1,3-Dimethyl-imidazolium dimethyl phosphate ([MMIM]⁺[DMP]⁻) analyzed by XPS and HAXPES [©]

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1,3-Dimethyl-imidazolium dimethyl phosphate ([MMIM]⁺[DMP]⁻) analyzed by XPS and HAXPES ⁽¹⁾

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Note: This paper is part of the 2023 Special Topic Collection on Higher Energy X-ray Photoelectron Spectroscopy.

ABSTRACT

The ionic liquid 1,3-dimethyl-imidazolium-dimethylphosphate ($[MMIM]^+[DMP]^-$) was analyzed using (hard) x-ray photoelectron spectroscopy. Here, XPS and HAXPES spectra are shown in comparison. For the acquisition of the XPS spectra, monochromatic Al K_{α} radiation at 1486.6 eV was used, while for the acquisition of the HAXPES spectra, monochromatic Cr K_{α} radiation at 5414.8 eV was applied. Here, survey scans and high-resolution spectra of P 2*p*, P 2*s*, C 1*s*, O 1*s*, and N 1*s* for both methods and P 1*s*, P $KL_{2,3}L_{2,3}$, and P $KL_1L_{2,3}$ for HAXPES are shown.

Key words: $C_7H_{15}N_2O_4P$, [MMIM]⁺[DMP]⁻, ionic liquid, hard x-ray photoelectron spectroscopy, HAXPES, Cr K_{α}

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Accession#: 01809, 01810, 01811, and 01812	Major Elements in Spectra: C, O, N, and P
Technique: XPS and XAES	Minor Elements in Spectra: None
Host Material: C ₇ H ₁₅ N ₂ O ₄ P ionic liquid	Published Spectra: 21
Instrument: ULVAC-PHI Quantes	Spectral Category: Comparison

INTRODUCTION

Ionic liquids (ILs) are organic salts that are liquid below temperatures of 100 °C. Due to their unique properties, they attract a lot of attraction in different fields like catalysis (Ref. 1), electrochemistry (Ref. 2), solar cells (Ref. 3), pharmaceutics, and medicine (Ref. 4).

Furthermore, they exhibit a high degree of lateral and depth homogeneity and have a well-defined stoichiometry, an extremely low vapor pressure, and high surface tension, which make them optimally suited as reference materials for the XPS analysis (Ref. 5).

Recently, spectra of the ionic liquid $[PMIM]^+[NTf_2]^-$ were published (Refs. 6 and 7) showing the suitability as reference of this material. In this work, the ionic liquid 1,3-dimethyl-imidazolium dimethyl phosphate ($[MMIM]^+[DMP]^-$) was analyzed, which is a vacuum ionic liquid with high thermal and hydrolytic stabilities (Ref. 8) consisting of C, O, N, and P. A lab-based ULVAC-PHI Quantes system equipped with a monochromatic Al K_{α} (at 1486.6 eV) and a Cr K_{α} x-ray source (at 5414.8 eV) was used for the measurements. This instrument allows a comparison of XPS and HAXPES data. The use of XPS detects photoemission from about 10 nm depths of the sample, while the detection depth of HAXPES is about three times as deep. This leads to a minimization of surface contamination effects. It is also possible to detect peaks at higher binding energy with HAXPES, here, e.g., the P 1*s* and the Auger peaks, due to the higher energy of the Cr K_{α} source compared to Al K_{α} .

Survey and high-resolution spectra are shown in this work. The spectra were obtained using monochromatic Al K_{α} and Cr K_{α} x-ray sources at 1486.6 and 5414.8 eV. High-resolution spectra of C 1s, O 1s, N 1s, P 2p, P 1s, P 2s, P $KL_{2,3}L_{2,3}$, and P $KL_{1}L_{2,3}$ are shown.

