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Bismuth acetate by XPS

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Bismuth (III) acetate was analyzed using x-ray photoelectron spectroscopy (XPS). The specimen is a powder purchased from Sigma Aldrich. Sample was fixed to a stainless-steel sample holder with copper 3M™ double-sided adhesive tape. Survey spectra, Bi 4f, O 1s, C 1s, Bi 4d, Bi 5d, Bi 6s core levels and valence band spectra were acquired. Results showed how the elements in the structure of bismuth acetate are related.

Keywords: Bismuth acetate; XPS; precursor; acetic acid bismuth

Accession#: 01648

Technique: XPS

Host Material: Bismuth acetate

Instrument: SPECS PHOIBOS 150

Major Elements in Spectra: Bi, O, C

Minor Elements in Spectra: None

Published Spectra: 6

Spectra in Electronic Record: 6

Spectral Category: comparison

INTRODUCTION

Bismuth (III) acetate is an inorganic compound consisting of positive bismuth ions (3+ charge) and negative acetate ions (1- charge) (Fig. 1). It is commonly employed as a precursor for the synthesis of several materials with different applications such as, bismuth sulfide for solar cells (Refs. 1 and 2), bismuth titanate nanorods as photocatalysts (Refs. 3 - 5), sensitization of TiO_2 nanotubes on photoelectrochemical reactions (Ref. 6) and it is also used in the synthesis of triarylbismuth compounds (Ref. 7).

C

Fig. 1. Structure of bismuth acetate

XPS survey spectrum shows that the sample does not present intrusive elements, since the presence of only carbon, oxygen and bismuth is evidenced.

Bi 4f high resolution spectrum was decomposed in four contributions corresponding to two different chemical environments. The peaks centered at 159.3 and 164.6 eV were assigned to bismuth bonding with oxygen, O-**Bi**-O, and signals at 160.9 and 166.2 eV were associated with bismuth bonding with oxygen and the last one with carbon, **Bi**-O-C. O 1s spectrum displayed five different species. First, at 530.3 eV corresponds to oxygen bonding with bismuth, **O**-Bi-**O**. Second, carbon double and single bonded with oxygen, **O**=C-**O** at 531.1 eV. Third, at 531.8 eV was assigned to (C=**O**)-O-Bi species. Fourth, oxygen single bounded with carbon, C-**O** at 532.6 eV. And finally, 533.4 eV has an overlap between (C=O)-**O**-Bi and adsorbed **O**H. High resolution spectrum of C 1s shows four chemical species. **C**-(C,H) at 284.8 eV, carbon carbon bonding or adventitious carbon used as a reference (Ref. 8). Carbon single bounded with oxygen, **C**-O at 286.3 eV. Carbon double and single bonded with oxygen, O=**C**-O at 288.4 eV, and carbon double bonded with oxygen related with bismuth, (**C**=O)-O-Bi at 290 eV. Bi 4d and Bi 5d signals were also fitted to confirm the surface chemical state found in Bi 4f. The results of this work evidenced the bonds between bismuth, oxygen and carbon in the structure of bismuth acetate.

SPECIMEN DESCRIPTION (ACCESSION # 01648)

Host Material: Bismuth acetate

CAS Registry #: 22306-37-2

Host Material Characteristics: homogeneous; solid; polycrystalline; unknown conductivity; inorganic compound; Powder

Chemical Name: Bismuth triacetate

Source: Sigma Aldrich

Host Composition: Bismuth (III) acetate (99.99%)

Form: Powder

Structure: $Bi(CH_3COO)_3$

History & Significance: Bismuth (III) acetate powder was ground and fixed to a sample holder with copper 3M™ double-sided adhesive tape. The sample was exposed to the environment for about 2 minutes, time that was spent to prepare the sample and then introduce it to the platform.

As Received Condition: As powder

Analyzed Region: same as host material

Ex Situ Preparation/Mounting: As received.

