

## Single Crystal Rare-earth Scandate Perovskites Analyzed Using X-ray Photoelectron Spectroscopy: 1. PrScO<sub>3</sub>(110)

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# Single Crystal Rare-earth Scandate Perovskites Analyzed Using X-ray Photoelectron Spectroscopy: 1. PrScO<sub>3</sub>(110)

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X-ray photoelectron spectroscopy (XPS) was used to analyze a commercially available PrScO<sub>3</sub> (110) bulk single crystal. XP spectra were obtained using incident monochromatic Al K<sub>α</sub> radiation at 0.83401 nm. A survey spectrum together with Pr 3d, O 1s, Sc 2p, C 1s, Pr 4p, Pr 4d, Sc 3s, Pr 5s, Sc 3p, O 2s, and Pr 5p core level spectra and the valence band are presented. The spectra indicate the principle core level photoelectron and Auger electron signals and show only minor carbon contamination. Making use of the O 1s, Sc 2p, Pr 4p lines and neglecting the components related to surface contaminants, XPS quantitative analysis reveals an altered stoichiometry of the air-exposed crystal surface of Pr<sub>1.12</sub>ScO<sub>2.29</sub>. © 2014 American Vacuum Society.  
[<http://dx.doi.org/10.1116/11.20140906>]

**Keywords:** praseodymium scandium oxide; rare-earth scandate; perovskite

## INTRODUCTION

Transition metal oxides present an impressive variety of functionality which is not available in more traditional systems such as group IV and III-V semiconductors or elemental metals. Among the many possible functionalities are, for instance, ferroelectricity (Ref. 1) and magnetism (Ref. 2), colossal magnetoresistance (Ref. 3), and high temperature superconductivity (Ref. 4), with transport character ranging from insulating to semiconducting to metallic. Furthermore, these properties are extremely sensitive to perturbations from chemistry, structural defects, strain and many other effects and this, in turn, provides the materials engineer a number of routes by which to engineer new functionalities in this class of materials (Ref. 5). While even simple oxide systems, such as binary oxides, exhibit a broad diversity of properties, it is the ternary systems which have received the most attention in recent years. In particular, materials possessing the perovskite structure (with chemical formula ABO<sub>3</sub>) have been observed to exhibit an incredible variety of functionality and phenomena. Advances in thin film epitaxy, particularly pulsed laser deposition, RF magnetron sputtering, and molecular beam epitaxy, have enabled researchers to carefully tune material properties using epitaxial strain. Such approaches have provided an opportunity to apply large biaxial strains (as much as several percent in some cases) to nanoscale films of various materials which would lead to cracks in bulk materials under similar values of hydrostatic strain (Ref. 6).

<sup>a)</sup>Author to whom correspondence should be addressed.

## SPECIMEN DESCRIPTION (ACCESSION #01315)

**Host Material:** Single crystal PrScO<sub>3</sub>

**CAS Registry #:** unknown

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

**Chemical Name:** praseodymium scandium oxide

**Source:** Crystec, GmbH. Grown by the Czochralski method.

**Host Composition:** PrScO<sub>3</sub>

**Form:** single crystal

**Structure:** orthorhombic distorted perovskite-like structure Pnma  
Z = 4, a = 0.5780(1) nm, b = 0.8025(2) nm, c = 0.5608(1) nm,  
V = 0.2601 nm<sup>3</sup>, Rgt(F) = 0.025, wRref(F<sub>2</sub>) = 0.060, T = 298 K (Ref. 7)

**History & Significance:** Various perovskite-based compounds have been widely used as substrates for a number of important applications such as epitaxial substrates for high TC oxide superconductors (Ref. 8), ferroelectric materials (Ref. 9), high-quality optoelectronic semiconductors (Ref. 10), and colossal magnetoresistive materials (Ref. 11). One particular group of perovskite-based materials, rare-earth scandates, is gaining attention as a candidate for high-k dielectrics (Refs. 12 and 13). In order to gain an increased understanding of the surfaces and hetero-interfaces of perovskite-based materials, a PrScO<sub>3</sub> (110) bulk single crystal was analyzed using X-ray photoelectron spectroscopy.

**As Received Condition:** as grown

**Analyzed Region:** same as host material

**Ex Situ Preparation/Mounting:** Samples were cleaned ultrasonically for 5 min each in Formula 409<sup>®</sup>, methyl alcohol, and deionized water. Samples were mounted onto the sample holder using double-sided carbon tape (Pella product number 16074).

**In Situ Preparation:** none

**Pre-Analysis Beam Exposure:** less than 2 min; no x-ray degradation effects observed

**Charge Control:** low energy flood gun/magnetic immersion lens combination, filament current = 1.8 A, charge balance = 3 V, filament bias = 1 V

