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Z. Paál, M. Muhler and R. Schlögl

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Platinum Black by XPS

Zoltan Paál
Institute of Isotopes, Hungarian Academy of Sciences, P.O. Box 77, Budapest, H-1525 Hungary

M. Muhler and R. Schlögl
Fritz-Haber-Institut der MPG, D-14195 Berlin Faradayweg 4-6, Germany

XPS spectra of Pt black in the as received state showed O and C impurities along with Pt. An *in situ* treatment by O_2 and H_2 increased Pt intensity and removed a part of oxygen and carbon impurities. The quasihomogeneous model was used for quantitative evaluation applying atomic sensitivity factors published in the literature (Ref. 1). Decomposition of the O 1*s* region indicated the presence of adsorbed O, OH, and H_2O as well as C—O and C═O species, whereas the C 1*s* region could be decomposed to give Pt–C, graphite, C_xH_y polymer, and oxidized C entities. © *1997 American Vacuum Society.* [S1055-5269(96)00202-2]

Keywords: *platinum; Pt black; Pt catalyst; XPS*

PACS: *82.80.Pv, 82.65.Jv*

Accession #s 00280, 00281

Technique: XPS

Host Material: #00280: Pt black; #00281: Pt black, treated

Instrument: Leybold, LHS 12 SCD EA II

Major Elements in Spectrum: Pt, O, C

Minor Elements in Spectrum: none

Printed Spectra: 7

Spectra in Electronic Record: 36

Spectral Category: technical

Original Submission: 4/05/95

Accepted for Publication: 6/19/97

SPECIMEN DESCRIPTION (Accession #00280)

Host Material: Pt black

CAS Registry #: 7440-06-4

Host Material Characteristics: homogeneous; polycrystalline; conductor; metal; powder

Chemical Name: platinum

Source: Platinum black was prepared by boiling the solution of H_2PtCl_6 with hydrazine and stored in air (Ref. 2).

Host Composition: Pt

Form: powder

Structure: fcc polycrystalline

History & Significance: Pt catalysts are extensively used for naphtha reforming (Ref. 3). Platinum black represents a polycrystalline unsupported catalyst with a relatively high surface area and it is a good model of supported metals of practical importance (Refs. 2 and 4) and its study by electron spectroscopy is not hampered by any electric insulator support. The state of Pt in the as received state and after regeneration may well simulate those present in practical platinum catalysts which thus contain oxygen and carbon impurities when they first meet the hydrocarbon reactant (Ref. 5).

As Received Condition: sample reduced from H_2PtCl_6 by hydrazine

Analyzed Region: same as host material

***Ex Situ* Preparation/Mounting:** Pt powder was dry loaded into the cavity of a stainless steel sample holder to form a flat surface.

***In Situ* Preparation:** None

Charge Control: None

Temp. During Analysis: 300 K

Pressure During Analysis: $<1\times10^{-7}$ Pa

SPECIMEN DESCRIPTION (Accession #00281)

Host Material: Pt black, treated

CAS Registry #: 7440-06-4

Host Material Characteristics: homogeneous; polycrystalline; conductor; metal; powder

Chemical Name: platinum

Source: Platinum black was prepared by boiling the solution of H_2PtCl_6 with hydrazine and stored in air (Ref. 2).

Host Composition: Pt

Form: powder

Structure: fcc polycrystalline

History & Significance: Pt catalysts are extensively used for naphtha reforming (Ref. 3). Platinum black represents a polycrystalline unsupported catalyst with a relatively high surface area and it is a good model of supported metals of practical importance (Refs. 2 and 4) and its study by electron spectroscopy is not hampered by any electric insulator support. The state of Pt in the as received state and after regeneration may well simulate those present in practical platinum catalysts which thus contain oxygen and carbon impurities when they first meet the hydrocarbon reactant (Ref. 5).

As Received Condition: sample reduced from H_2PtCl_6 by hydrazine

Analyzed Region: same as host material

***Ex Situ* Preparation/Mounting:** Pt powder was dry loaded into the cavity of a stainless steel sample holder to form a flat surface.

