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## Physicochemical Properties of Lithium Bis(trifluoromethanesulfonyl)imide Solutions in Sulfolane

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**Abstract**—A study is performed of the physicochemical properties (ionic conductivity, viscosity, and density in the 30–50°C range of temperatures) and the electrochemical and thermal stability of lithium bis(trifluoromethanesulfonyl)imide solutions in sulfolane in the 0.001–1.89 M range of concentrations. It is shown that lithium bis(trifluoromethanesulfonyl)imide solutions in sulfolane exhibit high electrochemical stability (5.75 V vs. Li/Li<sup>+</sup>) and moderate ionic conductivity ( $2.75 \times 10^{-3} \Omega^{-1} \text{cm}^{-1}$  at 30°C for a 1 M solution), which are comparable to the respective parameters of sulfolane solutions of lithium hexafluorophosphate. The structure and transport properties of sulfolane solutions of lithium bis(trifluoromethanesulfonyl)imide are determined by their concentrations. In dilute solutions, lithium bis(trifluoromethanesulfonyl)imide is weakly associated. The degree of electrolytic dissociation of LiN(SO<sub>2</sub>CF<sub>3</sub>)<sub>2</sub> initially falls and then rises upon an increase in its concentration. The electrolytic dissociation of lithium bis(trifluoromethanesulfonyl)imide rises along with its concentration, due to the formation of ion triplets and more complex ion associates. The constant of association and limit equivalent conductivity of LiN(SO<sub>2</sub>CF<sub>3</sub>)<sub>2</sub> in sulfolane rise along with temperature, due to a drop in the degree of self-association and the degradation of the solvent's structure. A specific feature of sulfolane solutions of lithium bis(trifluoromethanesulfonyl)imide is their tendency to form stable supercooled solutions.

**Keywords:** lithium bis(trifluoromethanesulfonyl)imide, sulfolane, electrolyte solutions, lithium batteries

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### INTRODUCTION

In designing electrochemical energy storage devices with energy characteristics better than those of existing devices, it is important to develop electrolyte systems that are safe and have high ionic conductivity and chemical/electrochemical stability while remaining in a liquid phase state throughout a wide range of temperatures.

Properties of electrolyte systems are determined by the properties of electrolyte salts and solvents. A promising class of lithium salts for lithium-ion (LIB) and lithium battery (LB) electrolytes are such lithium imides as bis(fluorosulfonyl)imide (LiN(SO<sub>2</sub>F)<sub>2</sub>), bis(trifluoromethanesulfonyl)imide (LiN(SO<sub>2</sub>CF<sub>3</sub>)<sub>2</sub>), and bis(perfluoroethylsulfonyl)imide (LiN(SO<sub>2</sub>CF<sub>2</sub>CF<sub>3</sub>)<sub>2</sub>) [1–3]. Imide anions, unlike lithium salts with complex anions, are considerably larger than the anions of the most commonly used LiPF<sub>6</sub>, LiClO<sub>4</sub>, and LiBF<sub>4</sub> salts. This ensures fairly good solubility and a high degree of electrolytic dissociation of lithium imides in dipolar aprotic solvents (DASes) of different origins [4, 5]. Of considerable interest is lithium bis(trifluoromethanesulfonyl)imide (LiTFSI), which readily dissociates even in solvents with low dielectric permeabilities. It

also exhibits high thermal stability and resistance to hydrolysis because it has a weakly coordinating anion with a highly delocalized charge density (lithium bis(trifluoromethanesulfonyl)imide) [2, 4]. A disadvantage of electrolyte solutions based on this salt is the corrosion of aluminum (the material of the current collectors of positive electrodes of LIBs) at high potentials (>3.8 V vs. Li/Li<sup>+</sup> reference electrodes) [6, 7]. The use of lithium bis(trifluoromethanesulfonyl)imide in electrolyte solutions for LIBs is therefore problematic, but lithium bis(trifluoromethanesulfonyl)imide could be in demand for electrolyte systems of low-voltage LBs (e.g., Li–S and Li–O<sub>2</sub>) [1, 8]. The transport and electrochemical properties of LiTFSI solutions in carbonate solvents [9–12], acetonitrile [13], and glymes [14, 15] have been extensively studied so far.

Sulfones are promising DASes for LIB and LB electrolytes. The interest in sulfones as electrolyte solvents is due mainly to their high anodic (oxidative) stability [3, 16–20]. The limit of anodic stability is thus around 5.8 V (vs. Li/Li<sup>+</sup>) for LiTFSI solutions in sulfolane, ethyl methyl sulfone, and ethyl-*cis*-butyl sulfone [21–23]; in linear and cyclic sulfones containing ester functional groups (bifunctional sulfones), it is

4.9–5.6 V on a Pt electrode (vs. Li/Li<sup>+</sup>) [24]. Electrolyte solutions of LiTFSI in sulfones [15, 25–27] and mixtures of sulfones with carbonates [28], glymes [8], and polymer [29] and composite electrolytes [30] containing sulfones as plasticizers, have recently been studied.

