SURFACE SCIENCE SPECTRA

Calcium sulfide powder analyzed by XPS

Cite as: Surf. Sci. Spectra **30**, 014005 (2023); https://doi.org/10.1116/6.0002304
Submitted: 20 October 2022 • Accepted: 11 January 2023 • Published Online: 10 February 2023

Brian Butkus, Matthew Havel, Alexandros Kostogiannes, et al.

View Online Export Citation CrossMark

ARTICLES YOU MAY BE INTERESTED IN

Lanthanum sulfide powder analyzed by XPS
Surface Science Spectra **30**, 014001 (2023); https://doi.org/10.1116/6.0002252

1,3-Dimethyl-imidazolium dimethyl phosphate ($[MMIM]^{+}[DMP]^{-}$) analyzed by XPS and HAXPES
Surface Science Spectra **30**, 014006 (2023); https://doi.org/10.1116/6.0002297

Surface Y_2O_3 layer formed on air exposed Y powder characterized by XPS
Surface Science Spectra **27**, 024010 (2020); https://doi.org/10.1116/6.0000475

© 2023 Author(s).

Calcium sulfide powder analyzed by XPS

Cite as: Surface Science Spectra **30**, 014005 (2023); doi: 10.1116/6.0002304
Submitted: 20 October 2022 · Accepted: 11 January 2023 ·
Published Online: 10 February 2023

View Online | Export Citation | CrossMark

Brian Butkus,[1] Matthew Havel,[1,2] Alexandros Kostogiannes,[1,2] Andrew Howe,[2] Myungkoo Kang,[2] Romain Gaume,[2,3] Kathleen A. Richardson,[1,2] and Parag Banerjee[1,3,4,5]

AFFILIATIONS

[1]Department of Materials Science and Engineering, University of Central Florida, Orlando, Florida 32816
[2]College of Optics and Photonics, University of Central Florida, Florida 32816
[3]NanoScience Technology Center, University of Central Florida, Florida 32816
[4]Florida Solar Energy Center, University of Central Florida, Florida 32816
[5]REACT Faculty Cluster, University of Central Florida, Florida 32816

ABSTRACT

X-ray photoelectron spectroscopy was performed on as-received, calcium sulfide (CaS) powder (98.0%), doped with europium (Eu) 2.0 wt. %. The scans provide photoelectron spectroscopy investigation data for CaS to help with identification of sulfide compounds. This report includes charge corrected scans for the survey along with S *2s*, S *2p*, O *1s*, Eu *3d*, Ca *2s*, Ca *2p*, and C *1s* surface photoelectron signals.

Key words: CaS, sulfides, phosphor, x-ray photoelectron spectroscopy, XPS

Published under an exclusive license by the AVS. https://doi.org/10.1116/6.0002304

Accession #: 01818	**Major Elements in Spectra:** Ca, S
Technique: XPS	**Minor Elements in Spectra:** O, C, Eu
Host Material: CaS, powder doped with Eu	**Published Spectra:** 8
Instrument: XPS ESCALAB 250Xi Fisher Scientific	**Spectral Category:** Comparison

INTRODUCTION

Calcium sulfide (CaS) has historically been used as a phosphor. CaS doped with europium (Eu) is a common red phosphor with an emission at 645 nm (Ref. 1). Along with the enhanced color emission, the doping increases the thermal stability of the material. Other historical uses include the production of pharmaceutical sulfur-based drugs. But new interest has broadened the uses and research in electronics, optical glasses, and ceramics, along with use in catalysis (Ref. 2). Examples of applications in each of the areas include nanocrystal for light-emitting diode production, broadband glasses, and ceramic application (i.e., UV and IR glass and ceramics) (Refs. 1–3). Catalysis research includes using CaS for the removal of heavy metals from groundwater (Ref. 4) and the conversion of drywall (gypsum board) to sulfur (Ref. 5) While there are some reports that include x-ray photoelectron spectroscopy (XPS) of CaS (Refs. 6 and 7), the data included in the reports are not complete, i.e., missing survey spectra data on collection and identification of peaks in fine spectra.

In this study, we analyze CaS doped with Eu by XPS to study and quantify the bonding states of Ca and S, along with minor species Eu, C, and O. Eu has been introduced at 2.0 wt. % during the synthesis of the compound. The peak fitting done in this report aims to identify bonding states of CaS. The use of XPS also provides quantification of atomic percentages (i.e., stoichiometry) along with the bonding states of additional constituent elements and non-trace level impurities, if present.

