Near ambient pressure-x-ray photoelectron spectroscopy spectra of lithium bis (trifluoromethane-sulfonyl) imide in propylene carbonate

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Paul M. Dietrich, Lydia Gerlein, Julia Maibach, et al.

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Note: This paper is part of the 2023 Special Topic Collection on Materials for Energy and the Environment.
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ABSTRACT

Near ambient pressure–x-ray photoelectron spectroscopy (NAP-XPS) is a less traditional form of XPS that allows samples to be analyzed at relatively high pressures, i.e., at greater than 5000 Pa. NAP-XPS can probe moderately volatile liquids, biological samples, porous materials, and/or polymeric materials that outgas significantly. In this submission, we show the survey, Li *1s*, S *2p*, C *1s*, N *1s*, O *1s*, and F *1s* NAP-XPS spectra of a Li-based electrolyte solution, which is a material that would be difficult to analyze by conventional XPS. The measurements were performed at 200 Pa in ambient gas atmosphere to compensate for surface charging. Peak fits of the C *1s*, O *1s*, and F *1s* narrow scans are presented.

Key words: Electrolyte, lithium salt, Li-ion battery, near ambient pressure x-ray photoelectron spectroscopy, NAP-XPS

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Accession #: 01814
Technique: XPS
Host Material: Lithium bis(trifluoromethane-sulfonyl) imide in propylene carbonate
Instrument: SPECS Surface Nano Analysis GmbH, EnviroESCA
Major Elements in Spectra: Li, S, C, N, O, F
Minor Elements in Spectra: None
Published Spectra: 7
Spectra in Electronic Record: 7
Spectral Category: Comparison

INTRODUCTION

Here, we present the NAP-XPS characterization of a 1M solution of lithium bis(trifluoromethane-sulfonyl) imide (LiTFSI) in propylene carbonate (PC) (Fig. 1). This document is part of a series of submissions on NAP-XPS that is being submitted to Surface Science Spectra. This set of articles and the NAP-XPS technique have previously been introduced in this journal (Ref. 1); NAP-XPS spectra of various materials including liquids (Refs. 2–4), gases (Refs. 5–8), and biological samples (Refs. 9–13) have been published so far. The SPECS EnviroESCA instrument was used to acquire these data (Refs. 14–17).

Probing Li-ion batteries (LIBs) under operational conditions with NAP-XPS is of growing interest (Refs. 18 and 19). During charging and discharging, the LIBs are operated at voltages exceeding the stability window of the organic electrolytes. Especially during the first discharge of the battery, electrolyte reduction and formation of a solid electrolyte interphase (SEI) on the anode are observed. This intentionally created SEI prevents formation of Li

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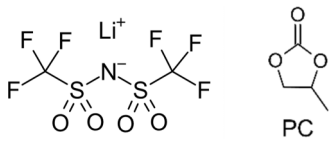

FIG. 1. Structure of LiTFSI and PC.

dendrites. At high voltages, the formation of a cathode-electrolyte interphase can also be observed during battery charging. The short- and long-term performance of a battery is significantly affected by these formed interphases.

The C *1s* core-level spectrum of LiTFSI shows *C*C/CH, *C*—O, and CO_3 peak components, at 285.0, 286.9, and 290.6 eV, related to the organic PC solvent together with an extra peak originating from the CF_3 moiety of the TFSI anion located at 292.3 eV. An additional component at 288.4 eV (C═O/O—C—O) is needed to fit the C *1s* spectrum, which is related to an unknown contamination or additional gas phase contributions from PC.

The O *1s* core-level spectrum of LiTFSI is also dominated by solvent-related *O*═C and *O*—C components at 532.3 and 534.0 eV with a nominal ratio of 1 to 2. An additional *O*═S contribution at 532.2 eV is due to TFSI and its sulfonimide groups $N(SO_2CF_3)_2$. In the respective F *1s* core-level spectrum, the LiTFSI solution shows a major peak at 688.5 eV (*F*—C) and a smaller one located at 684.6 eV (F^-). The latter peak is correlated to an inorganic F^- contamination of the LiTFSI salt, most probably LiF. The survey and core-level spectra of the LiTFSI indicate no other elements present in the electrolyte than Li, S, C, N, O, and F, originating from the organic solvent (PC), the organic lithium salt TFSI, or the inorganic LiF.

The LiTFSI concentration on the liquid surface appears to be higher than the nominal one as determined from the CF_3 to *C*—O ratio in C *1s* spectra yielding a TFSI to PC ratio of 1 to 8 compared to the expected ratio of 1 to 12. Similar findings were reported earlier using a different NAP-XPS setup (Ref. 18).

