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Dielectric Constants, Viscosities, Densities, and Refractive Indices of Some Alkylsydnones at Several Temperatures

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Dielectric constants, viscosities, densities, and refractive indices of four liquid alkylsydnones were measured at two to six temperatures within the range of 25–125 °C. All experimental data (except refractive indices) for 3-methylsydnone and 3-isopropylsydnone have been fitted to appropriate equations. Kirkwood–Fröhlich correlation factors for these two sydnones also have been calculated. The dielectric constant of each alkylsydnone is high, and particularly high in the case of 3-methylsydnone (ϵ = 144.0 at 40 °C) even though the calculated Kirkwood–Fröhlich correlation factor of 1.03 for this compound implies that there is little net parallel alignment of dipoles in the liquid. Factors which might affect the use of 3-methylsydnone as a nonaqueous solvent also are discussed.

The chemistry of the sydnones (see Figure 1) has been studied extensively since the original preparation of 3-phenylsydnone by Earl and Mackney (*7*). The structure, reactions, and some physicochemical properties of these interesting compounds have been reviewed by Baker and Ollis (*1*), by Stewart (*24*), and, more recently, by Ohta and Kato (*20*). One very notable feature of the sydnones is their unusually high dipole moments; values for many sydnones are greater than 6 D (*6*, *12*) and the value for 3-methylsydnone has been reported (*3*, *22*) as 7.3 D. While most sydnones are relatively high-melting solids, many of the 3-alkylsydnones are liquids at or slightly above room temperature. Nevertheless, it appears that no previous study considering bulk physical properties such as dielectric constants, viscosities, densities, and refractive indices of 3-alkylsydnones has been reported.

Considering the high dipole moments, the apparently convenient liquid range and the reasonably well described syntheses of these compounds, it appeared that one or more of these compounds might be a potentially useful nonaqueous solvent provided purification were feasible and the bulk physical properties were known.

Experimental Section

Preparation and Purification of the Sydnones. 3-Methylsydnone was prepared from *N*-methylglycine, commonly known as sarcosine, by the method of Vasil'eva and Yashunskii (*25*). A liquid–liquid extractor was used (*11*) for 12–24 h to recover the *N*-nitroso-*N*-methylglycine intermediate prior to its treatment with acetic anhydride. Yields of 70–80% of crude 3-methylsydnone were routinely achieved from 200-g portions of the *N*-methylglycine. *Warning: immediate introduction of air after vacuum distillation of the product from acetic anhydride* (*while the residual material was still hot*) *was found to lead to a mild explosion* (*see also ref 11*). The 3-methylsydnone was readily purified by fractional freezing (*2*). The compound appears to exist in two distinct colorless crystalline forms: needles which have a melting point near 36 °C (in agreement with the literature (*11*) and plates which melt several degrees lower.

The preparation of 3-isopropylsydnone starting with ethyl bromoacetate and isopropylamine has been reported (*10*, *15*); however, the method used to prepare 3-isopropylsydnone in quantity for the present work was similar to that used for the preparation of 3-*tert*-butylsydnone reported by Procházka and co-workers (*21*). Potassium cyanide (390 g) and isopropylamine (354 g) were dissolved together in water (360 mL). After the solution was cooled to 5 °C, concentrated aqueous HCl (575 mL) was added slowly. Formaldehyde (525 g of 37% aqueous solution) was added dropwise over 2 h at 10–15 °C. To the resulting brown solution, water (300 mL) was added followed by sodium hydroxide (270 g in 380 mL of water). The dark red solution was refluxed for 2 h. Water (1000 mL) was added and then removed by distillation together with any unreacted amine and aldehyde. The solution was acidified, the alkylamino acid was nitrosated (in portions), and the solution was allowed to stand overnight. The *N*-nitroso-*N*-isopropylglycine was then removed by filtration and dried (vacuum, 40 °C). The yield of *N*-nitroso-*N*-isopropylglycine was 65–70%. This material then was treated with an excess of acetic anhydride and the mixture was allowed to stand for several days at room temperature. The acetic anhydride and acetic acid were removed by vacuum distillation, and the crude isopropylsydnone was recovered by distillation (130 °C) under vacuum (yield 330 g). This compound also was purified by fractional freezing and was found to be colorless and to melt at approximately 57 °C (lit. (*15*), 54–55.5 °C).

