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Isobaric Vapor–Liquid Equilibrium for Dimethylsulfoxide with Chloroethanes and Chloroethenes

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The vapor–liquid equilibrium (VLE) and boiling point data have been measured at 95.3 kPa for the binary systems of dimethylsulfoxide (DMSO) with 1,2-dichloroethane, 1,1,1-trichloroethane, 1,1,2,2-tetrachloroethane, trichloroethene, and tetrachloroethene using a Swietoslawski-ebulliometer. The experimental results are analyzed in terms of intermolecular interactions between constituent molecules. The experimental temperature−composition (T, x) data were correlated with the Wilson, NRTL, and UNIQUAC models.

Introduction

Dimethylsulfoxide (DMSO) is a dipolar, aprotic, hygroscopic solvent for which a large number of pharmacologic properties have been claimed. This colorless liquid is an important solvent that dissolves both polar and nonpolar compounds and is miscible in a wide range of organic solvents as well as water. DMSO as a solvent has a high boiling point, thus its solutions are not typically evaporated but instead diluted to isolate the reaction product. It is a versatile organic liquid having a special solvent power to promote a chemical reaction when used as a reaction medium. It also exerts a solvent effect sufficient to accelerate a reaction brought about by another reagent. It is also used as a solvent for polymerization reaction displacement reactions because of its high dielectric constant value ($\epsilon = 46.45$).[1] Moreover, it is a highly polar liquid ($\mu = 4.06$)[1] and has the ability to participate in hydrogen bonding.

Chlorinated compounds are extensively used in industrial, agricultural, and commercial processes as solvents, lubricants, and intermediates in chemical industry, pesticides, pharmaceuticals, and medical equipment. The physical and chemical properties of chloroethenes differ strikingly from those of saturated aliphatic chlorine compounds. In recent years, the thermodynamic properties of highly polar compounds with chloroethanes and chloroethenes have received increasing attention because they serve as model compounds in biochemical considerations.[2–5] DMSO, chloroethanes, and chloroethenes are important liquids which find a variety of applications such as solvents for lacquers, oils, and resins.[4] These liquids were chosen in the present investigation on the basis of their industrial importance. Thermodynamic properties of these solvents are of interest because of the effects of chlorine with the S−O group, present in the DMSO, on the molecular interactions.

Vapor–liquid equilibria (VLE) data are required for design and operation of distillation equipment and for determining the optimal values of parameters in the thermodynamic models. In general, the VLE data are obtainable under two conditions, isothermal and isobaric. As part of our research program to explore the VLE of various molecular liquids,[3,6–9] we report here the VLE measurements for the systems of dimethylsulfoxide with 1,2-dichloroethane, 1,1,1-trichloroethane, 1,1,2,2-tetrachloroethane, trichloroethene, and tetrachloroethene at 95.3 kPa. A survey of the literature reveals no studies of VLE data for these mixtures. The Wilson,[10] NRTL,[11] and UNIQUAC[12] models were used to correlate the experimental VLE data of the binary systems.

Table 1. Densities (ρ) at 303.15 K and Boiling Point Temperatures (T) at 101.3 kPa (760 mmHg)

	ρ/g·cm^{-3}		T/K	
component	exptl	lit.	exptl	lit.[1]
DMSO	1.09040	1.090 42[14]	462.19	462.15
1,2-dichloroethane	1.238 41	1.238 43[2]	356.95	356.63
1,1,1-trichloroethane	1.320 97	1.320 94[2]	347.35	347.23
1,1,2,2-tetrachloroethane	1.578 59	1.578 57[2]	418.35	418.13
trichloroethene	1.451 42	1.451 39[2]	360.25	360.34
tetrachloroethene	1.606 39	1.606 41[2]	394.15	394.22

Experimental Section

Materials. In the present study, all the chemicals used were of analytical grade and purchased from commercial sources. DMSO, chloroethanes, and chloroethenes were purified by the standard methods described by Riddick et al.[1] The purity of the samples was checked by measuring densities and boiling points. The densities were measured using a standard bicapillary pycnometer, giving the uncertainty of 2 parts in 10^5 due to the mass measurements of liquids. Boiling points (Hg) were measured by a Swietoslawski-type ebulliometer[13] with the uncertainty of ± 0.2 K. The purities of the samples were further confirmed by GLC single sharp peaks. Good agreement between the measured densities as well as boiling points and literature values[1,2,14] is shown in Table 1.

