Low Temperature Synthesis Study on Metal-Organic Framework CPO-27: Investigating Metal, Solvent and Base Effects down to -78 °C

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1. PXRD Patterns

1.1 *CPO-27-Mg1* in MeOH/NaOH

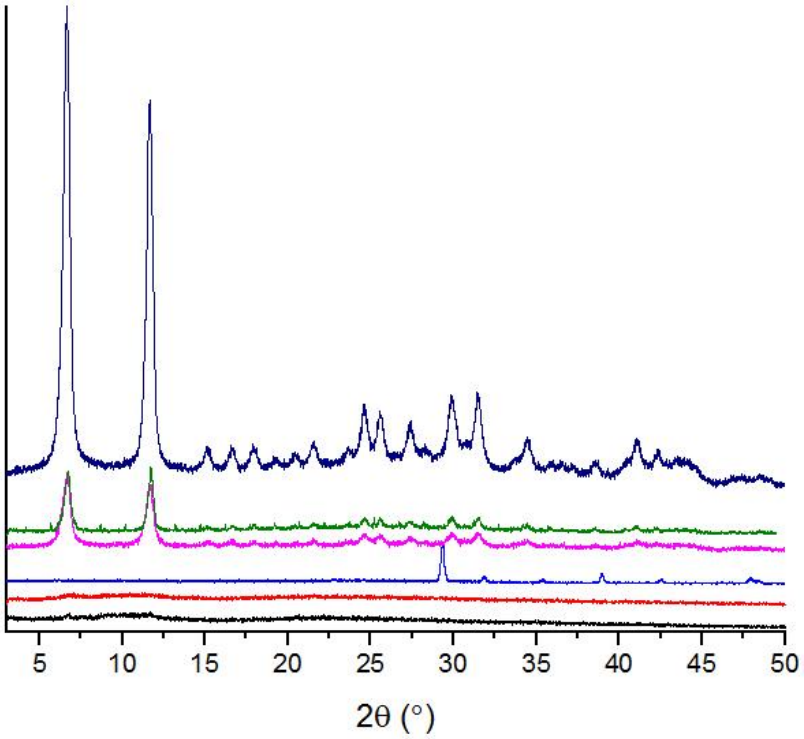

Fig. S1. Powder X-ray diffraction patterns for CPO-27-Mg**1** taken half-way through the synthesis (3h). -78 °C – black; -20 °C – red; -40 °C – blue; ambient – pink; 78 °C – green; 0 °C – navy. All diffraction run for 3 hours with the exception of 0 °C which was run for 12 hours.

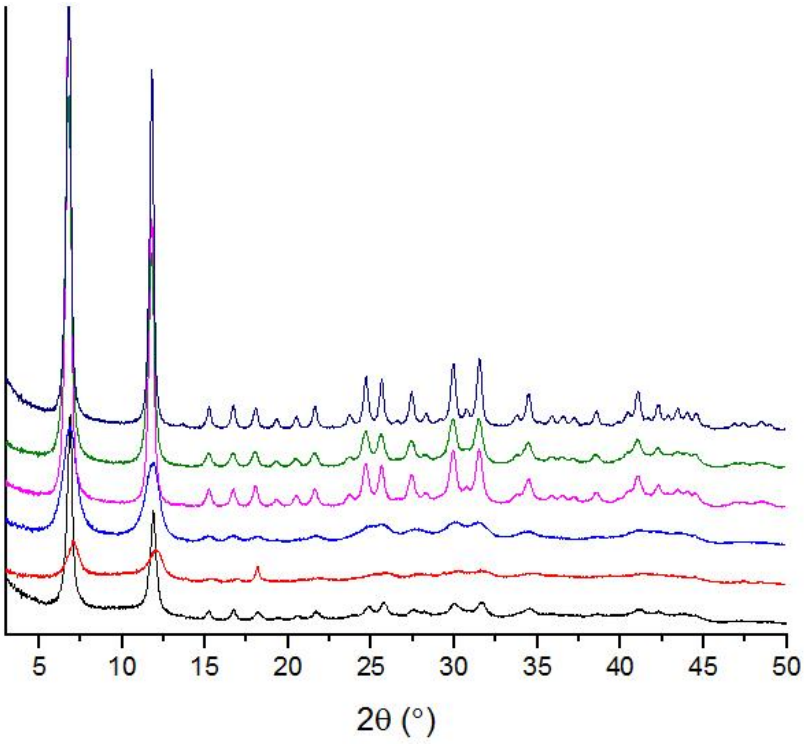

Fig. S2. Powder X-ray diffraction patterns for fully formed CPO-27-Mg**1**. -78 °C – black; -40 °C – red; -20 °C – blue; 0 °C – pink; ambient – green; 78 °C – navy.

