

Binary and Ternary Vapor-Liquid Equilibria of Methanol-Acetone-2,3-Dimethylbutane

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Vapor-liquid equilibrium data are presented for the binary systems 2,3-dimethylbutane-methanol, 2,3-dimethylbutane-acetone, and 2,3-dimethylbutane-chloroform, and for the ternary system 2,3-dimethylbutane-methanol-acetone at 760 mm. of mercury pressure. All systems contain minimum boiling azeotropes. The azeotrope in the ternary system is located at $x_{\text{DMB}} = 0.517$, $x_{\text{methanol}} = 0.254$, $x_{\text{acetone}} = 0.229$. The binary systems are fitted by the Wilson equations, and the multicomponent form of the Wilson equation is used to predict the multicomponent behavior from the binary data.

BECAUSE of experimental time involved in determining multicomponent vapor-liquid equilibrium data and their value in design, it is desirable to predict such data accurately from a minimum of information. This requires suitable equations and accurate multicomponent data to prove their effectiveness. Several sets of predictive equations have been developed, some requiring multicomponent interaction parameters and some requiring only binary parameters to describe the behavior of the system. The Wilson-type equations (12) require only binary parameters to describe a system of n components, with certain restrictions with regard to miscibility of the components in the liquid. The purpose of this investigation was to develop additional reliable multicomponent data at atmospheric pressure for a highly nonideal system and to determine the effectiveness of the Wilson-type equations in predicting the vapor-liquid equilibrium relationship for the system.

Experimental data were determined for the binary systems 2,3-dimethylbutane-methanol and 2,3-dimethylbutane-acetone. The ternary system 2,3-dimethylbutane-methanol-acetone was examined, and a limited number of experimental points were taken in the ternary system methanol-acetone-chloroform and the quaternary system 2,3-dimethylbutane-methanol-acetone-chloroform. All data

were taken at 760 mm. of Hg, at which pressure the vapor phase could be assumed to behave ideally.

EXPERIMENTAL

Materials. The 2,3-dimethylbutane was obtained from Phillips Petroleum Co. as the "pure" grade, with a guaranteed minimum purity of 99.0 mole %. The methanol and chloroform were Mallinckrodt Nanograde, and the acetone was Fisher Scientific ACS reagent grade. Boiling point and refractive index were measured for each component as an indication of component purity. These physical properties are listed in Table I, together with the accepted literature values (2, 3, 4, 9). All materials were used without further purification.

Apparatus and Procedure. A modified Colburn still (5) was used for determination of the equilibrium data. The data are presented in Tables II through VII, and Figures 1 through 3. The data for the system 2,3-dimethylbutane-methanol were taken in cooperation with Kirby (8). All temperatures were measured with copper-constantan thermocouples, which were calibrated for accuracy against a second set of thermocouples. This second set of thermocouples had been calibrated against a platinum resistance thermometer calibrated by the National Bureau of Standards. Thermocouple potential was measured with a Leeds

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Table I. Physical Properties of Pure Components

Component	Dimethylbutane	Methanol	Acetone	Chloroform
Molecular weight	86.17	32.04	58.08	119.39
Boiling point, °C				
Literature	57.988 (3)	64.7 (3)	56.1 (3)	61.3 (3)
Experimental	58.03	64.59	56.11	61.24
Refractive index				
n_D^{20}				
Literature	1.37495 (3)	1.3288 (4)	1.3588 (3)	...
Experimental	1.37493	1.32877	1.35841	1.44576
n_D^{25}				
Literature	1.37231 (2)	1.44293 (2)
Experimental	1.37257	1.32684	1.35841	1.44310
Specific gravity	0.668 ¹⁷ / ₄ (4)	0.7928 ²⁰ / ₄ (4)	0.792 ²⁰ / ₄ (4)	1.4984 ¹⁵ / ₄ (4)
Antoine constants				
A	6.80983 (2)	7.87863 (9)	7.02447 (9)	6.90328 (9)
B	1127.187	1473.11	1161.0	1163.03
C	228.9	230.0	224.0	227.4

