

Supplementary Information

Synthesis, crystal structure and gas sorption studies of two neutral octahedral $\{M^{II}_{24}\}$ -cages built from sulfonylthiacalix[4]arene tetranuclear clusters and triazine linkers

Ivan V. Khariushin, Véronique Bulach, Jas S. Ward, Kari Rissanen, Svetlana E. Solovieva, Igor S. Antipin, Alexander S. Ovsyannikov, and Sylvie Ferlay

Complex	1 ^a	2
Formula	C ₄₃₂ H ₃₆₀ Co ₂₄ N ₂₄ O ₁₂₆ S ₂₄	C ₄₃₂ H ₃₆₀ Ni ₂₄ O ₁₂₆ S ₂₄
Formula Weight (g mol ⁻¹)	10087.18	10081.90
Colour & Habit	Pink block	Colourless block
Crystal Dimensions (mm)	0.31 × 0.41 × 0.50	0.21 × 0.28 × 0.35
Crystal System	Tetragonal	Tetragonal
Space Group	<i>I4/m</i>	<i>I4/m</i>
<i>a</i> (Å)	33.0404(10)	32.9311(3)
<i>b</i> (Å)	33.0404(10)	32.9311(3)
<i>c</i> (Å)	54.083(2)	54.0192(6)
α (°)	90	90
β (°)	90	90
γ (°)	90	90
<i>V</i> (Å ³)	59041(4)	58581.7(12)
<i>Z</i>	2	2
ρ_{calc} (g cm ⁻³)	0.567	0.572
<i>F</i> (000)	10320	10368
μ (mm ⁻¹)	3.23	1.08
Temperature (K)	120.0(1)	120.0(1)
ϑ_{max} [°]	55.9	73.5
Total Reflections	72847	189192
Independent Reflections	19170	29635
Reflections (<i>I</i> _o > 2σ[<i>I</i> _o])	9172	22239
<i>R</i> _{int}	0.092	0.048
Parameters	733	733
Restraints	99	222
GooF (<i>F</i> ²)	1.03	1.03
<i>R</i> ₁ (<i>I</i> _o > 2σ[<i>I</i> _o])	0.086	0.090
<i>R</i> ₁ (all reflections)	0.151	0.114
<i>wR</i> ₂ (<i>I</i> _o > 2σ[<i>I</i> _o])	0.269	0.263
<i>wR</i> ₂ (all reflections)	0.312	0.298
Largest Peak (e Å ⁻³)	0.54	0.71
Largest Hole (e Å ⁻³)	-0.29	-0.57
CCDC Number	2384881	2384882

Table S1: The crystallographic data for **1** and **2**.

^a Isostructural to the compound reported in Ref. 1, but no corresponding deposition in the CSD database could be found.

