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Metallic antimony (Sb) by XPS

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Herein, survey and high-resolution core level, x-ray induced Auger, and valence photoelectron spectra recorded from a section of a mechanically polished antimony (Sb) rod are presented using monochromatic Al radiation. © *2017 American Vacuum Society*.
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Keywords: *Antimony, XPS, metal*

Accession #: 1422
Technique: XPS
Host Material: Antimony (Sb)
Instrument: Thermo Scientific K-Alpha+
Major Elements in Spectra: Sb
Minor Elements in Spectra: C
Published Spectra: 10
Spectra in Electronic Record: 10
Spectral Category: Reference

INTRODUCTION

Metallic antimony (Sb) is typically widely used in Sn-Pb alloys found in lead-acid batteries and used to quench hydrogen production and improve the characteristics of charging (Ref. 1). Further uses include alloys for antifriction applications (e.g., bullets or bearings from Babbitt-type metal alloys) (Ref. 2) and lead-free solders (Ref. 3) and as a fining agent for specialist glass production (Ref. 4). The toxicity of antimony is well documented (Ref. 5), which has potentially limited surface analysis to typically inorganic antimony materials (Ref. 6) and Sb containing semiconductors/photoconductors (Ref. 7).

The presented spectra serve as an up to date reference for oxygen free metallic Sb, which hitherto has been typically overlooked for analysis with the latest generation of photoelectron spectrometers.

For both proof and clarity of the absence of oxygen, Fig. 1 shows closeup sections of survey spectra from the presented data and Sb-containing oxides, which, due to weak Auger features close to the O KLL region, may be mistaken for oxygen.

SPECIMEN DESCRIPTION (ACCESSION # 1422)

Host Material: Antimony (Sb)

CAS Registry #: 7440-36-0

Host Material Characteristics: Homogeneous; solid; polycrystalline; conductor; metal; others

Chemical Name: Antimony

Source: Unknown

Host Composition: Sb

Form: Rod

Structure: Sb

History and Significance: The rod was one of the many stored in a screw-top glass jar for an unknown period of time. Spectra were recorded as the basis of reference spectra for oxidation and valence band studies.

As Received Condition: The rod was approximately 50 mm in length and 5 mm in diameter. The appearance of the rod was a dull gray color, indicating surface oxidation.

Analyzed Region: Center of the host material

***Ex Situ* Preparation/Mounting:** The sample was cleaved using a sharp blade after immersion of the rod in liquid nitrogen. The cleaved area was a lustrous silver color displaying a highly faceted area. The cleaved sample was wet polished using SiC paper until a visibly flat surface was obtained. The sample was subsequently rinsed and further ultrasonically cleaned with iso-propyl alcohol. The cleaned sample was mounted onto a sample plate using double sided conductive carbon tape.

***In Situ* Preparation:** 4 kV argon ion sputtering over an area of 4 × 4 mm. Cleanliness was assumed once no O KLL signal was detected in the survey spectra or notable contribution of antimony oxide to the Sb 3d spectrum.

Charge Control: None required as conducting

Temp. During Analysis: 300 K

Pressure During Analysis: 5.1×10^{-7} Pa

Preanalysis Beam Exposure: The analyzed region was exposed to less than 60 s of beam exposure during the analysis of auto-height procedures.

INSTRUMENT DESCRIPTION

Manufacturer and Model: Thermo Scientific K-Alpha+

Analyzer Type: Double focussing hemispherical analyzer

Detector: Multichannel resistive plate

Number of Detector Elements: 128

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

Spectrometer

Analyzer Mode: Constant pass energy

Throughput ($T = E^N$): Other

Excitation Source Window: None

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Beam Size: 400 × 400 μm

Signal Mode: Multichannel direct

Geometry

Incident Angle: 60°

Source-to-Analyzer Angle: 60°

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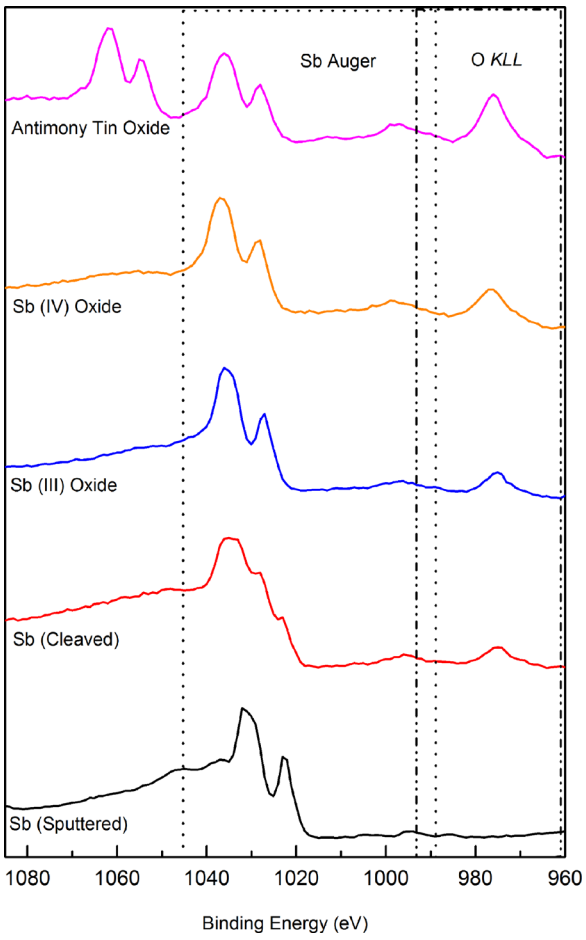

FIG. 1. (Color online) Sections of survey spectra for metallic Sb and associated Sb containing oxides.

