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Małgorzata Jóźwiak*

VISCOSIMETRIC INVESTIGATIONS OF NaCl AND NaBr IN HEXAMETHYLPHOSPHORAMIDE-WATER MIXTURES AT 298.15 K**

The maesurements of density and viscosity of hexamethylphosphoramide (HMPA)-water mixtures containing NaCl and NaBr were performed at 298.15 K. The B-coefficients of the Jones-Dole equation were calculated. The relative viscosity and the B-coefficient are discussed in this work together with the standard enthalpy of solution of the electrolytes in HMPA-H_2O mixtures at 298.15 K.

INTRODUCTION

In our previous papers [1, 2, 3] there were presented the measurements of the enthalpies of solution of NaI, KI, CsI, NaCl, KCl, Ph_4PCl and $NaBPh_4$ in HMPA-water mixtures. Different positions of the ΔH_s^o maxima for the salts containing various anions were observed.

In the other paper [4] the measurements of density and viscosity of HMPA-water mixtures and of solution of NaI in these mixtures were described at 293.15, 298.15, and 303.15 K. We suggested that the maximum of the hydrophobic hydration of HMPA molecules occurs at $x_2 = 0.05$ and the maximum ΔH_s^o is observed also for organic substances disolved in HMPA-H_2O mixtures [2]. The ΔH_s^o of inorganic salts also exhibit extrema corresponding to the different HMPA con-

*Department of Physical Chemistry, University of Łódź.

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tents in the binary solvent. The relative viscosity of NaI exhibit minimum in the same range of HMPA content as ΔH_s^o does.

In order to find out whether in the case of other electrolytes there is also a similarity of positions of ΔH_s^o and η_{rel} extrema we measured in this work density and viscosity of HMPA-water-NaCl and HMPA-water-NaBr systems.

EXPERIMENTAL

"Purum" hexamethylphosphoramide (Fluka) was purified as described previously [2].

Sodium chloride ("puriss" P.O.Ch. Gliwice) was crystallized from water and dried at 373 K. Its purity was tested by the potentiometric method and the chloride content was found to be 99.99 per cent.

Sodium bromide ("puriss" P.O.Ch. Gliwice) was crystallized from water-alcohol mixture and dried at 333 K under a reduced pressure. Its purity was tested by the argentometric method and the bromide content was found to be 99.9 per cent.

The density was measured using Lipkin's picnometers of approximately 14 cm^3 capacity.

The viscosity measurements were carried out using Ubbelohde's viscosimeters with the efflux time of the investigated solutions from 200 to 400 s. The efflux time was measured with accuracy to $\pm$ 0.1 s. The density and viscosity were measured at 298.15 K. The viscometers and the picnometers were thermostated with precision to $\pm$ 0.01°. They were calibrated by double-distilled water. The maximum value of the mean square error of the density measurements does not exceed $1 \cdot 10^{-4}$ g · cm^{-3} and of the viscosity measurements $5 \cdot 10^{-4}$ cP.

The density and the viscosity were measured in the molality m the range from 0.005 to 0.0501 mol · kg^{-1} for NaBr and 0.0021 to 0.0806 mol · kg^{-1} for NaCl.

The mole fraction (x_2) HMPA was 0 to 1 for NaBr and 0 to 0.2 for NaCl. At higher values of x_2 the density and the viscosity of NaCl in HMPA-H_2O mixtures could not be measured in view of the limited solubility of the electrolyte.

RESULTS

The desities (d_1) of NaBr in HMPA-water mixtures and NaCl in HMPA-water mixtures are presented in Tab. 1 and 2. As it can be seen from tables, the d_1 values reach maxima at the binary solvent composition, x_2 = 0.15. The maximum density of HMPA-H_2O mixtures [4] without an electrolyte corresponds also to this region (x_2 = = 0.15), indicating probably the highest molecule packing.

