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Liquid and Vapor Phase Enthalpy of Monomethylamine

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Enthalpy data have been obtained for liquid and vapor monomethylamine in the range of approximately 75° to 225° F. and pressures from 50 to 650 p.s.i.a. The data obtained are believed to be accurate to within ±1% of the actual enthalpy change relative to a base temperature of 75° F. The flow calorimeter used to make the measurements consists of a closed system in which the test fluid is recirculated at a constant and measurable rate and constant pressure through the calorimeter. A calorimeter fluid used to cool the test fluid is evaporated in the process. From the rate of evaporation of the calorimeter fluid and its known latent heat of vaporization, the enthalpy change of the test fluid can be calculated.

IN THE application of gas absorption and separation, data are required on specific heats of liquids and vapors and heats of vaporization, which sometimes cover wide ranges of temperature and pressure. Specifically, consideration of monomethylamine as a refrigerant in an absorption-refrigeration system requires knowledge of these properties in the temperature region of approximately 40° to 250° F. and pressures of 50 to 500 p.s.i.

Specific heat data available in the literature cover temperatures only to approximately 120° F. and for the saturated states. This paper presents data on the heat capacity of monomethylamine liquid and vapor at temperatures from 75° to 225° F. and pressures from 50 to 650 p.s.i.a. The experimental equipment is described also.

EXPERIMENTAL EQUIPMENT

The experimental technique employed and the principle of operation of the calorimeter have been described in part (*6*) and are patterned after those of Storvick (*7*). A schematic diagram of the flow system and essential pieces of equipment is given in Figure 1.

The adiabatic flow calorimeter is the most important item in the apparatus (Figure 2). The function of the calorimeter is to transfer to the boiling Freon (Freon 11–trichloromonofluoromethane, the calorimeter fluid) an amount of heat exactly equal to the decrease in energy of the methylamine that passes through the calorimeter coil. The amount of energy transferred is related to the quantity of vaporized Freon which is collected, condensed, and measured. With knowledge of the heat of vaporization of Freon, the enthalpy change of the methylamine between the temperatures at the

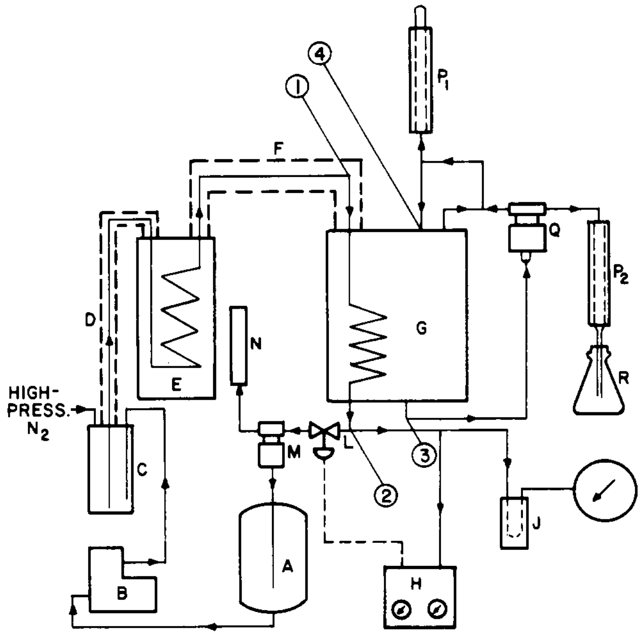
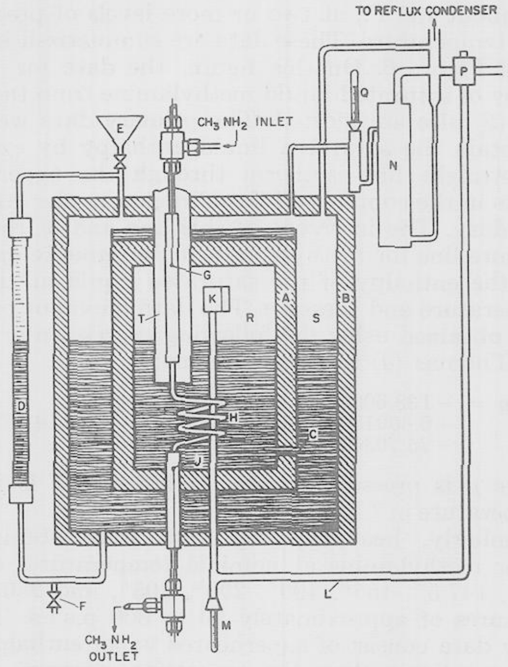


