Ibuprofen tablet characterized by XPS

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Ibuprofen tablet was characterized by x-ray photoelectron spectroscopy (XPS). Sample was fixed to a stainless-steel sample holder with copper double-sided adhesive tape. Survey spectra, C 1s, O 1s, N 1s, Si 2p and Na 1s core levels spectra were acquired. Results showed the presence of carbon and oxygen, elements that are part of the chemical structure of ibuprofen, however, these can also be part of the excipients. In addition, nitrogen, silicon and sodium were detected, which are associated with excipients.

Keywords: Ibuprofen; XPS; medicines; organic compounds; isobutylphenylpropionic acid

Accession#: 01687

Technique: XPS

Host Material: Ibuprofen tablet

Instrument: SPECS PHOIBOS 150

Major Elements in Spectra: C, O

Minor Elements in Spectra: Si, N, Na

Published Spectra: 6

Spectra in Electronic Record: 6

Spectral Category: comparison

INTRODUCTION

The 2- (4-isobutylphenyl) propanoic acid or ibuprofen (Fig. 1), as it is commonly known, is a drug in the nonsteroidal anti-inflammatory drug (NSAID), that is on the World Health Organization's List of Essential Medicines (Ref. 1). Its chemical formula is $C_{13}H_{18}O_2$ with a melting point of 77-78 ° C, soluble in ethanol and colorless or slightly yellow (Ref. 2). This is a very common over the counter drug that is widely used as a pain reliever, anti-inflammatory, and antipyretic (Ref. 3). Ibuprofen is marketed in different concentrations from 100mg to 800mg per tablet, this anti-inflammatory analgesic is used in complex treatments such as rheumatoid arthritis (Ref. 4), even cases of fever and chronic pain (Ref. 5). In this work we characterized the surface chemical state of an ibuprofen tablet using XPS.

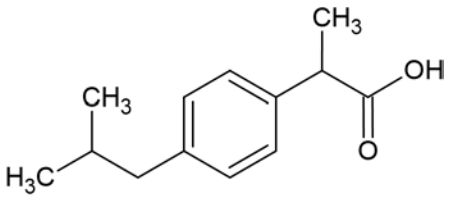

Fig 1. Chemical structure of Ibuprofen

SPECIMEN DESCRIPTION (ACCESSION # 01687)

Host Material: Ibuprofen tablet

CAS Registry #: 15687-27-1

Host Material Characteristics: homogeneous; solid; unknown crystallinity; unknown conductivity; organic compound; Other

Chemical Name: Ibuprofen

Source: Purchased from a pharmacy in Bucaramanga, Colombia, tablet from commercial brand of international distribution

Host Composition: Ibuprofen tablet

Form: Tablet

Structure: $C_{13}H_{18}O_2$

History & Significance: The tablet contains 500 mg of ibuprofen. The other components (excipients) are, sodium croscarmellose, hypromellose, polyvidone, lactose, microcrystalline cellulose, cornstarch, colloidal silicon dioxide, magnesium stearate, titanium dioxide, hypromellose, talc and propylene glycol. Ibuprofen tablet was ground and fixed to a sample holder with copper double sided adhesive tape. The sample was exposed to the environment for about 3 min, time that was spent to prepare the sample, and then introduce it to the platform.

As Received Condition: As tablet

Analyzed Region: same as host material

Ex Situ Preparation/Mounting: The preparation was carried out in a laboratory with controlled conditions, first the outer coating of the tablet was removed, then was gently grinded with an agate mortar and fixed to a sample holder with copper double sided adhesive tape, the conditions of humidity and temperature were 51% and 22 °C, respectively, and the tablet was exposed for about 3 min to the environment.

In Situ Preparation: None

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

Temp. During Analysis: 295 K

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

Pre-analysis Beam Exposure: 60 s

INSTRUMENT DESCRIPTION

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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: 100 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. 6) was used as reference to adjust the binding energy scale of the spectra.