SPECIMEN DESCRIPTION (ACCESSION # 01818)

Host Material: Calcium sulfide CaS bulk powder
CAS Registry #: 20548-54-3
Host Material Characteristics: Homogeneous; powder; polycrystalline; semiconductor; inorganic compound; powder
Chemical Name: Calcium sulfide 98.0 wt. % doped with europium 2.0 wt. %
Source: Lorad Chemical Corporation, 1200 19th Street North, St. Petersburg, FL 33713, USA

Published under an exclusive license by the AVS

Host Composition: CaS 98.0 wt. % and Eu 2.0 wt. %
Form: 98.0% purity powder CaS
Structure: Halite (cubic), cF8
History and Significance: The specimen was received in a vacuum sealed glass bottle and stored in an inert atmosphere chamber at room temperature. CaS is commonly used in Electronics, Optical glasses and ceramics, catalysts, pharmaceutical application.
As Received Condition: Light red powder
Analyzed Region: Same as host material
***Ex Situ* Preparation/Mounting:** Powder mounted on double-sided carbon tape.
***In Situ* Preparation:** Argon ion sputtering 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 μA) and a dual-beam low energy electron and ion coaxial flood source (2 eV, 100 μA).
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 [peak area/(PE × XSF)] versus log[KE/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_a monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Beam Size: $200 \times 200\ \mu m^2$
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° × 22.5°

■ Ion Gun

Manufacturer and Model: Thermo Fisher Scientific EX03 Ion Gun System
Energy: 3000 eV
Current: 0.02 mA
Current Measurement Method: Biased stage
Sputtering Species: Ar^+
Spot Size (unrastered): 500 μm
Raster Size: $4500 \times 4500\ \mu m^2$
Incident Angle: 40°
Polar Angle: 40°
Azimuthal Angle: 270°
Comment: These parameters correspond to ion cleaning methods used in typical operation requiring surface cleaning.

DATA ANALYSIS METHOD

Energy Scale Correction: Binding energy scale was referenced to C *1s* = 284.8 eV.
Recommended Energy Scale Shift: Shift +0.58 eV
Peak Shape and Background Method: Thermo Scientific Avantage software version 5.9902 was used for peak fitting and background subtraction. The smart (Shirley function) was used to subtract the background for Ca *2s*, Ca *2p*, S *2p*, S *2s*, O *1s*, C *1s*, and Eu *3d* peaks. Using the smart feature utilizes constraints that limit the background from having greater intensity than data from points in the collection region. After background subtraction, a mixed Gaussian (30%)-Lorentzian (70%) product function was used to fit the peaks after background subtraction.
Quantitation Method: Atomic percentages were calculated using Thermo Scientific Avantage software version 5.9902. Sensitivity factors were obtained from the Thermo Scientific Avantage software database and used to calculate elemental atomic percentages. The peak library is ALWAG (Ref. 8).

ACKNOWLEDGMENTS

The authors thank the Lockheed Martin University Engagement and Applied Research organizations, the U.S. Naval Surface Warfare Center, the U.S. Army Research Laboratory, and the U.S. Air Force Research Laboratory for their integral and collaborative support of this research. The authors acknowledge the NSF MRI: ECCS: 1726636 and the MCF-AMPAC facility, MSE and CECS along with Kirk Scammon MCF Research Engineer and Lorad Chemical Corporation. This research was supported, in part, by the Florida High Tech Corridor's Matching Grant Research Program at the University of Central Florida.

AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Published under an exclusive license by the AVS

Author Contributions

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

DATA AVAILABILITY

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

REFERENCES

[1] N. Ruelle, M. Pham-Thi, and C. Fouassier, Jpn. J. Appl. Phys. **31**, 2786 (1992).
[2] E. Shobhana, *Optical Characterization of Calcium Sulphide (CaS) Thin Films by Chemical Bath Deposition*, 2015.
[3] H. Nakamura, Y. Ogawa, A. Kasahara, and T. Kodama, J. Jpn. Inst. Met. **62**, 1137 (1998).
[4] C.-Y. Huang *et al.*, Water **13**, 2266 (2021).
[5] M. de Beer *et al.*, Waste Manage. **34**, 2373 (2014).
[6] S. Bahadur, D. Gong, and J. Anderegg, Wear **197**, 271 (1996).
[7] R. Vercaemst *et al.*, J. Lumin. **63**, 19 (1995).
[8] C. D. Wagner *et al.*, Surf. Interface Anal. **3**, 211 (1981).