Table 1

Densities, d_1, of NaBr in HMPA-H_2O mixtures at 298.15 K

$\frac{m}{mol \cdot kg^{-1}}$	$\frac{d_1}{g \cdot cm^{-3}}$	$\frac{m}{mol \cdot kg^{-1}}$	$\frac{d_1}{g \cdot cm^{-3}}$
1	2	3	4
x_2 = 0.00		x_2 = 0.35	
0.0050	0.9975	0.0050	1.0387
0.0071	0.9976	0.0071	1.0389
0.0099	0.9979	0.0099	1.0391
0.0100	0.9979	0.0100	1.0391
0.0200	0.9986	0.0200	1.0398
0.0350	0.9998	0.0350	1.0409
0.0501	1.0010	0.0501	1.0420
x_2 = 0.05		x_2 = 0.45	
0.0050	1.0299	0.0050	1.0342
0.0071	1.0301	0.0071	1.0344
0.0099	1.0303	0.0099	1.0346
0.0100	1.0303	0.0100	1.0346
0.0200	1.0311	0.0200	1.0354
0.0350	1.0323	0.0350	1.0365
0.0501	1.0334	0.0501	1.0377
x_2 = 0.15		x_2 = 0.55	
0.0050	1.0466	0.0050	1.0307
0.0071	1.0467	0.0071	1.0309
0.0099	1.0469	0.0099	1.0311
0.0100	1.0470	0.0100	1.0311
0.0200	1.0477	0.0200	1.0319
0.0350	1.0488	0.0350	1.0331
0.0501	1.0499	0.0501	1.0343
x_2 = 0.25 HMPA		x_2 = 0.65	
0.0050	1.0441	0.0050	1.0283
0.0071	1.0442	0.0071	1.0285
0.0099	1.0444	0.0099	1.0287

Table 1 (contd)

1	2	3	4
x_2 = 0.25 HMPA		x_2 = 0.65	
0.0100	1.0444	0.0100	1.0287
0.0200	1.0452	0.0200	1.0295
0.0350	1.0463	0.0350	1.0307
0.0501	1.0474	0.0501	1.0318
x_2 = 0.75		x_2 = 0.95	
0.0050	1.0255	0.0050	1.0216
0.0071	1.0257	0.0071	1.0218
0.0099	1.0259	0.0099	1.0220
0.0100	1.0259	0.0100	1.0220
0.0200	1.0267	0.0200	1.0229
0.0350	1.0279	0.0350	1.0242
0.0501	1.0291	0.0501	1.0255
x_2 = 0.85		x_2 = 1.0	
0.0050	1.0232	0.0050	1.0206
0.0071	1.0234	0.0071	1.0207
0.0099	1.0236	0.0099	1.0210
0.0100	1.0236	0.0100	1.0210
0.0200	1.0245	0.0200	1.0219
0.0350	1.0257	0.0350	1.0232
0.0501	1.0270	0.0501	1.0245

Table 2

Densities, d_1, of NaCl in HMPA-H_2O mixtures at 298.15 K

$\frac{m}{kg \cdot mol^{-1}}$	$\frac{d_1}{g \cdot cm^{-3}}$	$\frac{m}{kg \cdot mol^{-1}}$	$\frac{d_1}{g \cdot cm^{-3}}$
1	2	3	4
x_2 = 0.00		x_2 = 0.15	
0.0021	0.9971	0.0021	1.0463
0.0065	0.9973	0.0065	1.0464
0.0100	0.9975	0.0100	1.0466
0.0160	0.9978	0.0160	1.0468
0.0224	0.9981	0.0224	1.0470
0.0500	0.9993	0.0500	1.0480
0.0806	1.0007	0.0806	1.0491

Table 2 (contd)

1	2	3	4
x_2 = 0.05		x_2 = 0.20	
0.0021	1.0296	0.0021	1.0460
0.0065	1.0298	0.0065	1.0461
0.0100	1.0300	0.0100	1.0462
0.0160	1.0302	0.0160	1.0464
0.0224	1.0305	0.0224	1.0465
0.0500	1.0316	0.0500	1.0472
0.0806	1.0329	0.0806	1.0480
x_2 = 0.10			
0.0021	1.0432		
0.0065	1.0434		
0.0100	1.0435		
0.0160	1.0437		
0.0224	1.0440		
0.0500	1.0450		
0.0806	1.0462		

K e s s l e r et al., [5] on the basis of relaxation time determinations and electric permittivity measurements of the HMPA-H_2O mixture, suggest the most stable structure for the system to be at x_2 = 0.15. In this mixture these are still numerous hydrogen bonds between water molecules which together with HMPA give a more rigid system.

