Electronic Supplementary Material (ESI) for Dalton Transactions.
This journal is © The Royal Society of Chemistry 2017

Supporting Information

Lantern-Shaped 3d-4f High-Nuclearity Clusters with Magnetocaloric Effect

*Qingfang Lin,[a,c] JingLi[b], YayuDong[a], Guangpeng Zhou[a], You Song[b] and Yan Xu[a]**

[a]College of Chemistry and Chemical Engineering, State Key Laboratory of Materials-Oriented Chemical Engineering, Nanjing Tech University, Nanjing 210009, P. R. China

[b]State Key Laboratory of Coordination Chemistry, School of Chemistry and Chemical Engineering, Nanjing University, Nanjing 210023, P.R. China

[c]Department of of Chemistry，Bengbu Medical College，Bengbu 233030，PR China

Experimental Section

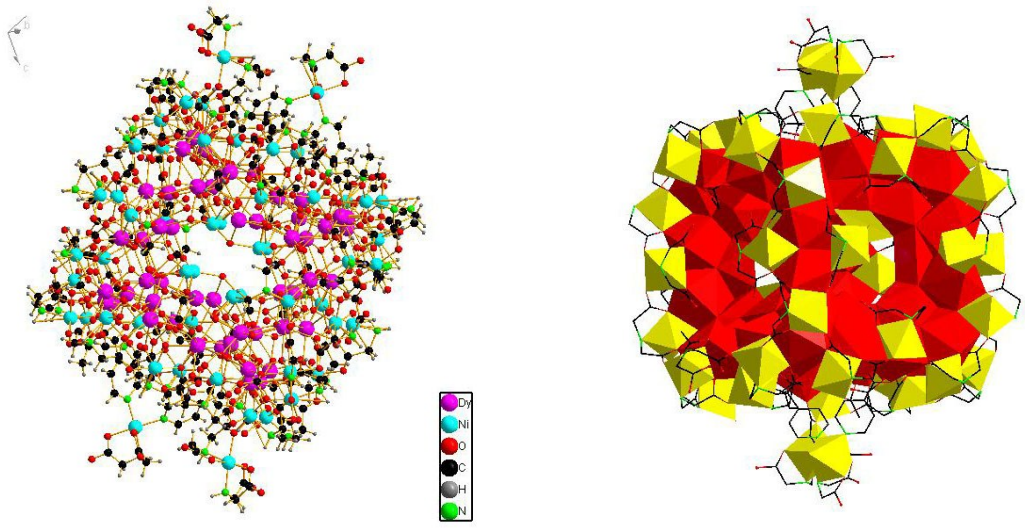
Synthesis of 1: A mixture of Gd_2O_3 (0.1812 g, 0.50 mmol), $Ni(OAc)_2{\cdot}4H_2O$ (0.1234 g,0.50 mmol), IDA (0.0665 g, 0.50 mmol), HIN (0.060 g, 0.50 mmol) in the mixture of distilled water (5 mL) and EtOH (10 mL) was stirred for 12 h at room temperature. The pH of resulting solution was adjusted to 5.0 by the addition of 1.0 M NaOH

aqueous solution. This solution was then sealed in a 25 mL Teflon-lined stainless steel autoclave and heated under autogenous pressure at 180 °C for 8 days. After cooling to room temperature, blue square slice crystals were filtered off, washed with ethanol, and dried at room temperature for 24 h (Yield 40% based on Gd). Calcd.(%) C: 13.60; H: 2.25; N: 3.45; Found (%): C: 13.56; H: 2.08; N: 3.34.

Synthesis of 2: Compound **2** was prepared in a procedure similar to that of **1**, except that Dy_2O_3(0.1865g, 0.50 mmol) was utilized instead of Gd_2O_3, blue square slice crystals were obtained. (Yield 46% based on Dy). Calcd.(%) : C:12.82; H: 2.28; N: 3.36, Found (%): C: 12.75; H: 2.08; N: 3.45.

Crystal data of **1 :** C_{248} H_{493} Gd_{52} N_{54} Ni_{52} $O_{401.5}$, *Mr*=21885.86, Monoclinic, $P2_1/n$, *a*=28.737(15) Å, *b*=41.67(2) Å, *c*=29.877(15) Å, *V*=34104(30) $Å^3$, Z=2, μ = 6.440 mm^{-1},ρ =2.129 $Mg{\cdot}m^{-3}$, *F* (000)= 20670, θ_{max}=25.005°.162404 measured reflections, 59047 independent reflections. Based on these and 1902 parameters, R_1=0.0857 (I>2σ(I)), wR_2=0.2682 (all data) were obtained. GOF=1.068.

Crystal data of **2**: C_{236} H_{503} Dy_{52} N_{53} Ni_{52} O_{407}, *M*r=22098.8, Monoclinic, $P2_1/n$, *a*=28.620(3), *b*=41.352 (4), *c*=29.564(3) Å, *V*=33259(6) $Å^3$, Z=2, μ = 7.287 mm^{-1},ρ =2.206 $Mg.m^{-3}$, *F* (000)= 20864, θ_{max}=25.005°.236957 measured reflections, 58585 independent reflections. Based on these and 1904 parameters, R_1=0.0737 (I>2σ(I)), wR_2=0.2421 (all data) were obtained. GOF=1.081. Crystal size (mm): 0.16 x 0.13 x 0.12. CCDC **1551098** and **1551099** contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre. The direct current magnetic data were measured at temperature between 1.8 and 300 K, and the magnetisation isothermal measurements were made in fields of between 0 and 7 T on MPMS-XL7 SQUID magnetometer. Experimental susceptibilities were corrected for the diamagnetism estimated Pascal's tables and for sample holder by previous calibration.



(a) (b)

Figure S1. (a) Ball and stick plot show the shell structure of compound **2** (b) The polyhedron structure of {$Dy_{52}Ni_{52}$}, Dy-O polyhedron(red),Ni-O(N) polyhedron(yellow)

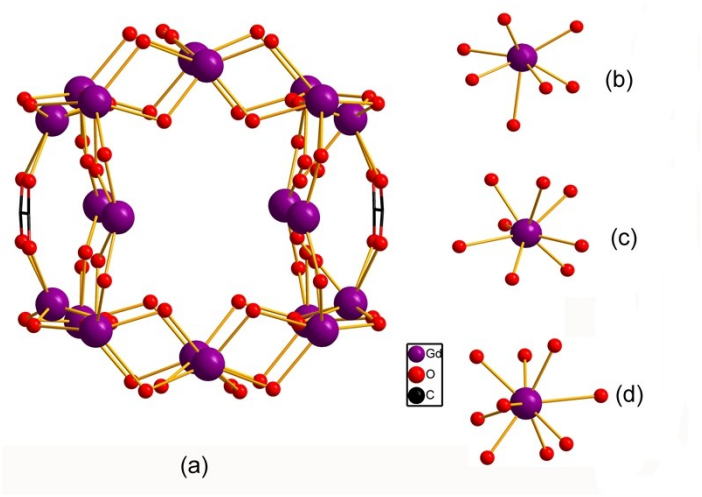


Figure S2. (a) View of 20 Gd^{III} ions in shell 2; (b) The coordination modes of Gd(heptacoordinate) ; (c) The coordination modes of Gd (octacoordinate) (d) The coordination modes of Gd (nonacoordinate).

