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Conductance Studies of Aqueous Ionic Liquids Solutions [emim][BF_4] and [bmim][BF_4] at Temperatures from (283.15 to 318.15) K

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We present for the first time the determination of molar conductances, Λ, of ionic liquids solutions (ILs) such as 1-ethyl-3-methylimidazolium tetrafluoroborate [emim][BF_4] and 1-butyl-3-methylimidazolium tetrafluoroborate [bmim][BF_4] as a function of temperature. The data was analyzed with Barthel's lcCM model to obtain ionic association constants, K_A, limiting molar conductances, Λ_o of these electrolytes. The examined ionic liquids are practically fully dissociated in water over the whole temperature range (283.15 to 318.15 K). From the temperature dependence of the limiting molar conductivities the Eyring's activation enthalpy of charge transport was determined. The Walden product values were also calculated.

Keywords: limiting molar conductance, 1-alkyl-3-methylimidazolium tetrafluoroborate, ion association, thermodynamic functions

1. INTRODUCTION

The data of physical and chemical properties on ionic liquids (ILs) are essential for both theoretical research and industrial application. A survey of literature indicates that physical properties of pure ionic liquids have been studied extensively, but the thermophysical and thermodynamic properties of ILs, and in particular their aqueous or organic mixtures, have not been studied in a systematic way so far. A literature review indicates that almost nothing is known about the temperature dependence of the transport properties of dilute ILs solutions, despite the importance of the infinite-dilution limit of such data in understanding the ion-ion and ion-solvent interactions and the possibility of prediction of ILs in specific applications.

The most intensively investigated ILs are those with imidazolium cation, but very little conductivity studies concerned the ionic association of ILs in molecular solvents [1-13]. From these papers results that the alkyl chain length of the cation, type of anion, and physical properties of the molecular solvents affect the ionic association constants. The ionic liquids are solvated to a different extent by the molecular solvents, and the ionic association is affected significantly by ionic solvation [11]. Studies on the ionic association and solvation behavior of ionic liquids in various solvents as a function of the temperature have been a subject of interest lately [14, 15]. The results of the conductance measurements of 1-ethyl-3-methylimidazolium tetrafluoroborate and 1-butyl-3-methylimidazolium tetrafluoroborate solutions in N,N-dimethylformamide [14] confirm that in this dipolar aprotic solvent a slight ion association occurs for the ionic liquids over the whole investigated temperature range (like other ionic liquids in acetonitrile, methanol and ethanol). In turn, in protic solvent 1-PrOH a strong ionic association was observed for the ILs [15] (similarly in 2-propanol, 1-butanol and 1-pentanol). Water promotes significantly dissociation of the various ionic liquids [3, 4, 8, 10, 11, 16]. In fact similar to the classical electrolytes, the ln K_A values of the ILs were found to increase linearly with the reverse of the dielectric constants of the solvents, which indicates that the electrostatic interaction between the ions are predominant for the ionic association of the ILs [1].

Conductometry is a reliable, affordable and convenient electrochemical technique, which provides valuable information on ion-solvent interactions, ion-ion association and solvent structure. Such studies are of considerable interest for the optimal use of ionic liquid solutions in high energy batteries and other electrochemical systems and for understanding the ion pair effect in such systems. Therefore, the studies allow the prediction of ILs in various specific applications.

To the best of our knowledge there are no conductometric data available in the literature about ILs tested in water at various temperatures. We present in this manuscript, precise conductivity measurements which have been carried out in dilute solutions of [emim][BF_4] and [bmim][BF_4] in water at temperatures range (283.15K - 308.15) K at atmospheric pressure. Imidazolium's ionic liquids were chosen because of their thermal and chemical stability and the insignificant impact of air and moisture. The obtained data were used to calculate the values of the limiting molar conductances, Λ_o, and the association constants, K_A on the basis of lcCM model. The Eyring activation enthalpy of charge transport, $\Delta H^{\ddagger}_{\lambda}$, as well as the Walden producs, $\Lambda_o\eta$, for the electrolytes have been evaluated.

2. EXPERIMENTAL

2.1. Reagents and chemicals

The specifications of used chemicals are summarized in Table 1.