**Temp. During Analysis:** 300 K

**Pressure During Analysis:**  $<3 \times 10^{-7}$  Pa

## INSTRUMENT DESCRIPTION

**Manufacturer and Model:** Kratos Axis Ultra

**Analyzer Type:** spherical sector

**Detector:** channeltron electron multiplier

**Number of Detector Elements:** 8

## INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

### ■ Spectrometer

**Analyzer Mode:** constant pass energy

**Throughput ( $T = E^M$ ):**  $N = 0$

**Excitation Source Window:** not specified

**Excitation Source:** Al  $K_{\alpha}$ , monochromatic

**Source Energy:** 1486.6 eV

**Source Strength:** 180 W

**Source Beam Size:** 2000  $\mu\text{m} \times 2000 \mu\text{m}$

**Signal Mode:** multichannel direct

### ■ Geometry

**Incident Angle:** 54°

**Source to Analyzer Angle:** 54°

**Emission Angle:** 0°

**Specimen Azimuthal Angle:** 45°

**Acceptance Angle from Analyzer Axis:** 0°

**Analyzer Angular Acceptance Width:** 40°  $\times$  40°

## DATA ANALYSIS METHOD

**Energy Scale Correction:** The binding energy scale was referenced to C 1s = 285.0 eV.

**Recommended Energy Scale Shift:** 2.039

**Peak Shape and Background Method:** Background: Custom three parameter Tougaard background (Ref. 14), U 4 Tougaard

(B, C, D, T0=0) (Ref. 15), was used. O 1s, C 1s, Pr 4p: B = 299 eV<sup>2</sup>, C = 550 eV<sup>2</sup>, D = 275 eV<sup>2</sup>, Sc 2p: B = 299 eV<sup>2</sup>, C = 350 eV<sup>2</sup>, D = 275 eV<sup>2</sup>

**Quantitation Method:** Quantification was done using region and component definitions with CasaXPS version 2.3.15. Sensitivity factors supplied by Kratos Analytical. Errors are given as  $\pm 1$  standard deviation. Standard deviations are calculated by CasaXPS using a Monte Carlo method for determining the error distribution for the computed areas.

## ACKNOWLEDGMENTS

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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
01315-02	Pr 3d <sub>5/2</sub>	928.0	2.37	...	...	...	PrScO <sub>3</sub>
01315-02	Pr 3d <sub>5/2</sub>	932.8	4.70	...	...	...	PrScO <sub>3</sub>
01315-02	Pr 3d <sub>3/2</sub>	949.2	3.24	...	...	...	PrScO <sub>3</sub>
01315-02	Pr 3d <sub>3/2</sub>	953.5	3.56	...	...	...	PrScO <sub>3</sub>
01315-02	Pr 3d <sub>3/2</sub>	957.4	3.57	...	...	...	PrScO <sub>3</sub>
01315-03	O 1s	529.2	0.96	23632.4	0.780	32.01	PrScO <sub>3</sub>
01315-03 <sup>a</sup>	O 1s	531.4	1.91	7295.7	0.780	9.88	hydroxide
01315-03 <sup>a</sup>	O 1s	533.0	1.48	3227.7	0.780	4.37	carbonate
01315-04	Sc 2p	...	...	24593	1.875	13.29	...
01315-04	Sc 2p <sub>3/2</sub>	401.0	1.01	...	...	...	PrScO <sub>3</sub>
01315-04 <sup>b</sup>	Sc 2p <sub>3/2</sub>	412.7	2.44	...	...	...	PrScO <sub>3</sub>
01315-04	Sc 2p <sub>1/2</sub>	405.4	1.40	...	...	...	PrScO <sub>3</sub>
01315-04 <sup>b</sup>	Sc 2p <sub>1/2</sub>	417.0	3.91	...	...	...	PrScO <sub>3</sub>
01315-05 <sup>a</sup>	C 1s	285.0	1.22	3781.4	0.278	14.48	hydrocarbon
01315-05 <sup>a</sup>	C 1s	286.6	1.18	1403.0	0.278	5.37	C-hydroxide
01315-05 <sup>a</sup>	C 1s	288.4	2.20	1121.1	0.278	4.29	carbonate
01315-06	Pr 4p	...	...	15571.8	1.061	15.63	...
01315-06	Pr 4p <sub>3/2</sub>	215.3	3.07	...	...	...	PrScO <sub>3</sub>
01315-06	Pr 4p <sub>3/2</sub>	218.8	4.11	...	...	...	PrScO <sub>3</sub>
01315-07	Pr 4d <sub>5/2</sub>	109.6	1.00	...	...	...	PrScO <sub>3</sub>
01315-07	Pr 4d <sub>5/2</sub>	112.8	3.25	...	...	...	PrScO <sub>3</sub>
01315-07	Pr 4d <sub>5/2</sub>	116.4	3.66	...	...	...	PrScO <sub>3</sub>
01315-07	Pr 4d <sub>3/2</sub>	119.9	3.00	...	...	...	PrScO <sub>3</sub>
01315-07	Pr 4d <sub>3/2</sub>	122.6	2.63	...	...	...	PrScO <sub>3</sub>
01315-08	Sc 3s	52.6	2.72	...	...	...	PrScO <sub>3</sub>
01315-09	Pr 5s	36.9	1.53	...	...	...	PrScO <sub>3</sub>
01315-09	Pr 5s	38.4	1.32	...	...	...	PrScO <sub>3</sub>
01315-09	Sc 3p	30.8	1.66	...	...	...	PrScO <sub>3</sub>
01315-09 <sup>c</sup>	Sc 3p	42.5	1.48	...	...	...	PrScO <sub>3</sub>
01315-09 <sup>c</sup>	Sc 3p	44.4	2.71	...	...	...	PrScO <sub>3</sub>
01315-09 <sup>d</sup>	O 2s	22.6	1.90	...	...	...	PrScO <sub>3</sub>
01315-09 <sup>d</sup>	Pr 5p	17.8	2.36	...	...	...	PrScO <sub>3</sub>
01315-09 <sup>d</sup>	Pr 5p	20.1	2.04	...	...	...	PrScO <sub>3</sub>
01315-09 <sup>d</sup>	Pr 5p	22.3	2.88	...	...	...	PrScO <sub>3</sub>
01315-09 <sup>e</sup>	valence band	6.5	1.43	...	...	...	PrScO <sub>3</sub>
01315-09 <sup>e</sup>	valence band	5.3	1.73	...	...	...	PrScO <sub>3</sub>
01315-09 <sup>e</sup>	valence band	3.6	1.69	...	...	...	PrScO <sub>3</sub>
01315-09 <sup>f</sup>	valence band maximum (VBM)	2.0	0.78	...	...	...	PrScO <sub>3</sub>

