Phase Equilibria of Desalination Solvents: Water-NaCl-Amines

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Heterogeneous equilibrium data for water-NaCl-amine systems are presented. Solvent selectivity is correlated with salinity and the mutual solubility of the amines and water.

A SOLVENT extraction process for saline water conversion has been developed (*2*, *4*) in which water is selectively extracted from salt water to produce a solvent phase containing considerable water but little salt, and a water phase of increased salt concentration. The solvents have lower consolute temperatures with water, and the fresh water is recovered by heating the extract to a higher temperature at which the solubility is greatly reduced.

Heterogeneous equilibrium data were obtained for a variety of solvents to find those having the best selectivity and also to find the optimum extraction conditions. The best solvents are amines—principally, secondary, and tertiary amines containing 5 or 6 carbon atoms—although other amines are included in this investigation. The water solubilities of these compounds have been discussed (*1*); so the present discussion emphasizes the distribution of sodium chloride between the solvent and water phases.

EXPERIMENTAL

The concentration of amine in both phases was determined by acid titration. Chloride concentration in the water phase was measured by titration with silver nitrate, and an equivalent amount of sodium was assumed to be present. Sodium and chloride concentrations in the solvent phase were determined by using ^{22}Na or ^{36}Cl as tracers. The water content of each phase was obtained by difference. The tracer technique reduced the sample size, allowing more samples to be run at once, and speeded analysis of the solvent phase. The salt content in the solvent was frequently so low that conventional chemical analysis was difficult without larger samples and considerable sample preparation.

The heterogeneous equilibria were established in an apparatus that consisted of an ampoule holding rack that could be made to rotate in a large constant temperature bath. The equilibrium vessels were either 20- or 60-ml. rubber-capped serum ampoules. Three identical mixtures were made at each concentration—i.e., the ratios of water to solvent were the same. One was labeled with ^{22}Na, another with ^{36}Cl, and the other was unlabeled. The ampoules were placed in the rack and after equilibrium was established the rack was rotated so that the necks of the bottles were just above the surface. Samples of each phase were withdrawn through a hypodermic needle for analysis. Amine in both phases and chloride in the water phase were measured using the unlabeled samples. The sodium and chloride contents of the solvent phase were determined by placing a small weighed quantity of both phases from the ampoules labeled with ^{22}Na and ^{36}Cl in planchets which were dried with a heat lamp and counted. The sodium and chloride in the solvent were then obtained from the ratio of counts in each phase and the known salt content of the water phase. Rather erratic results were obtained in the counting, owing to geometric differences in the salt deposits in the planchets. This was countered by sticking a piece of lens paper in the bottom of the planchet to give better distribution of the solutions as they dried.

Most of the secondary and tertiary amines were synthesized for this research (*3*), or were distilled from commercial products, and were of high purity with boiling point ranges of 2° C. or less. The primary amines were all commercial products. The compound called *tert*-nonylamine is actually a mixture of 1,1,3-trimethylhexylamine and 1-*n*-propyl-1,3-dimethylbutylamine.

RESULTS

The equilibrium data for salt, water, and the various amines are given in Table I. Many of these data were taken before the counting techniques were improved, and the variation in selectivity for duplicate samples was often as high as 10 to 20%. In Table I the composition of each phase is given along with the equilibrium constant for NaCl and the respective ions. Equivalent amounts of sodium and chloride need not be present in each phase, because hydrolysis of the amine increases the degrees of freedom. The difference is slight in the water phase, which contains the bulk of the salt; but in the amine phase, it may be large. For this reason a salt content for the amine phase is not given. In calculating the water content of the solvent, a mean value of the salt was used, but it makes little difference in the water content, since the quantity of salt is so small.

The selectivity or equilibrium constant is defined on a solvent-free basis.

$$K = \left(\frac{A}{A+B}\right)_w \times \left(\frac{A+B}{A}\right)_s$$

where w and s are water and solvent phases, A = weight fraction of salt, and B = weight fraction of water. For individual ions, the salt concentration is replaced by the ion concentration. Sodium selectivity is designated by K_+ and chloride by K_-. For a one-to-one salt the activity is equal to the square root of the product of the ionic activities, so K is assumed to equal $(K_+ K_-)^{1/2}$.

