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Reference survey spectra of elemental solid measured with Cr K_α photons as a tool for Quases analysis (2): Transition metals period 5 elements (Y, Zr, Nb, Mo, Ru, Rh, Pd, and Ag)

Special Collection: Higher Energy X-ray Photoelectron Spectroscopy 2022

C. Zborowski; T. Conard; A. Vanleenhove; ... et. al

Surface Science Spectra 29, 024003 (2022)
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Reference survey spectra of elemental solid measured with Cr K_α photons as a tool for Quases analysis (2): Transition metals period 5 elements (Y, Zr, Nb, Mo, Ru, Rh, Pd, and Ag)

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

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ABSTRACT

Several pure bulk materials were analyzed using laboratory-based hard x-ray photoelectron spectroscopy. The spectra are surveys measured using monochromatic Cr K_α radiation at 5414.8 eV after removal of surface contamination or oxidation. These aim to be references for inelastic background analysis using the Tougaard method.

Accession #: 01757, 01758, 01759, 01760, 01761, 01762, 01763, and 01764
Technique: XPS
Host Material: Y, Zr, Nb, Mo, Ru, Rh, Pd, and Ag
Instrument: ULVAC-PHI Quantes
Major Elements in Spectra: Y, Zr, Nb, Mo, Ru, Rh, Pd, and Ag
Minor Elements in Spectra: Ar
Published Spectra: 8
Spectral Category: Reference

INTRODUCTION

This work, similar to previous work (Refs. 1–5), aims to improve the accuracy of inelastic background analysis of XPS spectra.

The determination of the depth distribution of complex samples by inelastic background analysis of XPS spectra can be challenging if it involves different materials with widely different inelastic scattering cross sections and inelastic mean free paths.

However, it has been shown that the use of reference spectra to adjust the fit of the inelastic background (Ref. 1) with Quases-Analyze software (Ref. 6) significantly improves the accuracy of the depth distribution determined with the Tougaard method (Refs. 7 and 8).

With the development of the laboratory based HAXPES tools, the HAXPES technique is now easily accessible. The probing depth with the Tougaard method is ∼8 IMFP (Ref. 8) which is larger than the usual ∼3 IMFP which is the quoted value for classical XPS core-level peak analysis. In some typical HAXPES cases, the probing depth even exceeds 10 IMFP and structures at ∼50 nm or in some cases even more than 100 nm depths have been studied (Refs. 1–5 and 9–12).

That is the reason why these new laboratory based HAXPES tools make the inelastic background analysis even more useful to determine the depth distribution. The reference measurements of pure bulk samples are needed to improve accuracy of the depth distribution determination (Ref. 1). These survey measurements have been done after soft cleaning of the sample surface with Ar monoatomic sputtering until oxygen and carbon peaks are removed from the spectra recorded with the Al K_α source.

SPECIMEN DESCRIPTION (ACCESSION # 01757, 01758, 01759, 01760, 01761, 01762, 01763, AND 01764)

Host Material: Yttrium: 01757; Zirconium: 01758; Niobium: 01759; Molibdenum: 01760; Ruthenium: 01761; Rhodium: 01762; Paladium: 01763; and Silver: 01764
CAS Registry #: See Guide to Figures
Host Material Characteristics: Homogeneous; solid; polycrystalline; unknown conductivity; inorganic compound; and Other
Chemical Name: Same as Host Material
Source: Bulk samples
Host Composition: Y, Zr, Nb, Mo, Ru, Rh, Pd, and Ag
Form: Foil
Structure: Polycrystalline
History and Significance: Air exposed and Ar-sputtered
As Received Condition: Foil
Analyzed Region: Same as host materials
***Ex Situ* Preparation/Mounting:** Sample was taped on the sample holder using removable 3M double sided tape.
***In Situ* Preparation:** Monoatomic Ar^+ sputter clean until oxygen and carbon peaks are removed from the spectra recorded with the Al K_α source (standard cleaning 1 keV (lower for sensitive materials), sputter time dependent on surface contamination level).
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: $<5 \times 10^{-7}$ Pa
Preanalysis 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 [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: 5414.8 eV
Source Strength: 100 W
Source Beam Size: $100 \times 1400\,\mu m^2$
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
Energy: 10 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 charge neutralization

DATA ANALYSIS METHOD

Energy Scale Correction: For each spectrum, the mathematical average position of the "main" elemental peak as available in the NIST database (Ref. 13) was determined and the spectra were aligned on that specific peak. The peak selected for the energy calibration corresponds to the most often measured peak (Ref. 14).
Recommended Energy Scale Shift: See "guide to figure" table.
Peak Shape and Background Method: None
Quantitation Method: None

AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

C. Zborowski: Data curation (lead); Software (equal); Writing – review & editing (equal). **T. Conard:** Data curation (equal); Software (equal); Writing – original draft (lead). **A. Vanleenhove:** Writing – review & editing (lead). **I. Hoflijk:** Writing – review & editing (equal). **I. Vaesen:** Writing – review & editing (equal).

DATA AVAILABILITY

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

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[14]See the supplementary material at https://doi.org./10.1116/6.0001953 for ASCII data of all shown spectra.

