A C T A U N I V E R S I T A T I S L O D Z I E N S I S
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DENSIMETRIC INVESTIGATIONS OF WATER - ACETAMIDE - KNO_3 TERNARY SYSTEM WITHIN THE TEMPERATURE RANGE 25-85°C

The density of KNO_3 solutions in water - acetamide mixed solvents has been measured. The apparent molal volume ϕ_V of KNO_3 and coefficient of volume expansion α has been calculated.

The partial molal volume $\bar{V}_2^\circ$ of KNO_3 in mixed water - acetamide solvents by extrapolation up to c = 0 were obtained. The dependence of $\bar{V}^\circ_{KNO_3}$ and α coefficient of investigated solutions on the concentration, the composition of the mixed solvent and temperature has been discussed. The conclusions about the effect of KNO_3 on the structure of water - acetamide mixed solvents have been drawn.

From many papers [1-17] follows that the analysis for the partial molal volume of the electrolyte as a function of the composition of mixed solvent and temperature enables to drawn some conclusions concerning the interactions between the solute and the solvent. The previous investigations of water - acetamide system [18-21] showed that there exist spatial associates in which the molecules of water and acetamide are likely connected by hydrogen bonds. Moreover from papers [23-24] it follows that the hydrogen bonds in the associates water - acetamide are weaker than the hydrogen bonds in water.

In order to obtain further conclusions about the water - acetamide mixed solvents the measurements of the density of the ternary H_2O - $AcNH_2$ - KNO_3 system within the temperature range 25-85°C were carried out. It enables to calculate the apparent molal volume of KNO_3 and the coefficient of volume expansion α of

the investigated solutions. The analysis of change of these quantities as a function of the mole fraction of $AcNH_2$ and temperature of the mixed solvent made it possible to drawn certain conclusions concerning the influence of KNO_3 on the structure of water - acetamide mixtures.

Experimental

The solutions used for investigations were made by mixing the weighted amounts of twice destilled water with acetamide p.a. produced by Xenon - Łódź. The method of purification of acetamide and KNO_3 was described earlier [18,25]. The measurements of density of examined solutions were carried out with the float magnetic densimeter. The method of measurements of density was described previously [19].

The density of the solutions was calculated from the formula:

$$d = \frac{M + m + f \cdot i_o}{V + m/d_{Pt}} \qquad (1)$$

in which M - the weight of the float
m - the weight of the platinum rings on the float
f - the solenoid constant
i_o - the current intensity in the measuring solenoid at the moment of departure of the float from the bottom
V - the volume of the float
d_{Pt} - the density of the platinum at the temperature of the measurement

The accuracy of the density measurements was $1 \cdot 10^{-5}$ g/cm^3.

Results

The results of the measurements of the density of ternary H_2O - $AcNH_2$ - KNO_3 systems at the temperatures 25, 40, 60, 75 and 85°C are presented in tab. 1-5.

The values of density of the investigated solutions were used to calculate the apparent molal volume of KNO_3 according

Table 1. Density and apparent molal volume of KNO_3 in water - acetamide mixture containing 5 wt % of $AcNH_2$

m	25°C		40°C		60°C		75°C		85°C	
[mole/kg]	d [g/cm^3]	ϕ_v [cm^3/mole]	d [g/cm^3]	ϕ_v [cm^3/mole]	d [g/cm^3]	ϕ_v [cm^3/mole]	d [g/cm^3]	ϕ_v [cm^3/mole]	d [g/cm^3]	ϕ_v [cm^3/mole]
0.0200	1.00145	39.5	0.99625	40.9	0.98675	42.3	0.97814	42.7	0.97170	43.1
0.0495	1.00326	39.6	0.99803	40.7	0.98846	42.5	0.97984	42.8	0.97340	42.9
0.0694	1.00447	39.7	0.99921	40.9	0.98961	42.6	0.98098	42.9	0.97454	43.0
0.0989	1.00626	39.8	1.00096	41.0	0.99132	42.6	0.98267	42.9	0.97622	43.1
0.1979	1.01224	39.9	1.00680	41.2	0.99700	42.7	0.98833	42.9	0.98184	43.1
0.2968	1.01814	40.0	1.01251	41.5	1.00253	43.0	0.99393	42.9	0.98742	43.1
0.3956	1.02395	40.2	1.01817	41.6	1.00802	43.2	0.99941	43.1	0.99292	43.1
0.4948	1.02970	40.3	1.02377	41.8	1.01352	43.2	1.00481	43.3	0.99836	43.3
0.5937	1.03538	40.5	1.02928	41.9	1.01892	43.3	1.01036	43.1	1.00402	42.9
0.6926	1.04142	40.0	1.03503	41.6	1.02458	42.9	1.01601	42.7	1.00931	43.0

