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Crystal structure and Hirshfeld surface analysis of diaquabis(*N*,*N*-diethylnicotinamide-κN^1)bis(2,4,6-trimethylbenzoato-κO)manganese(II)

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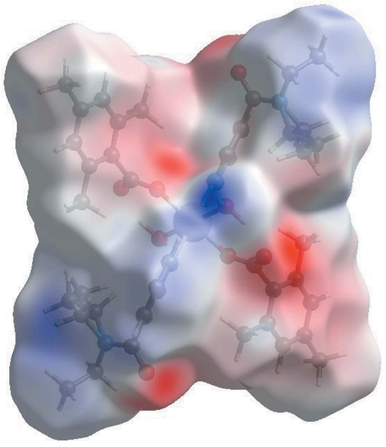
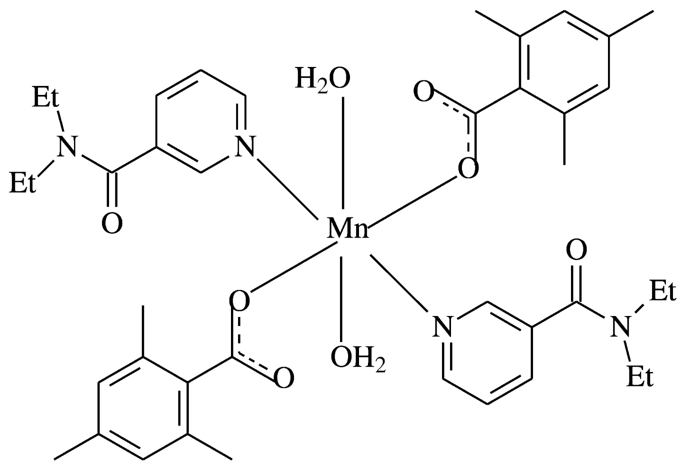
In the title centrosymmetric complex, $[Mn(C_{10}H_{11}O_2)_2(C_{10}H_{14}N_2O)_2(H_2O)_2]$, the Mn^{II} cation is located on an inversion centre. The four O atoms form a slightly distorted square-planar arrangement around the Mn^{II} cation, and the distorted octahedral coordination is completed by two pyridine N atoms at distances of 2.3289 (15) Å. The dihedral angle between the planar carboxylate group and the adjacent benzene ring is 87.73 (16)°, while the benzene and pyridine rings are oriented at a dihedral angle of 43.03 (8)°. In the crystal, the water molecules are involved in both intramolecular (to the non-coordinating carboxylate O atom) and intermolecular (to the amide carbonyl O atom) O—H⋯O hydrogen bonds. The latter lead to the formation of layers parallel to (100). These layers are further linked *via* weak C—H⋯O hydrogen bonds, resulting in a three-dimensional supramolecular network. The Hirshfeld surface analysis of the crystal structure indicates that the most important contributions for the crystal packing are from H⋯H (70.0%), H⋯O/O⋯H (15.5%) and H⋯C/C⋯H (14.0%) interactions. One of the ethyl groups of the diethylnicotinamide ligand is disordered over two sets of sites, with an occupancy ratio of 0.282 (10):0.718 (10).



1. Chemical context

Nicotinamide (NA) is one form of niacin. A deficiency of this vitamin leads to loss of copper from the body, known as pellagra disease. Victims of pellagra show unusually high serum and urinary copper levels (Krishnamachari, 1974). The nicotinic acid derivative *N*,*N*-Diethylnicotinamide (DENA) is an important respiratory stimulant (Bigoli *et al.*, 1972). The crystal structure of the complex $[Co(CH_3CO_2)_2(DENA)_2(H_2O)_2]$ (Mikelashvili, 1982) is isostructural with the analogous Ni, Mn, Zn and Cd complexes (Sergienko *et al.*, 1980). The structures of some complexes obtained from the reactions of transition metal(II) ions with nicotinamide (NA), *N*,*N*-Diethylnicotinamide (DENA) and some benzoic acid derivatives as ligands, *e.g.* $[Zn(NA)_2(C_7H_5O_3)_2]$ [(II); Necefoğlu *et al.*, 2002], $[Zn(NA)_2(C_8H_8NO_2)_2]\cdot H_2O$ [(III); Hökelek *et al.*, 2009*a*], $[Co(NA)(C_9H_{10}NO_2)_2(H_2O)_2]$ [(IV); Hökelek *et al.*, 2009*b*], $[Zn_2(DENA)_2(C_{11}H_{14}NO_2)_4]$ [(V); Hökelek *et al.*, 2009*c*], $[Mn(DENA)_2(C_7H_4ClO_2)_4(H_2O)_2$ [(VI); Hökelek *et al.*, 2009*d*], $[Mn(DENA)_2(NCS)_2]$ [(VII); Bigoli *et al.*, 1973*a*], $[Zn(DENA)_2(NCS)_2(H_2O)_2]$ [(VIII); Bigoli *et al.*, 1973*b*] and $[Cd(DENA)(SCN)_2]$ [(IX); Bigoli *et al.*, 1972] have been determined previously. In complex (VII), DENA is a bidentate ligand, while in complexes (V), (VI), (VIII) and (IX), DENA is a monodentate ligand. In complex (V), the four 4-(diethylamino)benzoate (DEAB) ions act as bidentate ligands bridging the two Zn atoms.



The structure–function–coordination relationships of the arylcarboxylate ion in Mn^{II} complexes of benzoic acid derivatives may change depending on the nature and position of the substituted groups on the benzene ring, the nature of the additional ligand molecule or solvent, and the pH and temperature of synthesis (Shnulin *et al.*, 1981; Nadzhafov *et al.*, 1981; Antsyshkina *et al.*, 1980; Adiwidjaja *et al.*, 1978). When pyridine and its derivatives are used instead of water molecules, the structure is completely different (Catterick *et al.*, 1974). In this context, the Mn^{II}-containing title compound, (I), with 2,4,6-trimethylbenzoate (TMB) and DENA ligands, namely diaquabis(*N*,*N*-diethylnicotinamide -κN^1)bis(2,4,6-trimethylbenzoato-κO^1) manganese(II), $[Mn(DENA)_2(TMB)_2(H_2O)_2]$, was synthesized and its crystal structure is reported on herein.

2. Structural commentary

The asymmetric unit of the crystal structure of the mononuclear title complex, contains one Mn^{II} cation located on an inversion centre, one 2,4,6-trimethylbenzoate (TMB) anion and one *N*,*N*-diethylnicotinamide (DENA) molecule together with the one water molecule, with all ligands coordinating to the Mn^{II} cation in a monodentate manner (Fig. 1).