VLE Measurements. The vapor–liquid equilibrium measurements were performed by a Swietoslawski-ebulliometer.[13] The detailed procedure has been discussed in our earlier articles.[6,7] The ebulliometer is connected to a vacuum pump and a dry nitrogen gas cylinder with a closed end manometer in line to enable the measurement and maintenance of the total pressure of the system at 95.3 kPa, by adjusting the opening of the needle valve of the gas cylinder/the opening of the bypass line of the vacuum pump. An open end manometer provided in line is frequently read (along with a Fortin's-type barometer), and the

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Table 2. Parameters of the Extended Antoine Equation[a]

compound	C_1	C_2	C_3	C_4	C_5	C_6	C_7	C_8/K	C_9/K
DMSO[15,16]	49.37	−7620.6	0	0	−4.63	4.38E−7	2	291.67	729.00
1,2-dichloroethane[17,18]	85.45	−6920.4	0	0	−10.65	9.14E−6	2	237.49	561.60
1,1,1-trichloroethane[17,19]	76.31	−6281.5	0	0	−9.33	7.87E−6	2	243.10	545.00
1,1,2,2-tetrachloroethane[17,20,21]	46.43	−6570.3	0	0	−4.33	2.31E−7	2	229.35	645.00
trichloroethene[22,23]	52.50	−5471.6	0	0	−5.83	4.51E−3	1	188.40	571.00
tetrachloroethene[24,25]	51.86	−6191.2	0	0	−5.33	2.13E−6	2	250.80	620.00

[a] Extended Antoine equation: $\ln(P^S) = C_1 + (C_2/(T + C_3)) + C_4T + C_5 \ln T + C_6T^{C_7}$ for $C_8 < T < C_9$, where P^S is in kPa and T is in K. The values of the coefficients were taken from the Aspen property databank.

Table 3. T, x Data for the Systems of Chloroethanes and Chloroethenes (1) with Dimethylsulfoxide (2) at 95.3 kPa

x_1	T_{exptl}/K	$y_{1,\text{calcd}}$	$\gamma_{1,\text{calcd}}$	$\gamma_{2,\text{calcd}}$
1,2-Dichloroethane (1) + DMSO (2)				
0.0	460.95	0.0		1
0.0748	436.85	0.5357	0.9341	0.9996
0.1307	423.25	0.7133	0.9392	0.9987
0.3107	395.25	0.9142	0.9573	0.9919
0.4286	383.85	0.9546	0.9689	0.9839
0.5197	376.95	0.9714	0.9771	0.9756
0.6433	369.55	0.9848	0.9867	0.9612
0.8440	360.45	0.9957	0.9973	0.9299
0.9253	357.45	0.9983	0.9994	0.9143
1.0	355.05	1.0	1	
1,1,1-Trichloroethane (1) + DMSO (2)				
0.0	460.95	0.0		1
0.0728	428.15	0.6432	1.1559	0.9997
0.1741	402.05	0.8673	1.1259	0.9977
0.3598	376.55	0.9623	1.1114	0.9866
0.4574	367.95	0.9781	1.1116	0.9768
0.5880	359.25	0.9890	1.1122	0.9620
0.7405	351.85	0.9951	1.1015	0.9649
0.8771	347.45	0.9978	1.0639	1.1233
0.9235	346.45	0.9984	1.0410	1.3703
1.0	345.45	1.0	1	
1,1,2,2-Tetrachloroethane (1) + DMSO (2)				
0.0	460.95	0.0		1
0.0728	454.55	0.2295	1.2148	1.0044
0.1447	449.35	0.3761	1.1229	1.0137
0.2880	441.35	0.5758	1.0370	1.0340
0.3829	437.05	0.6729	1.0139	1.0445
0.4774	433.25	0.7520	1.0031	1.0514
0.6190	428.15	0.8459	0.9980	1.0550
0.8025	422.45	0.9343	0.9988	1.0487
0.9125	419.35	0.9739	0.9997	1.0402
1.0	417.05	1.0	1	
Trichloroethene (1) + DMSO (2)				
0.0	460.95	0.0		1
0.0918	445.85	0.4326	0.5335	0.9873
0.1653	432.15	0.6550	0.6126	0.9620
0.3036	408.75	0.8738	0.7452	0.8910
0.3480	402.45	0.9080	0.7822	0.8648
0.6132	376.15	0.9837	0.9375	0.7098
0.7255	369.35	0.9921	0.9716	0.6528
0.8409	363.75	0.9967	0.9914	0.6006
0.9275	360.25	0.9988	0.9984	0.5655
1.0	357.75	1.0	1	
Tetrachloroethene (1) + DMSO (2)				
0.0	460.95	0.0		1
0.0512	444.95	0.4242	2.0897	1.0019
0.0948	435.35	0.5763	1.9946	1.0066
0.2189	418.95	0.7681	1.7340	1.0375
0.3133	411.85	0.8315	1.5625	1.0810
0.4814	404.35	0.8899	1.3210	1.2148
0.6256	400.25	0.9200	1.1718	1.4167
0.8069	396.25	0.9525	1.0496	1.8899
0.9123	393.95	0.9749	1.0111	2.3926
1.0	391.95	1.0	1	

