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High-energy x-ray photoelectron spectroscopy spectra of InP measured by Cr Kα

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High-energy x-ray photoelectron spectroscopy spectra of InP measured by Cr Kα

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Note: This paper is part of the 2022 Special Topic Collection on Higher Energy X-ray Photoelectron Spectroscopy.

ABSTRACT

Single crystal InP was analyzed using high-resolution high-energy x-ray photoelectron spectroscopy (HAXPES). The HAXPES spectra of InP obtained using monochromatic Cr Kα radiation at 5414.8 eV include two survey scans (Al Kα and Cr Kα) and high-resolution spectra of In 2s, In $2p_{3/2}$, In 3s, In 3p, In 3d, In 4s, In 4d, P 1s, P 2s, and P 2p.

Key words: InP, HAXPES, Cr Kα

Accession #: 01730 and 01743
Technique: XPS, XAES
Host Material: InP
Instrument: ULVAC-PHI Quantes
Major Elements in Spectra: In, P
Minor Elements in Spectra: None
Published Spectra: 12
Spectra in Electronic Record: 12
Spectral Category: Comparison

INTRODUCTION

While high-energy photoemission has been in use for decades, only very few reference spectra are available, even for common materials. The recent availability of performant lab-scale photoemission spectrometers (Refs. 1–3) requires reliable reference data.

Single crystal InP was analyzed using high-resolution high-energy x-ray photoelectron spectroscopy (HAXPES). The HAXPES spectra of InP obtained using monochromatic Cr Kα radiation at 5414.8 eV include two survey scans (Al Kα and Cr Kα) and high-resolution spectra of In 2s, In $2p_{3/2}$, In 3s, In 3p, In 3d, In 4s, In 4d, P 1s, P 2s, and P 2p.

SPECIMEN DESCRIPTION (ACCESSION # 01730)

Host Material: InP
CAS Registry #: 22398-80-7
Host Material Characteristics: Homogeneous; solid; single crystal; semiconductor; inorganic compound; other
Chemical Name: Indium phosphide
Source: Single crystal wafer purchased from AXT Inc. The n-type wafers had a (100) orientation and a carrier concentration in the range of $5–50 \times 10^{16}$ cm^{-3}.
Host Composition: In, P
Form: Bulk
Structure: Crystalline
History and Significance: Air exposed
As Received Condition: Piece of a wafer
Analyzed Region: Same as host materials
***Ex Situ* Preparation/Mounting:** Sample was taped on the sample holder using conducting double sided tape.
***In Situ* Preparation:** Before HAXPES measurement, the samples were cleaned with low energy Ar^+ ions (100 eV). The cleaning procedure was stopped when the carbon level was reduced to a negligible level using Al Kα radiation.
Charge Control: The charge neutralization has been performed using a dual beam setup combining a low energy electron beam (1 eV, filament 1.1 A) and low energy ion beam (10 eV, 5 mA emission).

Temp. During Analysis: 300 K
Pressure During Analysis: $<5 \times 10^{-7}$ Pa
Pre-analysis Beam Exposure: 0 s

SPECIMEN DESCRIPTION (ACCESSION # 01743)

Host Material: Au
CAS Registry #: 7440-57-5
Host Material Characteristics: Homogeneous; solid; amorphous; conductor; metal; other
Chemical Name: Gold
Source: 0.250 mm thick foil from Goodfellow, AU000372/5
Host Composition: Au
Form: Bulk
Structure: Amorphous
History and Significance: In-vacuum sputtered
As Received Condition: Foil
Analyzed Region: Same as host materials
***Ex Situ* Preparation/Mounting:** The sample was taped on the sample holder using insulating removable 3M double sided tape.
***In Situ* Preparation:** Sample was sputtered using 1 keV Ar^+ ions until no C or O was observed.
Charge Control: Low energy electrons (1 eV, filament 1.1 A) and low energy ions (10 eV, 5 mA emission)
Temp. During Analysis: 300 K
Pressure During Analysis: $<5 \times 10^{-7}$ Pa
Pre-analysis Beam Exposure: 0 s

INSTRUMENT DESCRIPTION

Manufacturer and Model: ULVAC-PHI Quantes
Analyzer Type: Spherical sector
Detector: Multichannel resistive plate
Number of Detector Elements: 32

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

Spectrometer

Analyzer Mode: Constant pass energy
Throughput ($T = E^N$): The energy dependence can be modeled using the following equation: $A/E_p = (a^2/(a^2 + R^2))^b$, where a and b are constants, E_p is the pass energy, A is the peak area, and R is the retard ratio equal to E/E_p, where E is the kinetic energy. Three spectral regions [Ag 2s (3790–3830 eV), Ag 3s (700–740 eV), and Ag 3d (350–390 eV)] are recorded on a sputter-cleaned silver sample at different pass energies. The values of a and b are then determined to be 576.9 and 6.3, respectively, by a linear least square fit of the data applying the equation described above (Ref. 4).
Excitation Source Window: Al
Excitation Source: Cr K_α monochromatic
Source Energy: 5414.8 eV
Source Strength: 52 W
Source Beam Size: $100 \times 100\ \mu m^2$
Signal Mode: Multichannel direct

Geometry

Incident Angle: 22°
Source-to-Analyzer Angle: 46°
Emission Angle: 45°
Specimen Azimuthal Angle: 0°
Acceptance Angle from Analyzer Axis: 0°
Analyzer Angular Acceptance Width: 20° × 20°

Ion Gun

Manufacturer and Model: ULVAC-PHI Quantes
Energy: 10 and 100 eV
Current: 5 mA
Current Measurement Method: Biased stage
Sputtering Species: Ar
Spot Size (unrastered): 10,000 μm
Raster Size: N/A
Incident Angle: 45°
Polar Angle: 45°
Azimuthal Angle: 45°
Comment: Gun used for neutralization

DATA ANALYSIS METHOD

Energy Scale Correction: Adsorbed air-born carbon contamination cannot be used for binding energy calibration due to its very low intensity in HAXPES. An alternative energy scale correction was developed by measuring the C 1s and In $3d_{5/2}$ peaks with Al Kα radiation and correcting the position of the In 3 $d_{5/2}$ peak toward the C 1s peak at 284.8 eV. Subsequently, the Al Kα determined In $3d_{5/2}$ position was used to calibrate the Cr Kα data. The determined binding energy position of In $3d_{5/2}$ using Al Kα is 444.24 eV. The binding energy shift of the Cr Kα data was then performed by shifting the In $3d_{5/2}$ peak to 444.24 eV.