Emission Angle: 90°

Specimen Azimuthal Angle: 45°

Acceptance Angle from Analyzer Axis: 60°

Analyzer Angular Acceptance Width: 45° × 0°

■ *Ion gun*

Manufacturer and Model: Thermo Scientific MAGCIS

Energy: 4000 eV

Current: 4.2 mA

Current Measurement Method: Faraday cup

Sputtering Species: Ar^+

Spot Size (unrastered): 120 μm

Raster Size: 4000 × 4000 μm

Incident Angle: 58°

Polar Angle: 58°

Azimuthal Angle: 90°

Comment: Enter ion beam comment.

DATA ANALYSIS METHOD

Energy Scale Correction: Not required

Recommended Energy Scale Shift: Not required

Peak Shape and Background Method: CasaXPS (v2.3.19 rev1.1h) was used to perform background subtraction using a Shirley background and the determination of peak positions and the full width at half maximum (FWHM) values. Asymmetric peaks were used to fit the Sb 3d core levels, while Gaussian peaks were used for all other levels.

Quantitation Method: Atomic concentrations were calculated in CasaXPS using Scofield sensitivity factors with an energy dependence of −0.6.

ACKNOWLEDGMENTS

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SPECTRAL FEATURE TABLE

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
N1422_01	Sb 4d	33.1	…	…	…	…	…
N1422_01	Sb 4p	100.2	…	…	…	…	…
N1422_01	Sb 4s	153.1	…	…	…	…	…
N1422_01	Sb $3d_{5/2}$	528.1	…	…	…	…	…
N1422_01	Sb $3d_{3/2}$	538.1	…	…	…	…	…
N1422_01	Sb $3p_{3/2}$	766.2	…	…	…	…	…
N1422_01	Sb $3p_{1/2}$	812.6	…	…	…	…	…
N1422_01	Sb 3s	945.5	…	…	…	…	…
N1422_01	Sb Auger	985	…	…	…	…	…
N1422_01	Sb Auger	1022.1	…	…	…	…	…
N1422_01	Sb Auger	1031.2	…	…	…	…	…
N1422_02	Valence	…	…	…	…	…	Valence band
N1422_03	Sb $4d_{5/2}$	32.0	0.87	391 845.10	1.85	…	Sb metal
N1422_03	Sb $4d_{3/2}$	33.3	0.87	261 243.10	1.29	…	Sb metal
N1422_04[a]	Sb 4p	99.0	…	…	2.88	…	Sb metal
N1422_05	Sb 4s	152.8	3.61	55 128.60	0.85	…	Sb metal
N1422_06	C 1s	284.5	1.75	4451.7	1.00	…	Residual carbon, possibly implanted by the ion beam
N1422_07	Sb $3d_{5/2}$	528.1	0.77	29 822 576.9	16.39	…	Sb metal
N1422_07	Sb $3d_{3/2}$	537.4	0.76	1 988 384.6	11.35	…	Sb metal
N1422_07[b]	Sb $3d_{5/2}$ satellite	543.8	5.07	691 459.0	…	…	Sb $3d_{5/2}$ satellite
N1422_07[b]	Sb $3d_{3/2}$ satellite	553.2	1.71	58 845.2	…	…	Sb $3d_{3/2}$ satellite
N1422_08[b]	Sb $3p_{3/2}$	766.0	3.33	1 589 019.4	9.77	…	Sb metal
N1422_08[b]	Sb $3p_{3/2}$ Loss	782.0	8.39	514 954.3	…	…	Sb $3p_{3/2}$ loss peak (1)
N1422_08[b]	Sb $3p_{3/2}$ Loss	797.9	4.15	23 715.3	…	…	Sb $3p_{3/2}$ loss peak (2)
N1422_08[b]	Sb $3p_{1/2}$	812.2	3.33	794 509.7	4.76	…	Sb metal
N1422_08[b]	Sb $3p_{1/2}$ Loss	828.2	8.39	257 477.1	…	…	Sb $3p_{3/2}$ loss peak (1)
N1422_08[b]	Sb $3p_{1/2}$ Loss	844.1	4.15	11 857.6	…	…	Sb $3p_{3/2}$ loss peak (2)
N1422_09[c]	Sb 3s	945.1	…	…	…	…	Sb metal
N1422_10[d]	Sb MNN	1022.4	…	…	…	…	Sb $M_4N_{45}N_{45}$
N1422_10[d]	Sb MNN	1031.7	…	…	…	…	Sn $M_5N_{45}N_{45}$

