



Short communication

Water as a solute in aprotic dipolar solvents. 2. D₂O–H₂O solute isotope effects on the enthalpy of water dissolution in nitromethane, acetonitrile and propylene carbonate at 298.15 K

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ABSTRACT

The enthalpies of solution of ordinary (H₂O) and heavy (D₂O) water in nitromethane (NM), acetonitrile (ACN) and propylene carbonate (PC) were measured calorimetrically at 298.15 K. Standard (at the infinite dilution) enthalpies of solution and solvation, along with D₂O–H₂O solute isotope effects on the quantities in question, were calculated. The enthalpies of solution of water H/D isotopologues were found to be positive by sign and substantially increasing in magnitude on going from ACN and PC to NM, whereas the corresponding positive solute H/D isotope effect changes in a consequence: NM > ACN > PC. The qualitative interrelations between the enthalpy–isotopic effect of dissolution (solvation) of water and the electron-accepting/donating ability of aprotic dipolar solvent (within a series considered) were found.

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1. Introduction

In previous report [1], we have studied the enthalpies of solution, $\Delta_{\text{sol}}H^\infty$, and solvation, $\Delta_{\text{solv}}H^\circ$, for ordinary (H₂O) and heavy (D₂O) water in acetone (AC), tetrahydrofuran (THF) and 1,4-dioxane (DO) being the predominantly electron-donating solvating media with low dielectric constants, as a whole. It has been shown that, in spite of appreciable differences in the structural properties of these solvents, there are obvious interrelations between the isotope effects (IEs) $\Delta_{\text{sol(v)}}H^\infty$ (H₂O → D₂O) for water in AC, THF and DO and the Gutmann's donor (DN) and acceptor (AN) numbers for the pure aprotic dipolar media studied. Also, it is worth noticing that D₂O–H₂O solute IEs on the enthalpy of water solvation are generally equal to the IE on the energy of water–water hydrogen bond, which amounts to $-(1.0 \pm 0.1)$ kJ mol⁻¹ at 298.15 K [2–4]. This circumstance indicates the molecules of water isotopologues are really capable of forming the rather strong hydrogen (H- or D-) bonds in the media considered.

In continuation of our thermochemical studies on the solvation peculiarities of water H/D isotopologues in aprotic dipolar solvents, we report in this paper the values of $\Delta_{\text{sol}}H^\infty$ and $\Delta_{\text{solv}}H^\circ$

for H₂O and D₂O in nitromethane (NM), acetonitrile (ACN) and propylene carbonate (PC) at 298.15 K, with a view to gaining information on the energy-related changes in the structural packing of a solvent under the influence of a solute and interactions between the latter and the former. Like solvents used in the first report [1], the specified aprotic dipolar solvating media were chosen primarily for a reason of comparability of their donor-accepting properties. It is known that NM, ACN and PC, being the solvents with noticeably differing structure-packing properties, are predominantly electron-accepting media having rather high dielectric constants (ϵ) and molecular dipole moments (μ) [4–6].¹ Herewith NM and ACN, forming the “close-to-ideal” (isodielectric) solvent mixtures [10,11], are closely spaced as to parameters of intermolecular interaction with a solute H₂O or D₂O, too [12]. As regards PC, its interactions with water seem to be not much different from those in pure water since the “interaction parameters” of PC and H₂O (D₂O), as a whole, does not differ appreciably [13].

As it has been emphasized previously [1,4], the H₂O–D₂O solute isotope substitution is a fine tool for analyzing structure-packing alterations (including effects of hydrogen-bonding) induced by

¹ Also, it should be noted that the solubility of NM in water isotopologues does not exceed (in mole fractions, x_2) ~ 0.063 for H₂O and ~ 0.056 for D₂O at 298.15 K [1,7]. The miscibility gap for (PC+H₂O) was found to extend from $x_2 = 0.318$ to 0.960 at the same temperature [8,9].

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Table 1
Sources, purities, densities (ρ , g cm⁻³) and refractive indexes (n_D) of solvents used at 298.15 K.