<sup>a</sup> Result of exposure to air<sup>b</sup> Bulk plasmon<sup>c</sup> Satellite<sup>d</sup> Estimated based on peak fitting<sup>e</sup> O 2p and Sc 3d (Ref. 16)<sup>f</sup> The position of VBM was estimated by subtracting 1/2 of the full width at half maximum (FWHM) from the position of the maximum intensity at the VBM.

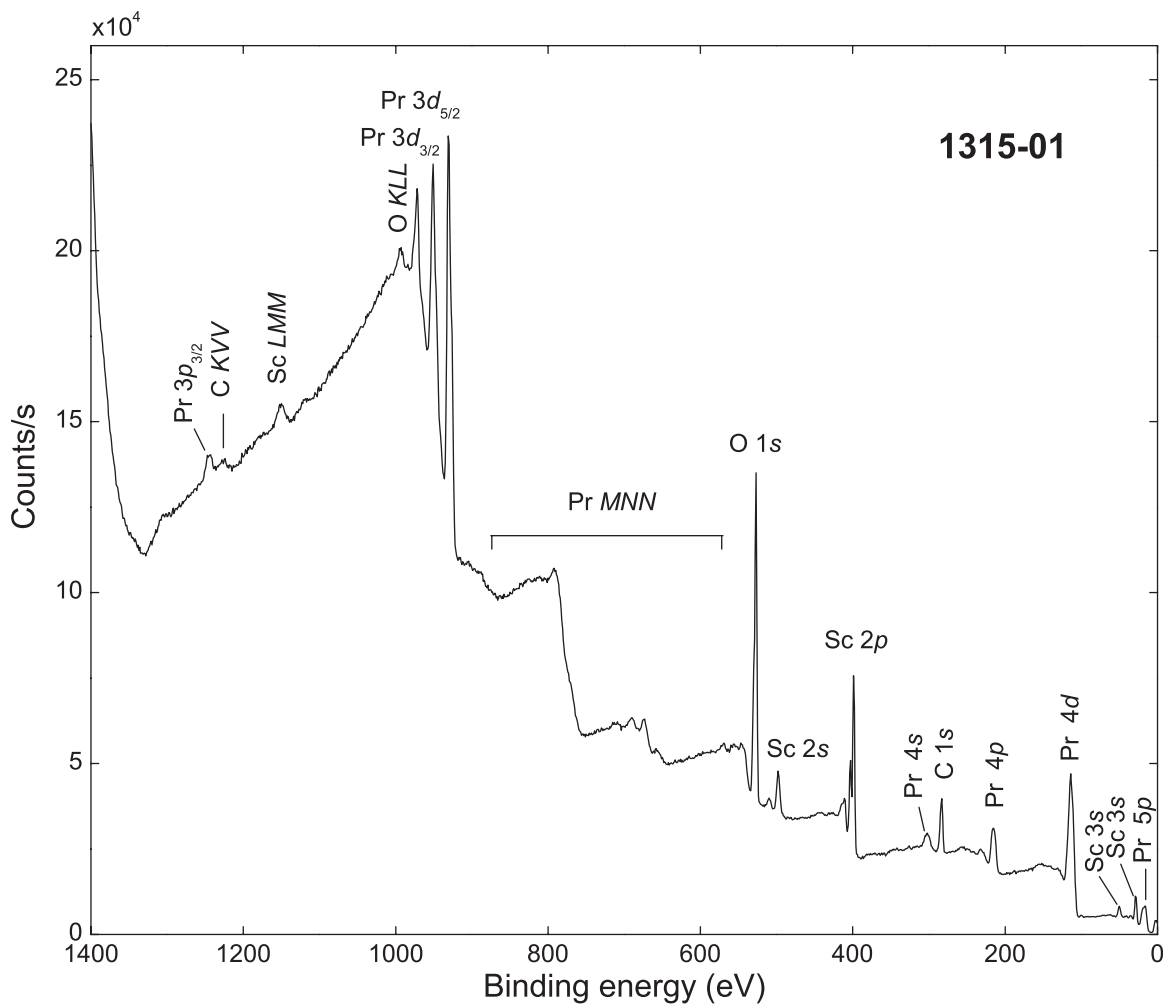
**ANALYZER CALIBRATION TABLE**

<b>Spectrum ID #</b>	<b>Element/Transition</b>	<b>Peak Energy (eV)</b>	<b>Peak Width FWHM (eV)</b>	<b>Peak Area (eV × cts/s)</b>	<b>Sensitivity Factor</b>	<b>Concentration (at. %)</b>	<b>Peak Assignment</b>
	Au 4f <sub>7/2</sub>	84.0	0.72	151917.9	...	...	...
	Ag 3d <sub>5/2</sub>	368.2	0.58	230506.2	...	...	...
	Cu 2p <sub>3/2</sub>	932.6	0.88	410979.8	...	...	...

