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HAXPES spectra of NaCl measured by Cr K_{α}

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HAXPES spectra of NaCl measured by Cr K_{α}

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

ABSTRACT

Single-crystalline NaCl was analyzed using high-resolution high-energy x-ray photoelectron spectroscopy (HAXPES). The HAXPES spectra of NaCl 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 Na 1s, Na 2p, Na 2s, Cl 1s, Cl 2p, Cl 2s, and Cl KLL.

Key words: GaAs, HAXPES Cr K_{α}

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Accession#: 01742 and 01743

Technique: XPS and XAES

Host Material: NaCl

Instrument: ULVAC-PHI Quantes

Major Elements in Spectra: Na and Cl

Minor Elements in Spectra: None

Published Spectra: 10

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.

Single-crystalline NaCl was analyzed using high-resolution high-energy x-ray photoelectron spectroscopy (HAXPES). The HAXPES spectra of NaCl 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 Na 1s, Na 2p, Na 2s, Cl 1s, Cl 2p, Cl 2s, and Cl KLL.

SPECIMEN DESCRIPTION (ACCESSION # 01742)

Host Material: NaCl

CAS Registry #: 7647-14-5

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

Chemical Name: Sodium chloride

Source: Single crystal

Host Composition: Na and Cl

Form: Single crystal

Structure: Single crystal

History and Significance: Air exposed

As Received Condition: Single crystal (100)

Analyzed Region: Same as the host material

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

In Situ Preparation: Sample was sputtered using 100 eV Ar⁺ ions until removal of surface C contamination and reduction of the oxygen signal

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

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 an insulating removable 3 M 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: $\frac{A}{E_p} = \left(\frac{a^2}{a^2 + R^2}\right)^b$, 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 squares 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: 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 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 Na 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 Na 1s is 1072.09 eV. The binding energy shift of the Cr K_α data was then performed by shifting the Na 1s peak to 1072.09 eV. This energy calibration method—assigning the same binding energy position to Na 1s peaks obtained with two different photons energies—may contain a small error as the recoil effect is neglected. However, this effect is very small and is expected to be of the order of 0.09 eV.

Recommended Energy Scale Shift: 1.44 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. Theoretical sensitivity factors were provided by the software. Theoretical RSFs have been calculated with consideration of matrix effects. Inelastic mean free paths were calculated by TPP-2M for the average matrix sample. Elastic-electron correction was calculated by a method based on ISO18118:2015. Back-scattering factor was ignored. Photoionization cross sections and asymmetry parameters were referred by Atomic Data and Nuclear Data Tables (Refs. 4 and 5) and Scofield (Ref. 6). Photoionization cross section of high kinetic energy region excited by Cr K_α was extrapolated based on a method developed by Verner et al. (Refs. 7 and 8). The RSF values are tabulated in the spectral features table. These RSF values do not include detector-related corrections.

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](#).⁹

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>

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⁹See the [supplementary material](https://www.scitation.org/doi/suppl/10.1116/6.0001531) at <https://www.scitation.org/doi/suppl/10.1116/6.0001531> for the figures.

SPECTRAL FEATURES TABLE

Spectrum ID #	Element/Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01742-03	Na 1s	1072.09	1.65	5 714	2.109	56.55	NaCl
01742-04	Na 2p	31.07	1.61	85	0.018	...	NaCl
01742-05	Na 2s	63.89	1.65	403	0.137	...	NaCl
01742-06	Cl 1s	2821.29	1.76	17 525	7.258	43.45	NaCl
01742-07	Cl 2p _{3/2}	199.04	1.56	646	0.198	...	NaCl
01742-08	Cl 2s	270.40	2.64	1 799	0.783	...	NaCl

ANALYZER CALIBRATION TABLE

Spectrum ID #	Element/Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × cts/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 Cr K_α photons and a pass energy of 112 eV corresponding to the presented high-resolution spectra.

