

$$\tau_{12} = (1/RT)(C_2 + D_2(273.15 - T))$$

$$\alpha_{12} = C_3 + D_3(273.15 - T)$$

Modified Wilson Equation

$$G^E/RT = -x_1 \ln(x_1 + x_2 \Lambda_{21}) - x_2 \ln(x_1 \Lambda_{12} + x_2) + x_1 \ln(x_1 + x_2 \rho_{21}) + x_2 \ln(x_1 \rho_{12} + x_2)$$

$$\ln \gamma_1 = -\ln(x_1 + x_2 \Lambda_{21}) + x_2 \left[\frac{\Lambda_{21}}{x_1 + x_2 \Lambda_{21}} - \frac{\Lambda_{12}}{x_1 \Lambda_{12} + x_2} \right] + \ln(x_1 + x_2 \rho_{21}) - x_2 \left[\frac{\rho_{21}}{x_1 + x_2 \rho_{21}} - \frac{\rho_{12}}{x_1 \rho_{12} + x_2} \right]$$

$$\ln \gamma_2 = -\ln(x_1 \Lambda_{12} + x_2) - x_1 \left[\frac{\Lambda_{21}}{x_1 + x_2 \Lambda_{21}} - \frac{\Lambda_{12}}{x_1 \Lambda_{12} + x_2} \right] + \ln(x_1 \rho_{12} + x_2) + x_1 \left[\frac{\rho_{21}}{x_1 + x_2 \rho_{21}} - \frac{\rho_{12}}{x_1 \rho_{12} + x_2} \right]$$

where

$$\rho_{12} = V_1^L/V_2^L$$

$$\rho_{21} = V_2^L/V_1^L$$

$$\Lambda_{21} = \rho_{21} \exp[-(1/RT)(C_1 + D_1(273.15 - T))]$$

$$\Lambda_{12} = \rho_{12} \exp[-(1/RT)(C_2 + D_2(273.15 - T))]$$

Glossary

C_1 – C_3	constants of the NRTL or modified Wilson equations at 273.15 K
D_1 – D_3	constants expressing the temperature dependence of the NRTL or modified Wilson constants
G	Gibbs function/J
G^E	excess Gibbs function/J mol ⁻¹
R	gas constant = 8.3143 J K ⁻¹ mol ⁻¹
T	temperature/K (IPTS 68)
T_c	critical temperature/K
P	pressure/N m ⁻²

P_i^0	vapor pressure of component i /N m ⁻²
P_c	critical pressure/N m ⁻²
V_i^L	molar volume of component i /m ³ mol ⁻¹
V_c	critical volume/m ³ mol ⁻¹
x_i	liquid-phase mole fraction of component i
y_i	vapor-phase mole fraction of component i

Greek Letters

α_{ij}	constant in NRTL equation
Λ_{ij}	constant in modified Wilson equation
η	vapor-phase association factor
μ	dipole moment/D
ρ_{ij}	constant in the modified Wilson equation = V_i^L/V_j^L
τ_{ij}	constant in the NRTL equation
ω	acentric factor
ω_H	acentric factor of homomorph

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Several Properties of the Dimethyl Sulfoxide–3-Methyl-2-oxazolidone System as Functions of Composition and Temperature

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Dielectric constants, viscosities, densities, and refractive indices for 11 binary mixtures of dimethyl sulfoxide and 3-methyl-2-oxazolidone were measured at 10 °C intervals within the range of 25–75 °C. The experimental data, excepting refractive indices, were fitted precisely to appropriate equations as separate functions of temperature and composition. Molar refractions also are discussed.

