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Ultrasonic speeds and thermodynamic properties for pentafluoropropyl alcohol, tetrafluoropropyl alcohol and trifluoroethyl alcohol under high pressures

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Abstract

The ultrasonic speeds u in pentafluoropropyl alcohol 5FP, tetrafluoropropyl alcohol 4FP and trifluoroethyl alcohol 3FE were measured by a sing-around technique operated at a frequency of 2 MHz. The measurements were carried out in the ranges of temperature from 283–348 K and pressure up to about 70 MPa with an uncertainty within $\pm 0.2\%$. The results for these compounds show lower absolute u values and smaller pressure dependence than those for primary alcohols. Among the compounds studied, the u values for 4FP differ greatly in their pressure effect from those for 5FP and 3FE. The relationship between $1/u(\partial u/\partial p)_T$ and isothermal compressibility κ_T for these alcohols is fitted well by a straight line having a slope of 5 ± 0.2, as well as those for other alcohols; this slope is distinguishable from that of 4 ± 0.15 for other hydrogen compounds reported elsewhere.

INTRODUCTION

1,1,2-Trichlorotrifluoroethane ($CCl_2F{-}CClF_2$, CFC-113), which has outstanding characteristics as a cleaning agent, has been used for many years in the microelectronics, precision machinery and dry cleaning industries. However, since the destruction of the ozone layer caused by the emission of chlorofluorocarbons (CFCs) has become a serious problem, the use of CFC-113 is becoming increasingly restricted, especially in the field of cleaning under open-system conditions.

Non-flon fluid, pentafluoropropyl alcohol (5FP), has been developed as a powerful replacement compound for $CCl_2F{-}CClF_2$, and is effective for the cleaning and/or drying of flax, machine oil or water. In the precision industrial sphere it is used as a solvent for ultrasonic cleaning, and therefore the ultrasonic speed in the fluid is an important property in analyzing the mechanism of the cleaning process. The authors have re-

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TABLE 1

Physical properties for each compound

	5FP	4FP	3FE	PrOH [b]	EtOH [b]
Chem. formula	$C_3F_5H_2OH$	$C_3F_4H_3OH$	$C_2F_3H_2OH$	C_3H_7OH	C_2H_5OH
Molecular weight	150.0	132.0	100.0	60.0	46.0
Boiling temp. (K)	353.8 [a]	382.1	347.2	370.3	351.4
Critical constants					
Temp. (K)	497.7	557.2	501.9	536.7	516.2
Press. (MPa)	5.23	4.85	5.11	5.17	6.38
Density (kg m^{-3})	523	495	432	275	276
At 298.15 K and 0.1 MPa:					
ρ (kg m^{-3})	1510 [a]	1482	1381	799.65 [c]	784.93 [c]
α (10^{-3} K^{-1})	1.29	0.83	1.41	0.995 [c]	1.093 [c]
n_D	1.287	1.319	1.292	1.3837	1.3594
η (Pa s)	3419 [a]	4872	1749	1722 [d]	1078
κ_S (GPa^{-1})	1.146	0.688	1.039	0.859	0.971
κ_T (GPa^{-1})	1.375	0.771	1.315	0.993	1.173
C_p (J mol^{-1} K^{-1})	214.4	214.2	155.8	143.8	114.1
$C_p - C_V$ (J mol^{-1} K^{-1})	35.8	23.1	32.6	20.7	16.3

Key: ρ = density; α = expansion coefficient; n_D = refractive index; η = viscosity. [a] Ref. 3 [b] Ref. 4 [c] Ref. 5 [d] 303.15 K.

ported previously [1,2] the ultrasonic speeds in liquid phases of hydrochlorofluorocarbons HCFC-123 and HCFC-123a under high pressures.

For alcohols containing fluorine atoms, the study of their thermodynamic properties is an interesting subject from the viewpoint of physical chemistry. This paper reports the results of experimental measurements of ultrasonic speed u in liquid 5FP, tetrafluoropropyl alcohol (4FP) and trifluoroethyl alcohol (3FE) under several conditions of temperature (T) and pressure (p). The temperature and pressure effects on u and related thermodynamic properties caused by the difference in molecular structure of these fluoro alcohols are discussed, together with the corresponding properties for primary alcohols.

