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Lanthanum Sulfide powder Analyzed by XPS

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X-ray photoelectron spectroscopy was performed on as-received, gamma lanthanum sulfide (γ-La_2S_3) powder (99.9%), doped with sodium sulfide (Na_2S) 0.1% wt%. The scans provide photoelectron spectroscopy investigation data for cubic La_2S_3 to help with identification of lanthanum compounds. This report includes charge corrected scans for the survey along with S *2s*, S *2p*, La *4s*, La *4p*, La *4d*, La $3p_{3/2}$, La $3p_{1/2}$, La *3d*, Na *1s*, O *1s*, and C *1s* surface photoelectron signals.

Keywords: La_2S_3; Lanthanides; transition metal dichalcogenide; x-ray photoelectron spectroscopy; XPS

Accession#: 01808

Technique: XPS

Host Material: γ-La_2S_3, powder doped with Na_2S

Instrument: XPS ESCALAB 250Xi Fisher Scientific

Major Elements in Spectra: La, S

Minor Elements in Spectra: O, C, Na

Published Spectra: 12

Spectral Category: Comparison

INTRODUCTION

Lanthanum sulfide (La_2S_3) is a commodity chemical for production of arsenic-free, chalcogenide glasses and ceramics[1-4] that transmit infrared radiation. For example, La_2S_3 is a major precursor in the production of gallium lanthanum sulfide and strontium lanthanum sulfide, both of which are chalcogenides.[4-7] La_2S_3-based glasses have unique characteristics such as, high refractive index (2.41n ~ 0.01),[1, 8, 9] and an optical bandgap of ~2.6 eV. [5, 8, 10] In the glassy phase, the high content of lanthanum results in a high dispersion of ions in the matrix giving greater ionic conductivity ($1.01x10^{-3}$ S/cm).[6, 11] Optically, these glasses have high transmission from the visible wavelength (380-750 nm) to infrared (~ 8 μm).[1, 4, 12] However, there are no reports on the x-ray photoelectron spectroscopy (XPS) of La_2S_3.

In this study, we analyze γ-La_2S_3 by XPS to study and quantify the bonding states of observable electron shells for La and S, along with minor species Na, C, and O. Sodium sulfide has been introduced at a 0.1 wt% level during the synthesis of the compound to stabilize the cubic phase of La_2S_3 at room temperature. [13]. 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. From the lanthanum fine spectra La *3d* along with the O *1s* spectra we see that the material is not a pure metal sulfide but a mixture of Lanthanum Oxide and Lanthanum Sulfide. The shake-up peaks in the La *3d* along with the O *1s* metal oxide peak resemble the feature from M. F. Sunding et al.[14] study on lanthanum oxide. There are some notable differences to peak height and shape but do give insights to the material not being pure γ-La_2S_3. While looking at the Sulfur fine spectra there is no evidence of oxidized sulfur that would be contributing to the O *1s* peak.

SPECIMEN DESCRIPTION (ACCESSION # 01808)

Host Material: Lanthanum Sulfide γ-La_2S_3 Bulk Powder

CAS Registry #: 12031-49-1

Host Material Characteristics: homogeneous; powder; polycrystalline; semiconductor; inorganic compound; Powder

Chemical Name: Lanthanum Sulfide 99.9% wt% doped with Sodium Sulfide 0.1% wt%

Source: Lorad Chemical Corporation 1200 19th Street North St. Petersburg, FL 33713 USA

Host Composition: La_2S_3 99.9% wt% & Na_2S 0.1% wt%

Form: 99.9% purity powder gamma phase La_2S_3

Structure: Modified Th_3P_4

History & Significance: The specimen was received in a vacuum sealed glass bottle and stored in an inert atmosphere chamber at room temperature. La_2S_3 is commonly used in glasses, ceramics, and optical fibers.

As Received Condition: Light yellow powder

Analyzed Region: Same as host material

Ex Situ Preparation/Mounting: Powder mounted on double-sides carbon tape.

In Situ Preparation: 120 second 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,100μA) and a dual-beam low energy electron and ion coaxial flood source (2 eV, 100μA).

Temp. During Analysis: 300K

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Pressure During Analysis: 5 x 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 x 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 μm x 200 μm

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 ° x 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 μm x 4500 μm

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 reference to O *1s* = 531 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 La *3d*, S *2p*, O *1s*, C *1s*, Na *1s* peaks. Using the smart feature, utilizes 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. Along with atomic percentages Thermo scientific Avantage software sensitivity factors. The peak library is ALWAG [15]

ACKNOWLEDGMENTS

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AUTHOR DECLARATION

Conflict of Interest: The authors have no conflicts to disclose.

DATA AVAILABILITY STATEMENT

The data that supports 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 x cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01808-02	S *2s*	224.62	2.78	8974.73	1.294	…	La_2S_3
01808-03	S *2p*	161	2.58	12818.31	1.881	39.44	La_2S_3
01808-04[a]	O *1s*	528.14	1.33	13393.67	2.881	29.49	La_2O_3/ Carbonates / Hydroxyls
01808-05	Na *1s*	1071.23	1.85	4161.84	10.588	3.33	Na
01808-06	La *4s*	274.09	6.3	4010.16	1.19	...	La_2S_3 / La_2O_3
01808-07[a]	La *4p*	195.29	5.5	19642.48	4.33	…	La_2S_3 / La_2O_3
01808-08[a]	La *4d*	104.27	5.95	51987.48	10.733	27.74	La_2S_3 / La_2O_3
01808-09[a]	La $3p_{3/2}$	1127.2	7.53	46502.55	14	…	La_2S_3 / La_2O_3
01808-10[a]	La $3p_{1/2}$	1206.72	7.26	18993.02	6.1	…	La_2S_3 / La_2O_3
01808-11	La $3d_{5/2}$	837.11	2.28	196155.2	32.944	…	La_2S_3 / La_2O_3
01808-12[b]	C *1s*	284.43	1	838.92	1	…	C-C/ Carbonates

[a] Peak assignment for La_2O_3 sourced from M. F. Sunding et al.[14]

[b] Quantification of Carbon not included due to low confidence as high noise to signal ratio on fine spectra

ANALYZER CALIBRATION TABLE

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV x cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
…	Au *4f*	84.05	0.57	262830.05	20.735	…	Au
…	Ag *3d*	368.36	0.48	386600.57	22.131	…	Ag
…	Cu *2p*	932.8	0.77	655133.11	26.513	…	Cu

GUIDE TO FIGURES

Spectrum (Accession) #	Spectral Region	Voltage Shift*	Multiplier	Baseline	Comment #
01808-01	Survey	-0.58	1	0	1
01808-02	S *2s*	-0.58	1	0	1
01808-03	S *2p*	-0.58	1	0	1
01808-04	O *1s*	-0.58	1	0	1
01808-05	Na *1s*	-0.58	1	0	1
01808-06	La *4s*	-0.58	1	0	1
01808-07	La *4p*	-0.58	1	0	1
01808-08	La *4d*	-0.58	1	0	1
01808-09	La $3p_{3/2}$	-0.58	1	0	1
01808-10	La $3p_{1/2}$	-0.58	1	0	1
01808-11	La *3d*	-0.58	1	0	1
01808-12	C *1s*	-0.58	1	0	1

*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. La_2S_3 powder

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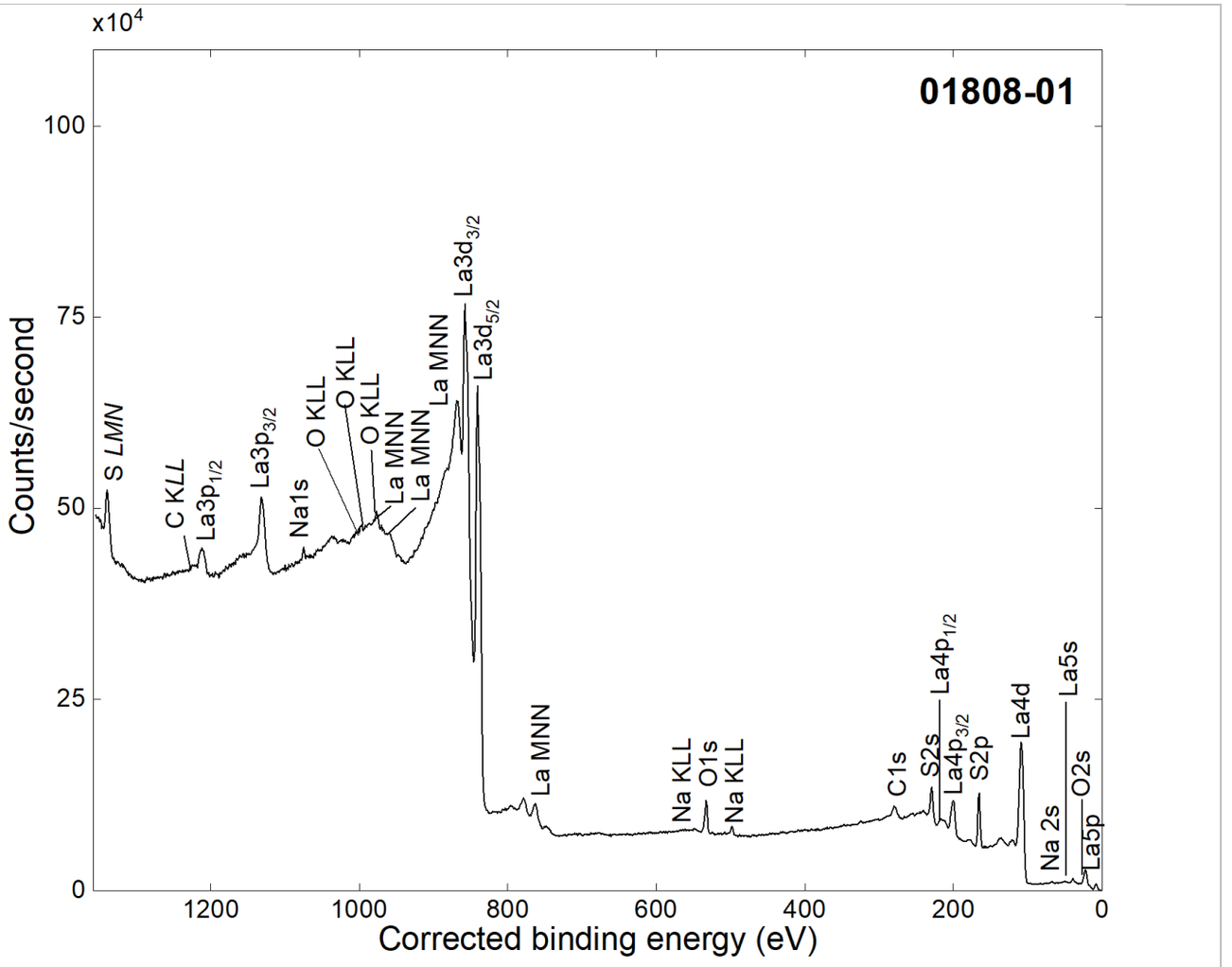

