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# Core-level Spectra of Powdered Tungsten Disulfide, WS<sub>2</sub>

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(Received day Month year; accepted day Month year; published day Month year)

Core level spectra, generated by monochromatic Al K $\alpha$  x-rays for a commercial, powdered tungsten disulfide (WS<sub>2</sub>) specimen have been recorded and presented. The XPS spectra of WS<sub>2</sub> obtained include a survey scan, high-resolution spectra of W 4f, S 2p, S 2s, W 4d, W 4p, O 1s, W 4s, S LMM and the valance band. Quantitative analysis has been achieved using a 2-parameter Tougaard background and line shapes indicated for fitting; using these methods a surface composition of WS<sub>2</sub> is found.

**Keywords:** WS<sub>2</sub>, tungsten, XPS, disulfide, dichalcogenide

## INTRODUCTION

There is currently a great interest in the study of transition metal dichalcogenides (TMDs) of the type MX<sub>2</sub>, where M is a metal atom (such as W or Mo) and where X is a chalcogen atom (S, Se or Te) (Ref. 1). Tungsten disulfide, WS<sub>2</sub>, is one such TMD which exhibits a diverse range of applications, including catalysis in crude oil hydrotreating and carbon dioxide hydrogenation (Refs. 2 – 3) and lubrication, especially for industrial applications (Ref. 4). Many TMDs, including WS<sub>2</sub>, exhibit a direct band gap which can be exploited in electronic transistors (Ref. 5), whilst nanostructured WS<sub>2</sub> is also being tested for hydrogen and lithium storage (Ref. 6). With diverse applications, the surface chemistry of such TMDs is of great interest, and it is with that in mind the spectra of WS<sub>2</sub> is presented herein.

## SPECIMEN DESCRIPTION (ACCESSION #00000)

**Host Material:** Tungsten disulfide, WS<sub>2</sub>

**CAS Registry #:** 12138-09-9

**Host Material Characteristics:** homogeneous; powder; unknown crystallinity; semiconductor; inorganic compound; Powder

**Chemical Name:** Tungsten disulfide

**Source:** Sigma-Aldrich

**Host Composition:** WS<sub>2</sub>

**Form:** Powder

**Structure:** polycrystalline

**History & Significance:** Received direct from manufacturer

**As Received Condition:** Supplied as new in glass bottle

**Analyzed Region:** Same as host material

**Ex Situ Preparation/Mounting:** Mounted ex-situ by pressing in to a well of a Thermo scientific powder sample holder.

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**Accession#:**

**Technique:** XPS

**Host Material:** Tungsten disulfide, WS<sub>2</sub>

**Instrument:** Thermo Scientific K-Alpha<sup>+</sup>

**Major Elements in Spectra:** W, S

**Minor Elements in Spectra:** O

**Published Spectra:** 10

**Spectra in Electronic Record:** 10

**Spectral Category:** reference

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**In Situ Preparation:** None

**Charge Control:** Combined low energy electrons and argon ions

**Temp. During Analysis:** 300K

**Pressure During Analysis:** 1.3x10<sup>-5</sup> Pa

**Pre-analysis Beam Exposure:** 30 s.

## INSTRUMENT DESCRIPTION

**Manufacturer and Model:** Thermo Scientific K-Alpha+

**Analyzer Type:** double focussing hemispherical analyser

**Detector:** multichannel resistive plate

**Number of Detector Elements:** 128

## INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

### ■ Spectrometer

**Analyzer Mode:** constant pass energy

**Throughput (T=E<sup>N</sup>):** N=0

**Excitation Source Window:** None

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

**Source Energy:** 1486.6 eV

**Source Strength:** 72 W

**Source Beam Size:** 800  $\mu$ m x 400  $\mu$ m

**Signal Mode:** multichannel direct

### ■ Geometry

**Incident Angle:** 60 °

**Source-to-Analyzer Angle:** 60 °

**Emission Angle:** 90 °

**Specimen Azimuthal Angle:** 45 °

<sup>a</sup>)Electronic mailMorganDJ3@Cardiff.ac.uk

**Acceptance Angle from Analyzer Axis:** 60 °

**Analyzer Angular Acceptance Width:** 45 ° x 0 °

■ **Ion Gun**

**Manufacturer and Model:** Thermo Scientific MAGCIS

**Energy:** 1000 eV

**Current:** 1 mA

**Current Measurement Method:** biased stage

**Sputtering Species:** None

**Spot Size (unrastered):** 120 μm

**Raster Size:** 4000 μm x 4000 μm

**Incident Angle:** 58 °

**Polar Angle:** 58 °

**Azimuthal Angle:** 90 °

**Comment:** No in-situ cleaning was performed

**DATA ANALYSIS METHOD**

**Energy Scale Correction:** None

**Recommended Energy Scale Shift:** None

**Peak Shape and Background Method:** Background: A custom 2-parameter Tougaard (U 2) background was used, with a 'C' parameter value of -650. For W 4p peaks, the loss structure should be included in defining the background, but the loss structure given a zero sensitivity factor in quantification. Peak Shapes: Finite Lorentzian (LF), Gaussian-Lorentzian (GL) mixtures or asymmetric Lorentzian (LA) lineshape were used as follows; W 4f LF(0.7,0.9,525,280,4); S 2p LF(1,1,25,180); W 4p loss peaks and W 5p<sub>3/2</sub> GL(90); S 2s LA(1,1,80); W 4p LF(0.6,1,45,80). For W 4d peaks, peaks are readily described using an asymmetric shape, modified by Gaussian-Lorentzian sum function as described by: W 4d<sub>5/2</sub> A(0.3,0.53,0)SGL(75); W 4d<sub>3/2</sub> A(0.3,0.1,0)SGL(75)

**Quantitation Method:** Quantification was performed using component definitions within CasaXPS v2.3.19rev1.2w, utilizing Scofield sensitivity factors corrected with an energy dependence of -0.6. Using this method, the ratio of S:W is found to be 1.97:1, in excellent agreement with expected 2:1 ratio.

