# Analysis of Sawdust by X-ray Photoelectron Spectroscopy

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Determining the chemical structure and composition of biomass fuels using x-ray photoelectron spectroscopy (XPS) can provide fundamental knowledge of their structures that is useful in understanding and predicting their combustion behavior. Sawdust is an example of a forest product residue (byproduct of paper and lumber production) of potential interest for biomass combustion. The XPS spectra of sawdust provide both its elemental composition and indications of its bonding. Traditional fuel analyses of this fuel are also provided. These include: ultimate analysis — the elemental composition of the overall fuel (C, H, N, S, and O); chlorine analysis — reported here as part of the ultimate analysis but formally a separate procedure; proximate analysis — the proximate composition of the fuel (moisture, fixed carbon, volatiles, and ash); heating value — the specific heat of combustion; ash chemistry analysis — an elemental analysis of the ash content, expressed as oxides (which does not imply that they occur as oxides in the fuel). These data are summarized with the XPS spectra. © 2005 American Vacuum Society. [DOI: 10.1116/11.20040806]

| | |
|---|---|
| **Accession #** | 00900 |
| **Technique:** | XPS |
| **Host Material:** | sawdust |
| **Instrument:** | Surface Science Instruments SSX-100 |
| **Major Elements in Spectrum:** | C, O |
| **Minor Elements in Spectrum:** | N, Si |
| **Printed Spectra:** | 8 |
| **Spectra in Electronic Record:** | 8 |
| **Spectral Category:** | technical |

## SPECIMEN DESCRIPTION

**Host Material:** sawdust

**Host Material Characteristics:** homogeneous; amorphous; unknown electrical characteristics; biological material; powder

**Chemical Name:** cellulose

**Host Composition:** see entry for History & Significance

**Form:** powder

**History & Significance:** Sawdust is an example of a forest product residue (byproduct of paper and lumber production) of potential interest for biomass combustion. Agricultural residues studies here came from 2002 harvests of wood and other agricultural materials in the U.S. and in Europe. The wood came from a commercial saw mill in the western U.S. It is of fairly typical composition of waste materials produced in sawmills. All materials underwent extensive homogenization and particle size classification to produce suitable feed materials for combustion tests conducted in the U.S. Some of the materials were prepared by us and others by collaborators in the U.S. and in Europe.

The XPS spectra of sawdust provide both its elemental composition and indications of its bonding. Traditional fuel analyses of this fuel are also provided. These include: ultimate analysis — the elemental composition of the overall fuel (C, H, N, S, and O); chlorine analysis — reported here as part of the ultimate analysis but formally a separate procedure; proximate analysis — the proximate composition of the fuel (moisture, fixed carbon, volatiles, and ash); heating value — the specific heat of combustion; ash chemistry analysis — an elemental analysis of the ash content, expressed as oxides (which does not imply they occur as oxides in the fuel). These data are summarized with the XPS spectra.

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**Table 1: Fuel analysis of bio-fuel sunflower shells (ash free basis except for ash and LHV, which are on an as-received basis).**

| | % by weight |
|---|---|
| Moisture | 9.84 |
| C | 46.40 |
| H | 5.85 |
| O | 34.33 |
| N | 0.88 |
| S | 0.18 |
| Ash | 2.52 |
| Sum | 100.0 |
| LHV*, MJ/kg | 18.816 |

*Lower heating value

**Table 2: Ash composition of sunflower shells (percent of ash basis).**

| | Mass % |
|---|---|
| $SiO_2$ | 1.1 |
| $Al_2O_3$ | 0.5 |
| $Fe_2O_3$ | 0.9 |
| CaO | 16 |
| MgO | 13.1 |
| $Na_2O$ | <0.2 |
| $K_2O$ | 45.1 |
| $SO_3$ | 11.7 |
| $P_2O_5$ | 10.1 |
| Cl | 1.2 |
| Other | 0.3 |
| Sum | 100 |

**As Received Condition:** powder

**Analyzed Region:** same as host material

***Ex Situ* Preparation/Mounting:** Sawdust powders were used as received. The powders were pressed onto a piece of nonconductive double-sticky tape mounted on a piece of silicon, which was then mounted on the sample stage with a piece of the same tape.