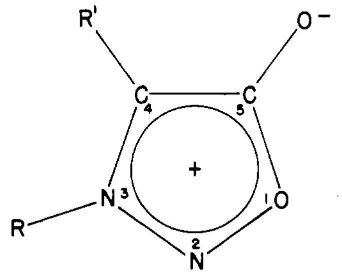

Figure 1. Structure and numbering of ring atoms for sydnones.

3-*n*-Propyl-4-methylsydnone was prepared by the method of Dhawan and Kier (*5*). Again, as with the methyl compound, release of air into the hot distillation apparatus may result in a mild explosion. The viscous red-brown liquid initially obtained as a product could not be made to crystallize, even at very low temperatures. Distillation under vacuum resulted in a limited amount of purification but also tended to decompose the compound. The distillate had a dark yellow color which tended to become darker on standing in contact with the laboratory atmosphere over a period of several days. Nevertheless, a proton NMR spectrum indicated that the compound was fairly pure. All physical measurements on this compound should, however, be considered only approximate and, indeed, the density and dielectric constant values both appeared to decrease slightly with time.

3-*n*-Butylsydnone was prepared by the method which Kier and Dhawan used for the preparation of other 3-alkylsydnones (*15*). The crude sydnone was a dark brown liquid. As with the 3-*n*-propyl-4-methylsydnone, purification was limited to a single further vacuum distillation. There was, however, no apparent decrease in the purity of the 3-*n*-butylsydnone with time. The proton NMR spectrum again indicated that the sydnone was reasonably pure but all physical property measurements on this compound from the present work should be regarded as approximate.

Experimental Measurements. A General Radio Type 821-A Twin-T impedance measuring assembly (*17, 23*) was used in conjunction with two-terminal parallel-plate cells for capacitance measurements at 1 MHz. The assembly and cells were similar to those used in previous studies (*4, 14*). The standard media used in the determination of the cell constants were air and water which have dielectric constants of 1.0005 (*26*) and 76.546 (*19*), respectively, at 30 °C. The measurements were made in duplicate, and the precision obtained was within 0.2%.

Dielectric constant measurements at 10 MHz were made using the same bridge assembly in conjunction with jacketed metal plug-in cells (*17*). These cells were calibrated at 25 and 40 °C again using air and water. The dielectric constant cells were maintained at a given temperature by rapidly circulating water from a thermostated bath (controlled within 0.05 °C) through the cell jackets. The precision of the duplicate measurements was within 0.3% except for the measurements on 3-*n*-propyl-4-methylsydnone for which apparent decomposition of the sydnone caused differences of about 1%. The higher value is reported for this compound at 40 °C. The value of the dielectric constant of 3-methylsydnone at 40 °C and 10 MHz is based on a single measurement. Relatively high conductances of the somewhat impure 3-*n*-butylsydnone and 3-*n*-propyl-4-methylsydnone made capacitance measurements for these compounds at 1 MHz impossible with the available bridge assembly and cells. This necessitated the measurements at 10 MHz.

The viscosities were measured with sizes 50, 75, and 100 Cannon-Ubbelohde viscometers which were previously calibrated by the Cannon Instrument Co. Efflux times were always greater than 200 s making corrections for kinetic energy effects unnecessary. To minimize atmospheric contamination, a dry nitrogen atmosphere was maintained over the liquids during these determinations. Efflux times were measured to 0.1 s using electric timers. The measurements were made in duplicate or triplicate and, except those for 3-*n*-propyl-4-methylsydnone, a precision of 0.3% was achieved.

