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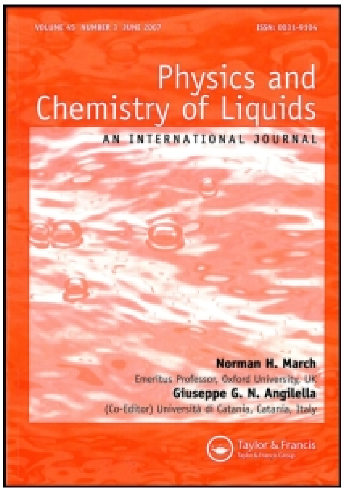

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# Conductivity Studies on LiBr, NaBr, KBr and CsBr Solutions in Binary Mixtures of *N,N* Dimethylformamide with Water at 298.15 K

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# CONDUCTIVITY STUDIES ON LiBr, NaBr, KBr AND CsBr SOLUTIONS IN BINARY MIXTURES OF *N,N*+DIMETHYLFORMAMIDE WITH WATER AT 298.15 K

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The electrical conductivities of LiBr, NaBr, KBr and CsBr in the mixtures of *N,N*-dimethylformamide with water, within the whole composition range (up to 80 mol% DMF for CsBr) have been measured. The obtained results were analysed using the Fuoss-Justice equation. The dependencies of the limiting molar conductances ($\Lambda_0$), Walden products ($\Lambda_0\eta$) and association constants ($K_A$) versus mixed solvent composition has been discussed.

*Keywords:* Conductance; walden products; *N, N*-dimethylformamide aqueous solutions

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## INTRODUCTION

The investigations of electrolyte solutions in the mixed solvents are our field of interest for many years. We have analysed the results obtained with the use of different experimental technics, such as conductometry [1–17], calorimetry [2,5–8,12,14,15,18,19], potentiometry [7,8,12–17], viscosimetry [3,4,11,18]. The conductometric method is well known to give valuable information regarding ion-solvent and ion-ion interactions in non-aqueous or mixed solvents. Moreover it, the values of association constant of electrolyte, $K_A$, obtained from conductometry, are necessary for correct description of thermodynamic [2,5–9,12–17,19] and viscosimetric data [3,4,11,18].

This work is a continuation of ours earlier studies [1] on conductometric properties of electrolyte solutions in the water + *N,N*-dimethylformamide system.

## EXPERIMENTAL

N,N-dimethylformamide, DMF, (Apolda, Germany) was purified according to the standard procedure, as it was described in previous paper [1]. The electric conductivity of the pure solvent was less than $10^{-7}\,\mathrm{S\,cm^{-1}}$, which correspond to the values cited in the literature (for example [20]). Conductivity water was prepared by triple distillation of water in presence of alkaline $KMnO_4$, and had a specific conductance less than $10^{-6}\,\mathrm{S\,cm^{-1}}$. The reagents used; LiBr suprapur grade (Merck) was recristallized from aceton and dried under reduced pressure at ca. 373.15 K; NaBr and KBr of spectral grade (Merck), were used without further purification and were only dried under reduced pressure at ca 353.15 K for ca. 10 hours; CsBr suprapur grade (Fluka) was recristallized from the conductivity water and dried under reduced pressure at ca. 353.15 K for ca. 10 hours. The salts were dried immediately prior to use. Solution of alkali metal bromides in DMF + water mixtures were prepared by weight. The measurements of the conductivity were performed in the range of 0.0002 – 0.012 $mol\ dm^{-3}$, usually for 16 concentration. Molar conductances were calculated after correction for solvent conductance. Details of the experimental procedure have been described previously [1]. The measurements of the conductivity were made using an E-315 A type bridge produced by "Mera-Tronic" (Poland), with the accuracy greater than 0.05%. The measuring cell was immersed into a thermostated bath keeping the temperature constant within ±0.005 K. Taking into account the reagents purity, equipment, method of measurements and accuracy of the temperature control, the uncertainty of the measured values was less than 0.05 per cent. More details are presented elsewhere [1 – 17]. The relative permittivity and viscosity of water + DMF mixtures were derived from the literature [21 – 23] as well as were numerically interpolated when necessary.

