

## Single Crystal Rare-earth Scandate Perovskites Analyzed Using X-ray Photoelectron Spectroscopy: 5. DyScO<sub>3</sub>(110)

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# Single Crystal Rare-earth Scandate Perovskites Analyzed Using X-ray Photoelectron Spectroscopy: 5. DyScO<sub>3</sub>(110)

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X-ray photoelectron spectroscopy (XPS) was used to analyze a commercially available DyScO<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 Dy 3d, O 1s, Sc 2p, Dy 4p, C 1s, Dy 4d, Sc 3s, Dy 5s, Sc 3p, Tb 5p, and O 2s 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, Dy 4d lines and neglecting the components related to surface contaminants, XPS quantitative analysis reveals an altered stoichiometry of the air-exposed crystal surface of DySc<sub>1.03</sub>O<sub>2.29</sub>. © 2014 American Vacuum Society.  
[<http://dx.doi.org/10.1116/11.20140910>]

**Keywords:** dysprosium 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).

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## SPECIMEN DESCRIPTION (ACCESSION #01319)

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

**CAS Registry #:** unknown

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

**Chemical Name:** dysprosium scandium oxide.

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

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

**Form:** single crystal

**Structure:** orthorhombic distorted perovskite-like structure Pnma Z = 4, a = 0.57175(2) nm, b = 0.7901(2) nm, c = 0.5443(2) nm, V = 0.2459(1) nm<sup>3</sup> (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 DyScO<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

Accession #: 01319

Technique: XPS

Host Material: Single crystal DyScO<sub>3</sub>

Instrument: Kratos Axis Ultra

Major Elements in Spectra: Dy, Sc, O

Minor Elements in Spectra: C

Published Spectra: 7

Spectra in Electronic Record: 7

Spectral Category: comparison

**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 = 3.25 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:** +2.103 eV for high-resolution spectra

**Peak Shape and Background Method:** Background: Custom three parameter Tougaard background (Ref. 14), U 4 Tougaard (B, C, D,  $T_0=0$ ) (Ref. 15), was used. O 1s, Sc 2p:

B = 299 eV<sup>2</sup>, C = 300 eV<sup>2</sup>, D = 275 eV<sup>2</sup>, C 1s: B = 299 eV<sup>2</sup>, C = 150 eV<sup>2</sup>, D = 275 eV<sup>2</sup>, Tb 4d: B = 299 eV<sup>2</sup>, C = 210 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
01319-02	Dy 3d <sub>5/2</sub>	1298.2	7.66	...	...	...	DyScO <sub>3</sub>
01319-02	Dy 3d <sub>3/2</sub>	1333.4	4.04	...	...	...	DyScO <sub>3</sub>
01319-02	Dy 3d <sub>3/2</sub>	1338.3	6.96	...	...	...	DyScO <sub>3</sub>
01319-03	O 1s	529.3	1.00	15511.2	0.780	27.12	DyScO <sub>3</sub>
01319-03 <sup>a</sup>	O 1s	531.4	1.90	6243.7	0.780	10.92	hydroxide
01319-03 <sup>a</sup>	O 1s	532.9	1.70	3362.9	0.780	5.88	carbonate
01319-04	Sc 2p	...	...	16715.6	1.875	12.53	...
01319-04	Sc 2p <sub>3/2</sub>	401.1	1.11	...	...	...	DyScO <sub>3</sub>
01319-04	Sc 2p <sub>3/2</sub>	413.2	2.91	...	...	...	DyScO <sub>3</sub>
01319-04	Sc 2p <sub>1/2</sub>	405.5	1.39	...	...	...	DyScO <sub>3</sub>
01319-04	Sc 2p <sub>1/2</sub>	417.6	2.91	...	...	...	DyScO <sub>3</sub>
01319-05	Dy 4p <sub>3/2</sub>	296.4	7.34	...	...	...	DyScO <sub>3</sub>
01319-05 <sup>a</sup>	C 1s	285.0	1.25	4263.9	0.278	20.92	hydrocarbon
01319-05 <sup>a</sup>	C 1s	286.5	1.48	1306.3	0.278	6.41	C-hydroxide
01319-05 <sup>a</sup>	C 1s	288.2	1.53	970.4	0.278	4.76	carbonate
01319-06 <sup>b</sup>	Dy 4d	...	...	21547.6	2.484	11.83	...
01319-06	Dy 4d	153.5	3.33	...	...	...	DyScO <sub>3</sub>
01319-06	Dy 4d	156.1	1.65	...	...	...	DyScO <sub>3</sub>
01319-06	Dy 4d	157.8	2.84	...	...	...	DyScO <sub>3</sub>
01319-06	Dy 4d	161.2	3.52	...	...	...	DyScO <sub>3</sub>
01319-06	Dy 4d	164.4	1.85	...	...	...	DyScO <sub>3</sub>
01319-06	Dy 4d	170.0	3.93	...	...	...	DyScO <sub>3</sub>
01319-06	Dy 4d	178.1	5.80	...	...	...	DyScO <sub>3</sub>
01319-06	Dy 4d	183.2	6.12	...	...	...	DyScO <sub>3</sub>
01319-07	Sc 3s	52.7	2.93	...	...	...	DyScO <sub>3</sub>
01319-07	Dy 5s	43.8	3.25	...	...	...	DyScO <sub>3</sub>
01319-07	Dy 5s	47.6	3.00	...	...	...	DyScO <sub>3</sub>
01319-07	Dy 5s	50.0	1.75	...	...	...	DyScO <sub>3</sub>
01319-07	Sc 3p	31.0	1.34	...	...	...	DyScO <sub>3</sub>
01319-07	Dy 5p <sub>3/2</sub>	20.4	3.89	...	...	...	DyScO <sub>3</sub>
01319-07	Dy 5p <sub>1/2</sub>	29.1	2.55	...	...	...	DyScO <sub>3</sub>
01319-07	Dy 5p <sub>1/2</sub>	32.5	3.55	...	...	...	DyScO <sub>3</sub>
01319-07	O 2s	24.0	3.27	...	...	...	DyScO <sub>3</sub>
01319-07 <sup>c</sup>	valence band	11.5	5.39	...	...	...	DyScO <sub>3</sub>
01319-07 <sup>c</sup>	valence band	9.5	2.31	...	...	...	DyScO <sub>3</sub>
01319-07 <sup>d</sup>	valence band	6.3	3.03	...	...	...	DyScO <sub>3</sub>
01319-07 <sup>e</sup>	valence band maximum (VBM)	3.1	1.33	...	...	...	DyScO <sub>3</sub>

<sup>a</sup> Result of exposure to air

<sup>b</sup> Energy range for quantification 166.4–149.1 eV

<sup>c</sup> O 2p, Sc 3d and Dy 4f (Ref. 16)

<sup>d</sup> O 2p and Sc 3d (Ref. 16)

<sup>e</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.

