HAXPES of Al with Cr Ka excitation

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Monochromatic Cr Kα radiation (5414.8 eV) was used to acquire high-energy photoelectron spectroscopy (HAXPES) data on pure Al. The reported spectra include a survey scan and high-resolution Al 1s, Al 2s, Al 2p and O 1s core-levels. The data will be useful as a comparison for the study in this field.

Keywords: Al, HAXPES, Cr Kα

Accession#: 01709

Technique: XPS

Host Material: Al

Instrument: ULVAC-PHI Quantes

Major Elements in Spectra: Al

Minor Elements in Spectra: O

Published Spectra: 4

Spectra in Electronic Record: 4

Spectral Category: comparison

INTRODUCTION

Recently developed laboratory based hard x-ray photoelectron spectrometers (HAXPES) allow to excite deeper core-levels, enabling different depth probing as a function of the studied core-level. In the case of aluminum, Cr Kα radiation (5414.8 eV) allows to ionize all Al core-levels.

In this work, we report HAXPES spectra recorded on a pure Al sample. The data include a survey scan and high-resolution spectra of Al 1s, Al 2s, Al 2p and O 1s regions.

SPECIMEN DESCRIPTION (ACCESSION # 01709)

Host Material: Al

CAS Registry #: 7429-90-5

Host Material Characteristics: Homogeneous; solid; polycrystalline; conductor; metal; Other

Chemical Name: Aluminum

Source: Goodfellow

Host Composition: Al

Form: Polycrystalline solid

Structure: Face-centered cubic

History & Significance: Air exposed thick Al metal sample

As Received Condition: Bulk Al metal

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 Ar^+ ions (2 keV) for 10 minutes prior to measurements to remove carbon and oxygen contamination.

Charge Control: Low-energy electrons (1 eV, filament 1.1 A) and low-energy Ar^+ ions (100 eV)

Temp. During Analysis: 300 K

Pressure During Analysis: $< 2.10^{-7}$ 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: $\frac{A}{E_p} = (\frac{a^2}{a^2+R^2})^b$, 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 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 °

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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: 100 and 2000 eV

Current: 0.001 mA

Current Measurement Method: Faraday cup

Sputtering Species: Ar

Spot Size (unrastered): 100 μm

Raster Size: 3000 μm x 3000 μm

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 Kα radiation after shifting the C 1s peak to 284.8 eV. Doing so, the Al 2p binding energy was 72.4 eV. The spectra recorded with the Cr Kα source were then rescaled by shifting the Al 2p to 72.4 eV.

Recommended Energy Scale Shift: 0.4 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 Kα source. They therefore account for the decrease of cross-section and different escape depth of photoelectrons using higher energy photons. Plasmon losses are not included in the derivation of RSFs which might lead to an underestimation of the Al content and, therefore, overestimate the relative concentration of surface contaminants. 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 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

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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.

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
01709-02	Al 1s	1558.4	2.00	17299	4.238	93.1	Al
01709-03	Al 2s	117.4	2.38	1345	0.369	…	Al
01709-03	Al 2p	72.4	2.02	344	0.075	…	Al
01709-04	O 1s	531.7	2.27	160	0.589	6.9	Contamination