EXPERIMENTAL

Research grade pentafluoropropyl alcohol ($CF_3CF_2CH_2OH$), trifluoropropyl alcohol ($CHF_2CF_2CH_2OH$) and trifluoroethyl alcohol (CF_3CH_3OH) were purified products from Daikin Industrials Ltd.; all had purities better than 99.8 mol%. These substances were used without further purification except that they were carefully dried with molecular sieve 5A supplied by Wako Pure Chemicals Co. The general physicochemical properties for these alcohols are listed in Table 1 together with those for *n*-propyl alcohol (C_3H_7OH, PrOH) and ethyl alcohol (C_2H_5OH, EtOH).

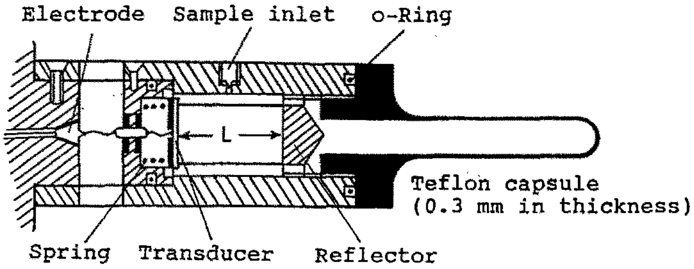

Fig. 1. Acoustic interferometer.

The method used to measure ultrasonic speed was a sing-round technique operated at a frequency of 2 MHz, similar to that outlined previously [6,7]. In this work, a fixed-path acoustic interferometer was newly constructed from stainless steel SUS 306, as illustrated in Fig. 1. The ultrasonic speed was obtained by measuring the transit time t of a short acoustic pulse travelling over a known distance L. The value of L was 31.234 mm at 298.15 K and 0.1 MPa, and its response to temperature and pressure changes was calibrated by measuring the period in pure toluene as reported by Muringer et al. [8]. The temperature, which was thermostatically controlled within ± 0.02 K, was measured by a quartz thermometer with resolution of ± 0.001 K. The temperature change in the sample caused by elevated pressure was monitored by a T-type thermocouple as the temperature difference in between a high pressure vessel and thermostat through a personal computer. The pressure generated by a manual oil pump was transmitted to the sample via a Teflon pressure capsule, and was observed by two precise pressure gauges (10 and 100 MPa maximum reading calibrated by a dead weight tested within ± 0.005 and ± 0.08 MPa, respectively. The probable uncertainty of the measured ultrasonic speed was better than $\pm 0.2\%$ overall under the conditions used.

RESULTS AND DISCUSSION

The experimental results for ultrasonic speed u (in m s^{-1}) at various temperatures T (in K) and pressures p (in MPa) in 5FP, 4FP and 3FE are listed in Table 2. For these alcohols, no experimental study on the thermodynamic properties, including the u value, has so far been reported. We therefore measured u in pure tetrachloromethane at various conditions of T and p, and confirmed the reliability of the apparatus by comparison with the values accurately measured by Bobic et al. [9].

The results thus obtained for each alcohol behaved smoothly in response to temperature and pressure changes, as illustrated in Fig. 2, and are well represented by the following polynomial equation

$$u = \sum_{i=0}^{2} \sum_{j=0}^{3} a_{ij}(T - 298.15)^i p^j \tag{1}$$

The values of the coefficients a_{ij}, calculated by least-squares analysis of all the experimental values are listed in Table 3 with the maximum and mean deviations from the above equation. The maximum deviations occurred chiefly in the vicinity of 0.1 MPa in the higher temperature region.

