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High Resolution XPS Study of a Thin CoO(111) Film Grown on Co(0001)

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Accession #s 00298, 00299
Technique: XPS
Host Material: #00298: Co(0001) metal; #00299: CoO(111) on Co
Instrument: Leybold-Heraeus EA 11
Major Elements in Spectrum: Co, O
Minor Elements in Spectrum: none
Printed Spectra: 6
Spectra in Electronic Record: 6
Spectral Category: comparison
Original Submission: 12/04/95
Accepted for Publication: 8/13/96

Transition metal oxides are often used as the active components in heterogenous catalysis. Therefore the investigation of single crystal oxides as model systems is important to understand the reaction mechanisms on a microscopic level. The reactivity of a (111) surface of ionic rocksalt type structures seems to be rather high as has been established for NiO(111). The ideal (111) surface is polar, and thus unstable, which means that stabilization mechanisms must exist. Here, high resolution XPS measurements of a thin epitaxial CoO(111) film grown on CO(0001) by exposing the surface to ≈10000 L O_2 are reported. © *1998 American Vacuum Society.*
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Keywords: *x-ray photoelectron spectroscopy; oxidation; cobalt oxide*

PACS: *79.60.Dp, 82.65.Jv, 82.80.Pv*

SPECIMEN DESCRIPTION (Accession #00298)

Host Material: Co(0001) metal

Host Material Characteristics: homogeneous; solid; single crystal; conductor; metal

Chemical Name: cobalt

Host Composition: Co

Form: single crystal

Structure: (0001) hexagonal close packed

As Received Condition: single crystal as grown

Analyzed Region: (0001) surface

***Ex Situ* Preparation/Mounting:** single crystal cut in (0001) direction and polished, then spot-welded on the sample holder

***In Situ* Preparation:** ion sputter cleaning and annealing

Charge Control: no charge control necessary as host material is metallic

Temp. During Analysis: 300 K

Pressure During Analysis: $<1\times10^{-7}$ Pa

SPECIMEN DESCRIPTION (Accession #00299)

Host Material: CoO(111) on Co

Host Material Characteristics: homogeneous; solid; single crystal; dielectric; inorganic compound; thin film

Chemical Name: cobalt oxide

Host Composition: Co, O

Form: thin film

Structure: CoO(111)

History & Significance: The analyzed sample was grown by oxidation of Co(0001) for 3 h with a background pressure of 10^{-6} mbar. During this time the sample was slowly heated up to 450 K. After this it was annealed for 1 h without oxygen background pressure (Ref. 1).

As Received Condition: not specified

Analyzed Region: oxidized host material

***Ex Situ* Preparation/Mounting:** single crystal cut in (0001) direction and polished, then spot-welded to the manipulator

***In Situ* Preparation:** no additional preparation after oxide growth (see History and Significance)

Charge Control: no charge control necessary as host material is metallic

Temp. During Analysis: 300 K

Pressure During Analysis: $<1\times10^{-7}$ Pa

INSTRUMENT DESCRIPTION

Manufacturer and Model: Leybold-Heraeus EA 11

Analyzer Type: spherical sector

Detector: 2 multichannel plates

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

■ *Spectrometer*

Analyzer Mode: constant pass energy

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

Excitation Source Window: 1.5 μm Al window

Excitation Source: Al K_α monochromatic

Source Energy: 1486.6 eV

Source Strength: 450 W

Analyzer Width: 3000 μm × 3000 μm

Signal Mode: multichannel direct

■ *Geometry*

Incident Angle: 45°

Source to Analyzer Angle: 45°

Emission Angle: 0°

Specimen Azimuthal Angle: 0°

Acceptance Angle from Analyzer Axis: 1.5°

Analyzer Angular Acceptance Width: 3° × 3°

■ *Ion Gun*

Manufacturer and Model: Leybold-Heraeus IQE 12/38

Energy: 500 eV

Current: 10 μA

Current Measurement Method: biased stage

Sputtering Species: Ne

Raster Size: 10000 μm × 10000 μm

Incident Angle: 45°

Polar Angle: 45°

Azimuthal Angle: 0°

DATA ANALYSIS METHOD

Energy Scale Correction: energy scale calibration to Fermi level of Co metal

ACKNOWLEDGMENTS

Several agencies have supported our work: Deutsche Forschungsgemeinschaft, Ministerium fur Wissenschaft und Forschung des Landes NRW, and Fonds der Chemischen Industrie.