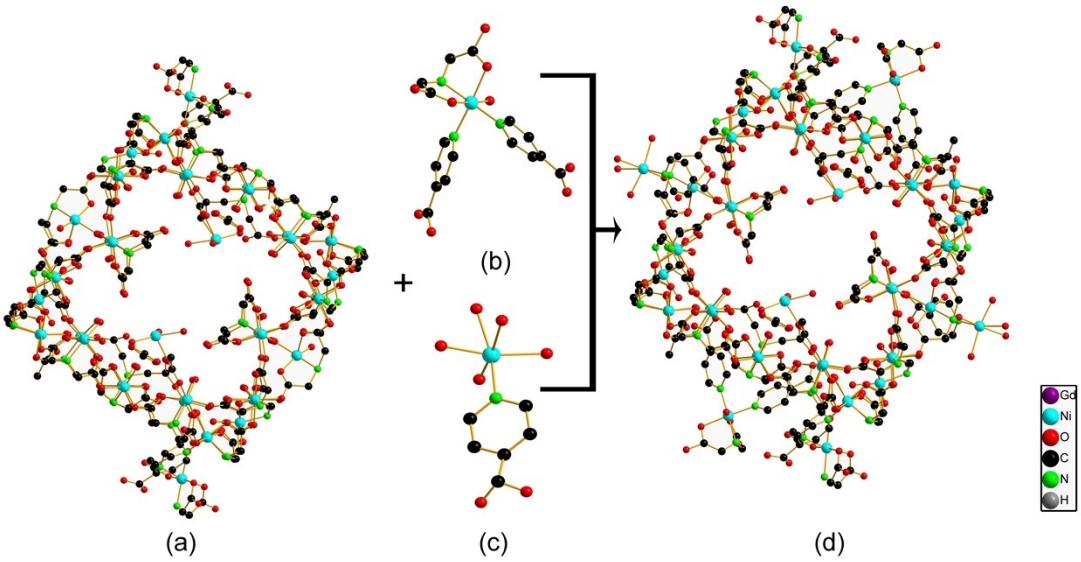


Figure S3. (a) Ball-and-stick view of the outermost shell $\{Ni_{41}\}$; (b) Ball-and-stick view of the $[Ni(IDA)(IN)_2]^{2-}$ unit; (c) Ball-and-stick view of the $[Ni(IN)(H_2O)_5]^{+}$unit;(d) Ball-and-stick view of the outermost shell $\{Ni_{44}\}$

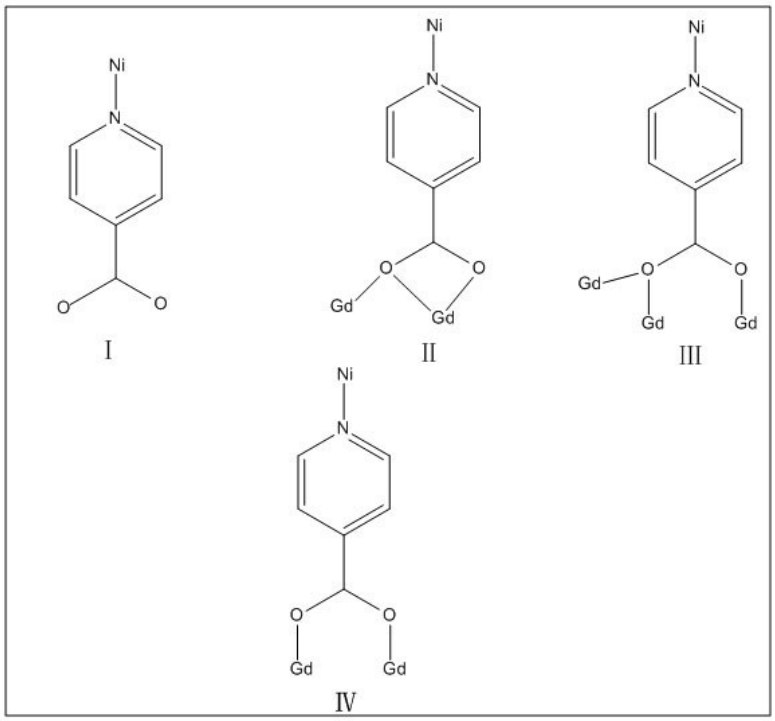


Figure S4. The coordination modes of HIN ligand in **1**

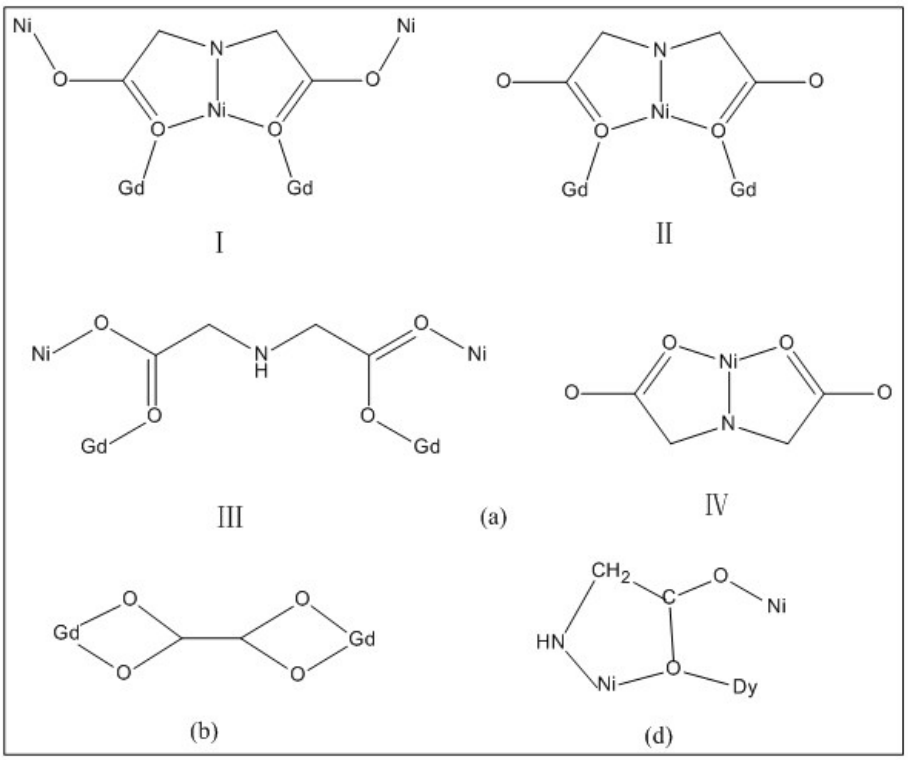


Figure S5. The coordination modes of IDA(a) and OX(b) ligands in **1** and glycine **in 2**

