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**Authors**

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# Analysis of Sugar Beet Pulp 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. Sugar beet pulp is an example of an agricultural residue (byproduct of food and feed production) of potential interest for biomass combustion. The XPS spectra of sugar beet pulp 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. These data are summarized with the XPS spectra. © 2005 American Vacuum Society. [DOI: 10.1116/11.20040803]

**Keywords:** biomass; sugar beet pulp; XPS; fuel

**PACS:** 82.80.Pv, 01.30.Kj, 84.60.Rb, 82.33.Vx, 82.60.Cx

## SPECIMEN DESCRIPTION

**Host Material:** sugar beet pulp

**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:** Sugar beet pulp is an example of an agricultural residue (byproduct of food and feed production) of potential interest for biomass combustion. The XPS spectra of sugar beet pulp 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. These data are summarized with the XPS spectra. The chemical composition of sugar beet pulp is summarized in Table 1.

**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.

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Accession # 00897

Technique: XPS

Host Material: sugar beet pulp

Instrument: Surface Science Instruments SSX-100

Major Elements in Spectrum: C, O

Minor Elements in Spectrum: N

Printed Spectra: 7

Spectra in Electronic Record: 7

Spectral Category: technical

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<sub>3/2</sub> peak positions under different flood gun settings. XPS spectra showed a Zr 3d<sub>5/2</sub> 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:** <2.0×10<sup>-6</sup> 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

**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

## **INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA**

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### ■ **Spectrometer**

**Analyzer Mode:** constant pass energy

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

**Excitation Source Window:** 12  $\mu\text{m}$  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**

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

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The authors acknowledge U.S. DOE Biomass Power Program for financial support, and Elsam engineering and Eltra, both Danish companies, which provided complementary analyses and some financial support for this investigation.

**SPECTRAL FEATURES TABLE**

<b>Spectrum ID #</b>	<b>Element/ Transition</b>	<b>Peak Energy (eV)</b>	<b>Peak Width FWHM (eV)</b>	<b>Peak Area (counts)</b>	<b>Sensitivity Factor</b>	<b>Concentration (at. %)</b>	<b>Peak Assignment</b>
00897-02	O 1s	527.8	3.0	138000	2.5	23.6	...
00897-03	C 1s	281.0	3.9	175000	1	74.6	...
00897-04	N 1s	395.6	3.9	71800	1.68	1.8	...
00897-05	O 1s	527.8	2.4	50700	2.5	...	...
00897-06	C 1s	280.1	2.7	60200	1	...	...
00897-07	N 1s	395.1	1.6	92200	1.68	...	...

