

Characteristics of the electrolyte with fluoro organic lithium salts

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Abstract

Some fluoro organic lithium salts have attractive features as organic electrolyte such as solubility, conductivity, and oxidation potentials. The fluoro organic lithium salts with SO₂ groups show higher conductivities than those with CO groups. Methide (CF₃SO₂)₃CLi and imide (RfSO₂)₂NLi salts show higher conductivities than oxide salts RfSO₂OLi with only one RfSO₂ group. The lithium salts with long fluoroalkyl groups and two or more RfSO₂ groups show high oxidation potentials and high aluminum corrosion potentials over 4.3 V. The battery performances of modified imide salts such as (C₂F₅SO₂)₂NLi, (C₄F₉SO₂)(CF₃SO₂)NLi, and ((CF₃)₂CHOSO₂)₂NLi are satisfactory. The cycle characteristics of the imide salt cells are better than those of the LiPF₆ cell. Especially, the ((CF₃)₂CHOSO₂)₂NLi shows the best cycle characteristics. In XPS analysis the anode surfaces of the imide cells are covered with imide salt components. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Fluoro organic lithium salt; Conductivity; Oxidation potential; Aluminum corrosion potential; Cycle characteristics

1. Introduction

Many types of organic lithium salts such as LiB(C₆H₅)₄, Li₂C₄O₄, LiSO₃C₂F₄SO₃Li, CF₃SO₃Li, CF₃CO₂Li, C₄F₉SO₃Li, (CF₃SO₂)₂NLi, (CF₃CO)₂NLi, (CF₃SO₂)₃CLi, (CF₃SO₂)₂CFLi, (C₂F₅SO₂)₂NLi, LiB(C₆H₄O₂)₂, and LiB(COC₆H₄O)₂ have been reported as lithium battery electrolyte [1–9]. Among them CF₃SO₃Li, C₄F₉SO₃Li, and (CF₃SO₂)₂NLi have been used or were used as lithium battery electrolytes.

The following features are required for lithium salts as electrolytes.

1. Good conductivity
2. Good electrochemical and thermal stability (high oxidation potential)
3. Safety and nontoxicity
4. Low cost

The most advantageous features of organic lithium salts are their characteristics to be improved by structural modification. We report here the structural effects of fluoro organic lithium salts on the conductivity, electrochemical stability, aluminum corrosion at high voltage, and cell performance

2. Experimental

2.1. HOMO calculation

The energy levels for the highest occupied molecular orbital (HOMO) of different kinds of geometry-optimized electrolyte anions were calculated with MNDO, PM3, HF/3-21G*, and B3LYP/6-31G* || HF/3-21G* methods by SPARTAN V 5.0.

2.2. Conductivity

The conductivities of several kinds of electrolyte solutions (0.1 mol/l lithium salts in 1/2 v/v PC/DME) were

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Table 1

Conductivities of electrolytes with fluoro organic lithium salts at 25°C
0.1 mol/l salts in PC/DME(1/2 v/v).

Lithium salt	Conductivity (mS/cm)	Molecular weight
$(C_2F_5)_2P(=O)OLi$	0.6	308
CF_3CO_2Li	0.4	120
$(CF_3CO)_2NLi$	0.8	215
CF_3SO_3Li	2.3	156
CH_3SO_3Li	Practically insoluble	102
$C_4F_9SO_3Li$	2.3	306
$C_6F_5SO_3Li$	1.1	254
$C_6H_5SO_3Li$	0.1–0.2 (ca. 0.02 mol/l)	164
$C_8F_{17}SO_3Li$	1.9	506
$(CF_3SO_2)_2NLi$	4.0	287
$(C_2F_5SO_2)_2NLi$	3.8	387
$(C_4F_9SO_2)(CF_3SO_2)NLi$	3.5	437
$(FSO_2C_6F_4)(CF_3SO_2)NLi$	3.0	347
$(C_8F_{17}SO_2)(CF_3SO_2)NLi$	3.2	637
$(CF_3CH_2OSO_2)_2NLi$	3.0	347
$(CF_3CF_2CH_2OSO_2)_2NLi$	3.0	447
$(HCF_2CF_2CH_2OSO_2)_2NLi$	2.9	411
$((CF_3)_2CHOSO_2)_2NLi$	3.1	483
$(CF_3SO_2)_3CLi$	3.6	418
$(CF_3CH_2OSO_2)_3CLi$	2.9	508
$LiTFPB^a$	2.7	870
$LiPF_6$	4.4	152

