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Bis[4-(2-hydroxybenzylideneamino)-benzoato- κ O]tetrakis(methanol- κ O)-manganese(II)

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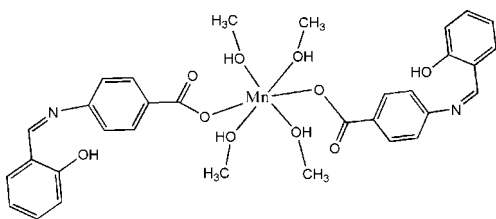
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.039; wR factor = 0.102; data-to-parameter ratio = 16.5.

In the title mononuclear complex, $[\text{Mn}(\text{C}_{14}\text{H}_{10}\text{NO}_3)_2(\text{CH}_3\text{OH})_4]$, the Mn^{II} atom, lying on an inversion centre, exhibits a distorted octahedral geometry, defined by two O atoms from two monodentate ligands and four O atoms from four methanol molecules. The crystal structure involves intramolecular $\text{O}-\text{H}\cdots\text{N}$ and $\text{O}-\text{H}\cdots\text{O}$ and intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For general background, see: Deeth (2008); Dubois *et al.* (2008); Huang *et al.* (2004).



Experimental

Crystal data

$[\text{Mn}(\text{C}_{14}\text{H}_{10}\text{NO}_3)_2(\text{CH}_3\text{O})_4]$
 $M_r = 663.57$
 Monoclinic, $P2_1/c$
 $a = 15.0341$ (6) Å
 $b = 11.8819$ (4) Å
 $c = 8.8178$ (3) Å
 $\beta = 98.912$ (4)°

$V = 1556.14$ (10) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.49$ mm⁻¹
 $T = 293$ K
 $0.6 \times 0.6 \times 0.3$ mm

Data collection

Oxford Diffraction Gemini S Ultra diffractometer
 Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)

$T_{\text{min}} = 0.823$, $T_{\text{max}} = 1.000$ (expected range = 0.711–0.865)
 10167 measured reflections
 3374 independent reflections
 2008 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.102$
 $S = 0.92$
 3374 reflections

205 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.55$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.31$ e Å⁻³

Table 1

Selected bond lengths (Å).

Mn1—O3	2.1275 (15)	Mn1—O4	2.2023 (14)
Mn1—O5	2.1803 (13)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1A \cdots N1	0.85	1.83	2.619 (2)	153
O4—H4B \cdots O2 ⁱ	0.85	1.84	2.621 (2)	151
O5—H5B \cdots O2	0.85	1.83	2.618 (2)	153

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2192).

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supporting information

Acta Cryst. (2009). E65, m634 [doi:10.1107/S160053680901681X]

Bis[4-(2-hydroxybenzylideneamino)benzoato- κ O]tetrakis(methanol- κ O)manganese(II)

Min Zhong, Jia-Huang Lin, Jing Shang, Ting-Hong Huang and Xiu-Jian Wang

S1. Comment

General molecular mechanics method for transition metal carboxylates and the multiple coordination modes in manganese(II) complexes have been reported recently (Deeth, 2008). Information on the structures of manganese(II) carboxylates continues to be collected, and at the same time new applications of such complexes are being discovered in magnetic properties, potential biological significance and ferrimagnet (Huang *et al.*, 2004). The chemistry of organo-manganese(II) complexes of Schiff base has stemmed from the reported biocidal and catalytic activities of organo-manganese(II) compounds (Dubois *et al.*, 2008). We report here a new monomeric manganese(II) compound, which contains the Schiff base ligand, *N*-(4-carboxyphenyl)salicylideneimine (Fig.1). The Mn^{II} atom has a distorted octahedral geometry (Table 1). There exist intra- and intermolecular hydrogen bonds in the crystal structure (Table 2). The intermolecular hydrogen bonds is used to form a two-dimensional supramolecular network (Fig. 2).

