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Excess molar enthalpies of binary mixtures of 2-bromo-2-chloro-1,1,1-trifluoroethane (halothane) with oxygenated and hydrocarbon solvents

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Abstract

Excess molar enthalpies, H^E, were determined at 298.15 K for the following eighteen mixtures: 2-bromo-2-chloro-1,1,1-trifluoroethane (halothane) + acetone, + dipropyl ether, + diisopropyl ether, + methyl *tert*-butyl ether, + ethylene glycol dimethyl ether or monoglyme, + diethylene glycol dimethyl ether or diglyme, + tetraethylene glycol dimethyl ether or tetraglyme, + methyl phenyl ether, + tetrahydrofuran, + 1,4-dioxane, + methyl acetate, + benzene, + cyclopentane, + cyclohexane, + *n*-hexane, + *n*-heptane, + 2,2-dimethylbutane and + 2,4-dimethylpentane. The value of H^E for all oxygenated solvents is strongly negative whereas for the equal-structure hydrocarbon solvents (taken as homomorphs) it is positive. This is a clear indication of the formation of a halothane–oxygenated-compound complex. The complexation is through a H-bond between the oxygen atom in the solvent molecules and the hydrogen atom in halothane. As the number of oxygen atoms in the solvent increases, H^E becomes more negative and asymmetrical, its minimum being displaced towards higher halothane concentrations. The H^E values for halothane mixed with the glymes are amongst the largest ever found for mixtures of non-electrolytes. The increase in asymmetry in going from monoglyme to tetraglyme is indicative of the presence of multisolvated species in the solution. A clear correlation between the solvent Kamlet–Taft β parameter (H-bonding acceptor ability) and H^E was found. A comparison between the H^E vs. β correlation for the present data and that for chloroform showed that for a given oxygenated solvent H^E with halothane is always more negative than with chloroform. This indicates that the acidity of the hydrogen atom in halothane is greater than that in chloroform.

INTRODUCTION

The family of compounds known as Freons can be divided into two broad groups depending on whether or not the molecule is fully

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halogenated. For Freons which are fully halogenated their molecular surface has an electron acceptor character owing to the strong electronegativity of the halogen atoms. Freons of this kind, e.g. Freon 113, can form complexes with electron donors such as oxygenated compounds via a charge transfer mechanism [1–5]. However, Freons which contain one or more hydrogen atoms in their chemical structures are particularly interesting because their molecular surface is constituted of two sections with different degrees of electron affinity, corresponding to the halogen and hydrogen atoms. Here the hydrogen atoms have been considered (using the Lewis acid–base concept) as electron acceptors. An alternative treatment is that in which the Freon molecule is taken as a proton donor (i.e. the Brønsted acid–base concept is used). The special molecular surface characteristics of these Freons, present in 2-bromo-2-chloro-1,1,1-trifluoroethane (commonly known as halothane), prompted us to study the thermodynamic behaviour of its mixtures with oxygen-containing solvents and, for comparison purposes, with several hydrocarbons whose chemical structures are identical to those of the oxygenated compounds. These thermodynamic studies include measurements of molar excess heat capacities (C_p^E) and molar excess volumes (V^E) [6] and of vapour–liquid equilibria (VLE) [7]. In this work we present and qualitatively discuss molar excess enthalpies (H^E) for a large number of binary mixtures of halothane with oxygenated or hydrocarbon solvents. The quantitative analysis through several association models of all the thermodynamic data accumulated (VLE, H^E and C_p^E) for several of these mixtures is presented elsewhere [8].

EXPERIMENTAL

Halothane (Spofa and Aldrich Chem. Co. stabilized with 0.01 mass.% thymol.) was distilled to separate the stabilizing agent. After distillation, no impurity was found through a gas chromatography analysis. All other materials were obtained from Aldrich Chem. Co. or Merck Chem. Co. with stated purities of 99–99.99 mol.%. These chemicals were used without further purification; however, before each experiment, some liquids were kept over molecular sieves and all liquids were partially degassed.

