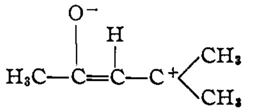
and the polar structure



According to an earlier explanation based on the MO theory,[22] this can be replaced by the interaction between the highest occupied orbital of the C=C bond ($H_{C=C}$) and the lowest vacant one of the carbonyl group ($V_{C=O}$) as shown in Fig. 4a.[22] As the result of the interaction between these two orbitals, we can expect the appearance of two new orbitals ψ_n and ψ_e for mesityl oxide.[23] One of them which is filled with two electrons in the normal state W_n can be represented by a linear combination of wave functions for orbitals $H_{C=C}$ and $V_{C=O}$, namely, $\psi_n = a\psi_{C=C} + b\psi_{C=O}$. The value of b, which represents a measure of the electron migration from the C=C bond to the C=O bond in the normal state, or in other words the degree of the contribution of the polar structure to the resonance in that state, is usually small. On the other hand, the wave function of the excited level W_e, which is orthogonal to that of W_n, contains one electron in ψ_n and the second in the orbital ψ_e of the form $\psi_e = b\psi_{C=C} - a\psi_{C=O}$ $(a > b)$. Then in the W_e state, the π electron on the C=C bond migrates to the C=O bond to a large extent. Thus it may be expected that the transition from W_n to W_e can be accompanied by a large electron transfer and therefore by a strong absorption. This absorption may be called an intramolecular charge transfer absorption in analogy to the intermolecular charge transfer absorption discussed by Mulliken.[8]

According to the foregoing discussion of the absorption, the excited state of the 231 mμ band of mesityl oxide is more polar than the normal state. In other words, the oxygen atom is more negatively charged in the former state than in the latter. Then it is reasonable to consider that the attachment of a proton to oxygen results in larger energy stabilization in the upper state than in the lower state and therefore in a large red shift of the absorption band. Furthermore, the solvation energy in polar medium, which is conceivably larger in the excited state than in the normal one, should also help the red shift. Thus, the large red shift seems to be explained qualitatively on the basis of the conception of charge transfer absorption.

Experimental

C.P. acetone was treated with an aqueous alkaline solution of $AgNO_3$, distilled, dried with anhydrous K_2CO_3 and finally fractionally distilled before use. White label Eastman mesityl oxide and mesitylene were fractionally distilled. Commercial phorone was four times recrystallized from methanol. *n*-Heptane was stirred with concentrated sulfuric acid for one or two days, washed with water and aqueous alkaline solution, dried with $CaCl_2$ and finally fractionally distilled with sodium metal. Water treated by ion exchange resin was distilled with $KMnO_4$. C.P. concentrated sulfuric acid was used without further purification.

The absorption spectra were measured with a Beckman spectrophotometer model DU, using the quartz absorption cell with 10 mm. light path. The temperature of the cell compartment was kept at 25° by the use of the thermospacer.

Acknowledgments.—The authors wish to express their sincere thanks to Professor Mulliken for his kindness in reading this manuscript and in giving them some valuable advice. One of the authors (S. N.) is also indebted to Professor Mulliken and Professor Platt and other members of the Laboratory for the hospitality shown during his stay at Chicago.

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[22] S. Nagakura and J. Tanaka, *J. Chem. Phys.*, **22**, 236 (1954); S. Nagakura, *ibid.*, **23**, 1441 (1955).

[23] A more accurate treatment will be published shortly.

[CONTRIBUTION FROM THE RESEARCH DEPARTMENT, PLASTICS DIVISION, MONSANTO CHEMICAL COMPANY]

Vapor–Liquid Equilibria in Binary Systems

BY W. F. YATES

RECEIVED OCTOBER 5, 1956

An equation containing two parameters has been derived from kinetic considerations which relates instantaneous vapor–liquid equilibria in binary systems. The two constants appearing in the equation have been described in terms of the thermodynamic properties of the mixtures concerned. The validity of the assumptions involved has been tested by applying them to data selected from the literature. Until recently the only adequate means of representing vapor–liquid equilibrium data involved the use of activity coefficients and rely on various integrations of the Gibbs–Duhem equation. The relationships existing, however, do not greatly contribute to the understanding of the phenomena involved in evaporation or condensation.