As can be seen in Fig. 2, among the compounds studied, the absolute values of u in 5FP are notably low, and the values increase in the order 5FP < 3FE < 4FP under the experimental conditions used. At 298.15 K, the temperature variation of u, $(\partial u/\partial T)_{p=0.1\text{ MPa}}$, for 5FP and 4FP had nearly the same value, around -2.85 m s^{-1} K^{-1}, which was higher than that for 3FE: 2.71 m s^{-1} K^{-1}. On the other hand, at elevated pressures, these fluoro alcohols show interesting behaviour of the u values, which differs markedly from that for $(\partial u/\partial T)_p$. That is, the u value for 5FP is found to be affected much more by pressure changes compared with those for the other two alcohols, and the value of $(\partial u/\partial p)_T$ decreases in the order 5FP > 3FE > 4FP.

As is well known, the thermodynamic properties for primary alcohols increase or decrease in general with an increasing carbon number in the molecule. For these fluoro alcohols the densities ρ, measured using a vibrating densimeter at atmospheric pressure, indicate a large absolute difference from those for primary alcohols, as shown in Fig. 3(a), but the measure in ρ for 5FP and 3FE corresponds to that for PrOH and EtOH, respectively.

According to the experimental studies on the ultrasonic speed in some primary alcohols reported by Wilson and Bradley [10] and by Sun et al. [11], the values of u also rise with increasing carbon number in alcohols, as shown in Fig. 2. Weissler [12] reported that the order for u was compatible with that for ρ for n-alcohols of C_2 to C_9. However, when the results presented here were compared with those for primary alcohols, the ultrasonic speeds differed greatly for the corresponding two substances. That is, the fluoro alcohols show a lower absolute value for u and for $(\partial u/\partial p)_T$ than do the n-alcohols. Interestingly, the order of u in 5FP and 3FE is found to be the reverse of that for PrOH and EtOH, although the order of ρ is 5FP > 3FE. Figure 2(b) shows the isentropic compressibility κ_S $[=(\rho u^2)^{-1}]$ at 0.1 MPa estimated from the experimental u and ρ values. These results for 5FP and 3FE also indicate the reverse order from those for primary alcohols. Moreover, these curves for u and κ_S vs. temperature for 4FP suggested that this alcohol has different molecular characteristics.

On the basis of the oscillator model of a liquid [13], the relation between the pressure effect coefficient of ultrasonic speed $1/u(\partial u/\partial p)_T$ and the isothermal compressibility κ_T is given by

$$\frac{1/u(\partial u/\partial p)_T}{\kappa_T} = C \tag{2}$$

where C is a constant with a very small, or zero, dependence on tempera-

TABLE 2

Experimental values of ultrasonic speed u (m s^{-1}) in fluorine-containing alcohols at various pressures p (MPa)

p	u	p	u	p	u	p	u
5FP							
283.15 K							
0.101	802.4	15.86	880.4	40.72	979.1	65.22	1060.0
3.280	819.4	20.86	902.0	45.63	996.6	70.22	1075.3
5.728	832.1	25.99	923.2	51.00	1014.6	75.06	1089.6
8.30	845.1	30.98	942.9	55.64	1029.9		
11.28	859.7	35.91	961.6	60.24	1044.7		
298.15 K							
0.101	760.0	20.12	862.4	40.46	945.3	60.58	1015.5
7.709	802.4	25.66	886.4	45.26	963.1	65.41	1030.9
11.61	822.2	30.44	906.3	50.19	980.6	70.24	1046.0
15.70	842.1	35.49	926.4	55.35	998.2	74.21	1058.1
303.15 K							
0.101	745.1	11.40	807.9	35.77	916.1	60.18	1003.7
2.655	760.3	15.26	827.1	41.05	936.4	65.13	1019.8
4.351	770.0	19.81	848.6	45.55	953.1	69.83	1034.6
6.350	781.2	25.38	873.4	50.61	971.1	75.00	1050.3
8.60	793.2	30.55	895.2	55.44	988.0		
313.15 K							
0.101	716.7	15.51	803.8	40.52	913.1	64.90	999.4
3.480	737.6	20.71	829.1	45.40	931.6	69.43	1013.9
6.081	753.0	25.60	851.5	49.86	948.1	75.17	1031.6
8.72	767.9	30.28	871.7	55.07	966.5		
10.71	778.8	35.32	892.5	60.23	984.2		
323.15 K							
0.101	688.7	21.40	809.0	46.22	914.9	70.49	999.5
3.723	712.2	26.01	830.6	50.73	931.8	75.29	1014.8
5.941	725.9	31.09	853.4	55.86	949.9		
11.84	759.7	36.00	874.1	60.68	967.0		
16.10	782.5	41.56	896.5	65.35	982.8		
333.15 K							
0.101	659.0	15.71	755.7	40.57	871.7	64.85	961.8
3.695	683.7	20.42	780.3	46.33	894.7	69.79	978.2
6.537	702.1	26.36	809.6	50.61	911.2	74.73	994.1
9.16	718.5	30.84	830.2	55.42	928.9		
11.52	732.4	36.08	853.0	60.74	947.7		
348.15 K							
0.101	616.3	16.11	722.3	41.03	843.6	65.47	937.3
4.077	645.9	21.18	750.0	45.86	863.8	70.56	954.4
7.123	666.8	25.79	773.9	50.77	883.2	75.57	971.1
8.88	678.4	31.22	800.1	55.72	901.9		
11.47	694.8	36.11	822.2	60.75	920.5		
4FP							
293.15 K							
0.101	1003.5	21.53	1083.8	45.53	1160.9	65.23	1217.2
5.678	1025.8	26.01	1099.4	50.56	1175.9	69.81	1229.6
10.92	1045.9	30.58	1114.5	55.23	1189.4	74.99	1243.1
16.06	1064.8	35.95	1131.5	60.42	1204.0		