**ACKNOWLEDGMENTS**

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SPECTRAL FEATURES TABLE							
Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV x cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
3 <sup>a</sup>	W 4f <sub>7/2</sub>	32.6	0.67	23466.8	9.80	30.44	W in WS <sub>2</sub>
3 <sup>a</sup>	W 4f <sub>5/2</sub>	34.7	0.62	17600.1	...	...	...
3 <sup>a</sup>	W 5p <sub>3/2</sub>	38.3	2.2	5361.7	...	...	...
4	S 2p <sub>3/2</sub>	162.2	0.63	9572.3	1.68	60.20	S in WS <sub>2</sub>
4	S 2p <sub>1/2</sub>	163.4	0.63	4786.2	...	...	...
5	S 2s	226.6	1.43	11901.0	1.43	...	...
6 <sup>b</sup>	W 4d <sub>5/2</sub>	244.6	3.85	71729.7	16.33	...	...
6 <sup>b</sup>	W 4d <sub>3/2</sub>	257.2	4.37	...	...	...	...
8 <sup>c</sup>	W 4p <sub>3/2</sub>	425.1	5.50	27985.0	5.16	...	...
8 <sup>c</sup>	Loss Peak	448.6	13.1	8329.5	...	...	Satellite / loss peak
8 <sup>c</sup>	W 4p <sub>1/2</sub>	491.9	5.50	9998.9	2.10	...	...
8 <sup>c</sup>	Loss Peak	514.5	13.1	3356.0	...	...	Satellite / loss peak
8 & 9	O 1s	530.9	...	...	2.93	9.36	Oxide / hydroxide species
10	W 4s	595.1	8.9	7393.6	1.81	...	...
11 <sup>d</sup>	S LMM	149.7	...	...	...	...	LMM Auger

<sup>a</sup> The reported sensitivity factor for W 4f is for both 7/2 and 5/2 peaks. For quantification, the background should include the W 5p<sub>3/2</sub> component, but the sensitivity factor set to zero for quantification.

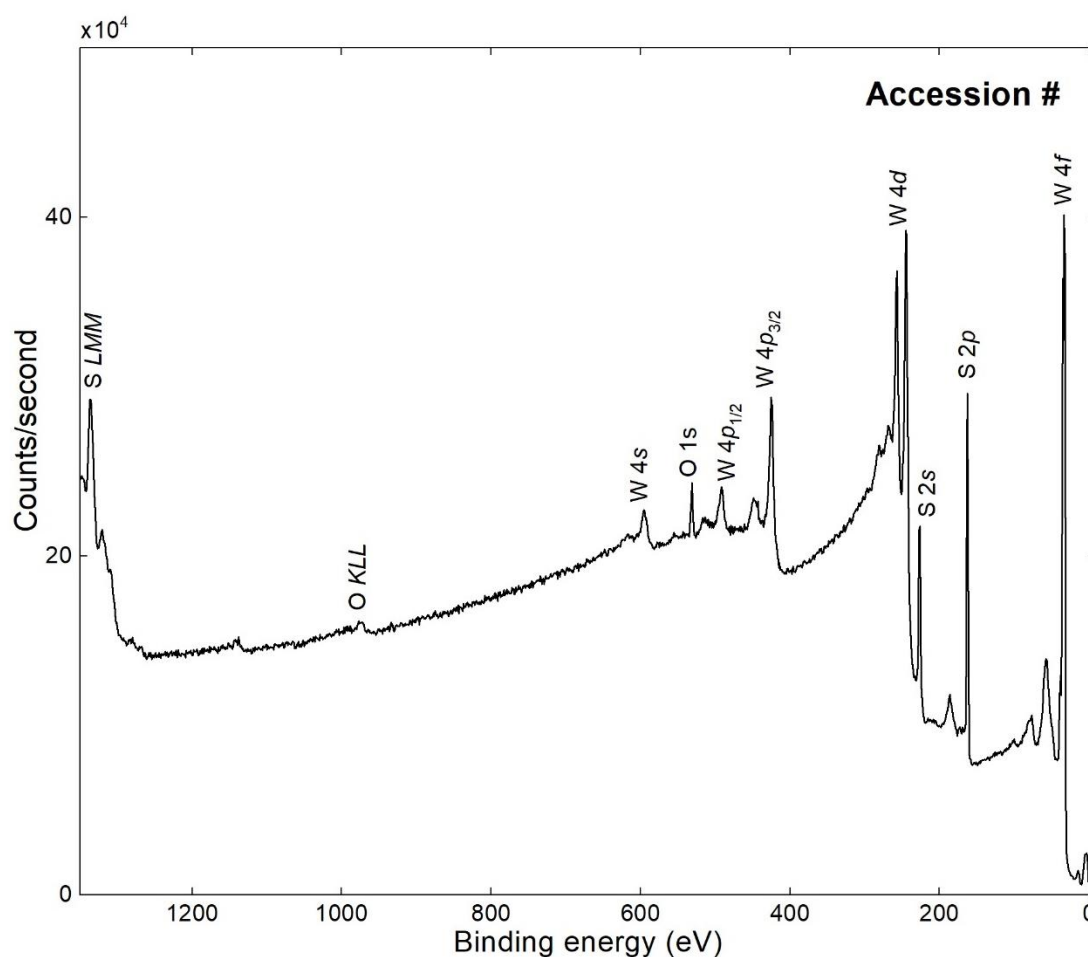
<sup>b</sup> The reported sensitivity factor for W 4d is for both 5/2 and 3/2 peaks, whilst the peak area is the total for both spin-orbit split components