***In Situ* Preparation:** 20 kPa O_2 for 3 min, evacuation for 5 min, and 200 kPa H_2 for 10 min at 603 K (regenerated sample)

Pre-Analysis Beam Exposure: A survey spectrum of the as received sample (Accession #00280) was taken first, without pre-analysis beam exposure, followed by high-resolution region spectra. Then the regeneration was performed (see *In Situ* Preparation) prior to survey and high-resolution region spectra for the regenerated sample.

Charge Control: None

Temp. During Analysis: 300 K

Pressure During Analysis: $<1\times10^{-7}$ Pa

SPECTROMETER DESCRIPTION

Manufacturer and Model: Leybold, LHS 12 SCD EA II

Analyzer Type: spherical sector

Detector: dynode multiplier

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

■ Spectrometer

Analyzer Mode: constant pass energy

Throughput ($T = E^N$): $N = -1$

Excitation Source: Mg K_α

Excitation Source Window: 2 μm Al

Source Energy: 1253.6 eV

Source Strength: 240 W

■ Geometry

Incident Angle: 75°

Source to Analyzer Angle: 75°

Emission Angle: 0°

Specimen Azimuthal Angle: 0°

■ Ion Gun

Manufacturer and Model: Leybold IQE 10135

DATA ANALYSIS METHOD

Peak Shape and Background Method: SCIPLOT software (shareware, Version 4.01, Copyright M. Wesemann, Berlin, Fritz-Haber-Institut) was used for data processing, including x-ray satellite subtraction, and Shirley background subtraction and integration. Line decomposition of O 1*s* and C 1*s* regions was done by using the mixture of Gaussian and Lorentzian curves (G/L ratio 0.5 for O 1*s*, and 0.4 for C 1*s*).

Quantitation Method: Atomic sensitivity factors used are listed in Ref. 1.

REFERENCES

1. D. Briggs and M. P. Seah, in *Practical Surface Analysis*, 2nd ed. (Wiley, Chichester, 1990), Vol. 1, Appendix 6, p. 635.
2. Z. Paál, Z. Zhan, E. Fülöp, and B. Tesche, J. Catal. **156**, 19 (1995).
3. Z. Paál, in *Catalytic Naphtha Reforming*, edited by G. M. Antos, A. M. Aitani, and J. M. Parera (Marcel Dekker, New York, 1995), p. 19.
4. Z. Paál, R. Schlögl, and G. Ertl, J. Chem. Soc. Faraday Trans. **88**, 1179 (1992).
5. Z. Paál and R. Schlögl, Surf. Interf. Anal. **19**, 524 (1992).
6. Z. Paál and Z. Zhan, Langmuir **13**, 3752 (1997).

