Analysis of 7,13-Bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane by XPS

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Highly toxic metals abound in the environment as a result of pollution. Macrocyclic ligands have been designed that are selective in binding certain toxic metals. This allows for a sensitive means of detecting these poisonous metals. Here we report the XPS analysis of 7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane. © *2004 American Vacuum Society.* [DOI: 10.1116/11.20030301]

Keywords: *x-ray photoelectron spectroscopy; tetraazacrown ethers; heavy metals*

PACS: *82.80.Pv, 79.60.Fr*

Accession # 00771

Technique: XPS

Host Material: 7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane

Instrument: Surface Science Laboratories, Inc. 101

Major Elements in Spectrum: C, O, N, S

Minor Elements in Spectrum: none

Printed Spectra: 9

Spectra in Electronic Record: 14

Spectral Category: technical

INTRODUCTION

There is great need for monitoring heavy metal levels in the environment, as many of them have detrimental effects on human health and the environment. A number of methods have been developed for detecting toxic metals, the majority of which rely on batch-test analysis of samples removed from waste streams. This technique requires extensive equipment and training. A better approach would be to monitor heavy metals in waste streams continuously. This aim can be achieved with fluroionophores such as 7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane. Fluoroionophores are chelating agents that allow detection of metals through optical techniques.

SPECIMEN DESCRIPTION

Host Material: 7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane

Host Material Characteristics: homogeneous; unknown crystallinity; dielectric; organic compound; powder

Chemical Name: 7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane

Source: compound synthesized in Dr. Paul Savage's laboratory, Brigham Young University, Provo, UT

Host Composition: $C_{32}H_{43}N_6O_2S$

Form: powder

Structure: $C_{32}H_{43}N_6O_2S$

History & Significance: This compound provides a sensitive and selective method for continuously monitoring heavy metal levels in waste streams.

As Received Condition: powder

Analyzed Region: same as host material

***Ex Situ* Preparation/Mounting:** A silicon surface (Si/SiO_2) was first cleansed with a solution of NH_4OH (conc.), H_2O_2 (conc.) (50:50) (v/v) for 30 min at room temperature. It was then rinsed with water and finally washed with 5% vol. HCl (conc.) for 1 h. Double-sided sticky tape was pressed into the organic sample and attached to the surface of clean silicon.

***In Situ* Preparation:** none

Charge Control: target bias 3 eV, flood gun voltage 2.5 eV, no metal screens were used

Temp. During Analysis: 298 K

Pressure During Analysis: $<1.97\times10^{-7}$ Pa

INSTRUMENT DESCRIPTION

Manufacturer and Model: Surface Science Laboratories, Inc. 101

Analyzer Type: spherical sector

Detector: resistive anode position detector

Number of Detector Elements: 128

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

■ *Spectrometer*

Analyzer Mode: constant pass energy

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

Excitation Source Window: 12 μm Al

Excitation Source: Al K_α monochromatic

Source Energy: 1486.6 eV

Source Strength: 200 W

Source Beam Size: 0.8 mm × 0.8 mm

Analyzer Width at 84 eV: 1500 μm × 12000 μm

Signal Mode: multichannel direct

■ *Geometry*

Incident Angle: 55°

Source to Analyzer Angle: 70.8°

Emission Angle: 55°

Specimen Azimuthal Angle: 0°

Acceptance Angle from Analyzer Axis: 0°

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DATA ANALYSIS METHOD

Quantitation Method: Sensitivity factors were obtained from ESCA 2000 NT software supplied by Service Physics. The peak areas are the areas above a linear background.

REFERENCES

1. Z. Yang, J. S. Bradshaw, X. Y. Yang, P. B. Savage, K. E. Krakowiak, N. K. Dalley, N. Su, T. Bronson, and R. M. Izatt, J. Org. Chem. **64**, 3162 (1999).

SPECTRAL FEATURES TABLE

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (counts)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
00771-02	C 1*s*	284.8	4.3	199	1.00	60.6	···
00771-03	C 1*s*	284.5	4.1	455	1.00	61.2	···
00771-04	N 1*s*	398.8	5.7	33	1.68	10.1	···
00771-05	N 1*s*	398.2	4.1	72	1.68	9.7	···
00771-06	O 1*s*	5530.6	4.4	49	2.50	15.0	···
00771-07	O 1*s*	530.3	3.7	102	2.50	13.8	···
00771-08	S 2*p*	162.4	3.8	47	1.79	14.3	···
00771-09	S 2*p*	162.e	3.9	114	1.79	15.3	···

