

RESEARCH ARTICLE | MAY 10 2022

High-energy x-ray photoelectron spectroscopy spectra of Al_2O_3 measured by Cr K α

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Surface Science Spectra 29, 014021 (2022)

<https://doi.org/10.1116/6.0001577>



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High-energy x-ray photoelectron spectroscopy spectra of Al₂O₃ measured by Cr K α

Cite as: Surf. Sci. Spectra 29, 014021 (2022); doi: 10.1116/6.0001577

Submitted: 22 October 2021 · Accepted: 13 April 2022 ·

Published Online: 10 May 2022



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Note: This paper is part of the 2022 Special Topic Collection on Higher Energy X-ray Photoelectron Spectroscopy.

ABSTRACT

An Al₂O₃ sapphire sample was analyzed using high-resolution high-energy x-ray photoelectron spectroscopy (HAXPES). The HAXPES spectra of Al₂O₃ obtained using monochromatic Cr K α radiation at 5414.8 eV include two survey scans (Al K α and Cr K α) and high-resolution spectra of Al 2p, Al 2s, Al 1s, and O 1s.

Key words: Al₂O₃, HAXPES, Cr K α

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Accession #: 01740 and 01743

Technique: XPS

Host Material: Al₂O₃

Instrument: ULVAC-PHI Quantes

Major Elements in Spectra: Al, O

Minor Elements in Spectra: None

Published Spectra: 7

Spectra in Electronic Record: 7

Spectral Category: Comparison

INTRODUCTION

While high-energy photoemission has been in use for decades, only very few reference spectra are available, even for common materials. The recent availability of performant lab-scale photoemission spectrometers (Refs. 1–3) requires reliable reference data.

In this work, we present reference spectra from an Al₂O₃ sapphire sample, which was analyzed using high-resolution high-energy x-ray photoelectron spectroscopy (HAXPES). The HAXPES spectra of Al₂O₃ obtained using monochromatic Cr K α radiation at 5414.8 eV include two survey scans (Al K α and Cr K α) and high-resolution spectra of Al 2p, Al 2s, Al 1s, and O 1s.

SPECIMEN DESCRIPTION (ACCESSION # 01740)

Host Material: Al₂O₃

CAS Registry #: 1344-28-1

Host Material Characteristics: Homogeneous; solid; single crystal; dielectric; inorganic compound; other

Chemical Name: Aluminum oxide

Source: Single crystal, 2 in. c-plane sapphire substrates (0001) obtained from Roditi International Corporation Ltd.

Host Composition: Al, O

Form: Bulk

Structure: Single crystal

History and Significance: Air exposed monocrystalline and bulk 2 in. c-plane sapphire substrates (0001).

As Received Condition: Piece of a 5 in. wafer

Analyzed Region: Same as host materials

Ex Situ Preparation/Mounting: The sample was taped on the sample holder using insulating removable 3M double sided tape.

In Situ Preparation: The sample was presputtered using 100 eV Ar⁺ ions while monitoring the C 1s signal. Sputtering was stopped when the C 1s presented only background levels.

Charge Control: Low energy electrons (1 eV, filament 1.1 A) and low energy ions (10 eV, 5 mA emission)

Temp. During Analysis: 300 K

Pressure During Analysis: <5 × 10⁻⁷ Pa

Pre-analysis Beam Exposure: 0 s

SPECIMEN DESCRIPTION (ACCESSION # 01743)