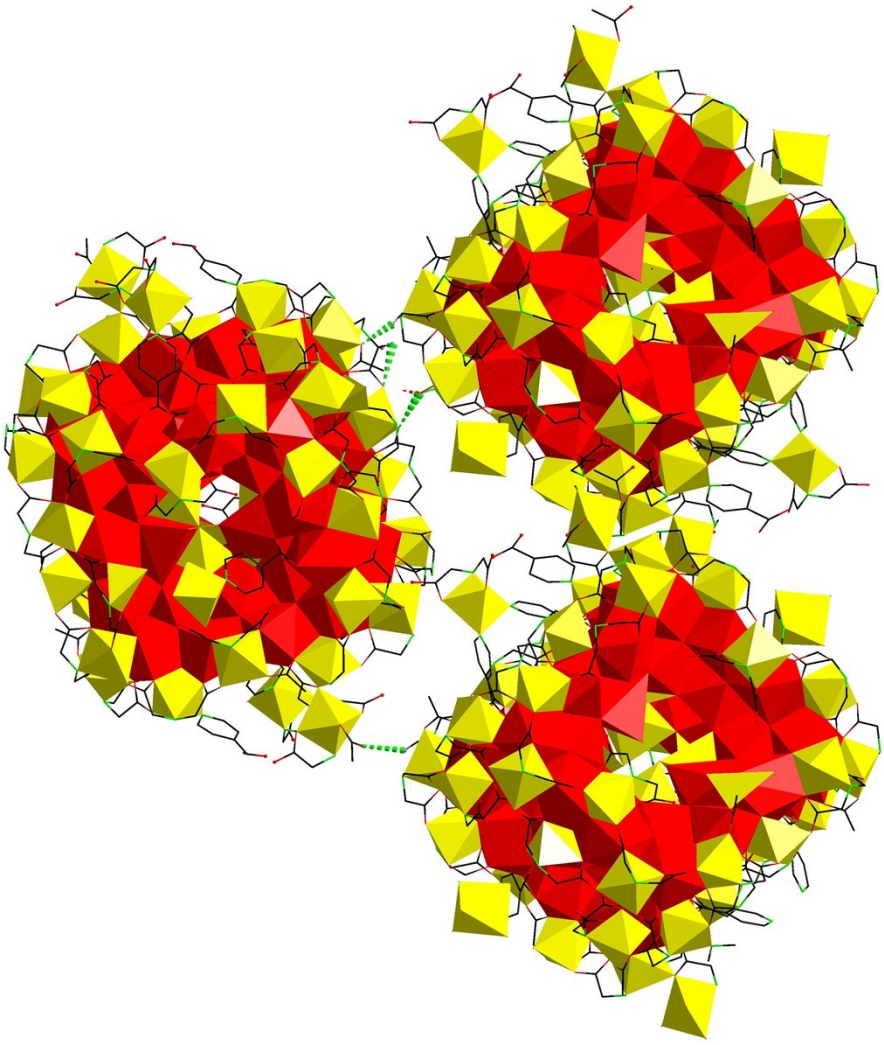
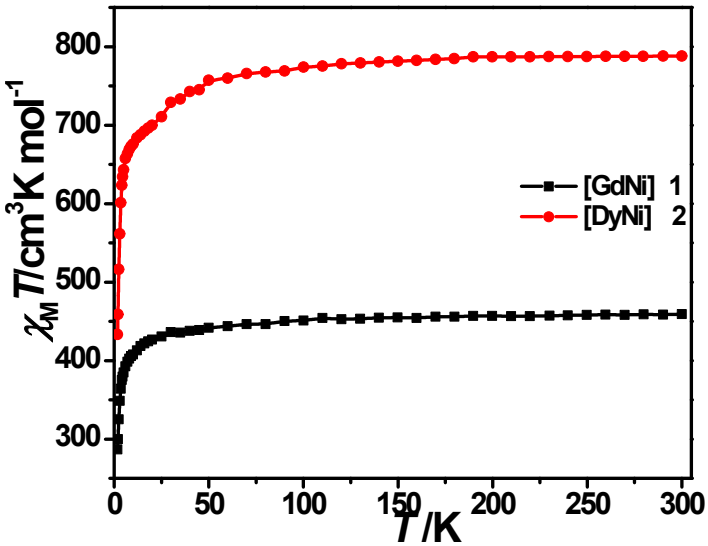
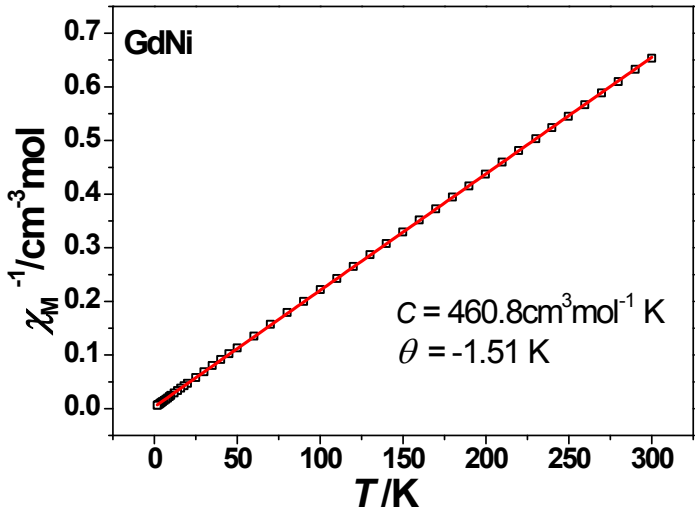


Figure S6. The 3D stacking structure of **1** that formed by packing of adjacent cluster units through strong hydrogen-bonding interactions.



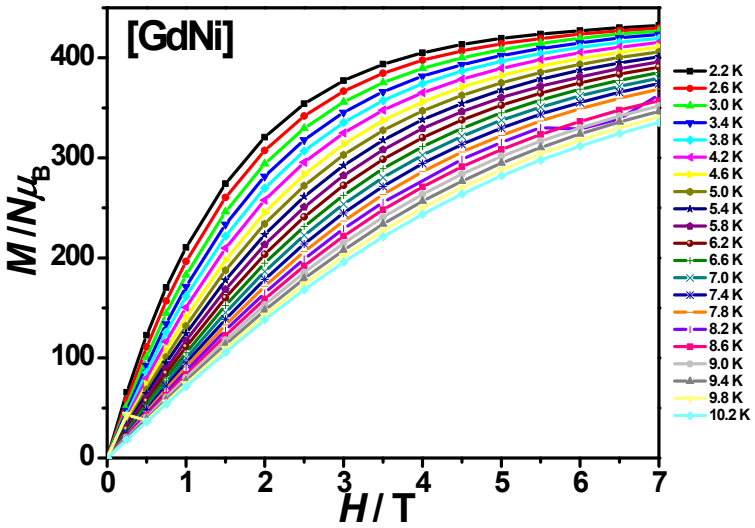
(a)



(b)

Figure S7. (a) Magnetic susceptibility versus temperature from 1.8 to 300 K under 2.0 kOe dc field for **1** and **2**.

