three thermodynamic properties for *n*-hexane, cyclohexane, and benzene measured previously by us (*4*, *6*, *9*, *10*). Results of this work are compared with the literature data (in parentheses). All results are in agreement with literature data except α for bicyclohexyl, cyclohexylbenzene, and tetralin. This is due to the fact that our values of α at 298.15 K are obtained by measuring 12 volumes between 296.65 and 299.65 K while Letcher and Baxter (*1*) obtain them with less measurements of density and in a wider range of temperatures (between 283.15 and 313.15 K).

The isentropic compressibility $\kappa_S = -V^{-1}(\partial V/\partial p)_S$ is related to the speed of sound through the density ρ of the substance by

$$\kappa_S = 1/\rho u^2 \qquad (1)$$

Values of κ_S calculated for all substances measured in this work are reported in the last column of Table I. Results of the internal pressure coefficient $\gamma = \alpha/\kappa_T$ can be easily obtained from the data of Table I, and the values are therefore omitted.

Registry No. Hexane, 110-54-3; 1-hexene, 592-41-6; 1-hexyne, 693-02-7; cyclopentane, 287-92-3; cyclohexane, 110-82-7; cyclohexene, 110-83-8; 1,3-cyclohexadiene, 592-57-4; 1,4-cyclohexadiene, 628-41-1; benzene, 71-43-2; bicyclohexyl, 92-51-3; cyclohexylbenzene, 827-52-1; tetralin, 119-64-2.

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Excess Volumes, Ultrasonic Sound Velocities, and Isentropic Compressibilities of 1-Chlorobutane with Isopropyl, Isobutyl, and Isopentyl Alcohols

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New experimental data on excess volumes, sound velocities, and isentropic compressibilities for the mixtures of 1-chlorobutane with isopropyl, isobutyl, and isopentyl alcohols are reported. The sound velocities have been analyzed in terms of free length theory (FLT) and collision factor theory (CFT). The positive excess volumes and positive deviations in isentropic compressibilities have been attributed to the dissociation of self-associated alcohols and chlorobutane.

Introduction

Excess volumes, sound velocities, and isentropic compressibilities of 1-chlorobutane with normal alcohols have been reported by us (*1*, *2*). To study the effect of branching of alcohols on these properties, further measurements on excess volumes, sound velocities, and isentropic compressibilities have been carried out. We report here excess volumes (at 303.15 and 313.15 K), sound velocities, and isentropic compressibilities (at 303.15 K) of 1-chlorobutane with isoalcohols. The isoalcohols include isopropyl, isobutyl, and isopentyl alcohols. The sound velocities have been analyzed in terms of Jacobson's free length theory (FLT) (*3*, *4*) and Schaaf's collision factor theory (CFT) (*5*, *6*).

Table I. Boiling Points and Densities of Pure Components along with the Literature Values

component	boiling point/K expt	boiling point/K lit. (*10*)	density(303.15 K)/(g·cm^{-3}) expt	density(303.15 K)/(g·cm^{-3}) lit. (*11*)
1-chlorobutane	381.40	381.59	0.87543	0.87549
isopropyl alcohol	385.20	385.41	0.77692	0.77690
isobutyl alcohol	410.55	410.81	0.79439	0.79437
isopentyl alcohol	433.30	433.65	0.80177	0.80179

Experimental Section

Excess volume (V_m^E) was measured by using the batch dilatometers described earlier (*7*, *8*). The mixing cell contained two bulbs of different capacities that were connected through a U-tube having mercury to separate the two compartments. One end of the first bulb was fitted with a capillary outlet and the opposite end of the second bulb was closed with a ground glass stopper. Four dilatometers of this type were used to cover the whole range of composition. The composition of each mixture was determined directly by weighing. All weights were corrected for buoyancy. The V_m^E values were accurate to ±0.003 cm^3·mol^{-1}. The ultrasonic sound velocity was measured with a single-crystal interferometer at a frequency

Table II. Excess Volumes, $V_m^E/(\mathrm{cm^3 \cdot mol^{-1}})$, of 1-Chlorobutane with Isoalcohols at 303.15 and 313.15 K

