

Analysis of 10,16-Diaza-1,4,7,13-tetrathiacyclooctane-9,17-dione by XPS

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(Received 1 April 2003; accepted 14 October 2003; published 8 March 2004)

10,16-Diaza-1,4,7,13-tetrathiacyclooctane-9,17-dione is important for its use as a fluorophoric metal detector. XPS was used to analyze the compound. The compound was mounted on a silicon plate using sticky tape. © 2004 American Vacuum Society. [DOI: 10.1116/11.20030402]

Keywords: XPS; crown-ethers; elemental analysis

PACS: 82.80.Pv, 79.60.Fr

INTRODUCTION

The development of compounds that selectively respond to certain metal ions is an important area of research. 10,16-Diaza-1,4,7,13-tetrathiacyclooctane-9,17-dione is important for its use as a fluorophoric metal detector. In this experiment the XPS spectra of 10,16-diaza-1,4,7,13-tetrathiacyclooctane-9,17-dione were analyzed. The ligand 8-hydroxyquinoline changes fluorescence characteristics when it is complexed with a metal ion. This characteristic is important for its use as a metal ion detector (Ref. 1).

SPECIMEN DESCRIPTION

Host Material: 10,16-diaza-1,4,7,13-tetrathiacyclooctane-9,17-dione

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

Chemical Name: 10,16-diaza-1,4,7,13-tetrathiacyclooctane-9,17-dione

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

Host Composition: C₁₂H₂₂N₂O₂S₄

Form: powder

Structure: C₁₂H₂₂N₂O₂S₄

History & Significance: see the Introduction

As Received Condition: powder

Analyzed Region: same as host material

Ex Situ Preparation/Mounting: The silicon surfaces were first cleaned with a solution of NH₄OH (conc.) H₂O₂ (conc.) (50:50) (v/v) for 30 min at room temperature. A piece of double sided sticky tape was attached to the surface of the clean wafer. The powder of the compound (10,16-Diaza-1,4,7,13-tetrathiacyclooctane-9,17-dione) was mounted on the sticky tape. The wafer was then mounted into the XPS.

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

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Accession # 00776

Technique: XPS

Host Material: 10,16-Diaza-1,4,7,13-tetrathiacyclooctane-9,17-dione

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: 9

Spectral Category: technical

Pressure During Analysis: $<1.33 \times 10^{-7}$ 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 μ m 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 μ m \times 12000 μ m

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. R. T. Brown, J. S. Bradshaw, P. B. Savage, S. Fuangswasdi, S. C. Lee, K. Krakowiak, and R. M. Izatt, *J. Org. Chem.* **66**, 4752 (2001).

SPECTRAL FEATURES TABLE

Spectrum ID #	Element/Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (counts)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
00776-02	S 2 <i>p</i>	157.17	4.83	12370	1.93	11.27	...
00776-03	S 2 <i>p</i>	154.25	4.40	36729	1.93	24.02	...
00776-04	C 1 <i>s</i>	279.99	1.45	12029	1.00	61.63	...
00776-05	C 1 <i>s</i>	279.86	5.05	60712	1.00	44.87	...
00776-06	N 1 <i>s</i>	392.65	8.08	6328	1.61	19.36	...
00776-07	N 1 <i>s</i>	392.45	5.57	12283	1.61	14.68	...
00776-08	O 1 <i>s</i>	524.64	4.68	10992	2.5	2.5	...
00776-09	O 1 <i>s</i>	523.79	4.34	27748	2.5	16.44	...

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 4 <i>f</i> _{7/2}	83.92	0.98	2200	10.67
... ^b	Au 4 <i>f</i> _{7/2}	83.92	1.6	6000	10.67
... ^c	Cu 3 <i>s</i>	122.36	3.0	1600	1.05
... ^b	Cu 2 <i>p</i> _{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.