<sup>c</sup> The reported sensitivity factors are for W 4p 5/2 and 3/2 peaks only, whilst the background is extended to include the loss structure, the sensitivity factor for the peak component is set to zero for quantification.

<sup>d</sup> The Auger peak energy is reported as a kinetic energy, the corresponding binding energy is 1336.9 eV.

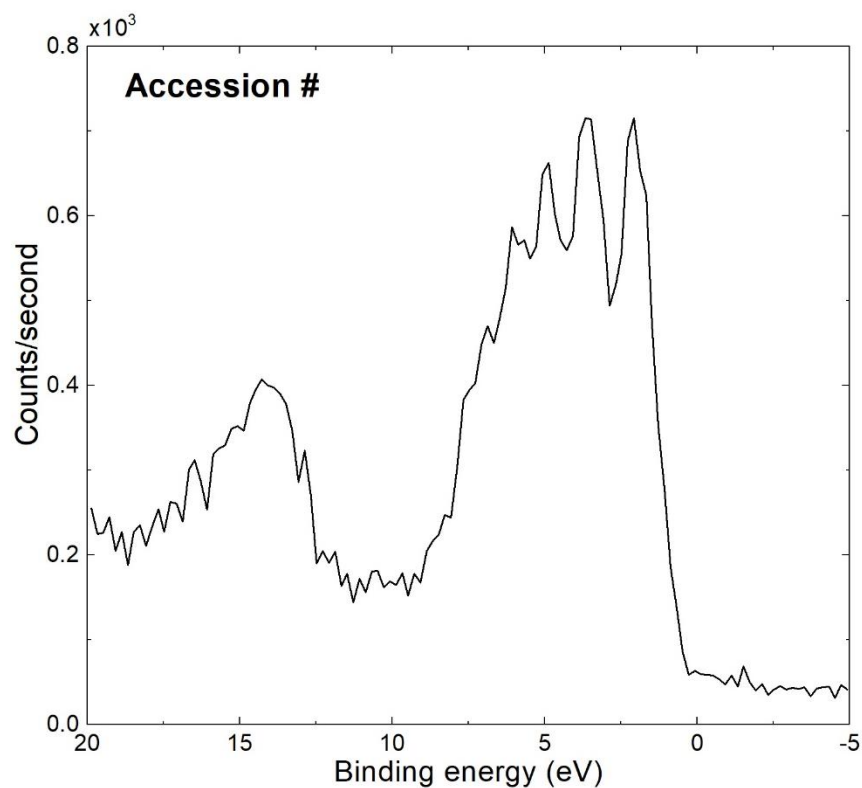
ANALYZER CALIBRATION TABLE							
Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV x cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
1	Au 4f <sub>7/2</sub>	83.99	0.76	1597652	9.580	100	Gold metal
2	Ag 3d <sub>5/2</sub>	368.28	0.58	1876744	7.38	100	Silver metal
3	Cu 2p <sub>3/2</sub>	932.67	0.83	2205571	16.73	100	Copper metal

GUIDE TO FIGURES					
Spectrum (Accession) #	Spectral Region	Voltage Shift*	Multiplier	Baseline	Comment #
1	Survey	0	1	0	
2	Valence	0	1	0	
3	W 4f	0	1	0	
4	S 2p	0	1	0	
5	S 2s	0	1	0	
6	W 4d	0	1	0	
7	C 1s	0	1	0	
8	W 4p & O 1s	0	1	0	
9	O 1s	0	1	0	
10	W 4s	0	1	0	
11	S LMM	0	1	0	



Publish in *Surface Science Spectra*: Yes ☒ No ☐

Accession #	01312-01
Host Material	Tungsten disulfide
Technique	XPS
Spectral Region	survey
Instrument	Thermo Scientific K-Alpha+
Excitation Source	Al Ka monochromatic
Source Energy	1486.6 eV
Source Strength	72 W
Source Size	0.8 mm x 0.4 mm
Analyzer Type	double focussing hemispherical analyser
Incident Angle	60°
Emission Angle	90°
Analyzer Pass Energy	150 eV
Analyzer Resolution	1 eV
Total Signal Accumulation Time	130 s
Total Elapsed Time	260 s
Number of Scans	10
Effective Detector Width	1 eV



