



STRUCTURAL SCIENCE
CRYSTAL ENGINEERING
MATERIALS

Volume 76 (2020)

Supporting information for article:

**CRYSTAL STRUCTURES AND PHASE TRANSITIONS OF
IMIDAZOLIUM HYPODIPHOSPHATES**

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Ślepokura**

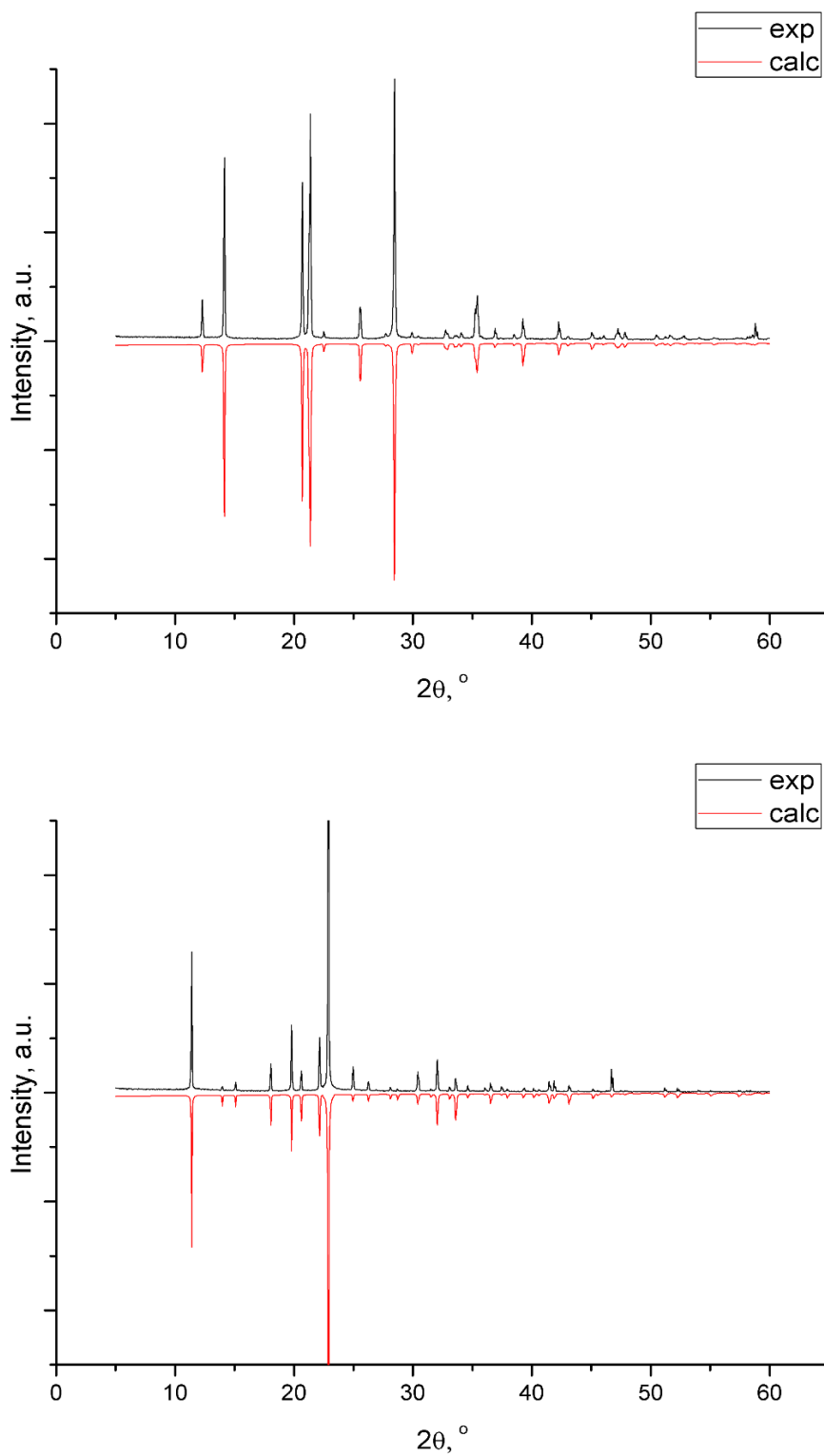


Figure S1 Experimental (black line) X-ray powder diffraction patterns for **(I)** (top) and **(II)** (bottom) compared with their theoretical diffraction patterns (red line; shown upside down).