ANALYZER CALIBRATION TABLE

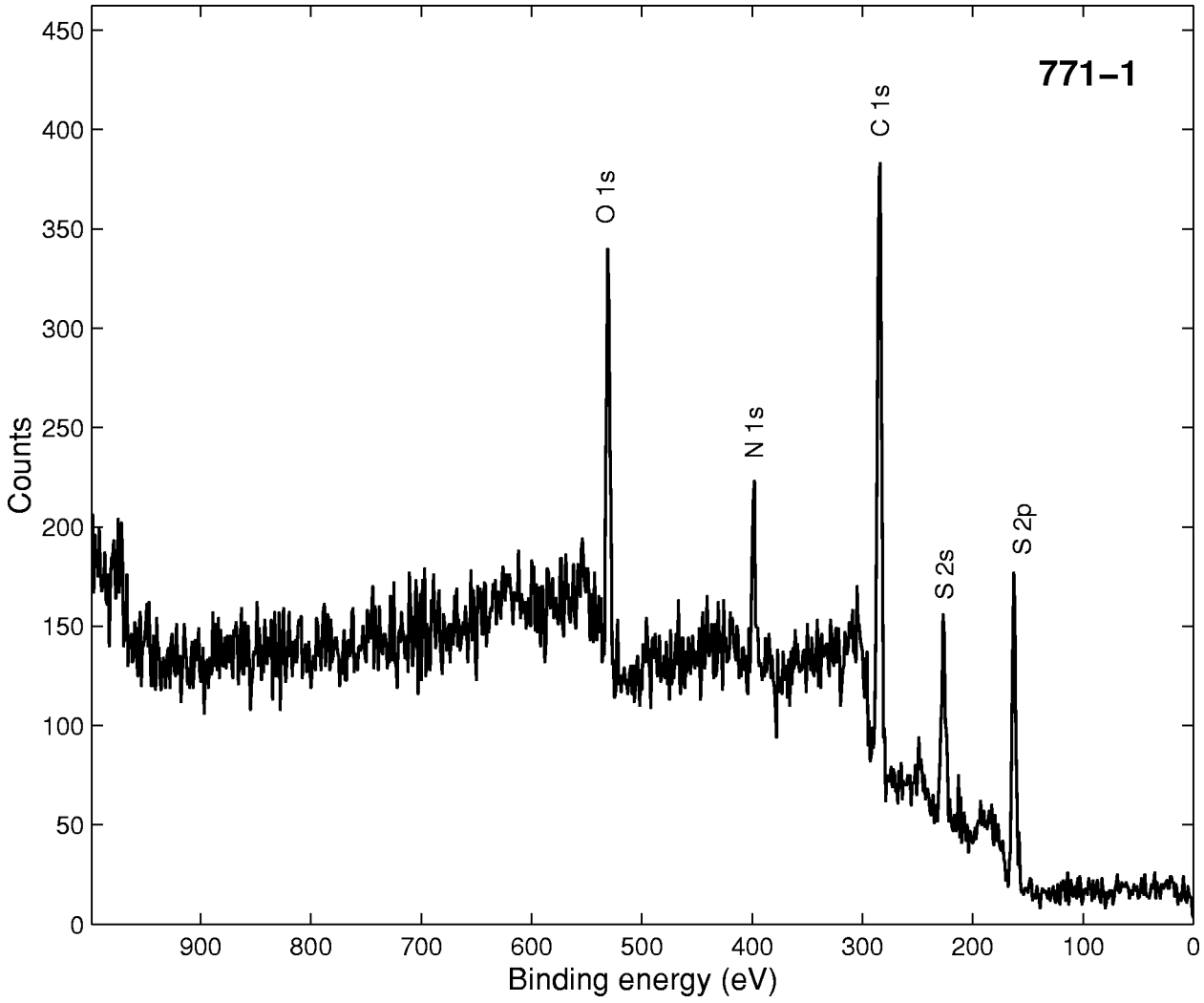
Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (counts)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
...[a]	Au $4f_{7/2}$	83.92	0.98	2200	10.67	···	···
...[b]	Au $4f_{7/2}$	83.92	1.6	6000	10.67	···	···
...[c]	Cu 3*s*	122.36	3.0	1600	1.05	···	···
...[b]	Cu $2p_{3/2}$	932.45	1.78	4000	9.73	···	···

[a] Spot size 300 μm, pass energy 50 eV, 2 scans.
[b] Spot size 800 μm, pass energy 150 eV, 1 scan.
[c] Spot size 800 μm, pass energy 150 eV, 3 scans.