***In Situ* Preparation:** none

**Pre-Analysis Beam Exposure:** No damage was observed in the sample even after several hours of exposure to x-ray radiation. After 4 h of exposure to x rays, the intensity of the N $1s$ scan did not change.

**Charge Control:** A flood gun was applied. The flood gun voltage was 4 V, and its current was less than 50 mA. A metal screen was used to mask the sample. The charge control was determined by observing zirconia Zr $3p_{3/2}$ peak positions under different flood gun settings. XPS spectra showed a Zr $3d_{5/2}$ at 182.3 eV. The metal screen used was nickel, 1 mm distance, 70 lines/in. and 90% transmission.

**Temp. During Analysis:** 298 K

**Pressure During Analysis:** $<2.0 \times 10^{-6}$ Pa

## INSTRUMENT DESCRIPTION

**Manufacturer and Model:** Surface Science Instruments SSX-100

**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 $\mu$m aluminum foil

**Excitation Source:** Al $K_\alpha$ monochromatic

**Source Energy:** 1486.6 eV

**Source Strength:** 200 W

**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

**Peak Shape and Background Method:** background Shirley function

**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.

## ACKNOWLEDGMENTS

## SPECTRAL FEATURES TABLE

| Spectrum ID # | Element/ Transition | Peak Energy (eV) | Peak Width FWHM (eV) | Peak Area (counts) | Sensitivity Factor | Concen- tration (at. %) | Peak Assignment |
|---|---|---|---|---|---|---|---|
| 00900-02 | O 1s | 528.2 | 3.9 | 88500 | 2.5 | 21.3 | ⋯ |
| 00900-03 | C 1s | 280.9 | 4.3 | 126000 | 1 | 76 | ⋯ |
| 00900-04 | N 1s | 396.7 | 5.2 | 16900 | 1.68 | 0.6 | ⋯ |
| 00900-05 | Si 2p | 99.3 | 4.8 | 12200 | 0.9 | 2 | ⋯ |
| 00900-06 | O 1s | 528.3 | 3.5 | 24000 | 2.5 | ⋯ | ⋯ |
| 00900-07 | C 1s | 281.0 | 4.1 | 32100 | 1 | ⋯ | ⋯ |
| 00900-08 | N 1s | 396.7 | 4.8 | 20300 | 1.68 | ⋯ | ⋯ |

## GUIDE TO FIGURES

| Spectrum (Accession) # | Spectral Region | Voltage Shift* | Multiplier | Baseline | Comment # |
|---|---|---|---|---|---|
| **900-1** | Survey | 0 | 1 | 0 | 1 |
| **900-2** | O 1s | 0 | 1 | 0 | 1 |
| **900-3** | C 1s | 0 | 1 | 0 | 1 |
| **900-4** | N 1s | 0 | 1 | 0 | 1 |
| **900-5** | Si 2p | 0 | 1 | 0 | 1 |
| **900-6** | O 1s | 0 | 1 | 0 | 2 |
| **900-7** | C 1s | 0 | 1 | 0 | 2 |
| **900-8** | N 1s | 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. 800 $\mu$m x-ray beam diameter, 150 eV pass energy
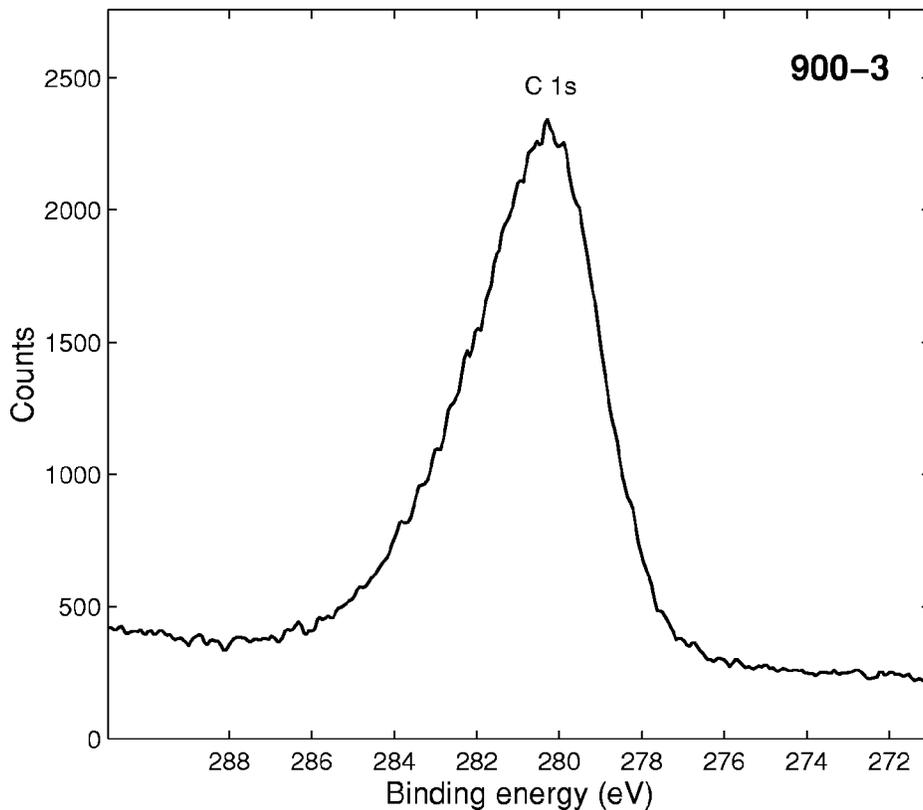2. 300 $\mu$m x-ray beam diameter, 50 eV pass energy