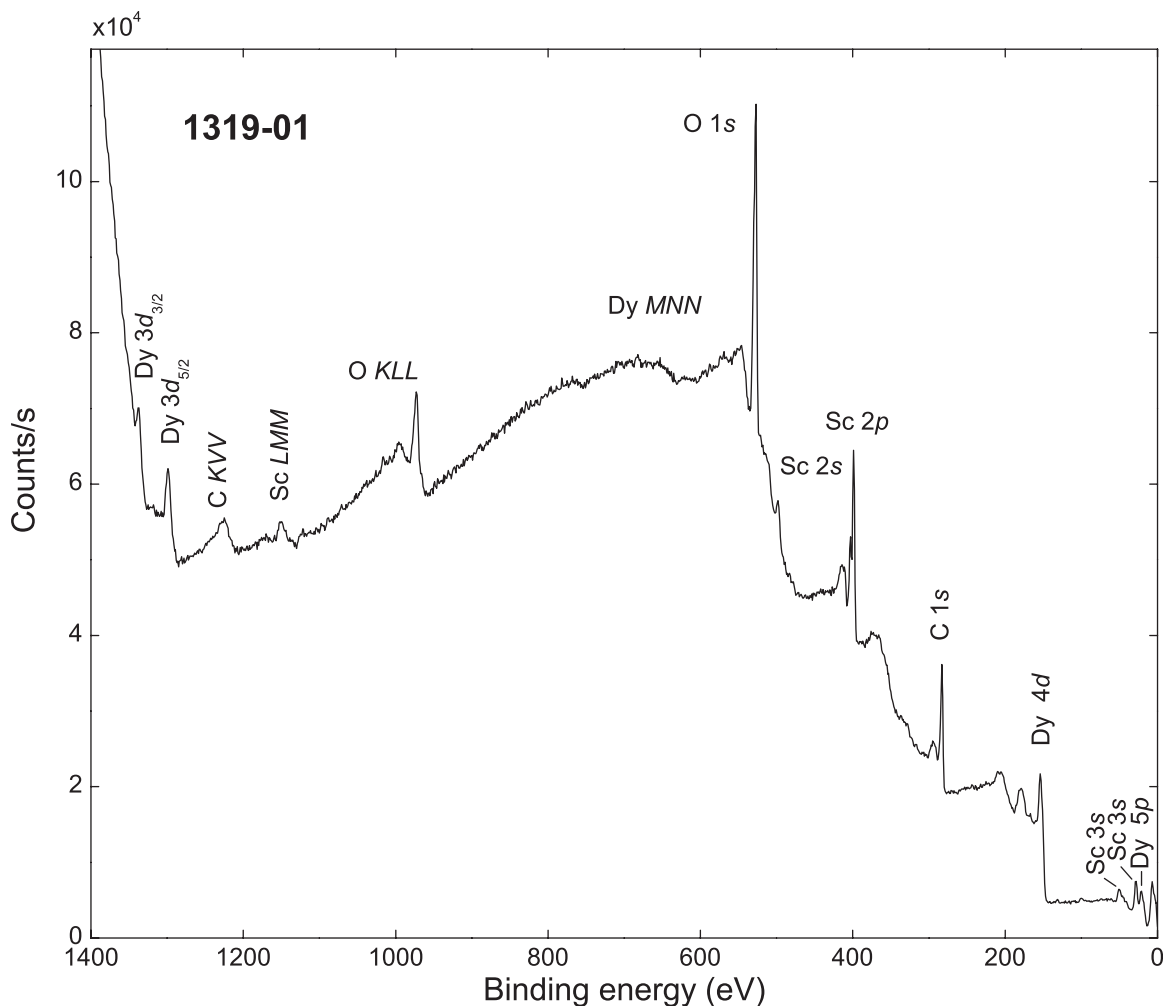
**ANALYZER CALIBRATION TABLE**

<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	...	...	...