ANALYZER CALIBRATION TABLE

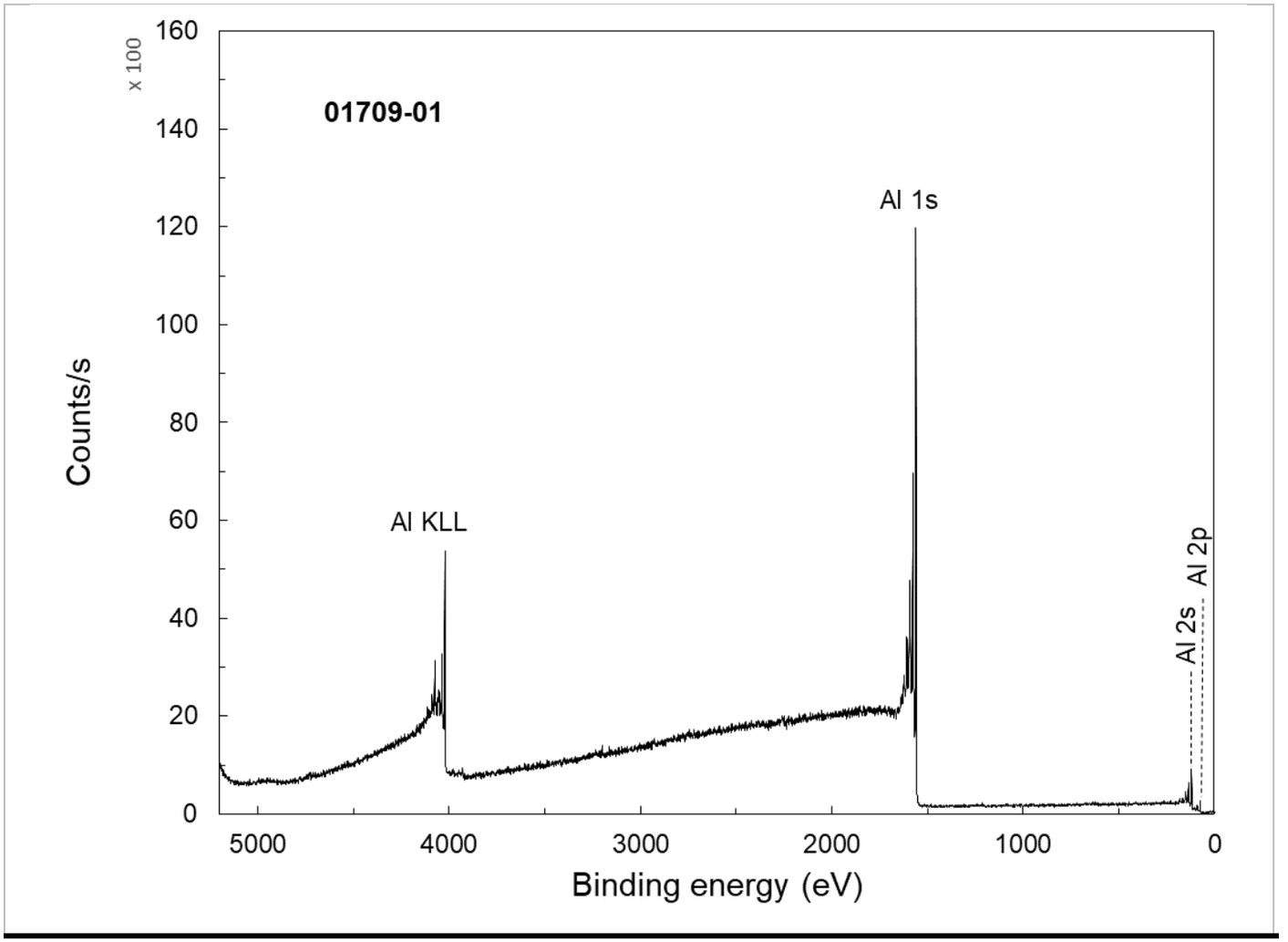
Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV x cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
...	Ag $3d_{5/2}$	368.12	0.63	114999	...	...	...
...	Cu $2p_{3/2}$	932.61	0.96	40205	...	...	...
...	Au $4f_{7/2}$	83.89	0.78	100500	...	...	...