Publish in SSS: Yes ☒ No ☐

■ **Accession #:**

■ **Host Material:** Tungsten disulfide

■ **Technique:** XPS

■ **Spectral Region:** Valence

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al Ka  
monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.8 mm x 0.4 mm

Analyzer Type: spherical sector

Incident Angle: 60 °

Emission Angle: 90 °

Analyzer Pass Energy 20 eV

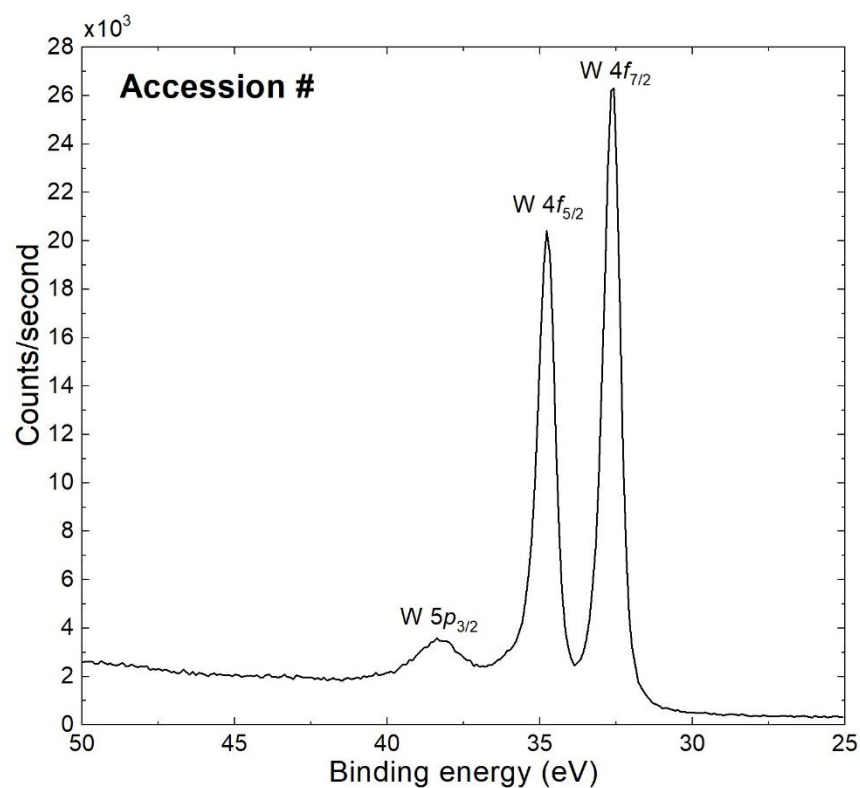
Analyzer Resolution: 0.45 eV

Total Signal Accumulation Time: 200 s

Total Elapsed Time: 226 s

Number of Scans: 20

Effective Detector Width: 0.2 eV



Publish in SSS: Yes ☒ No ☐

■ Accession #:

■ Host Material: Tungsten disulfide

■ Technique: XPS

■ Spectral Region: W 4f

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al K $\alpha$  monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.8 mm x 0.4 mm

Analyzer Type: double focussing hemispherical analyser

Incident Angle: 60 °

Emission Angle: 90 °

Analyzer Pass Energy 20 eV

Analyzer Resolution: 0.45 eV

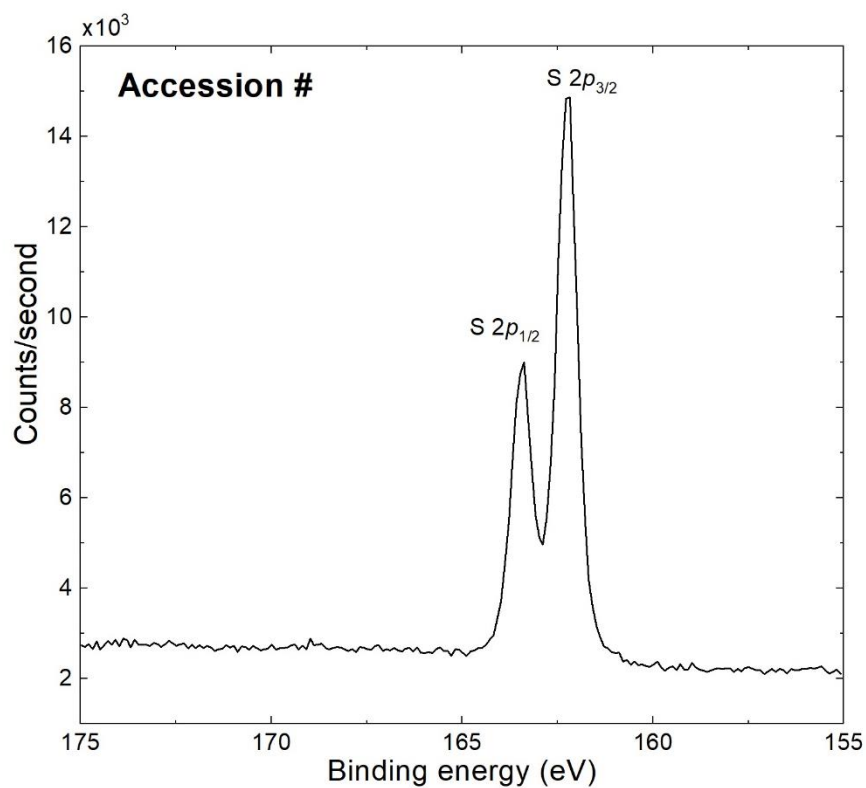
Total Signal Accumulation Time: 240 s

Total Elapsed Time: 251 s

Number of Scans: 20

Effective Detector Width: 0.1 eV





Publish in SSS: Yes ☒ No ☐

■ **Accession #:**

■ **Host Material:** Tungsten disulfide

■ **Technique:** XPS

■ **Spectral Region:** S 2p

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al K $\alpha$  monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.8 mm x 0.4 mm