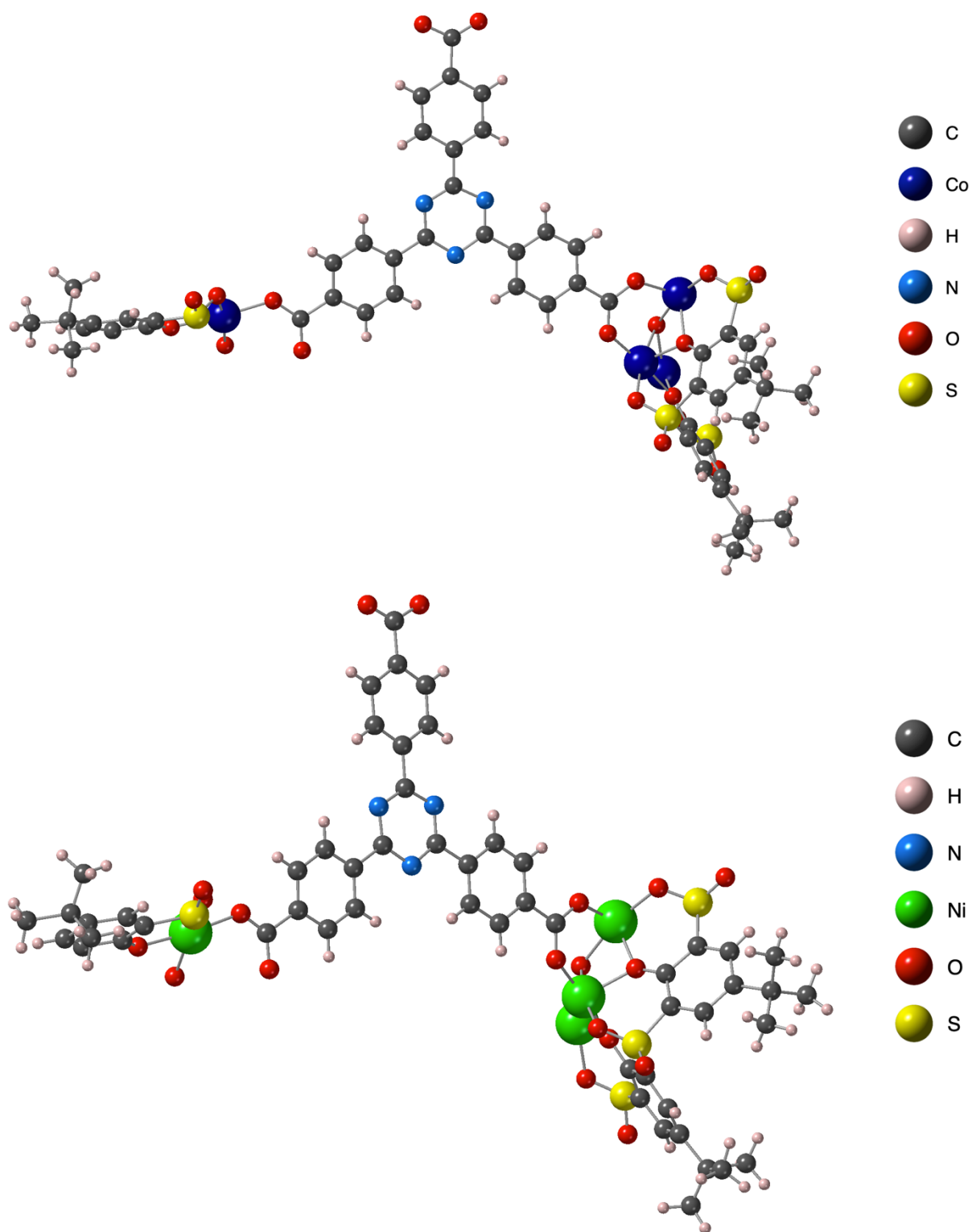


Figure S1. For **1** (top) and **2** (bottom), view of the asymmetric units, obtained from X-ray diffraction data.

