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XPS of the surface chemical environment of CsMAFAPbBrI trication-mixed halide perovskite film

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

Acccession#: 01650

Technique: XPS

Host Material: $Cs_{0.08}MA_{0.18}FA_{0.78}PbBr_{0.42}I_{2.58}$ perovskite

Instrument: SPECS PHOIBOS 150

Major Elements in Spectra: Pb, I, C, N

Minor Elements in Spectra: Br, Cs, O

Published Spectra: 9

Spectra in Electronic Record: 9

Spectral Category: comparison

Perovskite based materials have been considered as the most promising materials in several solar driven processes, specially photovoltaics. Some features as high sunlight harvesting and improved carrier transport has been highlighted to impact in the efficiency of the above topics, but limited studies have pointed out to their surface composition, which mainly influence on above abilities. As starting point to recognize the surface environment and chemical states of this materials, X-ray photoelectron spectroscopy measurements were performed. A trication mixed halide perovskite films based on $Cs_{0.08}MA_{0.18}FA_{0.78}PbBr_{0.42}I_{2.58}$ was prepared by one step process and deposited on an indium tin oxide substrate by spin-coating. Survey spectra, Pb 4f, I 3d, Br 3d, Cs 3d, C 1s, N 1s, Pb 4d, Pb 5d core levels and valence band spectra were measured for the semiconductor sample. Results exhibit symmetrical peaks, indicating that a homogenous solid solution was achieved. Main chemical states of this kind of perovskite such as Cs-Br and Pb-I species are also showed. We deduced the later signals are associated to the $[PbI_6]^{4-}$ octahedra existing into the perovskite lattice, while the former one is related Cs^+ bounded to $[PbBr_6]^{4-}$ units on the perovskite surface. Interestingly, NR_4^+ signal was also identified, associated to the presence of methylammonium and formamidinium cations.

Keywords: trication-mixed halide perovskite; surface environment; XPS

INTRODUCTION

In optoelectronics, $APbX_3$ perovskite-based materials, (A= cesium, Cs+; methylammonium, MA+; formamidinium, FA+; X=Cl, Br, I, Br/I) have been recognized as the most competitive materials due to their unique characteristics as high sunlight harvesting, improved charrier transport ability and long exciton diffusion length (Ref. 1). These features have allowed, for instance, the fabrication of emerging perovskite solar devices with photoconversion efficiencies higher than 25.2 % (Ref. 2). One of the most studied perovskites is based on black phase CsMAFAPbBrI trication mixed halide structure, due to this composition prevent some important issues as phase segregation and the α-to-γ phase transition (Ref. 3). This characteristic can be achieved since the combination of Cs/MA/FA cations tunes the Goldschmidt tolerance factor of the perovskite, mediating the thermal and humidity stability (Ref. 3). By obtaining information of the surface environment, the chemical speciation can be established as the first step to future investigations about how the mixture of cations and anions can control the stability of this kind of perovskites. Hence, we carried out the XPS analysis of a perovskite sampled based on the $Cs_{0.08}MA_{0.18}FA_{0.78}PbBr_{0.42}I_{2.58}$ composition, where their characteristic chemical species were elucidated.

XPS survey spectrum of the specimen reveals the presence of lead, iodine, bromine, cesium, carbon, nitrogen and oxygen.

High resolution spectrum of Pb 4f was fitted in two contributions corresponding to one chemical species, centered at 138.2 and 143.1 eV, attributed to lead bonding with iodine, **Pb**-I (Refs. 4 and 5). I 3d high resolution spectrum also displayed the Pb-**I** species with the doublet centered at 619.3 and 630.8 eV.

Br 3d signal shows the spin-orbit splitting at 68.3 and 69.3 eV corresponding to bromine bonding with cesium, **Br**-Cs (Refs. 5

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and 6). The same species Br-**Cs** was observed in Cs 3d spectra with its doublet centered at 725.1 and 739.1 eV.

C 1s signal shows three chemical species. **C**-(C,H) at 284.8 eV, that is adventitious carbon used as a reference (Ref. 7). Carbon bounded to nitrogen, (C=O)-N-**C** at 286.1 eV. And carbon double bounded with oxygen and related with nitrogen (**C**=O)-N-C at 288.2 eV. N 1s spectrum was decomposed in two chemical species. At 400.5 eV assigned to (C=O)-**N**-C and at 402.3 eV (Ref. 7) attributed to NR_4^+ (Ref. 5).

We believe that the presence of MA^+/FA^+ can be associated to this signal. Since Br and I are well-known to form $[PbX_6]^{4-}$ octahedra as the building blocks for the perovskite structure (Ref. 8), we only observed the Pb-I signal from the material, attributed to the presence a high density of $[PbI_6]^{4-}$ units presented into the perovskite matrix. This fact is expected since iodide content is nominally higher than bromide. However, the presence of Cs-Br (Cs^+ is in lower content than MA^+ and FA^+, nominally 0.7 at. %) allowed to deduce that trication mixed halide perovskite also exhibits $[PbBr_6]$ terminals bounded with Cs^+ cations on its surface. The latter feature could be a main factor to explain some intrinsic properties of the material to be suitable for light-driven processes as photovoltaics or water splitting.

This contribution opens the door to a better understanding about the species composing the perovskite lattice, which could be interesting to explain the surface properties of the materials in solar-driven applications.

SPECIMEN DESCRIPTION (ACCESSION # 01650)

Host Material: $Cs_{0.08}MA_{0.18}FA_{0.78}PbBr_{0.42}I_{2.58}$ perovskite

CAS Registry #: unknown

Host Material Characteristics: homogeneous; solid; polycrystalline; semiconductor; semiconductor; Thin Film

Chemical Name: $Cs_{0.08}MA_{0.18}FA_{0.78}PbBr_{0.42}I_{2.58}$ trication-mixed halide perovskite

Source: 500 nm of CsFAMAPbIBr perovskite layer was fabricated employing one step deposition process as follows. CsFAMAPbIBr solution was prepared by mixing 1.15 M PbI_2 (Alfa Aesar), 0.19 M $PbBr_2$ (TCI), 1.1 M formamidinium iodide (FAI, Greatcell Solar) and 0.19 M methylammonium bromide (MABr, Greatcell Solar) in a mixture of N,N-Dimethylformamide (DMF) and Dimethyl sulfoxide (DMSO) with a 4:1 volume ratio (solution A). Subsequently, a solution B was fabricated by mixing 1.15 M CsI (TCI) and 1.15 M PbI_2 in DMSO. Then solution A and B were mixed in a volume ratio of 10:1 to have the triple cation perovskite precursor. The deposition on Indium Thin Oxide glass (ITO) was carried out by spin-coating the aforementioned solution in two steps: first, at 2000 rpm for 12 s (200 rpm s^{-1}), and then 5000 rpm for 25 s (2000 rpm s^{-1}). We used these values of RPM in order to obtain a homogenous layer for material characterization, which has been reported elsewhere (Ref. 9) In this step, an aliquot of chlorobenzene (anti-solvent) was dropped 9 s before the end of the process. Afterwards, films were annealed at 100°C for 60 min.

