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Refractive Indices of Binary Solutions of Nitroparaffins in Carbon Tetrachloride

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Abstract—The refractive indices of binary solutions of nitromethane and nitroethane in carbon tetrachloride were determined at 30 and 35 °C. Various refractive index-concentration and refractive index-temperature relations were calculated. The nitromethane solutions showed deviations from ideality; the nitroethane solutions did not. The deviations are within the limits of empirical mixture laws.

1. Introduction

This investigation is a continuation of the series (cf. Gunter *et al.*, 1971 for most recent paper in the series) whereby simple physical measurements are used to determine the nature of binary solutions of nitroparaffins in carbon tetrachloride. As in the previous papers, nitromethane and nitroethane are the nitroparaffins used. In this work, the simple physical measurement is refractive index.

2. Experimental

Fisher certified grades of carbon tetrachloride and nitromethane, high purity research samples of nitromethane and nitroethane (Commercial Solvents), and highest purity nitroethane (Brothers Chemical Co.) were used without further purification. The solutions were prepared by mixing volumes of pure components. The estimated cumulative transfer error was ± 0.005 mole fraction.

Refractive index measurements were made with a Bausch and Lomb dipping refractometer (# 33-45-25) equipped with a metal beaker attachment. The *B* (1.36435-1.40601), *C* (1.39868-1.43827), and *D* (1.43626-1.47557) interchangeable prisms were utilized in making the measurements. Both sunlight and artificial lighting were used as a light source. Each solution was allowed 20 to 40 minutes

to reach thermal equilibrium before a reading was taken. Scale readings, ± 0.05 , on each solution were made at 10 minute intervals. Readings were always made from low to high scale values. Each scale reading was converted to a refractive index value with tables provided by Bausch and Lomb.

Constant temperature, $\pm 0.1^\circ\text{C}$, was maintained by use of a water circulating device (Precision Scientific Co., Cat. No. 6600, Serial No. C-1) which was connected to a water trough by use of a lead and a return rubber hose. The water trough is secondary equipment to the dipping refractometer. Temperature readings were made by use of an uncalibrated thermometer graduated in tenths of a degree.

All calculations were performed on an IBM 1620 computer with 40K storage using an IBM PR 025 monitor with programs written in FORTRAN II.

3. Results and Discussion

The refractive indices of $\text{CH}_3\text{NO}_2\text{-CCl}_4$ and $\text{C}_2\text{H}_5\text{NO}_2\text{-CCl}_4$ solutions at 30°C and 35°C are given in Table 1. Also tabulated in Table 1 are the average deviations and the probable errors (cf. Daniels *et al.*, 1962). These probable errors, due to statistical fluctuations in repeated observations, are much smaller than the theoretical limit of the refractometer (4×10^{-5}). For the 30°C measurements, each tabulated entry represents an average of three to six readings for a single stock solution; for the 35°C measurements, each entry is an average of 18 to 75 readings taken upon two to three stock solutions. The average deviations increase with the increase in the number of readings, but the probable errors are only slightly greater, indicating a slightly poorer quality in the readings, probably due to evaporation, at the higher temperature. The 0.3 mole fraction readings at 30°C were not obtained because the refractive index of these solutions occurs at the transition between the *C* and *D* prisms. A special study of a 0.4 mole fraction CH_3NO_2 solution at 30°C indicated, by maximum differences of 1×10^{-5} , no aging (one measurement each of several consecutive days) effect or evaporation (all solutions were stored in glass-stoppered bottles) effect.

The only available literature values at the temperatures of

TABLE I Refractive indices of carbon tetrachloride-nitroparaffin solutions.

Mole Fraction (RNO ₂)	<i>n_D</i>	30 °C		<i>n_D</i>	35 °C	
		Average Deviation × 10 ⁵	Probable Error × 10 ⁵		Average Deviation × 10 ⁵	Probable Error × 10 ⁵
Nitromethane						
0.0	1.45481	2	0.8	1.45207	2	0.3
0.1	1.44968	0.6	0.2	1.44701	0.8	0.2
0.2	1.44431	0.7	0.3	1.44184	0.2	0.05
0.3				1.43522	2	0.3
0.4	1.43195	0.2	0.1	1.42969	2	0.3
0.5	1.42489	0.5	0.3	1.42313	2	0.3
0.6	1.41806	0.3	0.2	1.41593	2	0.4
0.7	1.40996	0.3	0.1	1.40764	6	0.8
0.8	1.40044	0.2	0.08	1.39735	4	0.5
0.9	1.39002	0.2	0.1	1.38703	0.9	0.1
1.0	1.37811	0.3	0.2	1.37520	1	0.2
1.0	1.37730 ^a					
1.0	1.37738 ^b					
Nitroethane						
0.0	1.45481	2	0.8	1.45207	2	0.3
0.1	1.44945	2	0.6	1.44667	2	0.2
0.2	1.44416	0.6	0.2	1.44142	5	0.7
0.3				1.43505	1	0.2
0.4	1.43198	0.3	0.1	1.42950	6	0.8
0.5	1.42573	0.2	0.08	1.42328	2	0.3
0.6	1.41926	0.2	0.08	1.41694	2	0.3
0.7	1.41216	0.2	0.08	1.40989	6	0.8
0.8	1.40454	2	0.6	1.40112	12	1
0.9	1.39644	0.2	0.1	1.39334	5	0.6
1.0	1.38772	0.2	0.09	1.38465	2	0.2
1.0	1.38754 ^b					