The Mn^{II} cation is coordinated monodentately through the two carboxylate O atoms (O1 and $O1^i$) of the two symmetry-related TMB anions and the two symmetry-related water O atoms (O4 and $O4^i$) at distances of 2.0999 (14) and 2.2230 (15) Å, respectively, to form a slightly distorted square-planar arrangement, while the slightly distorted octahedral coordination sphere is completed by the two pyridine N atoms (N1 and $N1^i$) at distances of 2.3289 (15) Å of the two symmetry-related DENA ligands in the axial positions [symmetry code: (i) $-x$, $-y$, $-z$] (Fig. 1).

The near equalities of the C1—O1 [1.254 (3) Å] and C1—O2 [1.243 (3) Å] bonds in the carboxylate groups indicate delocalized bonding arrangements, rather than localized single and double bonds. The Mn—O bond lengths [2.2230 (15) Å] for water oxygen atoms are by *ca* 0.1 Å longer than those involving the benzoate oxygen atoms [2.0999 (14) Å]. The Mn—N bond length [2.3289 (15) Å] is the longest one in the MnO_4N_2 octahedron. The Mn1 atom lies 0.0697 (1) Å above the planar (O1/O2/C1) carboxylate group. The O2—C1—O1 bond angle [125.5 (2)°] seems to be significantly increased than that present in a free acid [122.2°], in which the O2—C1—O1 bond angle may be compared with the corresponding values of 123.5 (2) and 120.4 (2)° in (II), 119.2 (3) and 123.8 (2)° in (III), 123.86 (13) and 118.49 (14)° in (IV), 125.11 (13) and 124.80 (14)° in (V) and 126.65 (14)° in (VI), where the benzoate ions are coordinated to the metal atoms only bidentately in (V), only monodentately in (VI) and both monodentately and bidentately in (II), (III) and (IV). The O—Mn—O and O–Mn—N bond angles [range 87.88 (6) to 92.12 (6)° for *cis* angles; all *trans* angles are 180° due to symmetry] deviate slightly from ideal values, with same average values of 90.00 (6)°.

Table 1
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O4—H41$\cdots$O3^{i}	0.85 (3)	2.00 (3)	2.838 (2)	171 (3)
O4—H42$\cdots$O2^{ii}	0.80 (3)	1.90 (3)	2.660 (3)	157 (3)
C9—H9$C\cdots$O2^{ix}	0.96	2.48	3.366 (5)	154
C11—H11$\cdots$O3^{i}	0.93	2.52	3.447 (3)	179

Symmetry codes: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $-x, -y, -z$; (ix) $-x-1, y+\frac{1}{2}, -z-\frac{1}{2}$.

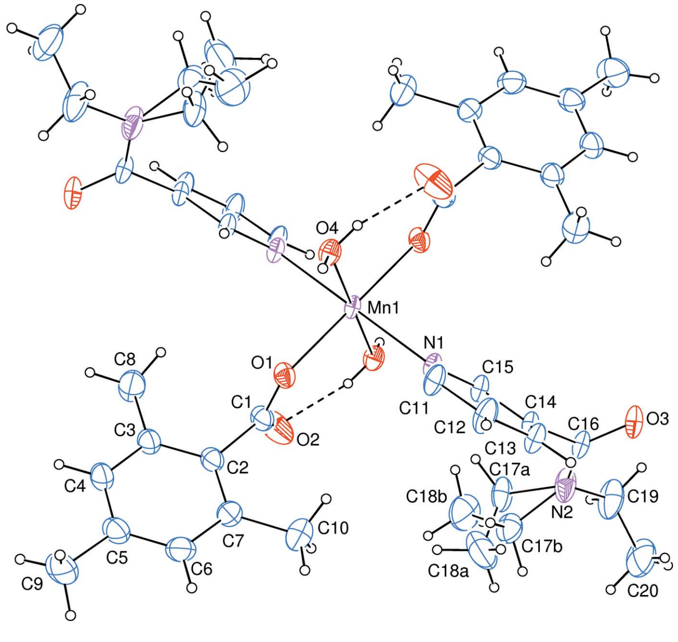


Figure 1
The molecular structure of the title complex with the atom-numbering scheme for the asymmetric unit. Unlabelled atoms are related to labelled ones by the symmetry operation ($-x$, $-y$, $-z$). Displacement ellipsoids are drawn at the 50% probability level. Intramolecular O—H$\cdots$O hydrogen bonds, enclosing S(6) ring motifs, are shown as dashed lines.

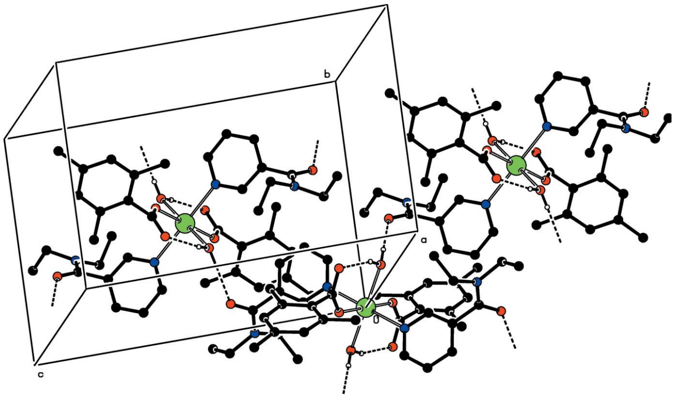


Figure 2
Part of the crystal structure. Only $O-H_W\cdots O_{TMB}$ and $O-H_W\cdots O_{DENA}$ (W = water, TMB = 2,4,6-trimethylbenzoate and DENA = *N*,*N*-diethylnicotinamide) hydrogen bonds, enclosing *S*(6) ring motifs, are shown as dashed lines. Only one part of the disordered group has been included and the C-bound hydrogen atoms have been omitted for clarity.

The dihedral angle between the planar carboxylate group (O1/O2/C1) and the adjacent benzene *A* (C2–C7) ring is 87.73 (16)°, while the benzene *A* and pyridine *B* (N1/C11–C15) rings are oriented at a dihedral angle of A/B = 43.03 (8)°.

3. Supramolecular features

Intramolecular $O-H_w\cdots O_c$ (w = water, c = non-coordinating carboxylate O atom) hydrogen bonds (Table 1) link two of the water ligands to the TMB anions, enclosing an *S*(6) ring motif (Fig. 1). The other water H atom is involved in intermolecular $O-H_w\cdots O_{DENA}$ (O_{DENA} = carbonyl O atom of *N*,*N*-diethylnicotinamide) hydrogen bonds (Table 1), leading to the formation of layers parallel to (100) (Fig. 2). These layers are further linked into a three-dimensional network structure *via* weak $C-H_{TMB}\cdots O_c$ (TMB = 2,4,6-trimethylbenzoate) and $C-H_{DENA}\cdots O_{DENA}$ hdyrogen bonds (Table 1).

