

Carbon monoxide, CO(g), by high-resolution near-ambient-pressure X-ray photoelectron spectroscopy

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Near-ambient-pressure X-ray photoelectron spectroscopy (NAP-XPS) and X-ray induced Auger electron spectroscopy was used to characterize gas-phase carbon monoxide, CO(g). In this submission, we show the survey, valence band, O 1s, C 1s, O KLL Auger, and C KLL Auger spectra acquired using high-resolution synchrotron NAP-XPS with a photon energy of 642.65 eV.

Keywords: near-ambient-pressure X-ray photoelectron spectroscopy, NAP-XPS, XPS, carbon monoxide, CO, CO (g)

INTRODUCTION

Near-ambient-pressure X-ray photoelectron spectroscopy (NAP-XPS) has been used extensively to examine the evolution of catalyst materials under reaction conditions and provide insight into reaction pathways and intermediates.^{1,2} These studies are performed by illumination with lab-based light sources, including Mg K α (1253.6 eV) and Al K α (1486.7 eV), and synchrotron-based light sources, which have variable photon energies. The interpretation of the results can be difficult because of the observation of both gas-phase and surface species through the detection of both XPS and Auger electrons, which for observation by synchrotron-based light sources will change positions with respect to XPS peaks as the photon energy is adjusted. Further, the much higher photon intensity of synchrotron-based light sources allows for high-resolution spectra to be obtained, which can resolve features that cannot be observed with lab-based light sources. Therefore, it is desirable to characterize gas-phase reactant and product species using both synchrotron-based and lab-based NAP-XPS.³ Recently, common gas-phase reactants and products, including O₂(g) and CO₂(g), have been characterized using lab-based NAP-XPS.³⁻⁵

Carbon monoxide (CO) is an important molecule that is used industrially in the catalytic production of methanol and synthetic fuels, and also of considerable research interest is the CO oxidation to CO₂.⁶ This work presents a complete collection of the survey, valence band, O 1s, C 1s, O KLL Auger, and C KLL Auger spectra acquired from the illumination of gas-phase CO with a high-intensity synchrotron-based light source (642.65 eV) using NAP-XPS. Furthermore, this full set was collected with

identical beamline and analyzer settings for optimal comparison. Additionally, the O 1s and C 1s regions with higher resolution (2-eV pass energy) are vibrationally resolved⁷⁻¹² but are complicated by the presence of shake-up satellites. The vibrationally resolved levels of the O 1s and C 1s region are designated by v_x, where x is the integer value of the vibrational level.¹⁰ The valence band binding energies^{13,14}, XPS binding energies^{7-12,15-17} and Auger kinetic energies^{10,18} are in agreement with previous assignments. The satellite Auger lines of the O KLL and C KLL are designated by the nomenclature used by Moddeman et. al.¹⁸

The given analyzer resolution is based on the full-width at half maximum of the Ag 3d_{5/2} collected with the same beamline and analyzer settings. As such, it reflects the total experimental resolution.

SPECIMEN DESCRIPTION (ACCESSION #01629)

Host Material: Carbon monoxide, CO(g)

CAS Registry #: 630-08-0

Host Material Characteristics: homogeneous; gas; amorphous; dielectric; inorganic compound; Other

Chemical Name: Carbon monoxide, CO(g)

Source: Matheson research purity (99.999%)

Host Composition: Carbon monoxide, CO(g)

Form: Gas

Structure: CO, C=O

Accession#: 01629

Technique: XPS, XAES

Host Material: Carbon monoxide, CO(g)

Instrument: SPECS Phoibos 150R6 NAP

Major Elements in Spectra: C, O

Minor Elements in Spectra: N/A

Published Spectra: 8

Spectra in Electronic Record: 8

Spectral Category: reference

^aElectronic mail: author@institution.edu

History & Significance: CO is an important reactant in the production of methanol and synthetic fuels.

As Received Condition: Compressed gas cylinder

Analyzed Region: Carbon monoxide gas molecules encountered by the X-ray beam

Ex Situ Preparation/Mounting: N/A

In Situ Preparation: A Ni-carbonyl trap was used to ensure pure CO was exposed to the chamber.

Charge Control: 266 Pa CO

Temp. During Analysis: 300 K

Pressure During Analysis: 266 Pa

Pre-analysis Beam Exposure: 30 s

INSTRUMENT DESCRIPTION

Manufacturer and Model: SPECS Phoibos 150R6 NAP

Analyzer Type: spherical sector

Detector: Surface Concept 1D-DLD detector (model number: 1D-DLD64_2-150HV)

Number of Detector Elements: 100

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

■Spectrometer

Analyzer Mode: constant pass energy

Throughput ($T=E^N$): $N = -1$

Excitation Source Window: 100-nm-thick SiN_x coated with 5 nm Cr and 10 nm Au (SPI supplies)

Excitation Source: Elliptically polarizing undulator at beamline 23-ID-2 of the National Synchrotron Light Source II¹⁹

Source Energy: 642.65 eV

Source Strength: $10^{12} - 10^{13}$ photons/s

Source Beam Size: $16 \mu\text{m} \times 70 \mu\text{m}$

Signal Mode: multichannel direct

■Geometry

Incident Angle: N/A

Source-to-Analyzer Angle: 70°

Emission Angle: N/A

Specimen Azimuthal Angle: N/A

Acceptance Angle from Analyzer Axis: 22°

Analyzer Angular Acceptance Width: 44°

DATA ANALYSIS METHOD

Energy Scale Correction: The energy scale was corrected for the work function of the detector and the photon energy. The work function of the detector was determined by setting the kinetic energy of the Ag M₄NN transition of a clean reference Ag(111) sample to 353.38 eV.²⁰ The photon energy was determined by setting the Ag3d_{5/2} binding energy of a reference Ag(111) sample to 368.20 eV. This correction procedure yields the Fermi level of a reference Ag(111) crystal to be 0.00 eV.

Recommended Energy Scale Shift: 0 eV.