(b) χ_M^{-1} versus T plot of complex **1**. The red line is fitting result with $\chi_M = C/(T-\theta)$.



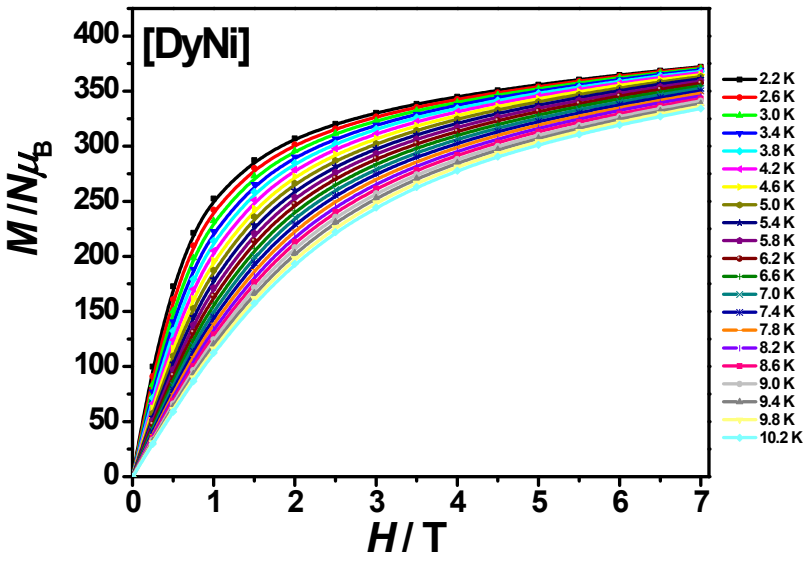


Figure S8. The field-dependent magnetization of **1** and **2** at temperature range 2.2 – 10.2 K.

Table S1 The Crystallographic data of compound **1**.

Compound	**1**
Empirical formula	C248 H473 Gd52 N54 Ni52 O401.50
Formula weight	21865.70
Crystal system	Monoclinic
Space group	$P2_1$/n
a (Å)	28.737(15)
b (Å)	41.67(2)
c (Å)	29.877(15)
V (Å)	34104(30)
Z	2
D_c (Mg·m^{-3})	2.129
μ (mm^{-1})	6.466
F (000)	20670
Crystal size (mm)	0.160 x 0.130 x 0.120
θ Range	0.86-25.005
Reflections collected	162404
Independent reflections	59047 [R_{int} = 0.2260]
Reflections observed [I>2σ(I)]	32207
Data/restraints/parameters	59047/92/1902
Goodness-of-fit on F^2	1.068
R/wR [I>2σ(I)]	0.0857/0.2312
R/wR (all data)	0.1625/0.2682
Max., Min. $\Delta\rho$ (e·Å^3)	6.633, -3.067

Table S2 Selected bond distances (Å) for compound **1**

Bond lengths

Gd(1)-O(10)	2.418(12)	Gd(5)-O(61)	2.348(14)
Gd(1)-O(5)	2.416(13)	Gd(5)-O(160)	2.389(18)
Gd(1)-O(16)	2.430(14)	Gd(5)-O(127)	2.400(15)
Gd(1)-O(21)	2.463(13)	Gd(5)-O(171)	2.43(3)
Gd(1)-O(33)	2.472(14)	Gd(5)-O(60)	2.451(15)
Gd(1)-O(38)	2.474(14)	Gd(5)-O(71)	2.463(15)
Gd(1)-O(47)	2.478(14)	Gd(5)-O(56)	2.475(15)
Gd(1)-O(17)	2.485(13)	Gd(5)-O(95)	2.548(16)
Gd(1)-O(11)	2.496(14)	Gd(5)-O(177)	2.70(4)
Gd(1)-Gd(13)	3.464(2)	Gd(6)-O(50)	2.346(13)
Gd(3)-Gd(19)	3.801(2)	Gd(6)-O(30)	2.407(13)
Gd(4)-O(48)	2.347(13)	Gd(6)-O(26)	2.441(14)
Gd(4)-O(64)	2.362(14)	Gd(6)-O(130)	2.449(15)
Gd(4)-O(29)	2.406(14)	Gd(6)-O(53)	2.455(13)
Gd(4)-O(173)	2.451(17)	Gd(6)-O(96)	2.510(15)
Gd(8)-O(110)	2.323(17)	Gd(11)-O(57)	2.372(14)
Gd(8)-O(10)	2.349(12)	Gd(11)-O(64)	2.388(13)
Gd(8)-O(58)	2.403(14)	Gd(11)-O(19)	2.420(13)
Gd(8)-O(9)	2.425(14)	Gd(11)-O(161)	2.422(15)
Gd(8)-O(72)	2.441(13)	Gd(11)-O(8)	2.445(16)
Gd(8)-O(84)	2.452(15)	Gd(11)-O(158)	2.58(3)
Gd(8)-O(17)	2.459(13)	Gd(11)-O(156)	2.64(2)
Gd(8)-O(25)	2.547(15)	Gd(11)-O(131)	2.782(18)
Gd(10)-O(39)	2.344(13)	Gd(11)-O(19W)	2.86(4)
Gd(10)-O(132)	2.370(18)	Gd(11)-O(57)	2.372(14)
Gd(10)-O(19)	2.380(13)	Gd(11)-O(64)	2.388(13)
Gd(10)-O(57)	2.415(14)	Gd(12)-O(15)#1	2.365(14)

Table S3 Selected bond angles(°)for compound **1**

O(10)-Gd(1)-O(5)	73.0(4)	O(16)#1-Gd(18)-O(54)	75.7(5)
O(10)-Gd(1)-O(16)	74.7(4)	O(30)-Gd(18)-O(54)	68.5(4)
O(5)-Gd(1)-O(16)	73.7(4)	O(81)#1-Gd(18)-O(54)	125.5(5)
O(10)-Gd(1)-O(21)	132.7(4)	O(53)-Gd(18)-O(54)	69.9(5)
O(5)-Gd(1)-O(21)	69.9(4)	O(144)-Gd(18)-O(54)	131.0(5)
O(16)-Gd(1)-O(21)	67.6(4)	O(21)#1-Gd(18)-O(54)	119.8(4)
O(10)-Gd(1)-O(33)	142.6(4)	O(63)#1-Gd(18)-O(54)	68.5(5)
O(5)-Gd(1)-O(33)	137.6(4)	O(16)#1-Gd(18)-O(159)	133.2(6)
O(28)-Ni(4)-O(88)	97.9(6)	O(67)-Gd(23)-O(15W)	144.8(8)
O(28)-Ni(4)-O(55)	87.0(6)	O(37)-Gd(23)-O(15W)	73.9(8)