1.2. *CPO-27-Ni1* in MeOH/NaOH

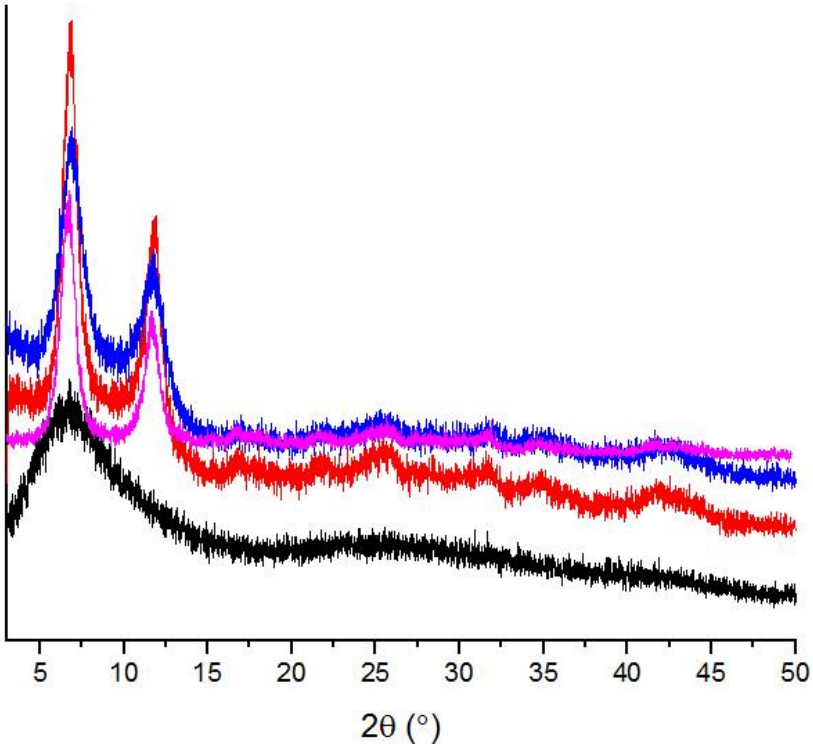

Fig. S3. Powder X-ray diffraction patterns for CPO-27-Ni**1** taken half-way through the synthesis (3h). -20 °C – black; 0 °C – red; ambient – blue; 78 °C – pink.

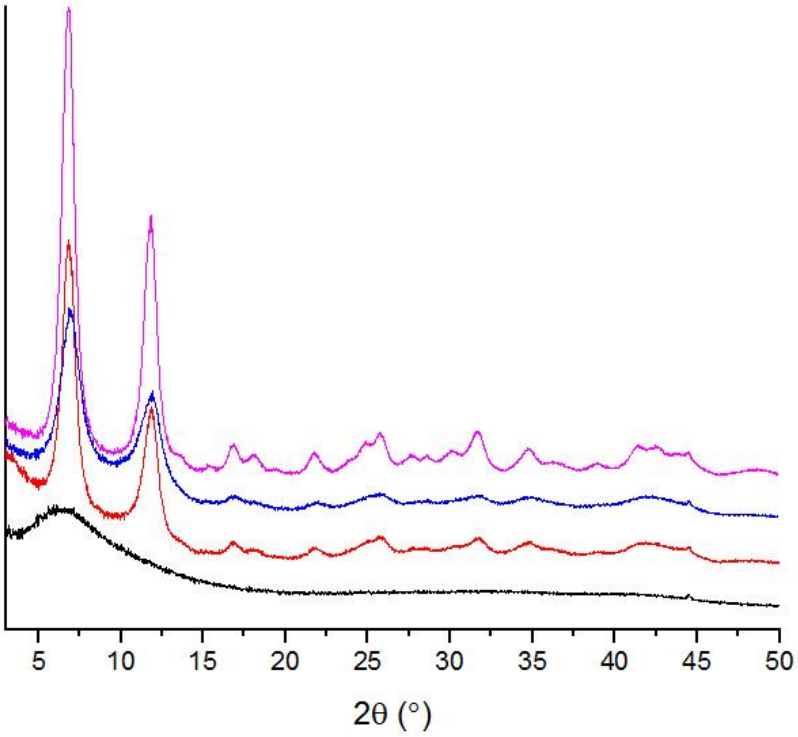

Fig. S4. Powder X-ray diffraction patterns for fully formed CPO-27-Ni**1**. -20 °C – black; 0 °C – red; ambient – blue; 78 °C – pink.

