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KBr Crystal: Cleaved in Air

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A crystal of infrared-grade potassium bromide (KBr) was cleaved in air using a clean single-edged razor blade. The crystal was adhered to double-sided adhesive tape and analyzed by XPS at an angle that maximizes the information associated with subsurface bulk. The crystal's dimensions were 3 mm × 3 mm × 10 mm ($w \times h \times l$), and only the freshly exposed surface was analyzed. A mesh made of nickel wire was placed about 1 mm above the surface of the sample to flatten the electric fields on the sample.

Keywords: XPS; K; Br; crystal; nonconductive; insulator

PACS: 79.60.Eq, 82.80.Pv

Accession #: 00046
Technique: XPS
Host Material: KBr
Instrument: Surface Science Instruments X-Probe 206
Major Elements in Spectrum: K, Br
Minor Elements in Spectrum: C
Printed Spectra: 4
Spectra in Electronic Record: 4
Spectral Category: comparison

SPECIMEN DESCRIPTION

Host Material: KBr

CAS Registry #: 7758-02-3

Host Material Characteristics: homogeneous; solid; single crystal; dielectric; inorganic compound

Chemical Name: potassium bromide

Source: unknown

Host Composition: K, Br

Form: single crystal

Lot #: N/A

Structure: N/A

History Significance: N/A

As Received Condition: as grown, stored in closed bottle

Analyzed Region: freshly exposed "bulk" surface

***Ex Situ* Preparation/Mounting:** The specimen was cleaved in air using a freshly cleaned single-edged razor blade, and mounted with double-sided adhesive tape. A charge control mesh was placed ~1 mm above surface.

***In Situ* Preparation:** no treatments

Pre-Analysis Beam Exposure: kept in an analytical chamber at $\sim 10^{-9}$ Torr for 17 h before analysis

Charge Control: An electron "flood" gun (~3 eV, 1 mA) at normal incidence, and a charge control metal (Ni) mesh at a 1 mm height from the sample surface were used to control charging.

Temp. During Analysis: 320 K

Pressure During Analysis: $< 1.00 \times 10^{-6}$ Pa

SPECTROMETER DESCRIPTION

Manufacturer and Model: Surface Science Instruments X-Probe 206

Analyzer Type: spherical sector

Detector: 128 channel

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

Spectrometer

Analyzer Mode: constant pass energy

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

Excitation Source: Al K_α monochromatic

Excitation Source Window: 1 μm Mylar

Source Energy: 1486.6 eV

Analyzer Width at 1000 eV: 2000 μm × 2000 μm

Signal Mode: multichannel direct

Simultaneous Channels: 128

Geometry

Incident Angle: 20°

Source to Analyzer Angle: 71°

Emission Angle: 0°

Specimen Azimuthal Angle: 90°

Acceptance Angle from Analyzer Axis: 0°

Analyzer Angular Acceptance Width: 30° × 30° at 1000 eV

Comments: Sampling depth was maximized to provide data which represents subsurface bulk.

DATA ANALYSIS METHOD

Energy Scale Correction: All peak energies were adjusted such that the adventitious hydrocarbon C (1*s*) BE equaled 285.0 eV.

Peak Shape and Background Method: A Shirley function was used to define the baseline of the background and Voight peak shapes (80:20 Gaussian–Lorentzian) were used to define the shape of each peak. Peak area ratios were constrained to the theoretical values.

Quantitation Method: Peak areas were used to calculate atomic percentages. Relative sensitivity factors were calculated by modifying Scofield values according to the following equa-

tion: RSF(X-Probe) = RSF(Scofield, $C=1.0$) $\times$ [(1487 − observed BE)/(1487−285)]$^{1.0}$, where 1.0 is an exponent. A 150 eV Pass Energy was used for data collection.