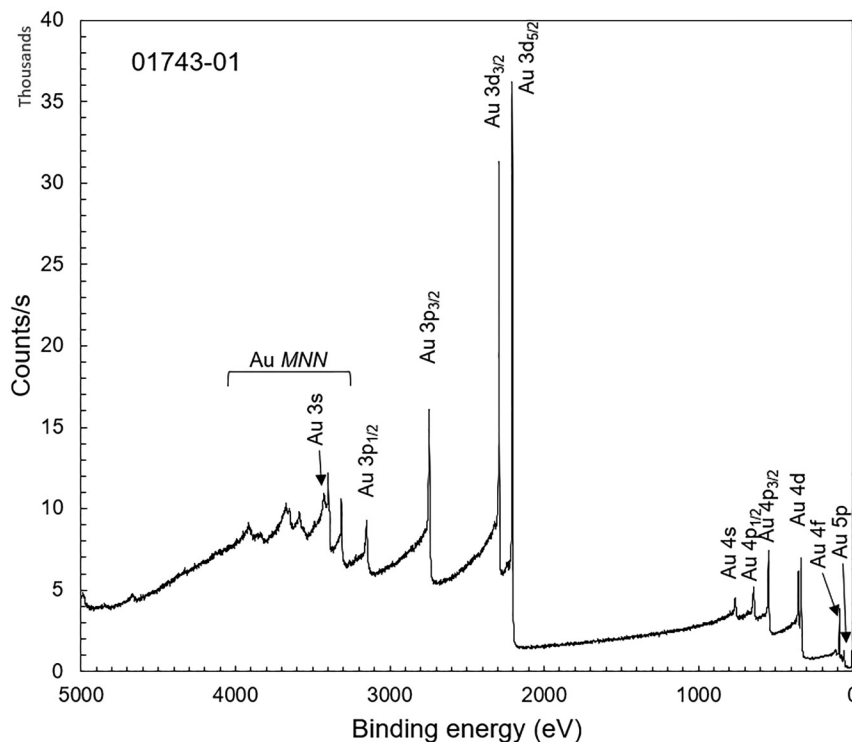
GUIDE TO FIGURES

Spectrum (Accession) #	Spectral Region	Voltage Shift ^a	Multiplier	Baseline	Comment #
01743-01	Survey	0	1	0	^b Au survey with x-ray source Cr _{Kα}
01742-01	Survey	0	1	0	X-ray source Al K _α
01742-02	Survey	0	1	0	X-ray source Cr K _α
01742-03	Na 1s	−1.44 eV	1	0	...
01742-04	Na 2p	−1.44 eV	1	0	...
01742-05	Na 2s	−1.44 eV	1	0	...
01742-06	Cl 1s	−1.44 eV	1	0	...
01742-07	Cl 2p	−1.44 eV	1	0	...
01742-08	Cl 2s	−1.44 eV	1	0	...
01742-09	Cl KL _{2,3} L _{2,3}	−1.44 eV	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.

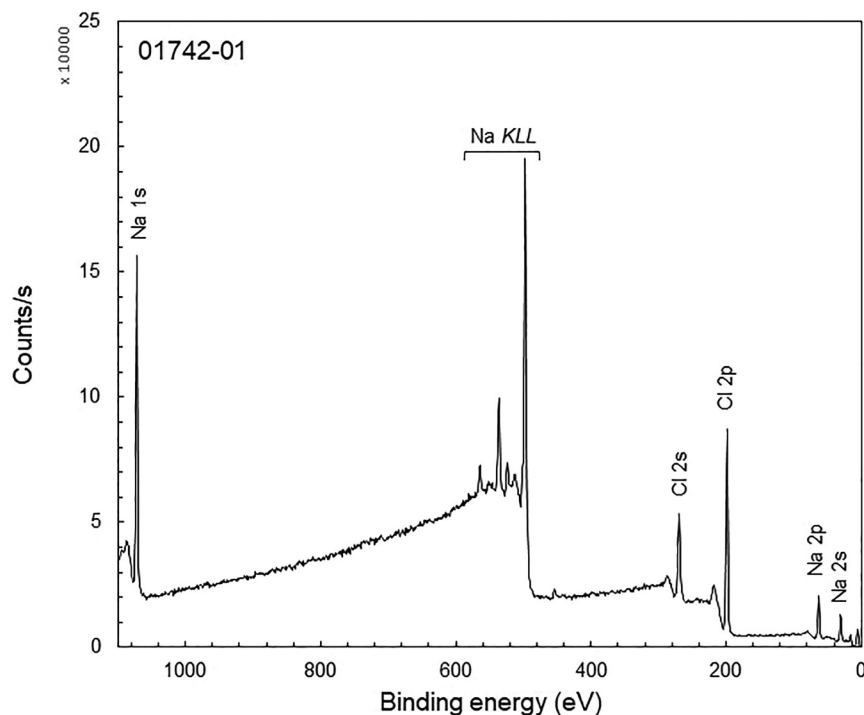
^bThe reference spectrum included in this article is the same as those included with other articles in this collection that use this x-ray source. This was intentional and not an error as submissions were requested to be accompanied by a wide-scan spectrum of sputter-cleaned gold taken on the same instrument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