High thermal stability is another property that makes sulfonic electrolytes compare favorably to electrolyte systems based on other DASes. Most sulfones have high boiling and flash points (287.3 and 165°C for sulfolane, respectively [31]). The flash point of a 0.75 *m* LiTFSI solution in sulfolane is 151°C [25]. Electrolyte systems based on sulfones combined with thermally stable salts are therefore classified as safe and nonflammable [3].

Despite considerable interest in LiTFSI–sulfolane electrolyte systems, experimental studies of their physicochemical properties are still fragmentary and incomplete. The region of dilute solutions remains poorly understood, so information about the degree of association of lithium bis(trifluoromethanesulfonyl)imide in sulfones is largely speculative. The aim of this work was thus a detailed study of the main physicochemical properties (ionic conductivity, viscosity, density, and thermal and electrochemical stability) of lithium bis(trifluoromethanesulfonyl)imide solutions in sulfolane in wide ranges of concentration and temperature.

## EXPERIMENTAL

Tetramethylene sulfone (sulfolane; Sigma Aldrich, 99%) purified via vacuum distillation was used in this work. Lithium bis(trifluoromethanesulfonyl)imide (anhydrous, 99.99%, Sigma-Aldrich) was used without further purification or drying. Electrolyte solutions were prepared volumetrically and gravimetrically in a glove box in a medium of dried air (dew point, –56°C). A detailed procedure for preparing electrolyte solutions is described below in Supplementary Information.

The content of water in the initial components and electrolyte solutions was determined via coulometric titration in a medium of Fischer reagent using a Titroline 7500 KF trace automatic titrator (SI Analytics, Germany). According to results from coulometric titration, the content of water in a 1 M LiTFSI solution in sulfolane was 25 ± 4 ppm.

Thermogravimetric studies of solid samples and electrolyte solutions were performed on an upgraded Paulik–Paulik–Erdei MOM-1000 derivatograph (Hungary) at a heating rate of 5 K/min.

Differential scanning calorimetry (DSC) studies were conducted on a DSC 214 Polyma differential scanning calorimeter (Netzsch, Germany) in an argon atmosphere in the –70 to +50°C range of temperatures at a cooling/heating rate of 1 K/min. The DSC thermograms were recorded by heating a sample from

room temperature to +50°C, keeping it under isothermal conditions for 30 min, and then cooling it to –70°C and holding it at that temperature for 1 h. After cooling, the sample was again heated to +50°C and kept at that temperature for 30 min. The temperature scanning procedure was then repeated three times in this mode. In processing the DSC thermograms, it was assumed according to [32–34] that the temperature of the beginning of the first endothermic peak was the solidus temperature, while that of the maximum of the second endothermic peak in the heating curves was the liquidus temperature.

The electrochemical stability of electrolyte solutions was studied via cyclic voltammetry in a three-electrode glass cell with a platinum working electrode ( $S = 0.1 \text{ cm}^2$ ) in the –0.1 to 6 V range of potentials vs. a Li/Li<sup>+</sup> reference electrode at a temperature of 30°C. The rate of a potential sweep was 2 mV/s.

The physicochemical properties of electrolyte solutions were studied as described in [35]. Specific ionic conductivity was determined in conductometric cells with platinized platinum electrodes calibrated against KCl solutions. Kinematic viscosity was determined in Ubbelohde viscometers upgraded to operating with anhydrous solutions. Density was measured pycnometrically.

The dynamic viscosity of solutions was calculated according to kinematic viscosity and density using the formula

$$\eta = \nu \rho,$$

where  $\nu$  is the kinematic viscosity of the solution, cSt;  $\rho$  is the density of the solution, g/cm<sup>3</sup>.