Accession #	01808-01
■ Host Material	La_2S_3
■ Technique	XPS
■ Spectral Region	survey
Instrument	Thermo Fisher Scientific ESCALAB 250Xi
Excitation Source	Al K_a monochromatic
Source Energy	1486.6 eV
Source Strength	200 W
Source Size	0.9 mm x 0.9 mm
Analyzer Type	spherical sector analyzer
Incident Angle	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

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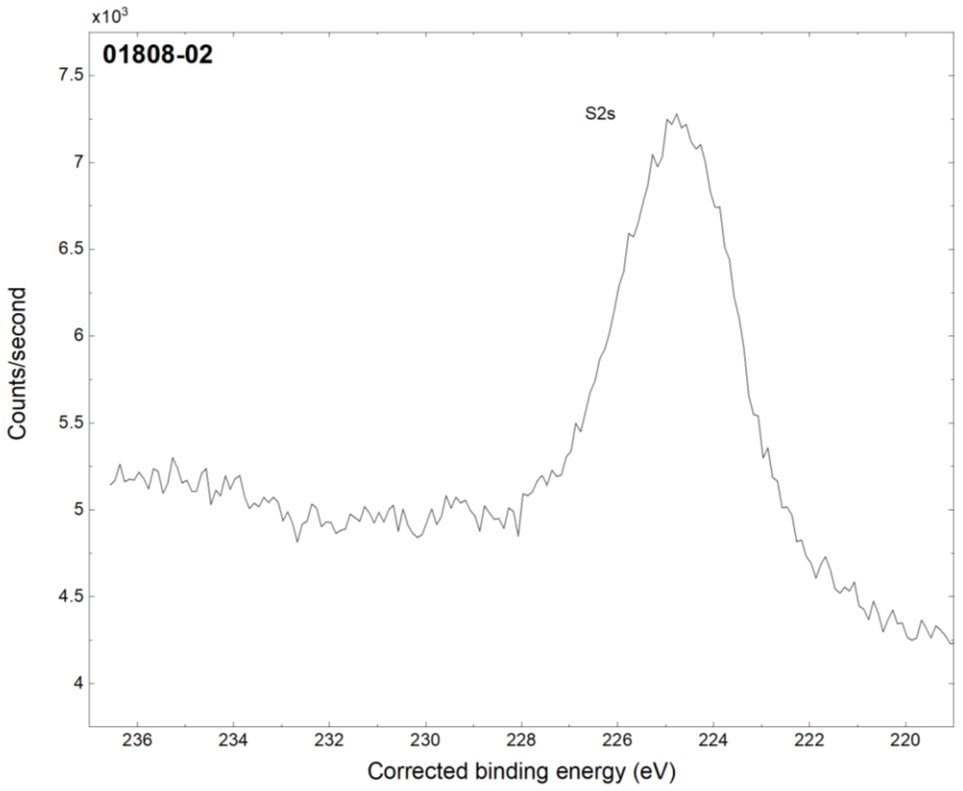
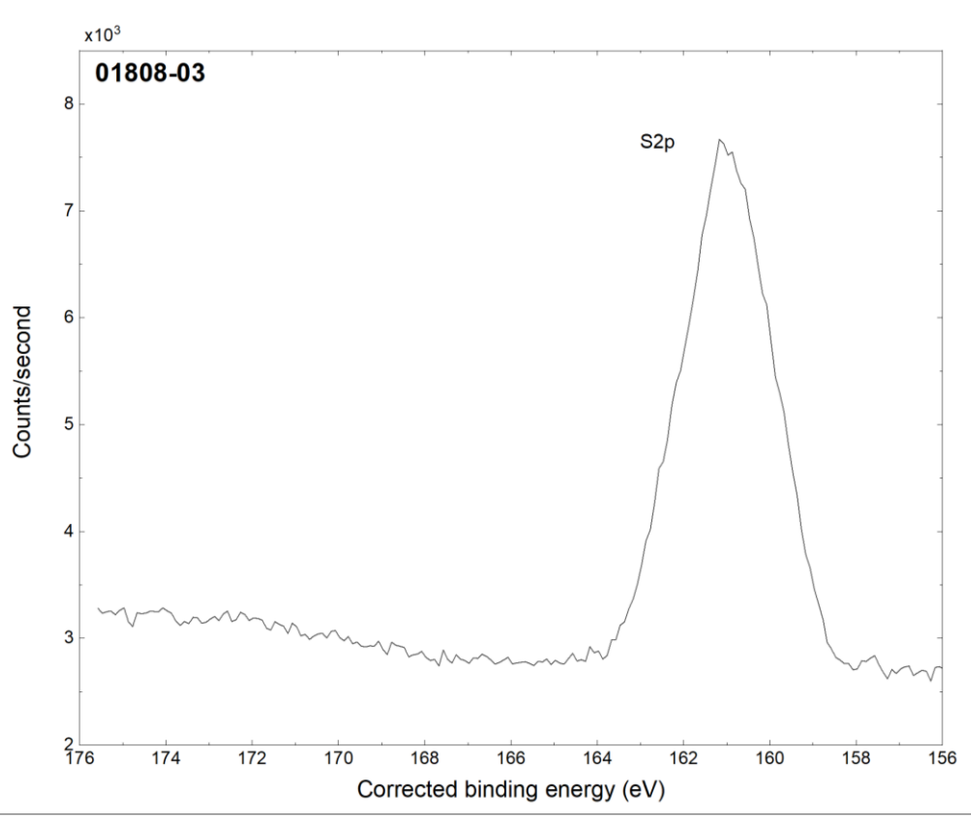


- **Accession #:** 01808-02
- **Host Material:** Lanthanum Sulfide La_2S_3 Bulk Powder
- **Technique:** XPS
- **Spectral Region:** S *2s*

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 mm x 0.9 mm
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 #:** 01808-03.
- **Host Material:** Lanthanum Sulfide La_2S_3 Bulk Powder
- **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 mm x 0.9 mm
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

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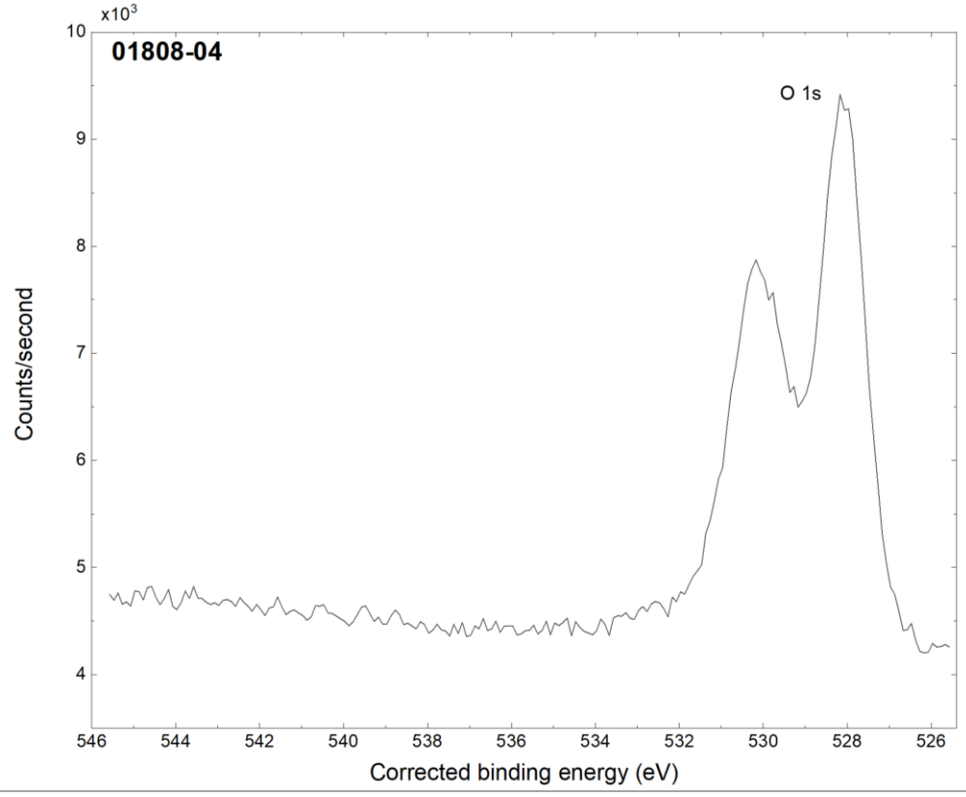


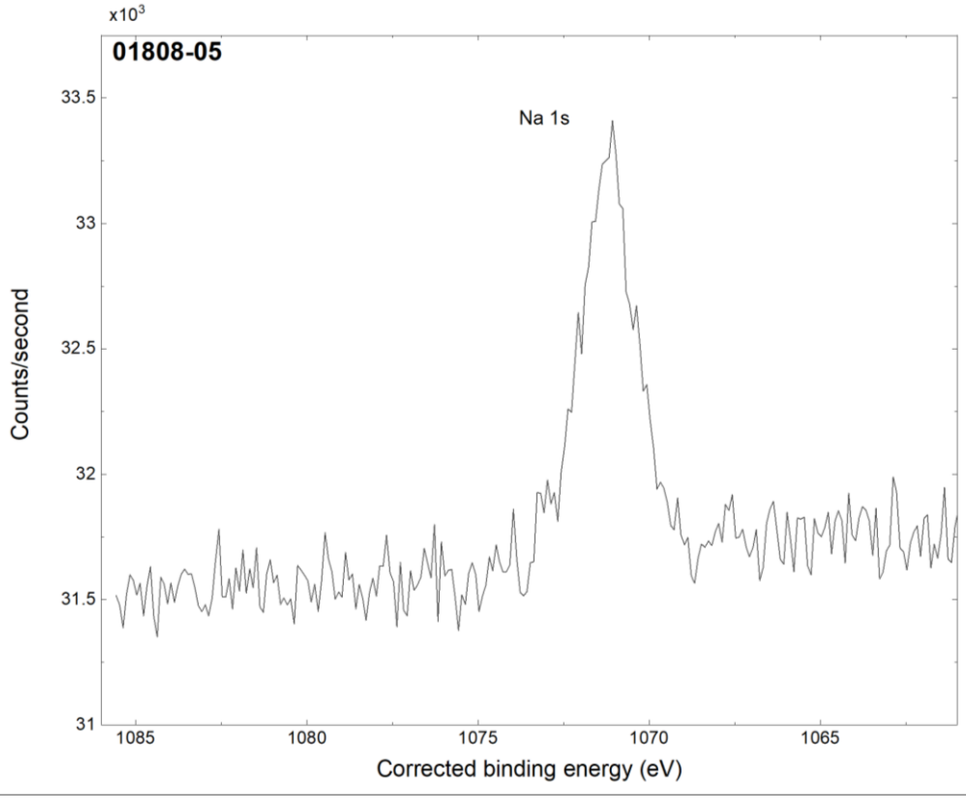
■ **Accession #:** 01808-04

■ **Host Material:** Lanthanum Sulfide La_2S_3 Bulk Powder

■ **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 mm x 0.9 mm
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: 253 s
Number of Scans: 20
Effective Detector Width: 0.100 eV