Spinner, Lu and Graydon[1] have observed the similarity between representations of vapor–liquid equilibrium data and copolymer–monomer composition plots and have applied the Alfrey–Price[2] relationship to predict vapor–liquid equilibria. The remarkable success of these investigations has prompted closer study to see if there is any basis in theory for the use of the equations they propose.

Considered from the standpoint of a binary liquid mixture containing components A and B in the process of evaporating, four possible processes are occurring

$$-A - A(l) \xrightarrow{k_{11}} -A(l) + A(g) \qquad (1)$$

$$-B - A(l) \xrightarrow{k_{21}} -B(l) + A(g) \qquad (2)$$

$$-B - B(l) \xrightarrow{k_{22}} -B(l) + B(g) \qquad (3)$$

$$-A - B(l) \xrightarrow{k_{12}} -A(l) + B(g) \qquad (4)$$

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[1] I. H. Spinner, B. C.-Y. Lu and W. F. Graydon, *Ind. Eng. Chem.*, **48**, 147 (1956).

[2] T. Alfrey and C. C. Price, *J. Polymer Sci.*, **2**, 101 (1947).

TABLE I

CALCULATION OF r_1 AND r_2 FROM EXCESS CHEMICAL POTENTIALS

System	t, °C.	μ_{12}^E	μ_{21}^E	$P_1°/P_2°$	r_1	r_2	Ref.
Benzene–cyclohexane	40	306.5	283	0.9895	0.6045	0.6411	3
	70	261	241.5	1.0129	0.6906	.6927	
Carbon tetrachloride–cyclohexane	40	65.3	61.3	1.1559	1.0407	.7839	4
	70	61.0	54.8	1.1351	1.0379	.8129	
Carbon tetrachloride–benzene	40	75.5	76.1	1.1682	1.0347	.7574	5
	70	71.5	72.8	1.1207	1.0091	.8019	
Methanol–benzene	35	1240	1597	1.4147	0.1867	.0521	6
	55	1277	1557	1.5781	.2224	.0581	
Methanol–carbon tetrachloride	35	1242	1652	1.2014	.1580	.0561	7
	55	1340	1681	1.3820	.1768	.0549	
Methyl acetate–benzene	25	328	504	2.2615	1.3000	.1889	8
	35	337	476	2.2296	1.2858	.2061	
Dimethoxymethane–chloroform	35	−550	−636	1.9383	4.7585	1.4580	8
Dimethoxymethane–benzene	35	50.5	169	3.9782	3.6635	0.1908	8
Acetone–chloroform[9]	50	−500	−500	1.1646	2.5381	1.8714	8
	60			1.1636	2.3311	1.7257	
Nitrogen–oxygen	−198	42.9	32.4	5.2273	3.9200	0.1539	8
	−178	56.0	− 7.6	3.3061	2.4574	.2905	
	−148	138.1	− 51.4	2.3652	1.3572	.5200	
Ethanol–water	50	511	1326	2.4029	1.084	.0528	10
	60	579	1426	2.3624	0.985	.0491	

where k_{11}, etc., are the rate constants for the respective processes. The rate of change in liquid composition for each component is then given by

$$\frac{-\mathrm{d}N_A}{\mathrm{d}t} = k_{11}(-A-A) + k_{21}(-B-A) \quad (5a)$$

and

$$\frac{-\mathrm{d}N_B}{\mathrm{d}t} = k_{22}(-B-B) + k_{12}(-A-B) \quad (5b)$$

By setting up steady-state conditions for equations 1 through 4 it can be seen that

$$\frac{\mathrm{d}(-A)}{\mathrm{d}t} = 0 = k_{12}(-A-B) - k_{21}(-B-A) \text{ or}$$

$$k_{12}(-A-B) = k_{21}(-B-A) \quad (6)$$

By dividing equation 5a by 5b and substituting equation 6 into the result

$$\frac{\mathrm{d}N_A}{\mathrm{d}N_B} = \frac{\dfrac{k_{11}(-A-A)}{k_{12}(-A-B)} + 1}{\dfrac{k_{22}(-B-B)}{k_{21}(-B-A)} + 1} \quad (7)$$

