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Viscosities and Densities of Very Concentrated Aqueous Solutions of $AgNO_3$, $TlNO_3$, and $Cd(NO_3)_2$

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Densities and viscosities of aqueous solutions of the mixed melt $(Ag,Tl,Cd)NO_3$ have been obtained at 98.5 °C over the entire water mole fraction (x_w) range. The Ag/Tl ratio was fixed at 1.06, and the Cd mole fraction in the melt was varied between 0 and 0.125. The techniques of measurement are those commonly used at room temperature, and thus the precision of measurement was greater than is usually the case with molten salts. The excess volume V^E of the two-component system (melt + water) is negative under all conditions studied and the V^E-x_w relation is slightly skewed. Since few, if any, electrolyte solution viscosities have been measured in the water-poor region, it is somewhat noteworthy to find that the viscosity-concentration behavior can be represented by a simple semilog relation over most of the x_w range.

Introduction

In the study of electrolyte solutions, water has been regarded as the solvent since solubility usually limits the practical concentration range considered. Conceptually, however, the solubility limit is an accidental phenomenon. It is evident that the search for adequate models of electrolyte solutions may be approached profitably from both the molten-salt and the pure-water ends of the concentration scale. Until recently, the domains of molten salts and electrolyte solutions did not overlap; this, fortunately, is now changing (*1*).

The system $(Ag,Tl)NO_3$ near its eutectic compositions is one of the few which are liquid over the entire concentration range in aqueous solution. Thus, at a constant Ag/Tl ratio of 1.06, the melt enables vapor pressure measurements (for example) to be made below 100 °C (*2*). A third cation (e.g., Cd^{2+}) may be added to the melt, and information on its relative hydration in the system has been obtained (*3*, *4*). The low operating temperatures accessible to these systems mean that physicochemical measurements may be done more precisely than is normally possible. We report here measurements on the densities and viscosities of $(Ag,Tl,Cd)NO_3$ in aqueous solution at 98.5 °C, over the whole water concentration range, and for various cadmium contents in the melt.

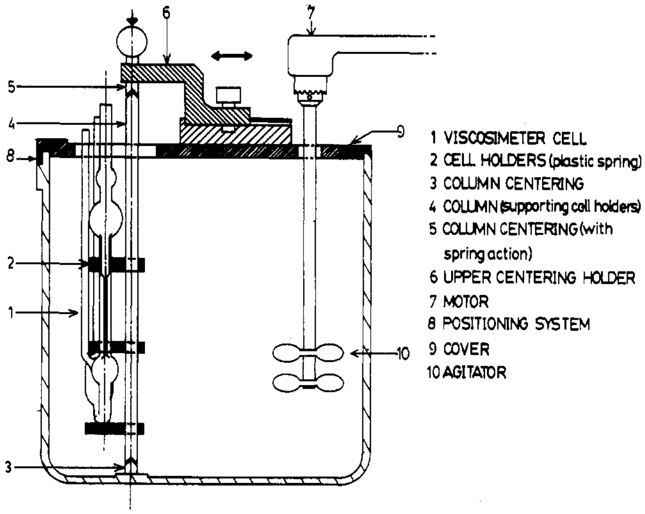

Figure 1. Mounting and alignment of viscometer in bath.

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Table I. Densities, Molar Volumes, and Excess Molar Volumes of Solutions (Ag,Tl,Cd)NO_3 + H_2O at 98.5 °C for Various Cadmium Contents of the Melt