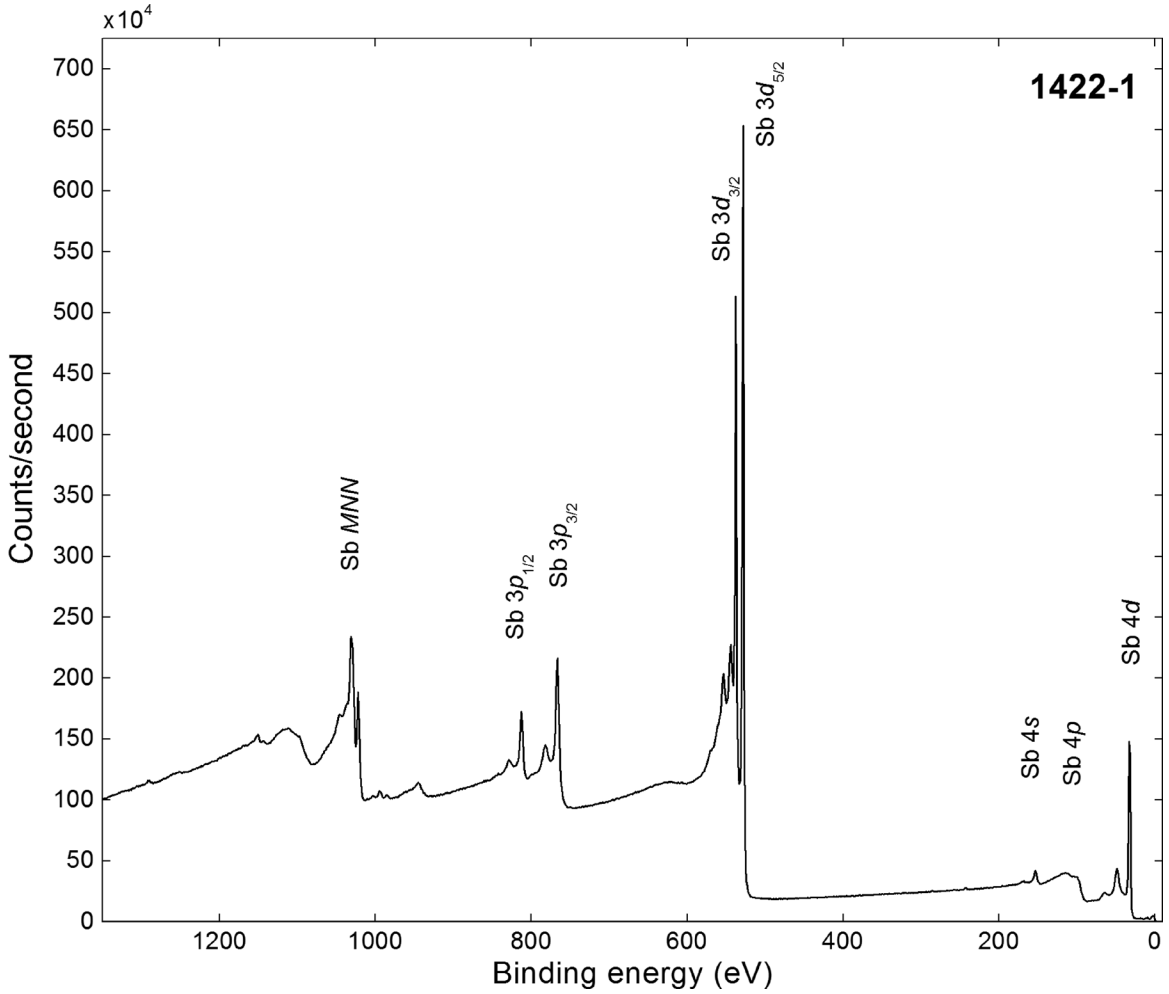
[a]Binding energy determined by taking the differential of a fitted polynomial regression (PR) background type in CasaXPS.
[b]For quantification purposes, the satellite structure should be included in the peak area calculation if the metallic content is sufficiently high.
[c]Very broad structured peak, binding energy taken to be the highest peak position.
[d]Sb also exhibits weak Auger transitions around 945 eV, which are not to be confused with neighboring O KLL Auger peaks.