SPECTRAL FEATURES TABLE[a]

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × counts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01757-01	Y 3d	155.7[b]	…	…	…	…	…
	Y $3p_{3/2}$	298.9	…	…	…	…	…
	Y $3p_{1/2}$	310.7	…	…	…	…	…
	Y 3s	393.6	…	…	…	…	…
	Y $2p_{3/2}$	2078.5	…	…	…	…	…
	Y $2p_{1/2}$	2153.5	…	…	…	…	…
	Y 2s	2371.4	…	…	…	…	…
	Y LMM	1743.5[c]	…	…	…	…	…
01758-01	Zr 4p	27.8	…	…	…	…	…
	Zr 4s	50.5	…	…	…	…	…
	Zr $3d_{5/2}$	178.9[b]	…	…	…	…	…
	Zr $3d_{3/2}$	180.9	…	…	…	…	…
	Zr $3p_{3/2}$	329.7	…	…	…	…	…
	Zr $3p_{1/2}$	343.4	…	…	…	…	…
	Zr 3s	429.7	…	…	…	…	…
	Zr $2p_{3/2}$	2221.4	…	…	…	…	…
	Zr $2p_{1/2}$	2305.7	…	…	…	…	…
	Zr 2s	2531.0	…	…	…	…	…
	Zr LMM	1839.3[c]	…	…	…	…	…
01759-01	Nb 4p	31.2	…	…	…	…	…
	Nb 4s	56.0	…	…	…	…	…
	Nb $3d_{5/2}$	202.2[b]	…	…	…	…	…
	Nb $3d_{3/2}$	204.7	…	…	…	…	…
	Nb $3p_{3/2}$	360.5	…	…	…	…	…
	Nb $3p_{1/2}$	376.1	…	…	…	…	…
	Nb 3s	466.3	…	…	…	…	…
	Nb $2p_{3/2}$	2368.3	…	…	…	…	…
	Nb $2p_{1/2}$	2462.5	…	…	…	…	…
	Nb 2s	2695.5	…	…	…	…	…
	Nb LMM	1938.2[c]	…	…	…	…	…
01760-01	Mo 4p	35.8	…	…	…	…	…
	Mo 4s	62.8	…	…	…	…	…
	Mo $3d_{5/2}$	227.8[b]	…	…	…	…	…
	Mo $3d_{3/2}$	230.9	…	…	…	…	…
	Mo $3p_{3/2}$	393.8	…	…	…	…	…
	Mo $3p_{1/2}$	411.5	…	…	…	…	…
	Mo 3s	505.8	…	…	…	…	…
	Mo $2p_{3/2}$	2521.0	…	…	…	…	…
	Mo $2p_{1/2}$	2625.8	…	…	…	…	…
	Mo 2s	2867.0	…	…	…	…	…
	Mo LMM	2039.0[c]	…	…	…	…	…
01761-01	Ru 4p	43.8	…	…	…	…	…
	Ru 4s	75.4	…	…	…	…	…
	Ru $3d_{5/2}$	280.0[b]	…	…	…	…	…
	Ru $3d_{3/2}$	284.1	…	…	…	…	…
	Ru $3p_{3/2}$	461.5	…	…	…	…	…
	Ru $3p_{1/2}$	483.8	…	…	…	…	…
	Ru 3s	586.2	…	…	…	…	…
	Ru $2p_{3/2}$	2838.8	…	…	…	…	…
	Ru $2p_{1/2}$	2967.7	…	…	…	…	…
	Ru 2s	3225.2	…	…	…	…	…
	Ru LMM	2249.7[c]	…	…	…	…	…

SPECTRAL FEATURES TABLE[a] (*Continued.*)

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × counts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01762-01	Rh 4p	47.8	…	…	…	…	…
	Rh 4s	81.3	…	…	…	…	…
	Rh $3d_{5/2}$	307.1[b]	…	…	…	…	…
	Rh $3d_{3/2}$	311.9	…	…	…	…	…
	Rh $3p_{3/2}$	496.3	…	…	…	…	…
	Rh $3p_{1/2}$	521.3	…	…	…	…	…
	Rh 3s	628.5	…	…	…	…	…
	Rh $2p_{3/2}$	3004.1	…	…	…	…	…
	Rh $2p_{1/2}$	3146.4	…	…	…	…	…
	Rh 2s	3412.0	…	…	…	…	…
	Rh LMM	2359.5[c]	…	…	…	…	…
01763-01	Pd 4p	52.9	…	…	…	…	…
	Pd 4s	88.2	…	…	…	…	…
	Pd $3d_{5/2}$	335.5[b]	…	…	…	…	…
	Pd $3d_{3/2}$	340.8	…	…	…	…	…
	Pd $3p_{3/2}$	532.6	…	…	…	…	…
	Pd $3p_{1/2}$	560.3	…	…	…	…	…
	Pd 3s	672.0	…	…	…	…	…
	Pd $2p_{3/2}$	3174.3	…	…	…	…	…
	Pd $2p_{1/2}$	3331.2	…	…	…	…	…
	Pd 2s	3605.3	…	…	…	…	…
	Pd LMM	2469.2[c]	…	…	…	…	…
01764-01	Ag 4p	60.4	…	…	…	…	…
	Ag 4s	97.5	…	…	…	…	…
	Ag $3d_{5/2}$	368.2[b]	…	…	…	…	…
	Ag $3d_{3/2}$	374.2	…	…	…	…	…
	Ag $3p_{3/2}$	573.2	…	…	…	…	…
	Ag $3p_{1/2}$	604.0	…	…	…	…	…
	Ag 3s	719.4	…	…	…	…	…
	Ag $2p_{3/2}$	3352.6	…	…	…	…	…
	Ag $2p_{1/2}$	3525.3	…	…	…	…	…
	Ag 2s	3807.6	…	…	…	…	…
	Ag LMM	2577.2[c]	…	…	…	…	…

[a]Applicable to all peak energy values: Peak energies were determined from the centroid of the peak. Due to the 0.5 eV data point spacing, they are reported to 0.3 eV precision.
[b]Peak used for binding energy referencing.
[c]Peak position is given in kinetic energy.