x_w	V, cm^3 mol^{-1}	ρ, g cm^{-3}	V^E, cm^3 mol^{-1}
$X_{Cd} = 0$			
0	46.38	4.672	0
0.1271	42.67	4.486	−0.20
0.1857	40.94	4.384	−0.31
0.2620	38.72	4.252	−0.43
0.3190	37.05	4.137	−0.53
0.3370	36.53	4.099	−0.55
0.3818	35.22	3.998	−0.62
0.4449	33.40	3.842	−0.70
0.5250	31.13	3.611	−0.76
0.6133	28.66	3.309	−0.79
0.6757	26.95	3.059	−0.78
0.7654	24.51	2.637	−0.74
0.8496	22.34	2.144	−0.59
0.9005	21.06	1.794	−0.46
0.9283	20.36	1.584	−0.40
0.9860	19.04	1.092	−0.12
1.0000	18.778[a]	0.9594	0
$X_{Cd} = 0.050$			
0	47.77	4.557	0
0.0762	45.40	4.458	−0.16
0.1143	44.21	4.407	−0.25
0.1887	41.90	4.296	−0.40
0.2502	40.00	4.193	−0.52
0.2865	38.87	4.128	−0.59
0.3206	37.82	4.063	−0.65
0.3398	37.25	4.022	−0.68
0.4119	35.04	3.865	−0.79
0.4757	33.13	3.703	−0.85
0.5464	31.02	3.500	−0.91
0.6016	29.41	3.318	−0.91
0.6450	28.15	3.158	−0.92
0.6698	27.42	3.062	−0.92
0.7876	24.09	2.508	−0.85
0.8474	22.50	2.154	−0.70
0.8993	21.12	1.805	−0.58
0.9297	20.38	1.573	−0.44
1.0000	18.778[a]	0.9594	0
$X_{Cd} = 0.075$			
0	48.45	4.503	0
0.1013	45.21	4.377	−0.23
0.1550	43.52	4.300	−0.34
0.1904	42.37	4.249	−0.43
0.2754	39.68	4.109	−0.60
0.3251	38.11	4.017	−0.69
0.3265	38.06	4.015	−0.70
0.3870	36.17	3.890	−0.80
0.4463	34.33	3.753	−0.88
0.4522	34.16	3.737	−0.87
0.4828	33.20	3.660	−0.92
0.5506	31.15	3.466	−0.96
0.6061	29.52	3.280	−0.95
0.6955	26.84	2.942	−0.97
0.7605	24.95	2.643	−0.93
0.7710	24.65	2.590	−0.92
0.8572	22.28	2.092	−0.74
0.8976	21.21	1.816	−0.61
0.9390	20.16	1.450	−0.43
0.9529	19.81	1.386	−0.37
1.0000	18.778[a]	0.9594	0
$X_{Cd} = 0.100$			
0	49.15	4.449	0
0.1042	45.73	4.325	−0.26
0.1707	43.55	4.235	−0.42
0.2153	42.08	4.170	−0.53
0.2824	39.91	4.059	−0.66
0.3380	38.11	3.958	−0.77
0.3520	37.65	3.931	−0.80
0.4234	35.37	3.781	−0.92
0.5092	32.70	3.562	−0.99
0.5764	30.59	3.368	−1.03
0.6875	27.23	2.964	−1.04
0.7321	25.92	2.769	−0.99
0.7930	24.13	2.467	−0.93
0.8916	21.42	1.857	−0.65
0.9272	20.48	1.593	−0.51
0.9753	19.30	1.190	−0.23
1.0000	18.778[a]	0.9594	0
$X_{Cd} = 0.125$			
0	49.84	4.397	0
0.1355	45.29	4.237	−0.34
0.1865	43.58	4.168	−0.47
0.2401	41.78	4.090	−0.61
0.2685	40.82	4.045	−0.68
0.3149	39.28	3.967	−0.78
0.3994	36.51	3.803	−0.93
0.4543	34.73	3.679	−1.00
0.5365	32.09	3.466	−1.08
0.6070	29.87	3.249	−1.11
0.6752	27.78	3.000	−1.09
0.7119	26.68	2.848	−1.05
0.7611	25.16	2.626	−1.03
0.8233	23.36	2.293	−0.91
0.9010	21.21	1.788	−0.64
0.9542	19.81	1.375	−0.39
1.0000	18.778[a]	0.9594	0

[a] Reference 6.