Host Composition: $Cs_{0.08}MA_{0.18}FA_{0.78}PbBr_{0.42}I_{2.58}$ deposited on ITO

Form: Thin film

Structure: $Cs_{0.08}MA_{0.18}FA_{0.78}PbBr_{0.42}I_{2.58}$

History & Significance: The importance of trication perovskites bases on the improvement in the light harvesting and carrier transport in order to increase the photoconversion efficiency in perovskite solar cells. The presence of both $[PbBr_6]^{4-}$ and $[PbI_6]^{4-}$ terminals on the perovskite surface allows to deduce that these structures are in direct contact with the other components during perovskite solar cells (PSCs) fabrication (for instance, electron and hole transporter materials). Thus, depending on the density of formed $[PbBr_6]^{4-}$ and $[PbI_6]^{4-}$ units, the photocarriers transport into PSCs is altered, which would be the first step to understand why the photoconversion efficiency of PSCs is directly related with the amount of halides into the perovskite active layer.

As Received Condition: As grown

Analyzed Region: same as host material

Ex Situ Preparation/Mounting: As received, sample was fixed with carbon double-sided conductive adhesive tape.

In Situ Preparation: None

Charge Control: Electron flood gun (SPECS FG-500) operated at 58μA and 2eV.

Temp. During Analysis: 300 K

Pressure During Analysis: $< 1 \times 10^{-7}$ Pa

Pre-analysis Beam Exposure: Not applicable s

INSTRUMENT DESCRIPTION

Manufacturer and Model: SPECS PHOIBOS 150 - SPECS Surface Nano Analysis GmbH

Analyzer Type: spherical sector

Detector: other

Number of Detector Elements: 25

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

■Spectrometer

Analyzer Mode: constant pass energy

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

Excitation Source Window: Mylar window, allows high X-ray transmission: 88% for Al Kα.

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 100 W

Source Beam Size: 2000 μm x 2000 μm

Signal Mode: multichannel direct

■Geometry

Incident Angle: 55 °

Source-to-Analyzer Angle: 55 °

Emission Angle: 0 °

Specimen Azimuthal Angle: Not applicable

Acceptance Angle from Analyzer Axis: 16 °

Analyzer Angular Acceptance Width: 16 ° x 16 °

■*Ion Gun*

Manufacturer and Model: SPECS IQE 12/38

Energy: 5000 eV

Current: 70 mA

Current Measurement Method: biased stage

Sputtering Species: Ar^+

Spot Size (unrastered): 3000 μm x 3000 μm

Raster Size: Not applicable μm x μm

Incident Angle: 54 °

Polar Angle: Not applicable

Azimuthal Angle: Not applicable

Comment: The specimens were analyzed as loaded. The ion gun was used only for cleaning the Ag reference foil.

DATA ANALYSIS METHOD

Energy Scale Correction: The binding energy of the adventitious carbon, C-(C,H) at 284.8 eV (Ref. 7) was used as reference to adjust the binding energy scale of the spectra.

Recommended Energy Scale Shift: 3.37 eV

Peak Shape and Background Method: Peak position and width were determined from fitting the spectra using a mixed Gaussian– Lorentzian function after subtraction of a Shirley background using the CasaXPS Software.

Quantitation Method: Peak areas were obtained from fitting the spectra and relative sensitivity factors from the atomic photoionization cross section of each core level provided by SPECS Prodigy library.

ACKNOWLEDGMENTS

Authors thank to Laboratorio Central en Ciencia de Superficies (SurfLab-UIS) from Universidad Industrial de Santander for providing their facilities to perform the XPS measurements. This work was partially supported by Universidad de Pamplona, through GA313-BP-2017.

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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
01650-02	Pb 4f	…	…	2.79x10^{4}	23.59	12.4	…
01650-02	Pb 4f$_{7/2}$	138.2	0.99	1.59 x10^{4}	…	…	**Pb**-I
01650-02	Pb 4f$_{5/2}$	143.1	0.98	1.19 x10^{4}	…	…	**Pb**-I
01650-03	I 3d	…	…	9.82x10^{4}	31.29	38.68	…
01650-03	I 3d$_{5/2}$	619.3	1.16	5.89x10^{4}	…	…	Pb-**I**
01650-03	I 3d$_{3/2}$	630.8	1.16	3.93 x10^{4}	…	…	Pb-**I**
01650-04	Br 3d	…	…	1.04x10^{3}	2.98	3.2	…
01650-04	Br 3d$_{5/2}$	68.3	1.16	0.62 x10^{3}	…	…	Cs-**Br**
01650-04	Br 3d$_{3/2}$	69.3	1.16	0.41 x10^{3}	…	…	Cs-**Br**
01650-05	Cs 3d	…	…	1.91x10^{3}	36.23	0.7	…
01650-05	Cs 3d$_{5/2}$	725.1	1.34	1.14 x10^{3}	…	…	**Cs**-Br
01650-05	Cs 3d$_{3/2}$	739.1	1.34	0.76 x10^{3}	…	…	**Cs**-Br
01650-06	C 1s	…	…	2.74x10^{3}	1.00	27.3	…
01650-06	C 1s	284.8	1.53	1.39 x10^{3}	…	…	**C**-(C,H)
01650-06	C 1s	286.1	1.53	0.49 x10^{3}	…	…	(C=O)-N-**C**
01650-06	C 1s	288.2	1.53	0.86 x10^{3}	…	…	(**C**=O)-N-C
01650-07	N 1s	…	…	2.41x10^{3}	1.76	14.6	…
01650-07	N 1s	400.5	1.25	2.16 x10^{3}	…	…	(C=O)-**N**-C
01650-07	N 1s	402.3	1.25	0.24 x10^{3}	…	…	**N**-R$_{4}^{+}$
01650-07	Pb 4d$_{5/2}$	413.0	3.68	1.25x10^{4}	…	…	…
01650-07	Pb 4d$_{3/2}$	436.0	3.68	8.37x10^{3}	…	…	…
01650-08	O 1s	…	…	7.49x10^{2}	2.77	3.1	…
01650-08	O 1s	532.2	2.40	…	…	…	C=**O**
01650-09	Pb 5d$_{5/2}$	19.3	0.99	2.47x10^{3}	…	…	…
01650-09	Pb 5d$_{3/2}$	21.9	0.98	1.65x10^{3}	…	…	…
01650-09[a]	VBM	0.94	…	…	…	…	…