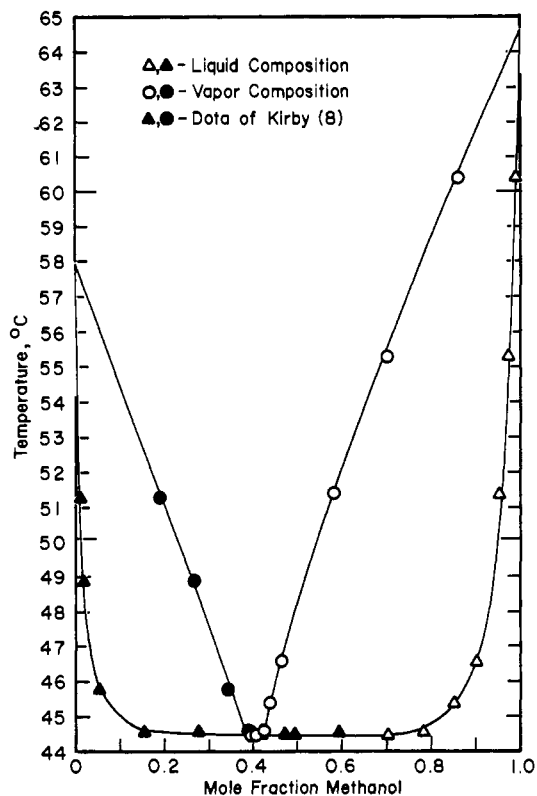


Figure 1. Temperature-composition diagram for dimethylbutane-methanol system

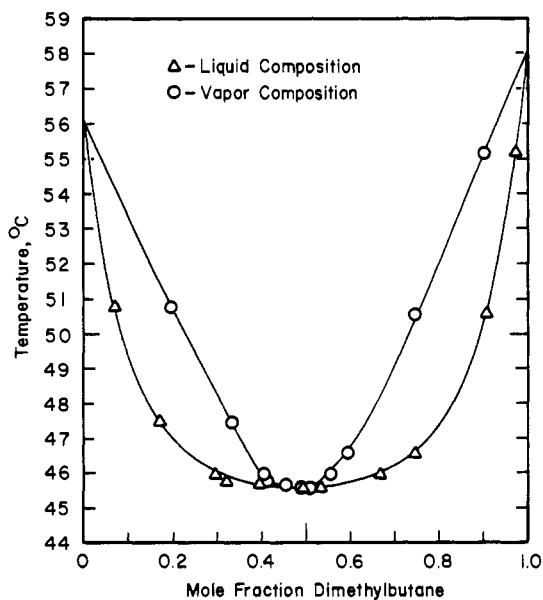


Figure 2. Temperature-composition diagram for dimethylbutane-acetone system

& Northrup portable precision potentiometer. Pressure in the equilibrium apparatus was measured with a mercury-in-glass manometer read with a cathetometer. Accuracy of experimental measurements is estimated at ± 0.10 mm. of Hg and $\pm 0.10^\circ\text{C}$.

Samples of the equilibrium vapor and liquid were analyzed using a Beckman GC-2 or GC-2A gas chromatograph. Accuracy of analysis was maintained by sequential analysis of a sample of known composition and an

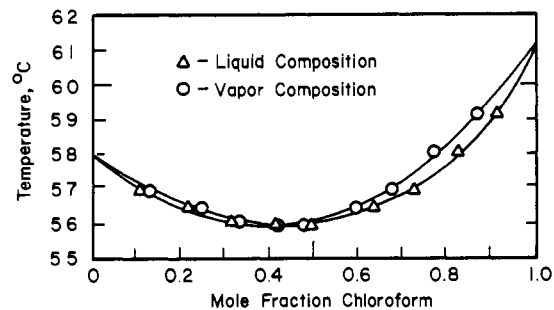


Figure 3. Temperature-composition diagram for dimethylbutane-chloroform system

Table II. Vapor-Liquid Equilibrium Data for Binary System Dimethylbutane (1)-Methanol(2)^a