O(88)-Ni(4)-O(55)	90.2(6)	O(35)-Gd(23)-O(15W)	118.1(8)
O(28)-Ni(4)-O(172)	94.1(6)	O(89)#1-Gd(23)-O(15W)	132.1(8)
O(88)-Ni(4)-O(172)	88.3(6)	O(129)-Gd(23)-O(15W)	99.5(8)
O(55)-Ni(4)-O(172)	178.3(6)	O(13W)-Gd(23)-O(15W)	51.7(11)
O(28)-Ni(4)-O(40)	84.1(5)	O(67)-Gd(23)-O(4)	65.5(6)
O(88)-Ni(4)-O(40)	178.0(6)	O(37)-Gd(23)-O(4)	66.5(6)
O(55)-Ni(4)-O(40)	89.9(6)	O(94)-Ni(1)-O(174)#1	92.5(6)
O(16)-Gd(1)-O(33)	92.3(3)	O(14)-Ni(1)-O(68)	95.2(5)
O(21)-Gd(1)-O(33)	67.6(3)	O(94)-Ni(1)-O(68)	178.4(6)
O(38)-Gd(1)-O(33)	133.0(3)	O(174)#1-Ni(1)-O(68)	86.6(6)
O(47)-Gd(1)-O(33)	72.2(3)	O(14)-Ni(1)-O(32)	84.5(6)
O(5)-Gd(1)-O(17)	134.0(3)	O(94)-Ni(1)-O(32)	87.7(6)
O(8)-Gd(2)-O(62)	80.1(3)	O(174)#1-Ni(1)-O(32)	175.5(6)
O(39)-Gd(2)-O(62)	68.4(3)	O(68)-Ni(1)-O(32)	93.2(6)
O(12)-Gd(2)-O(62)	66.8(3)	O(174)#1-Ni(1)-N(5)	92.6(7)
O(13)-Gd(2)-O(62)	130.2(3)	O(68)-Ni(1)-N(5)	101.0(6)
O(28)-Ni(4)-O(88)	97.9(6)	O(32)-Ni(1)-N(5)	83.0(6)
O(28)-Ni(4)-O(55)	87.0(6)	O(38)-Ni(2)-O(45)	97.1(6)
O(88)-Ni(4)-O(55)	90.2(6)	O(38)-Ni(2)-O(46)	97.5(6)
O(28)-Ni(4)-O(172)	94.1(6)	O(45)-Ni(2)-O(46)	90.3(6)
O(88)-Ni(4)-O(172)	88.3(6)	O(38)-Ni(2)-O(76)	86.4(6)
O(55)-Ni(4)-O(172)	178.3(6)	O(45)-Ni(2)-O(76)	175.9(6)
O(28)-Ni(4)-O(40)	84.1(5)	O(46)-Ni(2)-O(76)	91.4(6)
O(88)-Ni(4)-O(40)	178.0(6)	O(38)-Ni(2)-O(47)	84.1(6)
O(55)-Ni(4)-O(40)	89.9(6)	O(45)-Ni(2)-O(47)	91.5(6)
O(22)-Gd(26)-O(20W)	139.7(11)	O(46)-Ni(2)-O(47)	177.5(6)
O(98)-Gd(26)-O(20W)	117.1(11)	O(76)-Ni(2)-O(47)	86.7(5)
O(62)-Gd(26)-O(20W)	70.1(10)	O(38)-Ni(2)-N(1)	161.2(6)
O(165)-Gd(26)-O(20W)	101.1(11)	O(84)-Ni(3)-O(85)	90.7(6)
O(163)-Gd(26)-O(20W)	138.1(11)	O(9)-Ni(3)-O(111)#1	95.4(6)
O(22)-Gd(26)-O(10W)	149.0(8)	O(84)-Ni(3)-O(111)#1	176.8(6)
O(98)-Gd(26)-O(10W)	114.3(8)	O(85)-Ni(3)-O(111)#1	91.3(6)
O(22)-Gd(26)-O(148)	64.0(5)	O(9)-Ni(3)-N(15)	164.0(7)
O(98)-Gd(26)-O(148)	65.4(6)	O(84)-Ni(3)-N(15)	82.3(7)
O(62)-Gd(26)-O(148)	65.6(6)	O(85)-Ni(3)-N(15)	82.8(7)
O(165)-Gd(26)-O(148)	128.4(6)	O(111)#1-Ni(3)-N(15)	95.5(7)
O(163)-Gd(26)-O(148)	129.6(6)	O(9)-Ni(3)-O(83)	97.6(6)
O(20W)-Gd(26)-O(148)	85.5(11)	O(84)-Ni(3)-O(83)	88.8(6)

O(10W)-Gd(26)-O(148)	147.0(8)	O(79)-Ni(6)-O(26)	97.8(6)
O(46)-Ni(2)-O(76)	91.4(6)	O(79)-Ni(6)-O(96)	177.1(7)
O(38)-Ni(2)-O(47)	84.1(6)	O(26)-Ni(6)-O(96)	83.5(6)
O(45)-Ni(2)-O(47)	91.5(6)	O(79)-Ni(6)-O(44)	93.2(6)
O(46)-Ni(2)-O(47)	177.5(6)	O(26)-Ni(6)-O(44)	85.6(6)
O(76)-Ni(2)-O(47)	86.7(5)	O(96)-Ni(6)-O(44)	89.5(6)
O(38)-Ni(2)-N(1)	161.2(6)	O(79)-Ni(6)-N(8)	95.7(7)

Table S4 Hydrogen bond interactions in compound **1**

D—H···A	d(D···H) /Å	d(H···A)/ Å	d(D···A)/Å	D—H···A (°)
C3-H3A··O12W	0.97	2.58	3.53(3)	165
C13-H13B··O9W	0.97	2.55	3.23(3)	127
C10A-H10A··O132	0.93	2.56	2.88(3)	100
C13A-H13A··O78	0.93	2.43	3.04(5)	123
C14A-H14A··O145	0.93	2.56	2.87(6)	100
C17A-H17A··O4W	0.93	2.28	3.11(4)	148
C29-H29B O138	0.97	2.51	3.32(3)	141
C43-H43A O69	0.97	2.52	3.14(3)	122
C49-H49A··O133	0.97	2.43	3.36(2)	161
C55-H55B O27W	0.97	2.57	3.24(6)	126
C75-H75A O46	0.97	2.59	3.54(4)	167
N1-H1C··O138	0.98	1.99	2.86(3)	147
N4-H4A··O9W	0.98	2.44	3.22(5)	136
N5-H5C··O43	0.98	2.130	3.01(2)	148
N6-H6A··O32W	0.98	1.96	2.93(4)	167
N10-H10B··O136	0.98	1.89	3.77(3)	149
N11-H11D··O69	0.98	2.10	2.89(2)	136
N12-H12A··O34W	0.98	1.87	2.84(6)	166
N14-H14C··O174	0.98	2.24	3.14(2)	153
N15-H15A··O21W	0.98	2.51	3.29(5)	137
N16-H16E··O11W	0.98	2.40	3.16(5)	134
N18-H18C··O145	0.98	2.35	3.30(5)	163

N20-H20J··O33W	0.98	2.32	3.29(6)	169

Table S5 The Crystallographic data of compound **2**.