Published under an exclusive license by the AVS

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
01818-02	S *2s*	224.54	2.63	13 864.86	1.294	…	CaS
01818-03	S $2p_{3/2}$	160.78	2.45	21 373.11	1.881	32.7	CaS
01818-04	O *1s*	530.89	2.81	15 477.98	2.881	16.96	Metal oxide/ carbonates
01818-05	Eu $3d_{5/2}$	1133.83	3.56	12 820.81	48.796	1.18	Eu
01818-06	Ca *2s*	437.46	3.3	17 085.82	2.106	…	CaS, CaO,$CaCO_3$
01818-07	Ca $2p_{3/2}$	345.78	1.52	57 501.87	5.97	28.86	CaS, CaO,$CaCO_3$
01818-08	C *1s*	284.64	2.22	6 873.88	1	20.3	C–C, C–H, carbonates

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

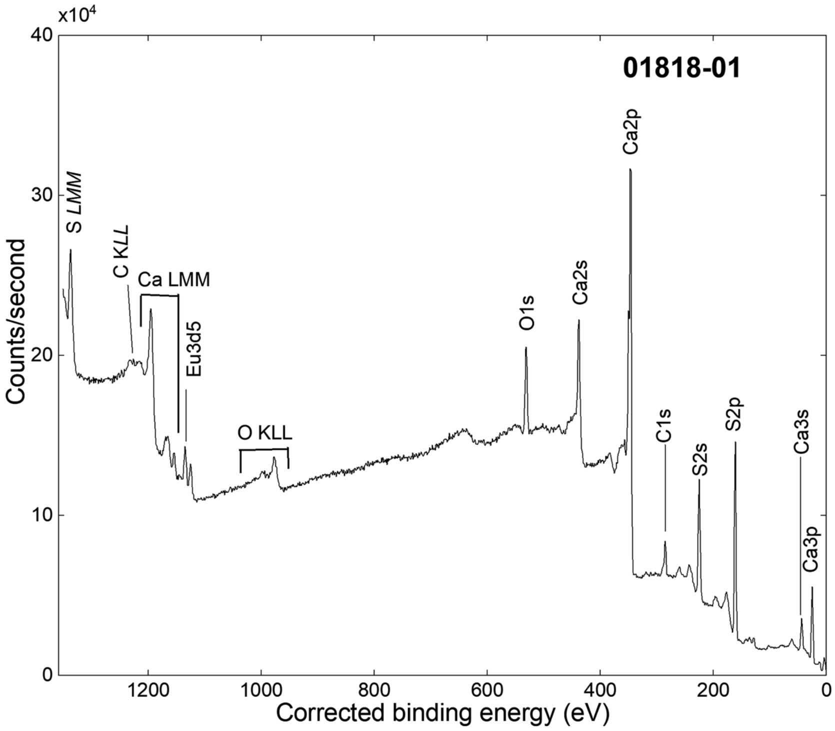
GUIDE TO FIGURES

Spectrum (Accession) #	Spectral Region	Voltage Shift[a]	Multiplier	Baseline	Comment #
01818-01	Survey	−0.58	1	0	1
01818-02	S *2s*	−0.58	1	0	1
01818-03	S *2p*	−0.58	1	0	1
01818-04	O *1s*	−0.58	1	0	1
01818-05	Eu *3d*	−0.58	1	0	1
01818-06	Ca *2s*	−0.58	1	0	1
01818-07	Ca *2p*	−0.58	1	0	1
01818-08	C *1s*	−0.58	1	0	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.