Table 2. Density and apparent molal volume of KNO_3 in water - acetamide mixture containing 15 wt % of $AcNH_2$.

m	25°C		40°C		60°C		75°C		85°C	
	d	ϕ_v	d	ϕ_v	d	ϕ_v	d	ϕ_v	d	ϕ_v
[mole/kg]	[g/cm^3]	[cm^3/mole]	[g/cm^3]	[cm^3/mole]	[g/cm^3]	[cm^3/mole]	[g/cm^3]	[cm^3/mole]	[g/cm^3]	[cm^3/mole]
0.0201	1.00817	40.5	1.00199	42.4	0.99139	44.7	0.98214	45.1	0.97534	45.0
0.0347	1.00905	40.6	1.00284	42.5	0.99222	44.4	0.98295	45.2	0.97616	44.8
0.0497	1.00996	40.6	1.00371	42.6	0.99307	44.3	0.98379	45.0	0.97700	44.7
0.0694	1.01115	40.6	1.00486	42.6	0.99418	44.3	0.98488	45.1	0.97810	44.7
0.0999	1.01296	40.8	1.00662	42.7	0.99590	44.3	0.98658	45.0	0.97980	44.7
0.1980	1.01878	41.0	1.01227	42.7	1.00143	44.1	0.99204	44.7	0.98524	44.6
0.2968	1.02458	41.1	1.01790	42.8	1.00691	44.1	0.99745	44.8	0.99070	44.5
0.3956	1.03036	41.1	1.02351	42.8	1.01241	44.0	1.00291	44.5	0.99612	44.4
0.4948	1.03602	41.3	1.02911	42.7	1.01788	43.9	1.00838	44.4	1.00155	44.3
0.5937	1.04154	41.5	1.03446	43.0	1.02326	43.9	1.01382	44.2	1.00698	44.1

Table 3. Density and apparent molal volume of KNO_3 in water-acetamide mixture containing 30 wt % of $AcNH_2$.

m	25°C		40°C		60°C		75°C		85°C	
[mole/kg]	d [g/cm^3]	Φ_v [cm^3/mole]	d [g/cm^3]	Φ_v [cm^3/mole]	d [g/cm^3]	Φ_v [cm^3/mole]	d [g/cm^3]	Φ_v [cm^3/mole]	d [g/cm^3]	Φ_v [cm^3/mole]
0.0200	1.01830	43.8	1.01046	45.1	0.99806	46.5	0.98833	47.5	0.98079	47.4
0.0346	1.01914	43.7	1.01127	45.3	0.99886	46.4	0.98910	47.7	0.98158	47.1
0.0494	1.01998	43.9	1.01210	45.2	0.99967	46.3	0.98990	47.4	0.98239	46.7
0.0693	1.02112	43.8	1.01322	45.0	1.00077	46.1	0.99098	47.2	0.98347	46.6
0.0991	1.02283	43.7	1.01490	44.8	1.00240	46.0	0.99259	47.0	0.98509	46.5
0.1981	1.02848	43.6	1.02046	44.6	1.00785	45.6	0.99794	46.6	0.99047	46.2
0.2967	1.03404	43.6	1.02598	44.4	1.01332	45.2	1.00327	46.3	0.99585	45.8
0.3957	1.03960	43.6	1.03146	44.3	1.01892	44.6	1.00863	46.0	1.00125	45.5
0.4946	1.04518	43.4	1.03703	44.0	1.02436	44.5	1.01402	45.7	1.00672	45.1
0.5935	1.05115	42.6	1.04261	43.7	1.03011	43.8	1.01924	45.6	1.01222	44.7

Table 4. Density and apparent molal volume of KNO_3 in water-acetamide mixture containing 50 wt % of $AcNH_2$.