Compound	**2**
Empirical formula	C236 H501 Dy52 N53 Ni52 O407
Formula weight	22096.79
Crystal system	Monoclinic
Space group	$P2_1/n$
a (Å)	28.620(3)
b (Å)	41.352(4)
c (Å)	29.564(3)
V (Å)	33259(6)
Z	2
D_c (g·cm^{-3})	2.206
μ (mm^{-1})	7.287
F (000)	20864
Crystal size (mm)	1.120 x 0.150 x 0.130
θ Range	0.865-25.005
Reflections collected	237024
Independent reflections	58601 [R_{int} = 0.2260]
Reflections observed [I>2σ(I)]	36895
Data/restraints/parameters	58601/80/1904
Goodness-of-fit on F^2	1.081
R/wR [I>2σ(I)]	0. 0737/0.2128
R/wR (all data)	0.1259/0.2421
Max., Min. $\Delta\rho$ (e·Å^3)	4.484, -2.414

Table S6 Selected bond distances (Å) for compound **2**

Bond distances			
Dy(1)-O(146)	2.387(11)	Ni(1)-O(143)	2.002(11)
Dy(1)-O(139)	2.396(11)	Ni(1)-O(171)	2.014(15)
Dy(1)-O(154)	2.407(11)	Ni(1)-O(18)	2.024(12)
Dy(1)-O(12)	2.438(11)	Ni(1)-O(21)	2.038(12)
Dy(1)-O(134)	2.444(11)	Ni(1)-O(10)	2.042(11)
Dy(1)-O(46)	2.445(12)	Ni(1)-N(12)	2.089(13)
Dy(1)-O(149)	2.447(11)	Ni(2)-O(136)	2.014(11)
Dy(1)-O(26)	2.454(12)	Ni(2)-O(41)#1	2.018(11)
Dy(1)-O(135)	2.490(11)	Ni(2)-O(51)#1	2.026(12)
Dy(3)-O(9)	2.322(11)	Ni(2)-O(13)#1	2.049(11)
Dy(3)-O(67)	2.330(16)	Ni(4)-O(37)	2.035(14)
Dy(3)-O(155)	2.367(12)	Ni(4)-O(16)	2.040(12)

Dy(3)-O(141)	2.383(11)	Ni(4)-O(145)	2.045(12)
Dy(3)-O(167)	2.395(12)	Ni(4)-O(15)	2.069(12)
Dy(3)-O(138)	2.403(11)	Ni(4)-N(21)	2.069(15)
Dy(3)-O(23)	2.407(12)	Ni(4)-O(38)	2.070(12)
Dy(3)-O(17)	2.478(12)	Ni(5)-O(138)	2.020(11)
Dy(14)-O(174)	2.31(2)	Ni(5)-O(17)	2.044(13)
Dy(14)-O(160)	2.314(12)	Ni(5)-O(14)	2.049(12)
Dy(14)-O(165)	2.350(12)	Ni(5)-O(70)	2.055(14)
Dy(14)-O(152)	2.415(12)	Ni(6)-O(135)	2.022(12)
Dy(14)-O(166)	2.430(11)	Ni(6)-O(31)	2.024(13)
Dy(14)-O(58)	2.453(12)	Ni(6)-O(36)	2.048(13)
Dy(14)-O(44)	2.467(14)	Ni(6)-O(46)	2.054(12)
Dy(14)-O(2)	2.51(2)	Ni(6)-O(102)	2.102(17)
Dy(15)-O(151)	2.322(12)	Ni(6)-N(1)	2.103(16)
Dy(11)-O(177)#1	2.72(2)	Ni(7)-O(20)	2.045(12)
Dy(13)-O(100)	2.301(9)	Ni(7)-O(12)	2.076(12)
Dy(13)-O(157)	2.359(8)	Ni(7)-O(28)	2.090(12)
Dy(13)-O(81)#1	2.362(11)	Ni(7)-N(4)	2.105(14)
Dy(13)-O(90)	2.367(10)	Ni(8)-O(140)	2.024(11)
Dy(13)-O(160)	2.382(9)	Ni(8)-O(66)	2.024(13)
Dy(13)-O(151)	2.383(8)	Ni(8)-O(53)	2.033(12)
Dy(13)-O(172)#1	2.549(10)	Ni(8)-N(5)	2.038(15)
Dy(13)-O(170)	2.551(10)	Ni(8)-O(42)	2.048(13)
Dy(13)-O(174)	2.854(13)	Ni(8)-O(72)	2.061(14)
Dy(17)-O(109)	2.329(10)	Ni(9)-O(159)	2.081(14)
Dy(17)-O(115)	2.348(11)	Ni(9)-N(15)	2.096(15)
Dy(17)-O(85)	2.357(9)	Ni(10)-O(59)	2.020(13)
Dy(17)-O(9)	2.369(8)	Ni(10)-O(155)	2.033(12)
Dy(17)-O(137)	2.370(7)	Ni(10)-O(45)	2.051(13)
Dy(17)-O(6)	2.373(8)	Ni(10)-O(23)	2.055(12)
Dy(17)-O(167)	2.591(9)	Ni(10)-O(56)#1	2.068(14)
Dy(17)-O(97)	2.660(11)	Ni(10)-N(11)	2.069(16)
Dy(18)-O(141)	2.308(7)	Ni(11)-O(142)	1.996(12)
Dy(18)-O(167)	2.314(8)	Ni(11)-O(63)	2.041(13)
Dy(18)-O(62)	2.408(10)	Ni(15)-O(121)	2.08(2)
Dy(18)-O(73)	2.411(10)	Ni(15)-N(13)	2.122(17)
Dy(18)-O(161)	2.42(3)	Ni(16)-O(168)	1.984(12)
Dy(18)-O(34W)	2.41(2)	Ni(16)-O(54)	2.034(13)