303.15 K		313.15 K	
x	$V_m^E/(\mathrm{cm^3 \cdot mol^{-1}})$	x	$V_m^E/(\mathrm{cm^3 \cdot mol^{-1}})$
1-Chlorobutane + Isopropyl Alcohol			
0.1126	0.150	0.0789	0.142
0.2162	0.269	0.2762	0.368
0.3004	0.344	0.2957	0.402
0.4216	0.415	0.4215	0.492
0.5449	0.457	0.5056	0.530
0.6584	0.444	0.6483	0.552
0.8113	0.340	0.6637	0.545
0.8222	0.329	0.8678	0.366
1-Chlorobutane + Isobutyl Alcohol			
0.1438	0.120	0.1278	0.152
0.2326	0.183	0.2839	0.282
0.2702	0.215	0.3399	0.321
0.4119	0.280	0.5719	0.382
0.5972	0.310	0.7259	0.333
0.6749	0.285	0.7307	0.317
0.7528	0.244	0.8350	0.226
0.8249	0.176	0.8631	0.195
1-Chlorobutane + Isopentyl Alcohol			
0.1355	0.055	0.1319	0.076
0.2672	0.105	0.2346	0.132
0.3035	0.115	0.2700	0.162
0.3746	0.140	0.3540	0.186
0.5491	0.160	0.3602	0.192
0.6986	0.128	0.5634	0.231
0.7254	0.120	0.7239	0.179
0.8483	0.068	0.8735	0.096

of 2 MHz, and the data were accurate to ±0.15% (*9*). Isentropic compressibilities were calculated from precise densities and sound velocity data determined at 303.15 K. Density was computed from the measured excess volume data, using the relation

$$\rho = \frac{x_1M_1 + x_2M_2}{V_m + V_m^E} \qquad (1)$$

where ρ is the density, x_i, the mole fraction, M_i, the molecular weight, and V_m the molar volume of the ideal mixture. All the measurements were made at a constant temperature, employing a thermostat that could be maintained to ±0.01 K.

Purification of Materials

1-Chlorobutane supplied by Riedel was further purified by refluxing with concentrated sulfuric acid, washing several times with water, drying over two portions of calcium chloride, and fractionally distilling. All the three isoalcohols supplied by BDH, England, were further purified by the methods described by Riddick and Bunger (*10*). The isoalcohols were refluxed over freshly ignited lime for 6 h and fractionally distilled, collecting the middle fraction. The middle fraction so collected is distilled again. The purity of the samples was checked by comparing the measured boiling points and densities of the components with those reported in the literature (*11*, *12*). Densities were determined by using a bicapillary-type pycnometer with an accuracy of 2 parts in 10^5. The measured boiling points and density data are reported in Table I along with those reported in the literature. The differences between the experimental boiling points and densities of the pure substances and the literature data fall within the experimental error, as seen from the Table I.

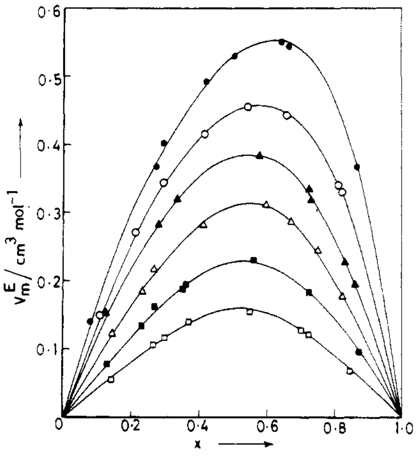

Figure 1. Excess volumes V_m^E plotted against mole fraction of 1-chlorobutane at 303.15 and 313.15 K for the mixtures of 1-chlorobutane with isoalcohols: ○, isopropyl alcohol at 303.15 K; ●, isopropyl alcohol at 313.15 K; △, isobutyl alcohol at 303.15 K; ▲, isobutyl alcohol at 313.15 K; □, isopentyl alcohol at 303.15 K; ■, isopentyl alcohol at 313.15 K; —, calculated from smoothing eq 2 by using the data given in Table III.

Results

Experimental V_m^E values for the three binary mixtures are presented in Table II and are also graphically represented in Figure 1. The values of V_m^E are well represented by the following empirical equation truncated to third or fourth term

$$V_m^E/(\mathrm{cm^3 \cdot mol^{-1}}) = x(1 - x)\sum_{i=0} A_i(2x - 1)^i \qquad (2)$$

where x is the mole fraction of 1-chlorobutane and A_i are the adjustable parameters. The values of the parameters obtained