Table 1. Specification of chemical samples

chemical name	source	initial mass fraction purity	purification method	final water mass fraction
[emim][BF_4]	Fluka	0.990	none	$<0.0002^a$ $<0.00015^b$

[bmim][BF_4]	Fluka	0.985	none	<0.0005[a] <0.0005[b]

[a] Manufacturer's analysis. [b] Our analysis (Karl Fischer coulometric titration).

Double distilled, deionized and degassed water with a specific conductance better than $0.5 \cdot 10^{-6}$ S · cm^{-1} was used for the preparation of the mixed solvents.

2.2. Apparatus

All the solutions were prepared by mass using an analytical balance (Sartorius RC 210D) with a precision of $\pm 1 \cdot 10^{-5}$ g.

The measurement procedure was based on the method described by Bešter-Rogač *et al.* [8, 17] and used by us in our previous works [14, 15, 18, 19]. Conductivity measurements were performed with a three-electrode cell with the use of a Precise Component Analyser type 6430B (Wayne-Kerr, UK) under argon atmosphere and at the different frequencies, v, (0.2, 0.5, 1, 2, 3, 5, 10, 20) kHz. The temperature was kept constant within 0.003 K (Calibration Thermostat Ultra UB 20F with Through-flow cooler DLK 25, Lauda, Germany). The details of the experimental procedure for conductometric measurements were described in our previous paper [14]. The uncertainty of the measured values of conductivity was 0.03%.

3. RESULTS AND DISCUSSION

Table 2. Molar conductances, Λ, corresponding molalities, m, and density gradients, b, for aqueous solutions of [emim][BF_4] and [bmim][BF_4] over the temperature range from (283.15 to 318.15) K

$10^3 m$ mol kg^{-1}	Λ S cm^2 mol^{-1}	$10^3 m$ mol kg^{-1}	Λ S cm^2 mol^{-1}	$10^3 m$ mol kg^{-1}	Λ S cm^2 mol^{-1}	$10^3 m$ mol kg^{-1}	Λ S cm^2 mol^{-1}
[emim][BF_4]							
T = 283.15 K		T = 288.15 K		T = 293.15 K		T = 298.15 K	
b = 0.0460 kg^2 dm^{-3}		b = 0.0449 kg^2 dm^{-3}		b = 0.0439 kg^2 dm^{-3} mol^{-1}		b = 0.0645 kg^2 dm^{-3} mol^{-1}	
0.7604	77.722	0.4864	88.836	0.5901	100.118	0.6182	112.089
1.3046	77.258	0.9779	88.282	1.0861	99.529	1.7167	110.813
2.1105	76.712	1.9749	87.476	2.0802	98.661	3.7337	109.289
3.4936	75.960	3.4419	86.589	3.5414	97.699	6.9897	107.532
5.1001	75.235	4.8963	85.872	5.0155	96.909	11.941	105.513
6.9799	74.500	6.8990	85.028	7.0330	95.990	15.219	104.397
10.911	73.188	11.047	83.574	10.837	94.548	17.542	103.676
14.108	72.254	14.152	82.639	14.143	93.480	20.093	102.935
17.413	71.371	17.480	81.729	17.579	92.486	23.128	102.110
20.028	70.716	19.998	81.089	20.151	91.799	25.205	101.575

