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Niobium ethoxide analyzed by XPS

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Niobium (V) ethoxide was characterized by 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 double-sided adhesive tape. Survey spectra, Nb 3d, O 1s, C 1s, Nb 3p, Nb 4p, O 2s core levels and valence band spectra were acquired. Results showed how the elements in niobium ethoxide structure are bonded.

Keywords: Niobium ethoxide; XPS; precursor; niobium ethanolate

Accession#: 01676

Technique: XPS

Host Material: Niobium ethoxide

Instrument: SPECS PHOIBOS 150

Major Elements in Spectra: Nb, O, C

Minor Elements in Spectra: None

Published Spectra: 6

Spectra in Electronic Record: 6

Spectral Category: comparison

INTRODUCTION

Niobium (V) ethoxide (Fig. 1) is a colorless to yellow organometallic compound, easily hydrolyzable and soluble in some organic solvents such as ethanol, it has a relative density of 1.268 g/cm^3 at 25 °C. It is widely used as a precursor for different processes, such as the generation of niobium oxide films by CVD (Refs 1 - 3), for applications in devices in the electronic and optical industry. Another important method that uses niobium ethoxide as a precursor to generate NbO or $LiNbO_3$ films is the sol-gel Refs. 3 - 5). These niobium oxides have a very high dielectric constant in thin films, being used by this property for memories (DRAMs) (Ref. 6). Furthermore, it is also used to produce Nb_2O_5, which is widely used for photocatalytic processes (Ref. 7).

C

Fig. 1. Structure of niobium ethoxide

XPS survey spectrum indicates that there are no intrusive elements in the sample, since the presence of just carbon, oxygen and niobium is evidenced.

Nb 3d high resolution spectrum was fitted with four contributions corresponding to two different chemical species. The peaks centered at 207.2 and 209.9 eV were assigned to niobium bonded with oxygen, O-**Nb**-O, and signals at 208.4 and 211.1 eV were associated with niobium bonded with oxygen related with carbon, **Nb**-O-C. High resolution spectrum of O 1s shows three chemical environments. **O**-Nb-**O** at 530.5 eV, oxygen bonded with bismuth. C-**O** at 532.2 eV, oxygen single bonded with carbon. And, C-**O**-Nb at 533.7 eV, oxygen bonded with carbon and niobium. C 1s spectrum displays three chemical species. First, carbon carbon bonding or adventitious carbon, **C**-(C,H) at 284.8 eV used as a reference (Ref. 8). Second, carbon single bounded with oxygen, **C**-O at 286.5 eV. And, carbon sinlge bonded with oxygen related with niobium, **C**-O-Nb at 288.8 eV.

SPECIMEN DESCRIPTION (ACCESSION # 01676)

Host Material: Niobium ethoxide

CAS Registry #: 3236-82-6

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

Chemical Name: Niobium ethoxide

Source: Sigma Aldrich

Host Composition: Niobium (V) ethoxide (99.95%)

Form: Powder

Structure: $Nb(OCH_2CH_3)_5$

History & Significance: Niobium (V) ethoxide powder was ground and fixed to a sample holder with copper 3M^{TM} 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.

In Situ Preparation: None

a)Electronic mail: rospinao@uis.edu.co

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: 60 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: 2.76 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
01676-02	Nb 3d	…	…	1.35×10^4	8.21	17.72	…
01676-02	Nb $3d_{5/2}$	207.2	1.37	…	…	…	O-**Nb**-O
01676-02	Nb $3d_{3/2}$	209.9	1.37	…	…	…	O-**Nb**-O
01676-02	Nb $3d_{5/2}$	208.4	1.78	…	…	…	**Nb**-O-C
01676-02	Nb $3d_{3/2}$	211.1	1.78	…	…	…	**Nb**-O-C
01676-03	O 1s	…	…	1.03×10^4	2.77	44.79	…
01676-03	O 1s	530.5	1.62	…	…	…	**O**-Nb-**O**
01676-03	O 1s	532.2	1.62	…	…	…	C-**O**
01676-03	O 1s	533.7	1.62	…	…	…	C-**O**-Nb
01676-04	C 1s	…	…	3.42×10^3	1.00	37.49	…
01676-04	C 1s	284.8	1.79	…	…	…	**C**-(C,H)
01676-04	C 1s	286.5	1.79	…	…	…	**C**-O
01676-04	C 1s	288.8	1.79	…	…	…	**C**-O-Nb
01676-05	Nb $3p_{3/2}$	365.9	2.71	6.42×10^3	…	…	…
01676-05	Nb $3p_{1/2}$	381.1	2.83	4.35×10^3	…	…	…
01676-06	Nb 4p	34.9	3.58	1.73×10^3	…	…	…
01676-06	O 2s	22.7	2.52	5.74×10^2	…	…	…
01676-06[a]	VBM	2.35	…	…	…	…	…

