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Crystal structure of a heterotrimetallic 12-metallacrown-4 with 2-propylvalerate anion bridges

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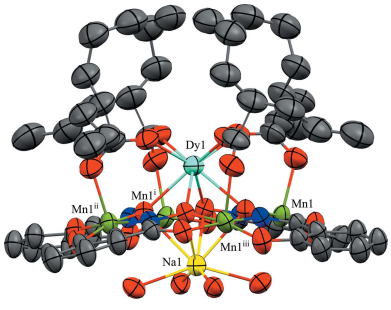
The synthesis and single-crystal X-ray structure for tetraaquatetrakis(μ-2-propylvalerato)tetrakis(μ_4-salicylhydroximato)dysprosiumtetramanganesesodium dimethylformamide tetrasolvate, $[DyMn_4Na(C_7H4NO_3)_4(C_8H_{15}O_2)_4(H_2O)_4]\cdot 4C_3H_7NO$ or $DyNa(2\text{-}PV)_4[12\text{-}MC_{Mn^{III}Nshi}\text{-}4](H_2O)_4\cdot 4DMF$, **1**, where MC is metallacrown, shi^{3-} is salicylhydroximate, 2-PV is 2-propylvalerate, and DMF is *N*,*N*-dimethylformamide, is reported. The slightly domed metallamacrocycle contains four ring Mn^{III} ions and four shi^{3-} ligands that generate an [Mn^{III}—N—O] repeat unit that recurs four times. The ring Mn^{III} ions are five-coordinate with a square-pyramidal shape. Furthermore, the metallacrown binds both a Dy^{III} ion and a Na^+ ion in the central cavity. The central ions are located on opposite faces of the cavity with the Dy^{III} ion located on the convex side of the MC and the Na^+ ion located on the concave side. Each central ion is eight-coordinate although they possess different geometries. The Dy^{III} ion has a square-antiprismatic shape, while the Na^+ ion has an extremely distorted biaugmented trigonal–prismatic shape. The four 2-propylvalerate anions help to tether the Dy^{III} to the MC cavity by forming bridges between the Dy^{III} ion and each ring Mn^{III} ion. Moreover, the interstitial DMF molecules are hydrogen bonded to the water molecules that complete the coordination environment of the Na^+ ion. The metallacrown framework (excluding the Dy^{III} and Na^+ ions), the bridging 2-propylvalerate, and the interstitial DMF molecule experience whole-molecule disorder due to the rotational orientation of the metallamacrocycle. Additionally, the main moiety alkyl chain of the 2-propylvalerate is disordered over two additional orientations, and the main moiety interstitial DMF molecule is disordered over one additional orientation. The main moiety of the metallacrown framework refined to 0.9030 (14), while the minor B-moiety refined to 0.0971 (14). The main moiety alkyl chain of the 2-propylvalerate refined to 0.287 (3): 0.309 (3): 0.307 (3), and the minor B-moiety alkyl chain refined to 0.0971 (14). Lastly, the main moiety interstitial DMF refined to 0.549 (3): 0.354 (3), and the minor B-moiety DMF refined to 0.0971 (14).

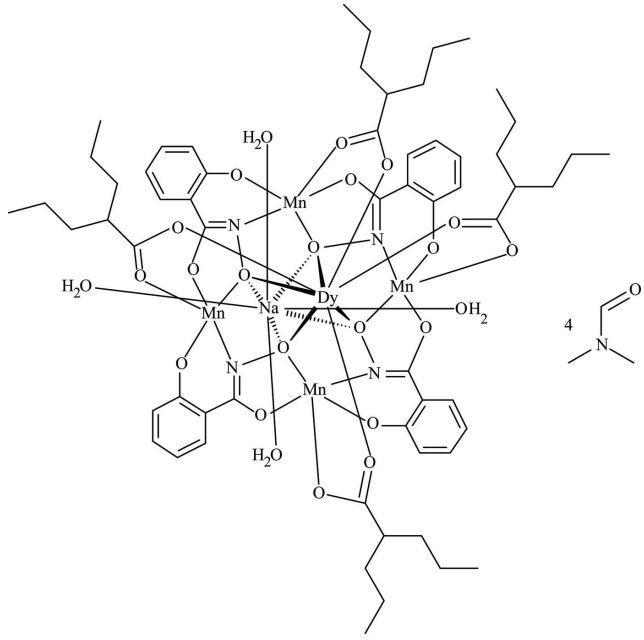
1. Chemical context

Materials that combine both 3*d* and 4*f* metal ions have potentially interesting magnetic properties as a result of the interaction between the paramagnetic centers. In particular, 3*d*–4*f* materials have applications in the areas of single-molecule magnetism (Liu *et al.*, 2015), single-chain magnetism (Wang *et al.*, 2014), and magnetorefrigeration (Lun *et al.*, 2021). The systematic synthesis of these heterometallic compounds is of interest to chemists and material scientists, and metallacrowns (MC) are a class of molecules particularly suited for the investigation of such materials because of their ability to interchange components of the molecule while maintaining the overall structural features (Mezei *et al.*, 2007; Lutter *et al.*, 2018).



Metallacrowns are metallamacrocyclic compounds with a metal–nitrogen–oxygen repeat unit about the inner ring. Several 3*d*–4*f* metallacrown systems have proven to be single-molecule magnets (SMM) (Boron, 2022) and magnetorefrigerates (Lutter *et al.*, 2021; Salerno *et al.*, 2021; Saha *et al.*, 2022). Indeed, we have been particularity focused on a lanthanide–manganese 12-metallacrown-4 system, *Ln*Na*Y*$_4$[12-MC$_{Mn^{III}Nshi}$-4], where *Y* is a carboxylate anion and shi^{3-} is salicylhydroximate (Azar *et al.*, 2014). These metallacrowns are based on a 12-membered MC ring with four oxygen atoms comprising the MC cavity. In addition, four Mn^{III} are part of the MC ring and the central cavity captures two cations in the central cavity: an *Ln*^{III} ion and a Na$^+$ ion. The two cations bind to opposite sides of the MC cavity. Furthermore, four carboxylate anions bridge between each ring Mn^{III} ions and the central *Ln*^{III} ion. We first reported these heterotrimetallic compounds with acetate bridges in 2014 (Azar *et al.*, 2014) and demonstrated that the MCs could bind a range of *Ln*^{III} ions in the central cavity, Pr to Yb (except Pm). Subsequently, we investigated the single-molecule magnet behavior of a series of DyNa*Y*$_4$[12-MC$_{Mn^{III}Nshi}$-4] compounds, where *Y* is acetate, trimethylacetate, benzoate, or 2-hydroxybenzoate (*i.e.* salicylate) (Boron *et al.*, 2016). In this series, only the MCs with bridging 2-hydroxybenzoate anions displayed single-molecule magnet properties. We hypothesized that the Lewis basicity of the bridging ligand may affect the magnetic coupling between the metal centers, thus switching on or off the SMM behavior. The pK_a of the parent carboxylic acid was used as a proxy for the Lewis basicity of the carboxylate, with lower pK_a values indicating greater electron-withdrawing ability for the subsequent carboxylate anion. For the investigated carboxylate anions, 2-hydroxybenzoic acid has the lowest pK_a (2.98) while the other acids are of comparable pK_a values, 4.20 for benzoic acid, 4.76 for acetic acid, and 5.03 for trimethylacetic acid (Haynes, 2010). Therefore, 2-hydroxybenzoate is the most electron-withdrawing in the series, and this may affect the magnetic coupling between the Dy^{III} and Mn^{III} ions. We have also extended the types of DyNa*Y*$_4$[12-MC$_{Mn^{III}Nshi}$-4] structures by synthesizing complexes with 3-hydroxy- and 4-hydroxybenzoate (Manickas *et al.*, 2020) and with halogenated benzoate anions (2-fluoro-, 3-fluoro-, 4-fluoro-, 2-chloro-, 3-chloro-, 3-bromo-, and 2-iodobenzoate; (Michael *et al.*, 2021). These carboxylates have a range of pK_a values that spans from 2.86 to 4.57 (Haynes, 2010), although at this time we have not investigated the SMM properties of these compounds.

Seeking to expand the types of DyNa*Y*$_4$[12-MC$_{Mn^{III}Nshi}$-4] structures beyond benzoate anions, we decided to determine if 2-propylvalerate, which has two propyl chains off the carboxylate carbon atom, could lead to MC formation. Herein we report the synthesis and crystal structure DyNa(2-PV)$_4$[12-MC$_{Mn^{III}Nshi}$-4](H$_2$O)$_4$·4DMF, **1**, where 2-PV is 2-propylvalerate and DMF is *N*,*N*-dimethylformamide. Although the pK_a of the parent 2-propylvaleric acid is 4.6 (Haynes, 2010) and the MC is not expected to possess SMM based on previous results, the 3*d*–4*f* compound will allow further investigation into the molecular characteristics that lead to single-molecule magnetism.



2. Structural commentary

DyNa(2-PV)$_4$[12-MC$_{Mn^{III}Nshi}$-4](H$_2$O)$_4$·4DMF, **1**, is centered about a crystallographic C_4 axis along the Dy^{III} and Na$^+$ ions located in the central cavity of the metallamacrocycle. The fourfold rotational axis generates a metallacrown with four ring Mn^{III} ions and four shi^{3-} ligands that yield the Mn^{III}–N–O repeat unit. In addition, four 2-propylvalerate anions form bridges between each ring Mn^{III} ion and the central Dy^{III} ion, and four interstitial DMF molecules are hydrogen bonded to the sodium-coordinated water molecules of the metallacrown. The metallacrown framework (excluding the Dy^{III} and Na$^+$ ions), the bridging 2-propylvalerate, and the interstitial DMF molecule experience whole molecule disorder as a result of the rotational orientation of the macrocycle. The main moiety of the molecule has an Mn^{III}–N–O counterclockwise rotation about the C_4 axis, while the minor B-moiety has an Mn^{III}–N–O clockwise rotation about the C_4 axis. Furthermore, the main moiety alkyl chain of the 2-propylvalerate is disordered over two additional orientations, and the main moiety interstitial DMF molecule is disordered over one additional orientation. The main moiety of the metallacrown framework occupancy refined to 0.9030 (14), while the minor B-moiety refined to 0.0971 (14). The main moiety alkyl chain of the 2-propylvalerate occupancies refined to 0.287 (3):0.309 (3):0.307 (3), and the minor B-moiety alkyl chain occupancy refined to 0.0971 (14). Lastly, the main moiety interstitial DMF refined to 0.549 (3):0.354 (3), and the minor B-moiety DMF refined to 0.0971 (14). In the following sections, all numbers refer to the major component, unless stated otherwise. The *Refinement* section contains complete details of the treatment of the disorder.

The oxidation states of the metal ions were determined based on overall molecular charge considerations, structure

Table 1
Average bond length (Å) and bond-valence-sum (BVS) values (v.u.) used to support assigned oxidation states of the dysprosium and manganese ions of **1**.

	Avg. Bond length	BVS value	Assigned oxidation state
Dy1	2.361	3.14	3+
Mn1	1.953	3.02	3+

features of the manganese ion, and bond-valence sum (BVS) values. The four triply deprotonated shi^{3-} ligands and four 2-propylvalerate anions provide an overall 16- charge, which is counterbalanced by one Dy^{III} ion, one Na^{+} ion, and four Mn^{III} ions (16+ total charge). In addition, the Mn^{III} ion possesses an elongated bond along the *z*-axis [2.126 (5) Å] and compressed bonds in the *xy* lane [1.840 (4) to 1.946 (5) Å], which are typical of high spin $3d^4$ ions. Lastly, the BVS values (Liu & Thorp, 1993; Trzesowska *et al.*, 2004) indicate that the Dy^{III} and Mn^{III} ions have a 3+ charge (Table 1).

Both central ions, Dy^{III} and Na^{+}, are eight-coordinate although with different coordination geometries (Fig. 1; Table 2). The metallacrown framework of **1** is slightly domed with the Dy^{III} ion located on the convex side of the metallamacrocycle and the Na^{+} ion located on the concave side. The Dy^{III} ion is bound to the four oxime oxygen atoms of the MC cavity, which are provided by four different shi^{3-} ligands, and four carboxylate oxygen atoms of four different 2-propylvalerate anions. The carboxylate groups of the 2-propylvalerate anions form three atom bridges to each ring Mn^{III} ion. An analysis of the geometry with the program *SHAPE 2.1* (Llunell *et al.*, 2013; Pinsky & Avnir, 1998) best describes the shape as a square antiprism (Casanova *et al.*, 2005). The Continuous Shape Measure (CShM) value is 0.747, indicating

Table 2
Continuous Shape Measure (CShM) values for the geometry about the eight-coordinate central Dy^{III} and Na^{+} ions of **1**.