Dy(18)-O(109)	2.452(10)	Ni(16)-O(25)	2.037(13)
Dy(18)-O(119)	2.617(16)	Ni(16)-O(82)	2.048(15)
Dy(18)-O(163)	2.746(16)	Ni(16)-O(20W)	2.073(18)
Dy(19)-O(73)	2.319(9)	Ni(16)-N(19)	2.087(16)
Dy(19)-O(156)#1	2.370(9)	Ni(17)-O(158)	2.004(12)
Dy(19)-O(147)	2.370(8)	Ni(17)-O(40)	2.024(13)
Dy(19)-O(79)	2.375(10)	Ni(23)-O(93)	2.251(15)
Dy(19)-O(150)	2.384(8)	Ni(23)-O(94)	2.260(15)
Dy(19)-O(92)#1	2.383(12)	Ni(23)-O(98)	2.309(16)
Dy(19)-O(62)	2.600(10)	Ni(23)-O(19X)	2.39(3)
Dy(19)-O(77)	2.619(10)	Ni(24)-O(73)	2.234(13)
Dy(19)-O(114)	2.734(11)	Ni(24)-O(79)	2.256(15)
Dy(18)-O(141)	2.308(7)	Ni(24)-O(92)#1	2.257(15)
Dy(24)-O(166)	2.309(12)	Ni(24)-O(35W)	2.28(3)
Dy(20)-O(158)	2.355(9)	Ni(25)-O(88)	2.051(18)
Dy(20)-O(148)	2.386(9)	Ni(25)-O(5W)	2.10(4)
Dy(20)-O(77)#1	2.382(10)	Ni(14)-N(18)	2.037(13)
Dy(20)-O(169)	2.393(9)	Ni(14)-O(30)	2.043(9)
Dy(20)-O(132)	2.464(11)	Ni(14)-O(55)	2.066(8)
Dy(20)-O(22)	2.475(10)	Ni(14)-O(17W)	2.084(13)
Dy(15)-O(2B)	2.48(3)	Ni(15)-O(148)	1.995(9)
Dy(15)-O(32)	2.490(11)	Ni(15)-O(22)	2.030(10)
Dy(15)-O(13)	2.519(11)	Ni(15)-O(33)	2.035(10)
Dy(15)-O(177)	2.78(3)	Ni(15)-O(39)	2.076(8)
Dy(16)-O(142)	2.387(13)	Ni(15)-O(121)	2.094(10)
Dy(16)-O(150)	2.415(11)	Ni(15)-N(13)	2.121(12)
Dy(16)-O(147)	2.429(11)	Ni(16)-O(168)	1.990(8)
Dy(16)-O(156)#1	2.439(12)	Ni(16)-O(54)	2.025(10)
Dy(16)-O(19)#1	2.444(12)	Ni(16)-O(82)	2.041(10)
Dy(16)-O(39)#1	2.459(13)	Ni(16)-O(25)	2.038(9)
Dy(17)-O(109)	2.321(14)	Ni(16)-O(20W)	2.096(13)
Dy(17)-O(115)	2.338(15)	Ni(16)-N(19)	2.097(11)
Dy(17)-O(85)	2.347(13)	Ni(17)-O(158)	2.011(8)
Dy(17)-O(9)	2.360(11)	Ni(17)-O(34)	2.018(9)

Table S7 Selected bond angles (°) for compound **2**

O(146)-Dy(1)-O(139)	75.0(4)	O(143)-Ni(1)-O(171)	96.6(5)
O(146)-Dy(1)-O(154)	74.9(4)	O(143)-Ni(1)-O(18)	98.8(5)
O(139)-Dy(1)-O(154)	72.8(4)	O(171)-Ni(1)-O(18)	89.2(6)

O(146)-Dy(1)-O(12)	139.1(4)	O(143)-Ni(1)-O(21)	84.6(4)
O(139)-Dy(1)-O(12)	93.0(4)	O(171)-Ni(1)-O(21)	92.2(6)
O(154)-Dy(1)-O(12)	139.3(4)	O(18)-Ni(1)-O(21)	176.2(5)
O(146)-Dy(1)-O(134)	66.8(4)	O(143)-Ni(1)-O(10)	83.6(4)
O(139)-Dy(1)-O(134)	73.2(4)	O(171)-Ni(1)-O(10)	178.3(6)
O(154)-Dy(1)-O(134)	133.8(4)	O(18)-Ni(1)-O(10)	92.4(5)
O(12)-Dy(1)-O(134)	72.3(4)	O(21)-Ni(1)-O(10)	86.2(5)
O(146)-Dy(1)-O(46)	138.4(4)	O(143)-Ni(1)-N(12)	160.5(5)
O(139)-Dy(1)-O(46)	139.3(4)	O(171)-Ni(1)-N(12)	97.1(6)
O(154)-Dy(1)-O(46)	92.2(4)	O(18)-Ni(1)-N(12)	95.1(5)
O(12)-Dy(1)-O(46)	73.9(4)	O(21)-Ni(1)-N(12)	81.2(5)
O(134)-Dy(1)-O(46)	133.7(4)	O(10)-Ni(1)-N(12)	82.3(5)
O(146)-Dy(1)-O(149)	132.7(4)	O(136)-Ni(2)-O(41)#1	84.2(5)
O(139)-Dy(1)-O(149)	65.4(4)	O(136)-Ni(2)-O(51)#1	96.8(5)
O(139)-Dy(1)-O(135)	133.7(4)	O(41)#1-Ni(2)-O(27)	91.1(5)
O(154)-Dy(1)-O(135)	68.3(4)	O(51)#1-Ni(2)-O(27)	88.0(5)
O(12)-Dy(1)-O(135)	133.1(4)	O(13)#1-Ni(2)-O(27)	178.5(5)
O(134)-Dy(1)-O(135)	119.2(4)	O(136)-Ni(2)-N(14)#1	163.4(5)
O(46)-Dy(1)-O(135)	66.9(4)	O(41)#1-Ni(2)-N(14)#1	83.9(5)
O(149)-Dy(1)-O(135)	119.8(4)	O(51)#1-Ni(2)-N(14)#1	95.2(5)
O(26)-Dy(1)-O(135)	72.7(4)	O(13)#1-Ni(2)-N(14)#1	82.7(5)
O(9)-Dy(2)-O(133)	133.1(4)	O(27)-Ni(2)-N(14)#1	96.1(5)
O(9)-Dy(2)-O(6)	74.5(4)	O(134)-Ni(3)-O(64)	101.4(5)
O(133)-Dy(2)-O(6)	67.3(4)	O(134)-Ni(3)-O(26)	84.0(5)
O(9)-Dy(2)-O(137)	72.9(4)	O(64)-Ni(3)-O(26)	174.6(5)
O(133)-Dy(2)-O(137)	71.4(4)	O(134)-Ni(3)-O(49)	83.6(5)
O(6)-Dy(2)-O(137)	74.2(4)	O(64)-Ni(3)-O(49)	93.1(5)
O(9)-Dy(2)-O(10)	137.1(4)	O(26)-Ni(3)-O(49)	87.5(5)
O(133)-Dy(2)-O(10)	74.7(4)	O(134)-Ni(3)-O(35)	95.4(5)
O(6)-Dy(2)-O(10)	142.0(4)	O(64)-Ni(3)-O(35)	87.0(5)
O(137)-Dy(2)-O(10)	93.7(4)	O(26)-Ni(3)-O(35)	92.5(5)
O(9)-Dy(2)-O(7)	142.7(4)	O(49)-Ni(3)-O(35)	179.0(5)
O(133)-Dy(2)-O(7)	66.5(4)	O(134)-Ni(3)-N(7)	159.4(5)
O(6)-Dy(2)-O(7)	92.4(4)	O(64)-Ni(3)-N(7)	92.2(6)
O(137)-Dy(2)-O(7)	137.8(4)	O(26)-Ni(3)-N(7)	82.7(5)
O(10)-Dy(2)-O(7)	72.3(4)	O(49)-Ni(3)-N(7)	80.2(5)
O(9)-Dy(2)-O(138)	68.4(4)	O(35)-Ni(3)-N(7)	100.7(5)
O(133)-Dy(2)-O(138)	120.0(4)	O(37)-Ni(4)-O(16)	176.8(5)