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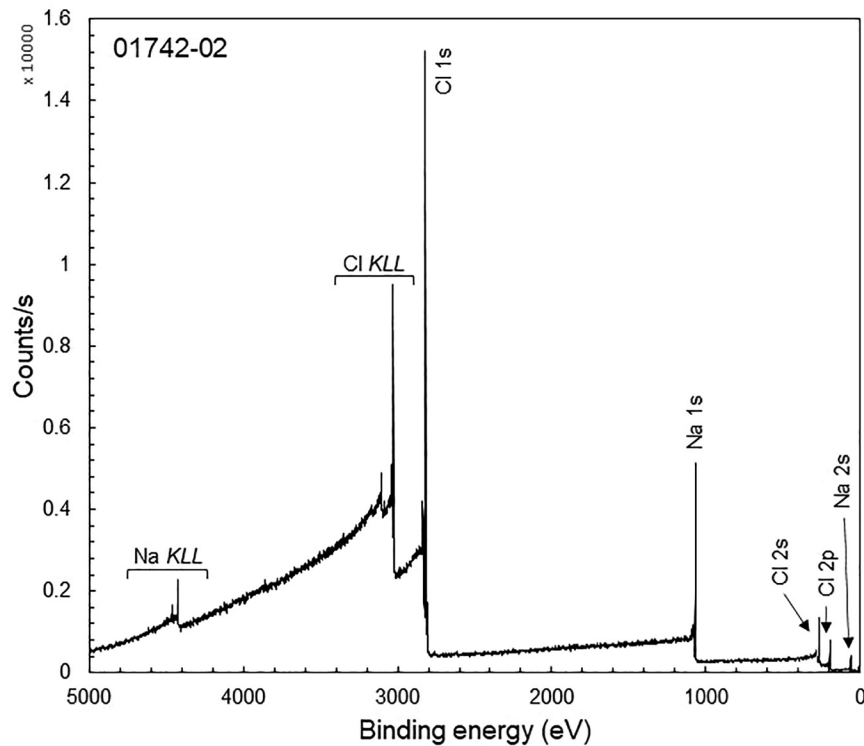
Accession #	01743-01
Host Material:	Au
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Cr K _α monochromatic
Source Energy:	5414.8 eV
Source Strength:	49 W
Source Size:	0.1 × 0.1 mm ²
Analyzer Type:	Spherical sector analyzer
Incident Angle:	22°
Emission Angle:	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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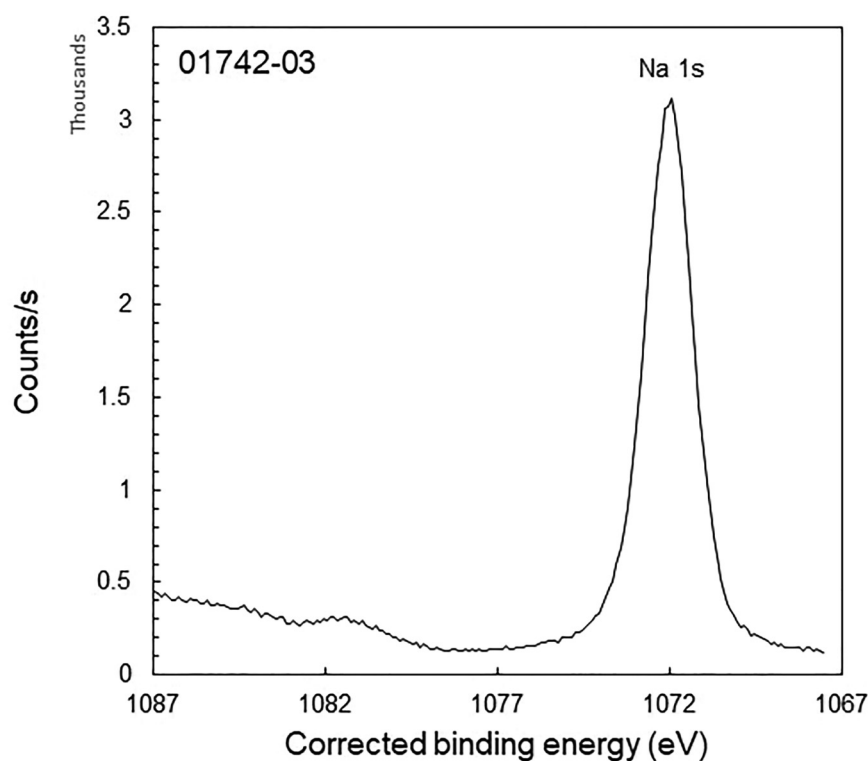
Accession #	01742-01
Host Material:	NaCl
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Al K_{α} monochromatic
Source Energy:	1486.6 eV
Source Strength:	25 W
Source Size:	$0.1 \times 0.1 \text{ mm}^2$
Analyzer Type:	Spherical sector analyzer
Incident Angle:	22°
Emission Angle:	45°
Analyzer Pass Energy:	280 eV
Analyzer Resolution:	1.9 eV
Total Signal Accumulation Time:	110 s
Total Elapsed Time:	130 s
Number of Scans:	1
Effective Detector Width:	31 eV

