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γ -Al₂O₃ by Ag L α , Hard X-ray Photoelectron Spectroscopy

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γ -alumina (γ -Al₂O₃) powder was characterized by hard x-ray photoelectron spectroscopy (HAXPES) with an Ag L α (2984.2 eV) excitation source. Sample was fixed to a stainless-steel sample holder with copper double-sided adhesive tape. Survey spectrum, C 1s, O 1s, Al 1s, Al KLL, Al 2s and Al 2p core levels spectra were acquired.

Keywords: Alumina; HAXPES; Ag L α excitation; γ -Al₂O₃

INTRODUCTION

Aluminum oxide (Al₂O₃) has several phases, however the most important one for catalysis is the γ -Al₂O₃, since is a useful catalyst support, due to its high surface area and high thermal stability (Ref. 1). In this work we analyzed γ -Al₂O₃ powder sample by HAXPES with a monochromatic Ag L α (2984.3 eV) excitation source. HAXPES allows to identify the inner core level of some elements that conventional XPS does not allow, which in some cases could be beneficial for the analysis, for example, when there is overlapping of signals measured by conventional XPS. With conventional radiation sources (Al K α and/or Mg K α) it is only possible to identify the Al 2s and Al 2p signals of Aluminum (Ref. 2). In this work it was possible to identify, in addition to the aforementioned signals, the inner core level Al 1s and the Al KLL Auger signals. The results showed here include survey spectrum, as well as high resolution spectra of C 1s, O 1s, Al 1s, Al KLL, Al 2s and Al 2p, which can be used for comparison.

SPECIMEN DESCRIPTION (ACCESSION # 01806)

Host Material: γ -Al₂O₃

CAS Registry #: 1344-28-1

Host Material Characteristics: homogeneous; solid; amorphous; dielectric; inorganic compound; Powder

Chemical Name: Aluminum oxide

Source: Procatalyse

Host Composition: γ -Al₂O₃

Form: Powder

Structure: Al₂O₃

History & Significance: Air exposed alumina powder. With 215 m²/g of surface area. 99.99% metal basis purity as specified by the manufacturer.

Accession#: 01806

Technique: XPS, XAES

Host Material: γ -Al₂O₃

Instrument: SPECS PHOIBOS 150

Major Elements in Spectra: O, Al

Minor Elements in Spectra: C

Published Spectra: 5

Spectra in Electronic Record: 5

Spectral Category: comparison

As Received Condition: As powder

Analyzed Region: same as host material

Ex Situ Preparation/Mounting: The sample was grounded and fixed to a sample holder with copper double sided adhesive tape.

In Situ Preparation: None

Charge Control: Electron flood gun (SPECS FG-500) operated at 96 μ A and 3.6eV

Temp. During Analysis: 295 K

Pressure During Analysis: < 1 x 10⁻⁷ Pa

Pre-analysis Beam Exposure: 60 s

INSTRUMENT DESCRIPTION

Manufacturer and Model: SPECS PHOIBOS 150 – 2D-DLD - SPECS Surface Nano Analysis GmbH

Analyzer Type: spherical sector

Detector: other

Number of Detector Elements: 25

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

■ Spectrometer

Analyzer Mode: constant pass energy

Throughput (T=E^N): N=0

Excitation Source Window: Mylar window.

Excitation Source: Ag La monochromatic

Source Energy: 2984.2 eV

Source Strength: 300 W

Source Beam Size: 2000 μ m x 2000 μ m

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Signal Mode: multichannel direct

■ Geometry

Incident Angle: 55 °

Source-to-Analyzer Angle: 55 °

Emission Angle: 0 °

Specimen Azimuthal Angle: Not applicable

Acceptance Angle from Analyzer Axis: 16 °

Analyzer Angular Acceptance Width: 16 ° x 16 °

■ Ion Gun

Manufacturer and Model: SPECS IQE 12/38

Energy: 5000 eV

Current: 70 mA

Current Measurement Method: biased stage

Sputtering Species: Ar⁺

Spot Size (unrastered): 3000 μm x 3000 μm

Raster Size: Not applicable μm x μm

Incident Angle: 54 °

Polar Angle: 55 °

Azimuthal Angle: 45 °

Comment: The specimen was analyzed as loaded. The ion gun was used only for cleaning the Ag reference foil.