The viscosity of NaBr and NaCl in HMPA-water mixtures is listed in Tab. 3 and 4. The function η (NaBr-HMPA-H_2O) = f(x_2) exhibits a maximum in the vicinity of x_2 = 0.20 similar to that for ΔH^E, η_o of HMPA-H_2O, V_ϕ(NaI) and $\Delta\eta_{rel}/\Delta T$ for NaI in HMPA-H_2O mixtures [3]. The extremum of viscosity for NaCl in HMPA-H_2O was not observed, because of the limited solubility of NaCl.

The concentration dependence of the viscosity of electrolyte solutions can be described by Jones-Dole semi-empirical equation [6].

$$\eta_{rel} = 1 + A \cdot c^{1/2} + B \cdot c$$

where:

η_{rel} - the relative viscosity of solution,
c - the molar concentration of electrolyte,
A - the coefficient for ion-ion electrostatic interaction,

B - the coefficient denoting an empirical constant connected with ion-solvent and solvent-solvent interactions.

Table 3

Viscosities, η_1, of NaBr in HMPA-H_2O mixtures at 298.15 K

$\frac{m}{mol \cdot kg^{-1}}$	$\frac{\eta_1}{cP}$	$\frac{m}{mol \cdot kg^{-1}}$	$\frac{\eta_1}{cP}$
1	2	3	4
x_2 = 0.00		x_2 = 0.35	
0.0050	0.8909	0.0050	6.7589
0.0071	0.8910	0.0071	6.7681
0.0099	0.8912	0.0099	6.7797
0.0100	0.8912	0.0100	6.7803
0.0200	0.8919	0.0200	6.8216
0.0350	0.8927	0.0350	6.8817
0.0501	0.8935	0.0501	6.9417
x_2 = 0.05		x_2 = 0.45	
0.0050	3.5518	0.0050	5.8128
0.0071	3.5524	0.0071	5.8241
0.0099	3.5532	0.0099	5.8383
0.0100	3.5533	0.0100	5.8390
0.0200	3.5559	0.0200	5.8901
0.0350	3.5595	0.0350	5.9646
0.0501	3.5630	0.0501	6.0391
x_2 = 0.15		x_2 = 0.55	
0.0050	7.8447	0.0050	5.1129
0.0071	7.8486	0.0071	5.1248
0.0099	7.8535	0.0099	5.1400
0.0100	7.8538	0.0100	5.1408
0.0200	7.8713	0.0200	5.1952
0.0350	7.8966	0.0350	5.2743
0.0501	7.9217	0.0501	5.3538
x_2 = 0.25		x_2 = 0.65	
0.0050	7.9879	0.0050	4.5191
0.0071	7.9948	0.0071	4.5318
0.0099	8.0037	0.0099	4.5479
0.0100	8.0037	0.0100	4.5488
0.0200	8.0346	0.0200	4.6070
0.0350	8.0791	0.0350	4.6924
0.0501	8.1235	0.0501	4.7780

Table 3 (contd)

1	2	3	4
x_2 = 0.75		x_2 = 0.95	
0.0050	4.0953	0.0050	3.3928
0.0071	4.1085	0.0071	3.4068
0.0099	4.1251	0.0099	3.4245
0.0100	4.1259	0.0100	3.4253
0.0200	4.1859	0.0200	3.4894
0.0350	4.2743	0.0350	3.5840
0.0501	4.3628	0.0501	3.6789
x_2 = 0.85		x_2 = 1.00	
0.0050	3.7190	0.0050	3.2449
0.0071	3.7326	0.0071	3.2588
0.0099	3.7498	0.0099	3.2766
0.0100	3.7507	0.0100	3.2775
0.0200	3.8131	0.0200	3.3420
0.0350	3.9050	0.0350	3.4370
0.0501	3.9973	0.0501	3.5325