Experimental Section

Chemicals. $AgNO_3$ (ACS Johnson Mathey) was used as received. $TlNO_3$ (Alfa) was recrystallized 3 times in water, filtered, and dried at 120 °C in vacuo. $Cd(NO_3)_2$ was introduced into the melt as $Cd(NO_3)_2 \cdot 4H_2O$ (Fisher Certified), and the water was driven off by heating in vacuo at about 200 °C. Solutions were prepared by weight with distilled water. The melts were stored in a closed container in a hot-air oven at 150 °C in the absence of light.

Measurements. The densities were measured pycnometrically with a 25-cm^3 Pyrex volumetric flask, calibrated twice with bromobenzene (*5*) (Fisher Certified) at the experimental temperature. The uncertainty of this calibration is ±0.005 cm^3. The flask was placed in a thermostated oil bath at 98.5 ± 0.05 °C. The level of liquid in the flask was observed horizontally with a magnifying glass through the glass wall of the bath and adjusted at the experimental temperature by means of liquid from a preheated pipet. The temperature was measured with calibrated thermometers (EverReady Thermometer Co.). To check this pycnometric method, we made measurements on water between 80 and 100 °C, the density being known with particular precision (*6*). The results were within 0.03% of the published values.

The viscosities were measured with an Ostwald-Ubbelhode viscometer (Cannon Instrument Co.) whose constant, C, was about 0.01 cSt s^{-1}. The flow times, t, lay in the range 300–1000 s, and the kinetic energy correction thus was negligible. The absolute viscosity η was calculated from the relation

$$\eta = \nu\rho = Ct\rho \qquad (1)$$

where ν is the kinematic viscosity and ρ the density. The flow times were determined between 2 and 5 times with an electronic timer (Accusplit, Siliconix, Inc.).

The viscometer constant C was determined with water, aniline, and aqueous glycerin solutions whose densities (*5–7*) and viscosities (*5*, *8*, *9*) are known at several temperatures between 25 and 60 °C. One of these liquids was taken as standard, and the viscosities of the others were reproduced with 1–7 ppt. The measurements were reproducible within 1 ppt,

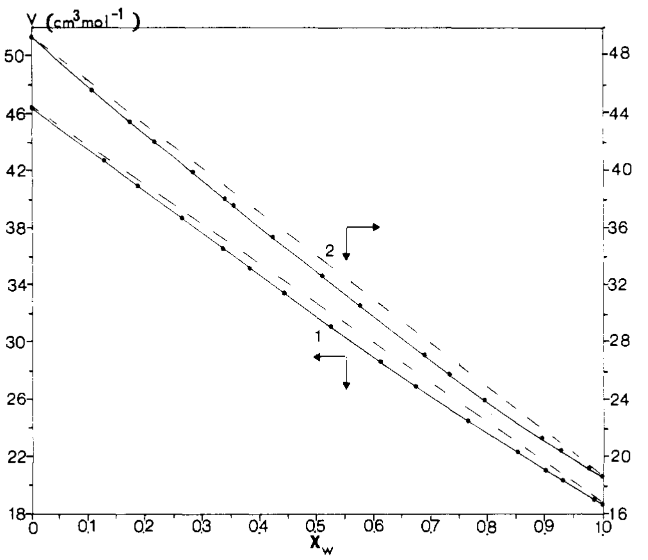

Figure 2. Molar volumes of the title system at 98.5 °C: (1) X_{Cd} = 0; (2) X_{Cd} = 0.100; (···) ideal behavior (eq 2).

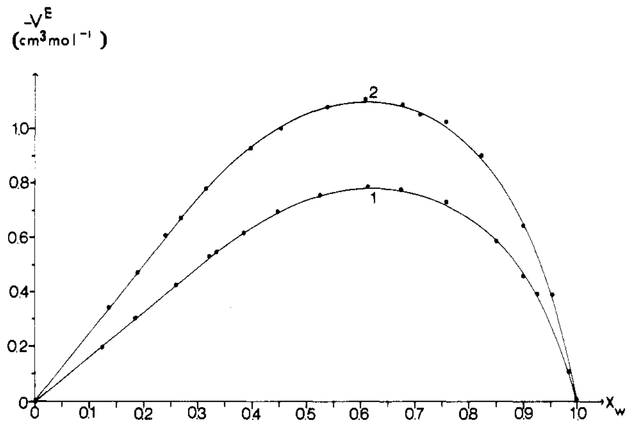

Figure 3. Excess volumes of the title system at 98.5 °C: (1) X_{Cd} = 0; (2) X_{Cd} = 0.125.

