Analysis of the Li KLL Auger Transition on Freshly Exposed Lithium and Lithium Surface Oxide by AES

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Lithium scribed in vacuum and a particle of lithium oxide were analysed by AES and lithium metal exposed to atmosphere for <1 min was analysed by XPS. The oxide particle embedded in the host metal was analysed at low and high take off angles. AES spectra acquired demonstrate the change in the KLL structure with increasing oxygen concentration. Survey spectra as well as high resolution narrow spectra were acquired and are presented. The presence of lithium carbonate, together with the oxide, formed in air, was confirmed by XPS as it is not easily resolvable using AES. © 2013 American Vacuum Society. [http://dx.doi.org/10.1116/1.116/11.20130901]

Keywords: lithium Li; lithium oxide Li₂O; Auger electron spectroscopy AES; x-ray photoelectron spectroscopy XPS

Accession #: 01274
Technique: AES, XPS
Host Material: lithium
Instrument: Thermo Scientific Theta Probe, Thermo MicroLab 350
Major Elements in Spectra: Li, O
Minor Elements in Spectra: Na, C, Ar
Published Spectra: 11
Spectra in Electronic Record: 24
Spectral Category: comparison

INTRODUCTION

Lithium, the third element of the periodic table, is notable for its widespread use in electrical batteries (Ref. 1), in quartz crystal vibrators, where it improves the Q factor (Ref. 2), in silicon-based x-ray detectors (Ref. 3), and in specialised electronic applications, such as electrochromic thin films (Ref. 4). LiOH has been used in water treatment for water cooled nuclear power plants (Ref. 5), and, as an alloying element with aluminium alloys (Ref. 6), it is used to gain both weight reduction and improved mechanical properties. Because of its low atomic number it has only a single core level, the 1s transition in the metal giving a peak at a binding energy of 54.7 eV – a value very close to the 3p level of iron. It is thus easily masked within the spectrum.

In this work spectra are presented that show the main Auger transition for lithium and lithium oxide. The lithium metal differential spectrum shows the primary peak at 53.8 eV together with a loss satellite at 48.8 eV. The differential spectrum of the oxide particle, collected with an emission angle of 60 degrees, relative to the sample normal, and a depth of analysis ~0.6 nm, shows the metal components at 53.8 and 48.5 eV together with two oxide components at 42.5 and 36 eV. By tilting the sample 15 degrees from the analyser the take-off angle is reduced and the metal components further suppressed. The differential spectrum of the oxide particle, collected with an emission angle of 75 degrees and a depth of analysis ~0.4 nm, shows a broad feature at 52.7 eV, which is likely the remaining metal signal, together with the two main oxide components at 42.5 and 36 eV.

A sharp peak at 29 eV in the survey spectrum for scribed lithium metal was been identified as the sodium L_{2,3}VV transition, although a narrow scan over the 950-1050 eV region did not show the presence of the sodium KLL transition. The high mobility of sodium in lithium leads to surface segregation with monolayer thickness (Ref. 7).

The kinetic energy of the Li KLL and Li₂O transitions presented here are in good agreement with those previously reported when referencing to the Fermi level is taken into account (Ref. 8).

Gradual oxidation is observed for the bare metal surface over the course of a single analysis even with chamber pressure of 1×10^{-9} mb. Re-examining the lithium metal KLL transition at the same sample position after an hour of analysis shows a drop in the intensity of the metal peaks and the appearance of slight oxide peaks. Gradual oxidation can also be observed in the generated SEM image with the formation of islands of oxide.

XPS analysis carried out on a piece of lithium metal exposed to atmosphere for less than 1 min showed the expected significant oxidation of the surface. The surface was argon ion etched for 10 min to remove carbon contamination and further etching had little effect on the oxide concentration. The lithium 1s peak shows a strong oxide component at 57.0 eV and weaker carbonate and metal components at 58.3 and 55.5 eV respectively. Features believed to be surface and bulk plasmons are observed after the Li 1s, O 1s and O KLL peaks, with an energy separation of approximately 10, 18.5, and 25.6 eV from the main peaks. Sodium is not observed in the initial XPS survey spectrum of freshly cut lithium but becomes gradually enriched at the surface during analysis.

