_t' - P_w'}\right)$$

where

P_w = vapor pressure of sample

P_w' = vapor pressure of distilled water

g_w = weight of water absorbed in sample drying tube

g_w' = weight of water absorbed in distilled water drying tube

P_t = total pressure above sample

P_t' = total pressure above distilled water (atmospheric).

Vapor pressures were measured at 20°, 40°, 60°, and 80° C for each mixture. Duplicate measurements were averaged.

SOLUBILITY MEASUREMENTS

Solubilities of salts were measured by analyzing the aqueous phase after the synthetic waste mixture had been equilibrated at the specified temperature for three to seven days. After this time the mixtures were rapidly filtered using a preheated plastic Millipore® (Millipore Corp.) syringe filter [with a Solvinert® (Millipore Corp.) membrane]. The filtrates were diluted 1:10 with distilled water to prevent crystallization during cooling, and the dilute solutions analyzed for OH^-, $Al(OH)_4^-$, NO_3^-, NO_2^-, $SO_4^=$, and $CO_3^=$. The methods used for these analyses are given in Table II. The filtrates were also diluted 1:10 with 6*M* NaOH to prevent precipitation of gibbsite [$Al(OH)_3$] by the reaction

$$Al(OH)_4^- \rightarrow \underset{\text{(gibbsite)}}{Al(OH)_3} + OH^-$$

TABLE II
METHODS OF ANALYSIS OF DILUTED SOLUTIONS

Ion Analyzed	Method
OH^-	Potentiometric acid titration[6]
$Al(OH)_4^-$	Potentiometric acid titration[6]
NO_3^-	Specific ion electrode
NO_2^-	Iodo-Permanganate titration[7]
$SO_4^=$	Gravimetric barium precipitation[8]
$CO_3^=$	Barium carbonate precipitation[6]

Gibbsite precipitated from most of the water-diluted samples. Therefore, to measure OH^- and $Al(OH)_4^-$ concentrations accurately, aluminum analyses were performed on both the water-diluted and NaOH-diluted samples. The difference in $Al(OH)_4^-$ concentration equaled the increase in OH^- concentration (due to gibbsite precipitation) in the water-diluted samples.

The establishment of equilibrium during measurements of vapor pressure and solubility was determined as follows. Two identical synthetic waste mixtures were prepared. The composition of the aqueous phase of the first mixture was measured at 80° C (after an equilibration period). This was repeated at 60°, 40°, and 20° C. The aqueous phase composition of the second mixture was determined similarly but in the order 20°, 40°, 60°, and 80° C. If the results for both mixtures were comparable, the mixture was assumed to be at equilibrium.

RESULTS AND DISCUSSION

COMPOSITION OF SALT CAKE AND ASSOCIATED MOTHER LIQUOR

Recent analyses of salt cake and mother liquor products of the Hanford evaporator-crystallizer have shown that the crystalline phase is composed primarily of $NaNO_2$ and Na_2CO_3.[9] Sodium sulfate was not determined, but is also present. The major components of the mother liquor were found to be NaOH, $NaNO_3$, $NaNO_2$, $NaAlO_2$, and Na_2CO_3. Concentrations of these salts for several mother liquor products are shown in Table III.

TABLE III

COMPOSITION* OF MOTHER LIQUOR PRODUCTS

Component	Molar Concentration of Component in Mother Liquor from Tank No.			
	105-S	106-S	108-S	110-S
NaOH	6.46	5.33	5.18	3.19
$NaNO_3$	1.95	2.49	2.30	3.72
$NaNO_2$	0.99	0.66	1.14	1.65
$NaAlO_2$	1.69	1.56	1.05	1.48
Na_2CO_3	0.07	0.01	0.09	0.20

*[Na_2SO_4] was not determined although known to be present.

To closely simulate the behavior of actual wastes, the synthetic waste mixtures used in obtaining the vapor pressure and solubility data contained each of the above salts in addition to Na_2SO_4 and water.

VAPOR PRESSURE RESULTS

Water vapor pressures of synthetic waste solutions were measured for various NaOH concentrations (1.1 to 27*M*) and temperatures (20° to 80° C). These solutions were saturated with $NaNO_3$, $NaNO_2$, $NaAlO_2$, Na_2SO_4, and Na_2CO_3. The results are presented graphically in Figure 1. Since the NaOH concentration varied somewhat at different temperatures for a given sample mixture, the average concentration is given in the Figure (except for the solutions saturated with NaOH).

Deliquescence of salt cake products during storage can be predicted using the vapor pressure data in Figure 1. However, the storage conditions (temperature and water vapor pressure of waste environment) and the NaOH concentration of the interstitial liquor must be known. For example, if the water vapor pressure of the waste is less than that of the storage environment, water will be absorbed and the soluble salts will deliquesce. If the situation is reversed, the waste will become drier.

There appear to be two possible air environments for waste stored in underground tanks. First, the waste in the tanks may be exposed to atmospheric air, which has a maximum monthly average water vapor pressure of about 4.5 mm Hg at the Hanford site.[10] Second, the waste may be exposed to air in contact with soil (because of formation of cracks in the tank walls). This air will be nearly saturated with water (∿15 mm Hg partial pressure at 17° C) because of the vapor pressure of soil pore water.[11] Deliquescence temperatures (temperatures below which the waste will deliquesce) are plotted as a function of NaOH concentration in