Shape	Dy^{III}	Na^{+}
Octagon (D_{8h})	32.056	32.142
Heptagonal pyramid (C_{7v})	23.666	26.075
Hexagonal bipyramid (D_{6h})	16.870	13.023
Cube (O_h)	9.253	5.054
Square antiprism (D_{4d})	0.747	3.803
Triangular dodecahedron (D_{2d})	2.730	4.048
Johnson gyrobifastigium (J26; D_{2s})	17.553	16.887
Johnson elongated triangular bipyramid (J14; D_{3h})	30.395	28.728
Johnson biaugmented trigonal prism (J50; C_{2v})	3.036	5.420
Biaugmented trigonal prism (C_{2v})	1.991	3.667
Johnson snub diphenoid (J84; D_{2d})	5.971	8.256
Triakis tetrahedron (T_d)	10.118	5.959
Elongated trigonal bipyramid (D_{3h})	25.708	25.581

that the geometry approaches that of an ideal square antiprism. The Na^{+} ion is also bound to the four oxime oxygen atoms of the MC cavity, and the coordination environment is completed by four water molecules. Each water molecule also hydrogen bonds (Fig. 2) to two interstitial DMF molecules. The geometry of the Na^{+} ion cannot be clearly defined based on CShM values as the two lowest values are 3.667 for a biaugmented trigonal prism and 3.803 for a square antiprism. Typically values above 3.0 indicate significant distortions from ideal geometry; thus, for the Na^{+} ion the geometry cannot be unambiguously specified (Cirera *et al.*, 2005).

The ring Mn^{III} ion is five-coordinate with a square-pyramidal shape (Table 3). The basal region of the ligand environment consists of two *trans* chelate rings from two different shi^{3-} ligands. One shi^{3-} ligand forms a five-membered chelate ring by binding with the oxime and carbonyl oxygen atoms of

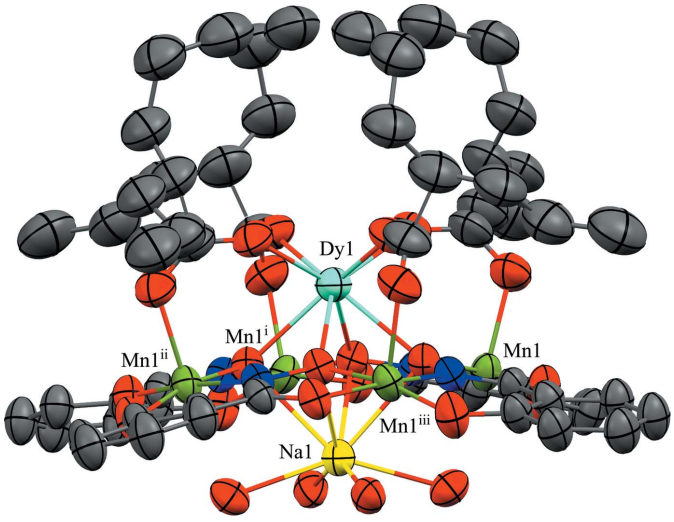


Figure 1
The single-crystal X-ray structure of DyNa$(O_2C_8H_{15})_4$[12-MC$_{Mn^{III}N(shi)}$-4]$(H_2O)_4$·4DMF, **1**. A side view with only the metal atoms labeled for clarity. The displacement ellipsoids are at the 50% probability level. For clarity, hydrogen atoms, solvent molecules, and disorder have been omitted. Color scheme: aqua – Dy^{III}, green – Mn^{III}, yellow – Na^{+}, red – oxygen, blue – nitrogen, and gray – carbon. All figures were generated with the program *Mercury* (Macrae *et al.*, 2020). [Symmetry codes: (i) $+x$, $-y+\frac{1}{2}$, z; (ii) $-x+\frac{1}{2}$, $-y+\frac{1}{2}$, z; (iii) $-x+\frac{1}{2}$, y, z.]

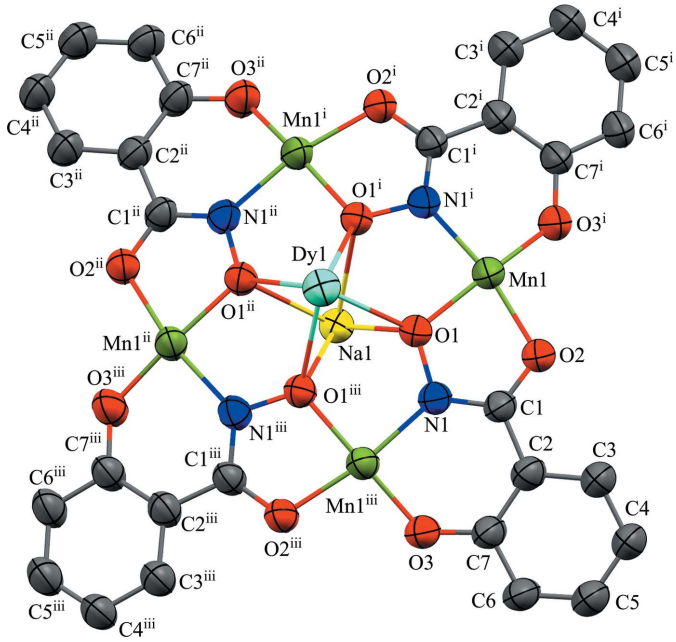


Figure 2
The single-crystal X-ray structure of DyNa$(O_2C_8H_{15})_4$[12-MC$_{Mn^{III}N(shi)}$-4]$(H_2O)_4$·4DMF, **1**. A top view with the four bridging 2-propylvalerate anions removed for clarity. See Fig. 1 for additional display details. [Symmetry codes: (i) x, $-y+\frac{1}{2}$, z; (ii) $-x+\frac{1}{2}$, $-y+\frac{1}{2}$, z; (iii) $-x+\frac{1}{2}$, y, z.]

Table 3
Continuous Shape Measure (CShM) values for the geometry about the five-coordinate ring Mn^{III} ion of **1**.

Shape	Pentagon (D_{5h})	Vacant octahedron (C_{4v})	Trigonal bipyramid (D_{3h})	Spherical square pyramid (C_{4v})	Johnson trigonal bipyramid (J12; D_{3h})
Mn1	30.515	0.832	5.422	0.739	7.504

Table 4
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6A···O7	0.86 (2)	2.02 (2)	2.860 (15)	165 (5)
O6—H6E···O7^{i}	0.83 (2)	1.99 (2)	2.809 (19)	167 (5)
O6B—H6C···O7B^{i}	0.84 (2)	2.00 (2)	2.80 (3)	159 (10)
O6B—H6D···O7B	0.85 (2)	2.01 (2)	2.859 (19)	177 (10)

Symmetry code: (i) $-y+\frac{1}{2}, x, z$.

the ligand, and the other shi^{3-} ligand forms a six-membered chelate ring by binding with the oxime nitrogen and phenolate oxygen atom of the ligand. The apical position is occupied by the carboxylate oxygen atom of the bridging 2-propylvalerate anion. Lastly, the water molecule that is coordinated to the Na^{+} ion forms a long interaction [2.527 (5) Å] with the Mn^{III} ion.

3. Supramolecular features

For **1**, the main metallacrown molecule forms hydrogen bonds to the interstitial DMF molecules *via* the water molecules bound to the central sodium ion. Each water molecule is hydrogen bonded to the carbonyl oxygen atom of two adjacent DMF molecules (Table 4, Fig. 3). This generates a small hydrogen-bonding network on the concave side of the metallacrown between the four sodium-bound water molecules and the four interstitial DMF molecules. A similar hydrogen-bonding connectivity is also observed for the minor B-moiety of the compound. These hydrogen bonds and pure van der Waals forces contribute to the overall packing of the molecules.

4. Database survey

A survey of the Cambridge Structural Database (CSD version 5.43, update September 2022, Groom *et al.*, 2016) reveals forty $LnXY_4$[12-$MC_{Mn^{III}N(shi)}$-4] structures, where X is a counter-cation with a 1+ charge and Y is a carboxylate anion (Azar *et al.*, 2014; Travis *et al.*, 2015, 2016; Boron *et al.*, 2016; Cao *et al.*, 2016; Qin *et al.*, 2017; Anthanasopoulou *et al.*, 2018; Manickas *et al.*, 2020; Michael *et al.*, 2021). The central Ln^{III} metal ions include the lanthanide ions from Pr to Yb (except Pm) and yttrium. The counter-cation X is usually an Na^{+} or K^{+} ion that is also bound to the central cavity, but other unbound counter-cations such as tetrabutylammonium, tetraethylammonium, and triethylammonium have been employed. A range of bridging carboxylate anions (Y) have been used including acetate (OAc), trimethylacetate (TMA), benzoate (ben), 2-hydroxybenzoate (2-OHben), 3-hydroxybenzoate (3-OHben), 4-hydroxylbenzoate (4-OHben), 2-fluorobenzoate (2-Fben), 3-fluorobenzoate (3-Fben), 4-fluorobenzoate (4-Fben), 2-chlorobenzoate (2-Clben), 4-chlorobenzoate (4-Clben), 3-bromobenzoate (3-Brben), and 2-iodobenzoate (2-Iben). Of the forty structures, thirteen contain both Dy^{III} and Na^{+} as in **1** (Azar *et al.*, 2014; Boron *et al.*, 2016; Manickas *et al.*, 2020; Michael *et al.*, 2021). The structural comparison of **1** will be limited to the MCs that contain Dy^{III} and Na^{+} ions captured in the central MC cavity (Table 5). Analysis of the parameters that define the MC cavity and framework such as the cavity radius, the cross cavity Mn^{III}—Mn^{III} distance, the distance between adjacent Mn^{III} ions, the cross cavity oxime oxygen–oxime oxygen distance, and the distance of the Dy^{III} ion from the oxime oxygen mean plane reveals that the identity of the bridging carboxylate has little impact on the overall metallacrown structure. [These parameters were determined as previously defined (Azar *et al.*, 2014)]. Indeed, this is a hallmark of metallacrown chemistry, the ability to switch components of the molecular systems without significantly affecting the overall structure. This asset allows the systematic investigation of chemical and physical properties such as

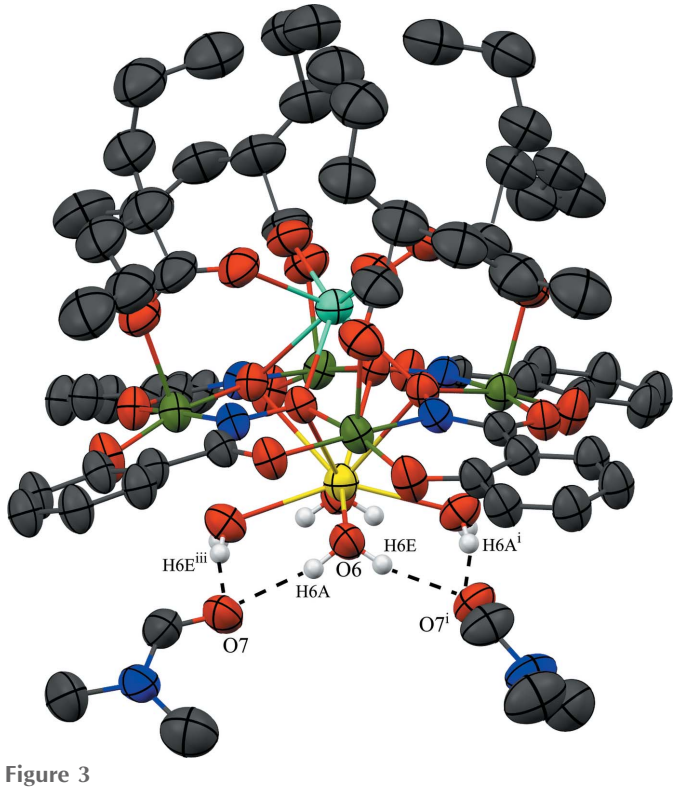


Figure 3
Intermolecular hydrogen bonding between the water molecules coordinated to the Na^{+} ion of **1** and the interstitial DMF molecules. Each coordinated water molecule forms hydrogen bonds with two neighboring DMF molecules. For clarity, only the hydrogen atoms (white) of the water molecules and only two of the four DMF molecules are displayed. See Fig. 1 for additional display details. [Symmetry codes: (i) $x, -y+\frac{1}{2}, z$; (iii) $-x+\frac{1}{2}, y, z$.]