Recommended Energy Scale Shift: 0.7 eV for binding energy

Peak Shape and Background Method: Data treatment was performed using Shirley background and Gaussian–Lorentzian peak shapes.

Quantitation Method: Quantification was done using PHI MULTIPAK Software Version 9.9.0.8. Theoretical sensitivity factors were provided by the software. Theoretical relative sensitivity factors have been calculated with consideration of matrix effects. Inelastic mean free paths were calculated by TPP-2M for the average matrix sample. Elastic-electron correction was calculated by a method based on ISO18118:2015. Back-scattering factor was ignored. Photoionization cross sections and asymmetry parameters were referred to by Atomic Data and Nuclear Data Tables (Refs. 5 and 6) and Scofield (Ref. 7). Photoionization cross section of high kinetic energy region of excited by Cr Kα was extrapolated based on a method developed by Verner *et al.* (Refs. 8 and 9).

AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

DATA AVAILABILITY

The data that support the findings of this study are available within the article and its supplementary material (Ref. 10).

REFERENCES

[1]See: https://www.ulvac-phi.com/en/products/xps/quantes/.
[2]See: https://www.kratos.com/products/axis-supra-xps-surface-analysis-instrument.
[3]See: https://scientaomicron.com/en/system-solutions/electron-spectroscopy/HAXPES-Lab.
[4]Ulvac-PHI, private communication (March 3, 2021).
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[9]D. A. Verner, G. J. Ferland, K. T. Korista, and D. G. Yakovlev, Astrophys. J. **465**, 487 (1996).
[10]See supplementary material at https://doi.org/10.1116/6.0001521 for ASCII data of all shown spectra..

SPECTRAL FEATURES TABLE

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × counts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01730-03	In 2s	4238.95	3.88	7860	4.831	…	InP
01730-04	In $2p_{3/2}$	3731.32	2.83	33 138	15.791	…	InP
01730-05	In 3s	827.23	8.08	3928	2.986	…	InP
01730-06	In $3p_{3/2}$	665.52	3.51	9759	4.997	…	InP
01730-07	In $3d_{5/2}$	444.24	1.34	4371	1.849	56.7	InP
01730-07	In $3d_{3/2}$	451.83	1.32	3034	1.22	…	InP
01730-08	In 4s	123.21	4.63	1061	0.665	…	InP
01730-08	P 2p	128.85	1.68	281	0.141	43.3	InP
01730-09	In 4d	17.66	1.89	997	0.414	…	InP
01730-10	P 1s	2142.19	1.37	6989	5.548	…	InP
01730-11	P 2s	186.34	1.91	718	0.493	…	InP

ANALYZER CALIBRATION TABLE[a]

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × counts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01743-01	Au $3d_{5/2}$	2206.8	2.59	47 379	…	…	…
01743-01	Au $4f_{7/2}$	84.2	1.24	2115	…	…	…
…	Cu $2p_{3/2}$	932.9	1.34	7090	…	…	…
…	Ag $2p_{3/2}$	3352.7	2.61	30 352	…	…	…
…	Ag $3d_{5/2}$	368.4	1.15	2978	…	…	…

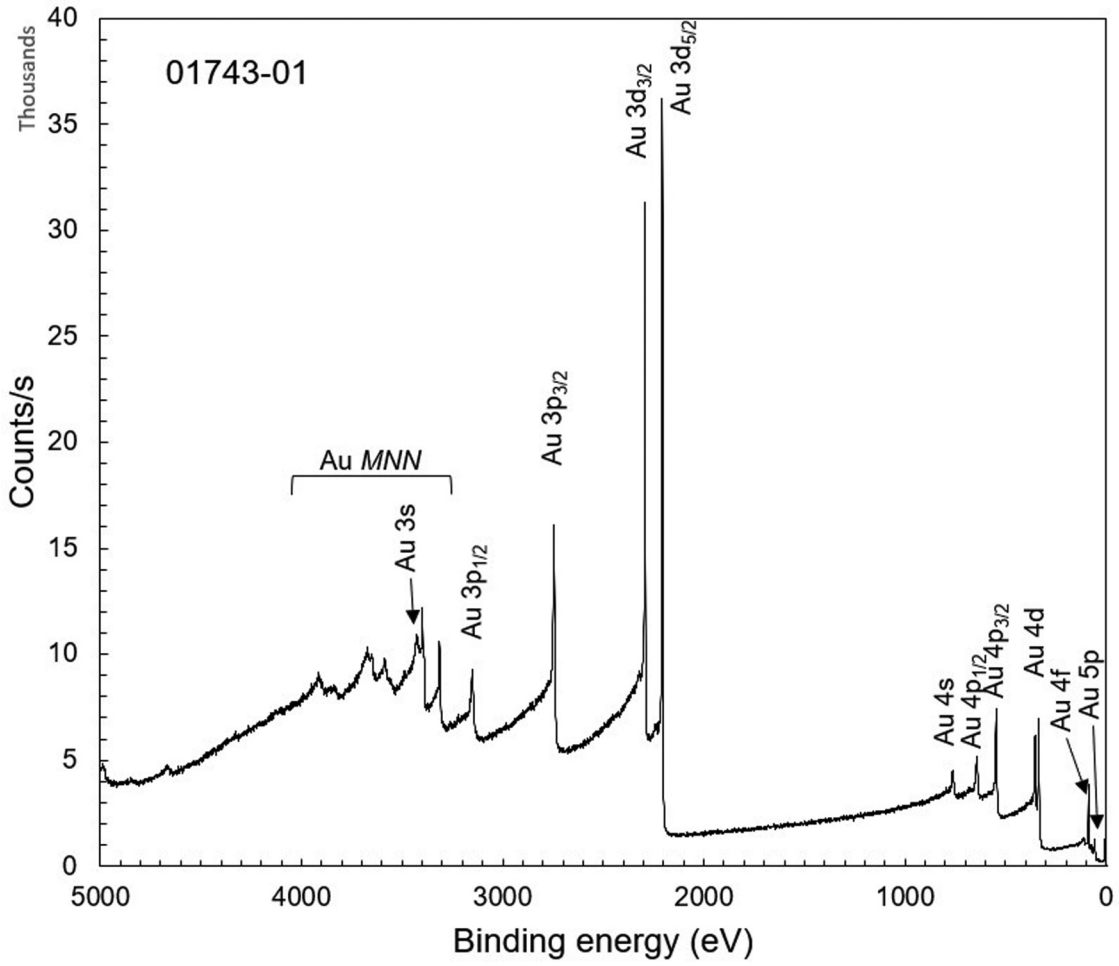
[a]The calibration table is established using the Cr Kα photons and a pass energy of 112 eV corresponding to the presented high-resolution spectra.

