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Supplementary information

2D networks of metallo-capsules and other coordination polymers from a
hexapodal ligand.

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- 1. NMR spectroscopy**
- 2. Mass spectrometry**
- 3. Infrared spectroscopy**
- 4. Thermogravimetric Analysis**
- 5. Elemental analysis**
- 6. Single crystal X-ray diffraction**

1. NMR spectra

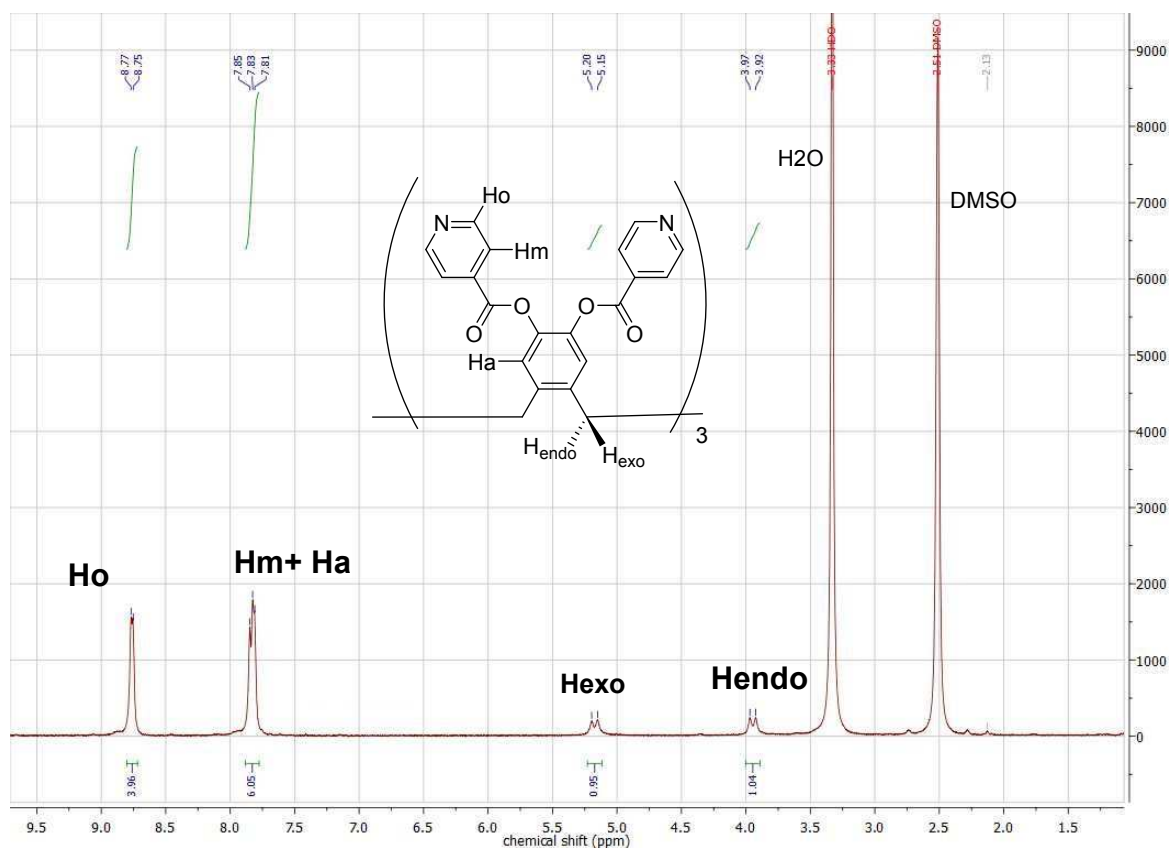


Figure S1. ^1H NMR spectrum (d_6 -DMSO, 300 MHz) of ligand hexakis(isonicotinoyl)cyclotricatechylene, L1

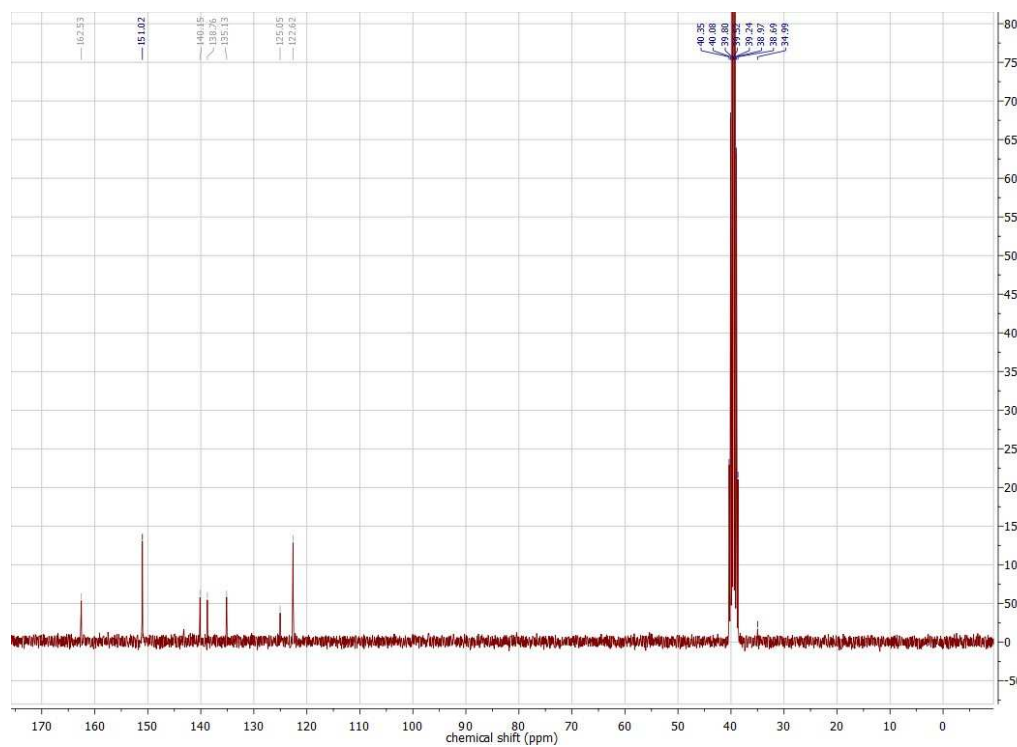


Figure S2. ^{13}C NMR spectrum (d_6 -DMSO, 75 MHz) of L1.

2. Mass spectrometry

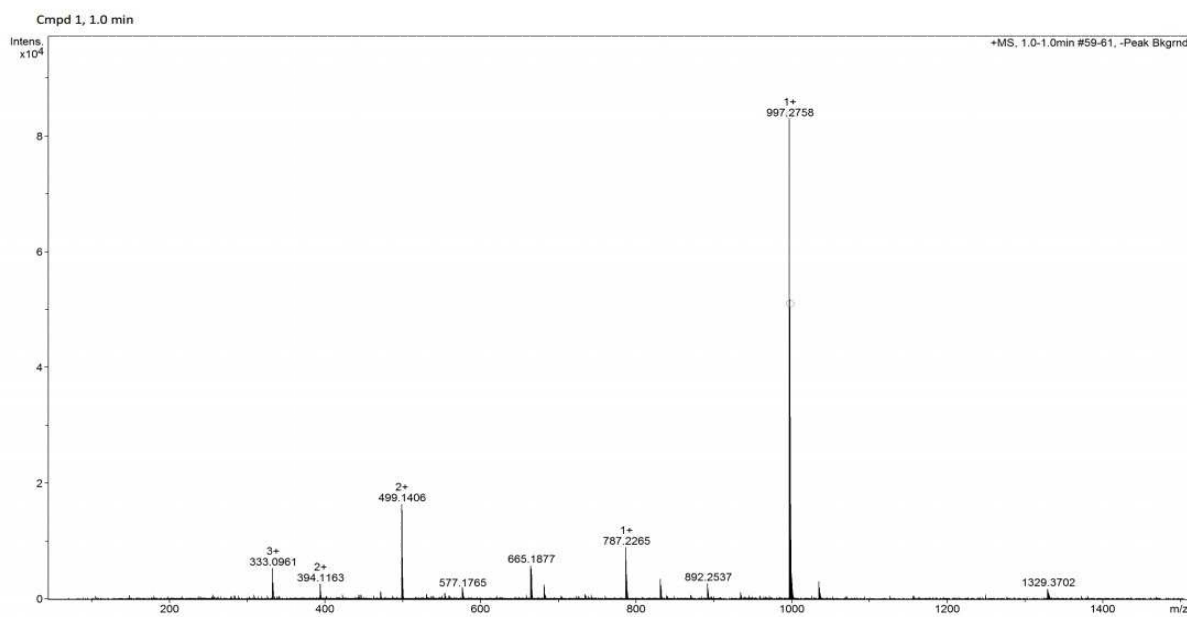


Figure S3. Mass spectrum of L1.

3. Infrared spectroscopy

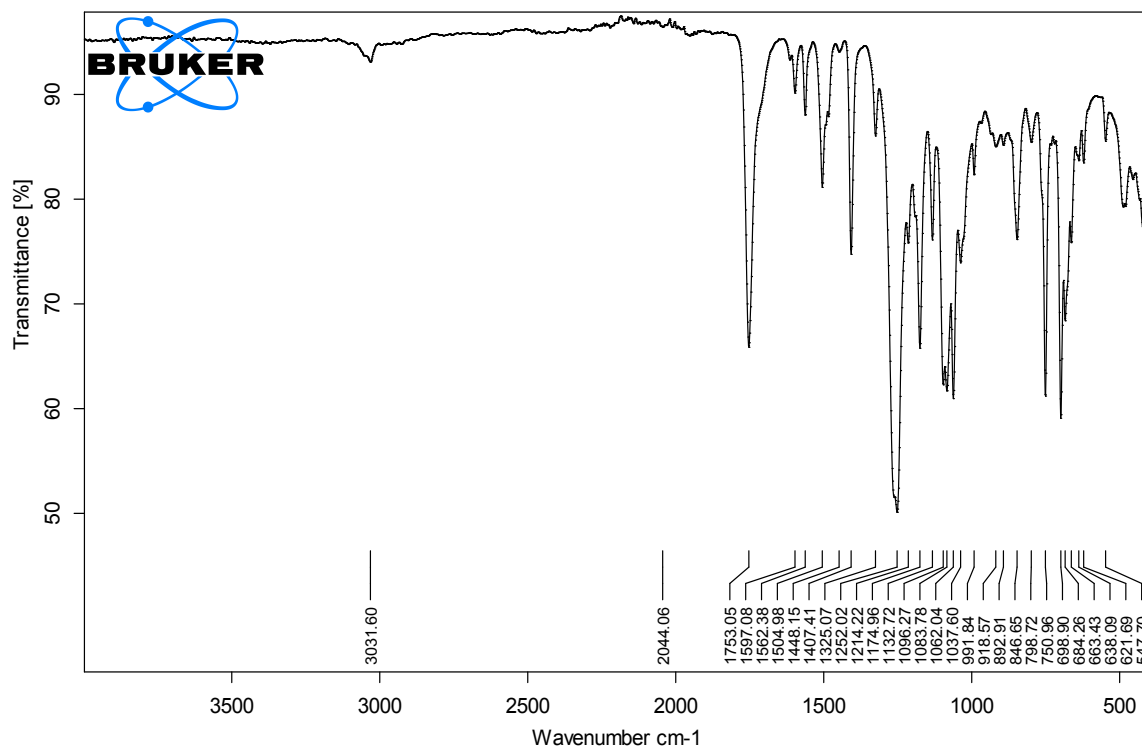


Figure S4. Infrared spectrum of L1.

