

Enthalpy of Cooperative Hydrogen Bonding in the Complexes of Triethyl- and Tri-*n*-butylamines with Alcohols: Effect of the Alkyl Chain Length

M. A. Varfolomeev, K. V. Zaitseva, I. T. Rakipov, and B. N. Solomonov

*Butlerov Chemical Institute, Kazan State University,
ul. Kremlevskaya 18, Kazan, 420008 Tatarstan, Russia
e-mail: mikhail.varfolomeev@ksu.ru*

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Abstract—The measurements of enthalpies of triethylamine and tri-*n*-butylamine dissolution in aliphatic alcohols and, vice versa, of aliphatic alcohols in amines were carried out. Enthalpies of specific interactions in the studied systems were calculated. The enthalpy of specific interaction determined in the alcohol medium, are significantly less than those obtained at dissolving alcohols in amines. The mechanism of specific interaction of amines with alcohols is discussed. Enthalpies of cooperative hydrogen bonds of tertiary amines with alcohol clusters are calculated. The dependences between the enthalpies of hydrogen bond and the spatial structure of interacting molecules are revealed.

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Cooperativity is one of the basic features of intermolecular interactions [1, 2]. This phenomenon is observed especially in the formation of hydrogen bonds [3–6]. The cooperativity consists in hydrogen bonding strengthening or weakening in the multi-component complexes in comparison with binary complexes. For the first time this phenomenon was observed in the study of the structure of liquid water [7]. The cooperativity of hydrogen bonding plays an important role in supramolecular chemistry and biochemistry [4, 8–10]. The phenomenon of cooperativity of hydrogen bonds is studied by various methods: infrared spectroscopy [5, 11, 12], NMR spectroscopy [13, 14], dissolution calorimetry [15–18], and gas-liquid chromatography [19, 20]. The methods of quantum chemistry are extensively used [21] for the estimation of the cooperativity parameters in various systems.

An important problem in the study of cooperative interactions is the experimental determination of thermodynamic parameters of the formation of cooperative hydrogen bonds in the solvents associated through the hydrogen bonding, such as aliphatic alcohols, water, etc. It should be emphasized that the knowledge of the values of these parameters is of great importance, since the reactivity of the dissolved

molecules in the associated solvent depends, besides other factors, on the solute-solvent hydrogen bonding. There are reasons to believe that solute molecule in associated solvent forms H-complexes not only with one solvent molecule, but also with the collection of solvent molecules that form supramolecular clusters. To our knowledge, the methods of NMR and IR spectroscopy are insufficient for solving the problem of determining the thermodynamic parameters of hydrogen bonding in “pure” aliphatic alcohols or water. Recently, we proposed a new method of determining the enthalpy of the cooperative binding of a proton-acceptor, pyridine, with aliphatic alcohols [18]. It was found that the average value of enthalpy of the cooperative hydrogen bonding of pyridine in a series of aliphatic alcohols is -19.8 ± 0.6 kJ mol⁻¹. At the same time, the average value of enthalpy of pyridine-alcohol H-complexes of 1:1 composition in the medium of pyridine was -15.8 ± 0.2 kJ mol⁻¹. It was shown in [18] that the cooperative enhancement in the complexes of pyridine with clusters of alcohol compared with complexes with the monomer is on the average of 20–30%. This value agrees well with the data of IR spectral studies [5].

In this work we carried out calorimetric study of cooperative hydrogen bonding of aliphatic amines with

a series of aliphatic alcohols in order to study the influence of spatial effects on the enthalpy of the cooperative hydrogen bonding.

Our study is based on the following principles and definitions.

