

# Reference survey spectra of elemental solid measured with Cr $K_{\alpha}$ photons as a tool for Quases analysis (1): Transition metals period 4 elements (Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn)

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- reactive sputter process control
- vacuum diagnostics
- vacuum coating process monitoring

# Reference survey spectra of elemental solid measured with Cr $K_{\alpha}$ photons as a tool for Quases analysis (1): Transition metals period 4 elements (Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn)

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## AFFILIATIONS

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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  $K_{\alpha}$  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.

**Key words:** HAXPES, Cr  $K_{\alpha}$ , Tougaard, Quases, inelastic background, elements, survey

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**Accession #s:** 01747, 01748, 01749, 01750, 01751, 01752, 01753, 01754, 01755, 01756

**Technique:** XPS

**Host Material:** Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn

**Instrument:** ULVAC-PHI Quantes

**Major Elements in Spectra:** Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn

**Minor Elements in Spectra:** Ar, Pb

**Published Spectra:** 10

**Spectral Category:** Reference

## INTRODUCTION

This work as 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 hard x-ray photoelectron spectroscopy (HAXPES) tools, the HAXPES technique is now easily accessible. The probing depth with the Tougaard method is  $\sim 8$  IMFP (Ref. 8), which is larger than the usual  $\sim 3$  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  $\sim 50$  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 the 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  $K_{\alpha}$  source.

### SPECIMEN DESCRIPTION (ACCESSION #s 01747, 01748, 01749, 01750, 01751, 01752, 01753, 01754, 01755, 01756)

**Host Material:** Scandium: Accession #01747; Titanium: Accession #01748; Vanadium: Accession #01749 Chromium: Accession #01750; Manganese: Accession #01751; Iron: Accession #01752; Cobalt: Accession #01753; Nickel: Accession #01754; Copper: Accession #01755; Zinc: Accession #01756

**CAS Registry #:** See Guide to Figures

**Host Material Characteristics:** Homogeneous; solid; polycrystalline; unknown conductivity; inorganic compound; other

**Chemical Name:** Same as host material

**Source:** Bulk samples

**Host Composition:** Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn

**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<sup>+</sup> sputter clean until oxygen and carbon peaks are removed from the spectra recorded with the Al  $K_{\alpha}$  source [standard cleaning 1 keV (lower for sensitive materials), sputter time dependent on the 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

**Pre-analysis Beam Exposure:** 0 s

### INSTRUMENT DESCRIPTION

**Manufacturer and Model:** ULVAC-PHI, Quantex

**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  $K_{\alpha}$  monochromatic

**Source Energy:** 5414.8 eV

**Source Strength:** 100 W

**Source Beam Size:**  $100 \times 1400 \mu\text{m}^2$

**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  $\mu\text{m}$

**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 alignment corresponds to the most often measured peak.

**Recommended Energy Scale Shift:** See “Guide to figure” table

**Peak Shape and Background Method:** None

**Quantitation Method:** None

#### AUTHOR DECLARATIONS

##### Conflicts 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 (lead); Writing – original draft (equal). **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 (Ref. 14).

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- <sup>13</sup>See <https://srdata.nist.gov/xps/ElmComposition.aspx>.
- <sup>14</sup>See [supplementary material](https://doi.org/10.1116/6.0001952) at <https://doi.org/10.1116/6.0001952> for ASCII data of all shown spectra.

SPECTRAL FEATURES TABLE

Spectrum ID #	Element/Transition	Peak Energy (eV) <sup>a</sup>	Peak Width FWHM (eV)	Peak Area (eV counts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01747-01	Sc 3p	28.7	...	...	...	...	...
	Sc 3s	50.9	...	...	...	...	...
	Sc 3p <sub>3/2</sub>	398.5 <sup>b</sup>	...	...	...	...	...
	Sc 3p <sub>1/2</sub>	403.4	...	...	...	...	...
	Sc KLL	3651.5 <sup>c</sup>	...	...	...	...	...
	Sc 1s	4489.2	...	...	...	...	...
01748-01	Ti 3p	32.8	...	...	...	...	...
	Ti 3s	58.5	...	...	...	...	...
	Ti 2p <sub>3/2</sub>	453.9 <sup>b</sup>	...	...	...	...	...
	Ti 2p <sub>1/2</sub>	460.0	...	...	...	...	...
	Ti 2s	560.4	...	...	...	...	...
	Ti KLL	4013.5 <sup>c</sup>	...	...	...	...	...
01749-01	Ti 1s	4964.7	...	...	...	...	...
	V 3p	37.2	...	...	...	...	...
	V 3s	66.1	...	...	...	...	...
	V 2p <sub>3/2</sub>	512.4 <sup>b</sup>	...	...	...	...	...
	V 2p <sub>1/2</sub>	520.0	...	...	...	...	...
	V 2s	626.5	...	...	...	...	...
01750-01	V L <sub>3</sub> M <sub>2,3</sub> M <sub>4,5</sub>	472.7 <sup>c</sup>	...	...	...	...	...
	Cr 3p	42.9	...	...	...	...	...
	Cr 3s	75.1	...	...	...	...	...
	Cr 2p <sub>3/2</sub>	574.8 <sup>b</sup>	...	...	...	...	...
	Cr 2p <sub>1/2</sub>	584.1	...	...	...	...	...
	Cr 2s	696.5	...	...	...	...	...
01751-01	Mn 3p	47.6	...	...	...	...	...
	Mn 3s	82.8	...	...	...	...	...
	Mn 2p <sub>3/2</sub>	639.5 <sup>b</sup>	...	...	...	...	...
	Mn 2p <sub>1/2</sub>	650.4	...	...	...	...	...
	Mn L <sub>3</sub> M <sub>4,5</sub> M <sub>4,5</sub>	635.2 <sup>c</sup>	...	...	...	...	...
	Mn L <sub>3</sub> M <sub>2,3</sub> M <sub>4,5</sub>	586.3 <sup>c</sup>	...	...	...	...	...
01752-01	Fe 3p	52.9	...	...	...	...	...
	Fe 3s	91.2	...	...	...	...	...
	Fe 2p <sub>3/2</sub>	707.1 <sup>b</sup>	...	...	...	...	...
	Fe 2p <sub>1/2</sub>	720.1	...	...	...	...	...
	Fe 2s	845.7	...	...	...	...	...
	Fe L <sub>3</sub> M <sub>4,5</sub> M <sub>4,5</sub>	703.2 <sup>c</sup>	...	...	...	...	...
01753-01	Co 3p	59.3	...	...	...	...	...
	Co 3s	100.9	...	...	...	...	...
	Co 2p <sub>3/2</sub>	778.2 <sup>b</sup>	...	...	...	...	...
	Co 2p <sub>1/2</sub>	793.5	...	...	...	...	...
	Co 2s	925.3	...	...	...	...	...
	Co L <sub>2</sub> M <sub>2,3</sub> M <sub>4,5</sub>	774.1 <sup>c</sup>	...	...	...	...	...
01754-01	Ni 3p	66.5	...	...	...	...	...
	Ni 3s	110.6	...	...	...	...	...
	Ni 2p <sub>3/2</sub>	852.7 <sup>b</sup>	...	...	...	...	...
	Ni 2p <sub>1/2</sub>	870.0	...	...	...	...	...
	Ni 2s	1008.9	...	...	...	...	...
	Ni L <sub>2</sub> M <sub>2,3</sub> M <sub>4,5</sub>	847.8 <sup>c</sup>	...	...	...	...	...
01755-01	Cu 3p	75.8	...	...	...	...	...
	Cu 3s	122.4	...	...	...	...	...
	Cu 2p <sub>3/2</sub>	932.6 <sup>b</sup>	...	...	...	...	...