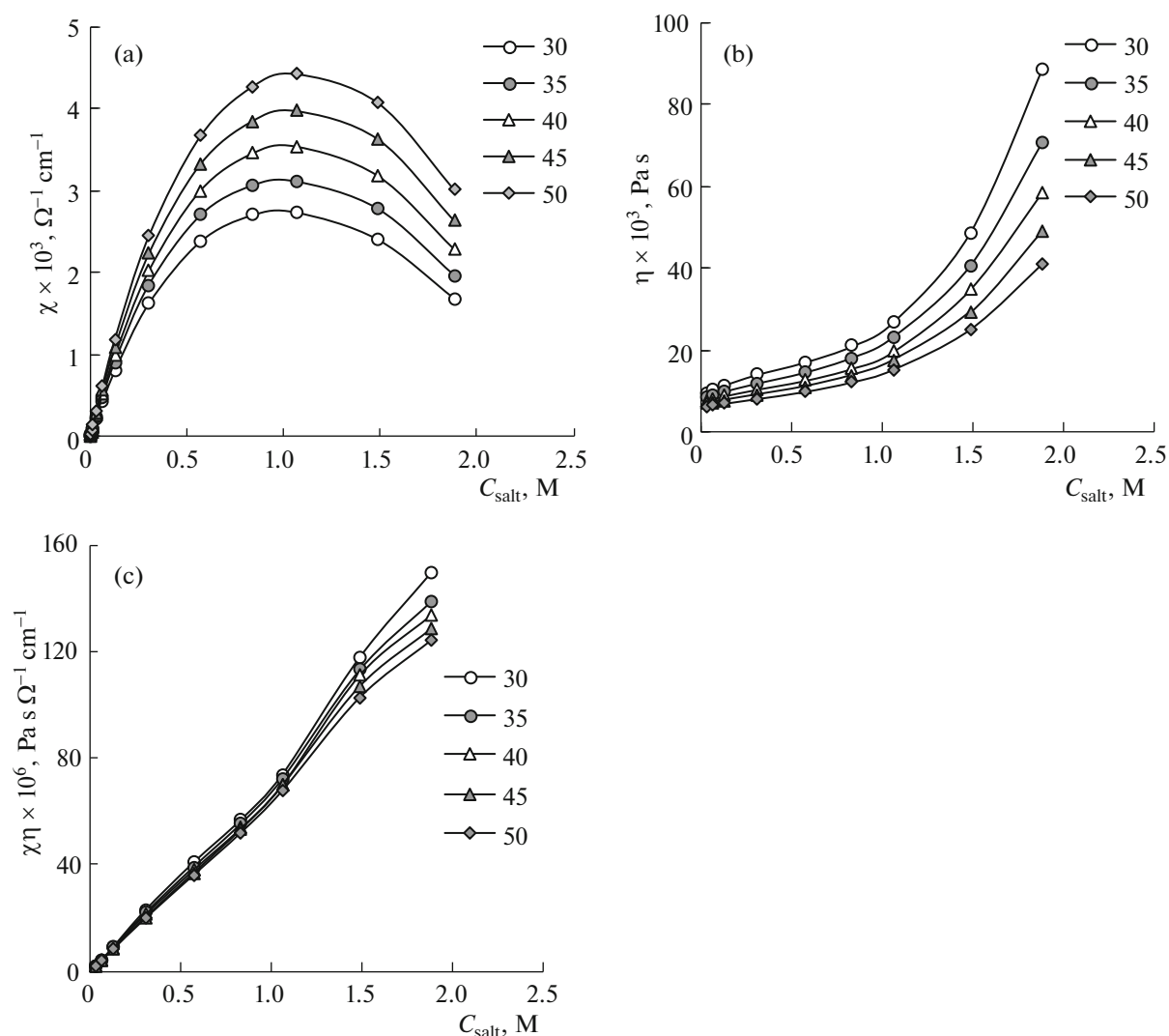
The errors in determining physicochemical properties were 0.5–1.0%.

Limiting equivalent conductivity ( $\lambda_0$ ) and constants of association ( $K_{\text{ass}}$ ) were determined from the equivalent conductivity of dilute electrolyte solutions using the Krauss–Bray formula [36].

The degree of electrolytic dissociation ( $\alpha$ ) of sulfolane solutions was determined as the ratio of the product of equivalent conductivity times viscosity to the product of limiting equivalent conductivity times the viscosity of the solvent:

$$\alpha = \frac{\lambda_C \eta_C}{\lambda_0 \eta_0}, \quad (1)$$

where  $\lambda_C$  is the equivalent conductivity of a solution with concentration  $C$ ;  $\eta_C$  is the dynamic viscosity of a solution with concentration  $C$ ;  $\lambda_0$  is the equivalent conductivity at infinite dilution; and  $\eta_0$  is the dynamic viscosity of the solvent.



**Fig. 1.** Isotherms of (a) ionic conductivity, (b) dynamic viscosity, and (c) corrected ionic conductivity of  $\text{LiN}(\text{SO}_2\text{CF}_3)_2$  solutions in sulfolane. Temperatures are indicated by the symbols ( $^{\circ}\text{C}$ ).