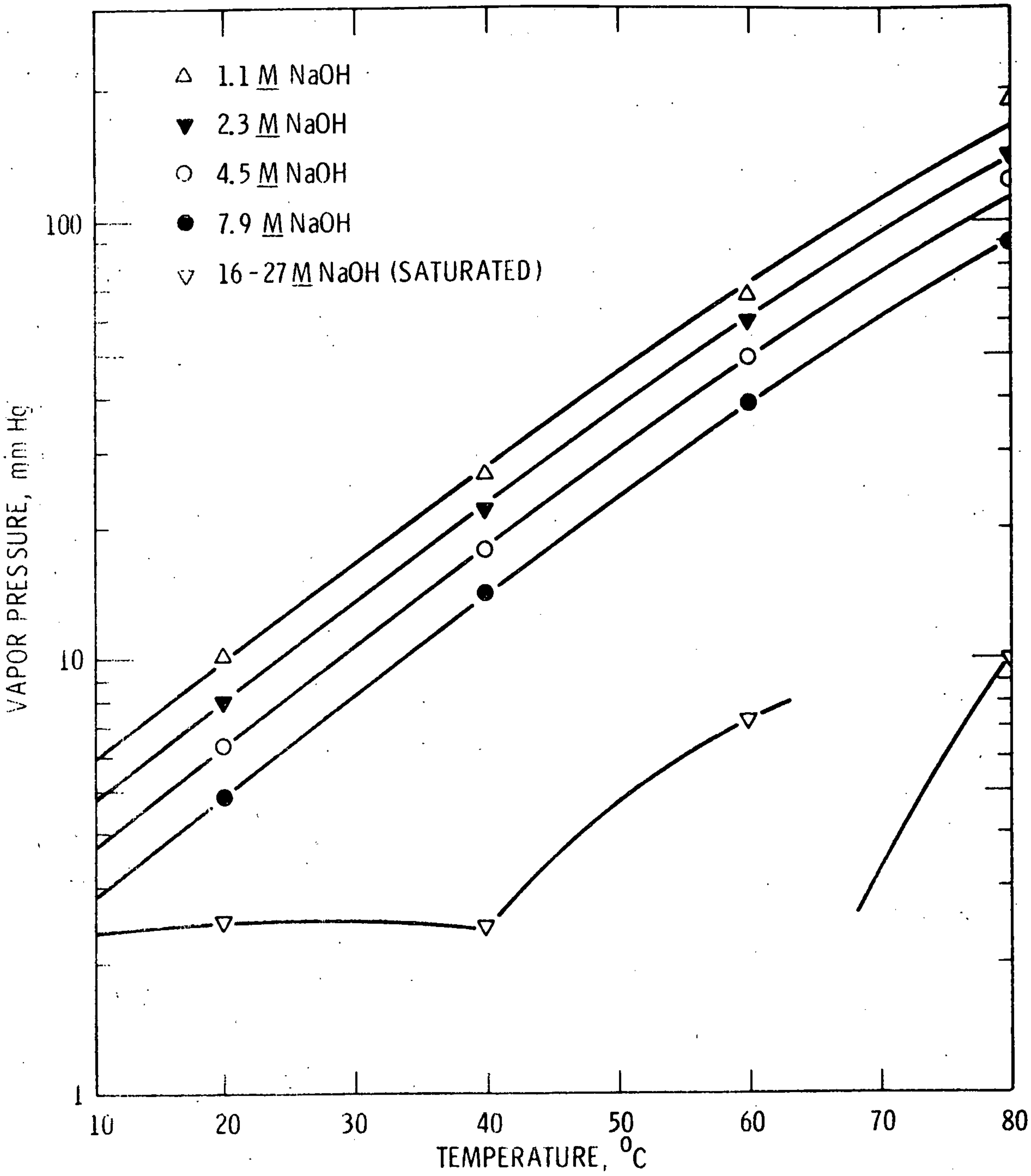

FIGURE 1

VAPOR PRESSURES OF SYNTHETIC WASTE SOLUTIONS
(SATURATED WITH $NaNO_3$, $NaNO_2$, $NaAlO_2$, Na_2CO_3, AND Na_2SO_4)

Figure 2 for both storage environments. Assuming a minimum storage temperature of 17° C (soil temperature), the curve for Hanford air shows that the waste will absorb water only when NaOH concentration is greater than ∿6.4*M* (at 17° C). However, if the waste is exposed to air in contact with soil, it will deliquesce at all concentrations of NaOH depending on the temperature of the waste. Of course, the deliquescence temperature decreases as the NaOH concentration in the interstitial liquor decreases.

The amount of water absorbed by the waste from soil can be calculated from the vapor pressure data, given an initial NaOH concentration in the interstitial liquor and a final temperature. For example, at a constant temperature of 30° C, waste having an initial NaOH concentration of 6.0*M* would absorb water until an equilibrium concentration of 1.7*M* NaOH was reached. The volume of the interstitial liquor would increase by a factor of 3.5 in this case.

The rate of diffusion of water vapor from soil into a tank has been estimated. Details of the method and results of these calculations are given in the Appendix. In general, the diffusion rates are only a few liters/year for almost any plausible storage conditions.

As mentioned in the Introduction, vapor pressure measurements can be used to predict the composition of mother liquor from the evaporator-crystallizer. Assuming equilibrium conditions and given an operating temperature and pressure, the NaOH concentration in the mother liquor can be obtained from the data in Figure 1. The composition of the crystalline product and concentrations of other components in the mother liquor can be obtained from solubility measurements given below.