Solvent	Source	Initial purity ^a	Water content ^b	ρ		n_D	
				Expt.	Literature	Expt.	Literature
NM	Aldrich	≥99.0	≤0.005	1.13118	1.13117 ^c , 1.13124 ^d	1.3795	1.3796 ^d
ACN	Fluka	≥99.9	≤0.003	0.77650	0.77645 ^e , 0.77652 ^f	1.3414	1.3416 ^g
PC	Merk	≥99.0	≤0.003	1.19941	1.19942 ^h , 1.19957 ⁱ	1.4197	1.4195 ⁱ

^a In wt.%.^b After purification.^c Ref. [14].^d Ref. [15].^e Ref. [16].^f Ref. [17].^g Ref. [18].^h Ref. [19].ⁱ Ref. [20].

the presence of dissolved water molecules. The point is that the replacement of H₂O by D₂O is dictated by changing both the vibrational/translational motions and donor-accepting ability of water molecules that brings about the formation of deuterium bonds being stronger compared with the similar protium bonds [2,3]. Therefore, substituting H₂O with D₂O in the cases considered here, it is important to use the solutes of the same (high) quality and to carry out the calorimetric measurements under the same conditions.

2. Experimental

Water was prepared as described previously [1]. Heavy water (Izotop Co., St. Petersburg; conductivity: 1.0×10^{-6} S cm⁻¹) with natural ¹⁸O-content and deuterium content being (99.95 ± 0.02) at.% D was used as such. Characterization data for the solvents are listed in Table 1. NM and ACN were initially treated with 0.3-nm molecular sieves for several days and then fractionally distilled in the presence of P₂O₅ under atmospheric pressure [21]. PC (4-methyl-1,3-dioxolan-2-one) was dried at first over molecular sieves (0.4-nm) for several days, too, and then fractionally distilled at reduced pressure (~2 mm Hg), according to procedure [22]. The middle fraction (of about 70%) of each solvent sample was kept over molecular sieves (0.3/0.4-nm) and distilled immediately before use, discarding the first and last fractions. The water content of these liquids (see Table 1) was determined with a Karl Fischer titration. The purity of each solvent was checked by measuring the density (ρ) and refractive index (n_D) at 298.15 K, using the procedure detailed in the paper [1]. Table 1 shows that our results are in rather good agreement with values obtained from other sources. All the solvents were stored in dark glass bottles under air-tight conditions.

The experimental enthalpies of solution of water isotopologues, $\Delta_{sol}H^m$ (m is the solution molality), in the aprotic dipolar media studied were measured at (298.15 ± 0.005) K using an automated isoperibol (ampoule-type) calorimeter fitted with a 60 cm³ reaction titanium vessel and electrical calibration (before each experiment). The calorimeter setup and experimental procedure were described in detail earlier [23].

3. Results

The measurements showed that $\Delta_{sol}H^m$ in the high dilution region do not depend (within the experimental error) on m ranging up to 0.18 mol of H₂O (D₂O) per 1 kg of the solvent. Therefore, the molar enthalpy of water dissolution at the infinite dilution, $\Delta_{sol}H^\infty$, has been calculated as the average value $|\Delta_{sol}H^m|_{av}$ over five concentration-dependent measurements, according to the procedure [24]. The experimental data on $\Delta_{sol}H^\infty$ for H₂O and

D₂O in the solvents in question are listed in Table 2 together with the results reported by others. Also, the table contains the $\Delta_{sol}H^\circ (= \Delta_{sol}H^\infty - \Delta_{vap}H^\circ)$ values with the corresponding IEs (here, $\Delta_{vap}H^\circ$ is the standard molar enthalpy of vaporization of ordinary or heavy water).

As can be seen from data of Table 2, the literature values of $\Delta_{sol}H^\infty$ are in fairly good agreement with our own, excepting those reported by Trampe and Eckert [26], and authors [25] for H₂O in NM as well. In the latter case, the enthalpic effect of solution of water differs from ours by almost 3 kJ mol⁻¹ (!). We believe that such an appreciable distinction in $\Delta_{sol}H^\infty$ may be due to both differences in the quality of a solvent sample (procedures of its preparing and testing are of prime importance) and in the experimental details. In addition, as it was mentioned in Section 1, it is important to carry out the calorimetric measurements under the same conditions for all the systems under investigated.

In Table 3, the dielectric, donor–acceptor and some other interaction characteristics of the pure solvents are collected.

