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Thermochimica Acta 285 (1996) 199–209

thermochimica
acta

Calorimetric and infrared study of methyl iodide and some monosubstituted butyl halides. Does methyl iodide form hydrogen bonds as a proton donor?

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Received 24 May 1995; accepted 16 February 1996

Abstract

The solution enthalpies of methyl iodide, *n*- and *t*-butyl chlorides, *n*- and *t*-butyl iodides in cyclohexane, carbon tetrachloride, acetone and dimethyl sulfoxide (DMSO) were measured calorimetrically and the transfer enthalpies of the solutes from cyclohexane were calculated. The comparative analysis of the transfer enthalpies shows no remarkable differences that could be attributed to specific interaction between the solvents and the solutes. In addition, the infrared (IR) spectra of the butyl halides dissolved in CCl_4 and deuterated DMSO, and the IR spectra of methyl iodide in CCl_4, deuterated acetonitrile, acetone and DMSO, were investigated. Significant enhancement of the CH_3 stretching band intensity was observed for methyl iodide solutions when going from carbon tetrachloride to H-bond-acceptor solvents. This enhancement can be ascribed to the hydrogen bonding of methyl iodide with the solvents. Meanwhile, no IR spectroscopic evidence of such hydrogen bonding was observed when the solutions of *n*- and *t*-butyl halides in organic solvents were considered. A possible reason for the disagreement between the IR and calorimetric measurements is discussed.

Keywords: Hydrogen bonding; Intermolecular interactions; IR spectra; Methyl iodide; Solvation

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1. Introduction

It is well known [1–3] that CH-groups adjacent to electron-withdrawing substituents, i.e. CH-groups in $CHCl_3$, $CHBr_3$, CH_2Cl_2, and other compounds, are able to

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PII S0040-6031(96)02908-5

display proton-donor abilities via hydrogen bond formation. The CH-groups of acetylenes are also able to form hydrogen bonds, [1,2,4,5]. There is also evidence that CH_3NO_2 and CH_3CN are the donors of the hydrogen bond [6,7]. The hydrogen bonds formed by such CH-groups are not usually very strong. However, activation of CH-groups due to the presence of an electron-acceptor substituent in the molecule can be wide-spread. Hence, estimation of the energies for such hydrogen bonding is of importance for understanding the factors affecting the solvation and reactivity of various organic compounds.

Methyl iodide (CH_3I) is the model compound used in studying the vibrational and orientational relaxation in condensed phases [8–10]. Several attempts have been made to estimate the proton donor ability of CH_3I by infrared (IR) and nuclear magnetic resonance (NMR) spectroscopy [1]. It was shown that neither CH_3I stretching frequencies not ^1H chemical shifts change significantly when going from carbon tetrachloride to proton acceptor solvents. Thus, no manifestation of hydrogen bonding was found for CH_3I.

However, more recently the considerably increased intensity of the CH_3 stretching band was established for CH_3I when going from its solution in carbon tetrachloride to that in acetone [11]. This was thought to be due to the weak hydrogen bonds formed by the methyl group of CH_3I.

The aim of this paper is a more thorough examination of the proton donor ability of methyl iodide and some other monosubstituted alkyl halides by solution calorimetry and infrared spectroscopy. Earlier, using these methods, we determined the enthalpies of hydrogen bond formation for various organic molecules [5,12–16] and comparable estimates were obtained.

2. Experimental

The substances and solvents used in the study (except *t*-butyl iodide) were commercial. *t*-Butyl iodide was synthesized by the method described in Ref. [17]. All compounds and solvents were purified and dried according to the recommended methods [18].

Solution enthalpies of the compounds were measured at 298 K with the differential quasi-adiabatic calorimeter designed by our group. The technique for the determination of the heat was described in more detail earlier [19]. The calorimeter was calibrated using the Joule effect. The solution enthalpy of potassium chloride in water was also determined to check the accuracy of the calorimeter. The solution enthalpy measured at 0.0347 mol l^{-1} corresponded to the recommended value [20].

The determined values of the solution enthalpies are the average of 4 to 6 measurements. The solute concentration (C_A) was changed in the range from 0.02 to 0.12 mol l^{-1}. No dependence of the heat effect on the solute concentration was observed. Hence, the measured heat effects were considered as the solution enthalpies at infinite dilution of the solute A in the solvent S ($\Delta_{\text{sin}}H^{A/S}$).