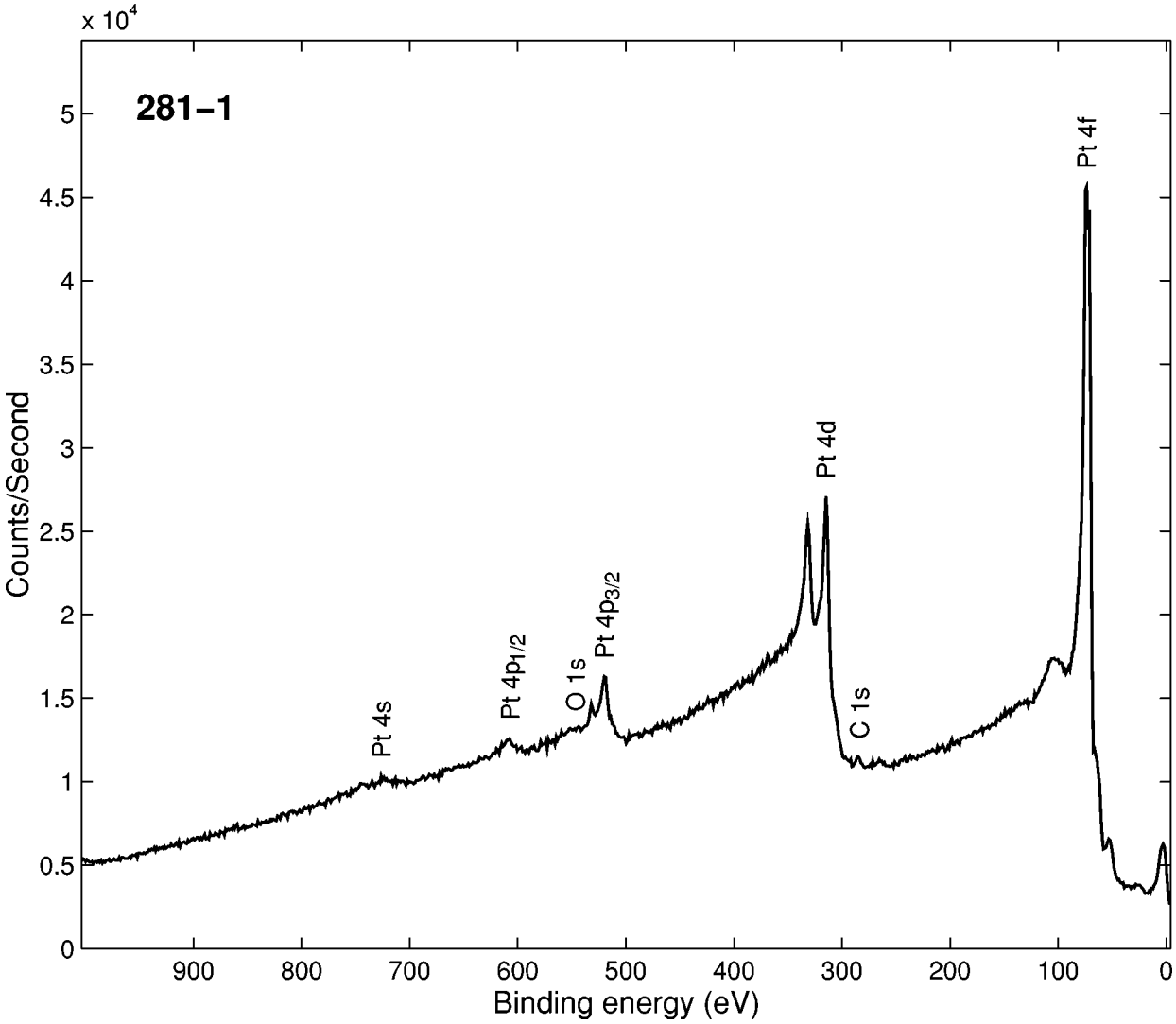
SPECTRAL FEATURES TABLE

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
00280-01	O 1*s*	531	2.7	250	0.66	30.1	···
00280-02	C 1*s*	285	2.5	100	0.25	27.8	···
00280-03	Pt $4f_{7/2}$	71.15	1.6	1700	4.40	42.1	···