**900-1**
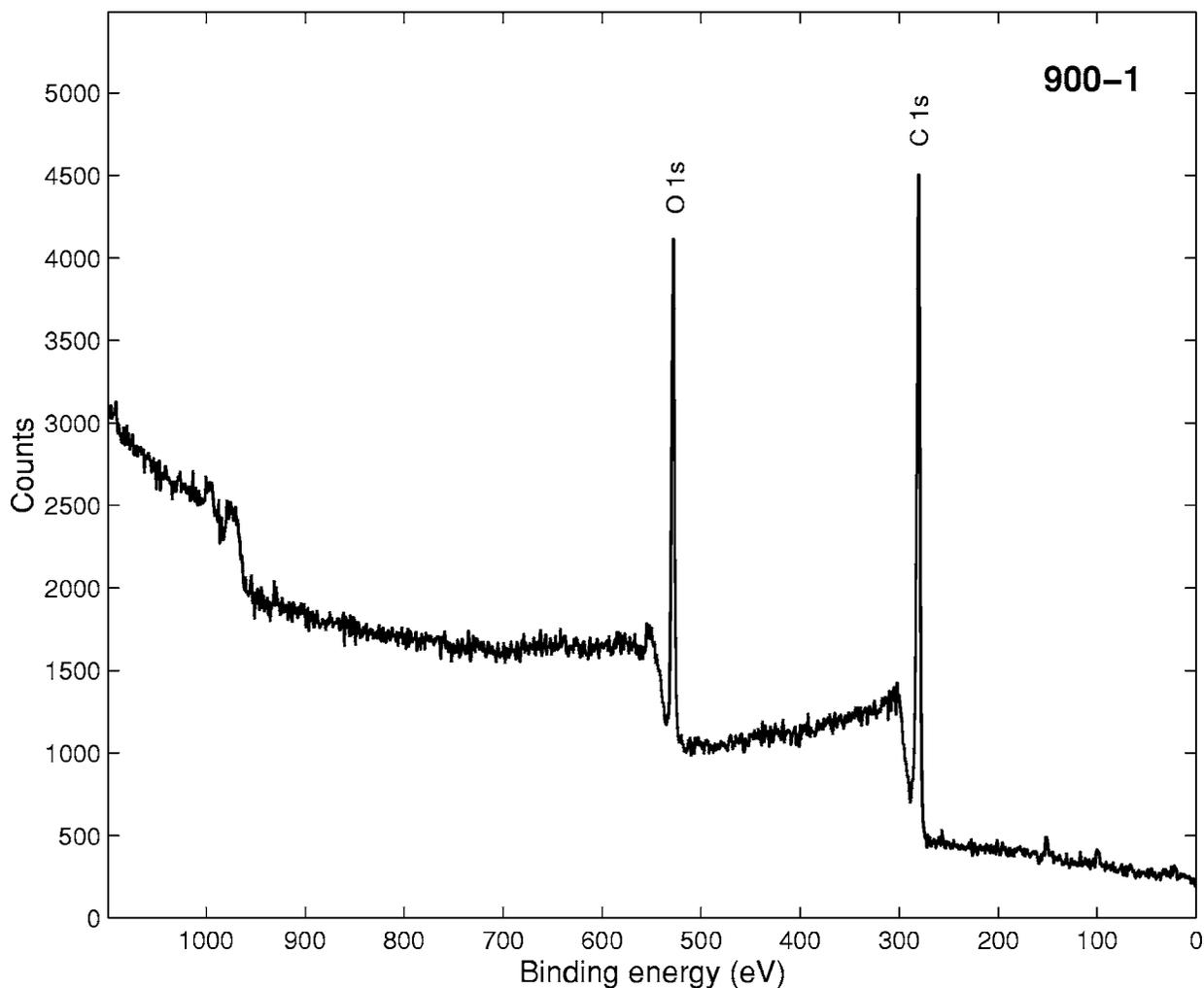
| Accession # | 00900-01 |
|---|---|
| Host Material | sawdust |
| Technique | XPS |
| Spectral Region | survey |
| Instrument | Surface Science Instruments SSX-100 |
| Excitation Source | Al $K_\alpha$ monochromatic |
| Source Energy | 1486.6 eV |
| Source Strength | 200 W |
| Source Size | 0.8 mm $\times$ 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 | 2200 s |
| Total Elapsed Time | 2400 s |
| Number of Scans | 10 |
| Source Beam Size at Specimen Surface | 0.8 mm $\times$ 1.392 mm |
| Effective Detector Width | 19 eV |
| Analyzer Width | 1500 $\mu$m $\times$ 12000 $\mu$m at 84 eV |