$T, ^\circ\text{C}$	x_1	x_2	y_1	y_2	γ_1	γ_2
60.4	0.009	0.991	0.139	0.861	14.322	1.032
55.3	0.024	0.976	0.297	0.703	13.480	1.055
51.4	0.045	0.955	0.420	0.580	11.545	1.048
46.6	0.096	0.904	0.536	0.464	8.115	1.092
45.4	0.149	0.851	0.563	0.437	5.723	1.153
44.6	0.216	0.784	0.579	0.421	4.174	1.249
44.5	0.296	0.704	0.594	0.406	3.136	1.348
44.6 ^b	0.408	0.592	0.604	0.396	2.305	1.556
44.5 ^b	0.507	0.493	0.608	0.392	1.874	1.858
44.5 ^b	0.532	0.468	0.606	0.394	1.780	1.968
44.5 ^b	0.585	0.415	0.608	0.392	1.624	2.208
44.5 ^b	0.610	0.390	0.607	0.393	1.555	2.355
44.6 ^b	0.726	0.274	0.609	0.391	1.306	3.320
44.6 ^b	0.847	0.153	0.615	0.385	1.131	5.855
45.8 ^b	0.949	0.051	0.658	0.342	1.036	14.787
48.9 ^b	0.983	0.017	0.734	0.266	1.004	30.093
51.3 ^b	0.991	0.009	0.810	0.190	1.014	36.600

^aSystem pressure, 760 ± 0.1 mm. of Hg. ^bKirby (8).

Table III. Vapor-Liquid Equilibrium Data for Binary System Dimethylbutane(1)-Acetone(3)^a

$T, ^\circ\text{C}$	x_1	x_3	y_1	y_3	γ_1	γ_3
50.8	0.071	0.929	0.198	0.802	3.519	1.041
47.5	0.173	0.827	0.337	0.663	2.746	1.088
46.0	0.297	0.703	0.407	0.593	2.033	1.209
45.8	0.322	0.678	0.415	0.585	1.925	1.246
45.7	0.397	0.603	0.456	0.544	1.722	1.307
45.6	0.496	0.504	0.494	0.506	1.498	1.460
45.6	0.536	0.464	0.511	0.489	1.434	1.533
46.0	0.669	0.331	0.557	0.443	1.235	1.918
46.6	0.749	0.251	0.595	0.405	1.155	2.262
50.6	0.910	0.090	0.748	0.252	1.044	3.400
55.2	0.975	0.025	0.904	0.096	1.013	3.972

^aSystem pressure, 760 ± 0.1 mm. of Hg.

Table IV. Vapor-Liquid Equilibrium Data for Binary System Dimethylbutane(1)-Chloroform(4)^a

$T, ^\circ\text{C}$	x_1	x_4	y_1	y_4	γ_1	γ_4
59.2	0.087	0.913	0.130	0.870	1.438	1.034
58.1	0.176	0.824	0.230	0.770	1.302	1.051
57.0	0.275	0.725	0.326	0.674	1.223	1.085
56.5	0.367	0.633	0.406	0.594	1.160	1.113
56.0	0.509	0.491	0.525	0.475	1.099	1.167
56.0	0.588	0.412	0.588	0.412	1.065	1.206
56.1	0.688	0.312	0.671	0.329	1.036	1.268
56.5	0.785	0.215	0.760	0.240	1.015	1.324
57.0	0.894	0.106	0.872	0.106	1.409	1.409

^aSystem pressure, 760 ± 0.1 mm. of Hg.