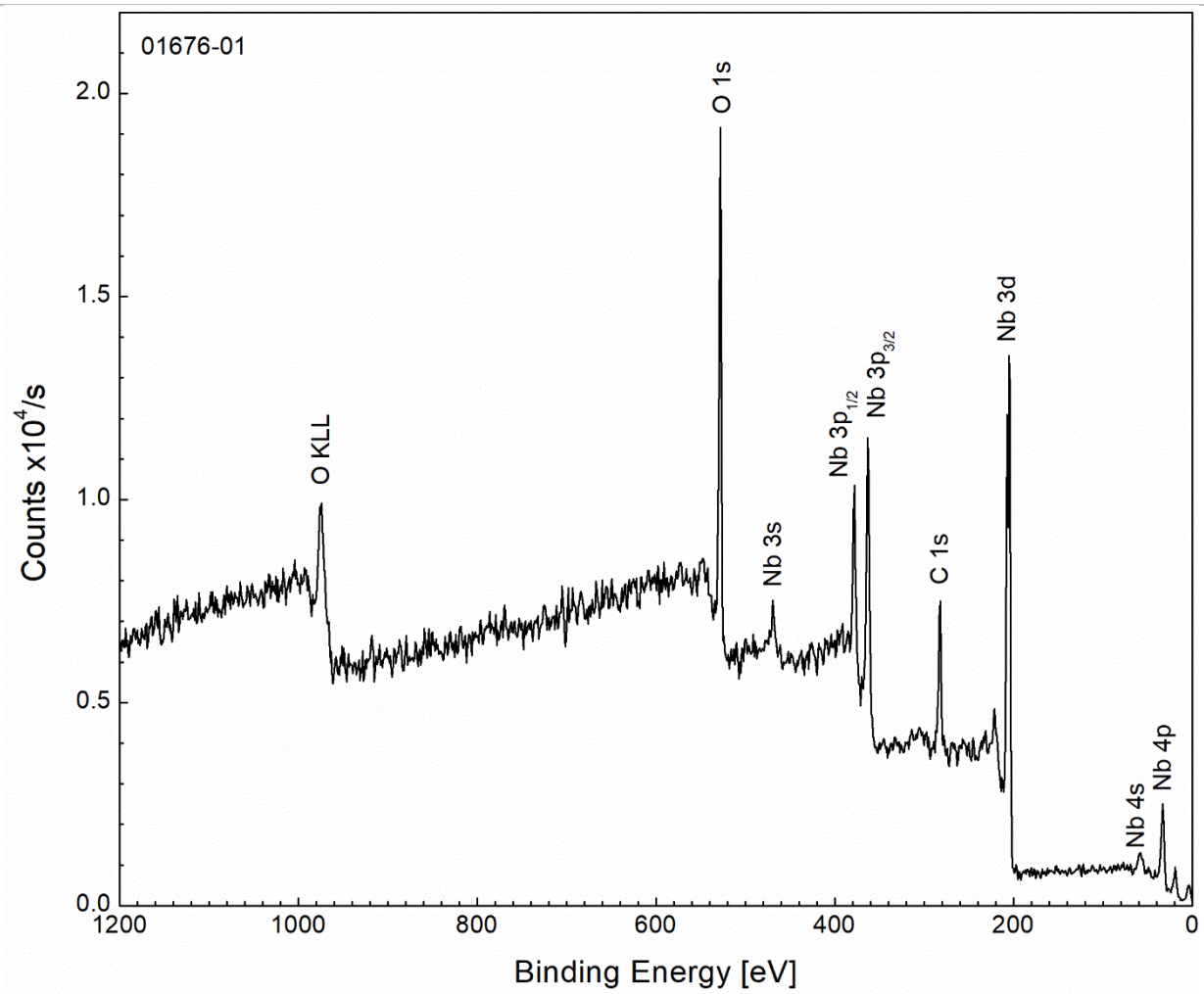
[a] Valence band maximum (VBM)

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	...	...	...