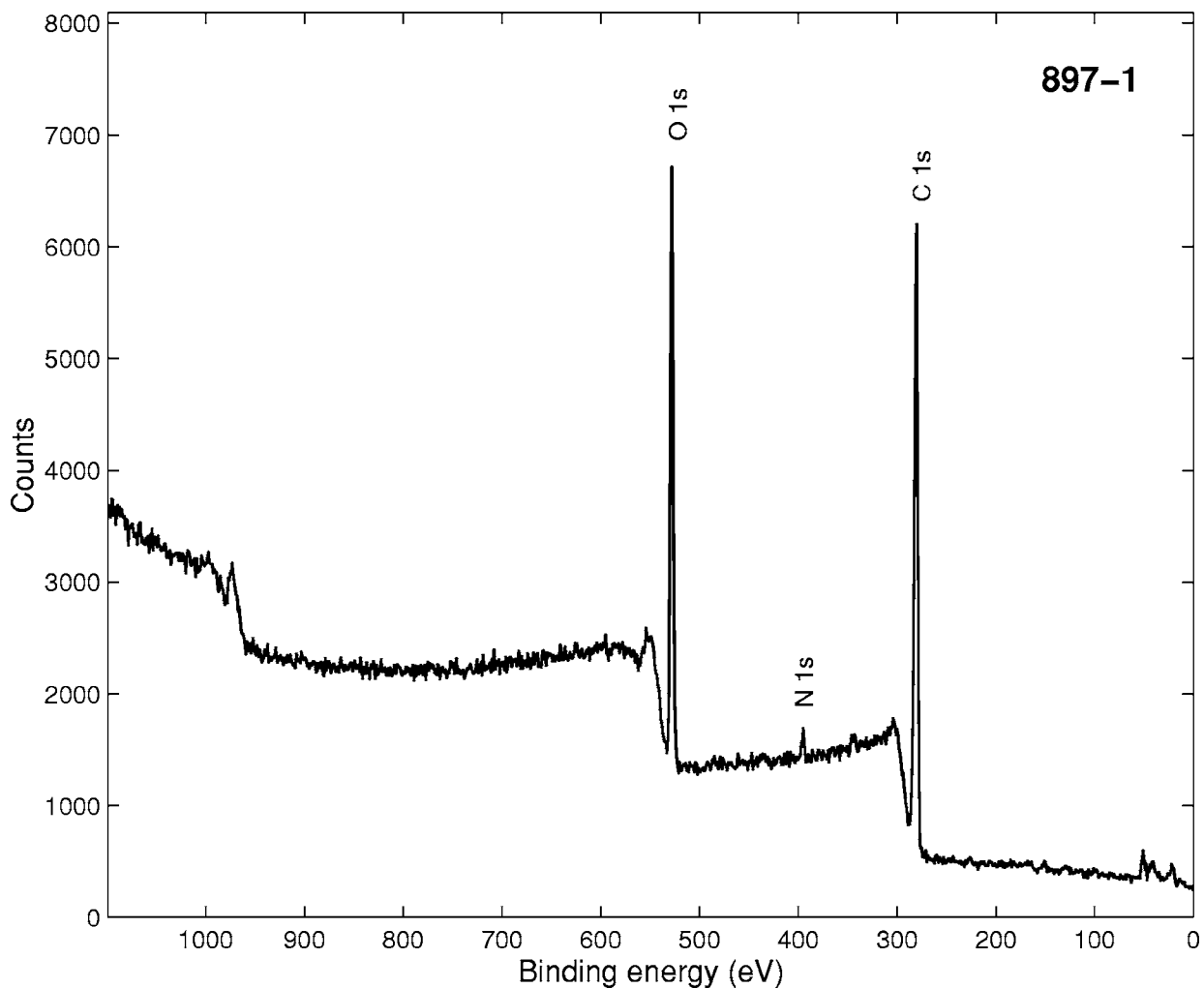
**GUIDE TO FIGURES**

<b>Spectrum (Accession) #</b>	<b>Spectral Region</b>	<b>Voltage Shift*</b>	<b>Multiplier</b>	<b>Baseline</b>	<b>Comment #</b>
<b>897-1</b>	Survey	0	1	0	1
<b>897-2</b>	O 1s	0	1	0	1
<b>897-3</b>	C 1s	0	1	0	1
<b>897-4</b>	N 1s	0	1	0	1
<b>897-5</b>	O 1s	0	1	0	2
<b>897-6</b>	C 1s	0	1	0	2
<b>897-7</b>	N 1s	0	1	0	2