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In Situ Preparation: None

Charge Control: Electron flood gun (SPECS FG-500) operated at 70 μA and 4eV

Temp. During Analysis: 300 K

Pressure During Analysis: < 1 x 10^{-7} Pa

Pre-analysis Beam Exposure: Not applicable s

INSTRUMENT DESCRIPTION

Manufacturer and Model: SPECS PHOIBOS 150 – 2D-DLD - SPECS Surface Nano Analysis GmbH

Analyzer Type: spherical sector

Detector: other

Number of Detector Elements: 25

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

■*Spectrometer*

Analyzer Mode: constant pass energy

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

Excitation Source Window: Mylar window, allows high X-ray transmission: 88% for Al Kα.

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 200 W

Source Beam Size: 2000 μm x 2000 μm

Signal Mode: multichannel direct

■*Geometry*

Incident Angle: 55 °

Source-to-Analyzer Angle: 55 °

Emission Angle: 0 °

Specimen Azimuthal Angle: Not applicable

Acceptance Angle from Analyzer Axis: 16 °

Analyzer Angular Acceptance Width: 16 ° x 16 °

■*Ion Gun*

Manufacturer and Model: SPECS IQE 12/38

Energy: 5000 eV

Current: 70 mA

Current Measurement Method: biased stage

Sputtering Species: Ar^+

Spot Size (unrastered): 3000 μm x 3000 μm

Raster Size: Not applicable μm x μm

Incident Angle: 54°

Polar Angle: 55°

Azimuthal Angle: 45°

Comment: The specimen was analyzed as loaded. The ion gun was used only for cleaning the Ag reference foil.

DATA ANALYSIS METHOD

Energy Scale Correction: Binding energy of the adventitious carbon, C-(C,H) at 284.8 eV (Ref. 8) was used as reference to adjust the binding energy scale of the spectra.

Recommended Energy Scale Shift: 2.54 eV

Peak Shape and Background Method: Peak position and width were determined from fitting the spectra using a mixed Gaussian– Lorentzian, GL (30) function after subtraction of a Shirley background using the CasaXPS Software.

Quantitation Method: Peak areas were obtained from fitting the spectra and relative sensitivity factors from the atomic photoionization cross section of each core level provided by SPECS Prodigy library.

ACKNOWLEDGMENTS

Authors thank to Laboratorio Central en Ciencia de Superficies (SurfLab-UIS) from Universidad Industrial de Santander for providing their facilities to perform the XPS measurements and to project 2321 of Vicerrectoría de Investigación y Extensión (VIE-UIS) from Universidad Industrial de Santander.

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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**
01648-02	Bi 4f	…	…	2.98×10^4	25.66	15.28	…
01648-02	Bi $4f_{7/2}$	159.3	1.52	…	…	…	O-**Bi**-O
01648-02	Bi $4f_{5/2}$	164.6	1.52	…	…	…	O-**Bi**-O
01648-02	Bi $4f_{7/2}$	160.9	1.90	…	…	…	**Bi**-O-C
01648-02	Bi $4f_{5/2}$	166.2	1.90	…	…	…	**Bi**-O-C
01648-03	O 1s	…	…	7.49×10^3	2.77	40.15	…
01648-03	O 1s	530.3	2.00	…	…	…	**O**-Bi-**O**
01648-03	O 1s	531.1	2.00	…	…	…	**O**=C-**O**
01648-03	O 1s	531.8	2.00	…	…	…	(C=**O**)-O-Bi
01648-03	O 1s	532.6	2.00	…	…	…	C-**O**
01648-03	O 1s	533.4	2.00	…	…	…	(C=O)-**O**-Bi, **O**H
01648-04	C 1s	…	…	3.30×10^3	1.00	44.57	…
01648-04	C 1s	284.8	1.74	…	…	…	**C**-(C,H)
01648-04	C 1s	286.3	1.74	…	…	…	**C**-O
01648-04	C 1s	288.4	1.74	…	…	…	O=**C**-O
01648-04	C 1s	290.0	1.74	…	…	…	(**C**=O)-O-Bi
01648-05	Bi 4d	…	…	1.90×10^4	…	…	…
01648-05	Bi $4d_{5/2}$	442.1	4.00	…	…	…	O-**Bi**-O
01648-05	Bi $4d_{3/2}$	465.9	4.00	…	…	…	O-**Bi**-O
01648-05	Bi $4d_{5/2}$	444.4	3.50	…	…	…	**Bi**-O-C
01648-05	Bi $4d_{3/2}$	468.2	3.50	…	…	…	**Bi**-O-C
01648-06	Bi 5d	…	…	4.14×10^3	…	…	…
01648-06	Bi $5d_{5/2}$	26.1	1.58	…	…	…	O-**Bi**-O
01648-06	Bi $5d_{3/2}$	29.1	1.58	…	…	…	O-**Bi**-O
01648-06	Bi $5d_{5/2}$	27.9	2.00	…	…	…	**Bi**-O-C
01648-06	Bi $5d_{3/2}$	30.9	2.00	…	…	…	**Bi**-O-C
01648-06	Bi 6s	11.7	2.99	0.74×10^2	…	…	…
01648-06[a]	VBM	1.73	…	…	…	…	…