Table 4

Viscosities, η_1, of NaCl in HMPA-H_2O mixtures at 298.15 K

$\frac{m}{mol \cdot kg^{-1}}$	$\frac{\eta_1}{cP}$	$\frac{m}{mol \cdot kg^{-1}}$	$\frac{\eta_1}{cP}$
x_2 = 0.00		x_2 = 0.15	
0.0021	0.8907	0.0021	7.8386
0.0065	0.8912	0.0065	7.8459
0.0100	0.8915	0.0100	7.8516
0.0160	0.8921	0.0160	7.8607
0.0224	0.8927	0.0224	7.8702
0.0500	0.8950	0.0500	7.9100
0.0806	0.8975	0.0806	7.9529
x_2 = 0.05		x_2 = 0.20	
0.0021	3.5511	0.0021	8.3210
0.0065	3.5531	0.0065	8.3323
0.0100	3.5546	0.0100	8.3408
0.0160	3.5570	0.0160	8.3549
0.0224	3.5594	0.0224	8.3650
0.0500	3.5696	0.0500	8.4310
0.0806	3.5804	0.0806	8.4977

Table 4 (contd)

1	2	3	4
x_2 = 0.10			
0.0021	6.2647		
0.0065	6.2687		
0.0100	6.2715		
0.0160	6.2762		
0.0224	6.2810		
0.0500	6.3007		
0.0806	6.3218		

The A coefficient is a measure of long-range coulombic forces, i.e. ion-ion interactions, and it can be calculated by the F a l k e n h a g e n equation [7, 8]

$$A = \frac{0.2577\Lambda_o}{\eta(\varepsilon T)^{1/2}\Lambda_o^+ \Lambda_o^-} \cdot \left[1 - 0.6863\left(\frac{\Lambda_o^+ - \Lambda_o^-}{\Lambda_o}\right)^2\right]$$

where:

Λ_o - the limiting equivalent conductance of the salt,

Λ_o^+ and Λ_o^- - the limiting equivalent conductance of the ions,

ε - the electric permittivity of the solvent,

η_o - the viscosity of the solvent,

T - the temperature.

The values of physical constants and of limiting equivalent conductances were reported by G a l et al. [9]. The coefficients A for HMPA-H_2O-NaBr and HMPA-H_2O-NaCl systems used by us are shown in Tab. 5.

T a b l e 5

Theoretical A-coefficients in HMPA-water at 298.15 K

x_2	NaBr	x_2	NaCl
	A / $dm^{3/2}mol^{-1/2}$		A / $dm^{3/2}mol^{-1/2}$
1	2	3	4
0.00	0.0059	0.00	0.0061
0.05	0.0050	0.05	0.0050
0.15	0.0059	0.10	0.0055

Table 5 (contd)

1	2	3	4
0.25	0.0094	0.15	0.0061
0.35	0.0119	0.20	0.0081
0.45	0.0145		
0.55	0.0154		
0.65	0.0159		
0.75	0.0159		
0.85	0.0159		
0.95	0.0160		
1.00	0.0161		