DATA ANALYSIS METHOD

Energy Scale Correction: Due to the decreased photoionization cross sections in HAXPES (Refs. 3 and 4) the intensity of C 1s is very low. For this reason, the binding energy scale was corrected using the Al 2p binding energy position measured with Al K α radiation after correction with C 1s at 284.8 eV (Ref. 5). After this the binding energy of Al 2p was 74.0 eV. The spectra acquired with the Ag L α source were corrected with respect to this last value.

Recommended Energy Scale Shift: 0.75 eV

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

Quantitation Method: Peak areas were obtained from Shirley background and the relative sensitivity factors (R.S.F.) were taken from Ref. 6.

ACKNOWLEDGMENTS

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AUTHOR DECLARATIONS

Conflicts of Interest (required)

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 (Ref..6).

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7. See supplementary material at [http....](http://...) for the XPS raw data of γ -Al₂O₃. Survey spectrum, C 1s, O1s, Al 1s, Al 2s and Al 2p.

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
01806-02	C 1s	285.2	3.68	1.82x10 ²	1.00	3.79	Adventitious carbon
01806-03	O 1s	530.9	3.18	7.71x10 ³	2.93	55.72	Al ₂ O ₃
01806-04	Al 1s	1561.2	2.40	1.61x10 ⁴	11.0	40.50	Al ₂ O ₃
01806-04	Al KLL	1387.9 ^a	2.56	1.03x10 ⁴	Al ₂ O ₃
01806-05	Al 2s	118.9	2.95	1.38x10 ³	Al ₂ O ₃
01806-06	Al 2p	74.0	2.33	5.33x10 ²	Al ₂ O ₃

^a Peak position is given in kinetic energy.

ANALYZER CALIBRATION TABLE

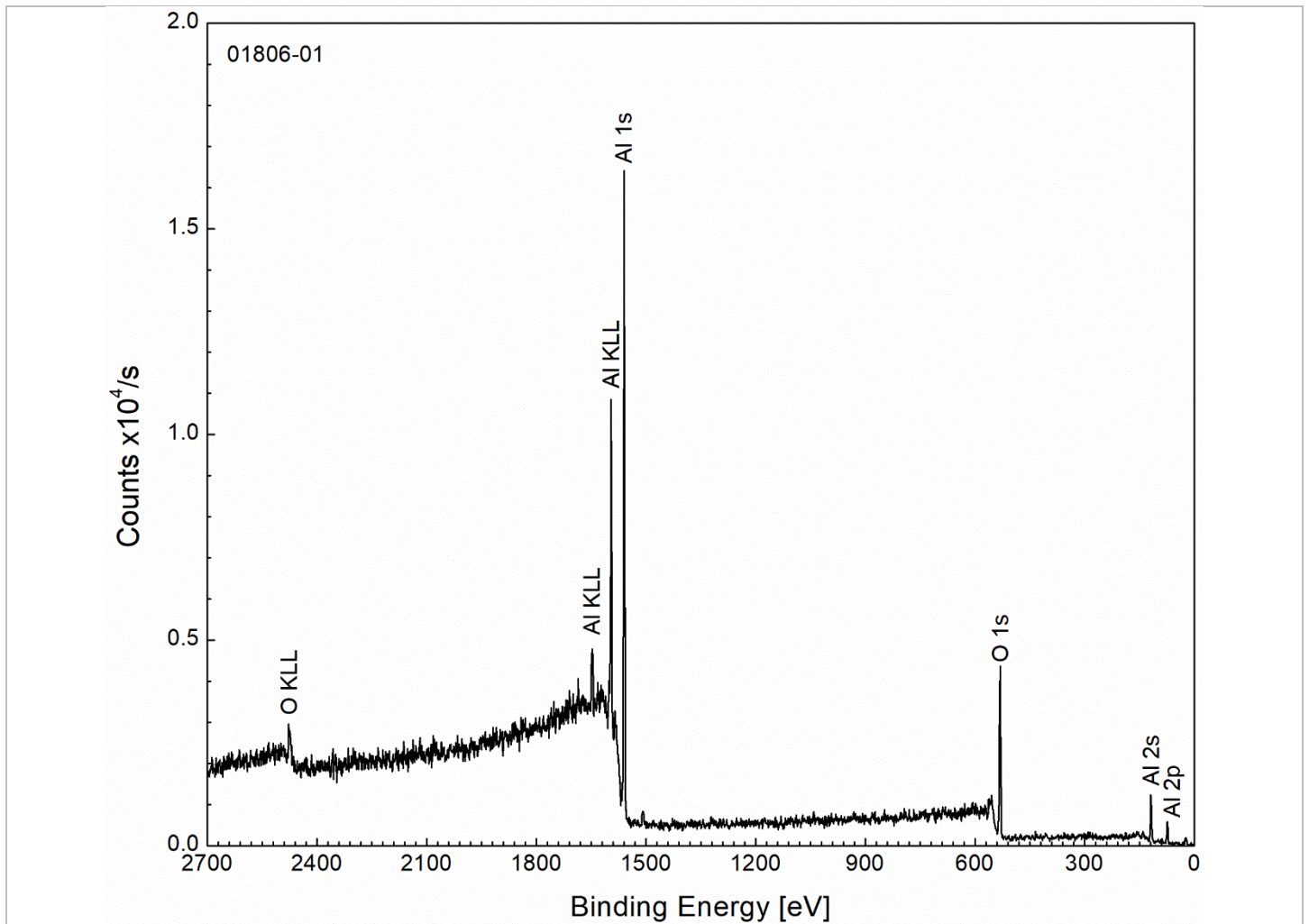
Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV x cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
...	Ag 3d _{5/2}	368.3	0.50	0.15x10 ⁶