^a $LiB[C_6H_3(CF_3)_2-3,5]_4$.

measured by a conductance meter (CM40S, Toa Electronics).

2.3. Oxidation potential and aluminum corrosion potential

Anodic oxidation potentials were measured by potential sweep method. Platinum wire (0.3-mm \varnothing , Nilaco) was used for working electrode, and 21-mm-wide lithium foil (Kyokuto) was used for counter electrode (substantial area = 0.2 cm²). A polyethylene separator was placed between the working electrode and the counter electrode. Cell potential was swept at a rate of 50 mV/s with a potentiostat (HA-501, Hokuto Denko) and a function generator

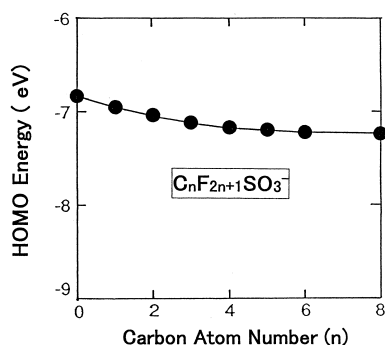


Fig. 1. The effects of alkyl-chain length on calculated HOMO energy levels by MNDO method.

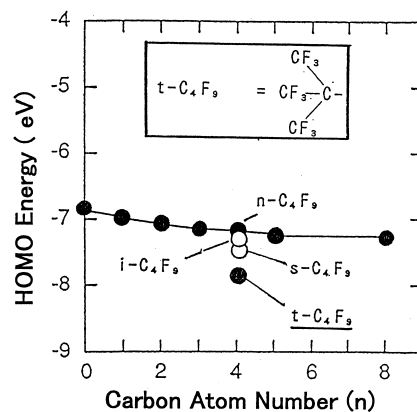


Fig. 2. The effects of branched structure on calculated HOMO energy levels by MNDO.

(HB-104, Hokuto Denko). The potential at a current density of 0.5 mA/cm² was regarded as the oxidation potential.

Aluminum dissolution potential was measured at a rate of 5 mV/s by using aluminum plate (1 cm × 1 cm).

2.4. Battery-performance test

LiCoO₂ was used as a cathode with PVDF binder and conductive carbon. Graphite was used as an anode with PVDF binder. The 14500 cell (14 mm diameter and 50 mm height) was made using LiPF₆ or lithium imide electrolyte. After precharging and aging, the capacity and cycle characteristics of the batteries were checked.

3. Results and discussion

3.1. Conductivity

The conductivities of several kinds of electrolyte solution (0.1 mol/l lithium salts in 1/2 v/v PC/DME) with polyfluorinated organic lithium salts at 25°C are shown in Table 1. The solubility of organic lithium salt in organic solvents for electrolyte is an important factor to be checked.

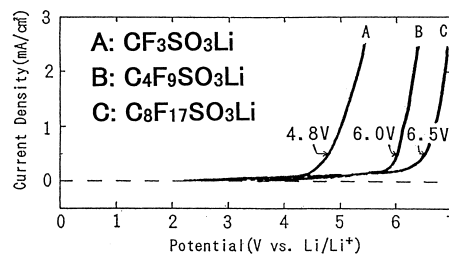


Fig. 3. The effects of alkyl-chain length on oxidation potential. Electrolyte: 0.1 mol/l Li salt/PC. Electrode: Pt (0.2 cm²), sweep rate: 50 mV/s.

