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Accessory Publication

Charge-assisted hydrogen-bonded linear second-sphere assemblies $[M^{II}(DABP)_3]_3[Cr(C_2O_4)_3]_2 \cdot 14H_2O$ (M = Cu, Ni, Fe, Zn and Mn) with a discrete $(H_2O)_{14}$ cluster of S_6 symmetry

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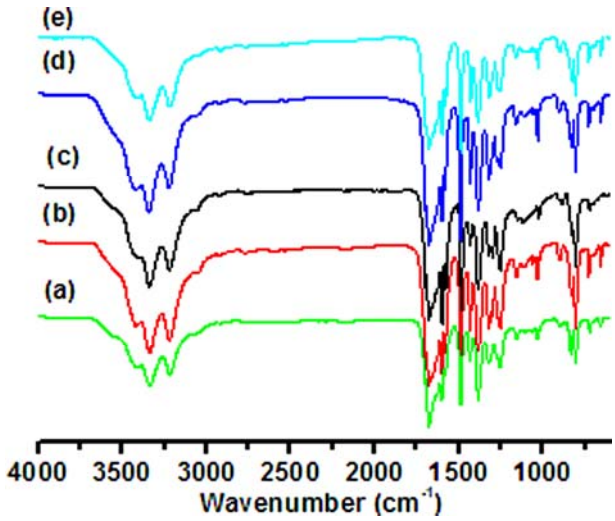

Fig. S1. FT-IR spectra of the five compounds. (a) $[Cu(DABP)_3]_3[Cr(C_2O_4)_3]_2 \cdot 14H_2O$ (**1**); (b) $[Ni(DABP)_3]_3[Cr(C_2O_4)_3]_2 \cdot 14H_2O$ (**2**); (c) $[Fe(DABP)_3]_3[Cr(C_2O_4)_3]_2 \cdot 14H_2O$ (**3**); (d) $[Zn(DABP)_3]_3[Cr(C_2O_4)_3]_2 \cdot 14H_2O$ (**4**); (e) $[Mn(DABP)_3]_3[Cr(C_2O_4)_3]_2 \cdot 14H_2O$ (**5**).

Table S1. Crystal data and refinement details for compounds **1**–**5**.

Compound	**1**	**2**	**3**	**4**	**5**

Empirical formula	$C_{102}H_{118}Cr_2Cu_3N_{36}O_{38}$	$C_{102}H_{118}Cr_2Ni_3N_{36}O_{38}$	$C_{102}H_{118}Cr_2Fe_3N_{36}O_{38}$	$C_{102}H_{118}Cr_2Zn_3N_{36}O_{38}$	$C_{102}H_{118}Cr_2Mn_3N_{36}O_{38}$
*F*w	2750.94	2736.45	2727.87	2756.43	2725.14
Crystal color	deep green	deep orange	red	brown	deep orange
Crystal system	trigonal	trigonal	trigonal	trigonal	trigonal
Space group	*R*-3*c*	*R*-3*c*	*R*-3*c*	*R*-3*c*	*R*-3*c*
a [Å]	27.469(3)	27.478(6)	27.399(5)	27.445(6)	27.544(3)
b [Å]	27.469(3)	27.478(6)	27.399(5)	27.445(6)	27.544(3)
c [Å]	27.469(3)	27.478(6)	27.399(5)	27.445(6)	27.544(3)
α [°]	34.775(7)	34.720(8)	34.374(7)	34.88(1)	34.87(2)
β [°]	34.775(7)	34.720(8)	34.374(7)	34.88(1)	34.87(2)
γ [°]	34.775(7)	34.720(8)	34.374(7)	34.88(1)	34.87(2)
V [Å^3]	6018(1)	6007(2)	5848(2)	6035(2)	6097(1)
Z	2	2	2	2	2
$D_{calcd.}$ [g cm^{-3}]	1.518	1.513	1.549	1.517	1.484
F(000)	2842	2836	2824	2848	2818
μ [mm^{-1}]	0.794	0.734	0.644	0.859	0.571
Measured reflns	32193	32018	31299	33396	31873
Unique reflns. (R_{int})	4180 (0.0756)	4154 (0.1503)	4041 (0.1648)	4478 (0.1398)	4174 (0.0760)
Observed reflns [$I > 2\sigma(I)$]	1502	1081	1114	1177	1628
Parameters/restraints	273/0	276/0	273/18	273/18	273/0
Max./min. $\Delta\rho$/e Å^{-3} [a]	0.812/–0.330	0.910/–0.420	0.888/–0.432	0.882/–0.834	1.023/–0.793
*R*1/*wR*2 [$I > 2\sigma(I)$] [b]	0.0499/0.1397	0.0525/0.1342	0.0520/0.1297	0.0623/0.1536	0.0627/0.1896
*R*1/*wR*2 (all reflections) [b]	0.1324/0.1677	0.2038/0.1722	0.2072/0.1693	0.1876/0.1857	0.1427/0.2318
GOF on F^2 [c]	0.785	0.699	0.737	0.695	0.851
Weight. scheme *w*; *a*/*b* [d]	0.1003/0.0000	0.0891/0.0000	0.0812/0.0000	0.1003/0.0000	0.1482/0.0000