GUIDE TO FIGURES

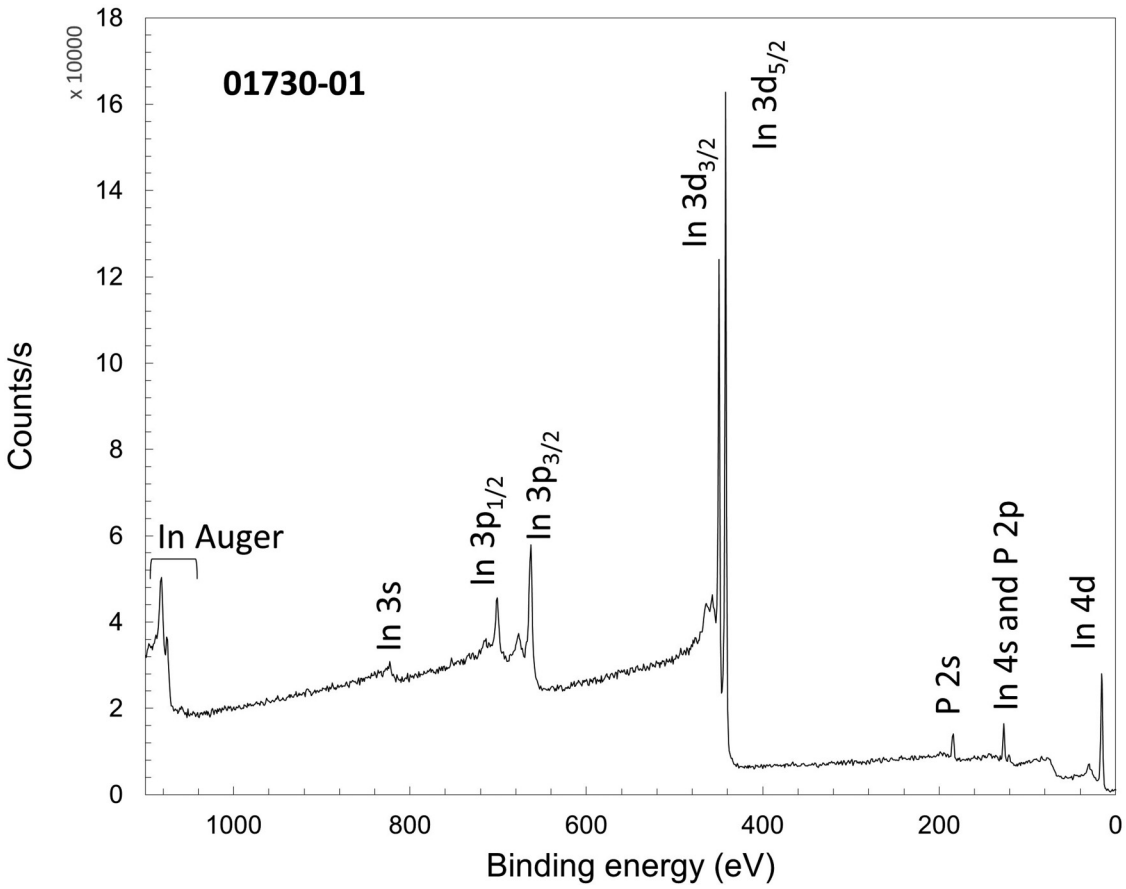
Spectrum (Accession) #	Spectral Region	Voltage Shift[a] (eV)	Multiplier	Baseline	Comment #
01743-01	Survey	0	1	0	Au with x-ray source Cr Kα[b]
01730-01	Survey	0	1	0	X-ray source Al Kα
01730-02	Survey	0	1	0	X-ray source Cr Kα
01730-03	In 2s	−0.7	1	0	…
01730-04	In $2p_{3/2}$	−0.7	1	0	…
01730-05	In 3s	−0.7	1	0	…
01730-06	In $3p_{3/2}$	−0.7	1	0	…
01730-07	In 3d	−0.7	1	0	…
01730-08	In 4s and P 2p	−0.7	1	0	…
01730-09	In 4d	−0.7	1	0	…
01730-10	P 1s	−0.7	1	0	…
01730-11	P 2s	−0.7	1	0	…

[a]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.