REFERENCES

1. J. H. Scofield, J. Electron. Spectrosc. Relat. Phenom. **8**, 129 (1976).

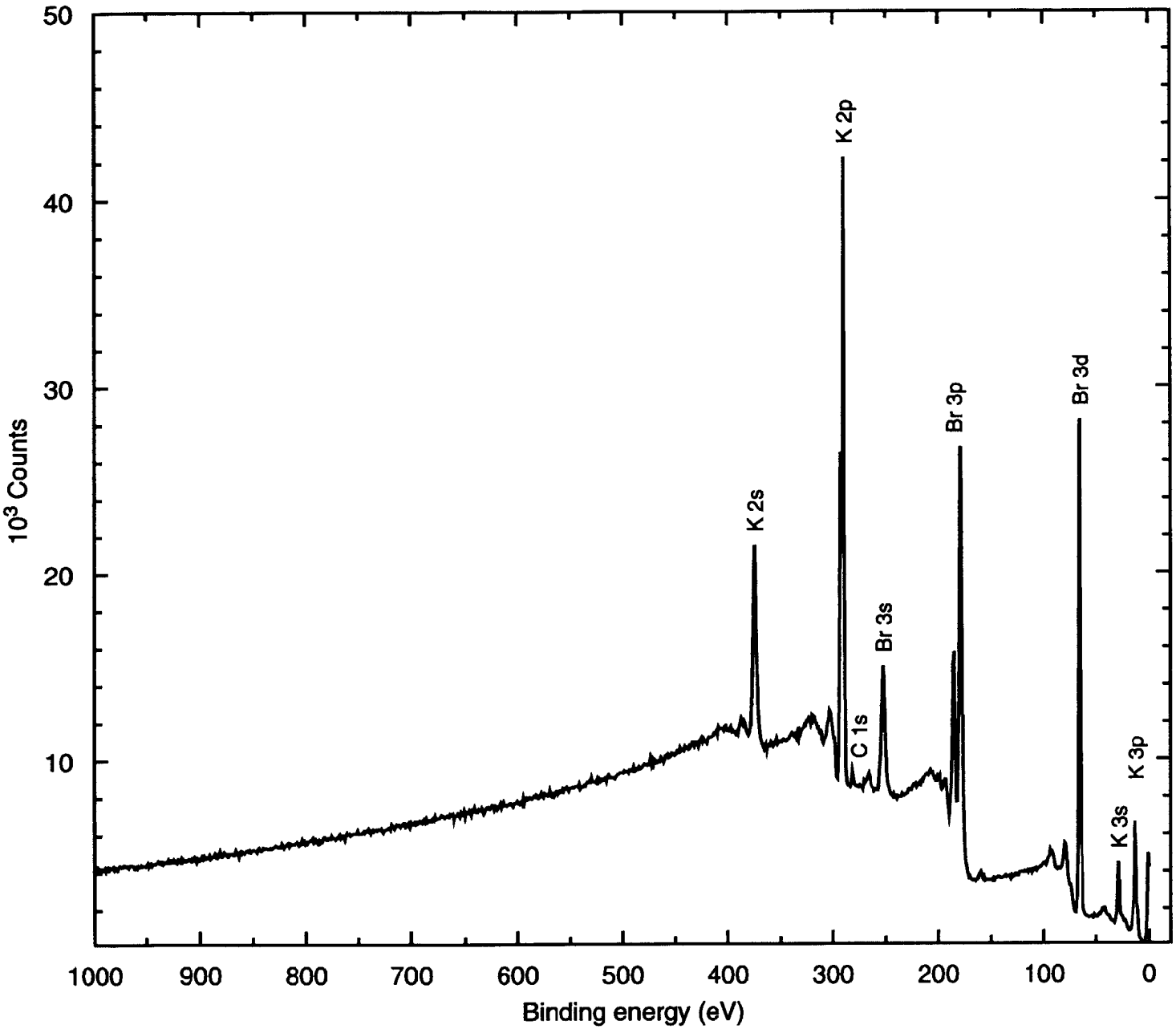
SPECTRAL FEATURES TABLE

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (counts)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
1	Br $3d_{5/2,3/2}$	67	...	169762	3.36	49.6	Br
1	K $2s$	377	...	100049	2.11	46.7	K
1	C $1s$	285	...	3787	1.00	3.7	C
2	Br $3d_{5/2}$	68.5	0.9	...	...	...	Br
2	Br $3d_{3/2}$	69.6	0.96	...	...	...	Br
3	K $2p_{3/2}$	293.2	1.09	...	...	...	K
3	K $2p_{1/2}$	296.0	1.07	...	...	...	K
4	C $1s$	285.0	1.68	...	...	...	C

ANALYZER CALIBRATION TABLE

Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (counts)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
...	Cu $2p_{3/2}$	932.47±0.08	...	...	...	...	...
...	Cu $3s$	122.39±0.08	...	...	...	...	...
...	Au $4f_{7/2}$	83.96±0.08	...	...	...	...	...
...	Au $4f_{5/2}$	87.64±0.08	...	...	...	...	...