ANALYZER CALIBRATION TABLE

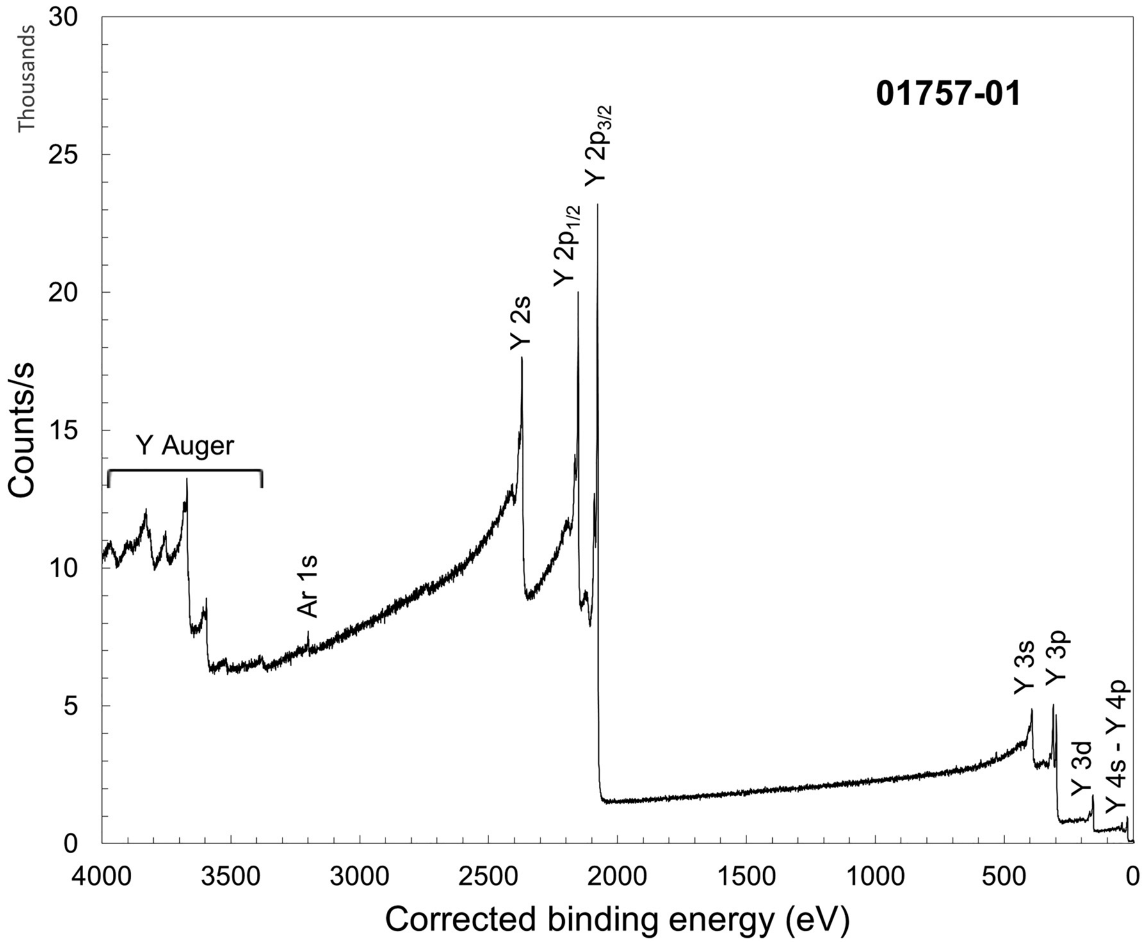
Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × counts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
…	Cu $2p_{3/2}$	932.66	0.88	79 646	…	…	…
…	Ag $3d_{5/2}$	368.25	0.63	79 262	…	…	…
…	Au $4f_{7/2}$	84.02	0.73	35 042	…	…	…

The calibration table is established using the Cr K_{α} photons and a pass energy of 112 eV (analyzer resolution 0.86 eV).