Table 2

The HOMO energies and oxidation potentials for $(\text{CF}_3\text{SO}_2)_n\text{XLi}$ type electrolytes (X = O,N,C)

Lithium salts	HOMO energy of anion (eV)			Oxidation potential at 0.5 mA/cm ² (V vs. Li/Li+)
	PM3	HF/3-21G*	B3LYP/6-31G* HF/3-21G*	
$\text{CF}_3\text{SO}_3\text{Li}$	-6.39	-6.41	-1.98	4.8
$(\text{CF}_3\text{SO}_2)_2\text{NLi}$	-7.18	-7.61	-3.32	5.2
$(\text{CF}_3\text{SO}_2)_3\text{CLi}$	-8.15	-8.00	-3.78	5.3

In general, the organic lithium salts with no fluorine atoms and with no conjugated resonance structures show low solubility.

The conductivity of electrolytes relates to the mobility and concentration of dissociated ions. The mobility of organic anions decreases with the increase of their molecular size. However, as shown in Table 1, the conductivity of LiTFPB whose molecular weight is six times larger than that of $\text{CF}_3\text{SO}_3\text{Li}$ is higher than that of $\text{CF}_3\text{SO}_3\text{Li}$. This indicates that ion dissociation is another important factor for organic electrolytes.

The imide and methide salts with two or three CF_3SO_2 groups show higher conductivities than $\text{CF}_3\text{SO}_3\text{Li}$ with only one CF_3SO_2 group [$(\text{CF}_3\text{SO}_2)_2\text{NLi}$ (4.0 mS/cm), $(\text{CF}_3\text{SO}_2)_3\text{CLi}$ (3.6 mS/cm) > $\text{CF}_3\text{SO}_3\text{Li}$ (2.3 mS/cm)].

The $\text{CF}_3\text{SO}_2\text{OLi}$ with SO_2 group shows higher conductivity than CF_3COOLi with CO group and $(\text{C}_2\text{F}_5)_2\text{POOLi}$ with PO_x group [$\text{CF}_3\text{SO}_3\text{Li}$ (2.3 mS/cm) > $\text{CF}_3\text{CO}_2\text{Li}$ (0.4 mS/cm), $(\text{C}_2\text{F}_5)_2\text{POOLi}$ (0.6 mS/cm)].

The length of fluoroalkyl groups also affects the conductivity. The conductivities of $\text{CF}_3\text{SO}_3\text{Li}$, $\text{C}_4\text{F}_9\text{SO}_3\text{Li}$, and $\text{C}_8\text{F}_{17}\text{SO}_3\text{Li}$ are 2.3, 2.3, and 1.9 mS/cm, respectively.

These results show that the lithium salts with more than two RfSO_2 groups with fluoroalkyl groups from CF_3 to C_4F_9 are preferable for high conductivity.

3.2. Electrochemical stability

The oxidation stability is estimated by HOMO energy level calculation. The high oxidation potential is for the

anions with low HOMO energy level. As shown in Figs. 1 and 2, the anions of organic lithium salts with longer or branched fluoroalkyl groups show lower HOMO energy levels. We measured the oxidation potentials by the potential sweep method on Pt electrode in 0.1 mol/l lithium salts/PC electrolyte (50 mV/s). The potential-current profiles of $\text{C}_n\text{F}_{2n+1}\text{SO}_3\text{Li}$ ($n = 1, 4, 8$) electrolytes are shown in Fig. 3. The HOMO levels of the corresponding anions correlate well with the measured oxidation potentials in Fig. 3.

The number of CF_3SO_2 groups in lithium salts also affects their oxidation potentials. Table 2 shows HOMO energies and measured oxidation potentials for $(\text{CF}_3\text{SO}_2)_n\text{XLi}$ electrolyte (X = O,N,C). The anion of $(\text{CF}_3\text{SO}_2)_3\text{CLi}$ shows the lowest HOMO energy among the anions with CF_3SO_2 groups.