Peak Shape and Background Method: N/A

Quantitation Method: N/A

ACKNOWLEDGMENTS

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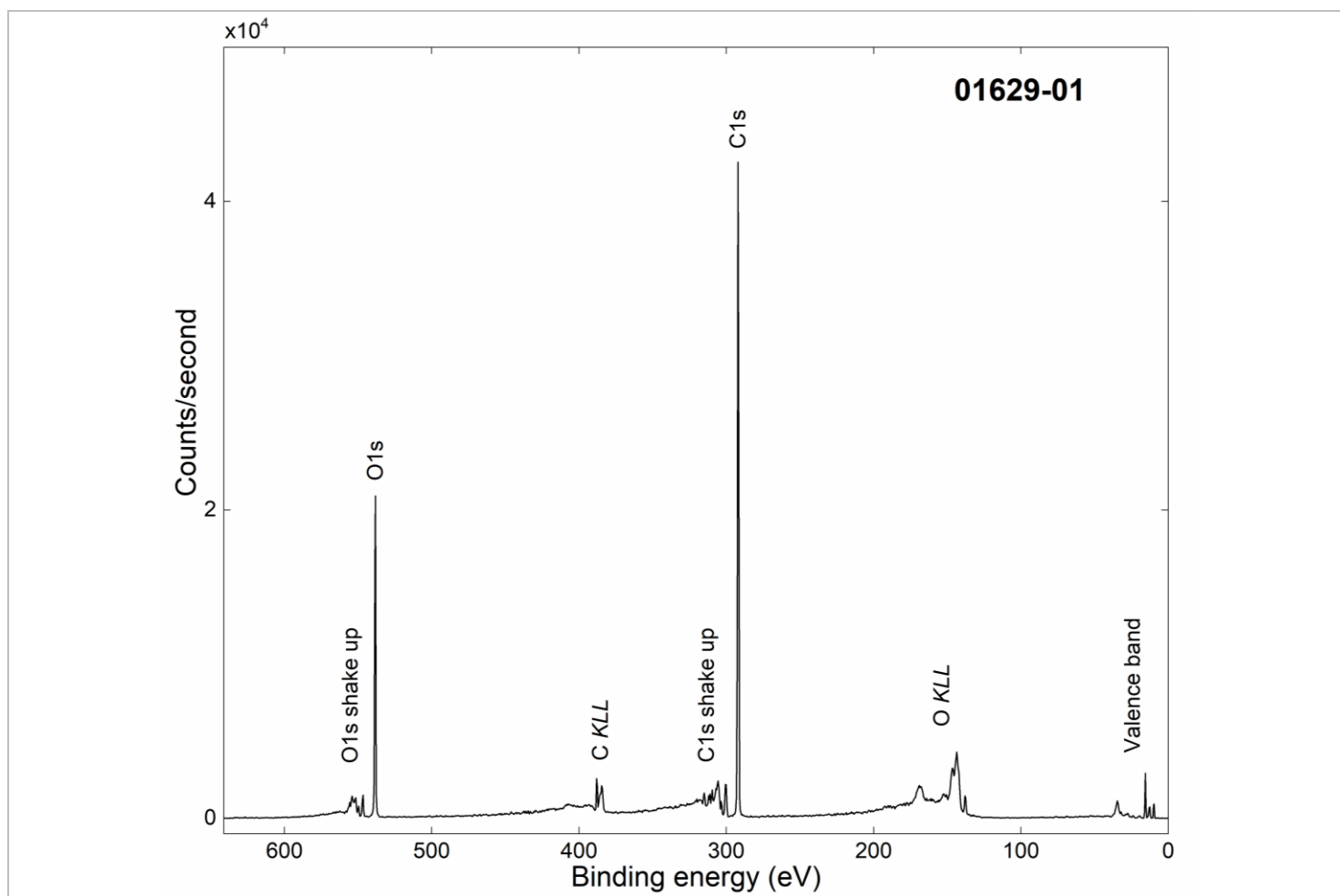
SPECTRAL FEATURES TABLE							
Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV x cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01629-01	Valence band	0-50	CO(g)
01629-01	O KLL	499.2 ^a	CO(g)
01629-01	C 1s	291.8	CO(g)
01629-01	C KLL	254.8 ^a	CO(g)
01629-01	O 1s	538.3	CO(g)
01629-02	Valence band	9.80	CO(g), 3σ
01629-02	Valence band	12.75	CO(g), 1π
01629-02	Valence band	15.45	CO(g), 2σ
01629-02	Valence band	34.55	CO(g), 1σ
01629-03	O KLL	514.70 ^a	CO(g), A-8
01629-03	O KLL	510.75 ^a	CO(g), A-10
01629-03	O KLL	504.75 ^a	CO(g), B-1
01629-03	O KLL	500.45 ^a	CO(g), B-4
01629-03	O KLL	499.20 ^a	CO(g), B-5
01629-03	O KLL	496.00 ^a	CO(g), B-7
01629-03	O KLL	492.20 ^a	CO(g), B-9
01629-03	O KLL	489.65 ^a	CO(g), B-11
01629-03	O KLL	474.00 ^a	CO(g), C-2
01629-04	C 1s	300.3	CO(g), shake up
01629-04	C 1s	291.9	CO(g)
01629-05	C 1s	291.82	CO(g), v ₀
01629-05	C 1s	292.11	CO(g), v ₁
01629-05	C 1s	292.41	CO(g), v ₂
01629-06	C KLL	258.35 ^a	CO(g), B-1
01629-06	C KLL	256.80 ^a	CO(g), B-2
01629-06	C KLL	254.75 ^a	CO(g), B-3
01629-07	O 1s	546.6	CO(g), shake up
01629-07	O 1s	538.3	CO(g)
01629-08	O 1s	538.25	CO(g), v ₀
01629-08	O 1s	538.46	CO(g), v ₁

^aPeak energy value indicated as 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}	368.20	0.46 eV	1177774.1

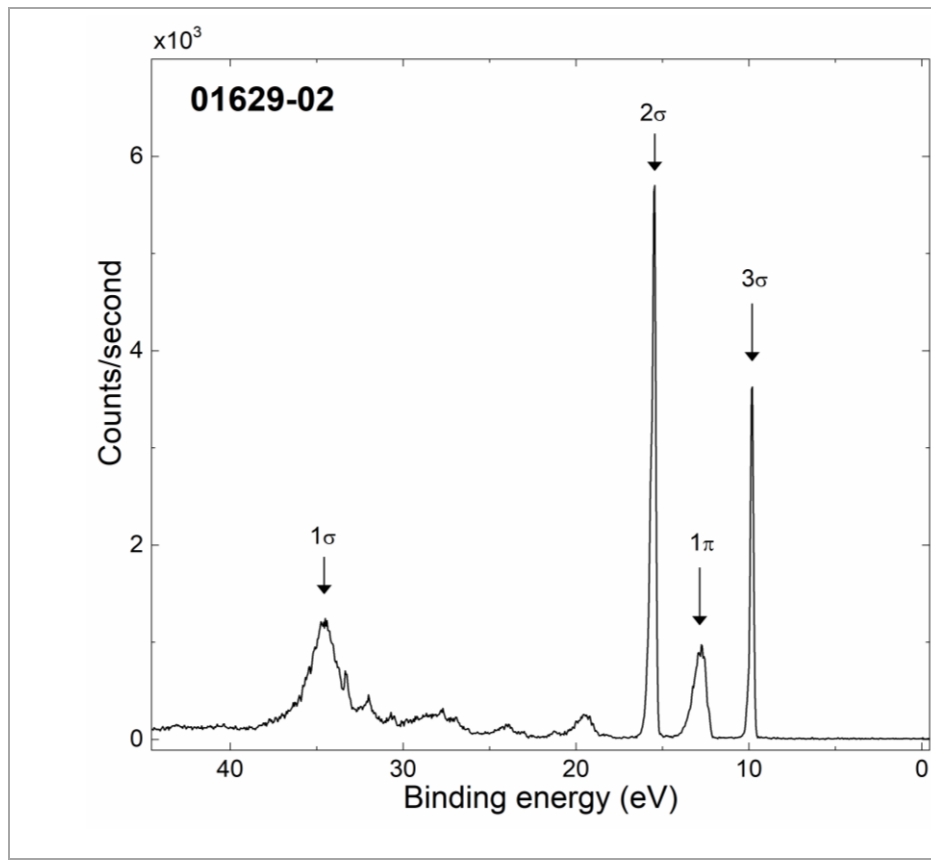
GUIDE TO FIGURES						
Spectrum (Accession) #	Spectral Region	Voltage Shift	Multiplier	Baseline	Comment #	
01629-01	Survey	0	1	0	From CO(g)	
01629-02	Valence band	0	1	0	From CO(g)	
01629-03	O KLL	0	1	0	From CO(g)	
01629-04	C 1s	0	1	0	From CO(g)	
01629-05	C 1s	0	1	0	From CO(g)	
01629-06	C KLL	0	1	0	From CO(g)	
01629-07	O 1s	0	1	0	From CO(g)	
01629-08	O 1s	0	1	0	From CO(g)	