4. Hirshfeld surface analysis

Visulization and exploration of intermolecular close contacts in the crystal structure of the title complex is invaluable. Thus, a Hirshfeld surface (HS) analysis (Hirshfeld, 1977; Spackman & Jayatilaka, 2009) was carried out by using Crystal-Explorer*17.5* (Turner *et al.*, 2017) to investigate the locations of atom–atom short contacts with potential to form hydrogen bonds and the quantitative ratios of these interactions and those of the π-stacking interactions. In the HS plotted over d_{norm} (Fig. 3), the white surface indicates contacts with distances equal to the sum of van der Waals radii, and the red and blue colours indicate distances shorter (in close contact) or longer (distinct contact) than the van der Waals radii, respectively (Venkatesan *et al.*, 2016). The bright-red spots appearing near DENA-O3, carboxylate-O2, and hydrogen atoms H41, H42, H9*C* and H11 indicate their roles as the respective donors and acceptors in the dominant $O-H\cdots O$ and $C-H\cdots O$ hydrogen bonds; they also appear as blue and red regions corresponding to positive and negative potentials on the HS mapped over electrostatic potential (Spackman *et al.*, 2008; Jayatilaka *et al.*, 2005) as shown in Fig. 4. The blue regions indicate the positive electrostatic potential (hydrogen-bond donors), while the red regions indicate the negative electrostatic potential (hydrogen-bond acceptors). The shape-index of the HS is a tool to visualize the π–π stacking interactions by the presence of adjacent red and blue triangles; if there are no adjacent red and/or blue triangles, then there are no π–π interactions. Fig. 5 clearly suggests that there are no $\pi\cdots\pi$ interactions in (I).

Table 2
Selected interatomic distances (Å).

$O1\cdots H10B$	2.87	$C17A\cdots H15$	2.78
$O1\cdots H13^{i}$	2.65	$C17B\cdots H20B$	2.75
$O1\cdots H8C$	2.82	$C18A\cdots H9B^{v}$	2.87
$O2\cdots H42^{ii}$	1.90 (3)	$C18B\cdots H8B^{vi}$	2.79
$O2\cdots H9C^{iii}$	2.48	$C20\cdots H17C$	2.76
$O3\cdots H12^{iv}$	2.85	$H4\cdots H8A$	2.37
$O3\cdots H11^{v}$	2.52	$H4\cdots H9A$	2.38
$O3\cdots H41^{v}$	2.00 (3)	$H6\cdots H10A$	2.37
$O3\cdots H19B$	2.35	$H6\cdots H9C$	2.50
$O4\cdots H15^{ii}$	2.62	$H8A\cdots H20A^{vii}$	2.31
$O4\cdots H11$	2.89	$H8B\cdots H17A^{viii}$	2.44
$C1\cdots H42^{ii}$	2.61 (3)	$H8B\cdots H18E^{viii}$	2.14
$C1\cdots H8C$	2.59	$H11\cdots H41$	2.52
$C1\cdots H10B$	2.71	$H15\cdots H18F$	2.48
$C14\cdots H17D$	2.40	$H15\cdots H17B$	2.00
$C14\cdots H17B$	2.74	$H17A\cdots H19A$	1.96
$C14\cdots H18B$	2.82	$H17C\cdots H20B$	2.16
$C15\cdots H17D$	2.88	$H18A\cdots H9B^{v}$	2.50
$C15\cdots H17B$	2.44	$H18E\cdots H19A$	2.46
$C16\cdots H18B$	2.80		

Symmetry codes: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $-x, -y, -z$; (iii) $-x-1, y-\frac{1}{2}, -z-\frac{1}{2}$; (iv) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$; (v) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (vi) $x, -y-\frac{1}{2}, z+\frac{1}{2}$; (vii) $x, y, z-1$; (viii) $x, -y-\frac{1}{2}, z-\frac{1}{2}$.

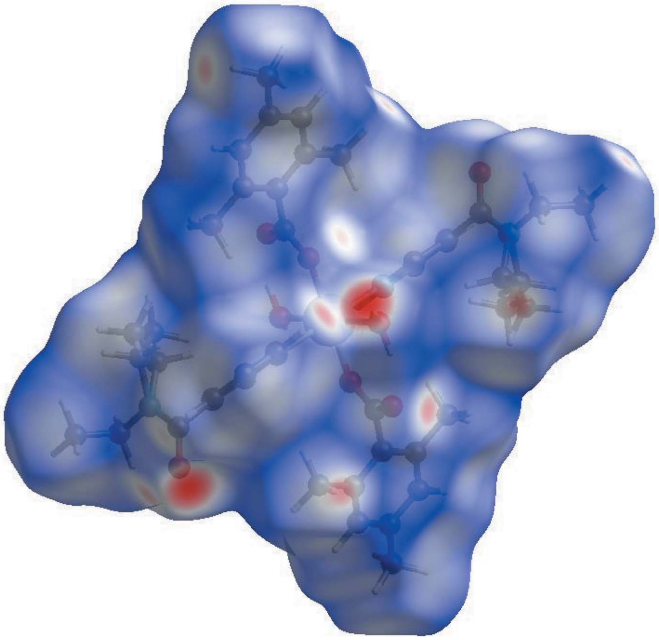


Figure 3
View of the three-dimensional Hirshfeld surface of the title complex plotted over d_{norm} in the range −0.6741 to 1.6440 a.u.

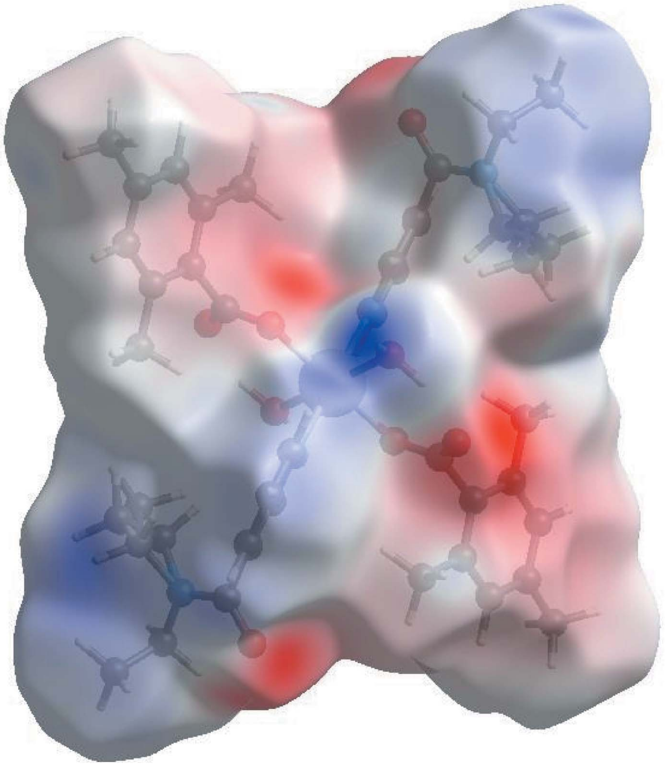


Figure 4
View of the three-dimensional Hirshfeld surface of the title complex plotted over electrostatic potential energy in the range −0.1032 to 0.1415 a.u. using the STO-3G basis set at the Hartree–Fock level of theory. The O—H$\cdots$O and C—H$\cdots$O hydrogen-bond donors and acceptors are viewed as blue and red regions around the atoms corresponding to positive and negative potentials, respectively.