The enthalpy of solvation of compound A in solvent S ($\Delta_{\text{solv}}H^{A/S}$) is the enthalpy of the isothermal transport of A from the ideal gas into an infinitely diluted solution at 298.15 K and 1 atm. Enthalpies of dissolution of A in solvent S ($\Delta_{\text{dissol}}H^{A/S}$) is the enthalpy of transfer of A from the standard state (solid or liquid) in an infinitely diluted solution at 298.15 K and 1 atm. Enthalpies of dissolution and solvation are related by the Eq. (1):

$$\Delta_{\text{solv}}H^{A/S} = \Delta_{\text{dissol}}H^{A/S} - \Delta_{\text{vap}}H^A, \quad (1)$$

where $\Delta_{\text{vap}}H^A$ is molar enthalpy of vaporization at 298.15 K. Enthalpies of solvation can be represented as the sum of the enthalpies of nonspecific solvation [$\Delta_{\text{solv(nonspecific)}}H^{A/S}$] and the enthalpy of specific interaction [$\Delta_{\text{solv(specific)}}H^{A/S}$]:

$$\Delta_{\text{solv}}H^{A/S} = \Delta_{\text{solv(nonspecific)}}H^{A/S} + \Delta_{\text{solv(specific)}}H^{A/S}. \quad (2)$$

In turn, the enthalpy of nonspecific solvation can be represented as the sum of two terms: the enthalpy of formation of a cavity in the solvent and the enthalpy of nonspecific interactions, which are caused by van der Waals interactions.

In [17], we proposed a method for calculating the enthalpy of nonspecific solvation based on Eq. (3):

$$\begin{aligned} \Delta_{\text{solv(nonspecific)}}H^{A/S} = & (\delta_{\text{total}}h^S - \delta_{\text{total}}h^{C_6H_{12}})V_X^A + \Delta_{\text{dissol}}H^{A/C_6H_{12}} \\ & + (a_R + b_R\sqrt{\delta_{\text{total}}h^S})[(\Delta_{\text{dissol}}H^{A/R} - \Delta_{\text{dissol}}H^{A/C_6H_{12}}) \\ & - (\delta_{\text{total}}h^R - \delta_{\text{total}}h^{C_6H_{12}})V_X^A]. \end{aligned} \quad (3)$$

Here $\Delta_{\text{dissol}}H^{A/S}$ and $\Delta_{\text{dissol}}H^{A/C_6H_{12}}$ are enthalpies of dissolution of A in the standard solvent R (incapable of specific interactions with the solute) and cyclohexane, respectively; V_X^A is characteristic volume according to Mac-Gauvin; $\delta_{\text{total}}h^S$, $\delta_{\text{total}}h^R$ and $\delta_{\text{total}}h^{C_6H_{12}}$ are specific relative enthalpies of formation of cavities in the solvent S, R, and cyclohexane, characterizing the capability of these substances of nonspecific interactions. The value of $\delta_{\text{total}}h^S$ can be found by Eq. (4):

$$\delta_{\text{total}}h^S = \frac{\Delta_{\text{dissol}}H^{C_nH_{2n+2}/S}}{V_X^{C_nH_{2n+2}/S}}. \quad (4)$$

From Eqs. (1), (2), and (3) was obtained the equation for determining the enthalpy of specific interaction (5):

$$\begin{aligned} \Delta_{\text{int(specific)}}H^{A/S} = & \Delta_{\text{dissol}}H^{A/S} - (\delta_{\text{total}}h^S - \delta_{\text{total}}h^{C_6H_{12}})V_X^A \\ & - \Delta_{\text{dissol}}H^{A/C_6H_{12}} - (a_R + b_R\sqrt{\delta_{\text{total}}h^S})[(\Delta_{\text{dissol}}H^{A/R} \\ & - \Delta_{\text{dissol}}H^{A/C_6H_{12}}) - (\delta_{\text{total}}h^R - \delta_{\text{total}}h^{C_6H_{12}})V_X^A]. \end{aligned} \quad (5)$$

For the calculation of enthalpy of specific interaction of aliphatic alcohol in triethylamine and in tri-*n*-butylamine, as the standard solvent R was taken carbon tetrachloride. For the calculation of enthalpy of specific interaction of triethylamine and tri-*n*-butylamine in aliphatic alcohols was taken benzene, as carbon tetrachloride was capable of specific interaction with amines [22]. For carbon tetrachloride $a_R = 0.34$ and $b_R = 0.61$, for benzene, $a_R = 0.20$ and $b_R = 0.38$ [18]. The values of the specific relative enthalpies of cavity formation in the different solvents used in this study are taken from [17, 18].