needed corrective action of adjusting the opening of the needle valve attached to the gas cylinder or the bypass line of the vacuum pump is performed according to the requirement. The pressure was controlled by a Cartesian driver and was measured with this mercury manometer with an uncertainty of ± 0.13 kPa (1 mmHg). A mercury-in-glass thermometer, calibrated by means of point to point comparison with a platinum resistance thermometer (certified by the National Institute of Standards and Technology, Boulder, USA), is used to measure the equilibrium temperature to the uncertainty of ± 0.1 K. The thermometer is placed in a thermo well (in the apparatus) whose outer surface is constantly impinged by an equilibrium vapor–liquid mixture. The thermo well contains a small quantity of mercury to provide for good contact.

Each solvent was degassed by magnetic stirring under its vapor pressure before mixing. The boiling points of mixtures of DMSO with chloroethanes and chloroethenes were determined at 95.3 kPa over the entire range of compositions; mixtures of different compositions were prepared gravimetrically by weighing the needed quantities of the pure components, making use of a Mettler balance uncertainty to 0.0001 g. Boiling points of mixtures were also measured using an ebulliometer, giving the uncertainty of ± 0.2 K. The uncertainty in solution composition was $\pm\ 1 \cdot 10^{-4}$ in mole fraction. The heating rate is maintained at a level capable of producing a condensate drop rate of 30 drops per minute, following the suggestion of Hala et al.,[13] by carefully adjusting the energy supply to the heater. Equilibrium temperature is recorded, when the steady-state conditions (observation of constant temperature and uniform boiling rate indicated by the maintenance of the chosen condensate liquid drop rate of 30 drops per minute) are observed for at least 30 min.

Results and Discussion

The isobaric VLE data (T and x_i) were correlated with the $\gamma-\varphi$ method by using one of the activity coefficient models (Wilson,[10] NRTL,[11] or UNIQUAC[12]) and assuming that the vapor phase was ideal gas mixtures. The optimal values of the binary parameters were determined by the minimization of the following objective function π

$$\pi = \sum_{k=1}^{n} \left\{ \left[\frac{(P_k^{\text{calcd}} - P_k^{\text{exptl}})}{\sigma_P} \right]^2 + \left[\frac{(T_k^{\text{calcd}} - T_k^{\text{exptl}})}{\sigma_T} \right]^2 + \left[\frac{x_{1,k}^{\text{calcd}} - x_{1,k}^{\text{exptl}}}{\sigma_{x_1}} \right]^2 \right\} \quad (1)$$

where T_k^{exptl} and T_k^{calcd} are the experimental and the calculated boiling point temperatures, respectively, and summation is performed over all experimental points. In the above VLE calculations, the standard deviation σ was set to 0.01 kPa for pressure, 0.03 K for temperature, and 0.0002 for liquid composition.