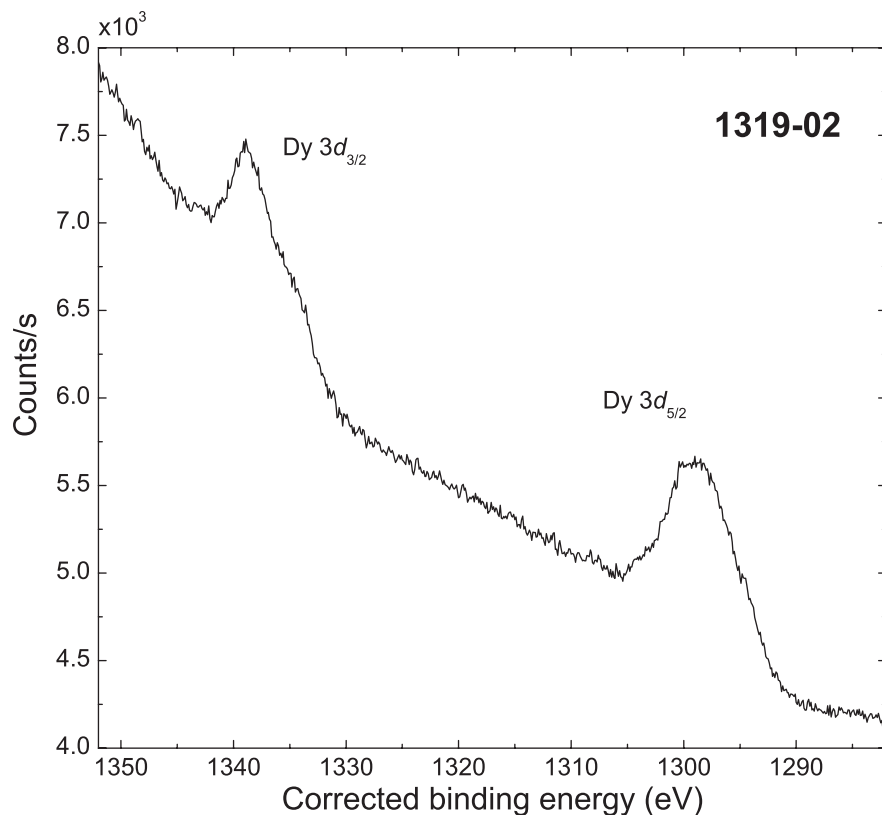
**GUIDE TO FIGURES**

<b>Spectrum (Accession) #</b>	<b>Spectral Region</b>	<b>Voltage Shift*</b>	<b>Multiplier</b>	<b>Baseline</b>	<b>Comment #</b>
1319-01	survey	0	1	0	
1319-02	Dy 3d	-2.103	1	0	
1319-03	O 1s	-2.103	1	0	
1319-04	Sc 2p	-2.103	1	0	
1319-05	Dy 4p, C 1s	-2.103	1	0	
1319-06	Dy 4d	-2.103	1	0	
1319-07	Sc 3s, Dy 5s, Sc 3p, Dy 5p, O 2s, valence band	-2.103	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.

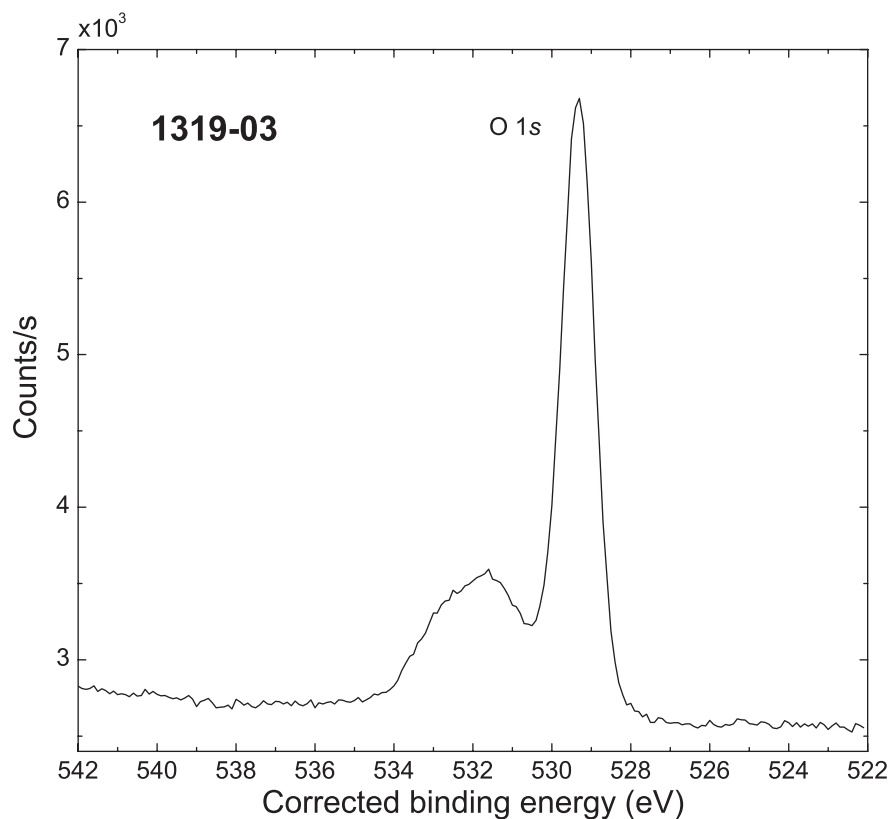


<b>Accession #</b>	<b>01319-01</b>
<b>Host Material</b>	Single crystal DyScO <sub>3</sub>
<b>Technique</b>	XPS
<b>Spectral Region</b>	survey
<b>Instrument</b>	Kratos Axis Ultra
<b>Excitation Source</b>	Al K <sub>α</sub> monochromatic
<b>Source Energy</b>	1486.6 eV
<b>Source Strength</b>	180 W
<b>Source Size</b>	2 mm × 2 mm
<b>Analyzer Type</b>	spherical sector
<b>Incident Angle</b>	54°
<b>Emission Angle</b>	0°
<b>Analyzer Pass Energy:</b>	160 eV
<b>Analyzer Resolution</b>	2.4 eV
<b>Total Signal Accumulation Time</b>	560 s
<b>Total Elapsed Time</b>	1120 s
<b>Number of Scans</b>	4
<b>Effective Detector Width</b>	33.6 eV