SPECIMEN DESCRIPTION (ACCESSION #01274)

Host Material: lithium
CAS Registry #: 7439-93-2
Host Material Characteristics: homogeneous; solid; polycrystalline; conductor; metal
Chemical Name: Li
Source: Sigma-Aldrich
Host Composition: Li 99.9%
Form: 12.7 mm diameter rod
Lot #: 265969 Aldrich

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As Received Condition: Lithium rod was stored under argon atmosphere in a glass Winchester.

Analyzed Region: not specified

Ex Situ Preparation/Mounting: Rapid oxidation of the sample occurred after removal from inert atmosphere. A 2 mm thick sample was cut from the rod using a scalpel. This was mounted in a sample holder and placed into the spectrometer entry lock within 1 min.

In Situ Preparation: The black surface oxide/carbonate was removed in vacuum using the end of a wobble stick, exposing the bare metal surface. Analysis was carried out on the metal and a small oxide particle embedded in the surface.

Pre-Analysis Beam Exposure: One min to atmospheric conditions while the sample was cut and mounted

Charge Control: A shift of +0.65 eV is applied for the XPS spectra to correct for C 1s at 285 eV

Temp. During Analysis: 300 K

Pressure During Analysis: $<1 \times 10^{-9}$ Pa

INSTRUMENT DESCRIPTION (INSTRUMENT 1 OF 2) —

Manufacturer and Model: Thermo Scientific Theta Probe

Analyzer Type: spherical sector

Detector: multi-channel plate detector

Number of Detector Elements: 128

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA —

■ Spectrometer

Analyzer Mode: constant pass energy

Throughput ($T = E^N$): N=See comment below

Throughput Comment: The transmission function is calculated from a cubic polynomial fit to a plot of LOG (Peak Area * XSF/PE) vs. LOG (RR)RR = KE/PE (Retard Ratio = Kinetic Energy/Pass Energy) XSF is a term to account for the difference in absolute sensitivities of the Ag MNN and Ag 3d peaks.

Excitation Source Window: none

Excitation Source: Al K_{α} , monochromatic

Source Energy: 1486.68 eV

Source Strength: 300 W

Source Beam Size: $200 \mu\text{m} \times 200 \mu\text{m}$

Signal Mode: pulse single channel

■ Geometry

Incident Angle: 30°

Source to Analyzer Angle: 67.4°

Emission Angle: 53°

Specimen Azimuthal Angle: 70°

Acceptance Angle from Analyzer Axis: $50/+30^\circ$

Analyzer Angular Acceptance Width: $25^\circ \times 25^\circ$

■ Ion Gun

Manufacturer and Model: Thermo EX05

Energy: 3000 eV

Current: 2 mA

Current Measurement Method: biased stage

Sputtering Species: Ar^+

Spot Size (unrastered): $200 \mu\text{m}$

Raster Size: $2000 \mu\text{m} \times 2000 \mu\text{m}$

Incident Angle: 45°

Polar Angle: 58.4°

Azimuthal Angle: 100°

Comment: differentially pumped ion gun

INSTRUMENT DESCRIPTION (INSTRUMENT 2 OF 2) —

Manufacturer and Model: Thermo Microlab 350

Analyzer Type: spherical sector

Detector: multi-channel parallel detector

Number of Detector Elements: 6

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA —

■ Spectrometer

Analyzer Mode: constant retard ratio

Throughput ($T = E^N$): N=See comment below

Throughput Comment: The transmission function is calculated from a cubic polynomial fit to a plot of LOG (Peak Area * XSF/PE) vs. LOG (RR)RR = KE/PE (Retard Ratio = Kinetic Energy/Pass Energy) XSF is a term to account for the difference in absolute sensitivities of the Ag MNN and Ag 3d peaks.