XPS and HAXPES results agree broadly. The element composition measured in this study is in close agreement with the expected stoichiometric ratio of [MMIM]⁺[DMP]⁻ with an average



relative deviation of 3.6%, indicating that the sample is homogeneous in the information depth of HAXPES (Refs. 9 and 10).

SPECIMEN DESCRIPTION (ACCESSION # 01809)

Host Material: C7H15N2O4P ionic liquid

CAS Registry #: 654058-04-5

- Host Material Characteristics: Homogeneous; liquid; unknown crystallinity; unknown conductivity; organic compound; other
- **Chemical Name:** 1,3-Dimethyl-imidazolium dimethyl phosphate ([MMIM]⁺[DMP]⁻)

Source: Merck KGaA, Darmstadt, Germany

Host Composition: C₇H₁₅N₂O₄P

Form: Ionic liquid

Structure: Ionic liquid

History and Significance: [MMIM]⁺[DMP]⁻ is commercially available from Merck.

As Received Condition: In a bottle

Analyzed Region: Same as the host material

Ex Situ **Preparation/Mounting:** In a sample holder, which was mounted on a platen using an insulating double-sided tape, a drop of approx. 5μ l of the ionic liquid was filled in. This sample was stored in the intro chamber of the spectrometer at ~ 10^{-5} Pa for 7 days to remove vapors before the measurement started.

In Situ Preparation: None

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:** <10⁻⁶ Pa

Pre-analysis Beam Exposure: 0 s

SPECIMEN DESCRIPTION (ACCESSION # 01810)

Host Material: Au

CAS Registry #: 7440-57-5

Host Material Characteristics: Homogeneous; solid; polycrystalline; conductor; metal; other

Chemical Name: Au

Source: Alfa Aesar

Host Composition: Au

Form: Solid foil

Structure: Face centered cubic

History and Significance: The calibration sample was mounted and provided by the manufacturer of the spectrometer (ULVAC PHI). As Received Condition: Mounted foil

As Received Condition: Mounted Ion

Analyzed Region: Same as the host material

- *Ex Situ* **Preparation/Mounting:** The foil was mounted on a sample holder on a platen by the manufacturer. This platen did not leave the vacuum chamber of the spectrometer for several months.
- *In Situ* **Preparation:** The Au foil was sputtered with 4 kV with an Argon ion gun over a $2 \times 2 \text{ mm}^2$ area for 5 min before the measurement to remove surface contamination.
- 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:** <10⁻⁶ Pa

Pre-analysis Beam Exposure: 0 s

SPECIMEN DESCRIPTION (ACCESSION # 01811)

Host Material: Ag

CAS Registry #: 7440-22-4

Host Material Characteristics: Homogeneous; solid; polycrystalline; conductor; metal; other

Chemical Name: Ag

Source: Alfa Aesar

Host Composition: Ag

Form: Solid foil

Structure: Face centered cubic

History and Significance: The calibration sample was mounted and provided by the manufacturer of the spectrometer, ULVAC PHI.

As Received Condition: Mounted foil

Analyzed Region: Same as the host material

- *Ex Situ* **Preparation/Mounting:** The foil was mounted on a sample holder on a platen by the manufacturer. This platen did not leave the vacuum chamber of the spectrometer for several months.
- *In Situ* **Preparation:** The Ag foil was sputtered with 4 kV with an Argon ion gun over a $2 \times 2 \text{ mm}^2$ area for 5 min before the measurement to remove surface contamination.

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: <10⁻⁶ Pa

Pre-analysis Beam Exposure: 0 s

SPECIMEN DESCRIPTION (ACCESSION # 01812)

Host Material: Cu0

- **CAS Registry #:** 7440-50-8
- Host Material Characteristics: Homogeneous; solid; polycrystalline; conductor; metal; other

Chemical Name: Cu

Source: Alfa Aesar

Host Composition: Cu

- Form: Solid foil
- **Structure:** Face centered cubic
- **History and Significance:** The calibration sample was mounted and provided by the manufacturer of the spectrometer, ULVAC PHI.