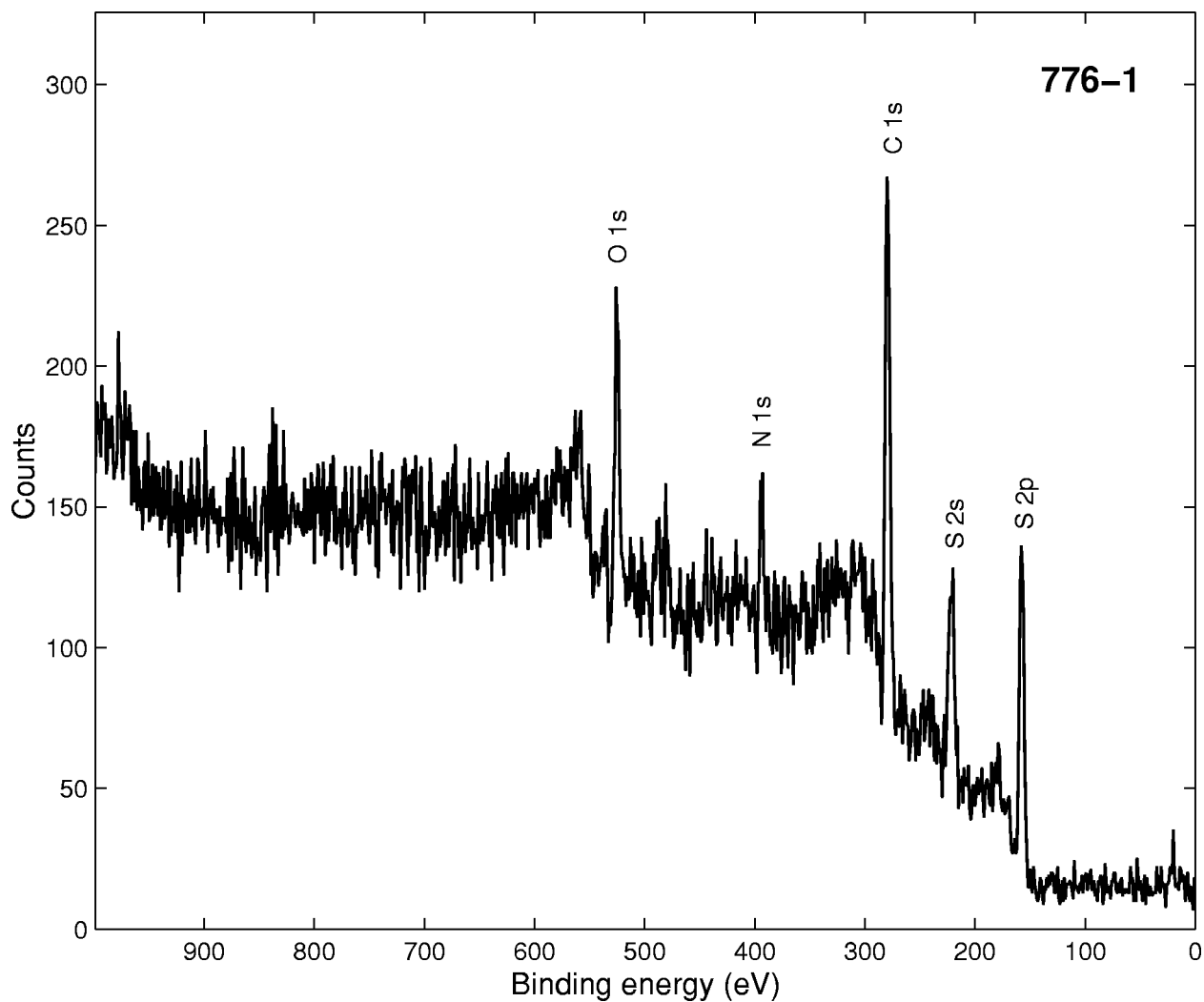
GUIDE TO FIGURES

Spectrum (Accession) #	Spectral Region	Voltage Shift*	Multiplier	Baseline	Comment #
776-1	Survey	0	1	0	1
776-2	S 2 <i>p</i>	0	1	0	2
776-3	S 2 <i>p</i>	0	1	0	1
776-4	C 1 <i>s</i>	0	1	0	2
776-5	C 1 <i>s</i>	0	1	0	1
776-6	N 1 <i>s</i>	0	1	0	2
776-7	N 1 <i>s</i>	0	1	0	1
776-8	O 1 <i>s</i>	0	1	0	1
776-9	O 1 <i>s</i>	0	1	0	2