SPECIMEN DESCRIPTION (ACCESSION # 01814)

Host Material: Lithium bis(trifluoromethane-sulfonyl) imide in propylene carbonate
CAS Registry #: 90076-65-6 LiTFSI; 108-32-7 (PC)
Host Material Characteristics: Liquid; unknown crystallinity; conductor; composite; other
Chemical Name: Lithium bis(trifluoromethylsulfonyl) azanide
Source: LiTFSI, BASF, purity 99.9 wt. %, PC, BASF, purity 99.9 wt. %
Host Composition: 1M LiTFSI ($LiC_2F_6NO_4S_2$) in propylene carbonate ($C_4H_6O_3$)
Form: Liquid
Structure: None
History and Significance: Solutions of lithium bis (trifluoromethane-sulfonyl) imide in propylene carbonate is a common liquid carbonate-based electrolyte for LIBs.
As Received Condition: Under argon.
Analyzed Region: Surface of the liquid
***Ex Situ* Preparation/Mounting:** The LiTFSI electrolyte was prepared by dissolving 1M bis(trifluoromethane) sulfonimide lithium salt in PC. The salt was dried overnight at 120 °C under vacuum. All chemicals were handled under an inert argon atmosphere inside a glove box (H_2O ~1 ppm, O_2 ~1 ppm) and introduced into the EnviroESCA NAP-XPS instrument via a glove bag, which was Ar-flushed for a minimum of four times directly before any new sample preparation. For transport, all materials and vials containing solvent or electrolyte were sealed in individual vacuum pouch cells to avoid contact with atmospheric conditions. Still a small amount of oxygen (O_2) from ambient air is detected in the corresponding O *1s* spectrum but that vanishes completely during the experiments.
***In Situ* Preparation:** None
Charge Control: Charge compensation and stabilization by photo-, Auger, and secondary electrons emitted by photoionized ambient gas atoms and molecules (nitrogen and solvent vapor).
Temp. During Analysis: 300 K
Pressure During Analysis: 200 Pa
Pre-analysis Beam Exposure: 300 s

INSTRUMENT DESCRIPTION

Manufacturer and Model: SPECS Surface Nano Analysis GmbH, EnviroESCA
Analyzer Type: Spherical sector
Detector: Other, 1D delay line detector
Number of Detector Elements: 25

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

Spectrometer

Analyzer Mode: Constant pass energy
Throughput ($T = E^N$): N = 0
Excitation Source Window: Silicon nitride
Excitation Source: Al K_α
Source Energy: 1486.6 eV
Source Strength: 42 W
Source Beam Size: $270 \times 160\ \mu m^2$
Signal Mode: Multichannel direct

Geometry

Incident Angle: 55°
Source-to-Analyzer Angle: 55°
Emission Angle: 0°
Specimen Azimuthal Angle: 0°
Acceptance Angle from Analyzer Axis: 22°
Analyzer Angular Acceptance Width: 44°

DATA ANALYSIS METHOD

Energy Scale Correction: The binding energy scale was corrected for surface charging with respect to the C *1s* component peak of aliphatic carbon at 285.0 eV, in accordance with ISO 19318:2004 and ISO 15472:2010.

Recommended Energy Scale Shift: −0.7 eV

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Peak Shape and Background Method: Curve fitting of core-level spectra was done with the SPECSLAB PRODIGY software (SPECS GmbH) using a Gaussian/Lorentzian product function peak shape model in combination with a Shirley background. Generally, the full width at half maximum (FWHM) was set as a free parameter but constrained to be the same for all peaks within the same core-level spectrum. The spectra were fitted with a minimum set of peak components except for the O *1s* core level. Here, the O *1s* peak components (O═C, O─C) from PC were used with identical FWHM and a constrained peak area ratio of 1 to 2 according to the PC stoichiometry. The third component for TFSI (O═S) was then added to complete the fit of the O *1s* core-level data.

Quantitation Method: Elemental compositions were calculated using the standard SPECS software SPECSLAB PRODIGY.

ACKNOWLEDGMENT

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Paul M. Dietrich: Conceptualization (equal); Data curation (equal); Formal analysis (equal); Investigation (equal); Validation (equal); Writing – original draft (lead); Writing – review & editing (lead). **Lydia Gerlein:** Conceptualization (equal); Data curation (equal); Formal analysis (equal); Investigation (equal); Validation (equal). **Julia Maibach:** Conceptualization (equal); Data curation (equal); Formal analysis (equal); Investigation (equal); Validation (equal). **Andreas Thissen:** Conceptualization (equal); Data curation (equal); Formal analysis (equal); Investigation (equal); Validation (equal).

DATA AVAILABILITY

The data that support the findings of this study are available within the article and its supplementary material.

