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Single Crystal Perovskites Analyzed Using X-ray Photoelectron Spectroscopy: 4. $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$(001)

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X-ray photoelectron spectroscopy (XPS) was used to analyze a commercially available $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$ (001) bulk single crystal. XP spectra were obtained using incident monochromatic Al K_α radiation at 0.83401 nm. A survey spectrum together with La 3d, O 1s, C 1s, Sr 3p, Ta 4d, La 4p, Sr 3d, Al 2s, La 4d, Al 2p, Ta 5p, La 5s, Ta 4f, O 2s, Sr 4p and La 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, Sr 3d, La 4d, Al 2p Ta 4f lines and neglecting the components related to surface contaminants, XPS quantitative analysis reveals an altered stoichiometry of the air-exposed crystal surface of $La_{0.34}Sr_{0.88}Al_{0.82}Ta_{0.43}O_3$. © *2014 American Vacuum Society*.
[http://dx.doi.org/10.1116/11.20140904]

Keywords: *lanthanum strontium aluminum tantalum oxide; perovskite*

Accession #: 01313
Technique: XPS
Host Material: Single crystal $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
Instrument: Kratos Axis Ultra
Major Elements in Spectra: La, Sr, Al, Ta, O
Minor Elements in Spectra: C
Published Spectra: 11
Spectra in Electronic Record: 11
Spectral Category: comparison

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 engineering 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 complex oxide systems which have received the most attention in recent years. In particular, materials possessing the perovskite and substituded perovskite structure (with chemical formula ABO_3) 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 #01313)

Host Material: Single crystal $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$

CAS Registry #: 272780-20-8

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

Chemical Name: Lanthanum strontium aluminum tantalum oxide

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

Host Composition: $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$

Form: single crystal

Structure: cubic Pn3(bar)m mixed perovskite, a = 0.7730 nm (Ref. 7)

History & Significance: $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$ has been widely used as a designed substrate for a number of important applications. It originally rose to prominence as a competitive substrate for high TC oxide superconductors due to its low dielectric response, exceptional crystallinity, cubic symmetry, and small lattice constant (Ref. 8). Since then, it has been used as an epitaxial substrate for ferroelectric materials (Ref. 9), high-quality optoelectronic semiconductors (Ref. 10), and colossal magnetoresistive materials (Ref. 11). In order to gain an increased understanding of the surfaces and hetero-interfaces of perovskite-based materials, $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$ 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®, 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 = 1.8A, charge balance = 3V, filament bias = 1V

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 ($T = E^N$): $N = 0$

Excitation Source Window: not specified

Excitation Source: Al K_α, monochromatic

Source Energy: 1486.6 eV

Source Strength: 180 W

Source Beam Size: 2000 μm $\times$ 2000 μm

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: +1.956 eV for high-resolution spectra

Peak Shape and Background Method: Background: Custom three parameter Tougaard background (Ref. 12), U 4 Tougaard (B, C, D, T0 = 0) (Ref. 13), was used. O 1s, C 1s, Sr 3d, La 4d, Al 2p, Ta 4f: B = 299 eV^2, C = 542 eV^2, D = 275 eV^2.

Quantitation Method: Quantification was done using region and component definitions with CasaXPS version 2.3.15. Sensitivity factors were supplied by Kratos Analytical. Errors are given as ± 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
01313-02	La $3d_{5/2}$	834.1	1.80	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-02	La $3d_{5/2}$	838.4	2.02	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-02	La $3d_{3/2}$	850.9	1.80	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-02	La $3d_{3/2}$	855.2	2.19	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-03	O 1s	529.9	1.40	34260.9	0.780	40.85	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-03[a]	O 1s	531.0	1.46	4141.1	0.780	4.94	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-03[a]	O 1s	532.9	1.47	1608.4	0.780	1.92	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-04[a]	C 1s	285.0	1.19	3912.5	0.278	13.09	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-04[a]	C 1s	286.5	1.60	1063.9	0.278	3.56	C-hydroxide
01313-04[a]	C 1s	289.0	1.80	634.2	0.278	2.12	carbonate
01313-04	Sr $3p_{3/2}$	268.7	2.20	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-04	Sr $3p_{1/2}$	279.0	2.16	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-05	Ta $4d_{5/2}$	230.0	4.02	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-05	Ta $4d_{3/2}$	241.4	4.30	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-06	La $4p_{3/2}$	195.6	1.97	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-06	La $4p_{1/2}$	198.7	3.69	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-07	Sr 3d	···	···	23706.5	1.843	11.97	···
01313-07	Sr $3d_{5/2}$	132.9	1.00	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-07	Sr $3d_{3/2}$	134.7	1.00	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-08	Al 2s	118.5	1.75	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-09	La 4d	···	···	12145.2	2.475	4.56	···
01313-09	La $4d_{5/2}$	102.2	1.52	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-09	La $4d_{5/2}$	105.4	2.80	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-09	La $4d_{3/2}$	105.3	1.52	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-09	La $4d_{3/2}$	108.5	2.80	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-10	Al 2p	···	···	2322.7	0.193	11.19	···
01313-10	Al 2p	73.7	1.29	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-11	Ta $5p_{3/2}$	37.1	2.07	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-11	La 5s	34.9	1.39	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-11	Ta 4f	···	···	19224.5	3.082	5.80	···
01313-11	Ta $4f_{7/2}$	25.9	1.01	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-11	Ta $4f_{5/2}$	27.7	1.01	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-11	O 2s	21.4	2.33	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-11	Sr $4p_{3/2}$	18.7	1.23	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-11	Sr $4p_{1/2}$	19.8	1.23	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-11	La $5p_{3/2}$	17.1	1.44	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-11	La $5p_{1/2}$	19.5	1.44	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-11[b]	valence band	8.9	2.00	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-11[c]	valence band	7.4	1.50	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-11[c]	valence band	5.9	1.50	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
01313-11[d]	valence band maximum (VBM)	3.3	2.11	···	···	···	$(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$