Excitation Source Window: none

Excitation Source: electron beam

Source Energy: 10,000 eV

Source Strength: 1.2 nA

Source Beam Size: $0.1 \mu\text{m} \times 0.1 \mu\text{m}$

Signal Mode: pulse single channel

■ Geometry

Incident Angle: 60°

Source to Analyzer Angle: 60°

Emission Angle: varies by spectrum

Specimen Azimuthal Angle: 0°

Acceptance Angle from Analyzer Axis: $50/+25^\circ$

Analyzer Angular Acceptance Width: $30^\circ \times 30^\circ$

■ Ion Gun

Manufacturer and Model: Thermo EX05

Energy: 3000 eV

Current: 1 mA

Current Measurement Method: biased stage

Sputtering Species: Ar⁺

Spot Size (unrastered): 200 μm

Raster Size: 2000 $\mu\text{m} \times 2000 \mu\text{m}$

Incident Angle: 67.5°

Polar Angle: 43°

Azimuthal Angle: 228°

Comment: differentially pumped ion gun

DATA ANALYSIS METHOD

Energy Scale Correction: For the lithium XPS data the binding energy values can be corrected using C 1s peak at 285.0 eV using a shift of +0.65 eV.

Recommended Energy Scale Shift: For XPS spectra, Accession #s 1274-10 through -12, +0.65 eV

Peak Shape and Background Method: The peak fitting function used is a Gaussian - Lorentzian product function with a maximum of 30% Lorentzian mix. The Shirley function was used for background subtraction.

Quantitation Method: Peak height has used for peak amplitude and the FWHM of each peak measured using a Shirley background for AES spectra. Peak area used for peak amplitude for XPS spectra. Peaks were fitted using

ThermoScientific Advantage v4.54 software. Sensitivity factors used in Thermo Scientific Advantage software. Peak library is ALWAG from C.D. Wagner, *et al.*, Surf. Interface Anal., 3, 211 (1981)

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SPECTRAL FEATURES TABLE							
Spectrum ID #	Element/Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01274-02 ^a	Li KLL	52.3, 46.5 : 53	6.8	15274	...	...	Li metal
01274-09 ^b	Ox KLL	509.5	6.2	626	...	...	O in Li ₂ O/LiOH
01274-04 ^c	Li KLL	51.8, 40.8 : 53	8.9	49274	...	...	Li in Li ₂ O and metal
01274-05 ^b	Ox KLL	509.8	5.6	89707	...	...	O in Li ₂ O/LiOH
01274-07 ^d	Li KLL	40.7, 34.3 : 42	3.0	16603	...	...	Li in Li ₂ O
01274-08 ^b	Ox KLL	509.8	5.0	27809	...	...	O in Li ₂ O/LiOH
01274-06 ^e	Ca KLL	264.0	-	1392	...	...	Contamination or carbonate
01274-11	Li 1s	55.5	0.78	154	0.02	2.59	Li metal
01274-11	Li 1s	57.0	1.37	3295	0.02	55.5	Li in Li ₂ O/LiOH
01274-11 ^f	Li 1s	58.3	1.37	116	0.02	1.96	Li in Li ₂ CO ₃
01274-12 ^f	O 1s	534.7	1.43	3310	0.66	4.15	O in Li ₂ CO ₃
01274-12	O 1s	531.9	1.16	27922	0.66	34.97	O in Li ₂ O/LiOH
01274-13 ^g	Ar 2p	245.8	1.27	90.8	0.96	0.03	Ar
01274-14 ^h	C 1s	285.0	1.72	181	0.25	0.27	Hydrocarbon contamination
01274-14 ^f	C 1s	288.8	2.41	343	0.25	0.52	C in Li ₂ CO ₃

^a Scribed in vacuum sample

^b Oxygen could be present at surface in the form of lithium oxide or hydroxide.

^c Oxide particle sample

^d Tilted oxide particle sample

^e Carbon may be present as trace surface contamination or as lithium carbonate.

^f Lithium carbonate is formed when lithium reacts with carbon dioxide in the atmosphere.

^g Argon is gradually implanted into the lithium surface during argon ion etching.