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SPECTRAL FEATURES TABLE

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV counts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01814-01	Survey	…	…	…	…	…	…
01814-02	Li *1s*	55.6	…	468	0.06	11.2	Li^+, from LiTFSI (and LiF)
01814-03	S *2p*	169.0	…	6079	2.25	4.2	O═*S*═O, sulfur from bis (trifluoromethane-sulfonyl) imide
01814-04	C *1s*	285.0	…	23 816	1.00	36.8	Carbon
01814-04[a]	C *1s*	285.0	1.4	…	…	28.0	*C*C/*C*H, aliphatic carbon (solvent, hydrocarbon contamination)
01814-04[a]	C *1s*	286.9	1.4	…	…	40.9	*C*—O, carbon in single bonds to O (solvent)
01814-04[a]	C *1s*	288.4	1.4	…	…	6.0	*C*═O/O—*C*—O, carbon in carbonyl or acetal moieties (unknown contamination)
01814-04[a]	C *1s*	290.6	1.4	…	…	19.7	CO_3, carbon in carbonate moiety (solvent)
01814-04[a]	C *1s*	292.3	1.4	…	…	5.4	CF_3, carbon in trifluoromethane moiety
01814-05	N *1s*	399.2	…	2215	1.67	2.1	N^-, nitrogen from bis (trifluoromethane-sulfonyl) imide
01814-05	N *1s*	405.0	…	…	…	…	N_2, molecular nitrogen (venting gas)
01814-06	O *1s*	533.0	…	58 700	2.47	36.7	Oxygen
01814-06[a]	O *1s*	532.2	1.5	…	…	27.6	*O*═S, oxygen in double bonds to S, bis(trifluoromethane-sulfonyl) imide
01814-06[a]	O *1s*	532.3	1.9	…	…	24.1	*O*═C, oxygen in double bonds to C (solvent)
01814-06[a]	O *1s*	534.0	1.9	…	…	48.3	*O*—C, oxygen in single bonds to C (solvent)
01814-06[a]	O *1s*	538.4	…	…	…	…	O_2, molecular oxygen
01814-07	F *1s*	688.5	…	19 161	3.27	9.0	fluorine
01814-07[a]	F *1s*	684.7	1.7	…	…	30.0	F^-, fluoride from LiF
01814-07[a]	F *1s*	688.5	1.9	…	…	70.0	*F*—C, fluorine in single bonds to C, from trifluoromethane moiety

Comment to Spectral Features Table: All spectra acquired for sample held in 2 mbar residual ambient gas atmosphere (NAP-XPS spectra).
[a]Peak energy obtained from the fitted data. Concentrations reported as component peak area percentages of the corresponding peak fits.

ANALYZER CALIBRATION TABLE

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV counts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
…	Au *4f*	83.9	0.67	8 940.7	…	…	…
…	Ag *3d*	368.1	0.60	13 854.9	…	…	…
…	Ge *2p*	1217.5	1.15	8 064.6	…	…	…

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GUIDE TO FIGURES

Spectrum (Accession) #	Spectral Region	Voltage Shift[a] (eV)	Multiplier	Baseline	Comment #
01814-01	Survey	0.7	…	…	
01814-02	Li *1s*	0.7	…	…	1,2
01814-03	S *2p*	0.7	…	…	1,2
01814-04	C *1s*	0.7	…	…	1,2
01814-05	N *1s*	0.7	…	…	1,2
01814-06	O *1s*	0.7	…	…	1,2
01814-07	F *1s*	0.7	…	…	1

[a]Voltage shift of the archived (as-measured) spectrum relative to the printed figure. The figure reflects the recommended energy scale correction due to a calibration correction, sample charging, flood gun, or other phenomenon.

Comments:

1. Energy scale corrected in the spectrum displayed.
2. No peak fitting analysis reported.

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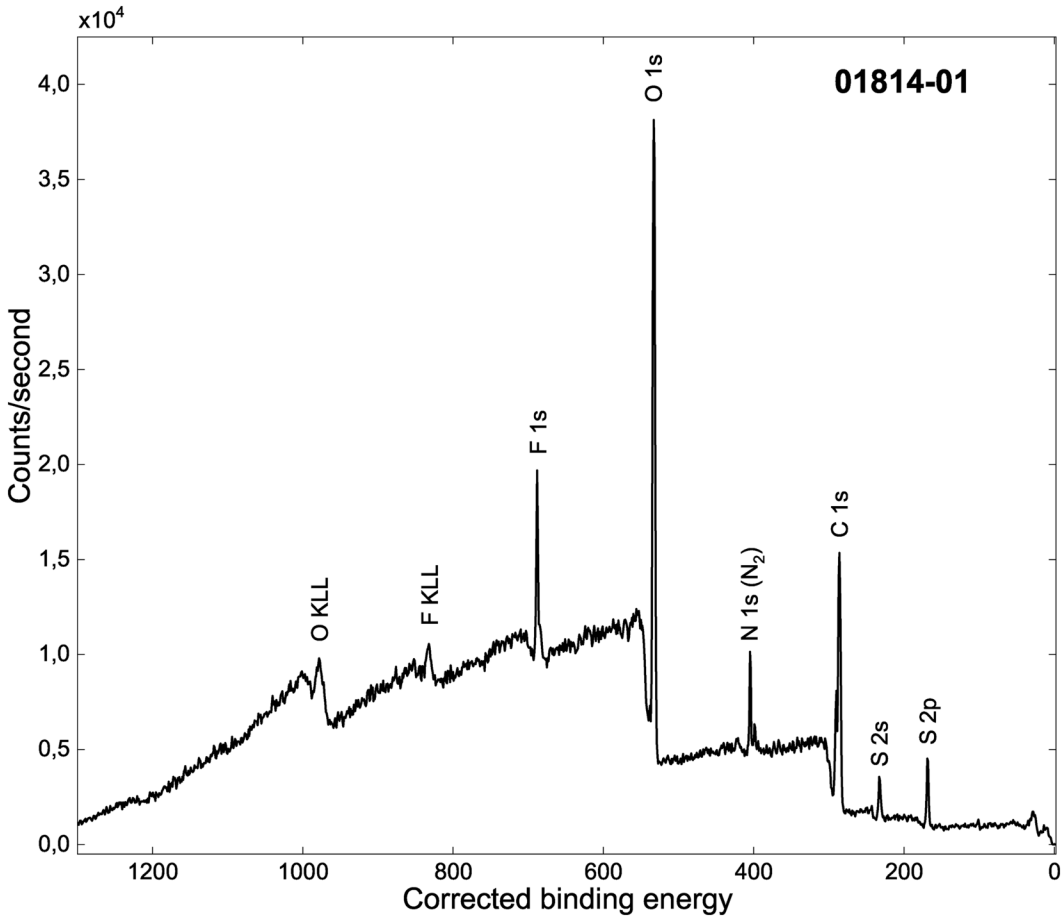

