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Zinc sulfide chemical vapor deposition optical ceramic analyzed by XPS

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Zinc sulfide chemical vapor deposition optical ceramic analyzed by XPS

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Kathleen A. Richardson,^{1,2}  and Parag Banerjee^{1,3,4,5,a)} 

AFFILIATIONS

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ABSTRACT

Commercially available zinc sulfide (ZnS) ceramics, synthesized using chemical vapor deposition and subsequently hot isostatically pressed, are optically transparent as compared to other zinc sulfide chemical vapor deposited films and ceramics. The unique optical transparency along with the hardness and chemical resistance of ZnS makes it an important material for infrared transmission applications. X-ray photoelectron spectroscopy and x-ray induced Auger electron spectroscopy were performed on an optically transparent ZnS ceramic. This report includes charge corrected scans for the survey along with S 2s, S 2p, S LMM, Zn 2p, Zn 2s, Zn 3d, Zn 3p, Zn 3s, Zn LMM, O 1s, and C 1s surface photoelectron signals. The scans provide photoelectron spectroscopy investigation data to help with the identification of metal sulfide compounds.

Key words: ZnS, sulfides, chemical vapor deposition, CVD, ceramics, x-ray photoelectron spectroscopy, XPS

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Accession #: 01862

Technique: XPS, XAES

Host Material: Zinc Sulfide CVD Optical Ceramic

Instrument: XPS ESCALAB 250Xi Fisher Scientific

Major Elements in Spectra: Zn, S

Minor Elements in Spectra: O, C

Published Spectra: 12

Spectral Category: Comparison

INTRODUCTION

Zinc sulfide (ZnS) ceramics transmit in the infrared wavelength range between 1 and 12 μm (Ref. 1). This material is also semiconducting with a bandgap of 3.67 eV and exhibits unique optical and electrical properties, making it a popular choice for various applications, such as thermal imaging cameras, night vision devices, and lasers (Refs. 2–4).

Chemical vapor deposition (CVD) allows for the deposition of bulk ZnS ceramics with precise control over thickness, uniformity, and crystalline structure. This makes it possible to produce high-quality ZnS with low defects and excellent optical properties,

including high optical transparency, low absorption, and high refractive index (2.35 ± 0.01) (Ref. 5). The resulting ZnS are also durable and resistant to environmental degradation. While the ceramics made by CVD are of high quality, the visual clarity of the material is not clear. Upon hot isostatic pressing, the ceramics transform into optically transparent substrates.

There are little to no reports of the x-ray photoelectron spectroscopy (XPS) of ZnS transparent ceramics. The reports available are either incomplete or the available XPS spectral characterization is for ZnS with other form factors such as thin films, powders, and nanoparticles (Refs. 2 and 6–8).

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In this study, we analyze ZnS CVD transparent ceramic by XPS to study and quantify the bonding states of observable electron shells for Zn and S, along with minor species C and O. The use of XPS also provides quantification of atomic percentages (i.e., stoichiometry) along with the bonding states of trace level impurities, if present. From the oxygen (O 1s) fine spectra, we see that the material has surface level oxidation. The sulfur fine spectrum (S 2p) does not show evidence of oxidized sulfur, indicating that O is associated primarily with zinc.

SPECIMEN DESCRIPTION (ACCESSION # 01862)

Host Material: Zinc Sulfide Bulk Ceramic

CAS Registry #: 1314-98-3

Host Material Characteristics: Homogeneous; solid; polycrystalline; semiconductor; inorganic compound; Ceramic

Chemical Name: Zinc Sulfide

Source: APC American Photonics, 6621 19th Street East Sarasota, FL 34243, USA

Host Composition: ZnS

Form: CVD Zinc Sulfide Ceramic

Structure: Microcrystalline cubic zinc blend

History & Significance: The specimen was received from APC American Photonics in an optics case with lens tissue and lining and stored in normal atmosphere at room temperature. ZnS is commonly used in infrared windows, domes, and optical elements. While the ZnS nanocrystalline CVD thin film has been cataloged in the past, this work is done on a commercially available ZnS CVD ceramic sample that is optically transparent. The samples were used as received.

As Received Condition: Optically clear ceramic window

Analyzed Region: Same as the host material

Ex Situ Preparation/Mounting: Ceramic was sonicated for 5 min in hexane, then transferred to isopropanol and sonicated for 5 min, and finally transferred to de-ionized water for a final 5 min of sonication. The sample was then dried by compressed air. The sample was mounted on the XPS stage using double-sided carbon tape.

In Situ Preparation: 600 s argon ion sputtering at 500 eV was used to clean the surface before analysis.

Charge Control: Charge compensation is delivered by both an in-lens electrostatic electron flood source (1 eV, 100 μ A) and a dual-beam low-energy electron and ion coaxial flood source (2 eV, 100 μ A).

Temp. During Analysis: 300 K

Pressure During Analysis: 5×10^{-8} Pa

Pre-analysis Beam Exposure: 0 s

INSTRUMENT DESCRIPTION

Manufacturer and Model: Thermo Fisher Scientific ESCALAB 250Xi

Analyzer Type: Spherical sector

Detector: Channeltron

Number of Detector Elements: 6

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

Spectrometer

Analyzer Mode: Constant pass energy

Throughput ($T = E^N$): Calculated from a polynomial fit to a plot of $\log[\text{peak area}/(\text{PE} \times \text{XSF})]$ versus $\log[\text{KE}/\text{PE}]$, where PE is the pass energy, KE is the kinetic energy, and XSF is the relative sensitivity factor.

Excitation Source Window: None

Excitation Source: Al K_{α} monochromatic

Source Energy: 1486.6 eV

Source Strength: 200 W

Source Beam Size: $650 \times 650 \mu\text{m}^2$

Signal Mode: Single channel direct

Geometry

Incident Angle: 58°

Source-to-Analyzer Angle: 58°

Emission Angle: 0°

Specimen Azimuthal Angle: 90°

Acceptance Angle from Analyzer Axis: 45°

Analyzer Angular Acceptance Width: $22.5^\circ \times 22.5^\circ$

Ion Gun

Manufacturer and Model: Thermo Fisher Scientific EX03 Ion Gun System

Energy: 500 eV

Current: 0.02 mA

Current Measurement Method: biased stage

Sputtering Species: Ar⁺

Spot Size (unrastered): 500 μm

Raster Size: $4500 \times 4500 \mu\text{m}^2$

Incident Angle: 40°

Polar Angle: 40°

Azimuthal Angle: 270°

Comment: These parameters correspond to ion-cleaning methods used in optical materials requiring surface cleaning.

