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Item Type	Peer-Reviewed;Article;Published version
Authors	Hoggard, Jamin;Carlson, Eric D.;Frederickson, Scott H.;Monson, Chris F.;Gertsch, Kevin R;Bronson, R. Todd;Savage, Paul B.;Linford, Matthew R.;Husseini, Ghaleb
Citation	Hoggard, J., Carlson, E. D., Frederickson, S. H., Monson, C. F., Gertsch, K. R., Bronson, R. T., Savage, P. B., Linford, M. R., & Husseini, G. A. (2002). Analysis of 7,13-Bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane by XPS. <i>Surface Science Spectra</i> , 9(1), 227-233. https://doi.org/10.1116/11.20030301
DOI	10.1116/11.20030301
Publisher	American Vacuum Society
Download date	2026-03-08 13:57:32
Link to Item	http://hdl.handle.net/11073/21334

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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(Received 31 March 2003; accepted 14 October 2003; published 8 March 2004)

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

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 fluoroionophores 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: C₃₂H₄₃N₆O₂S

Form: powder

Structure: C₃₂H₄₃N₆O₂S

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/SiO₂) was first cleansed with a solution of NH₄OH (conc.), H₂O₂ (conc.)

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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

(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: $<1.97 \times 10^{-7}$ 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 \times 0.8 mm

Analyzer Width at 84 eV: 1500 μm \times 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°

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 1s	284.8	4.3	199	1.00	60.6	...
00771-03	C 1s	284.5	4.1	455	1.00	61.2	...
00771-04	N 1s	398.8	5.7	33	1.68	10.1	...
00771-05	N 1s	398.2	4.1	72	1.68	9.7	...
00771-06	O 1s	5530.6	4.4	49	2.50	15.0	...
00771-07	O 1s	530.3	3.7	102	2.50	13.8	...
00771-08	S 2p	162.4	3.8	47	1.79	14.3	...
00771-09	S 2p	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 3s	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.