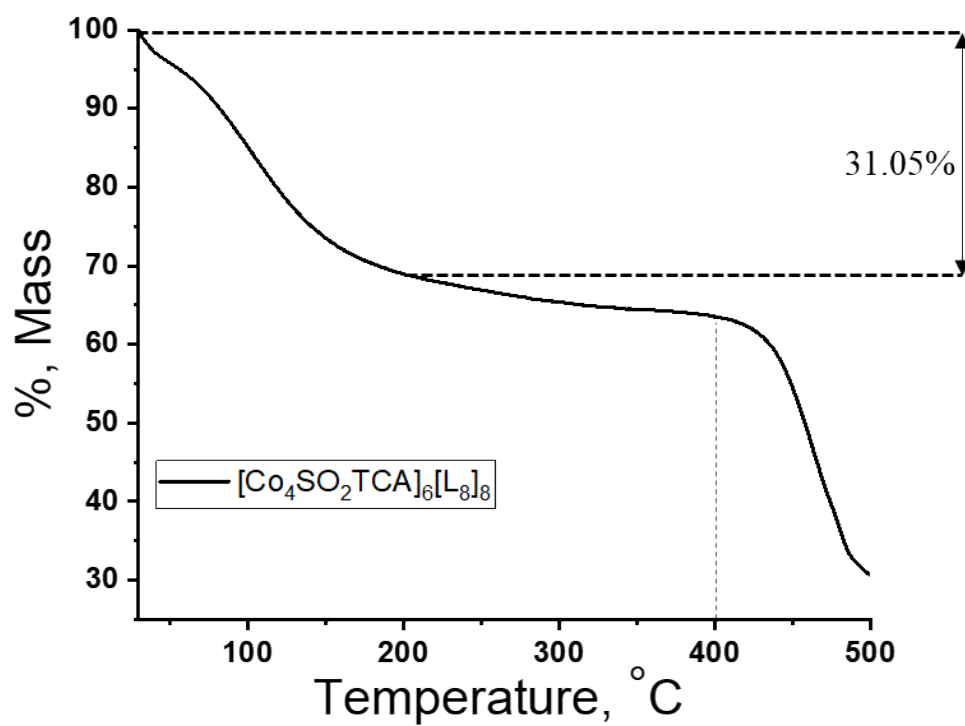


Figure S2. TGA trace for freshly prepared 1

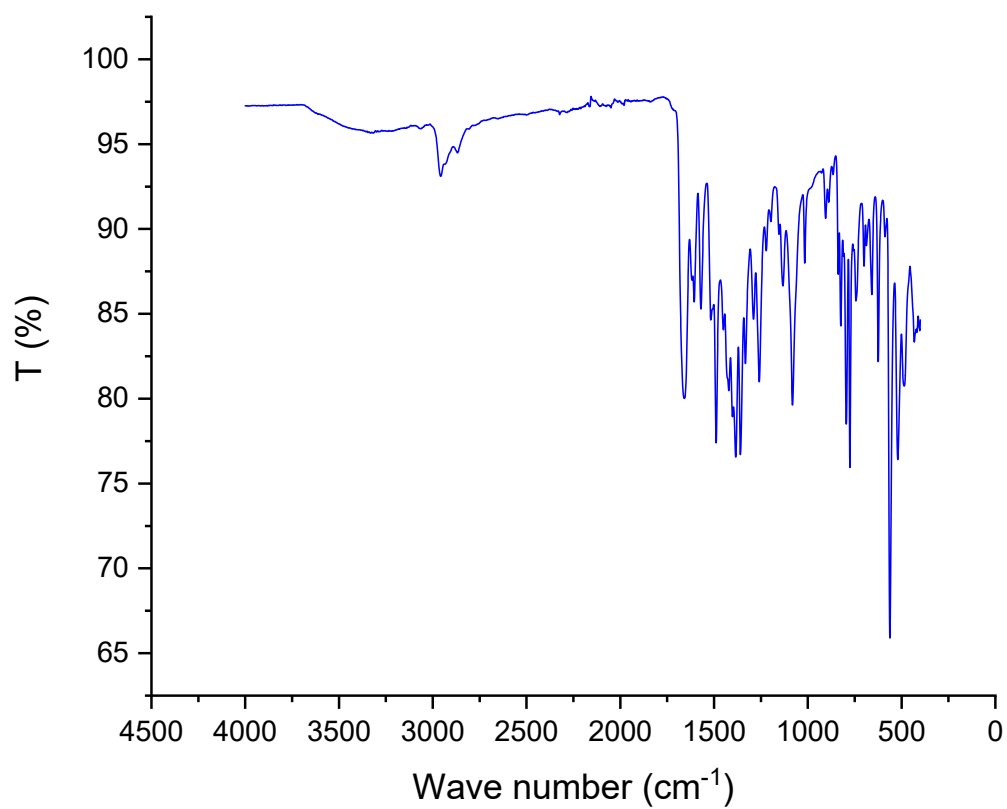


Figure S3. FTIR data for activated for **1**

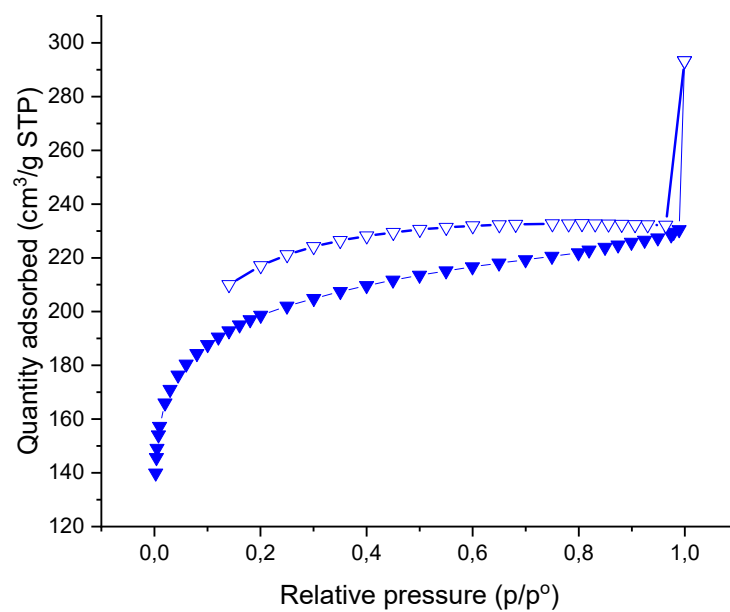


Figure S4. $N_{2(g)}$ adsorption-desorption isotherm at 77K for **1** (filled symbols – adsorption, hollow symbols – desorption)