Table 6

Viscosity B-coefficients of NaCl in HMPA-water mixtures at 298.15 K

$\frac{m}{mol \cdot kg^{-1}}$	$\frac{c}{mol \cdot l^{-1}}$	η_{rel}	$\frac{\bar{B}}{dm^3 \cdot mol^{-1}}$
1	2	3	4
$x_2 = 0.00$			
0.0021	0.0021	1.0004	
0.0065	0.0065	1.0010	
0.0100	0.0100	1.0013	
0.0160	0.0160	1.0020	0.075
0.0224	0.0223	1.0027	
0.0500	0.0493	1.0053	
0.0806	0.0803	1.0081	
$x_2 = 0.05$			
0.0021	0.0022	1.0004	
0.0065	0.0067	1.0010	
0.0100	0.0103	1.0014	
0.0160	0.0165	1.0021	0.086
0.0224	0.0230	1.0028	
0.0500	0.0514	1.0056	
0.0806	0.0829	1.0087	
$x_2 = 0.10$			
0.0021	0.0022	1.0005	
0.0065	0.0068	1.0011	
0.0100	0.0104	1.0015	
0.0160	0.0167	1.0023	0.097
0.0224	0.0233	1.0031	
0.0500	0.0521	1.0062	
0.0806	0.0840	1.0096	

Table 6 (contd)

1	2	3	4
x_2 = 0.15			
0.0021	0.0022	1.0006	
0.0065	0.0067	1.0016	
0.0100	0.0105	1.0023	
0.0160	0.0167	1.0035	0.159
0.0224	0.0234	1.0047	
0.0500	0.0523	1.0098	
0.0806	0.0842	1.0152	
x_2 = 0.20			
0.0021	0.0022	1.0009	
0.0065	0.0068	1.0023	
0.0100	0.0105	1.0033	
0.0160	0.0167	1.0050	0.234
0.0224	0.0234	1.0062	
0.0500	0.0522	1.0141	
0.0806	0.0841	1.0222	

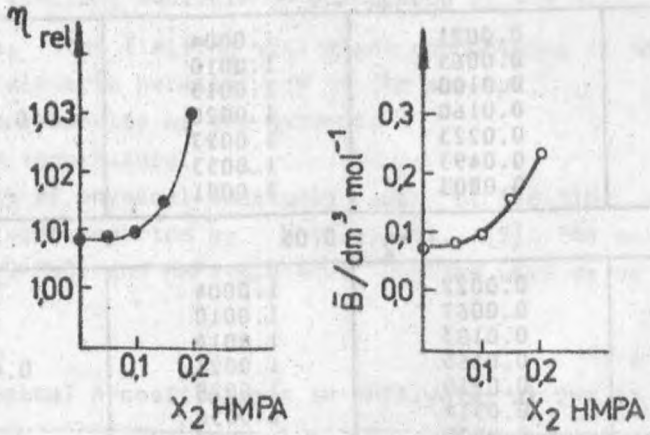

Fig. 1. Relative viscosities (•) and the B-coefficients (o) of NaCl solutions in water-HMPA mixtures at 298.15 K

The relative viscosity and the B-coefficient are presented in Tab. 6 and in Fig. 1 for HMPA-H_2O-NaCl system, in Tab. 7 and Fig. 2 for HMPA-H_2O-NaBr. The values of B-coefficient obtained in this work are in satisfactory agreement with those from the literature for NaCl at x_2 = 0.05, 0.1 and 0.2, for NaBr at x_2 = 0.05 [10].

Table 7

Viscosity B-coefficients of NaBr in HMPA-water mixtures at 298.15 K

$\frac{m}{mol \cdot kg^{-1}}$	$\frac{c}{mol \cdot l^{-1}}$	η_{rel}	$\frac{\bar{B}}{dm^3 \cdot mol^{-1}}$
1	2	3	4
$x_2 = 0.00$			
0.0050	0.0050	1.0007	
0.0071	0.0071	1.0008	
0.0099	0.0098	1.0010	
0.0100	0.0100	1.0010	0.046
0.0200	0.0200	1.0018	
0.0350	0.0349	1.0027	
0.0501	0.0499	1.0036	
$x_2 = 0.05$			
0.0050	0.0051	1.0006	
0.0071	0.0073	1.0008	
0.0099	0.0102	1.0010	
0.0100	0.0103	1.0010	0.051
0.0200	0.0206	1.0018	
0.0350	0.0361	1.0028	
0.0501	0.0516	1.0038	
$x_2 = 0.15$			
0.0050	0.0052	1.0014	
0.0071	0.0075	1.0019	
0.0099	0.0103	1.0025	
0.0100	0.0105	1.0026	0.188
0.0200	0.0210	1.0048	
0.0350	0.0366	1.0080	
0.0501	0.0523	1.0113	
$x_2 = 0.25$			
0.0050	0.0052	1.0024	
0.0071	0.0074	1.0033	
0.0099	0.0103	1.0044	
0.0100	0.0104	1.0044	0.332
0.0200	0.0209	1.0083	
0.0350	0.0365	1.0139	
0.0501	0.0522	1.0194	