23.790	69.825	23.530	80.245	23.585	90.942	27.641	100.974
26.761	69.158	26.595	79.554	26.483	90.262	30.099	100.393
		29.956	78.833	29.992	89.482		
T = 303.15K		T = 308.15 K		T = 313.15 K		T = 318.15 K	
b = 0.0422 kg^2 dm^{-3}		b = 0.0414 kg^2 dm^{-3}		b = 0.0407 kg^2 dm^{-3} mol^{-1}		b = 0.0400 kg^2 dm^{-3} mol^{-1}	
1.0182	124.103	0.5551	138.075	1.0938	150.858	1.0203	165.351
2.0033	122.983	1.1630	137.040	1.7851	149.834	1.9135	163.877
3.4776	121.742	2.0121	135.981	3.4032	148.031	3.5133	161.923
4.8980	120.777	3.3852	134.666	4.8945	146.723	5.0815	160.400
7.1251	119.511	5.0522	133.389	6.9612	145.200	7.0203	158.804
10.107	118.091	7.0673	132.100	11.287	142.606	10.999	156.088
14.814	116.221	9.9919	130.519	14.048	141.202	14.102	154.285
17.543	115.271	14.728	128.382	17.400	139.667	17.430	152.549
20.068	114.454	17.660	127.226	19.942	138.595	19.939	151.341
23.310	113.476	20.169	126.310	23.419	137.231	23.406	149.783
26.372	112.611	23.675	125.119	26.530	136.091	26.489	148.486
		26.540	124.209	29.799	134.960	31.271	146.605
		29.360	123.358				
[bmim][BF$_4$]							
T = 283.15 K		T = 288.15 K		T = 293.15 K		T = 298.15 K	
b = 0.0351kg^2 dm^{-3} mol^{-}		b = 0.0333 kg^2 dm^{-3}		b = 0.0292 kg^2 dm^{-3} mol^{-1}		b = 0.0284 kg^2 dm^{-3} mol^{-1}	
0.5908	73.488	0.5070	83.270	0.4904	93.936	0.4447	105.616
1.0714	73.015	1.1300	82.613	1.0199	93.301	0.9793	104.873
2.1409	72.240	2.0214	81.943	1.9947	92.487	1.9562	103.944
5.1364	70.735	5.0567	80.403	5.0355	90.841	3.0045	103.194
7.1697	69.939	7.8926	79.362	7.0342	90.052	3.9332	102.641
9.9741	68.987	9.5425	78.844	10.032	89.074	5.8783	101.677
11.954	68.382	11.853	78.191	12.153	88.478	7.9552	100.826
14.374	67.696	15.068	77.382	15.047	87.755	9.9835	100.108
16.461	67.142	17.155	76.904	16.952	87.322	11.963	99.483
18.060	66.737	19.942	76.309	18.862	86.915	14.904	98.657
20.113	66.236	21.942	75.909			17.719	97.950
22.134	65.764	24.022	75.512				
24.254	65.285						
T = 303.15K		T = 308.15 K		T = 313.15 K		T = 318.15 K	
b = 0.0278kg^2 dm^{-3} mol^{-}		b = 0.0269 kg^2 dm^{-3}		b = 0.0240 kg^2 dm^{-3} mol^{-1}		b = 0.0230 kg^2 dm^{-3} mol^{-1}	
0.5299	118.022	0.6180	131.357	0.5476	145.957	0.5466	161.384
1.0060	117.300	1.0803	130.591	0.9973	145.052	1.1178	160.095
2.0099	116.218	1.9630	129.492	2.0170	143.553	2.1061	158.447
5.0215	114.096	3.5103	128.049	3.5474	141.869	3.4358	156.707
7.1274	113.003	5.0418	126.908	5.0552	140.517	5.0327	154.965

9.9539	111.778	7.0504	125.644	6.9851	139.029	7.1848	152.948
12.023	110.996	9.9556	124.094	9.9726	137.046	10.062	150.598
14.027	110.305	13.059	122.665	15.030	134.204	15.012	147.093
15.918	109.700	17.160	121.008	18.063	132.695	18.067	145.146
18.023	109.070	20.261	119.879	20.165	131.710	20.047	143.948
20.022	108.509	23.047	118.933	23.059	130.420	22.991	142.240
22.083	107.961	25.300	118.207	25.340	129.450	25.114	141.057
24.005	107.476						

The densities, viscosities, and relative water’s permittivity were taken from ref. [20].

To convert molonity, $\tilde{m}$, (moles of electrolyte per kilogram of solution) into molarity, c, the values of density gradients, b, have been determined independently and used in the equation

$$c / \tilde{m} = \rho = \rho_{\mathrm{o}} + b\,\tilde{m} \qquad \text{(1a)}$$

where ρ_{o} is the density of the solvent. Molar concentrations, c, were necessary to use the conductivity equation. The density gradients and the molar conductances of the ILs in solution, Λ, as a function of IL molality, m, (moles of electrolyte per kilogram of solvent) and temperature are presented in Table 2. The relationship among m, $\tilde{m}$, and c is the following

$$\tilde{m} = c/\rho = 1 / (1 + mM) \qquad \text{(1b)}$$

where M is the molar mass of electrolyte.

The plot of molar conductances, Λ, versus the square root of the molar concentration, $c^{1/2}$, for the investigated systems monotonically decreases as shown in Figures 1 and 2, respectively over the temperature range from 318.15 to 283.15 K.