Analyzer Type: double focussing hemispherical analyser

Incident Angle: 60 °

Emission Angle: 90 °

Analyzer Pass Energy 20 eV

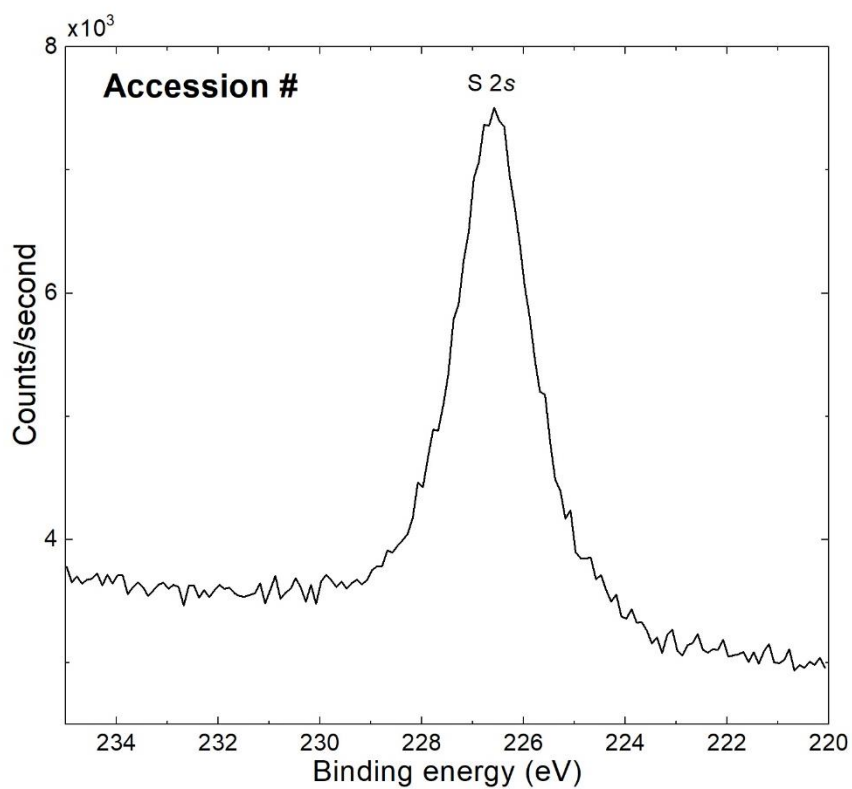
Analyzer Resolution: 0.45 eV

Total Signal Accumulation Time: 200 s

Total Elapsed Time: 226 s

Number of Scans: 20

Effective Detector Width: 0.1 eV



Publish in SSS: Yes ☒ No ☐

■ **Accession #:**

■ **Host Material:** Tungsten disulfide

■ **Technique:** XPS

■ **Spectral Region:** S 2s

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al Ka  
monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.8 mm x 0.4 mm

