Correlating Vapor Pressures and Heats of Solution for the Ammonium Nitrate—Water System:

An Enthalpy-Concentration Diagram

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The usual tedious method of construction of enthalpy-concentration charts for solutions of solids requires data seldom available. A new, simple method is therefore presented that utilizes more readily available data, that is vapor pressures of the solutions. These plot as straight lines on a logarithmic-reference substance plot. Differences from unity of the slopes of these lines represent heats of solution, and when this difference function is integrated between concentration limits the integral heat of solution is obtained. The enthalpy chart is then readily constructed by use of the specific heats of liquid and solid.

Ammonium nitrate is the solid used in this example, and water is the liquid because of the industrial importance of aqueous solutions. Vapor pressures were carefully determined experimentally, and the enthalpy chart was developed from the straight lines of the logarithmic plot and available heat data. Constants for the vapor-pressure curves for ammonium nitrate solutions and equations for enthalpies of solid ammonium nitrate are given for the temperature range 0° to 170°C.

With production of almost 3,000,000 tons/yr. in the United States, ammonium nitrate is of outstanding importance as a nitrogen fertilizer, more recently as an explosive, and in other major uses. Properties of the ammonium nitrate–water system are very important in the design of equipment to produce and use ammonium nitrate.

While more complex systems involving ammonium nitrate were being studied (*4*), pressures were measured above aqueous ammonium nitrate solutions; these were correlated with available vapor-pressure data as the logarithms of the vapor pressures vs. the vapor pressures of water at the same temperatures as previously described (*11*). From the slope of the lines of constant concentration, differential heats of dilution were calculated, and these were used together with integral heats of solution and heat capacity from the literature to construct an enthalpy-concentration diagram, as described by Othmer, Kowalski, and Napthali (*12*).

EXPERIMENTAL DETERMINATION OF VAPOR-PRESSURE DATA

Two ebulliometers, one containing the ammonium nitrate solution and the other pure water, were connected in parallel to a pressure system. The boiling-point difference was measured directly, and the temperature of the water was determined from the pressure of the system. Pressure readings were accurate to ± 0.1 mm. Hg and temperature readings to ± 0.05°C. Below 50°C. and above 65 wt.% ammonium nitrate excessive bumping of the liquid made measurements unreliable. The experimental data are shown in Table 1 and plotted in Figure 1 according to the method previously described (*11*), no point deviating by more than 1% from the line representing all the points.

DISCUSSION OF VAPOR-PRESSURE DATA

After completion of these determinations Campbell *et al.* (*1*) published similar vapor pressures determined by a static method. The experimental data and those of the literature (*1, 3, 8*) correlate well on the logarithmic-reference substance plot. The basic equation when vapor pressure of the solution is always taken at the same temperature as vapor pressure of water is

$$\log P = m \log P^\circ + C \qquad (1)$$

where $m = L_B/L^\circ_B$

Slopes and intercepts of the straight lines were plotted vs. concentration in Figure 2. Slopes derived from vapor-pressure data were calculated by statistical means (*16*), slopes have also been calculated from heats of dilution data (*2, 10*).

The slopes from the experimental

TABLE 1. EXPERIMENTAL PARTIAL PRESSURE OF WATER ABOVE AMMONIUM NITRATE SOLUTIONS

Concentration in wt. % NH_4NO_3

20.6		40.1		46.6		60.3	
t, °C.	P_{H_2O}, mm. Hg	t, °C.	P_{H_2O}, mm. Hg	t, °C.	P_{H_2O}, mm. Hg	t, °C.	P_{H_2O}, mm. Hg
61.8	147.0	61.6	128.0	51.4	77.4	55.5	84.6
76.3	280.3	80.1	282.9	61.0	120.6	61.1	105.0
87.6	456.8	83.7	316.3	69.9	182.4	69.5	147.1
101.9	750.3	92.6	464.4	85.4	341.2	80.8	237.0
		94.6	479.9	97.5	520.3	85.1	278.5
		98.9	567.5	98.9	540.6	93.3	378.6
		99.4	578.8	103.9	647.6	98.9	461.3
		99.7	584.4	106.4	742.9	99.3	485.3
		102.1	634.7			104.9	593.2
		105.6	751.4			106.5	632.6
		105.8	727.9			113.1	749.1
		106.6	743.1				