Table II. Parameters of Eq 3 Fitting Excess Volumes of Solutions (Ag,Tl,Cd)NO_3 + H_2O at 98.5 °C for Various Cadmium Contents of the Melt

	parameters of eq 3		
X_{Cd}	a, cm³ mol⁻¹	b, cm³ mol⁻¹	r[a]
0	−3.12	−1.43	0.999
0.050	−2.92	−2.05	0.999
0.075	−3.04	−2.18	0.999
0.100	−3.01	−2.45	0.999
0.125	−3.49	−2.49	0.999

[a] r: coefficient of correlation for linear fitting of $V^E/(x_m x_w) = a x_w + b$.

Table III. Experimental Viscosities of Solutions (Ag,Tl,Cd)NO_3 + H_2O at 98.5 °C for Various Cadmium Melt Contents

x_w	η, cP	x_w	η, cP
X_{Cd} = 0			
0	22.53	0.5957	2.14
0.1070	14.40	0.6667	1.61
0.1883	10.39	0.7238	1.25
0.2556	7.90	0.7932	0.93
0.3463	5.62	0.8470	0.71
0.4474	3.84	0.9504	0.43
0.5188	2.91	1.0000	0.287[a]
X_{Cd} = 0.050			
0	30.60	0.6529	2.01
0.1017	20.03	0.6945	1.66
0.2294	11.85	0.7477	1.28
0.3153	8.39	0.8247	0.87
0.3679	6.89	0.8684	0.71
0.4728	4.37	0.9278	0.49
0.5306	3.43	1.0000	0.287[a]
0.5711	2.92		
X_{Cd} = 0.075			
0	35.89	0.5246	3.97
0.0508	28.76	0.5783	3.17
0.1957	15.88	0.6514	2.24
0.2509	12.64	0.6682	2.04
0.3086	9.93	0.7610	1.30
0.3460	8.66	0.8532	0.80
0.4006	6.76	0.9470	0.48
0.4804	4.85	1.0000	0.287[a]
X_{Cd} = 0.100			
0	42.86	0.5769	3.50
0.0811	29.87	0.6176	2.87
0.1468	22.61	0.7072	1.82
0.2552	14.24	0.8090	1.05
0.3610	9.11	0.8707	0.73
0.4242	6.94	0.9522	0.47
0.4556	6.11	1.000	0.287[a]
0.5311	4.29		
X_{Cd} = 0.125			
0	51.37	0.5782	3.83
0.1140	30.00	0.6175	3.12
0.1834	22.62	0.6586	2.54
0.2876	14.20	0.7250	1.76
0.3522	10.64	0.8049	1.13
0.4190	8.03	0.8853	0.68
0.4871	5.83	1.0000	0.287[a]
0.5293	4.83		

[a] Reference *15*.

over a period of 1 h. Water evaporation was negligible over the time of measurements.

More than usual attention was taken in mounting the viscometer in the thermostated bath to ensure perpendicularity of the capillary tube, for each measurement, in spite of repeated handling.

***System of Alignment* (*Figure 1*).** Two of the Teflon spring holders (2) of the viscometer have two holes each, one of which has the same diameter as the support column (4). The other has the same diameter as the capillary tube of the viscometer and has a notch on its front to permit the insertion of the capillary tube by clipping. A small spring, made of music wire, can be inserted in the front of these clips to ensure better positioning. These clips (2) are aligned on the support column (4) on which they are attached by cotter pins to prevent them from moving. A sliding upper centering holder (6) is installed on the cover (9) which can be stopped, bolted against a ridge, in order to maintain its identical forward position. It is moved back to clear the hole through which the viscometer and its support column (4) can be introduced or removed. There is a spring-loaded knob (5) on top of this slide, allowing for vertical thermal expansion, with a female cone at its lower end. The support column (4) can be positioned accurately as it rests on the male cone (3) glued on the bottom of the tank.