[a] Valence band maximum (VBM)

ANALYZER CALIBRATION TABLE

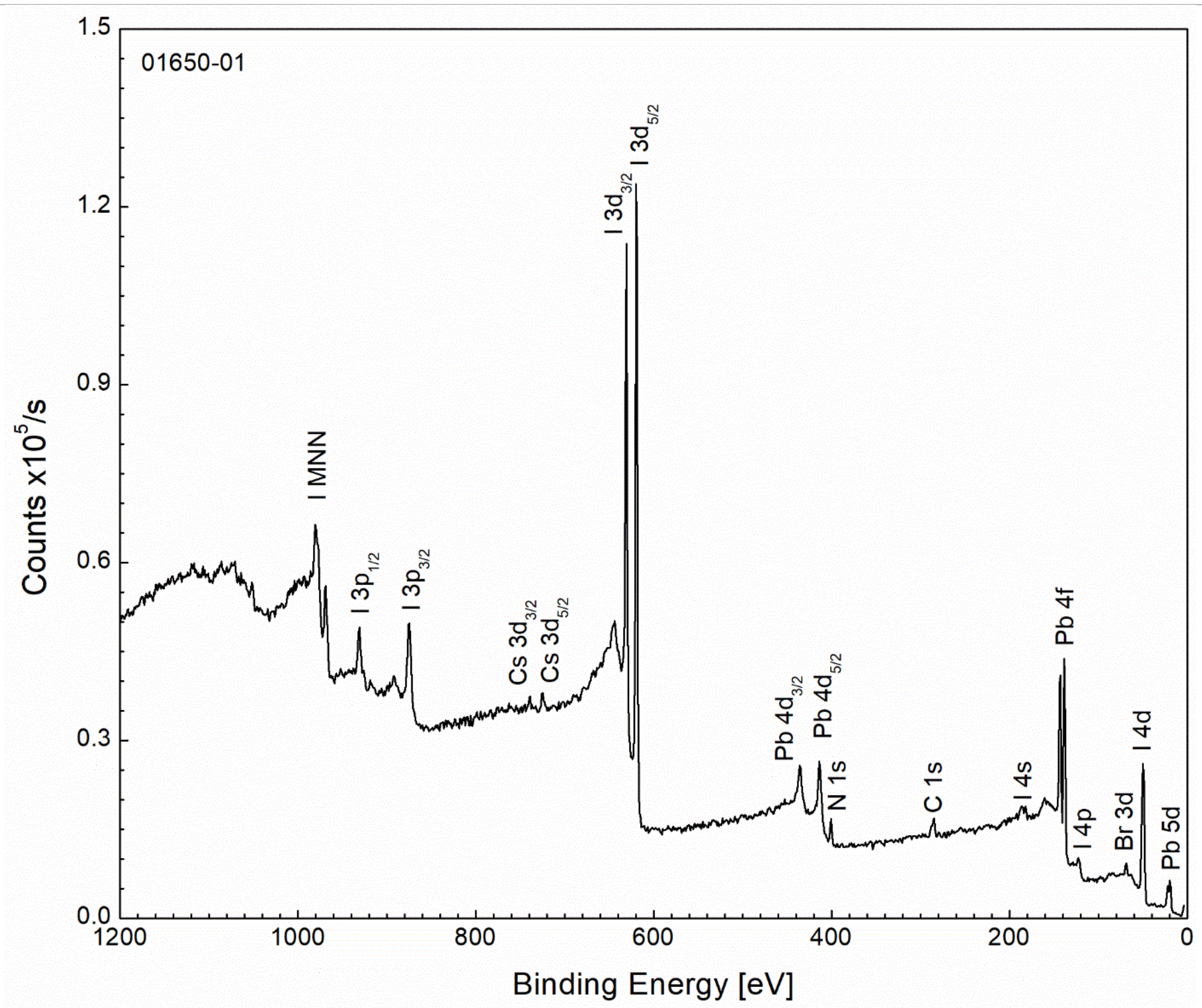
Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV x cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
…	Ag 3d$_{5/2}$	368.3	0.50	0.15x10^{6}	...	...	...

GUIDE TO FIGURES

Spectrum (Accession) #	Spectral Region	Voltage Shift*	Multiplier	Baseline	Comment #
01650-01	Survey	0	1	0	1
01650-02	Pb 4f	-3.37	1	0	1
01650-03	I 3d	-3.37	1	0	1
01650-04	Br 3d	-3.37	1	0	1
01650-05	Cs 3d	-3.37	1	0	1
01650-06	C 1s	-3.37	1	0	1
01650-07	N 1s, Pb 4d	-3.37	1	0	1
01650-08	O 1s	-3.37	1	0	1
01650-09	Pb 5d, VB	-3.37	1	0	1