Since A and B must each distribute themselves in the liquid by attaching to molecules according to the ratio of their mole fractions

$$\frac{(-A-A)}{(-A-B)} = \frac{x_1}{x_2} \quad (8a)$$

and

$$\frac{(-B-B)}{(-B-A)} = \frac{x_2}{x_1} \quad (8b)$$

Substituting equations 8a and 8b into 7 and by defining $r_1 = k_{11}/k_{12}$ and $r_2 = k_{22}/k_{21}$

$$\frac{\mathrm{d}N_A}{\mathrm{d}N_B} = \frac{x_1(r_1x_1 + x_2)}{x_2(r_2x_2 + x_1)} \quad (9)$$

The change in liquid composition, $\mathrm{d}N_A/\mathrm{d}N_B$, must define the vapor composition. If the vapor composition is expressed in terms of mole fractions equation 9 becomes

$$\frac{y_1}{y_2} = \frac{x_1\,(r_1x_1 + x_2)}{x_2\,(r_2x_2 + x_1)} \quad (10)$$

Equation 10 is seen to be identical with the vapor–liquid equilibrium equation proposed by Spinner, Lu and Graydon.[1]

The derivation of equation 10 is valid only for systems at constant temperature. Spinner has presented a number of calculations of r_1 and r_2 values for systems at constant pressure and has demonstrated that the equation describes the data in most cases.

Calculation of Binary Constants.—In order to predict vapor–liquid equilibria for binary systems it is necessary to have an independent means of computing values of r_1 and r_2. Spinner used the empirical relationship

$$r_1 = \frac{A_1}{A_2}\,e^{-B_1(B_1 - B_2)} \quad (11a)$$

$$r_2 = \frac{A_2}{A_1}\,e^{-B_2(B_2 - B_1)} \quad (11b)$$

and evaluated the constants A and B from x-y data. It would be advantageous to be able to predict r_1 and r_2 values from thermodynamic properties of each system.

The rate of evaporation of a pure compound k_{11}, should be given by the Arrhenius equation when it is considered that the energy barrier to evaporation is equal to the heat of vaporization, L_{11}

$$k_{11} = s_{11}e^{-L_{11}/RT} \quad (12a)$$

Similarly, for molecules of B evaporating from molecules of A

$$k_{12} = s_{12}e^{-L_{12}/RT} \quad (12b)$$

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(3) G. Scatchard, S. E. Wood and J. M. Mochel, *J. Phys. Chem.*, **43**, 119 (1939).

(4) G. Scatchard, S. E. Wood and J. M. Mochel, THIS JOURNAL, **61**, 3206 (1939).

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(7) G. Scatchard, S. E. Wood and J. M. Mochel, *ibid.*, **68**, 1960 (1946).

(8) J. C. Chu, "Distillation Equilibrium Data," Reinhold Publ. Corp., New York, N. Y., 1950.

(9) r_1 and r_2 values at 60° calcd. using equation 14 with $H_{12}^m = -1800$ and $H_{21}^m = -1750$.

(10) C. A. Jones, E. M. Schoenborn and A. P. Colburn, *Ind. Eng. Chem.*, **35**, 606 (1943).

In order to evaluate L_{12} one can consider it to be equal to the heat required to unmix a mole of B from an infinitely dilute solution in A (where no B–B bonds are to be found), and vaporize it

$$L_{12} = -H^{\mathrm{m}}_{12} + L_{22}$$

where H^{m}_{12} is equal to $x_2 \xrightarrow{\text{limit}} 0\ (\partial H^{\mathrm{m}}_{\mathrm{x}})/(\partial x_2)$ and $H^{\mathrm{m}}_{\mathrm{x}}$ is the heat of mixing to form a mole of solution of specified composition. From these relationships

$$r_1 = s_{11}/s_{12}e^{(-L_{11} + L_{22} - H^{\mathrm{m}}_{12})/RT} \qquad (13)$$