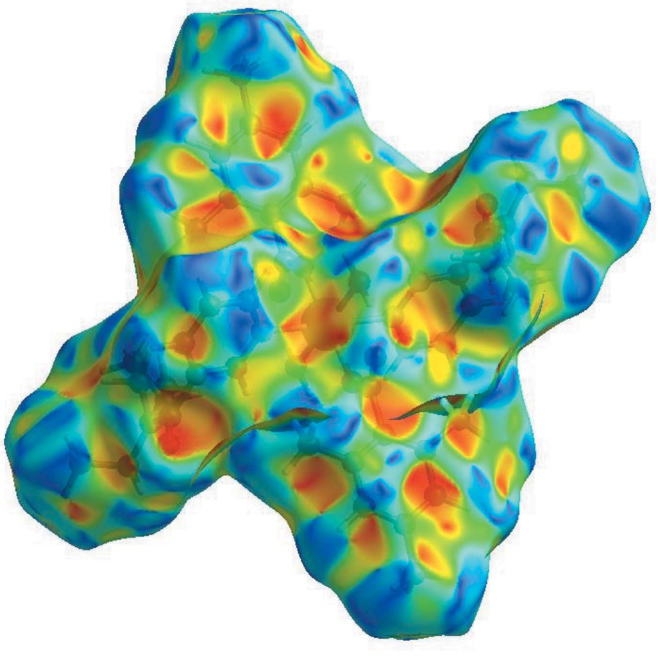


Figure 5
Hirshfeld surface of the title complex plotted over shape-index.

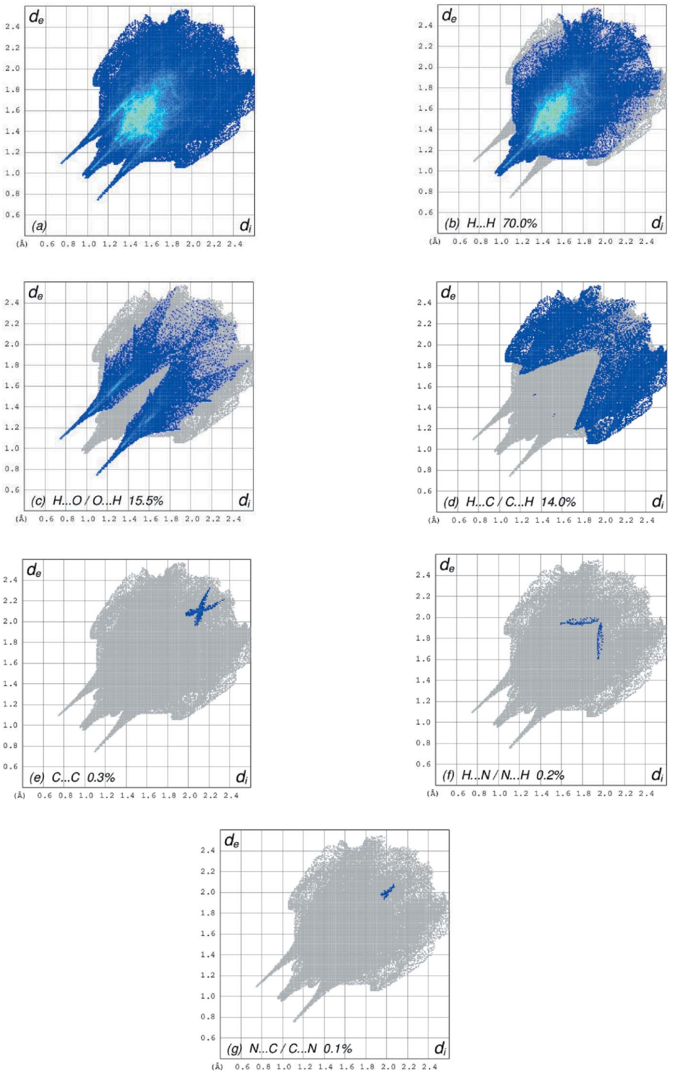
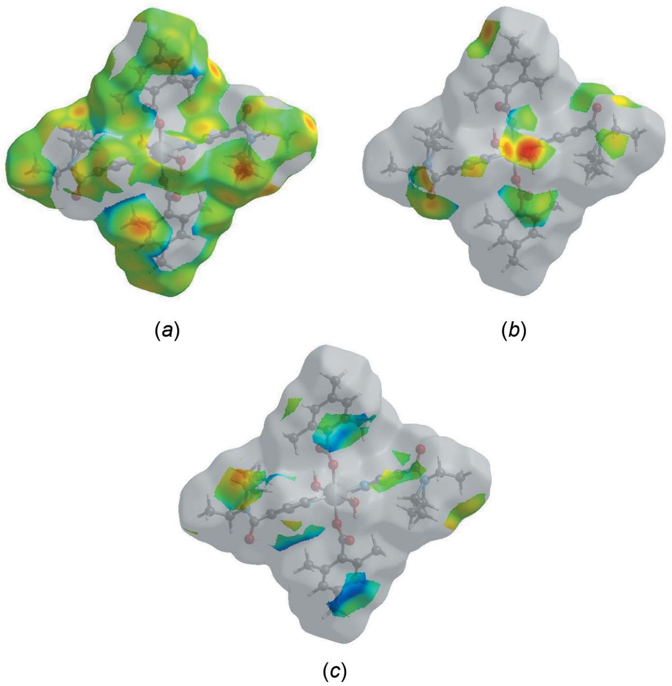


Figure 6
The full two-dimensional fingerprint plots for the title complex, showing (*a*) all interactions, and delineated into (*b*) H$\cdots$H, (*c*) H$\cdots$O/O$\cdots$H, (*d*) H$\cdots$C/C$\cdots$H, (*e*) C$\cdots$C, (*f*) H$\cdots$N/N$\cdots$H and (*g*) N$\cdots$C/C$\cdots$N interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface contacts.

