Table V. Comparison of Experimental Enthalpy Deviations of *n*-Heptane of This Work, Lenoir and Hipkin (*7*), and Wilson et al. (*14, 15*) with Those Calculated by Methods of Starling and Kwok (*9, 10*) and BWR (*3, 4*)

	Enthalpy, Btu/lbm (Datum: $H = 0$, saturated liquid *n*-heptane)			Enthalpy dev ($H° - H$), Btu/lbm									
	0 psi			50 psia					100 psia				
T, °F	API and this work	Lenoir	Wilson	This work[a]	Lenoir[b]	Wilson[c]	Starling	BWR	This work[a]	Lenoir[b]	Wilson[c]	Starling	BWR
400	315.7	316.8	318.3	3.7	4.9	6.2	4.2	4.4	8.7	10.4	10.9	9.0	9.5
500	376.0	376.0	379.9	3	4.1	5.0	3.1	3.2	7.0	8.3	8.4	6.6	6.8
550	408.0	408.3	412.5	3	3.7	4.0	2.8	2.9	6.5	7.3	7.0	5.9	5.9

[a] Enthalpy deviation from API ideal gas enthalpy. [b] Enthalpy deviation from Lenoir's ideal gas enthalpy. [c] Enthalpy deviation from Wilson's ideal gas enthalpy.

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Supplementary Material Available. The complete Table I and two large-scale graphs containing enthalpy–temperature and pressure–enthalpy data for *n*-heptane will appear following these pages in the microfilm edition of this volume of the journal. Photocopies of the supplementary material from this paper only or microfiche (105 × 148 mm, 24× reduction, negatives) containing all of the supplementary material for the papers in this issue may be obtained from the Journals Department, American Chemical Society, 1155 16th St., N.W., Washington, D.C. 20036. Remit check or money order for $3.00 for photocopy or $2.00 for microfiche, referring to code number JCED-74-193.

Dielectric Constants, Viscosities, and Related Physical Properties of 10 Liquid Sulfoxides and Sulfones at Several Temperatures

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Dielectric constants, viscosities, densities, and refractive indices of 10 liquid dialkyl and cyclic sulfoxides and sulfones were measured at several temperatures ranging from 25° to 125°C. The experimental data as functions of temperature were fitted precisely to appropriate equations. Values of activation energies of viscous flow and Kirkwood correlation factors were also calculated. At comparable temperatures, sulfones consistently had higher dielectric constants, densities, viscosities, and activation energies of viscous flow but lower refractive indices than those of corresponding sulfoxides. Kirkwood correlation factors for the sulfones and sulfoxides ranged from 0.69 to 1.48 without any obvious general trend, and the magnitude of these factors implied only slight amounts of net parallel or antiparallel configurations of dipoles in the liquids.

Sulfoxides and sulfones are recognized as important aprotic compounds having broad liquid ranges, large dipole moments, and moderately high dielectric constants and viscosities. Dimethyl sulfoxide (DMSO) is by far the best known and most widely used sulfoxide (*11, 18*); tetramethylenesulfoxide (TMSO) quite recently has received considerable attention as a ligand in inorganic complexes (*2, 3*). Tetramethylenesulfone or sulfolane ($TMSO_2$) and, to a lesser extent, its 3-methyl and 2,4-dimethyl derivatives ($3MeTMSO_2$ and $2{,}4MeTMSO_2$) have been used as electrolytic solvents (*6, 20*) and media for several other types of investigations (*4, 21, 34*).

The general objective of this research was to investigate the physical properties of sulfoxides and sulfones as functions of structure and temperature. This study was designed to yield the first values for the dielectric constants, viscosities, densities, activation energies of viscous flow, and Kirkwood correlation factors at several temperatures in the range of 25–125° for five compounds, namely, di-*n*-propyl sulfoxide (DnPSO), di-*n*-butyl sulfoxide (DnBSO), dimethyl sulfone ($DMSO_2$), di-*n*-propyl sulfone ($DnPSO_2$), and di-*n*-butyl sulfone ($DnBSO_2$). Of equal importance, this study was further designed not only to check limited literature data but also to significantly extend knowledge concerning several physical properties of the other five compounds named in the preceding paragraph which heretofore have been studied over considerably narrower ranges of temperature.

Experimental

The experimental compounds were obtained from Phillips Petroleum Co., Crown Zellerbach Corp., and Aldrich

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Chemical Co. Each sulfoxide was purified through three cycles of first drying the sulfoxide at least 24 hr over Linde 3A molecular sieves and then fractionally distilling the decantate at <1 mm. Each sulfone was purified by two fractional distillations over solid KOH at <1 mm followed by a third distillation in the absence of KOH. In most cases, each compound was purified in sufficient quantity to permit a separate portion to be used for each type of measurement. In the event insufficient sample was available for all of the determinations at once, the sample was distilled again before proceeding with measurements.