The energies of activation for ionic conductivity and viscous flow were calculated according to the Arrhenius equations

$$\eta = \eta_0 \exp \left[ \frac{1}{R} \left( \frac{E_{a\eta}}{T} \right) \right], \quad (2)$$

$$\chi = \chi_0 \exp \left[ \frac{1}{R} \left( \frac{E_{a\chi}}{T} \right) \right]. \quad (3)$$

## RESULTS AND DISCUSSION

### *Physicochemical and Electrochemical Properties of Sulfolane Solutions of Lithium Bis(trifluoromethanesulfonyl)imide*

Isotherms of the ionic conductivity, viscosity, and density of lithium bis(trifluoromethanesulfonyl)imide solutions in sulfolane are shown in Fig. 1 and Table 1S

in the below Supplementary Information. The ionic conductivity isotherms of  $\text{LiTFSI}$  sulfolane solutions exhibit maxima in the 0.9–1.1 mol/L range of salt concentrations, and the positions of the maxima are almost independent of temperature (Fig. 1a). The dynamic viscosity isotherms are not linear: there was an abrupt increase in viscosity at 0.8–1.1 M concentrations of salt (Fig. 1b). The isotherms of corrected ionic conductivity (specific ionic conductivity normalized to unit viscosity) are almost linear up to a 1.0 M concentration of salt. Kinks in the corrected ionic conductivity isotherms are observed at higher concentrations (Fig. 1c).

The ionic conductivity of the solutions grows along with temperature, while viscosity falls. The ionic conductivity corrected for viscosity falls as the temperature rises. The linearity of the temperature dependences of the ionic conductivity and viscosity of sulfo-

**Table 1.** Energies of activation for ionic conductivity and viscous flow ( $\text{kJ mol}^{-1}$ ) and coefficients of transmission for  $\text{LiN}(\text{SO}_2\text{CF}_3)_2$  solutions in sulfolane in the 30–50°C range of temperatures

Parameter	$C_{\text{salt}}, \text{M}$								
	0.031	0.063	0.126	0.303	0.570	0.836	1.064	1.490	1.890
$n$	338.5	165.6	82.1	33.2	17.0	11.1	8.4	5.5	4.1
$E_{\text{act}}(\chi) \pm 0.1$	16.2	16.0	16.0	16.5	17.5	18.6	19.6	21.5	23.7
$E_{\text{act}}(\eta) \pm 0.1$	17.8	18.9	20.5	22.5	22.6	22.3	23.1	27.0	31.1
$E_{\text{act}}(\chi)/E_{\text{act}}(\eta)$	0.91	0.84	0.78	0.73	0.77	0.83	0.85	0.80	0.76

$n$  is the solvation number.

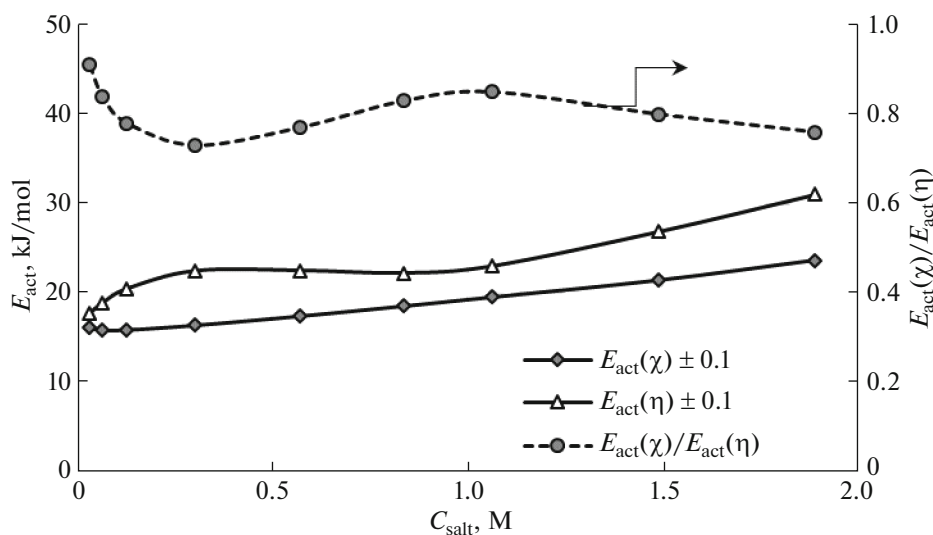
lane solutions of LiTFSI in the 30–50°C range in the coordinates of the Arrhenius equation allowed us to calculate the energies of activation for ionic conductivity and viscous flow and the coefficients of transmission (Table 1). The data suggest that the variation in the energies of activation for ionic conductivity and viscous flow and the coefficients of transmission has a complex pattern as a function of the concentration of the solution (Fig. 2). Raising the concentration of salt led to a weakly nonlinear increase in the energy of activation for ionic conductivity. The energy of activation for the viscous flow initially rose upon raising the concentration of salt. It then stabilized and began to rise again at a LiTFSI concentration of around 1 M. The concentration dependence of the coefficient of transmission exhibits antitabate behavior with the concentration dependence of the energy of activation for viscous flow.

