Supporting Information

Single Molecule Magnet behaviour in a $\{Dy_4P_2\}$ Octahedron

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Experimental Details

All reagents, metal salts and ligands were used as obtained from Aldrich. Analytical data were obtained by the microanalytical service of the University of Manchester, $[Co_3(\mu_3\text{-}O)(O_2C^tBu)_6(py)_3]\bullet O_2C^tBu$ was prepared as reported.[1]

Preparation of compounds 1 and 2

Compound **1** was synthesised by reacting a mixture of gadolinium nitrate $Gd(NO_3)_3\bullet 6H_2O$ (0.13 g, 0.30 mmol), $[Co_3(\mu_3\text{-}O)(O_2C^tBu)_6(py)_3]\bullet O_2C^tBu$ (1g, 0.88mmol), *tert*-butyl phosphonic acid ($H_2O_3P^tBu$) (0.07g, 0.507 mmol) and pyridine (C_5H_5N) (0.5mL, 6.20mmol) in acetonitrile (CH_3CN) (15 ml) for 7 hrs to form a brown solution. The solution was filtered and then allowed to stand untouched at room temperature for two weeks. Light brown colour crystals of the compound **1** suitable for X-ray were collected. Yield 65 mg (25%), based on $Gd(NO_3)_3\bullet 6H_2O$. EA for $C_{122}H_{202}Co_6Gd_4N_{14}O_{60}P_2$, found (calc); C 37.85 (37.87); H 5.20 (5.26); N 5.04 (5.07); Gd. 16.22 (16.25); P 1.55 (1.60); Co 9.10 (9.14).

A parallel procedure to that used for synthesis of compound **1** was used for compound **2**, using $Dy(NO_3)_3\bullet 5H_2O$ (0.30 mmol) in place of $Gd(NO_3)_3\bullet 6H_2O$. Yield 50 mg (20%), based on $Dy(NO_3)_3\bullet 5H_2O$. EA for $C_{122}H_{202}Co_6Dy_4N_{14}O_{60}P_2$, found (calc); C 37.70 (37.66); H 5.20 (5.23); N 5.01 (5.04); Dy 16.65 (16.71); P 1.57 (1.59); Co 9.06 (9.09).

Crystallography

The data were collected on Agilent SuperNova CCD diffractometer with MoK_α radiation (λ = 0.71073 Å). The structures were solved by direct methods and refined on *F2* using SHELXTL. CCDC 1051263 and 1051264 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via

[1] G. Aromi, A. S. Batsanov, P. Christian, M. Helliwell, A. Parkin, S. Parsons, A. A. Smith, G. A. Timco, R. E. P. Winpenny, *Chem. Eur. J.*, 2003, **9**, 5142.

www.ccdc.cam.ac.uk/conts/retrieving.html (or from Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB21EZ, UK; fax: (+44)1223-336-033; or deposit@ccdc.cam.ac.uk).

Crystal data for **1** [$C_{122}H_{202}Co_6Gd_4N_{14}O_{60}P_2$]: Mr = 3869.48, monoclinic, $P2_1/n$, T = 128(4) K, a = 11.7554(2), b = 19.4689(4), c = 36.4118(7) Å, α /° = 90, β/° = 97.9131(17), γ/° = 90, V = 8254.0(3) Å^3, Z = 2, ρ = 1.557 g cm^{-3}, total data = 64303, independent reflections 16879 (R_{int} = 0.0631), μ = 2.274 mm^{-1}, 1053 parameters, R_1 = 0.0479 for $I \geq 2\sigma$ (I) and wR_2 = 0.0687 CCDC 1051264.

Crystal data for **2** [$C_{106}H_{196}Co_6Gd_4N_{12}O_{60}P_2$]: Mr = 3890.48, monoclinic, $P2_1/n$, T = 150.02(10) K, a = 11.7542(2), b = 19.4585(4), c = 36.4590(7) Å, α /° = 90, β/° = 97.9842(19), γ/° = 90, V = 8258.0(3) Å^3, Z = 2, ρ = 1.565 g cm^{-3}, total data = 41706, independent reflections 18920 (R_{int} = 0.0750), μ = 2.476 mm^{-1}, 1059 parameters, R_1 = 0.0448 for $I \geq 2\sigma$ (I) and wR_2 = 0.0900 CCDC 1051263.