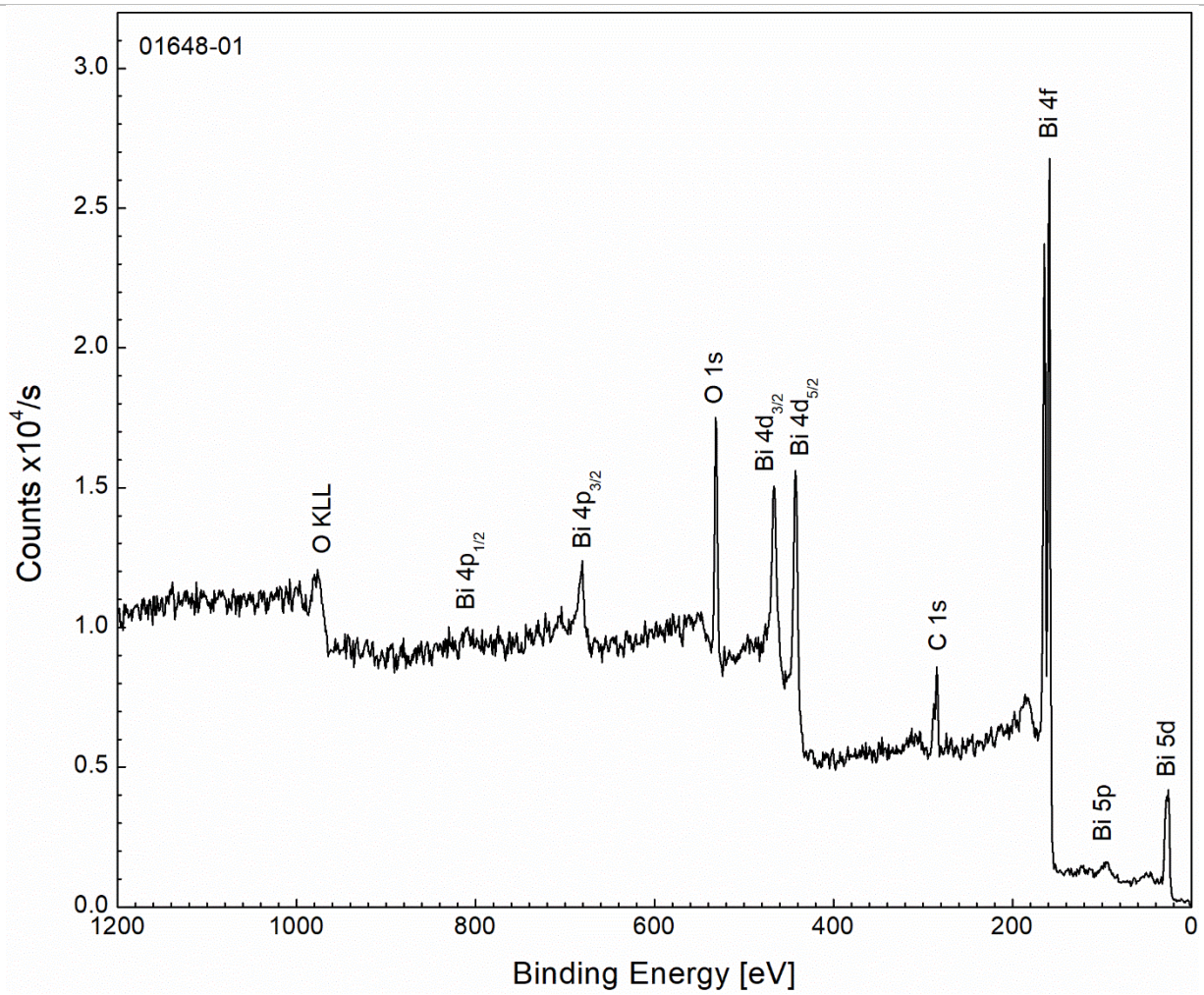
[a] Valence band maximum (VBM)