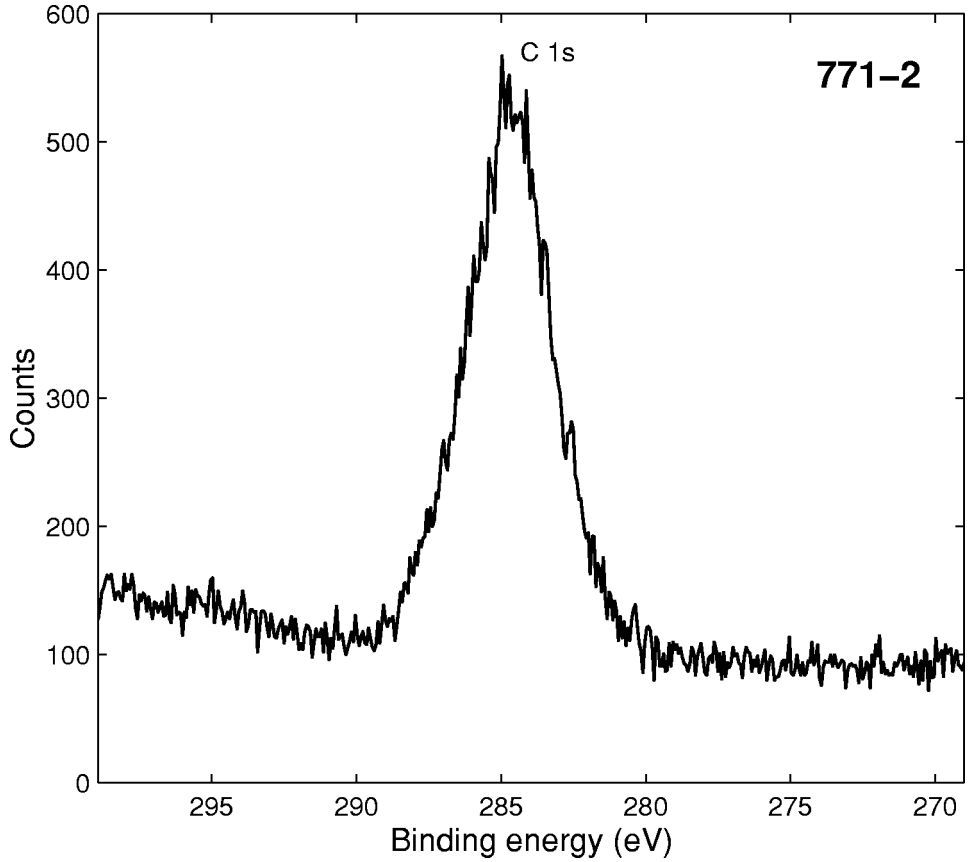
GUIDE TO FIGURES

Spectrum (Accession) #	Spectral Region	Voltage Shift*	Multiplier	Baseline	Comment #
771-1	Survey	0	1	0	1
771-2	C 1*s*	0	1	0	2
771-3	C 1*s*	0	1	0	1
771-4	N 1*s*	0	1	0	2
771-5	N 1*s*	0	1	0	1
771-6	O 1*s*	0	1	0	2
771-7	O 1*s*	0	1	0	1
771-8	S 2*p*	0	1	0	2
771-9	S 2*p*	0	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. Pass energy 150 eV
2. Pass energy 50 eV



Accession #	00771-01
Host Material	7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl 1,4,7,13-tetraaza-10-thiacyclopentadecane
Technique	XPS
Spectral Region	survey
Instrument	Surface Science Laboratories, Inc. 101
Excitation Source	Al K_α monochromatic
Source Energy	1486.6 eV
Source Strength	200 W
Source Size	0.8 mm × 0.8 mm
Analyzer Type	spherical sector
Incident Angle	55°
Emission Angle	55°
Analyzer Pass Energy	150 eV
Analyzer Resolution	1.5 eV
Total Signal Accumulation Time	220 s
Total Elapsed Time	420 s
Number of Scans	1
Effective Detector Width	15.1 eV



- **Accession #:** 00771-02
- **Host Material:** 7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane
- **Technique:** XPS
- **Spectral Region:** C 1*s*