Introduction

The utility of dimethyl sulfoxide (Me₂SO) as an outstanding solvent for both electrolytes and nonelectrolytes has become

well established and known during the last 2 decades. Studies in this laboratory within the last few years (1, 5, 6) have indicated that 3-methyl-2-oxazolidone (3Me₂Ox), which has a dielectric constant essentially equal to that of water, similarly appears to have great potential as an electrolytic solvent. Me₂SO and 3Me₂Ox have dielectric constants of 46.45 and 77.54 and viscosities of 1.991 and 2.450 cP, respectively, at 25 °C. Both Me₂SO and 3Me₂Ox can be highly purified through a series of fractional freezings under nitrogen. The useful data reported herein concerning dielectric constants, viscosities, and other physical properties of Me₂SO–3Me₂Ox mixtures were obtained in the course of systematic physicochemical investigations dealing with pure and mixed nonaqueous media.

Experimental Section

Materials. Me₂SO was obtained commercially (Mallinckrodt Analytical Reagent). 3Me₂Ox was available from a laboratory supply which had been prepared as described previously (6). Me₂SO and 3Me₂Ox were purified similarly through a sequence of fractional freezings under nitrogen (4) until a product with a constant, maximum freezing point was obtained, i.e., 18.5 and 15.9 °C for Me₂SO and 3Me₂Ox, respectively. Each Me₂SO–3Me₂Ox mixture was prepared in a glass-stoppered flask on a weight basis in sufficient quantity to permit a separate portion to be used for each measurement.

Experimental Measurements. A General Radio Type 821-A Twin-T impedance measuring assembly (9, 11) was used in conjunction with two-terminal parallel plate cells for capacitance measurements at 1 MHz. The assembly and cells were the same as those used in previous studies (2, 7). The principal aspects for determining cell constants and for calculating dielectric constants have been described previously (9, 13). The standard media used in the determination of cell constants were air and water which have dielectric constants of 1.0005 and 78.304 (10), respectively, at 25 °C.

The viscosities were determined by using sizes 25 and 50 Cannon-Ubbelohde viscometers which previously were calibrated by the Cannon Instrument Co. Efflux times always exceeded 200 s, making unnecessary any corrections due to kinetic energy effects. To minimize atmospheric contamination, a dry nitrogen atmosphere was maintained over the experimental liquids in the viscometers which were equipped with top adapters and vented through tubes filled with Drierite and Ascarite. Efflux times were measured to 0.1 s by using electric timers. The viscosities were reported relative to a viscosity of 1.0019 cP for water at 20 °C (12).

Densities were determined pycnometrically and dilatometrically with 15-mL pycnometer–dilatomers which had been calibrated using freshly distilled water at 25 °C and are based on 0.997 04 g cm⁻³ as the density of water at this temperature. Volumes of the pycnometer–dilatomers at higher temperatures were calculated with a standard volume expansion formula by using $9.6 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ as the cubical coefficient of expansion of Pyrex glass (8). Excess liquid in a pycnometer–dilatomer was removed when necessary by strong suction through a fine capillary. Buoyancy corrections were applied to all experimental weights prior to calculations of densities (8).

Refractive indices were measured with Spencer Abbe Model 2214 refractometers equipped with sodium D line compensating prisms and with jackets through which water from a constant-temperature bath could be rapidly circulated.

Measurements of dielectric constants, viscosities, densities, and refractive indices were made in duplicate with precisions and estimated equivalent accuracies of 0.2, 0.2, 0.02, and 0.02%, respectively.

The temperature for the dielectric constant, viscosity, and density determinations was controlled within 0.04 °C with a Cannon Model H-1 constant-temperature bath filled with light paraffin oil. The bath thermometers with 0.1 °C subdivisions were compared with a similar thermometer for which NBS calibration data were available. Appropriate corrections were applied, the largest being +0.13 °C (stem correction of +0.22 °C plus a scale correction of -0.09 °C) at a bath temperature of 75 °C.