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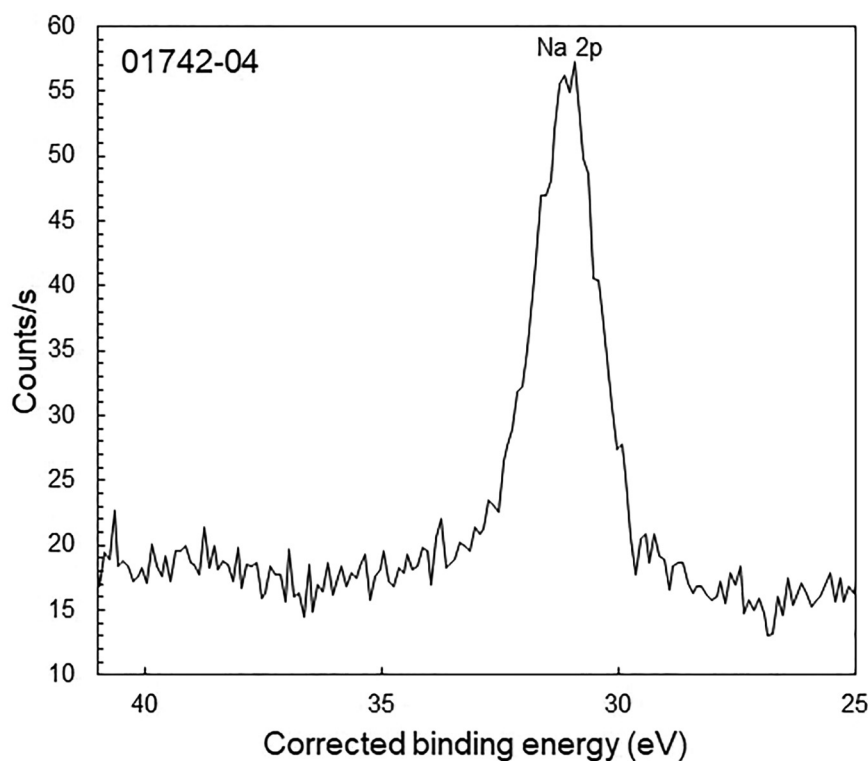
Accession #	01742-02
Host Material:	NaCl
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Cr K_{α} monochromatic
Source Energy:	5414.8 eV
Source Strength:	43 W
Source Size:	0.1 \times 0.1 mm ²
Analyzer Type:	Spherical sector analyzer
Incident Angle:	22°
Emission Angle:	45°
Analyzer Pass Energy:	280 eV
Analyzer Resolution:	1.9 eV
Total Signal Accumulation Time:	4500 s
Total Elapsed Time:	4950 s
Number of Scans:	9
Effective Detector Width:	31 eV