1.3. *CPO-27-Zn1* in MeOH/NaOH

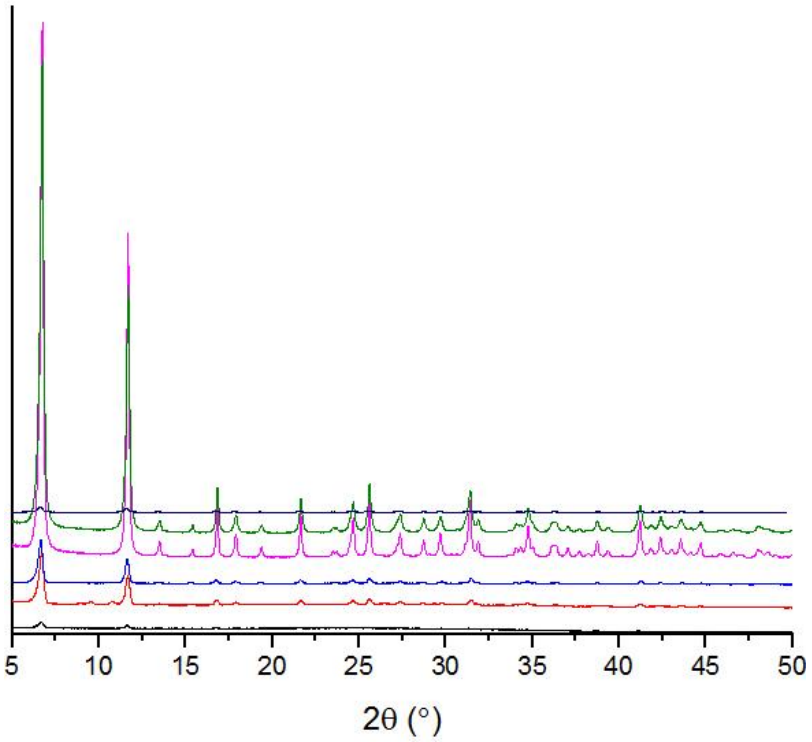

Fig. S5. Powder X-ray diffraction patterns for CPO-27-Zn**1** in MeOH/NaOH taken half-way through the synthesis (3h). -78 °C – black; -40 °C – red; -20 °C – blue; 0 °C – pink; ambient – green; 78 °C – navy.

1.4. *CPO-27-Zn2* in MeOH/TEA

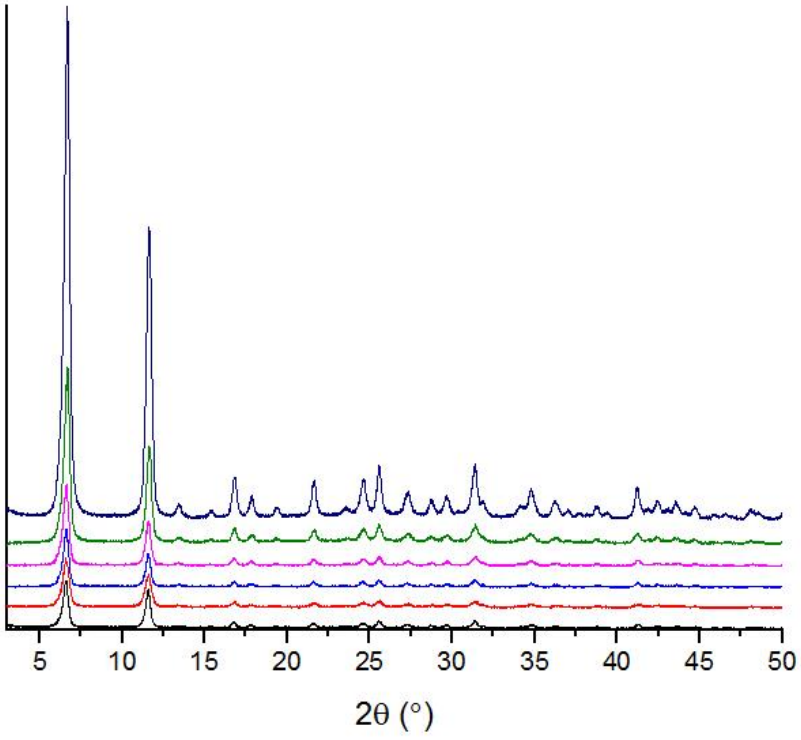

Fig. S6. Powder X-ray diffraction patterns for CPO-27-Zn**2** in MeOH/TEA taken half-way through the synthesis (3h). -78 °C – navy; -40 °C – green; -20 °C – pink; 0 °C – blue; ambient – red; 78 °C – black