For most of the amines tested the amine ions produced by hydrolysis are more soluble in the solvent than sodium ions. Accumulation of these positive ions in the amine phase increases the chloride ion to maintain equivalence with a corresponding suppression of the sodium content. In this event, the sodium selectivity will be higher than the chloride selectivity. The solubility of the amine ion in water will parallel that of the amine; for the less soluble amines, the amine ions will remain principally in the solvent, increasing the sodium and decreasing the chloride selectivity (Figure 1).

The data used in Figure 1 are those in which the salt content of the water phase was approximately 0.1%. The

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Table I. Heterogeneous Equilibrium in Amine-Water-NaCl

Solvent Phase			Water Phase					
Mole % H_2O	Weight % H_2O	Weight % solvent	Weight % H_2O	Weight % solvent	Weight % NaCl	K_s	K_w	$(K_sK_w)^{1/2}$
2-Ethylhexylamine (30° C.)								
	23.0	77.0	99.76	0.24[a]	0			
67.5	22.4	77.6	99.66	0.24	0.10	46.7	1.2	7.5
66.6	21.7	78.3	99.23	0.24	0.53	17.9	2.0	6.0
65.8	21.1	78.9	98.72	0.24	1.04	12.9	2.2	5.3
64.4	20.1	79.6	94.77	0.24	4.99	4.4	3.0	3.6
61.3	18.1	81.4	90.21	0.24	9.55	4.6	2.9	3.7
1-Methyldecylamine (30° C.)								
	23.3	76.7	99.90	0.1[a]	0			
73.8	22.8	77.2	99.78	0.1	0.12	25.6	0.35	3.0
73.0	22.1	77.9	99.38	0.1	0.52	9.0	0.80	2.7
71.9	21.2	78.7	98.87	0.1	1.03	5.6	1.26	2.7
70.0	19.7	79.9	94.78	0.1	5.12	3.1	2.06	2.5
68.6	18.7	80.4	89.68	0.1	10.22	2.6	1.97	2.3
1-Methyldodecylamine (30° C.)								
	19.4	80.6	99.90	0.1[a]	0			
71.4	18.6	81.4	99.80	0.1	0.10	25.2	0.27	2.6
70.8	18.1	81.6	99.38	0.1	0.52	8.1	0.63	2.3
70.0	17.4	82.5	98.87	0.1	1.03	4.5	0.9	2.0
68.8	16.6	82.9	94.78	0.1	5.12	2.70	1.6	2.1
67.0	15.5	83.7	89.70	0.1	10.20	2.26	1.8	2.0
1-Methyloctylamine (30° C.)								
	29.9	70.1	99.9	0.2[a]	0			
76.7	29.3	70.7	99.68	0.2	0.11	15.1	0.50	2.8