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 $3d_{5/2}$	368.3	0.50	0.15×10^6	...	...	...

GUIDE TO FIGURES					
Spectrum (Accession) #	**Spectral Region**	**Voltage Shift***	**Multiplier**	**Baseline**	**Comment #**
01648-01	Survey	0	1	0	1
01648-02	Bi 4f	-2.54	1	0	1
01648-03	O 1s	-2.54	1	0	1
01648-04	C 1s	-2.54	1	0	1
01648-05	Bi 4d	-2.54	1	0	1
01648-06	Bi 5d, Bi 6s, VB	-2.54	1	0	1

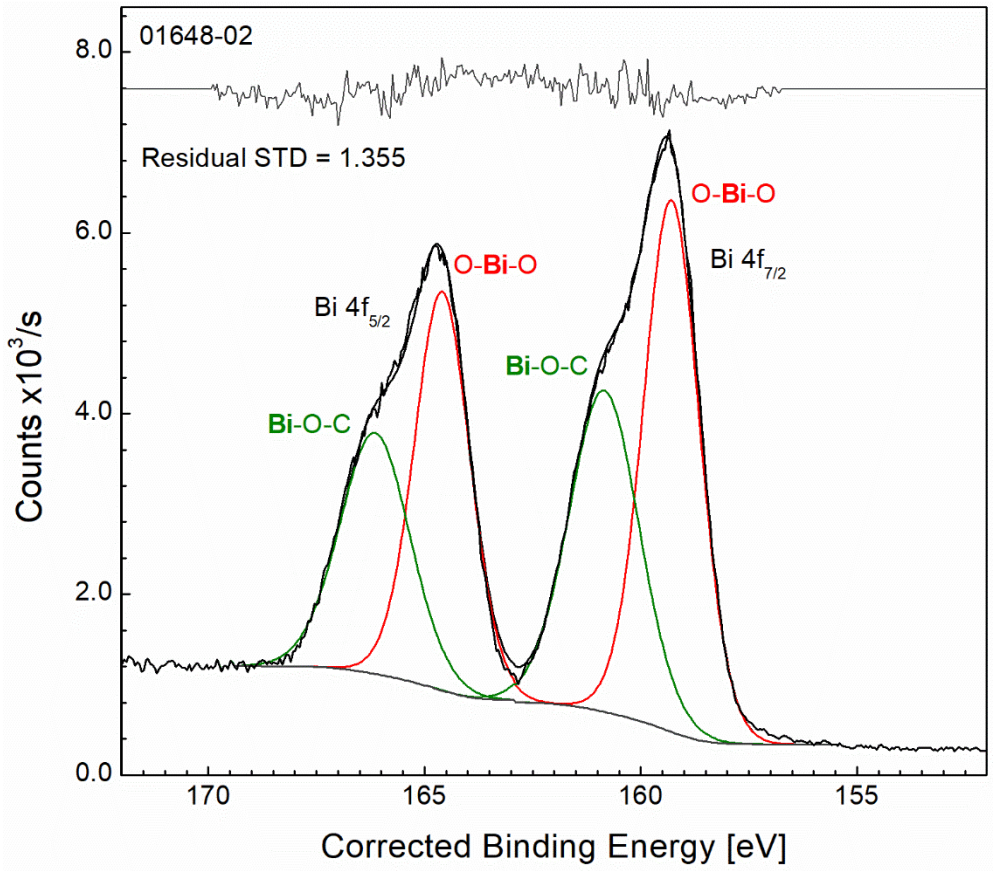
*Voltage shift of the archived (as-measured) spectrum relative to the printed figure. The figure reflects the recommended energy scale correction due to a calibration correction, sample charging, flood gun, or other phenomenon.