The spectrum in the analyzer calibration table was recorded using Al K α radiation.

GUIDE TO FIGURES

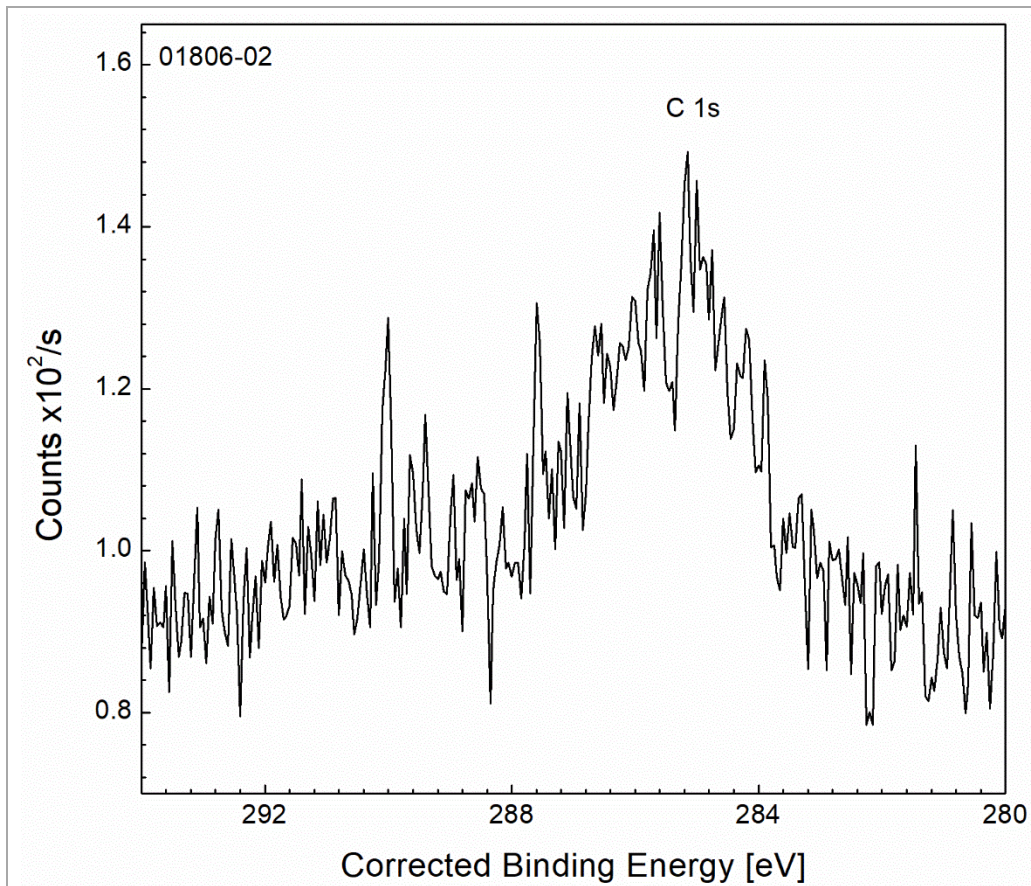
Spectrum (Accession) #	Spectral Region	Voltage Shift*	Multiplier	Baseline	Comment #
01806-01	Survey	0	1	0	-
01806-02	C 1s	-0.75	1	0	-
01806-03	O 1s	-0.75	1	0	-
01806-04	Al 1s	-0.75	1	0	-
01806-04	Al KLL	-0.75	1	0	-
01806-05	Al 2s	-0.75	1	0	-
01806-05	Al 2p	-0.75	1	0	-
01806-06	Na 1s	-0.75	1	0	-

*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.



