

THERMOCHEMICAL INVESTIGATIONS OF SOME ELECTROLYTES IN WATER–ACETAMIDE MIXTURES AT 298.15 K

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ABSTRACT

The enthalpies of solution of NaCl, KCl, KBr, KNO₃ and CsI have been measured in mixtures of acetamide with water at 298.15 K in the miscibility range. The standard enthalpies of solutions (ΔH^0) of the investigated electrolytes have been evaluated. The corresponding enthalpies of solvation (ΔH_s^0) and enthalpies of transfer (ΔH_t^0) from water to water–acetamide mixtures have been calculated. The dependence of the standard enthalpies of solution and enthalpies of transfer of the investigated electrolytes on the solvent composition is discussed.

INTRODUCTION

Amides are of considerable interest due to their ability to dissolve various substances. For this reason they are used as solvents in a variety of technological processes. Owing to their high electrical permeability they are also good solvents of electrolytes. In particular, molten acetamide has been found to be a particularly good solvent of electrolytes [1]. Very recently, the enthalpies of solution of some electrolytes in the molten acetamide were reported [2]. It has been shown [2] that the thermodynamic similarity of molten acetamide to water used as a solvent of electrolytes may be related to the fact that the three-dimensional network of H-bonds existing both in crystalline acetamide [3–5] and in ice remain in the liquid state [6]. For this reason, it seemed interesting to measure the enthalpies of solution of chosen electrolytes in water–acetamide mixtures. In the present investigation, the enthalpies of solution of NaCl, KCl, KBr, KNO₃ and CsI were measured in binary mixtures of acetamide and water in the miscibility range at 298.15 K.

EXPERIMENTAL

Acetamide (Xenon, A.R.) was crystallized twice from ethanol and dried under reduced pressure at 333 K. The melting point of acetamide was

81–82°C (354.15–355.15 K) which is in good accordance with literature data [7]. The electrolytes were purified and dehydrated by the usual methods. The solvent mixtures were prepared by weighing using double-distilled water.

The enthalpy of solution was measured using an “isoperibol” calorimeter. The calorimeter consisted of a 200-cm³ glass vessel with a tightly fitting teflon stopper. The calorimetric vessel contained two thermistors (10 kohm each) encased in stainless chromed steel, a glass stirrer and a calorimeter heater made from constantan wire ($R \approx 14$ ohms). The stirrer ended with a funnel for attaching a thin-walled glass ampoule containing the investigated salt. The ampoule was crushed against the bottom of the calorimetric vessel. A speed-controlled motor drove the stirrer. A heater coil immersed in paraffin oil and contained in a thin glass tube was used for calibration. The energy for the heater was furnished by a power supply (type ZT-980-2M, Unitra-Unima, Poland). The current was determined by measuring the potential drop across a standard 1-ohm resistance in series with the heater by means of digital voltmeter (type V540, Mera-Tronik, Poland). The time of heating is given by an electronic centisecond stopclock synchronized with the heater switch. Electrical calibration was performed after each experiment. The calorimeter was placed in a thermostated water bath. The temperature of this bath was regulated at 298.15 ± 0.005 K. The calorimeter temperature was measured by a thermistor system. The thermistors were connected to an astable multivibrator system generating a rectangular wave whose frequency is a function of the temperature inside the calorimeter vessel. Changes in the frequency of the wave were determined using a digital frequency meter (type PFL-21, Kabid, Poland) with a digital readout. The temperature sensitivity was 1×10^{-4} K. The calorimeter was checked for overall accuracy by measurements of the heat of solution of KCl(s) in water. The mean of four determinations corrected to a molar ratio of 1 : 200 for KCl/H₂O (17.25 ± 0.05 kJ mol⁻¹) deviated 0.2% from the average based on literature data (17.234 kJ mol⁻¹) [8,9]. In general, two or more determinations were made for a particular concentration and the results were averaged.