1, Bismuth (III) acetate powder



Publish in *Surface Science Spectra*: Yes ☒ No ☐

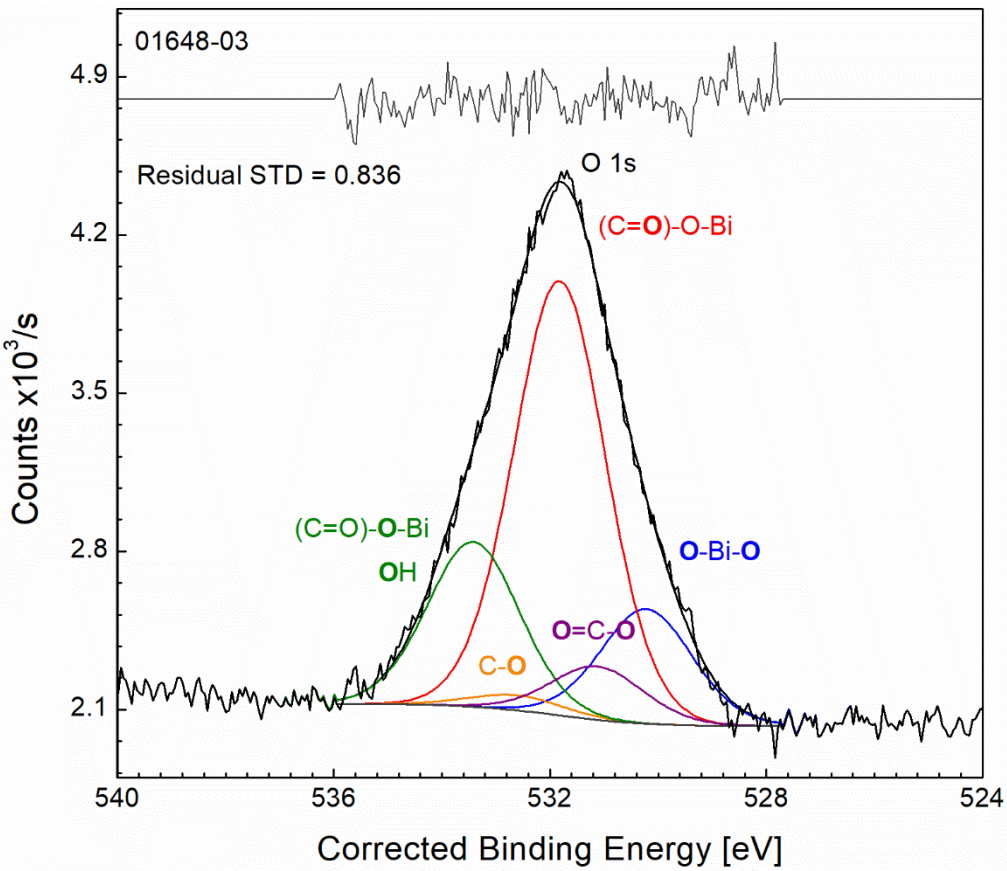
Accession #	01648-01
Host Material	Bismuth acetate
Technique	XPS
Spectral Region	survey
Instrument	SPECS PHOIBOS 150
Excitation Source	Al Ka monochromatic
Source Energy	1486.6 eV
Source Strength	200 W
Source Size	2 mm x 2 mm
Analyzer Type	spherical sector analyzer
Incident Angle	55˚
Emission Angle	0˚
Analyzer Pass Energy	100 eV
Analyzer Resolution	1.7 eV
Total Signal Accumulation Time	122 s
Total Elapsed Time	260 s
Number of Scans	1
Effective Detector Width	5.28 eV



Publish in *SSS*: Yes ☒ No ☐

- **Accession #:** 01648-02
- **Host Material:** Bismuth acetate
- **Technique:** XPS
- **Spectral Region:** Bi 4f