Infrared spectra in the range 4000 to 2300 cm^{-1} were registered at 298 K with a Specord M80 spectrometer combined with a computer.

Carbon tetrachloride and deuterated acetonitrile, acetone and dimethyl sulfoxide were used as the solvents for the infrared measurements. The concentrations of the solutes in carbon tetrachloride were in the range $0.05 < C_A < 0.25$ mol l^{-1}. The C–H absorption coefficients of the solutes did not show any dependence on C_A in the above range. This indicates that there is no self-association of solutes in carbon tetrachloride. The solute concentrations in other solvents were within the limits $0.4 < C_A < 2.5$ mol l^{-1}.

3. Results and discussion

3.1. *Infrared study of methyl iodode*

There are two main manifestations of the A–H...S hydrogen bonding in the infrared spectra: the frequency shift of the A-H stretching vibration and the increase of the integral absorption coefficient (integral intensity) of this band [21–23]. However, some hydrogen bonds do not show this remarkable frequency shift, while the enhancement of the integral intensity can be dramatic. Haloforms [24,25] and dihalomethanes [26] are well-known examples of such systems. Therefore, we paid paticular attention to the integral absorption coefficients in the C–H stretching region.

Infrared spectra of methyl iodide in solutions are shown in Fig. 1. Four bands fall in the region of CH_3 stretchings. The fundamentals 2958 and 3060 cm^{-1} represent symmetric and asymmetric vibrations ν_1 and ν_4; the bands 2833 and 2810 cm^{-1} are assigned to combination transitions of $2\nu_5$ and $\nu_3 + \nu_5 + \nu_6$ [10, 27]. It can be seen that the intensities of the fundamentals increase in the series of the media: $CCl_4 < CH_3I < CD_3CN < (CD_3)_2CO < (CD_3)_2SO$. In order to determine the integral absorption coefficients, the base lines were drawn as the straight lines connecting the smallest optical density points on the high- and low-frequency sides of the spectral range under study. Then, the combination bands were truncated by straight lines, and the total areas under the symmetric and asymmetric bands were determined. Eq. (1) was used for calculating the integral absorption coefficients of the bands ($\alpha_A/_S$, 10^4 cm $mmol^{-1}$)

$$\alpha_{A/S} = 2.303\, l^{-1}\, C_A^{-1} \int_{band} \lg(I_0/I)\, d\nu \qquad (1)$$

Where l is the cell pathlength, and I and I_0 are the percentage transmittance readings for the solution and solvent spectra. The integration in Eq. (1) covers the total area of the ν_1 and ν_4 bands. The calculated values together with the ν_1 and ν_4 band frequencies are listed in Table 1. One can see that the band positions do not change considerably when going from carbon tetrachloride to dimethly sulfoxide, while the integral absorption coefficient increases approximately four times. Such enhancement of the band intensity is too large to be explained by nonspecific interactions [25, 28].

We calculated the change of the square root of the $\alpha_{A/S}$ value for for CH_3I when going from its solution in carbon tetrachloride to other solvents: $\Delta\alpha^{1/2} = (\alpha_{A/S})^{1/2} - (\alpha_{A/CCl_4})^{1/2}$. This difference was further correlated with the polarizability, "polarity" and basicity of the solvents used. The electronic polarizability of the solvent was described