1.5. *CPO-27-Zn3* in MeOH/no base

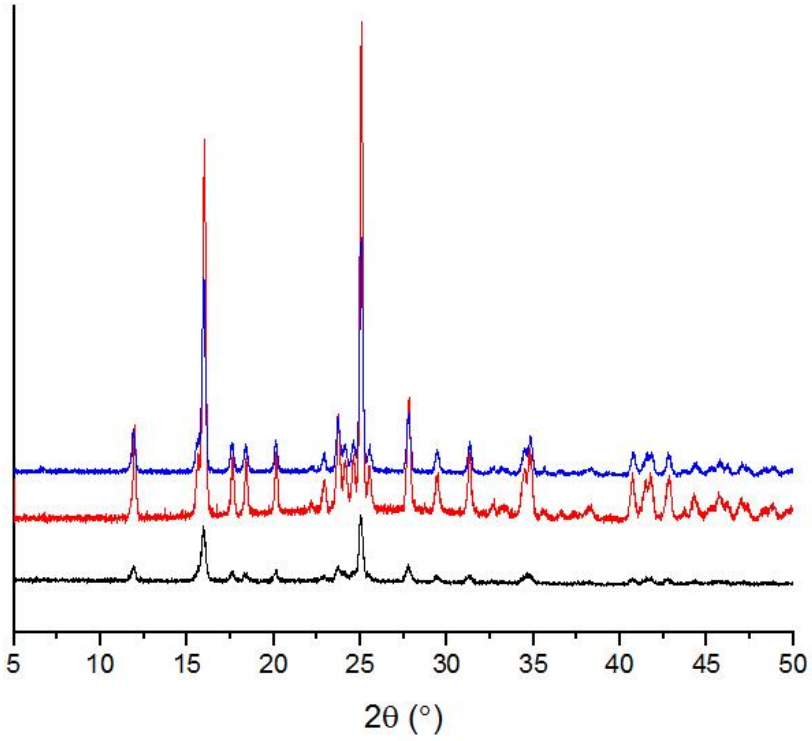

Fig. S7. Powder X-ray diffraction patterns for CPO-27-Zn**3** in MeOH without base taken half-way through the synthesis (3h). 0 °C – black; ambient – red; 78 °C – blue.

1.6. *CPO-27-Zn5* in THF/NaOH

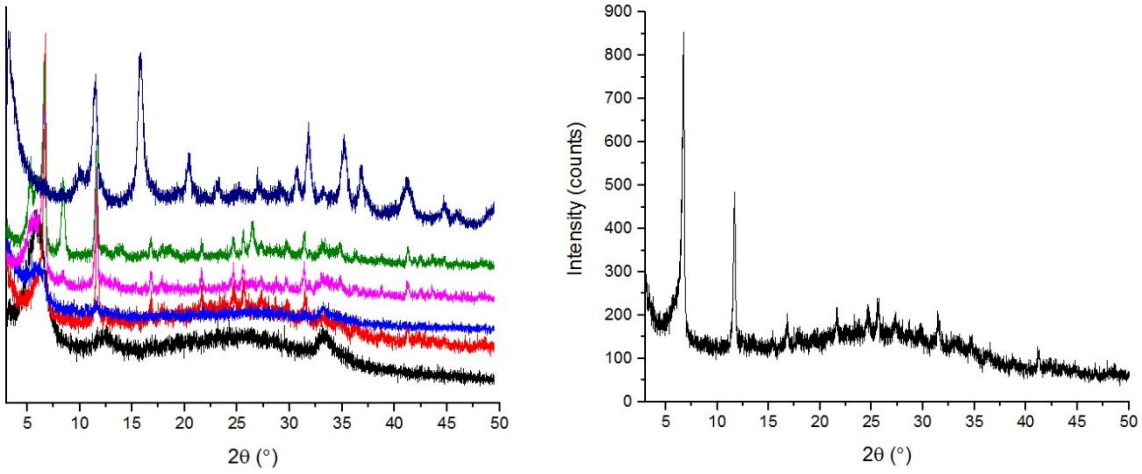

Fig. S8. Left - Powder X-ray diffraction patterns for CPO-27-Zn**5** in THF/NaOH taken half-way through the synthesis (3h). -78 °C – navy; -40 °C – green; -20 °C – pink; 0 °C – blue; ambient – red; 78 °C – black. Right – Powder X-ray diffraction pattern for CPO-27-Zn**5** in THF/NaOH at -40 °C.

