Supplementary Information

Order in disorder: solution and solid-state studies of $[M^{III}_2M^{II}_5]$ wheels (M^{III} = Cr, Al; M^{II} = Ni, Zn)

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Low temperature single-crystal X-ray crystallography

Low temperature single-crystal data of compound **2** were collected on XIPHOS I, a four-circle Huber goniometer equipped with an APEXII detector and rotating anode MoK_α radiation (λ = 0.71073 Å) focused using Helios optics.[1] The sample was mounted to a modified APD 202E Displex cryogenic refrigerator and cooled a rate of 1 K/minuet to a base temperature of 3.4 K. Cell indexing was carried out using the Bruker APEX III software. The low temperature sample environment allowed only unit cell parameters to be extracted at 3.4 K. On cooling the sample remains in the rhombohedral crystal system with unit cell parameters showing the expected thermal contraction from those determined at 120 K. This suggests that no large structural rearrangement occurs upon cooling. a,b = 14.522(0.001) Å, c = 36.2145(0.0029) Å, $\alpha = \beta = 90^o$, $\gamma = 120^o$, V = 6614.007(1.308) Å^3.

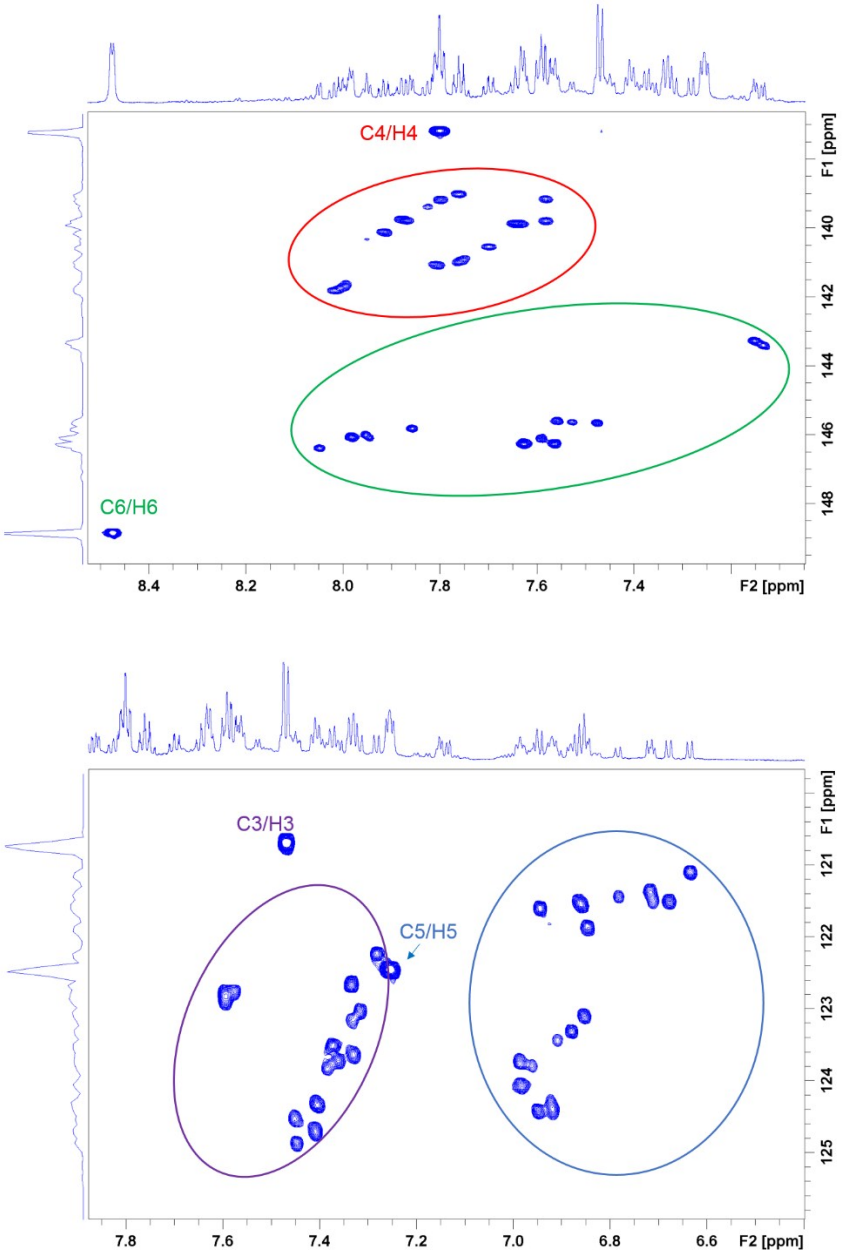

Figure S1. Partial 2D ^{1}H, ^{13}C HSQC spectrum of **4** showing (top) the C4/H4 and C6/H6 and (bottom) C3/H3 and C5/H5 cross peaks.

Table S1. Crystallographic information for compounds **1**-**4**.

Compound	**1**	**2**	**3**	**4**
Formula	$C_{79}H_{100}Cl_4Cr_2N_{12}Ni_5O_{35}$	$C_{81}H_{108}Cl_4Cr_2N_{12}O_{37}Zn_5$	$C_{81}H_{108}Al_2Cl_4N_{12}Ni_5O_{37}$	$C_{80}H_{104}Al_2Cl_4N_{12}O_{36}Zn_5$