In the case of non-associated solvents the enthalpy of specific interaction is the enthalpy of localized donor-acceptor interaction, including hydrogen bonding. In the case of an associated solvent the solute molecule capable of hydrogen bonding with the solvent, can compete for the proton-acceptor or proton-donor sites in the solvent with the solvent molecules. Accordingly, the enthalpy of specific interaction in the associated solvent may differ from the enthalpy of hydrogen bonding in 1:1 complexes between the molecules of the solute and solvent. According to our previous work [18], for associated solvents the value of $\Delta_{\text{int(specific)}}H^{A/S}$ is defined by Eq. (6):

$$\Delta_{\text{int(specific)}}H^{A/S} = \Delta_{\text{HB}}H^{A/S} + \Delta_{\text{reorg}}H^{A/S}, \quad (6)$$

where $\Delta_{\text{HB}}H^{A/S}$ is enthalpies of formation of A with the solvent associates and with the solvent, and $\Delta_{\text{reorg}}H^{A/S}$ is the enthalpy of the reorganization, which represents the enthalpy of the process associated with the rupture of solvent-solvent hydrogen bonds (*endo*-effect).

Table 1 shows the enthalpies of dissolution of aliphatic alcohols in triethylamine and tri-*n*-butylamine and the data on the enthalpies of dissolution of aliphatic alcohols in carbon tetrachloride (standard solvent) and cyclohexane taken from published sources, which are required for the calculation of specific interaction enthalpy of aliphatic alcohols in amines by Eq. (5). The characteristic volumes for the aliphatic alcohols are calculated by the additive scheme [23]. To determine the specific relative enthalpy of formation of cavities in the tri-*n*-butylamine according to [17], we have measured the enthalpies of dissolution of *n*-hexane, *n*-heptane and *n*-octane in this solvent

$[\Delta_{\text{dissol}}H^{C_6H_{14}/(C_4H_9)_3N} = 0.02 \text{ kJ mol}^{-1}$; $\Delta_{\text{dissol}}H^{C_7H_{16}/(C_4H_9)_3N} = 0.02 \text{ kJ mol}^{-1}$; $\Delta_{\text{dissol}}H^{C_8H_{18}/(C_4H_9)_3N} = 0.03 \text{ kJ mol}^{-1}$]. The value of the specific relative enthalpy of formation of cavities in the tri-*n*-butylamine was $0.02 \text{ kJ cm}^{-3} \times 10^2$. Enthalpies of specific interaction of aliphatic alcohols with triethylamine and tri-*n*-butylamine, calculated using Eq. (5), are shown in Table 1.

The values of the enthalpies of specific interaction of aliphatic alcohols with triethylamine are in good agreement with the published data on the enthalpies of formation of hydrogen bonds for 1:1 complexes for methanol, butanol, and octanol [32, 33]. The values of the enthalpy of specific interaction $\Delta_{\text{int(specific)}}H^{\text{ROH}/(C_2H_5)_3N}$ and $\Delta_{\text{int(specific)}}H^{\text{ROH}/(C_4H_9)_3N}$ do not depend on the length of the alcohol hydrocarbon chain. The enthalpy of specific interaction of aliphatic alcohols with tri-*n*-butylamine is somewhat smaller in the absolute value. This, apparently, is due to steric factors arising from the presence of bulky *n*-butyl groups in tri-*n*-butylamine. The presence of steric constraints reduces the strength of H-bonds.