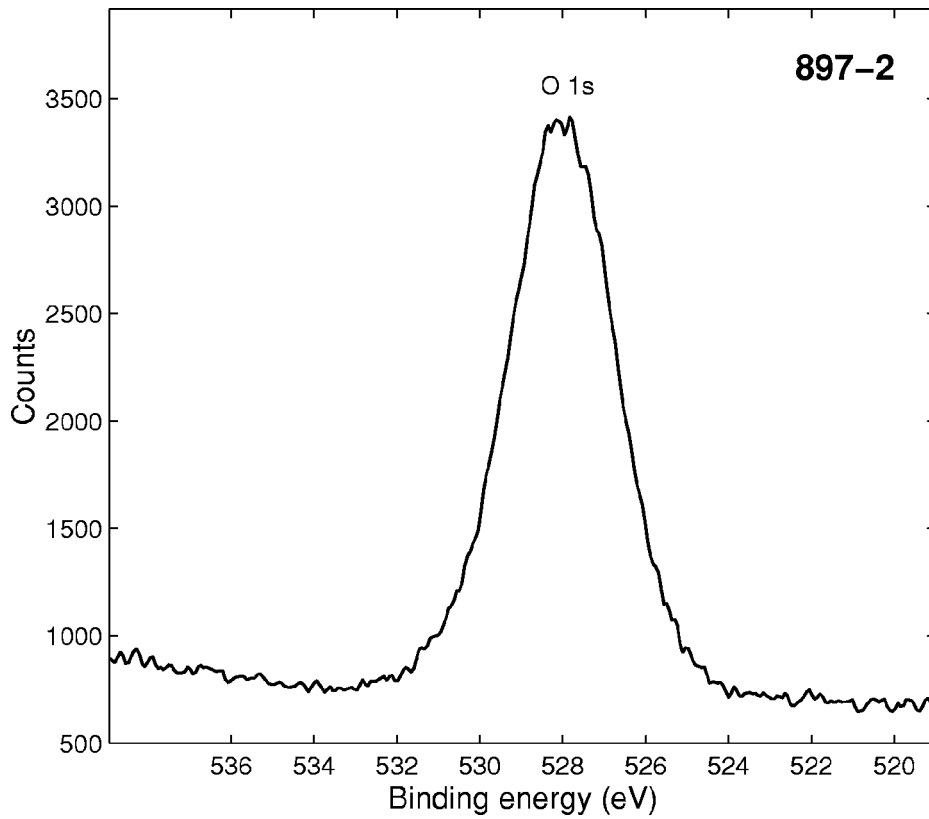
\* Voltage shift of the archived (as-measured) spectrum relative to the printed figure. The figure reflects the recommended energy scale correction due to a calibration correction, sample charging, flood gun, or other phenomenon.