Host Material: Au
CAS Registry #: 7440-57-5
Host Material Characteristics: Homogeneous; solid; amorphous; conductor; metal; other
Chemical Name: Gold
Source: 0.250 mm thick foil from Goodfellow, AU000372/5
Host Composition: Au
Form: Bulk
Structure: Amorphous
History and Significance: In-vacuum sputtered
As Received Condition: Foil
Analyzed Region: Same as host materials
Ex Situ Preparation/Mounting: The sample was taped on the sample holder using insulating removable 3M double sided tape.
In Situ Preparation: Sample was sputtered using 1 keV Ar⁺ ions until no C or O was observed.
Charge Control: Low energy electrons (1 eV, filament 1.1 A) and low energy ions (10 eV, 5 mA emission)
Temp. During Analysis: 300 K
Pressure During Analysis: <5 × 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: $A/E_p = (a^2/(a^2 + R^2))^b$, 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 [Ag 2s (3790–3830 eV), Ag 3s (700–740 eV), and Ag 3d (350–390 eV)] are recorded on a sputter-cleaned silver sample at different pass energies. The values of *a* and *b* are then determined to be 576.9 and 6.3, respectively, 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: 5417.8 eV
Source Strength: 43 W
Source Beam Size: 100 × 100 μm²
Excitation Source: Al K_α monochromatic
Source Energy: 1486.6 eV
Source Strength: 43 W
Source Beam Size: 100 × 100 μm²
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° × 20°

Ion Gun

Manufacturer and Model: ULVAC-PHI Quantes
Energy: 10 and 100 eV
Current: 5 mA
Current Measurement Method: Biased stage
Sputtering Species: Ar
Spot Size (unrastered): 10 000 μm
Raster Size: N/A
Incident Angle: 45°
Polar Angle: 45°
Azimuthal Angle: 45°
Comment: Gun used for neutralization

DATA ANALYSIS METHOD

Energy Scale Correction: Due to the too low intensity of the C 1s peak with Cr K_α, the binding energy was referenced by determining the binding energy position of the O 1s peak recorded by Al K_α XPS measurement and subsequent correction shift of the C 1s peak to 284.8 eV. The determined binding energy position of O 1s is 530.6 eV. The binding energy shift of the Cr K_α data was then performed by shifting the O 1s peak to 530.6 eV.
Recommended Energy Scale Shift: 1.15 eV
Peak Shape and Background Method: Data treatment was performed using Shirley background and Gaussian–Lorentzian peak shapes.
Quantitation Method: Quantification was done using PHI MULTIPAK Software Version 9.9.0.8. The elemental relative sensitivity factors were derived according to ISO 18118 Equation (A.7): “The pure-element relative sensitivity factor (PERSF), Si(E_p), can be obtained from measurements of Si(ref) for the selected element and a measurement of the peak intensity for the selected key material, I(key), as given in Formula (A.7): Si(E_p) = (I(ref))/(I(key)).”

AUTHOR DECLARATIONS**Conflict of Interest**

The authors have no conflicts to disclose.

DATA AVAILABILITY

The data that support the findings of this study are available within the article and its supplementary material (Ref. 4).

REFERENCES

- ¹See: <https://www.ulvac-phi.com/en/products/xps/quantes/>.
- ²See: <https://www.kratos.com/products/axis-supra-xps-surface-analysis-instrument>.
- ³See: <https://scientaomicron.com/en/system-solutions/electron-spectroscopy/HAXPES-Lab>.
- ⁴See supplementary material at <https://doi.org/10.1116/6.0001577> for ASCII data of all shown spectra.

SPECTRAL FEATURES TABLE

Spectrum ID #	Element/Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × counts/s)	Sensitivity Factor	Concentration (at. %) ^a	Peak Assignment
01740-03	Al 2p	73.8	1.58	236	0.075	36.4	Al ₂ O ₃
01740-04	Al 2s	118.6	2.17	865	0.368	35.3	Al ₂ O ₃
01740-05	Al 1s	1560.7	1.73	11 574	4.254	37.4	Al ₂ O ₃
01740-06	1s	530.6	1.66	2441	0.589	...	Al ₂ O ₃

^aThe concentration is calculated by taking into account only the O 1s peak and the Al peak from the given transition.