Table 2 presents the data on the enthalpies of dissolution of triethylamine and tri-*n*-butylamine in aliphatic alcohols. The values of characteristic volumes V_X^A were calculated by the additive scheme [23]; the values obtained for triethylamine and tri-*n*-butylamine are 1.0538×10^{-2} and $1.8992 \times 10^{-2} \text{ cm}^3 \text{ mol}^{-1}$, respectively. Enthalpies of specific interaction were determined according to Eq. (5). Since carbon tetrachloride is capable of a donor-acceptor interaction with amines, as standard solvents have been used benzene and cyclohexane. The enthalpies of dissolution of amines in cyclohexane and benzene are respectively: $\Delta_{\text{dissol}}H^{(C_2H_5)_3/C_6H_{12}} = 1.1 \text{ kJ mol}^{-1}$ [34], $\Delta_{\text{dissol}}H^{(C_4H_9)_3/C_6H_{12}} = 2.1 \text{ kJ mol}^{-1}$ [35], $\Delta_{\text{dissol}}H^{(C_2H_5)_3/C_6H_6} =$

2.1 kJ mol^{-1} [32] and $\Delta_{\text{dissol}}H^{(C_2H_5)_3/C_6H_6} = 5.2 \text{ kJ mol}^{-1}$ [35]. The values of specific relative cavity formation enthalpies of aliphatic alcohols required for the calculation are also given in Table 2.

Table 2 presents the data on the enthalpies of specific interaction of aliphatic amines with alcohols. These data have two special features. Firstly, they are much smaller in magnitude than the enthalpies of specific interaction of aliphatic alcohols in amines (Table 1), which correspond to the enthalpy of formation of 1:1 H-complexes in the medium of amine. This difference is governed at least by two reasons. On the one hand the enthalpy of specific interaction of amines in aliphatic alcohols decreases due to the reorganization of solvent molecules [Eq. (6)]. On the other hand, it increases due to the fact that the amines form hydrogen bonds not only with one molecule of alcohol, but also with their associates. The strength of the latter exceeds the strength of hydrogen bonds in the 1:1 complexes due to the cooperative effect [30]. Secondly, the inequality of the enthalpies of specific interaction suggests that the accounting for the influence of hydrogen bonding on various physical, chemical and biological processes in associated solvents based on the thermodynamic parameters of the 1:1 complexation is not correct. More correct is to use $\Delta_{\text{int(specific)}}H^{A/S}$ value, which reflects the energy of the process of hydrogen bonding of the dissolved compounds A with the clusters of associated liquids.

The problem of the value of reorganization enthalpy [Eq. (6)] was considered in several papers [11, 15, 16]. For example, Symons et al. [11] suggested that the contribution of the reorganization to the enthalpy of solvation in aliphatic alcohols was not

Table 1. Enthalpies of dissolution of aliphatic alcohols in carbon tetrachloride and cyclohexane, the enthalpy of dissolution in triethylamine and tri-*n*-butylamine, and the enthalpy of specific interaction of alcohols with triethylamine and tri-*n*-butylamine at 298.15 K (kJ mol⁻¹)

Alcohol	$\Delta_{\text{dissol}}H^{\text{ROH}/\text{CCl}_4}$	$\Delta_{\text{dissol}}H^{\text{ROH}/\text{C}_6\text{H}_{12}}$	$\Delta_{\text{dissol}}H^{\text{ROH}/(\text{C}_2\text{H}_5)_3\text{N}}$	$\Delta_{\text{dissol}}H^{\text{ROH}/(\text{C}_4\text{H}_9)_3\text{N}}$	$\Delta_{\text{int(specific)}}H^{\text{ROH}/(\text{C}_2\text{H}_5)_3\text{N}}$	$\Delta_{\text{int(specific)}}H^{\text{ROH}/(\text{C}_4\text{H}_9)_3\text{N}}$
Methanol	18.2 ²⁴	24.3 ²⁵	-4.8 ²⁶	-0.9	-24.2 (-25.1 ³³)	-22.1
Ethanol	18.4 ²⁷	23.3 ²⁸	-3.6 ²⁹	1.8	-22.7	-18.7
Propanol-1	18.5 ³⁰	24.5 ³⁰	-4.1	1.1	-23.4	-19.9
Butanol-1	19.2 ²⁵	24.5 ³¹	-4.8 ³¹	0.9	-24.4 (-23.0 ³³)	-20.2
Hexanol-1	18.7 ¹⁷	24.4 ²⁵	-4.8 ¹⁷	0.3	-23.6	-20.0
Octanol-1	19.9 ³²	24.6 ²⁵	-5.1 ³²	0.2	-24.5 (-26.4 ³²)	-20.3