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

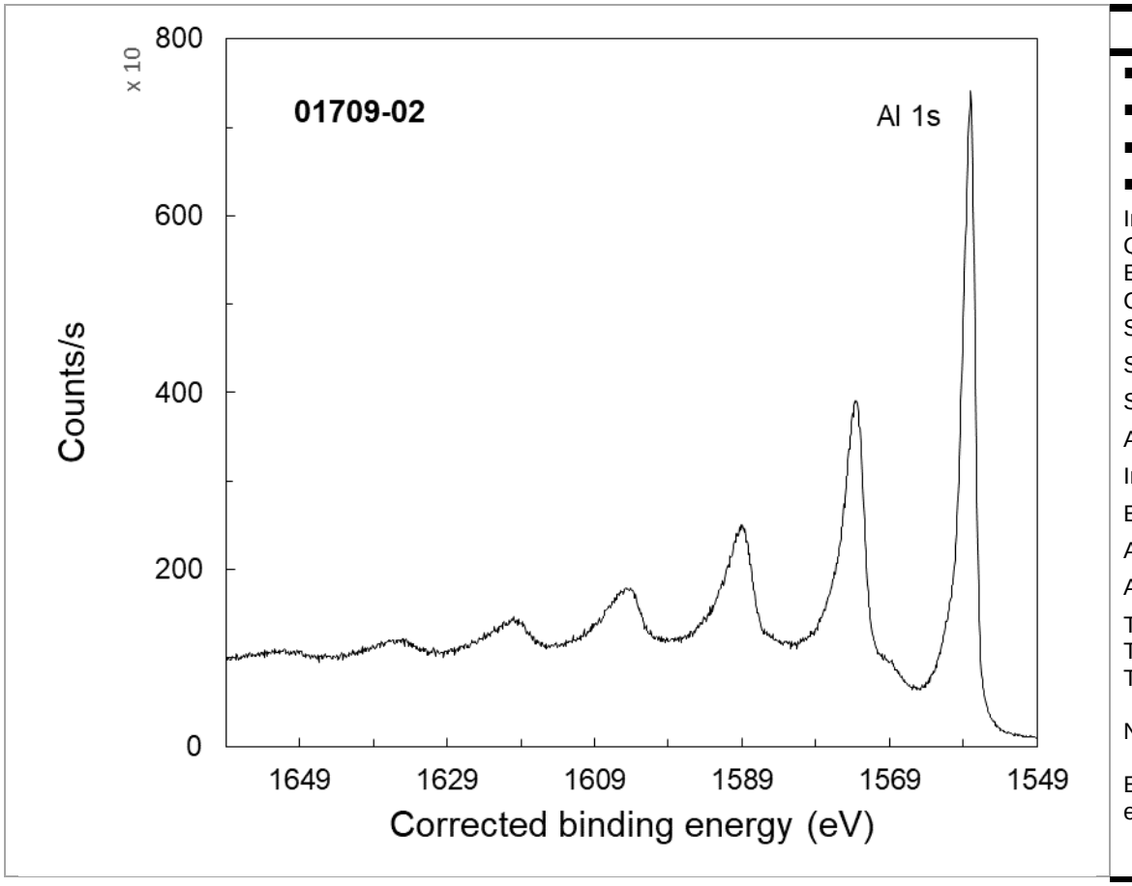
GUIDE TO FIGURES

Spectrum (Accession) #	Spectral Region	Voltage Shift*	Multiplier	Baseline	Comment #
01709-01	Survey	0	1	0	…
01709-02	Al 1s	-0.4	1	0	…
01709-03	Al 2s, Al 2p	-0.4	1	0	…
01709-04	O 1s	-0.4	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.

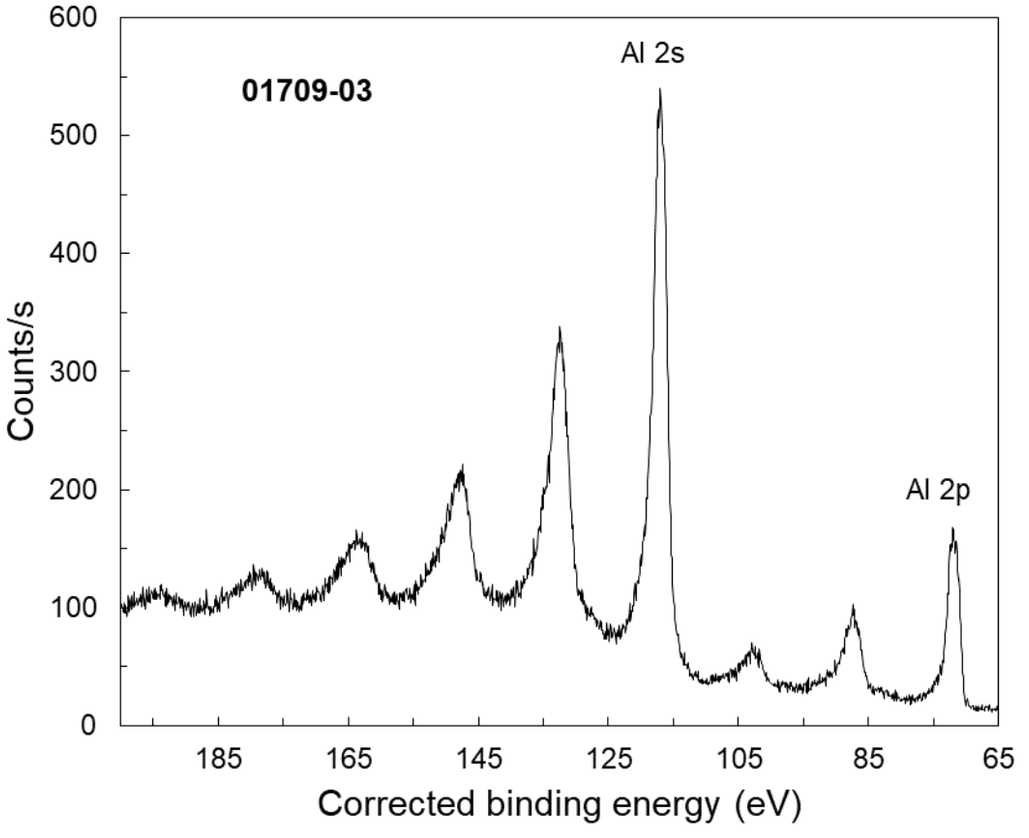


Accession #	01709-01
Host Material	Al
Technique	XPS
Spectral Region	survey
Instrument	ULVAC-PHI Quantes
Excitation Source	Cr K_α monochromatic
Source Energy	5414.8 eV
Source Strength	50 W
Source Size	0.1 mm x 0.1 mm
Analyzer Type	spherical sector analyzer
Incident Angle	22˚
Emission Angle	45˚
Analyzer 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