Publish in *Surface Science Spectra*: Yes No

Accession #	01806-01
Host Material	γ -Al ₂ O ₃
Technique	XPS
Spectral Region	survey
Instrument	SPECS PHOIBOS 150
Excitation Source	Ag L α monochromatic
Source Energy	2984.2 eV
Source Strength	300 W
Source Size	2 mm x 2 mm
Analyzer Type	spherical sector analyzer
Incident Angle	55°
Emission Angle	0°
Analyzer Pass Energy	100 eV
Analyzer Resolution	3.2 eV
Total Signal Accumulation Time	270 s
Total Elapsed Time	385 s
Number of Scans	1
Effective Detector Width	5.28 eV



Publish in SSS: Yes No

■ Accession #: 01806-02

■ Host Material: γ -Al₂O₃

■ Technique: XPS

■ Spectral Region: C 1s

Instrument: SPECS PHOIBOS 150

Excitation Source: Ag L α
monochromatic

Source Energy: 2984.2 eV

Source Strength: 300 W

Source Size: 2 mm x 2 mm

Analyzer Type: spherical sector

Incident Angle: 55 °

Emission Angle: 0 °

Analyzer Pass Energy 60 eV

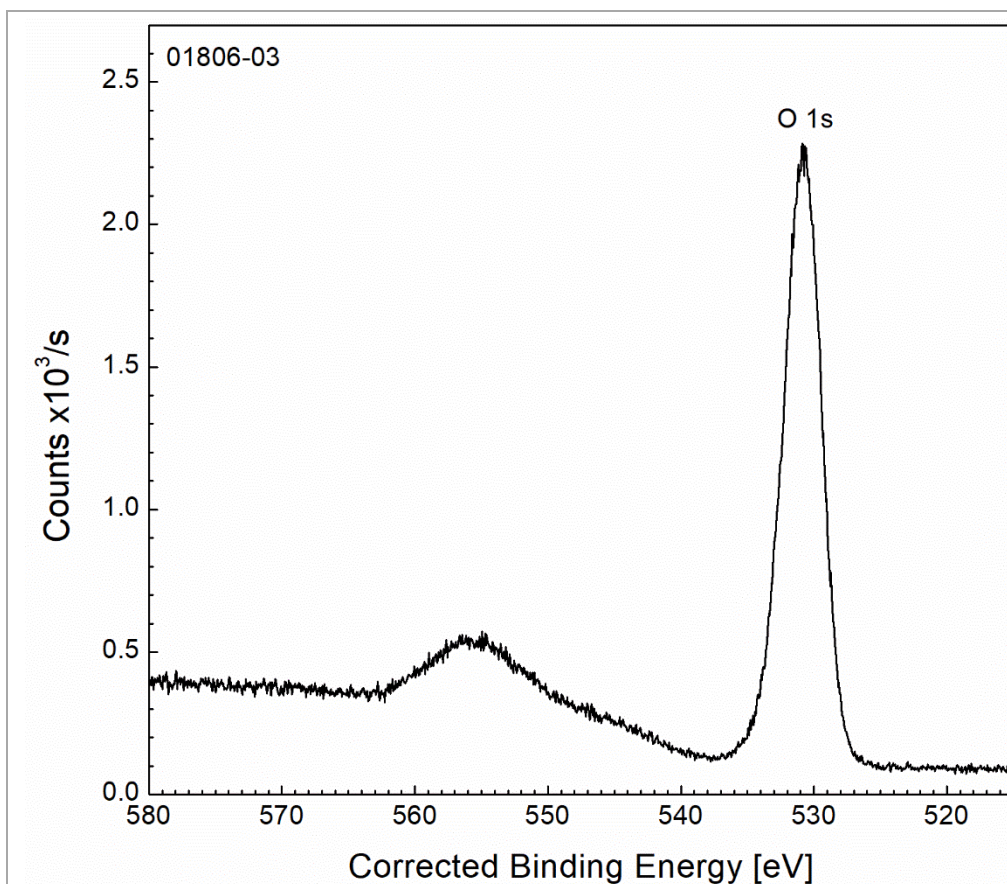