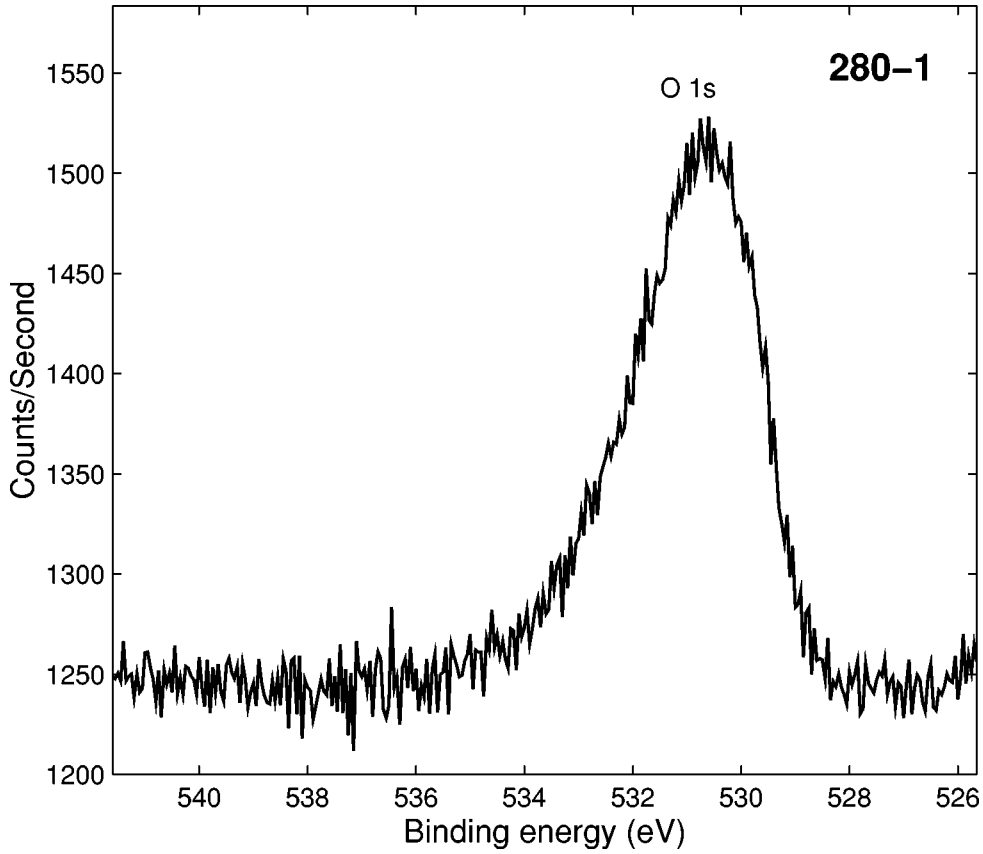
ANALYZER CALIBRATION TABLE

Spectrum ID #	Element/ Transition	Peak Energy[a] (eV)	Peak Width FWHM (eV)	Peak Area (cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
···	Pt E_F	0.0	···	···	···	···	···
···	Au $4f_{7/2}$	84.0	1.3	···	···	···	···

[a] Energy calibration only.



Accession #	00281-01
Host Material	Pt black, treated
Technique	XPS
Spectral Region	survey
Instrument	Leybold, LHS 12 SCD EA II
Excitation Source	Mg K_α
Source Energy	1253.6 eV
Source Strength	240 W
Source Size	not specified
Analyzer Type	spherical sector
Incident Angle	75°
Emission Angle	0°
Analyzer Retard Ratio	4
Analyzer Resolution	0.25%
Total Signal Accumulation Time	not specified
Total Elapsed Time	252.3 s
Number of Scans	15
Comment	Survey spectrum of sample regenerated by O_2 and subsequent H_2 treatment at 600 K



- **Accession #:** 00280-01
- **Host Material:** Pt black
- **Technique:** XPS
- **Spectral Region:** O 1*s*

