

Analysis of 5-chloro-8-methoxy-2-(bromomethyl)quinoline by XPS

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Macrocyclic ethers are used extensively for their ability to form strong and selective interactions with charged species. It has been shown that the incorporation of heterocyclic groups into macrocyclic ethers increase rigidity and interaction with certain ions. Alkylation of these azacrowns with benzylic halides can attach fluoronophoric substituents onto the ring. One of the molecules used for this alkylation is 5-chloro-8-methoxy-2-(bromomethyl)quinoline. Azacrowns with this alkyl ligand have displayed interesting ion complexation properties. © 2004 American Vacuum Society. [DOI: 10.1116/11.20030403]

Keywords: *x-ray photoelectron spectroscopy; pyridinoazacrown ethers; 5-chloro-8-methoxy-2-(bromoethyl)quinoline*

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INTRODUCTION

Macrocyclic ethers are known to display strong and selective interactions with charged species. Because of these unique properties, these macrocycles have been used extensively in ion-exchange resins and other chemical separation techniques (Ref. 1). Additionally, it has been shown that alkylation of these azacrowns with active benzylic halides has been effective in adding a fluoronophoric substituent onto the macrocyclic ring. One of the alkyl ligands used in these reactions is 5-chloro-8-methoxy-2-(bromomethyl)quinoline (Ref. 2). XPS spectra of this compound were obtained and analyzed to determine the atomic percentage of C, O, N, Br, and Cl.

SPECIMEN DESCRIPTION

Host Material: 5-chloro-8-methoxy-2-(bromomethyl)quinoline
Host Material Characteristics: homogeneous; unknown crystallinity; dielectric; organic compound; powder
Chemical Name: 5-chloro-8-methoxy-2-(bromomethyl)quinoline
Source: compound synthesized in Dr. Paul Savage's laboratory, Brigham Young University, Provo, UT
Host Composition: C₁₁H₉NOCIBr
Form: powder
Structure: C₁₁H₉NOCIBr
History & Significance: see the Introduction
As Received Condition: powder
Analyzed Region: same as host material
Ex Situ Preparation/Mounting: A 125 mm diam silicon wafer was first cleaned with a solution of 50:50 concentrated NH₄OH and H₂O₂ for 30 min at room temperature. A piece of double sided sticky tape was then attached to the surface of the clean wafer. A sample of 5-chloro-8-methoxy-2-(bromomethyl)quinoline was mounted to the sticky tape and then mounted

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Accession # 00777

Technique: XPS

Host Material: 5-chloro-8-methoxy-2-(bromomethyl)quinoline

Instrument: Surface Science Laboratories, Inc. 101

Major Elements in Spectrum: C, O, N, Br, Cl

Minor Elements in Spectrum: none

Printed Spectra: 11

Spectra in Electronic Record: 11

Spectral Category: technical

into the XPS instrument.

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: $<8.8 \times 10^{-7}$ 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. M. Newcomb, J. M. Timko, D. M. Walba, and D. J. Cram, *J. Org. Chem.* **49**, 353 (1984).
2. A. V. Burdunov, J. S. Hellier, N. K. Dalley, X. Kou, X. X. Zhang, and R. M. Izatt, *J. Org. Chem.* **60**, 6097 (1995).

SPECTRAL FEATURES TABLE

Spectrum ID #	Element/Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (counts)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
00777-02 ^a	C 1s	280.9	2.749	1166
00777-03 ^b	C 1s	280.7	2.841	3314	1.00	78.47	...
00777-04 ^a	O 1s	528.9	3.944	300
00777-05 ^b	O 1s	528.5	3.965	769	2.50	7.19	...
00777-06 ^a	Br 3d	65.8	2.899	176
00777-07 ^b	Br 3d	66.0	2.912	592	3.19	4.10	...
00777-08 ^a	N 1s	394.0	4.054	185
00777-09 ^b	N 1s	394.4	4.036	415	1.68	5.70	...
00777-10 ^a	Cl 2s	266.6	3.826	176
00777-11 ^b	Cl 2s	266.5	3.830	156	1.71	4.54	...

^a Pass energy 50 eV

^b Pass energy 150 eV

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.