The overall two-dimensional fingerprint plot, Fig. 6*a*, and those delineated into H$\cdots$H, H$\cdots$O/O$\cdots$H, H$\cdots$C/C$\cdots$H, C$\cdots$C, H$\cdots$N/N$\cdots$H and N$\cdots$C/C$\cdots$N contacts (McKinnon *et al.*, 2007) are illustrated in Fig. 6*b*–*g*, respectively, together with their relative contributions to the Hirshfeld surface. The most important interaction is H$\cdots$H, contributing 70.0% to the overall crystal packing, which is reflected in Fig. 6*b* as widely scattered points of high density due to the large hydrogen content of the molecule. The single spike in the centre at $d_e = d_i = 0.96$ Å in Fig. 6*b* is due to a short interatomic H$\cdots$H contact (Table 2). In the fingerprint plot delineated into H$\cdots$O/O$\cdots$H contacts Fig. 6*c*, the 15.5% contribution to the HS arises from intermolecular O—H$\cdots$O hydrogen bonding and is viewed as pair of spikes with the tip at $d_e + d_i$ ~1.84 Å. The short H$\cdots$O/O$\cdots$H contacts may be masked by strong O—H$\cdots$O hydrogen bonding in this plot. In the presence of a



(*a*) (*b*) (*c*)

Figure 7
The Hirshfeld surface representations with the function $d_{\rm norm}$ plotted onto the surface for (*a*) H···H, (*b*) H···O/O···H and (*c*) H···C/C···H interactions.

weak C—H···π interaction in the crystal, the two pairs of characteristic wings in the fingerprint plot delineated into H···C/C···H contacts with 14.0% contribution to the HS, Fig. 6*d*, and the two pairs of thin and thick edges at $d_e + d_i$ ~2.91 and 2.89 Å, respectively, result from short interatomic H···C/C···H contacts (Table 2). The Hirshfeld surface representations with the function $d_{\rm norm}$ plotted onto the surface are shown for the H···H, H···O/O···H and H···C/C···H interactions in Fig. 7*a*–*c*, respectively.

The Hirshfeld surface analysis confirms the importance of H-atom contacts in establishing the packing. The large number of H···H, H···O/O···H and H···C/C···H interactions suggest that van der Waals interactions and hydrogen bonding play the major roles in the crystal packing (Hartwar *et al.*, 2015).

5. Synthesis and crystallization

The title compound was prepared by the reaction of $MnSO_4 \cdot H_2O$ (0.85 g, 5 mmol) in H_2O (100 ml) and *N*,*N*-diethylnicotinamide (1.78 g, 10 mmol) in H_2O (10 ml) with sodium 2,4,6-trimethylbenzoate (1.86 g, 10 mmol) in H_2O (150 ml). The mixture was filtered and set aside to crystallize at ambient temperature for three weeks, giving colourless single crystals.

6. Refinement

Table 3
Experimental details.

Crystal data	
Chemical formula	$[Mn(C_{10}H_{11}O_2)_2(C_{10}H_{14}N_2O)_2(H_2O)_2]$
M_r	773.83
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	13.1040 (4), 10.8828 (3), 15.7167 (4)
β (°)	111.570 (2)
V (Å^3)	2084.37 (10)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.37
Crystal size (mm)	0.45 × 0.37 × 0.35
Data collection	
Diffractometer	Bruker *SMART* BREEZE CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)
$T_{\rm min}$, $T_{\rm max}$	0.851, 0.882
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	36139, 5178, 3995
$R_{\rm int}$	0.030
$(\sin \theta/\lambda)_{\rm max}$ (Å^{-1})	0.667
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.048, 0.144, 1.05
No. of reflections	5178
No. of parameters	274
No. of restraints	42
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\rm max}$, $\Delta\rho_{\rm min}$ (e Å^{-3})	0.51, −0.37

Computer programs: *APEX2* (Bruker, 2012), *SAINT* (Bruker, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008), *ORTEP-3* for Windows (Farrugia, 2012), *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

The experimental details including the crystal data, data collection and refinement are summarized in Table 3. Water H atoms H41 and H42 were located in a difference-Fourier map and freely refined. C-bound H atoms were positioned geometrically, with C—H = 0.93, 0.96 and 0.97 Å for aromatic, methyl and methylene H atoms, respectively, and constrained to ride on their parent atoms, with $U_{\rm iso}({\rm H}) = k \times U_{\rm eq}({\rm C})$, where $k = 1.5$ for methyl H atoms and $k = 1.2$ for other H atoms. The disordered ethyl group (C17, C18) was refined over two sets of sites with distance restraints and SIMU and DELU restraints (Sheldrick, 2008). The refined occupancy ratio of the two orientations is 0.282 (10):0.718 (10).

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Crystal structure and Hirshfeld surface analysis of diaquabis(*N*,*N*-diethylnicotinamide-*κN*^{1})bis(2,4,6-trimethylbenzoato-*κO*)manganese(II)

Tuncer Hökelek, Safiye Özkaya and Hacali Necefoğlu

Computing details

Data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT* (Bruker, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* for Windows (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

Diaquabis(*N*,*N*-diethylnicotinamide-*κN*^{1})bis(2,4,6-trimethylbenzoato-*κO*)manganese(II)

Crystal data

$[Mn(C_{10}H_{11}O_2)_2(C_{10}H_{14}N_2O)_2(H_2O)_2]$
$M_r = 773.83$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 13.1040\ (4)$ Å
$b = 10.8828\ (3)$ Å
$c = 15.7167\ (4)$ Å
$\beta = 111.570\ (2)°$
$V = 2084.37\ (10)$ Å^3
$Z = 2$
$F(000) = 822$
$D_x = 1.233$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9985 reflections
$\theta = 2.5–28.2°$
$\mu = 0.37$ mm^{-1}
$T = 296$ K
Block, translucent light colourless
0.45 × 0.37 × 0.35 mm