Table V. Vapor-Liquid Equilibrium Data for Ternary System Dimethylbutane(1)-Methanol(2)-Acetone(3)^a

(Comparison of experimental and predicted values)

$T, ^\circ\text{C}_e$	x_{1e}	x_{2e}	x_{3e}	y_{1e}	y_{2e}	y_{3e}	γ_{1e}	γ_{2e}	γ_{3e}
$T, ^\circ\text{C}_p$				y_{1p}	y_{2p}	y_{3p}			
44.7	0.150	0.631	0.219	0.459	0.340	0.201	4.772	1.248	1.382
45.1				0.458	0.338	0.204			
43.6	0.303	0.311	0.386	0.447	0.238	0.315	2.373	1.868	1.279
44.7				0.461	0.228	0.311			
47.6	0.072	0.773	0.155	0.385	0.442	0.173	7.490	1.163	1.514
49.3				0.393	0.431	0.176			
46.6	0.091	0.609	0.300	0.371	0.350	0.279	5.883	1.224	1.303
48.0				0.367	0.347	0.286			
44.6	0.196	0.310	0.494	0.399	0.217	0.384	3.168	1.629	1.172
45.8				0.402	0.216	0.382			
49.5	0.049	0.748	0.203	0.316	0.453	0.231	8.472	1.131	1.439
51.0				0.312	0.449	0.239			
43.8	0.397	0.165	0.438	0.456	0.163	0.381	1.844	2.390	1.350
44.7				0.466	0.167	0.367			
43.8	0.766	0.145	0.089	0.584	0.282	0.134	1.218	4.687	2.354
44.5				0.581	0.295	0.124			
44.3	0.771	0.066	0.163	0.573	0.181	0.246	1.167	6.506	2.311
45.1				0.567	0.193	0.240			
44.3	0.642	0.317	0.041	0.589	0.353	0.058	1.444	2.626	2.138
44.6				0.599	0.350	0.051			
46.9	0.131	0.152	0.717	0.313	0.133	0.554	3.453	1.829	1.073
47.9				0.312	0.133	0.555			
47.0	0.107	0.268	0.625	0.297	0.203	0.500	3.964	1.580	1.106
48.1				0.301	0.202	0.497			
50.3	0.056	0.132	0.812	0.189	0.127	0.684	4.333	1.744	1.035
51.0				0.186	0.127	0.687			
51.1	0.036	0.246	0.718	0.148	0.213	0.639	5.074	1.508	1.061
51.8				0.146	0.212	0.642			
43.2	0.460	0.243	0.297	0.496	0.231	0.273	1.758	2.353	1.464
44.3				0.501	0.230	0.269			
44.1	0.509	0.081	0.410	0.487	0.116	0.397	1.514	3.409	1.491
45.0				0.489	0.118	0.393			
44.0	0.379	0.574	0.047	0.567	0.378	0.055	2.376	1.576	1.787
44.7				0.587	0.362	0.051			
44.5	0.182	0.419	0.399	0.418	0.262	0.320	3.600	1.464	1.215
45.7				0.418	0.260	0.322			
46.1	0.109	0.463	0.428	0.344	0.292	0.364	4.691	1.375	1.215
47.5				0.350	0.285	0.365			
44.3	0.300	0.158	0.542	0.423	0.147	0.430	2.220	2.195	1.209
45.3				0.430	0.147	0.423			
44.1	0.659	0.071	0.270	0.531	0.144	0.325	1.278	4.833	1.849
45.0				0.530	0.148	0.322			
43.4	0.561	0.223	0.216	0.524	0.248	0.228	1.521	2.736	1.661
44.2				0.528	0.251	0.221			
43.4	0.411	0.389	0.200	0.514	0.298	0.188	2.034	1.881	1.488
44.3				0.527	0.287	0.186			
43.7	0.276	0.507	0.217	0.495	0.310	0.195	2.883	1.488	1.402
44.7				0.505	0.302	0.193			
44.4	0.174	0.702	0.124	0.504	0.371	0.125	4.534	1.240	1.532
45.8				0.511	0.365	0.124			
43.5	0.504	0.241	0.255	0.510	0.242	0.248	1.638	2.458	1.528
44.2				0.515	0.242	0.243			
43.2	0.517	0.253	0.230	0.517	0.254	0.229	1.636	2.491	1.587
44.2				0.523	0.253	0.224			