■ **Accession #:** 01808-05

■ **Host Material:** Lanthanum Sulfide La_2S_3 Bulk Powder

■ **Technique:** XPS

■ **Spectral Region:** Na *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 mm x 0.9 mm
Analyzer Type: spherical sector
Incident Angle: 58 ˚
Emission Angle: 0 ˚
Analyzer Pass Energy 20 eV
Analyzer Resolution: 0.100 eV
Total Signal Accumulation Time: 627.2 s
Total Elapsed Time: 762 s
Number of Scans: 50
Effective Detector Width: 0.100 eV

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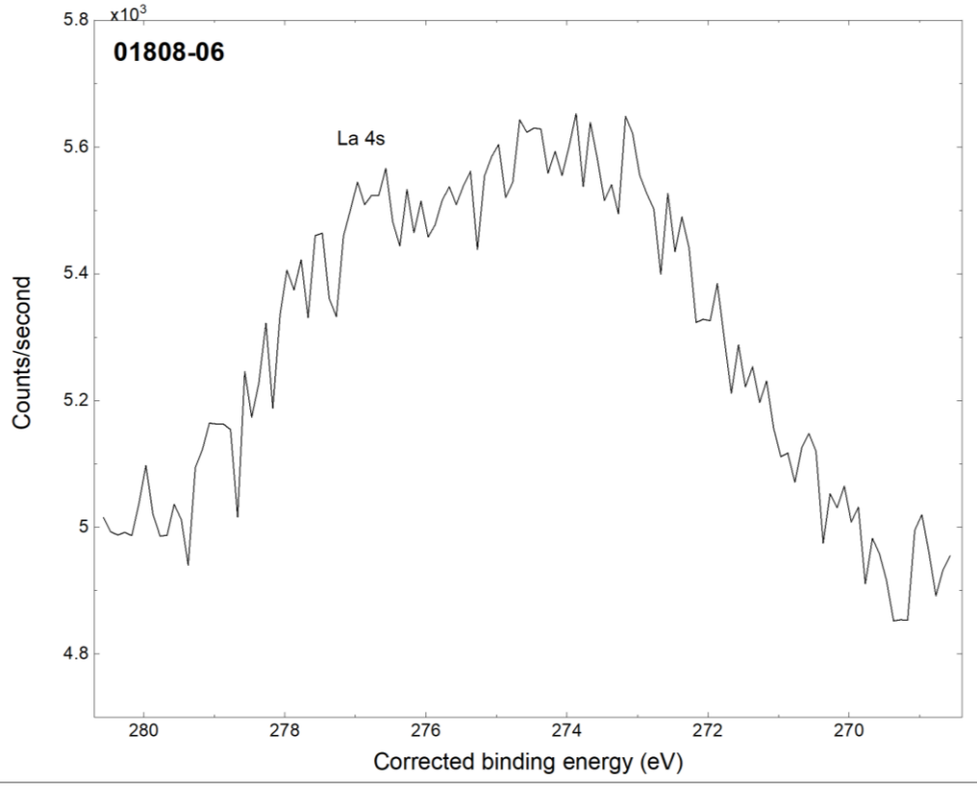
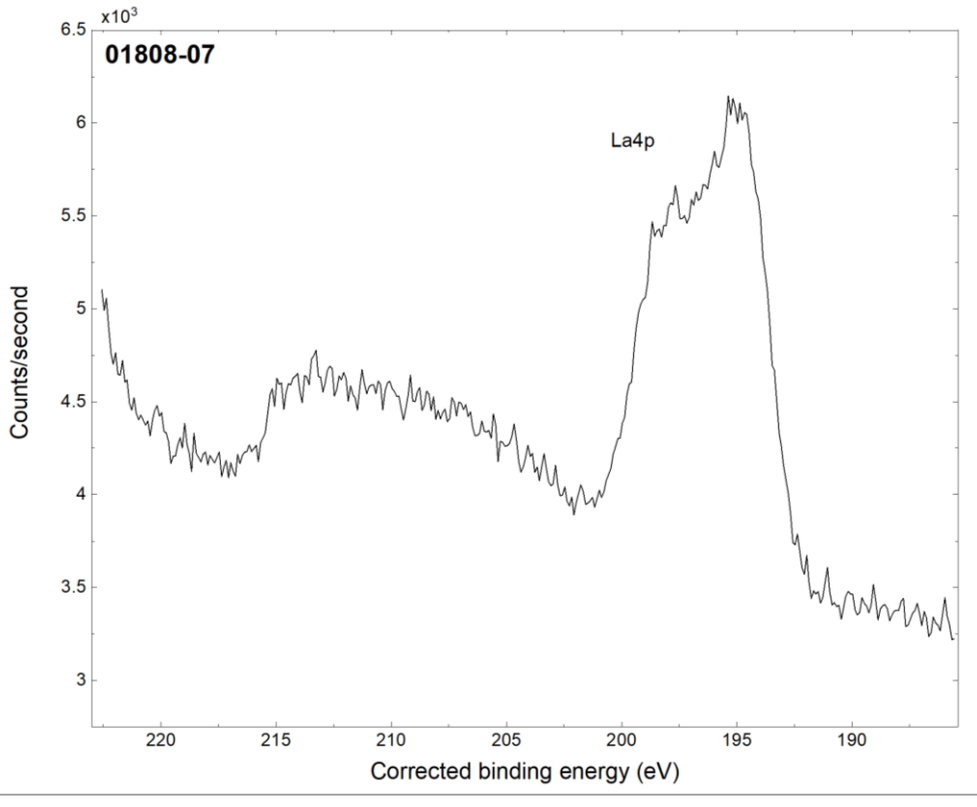


- **Accession #:** 01808-06
- **Host Material:** Lanthanum Sulfide La_2S_3 Bulk Powder
- **Technique:** XPS
- **Spectral Region:** La *4s*

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 mm x 0.9 mm
Analyzer Type: spherical sector
Incident Angle: 58 ˚
Emission Angle: 0 ˚
Analyzer Pass Energy 20 eV
Analyzer Resolution: 0.100 eV
Total Signal Accumulation Time: 121.0 s
Total Elapsed Time: 166 s

Number of Scans: 20

Effective Detector Width: 0.100 eV



- **Accession #:** 01808-07
- **Host Material:** Lanthanum Sulfide La_2S_3 Bulk Powder
- **Technique:** XPS
- **Spectral Region:** La *4p*

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 mm x 0.9 mm
Analyzer Type: spherical sector
Incident Angle: 58 ˚
Emission Angle: 0 ˚
Analyzer Pass Energy 20 eV
Analyzer Resolution: 0.100 eV
Total Signal Accumulation Time: 370.9 s
Total Elapsed Time: 432 s

Number of Scans: 20

Effective Detector Width: 0.100 eV

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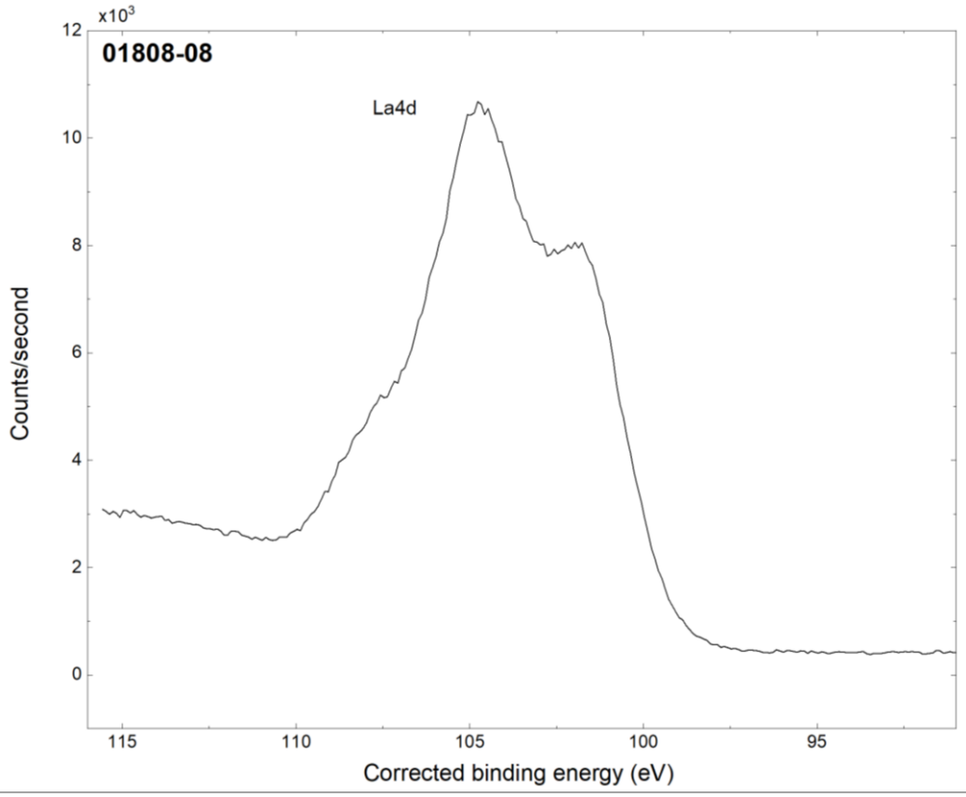