O(6)-Dy(2)-O(138)	71.0(4)	O(37)-Ni(4)-O(145)	96.5(5)
O(137)-Dy(2)-O(138)	133.2(4)	O(16)-Ni(4)-O(145)	86.7(5)
O(10)-Dy(2)-O(138)	132.7(4)	O(37)-Ni(4)-O(15)	91.7(5)
O(7)-Dy(2)-O(138)	74.4(4)	O(16)-Ni(4)-O(15)	89.1(5)
O(9)-Dy(2)-O(14)	91.5(4)	O(145)-Ni(4)-O(15)	85.4(5)
O(133)-Dy(2)-O(14)	135.2(4)	O(37)-Ni(4)-N(21)	95.1(6)
O(155)-Dy(3)-O(141)	68.9(4)	O(16)-Ni(4)-N(21)	81.9(6)
O(9)-Dy(3)-O(167)	71.4(4)	O(145)-Ni(4)-N(21)	162.4(6)
O(67)-Dy(3)-O(167)	84.8(5)	O(15)-Ni(4)-N(21)	81.1(6)
O(155)-Dy(3)-O(167)	127.9(4)	O(37)-Ni(4)-O(38)	86.5(5)
O(141)-Dy(3)-O(167)	71.7(4)	O(16)-Ni(4)-O(38)	92.5(5)
O(9)-Dy(3)-O(138)	70.3(4)	O(145)-Ni(4)-O(38)	97.3(5)
O(67)-Dy(3)-O(138)	73.9(5)	O(15)-Ni(4)-O(38)	176.9(5)
O(155)-Dy(3)-O(138)	130.4(4)	O(138)-Ni(5)-O(17)	82.6(5)
O(154)-Dy(4)-O(145)	148.0(4)	O(138)-Ni(5)-O(14)	83.3(5)
O(154)-Dy(4)-O(80)	71.1(4)	O(17)-Ni(5)-O(14)	89.3(5)
O(145)-Dy(4)-O(80)	128.4(4)	O(138)-Ni(5)-O(70)	102.1(5)
O(154)-Dy(4)-O(88)	140.7(5)	O(17)-Ni(5)-O(70)	175.3(5)
O(145)-Dy(4)-O(88)	71.2(5)	O(14)-Ni(5)-O(70)	90.8(5)
O(80)-Dy(4)-O(88)	83.2(5)	O(138)-Ni(5)-O(15W)	95.7(6)
O(154)-Dy(4)-O(11)	98.3(4)	O(17)-Ni(5)-O(15W)	89.1(6)
O(145)-Dy(4)-O(11)	70.2(4)	O(14)-Ni(5)-O(15W)	178.2(7)
O(80)-Dy(4)-O(11)	72.3(4)	O(70)-Ni(5)-O(15W)	90.8(7)
O(88)-Dy(4)-O(11)	101.6(5)	O(138)-Ni(5)-N(3)	158.3(6)
O(145)-Dy(4)-O(135)	128.3(4)	O(17)-Ni(5)-N(3)	81.5(5)
O(80)-Dy(4)-O(135)	85.9(4)	O(14)-Ni(5)-N(3)	81.8(5)
O(88)-Dy(4)-O(135)	77.6(5)	O(70)-Ni(5)-N(3)	93.8(6)
O(11)-Dy(4)-O(135)	158.0(4)	O(15W)-Ni(5)-N(3)	98.8(7)
O(154)-Dy(4)-O(15)	76.9(4)	O(135)-Ni(6)-O(31)	83.8(5)
O(145)-Dy(4)-O(15)	71.1(4)	O(135)-Ni(6)-O(36)	96.7(5)
O(80)-Dy(4)-O(15)	127.4(4)	O(31)-Ni(6)-O(36)	178.6(5)
O(88)-Dy(4)-O(15)	141.6(5)	O(31)-Ni(6)-N(1)	82.4(6)
O(11)-Dy(4)-O(15)	72.3(4)	O(36)-Ni(6)-N(1)	97.5(6)
O(135)-Dy(4)-O(15)	121.9(4)	O(46)-Ni(6)-N(1)	81.4(5)
O(157)-Dy(5)-O(140)	146.9(4)	O(102)-Ni(6)-N(1)	96.8(7)
O(170)-Dy(5)-O(140)	129.5(4)	O(78)-Ni(7)-O(149)	95.7(5)
O(157)-Dy(5)-O(153)	99.7(4)	O(78)-Ni(7)-O(20)	88.7(5)
O(170)-Dy(5)-O(153)	72.2(4)	O(149)-Ni(7)-O(20)	98.8(5)

O(140)-Dy(5)-O(153)	70.2(4)	O(78)-Ni(7)-O(12)	177.4(5)
O(157)-Dy(5)-O(108)	137.9(5)	O(20)-Ni(7)-O(12)	89.0(5)
O(170)-Dy(5)-O(108)	88.8(5)	O(78)-Ni(7)-O(28)	92.7(5)
O(140)-Dy(5)-O(108)	73.6(5)	O(149)-Ni(7)-O(28)	83.0(4)
O(153)-Dy(5)-O(108)	109.4(5)	O(20)-Ni(7)-O(28)	177.6(5)
O(157)-Dy(5)-O(168)	69.1(4)	O(12)-Ni(7)-O(28)	89.6(5)

Table S8 Hydrogen bond interactions in compound **2**

D-H···A	d(D···H) /Å	d(H···A)/ Å	d(D···A)/Å	D-H···A (°)
N1-H1A··O1	0.98	2.19	2.90(4)	128
N2-H2A··O123	0.98	2.39	3.33(3)	160
N3-H3A··O13W	0.98	1.91	2.86(4)	161
N4-H4A··O24	0.98	2.11	2.911(18)	137
N7-H7A··O51	0.98	2.40	3.15(2)	133
N7-H7A··O27	0.98	2.17	3. 03 (2)	145
N8-H8A··O191	0.98	2.02	2.98(5)	167
N12-H12A··O74	0.98	2.00	2.86(2)	149
N1C-H1CB··O14W	0.89	1.59	2.40(9)	149
N14-H14A··O64	0.98	2.19	3.124(19)	158
N15-H15A··O6W	0.98	2.19	3.08(3)	151
N17-H17A··O14W	0.98	2.57	3.41(7)	144
N20-H20D··O29W	0.98	2.16	3.00(6)	142
N22-H22A··O190	0.98	2.10	3.06(2)	165
C2-H2B··O74	0.97	2.51	3.29(3)	137
C4A-H4AA··O67	0.93	2.46	2.79(2)	101
C6-H6C··O101	0.97	2.41	2.32(3)	155
C14-H14C··O159	0.97	2.16	2.99(2)	142
C15-H15B··O24	0.97	2.51	3.15(2)	123
C46-H46B··O12W	0.97	2.60	3.51(4)	157
C46-H46B··O74	0.97	2.59	3.17(2)	119
C75-H75B··O131	0.97	2.51	3.46(3)	167

C91-H91A··O66	0.97	2.41	3.32(5)	156