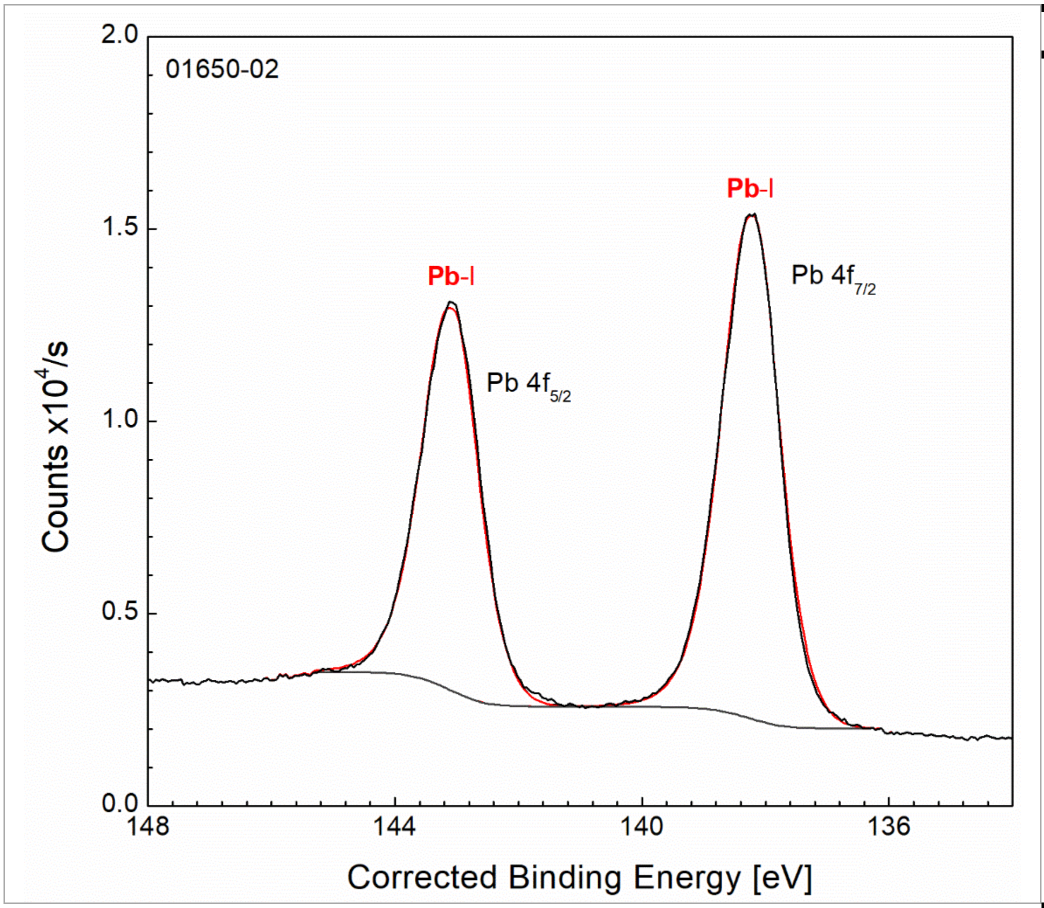
*Voltage shift of the archived (as-measured) spectrum relative to the printed figure. The figure reflects the recommended energy scale correction due to a calibration correction, sample charging, flood gun, or other phenomenon.

1, refers to $Cs_{0.08}MA_{0.18}FA_{0.78}PbBr_{0.42}I_{2.58}$ perovskite



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

Accession #	**01650-01**
Host Material	$Cs_{0.08}MA_{0.18}FA_{0.78}PbBr_{0.42}I_{2.58}$ perovskite
Technique	XPS
Spectral Region	survey
Instrument	SPECS PHOIBOS 150
Excitation Source	Al Ka monochromatic
Source Energy	1486.6 eV
Source Strength	100 W
Source Size	2 mm x 2 mm
Analyzer Type	spherical sector analyzer
Incident Angle	55˚
Emission Angle	0˚
Analyzer Pass Energy	100 eV
Analyzer Resolution	0.5 eV
Total Signal Accumulation Time	122 s
Total Elapsed Time	260 s
Number of Scans	1
Effective Detector Width	5.28 eV
Energy Step	1 eV



Publish in *SSS*: Yes ☒ No ☐

- **Accession #: 01650-02**
- **Host Material:** $Cs_{0.08}MA_{0.18}FA_{0.78}PbBr_{0.42}I_{2.58}$ perovskite
- **Technique:** XPS
- **Spectral Region:** Pb 4f