m	25°C		40°C		60°C		75°C		85°C	
	d	ϕ_v	d	ϕ_v	d	ϕ_v	d	ϕ_v	d	ϕ_v
[mole/kg]	[g/cm^3]	[cm^3/mole]	[g/cm^3]	[cm^3/mole]	[g/cm^3]	[cm^3/mole]	[g/cm^3]	[cm^3/mole]	[g/cm^3]	[cm^3/mole]
0.0200	1.03131	47.7	1.02100	48.6	1.00674	49.0	0.99535	49.1	0.98721	48.5
0.0353	1.03214	47.4	1.02182	48.1	1.00754	48.9	0.99615	48.9	0.98801	48.5
0.0499	1.03293	47.2	1.02260	47.9	1.00831	48.7	0.99693	48.4	0.98879	48.1
0.0693	1.03398	47.1	1.02364	47.7	1.00934	48.5	0.99796	48.2	0.98983	47.8
0.0994	1.03561	47.0	1.02526	47.5	1.01094	48.1	0.99956	48.0	0.99143	47.7
0.1979	1.04099	46.4	1.03062	46.8	1.01619	47.6	1.00482	47.5	0.99677	46.9
0.2975	1.04640	46.2	1.03610	46.2	1.02162	46.9	1.01018	47.0	1.00219	46.3
0.3962	1.05187	45.7	1.04154	45.8	1.02699	46.4	1.01558	46.4	1.00764	45.8
0.4952	1.05733	45.4	1.04698	45.4	1.03236	46.1	1.02096	46.0	1.01306	45.4
0.5935	1.06261	45.3	1.05234	45.2	1.03783	45.5	1.02640	45.5	1.01847	45.1

Table 5. Density and apparent molal volume of KNO_3 in water-acetamide mixture containing 70 wt % $AcNH_2$.

m	40°C		60°C		75°C		85°C	
	d	Φ_V	d	Φ_V	d	Φ_V	d	Φ_V
[mole/kg]	[g/cm^3]	[cm^3/mole]	[g/cm^3]	[cm^3/mole]	[g/cm^3]	[cm^3/mole]	[g/cm^3]	[cm^3/mole]
0.0205	1.02808	50.3	1.01267	50.3	1.00065	49.8	0.99176	49.3
0.0410	1.02913	50.0	1.01372	50.0	1.00171	49.5	0.99284	48.8
0.0786	1.03110	49.3	1.01567	49.5	1.00368	49.0	0.99484	48.2
0.1003	1.03222	49.2	1.01682	49.2	1.00483	48.7	0.99599	48.0
0.2000	1.03747	48.5	1.02204	48.6	1.01008	48.2	1.00137	47.2
0.3000	1.04276	48.0	1.02733	48.0	1.01542	47.6	1.00684	46.5
0.3977	1.04800	47.5	1.03262	47.4	1.02068	47.1	1.01224	45.9
0.4963	1.05330	47.1	1.03778	47.2	1.02608	46.5	1.01758	45.6
0.5936	1.05857	46.7	1.04302	46.8	1.03136	46.1	1.02301	45.1
0.6925	1.06390	46.3	1.04854	46.2	1.03656	46.0	1.02841	44.9

to

$$\phi_v = \frac{1000(d_o - d)}{mdd_o} + \frac{M}{d} \qquad (2)$$

where d_o - the density of the solvent
d - the density of the solution
m - the concentration of the solution
M - the molecular weight of the electrolyte.

The values ϕ_v calculated for all the examined solutions are given in tab. 1-5. In the case of diluted solutions ϕ_v is given by Masson's equation

$$\phi_v = \phi_v^o + A\sqrt{c} \qquad (3)$$

By extrapolation of the equation (3) up to $c = 0$ the values of ϕ_v^o were obtained which corresponds to the partial molal volume of electrolyte at infinite dilution ($\bar{V}_2^o$). The values of $\bar{V}^o_{KNO_3}$ in water - acetamide mixed solvents are presented in tab. 6. and shown in fig. 1 as a function of the composition of mixed solvent and on temperature of the solution.