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**SPECTRAL FEATURES TABLE** (Continued.)

Spectrum ID #	Element/Transition	Peak Energy (eV) <sup>a</sup>	Peak Width FWHM (eV)	Peak Area (eV counts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01756-01	Cu 2p <sub>1/2</sub>	952.6	...	...	...	...	...
	Cu 2s	1097.1	...	...	...	...	...
	Cu L <sub>3</sub> M <sub>4,5</sub> M <sub>4,5</sub>	918.8 <sup>c</sup>	...	...	...	...	...
	Zn 3p	88.9	...	...	...	...	...
	Zn 3s	139.7	...	...	...	...	...
	Zn 2p <sub>3/2</sub>	1021.7 <sup>b</sup>	...	...	...	...	...
	Zn 2p <sub>1/2</sub>	1044.8	...	...	...	...	...
	Zn 2s	1196.1	...	...	...	...	...
Zn L <sub>3</sub> M <sub>4,5</sub> M <sub>4,5</sub>	992.5 <sup>c</sup>	...	...	...	...	...	

<sup>a</sup>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.

<sup>b</sup>Peak used for binding energy referencing.

<sup>c</sup>Peak position is given in kinetic energy.

**ANALYZER CALIBRATION TABLE**

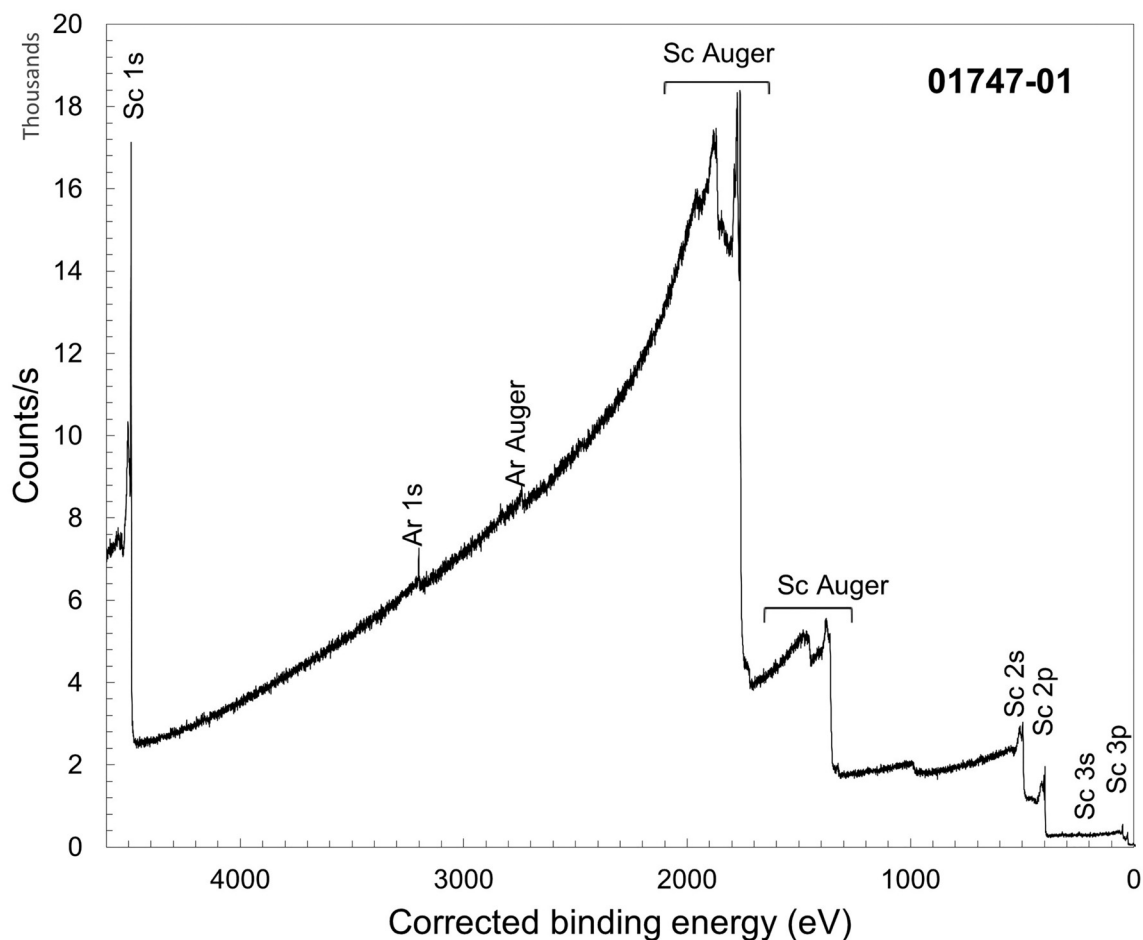
Spectrum ID #	Element/Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV counts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
...	Cu 2p <sub>3/2</sub>	932.66	0.88	79 646	...	...	...
...	Ag 3d <sub>5/2</sub>	368.25	0.63	79 262	...	...	...
...	Au 4f <sub>7/2</sub>	84.02	0.73	35 042	...	...	...

The calibration table is established using the Cr K<sub>α</sub> photons and a pass energy of 112 eV (analyzer resolution 0.86 eV).

