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HAXPES of Al₂O₃ with Cr K α excitation

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A thick alumina sample was analyzed by hard x-ray photoelectron spectroscopy (HAXPES) by means of a lab-scale spectrometer equipped with a Cr K α (5414.8 eV) excitation source. The reported spectra include a survey scan as well as Al 1s, Al 2s, Al 2p, O 1s and C 1s core-level spectra.

Keywords: Al₂O₃, HAXPES, Cr K α

INTRODUCTION

Alumina has attracted a lot of attention for its use as gate insulator material in high electron mobility transistor (HEMT) applications. X-ray photoelectron spectroscopy (XPS) is a powerful method to investigate the interface between Al₂O₃ and the underlying semiconductor. However, the low probing depth of XPS prevents from studying realistic devices. Thanks to the increase of the inelastic mean free path of electrons with increasing photon energy, hard x-ray photoelectron spectroscopy (HAXPES) has become a useful tool to probe deeper into materials and, therefore, study more realistic stacks. Interestingly, the recent advent of novel lab-based instruments brings this technique to the expected access level compared to synchrotron facilities.

In this work, we analyzed a thick alumina sample by HAXPES with a monochromatic Cr K α (5414.8 eV) source. We provide data that can be used for comparison which include a survey scan as well as high-resolution spectra of Al 1s, Al 2s, Al 2p, O 1s and C 1s.

SPECIMEN DESCRIPTION (ACCESSION # 01710)

Host Material: Al₂O₃

CAS Registry #: 1344-28-1

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

Chemical Name: Aluminum oxide

Source: Unknown

Host Composition: Al₂O₃

Form: Thick sheet

Structure: Trigonal

History & Significance: Air exposed polycrystalline solid

As Received Condition: 20x25mm alumina sheet

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Accession#: 01710

Technique: XPS

Host Material: Al₂O₃

Instrument: ULVAC-PHI Quantes

Major Elements in Spectra: Al, O

Minor Elements in Spectra: C

Published Spectra: 6

Spectra in Electronic Record: 6

Spectral Category: comparison

Analyzed Region: Same as host material

Ex Situ Preparation/Mounting: The sample was mounted on the sample holder using double sided conductive tape.

In Situ Preparation: The sample was sputter cleaned by low energy Ar⁺ ions (100 eV) for 1h prior to measurements to remove carbon contamination.

Charge Control: Low-energy electrons (1 eV, filament 1.1 A) and low-energy ions (10 eV)

Temp. During Analysis: 300 K

Pressure During Analysis: < 6.10⁻⁷ Pa

Pre-analysis Beam Exposure: 0 s.

INSTRUMENT DESCRIPTION

Manufacturer and Model: ULVAC-PHI Quantes

Analyzer Type: spherical sector

Detector: multichannel resistive plate

Number of Detector Elements: 32

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

■ Spectrometer

Analyzer Mode: constant pass energy

Throughput (T=E^N): The energy dependence can be modeled using the following equation: $\frac{A}{E_p} = \left(\frac{a^2}{a^2 + R^2}\right)^b$, where a and b are constants, E_p is the pass energy, A is the peak area and R is the retard ratio equal to E/E_p, where E is the kinetic energy. Three spectral regions are recorded on a sputter cleaned sample at different pass energies. The values of a and b are then determined by a linear least square fit of the data applying the equation described above.

Excitation Source Window: Al

Excitation Source: Cr K α monochromatic

Source Energy: 5414.8 eV

Source Strength: 50 W

Source Beam Size: 100 μm x 100 μm

Signal Mode: multichannel direct

■ Geometry

Incident Angle: 22 °

Source-to-Analyzer Angle: 46 °

Emission Angle: 45 °

Specimen Azimuthal Angle: 0 °

Acceptance Angle from Analyzer Axis: 0 °

Analyzer Angular Acceptance Width: 20 ° x 20 °

■ Ion Gun

Manufacturer and Model: ULVAC-PHI Quantes

Energy: 10 and 100 eV

Current: 1.4×10^{-5} mA

Current Measurement Method: Faraday cup

Sputtering Species: Ar

Spot Size (unrastered): 100 μm

Raster Size: N/A

Incident Angle: 45 °

Polar Angle: 45 °

Azimuthal Angle: 45 °

Comment: Differentially pumped ion gun used for pre-sputtering of the sample and to prevent reoxidation during analysis.

DATA ANALYSIS METHOD

Energy Scale Correction: The decrease of photoionization cross-sections in HAXPES (Refs. 1 and 2) leads to a very low C 1s intensity. Therefore, the binding energy was referenced to the Al 2p binding energy position measured with Al K α radiation after shifting the C 1s peak to 284.8 eV. Doing so, the Al 2p binding energy was 75.1 eV. The spectra recorded with the Cr K α source were then rescaled by shifting the Al 2p to 75.1 eV.

Recommended Energy Scale Shift: 2.0 eV for binding energy

Peak Shape and Background Method: Shirley background was employed for peak area determination. No curve fitting was performed on the spectra.

Quantitation Method: PHI Multipak software Version 9.9.0.8 was used to perform quantification. Empirically determined sensitivity factors (RSFs) were provided by the software. The RSFs were derived from the pure-element relative sensitivity factor as defined in ISO 18118:2015 (Ref 3) which were measured on pure element samples using a Cr K α source. They therefore account for the decrease of cross-section and different escape depth of photoelectrons using higher energy photons. RSFs are reported proportional to the RSF of F 1s equal to 1. The reported concentrations were calculated using these RSFs corrected to include the transmission function and the asymmetry parameter.

ACKNOWLEDGMENTS

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DATA AVAILABILITY STATEMENT

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

REFERENCES

1. M.B. Trzhaskovskaya and V.G. Yarzhemsky, *At. Data Nucl. Data Tables* **119**, 99 (2018).
2. M.B. Trzhaskovskaya and V.G. Yarzhemsky, *At. Data Nucl. Data Tables* **129-130**, 101280 (2019).
3. International Organization for Standardization 2015, *Surface chemical analysis - Auger electron spectroscopy and X-ray photoelectron spectroscopy - Guide to the use of experimentally determined relative sensitivity factors for the quantitative analysis of homogeneous materials*, ISO 18118:2015.