76.0	28.4	71.5	99.26	0.2	0.54	7.46	0.90	2.6
75.3	27.7	72.2	98.72	0.2	1.08	5.75	1.27	2.7
74.6	26.9	72.6	94.5	0.2	5.31	3.32	2.18	2.7
72.0	24.4	74.4	89.41	0.2	10.39	2.23	2.00	2.1
tert-Nonylamine (30° C.)								
	15.8	84.2	99.68	0.32	0			
58.2	14.9	85.1	99.58	0.32	0.10	45.3	4.7	14.6
57.5	14.5	85.5	99.18	0.32	0.50	38.1	5.7	14.7
56.2	13.9	86.1	98.68	0.31	1.01	26.9	5.9	12.6
55.0	13.3	86.6	94.72	0.23	5.05	17.4	7.4	11.3
50.1	11.2	88.7	89.77	0.17	10.06	14.7	7.8	10.7
1,1,3,3-Tetramethylbutylamine (30° C.)								
	23.6	76.4	99.20	0.80	0			
68.0	22.8	77.2	99.10	0.80	0.10	17.8	3.2	7.4
67.0	22.1	77.9	98.72	0.75	0.53	13.8	4.2	7.6
66.3	21.5	78.5	98.23	0.72	1.05	10.5	4.2	6.6
64.6	20.3	79.6	94.30	0.51	5.19	7.1	5.1	6.0
59.9	17.2	82.5	89.39	0.33	10.23	5.7	4.8	5.2
N-Ethyl-*sec*-butylamine (32° C.)								
	31.2	68.8	92.01	7.99	0			
71.2	30.6	69.4	91.99	7.90	0.11	8.2	5.9	7.0
70.7	30.1	69.9	91.72	7.80	0.48	9.1	7.8	8.4
70.1	29.5	70.5	91.32	7.63	1.05	9.4	8.0	8.7
71.0	21.8	78.1	90.53	4.50	4.97	14.8	13.5	14.1
54.3	17.4	82.5	87.71	2.65	9.64	27.8	25.1	26.4
N-Ethyl-*tert*-butylamine (40° C.)								
	42.5	57.5	89.8	10.2	0			
80.2	41.8	58.2	89.4	10.5	0.09	3.3	2.6	2.9
78.2	38.8	61.1	90.5	9.0	0.47	3.7	3.1	3.4
76.2	36.2	63.7	90.9	8.1	0.97	3.9	3.4	3.6
64.2	24.1	75.7	90.2	5.0	4.76	7.7	6.7	7.2
51.9	16.1	83.8	87.3	3.3	9.43	15.2	15.2	15.2
N-Methyl-1,3-dimethylbutylamine (30° C.)								
	28.4	71.6	98.64	1.36	0			
71.4	28.1	71.9	98.50	1.39	0.106	19.1	5.3	10.1
71.3	28.0	72.0	98.08	1.37	0.55	14.3	7.0	10.0
70.0	26.7	73.3	97.59	1.35	1.06	11.8	7.0	9.1
64.7	22.3	77.6	93.56	0.89	5.55	10.2	8.0	9.0
58.6	18.1	81.7	89.12	0.59	10.29	8.5	9.8	9.1
Diisopropylamine-Solvent/Water, ⅓ (33.5° C.)								
71.3	30.7[b]	69.3	91.9	8.1	0			
70.6	29.9	70.1	91.8	8.1	0.095	5.1	8.1	6.4
69.8	27.9	72.1	91.9	7.6	0.48	5.3	7.1	6.1
65.5	27.1	72.9	91.9	7.1	0.98	8.1	6.6	7.3
54.9	17.8	82.2	90.7	4.5	4.80	17.5	21.8	19.5
42.9	11.8	88.2	87.8	2.9	9.31	27.4	33.7	30.4