Instrument: Surface Science Laboratories, Inc. 101

Excitation Source: Al K_α monochromatic

Source Energy: 1486.6 eV

Source Strength: 200 W

Source Size: 0.8 mm × 0.8 mm

Incident Angle: 55°

Analyzer Type: spherical sector

Analyzer Pass Energy: 50 eV

Analyzer Resolution: 0.5 eV

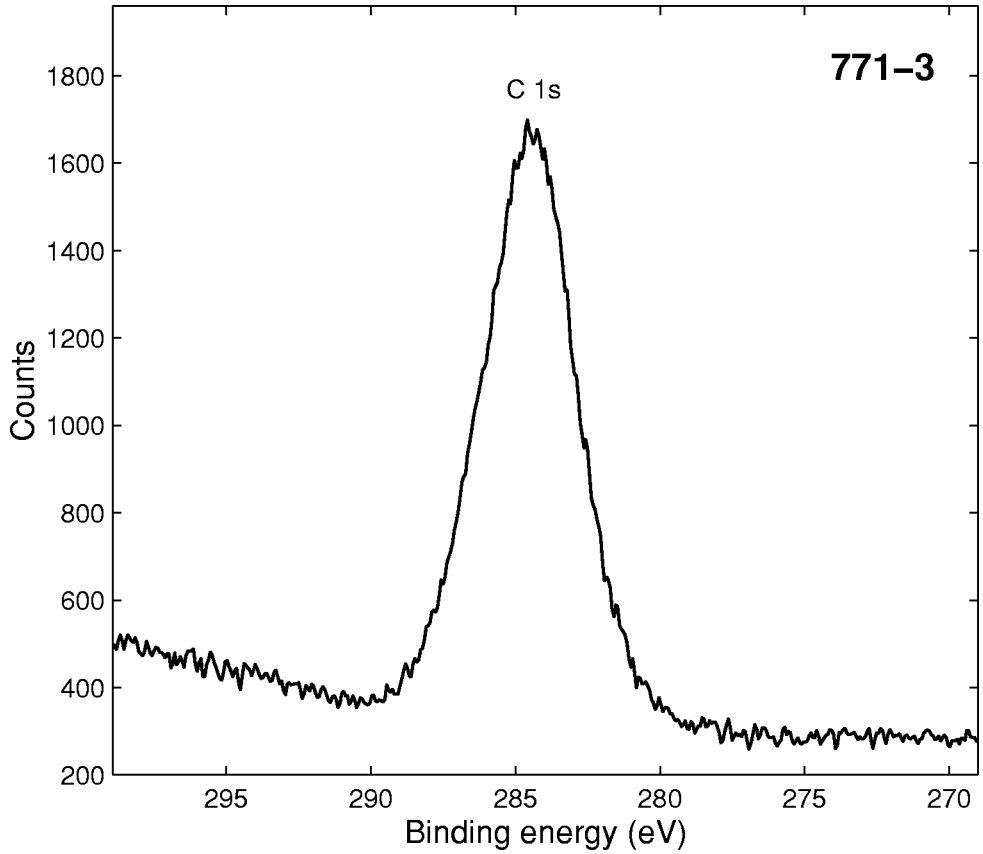
Emission Angle: 55°

Total Signal Accumulation Time: 552 s

Total Elapsed Time: 721 s

Number of Scans: 8

Effective Detector Width: 6.09 eV



- **Accession #:** 00771-03
- **Host Material:** 7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane
- **Technique:** XPS
- **Spectral Region:** C 1*s*