A Twin-T impedance bridge assembly was used in conjunction with two-terminal parallel plate cells for capacitance measurements at 1 MHz. Similar glass cells with platinum electrodes have been found by other investigators (*1, 13*) to be structurally satisfactory and convenient to use for measurements on low conducting liquids at several temperatures. The ends of the external leads to the cell electrodes were fitted with special adapters so that the cell, which was suspended in an oil thermostat during measurements, could be easily attached to the Twin-T bridge by a shielded coaxial cable having a dual banana plug connection at each end. The principal aspects for determining the cell constants and for calculating dielectric constants have been described previously (*16, 30*). The standard media used in the determination of cell constants were air and water which have dielectric constants of 1.0005 and 78.304 (*17*), respectively, at 25°. The cell constants were assumed to be temperature independent over the experimental range. In check determinations on known systems, experimental values for the dielectric constants of water at 35°, 45°, 55°, 65°, and 75°C, and of acetone, methanol, and nitrobenzene at 25°C in each case agreed within 0.1 dielectric constant unit with widely accepted comparison data in the literature (*17, 19*).

Other values of 78.37 and 78.36 for the dielectric constant of water at 25°C have been reported by Vidulich and Kay (*31*) and Owen et al. (*24*), respectively. These values are slightly higher (<0.1%) than that of 78.30 by Malmberg and Maryott (*17*) which was used for cell calibration in this work. If one prefers results based on the data of Vidulich and Kay or of Owen et al. rather than those of Malmberg and Maryott, the experimental dielectric constant data in this work should be multiplied by a factor of 1.0008 or 1.0007, respectively.

A size 25 or 50 Cannon-Ubbelohde viscometer was selected for each liquid to obtain an efflux time exceeding 200 sec at each temperature and thereby maintain negligible kinetic energy errors. Calibration data for the viscometers were provided by the Cannon Instrument Co. 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 with electric timers. The viscosities are reported relative to a viscosity of 1.0019 cP for water at 20° (*28*).

Densities were determined pycnometrically and dilatometrically with 15-ml pycnometer-dilatometers which were calibrated with freshly distilled water at 25°C and were based on 0.99704 g/cm^3 as the density of water at this temperature (*15*). Volumes of the pycnometer-dilatometers at higher temperatures were calculated with a standard volume expansion formula and by use of 9.9×10^{-6} 1/°C as the cubical coefficient of expansion of Pyrex glass (*10*). Appropriate buoyancy corrections were applied to weights of liquids prior to calculation of densities (*15*).

Refractive indices were measured with a Spencer Abbe Model 2214 refractometer equipped with sodium D-line compensating prisms and with jackets through which water from a thermostat was circulated rapidly with a Sargent S-71598 pump.

The temperature for the dielectric constant, viscosity, and density determinations was controlled within 0.03°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 an NBS-calibrated thermometer; appropriate corrections were applied.

All determinations were made in duplicate or triplicate. The precision of multiple experimental values was within 0.2% for dielectric constants and viscosities and 0.02% for densities and refractive indices. Mean values of experimental data, which are provided in Table I, were fitted to various equations by least-squares analyses performed with an IBM 360/65 computer by use of double-precision Fortran IV programming.

Results and Discussion

All experimental data are summarized in Table I along with some comparison data for five of the compounds from other studies. In comparison with matching literature data, the results of this study exhibit excellent agreement in the cases of DMSO and TMSO$_2$, show moderate agreement for 3MeTMSO$_2$ and 2,4MeTMSO$_2$, and differ significantly for TMSO which is difficult to obtain in a state of high purity.

The dielectric constant data for each of the sulfoxides and sulfones as a function of temperature were fitted precisely to the following empirical equation:

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

The values for *A*, *B*, and *C* obtained through a standard least-squares analysis of the data for the 10 compounds are summarized in Table II. The excellent fitting of the data to Equation 1 is manifest from other data in Table II which show that the average deviation between experimental and calculated values is 0.06% or less and that the maximum deviation never exceeds 0.1%. The dielectric constant of a sulfone in each case is greater than that of the corresponding sulfoxide. In most instances the difference is about three dielectric units; however, at 125° the value of ϵ for DMSO$_2$ exceeds that for DMSO by 11 units. The values of ϵ for 3MeTMSO$_2$ and 2,4MeTMSO$_2$ surprisingly differ very little but are substantially lower than comparable values for TMSO$_2$.

