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Densities and Refractive Indices of Aqueous Solutions

Diethylenetriamine, Triethylenetetramine, and Tetraethylenepentamine

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CONTINUING AN INVESTIGATION of the effect of chemical structure on density and refractive index, data have been obtained for aqueous solutions of diethylenetriamine, triethylenetetramine, and tetraethylenepentamine. The purification procedures and methods of analysis described by Chu and Thompson (*4*) were employed.

Refractive index and density data were determined at 25° C. Data on these properties have been reported (*1*) for commercially available compounds but no values have been given for aqueous solutions.

PURIFICATION AND PREPARATION

Following purification by distillation (*4*), the water contents by weight were determined by Karl Fischer reagent to be as follows: diethylenetriamine 0.009%, triethylenetetramine 0.008% and tetraethylenepentamine 0.011%. Within the limits of the experimental measurements, these purified materials gave the same values for density and refractive index as those for pure compounds determined by extrapolation to 100% in each case.

Data for the pure amines are compared in Table I with those reported earlier (*1*) for commercial products.

Solutions covering the entire composition range at approximately 10 weight % increments were prepared according to the established procedure (*4*). Based on amounts of material and the precision possible, compositions were known to within ±0.003%.

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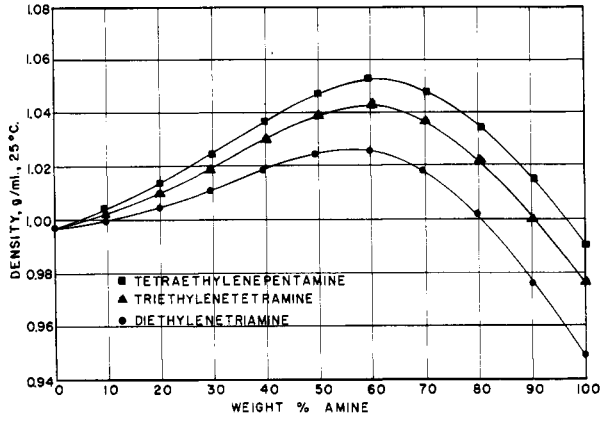

Figure 1. Densities of aqueous amine solutions at 25° C.

DENSITY MEASUREMENTS

Densities of all solutions were determined at 25.00° ±0.01° C. in a constant temperature bath using calibrated 10-ml. Weld-type, capped specific gravity bottles. A precision of ±0.0001 gram per ml. was possible by this procedure. The experimental data for the densities of the three amines are presented in Table II.

The density-composition curves for diethylenetriamine, triethylenetetramine, and tetraethylenepentamine are shown in Figure 1. All three curves show pronounced maxima in the midcomposition range similar to those for many compounds, including aqueous solutions of propylene glycol (*7*), dipropylene glycol (*2*), ethylene glycol monomethyl ether (*4*), diethylene glycol monomethyl ether (*3*) and diethylene glycol monoethyl ether (*3*).

Table I. Properties of Pure Compounds

Compound	Specific Gravity, 25°/25° C. Earlier Data[a](1)	Density, 25° C., G./Ml. Authors	R.I., n_D, 25° C. Earlier Data[a](1)	R.I., n_D, 25° C. Authors
Diethylenetriamine	0.950	0.9492	1.483	1.4815
Triethylenetetramine	0.977	0.9769	1.496	1.4954
Tetraethylenepentamine	0.992	0.9906	1.503	1.5029

[a] Data for commercially available product.