Instrument: Leybold, LHS 12 SCD EA II

Excitation Source: Mg K_α

Source Energy: 1253.6 eV

Source Strength: 240 W

Source Size: not specified

Incident Angle: 75°

Analyzer Type: spherical sector

Analyzer Pass Energy: 50 eV

Analyzer Resolution: 1.0 eV

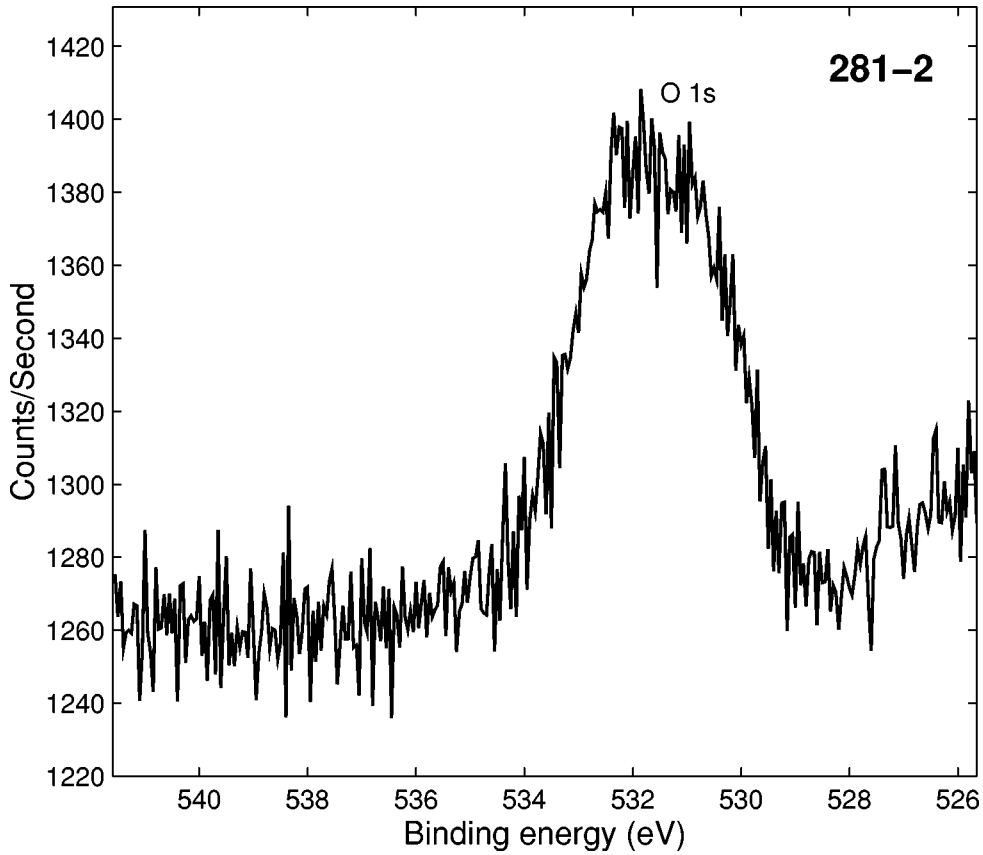
Emission Angle: 0°

Total Signal Accumulation Time: not specified

Total Elapsed Time: 6400 s

Number of Scans: 1000

Comment: O 1*s* line of the as received sample



- **Accession #:** 00281-02
- **Host Material:** Pt black, treated
- **Technique:** XPS
- **Spectral Region:** O 1*s*