Recommended Energy Scale Shift: 2.96 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

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SPECTRAL FEATURES TABLE

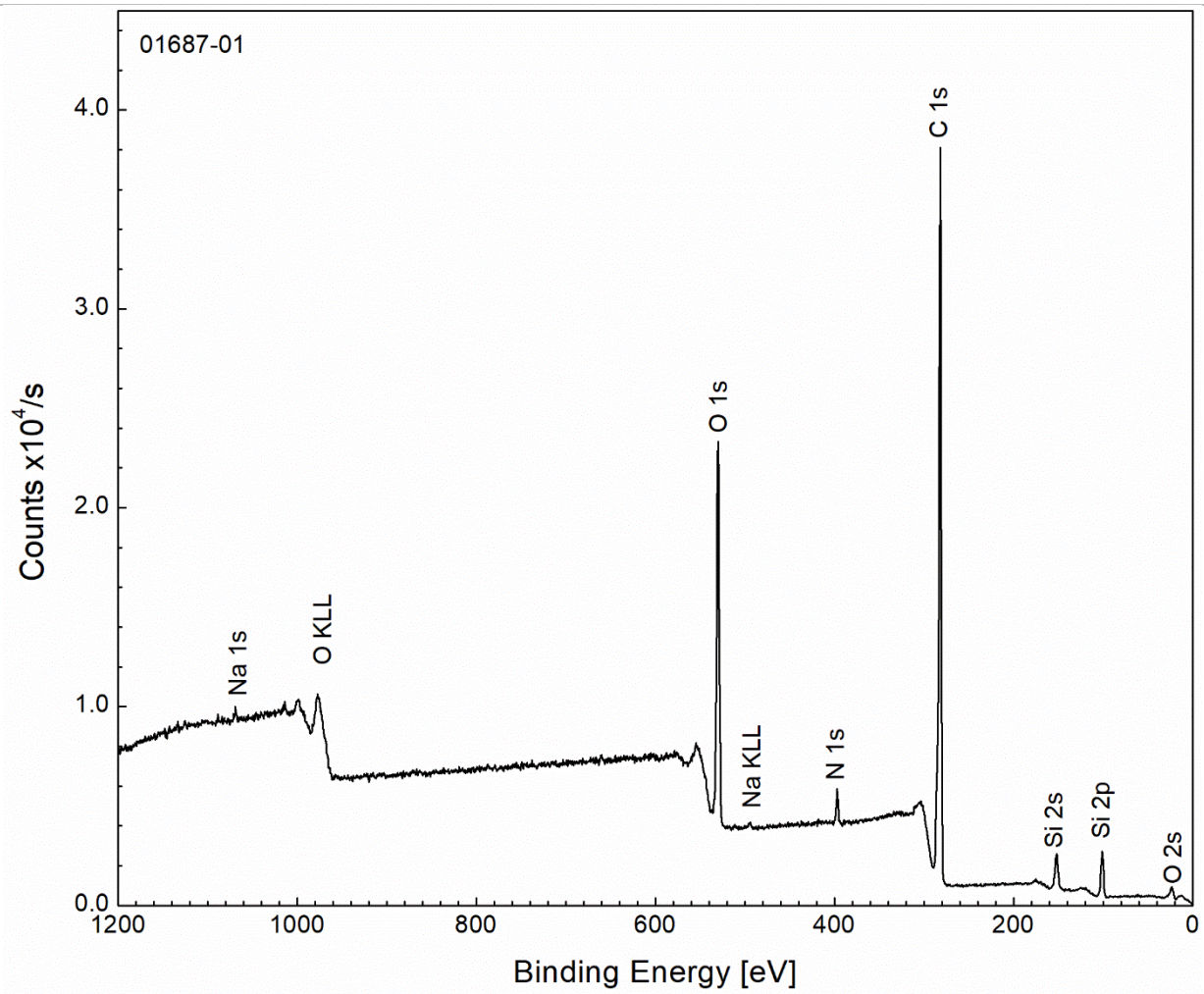
Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV x cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01687-02	C 1s	…	…	1.48×10^4	1.00	75.04	…
01687-02	C 1s	284.8	1.37	…	…	…	**C**-(C,H)
01687-02	C 1s	285.9	1.37	…	…	…	**C**-N
01687-02	C 1s	287.0	1.37	…	…	…	**C**-O
01687-02	C 1s	288.7	1.37	…	…	…	(**C**=O)-OH
01687-03	O 1s	…	…	1.06×10^4	2.77	19.48	…
01687-03	O 1s	532.0	1.88	…	…	…	Si-**O**, (C=**O**)-OH
01687-03	O 1s	532.7	1.88	…	…	…	C-**O**
01687-03	O 1s	533.2	1.88	…	…	…	(C=O)-**O**H
01687-03	O 1s	534.7	1.88	…	…	…	**O**H ads
01687-04	N 1s	…	….	5.71×10^2	1.76	1.79	…
01687-04	N 1s	399.6	1.49	…	…	…	**C**-N
01687-05	Si 2p	…	…	6.23×10^2	0.85	3.45	…
01687-05	Si 2p	103.5	2.00	…	…	…	Si-**O**
01687-06	Na 1s	…	…	2.12×10^2	6.59	0.24	
01687-06	Na 1s	1071.6	2.50	…	…	…	**Na^+**