1. 800  $\mu\text{m}$  x-ray beam diameter, 150 eV pass energy

2. 300  $\mu\text{m}$  x-ray beam diameter, 50 eV pass energy

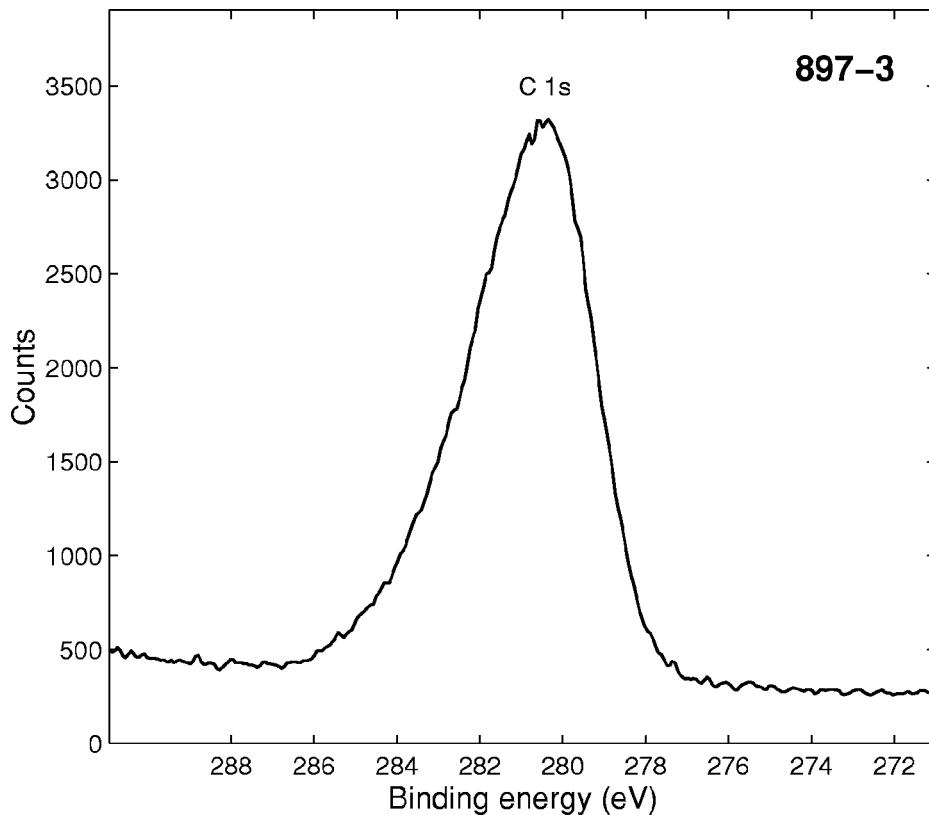


<b>Accession #</b>	<b>00897-01</b>
<b>Host Material</b>	sugar beet pulp
<b>Technique</b>	XPS
<b>Spectral Region</b>	survey
<b>Instrument</b>	Surface Science Instruments SSX-100
<b>Excitation Source</b>	Al $K_{\alpha}$ monochromatic
<b>Source Energy</b>	1486.6 eV
<b>Source Strength</b>	200 W
<b>Source Size</b>	0.8 mm $\times$ 0.8 mm
<b>Analyzer Type</b>	spherical sector
<b>Incident Angle</b>	55°
<b>Emission Angle</b>	55°
<b>Analyzer Pass Energy</b>	150 eV
<b>Analyzer Resolution</b>	1.5 eV
<b>Total Signal Accumulation Time</b>	2200 s
<b>Total Elapsed Time</b>	2400 s
<b>Number of Scans</b>	10
<b>Source Beam Size at Specimen Surface</b>	0.8 mm $\times$ 1.392 mm
<b>Effective Detector Width</b>	19 eV
<b>Analyzer Width</b>	1500 $\mu$ m $\times$ 12000 $\mu$ m at 84 eV



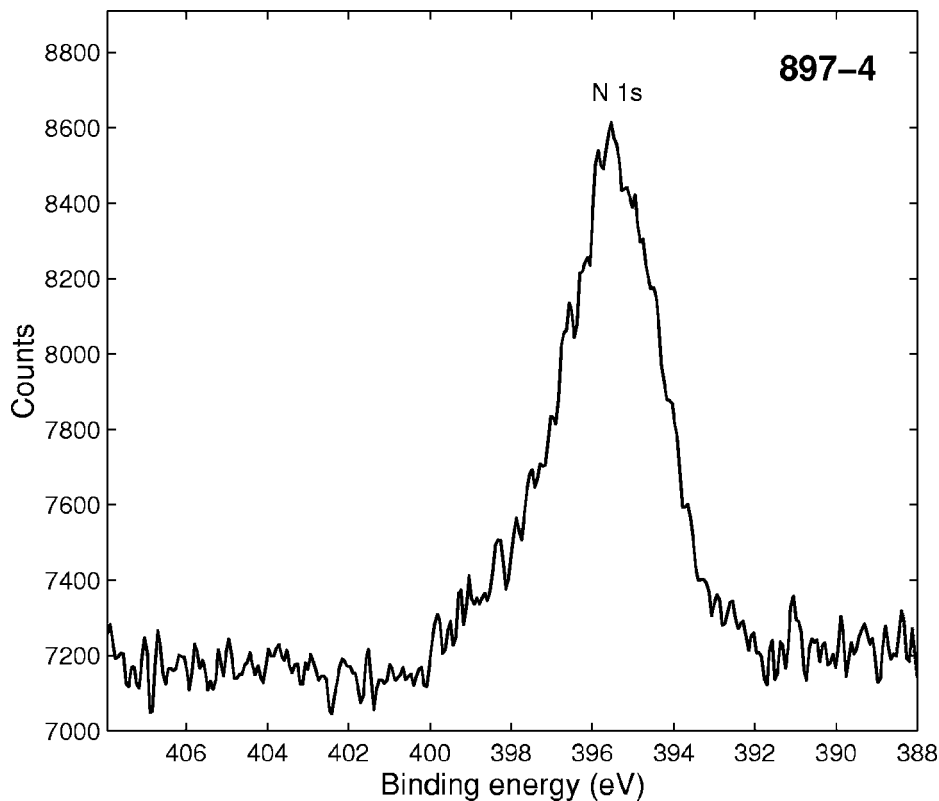
■ **Accession #:** 00897-02  
 ■ **Host Material:** sugar beet pulp  
 ■ **Technique:** XPS  
 ■ **Spectral Region:** O 1s