Excess molar enthalpies, H^E, were determined at 298.15 K using a Picker flow microcalorimeter (Sodev Inc., Sherbrooke, Canada) for the following eighteen mixtures: 2-bromo-2-chloro-1,1,1-trifluoroethane (halothane) +acetone (ACT), +dipropyl ether (DPE), +diisopropyl ether (DIPE), +methyl *tert*-butyl ether (MTBE), +ethylene glycol dimethyl ether or monoglyme (G1), +diethylene glycol dimethyl ether or diglyme (G2), +tetraethylene glycol dimethyl ether or tetraglyme (G4), +methyl phenyl ether (MPE), +tetrahydrofuran (THF), +1,4-dioxane (DIOX),

+methyl acetate (MAC), +benzene (BENZ), +cyclopentane (cC5), +cyclohexane (cC6), +*n*-hexane (nC6), +*n*-heptane (nC7), +2,2-dimethylbutane (22DMB) and +2,4-dimethylpentane (24DMP). The instrument was operated in the discontinuous mode. For each mixture we started the collection of results at a volume-flow ratio of 0.5, decreasing it to its lower bound then increasing it gradually to its upper bound, finally returning to the starting point. The performance of the calorimeter was checked by measuring H^E of two test mixtures: chloroform + acetone and diethylene glycol dimethyl ether + *n*-heptane. Agreement with reliable literature values [9–10] was on average within 1%. For the mixtures halothane +cC5, +MPE, +G1 and +G2 the H^E values reported here were obtained assuming that VE = 0; we estimate that the effect of this approximation is well within the accuracy of our calorimetric measurements (2%).

RESULTS AND DISCUSSION

The experimental values of the excess molar enthalpies H^E are given in Table 1. For each mixture, the results were correlated with a Redlich–Kister equation

$$H^E\ (\mathrm{J\ mol^{-1}}) = x_1(1 - x_1)\sum_{i=0}^{p} A_i(2x_1 - 1)^i \qquad (1)$$

whose parameters A_i, along with the standard deviations $s(H^E)$, are given in Table 2. These parameters were evaluated by the least-squares method with equal weighing for all values. For each system the number of parameters p in eqn. (1) was chosen on the basis of a statistical criterion (F-test) and the analysis of the statistical behaviour of the residuals [11]. Comparison with previously reported H^E measurements is only possible for the mixtures halothane +DIOX, +THF and +ACT [12]; this comparison shows that the data are in agreement within the reported experimental errors.

The molar excess enthalpies, plotted against the mole fraction of halothane (component 1), for a series of saturated hydrocarbons are shown in Fig. 1. Here, the H^E values are positive, ranging from 600 to 850 J mol^{-1}. In contrast, Fig. 2 shows that the H^E values for a series of ethers are strongly negative, their values ranging from −1000 to −3800 J mol^{-1}. These large negative H^E values are strong evidence in favour of the existence of a halothane–ether complex in solution. In Figs. 1 and 2 there are four hydrocarbon–ether pairs whose molecules have the same chemical structure (cC5–THF, nC7–DPE, 22DMB–MTBE and 24DMP–DIPE). These hydrocarbons can be taken as the ethers' homomorphs, their H^E values representing the so-called "physical contribution" to H^E arising from effects other than association. The comparison between

TABLE 1

Experimental values of the excess molar enthalpy H^E at 298.15 K [a]