Table 7 (contd)

1	2	3	4
		x_2 = 0.35	
0.0050	0.0052	1.0037	
0.0071	0.0074	1.0050	
0.0099	0.0102	1.0067	
0.0100	0.0104	1.0068	0.540
0.0200	0.0208	1.0130	
0.0350	0.0363	1.0219	
0.0501	0.0519	1.0308	
		x_2 = 0.45	
0.0050	0.0052	1.0051	
0.0071	0.0074	1.0071	
0.0099	0.0102	1.0095	
0.0100	0.0103	1.0096	0.789
0.0200	0.0207	1.0185	
0.0350	0.0362	1.0314	
0.0501	0.0517	1.0442	
		x_2 = 0.55	
0.0050	0.0051	1.0061	
0.0071	0.0073	1.0084	
0.0099	0.0102	1.0114	
0.0100	0.0103	1.0116	0.965
0.0200	0.0206	1.0213	
0.0350	0.0360	1.0378	
0.0501	0.0516	1.0535	
		x_2 = 0.65	
0.0050	0.0051	1.0073	
0.0071	0.0073	1.0101	
0.0099	0.0101	1.0137	
0.0100	0.0103	1.0139	1.197
0.0200	0.0206	1.0269	
0.0350	0.0359	1.0459	
0.0501	0.0514	1.0650	
		x_2 = 0.75	
0.0050	0.0051	1.0081	
0.0071	0.0073	1.0114	
0.0099	0.0101	1.0155	
0.0100	0.0103	1.0157	1.372
0.0200	0.0205	1.0305	
0.0350	0.0359	1.0522	
0.0501	0.0513	1.0740	

Table 7 (contd)

1	2	3	4
x_2 = 0.85			
0.0050	0.0051	1.0092	
0.0071	0.0073	1.0129	
0.0099	0.0101	1.0176	
0.0100	0.0102	1.0178	1.584
0.0200	0.0205	1.0348	
0.0350	0.0358	1.0597	
0.0501	0.0512	1.0848	
x_2 = 0.95			
0.0050	0.0051	1.0103	
0.0071	0.0073	1.0144	
0.0099	0.0101	1.0197	
0.0100	0.0102	1.0200	1.796
0.0200	0.0205	1.0390	
0.0350	0.0357	1.0672	
0.0501	0.0511	1.0955	
x_2 = 1.00			
0.0050	0.0051	1.0108	
0.0071	0.0073	1.0151	
0.0099	0.0101	1.0207	
0.0100	0.0102	1.0210	1.893
0.0200	0.0204	1.0411	
0.0350	0.0357	1.0706	
0.0501	0.0511	1.1004	

The analysis of the η_{rel} and B-coefficients of the salts studied in the water-HMPA mixtures provides a few interesting results. It can be noted that at 298.15 K, the η_{rel} and B-coefficient, in passing from water to x_2 = 0.05 for NaBr and x_2 = 0.10 for NaCl increase very slowly, and then the increase is much more intensive up to x_2 = 0.2 for NaCl and x_2 = 1 for NaBr. We observed a very close similarity of the relative viscosity to the B-coefficient vs. concentration of mixed solvent for both salts. The analogous similarity was observed for the other solvents [11, 12]. As can be seen in Fig. 1 and 2 respectively, η_{rel} and B-coefficient are different for NaCl and NaBr. Therefore, we observed a different influence of the anion on the course of functions $\eta_{rel} = f(x_2$ HMPA) and $B = f(x_2$ HMPA). Earlier [2, 3], we drew similar conclusions