Figure 1. Schematic diagram of calorimeter unit



A	INNER CAN	K	FREON ENTRAINMENT SEPARATOR
B	OUTER CAN	L	FREON VAPOR-REMOVAL LINE
C	FREON PORT	M	FREON VAPOR THERMOMETER
D	FREON LEVEL GAGE	N	FREON LIQUID PREHEATER
E	FREON RETURN FUNNEL	P	FREON VAPOR SOLENOID VALVE
F	FREON DRAIN LINE	Q	REFLUX FREON THERMOMETER
G	INLET CH_3NH_2 THERMOCOUPLE	R	INNER VAPOR SPACE
H	CH_3NH_2 COOLING COIL	S	OUTER VAPOR SPACE
J	OUTLET CH_3NH_2 THERMOCOUPLE	T	HEAT SHIELD

Figure 2. The calorimeter

calorimeter inlet and outlet, at constant pressure, can be calculated. The remainder of the equipment is necessary to operate the calorimeter at the desired levels of temperature and pressure.

Liquid methylamine leaves the slightly pressurized feed tank (Figure 1, *A*) and enters the high-pressure pump, *B*, which forces the fluid into the pressurized surge tank, *C*. The fluid then passes through the preheater, *D*, and enters the constant temperature bath, *E*, where it is heated to the desired temperature. The shroud heater, *F*, maintains this temperature until the methylamine enters the calorimeter, *G*. Thermocouples 1 and 2 measure the temperature of the methylamine as it enters and leaves the inner chamber of the calorimeter. The methylamine pressure is measured through a mercury tube, *J*, by a gage, *K*. A pressure controller, *H*, operates a microflow control valve, *L*, which allows the methylamine to discharge at feed tank pressure. The methylamine then enters the solenoid, *M*, and is either returned to feed tank *A* or, during an actual run, enters the collecting bomb, *N*.

The Freon evaporated in the inner calorimeter chamber flows through solenoid *Q* to the reflux condenser, P_1, and returns to the calorimeter or, during an actual run, to condenser P_2 and subsequently to a collection bottle, *R*. Thermometers 3 and 4 measure the temperatures of the outgoing and returning Freon, respectively.

During an experimental run, both solenoids, *Q* and *M*, are activated simultaneously by a timer to allow an amount of the methylamine and a corresponding amount of Freon to be collected during a given time interval.

EXPERIMENTAL PROCEDURES

The calorimeter is designed so that the transfer of energy from the methylamine to the calorimeter fluid approaches adiabatic, and will occur only in the inner calorimeter chamber, can *A*. This is accomplished by the double-walled, insulated construction of the inner can, by the pool of boiling Freon maintained on top of this can, and by the presence of a quantity of Freon between cans *A* and *B*, all of which minimize heat leaks to or from an outside source.

This calorimeter will operate properly only when the room temperature is equal to or above the normal boiling point of Freon. If the room temperature is lower, there would be a net heat flux out from can *A*, giving correspondingly low values of enthalpy change for the methylamine. Further, the Freon liquid passing into can *A* from can *B* would be below its normal boiling point and would require some added heat to bring it to the boiling point. This also would give low readings.

Freon 11 was chosen as the calorimeter fluid because it has these properties: It has a relatively low heat of vaporization, 78.4 B.t.u./lb.; it can be water-condensed because its normal boiling point is 74.6° F., and the latent heat of vaporization is known very accurately.

Calorimeter Calibration and Operation. The pressure-measuring Heise gage of the unit was calibrated to ±1 p.s.i.a. by means of a deadweight tester. All thermocouples used were calibrated to ±0.1° F. for temperatures of 70° to 455° F. with a platinum resistance thermometer calibrated by the U. S. National Bureau of Standards.