Instrument: SPECS PHOIBOS 150

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 100 W

Source Size: 2 mm x 2 mm

Analyzer Type: spherical sector

Incident Angle: 55 °

Emission Angle: 0 °

Analyzer Pass Energy 30 eV

Analyzer Resolution: 0.5 eV

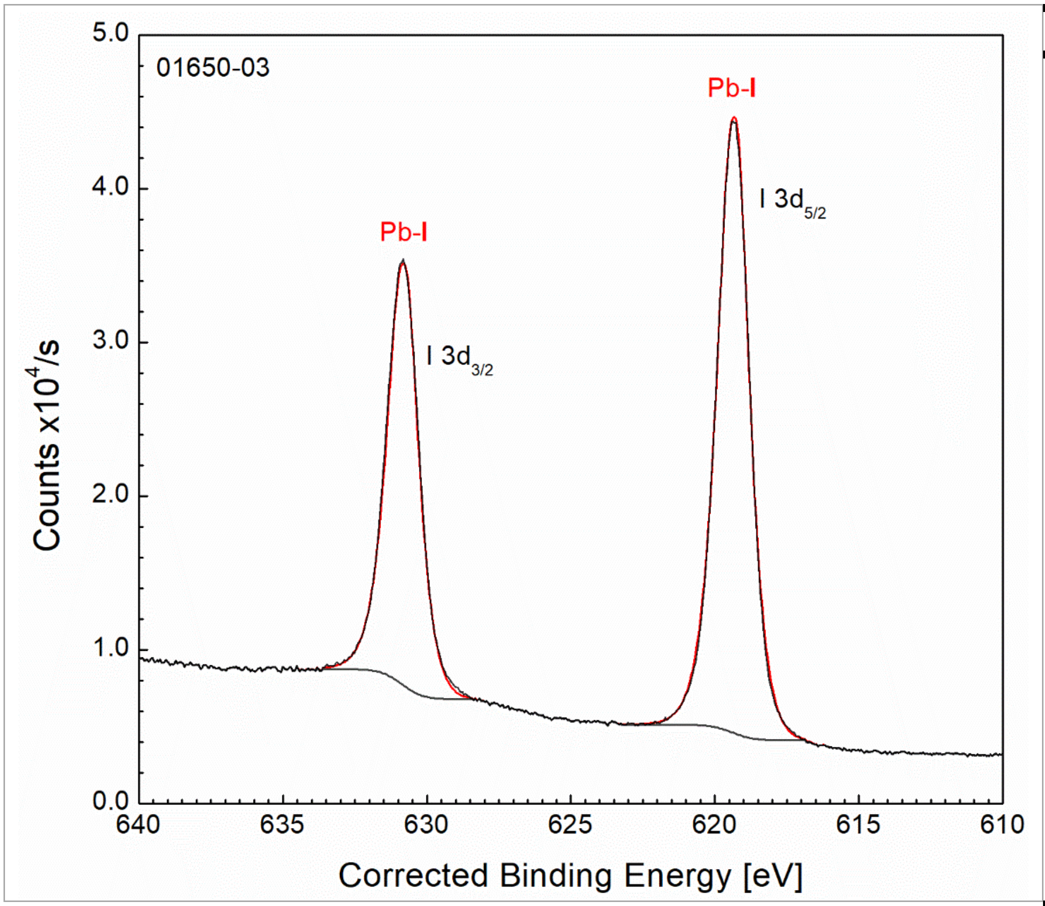
Total Signal Accumulation Time: 542 s

Total Elapsed Time: 797 s

Number of Scans: 15

Effective Detector Width: 2.64 eV

Energy Step: 0.05 eV



Publish in *SSS*: Yes ☒ No ☐

- **Accession #: 01650-03**
- **Host Material:** $Cs_{0.08}MA_{0.18}FA_{0.78}PbBr_{0.42}I_{2.58}$ perovskite
- **Technique:** XPS
- **Spectral Region:** I 3d

Instrument: SPECS PHOIBOS 150

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 100 W

Source Size: 2 mm x 2 mm

Analyzer Type: spherical sector

Incident Angle: 55 °

Emission Angle: 0 °

Analyzer Pass Energy 30 eV

Analyzer Resolution: 0.5 eV

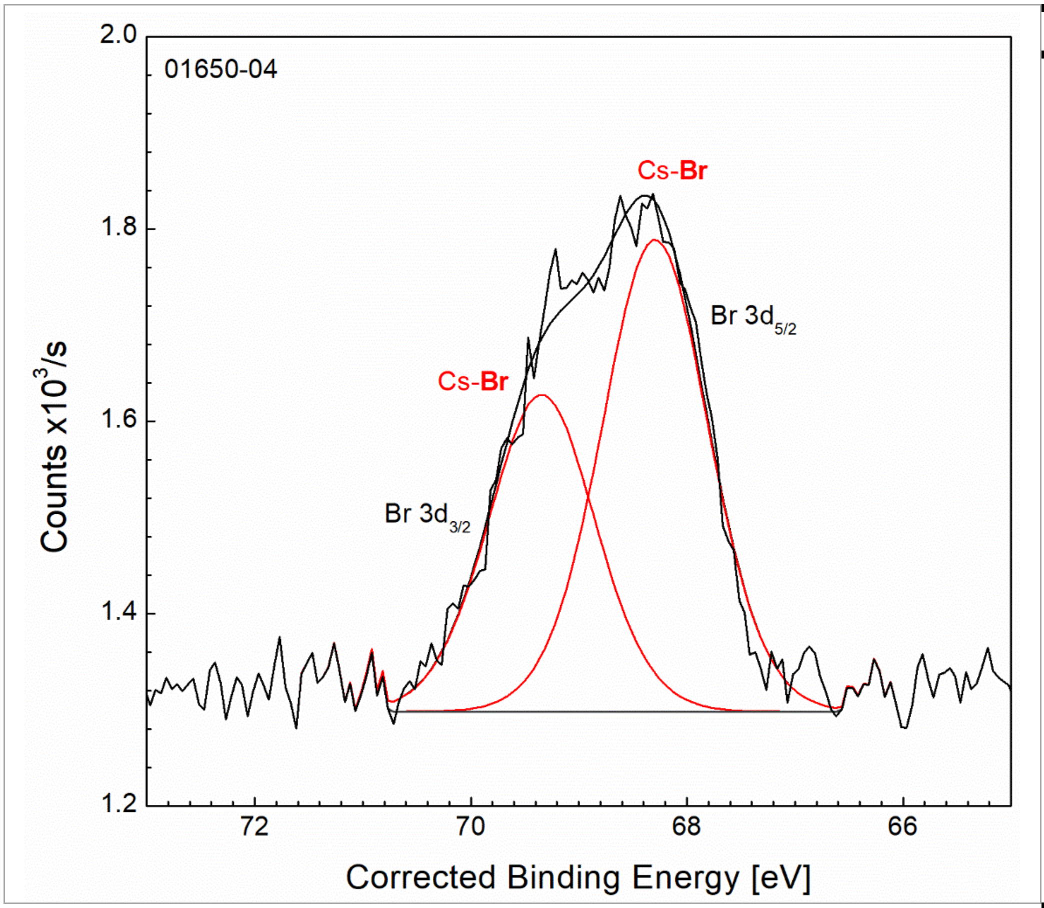
Total Signal Accumulation Time: 640 s

Total Elapsed Time: 941 s

Number of Scans: 8

Effective Detector Width: 2.64 eV

Energy Step: 0.05 eV



Publish in *SSS*: Yes ☒ No ☐

- **Accession #: 01650-04**
- **Host Material:** $\mathrm{Cs_{0.08}MA_{0.18}FA_{0.78}PbBr_{0.42}I_{2.58}}$ perovskite
- **Technique:** XPS
- **Spectral Region:** Br 3d