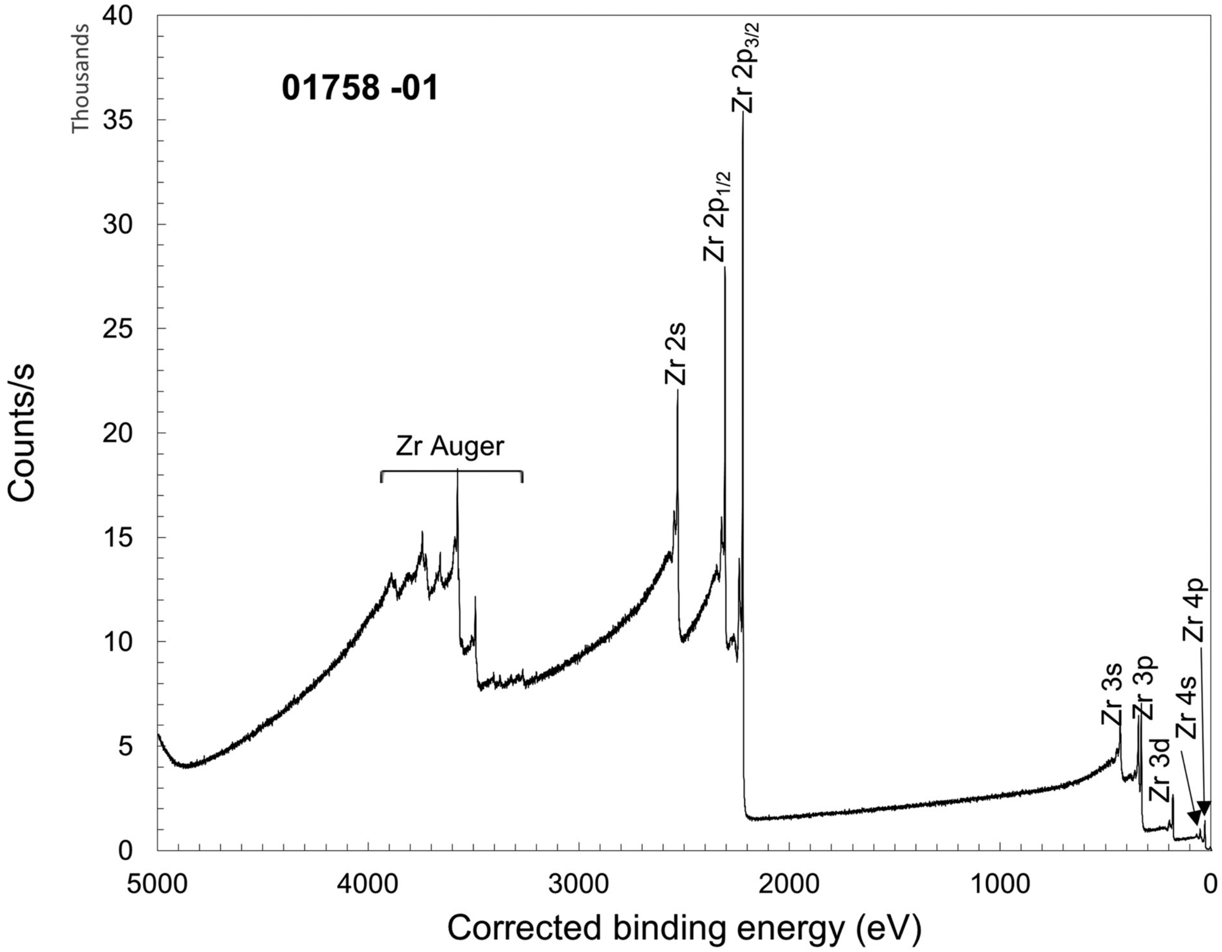
GUIDE TO FIGURES

Spectrum (Accession) #	Spectral Region	Voltage Shift[a]	Multiplier	Baseline	Comment #
01757-01	Survey	−1.1	1	0	Y: CAS 7440-65-5; Goodfellow: 913-774-78
01758-01	Survey	−1.2	1	0	Zr: CAS 7440-67-7; Goodfellow:157-925-54
01759-01	Survey	−1.1	1	0	Nb: CAS 7440-03-1; Goodfellow: 122-201-42
01760-01	Survey	−1.0	1	0	Mo: CAS 7439-98-7; Goodfellow: 278-943-70
01761-01	Survey	−0.9	1	0	Ru: CAS 7440-18-8; Goodfellow: 407-382-60
01762-01	Survey	−1.5	1	0	Rh: CAS 7440-16-6; Goodfellow: 213-336-42
01763-01	Survey	−0.8	1	0	Pd: CAS 7440-05-3; Goodfellow 671-051-11
01764-01	Survey	−0.8	1	0	Ag: CAS 7440-22-4; Goodfellow: 299-906-61

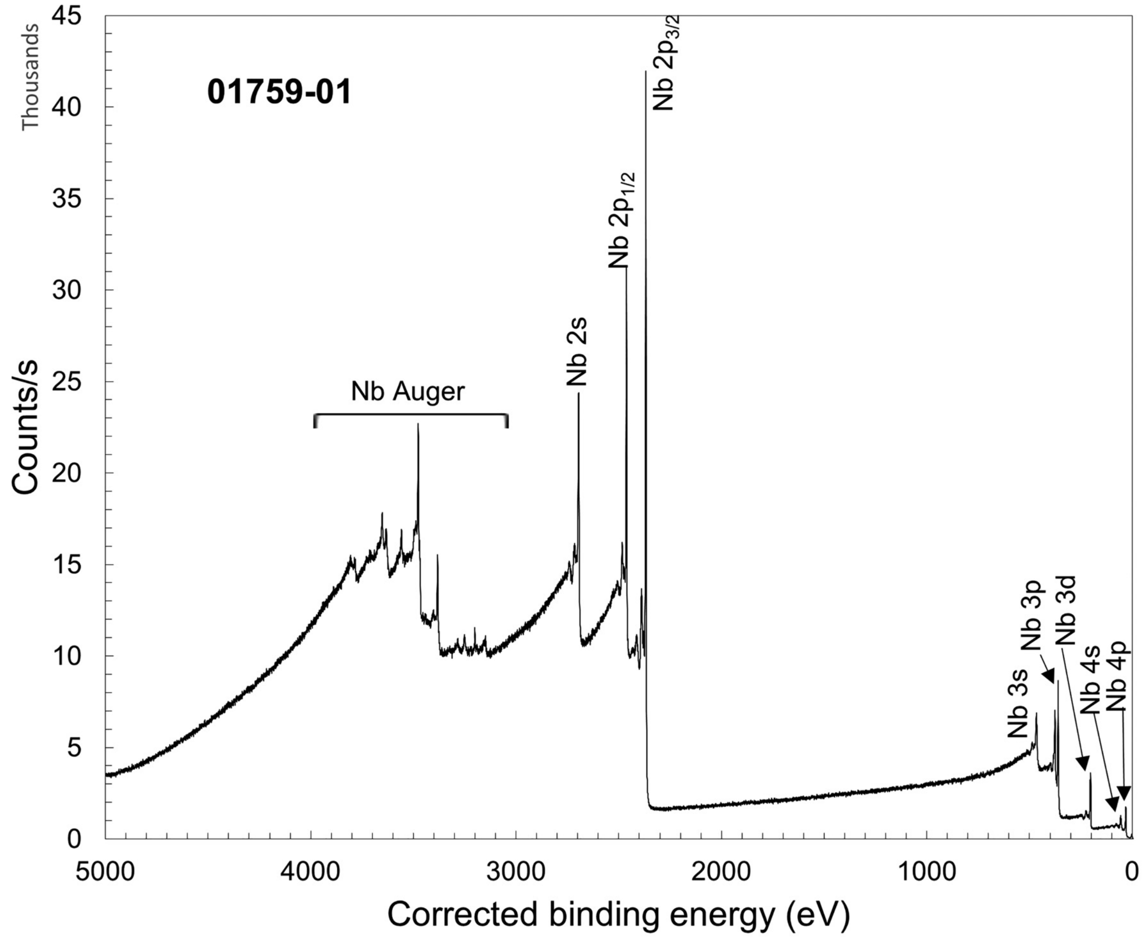
[a]Voltage shift of the archived (as-measured) spectrum relative to the printed figure. The figure reflects the recommended energy scale correction due to a calibration correction, sample charging, flood gun, or other phenomenon.