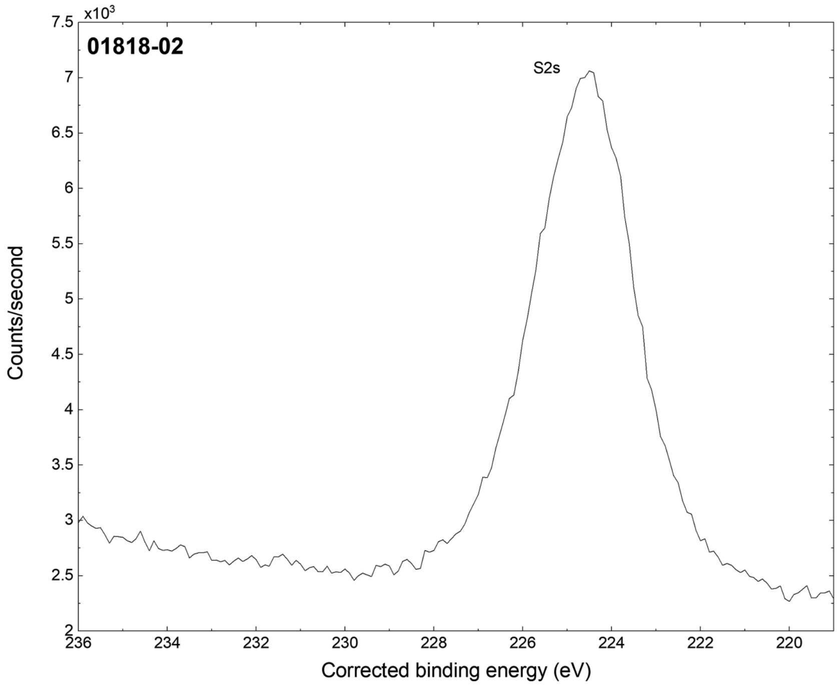
1. CaS powder.

Published under an exclusive license by the AVS



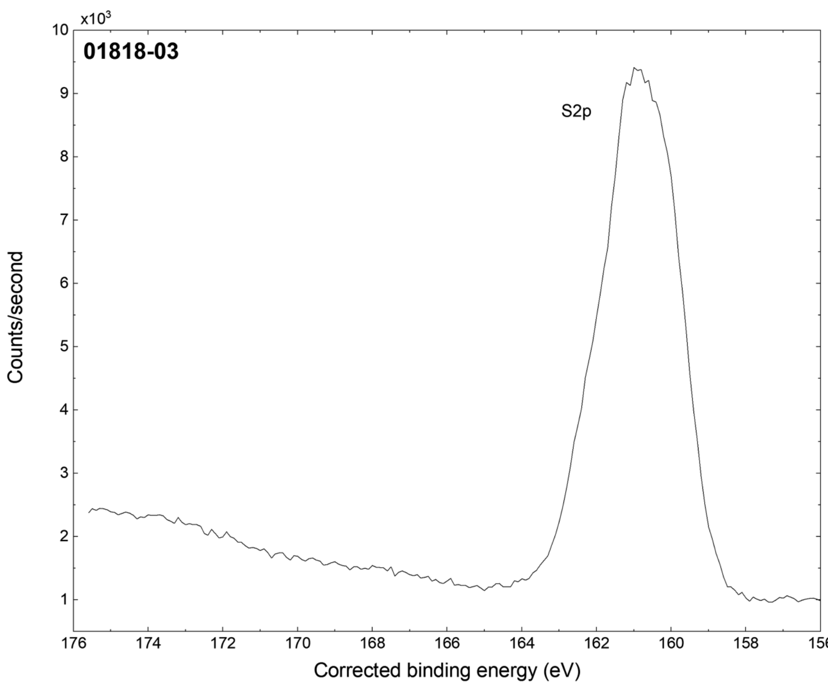
Accession #:	01818-01
■ **Host Material:**	Calcium Sulfide CaS Bulk Powder
■ **Technique:**	XPS
■ **Spectral Region:**	Survey
Instrument:	Thermo Fisher Scientific ESCALAB 250Xi
Excitation Source:	Al K_a monochromatic
Source Energy:	1486.6 eV
Source Strength:	200 W
Source Size:	0.9 × 0.9 mm^2
Analyzer 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:	222 s
Number of Scans:	5
Effective Detector Width:	1.00 eV
Comments:	Number of energy steps 1361

Published under an exclusive license by the AVS



- **Accession #:** 01818-02
- **Host Material:** Calcium Sulfide CaS Bulk Powder
- **Technique:** XPS
- **Spectral Region:** S *2s*