^h Contamination from the atmosphere or the scalpel blade used to cut the lithium sample

ANALYZER CALIBRATION TABLE							
Spectrum ID #	Element/Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
17	Au M ₅ N _{6,7} N _{6,7}	2014.0	22	19676	...	...	Au metal
16	Au N _{6,7} VV	69.5, 71.5	7.4	38605	...	...	Au metal
22	Ag M ₄ NN	351.8, 357.5	4.4	111196	...	...	Ag metal
20	Cu L ₃ VV	60.2, 62.3	6.2	19337	...	...	Cu metal
19	Cu M _{2,3} VV	917.3	7.2	41899	...	...	Cu metal
23	Au 4f _{7/2}	84.1	0.74	17347	4.95	100	Au metal
24	Cu 2p _{3/2}	932.6	0.87	108427	4.2	100	Cu metal

GUIDE TO FIGURES					
Spectrum (Accession) #	Spectral Region	Voltage Shift*	Multiplier	Baseline	Comment #
1274-01	survey		1	0	1
1274-02	Li KLL		1	0	1
1274-03	survey		1	0	2
1274-04	Li KLL		1	0	2
1274-05	O KLL		1	0	2
1274-06	survey		1	0	3
1274-07	Li KLL		1	0	3
1274-08	O KLL		1	0	3
1274-09 [NP]**	O KLL		1	0	1
1274-10	survey	−0.65	1	0	4
1274-11	Li 1s	−0.65	1	0	4
1274-12	O 1s	−0.65	1	0	4
1274-13 [NP]	Ar 2p	−0.65	1	0	4
1274-14 [NP]	C 1s	−0.65	1	0	4
1275-01 [NP]	Au NVV, Au MNN		1	0	5
1275-02 [NP]	Au NVV		1	0	5
1275-03 [NP]	Au MNN		1	0	5
1275-04 [NP]	Au 4f		1	0	5
1276-01 [NP]	Cu MVV, Cu LVV		1	0	6
1276-02 [NP]	Cu MVV		1	0	6
1276-03 [NP]	Cu LVV		1	0	6
1276-04 [NP]	Cu 2p		1	0	6
1277-01 [NP]	Ag MNN		1	0	7
1277-02 [NP]	Ag MNN		1	0	7

* 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.

** [NP] signifies not published; digital spectra are archived in SSS database but not reproduced in the printed journal.

1. fresh Li metal surface, scribed *in situ*, take-off angle = 30°
2. oxide particle embedded in Li sample, take-off angle = 30°
3. oxide particle embedded in Li sample, take-off angle = 15°
4. surface of Li sample exposed to air for <1 min, take-off angle = 30°
5. Au calibration
6. Cu calibration
7. Ag calibration

Accession #	01274-01
Host Material	lithium
Technique	AES
Spectral Region	survey
Instrument	Thermo MicroLab 350
Excitation Source	electron beam
Source Energy	10000 eV
Source Strength	1.2 nA
Source Size	0.1 μm $\times$ 0.1 μm
Analyzer Type	spherical sector
Incident Angle	30°
Emission Angle	60°
Analyzer Retard Ratio:	4
Analyzer Resolution	2%
Total Signal Accumulation Time	267 s
Total Elapsed Time	361 s
Number of Scans	3
Source Beam Size at Specimen Surface	0.011 μm $\times$ 0.011 μm
Effective Detector Width	1 eV
Analyzer Angular Acceptance Width	25° $\times$ 25°
Comment	fresh Li metal surface, scribed <i>in situ</i> , take-off angle = 30°

■ Accession #: 01274-02
 ■ Host Material: lithium
 ■ Technique: AES
 ■ Spectral Region: Li KLL

Instrument: Thermo MicroLab 350
 Excitation Source: electron beam
 Source Energy: 10000 eV
 Source Strength: 1.2 nA
 Source Size: 0.1 μm $\times$ 0.1 μm
 Analyzer Type: spherical sector
 Incident Angle: 30°
 Emission Angle: 60°
 Analyzer Retard Ratio: 4
 Analyzer Resolution: 2%
 Total Signal Accumulation Time: 203 s
 Total Elapsed Time: 272 s
 Number of Scans: 50
 Source Beam Size at Specimen Surface: 0.011 μm $\times$ 0.011 μm
 Effective Detector Width: 0.5 eV
 Analyzer Angular Acceptance Width: 25° $\times$ 25°
 Comment: fresh Li metal surface, scribed *in situ*, take-off angle 30°