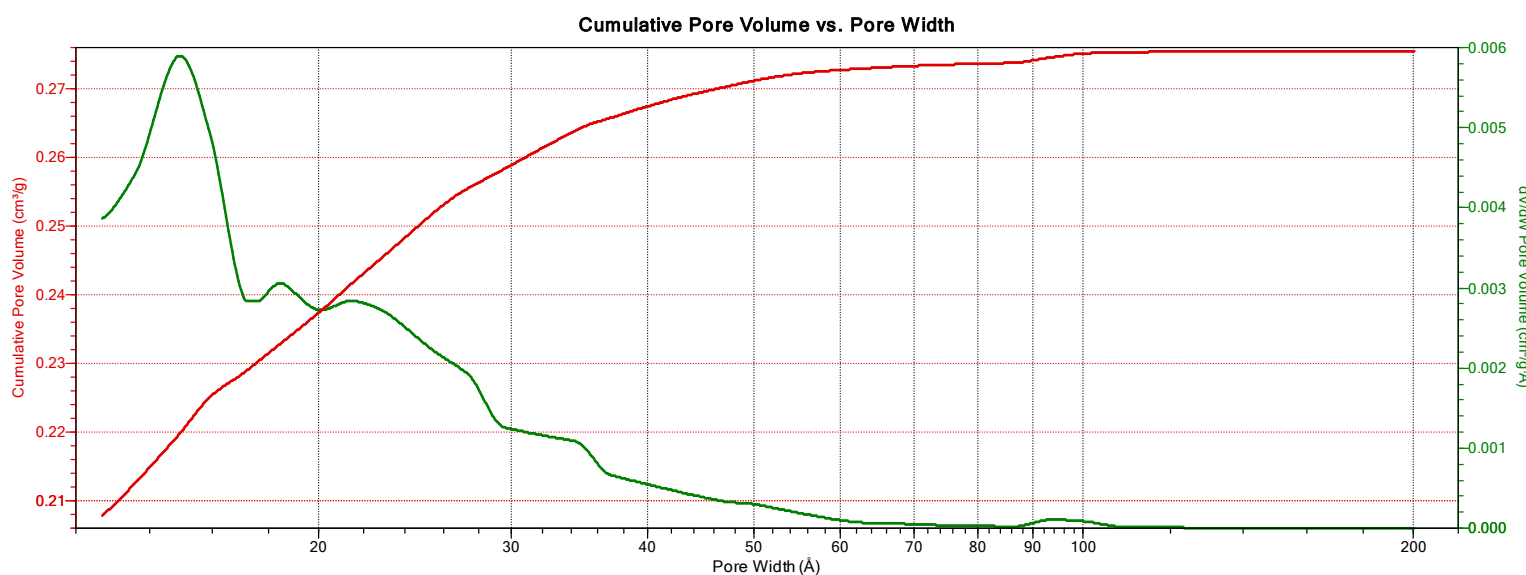


Figure S5. Pore size distribution (green line) and cumulative pore volumes (red line) for **1** calculated by N_2 -DFT Model using MicroActive software

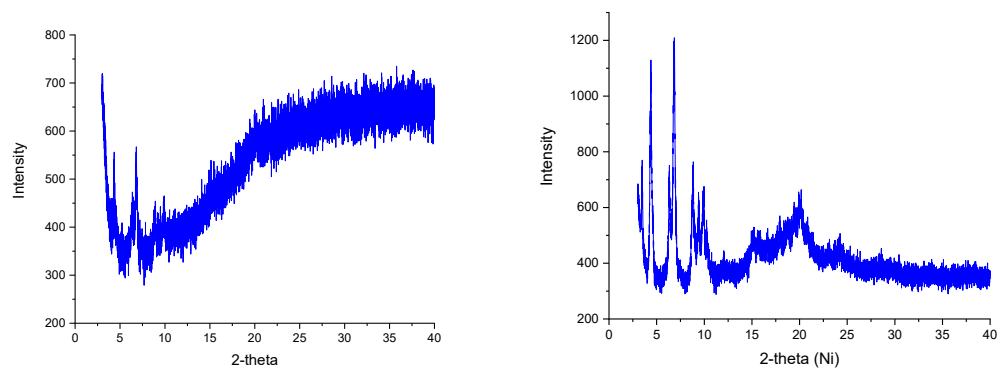


Figure S6. XRPD pattern for **1** (left) and **2** (right)