^a Thompson, Coleman, and Helm (1954).

^b Toops (1956).

measurement are given in Table 1 for comparison purposes. The agreement with the literature is better for C₂H₅NO₂ than for CH₃NO₂ and in each case the present values are higher than those previously reported. Extrapolation of literature values for CCl₄ indicate that the present values for CCl₄ are also high. The discrepancy between current and previous values is greater than the usually accepted experimental error of the dipping refractometer, i.e. $\pm 3\frac{1}{2} \times 10^{-5}$

(Weissberger, 1959) to $\pm 4 \times 10^{-5}$ (Berl, 1960). Dissolved air is known to affect the 5th decimal place while the changes in pressure during the present investigation would cause an error of $\pm 0.1 \times 10^{-5}$ (Weissberger, 1959). For the 0.1°C temperature control used in this work, an error of $\pm 5 \times 10^{-5}$ (Weissberger, 1959) is expected. Williams (1925) showed that 0.2% water contamination of nitromethane at 22°C increased the refractive index by $3\text{--}4 \times 10^{-4}$. The consistently high results could be explained by the presence of $\sim 0.5\%$ H_2O in the CH_3NO_2 and (assuming the Williams results are valid for $\text{C}_2\text{H}_5\text{NO}_2$) $\sim 0.1\%$ H_2O in the $\text{C}_2\text{H}_5\text{NO}_2$. These impurities are compatible with the more hygroscopic nature of CH_3NO_2 and with measurements made in the high summer humidity of Carbondale.

In order to ascertain the effect of the error (± 0.005) inherent in the mole fraction, due to volume techniques, a 0.9 mole fraction CH_3NO_2 solution was prepared by weight techniques and the refractive index measured at 35°C . The resulting value of 1.38674 differs from the value given in Table 1 by approximately the same amount as the present values from the literature values.

For each mole fraction, the refractive index was assumed to be a linear function of centigrade temperature, and the coefficients were determined by the method of least squares (Daniels *et al.*, 1962). The values of the intercept, a_0 , and the slope, a_1 , are given in Table 2. Literature values and coefficients calculated from literature data are also given in Table 2 for comparison purposes. For the pure nitroparaffins the intercepts agree to within 0.5% but the intercepts are about 30% high. These results, too, can be understood in terms of water absorption. The temperature coefficient of the refractive index of water is about 20% that of the refractive index of organic liquids (Weissberger, 1959) indicating that at the higher temperature the experimental value should be in closer agreement with the literature determinations. The coefficients obtained for pure CCl_4 are in excellent agreement with previous calculations.

The variation of a_1 with mole fraction indicates a definite minimal value, *circa* 0.5 to 0.6 mole fraction of nitroparaffin. The scatter in this functional behavior is attributed to the fact that a_1 represents a second order effect. The errors due to water impurity are not sufficient to explain the presence of a minimum in this function.

TABLE 2 Coefficients for refractive index as a linear function of centigrade temperature

Mole Fraction (RNO ₂)	Nitromethane		Nitroethane	
	a_0	$-a_1 \times 10^4$ (deg ⁻¹)	a_0	$-a_1 \times 10^4$ (deg ⁻¹)
0.0 ^a	1.47209	6.020	1.47209	6.020
0.0 ^b		5.5		5.5
0.0 ^c		5.74		5.74
0.0	1.47130	5.494	1.47130	5.494
0.1	1.46568	5.334	1.46610	5.550
0.2	1.45913	4.940	1.46061	5.482
0.4	1.44552	4.524	1.44684	4.954
0.5	1.43543	3.514	1.44042	4.896
0.6	1.43083	4.258	1.43316	4.632
0.7	1.42392	4.654	1.42576	4.532
0.8	1.41901	6.188	1.42507	6.844
0.9	1.40798	5.984	1.41500	6.188
1.0	1.39560	5.830	1.40615	6.142
1.0 ^a	1.39149	4.800		
1.0 ^d	1.39130	4.670		
1.0 ^e	1.39088	4.500	1.40070	4.390
1.0 ^f	1.39125	4.648	1.40104	4.426

^a Dreisbach and Martin (1949). 2 points, 20–25 °C.