x_1	H^E (J mol^{-1})	x_1	H^E (J mol^{-1})	x_1	H^E (J mol^{-1})
Halothane(1) + ACT(2)					
0.0735	−588	0.3162	−2056	0.5661	−2717
0.1107	−770	0.3617	−2312	0.6226	−2626
0.1492	−1093	0.4088	−2457	0.6226	−2634
0.1888	−1309	0.4088	−2454	0.6814	−2469
0.2299	−1618	0.4593	−2615	0.7473	−2128
0.2299	−1594	0.5116	−2701	0.8161	−1709
0.2724	−1840	0.5116	−2665	0.8881	−1124
Halothane(1) + DPE(2)					
0.1881	−1275	0.5133	−2285	0.7083	−1754
0.2460	−1616	0.5627	−2222	0.7543	−1510
0.3022	−1874	0.5627	−2233	0.7992	−1251
0.3572	−2093	0.5627	−2249	0.7992	−1235
0.4106	−2217	0.6125	−2117	0.8462	−966
0.4626	−2290	0.6125	−2107	0.8920	−667
0.4626	−2279	0.6609	−1963	0.9366	−364
Halothane(1) + DIPE(2)					
0.1322	−1486	0.5211	−3235	0.7601	−2057
0.2518	−2418	0.5704	−3166	0.8042	−1665
0.3089	−2738	0.6199	−2969	0.8503	−1282
0.3644	−3048	0.6679	−2746	0.8949	−863
0.4181	−3197	0.7147	−2389	0.9384	−470
0.4704	−3308				
Halothane(1) + MTBE(2)					
0.0574	−636	0.3775	−3423	0.6292	−3384
0.1139	−1121	0.4284	−3645	0.6788	−3064
0.2212	−2136	0.4284	−3692	0.7760	−2281
0.2738	−2713	0.4786	−3773	0.8273	−1800
0.3260	−3117	0.5284	−3800	0.8779	−1311
0.3260	−3116	0.5791	−3688	0.9279	−741
Halothane(1) + G1(2)					
0.0504	−682	0.4446	−4908	0.7513	−3980
0.1007	−1336	0.4942	−5162	0.8069	−3252
0.1985	−2506	0.5454	−5297	0.8069	−3204
0.2966	−3745	0.6482	−4931	0.8624	−2300
0.3458	−4161	0.6997	−4603	0.9181	−1428
0.3952	−4667				
Halothane(1) + G2(2)					
0.0676	−788	0.4195	−4424	0.7158	−4922
0.1328	−1517	0.4195	−4435	0.7611	−4483
0.1938	−2132	0.4718	−4949	0.8051	−3884
0.2529	−2754	0.5225	−5134	0.8510	−3150
0.3101	−3392	0.5718	−5425	0.8955	−2331
0.3657	−3942	0.6212	−5426	0.9388	−1451
0.3657	−3943				

TABLE 1 (continued)

x_1	H^E (J mol^{-1})	x_1	H^E (J mol^{-1})	x_1	H^E (J mol^{-1})
Halothane(1) + G4(2)					
0.1008	−1066	0.5799	−5746	0.7576	−5653
0.1914	−1978	0.6284	−5931	0.7956	−5248
0.2708	−2841	0.6736	−6038	0.8311	−4615
0.4098	−4337	0.6736	−6047	0.8645	−3941
0.4711	−4962	0.6736	−6060	0.8982	−3065
0.5276	−5374	0.7170	−5964	0.9298	−2231
Halothane(1) + MPE(2)					
0.1049	−239.9	0.4060	−890.1	0.7090	−359.0
0.1553	−343.7	0.4557	−948.0	0.7596	−858.9
0.2057	−464.3	0.5053	−1013.0	0.8137	−740.4
0.2558	−571.0	0.5564	−1045.1	0.8676	−554.7
0.3060	−699.2	0.6074	−1065.3	0.9214	−359.0
0.3560	−787.4	0.6584	−1018.9		
Halothane(1) + THF(2)					
0.0806	−784	0.2925	−2492	0.5364	−3324
0.0806	−771	0.3382	−2826	0.5904	−3215
0.1209	−1137	0.3850	−3020	0.6457	−2917
0.1622	−1487	0.3850	−3054	0.7027	−2586
0.1622	−1450	0.4331	−3221	0.7657	−2145
0.2045	−1842	0.4331	−3235	0.8306	−1624
0.2480	−2221	0.4841	−3261	0.8977	−985
Halothane(1) + DIOX(2)					
0.0417	−302	0.3489	−2018	0.6018	−2692
0.0841	−529	0.3963	−2216	0.6018	−2670
0.1260	−780	0.4447	−2419	0.6565	−2589
0.1688	−978	0.4447	−2405	0.7124	−2446
0.1688	−1030	0.4959	−2550	0.7740	−2126
0.2123	−1240	0.5482	−2656	0.8371	−1751
0.2570	−1516	0.5482	−2634	0.9019	−1174
0.3024	−1757				
Halothane(1) + MAC(2)					
0.0389	−314	0.3326	−1891	0.6399	−2275
0.0787	−519	0.3790	−2083	0.6973	−2070
0.1182	−770	0.4269	−2236	0.6973	−2052
0.1588	−988	0.4778	−2338	0.7611	−1765
0.2005	−1239	0.5301	−2396	0.8270	−1377
0.2433	−1460	0.5842	−2384	0.8953	−861
0.2873	−1672				
Halothane(1) + BENZ(2)					
0.0874	−133.0	0.3585	−551.1	0.6118	−614.6
0.1307	−204.5	0.4064	−579.4	0.6659	−585.4
0.1748	−285.9	0.5064	−629.1	0.7813	−441.5
0.2651	−415.5	0.5586	−626.5	0.9194	−210.6