ANALYZER CALIBRATION TABLE^a

Spectrum ID #	Element/Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × counts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01743-01	Au 3d _{5/2}	2206.8	2.59	47 379
01743-01	Au 4f _{7/2}	84.2	1.24	2 115
...	Cu 2p _{3/2}	932.9	1.34	7 090
...	Ag 2p _{3/2}	3352.7	2.61	30 352
...	Ag 3d _{5/2}	368.4	1.15	2 978

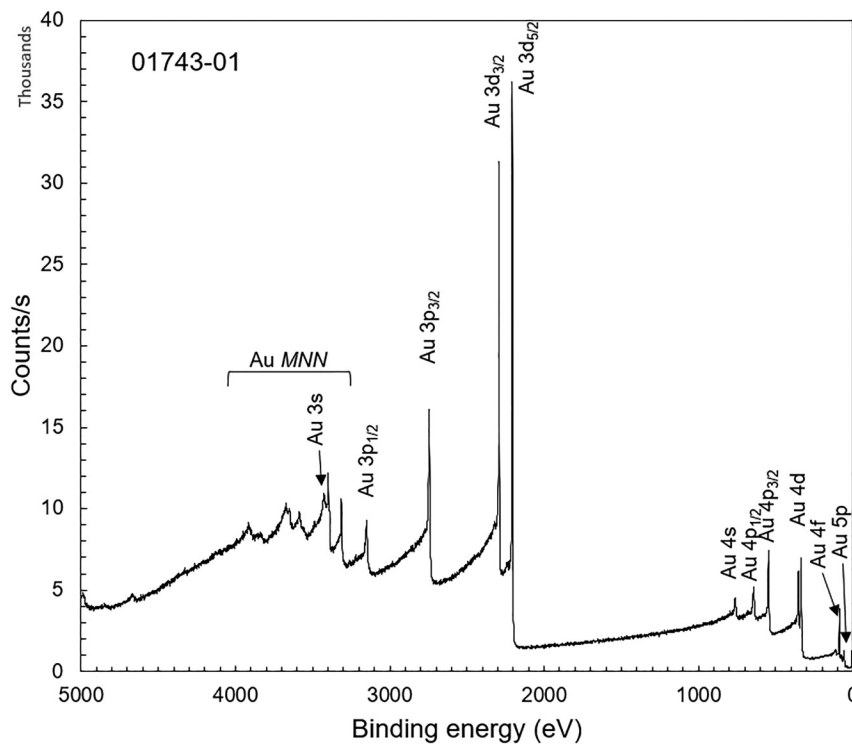
^aThe calibration table is established using the Cr K α photons and a pass energy of 112 eV corresponding to the presented high-resolution spectra.

GUIDE TO FIGURES

Spectrum (Accession) #	Spectral Region	Voltage Shift ^a	Multiplier	Baseline	Comment #
01743-01	Survey	0	1	0	Au survey with x-ray source Cr K α ^b
01740-01	Survey	0	1	0	X-ray source Al K α
01740-02	Survey	0	1	0	X-ray source Cr K α
01740-03	Al 2p	-1.15	1	0	...
01740-04	Al 2s	-1.15	1	0	...
01740-05	Al 1s	-1.15	1	0	...
01740-06	O 1s	-1.15	1	0	...

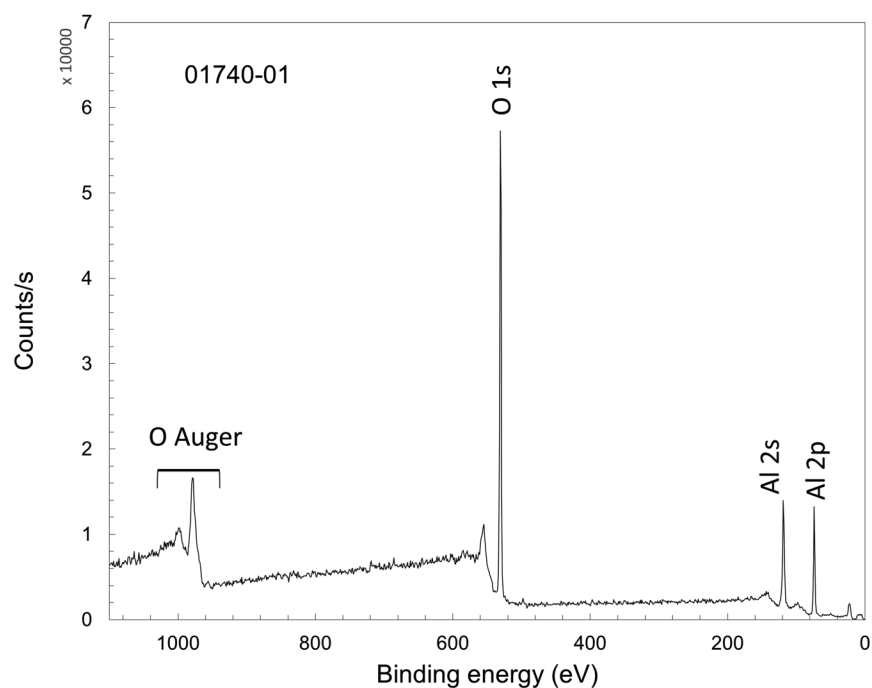
^aVoltage shift of the archived (as-measured) spectrum relative to the printed figure. The figure reflects the recommended energy scale correction due to a calibration correction, sample charging, flood gun, or other phenomenon.