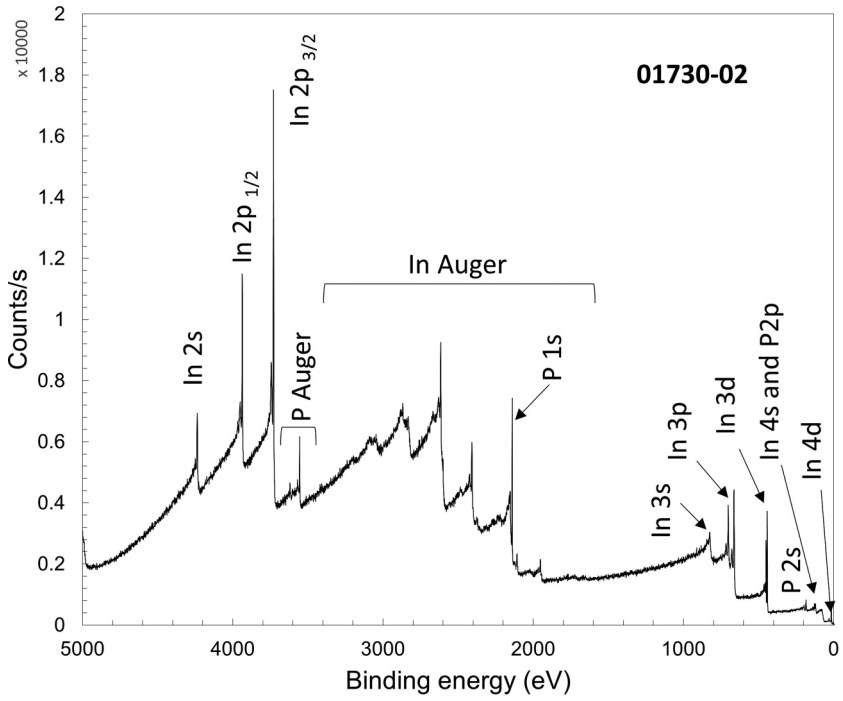
[b]The reference spectrum included in this paper is the same as those included with other papers in this collection that use this x-ray source. This was intentional and not an error as submissions were requested to be accompanied by a wide-scan spectrum of sputter-cleaned gold taken on the same instrument and using the same settings as the wide-scan spectrum of the material, or materials in the submission.



Accession #	01743-01
Host Material:	Au
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Cr K_α monochromatic
Source Energy:	5414.8 eV
Source Strength:	49 W
Source Size:	0.1 × 0.1 mm^2
Analyzer Type:	Spherical sector analyzer
Incident Angle:	22°
Emission Angle:	45°
Analyzer Pass Energy:	280 eV
Analyzer Resolution:	1.9 eV
Total Signal Accumulation Time:	5210 s
Total Elapsed Time:	5700 s
Number of Scans:	1
Effective Detector Width:	31 eV

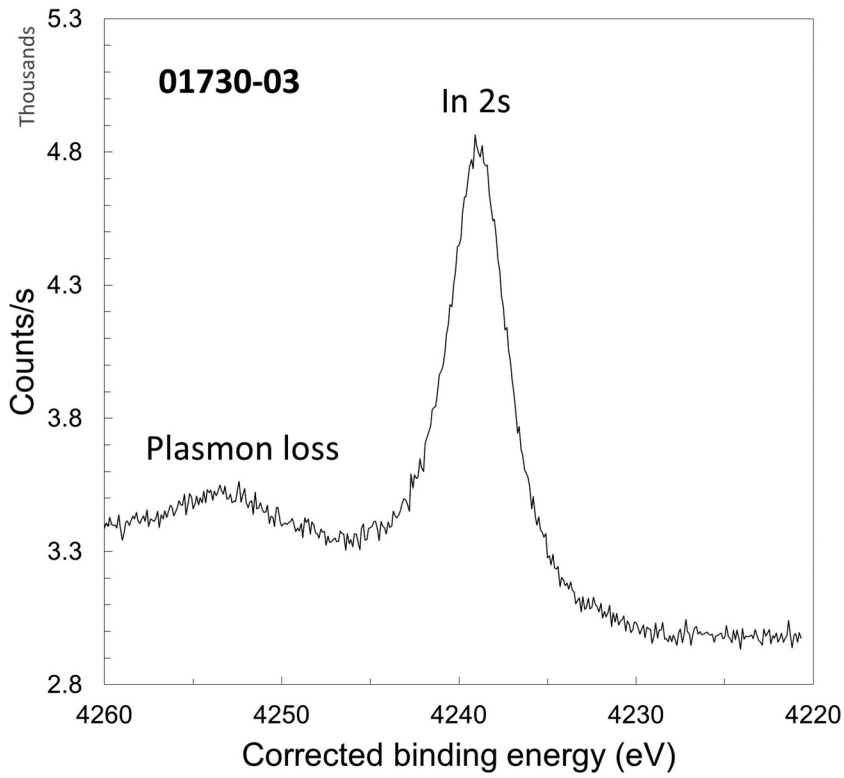


Accession #	01730-01
Host Material:	InP
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Al K_α monochromatic
Source Energy:	1486.6 eV
Source Strength:	25 W
Source Size:	0.1 × 0.1 mm^2
Analyzer Type:	Spherical sector analyzer
Incident Angle:	22°
Emission Angle:	45°
Analyzer Pass Energy:	280 eV
Analyzer Resolution:	1.9 eV
Total Signal Accumulation Time:	110 s
Total Elapsed Time:	130 s
Number of Scans:	1
Effective Detector Width:	31 eV