4. Discussion

As it follows from the data of Table 2, the endothermic effects of dissolution of water H/D isotopologues are arranged (in magnitude) in ascending order for the solvents studied as ACN ≤ PC < NM; the negative by sign $\Delta_{sol}H^\circ$ values undergo the directly opposite changes, pointing to weakening the solvation of a water (H₂O or D₂O) molecule in the specified series of aprotic dipolar solvents, on the whole. Herewith, if $\Delta_{sol(v)}H^\infty$ (H₂O) for ACN and PC differ from each other by (0.33 ± 0.08) kJ mol⁻¹, going to D₂O, these enthalpy characteristics for both solvents in question become comparable within the experimental error. Noteworthy is also that the discussed $\Delta_{sol}H^\infty$ quantities (Table 2) are markedly higher than those determined previously [1] for water H/D isotopologues in THF, AC and DO that are the predominantly electron-donating solvents with low ϵ . When examined $\Delta_{sol}H^\infty$ or $\Delta_{sol}H^\circ$ (Table 2) in terms of variation of the “interaction parameters” presented in Table 3, no definite correlations can be found (perhaps, except for the dependences of $\Delta_{sol}H^\infty$ from the Ohtaki’s structuredness parameter S_p [31] depicted in Fig. 1 and donor–acceptor numbers as well) although it is evident that the individuality of the structure (details of a molecule) of both solvents and dissolved water H/D isotopologues is generally reflected in the order of changing the enthalpy effects considered. In the previous case [1], the similar situation took place, too.

Meanwhile, considering the data of Tables 2 and 3 jointly, one may do the conclusion that there are qualitative interrelations between IEs on $\Delta_{sol(v)}H^\infty$ and donor/acceptor numbers for the pure solvating media studied. Namely, the $\delta\Delta_{sol}H^\infty$ (H₂O → D₂O) value increases with both decreasing DN and increasing AN in a con-

Table 2
Standard molar enthalpies of solution, $\Delta_{\text{sol}}H^\circ/(\text{kJ mol}^{-1})$, and solvation, $\Delta_{\text{solv}}H^\circ/(\text{kJ mol}^{-1})^a$, of ordinary and heavy water in nitromethane (NM), acetonitrile (ACN) and propylene carbonate (PC) at 298.15 K.

Solvent	Solute H ₂ O			Solute D ₂ O			Isotope effect (D ₂ O–H ₂ O)	
	Molalities (<i>m</i>) ^b	$\Delta_{\text{sol}}H^\circ$	$-\Delta_{\text{solv}}H^\circ$	Molalities (<i>m</i>) ^b	$\Delta_{\text{sol}}H^\circ$	$-\Delta_{\text{solv}}H^\circ$	$\delta\Delta_{\text{sol}}H^\circ$	$-\delta\Delta_{\text{solv}}H^\circ$
NM	0.112 – 0.183	12.82 ± 0.02	31.19 ± 0.02	0.149 – 0.170	13.85 ± 0.03	31.54 ± 0.03	1.03 ± 0.04	0.35 ± 0.04
		10.04 ± 0.10 ^c	33.97 ± 0.10 ^c		13.47 ± 0.04 ^e	31.92 ± 0.04 ^e	0.84 ± 0.05 ^e	0.54 ± 0.05 ^e
		14.00 ± 0.30 ^d	30.01 ± 0.30 ^d		8.63 ± 0.06	36.76 ± 0.06	0.71 ± 0.08	0.67 ± 0.08
		12.63 ± 0.03 ^e	31.38 ± 0.03 ^e					
ACN	0.116 – 0.131	7.92 ± 0.05	36.09 ± 0.05	0.146 – 0.165	8.58 ± 0.03 ^{f*}	36.81 ± 0.03 ^f	0.78 ± 0.04 ^f	0.60 ± 0.04 ^f
		7.91 ± 0.10 ^c	36.10 ± 0.10 ^c					
		7.44 ± 0.17 ^d	36.57 ± 0.17 ^d					
		7.80 ± 0.02 ^{f*}	36.21 ± 0.02 ^f					
		8.26 ± 0.01 ^{g*}	35.75 ± 0.01 ^g					
7.91 ± 0.05 ^h	36.10 ± 0.05 ^h							
PC	0.072 – 0.110	8.25 ± 0.06	35.76 ± 0.06	0.143 – 0.178	8.60 ± 0.05	36.79 ± 0.05	0.35 ± 0.08	1.03 ± 0.08
		8.19 ± 0.05 ^h	35.82 ± 0.05 ^h					
		8.08 ± 0.08 ^{i*}	35.93 ± 0.08 ⁱ					

^a The values of $\Delta_{\text{vap}}H^\circ$ (298.15 K) are presented below, in Table 3.

^b Units: mole of water (H₂O or D₂O) per 1 kg of solvent.