Data collection

Bruker SMART BREEZE CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2012)
$T_{min} = 0.851$, $T_{max} = 0.882$
36139 measured reflections
5178 independent reflections
3995 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 28.3°$, $\theta_{min} = 1.7°$
$h = -17 \rightarrow 17$
$k = -14 \rightarrow 13$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.144$
$S = 1.05$
5178 reflections
274 parameters
42 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0696P)^2 + 0.7599P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ 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. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of F^2 > 2sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Mn1	0.0000	0.0000	0.0000	0.04559 (14)	
O1	−0.14717 (12)	0.04378 (15)	−0.10765 (9)	0.0615 (4)	
O2	−0.25400 (18)	−0.1155 (2)	−0.11016 (17)	0.1263 (10)	
O3	−0.02706 (15)	0.12536 (14)	0.39218 (9)	0.0699 (4)	
O4	0.08409 (16)	0.14917 (14)	−0.04553 (11)	0.0590 (4)	
H41	0.058 (2)	0.220 (3)	−0.0636 (19)	0.077 (8)*	
H42	0.143 (2)	0.153 (2)	−0.005 (2)	0.075 (9)*	
N1	−0.04496 (14)	0.13195 (15)	0.09755 (10)	0.0534 (4)	
N2	−0.1788 (2)	0.0256 (3)	0.30478 (15)	0.0975 (8)	
C1	−0.23618 (19)	−0.0138 (2)	−0.13796 (16)	0.0667 (6)	
C2	−0.32465 (17)	0.0468 (2)	−0.21779 (15)	0.0633 (5)	
C3	−0.3318 (2)	0.0191 (2)	−0.30595 (17)	0.0696 (6)	
C4	−0.4057 (2)	0.0847 (3)	−0.37831 (17)	0.0805 (7)	
H4	−0.4101	0.0677	−0.4375	0.097*	
C5	−0.4726 (2)	0.1739 (3)	−0.36515 (19)	0.0837 (8)	
C6	−0.4672 (2)	0.1957 (3)	−0.2775 (2)	0.0871 (8)	
H6	−0.5142	0.2536	−0.2680	0.104*	
C7	−0.3934 (2)	0.1337 (3)	−0.20238 (17)	0.0763 (6)	
C8	−0.2595 (3)	−0.0781 (3)	−0.3224 (2)	0.0988 (9)	
H8A	−0.2585	−0.0688	−0.3828	0.148*	
H8B	−0.2875	−0.1579	−0.3166	0.148*	
H8C	−0.1862	−0.0697	−0.2781	0.148*	
C9	−0.5484 (3)	0.2479 (4)	−0.4452 (3)	0.1240 (14)	
H9A	−0.5621	0.2031	−0.5009	0.186*	
H9B	−0.5145	0.3251	−0.4483	0.186*	
H9C	−0.6165	0.2623	−0.4370	0.186*	
C10	−0.3879 (3)	0.1594 (4)	−0.1064 (2)	0.1176 (12)	
H10A	−0.4327	0.2295	−0.1070	0.176*	
H10B	−0.3134	0.1760	−0.0676	0.176*	
H10C	−0.4143	0.0892	−0.0836	0.176*	
C11	−0.0577 (2)	0.2526 (2)	0.08377 (14)	0.0671 (6)	
H11	−0.0501	0.2856	0.0319	0.081*	

C12	−0.0817 (3)	0.3307 (2)	0.14275 (16)	0.0805 (8)	
H12	−0.0887	0.4147	0.1311	0.097*	
C13	−0.0952 (2)	0.2834 (2)	0.21932 (14)	0.0661 (6)	
H13	−0.1108	0.3345	0.2604	0.079*	
C14	−0.08498 (16)	0.15840 (18)	0.23330 (11)	0.0508 (4)	
C15	−0.05883 (18)	0.08718 (18)	0.17157 (12)	0.0532 (4)	
H15	−0.0504	0.0030	0.1822	0.064*	
C16	−0.09574 (19)	0.10153 (18)	0.31679 (12)	0.0580 (5)	
C17A	−0.2372 (10)	−0.0494 (10)	0.2202 (6)	0.085 (4)	0.282 (10)
H17A	−0.2608	−0.1285	0.2348	0.102*	0.282 (10)
H17B	−0.1926	−0.0612	0.1835	0.102*	0.282 (10)
C17B	−0.2820 (5)	0.0332 (6)	0.2173 (4)	0.0939 (18)	0.718 (10)
H17C	−0.3462	0.0479	0.2326	0.113*	0.718 (10)
H17D	−0.2752	0.1001	0.1790	0.113*	0.718 (10)
C18A	−0.3339 (15)	0.036 (2)	0.1740 (14)	0.155 (8)	0.282 (10)
H18A	−0.3582	0.0269	0.1089	0.233*	0.282 (10)
H18B	−0.3114	0.1199	0.1902	0.233*	0.282 (10)
H18C	−0.3929	0.0165	0.1940	0.233*	0.282 (10)
C18B	−0.2932 (6)	−0.0862 (6)	0.1678 (5)	0.142 (3)	0.718 (10)
H18D	−0.3531	−0.0811	0.1100	0.213*	0.718 (10)
H18E	−0.3071	−0.1507	0.2038	0.213*	0.718 (10)
H18F	−0.2266	−0.1034	0.1579	0.213*	0.718 (10)
C19	−0.1838 (4)	−0.0419 (4)	0.3858 (2)	0.1213 (14)	
H19A	−0.2042	−0.1267	0.3691	0.146*	
H19B	−0.1118	−0.0415	0.4342	0.146*	
C20	−0.2624 (5)	0.0130 (5)	0.4194 (3)	0.171 (3)	
H20A	−0.2656	−0.0343	0.4699	0.257*	
H20B	−0.3335	0.0141	0.3713	0.257*	
H20C	−0.2402	0.0955	0.4392	0.257*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0618 (2)	0.0489 (2)	0.02981 (18)	−0.00067 (16)	0.02117 (16)	0.00058 (13)
O1	0.0652 (9)	0.0672 (8)	0.0465 (7)	−0.0039 (7)	0.0140 (6)	0.0079 (6)
O2	0.0932 (14)	0.1235 (18)	0.1255 (18)	−0.0337 (13)	−0.0030 (13)	0.0690 (15)
O3	0.1131 (12)	0.0614 (8)	0.0348 (7)	−0.0133 (8)	0.0266 (7)	−0.0056 (6)
O4	0.0774 (10)	0.0539 (8)	0.0500 (8)	0.0010 (7)	0.0286 (8)	0.0088 (6)
N1	0.0761 (10)	0.0536 (9)	0.0367 (7)	0.0019 (7)	0.0281 (7)	0.0001 (6)
N2	0.127 (2)	0.1230 (19)	0.0551 (12)	−0.0580 (16)	0.0490 (13)	−0.0147 (12)
C1	0.0634 (12)	0.0778 (15)	0.0558 (12)	−0.0048 (10)	0.0184 (10)	0.0167 (10)
C2	0.0560 (11)	0.0751 (13)	0.0557 (11)	−0.0062 (10)	0.0170 (9)	0.0126 (10)
C3	0.0635 (12)	0.0839 (16)	0.0574 (12)	−0.0026 (11)	0.0178 (10)	0.0092 (10)
C4	0.0739 (15)	0.107 (2)	0.0550 (13)	−0.0008 (14)	0.0166 (11)	0.0126 (12)
C5	0.0642 (14)	0.107 (2)	0.0733 (16)	0.0069 (13)	0.0180 (12)	0.0277 (14)
C6	0.0677 (15)	0.105 (2)	0.0923 (19)	0.0159 (14)	0.0339 (14)	0.0174 (16)
C7	0.0677 (13)	0.0976 (18)	0.0669 (14)	−0.0037 (13)	0.0287 (11)	0.0072 (12)
C8	0.117 (2)	0.098 (2)	0.0807 (19)	0.0161 (18)	0.0365 (17)	0.0007 (16)