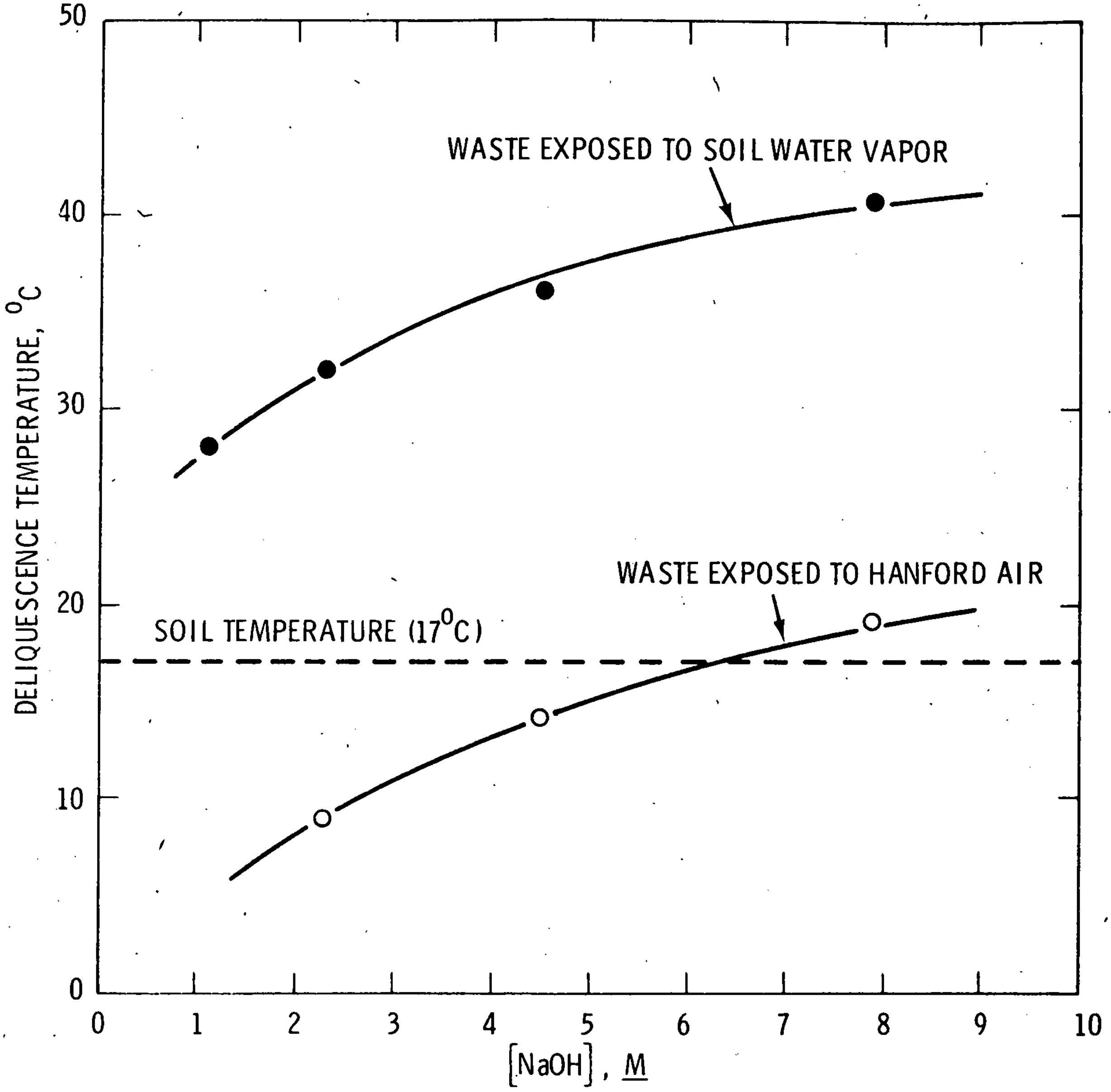

FIGURE 2

DELIQUESCENCE TEMPERATURE (TEMPERATURE BELOW WHICH THE THE WASTE WILL ABSORB WATER FROM STORAGE ENVIRONMENT) AS A FUNCTION OF NaOH CONCENTRATION IN THE INTERSTITIAL LIQUOR

SOLUBILITY RESULTS

To control the evaporation-crystallization of Hanford wastes accurately it is necessary to be able to predict the identity of crystalline products. For example, precipitation of aluminum compounds [$Al(OH)_3$ or $NaAlO_2$] must be avoided since they precipitate as very small crystals which are difficult to separate from the liquid phase. Solubility curves for these compounds give conditions which must be met in order to keep the aluminum in solution.

Figure 3 shows the solubilities of $Al(OH)_3$ and $NaAlO_2$ as a function of NaOH concentration and temperature. As with all the solubility and vapor pressure measurements, the aqueous phase was saturated with $NaNO$, $NaNO_3$, $NaAlO_2$, Na_2SO_4, and Na_2CO_3. The left-hand curve of Figure 3 is the solubility curve for $Al(OH)_3$ (gibbsite) while the right-hand curve is the solubility curve for $NaAlO_2 \cdot 1.25H_2O$ (assuming the same sodium aluminate solid phase as for the Na_2O-Al_2O_3-H_2O system). At the point of intersection of these curves both solid phases and solution are present. According to these curves the maximum aluminum solubility ($\sim 4M$) occurs at $\sim 2M$ NaOH. This maximum appears at a much lower NaOH concentration than for the pure Na_2O-Al_2O_3-H_2O system, as shown by the broken line curves.[12] Surprisingly, temperature seems to have little effect on the solubility. No significant trend was observed between 20° and 80° C. It is interesting to note that all available measurements of $NaAlO_2$ and NaOH concentrations in actual evaporator-crystallizer mother liquors fall below these solubility curves.

Solubility curves for $NaNO_3$, $NaNO_2$, Na_2SO_4, and Na_2CO_3 are shown in Figures 4, 5, 6, and 7, respectively. In general the solubility decreased in the order: $NaNO_2 > NaNO_3 >> Na_2CO_3 > Na_2SO_4$. A comparison of these