REFERENCES

1. M. Hassel and H.-J. Freund, Surf. Sci. **325**, 163 (1995).

SPECTRAL FEATURES TABLE

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area	Sensitivity Factor	Concentration (at. %)	Peak Assignment
00299-02	O 1*s*	530.0	1.0	···	···	···	CoO(111)
00299-02	O 1*s*	531.8	1.3	···	···	···	OH groups
00299-03	Co $2p_{1/2}$	796.8	4.5	···	···	···	CoO(111)
00299-03	Co $2p_{3/2}$	780.6	5.0	···	···	···	CoO(111)
00299-04	Co 3*s*	101.4	5.0	···	···	···	CoO(111)
00299-04	Co 3*p*	59.4	4.0	···	···	···	CoO(111)
00299-05	Co/O valence band	1.4	5.0	···	···	···	CoO(111)

ANALYZER CALIBRATION TABLE

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
···	···	···	···	···	···	···

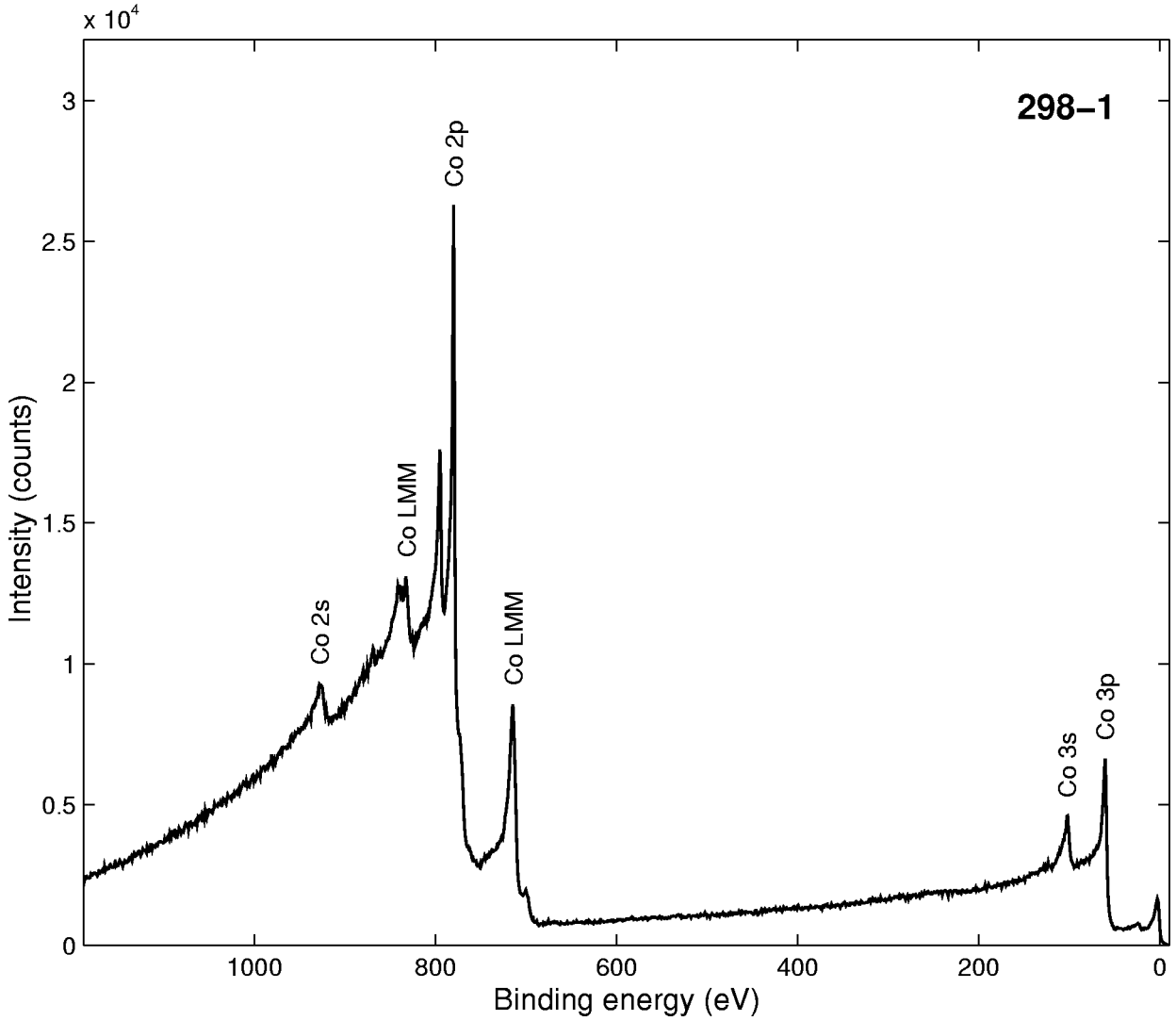
Comment to Analyzer Calibration Table: The energy scale was calibrated by determining the Fermi edge of the Co(0001) substrate.