DATA ANALYSIS METHOD

Energy Scale Correction: Binding energy scale was referenced to C 1s = 284.8 eV fine spectra pre and post sputter

Recommended Energy Scale Shift: Shift +0.15 eV

Peak Shape and Background Method: THERMO SCIENTIFIC AVANTAGE software version 5.9902 was used for peak shape and background subtraction. The smart (Shirley function) was used to subtract the background for Zn 2p, S 2p, O 1s, and C 1s peaks. Using the smart feature utilizes revised constraints that limit the background from having greater intensity than data from points in the collection region.

Quantitation Method: Atomic percentages were calculated using the THERMO SCIENTIFIC AVANTAGE software version 5.9902. Sensitivity factors were obtained from the THERMO SCIENTIFIC AVANTAGE software database and were used in the calculation of elemental atomic percentages. The peak library is ALWAG (Ref. 9).

ACKNOWLEDGMENTS

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Brian Butkus: Conceptualization (lead); Data curation (lead); Formal analysis (lead); Investigation (lead); Methodology (lead); Software (lead); Visualization (lead); Writing – original draft (lead); Writing – review & editing (equal). **Alexandros Kostogiannes:** Investigation (supporting); Validation (supporting); Writing – review & editing (supporting). **Andrew Howe:** Investigation (supporting); Visualization (supporting); Writing – review & editing (supporting). **Myungkoo Kang:** Validation (supporting); Visualization (supporting); Writing – review & editing (supporting). **Romain Gaume:** Conceptualization (supporting); Formal analysis (supporting); Funding acquisition (equal); Project administration (equal); Resources (equal); Supervision (supporting); Validation (supporting); Writing – review & editing (supporting). **Kathleen A. Richardson:** Conceptualization (supporting); Formal analysis (supporting); Funding acquisition (lead); Project administration (equal); Resources (equal); Supervision (supporting); Validation (supporting); Visualization (supporting); Writing – review & editing

(equal). **Parag Banerjee:** Conceptualization (equal); Formal analysis (supporting); Funding acquisition (equal); Investigation (supporting); Project administration (equal); Resources (equal); Supervision (lead); Validation (equal); Visualization (supporting); Writing – review & editing (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
01862-02	S 2s	226.29	2.10	21 373.08	1.294	...	Zinc sulfide
01862-03	S 2p _{3/2}	162.17	1.97	34 340.82	1.881	45.22	Zinc sulfide
01862-03	S 2p _{1/2}	163.35	Zinc sulfide
01862-04 ^a	S LMM	151.32	7.09	78 819.67	0.460	...	Zinc sulfide
01862-05	Zn 2p _{3/2}	1021.74	1.45	326 328.37	21.391	...	Zinc sulfide
01862-05	Zn 2p _{1/2}	1044.81	1.52	157 131.76	10.471	...	Zinc sulfide
01862-06	Zn 2s	1195.70	5.15	48 691.46	4.200	...	Zinc sulfide
01862-07	Zn 3d	10.75	1.28	21 996.46	0.716	...	Zinc sulfide
01862-08	Zn 3p _{3/2}	89.24	2.63	50 018.35	2.600	46.19	Zinc sulfide
01862-08	Zn 3p _{1/2}	92.00	Zinc sulfide
01862-09	Zn 3s	140.24	2.37	17 085.14	0.800	...	Zinc sulfide
01862-10 ^a	Zn LMM	989.58	2.14	66 530.64	4.916	...	Zinc sulfide
01862-10 ^a	Zn LMM	992.59	3.37	43 942.32	4.916	...	Zinc sulfide
01862-11 ^{b,c}	O 1s	531.24	...	4 177.20	2.881	3.90	Metal carbonate, metal oxide
01862-12	C 1s	284.75	1.63	1 861.12	1.000	4.69	Carbon

^aPeak energy in kinetic energy (KE).

^bQuantification of oxygen is not included due to interaction with Auger electron peaks.

^cOverlap between Zn LMM Auger electron signal and O 1s peak [see comment to (01862-01)]; due to this overlap, we have avoided the evaluation of the O 1s FWHM and estimated only the BE (footnote to 01862-11).

Footnote to 01862-03: The measured S 2p is used for atomic percentage calculation for more accurate representation when compared to Zn 3d and can be more representative of the actual atomic percentage. The S 2p position was centered at BEs typical for sulfide species BE (S 2p_{3/2})=162.17 eV; FWHM=1.97 eV, and the peak for a sulfite or sulfate are shifted to the higher BEs with the sulfates at BEs≈7.0 eV higher (Refs. 6 and 10).

Footnote to 01862-05: The measured Zn 2p_{3/2} position binding energy (BE)=1021.74 eV, and a full width at half maximum (FWHM)=1.45 eV was not consistent with handbook data on zinc sulfide but was consistent with D. Barreca *et al.*, since reported values for the Zn 2p_{3/2} BEs for both zinc sulfide and zinc oxide are similar ZnS (1021.6 eV) and ZnO (1022.1 eV) (Refs. 8, 11, and 12). As with previous articles on ZnS, we evaluated the Auger electron α parameter, α =BE(Zn 2p_{3/2}) + KE(Zn LMM) (Ref. 13) was used to confirm our evaluations to identify ZnS presence. The obtained value (2011.32 eV) agreed with those previously reported in the literature for zinc sulfide (Refs. 8, 10, 14, and 15).

Footnote to 01862-08: The Zn 3p photoelectron peak was used to obtain a more accurate evaluation of atomic percentages, the S/Zn ratio. If the Zn 2p_{3/2} peak is used, the intensity of the Zn signal peak is non-congruent. This intensity difference would be represented as different escape depths and would result in off stoichiometry for the Zn:S ratio (Refs. 8 and 13).

Footnote to 01862-10: The KE of the Zn LMM peak (989.62 eV) was evaluated by the most intense Auger electron peak component.

Footnote to 01862-11: The O 1s signal was fitted by a single component (BE=531.24) (Refs. 8 and 16). The O 1s peak signal intensity was reduced to just above noise level after a sputtering Ar⁺ erosion. Due to the overlap between Zn LMM Auger electron signal and O 1s peak (see footnote to 01862-10), the relative percentages could not be determined by direct measurement of the O 1s peak area. And for this reason, at.% will be left out of these calculations.

Footnote to 01862-12: Carbon presence was limited to surface contamination since the post-sputtering Ar⁺, C 1s peak reduced in intensity to just above the noise in the fine spectra and noise level in the survey.