[a] Result of exposure to air
[b] O 2p and Al 3s (Refs. 14–16)
[c] O 2p, Al 3p, and Ta 5d (Refs. 14–16)
[d] 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.

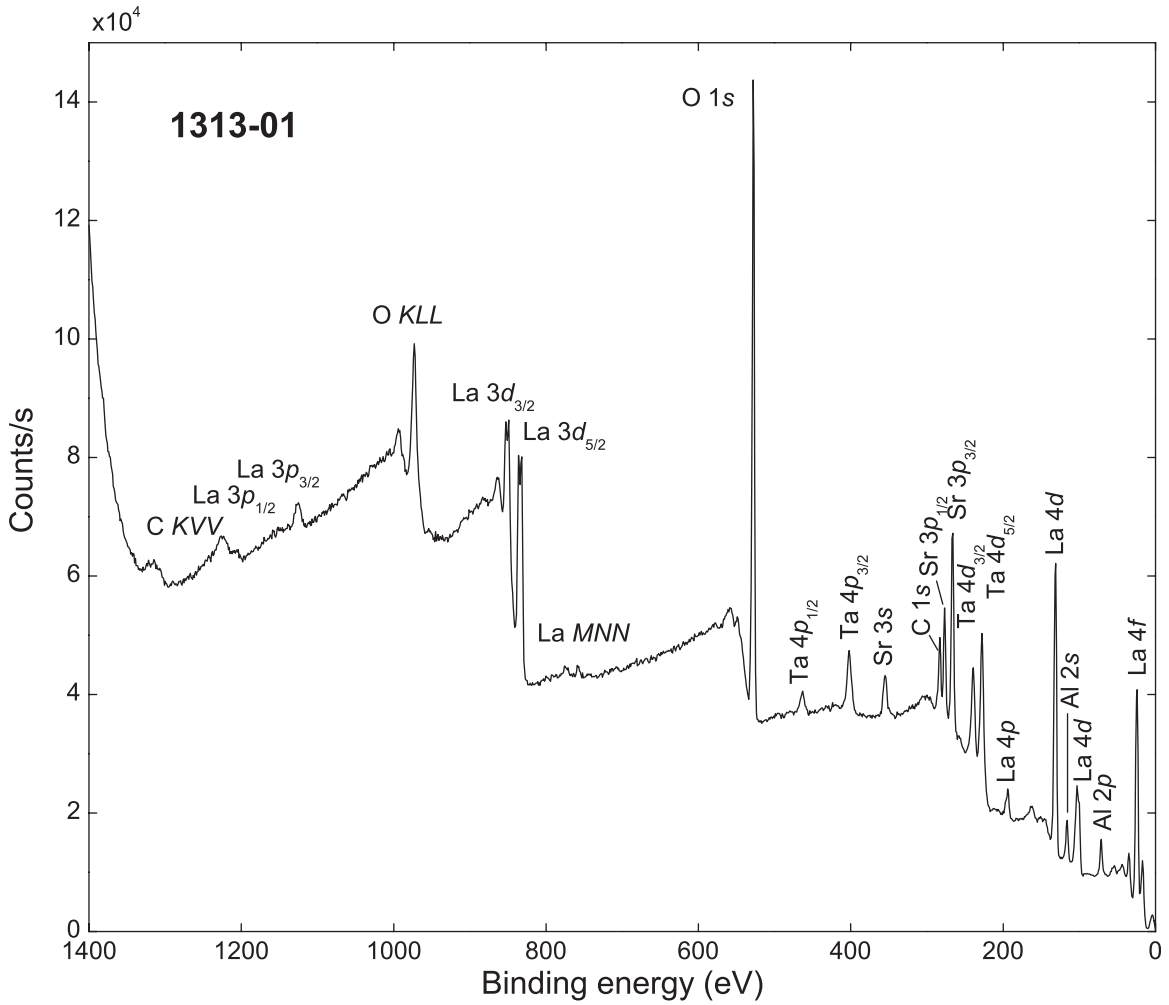
ANALYZER CALIBRATION TABLE

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
	Au $4f_{7/2}$	84.0	0.72	151917.9	···	···	···
	Ag $3d_{5/2}$	368.2	0.58	230506.2	···	···	···
	Cu $2p_{3/2}$	932.6	0.88	410979.8	···	···	···

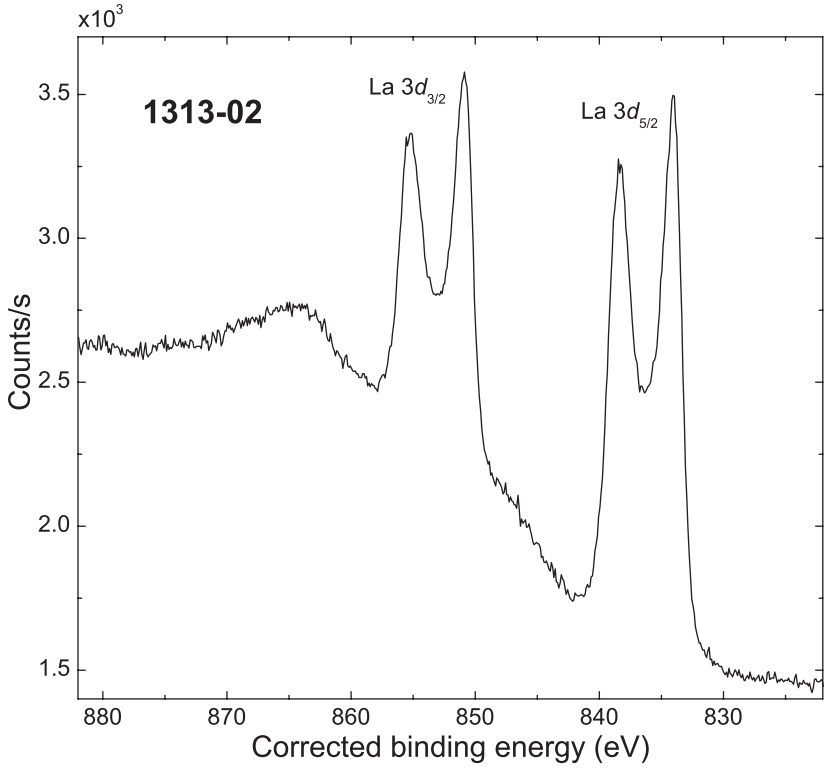
GUIDE TO FIGURES

Spectrum (Accession) #	Spectral Region	Voltage Shift*	Multiplier	Baseline	Comment #
1313-01	survey	0	1	0	
1313-02	La 3d	−1.956	1	0	
1313-03	O 1s	−1.956	1	0	
1313-04	C 1s, Sr 3p	−1.956	1	0	
1313-05	Ta 4d	−1.956	1	0	
1313-06	La 4p	−1.956	1	0	
1313-07	Sr 3d	−1.956	1	0	
1313-08	Al 2s	−1.956	1	0	
1313-09	La 4d	−1.956	1	0	
1313-10	Al 2p	−1.956	1	0	
1313-11	Ta $5p_{3/2}$, La 5s, Ta 4f, O 2s, Sr 4p	−1.956	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.