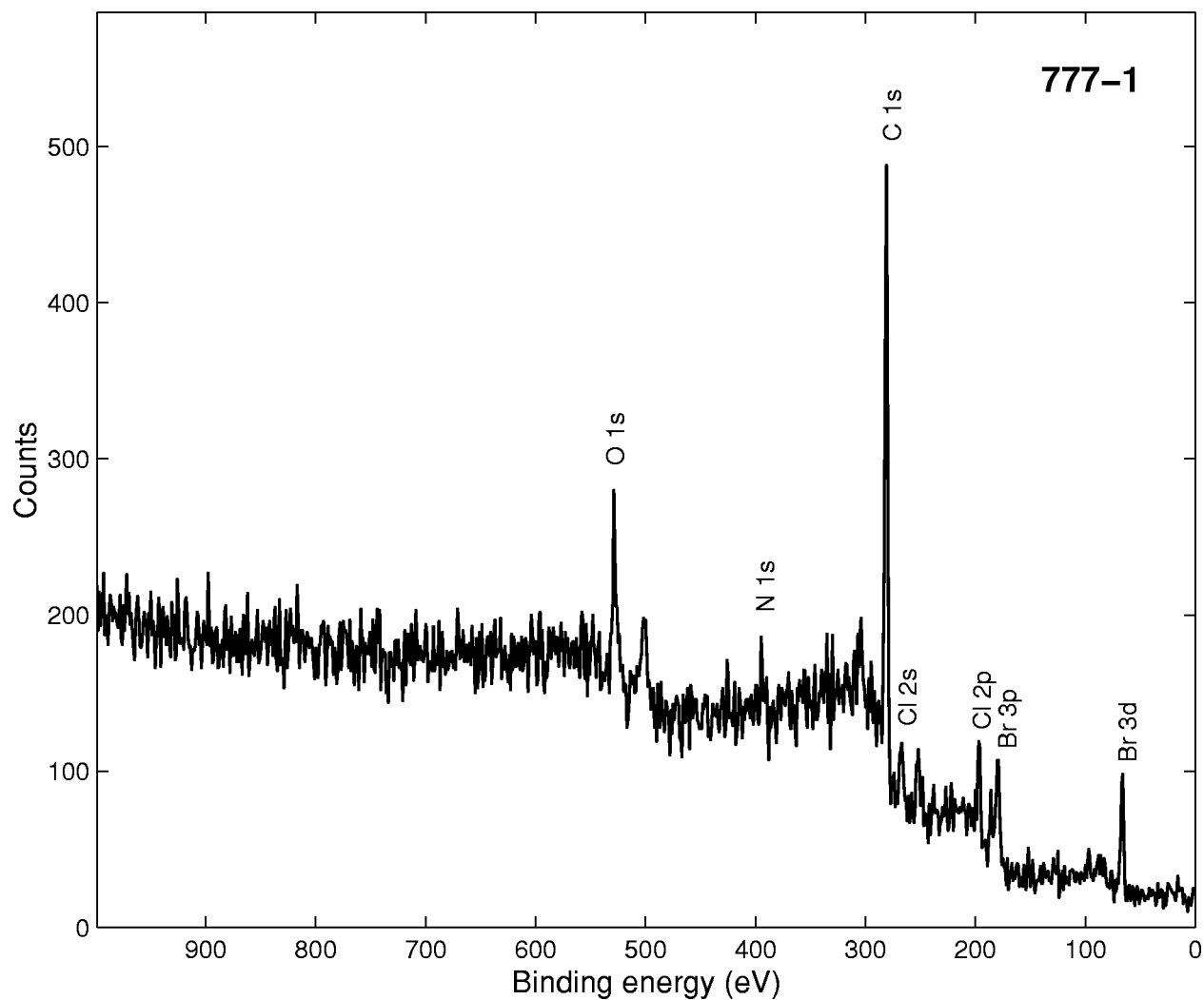
^c Spot size 800 μm, pass energy 150 eV, 3 scans.

GUIDE TO FIGURES

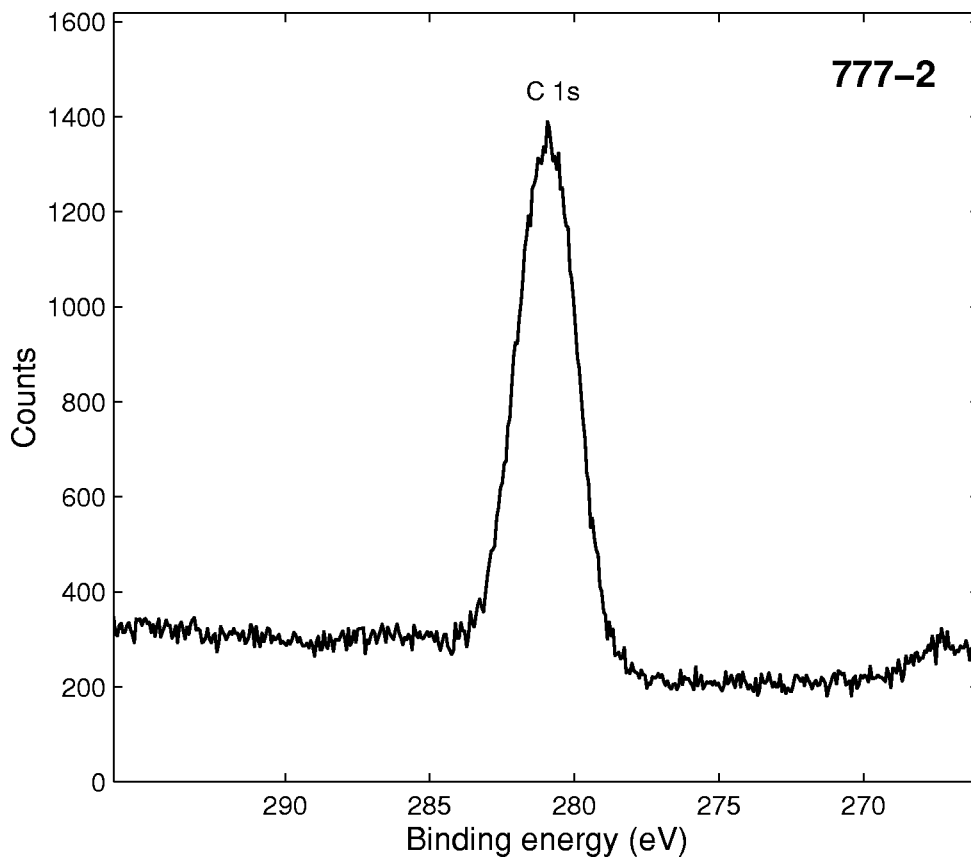
Spectrum (Accession) #	Spectral Region	Voltage Shift*	Multiplier	Baseline	Comment #
777-1	Survey	0	1	0	1
777-2	C 1s	0	1	0	2
777-3	C 1s	0	1	0	1
777-4	O 1s	0	1	0	2
777-5	O 1s	0	1	0	1
777-6	Br 3d	0	1	0	2
777-7	Br 3d	0	1	0	1
777-8	N 1s	0	1	0	2
777-9	N 1s	0	1	0	1
777-10	Cl 2s	0	1	0	2
777-11	Cl 2s	0	1	0	1