Instrument: Surface Science Laboratories, Inc. 101

Excitation Source: Al K_α monochromatic

Source Energy: 1486.6 eV

Source Strength: 200 W

Source Size: 0.8 mm × 0.8 mm

Incident Angle: 55°

Analyzer Type: spherical sector

Analyzer Pass Energy: 150 eV

Analyzer Resolution: 1.5 eV

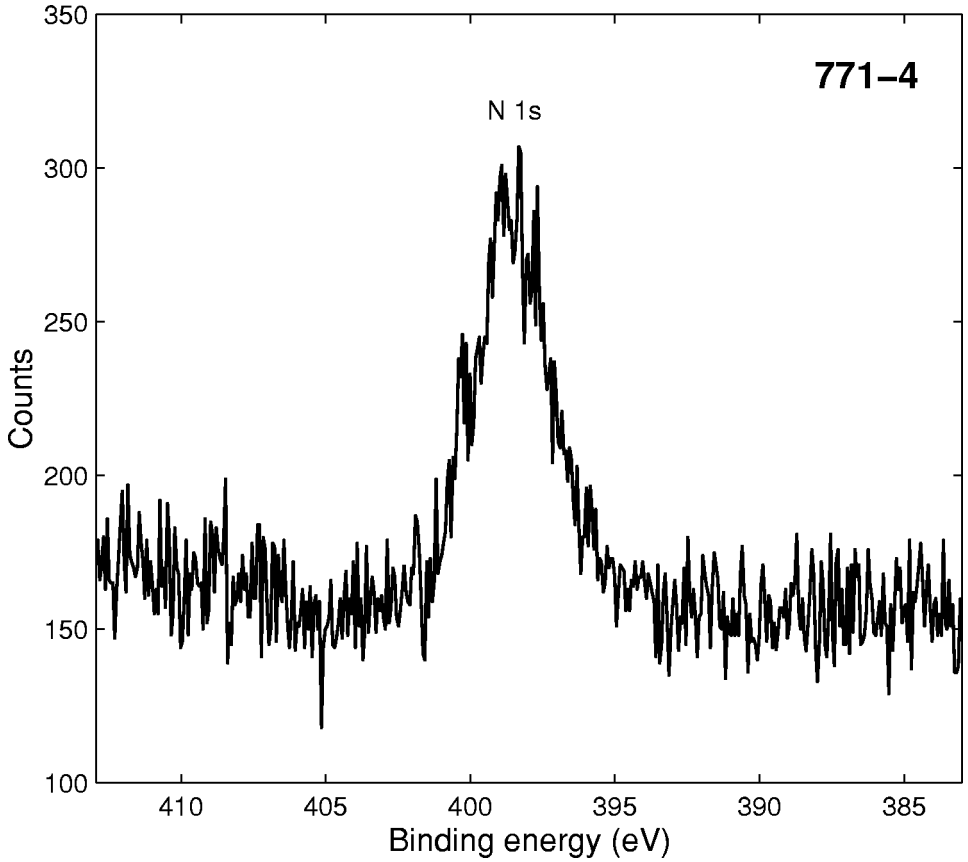
Emission Angle: 55°

Total Signal Accumulation Time: 276 s

Total Elapsed Time: 445 s

Number of Scans: 4

Effective Detector Width: 15.1 eV



- **Accession #:** 00771-04
- **Host Material:** 7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane
- **Technique:** XPS
- **Spectral Region:** N 1*s*

Instrument: Surface Science Laboratories, Inc. 101

Excitation Source: Al K_α monochromatic

Source Energy: 1486.6 eV

Source Strength: 200 W

Source Size: 0.8 mm × 0.8 mm

Incident Angle: 55°

Analyzer Type: spherical sector

Analyzer Pass Energy: 50 eV

Analyzer Resolution: 0.5 eV

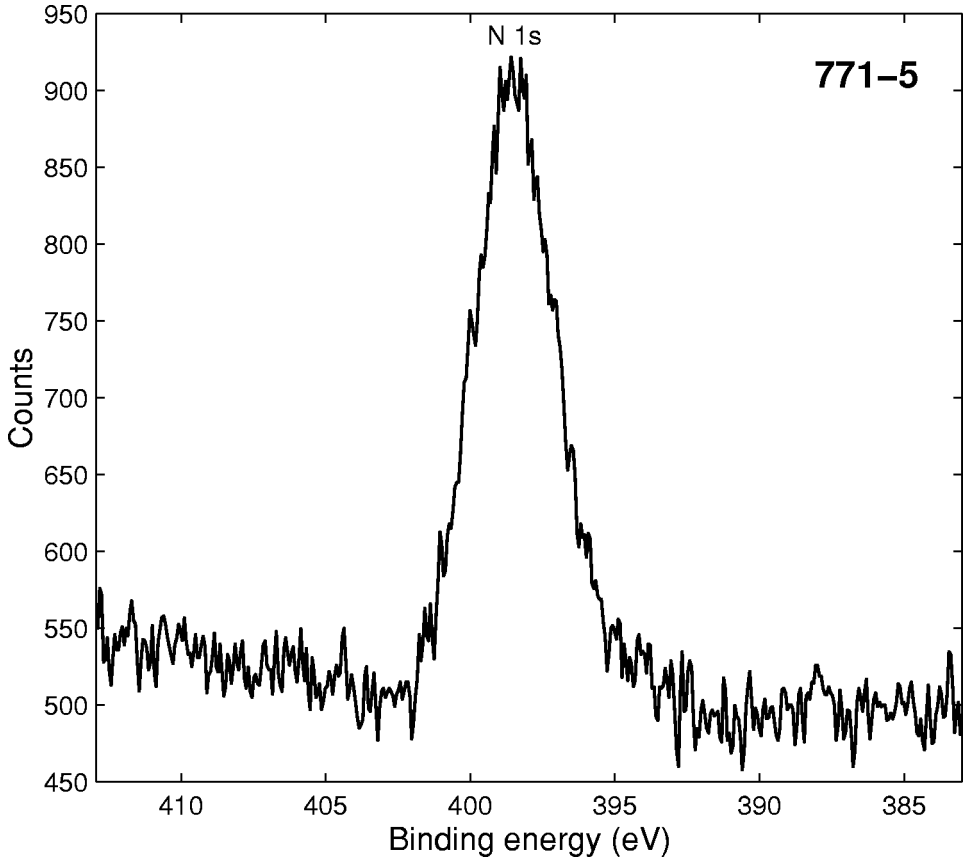
Emission Angle: 55°

Total Signal Accumulation Time: 552 s

Total Elapsed Time: 721 s

Number of Scans: 8

Effective Detector Width: 6.09 eV



- **Accession #:** 00771-05
- **Host Material:** 7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane
- **Technique:** XPS
- **Spectral Region:** N 1*s*