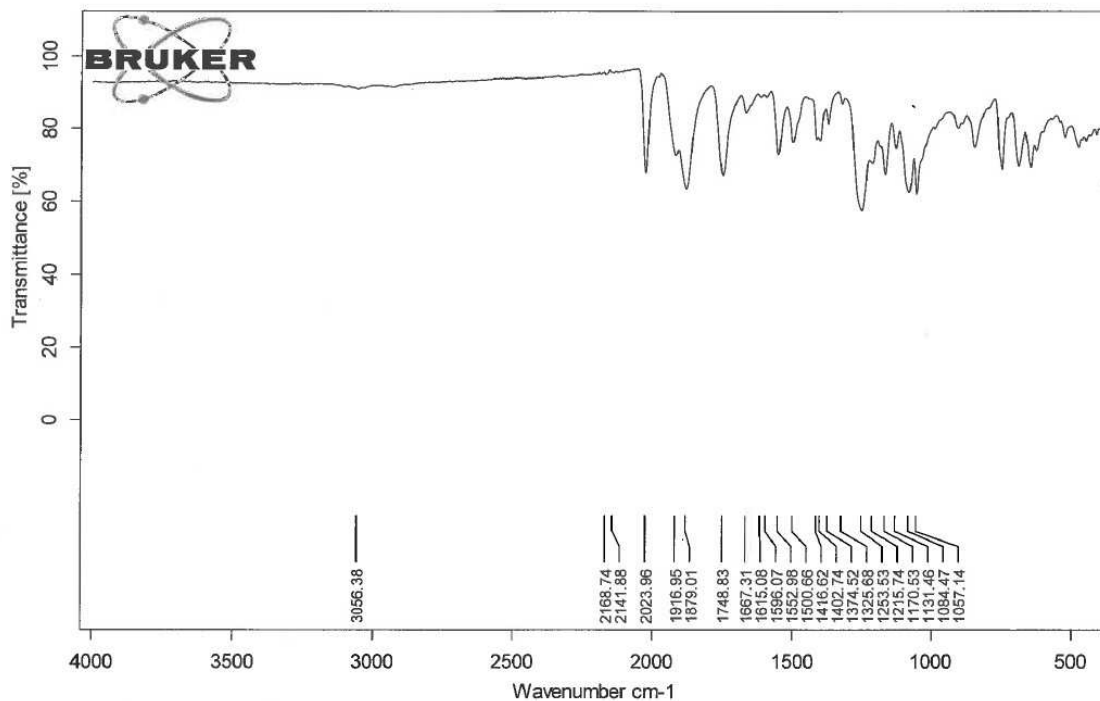


Figure S5. Infrared spectrum of $[\text{Re}_3(\text{L}1)_2\text{Br}_3(\text{CO})_3] \cdot n(\text{CH}_3\text{NO}_2) \cdot m(\text{H}_2\text{O})$ **1**.

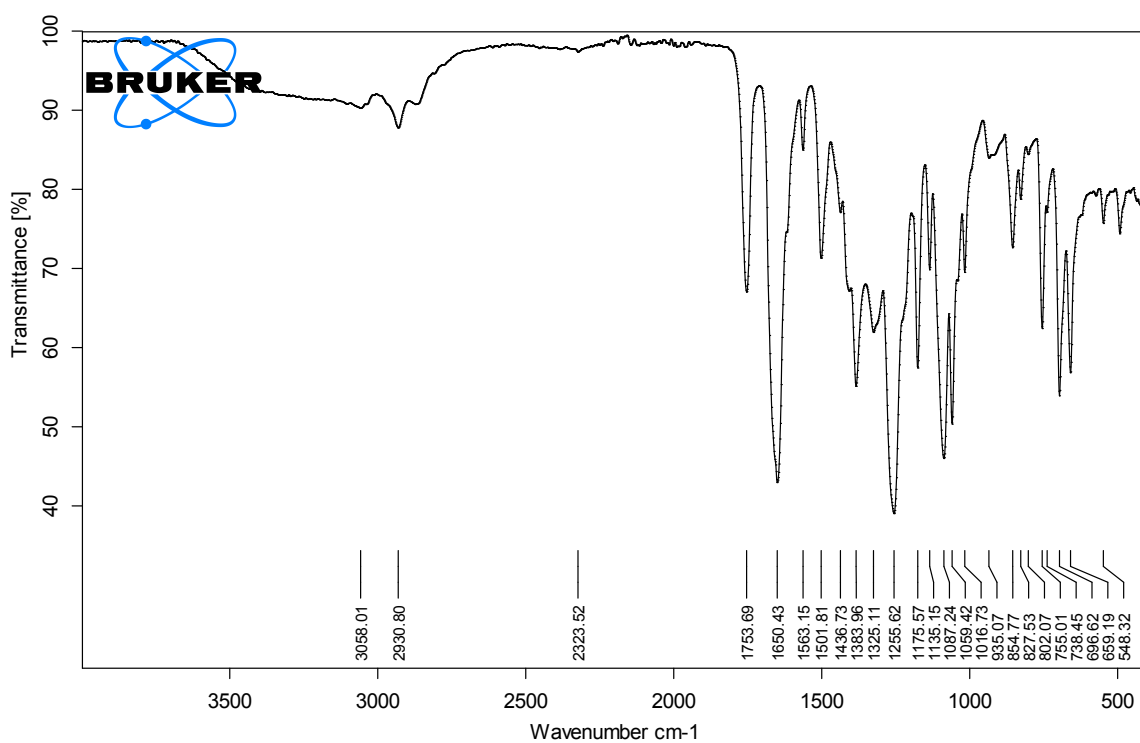
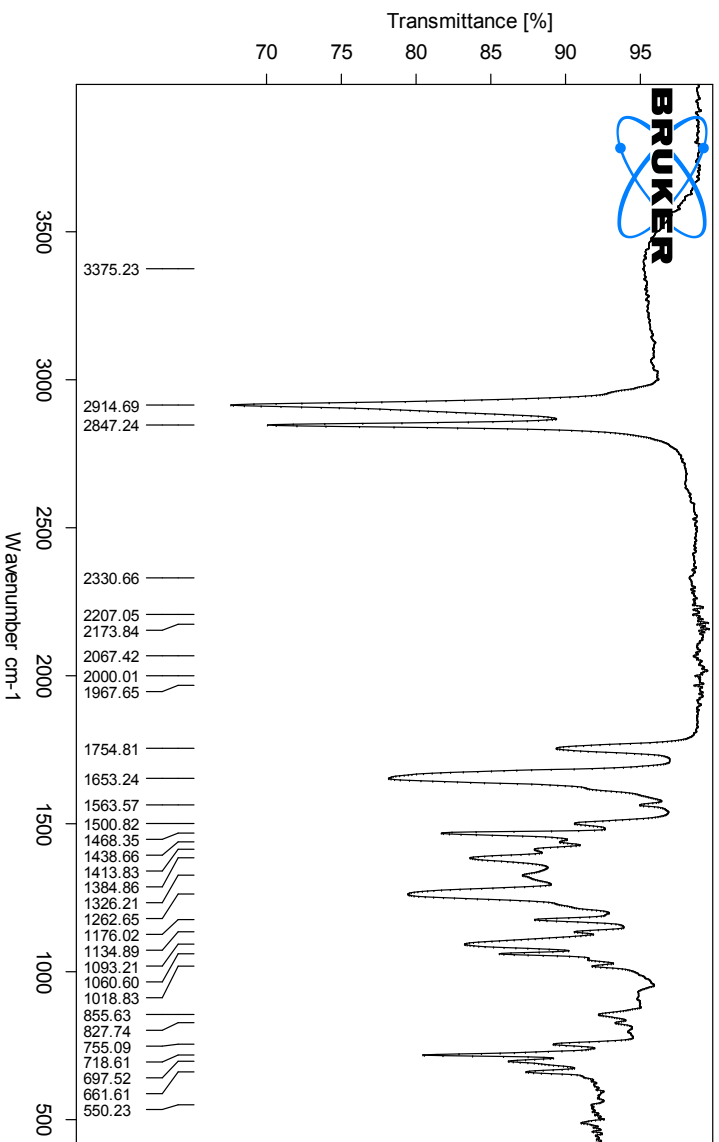
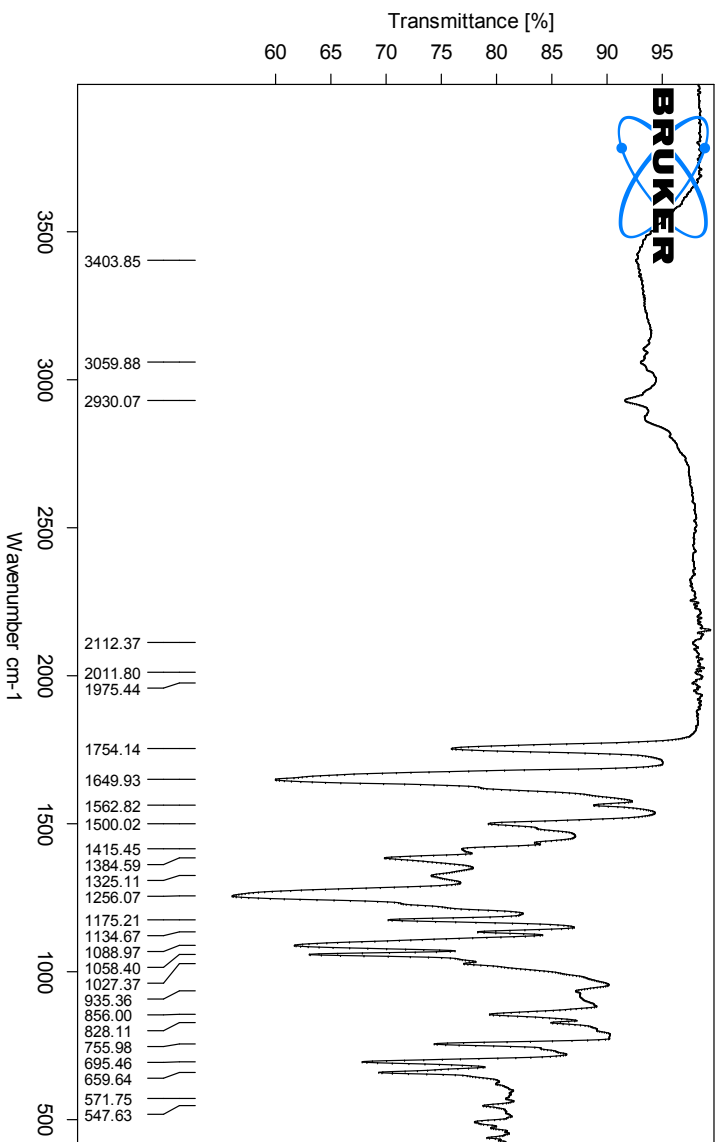
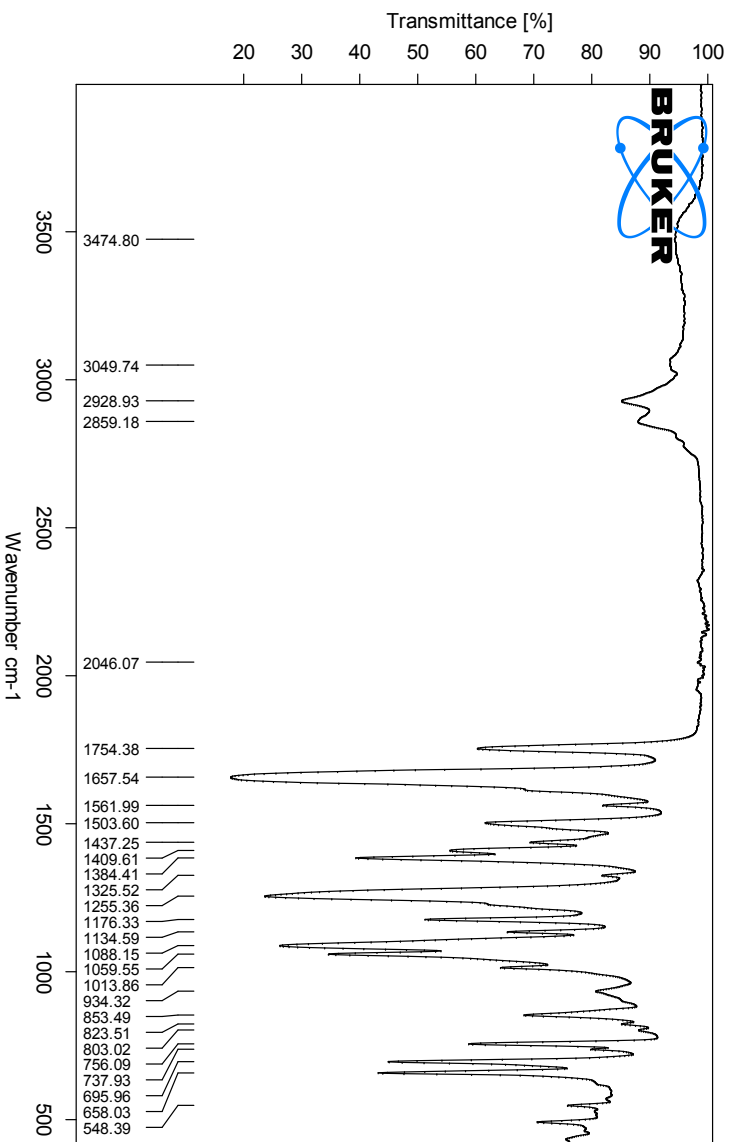
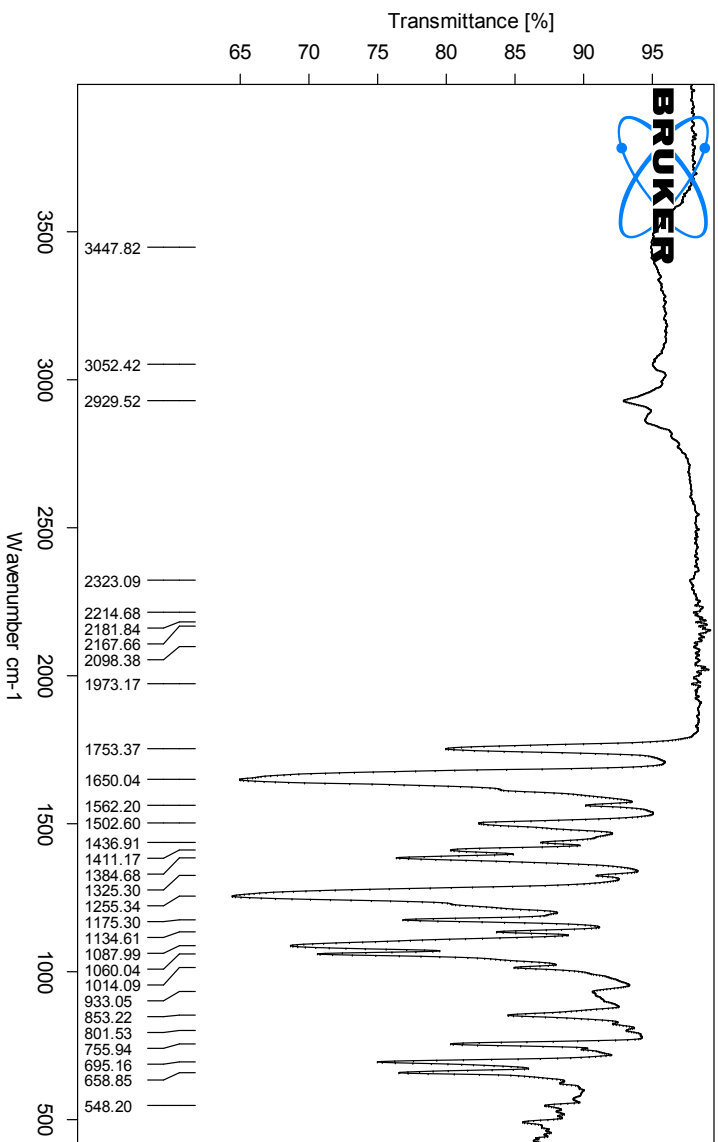