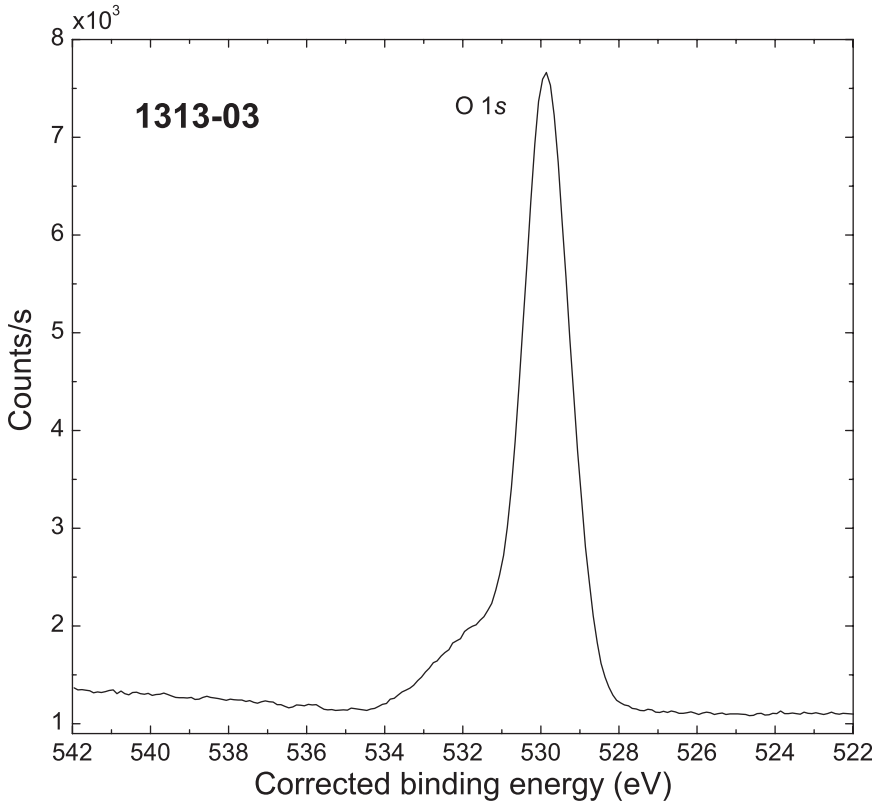


Accession #	01313–01
Host Material	Single crystal $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
Technique	XPS
Spectral Region	survey
Instrument	Kratos Axis Ultra
Excitation Source	Al K_α monochromatic
Source Energy	1486.6 eV
Source Strength	180 W
Source Size	2 mm × 2 mm
Analyzer Type	spherical sector
Incident 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



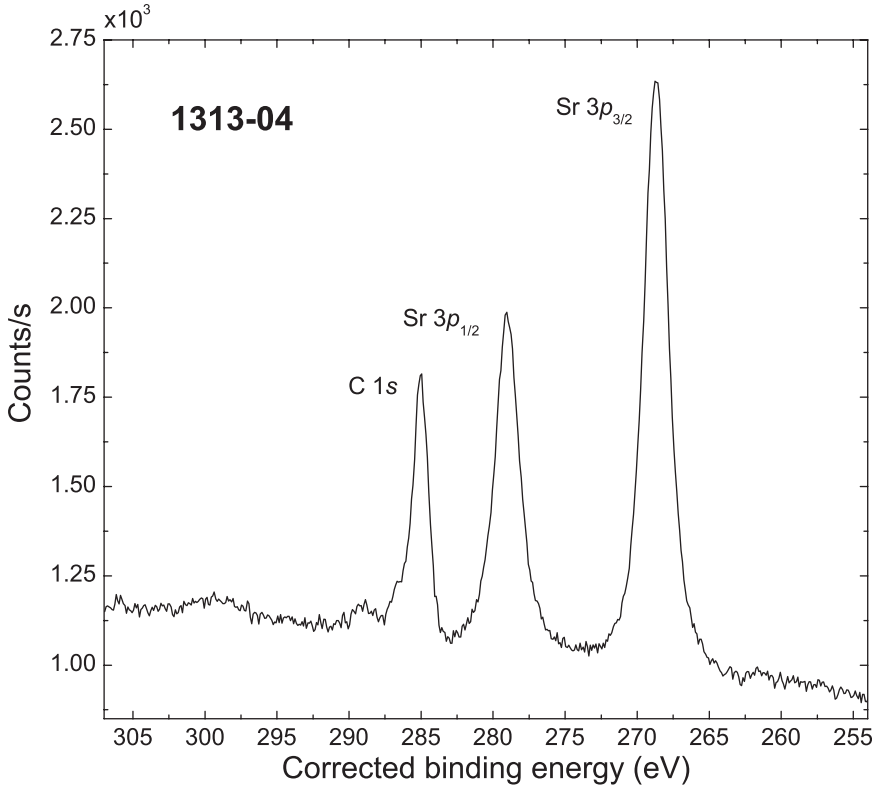
- **Accession #:** 01313–02
- **Host Material:** Single crystal $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
- **Technique:** XPS
- **Spectral Region:** La 3*d*

Instrument: Kratos Axis Ultra
Excitation Source: Al K_α 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