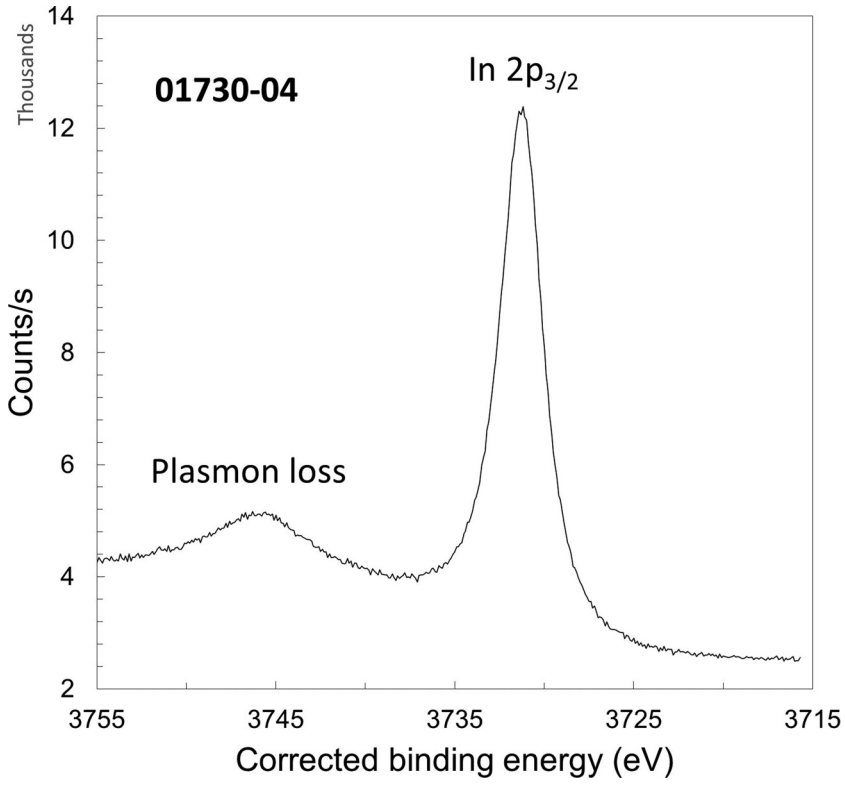
- **Accession #:** 01730-02
- **Host Material:** InP
- **Technique:** XPS
- **Spectral Region:** Survey

Instrument: ULVAC-PHI Quantes
Excitation Source: Cr K_α monochromatic
Source Energy: 5414.8 eV
Source Strength: 43 W
Source Size: 0.1 × 0.1 mm^2
Analyzer Type: Spherical sector analyzer
Incident Angle: 22°
Emission Angle: 45°
Analyzer Pass Energy: 280 eV
Analyzer Resolution: 1.9 eV
Total Signal Accumulation Time: 5000 s
Total Elapsed Time: 5500 s
Number of Scans: 10
Effective Detector Width: 31 eV



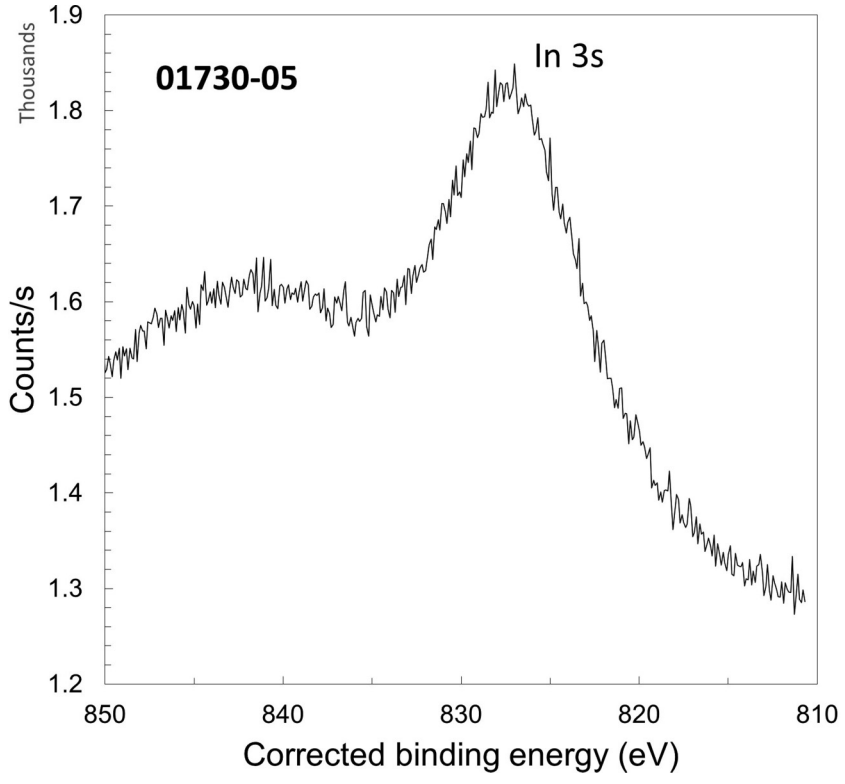
- **Accession #:** 01730-03
- **Host Material:** InP
- **Technique:** XPS
- **Spectral Region:** In 2s

Instrument: ULVAC-PHI Quantes
Excitation Source: Cr K_α monochromatic
Source Energy: 5414.8 eV
Source Strength: 52 W
Source Size: 0.1 × 0.1 mm^2
Analyzer Type: Spherical sector
Incident Angle: 22°
Emission Angle: 45°
Analyzer Pass Energy: 112 eV
Analyzer Resolution: 0.86 eV
Total Signal Accumulation Time: 2890 s
Total Elapsed Time: 3175 s
Number of Scans: 72
Effective Detector Width: 12.4 eV