The Antoine constants for vapor pressures of the pure components were taken from the Aspen property databank and are presented in Table 2. The vapor pressure data of each

Table 4. Wilson, NRTL, and UNIQUAC Correlated Parameters at 95.3 kPa for the Systems Studied

system	model	parameter/K		rmsd ΔT/K[a]	rmsd ΔP/kPa[a]	rmsd Δx_1[a]
1,2-dichloroethane + DMSO	Wilson	$\Delta\lambda_{12}/R$	51.55	0.07	0.003	0.0002
		$\Delta\lambda_{21}/R$	−17.48			
	NRTL[b]	$\Delta g_{12}/R$	37.14	0.07	0.003	0.0002
		$\Delta g_{21}/R$	−70.69			
	UNIQUAC	$\Delta u_{12}/R$	−29.88	0.07	0.003	0.0001
		$\Delta u_{21}/R$	42.57			
1,1,1-trichloroethane + DMSO	Wilson	$\Delta\lambda_{12}/R$	346.77	0.04	0.002	0.0004
		$\Delta\lambda_{21}/R$	−1258.74			
	NRTL[b]	$\Delta g_{12}/R$	959.42	0.53	0.021	0.0020
		$\Delta g_{21}/R$	−458.68			
	UNIQUAC	$\Delta u_{12}/R$	−680.70	0.32	0.013	0.0011
		$\Delta u_{21}/R$	322.30			
1,1,2,2-tetrachloroethane + DMSO	Wilson	$\Delta\lambda_{12}/R$	−563.57	0.35	0.015	0.0005
		$\Delta\lambda_{21}/R$	296.18			
	NRTL[b]	$\Delta g_{12}/R$	−414.72	0.31	0.013	0.0004
		$\Delta g_{21}/R$	675.50			
	UNIQUAC	$\Delta u_{12}/R$	212.26	0.35	0.015	0.0005
		$\Delta u_{21}/R$	−319.43			
trichloroethene + DMSO	Wilson	$\Delta\lambda_{12}/R$	−269.95	0.21	0.010	0.0022
		$\Delta\lambda_{21}/R$	410.81			
	NRTL[b]	$\Delta g_{12}/R$	−514.06	0.29	0.013	0.0015
		$\Delta g_{21}/R$	380.99			
	UNIQUAC	$\Delta u_{12}/R$	262.77	0.24	0.010	0.0016
		$\Delta u_{21}/R$	−228.70			
tetrachloroethene + DMSO	Wilson	$\Delta\lambda_{12}/R$	−125.19	0.73	0.030	0.0039
		$\Delta\lambda_{21}/R$	−333.24			
	NRTL[b]	$\Delta g_{12}/R$	360.99	0.74	0.030	0.0040
		$\Delta g_{21}/R$	71.76			
	UNIQUAC	$\Delta u_{12}/R$	−307.78	0.73	0.030	0.0039
		$\Delta u_{21}/R$	109.48			

[a] rmsd $\Delta M = \sqrt{1 / n_P \sum_{k=1}^{n_P} (M_k^{\text{calcd}} - M_k^{\text{exptl}})^2}$, where n_p is the number of data points and M represents T, P, x_1. [b] The value of α was fixed as 0.3 for each binary system.

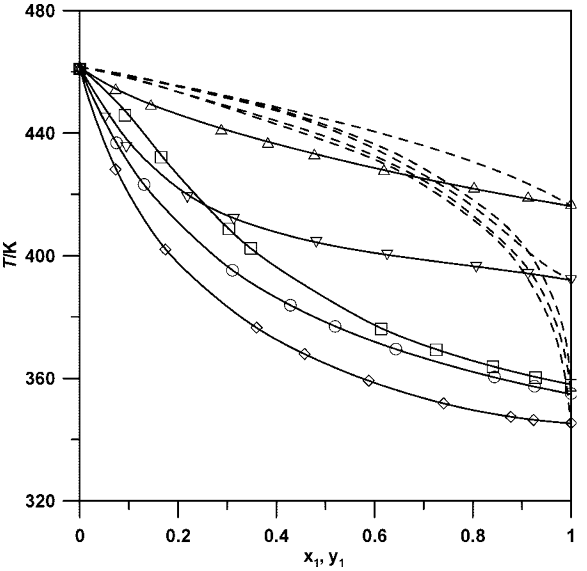

Figure 1. Experimental boiling points at 95.3 kPa against mole fraction of chloroethanes and chloroethenes (x_1) for the mixtures of ○, DMSO + 1,2-dichloroethane; or ◇, + 1,1,1-trichloroethane; or △, + 1,1,2,2-tetrachloroethane; or □, + trichloroethene; or ▽, + tetrachloroethene. —, The calculated liquid composition from the Wilson model and -----, the calculated vapor composition.