* 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

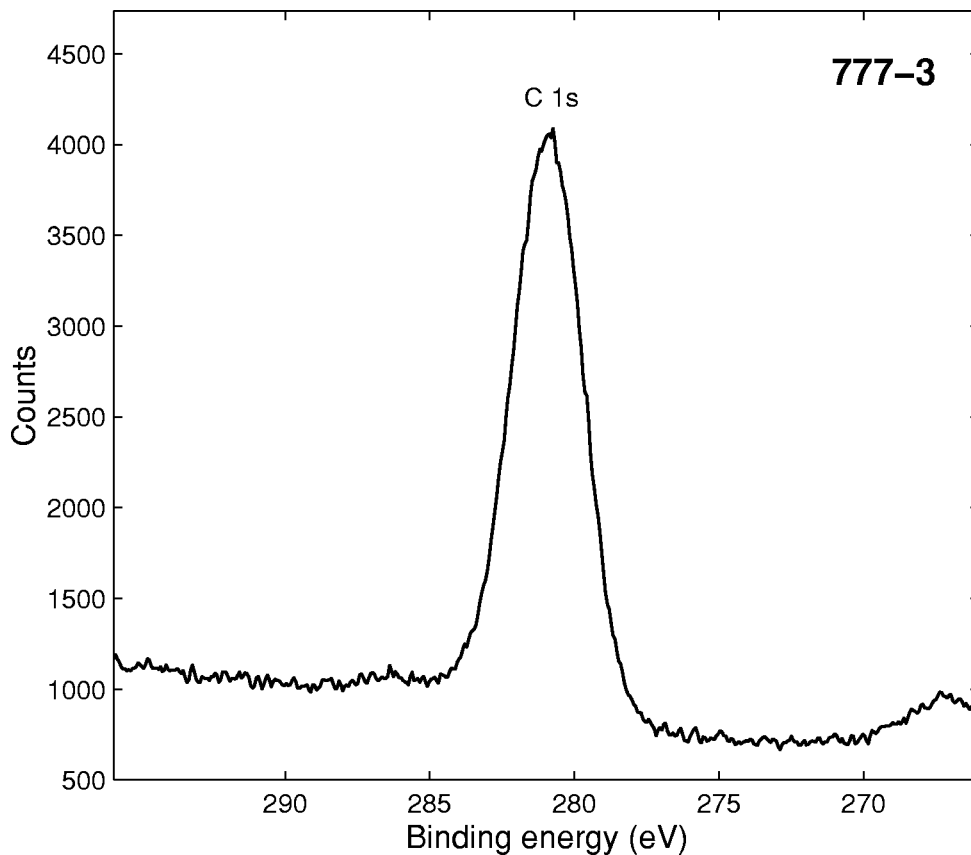


Accession #	00777-01
Host Material	5-chloro-8-methoxy-2-(bromomethyl)quinoline
Technique	XPS
Spectral Region	survey
Instrument	Surface Science Laboratories, Inc. 101
Excitation Source	Al K_{α} monochromatic
Source Energy	1486.6 eV
Source Strength	200 W
Source Size	0.8 mm \times 0.8 mm
Analyzer Type	spherical 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