RESULTS

The enthalpies of solution of the investigated electrolytes have been measured over the concentration range 0.01–0.2 mol l⁻¹. The results of the measurements of the integral enthalpies of solution in water–acetamide mixtures are given in Tables 1–5. The plots of the integral enthalpy of solution (ΔH_m) vs. the square-root of the concentration of the investigated electrolytes are linear and they have been extrapolated to zero concentration for evaluation of the standard enthalpies of solution (ΔH^0). The lack of the necessary data makes it impossible to calculate ΔH^0 by the Criss–Cobble method [10]. The values of the standard enthalpies of solution of the

TABLE 1
The enthalpies of solution, ΔH_m , of NaCl in water-acetamide mixtures at 298.15 K (kJ mol⁻¹)

m (mol kg ⁻¹)	5 wt% (1.58 mol%) AcNH ₂	m (mol kg ⁻¹)	15 wt% (5.10 mol%) AcNH ₂	m (mol kg ⁻¹)	30 wt% (11.55 mol%) AcNH ₂	m (mol kg ⁻¹)	50 wt% (23.36 mol%) AcNH ₂
0.0120	3.90	0.0147	3.71	0.0122	3.33	0.0115	2.50
0.0188	3.92	0.0195	3.72	0.0178	3.34	0.0190	2.51
0.0250	3.93	0.0273	3.73	0.0268	3.35	0.0274	2.52
0.0331	3.95	0.0402	3.76	0.0410	3.37	0.0410	2.53
0.0399	3.97	0.0550	3.78	0.0545	3.39	0.0575	2.54
0.0529	3.99	0.0683	3.79	0.0675	3.40	0.0809	2.54
0.0651	4.01	0.0821	3.80	0.0801	3.41	0.1007	2.53
0.0760	4.03	0.1001	3.81	0.1004	3.42	0.1215	2.52
0.0992	4.07	0.1211	3.82	0.1218	3.42	0.1507	2.50
0.1204	4.08	0.1501	3.83	0.1502	3.41	0.1721	2.47
0.1502	4.09	0.1698	3.83	0.1711	3.40	0.2017	2.45
0.1713	4.09	0.1985	3.82	0.2021	3.39	—	—
0.2007	4.09	—	—	—	—	—	—

TABLE 2
The enthalpies of solution, ΔH_m , of KCl in water-acetamide mixtures at 298.15 K (kJ mol^{-1})

m (mol kg^{-1})	5 wt% (1.58 mol%) AcNH ₂	m (mol kg^{-1})	15 wt% (5.10 mol%) AcNH ₂	m (mol kg^{-1})	30 wt% (11.55 mol%) AcNH ₂	m (mol kg^{-1})	50 wt% (23.36 mol%) AcNH ₂
0.0095	17.28	0.0101	16.84	0.0103	16.21	0.0084	15.08
0.0160	17.31	0.0171	16.86	0.0202	16.23	0.0199	15.10
0.0222	17.32	0.0237	16.87	0.0362	16.25	0.0262	15.11
0.0301	17.35	0.0292	16.88	0.0403	16.27	0.0337	15.12
0.0407	17.36	0.0402	16.90	0.0512	16.28	0.0441	15.13
0.0569	17.40	0.0562	16.92	0.0630	16.30	0.0598	15.14
0.0746	17.43	0.0744	16.93	0.0799	16.31	0.0798	15.16
0.0999	17.45	0.1002	16.96	0.1003	16.33	0.1012	15.17
0.1202	17.46	0.1207	16.98	0.1244	16.36	0.1249	15.19
0.1503	17.47	0.1499	16.99	0.1502	16.38	0.1503	15.20
0.1745	17.48	0.1744	17.00	0.1745	16.39	0.1750	15.20
0.2001	17.48	0.1999	17.01	0.2003	16.39	0.2012	15.21

TABLE 3
The enthalpies of solution, ΔH_m , of KBr in water-acetamide mixtures at 298.15 K (kJ mol^{-1})

m (mol kg^{-1})	5 wt% (1.58 mol%) AcNH ₂	m (mol kg^{-1})	15 wt% (5.10 mol%) AcNH ₂	m (mol kg^{-1})	30 wt% (11.55 mol%) AcNH ₂	m (mol kg^{-1})	50 wt% (23.36 mol%) AcNH ₂
0.0090	19.87	0.0102	19.10	0.0097	17.87	0.0102	15.61
0.0146	19.90	0.0174	19.13	0.0148	17.89	0.0172	15.63
0.0182	19.91	0.0249	19.16	0.0238	17.92	0.0260	15.66
0.0232	19.92	0.0363	19.18	0.0335	17.94	0.0351	15.68
0.0370	19.96	0.0455	19.21	0.0505	17.97	0.0502	15.70
0.0490	20.01	0.0580	19.24	0.0747	18.00	0.0714	15.73
0.0611	20.04	0.0725	19.26	0.0999	18.04	0.0904	15.76
0.0731	20.07	0.1002	19.29	0.1237	18.06	0.1219	15.79
0.0998	20.09	0.1240	19.30	0.1507	18.08	0.1515	15.80
0.1244	20.09	0.1505	19.30	0.1698	18.09	0.1718	15.81
0.1501	20.09	0.1714	19.29	0.2022	18.10	0.1995	15.82
0.1724	20.08	0.1998	19.29	—	—	—	—
0.2004	20.07	—	—	—	—	—	—