Table 2. Enthalpies of specific relative cavity formation ($\text{kJ cm}^{-3} \times 10^2$) in aliphatic alcohols, the enthalpies of dissolution of amines in alcohols, and the enthalpies of specific interaction of amines with alcohols at 298 K (kJ mol^{-1})

Alcohol	$\delta_{\text{total}}H^{\text{ROH}}$	$\Delta_{\text{dissol}}H^{(\text{C}_2\text{H}_5)_3\text{N}/\text{ROH}}$	$\Delta_{\text{dissol}}H^{(\text{C}_4\text{H}_9)_3\text{N}/\text{ROH}}$	$\Delta_{\text{int(specific)}}H^{(\text{C}_2\text{H}_5)_3\text{N}/\text{ROH}}$	$\Delta_{\text{int(specific)}}H^{(\text{C}_4\text{H}_9)_3\text{N}/\text{ROH}}$
Methanol	5.1 ¹⁸	-13.0 ³⁶	-4.7	-15.6	-9.9
Ethanol	2.8 ¹⁸	-11.3	-2.5	-11.5	-4.1
Propanol-1	1.5 ¹⁸	-10.0	-3.5	-9.3	-3.3
Butanol-1	1.6 ¹⁸	-9.6	-3.2	-9.0	-3.1
Hexanol-1	1.2 ¹⁸	-8.6	-3.4	-7.7	-2.8
Octanol-1	1.1 ¹⁸	-8.6 ³⁷	-3.0	-7.7	-2.3

Table 3. Enthalpies of self-association of alcohols, the enthalpy of formation of hydrogen bonds of triethylamine and tri-*n*-butylamine with clusters of alcohols at 298 K (kJ mol^{-1})

Alcohol	$\Delta_{\text{ass}}H^{\text{ROH}}$	$\Delta_{\text{int(specific)}}H^{(\text{ROH})_n \dots \text{N}(\text{C}_2\text{H}_5)_3}$	$\Delta_{\text{int(specific)}}H^{(\text{ROH})_n \dots \text{N}(\text{C}_4\text{H}_9)_3}$
Methanol	-15.1 ³⁰	-30.7	-25.0
Ethanol	-16.9 ³⁰	-28.4	-21.0
Propanol-1	-17.7 ³⁰	-27.0	-21.0
Butanol-1	-17.7 ³⁰	-26.7	-20.8
Hexanol-1	-17.7 ³⁰	-25.4	-20.5
Octanol-1	-17.7 ³⁰	-25.4	-20.0

constant and depended on the structure of associates of alcohol. We believe that the enthalpy of reorganization is the enthalpy of self-association, taken with the opposite sign:

$$\Delta_{\text{reorg}}H^S = \Delta_{\text{ass}}H^S. \quad (7)$$

The enthalpy of self-association is the mean value of enthalpy of formation of the hydrogen bonding in various associates of the alcohol [30].

In accordance with this model the enthalpy of the cooperative hydrogen bonding in the environment of associated alcohol is given by Eq. (8):

$$\Delta_{\text{HB}}H^{(\text{ROH})_m \dots \text{B}} = \Delta_{\text{int(specific)}}H^{B/(\text{ROH})_n} + \Delta_{\text{ass}}H^{\text{ROH}}. \quad (8)$$

In order to estimate the energy of the cooperative hydrogen bonding by Eq. (8), we used the data on the enthalpies of self-association of aliphatic alcohols from [18] (Table 3). Table 3 shows the values of $\Delta_{\text{HB}}H^{(\text{ROH})_m \dots \text{B}}$ for the complexes of amines with aliphatic alcohols. In the case of triethylamine we observed a 10–30% increase in the strength of hydrogen bonding in comparison with the 1:1 complexes. Despite the fact that the enthalpy of hydrogen bonding of pyridine with aliphatic alcohols is by 7–8 kJ mol^{-1} less than that of

triethylamine, the effect of strengthening hydrogen bonding in the complexes with associates of alcohol is on the same level (20–30%) [18].