Accession #:	01814-01
Host Material:	Lithium bis(trifluoromethane-sulfonyl) imide in propylene carbonate
Technique:	XPS
Spectral Region:	Survey
Instrument:	SPECS EnviroESCA
Excitation Source:	Al K_a monochromatic
Source Energy:	1486.6 eV
Source Strength:	42 W
Source Size:	270 × 160 mm^2
Analyzer Type:	Spherical sector analyzer
Incident Angle:	54.7°
Emission Angle:	0°
Analyzer Pass Energy:	100 eV
Analyzer Resolution:	1.7 eV
Total Signal Accumulation Time:	130 s
Total Elapsed Time:	133 s
Number of Scans:	1
Effective Detector Width:	25 eV

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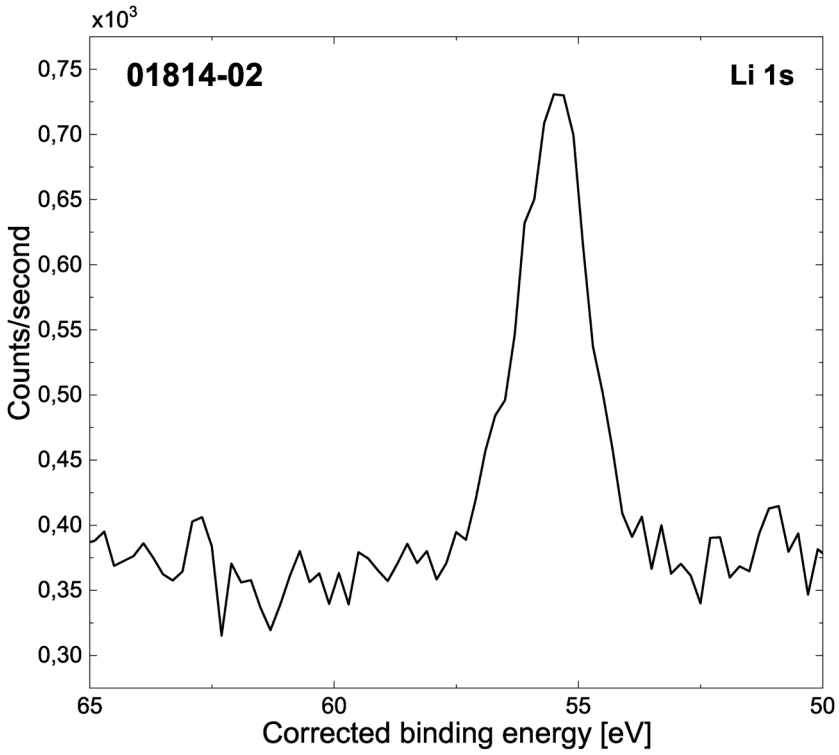
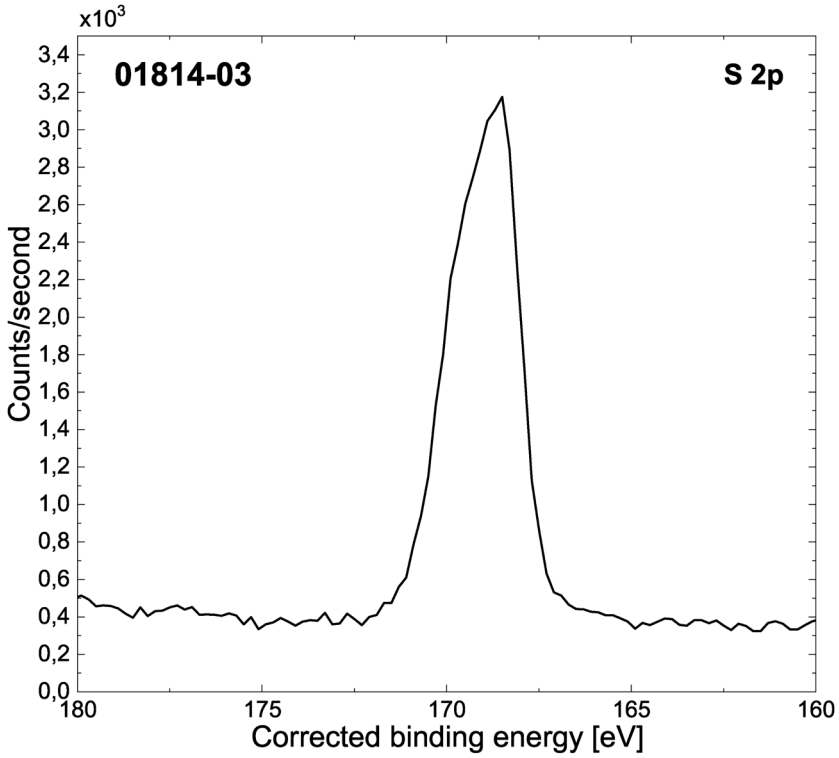