GUIDE TO FIGURES

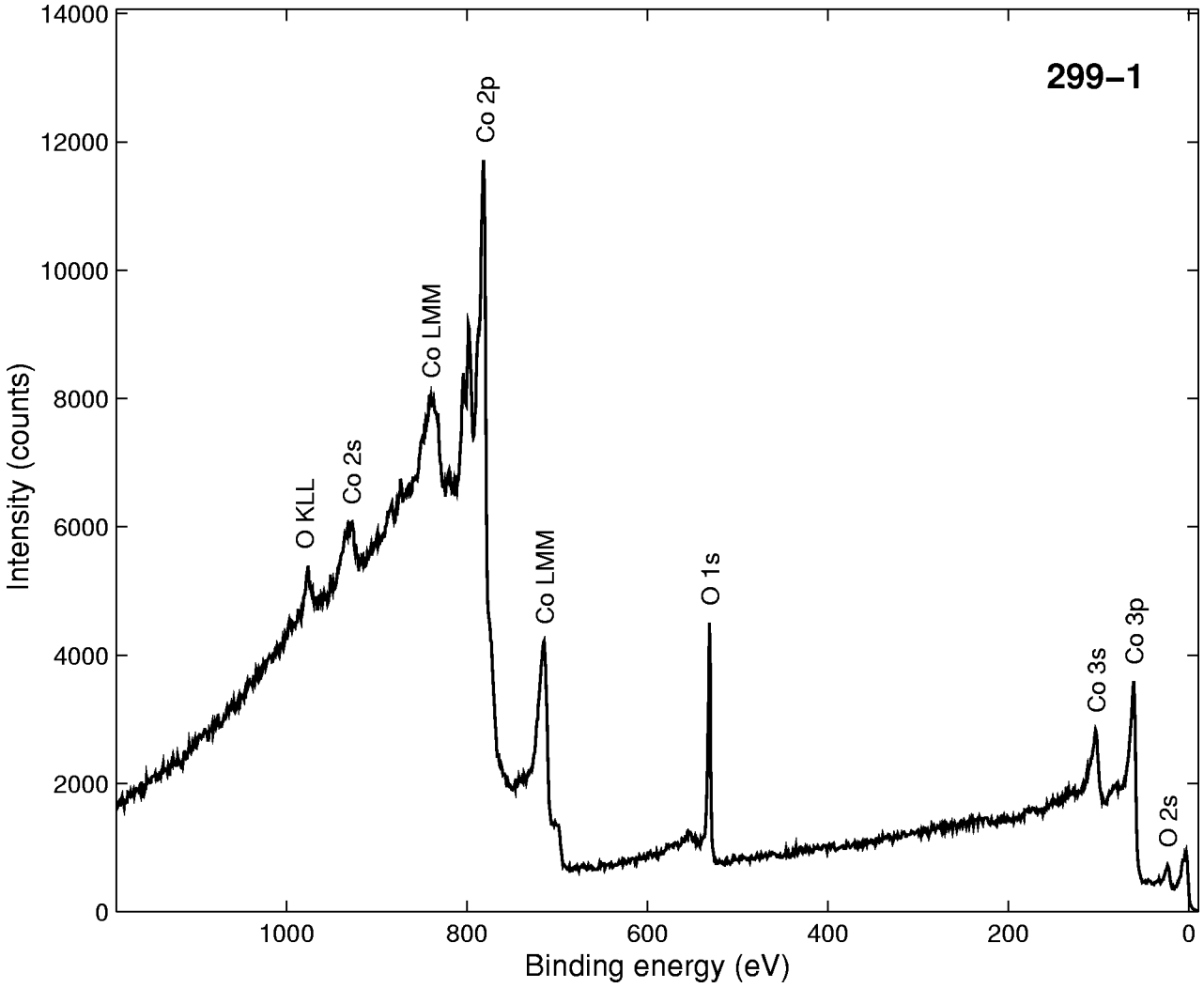
Spectrum (Accession) #	Sample Voltage*	Multiplier	Baseline	Comment #
298-1	0	1.000	0	-
299-1	0	1.000	0	1
298-2	0	1.000	0	1
299-3	0	1.000	0	1
299-4	0	1.000	0	1
299-5	0	1.000	0	1