The pressure drop in the calorimeter was about 1.5 p.s.i.a. for a nominal 500-p.s.i.a. system pressure. Figure 3 shows the relative upstream and downstream pressure variations relative to time.

A cycling effect of the back-pressure controller caused the flow rate and temperature to follow a cycle as well. At the peak of the pressure cycle, the increased signal pressure would close the relief valve. Consequently, there would be a very brief decrease in the flow rate. This would cause a temperature drop in the entering hot stream because a constant heat flux exists between the incoming stream and its surrounding insulated path. This is demonstrated in Figure 4.

The best method of operation was close observation of the temperature after the system had achieved a steady state, and removal of the samples of Freon and water during the constant temperature period of the cycle. This corresponds to the region indicated as stable on Figure 4.

In addition to calibration of the Heise gage and inlet and outlet thermocouples, the calorimeter was calibrated for heat leaks by running water through it, measuring the specific heat difference between inlet and outlet conditions, and comparing this to known values from the tables of Keenan and Keyes (*5*). The influence upon the accuracy of the data of the following factors was determined:

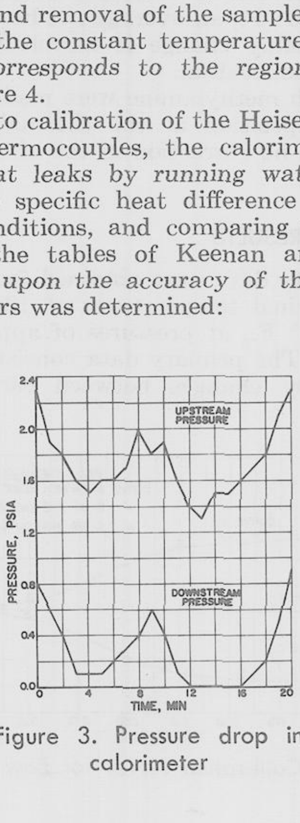


Figure 3. Pressure drop in calorimeter

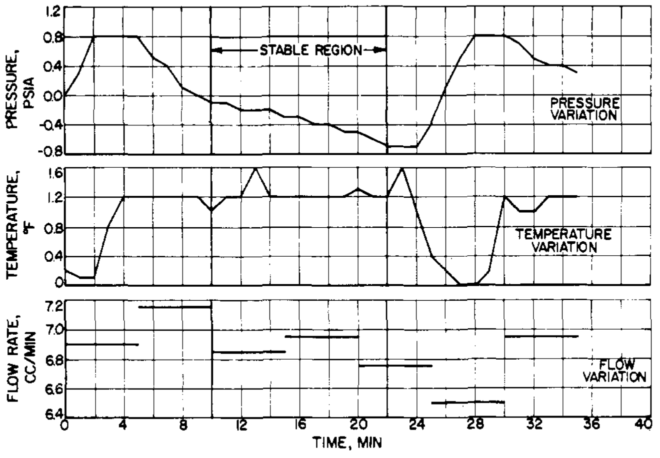


Figure 4. Pressure, temperature, and flow variations in the calorimeter

Fluctuations of the Freon level in the calorimeter inner chamber.

Fluctuations of the methylamine flow rate through the calorimeter.

Temperature fluctuations of the hot methylamine entering the calorimeter.

Location of the inlet thermocouple.

Of these four factors, only the last affected the accuracy of the results appreciably. A number of runs were made in which the inlet thermocouple was positioned $^1/_4$ inch inside, $^1/_4$ inch outside, and at the entrance to inner space *R* (Figure 2). The results obtained are shown in Figure 5. Curve *A* (Figure 5) represents the data when the inner thermocouple was $^1/_4$ inch inside space *R*, curve *B* at the entrance to space *R*, and curve *C* at $^1/_4$ inch outside area *R* and into insulated area *A*. Figure 5 shows that the thermal center of the inlet area lies about $^1/_4$ inch inside the space *R*. This is believed to be due to the requirements of the geometry of the inlet area. Figure 5 also shows that, for sensible heat losses of the inlet stream corresponding to temperatures higher than 300° F., an average net heat flux of 2% of the total enthalpy change is established irrespective of thermocouple location.