TABLE 1 (continued)

x_1	H^E (J mol^{-1})	x_1	H^E (J mol^{-1})	x_1	H^E (J mol^{-1})
Halothane(1) + *cC*5(2)					
0.0922	243.8	0.3719	609.2	0.6254	571.3
0.1374	341.5	0.4204	633.0	0.6786	527.0
0.1833	400.6	0.4695	640.1	0.7325	456.6
0.2295	481.1	0.4695	633.2	0.7910	380.5
0.2765	538.5	0.5209	626.7	0.8503	280.4
0.3239	576.7	0.5728	613.4	0.9104	168.4
Halothane(1) + cC6(2)					
0.1044	349.9	0.4047	843.9	0.6572	741.6
0.1546	451.1	0.4544	850.1	0.7080	680.7
0.2049	569.4	0.5552	834.6	0.8130	487.6
0.3050	734.5	0.6062	791.4	0.9211	226.9
Halothane(1) + nC6(2)					
0.1236	324.9	0.3996	724.4	0.6988	640.9
0.1236	315.4	0.4514	742.7	0.7458	573.2
0.1812	418.9	0.5019	748.1	0.7918	485.9
0.2376	517.2	0.5515	747.9	0.8402	399.6
0.2927	608.5	0.5515	738.9	0.8875	284.3
0.3468	665.0	0.6016	727.7	0.9339	167.7
0.3468	664.0	0.6507	693.7		
Halothane(1) + nC7(2)					
0.1365	367.7	0.5304	825.3	0.7222	684.1
0.1987	468.2	0.5795	815.6	0.7668	615.3
0.2589	585.8	0.5795	808.3	0.8549	425.3
0.3730	740.5	0.6286	789.4	0.9406	198.1
0.4797	818.2	0.6761	740.5		
Halothane(1) + 22DMB(2)					
0.1254	272.4	0.5059	639.2	0.7021	535.4
0.1836	373.3	0.5555	633.5	0.7488	479.2
0.2406	457.4	0.6055	613.6	0.8424	337.1
0.3504	574.0	0.6543	580.3	0.9348	160.9
0.4553	633.8				
Halothane(1) + 24DMP(2)					
0.2014	383.9	0.4839	679.3	0.6798	621.0
0.2621	489.6	0.5345	687.7	0.7255	569.5
0.3770	624.9	0.5836	679.3	0.7697	508.6
0.4313	662.0	0.6325	654.6	0.8570	329.6

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[a] For abbreviations of compounds used, see text.

TABLE 2

Parameters A_i and standard deviations $s(H^E)$ for representation of excess molar enthalpies at 298.15 K by eqn. (1) [a]

Halothane(1)+	A_0	A_1	A_2	A_3	A_4	$s(H^E)$ (J mol^{-1})
ACT	−10698	−2509	1664	1006		22
DPE	−9185	526	3028			9.4
DIPE	−13129	782	4648	2678	−2239	21
MTBE	−15204	−113	9247	−477	−5500	35
G1	−20759	−5650	6381	4672		42
G2	−20261	−11825	2487	6845		41
G4	−20676	−17749	−8509	5533	8419	39
MPE	−4030	−1900	559	617		8.6
THF	−13272	−380	6345	222	−3610	26
DIOX	−10294	−4521	1988	875	−2724	17
MAC	−9512	−2149	3283	1501	−2272	14
BENZ	−2511.2	−348.1	473.0	−402.4		7.7
cC5	2536.4	−289.7	−81.3	−298.0		5.1
cC6	3390.7	−258.0				9.2
nC6	3009.0	201.6	−210.1	−383.4		6.7
nC7	3264.6	359.8				8.4
22DMB	2555.2	53.1	−77.1			2.5
24DMP	2756.6	344.6	−399.9			6.7

[a] For abbreviations of second components, see text.

these two sets of H^E values shows that the substitution of a methylene group in the hydrocarbon solvent by an oxygen atom displaces H^E from being positive to highly negative. It is clear then that there is a specific interaction between halothane and the oxygen atoms giving rise to a halothane–ether complex.