Table I. Experimental Data

Temp, °C	Dielectric constant	Viscosity, cP	Density, g cm^{-3}	Refractive index
		3-Methylsydnone		
40	144.0[a] (1 MHz)	5.501	1.3085	1.515_0
50	135.8	4.367	1.2991	1.511_5
60	128.3	3.546	1.2896	1.508_0
75	118.3	2.731	1.2758	
100	104.1	1.899	1.2526	
125	92.5	1.413	1.2294	
		3-Isopropylsydnone		
60	66.0 (1 MHz)	3.864	1.1324	1.485_2
75	62.3	2.879	1.1198	
100	56.5	1.913	1.0983	
125	51.4	1.379	1.0771	
		3-*n*-Butylsydnone		
25	52.8 (10 MHz)	21.4	1.097	1.487
40	49.5	11.5	1.085	1.481
		3-*n*-Propyl-4-methylsydnone		
25	66.4 (10 MHz)	14.7	1.124	1.497
40	62.9	9.3	1.112	1.492

[a] 143.7 (10 MHz) at 40 °C.

Densities were determined pycnometrically and dilatometrically using 15-mL pycnometer–dilatometers which were calibrated with distilled water at 30 °C (*13*). Volumes of the pycnometer–dilatometers at other temperatures were calculated with a standard volume expansion formula using 9.6×10^{-6} °C^{-1} as the cubical coefficient of expansion of Pyrex glass (*16*). Buoyancy corrections were applied to all experimental weights (*16*). Density measurements were done in duplicate or triplicate and a precision of 0.02% was attained except again for the determinations of the density of 3-*n*-propyl-4-methylsydnone which were reproducible within 0.2%.

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 constant temperature bath was circulated.

The temperature for the dielectric constant (1 MHz), 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 (0.2 °C subdivisions at 125 °C) were compared with thermometers previously calibrated against NBS-calibrated thermometers (H-B Instrument Co.) or compared directly against an NBS-calibrated thermometer.

Mean values of the experimental data for 3-methylsydnone and 3-isopropylsydnone were fitted, as functions of temperature, to various equations by least-squares analysis using an IBM 370-165-II computer. These equations then were used to calculate small corrections to the values of the physical constants, from the temperatures at which the measurements were made to the temperatures shown in Table I ($\Delta T < 0.5$ °C). The values in Table I, therefore, are corrected but not smoothed values.

Results and Discussion

The experimental data are summarized in Table I. There appear to be no values available in the literature for comparison.

The dielectric constant data for 3-methylsydnone were fitted to the empirical equation

$$\epsilon = 7.759 + 964.18/T + 1.3054(10^7/T^2) \qquad (1)$$

and the data for 3-isopropylsydnone to the parallel equation

$$\epsilon = -34.830 + 3.7933(10^4/T) - 1.4395(10^6/T^2) \qquad (2)$$

The maximum deviation between experimental and calculated values was found to be <0.1%. The dielectric constants are quite high compared to those for most organic liquids and, above 100 °C, the dielectric constant of 3-methylsydnone is even greater than that of *N*-methylacetamide (*18*). Increasing the chain length of the alkyl group at the 3-position of the sydnone ring appears to markedly decrease the dielectric constant. The agreement between the 1-MHz and 10-MHz measurements at 40 °C for 3-methylsydnone is quite good.

The density data for 3-methylsydnone is described excellently as a function of temperature by the linear equation

$$\rho = 1.34558 - 9.2986(10^{-4}t) \qquad (3)$$

with a maximum deviation of 0.012%. Similarly, the temperature dependence of the density of 3-isopropylsydnone can be expressed as

$$\rho = 1.18364 - 8.5336(10^{-4}t) \qquad (4)$$

with a maximum deviation of 0.015%. The density of 3-methylsydnone is fairly high, similar to many organic nitro compounds. As might be expected, the density decreases markedly as the organic group on the 3-position of the sydnone ring becomes larger.