All runs with methylamine were made with the inlet thermocouple position at the thermal center of the inlet area, and no correction of the obtained data was applied.

EXPERIMENTAL RESULTS

Heat capacity data were obtained for liquid methylamine at nominal temperatures of 125°, 150°, 175°, 195°, and 225° F., at pressures of approximately 300 to 650 p.s.i.a. The primary data consist of compressed liquid enthalpy changes between each temperature and about 75° F., at two or more levels of pressure at each temperature. These data are summarized in Table I and Figure 6. On this figure, the data for the enthalpy of saturated liquid methylamine from the literature (*2*) also are plotted. The primary data were used to obtain the saturated liquid enthalpy by extending the straight line isotherm through the experimental points in the compressed liquid region to the saturation boundary. The intercept of this line and of the vapor pressure line for methylamine at the same temperature was the enthalpy of the saturated liquid at the same temperature and pressure. The data on vapor pressures were obtained using the following expression of Felsing and Thomas (*4*) for methylamine:

$$\log_{10} p = -138.60647/T + 38.730167 \log T - 6.600156 \times 10^{-2} T + 3.870056 \times 10^{-5} T^2 - 75.7030015 \qquad (1)$$

where *p* is pressure in mm. of Hg and *T* is absolute temperature in ° K.

Similarly, heat capacity data were obtained for vapor methylamine at nominal temperatures of 120°, 140°, 147.5°, 155°, 190°, 200°, 205°, and 230° F., at pressures of approximately 90 to 300 p.s.i.a. The primary data consist of superheated vapor enthalpies at a pressure lower than the saturation pressure at each temperature. These data are summarized in Table II and Figure 7. On this figure, the data for the enthalpy of saturated methylamine vapor from the literature (*2*) also are plotted. The saturated vapor enthalpy was obtained by drawing a straight line through the experimental points at each temperature and the atmospheric pressure enthalpy at that temperature. The intercept of this line and of the vapor pressure line at the same temperature is the enthalpy of the saturated vapor at the same temperature and pressure. The ideal or atmospheric pressure enthalpy for methylamine at

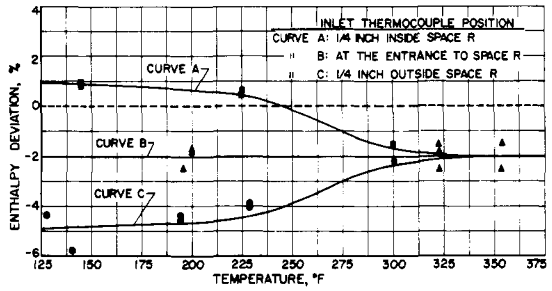


Figure 5. Calibration curves for flow calorimeter

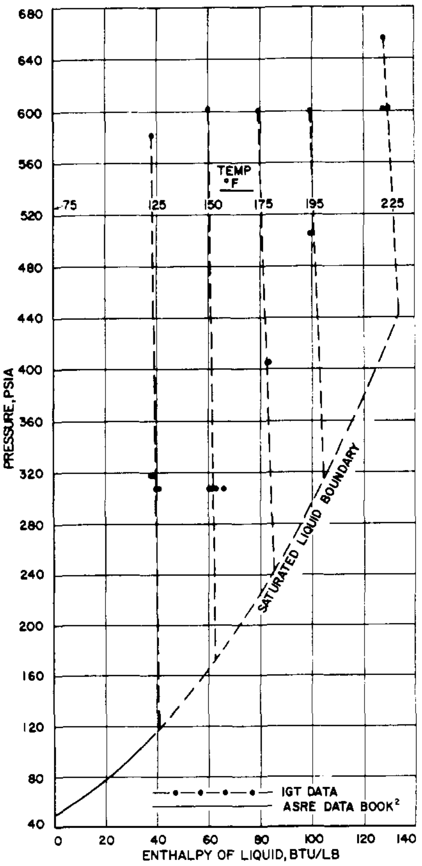


Figure 6. Experimental enthalpy data for liquid methylamine

Table I. Experimental Liquid Enthalpy of Methylamine between 120° and 225° F.