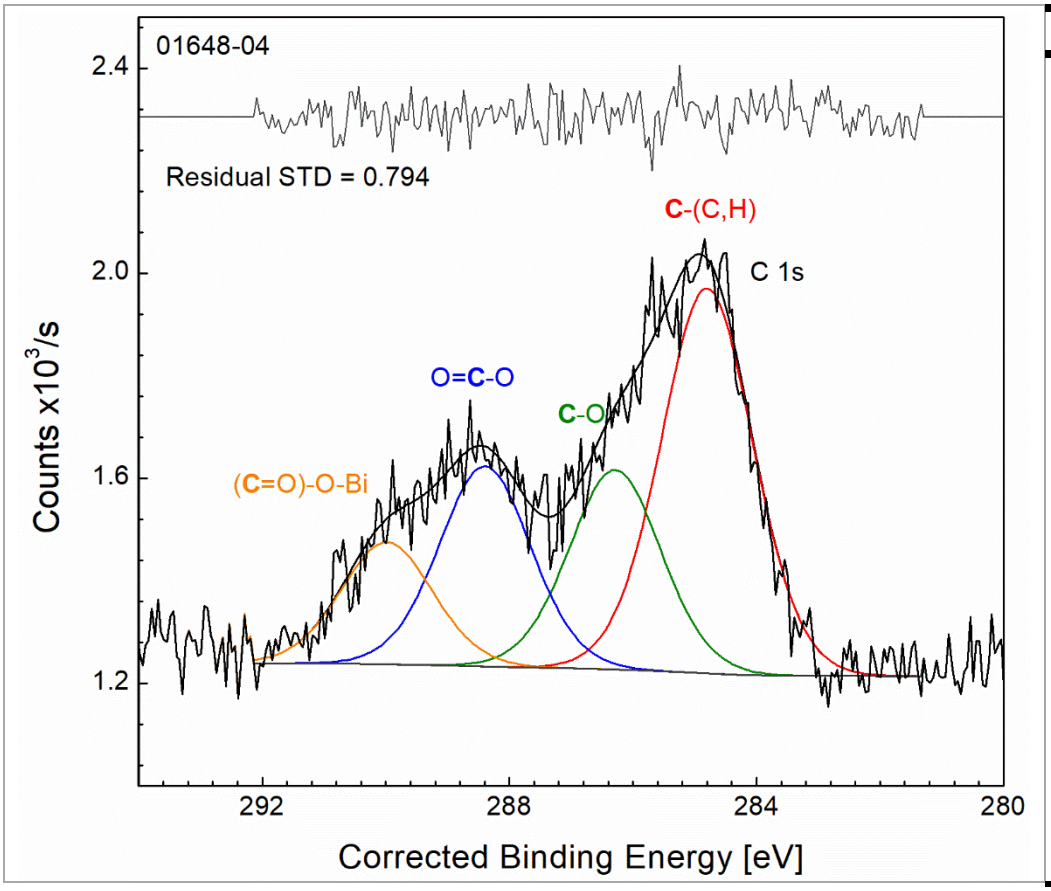
Instrument: SPECS PHOIBOS 150
Excitation Source: Al Ka monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 2 mm x 2 mm
Analyzer Type: spherical sector
Incident Angle: 55 ˚
Emission Angle: 0 ˚
Analyzer Pass Energy 30 eV
Analyzer Resolution: 0.5 eV
Total Signal Accumulation Time: 601 s
Total Elapsed Time: 954 s
Number of Scans: 8
Effective Detector Width: 2.64 eV



Publish in *SSS*: Yes ☒ No ☐

- **Accession #:** 01648-03
- **Host Material:** Bismuth acetate
- **Technique:** XPS
- **Spectral Region:** O 1s

Instrument: SPECS PHOIBOS 150
Excitation Source: Al Ka monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 2 mm x 2 mm
Analyzer Type: spherical sector
Incident Angle: 55 ˚
Emission Angle: 0 ˚
Analyzer Pass Energy 30 eV
Analyzer Resolution: 0.5 eV
Total Signal Accumulation Time: 421 s
Total Elapsed Time: 668 s
Number of Scans: 8
Effective Detector Width: 2.64 eV