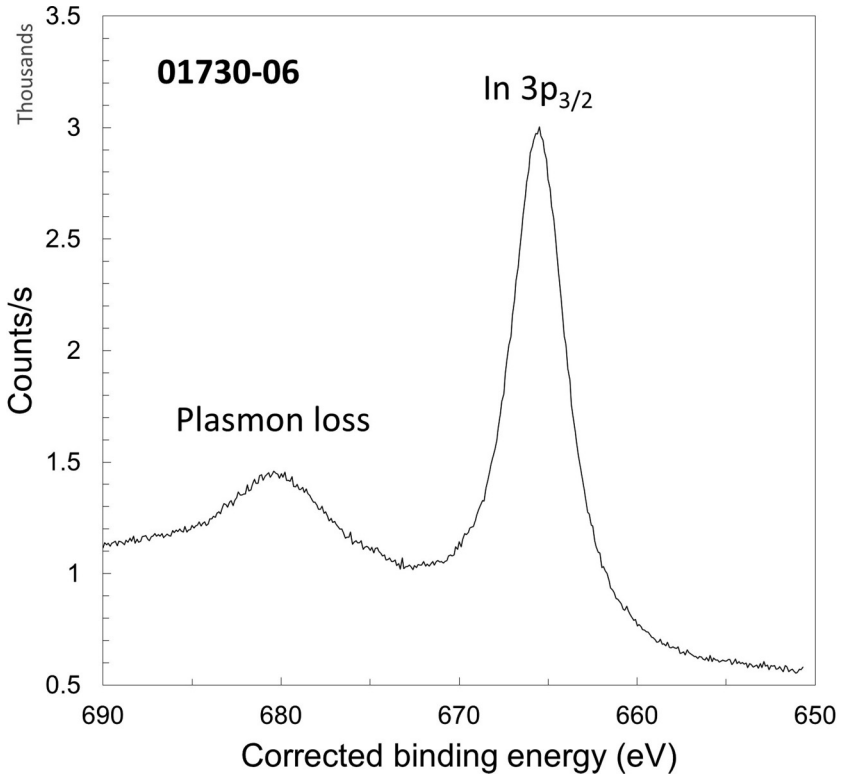
- **Accession #:** 01730-04
- **Host Material:** InP
- **Technique:** XPS
- **Spectral Region:** In

Instrument: ULVAC-PHI Quantes
Excitation Source: Cr K_α monochromatic
Source Energy: 5414.8 eV
Source Strength: 52 W
Source Size: 0.1 × 0.1 mm^2
Analyzer Type: Spherical sector
Incident Angle: 22°
Emission Angle: 45°
Analyzer Pass Energy: 112 eV
Analyzer Resolution: 0.86 eV
Total Signal Accumulation Time: 1450 s
Total Elapsed Time: 1450 s
Number of Scans: 36
Effective Detector Width: 12.4 eV



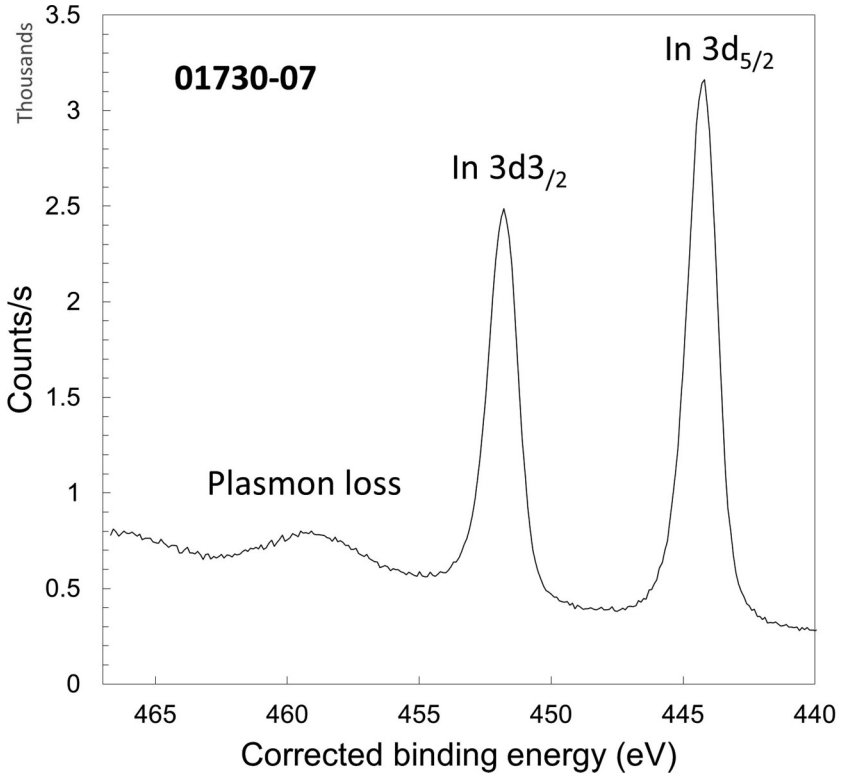
- **Accession #:** 01730-05
- **Host Material:** InP
- **Technique:** XPS
- **Spectral Region:** In 3s

Instrument: ULVAC-PHI Quantes
Excitation Source: Cr K_α monochromatic
Source Energy: 5414.8 eV
Source Strength: 52 W
Source Size: 0.1 × 0.1 mm^2
Analyzer Type: Spherical sector
Incident Angle: 22°
Emission Angle: 45°
Analyzer Pass Energy: 112 eV
Analyzer Resolution: 0.86 eV
Total Signal Accumulation Time: 3530 s
Total Elapsed Time: 3530 s
Number of Scans: 88
Effective Detector Width: 12.4 eV