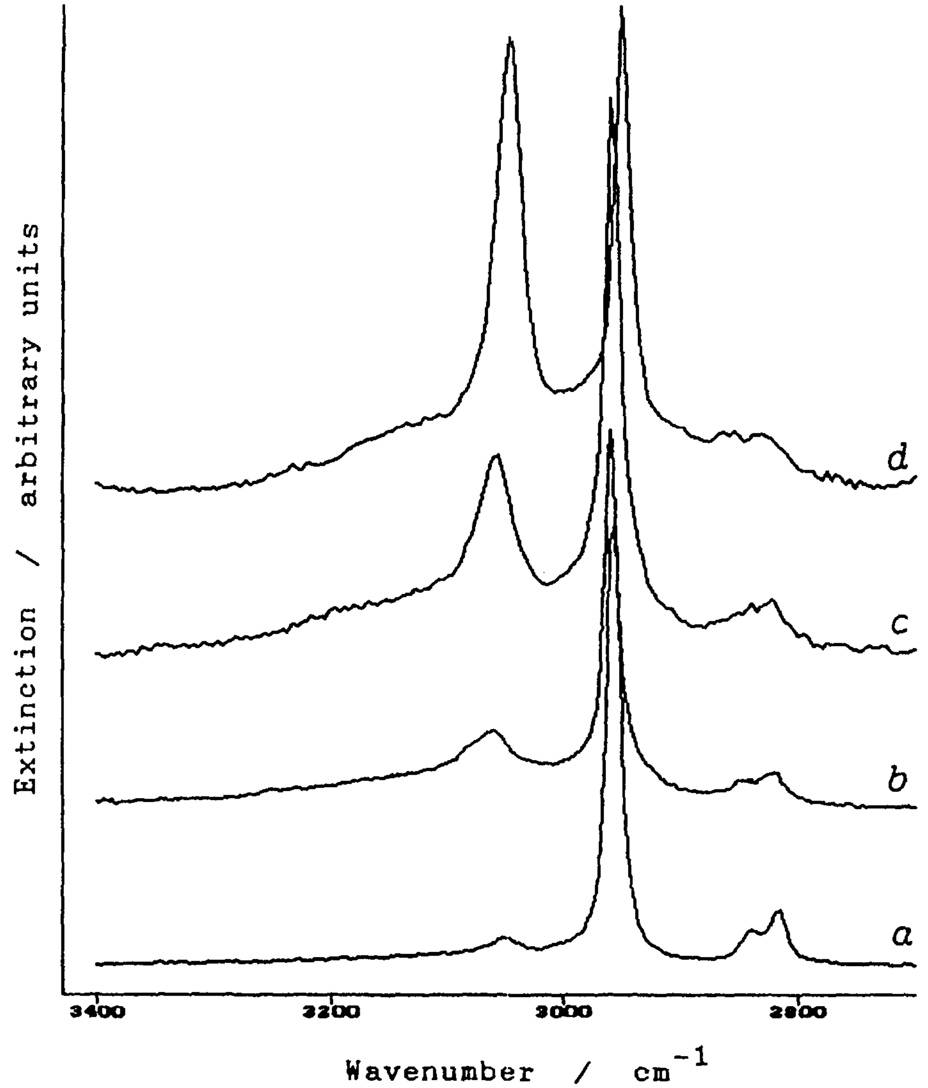

Fig. 1. IR spectra of methyl iodide in (a) carbon tetrachloride, (b) deuterated acetonitrile, (c) deuterated acetone and (d) deuterated dimethyl sulfoxide.
Extinction equals $\lg(I_0/I)/(l\,C_A)$; symbols are given in the text.

by the $(n^2-1)/(n^2+2)$ value, where n is the refractive index of the solvent. Kirkwood's function $(\varepsilon-1)/(\varepsilon+2)$ of the dielectric constant ε of the solvent is the traditional measure of the medium polarity. The enthalpy of the hydrogen bonding of phenol with a solvent molecule $\Delta_{\text{h.b.}}H^{\text{PhOH/S}}$ was considered as a measure of the basicity of the solvent. These values for the solvents under consideration, together with the parameters of the above linear correlations, are listed in Table 1. One can see that the

Table 1
Infrared data for methyl iodide in solutions

Solvent	ν_1 [a]	ν_4 [a]	$\alpha_{A/S}$ [b]	$\Delta\alpha^{1/2}$ [c]	$\frac{n^2-1}{n^2+2}$	$\frac{\varepsilon-1}{2\varepsilon+2}$	$-\Delta_{h.b.}H^{PhOH/S}$ [d]	$-\Delta_{sp.int}H^{CH_3I/S}$ [e]
CCl_4	2958	3051	0.096 ± 0.005	0.0	0.215	0.225	0.0	0.0
CH_3I	2951	3044	0.126 ± 0.006	4.5 ± 0.7	0.238	0.400	5.9	0.5 ± 0.2
CD_3CN	2960	3061	0.17 ± 0.02	10.2 ± 1.0	0.175	0.480	15.5	1.3 ± 0.4
$(CD_3)_2CO$	2959	3059	0.20 ± 0.01	13.7 ± 0.7	0.180	0.465	20.9	1.7 ± 0.2
$(CD_3)_2SO$	2951	3048	0.36 ± 0.02	29.2 ± 1.1	0.220	0.495	28.4	3.4 ± 0.4
Slope					−31 ± 658	75 ± 107	0.94 ± 0.48	
Intercept					18 ± 136	−19 ± 45	−1.7 ± 8.3	
σ [f]					12.9	8.6	3.9	
r [g]					−0.079	0.749	0.953	