Table 6. Partial molal volume of KNO_3 in water-acetamide solutions within the temperature range 25-85°C [cm^3/mole].

wt % of $AcNH_2$ \ T °C	25	40	60	75	85
water	38.0	39.3	40.4	41.0	41.1
5(1.58 mol %)	39.0	40.4	42.0	42.7	42.7
15(5.10 mol %)	41.0	42.4	44.6	45.2	45.0
30(11.55 mol %)	44.0	45.6	47.0	48.0	47.7
50(23.36 mol %)	48.0	49.0	49.9	49.8	49.4
70(41.55 mol %)	-	50.9	51.2	50.6	50.0

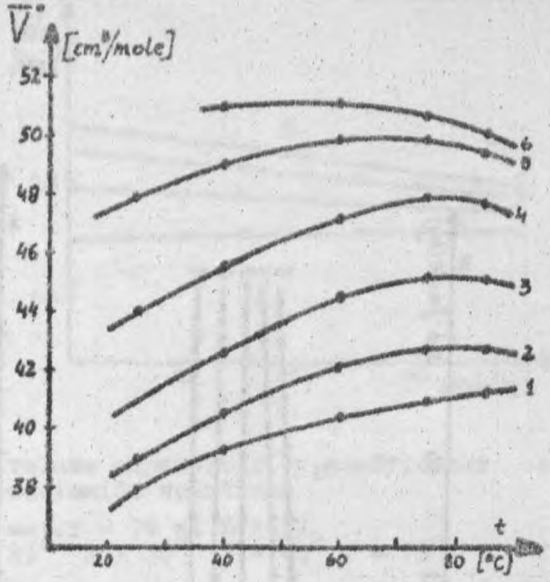

Fig. 1. The partial molal volume $\bar{V}_2^o$ of KNO_3 in water-acetamide solutions 1 - water, 2 - water- 5 wt % (1.58 mol %)$AcNH_2$, 3 - water - 15 wt %(5.10 mol %) $AcNH_2$, 4 - water - 30 wt % (11.55 mol %) $AcNH_2$, 5 - water - 50 wt % (23.36 mol %) $AcNH_2$, 6 - water - 70 wt %(41.55 mol %) $AcNH_2$.

The measurements of density carried out within the larger temperature range enable to calculate the values of the coefficients of the equation

$$d_{x,m} = a + bT + cT^2 \qquad (4)$$

describing the dependence of density of the examined solutions on the temperature. If we know the values of the coefficients a, b and c of the eq. 4 we can calculate the derivative $[\partial d/\partial T]_{x,m}$ and then the coefficient of volume expansion α of the examined solutions from formula

$$\alpha = -\frac{1}{d} \Big/ \frac{\partial d}{\partial T} \Big/_{x,m} \qquad (5)$$

The values of coefficient of volume expansion α of H_2O - $AcNH_2$-KNO_3 systems are presented on fig. 2.