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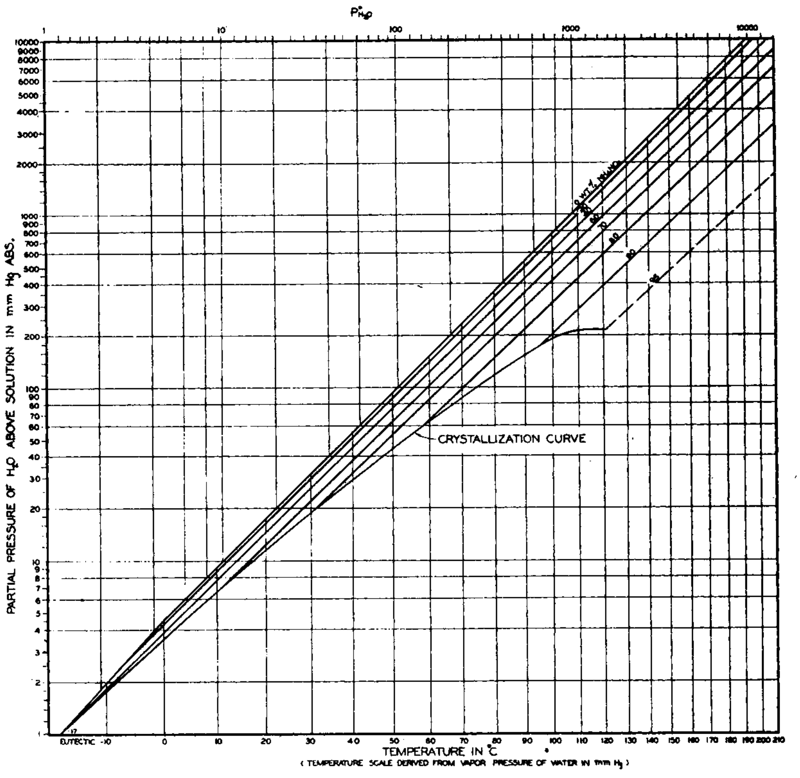

Fig. 1. Equilibrium vapor pressure of water above ammonium-nitrate solutions of constant concentration as a function of temperature.

data and those from Campbell *et al.* (*1*) for the high concentration range are within the confidence range of those calculated from heat of dilution data. Slopes from the Campbell *et al.* data for the lower concentration range are not consistent with those for high concentrations, nor with those presently found. They are disregarded in plotting the smooth curve of Figure 2*a* representing the slopes of the lines of constant composition of the best data as determined by the three indicated methods.

Constant *C* of Equation (1) was calculated for each concentration with mean values of the experimental data and the slope from Figure 2*a*. Data of Campbell *et al.* were used to establish *C* at 70, 80, and 90 wt.% ammonium nitrate. A smooth curve was drawn through the plotted points in Figure 2*b*.

Gerlach (*5, 8, 9*) gives the boiling points at 760 mm. also for 95 and 96 wt.% ammonium nitrate solutions. These may be used to estimate the pressures above the solution at concentrations higher than 90 wt.% by extrapolating the slope *m* in Figure 2*a* to the respective composition and then calculating *C* from Equation (1) to extend the correlation of Figure 2*b*. Equation (1) can then be used to calculate partial pressures at other conditions, also above 90 wt.% ammonium nitrate. However further experimental verification of the pressures for solutions higher than 90 wt.% ammonium nitrate would be desirable. Table 2 summarizes the results.

The lines of Figure 1 correlate the experimental data, according to the vapor-pressure equations given in Table 2, on a temperature scale derived from the vapor pressure of pure water. At lower temperatures the lines intersect the liquid-solid saturation line, which was derived from data taken from D'Ans-Lax (*2*). Vapor pressures along this crystallization curve determine the operating conditions for vacuum and air-stripping crystallizers for ammonium nitrate; also they define humidity conditions under which solid ammonium nitrate will absorb moisture from the air.