When an oxygenated solvent is brought into contact with a Freon, complexation can occur via charge transfer mechanism or via the formation of a H-bond, depending on whether the Freon molecule is fully halogenated or not. An example of the first case is given by 1,1,2-trichlorotrifluoroethane (Freon 113) where the complexation Freon–oxygenated-solvent occurs between the electron donor oxygen atoms and the electron acceptor molecular surface of Freon 113 [1–5]. For single-hydrogen-containing Freons, such as halothane, the strong electronegativity of the halogen atoms gives a highly acidic character to the hydrogen atom. The presence of this readily available proton implies that for the oxygen atoms in the solvent molecules it is energetically more favourable to capture this proton than to share its electrons with the halogen atoms. Hence, for halothane + oxygenated-solvents, the complexation is through an H-bond between the oxygen atom in the solvent and the hydrogen atom

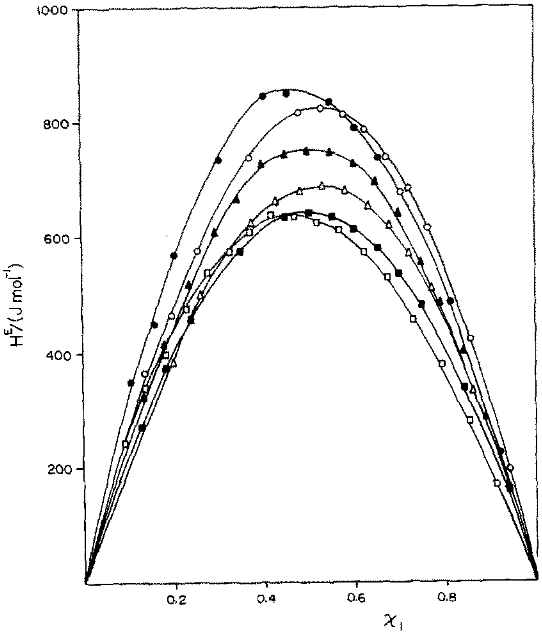

Fig. 1. Excess molar enthalpies at 298.15 K for halothane (component 1) mixed with cC6 (●), nC7 (○), nC6 (▲), 24DMP (△), 22DMB (■) and cC5 (□) (for abbreviations see text).

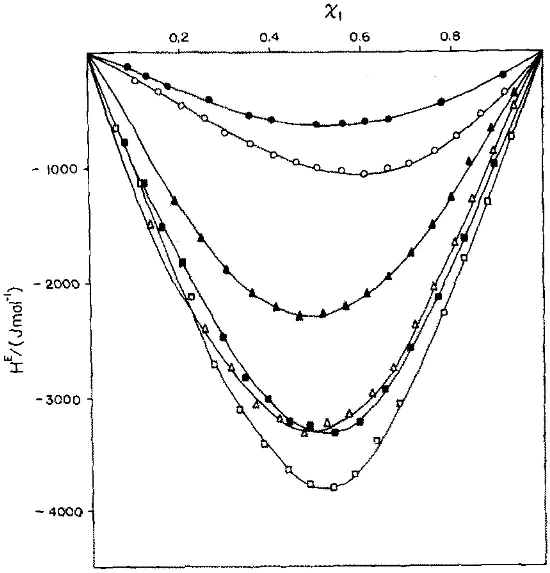

Fig. 2. Excess molar enthalpies at 298.15 K for halothane (component 1) mixed with BENZ (●), MPE (○), DPE (▲), DIPE (△), THF (■) and MTBE (□) (for abbreviations see text).

in halothane. In this context, it is interesting to note that the negative H^E value for halothane + BENZ in Fig. 2 is consistent with the well established electron donor character of benzene [13]. When benzene is substituted by MPE i.e. by a solvent which has not only an aromatic ring but also an oxygen atom, H^E becomes more negative and skewed to high halothane concentration (Fig. 2), indicating that the halothane–MPE interaction is probably of a multisolvation dual character, i.e. composed of an H-bond with the oxygen atom in MPE and of a charge transfer complex with the aromatic ring.