Instrument: Leybold, LHS 12 SCD EA II

Excitation Source: Mg K_α

Source Energy: 1253.6 eV

Source Strength: 240 W

Source Size: not specified

Incident Angle: 75°

Analyzer Type: spherical sector

Analyzer Pass Energy: 50 eV

Analyzer Resolution: 1.0 eV

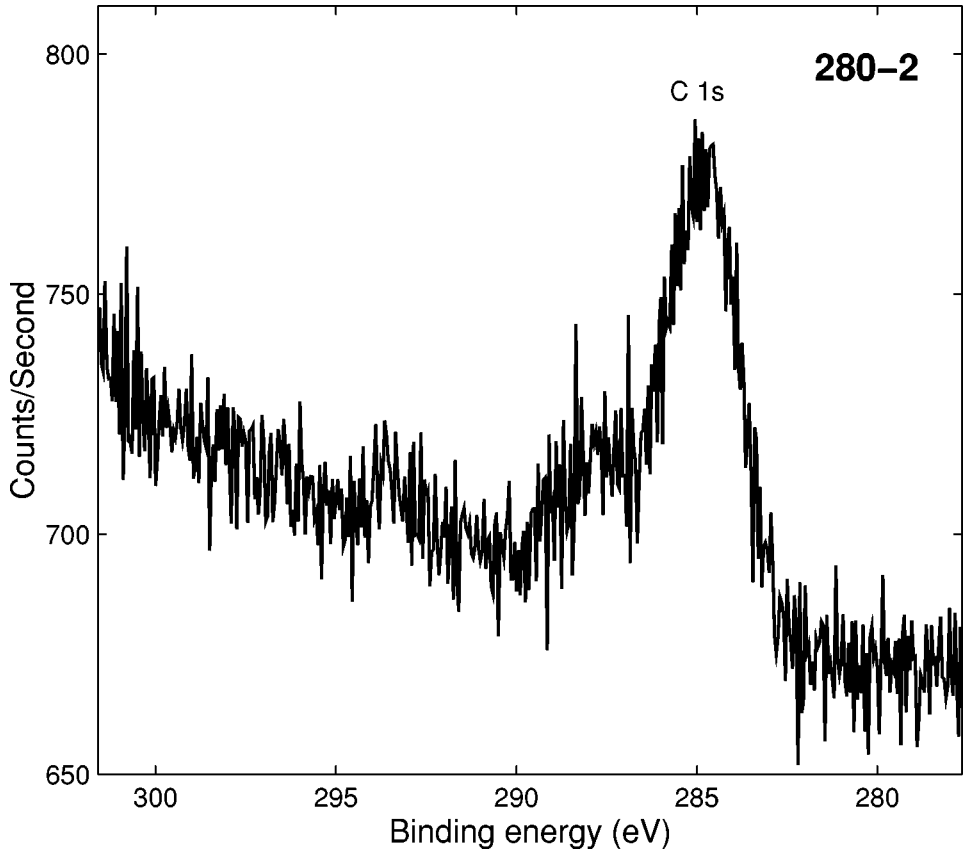
Emission Angle: 0°

Total Signal Accumulation Time: not specified

Total Elapsed Time: 6400 s

Number of Scans: 1000

Comment: O 1*s* line of the regenerated sample



- **Accession #:** 00280-02
- **Host Material:** Pt black
- **Technique:** XPS
- **Spectral Region:** C 1*s*