^b Timmermans (1950).

^c Miller and Lee (1968).

^d Thompson, Coleman, and Helm (1954). 3 points, 20–30 °C.

^e Toops (1956). 3 points, 20–30 °C.

^f Urbánski (1964). 3 points, 20–50 °C.

Partington (1953) gives the following relationship between the differential of the refractive index and the differential of the density, ρ .

$$\frac{dn}{n} = \frac{(n^2 - 1)(n^2 + 2)}{6n^2} \frac{d\rho}{\rho} \quad (1)$$

From Eq. (1), the value of a_1 is found to be the product of a monotonically increasing function (n/ρ) and a monotonically decreasing function (the remaining terms). Conceivably such a product could produce the observed minimum in the $a_1 - x_2$ function. An alternate explanation could be related to the deviations from ideality in these solutions which have been observed in density

(Gunter *et al.*, 1967) and viscosity measurements (Wettaw *et al.*, 1969).

The values of n_D are almost independent of the nitroparaffin component for values of mole fraction of nitroparaffin from 0.0 to 0.5. That is, for these solutions the properties associated with the transmission of light rays is dependent only on the presence of solute molecules, not upon the type of solute molecule.

Using the method of least squares (Daniels *et al.*, 1962), the coefficients were individually determined for refractive index as a linear function of, respectively, mole fraction, weight percent, and volume percent, and for refractive index as a quadratic function of these same concentration variables. The coefficients for the linear functions are given in Table 3 and the coefficients for the quadratic

TABLE 3 Coefficients for refractive index a as linear function of concentration

Temperature (°C)	Nitromethane			Nitroethane		
	a_0	$-a_1$ (conc ⁻¹)	Error Percent	a_0	$-a_1$ (conc ⁻¹)	Error Percent
	n_D vs. x_2					
30	1.459035	0.07463534	0.109	1.457095	0.06628831	0.0559
35	1.456858	0.07515127	0.108	1.454639	0.06674273	0.0575
	n_D vs. w_2					
30	1.450108	0.07712932	0.105	1.450701	0.06741858	0.0905
35	1.447571	0.07741101	0.0890	1.447934	0.06762644	0.0781
	n_D vs. v_2					
30	1.453544	0.07653148	0.0263	1.454532	0.06689609	0.00630
35	1.451008	0.07677309	0.0197	1.451908	0.06714755	0.0110

function are given in Table 4. In addition, the error percent values are given for each function and these values were interpreted by means of the techniques given by Musulin (1971). A comparison of the error percent values in Tables 3 and 4 shows that refractive index as a function of volume percent is a linear function. In general, volume percent is the best independent variable as previously concluded for other systems by MacFarlane and Wright (1933) and Ioffe (1954). For the CH_3NO_2 data weight percent is a slightly better

TABLE 4 Coefficients for refractive index as a quadratic function of concentration

Temperature (°C)	Nitromethane					Nitroethane				
	a_0	$-a_1$ (conc ⁻¹)	a_2 (conc ⁻²)	Error Percent	a_0	$-a_1$ (conc ⁻¹)	a_2 (conc ⁻²)	Error Percent		
30	1.454132	0.03942167	-0.03565941	0.0211	1.454571	0.04816155	-0.01835621	0.00789		
35	1.451177	0.03727901	-0.03787226	0.0226	1.451631	0.04668468	-0.02005804	0.0132		
				n_D vs. x_2						
30	1.454040	0.1087175	0.03333814	0.0194	1.454263	0.09509793	0.02901586	0.0137		
35	1.451260	0.1070946	0.03133756	0.0165	1.451364	0.09390086	0.02746928	0.0148		
				n_D vs. w_2						
30	1.454544	0.08415969	0.007954401	0.00947	1.454702	0.06815061	0.001291054	0.00492		
35	1.451740	0.08221383	0.005646435	0.0119	1.451786	0.06629148	0.0008728648	0.0107		

independent variable than mole fraction for describing refractive index; a conclusion again in agreement with MacFarlane and Wright. However, the converse is true for $C_2H_5NO_2$ data in agreement with Weissberger (1959).

For each temperature, the deviations of experimental CH_3NO_2 refractive index values from predicted values, using the linear function of volume percent, are negative with the amount of deviation decreasing with increasing temperature. The sign of the deviations and their magnitude are consistent with the fact that these deviations are related to volume changes upon mixing (Ioffe, 1954). Conceivably the scatter of $C_2H_5NO_2$ n_D values for the volume percent linear function could be explained in the same manner but the deviations are too small to be significant within the experimental accuracy of the present investigation.