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ANALYZER CALIBRATION TABLE

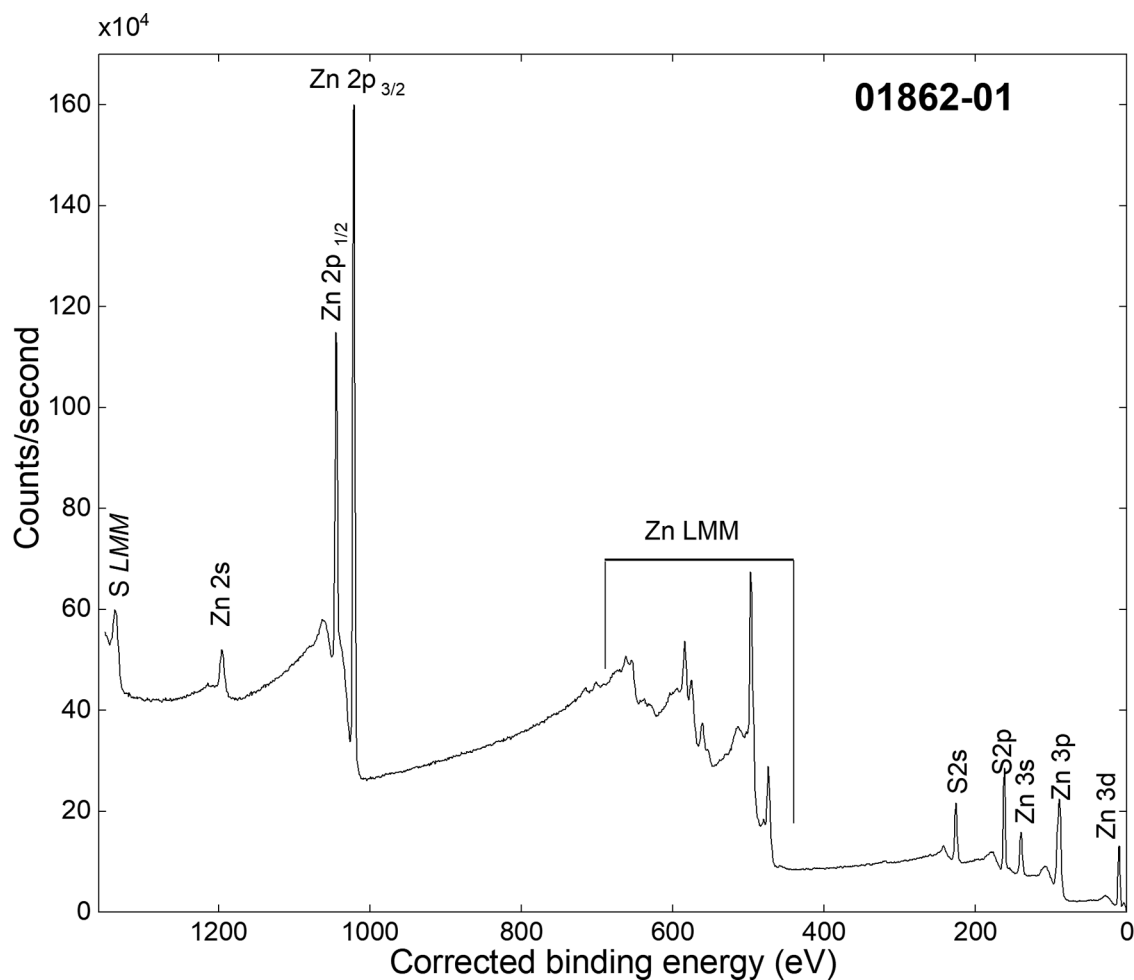
Spectrum ID #	Element/Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV counts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
...	Au 4f	84.05	0.57	262 830.05	20.735	...	Au
...	Ag 3d	368.36	0.48	386 600.57	22.131	...	Ag
...	Cu 2p	932.8	0.77	655 133.11	26.513	...	Cu

GUIDE TO FIGURES

Spectrum (Accession) #	Spectral Region	Voltage Shift ^a	Multiplier	Baseline	Comment #
01862-01	Survey	-0.15	1	0	1
01862-02	S 2s	-0.15	1	0	1
01862-03	S 2p	-0.15	1	0	1
01862-04	S LMM	+0.15	1	0	1
01862-05	Zn 2p	-0.15	1	0	1
01862-06	Zn 2s	-0.15	1	0	1
01862-07	Zn 3d	-0.15	1	0	1
01862-08	Zn 3p	-0.15	1	0	1
01862-09	Zn 3s	-0.15	1	0	1
01862-10	Zn LMM	+0.15	1	0	1
01862-11	O 1s	-0.15	1	0	1
01862-12	C 1s	-0.15	1	0	1

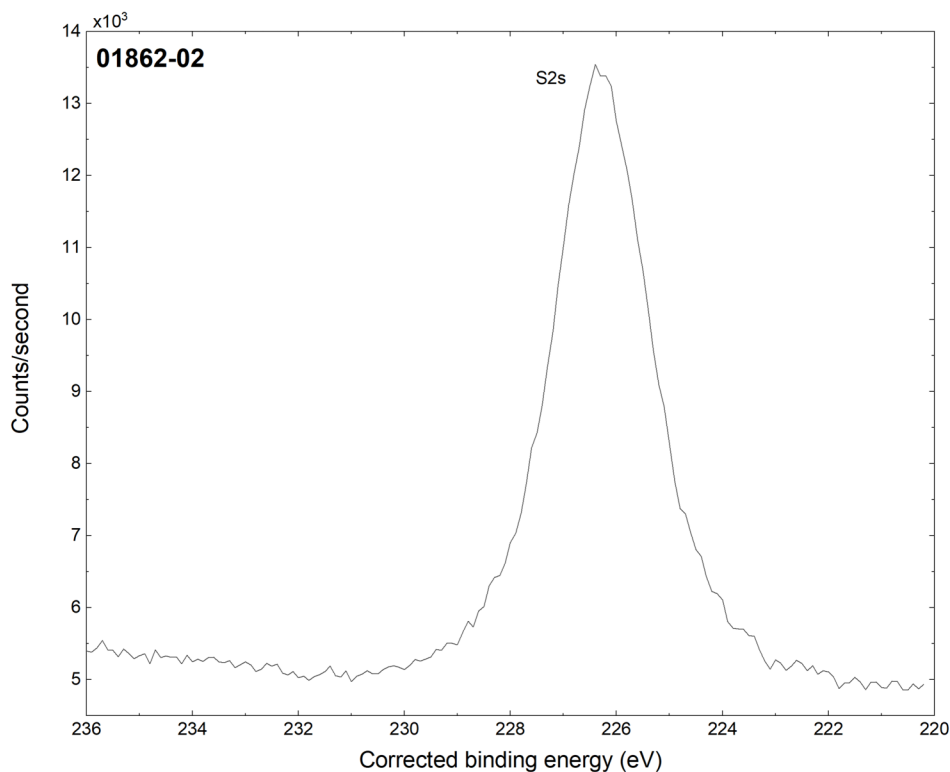
^aVoltage shift of the archived (as-measured) spectrum relative to the printed figure. The figure reflects the recommended energy scale correction due to a calibration correction, sample charging, flood gun, or other phenomenon.