Figure S6. Infrared spectrum of $[\text{Co}_3(\text{H}_2\text{O})_6(\text{L}1)_2] \cdot 6(\text{NO}_3) \cdot n(\text{DMF})$ **2a**.





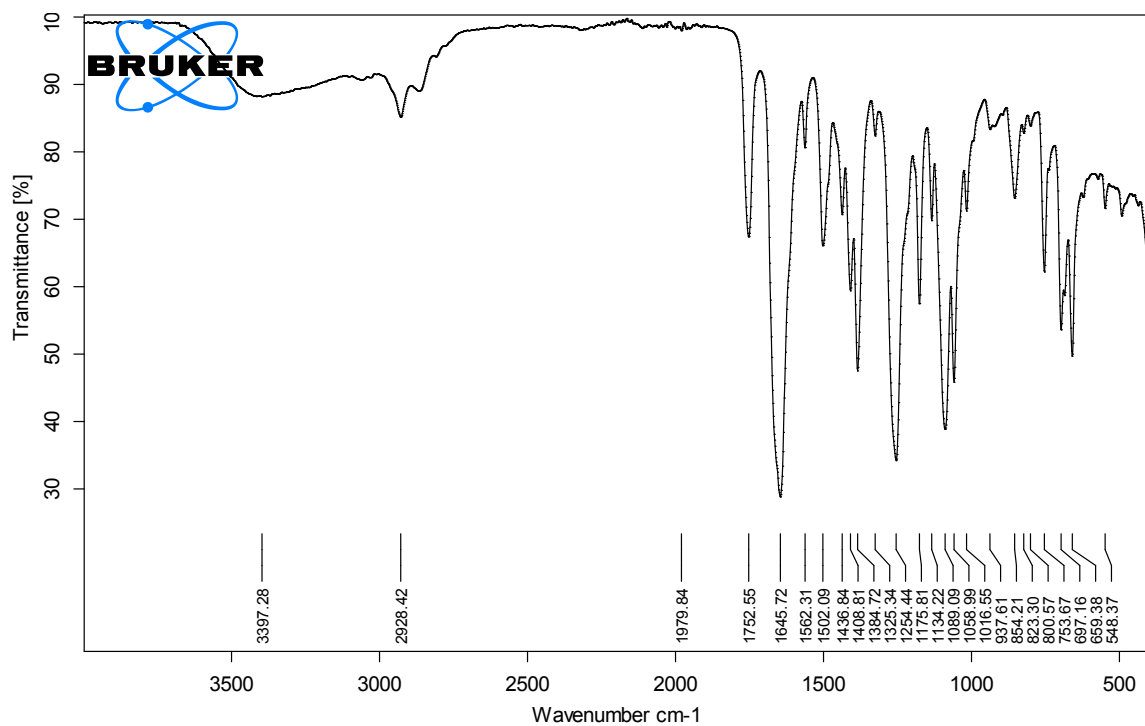


Figure S11. Infrared spectrum of $[\text{Co}_3]_{1.5}(\text{H}_2\text{O})_{4.5}(\text{L1})_2] \cdot 4.5\text{I} \cdot m(\text{DMF})$ **4**.

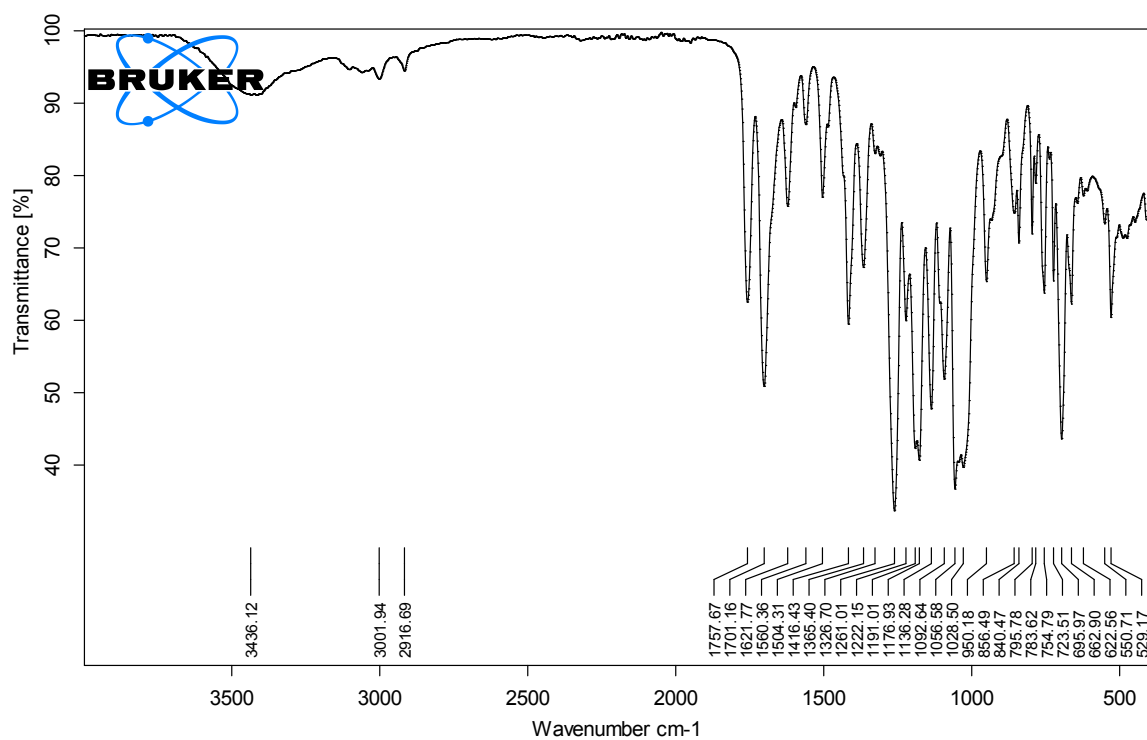


Figure S12. Infrared spectrum of $[\text{Cu}_2(\text{L1})(\text{TFA})_3(\text{isonic})] \cdot n(\text{DMF})$ **5**, where tfa = trifluoroacetate and isonic = isonicotinate.

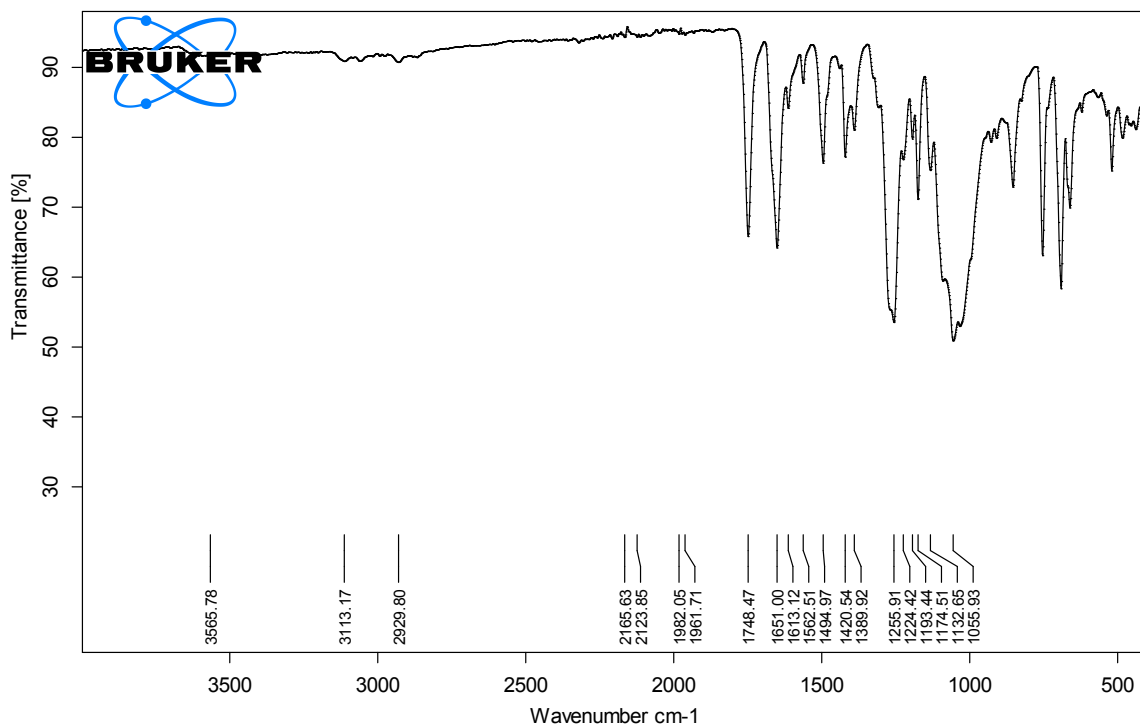


Figure S13. Infrared spectrum of $[Ag_2(L1)(DMF)_2] \cdot 2BF_4 \cdot 2(H_2O) \cdot 6(DMF)$ 6.