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- Accession #: 01742-03
- Host Material: NaCl
- Technique: XPS
- Spectral Region: Na 1s

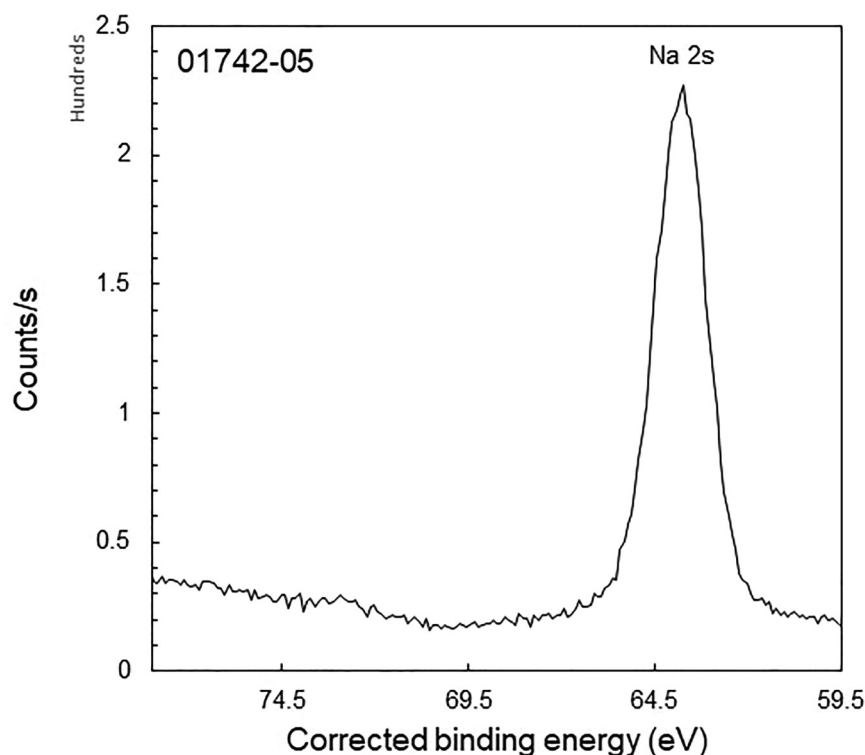
Instrument: ULVAC-PHI Quantes
 Excitation Source: Cr K_{α} monochromatic
 Source Energy: 5414.8 eV
 Source Strength: 43 W
 Source Size: $0.1 \times 0.1 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 22°
 Emission Angle: 45°
 Analyzer Pass Energy: 112 eV
 Analyzer Resolution: 0.86 eV
 Total Signal Accumulation Time: 800 s
 Total Elapsed Time: 880 s
 Number of Scans: 40
 Effective Detector Width: 12.4 eV



- Accession #: 01742-04
- Host Material: NaCl
- Technique: XPS
- Spectral Region: Na 2p