***General Assembly*.** The Pyrex tank is glued in position on the workbench. The cover (9) has a ridge which fits against the inner wall of the tank, but not too tightly to allow for thermal expansion. The final rigorous positioning of the cover is ensured by three parts (8), female V glued on the tank and male V on the cover, preventing any movement, especially rotation. The sliding element (6) being pushed foward, the knob (5) is lifted and replaced by a bushing pierced in its center with a hole large enough for the insertion of a plumb line. The bottom rest (3) of the support axis (4) is then glued on the tank, just under the point of the plumb line.

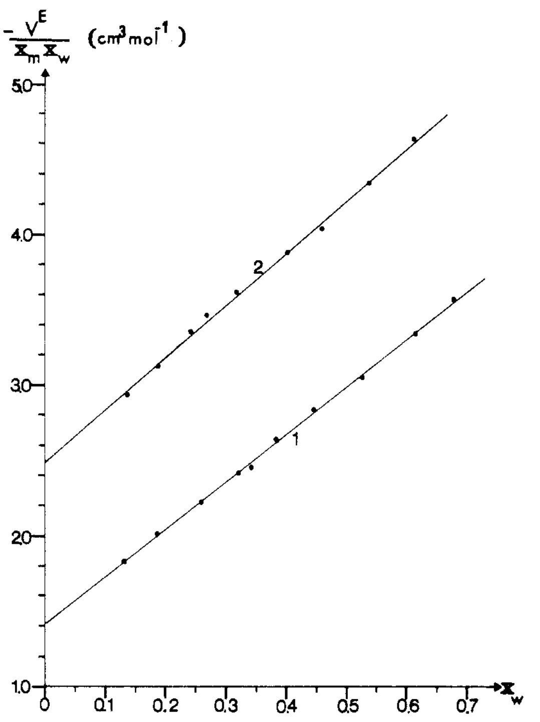

Figure 4. $-V^E/(x_m x_w)$ as a function of x_w of the title system at 98.5 °C: (1) $X_{Cd} = 0$; (2) $X_{Cd} = 0.125$.

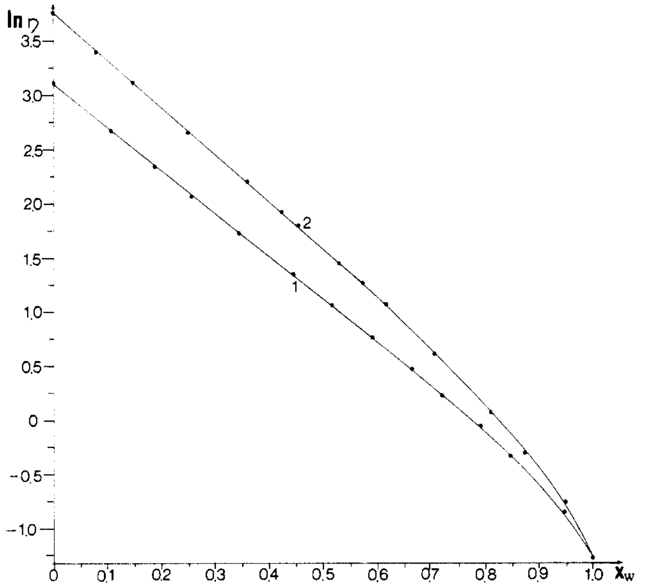

Figure 5. ln of absolute viscosities of the title system at 98.5 °C: (1) $X_{Cd} = 0$; (2) $X_{Cd} = 0.100$.

In this work, viscosities are measured relative to the viscosity of water at 30 °C.