Footnote to Analyzer Calibration Table: All of these BEs are used to calibrate the energy scale of this XPS system.



- **Accession #:** 00046-01
- **Host Material:** KBr
- **Technique:** XPS
- **Spectral Region:** survey

Instrument: Surface Science Instruments X-Probe 206

Excitation Source: Al K_α monochromatic

Source Energy: 1486.6 eV

Source Strength: 100 W

Source Size: 600 μm × 600 μm

Incident Angle: 20°

Analyzer Type: spherical sector

Analyzer Pass Energy: 150 eV

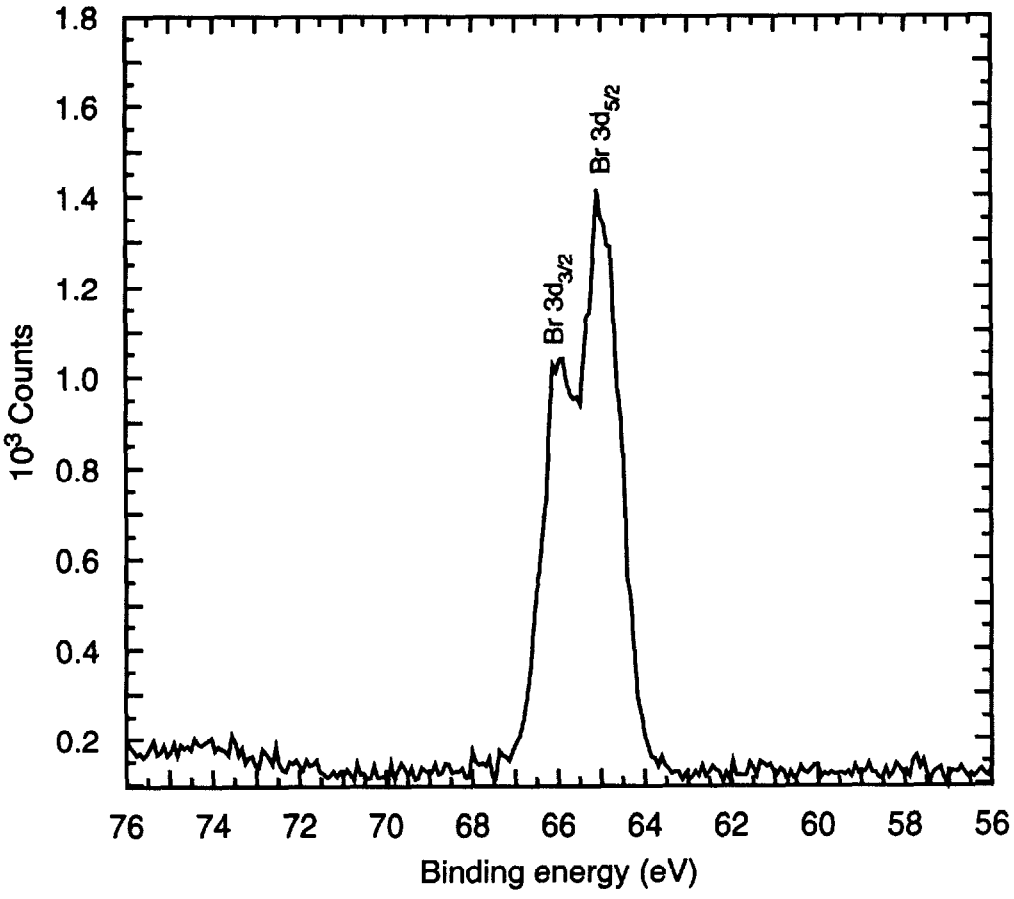
Analyzer Resolution: 0.68 eV

Emission Angle: 0°

Data Acquisition Time: 1260 s

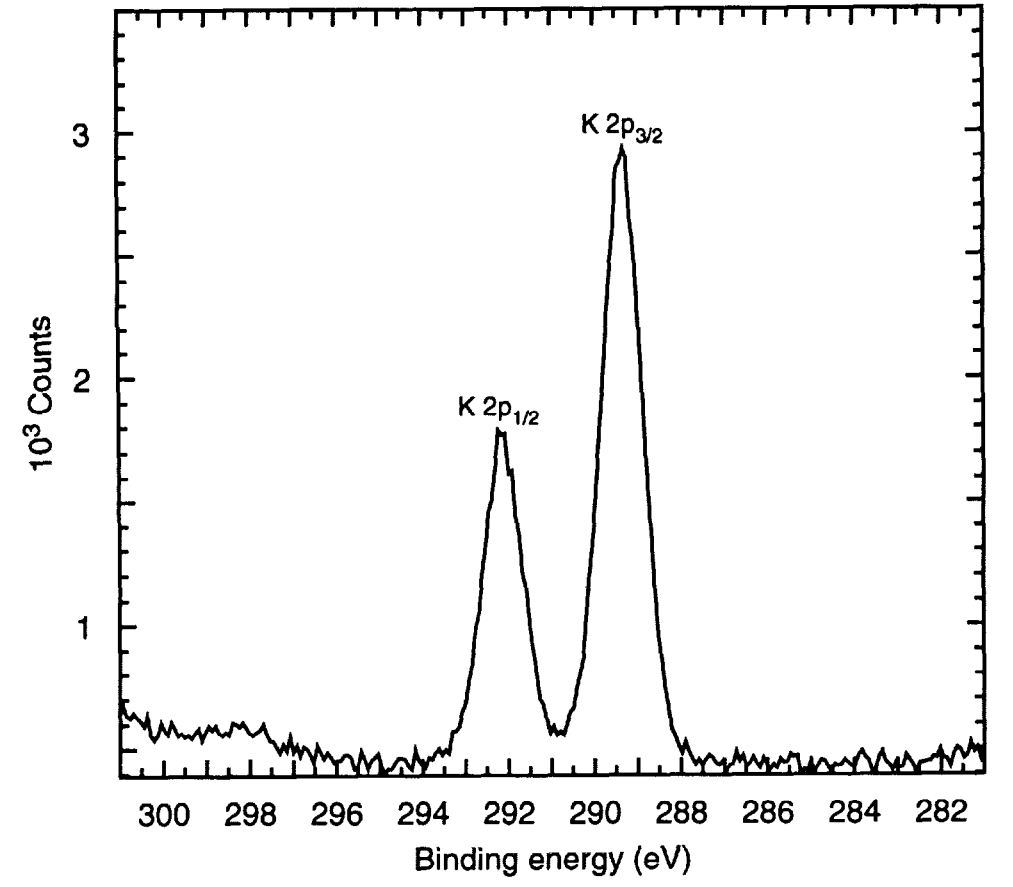
Dead Time Correction: not specified

Number of Scans: 4



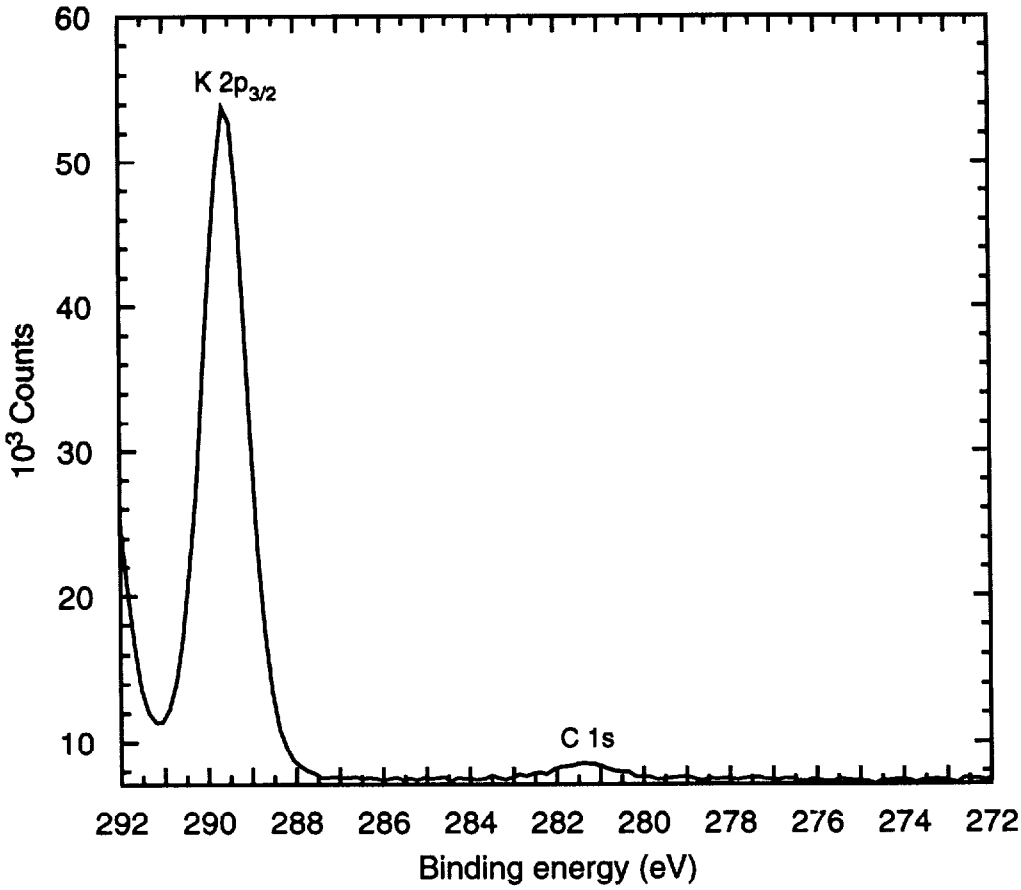
- **Accession #:** 00046-02
- **Host Material:** KBr
- **Technique:** XPS
- **Spectral Region:** Br $3d_{5/2}$; Br $3d_{3/2}$