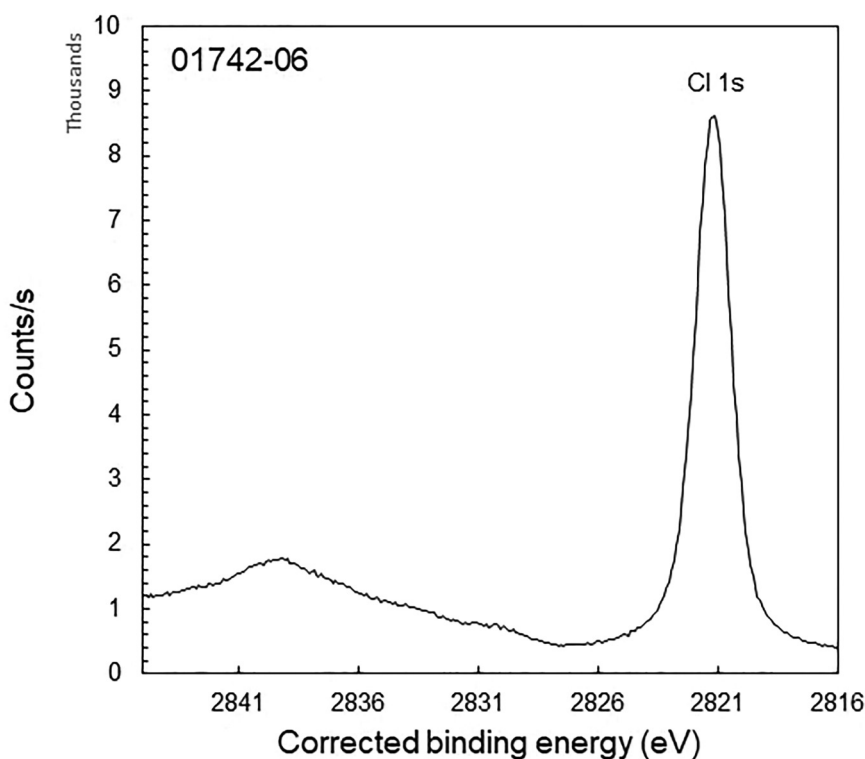
Instrument: ULVAC-PHI Quantes
 Excitation Source: Cr K_{α} monochromatic
 Source Energy: 5414.8 eV
 Source Strength: 43 W
 Source Size: $0.1 \times 0.1 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 22°
 Emission Angle: 45°
 Analyzer Pass Energy: 112 eV
 Analyzer Resolution: 0.86 eV
 Total Signal Accumulation Time: 2400 s
 Total Elapsed Time: 2650 s
 Number of Scans: 120
 Effective Detector Width: 12.4 eV

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■ Accession #: [01742-05](#)
 ■ Host Material: NaCl
 ■ Technique: XPS
 ■ Spectral Region: Na 2s

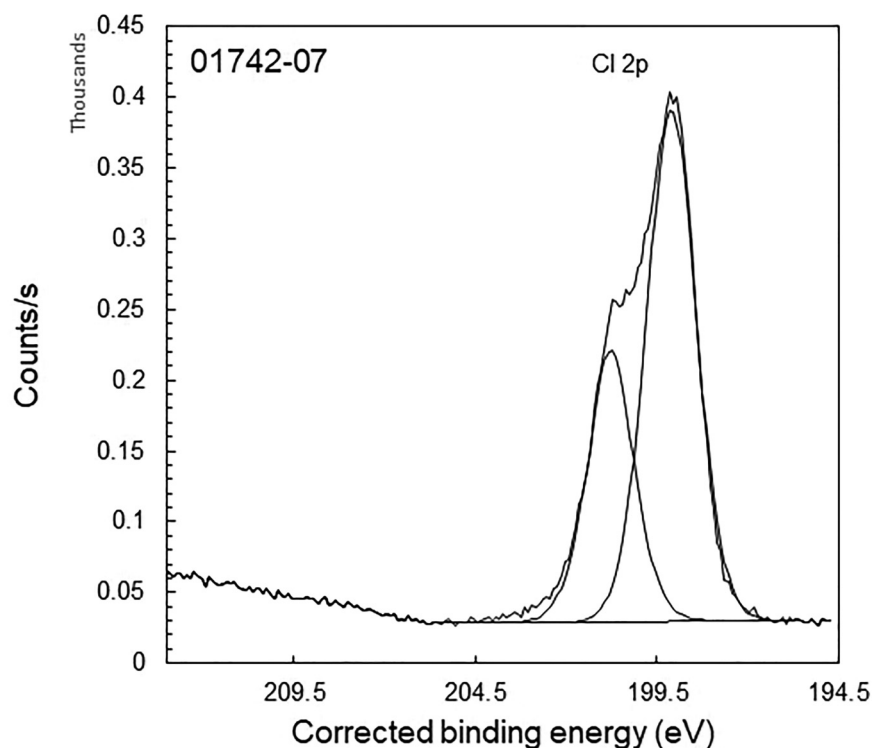
Instrument: ULVAC-PHI Quantes
 Excitation Source: Cr K_{α} monochromatic
 Source Energy: 5414.8 eV
 Source Strength: 43 W
 Source Size: $0.1 \times 0.1 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 22°
 Emission Angle: 45°
 Analyzer Pass Energy: 112 eV
 Analyzer Resolution: 0.86 eV
 Total Signal Accumulation Time: 2400 s
 Total Elapsed Time: 2650 s
 Number of Scans: 120
 Effective Detector Width: 12.4 eV