ANALYZER CALIBRATION TABLE

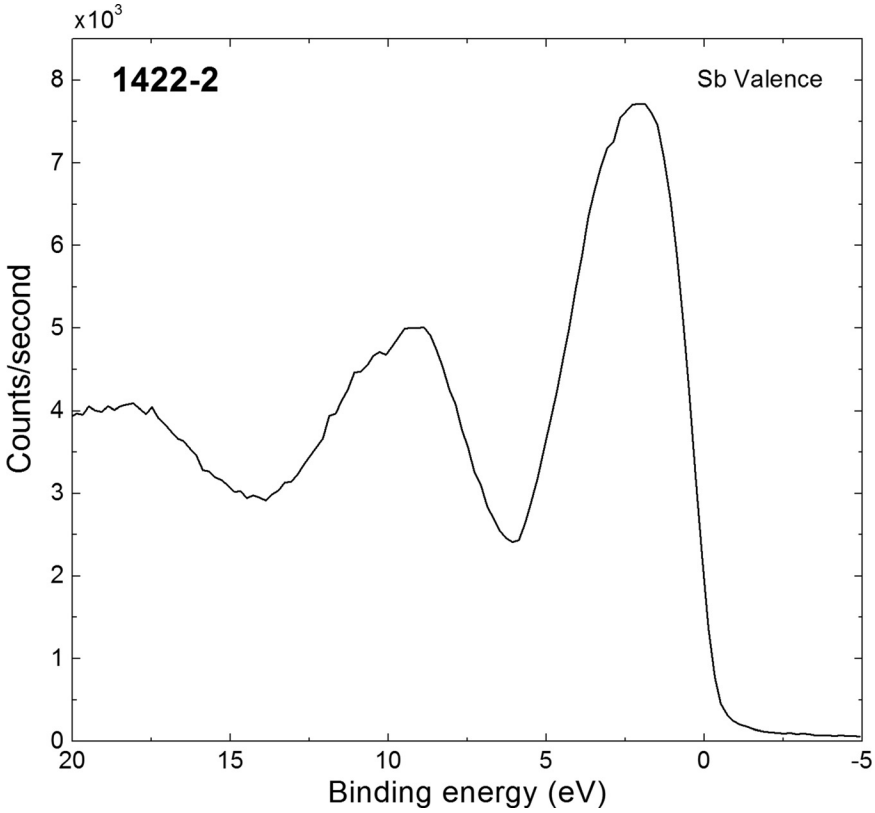
Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
1	Au $4f_{7/2}$	83.99	0.76	1 597 652	9.58	100	Gold metal
2	Ag $3d_{5/2}$	368.28	0.58	1 876 744	7.38	100	Silver metal
3	Cu $2p_{3/2}$	932.67	0.83	2 205 571	16.73	100	Copper metal

GUIDE TO FIGURES

Spectrum (Accession) #	Spectral Region	Voltage Shift	Multiplier	Baseline	Comment #
N1422_01	Survey	0	1	0	…
N1422_02	Valence	0	1	0	…
N1422_03	Sb 4d	0	1	0	…
N1422_04	Sb 4p	0	1	0	…
N1422_05	Sb 4s	0	1	0	…
N1422_06	C 1s	0	1	0	…
N1422_07	Sb 3d	0	1	0	…
N1422_08	Sb 3p	0	1	0	…
N1422_09	Sb 3s	0	1	0	…
N1422_10	Sb MNN	0	1	0	…



Accession #	1422–01
Host Material	Antimony (Sb)
Technique	XPS
Spectral Region	Survey
Instrument	Thermo Scientific K-Alpha^{+}
Excitation Source	Al Ka monochromatic
Source Energy	1486.6 eV
Source Strength	72 W
Source Size	0.004 × 0.004 mm
Analyzer Type	Double focussing hemispherical analyzer
Incident Angle	60°
Emission Angle	90°
Analyzer Pass Energy	150 eV
Analyzer Resolution	0.1 eV
Total Signal Accumulation Time	130 s
Total Elapsed Time	260 s
Number of Scans	10
Effective Detector Width	1 eV



- **Accession #:** 1422-02
- **Host Material:** Antimony (Sb)
- **Technique:** XPS
- **Spectral Region:** Valence

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.004 × 0.004 mm

Analyzer Type: Double focussing hemispherical analyzer

Incident Angle: 60°

Emission Angle: 90°

Analyzer Pass Energy: 40 eV

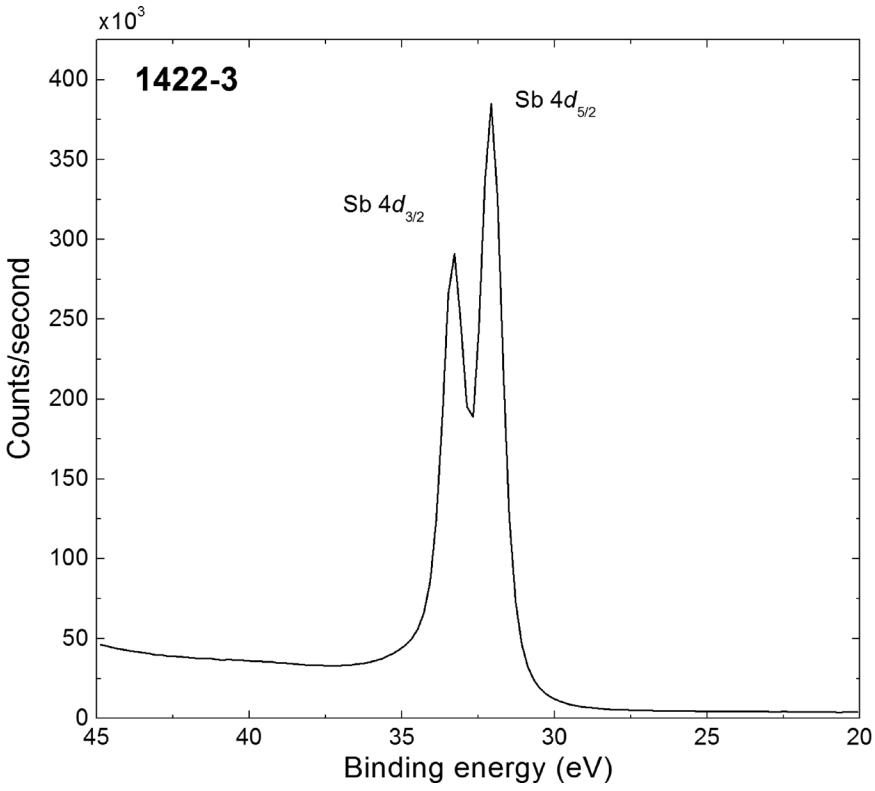
Analyzer Resolution: 0.1 eV

Total Signal Accumulation Time: 120 s

Total Elapsed Time: 153 s

Number of Scans: 20

Effective Detector Width: 0.1 eV



- **Accession #:** 1422-03
- **Host Material:** Antimony (Sb)
- **Technique:** XPS
- **Spectral Region:** Sb 4d