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  
 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  $\times$  1.392 mm  
 Effective Detector Width: 19 eV  
 Analyzer Width: 1500  $\mu\text{m}$   $\times$  12000  $\mu\text{m}$  at 84 eV



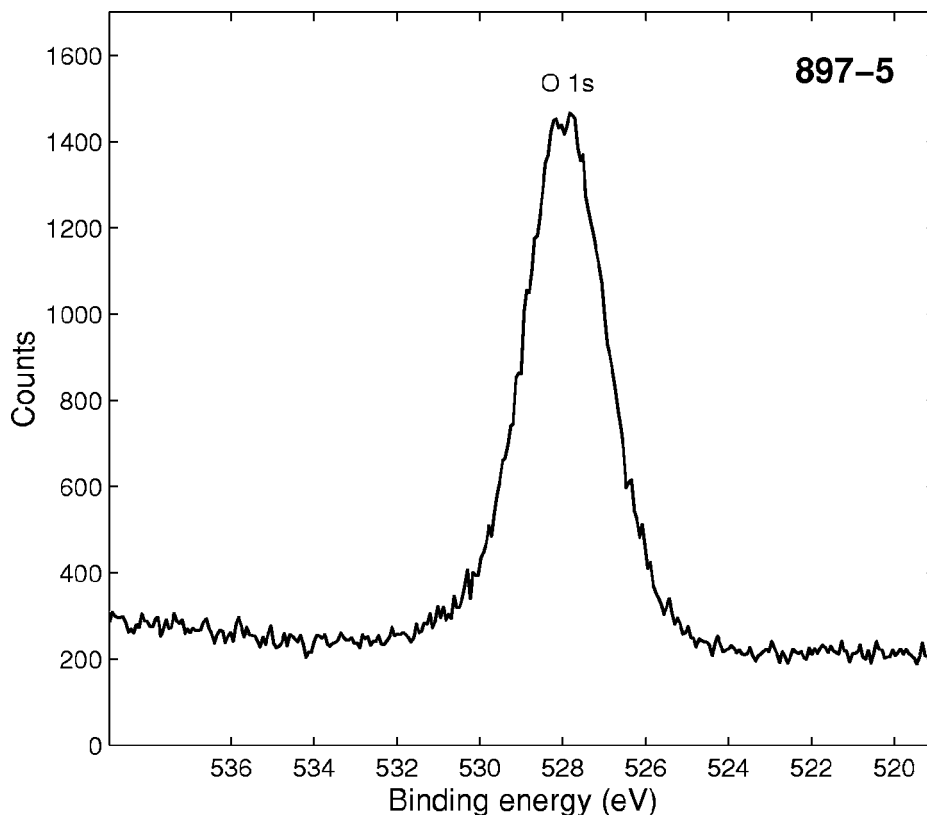
■ **Accession #:** 00897-03  
 ■ **Host Material:** sugar beet pulp  
 ■ **Technique:** XPS  
 ■ **Spectral Region:** C 1s