Mean values of the experimental data for each mixture were fitted, as functions of temperature, to various equations by least-squares analysis with an IBM 370-165-II computer. These equations then were used to calculate small corrections which were applied to the actual experimental values of the physical properties in converting these values from the slightly nonintegral temperatures at which the measurements were made to the integral temperatures shown in Table I ($\Delta T < 0.30 \text{ }^\circ\text{C}$). The

Table I. Experimental Data

$10^2 X_2$	25 °C	35 °C	45 °C	55 °C	65 °C	75 °C
Dielectric Constants (1 MHz)						
0.00 ^a	46.45	45.18	43.89	42.60	41.30	40.02
4.65	48.01	46.62	45.21	43.82	42.43	41.06
8.41	49.50	48.05	46.61	45.18	43.77	42.38
16.76	52.53	50.92	49.32	47.75	46.21	44.69
24.19	55.28	53.54	51.82	50.11	48.44	46.79
34.15	58.51	56.46	54.50	52.61	50.80	49.07
42.75	61.40	59.22	57.12	55.08	53.13	51.24
54.01	64.89	62.44	60.08	57.81	55.61	53.50
63.95	67.74	65.09	62.57	60.17	57.88	55.70
75.24	70.90	68.09	65.41	62.85	60.41	58.08
87.85	74.20	71.25	68.42	65.70	63.09	60.58
95.14	75.99	72.94	70.04	67.26	64.60	62.07
100.00 ^b	77.54	74.31	71.28	68.44	65.76	63.24
Densities, g cm ⁻³						
0.00 ^a	1.0954	1.0854	1.0754	1.0654	1.0553	1.0452
4.65	1.0993	1.0894	1.0794	1.0693	1.0593	1.0492
8.41	1.1023	1.0923	1.0824	1.0724	1.0624	1.0524
16.76	1.1092	1.0993	1.0894	1.0795	1.0695	1.0595
24.19	1.1152	1.1053	1.0955	1.0856	1.0758	1.0659
34.15	1.1229	1.1132	1.1034	1.0936	1.0839	1.0741
42.75	1.1293	1.1196	1.1100	1.1003	1.0905	1.0808
54.01	1.1375	1.1279	1.1183	1.1087	1.0991	1.0894
63.95	1.1451	1.1354	1.1257	1.1161	1.1065	1.0970
75.24	1.1529	1.1434	1.1338	1.1243	1.1147	1.1052
87.85	1.1616	1.1521	1.1426	1.1331	1.1236	1.1140
95.14	1.1669	1.1574	1.1479	1.1384	1.1289	1.1195
100.00 ^b	1.1702	1.1608	1.1515	1.1421	1.1328	1.1235
Viscosities, cP						
0.00 ^a	1.991	1.648	1.391	1.194	1.039	0.915
4.65	2.004	1.663	1.404	1.204	1.048	0.925
8.41	2.012	1.673	1.413	1.212	1.055	0.931
16.76	2.037	1.698	1.437	1.236	1.077	0.951
24.19	2.060	1.720	1.459	1.256	1.097	0.969
34.15	2.090	1.751	1.490	1.286	1.124	0.995
42.75	2.126	1.782	1.518	1.311	1.148	1.017
54.01	2.171	1.826	1.558	1.349	1.182	1.048
63.95	2.218	1.872	1.603	1.387	1.215	1.077
75.24	2.280	1.923	1.645	1.426	1.252	1.111
87.85	2.359	1.990	1.703	1.478	1.298	1.153
95.14	2.415	2.038	1.744	1.513	1.329	1.180
100.00 ^b	2.450	2.067	1.770	1.536	1.349	1.197
Refractive Indices						
0.00 ^a	1.4771					
4.65	1.4748	1.4707	1.4660			
8.41	1.4735	1.4695	1.4646			
16.76	1.4707	1.4668	1.4625			
24.19	1.4690	1.4646	1.4611			
34.15	1.4666	1.4621	1.4582			
42.75	1.4643	1.4604	1.4566			
54.01	1.4619	1.4582	1.4542			
63.95	1.4600	1.4559	1.4522			
75.24	1.4576	1.4540	1.4502			
87.85	1.4549	1.4519	1.4482			
95.14	1.4536	1.4506	1.4470			
100.00 ^b	1.4522					

^a Reference 3. ^b Reference 6.

values in Table I, therefore, are experimental values to which small smoothed corrections or adjustments have been applied to facilitate a convenient, more useful presentation of the data at integral temperatures.