	Freon[a]		Monomethylamine								
			Temperature			Inlet Pressure		Enthalpy Change		Adj.	Adj.
Run No.	Boiling Point, t_{Fr}, ° F.	Weight, W_{Fr}, grams	Inlet, t_i, ° F.	Outlet, t_o, ° F.	Weight, W_m, grams	Exp., p_i, p.s.i.a.	Sat. at t_i, p_s, p.s.i.a.	Measured, ΔH_m,[b] B.t.u./lb.	Corrected $c\Delta H_m$,[c] B.t.u./lb.	Inlet Temp., t_a, ° F.	Enth. Change $a\Delta H_m$,[d] B.t.u./lb.
35	75.2	58.5	126.5	75.2	118.0	582	117	38.87	39.0	125	37.9
36	75.2	58.7	126.6	75.2	116.1	582	117	39.57	39.7	125	38.5
37	75.2	66.3	127.1	75.2	128.8	317	119	40.36	40.5	125	38.9
38	75.2	64.4	127.2	75.2	126.9	317	119	39.79	39.9	125	38.2
39	74.9	61.7	124.0	74.9	120.1	307	113	40.28	40.2	125	41.0
40	75.1	62.3	124.8	75.1	122.8	307	115	39.77	39.8	125	40.0
41	75.0	92.6	153.1	75.0	105.2	307	176	69.01	69.0	150	66.3
42	75.0	85.9	153.6	75.0	102.5	307	177	65.70	65.7	150	62.7
43	74.9	101.9	150.6	74.9	128.9	307	169	61.98	61.9	150	61.4
44	75.0	102.9	150.7	75.0	129.8	307	169	62.15	62.2	150	61.6
45	75.0	93.5	151.0	75.0	119.1	602	170	61.55	61.6	150	60.7
46	75.0	92.9	151.4	75.0	119.1	602	171	61.15	61.2	150	60.0
47	75.2	115.8	175.7	75.2	113.3	601	242	80.13	80.3	175	79.7
48	75.3	119.9	176.7	75.3	115.9	601	246	81.11	81.3	175	80.0
49	74.8	136.7	177.8	74.8	123.8	405	249	86.57	86.4	175	84.0
50	74.8	137.5	178.1	74.8	125.3	405	250	86.04	85.9	175	83.3
51	75.0	151.4	195.9	75.0	117.6	505	317	100.93	100.9	195	100.2
52	75.0	151.8	195.8	75.0	118.4	505	317	100.52	100.5	195	99.9
53	74.8	145.9	196.3	74.8	112.9	602	319	101.31	101.1	195	100.1
54	74.8	147.9	196.9	74.8	113.5	602	321	102.16	102.0	195	100.4
55	74.6	191.6	225.2	74.6	115.0	602	454	130.62	130.3	225	130.1
56	74.5	187.9	224.8	74.5	114.9	602	451	128.21	127.8	225	128.0
57	74.3	189.2	225.5	74.3	114.3	656	455	129.77	129.2	225	128.7
58	74.3	186.8	225.4	74.3	114.0	656	455	128.47	127.9	225	127.4

[a] Heat of vaporization (*1*), h_{Fr}, = 78.4 B.t.u./lb. for all runs.
[b] $\Delta H_m = (W_{\mathrm{Fr}}/W_m)h_{\mathrm{Fr}}$.
[c] $c\Delta H_m = \Delta H_m(t_i - 75)/(t_i - t_o) \rightarrow$ adjusted for $t_o = 75°$ F.
[d] $a\Delta H_m = \Delta H_m(t_a - 75)/(t_i - t_o) \rightarrow$ adjusted for $t_i = t_a$ and $t_o = 75°$ F.

Table II. Experimental Vapor Enthalpy of Methylamine between 120° and 230° F.