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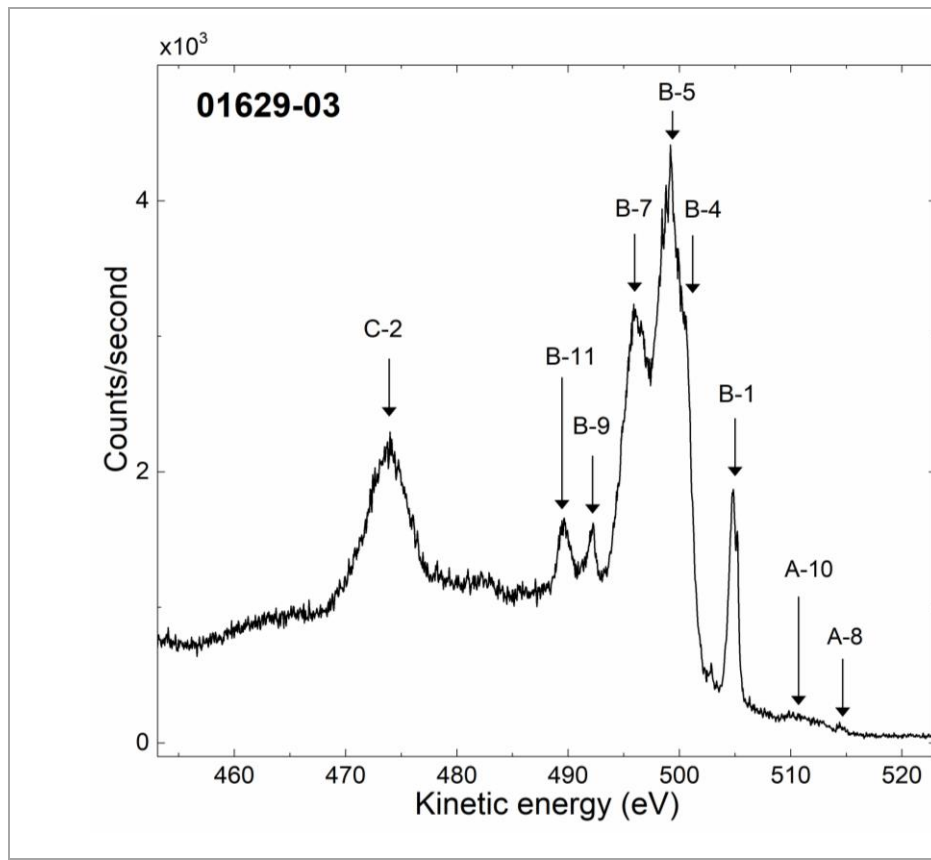
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Host Material	Carbon monoxide, CO(g)
Technique	XPS/XAES
Spectral Region	Survey
Instrument	SPECS Phoibos 150R6 NAP
Excitation Source	Elliptically polarizing undulator at beamline 23-ID-2 of the National Synchrotron Light Source II
Source Energy	642.65 eV
Source Strength	$10^{12} - 10^{13}$ photon/s
Source Size	$16 \mu\text{m} \times 70 \mu\text{m}$
Analyzer Type	spherical sector analyzer
Incident Angle	N/A
Emission Angle	N/A
Analyzer Pass Energy	10 eV
Analyzer Resolution	0.46 eV
Total Signal Accumulation Time	260 s
Total Elapsed Time	300 s
Number of Scans	2
Effective Detector Width	50 eV

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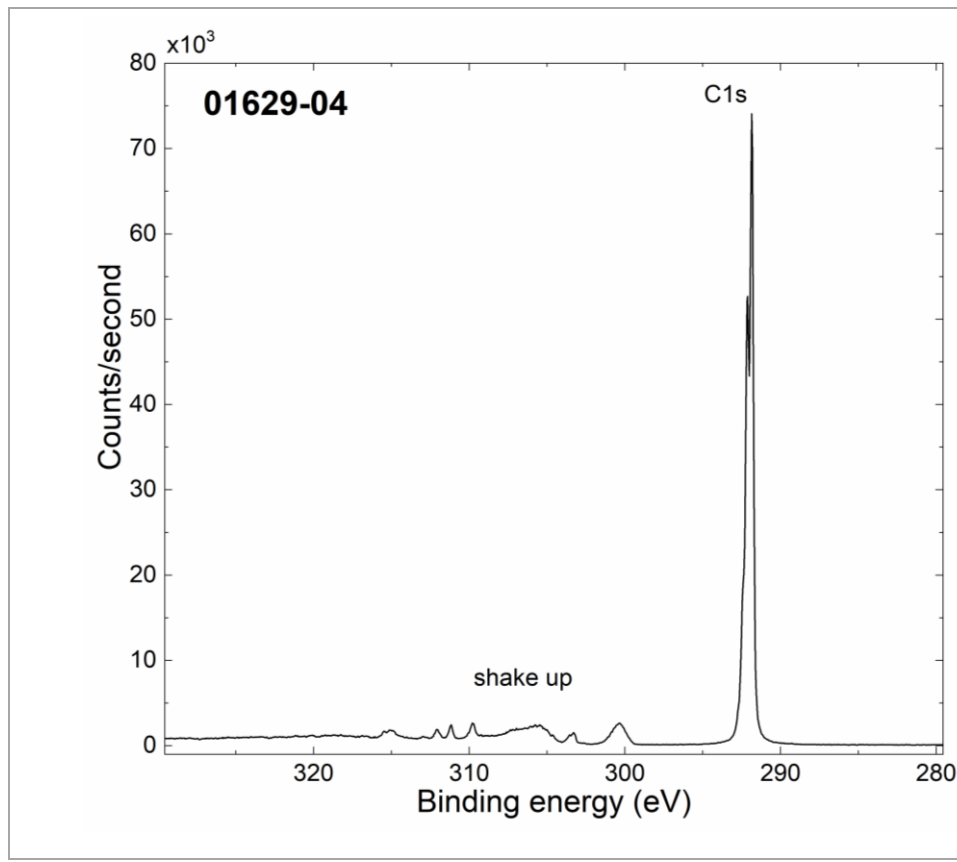
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 ■ **Technique:** XPS
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 Source Energy: 642.65 eV
 Source Strength: $10^{12} - 10^{13}$ photon/s
 Source Size: $16 \mu\text{m} \times 70 \mu\text{m}$
 Analyzer Type: spherical sector
 Incident Angle: N/A
 Emission Angle: N/A
 Analyzer Pass Energy 10 eV
 Analyzer Resolution: 0.46 eV
 Total Signal Accumulation Time: 450 s
 Total Elapsed Time: 550 s
 Number of Scans: 5
 Effective Detector Width: 5 eV