The conductivity data were analyzed in the framework of the low concentration Chemical Model (lcCM) [21]. This approach uses the set of equations

$$\Lambda = \alpha\,[\Lambda_{\mathrm{o}} - S(\alpha c)^{1/2} + E(\alpha c)\ln(\alpha c) + J(\alpha c) + J_{3/2}(\alpha c)^{3/2}] \qquad \text{(2)}$$

$$K_{\mathrm{A}} = (1 - \alpha) / (\alpha^2 c y_{\pm}^2) \qquad \text{(3)}$$

and

$$\ln y_{\pm} = -\,(A\alpha^{1/2}c^{1/2}) / (1 + BR\alpha^{1/2}c^{1/2}) \qquad \text{(4)}$$

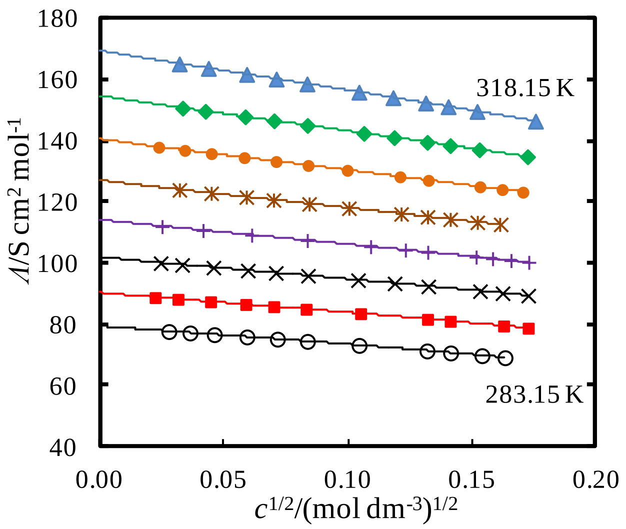

Figure 1. Molar conductance, Λ, of aqueous [emim]BF_4 solutions versus $c^{1/2}$ at experimental temperatures; ○, 283.15 K; ■, 288.15 K; ×, 293.15 K; +, 298.15 K; x, 303.15 K; ●, 308.15 K; ♦, 313.15 K; ▲, 318.15 K. The lines represent the calculations according to Eqs (2) through (4).

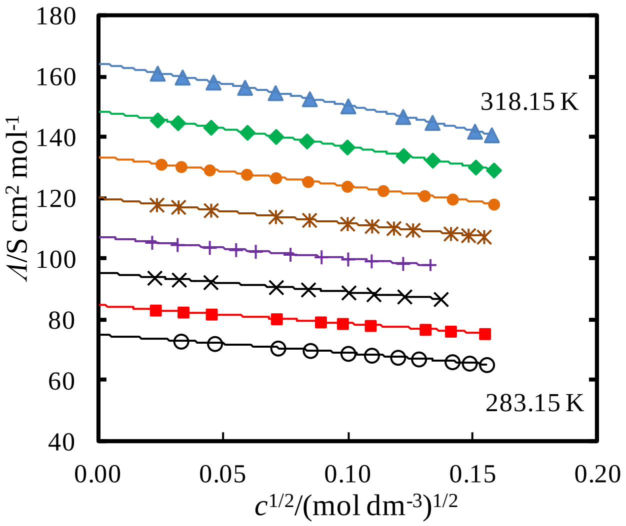

Figure 2. Molar conductance, Λ, of aqueous [bmim]BF_4 versus $c^{1/2}$ at experimental temperatures; ○, 283.15 K; ■, 288.15 K; ×, 293.15 K; +, 298.15 K; x, 303.15 K; ●, 308.15 K; ♦, 313.15 K; ▲, 318.15 K. The lines represent the calculations according to Eqs (2) through (4).

In these equations, Λ_o is the limiting molar conductance; α is the dissociation degree of an electrolyte; K_A is the ionic association constant; R is the distance parameter of ions; $y_\pm$ is the activity coefficient of ions on the molar scale; A and B are the Debye–Hückel equation coefficients. The analytical form of the parameters S, E, J, and $J_{3/2}$ was presented previously [21]. The values of Λ_o, K_A, and R were obtained using the well-known procedure given by Fuoss [22] and are collected in Table 3.