**GUIDE TO FIGURES**

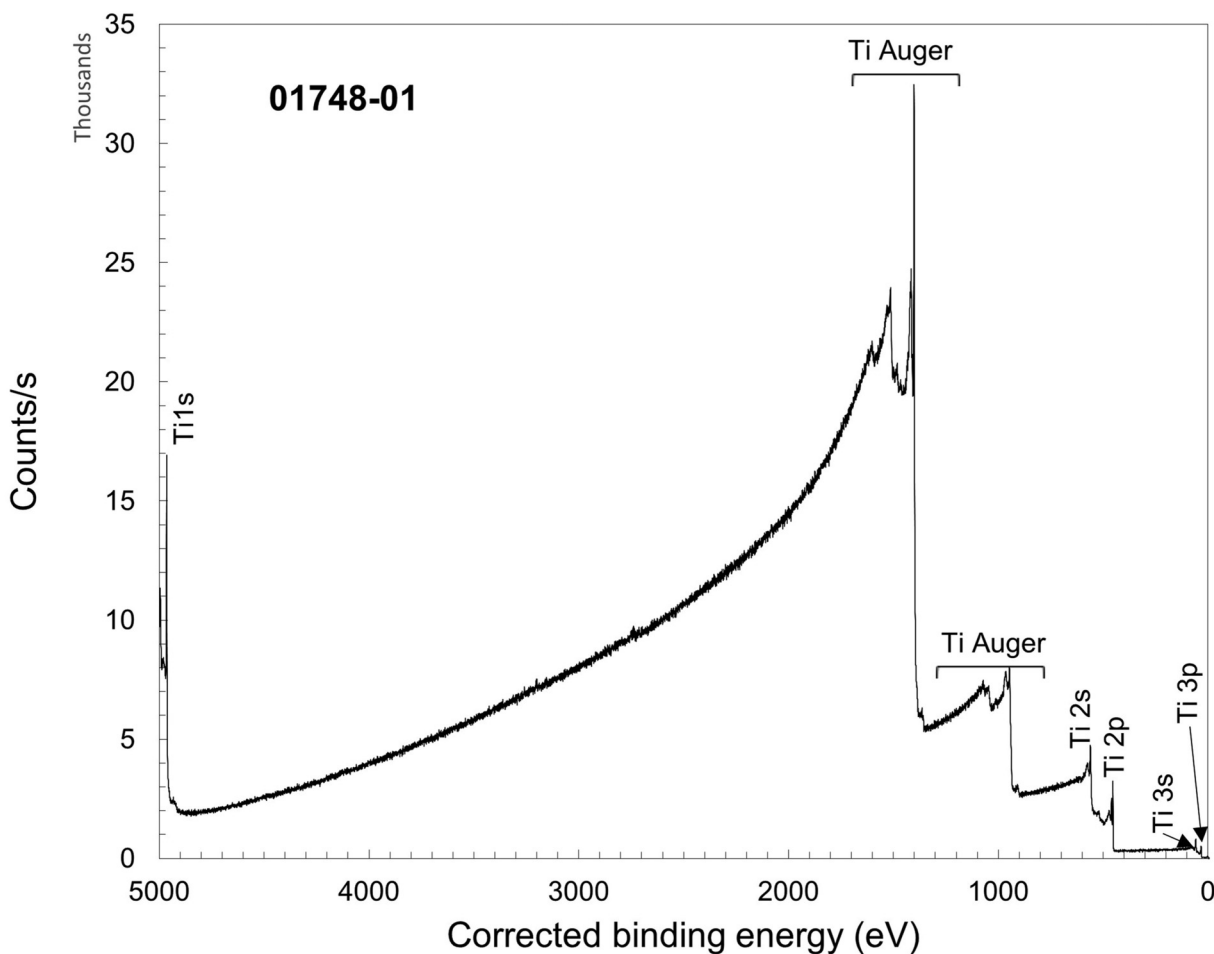
Spectrum (Accession) #	Spectral Region	Voltage Shift <sup>a</sup>	Multiplier	Baseline	Comment #
01747-01	Survey	-1.4	1	0	Sc: CAS 7440-20-2; Goodfellow: 494-421-37
01748-01	Survey	-1.8	1	0	Ti: CAS 7440-32-6; Goodfellow 832- 171-02
01749-01	Survey	-0.5	1	0	V: CAS 7440-62-2; Goodfellow: 827-358-69
01750-01	Survey	-0.3	1	0	Cr: CAS 7440-47-3; Goodfellow: 182-300-60
01751-01	Survey	2.1	1	0	Mn: CAS: 7439-96-5; Goodfellow 244-303-95
01752-01	Survey	-0.9	1	0	Fe: CAS 7439-89-6; Goodfellow: 446-762-65
01753-01	Survey	1.7	1	0	Co: CAS 7440-48-4 PVD film on Si
01754-01	Survey	-0.4	1	0	Ni: CAS 7440-02-0; Goodfellow: 194-552-06
01755-01	Survey	-0.5	1	0	Cu: CAS 7440-50-8; PVD film on Si
01756-01	Survey	0.0	1	0	Zn: CAS 7440-66-6; Goodfellow: 260-142-05