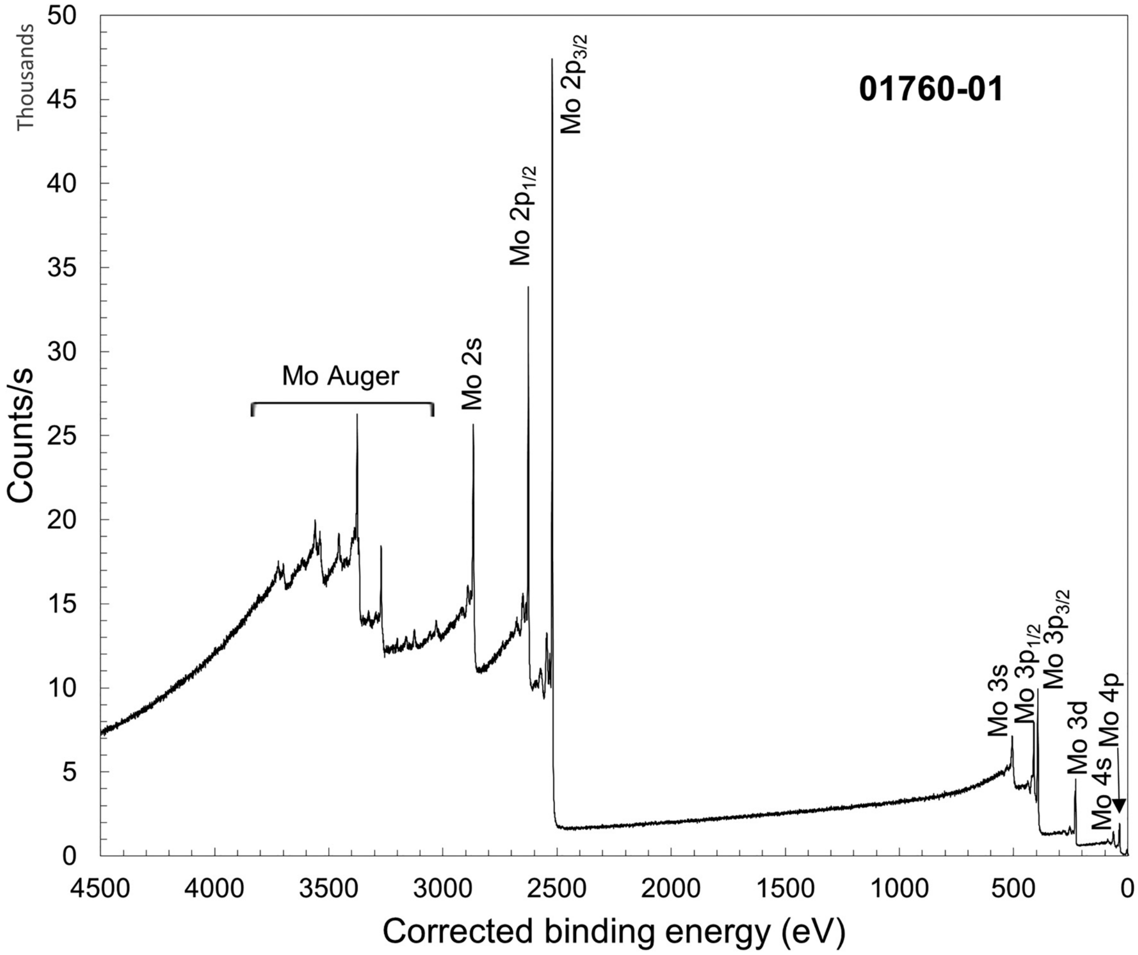
Accession #	01757-01
Host Material	Y
Technique	XPS
Spectral Region	Survey
Instrument	ULVAC-PHI Quantes
Excitation Source	Cr K_α monochromatic
Source Energy	5414.8 eV
Source Strength	100 W
Source Size	0.1 × 1.4 mm^2
Analyzer Type	Spherical sector analyzer
Incident Angle	22°
Emission Angle	45°
Analyzer Pass Energy	280 eV
Analyzer Resolution	1.9 eV
Total Signal Accumulation Time	10 000 s
Total Elapsed Time	11 300 s
Number of Scans	10
Effective Detector Width	31 eV