[a] In cm^{-1}. [b] In 10^4 cm $mmol^{-1}$. [c] In $cm^{1/2}$ $mmol^{-1/2}$. [d] In kJ mol^{-1}; the $\Delta_{h.b.}H^{PhOH/S}$ values averaged over data from Ref. [2]. Instead of $\Delta_{h.b.}H^{PhOH/CH_3I}$, the average over the enthalpies of hydrogen bonding of phenol with *n*-butyl, *n*-hexyl, *n*-heptyl and cyclohexyl iodides [2] is used. [e] In kJ mol^{-1}. [f] σ is the standard deviation in $cm^{1/2}$ $mmol^{-1/2}$. [g] r is the correlation coefficient.

correlation is good only if the $\Delta_{\mathrm{h.b.}}H^{\mathrm{PhOH/S}}$ value is considered as the independent variable. Thus, according to the spectral criteria [21–32] and the observed correlation with the solvent's basicity, one can assume here the existence of hydrogen bonding between the methyl group of CH_3I and the proton acceptors.

To estimate the specific interaction enthalpy per mole of proton donor ($\Delta_{\mathrm{sp.int.}}H^{\mathrm{A/S}}$, kJ mol^{-1}), relationship (2) known as the intensity rule [23] was used.

$$-\Delta_{\mathrm{sp.int.}}H^{\mathrm{A/S}} = 12.1\ \Delta\alpha^{1/2} \tag{2}$$

The $\Delta_{\mathrm{sp.int.}}H^{\mathrm{A/S}}$ values calculated from Eq. (2) are also presented in Table 1. One can see that the largest $-\Delta_{\mathrm{sp.int}}H^{\mathrm{A/S}}$ value does not exceed 3.4 kJ mol^{-1}.

3.2. Analysis of solution enthalpies for methyl iodide

To evaluate calorimetrically the specific interaction enthalpies for CH_3I/solvent systems, we used the approach based on the additivity analysis of the transfer enthalpies of methyl iodide and related compounds. The solution enthalpies of methyl iodide and some other monosubstituted alkyl halides in four organic solvents were measured (Table 2).

As cyclohexane cannot form hydrogen bonds with organic compounds, it can be used as the reference solvent. The transfer enthalpies of solutes A from cyclohexane Y to other solvents S ($\Delta_t H^{\mathrm{A}}$) were calculated from the solution enthalpies by the equation

$$\Delta_t H^{\mathrm{A}} = \Delta_{\mathrm{sln}} H^{\mathrm{A/S}} - \Delta_{\mathrm{sln}} H^{\mathrm{A/Y}} \tag{3}$$

where $\Delta_{\mathrm{sln}} H^{\mathrm{A/Y}}$ is the solution enthalpy of solute A in cyclohexane. The values of the transfer enthalpies are presented in Table 3.

Both the solvation enthalpies of organic compounds and the transfer enthalpies are known to be linearly correlated with the number of methylene groups in a molecule [32–37]. Hence, the transfer enthalpy for methyl iodide from cyclohexane Y to the solvent S ($\Delta_t H^{\mathrm{CH_3I}}$) can be considered as the difference between two terms

$$\Delta_t H^{\mathrm{CH_3I}} = \Delta_t H^{\mathrm{CH_3(CH_2)_3I}} - \Delta_t H^{\mathrm{(CH_2)_3}} \tag{4}$$