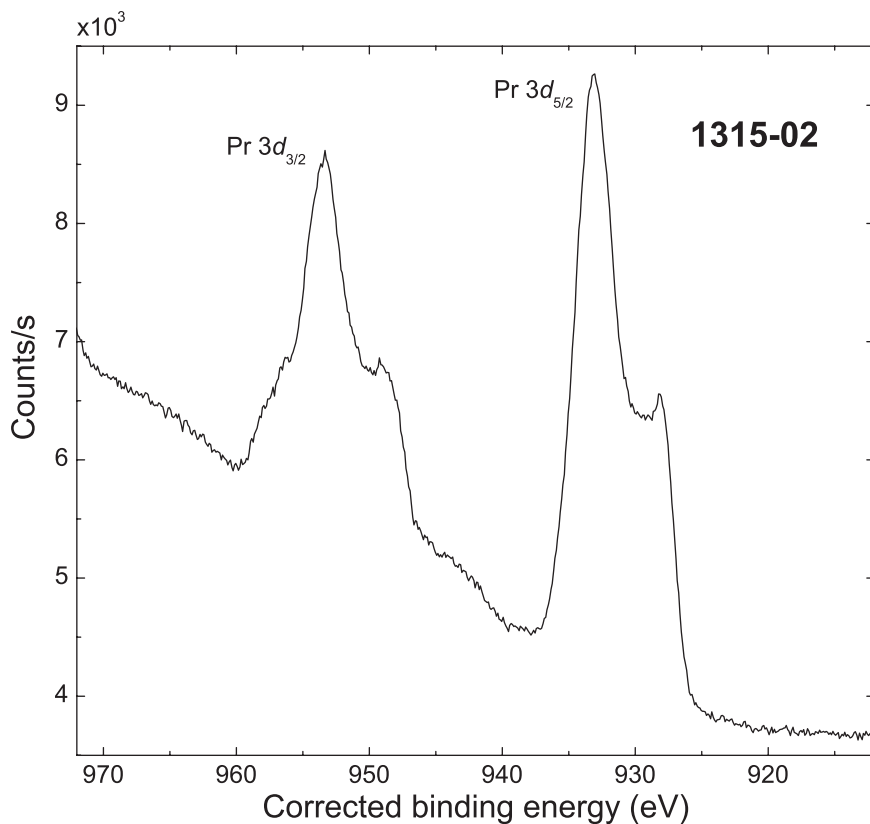
**GUIDE TO FIGURES**

<b>Spectrum (Accession) #</b>	<b>Spectral Region</b>	<b>Voltage Shift*</b>	<b>Multiplier</b>	<b>Baseline</b>	<b>Comment #</b>
1315-01	survey	0	1	0	
1315-02	Pr 3d	-2.039	1	0	
1315-03	O 1s	-2.039	1	0	
1315-04	Sc 2p	-2.039	1	0	
1315-05	C 1s	-2.039	1	0	
1315-06	Pr 4p	-2.039	1	0	
1315-07	Pr 4d	-2.039	1	0	
1315-08	Sc 3s	-2.039	1	0	
1315-09	Pr 5s, Sc 3p, O 2s, Pr 5p, valence band	-2.039	1	0	

\* 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.