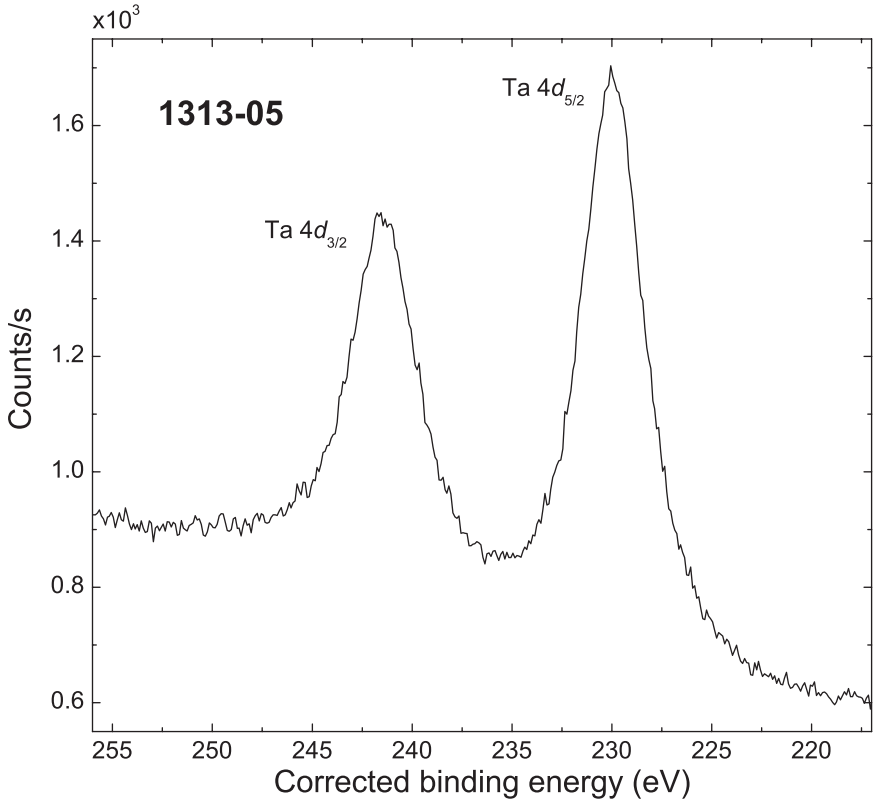
- **Accession #:** 01313–03
- **Host Material:** Single crystal $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
- **Technique:** XPS
- **Spectral Region:** O 1*s*

Instrument: Kratos Axis Ultra
Excitation Source: Al K_α 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: 1333 s
Total Elapsed Time: 3665.75 s
Number of Scans: 20
Effective Detector Width: 4.2 eV



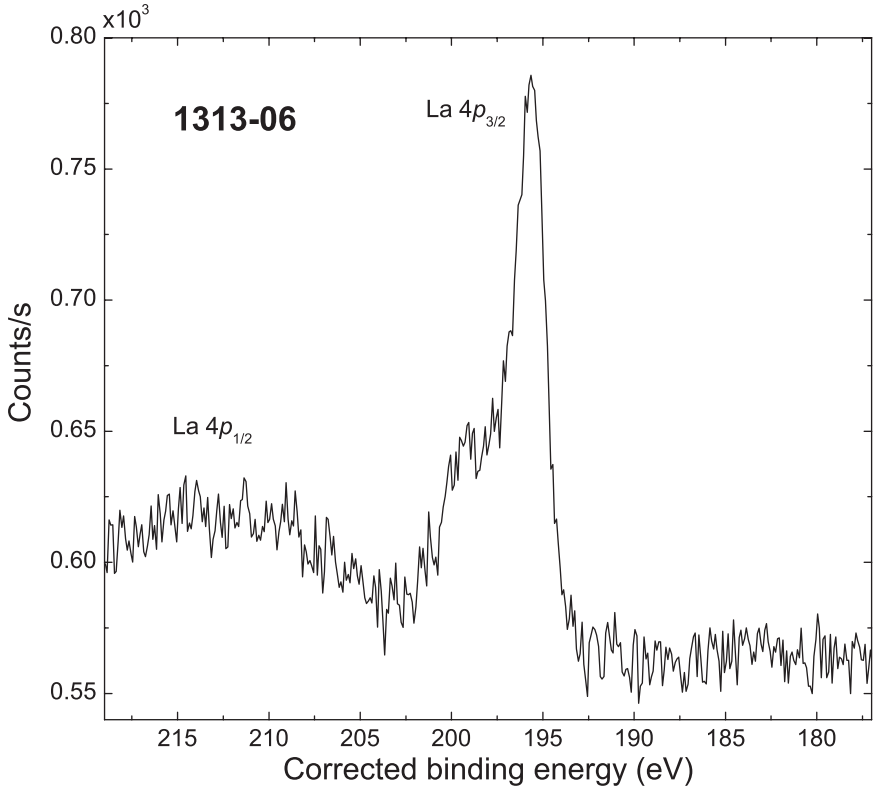
- **Accession #:** 01313–04
- **Host Material:** Single crystal $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
- **Technique:** XPS
- **Spectral Region:** C 1*s*; Sr 3*p*

Instrument: Kratos Axis Ultra
Excitation Source: Al K_α 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: 3186 s
Total Elapsed Time: 8761.5 s
Number of Scans: 20
Effective Detector Width: 4.2 eV