**900-2**

O 1s

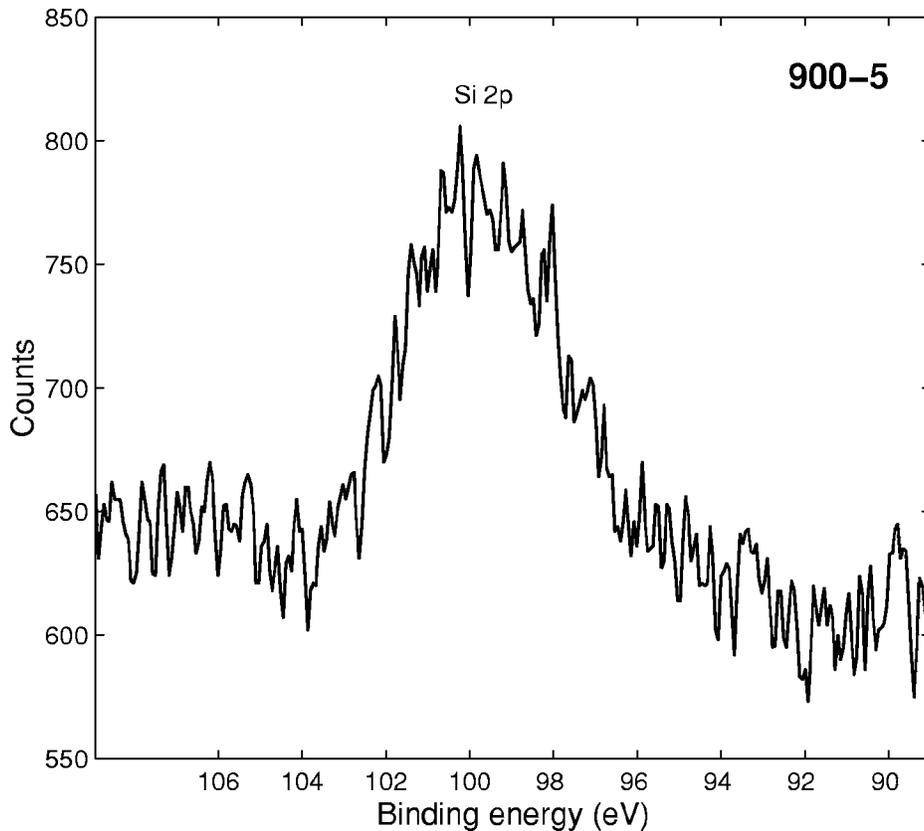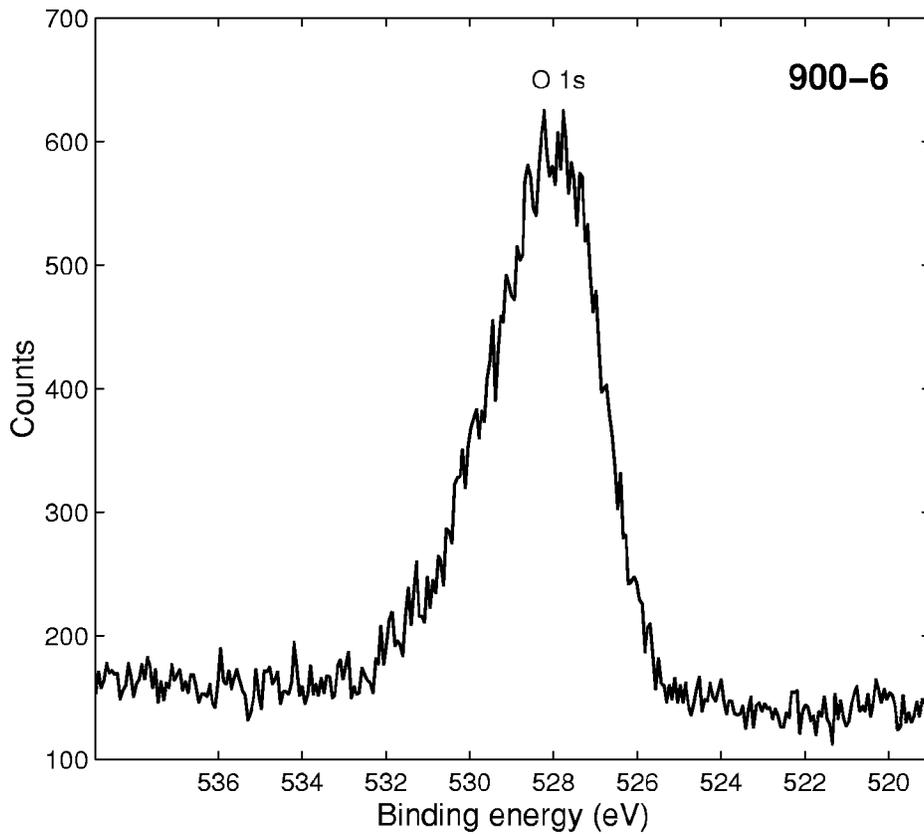Counts vs Binding energy (eV)