Instrument: SPECS PHOIBOS 150

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 100 W

Source Size: 2 mm x 2 mm

Analyzer Type: spherical sector

Incident Angle: 55 °

Emission Angle: 0 °

Analyzer Pass Energy 30 eV

Analyzer Resolution: 0.5 eV

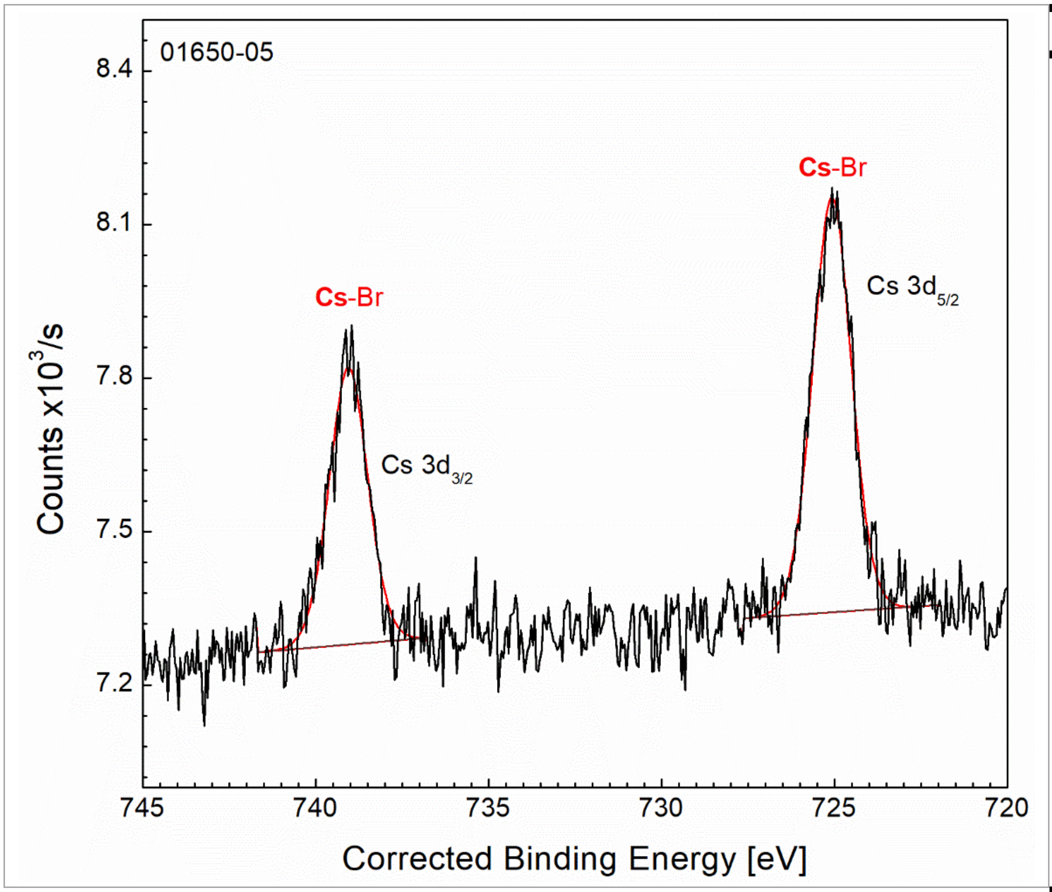
Total Signal Accumulation Time: 1402 s

Total Elapsed Time: 2061 s

Number of Scans: 25

Effective Detector Width: 2.64 eV

Energy Step: 0.05 eV



Publish in *SSS*: Yes ☒ No ☐

- **Accession #: 01650-05**
- **Host Material:** $\mathrm{Cs_{0.08}MA_{0.18}FA_{0.78}PbBr_{0.42}I_{2.58}}$ perovskite
- **Technique:** XPS
- **Spectral Region:** Cs 3d

Instrument: SPECS PHOIBOS 150

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 100 W

Source Size: 2 mm x 2 mm

Analyzer Type: spherical sector

Incident Angle: 55 °

Emission Angle: 0 °

Analyzer Pass Energy 30 eV

Analyzer Resolution: 0.5 eV

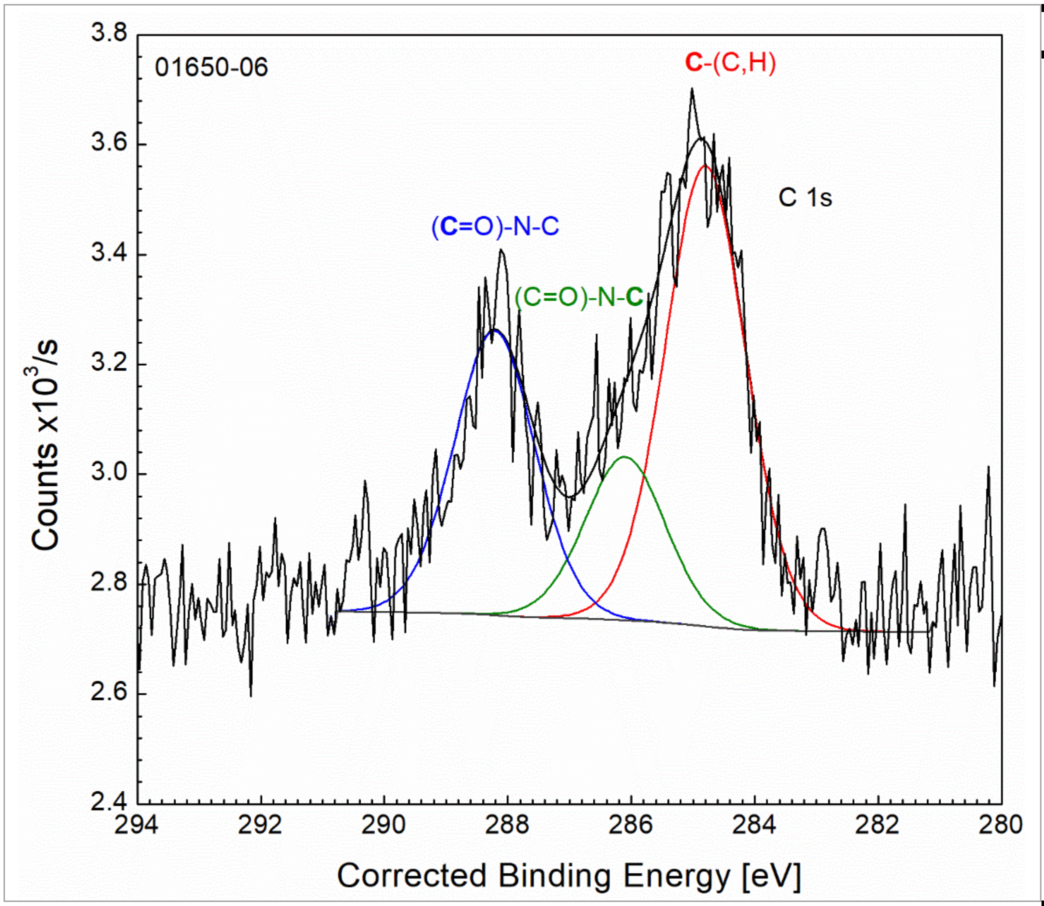
Total Signal Accumulation Time: 2752 s

Total Elapsed Time: 4047 s

Number of Scans: 25

Effective Detector Width: 2.64 eV

Energy Step: 0.05 eV



Publish in *SSS*: Yes ☒ No ☐

- **Accession #: 01650-06**
- **Host Material:** $Cs_{0.08}MA_{0.18}FA_{0.78}PbBr_{0.42}I_{2.58}$ perovskite
- **Technique:** XPS
- **Spectral Region:** C 1s