CONSTRUCTION OF THE ENTHALPY-CONCENTRATION DIAGRAM

The enthalpy of a solution above a fixed datum temperature for two pure components mixed together is

$$H_s = x_A H_A + x_B H_B - x_A q \qquad (2)$$

where the integral heat of solution *q*

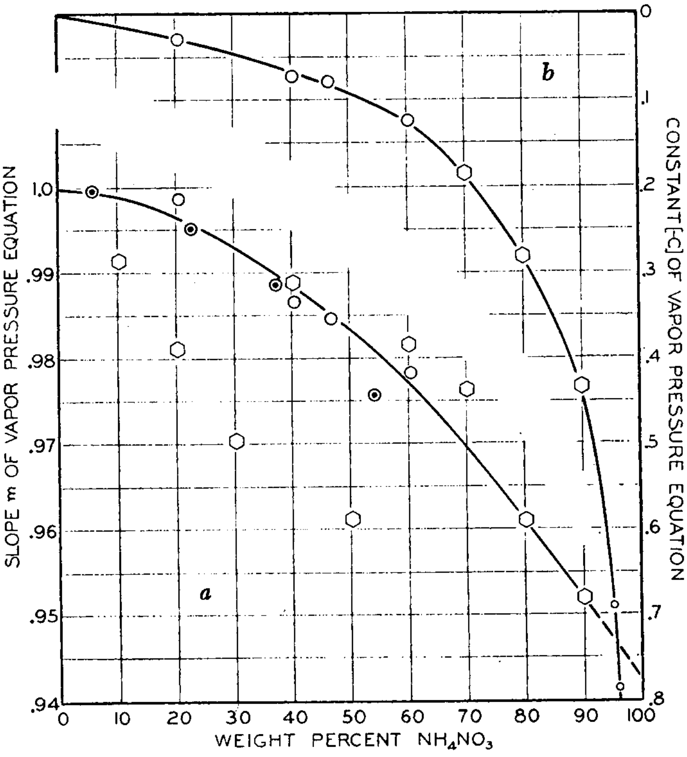

Fig. 2. The upper curve represents the intercept C of the vapor pressure Equation (1) as a function of the ammonium nitrate concentration. These values from Table 2 are all less than zero and hence are negative. The lower curve refers to the slope *m* of the vapor pressure Equation (1) as a function of the ammonium nitrate concentration. ○ This investigation, ⬡ Campbell *et al.* (*1*), ⊙ Calculated from heats of dilution (*2, 10*), ○ Gerlach (*5, 8, 9*).

TABLE 2. PARTIAL PRESSURE OF WATER ABOVE AMMONIUM NITRATE-WATER SOLUTIONS

NH_4NO_3-Concentration, wt. %	Slope of isostere in Equation (1) m	Intercept of isostere in Equation (1) C
10	0.9988	—0.0125
20	0.9968	—0.0275
30	0.9935	—0.0460
40	0.9889	—0.0670
50	0.9837	—0.0910
60	0.9770	—0.1245
70	0.9693	—0.1853
80	0.9611	—0.2829
90	0.9523	—0.4360
95	0.9475	—0.6950

can be shown to be (*12*)

$$q = L^\circ_B \int_0^{x_B/x_A} (m-1)\, d\frac{x_B}{x_A} \quad (3)$$

The reference state for a solid solute is usually the state of infinite dilution, and Equation (2) becomes

$$H_s = x_A \overline{H'}_A + x_B H_B + x_A q_\infty \quad (4)$$

Here, instead of the enthalpy of the pure component H_A, the partial enthalpy of the solute at infinite dilution $\overline{H'}_A$ has been used, and the infinite heat of solution is substituted by the infinite heat of dilution:

$$q_\infty = L^\circ_B \int_{x_B/x_A}^{\infty} (m-1)\, d\frac{x_B}{x_A} \quad (5)$$

For many systems of partial miscibility, such as salt-water systems, only Equations (4) and (5) can be used. However where vapor pressures at high concentrations can be determined, Equation (3) can be integrated at least as accurately as Equation (5); thus either may be used.

TABLE 3.