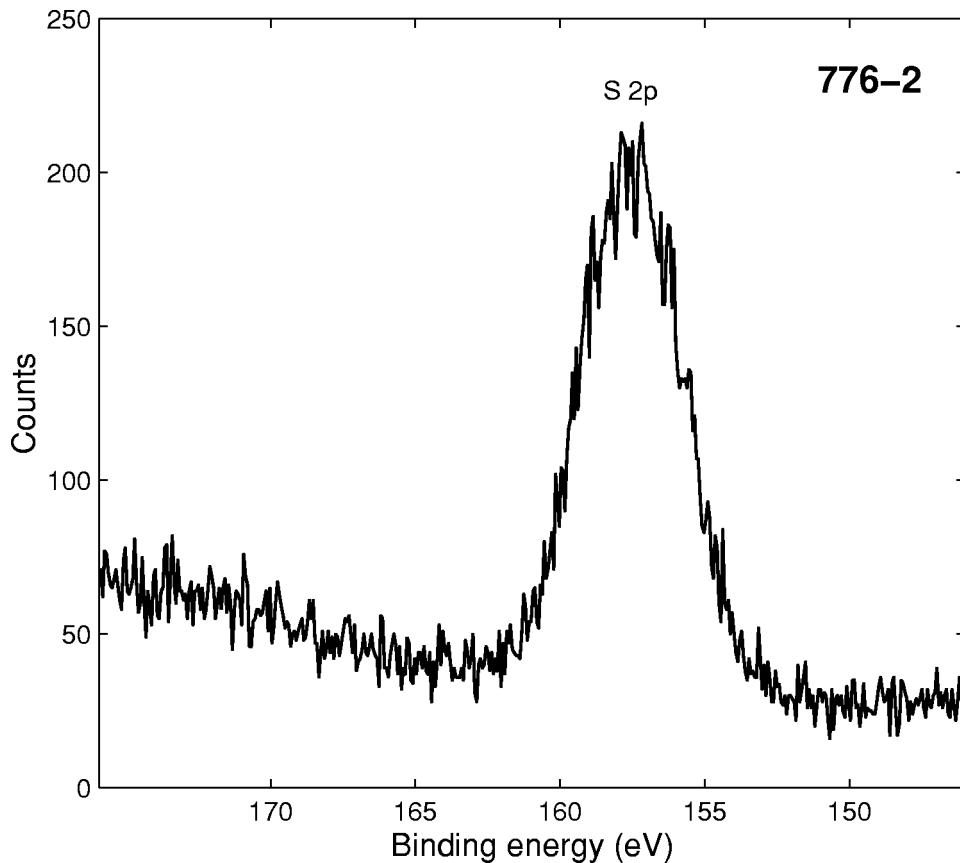
*Voltage shift of the archived (as-measured) spectrum relative to the printed figure. The figure reflects the recommended energy scale correction due to a calibration correction, sample charging, flood gun, or other phenomenon.