As Received Condition: Mounted foil

Analyzed Region: Same as the host material

- *Ex Situ* **Preparation/Mounting:** The foil was mounted on a sample holder on a platen by the manufacturer. This platen did not leave the vacuum chamber of the spectrometer for several months.
- *In Situ* **Preparation:** The Cu foil was sputtered with 4 kV with an Argon ion gun over a $2 \times 2 \text{ mm}^2$ area for 5 min before the measurement to remove surface contamination.
- **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: $<10^{-6}$ 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 ($\mathbf{T} = \mathbf{E}^{\mathbf{N}}$): The energy dependence can be modeled using the following equation: $\frac{A}{E_p} = \left(\frac{a^2}{a^2 + R^2}\right)^b$, 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 are recorded on a sputter cleaned sample at different pass energies. The values of *a* and *b* are then determined by a linear least square fit of the data applying the equation described above.

Excitation Source Window: Al

Excitation Source: Al K_{α} and Cr K_{α} monochromatic **Source Energy:** 1486.6 and 5414.8 eV **Source Strength:** 24.0 and 51.4 W **Source Beam Size:** $100 \times 100 \,\mu\text{m}^2$ **Signal Mode:** Multichannel direct

Geometry

Incident Angle: Al: 0°, Cr: 22° Source-to-Analyzer Angle: Al: 45°, Cr: 49° Emission Angle: 45° Specimen Azimuthal Angle: 0° Acceptance Angle from Analyzer Axis: 0° Analyzer Angular Acceptance Width: 20° × 20°

lon Gun

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

DATA ANALYSIS METHOD

Energy Scale Correction: The energy scale shift was calculated by setting the C 1s binding energy to 284.80 eV. The XPS and

HAXPES high-resolution spectra of the C 1s peak give a position of 283.97 and 284.22 eV; therefore, the correction shift is 0.83 eV for XPS and 0.58 eV for HAXPES.

- **Recommended Energy Scale Shift:** XPS: 0.83 eV; HAXPES: 0.58 eV for binding energy (photoelectron spectra) and -0.58 eV for kinetic energy (Auger electron spectra)
- **Peak Shape and Background Method:** The inbuilt Gaussian– Lorentzian peak shapes routine of MultiPak and a Shirley background method were used.
- **Quantitation Method:** For Quantification, the PHI MultiPak software version 9.9.2 was used applying a nonlinear least square routine (Ref. 11) with relative empirical sensitivity factors that were provided by the manufacturer.

ACKNOWLEDGMENTS

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

X. Knigge: Formal analysis (equal); Writing – original draft (equal). **J. Radnik:** Formal analysis (equal); Writing – original draft (equal).

DATA AVAILABILITY

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

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SPECTRAL FEATURES TABLE							
Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01809-02	C 1s	284.80	1.47	7073	0.314	49.7	C ₇ H ₁₅ N ₂ O ₄ P
01809-03	O 1s	528.79	1.29	4381	0.733	29.3	C ₇ H ₁₅ N ₂ O ₄ P
01809-03	O 1s	531.03	1.29	4711	0.733		C ₇ H ₁₅ N ₂ O ₄ P
01809-04	N 1s	400.17	1.15	3144	0.499	14.0	C ₇ H ₁₅ N ₂ O ₄ P
01809-05	P 2p	131.76	1.41	1231	0.525	7.0	C ₇ H ₁₅ N ₂ O ₄ P
01809-06	P 2s	188.82	2.19	1299	0.380		C ₇ H ₁₅ N ₂ O ₄ P
01809-08	C 1s	284.80	1.47	148	0.199	48.6	C ₇ H ₁₅ N ₂ O ₄ P
01809-09	O 1s	528.75	1.45	139	0.589	28.8	C ₇ H ₁₅ N ₂ O ₄ P
01809-09	O 1s	530.98	1.45	148	0.589		C ₇ H ₁₅ N ₂ O ₄ P
01809-10	N 1s	400.15	1.36	85	0.331	15.4	C ₇ H ₁₅ N ₂ O ₄ P
01809-11	P 2p	131.15	1.34	20	0.156		C ₇ H ₁₅ N ₂ O ₄ P
01809-12	P 2s	188.67	2.15	55	0.450		C ₇ H ₁₅ N ₂ O ₄ P
01809-13	P 1s	2145.66	1.56	874	5.136	7.1	C ₇ H ₁₅ N ₂ O ₄ P
01809-14 ^a	P <i>KL</i> _{2.3} <i>L</i> _{2.3}	1852.05	1.41	345			C ₇ H ₁₅ N ₂ O ₄ P
01809-15 ^a	P KL ₁ L _{2,3}	1787.35	3.02	82			C ₇ H ₁₅ N ₂ O ₄ P