^bThe reference spectrum included in this paper is the same as those included with other papers in this collection that use this x-ray source. This was intentional and not an error as submissions were requested to be accompanied by a wide-scan spectrum of sputter-cleaned gold taken on the same instrument and using the same settings as the wide-scan spectrum of the material or materials in the submission.



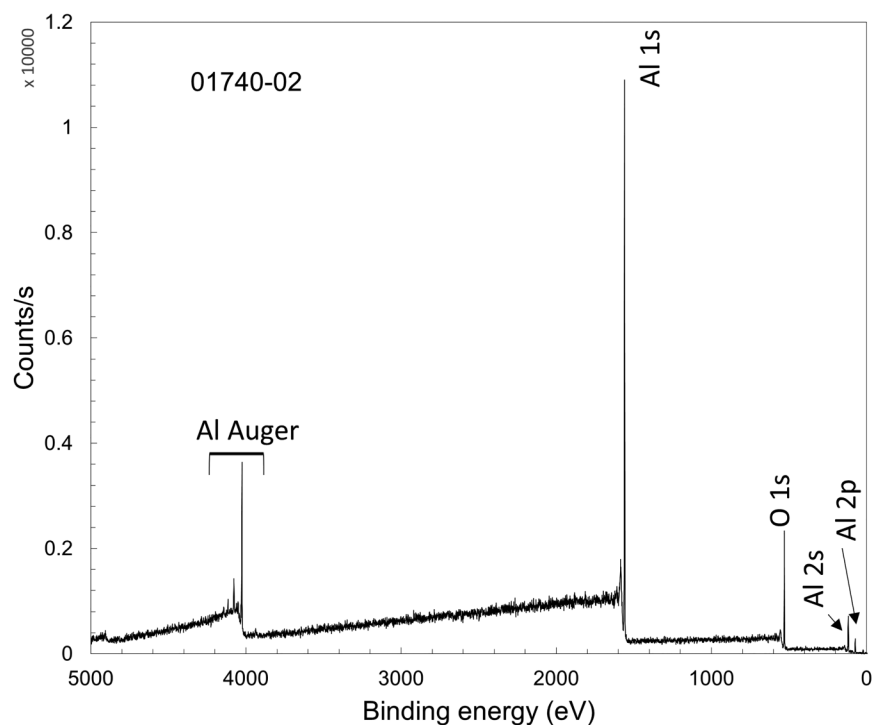
Accession #	01743-01
Host Material:	Au
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Cr K _α monochromatic
Source Energy:	5414.8 eV
Source Strength:	48 W
Source Size:	0.1 × 0.1 mm ²
Analyzer Type:	Spherical sector analyzer
Incident Angle:	22°
Emission Angle:	45°
Analyzer Pass Energy:	280 eV
Analyzer Resolution:	1.9 eV
Total Signal Accumulation Time:	5210 s
Total Elapsed Time:	5700 s
Number of Scans:	1
Effective Detector Width:	31 eV