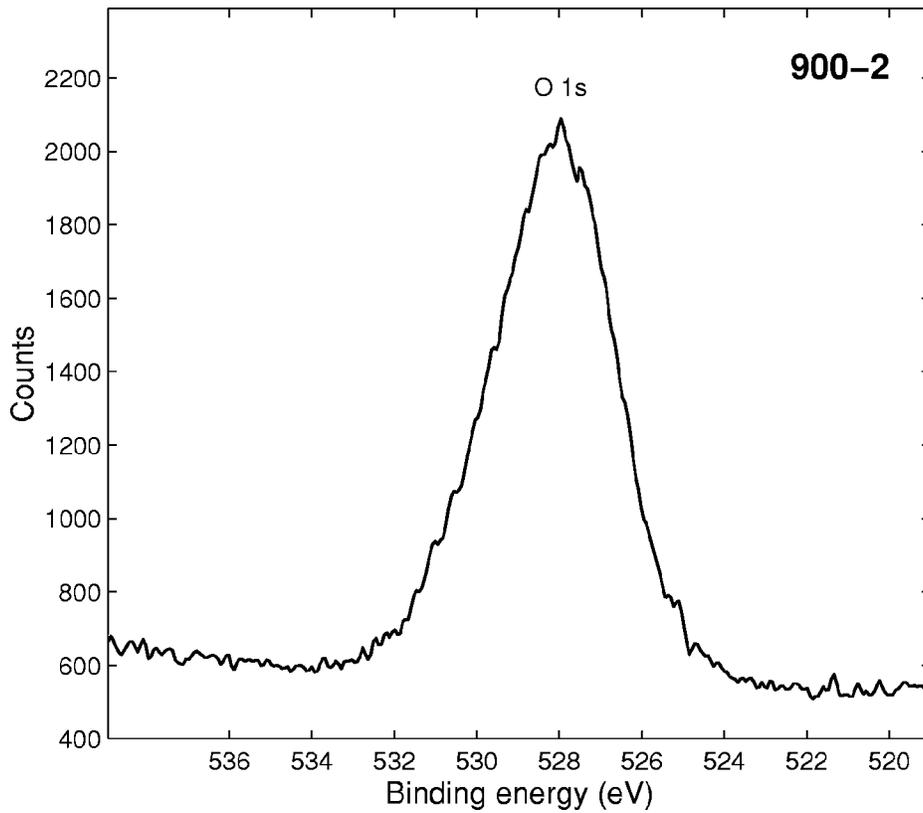
■ **Accession #:** 00900-02
■ **Host Material:** sawdust
■ **Technique:** XPS
■ **Spectral Region:** O 1$s$