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.004 × 0.004 mm

Analyzer Type: Double focussing hemispherical analyzer

Incident Angle: 60°

Emission Angle: **90**°

Analyzer Pass Energy: 40 eV

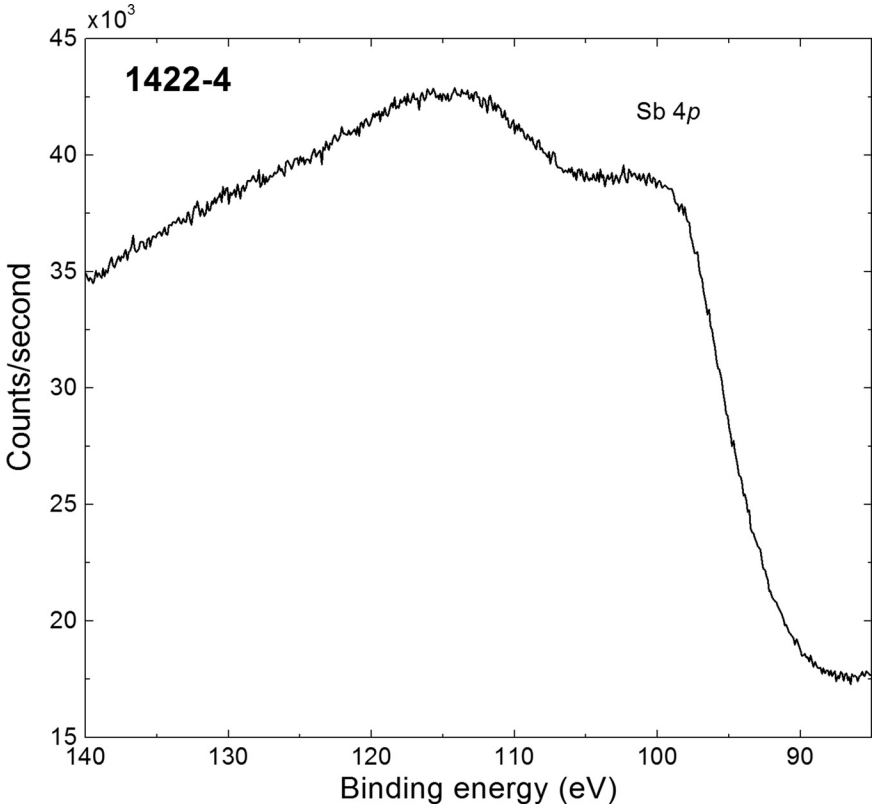
Analyzer Resolution: 0.1 eV

Total Signal Accumulation: Time: 120 s

Total Elapsed Time: 153 s

Number of Scans: 20

Effective Detector Width: 0.1 eV



- **Accession #:** 1422-04
- **Host Material:** Antimony (Sb)
- **Technique:** XPS
- **Spectral Region:** Sb 4p