TABLE 2 (continued)

p	*u*	*p*	*u*	*p*	*u*	*p*	*u*
4FP							
298.15 K							
0.101	990.4	19.29	1064.4	45.65	1150.9	69.97	1219.7
5.936	1014.1	23.92	1080.7	50.81	1166.1	75.05	1233.2
7.010	1018.6	29.77	1100.4	55.99	1181.2		
10.225	1031.0	34.13	1114.9	61.33	1196.2		
14.26	1046.3	41.60	1138.4	65.72	1208.0		
303.15 K							
0.101	974.9	21.48	1058.5	41.49	1125.4	60.87	1182.9
4.560	993.7	26.60	1076.4	46.68	1141.2	65.91	1196.6
10.65	1018.2	31.22	1091.9	51.72	1156.3	70.74	1210.0
15.64	1037.2	36.30	1108.8	56.00	1169.0	74.72	1221.0
313.15 K							
0.101	945.5	20.68	1029.5	45.41	1114.0	69.86	1185.9
3.976	962.6	25.90	1048.6	50.71	1130.3	72.92	1194.4
6.716	974.3	31.16	1067.0	55.36	1144.4		
10.08	988.2	36.16	1084.0	59.91	1157.8		
14.75	1006.8	40.85	1099.4	64.69	1171.5		
323.15 K							
0.101	918.8	15.77	984.8	40.56	1075.5	70.88	1168.0
0.602	924.9	21.11	1005.6	45.23	1090.9	75.24	1180.3
4.411	936.2	25.29	1021.6	50.47	1107.6		
7.408	949.5	31.54	1044.4	55.92	1124.4		
10.58	963.3	35.77	1059.2	65.25	1152.0		
333.15 K							
0.101	886.7	21.23	980.5	45.27	1068.0	69.89	1144.3
6.127	915.7	26.11	999.5	50.74	1085.8	75.06	1159.1
8.53	926.8	31.60	1019.9	55.47	1100.9		
11.71	940.8	36.04	1036.0	60.68	1116.9		
16.36	960.6	40.88	1053.0	64.97	1129.9		
348.15 K							
0.101	841.6	20.30	937.5	46.10	1036.1	70.12	1113.1
5.115	867.7	25.75	960.1	51.10	1053.1	75.39	1128.6
8.86	886.3	30.80	980.2	55.90	1068.9		
13.29	906.7	36.39	1001.3	60.88	1084.9		
14.97	914.3	41.37	1019.5	65.36	1098.8		
3FE							
293.15 K							
0.101	848.0	15.58	920.7	40.80	1017.7	64.57	1094.2
3.739	866.2	20.04	939.4	45.06	1032.3	69.30	1108.2
6.220	878.2	24.71	958.2	49.81	1048.0	74.66	1123.6
8.55	889.2	30.14	979.1	54.90	1064.0		
10.97	900.3	35.35	998.3	60.88	1082.1		
298.15 K							
0.101	834.8	15.54	909.1	40.58	1006.9	65.17	1086.6
3.673	853.3	20.51	930.1	44.55	1020.7	69.95	1100.8
6.476	867.2	25.84	951.7	50.25	1039.8	74.47	1113.8
8.42	876.6	30.66	970.4	55.01	1055.1		
11.22	889.7	35.38	988.1	60.04	1070.9		