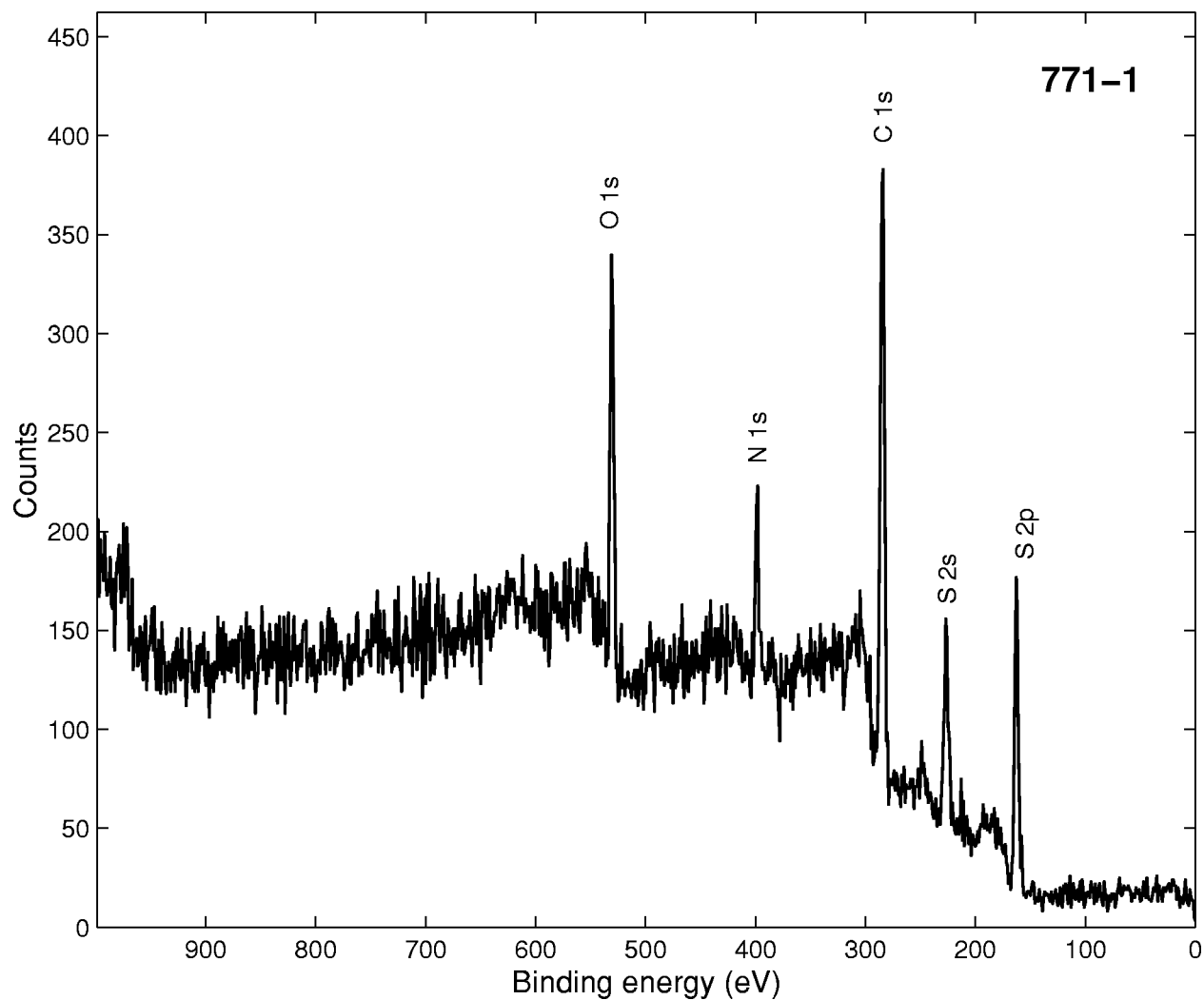
GUIDE TO FIGURES

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

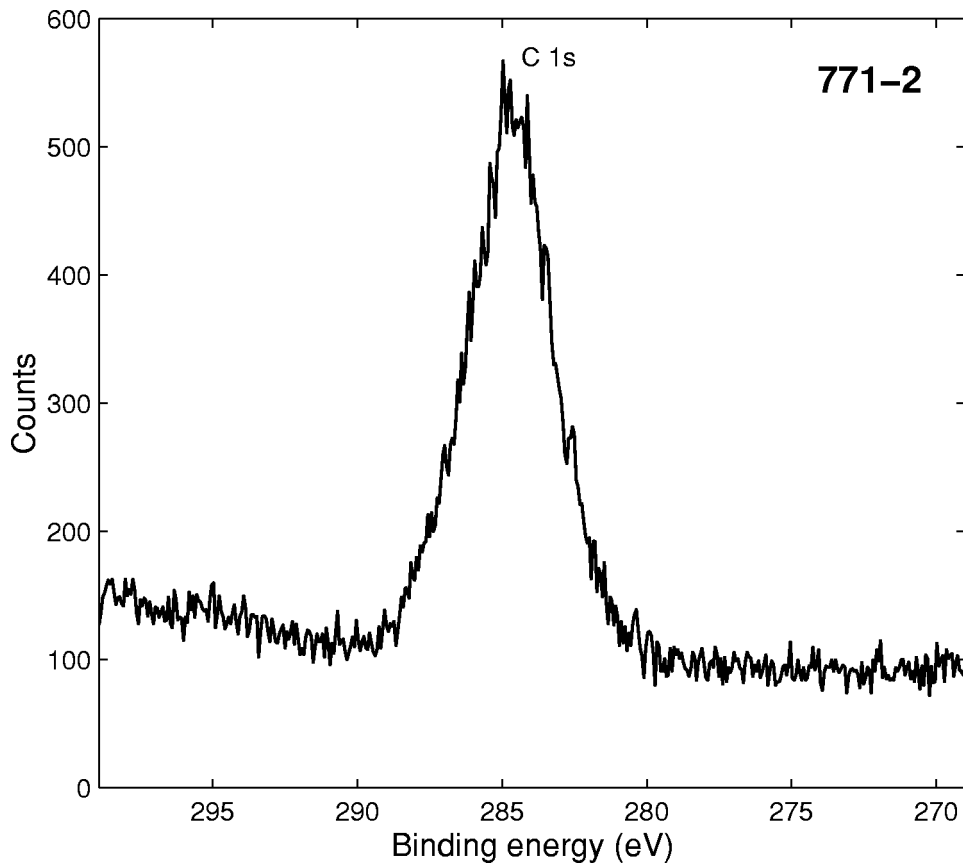
* Voltage shift of the archived (as-measured) spectrum relative to the printed figure. The figure reflects the recommended energy scale correction due to a calibration correction, sample charging, flood gun, or other phenomenon.