Temperature t, °C.	Enthalpies for solid NH_4NO_3 in kcal./kg.-mole ammonium nitrate derived from data of reference *8, 14* (base temperature 0°C.)
—60 to —17	$H^*_A = 3{,}890. - 3.10T - 0.0551T^2$
—17	$\Delta H_{TR} = 120$ (V → IV)
—17 to +32	$H^*_A = -5{,}580. + 9.54T + 0.0398T^2$
+32	$\Delta H_{TR} = 399.$ (IV → III)
+32 to 83	$H^*_A = -5{,}452. + 14.11T + 0.0235T^2$
+83	$\Delta H_{TR} = 311$ (III → II)
+83 to +125	$H^*_A = -9{,}696. + 27.22T$
+125	$\Delta H^*_{TR} = 1{,}027$ (II → I)
+125 to +170	$H^*_A = -10{,}840 + 27.22T$
+170	$\Delta H_{(fusion)} = 1{,}460$ (I → liquid)

Equations (2) and (3) were used in the present case. Liquid ammonium nitrate can exist only in its pure form above the melting point of 170°C. Below 170°C. the enthalpy of the molten salt in a subcooled state would be indicated and shall be designated as $\overline{H}_A$ in contrast to H^*_A.

The slope of the isosteres is a function of the concentration only and not of temperature; hence the integration of Equation (3) covers also the range of immiscibility at temperatures below the melting point of the solute.

It is necessary only that at some temperature the term $(m-1)$ can be extrapolated with reasonable accuracy to $x_B/x_A = 0$ in the same way as $(m-1)$ is extrapolated to $x_B/x_A = \infty$ when the reference state of infinite dilution for the solute is used.

The reference states of pure liquid water and of pure solid ammonium nitrate were both taken at 0°C. The integral heat of solution to dissolve solid ammonium nitrate in water isothermally at 25°C. can be taken from reference *15*. The 25°C. isotherm may therefore be used as a starting point for constructing the enthalpy-concentration diagram, although the reference temperature is 0°C.

The steam tables (*7*) give the enthalpy of pure water. Heat capacities and heat of transformations for solid ammonium nitrate have been used as shown in Table 3. Heat capacity data of Rutskov (*13*) and Gucker *et al.* (*6*) for the ammonium nitrate solutions have been used. The 25°C. isotherm is thus calculated:

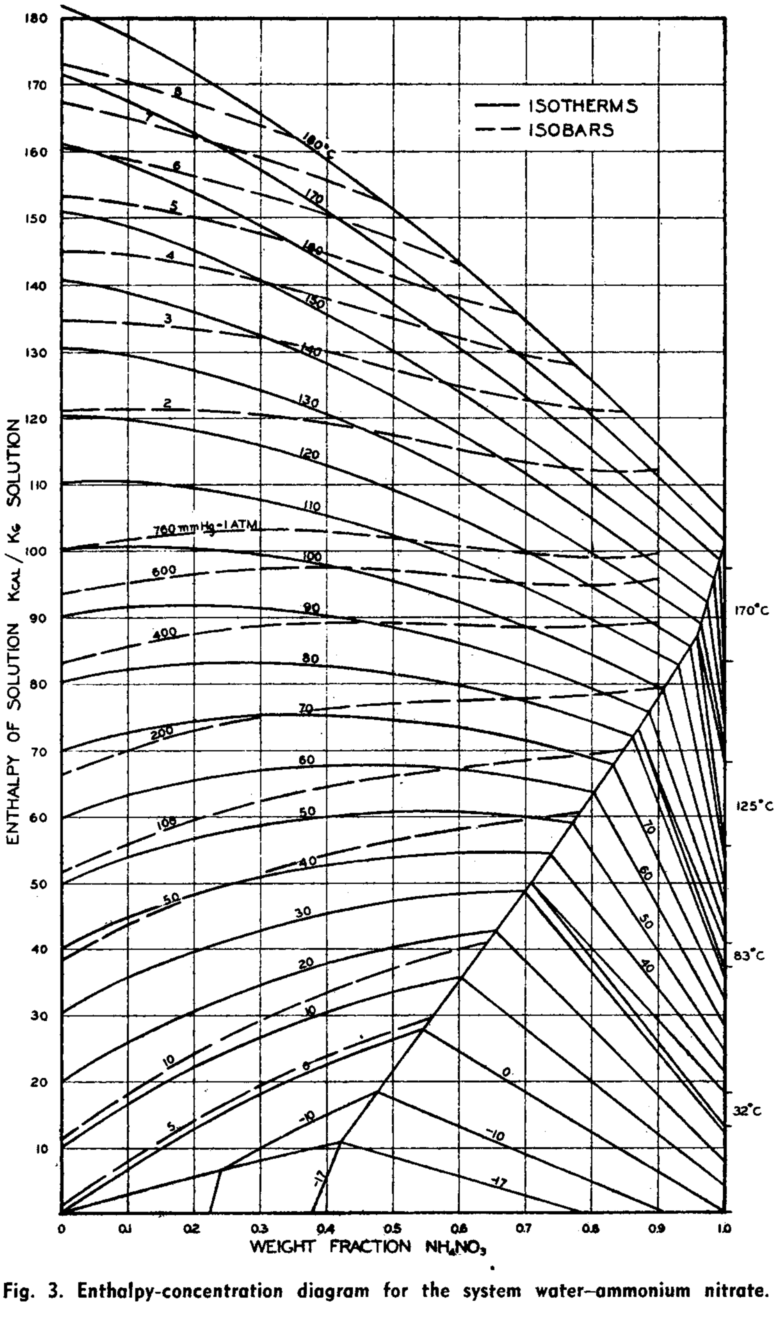

Fig. 3. Enthalpy-concentration diagram for the system water–ammonium nitrate.

TABLE 4. VALUES OF

$$\frac{x_{A}q}{L^{\circ}_{B}} = x_A \int_0^{x_B/x_A} (m-1)\, d\frac{x_B}{x_A} = x_A Z$$

Wt. % NH_4NO_3	x_AZ
2.20	0.00186
5.0	0.00378
10.0	0.00687
20.0	0.01240
30.0	0.01760
40.0	0.02195
50.0	0.02525
60.0	0.02758
70.0	0.02785
80.0	0.02530
90.0	0.01900

First the enthalpy of a 9.09 mole % solution at 25°C. is calculated:

$$H_{s_1} = x_{A_1} H^{*}_{A} + x_{B_1} H_B + x_{A_1} Q \qquad (6)$$

$$= (0.0909 \times 805) + (0.9091 \times 451) + (0.0909 \times 4{,}810)$$

$$= 921.6 \text{ kcal./kg.-mole solution}$$

$$= 38.92 \text{ kcal./kg. solution}$$

The area under the curve $(m-1)$ vs. x_B/x_A was evaluated by a step-by-step integration, and the values x_AZ were calculated (Table 4), where

$$Z = \int_0^{x_B/x_A} (m-1)\, d\frac{x_B}{x_A} \qquad (7)$$

Multiplication of Z by the molar heat of vaporization of pure water (7) gives the desired q value at any desired temperature and pressure.

The enthalpy of any solution along the 25°C. isotherm can now be calculated as

$$H_{s_2} = H_{s_1} + (x_{A_2} - x_{A_1})\, \overline{H}_A + (x_{A_1} - x_{A_2})\, H_B - x_{A_2}\, q\Big|_0^{x_{A_2}} + x_{A_1}\, q\Big|_0^{x_{A_1}} \qquad (8)$$

$$= 921.6 + (x_{A_2} - 0.0909)\, 3{,}540 - (x_{A_2} - 0.0909)\, 451 - 188.88 - x_{A_2}\, q\Big|_0^{x_{A_2}}$$

where $\overline{H}_A$ is obtained from

$$\overline{H}_A = \frac{H_{s_1} - x_{B_1} H_B + x_{A_1}\, q\Big|_0^{x_{A_1}}}{x_{A_1}} \qquad (9)$$

and specifically for 25°C.

$$\overline{H}_A = \frac{921.6 - 0.9091 \times 451 - 188.88}{0.0909}$$

$$= 3{,}540 \text{ kcal./kg.-mole ammonium nitrate} \qquad (10)$$

$$= 44.17 \text{ kcal./kg. ammonium nitrate}$$

TABLE 5. ENTHALPIES FOR THE WATER—AMMONIUM NITRATE SYSTEM

(Enthalpies in Kcal/Kg. Solution)