Instrument: Leybold, LHS 12 SCD EA II

Excitation Source: Mg K_α

Source Energy: 1253.6 eV

Source Strength: 240 W

Source Size: not specified

Incident Angle: 75°

Analyzer Type: spherical sector

Analyzer Pass Energy: 50 eV

Analyzer Resolution: 1.0 eV

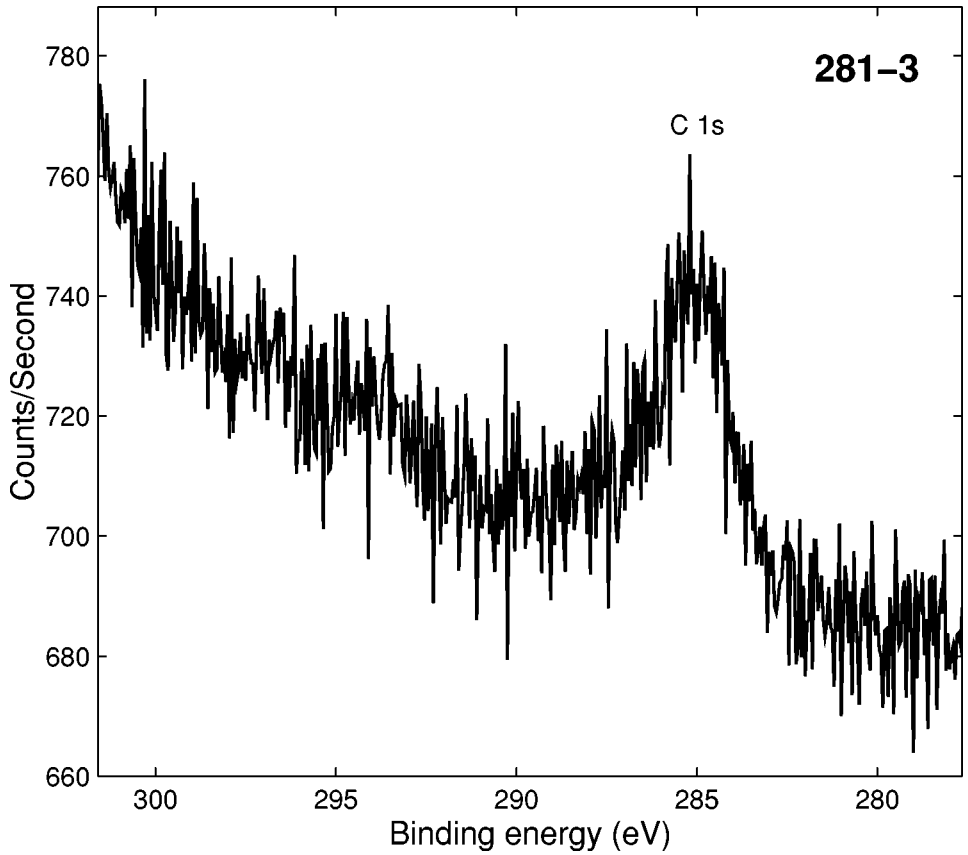
Emission Angle: 0°

Total Signal Accumulation Time: not specified

Total Elapsed Time: 9600 s

Number of Scans: 1000

Comment: C 1*s* line of the as received sample



- **Accession #:** 00281-03
- **Host Material:** Pt black, treated
- **Technique:** XPS
- **Spectral Region:** C 1*s*

Instrument: Leybold, LHS 12 SCD EA II

Excitation Source: Mg K_α

Source Energy: 1253.6 eV

Source Strength: 240 W

Source Size: not specified

Incident Angle: 75°

Analyzer Type: spherical sector

Analyzer Pass Energy: 50 eV

Analyzer Resolution: 1.0 eV

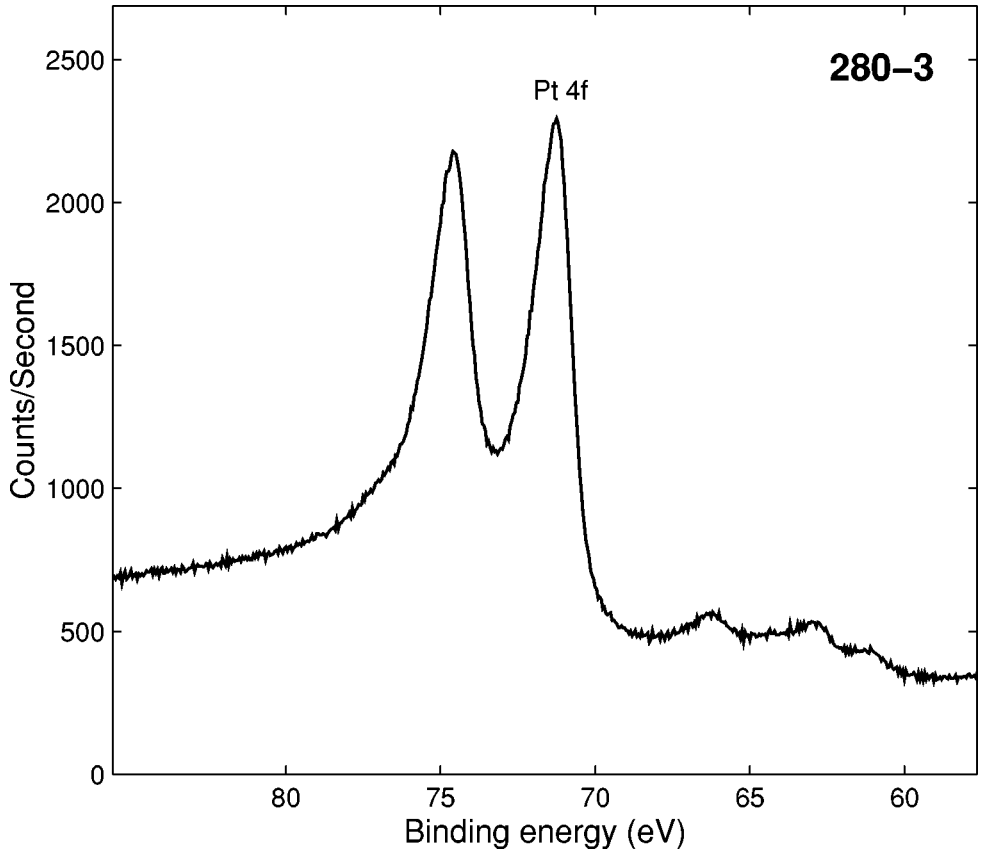
Emission Angle: 0°

Total Signal Accumulation Time: not specified

Total Elapsed Time: 9600 s

Number of Scans: 1000

Comment: C 1*s* line of the regenerated sample



- **Accession #:** 00280-03
- **Host Material:** Pt black
- **Technique:** XPS
- **Spectral Region:** Pt 4*f*