1. Pass energy 150 eV

2. Pass energy 50 eV

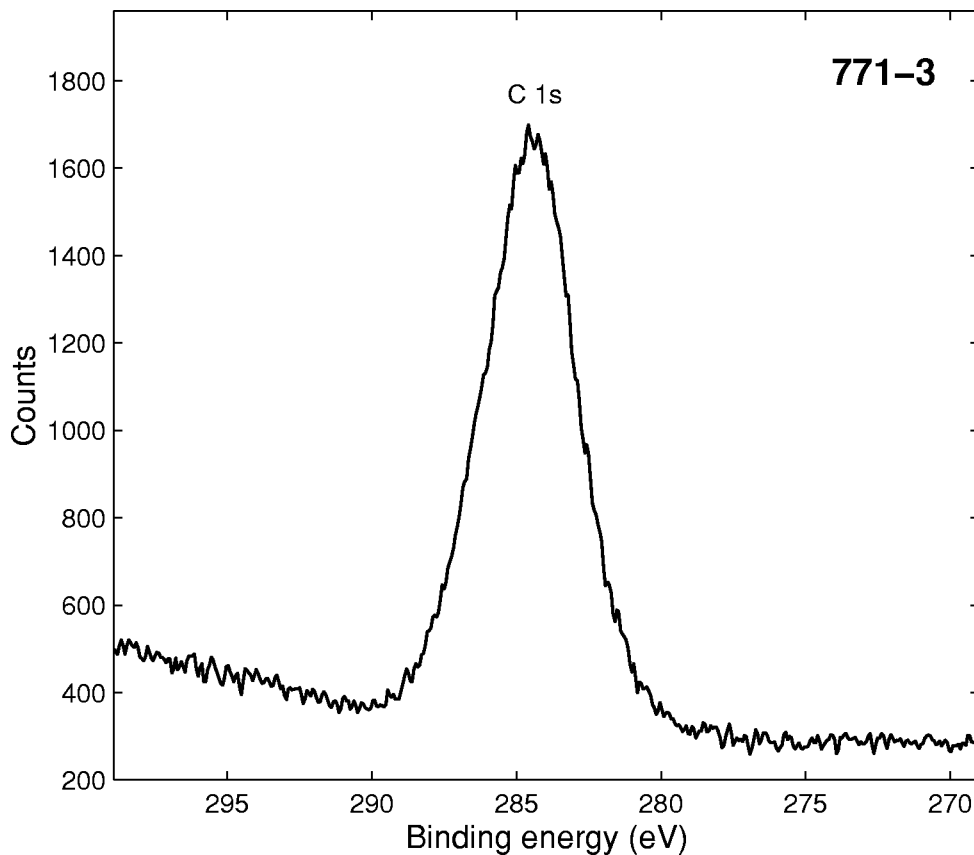


Accession #	00771-01
Host Material	7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl 1,4,7,13-tetraaza-10-thiacyclopentadecane
Technique	XPS
Spectral Region	survey
Instrument	Surface Science Laboratories, Inc. 101
Excitation Source	Al K_{α} monochromatic
Source Energy	1486.6 eV
Source Strength	200 W
Source Size	0.8 mm \times 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