Table 5
Structural feature comparison of **1** and other DyNa(carboxylate)$_4$[12-MC$_{Mn^{III}N(shi)}$-4] compounds (Å).

Compound	MC cavity radius[a]	Avg. cross-cavity Mn^{III}—Mn^{III} distance[a]	Avg. adjacent Mn^{III}—Mn^{III} distance[a]	Avg. cross-cavity O_{oxime}—O_{oxime} distance[a]	Dy^{III}–oxime oxygen mean plane distance[b]
DyNa(2-PV)$_4$[12-MC-4], **1**	0.54	6.51	4.60	3.68	1.6069 (37)
DyNa(OAc)$_4$[12-MC-4]	0.55	6.52	4.61	3.71	1.5918 (7)
DyNa(TMA)$_4$[12-MC-4]	0.56	6.51	4.60	3.73	1.5790 (19)
DyNa(ben)$_4$[12-MC-4]	0.54	6.51	4.60	3.69	1.5811 (18)
DyNa(2-OHben)$_4$[12-MC-4]	0.54	6.47	4.57	3.68	1.5094 (46)
DyNa(3-OHben)$_4$[12-MC-4]	0.56	6.53	4.62	3.72	1.5463 (25)
DyNa(4-OHben)$_4$[12-MC-4]	0.54	6.49	4.59	3.69	1.5925 (66)
DyNa(2-Fben)$_4$[12-MC-4]	0.55	6.51	4.60	3.71	1.5424 (52)
DyNa(3-Fben)$_4$[12-MC-4]	0.56	6.51	4.60	3.72	1.5567 (34)
DyNa(4-Fben)$_4$[12-MC-4]	0.54	6.52	4.61	3.68	1.5589 (45)
DyNa(2-Clben)$_4$[12-MC-4]	0.53	6.50	4.60	3.67	1.5883 (79)
DyNa(4-Clben)$_4$[12-MC-4]	0.55	6.54	4.62	3.71	1.5593 (13)
DyNa(3-Brben)$_4$[12-MC-4]	0.56	6.51	4.60	3.71	1.5685 (13)
DyNa(2-Iben)$_4$[12-MC-4]	0.54	6.50	4.59	3.68	1.5764 (117)

Notes: (*a*) Measured with *Mercury* (Macrae *et al.*, 2020); (*b*) measured with *SHELXL* (Sheldrick, 2015*b*).

magnetism or luminescence across a range of structures (Boron *et al.*, 2016; Chow *et al.*, 2016).

5. Synthesis and crystallization

Materials

Dysprosium(III) nitrate pentahydrate (99.99%) and manganese(II) nitrate tetrahydrate (98%) were purchased from Alfa Aesar. Salicylhydroxamic acid (H_3shi, >98%) and sodium 2-propylvalerate (>98.0%) were purchased from TCI America. DMF (ACS grade) was purchased from VWR Chemicals BDH. All reagents were used as received and without further purification.

Synthesis

DyNa(2-PV)$_4$[12-MC$_{Mn^{III}N(shi)}$-4](H$_2$O)$_4$·4DMF, **1.** Manganese(II) nitrate tetrahydrate (2 mmol, 0.5020 g) was dissolved in 10 mL of DMF to produce a clear and colorless solution. Dysprosium(III) nitrate pentahydrate (0.125 mmol, 0.0548 g), salicylhydroxamic acid (2 mmol, 0.3063 g), and sodium 2-propylvalerate (4 mmol, 0.4648 g) were then dissolved in 10 mL of DMF, resulting in a clear, slightly tan solution. Next the manganese(II) nitrate solution was added to the latter solution resulting in a clear yellow solution. Then the solution slowly darkened, eventually producing a dark-brown/black color. The solution was stirred overnight and then gravity filtered the next day. No precipitate was recovered, and the dark-brown/black filtrate was allowed to slowly evaporate to aid crystal growth. After 11 days, dark-brown/black X-ray-quality crystals formed. The crystals were isolated and washed with cold DMF. The percentage yield was 64% (0.1562 g) based on dysprosium(III) nitrate pentahydrate. FT–IR (ATR, cm^{-1}): 1656, 1600, 1571, 1551, 1514, 1465, 1435, 1390, 1319, 1258, 1249, 1155, 1100, 1060, 1030, 932, 866, 820, 769, 754, 679, 665, 649, 606.

6. Refinement

Minor whole molecule disorder was detected for the metallacrown molecule (excluding the dysprosium and sodium ions) and all organic fragments, and the disorder was refined. The metallacrown is disordered by clockwise *versus* counter-clockwise rotation orientation of the metallamacrocycle, as are the 2-propylvalerate anion and the interstitial DMF molecule (major and minor components). In addition, the main moiety alkyl chain of the 2-propylvalerate is disordered over two additional sites, and the main moiety interstitial DMF molecule is disordered over one additional site. The U_{ij} components of ADPs for disordered atoms closer to each other than 2.7 Å were restrained to be similar (SIMU command of *SHELXL*). Occupancies were constrained to sum to unity for all sites using SUMP commands. The major and minor (B-moiety) metallacrown units were restrained to have similar geometries. For the B-moiety benzene ring of the salicylhydroximate, the C atoms were restrained to be close to planar (FLAT command of *SHELXL*). For the 2-propylvalerate anion, the major (including the additional alkyl-chain disorder) and B-moieties were restrained to have similar geometries. The C-atom positions of the 2-propylvalerate were further restrained based on typical carbon–carbon bond distances and angles. The B-moiety carboxylate carbon atom (C8*B*) was restrained to planarity (CHIV 0 command of *SHELXL*).

For the interstitial DMF molecule, the N atom was restrained to be equidistant from both carbon atoms of the methyl groups. In addition, the major (including the additional disorder) and B-moieties were restrained to have similar geometries. Hydrogen-atom positions of the water molecule coordinated to the sodium ion were refined and O—H and H···H distances were restrained to 0.84 (2) and 1.36 (2) Å, respectively. The water-O and H-atom positions were further restrained based on hydrogen-bonding considerations to the interstitial DMF molecule, and distances of all water H atoms to the sodium ion were restrained to be similar.

All other hydrogen atoms were placed in calculated positions and refined as riding on their carrier atoms with C—H distances of 0.95 Å for sp^2 carbon atoms and to 1.00, 0.99 and 0.98 Å for aliphatic C—H, CH_2 and CH_3 moieties, respectively. The U_{iso} values for hydrogen atoms were set to a multiple of the U_{eq} value of the carrying carbon or oxygen

Table 6
Experimental details.

Crystal data	
Chemical formula	$[DyMn_4Na(C_7H_4NO_3)_4(C_8H_{15}O_2)_4(H_2O)_4]\cdot 4C_3H_7NO$
M_r	1942.94
Crystal system, space group	Tetragonal, *P*4/*n*
Temperature (K)	150
a, *c* (Å)	18.2654 (9), 13.4219 (9)
V (Å^3)	4477.9 (5)
Z	2
Radiation type	Mo *Kα*
μ (mm^{-1})	1.45
Crystal size (mm)	0.45 × 0.43 × 0.15
Data collection	
Diffractometer	Bruker AXS D8 Quest
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.020, 0.060
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	87812, 7801, 5435
R_{int}	0.070
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.747
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.072, 0.257, 1.07
No. of reflections	7801
No. of parameters	708
No. of restraints	2628
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.36, −1.96

Computer programs: *APEX4* and *SAINT* (Bruker, 2022), SHELXT (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), *ShelXle* (Hübschle *et al.*, 2011), *Mercury* (Macrae *et al.*, 2020), and *publCIF* (Westrip, 2010).

atom (1.2 times for C—H and CH_2 groups and 1.5 for water molecules and methyl groups).

Subject to these conditions, the occupancy ratios refined as follows: main moiety metallacrown unit, 0.9030 (14); B-moiety metallacrown unit: 0.0971 (14); main alkyl chain of 2-propylvalerate, 0.287 (3):0.309 (3):0.307 (3); B-moiety alkyl chain, 0.0971 (14); main moiety interstitial DMF, 0.549 (3):0.354 (3); and B-moiety DMF, 0.0971 (14). Additional crystal data, data collection, and structure refinement details are summarized in Table 6.

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supporting information

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Crystal structure of a heterotrimetallic 12-metallacrown-4 with 2-propylvalerate anion bridges

Ozha A. Aziz, Matthias Zeller and Curtis M. Zaleski

Computing details

Data collection: *APEX4* (Bruker, 2022); cell refinement: *SAINT* (Bruker, 2022); data reduction: *SAINT* (Bruker, 2022); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015b), *ShelXle* (Hübschle *et al.*, 2011); molecular graphics: *Mercury* (Macrae *et al.*, 2020); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Tetraaquatetrakis(*μ*-2-propylvalerato)tetrakis(*μ*$_4$-salicylhydroximato)dysprosiumtetramanganesesodium dimethylformamide tetrasolvate,

Crystal data

$[DyMn_4Na(C_7H_4NO_3)_4(C_8H_{15}O_2)_4(H_2O)_4]\cdot 4C_3H_7NO$
M_r = 1942.94
Tetragonal, *P*4/*n*
a = 18.2654 (9) Å
c = 13.4219 (9) Å
V = 4477.9 (5) Å^3
Z = 2
F(000) = 2002.2
D_x = 1.441 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 9870 reflections
θ = 2.5–32.0°
μ = 1.45 mm^{-1}
T = 150 K
Plate, brown
0.45 × 0.43 × 0.15 mm

Data collection

Bruker AXS D8 Quest diffractometer
Radiation source: fine focus sealed tube X-ray source
Triumph curved graphite crystal monochromator
Detector resolution: 7.4074 pixels mm^{-1}
ω and phi scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.020, T_{max} = 0.060
87812 measured reflections
7801 independent reflections
5435 reflections with $I > 2\sigma(I)$
R_{int} = 0.070
θ_{max} = 32.1°, θ_{min} = 2.2°
h = −27→24
k = −27→27
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.257
S = 1.07
7801 reflections
708 parameters
2628 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1326P)^2 + 7.8338P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.013
$\Delta\rho_{max}$ = 1.36 e Å^{-3}
$\Delta\rho_{min}$ = −1.96 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Minor whole molecule disorder was detected for the metallacrown molecule and all organic fragments (excluding the dysprosium and sodium ions), and the disorder was refined. The metallacrown is disordered by clockwise vs counterclockwise rotation orientation of the metallacycle, as are the 2-propylvalerate anion and the interstitial DMF molecule (main moiety and B-moiety). In addition, the alkyl chain of the 2-propylvalerate of the main moiety is disordered over two additional sites, and the interstitial DMF molecule of the main moiety is disordered over one additional site. The Uij components of ADPs for disordered atoms closer to each other than 2.7 Angstrom were restrained to be similar (SIMU command of Shelxl). Occupancies were constrained to sum up to unity for all sites using SUMP commands.

The major and minor (B-moiety) metallacrown units were restrained to have similar geometries. For the B-moiety benzene ring of the salicylhydroximate, the C atoms were restrained to be close to planar (FLAT command of Shelxl). For the 2-propylvalerate anion, the major (including the additional alkyl chain disorder) and B moieties were restrained to have similar geometries. The C atoms positions 2-propylvalerate were further restrained based on typical carbon-carbon bond distances and angles. The B-moiety carboxylate carbon atom (C8B) was restrained to planarity (CHIV command of Shelxl).

For the interstitial DMF molecule, the N atom was restrained to be equidistant from both carbon atoms of the methyl groups. In addition, the major (including the additional disorder) and B moieties were restrained to have similar geometries.

H atom positions of the water molecule coordinated to the sodium ion were refined and O-H and H···H distances were restrained to 0.84 (2) and 1.36 (2) Angstrom, respectively. The water O and H atom positions were further restrained based on hydrogen bonding considerations to the interstitial DMF molecule and distances of all water H atoms to the sodium ion were restrained to be similar.

All other hydrogen atoms were placed in calculated positions and refined as riding on their carrier atoms with C-H distances of 0.95 Angstrom for sp2 carbon atoms. The Uiso values for hydrogen atoms were set to a multiple of the Ueq value of the carrying carbon atom (1.2 times for sp2 hybridized carbon atoms, 1.5 for water molecules and methyl groups).