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## RESULTS AND DISCUSSION

The molar conductance data were analysed using the Fuoss-Justice [24–26] equation in the form:

$$\Lambda = \alpha[\Lambda_0 - S(\alpha c)^{1/2} + E(\alpha c)\ln(\alpha c) + J(\alpha c) + J_{3/2}(\alpha c)^{3/2}] \quad (1)$$

and

$$K_A = (1 - \alpha)/(\alpha^2 c y_{\pm}^2) \quad (2)$$

and

$$\ln y_{\pm} = -(A\alpha^{1/2}c^{1/2})/(1 + BR\alpha^{1/2}c^{1/2}) \quad (3)$$

In these equations $\Lambda_0$ is the limiting molar conductance, $\alpha$ is the dissociation degree of an electrolyte, $K_A$ is the ion association constant, and $y_{\pm}$ is the mean activity coefficient of ions on the molar scale; (the activity coefficient of the ion pairs is assumed to be equal to unity, as usual for diluted solutions). $A$ and $B$ are the Debye-Huckel equation coefficients. The analytical form of the remaining parameters i.e. $S$, $E$, $J$ and $J_{3/2}$ have been presented elsewhere [24–27]. The values of $\Lambda_0, K_A$ and $R$ were obtained with the use of the well known procedure (see for example paper of Fuoss [28]). In this method, the optimized values of $\Lambda_0$ and $K_A$ were acceptable, for which the function $\sigma_\Lambda = f(R)$ did have a sharp minimum ($\sigma_\Lambda$ is the standard deviation of the experimental results).

The error of the $R$ value i.e. $\Delta R$ was estimated as corresponding to $0.1(\sigma_\Lambda)_{\min}$.

This mean, that the values of $R$ were closed in the interval for which $\sigma_\Lambda < 1.1(\sigma_\Lambda)_{\min}$. The values of $\Lambda_0, K_A$ and $R$ thus obtained together with their standard deviations are collected in Tables I–IV. The values of the Walden products for investigated bromides as well as the values of the viscosity used for calculations are presented in Table V.

The courses of the dependencies of limiting molar conductances ($\Lambda_0$), and viscosity ($\eta$) of the mixed solvent versus mixed solvent composition are presented in Figure 1.

TABLE I Limiting molar conductance ($\Lambda_0$), ionic association constants ($K_A$) their standard deviations ($\sigma\Lambda_0$ and $\sigma K_A$), parameters $R$ and their estimated errors ($\Delta R$) for LiBr in water + DMF mixtures at 298.15 K

| *DMF/mol%* | $\Lambda_0$ | $\sigma\Lambda_0$ | $K_A$ | $\sigma K_A$ | $R$ |
|---|---|---|---|---|---|
| 0.0 | 116.95 | 0.03 | | | 4.0 |
| 2.5 | 94.00 | 0.02 | | | 4.0 |
| 5.0 | 77.59 | 0.02 | | | 4.5 |
| 10.0 | 57.62 | 0.03 | | | 4.5 |
| 20.0 | 39.28 | 0.02 | | | 4.5 |
| 25.0 | 35.25 | 0.03 | 1.0 | 0.5 | 4.5 |
| 35.0 | 32.73 | 0.01 | 1.9 | 0.5 | 5.0 |
| 50.0 | 36.62 | 0.01 | 2.7 | 0.8 | 5.0 |
| 70.0 | 49.09 | 0.02 | 5.9 | 1.3 | 5.0 |
| 80.0 | 58.12 | 0.03 | 6.9 | 1.9 | 5.5 |
| 90.0 | 68.05 | 0.02 | 8.0 | 2.1 | 6.5 |
| 100.0 | 79.30 | 0.03 | 12.1 | 3.1 | 7.0 |

in all cases $\Delta R = 0.5\ 10^{-8}$cm, except for 100 mol% DMF, where $\Delta R$ are 1.0 $10^{-8}$cm