Instrument: SPECS PHOIBOS 150

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 100 W

Source Size: 2 mm x 2 mm

Analyzer Type: spherical sector

Incident Angle: 55 °

Emission Angle: 0 °

Analyzer Pass Energy 30 eV

Analyzer Resolution: 0.5 eV

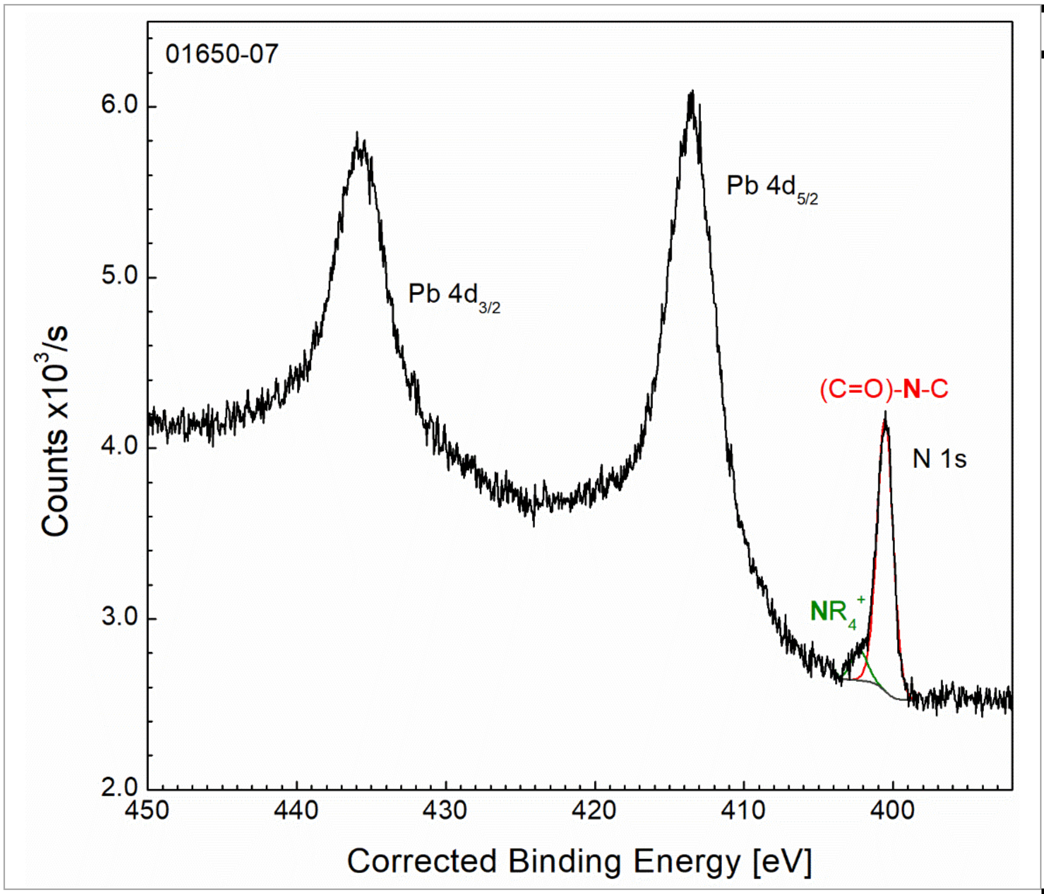
Total Signal Accumulation Time: 167 s

Total Elapsed Time: 246 s

Number of Scans: 4

Effective Detector Width: 2.64 eV

Energy Step: 0.05 eV



Publish in *SSS*: Yes ☒ No ☐

- **Accession #: 01650-07**
- **Host Material:** $Cs_{0.08}MA_{0.18}FA_{0.78}PbBr_{0.42}I_{2.58}$ perovskite
- **Technique:** XPS
- **Spectral Region:** N 1s, Pb 4d