The experimental dielectric constant, density, and refractive index data were used with available dipole moment data from the literature to obtain information concerning the nature and extent of association in two of the liquid sydnones. This was done by calculation of the Kirkwood–Fröhlich *g* factor (*8*) using the equation

$$g = [(\epsilon - \epsilon_\infty)(2\epsilon + \epsilon_\infty)9MkT]/[\epsilon(\epsilon_\infty + 2)^2 4\pi N\rho\mu_0{}^2] \qquad (5)$$

The *g* factor gives an indication of the type of dipole–dipole alignment taking place in a polar liquid. Values of $g > 1$ indicate net parallel asignment of the dipoles while $g < 1$ suggests net antiparallel alignment. Values of *g* which are close to unity, as found for 3-methylsydnone and 3-isopropylsydnone (see Table II), suggest that the intermolecular forces in such liquids are nonspecific or random.

The temperature dependence of the viscosity of 3-methylsydnone can be described, with a maximum deviation of 0.22%, by the equation

$$\eta = \exp[-9.3507 + 8.4545(10^3/T) - 2.8078(10^6/T^2) + 3.8971(10^8/T^3)] \qquad (6)$$

For 3-isopropylsydnone, the results fit eq 7 with a maximum deviation of 0.03%.

$$\eta = \exp[-1.2760 - 5.9164(10^2/T) + 4.8878(10^5/T^2)] \qquad (7)$$

The sydnones, as indicated by the viscosity data in Table I, are moderately viscous liquids. Apparently the viscosity is enhanced if a longer linear alkyl group is attached to the 3-position of the sydnone ring.

Other Findings Relative to 3-Methylsydnone

A number of common alkali metal, alkaline earth metal, and quaternary ammonium salts were found to be soluble in 3-methylsydnone to the extent of more than 0.075 *m*. Water, benzene, methanol, acetone, and chloroform are miscible with 3-methylsydnone in all proportions while carbon tetrachloride is only slightly miscible and hexane is practically immiscible with this sydnone. Qualitative experiments indicate that 3-methylsydnone is only slightly hygroscopic at 40 °C. The work of Garrett (*9*) previously has shown that the hydrolysis of 3-methylsydnone is quite slow, particularly in neutral aqueous solution near room temperature. The minimum specific conductance thus far observed for purified 3-methylsydnone is $7.5 \times 10^{-8}\ \Omega^{-1}\ \mathrm{cm}^{-1}$ at 40 °C; at this temperature, the specific conductance changes approximately 2.7% $°\mathrm{C}^{-1}$.

Table II. Kirkwood–Fröhlich Correlation Factors

Compd	Dipole moment, D	Temp, °C	Correlation factor (*g*)
3-Methylsydnone	7.31[a]	40	1.03
3-Isopropylsydnone	6.70[b]	60	0.91

[a] Reference 22. [b] Approximated as being equal to the dipole moment of 3-cyclohexylsydnone (ref 12).

Conclusion

The reasonably convenient liquid range, high purity, low specific conductance, very high dielectric constant, large dipole moment, moderate viscosity, and fairly good dissolving power of 3-methylsydnone indicate that it has excellent potential and is indeed worthy of investigation as a new nonaqueous solvent. The other sydnones discussed in this work appear to have considerably less potential.

Acknowledgment

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Glossary

cP	centipoise
g	Kirkwood–Fröhlich correlation factor
k	Boltzmann constant
M	formula weight, g mol^{-1}
N	Avogadro number
n_D	refractive index (sodium D line)
T	temperature, K
t	temperature, °C

Greek Letters

ϵ	low-frequency dielectric constant
ϵ_∞	high-frequency dielectric constant, approximated as $1.10\ n_D{}^2$
η	viscosity, cP
μ_0	dipole moment
ρ	density, g cm^{-3}

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Absorption and Fluorescence Properties of Cyanine Dyes

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The absorption and fluorescence properties of a variety of cyanine dyes in dimethyl sulfoxide solutions have been determined. The wavelengths of the absorption and fluorescence maxima and the fluorescence quantum yield relative to sodium fluorescein have been measured. The results are discussed in relation to the molecular structure.