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	...	...	...

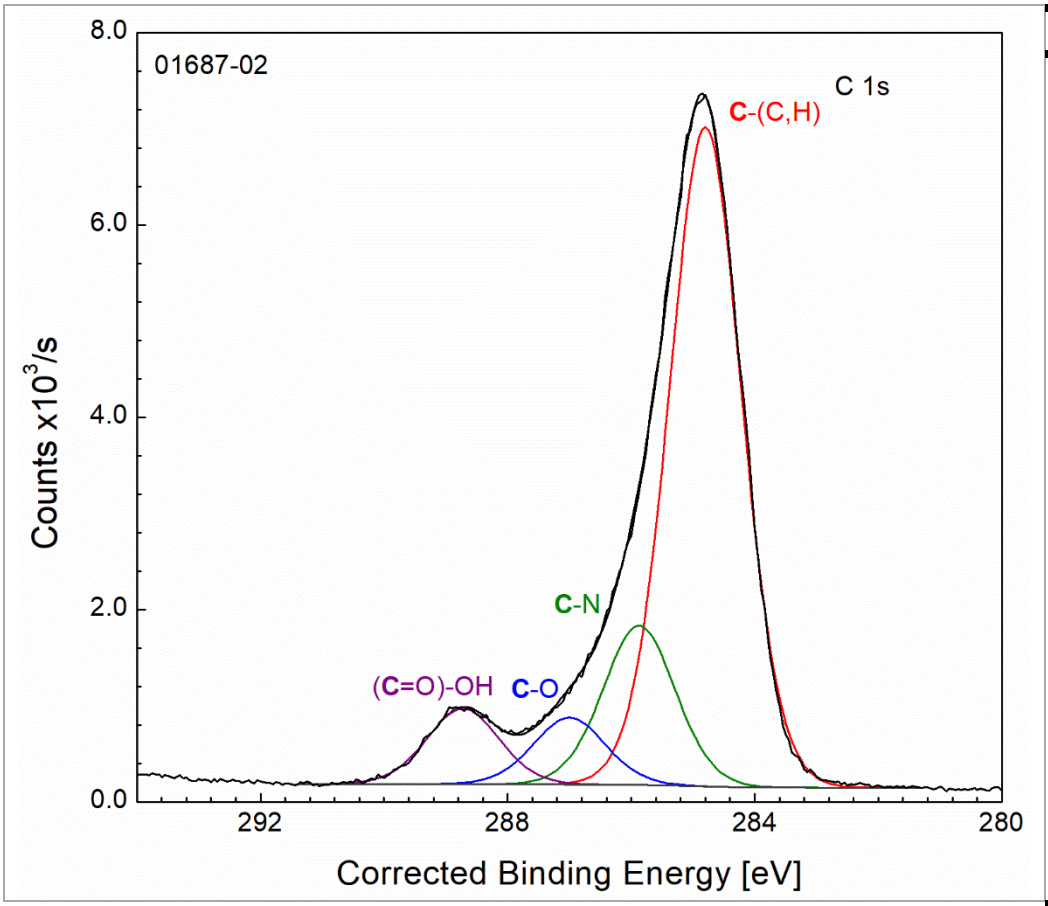
GUIDE TO FIGURES					
Spectrum (Accession) #	**Spectral Region**	**Voltage Shift***	**Multiplier**	**Baseline**	**Comment #**
01687-01	Survey	0	1	0	-
01687-02	C 1s	-2.96	1	0	-
01687-03	O 1s	-2.96	1	0	-
01687-04	N 1s	-2.96	1	0	-
01687-05	Si 2p	-2.96	1	0	-
01687-06	Na 1s	-2.96	1	0	-