1.7. *CPO-27-Zn6* in THF/TEA

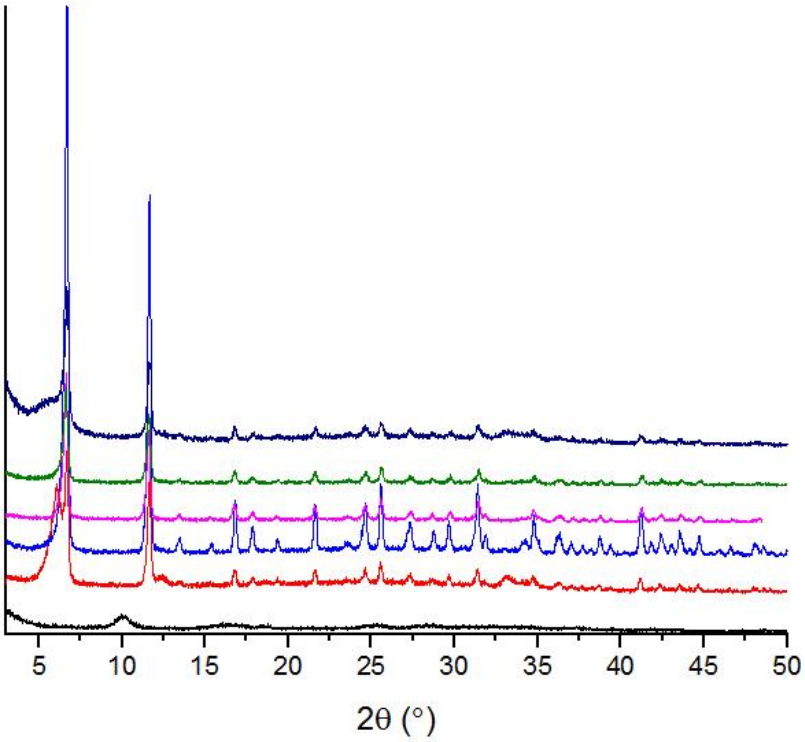

Fig. S9. Powder X-ray diffraction patterns for CPO-27-Zn**6** in THF/TEA taken half-way through the synthesis (3h). -78 °C – navy; -40 °C – green; -20 °C – pink; -0 °C – blue; ambient – red; 78 °C – black.

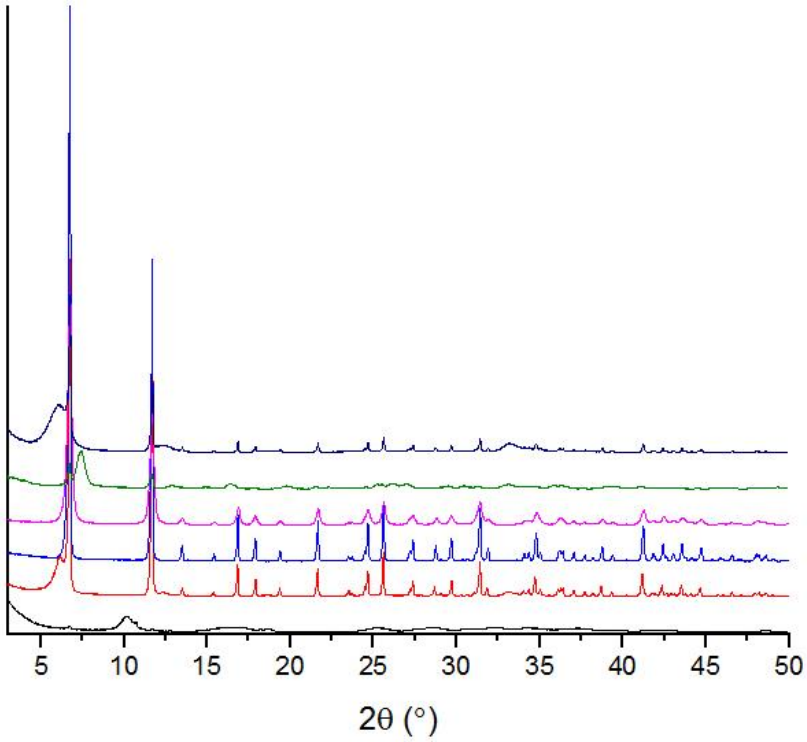

Fig. S10. Powder X-ray diffraction patterns for fully formed CPO-27-Zn**6** in THF/TEA. -78 °C – navy; -40 °C – green; -20 °C – pink; 0 °C – blue; ambient – red; 78 °C – black.