Analyzer Resolution: 1.3 eV

Total Signal Accumulation Time: 621 s

Total Elapsed Time: 993 s

Number of Scans: 16

Effective Detector Width: 2.64 eV



Publish in SSS: Yes No

■ Accession #: 01806-03

■ Host Material: γ -Al₂O₃

■ Technique: XPS

■ Spectral Region: O 1s

Instrument: SPECS PHOIBOS 150

Excitation Source: Ag L α
monochromatic

Source Energy: 2984.2 eV

Source Strength: 300 W

Source Size: 2 mm x 2 mm

Analyzer Type: spherical sector

Incident Angle: 55 °

Emission Angle: 0 °

Analyzer Pass Energy 60 eV

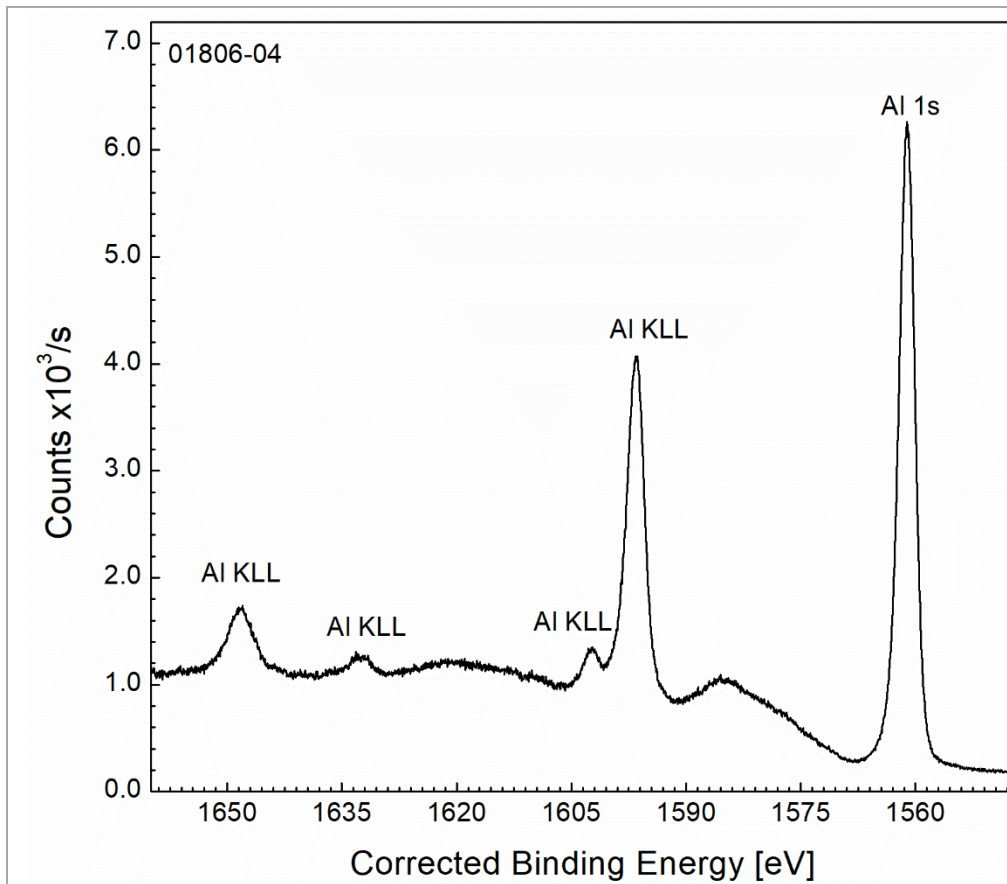
Analyzer Resolution: 1.3 eV

Total Signal Accumulation Time: 1481
s

Total Elapsed Time: 2369 s

Number of Scans: 16

Effective Detector Width: 2.64 eV



Publish in SSS: Yes No

■ Accession #: 01806-04

■ Host Material: γ -Al₂O₃

■ Technique: XPS, XAES

■ Spectral Region: Al 1s + Al KLL

Instrument: SPECS PHOIBOS 150

Excitation Source: Ag L α