TABLE 2 (continued)

p	u	p	u	p	u	p	u
3FE							
303.15 K							
0.101	821.0	15.77	897.5	44.94	1011.0	70.99	1094.2
3.570	839.1	20.98	920.0	50.60	1030.5	75.29	1106.9
6.335	853.2	25.86	940.2	55.60	1046.8		
8.68	864.7	31.02	960.3	60.06	1061.1		
11.07	876.1	35.87	978.5	65.19	1076.9		
313.15 K							
0.101	794.0	16.36	876.6	40.86	976.5	64.95	1057.9
3.408	812.4	20.93	897.4	45.17	992.3	69.49	1071.8
6.294	827.8	25.75	918.0	50.41	1010.4	74.61	1087.1
8.77	840.5	31.08	939.4	54.94	1025.8		
11.38	853.4	35.39	956.4	60.34	1043.3		
323.15 K							
0.101	766.6	15.31	848.6	40.70	955.8	64.99	1039.6
3.746	787.9	20.84	874.4	45.18	972.3	69.73	1054.4
6.316	802.2	25.81	896.2	50.30	990.7	74.98	1070.3
8.46	813.7	30.73	916.8	55.56	1008.6		
11.29	818.3	35.35	935.2	60.05	1023.7		
333.15 K							
0.101	740.3	16.02	828.7	40.52	935.4	65.04	1022.2
3.068	757.9	20.97	852.5	45.47	954.4	69.77	1037.2
5.652	773.0	25.67	874.1	50.93	974.2	74.83	1053.0
8.12	787.2	31.00	897.1	55.61	990.7		
10.85	802.0	35.95	917.4	60.44	1007.0		
348.15 K							
0.101	697.9	15.54	790.8	40.65	905.8	64.84	994.6
3.582	720.5	21.03	819.0	44.95	923.1	69.71	1010.6
6.565	739.5	26.20	843.6	50.67	944.7	74.97	1027.6
8.58	751.7	30.62	863.6	54.99	960.4		
11.43	768.2	35.65	885.3	60.20	978.8		

ture and pressure. For some organic liquids, C has a value of ≈ 4 [14]. To measure the above relation for fluoro alcohols showing marked differences in their u values, we estimated the C value. At first, the values of κ_T required in this calculation was determined thermodynamically by the following equation

$$\kappa_T = \kappa_S + \frac{T\alpha^2}{\rho C_p} \tag{3}$$

where ρ and α are the density and the thermal expansion coefficient, respectively. For these substances, the values of C_p were also estimated by the Missenard group contribution method [15]. The comparison of C_p obtained: 142.0 and 113.8 J mol^{-1} K^{-1} for PrOH and EtOH respectively at 298.15 K and 0.1 MPa, seems reasonable with the data obtained experi-