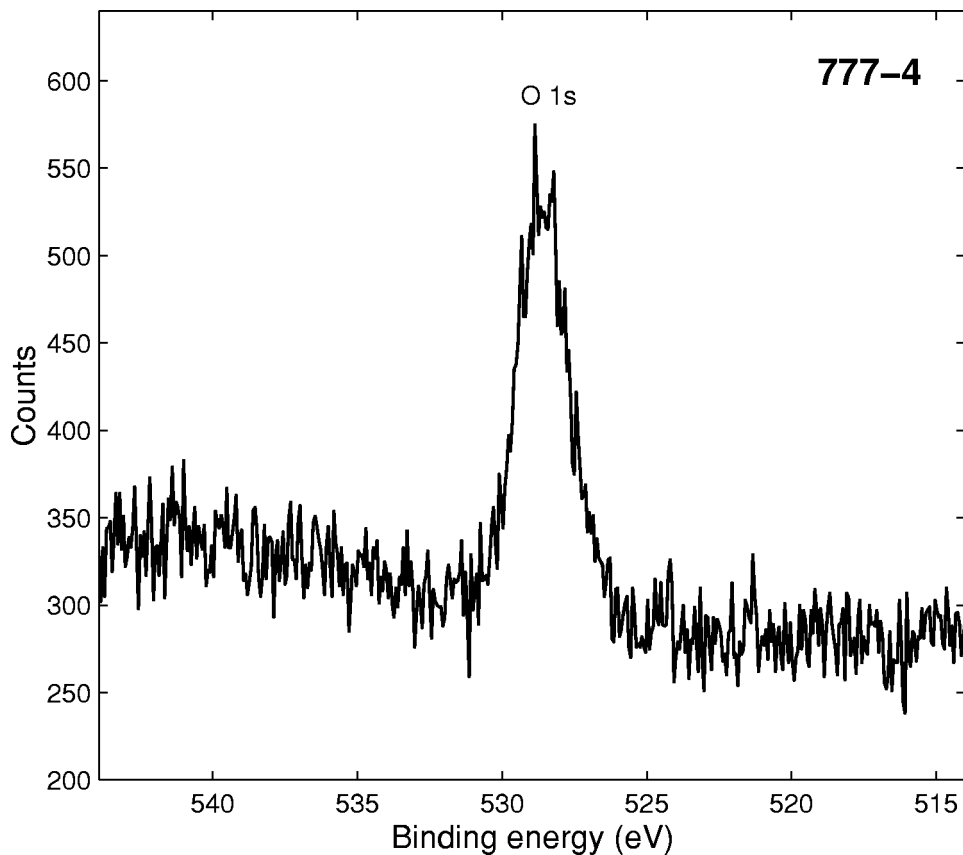
■ **Accession #:** 00777-02
 ■ **Host Material:** 5-chloro-8-methoxy-2-(bromomethyl)-quinoline
 ■ **Technique:** XPS
 ■ **Spectral Region:** C 1s

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



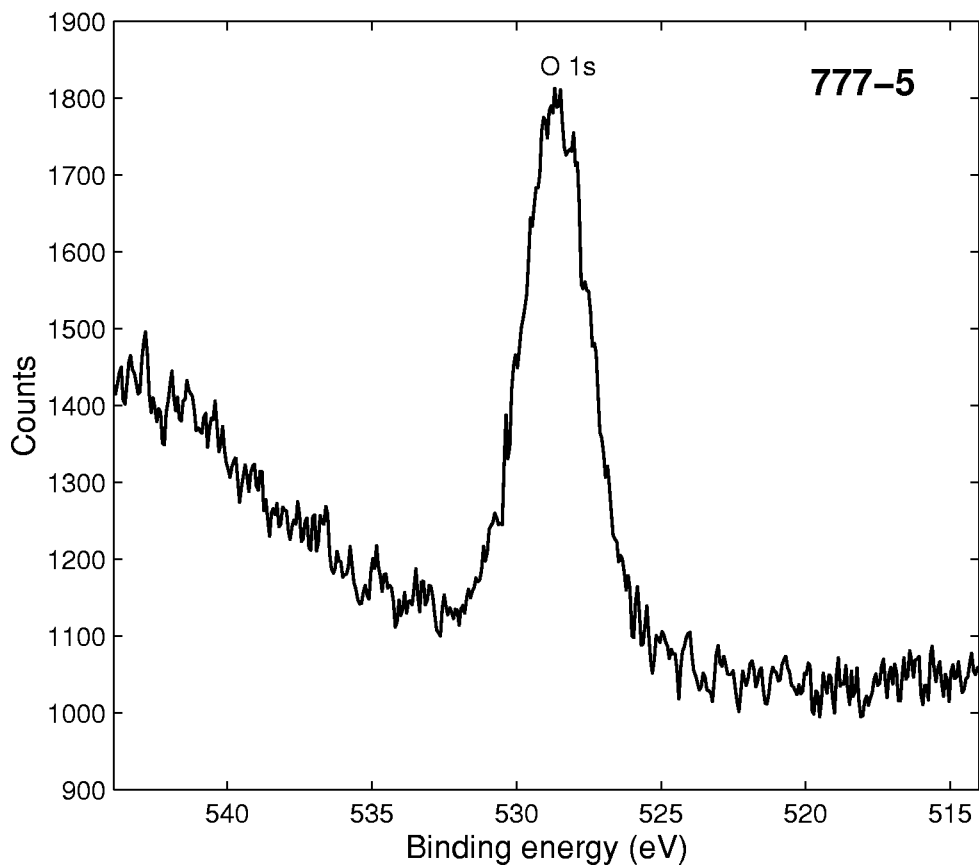
■ **Accession #:** 00777-03
 ■ **Host Material:** 5-chloro-8-methoxy-2-(bromomethyl)-quinoline
 ■ **Technique:** XPS
 ■ **Spectral Region:** C 1s