* Sample voltage due to charging unless otherwise noted.

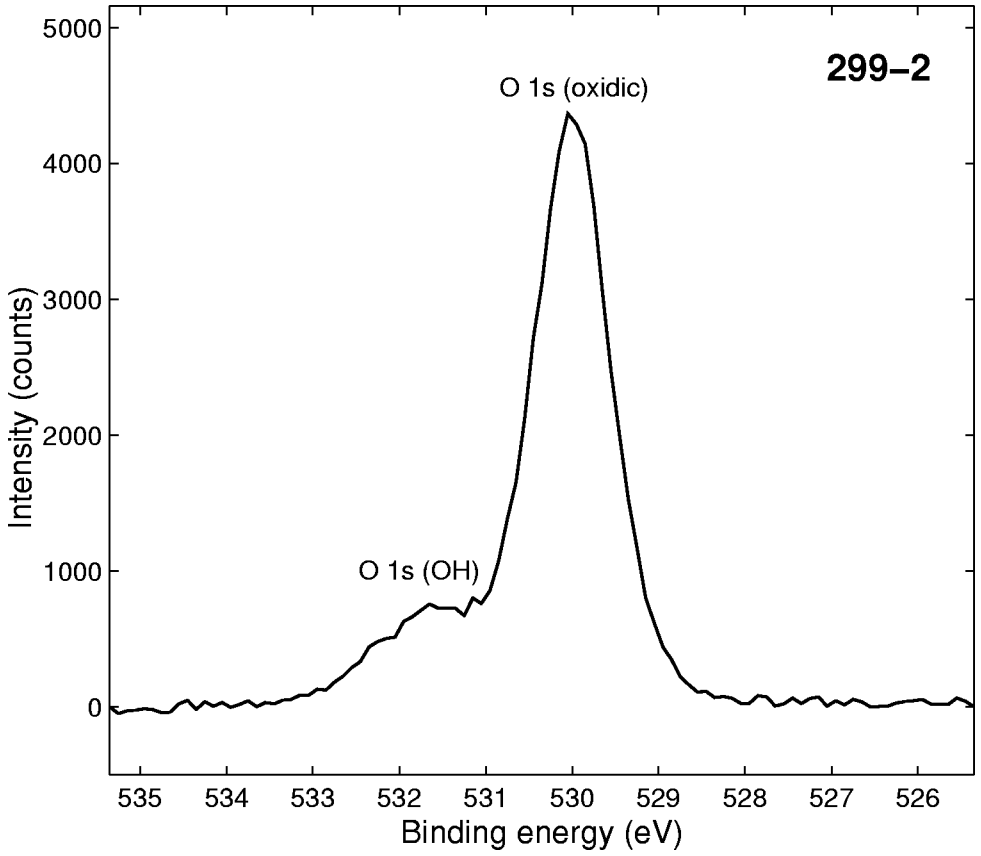
1. Peak energies were referenced to Fermi level of Co metal substrate which could be measured through thin oxide (see 299-5).