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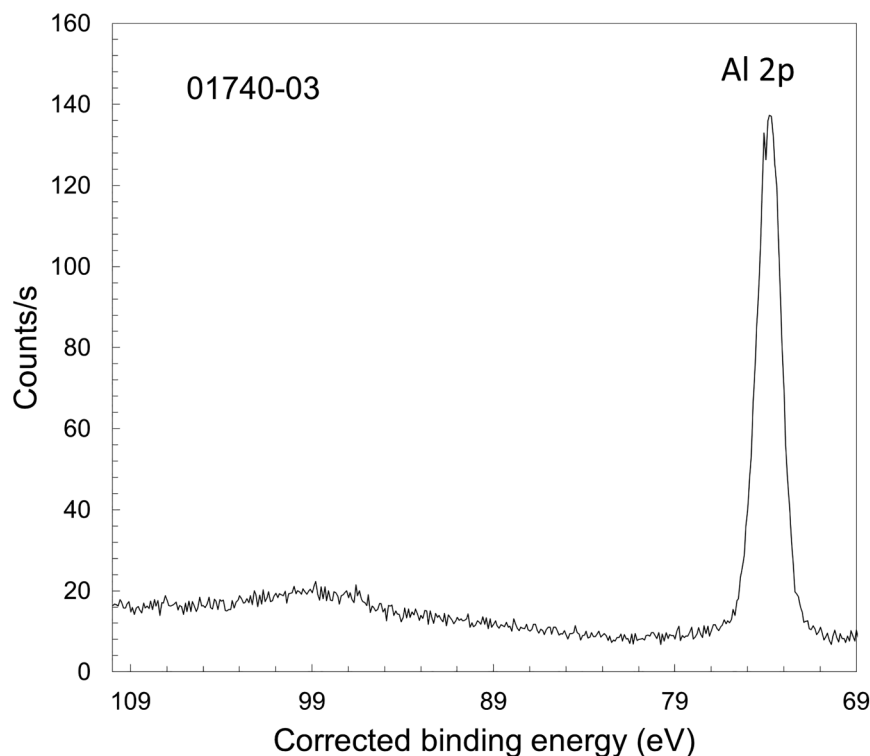
Accession #	01740-01
Host Material:	Al ₂ O ₃
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Al K _α monochromatic
Source Energy:	1486.6 eV
Source Strength:	43 W
Source Size:	0.1 × 0.1 mm ²
Analyzer Type:	Spherical sector analyzer
Incident Angle:	22°
Emission Angle:	45°
Analyzer Pass Energy:	140 eV
Analyzer Resolution:	1.9 eV
Total Signal Accumulation Time:	114 s
Total Elapsed Time:	125 s
Number of Scans:	1
Effective Detector Width:	31 eV

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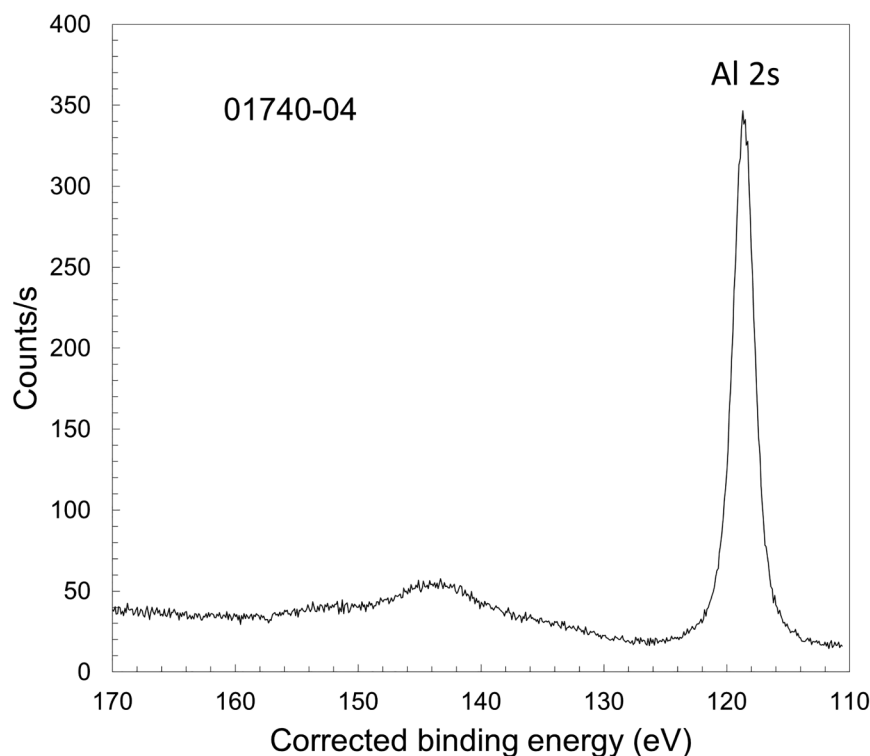
Accession #	01740-02
Host Material:	Al ₂ O ₃
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Cr K _α monochromatic
Source Energy:	5414.8 eV
Source Strength:	43 W
Source Size:	0.1 × 0.1 mm ²
Analyzer Type:	Spherical sector analyzer
Incident Angle:	22°
Emission Angle:	45°
Analyzer Pass Energy:	280 eV
Analyzer Resolution:	1.9 eV
Total Signal Accumulation Time:	1500 s
Total Elapsed Time:	1650 s
Number of Scans:	1
Effective Detector Width:	31 eV