*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.



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

Accession #	01687-01
Host Material	Ibuprofen tablet
Technique	XPS
Spectral Region	survey
Instrument	SPECS PHOIBOS 150
Excitation Source	Al Ka monochromatic
Source Energy	1486.6 eV
Source Strength	100 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	732 s
Total Elapsed Time	1560 s
Number of Scans	3
Effective Detector Width	5.28 eV



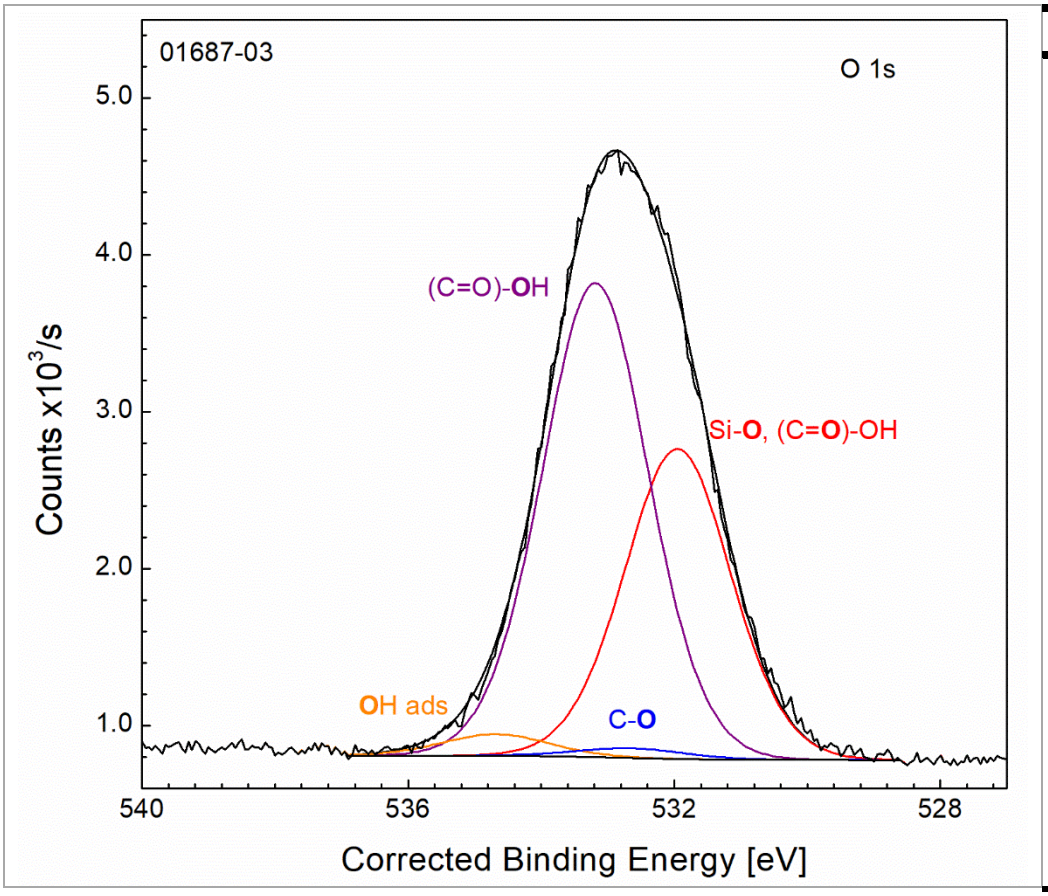
Publish in *SSS*: Yes ☒ No ☐