Subject to these conditions the occupancy ratios were refined as follows:
main metallacrown unit: 0.9030 (14) B-moiety metallacrown unit: 0.0971 (14)
main alkyl chain of 2-propylvalerate: 0.287 (3): 0.309 (3): 0.307 (3) B-moiety alkyl chain: 0.0971 (14)
main interstitial DMF: 0.549 (3): 0.354 (3) B-moiety DMF: 0.0971 (14)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Dy1	0.250000	0.250000	0.78820 (3)	0.05635 (17)	
Na1	0.250000	0.250000	0.5233 (3)	0.0524 (8)	
Mn1	0.14036 (4)	0.39036 (4)	0.64460 (7)	0.0514 (2)	0.9030 (14)
O1	0.23534 (18)	0.34977 (18)	0.6685 (3)	0.0490 (7)	0.9030 (14)
O2	0.19756 (19)	0.4788 (2)	0.6261 (3)	0.0563 (8)	0.9030 (14)
O3	0.4255 (2)	0.4468 (2)	0.5953 (4)	0.0683 (11)	0.9030 (14)
N1	0.2918 (3)	0.4000 (2)	0.6508 (9)	0.0499 (9)	0.9030 (14)
C1	0.2672 (3)	0.4663 (3)	0.6306 (4)	0.0504 (9)	0.9030 (14)
C2	0.3191 (3)	0.5252 (3)	0.6105 (5)	0.0563 (11)	0.9030 (14)
C3	0.2920 (3)	0.5972 (3)	0.6059 (6)	0.0631 (12)	0.9030 (14)
H3	0.241645	0.605935	0.618711	0.076*	0.9030 (14)
C4	0.3373 (4)	0.6556 (3)	0.5831 (7)	0.0696 (15)	0.9030 (14)

H4	0.318007	0.703811	0.579281	0.084*	0.9030 (14)
C5	0.4104 (4)	0.6431 (3)	0.5662 (7)	0.0706 (16)	0.9030 (14)
H5	0.441614	0.683117	0.550566	0.085*	0.9030 (14)
C6	0.4391 (3)	0.5732 (3)	0.5714 (5)	0.0664 (14)	0.9030 (14)
H6	0.489816	0.565843	0.559009	0.080*	0.9030 (14)
C7	0.3947 (3)	0.5131 (3)	0.5948 (5)	0.0581 (11)	0.9030 (14)
O4	0.1581 (3)	0.3123 (3)	0.8654 (4)	0.0740 (12)	0.9030 (14)
O5	0.1146 (3)	0.4144 (3)	0.7956 (4)	0.0773 (12)	0.9030 (14)
C8	0.1192 (5)	0.3701 (5)	0.8633 (6)	0.0876 (18)	0.9030 (14)
C9	0.0809 (12)	0.3783 (19)	0.9652 (13)	0.104 (3)	0.287 (3)
H9	0.083089	0.327758	0.993659	0.125*	0.287 (3)
C10	0.0002 (12)	0.3888 (18)	0.943 (2)	0.107 (4)	0.287 (3)
H10A	−0.026153	0.392395	1.007540	0.128*	0.287 (3)
H10B	−0.005916	0.436207	0.908509	0.128*	0.287 (3)
C11	−0.0354 (14)	0.3317 (19)	0.883 (3)	0.112 (4)	0.287 (3)
H11A	−0.024496	0.283824	0.914116	0.135*	0.287 (3)
H11B	−0.012164	0.331805	0.816430	0.135*	0.287 (3)
C12	−0.1184 (14)	0.336 (2)	0.868 (3)	0.134 (8)	0.287 (3)
H12A	−0.134736	0.295366	0.826735	0.201*	0.287 (3)
H12B	−0.142903	0.334483	0.932844	0.201*	0.287 (3)
H12C	−0.130515	0.382683	0.834713	0.201*	0.287 (3)
C13	0.1196 (14)	0.4221 (16)	1.0385 (17)	0.110 (3)	0.287 (3)
H13A	0.102073	0.473193	1.032217	0.132*	0.287 (3)
H13B	0.172113	0.422004	1.020370	0.132*	0.287 (3)
C14	0.1138 (17)	0.401 (2)	1.1475 (15)	0.118 (4)	0.287 (3)
H14A	0.065666	0.377546	1.159867	0.142*	0.287 (3)
H14B	0.117293	0.445042	1.189532	0.142*	0.287 (3)
C15	0.177 (2)	0.345 (2)	1.177 (2)	0.126 (7)	0.287 (3)
H15A	0.155796	0.298178	1.197504	0.188*	0.287 (3)
H15B	0.205757	0.365358	1.232659	0.188*	0.287 (3)
H15C	0.209632	0.337212	1.119868	0.188*	0.287 (3)
O6	0.1794 (2)	0.3562 (3)	0.4701 (3)	0.0613 (9)	0.9030 (14)
H6A	0.207 (2)	0.383 (3)	0.433 (5)	0.092*	0.9030 (14)
H6E	0.144 (3)	0.343 (3)	0.435 (5)	0.092*	0.9030 (14)
C9C	0.0715 (15)	0.3936 (16)	0.9511 (19)	0.101 (3)	0.309 (3)
H9C	0.106691	0.370299	0.998520	0.122*	0.309 (3)
C10C	0.0167 (16)	0.3347 (15)	0.9718 (19)	0.109 (4)	0.309 (3)
H10C	0.039180	0.302612	1.022895	0.131*	0.309 (3)
H10D	−0.025323	0.359039	1.004438	0.131*	0.309 (3)
C11C	−0.0138 (15)	0.2868 (18)	0.898 (2)	0.111 (4)	0.309 (3)
H11C	0.009971	0.238535	0.907550	0.133*	0.309 (3)
H11D	0.002898	0.305348	0.832615	0.133*	0.309 (3)
C12C	−0.0956 (15)	0.272 (2)	0.888 (3)	0.122 (7)	0.309 (3)
H12D	−0.103422	0.228717	0.846672	0.183*	0.309 (3)
H12E	−0.119444	0.314501	0.857290	0.183*	0.309 (3)
H12F	−0.116690	0.263779	0.954439	0.183*	0.309 (3)
C13C	0.0880 (15)	0.4648 (13)	0.992 (2)	0.109 (3)	0.309 (3)
H13C	0.047967	0.481026	1.036652	0.131*	0.309 (3)

H13D	0.093563	0.501176	0.937993	0.131*	0.309 (3)
C14C	0.1596 (16)	0.4577 (15)	1.051 (2)	0.115 (4)	0.309 (3)
H14C	0.157564	0.485240	1.114240	0.138*	0.309 (3)
H14D	0.202082	0.474735	1.011202	0.138*	0.309 (3)
C15C	0.162 (2)	0.3702 (17)	1.070 (3)	0.120 (6)	0.309 (3)
H15D	0.212086	0.352187	1.056624	0.180*	0.309 (3)
H15E	0.127934	0.345735	1.024615	0.180*	0.309 (3)
H15F	0.149029	0.359666	1.138777	0.180*	0.309 (3)
C9D	0.0619 (11)	0.375 (2)	0.9491 (15)	0.103 (3)	0.307 (3)
H9D	0.064660	0.428430	0.964243	0.124*	0.307 (3)
C10D	−0.0186 (13)	0.3685 (16)	0.922 (2)	0.105 (4)	0.307 (3)
H10E	−0.046788	0.388208	0.979130	0.126*	0.307 (3)
H10F	−0.027281	0.401942	0.865313	0.126*	0.307 (3)
C11D	−0.0511 (17)	0.2995 (18)	0.896 (3)	0.118 (5)	0.307 (3)
H11E	−0.034898	0.263808	0.947376	0.142*	0.307 (3)
H11F	−0.028719	0.284003	0.832606	0.142*	0.307 (3)
C12D	−0.1338 (17)	0.290 (2)	0.885 (4)	0.132 (8)	0.307 (3)
H12G	−0.153174	0.264001	0.943212	0.198*	0.307 (3)
H12H	−0.144137	0.261731	0.824655	0.198*	0.307 (3)
H12I	−0.157015	0.338228	0.880081	0.198*	0.307 (3)
C13D	0.0917 (15)	0.3450 (15)	1.0424 (18)	0.108 (3)	0.307 (3)
H13E	0.114151	0.296643	1.029356	0.130*	0.307 (3)
H13F	0.051448	0.338078	1.090934	0.130*	0.307 (3)
C14D	0.1487 (15)	0.3961 (19)	1.086 (2)	0.113 (4)	0.307 (3)
H14E	0.174549	0.423623	1.033458	0.136*	0.307 (3)
H14F	0.184933	0.368783	1.126639	0.136*	0.307 (3)
C15D	0.100 (2)	0.4496 (19)	1.155 (3)	0.130 (7)	0.307 (3)
H15G	0.095446	0.497242	1.121771	0.194*	0.307 (3)
H15H	0.124177	0.455943	1.219388	0.194*	0.307 (3)
H15I	0.051571	0.428170	1.164385	0.194*	0.307 (3)
Mn1B	0.4210 (3)	0.3002 (3)	0.6438 (6)	0.0469 (15)	0.0971 (14)
O1B	0.3183 (10)	0.3217 (11)	0.655 (3)	0.050 (4)	0.0971 (14)
O2B	0.4248 (12)	0.4055 (10)	0.618 (3)	0.052 (3)	0.0971 (14)
O3B	0.2210 (14)	0.5183 (15)	0.620 (3)	0.058 (4)	0.0971 (14)
N1B	0.304 (2)	0.3966 (16)	0.648 (11)	0.054 (4)	0.0971 (14)
C1B	0.3622 (14)	0.4357 (11)	0.627 (4)	0.054 (3)	0.0971 (14)
C2B	0.3539 (14)	0.5138 (11)	0.608 (2)	0.056 (3)	0.0971 (14)
C3B	0.4195 (17)	0.5499 (18)	0.584 (4)	0.060 (3)	0.0971 (14)
H3B	0.463964	0.522861	0.582859	0.073*	0.0971 (14)
C4B	0.421 (2)	0.6238 (19)	0.563 (5)	0.062 (4)	0.0971 (14)
H4B	0.465052	0.648291	0.546111	0.074*	0.0971 (14)
C5B	0.355 (2)	0.660 (2)	0.567 (5)	0.063 (4)	0.0971 (14)
H5B	0.355061	0.711149	0.552398	0.076*	0.0971 (14)
C6B	0.290 (2)	0.6277 (15)	0.591 (4)	0.060 (4)	0.0971 (14)
H6B	0.246663	0.656543	0.595648	0.072*	0.0971 (14)
C7B	0.2863 (14)	0.5516 (15)	0.609 (4)	0.058 (3)	0.0971 (14)
O4B	0.249 (3)	0.352 (2)	0.881 (3)	0.090 (5)	0.0971 (14)
O5B	0.190 (3)	0.437 (3)	0.793 (3)	0.075 (6)	0.0971 (14)