Accession #	01274-03
Host Material	lithium
Technique	AES
Spectral Region	survey
Instrument	Thermo MicroLab 350
Excitation Source	electron beam
Source Energy	10000 eV
Source Strength	1.2 nA
Source Size	0.1 μm $\times$ 0.1 μm
Analyzer Type	spherical sector
Incident Angle	30°
Emission Angle	60°
Analyzer Retard Ratio:	4
Analyzer Resolution	2%
Total Signal Accumulation Time	267 s
Total Elapsed Time	361 s
Number of Scans	3
Source Beam Size at Specimen Surface	0.011 μm $\times$ 0.011 μm
Effective Detector Width	1 eV
Analyzer Angular Acceptance Width	25° $\times$ 25°
Comment	oxide particle embedded in Li sample, take-off angle = 30°

■ Accession #: 01274-04
 ■ Host Material: lithium
 ■ Technique: AES
 ■ Spectral Region: Li KLL

Instrument: Thermo MicroLab 350
 Excitation Source: electron beam
 Source Energy: 10000 eV
 Source Strength: 1.2 nA
 Source Size: $0.1 \mu\text{m} \times 0.1 \mu\text{m}$
 Analyzer Type: spherical sector
 Incident Angle: 30°
 Emission Angle: 60°
 Analyzer Retard Ratio: 4
 Analyzer Resolution: 2%
 Total Signal Accumulation Time: 203 s
 Total Elapsed Time: 272 s
 Number of Scans: 50
 Source Beam Size at Specimen Surface: $0.011 \mu\text{m} \times 0.011 \mu\text{m}$
 Effective Detector Width: 0.5 eV
 Analyzer Angular Acceptance Width: $25^\circ \times 25^\circ$
 Comment: oxide particle embedded in Li sample, take-off angle = 30°

■ Accession #: 01274-05
 ■ Host Material: lithium
 ■ Technique: AES
 ■ Spectral Region: O KLL

Instrument: Thermo MicroLab 350
 Excitation Source: electron beam
 Source Energy: 10000 eV
 Source Strength: 1.2 nA
 Source Size: $0.1 \mu\text{m} \times 0.1 \mu\text{m}$
 Analyzer Type: spherical sector
 Incident Angle: 30°
 Emission Angle: 60°
 Analyzer Retard Ratio: 4
 Analyzer Resolution: 2%
 Total Signal Accumulation Time: 402 s
 Total Elapsed Time: 510 s
 Number of Scans: 50
 Source Beam Size at Specimen Surface: $0.011 \mu\text{m} \times 0.011 \mu\text{m}$
 Effective Detector Width: 0.5 eV
 Analyzer Angular Acceptance Width: $25^\circ \times 25^\circ$
 Comment: oxide particle embedded in Li sample, take-off angle = 30°

Accession #	01274-06
Host Material	lithium
Technique	AES
Spectral Region	survey
Instrument	Thermo MicroLab 350
Excitation Source	electron beam
Source Energy	10000 eV
Source Strength	1.2 nA
Source Size	0.1 μm $\times$ 0.1 μm
Analyzer Type	spherical sector
Incident Angle	30°
Emission Angle	75°
Analyzer Retard Ratio:	4
Analyzer Resolution	2%
Total Signal Accumulation Time	267 s
Total Elapsed Time	361 s
Number of Scans	3
Source Beam Size at Specimen Surface	0.011 μm $\times$ 0.011 μm
Effective Detector Width	1 eV
Analyzer Angular Acceptance Width	25° $\times$ 25°
Comment	oxide particle embedded in Li sample, take-off angle = 15°

■ Accession #: 01274-07
 ■ Host Material: lithium
 ■ Technique: AES
 ■ Spectral Region: Li KLL

Instrument: Thermo MicroLab 350
 Excitation Source: electron beam
 Source Energy: 10000 eV
 Source Strength: 1.2 nA
 Source Size: $0.1 \mu\text{m} \times 0.1 \mu\text{m}$
 Analyzer Type: spherical sector
 Incident Angle: 30°
 Emission Angle: 75°
 Analyzer Retard Ratio: 4
 Analyzer Resolution: 2%
 Total Signal Accumulation Time: 203 s
 Total Elapsed Time: 272 s
 Number of Scans: 50
 Source Beam Size at Specimen Surface: $0.011 \mu\text{m} \times 0.011 \mu\text{m}$
 Effective Detector Width: 0.5 eV
 Analyzer Angular Acceptance Width: $25^\circ \times 25^\circ$
 Comment: oxide particle embedded in Li sample, take-off angle = 15°