Results and Discussion

The experimental data are summarized in Table I; the data for pure Me₂SO and pure 3Me₂Ox were obtained in earlier studies in this laboratory and were taken from ref 3 and 6, respectively.

The dielectric constant data for each experimental mixture as a function of temperature were fitted to the empirical equation

$$\epsilon = A + B/T + C/T^2 \quad (1)$$

Table II. Results for Dielectric Constant Data Fitted to Equation 1

$10^2 X_2$	$-A$	$B \times 10^{-4}$	$-C \times 10^{-6}$	% dev $\times 10^2$ ^c	
				mean	max
0.00 ^a	40.574	4.064 26	4.380 87	2	5
4.65	41.180	4.079 35	4.233 69	3	6
8.41	38.498	3.961 71	3.989 61	4	9
16.76	40.538	4.113 97	3.992 51	2	5
24.19	46.343	4.509 74	4.411 91	4	6
34.15	28.100	3.308 98	2.166 71	3	6
42.75	36.350	3.853 51	2.799 28	3	6
54.01	44.279	4.294 64	3.099 81	6	11
63.95	32.876	3.584 10	1.741 43	7	15
75.24	38.894	3.987 25	2.127 71	4	4
87.85	50.539	4.761 90	3.109 09	2	6
95.14	44.577	4.417 60	2.453 35	2	4
100.00 ^b	22.01	2.968 2		7	16

^a Reference 3. ^b Reference 6. ^c % deviation = $100|\epsilon_{\text{exptl}} - \epsilon_{\text{calcd}}|/\epsilon_{\text{exptl}}$.

Table III. Results for Dielectric Constant Data Fitted to Equation 2

temp, °C	J	K	$-P$	Q	% dev $\times 10^2$ ^a	
					mean	max
25	46.32	39.07	10.04	2.02	13	29
35	45.05	36.95	10.37	2.58	13	28
45	43.76	35.20	11.07	3.35	13	35
55	42.46	33.60	11.75	4.10	14	40
65	41.15	32.38	12.96	5.16	15	47
75	39.85	31.24	14.01	6.09	17	53

^a % deviation = $100|\epsilon_{\text{exptl}} - \epsilon_{\text{calcd}}|/\epsilon_{\text{exptl}}$.

The values obtained for A , B , and C through least-squares analyses of the data are summarized in Table II. The excellent fitting of the data to eq 1 is manifest from the average and maximum deviations between experimental and calculated values never exceeding 0.07 and 0.16%, respectively.

At each temperature the dielectric constant data as a function of composition were fitted to a polynomial equation of the type

$$\epsilon = J + KX_2 + PX_2^2 + QX_2^3 \quad (2)$$

The least-squares values for the constants in eq 2 are compiled in Table III, which also contain other data showing that the mean and maximum deviations between experimental and calculated values never exceed 0.17 and 0.53%, respectively.

The density data for each Me₂SO–3Me₂Ox mixture as a function of temperature can be fitted very precisely to the equation

$$\rho = a + bt + ct^2 \quad (3)$$

The calculated least-squares values for a , b , and c are summarized in Table IV, which provides additional data showing that the mean and maximum deviations between experimental and calculated densities never exceed 0.011 and 0.026%, respectively.

At each temperature the density data as a function of composition were fitted within 0.032% using a polynomial equation of the type

$$\rho = j + kX_2 + pX_2^2 + qX_2^3 \quad (4)$$

The least-squares values for the constants in eq 4 and for the deviations between experimental and calculated densities are compiled in Table V.

The temperature coefficient of refractive index, dn_D/dt , is approximately $-4 \times 10^{-4} \text{ }^\circ\text{C}^{-1}$ over the entire composition range of the Me₂SO–3Me₂Ox system. This magnitude of temperature coefficient is typical of most organic systems.