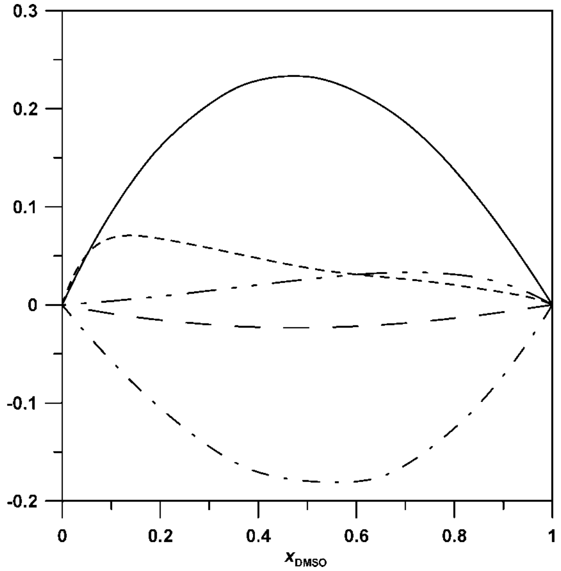

Figure 2. Excess Gibbs free energy against mole fraction of DMSO for ——, DMSO + 1,2-dichloroethane; ------, DMSO + 1,1,1-trichloroethane; — - - - —, DMSO + 1,1,2,2-tetrachloroethane; — - —, DMSO + trichloroethene; and —, DMSO + tetrachloroethene.

constituent component are available from the literature.[15–25] The related references are also given in Table 2. The experimental and calculated results from the Wilson model are presented in Table 3, while the optimized values of the parameters, including $\Delta\lambda_{12}$ and $\Delta\lambda_{21}$ of the Wilson, Δg_{12}, Δg_{21}, and α of the NRTL, and Δu_{12} and Δu_{21} of the UNIQUAC, are collected in Table 4. The boiling point temperature versus mole fraction curves are presented graphically in Figure 1. The smooth curves in the graph are the calculated values from the Wilson models for both the liquid and vapor phases. For the sake of clarity, we did not show here the other two models, since three models are merging. Close observation of Figure 1 reveals that the curves of three models are overlapping one another. All these three models with the tabulated parameters represent good agreement with the VLE properties of DMSO with chloroethanes and chloroethenes at 95.3 kPa, except for the system of DMSO with tetrachloroethene, which exhibits larger deviations than those of the other investigated systems.

The results as reported in Table 3 show very different deviations from ideality for the investigated systems. As we can see from the results, the binary systems containing 1,2-dichloroethane, 1,1,1-trichloroethane, and 1,1,2,2-tetrachloroethane behave as near ideal solutions. However, the chloroethene systems reveal large deviations from ideal solutions. Negative deviations are exhibited in the trichloroethene + DMSO system, whereas positive deviations are found in the tetrachloroethene + DMSO system. The probable explanation is the structural arrangement of the chlorine molecules situated around the carbon atoms and unsaturation of the ethane molecules. Moreover, the chloroethene systems present larger deviations. There is a pronounced decrease in reactivity of a chlorine atom on an ethylenic carbon, since they possess double bond character in their structures.

Using the Wilson parameters, we calculated the excess Gibbs Free energies (G^E) from the following equation

$$\frac{G^E}{RT} = x_1 \ln \gamma_1 + x_2 \ln \gamma_2 \qquad (2)$$

The excess Gibbs free energies versus mole fraction of DMSO are presented graphically in Figure 2. The G^E/RT values are negative over the entire range of compositions for the mixtures of DMSO with 1,2-dichloroethane and trichloroethene. However, the quantity is positive in the binary mixtures of DMSO with 1,1,1-trichloroethane, 1,1,2,2-tetrachloroethane, and tetrachloroethene. At the same concentration, the algebraic values of G^E for the systems of DMSO with chloroethanes and chloroethenes fall in the order:

tetrachloroethane > 1,1,1-trichloroethane > 1,1,2,2-tetrachloroethane > 1,2-dichloroethane > trichloroethene.

Conclusions

Isobaric VLE data have been determined experimentally for binary mixtures composed of DMSO with chloroethanes and chloroethenes at 95.3 kPa over the entire range of compositions. The Wilson, NRTL, and UNIQUAC models are capable of accurately correlating the VLE data of the investigated systems. The reported experimental results are satisfactorily correlated with these three models, except DMSO with tetrachloroethene, which exhibits larger deviations than those of the other investigated systems.

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