■ Accession #: [01742-06](#)
 ■ Host Material: NaCl
 ■ Technique: XPS
 ■ Spectral Region: Cl 1s

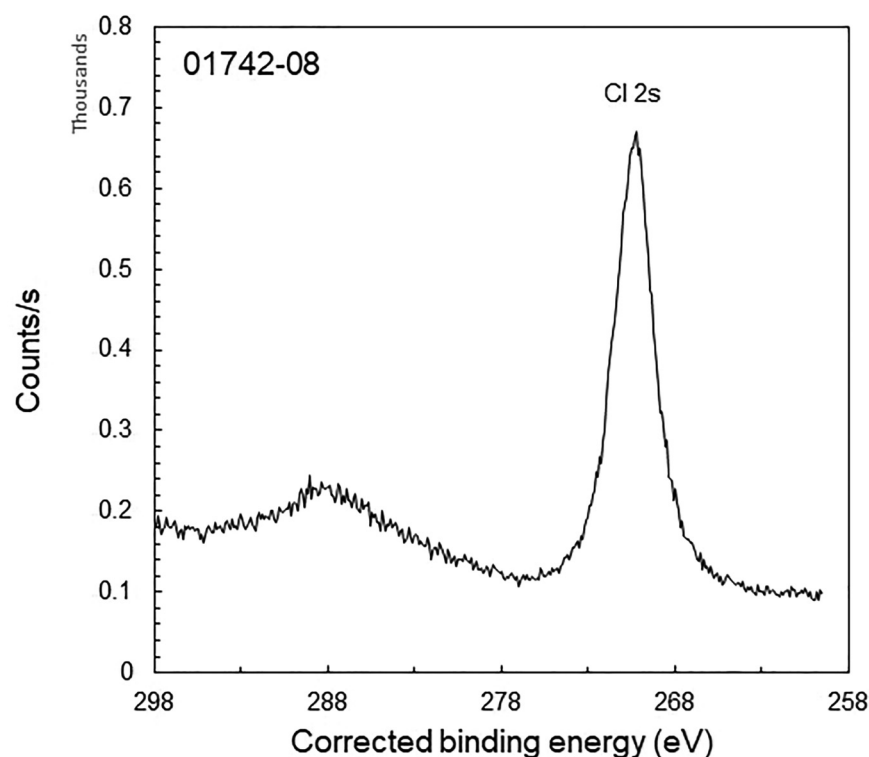
Instrument: ULVAC-PHI Quantes
 Excitation Source: Cr K_{α} monochromatic
 Source Energy: 5414.8 eV
 Source Strength: 43 W
 Source Size: $0.1 \times 0.1 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 22°
 Emission Angle: 45°
 Analyzer Pass Energy: 112 eV
 Analyzer Resolution: 0.86 eV
 Total Signal Accumulation Time: 1200 s
 Total Elapsed Time: 1320 s
 Number of Scans: 40
 Effective Detector Width: 12.4 eV