Results and Dicussion

Concentrations are reported here in mole fractions. Molecular weights used in the calculations are as follows: $AgNO_3$, 169.87; $TlNO_3$, 266.37; $Cd(NO_3)_2$, 236.41; H_2O, 18.015. For convenience, the solutions are considered to be two component, i.e., water and the melt. For example, since the Ag/Tl mole ratio is always 1.06, the molecular weight of the melt $(Ag,Tl)NO_3$ (without cadmium) is (0.515)(169.87) + (0.485)(266.37) = 216.68 g mol^{-1}. The mole fraction of water is x_w and the mole fraction of cadmium *in the melt* is X_{Cd}.

Table IV. Parameters of Eq 4 Fitting Viscosities of Solutions $(Ag,Tl,Cd)NO_3 + H_2O$ at 98.5 °C for Various Cadmium Contents of the Melt

X_{Cd}	parameters of eq 4 $-c$	 d	$x_w(\max)$[a]	r[b]
0	3.938	3.095	0.5	0.9999
0.050	4.108	3.420	0.6	0.9999
0.075	4.181	3.583	0.6	0.9999
0.100	4.336	3.761	0.6	0.9999
0.125	4.454	3.932	0.6	0.9999

[a] Upper limit of water mole fraction, beyond which [ln η(exptl) − ln η(calcd)]/ln η(exptl) > 0.01. [b] r: coefficient of correlation for linear fitting.

Densities. Experimental values of density, molar volume, and excess molar volume are given in Table I.

It is more revealing to present the data in terms of the excess volume V^E, viewing the solution as the result of mixing two liquids. Thus $V^E = V - V^{id}$, where V^{id} is the molar volume of an ideal solution. If V_m° and V_w° are the molar volumes of melt and water respectively, then

$$V^{id} = x_m V_m^\circ + x_w V_w^\circ \tag{2}$$

The data may be represented adequately, in the concentration range $0 < x_w < 0.7$, by the equation

$$V^E = x_m x_w (a x_w + b) \tag{3}$$

where $x_m = 1 - x_w$ and the parameters are given in Table II. The V, V^E, and $V^E/(x_m x_w)$ data for two of the systems are shown in Figures 2–4.

The excess volumes of all systems measured in this work are negative. This water contraction effect is quite common and has been observed in other electrolyte solutions (*1*) studied over wide x_w ranges: $(Li,K)NO_3$, $(NH_4,Li)NO_3 + NH_4Cl$, and $Ca(NO_3)_2$. In the latter cases, a linear dependence of V upon x_w in concentrated solution has been claimed (*1*). A glance at Figures 2 and 3 shows that V and V^E are not linear for the systems studied here.

Viscosities. The viscosity (η) data for solutions under the same conditions as in the previous section are presented in Table III. The relation ln η − x_w is linear in a large concentration range. The data representing the linear portion of the curve are fitted to the equation

$$\ln \eta(cP) = c x_w + d \tag{4}$$

and the parameters appear in Table IV. The column labeled $x_w(\max)$ gives the upper x_w limit, beyond which the experimental and calculated values of ln η deviate by more than 1%. Data for two of the systems are plotted in Figure 5. This is the first time, we believe, such an observation is made.

The most successful models for the transport properties of concentrated electrolyte solutions have been derived from transition-state theory (*1*). The empirical Vogel–Tammann–Fulcher equation was applied successfully by Angell and Bressel (*10*) to account for both concentration and temperature dependences. Another transition-state treatment is due to Goldsack (*11*, *12*). These and other theories have been applied (*13*, *14*) with more or less success, for solutions of water mole fractions more than about 0.73. It can be seen from Figure 5 that the simple semilog relation, eq 4, applies in just that concentration region not previously covered, i.e., $x_w < 0.7$.

Registry No. $AgNO_3$, 7761-88-8; $TlNO_3$, 10102-45-1; $Cd(NO_3)_2$, 10325-94-7.