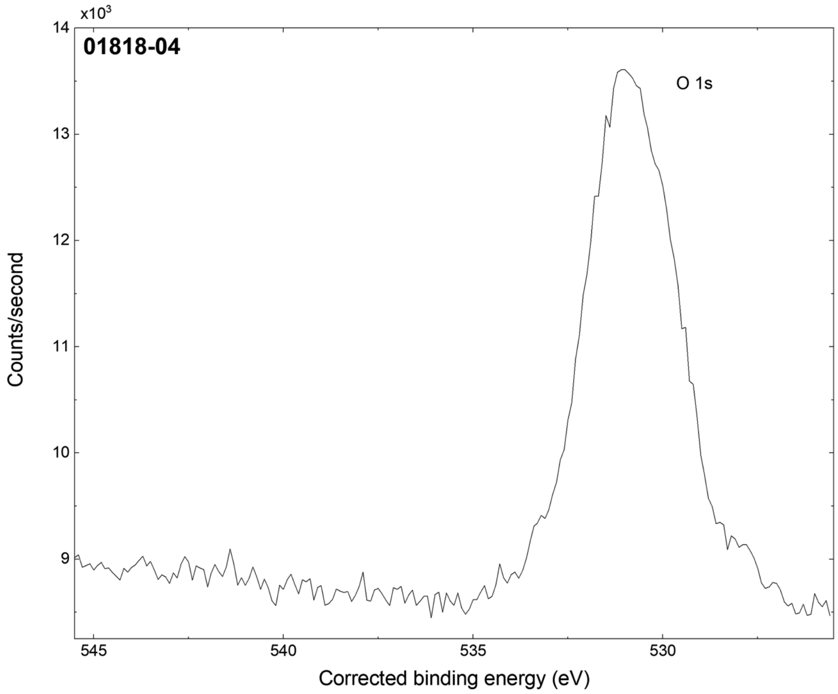
Instrument: Thermo Fisher Scientific ESCALAB 250Xi
Excitation Source: Al K_a monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.9 × 0.9 mm^2
Analyzer Type: Spherical sector
Incident Angle: 58°
Emission Angle: 0°
Analyzer Pass Energy: 20 eV
Analyzer Resolution: 0.100 eV
Total Signal Accumulation Time: 180.9 s
Total Elapsed Time: 230 s
Number of Scans: 20
Effective Detector Width: 0.100 eV



- **Accession #:** 01818-03
- **Technique:** XPS
- **Spectral Region:** S *2p*

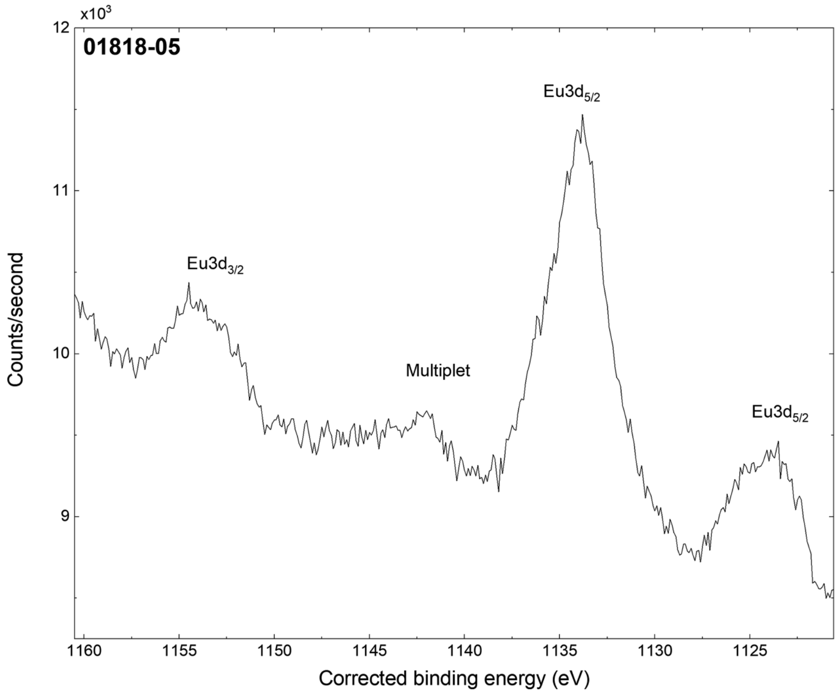
Instrument: Thermo Fisher Scientific ESCALAB 250Xi
Excitation Source: Al K_a monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.9 × 0.9 mm^2
Analyzer Type: Spherical sector
Incident Angle: 58°
Emission Angle: 0°
Analyzer Pass Energy: 20 eV
Analyzer Resolution: 0.100 eV
Total Signal Accumulation Time: 200.9 s
Total Elapsed Time: 259 s
Number of Scans: 20
Effective Detector Width: 0.100 eV