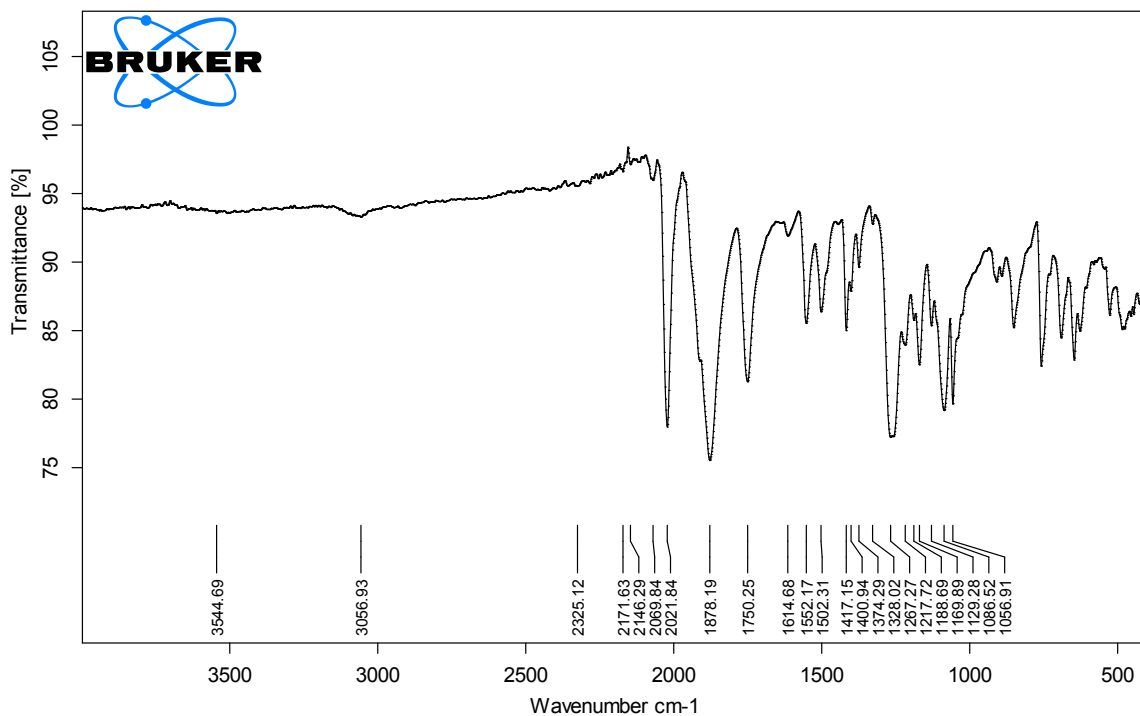
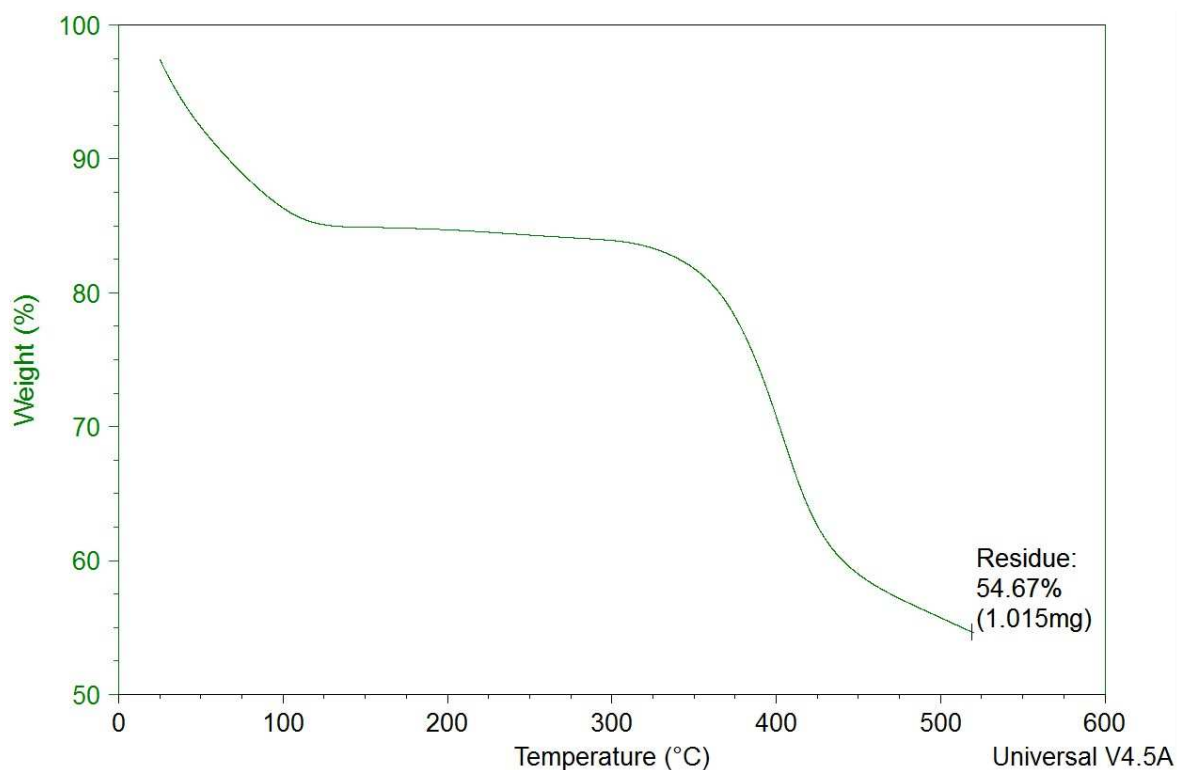


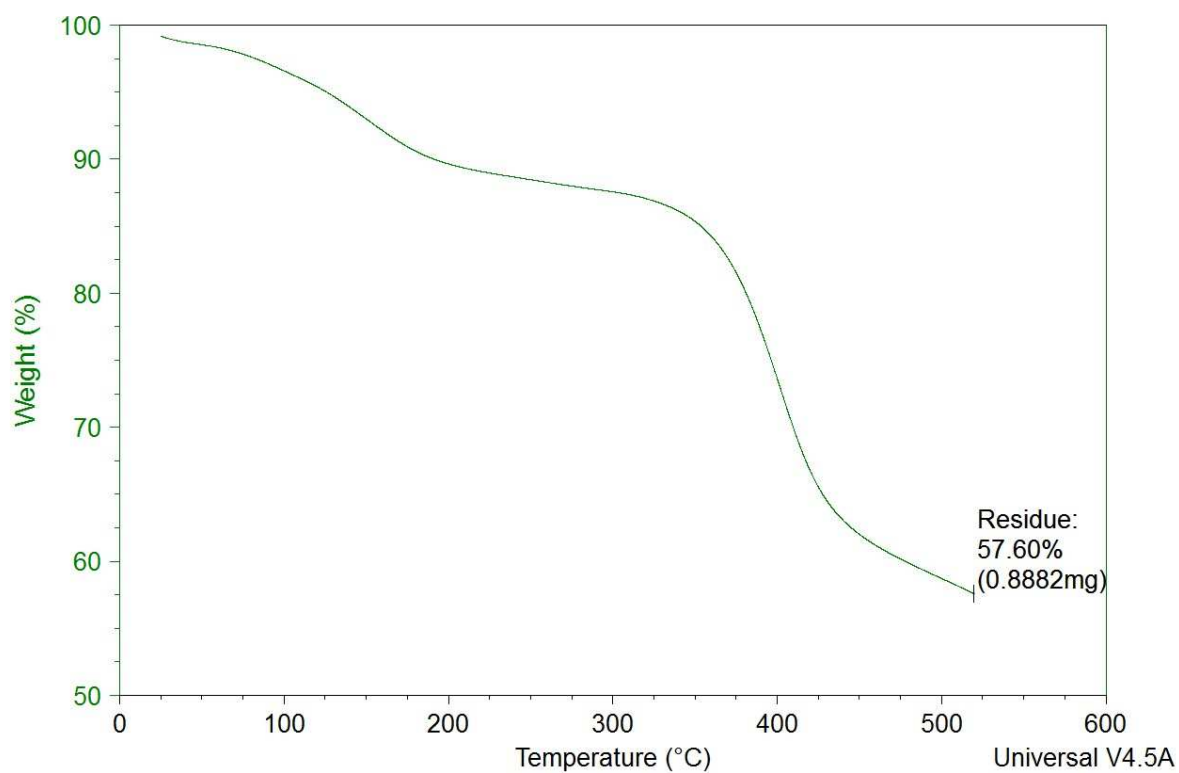
Figure S14. Infrared spectrum of $[Re_3(L1)_2Br_3(CO)_3] \cdot n(CH_3NO_2) \cdot m(H_2O)$ 1 after I_2 up-take.

4. Thermogravimetric Analysis

$[\text{Re}_3(\text{L1})_2\text{Br}_3(\text{CO})_3] \cdot n(\text{CH}_3\text{NO}_2) \cdot m(\text{H}_2\text{O})$ **1**



(a)



(b)

Figure S15. TGA of (a) $[\text{Re}_3(\text{L1})_2\text{Br}_3(\text{CO})_3] \cdot n(\text{CH}_3\text{NO}_2) \cdot m(\text{H}_2\text{O})$ **1**; and (b) $\text{Re}_3(\text{L1})_2\text{Br}_3(\text{CO})_3] \cdot n(\text{CH}_3\text{NO}_2) \cdot m(\text{H}_2\text{O})$ **1** after I_2 up-take.

[Co₃(H₂O)₆(L1)₂]-6(NO₃)·n(DMF) 2a

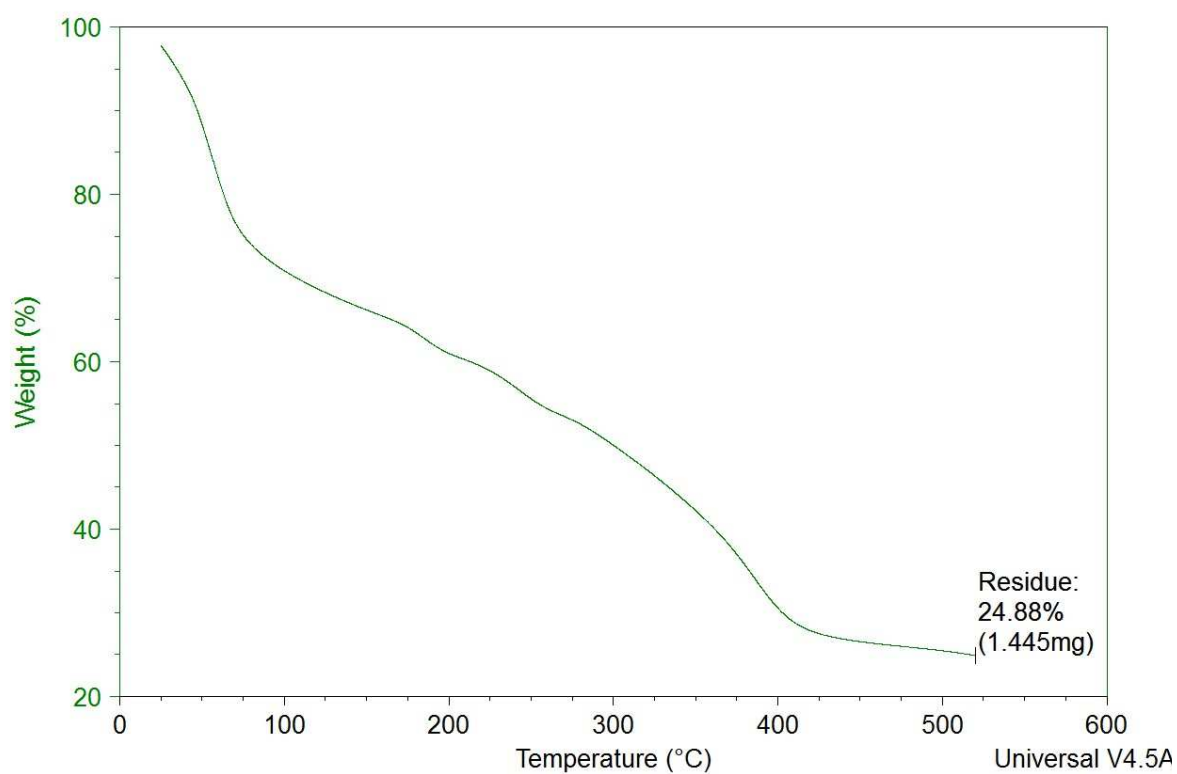


Figure S16. TGA of [Co₃(H₂O)₆(L1)₂]-6(NO₃)·n(DMF) 2a.