■ **Accession #:** 01814-02
■ **Host Material:** Lithium bis(trifluoromethanesulfonyl) imide in propylene carbonate
■ **Technique:** XPS
■ **Spectral Region:** Li *1s*

Instrument: SPECS EnviroESCA
Excitation Source: Al K_a monochromatic
Source Energy: 1486.6 eV
Source Strength: 42 W
Source Size: 270 × 160 mm^2
Analyzer Type: Spherical sector
Incident Angle: 54.7°
Emission Angle: 0°
Analyzer Pass Energy: 50 eV
Analyzer Resolution: 1.02 eV
Total Signal Accumulation Time: 51 s
Total Elapsed Time: 75 s
Number of Scans: 5
Effective Detector Width: 5 eV



■ **Accession #:** 01814-03
■ **Host Material:** Lithium bis(trifluoromethanesulfonyl) imide in propylene carbonate
■ **Technique:** XPS
■ **Spectral Region:** S *2p*

Instrument: SPECS EnviroESCA
Excitation Source: Al K_a monochromatic
Source Energy: 1486.6 eV
Source Strength: 42 W
Source Size: 270 × 160 mm^2
Analyzer Type: Spherical sector
Incident Angle: 54.7°
Emission Angle: 0°
Analyzer Pass Energy: 50 eV
Analyzer Resolution: 1.02 eV
Total Signal Accumulation Time: 81 s
Total Elapsed Time: 105 s
Number of Scans: 5
Effective Detector Width: 5 eV

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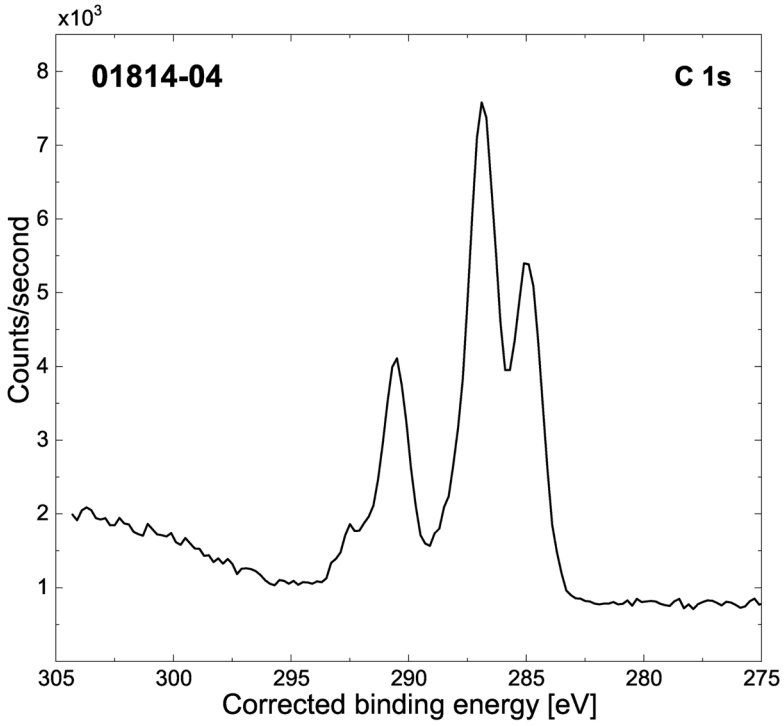