Temperature, °C	Concentration in Wt.% NH_4NO_3 0	10	20	30	40	50	60	70	80	90	100
-17	-17.02			4.41	9.88	14.55					-6.45
-10	-10.10		4.30	9.16	15.19	19.42					-3.81
0	0.00	7.21	13.10	18.34	22.74	26.36	29.36				0.00
10	10.06	16.54	22.26	25.52	30.30	33.30	35.70				4.01
20	20.06	25.99	30.63	34.61	37.79	40.19	41.98	43.01			7.88
30	30.04	36.24	39.34	42.71	45.27	47.06	48.27	48.70			12.24
40	40.02	44.67	48.06	50.81	52.76	53.95	54.55	54.40	53.61		21.06
50	49.99	54.01	56.76	58.90	60.24	60.82	60.83	60.09	58.72		24.67
60	59.97	63.34	65.48	66.99	67.73	67.71	67.11	65.78	63.82	61.44	28.37
70	69.98	72.72	74.22	75.11	75.22	74.60	73.40	71.48	68.93	65.97	32.10
80	80.01	82.10	82.98	83.24	82.73	81.50	79.70	77.36	74.05	70.50	35.91
90	90.07	91.51	91.75	91.39	90.25	88.40	86.01	82.89	79.17	75.03	43.33
100	100.2	100.99	100.58	99.57	97.82	95.36	92.34	88.61	84.29	79.57	46.74
110	110.3	110.43	109.38	107.75	105.37	102.29	98.66	94.34	89.41	84.10	50.13
120	120.4	119.88	118.18	115.92	112.91	109.22	104.97	100.04	94.53	88.63	53.54
130	130.6	129.40	127.06	124.14	120.50	116.18	111.32	105.78	99.67	93.17	69.78
140	140.8	138.93	135.94	132.37	128.10	123.15	117.67	111.53	104.81	97.72	73.18
150	151.0	148.46	145.81	140.61	135.70	130.11	124.02	117.26	109.86	102.25	76.58
160	161.3	158.08	153.76	148.89	143.34	137.13	130.39	123.02	115.10	106.81	79.98
170	171.6	167.69	162.71	157.19	150.98	144.14	136.78	128.78	120.24	111.35	83.29
180	181.9	177.31	171.66	165.48	158.62	151.13	143.15	134.54	125.39	115.89	105.48

Heterogeneous Area
(Figures only meaningful as aid to drawing isotherms up to the crystallization line.)

With the aid of heat capacity data for the solution, $\overline{H}_A$ is calculated at any other temperature

$$\overline{H}_A = \frac{H_{s(25)} + \overline{C_{p_s}}\,(t-25) - x_B H_B + x_A\, q\Big|_0^{x_A}}{x_A} \qquad (11)$$

At 170°C. $\overline{H}_A$ should become equal to the enthalpy of pure molten ammonium nitrate. This value can be calculated independently by using heat capacities of the solid and heats of transition and fusion for pure ammonium nitrate. Within the accuracy of the heat-capacity data for solid ammonium nitrate and for the solutions there is calculated

$$\overline{H}_A = 2{,}744 + 31.67t \quad \text{(kcal./kg.-mole)}$$

With the above information the complete enthalpy-concentration diagram was calculated as shown in Figure 3. In addition to the isotherms, isobars for concentrations up to 90 wt.% ammonium nitrate were calculated from the equation of Table 2 and plotted in the enthalpy-concentration diagram. Tables 5 and 6 show the values of enthalpy along isotherms and isobars respectively; by use of these numbers, for more precise use, there may be prepared large-scale plots.

ACKNOWLEDGMENT

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NOTATION

C = integration constant (intercept of partial-pressure equation)

$\overline{C_{p_s}}$ = mean molar heat capacity for ammonium nitrate solutions, kcal./kg.-mole solution and °C.

TABLE 6. ISOBARS FOR THE WATER—AMMONIUM NITRATE SYSTEM
(VALUES GIVEN ARE OF TEMPERATURES IN °C.)