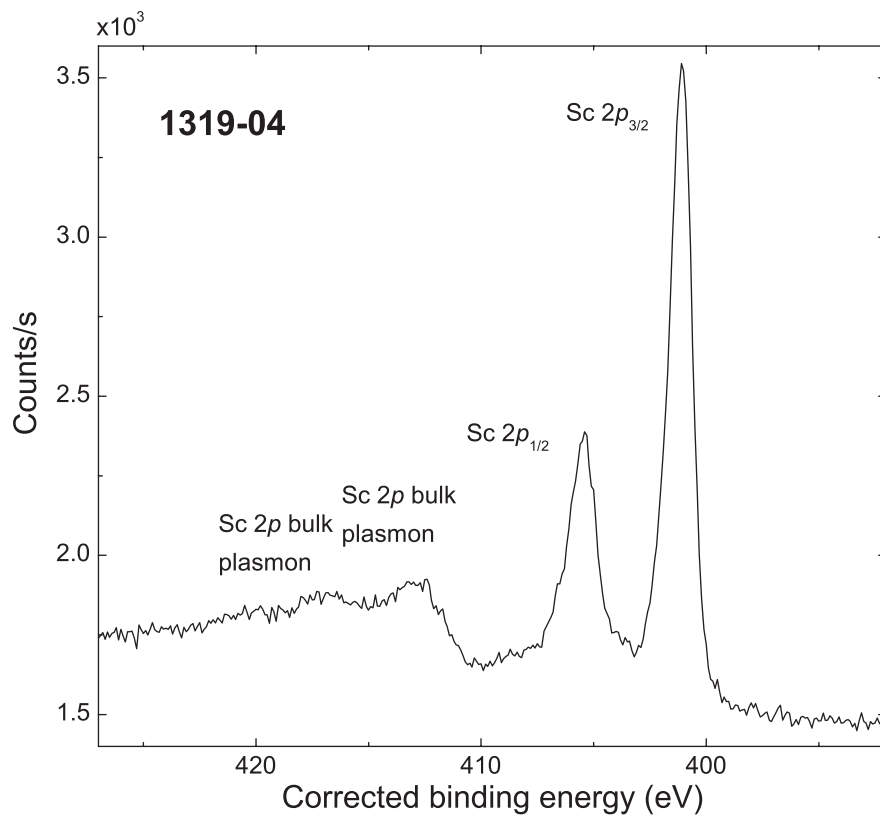
- Accession #: 01319-02
- Host Material: Single crystal DyScO<sub>3</sub>
- Technique: XPS
- Spectral Region: Dy 3d

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: 4206 s  
 Total Elapsed Time: 11566.5 s  
 Number of Scans: 20  
 Effective Detector Width: 4.2 eV