Analyzer Type: double focussing  
hemispherical analyser

Incident Angle: 60 °

Emission Angle: 90 °

Analyzer Pass Energy 20 eV

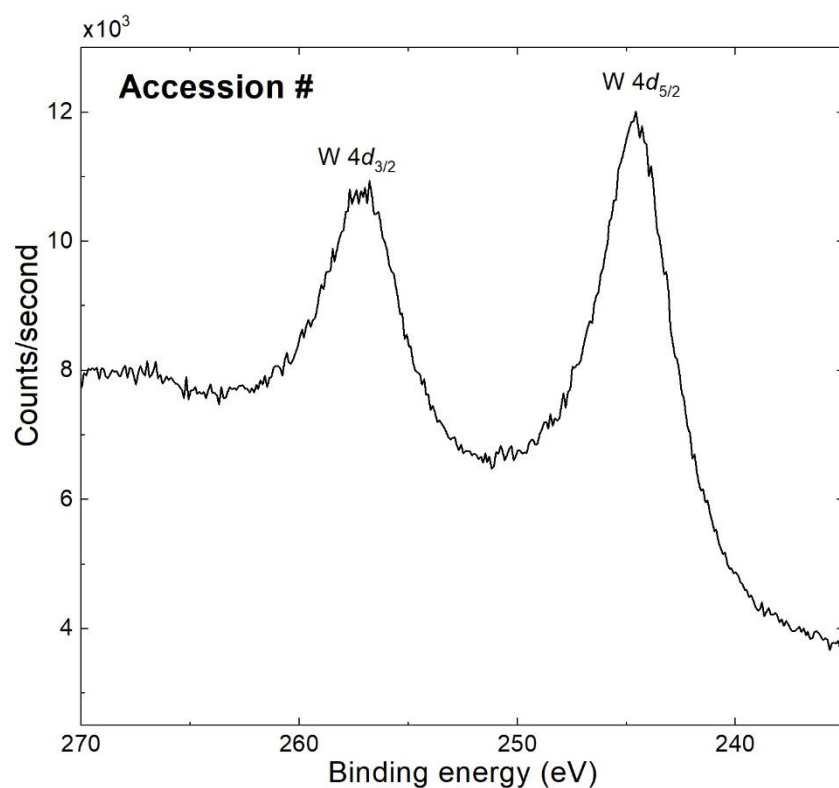
Analyzer Resolution: 0.45 eV

Total Signal Accumulation Time: 120 s

Total Elapsed Time: 131 s

Number of Scans: 20

Effective Detector Width: 0.1 eV



Publish in SSS: Yes ☒ No ☐

■ **Accession #:**

■ **Host Material:** Tungsten disulfide

■ **Technique:** XPS

■ **Spectral Region:** W 4d

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al Ka  
monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.8 mm x 0.4 mm

Analyzer Type: double focussing  
hemispherical analyser

Incident Angle: 60 °

Emission Angle: 90 °

Analyzer Pass Energy 20 eV

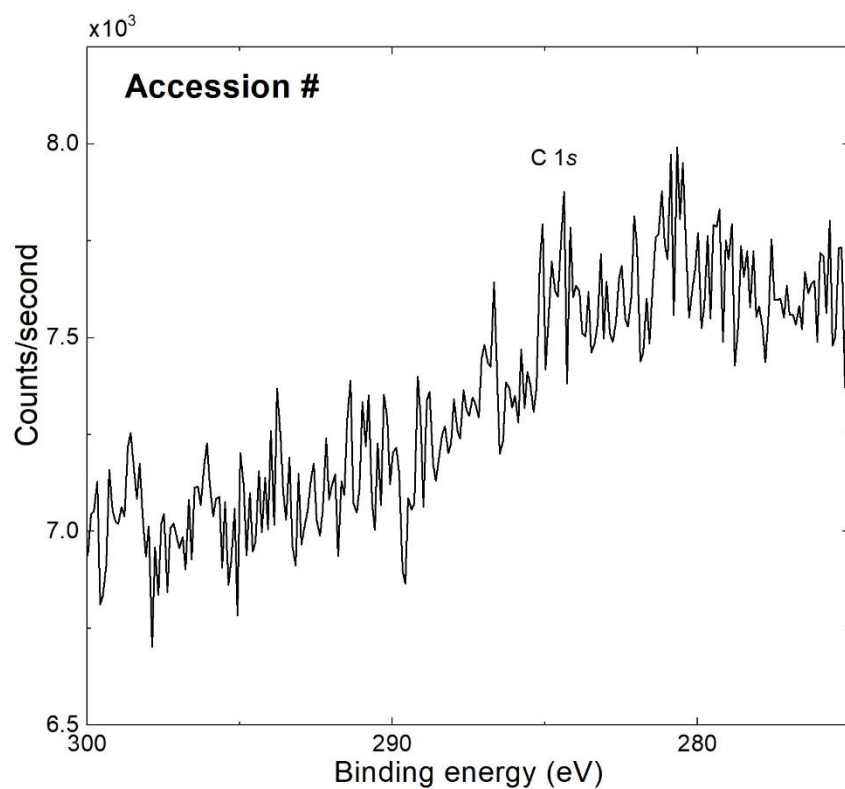
Analyzer Resolution: 0.45 eV

Total Signal Accumulation Time: 300 s

Total Elapsed Time: 351 s

Number of Scans: 20

Effective Detector Width: 0.1 eV



Publish in SSS: Yes ☒ No ☐

■ **Accession #:**

■ **Host Material:** Tungsten disulfide

■ **Technique:** XPS

■ **Spectral Region:** C 1s

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al Ka  
monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.8 mm x 0.4 mm