Instrument: Surface Science Laboratories, Inc. 101

Excitation Source: Al K_α monochromatic

Source Energy: 1486.6 eV

Source Strength: 200 W

Source Size: 0.8 mm × 0.8 mm

Incident Angle: 55°

Analyzer Type: spherical sector

Analyzer Pass Energy: 150 eV

Analyzer Resolution: 1.5 eV

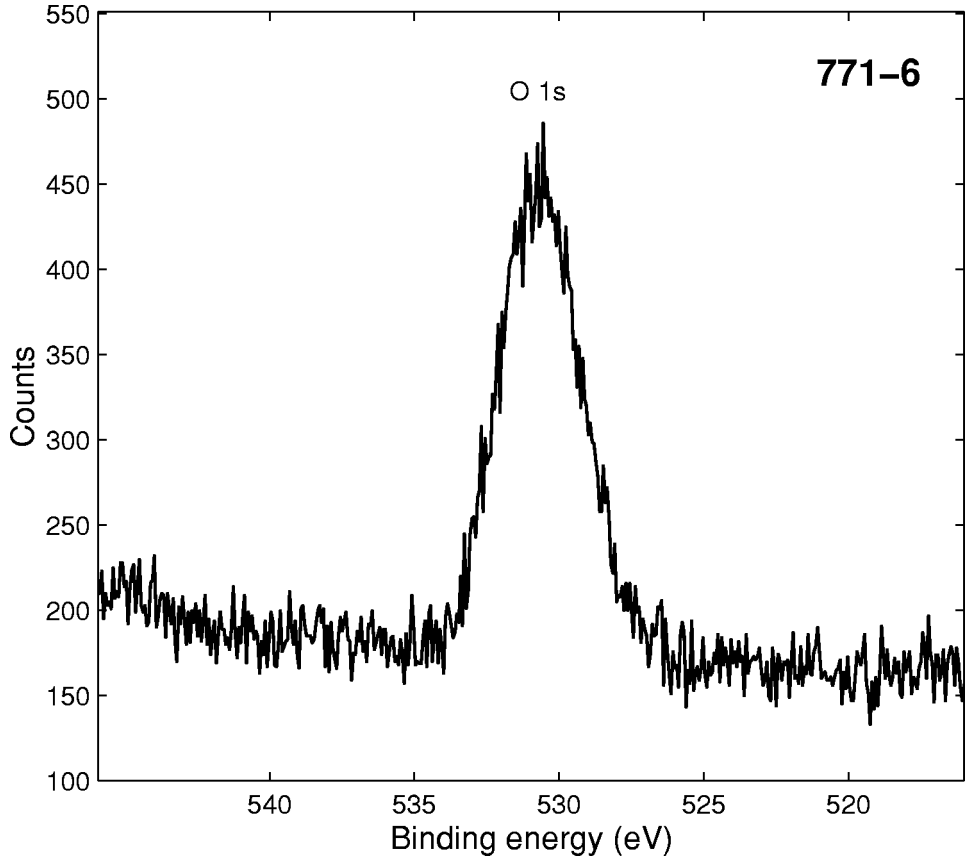
Emission Angle: 55°

Total Signal Accumulation Time: 296 s

Total Elapsed Time: 445 s

Number of Scans: 4

Effective Detector Width: 15.1 eV



- **Accession #:** 00771-06
- **Host Material:** 7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane
- **Technique:** XPS
- **Spectral Region:** O 1*s*