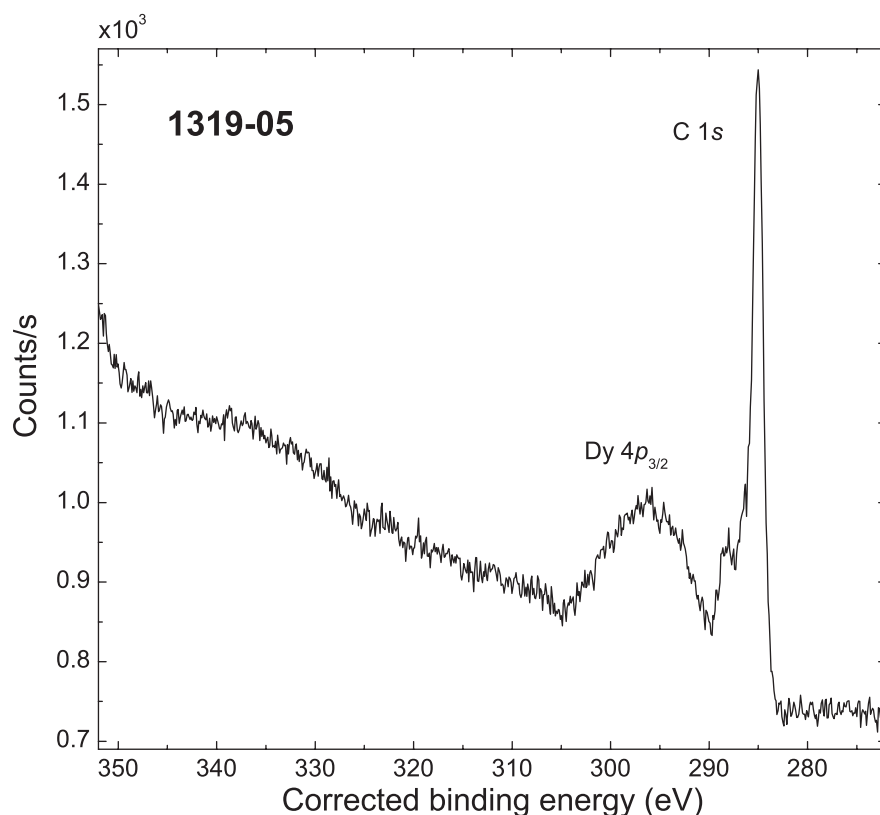
- Accession #: 01319-03
- Host Material: Single crystal DyScO<sub>3</sub>
- Technique: XPS
- Spectral Region: O 1s

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 #: 01319-04
- Host Material: Single crystal DyScO<sub>3</sub>
- Technique: XPS
- Spectral Region: Sc 2p

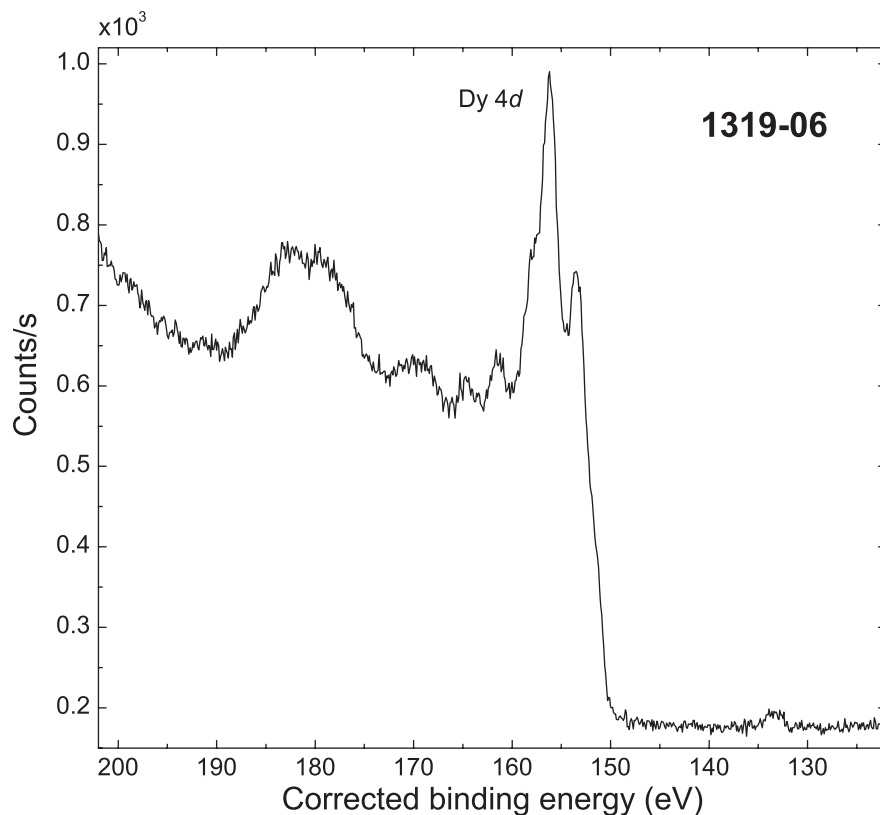
Instrument: Kratos Axis Ultra  
 Excitation Source: Al K<sub>α</sub> monochromatic  
 Source Energy: 1486.6 eV  
 Source Strength: 180 W  
 Source Size: 2 mm × 2 mm  
 Analyzer Type: spherical sector  
 Incident Angle: 54°  
 Emission Angle: 0°  
 Analyzer Pass Energy: 20 eV  
 Analyzer Resolution: 0.3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