Table II. Experimental Data

Amine, Wt. %	Density, d, G.Ml. at 25° C.	R.I. n_D at 25° C.	Refractivity Intercept $n_D - d/2$ at 25° C.
	Diethylenetriamine		
0	0.99707(6)	1.3325	0.8340
9.13	0.9999	1.3489	0.8490
19.35	1.0045	1.3674	0.8652
28.75	1.0112	1.3855	0.8799
38.56	1.0187	1.4049	0.8956
48.55	1.0250	1.4254	0.9129
58.79	1.0260	1.4436	0.9306
68.99	1.0186	1.4585	0.9492
79.24	1.0020	1.4699	0.9689
89.62	0.9766	1.4775	0.9892
Purified Material (0.009% H_2O)	0.9492	1.4815	1.0069
	Triethylenetetramine		
0	0.99707(6)	1.3325	0.8340
9.20	1.0026	1.3495	0.8482
19.43	1.0105	1.3694	0.8642
28.39	1.0191	1.3881	0.8786
40.04	1.0306	1.4118	0.8965
49.79	1.0392	1.4328	0.9132
60.08	1.0431	1.4541	0.9326
70.02	1.0369	1.4700	0.9516
80.38	1.0220	1.4822	0.9712
90.47	1.0012	1.4904	0.9898
Purified Material (0.008% H_2O)	0.9769	1.4954	1.0070
	Tetraethylenepentamine		
0	0.99707(6)	1.3325	0.8340
8.70	1.0048	1.3514	0.8490
19.60	1.0138	1.3702	0.8633
29.53	1.0250	1.3920	0.8795
39.43	1.0366	1.4132	0.8949
49.28	1.0474	1.4357	0.9120
58.86	1.0527	1.4555	0.9292
70.29	1.0481	1.4755	0.9515
80.74	1.0350	1.4875	0.9700
90.22	1.0154	1.4964	0.9887
Purified Material (0.011% H_2O)	0.9906	1.5029	1.0076

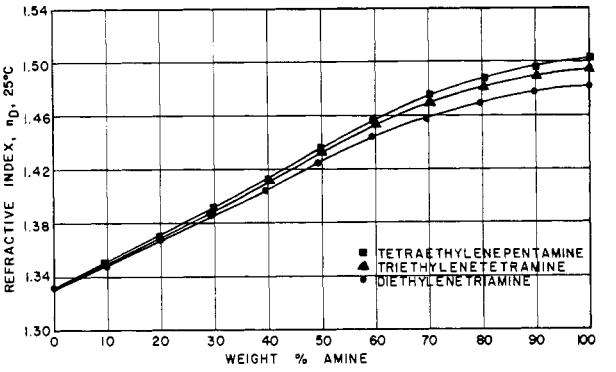

Figure 2. Refractive indices of aqueous amine solutions at 25° C.

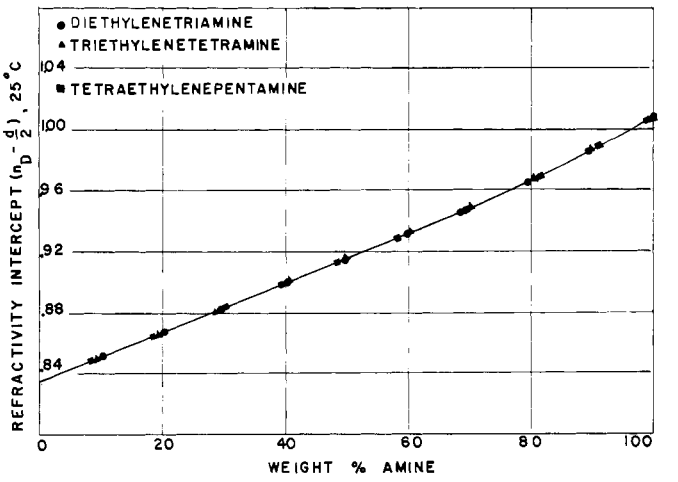

Figure 3. Refractivity intercepts for aqueous amine solutions at 25° C.

REFRACTIVE INDEX

An improved precision Valentine refractometer was used to determine refractive index values to 0.0001 with the temperature controlled at 25.00 ± 0.01° C. (Figure 2, Table II).

It is possible to evaluate the amine content of aqueous solutions of all three compounds to within ±0.15% using a refractive index measurement.

REFRACTIVITY INTERCEPTS

An interesting method of correlating refractive index and density data which appears to be useful has been suggested by Kurtz (5). This consists of plotting the refractivity intercept, $n_D - d/2$, *vs.* the composition of the binary solution. In most cases this procedure gives very close to a linear relationship. As shown in Figure 3, the three amines investigated reveal a striking fact. Within the limits of the experimental measurements the same line fits the data for all three compounds.

This method of correlation emphasizes the value of determining density data simultaneously with refractive index measurements. An article discussing the theoretical basis for the use of the refractivity intercept, in which a number of compounds will be used as examples, is planned.

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