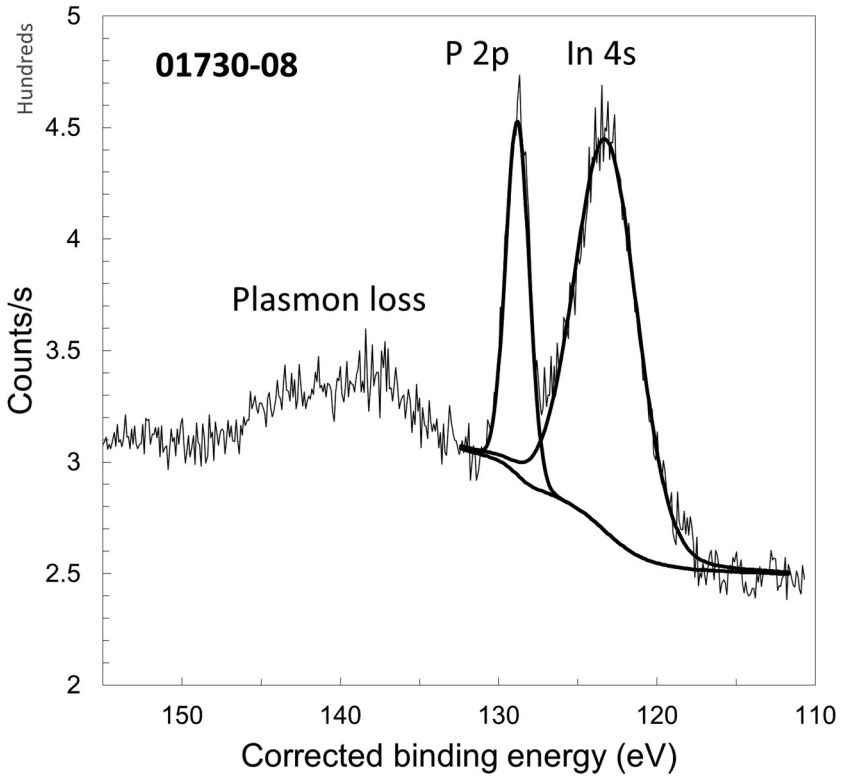
- **Accession #:** 01730-06
- **Host Material:** InP
- **Technique:** XPS
- **Spectral Region:** In $3p_{3/2}$

Instrument: ULVAC-PHI Quantes
Excitation Source: Cr K_α monochromatic
Source Energy: 5414.8 eV
Source Strength: 52 W
Source Size: 0.1 × 0.1 mm^2
Analyzer Type: Spherical sector
Incident Angle: 22°
Emission Angle: 45°
Analyzer Pass Energy: 112 eV
Analyzer Resolution: 0.86 eV
Total Signal Accumulation Time: 3530 s
Total Elapsed Time: 3530 s
Number of Scans: 88
Effective Detector Width: 12.4 eV



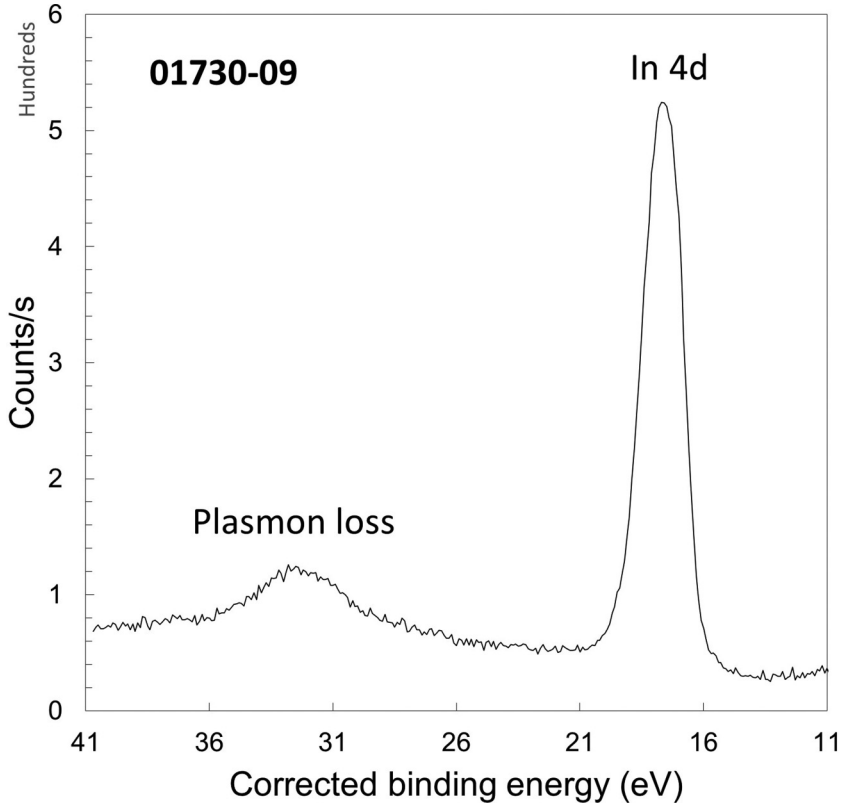
- **Accession #:** 01730-07
- **Host Material:** InP
- **Technique:** XPS
- **Spectral Region:** In 3d

Instrument: ULVAC-PHI Quantes
Excitation Source: Cr K_α monochromatic
Source Energy: 5414.8 eV
Source Strength: 52 W
Source Size: 0.1 × 0.1 mm^2
Analyzer Type: Spherical sector
Incident Angle: 22°
Emission Angle: 45°
Analyzer Pass Energy: 112 eV
Analyzer Resolution: 0.86 eV
Total Signal Accumulation Time: 2475 s
Total Elapsed Time: 2475 s
Number of Scans: 112
Effective Detector Width: 12.4 eV