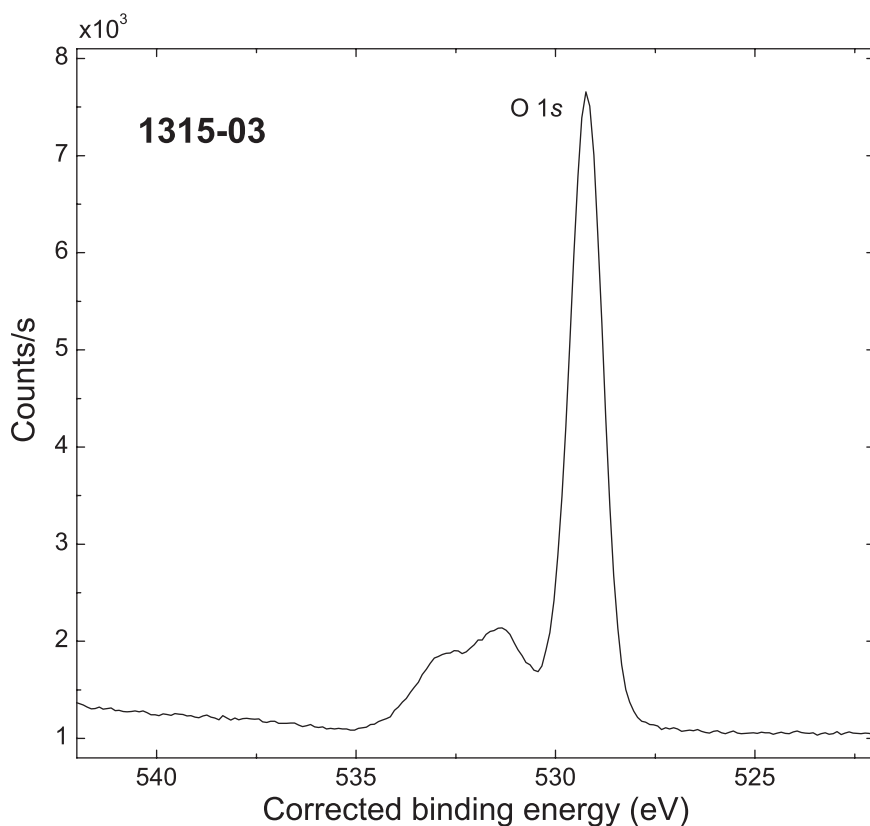


Accession #	01315-01
Host Material	Single crystal PrScO <sub>3</sub>
Technique	XPS
Spectral Region	survey
Instrument	Kratos Axis Ultra
Excitation Source	Al K <sub>α</sub> monochromatic
Source Energy	1486.6 eV
Source Strength	180 W
Source Size	2 mm × 2 mm
Analyzer Type	spherical sector
Incident Angle	54°
Emission Angle	0°
Analyzer Pass Energy:	160 eV
Analyzer Resolution	2.4 eV
Total Signal Accumulation Time	560 s
Total Elapsed Time	1120 s
Number of Scans	4
Effective Detector Width	33.6 eV



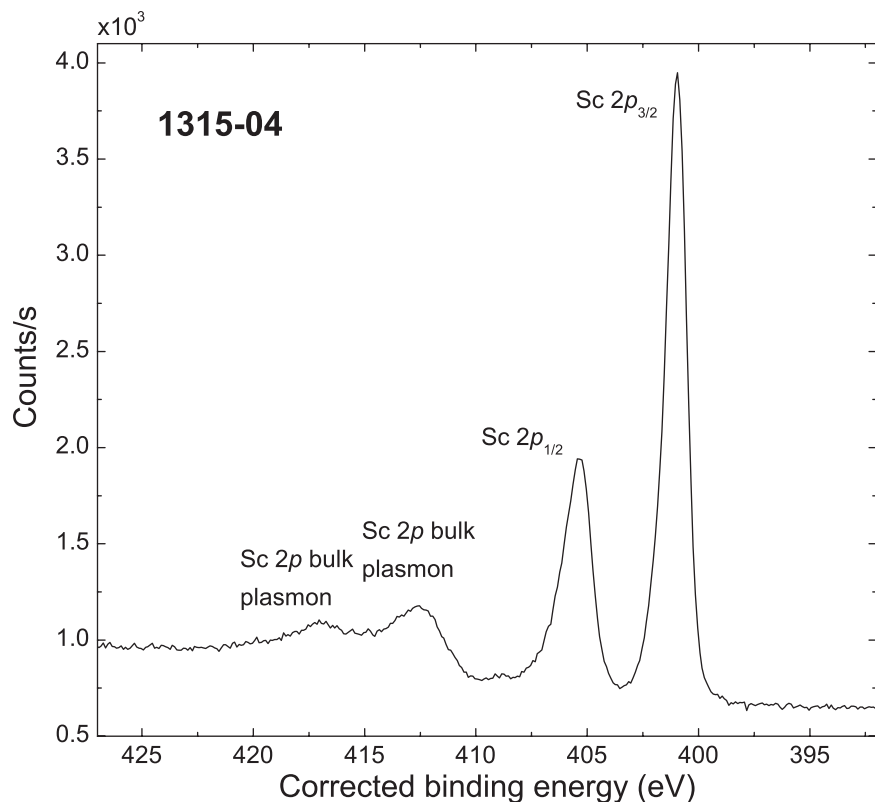
- Accession #: 01315-02
- Host Material: Single crystal PrScO<sub>3</sub>
- Technique: XPS
- Spectral Region: Pr 3d

Instrument: Kratos Axis Ultra  
 Excitation Source: Al K<sub>α</sub> monochromatic  
 Source Energy: 1486.6 eV  
 Source Strength: 180 W  
 Source Size: 2 mm × 2 mm  
 Analyzer Type: spherical sector  
 Incident Angle: 54°  
 Emission Angle: 0°  
 Analyzer Pass Energy: 20 eV  
 Analyzer Resolution: 0.3 eV  
 Total Signal Accumulation Time: 3606 s  
 Total Elapsed Time: 9916.5 s  
 Number of Scans: 20  
 Effective Detector Width: 4.2 eV