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- Accession #: 01740-03
- Host Material: Al₂O₃
- Technique: XPS
- Spectral Region: Al 2p

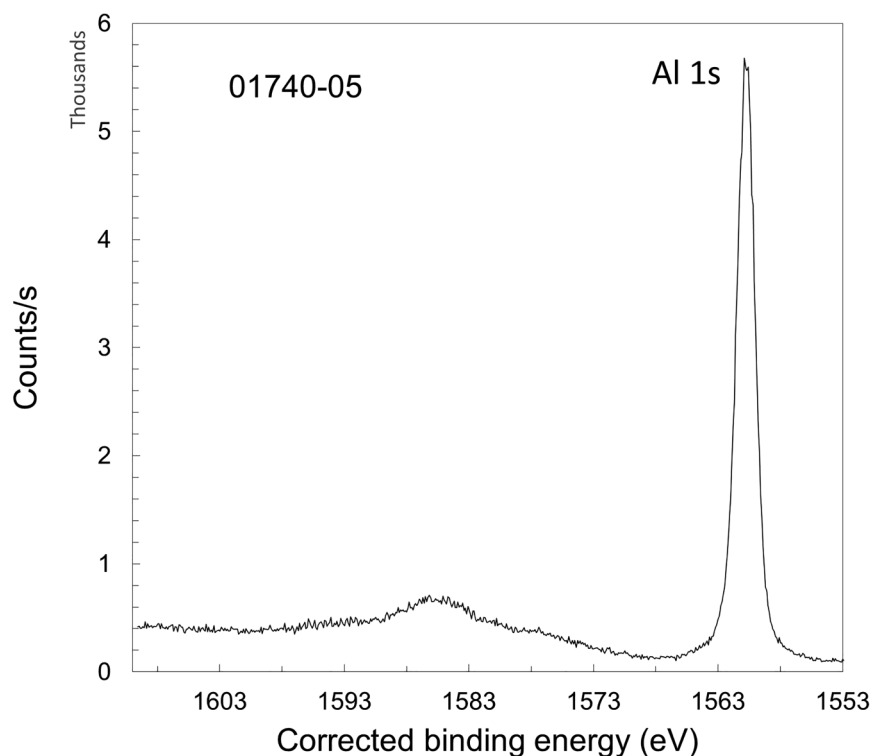
Instrument: ULVAC-PHI Quantes
 Excitation Source: Cr K_α monochromatic
 Source Energy: 5414.8 eV
 Source Strength: 43 W
 Source Size: 0.1 × 0.1 mm²
 Analyzer Type: Spherical sector
 Incident Angle: 22°
 Emission Angle: 45°
 Analyzer Pass Energy: 112 eV
 Analyzer Resolution: 0.86 eV
 Total Signal Accumulation Time: 7440 s
 Total Elapsed Time: 8180 s
 Number of Scans: 140
 Effective Detector Width: 12.4 eV