The oxidation potentials of $\text{CF}_3\text{SO}_3\text{Li}$, $(\text{CF}_3\text{SO}_2)_2\text{NLi}$, and $(\text{CF}_3\text{SO}_2)_3\text{CLi}$ are 4.8, 5.2, and 5.3 V, respectively, in 0.1 mol/l lithium salt/PC electrolyte. Table 2 shows a good correlation between the HOMO energies and the measured oxidation potentials for $(\text{CF}_3\text{SO}_2)_n\text{XLi}$ type electrolyte (X = O,N,C, $n = 1, 2, 3$).

Next we examined the effects of the length of fluoroalkyl groups on the oxidation potentials of the imide electrolytes. Imide electrolytes with longer fluoroalkyl groups such as C_4F_9 and C_8F_{17} groups show their high oxidation potentials. The oxidation potentials of $(\text{CF}_3\text{SO}_2)_2\text{NLi}$, $(\text{C}_4\text{F}_9\text{SO}_2)(\text{CF}_3\text{SO}_2)\text{NLi}$, and $(\text{C}_8\text{F}_{17}\text{SO}_2)(\text{CF}_3\text{SO}_2)\text{NLi}$ are 5.2, 5.9, and 6.0 V, respectively.

We also checked the oxidation stability of new ester-type imide electrolytes such as $((\text{CF}_3)_2\text{CHOSO}_2)_2\text{NLi}$. In Table 3, with increase in number of F atoms of imide ester salts, their HOMO energy levels are decreased. Imide ester salt $(\text{CF}_3\text{CH}_2\text{OSO}_2)_2\text{NLi}$ shows a higher oxidation potential than imide salt $(\text{CF}_3\text{SO}_2)_2\text{NLi}$. Branched imide ester salt $((\text{CF}_3)_2\text{CHOSO}_2)_2\text{NLi}$ shows the highest oxidation potential (5.8 V) among the measured imide ester salts.

The reduction stability is predicted by the LUMO energy level calculation. The lithium salts with low HOMO energy level always show low LUMO energies as shown in Fig. 4. We checked the cycle life of fluoro organic lithium salts in EC:Diox (1/2 v/v) to compare their

Table 3

The HOMO energies and the oxidation potentials for imide ester salts $(\text{RfOSO}_2)_2\text{NLi}$ electrolytes

Lithium salts	HOMO energy of anion (eV)			Oxidation potential at 0.5 mA/cm ² (V vs. Li/Li+)
	PM3	HF/3-21G*	B3LYP/6-31G* HF/3-21G*	
$(\text{CF}_3\text{CH}_2\text{OSO}_2)_2\text{NLi}$	-6.89	-7.32	-3.13	5.4
$(\text{HCF}_2\text{CF}_2\text{CH}_2\text{OSO}_2)_2\text{NLi}$	-6.63	-7.33	-3.27	5.5
$(\text{CF}_3\text{CF}_2\text{CH}_2\text{OSO}_2)_2\text{NLi}$	-6.95	-7.63	-3.40	5.6
$((\text{CF}_3)_2\text{CHOSO}_2)_2\text{NLi}$	-7.29	-8.03	-3.84	5.8

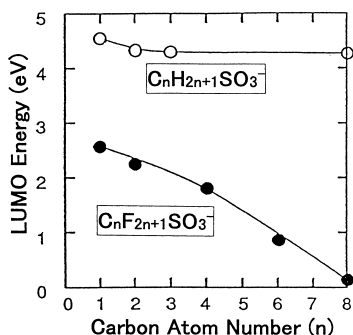


Fig. 4. The effects of alkyl-chain length on calculated LUMO energy levels by MNDO.

reactivity with lithium. In Table 4, $C_4F_9SO_3$ anion and $(CF_3SO_2)_2N$ anion with lower LUMO energy level show good cycle life compared with CF_3SO_3 anion. This result suggests that the surface passivation film on lithium foil might suppress the reaction of electrolyte with lithium and also decrease the dendritic lithium formation.