^c Ref. [25] (hereinafter, the asterisk labels the value calculated with using the experimental enthalpies of mixing).

^d Ref. [26].

^e Ref. [27].

^f Ref. [28].

^g Ref. [29].

^h Ref. [30].

ⁱ Ref. [8].

sequence PC, ACN, and NM, and vice versa, when the question is how the $\delta\Delta_{\text{solv}}H^\circ$ (H₂O → D₂O) changes in magnitude. This seems to indicate a dominating effect of donor–acceptor interactions, including hydrogen-bonding, on the process of water solvation in the media studied. Also, the observed sequence of changes in IEs (Table 2) seems to suggest that the intermolecular interactions under discussion are the weakest in water – NM “solvate complexes” and the strongest in water – PC those because the replacement of H₂O by D₂O in the given cases is dictated by changes in the hydrogen-bonding between the interacting like or unlike molecules [1–4,12,28]. Herewith in the latter case (where water as a solute in PC), the $\delta\Delta_{\text{solv}}H^\circ$ quantity is about -1 kJ mol^{-1} that is close to the difference in the strength of the hydrogen bonds between D₂O and H₂O in the pure state [2,3,34]. This fact is fairly surprising because the same inference was made earlier [1] in respect of effects of water solvation in DO and AC being the aprotic dipolar solvents with other donor–acceptor nature (see in Section 1 also).

It was mentioned previously [1] that a positive sign at $\Delta_{\text{sol}}H^\circ$ suggests that the water–solvent interaction is weaker than the interaction between H₂O or D₂O molecules in the own aque-

ous medium. Indeed, taking into account that $\Delta_{\text{vap}}H^\circ = -\Delta_{\text{cond}}H^\circ$ (where $\Delta_{\text{cond}}H^\circ$ is the standard enthalpy of solute condensation), the sign and magnitude of $\Delta_{\text{sol}}H^\circ$ are determined by the difference between solute–solvent and solute–solute interactions, because the solvation of a solute can be identified with the condensation of 1 mol of its gaseous molecules in an infinitely large amount of a solvent [1,35]. Hence the main contributions in $(\delta)\Delta_{\text{sol}}H^\circ$ are two enthalpic effects connected with the energy expenditure at creating the solvation cavity in a solvent (a positive contribution) and the heat evolution at associating the ordinary or heavy water with a surrounding solvent generally through hydrogen-bonding and non-specific interactions (a negative contribution).

Seen in this context, the comparability of results for PC and ACN (Table 2) points to a certain similarity in the character of interaction between molecules of water and surrounding solvent. According to results [13,36], the volumes and isentropic compressibilities of pure water and water dissolved in both PC and ACN do not differ appreciably, whereas the ϵ , S_p and $\Delta_{\text{vap}}H^\circ$ values of the latter solvent are substantially different from those of water and PC (Table 3). Taking it into account, along with the observed parity between

Table 3
Dielectric constants (ϵ), dipole moments (μ), enthalpies of vaporization ($\Delta_{\text{vap}}H^\circ$), Gutmann's donor (DN) and acceptor (AN) numbers and Ohtaki's structuredness parameter (S_p) of water H/D isotopologues and aprotic dipolar solvents under study at 298.15 K.

Property	H ₂ O	D ₂ O	NM	ACN	PC
ϵ	78.3 ^c , 78.4 ^{d,e}	78.2 ^c	36.7 ^d , 35.87 ^e	36.0 ^d , 35.94 ^f	65.0 ^d , 64.92 ^f
$10^{30}\mu$ (Cm)	6.12 ^e , 6.14 ^g	6.21 ^g	11.9 ^e , 11.88 ^e	11.5 ^e , 11.78 ^f	16.6 ^e , 16.48 ^f
$\Delta_{\text{vap}}H^\circ$ (kJ mol ⁻¹)	44.01 ^c , 44.0 ^e	45.39 ^c	38.3 ^e	33.2 ^e	42.8 ^e
DN ^a	18.0 ^h	≤18.0 ^g	2.7 ^h	14.1 ^h	15.1 ^h
AN	54.8 ^h	≥54.8 ^g	20.5 ^h	18.9 ^h	18.3 ^h
S_p ^b	19.3	20.7	24.0	12.2	12.4

^a The units of the Gutmann's donor number (DN_{SbCl_5}), according to the original definition being the agreed-upon one now, are in kcal mol⁻¹. Hence, for convenience, we did not convert these units in SI.