- **Accession #:** 01709-02
- **Host Material:** Al
- **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: 3918 s
Total Elapsed Time: 4308 s
Number of Scans: 8
Effective Detector Width: 12.4 eV



■ **Accession #:** 01709-03

■ **Host Material:** Al

■ **Technique:** XPS

■ **Spectral Region:** Al 2s, 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 °

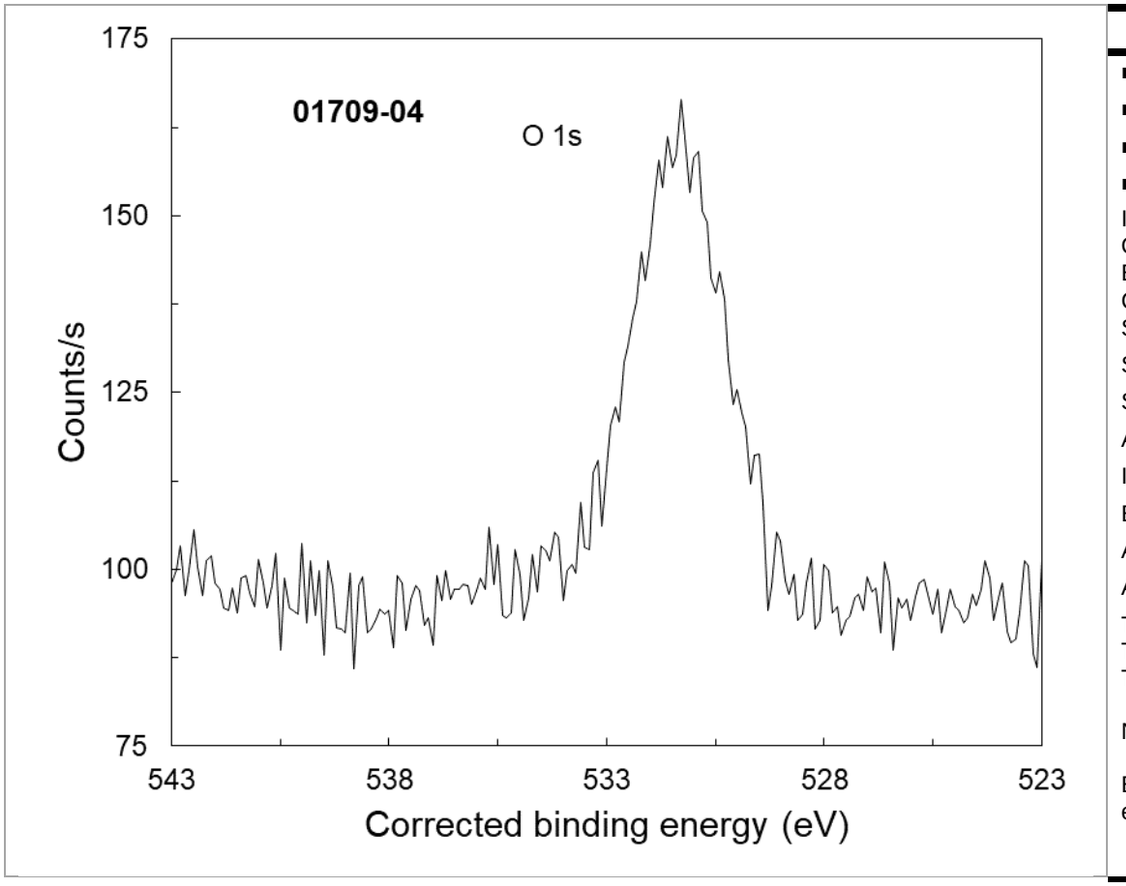
Emission Angle: 45 °

Analyzer Pass Energy 112 eV
Analyzer Resolution: 1.17 eV

Total Signal Accumulation Time: 9432 s
Total Elapsed Time: 10380 s

Number of Scans: 16

Effective Detector Width: 12.4 eV



- **Accession #:** 01709-04
- **Host Material:** Al
- **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: 2592 s
Total Elapsed Time: 2856 s

Number of Scans: 20

Effective Detector Width: 12.4 eV