The constants of association and the values of the limiting molar conductivity were determined according to the Krauss–Bray formula using isotherms of the

molar conductivity of dilute sulfolane solutions of LiTFSI (Table 2). According to calculations, the limiting equivalent conductivity ( $\lambda_0$ ) of LiTFSI solutions and the constant of association of the salt ( $K_{\text{ass}}$ ) rise along with temperature, while the corrected equivalent conductivity falls.

The calculated degrees of dissociation initially fell as the concentration of salt rose (Fig. 3). They began to rise again at a concentration of 0.6 M (a salt : sulfolane molar ratio of  $\approx 16$ ) and reach a maximum at a concentration of around 1.5 M (a salt : sulfolane molar ratio of  $\approx 6$ ).

The shape of the corrected ionic conductivity isotherms suggests that the main reason for the maxima in the ionic conductivity isotherms was an increase in the viscosity of the electrolyte solutions along with the concentration of salt. The probable cause of the drop in corrected ionic conductivity could be a rise in the degree of association of LiTFSI, due to degradation of the solvent's structure upon an increase in temperature.

**Fig. 2.** Concentration dependences of the energies of activation for ionic conductivity and viscous flow, and the coefficients of transmission for  $\text{LiN}(\text{SO}_2\text{CF}_3)_2$  solutions in sulfolane.

**Table 2.** Limiting equivalent conductivity ( $\lambda_0$ ) and constants of association ( $K_{\text{ass}}$ ) for  $\text{LiN}(\text{SO}_2\text{CF}_3)_2$  in sulfolane

Parameter	$T, ^\circ\text{C}$				
	30	35	40	45	50
$\lambda_0 \times 10^4, \Omega^{-1} \text{ m}^2 \text{ mol}^{-1}$	7.4	8.4	9.4	10.4	11.4
$\lambda_0 \eta_0 \times 10^6, \Omega^{-1} \text{ m}^2 \text{ mol}^{-1} \text{ Pa s}$	75.9	75.9	75.4	74.4	73.0
$K_{\text{ass}}$	1.67	1.75	1.79	1.82	1.85

The maxima in the ionic conductivity isotherms and the complex shape of the concentration dependences of the energies of activation for viscous flows and the coefficients of transmission suggest the concentration of salt has a strong effect on the structure of the resulting sulfolane solutions of LiTFSI.

Sulfolane solutions of  $\text{LiN}(\text{SO}_2\text{CF}_3)_2$  are characterized by a wide window of electrochemical stability. According to cyclic voltammetry, the anodic stability of a 1 M  $\text{LiN}(\text{CF}_3\text{SO}_2)_2$  solution in sulfolane on a platinum electrode was 5.75 V vs. the lithium reference electrode (Fig. 4).

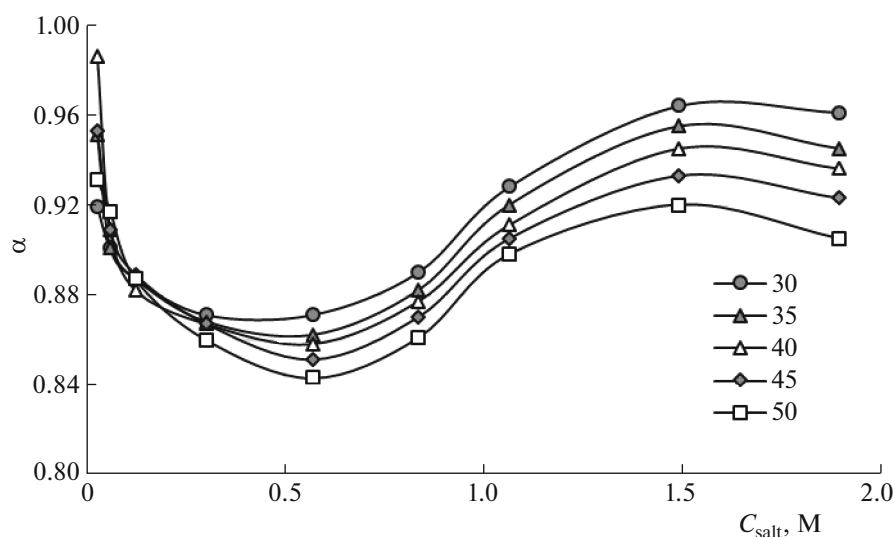
#### *Thermal Properties of Sulfolane Solutions of Lithium Bis(trifluoromethanesulfonyl)imide*