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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
01710-02	Al 1s	1562.0	2.13	14593	4.238	35.9	Al ₂ O ₃
01710-03	Al 2s	120.1	2.49	1089	0.369	...	Al ₂ O ₃
01710-04	Al 2p	75.1	1.96	292	0.075	...	Al ₂ O ₃
01710-05	O 1s	531.9	2.13	3048	0.589	60.1	Al ₂ O ₃
01710-06	C 1s	285.0	2.15	68	0.199	4.1	Contamination

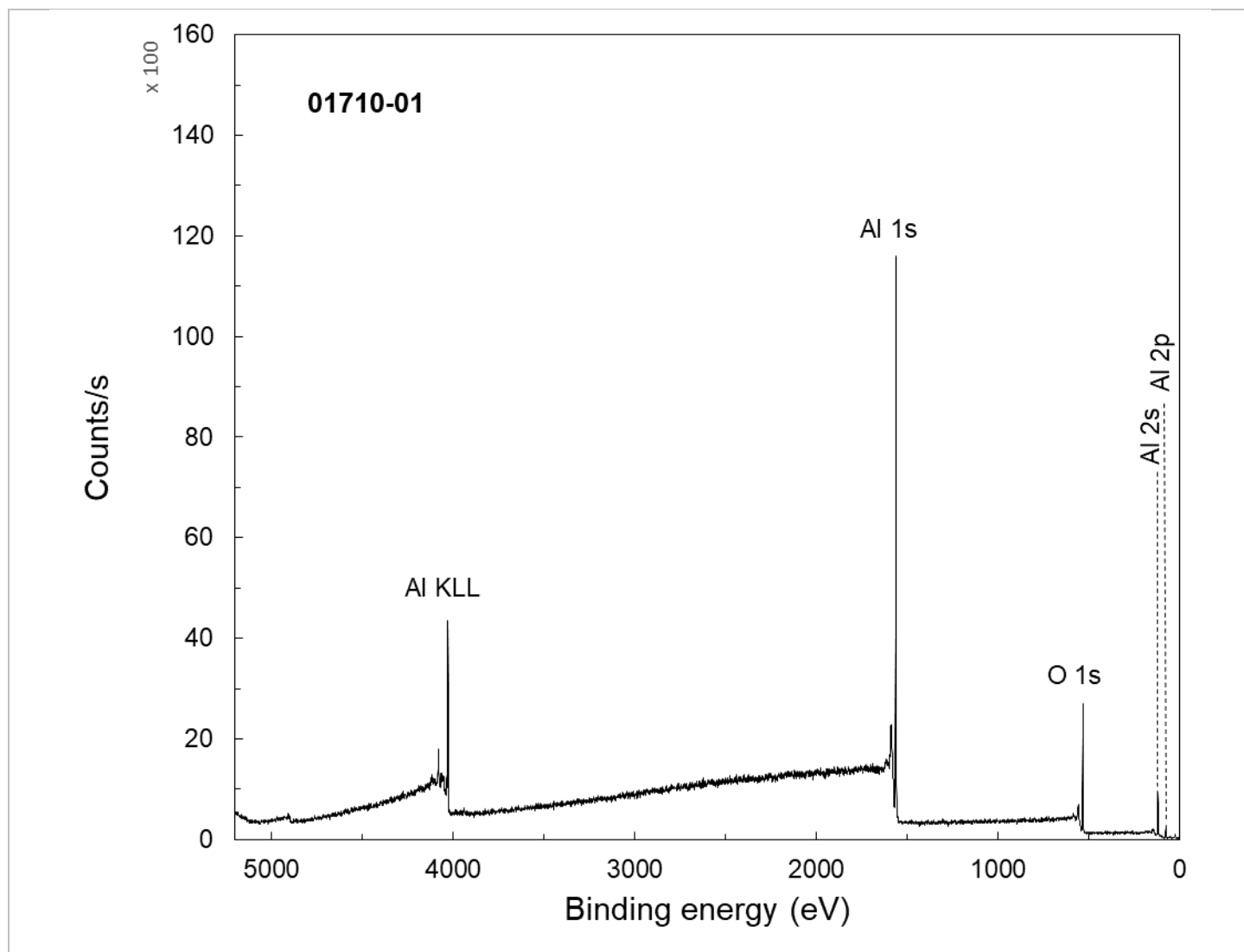
ANALYZER CALIBRATION TABLE							
Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV x cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
...	Ag 3d _{5/2}	368.12	0.63	114999
...	Cu 2p _{3/2}	932.61	0.96	40205
...	Au 4f _{7/2}	83.89	0.78	100500

The spectra in the analyzer calibration table were recorded using Al K α photons.

GUIDE TO FIGURES					
Spectrum (Accession) #	Spectral Region	Voltage Shift*	Multiplier	Baseline	Comment #
01710-01	Survey	0	1	0	...
01710-02	Al 1s	-2.0	1	0	...
01710-03	Al 2s	-2.0	1	0	...
01710-04	Al 2p	-2.0	1	0	...
01710-05	O 1s	-2.0	1	0	...
01710-06	C 1s	-2.0	1	0	...