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



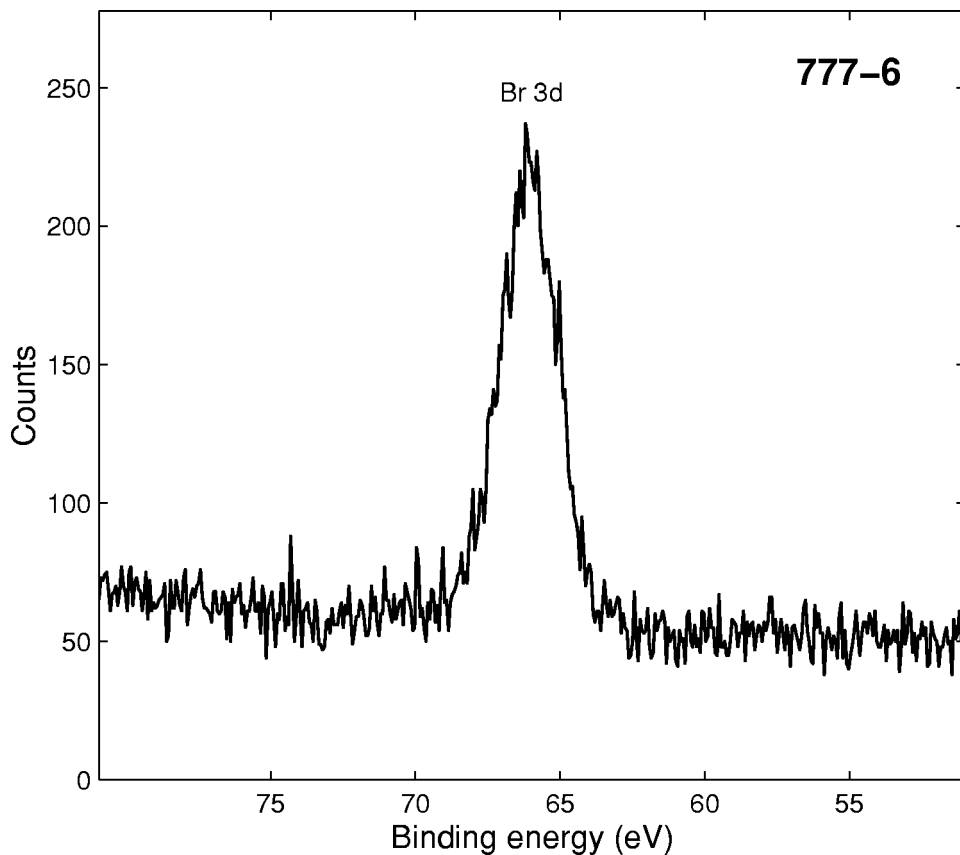
■ **Accession #:** 00777-04
 ■ **Host Material:** 5-chloro-8-methoxy-2-(bromomethyl)-quinoline
 ■ **Technique:** XPS
 ■ **Spectral Region:** O 1s

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 #:** 00777-05
 ■ **Host Material:** 5-chloro-8-methoxy-2-(bromomethyl)-quinoline
 ■ **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 #:** 00777-06