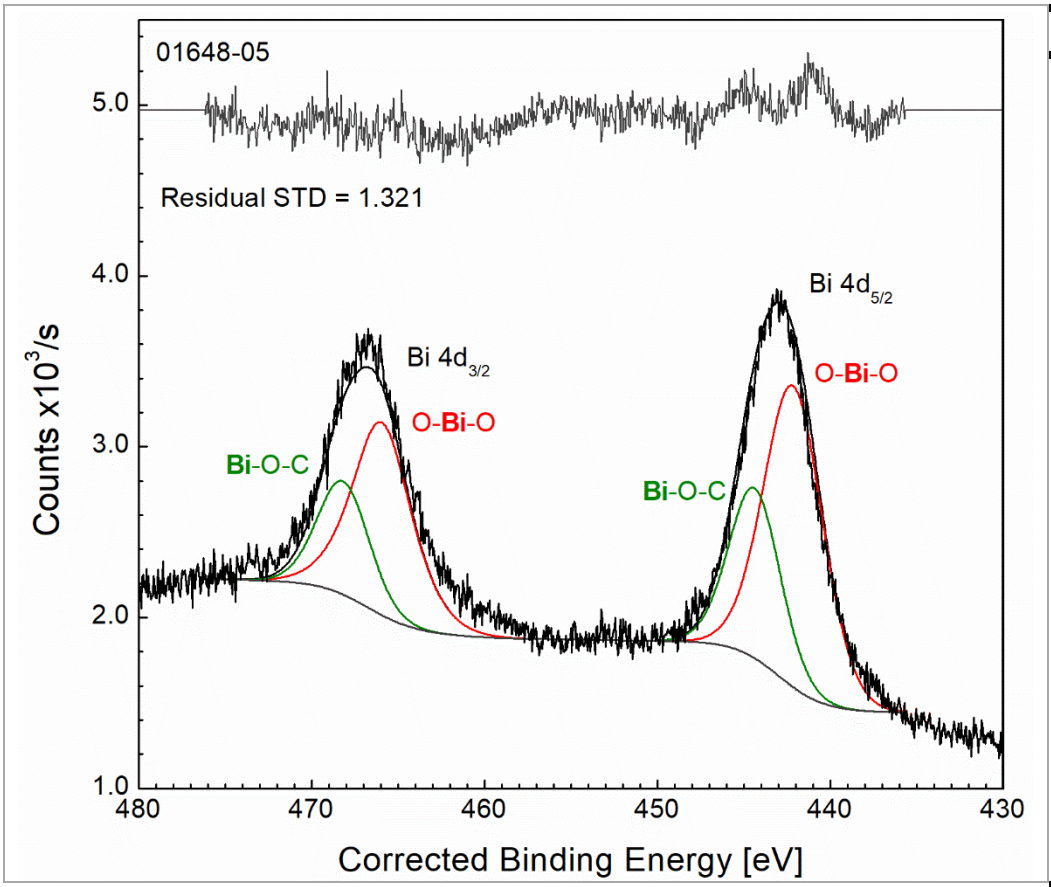
Publish in *SSS*: Yes ☒ No ☐

- **Accession #:** 01648-04
- **Host Material:** Bismuth acetate
- **Technique:** XPS
- **Spectral Region:** C 1s

Instrument: SPECS PHOIBOS 150
Excitation Source: Al Ka monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 2 mm x 2 mm
Analyzer Type: spherical sector
Incident Angle: 55 ˚
Emission Angle: 0 ˚
Analyzer Pass Energy 30 eV
Analyzer Resolution: 0.5 eV
Total Signal Accumulation Time: 211 s
Total Elapsed Time: 335 s