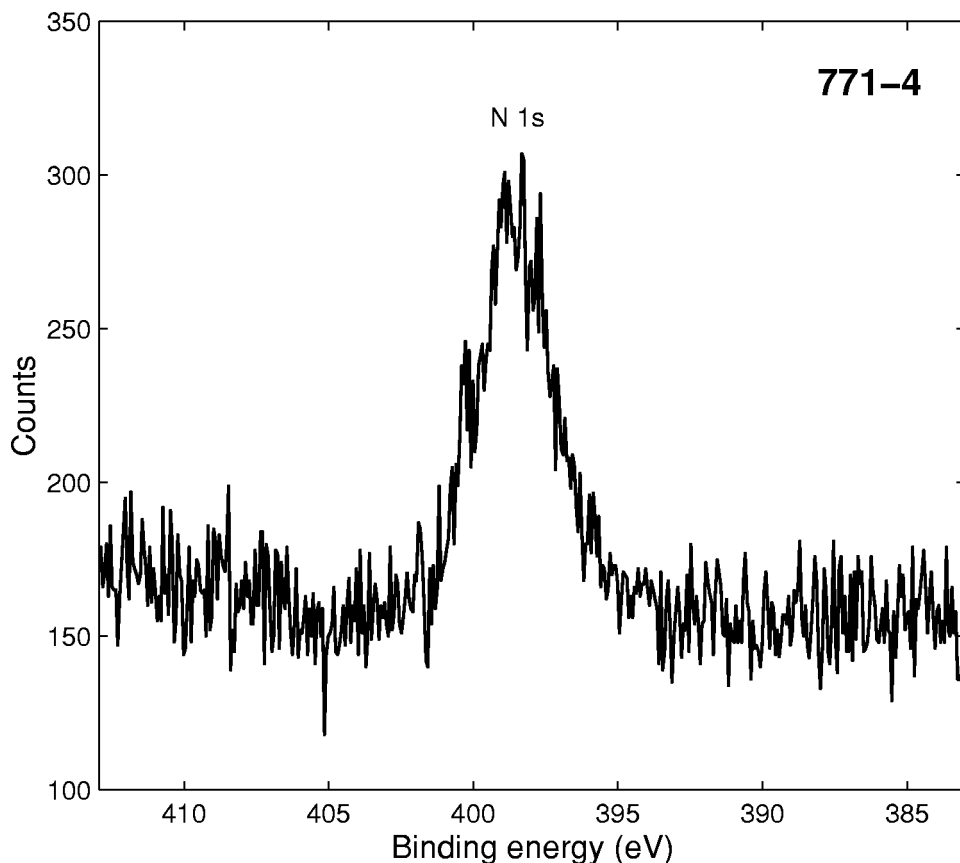
■ **Accession #:** 00771-02
 ■ **Host Material:** 7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane
 ■ **Technique:** XPS
 ■ **Spectral Region:** C 1s

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 \times 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



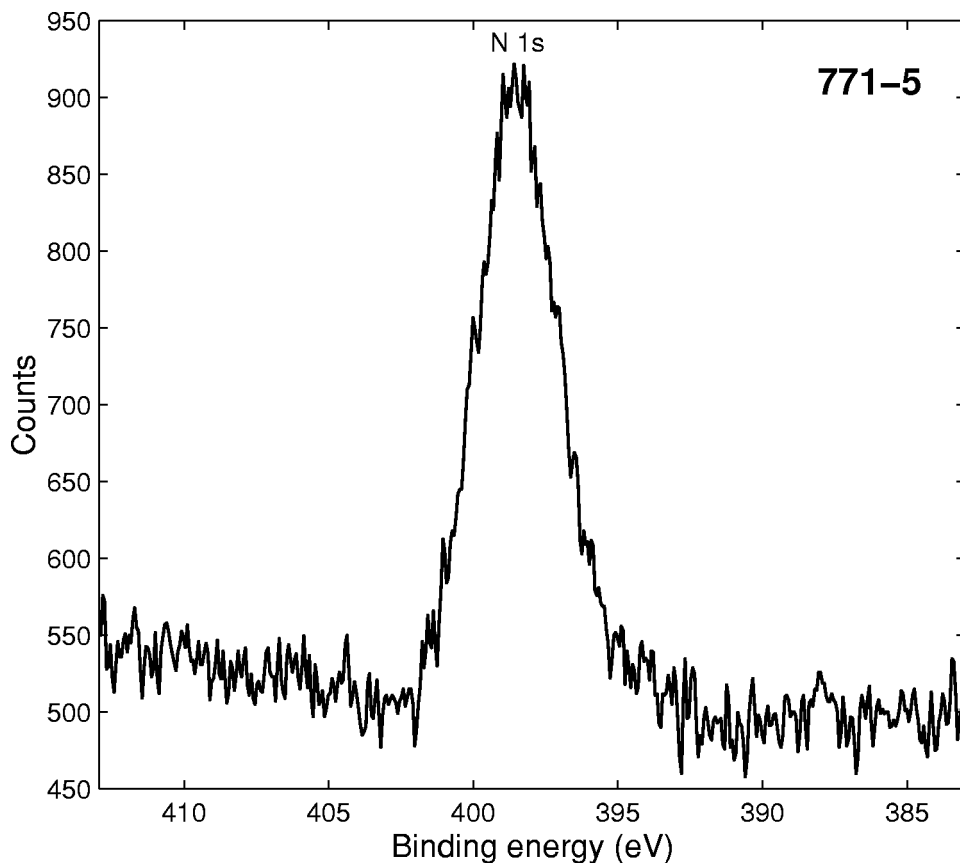
■ **Accession #:** 00771-03
 ■ **Host Material:** 7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane
 ■ **Technique:** XPS
 ■ **Spectral Region:** C 1s