Accession #	00298-01
Host Material	Co(0001) metal
Technique	XPS
Spectral Region	survey
Instrument	Leybold-Heraeus EA 11
Excitation Source	Al K_α monochromatic
Source Energy	1486.6 eV
Source Strength	450 W
Source Size	not specified
Analyzer Type	spherical sector
Incident Angle	45°
Emission Angle	0°
Analyzer Retard Ratio	4
Analyzer Resolution	0.125 eV
Total Signal Accumulation Time	not specified
Total Elapsed Time	330 s
Number of Scans	5
Comment	survey of the clean Co single crystal



Accession #	00299-01
Host Material	CoO(111) on Co
Technique	XPS
Spectral Region	survey
Instrument	Leybold-Heraeus EA 11
Excitation Source	Al K_α monochromatic
Source Energy	1486.6 eV
Source Strength	450 W
Source Size	not specified
Analyzer Type	spherical sector
Incident Angle	45°
Emission Angle	0°
Analyzer Retard Ratio	4
Analyzer Resolution	0.125 eV
Total Signal Accumulation Time	not specified
Total Elapsed Time	330 s
Number of Scans	5
Comment	survey of the oxidized Co single crystal



- **Accession #:** 00299-02
- **Host Material:** CoO(111) on Co
- **Technique:** XPS
- **Spectral Region:** O 1*s*