C8B	0.215 (2)	0.414 (2)	0.872 (2)	0.091 (5)	0.0971 (14)
C9B	0.212 (3)	0.450 (3)	0.976 (3)	0.105 (4)	0.0971 (14)
H9B	0.244392	0.493722	0.971060	0.126*	0.0971 (14)
C10B	0.135 (3)	0.480 (4)	0.988 (4)	0.106 (4)	0.0971 (14)
H10G	0.102155	0.436896	0.990354	0.127*	0.0971 (14)
H10H	0.133232	0.502994	1.054169	0.127*	0.0971 (14)
C11B	0.102 (3)	0.531 (4)	0.920 (6)	0.107 (5)	0.0971 (14)
H11G	0.126655	0.524595	0.854898	0.128*	0.0971 (14)
H11H	0.114377	0.580442	0.944008	0.128*	0.0971 (14)
C12B	0.020 (3)	0.531 (5)	0.899 (6)	0.109 (11)	0.0971 (14)
H12J	0.007978	0.569567	0.850948	0.163*	0.0971 (14)
H12K	0.005171	0.483275	0.871238	0.163*	0.0971 (14)
H12L	−0.007204	0.539560	0.961048	0.163*	0.0971 (14)
C13B	0.245 (3)	0.404 (4)	1.054 (4)	0.107 (5)	0.0971 (14)
H13G	0.275633	0.434715	1.097827	0.128*	0.0971 (14)
H13H	0.277073	0.366752	1.022537	0.128*	0.0971 (14)
C14B	0.187 (4)	0.365 (5)	1.116 (7)	0.112 (5)	0.0971 (14)
H14G	0.146656	0.347873	1.073754	0.135*	0.0971 (14)
H14H	0.167172	0.398959	1.167225	0.135*	0.0971 (14)
C15B	0.227 (6)	0.297 (4)	1.169 (6)	0.115 (10)	0.0971 (14)
H15J	0.191748	0.270452	1.210549	0.173*	0.0971 (14)
H15K	0.246830	0.264050	1.118237	0.173*	0.0971 (14)
H15L	0.267251	0.314898	1.211273	0.173*	0.0971 (14)
O6B	0.2072 (19)	0.383 (3)	0.472 (3)	0.065 (6)	0.0971 (14)
H6C	0.178 (4)	0.368 (3)	0.428 (7)	0.097*	0.0971 (14)
H6D	0.247 (3)	0.393 (4)	0.443 (7)	0.097*	0.0971 (14)
O7	0.2905 (12)	0.4390 (12)	0.371 (2)	0.071 (4)	0.549 (3)
C16	0.3400 (8)	0.4850 (8)	0.3568 (10)	0.086 (3)	0.549 (3)
H16	0.381901	0.482146	0.398582	0.104*	0.549 (3)
N2	0.3391 (8)	0.5377 (9)	0.2882 (13)	0.082 (3)	0.549 (3)
C17	0.4050 (11)	0.5828 (11)	0.2738 (15)	0.120 (5)	0.549 (3)
H17A	0.446742	0.551047	0.258363	0.179*	0.549 (3)
H17B	0.415204	0.610433	0.334803	0.179*	0.549 (3)
H17C	0.396990	0.616954	0.218513	0.179*	0.549 (3)
C18	0.2743 (11)	0.5436 (12)	0.2230 (15)	0.122 (5)	0.549 (3)
H18A	0.272351	0.592718	0.193768	0.183*	0.549 (3)
H18B	0.229911	0.534844	0.262163	0.183*	0.549 (3)
H18C	0.277733	0.507079	0.169677	0.183*	0.549 (3)
O7B	0.338 (2)	0.417 (2)	0.366 (4)	0.086 (7)	0.0971 (14)
C16B	0.293 (3)	0.467 (3)	0.352 (7)	0.088 (5)	0.0971 (14)
H16B	0.242889	0.459512	0.370384	0.105*	0.0971 (14)
N2B	0.313 (3)	0.532 (2)	0.311 (5)	0.089 (5)	0.0971 (14)
C17B	0.255 (4)	0.588 (3)	0.313 (7)	0.097 (8)	0.0971 (14)
H17D	0.273533	0.633174	0.283004	0.145*	0.0971 (14)
H17E	0.212737	0.570271	0.276391	0.145*	0.0971 (14)
H17F	0.241486	0.597465	0.382769	0.145*	0.0971 (14)
C18B	0.392 (3)	0.546 (4)	0.323 (7)	0.095 (7)	0.0971 (14)
H18D	0.404240	0.593394	0.291920	0.142*	0.0971 (14)

H18E	0.404357	0.547736	0.393683	0.142*	0.0971 (14)
H18F	0.420059	0.507229	0.290140	0.142*	0.0971 (14)
O7C	0.296 (2)	0.441 (2)	0.386 (4)	0.077 (6)	0.354 (3)
C16C	0.2949 (12)	0.4984 (12)	0.3350 (17)	0.087 (4)	0.354 (3)
H16C	0.250026	0.522809	0.321376	0.104*	0.354 (3)
N2C	0.3591 (11)	0.5251 (12)	0.299 (2)	0.080 (4)	0.354 (3)
C17C	0.3557 (17)	0.5992 (13)	0.257 (2)	0.112 (6)	0.354 (3)
H17G	0.404347	0.613588	0.233349	0.168*	0.354 (3)
H17H	0.321143	0.599778	0.201064	0.168*	0.354 (3)
H17I	0.339192	0.633706	0.308216	0.168*	0.354 (3)
C18C	0.4231 (14)	0.4784 (15)	0.303 (2)	0.118 (6)	0.354 (3)
H18G	0.465149	0.504563	0.274964	0.176*	0.354 (3)
H18H	0.433399	0.465172	0.372309	0.176*	0.354 (3)
H18I	0.413916	0.433936	0.264112	0.176*	0.354 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Dy1	0.0591 (2)	0.0591 (2)	0.0508 (3)	0.000	0.000	0.000
Na1	0.0498 (10)	0.0498 (10)	0.058 (2)	0.000	0.000	0.000
Mn1	0.0453 (4)	0.0447 (4)	0.0641 (5)	0.0015 (2)	0.0072 (3)	0.0063 (3)
O1	0.0445 (15)	0.0446 (15)	0.0580 (19)	0.0001 (12)	0.0063 (13)	0.0018 (13)
O2	0.0482 (16)	0.0458 (17)	0.075 (2)	0.0004 (13)	0.0103 (16)	0.0038 (16)
O3	0.0549 (19)	0.0484 (18)	0.102 (3)	−0.0038 (15)	0.022 (2)	−0.0030 (19)
N1	0.049 (2)	0.0442 (18)	0.057 (2)	−0.0025 (15)	0.009 (3)	−0.0048 (17)
C1	0.051 (2)	0.0420 (19)	0.058 (2)	−0.0031 (16)	0.0111 (18)	−0.0014 (17)
C2	0.055 (2)	0.046 (2)	0.068 (3)	−0.0066 (18)	0.005 (2)	−0.006 (2)
C3	0.057 (2)	0.048 (2)	0.085 (3)	−0.003 (2)	0.007 (2)	0.001 (2)
C4	0.062 (3)	0.045 (2)	0.101 (4)	−0.004 (2)	0.011 (3)	0.001 (3)
C5	0.066 (3)	0.051 (3)	0.095 (4)	−0.011 (2)	0.010 (3)	0.000 (3)
C6	0.059 (3)	0.050 (2)	0.090 (4)	−0.011 (2)	0.018 (3)	−0.004 (3)
C7	0.056 (2)	0.044 (2)	0.074 (3)	−0.0055 (18)	0.016 (2)	−0.005 (2)
O4	0.086 (3)	0.076 (3)	0.060 (2)	0.006 (2)	0.019 (2)	−0.006 (2)
O5	0.087 (3)	0.076 (3)	0.069 (3)	0.003 (2)	0.021 (2)	−0.002 (2)
C8	0.100 (4)	0.095 (4)	0.067 (3)	0.010 (4)	0.026 (3)	−0.015 (3)
C9	0.117 (6)	0.113 (6)	0.083 (5)	0.015 (6)	0.024 (5)	−0.012 (5)
C10	0.115 (7)	0.121 (7)	0.084 (6)	0.015 (6)	0.029 (6)	−0.011 (6)
C11	0.121 (8)	0.123 (8)	0.093 (7)	0.006 (8)	0.026 (7)	−0.010 (8)
C12	0.140 (15)	0.155 (16)	0.107 (14)	0.016 (15)	0.038 (13)	−0.003 (14)
C13	0.121 (6)	0.121 (6)	0.087 (6)	0.011 (6)	0.019 (6)	−0.013 (6)
C14	0.132 (8)	0.128 (8)	0.095 (7)	0.008 (8)	0.023 (7)	−0.015 (7)
C15	0.143 (13)	0.146 (13)	0.088 (11)	−0.007 (12)	0.018 (12)	−0.011 (12)
O6	0.057 (2)	0.064 (2)	0.063 (2)	−0.0103 (17)	−0.0029 (18)	0.0144 (19)
C9C	0.114 (6)	0.110 (6)	0.080 (5)	0.017 (5)	0.025 (5)	−0.013 (5)
C10C	0.119 (7)	0.122 (7)	0.086 (6)	0.013 (7)	0.026 (6)	−0.011 (6)
C11C	0.109 (8)	0.125 (8)	0.098 (7)	0.019 (7)	0.027 (7)	−0.013 (7)
C12C	0.111 (14)	0.145 (15)	0.110 (13)	−0.013 (14)	0.036 (13)	−0.011 (13)
C13C	0.123 (7)	0.118 (7)	0.086 (6)	0.013 (6)	0.024 (6)	−0.021 (6)

C14C	0.127 (7)	0.121 (7)	0.096 (7)	0.007 (7)	0.022 (6)	−0.014 (6)
C15C	0.137 (11)	0.126 (11)	0.098 (10)	0.021 (10)	0.023 (10)	−0.014 (10)
C9D	0.114 (6)	0.113 (6)	0.082 (5)	0.019 (6)	0.025 (5)	−0.009 (5)
C10D	0.115 (7)	0.118 (7)	0.083 (7)	0.012 (7)	0.031 (6)	−0.013 (7)
C11D	0.127 (9)	0.131 (9)	0.096 (8)	0.010 (9)	0.025 (9)	−0.004 (8)
C12D	0.136 (16)	0.130 (16)	0.130 (15)	0.016 (15)	0.020 (15)	−0.022 (15)
C13D	0.120 (6)	0.123 (7)	0.082 (6)	0.011 (6)	0.025 (6)	−0.011 (6)
C14D	0.127 (7)	0.121 (7)	0.091 (7)	0.014 (7)	0.022 (6)	−0.012 (6)
C15D	0.140 (13)	0.131 (13)	0.118 (12)	0.018 (12)	0.035 (11)	−0.004 (12)
Mn1B	0.036 (3)	0.038 (3)	0.066 (4)	0.001 (2)	−0.001 (2)	−0.002 (2)
O1B	0.042 (7)	0.032 (7)	0.075 (8)	0.003 (6)	0.009 (7)	0.007 (7)
O2B	0.048 (6)	0.035 (6)	0.073 (7)	−0.002 (6)	0.006 (6)	0.001 (6)
O3B	0.054 (7)	0.047 (7)	0.074 (7)	−0.004 (7)	0.009 (7)	−0.005 (7)
N1B	0.052 (7)	0.040 (7)	0.070 (7)	−0.006 (6)	0.010 (7)	0.000 (7)
C1B	0.055 (6)	0.037 (6)	0.072 (6)	−0.004 (6)	0.012 (6)	0.000 (6)
C2B	0.052 (5)	0.040 (5)	0.076 (5)	−0.006 (5)	0.012 (5)	−0.001 (5)
C3B	0.056 (6)	0.043 (6)	0.083 (6)	−0.004 (6)	0.012 (6)	−0.002 (6)
C4B	0.057 (7)	0.045 (7)	0.084 (7)	−0.007 (7)	0.011 (7)	−0.002 (7)
C5B	0.058 (7)	0.046 (7)	0.085 (7)	−0.012 (7)	0.015 (7)	0.002 (7)
C6B	0.055 (7)	0.043 (7)	0.081 (7)	−0.007 (7)	0.013 (7)	−0.001 (7)
C7B	0.055 (6)	0.042 (6)	0.078 (6)	−0.007 (6)	0.010 (6)	−0.003 (6)
O4B	0.104 (10)	0.094 (9)	0.071 (9)	0.012 (9)	0.012 (9)	−0.019 (9)
O5B	0.076 (12)	0.081 (12)	0.068 (12)	0.001 (11)	−0.006 (11)	−0.009 (11)
C8B	0.103 (8)	0.097 (8)	0.072 (8)	0.011 (8)	0.019 (8)	−0.015 (8)
C9B	0.118 (7)	0.112 (7)	0.083 (7)	0.012 (7)	0.020 (7)	−0.016 (7)
C10B	0.118 (7)	0.115 (7)	0.084 (6)	0.014 (6)	0.023 (6)	−0.015 (6)
C11B	0.120 (9)	0.115 (10)	0.086 (9)	0.013 (9)	0.023 (9)	−0.015 (9)
C12B	0.12 (2)	0.12 (2)	0.084 (19)	0.02 (2)	0.038 (19)	−0.017 (19)
C13B	0.120 (8)	0.116 (8)	0.085 (8)	0.011 (8)	0.019 (8)	−0.014 (8)
C14B	0.126 (9)	0.122 (9)	0.088 (8)	0.011 (8)	0.023 (8)	−0.015 (8)
C15B	0.132 (19)	0.131 (19)	0.082 (18)	−0.005 (19)	0.019 (18)	−0.010 (18)
O6B	0.058 (11)	0.070 (12)	0.066 (12)	−0.008 (11)	0.004 (11)	0.012 (11)
O7	0.071 (6)	0.071 (6)	0.070 (9)	−0.005 (5)	−0.001 (5)	0.021 (6)
C16	0.098 (7)	0.089 (6)	0.072 (5)	−0.030 (6)	−0.022 (5)	0.010 (5)
N2	0.114 (7)	0.071 (5)	0.062 (6)	−0.032 (5)	−0.007 (6)	0.012 (4)
C17	0.133 (11)	0.110 (9)	0.116 (10)	−0.054 (9)	−0.015 (9)	0.025 (8)
C18	0.139 (11)	0.117 (10)	0.111 (10)	0.006 (9)	0.001 (9)	0.014 (8)
O7B	0.100 (13)	0.080 (12)	0.077 (12)	−0.026 (12)	0.005 (12)	0.019 (12)
C16B	0.106 (9)	0.083 (9)	0.074 (9)	−0.026 (9)	−0.002 (9)	0.013 (9)
N2B	0.108 (9)	0.083 (8)	0.076 (9)	−0.026 (8)	−0.003 (8)	0.012 (8)
C17B	0.118 (15)	0.094 (14)	0.078 (14)	−0.025 (14)	−0.003 (14)	0.007 (13)
C18B	0.113 (13)	0.089 (12)	0.082 (12)	−0.027 (12)	0.008 (12)	0.018 (12)
O7C	0.082 (10)	0.075 (9)	0.075 (12)	−0.007 (8)	0.001 (9)	0.018 (8)
C16C	0.110 (8)	0.075 (7)	0.076 (7)	−0.016 (7)	0.012 (7)	0.017 (6)
N2C	0.102 (8)	0.076 (7)	0.062 (7)	−0.033 (7)	−0.004 (7)	0.004 (6)
C17C	0.136 (12)	0.095 (11)	0.105 (11)	−0.018 (11)	0.012 (11)	0.014 (10)
C18C	0.126 (11)	0.116 (11)	0.110 (11)	−0.026 (10)	−0.001 (10)	0.017 (10)