^b Dimensionless parameter identical with the intermolecular interaction energy ($STR/\text{kJ mol}^{-1}$) due to the “three-dimensional molecular ordering” in the liquid: $\Delta_{\text{vap}}H^\circ - (DA + VDW)$ where DA and VDW are energies of the donor–acceptor and some other (of Van der Waals type) interaction energies [31].

^c Ref. [2].

^d Ref. [5].

^e Ref. [6].

^f Ref. [32].

^g Ref. [4].

^h Ref. [33].

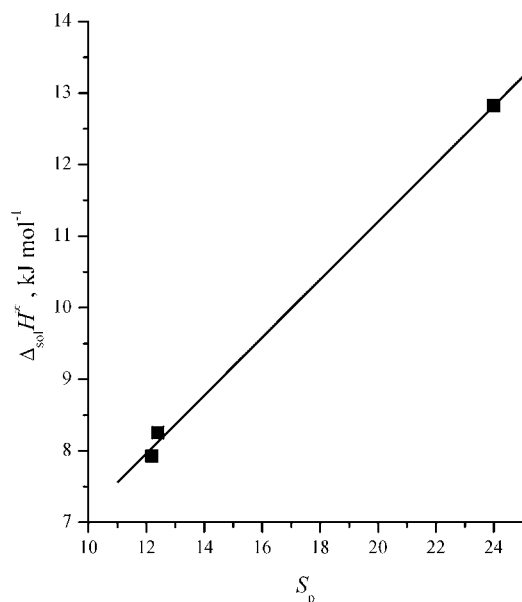


Fig. 1. The correlation between the standard enthalpy of H₂O dissolution in aprotic dipolar solvents studied and the Ohtaki's structuredness parameter of the letters (the values of 95%-confidence interval half-width for $\Delta_{\text{sol}}H^\circ$ are given in Table 2).

the enthalpy-isotope effects of water solvation in PC (see Table 2) and hydrogen-bonding in the pure water, one can agree with the assumption [13] about a similarity of water–water and PC–water interactions, in terms of “the extent of hydrogen-bond complexing” (or “strong dipole–dipole correlations”).

As seen in Table 2, the H(D)-bonding interaction in the infinitely dilute solutions of water H/D isotopologues in ACN and NM is possible too, corroborating the data of spectroscopic [25,37,38] and thermodynamic [12,28,39] studies. But, it appears to be the less pronounced, in comparison with solutions of H₂O (D₂O) in PC, due to differences in the proton-accepting and dielectric properties of aprotic dipolar media in question. Ideally, in the (ACN + water) and (NM + water) systems being isodielectric those (see above), solute–solvent interactions should be of similar nature and extent; however, such interactions as well as a water solvation on the whole are comparatively stronger in ACN (see Table 2 and [12,25,36] as well) because its *DN* is the substantially higher whereas the value of *S_p* parameter is half as large. In accord with results [25], the energy difference between H-bonding interactions of water with ACN and NM amounts to ca. 1 kJ mol^{−1} in magnitude.

Perhaps the most interesting here is the (NM + water) system, whose the data on enthalpy-isotope effects (Table 2), along with *ab initio* calculations [38] and IR-spectra [40], indicate that the solute isotopologues are weakly hydrogen-bonded with this solvent. Steric effects can account for the weakening solvation of water in NM because of the structure nature of the molecule containing two nitro-oxygen atoms. In opinion of Bonner and Choi [40], if one does not wish to believe that hydrogen bonding in a “water-oxygenated solvent” complex can be so weak, there is a possible alternative explanation. It is possible that the attraction between water and NM is purely dipolar in nature and that because of the similar geometry of the H–O–H and O–N–O bonds (with protons being positive in water and nitrogen being positive in CH₃NO₂) the water molecule assumes a position so that three oppositely

charged atoms of each of the molecules are adjacent to one another in the form of two superimposed V's. H-bonding in the usual sense is thus problematic. This is a question worth answering in future.

Thus, proceeding from the results of the present calorimetric study, one can presume that the hydrogen-bonding between water H/D isotopologues and aprotic dipolar solvents under investigation are strengthened in a series NM < ACN < PC, on the background of solute–solvent interactions in total. Interestingly, the same conclusions was made earlier by authors [41] on the basis of calorimetrically obtained data on the intermolecular interactions (donor-accepting and hydrogen-bonding) between the solvents studied and methanol. However, to understand this similarity in the interaction behavior clearly, further detailed studies are needed.

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