Instrument: Leybold-Heraeus EA 11

Excitation Source: Al K_α monochromatic

Source Energy: 1486.6 eV

Source Strength: 450 W

Source Size: not specified

Incident Angle: 45°

Analyzer Type: spherical sector

Analyzer Pass Energy: 25.2 eV

Analyzer Resolution: 0.3 eV

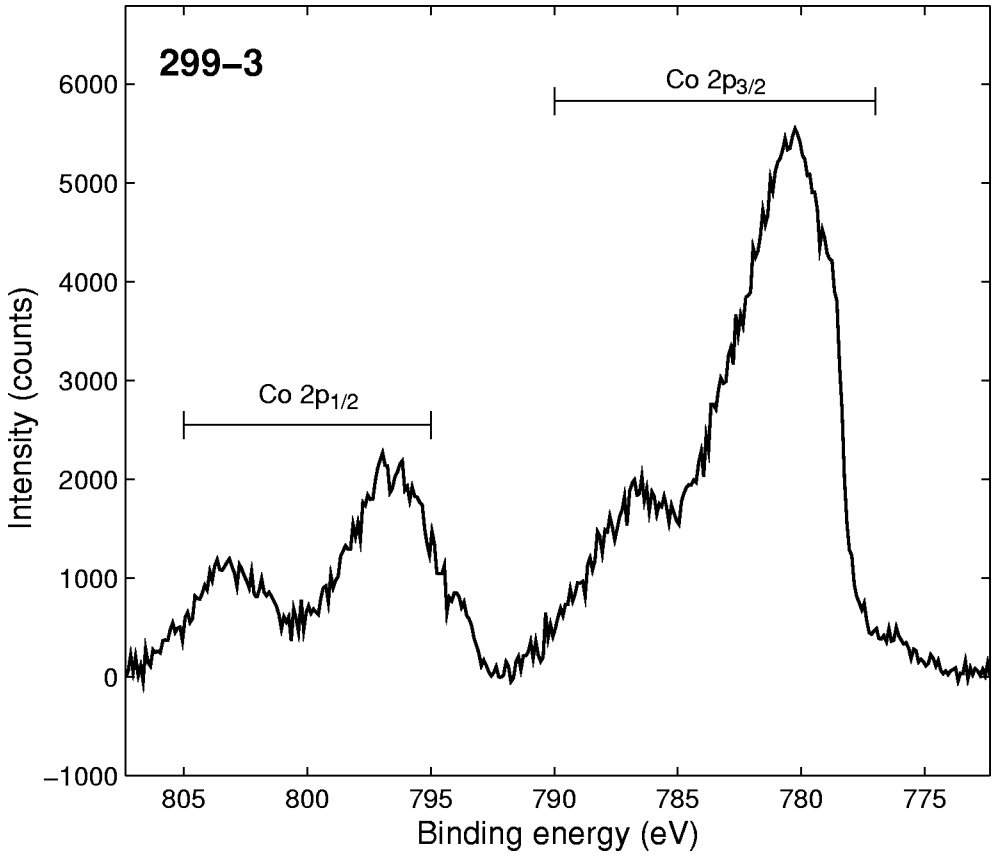
Emission Angle: 0°

Total Signal Accumulation Time: not specified

Total Elapsed Time: 900 s

Number of Scans: 50

Comment: oxidized Co single crystal



- **Accession #:** 00299-03
- **Host Material:** CoO(111) on Co
- **Technique:** XPS
- **Spectral Region:** Co 2$p_{1/2}$; Co 2$p_{3/2}$

Instrument: Leybold-Heraeus EA 11

Excitation Source: Al K_α monochromatic

Source Energy: 1486.6 eV

Source Strength: 450 W

Source Size: not specified

Incident Angle: 45°

Analyzer Type: spherical sector

Analyzer Pass Energy: 25.2 eV

Analyzer Resolution: 0.3 eV

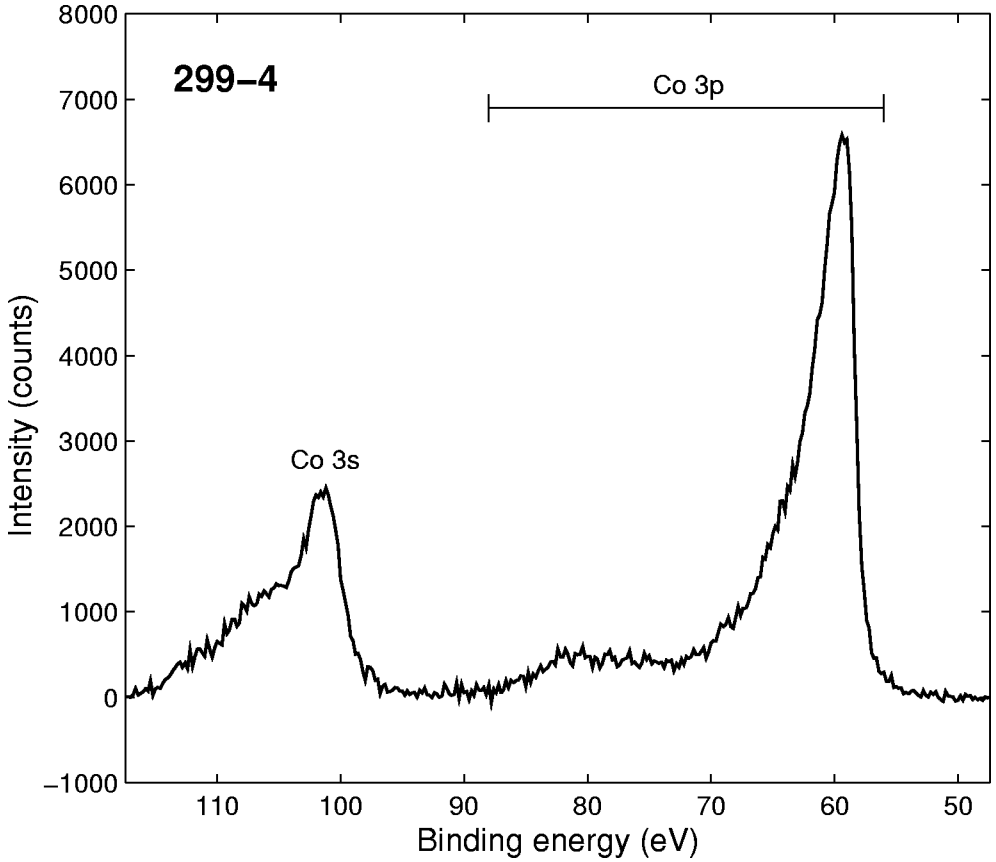
Emission Angle: 0°

Total Signal Accumulation Time: not specified

Total Elapsed Time: 2150 s

Number of Scans: 50

Comment: oxidized Co single crystal



- **Accession #:** 00299-04
- **Host Material:** CoO(111) on Co
- **Technique:** XPS
- **Spectral Region:** survey