<sup>a</sup>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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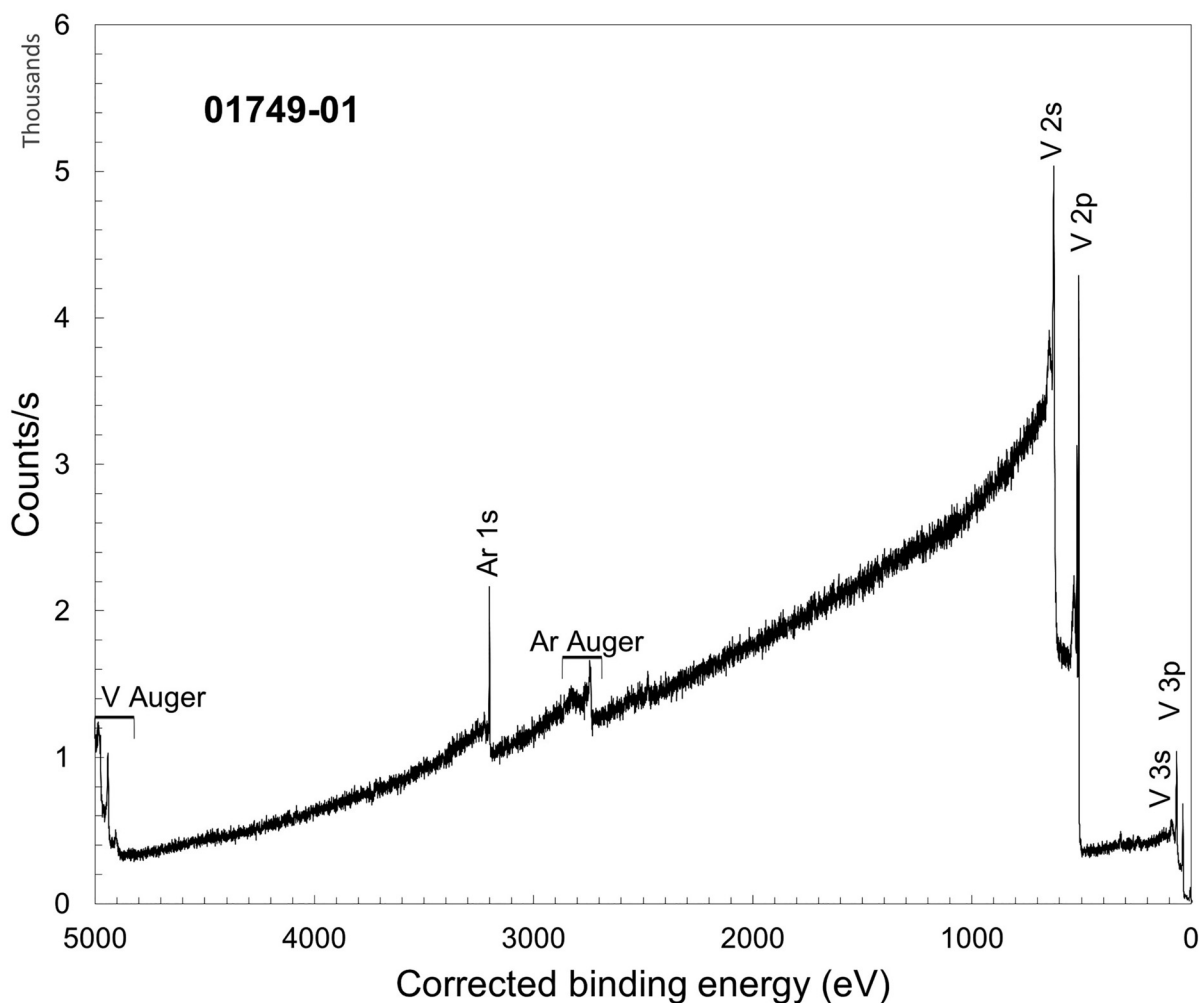
Accession #:	01747-01
Host Material:	Sc
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Cr K <sub>α</sub> monochromatic
Source Energy:	5414.8 eV
Source Strength:	100 W
Source Size:	0.1 × 1.4 mm <sup>2</sup>
Analyzer Type:	Spherical sector analyzer
Incident Angle:	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



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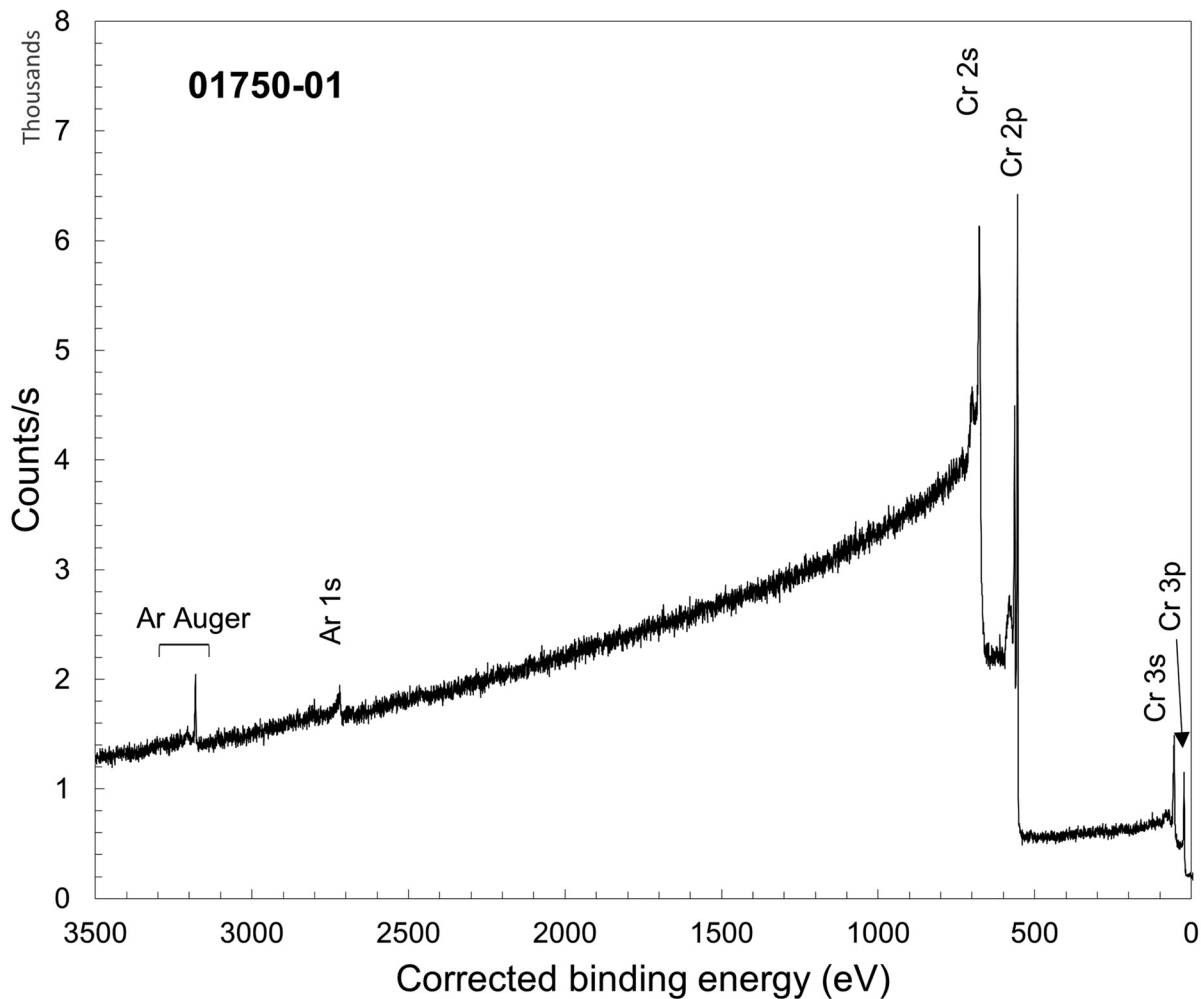
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<b>Host Material:</b>	Ti
<b>Technique:</b>	XPS
<b>Spectral Region:</b>	Survey
<b>Instrument:</b>	ULVAC-PHI Quantes
<b>Excitation Source:</b>	Cr K <sub>α</sub> monochromatic
<b>Source Energy:</b>	5414.8 eV
<b>Source Strength:</b>	100 W
<b>Source Size:</b>	0.1 × 1.4 mm <sup>2</sup>
<b>Analyzer Type:</b>	Spherical sector analyzer
<b>Incident Angle:</b>	22°
<b>Emission Angle:</b>	45°
<b>Analyzer Pass Energy:</b>	280 eV
<b>Analyzer Resolution:</b>	1.9 eV
<b>Total Signal Accumulation Time:</b>	10 000 s
<b>Total Elapsed Time:</b>	11 300 s
<b>Number of Scans:</b>	10
<b>Effective Detector Width:</b>	31 eV