S2. Experimental

Manganese(II) acetate tetrahydrate (0.049 g, 0.2 mmol) was dissolved in 8 ml deionized water, giving a transparent solution (A), and *N*-(4-carboxyphenyl)salicylideneimine (0.097 g, 0.4 mmol) was dissolved in 10 ml methanol (B). Then solution B was mixed with A and a suspension was obtained. Ammonia was added to the above mixture dropwise under magnetic stirring until pH value is neutral. The resulting suspension was transferred into a 25 ml Teflon-lined stainless-steel autoclave. The autoclave was sealed and maintained at 363 K for 12 h under autogenous pressure. After the reaction was completed, the resulting colourless block crystals were collected by filtration.

S3. Refinement

H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 (aromatic) and 0.96 (CH₃) Å and O—H = 0.85 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{O})$.

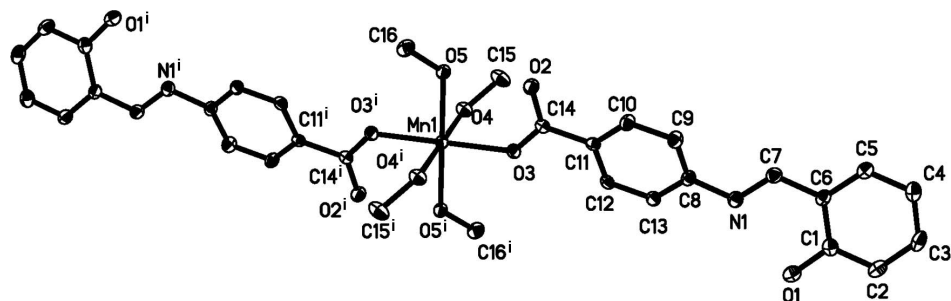


Figure 1

Molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. [Symmetry code: (i) $-x + 1, -y, -z$.]

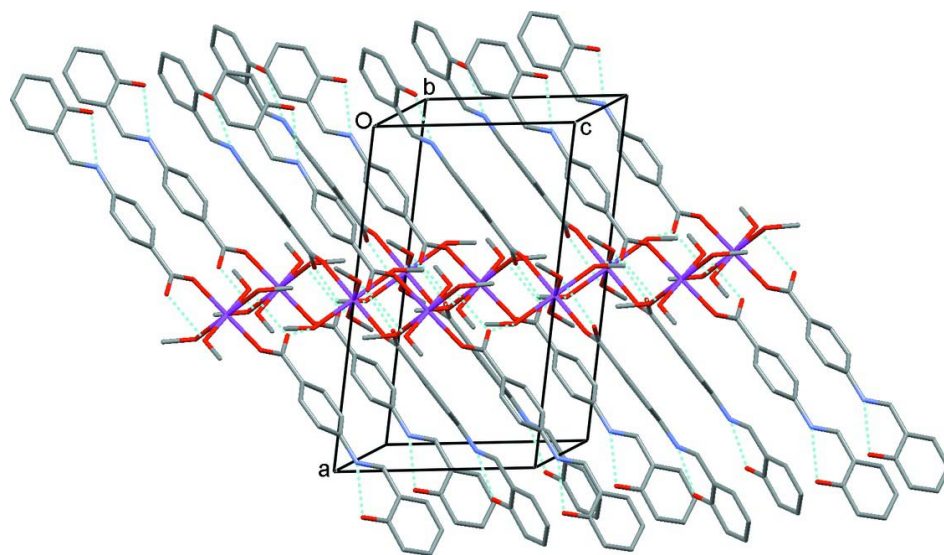


Figure 2

Crystal packing of the title compound, showing hydrogen bonds (dashed lines).