Geometric parameters (Å, º)

Dy1—O4B	2.24 (4)	C10D—C11D	1.43 (2)
Dy1—O4^{i}	2.277 (5)	C10D—H10E	0.9900
Dy1—O4^{ii}	2.277 (5)	C10D—H10F	0.9900
Dy1—O4^{iii}	2.277 (5)	C11D—C12D	1.53 (2)
Dy1—O4	2.277 (5)	C11D—H11E	0.9900
Dy1—O1	2.444 (3)	C11D—H11F	0.9900
Dy1—O1^{ii}	2.444 (3)	C12D—H12G	0.9800
Dy1—O1^{iii}	2.444 (3)	C12D—H12H	0.9800
Dy1—O1^{i}	2.444 (3)	C12D—H12I	0.9800
Dy1—O1B^{iii}	2.54 (3)	C13D—C14D	1.52 (2)
Dy1—O1B^{ii}	2.54 (3)	C13D—H13E	0.9900
Dy1—O1B^{i}	2.54 (3)	C13D—H13F	0.9900
Na1—O6	2.436 (5)	C14D—C15D	1.61 (2)
Na1—O6^{ii}	2.436 (5)	C14D—H14E	0.9900
Na1—O6^{i}	2.436 (5)	C14D—H14F	0.9900
Na1—O6^{iii}	2.436 (5)	C15D—H15G	0.9800
Na1—O1B^{i}	2.53 (3)	C15D—H15H	0.9800
Na1—O1B^{ii}	2.53 (3)	C15D—H15I	0.9800
Na1—O1B^{iii}	2.53 (3)	Mn1B—O3B^{i}	1.85 (3)
Na1—O1B	2.53 (3)	Mn1B—O1B	1.923 (17)
Na1—O6B^{ii}	2.64 (5)	Mn1B—N1B^{i}	1.95 (4)
Na1—O6B^{i}	2.64 (5)	Mn1B—O2B	1.955 (17)
Na1—O6B^{iii}	2.64 (5)	Mn1B—O6B^{i}	2.41 (4)
Na1—O6B	2.64 (5)	O1B—N1B	1.397 (18)
Mn1—O3^{ii}	1.840 (4)	O2B—C1B	1.275 (18)
Mn1—O1	1.914 (3)	O3B—C7B	1.349 (19)
Mn1—O2	1.939 (4)	N1B—C1B	1.317 (19)
Mn1—N1^{ii}	1.946 (5)	C1B—C2B	1.459 (17)
Mn1—O5	2.126 (5)	C2B—C3B	1.404 (19)
Mn1—O6	2.527 (5)	C2B—C7B	1.414 (19)
O1—N1	1.401 (5)	C3B—C4B	1.38 (2)
O2—C1	1.294 (6)	C3B—H3B	0.9500
O3—C7	1.335 (7)	C4B—C5B	1.37 (2)
N1—C1	1.319 (7)	C4B—H4B	0.9500
C1—C2	1.459 (7)	C5B—C6B	1.37 (2)
C2—C3	1.406 (8)	C5B—H5B	0.9500
C2—C7	1.415 (8)	C6B—C7B	1.412 (19)
C3—C4	1.384 (8)	C6B—H6B	0.9500
C3—H3	0.9500	O4B—C8B	1.29 (2)
C4—C5	1.373 (9)	O5B—C8B	1.23 (2)
C4—H4	0.9500	C8B—C9B	1.54 (2)
C5—C6	1.383 (9)	C9B—C13B	1.47 (2)
C5—H5	0.9500	C9B—C10B	1.52 (2)
C6—C7	1.402 (7)	C9B—H9B	1.0000
C6—H6	0.9500	C10B—C11B	1.44 (2)
O4—C8	1.274 (10)	C10B—H10G	0.9900

O5—C8	1.220 (10)	C10B—H10H	0.9900
C8—C9C	1.526 (16)	C11B—C12B	1.53 (2)
C8—C9	1.543 (16)	C11B—H11G	0.9900
C8—C9D	1.558 (16)	C11B—H11H	0.9900
C9—C13	1.452 (16)	C12B—H12J	0.9800
C9—C10	1.515 (15)	C12B—H12K	0.9800
C9—H9	1.0000	C12B—H12L	0.9800
C10—C11	1.470 (16)	C13B—C14B	1.52 (2)
C10—H10A	0.9900	C13B—H13G	0.9900
C10—H10B	0.9900	C13B—H13H	0.9900
C11—C12	1.530 (16)	C14B—C15B	1.61 (2)
C11—H11A	0.9900	C14B—H14G	0.9900
C11—H11B	0.9900	C14B—H14H	0.9900
C12—H12A	0.9800	C15B—H15J	0.9800
C12—H12B	0.9800	C15B—H15K	0.9800
C12—H12C	0.9800	C15B—H15L	0.9800
C13—C14	1.517 (17)	O6B—H6C	0.84 (2)
C13—H13A	0.9900	O6B—H6D	0.850 (18)
C13—H13B	0.9900	O7—C16	1.248 (18)
C14—C15	1.594 (17)	C16—N2	1.333 (15)
C14—H14A	0.9900	C16—H16	0.9500
C14—H14B	0.9900	N2—C17	1.472 (14)
C15—H15A	0.9800	N2—C18	1.475 (16)
C15—H15B	0.9800	C17—H17A	0.9800
C15—H15C	0.9800	C17—H17B	0.9800
O6—H6A	0.857 (17)	C17—H17C	0.9800
O6—H6E	0.832 (18)	C18—H18A	0.9800
C9C—C13C	1.44 (2)	C18—H18B	0.9800
C9C—C10C	1.50 (2)	C18—H18C	0.9800
C9C—H9C	1.0000	O7B—C16B	1.25 (2)
C10C—C11C	1.43 (2)	C16B—N2B	1.36 (2)
C10C—H10C	0.9900	C16B—H16B	0.9500
C10C—H10D	0.9900	N2B—C18B	1.47 (2)
C11C—C12C	1.53 (2)	N2B—C17B	1.47 (2)
C11C—H11C	0.9900	C17B—H17D	0.9800
C11C—H11D	0.9900	C17B—H17E	0.9800
C12C—H12D	0.9800	C17B—H17F	0.9800
C12C—H12E	0.9800	C18B—H18D	0.9800
C12C—H12F	0.9800	C18B—H18E	0.9800
C13C—C14C	1.53 (2)	C18B—H18F	0.9800
C13C—H13C	0.9900	O7C—C16C	1.25 (2)
C13C—H13D	0.9900	C16C—N2C	1.358 (18)
C14C—C15C	1.62 (2)	C16C—H16C	0.9500
C14C—H14C	0.9900	N2C—C18C	1.448 (18)
C14C—H14D	0.9900	N2C—C17C	1.469 (17)
C15C—H15D	0.9800	C17C—H17G	0.9800
C15C—H15E	0.9800	C17C—H17H	0.9800
C15C—H15F	0.9800	C17C—H17I	0.9800

C9D—C13D	1.47 (2)	C18C—H18G	0.9800
C9D—C10D	1.52 (2)	C18C—H18H	0.9800
C9D—H9D	1.0000	C18C—H18I	0.9800
O4B—Dy1—O4^{ii}	111.0 (14)	C10C—C9C—C8	108.9 (16)
O4^{i}—Dy1—O4^{ii}	125.9 (3)	C13C—C9C—H9C	90.3
O4B—Dy1—O4^{iii}	99.8 (12)	C10C—C9C—H9C	90.3
O4^{i}—Dy1—O4^{iii}	78.06 (11)	C8—C9C—H9C	90.3
O4^{ii}—Dy1—O4^{iii}	78.06 (11)	C11C—C10C—C9C	125 (2)
O4^{i}—Dy1—O4	78.06 (11)	C11C—C10C—H10C	106.1
O4^{ii}—Dy1—O4	78.06 (11)	C9C—C10C—H10C	106.1
O4^{iii}—Dy1—O4	125.9 (3)	C11C—C10C—H10D	106.1
O4^{i}—Dy1—O1	78.69 (16)	C9C—C10C—H10D	106.1
O4^{ii}—Dy1—O1	142.57 (15)	H10C—C10C—H10D	106.3
O4^{iii}—Dy1—O1	138.77 (16)	C10C—C11C—C12C	123 (2)
O4—Dy1—O1	81.14 (15)	C10C—C11C—H11C	106.5
O4B—Dy1—O1^{ii}	116.6 (12)	C12C—C11C—H11C	106.5
O4^{i}—Dy1—O1^{ii}	138.77 (16)	C10C—C11C—H11D	106.5
O4^{ii}—Dy1—O1^{ii}	81.14 (15)	C12C—C11C—H11D	106.5
O4^{iii}—Dy1—O1^{ii}	142.57 (15)	H11C—C11C—H11D	106.5
O4—Dy1—O1^{ii}	78.69 (16)	C11C—C12C—H12D	109.5
O1—Dy1—O1^{ii}	64.39 (9)	C11C—C12C—H12E	109.5
O4B—Dy1—O1^{iii}	170.3 (14)	H12D—C12C—H12E	109.5
O4^{i}—Dy1—O1^{iii}	142.57 (15)	C11C—C12C—H12F	109.5
O4^{ii}—Dy1—O1^{iii}	78.69 (16)	H12D—C12C—H12F	109.5
O4^{iii}—Dy1—O1^{iii}	81.14 (15)	H12E—C12C—H12F	109.5
O4—Dy1—O1^{iii}	138.77 (16)	C9C—C13C—C14C	107.4 (19)
O1—Dy1—O1^{iii}	97.80 (17)	C9C—C13C—H13C	110.2
O1^{ii}—Dy1—O1^{iii}	64.40 (9)	C14C—C13C—H13C	110.2
O4B—Dy1—O1^{i}	106.2 (15)	C9C—C13C—H13D	110.2
O4^{i}—Dy1—O1^{i}	81.14 (15)	C14C—C13C—H13D	110.2
O4^{ii}—Dy1—O1^{i}	138.77 (16)	H13C—C13C—H13D	108.5
O4^{iii}—Dy1—O1^{i}	78.69 (16)	C13C—C14C—C15C	101.0 (19)
O4—Dy1—O1^{i}	142.57 (15)	C13C—C14C—H14C	111.6
O1—Dy1—O1^{i}	64.40 (9)	C15C—C14C—H14C	111.6
O1^{ii}—Dy1—O1^{i}	97.80 (17)	C13C—C14C—H14D	111.6
O1^{iii}—Dy1—O1^{i}	64.40 (9)	C15C—C14C—H14D	111.6
O4B—Dy1—O1B^{iii}	144.4 (13)	H14C—C14C—H14D	109.4
O4^{i}—Dy1—O1B^{iii}	160.8 (7)	C14C—C15C—H15D	109.5
O4^{ii}—Dy1—O1B^{iii}	72.2 (7)	C14C—C15C—H15E	109.5
O4^{iii}—Dy1—O1B^{iii}	115.1 (5)	H15D—C15C—H15E	109.5
O4—Dy1—O1B^{iii}	102.4 (5)	C14C—C15C—H15F	109.5
O1—Dy1—O1B^{iii}	82.4 (7)	H15D—C15C—H15F	109.5
O1^{ii}—Dy1—O1B^{iii}	27.9 (4)	H15E—C15C—H15F	109.5
O1^{iii}—Dy1—O1B^{iii}	37.6 (4)	C13D—C9D—C10D	122 (2)
O1^{i}—Dy1—O1B^{iii}	87.7 (7)	C13D—C9D—C8	111.2 (19)
O4B—Dy1—O1B^{ii}	88.6 (13)	C10D—C9D—C8	118.0 (18)
O4^{i}—Dy1—O1B^{ii}	102.4 (5)	C13D—C9D—H9D	99.9