Instrument: Surface Science Instruments X-Probe 206
Excitation Source: Al K_α monochromatic
Source Energy: 1486.6 eV
Source Strength: 10 W
Source Size: 150 μm × 150 μm
Incident Angle: 20°
Analyzer Type: spherical sector
Analyzer Pass Energy: 25 eV
Analyzer Resolution: 0.25 eV
Emission Angle: 0°
Data Acquisition Time: 1020 s
Dead Time Correction: not specified
Number of Scans: 20
Comment: The 3*d* signal shown here is resolved into the $3d_{5/2}$ and $3d_{3/2}$ transitions.



- **Accession #:** 00046-03
- **Host Material:** KBr
- **Technique:** XPS
- **Spectral Region:** K $2p_{3/2}$; K $2p_{1/2}$

Instrument: Surface Science Instruments X-Probe 206
Excitation Source: Al K_α monochromatic
Source Energy: 1486.6 eV
Source Strength: 10 W
Source Size: 150 μm × 150 μm
Incident Angle: 20°
Analyzer Type: spherical sector
Analyzer Pass Energy: 25 eV
Analyzer Resolution: 0.25 eV
Emission Angle: 0°
Data Acquisition Time: 1020 s
Dead Time Correction: not specified
Number of Scans: 20
Comment: The 2*p* signal shown here is resolved into the $2p_{3/2}$ and $2p_{1/2}$ transitions.



- **Accession #:** 00046-04
- **Host Material:** KBr
- **Technique:** XPS
- **Spectral Region:** C 1*s*; K $2p_{3/2}$

Instrument: Surface Science Instruments X-Probe 206
Excitation Source: Al K_α monochromatic
Source Energy: 1486.6 eV
Source Strength: 100 W
Source Size: 600 μm × 600 μm
Incident Angle: 20°
Analyzer Type: spherical sector
Analyzer Pass Energy: 50 eV
Analyzer Resolution: 0.45 eV
Emission Angle: 0°
Data Acquisition Time: 260 s
Dead Time Correction: not specified
Number of Scans: 10
Comment: The C 1*s* signal is shown together with the K $2p_{3/2}$ signal to assist with energy referencing.