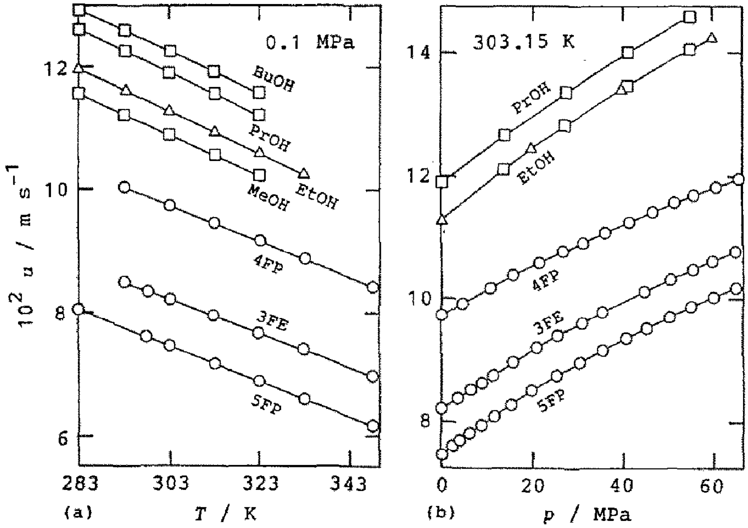

Fig. 2. Temperature T (a) and pressure p (b) dependences of ultrasonic speeds u for fluoro alcohols and n-alcohols. (○) This work; (□) ref. 10; (△) ref. 11.

mentally elsewhere [4]. Using the estimated C_p values, the κ_T for fluoro alcohols were computed by eqn. (3) and the relation of eqn. (2) is plotted in Fig. 4. Unexpectedly, the results for all alcohols, including benzyl alcohol and some primary alcohols, are found to be fitted well by a straight line

TABLE 3

Coefficients a_{ij} of eqn. (1) and maximum (δ_{max}) and mean (δ_{mean}) deviations from experimental values

i	j			
	0	1	2	3
5FP				
0	7.59661×10^{2}	5.71223×10^{0}	-3.34721×10^{-2}	1.43846×10^{-4}
1	-2.85116×10^{0}	2.67412×10^{-2}	-3.04082×10^{-4}	1.50243×10^{-6}
2	-1.20551×10^{-4}	1.05157×10^{-4}	-1.86142×10^{-6}	1.15871×10^{-8}
	$\delta_{max}=0.11\%$	$\delta_{mean}=0.044\%$		
4FP				
0	9.89310×10^{2}	4.15479×10^{0}	-1.65328×10^{-2}	5.84684×10^{-5}
1	-2.90499×10^{0}	1.91624×10^{-2}	-1.93860×10^{-4}	9.89190×10^{-7}
2	-9.99962×10^{-4}	4.43118×10^{-5}	-6.95964×10^{-8}	-2.38330×10^{-9}
	$\delta_{max}=0.09\%$	$\delta_{mean}=0.031\%$		
3FE				
0	8.34424×10^{2}	5.16073×10^{0}	-2.70147×10^{-2}	1.09092×10^{-4}
1	-2.68376×10^{0}	2.35598×10^{-2}	-2.47181×10^{-4}	1.15034×10^{-6}
2	-9.29820×10^{-4}	1.00389×10^{-4}	-1.83644×10^{-6}	1.24959×10^{-8}
	$\delta_{max}=0.10\%$	$\delta_{mean}=0.030\%$		

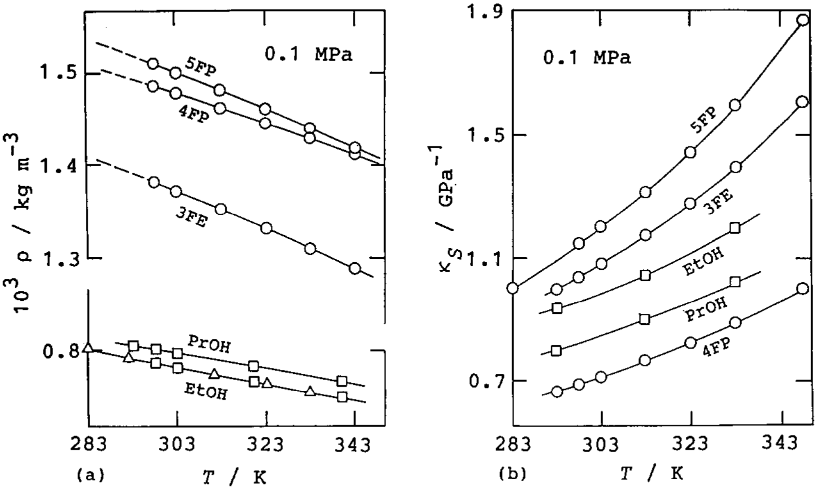

Fig. 3. Temperature dependences of density (a) and isentropic compressibility κ_S (b) for fluoro alcohols and *n*-alcohols. (o) This work; (□) ref. 5 for ρ determined from ρ in ref. 5 and u in ref. 10 for κ_S; (△) ref. 11.