Figure 3 shows H^E for halothane mixed with MA, ACT, DIOX, G1, G2 and G4. Here, the marked asymmetry of the H^E curves implies that with these solvents multisolvation (via H-bonding) is the predominant

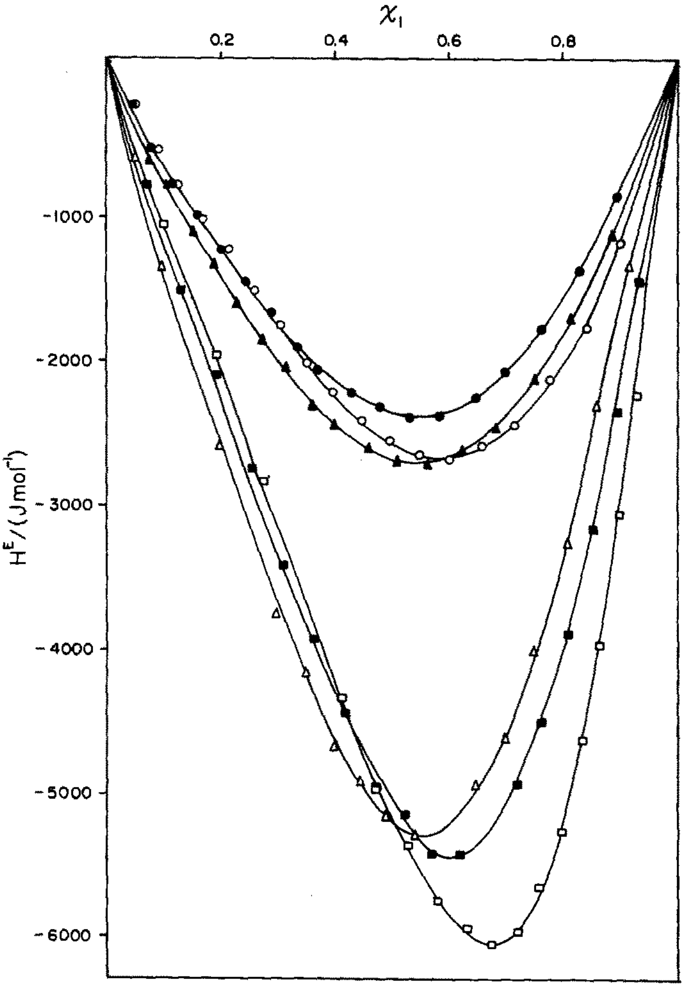

Fig. 3. Excess molar enthalpies at 298.15 K for halothane (component 1) mixed with MAC (●), DIOX (○), ACT (▲), G1 (△), G2 (■) and G4 (□) (for abbreviations see text).

feature. The application of several association models [8] to the data available for ACT and MAC (VLE, H^E and C_p^E) indicated that both solvents form two H-bonds with halothane. Comparison between the H^E results for DIOX in Fig. 3 and those for its homomorph cC6 in Fig. 1 shows again that the introduction of oxygen atoms has a marked effect on the magnitude of H^E, which changes from being positive and symmetrical for cC6 to strongly negative and asymmetrical for DIOX. The asymmetry here is clearly caused by the presence of two oxygen atoms in DIOX, each of which is capable of H-bonding with a halothane molecule. The presence of more than one oxygen atom in the solvent molecule has a dramatic effect on the magnitude of H^E, as illustrated by the series of linear ethers DPE, G1, G2 and G4. When the single oxygen ether DPE in Fig. 2 is substituted by the two-oxygen-containing G1, Fig. 3 indicates that H^E doubles its magnitude and becomes asymmetrical. The extremely negative H^E values for halothane + G1, +G2 and +G4 in Fig. 3 are amongst the largest ever found for mixtures of non-electrolytes [14]. In going from G1 to G2 and to G4, Fig. 3 shows that the introduction of a third and fifth oxygen atom in the solvent molecules increases the magnitude of H^E and makes the H^E curves even more asymmetrical, displacing their minima to higher halothane concentrations. The further skewedness of the H^E curve for G2 compared to G1, and for G4, compared to G2, is due to the presence of higher multisolvated species in solution. Evidence in favour of the presence of progressively higher species in the G2 and G4 mixtures compared to the G1 mixture is given by the partial molar enthalpies at infinite dilution obtained from the Redlich-Kister constants in Table 2: G1, $\bar{H}_2^{E,\infty} = -15.4 \pm 0.6\ \mathrm{kJ\ mol^{-1}}$; G2, $\bar{H}_2^{E,\infty} = -25.9 \pm 1.1\ \mathrm{kJ\ mol^{-1}}$; G4, $\bar{H}_2^{E,\infty} = -33.0 \pm 0.8$; these values indicate a higher degree of interaction for halothane–G4 contacts. It is interesting to note that, according to the H^E data here and in ref. 12, when a cyclic ether containing only one oxygen atom is replaced by a cyclic ether with two oxygen atoms, the H^E value with halothane becomes less negative, a trend which is opposite to that displayed by the linear ethers. It is possible that the measurement of more thermodynamic data (C_p^E and VLE) for halothane mixed with cyclic and linear ethers might be very helpful in understanding the behaviour of both kinds of ethers.