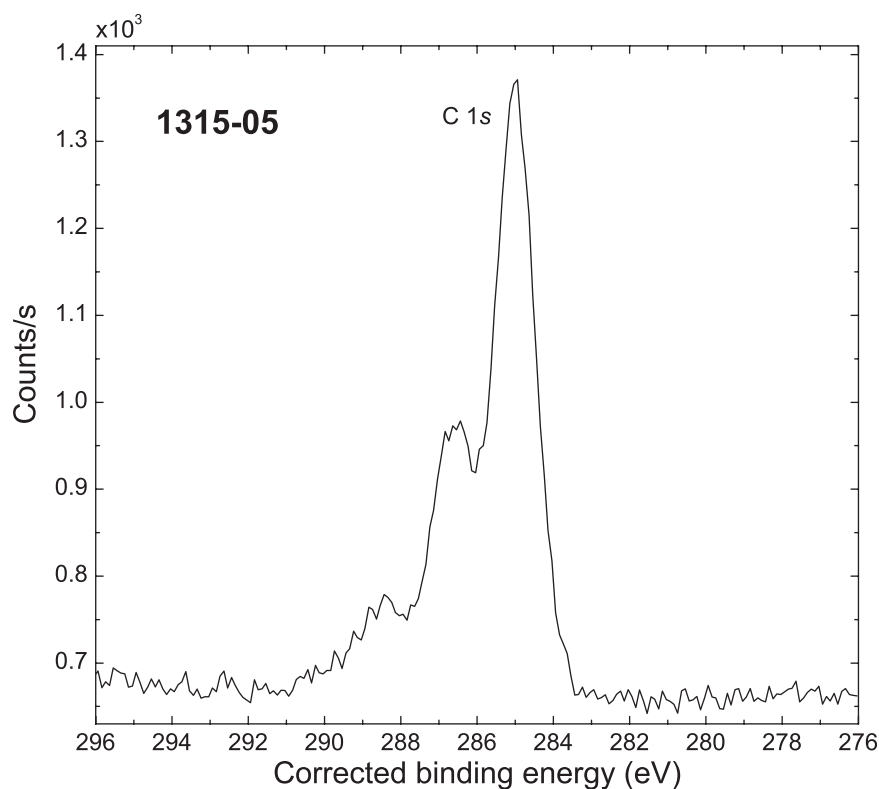
- Accession #: 01315-03
- Host Material: Single crystal PrScO<sub>3</sub>
- Technique: XPS
- Spectral Region: O 1s

Instrument: Kratos Axis Ultra  
 Excitation Source: Al K<sub>α</sub> monochromatic  
 Source Energy: 1486.6 eV  
 Source Strength: 180 W  
 Source Size: 2 mm × 2 mm  
 Analyzer Type: spherical sector  
 Incident Angle: 54°  
 Emission Angle: 0°  
 Analyzer Pass Energy: 20 eV  
 Analyzer Resolution: 0.3 eV  
 Total Signal Accumulation Time: 1206 s  
 Total Elapsed Time: 3316.5 s  
 Number of Scans: 20  
 Effective Detector Width: 4.2 eV



- Accession #: 01315-04
- Host Material: Single crystal PrScO<sub>3</sub>
- Technique: XPS
- Spectral Region: Sc 2p

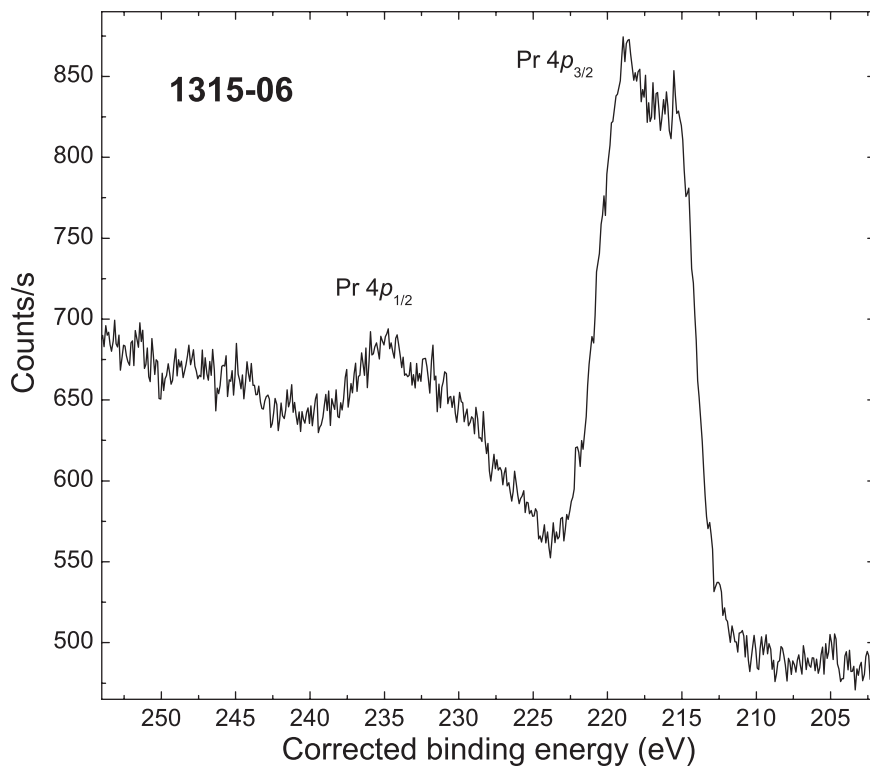
Instrument: Kratos Axis Ultra  
 Excitation Source: Al K<sub>α</sub> monochromatic  
 Source Energy: 1486.6 eV  
 Source Strength: 180 W  
 Source Size: 2 mm × 2 mm  
 Analyzer Type: spherical sector  
 Incident Angle: 54°  
 Emission Angle: 0°  
 Analyzer Pass Energy: 20 eV  
 Analyzer Resolution: 0.3 eV  
 Total Signal Accumulation Time: 2106 s  
 Total Elapsed Time: 5791.5 s  
 Number of Scans: 20  
 Effective Detector Width: 4.2 eV



- Accession #: 01315-05
- Host Material: Single crystal PrScO<sub>3</sub>
- Technique: XPS
- Spectral Region: C 1s