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 \times 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



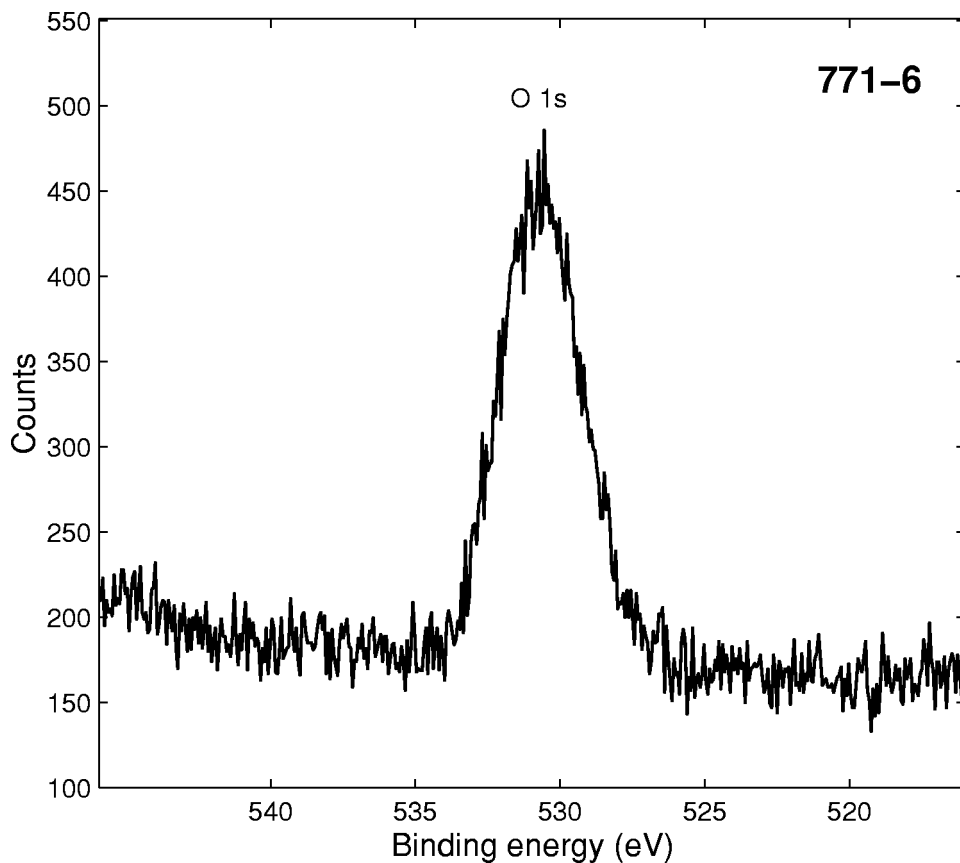
■ **Accession #:** 00771-04
 ■ **Host Material:** 7,13-bis(8-hydroxy-2-quinolinyl)methyl-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane
 ■ **Technique:** XPS
 ■ **Spectral Region:** N 1s

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 \times 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