Instrument: Leybold, LHS 12 SCD EA II

Excitation Source: Mg K_α

Source Energy: 1253.6 eV

Source Strength: 240 W

Source Size: not specified

Incident Angle: 75°

Analyzer Type: spherical sector

Analyzer Pass Energy: 50 eV

Analyzer Resolution: 1.0 eV

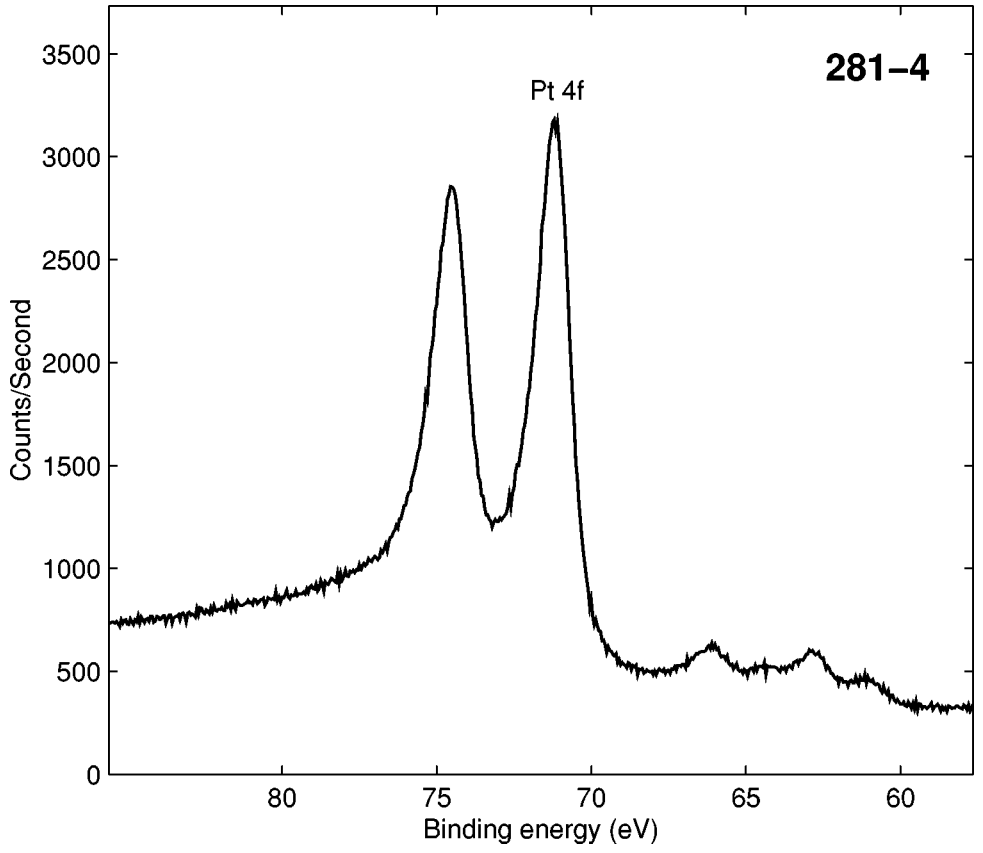
Emission Angle: 0°

Total Signal Accumulation Time: not specified

Total Elapsed Time: 11200 s

Number of Scans: 1000

Comment: Pt 4*f* line of the as received sample



- **Accession #:** 00281-04
- **Host Material:** Pt black, treated
- **Technique:** XPS
- **Spectral Region:** Pt 4*f*

Instrument: Leybold, LHS 12 SCD EA II

Excitation Source: Mg K_α

Source Energy: 1253.6 eV

Source Strength: 240 W

Source Size: not specified

Incident Angle: 75°

Analyzer Type: spherical sector

Analyzer Pass Energy: 50 eV

Analyzer Resolution: 1.0 eV

Emission Angle: 0°

Total Signal Accumulation Time: not specified

Total Elapsed Time: 4480 s

Number of Scans: 400

Comment: Pt 4*f* line of the regenerated sample