Table IV. Results for Density Data Fitted to Equation 3

$10^2 X_2$	a	$-b \times 10^4$	$-c \times 10^7$	% dev $\times 10^3$ ^c	
				mean	max
0.00 ^a	1.120 32	9.9431	0.887	7	15
4.65	1.124 10	9.8674	1.665	6	13
8.41	1.127 16	9.9304	0.580	5	10
16.76	1.133 78	9.7897	1.508	8	18
24.19	1.139 76	9.8200	0.413	10	25
34.15	1.147 31	9.7494	0.178	5	26
42.75	1.153 37	9.6012	0.987	5	12
54.01	1.161 55	9.5956	0.270	7	16
63.95	1.169 55	9.8549	-2.454	4	9
75.24	1.176 84	9.5718	-0.269	5	11
87.85	1.185 32	9.4931	0.134	6	12
95.14	1.190 56	9.4789	0.025	7	17
100.00 ^b	1.193 5	9.3400		7	11

^a Reference 3. ^b Reference 6. ^c % deviation = $100|\rho_{\text{exptl}} - \rho_{\text{calcd}}|/\rho_{\text{exptl}}$.

Table V. Results for Density Data Fitted to Equation 4

temp, °C	j	$k \times 10^2$	$-p \times 10^2$	$q \times 10^3$	% dev $\times 10^3$ ^a	
					mean	max
25	1.0954	8.503	1.491	4.72	7	20
35	1.0854	8.578	1.511	4.74	7	15
45	1.0753	8.728	1.745	6.24	7	14
55	1.0653	8.802	1.716	5.82	7	17
65	1.0552	8.959	1.921	7.04	9	20
75	1.0450	9.078	2.000	7.40	12	32

^a % deviation = $100|\rho_{\text{exptl}} - \rho_{\text{calcd}}|/\rho_{\text{exptl}}$.

Although the refractive index is a nonlinear function, the molar refraction is essentially a linear function of mole fraction composition for the Me₂SO–3Me₂Ox system at 25 °C. Values of the molar refraction calculated by eq 5 show a maximum

$$(MR)_{12} = [(n_D^2 - 1)/(n_D^2 + 2)][(X_1M_1 + X_2M_2)/\rho_{12}] \quad (5)$$

difference of only $\pm 0.04 \text{ cm}^3 \text{ mol}^{-1}$, or $\pm 0.2\%$, from values for ideal mixtures calculated by eq 6. Least-squares treatment of

$$(MR)_{12} = X_1(MR)_1 + X_2(MR)_2 = (MR)_1 + [(MR)_2 - (MR)_1]X_2 \quad (6)$$

experimental molar refraction values (calculated by eq 5) as a linear function of mole fraction composition yields eq 7 for

$$(MR)_{12} = 20.128 + 3.223X_2 \quad (7)$$

Me₂SO–3Me₂Ox mixtures at 25 °C. The average and maximum deviations between values calculated by eq 5 and 7 are 0.04 and 0.11%, respectively.

The viscosity and activation energy of viscous flow for each Me₂SO–3Me₂Ox mixture can be described excellently as functions of temperature by the equations, respectively

$$\eta = \exp(\alpha + \beta/T + \gamma/T^2 + \delta/T^3) \quad (8)$$

$$E_{\text{vis}} = R \ln \eta / d(1/T) = R(\beta + 2\gamma/T + 3\delta/T^2) \quad (9)$$

The viscosity data were fitted to eq 8 with a nonlinear least-squares program of the type described by Wentworth (14). Resulting values of α , β , γ , and δ are summarized in Table VI along with data showing that the average and maximum deviations between experimental and calculated values never exceed 0.19 and 0.43%, respectively. Values of E_{vis} range from 3540 cal mol⁻¹ for pure Me₂SO to 3145 cal mol⁻¹ for pure 3Me₂Ox at 25 °C.