Lithium bis(trifluoromethanesulfonyl)imide exhibits high thermal stability. Its thermal degradation begins at temperatures above 320°C. It occurs in a single stage and is accompanied by a strong exothermic effect (Fig. 5). Sulfolane is a high-boiling liquid; under conditions thermogravimetric analysis, it evaporates with no thermochemical degradation. When 1 M  $\text{LiN}(\text{SO}_2\text{CF}_3)_2$  solution in sulfolane is heated, the

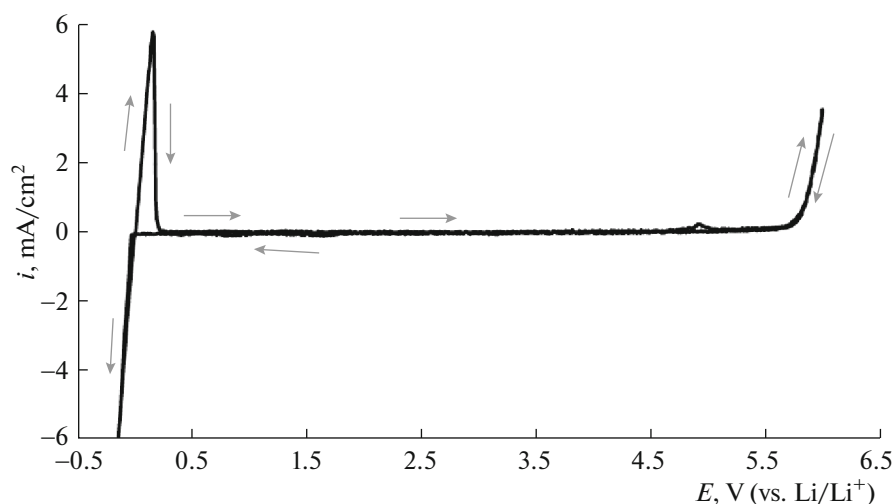
sulfolane evaporates before the thermal degradation of the salt. It should be noted that sulfolane's temperature of evaporation from a salt solution is higher than that of pure sulfolane, and the difference between the temperatures of evaporation for only sulfolane and sulfolane from solution grows along with the concentration of salt. According to the shape of the differential thermal analysis (DTA) curves, the sulfolane does not interact chemically with salt anions during heating.

#### *Low-Temperature Properties of Sulfolane Solutions of Lithium Bis(trifluoromethanesulfonyl)imide*

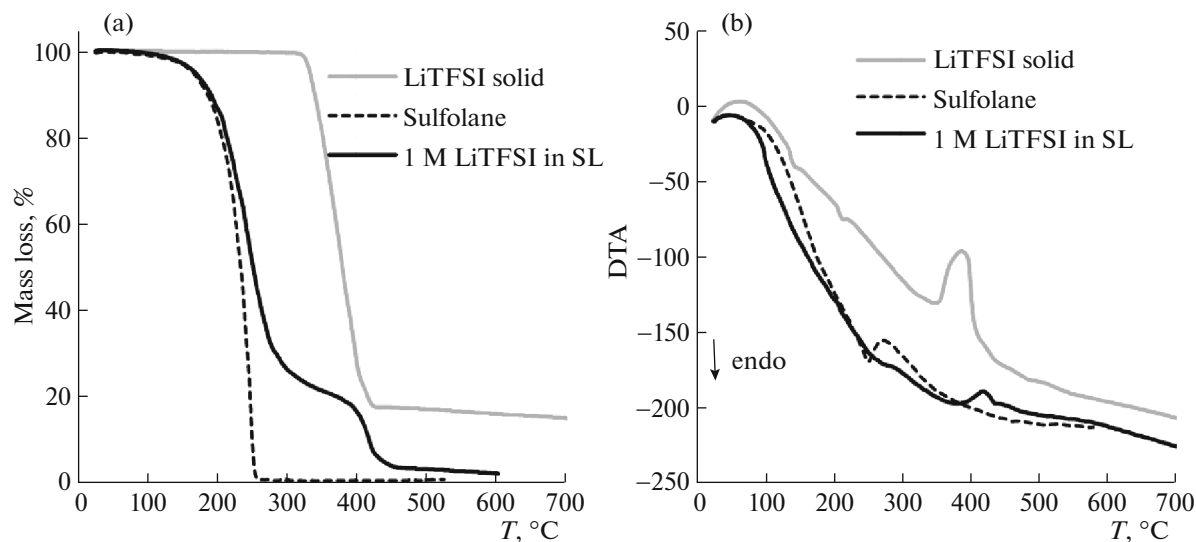
Under normal conditions, sulfolane is in a crystalline state. The heating curve of sulfolane (Fig. 6) exhibits two endothermic effects corresponding to the transition from the crystalline to the mesomorphic phase (14.8°C) and the melting of the mesomorphic phase (27.7°C) [34, 37–39]. The cooling curves of sulfolane also exhibit two thermal effects, but their temperatures differ considerably from those of the thermal effects in the heating curves. The first exothermic effect in the cooling curves is observed at a temperature of 25°C; the second effect, at a temperature of –10°C. The first exothermic effect apparently corre-