TABLE 4
The enthalpies of solution, ΔH_m , of KNO_3 in water-acetamide mixtures at 298.15 K (kJ mol^{-1})

m (mol kg^{-1})	5 wt% (1.58 mol\%) AcNH_2	m (mol kg^{-1})	15 wt% (5.10 mol\%) AcNH_2	m (mol kg^{-1})	30 wt% (11.55 mol\%) AcNH_2	m (mol kg^{-1})	50 wt% (23.36 mol\%) AcNH_2
0.0065	34.74	0.0106	34.04	0.0079	32.81	0.0085	30.74
0.0105	34.75	0.0153	34.06	0.0161	32.82	0.0139	30.76
0.0194	34.77	0.0190	34.08	0.0258	32.83	0.0221	30.78
0.0330	34.78	0.0230	34.09	0.0385	32.84	0.0332	30.80
0.0402	34.79	0.0288	34.10	0.0502	32.83	0.0466	30.82
0.0582	34.78	0.0498	34.09	0.0737	32.78	0.0710	30.81
0.0796	34.75	0.0732	34.05	0.0928	32.72	0.0942	30.80
0.0998	34.70	0.0938	33.98	0.1153	32.65	0.1196	30.78
0.1202	34.62	0.1140	33.94	0.1412	32.55	0.1464	30.75
0.1501	34.55	0.1457	33.83	0.1712	32.42	0.1732	30.71
0.1733	34.46	0.1711	33.74	0.2078	32.27	0.2035	30.66
0.2007	34.38	0.2036	33.59	—	—	—	—

TABLE 5

The enthalpies of solution, ΔH_m , of CsI in water-acetamide mixtures at 298.15 K (kJ mol^{-1})

m (mol kg^{-1})	5 wt% (1.58 mol%) AcNH ₂	m (mol kg^{-1})	15 wt% (5.10 mol%) AcNH ₂	m (mol kg^{-1})	30 wt% (11.55 mol%) AcNH ₂	m (mol kg^{-1})	50 wt% (23.36 mol%) AcNH ₂
0.0070	32.81	0.0084	31.36	0.0094	29.38	0.0070	25.50
0.0112	32.82	0.0115	31.37	0.0132	29.39	0.0151	25.51
0.0190	32.83	0.0193	31.39	0.0202	29.40	0.0226	25.52
0.0243	32.84	0.0260	31.40	0.0273	29.41	0.0375	25.51
0.0392	32.85	0.0377	31.42	0.0375	29.42	0.0499	25.49
0.0505	32.84	0.0503	31.42	0.0502	29.41	0.0630	25.44
0.0630	32.83	0.0612	31.41	0.0647	29.36	0.0822	25.38
0.0749	32.82	0.0748	31.39	0.0802	29.31	0.0999	25.32
0.0955	32.81	0.0998	31.35	0.1002	29.27	0.1375	25.17
0.1163	32.78	0.1203	31.31	0.1248	29.19	0.1650	25.05
0.1501	32.73	0.1498	31.24	0.1556	29.09	0.2001	24.91
0.2002	32.67	0.1978	31.10	0.1987	28.94	—	—

TABLE 6

The standard enthalpies of solution, ΔH^0 , of some electrolytes in water-acetamide mixtures at 298.15 K (kJ mol^{-1})

Electrolyte	Water [9,11,13]	5 wt% (1.58 mol%) AcNH ₂	5 wt% (5.10 mol%) AcNH ₂	30 wt% (11.55 mol%) AcNH ₂	50 wt% (23.36 mol%) AcNH ₂
NaCl	3.89	3.81	3.64	3.27	2.47
NaI [14]	-7.58	-7.87	-8.58	-9.80	-12.22
KCl	17.25	17.16	16.79	16.16	15.05
KBr	20.05	19.76	19.00	17.79	15.53
KNO ₃	34.95	34.70	33.95	32.78	30.68
CsI	33.07	32.78	31.31	29.34	25.49