(*Continued on page 306*)

Table I. Heterogeneous Equilibrium in Amine-Water-NaCl (*Continued*)

Solvent Phase			Water Phase					
Mole % H_2O	Weight % H_2O	Weight % solvent	Weight % H_2O	Weight % solvent	Weight % NaCl	K_+	K_-	$(K_+K_-)^{1/2}$
Diisopropylamine-Solvent/Water, ⅔ (33.5° C.)								
74.8	34.5	65.5	90.8	9.2	0			
72.9	32.3	67.7	90.8	9.0	0.11	7.3	5.1	6.2
71.6	31.0	69.0	91.2	8.3	0.54	6.0	6.1	6.0
69.8	28.2	71.8	91.3	7.6	1.05	8.0	6.9	7.4
54.8	17.7	82.2	90.2	4.7	5.10	14.9	14.6	14.7
47.4	13.8	86.1	97.1	3.2	9.66	18.7	36.7	26.3
Diisopropylamine-Solvent/Water, 3/2 (33.5° C.)								
75.1	34.9	65.1	90.0	10.0	0			
73.9	33.5	66.5	90.1	9.8	0.15	8.3	5.4	6.7
70.7	30.1	69.9	90.8	8.5	0.71	7.8	5.9	6.8
68.1	27.6	72.4	90.7	7.9	1.36	8.9	7.0	7.9
54.4	17.5	82.4	89.7	4.5	5.85	6.4	17.5	10.6
44.0	12.3	87.7	86.6	2.7	10.73	40.8	23.5	31.0
N,*N*-Dimethyl-*tert*-butylamine (49° C.)								
	31.1	68.9	93.2	6.8	0			
71.2	30.6	69.4	93.2	6.7	0.096	3.9	8.8	5.9
69.2	28.6	71.4	93.3	6.2	0.49	6.5	9.5	7.9
67.0	26.6	73.4	93.2	5.8	0.98	7.1	9.7	8.3
53.2	16.8	83.2	91.5	3.7	4.77	14.7	21.2	17.6
39.7	10.5	89.5	88.5	2.3	9.23	28.6	24.7	26.6
Methyldiethylamine (52° C.)								
	37.7	62.3	87.0	13.0	0			
73.6	36.5	63.5	87.5	12.4	0.091	2.62	6.54	4.1
68.8	31.3	68.7	88.7	10.8	0.46	2.60	6.07	4.0
64.6	27.4	72.6	89.2	9.9	0.94	4.30	5.93	5.1
46.5	15.2	84.8	90.1	5.2	4.70	6.23	25.0	12.5
40.0	10.8	89.2	87.5	3.2	9.26	20.8	50.7	32.5
½ Mixture-Triethylamine/Methyldiethylamine (39.1° C.)								
	40.5	59.5	85.9	14.1	0			
75.4	37.6	62.4	86.9	13.0	0.15	4.96	3.80	4.3
70.1	31.5	68.5	89.1	10.2	0.72	5.82	5.55	5.7
64.5	26.3	73.6	89.8	8.8	1.41	6.75	7.77	7.2
47.7	15.2	84.8	89.9	4.7	5.44	14.8	19.5	17.0
35.1	9.6	90.4	87.3	2.9	9.71	70.0	29.0	45.1
½ Mixture-Triethylamine/Methyldiethylamine (40° C.)								
	35.4	64.6	83.4	16.6	0			
72.3	33.9	66.1	84.6	15.3	0.09	4.5	5.0	4.7
68.1	29.5	70.5	86.0	13.3	0.69	6.4	7.1	6.7
64.4	26.2	73.8	87.5	11.3	1.18	8.0	10.6	9.2
46.2	14.4	85.6	88.6	6.2	5.15	9.0	34.3	25.5
36.7	10.2	89.8	86.6	3.7	9.67	50.4	73.3	60.7
½ Mixture-Triethylamine/Methyldiethylamine (41° C.)								
	31.0	69.0	85.1	14.9	0			
68.3	29.7	70.3	87.8	12.1	0.11	5.7	7.3	6.5
64.0	25.9	74.1	89.4	9.9	0.65	7.6	9.2	8.4
59.7	22.5	77.5	90.4	8.3	1.33	10.3	12.2	11.2
43.2	13.0	87.0	90.0	4.8	5.24	17.1	23.1	19.9
35.6	9.8	90.2	87.3	2.9	9.77	59.0	79.0	66.5
½ Mixture-Triethylamine/Methyldiethylamine (42° C.)								
	28.0	72.0	89.0	11.0	0			
65.1	26.8	73.2	88.8	11.1	0.12	8.6	10.6	9.6
60.9	23.4	76.6	89.9	9.5	0.63	9.3	12.6	10.8
56.2	20.1	79.9	89.9	8.8	1.28	11.7	16.0	13.7
40.5	11.8	88.2	90.1	4.8	5.10	18.2	31.8	24.1
32.1	8.5	91.5	87.5	2.8	9.73	34.0	26.5	30.0

[a] Approximate values. Emulsification made analysis difficult. [b] Difference between % water in solvent phase in this set of data and next two indicates error in temperature.

ratio K_+/K_- is strongly affected by the salt content and is greatest at low salt concentration, since increasing the quantity of salt cannot produce a corresponding ionization of the amine.

Some of the deviation in Figure 1 may result fron variations in the salt content among the data points and from variation in the relative quantity of the phases. Probably random error accounts for most of the scatter. The two low points for tertiary amines come from erratic data. The low point for secondary amines was obtained with diisopropylamine and differs sharply from two other data points for the same compound. The difference in dissociation constants for primary, secondary, and tertiary amines is not sufficient to produce a marked effect on K_+/K_-.

Figures 2, 3, and 4 indicate that the principal variable affecting selectivity is mole per cent water in the solvent phase. The primary amines show a significant relation between selectivity and water phase salt content. The curves drawn through the primary amine data are somewhat idealized, since there is too much scatter to draw