The degree of acidity of the hydrogen atom in halothane compared to other acidic-type hydrogen atoms, for example the hydrogen atom in chloroform, can be tested using H^E data and the solvent Kamlet–Taft β parameter [13], which is a measure of its H-bonding acceptor ability. In Fig. 4 equimolar H^E values for halothane and chloroform mixed with several solvents are plotted against the solvent β parameter value. In Fig. 4, H^E data for halothane mixed with an amide (dimethyl formamide, DMF) and a nitrile (acetonitrile, ACN) [15] have been added to the data for oxygenated solvents reported here, whereas for chloroform H^E data

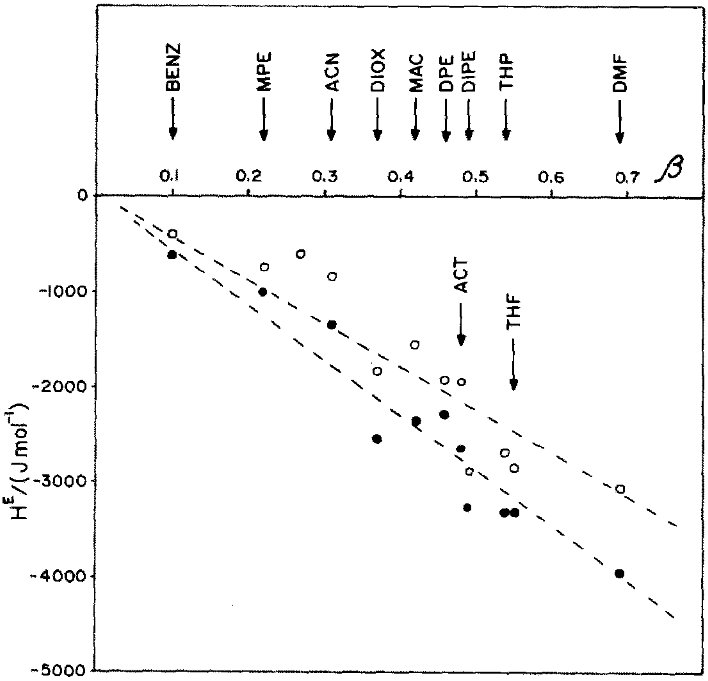

Fig. 4. Equimolar excess enthalpies at 298.15 K vs. the solvent Kamlet-Taft β parameter for halothane (●) and chloroform (○) mixed with the solvents indicated. All H^E data for halothane are from this work (Tables 1 and 2) with the exceptions of ACN and DMF [15] and tetrahydropyran (THP) [12]. The H^E data for chloroform were taken from: BENZ, [16]; MPE, [17] (at 276.15 K); ACN, [18]; DIOX, [19]; MAC, [20] (at 308.15 K); DPE, [21]; ACT, [22]; DIPE, [21]; THP, [23]; THF, [24] and DMF, [15].

have been taken from the literature. Figure 4 indicates that for a wide range of β values (in ref. 13 β changes from 0 to 1 for a very large number of compounds), for both halothane and chloroform there is a clear correlation between H^E and the basicity of the solvents expressed by their β parameters: as the H-bonding acceptor ability of the solvent increases H^E becomes more negative. The comparison between halothane and chloroform H^E vs. β correlations shows that for all solvents H^E for the chloroform mixture is less negative than for the corresponding halothane mixture, indicating that the acidity of the hydrogen atom in halothane is greater than that in chloroform. Unfortunately, owing to the lack of β values for MTBE, G1, G2 and G4, these solvents are not included in Fig. 4. However, their H^E values are in agreement with the trend seen in Fig. 4: the equimolar H^E (in J mol^{-1}) for halothane and chloroform respectively [15] are: −3800 and −2720 for MTBE; −5190 and −4250 for G1; −5065 and −4405 for G2.

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