 ^{a}The P $\textit{KL}_{2,3}\textit{L}_{2,3}$ and the P $\textit{KL}_{1}\textit{L}_{2,3}$ peak energies are listed as kinetic energy.

ANALYZER CALIBRATION TABLE							
Spectrum ID #	Element/ Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × cts/s)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01810-01	Au 4f _{7/2}	84.1	2.17	392 520	3.849		XPS Au
01811-01	Ag 3d5/2	368.4	1.70	470 847	3.742		XPS Aq
01812-01	Cu 2p _{3/2}	932.7	2.00	396 795	2.626		XPS Cu
01810-02	Au 4f7/2	83.9	2.17	7 293	1.103		HAXPES Au
01811-02	Ag 3d5/2	368.0	1.97	10 340	1.996		HAXPES Ag
01812-02	Cu 2p _{3/2}	932.5	2.15	24 495	3.829		HAXPES Cu



GUIDE TO FIGURES					
Spectrum (Accession) #	Spectral Region	Voltage Shift ^a	Multiplier	Baseline	Comment #
01809-01	Survey	-0.83	1	0	1
01809-02	C 1s	-0.83	1	0	1
01809-03	O 1s	-0.83	1	0	1
01809-04	N 1 <i>s</i>	-0.83	1	0	1
01809-05	P 2p	-0.83	1	0	1
01809-06	P 2s	-0.83	1	0	1
01809-07	Survey	-0.58	1	0	2
01809-08	C 1s	-0.58	1	0	2
01809-09	O 1 <i>s</i>	-0.58	1	0	2
01809-10	N 1 <i>s</i>	-0.58	1	0	2
01809-11	P 2p	-0.58	1	0	2
01809-12	P 2s	-0.58	1	0	2
01809-13	P 1s	-0.58	1	0	2
01809-14	P KL _{2.3} L _{2.3}	+0.58	1	0	2
01809-15	P KL1L2.3	+0.58	1	0	2
01810-01	Survey (Au)		1	0	3
01811-01	Survey (Ag)		1	0	3
01812-01	Survey (Cu)		1	0	3
01810-02	Survey (Au)		1	0	4
01811-02	Survey (Ag)		1	0	4
01812-02	Survey (Cu)		1	0	4

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

1. C₇H₁₅N₂O₄C ionic liquid XPS

2. $C_7H_{15}N_2O_4C$ ionic liquid HAXPES

3. Calibration XPS

4. Calibration HAXPES





Accession #:	01809-01
Host Material:	C ₇ H ₁₅ N ₂ O ₄ P ionic liquid
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Al K_{α} monochromatic
Source Energy:	1486.6 eV
Source Strength:	24.0 W
Source Size:	0.1 × 0.1 mm ²
Analyzer Type:	Spherical sector analyzer
Incident Angle:	0°
Emission Angle:	45°
Analyzer Pass Energy:	280 eV
Analyzer Resolution:	1.9 eV
Total Signal Accumulation Time:	1434 s
Total Elapsed Time:	1572 s
Number of Scans:	3
Effective Detector Width:	31 eV



