Bis[4-(2-hydroxybenzylideneamino)benzoato- κ O]tetrakis(methanol- κ O)manganese(II)

Crystal data

$[\text{Mn}(\text{C}_{14}\text{H}_{10}\text{NO}_3)_2(\text{CH}_4\text{O})_4]$

$M_r = 663.57$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 15.0341(6)\ \text{\AA}$

$b = 11.8819(4)\ \text{\AA}$

$c = 8.8178(3)\ \text{\AA}$

$\beta = 98.912(4)^\circ$

$V = 1556.14(10)\ \text{\AA}^3$

$Z = 2$

$F(000) = 694$

$D_x = 1.416\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 3622 reflections

$\theta = 2.7\text{--}29.8^\circ$

$\mu = 0.49\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, colorless

$0.6 \times 0.6 \times 0.3\ \text{mm}$

Data collection

Oxford Diffraction Gemini S Ultra
diffractometer

Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator

Detector resolution: $16.0855\ \text{pixels mm}^{-1}$

ω scans

Absorption correction: multi-scan

(*CrysAlis RED*; Oxford Diffraction, 2006)

$T_{\min} = 0.823$, $T_{\max} = 1.000$
 10167 measured reflections
 3374 independent reflections
 2008 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$

$\theta_{\max} = 27.0^\circ$, $\theta_{\min} = 2.7^\circ$
 $h = -19 \rightarrow 16$
 $k = -15 \rightarrow 14$
 $l = -11 \rightarrow 11$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.102$
 $S = 0.92$
 3374 reflections
 205 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.058P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.55 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.31 \text{ e } \text{\AA}^{-3}$

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Mn1	0.5000	0.0000	0.0000	0.02171 (15)
O1	1.09324 (11)	0.03911 (14)	0.80222 (19)	0.0404 (4)
H1A	1.0420	0.0210	0.7533	0.048*
O2	0.57922 (10)	-0.20216 (13)	0.26345 (18)	0.0350 (4)
O3	0.60735 (10)	-0.03061 (12)	0.18268 (17)	0.0291 (4)
N1	0.94227 (12)	-0.06941 (15)	0.7155 (2)	0.0295 (5)
C1	1.11213 (15)	-0.0410 (2)	0.9117 (3)	0.0303 (6)
C2	1.19528 (16)	-0.0391 (2)	1.0049 (3)	0.0360 (6)
H2A	1.2378	0.0151	0.9904	0.043*
C3	1.21437 (16)	-0.1187 (2)	1.1196 (3)	0.0397 (6)
H3A	1.2704	-0.1177	1.1815	0.048*
C4	1.15283 (15)	-0.1989 (2)	1.1442 (3)	0.0377 (6)
H4A	1.1665	-0.2511	1.2229	0.045*
C5	1.07051 (15)	-0.2014 (2)	1.0510 (3)	0.0332 (6)
H5A	1.0288	-0.2561	1.0675	0.040*
C6	1.04831 (14)	-0.12450 (18)	0.9335 (2)	0.0276 (5)
C7	0.96278 (15)	-0.13309 (19)	0.8321 (3)	0.0306 (5)
H7A	0.9215	-0.1870	0.8529	0.037*
C8	0.86185 (14)	-0.08573 (19)	0.6112 (2)	0.0269 (5)
C9	0.81878 (14)	-0.18938 (19)	0.5844 (3)	0.0312 (6)
H9A	0.8412	-0.2524	0.6402	0.037*
C10	0.74265 (14)	-0.19842 (19)	0.4748 (3)	0.0299 (5)
H10A	0.7137	-0.2675	0.4587	0.036*
C11	0.70884 (14)	-0.10594 (17)	0.3887 (2)	0.0225 (5)
C12	0.75350 (15)	-0.00380 (18)	0.4146 (2)	0.0259 (5)
H12A	0.7322	0.0587	0.3567	0.031*
C13	0.82848 (15)	0.00641 (19)	0.5241 (2)	0.0279 (5)
H13A	0.8572	0.0757	0.5401	0.033*
C14	0.62646 (14)	-0.11436 (18)	0.2699 (2)	0.0234 (5)