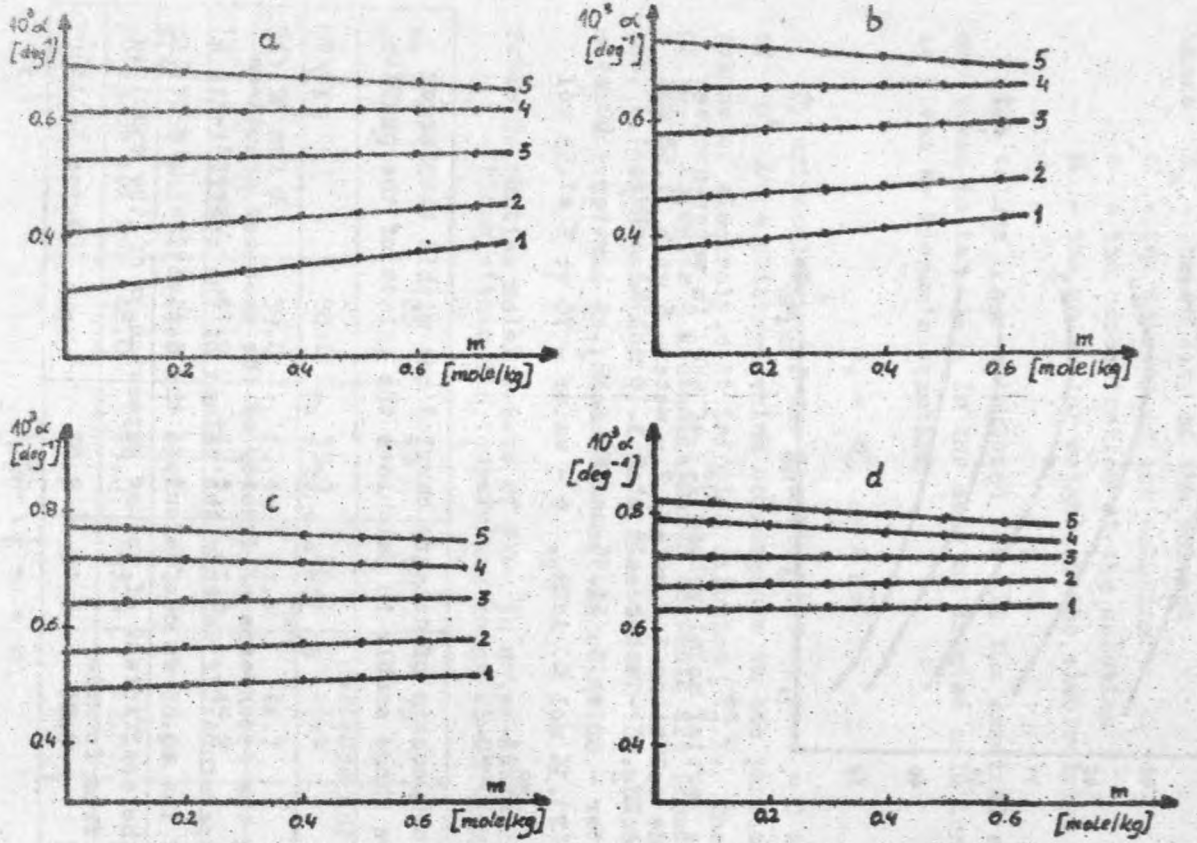

Fig. 2. The volume expansibility coefficient of KNO_3 in water-acetamide solutions
a - water - 5 wt % $AcNH_2$ b - water - 15 wt % $AcNH_2$ c - water - 30 wt % $AcNH_2$
d - water - 50 wt % $AcNH_2$ 1-25, 2-40, 3-60, 4-75, 5-85°C

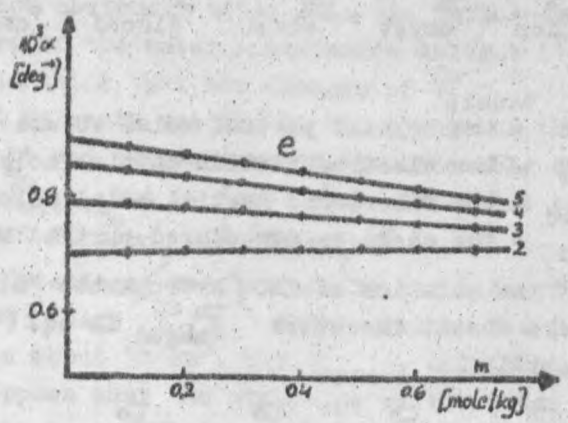

Fig. 2. The volume expansibility coefficient of KNO_3 in water-acetamide solutions

e - water - 70 wt % $AcNH_2$
1 - 25° 2 - 40° 3 - 60° 4 - 75° 5 - 85°C.

Discussion

As it can be seen from fig. 1 the partial molal volume of KNO_3 in the water - acetamide mixed solvents rises with the increase of acetamide concentration in the mixed solvent. The values of $\bar{V}^o_{KNO_3}$ increase as well with the increase of temperature and at certain temperature the value of $\bar{V}^o_{KNO_3}$ passes a maximum and then decreases. The temperature maximum T_{max} is highest in case of solutions KNO_3 in water (about 100°C [14]) and decreases with the increase of acetamide concentration in the mixed solvent. In case of the solvent containing 70 wt % (41.55 mol %) of $AcNH_2$ the temperature max. is ~55°C. Presented here character of changes of $V^o_{KNO_3}$ as a function of concentration of acetamide and temperature can be explained with the multilayer hydration model used by G u r n e y [27] and F r a n k - W e n [28]. Using this model the partial molal volume of an ion at infinite dilution $\bar{V}^o_{ion}$ can be dissected in to following components:

$$\bar{V}^{o}_{ion} = \bar{V}^{o}_{cryst} + \bar{V}^{o}_{elect} + \bar{V}^{o}_{disord} + \bar{V}^{o}_{caged} \qquad (6)$$

where $\bar{V}^{o}_{cryst}$ - the crystal partial molal volume
$\bar{V}^{o}_{elect}$ - the electrostriction partial molal volume
$\bar{V}^{o}_{disord}$ - the disordered partial molal volume
$\bar{V}^{o}_{caged}$ - the caged or structured partial molal volume