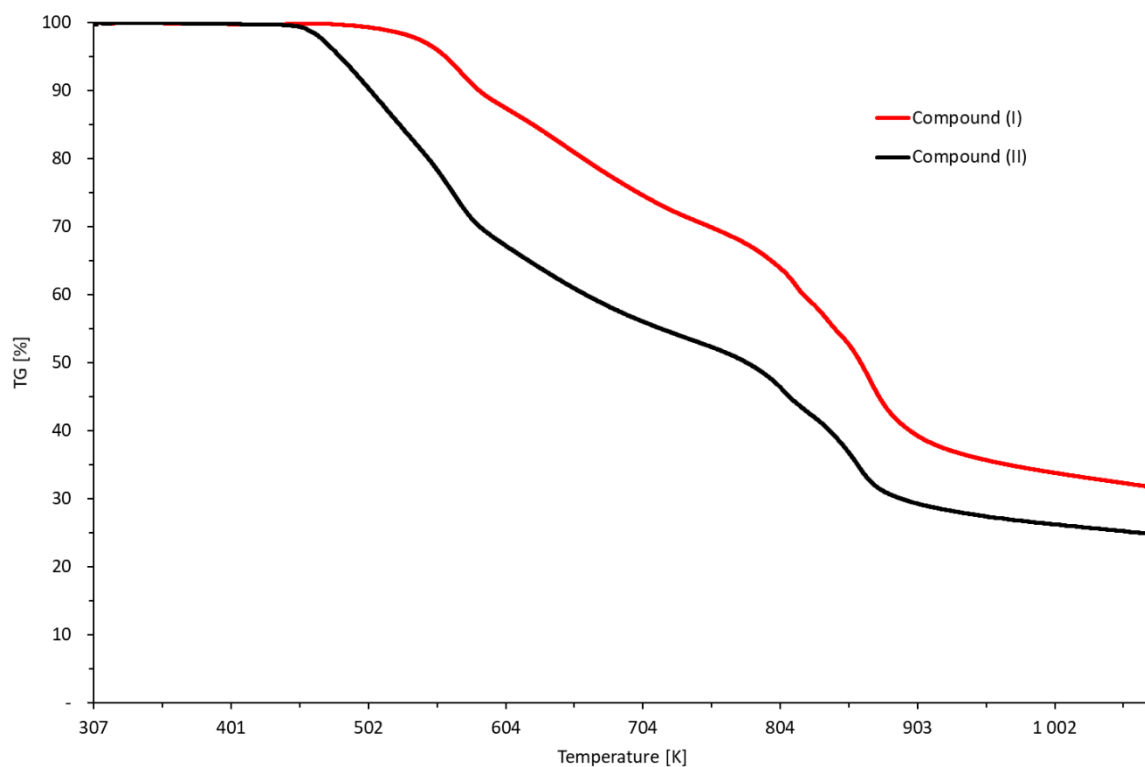


Figure S2 TG curves for **(I)** (red, $m = 4.44$ mg) and **(II)** (black, $m = 3.12$ mg).