Instrument: Leybold-Heraeus EA 11

Excitation Source: Al K_α monochromatic

Source Energy: 1486.6 eV

Source Strength: 450 W

Source Size: not specified

Analyzer type: spherical sector

incident Angle: 45°

Emission Angle: 0°

Analyzer Retard Ratio: 4

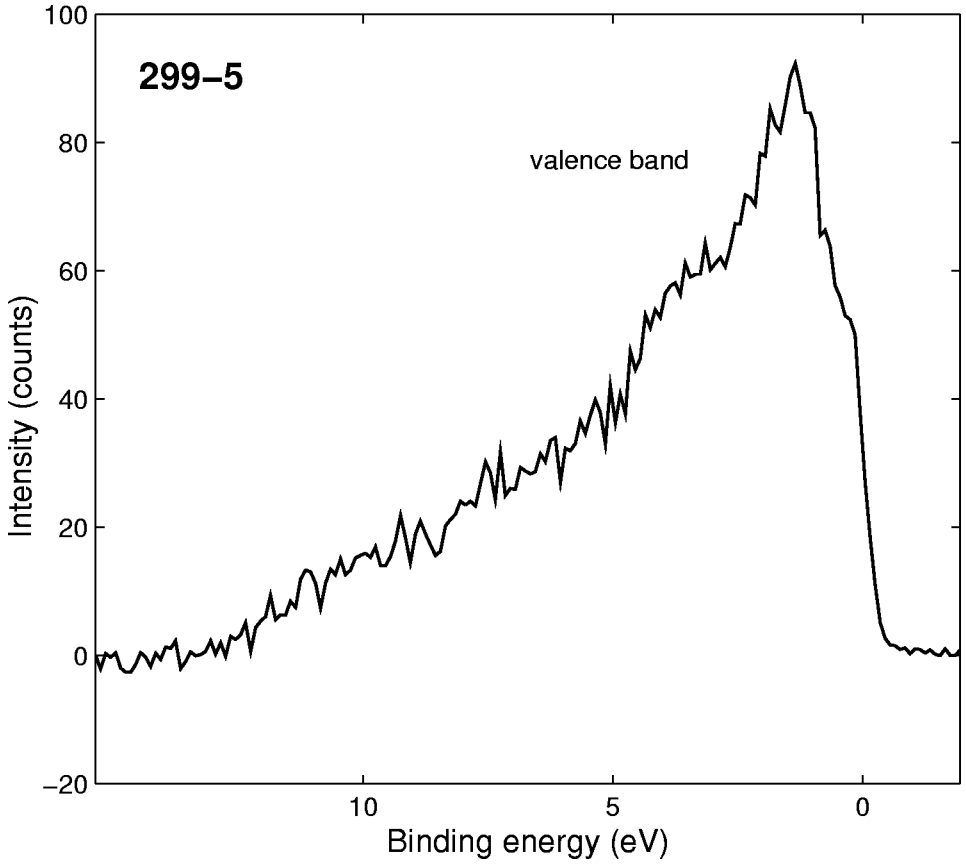
Analyzer Resolution: 0.125 eV

Total Signal Accumulation Time: not specified

Total Elapsed Time: 330 s

Number of Scans: 5

Comment: survey of the oxidized Co single crystal



- **Accession #:** 00299-05
- **Host Material:** CoO(111) on Co
- **Technique:** XPS
- **Spectral Region:** Co valence band; O valence band

Instrument: Leybold-Heraeus EA 11

Excitation Source: Al K_α monochromatic

Source Energy: 1486.6 eV

Source Strength: 450 W

Source Size: not specified

Incident Angle: 45°

Analyzer Type: spherical sector

Analyzer Pass Energy: 25.2 eV

Analyzer Resolution: 0.3 eV

Emission Angle: 0°

Total Signal Accumulation Time: not specified

Total Elapsed Time: 4400 s

Number of Scans: 200

Comment: oxidized Co single crystal