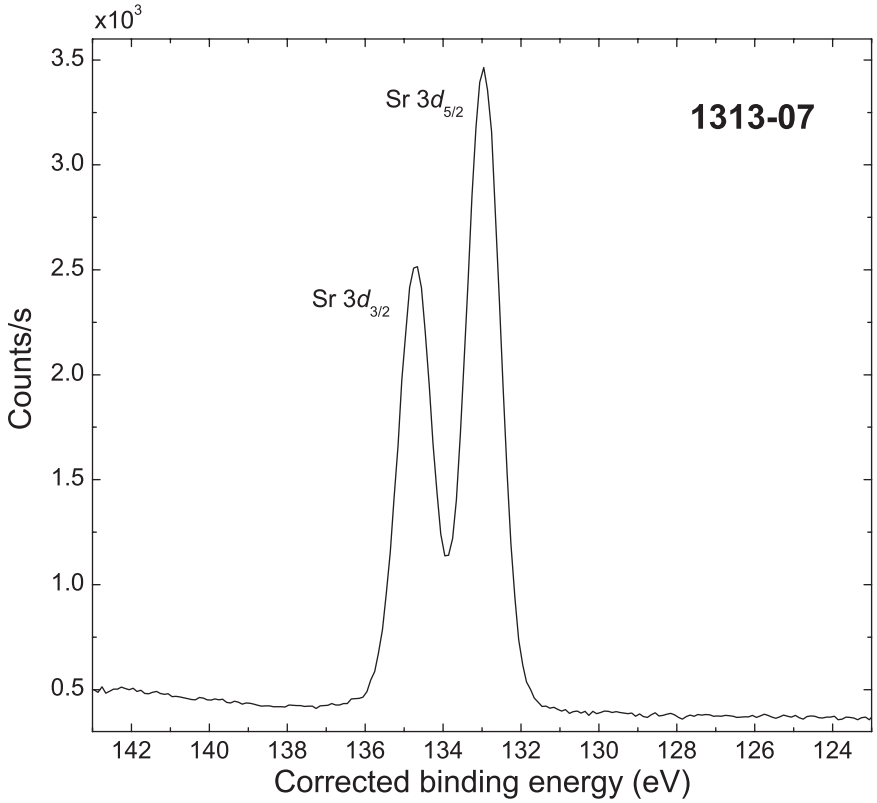
- **Accession #:** 01313–05
- **Host Material:** Single crystal $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
- **Technique:** XPS
- **Spectral Region:** Ta 4*d*

Instrument: Kratos Axis Ultra
Excitation Source: Al K_α 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: 2346 s
Total Elapsed Time: 6451.5 s
Number of Scans: 20
Effective Detector Width: 4.2 eV



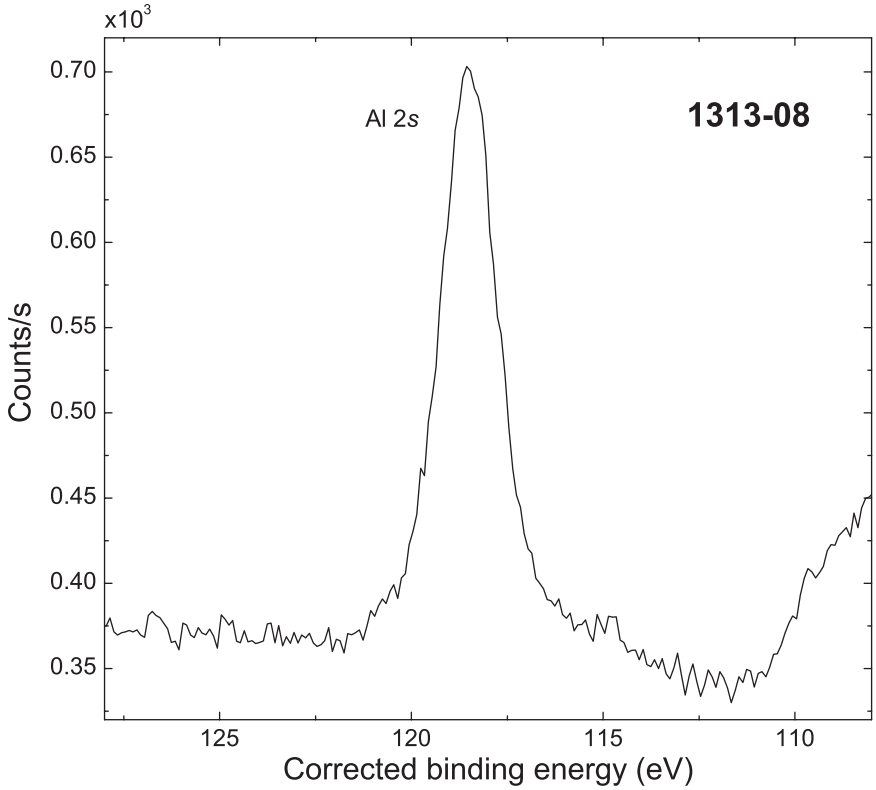
- **Accession #:** 01313–06
- **Host Material:** Single crystal $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
- **Technique:** XPS
- **Spectral Region:** La 4*p*