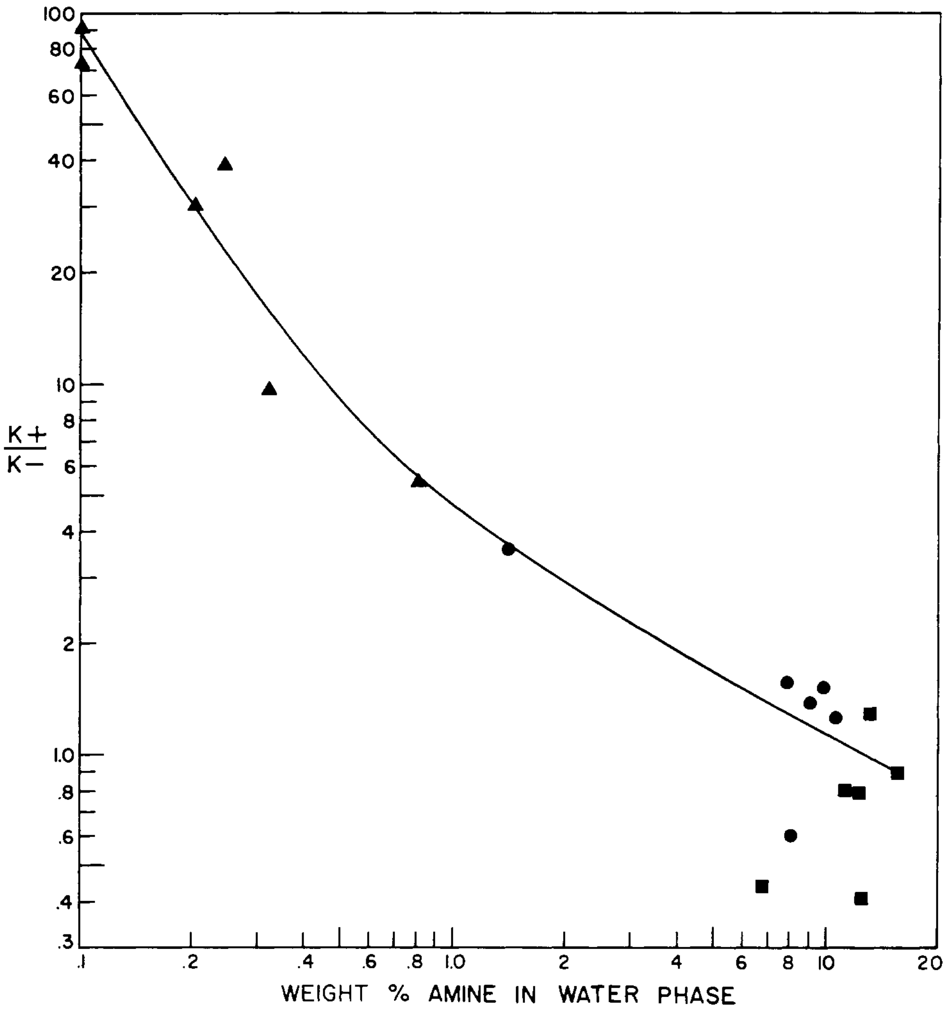

Figure 1. Effect of amine solubility in water on ratio of Na and Cl selectivities

▲ Primary amine ● Secondary amines ■ Tertiary amines

precise curves, but the trend indicated is definitely present. All curves are brought together at a common point at a selectivity of one which will correspond to the consolute composition, assuming that this composition is relatively unaffected by salt content.

The line drawn in Figure 4 was obtained from a secondary source. A desalination pilot plant was operated using various mixtures of methyldiethylamine and triethylamine over various temperature ranges. A statistical analysis of the data showed that extraction was independent of mixer speed, from which it was concluded that equilibrium was being closely approached in each stage.

An attempt was made to correlate the 283 data points by the equation

$$\log K = A + B(\text{mole \% water in amine}) + C(\text{weight \% salt in water})$$

Student's T test indicated a low significance for coefficient C and a very high significance for B. Omission of C left the mean square error and correlation coefficient unchanged. The final correlation was

$$\log K = 2.71631 - 0.0284225 \text{ mole \% water}$$

with a correlation coefficient of 0.859, a square root of mean square error of 0.149, and a standard deviation of 0.0010 for coefficient B.