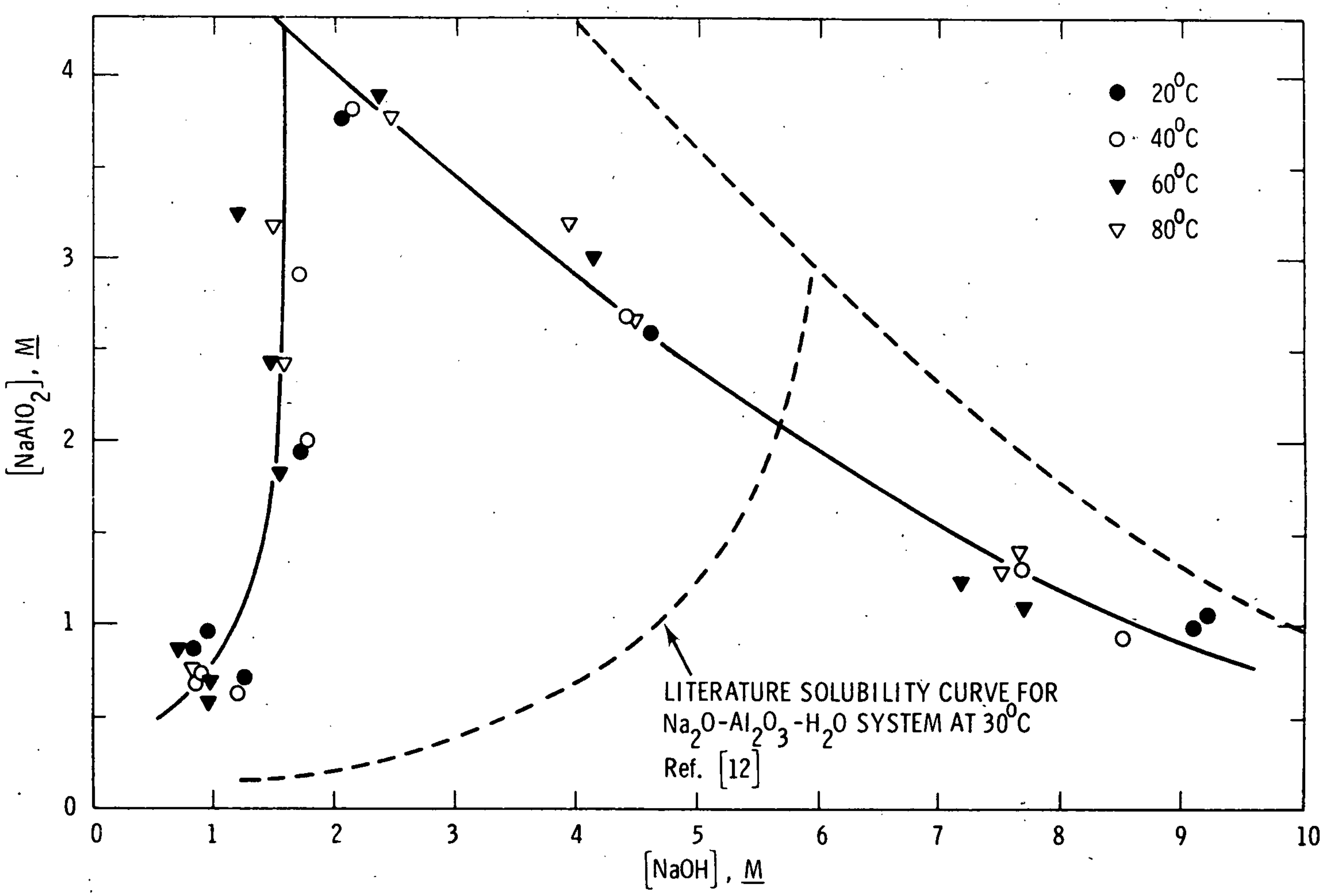

FIGURE 3

SOLUBILITY OF SODIUM ALUMINATE IN SYNTHETIC WASTE SOLUTIONS
(SATURATED WITH $NaNO_3$, $NaNO_2$, Na_2SO_4, AND Na_2CO_3)