■ **Host Material:** 5-chloro-8-methoxy-2-(bromomethyl)-quinoline

■ **Technique:** XPS

■ **Spectral Region:** Br 3d

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

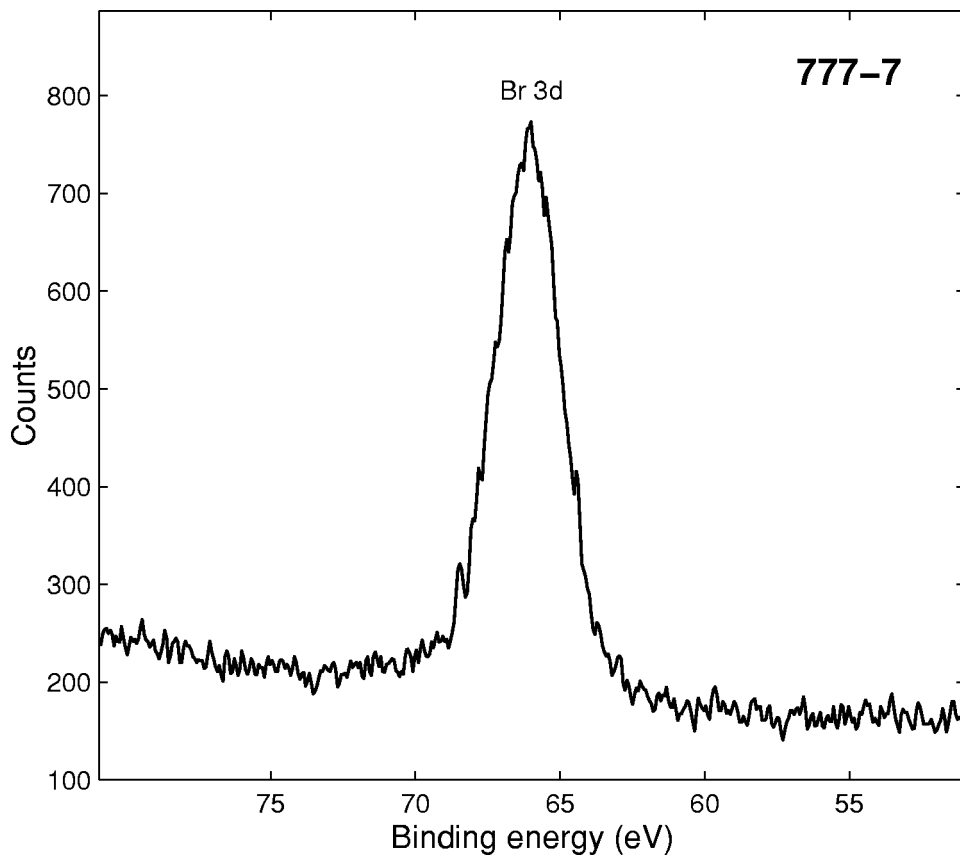
Emission Angle: 55°

Total Signal Accumulation Time: 552 s

Total Elapsed Time: 721 s

Number of Scans: 8

Effective Detector Width: 6.09 eV



■ **Accession #:** 00777-07

■ **Host Material:** 5-chloro-8-methoxy-2-(bromomethyl)-quinoline

■ **Technique:** XPS