3.3. Aluminum corrosion

Some organic lithium salts such as CF_3SO_3Li and $(CF_3SO_2)_2NLi$ dissolve aluminum (Al) positive current collector at 4.2 V in PC electrolyte, shown in Table 5. These salts are therefore difficult to use in common lithium ion batteries charged over 4.2 V. However, imide salts with longer fluoroalkyl groups and methide salts show corrosion potentials of more than 4.3 V. The aluminum dissolution potentials of CF_3SO_3Li , $(CF_3SO_2)_2NLi$, $(CF_3SO_2)_3CLi$, $(CF_3CH_2OSO_2)_3CLi$, $(C_2F_5SO_2)_2NLi$, $(C_4F_9SO_2)(CF_3SO_2)NLi$, and $((CF_3)_2CHOSO_2)_2NLi$ are 3.1, 4.0, 4.6, 5.5, 4.5, 4.8, and 4.3 V, respectively, in 0.1 mol/l Li salt/. These corrosion potentials are well correlated with anion size.

3.4. Cell performance with imide salts

We investigated the structural effects of the imide salt electrolytes on cell performance by using some imide salts with corrosion potentials over 4.3 V and good conductivities.

Table 4

Cycle life of lithium anode in EC:diox with fluoro-organic lithium salts 0.6 mol/l lithium salts in EC:Diox (1/2 v/v). CR2016-type coin cell (positive: Li_xMnO_2 , negative: lithium foil 0.05 mm).

Lithium salt	Cycle no. (F.O.M. of anode Li foil)		
	0.25 mA/cm ²	0.5 mA/cm ²	1.0 mA/cm ²
CF_3SO_3Li	102 (9.9)	115 (11.1)	85 (8.2)
$C_4F_9SO_3Li$	174 (16.8)	147 (14.2)	96 (9.3)
$(CF_3SO_2)_2NLi$	196 (18.9)	126 (12.2)	99 (9.6)

Table 5

Cell performance of 14500 cell with imide salts

Li salts ^a	Capacity (mAh) at 25°C		
	0.2 C (140 mA)	1 C (600 mA)	2 C (1200 mA)
$LiPF_6$	590(100)	577(97.8)	567(96.1)
$(C_2F_5SO_2)_2NLi$	589(100)	575(97.6)	571(96.9)
$(C_2F_5SO_2)_2NLi^b$	592(100)	576(97.2)	573(96.8)
$(C_4F_9SO_2)(CF_3SO_2)NLi^b$	592(100)	571(96.4)	566(95.6)
$(CF_3)_2CHOSO_2)_2NLi^b$	599(100)	580(96.8)	573(95.7)

^aElectrolyte; 1.0 mol/l Li salt/EC/MEC 1/2 v/v 14500 cell (14 mm \varnothing , 50 mm height, (+) $LiCoO_2$, (-) graphite). Charge: 600 mA h, 2.5 h, 4.1 V CCCV. Discharge: 140–600 mA, 2.75 V cut off.

^bWith 0.05 mol/l $LiPF_6$ added.

Since the conductivity (6.5 mS/cm) of 1.0 mol/l imide electrolyte ($(C_2F_5SO_2)_2NLi$) in EC:MEC (1/2 v/v) is a little lower than that of $LiPF_6$ (9.5 mS/cm), the impedance of the $(C_2F_5SO_2)_2NLi$ cell is higher than that of the $LiPF_6$ cell. However, the impedance increase of the imide cell after 20 days storage at 60°C is smaller than that of the $LiPF_6$ cell because of the better stability of the imide ($(C_2F_5SO_2)_2NLi$: 13%, $LiPF_6$: 22%). Moreover, the $(C_2F_5SO_2)_2NLi$ cell with 1.0 mol/l imide and 0.05 mol/l $LiPF_6$ shows only 9% impedance increase.