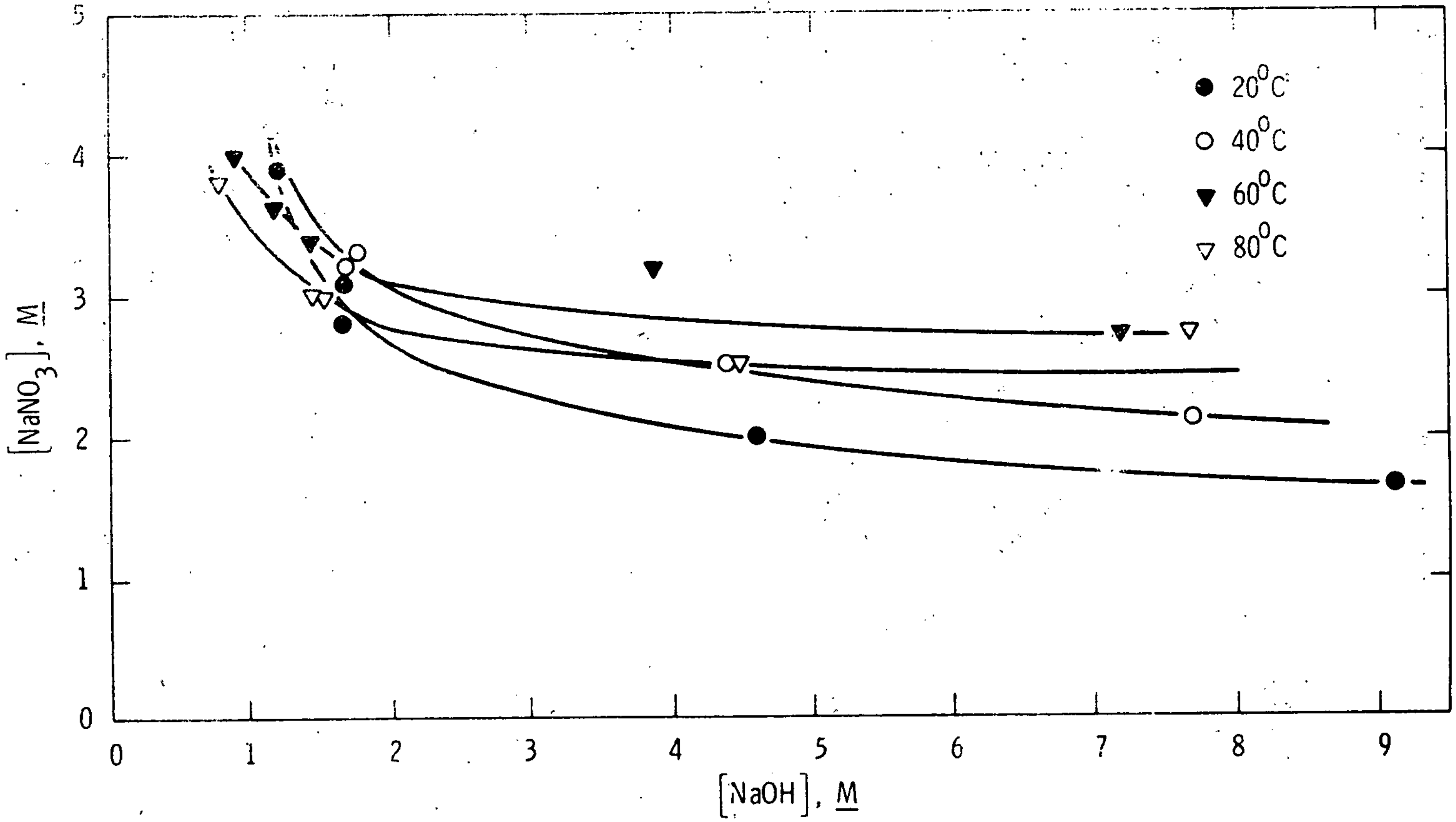

FIGURE 4

SOLUBILITY OF SODIUM NITRATE IN SYNTHETIC WASTE SOLUTIONS (SATURATED)

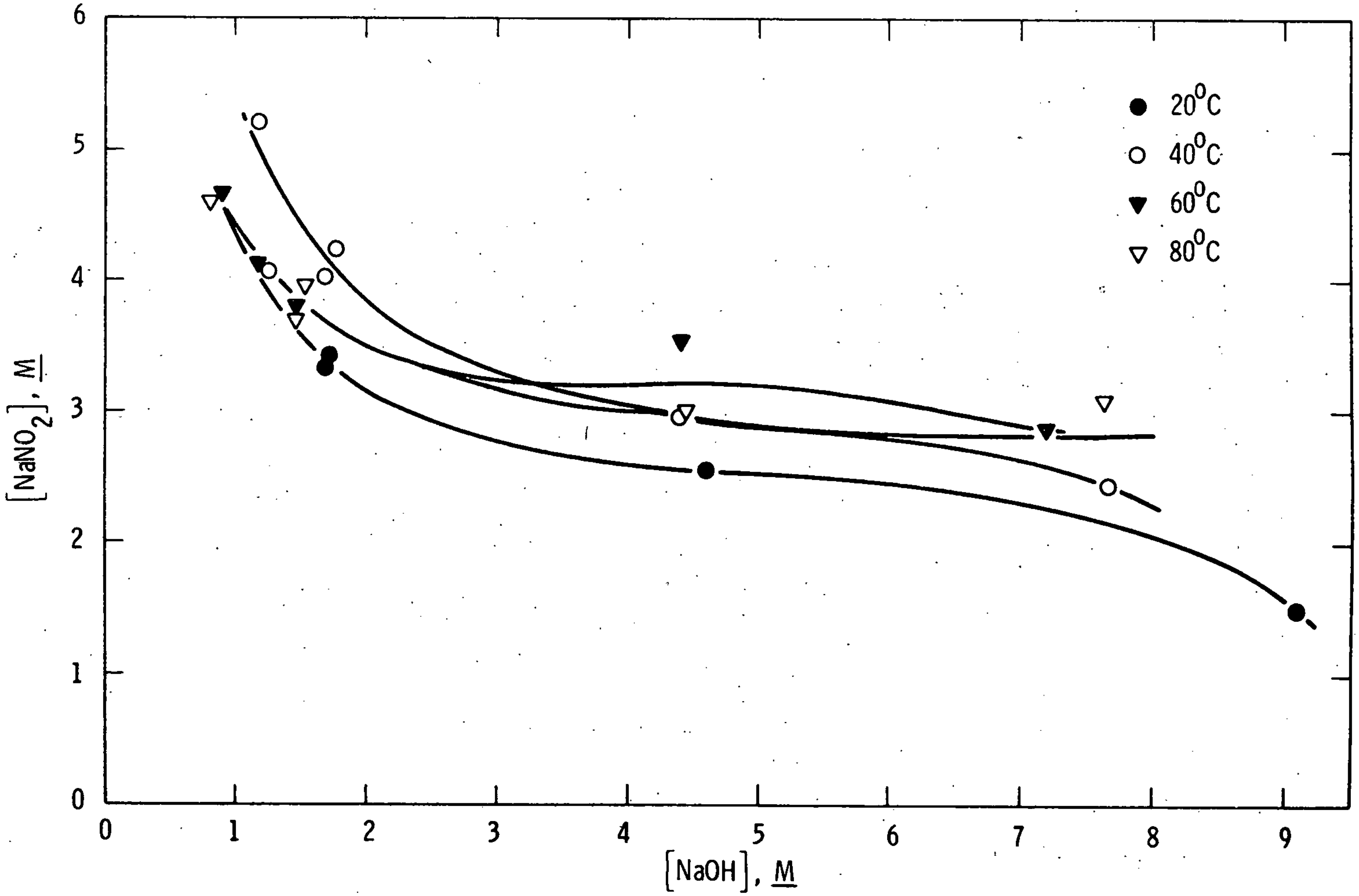

FIGURE 5

SOLUBILITY OF SODIUM NITRITE IN SYNTHETIC WASTE SOLUTIONS (SATURATED)