Table 2
Solution enthalpies at infinite dilution in kJ mol^{-1} at 298 K

Solutes	Solvents			
	$c\text{-}C_6H_{12}$	CCl_4	CH_3COCH_3	CH_3SOCH_3
CH_3I	4.23 ± 0.13	1.21 ± 0.08	1.76 ± 0.08	2.01 ± 0.04
$n\text{-}C_4H_9I$	3.05 ± 0.13	0.21 ± 0.04	3.81 ± 0.17	7.11 ± 0.04
$t\text{-}C_4H_9I$	2.01 ± 0.08	−0.17 ± 0.08	3.26 ± 0.04	–
$n\text{-}C_4H_9Cl$	2.97 ± 0.16[a]	–	1.92 ± 0.04	5.82 ± 0.13
$t\text{-}C_4H_9Cl$	2.26 ± 0.08[a]	–	1.92 ± 0.04	6.28 ± 0.25
$-k_s$/kJ cm^{-3}[b]	1.05	1.00	0.81	0.62

[a] Ref. [29]. [b] Refs. [30, 31].

Table 3
Transfer enthalpies of solutes in kJ mol^{-1} at 298 K[a]

Solutes	Solvents		
	CCl_4	CH_3COCH_3	CH_3SOCH_3
CH_3I	-3.0 ± 0.2	-2.5 ± 0.2	-2.2 ± 0.2
n-C_4H_9I	-2.8 ± 0.2	0.8 ± 0.3	4.1 ± 0.2
t-C_4H_9I	-2.2 ± 0.2	1.3 ± 0.1	–
n-C_4H_9Cl	–	-1.1 ± 0.2	2.9 ± 0.3
t-C_4H_9Cl	–	-0.3 ± 0.1	4.0 ± 0.3

[a] Error is the sum of the accuracy of the solution enthalpies used.

where $\Delta_t H^{CH_3(CH_2)_3I}$ is the transfer enthalpy for n-butyl iodide, and $\Delta_t H^{(CH_2)_3}$ is the transfer enthalpy for three methylene groups. The last value can be calculated if the increment for the transfer enthalpy of one methylene group is known. We calculated $\Delta_t H^{(CH_2)_3}$ in a slightly different manner.

It was shown earlier [30, 31] that solution enthalpies of liquid saturated hydrocarbons in a solvent are proportional to the molecular refraction $(MR)^H$ of hydrocarbons. Hence, the transfer enthalpies for alkanes H $(\Delta_t H^H)$ should also be proportional to their molecular refraction $(MR)^H$. The vaporization enthalpies of the saturated hydrocarbons are known to be linearly correlated with the $(MR)^H$ values [31]. The intercept of this correlation depends on the kind of hydrocarbon (normal, cyclic or branched); however, the slope is the same for various classes of hydrocarbons [31]. It follows from these facts that the $\Delta_t H^H$ values can be written as

$$\Delta_t H^H = (k_S - k_Y) MR^H \tag{5}$$

where k_S and k_Y are the slopes of the linear dependences of the solvation enthalpies of the hydrocarbons upon their molecular refraction in the solvents S and Y, respectively. It is clear that the k_S and k_Y values do not depend on the nature of the saturated hydrocarbon (normal, cyclic or branched). Using the known k_S and k_Y values listed in Table 2, the transfer enthalpies $\Delta_t H^{(CH_2)_3}$ can easily be calculated by Eq. (5) from the molecular refraction of the three methylene groups, which is equal to 14.0 cm^3 mol^{-1} [38].

We evaluated the $\Delta_t H^{CH_3I}$ magnitudes by Eqs. (4) and (5): -3.5 kJ mol^{-1} in carbon tetrachloride, -2.6 kJ mol^{-1} in acetone and -1.9 kJ mol^{-1} in dimethyl sulfoxide. One can see that calculated values are in good agreement with the experimental transfer enthalpies of methyl iodide presented in Table 3. This means that the additivity (4) is valid. Therefore, there is no essential difference in the proton donor ability of methyl iodide and n-butyl iodide.

3.3. *Inspection of the proton donor ability of butyl halides*

The proton donor ability of the series of butyl halides was examined both by infrared spectroscopy and calorimetry. According to the data obtained for methyl iodide, the most distinct manifestations of hydrogen bonding in the IR spectra were expected for solutions of the compounds in deuterated dimethyl sulfoxide. Thus, only solutions of butyl halides in carbon tetrachloride and dimethyl sulfoxide were investigated using IR spectroscopy.

As an example, the IR spectra of *n*-butyl iodide are shown in Fig. 2.