O4^{ii}—Dy1—O1B^{ii}	115.1 (5)	C10D—C9D—H9D	99.9
O4^{iii}—Dy1—O1B^{ii}	160.8 (7)	C8—C9D—H9D	99.9
O4—Dy1—O1B^{ii}	72.2 (7)	C11D—C10D—C9D	122 (2)
O1—Dy1—O1B^{ii}	27.9 (4)	C11D—C10D—H10E	106.9
O1^{ii}—Dy1—O1B^{ii}	37.6 (4)	C9D—C10D—H10E	106.9
O1^{iii}—Dy1—O1B^{ii}	87.7 (7)	C11D—C10D—H10F	106.9
O1^{i}—Dy1—O1B^{ii}	82.4 (7)	C9D—C10D—H10F	106.9
O1B^{iii}—Dy1—O1B^{ii}	60.4 (9)	H10E—C10D—H10F	106.7
O4B—Dy1—O1B^{i}	143.3 (15)	C10D—C11D—C12D	122 (2)
O4^{i}—Dy1—O1B^{i}	115.1 (5)	C10D—C11D—H11E	106.8
O4^{ii}—Dy1—O1B^{i}	102.4 (5)	C12D—C11D—H11E	106.8
O4^{iii}—Dy1—O1B^{i}	72.2 (7)	C10D—C11D—H11F	106.8
O4—Dy1—O1B^{i}	160.8 (7)	C12D—C11D—H11F	106.8
O1—Dy1—O1B^{i}	87.7 (7)	H11E—C11D—H11F	106.6
O1^{ii}—Dy1—O1B^{i}	82.4 (7)	C11D—C12D—H12G	109.5
O1^{iii}—Dy1—O1B^{i}	27.9 (4)	C11D—C12D—H12H	109.5
O1^{i}—Dy1—O1B^{i}	37.6 (4)	H12G—C12D—H12H	109.5
O1B^{iii}—Dy1—O1B^{i}	60.4 (9)	C11D—C12D—H12I	109.5
O1B^{ii}—Dy1—O1B^{i}	90.7 (15)	H12G—C12D—H12I	109.5
O6—Na1—O6^{ii}	85.06 (8)	H12H—C12D—H12I	109.5
O6—Na1—O6^{i}	85.06 (8)	C9D—C13D—C14D	111 (2)
O6^{ii}—Na1—O6^{i}	145.9 (3)	C9D—C13D—H13E	109.5
O6—Na1—O6^{iii}	145.9 (3)	C14D—C13D—H13E	109.5
O6^{ii}—Na1—O6^{iii}	85.06 (8)	C9D—C13D—H13F	109.5
O6^{i}—Na1—O6^{iii}	85.06 (8)	C14D—C13D—H13F	109.5
O6—Na1—O1B^{i}	150.7 (6)	H13E—C13D—H13F	108.1
O6^{ii}—Na1—O1B^{i}	110.9 (4)	C13D—C14D—C15D	103 (2)
O6^{i}—Na1—O1B^{i}	93.1 (5)	C13D—C14D—H14E	111.2
O6^{iii}—Na1—O1B^{i}	62.5 (6)	C15D—C14D—H14E	111.2
O6—Na1—O1B^{ii}	62.5 (6)	C13D—C14D—H14F	111.2
O6^{ii}—Na1—O1B^{ii}	93.1 (5)	C15D—C14D—H14F	111.2
O6^{i}—Na1—O1B^{ii}	110.9 (4)	H14E—C14D—H14F	109.2
O6^{iii}—Na1—O1B^{ii}	150.7 (6)	C14D—C15D—H15G	109.5
O1B^{i}—Na1—O1B^{ii}	91.4 (12)	C14D—C15D—H15H	109.5
O6—Na1—O1B^{iii}	110.9 (4)	H15G—C15D—H15H	109.5
O6^{ii}—Na1—O1B^{iii}	62.5 (6)	C14D—C15D—H15I	109.5
O6^{i}—Na1—O1B^{iii}	150.7 (6)	H15G—C15D—H15I	109.5
O6^{iii}—Na1—O1B^{iii}	93.1 (5)	H15H—C15D—H15I	109.5
O1B^{i}—Na1—O1B^{iii}	60.8 (7)	O3B^{i}—Mn1B—O1B	174.4 (18)
O1B^{ii}—Na1—O1B^{iii}	60.8 (7)	O3B^{i}—Mn1B—O2B	98.1 (10)
O1B^{i}—Na1—O1B	60.8 (7)	O1B—Mn1B—O2B	81.2 (8)
O1B^{ii}—Na1—O1B	60.8 (7)	N1B^{i}—Mn1B—O2B	166 (3)
O1B^{iii}—Na1—O1B	91.4 (12)	O3B^{i}—Mn1B—O6B^{i}	95.9 (17)
O6—Na1—O6B^{ii}	71.0 (7)	O1B—Mn1B—O6B^{i}	78.5 (16)
O6^{ii}—Na1—O6B^{ii}	15.2 (7)	N1B^{i}—Mn1B—O6B^{i}	85 (4)
O6^{i}—Na1—O6B^{ii}	144.3 (8)	O2B—Mn1B—O6B^{i}	84.1 (14)
O6^{iii}—Na1—O6B^{ii}	100.0 (8)	O3B^{i}—Mn1B—Na1	134.2 (11)
O1B^{i}—Na1—O6B^{ii}	120.7 (9)	O1B—Mn1B—Na1	41.2 (10)

O1B^{ii}—Na1—O6B^{ii}	81.5 (10)	N1B^{i}—Mn1B—Na1	65 (3)
O1B^{iii}—Na1—O6B^{ii}	64.8 (10)	O2B—Mn1B—Na1	101.6 (9)
O1B—Na1—O6B^{ii}	142.0 (10)	O6B^{i}—Mn1B—Na1	46.4 (12)
O6—Na1—O6B^{i}	100.0 (8)	N1B—O1B—Mn1B	112.6 (15)
O6^{ii}—Na1—O6B^{i}	144.3 (8)	N1B—O1B—Na1	112 (6)
O6^{i}—Na1—O6B^{i}	15.2 (7)	Mn1B—O1B—Na1	108.7 (13)
O6^{iii}—Na1—O6B^{i}	71.0 (7)	N1B—O1B—Dy1	117 (4)
O1B^{i}—Na1—O6B^{i}	81.5 (10)	Mn1B—O1B—Dy1	115.3 (12)
O1B^{ii}—Na1—O6B^{i}	120.7 (9)	Na1—O1B—Dy1	89.0 (6)
O1B^{iii}—Na1—O6B^{i}	142.0 (10)	C1B—O2B—Mn1B	112.2 (14)
O1B—Na1—O6B^{i}	64.8 (10)	C7B—O3B—Mn1B^{ii}	130 (2)
O6B^{ii}—Na1—O6B^{i}	149.9 (18)	C1B—N1B—O1B	113 (2)
O6—Na1—O6B^{iii}	144.3 (8)	C1B—N1B—Mn1B^{ii}	131 (3)
O6^{ii}—Na1—O6B^{iii}	71.0 (7)	O1B—N1B—Mn1B^{ii}	114 (2)
O6^{i}—Na1—O6B^{iii}	100.0 (8)	O2B—C1B—N1B	121.0 (18)
O6^{iii}—Na1—O6B^{iii}	15.2 (7)	O2B—C1B—C2B	119.9 (18)
O1B^{i}—Na1—O6B^{iii}	64.8 (10)	N1B—C1B—C2B	118.9 (19)
O1B^{ii}—Na1—O6B^{iii}	142.0 (10)	C3B—C2B—C7B	121.2 (19)
O1B^{iii}—Na1—O6B^{iii}	81.5 (10)	C3B—C2B—C1B	114.3 (19)
O1B—Na1—O6B^{iii}	120.7 (9)	C7B—C2B—C1B	124.5 (19)
O6B^{ii}—Na1—O6B^{iii}	86.1 (4)	C4B—C3B—C2B	121 (2)
O6B^{i}—Na1—O6B^{iii}	86.1 (4)	C4B—C3B—H3B	119.3
O1B^{i}—Na1—O6B	142.0 (10)	C2B—C3B—H3B	119.3
O1B^{ii}—Na1—O6B	64.8 (10)	C5B—C4B—C3B	117 (3)
O1B^{iii}—Na1—O6B	120.7 (9)	C5B—C4B—H4B	121.6
O1B—Na1—O6B	81.5 (10)	C3B—C4B—H4B	121.6
O6B^{ii}—Na1—O6B	86.1 (4)	C4B—C5B—C6B	124 (3)
O6B^{i}—Na1—O6B	86.1 (4)	C4B—C5B—H5B	118.0
O6B^{iii}—Na1—O6B	149.9 (18)	C6B—C5B—H5B	118.0
O3^{ii}—Mn1—O1	168.5 (2)	C5B—C6B—C7B	120 (2)
O3^{ii}—Mn1—O2	97.45 (17)	C5B—C6B—H6B	119.8
O1—Mn1—O2	81.70 (15)	C7B—C6B—H6B	119.8
O3^{ii}—Mn1—N1^{ii}	90.57 (19)	O3B—C7B—C6B	120 (2)
O1—Mn1—N1^{ii}	88.75 (16)	O3B—C7B—C2B	124 (2)
O2—Mn1—N1^{ii}	168.3 (3)	C6B—C7B—C2B	116.1 (19)
O3^{ii}—Mn1—O5	94.6 (2)	C8B—O4B—Dy1	133 (3)
O1—Mn1—O5	96.9 (2)	O5B—C8B—O4B	124 (3)
O2—Mn1—O5	94.0 (2)	O5B—C8B—C9B	128 (3)
N1^{ii}—Mn1—O5	93.8 (4)	O4B—C8B—C9B	108 (2)
O3^{ii}—Mn1—O6	89.8 (2)	C13B—C9B—C10B	121 (3)
O1—Mn1—O6	78.68 (15)	C13B—C9B—C8B	113 (3)
O2—Mn1—O6	86.28 (16)	C10B—C9B—C8B	106 (3)
N1^{ii}—Mn1—O6	85.3 (4)	C13B—C9B—H9B	105.3
O5—Mn1—O6	175.52 (19)	C10B—C9B—H9B	105.3
O3^{ii}—Mn1—Na1	124.15 (18)	C8B—C9B—H9B	105.3
O1—Mn1—Na1	45.80 (12)	C11B—C10B—C9B	124 (3)
O2—Mn1—Na1	103.48 (11)	C11B—C10B—H10G	106.3
N1^{ii}—Mn1—Na1	64.9 (3)	C9B—C10B—H10G	106.3

