Support information

In Situ template Generation via N-alkylation in the Syntheses of Open-framework Zinc Phosphites and Phosphate

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Figure S1. Simulated and experimental X-ray powder diffraction patterns of compounds I (a), II (b) and III (c).
Figure S2. IR spectra of compounds I-III.

Figure S3. View of the structure of I along the [001] direction, showing the inorganic layers intercalated with organic pmpip$^{2+}$ cations. Color code: ZnO$_4$ tetrahedra, green; HPO$_3$ pseudopyramids, purple.

Figure S4. View of the structure of compound II along the [100] direction, showing the 6- and 8-ring channels. Color code: ZnO$_4$ tetrahedra, green; HPO$_3$ pseudopyramids, purple.
**Figure S5.** View of the structure of II along the [011] direction, showing the puckered 12-ring channels. Color code: ZnO$_4$ tetrahedra, green; HPO$_3$ pseudopyramids, purple.

**Figure S6.** View of the structure of II along the [001] direction with dmdabco$_{2}^{2+}$ cations residing in the 10-ring channels. Color code: ZnO$_4$ tetrahedra, green; HPO$_3$ pseudopyramids, purple.

**Figure S7.** View of the structure of III along the [100] direction with mampy$_{2}^{2+}$ cations residing in the 8-ring channels. Color code: ZnO$_4$ tetrahedra, green; HPO$_3$ pseudopyramids, purple.