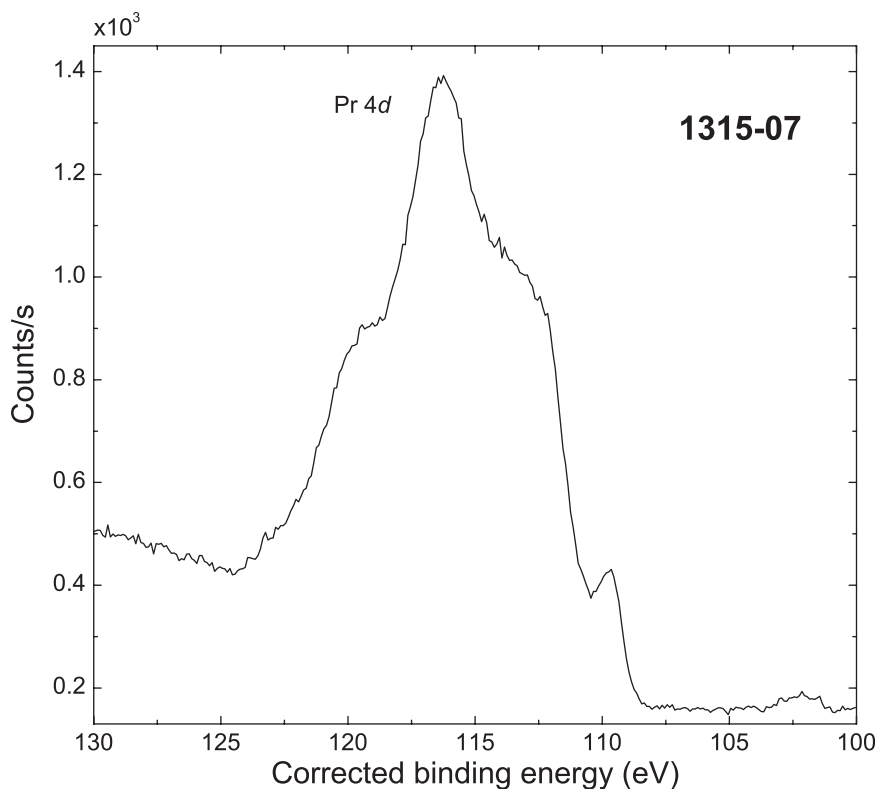
Instrument: Kratos Axis Ultra  
 Excitation Source: Al K<sub>α</sub> monochromatic  
 Source Energy: 1486.6 eV  
 Source Strength: 180 W  
 Source Size: 2 mm × 2 mm  
 Analyzer Type: spherical sector  
 Incident Angle: 54°  
 Emission Angle: 0°  
 Analyzer Pass Energy: 20 eV  
 Analyzer Resolution: 0.3 eV  
 Total Signal Accumulation Time: 1206 s  
 Total Elapsed Time: 3316.5 s  
 Number of Scans: 20  
 Effective Detector Width: 4.2 eV





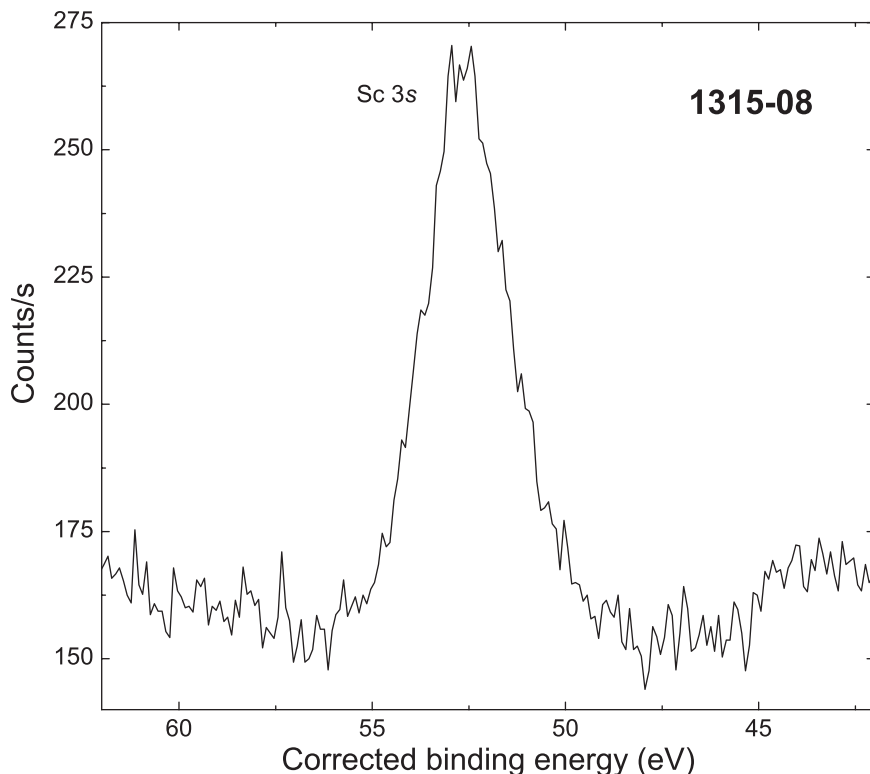
- Accession #: 01315-06
- Host Material: Single crystal PrScO<sub>3</sub>
- Technique: XPS
- Spectral Region: Pr 4p