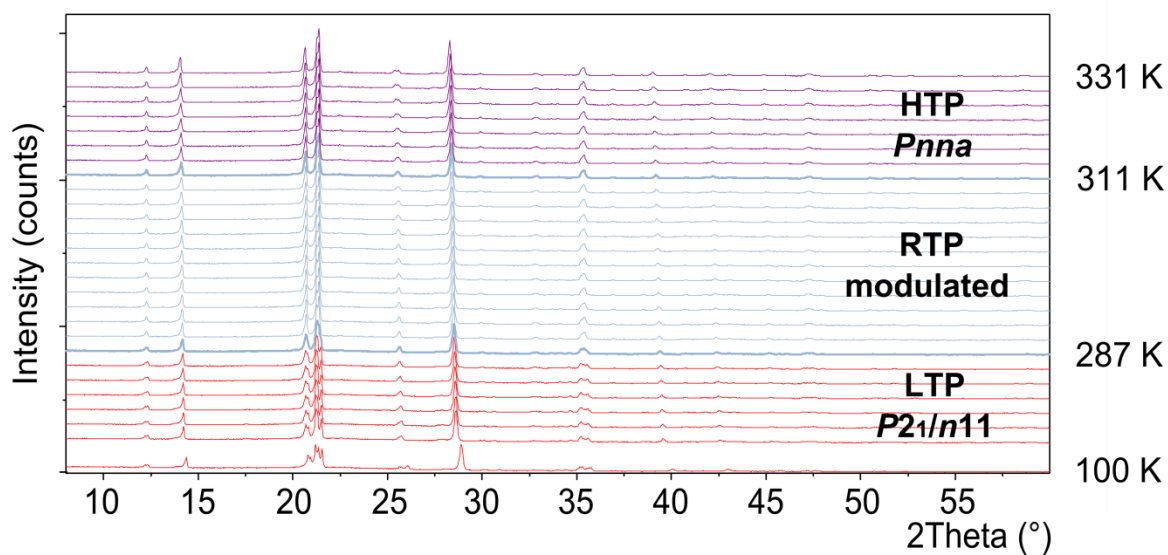


Figure S3 Experimental variable temperature X-ray powder diffraction patterns for **(I)** carried out at 100, 260-285 K (every 5 K), 287-321 K (every 2 K), 326 and 331. The different phases are shown in different colours.