By differentiating equation 13, and combining the results with the differential form of the Clapeyron equation it can be seen that

$$\frac{\mathrm{d} \ln (r_1P_2°/P_1°)}{\mathrm{d}(1/T)} = H^{\mathrm{m}}_{12}/R \qquad (14)$$

where $P_1°$ and $P_2°$ are the vapor pressures of the pure components. By regarding the constant $r_1 P_2°/P_1°$ as analogous to an equilibrium constant, it should be possible to evaluate it from the excess chemical potential of component B in an infinitely dilute solution in A

$$r_1 = P_1°/P_2°e^{-\mu^{\mathrm{E}}_{12}/RT} \qquad (15a)$$

In this case μ^{E}_{12} is equal to $x_2 \xrightarrow{\text{limit}} 0\ (\partial F_{\mathrm{x}}{}^{\mathrm{E}})/(\partial x_2)$, or is the change in excess free energy accompanying the solution of a mole of B into an infinite amount of A. It can further be shown that

$$r_2 = P_2°/P_1°e^{-\mu^{\mathrm{E}}_{21}/RT} \qquad (15b)$$

In order to test the validity of equations 15a and 15b a few systems have been selected from the literature for study. Table I is a compilation of the results of the calculations of r_1 and r_2 values therefrom and Table II shows the results obtained when the calculated r_1 and r_2 values are applied to equation 10.

TABLE II

CALCULATION OF VAPOR-LIQUID EQUILIBRIA

System							
Benzene–cyclohexane (40°)							
x_1	0.128	0.235	0.369	0.493	0.614	0.743	0.866
y_1 (calcd.)	.169	.278	.392	.489	.583	.692	.817
y_1 (obsd.)	.166	.277	.391	.495	.591	.698	.821
Benzene–cyclohexane (70°)							
x_1	0.119	0.241	0.376	0.495	0.618	0.725	0.866
y_1 (calcd.)	.151	.277	.397	.495	.597	.691	.831
y_1 (obsd.)	.149	.281	.398	.498	.603	.696	.831
Carbon tetrachloride–cyclohexane (40°)							
x_1	0.126	0.245	0.367	0.515	0.606	0.754	0.876
y_1 (calcd.)	.152	.282	.405	.548	.633	.770	.882
y_1 (obsd.)	.152	.282	.407	.547	.634	.770	.882
Carbon tetrachloride–cyclohexane (70°)							
x_1	0.125	0.247	0.364	0.515	0.607	0.754	0.876
y_1 (calcd.)	.146	.278	.397	.544	.631	.767	.882
y_1 (obsd.)	.146	.279	.398	.547	.632	.768	.882
Carbon tetrachloride–benzene (40°)							
x_1	0.140	0.238	0.374	0.492	0.620	0.759	0.872
y_1 (calcd.)	.171	.279	416	.529	.648	.774	.879
y_1 (obsd.)	.170	.277	.416	.530	.648	.774	.878
Carbon tetrachloride–benzene (70°)							
x_1	0.143	0.239	0.379	0.493	0.622	0.762	0.875
y_1 (calcd.)	.167	.271	.411	.521	.642	.772	.879
y_1 (obsd.)	.167	.270	.411	.520	.641	.772	.878
Methanol–benzene (35°)							
x_1	0.024	0.130	0.311	0.519	0.631	0.797	0.920
y_1 (calcd.)	.245	.432	.493	.534	.561	.631	.758
y_1 (obsd.)	.273	.486	.530	.557	.579	.642	.769
Methanol–benzene (55°)							
x_1	0.030	0.103	0.330	0.498	0.608	0.790	0.901
y_1 (calcd.)	.261	.405	.498	.536	.564	.644	.751
y_1 (obsd.)	.302	.484	.554	.586	.608	.672	.770
Methanol–carbon tetrachloride (35°)							
x_1	0.135	0.356	0.478	0.494	0.656	0.791	0.912
y_1 (calcd.)	.430	.497	.519	.522	.558	.612	.724
y_1 (obsd.)	.463	.492	.503	.506	.530	.579	.702
Methanol–carbon tetrachloride (55°)							
x_1	0.058	0.149	0.365	0.495	0.645	0.790	0.909
y_1 (calcd.)	.348	.440	.501	.526	.562	.622	.733
y_1 (obsd.)	.364	.498	.528	.544	.569	.619	.734
Methyl acetate–benzene (25°)							
x_1	0.100	0.200	0.400	0.500	0.600	0.800	0.900
y_1 (calcd.)	.298	.430	.593	.659	.724	.856	.926
y_1 (obsd.)	.323	.466	.621	.681	.738	.864	.931
Methyl–acetate–benzene (35°)							
x_1	0.100	0.200	0.400	0.500	0.600	0.800	0.900
y_1 (calcd.)	.286	.420	.587	.655	.720	.854	.925
y_1 (obsd.)	.306	.448	.616	.679	.741	.866	.935
Dimethoxymethane–chloroform (25°)							
x_1	0.100	0.200	0.400	0.500	0.600	0.800	0.900
y_1 (calcd.)	.098	.243	.567	.701	.805	.936	.974
y_1 (obsd.)	.092	.222	.534	.675	.788	.933	.972
Dimethoxymethane–benzene (35°)							
x_1	0.100	0.200	0.400	0.500	0.600	0.800	0.900
y_1 (calcd.)	.341	.521	.728	.797	.852	.937	.971
y_1 (obsd.)	.342	.520	.720	.788	.845	.936	.971
Acetone–chloroform (760 mm.)							
x_1	0.138	0.211	0.477	0.663	0.739	0.859	0.915
y_1 (calcd.)	.104	.179	.520	.749	.825	.922	.957
y_1 (obsd.)	.100	.176	.517	.751	.824	.917	.952
Nitrogen–oxygen (−198°)							
x_1	0.100	0.200	0.400	0.500	0.600	0.800	0.900
y_1 (calcd.)	.376	.551	.746	.810	.862	.941	.973
y_1 (obsd.)	.417	.601	.779	.833	.877	.945	.974
Nitrogen–oxygen (−178°)							
x_1	0.100	0.200	0.400	0.500	0.600	0.800	0.900
y_1 (calcd.)	.261	.428	.648	.728	.797	.910	.957
y_1 (obsd.)	.271	.445	.666	.743	.808	.913	.958
Nitrogen–oxygen (−148°)							
x_1	0.100	0.200	0.400	0.500	0.600	0.800	0.900
y_1 (calcd.)	.169	.303	.519	.608	.693	.851	.926
y_1 (obsd.)	.173	.315	.534	.622	.702	.851	.924
Ethanol–water (50°)							
x_1	0.093	0.123	0.158	0.343	0.513	0.824	0.908
y_1 (calcd.)	.423	.455	.484	.587	.671	.857	.921
y_1 (obsd.)	.424	.482	.507	.586	.649	.845	.910
Ethanol–water (60°)							
x_1	0.086	0.197	0.375	0.527	0.808	0.860	0.972
y_1 (calcd.)	.418	.508	.589	.668	.835	.875	.972
y_1 (obsd.)	.393	.517	.596	.660	.826	.867	.972