1. ZnS Ceramic.



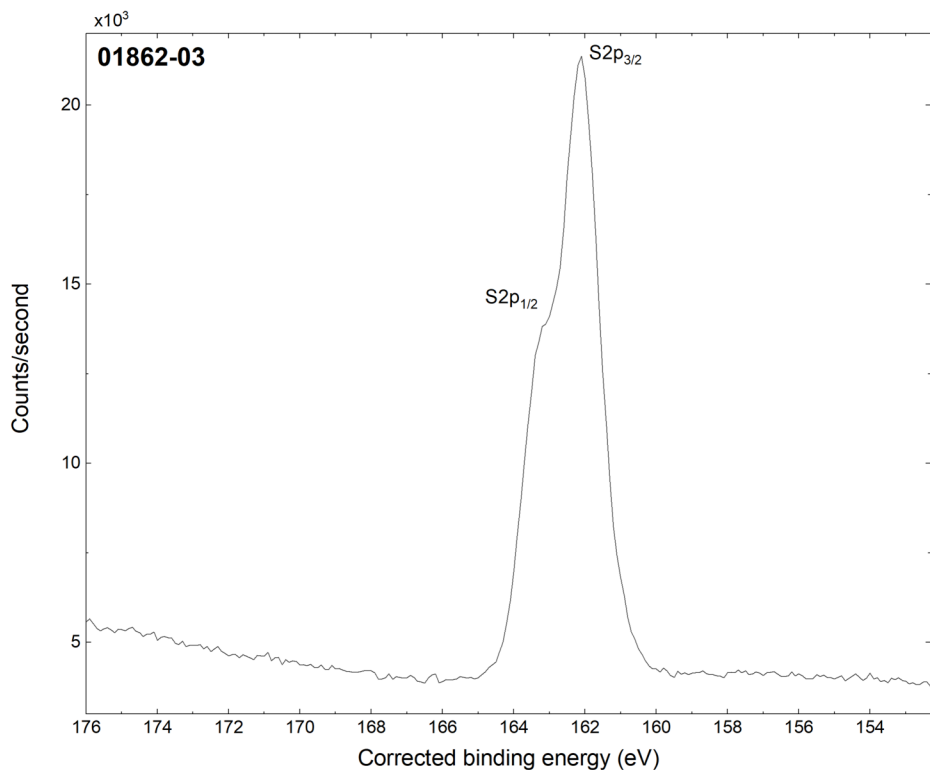
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Accession #	01862-01
Host Material:	ZnS
Technique:	XPS
Spectral Region:	Survey
Instrument:	Thermo Fisher Scientific ESCALAB 250Xi
Excitation Source:	Al K_{α} monochromatic
Source Energy:	1486.6 eV
Source Strength:	200 W
Source Size:	0.9 \times 0.9 mm ²
Analyzer Type:	Spherical sector analyzer
Incident Angle:	58°
Emission Angle:	0°
Analyzer Pass Energy:	150 eV
Analyzer Resolution:	1.000 eV
Total Signal Accumulation Time:	136 s
Total Elapsed Time:	229 s
Number of Scans:	5
Effective Detector Width:	25.441 eV
Comments:	Number of Energy Steps 1361



- **Accession #:** 01862-02
- **Host Material:** ZnS
- **Technique:** XPS
- **Spectral Region:** S 2s

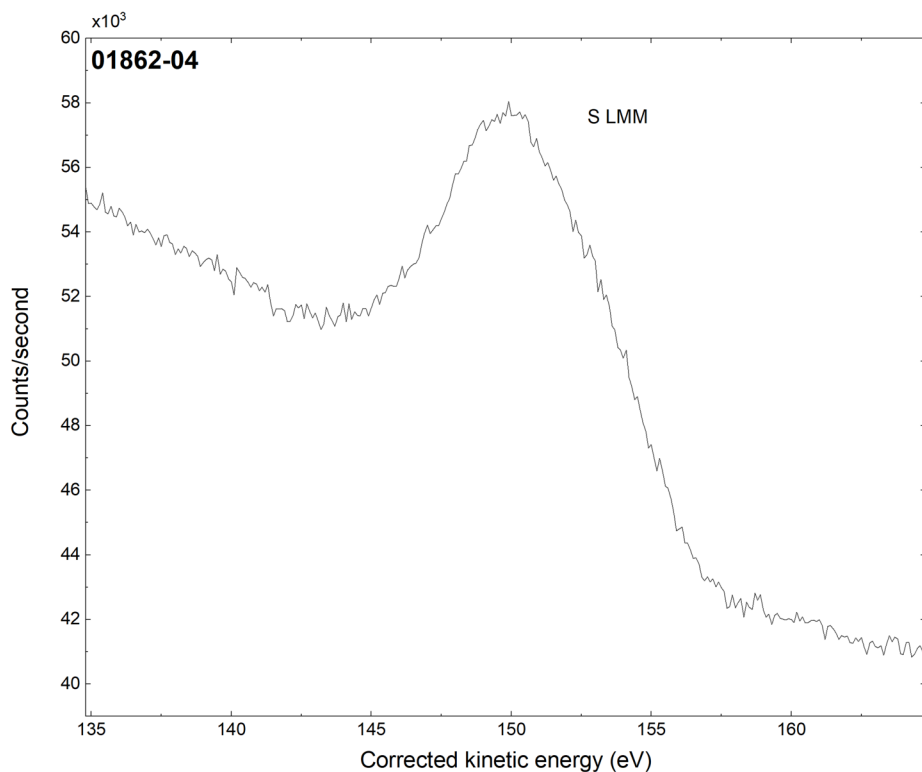
Instrument: Thermo Fisher Scientific
 ESCALAB 250Xi
 Excitation Source: Al K_{α} monochromatic
 Source Energy: 1486.6 eV
 Source Strength: 200 W
 Source Size: $0.9 \times 0.9 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 58°
 Emission Angle: 0°
 Analyzer Pass Energy: 20 eV
 Analyzer Resolution: 0.100 eV
 Total Signal Accumulation Time: 135.7 s
 Total Elapsed Time: 172 s
 Number of Scans: 15
 Effective Detector Width: 3.392 eV