2. Pawley Refinement of [Zn(H_2dhtp)(H_2O)$_2$]

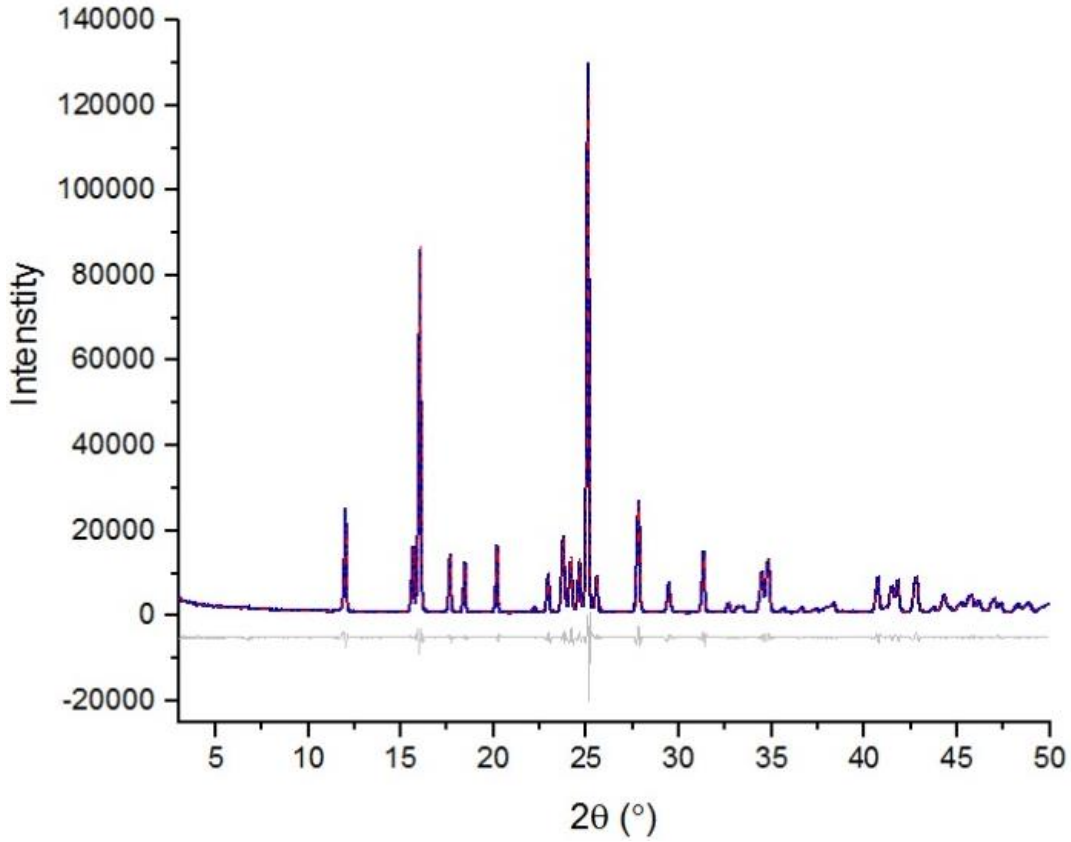

Fig. S11. Topas Pawley refinement for [Zn(H_2dhtp)(H_2O)$_2$] afforded from a CPO-27–Zn**3** synthesis attempt in MeOH. Experimental in blue, calculated model in red and the calculated difference plot in grey.[22]

3. SEM images

3.1. *CPO-27-Mg1* in MeOH/NaOH

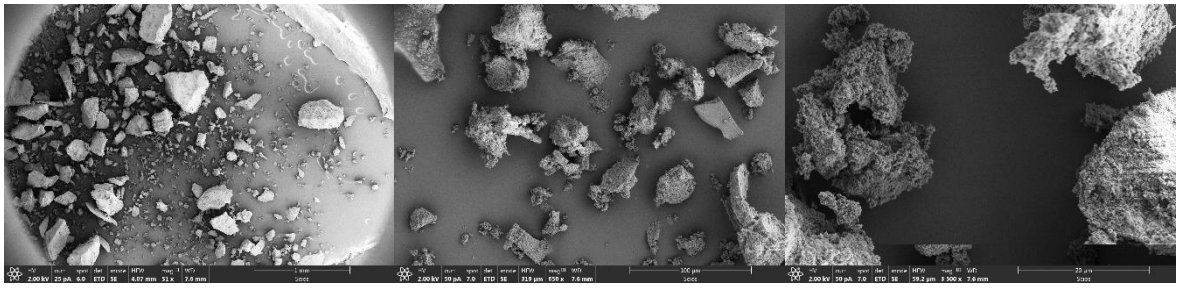

Fig. S12. SEM images for CPO-27-Mg**1** afforded from MeOH/NaOH. From left to right, 1 mm; 100 µm; 10 µm