Published under an exclusive license by the AVS



- **Accession #:** 01818-04
- **Host Material:** Calcium Sulfide CaS Bulk Powder
- **Technique:** XPS
- **Spectral Region:** O *1s*

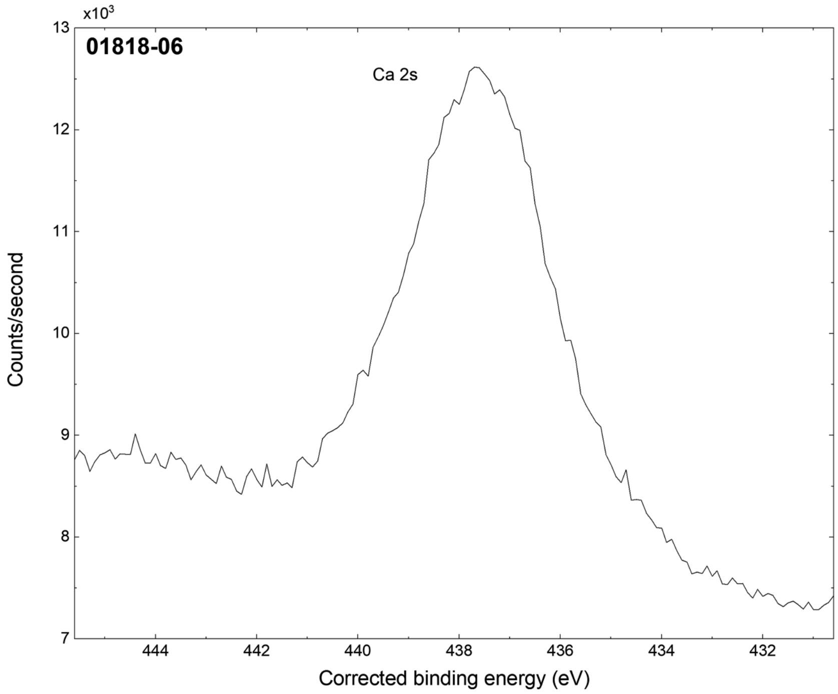
Instrument: Thermo Fisher Scientific ESCALAB 250Xi
Excitation Source: Al K_a monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.9 × 0.9 mm^2
Analyzer Type: Spherical sector
Incident Angle: 58°
Emission Angle: 0°
Analyzer Pass Energy: 20 eV
Analyzer Resolution: 0.100 eV
Total Signal Accumulation Time: 200.9 s
Total Elapsed Time: 251 s
Number of Scans: 20
Effective Detector Width: 0.100 eV



- **Accession #:** 01818-05
- **Host Material:** Calcium Sulfide CaS Bulk Powder
- **Technique:** XPS
- **Spectral Region:** Eu *3d*

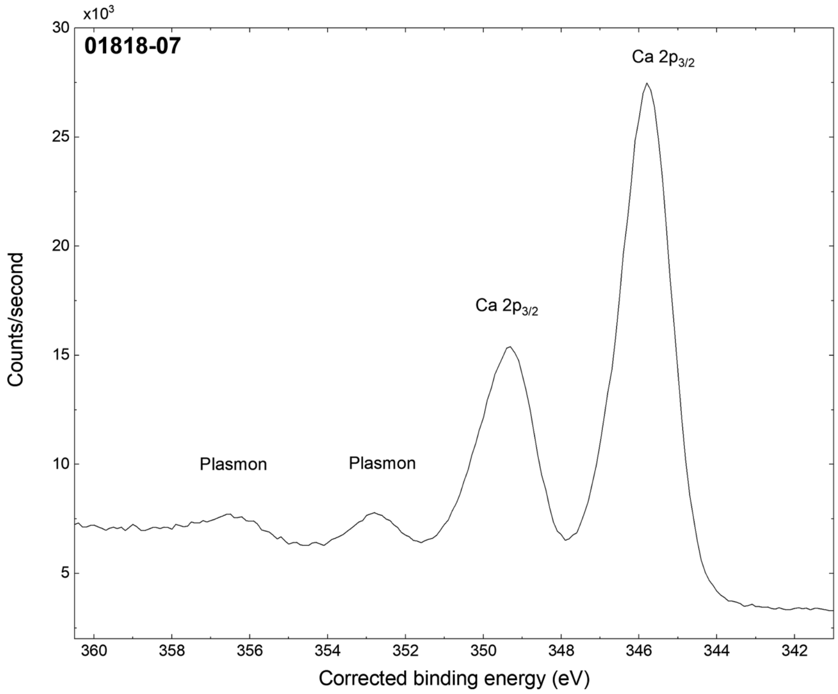
Instrument: Thermo Fisher Scientific ESCALAB 250Xi
Excitation Source: Al K_a monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.9 × 0.9 mm^2
Analyzer Type: Spherical sector
Incident Angle: 58°
Emission Angle: 0°
Analyzer Pass Energy: 20 eV
Analyzer Resolution: 0.100 eV
Total Signal Accumulation Time: 10 021 s
Total Elapsed Time: 1163 s
Number of Scans: 50
Effective Detector Width: 0.100 eV