Accession #:	01809-07
Host Material:	C ₇ H ₁₅ N ₂ O ₄ P ionic liquid
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Cr K_{α} monochromatic
Source Energy:	5414.8 eV
Source Strength:	51.4 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:	25 158 s
Total Elapsed Time:	27 672 s
Number of Scans:	5
Effective Detector Width:	31 eV













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Accession #:	01810-01
Host Material:	Au
Technique:	XPS
Spectral Region:	Survey
Instrument:	ULVAC-PHI Quantes
Excitation Source:	Al K_{α} monochromatic
Source Energy:	1486.6 eV
Source Strength:	24.0 W
Source Size:	$0.1 \times 0.1 \text{ mm}^2$
Analyzer Type:	Spherical sector analyzer
Incident Angle:	0°
Emission Angle:	45°
Analyzer Pass Energy:	280 eV
Analyzer Resolution:	1.9 eV
Total Signal Accumulation Time:	1434 s
Total Elapsed Time:	1572 s
Number of Scans:	1
Effective Detector Width:	31 eV



Accession #:	01811-01	
Host Material:	Ag	
Technique:	XPS	
Spectral Region:	Survey	
Instrument:	ULVAC-PHI Quantes	
Excitation Source:	Al K_{α} monochromatic	
Source Energy:	1486.6 eV	
Source Strength:	24.0 W	
Source Size:	0.1 × 0.1 mm ²	
Analyzer Type:	Spherical sector analyzer	
Incident Angle:	0°	
Emission Angle:	45°	
Analyzer Pass Energy:	280 eV	
Analyzer Resolution:	1.9 eV	
Total Signal Accumulation Time:	1434 s	
Total Elapsed Time:	1572 s	
Number of Scans:	1	
Effective Detector Width:	31 eV	





01812-01 Accession #: Cu Host Material: XPS Technique: Spectral Region: Survey Instrument: ULVAC-PHI Quantes **Excitation Source:** Al K_{α} monochromatic Source Energy: 1486.6 eV Source Strength: 24.0 W $0.1 \times 0.1 \text{ mm}^2$ Source Size: Analyzer Type: Spherical sector analyzer Incident Angle: 0° 45° Emission Angle: 280 eV Analyzer Pass Energy: Analyzer Resolution: 1.9 eV Total Signal Accumulation Time: 1434 s Total Elapsed Time: 1572 s Number of Scans: 1 31 eV **Effective Detector Width:**







Accession #:	01810-02	
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:	51.4 W	
Source Size:	$0.1 \times 0.1 \text{ mm}^2$	
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:	19 980 s	
Total Elapsed Time:	22 140 s	
Number of Scans:	4	
Effective Detector Width:	31 eV	

30, 014006-19

35	Ag 2p3	01811-02
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10 -	WW BY	g 3 <i>p1</i> 6 3 <i>p1</i> 15
5 -	- Mr Dille	
0 500	0 4000 3000 2000	1000 0
	Binding energy (eV)	

Accession #:	01811-02	
Host Material:	Ag	
Technique:	XPS	
Spectral Region:	Survey	
Instrument:	ULVAC-PHI Quantes	
Excitation Source:	Cr K_{α} monochromatic	
Source Energy:	5414.8 eV	
Source Strength:	51.4 W	
Source Size:	0.1 × 0.1 mm	
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:	19 980 s	
Total Elapsed Time:	22 140 s	
Number of Scans:	4	
Effective Detector Width:	31 eV	



x 10³

ARTICLE







Accession #:	01812-02	
Host Material:	Cu	
Technique:	XPS	
Spectral Region:	Survey	
Instrument:	ULVAC-PHI Quantes	
Excitation Source:	Cr K_{α} monochromatic	
Source Energy:	5414.8 eV	
Source Strength:	51.4 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:	19 980 s	
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Number of Scans:	4	
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