The integral absorption coefficients of the compounds are presented in Table 4. No significant changes in the $\alpha_{A/S}$ values are seen when going from carbon tetrachloride to

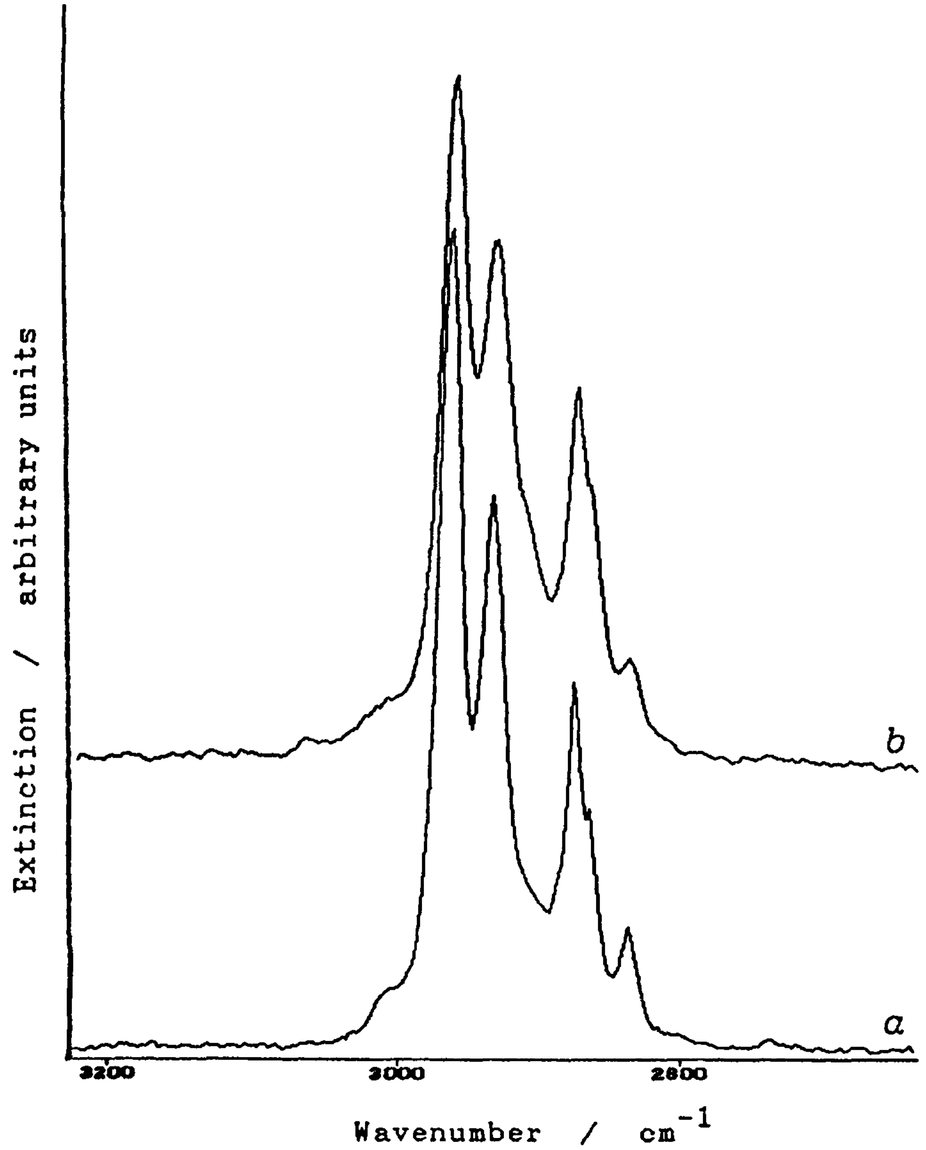

Fig. 2. IR spectra of *n*-butyl iodide in (a) carbon tetrachloride and (b) deuterated dimethyl sulfoxide.