3.2. *CPO-27-Ni1* in MeOH/NaOH

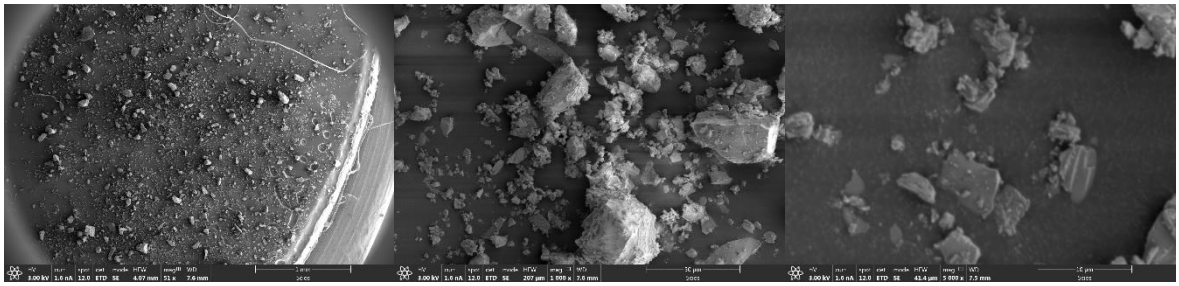

Fig. S13. SEM images for CPO-27-Ni**1** afforded from MeOH/NaOH. From left to right, 1 mm; 50 µm; 10 µm

3.3. *CPO-27-Zn2* in MeOH/TEA

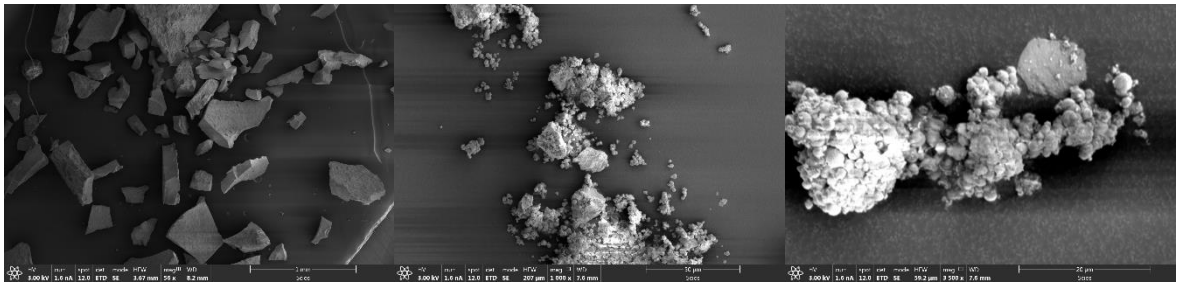

Fig. S14. SEM images for CPO-27-Zn**2** afforded from MeOH/TEA. From left to right, 1 mm; 50 µm; 20 µm

3.4. *CPO-27-Zn3* in MeOH/no base

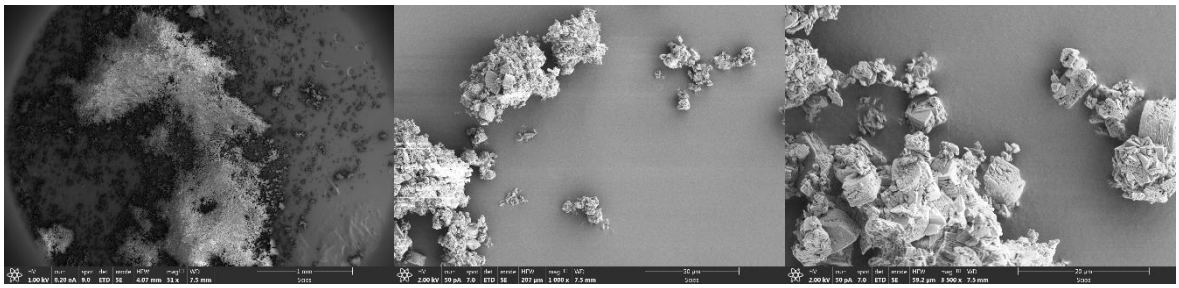

Fig. S15. SEM images for CPO-27-Zn**3** afforded from MeOH without base. From left to right, 1 mm; 50 µm; 20 µm