	Freon[a]		Monomethylamine								
			Temperature			Inlet Pressure		Enthalpy Change		Adj.	Adj.
Run No.	Boiling Point, t_{Fr}, ° F.	Weight, W_{Fr}, grams	Inlet, t_i, ° F.	Outlet, t_o, ° F.	Weight, W_m, grams	Exp., p_i, p.s.i.a.	Sat. at t_i, p_s, p.s.i.a.	Measured ΔH_m,[b] B.t.u./lb.	Corrected $c\Delta H_m$,[c] B.t.u./lb.	Inlet Temp., t_a, ° F.	Enth. Change, ΔH_m,[d] B.t.u./lb.
61	74.5	244.1	120.6	74.5	55.0	89	107	347.95	344.0	120	339.6
62	74.6	237.4	119.3	74.6	54.9	89	107	339.02	336.0	120	341.3
63	74.4	262.2	148.0	74.4	56.9	87	163	361.27	358.3	147.5	355.9
64	74.3	269.3	147.4	74.3	58.9	87	162	358.45	355.0	147.5	355.5
65	74.0	272.9	139.4	74.0	60.5	132	144	353.64	348.2	140	351.5
66	74.1	270.2	139.8	74.1	59.7	132	145	354.83	349.8	140	351.0
67	74.4	267.2	153.9	74.4	57.4	138	178	364.95	362.3	155	367.2
68	74.4	271.8	155.5	74.4	59.2	138	182	359.95	357.3	155	355.1
71	74.6	297.0	188.8	74.6	62.4	208	289	373.15	371.9	190	375.8
72	74.6	300.1	190.2	74.6	63.5	208	295	370.51	369.2	190	368.6
75	74.4	284.4	203.4	74.4	60.3	307	349	369.77	368.1	205	372.6
76	74.8	275.5	204.4	74.8	57.6	307	353	374.88	374.3	205	276.0
79	74.0	223.1	229.3	74.0	44.2	213	477	395.72	393.2	230	395.0
80	74.0	222.9	230.7	74.0	44.3	213	485	394.48	392.0	230	390.2
82	74.0	193.6	198.3	74.0	42.0	287	327	361.38	358.4	200	363.4

[a] Heat of vaporization (*1*), h_{Fr}, = 78.4 B.t.u./lb. for all runs.
[b] $\Delta H_m = (W_{\mathrm{Fr}}/W_m)h_{\mathrm{Fr}}$
[c] $c\Delta H_m = \Delta H_m(t_i - 75)/(t_i - t_o) \rightarrow$ adjusted for $t_o = 75°$ F.
[d] $a\Delta H_m = \Delta H_m(t_a - 75)/(t_i - t_o) \rightarrow$ adjusted for $t_i = t_a$ and $t_o = 75°$ F.

various temperatures was obtained using the following expression of Felsing and Jessen (*3*):

$$\tilde{C}p = -9.530 + 0.1108T - 1.212\ 10^{-4}T^2 \qquad (2)$$

where $\tilde{C}p$ represents the atmospheric pressure vapor heat capacity in gram-cal./mole-° C., and T the absolute temperature in ° K.

The smoothed data for the saturated phases as well as the vapor pressure and the heat of vaporization of methylamine between 75° and 225° F. are presented in Table III.

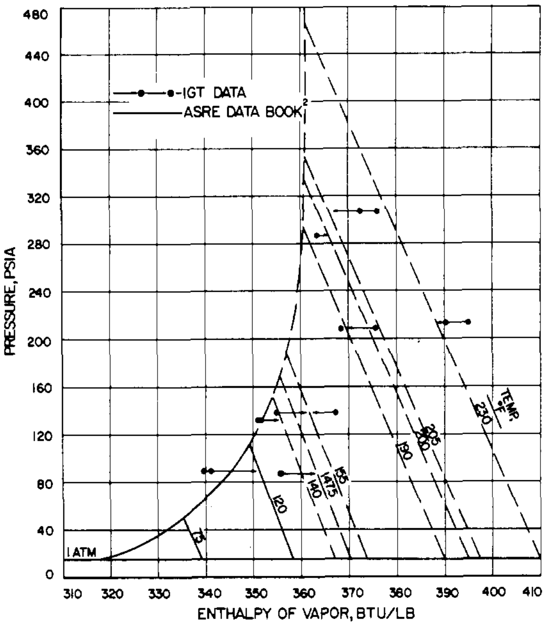


Figure 7. Experimental enthalpy data for saturated methylamine vapor

Figure 8 shows a pressure-enthalpy or Mollier chart for methylamine constructed with the data appearing in Table III with data from the literature (*2*, *3*, *4*).