Though secondary and tertiary amines do not show sufficient salt effect to justify drawing separate curves, the effect is present, but is obscured by the nature of the data. Nearly all the points corresponding to high salt content also represent low water content owing to suppression of the

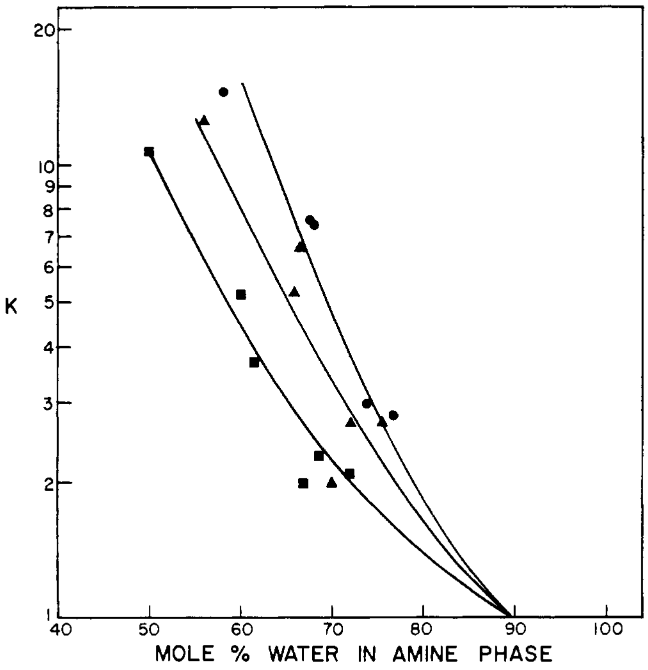

Figure 2. Selectivity of primary amines

● 0.1% NaCl

▲ 1% NaCl

■ 10% NaCl

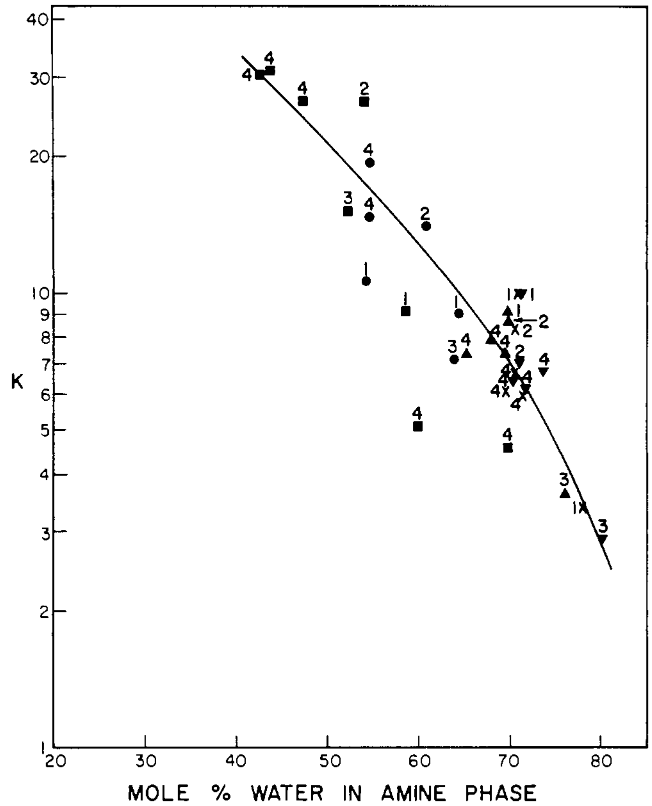

Figure 3. Selectivity of secondary amines

1. *N*-Methyl-1,3-dimethylamine
2. *N*-Ethyl-sec-butylamine
3. *N*-Ethyl-*tert*-butylamine
4. Diisopropylamine

▼ 0.1% NaCl
X 0.5% NaCl
▲ 1% NaCl
● 5% NaCl
■ 10% NaCl

Table II. Effect of Oleic and Adipic Acid on Diisopropylamine Selectivities at 33.5° C.

Weight % Acid in Amine	K_+	$(K_+K_-)^{1/2}$	K_-	Weight % H_2O in Amine	Mole % H_2O in Amine
		Oleic Acid			
0.1	5.2	5.2	5.1	34.3	74.6
0.25	3.8	4.5	5.4	36.0	76.0
0.5	2.2	3.3	4.9	36.6	76.4
1.0	1.2	2.5	5.2	40.9	76.5
		Adipic Acid			
0.1	12.5	6.9	3.8	33.2	73.7
0.25	14.9	7.1	3.4	33.4	73.9
0.5	18.5	7.2	2.8	31.0	71.6
1.0	28.0	8.4	2.5	28.5	68.9

solubility by salt, so that very little overlap in the data occurs. One secondary amine, *N*-methyl-1,3-dimethylbutylamine, has solubility characteristics similar to those of primary amines, in that the solubility of water in the compound is not so greatly reduced by salt. The data for this amine (No. 1 in Figure 3) show a trend very indicative of a salt effect. The two points for diisopropylamine on Figure 3 were obtained later using conventional chemical analysis and a temperature of only 17° C. in order to get a high water content at high salt content. The point farthest from the curve actually corresponds to over 12% salt. The data in Figure 5, which includes only results at 0.1% salt, show a trend decidedly different from the bulk trend in Figure 3.