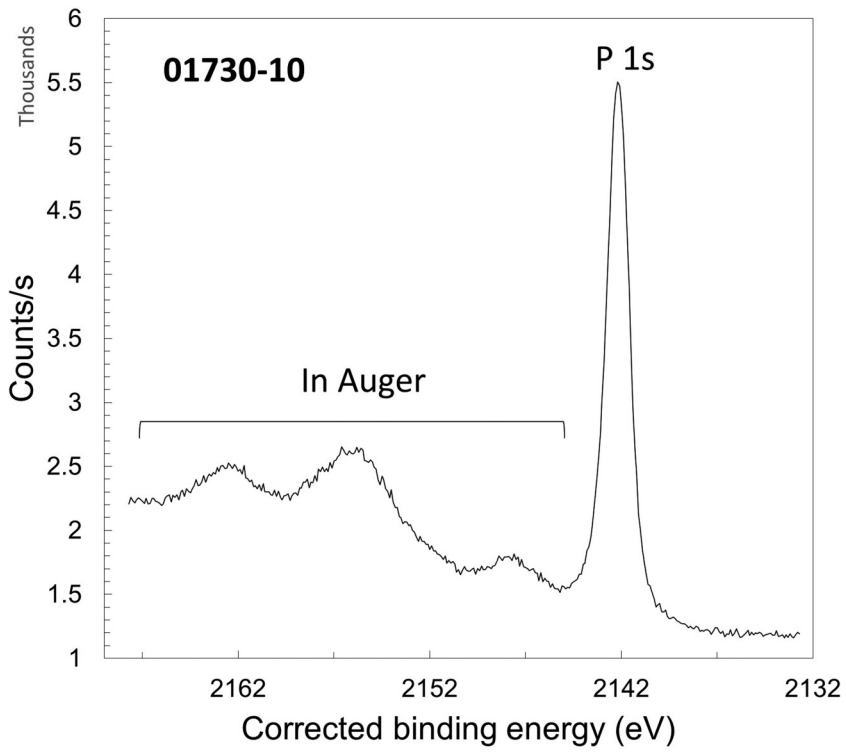
- **Accession #:** 01730-08
- **Host Material:** InP
- **Technique:** XPS
- **Spectral Region:** In 4s and P 2p

Instrument: ULVAC-PHI Quantes
Excitation Source: Cr K_α monochromatic
Source Energy: 5414.8 eV
Source Strength: 52 W
Source Size: 0.1 × 0.1 mm^2
Analyzer Type: Spherical sector
Incident Angle: 22°
Emission Angle: 45°
Analyzer Pass Energy: 112 eV
Analyzer Resolution: 0.86 eV
Total Signal Accumulation Time: 3600 s
Total Elapsed Time: 3600 s
Number of Scans: 80
Effective Detector Width: 12.4 eV



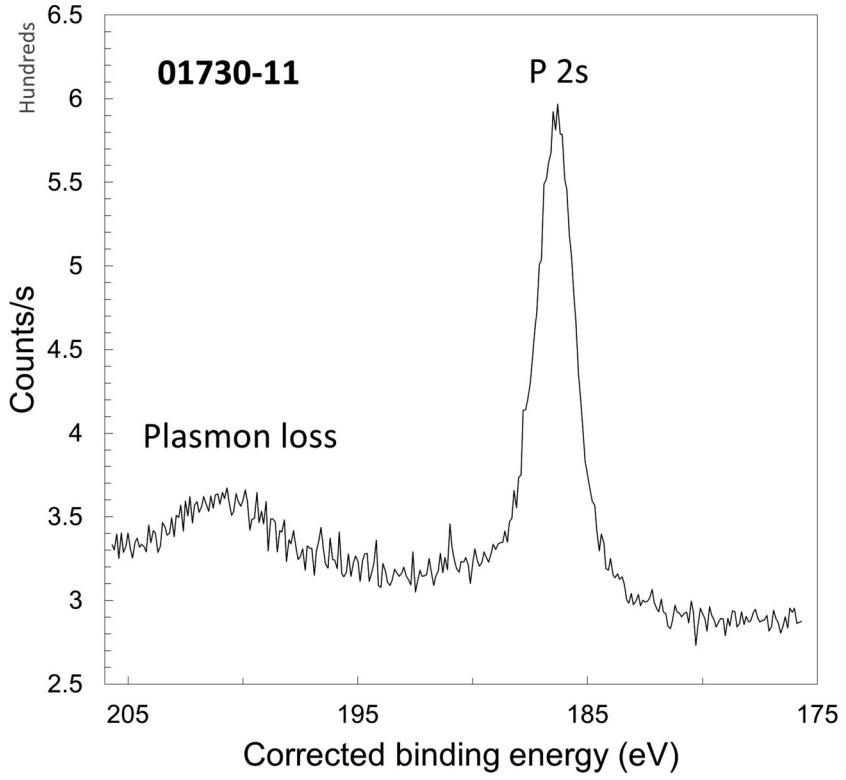
- **Accession #:** 01730-09
- **Host Material:** InP
- **Technique:** XPS
- **Spectral Region:** In 4d

Instrument: ULVAC-PHI Quantes
Excitation Source: Cr K_α monochromatic
Source Energy: 5414.8 eV
Source Strength: 52 W
Source Size: 0.1 × 0.1 mm^2
Analyzer Type: Spherical sector
Incident Angle: 22°
Emission Angle: 45°
Analyzer Pass Energy: 112 eV
Analyzer Resolution: 0.86 eV
Total Signal Accumulation Time: 3370 s
Total Elapsed Time: 3370 s
Number of Scans: 112
Effective Detector Width: 12.4 eV



- **Accession #:** 01730-10
- **Host Material:** InP
- **Technique:** XPS, XAES
- **Spectral Region:** P 1s

Instrument: ULVAC-PHI Quantes
Excitation Source: Cr K_α monochromatic
Source Energy: 5414.8 eV
Source Strength: 52 W
Source Size: 0.1 × 0.1 mm^2
Analyzer Type: Spherical sector
Incident Angle: 22°
Emission Angle: 45°
Analyzer Pass Energy: 112 eV
Analyzer Resolution: 0.86 eV
Total Signal Accumulation Time: 1545 s
Total Elapsed Time: 1545 s
Number of Scans: 44
Effective Detector Width: 12.4 eV



- **Accession #:** 01730-11
- **Host Material:** InP
- **Technique:** XPS
- **Spectral Region:** P 2s

Instrument: ULVAC-PHI Quantes
Excitation Source: Cr K_α monochromatic
Source Energy: 5414.8 eV
Source Strength: 52 W
Source Size: 0.1 × 0.1 mm^2
Analyzer Type: Spherical sector
Incident Angle: 22°
Emission Angle: 45°
Analyzer Pass Energy: 112 eV
Analyzer Resolution: 0.86 eV
Total Signal Accumulation Time: 3370 s
Total Elapsed Time: 3370 s
Number of Scans: 112
Effective Detector Width: 12.4 eV