[Ni₃(H₂O)₆(L1)₂]-6(NO₃)·n(DMF) 2c

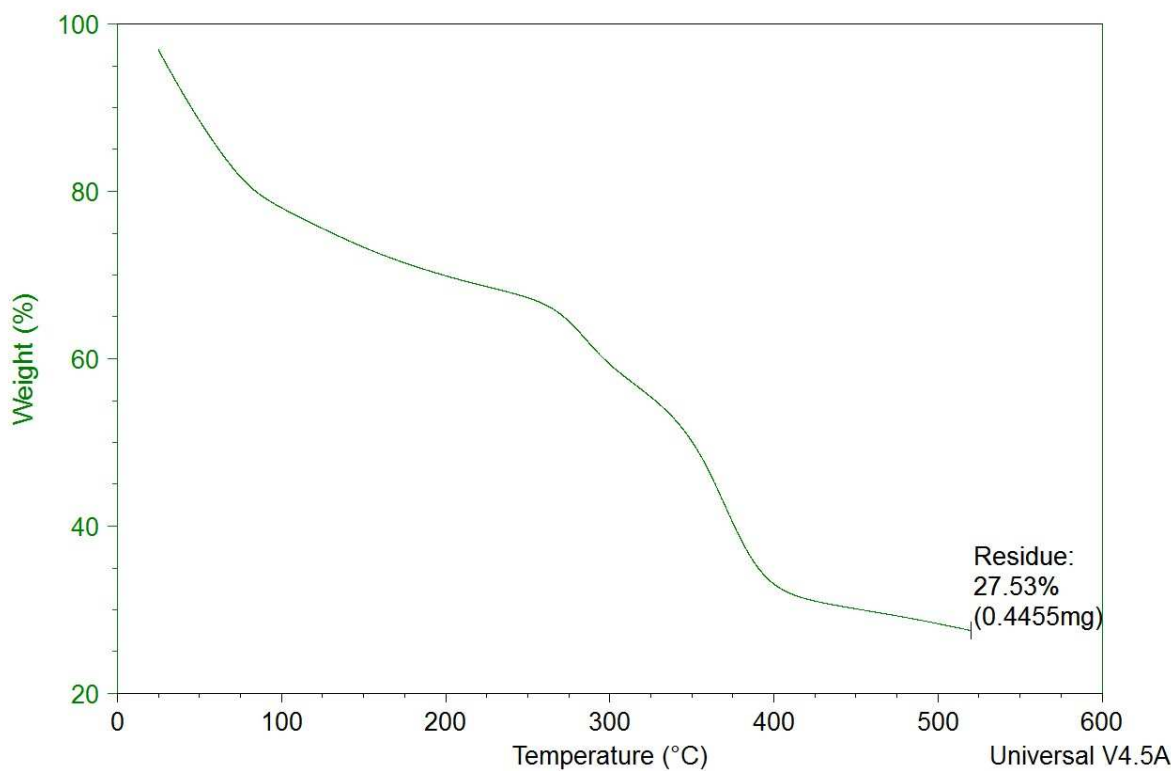


Figure S17. TGA of [Ni₃(H₂O)₆(L1)₂]-6(NO₃)·n(DMF) 2c.

[Co₃Cl₆(L1)₂] \cdot n(DMF) 3a

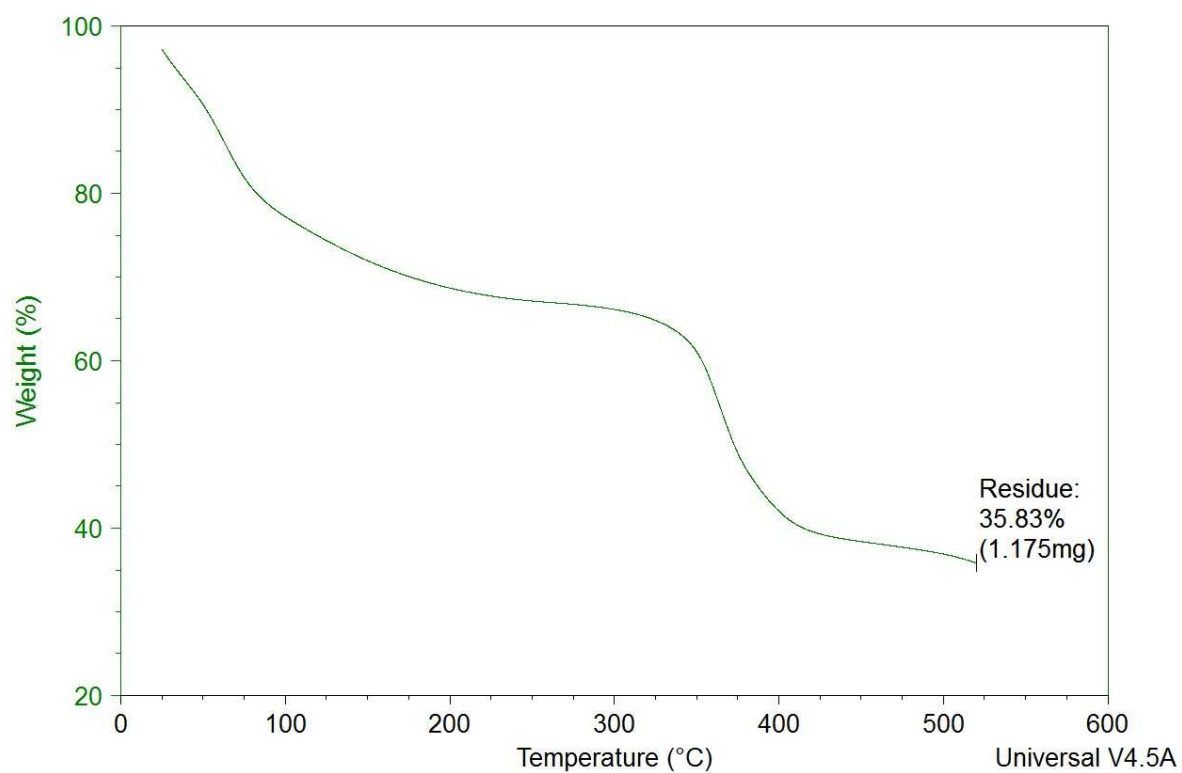


Figure S18. TGA of [Co₃Cl₆(L1)₂] \cdot n(DMF) 3a.

[Co₃Br₆(L1)₂] \cdot n(DMF) 3b

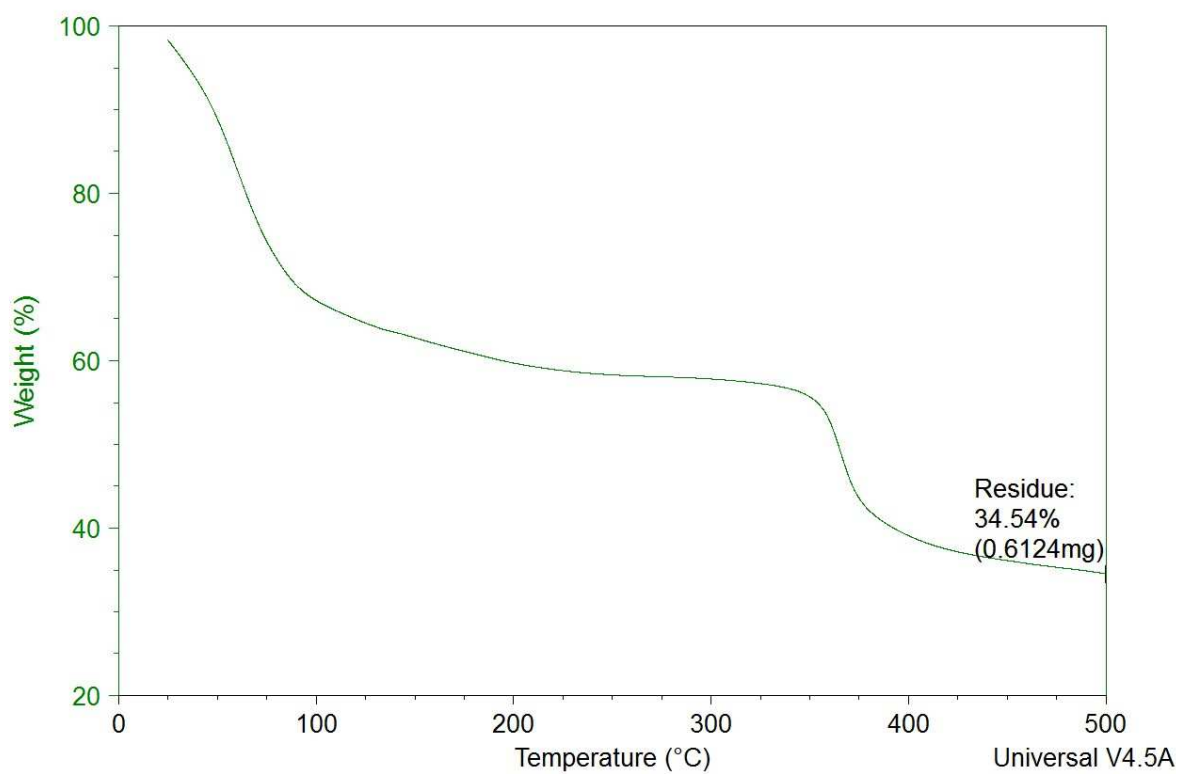


Figure S19. TGA of [Co₃Br₆(L1)₂] \cdot n(DMF) 3b.

[Co₃L_{1.5}(H₂O)_{4.5}L1₂]₂·4.5l·m(DMF) 4

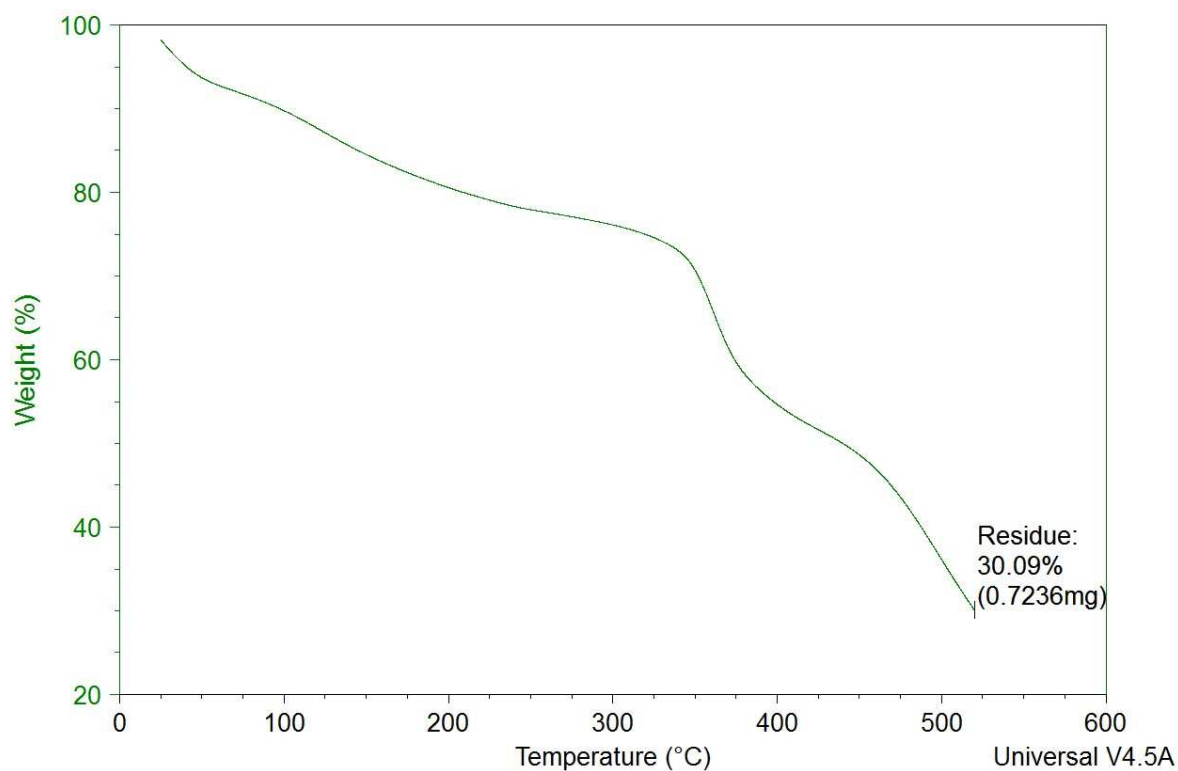


Figure S20. TGA of [Co₃L_{1.5}(H₂O)_{4.5}L1₂]₂·4.5l·m(DMF) 4.