In the case of tri-*n*-butylamine, we do not observe any strengthening in hydrogen bonding (Table 3) as compared with 1:1 complexes (Table 1). This can be caused either by the fact that, by virtue of steric hindrances, tri-*n*-butylamine forms only 1:1 complexes in the medium of alcohol, or there is the compensation of two factors affecting the hydrogen bonding enthalpy. On the one hand, the hydrogen bonding becomes stronger due to the cooperative effect, on the other hand, weakening of hydrogen bonding occurs due to the fact that the sterically hindered tri-*n*-butylamine is more “sensitive” to the steric hindrance associated with the structure of the alcohol associates.

Thus, we have studied the process of specific interaction of tertiary amines with aliphatic alcohols. We showed that triethylamine forms stronger hydrogen bonds than tri-*n*-butylamine. Enthalpies of hydrogen bonding in 1:1 complexes $\text{ROH} \cdots \text{B}$ (B is amine) in the medium of amines does not depend on the length of the alkyl chain of the alcohol molecule. The interaction

Table 4. Enthalpies of dissolution of triethylamine in methanol (kJ mol^{-1}) at various concentrations of the amine (mol l^{-1})

C	$\Delta_{\text{dissol}}H^{(\text{C}_2\text{H}_5)_3\text{N}/\text{CH}_3\text{OH}}$	C	$\Delta_{\text{dissol}}H^{(\text{C}_2\text{H}_5)_3\text{N}/\text{CH}_3\text{OH}}$
0.009	-12.7	0.043	-12.6
0.012	-12.9	0.047	-12.6
0.015	-12.9	0.053	-12.6
0.017	-13.1	0.057	-12.6
0.021	-12.8	0.072	-12.7
0.023	-12.6	0.081	-12.3
0.026	-12.5	0.082	-12.7
0.028	-12.4	0.087	-12.5
0.030	-12.9	0.091	-12.7
0.034	-12.6	0.097	-12.3
0.035	-12.5	0.102	-12.5
0.039	-12.9	0.108	-12.4

of amines with pure alcohol proceeds by a complex mechanism that includes the stage of breaking hydrogen bonds between solvent molecules and the formation of hydrogen bonding between the molecules of the proton acceptor and associates of the alcohol. Hydrogen bonds in the complexes of triethylamine with clusters of alcohol are shown to be stronger than in the complexes with the monomer due to the cooperative effect. In the case of tri-*n*-butylamine the cooperativity of hydrogen bonds is practically not observed due to the influence of steric factors. The data obtained showed that the formation of supra-molecular complexes depends strongly on the steric structure of the proton-acceptor.

EXPERIMENTAL

We used reagents from Acros Organics (content min. 99%). Before the experiment, they were further purified by standard methods [38].

Measurements of enthalpies of dissolution were carried out at 298 K using a differential quasi-adiabatic calorimeter. Methods of determining these values were described in detail previously [17]. The calorimeter was calibrated using the known value of the enthalpy of dissolution of KCl in water at maximal dilution [39]. Experimental and literature data correlate well with each other within experimental error of measurement. All values of the enthalpy of dissolution have been measured at the maximal dilution. This is confirmed by the lack of concentration dependence of

the thermal effects. Table 4 shows the enthalpy of dissolution of triethylamine in methanol at different concentrations of amine. The data obtained are practically unchanged in the range of concentrations from 0.009 to 0.108 mol l^{-1} . Therefore, changes in the degree of association do not occur. The values of dissolution enthalpy were averaged based on 6 measurements.

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