- **Accession #:** 01862-03
- **Host Material:** ZnS
- **Technique:** XPS
- **Spectral Region:** S 2p

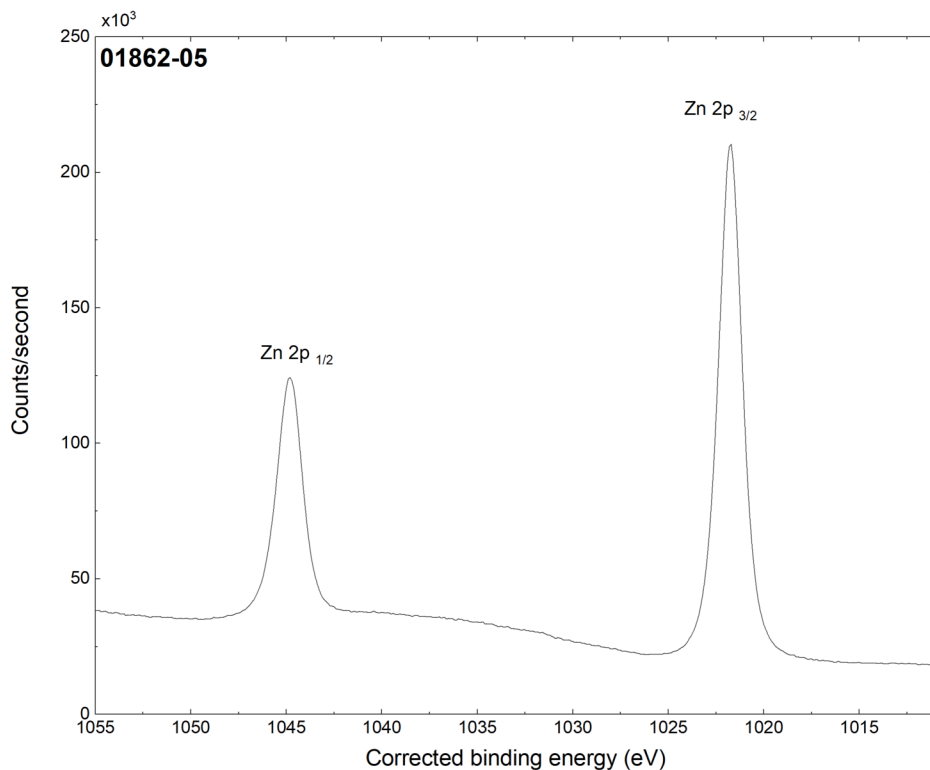
Instrument: Thermo Fisher Scientific
 ESCALAB 250Xi
 Excitation Source: Al K_{α} monochromatic
 Source Energy: 1486.6 eV
 Source Strength: 200 W
 Source Size: $0.9 \times 0.9 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 58°
 Emission Angle: 0°
 Analyzer Pass Energy: 20 eV
 Analyzer Resolution: 0.100 eV
 Total Signal Accumulation Time: 210.7 s
 Total Elapsed Time: 252 s
 Number of Scans: 15
 Effective Detector Width: 3.392 eV
 Comments: See footnotes SPECTRAL
 FEATURES TABLE.

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- Accession #: 01862-04
- Host Material: ZnS
- Technique: XAES
- Spectral Region: S LMM

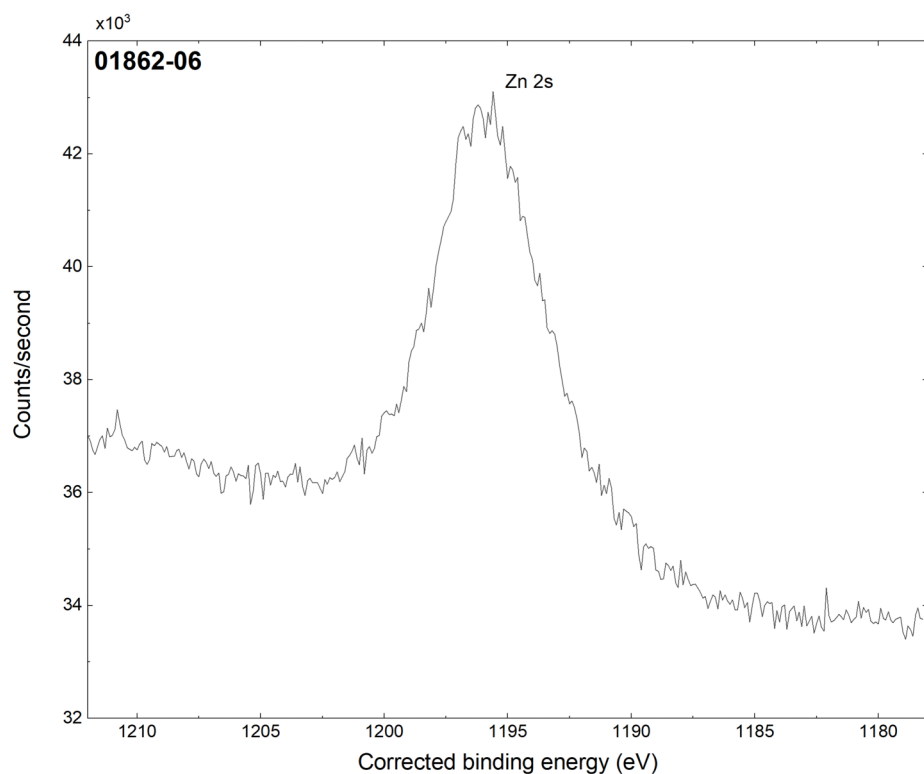
Instrument: Thermo Fisher Scientific ESCALAB 250Xi
 Excitation Source: Al K_{α} monochromatic
 Source Energy: 1486.6 eV
 Source Strength: 200 W
 Source Size: $0.9 \times 0.9 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 58°
 Emission Angle: 0°
 Analyzer Pass Energy: 20 eV
 Analyzer Resolution: 0.100 eV
 Total Signal Accumulation Time: 225.7 s
 Total Elapsed Time: 268 s
 Number of Scans: 15
 Effective Detector Width: 3.392 eV



- Accession #: 01862-05
- Host Material: ZnS
- Technique: XPS
- Spectral Region: Zn 2p