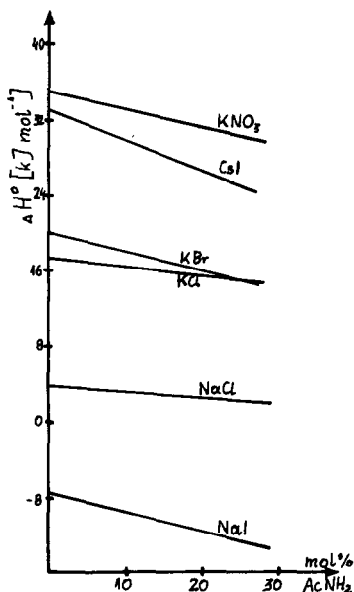


Fig. 1. The standard enthalpy of solution (ΔH_m^0) of some electrolytes in water-acetamide mixtures as a function of the mole fraction of AcNH_2 at 298.15 K.

investigated electrolytes in water-acetamide mixtures are given in Table 6 and they are presented as a function $\Delta H^0 = f(x)$ in Fig. 1.

The enthalpies of solvation (ΔH_s^0) for all the investigated electrolytes have been calculated from the standard enthalpies of solution (ΔH^0) and lattice enthalpy ($\Delta H_{\text{cryst.}}$) using the relation

$$-\Delta H_s^0 = \Delta H_{\text{cryst.}} - \Delta H^0 \quad (1)$$

The lattice enthalpies of the alkali halides at 298 K are taken from the literature [11]. The value of $\Delta H_{\text{cryst.}}$ for KNO_3 was calculated from Krestov and Zverev's data [12].

Finally, Table 7 lists the enthalpies of solvation of the investigated electrolytes both in water and water-acetamide mixtures calculated in accordance with eqn. (1). Using the values of ΔH^0 or ΔH_s^0 of the investigated electrolytes in water and water-acetamide mixtures the enthalpies of transfer, ΔH_t^0 , from water to water-acetamide mixed solvents can be calculated. Thus

$$\Delta H_t^0 = \Delta H_{(\text{ms})}^0 - \Delta H_{(\text{w})}^0 = \Delta H_{\text{s}(\text{ms})}^0 - \Delta H_{\text{s}(\text{w})}^0 \quad (2)$$

In eqn. (2), $\Delta H_{(\text{ms})}^0$ ($\Delta H_{\text{s}(\text{ms})}^0$) and $\Delta H_{(\text{w})}^0$ ($\Delta H_{\text{s}(\text{w})}^0$) are the enthalpies of solution (enthalpies of solvation) at infinite dilution of the salt in the mixed solvent, ms, and in water, w, respectively. The dependence of the enthalpies of transfer, ΔH_t^0 , of the investigated electrolytes in water-acetamide mixtures on the solvent composition is presented in Fig. 2.

TABLE 7

The enthalpies of solvation, $-\Delta H_s^0$, of some electrolytes in water–acetamide mixtures at 298.15 K (kJ mol^{-1})

Electrolyte	Water	5 wt% (1.58 mol%) AcNH ₂	15 wt% (5.10 mol%) AcNH ₂	30 wt% (11.55 mol%) AcNH ₂	50 wt% (23.36 mol%) AcNH ₂
NaCl	773.8	773.9	774.1	774.4	775.2
NaI	696.2	696.4	697.3	698.4	700.8
KCl	689.7	689.8	690.2	690.8	691.9
KBr	655.6	655.8	656.6	657.8	660.1
KNO ₃	652.5	652.8	653.5	654.7	656.8
CsI	554.2	554.5	556.0	558.0	561.8

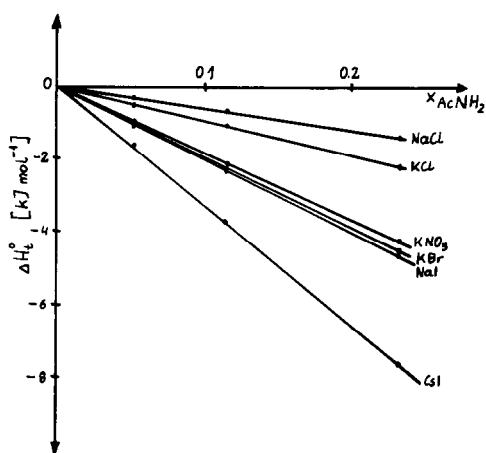


Fig. 2. The enthalpy of transfer (ΔH_t^0) of some electrolytes in water–acetamide mixtures as a function of the mole fraction of AcNH₂ at 298.15 K.