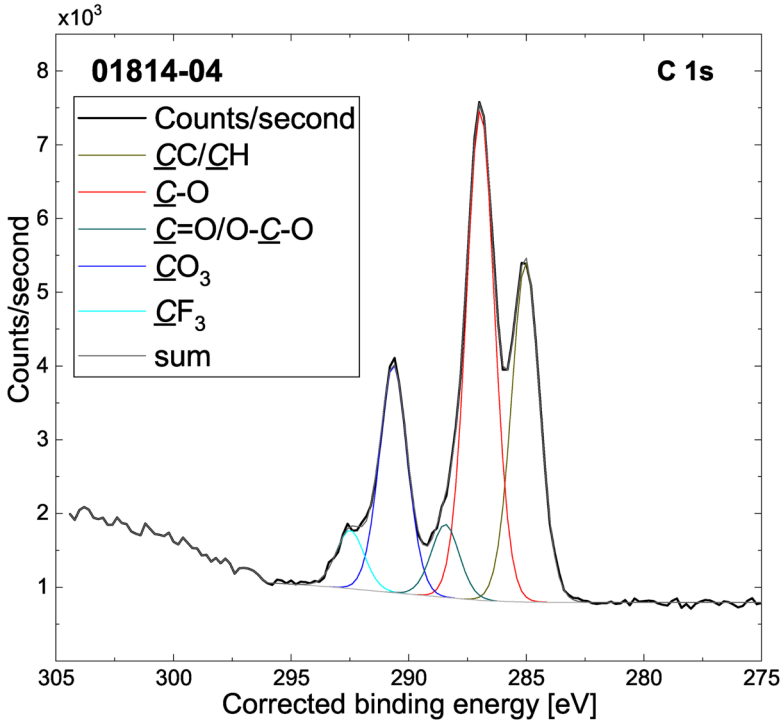

■ **Accession #:** 01814-04
■ **Host Material:** Lithium bis(trifluoromethane-sulfonyl) imide in propylene carbonate
■ **Technique:** XPS
■ **Spectral Region:** C *1s*

Instrument: SPECS EnviroESCA
Excitation Source: Al K_a monochromatic
Source Energy: 1486.6 eV
Source Strength: 42 W
Source Size: 270 × 160 mm^2
Analyzer Type: Spherical sector
Incident Angle: 54.7°
Emission Angle: 0°
Analyzer Pass Energy: 50 eV
Analyzer Resolution: 1.02 eV
Total Signal Accumulation Time: 78 s
Total Elapsed Time: 103 s
Number of Scans: 5
Effective Detector Width: 5 eV

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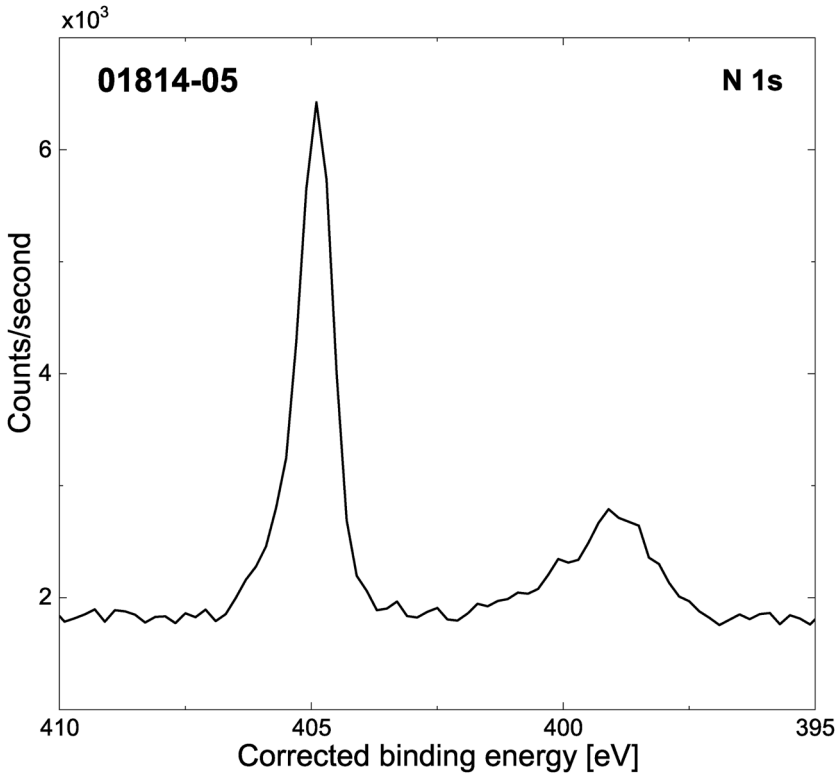

■ **Accession #:** 01814-05
■ **Host Material:** Lithium bis(trifluoromethane-sulfonyl) imide in propylene carbonate
■ **Technique:** XPS
■ **Spectral Region:** N *1s*