Analyzer Type: double focussing  
hemispherical analyser

Incident Angle: 60 °

Emission Angle: 90 °

Analyzer Pass Energy 20 eV

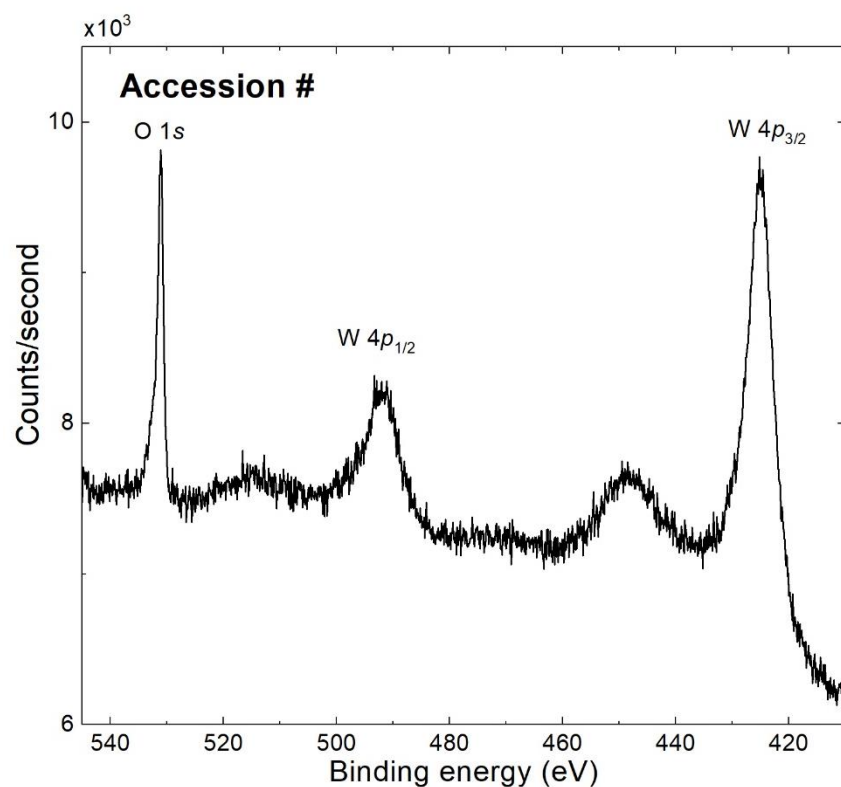
Analyzer Resolution: 0.45 eV

Total Signal Accumulation Time: 120 s

Total Elapsed Time: 131 s

Number of Scans: 20

Effective Detector Width: 0.1 eV



Publish in SSS: Yes ☒ No ☐

■ Accession #:

■ Host Material: Tungsten disulfide

■ Technique: XPS

■ Spectral Region: W4p & O 1s

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al Ka  
monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.8 mm x 0.4 mm

Analyzer Type: double focussing  
hemispherical analyser

Incident Angle: 60 °

Emission Angle: 90 °

Analyzer Pass Energy 20 eV

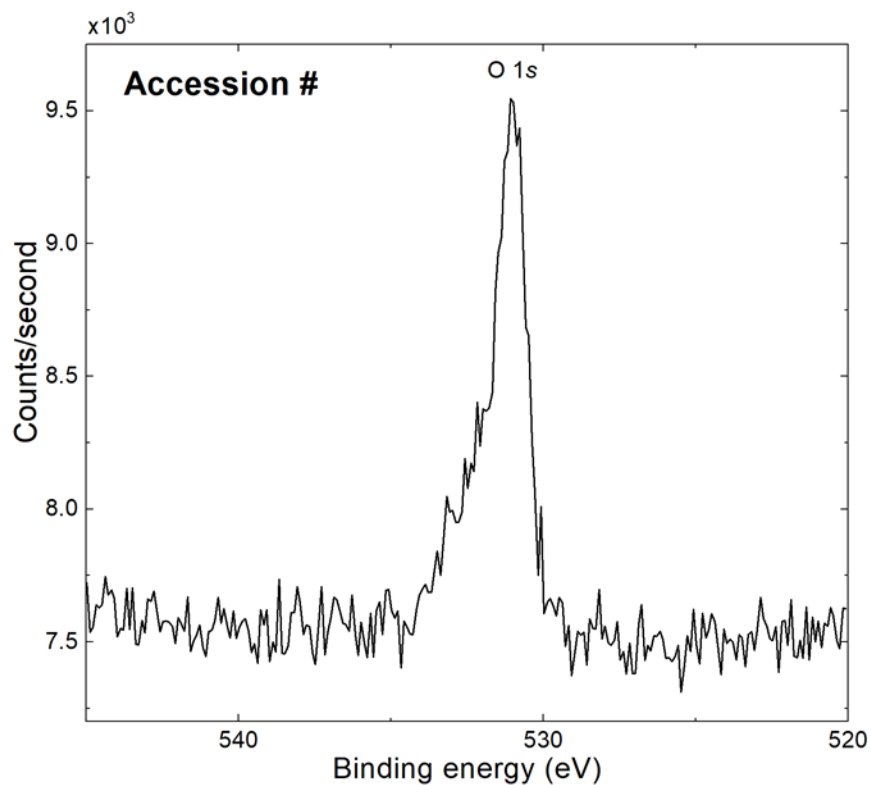
Analyzer Resolution: 0.45 eV

Total Signal Accumulation Time: 900 s

Total Elapsed Time: 935 s

Number of Scans: 20

Effective Detector Width: 0.1 eV



Publish in SSS: Yes ☒ No ☐

■ Accession #:

■ Host Material: Tungsten disulfide

■ Technique: XPS

■ Spectral Region: O 1s

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al Ka  
monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.8 mm x 0.4 mm