Instrument: Surface Science
  Instruments SSX-100
Excitation Source: Al $K_\alpha$
  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:
  306.5 s
Total Elapsed Time: 475.5 s
Number of Scans: 5
Source Beam Size at Specimen
  Surface: 0.8 mm × 1.392 mm
Effective Detector Width: 19 eV
Analyzer Width: 1500 $\mu$m ×
  12000 $\mu$m at 84 eV



**900-3**

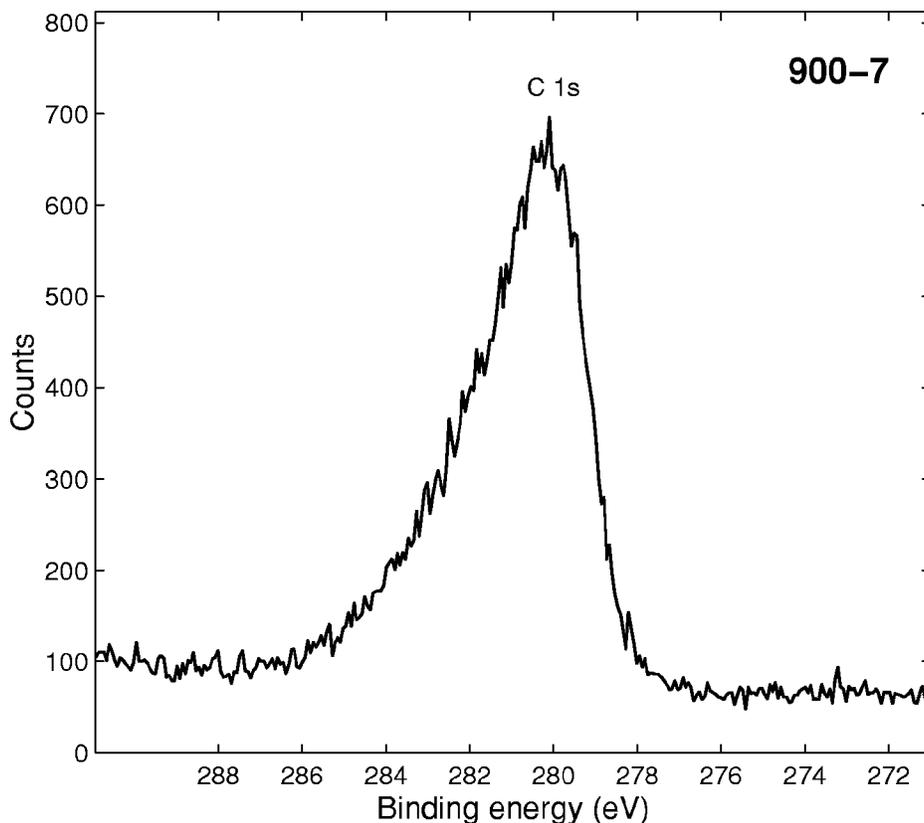C 1s

Counts vs Binding energy (eV)

■ **Accession #:** 00900-03
■ **Host Material:** sawdust
■ **Technique:** XPS
■ **Spectral Region:** C 1$s$