Discussion

By far the most reliable data used in these studies are those published by Scatchard and coworkers on benzene–cyclohexane, carbon tetrachloride–benzene, carbon tetrachloride–cyclohexane, methanol–benzene, and methanol–carbon tetrachloride. It is quite apparent that the agreement between calculated and observed values for y_1 in Table II is much poorer in the cases of systems containing polar materials such as methanol. The very good agreement in the set of systems containing benzene, cyclohexane and carbon tetrachloride makes it appear that the theory is sound for non-polar materials.

The validity of equations 11a and 11b as proposed by Spinner may find some justification in the expression derived from equations 12a and 12b

$$r_1 = \frac{s_{11}}{s_{12}} e(-L_{11} + L_{12})/RT$$

and

$$r_2 = \frac{s_{22}}{s_{21}} e(-L_{22} + L_{21})/RT$$

but any such justification involves a number of unwarranted assumptions.

TEXAS CITY, TEXAS

[A CONTRIBUTION FROM THE COATES CHEMICAL LABORATORY, LOUISIANA STATE UNIVERSITY]

The Distribution of Iodine between Carbon Tetrachloride and Water and a Proposed Mechanism for Dilute, Aqueous Iodine Reactions[1]

BY RAYMOND G. WILLE AND MARY L. GOOD

RECEIVED AUGUST 20, 1956

The distribution of iodine between carbon tetrachloride and water at 25° has been studied as a function of the total iodine concentration (10^{-1} to 10^{-5} *M*); the *p*H of the aqueous phase (1.0 to 7.0); and the time of mixing. Both tracer techniques and spectrophotometric methods were used. Discrepancies between calculated and experimental distribution coefficients for low iodine concentrations are reported. Some information on the chemical behavior of the iodine species in the water phase is presented along with a mechanism to explain the anomalous behavior of dilute aqueous iodine solutions.