[a] Largest difference peak and hole. – [b] $R_1 = [\Sigma(\| F_o \| - \| F_c \|)/\Sigma \| F_o \|]$; $wR_2 = [\Sigma [w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]]^{1/2}$. – [c] Goodness-of-fit = $[\Sigma [w(F_o^2 - F_c^2)^2] /(n - p)]^{1/2}$. – [d] $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$ where $P = (\max(F_o^2 \text{ or } 0)+2F_c^2)/3$.

Note: Despite numerous attempts the crystals of **1** and the analogous compounds (see Accessory) did not diffract very strongly as evidenced by the high R_{int} values of the data set and the large difference between the unique and observed reflections with [$I > 2\sigma(I)$]. This weak reflecting intensity may be traced to a loss in crystallinity from loss of crystal waters and to an incommensurate shift, that is by different A_x+B_y units, of the neighboring 1D $\{A_3B_2C\}_n$ chains (cf. Fig. 2b). Consequently, the high R_{int} values carry over to

large *wR*2 values and GOF values deviating from 1.

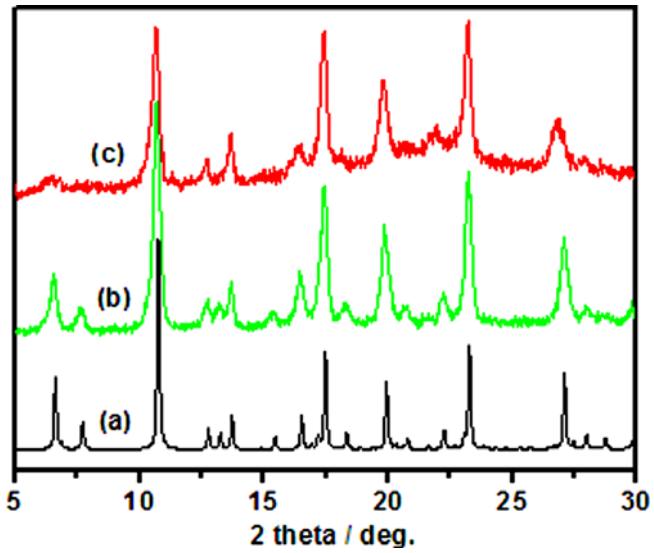

Fig. S2. Powder X-ray diffraction patterns of compound **1**. (a) Simulated from the crystallographic data of **1** obtained from single crystal X-ray diffraction. (b) Bulk crystals synthesized by a slow diffusion method. (c) Flower-like product synthesized by a precipitation method.

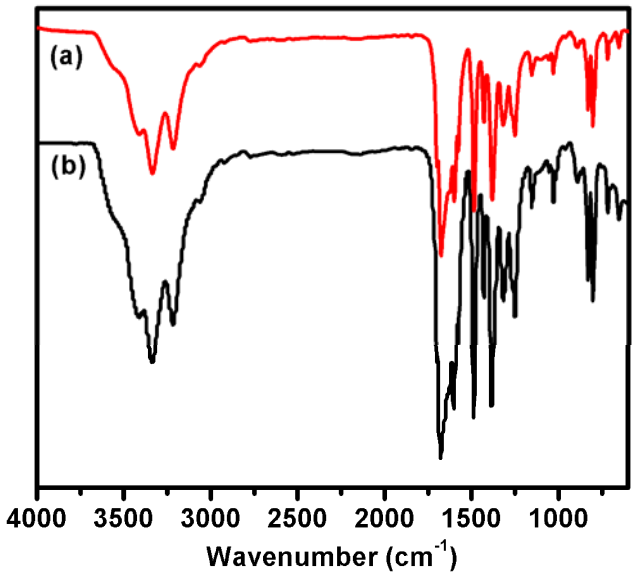

Fig. S3. FT-IR spectra of (a) Bulk crystals of **1** synthesized by a slow diffusion method; (b) Flower-like product synthesized by a precipitation method.