^aSystem pressure, 760 ± 0.1 mm. of Hg.

equilibrium sample. The sample of known composition was composed by difference weighing to approximately the same composition as the equilibrium sample. The approximate composition of each equilibrium sample was determined by chromatographic analysis of the sample. The relationship between peak area and composition was determined from analysis of the sample of known composition. This relationship, applied to the peak areas of the analysis of the equilibrium sample, allowed accurate determination of equilibrium sample composition. The accuracy of composition measurement is estimated at ±0.002 mole fraction unit.

Calculation of Activity Coefficient. Activity coefficients were calculated for each equilibrium point, using the equation

$$P y_i = \gamma_i P_i x_i \quad (1)$$

The vapor pressure for each component was determined using the corresponding Antoine constants listed in Table I. Vapor phase nonideality was neglected, since all data were taken at atmospheric pressure and calculations indicated the correction for pressure to be insignificant.

Correlation of Binary Data. The binary equilibrium data were correlated using the Wilson equations. For a binary system, the equations are

Table VI. Vapor-Liquid Equilibrium Data for Ternary System Methanol(2)-Acetone(3)-Chloroform(4)^a

(Comparison of experimental and predicted values)

T, °C _e T, °C _p	x _{2e}	x _{3e}	x _{4e}	y _{2e}	y _{3e}	y _{4e}	γ _{2e}	γ _{3e}	γ _{4e}
				y _{2P}	y _{3P}	y _{4P}			
58.1	0.292	0.368	0.340	0.357	0.337	0.306	1.588	0.861	1.014
57.5				0.360	0.332	0.308			
59.5	0.146	0.436	0.418	0.235	0.411	0.354	1.982	0.846	0.911
58.9				0.253	0.396	0.351			
56.7	0.400	0.188	0.412	0.419	0.143	0.438	1.445	0.746	1.250
56.5				0.414	0.150	0.436			
57.4	0.403	0.428	0.169	0.406	0.446	0.148	1.351	1.003	1.009
57.1				0.401	0.446	0.153			
57.7	0.495	0.264	0.241	0.475	0.264	0.261	1.274	0.948	1.232
57.2				0.464	0.268	0.268			

^aSystem pressure, 760 ± 0.1 mm. of Hg.

Table VII. Vapor-Liquid Equilibrium Data for Quaternary System Dimethylbutane(1)-Methanol(2)-Acetone(3)-Chloroform(4)^a

(Comparison of experimental and predicted values)

T, °C _e T, °C _p	x _{1e}	x _{2e}	x _{3e}	x _{4e}	y _{1e}	y _{2e}	y _{3e}	y _{4e}	γ _{1e}	γ _{2e}	γ _{3e}	γ _{4e}
					y _{1P}	y _{2P}	y _{3P}	y _{4P}				
49.4	0.175	0.251	0.291	0.283	0.333	0.262	0.225	0.180	2.514	1.967	0.976	0.962
49.6					0.341	0.266	0.215	0.178				
50.4	0.222	0.128	0.324	0.326	0.357	0.180	0.260	0.203	2.052	2.552	0.984	0.911
50.4					0.362	0.197	0.249	0.192				
53.1	0.070	0.281	0.334	0.315	0.195	0.306	0.266	0.233	3.252	1.751	0.888	0.983
52.9					0.201	0.303	0.263	0.233				
48.5	0.224	0.297	0.156	0.323	0.363	0.301	0.105	0.231	2.204	1.989	0.882	1.116
48.7					0.371	0.297	0.108	0.224				
47.1	0.210	0.302	0.334	0.154	0.391	0.263	0.262	0.084	2.667	1.819	1.078	0.898
47.6					0.397	0.259	0.258	0.086				

^aSystem pressure, 760 ± 0.1 mm. of Hg.