Table 3. Limiting molar conductances, Λ_o, association constants, K_A, distance parameters, R, and standard deviations, $\sigma(\Lambda)$, for the investigated electrolytes in water at different temperatures

T/K	Λ_o/S cm^2 mol^{-1}	K_A/dm^3 mol^{-1}	R/nm	$\sigma(\Lambda)$
[emim][BF$_4$]				
283.15	79.39 ± 0.01	1.36	7.98	0.02
288.15	90.38 ± 0.02	1.01	7.42	0.04
293.15	102.01 ± 0.02	0.85	7.20	0.04
298.15	114.27 ± 0.01	0.89	6.91	0.04
303.15	127.17 ± 0.02	0.92	6.92	0.03
308.15	140.66 ± 0.02	1.06	6.78	0.04
313.15	154.86 ± 0.01	1.39	6.62	0.03
318.15	169.61 ± 0.01	1.59	6.70	0.03
[bmim][BF$_4$]				
283.15	74.93 ± 0.01	1.87	7.81	0.01
288.15	84.73 ± 0.01	0.60	7.50	0.02
293.15	95.57 ± 0.01	0.21	7.21	0.02
298.15	107.39 ± 0.01	0.16	7.18	0.02
303.15	120.21 ± 0.01	0.59	6.92	0.03
308.15	134.02 ± 0.01	1.06	7.24	0.04
313.15	148.78 ± 0.02	1.54	7.78	0.04
318.15	164.54 ± 0.02	2.17	8.41	0.04

As seen from Table 3, both ionic liquids are practically fully dissociated in water. In DMF the values of association constants are also very small [14], but slightly higher than in water, whereas in 1-propanol both ILs were highly associated [15]. This may mean that an essential role in the ionic association process plays the relative permittivity of the solvent.

One should pay attention to the fact that the values of association constants for various ionic liquids in water are also very small as reported previously [4, 8, 10, 11], which confirms that the ionic liquids exist as free ions in aqueous solution. The values of K_A (and Λ_o) differ considerably from those of Wang et al. [1] who reported for [bmim][BF$_4$] at 298.15 K association constant of ~100 dm^3 mol^{-1}. Such a large value of the association constant is surprising given by the high permittivity of water. Wang et al. presented values of K_A and Λ_o in various solvents which also differ from other literature data [12, 13, 23, 24]. In turn, Katsuta et al. [3] illustrated for [bmim][BF$_4$] at 298.15 K an association constant of ~5 dm^3 mol^{-1}. This value also differs slightly from ours, which may result from another research method (capillary electrophoresis).

The limiting molar conductances increase as the temperature increases since the mobility of free ions is higher. The values of Λ_o for [emim][BF$_4$] are higher compared to those values for [bmim][BF$_4$], because the Λ_o values increase with decreasing alkyl chain length of the ILs.

From the temperature dependence of Λ_o, the Eyring activation enthalpy of charge transport, $\Delta H_\lambda^\ddagger$, was obtained by using equation 5

$$\ln \Lambda_o + 2/3 \ln \rho_o = -\frac{\Delta H_\lambda^\ddagger}{RT} + D \qquad (5)$$

where D is an empirical constant. From the slope of the linear dependencies of $\ln \Lambda_o + 2/3 \ln \rho_o$ versus the inverse of the temperature ($1/T$), which are shown in Figure 3, we obtained the following $\Delta H_\lambda^\ddagger$ values 16078 and 16727 J mol^{-1} for [emim][BF$_4$] and [bmim][BF$_4$], respectively. For [bmim][BF$_4$], the value of $\Delta H_\lambda^\ddagger$ is thus higher than [emim][BF$_4$] by 649 units. It is the result of the presence of a larger substituent in the [bmim]$^+$ cation compared to [emim]$^+$.