O4	0.42496 (10)	0.08439 (12)	0.16313 (16)	0.0306 (4)
H4B	0.4371	0.1543	0.1666	0.037*
O5	0.43893 (10)	-0.15779 (11)	0.05698 (16)	0.0271 (4)
H5B	0.4791	-0.1938	0.1163	0.033*
C15	0.42208 (19)	0.0436 (2)	0.3134 (3)	0.0436 (7)
H15A	0.3862	0.0932	0.3650	0.065*
H15B	0.4821	0.0403	0.3695	0.065*
H15C	0.3960	-0.0303	0.3075	0.065*
C16	0.39223 (16)	-0.23800 (19)	-0.0468 (3)	0.0368 (6)
H16A	0.3730	-0.2999	0.0103	0.055*
H16B	0.4316	-0.2651	-0.1145	0.055*
H16C	0.3406	-0.2029	-0.1058	0.055*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0265 (3)	0.0162 (2)	0.0218 (3)	-0.0006 (2)	0.00172 (19)	0.0002 (2)
O1	0.0353 (10)	0.0405 (10)	0.0447 (11)	-0.0090 (8)	0.0041 (8)	0.0001 (9)
O2	0.0365 (9)	0.0240 (9)	0.0407 (10)	-0.0064 (7)	-0.0061 (8)	0.0091 (8)
O3	0.0313 (9)	0.0259 (9)	0.0282 (9)	-0.0022 (7)	-0.0011 (7)	0.0067 (7)
N1	0.0296 (11)	0.0303 (11)	0.0282 (10)	-0.0001 (9)	0.0029 (9)	-0.0037 (9)
C1	0.0333 (14)	0.0307 (13)	0.0272 (12)	0.0057 (10)	0.0061 (11)	-0.0047 (11)
C2	0.0260 (13)	0.0373 (14)	0.0454 (15)	-0.0033 (11)	0.0077 (12)	-0.0122 (12)
C3	0.0247 (14)	0.0523 (17)	0.0392 (15)	0.0064 (12)	-0.0040 (11)	-0.0083 (13)
C4	0.0366 (14)	0.0417 (15)	0.0331 (14)	0.0070 (12)	0.0002 (12)	0.0001 (12)
C5	0.0286 (13)	0.0352 (14)	0.0354 (14)	-0.0022 (11)	0.0035 (11)	-0.0022 (12)
C6	0.0223 (12)	0.0336 (13)	0.0271 (12)	0.0010 (10)	0.0044 (10)	-0.0069 (11)
C7	0.0304 (13)	0.0313 (13)	0.0302 (13)	-0.0033 (10)	0.0053 (11)	-0.0027 (11)
C8	0.0245 (12)	0.0312 (13)	0.0249 (12)	-0.0004 (10)	0.0036 (10)	-0.0059 (10)
C9	0.0293 (13)	0.0247 (13)	0.0373 (14)	0.0055 (10)	-0.0015 (11)	0.0034 (11)
C10	0.0293 (13)	0.0226 (12)	0.0355 (14)	-0.0011 (10)	-0.0022 (11)	-0.0010 (11)
C11	0.0252 (12)	0.0214 (11)	0.0225 (11)	0.0003 (9)	0.0086 (10)	-0.0014 (9)
C12	0.0330 (12)	0.0224 (11)	0.0223 (11)	0.0000 (11)	0.0043 (9)	0.0032 (10)
C13	0.0336 (13)	0.0244 (12)	0.0251 (11)	-0.0048 (11)	0.0025 (10)	-0.0020 (11)
C14	0.0276 (12)	0.0201 (12)	0.0237 (11)	0.0025 (10)	0.0075 (10)	-0.0001 (10)
O4	0.0433 (10)	0.0183 (8)	0.0317 (9)	0.0003 (7)	0.0105 (7)	-0.0017 (7)
O5	0.0306 (8)	0.0180 (8)	0.0309 (9)	-0.0023 (7)	-0.0010 (7)	0.0019 (7)
C15	0.070 (2)	0.0313 (13)	0.0339 (14)	-0.0014 (13)	0.0207 (14)	0.0006 (12)
C16	0.0417 (15)	0.0264 (13)	0.0415 (15)	-0.0077 (11)	0.0041 (12)	-0.0096 (11)