[Cu₂(L1)(TFA)₃(isonic)]·n(DMF) 5

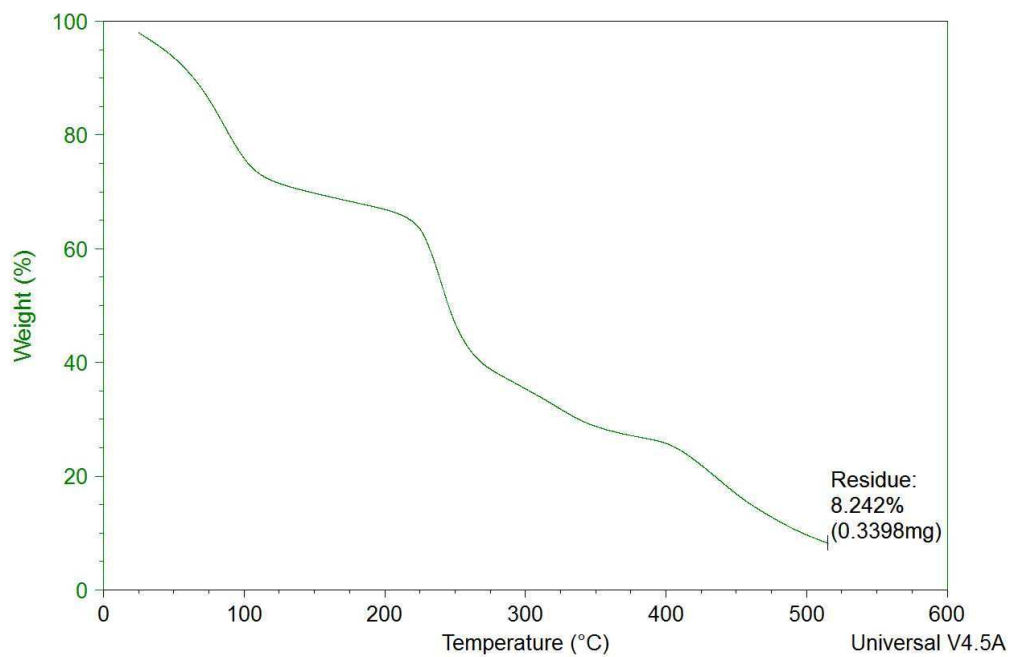


Figure S21. TGA of [Cu₂(L1)(TFA)₃(isonic)]·n(DMF) 5.

[Ag₂(L1)(DMF)₂]₂·2BF₄·2(H₂O)·6(DMF) 6

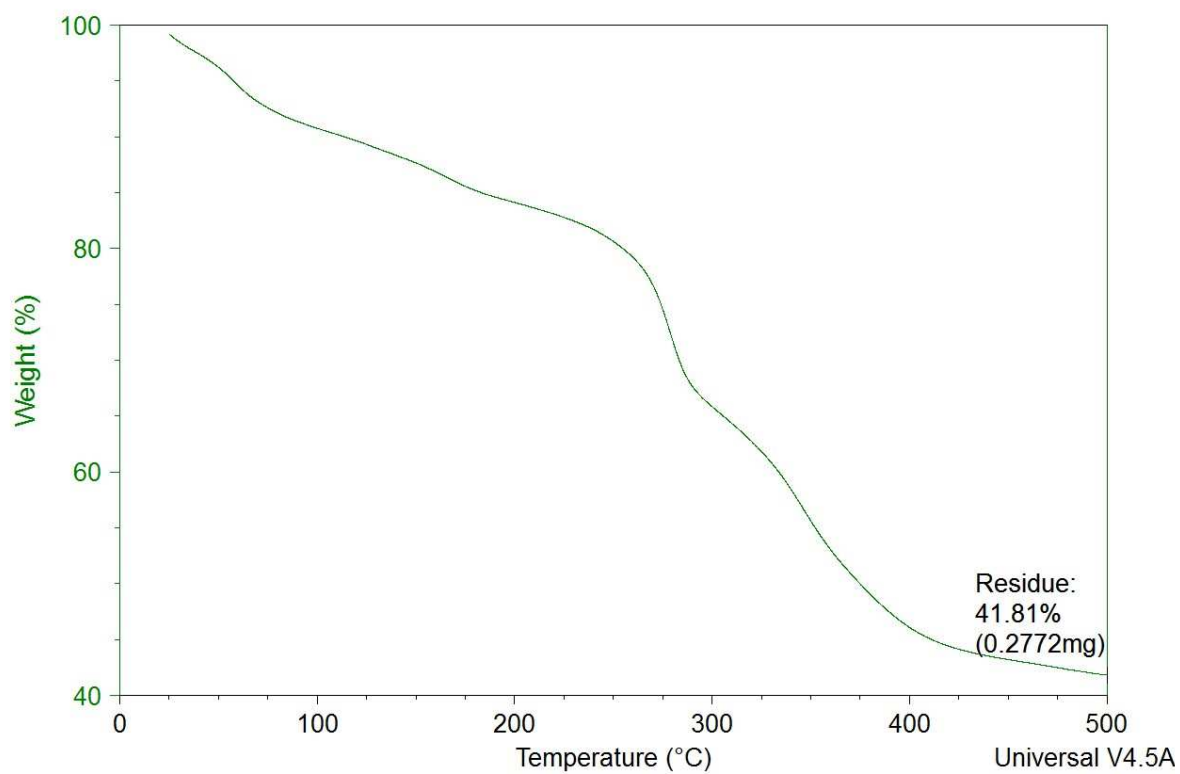


Figure S22. TGA of [Ag₂(L1)(DMF)₂]₂·2BF₄·2(H₂O)·6(DMF) 6.

5. Elemental analysis

CHN analysis

CHN analysis indicated high levels of solvation for materials **2-5** as expected, and satisfactory analysis cannot be established in some cases due to unknown levels of solvation (common for these types of materials) and small amounts of some amorphous powdered material. Presence of DMF and water are apparent from IR. Results obtained are given below (see main text for compound **1**, **4** and **6**).

Compound	Calc (%) CHN for desolvated material*	Calc (%) CHN for complex·n(DMF)·m(H ₂ O)	Found (%) CHN
2a	C 51.64, H 3.20, N 9.51	n = 10, m = 20 C 46.20, H 5.23, N 10.48	C 44.39, H 2.65, N 10.88
2b	C 51.37, H 3.18, N 9.46	n = 10, m = 20 C 46.03, H 5.21, N 10.44	C 45.18, H 2.14, N 6.56
2c	C 51.65, H 3.20, N 9.52	n = 9, m = 15 C 47.31, H 4.99, N 10.57	C 47.68, H 4.37, N 9.51
3a	C 57.43, H 3.05, N 7.05	n = 10, m = 20 C 49.76, H 5.28, N 8.87	C 49.54, H 4.05, N 7.65
3b	C 51.67, H 2.74, N 6.34	n = 10, m = 20 C 46.21, H 4.91, N 8.24	C 46.84, H 3.36, N 6.66
5	C 52.26, H 2.54, N 6.19	n = 10, m = 15 C 45.96, H 5.46, N 9.21	C 45.60, H 4.00, N 8.35

* Lattice solvent excluded

SEM-EDX

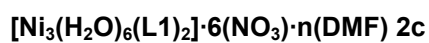
SEM/EDX analysis data for compound **1** after I₂ up-take (Weight %).

Sample reference	Carbon	Oxygen	Aluminium	Bromine	Iodine	Rhenium
particle 1	24.24	15.51	2.77	5.15	16.44	35.89
particle 2	24.56	17.74	1.97	6.20	14.07	35.47
particle 3	27.39	20.04	2.31	4.73	10.80	34.73
* particle 4	30.32	16.04	0.68	3.15	27.84	21.98
particle 5	23.68	18.50	2.04	4.88	14.13	36.77
particle 6	24.86	17.81	1.40	7.33	11.44	37.17
particle 7	30.79	17.99	0.44	7.16	13.96	29.65
particle 8	35.10	18.24	2.07	5.70	10.10	28.79
many particles	36.36	18.32	2.65	7.86	10.23	24.58
Ground sample 1	39.10	20.76	0.76	6.72	8.45	24.21
Ground sample 2	39.46	20.60	1.17	5.80	7.77	25.20
Ground sample 3	39.41	21.20	1.41	4.78	9.05	24.14
Ground sample 4	41.14	20.93	0.80	6.37	8.02	22.57

* Outlier excluded from analyses.

6. Single crystal X-ray diffraction

6.1 Unit cell information for isostructural complexes



Unit cell parameters Monoclinic $a = 35.0709(14)$, $b = 20.2572(15)$, $c = 33.454(3)$ Å. $\beta = 110.482(7)^\circ$

6.2 Additional diagrams of single crystal structures

Hexakis(isonicotinoyl)cyclotricatechylene (L1)