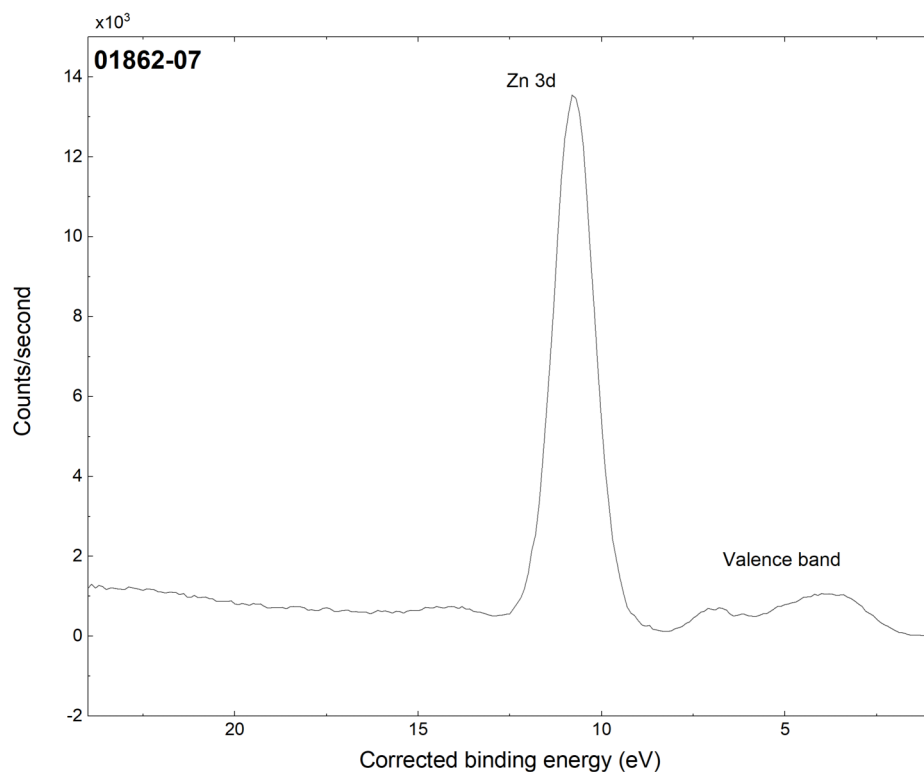
Instrument: Thermo Fisher Scientific ESCALAB 250Xi
 Excitation Source: Al K_{α} monochromatic
 Source Energy: 1486.6 eV
 Source Strength: 200 W
 Source Size: $0.9 \times 0.9 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 58°
 Emission Angle: 0°
 Analyzer Pass Energy: 20 eV
 Analyzer Resolution: 0.100 eV
 Total Signal Accumulation Time: 353.1 s
 Total Elapsed Time: 406 s
 Number of Scans: 15
 Effective Detector Width: 3.392 eV
 Comments: See footnotes SPECTRAL FEATURES TABLE

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■ **Accession #:** 01862-06
 ■ **Host Material:** ZnS
 ■ **Technique:** XPS
 ■ **Spectral Region:** Zn 2s

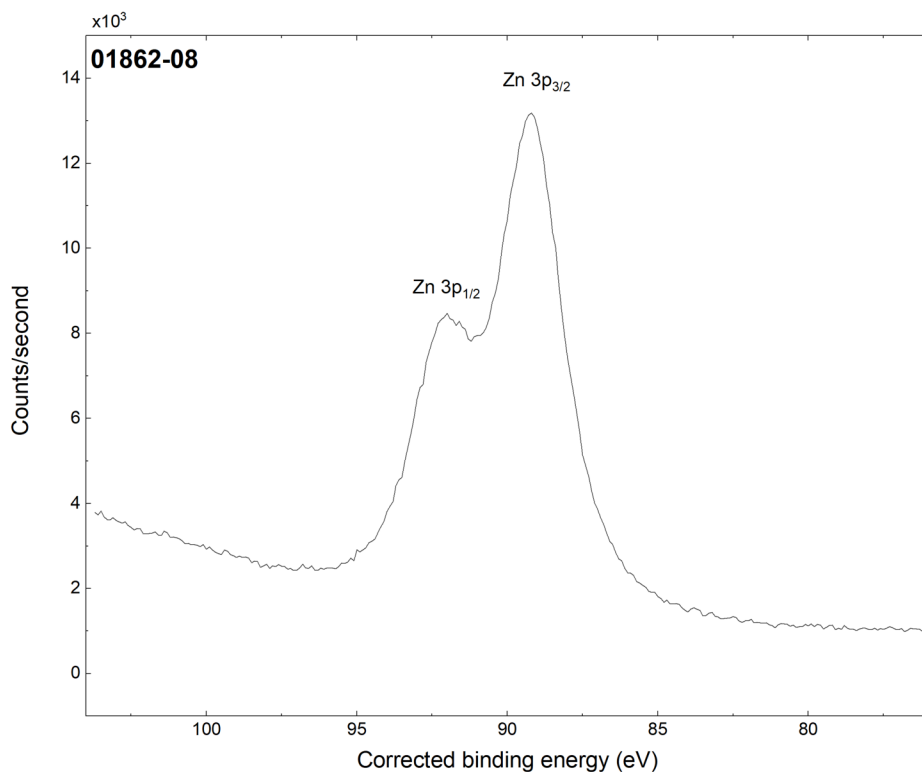
Instrument: Thermo Fisher Scientific
 ESCALAB 250Xi
 Excitation Source: Al K_{α} monochromatic
 Source Energy: 1486.6 eV
 Source Strength: 200 W
 Source Size: $0.9 \times 0.9 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 58°
 Emission Angle: 0°
 Analyzer Pass Energy: 20 eV
 Analyzer Resolution: 0.100 eV
 Total Signal Accumulation Time: 255.6 s
 Total Elapsed Time: 300 s
 Number of Scans: 15
 Effective Detector Width: 3.392 eV



■ **Accession #:** 01862-07
 ■ **Host Material:** ZnS
 ■ **Technique:** XPS
 ■ **Spectral Region:** Zn 3d