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Binary Systems of 1,1,2,2-Tetrachloroethane with Benzene, Toluene, *p*-Xylene, Acetone, and Cyclohexane. 1. Excess Volumes, Ultrasonic Velocities, and Adiabatic Compressibilities at 298.15 and 308.15 K

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Measurements of excess volumes (V^E), ultrasonic velocities (u), and adiabatic compressibilities (k_s) at 298.15 and 308.15 K have been made for binary liquid mixtures of 1,1,2,2-tetrachloroethane ($CHCl_2CHCl_2$) with benzene, toluene, *p*-xylene, acetone, and cyclohexane. The values of V^E at both temperatures have been fitted by the method of least squares to the equation $V^E = x_1x_2[A_0 + A_1(x_1 - x_2) + A_2(x_1 - x_2)^2]$ where x_1 refers to the mole fraction of $CHCl_2CHCl_2$, and A_0, A_1, and A_2 are constants characteristic of a system. The values of the quantity k_s^E, which refers to the deviations of the experimental values of the adiabatic compressibilities of the mixtures from the mole fraction mixture law values, have also been calculated and have been fitted by the method of least squares to the equation $k_s^E = x_1x_2[B_0 + B_1(x_1 - x_2) + B_2(x_1 - x_2)^2]$ where x_1 refers to the mole fraction of $CHCl_2CHCl_2$, and B_0, B_1, and B_2 are constants characteristic of a system. At both temperatures 298.15 and 308.15 K, values of V^E have been found to be negative for $CHCl_2CHCl_2$–benzene, $CHCl_2CHCl_2$–toluene, $CHCl_2CHCl_2$–*p*-xylene, and $CHCl_2CHCl_2$–acetone and positive for $CHCl_2CHCl_2$–cyclohexane. Further, the values of k_s^E at 298.15 and 308.15 K have been found to be negative for $CHCl_2CHCl_2$–benzene, $CHCl_2CHCl_2$–toluene, $CHCl_2CHCl_2$–acetone, and $CHCl_2CHCl_2$–cyclohexane and positive for $CHCl_2CHCl_2$–*p*-xylene. These results indicate the existence of specific interaction of $CHCl_2CHCl_2$ with the aromatic hydrocarbons and acetone.

Introduction

Binary systems of 1,1,2,2-tetrachloroethane ($CHCl_2CHCl_2$) with aromatic hydrocarbons, acetone, and cyclohexane are of considerable interest from the viewpoint of the existence of specific interaction between the components in the liquid state. The specific interaction of $CHCl_2CHCl_2$ with aromatics and acetone can be visualized to be due to the presence of four Cl atoms and two H atoms in $CHCl_2CHCl_2$ on account of which it can act as a σ acceptor toward, and be involved in hydrogen-bond formation with, the aromatics and acetone. The aromatics in their interaction with $CHCl_2CHCl_2$ will act as π donors, whereas acetone will act as an n donor. The system of cyclohexane with $CHCl_2CHCl_2$, in which case only the dispersion, dipolar, and induction forces are believed to be present, is of interest as it will act as a reference system. Extensive studies concerning the properties of these systems have not been made. In the present program, we have made measurements of ultrasonic velocities in, plus adiabatic compressibilities and excess volumes for, the binary liquid mixtures of $CHCl_2CHCl_2$ with benzene, toluene, *p*-xylene, acetone, and cyclohexane at 298.15 and 308.15 K, and the results obtained have been interpreted in this paper.

Table I. Values of Ultrasonic Velocities in, and Adiabatic Compressibilities for, Various Pure Liquids at 303.15 K

	u, m s^{-1}		10^6k_s, atm^{-1}	
liquid	this work	lit. value^a	this work	lit. value^a
benzene	1277	1278	71.6	71.5
toluene	1283	1284.5	71.8	71.8
acetone	1144	1146	99.3	99.1
chloroform	968	967.5	73.5	73.7
carbon tetrachloride	906	904	78.4	78.8

^{a} See ref *6*.

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