$D_{calc.}$/ g cm^{-3}	1.674	1.727	1.674	1.679
μ/mm^{-1}	1.439	1.706	3.195	1.511
Formula Weight	2317.05	2414.44	2331.10	2332.36
Colour	blue	light purple	pale blue	colourless
Shape	block	plate	plate	plate
Size/mm^3	0.32×0.11×0.10	0.32×0.11×0.10	0.23×0.19×0.09	0.28×0.23×0.04
T/K	120.0	120.0	120.0	120.0
Crystal System	trigonal	trigonal	trigonal	trigonal
Space Group	R-3	R-3	R-3	R-3
a/Å	14.6131(3)	14.5920(3)	14.53160(10)	14.4962(5)
b/Å	14.6131(3)	14.5920(3)	14.53160(10)	14.4962(5)
c/Å	37.2857(9)	37.7623(14)	37.9260(9)	38.022(2)
α/°	90	90	90	90
β/°	90	90	90	90
γ/°	120	120	120	120
V/Å^3	6895.4(3)	6963.4(4)	6935.77(19)	6919.4(6)
Z (Z')	3 (0.16667)	3 (0.16667)	3 (0.16667)	3 (0.16667)
Wavelength/Å	0.71073	0.71073	1.54178	0.71073
Radiation type	MoK_α	MoK_α	CuK_α	MoK_α
Θ_{min}/° - Θ_{max}/°	1.638 – 25.339	2.792 – 25.350	3.700 – 76.591	2.810 – 26.372
Measured Refl.	26509	35087	32013	13554
Independent Refl.	2823	2846	3217	3142
Reflections Used	2044	2599	3044	2492
R_{int}	0.0491	0.0660	0.0502	0.0577
Parameters	186	202	186	187
Restraints	0	45	0	0
Largest Peak	0.976	1.903	0.878	1.427
Deepest Hole	-1.227	-0.593	-1.627	-0.919
GooF	1.086	1.150	1.134	1.092
wR_2 (all data) (wR_2)	0.1752 (0.1512)	0.1438 (0.1412)	0.2395 (0.2373)	0.1459 (0.1384)
R_1 (all data) (R_1)	0.0779 (0.0548)	0.0721 (0.0660)	0.0838 (0.0818)	0.0849 (0.0647)

References

1. M. R. Probert, C. M. Robertson, J. A. Coome, J. A. K. Howard, B. C. Michell and A. E. Goeta. *J. Appl. Crystallogr*., 2010, **43**, 1415-1418.