Instrument: Surface Science
  Instruments SSX-100
Excitation Source: Al $K_\alpha$
  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:
  306.5 s
Total Elapsed Time: 475.5 s
Number of Scans: 5
Source Beam Size at Specimen
  Surface: 0.8 mm × 1.392 mm
Effective Detector Width: 19 eV
Analyzer Width: 1500 $\mu$m ×
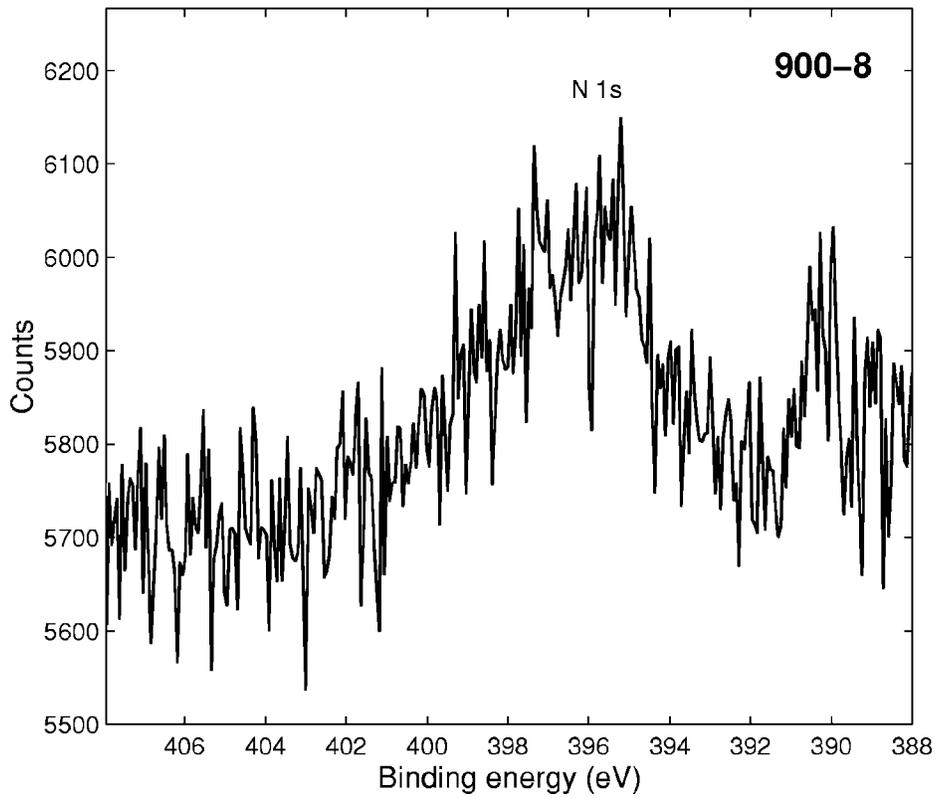  12000 $\mu$m at 84 eV

**900-4**

N 1s

**900-5**

Si 2p

900–6

O 1s

**Accession #:** 00900-06
**Host Material:** sawdust
**Technique:** XPS
**Spectral Region:** O 1$s$

Instrument: Surface Science Instruments SSX-100
Excitation Source: Al $K_\alpha$ monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.3 mm × 0.3 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: 613 s
Total Elapsed Time: 782 s
Number of Scans: 10
Source Beam Size at Specimen Surface: 0.3 mm × 0.523 mm
Effective Detector Width: 6.6 eV
Analyzer Width: 750 $\mu$m × 6000 $\mu$m at 84 eV



900–7

C 1s

**Accession #:** 00900-07
**Host Material:** sawdust
**Technique:** XPS
**Spectral Region:** C 1$s$

Instrument: Surface Science Instruments SSX-100
Excitation Source: Al $K_\alpha$ monochromatic
Source Energy: 1486.6 eV
Source Strength: 200 W
Source Size: 0.3 mm × 0.3 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: 613 s
Total Elapsed Time: 782 s
Number of Scans: 10
Source Beam Size at Specimen Surface: 0.3 mm × 0.523 mm
Effective Detector Width: 6.6 eV
Analyzer Width: 750 $\mu$m × 6000 $\mu$m at 84 eV

**900–8**

N 1s



- ■ **Accession #:** 00900-08
- ■ **Host Material:** sawdust
- ■ **Technique:** XPS
- ■ **Spectral Region:** N 1$s$

Instrument: Surface Science Instruments SSX-100

Excitation Source: Al $K_{\alpha}$ monochromatic

Source Energy: 1486.6 eV

Source Strength: 200 W

Source Size: 0.3 mm × 0.3 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: 24520 s

Total Elapsed Time: 24689 s

Number of Scans: 400

Source Beam Size at Specimen Surface: 0.3 mm × 0.523 mm

Effective Detector Width: 6.6 eV

Analyzer Width: 750 $\mu$m × 6000 $\mu$m at 84 eV