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.004 × 0.004 mm

Analyzer Type: Double focussing hemispherical analyzer

Incident Angle: 60°

Emission Angle: 90°

Analyzer Pass Energy: 40 eV

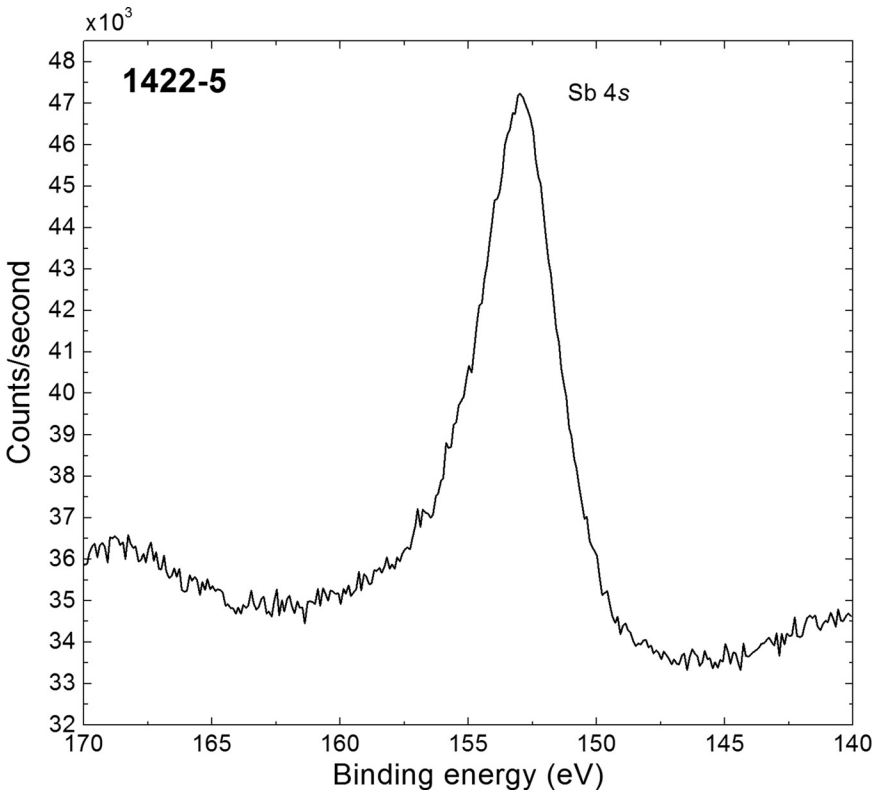
Analyzer Resolution: 0.1 eV

Total Signal Accumulation Time: 120 s

Total Elapsed Time: 153 s

Number of Scans: 20

Effective Detector Width: 0.1 eV



- **Accession #:** 1422-05
- **Host Material:** Antimony (Sb)
- **Technique:** XPS
- **Spectral Region:** Sb 4s

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.004 × 0.004 mm

Analyzer Type: Double focussing hemispherical analyzer

Incident Angle: 60°

Emission Angle: 90°

Analyzer Pass Energy: 40 eV

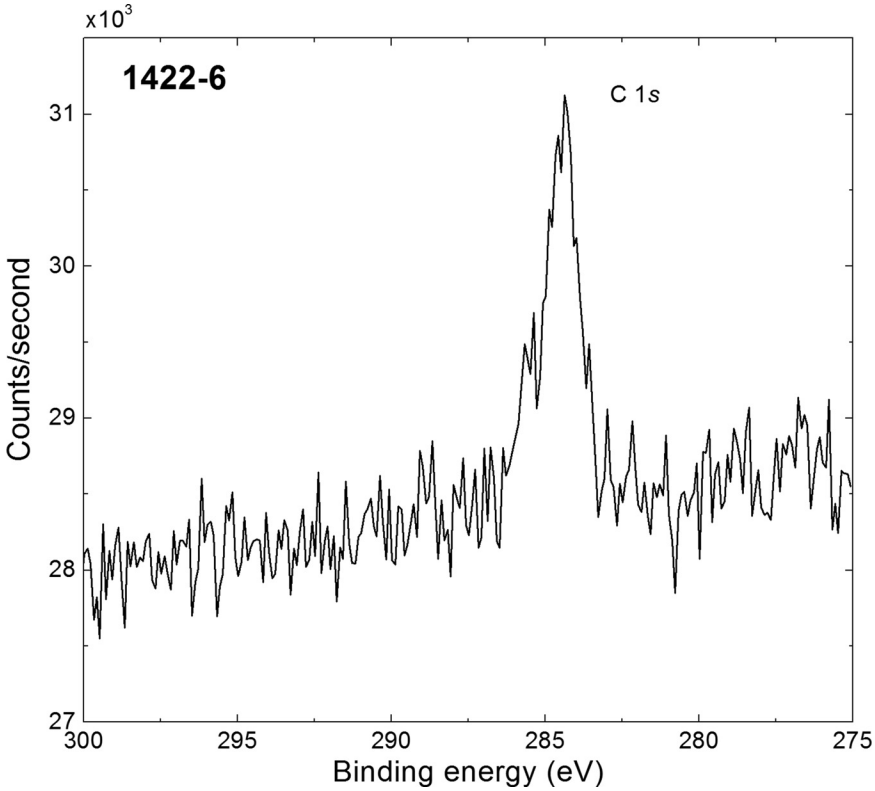
Analyzer Resolution: 0.1 eV

Total Signal Accumulation Time: 120 s

Total Elapsed Time: 153 s

Number of Scans: 20

Effective Detector Width: 0.1 eV



- **Accession #:** 1422-06
- **Host Material:** Antimony (Sb)
- **Technique:** XPS
- **Spectral Region:** C 1s