- Accession #: 01740-04
- Host Material: Al₂O₃
- Technique: XPS
- Spectral Region: Al 2s

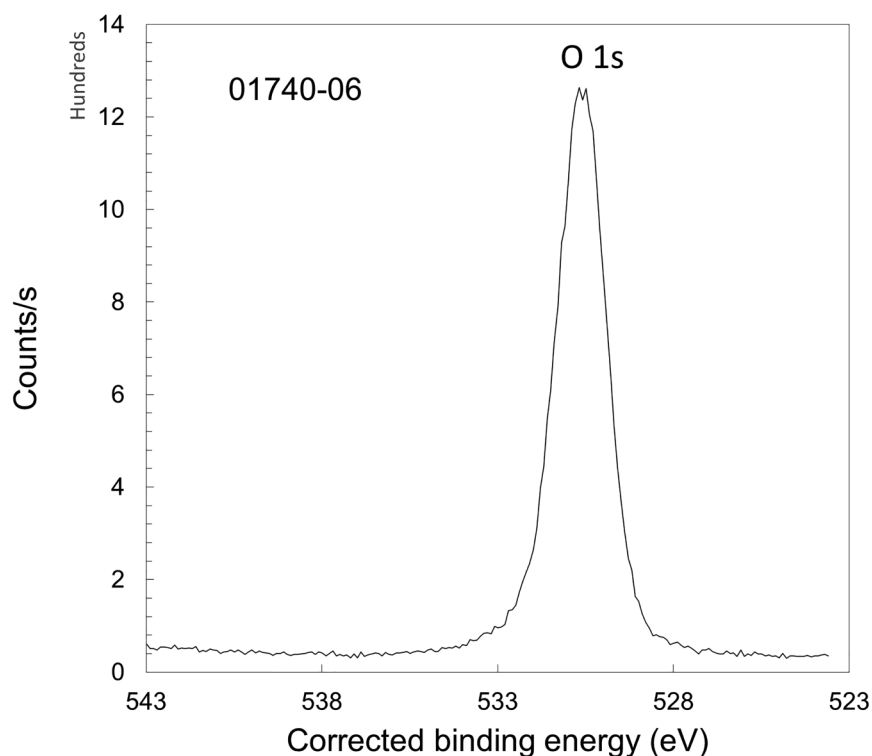
Instrument: ULVAC-PHI Quantes
 Excitation Source: Cr K_α monochromatic
 Source Energy: 5414.8 eV
 Source Strength: 43 W
 Source Size: 0.1 × 0.1 mm²
 Analyzer Type: Spherical sector
 Incident Angle: 22°
 Emission Angle: 45°
 Analyzer Pass Energy: 112 eV
 Analyzer Resolution: 0.86 eV
 Total Signal Accumulation Time: 8400 s
 Total Elapsed Time: 9250 s
 Number of Scans: 140
 Effective Detector Width: 12.4 eV

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- Accession #: [01740-05](#)
- 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: 43 W
 Source Size: 0.1 × 0.1 mm²
 Analyzer Type: Spherical sector
 Incident Angle: 22°
 Emission Angle: 45°
 Analyzer Pass Energy: 112 eV
 Analyzer Resolution: 0.86 eV
 Total Signal Accumulation Time: 480 s
 Total Elapsed Time: 520 s
 Number of Scans: 8
 Effective Detector Width: 12.4 eV



- Accession #: [01740-06](#)
- 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: 43 W
 Source Size: 0.1 × 0.1 mm²
 Analyzer Type: Spherical sector
 Incident Angle: 22°
 Emission Angle: 45°
 Analyzer Pass Energy: 112 eV
 Analyzer Resolution: 0.86 eV
 Total Signal Accumulation Time: 720 s
 Total Elapsed Time: 790 s
 Number of Scans: 36
 Effective Detector Width: 12.4 eV

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