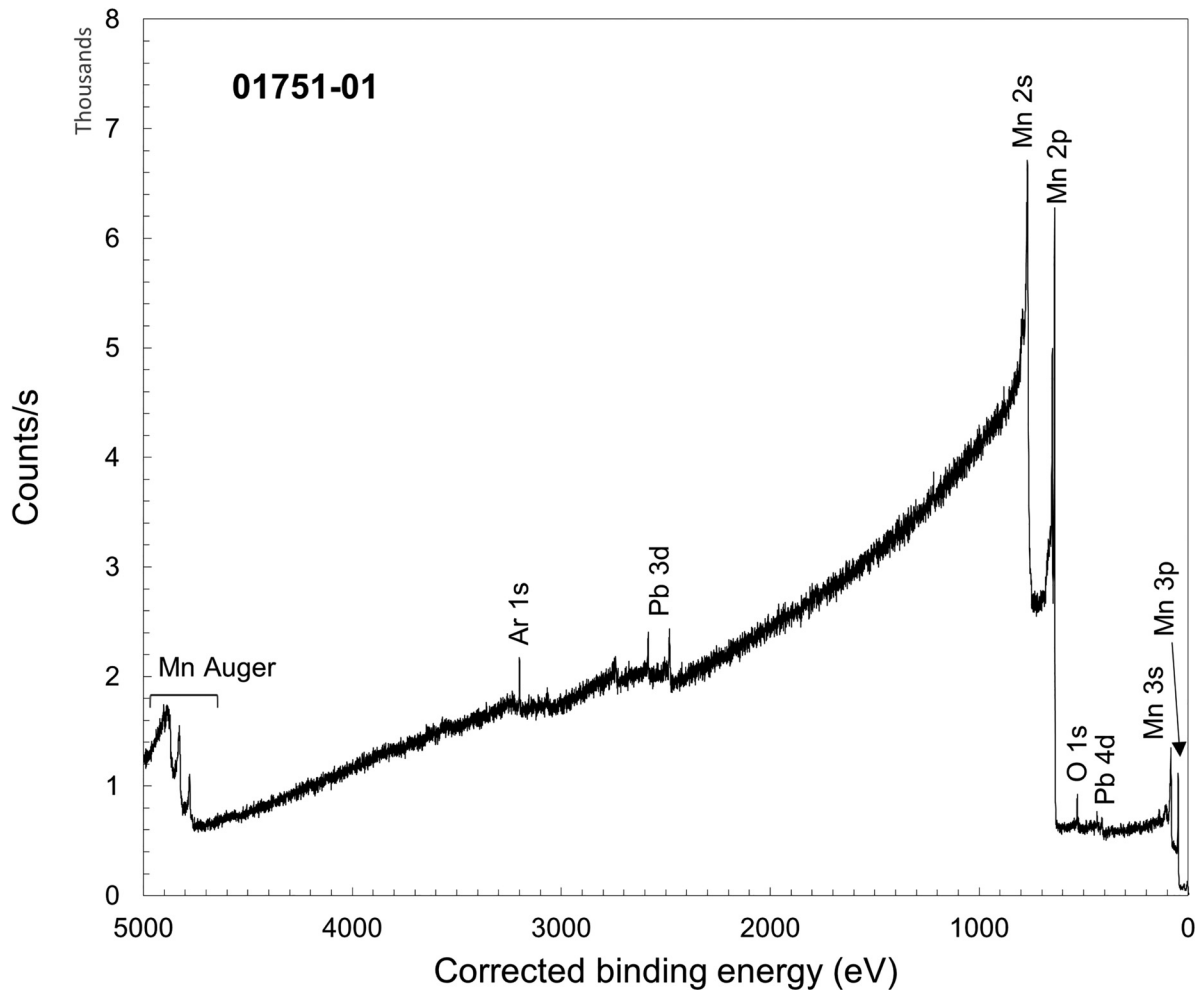
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Accession #:	01749-01
Host Material:	V
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Cr $K_{\alpha}$ monochromatic
Source Energy:	5414.8 eV
Source Strength:	100 W
Source Size:	0.1 × 1.4 mm <sup>2</sup>
Analyzer 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