Instrument: SPECS PHOIBOS 150

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 100 W

Source Size: 2 mm x 2 mm

Analyzer Type: spherical sector

Incident Angle: 55 °

Emission Angle: 0 °

Analyzer Pass Energy 30 eV

Analyzer Resolution: 0.5 eV

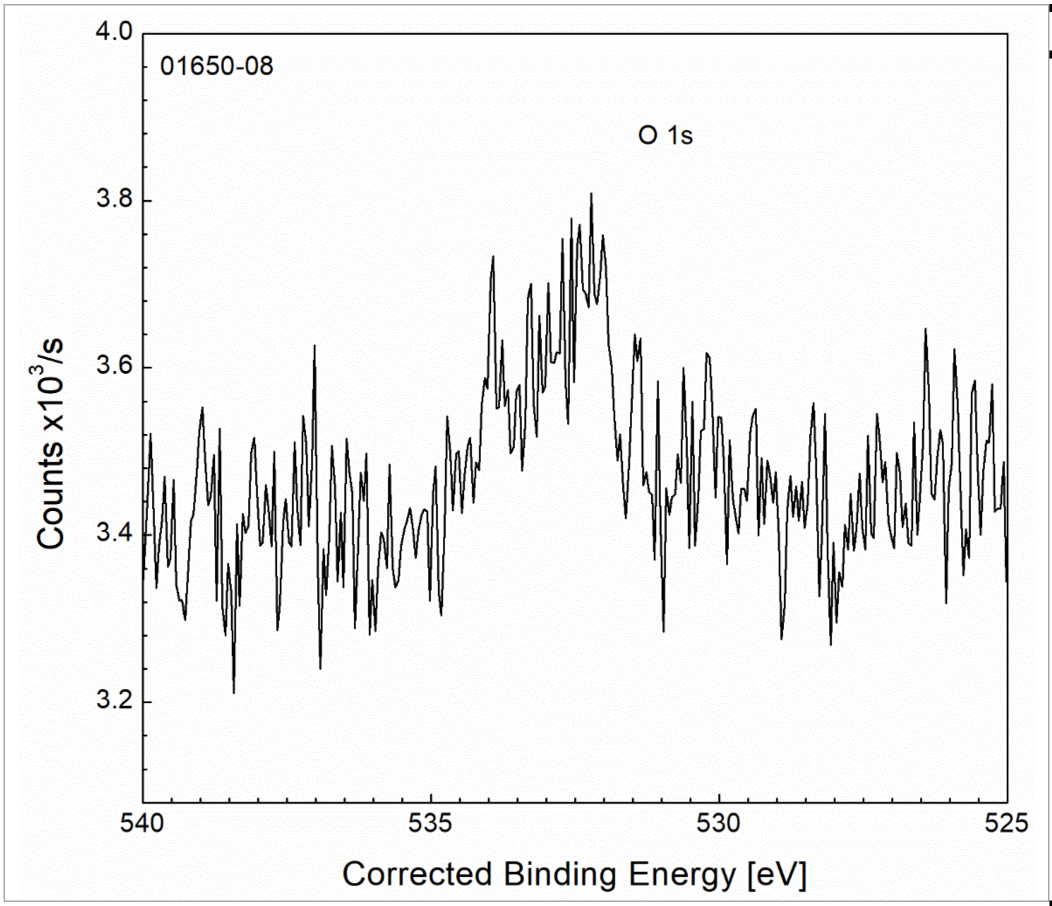
Total Signal Accumulation Time: 993 s

Total Elapsed Time: 1460 s

Number of Scans: 8

Effective Detector Width: 2.64 eV

Energy Step: 0.05 eV



Publish in *SSS*: Yes ☒ No ☐

■ Accession #: 01650-08

■ Host Material: $Cs_{0.08}MA_{0.18}FA_{0.78}PbBr_{0.42}I_{2.58}$ perovskite

■ Technique: XPS

■ Spectral Region: O 1s

Instrument: SPECS PHOIBOS 150

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 100 W

Source Size: 2 mm x 2 mm

Analyzer Type: spherical sector

Incident Angle: 55 °

Emission Angle: 0 °

Analyzer Pass Energy 30 eV

Analyzer Resolution: 0.5 eV

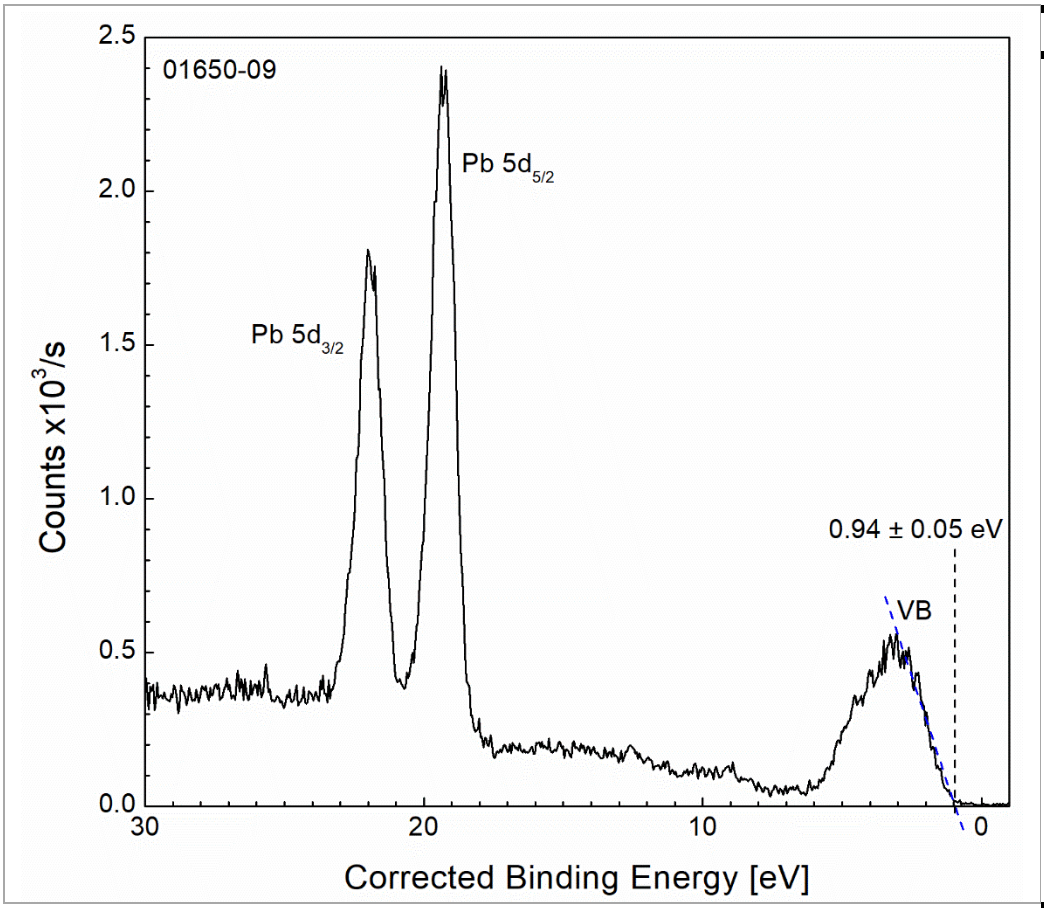
Total Signal Accumulation Time: 152 s

Total Elapsed Time: 224 s

Number of Scans: 4

Effective Detector Width: 2.64 eV

Energy Step: 0.05 eV



Publish in *SSS*: Yes ☒ No ☐

■ Accession #: 01650-09

■ Host Material: $Cs_{0.08}MA_{0.18}FA_{0.78}PbBr_{0.42}I_{2.58}$ perovskite

■ Technique: XPS

■ Spectral Region: VB, Pb 5d

Instrument: SPECS PHOIBOS 150

Excitation Source: Al Ka monochromatic

Source Energy: 1486.6 eV

Source Strength: 100 W

Source Size: 2 mm x 2 mm

Analyzer Type: spherical sector

Incident Angle: 55 °

Emission Angle: 0 °

Analyzer Pass Energy 30 eV

Analyzer Resolution: 0.5 eV

Total Signal Accumulation Time: 304 s

Total Elapsed Time: 447 s

Number of Scans: 4

Effective Detector Width: 2.64 eV

Energy Step: 0.05 eV