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■ **Accession #:** 01742-07
 ■ **Host Material:** NaCl
 ■ **Technique:** XPS
 ■ **Spectral Region:** Cl 2p

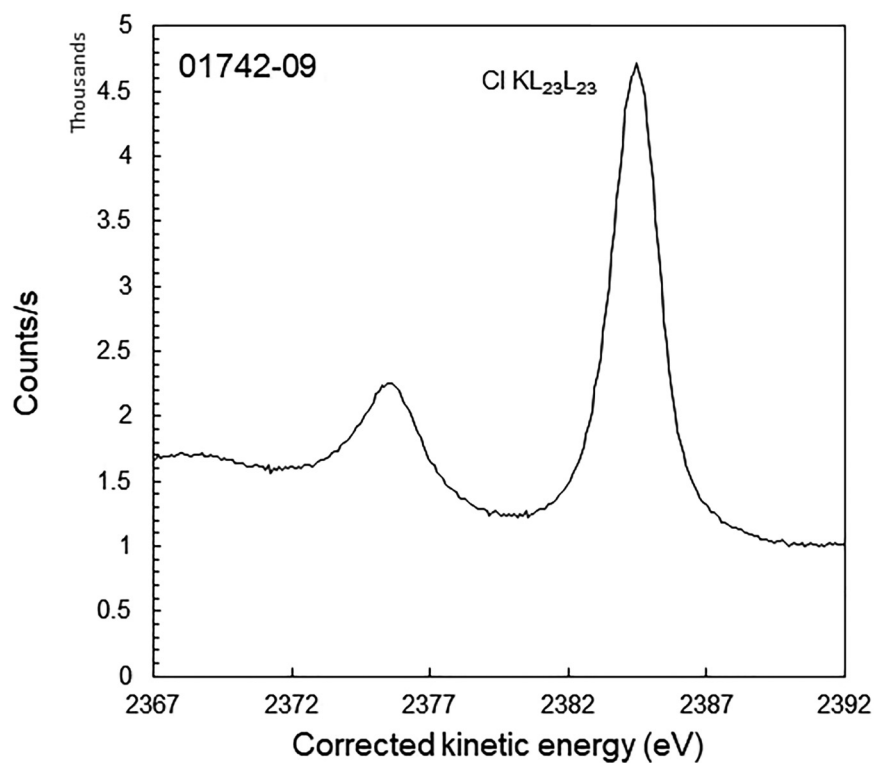
Instrument: ULVAC-PHI Quantes
 Excitation Source: Cr K_{α} monochromatic
 Source Energy: 5414.8 eV
 Source Strength: 43 W
 Source Size: $0.1 \times 0.1 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 22°
 Emission Angle: 45°
 Analyzer Pass Energy: 112 eV
 Analyzer Resolution: 0.86 eV
 Total Signal Accumulation Time: 2400 s
 Total Elapsed Time: 2650 s
 Number of Scans: 120
 Effective Detector Width: 12.4 eV



■ **Accession #:** 01742-08
 ■ **Host Material:** NaCl
 ■ **Technique:** XPS
 ■ **Spectral Region:** Cl 2s

Instrument: ULVAC-PHI Quantes
 Excitation Source: Cr K_{α} monochromatic
 Source Energy: 5414.8 eV
 Source Strength: 43 W
 Source Size: $0.1 \times 0.1 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 22°
 Emission Angle: 45°
 Analyzer Pass Energy: 112 eV
 Analyzer Resolution: 0.86 eV
 Total Signal Accumulation Time: 1760s
 Total Elapsed Time: 1940s
 Number of Scans: 44
 Effective Detector Width: 12.4 eV

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- Accession #: [01742-09](#)
- Host Material: NaCl
- Technique: XAES
- Spectral Region: Cl KLL

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: 300 s
 Total Elapsed Time: 3300 s
 Number of Scans: 120
 Effective Detector Width: 12.4 eV

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