TABLE II Limiting molar conductance ($\Lambda_0$), ionic association constants ($K_A$) their standard deviations ($\sigma\Lambda_0$ and $\sigma K_A$), parameters $R$ and their estimated errors ($\Delta R$) for NaBr in water + DMF mixtures at 298.15 K

| *DMF/mol%* | $\Lambda_0$ | $\sigma\Lambda_0$ | $K_A$ | $\sigma K_A$ | $R$ |
|---|---|---|---|---|---|
| 0.00 | 128.40 | 0.01 | | | 4.0 |
| 2.97 | 101.25 | 0.01 | | | 4.0 |
| 6.04 | 82.29 | 0.01 | | | 4.0 |
| 9.13 | 69.91 | 0.02 | | | 4.0 |
| 19.60 | 47.71 | 0.03 | | | 4.0 |
| 26.37 | 42.08 | 0.04 | 0.5 | 0.3 | 4.5 |
| 36.25 | 39.68 | 0.02 | 1.0 | 0.5 | 4.5 |
| 48.62 | 42.32 | 0.03 | 2.1 | 0.6 | 5.0 |
| 69.38 | 54.85 | 0.02 | 5.3 | 0.9 | 5.5 |
| 81.06 | 64.68 | 0.02 | 6.4 | 1.4 | 5.5 |
| 88.30 | 71.61 | 0.02 | 8.7 | 1.8 | 5.5 |
| 100.00 | 84.01 | 0.04 | 12.5 | 2.6 | 6.0 |

in all cases $\Delta R$= 0.5 $10^{-8}$cm, except for 19.60 mol % DMF and 81.06 mol% DMF, where values of $\Delta R$ are 1.0 $10^{-8}$cm

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TABLE III Limiting molar conductance ($\Lambda_0$), ionic association constants ($K_A$) their standard deviations ($\sigma\Lambda_0$ and $\sigma K_A$), parameters $R$ and their estimated errors ($\Delta R$) for KBr in water + DMF mixtures at 298.15 K

| *DMF/mol%* | $\Lambda_0$ | $\sigma\Lambda_0$ | *K* | $\sigma K$ | *R* |
|---|---|---|---|---|---|
| 0.00 | 151.86 | 0.04 | | | 4.0 |
| 2.50 | 123.23 | 0.02 | | | 4.0 |
| 5.00 | 102.75 | 0.04 | | | 4.0 |
| 10.00 | 77.28 | 0.03 | | | 4.5 |
| 20.00 | 53.35 | 0.03 | | | 4.5 |
| 25.00 | 47.97 | 0.02 | 1.1 | 0.7 | 5.0 |
| 35.00 | 44.11 | 0.03 | 1.8 | 0.9 | 5.0 |
| 50.00 | 44.65 | 0.02 | 3.2 | 1.2 | 5.5 |
| 70.00 | 58.13 | 0.03 | 5.4 | 1.5 | 5.5 |
| 80.00 | 66.31 | 0.02 | 6.4 | 1.9 | 5.5 |
| 90.00 | 75.65 | 0.03 | 8.9 | 2.1 | 6.5 |
| 100.00 | 84.97 | 0.04 | 9.7 | 2.7 | 6.5 |

in all cases $\Delta R = 0.5\ 10^{-8}$cm, except for 90.00 mol% DMF and 100.00 mol% DMF, where values of $\Delta R$ are $1.0\ 10^{-8}$cm

TABLE IV Limiting molar conductance ($\Lambda_0$), ionic association constants ($K_A$) their standard deviations ($\sigma\Lambda_0$ and $\sigma K_A$), parameters $R$ and their estimated errors ($\Delta R$) for CsBr in water + DMF mixtures at 298.15 K

| *DMF/mol%* | $\Lambda_0$ | $\sigma\Lambda_0$ | *K* | $\sigma K$ | *R* |
|---|---|---|---|---|---|
| 0.00 | 155.82 | 0.01 | 0.5 | 0.3 | 4.0 |
| 2.50 | 123.23 | 0.02 | 0.3 | 0.2 | 4.0 |
| 5.00 | 104.65 | 0.02 | 0.6 | 0.2 | 4.0 |
| 10.00 | 78.36 | 0.02 | 0.5 | 0.3 | 4.5 |
| 20.00 | 53.28 | 0.02 | 1.0 | 0.5 | 4.5 |
| 25.00 | 47.40 | 0.02 | 1.5 | 0.8 | 4.5 |
| 35.00 | 44.00 | 0.02 | 3.1 | 1.1 | 5.0 |
| 50.00 | 47.69 | 0.02 | 5.3 | 1.9 | 5.0 |
| 70.00 | 60.60 | 0.02 | 9.9 | 2.3 | 5.5 |
| 80.00 | 69.49 | 0.03 | 14.5 | 3.5 | 6.0 |

in all cases $\Delta R = 0.5\ 10^{-8}$cm, except for 19.60 mol% DMF and 81.06 mol% DMF, where values of $\Delta R$ are $1.0\ 10^{-8}$cm