■ **Accession #:** 01808-08

■ **Host Material:** Lanthanum Sulfide La_2S_3 Bulk Powder

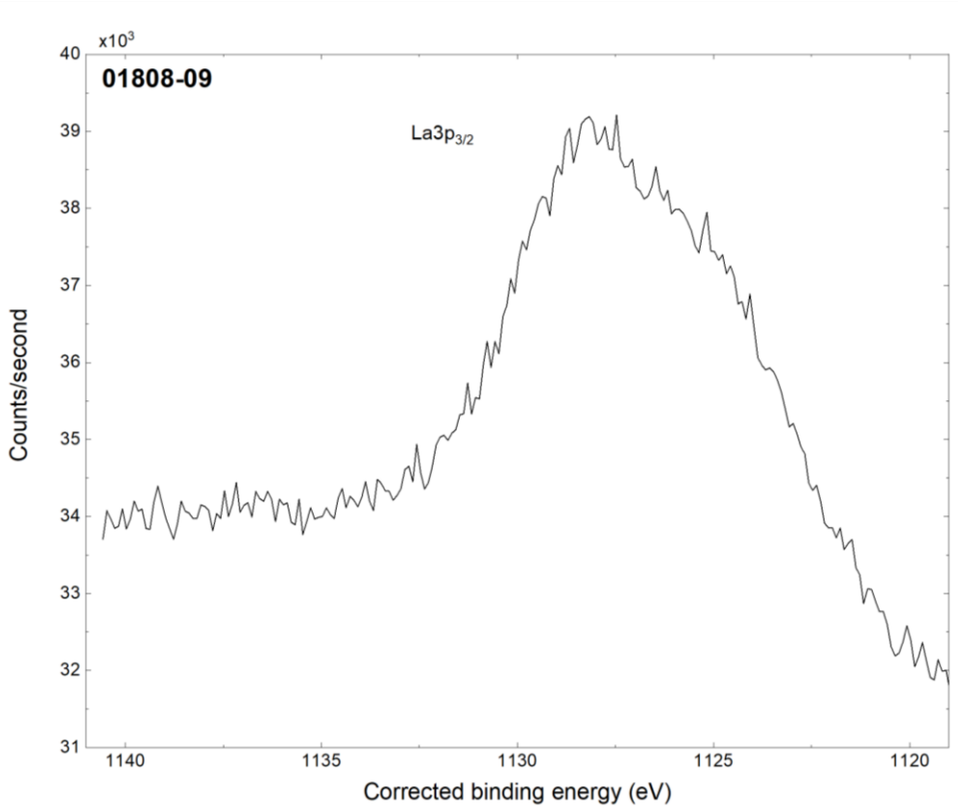
■ **Technique:** XPS

■ **Spectral Region:** La *4d*

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 mm x 0.9 mm
Analyzer Type: spherical sector
Incident Angle: 58 ˚
Emission Angle: 0 ˚
Analyzer Pass Energy 20 eV
Analyzer Resolution: 0.100 eV
Total Signal Accumulation Time: 250.9 s
Total Elapsed Time: 313 s

Number of Scans: 20

Effective Detector Width: 0.100 eV



■ **Accession #:** 01808-09

■ **Host Material:** Lanthanum Sulfide La_2S_3 Bulk Powder

■ **Technique:** XPS

■ **Spectral Region:** La $3p_{3/2}$

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 mm x 0.9 mm
Analyzer Type: spherical sector
Incident Angle: 58 ˚
Emission Angle: 0 ˚
Analyzer Pass Energy 20 eV
Analyzer Resolution: 0.100 eV
Total Signal Accumulation Time: 220.9 s
Total Elapsed Time: 273 s

Number of Scans: 20

Effective Detector Width: 0.100 eV

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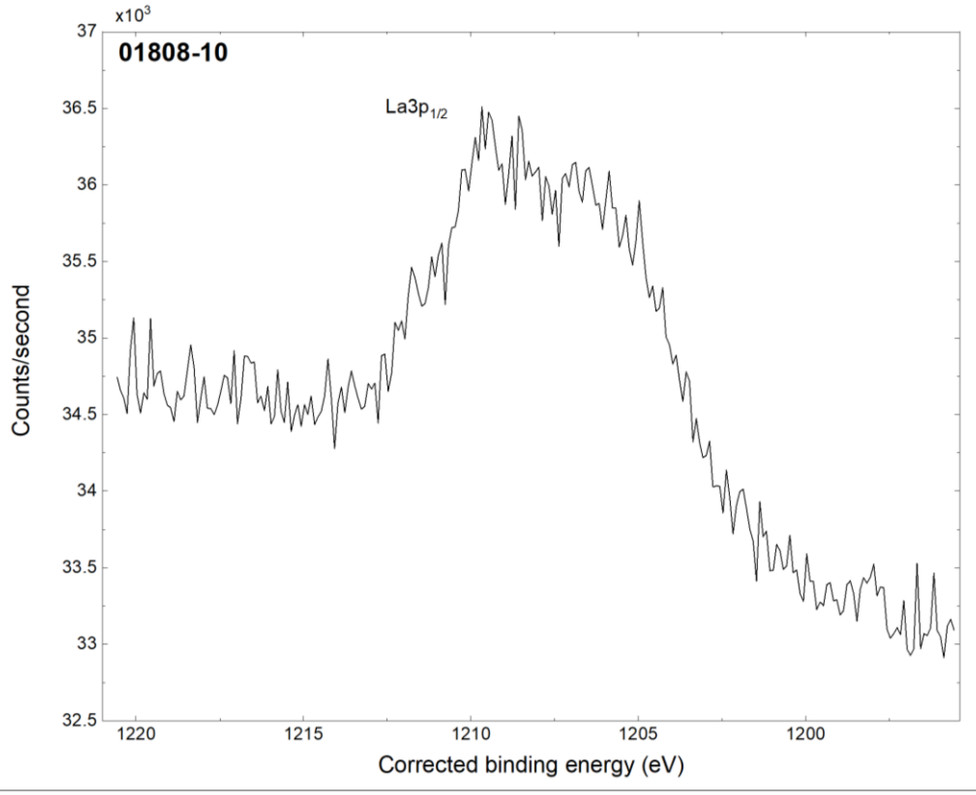
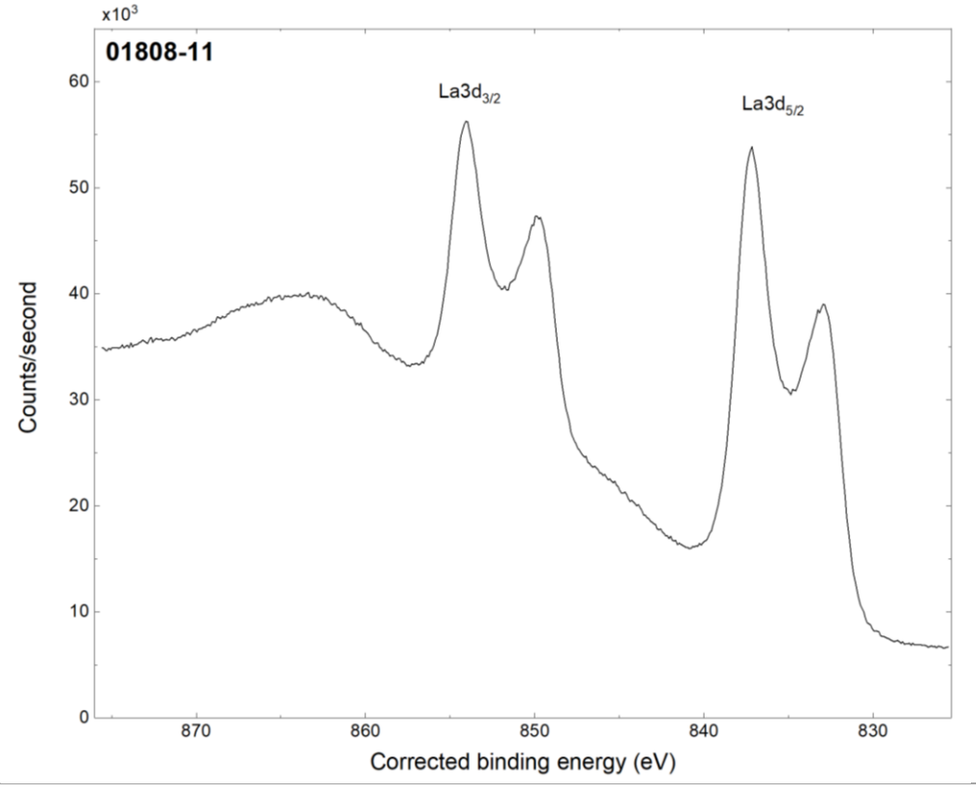


- **Accession #:** 01808-10
- **Host Material:** Lanthanum Sulfide La_2S_3 Bulk Powder
- **Technique:** XPS
- **Spectral Region:** La *3p1/2*

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 mm x 0.9 mm
Analyzer Type: spherical sector
Incident Angle: 58 ˚
Emission Angle: 0 ˚
Analyzer Pass Energy 20 eV
Analyzer Resolution: 0.100 eV
Total Signal Accumulation Time: 250.9 s
Total Elapsed Time: 304 s
Number of Scans: 20
Effective Detector Width: 0.100 eV



- **Accession #:** 01808-11
- **Host Material:** Lanthanum Sulfide La_2S_3 Bulk Powder
- **Technique:** XPS
- **Spectral Region:** La *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 mm x 0.9 mm
Analyzer Type: spherical sector
Incident Angle: 58 ˚
Emission Angle: 0 ˚
Analyzer Pass Energy 20 eV
Analyzer Resolution: 0.100 eV
Total Signal Accumulation Time: 500.8 s
Total Elapsed Time: 572 s
Number of Scans: 20
Effective Detector Width: 0.100 eV

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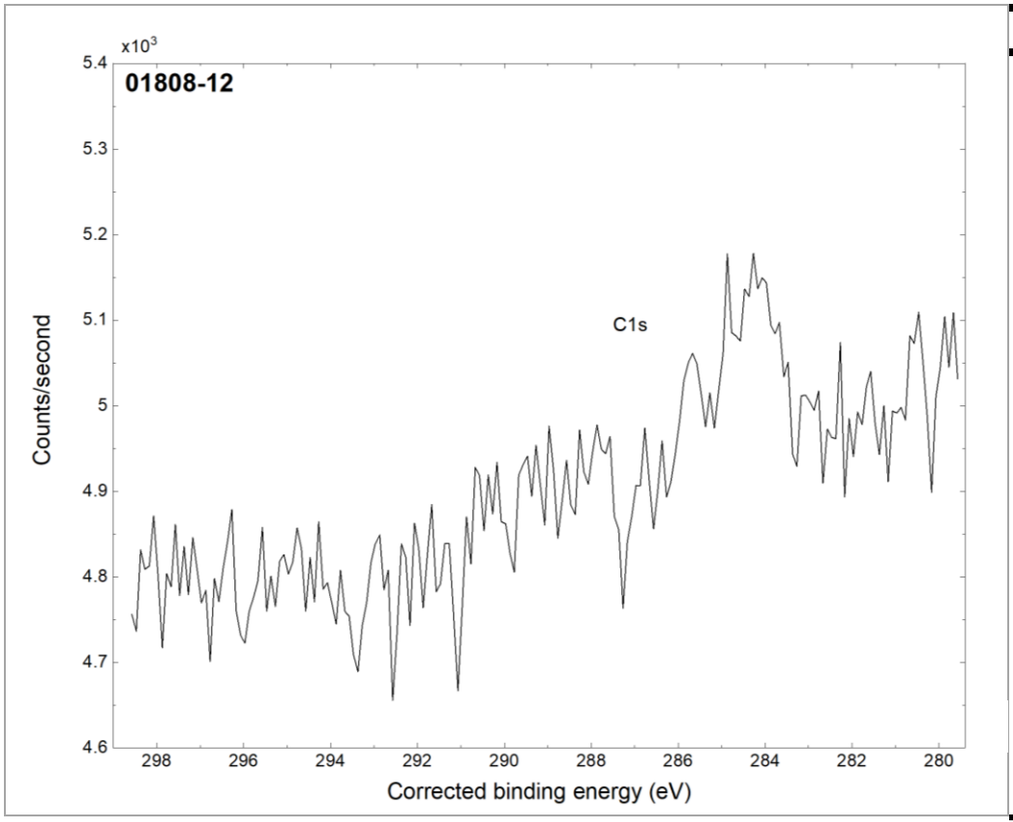