In a previous paper[2] an extensive study of the distribution of iodine between carbon disulfide and aqueous solutions was reported. In this study it was found that more iodine (in various chemical forms) appeared in the water phase than could be accounted for by considering the well-known iodine equilibrium reactions. The discrepancies became more pronounced as the total iodine concentration in the system was decreased. Attention was called to the fact that similar results were obtained by Kahn[3] in a study of the distribution of iodine between benzene and water. Also other investigators have found anomalous behavior in dilute iodine solutions. Examples are (1) the studies made by Reid and Mulliken[4] on dilute solutions of iodine in pyridine where excess triiodide appeared as the iodine concentration was decreased; and (2) the investigation of the ionization constant of iodine by Katzin[5] where the value obtained for the constant increased as the iodine concentration decreased. No definite conclusions were made to account for results reported in these cases. However, it was generally agreed that either impurities were playing an important role in these studies or there was some unknown reaction occurring at low iodine concentrations between iodine and the solvent in its "brown" solutions. The results of the carbon disulfide–water distribution study[2] indicated that the anomalous behavior was quite reproducible. This is not generally a characteristic of impurity reactions. Thus it seems the most likely explanation is that some iodine reaction, heretofore not taken into account, becomes important at low iodine concentrations.

To further study this interesting iodine behavior, a distribution study of iodine between carbon tetrachloride and aqueous solutions was carried out. Tracer techniques were employed to follow the distribution so that the lowest possible iodine concentrations could be used.

Experimental

The experimental procedure described previously[2] for the distribution study of iodine in the carbon disulfide–water system was essentially repeated. Reagent grade carbon tetrachloride was purified further by a method described by Edwards and Davies.[6] Buffers at *p*H 6.98, 5.01 and 2.98 were prepared using 0.1 *M* citric acid and 0.2 *M* disodium phosphate. A dilute solution of sulfuric acid was used for *p*H 1.02. All buffer systems were made up with conductivity water. A Beckman Model G *p*H meter was used to determine the *p*H of the buffer solutions. U. S. P. iodine (resublimed) was used to prepare carrier solutions of iodine in carbon tetrachloride. All carrier solutions were diluted from a 0.1 *M* stock solution. Eight day I^{131} obtained from the Oak Ridge National Laboratory in a "carrier-free" state, in a sodium sulfite solution, was used as a tracer. The radioactive solutions to be used in the distributions were obtained by shaking the carbon tetrachloride solutions of carrier iodine with diluted solutions of the Oak Ridge I^{131}. The two phases were separated leaving the iodine in the carbon tetrachloride phase tagged with I^{131} due to the exchange between the active iodide ions and the I_2 molecules. Radioactivity measurements were made on liquid samples using a well type NaI (thallium activated) scintillation counter. A Dumont 6292 photomultiplier tube; a Nuclear Corporation Power supply, Model 1090A; a Radiation Counter Laboratories linear amplifier, Model 2206; and a Nuclear Corporation scaling unit, Model 161, were used.

The distributions were made by adding 20 ml. of carbon tetrachloride containing radioactive iodine to an equal volume of buffered water in a 100-ml. glass-stoppered flask. The flask was immersed in a water-bath controlled at 25 ± 1°. The flask was mechanically shaken at a constant rate. The solutions were shaken for varying lengths of time from two minutes to several hours. After each period of mixing the layers were allowed to separate and equal volumes of solution were removed from each phase and counted. To obtain the reproducibility a second series of distributions were made. Results of the two separate runs agreed within experimental error (approximately 10%).

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(1) This work was supported in part by a Frederick Gardner Cottrell Grant from the Research Corporation. Abstracted from a portion of the thesis submitted by Raymond G. Wille to the Graduate School of the Louisiana State University, Baton Rouge, in partial fulfillment of the requirements for the degree of Master of Science.

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