■ **Spectral Region:** Br 3d

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

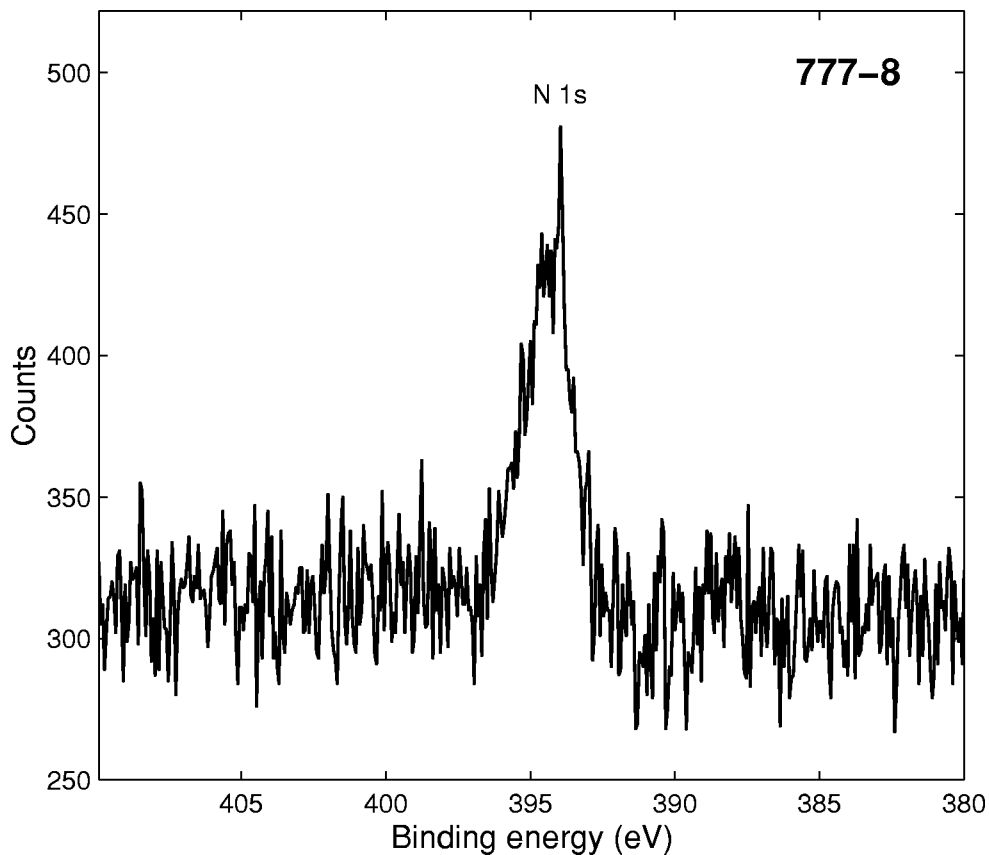
Emission Angle: 55°

Total Signal Accumulation Time: 276 s

Total Elapsed Time: 445 s

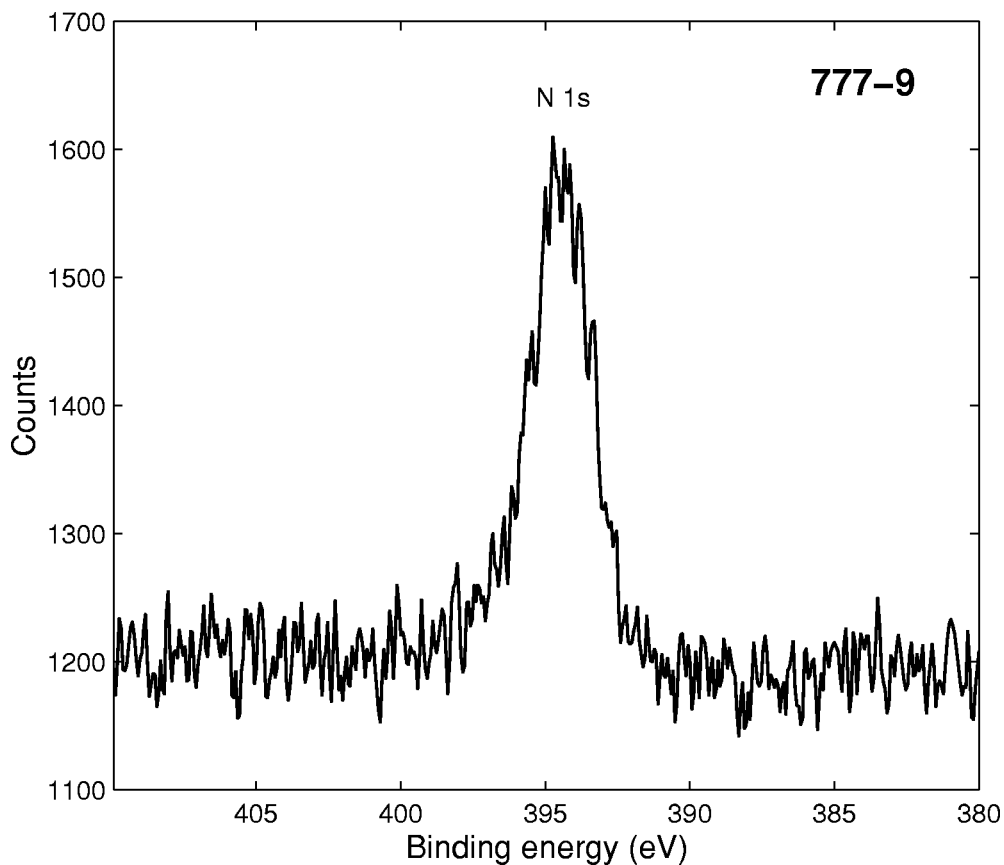
Number of Scans: 4

Effective Detector Width: 15.1 eV



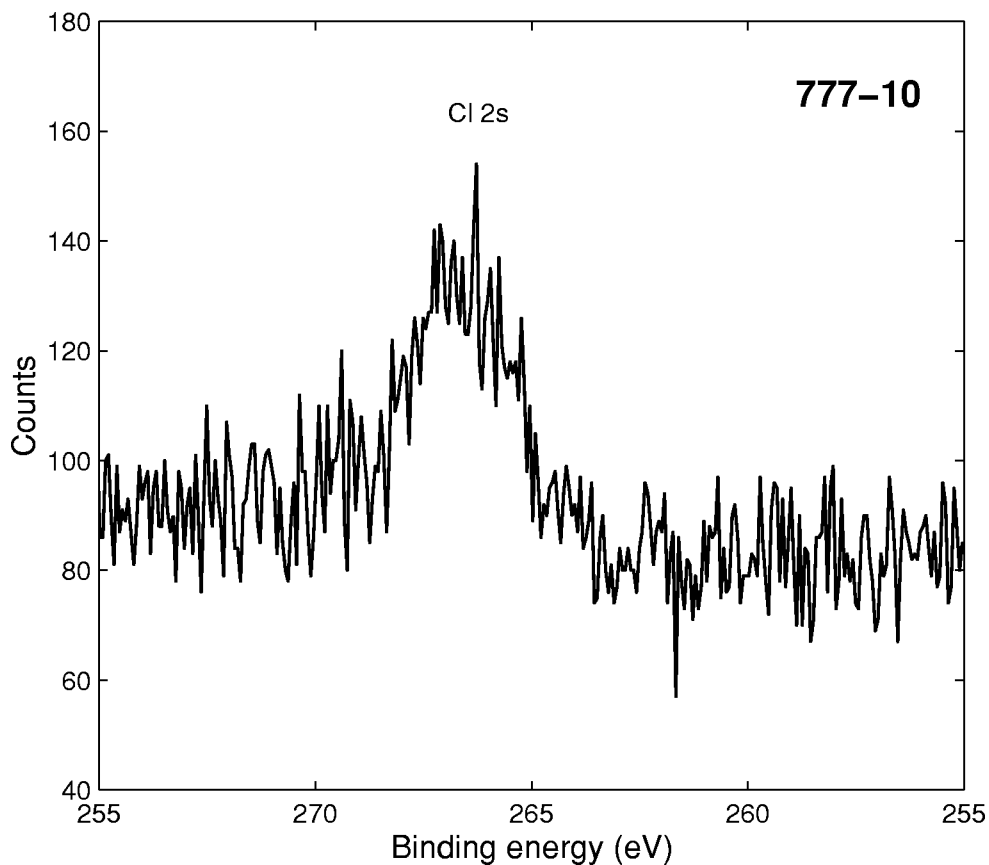
■ **Accession #:** 00777-08
 ■ **Host Material:** 5-chloro-8-methoxy-2-(bromomethyl)-quinoline
 ■ **Technique:** XPS
 ■ **Spectral Region:** N 1s