Units in Tables I–IV are: $\Lambda_0$ and $\sigma\Lambda_0/S$ cm$^2$ mol$^{-1}$; $K_A$ and $\sigma K_A/dm^3$ mol$^{-1}$; $R/10^{-8}$cm

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TABLE V Viscosity of the water + DMF mixtures taken for calculation and the values of the Walden products, $\Lambda_0\eta$, for (a) LiBr, (b) NaBr, (c) KBr, (d) CsBr at 298.15 K

| *DMF/mol%* | $\eta$ | $\Lambda_0\eta$ | | | |
|---|---|---|---|---|---|
| | | *a* | *b* | *c* | *d* |
| 0.00 | 0.890 | 1.041 | 1.146 | 1.352 | 1.387 |
| 2.50 | 1.100 | 1.034 | | 1.356 | 1.390 |
| 2.97 | 1.140 | | 1.154 | | |
| 5.00 | 1.325 | 1.028 | | 1.361 | 1.387 |
| 6.04 | 1.395 | | 1.148 | | |
| 9.13 | 1.659 | | 1.160 | | |
| 10.00 | 1.737 | 1.000 | | 1.342 | 1.361 |
| 19.60 | 2.345 | | 1.119 | | |
| 20.00 | 2.358 | 0.941 | | 1.258 | 1.256 |
| 25.00 | 2.485 | 0.876 | | 1.192 | 1.179 |
| 26.37 | 2.500 | | 1.052 | | |
| 35.00 | 2.405 | 0.757 | | 1.061 | 1.058 |
| 36.25 | 2.370 | | 0.940 | | |
| 48.62 | 1.910 | | 0.808 | | |
| 50.00 | 1.865 | 0.683 | | 0.870 | 0.889 |
| 69.38 | 1.260 | | 0.691 | | |
| 70.00 | 1.245 | 0.611 | | 0.724 | 0.755 |
| 80.00 | 1.040 | 0.604 | | 0.690 | 0.726 |
| 81.06 | 1.023 | | 0.662 | | |
| 88.30 | 0.922 | | 0.600 | | |
| 90.00 | 0.900 | 0.612 | | 0.690 | |
| 100.00 | 0.795 | 0.630 | 0.668 | 0.676 | |

The values of viscosity ($\eta$) were derived from ref. [23] as well as were interpolated numerically.
Units in Table V: $\eta$/P, $\Lambda_0\eta$/, $S\text{cm}^2\,\text{mol}^{-1}$P (P denote Poise units)

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The dependencies of the Walden products ($\Lambda_0\ \eta$), and association constants ($K_A$) versus mixed solvent composition are illustrated in Figures 2 and 3, respectively. As it is seen in Figure 1, the viscosity of the solvent mixtures increases rapidly with increasing of DMF content, reaching a maximum of nearly 3 times the viscosity of water at about 25 mol% of DMF in the system. When the maximum of the viscosity is attained, then the values of the viscosity decrease slower with the increasing of DMF content in the mixture, up to value 0.00795 Poise [23] for pure DMF. Limiting molar conductances ($\Lambda_0$) for the bromides investigated in this paper decrease with the increasing DMF content, reaching a minimum at about 35 mol% of DMF, and subsequently the values of $\Lambda_0$ increases gradually with the increasing DMF content. Similar dependencies of the limiting molar conductances ($\Lambda_0$) as the function of the mixed solvent composition of water