Instrument: Kratos Axis Ultra
Excitation Source: Al K_α 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: 2526 s
Total Elapsed Time: 6946.5 s
Number of Scans: 20
Effective Detector Width: 4.2 eV



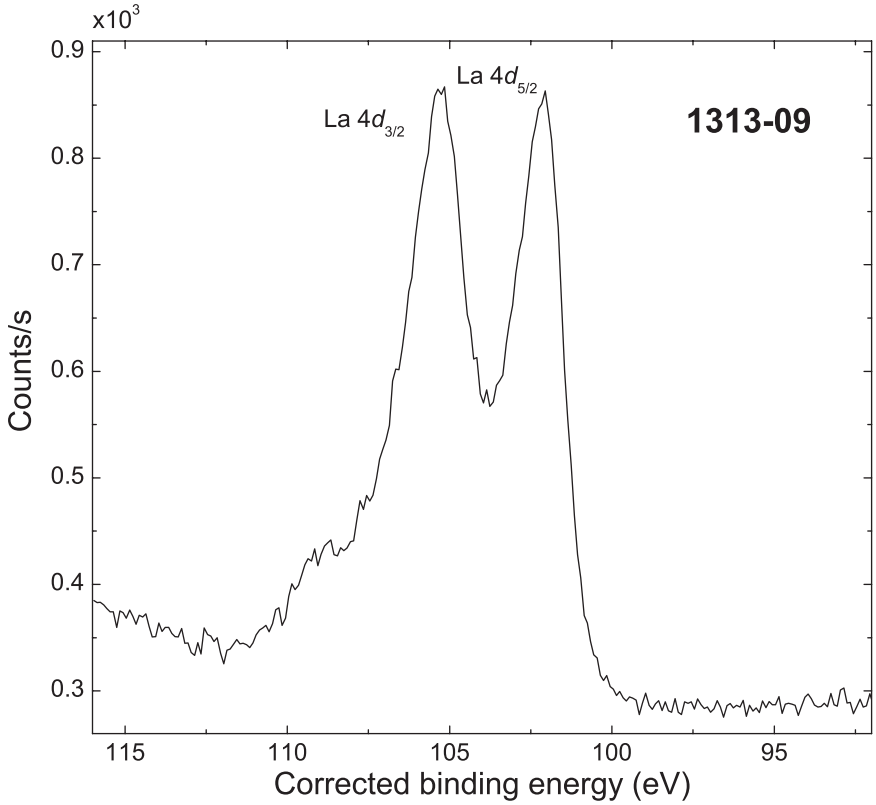
- **Accession #:** 01313–07
- **Host Material:** Single crystal $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
- **Technique:** XPS
- **Spectral Region:** Sr 3*d*

Instrument: Kratos Axis Ultra
Excitation Source: Al K_α 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



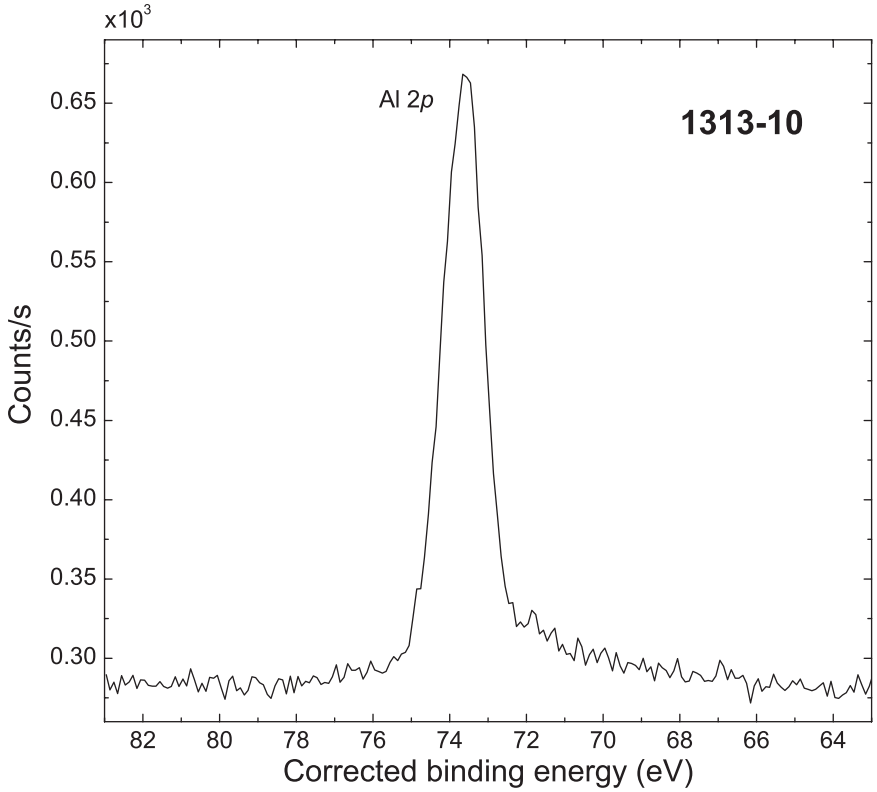
- **Accession #:** 01313–08
- **Host Material:** Single crystal $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
- **Technique:** XPS
- **Spectral Region:** Al 2*s*