Table III. Values of the Parameters A_i (in $\mathrm{cm^3 \cdot mol^{-1}}$) for Eq 2, the Standard Deviation $\sigma(V_m^E)$, and the Maximum Deviation, $\delta_{max}(V_m^E)$

system	T/K	A_0	A_1	A_2	A_3	$\sigma(V_m^E)$	$\delta_{max}(V_m^E)$
1-chlorobutane + isopropyl alcohol	303.15	1.7893	0.4723	0.2655	0.1640	0.0029	0.0047
	313.15	2.1052	0.8578	0.8109		0.0070	0.0086
1-chlorobutane + isobutyl alcohol	303.15	1.2407	0.3293	−0.2985	−0.3505	0.0047	0.0059
	313.15	1.5209	0.3245	−0.0330	−0.2471	0.0048	0.0070
1-chlorobutane + isopentyl alcohol	303.15	0.6281	0.0971	−0.2648	−0.1274	0.0024	0.0028
	313.15	0.9070	0.1426	−0.2663		0.0056	0.0077

Table IV. Values of Molar Volume V_m, Molar Volume at Absolute Zero V_0, Available Volume V_a, Free Length L_f, Surface Area Y, Collision Factor S, and Molecular Radius r_m of the Pure Liquid Components at 303.15 K

component	V_m/ $(\mathrm{cm^3 \cdot mol^{-1}})$	V_0/ $(\mathrm{cm^3 \cdot mol^{-1}})$	V_a/ $(\mathrm{cm^3 \cdot mol^{-1}})$	L_f/Å	Y	S	r_m/Å
1-chlorobutane	105.741	82.704	23.037	0.6165	74.740	1.4900	2.6793
isopropyl alcohol	77.353	58.921	18.432	0.6335	58.189	1.6497	2.3589
isobutyl alcohol	93.309	73.262	20.047	0.6041	66.373	1.6585	2.5373
isopentyl alcohol	109.946	88.076	21.869	0.5776	75.721	1.6738	2.7077

Table V. Values of Free Lengths of Mixtures, L_f^M, and Experimental and Predicted Sound Velocities for the Binary Mixtures of 1-Chlorobutane with Isoalcohols at 303.15 K

		$U/(\mathrm{m \cdot s^{-1}})$			% ΔU	
x	$L_f^M/\text{Å}$	FLT	CFT	exptl	FLT	CFT
1-Chlorobutane + Isopropyl Alcohol						
0.1126	0.6361	1116	1128	1128	−1.06	0.00
0.2162	0.6378	1105	1125	1121	−1.43	0.36
0.3004	0.6384	1098	1122	1115	−1.53	0.63
0.4216	0.6380	1091	1118	1109	−1.62	0.81
0.5449	0.6368	1085	1113	1106	−1.90	0.63
0.6584	0.6341	1083	1108	1102	−1.72	0.55
0.8113	0.6286	1084	1102	1099	−1.37	0.27
0.8222	0.6281	1084	1101	1098	−1.28	0.27
					av dev −1.49	av dev 0.44
1-Chlorobutane + Isobutyl Alcohol						
0.1438	0.6096	1153	1161	1156	−0.26	0.43
0.2326	0.6126	1142	1154	1147	−0.44	0.61
0.2702	0.6140	1137	1150	1143	−0.52	0.61
0.4119	0.6176	1123	1139	1130	−0.62	0.80
0.5972	0.6205	1108	1124	1114	−0.54	0.90
0.6749	0.6207	1104	1118	1109	−0.54	0.81
0.7528	0.6204	1100	1113	1104	−0.36	0.82
0.8249	0.6193	1098	1107	1102	−0.36	0.45
					av dev −0.46	av dev 0.68
1-Chlorobutane + Isopentyl Alcohol						
0.1355	0.5843	1199	1202	1198	0.08	0.34
0.2672	0.5907	1180	1185	1176	0.34	0.77
0.3035	0.5924	1175	1181	1170	0.43	0.94
0.3746	0.5958	1164	1171	1158	0.52	1.12
0.5491	0.6031	1141	1149	1134	0.62	1.32
0.6986	0.6081	1125	1131	1118	0.63	1.16
0.7254	0.6089	1122	1128	1114	0.72	1.26
0.8483	0.6123	1109	1113	1104	0.45	0.82
					av dev 0.47	av dev 0.99

by least-squares analysis are given in Table III together with standard deviation $\sigma(V_m^E)$ and maximum deviation $\delta_{\max}(V_m^E)$.