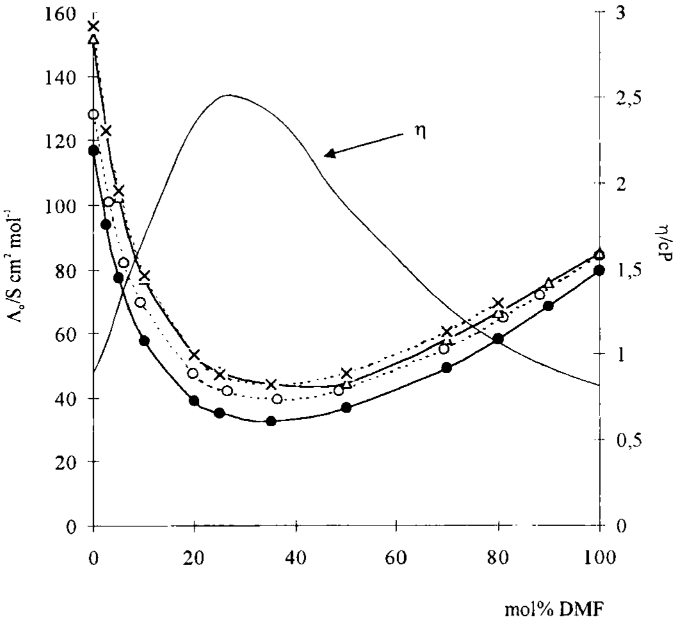

FIGURE 1 Viscosity ($\eta$) of water + DMF mixtures and limiting molar conductances ($\Lambda_0$) in water + DMF mixtures at 298.15 K for: LiBr -●-; KBr -△-; NaBr ··○·· and CsBr ··×··.

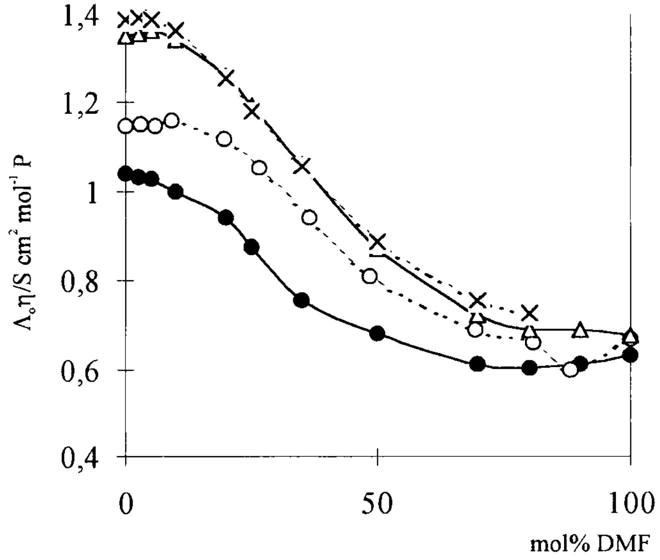

FIGURE 2 Walden product ($\Lambda_0\eta$) in water + DMF mixtures at 298.15 K for: LiBr -●-; KBr -△-; NaBr ··○·· and CsBr ··×··.

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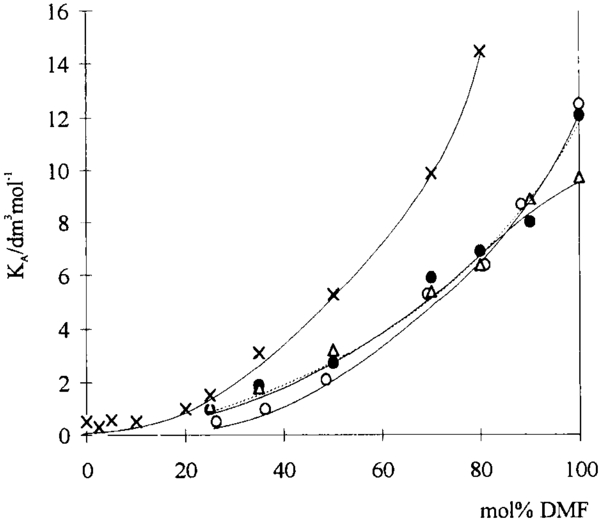

FIGURE 3 Values of $K_A$ as a function of the DMF content in water + DMF mixtures at 298.15 K for: LiBr •; KBr Δ; NaBr ο and CsBr ×.