passing through the origin, and the line has a slope of 5 ± 0.2 as the C value, except that for MeOH. This slope differs clearly from that of 4 ± 0.15 for other hydrocarbon liquids, as discussed elsewhere. This distinction between the slopes suggests that the intermolecular force of alcohol molecules is stronger than that for other organic substances, because the C value corresponds to the sum of the exponential terms, $n + m$ $(= 3C - 6)$, of the Mie potential equation [13]. This larger C value for alcohol compounds may derive from the association due to hydrogen bonding.

We have reported in previous work that the ultrasonic speeds in tetramethylsilane [18] and some fluorocarbon refrigerants [2,7] usually had values

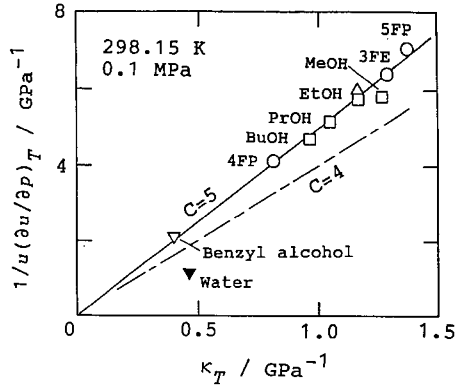

Fig. 4. Relation between pressure effect coefficient $1/u(\partial u/\partial p)_T$ and isothermal compressibility κ_T (———) For alcohols; (- — - — -) for other hydrocarbon liquids; (o) this work; (□) ref. 10; (△) ref. 11; (▽) ref. 16; (▼) ref. 17.

lower than those for other hydrocarbon substances. This phenomenon can be interpreted qualitatively by the differences in molecular structure based on the Eyring liquid free volume model [19]. That is, when the value for the intermolecular free volume V_f in a liquid is large, the speed has a low value in general. From this fact, a fluoro alcohol, which includes a larger van der Waals radius for the fluorine atom compared with that for the hydrogen atom [19], is presumed structurally to have a V_f larger than that for a primary alcohol, and therefore the speed should decrease with an increasing number of fluorine atoms as shown experimentally in this work. In the case of non-polar and weakly polar substances, it is confirmed for some liquids that the u value in fluids having a large free volume is influenced greatly by pressure changes.

Fluorine is the most electronegative of all the elements. A strengthening of the electronegativity for fluoro alcohols leads to an increase in the intermolecular force due to hydrogen bonding. Although fluoro alcohols are predicted to have a large V_f, the u values are largely unaffected by elevated pressure. This fact should result from a strong intermolecular interaction. This phenomenon is also supported thermodynamically in that the difference between the molar heat capacities at constant pressure and constant volume ($C_p - C_V$), which is a measure of intermolecular force, for fluoro alcohols has a large value compared with that for primary alcohols, as listed in Table 1. Among the present compounds, 4FP shows small values for $(\partial u/\partial p)_T$, κ_S and $C_p - C_V$, and is considered to have different intermolecular interaction behaviour from that for 5FP and 3FE.

CONCLUSION

The ultrasonic speed u in 5FP, which is noted as a safe alternative compound to $CCl_2F{-}CClF_2$, and in 4FP and 3FE has been observed at several conditions of T and p. The results for these fluoro alcohols displayed lower values than those for primary alcohols. At 300 K and 0.1 MPa, the difference of u between 5FP (743.6 m s^{-1}) and $CCl_2F{-}CClF_2$ (630.9 m s^{-1} [20]) is slight.

Ultrasonic cleaning is a process involving the effects of both agitation and cavitation. Therefore the low u value observed for 5FP corresponding to a short wavelength makes it useful as a cleaning agent, because fine agitation and cavitation can be achieved. 4FP and 3FE are also useful as cleaning agents, but their industrial use is precluded because these compounds are inflammable.

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