**Fig. 3.** Concentration dependences of the degree of electrolytic dissociation ( $\alpha$ ) of  $\text{LiN}(\text{SO}_2\text{CF}_3)_2$  in sulfolane at different temperatures indicated by the symbols ( $^\circ\text{C}$ ).



**Fig. 4.** Cyclic voltammogram of a 1 M  $\text{LiN}(\text{SO}_2\text{CF}_3)_2$  solution in sulfolane ( $30^\circ\text{C}$ ) on a Pt electrode vs.  $\text{Li}/\text{Li}^+$ . The rate of the potential sweep is 2 mV/s.



**Fig. 5.** Curves of (a) mass loss and (b) thermal effects (DTA) of LiTFSI (solid), sulfolane, and a 1 M LiTFSI solution in sulfolane (SL). The rate of heating is 5 K/min.

sponds to the transition of sulfolane from the liquid state to the mesomorphic phase, while the second effect corresponds to the transition from the mesomorphic to the crystalline phase. Strong differences in the temperatures of the phase transition during the heating and cooling of sulfolane suggest that it has a tendency to form supercooled states.

The DSC thermograms of dilute sulfolane solutions of LiTFSI also exhibit two peaks each. These peaks correspond to the melting of the crystalline and mesomorphic phases of sulfolane (Fig. 6, Table 2S in Supplementary Information). The melting points of

the crystalline and mesomorphic phases fall as the concentration of salt rises. Solutions of LiTFSI in sulfolane also have a tendency to form supercooled states. A highly concentrated (1.89 M) LiTFSI solution exhibits interesting properties. No thermal effects are observed in the cooling curves of this solution (Fig. 6), but the heating curves initially exhibit an exothermic effect at a temperature of  $-45.5^\circ\text{C}$ , and then an endothermic effect at a temperature of  $-6.3^\circ\text{C}$ . The experimental data suggest that the tendency of sulfolane solutions of LiTFSI to form supercooled states grows along with their concentrations.