with DMF, for others uni-univalent salts than those presented in this work, were observed in papers [1,20,29,30]. The mentioned above observations can suggest, that influence of the viscosity of mixed solvent on the values of the limiting molar conductances is significant. It is well known however, that the analyse of the dependencies of the Walden product ($\Lambda_0\eta$) vs. solvent composition gives more valuable informations on the solute-solvent interactions. In fact, as it is seen in Figure 2, the dependencies of $\Lambda_0\eta = f(x)$, (where $x$ denote mixed solvent composition), are similar for all four investigated bromides. In the range of the small amounts of the DMF contents the small maxima (or stabilised values as in the case of LiBr) are achieved, and subsequently the values of $\Lambda_0\eta$ decreases monotonically showing flat minum at about 85 mol% DMF. In paper [1] which deals with the conductivity of chlorides of lithium, sodium, potassium and cesium in the water + DMF mixtures at 298.15 K, the dependencies $\Lambda_0\eta = f(x)$ were more characteristic. This means, that the maximum in the range of the small amounts of the DMF contents, and minimum within the DMF rich region were more distinct. Similarly, more characteristic extremum for the dependencies $\Lambda_0\eta = f(x)$ were found for the

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solutions of $NaNO_2$, $CH_3COONa$, NaCl and for nitrates of sodium, potassium and ammonium in the water + DMF mixtures [29,30]. This can prove, that the differences in the interactions of anions with dipoles of the mixed solvent water + DMF are considerable, because cations are the same.

In papers [4,31 – 42] was also shown that the addition of organic solvent to the water leads to the excess cation mobility, which can be explained by so called "sorting effect" [32]. This phenomena results from the fact, that the composition of the solvation shell of cations are different than the mixed solvent composition, when the small amounts of organic solvents are added to the water. In this case, the microscopic viscosity (around ions) increases slower than macroscopic viscosity, which leads to the greater ionic mobility, as it results from the Stokes' low. This behaviour can also explain the changes in the mobilities of cations, which were investigated in this work. However, the character of the interactions of anions with the mixed solvent are less evident, particularly in the case of the mixtures of water with aprotic solvent, including in it DMF. Other authors [43] suggest, that the conductometric properties of anions, are connected with the breaking or ordering of the water structure by organic solvent.

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We intend to analyse the conductometric properties of anions in binary mixtures of DMF with water in the next papers. The differences between the limiting conductances as well as in the Walden products are sources of informations on differences between interactions of cations of lithium, sodium, potassium and cesium with dipoles of the mixed solvent. One can observe, that for the values of $(\Lambda_0\eta)$, in the entire range of the mixed solvent composition, the following relationship is fulfilled; $\Lambda_0\eta(Li^+) < \Lambda_0\eta(Na^+) < \Lambda_0\eta(K^+)\Lambda_0\eta(Cs^+)$. This can signify, that the effective radii of the lithium ion is always most greater. Taking into account this fact, that crystallografic radii of lithium ion is smallest, one can assume, that lithium ion is strongly solvated by mixed solvent molecules. In the contrary, cesium ion having their smaller charge density, will be smallest solvated. From the comparison of the $\Lambda_0\eta$ values, can also result, that the differences in the solvation of $Li^+$, $Na^+$, $K^+$and $Cs^+$ decreases with increasing of DMF contents in the mixture. Probably it results from more basicity of DMF than water. As it results from Figure 3, the values of the association constants $(K_A)$ are rather small and increase with the increasing of

DMF content in the mixture, which can be explained not only by the decreasing of the relative permittivity of the mixed solvent with the decreasing of the DMF content, but also by the fact, that the anions are weakly solvated by disubstituted amides. As it results from the data given in Tables I – IV, the values of the parameter $R$ are similar for all four investigated bromides and increases with the increasing of DMF contents. In our opinion, more detailed analyse of the conductometric properties of ions in terms of their ion-solvent interactions will be possible, when the conductivity studies in the water + DMF mixtures for many others uni-univalent electrolytes will be made, including the division into the ionic contribution. Appropriate studies are already under way and their results will be reported later.

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