The theoretical aspects of FLT and CFT have been discussed in detail in our earlier publications (*13*, *14*). The surface area and collision factor of the pure components, used in FLT and CFT, were calculated by using experimental sound velocities and densities. Critical temperature and surface tension data of the pure components, required for calculation of molar volume at absolute zero, and the molecular radius were taken from the literature (*15*, *16*). The data for molar volume, V_m, molar volume at absolute zero, V_0, available volume, V_a, free length, L_f, surface area, Y, collision factor, S, and molecular radius r_m of the pure components at 303.15 K are given in Table IV. Sound velocity data predicted in terms of FLT and CFT are given in Table V along with the experimental results in columns 2, 3, and 4. The percentage deviation between the experimental sound velocity data and those of FLT and CFT values are also given in columns 5 and 6.

Isentropic compressibility, K_s, was calculated by using the relation

$$K_s = U^{-2}\rho^{-1} \tag{3}$$

where U and ρ denote the sound velocity and density, respectively. The values of K_s are accurate to ±2 TPA^{-1}. The deviation in isentropic compressibility from the ideal value, assumed to be additive in terms of volume fraction, is estimated by using the equation

$$\Delta K_s = K_s - \{\phi K_{s,1} + (1 - \phi)K_{s,2}\} \tag{4}$$

where K_s, $K_{s,1}$, and $K_{s,2}$ are the isentropic compressibilities of the mixtures and the pure components, respectively. ϕ is the volume fraction of 1-chlorobutane.

Table VI. Volume Fraction, ϕ, of 1-Chlorobutane, Density ρ, Sound Velocity U, Isentropic Compressibility K_s, and Values of ΔK_s Computed from Eq 4 for 1-Chlorobutane + Isoalcohols at 303.15 K

ϕ	$\rho/(\mathrm{g \cdot cm^{-3}})$	$U/(\mathrm{m \cdot s^{-1}})$	$K_s/\mathrm{TPa^{-1}}$	$\Delta K_s/\mathrm{TPa^{-1}}$
1-Chlorobutane + Isopropyl Alcohol				
0.0000	0.77692	1130	1008	0
0.1478	0.79008	1128	995	3
0.2738	0.80131	1121	993	5
0.3699	0.81010	1115	992	7
0.4991	0.82226	1109	989	8
0.6207	0.83396	1106	980	7
0.7249	0.84442	1102	975	5
0.8546	0.85820	1099	965	3
0.8634	0.85917	1098	963	2
1.0000	0.87543	1094	954	0
1-Chlorobutane + Isopentyl Alcohol				
0.0000	0.80177	1220	838	0
0.1310	0.81101	1198	860	7
0.2596	0.82013	1176	882	14
0.2953	0.82265	1170	888	16
0.3655	0.82763	1158	904	21
0.5394	0.84026	1134	925	25
0.6903	0.85161	1118	940	22
0.7176	0.85367	1114	947	20
0.8432	0.86334	1104	952	14
1.0000	0.87543	1094	954	0
1-Chlorobutane + Isobutyl Alcohol				
0.0000	0.79439	1172	917	0
0.1600	0.80633	1156	928	5
0.2557	0.81356	1147	934	8
0.2956	0.81652	1143	937	9
0.4425	0.82789	1130	946	13
0.6269	0.84260	1114	956	16
0.7017	0.84889	1109	958	15
0.7753	0.85520	1104	958	12
0.8422	0.86119	1102	956	8
1.0000	0.87543	1094	954	0

Experimental data for the density, sound velocity, isentropic compressibility, and ΔK_s are included in Table VI. The dependence of U and ΔK_s on volume fraction is expressed by eq 5 and 6, respectively, where B_i are the adjustable parameters.

$$U = \sum_{i=0} B_i \phi^i \tag{5}$$

$$\Delta K_s = \phi(1 - \phi)\sum_{i=0} B_i(2\phi - 1)^i \tag{6}$$

The values of B_i, calculated by the method of least-squares, are given in Table VII along with the standard deviation $\sigma(U,\Delta K_s)$ and maximum deviation $\delta_{\max}(U,\Delta K_s)$.

Discussion

The values of V_m^E given in Table I are positive over the entire range of composition at both the temperatures. The positive excess volumes may be explained in terms of relative strengths of two opposing effects: (1) dissociation of self-associated alcohols and chlorobutane (*17*); (2) interstitial accommodation of chloroalkane molecules in the aggregates of alcohols and weak hydrogen-bond interactions (*7*, *8*, *18*) of the type Cl···H–O between the molecules of unlike components. While the former effect contributes to expansion in volume, the latter effect leads to contraction in volume. The positive excess volumes, observed for the three mixtures, suggest that the former effect is dominant.