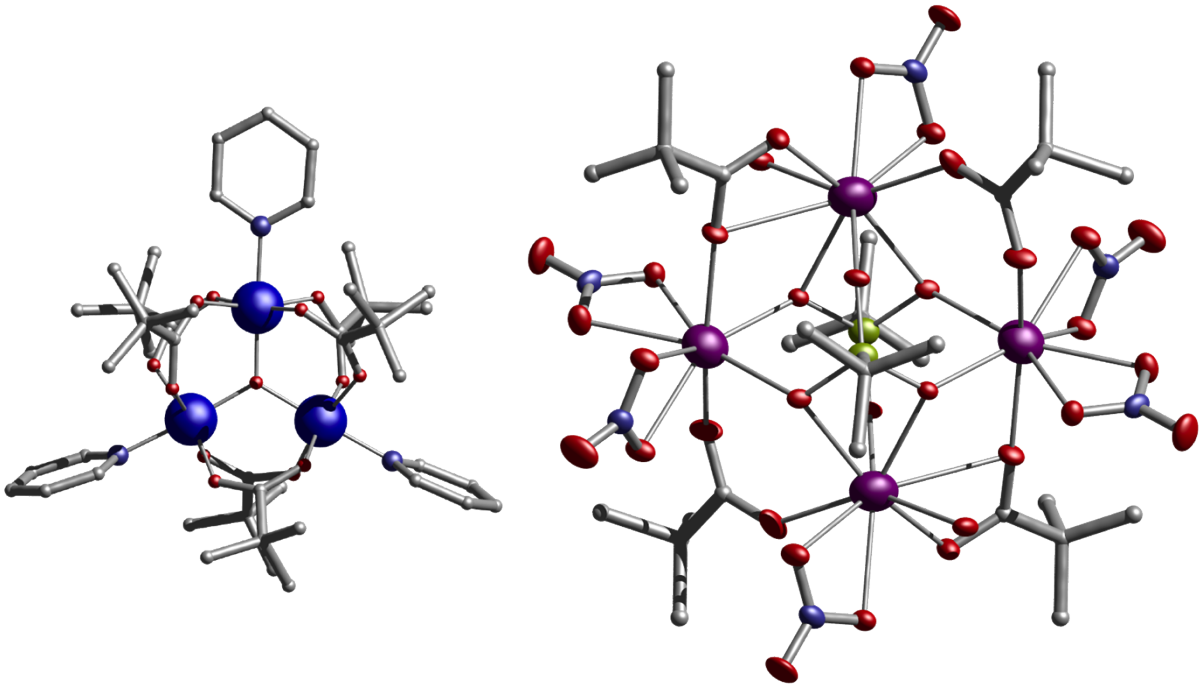

Figure S1. Crystal structure of **2**. Colour codes: Dy, purple; P, green; Co, blue; N, cyan; O, red; C, grey. H-atoms are omitted for clarity.

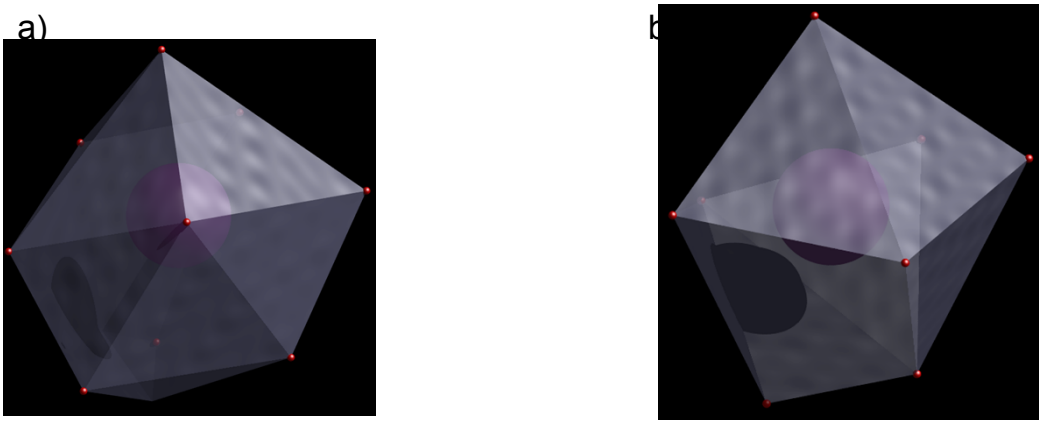

Figure S2. (a) and (b) are the polyhedral view of Dy1 and Dy2, respectively.

Table S1. CShM values for the Dy metal ion centres of **2**.