Most of the secondary and tertiary amines included in this study have very similar water solubility curves (*4*), and

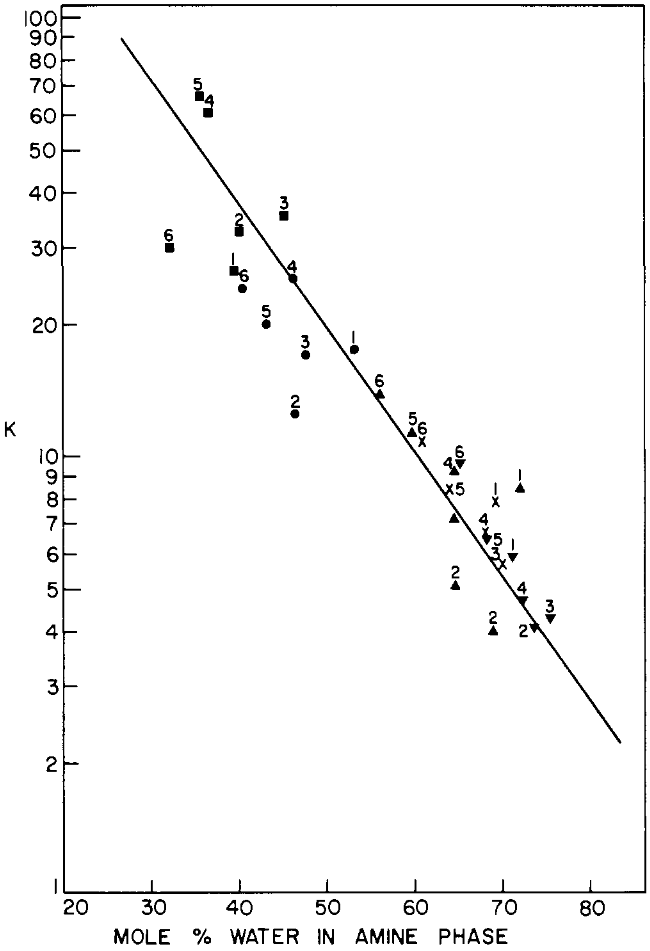

Figure 4. Selectivity of tertiary amines

1. *N,N*-Dimethyl-*tert*-butylamine
2. Methyldiethylamine

2 to 1 mixtures of methyldiethylamine/triethylamine

3. 30° C.
4. 40° C.
5. 41° C.
6. 42° C.

▼ 0.1% NaCl
X 0.5% NaCl
▲ 1% NaCl
● 5% M NaCl
■ 10% NaCl

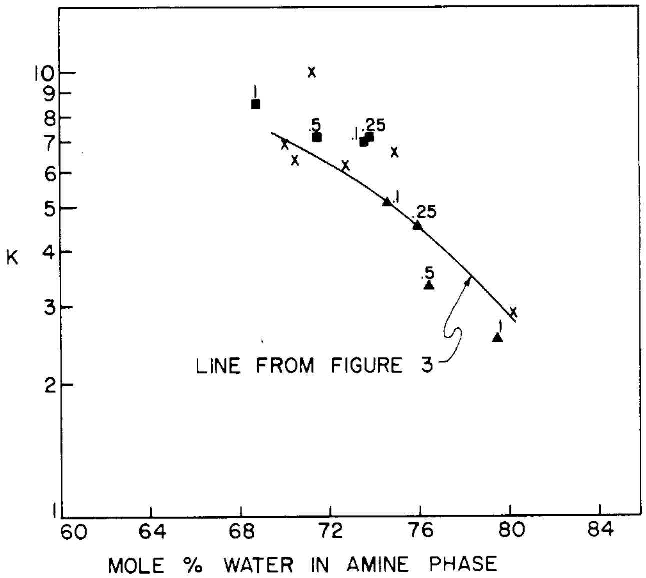

Figure 5. Effect of additions that change ionic selectivities on mean selectivity

▲ Oleic acid in diisopropylamine
■ Adipic acid in diisopropylamine
X Secondary amines, no acid

it is not surprising that they respond similarly toward salt solutions. It appears that secondary amines may be slightly superior to the tertiary amines.