- **Accession #:** 01687-02
- **Host Material:** Ibuprofen tablet
- **Technique:** XPS
- **Spectral Region:** C 1s

Instrument: SPECS PHOIBOS 150
Excitation Source: Al Ka monochromatic
Source Energy: 1486.6 eV
Source Strength: 100 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: 10

Effective Detector Width: 2.64 eV



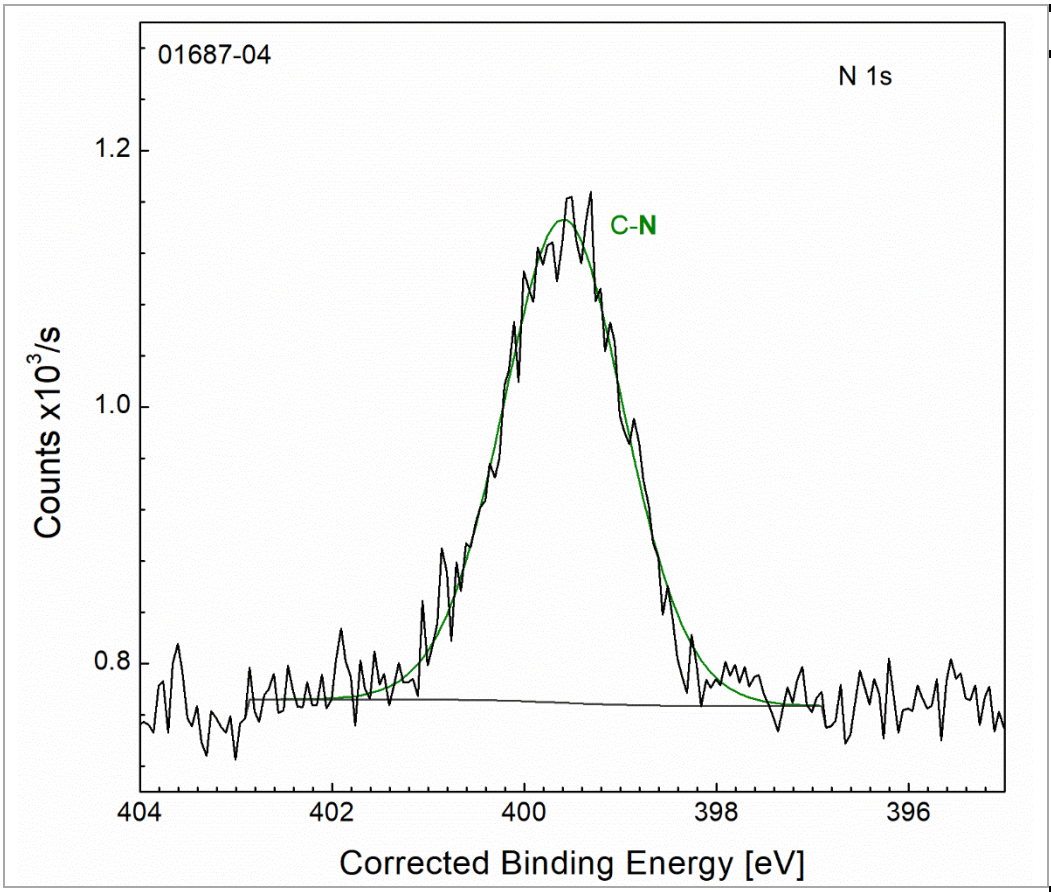
Publish in *SSS*: Yes ☒ No ☐

- **Accession #:** 01687-03
- **Host Material:** Ibuprofen tablet
- **Technique:** XPS
- **Spectral Region:** O 1s