Table VIII. Pure Component Molar Volumes

Component	Dimethylbutane	Methanol	Acetone	Chloroform
Temperature, °K.	273.15	273.15	228.15	273.15
Molar volume, cc./mole	126.80	39.556	67.380	78.218
Temperature, °K.	303.15	373.15	273.15	303.15
Molar volume, cc./mole	132.06	44.874	71.483	81.185
Temperature, °K.	333.15	473.15	323.15	333.15
Molar volume, cc./mole	138.03	57.939	76.826	84.500

Table IX. Values of Parameters (λ_{ij} - λ_{ii}), (λ_{ij} - λ_{jj}) for Binary Systems^a

Component i	Component j			
	Dimethylbutane	Methanol	Acetone	Chloroform
Dimethylbutane	0	449.08	234.96	-28.72
	0	2771.85	948.29	342.16
Methanol	2771.85	0	664.08	1703.68
	449.08	0	-214.95	-373.30
Acetone	948.29	-214.95	0	-72.20
	234.96	664.08	0	-332.23
Chloroform	342.16	-373.30	-332.23	0
	-28.72	1703.68	-72.20	0

^aFirst parameter in each set for a given pair of components i and j, is (λ_{ij} - λ_{ii}). Second parameter is (λ_{ij} - λ_{jj}), cal. per gram mole

$$\ln \gamma_i = -\ln(x_i + \Lambda_{12}x_2) + x_2 \left(\frac{\Lambda_{12}}{x_i + \Lambda_{12}x_2} - \frac{\Lambda_{21}}{\Lambda_{21}x_1 + x_2} \right) \quad (2)$$

$$\ln \gamma_2 = -\ln(\Lambda_{21}x_1 + x_2) - x_1 \left(\frac{\Lambda_{12}}{x_i + \Lambda_{12}x_2} - \frac{\Lambda_{21}}{\Lambda_{21}x_1 + x_2} \right) \quad (3)$$

where

$$\Lambda_{ij} \equiv \frac{V_{iL}}{V_{jL}} \exp - [(\lambda_{ij} - \lambda_{ii})/RT] \quad (4)$$

The quantity (λ_{ij} - λ_{ii}) is assumed to be independent of temperature over a small temperature range (11). Liquid molar volume for each component was expressed as a function of temperature by fitting a quadratic equation through the points of temperature and molar volume in Table VIII. The parameters (λ_{ij} - λ_{ii}) and (λ_{ij} - λ_{jj}) were determined for each binary system by a least squares fit of the Wilson

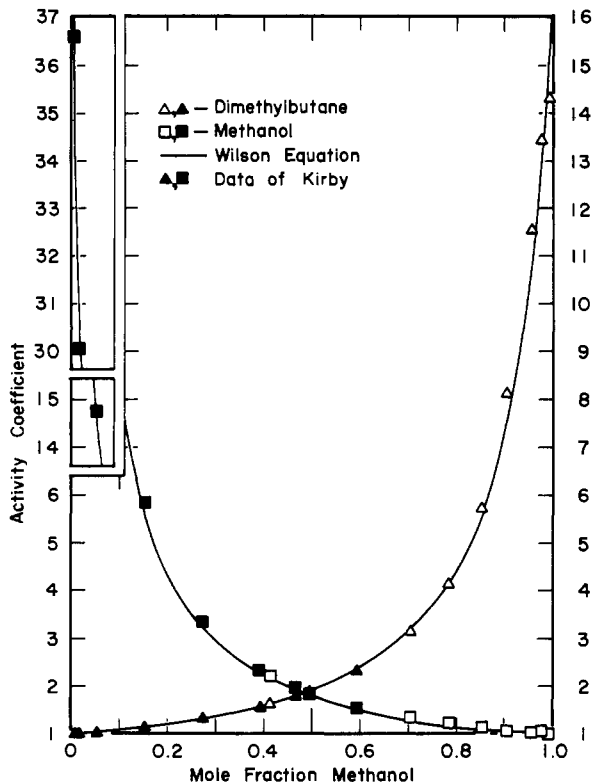


Figure 4. Activity coefficient-composition diagram for dimethylbutane-methanol system