1. Pass energy 150 eV

2. Pass energy 50 eV

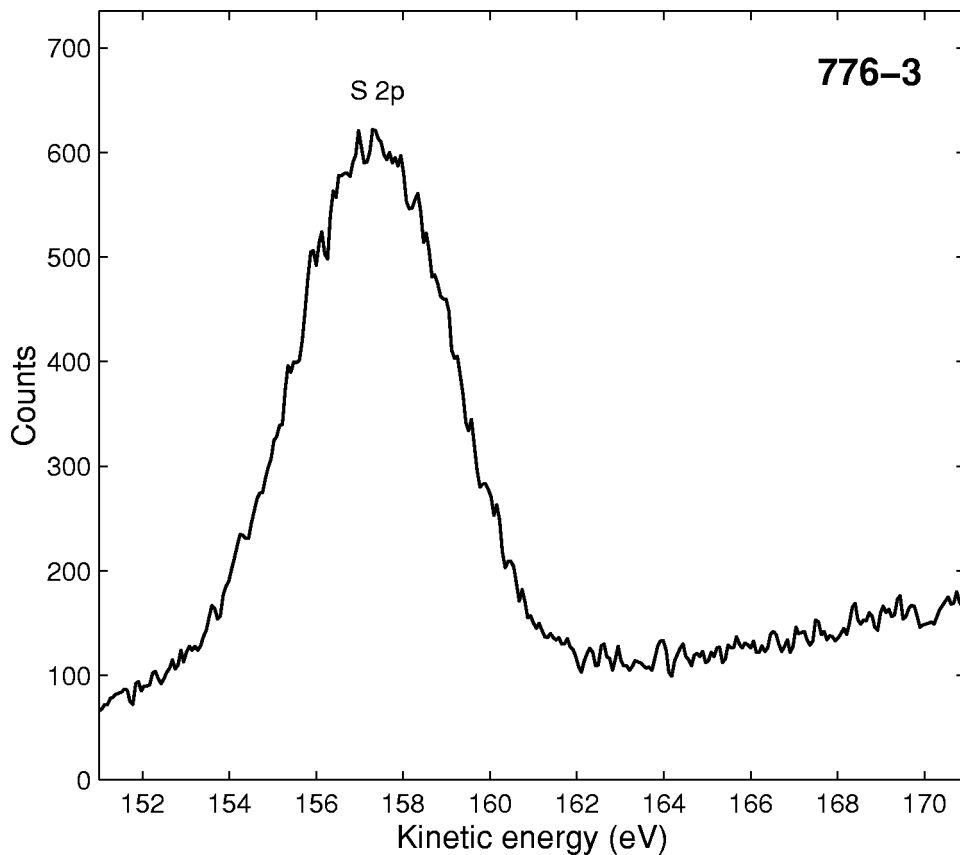


Accession #	00776-01
Host Material	10,16-diaza-1,4,7,13-tetrathiacyclooctane-9,17-dione
Technique	XPS
Spectral Region	survey
Instrument	Surface Science Laboratories, Inc. 101
Excitation Source	Al K_{α} monochromatic
Source Energy	1486.6 eV
Source Strength	200 W
Source Size	0.8 mm \times 0.8 mm
Analyzer 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	4
Effective Detector Width	15.1 eV



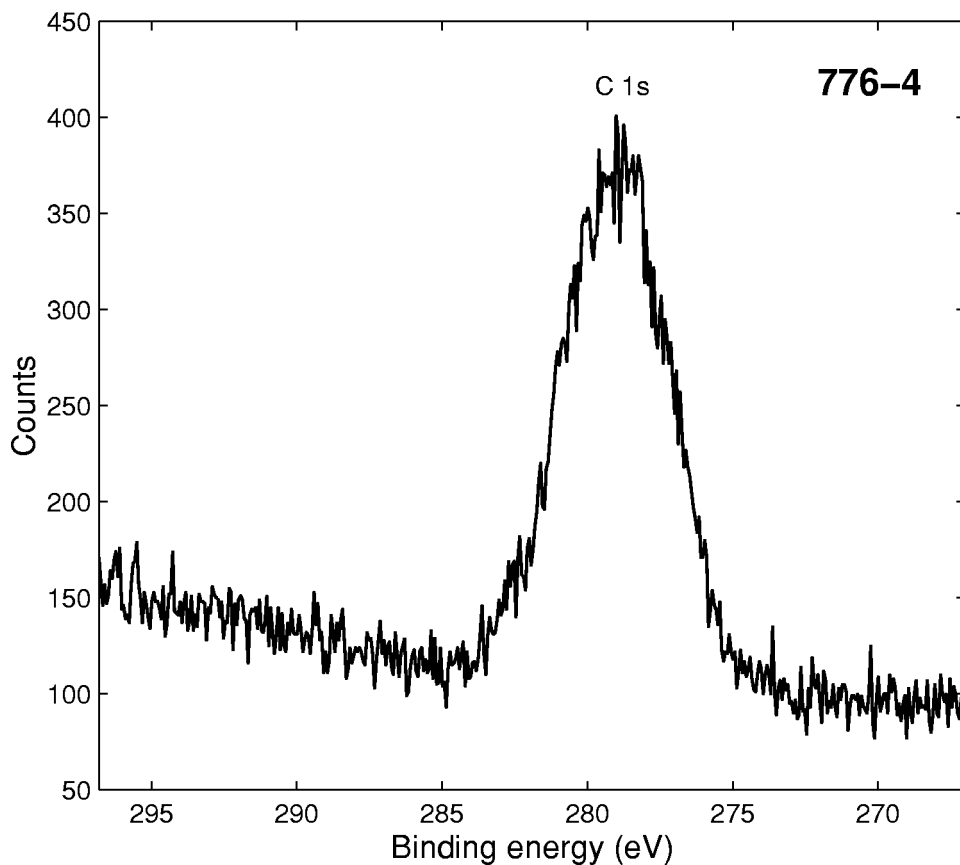
■ **Accession #:** 00776-02
 ■ **Host Material:** 10,16-diaza-1,4,7,13-tetrathiacyclooctane-9,17-dione
 ■ **Technique:** XPS
 ■ **Spectral Region:** S 2p