It is known that the enthalpy of solvation of the salts is additively built up from the individual values of the ions. This is demonstrated by the constant difference of the enthalpies of solvation of salts with one common ion. From the obtained values of ΔH_s^0 of the investigated salts in water–acetamide mixtures, it is possible to calculate the values of the enthalpies of solvation of non-investigated salts (NaBr, NaNO₃, KI, CsCl, CsBr). Thus

$$\Delta H_{s(\text{NaBr})}^0 = \Delta H_{s(\text{NaCl})}^0 + \Delta H_{s(\text{KBr})}^0 - \Delta H_{s(\text{KCl})}^0$$

$$\Delta H_{s(\text{KI})}^0 = \Delta H_{s(\text{KCl})}^0 + \Delta H_{s(\text{NaI})}^0 - \Delta H_{s(\text{NaCl})}^0$$

$$\Delta H_{s(\text{CsCl})}^0 = \Delta H_{s(\text{CsI})}^0 + \Delta H_{s(\text{NaCl})}^0 - \Delta H_{s(\text{NaI})}^0 \quad (3)$$

$$\Delta H_{s(\text{CsBr})}^0 = \Delta H_{s(\text{CsI})}^0 + \Delta H_{s(\text{KBr})}^0 - \Delta H_{s(\text{KI})}^0$$

$$\Delta H_{s(\text{NaNO}_3)}^0 = \Delta H_{s(\text{NaCl})}^0 + \Delta H_{s(\text{KNO}_3)}^0 - \Delta H_{s(\text{KCl})}^0$$

TABLE 8

The standard enthalpies of solution, ΔH^0 , and enthalpies of solvation, ΔH_s^0 , for NaBr, NaNO₃, KI, CsCl and CsBr calculated from eqn. (3) in water-acetamide mixtures at 298.15 K (kJ mol⁻¹)

Electrolyte	Water	5 wt% (1.58 mol%)		15 wt% (5.10 mol%)		30 wt% (11.55 mol%)		50 wt% (23.36 mol%)	
		ΔH^0	$-\Delta H_s^0$	ΔH^0	$-\Delta H_s^0$	ΔH^0	$-\Delta H_s^0$	ΔH^0	$-\Delta H_s^0$
NaBr	-0.8 [13]	739.8	739.9	-1.3	740.5	-2.2	741.4	-4.2	743.4
NaNO ₃	20.5 [15]	736.5	736.9	19.6	737.4	18.7	738.3	16.9	740.1
KI	20.5 [13]	612.1	612.3	19.2	613.4	17.8	614.8	15.1	617.5
CsCl	18.8 [16] 17.4 [17]	631.8	632.0	16.9	632.8	15.7	634.0	13.5	636.2
CsBr	26.1 [13]	597.6	598.0	24.5	599.2	22.7	601.0	19.3	604.4

The values of the standard enthalpy of solution for NaI in water–acetamide mixtures are taken from the literature [14]. From these values of ΔH_s^0 for NaBr, NaNO₃, KI, CsCl and CsBr in water–acetamide mixtures and ΔH_s^0 in water it is possible to calculate the standard enthalpies of solution of NaBr, NaNO₃, KI, CsCl and CsBr in water–acetamide mixtures. The values of ΔH^0 and ΔH_s^0 of these salts in water–acetamide mixtures are given in Table 8.

DISCUSSION

The relationship between the standard enthalpy of solution, ΔH^0 , of the investigated electrolytes in water–acetamide mixed solvent and the composition of the solvent for all salts is shown in Fig. 1. As it can be seen from Fig. 1 the course of the function $\Delta H^0 = f(x)$ of the investigated electrolytes in water–acetamide mixtures is linear suggesting that interactions between water and acetamide molecules in the mixed solvent are very weak and that the enthalpy of solution at infinite dilution of the salts is proportional to the mole fraction of acetamide in the mixed solvent. Therefore, one can draw the conclusion that the structure of water–acetamide mixtures changes gradually. Moreover, it can be noted (see Fig. 1) that the standard enthalpies of solution (ΔH^0) of the electrolytes in water–acetamide mixed solvents are more exothermic as the acetamide content in the mixed solvent increases.

These more exothermic enthalpies of solution of salts in water–acetamide mixtures indicate a stronger interaction of the electrolyte with the mixed solvent than those in water.