■ **Accession #:** 01808-12

■ **Host Material:** Lanthanum Sulfide La_2S_3 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 mm x 0.9 mm

Analyzer Type: spherical sector

Incident Angle: 58 ˚

Emission Angle: 0 ˚

Analyzer Pass Energy 20 eV

Analyzer Resolution: 0.100 eV

Total Signal Accumulation Time: 238.7 s

Total Elapsed Time: 301 s

Number of Scans: 25

Effective Detector Width: 0.100 eV

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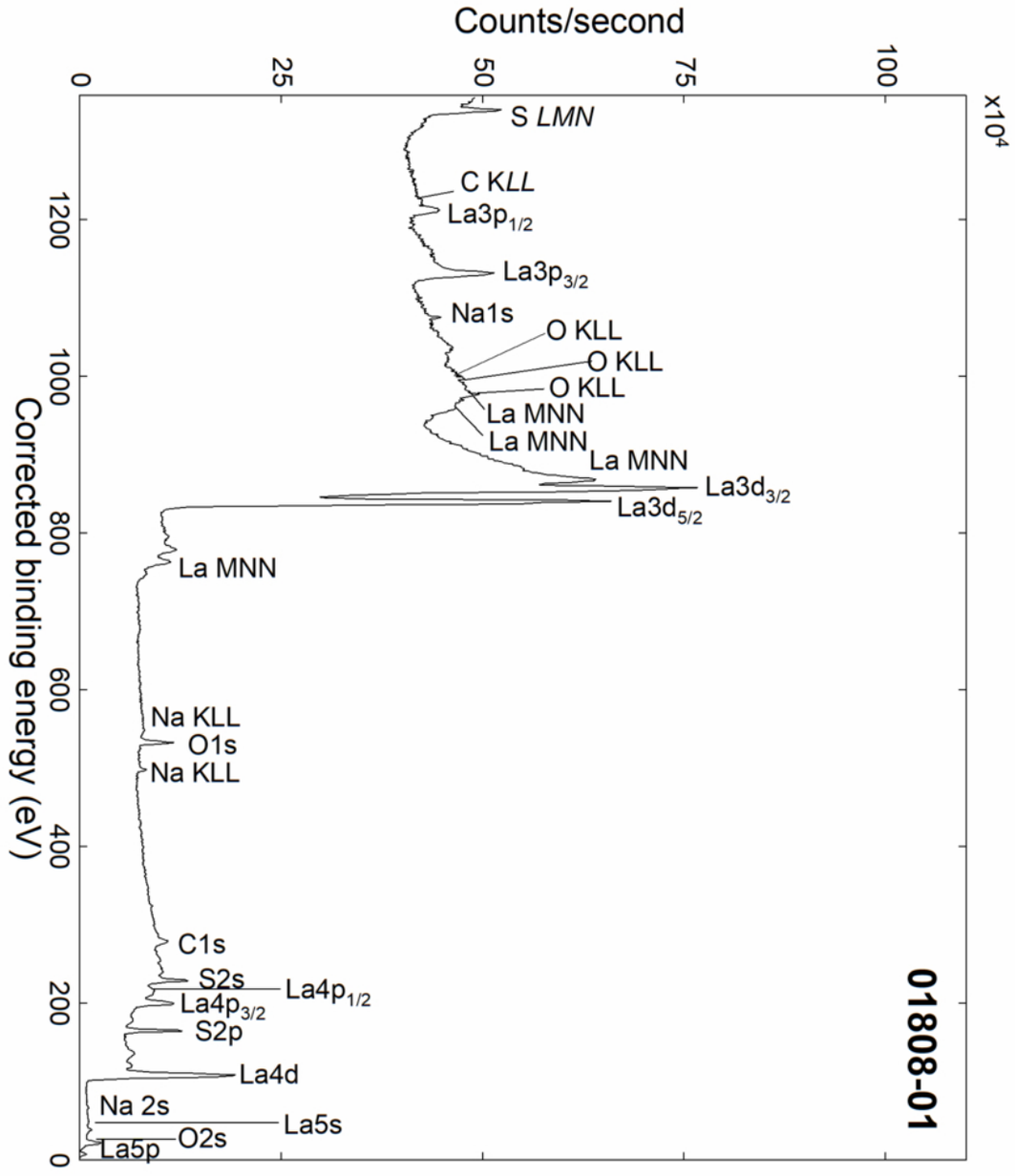

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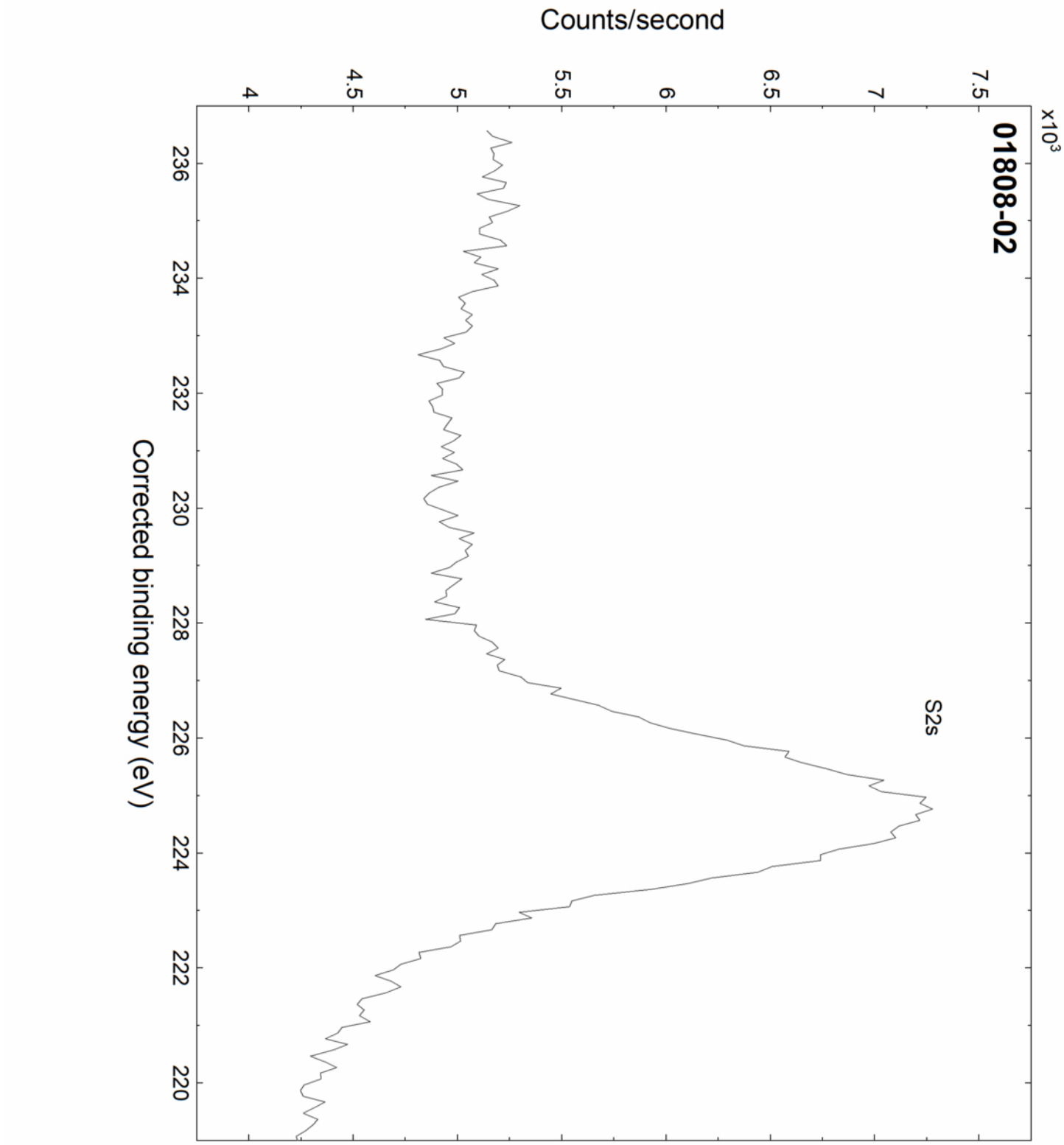

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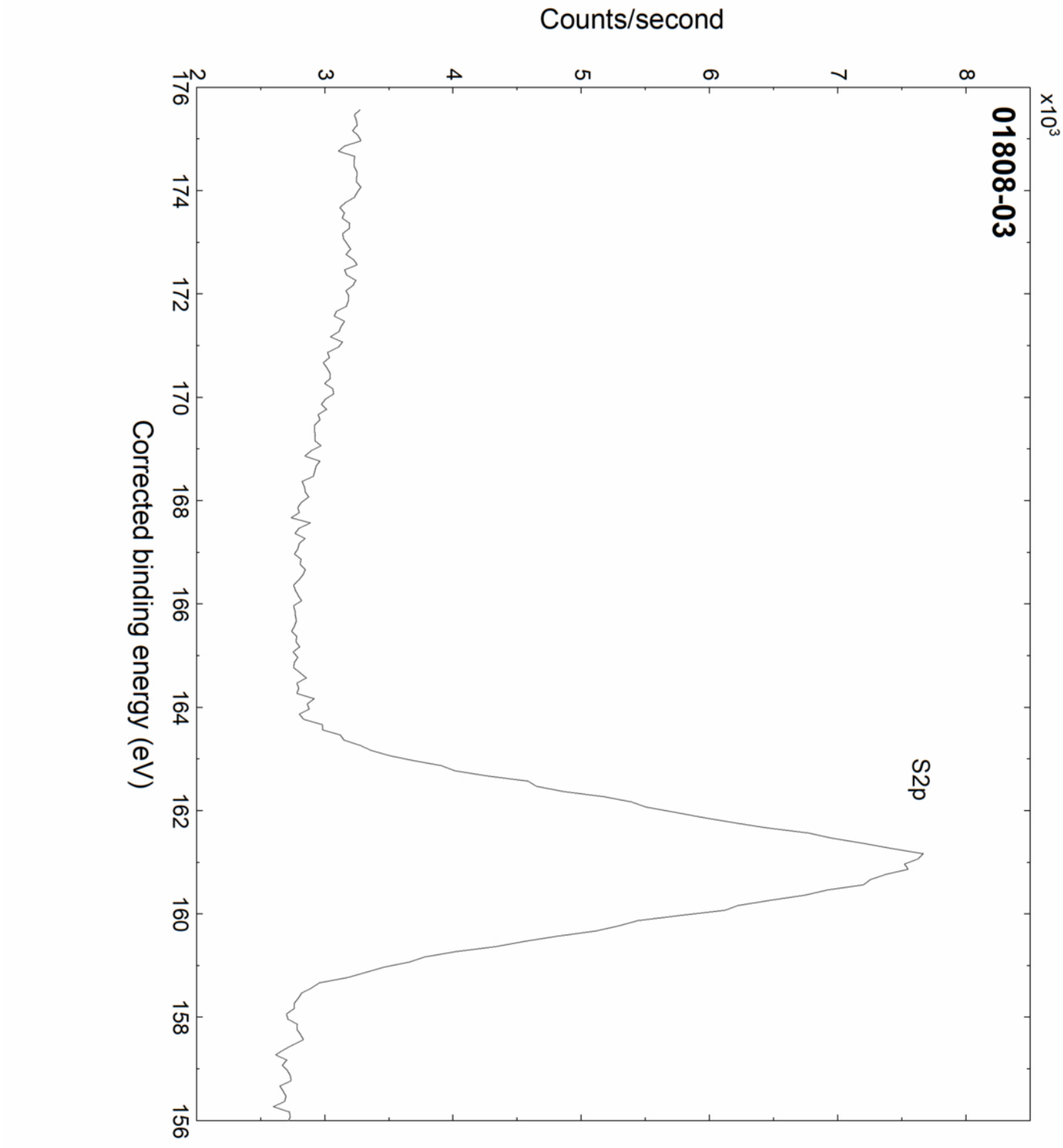