Table III. Thermodynamic Properties of Saturated Methylamine

Temp. °F.	Pressure (*4*), p.s.i.a.	Enthalpy Liquid, B.t.u./lb.	Enthalpy Vapor, B.t.u./lb.	Latent Heat, B.t.u./lb.
75	50.3	0.0	335.5	335.5
100	79.9	20.1	343.4	323.3
120	111.0	36.9	349.3	312.4
125	120.2	41.3	350.6	309.3
130	129.9	45.3	351.8	306.5
135	140.2	49.7	353.0	303.3
140	150.7	54.3	354.2	299.9
145	162.2	58.6	355.2	296.6
150	174.4	63.1	356.2	293.1
155	186.7	67.5	357.1	289.6
160	200.1	72.1	357.9	285.8
165	214.2	76.6	358.6	282.0
170	229.0	81.1	359.1	278.0
175	244.5	85.8	359.6	273.8
180	260.8	90.5	360.0	269.5
185	277.3	95.3	360.3	265.0
190	295.1	99.9	360.5	260.6
195	313.8	104.8	360.8	256.0
200	332.6	109.7	360.9	251.2
205	352.9	114.5	361.0	246.5
210	374.2	119.4	361.0	241.6
215	396.4	124.3	361.0	236.7
220	419.5	129.3	361.0	231.7
225	442.7	134.3	361.0	226.7

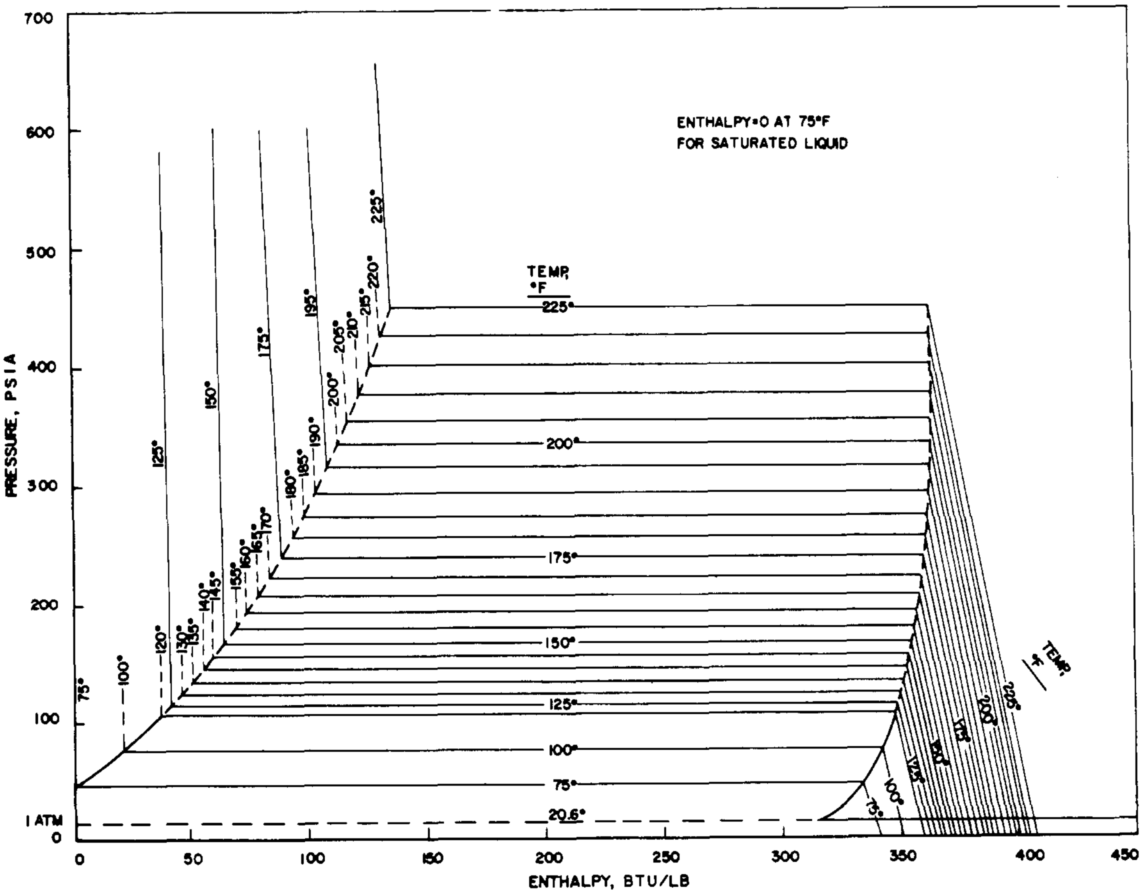


Figure 8. Mollier chart for monomethylamine

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Ternary Systems: Water-Acetonitrile-Salts