Number of Scans: 4

Effective Detector Width: 2.64 eV



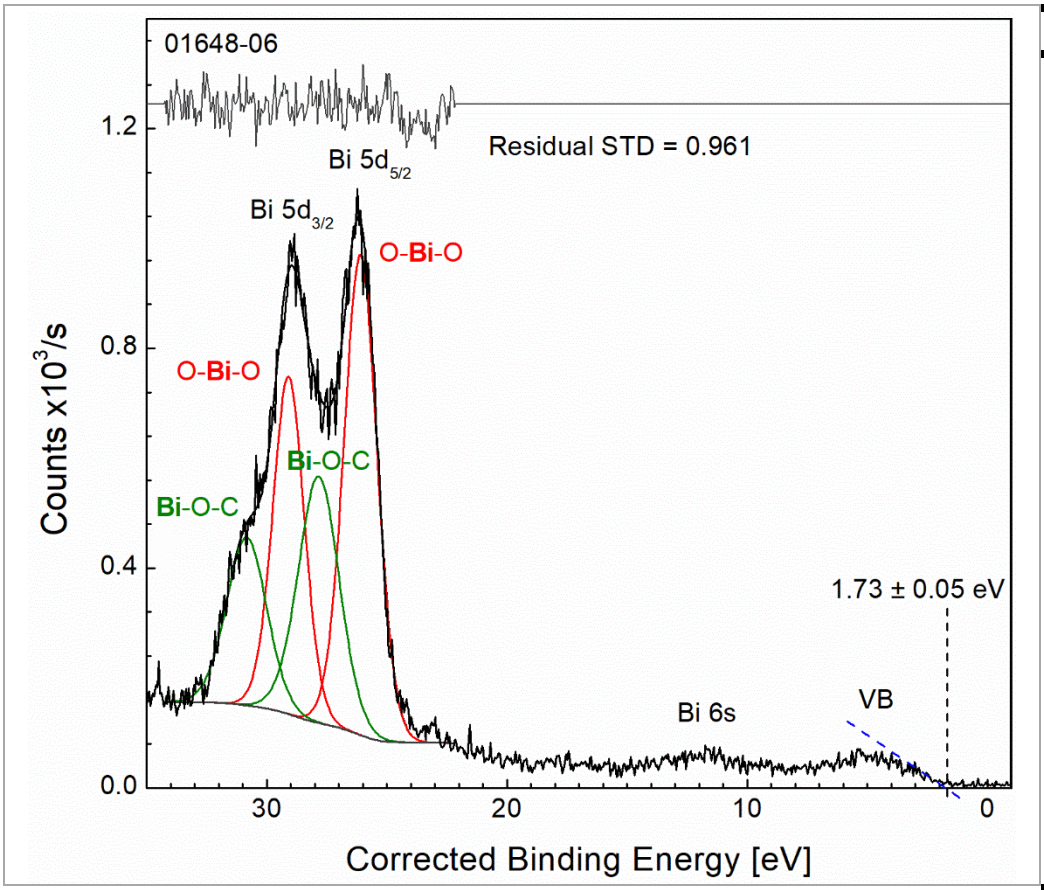
Publish in *SSS*: Yes ☒ No ☐

- **Accession #:** 01648-05
- **Host Material:** Bismuth acetate
- **Technique:** XPS
- **Spectral Region:** Bi 4d

Instrument: SPECS PHOIBOS 150
Excitation Source: Al Ka monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 2 mm x 2 mm
Analyzer Type: spherical sector
Incident Angle: 55 ˚
Emission Angle: 0 ˚
Analyzer Pass Energy 30 eV
Analyzer Resolution: 0.5 eV
Total Signal Accumulation Time: 1361 s
Total Elapsed Time: 2160 s

Number of Scans: 4

Effective Detector Width: 2.64 eV



Publish in *SSS*: Yes ☒ No ☐

- **Accession #:** 01648-06
- **Host Material:** Bismuth acetate
- **Technique:** XPS
- **Spectral Region:** Bi 5d, Bi 6s, VB

Instrument: SPECS PHOIBOS 150

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 200 W

Source Size: 2 mm x 2 mm

Analyzer Type: spherical sector

Incident Angle: 55 ˚

Emission Angle: 0 ˚

Analyzer Pass Energy 30 eV

Analyzer Resolution: 0.5 eV

Total Signal Accumulation Time: 761 s

Total Elapsed Time: 1207 s

Number of Scans: 4

Effective Detector Width: 2.64 eV