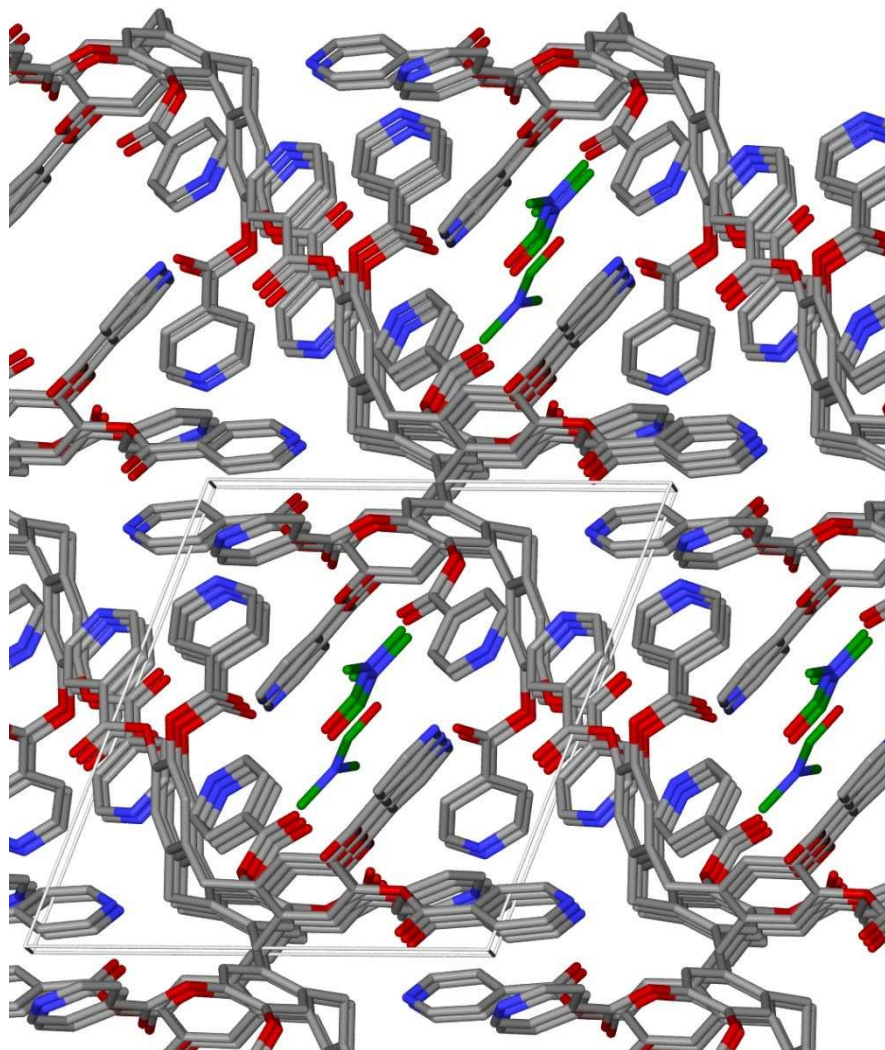
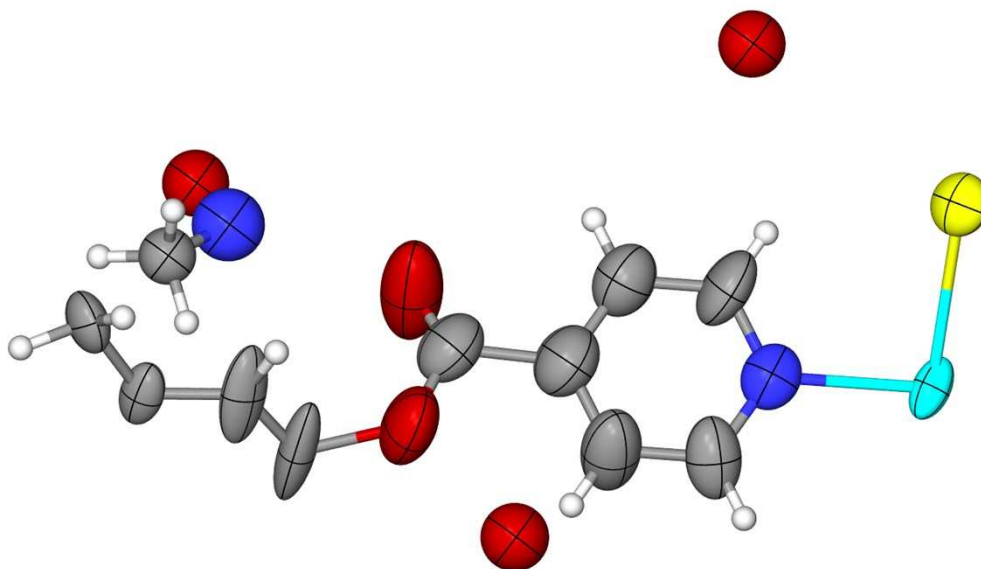
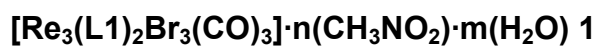
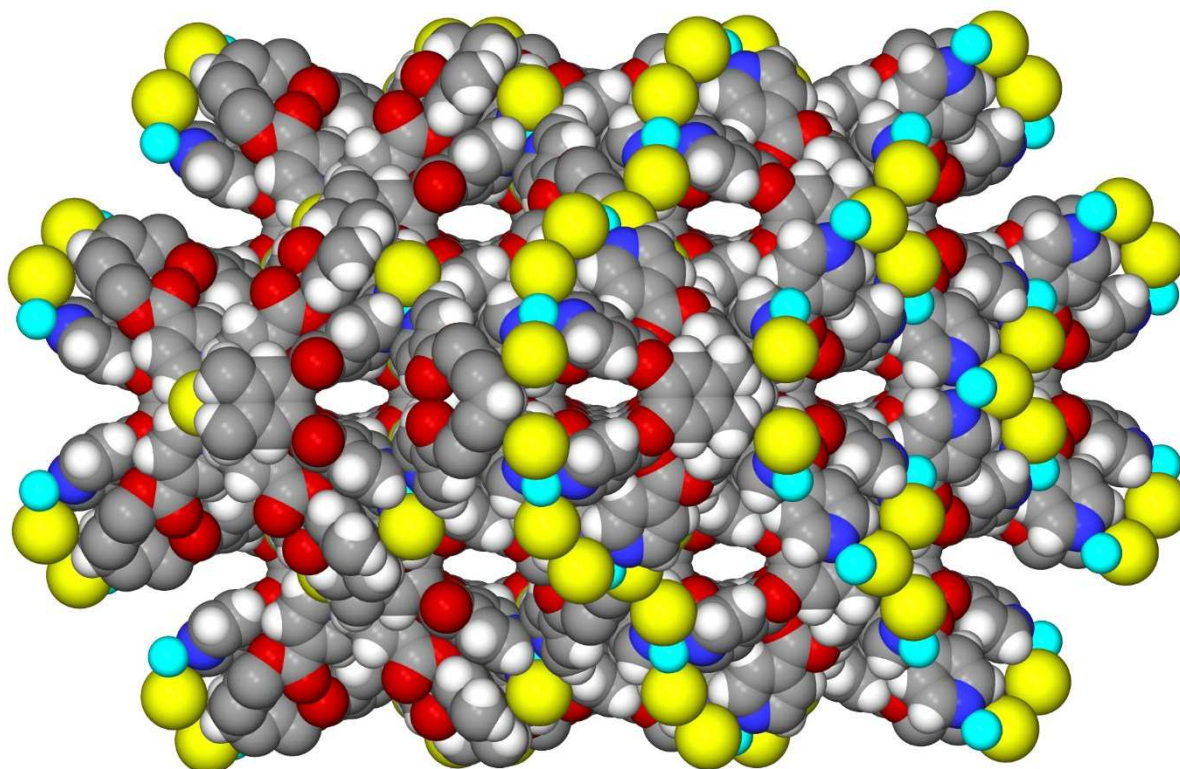


Figure S23: Packing diagram of L1-DMF viewed down b . Hydrogen atoms excluded for clarity and DMF shown in green.



*Figure S24: Asymmetric unit of **1** with ellipsoids shown at 50% probability levels. Isotropic atoms are solvent CH_3NO_2 and partly occupied water positions.*



*Figure S25: Packing diagram of **1b** in space-filing mode showing channels in structure.*

[Co₃(H₂O)₆(L1)₂·6(NO₃)·n(DMF) 2a

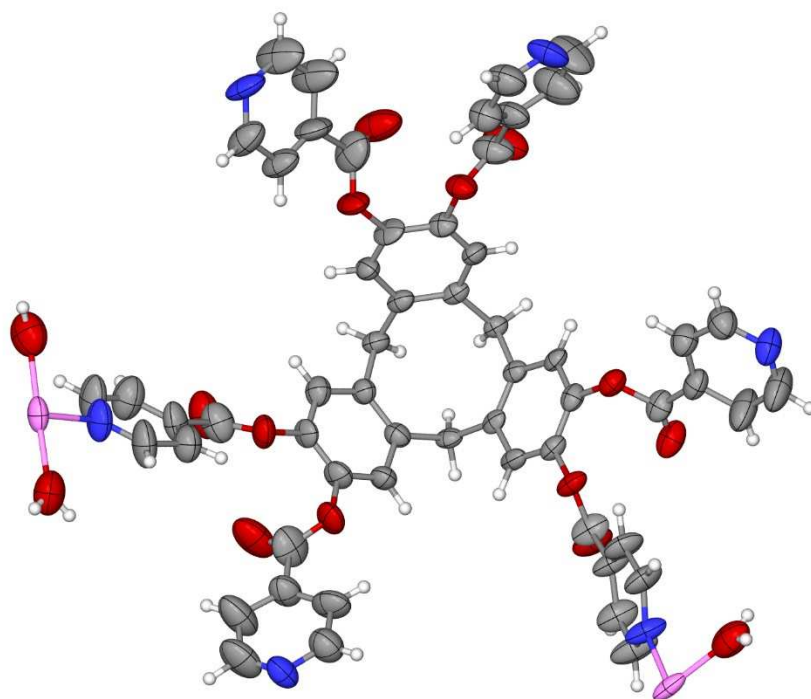


Figure S26: Asymmetric unit of 2a with ellipsoids shown at 50% probability levels.

[Cu₃(H₂O)₆(L1)₂·6(NO₃)·n(DMF) 2b

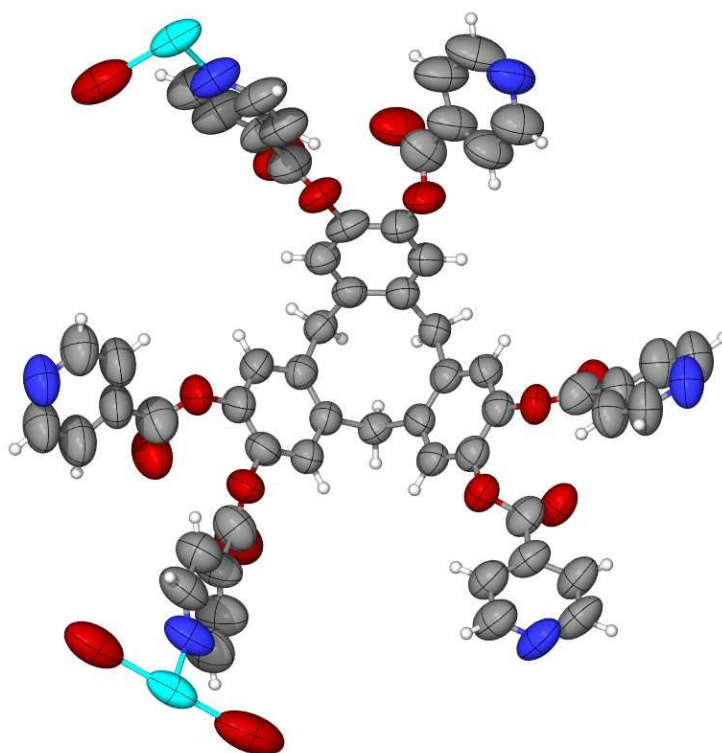


Figure S27: Asymmetric unit of 2b with ellipsoids shown at 50% probability levels.