Pressure mm.Hg.	Concentration in Wt. % NH_4NO_3 0	10	20	30	40	50	60	70	80	90
5	1.2	1.3	1.4	1.5	1.7	1.9				
10	11.3	11.7	12.3	13.1	14.0	15.1	16.6			
50	38.1	38.8	39.6	40.6	41.9	43.4	45.4	49.0		
100	51.6	52.3	53.2	54.4	55.9	57.6	60.0	64.0	70.4	
200	66.5	67.2	68.3	69.8	71.4	73.4	76.2	80.8	88.0	99.6
300	75.9	76.7	77.9	79.4	81.2	83.4	86.6	91.6	99.4	112.0
400	83.0	83.9	85.1	86.6	88.7	91.0	94.3	99.7	108.0	121.5
500	88.7	89.6	90.9	92.6	94.7	97.1	100.6	106.2	115.0	128.9
600	93.6	94.5	95.8	97.6	99.8	102.3	105.9	111.8	120.9	135.6
700	97.7	98.8	100.1	101.9	104.2	106.8	110.6	116.7	126.0	141.0
(atms.)										
1	100.0	101.0	102.5	104.2	106.6	109.3	113.1	119.3	128.9	144.1
1.25	106.6	107.2	108.9	110.8	113.3	116.2	120.2	126.7	136.7	152.8
1.5	111.8	112.9	114.4	116.4	119.0	122.0	126.1	132.9	143.4	160.0
2.0	120.7	121.9	123.4	125.6	128.3	131.6	136.1	143.4	154.6	172.4
2.5	127.9	129.1	130.8	133.1	136.0	139.4	144.1	151.8	163.6	182.5
3.0	134.0	135.3	137.0	139.4	142.5	146.0	151.0	159.0	171.4	191.1
3.5	139.3	140.7	142.5	144.9	148.1	151.8	157.0	165.4	178.2	198.6
4.0	144.1	145.5	147.3	149.9	153.2	157.0	162.4	171.0	184.3	
4.5	148.4	149.8	151.7	154.4	157.8	161.7	167.2	175.9	189.8	
5.0	152.3	153.8	155.7	158.5	162.0	166.0	171.7	180.8		
6.0	159.2	160.9	162.9	165.8	169.4	173.7	179.6	189.2		
7.0	166.0	167.1	169.2	172.1	175.9	180.4	186.6	196.6		
8.0	171.8	172.6	174.8	177.9	181.8	186.4	192.8			

Temperatures in degrees centigrade.

L°_B = molar heat of vaporization for pure water, kcal./kg.-mole water
L_B = molar heat of vaporization for water out of ammonium nitrate solutions, kcal./kg.-mole water
m = slope of an isostere on a logarithmic partial-pressure plot
H = enthalpy, kcal./kg-mole (except if otherwise noted)
P = partial pressure of water above ammonium nitrate solutions, mm. Hg abs.
P° = vapor pressure of pure water, mm. Hg abs.
Q = integral heat of solution for solid ammonium nitrate, kcal./kg.-mole
q = integral heat of dilution, kcal./kg.-mole
t = temperature, °C.
T = absolute temperature, °K.
x = mole fraction
Z = abbreviation for the integral of Equation (7)

Subscripts

A = ammonium nitrate
B = water
s = solution
TR = transition
∞ = infinite dilution
1 = concentration where integral heat of solution Q is known
2 = any concentration

Superscripts

$-$ = partial quantity
$'$ = infinite dilution
$\bullet$ = solid state
$\circ$ = pure compound

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Hydrocarbon Vapor-Liquid Equilibria and Solubility Parameter

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Hydrocarbon vapor-liquid equilibria are expressed in terms of K values, which are functions of composition, as well as pressure and temperature. The composition effect in the liquid phase is calculated by the Hildebrand-Scatchard equation for regular solutions. The parameters in this equation, called *solubility parameters*, can be calculated simply from heat of vaporization for the heavier hydrocarbons, but an indirect method of calculation must be used for the lighter components. Solubility parameters for hydrogen, methane, ethane, and propane were computed from gas-solubility data in several hydrocarbon solvents at various temperatures and pressures. This computation also yielded simultaneously the fugacities of the hypothetical liquid-standard states.

The results presented are not complete for practical applications, owing to the scarcity of suitable solubility data, especially at high temperatures and pressures. However solubility parameters appear to give the right liquid-phase corrections in the correlation and prediction of hydrocarbon phase equilibria. Calculated K values for light hydrocarbons in paraffinic, naphthenic, and aromatic absorption oils are compared with experimental results. The average deviation for the forty-two values tested is 13%.

The composition dependence of the vaporization equilibrium ratio in hydrocarbon systems has been frequently neglected in practical applications. While the composition-independent K values are approximately applicable to mixtures composed entirely of one class of hydrocarbons, like the aliphatics, large deviations are encountered for mixtures composed of different classes of hydrocarbons, notably those containing aromatics and, to a lesser extent, naphthenes.

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