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: 276 s
 Total Elapsed Time: 721 s
 Number of Scans: 8
 Effective Detector Width: 6.09 eV



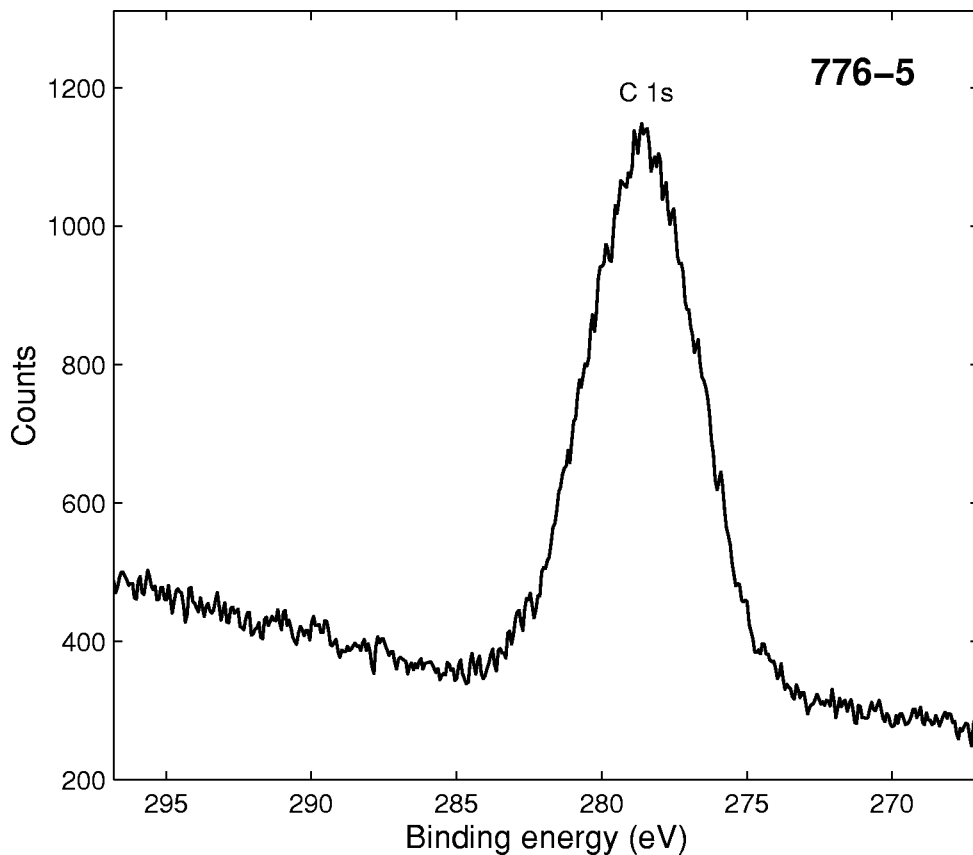
■ **Accession #:** 00776-03
 ■ **Host Material:** 10,16-diaza-1,4,7,13-tetrathiacyclooctane-9,17-dione
 ■ **Technique:** XPS
 ■ **Spectral Region:** S 2p