Fig. 5 and Table 5 show the battery discharge curves and capacities of imide cells and $LiPF_6$ cell at 0.2, 1.0, and 2.0 C rate. The discharge potentials of the imide cells are lower than that of $LiPF_6$ cell because of the difference of their conductivities, but discharge capacities of those imide cells are almost same even at 2 C rate.

The discharge capacities of $((CF_3)_2CHOSO_2)_2NLi$ cell are 599, 580, and 573 mAh at 0.2 C (140 mA), 1.0 C (600 mA), and 2.0 C (1200 mA), respectively, while those of $LiPF_6$ are 590, 577, and 567 mAh, respectively.

As shown in Fig. 6, the cycle characteristics of the $(C_2F_5SO_2)_2NLi$ cell with 1.0 mol/l lithium imide and 0.05 mol/l $LiPF_6$ is better than that of the $LiPF_6$ cell. The $(C_4F_9SO_2)(CF_3SO_2)NLi$ cell shows a lower capacity at

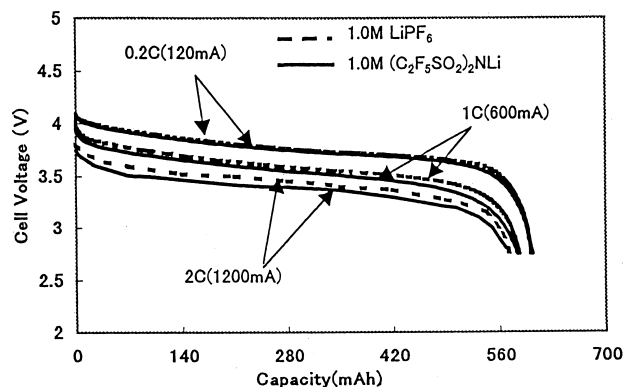


Fig. 5. Discharge curves of 14500 cell with $(C_2F_5SO_2)_2NLi$ salt. 14500 cell (14 mm \varnothing , 50 mm height, (+) $LiCoO_2$, (-) graphite).

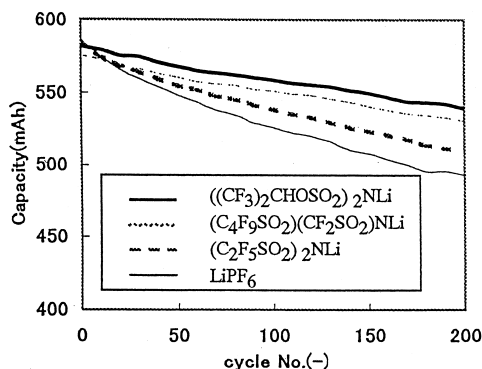


Fig. 6. Cycle characteristics of 14500 cells with imide salt. LiCoO₂/graphite-type cell of 14 mm Ø, 50 mm height; charge current = 600 mA for 2.5 h (4.1 V CCCV); discharge current = 600 mA (2.75 V cut off); electrolyte: 1.0 mol/l Li salt in EC/MEC (1/2 v/v).

1 C (600 mA) rate than the ((C₂F₅SO₂)₂NLi cell, while its cycle characteristic is better than that of the (C₂F₅SO₂)₂NLi cell. The ((CF₃)₂CHOSO₂)₂NLi cell shows the best cycle characteristics.