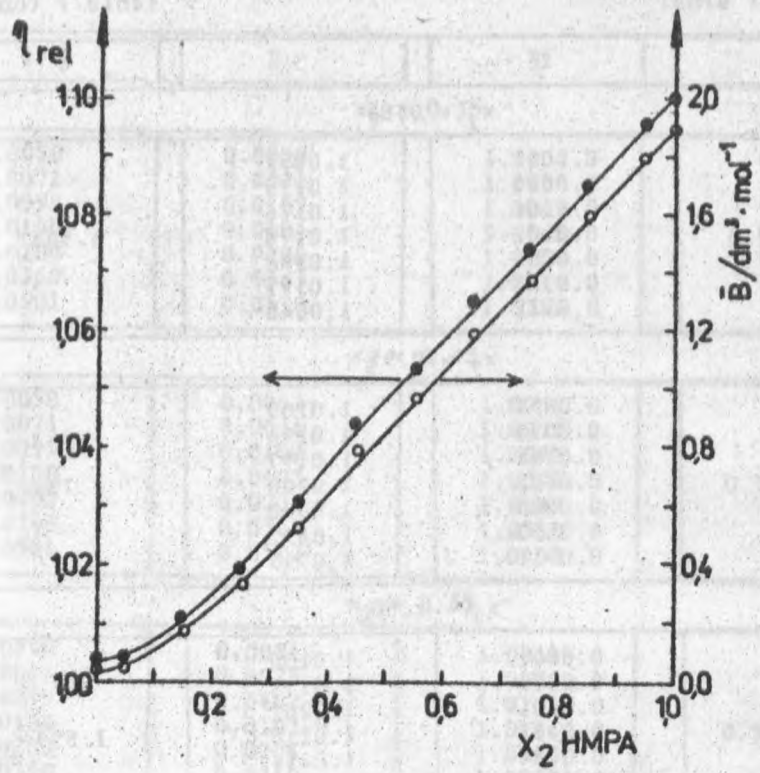

Fig. 2. Relative viscosities (●) and the B-coefficients (○) of NaBr solutions in water-HMPA mixtures at 298.15 K

from the standard enthalpy of solution of electrolytes in HMPA--water mixtures. The increase of η_{rel} and B-coefficient for both investigated salts suggested the structure-making effect. Taking into account the ionic contribution in B-coefficient presented by S a c c o et al., [13] (B_{Na^+} = 1.171 $dm^3 \cdot mol^{-1}$, B_{Cl^-} = 0.738 $dm^3 \cdot mol^{-1}$, B_{Br^-} = 0.732 $dm^3 \cdot mol^{-1}$, B_{I^-} = 0.602 $dm^3 \cdot mol^{-1}$), it can be supposed that Na^+ ion is a stronger structure-maker than anions (Br^-, Cl^-, I^-) are structure-breakers.

We can draw a conclusion from the results presented above that contrary to the HMPA-water-NaI system the plots $\Delta H_s^0 = f(x_2)$ and B(or η_{rel}) = $f(x_2)$ are not similar for solutions of NaCl and NaBr.

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Małgorzata Jóźwiak

WISKOZYMETRYCZNE BADANIA NaCl I NaBr W MIESZANINACH HEKSAMETYLOFOSFORTRIAMIDU Z WODĄ W TEMPERATURZE 298,15 K

Przeprowadzono badania gęstości i lepkości mieszanin heksametylofosfortriamidu z wodą zawierających NaCl i NaBr w temperaturze 298,15 K. Współczynniki B równania Jones'a-Dole'a były obliczone. Lepkość względna i współczynniki B są dyskutowane razem ze standardową entalpią rozpuszczania badanych elektrolitów w mieszaninie HMPA-H_2O w temperaturze 298,15 K.