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



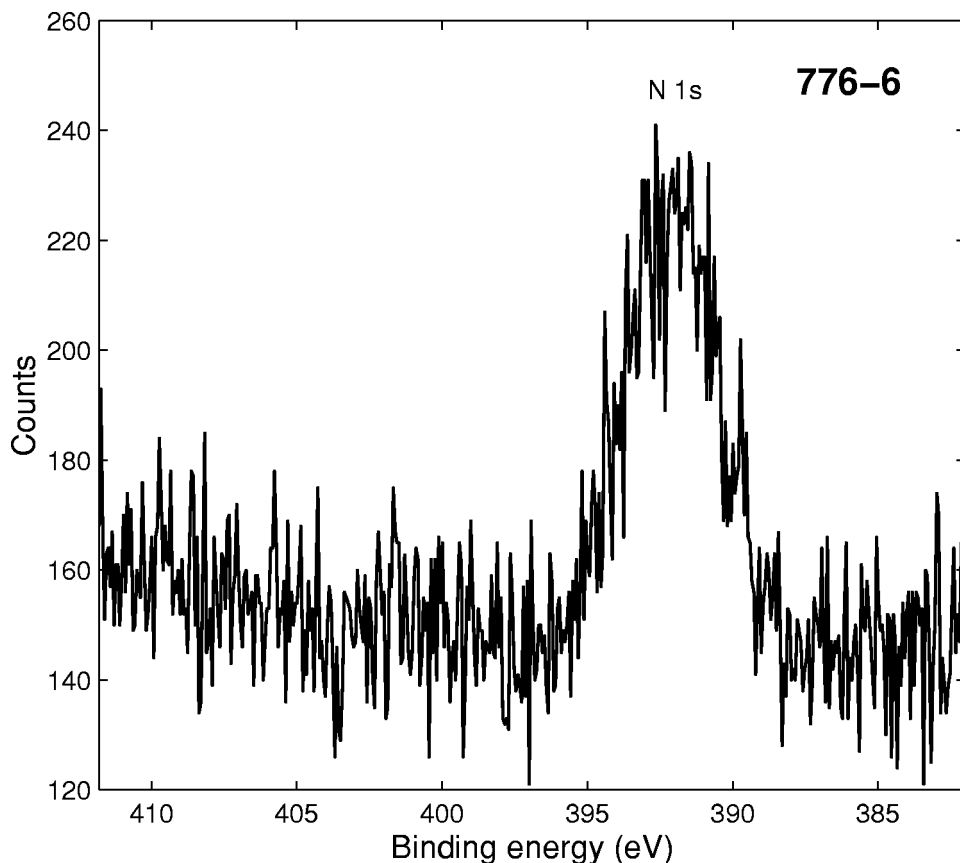
■ **Accession #:** 00776-04
 ■ **Host Material:** 10,16-diaza-1,4,7,13-tetrathiacyclooctane-9,17-dione
 ■ **Technique:** XPS
 ■ **Spectral Region:** C 1s

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: 276 s
 Total Elapsed Time: 721 s
 Number of Scans: 8
 Effective Detector Width: 6.09 eV