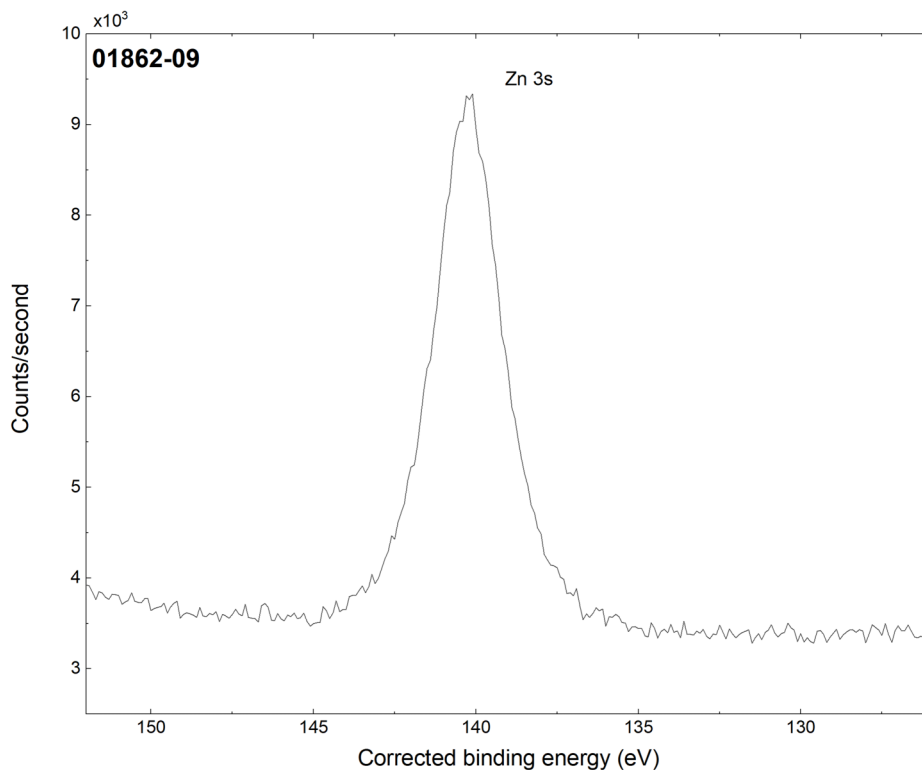
Instrument: Thermo Fisher Scientific
 ESCALAB 250Xi
 Excitation Source: Al K_{α} monochromatic
 Source Energy: 1486.6 eV
 Source Strength: 200 W
 Source Size: $0.9 \times 0.9 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 58°
 Emission Angle: 0°
 Analyzer Pass Energy: 20 eV
 Analyzer Resolution: 0.100 eV
 Total Signal Accumulation Time: 173.2 s
 Total Elapsed Time: 221 s
 Number of Scans: 15
 Effective Detector Width: 3.392 eV

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■ **Accession #:** 01862-08
 ■ **Host Material:** ZnS
 ■ **Technique:** XPS
 ■ **Spectral Region:** Zn 3p

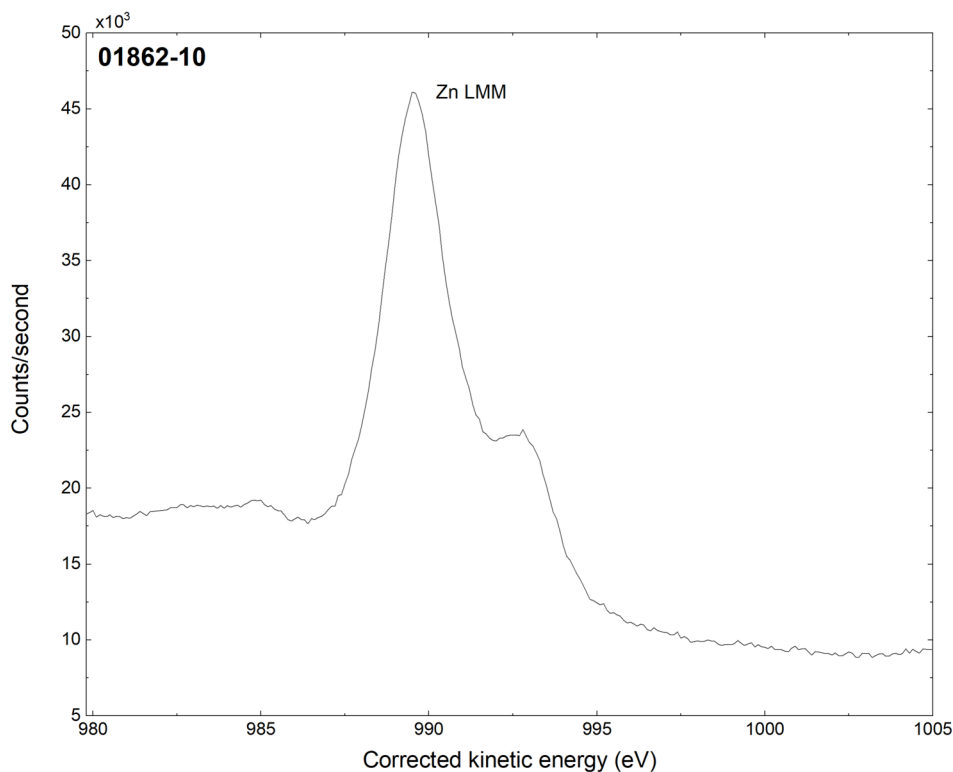
Instrument: Thermo Fisher Scientific
 ESCALAB 250Xi
 Excitation Source: Al K_α monochromatic
 Source Energy: 1486.6 eV
 Source Strength: 200 W
 Source Size: 0.9 × 0.9 mm²
 Analyzer Type: Spherical sector
 Incident Angle: 58°
 Emission Angle: 0°
 Analyzer Pass Energy: 20 eV
 Analyzer Resolution: 0.100 eV
 Total Signal Accumulation Time: 214.4 s
 Total Elapsed Time: 256 s
 Number of Scans: 15
 Effective Detector Width: 3.392 eV
 Comments: See footnotes SPECTRAL
 FEATURES TABLE.



■ **Accession #:** 01862-09
 ■ **Host Material:** ZnS
 ■ **Technique:** XPS
 ■ **Spectral Region:** Zn 3s