O5—Mn1—Na1	133.91 (17)	C11B—C10B—H10H	106.2
O6—Mn1—Na1	41.91 (11)	C9B—C10B—H10H	106.2
N1—O1—Mn1	112.7 (3)	H10G—C10B—H10H	106.4
N1—O1—Dy1	121.3 (4)	C10B—C11B—C12B	122 (3)
Mn1—O1—Dy1	119.88 (15)	C10B—C11B—H11G	106.8
N1—O1—Na1	104.4 (4)	C12B—C11B—H11G	106.8
Mn1—O1—Na1	103.43 (15)	C10B—C11B—H11H	106.8
Dy1—O1—Na1	87.70 (12)	C12B—C11B—H11H	106.8
C1—O2—Mn1	112.1 (3)	H11G—C11B—H11H	106.6
C7—O3—Mn1^{i}	129.8 (3)	C11B—C12B—H12J	109.5
C1—N1—O1	112.6 (5)	C11B—C12B—H12K	109.5
C1—N1—Mn1^{i}	130.9 (4)	H12J—C12B—H12K	109.5
O1—N1—Mn1^{i}	116.1 (3)	C11B—C12B—H12L	109.5
O2—C1—N1	120.4 (4)	H12J—C12B—H12L	109.5
O2—C1—C2	120.0 (4)	H12K—C12B—H12L	109.5
N1—C1—C2	119.6 (5)	C9B—C13B—C14B	112 (3)
C3—C2—C7	118.8 (5)	C9B—C13B—H13G	109.2
C3—C2—C1	118.0 (5)	C14B—C13B—H13G	109.2
C7—C2—C1	123.1 (5)	C9B—C13B—H13H	109.2
C4—C3—C2	121.4 (6)	C14B—C13B—H13H	109.2
C4—C3—H3	119.3	H13G—C13B—H13H	107.9
C2—C3—H3	119.3	C13B—C14B—C15B	107 (3)
C5—C4—C3	119.3 (6)	C13B—C14B—H14G	110.4
C5—C4—H4	120.3	C15B—C14B—H14G	110.4
C3—C4—H4	120.3	C13B—C14B—H14H	110.4
C4—C5—C6	120.9 (6)	C15B—C14B—H14H	110.4
C4—C5—H5	119.5	H14G—C14B—H14H	108.6
C6—C5—H5	119.5	C14B—C15B—H15J	109.5
C5—C6—C7	121.0 (6)	C14B—C15B—H15K	109.5
C5—C6—H6	119.5	H15J—C15B—H15K	109.5
C7—C6—H6	119.5	C14B—C15B—H15L	109.5
O3—C7—C6	117.9 (5)	H15J—C15B—H15L	109.5
O3—C7—C2	123.5 (5)	H15K—C15B—H15L	109.5
C6—C7—C2	118.5 (5)	Mn1B^{ii}—O6B—Na1	92.1 (13)
C8—O4—Dy1	144.7 (5)	Mn1B^{ii}—O6B—H6C	137 (7)
C8—O5—Mn1	124.0 (5)	Na1—O6B—H6C	95 (6)
O5—C8—O4	127.1 (7)	Mn1B^{ii}—O6B—H6D	115 (7)
O5—C8—C9C	110.5 (16)	Na1—O6B—H6D	95 (6)
O4—C8—C9C	122.3 (16)	H6C—O6B—H6D	107 (3)
O5—C8—C9	124.4 (14)	O7—C16—N2	125.5 (16)
O4—C8—C9	108.3 (14)	O7—C16—H16	117.2
O5—C8—C9D	117.9 (15)	N2—C16—H16	117.2
O4—C8—C9D	113.9 (16)	C16—N2—C17	119.0 (13)
C13—C9—C10	122.4 (16)	C16—N2—C18	118.2 (12)
C13—C9—C8	115.7 (17)	C17—N2—C18	122.5 (13)
C10—C9—C8	106.3 (18)	N2—C17—H17A	109.5
C13—C9—H9	103.3	N2—C17—H17B	109.5
C10—C9—H9	103.3	H17A—C17—H17B	109.5

C8—C9—H9	103.3	N2—C17—H17C	109.5
C11—C10—C9	116.6 (15)	H17A—C17—H17C	109.5
C11—C10—H10A	108.1	H17B—C17—H17C	109.5
C9—C10—H10A	108.1	N2—C18—H18A	109.5
C11—C10—H10B	108.1	N2—C18—H18B	109.5
C9—C10—H10B	108.1	H18A—C18—H18B	109.5
H10A—C10—H10B	107.3	N2—C18—H18C	109.5
C10—C11—C12	118.1 (17)	H18A—C18—H18C	109.5
C10—C11—H11A	107.8	H18B—C18—H18C	109.5
C12—C11—H11A	107.8	O7B—C16B—N2B	120 (3)
C10—C11—H11B	107.8	O7B—C16B—H16B	119.8
C12—C11—H11B	107.8	N2B—C16B—H16B	119.8
H11A—C11—H11B	107.1	C16B—N2B—C18B	113 (3)
C11—C12—H12A	109.5	C16B—N2B—C17B	113 (3)
C11—C12—H12B	109.5	C18B—N2B—C17B	125 (3)
H12A—C12—H12B	109.5	N2B—C17B—H17D	109.5
C11—C12—H12C	109.5	N2B—C17B—H17E	109.5
H12A—C12—H12C	109.5	H17D—C17B—H17E	109.5
H12B—C12—H12C	109.5	N2B—C17B—H17F	109.5
C9—C13—C14	118.5 (17)	H17D—C17B—H17F	109.5
C9—C13—H13A	107.7	H17E—C17B—H17F	109.5
C14—C13—H13A	107.7	N2B—C18B—H18D	109.5
C9—C13—H13B	107.7	N2B—C18B—H18E	109.5
C14—C13—H13B	107.7	H18D—C18B—H18E	109.5
H13A—C13—H13B	107.1	N2B—C18B—H18F	109.5
C13—C14—C15	110.9 (15)	H18D—C18B—H18F	109.5
C13—C14—H14A	109.5	H18E—C18B—H18F	109.5
C15—C14—H14A	109.5	O7C—C16C—N2C	119 (2)
C13—C14—H14B	109.5	O7C—C16C—H16C	120.5
C15—C14—H14B	109.5	N2C—C16C—H16C	120.5
H14A—C14—H14B	108.1	C16C—N2C—C18C	118.3 (16)
C14—C15—H15A	109.5	C16C—N2C—C17C	115.5 (18)
C14—C15—H15B	109.5	C18C—N2C—C17C	126.2 (19)
H15A—C15—H15B	109.5	N2C—C17C—H17G	109.5
C14—C15—H15C	109.5	N2C—C17C—H17H	109.5
H15A—C15—H15C	109.5	H17G—C17C—H17H	109.5
H15B—C15—H15C	109.5	N2C—C17C—H17I	109.5
Na1—O6—Mn1	94.24 (17)	H17G—C17C—H17I	109.5
Na1—O6—H6A	109 (4)	H17H—C17C—H17I	109.5
Mn1—O6—H6A	124 (6)	N2C—C18C—H18G	109.5
Na1—O6—H6E	110 (4)	N2C—C18C—H18H	109.5
Mn1—O6—H6E	112 (5)	H18G—C18C—H18H	109.5
H6A—O6—H6E	107 (3)	N2C—C18C—H18I	109.5
C13C—C9C—C10C	135.8 (18)	H18G—C18C—H18I	109.5
C13C—C9C—C8	115.4 (18)	H18H—C18C—H18I	109.5
Mn1—O1—N1—C1	6.0 (10)	O5—C8—C9D—C13D	−153.5 (19)
Dy1—O1—N1—C1	−146.3 (6)	O4—C8—C9D—C13D	37 (3)

Na1—O1—N1—C1	117.5 (7)	O5—C8—C9D—C10D	58 (3)
Mn1—O1—N1—Mn1^{i}	−167.2 (5)	O4—C8—C9D—C10D	−111 (2)
Dy1—O1—N1—Mn1^{i}	40.4 (9)	C13D—C9D—C10D—C11D	−70 (4)
Na1—O1—N1—Mn1^{i}	−55.7 (8)	C8—C9D—C10D—C11D	75 (4)
Mn1—O2—C1—N1	−3.1 (9)	C9D—C10D—C11D—C12D	170 (3)
Mn1—O2—C1—C2	175.1 (4)	C10D—C9D—C13D—C14D	−141 (3)
O1—N1—C1—O2	−1.9 (12)	C8—C9D—C13D—C14D	72 (3)
Mn1^{i}—N1—C1—O2	170.1 (7)	C9D—C13D—C14D—C15D	89 (3)
O1—N1—C1—C2	179.9 (6)	Mn1B—O1B—N1B—C1B	5 (12)
Mn1^{i}—N1—C1—C2	−8.2 (14)	Na1—O1B—N1B—C1B	−117 (9)
O2—C1—C2—C3	12.2 (9)	Dy1—O1B—N1B—C1B	143 (7)
N1—C1—C2—C3	−169.5 (8)	Mn1B—O1B—N1B—Mn1B^{ii}	173 (5)
O2—C1—C2—C7	−167.2 (6)	Na1—O1B—N1B—Mn1B^{ii}	51 (9)
N1—C1—C2—C7	11.0 (11)	Dy1—O1B—N1B—Mn1B^{ii}	−50 (10)
C7—C2—C3—C4	2.2 (11)	Mn1B—O2B—C1B—N1B	−2 (9)
C1—C2—C3—C4	−177.3 (7)	Mn1B—O2B—C1B—C2B	−178 (2)
C2—C3—C4—C5	−1.0 (12)	O1B—N1B—C1B—O2B	−2 (14)
C3—C4—C5—C6	0.1 (13)	Mn1B^{ii}—N1B—C1B—O2B	−167 (8)
C4—C5—C6—C7	−0.4 (12)	O1B—N1B—C1B—C2B	173 (6)
Mn1^{i}—O3—C7—C6	166.9 (5)	Mn1B^{ii}—N1B—C1B—C2B	8 (15)
Mn1^{i}—O3—C7—C2	−17.1 (10)	O2B—C1B—C2B—C3B	−3 (4)
C5—C6—C7—O3	177.8 (7)	N1B—C1B—C2B—C3B	−179 (8)
C5—C6—C7—C2	1.5 (11)	O2B—C1B—C2B—C7B	175 (5)
C3—C2—C7—O3	−178.4 (6)	N1B—C1B—C2B—C7B	0 (8)
C1—C2—C7—O3	1.0 (10)	C7B—C2B—C3B—C4B	1 (3)
C3—C2—C7—C6	−2.4 (10)	C1B—C2B—C3B—C4B	180 (3)
C1—C2—C7—C6	177.0 (6)	C2B—C3B—C4B—C5B	1 (5)
Mn1—O5—C8—O4	22.3 (14)	C3B—C4B—C5B—C6B	0 (7)
Mn1—O5—C8—C9C	−158.7 (11)	C4B—C5B—C6B—C7B	−3 (7)
Mn1—O5—C8—C9	−161.6 (12)	Mn1B^{ii}—O3B—C7B—C6B	−180 (3)
Mn1—O5—C8—C9D	−145.4 (13)	Mn1B^{ii}—O3B—C7B—C2B	4 (7)
Dy1—O4—C8—O5	12.0 (18)	C5B—C6B—C7B—O3B	−172 (5)
Dy1—O4—C8—C9C	−166.8 (12)	C5B—C6B—C7B—C2B	5 (6)
Dy1—O4—C8—C9	−164.6 (11)	C3B—C2B—C7B—O3B	172 (5)
Dy1—O4—C8—C9D	−179.9 (12)	C1B—C2B—C7B—O3B	−6 (5)
O5—C8—C9—C13	−84 (2)	C3B—C2B—C7B—C6B	−4 (4)
O4—C8—C9—C13	92 (2)	C1B—C2B—C7B—C6B	178 (3)
O5—C8—C9—C10	55 (2)	Dy1—O4B—C8B—O5B	−22 (5)
O4—C8—C9—C10	−128.2 (18)	Dy1—O4B—C8B—C9B	158 (4)
C13—C9—C10—C11	−168 (3)	O5B—C8B—C9B—C13B	178 (4)
C8—C9—C10—C11	56 (4)	O4B—C8B—C9B—C13B	−3 (4)
C9—C10—C11—C12	174 (3)	O5B—C8B—C9B—C10B	43 (5)
C10—C9—C13—C14	82 (3)	O4B—C8B—C9B—C10B	−137 (4)
C8—C9—C13—C14	−146 (2)	C13B—C9B—C10B—C11B	174 (7)
C9—C13—C14—C15	90 (3)	C8B—C9B—C10B—C11B	−57 (8)
O5—C8—C9C—C13C	−58 (3)	C9B—C10B—C11B—C12B	148 (7)
O4—C8—C9C—C13C	121 (2)	C10B—C9B—C13B—C14B	25 (8)
O5—C8—C9C—C10C	121 (2)	C8B—C9B—C13B—C14B	−102 (6)

O4—C8—C9C—C10C	−60 (3)	C9B—C13B—C14B—C15B	160 (6)
C13C—C9C—C10C—C11C	148 (4)	O7—C16—N2—C17	−173 (3)
C8—C9C—C10C—C11C	−31 (4)	O7—C16—N2—C18	0 (4)
C9C—C10C—C11C—C12C	−131 (4)	O7B—C16B—N2B—C18B	−22 (11)
C10C—C9C—C13C—C14C	108 (4)	O7B—C16B—N2B—C17B	−172 (9)
C8—C9C—C13C—C14C	−74 (3)	O7C—C16C—N2C—C18C	13 (5)
C9C—C13C—C14C—C15C	−20 (3)	O7C—C16C—N2C—C17C	−169 (4)

Symmetry codes: (i) *y*, −*x*+1/2, *z*; (ii) −*y*+1/2, *x*, *z*; (iii) −*x*+1/2, −*y*+1/2, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H6*A*···O7	0.86 (2)	2.02 (2)	2.860 (15)	165 (5)
O6—H6*E*···O7^{ii}	0.83 (2)	1.99 (2)	2.809 (19)	167 (5)
O6*B*—H6*C*···O7*B*^{ii}	0.84 (2)	2.00 (2)	2.80 (3)	159 (10)
O6*B*—H6*D*···O7*B*	0.85 (2)	2.01 (2)	2.859 (19)	177 (10)

Symmetry code: (ii) −*y*+1/2, *x*, *z*.