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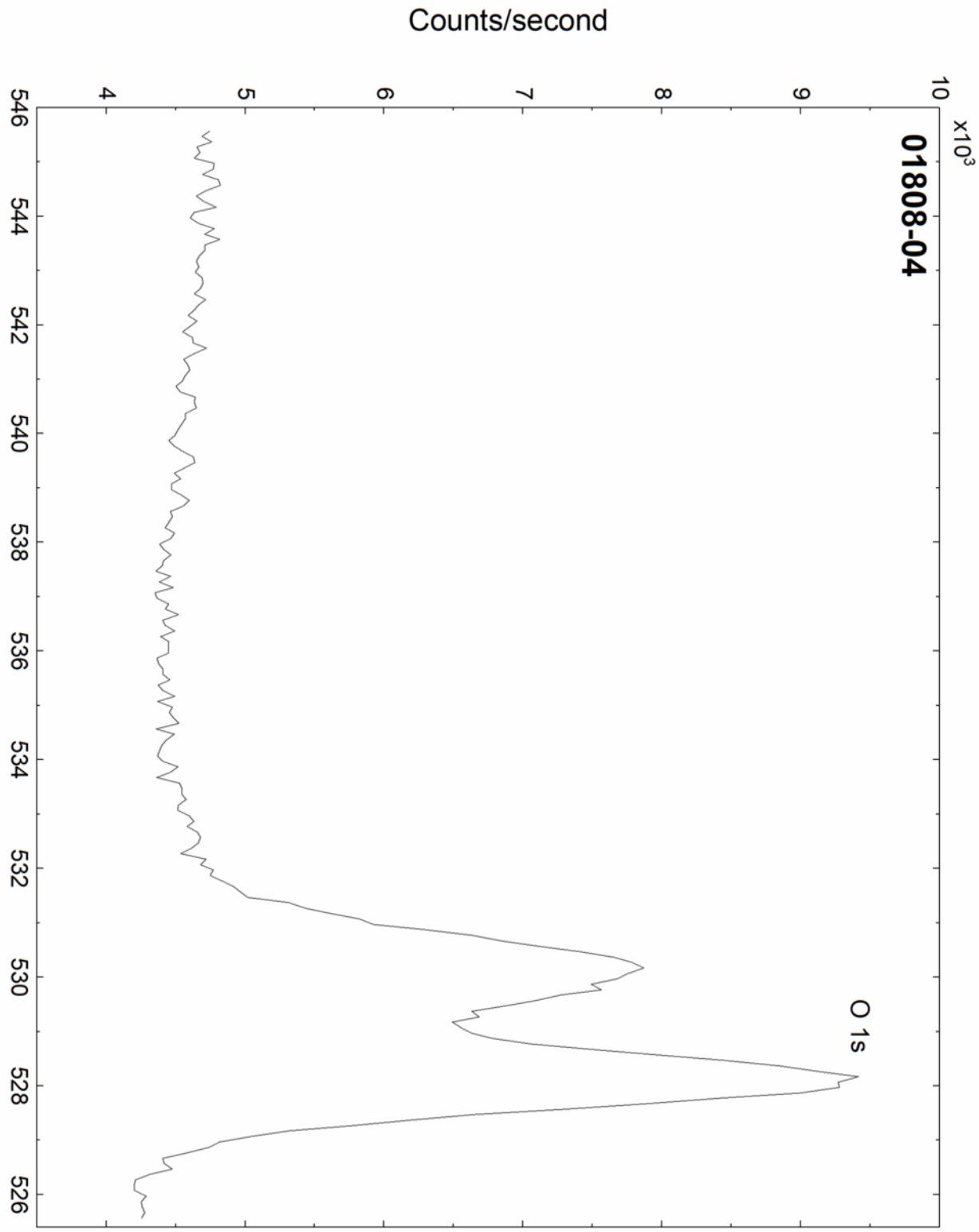

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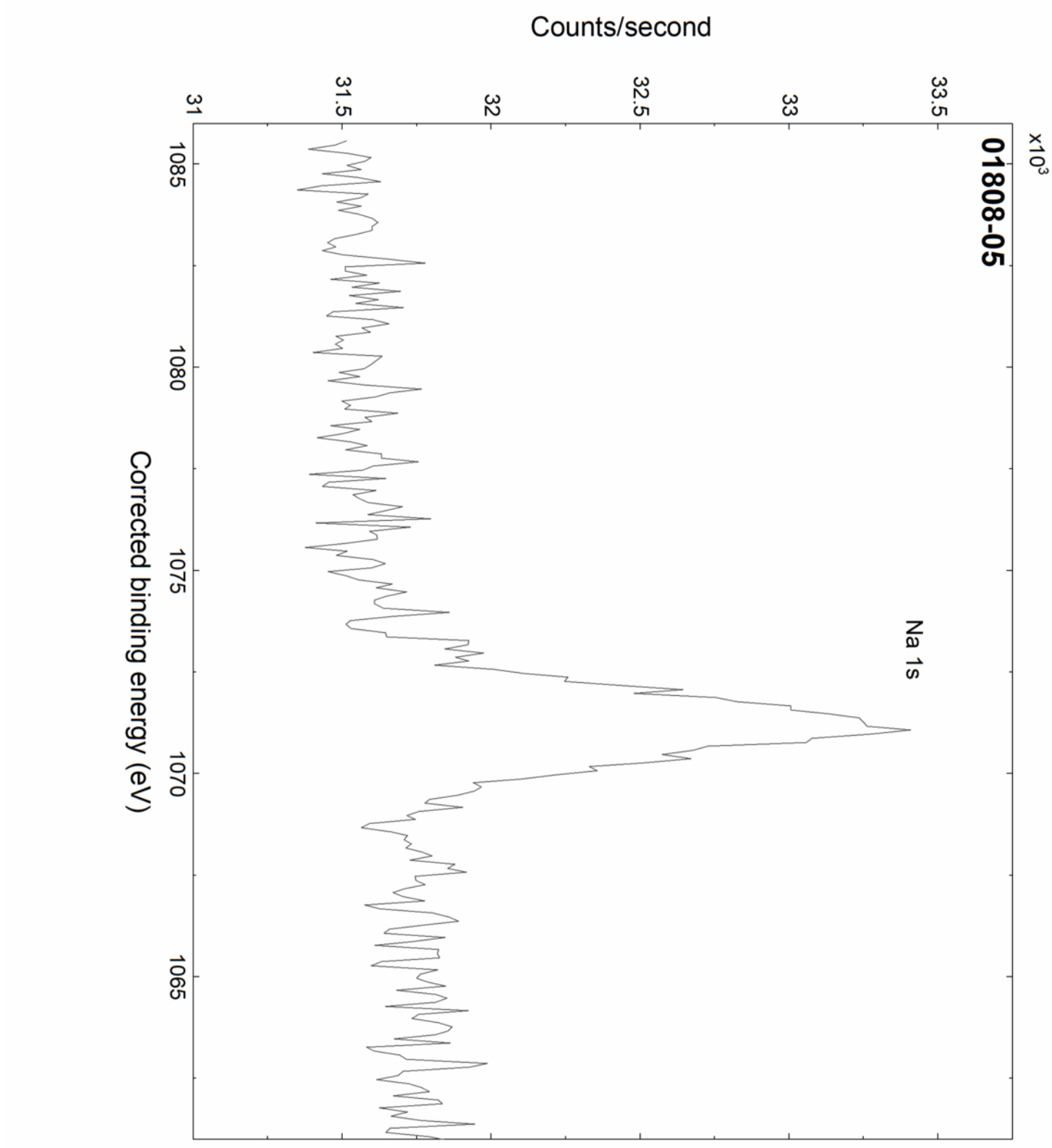