To ascertain the effect on the dielectric constant caused by a double bond in the ring structure of a cyclic sulfone, a concerted effort was made to measure the dielectric constants of three sulfolenes or unsaturated sulfones, namely, 3-sulfolene and its 3-methyl and 2,4-dimethyl derivatives which have melting points above 60°, 60°, and 40°, respectively. Unfortunately, however, each of these compounds undergoes decomposition upon distillation or fractional freezing and also fairly rapid change upon simple fusion, yielding a liquid so conducting that capacitance measurements at 1 MHz were not feasible.

The density data for each compound as a function of temperature are described excellently by the following equation:

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

The calculated least-squares values for *a*, *b*, and *c* are compiled in Table III which also contains data showing that the maximum difference between experimental and calculated densities in no case exceeds 0.026%. From data in Table I, the density of a sulfone consistently is

Table I. Experimental Data

Temp, °C	Dielectric constant, 1 MHz	Viscosity, cP	Density, g/cm^3	Refractive index
DMSO				
25	46.45	1.991	1.0955	1.4771
	46.4^{a}	1.998^{a}	1.0961^{a}	
	46.4^{b}	1.996^{b}	1.0955^{b}	
30	45.82	1.808	1.0904	1.4752
40	44.55	1.511	1.0803	1.4706
	44.6^{c}	1.513^{c}	1.0804^{c}	
50	43.25	1.286	1.0702	1.4654
75	40.02	0.916	1.0454	
100	36.86	0.691	1.0200	
125	33.88	0.546	0.9946	
DnPSO				
30	30.37	2.526	0.9531	1.4641
40	29.19	2.002	0.9449	1.4597
50	27.97	1.630	0.9367	1.4550
75	25.14	1.076	0.9157	
100	22.57	0.772	0.8949	
125	—	—	0.8739	
DnBSO				
40	24.73	3.149	0.9216	1.4606
50	23.64	2.454	0.9138	1.4559
60	22.53	1.972	0.9059	
75	21.04	1.498	0.8947	
100	18.73	1.028	0.8750	
125	16.70	0.749	0.8552	
TMSO				
25	42.86	5.252	1.1682	1.5192
	42.5^{d}			
30	42.04	4.697	1.1637	1.5168
		5.2^{d}	1.173^{d}	
40	40.48	3.809	1.1548	1.5130
			1.169^{d}	
50	38.92	3.154	1.1460	1.5087
75	35.38	2.109	1.1243	
100	32.24	1.505	1.1023	
125	29.46	1.132	1.0802	
DMSO$_2$				
110	47.39	1.368	1.1625	
112	—	—	1.1605	
115	46.56	1.285	1.1574	
120	45.78	1.208	1.1525	
125	45.00	1.140	1.1477	
DnPSO$_2$				
30	32.62	5.432	1.0413	1.4494
40	31.70	4.043	1.0330	1.4453
50	30.73	3.109	1.0246	1.4409
75	28.43	1.855	1.0039	
100	26.21	1.236	0.9829	
125	24.16	0.895	0.9616	
DnBSO$_2$				
50	25.72	4.711	0.9860	1.4433
60	24.87	3.595	0.9781	
75	23.65	2.556	0.9663	
100	21.68	1.601	0.9460	
125	19.91	1.111	0.9259	
TMSO$_2$				
30	43.26	10.284	1.2619	1.4810
	43.3^{e}	10.286^{e}	1.2614^{e}	
	43.33^{f}	10.29^{f}	1.2623^{f}	
40	41.99	7.953	1.2534	1.4777
	41.8^{e}	7.974^{e}	1.2525^{e}	
50	40.76	6.312	1.2447	1.4742
	40.7^{e}	6.312^{e}	1.2447^{e}	
75	37.84	3.846	1.2229	
100	35.16	2.570	1.2009	
125	32.74	1.835	1.1789	
3MeTMSO$_2$				
25	29.40	11.810	1.1884	1.4756
	29.4^{e}	11.67^{e}	1.1884^{e}	
30	29.08	10.090	1.1841	1.4738
	29.2^{e}	10.14^{e}	1.1833^{e}	
40	28.46	7.596	1.1755	1.4705
	28.6^{e}	7.65^{e}	1.1751^{e}	
50	27.80	5.882	1.1669	1.4670
	27.8^{e}	5.88^{e}	1.1672^{e}	
75	26.26	3.488	1.1457	
100	24.74	2.289	1.1241	
125	23.34	1.625	1.1028	
2,4MeTMSO$_2$				
25	29.69	9.079	1.1328	1.4707
	29.5^{g}	9.04^{h}	1.1314^{h}	
30	29.34	7.755	1.1286	1.4692
40	28.62	5.819	1.1202	1.4660
50	27.89	4.519	1.1118	1.4617
			1.1106^{g}	
75	26.16	2.693	1.0909	
			1.0914^{g}	
100	24.48	1.793	1.0699	
125	22.89	1.285	1.0488	

^{a} (33). ^{b} (25). ^{c} (26). ^{d} (12). ^{e} (29). ^{f} (7). ^{g} (9). ^{h} (5).

several percent greater than that of the corresponding sulfoxide.