The extremum values for n_D as a quadratic function of weight percent are found outside the range of physically meaningful weight percent values. Consequently, no evidence of a 1:1 complex is found by the methods of Souček (1938). Further, plots of n_D^2 vs. mole fraction gave no extremum in the physically meaningful range of mole fraction indicating no evidence of a 1:1 complex by the methods given by Weissberger (1959). Voronkov and Deich (1965) also attribute lack of additivity of n_D values in certain mixtures to complex formation but present work indicates a weaker interaction in the nitroparaffin mixtures.

The Lorentz-Lorenz molar refractions, R_E , (Weissberger, 1959) were calculated from the observed solution refractive indices and the experimental densities of Gunter *et al.* (1967). The Lorentz-Lorenz molar refractions, R_A , were calculated from the R_E values for the pure components assuming a linear, with respect to mole fraction, addition law (Prutton and Maron, 1951). These calculated values are given in Table 5 along with several literature values for comparison purposes. The usual slight increase of molar refractions with increasing temperature (Partington, 1953) is not clearly demonstrable from the values in Table 5, probably because of the small temperature range. The differences, $R_E - R_A$, at constant temperature are extremely small, except for the 35°C values of 0.4 and 0.5 $C_2H_5NO_2$ solutions. These small changes indicate that deviations from ideality for these solutions are less than average and that there are no

TABLE 5 Molar refractions of carbon tetrachloride-nitroparaffin solutions

Mole Fraction (RNO ₂)	Calculated from Solution		Calculated from Component	
	Refractive Indices, R_E		Refractive Indices, n_A	
	30°	35°	30°	35°
Nitromethane				
0.0	26.45 ^a			
0.0	26.49	26.50	26.49	26.50
0.1	25.09	25.09	25.10	25.11
0.2	23.70	23.72	23.71	23.71
0.3		22.30	22.32	22.32
0.4	20.93	20.95	20.93	20.93
0.5	19.50	19.56	19.55	19.54
0.6	18.15	18.17	18.16	18.15
0.7	16.77	16.79	16.77	16.76
0.8	15.37	15.35	15.38	15.36
0.9	13.99	13.97	13.99	13.97
1.0	12.60	12.58	12.60	12.58
1.0	12.49 ^{b,c}			
Nitroethane				
0.0	26.49	26.50	26.49	26.50
0.1	25.55	25.57	25.55	25.56
0.2	24.63	24.63	24.62	24.62
0.3		23.65	23.68	23.67
0.4	22.73	22.57	22.74	22.73
0.5	21.79	21.62	21.80	21.79
0.6	20.85	20.88	20.86	20.85
0.7	19.93	19.93	19.92	19.91
0.8	19.00	18.95	18.99	18.97
0.9	18.06	18.00	18.05	18.02
1.0	17.11	17.08	17.11	17.08
1.0	17.02 ^c			

^a Vogel (1948). Measured at 25°C.

^b Thompson, Coleman, and Helm (1954).

^c Toops (1956).

perceptible changes in electronic polarizability upon forming these solutions (Weissberger, 1959).

The molar refractions for the pure components calculated in an additive manner from bond refractions (Vogel *et al.*, 1950) are 12.38, 17.03, and 26.04 for CH₃NO₂, C₂H₅NO₂, and CCl₄, respectively. As with other additive properties of this type, CCl₄ shows a decided

deviation between calculated and experimental values, usually attributable to the excessively negative charge cloud (Mumford and Phillips, 1929). The same explanation could be used to explain the moderate deviation for CH_3NO_2 and the lack of deviation for $\text{C}_2\text{H}_5\text{NO}_2$. The difference in deviations for the two nitroparaffin values is also related to the diminishing inductive effect with increasing chain length (Urbánski, 1964). The smallness of the $R_E - R_A$ values indicates that no interaction occurs, upon mixing the pure components, between the negative charge clouds responsible for the deviations from additivity of the pure components.

Since the refractor suggested by Desai (1965) is the same type of additive property as the molar refraction and since no new information was obtained with it, these values are not separately tabulated. The same comments apply to two other new additive properties, R_T (Joshi, 1962) and R_P (Joshi, 1964), related to the refractive index.

4. Summary

The relationship between refractive index and volume percent, at constant temperature, indicates that $\text{CH}_3\text{NO}_2\text{-CCl}_4$ solutions deviate from ideality while $\text{C}_2\text{H}_5\text{NO}_2\text{-CCl}_4$ solutions do not. The deviations are attributed to volume changes upon mixing. The less sensitive criterion of molar refraction indicates no deviation from ideality for either type of solution. The additivity of molar refraction values can be used to predict solution refractive index values for nitroparaffin- CCl_4 solutions. The present investigation could be refined by (1) careful drying of the pure components, and (2) preparing the solutions by weight techniques. These refinements might provide data indicating some non-ideality in $\text{C}_2\text{H}_5\text{NO}_2\text{-CCl}_4$ solutions. A refined experiment is also necessary to determine if the refractive index-temperature functional relationship is a new technique for ascertaining non-ideality.

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