During the course of our research to evaluate indicator substances that could be used in the study of the retinal and choroidal circulation in the eye, a substantial amount of data was obtained regarding cyanine and merocyanine (or polymethine) dyes. Although the polymethine dyes are widely used as photographic sensitizers, in dye lasers, as saturable absorbers for passively Q-switching lasers, and as molecular probes of membrane potential, there does not appear to be a compilation of the spectral data of these compounds in the literature. In fact, very little has been published regarding fluorescence properties at wavelengths longer than 700 nm. We have correlated the spectral properties of the polymethine dyes with their molecular structure in order to predict the wavelengths of maximum absorption and fluorescence and to roughly estimate the fluorescence quantum yield.

Experimental Section

A Beckman DK-2 spectrometer was used to measure the absorption spectra. A schematic of the fluorescence apparatus is shown in Figure 1. Note that because our work was ultimately for ophthalmic applications, the fluorescence was detected in a direction parallel to the excitation light instead of the usual 90° configuration. A 75-W xenon lamp was used as the excitation source, and the output was filtered by a SPEX Model 1670 monochromator; the slit width was 2.5 mm (10 nm band-pass). The excitation light was focused on the sample cell (1-cm cuvette) by a 30-cm spherical mirror. The fluorescence from the sample was coupled out through a 5-cm diameter hole in the center of the mirror, modulated by a mechanical chopper, and focused on the slit (0.5 mm, 5 nm band-pass) of a Perkin-Elmer Model 83 monochromator. The fluorescence detector was an RCA 4832 photomultiplier tube (PMT) which has a rather flat response from 400 to 850 nm. (For wavelengths longer than 850 nm, an RCA C31004A PMT was used.) The PMT output was preamplified and phase-sensitive detected at the modulation frequency using a lock-in amplifier. The lock-in output was then chart recorded.

All measurements were made at ambient conditions without extensive temperature regulation or solvent degassing. The dyes were obtained from several manufacturers (see Table I) and were used without further purification.

The fluorescence quantum yield of the cyanine dyes was measured relative to sodium fluorescein in distilled water. The optimum fluorescein concentration for the 1-cm cuvette was 0.1 g/L (2.7×10^{-4} M). The quantum yields have been corrected for the spectral response of the apparatus components.

Results and Discussion

The spectral results are summarized in Table I according to the basic molecular structure. λ_{abs} refers to the wavelength at which the absorption was found to be a maximum. The excitation wavelength (λ_{ex}) refers to the actual wavelength setting of the source monochromator which produces the maximum sample fluorescence. Note that λ_{ex} was not corrected for the spectral variations of the source, and since the intensity of the xenon lamp varied with wavelength, λ_{ex} did not necessarily coincide with λ_{abs}.

Although we were primarily interested in aqueous solutions for the ophthalmic applications, the majority of the dyes were not sufficiently soluble in water. In order to compare the results from a variety of dyes, dimethyl sulfoxide (Me_2SO) was used since most of the dyes were readily soluble in it. Studies of the concentration dependence of fluorescence in Me_2SO for several dyes indicated the maximum fluorescence occurred at approximately 0.1 g/L. This concentration was used in all of the fluorescence measurements reported in Table I.

As mentioned above, the fluorescence quantum yield has been corrected for the spectral response of the apparatus. However, the majority of the dyes are rather impure (*3*) and, therefore, the quantum yields should be considered for general trends rather than for comparing individual dyes. For example, it is evident from Table I that dyes possessing the 4,4′- or 2,2′-quino structure and the merocyanines generally have low

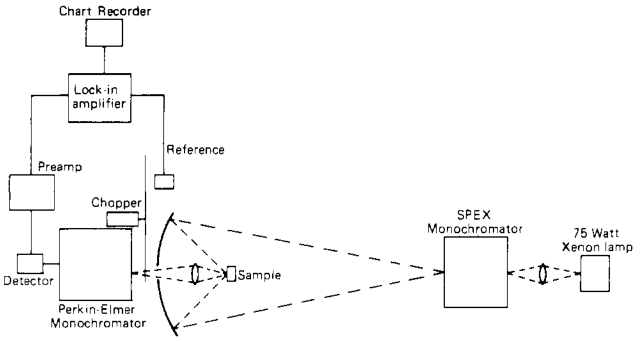

Figure 1. Schematic of apparatus used in measuring fluorescence spectra.