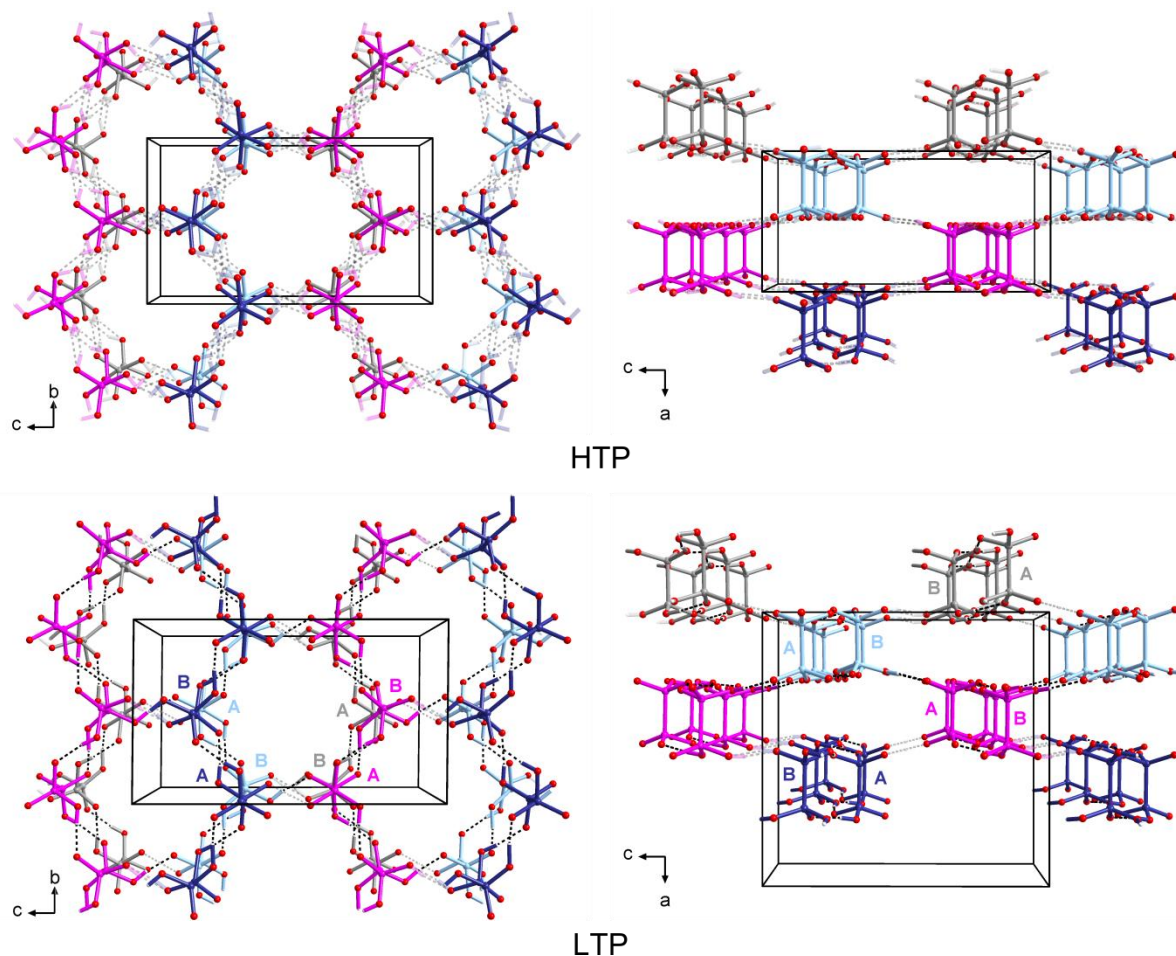


Figure S4 Perspective views of hydrogen-bonded anionic sublattice in HTP and LTP of (**I**) viewed down the **a**-axis (left) and **b**-axis (right). Disordered or partially occupied H atoms are represented by transparent spheres. Hydrogen bonds are shown with dashed lines.

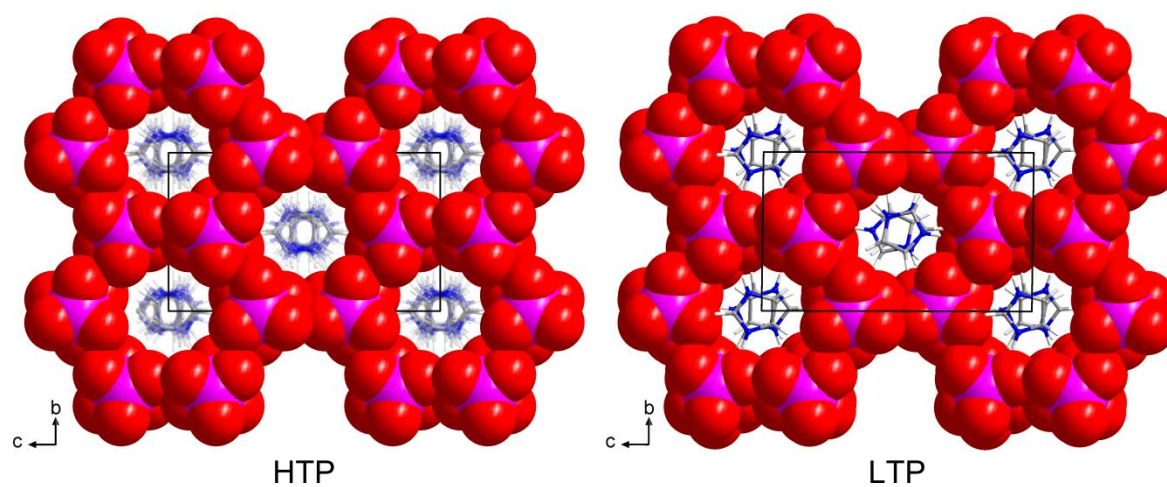


Figure S5 Space-filling views of hydrogen-bonded anionic sublattice in HTP and LTP of (**I**) viewed down the **a**-axis. The channels are occupied by the π - π stacks of imidazolium cations (shown as transparent in HTP).