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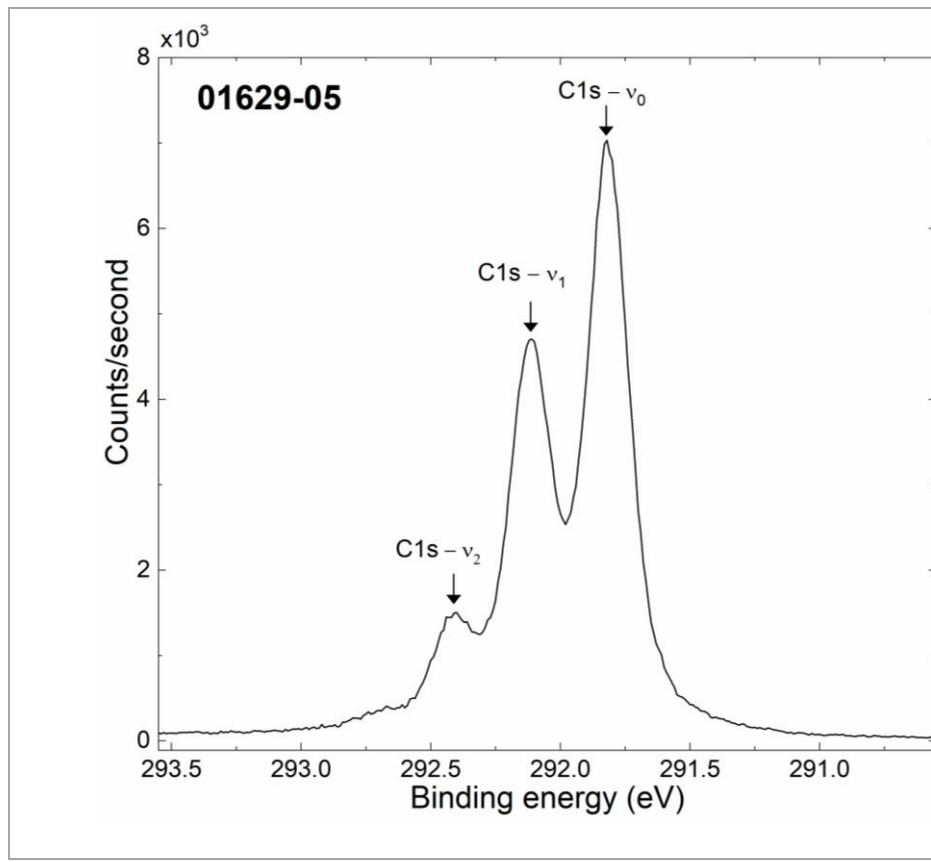
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 ■ **Technique:** XAES
 ■ **Spectral Region:** O KLL
 Instrument: Specs Phoibos 150R6 NAP
 Excitation Source: Elliptically polarizing undulator at beamline 23-ID-2 of the National Synchrotron Light Source II
 Source Energy: 642.65 eV
 Source Strength: $10^{12} - 10^{13}$ photon/s
 Source Size: $16 \mu\text{m} \times 70 \mu\text{m}$
 Analyzer Type: spherical sector
 Incident Angle: N/A
 Emission Angle: N/A
 Analyzer Pass Energy 10 eV
 Analyzer Resolution: 0.46 eV
 Total Signal Accumulation Time: 700 s
 Total Elapsed Time: 800 s
 Number of Scans: 5
 Effective Detector Width: 5 eV

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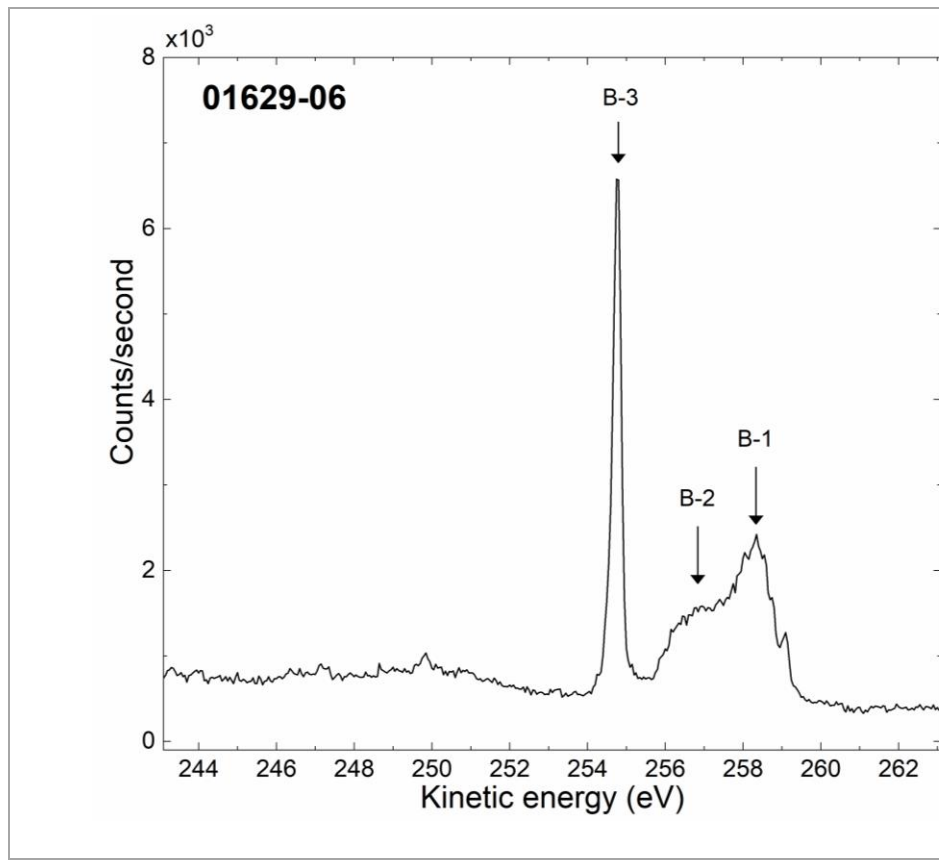
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 ■ **Technique:** XPS
 ■ **Spectral Region:** C 1s
 Instrument: Specs Phoibos 150R6 NAP
 Excitation Source: Elliptically polarizing undulator at beamline 23-ID-2 of the National Synchrotron Light Source II
 Source Energy: 642.65 eV
 Source Strength: $10^{12} - 10^{13}$ photon/s
 Source Size: $16 \mu\text{m} \times 70 \mu\text{m}$
 Analyzer Type: spherical sector
 Incident Angle: N/A
 Emission Angle: N/A
 Analyzer Pass Energy 10 eV
 Analyzer Resolution: 0.46 eV
 Total Signal Accumulation Time: 500 s
 Total Elapsed Time: 600 s
 Number of Scans: 5
 Effective Detector Width: 5 eV