In case of the solution of KNO_3 hydrophobic "Structure - making" ions are absent therefore $\bar{V}^{o}_{caged}$ in eq. (6) can be omitted. We obtain

$$\bar{V}^{o}_{ion} = \bar{V}^{o}_{cryst} + \bar{V}^{o}_{elect} + \bar{V}^{o}_{disord} \qquad (7)$$

Since $\bar{V}^{o}_{cryst}$ can be assumed to be a constant and equal to the volume of the ion in the crystal [27-31] so the changes of $\bar{V}^{o}_{ion}$ are caused by the changes of both terms $\bar{V}^{o}_{elect}$ and $\bar{V}^{o}_{disord}$.

From the theory [31, 32] it is known that $\bar{V}^{o}_{elect}$ is a function of the electric permeability of the solvent and passes a maximum as a function of temperature [33,34]. The temperature of the maximum value of $\bar{V}^{o}_{elect}$ (T_{max})is a function of the field strength of ion. For ions exerting high field strength on the solvation cosphere the maximum appears at lower temperature than for ions exerting weaker field strength. According to R o h d e w a l d and M o l d n e r [35] the electric permeability of the water - acetamide mixed solvent increases attaing the maximum value in the solution containing about 40 mol % of acetamide and then it decreases. On the other hand if we compare the values $\bar{V}^{o}_{2}$ of electrolytes in various solvents we infer that the values of $\bar{V}^{o}_{ion}$ rise with the increase of the electric permeability of the solvent. In case of the solvents which have a similar electric permeability the values $\bar{V}^{o}_{ion}$ are higher in the solvents with the strong association [22].

From these considerations it follows that the increase of $\bar{V}^{o}_{KNO_3}$ in the investigated water - acetamide solvents is probably due to the increase of the electric permeability of solvent for the course of the changes of the function $\bar{V}^{o}_{KNO_3} = f(x_{AcNH_2}, T)$ is similar to the changes of function

$\bar{V}^o_{elect}$ = F(electric permeability, temperature).

We can not obviously total omit the influence of KNO_3 on the structure of the water - acetamide solvent ($\bar{V}^o_{disord}$). However one can think that the changes of $\bar{V}^o_{disord}$ of KNO_3 in the examined solvents are due to the change of the composition of the solvent and temperature are considerably smaller than the changes $\bar{V}^o_{elect}$ and it does not decide about the course of the dependence of $\bar{V}^o_{KNO_3} = f(x_{AcNH_2}, T)$.

Similar dependence can be seen in the case of the aqueous solutions [36] where $\bar{V}^o_{elect}$ for ion with $r = 1$ Å from 0 to 200°C changes about 18 cm^3, but $\bar{V}^o_{disord}$ only about 2 cm^3/mol. So we can suppose that the structure of the water - acetamide mixed solvents is not fundamentally different in comparison with the structure of water. It is confirmed by the papers [18-24] from which follows that in the water - acetamide mixed solvents the molecules of acetamide form hydrogen bonds with water molecules into three - dimentional network.

In order to get additional information about KNO_3 effect on water - acetamide solvent structure the values of the difference between the $\bar{V}^o_{KNO_3} - \bar{V}^o_{cryst}$ were calculated (Tab. 7). As it can be seen from the eq. (7) the positive value of the difference $\bar{V}^o_{ion} - \bar{V}^o_{cryst}$ proves that $\bar{V}^o_{disord} > -\bar{V}^o_{elect}$ or the volume of the disordered region in the solvent around the solvated ion is larger than region of the electrostriction. In such case we can assume that the ions break the original structure of the solvent ("Structure-breaking" ions or negative hydrating ions). As it is seen from tab. 7 the values of the difference $\bar{V}^o_{KNO_3} - \bar{V}^o_{cryst} = \bar{V}^o_{disord} - \bar{V}^o_{elect}$ in the water - acetamide mixed solvents are positive. It proves that KNO_3 breakes the structure of water - acetamide mixed solvents. It can be added that the breaking effect of KNO_3 rises with the increase of the concentration of acetamide in the mixed solvent and the increase of temperature untill the temperature reaches the value T_{max} then begins to decreases. The intensity of interaction of KNO_3 in the solvents containing more acetamide can be explained by the breaking of the larger quantity of weaker hydrogen bonds by electrolyte. The increase of temperature causes probably the