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.004 × 0.004 mm

Analyzer Type: Double focussing hemispherical analyzer

Incident Angle: 60°

Emission Angle: 90°

Analyzer Pass Energy: 40 eV

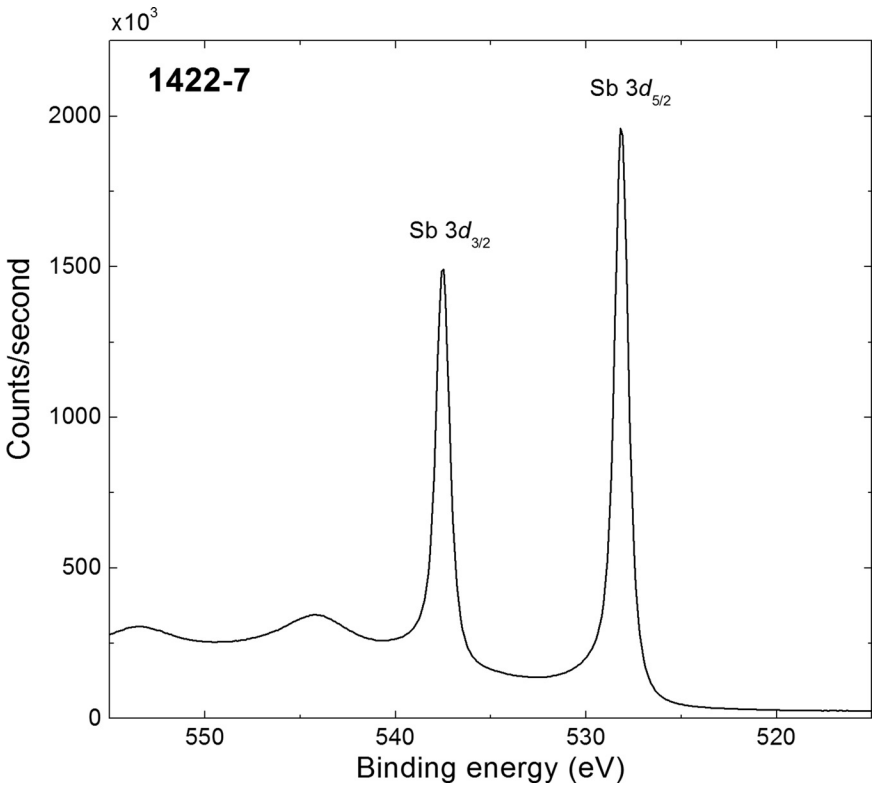
Analyzer Resolution: 0.1 eV

Total Signal Accumulation Time: 120 s

Total Elapsed Time: 153 s

Number of Scans: 20

Effective Detector Width: 0.1 eV



- **Accession #:** 1422-07
- **Host Material:** Antimony (Sb)
- **Technique:** XPS
- **Spectral Region:** Sb 3d

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.004 × 0.004 mm

Analyzer Type: Double focussing hemispherical analyzer

Incident Angle: 60°

Emission Angle: 90°

Analyzer Pass Energy: 40 eV

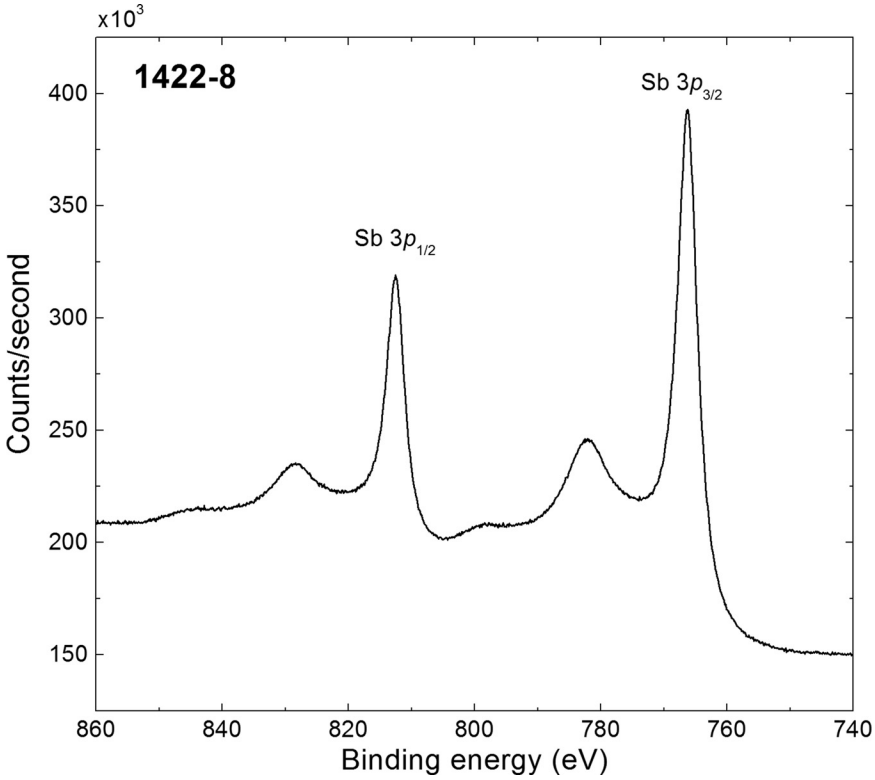
Analyzer Resolution: 0.1 eV

Total Signal Accumulation Time: 120 s

Total Elapsed Time: 153 s

Number of Scans: 20

Effective Detector Width: 0.1 eV



- **Accession #:** 1422-08
- **Host Material:** Antimony (Sb)
- **Technique:** XPS
- **Spectral Region:** Sb 3p