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Accession #: 01629-05
Host Material: Carbon monoxide, CO(g)
Technique: XPS
Spectral Region: C 1s
 Instrument: Specs Phoibos 150R6 NAP
 Excitation Source: Elliptically polarizing undulator at beamline 23-ID-2 of the National Synchrotron Light Source II
 Source Energy: 642.65 eV
 Source Strength: $10^{12} - 10^{13}$ photon/s
 Source Size: $16 \mu\text{m} \times 70 \mu\text{m}$
 Analyzer Type: spherical sector
 Incident Angle: N/A
 Emission Angle: N/A
 Analyzer Pass Energy 2 eV
 Analyzer Resolution: 0.42 eV
 Total Signal Accumulation Time: 300 s
 Total Elapsed Time: 500 s
 Number of Scans: 10
 Effective Detector Width: 1 eV

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■ **Accession #:** 01629-06

■ **Host Material:** Carbon monoxide, CO(g)

■ **Technique:** XAES

■ **Spectral Region:** C KLL

Instrument: Specs Phoibos 150R6 NAP

Excitation Source: Elliptically polarizing undulator at beamline 23-ID-2 of the National Synchrotron Light Source II

Source Energy: 642.65 eV

Source Strength: $10^{12} - 10^{13}$ photon/s

Source Size: $16 \mu\text{m} \times 70 \mu\text{m}$

Analyzer Type: spherical sector

Incident Angle: N/A

Emission Angle: N/A

Analyzer Pass Energy 10 eV

Analyzer Resolution: 0.46 eV

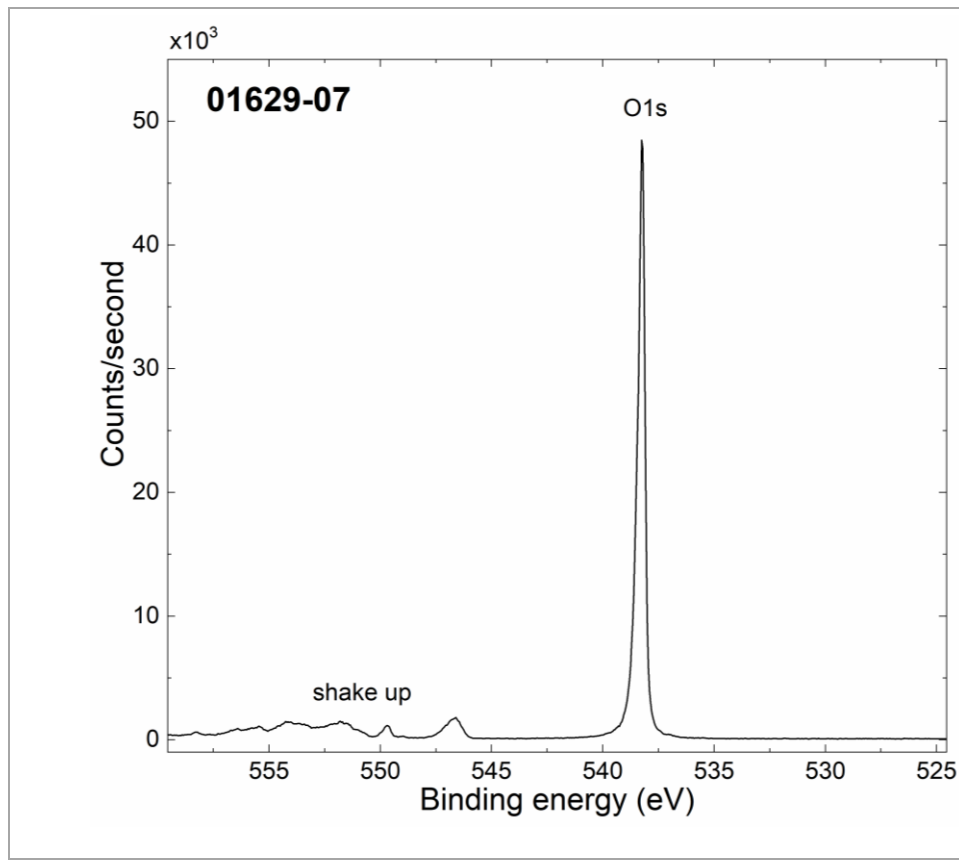
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Total Elapsed Time: 300 s