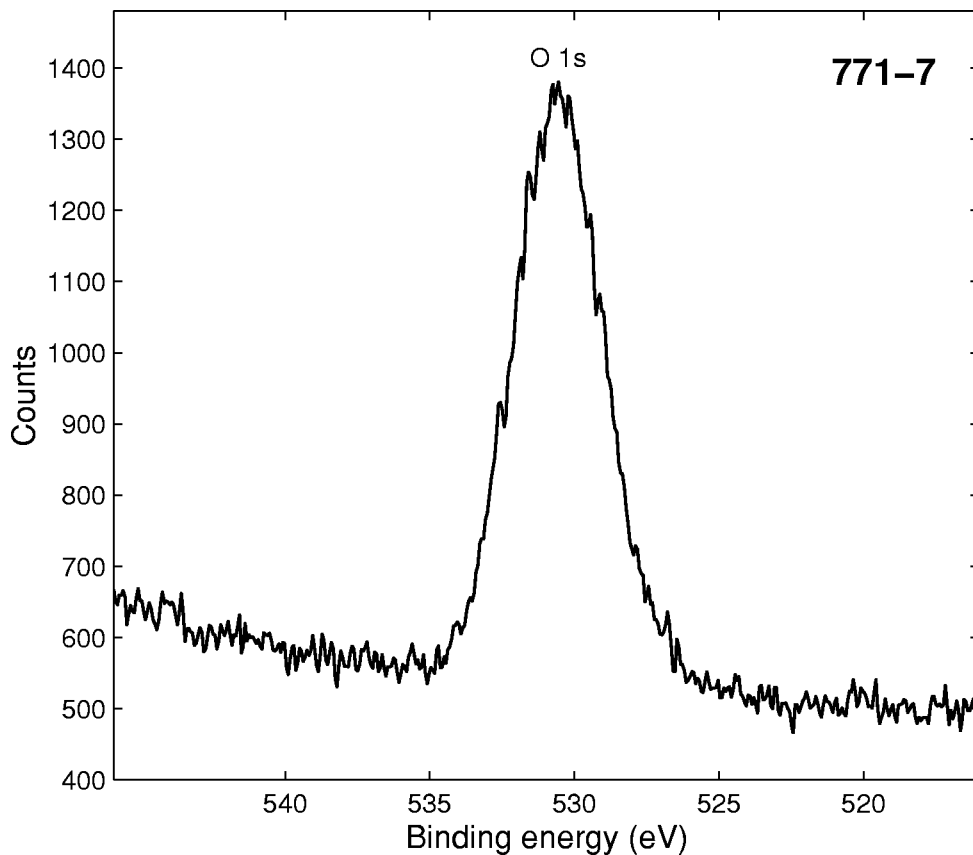
■ **Accession #:** 00771-05
 ■ **Host Material:** 7,13-bis(8-hydroxy-2-quinolinyl)methyl-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane
 ■ **Technique:** XPS
 ■ **Spectral Region:** N 1s

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 \times 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



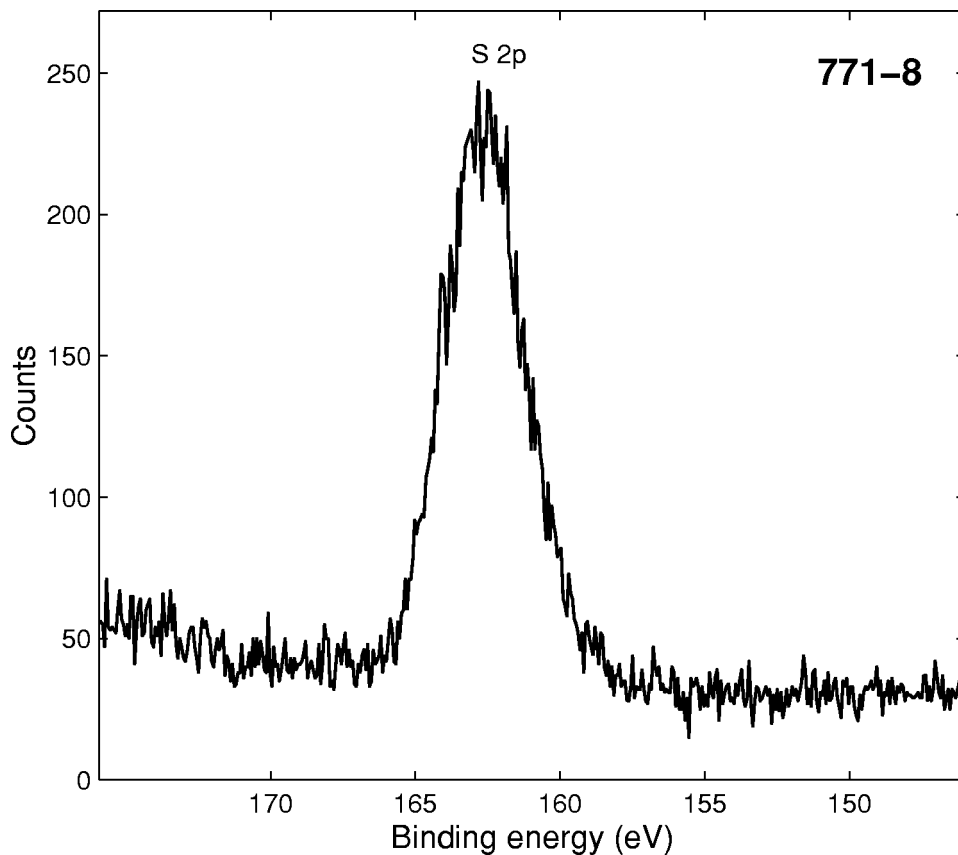
■ **Accession #:** 00771-06
 ■ **Host Material:** 7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane
 ■ **Technique:** XPS
 ■ **Spectral Region:** O 1s