Instrument: SPECS EnviroESCA
Excitation Source: Al K_a monochromatic
Source Energy: 1486.6 eV
Source Strength: 42 W
Source Size: 270 × 160 mm^2
Analyzer Type: Spherical sector
Incident Angle: 54.7°
Emission Angle: 0°
Analyzer Pass Energy: 50 eV
Analyzer Resolution: 1.02 eV
Total Signal Accumulation Time: 51 s
Total Elapsed Time: 75 s
Number of Scans: 5
Effective Detector Width: 5 eV

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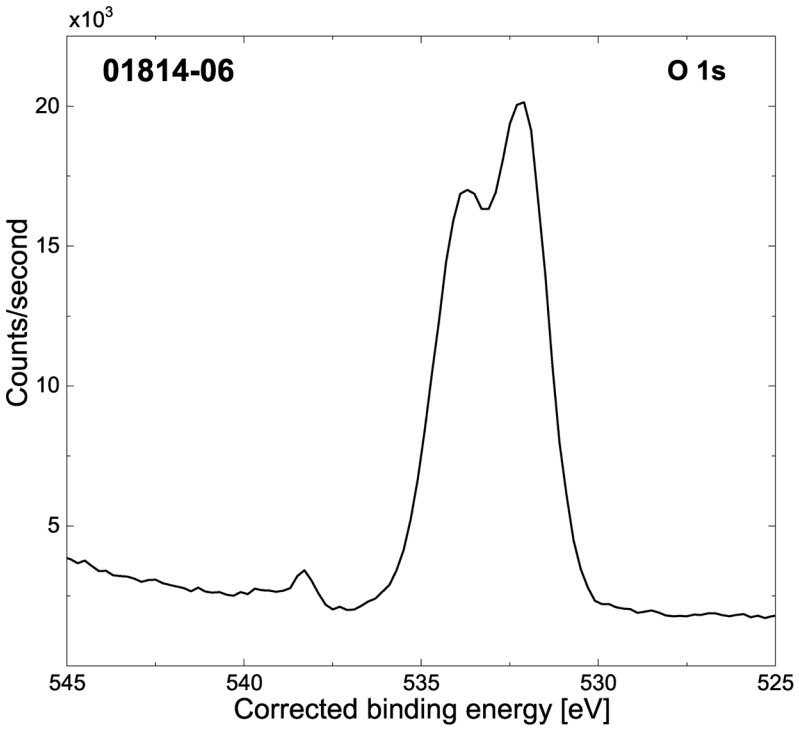

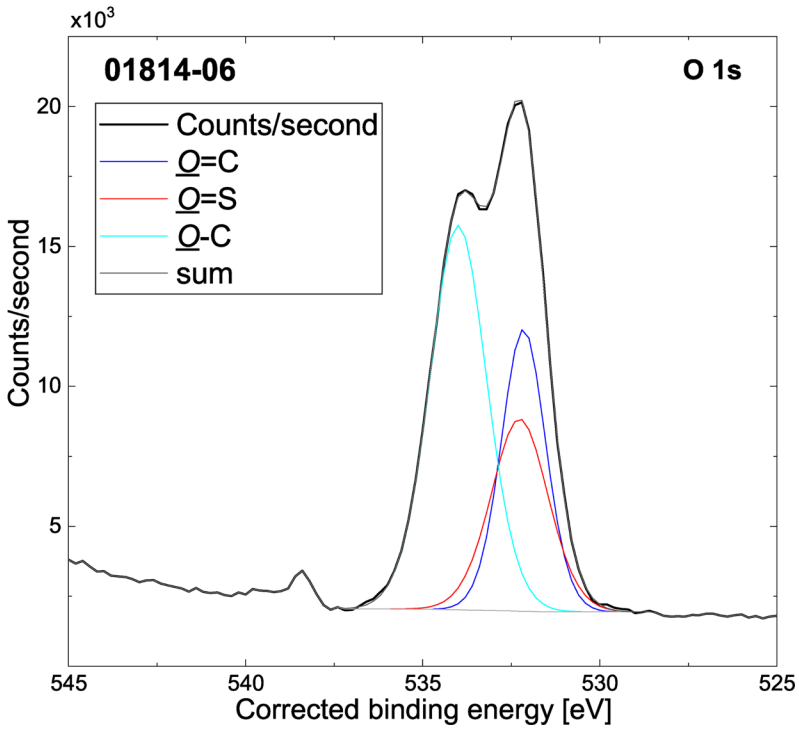

■ **Accession #:** 01814-06
■ **Host Material:** Lithium bis(trifluoromethane-sulfonyl) imide in propylene carbonate
■ **Technique:** XPS
■ **Spectral Region:** O *1s*

Instrument: SPECS EnviroESCA
Excitation Source: Al K_a monochromatic
Source Energy: 1486.6 eV
Source Strength: 42 W
Source Size: 270 × 160 mm^2
Analyzer Type: Spherical sector
Incident Angle: 54.7°
Emission Angle: 0°
Analyzer Pass Energy: 50 eV
Analyzer Resolution: 1.02 eV
Total Signal Accumulation Time: 63 s
Total Elapsed Time: 88 s
Number of Scans: 5
Effective Detector Width: 5 eV