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Salting-out characteristics have been determined for the fluoride, chloride, iodide, bromide, acetate, carbonate, sulfate, and nitrate salts of lithium. Binodal curves and tie-line data have been obtained at 25° C. for ternary systems of water-acetonitrile and potassium acetate, lithium acetate, lithium chloride, and lithium sulfate. Various tie-line correlations and methods of determining plait points have been applied to these systems. A novel method is proposed for plait point determination.

IN TWO preceding papers (*8, 9*) the authors have presented the salting-out characteristics of acetonitrile-water with the following salts: potassium carbonate, potassium fluoride, potassium bromide, sodium sulfate, sodium thiosulfate, sodium carbonate, sodium citrate, and ammonium sulfate. Acetonitrile having becoming an extremely versatile material, the authors decided to continue their investigation on phase equilibria of ternary acetonitrile–water–salts systems.

EXPERIMENTAL

A new series of qualitative tests on the salting-out power of other salts is presented in Table I. The ternary diagrams for water-acetonitrile–K acetate, water-aceonitrile–Li chloride, water-acetonitrile–Li acetate, and water-acetonitrile–Li sulfate were determined in a laboratory air conditioned to ±25° C. In addition, the equilibria were reached and maintained in a water bath thermostatically controlled at 25° ± 0.05° C. As described previously (*8, 9*), the binodal curves were determined by the well known cloud point method utilizing closed vials for the weighed components. Tie lines were constructed by preparing mixtures of known composition within the limits of the two-phase region, shaking mechanically for 20 minutes, and allowing the two layers to separate while immersed in the 25° C. bath. The two layers were analyzed for salt content by evaporation to dryness to constant weight. The plait point was determined by the method of Coolidge (*4*), and checked by the method of Othmer and Tobias (*7*), as well as by a novel method proposed below. The hydrated salts of Li acetate$\cdot 2H_2O$ and Li sulfate$\cdot H_2O$ were used. Because of some discrepancies in the quantities of moles of water of hydration, the water content was checked by thermal balance and the calculations were corrected to the basis of anhydrous salts. Weighings were made to 0.1 mg. (Mettler balance) and compositions were expressed to the nearest 0.1% by weight. Data for the systems are given in Tables II and III and binodal curves in Figures 1 and 2.

MATERIALS

The salts used (99.7+% purity) were potassium acetate (crystals), analytical reagent Mallinckrodt; lithium sulfate, Baker analyzed reagent; lithium acetate and lithium chloride, Fisher certified reagents. The acetonitrile was high-purity spectro grade (Matheson, Coleman and Bell) with a refractive index at 20° C. of 1.3440 [literature value = 1.3441 (*11*)]. Distilled water was used in all of the experimental work.

RESULTS

The data for the four systems show that K acetate has the greater salting-out power, followed successively by Li chloride, Li acetate, and Li sulfate. The solubility in the salt solutions increases from K acetate, Li sulfate, Li acetate, to Li chloride. Table I shows that of all the halogens of lithium, only Li chloride has salting-out properties.

CORRELATIONS

Correlations of tie-line data serve the dual purpose of assisting in interpolation of the tie-line data and extrapolation of the data to the plait point. The correlations involve graphs of the binodal curve, such as the diagrams in Figures 1 and 2, similar plots on rectangular coordinates, or plots of composition variables

Table I. Effectiveness of Salts with Acetonitrile

Effective	Ineffective	Precipitated
$LiCl$	$LiBr$	LiF
$LiC_2H_3O_2$	LiI	Li_2CO_3
Li_2SO_4	$LiNO_3$	