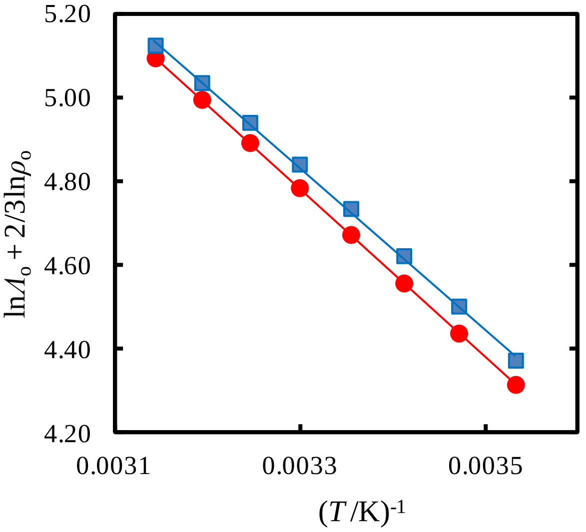

Figure 3. Plot of $\ln \Lambda_o + 2/3 \ln \rho_o$ as a function of $1/T$ for ■, [emim][BF$_4$]; and ●, [bmim][BF$_4$] in water.

In the case of protic 1-propanol the $\Delta H_\lambda^\ddagger$ value for [bmim][BF$_4$] was higher than [emim][BF$_4$] by 330 units [15] and for aprotic DMF the $\Delta H_\lambda^\ddagger$ value for [bmim][BF$_4$] was also higher than [emim][BF$_4$], but only by 128 units [14]. It should also be noted that in the case of protic solvents (water and 1-propanol) $\Delta H_\lambda^\ddagger$ values are very similar and almost twice bigger than those for DMF [14, 15]. It can be concluded that the value of $\Delta H_\lambda^\ddagger$ depends on the formation of hydrogen bonds between molecules of the solvent and its structure.

As shown in Table 4, the association constant values, K_A, are very small. This means that the test electrolytes are almost completely dissociated and can be treated as strong electrolytes. For this reason, the values of thermodynamic functions of the association were not determined because of their too large uncertainties.

In the next stage of this investigation we decided to calculate the Walden products, connected with Stokes radii, often called the hydrodynamic radius (Table 4)

$$\eta \lambda_o = r_S = 8.204 z_i / r_S \qquad (6)$$

where z_i is the charge number of the ion, η is the dynamic viscosity of water.

Table 4. Walden products , $\Lambda_o \eta$, in water

	$10^{-2} \Lambda_o \eta$ / S cm^2 mol^{-1}mPa s	
T/K	[emim][BF_4]	[bmim][BF_4]4
283.15	1.035	0.977
288.15	1.028	0.963
293.15	1.022	0.958
298.15	1.017	0.956
303.15	1.014	0.958
308.15	1.011	0.964
313.15	1.011	0.971
318.15	1.013	0.983

Table 4 shows the Walden products of [emim][BF_4] and [bmim][BF_4] ionic liquids which change very slightly with temperature. Similarly, small changes in the value of the products Walden can be observed for these ionic liquids in the case of N,N-dimethylformamide [14], and 1-propanol [15]. Considering equation (6) can be assumed that the temperature change in the used range does not practically affect the effective size of ions. The data contained in Table 4 and the equation (6) shows that the effective size of the ion [bmim]$^+$ are larger than those of the ions [emim]$^+$ over the range of applied temperatures. Comparing the $\Lambda_o\eta$ values from Table 4 with those given in [14, 15], can be seen that the Walden products in water, DMF and 1-propanol satisfy the following relations:

$\Lambda_o\eta$ [emim] [BF_4] > $\Lambda_o\eta$ [bmim] [BF_4]

and

$\Lambda_o\eta$ (water) > $\Lambda_o\eta$ (DMF) > $\Lambda_o\eta$ (1-propanol)

The first relationship, suggests a greater effective radius of the ion [bmim]$^+$ which corresponds well with the fact that the Eyring activation enthalpy of charge transfer is larger for this ion. However, a meaningful comparison of the effective size of ions will be possible only on the basis of values of ionic conductances and by use of equation (6).

4. CONCLUSIONS

Molar conductances of aqueous solutions of 1-ethyl-3-methylimidazolium tetrafluoroborate and 1-butyl-3-methylimidazolium tetrafluoroborate have been successfully reported for the first time at T = (283.15 to 318.15) K. Conductivity data were analyzed on the basis of Barthel's low concentration Chemical Model (lcCM) showing that these electrolytes exist as free ions in aqueous solution at used temperature range. This strategic, low cost and effective method allowed us to calculate the activation enthalpy of charge transfer and the Walden products and compared with the corresponding values for the DMF and 1-propanol.

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