The values of V_m^E of the mixtures containing isoalcohols are more positive than those observed for the corresponding 1-alcohols. This suggests that the branching of alcohols mitigates the strength of the hydrogen-bonding interaction between like molecules and also that between unlike molecules. The positive V_m^E values fall in the order isopropyl alcohol > isobutyl alcohol > isopentyl alcohol. This order is similar to that observed for the corresponding 1-alcohols. The data presented in Table II

Table VII. Values of the Parameters B_i of Eq 5 and 6, Standard Deviations, σ, and Maximum Deviation, δ_{max}, for 1-Chlorobutane with Isoalcohols at 303.15 K

system	property	B_0	B_1	B_2	B_3	σ	δ_{max}
1-chlorobutane + isopropyl alcohol	$U/(m \cdot s^{-1})$	1139.73	-86.98	65.07	-22.89	0.7	0.7
	$\Delta K_s/TPa^{-1}$	30.63	-1.39	-18.03		0.4	0.5
1-chlorobutane + isobutyl alcohol	$U/(m \cdot s^{-1})$	1168.82	-71.72	-70.13	71.96	0.5	0.6
	$\Delta K_s/TPa^{-1}$	60.12	43.96	-22.05	-58.23	0.4	0.5
1-chlorobutane + isopentyl alcohol	$U/(m \cdot s^{-1})$	1224.57	-209.86	79.03		0.6	0.7
	$\Delta K_s/TPa^{-1}$	95.77	29.10	-25.77		0.8	1.0

also reveal that the temperature coefficient of V_m^E is positive in all the three mixtures. This may be ascribed to a decrease in degree of self-association of pure components and association between unlike molecules with increasing temperature.

The sound velocity data presented in Table V show that the agreement between estimated values and experimental data is good. Maximum average deviation in case of FLT observed is 1.5% whereas the maximum individual deviation is 1.9%. In the case of CFT maximum individual deviation is 1.32% and maximum average deviation is 1%. Hence, it can be concluded that both FLT and CFT can satisfactorily estimate sound velocities of mixtures of haloethane with isoalcohols.

The values of ΔK_s given in Table VI are positive over the whole range of mole fraction. As in the case of V_m^E, positive ΔK_s values may also be explained in terms of (1) dissociation of self-associated alcohols and chlorobutane, and (2) interstitial accommodation of chloroalkane molecules in the aggregates of alcohols and weak hydrogen-bond interactions between the unlike molecules. The first effect contributes to an increase in free lengths described by Jacobson (*19*) (as is seen in Tables IV and V). This leads to negative deviation in U and positive deviation in K_s. The second effect, on the other hand, contributes to positive deviation in U and negative deviation in K_s. The actual value of the deviation would depend upon the relative strengths of the two opposing effects. The experimental values of ΔK_s, which are positive, point out that the first effect is stronger than the latter. This supports our earlier observations that the dissociation of self-associated alcohols and chlorobutane dominates over the other effect.

Registry No. 1-Chlorobutane, 109-69-3; isopropyl alcohol, 67-63-0; isobutyl alcohol, 78-83-1; isopentyl alcohol, 123-51-3.

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NEW COMPOUNDS

Synthesis and Spectral Studies of Some New Sulfides and Sulfones

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Sixteen new substituted benzylphenyl sulfides and sulfones have been synthesized. Their structures were proved by IR, ^{1}H NMR, and mass spectra. A linear relationship between δ-values of the benzylic protons and σ-(Hammett) values of the phenyl substituents has been found.

The therapeutic effects (*1*) of sulfides and sulfones together with their importance toward reagents have long been known since their synthesis (*2*, *6*).

Also, in the course of a study of the displacement reactions (*7*, *8*), and nucleophilic reactivities (*9*), a series of substituted benzylphenyl sulfides and sulfones were required. Therefore, we decided to study the synthesis of some new sulfides (**1a–h**) and new sulfones (**2a–h**), and investigate their structure by IR, ^{1}H NMR, and mass spectra.

Sulfides (**1a–h**) and sulfones (**2a–h**) have been prepared by conventional procedures (**10**, **11**), although in individual cases, experimental modifications were important for high yield of products.

The mass spectra (*12*, *13*) of (**1b**) and (**1h**) were studied. The relative intensities of the most prominent peaks in their fragmentation patterns are recorded in Table I.

IR spectra showed for sulfides and sulfones the expected bands. For example, the infrared spectra of sulfones showed, besides other, strong absorption bands at 1350–1300 cm^{-1} and at 1150 cm^{-1}. These bands arise from asymmetric and sym-

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