The experimental dielectric constant, density, and refractive index data may be used with dipole moment data from the literature to gain insight concerning the nature and extent of association of polar liquids such as the sulfones and sulfoxides (*23, 27*). The Kirkwood-Frohlich equation (*8, 14*) in the form

$$g = [(\epsilon - \epsilon_\infty)(2\epsilon + \epsilon_\infty)9MkT]/[\epsilon(\epsilon_\infty + 2)^2 4\pi N\rho\mu_0{}^2] \quad (3)$$

makes possible a description of polar liquids having specific short-range forces which hinder rotations of molecules. The correlation factor, *g*, is a measure of the short-range effects which hinder orientation of a molecule with respect to its surrounding neighbors. For systems in which intermolecular forces are nonspecific or perfectly random, *g* equals unity and the Kirkwood-Frohlich equation reduces to the Onsager equation (*22*) for a "normal" polar liquid. For liquids in which specific intermolecular forces orient neighboring dipoles in a net parallel configuration, *g* is greater than unity; for a net antiparallel configuration of dipoles, *g* is less than unity.

The calculated Kirkwood correlation factors for the sulfones and sulfoxides are presented in Table IV. The deviations of the correlation factors for the liquids from unity indicate possibly slight extents of net parallel or antiparallel association of dipoles, but none of the values of *g* is sufficiently above or below unity to substantiate any cases of predominantly parallel or antiparallel association of dipoles. It is interesting, nevertheless, that the *g*

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

Compound	$-A$	$B \times 10^{-4}$	$-C \times 10^{-6}$	% Deviation, 100[a] Mean	Max
DMSO	40.574	4.06426	4.38087	2	5
DnPSO	28.676	2.43801	1.96285	5	10
DnBSO	18.496	1.57478	0.69121	6	10
TMSO	20.240	2.26540	1.14335	5	9
$DMSO_2$	15.926	2.42584	0	1	2
$DnPSO_2$	23.780	2.54087	2.51810	4	7
$DnBSO_2$	17.769	1.90691	1.62035	4	8
$TMSO_2$	15.928	2.39354	1.81589	2	4
$3MeTMSO_2$	10.177	1.79390	1.83013	3	6
$2,4MeTMSO_2$	14.345	1.98829	2.01330	2	5

[a] % Deviation $= 100|\epsilon_{\text{exptl}} - \epsilon_{\text{calcd}}|/\epsilon_{\text{exptl}}$.

Table III. Results for Density Data Fitted to Equation 2

Compound	a	$-b \times 10^4$	$-c \times 10^7$	% Deviation $\times 10^3$ [a] Mean	Max
DMSO	1.12032	9.9431	0.887	7	15
DnPSO	0.97774	8.1836	0.971	4	10
DnBSO	0.95162	7.4615	2.020	12	26
TMSO	1.19003	8.7685	0.096	7	13
$DMSO_2$	1.27146	9.9082	0	4	5
$DnPSO_2$	1.06599	8.1894	1.255	3	9
$DnBSO_2$	1.02525	7.7637	1.509	7	15
$TMSO_2$	1.28783	8.5756	1.105	3	5
$3MeTMSO_2$	1.20980	8.5635	0.012	5	13
$2,4MeTMSO_2$	1.15370	8.3515	0.333	2	5

[a] % Deviation $= 100|\rho_{\text{exptl}} - \rho_{\text{calcd}}|/\rho_{\text{exptl}}$.