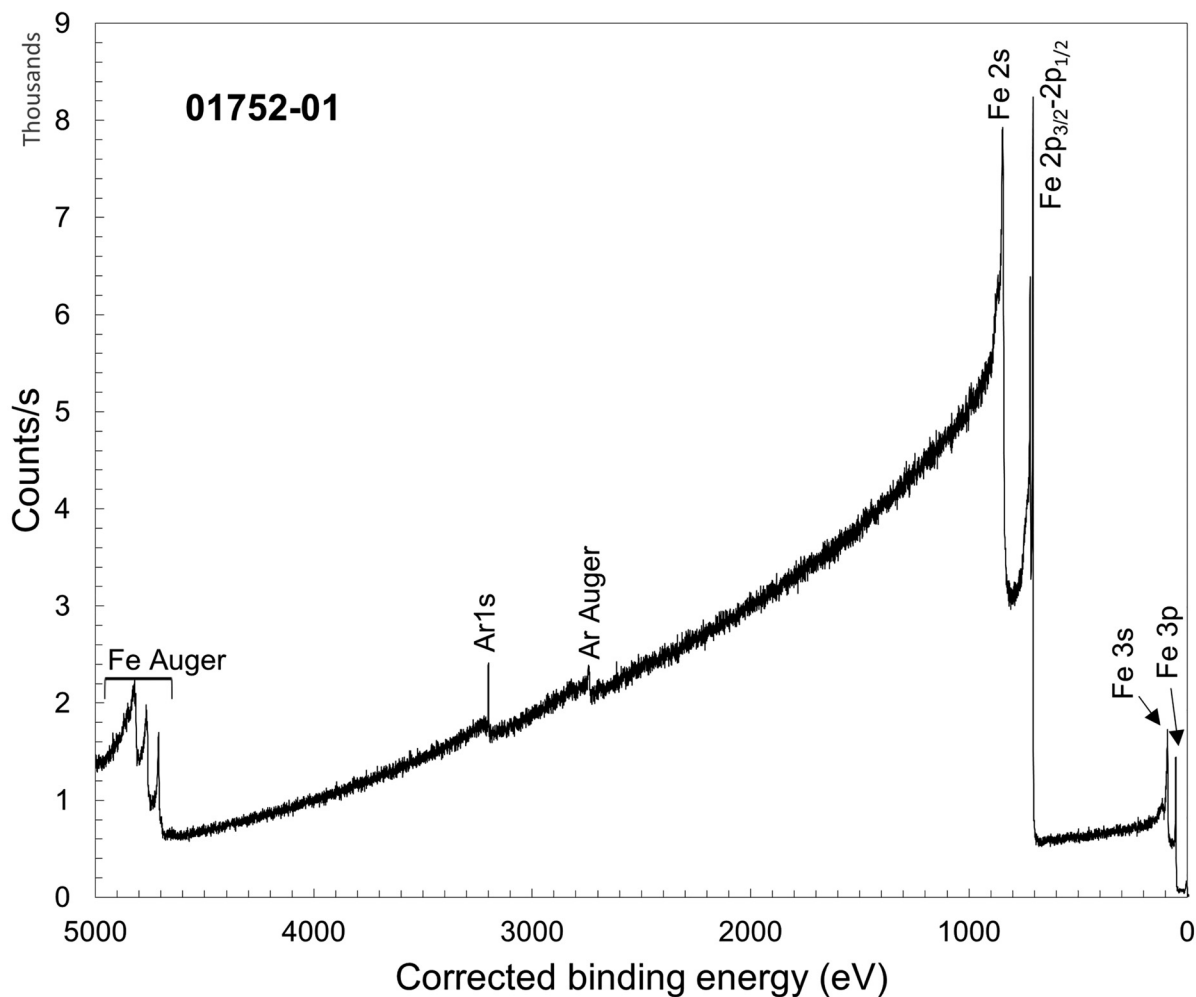
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<b>Accession #:</b>	01750-01
<b>Host Material:</b>	Cr
<b>Technique:</b>	XPS
<b>Spectral Region:</b>	Survey
<b>Instrument:</b>	ULVAC-PHI Quantes
<b>Excitation Source:</b>	Cr $K_{\alpha}$ monochromatic
<b>Source Energy:</b>	5414.8 eV
<b>Source Strength:</b>	100 W
<b>Source Size:</b>	0.1 × 1.4 mm <sup>2</sup>
<b>Analyzer Type:</b>	Spherical sector analyzer
<b>Incident Angle:</b>	22°
<b>Emission Angle:</b>	45°
<b>Analyzer Pass Energy:</b>	280 eV
<b>Analyzer Resolution:</b>	1.9 eV
<b>Total Signal Accumulation Time:</b>	10 000 s
<b>Total Elapsed Time:</b>	11 300 s
<b>Number of Scans:</b>	10
<b>Effective Detector Width:</b>	31 eV



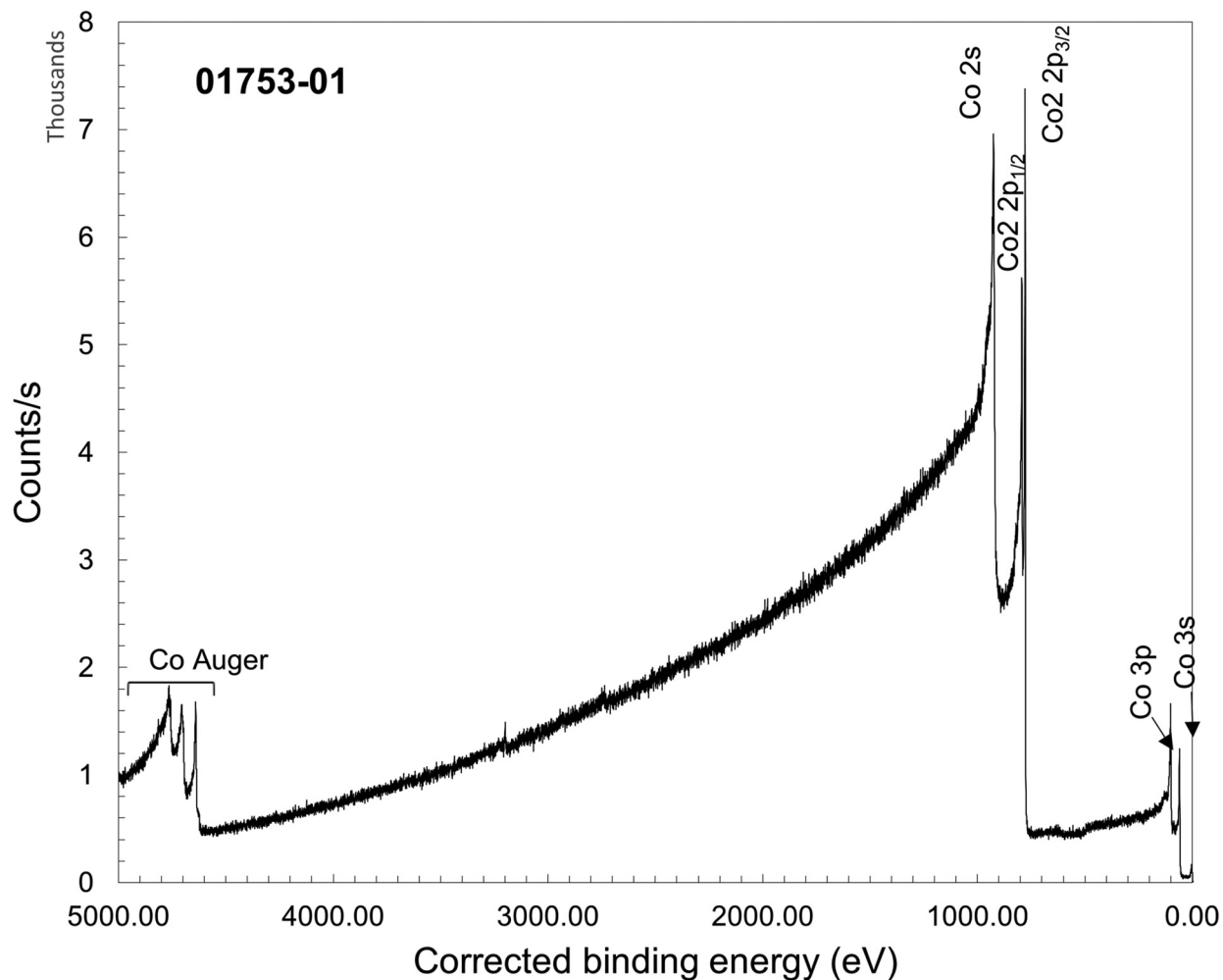
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Accession #:	01751-01
Host Material:	Mn
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Cr $K_{\alpha}$ monochromatic
Source Energy:	5414.8 eV
Source Strength:	100 W
Source Size:	0.1 × 1.4 mm <sup>2</sup>
Analyzer 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