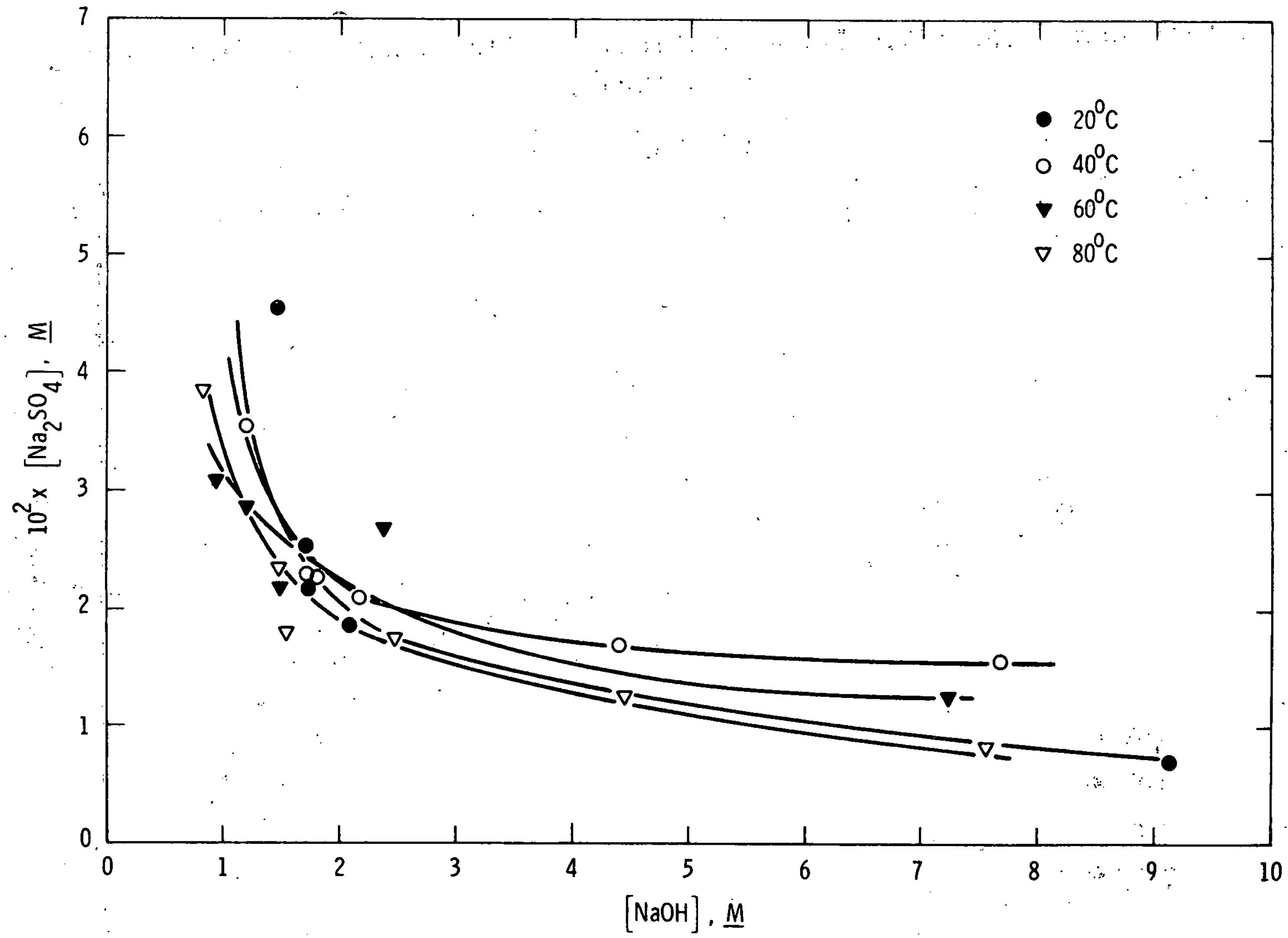

FIGURE 6

SOLUBILITY OF SODIUM SULFATE IN SYNTHETIC WASTE SOLUTIONS (SATURATED)