Instrument: SPECS PHOIBOS 150
Excitation Source: Al Ka monochromatic
Source Energy: 1486.6 eV
Source Strength: 100 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: 341 s
Total Elapsed Time: 541 s

Number of Scans: 10

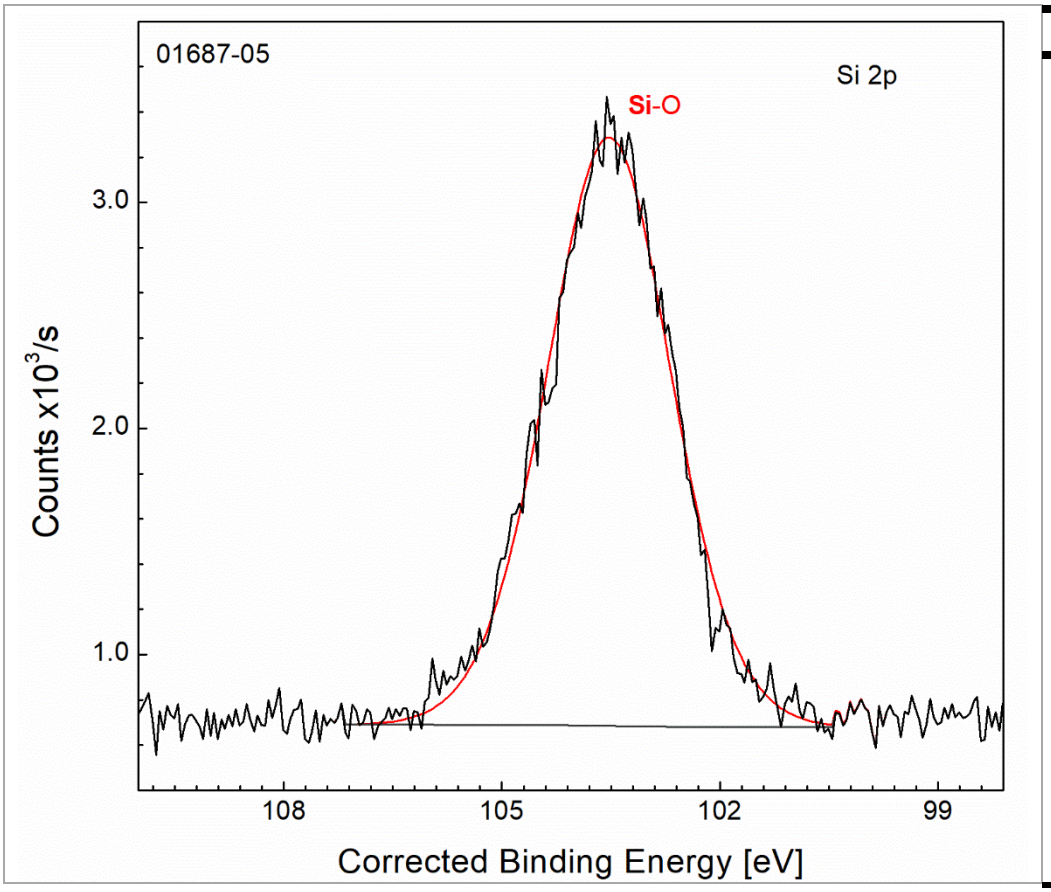
Effective Detector Width: 2.64 eV



Publish in *SSS*: Yes ☒ No ☐

- **Accession #:** 01687-04
- **Host Material:** Ibuprofen tablet
- **Technique:** XPS
- **Spectral Region:** N 1s