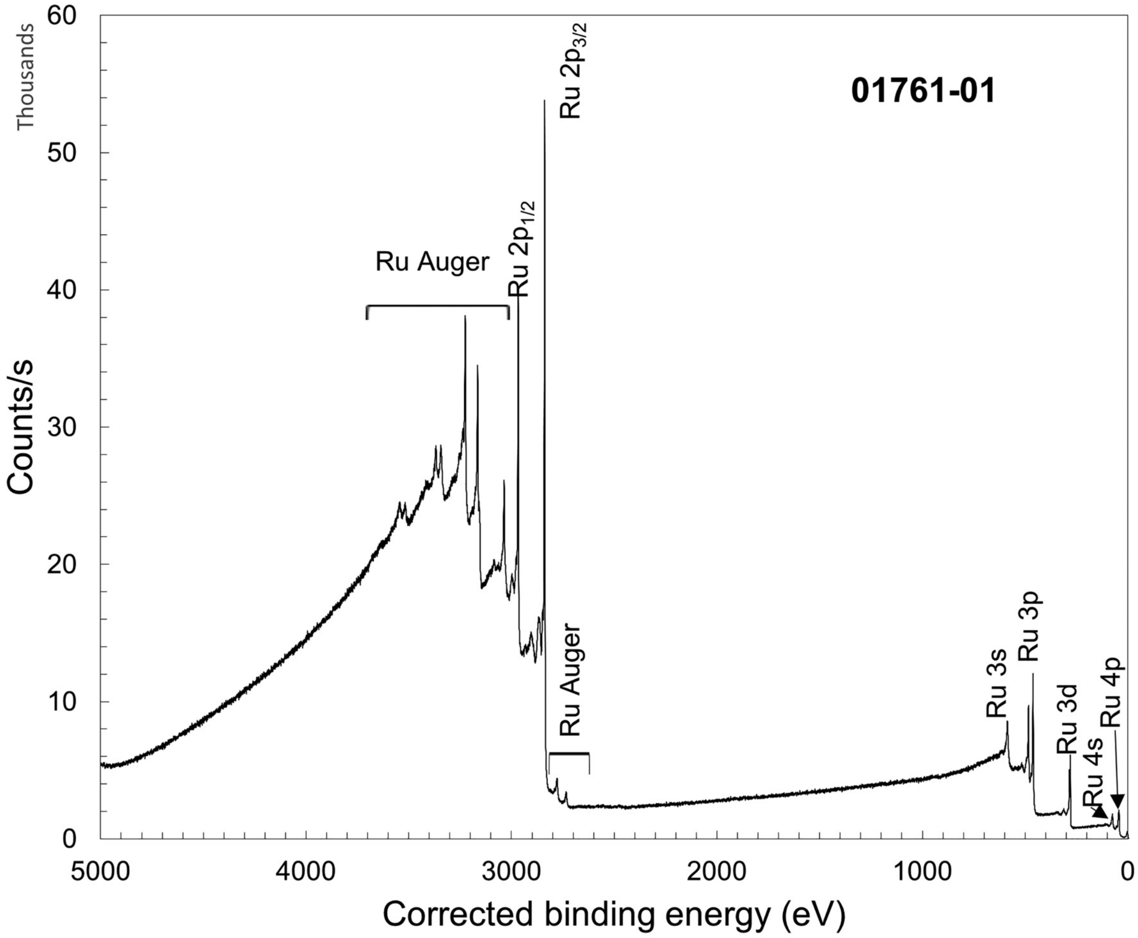
Accession #	01758-01
Host Material	Zr
Technique	XPS
Spectral Region	Survey
Instrument	ULVAC-PHI Quantes
Excitation Source	Cr K_α monochromatic
Source Energy	5414.8 eV
Source Strength	100 W
Source Size	0.1 × 1.4 mm^2
Analyzer Type	Spherical sector analyzer
Incident Angle	22°
Emission Angle	45°
Analyzer Pass Energy	280 eV
Analyzer Resolution	1.9 eV
Total Signal Accumulation Time	10 000 s
Total Elapsed Time	11 300 s
Number of Scans	10
Effective Detector Width	31 eV



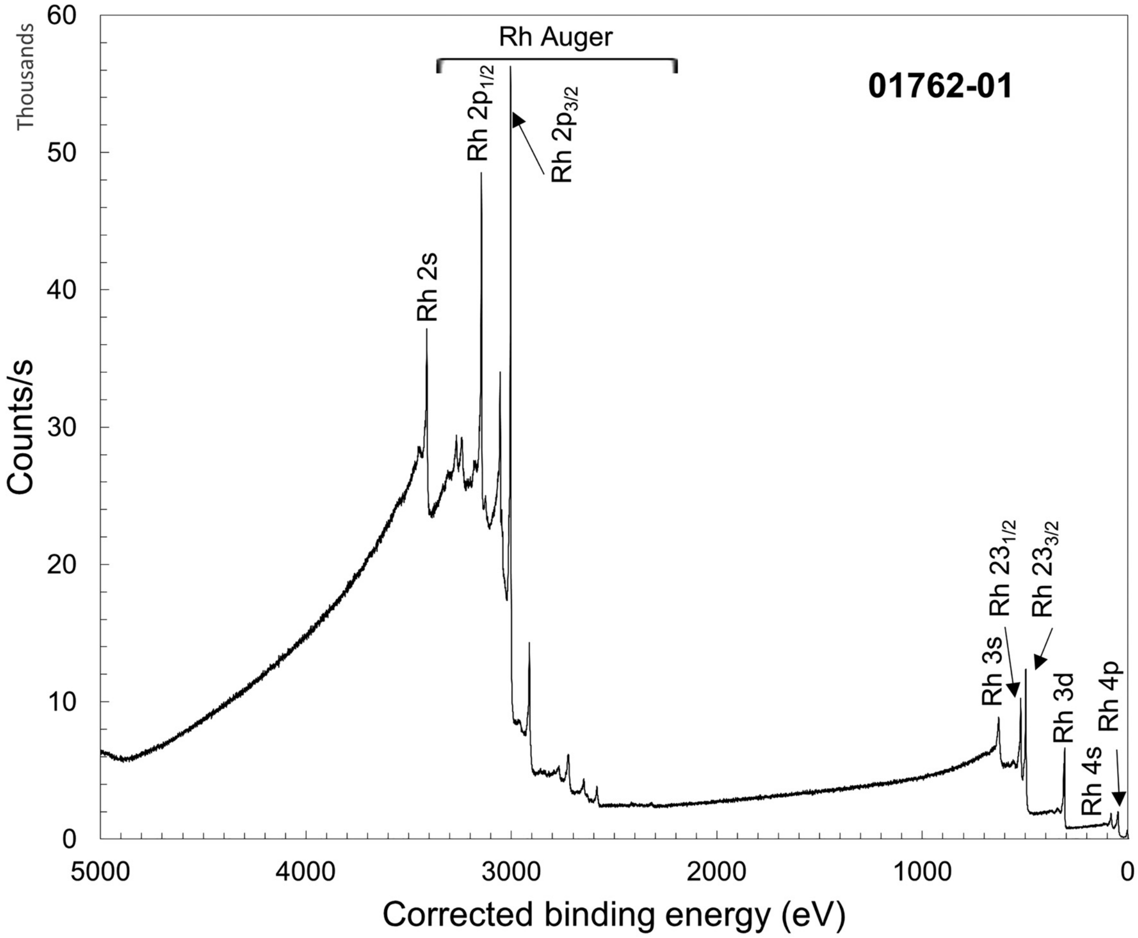
Accession #	01759-01
Host Material	Nb
Technique	XPS
Spectral Region	Survey
Instrument	ULVAC-PHI Quantes
Excitation Source	Cr K_α monochromatic
Source Energy	5414.8 eV
Source Strength	100 W
Source Size	0.1 × 1.4 mm^2
Analyzer Type	Spherical sector analyzer
Incident Angle	22°
Emission Angle	45°
Analyzer Pass Energy	280 eV
Analyzer Resolution	1.9 eV
Total Signal Accumulation Time	10 000 s
Total Elapsed Time	11 300 s
Number of Scans	10
Effective Detector Width	31 eV