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  
 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  $\times$  1.392 mm  
 Effective Detector Width: 19 eV  
 Analyzer Width: 1500  $\mu\text{m}$   $\times$  12000  $\mu\text{m}$  at 84 eV



■ **Accession #:** 00897-04  
 ■ **Host Material:** sugar beet pulp  
 ■ **Technique:** XPS  
 ■ **Spectral Region:** N 1s

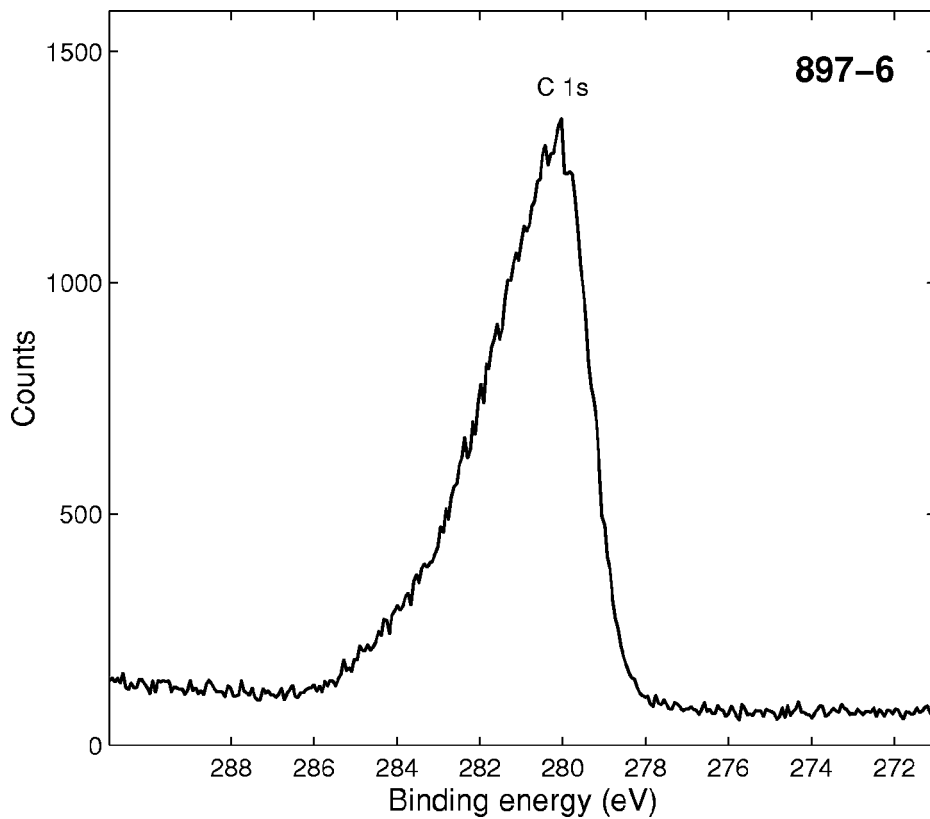
Instrument: Surface Science Instruments SSX-100  
 Excitation Source: Al  $K_{\alpha}$  monochromatic  
 Source Energy: 1486.6 eV  
 Source Strength: 200 W  
 Source Size: 0.8 mm  $\times$  0.8 mm  
 Incident Angle: 55°  
 Analyzer Type: spherical sector  
 Analyzer Pass Energy: 150 eV  
 Analyzer Resolution: 1.5 eV  
 Emission Angle: 55°  
 Total Signal Accumulation Time: 3065 s  
 Total Elapsed Time: 3234 s  
 Number of Scans: 50  
 Source Beam Size at Specimen Surface: 0.8 mm  $\times$  1.392 mm  
 Effective Detector Width: 19 eV  
 Analyzer Width: 1500  $\mu\text{m}$   $\times$  12000  $\mu\text{m}$  at 84 eV



■ **Accession #:** 00897-05  
 ■ **Host Material:** sugar beet pulp  
 ■ **Technique:** XPS  
 ■ **Spectral Region:** O 1s