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 \times 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



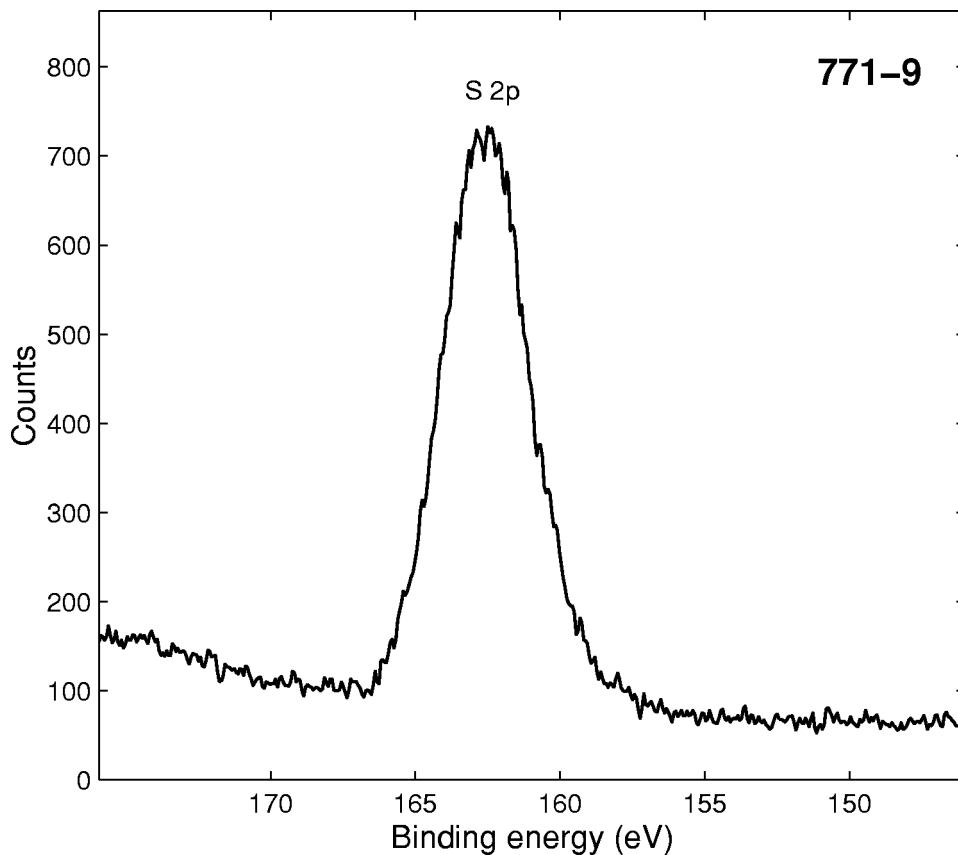
■ **Accession #:** 00771-07
 ■ **Host Material:** 7,13-bis((8-hydroxy-2-quinolinyl)methyl)-1,4-dimethyl-1,4,7,13-tetraaza-10-thiacyclopentadecane
 ■ **Technique:** XPS
 ■ **Spectral Region:** O 1s

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 \times 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 2p

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 \times 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 2p

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 \times 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