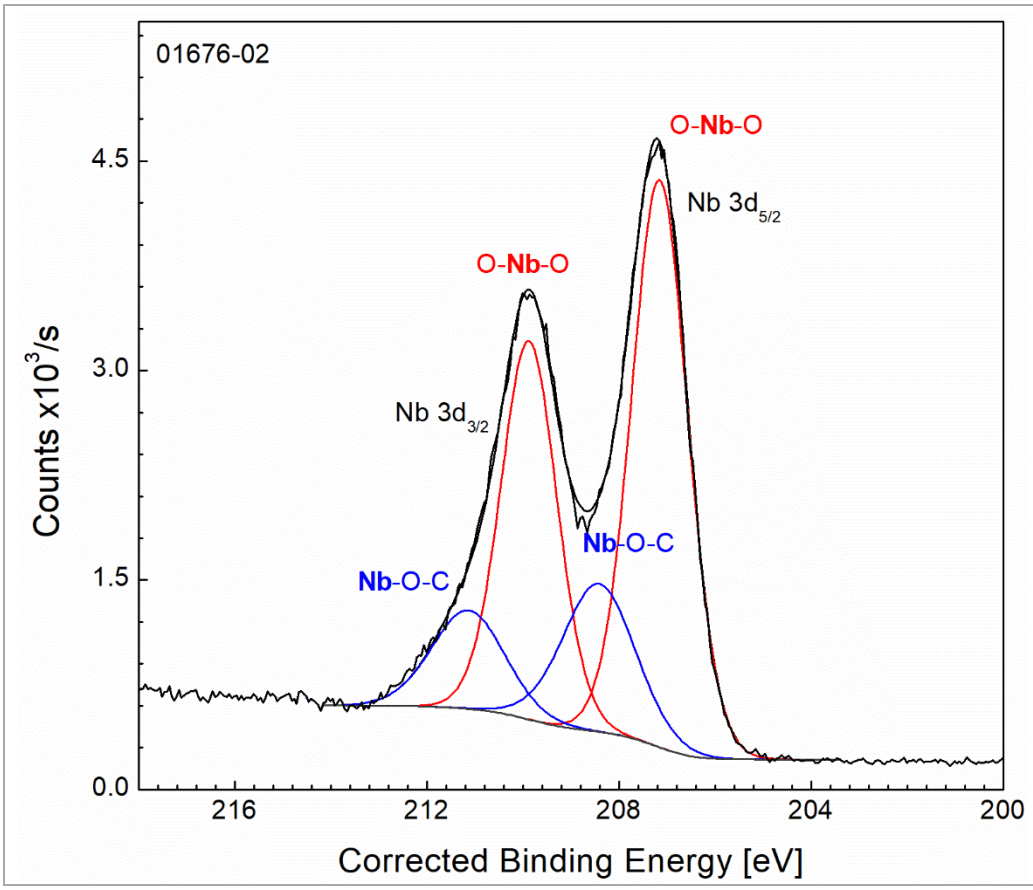
GUIDE TO FIGURES					
Spectrum (Accession) #	**Spectral Region**	**Voltage Shift***	**Multiplier**	**Baseline**	**Comment #**
01676-01	Survey	0	1	0	1
01676-02	Nb 3d	-2.76	1	0	1
01676-03	O 1s	-2.76	1	0	1
01676-04	C 1s	-2.76	1	0	1
01676-05	Nb 3p	-2.76	1	0	1
01676-06	Nb 4p, O 2s, VB	-2.76	1	0	1