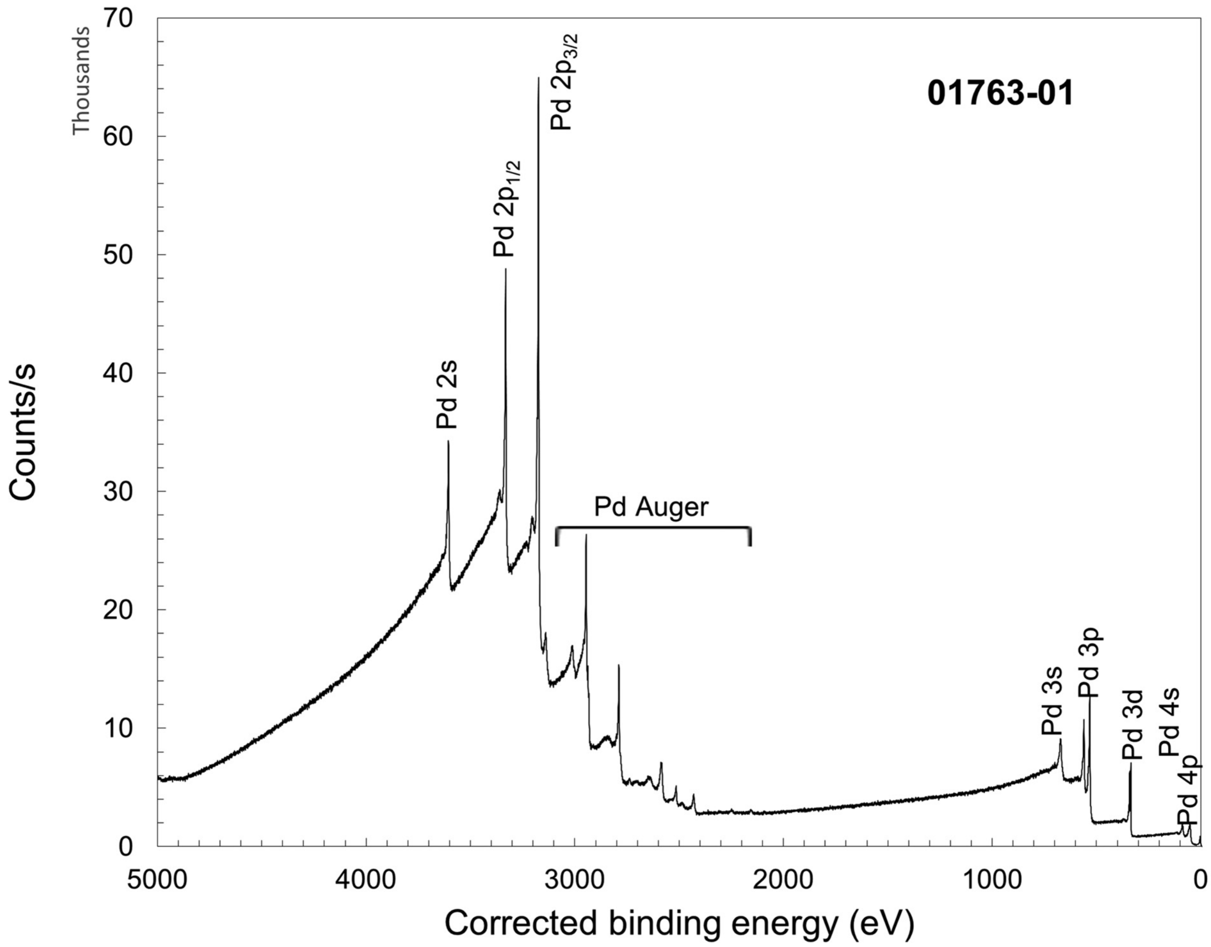
Accession #	01760-01
Host Material	Mo
Technique	XPS
Spectral Region	Survey
Instrument	ULVAC-PHI Quantes
Excitation Source	Cr K_α monochromatic
Source Energy	5414.8 eV
Source Strength	100 W
Source Size	0.1 × 1.4 mm^2
Analyzer Type	Spherical sector analyzer
Incident Angle	22°
Emission Angle	45°
Analyzer Pass Energy	280 eV
Analyzer Resolution	1.9 eV
Total Signal Accumulation Time	10 000 s
Total Elapsed Time	11 300 s
Number of Scans	10
Effective Detector Width	31 eV