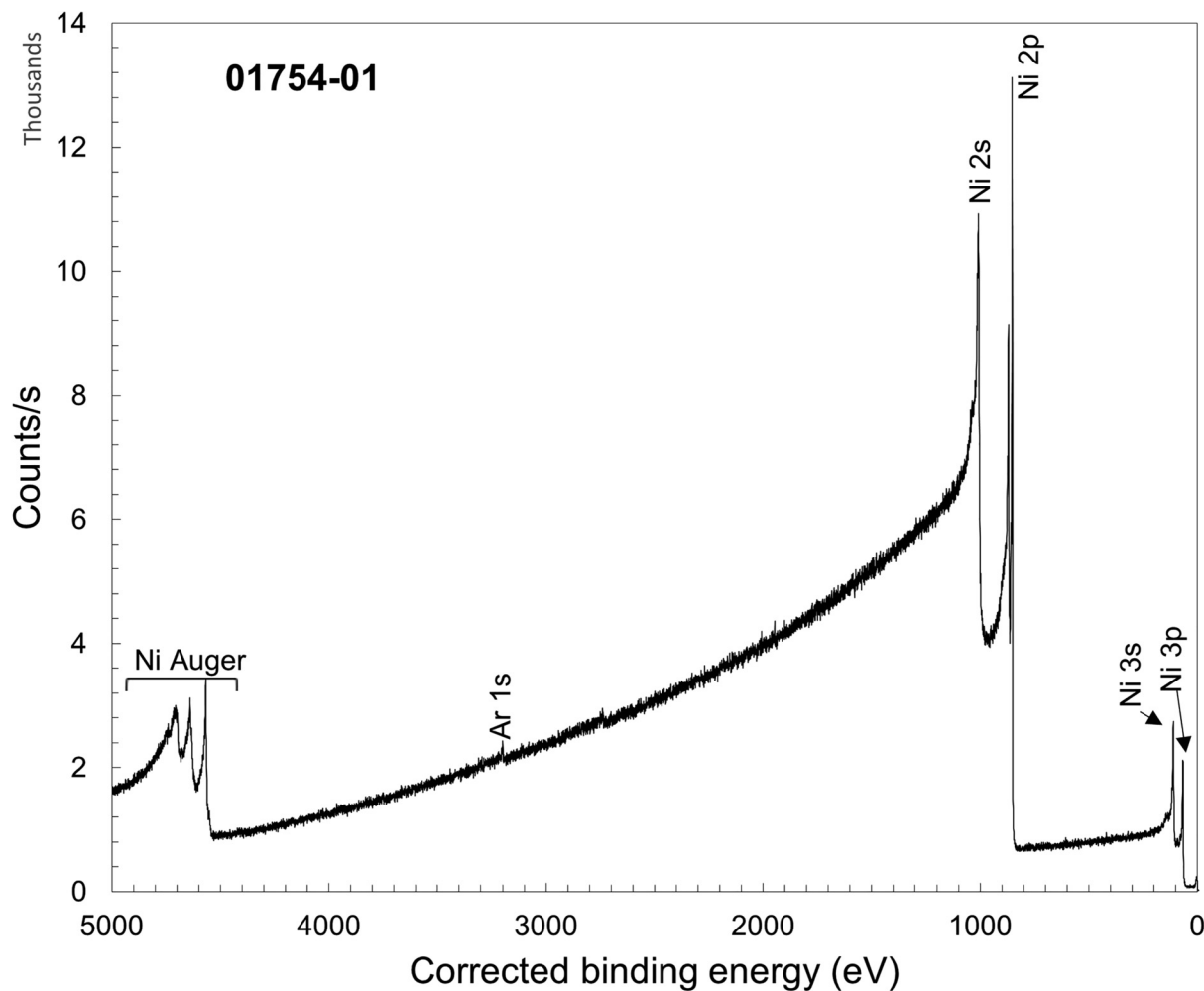
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Accession #:	01752-01
Host Material:	Fe
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Cr K <sub>α</sub> monochromatic
Source Energy:	5414.8 eV
Source Strength:	100 W
Source Size:	0.1 × 1.4 mm <sup>2</sup>
Analyzer 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



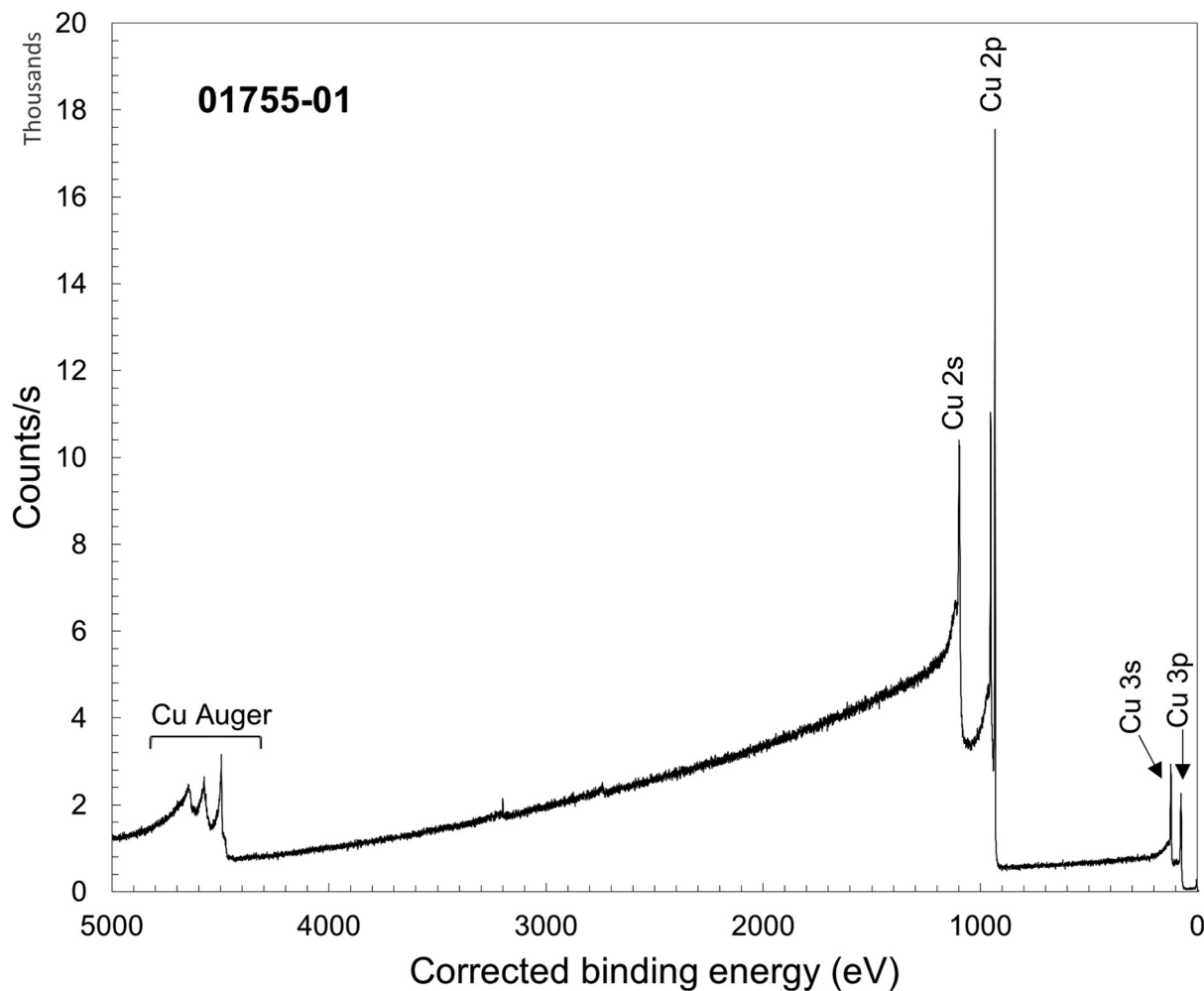
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Accession #:	01753-01
Host Material:	Co
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Cr K <sub>α</sub> monochromatic
Source Energy:	5414.8 eV
Source Strength:	100 W
Source Size:	0.1 × 1.4 mm <sup>2</sup>
Analyzer 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