■ Accession #: 01274-08
 ■ Host Material: lithium
 ■ Technique: AES
 ■ Spectral Region: O KLL

Instrument: Thermo MicroLab 350
 Excitation Source: electron beam
 Source Energy: 10000 eV
 Source Strength: 1.2 nA
 Source Size: $0.1 \mu\text{m} \times 0.1 \mu\text{m}$
 Analyzer Type: spherical sector
 Incident Angle: 30°
 Emission Angle: 75°
 Analyzer Retard Ratio: 4
 Analyzer Resolution: 2%
 Total Signal Accumulation Time: 402 s
 Total Elapsed Time: 510 s
 Number of Scans: 50
 Source Beam Size at Specimen Surface: $0.011 \mu\text{m} \times 0.011 \mu\text{m}$
 Effective Detector Width: 0.5 eV
 Analyzer Angular Acceptance Width: $25^\circ \times 25^\circ$
 Comment: oxide particle embedded in Li sample, take-off angle = 15°

Accession #	01274-10
Host Material	lithium
Technique	XPS
Spectral Region	survey
Instrument	Thermo Scientific Theta Probe
Excitation Source	Al K_{α} monochromatic
Source Energy	1486.68 eV
Source Strength	300 W
Source Size	200 μm $\times$ 200 μm
Analyzer Type	spherical sector
Incident Angle	30°
Emission Angle	53°
Analyzer Pass Energy:	300 eV
Analyzer Resolution	2.0 eV
Total Signal Accumulation Time	675 s
Total Elapsed Time	1235 s
Number of Scans	3
Source Beam Size at Specimen Surface	400 μm $\times$ 200 μm
Effective Detector Width	0.3 eV
Analyzer Angular Acceptance Width	30° $\times$ 30°
Comment	surface of Li sample exposed to atmosphere for <1 min, take-off angle = 30°

■ **Accession #:** 01274-11
 ■ **Host Material:** lithium
 ■ **Technique:** XPS
 ■ **Spectral Region:** Li 1s

Instrument: Thermo Scientific Theta Probe
 Excitation Source: Al K_{α} monochromatic
 Source Energy: 1486.68 eV
 Source Strength: 300 W
 Source Size: $400\ \mu\text{m} \times 200\ \mu\text{m}$
 Analyzer Type: spherical sector
 Incident Angle: 30°
 Emission Angle: 53°
 Analyzer Pass Energy: 80 eV
 Analyzer Resolution: 0.8 eV
 Total Signal Accumulation Time: 378 s
 Total Elapsed Time: 1020 s
 Number of Scans: 50
 Source Beam Size at Specimen Surface: $400\ \mu\text{m} \times 200\ \mu\text{m}$
 Effective Detector Width: 0.1 eV
 Analyzer Angular Acceptance Width: $30^{\circ} \times 30^{\circ}$
 Comment: surface of Li sample exposed to atmosphere for <1 min, take-off angle = 30°

■ **Accession #:** 01274-12
 ■ **Host Material:** lithium
 ■ **Technique:** XPS
 ■ **Spectral Region:** O 1s

Instrument: Thermo Scientific Theta Probe
 Excitation Source: Al K_{α} monochromatic
 Source Energy: 1486.68 eV
 Source Strength: 300 W
 Source Size: $400\ \mu\text{m} \times 200\ \mu\text{m}$
 Analyzer Type: spherical sector
 Incident Angle: 30°
 Emission Angle: 53°
 Analyzer Pass Energy: 50 eV
 Analyzer Resolution: 0.65 eV
 Total Signal Accumulation Time: 189 s
 Total Elapsed Time: 420 s
 Number of Scans: 25
 Source Beam Size at Specimen Surface: $400\ \mu\text{m} \times 200\ \mu\text{m}$
 Effective Detector Width: 0.1 eV
 Analyzer Angular Acceptance Width: $30^{\circ} \times 30^{\circ}$
 Comment: surface of Li sample exposed to atmosphere for <1 min, take-off angle = 30°