Instrument: Kratos Axis Ultra
Excitation Source: Al K_α 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: 1608 s
Total Elapsed Time: 4422 s
Number of Scans: 20
Effective Detector Width: 4.2 eV



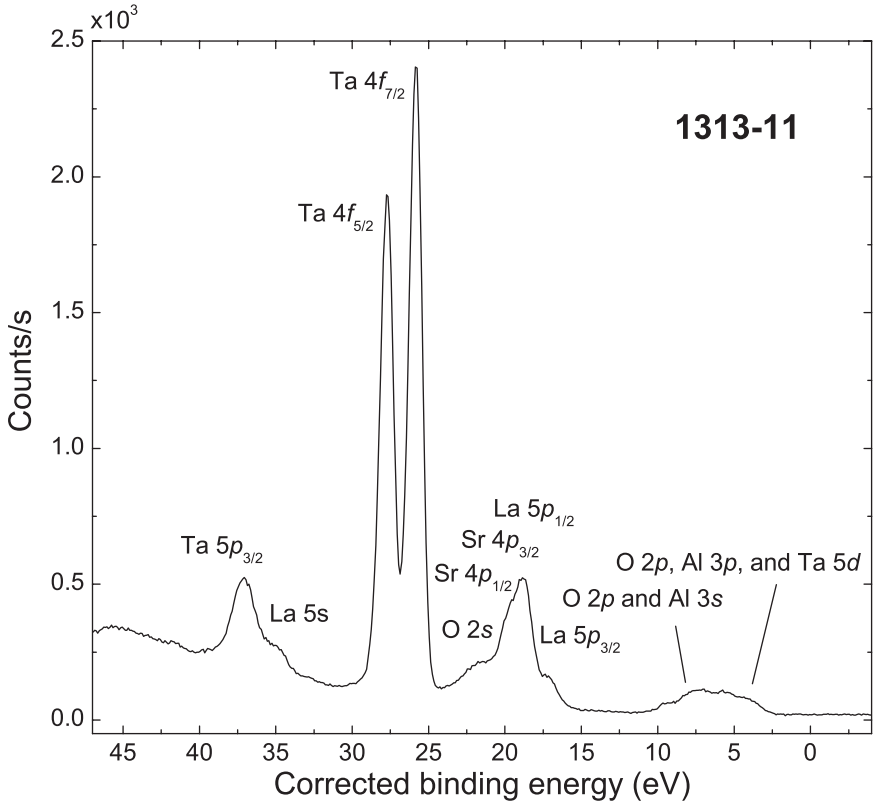
- **Accession #:** 01313–09
- **Host Material:** Single crystal $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
- **Technique:** XPS
- **Spectral Region:** La 4*d*

Instrument: Kratos Axis Ultra
Excitation Source: Al K_α 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: 1446 s
Total Elapsed Time: 4422 s
Number of Scans: 20
Effective Detector Width: 4.2 eV



- **Accession #:** 01313–10
- **Host Material:** Single crystal $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
- **Technique:** XPS
- **Spectral Region:** Al 2*p*

Instrument: Kratos Axis Ultra
Excitation Source: Al K_α 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: 1608 s
Total Elapsed Time: 4422 s
Number of Scans: 20
Effective Detector Width: 4.2 eV



- **Accession #:** 01313–11
- **Host Material:** Single crystal $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$
- **Technique:** XPS
- **Spectral Region:** Ta 5$p_{3/2}$; La 5*s*; Ta 4*f*; O 2*s*; Sr 4*p*; La 5*p*; valence band

Instrument: Kratos Axis Ultra
Excitation Source: Al K_α 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: 4088 s
Total Elapsed Time: 11242 s
Number of Scans: 20
Effective Detector Width: 4.2 eV