Table 7. The difference $\bar{V}^{o}_{ion} - \bar{V}^{o}_{cryst.}$ of KNO_3 in water - acetamide solutions within the temperature range 25-85°C [cm^3/mole].

T °C	25	40	60	75	85	T_{max}
Water	11.0	12.3	13.4	14.0	14.1	~100 [36]
1.58 mol % $AcNH_2$	12.0	13.4	15.0	15.7	15.7	~ 80
5.10 mil % $AcNH_2$	14.0	15.4	17.6	18.2	18.0	~ 75
11.55 mol % $AcNH_2$	17.0	18.6	20.0	21.0	20.7	~ 70
23.36 mol % $AcNH_2$	21.0	22.0	22.9	22.8	22.4	~ 60
41.55 mol % $AcNH_2$	-	23.9	24.2	23.6	23.0	~ 55

weakening of hydrogen bonds because of more intensive thermal motions of the molecules. This effect makes easier the breaking of weaker hydrogen bonds by KNO_3. The disordering effect of KNO_3 begins to decrease above the temperature T_{max} (see tab. 7).

The conclusions obtained from the analysis of the dependence $\bar{V}^{o}_{KNO_3} = f(x_{AcNH_2}, T)$ in the mixed solvent can be confirmed by the course of the dependence of volume expansion coefficient α of investigated systems as a function of the composition of the mixed solvent and temperature. As it is seen from Fig 2 the values of the coefficient α rise with the increase of the concentration of electrolyte in the mixed solvent untill a certain transitional temperature T_p above which a function $\alpha = f(c)$ decreases. The value of the transitional temperature T_p depends on concentration of acetamide in the mixed solvent and as can be seen from fig. 2 it decreases with the increase of the mole fraction of $AcNH_2$ in the mixed solvent. It is easy to notice the

convergence of the T_p and T_{max}. The changes of the coefficient α described here according with the conclusions from papers [37-39] proves that the growth of the coefficient α with the increase of concentration of the electrolyte in solution suggest the breaking effect of electrolyte on the structure of the solvent and vice versa. It appears that KNO_3 disorders the the structure of water - acetamide mixed solvent only below the temperature T_p but above this temperature it begins to reveal ordering effect of the hydration of ions. The increase of the coefficient α with the increase of concentration of acetamide in solution proves that the solution expands more easily. Thus it can be assumed that the hydrogen bonds in the water - acetamide mixed solvents are weaker than the hydrogen bonds in the water. This opinion is in agreement with the conclusion following from the papers of G o n c h a r o v et al. [23-24].

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DENSYMETRYCZNE BADANIA UKŁADU H_2O - $AcNH_2$ - KNO_3 W ZAKRESIE TEMPERATURY 25 - 85°C

Zmierzono gęstość roztworów KNO_3 w rozpuszczalnikach mieszanych woda - acetamid w zakresie temperatury 25-85°C. Wykorzystując otrzymane wartości gęstości obliczono pozorną molową objętość ϕ_v elektrolitu i współczynnik rozszerzalności objętościowej α. Poprzez ekstrapolację wartości ϕ_v do rozcieńczenia nieskończenie wielkiego otrzymano wartości cząstkowej molowej objętości $\overline{V}^o_{KNO_3}$ w badanych rozpuszczalnikach.

Przedyskutowano zależność $\bar{V}^{o}_{KNO_3}$ i współczynnika α w funkcji stężenia roztworu, składu rozpuszczalnika mieszanego i jego temperatury. Wysnuto wniosek odnośnie wpływu KNO_3 na strukturę badanych rozpuszczalników wodno-acetamidowych.