C9	0.099 (2)	0.162 (4)	0.100 (2)	0.040 (2)	0.0236 (18)	0.059 (2)
C10	0.123 (3)	0.161 (3)	0.080 (2)	0.015 (2)	0.051 (2)	−0.004 (2)
C11	0.1095 (18)	0.0582 (11)	0.0469 (10)	0.0063 (11)	0.0442 (11)	0.0089 (9)
C12	0.143 (2)	0.0507 (11)	0.0622 (13)	0.0147 (13)	0.0547 (15)	0.0080 (10)
C13	0.1038 (17)	0.0565 (11)	0.0481 (10)	0.0091 (11)	0.0397 (11)	−0.0032 (8)
C14	0.0689 (11)	0.0549 (10)	0.0320 (8)	−0.0057 (8)	0.0224 (8)	−0.0043 (7)
C15	0.0819 (13)	0.0471 (9)	0.0360 (8)	0.0005 (9)	0.0280 (8)	−0.0004 (7)
C16	0.0904 (14)	0.0546 (10)	0.0381 (9)	−0.0074 (10)	0.0344 (9)	−0.0077 (7)
C17A	0.129 (9)	0.068 (6)	0.059 (6)	−0.048 (6)	0.037 (5)	−0.022 (4)
C17B	0.098 (4)	0.109 (4)	0.085 (3)	−0.031 (3)	0.046 (3)	−0.017 (3)
C18A	0.121 (12)	0.197 (19)	0.103 (12)	−0.017 (10)	−0.011 (9)	−0.007 (13)
C18B	0.144 (5)	0.143 (5)	0.142 (5)	−0.059 (4)	0.057 (4)	−0.071 (4)
C19	0.183 (4)	0.124 (3)	0.080 (2)	−0.064 (3)	0.077 (2)	−0.0064 (19)
C20	0.164 (4)	0.283 (7)	0.100 (3)	−0.063 (4)	0.088 (3)	−0.019 (3)

Geometric parameters (Å, º)

Mn1—O1	2.0999 (14)	C9—H9C	0.9600
Mn1—O1$^{\rm i}$	2.0999 (14)	C10—H10A	0.9600
Mn1—O4	2.2230 (15)	C10—H10B	0.9600
Mn1—O4$^{\rm i}$	2.2230 (15)	C10—H10C	0.9600
Mn1—N1	2.3289 (15)	C11—C12	1.377 (3)
Mn1—N1$^{\rm i}$	2.3289 (15)	C11—H11	0.9300
O1—C1	1.254 (3)	C12—H12	0.9300
O2—C1	1.243 (3)	C13—C12	1.379 (3)
O3—C16	1.224 (3)	C13—H13	0.9300
O4—H41	0.85 (3)	C14—C13	1.376 (3)
O4—H42	0.80 (3)	C14—C15	1.380 (2)
N1—C11	1.331 (3)	C14—C16	1.504 (2)
N1—C15	1.334 (2)	C15—H15	0.9300
N2—C17A	1.506 (9)	C16—N2	1.324 (3)
N2—C17B	1.536 (7)	C17A—C18A	1.526 (17)
N2—C19	1.493 (3)	C17A—H17A	0.9700
C2—C1	1.511 (3)	C17A—H17B	0.9700
C2—C3	1.387 (3)	C17B—C18B	1.493 (8)
C2—C7	1.388 (4)	C17B—H17C	0.9700
C3—C8	1.505 (4)	C17B—H17D	0.9700
C4—C3	1.390 (3)	C18A—H18A	0.9600
C4—C5	1.373 (4)	C18A—H18B	0.9600
C4—H4	0.9300	C18A—H18C	0.9600
C5—C6	1.373 (4)	C18B—H18D	0.9600
C5—C9	1.516 (4)	C18B—H18E	0.9600
C6—H6	0.9300	C18B—H18F	0.9600
C7—C6	1.395 (4)	C19—C20	1.447 (6)
C7—C10	1.510 (4)	C19—H19A	0.9700
C8—H8A	0.9600	C19—H19B	0.9700
C8—H8B	0.9600	C20—H20A	0.9600
C8—H8C	0.9600	C20—H20B	0.9600

C9—H9A	0.9600	C20—H20C	0.9600
C9—H9B	0.9600		

O1···H10B	2.87	C16···H18B	2.80
O1···H13^{ii}	2.65	C16···H41^{v}	2.93 (3)
O1···H8C	2.82	C17A···H15	2.78
O2···H42^{i}	1.90 (3)	C17B···H20B	2.75
O2···H9C^{iii}	2.48	C18A···H9B^{v}	2.87
O3···H12^{iv}	2.85	C18B···H8B^{vi}	2.79
O3···H11^{v}	2.52	C18B···H19A	2.97
O3···H41^{v}	2.00 (3)	C19···H18E	2.97
O3···H19B	2.35	C20···H17C	2.76
O4···H15^{i}	2.62	H4···H8A	2.37
O4···H11	2.89	H4···H9A	2.38
C1···H10C	2.98	H6···H10A	2.37
C1···H42^{i}	2.61 (3)	H6···H9C	2.50
C1···H8C	2.59	H8A···H20A^{vii}	2.31
C1···H10B	2.71	H8B···H17A^{viii}	2.44
C5···H18B^{ii}	2.98	H8B···H18E^{viii}	2.14
C13···H17D	2.97	H11···H41	2.52
C14···H17D	2.40	H15···H18F	2.48
C14···H17B	2.74	H15···H17B	2.00
C14···H18B	2.82	H17A···H19A	1.96
C15···H17D	2.88	H17C···H20B	2.16
C15···H17B	2.44	H18A···H9B^{v}	2.50
C15···H18F	2.97	H18E···H19A	2.46

O1^{i}—Mn1—O1	180.00 (7)	H9A—C9—H9B	109.5
O1—Mn1—O4	89.54 (6)	H9A—C9—H9C	109.5
O1^{i}—Mn1—O4	90.46 (6)	H9B—C9—H9C	109.5
O1—Mn1—O4^{i}	90.46 (6)	C7—C10—H10A	109.5
O1^{i}—Mn1—O4^{i}	89.54 (6)	C7—C10—H10B	109.5
O1—Mn1—N1	90.62 (6)	C7—C10—H10C	109.5
O1^{i}—Mn1—N1	89.38 (6)	H10A—C10—H10B	109.5
O1—Mn1—N1^{i}	89.38 (6)	H10A—C10—H10C	109.5
O1^{i}—Mn1—N1^{i}	90.62 (6)	H10B—C10—H10C	109.5
O4^{i}—Mn1—O4	180.00 (9)	N1—C11—C12	123.08 (18)
O4—Mn1—N1	92.12 (6)	N1—C11—H11	118.5
O4^{i}—Mn1—N1	87.88 (6)	C12—C11—H11	118.5
O4—Mn1—N1^{i}	87.88 (6)	C11—C12—C13	119.4 (2)
O4^{i}—Mn1—N1^{i}	92.12 (6)	C11—C12—H12	120.3
N1—Mn1—N1^{i}	180.00 (7)	C13—C12—H12	120.3
Mn1—O4—H41	126.1 (18)	C12—C13—H13	120.9
Mn1—O4—H42	103 (2)	C14—C13—C12	118.18 (18)
H41—O4—H42	111 (3)	C14—C13—H13	120.9
C1—O1—Mn1	130.03 (14)	C13—C14—C15	118.53 (17)
C11—N1—Mn1	123.24 (12)	C13—C14—C16	120.74 (16)
C11—N1—C15	116.92 (16)	C15—C14—C16	120.64 (17)