$[\text{Co}_3\text{Cl}_6(\text{L1})_2] \cdot n(\text{DMF})$ **3a**

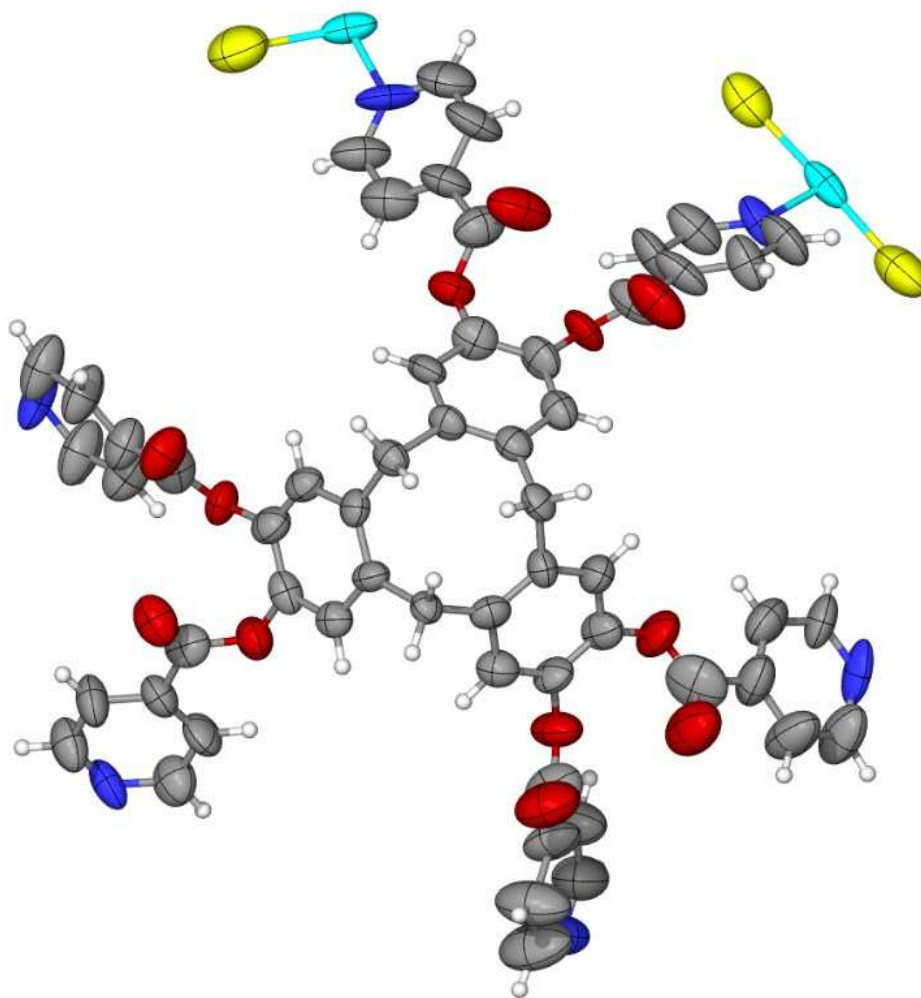
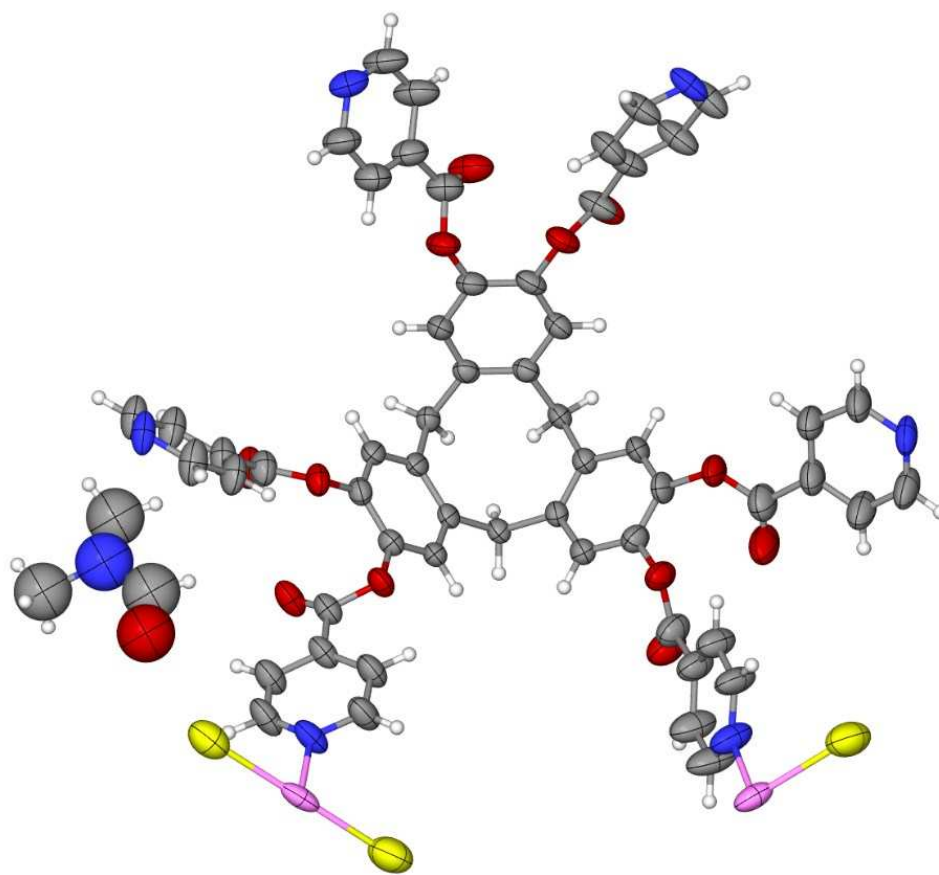
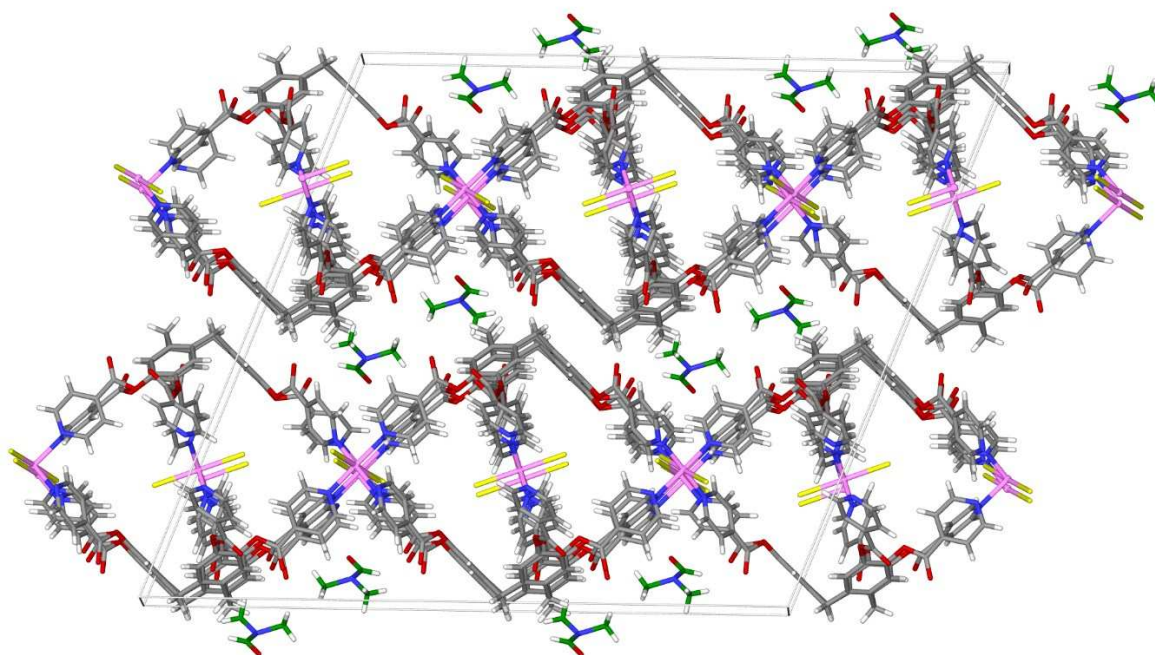


Figure S28: Asymmetric unit of **3a** with ellipsoids shown at 50% probability levels.

[Co₃Br₆(L1)₂·n(DMF) 3b



*Figure S29: Asymmetric unit of **3b** with ellipsoids shown at 50% probability levels. Isotropic atoms are solvent DMF.*



*Figure S30: Unit cell packing diagram of **3b** viewed down *b*. DMF shown in green.*

[Co₃I_{1.5}(H₂O)_{4.5}L1]₂·4.5I·m(DMF) 4

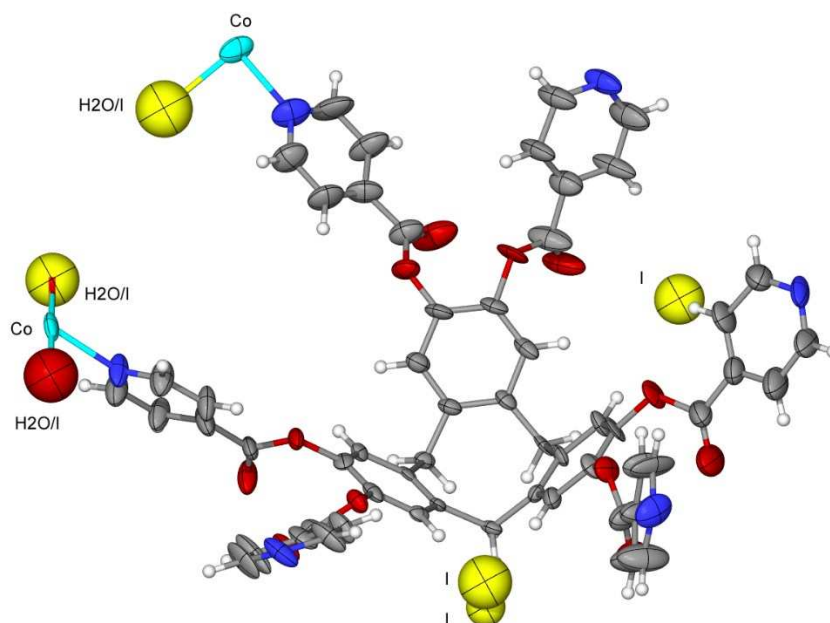


Figure S31: Asymmetric unit of 4 with ellipsoids shown at 50% probability levels. Isotropic atoms are disordered I- anions and disordered H₂O/I ligands with position refined as 25% I, 75% O.

[Ag₂(L1)(DMF)₂·2BF₄·2(H₂O)·6(DMF) 6

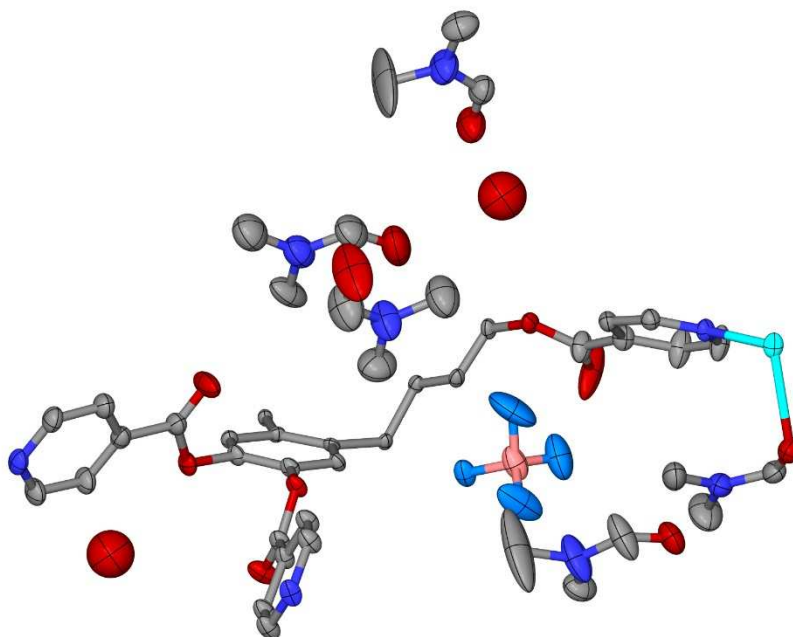


Figure S32: Asymmetric unit of 6 with ellipsoids shown at 50% probability levels. Isotropic atoms are disordered water. Hydrogen atoms excluded for clarity.

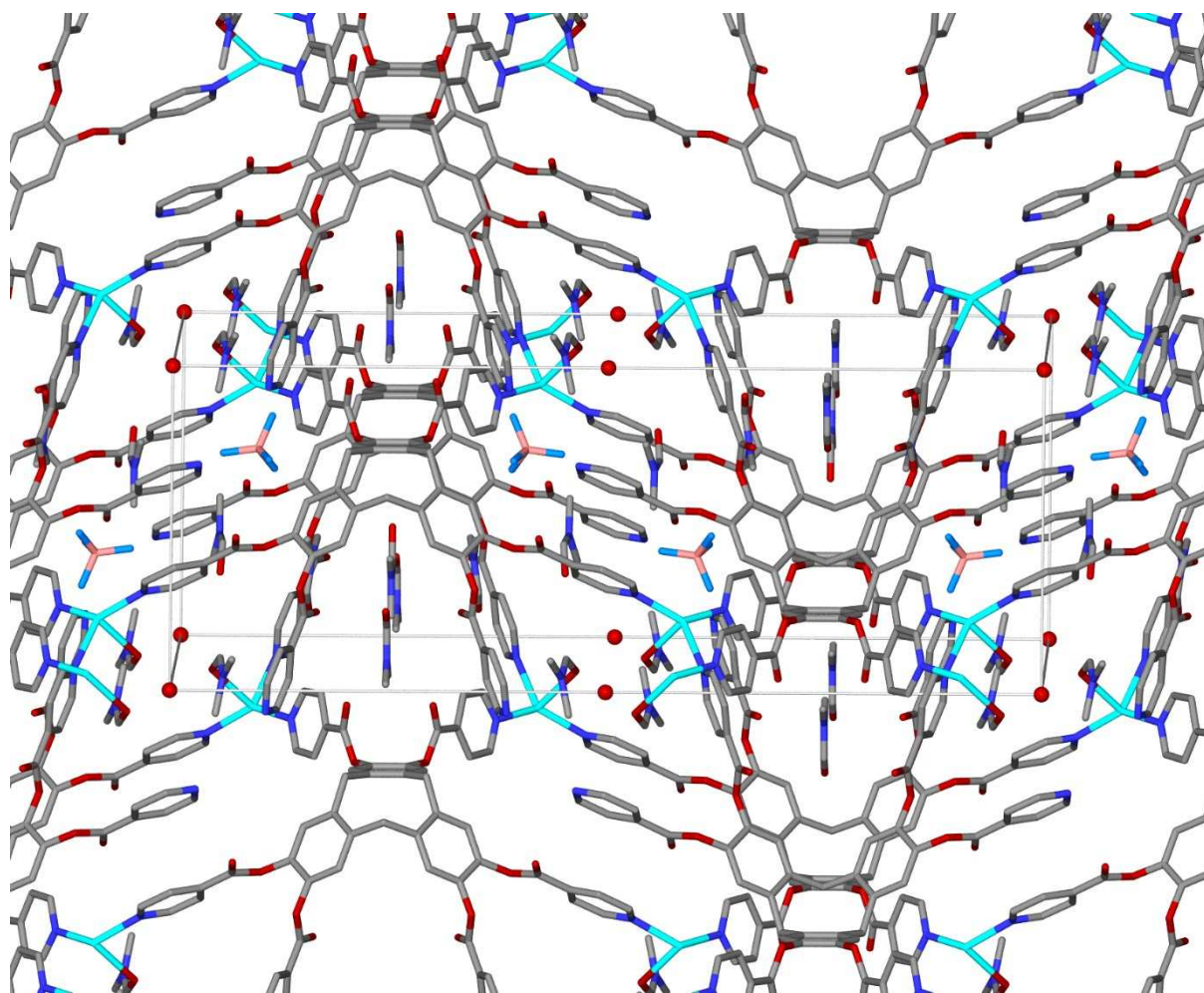


Figure S33: Packing diagram of 6 viewed down the a axis. Hydrogen atoms excluded for clarity