The course of the functions $\Delta H^0 = f(x)$ of NaI in water–formamide [18,19], water–*N,N*-dimethylformamide (DMF) [18,20] and water–DMSO [21,22] mixtures are expressed by monotonous curves with no extrema. The extrema were observed in the case of mixtures of alcohols with water [23–27], acetonitrile–water [28] and acetone–water [29]. It may be supposed that none of the factors responsible for the extrema which appear in the systems referred to the above ordering of water structure in the mixtures of alcohols with water. Complex formation in the case of acetonitrile and acetone, etc., plays a distinct role in the amide–water systems. Probably, on adding amide (F, AcNH₂, DMF) to water, the three-dimensional network of H-bonds in water is gradually destroyed and water–amide associates are being continuously formed. In the formamide–water system the structure of the mixed solvent is labile [30,31], while in case of water–DMF mixtures, associates of definite composition can be discerned [32–34]. In water–tetrahydrofuran (THF) mixtures the maximum of the standard enthalpy of solution for NaI occurs within the range 0.035–0.045 mole fraction THF [35], but in the case of NaCl this maximum does not occur [35–37]. The reason for the lack of a maximum for the NaCl solution is not clear. It seems

TABLE 9

Transfer parameters for various electrolytes from water to water–acetamide mixed solvent at 298.15 K

NaCl	0.057	KBr	0.194
NaI	0.195	KNO ₃	0.185
KCl	0.094	CsI	0.324

that the enthalpy of transfer (ΔH_t^0) is the function which better reflects the interactions between dissolved electrolyte and solvent. As it is seen from Fig. 2 the values of the enthalpy of transfer for the investigated electrolytes are more exothermic in the water–acetamide mixed solvents than those in water and they increase as the content of acetamide in the mixed solvent increases. Moreover, the dependence $\Delta H_t^0 = f(x)$ in the case of water–acetamide mixed solvents is linear. It is thus implied that ΔH_t^0 may be expressed by the equation $\Delta H_t^0 = ax_{\text{AcNH}_2}$ where x_{AcNH_2} is the mole fraction of acetamide in the mixed solvent.

From experimental data on the enthalpy of transfer, the slopes (coefficient a) of these lines can be calculated. They are summarized in Table 9. The coefficient a may be regarded as a measure of the interaction between the

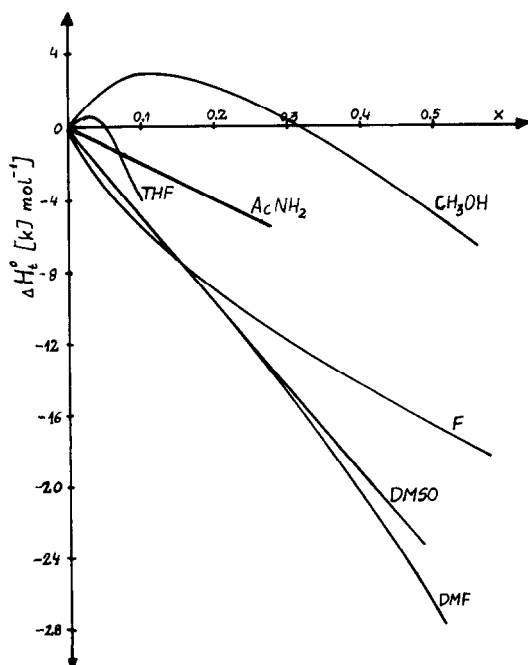


Fig. 3. The enthalpy of transfer (ΔH_t^0) of NaI in several water–organic mixed solvents as a function of the mole fraction of the non-electrolyte at 298.15 K.

electrolyte and the mixed solvent. From the obtained values it follows that the interaction between the dissolved electrolyte and the water–acetamide mixture rises as the size of the ion grows, and for cations and anions. Hence, CsI introduced into water–acetamide mixed solvents strongly disturbs the structure of the mixed solvent but NaCl acts only weakly.

For comparison, the course of ΔH_t^0 for NaI in several water–organic solvents as a function of the mole fraction of the non-electrolyte are presented in Fig. 3. As we can see from Fig. 3 the course of the function of $\Delta H_t^0 = f(x)$ for methanol–water (and other alcohols) and THF–water is completely different than for the remaining systems. The dependence of ΔH_t^0 on the composition of the mixture for DMF–water and DMSO–water is nearly identical. It is in agreement with the opinion that DMSO is an aprotic dipolar solvent, and shows, to a fair extent, the same solvent properties as DMF. The interaction of NaI with water–acetamide mixtures is smaller than those in F–water, DMSO–water and DMF–water systems.

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