*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 ☐

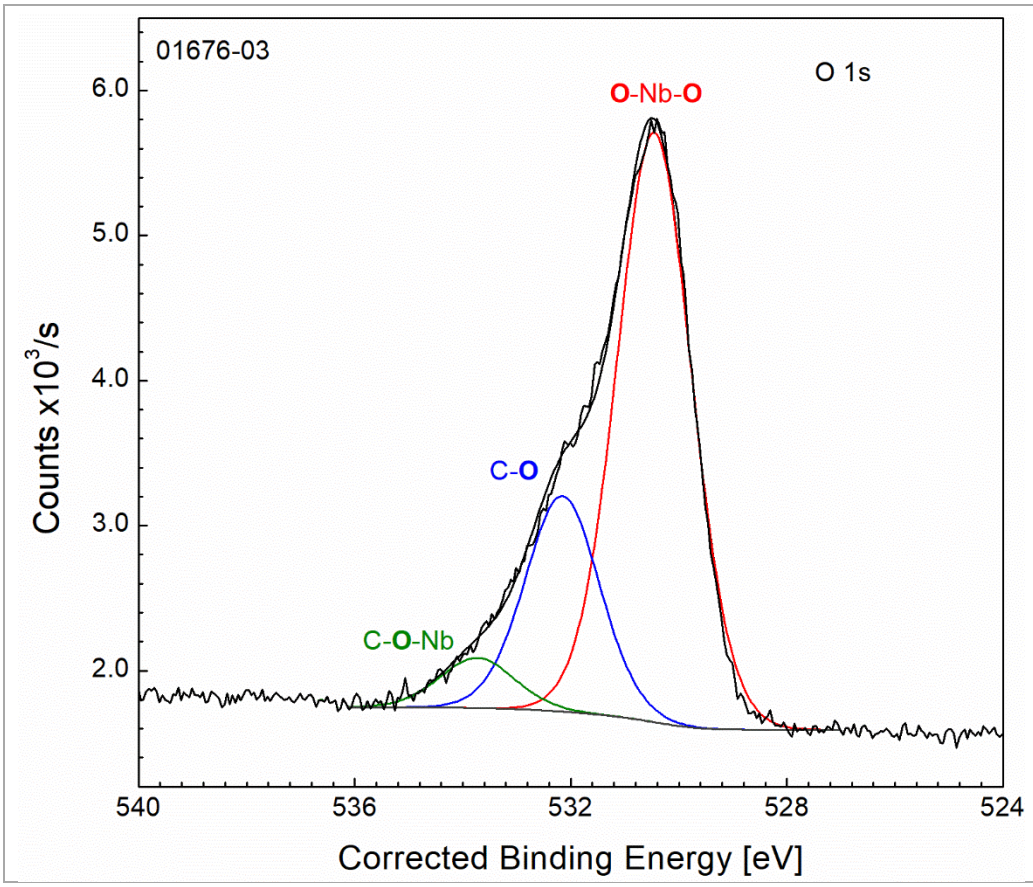
Accession #	01676-01
Host Material	Niobium ethoxide
Technique	XPS
Spectral Region	survey
Instrument	SPECS PHOIBOS 150
Excitation Source	Al Ka monochromatic
Source Energy	1486.6 eV
Source Strength	200 W
Source Size	2 mm x 2 mm
Analyzer Type	spherical sector analyzer
Incident Angle	55˚
Emission Angle	0˚
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 #:** 01676-02
- **Host Material:** Niobium ethoxide
- **Technique:** XPS
- **Spectral Region:** Nb 3d

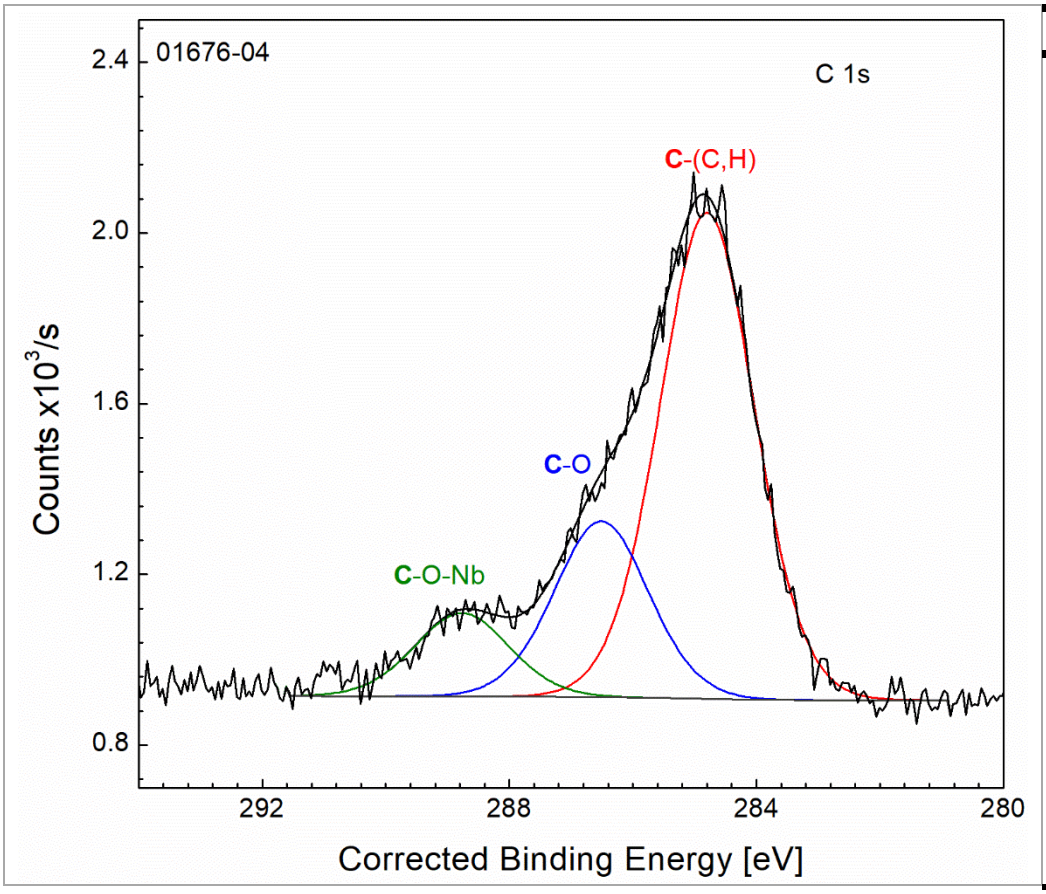
Instrument: SPECS PHOIBOS 150
Excitation Source: Al Ka monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 2 mm x 2 mm
Analyzer Type: spherical sector
Incident Angle: 55 ˚
Emission Angle: 0 ˚
Analyzer Pass Energy 30 eV
Analyzer Resolution: 0.5 eV
Total Signal Accumulation Time: 801 s
Total Elapsed Time: 1271 s
Number of Scans: 8
Effective Detector Width: 2.64 eV