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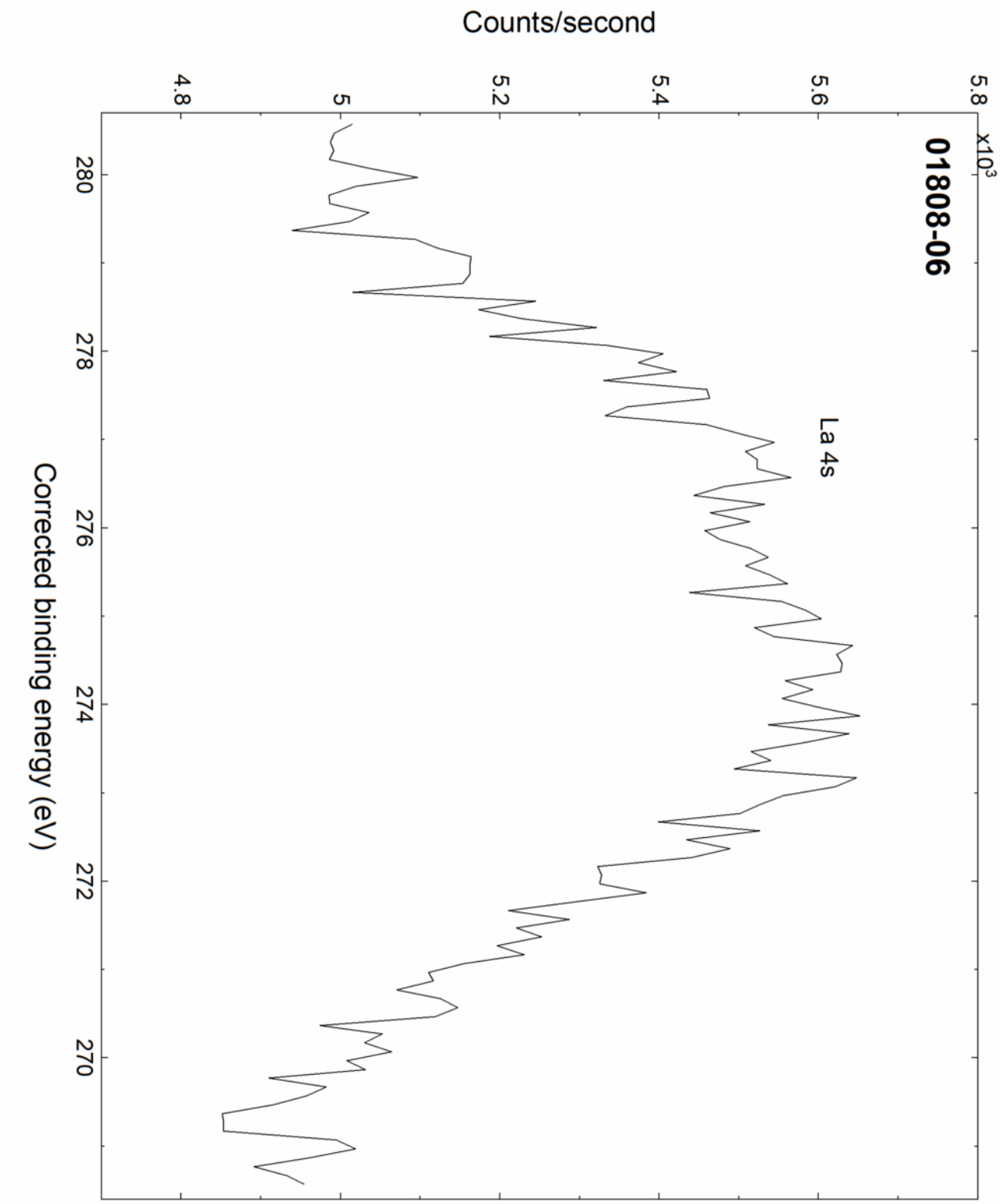

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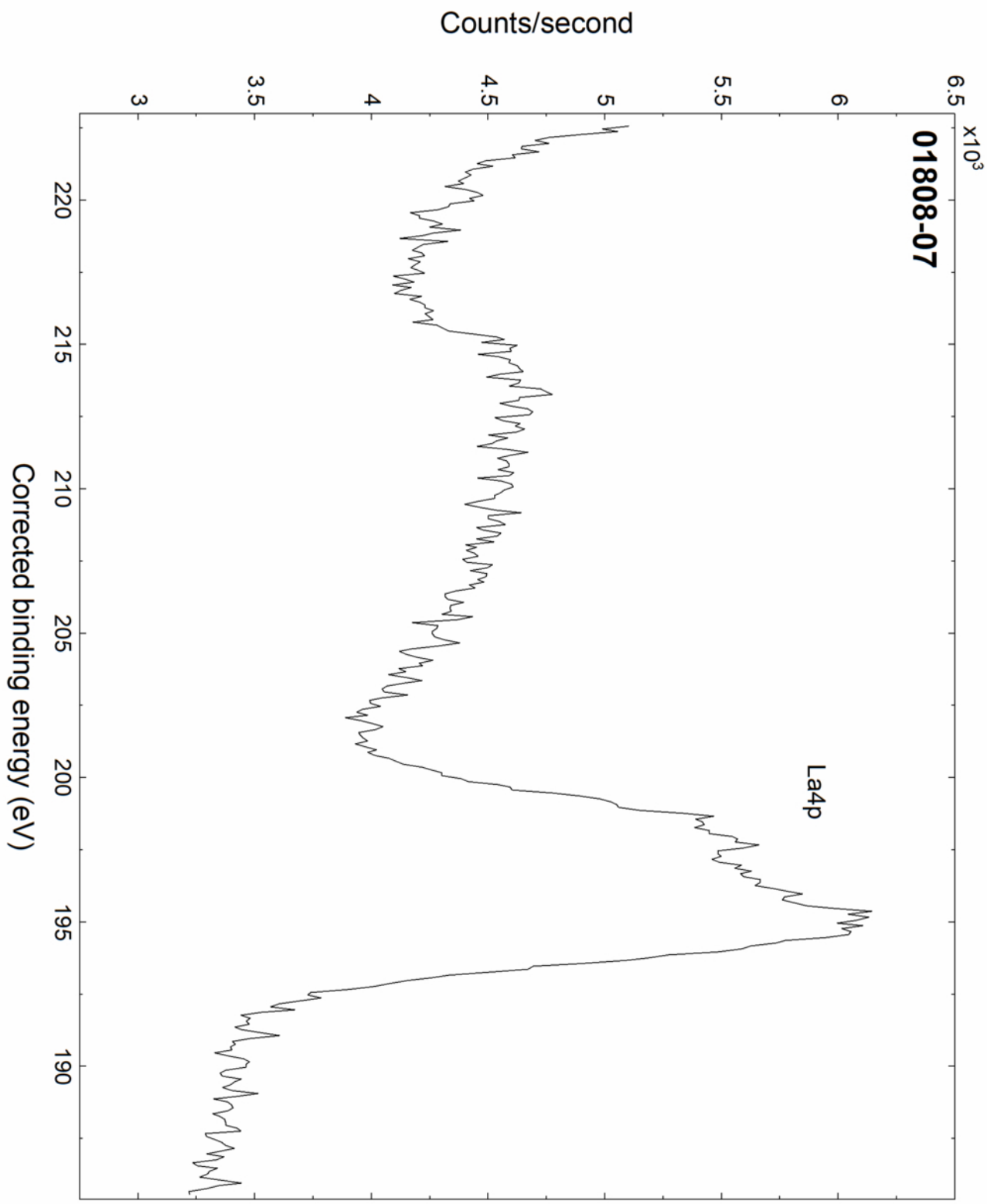

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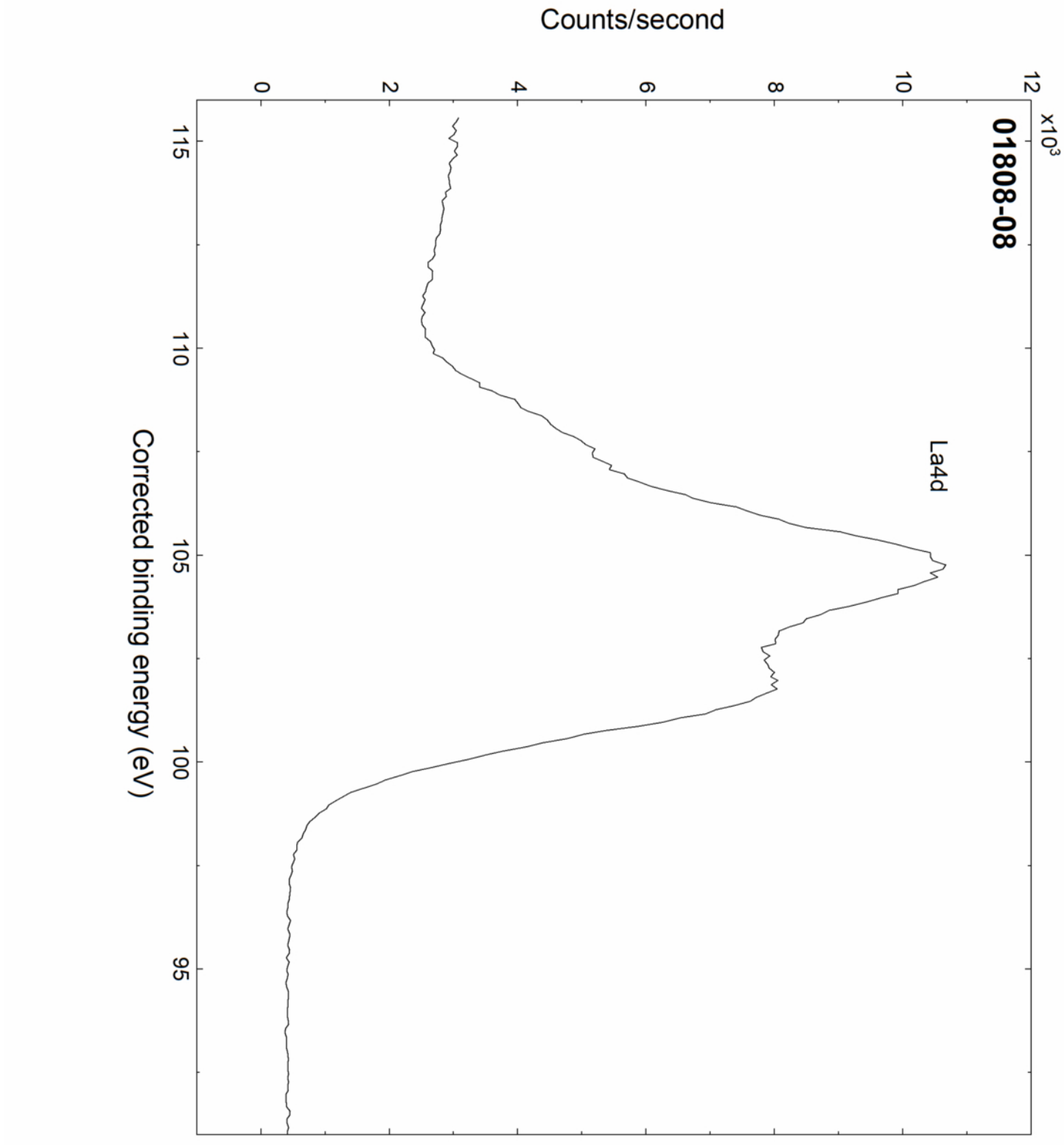