Number of Scans: 5

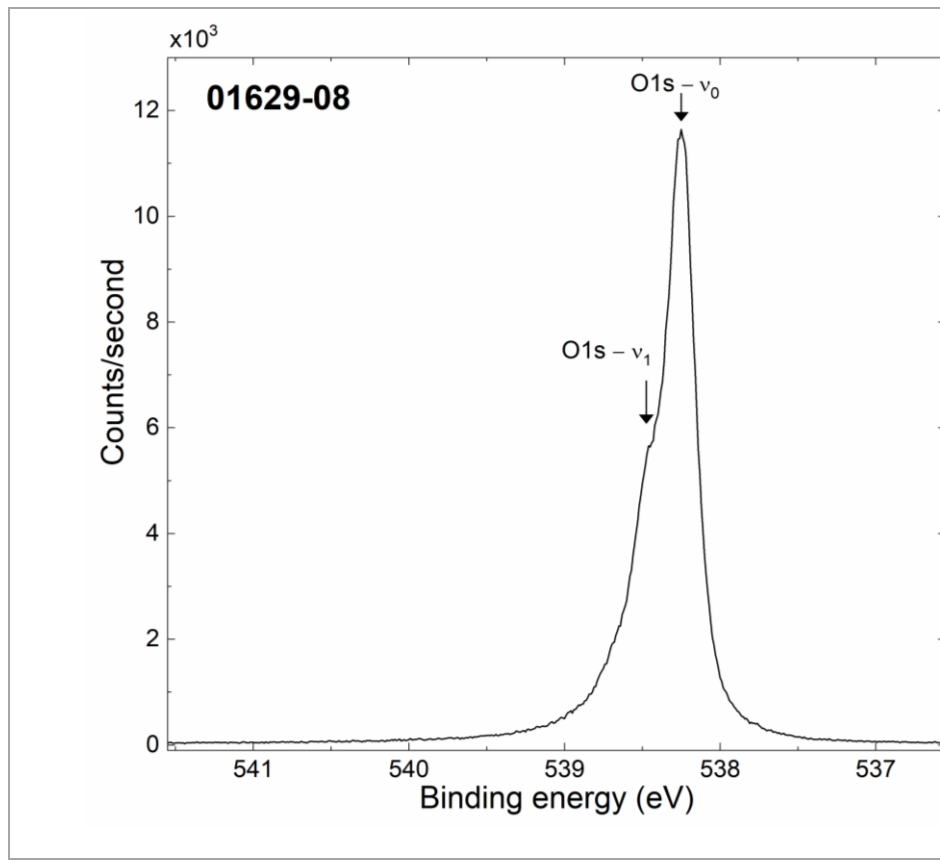
Effective Detector Width: 5 eV

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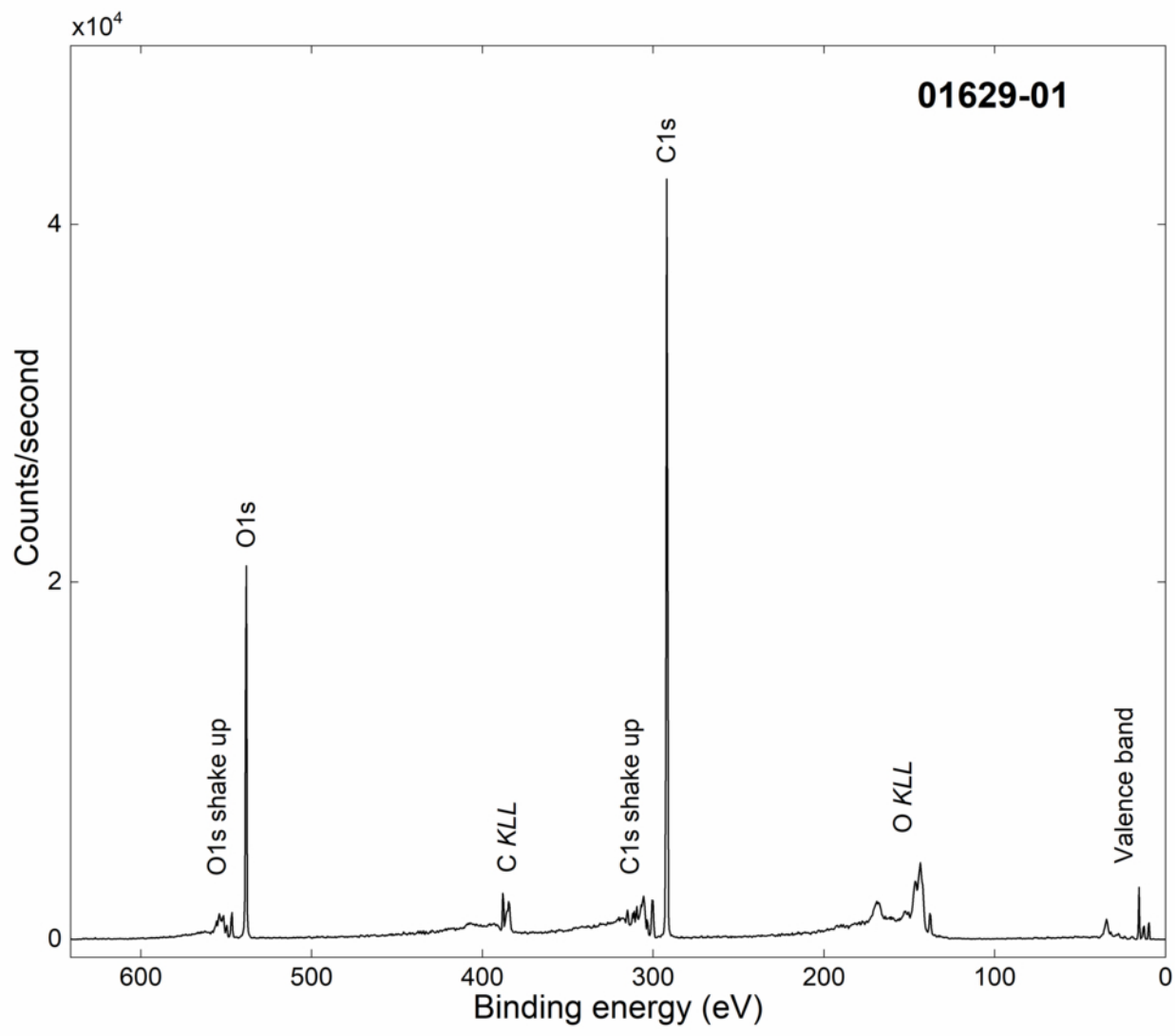
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 ■ **Technique:** XPS
 ■ **Spectral Region:** O 1s
 Instrument: Specs Phoibos 150R6 NAP
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 Source Energy: 642.65 eV
 Source Strength: $10^{12} - 10^{13}$ photon/s
 Source Size: $16 \mu\text{m} \times 70 \mu\text{m}$
 Analyzer Type: spherical sector
 Incident Angle: N/A
 Emission Angle: N/A
 Analyzer Pass Energy 10 eV
 Analyzer Resolution: 0.46 eV
 Total Signal Accumulation Time: 350 s
 Total Elapsed Time: 550 s
 Number of Scans: 10
 Effective Detector Width: 5 eV

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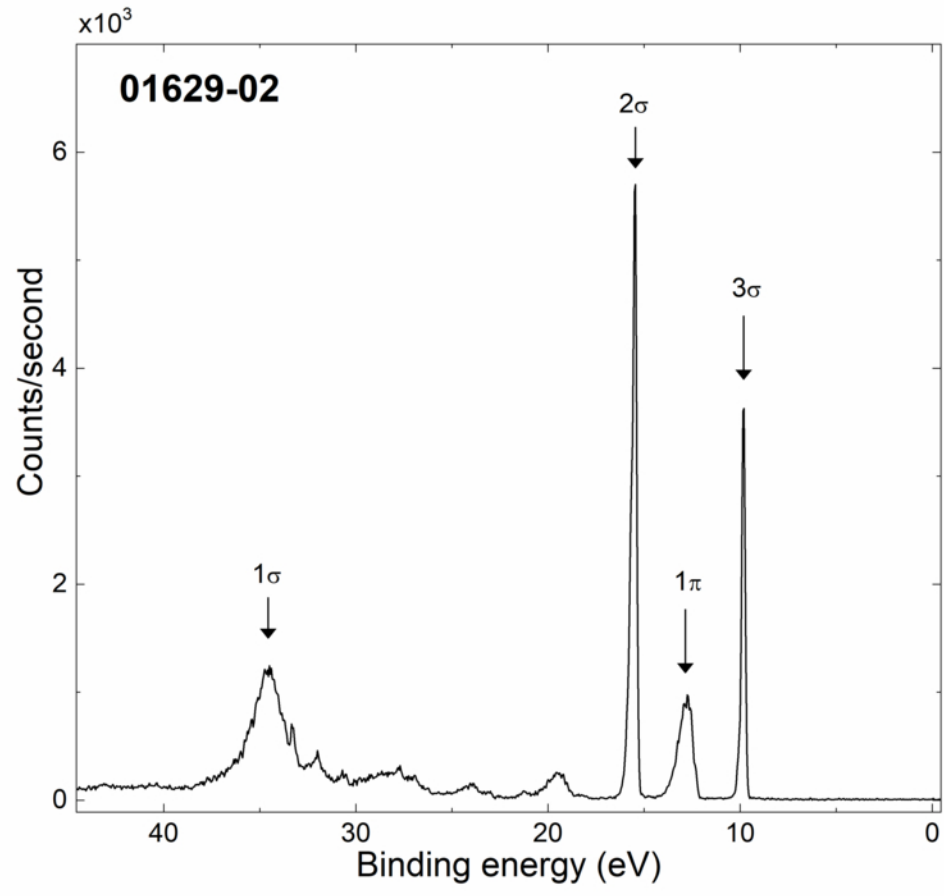