Publish in *SSS*: Yes ☒ No ☐

- **Accession #:** 01676-03
- **Host Material:** Niobium ethoxide
- **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



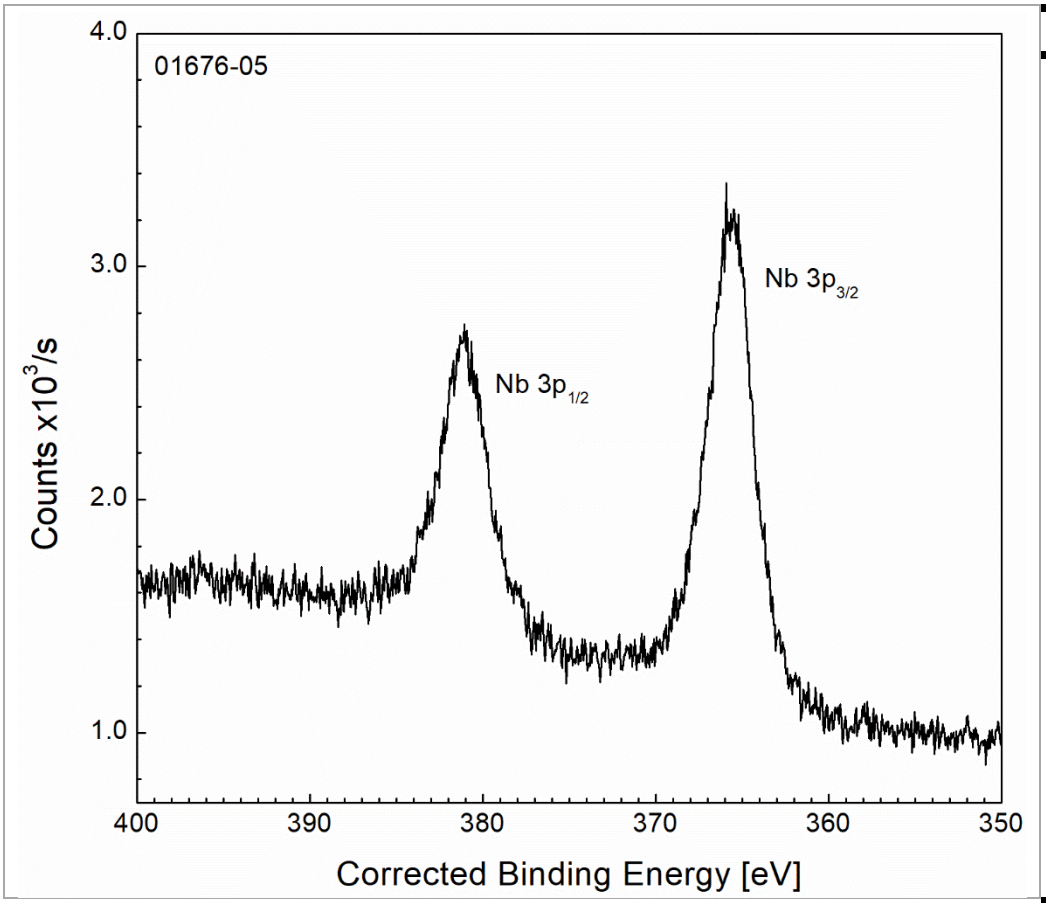
Publish in *SSS*: Yes ☒ No ☐

- **Accession #:** 01676-04
- **Host Material:** Niobium ethoxide
- **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: 421 s
Total Elapsed Time: 668 s

Number of Scans: 8

Effective Detector Width: 2.64 eV



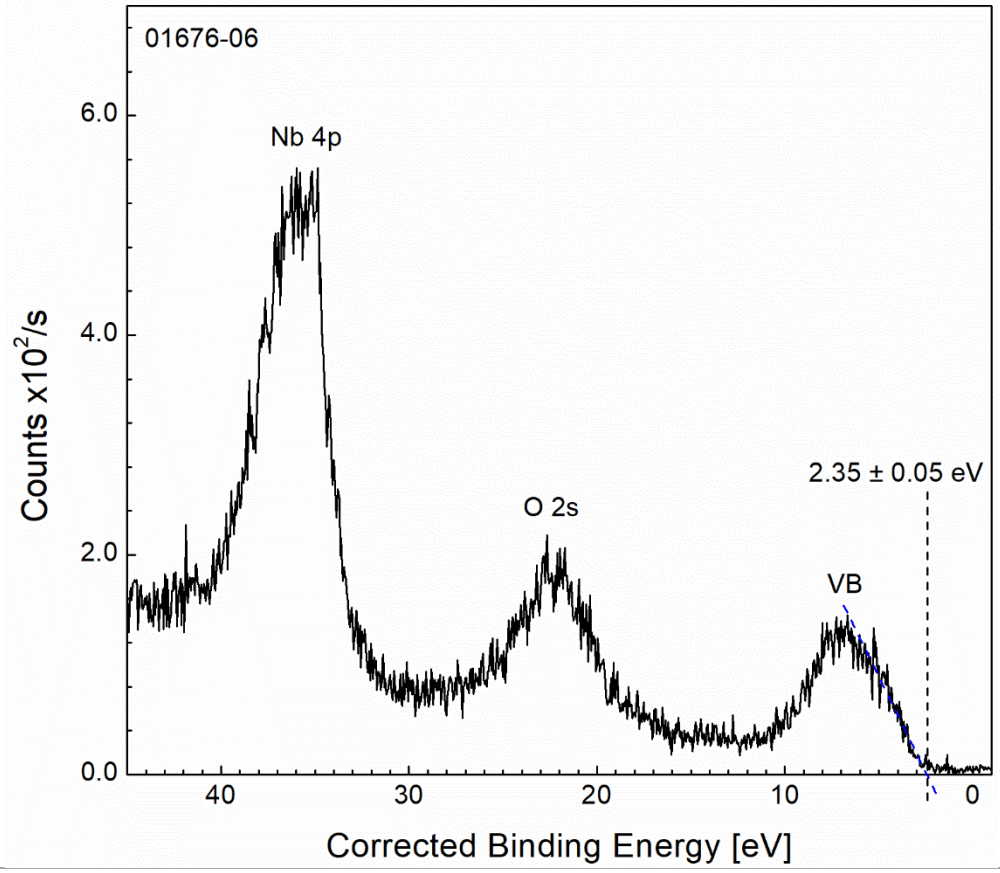
Publish in *SSS*: Yes ☒ No ☐

- **Accession #:** 01676-05
- **Host Material:** Niobium ethoxide
- **Technique:** XPS
- **Spectral Region:** Nb 3p

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: 1201 s
Total Elapsed Time: 1906 s

Number of Scans: 4

Effective Detector Width: 2.64 eV



Publish in *SSS*: Yes ☒ No ☐

- **Accession #:** 01676-06
- **Host Material:** Niobium ethoxide
- **Technique:** XPS
- **Spectral Region:** Nb 4p, O 2s, 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: 941 s

Total Elapsed Time: 1494 s

Number of Scans: 4

Effective Detector Width: 2.64 eV