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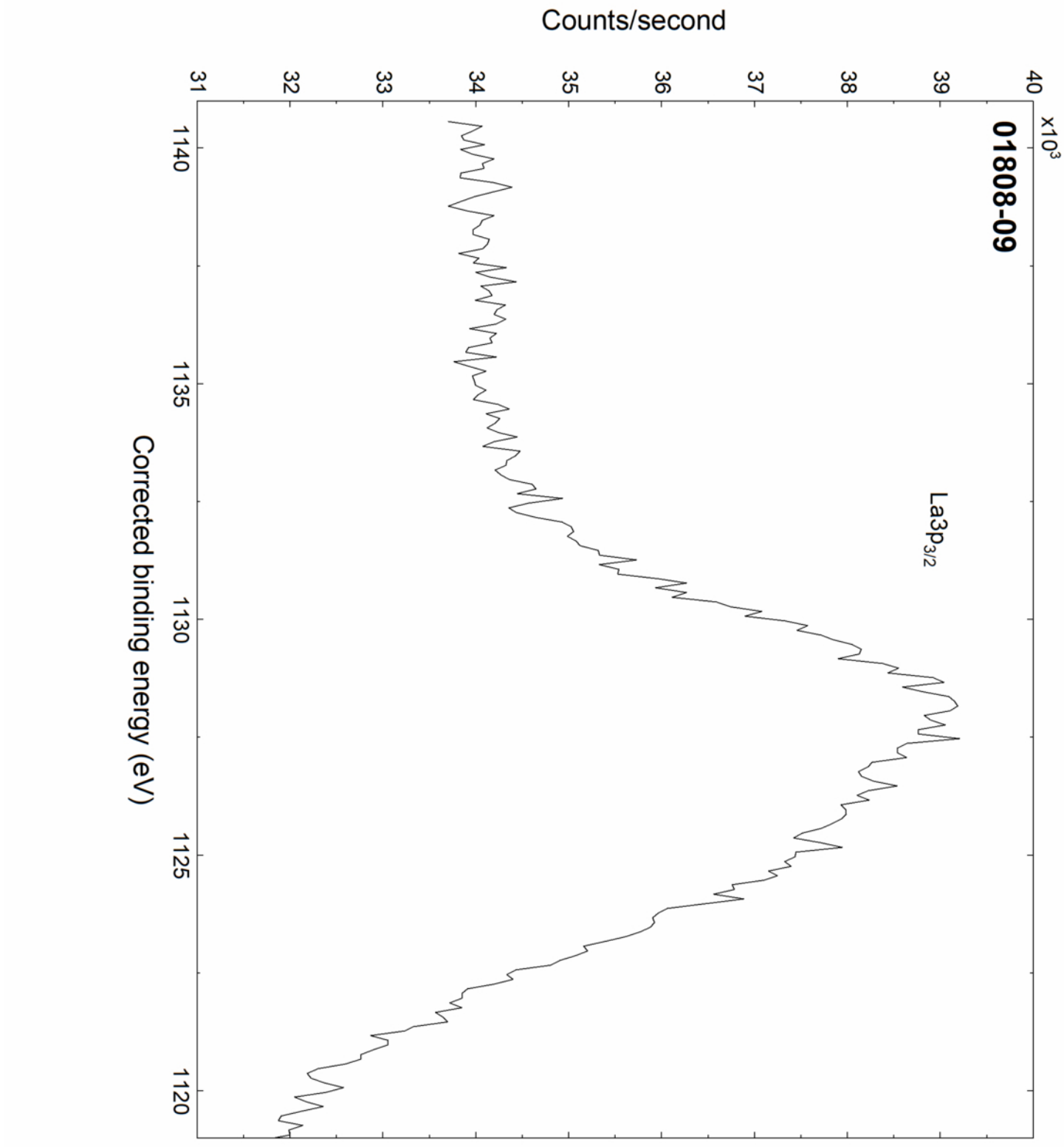

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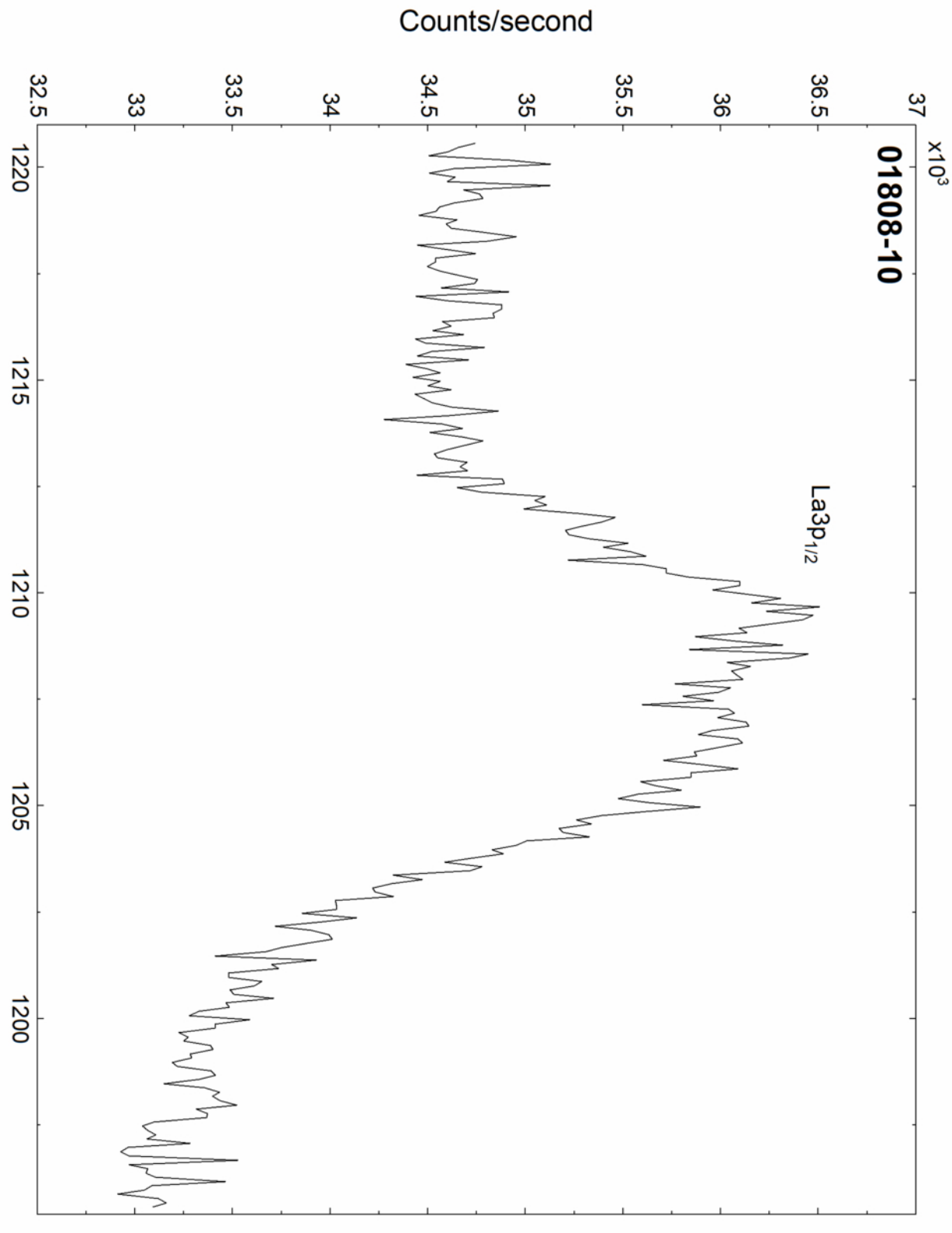

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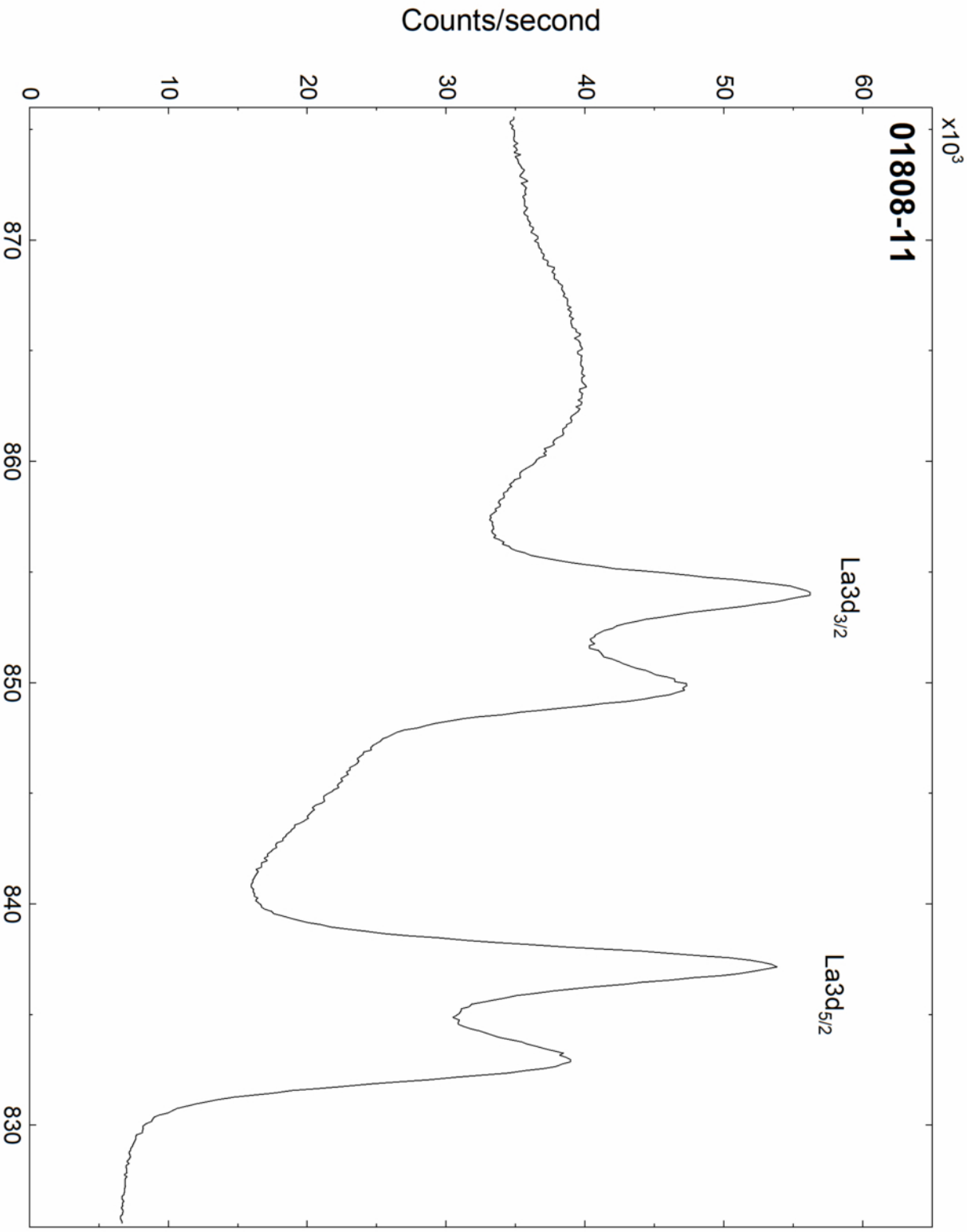

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