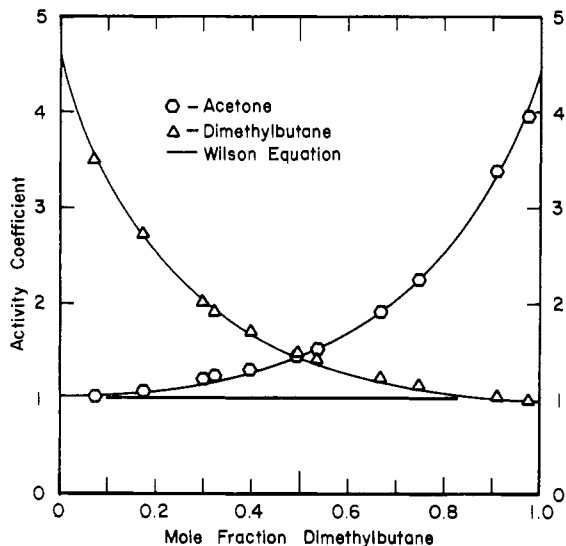


Figure 5. Activity coefficient-composition diagram for dimethylbutane-acetone system

equations to the experimental activity coefficient-composition-temperature data for the system. The solution was accomplished using the method of Holmes (6), which minimizes the total residual sum of squares of the activity coefficient-composition function for both components.

The parameters for each experimental binary system are presented in Table IX. Also tabulated are the parameters determined for the three additional binary systems for which the data were available in the literature (1, 7, 11). The correlation of the activity coefficient data by the Wilson equations is shown by the solid curves in Figures 4 through

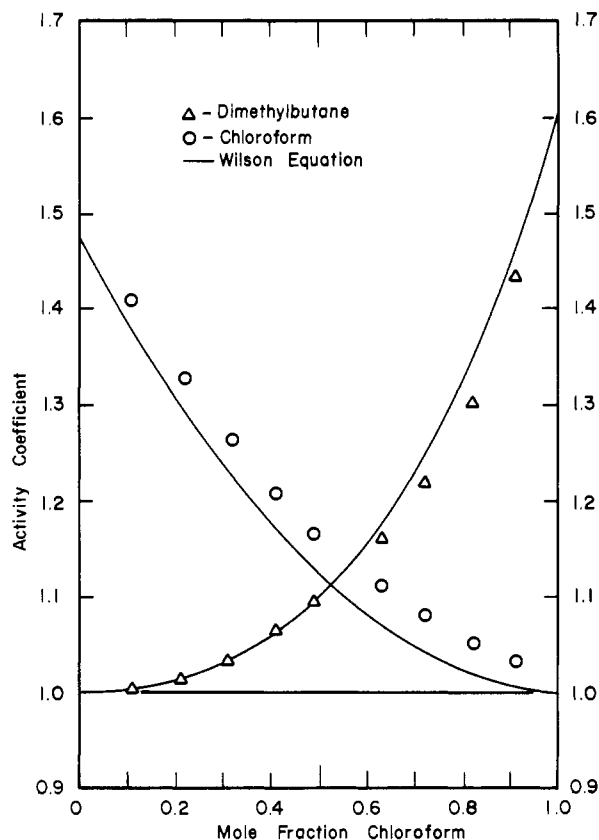


Figure 6. Activity coefficient-composition diagram for dimethylbutane-chloroform system

6. The deviation of the chloroform activity coefficients from the curve possibly can be attributed to vapor phase non-ideality.