Instrument: Surface Science Laboratories, Inc. 101

Excitation Source: Al K_α monochromatic

Source Energy: 1486.6 eV

Source Strength: 200 W

Source Size: 0.8 mm × 0.8 mm

Incident Angle: 55°

Analyzer Type: spherical sector

Analyzer Pass Energy: 50 eV

Analyzer Resolution: 0.5 eV

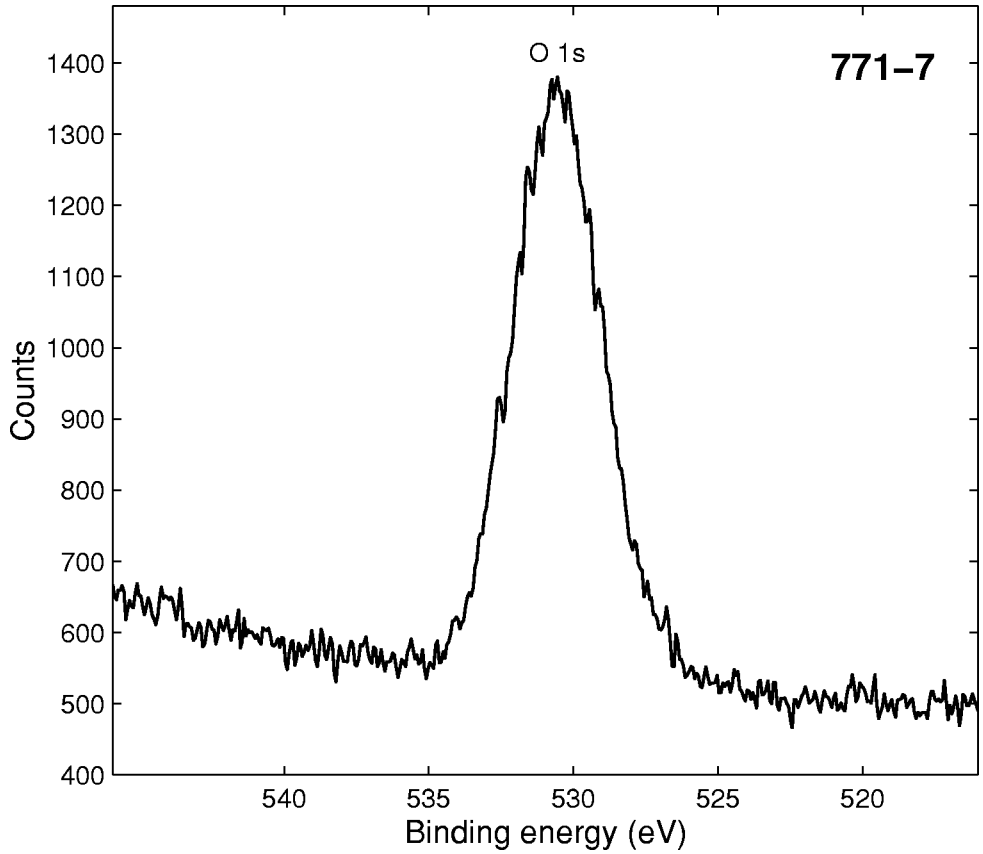
Emission Angle: 55°

Total Signal Accumulation Time: 552 s

Total Elapsed Time: 721 s

Number of Scans: 8

Effective Detector Width: 6.09 eV



- **Accession #:** 00771-07
- **Host Material:** 7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane
- **Technique:** XPS
- **Spectral Region:** O 1*s*