Table IV. Kirkwood Correlation Factors

Compound	μ_0, Debyes	Temp, °C	g
DMSO	3.90[a]	25	1.07
		30	1.08
		40	1.10
		50	1.12
DnPSO	3.90[b]	30	1.41
		40	1.42
		50	1.42
DnBSO	3.90[b]	40	1.48
		50	1.48
TMSO	4.17[a]	25	1.01
		30	1.02
		40	1.02
		50	1.03
$DMSO_2$	4.25[a]	125	1.46[c]
$DnPSO_2$	4.47[a]	30	1.22
		40	1.24
		50	1.26
$DnBSO_2$	4.46[a]	50	1.28
$TMSO_2$	4.81[a]	30	0.88
		40	0.90
		50	0.91
$3MeTMSO_2$	4.81[b]	25	0.69
		30	0.70
		40	0.72
		50	0.73
$2,4MeTMSO_2$	4.81[b]	25	0.82
		30	0.83
		40	0.84
		50	0.86

[a] (*12*). [b] Estimated to be equal to that for a closely related compound. [c] Based on an estimated value of 1.42 for refractive index.

values of the dialkyl sulfones are slightly greater than unity, whereas those for the cyclic sulfones are less than unity. The g values for the dialkyl sulfoxides likewise are relatively larger than the value for cyclic TMSO.

The viscosity and the activation energy of viscous flow for each liquid may be described as functions of temperature by the following equations, respectively:

$$\eta = e^{(\alpha + \beta/T + \gamma/T^2 + \delta/T^3)} \quad (4)$$

$$E_{\text{vis}} = R d(\ln \eta)/d(1/T) = R(\beta + 2\gamma/T + 3\delta/T^2) \quad (5)$$

The viscosity data were fitted to Equation 4 by a nonlinear least-squares program of the type described by Wentworth (*32*). Resulting values for α, β, γ, and δ are compiled in Table V along with data showing that the average and maximum deviations between experimental and calculated values of viscosity never exceed 0.16 and 0.32%, respectively. Generally, the viscosity of a sulfone is about twice that of the corresponding sulfoxide. This may be attributed to greater hindrance toward flow caused by the tetrahedral structure of a sulfone in contrast to the pyramidal structure of a sulfoxide and, probably to a lesser extent, to stronger dipole–dipole attractions in the sulfone which has a larger dipole moment as indicated in Table IV. An inverse correlation exists between the viscosities and molecular weights of $TMSO_2$ and its 3-methyl and 2,4-dimethyl derivatives at each experimental temperature. While relatively uncommon, this inverse viscosity–molecular weight relationship also applies for pyridine-*N*-oxide and its 2-methyl and 2,6-dimethyl derivatives as found recently in another study in this laboratory.

A value of E_{vis} for each liquid at one temperature also is presented in Table V. As expected on the basis of structural features and other physical properties, values of E_{vis} for sulfones are considerably larger than those of corresponding sulfoxides.

Nomenclature

n_D = refractive index (sodium *D*-line)
T = temperature, K
t = temperature, °C
g = Kirkwood correlation factor
M = formula weight in g/mol
k = Boltzmann constant
N = Avogadro number
E_{vis} = activation energy of viscous flow in cal/mol
R = molar gas constant in cal/(mol deg)
cP = centipoise
A, B, C = constants in Equation 1
a, b, c = constants in Equation 2

Greek Letters

ϵ = low-frequency dielectric constant
ϵ_∞ = high-frequency dielectric constant, approximated as 1.10 n^2D
η = viscosity in cP
ρ = density in g/cm^3
μ_0 = dipole moment in Debye units
$\alpha, \beta, \gamma, \delta$ = constants in Equations 4 and 5

Table V. Results for Viscosity Data Relative to Equations 4 and 5

Compound	$-\alpha$	β	γ	$\delta \times 10^{-8}$	% Deviation, 100[a] Mean	Max	E_{vis} (°C), cal/mol	SD in E_{vis}, cal/mol
DMSO	6.09732	3896.073	−1044563	1.449645	6	10	3171 (50)	5
DnPSO	13.09943	11197.79	−3624007	4.603172	5	11	3960 (50)	12
DnBSO	17.77489	16645.78	−5692011	7.311975	5	11	4816 (50)	14
TMSO	6.26920	4329.353	−1080863	1.475340	7	14	3732 (50)	7
$DMSO_2$	2.18771	23.878	358042	0	3	5	3761 (110)	68
$DnPSO_2$	8.93263	7386.321	−2435050	3.553887	16	31	5018 (50)	22
$DnBSO_2$	5.88679	4327.727	−1424757	2.594084	13	24	5886 (50)	120
$TMSO_2$	4.73939	2963.174	−599229	1.063099	2	6	4587 (50)	3
$3MeTMSO_2$	6.96166	5429.437	−1579581	2.382514	11	22	4963 (50)	11
$2,4MeTMSO_2$	8.09114	6594.420	−2055982	2.997189	3	6	4928 (50)	3

[a] % Deviation $= 100|\eta_{\text{exptl}} - \eta_{\text{calcd}}|/\eta_{\text{exptl}}$.

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