Published under an exclusive license by the AVS



■ **Accession #:** 01818-06
■ **Host Material:** Calcium Sulfide CaS Bulk Powder
■ **Technique:** XPS
■
■ **Spectral Region:** Ca *2s*

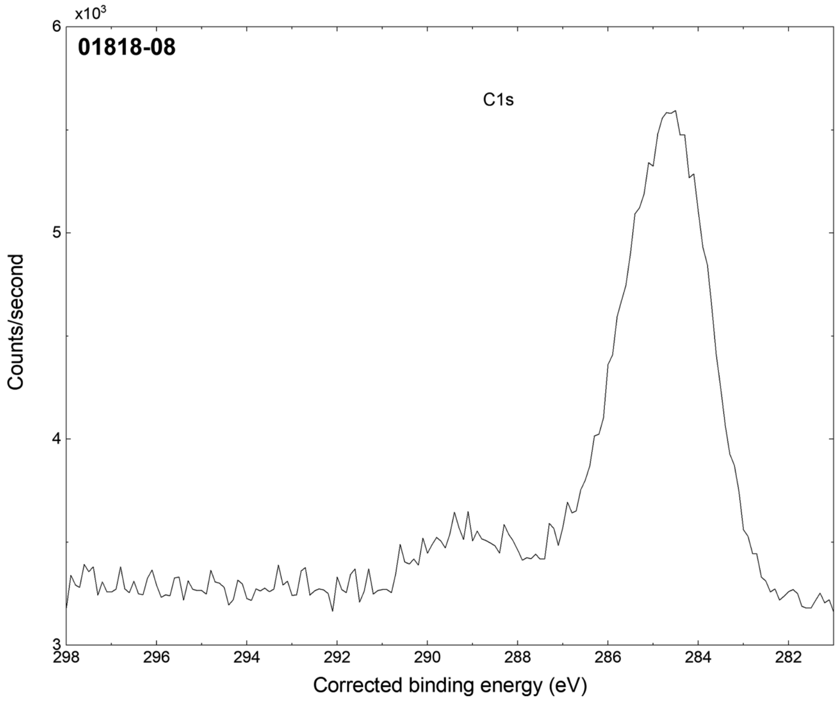
Instrument: Thermo Fisher Scientific ESCALAB 250Xi
Excitation Source: Al K_a monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.9 × 0.9 mm^2
Analyzer Type: Spherical sector
Incident Angle: 58°
Emission Angle: 0°
Analyzer Pass Energy: 20 eV
Analyzer Resolution: 0.100 eV
Total Signal Accumulation Time: 150.9 s
Total Elapsed Time: 199 s
Number of Scans: 20
Effective Detector Width: 0.100 eV



■ **Accession #:** 01818-07
■ **Host Material:** Calcium Sulfide CaS Bulk Powder
■ **Technique:** XPS
■
■ **Spectral Region:** Ca *2p*

Instrument: Thermo Fisher Scientific ESCALAB 250Xi
Excitation Source: Al K_a monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.9 × 0.9 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: 200.9 s
Total Elapsed Time: 251 s
Number of Scans: 20
Effective Detector Width: 0.100 eV

Published under an exclusive license by the AVS



- **Accession #:** 01818-08
- **Host Material:** Calcium Sulfide CaS Bulk Powder
- **Technique:** XPS
-
- **Spectral Region:** C *1s*

Instrument: Thermo Fisher Scientific ESCALAB 250Xi
Excitation Source: Al K_a monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.9 × 0.9 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: 190.9 s
Total Elapsed Time: 241 s
Number of Scans: 20
Effective Detector Width: 0.100 eV

Published under an exclusive license by the AVS