Prediction of Multicomponent Data. The multicomponent activity coefficients were predicted using the multicomponent form of the Wilson equation,

$$\ln \gamma_k = -\ln \left(\sum_{j=1}^n x_j \Lambda_{kj} \right) + 1 - \sum_{i=1}^n \frac{x_i \Lambda_{ik}}{\sum_{j=1}^n x_j \Lambda_{ij}} \quad (5)$$

For calculation purposes, liquid phase composition was fixed at the experimental value. Determination of the bubble point temperature at which

$$\sum_{i=1}^n y_i = \sum_{i=1}^n \frac{\gamma_i x_i P_i}{P} = 1.0 \quad (6)$$

then yielded the predicted equilibrium temperature and vapor composition. These predicted temperatures and vapor compositions are compared with the experimental values for each multicomponent system in Tables V through VII.

RESULTS AND CONCLUSIONS

The Wilson equations provide an excellent basis for correlation of the binary activity coefficient data and the multicomponent equilibria are accurately predicted using only the binary parameters and the multicomponent form of the Wilson equation. At pressures low enough to justify the assumption of ideal behavior in the vapor phase, the Wilson equation provides a simple method for predicting vapor-liquid equilibrium in systems of up to four components exhibiting considerable liquid phase nonideality.

NOMENCLATURE

- P = total pressure, mm. of Hg
 P_i = vapor pressure of component i , mm. of Hg
 R = gas constant, 1.987 cal. per gram mole-° K.
 T = temperature, ° K
 V_{iL} = liquid molar volume of component i , cm.³ per gram mole
 x_i = mole fraction of component i in liquid phase
 y_i = mole fraction of component i in vapor phase

Greek Letters

- γ_i = liquid phase activity coefficient of component i
 $(\lambda_{ij} - \lambda_{ii})$ = Wilson parameters for interaction of ij -pair relative to ii -pair
 Λ_{ij} = parameter of Wilson equation, defined in Equation 4
 Σ = summation of

Subscripts

- 1 = 2,3-dimethylbutane
 2 = methanol
 3 = acetone
 4 = chloroform
 e = experimental
 p = predicted

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Heat Capacity of Alcohol Vapors at Atmospheric Pressure

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Vapor-phase heat capacity was measured in a flow calorimeter at atmospheric pressure in the temperature range from saturation to approximately 330° C. for the following aliphatic alcohols: methyl, ethyl, *n*-propyl, isopropyl, *n*-butyl, isobutyl, *sec*-butyl, *tert*-butyl, *n*-pentyl, isopentyl-, and *tert*-pentyl. Equation 9 was derived as a suitable model for all the alcohols investigated and may be used for extrapolation to higher alcohols.

THE CHEMICAL engineering laboratories at the Technical University of Norway have for some time been investigating the temperature and pressure dependence of vapor-phase enthalpy. The pressure effect is measured by isothermal expansion in an apparatus described by Stris determined by measuring the heat capacity at atmospheric pressure in a flow calorimeter (9).

These measurements, carried out with a homologous series, may give a more accurate basis for estimation of the vapor-phase enthalpy. The first part of the investigation is concerned with 11 aliphatic alcohols. Their heat capacity at atmospheric pressure was measured from the normal boiling point to approximately 330° C. The measurement results and their mathematical correlation models are reported here.

EXPERIMENTAL

The heat capacity measurements were carried out as described by Lydersen and Rønne (9) in the flow calorimeter described by them.

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The performance of the equipment was examined by measuring the heat capacity of steam. Figure 1 gives the results and the values from the VDI-steam table (11). The average deviation is 0.002 kcal./kg., ° C. (0.4%).

The alcohols used in the experiments were of the following origins:

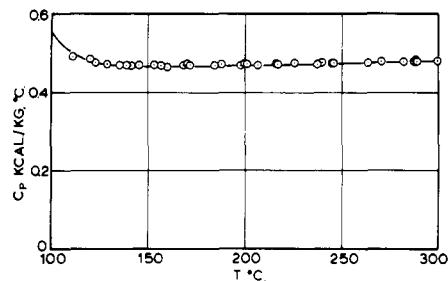


Figure 1. Heat capacity for steam measured at atmospheric pressure

Curve corresponds to steam table (11)

○ This work