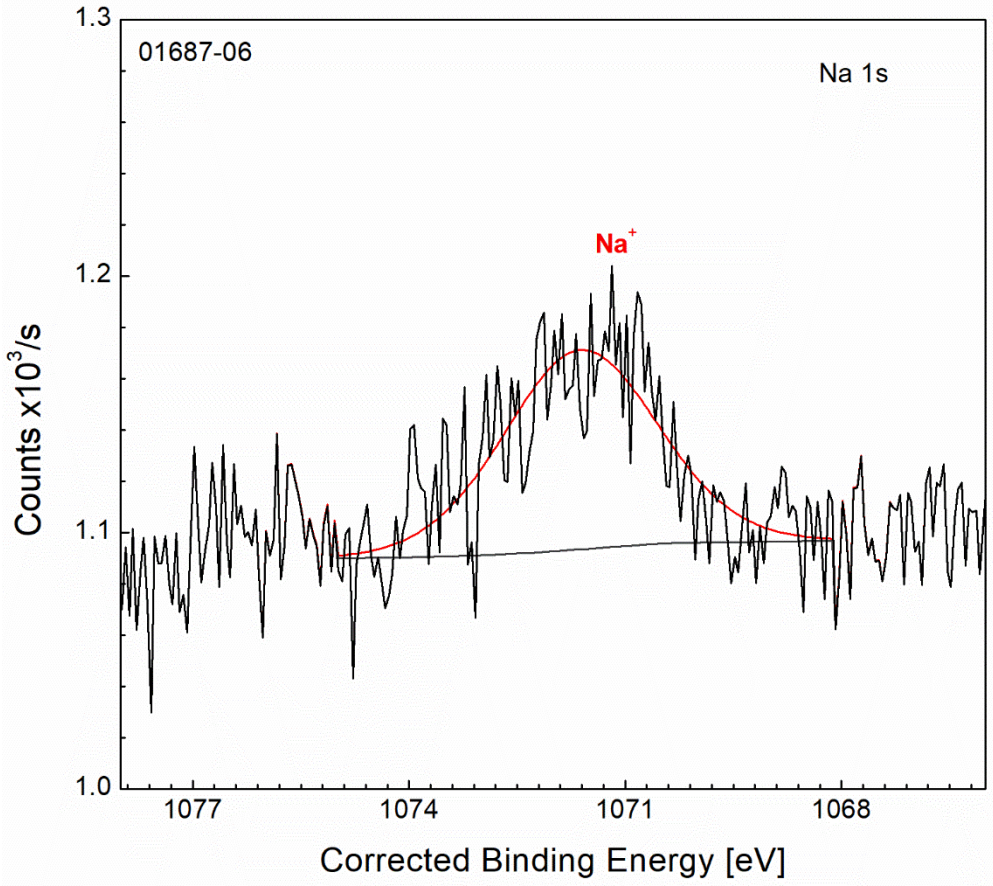
Instrument: SPECS PHOIBOS 150
Excitation Source: Al Ka monochromatic
Source Energy: 1486.6 eV
Source Strength: 100 W
Source Size: 2 mm x 2 mm
Analyzer Type: spherical sector
Incident Angle: 55 ˚
Emission Angle: 0 ˚
Analyzer Pass Energy 30 eV
Analyzer Resolution: 0.5 eV
Total Signal Accumulation Time: 261 s
Total Elapsed Time: 414 s
Number of Scans: 16
Effective Detector Width: 2.64 eV



Publish in *SSS*: Yes ☒ No ☐

- **Accession #:** 01687-05
- **Host Material:** Ibuprofen tablet
- **Technique:** XPS
- **Spectral Region:** Si 2p

Instrument: SPECS PHOIBOS 150
Excitation Source: Al Ka monochromatic
Source Energy: 1486.6 eV
Source Strength: 100 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: 281 s
Total Elapsed Time: 446 s
Number of Scans: 16
Effective Detector Width: 2.64 eV



Publish in *SSS*: Yes ☒ No ☐

- **Accession #:** 01687-06
- **Host Material:** Ibuprofen tablet
- **Technique:** XPS
- **Spectral Region:** Na 1s

Instrument: SPECS PHOIBOS 150

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 100 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: 361 s

Total Elapsed Time: 573 s

Number of Scans: 20

Effective Detector Width: 2.64 eV