Instrument: Surface Science Laboratories, Inc. 101

Excitation Source: Al K_α monochromatic

Source Energy: 1486.6 eV

Source Strength: 200 W

Source Size: 0.8 mm × 0.8 mm

Incident Angle: 55°

Analyzer Type: spherical sector

Analyzer Pass Energy: 150 eV

Analyzer Resolution: 1.5 eV

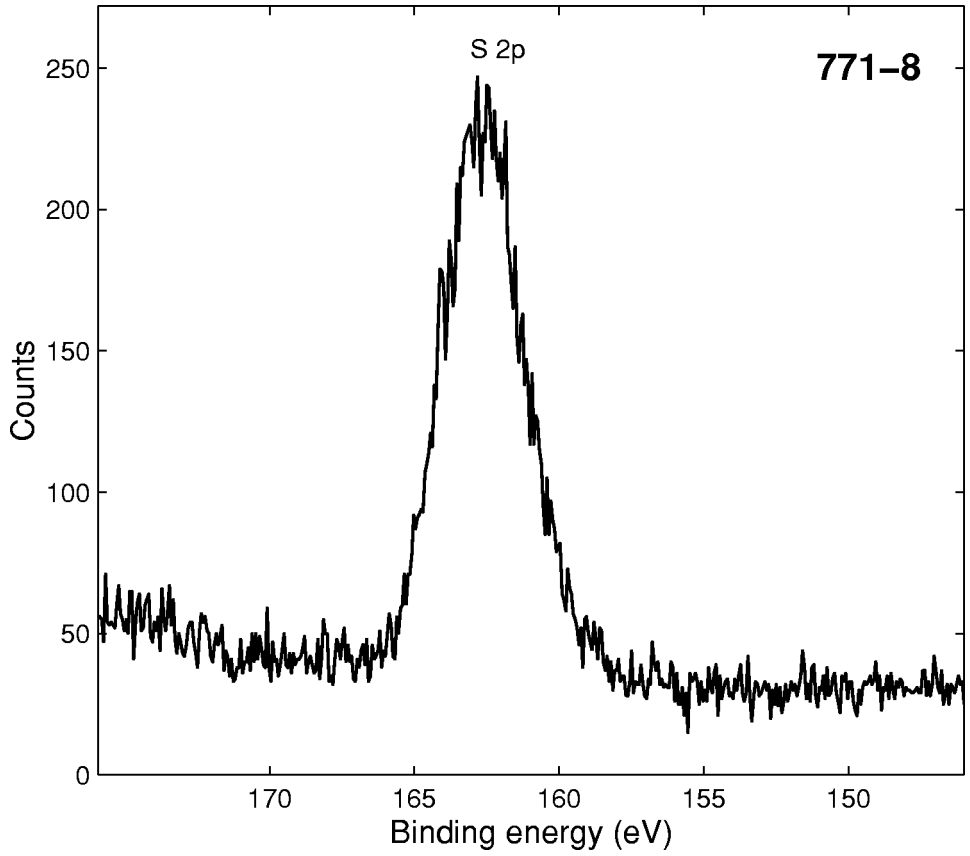
Emission Angle: 55°

Total Signal Accumulation Time: 296 s

Total Elapsed Time: 445 s

Number of Scans: 4

Effective Detector Width: 15.1 eV



- **Accession #:** 00771-08
- **Host Material:** 7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane
- **Technique:** XPS
- **Spectral Region:** S 2*p*

Instrument: Surface Science Laboratories, Inc. 101

Excitation Source: Al K_α monochromatic

Source Energy: 1486.6 eV

Source Strength: 200 W

Source Size: 0.8 mm × 0.8 mm

Incident Angle: 55°

Analyzer Type: spherical sector

Analyzer Pass Energy: 50 eV

Analyzer Resolution: 0.5 eV

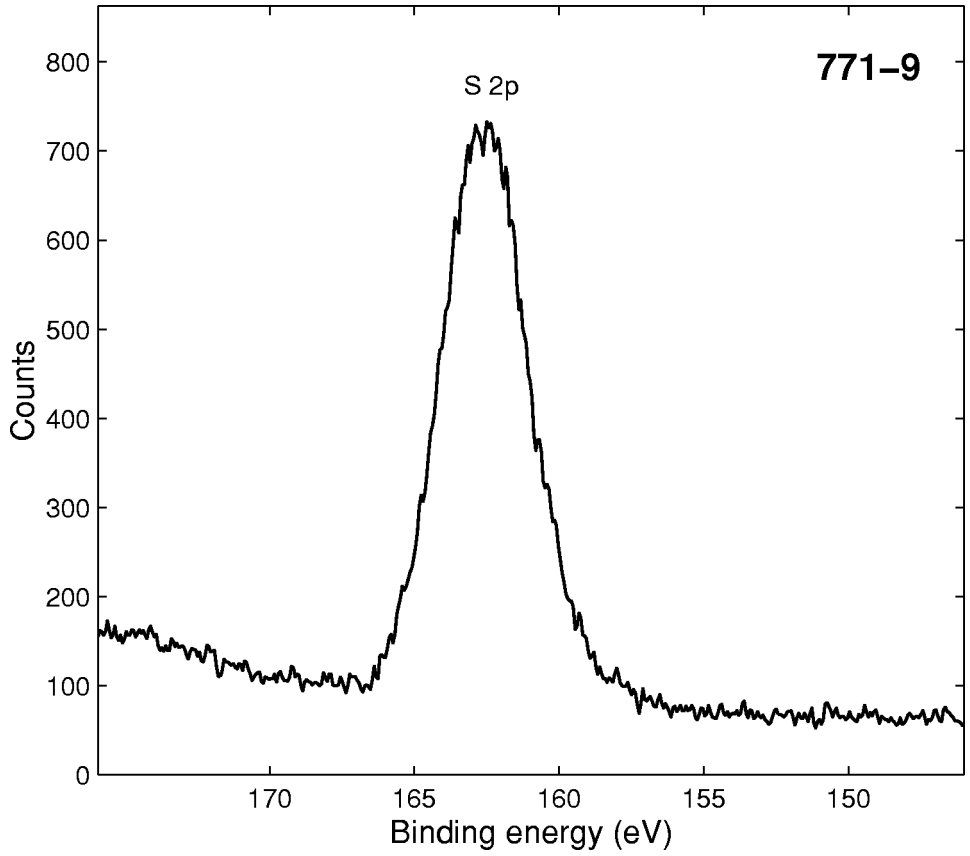
Emission Angle: 55°

Total Signal Accumulation Time: 552 s

Total Elapsed Time: 721 s

Number of Scans: 8

Effective Detector Width: 6.09 eV



- **Accession #:** 00771-09
- **Host Material:** 7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane
- **Technique:** XPS
- **Spectral Region:** S 2*p*

Instrument: Surface Science Laboratories, Inc. 101

Excitation Source: Al K_α monochromatic

Source Energy: 1486.6 eV

Source Strength: 200 W

Source Size: 0.8 mm × 0.8 mm

Incident Angle: 55°

Analyzer Type: spherical sector

Analyzer Pass Energy: 150 eV

Analyzer Resolution: 1.5 eV

Emission Angle: 55°

Total Signal Accumulation Time: 276 s

Total Elapsed Time: 445 s

Number of Scans: 4

Effective Detector Width: 15.1 eV