Analyzer Type: double focussing  
hemispherical analyser

Incident Angle: 60 °

Emission Angle: 90 °

Analyzer Pass Energy 20 eV

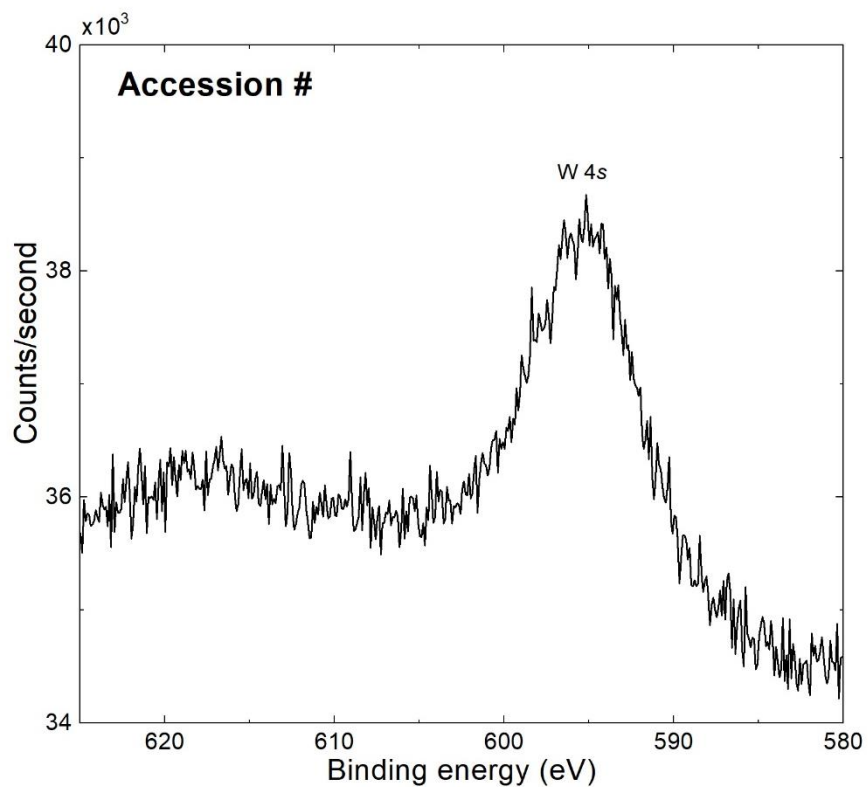
Analyzer Resolution: 0.45 eV

Total Signal Accumulation Time: 900 s

Total Elapsed Time: 935 s

Number of Scans: 20

Effective Detector Width: 0.1 eV



**Publish in SSS: Yes ☒ No ☐**

■ **Accession #:**

■ **Host Material:** Tungsten disulfide

■ **Technique:** XPS

■ **Spectral Region:** W 4s

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al Ka  
monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.4 mm x 0.4 mm

Analyzer Type: double focussing  
hemispherical analyser

Incident Angle: 60 °

Emission Angle: 90 °

Analyzer Pass Energy 20 eV

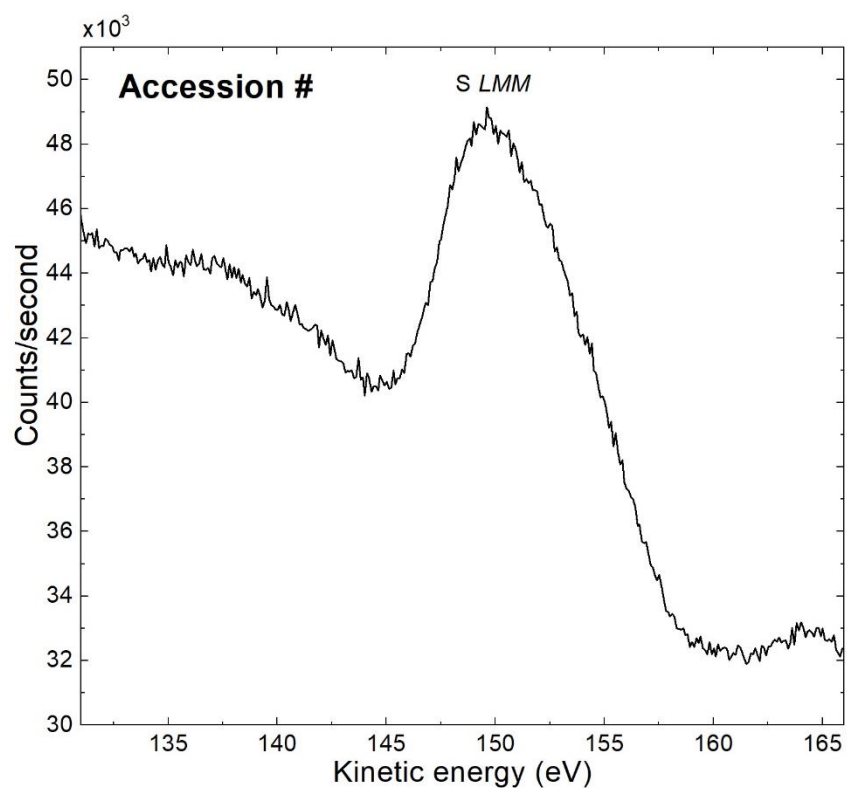
Analyzer Resolution: 0.45 eV

Total Signal Accumulation Time: 420 s

Total Elapsed Time: 451 s

Number of Scans: 20

Effective Detector Width: 0.1 eV



Publish in SSS: Yes ☒ No ☐

■ **Accession #:**

■ **Host Material:** Tungsten disulfide

■ **Technique:** XPS

■ **Spectral Region:** S LMM

Instrument: Thermo Scientific K-Alpha+

Excitation Source: Al Ka  
monochromatic

Source Energy: 1486.6 eV

Source Strength: 72 W

Source Size: 0.4 mm x 0.4 mm

Analyzer Type: double focussing  
hemispherical analyser

Incident Angle: 60 °

Emission Angle: 90 °

Analyzer Pass Energy 20 eV

Analyzer Resolution: 0.45 eV

Total Signal Accumulation Time: 900 s

Total Elapsed Time: 935 s

Number of Scans: 20

Effective Detector Width: 0.1 eV