3.5. XPS analysis results of anode surface of the cells with imide salts

We analyzed the anode surface characteristics by XPS analysis to investigate the reason for the difference in cycle characteristics. Tables 6 and 7 show the atomic ratio of anode surface by XPS and the calculated atomic ratio from N atom ratio for imide salts. The anode surfaces of the imide cells are mainly covered by the imide salt component and organic compounds. For the ((CF₃)₂CHOSO₂)₂NLi cell, 28.8 at.% organic F atom should exist as the imide salt or its derivative on anode surface based on N ratio. This calculated ratio of 28.8% is almost equal to the observed value of 32.8% (based on F atom ratio). Total amount of atomic ratio for imide ester ((CF₃)₂CHOSO₂)₂NLi is 67 at.%, whereas that for LiPF₆ was only 30 at.%. These results suggest that the atoms of imide salts would exist on anode surface or are involved as a surface film with other organic compounds. In contrast to the imide-anode surface, LiPF₆-anode surface is covered with organic compounds, LiPF₆ component, LiF, Li salts and PVDF binder. The difference in anode surface compo-

Table 6

The atomic ratio of anode surface by XPS analysis
ESCA LAB Mark 2 (VG) Mg-Kα 12 kV, 10 mA.

Salt used in the cell	C	F	Li	S	N	O
LiPF ₆	33.0	19.6	10.7	0.0	0.0	32.6
(C ₂ F ₅ SO ₂) ₂ NLi	33.1	27.7	9.2	5.2	2.1	22.4
(C ₄ F ₉ SO ₂)(CF ₃ SO ₂)NLi	28.8	32.8	10.9	3.2	1.5	20.7
(CF ₃) ₂ CHOSO ₂) ₂ NLi	26.5	32.8	7.3	6.5	2.4	24.3

Table 7

The calculated atomic ratio based on N atomic ratio for imide salts

Salt used in the cell	C	F	Li	S	N	O
(C ₂ F ₅ SO ₂) ₂ NLi	8.4	21.0	2.1	4.2	2.1	8.4
(C ₄ F ₉ SO ₂)(CF ₃ SO ₂)NLi	15.0	18.0	1.5	3.0	1.5	6
((CF ₃) ₂ CHOSO ₂) ₂ NLi	14.4	28.8	2.4	4.8	2.4	14.4

nents might be important to elucidate various effects on cycle characteristics.

4. Conclusion

The activity characteristics of fluoro organic lithium salts can be summarized as follows.

- (1) The organic lithium salts with fluoro alkyl groups show good solubility in organic solvents.
- (2) The fluoro organic lithium salts with SO₂ groups show better conductivities than those with CO groups.
- (3) The fluoro organic lithium salts with more than two RfSO₂ groups such as methide and imide salts show better conductivities than oxide salts RfSO₂OLi with only one RfSO₂ group.
- (4) The lithium salts with long fluoroalkyl groups show high oxidation potentials. The oxidation potentials of CF₃SO₃Li, C₄F₉SO₃Li, C₈F₁₇SO₃Li, (CF₃SO₂)₂NLi, (C₄F₉SO₂)(CF₃SO₂)NLi, and (C₈F₁₇SO₂)(CF₃SO₂)NLi are 4.8, 6.0, 6.5, 5.2, 5.9, and 6.0 V, respectively.
- (5) Imide and methide salts with two or more RfSO₂ groups show higher oxidation potentials than oxide salts RfSO₂OLi with only one RfSO₂ group.
- (6) Imide ester salt such as ((CF₃)₂CHOSO₂)₂NLi shows higher oxidation potentials than that of (CF₃SO₂)₂NLi.
- (7) Imide salts with longer fluoroalkyl groups and methide salts show corrosion potentials more than 4.3 V. Especially, methide salt (CF₃CH₂OSO₂)₃CLi electrolyte shows the highest Al dissolution potential (5.5V vs. Li/Li+) in PC electrolyte.
- (8) The battery performances of imide salts are satisfactory. The cycle characteristics of the imide salts cell are better than those of LiPF₆ cell. The ((CF₃)₂CHOSO₂)₂NLi cell shows the best cycle characteristics.
- (9) In XPS analysis, the anode surfaces of imide cells are covered with imide salt components.

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