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Host Material: Carbon monoxide, CO(g)
Technique: XPS
Spectral Region: O 1s
 Instrument: Specs Phoibos 150R6 NAP
 Excitation Source: Elliptically polarizing undulator at beamline 23-ID-2 of the National Synchrotron Light Source II
 Source Energy: 642.65 eV
 Source Strength: $10^{12} - 10^{13}$ photon/s
 Source Size: $16 \mu\text{m} \times 70 \mu\text{m}$
 Analyzer Type: spherical sector
 Incident Angle: N/A
 Emission Angle: N/A
 Analyzer Pass Energy 2 eV
 Analyzer Resolution: 0.42 eV
 Total Signal Accumulation Time: 500 s
 Total Elapsed Time: 700 s
 Number of Scans: 10
 Effective Detector Width: 1 eV

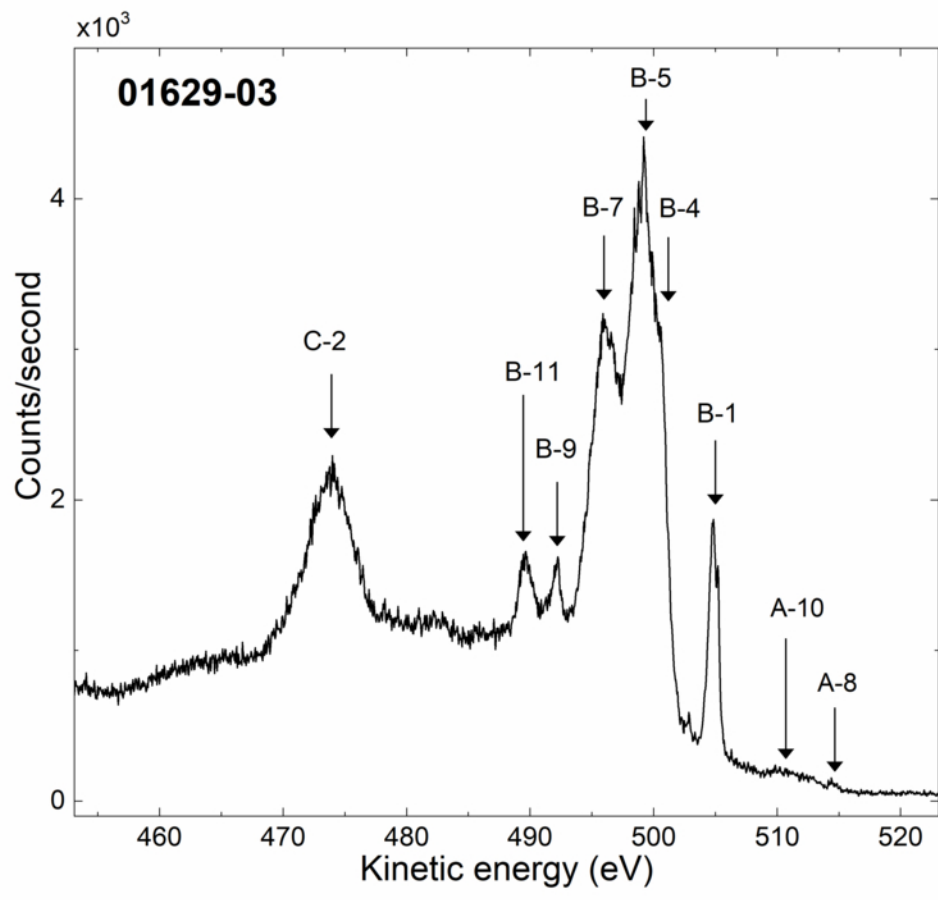
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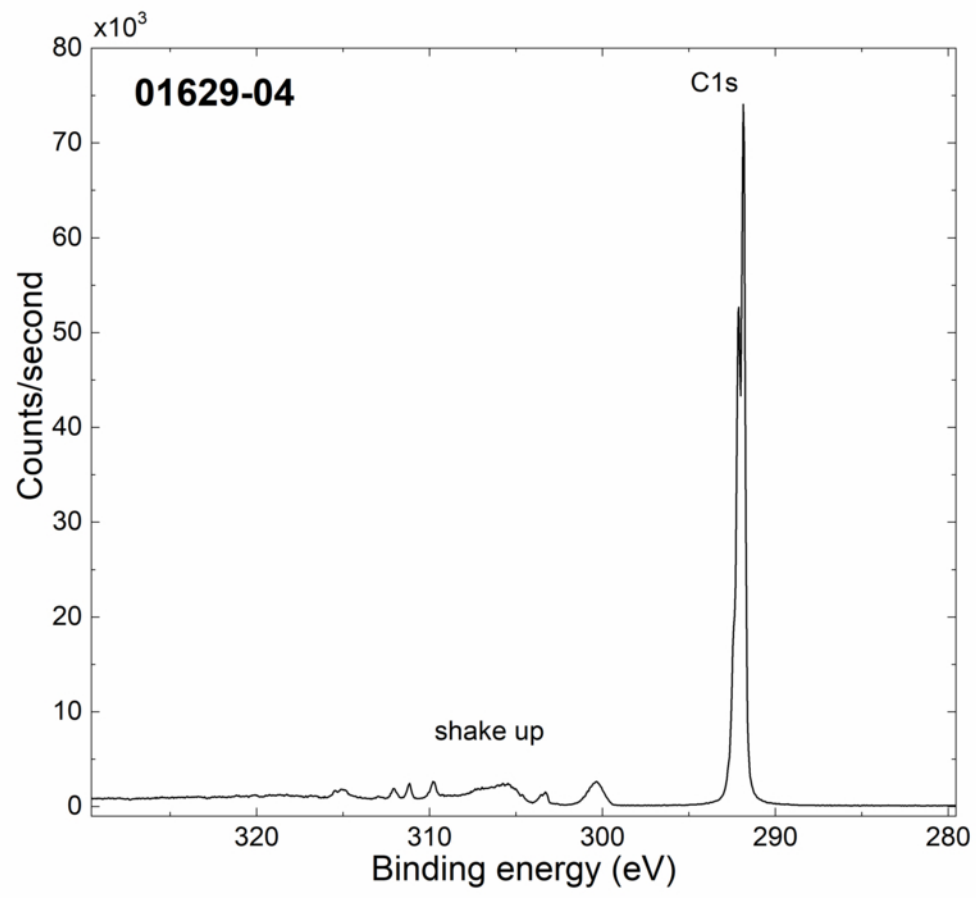
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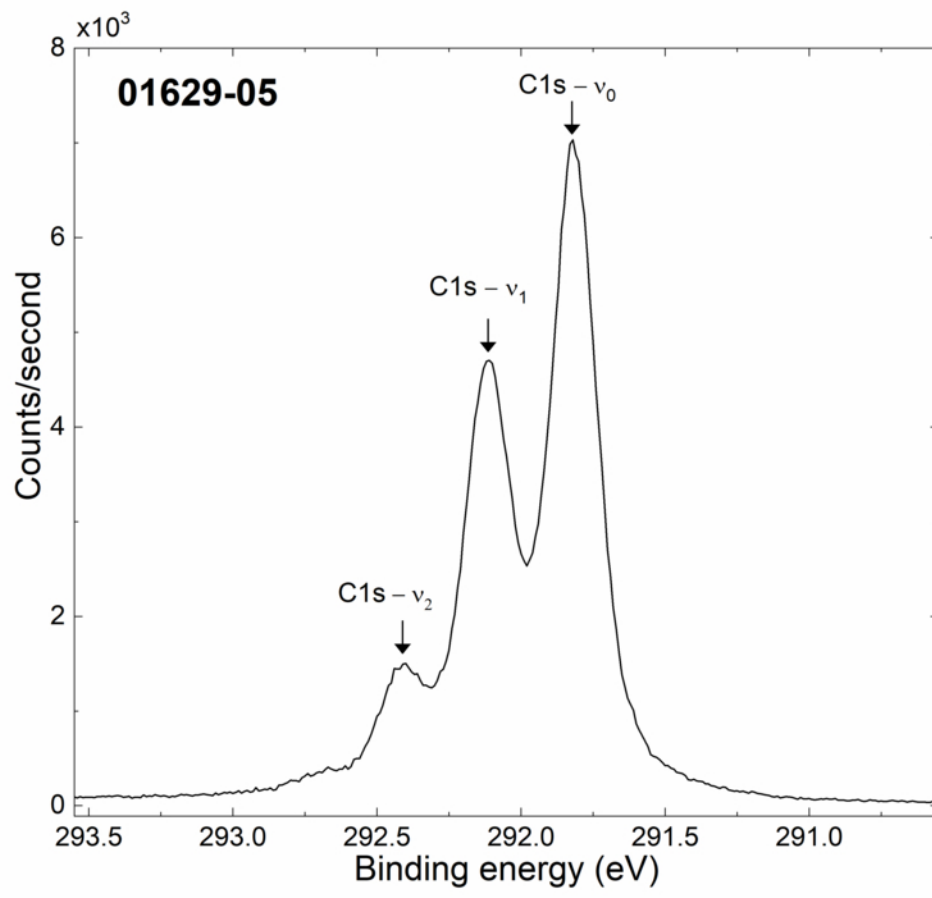
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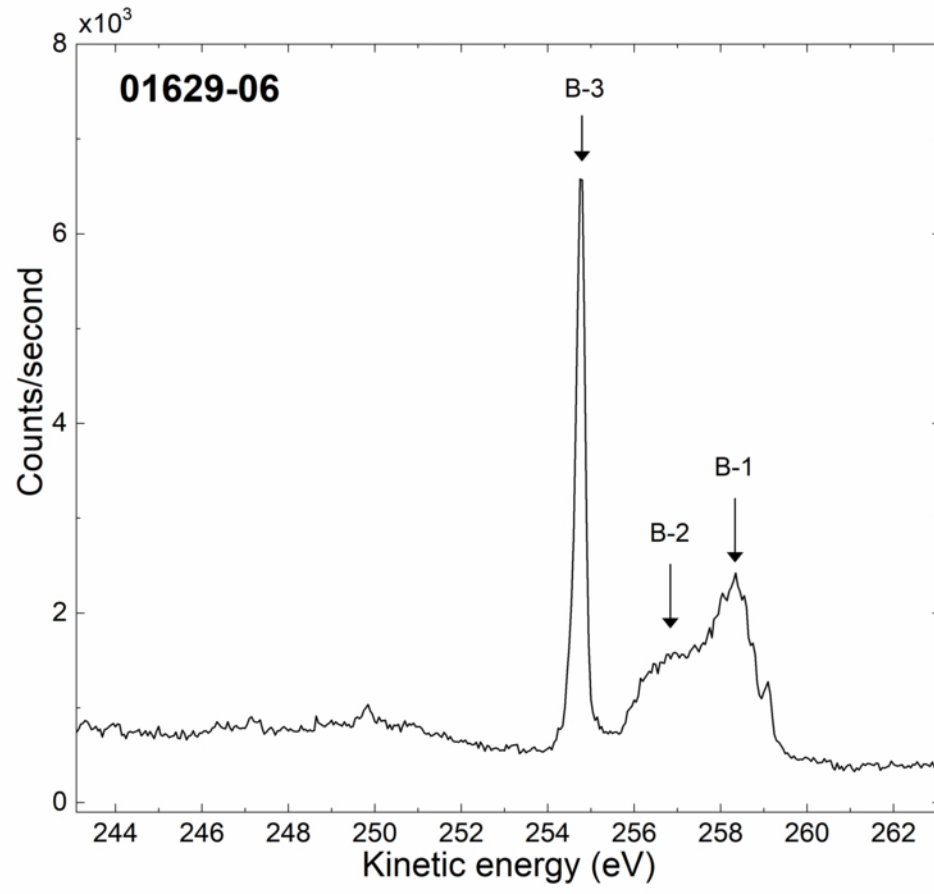
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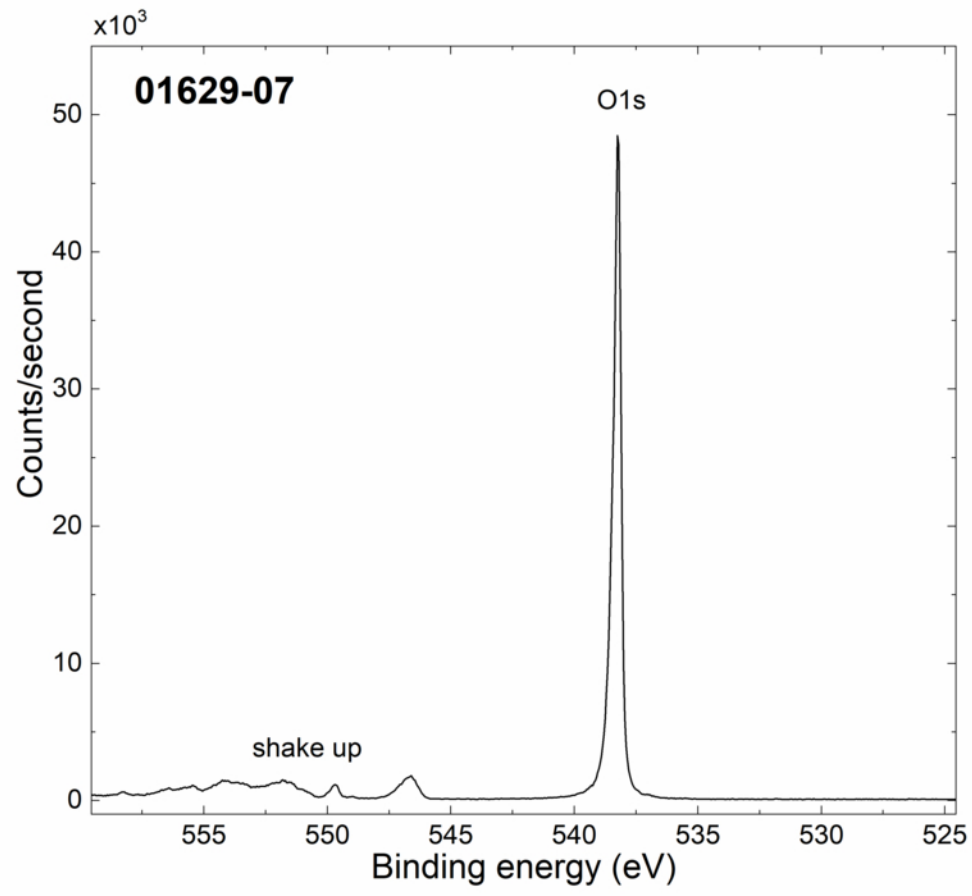
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