- Accession #: 01319-05
- Host Material: Single crystal DyScO<sub>3</sub>
- Technique: XPS
- Spectral Region: Dy 4p; C 1s

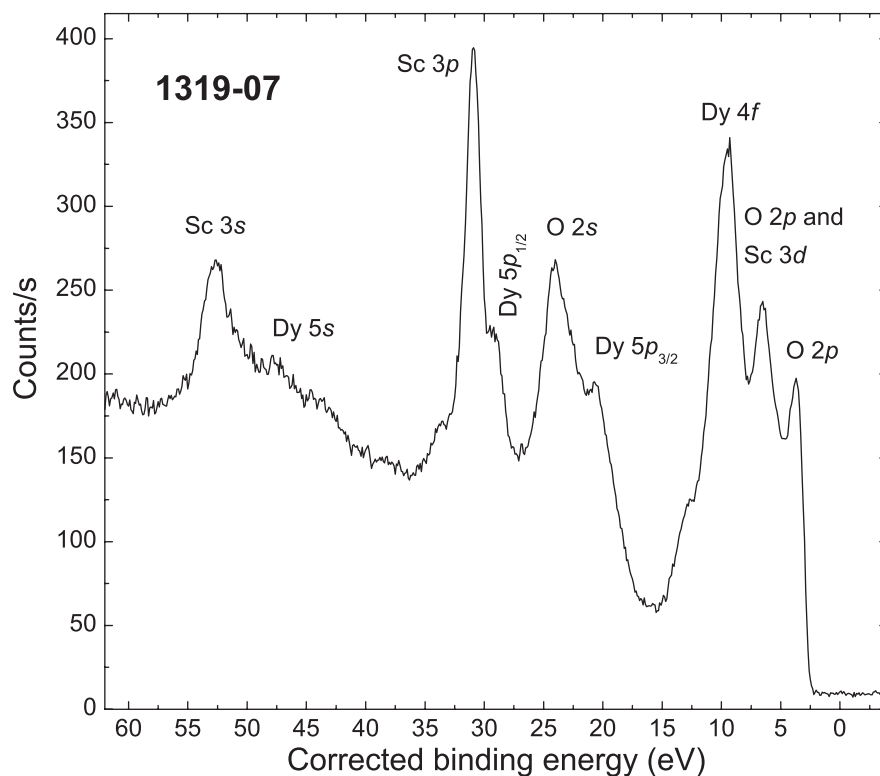
Instrument: Kratos Axis Ultra  
 Excitation Source: Al K<sub>α</sub> monochromatic  
 Source Energy: 1486.6 eV  
 Source Strength: 180 W  
 Source Size: 2 mm × 2 mm  
 Analyzer Type: spherical sector  
 Incident Angle: 54°  
 Emission Angle: 0°  
 Analyzer Pass Energy: 20 eV  
 Analyzer Resolution: 0.3 eV  
 Total Signal Accumulation Time: 4806 s  
 Total Elapsed Time: 13216.5 s  
 Number of Scans: 20  
 Effective Detector Width: 4.2 eV





- Accession #: 01319-06
- Host Material: Single crystal DyScO<sub>3</sub>
- Technique: XPS
- Spectral Region: Dy 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: 4806 s  
 Total Elapsed Time: 13216.5 s  
 Number of Scans: 20  
 Effective Detector Width: 4.2 eV



- Accession #: 01319-07
- Host Material: Single crystal DyScO<sub>3</sub>
- Technique: XPS
- Spectral Region: Sc 3s; Dy 5s; Sc 3p; Dy 5p; O 2s; 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: 9254 s  
 Total Elapsed Time: 25448.5 s  
 Number of Scans: 20  
 Effective Detector Width: 4.2 eV