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.004 × 0.004 mm

Analyzer Type: Double focussing hemispherical analyzer

Incident Angle: 60°

Emission Angle: 90°

Analyzer Pass Energy: 40 eV

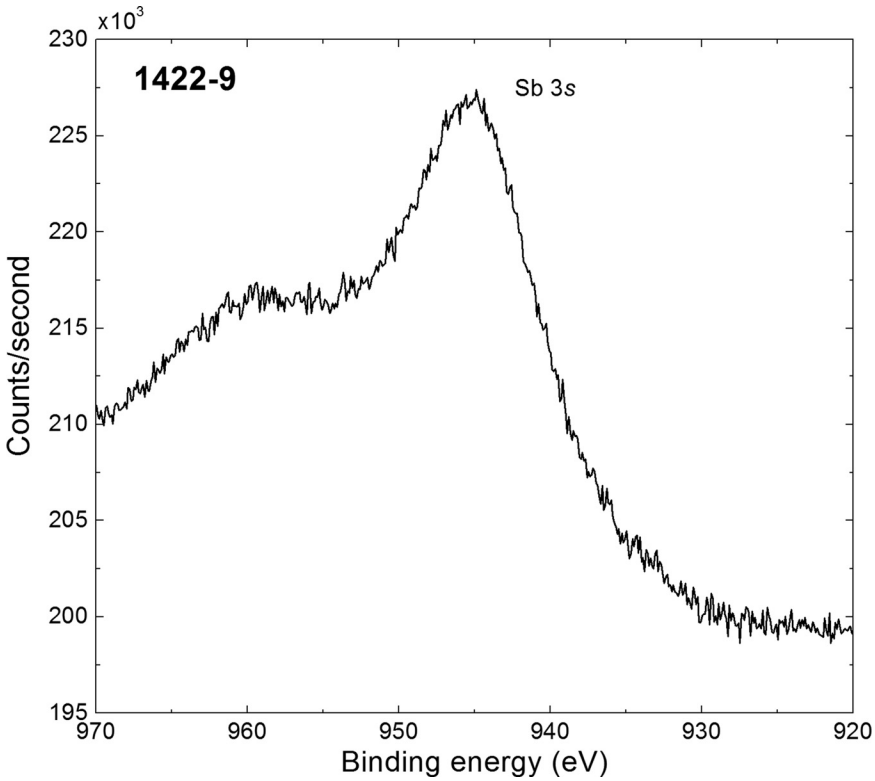
Analyzer Resolution: 0.1 eV

Total Signal Accumulation Time: 120 s

Total Elapsed Time: 153 s

Number of Scans: 20

Effective Detector Width: 0.1 eV



- **Accession #:** 1422-09
- **Host Material:** Antimony (Sb)
- **Technique:** XPS
- **Spectral Region:** Sb 3s

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.004 × 0.004 mm

Analyzer Type: Double focussing hemispherical analyzer

Incident Angle: 60°

Emission Angle: 90°

Analyzer Pass Energy: 40 eV

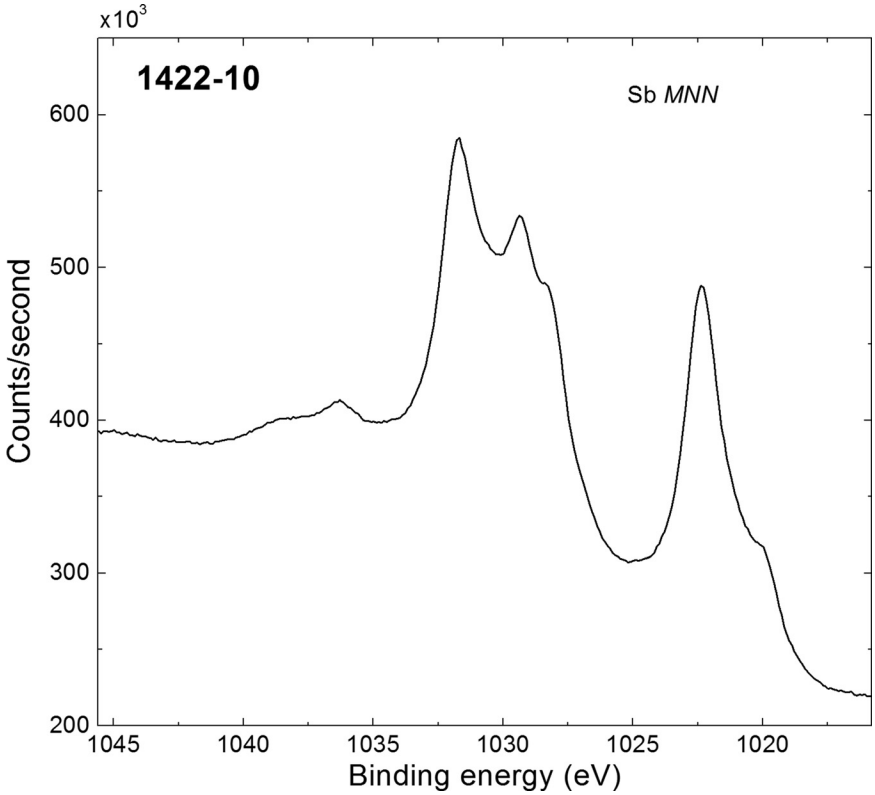
Analyzer Resolution: 0.1 eV

Total Signal Accumulation Time: 120 s

Total Elapsed Time: 153 s

Number of Scans: 20

Effective Detector Width: 0.1 eV



- **Accession #:** 1422-10
- **Host Material:** Antimony (Sb)
- **Technique:** XPS
- **Spectral Region:** Sb MNN

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.004 × 0.004 mm

Analyzer Type: Double focussing hemispherical analyzer

Incident Angle: 60°

Emission Angle: 90°

Analyzer Pass Energy: 40 eV

Analyzer Resolution: 0.1 eV

Total Signal Accumulation Time: 120 s

Total Elapsed Time: 153 s

Number of Scans: 20

Effective Detector Width: 0.1 eV