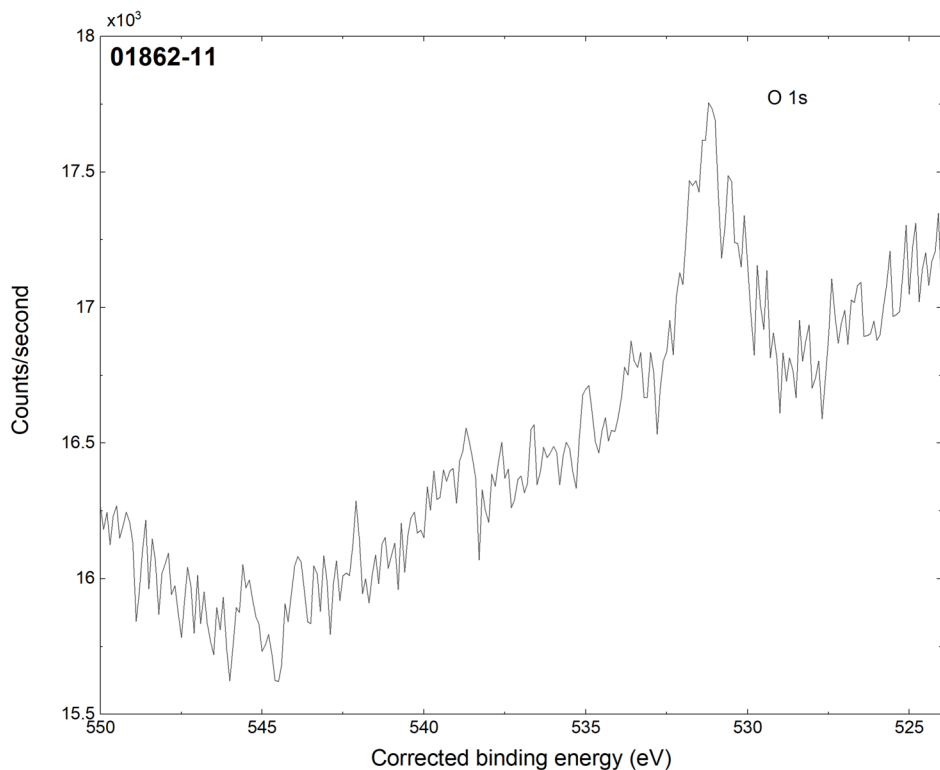
Instrument: Thermo Fisher Scientific
 ESCALAB 250Xi
 Excitation Source: Al K_α monochromatic
 Source Energy: 1486.6 eV
 Source Strength: 200 W
 Source Size: 0.9 × 0.9 mm²
 Analyzer Type: Spherical sector
 Incident Angle: 58°
 Emission Angle: 0°
 Analyzer Pass Energy: 20 eV
 Analyzer Resolution: 0.100 eV
 Total Signal Accumulation Time: 203.2 s
 Total Elapsed Time: 244 s
 Number of Scans: 15
 Effective Detector Width: 3.392 eV

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■ **Accession #:** 01862-10
 ■ **Host Material:** ZnS
 ■ **Technique:** XAES
 ■ **Spectral Region:** Zn LMM

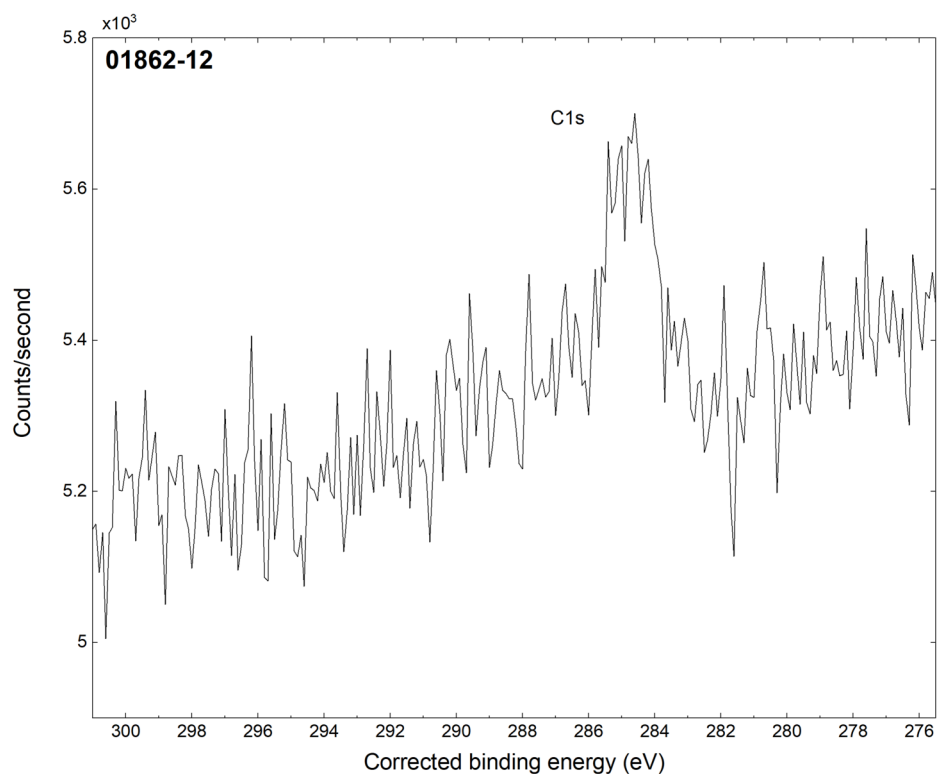
Instrument: Thermo Fisher Scientific
 ESCALAB 250Xi
 Excitation Source: Al K_{α} monochromatic
 Source Energy: 1486.6 eV
 Source Strength: 200 W
 Source Size: $0.9 \times 0.9 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 58°
 Emission Angle: 0°
 Analyzer Pass Energy: 20 eV
 Analyzer Resolution: 0.100 eV
 Total Signal Accumulation Time: 203.2 s
 Total Elapsed Time: 245 s
 Number of Scans: 15
 Effective Detector Width: 3.392 eV
 Comments: See footnotes SPECTRAL
 FEATURES TABLE



■ **Accession #:** 01862-11
 ■ **Host Material:** ZnS
 ■ **Technique:** XPS
 ■ **Spectral Region:** O 1s

Instrument: Thermo Fisher Scientific
 ESCALAB 250Xi
 Excitation Source: Al K_{α} monochromatic
 Source Energy: 1486.6 eV
 Source Strength: 200 W
 Source Size: $0.9 \times 0.9 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 58°
 Emission Angle: 0°
 Analyzer Pass Energy: 20 eV
 Analyzer Resolution: 0.100 eV
 Total Signal Accumulation Time: 225.7 s
 Total Elapsed Time: 268 s
 Number of Scans: 15
 Effective Detector Width: 3.392 eV
 Comments: See footnotes SPECTRAL
 FEATURES TABLE

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- **Accession #:** 01862-12
- **Host Material:** ZnS
- **Technique:** XPS
- **Spectral Region:** C 1s

Instrument: Thermo Fisher Scientific
 ESCALAB 250Xi
 Excitation Source: Al K_{α} monochromatic
 Source Energy: 1486.6 eV
 Source Strength: 200 W
 Source Size: $0.9 \times 0.9 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 58°
 Emission Angle: 0°
 Analyzer Pass Energy: 20 eV
 Analyzer Resolution: 0.100 eV
 Total Signal Accumulation Time: 218.2 s
 Total Elapsed Time: 259 s
 Number of Scans: 15
 Effective Detector Width: 3.392 eV
 Comments: See footnotes SPECTRAL
 FEATURES TABLE

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