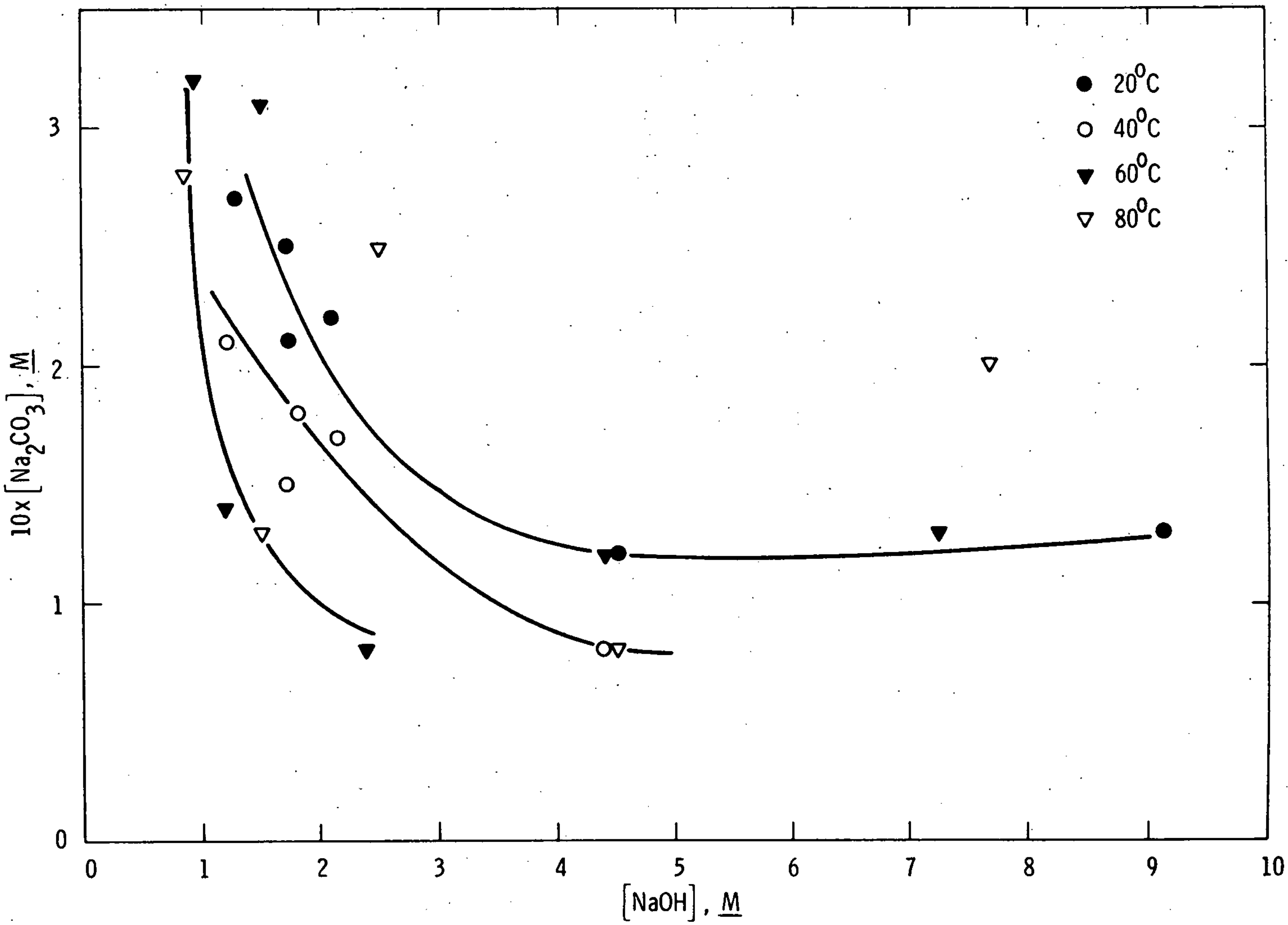

FIGURE 7

SOLUBILITY OF SODIUM CARBONATE IN SYNTHETIC WASTE SOLUTIONS (SATURATED) solubilities with analyses of actual waste liquors indicates that the aqueous waste is saturated with each of these salts except for $NaNO_2$.

The solubility of each of the salts increases sharply as the NaOH concentration drops below ∿2*M*. This may be a common ion effect due to the rapid <u>decrease</u> in solubility of $NaAlO_2$ below ∿2*M*. There is an increase in solubility with temperature only at the higher NaOH concentrations. The solubility is more complex at lower NaOH concentrations.

The formation of compounds between various components of the waste can, of course, affect the solubilities of the salts. Several compounds are known to exist between components of the system studied, for example: $Na_2CO_3 \cdot xH_2O$ (x = 1, 7, or 10), $Na_2SO_4 \cdot xH_2O$ (x = 7 or 10), $Na_2CO_3 \cdot 2Na_2SO_4$, and $NaNO_2 \cdot Na_2SO_4 \cdot 2H_2O$. In order to simplify the data, compound formation was ignored.

SUMMARY OF DATA

A summary of the data obtained in this study is given in Table IV. Values for water vapor pressure, solubilities, and densities of the saturated waste solutions are given at various temperatures and NaOH concentrations.

ACKNOWLEDGMENT

The author expresses appreciation to G. W. Cramer for his experimental assistance.

TABLE IV

VAPOR PRESSURE, COMPOSITION, AND DENSITY OF SATURATED WASTE SOLUTIONS[*]

					Solubility in Saturated Solution				
Sample No.	Temperature °C	Vapor Pressure mm Hg	Density g/ml	[NaOH] M	[$NaAlO_2$] M	[$NaNO_3$] M	[$NaNO_2$] M	[Na_2CO_3] M	[Na_2SO_4] M
1	20	2.5	1.51	15.9	0.17	0.52	0.72	0.023	-
1	40	2.4	1.59	17.5	0.26	0.34	0.82	0.021	-
1	60	7.1	1.61	17.2	0.17	0.34	0.94	0.019	-
1	80	9.9	1.68	26.3	0.17	0.27	0.80	0.037	-
6A	20	4.9	1.49	9.1	1.00	1.6	1.47	0.13	0.0069
6A	40	14.1	1.51	7.68	1.32	2.1	2.40	0.31	0.0151
6A	60	38.9	1.55	7.20	1.24	2.7	2.83	0.13	0.0122
6A	80	86.4	1.46	7.66	1.40	2.7	3.08	0.20	0.0080
2A	20	6.4	1.55	4.61	2.60	2.0	2.55	0.12	0.0353
2A	40	17.7	1.58	4.42	2.67	2.5	2.95	0.08	0.0169
2A	60	48.6	1.59	-	2.47	3.2	3.53	0.12	0.0554
2A	80	127	1.57	4.45	2.69	2.5	2.98	0.08	0.0125
3A	20	8.1	1.55	2.07	3.77	2.6[†]	2.85[†]	0.22	0.0186
3A	40	21.9	1.56	2.14	3.81	2.7[†]	2.91[†]	0.17	0.0208
3A	60	59.8	1.55	2.38	3.89	2.7[†]	2.66[†]	0.08	0.0266
3A	80	140	1.54	2.48	3.77	2.5[†]	2.86[†]	0.25	0.0173
3.5A	20	10.8	1.53	-	2.24	2.8	3.38	0.21	0.0213
3.5A	40	22.3	1.56	1.71	2.91	3.2	4.01	0.15	0.0224
3.5A	60	57.7	1.57	1.20	3.24	3.6	4.09	0.14	0.0285
3.5A	80	165	1.56	1.49	3.17	3.0	3.67	0.13	0.0231
4A	20	10.3	1.50	1.70	1.94	3.1	3.31	0.25	0.0252
4A	40	23.1	1.54	1.79	2.00	3.3	4.20	0.18	0.0225
4A	60	57.8	1.51	1.46	2.42	3.4	3.80	0.31	0.0215
4A	80	141	1.51	1.55	2.42	3.0	3.99	0.36	0.0179
5A	20	10.4	1.47	1.26	0.70	3.9	4.19	0.27	0.0453
5A	40	25.9	1.51	1.20	0.62	4.2	5.36	0.21	0.0353
5A	60	66.9	1.48	0.92	0.72	4.0	4.66	0.32	0.0309
5A	80	186	1.46	0.83	0.74	3.8	4.57	0.28	0.0383

[*] Solutions were saturated with $NaAlO_2$, $NaNO_3$, $NaNO_2$, Na_2CO_3, and Na_2SO_4.