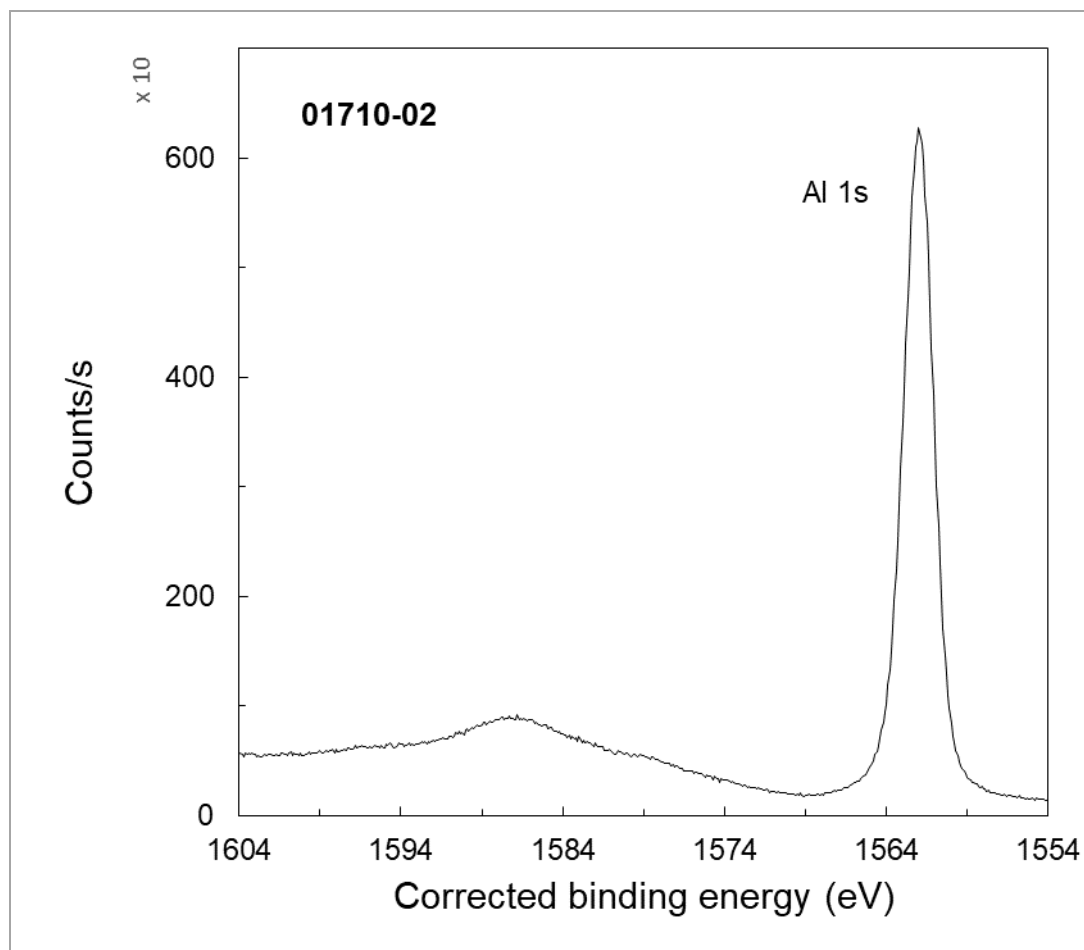
*Voltage shift of the archived (as-measured) spectrum relative to the printed figure. The figure reflects the recommended energy scale correction due to a calibration correction, sample charging, flood gun, or other phenomenon.

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Accession #	01710-01
Host Material	Al ₂ O ₃
Technique	XPS
Spectral Region	survey
Instrument	ULVAC-PHI Quantes
Excitation Source	Cr K _α monochromatic
Source Energy	5414.8 eV
Source Strength	50 W
Source Size	0.1 mm x 0.1 mm
Analyzer Type	spherical sector analyzer
Incident Angle	22°
Emission Angle	45°
Analyzer Pass Energy	280 eV
Analyzer Resolution	2.33 eV
Total Signal Accumulation Time	5240 s
Total Elapsed Time	5760 s
Number of Scans	10
Effective Detector Width	31 eV

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■ **Accession #:** 01710-02

■ **Host Material:** Al₂O₃

■ **Technique:** XPS

■ **Spectral Region:** Al 1s

Instrument: ULVAC-PHI

Quantes

Excitation Source:

Cr K_α monochromatic

Source Energy: 5414.8 eV

Source Strength: 50 W

Source Size: 0.1 mm x 0.1 mm

Analyzer Type: spherical sector

Incident Angle: 22 °

Emission Angle: 45 °

Analyzer Pass Energy 112 eV

Analyzer Resolution: 1.17 eV

Total Signal Accumulation

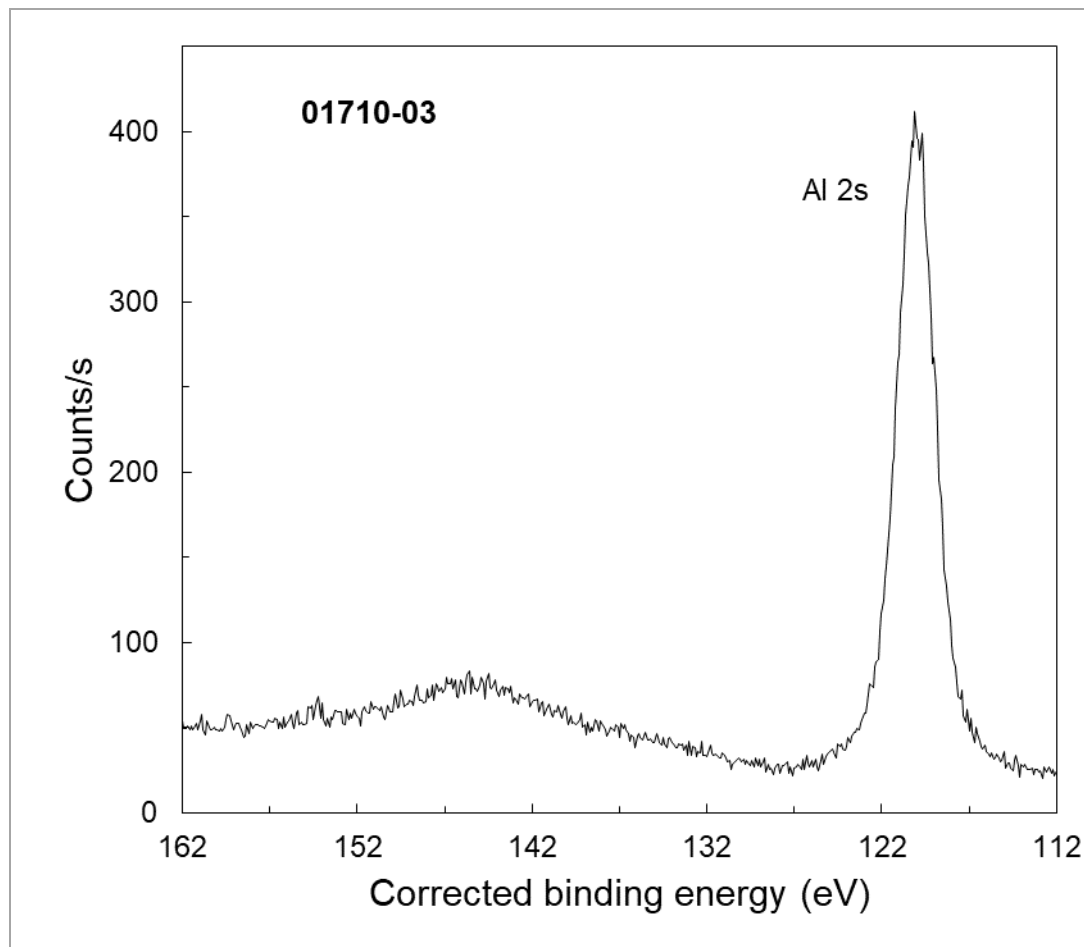
Time: 3740 s

Total Elapsed Time: 4120 s

Number of Scans: 15

Effective Detector Width: 12.4 eV

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■ Accession #: 01710-03

■ Host Material: Al₂O₃

■ Technique: XPS

■ Spectral Region: Al 2s

Instrument: ULVAC-PHI

Quantes

Excitation Source:

Cr K_α monochromatic

Source Energy: 5414.8 eV

Source Strength: 50 W

Source Size: 0.1 mm x 0.1 mm

Analyzer Type: spherical sector

Incident Angle: 22 °

Emission Angle: 45 °

Analyzer Pass Energy 112 eV

Analyzer Resolution: 1.17 eV

Total Signal Accumulation

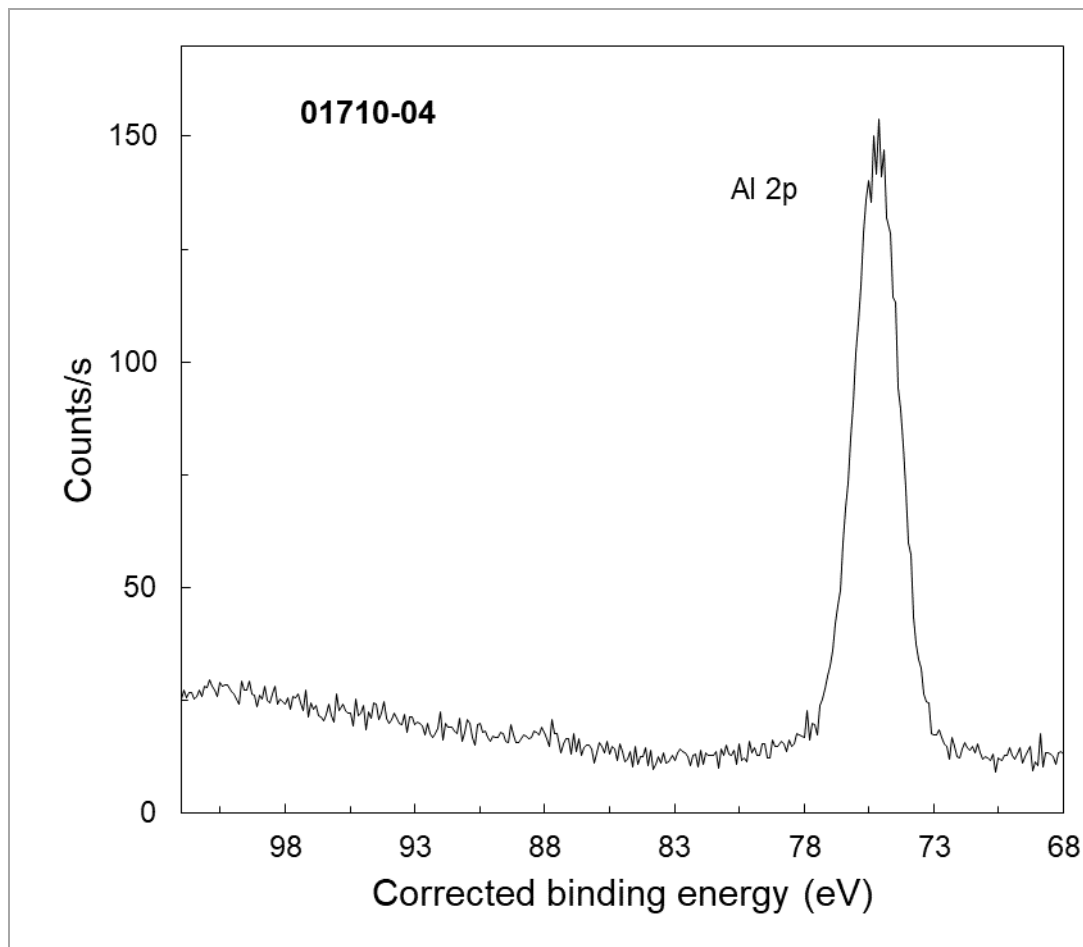
Time: 3740 s

Total Elapsed Time: 4120 s

Number of Scans: 15

Effective Detector Width: 12.4 eV

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■ Accession #: 01710-04

■ Host Material: Al₂O₃

■ Technique: XPS

■ Spectral Region: Al 2p

Instrument: ULVAC-PHI

Quantes

Excitation Source:

Cr K_α monochromatic

Source Energy: 5414.8 eV

Source Strength: 50 W

Source Size: 0.1 mm x 0.1 mm

Analyzer Type: spherical sector

Incident Angle: 22 °

Emission Angle: 45 °

Analyzer Pass Energy 112 eV

Analyzer Resolution: 1.17 eV

Total Signal Accumulation

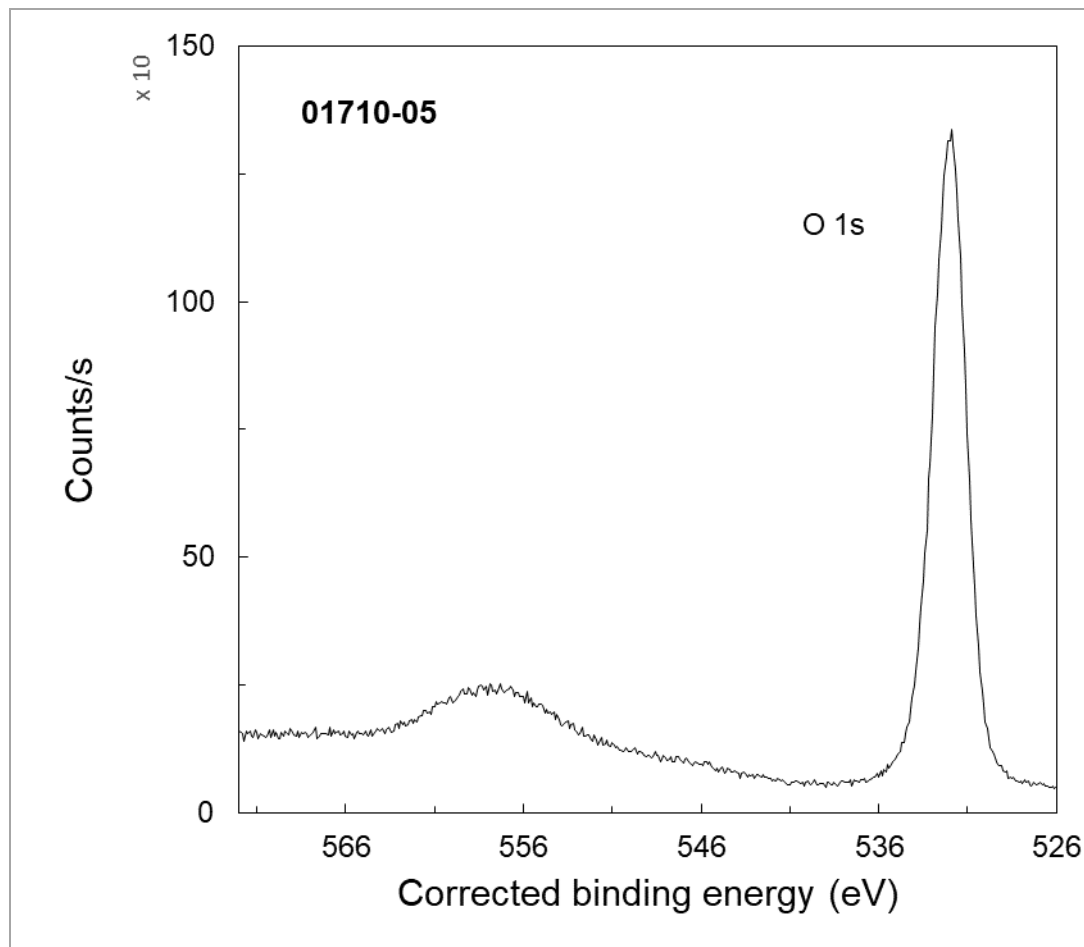
Time: 3710 s

Total Elapsed Time: 4090 s

Number of Scans: 20

Effective Detector Width: 12.4 eV

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■ Accession #: 01710-05

■ Host Material: Al₂O₃

■ Technique: XPS

■ Spectral Region: O 1s

Instrument: ULVAC-PHI

Quantes

Excitation Source:

Cr K α monochromatic

Source Energy: 5414.8 eV

Source Strength: 50 W

Source Size: 0.1 mm x 0.1 mm

Analyzer Type: spherical sector

Incident Angle: 22 °

Emission Angle: 45 °

Analyzer Pass Energy 112 eV

Analyzer Resolution: 1.17 eV

Total Signal Accumulation

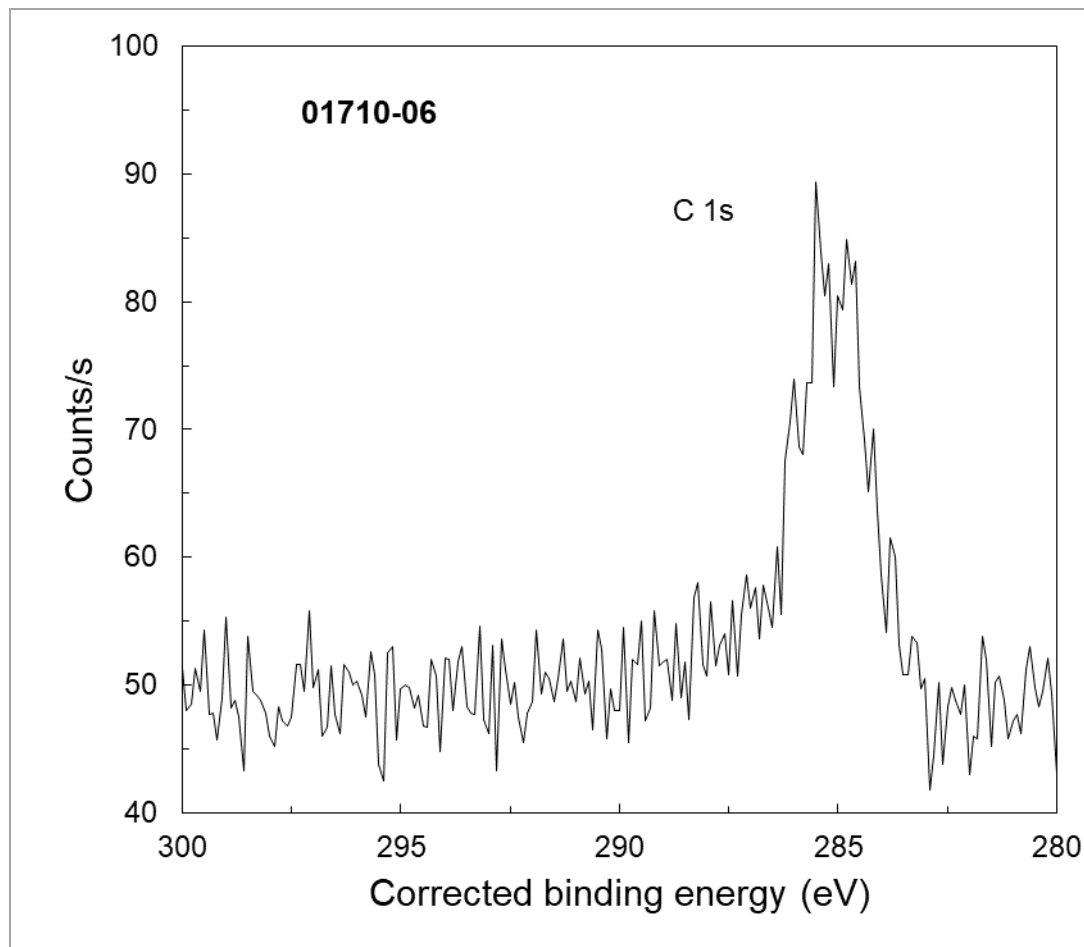
Time: 3970 s

Total Elapsed Time: 4370 s

Number of Scans: 17

Effective Detector Width: 12.4 eV

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■ Accession #: 01710-06

■ Host Material: Al₂O₃

■ Technique: XPS

■ Spectral Region: C 1s

Instrument: ULVAC-PHI

Quantes

Excitation Source:

Cr K_α monochromatic

Source Energy: 5414.8 eV

Source Strength: 50 W

Source Size: 0.1 mm x 0.1 mm

Analyzer Type: spherical sector

Incident Angle: 22 °

Emission Angle: 45 °

Analyzer Pass Energy 112 eV

Analyzer Resolution: 1.17 eV

Total Signal Accumulation

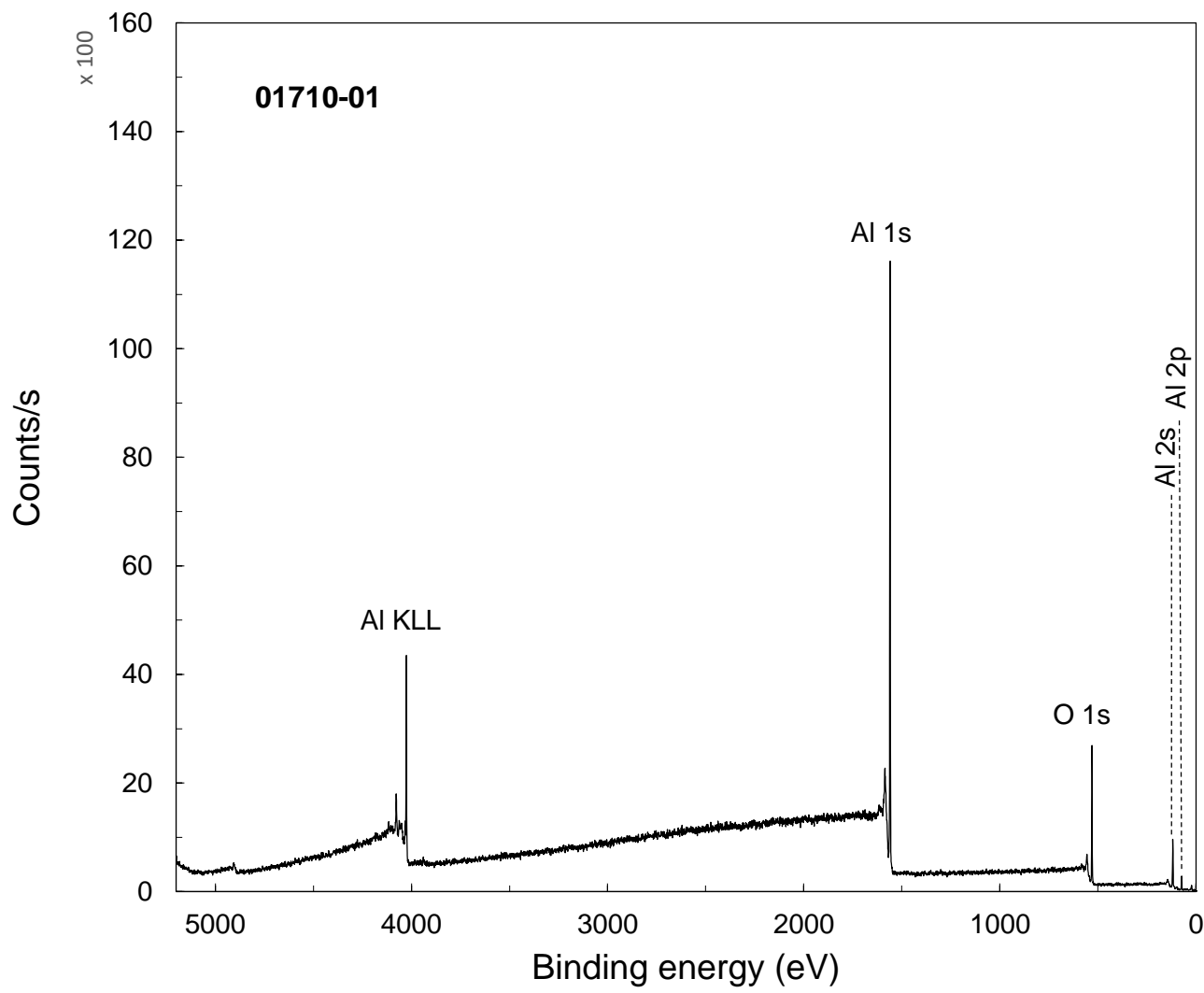
Time: 1940 s

Total Elapsed Time: 2140 s