Dy1		**Dy2**	
Geometry	CShM	Geometry	CShM
OP	31.623	**EP**	30.136
HPY	23.144	**OPY**	22.402
HBPY	13.442	**HBPY**	15.576
CU	12.503	**JTC**	14.218
SAPR	4.705	**JCCU**	8.029
TDD	2.745	**CCU**	6.926
JGBF	11.410	**JCSAPR**	5.858
JETBPY	26.724	**CSAPR**	4.840
JBTPR	4.333	**JTCTRP**	4.777
BTPR	3.905	**TCTPR**	5.830
JSD	3.575	**JTDIC**	10.421
TT	13.256	**HH**	5.955
ETBPY	24.075	**MFF**	3.265

Magnetic measurements

The magnetic properties in the temperature range 1.8 K–300 K were performed on polycrystalline samples either constrained in eicosane or powdered, using a Quantum Design MPMS-XL7 SQUID magnetometer armed with a 7 T magnet. Data were corrected for the diamagnetism of the compounds (Pascal constants) and for diamagnetic contribution of eicosane and the sample holder by measurement.

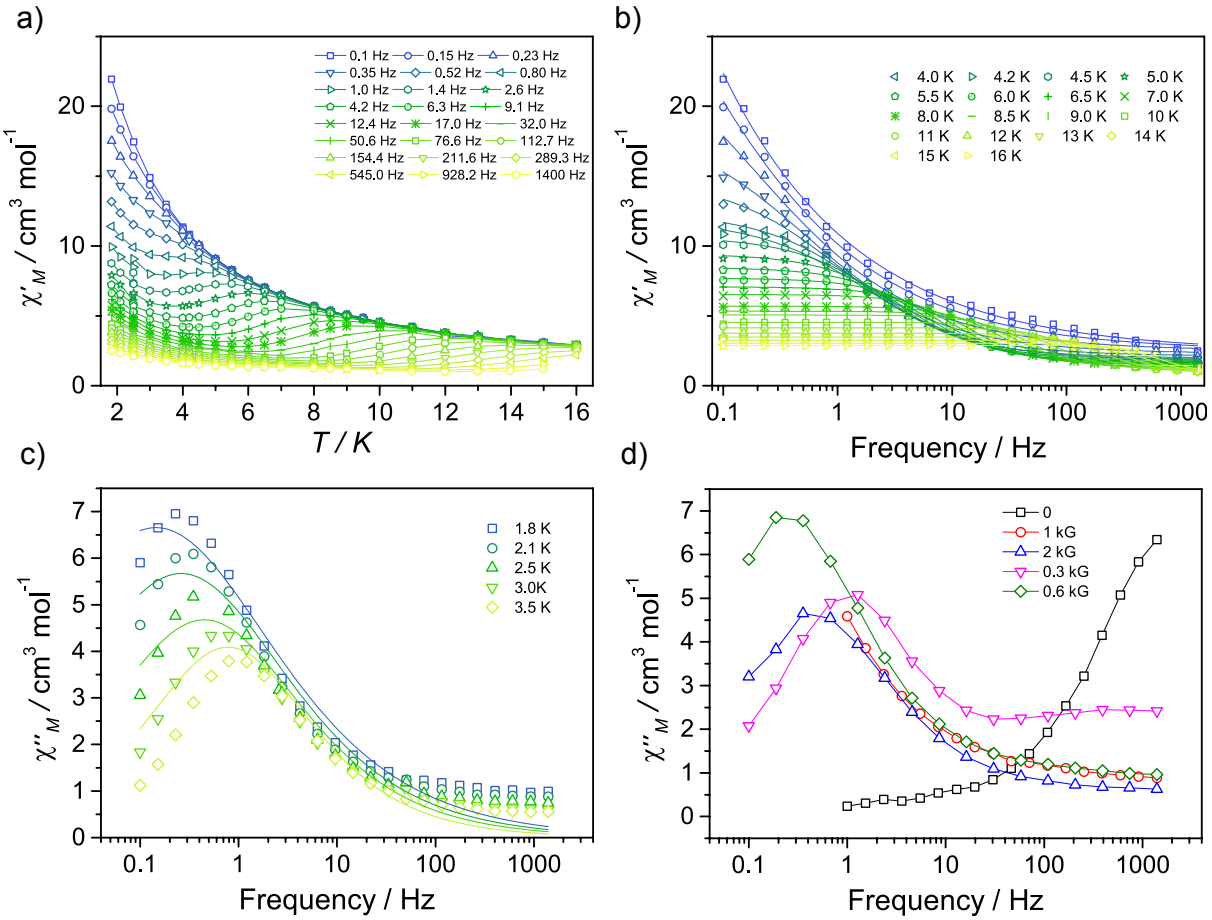

Figure S3. (a) χ_M'(T); (b) χ_M'(ν); (c) χ_M''(ν); (d) and field dependence of the χ_M''(ν) for compound **2**.