[†] Sample No. 3A was probably not completely saturated with $NaNO_3$ and $NaNO_2$.

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APPENDIX

APPENDIX

CALCULATION OF THE RATE OF DIFFUSION OF WATER VAPOR INTO AN UNDERGROUND WASTE STORAGE TANK

The rate of water vapor diffusion from surrounding soil into an underground waste storage tank (through cracks in tank walls) can be estimated using steady state diffusion equations.[1] The following assumptions must be made:

1. Air convection is not significant.
2. Water vapor behaves as an ideal gas.
3. The water vapor pressure at the soil-tank interface is constant (∿15 mm Hg @ 17.4° C).
4. Soil temperature is constant.

The steady state equation

$$J = -D\frac{(C_t - C_s)}{L} \tag{1}$$

can be used where J is the water vapor flux, moles/cm^2-sec; D is the diffusion coefficient, cm^2/sec, for water vapor in air; L is the length of diffusion path, cm; and C_t and C_s are the water vapor concentrations, moles/cm^3, in the tank and in the soil, respectively. Assuming that water vapor behaves ideally, then

$$C = \frac{n}{V} = \frac{P}{RT} \tag{2}$$

and

$$J = -\frac{D}{RTL}(P_t - P_s) \tag{3}$$

---

[1] W. Jost, Diffusion in Solids, Liquids, Gases, Academic Press, Inc., New York, 1952

where R is the gas constant, mm Hg-cm^3/g mole-°K, T is the temperature, °K; and P_t and P_s are water vapor pressures, mm Hg, in the tank and in the soil, respectively.

Equation (3) was used to calculate rates of water diffusion, liters/year, into a tank, assuming various sizes of openings in the tank wall and several values of ΔP ($\Delta P = P_s - P_t$). The results of these calculations are shown in Figure 8. The curves indicate that under almost any plausible conditions of ΔP and size of tank opening the rate of diffusion will be only a few liters/year.

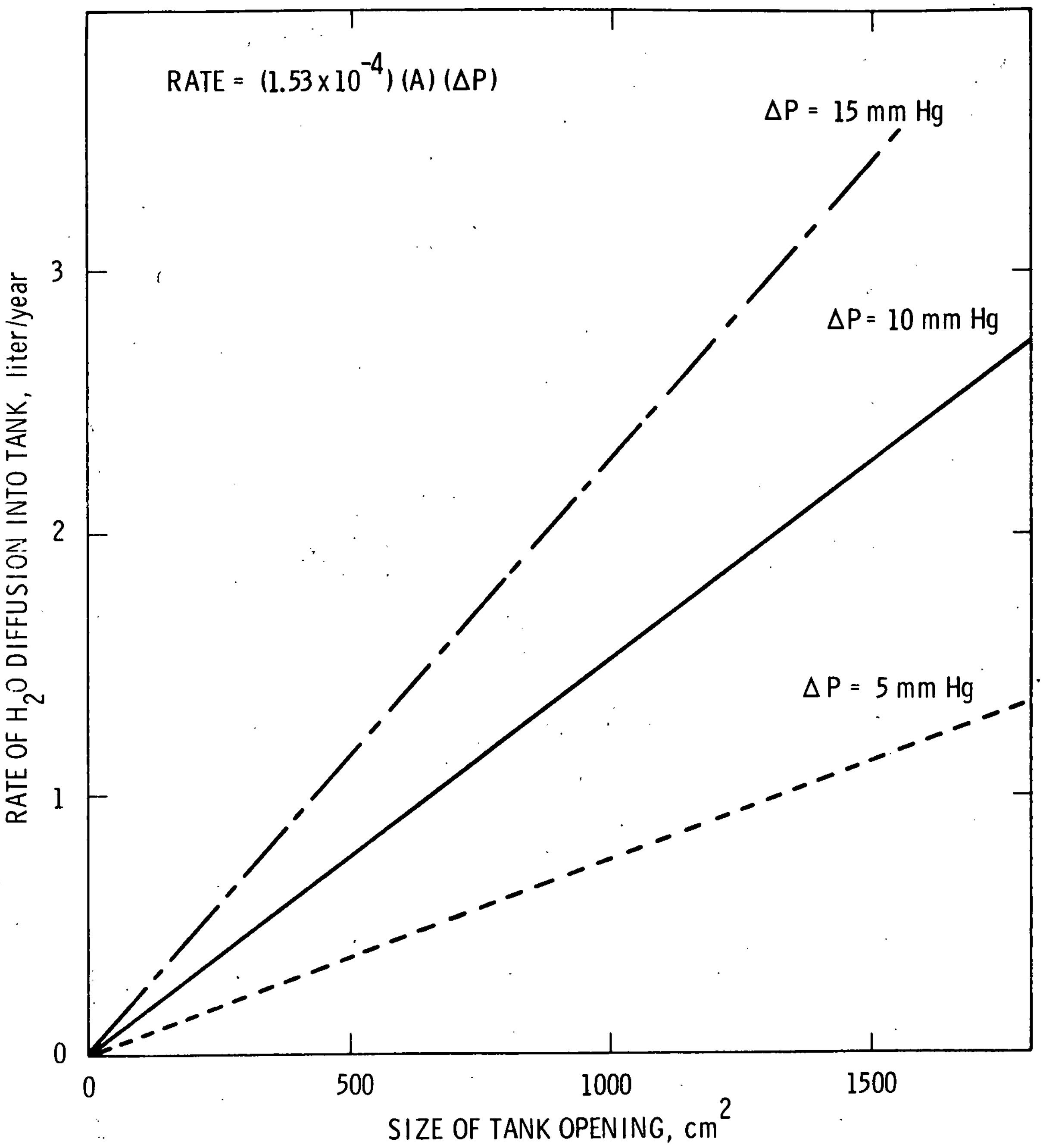

FIGURE 8

RATE OF WATER DIFFUSION INTO A WASTE TANK AS A FUNCTION OF ΔP AND AREA OF TANK OPENING

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