Table S1 Selected geometric parameters (Å, °) for **(I)** and **(II)**

| | HTP of (I) | LTP of (I) | | (II) |
|---|-------------------|-------------------|-------------|--------------|
| | | Anion A | Anion B | |
| P1—P1 ⁱ /P2 | 2.1805 (14) | 2.1863 (7) | 2.1902 (7) | 2.1741 (11) |
| P1—O1 | 1.5198 (13) | 1.5083 (12) | 1.4972 (12) | 1.4986 (17) |
| P1—O2 | 1.5252 (14) | 1.5299 (13) | 1.5299 (13) | 1.5157 (16) |
| P1—O3 | 1.5219 (13) | 1.5583 (12) | 1.5551 (12) | 1.5768 (17) |
| P2—O4 | | 1.5077 (12) | 1.4987 (12) | |
| P2—O5 | | 1.5222 (13) | 1.5331 (13) | |
| P2—O6 | | 1.5551 (12) | 1.5614 (13) | |
| O1—P1—O2 | 111.91 (7) | 114.69 (7) | 113.34 (7) | 117.29 (9) |
| O1—P1—O3 | 111.45 (9) | 109.61 (7) | 113.91 (7) | 111.45 (10) |
| O2—P1—O3 | 111.50 (8) | 109.86 (7) | 108.24 (7) | 106.66 (9) |
| O1—P1—P1 ⁱ /P2 | 107.67 (7) | 108.19 (5) | 108.56 (5) | 108.62 (7) |
| O2—P1—P1 ⁱ /P2 | 107.11 (7) | 106.20 (5) | 106.38 (5) | 107.62 (6) |
| O3—P1—P1 ⁱ /P2 | 106.89 (6) | 108.03 (5) | 105.89 (5) | 104.37 (7) |
| O4—P2—O5 | | 115.74 (7) | 113.44 (7) | |
| O4—P2—O6 | | 109.96 (7) | 113.32 (7) | |
| O5—P2—O6 | | 109.81 (7) | 107.01 (7) | |
| O4—P2—P1 | | 106.09 (5) | 108.13 (5) | |
| O5—P2—P1 | | 108.55 (6) | 107.79 (5) | |
| O6—P2—P1 | | 106.19 (5) | 106.82 (5) | |
| O1—P1—P1 ⁱ /P2—O1 ⁱ /O4 | 50.17 (11) | -68.32 (7) | 43.44 (8) | 67.61 (15) |
| O2—P1—P1 ⁱ /P2—O1 ⁱ /O4 | -70.35 (7) | 55.28 (7) | -78.85 (8) | -164.46 (9) |
| O3—P1—P1 ⁱ /P2—O1 ⁱ /O4 | 170.03 (7) | 173.10 (7) | 166.13 (7) | -51.39 (9) |
| O1—P1—P1 ⁱ /P2—O2 ⁱ /O5 | -70.35 (7) | 56.68 (7) | -79.56 (7) | -164.46 (9) |
| O2—P1—P1 ⁱ /P2—O2 ⁱ /O5 | 169.13 (10) | -179.72 (7) | 158.14 (7) | -36.54 (13) |
| O3—P1—P1 ⁱ /P2—O2 ⁱ /O5 | 49.51 (7) | -61.90 (7) | 43.13 (7) | 76.54 (9) |
| O1—P1—P1 ⁱ /P2—O3 ⁱ /O6 | 170.03 (7) | 174.70 (7) | 165.73 (7) | -51.39 (10) |
| O2—P1—P1 ⁱ /P2—O3 ⁱ /O6 | 49.51 (7) | -61.70 (7) | 43.43 (7) | 76.54 (9) |
| O3—P1—P1 ⁱ /P2—O3 ⁱ /O6 | -70.11 (11) | 56.11 (7) | -71.58 (7) | -170.38 (13) |

Symmetry code for HTP of **(I)**: (i) $-x+3/2, -y, z$. Symmetry code for **(II)**: (i) $y, x, -z+1$.