3.5. *CPO-27-Zn4* in THF/no base

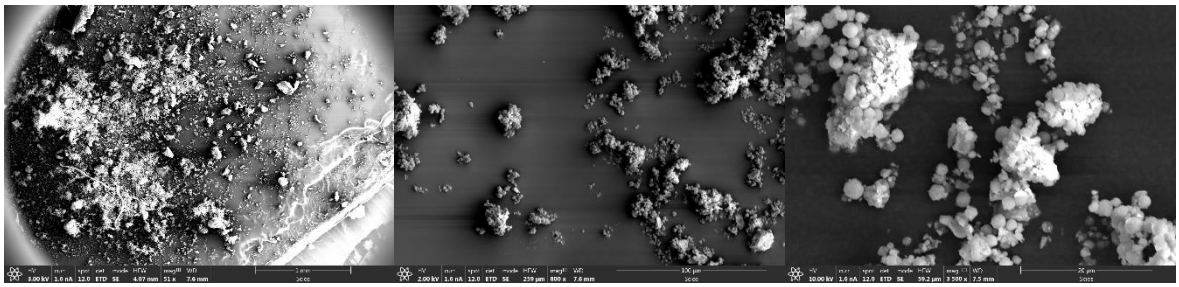

Fig. S16. SEM images for CPO-27-Zn**4** afforded from THF without base. From left to right, 1 mm; 100 µm; 20 µm

3.6. *CPO-27-Zn5* in THF/NaOH

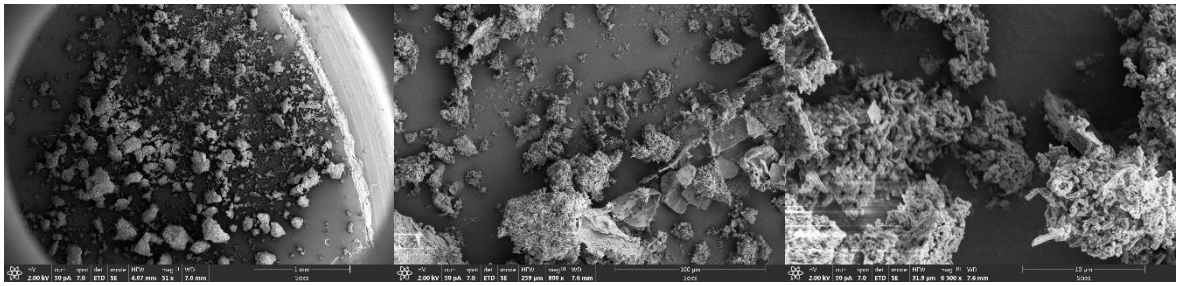

Fig. S17. SEM images for CPO-27-Zn**5** afforded from THF/NaOH. From left to right, 1 mm; 100 µm; 10 µm

3.7. *CPO-27-Zn6* in THF/TEA

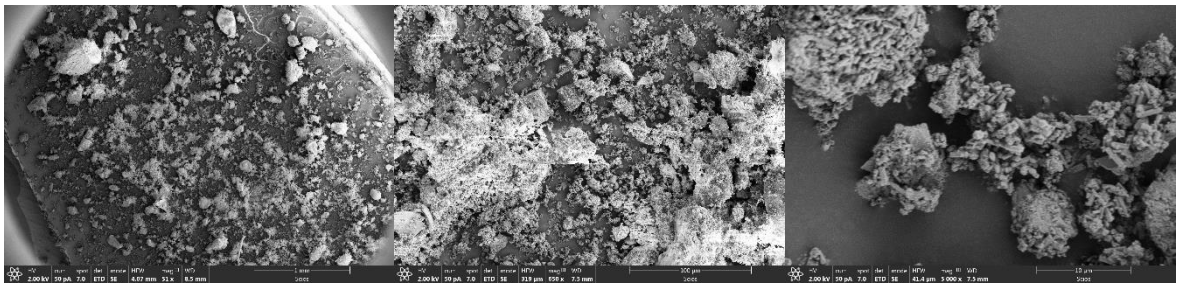

Fig. S18. SEM images for CPO-27-Zn**6** afforded from THF/TEA. From left to right, 1 mm; 100 µm; 10 µm