Table 4
IR intensities of butyl halides in solutions

Solute	Solvent	$\alpha_{A/S}$ [a]	$-\Delta_{sp.int.}H^{A/S}$ [b]
n-C_4H_9I	CCl_4	2.11 ± 0.05	–
	$(CD_3)_2SO$	2.17 ± 0.05	0.2 ± 0.5
t-C_4H_9I	CCl_4	1.95 ± 0.10	–
	$(CD_3)_2SO$	1.60 ± 0.10	-1.5 ± 0.7
n-C_4H_9Cl	CCl_4	2.30 ± 0.20	–
	$(CD_3)_2SO$	2.11 ± 0.05	-0.8 ± 1.0
t-C_4H_9Cl	CCl_4	1.44 ± 0.06	–
	$(CD_3)_2SO$	1.30 ± 0.05	-0.7 ± 0.6

[a] In 10^4 cm mmol^{-1}. [b] In kJ mol^{-1}.

dimethyl sulfoxide. Moreover, the $\alpha_{A/S}$ values even decrease slightly. The specific interaction enthalpies were calculated using Eq. (2) in the same way as was done for methyl iodide. The obtained $\Delta_{sp.int}H^{A/S}$ values (see Table 4) are negligibly small.

Thus, no manifestations of hydrogen bonding are seen in the infrared spectra of butyl halides dissolved in dimethly sulfoxide.

The next step was the calorimetric examiniation of butyl halides. The possible proton- donor ability of methyl iodide is believed to be due to the presence of the electron-withdrawing substituent neighboring the methyl group. It is reasonable to assume for n-butyl halides that only the protons geminal to a halogen substituent could be able to form hydrogen bonds with proton acceptors. Based on this assumption, t-butyl halides are not able to form hydrogen bonds since there are no such elements in their structure. Therefore, it is reasonable to assume that the difference (if any) between the transfer enthalpies of n-butyl iodide and t-butyl iodide has to be equal to the enthalpy of the specific interaction of n-butyl iodide with the solvent.

One can compare the transfer enthalpies for n- and t-butyl iodides (Table 3). The difference between the transfer enthalpies of the isomers from cyclohexane to acetone equals -0.5 ± 0.4 kJ mol^{-1}. This does not differ remarkably from zero. It appears that there is no considerable heat effect which could be attributed to the hydrogen bonding between n-butyl iodide and acetone.

Unfortunately, the attempt to measure the solution enthalpy of t-butyl iodide in dimethyl sulfoxide failed because an exothermic reaction is superimposed on the endothermic dissolution of t-butyl iodide.

To check the proton donor ability of the α-methylene groups in other monosubstituted alkyl halides, we also compared the transfer enthalpies for n- and t-butyl chlorides. From Table 3 one can see that the difference between such transfer enthalpies is -0.8 ± 0.3 kJ mol^{-1} in acetone and -1.1 ± 0.6 kJ mol^{-1} in dimethyl sulfoxide. These values are significantly smaller than the enthalpies of specific interaction of methyl iodide estimated from IR spectra.

Thus, the analysis of the solution enthalpies does not indicate the remarkable heat effect which could be attributed to the hydrogen bonding of butyl halides with the solvents.

4. Discussion

It should be noted that neither the calorimetric not the IR spectroscopic measurements show features of hydrogen bonding between *n*- and *t*-butyl halides and the solvents. Taking into account the additivity analysis of solution enthalpies (see Section 3.2), we have to admit that there is no calorimetric confirmation of the hydrogen bonding between methyl iodide and the solvents either.

However, manifestation of the hydrogen bonding of methyl iodide with the solvents is observed in the infrared spectra (see Section 3.1).

The disagreement between the two methods may be due to an additional process contributing to the measured heat and "masking" the solute–solvent hydrogen bonding, which is observable in the IR spectra. The rupture of the intermolecular solvent–solvent hydrogen bonds accompanying the solute–solvent hydrogen bonding can result in such a "masking" effect [39,40]. Hence, one should expect the IR spectroscopic manifestation of the hydrogen bonding between the dimethyl sulfoxide molecules. To check this, the integral absorption coefficients of the C–H stretchings of non-deuterated dimethyl sulfoxide were measured: $(0.26 \pm 0.02) \times 10^4$ cm mmol^{-1} for dimethyl sulfoxide dissolved in carbon tetrachloride and $(0.76 \pm 0.03) \times 10^4$ cm mmol^{-1} for pure dimethyl sulfoxide. The change in the integral absorption coefficient indicates a self-association of this solvent with the participation of the methyl groups.

Thus, The IR spectroscopic registration of the weak hydrogen bonding of a solute with a solvent is not a sufficient reason to assume that this interaction results in the decrease of the solvation enthalpy of a solute.

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