Instrument: Kratos Axis Ultra  
 Excitation Source: Al K<sub>α</sub> monochromatic  
 Source Energy: 1486.6 eV  
 Source Strength: 180 W  
 Source Size: 2 mm × 2 mm  
 Analyzer Type: spherical sector  
 Incident Angle: 54°  
 Emission Angle: 0°  
 Analyzer Pass Energy: 20 eV  
 Analyzer Resolution: 0.3 eV  
 Total Signal Accumulation Time: 3126 s  
 Total Elapsed Time: 8596.5 s  
 Number of Scans: 20  
 Effective Detector Width: eV



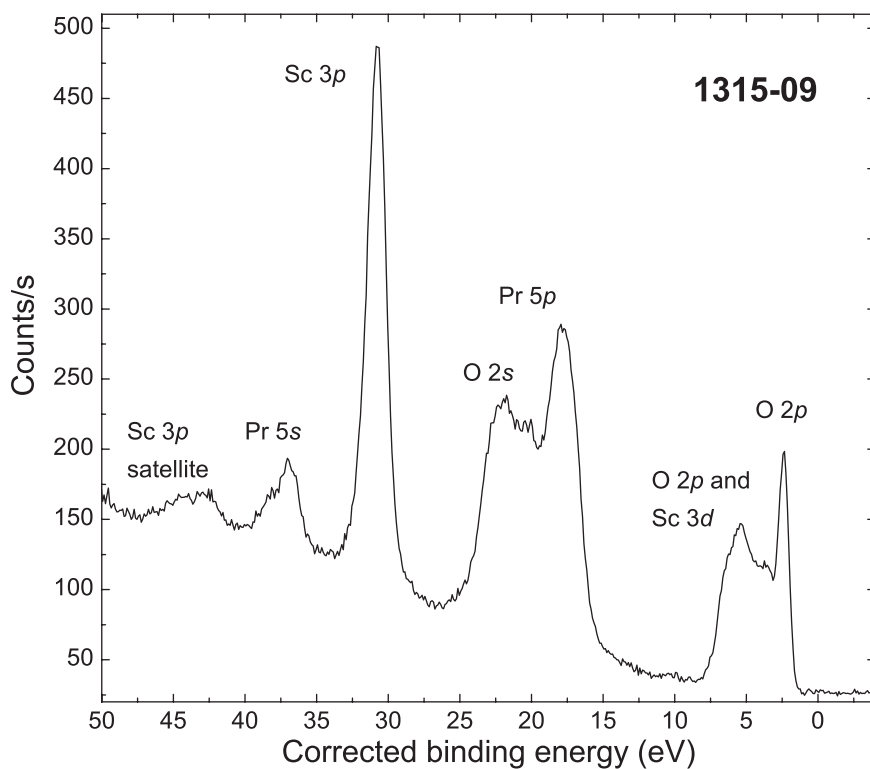
- Accession #: 01315-07
- Host Material: Single crystal PrScO<sub>3</sub>
- Technique: XPS
- Spectral Region: Pr 4d

Instrument: Kratos Axis Ultra  
 Excitation Source: Al K<sub>α</sub> monochromatic  
 Source Energy: 1486.6 eV  
 Source Strength: 180 W  
 Source Size: 2 mm × 2 mm  
 Analyzer Type: spherical sector  
 Incident Angle: 54°  
 Emission Angle: 0°  
 Analyzer Pass Energy: 20 eV  
 Analyzer Resolution: 0.3 eV  
 Total Signal Accumulation Time: 1806 s  
 Total Elapsed Time: 4966.5 s  
 Number of Scans: 20  
 Effective Detector Width: 4.2 eV



- Accession #: 01315-08
- Host Material: Single crystal PrScO<sub>3</sub>
- Technique: XPS
- Spectral Region: Sc 3s

Instrument: Kratos Axis Ultra  
 Excitation Source: Al K<sub>α</sub> monochromatic  
 Source Energy: 1486.6 eV  
 Source Strength: 180 W  
 Source Size: 2 mm × 2 mm  
 Analyzer Type: spherical sector  
 Incident Angle: 54°  
 Emission Angle: 0°  
 Analyzer Pass Energy: 20 eV  
 Analyzer Resolution: 0.3 eV  
 Total Signal Accumulation Time: 1206 s  
 Total Elapsed Time: 3316.5 s  
 Number of Scans: 20  
 Effective Detector Width: 4.2 eV



- Accession #: 01315-09
- Host Material: Single crystal PrScO<sub>3</sub>
- Technique: XPS
- Spectral Region: Pr 5s; Sc 3p; O 2s; Pr 5p; valence band

Instrument: Kratos Axis Ultra  
 Excitation Source: Al K<sub>α</sub> monochromatic  
 Source Energy: 1486.6 eV  
 Source Strength: 180 W  
 Source Size: 2 mm × 2 mm  
 Analyzer Type: spherical sector  
 Incident Angle: 54°  
 Emission Angle: 0°  
 Analyzer Pass Energy: 20 eV  
 Analyzer Resolution: 0.3 eV  
 Total Signal Accumulation Time: 7574 s  
 Total Elapsed Time: 20828.5 s  
 Number of Scans: 20  
 Effective Detector Width: 4.2 eV