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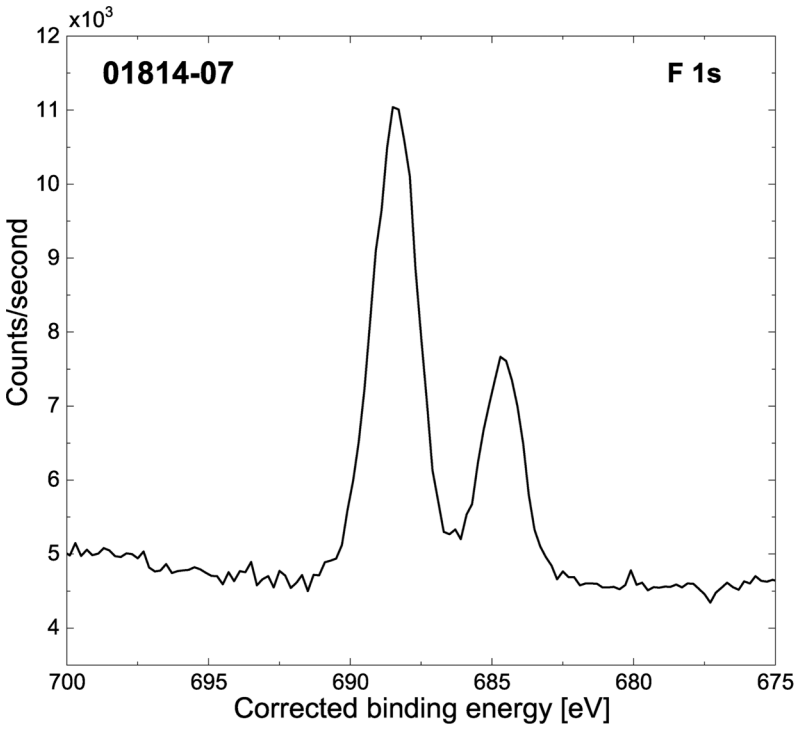

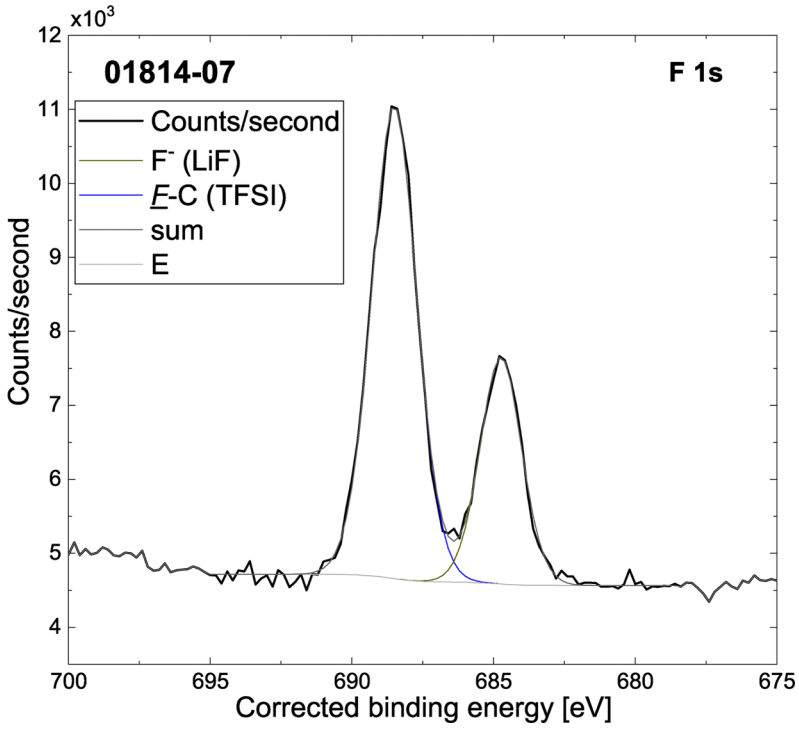

■ **Accession #:** 01814-07
■ **Host Material:** Lithium bis(trifluoromethanesulfonyl) imide in propylene carbonate
■ **Technique:** XPS
■ **Spectral Region:** F *1s*

Instrument: SPECS EnviroESCA
Excitation Source: Al K_a monochromatic
Source Energy: 1486.6 eV
Source Strength: 42 W
Source Size: 270 × 160 mm^2
Analyzer Type: Spherical sector
Incident Angle: 54.7°
Emission Angle: 0°
Analyzer Pass Energy: 50 eV
Analyzer Resolution: 1.02 eV
Total Signal Accumulation Time: 78 s
Elapsed Time: 103 s
Number of Scans: 5
Effective Detector Width: 5 eV

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