At each temperature the viscosity data as a function of composition were fitted within 0.30% to a polynomial equation of the type

$$\eta = S + VX_2 + WX_2^2 + ZX_2^3 \quad (10)$$

Table VI. Results for Viscosity Data Relative to Equations 8 and 9

$10^2 X_2$	α	$-\beta$	γ	$-\delta \times 10^{-8}$	% dev $\times 10^{2c}$		E_{vis} , cal mol $^{-1}$	std dev in E_{vis} , cal mol $^{-1}$
					mean	max		
0.00 ^a	-6.097 32	-3896.073	-1 044 563	-1.449 645	6	10	3540	22
4.65	3.875 60	5723.723	2 048 709	1.863 156	6	15	3440	60
8.41	4.316 59	6194.696	2 217 607	2.063 785	7	18	3410	66
16.76	3.268 80	5169.327	1 895 591	1.734 208	10	24	3365	95
24.19	1.959 67	3893.710	1 492 091	1.315 246	9	21	3332	79
34.15	1.068 35	3024.581	1 224 407	1.049 582	10	23	3272	88
42.75	1.565 52	3444.017	1 349 234	1.176 247	9	22	3253	88
54.01	1.136 71	3044.616	1 239 655	1.085 359	8	16	3195	67
63.95	1.632 54	3678.147	1 505 561	1.440 958	19	43	3096	165
75.24	1.359 44	3224.915	1 309 778	1.180 200	8	19	3136	73
87.85	1.121 57	2906.544	1 191 796	1.039 384	9	20	3140	78
95.14	0.324 90	2139.862	953 729	0.793 746	8	19	3138	73
100.00 ^b	-0.947 14	-109.463	86 160		1	3	3145	4

^a Reference 3. ^b Reference 6. ^c % deviation = $100|\eta_{\text{exptl}} - \eta_{\text{calcd}}|/\eta_{\text{exptl}}$.

Table VII. Results for Viscosity Data Fitted to Equation 10

temp, °C	S	$V \times 10$	$W \times 10^2$	$Z \times 10$	% dev $\times 10^{2a}$	
					mean	max
25	1.991	2.61	5.89	1.40	4	9
35	1.649	2.77	4.12	1.00	5	14
45	1.391	2.63	5.98	0.55	7	30
55	1.193	2.46	6.13	0.35	7	20
65	1.037	2.30	5.66	0.25	7	20
75	0.914	2.14	5.13	0.17	7	17

^a % deviation = $100|\eta_{\text{exptl}} - \eta_{\text{calcd}}|/\eta_{\text{exptl}}$.

The least-squares values for the constants in eq 10 and for the deviations between experimental and calculated viscosities are provided in Table VII.

Concluding Remarks

The dielectric constants, viscosities, densities, and other physical properties of 11 mixtures of two important solvents, Me₂SO and 3Me₂Ox, have been determined at several temperatures. At each temperature, the value of each physical property determined for a mixture was found to be intermediate between the values of the same property for the pure components. This suggests that molecular interactions in Me₂SO-3Me₂Ox mixtures do not differ appreciably from molecular interactions in the pure liquids.

Glossary

A, B, C	constants in eq 1
a, b, c	constants in eq 3
cP	centipoise
E_{vis}	activation energy of viscous flow, cal mol $^{-1}$
$J, K, P,$ Q	constants in eq 2
$j, k, p,$ q	constants in eq 4
M	formula weight
MR	molar refraction, cm ³ mol $^{-1}$
n_D	refractive index (sodium D line)

R	molar gas constant, cal mol $^{-1}$ deg $^{-1}$
$S, V,$ W, Z	constants in eq 10
T	temperature, K
t	temperature, °C
X	mole fraction

Acronyms

Me ₂ SO	dimethyl sulfoxide
3Me ₂ Ox	3-methyl-2-oxazolidone

Greek Letters

$\alpha, \beta, \gamma,$ δ	constants in eq 8 and 9
ϵ	low-frequency dielectric constant
η	viscosity, cP
ρ	density, g cm $^{-3}$

Numerical Subscripts

1	Me ₂ SO
2	3Me ₂ Ox
12	Me ₂ SO-3Me ₂ Ox mixture

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