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



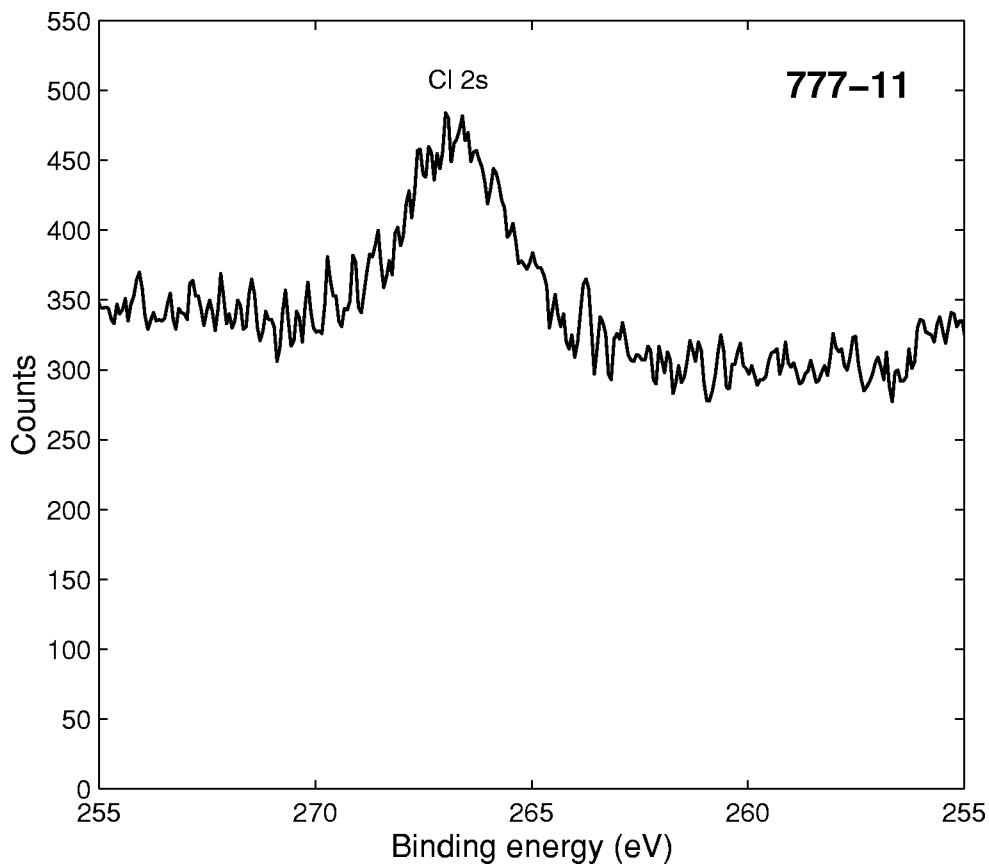
■ **Accession #:** 00777-09
 ■ **Host Material:** 5-chloro-8-methoxy-2-(bromomethyl)-quinoline
 ■ **Technique:** XPS
 ■ **Spectral Region:** N 1s

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 #:** 00777-10
 ■ **Host Material:** 5-chloro-8-methoxy-2-(bromomethyl)-quinoline
 ■ **Technique:** XPS
 ■ **Spectral Region:** Cl 2s

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 #:** 00777-11
 ■ **Host Material:** 5-chloro-8-methoxy-2-(bromomethyl)-quinoline
 ■ **Technique:** XPS
 ■ **Spectral Region:** Cl 2s

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