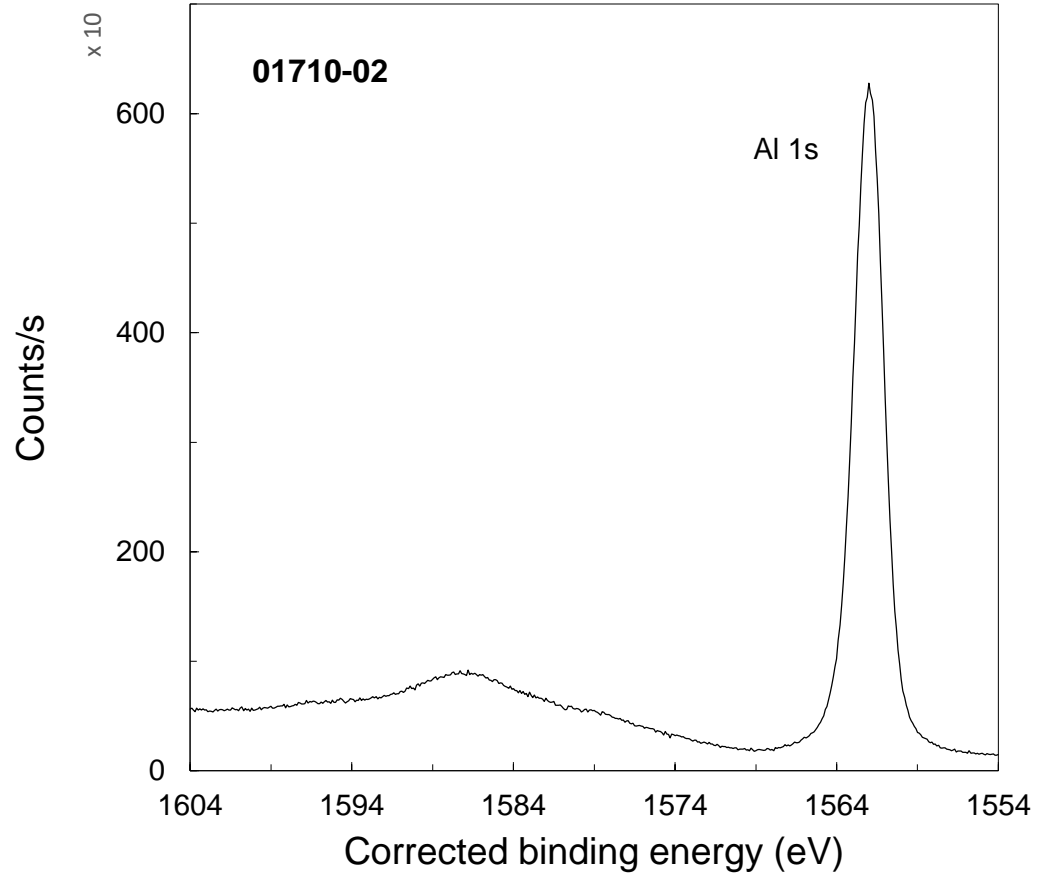
Number of Scans: 15

Effective Detector Width: 12.4 eV

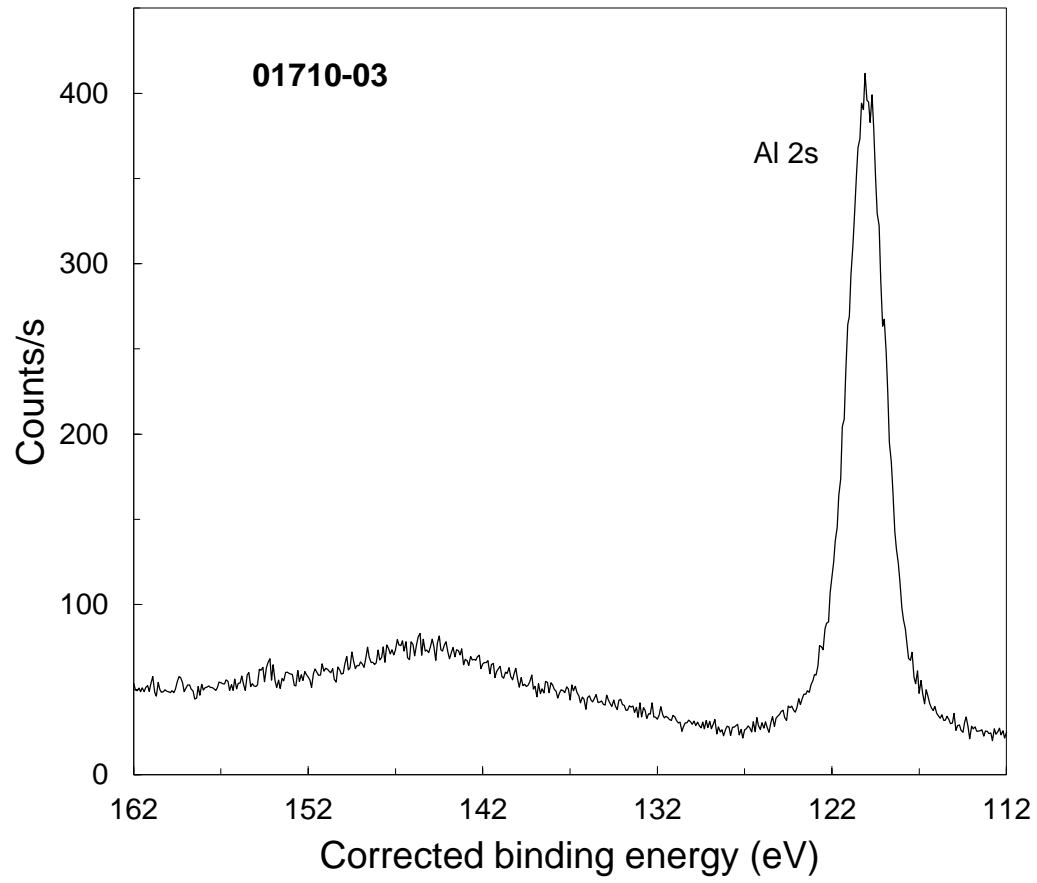
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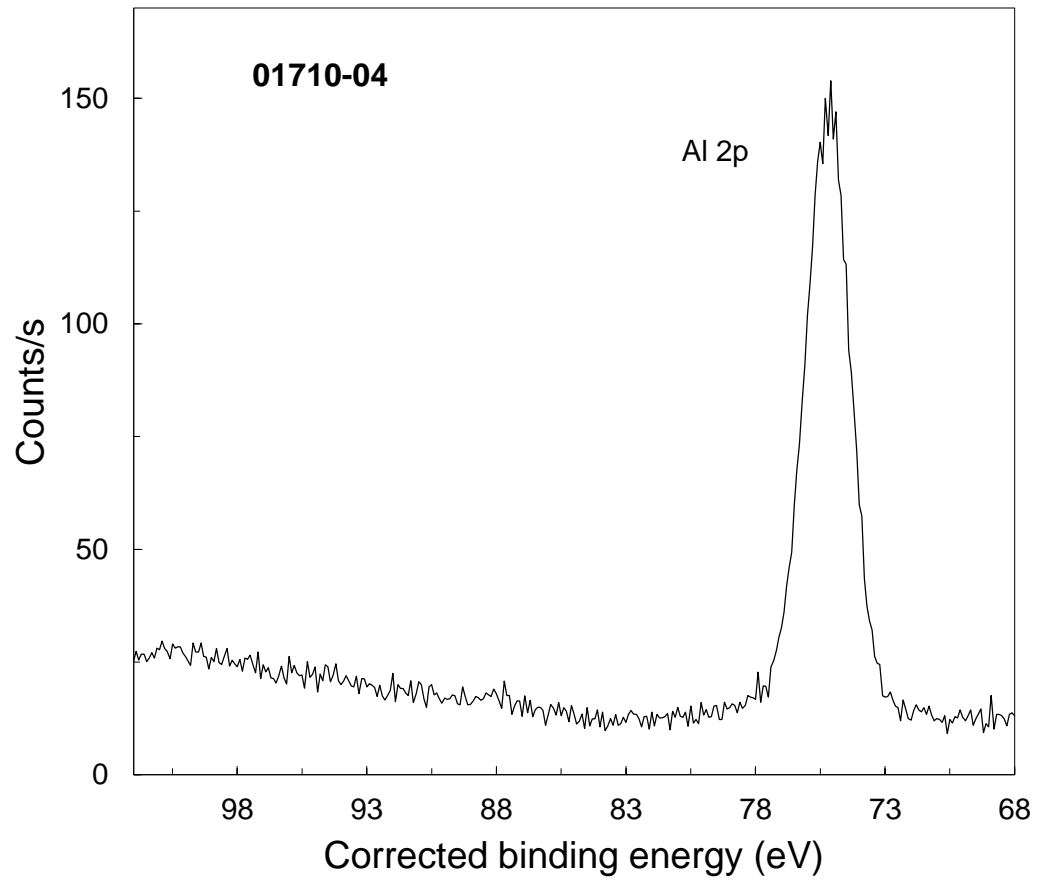
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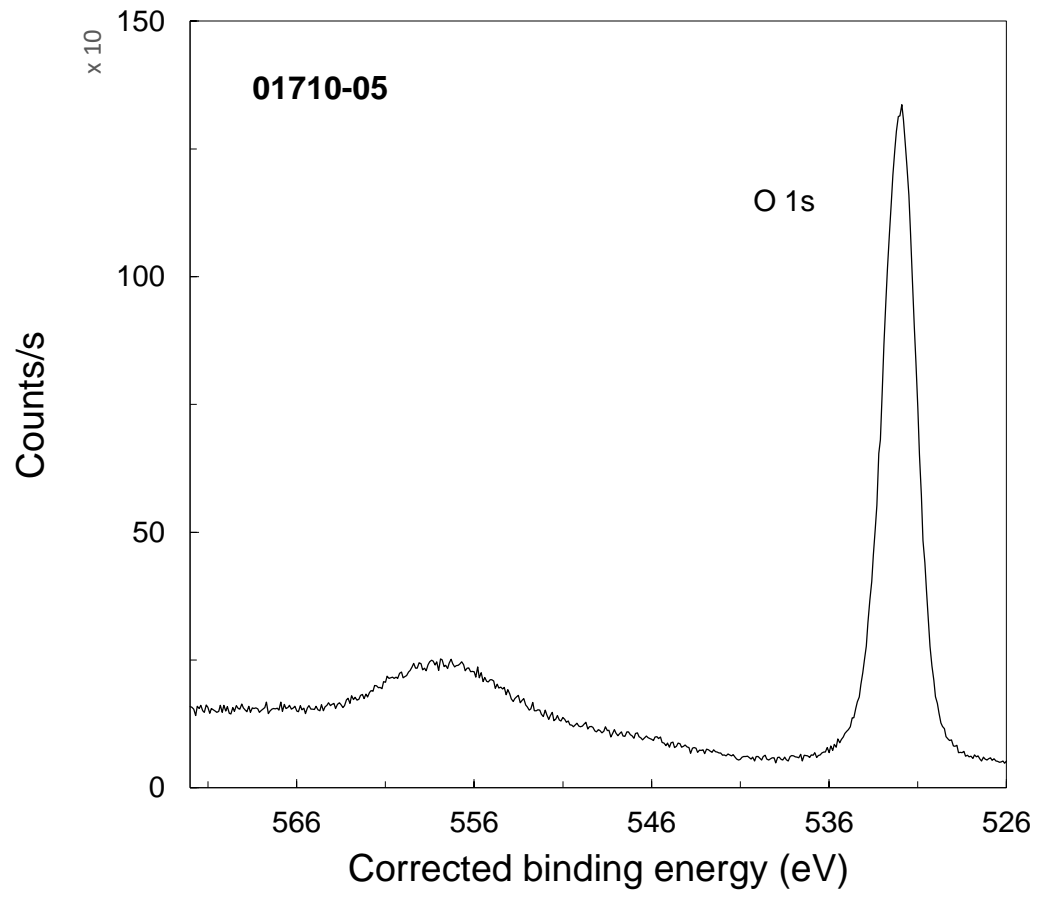
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