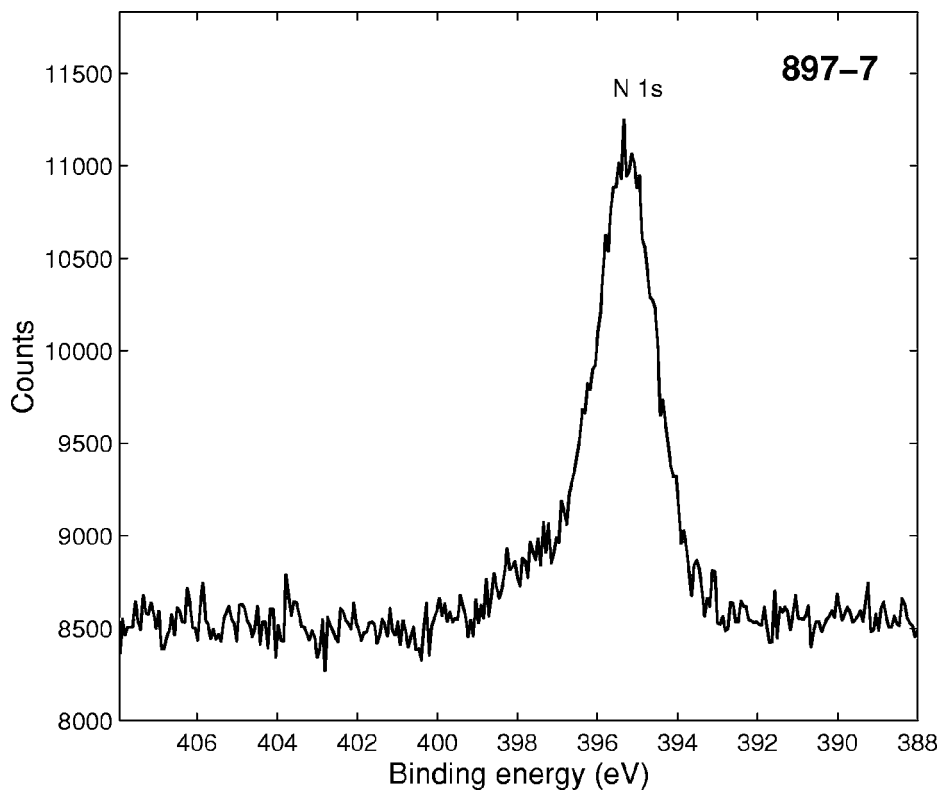
Instrument: Surface Science Instruments SSX-100  
 Excitation Source: Al  $K_{\alpha}$  monochromatic  
 Source Energy: 1486.6 eV  
 Source Strength: 200 W  
 Source Size: 0.3 mm  $\times$  0.3 mm  
 Incident Angle: 55°  
 Analyzer Type: spherical sector  
 Analyzer Pass Energy: 50 eV  
 Analyzer Resolution: 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  $\times$  0.523 mm  
 Effective Detector Width: 6.6 eV  
 Analyzer Width: 750  $\mu\text{m}$   $\times$  6000  $\mu\text{m}$  at 84 eV





■ **Accession #:** 00897-06  
 ■ **Host Material:** sugar beet pulp  
 ■ **Technique:** XPS  
 ■ **Spectral Region:** C 1s

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  $\times$  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  $\times$  0.523 mm  
 Effective Detector Width: 6.6 eV  
 Analyzer Width: 750  $\mu$ m  $\times$  6000  $\mu$ m at 84 eV



■ **Accession #:** 00897-07  
 ■ **Host Material:** sugar beet pulp  
 ■ **Technique:** XPS  
 ■ **Spectral Region:** N 1s

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  $\times$  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  $\times$  0.523 mm  
 Effective Detector Width: 6.6 eV  
 Analyzer Width: 750  $\mu$ m  $\times$  6000  $\mu$ m at 84 eV