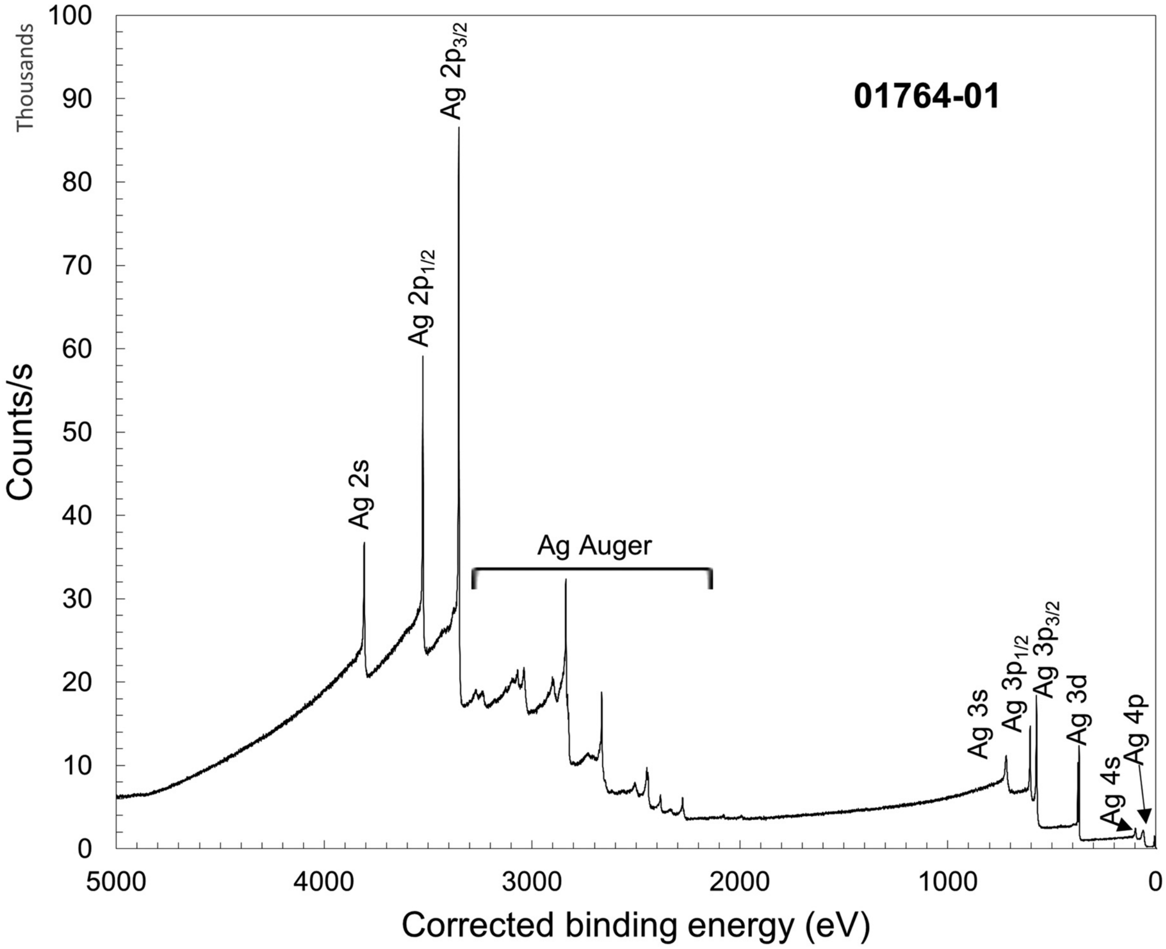
Accession #	01761-01
Host Material	Ru
Technique	XPS
Spectral Region	Survey
Instrument	ULVAC-PHI Quantes
Excitation Source	Cr K_{α} monochromatic
Source Energy	5414.8 eV
Source Strength	100 W
Source Size	0.1 × 1.4 mm^2
Analyzer Type	Spherical sector analyzer
Incident Angle	22°
Emission Angle	45°
Analyzer Pass Energy	280 Ev
Analyzer Resolution	1.9 eV
Total Signal Accumulation Time	10 000 s
Total Elapsed Time	11 300 s
Number of Scans	10
Effective Detector Width	31 eV



Accession #	01762-01
Host Material	Rh
Technique	XPS
Spectral Region	Survey
Instrument	ULVAC-PHI Quantes
Excitation Source	Cr K_α monochromatic
Source Energy	5414.8 eV
Source Strength	100 W
Source Size	0.1 × 1.4 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	10 000 s
Total Elapsed Time	11 300 s
Number of Scans	10
Effective Detector Width	31 eV



Accession #	01763-01
Host Material	Pd
Technique	XPS
Spectral Region	Survey
Instrument	ULVAC-PHI Quantes
Excitation Source	Cr K_α monochromatic
Source Energy	5414.8 eV
Source Strength	100 W
Source Size	0.1 × 1.4 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	10 000 s
Total Elapsed Time	11 300 s
Number of Scans	10
Effective Detector Width	31 eV



Accession #	01764-01
Host Material	Ag
Technique	XPS
Spectral Region	Survey
Instrument	ULVAC-PHI Quantes
Excitation Source	Cr K_{α} monochromatic
Source Energy	5414.8 eV
Source Strength	100 W
Source Size	0.1 × 1.4 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	10 000 s
Total Elapsed Time	11 300 s
Number of Scans	10
Effective Detector Width	31 eV