Table II and Figure 5 show the results of a study which confirms the validity of the expression $K = (K_+ K_-)^{1/2}$. Gradient quantities of adipic and oleic acid were added to diisopropylamine and equilibrated with 0.1% salt solution. These acids add foreign ions to the system and increase considerably the supply of amine ions.

The adipic acid, being water-soluble, increases the supply of anions in the water phase, raising the sodium selectivity. The water content of the amine phase is reduced, probably through increase in the electrolyte content of the water phase. Oleic acid suppresses the sodium selectivity. The water content of the amine is increased, probably through solvation with the oleate ions. Although the individual ionic selectivities were affected radically, the mean selectivity was unaffected, except by the variation in water content. The results obtained with these acids agree with the other secondary amine data.

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High Temperature *PVT* Properties of Sodium, Potassium, and Cesium

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Pressure-volume-temperature properties are presented for three alkali metals—sodium, potassium, and cesium. The data for each metal cover an extended temperature range from the normal boiling point to 2550° F.

ALKALI METALS, because of their high latent heats of vaporization and heat-transfer coefficients, are prominent candidates for working fluids in turbine power converters. The selection of a particular metal will be largely determined by the maximum cycle temperature, which is limited and effectively fixed by the state of the art for turbine, reactor, and containment materials. Potassium and cesium have vapor pressure ranges which make them attractive for the current state of the art. However, as technology progresses, higher boiling metals such as sodium and lithium will be considered. To evaluate the performance of these alkali metals, a reliable body of experimentally determined physical and thermodynamic properties at proposed operating conditions is required. A program was established to determine several physical and thermodynamic properties of sodium to 2500° F., potassium to 2300° F., and cesium to 2300° F.

This article, which describes pressure-volume-temperature measurements for sodium, potassium, and cesium, is the first publication derived from this program. An ultimate objective is the derivation of reliable engineering and thermodynamic properties—enthalpy, entropy, specific heat, and specific volume. The *PVT* data reported in this article, combined with measurements now in progress of saturation pressures, liquid specific volumes, and liquid specific heats, will provide the experimental data required to obtain these properties.

EXPERIMENTAL

Apparatus and Method. A *PVT* determination requires the measurement of four variables—weight, volume, temperature, and pressure of the vapor involved. For the alkali metals all four of these had to be obtained with high accuracy in order to permit the generation of reliable thermodynamic properties. This was accomplished with a simple constant-volume apparatus using a thin metal diaphragm operating at temperature. The important variable, pressure, was read with sensors at room temperature by balancing the external gas pressure against that of the metal vapor. Temperatures were observed with thermocouples; the alkali metal charge was determined by weighing, and the volume by dimensioning. The method thus permitted the use of direct and positive techniques for obtaining the four basic measurements. The principal disadvantage was that a new or a completely reworked apparatus was required for each experiment, but even this increased the over-all accuracy by reducing systematic errors.

The apparatus, shown in Figure 1, was a small pot equipped with the pressure-sensing diaphragm and a small tube for filling, evacuating, and closing. The principle of the diaphragm design was taken from White and Hilsenrath (*5*). Although it was necessary to redesign their apparatus completely to permit high-temperature operation, the three basic criteria which they suggested for an apparatus of this type were met. The modified apparatus had high sensitivity over the entire temperature and pressure range, a reproducible null point, and a negligible change in volume upon actuation of the diaphragm.

To meet the experimental conditions imposed by temperature and containment, the entire apparatus except for the mullite insulator was fabricated from a refractory alloy, columbium-1% zirconium. This alloy, with a melting point of 4375° F. and a yield strength of around 6000 p.s.i. at 2500° F., proved to be an excellent choice. The mechanical properties of electron beam-melted material were particularly well suited to this work. The sensitivities of diaphragms made from the material were shown experimentally to remain unchanged after repeated cycling from room temperature to 2550° F. Dimensional changes of apparatus after firing were surprisingly low and generally within the expected error of the dimensioning technique. The ma-