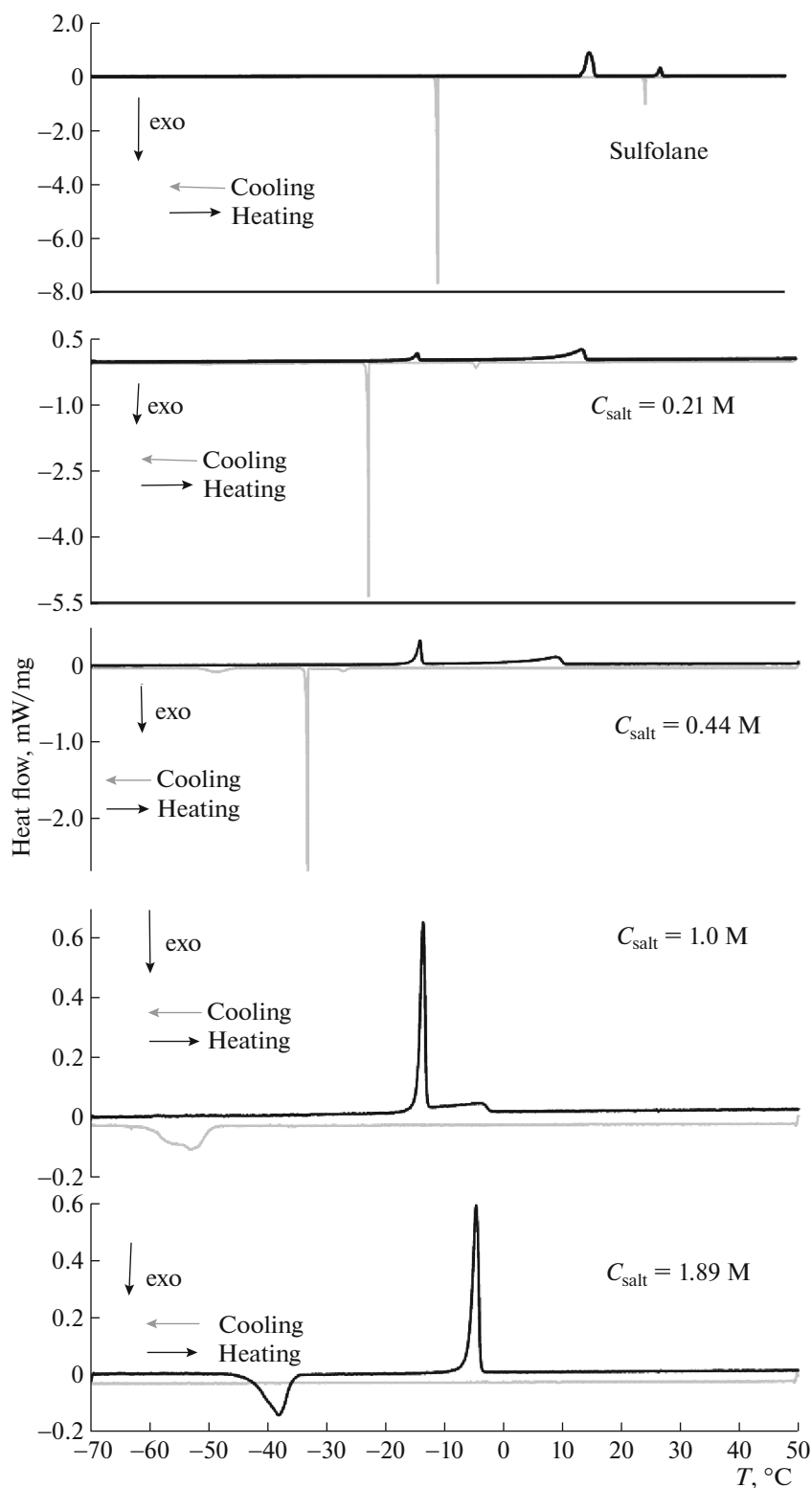


Fig. 6. Differential scanning calorimetry thermograms of sulfolane and  $\text{LiN}(\text{SO}_2\text{CF}_3)_2$  solutions in sulfolane.

## CONCLUSIONS

Our studies have shown that lithium bis(trifluoromethanesulfonyl)imide solutions in sulfolane exhibit

high thermal stability, electrochemical stability in a wide range of electrode potentials, and moderate ionic conductivity comparable to that of sulfolane solutions

of lithium salts with such bulky anions as those of lithium perchlorate and lithium hexafluorophosphate.

The structure and transport properties of sulfolane solutions of lithium bis(trifluoromethanesulfonyl)imide are largely determined by their concentrations. Lithium bis(trifluoromethanesulfonyl)imide is weakly associated in dilute solutions. The degree of electrolytic dissociation of  $\text{LiN}(\text{SO}_2\text{CF}_3)_2$  initially falls and then starts to rise along with the concentrations of the solutions. The rise in the degree of electrolytic dissociation of  $\text{LiN}(\text{SO}_2\text{CF}_3)_2$  along with the concentration of the solution can be attributed to the formation of ion triplets and more complex ion associates.

The mechanism of ion transport also depends largely on the concentration of a solution. In dilute solutions, ion transport mostly proceeds in accordance with the Stokes mechanism. The mechanism of ion transport changes upon an increase in the concentration of the  $\text{LiN}(\text{SO}_2\text{CF}_3)_2$  solution. According to the pattern of change in the coefficients of transmission, the contribution of the relay-race ion transport mechanism initially rises and then falls upon an increase in the concentration of salt. It then begins to rise again at concentrations of salt greater than 1 M.

Since sulfolane is (like most strongly polar solvents) an associated solvent, temperature has a great effect on the thermodynamic and transport properties of  $\text{LiN}(\text{SO}_2\text{CF}_3)_2$  solutions. An increase in temperature raises the constants of association and limit equivalent conductivity of  $\text{LiN}(\text{SO}_2\text{CF}_3)_2$  solutions due to a drop in the degree of self-association and degradation of the sulfolane structure.

A specific feature of sulfolane solutions of lithium bis(trifluoromethylsulfonyl)imide is their tendency to form supercooled solutions.

According to their set of physicochemical, electrochemical, and thermal properties, lithium bis(trifluoromethanesulfonyl)imide solutions can be considered promising electrolyte systems for medium-temperature energy-intensive lithium-ion and lithium energy storage devices.

#### SUPPLEMENTARY INFORMATION

The online version of this work contains supplementary material available at <https://doi.org/10.1134/S0036024424030269>.

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#### ETHICS APPROVAL AND CONSENT TO PARTICIPATE

This work did not involve any studies using human and animal subjects.

#### CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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