C15—N1—Mn1	119.84 (12)	N1—C15—C14	123.83 (18)
C16—N2—C17A	126.0 (4)	N1—C15—H15	118.1
C16—N2—C17B	120.1 (3)	C14—C15—H15	118.1
C16—N2—C19	118.5 (2)	O3—C16—N2	122.95 (19)
C19—N2—C17A	108.6 (4)	O3—C16—C14	119.10 (19)
C19—N2—C17B	119.3 (3)	N2—C16—C14	117.94 (18)
O1—C1—C2	114.81 (19)	N2—C17A—C18A	98.7 (12)
O2—C1—O1	125.5 (2)	N2—C17A—H17A	112.0
O2—C1—C2	119.7 (2)	N2—C17A—H17B	112.0
C3—C2—C1	118.9 (2)	C18A—C17A—H17A	112.0
C3—C2—C7	120.9 (2)	C18A—C17A—H17B	112.0
C7—C2—C1	120.1 (2)	H17A—C17A—H17B	109.7
C2—C3—C4	118.4 (2)	N2—C17B—H17C	110.1
C2—C3—C8	120.6 (2)	N2—C17B—H17D	110.1
C4—C3—C8	121.0 (2)	C18B—C17B—N2	107.8 (6)
C3—C4—H4	118.9	C18B—C17B—H17C	110.1
C5—C4—C3	122.1 (3)	C18B—C17B—H17D	110.1
C5—C4—H4	118.9	H17C—C17B—H17D	108.5
C4—C5—C6	118.2 (2)	C17B—C18B—H18D	109.5
C4—C5—C9	120.7 (3)	C17B—C18B—H18E	109.5
C6—C5—C9	121.1 (3)	C17B—C18B—H18F	109.5
C5—C6—C7	122.0 (3)	H18D—C18B—H18E	109.5
C5—C6—H6	119.0	H18D—C18B—H18F	109.5
C7—C6—H6	119.0	H18E—C18B—H18F	109.5
C2—C7—C6	118.3 (2)	N2—C19—H19A	109.3
C2—C7—C10	120.4 (3)	N2—C19—H19B	109.3
C6—C7—C10	121.3 (3)	C20—C19—N2	111.6 (4)
C3—C8—H8A	109.5	C20—C19—H19A	109.3
C3—C8—H8B	109.5	C20—C19—H19B	109.3
C3—C8—H8C	109.5	H19A—C19—H19B	108.0
H8A—C8—H8B	109.5	C19—C20—H20A	109.5
H8A—C8—H8C	109.5	C19—C20—H20B	109.5
H8B—C8—H8C	109.5	C19—C20—H20C	109.5
C5—C9—H9A	109.5	H20A—C20—H20B	109.5
C5—C9—H9B	109.5	H20A—C20—H20C	109.5
C5—C9—H9C	109.5	H20B—C20—H20C	109.5

O4—Mn1—O1—C1	168.4 (2)	C1—C2—C3—C8	5.5 (4)
O4^{i}—Mn1—O1—C1	−11.6 (2)	C7—C2—C3—C4	3.1 (4)
N1—Mn1—O1—C1	−99.5 (2)	C7—C2—C3—C8	−178.0 (2)
N1^{i}—Mn1—O1—C1	80.5 (2)	C1—C2—C7—C6	174.2 (2)
O1—Mn1—N1—C11	−62.66 (19)	C1—C2—C7—C10	−6.0 (4)
O1^{i}—Mn1—N1—C11	117.34 (19)	C3—C2—C7—C6	−2.3 (4)
O1—Mn1—N1—C15	117.53 (16)	C3—C2—C7—C10	177.6 (3)
O1^{i}—Mn1—N1—C15	−62.47 (16)	C5—C4—C3—C2	−1.1 (4)
O4—Mn1—N1—C11	26.91 (19)	C5—C4—C3—C8	−179.9 (3)
O4^{i}—Mn1—N1—C11	−153.09 (19)	C3—C4—C5—C6	−1.7 (4)
O4—Mn1—N1—C15	−152.91 (16)	C3—C4—C5—C9	177.1 (3)

O4^{i}—Mn1—N1—C15	27.09 (16)	C4—C5—C6—C7	2.6 (4)
Mn1—O1—C1—O2	−2.5 (4)	C9—C5—C6—C7	−176.2 (3)
Mn1—O1—C1—C2	−179.51 (14)	C2—C7—C6—C5	−0.6 (4)
Mn1—N1—C11—C12	−178.4 (2)	C10—C7—C6—C5	179.5 (3)
C15—N1—C11—C12	1.4 (4)	N1—C11—C12—C13	−1.1 (4)
Mn1—N1—C15—C14	179.68 (16)	C14—C13—C12—C11	−0.6 (4)
C11—N1—C15—C14	−0.1 (3)	C15—C14—C13—C12	1.7 (4)
C16—N2—C17A—C18A	−96.6 (11)	C16—C14—C13—C12	178.3 (2)
C17B—N2—C17A—C18A	−0.4 (10)	C13—C14—C15—N1	−1.5 (3)
C19—N2—C17A—C18A	113.2 (10)	C16—C14—C15—N1	−178.04 (19)
C16—N2—C17B—C18B	117.7 (4)	C13—C14—C16—O3	−65.7 (3)
C17A—N2—C17B—C18B	6.0 (7)	C13—C14—C16—N2	115.6 (3)
C19—N2—C17B—C18B	−78.9 (5)	C15—C14—C16—O3	110.9 (2)
C16—N2—C19—C20	102.2 (4)	C15—C14—C16—N2	−67.9 (3)
C17A—N2—C19—C20	−105.0 (7)	O3—C16—N2—C17A	−152.8 (7)
C17B—N2—C19—C20	−61.5 (5)	O3—C16—N2—C17B	158.4 (3)
C3—C2—C1—O1	89.8 (3)	O3—C16—N2—C19	−5.2 (4)
C3—C2—C1—O2	−87.4 (3)	C14—C16—N2—C17A	26.0 (8)
C7—C2—C1—O1	−86.7 (3)	C14—C16—N2—C17B	−22.9 (4)
C7—C2—C1—O2	96.0 (3)	C14—C16—N2—C19	173.5 (3)
C1—C2—C3—C4	−173.4 (2)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H41···O3^{ii}	0.85 (3)	2.00 (3)	2.838 (2)	171 (3)
O4—H42···O2^{i}	0.80 (3)	1.90 (3)	2.660 (3)	157 (3)
C9—H9*C*···O2^{ix}	0.96	2.48	3.366 (5)	154
C11—H11···O3^{ii}	0.93	2.52	3.447 (3)	179

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