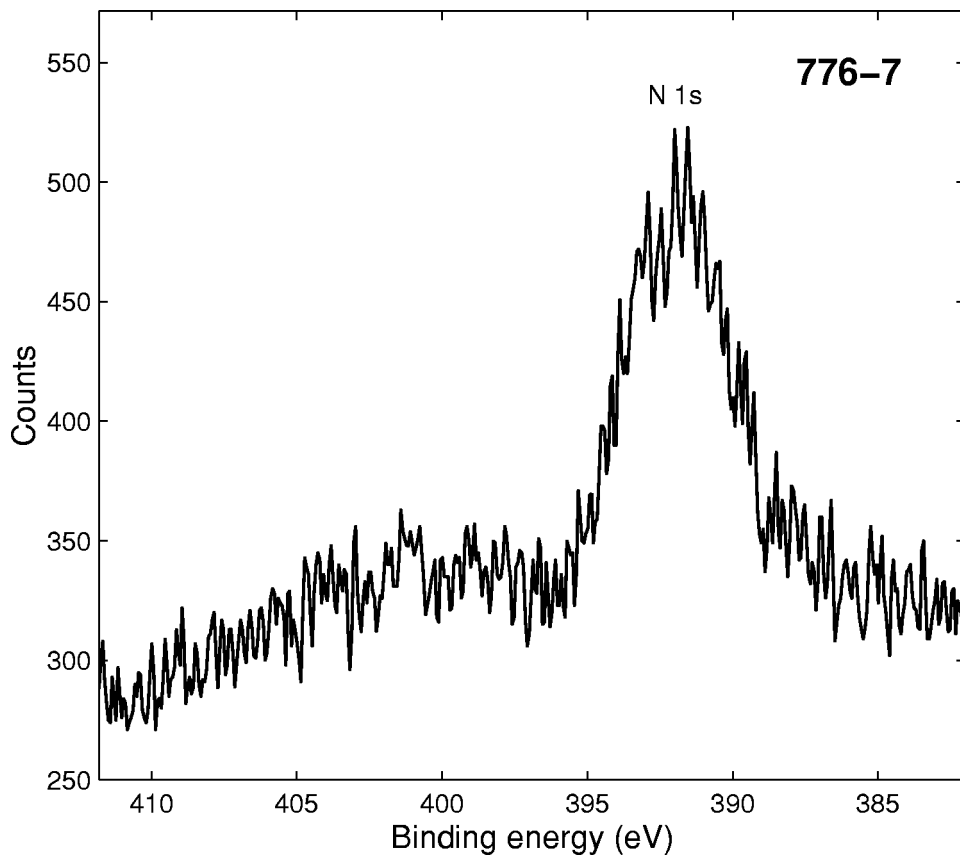
■ **Accession #:** 00776-05
 ■ **Host Material:** 10,16-diaza-1,4,7,13-tetrathiacyclooctane-9,17-dione
 ■ **Technique:** XPS
 ■ **Spectral Region:** C 1s

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



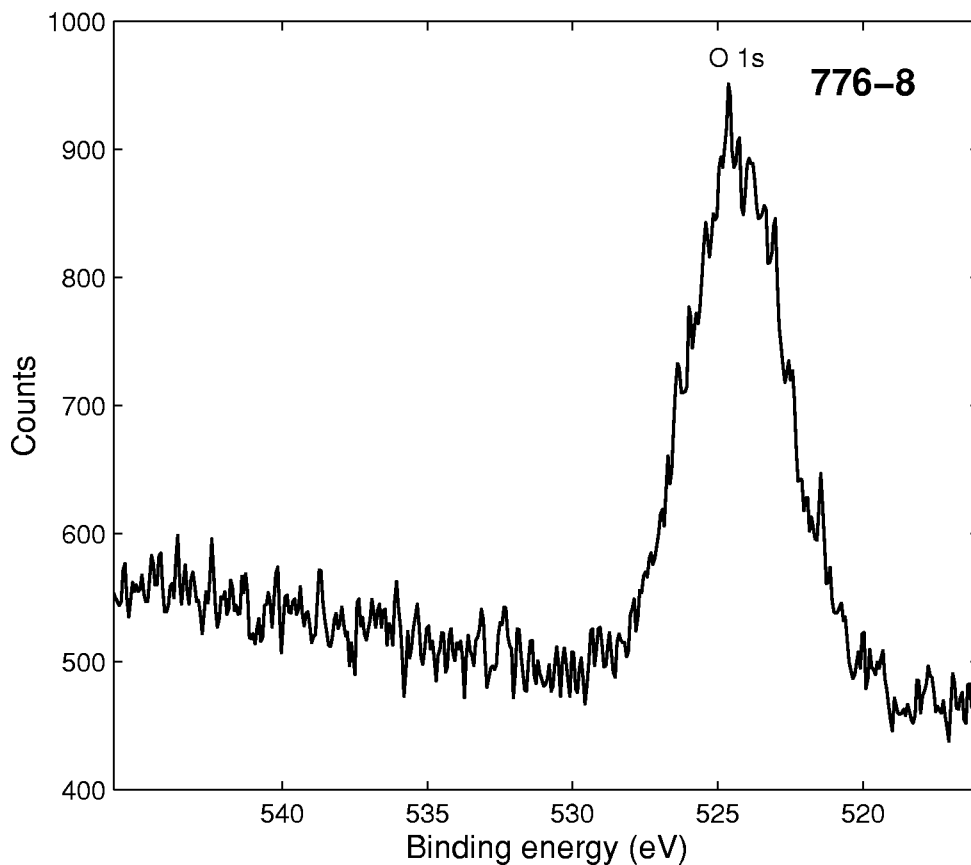
■ **Accession #:** 00776-06
 ■ **Host Material:** 10,16-diaza-1,4,7,13-tetrathiacyclooctane-9,17-dione
 ■ **Technique:** XPS
 ■ **Spectral Region:** N 1s