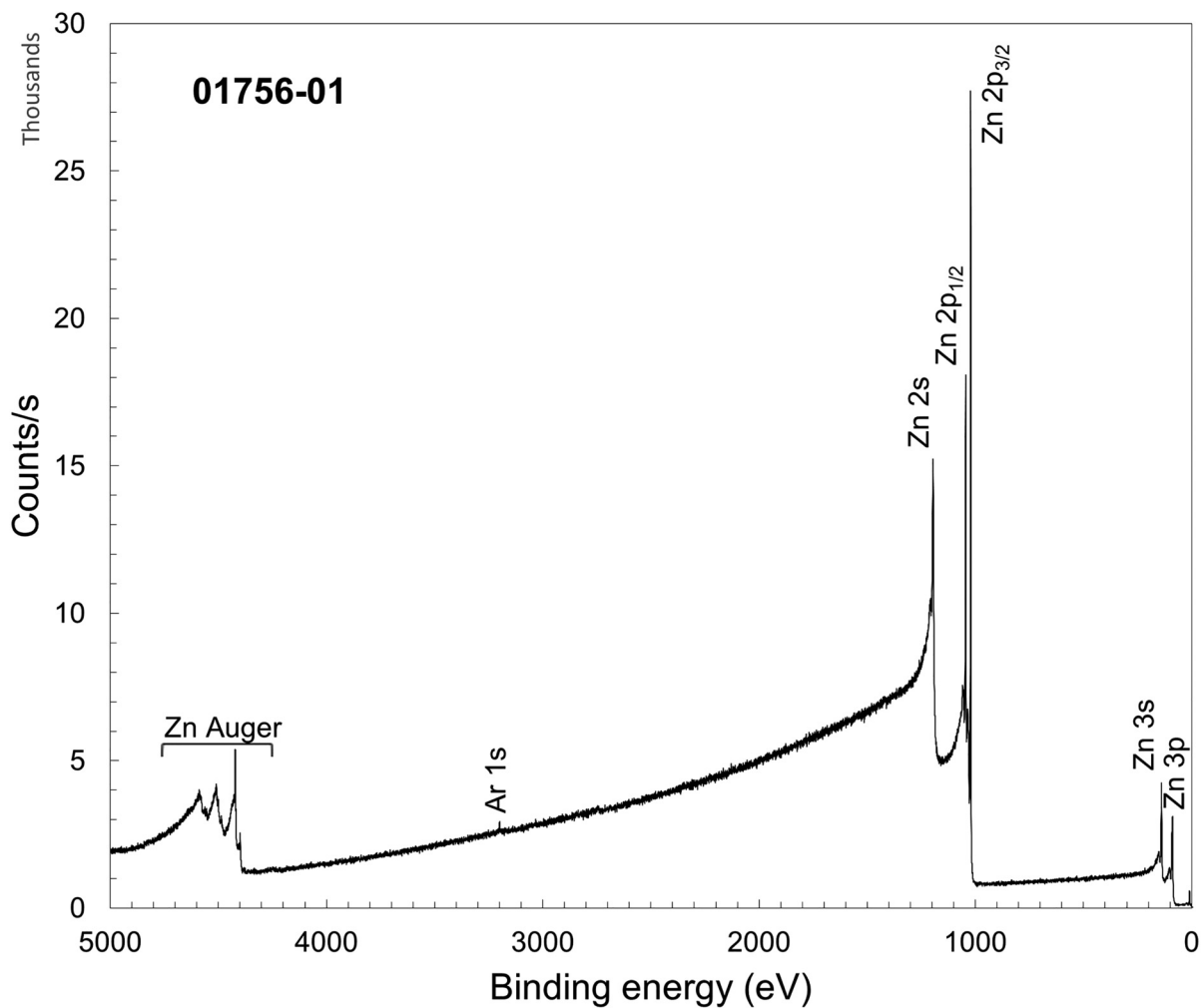
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Accession #:	01754-01
Host Material:	Ni
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Cr $K_{\alpha}$ monochromatic
Source Energy:	5414.8 eV
Source Strength:	100 W
Source Size:	0.1 × 1.4 mm <sup>2</sup>
Analyzer 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



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Accession #:	01755-01
Host Material:	Cu
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Cr K <sub>α</sub> monochromatic
Source Energy:	5414.8 eV
Source Strength:	100 W
Source Size:	0.1 × 1.4 mm <sup>2</sup>
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



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Accession #:	01756-01
Host Material:	Zn
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Cr K <sub>α</sub> monochromatic
Source Energy:	5414.8 eV
Source Strength:	100 W
Source Size:	0.1 × 1.4 mm <sup>2</sup>
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