monochromatic

Source Energy: 2984.2 eV

Source Strength: 300 W

Source Size: 2 mm x 2 mm

Analyzer Type: spherical sector

Incident Angle: 55 °

Emission Angle: 0 °

Analyzer Pass Energy 60 eV

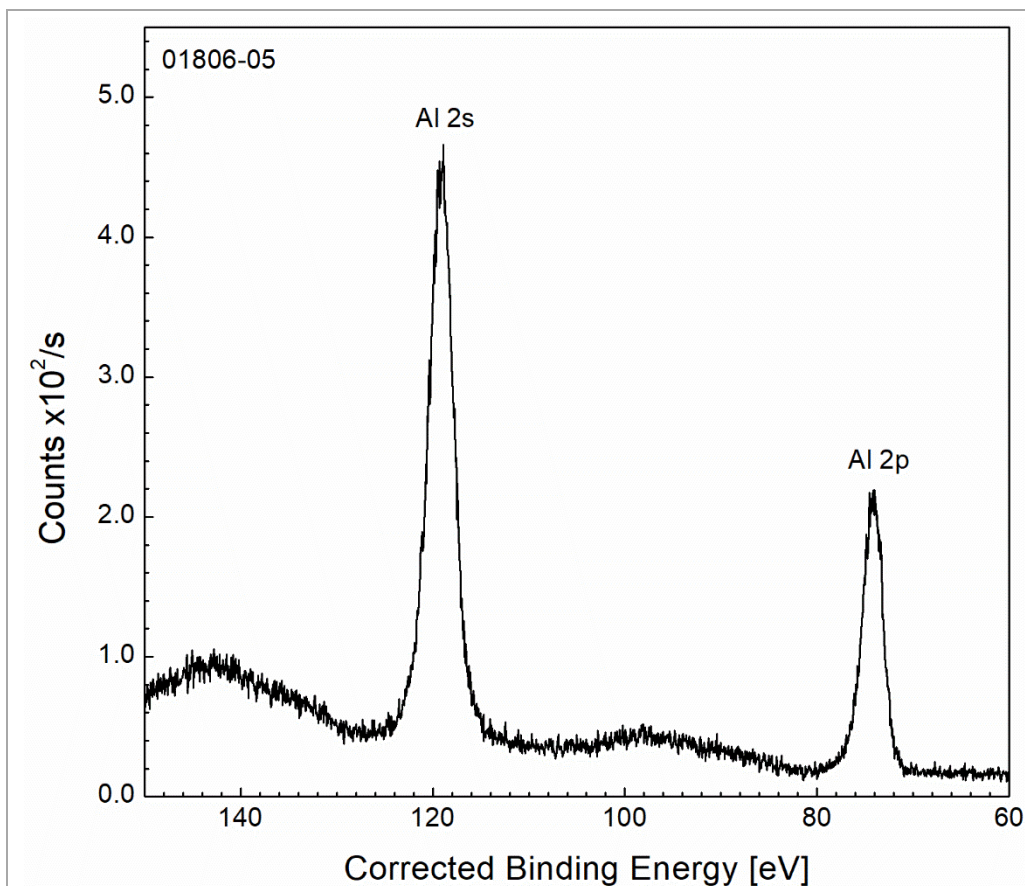
Analyzer Resolution: 1.3 eV

Total Signal Accumulation Time: 2681
s

Total Elapsed Time: 6702 s

Number of Scans: 25

Effective Detector Width: 2.64 eV



Publish in SSS: Yes No

■ Accession #: 01806-05

■ Host Material: γ -Al₂O₃

■ Technique: XPS

■ Spectral Region: Al 2s + Al 2p

Instrument: SPECS PHOIBOS 150

Excitation Source: Ag L α
monochromatic

Source Energy: 2984.2 eV

Source Strength: 300 W

Source Size: 2 mm x 2 mm

Analyzer Type: spherical sector

Incident Angle: 55 °

Emission Angle: 0 °

Analyzer Pass Energy 60 eV

Analyzer Resolution: 1.3 eV

Total Signal Accumulation Time: 1881
s

Total Elapsed Time: 3762 s

Number of Scans: 20

Effective Detector Width: 2.64 eV