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: 276 s
 Total Elapsed Time: 721 s
 Number of Scans: 8
 Effective Detector Width: 6.09 eV



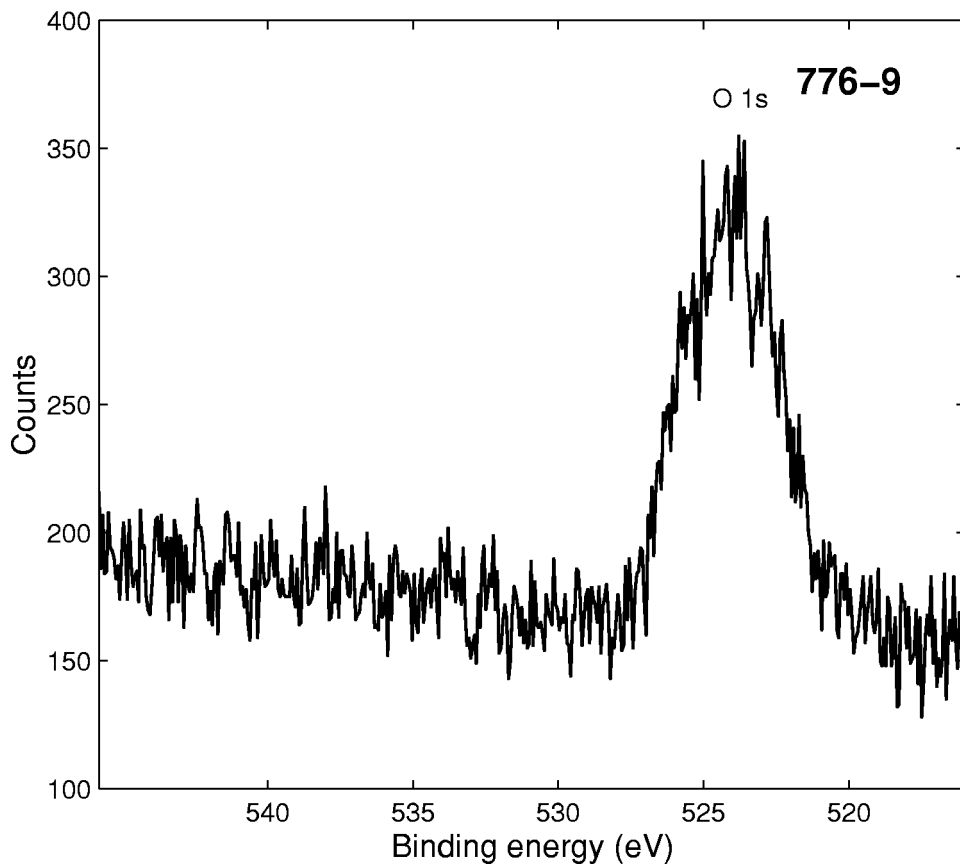
■ **Accession #:** 00776-07
 ■ **Host Material:** 10,16-diaza-1,4,7,13-tetrathiacyclooctane-9,17-dione
 ■ **Technique:** XPS
 ■ **Spectral Region:** N 1s

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: 276 s
 Total Elapsed Time: 445 s
 Number of Scans: 4
 Effective Detector Width: 15.1 eV



■ **Accession #:** 00776-08
 ■ **Host Material:** 10,16-diaza-1,4,7,13-tetrathiacyclooctane-9,17-dione
 ■ **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: 276 s
 Total Elapsed Time: 445 s
 Number of Scans: 4
 Effective Detector Width: 15.1 eV



■ **Accession #:** 00776-09
 ■ **Host Material:** 10,16-diaza-1,4,7,13-tetrathiacyclooctane-9,17-dione
 ■ **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: 276 s
 Total Elapsed Time: 721 s
 Number of Scans: 8
 Effective Detector Width: 6.09 eV