Geometric parameters (Å, °)

Mn1—O3	2.1275 (15)	C7—H7A	0.9300
Mn1—O3 ⁱ	2.1275 (15)	C8—C13	1.386 (3)
Mn1—O5 ⁱ	2.1802 (13)	C8—C9	1.394 (3)
Mn1—O5	2.1803 (13)	C9—C10	1.383 (3)
Mn1—O4	2.2023 (14)	C9—H9A	0.9300
Mn1—O4 ⁱ	2.2023 (14)	C10—C11	1.387 (3)

O1—C1	1.354 (3)	C10—H10A	0.9300
O1—H1A	0.8500	C11—C12	1.389 (3)
O2—C14	1.258 (2)	C11—C14	1.496 (3)
O3—C14	1.263 (2)	C12—C13	1.372 (3)
N1—C7	1.275 (3)	C12—H12A	0.9300
N1—C8	1.415 (3)	C13—H13A	0.9300
C1—C2	1.386 (3)	O4—C15	1.418 (3)
C1—C6	1.414 (3)	O4—H4B	0.8500
C2—C3	1.382 (3)	O5—C16	1.428 (2)
C2—H2A	0.9300	O5—H5B	0.8500
C3—C4	1.369 (3)	C15—H15A	0.9600
C3—H3A	0.9300	C15—H15B	0.9600
C4—C5	1.376 (3)	C15—H15C	0.9600
C4—H4A	0.9300	C16—H16A	0.9600
C5—C6	1.383 (3)	C16—H16B	0.9600
C5—H5A	0.9300	C16—H16C	0.9600
C6—C7	1.452 (3)		
O3—Mn1—O3 ⁱ	180.00 (10)	C13—C8—C9	119.0 (2)
O3—Mn1—O5 ⁱ	91.39 (5)	C13—C8—N1	116.85 (19)
O3 ⁱ —Mn1—O5 ⁱ	88.61 (5)	C9—C8—N1	124.0 (2)
O3—Mn1—O5	88.61 (5)	C10—C9—C8	120.0 (2)
O3 ⁱ —Mn1—O5	91.39 (5)	C10—C9—H9A	120.0
O5 ⁱ —Mn1—O5	180.00 (7)	C8—C9—H9A	120.0
O3—Mn1—O4	89.34 (6)	C9—C10—C11	121.0 (2)
O3 ⁱ —Mn1—O4	90.66 (6)	C9—C10—H10A	119.5
O5 ⁱ —Mn1—O4	92.04 (5)	C11—C10—H10A	119.5
O5—Mn1—O4	87.96 (5)	C10—C11—C12	118.4 (2)
O3—Mn1—O4 ⁱ	90.66 (6)	C10—C11—C14	121.60 (19)
O3 ⁱ —Mn1—O4 ⁱ	89.34 (6)	C12—C11—C14	120.02 (19)
O5 ⁱ —Mn1—O4 ⁱ	87.96 (5)	C13—C12—C11	121.1 (2)
O5—Mn1—O4 ⁱ	92.04 (5)	C13—C12—H12A	119.4
O4—Mn1—O4 ⁱ	180.00 (7)	C11—C12—H12A	119.4
C1—O1—H1A	104.9	C12—C13—C8	120.5 (2)
C14—O3—Mn1	132.05 (14)	C12—C13—H13A	119.7
C7—N1—C8	121.23 (19)	C8—C13—H13A	119.7
O1—C1—C2	118.8 (2)	O2—C14—O3	123.5 (2)
O1—C1—C6	121.2 (2)	O2—C14—C11	119.14 (19)
C2—C1—C6	120.0 (2)	O3—C14—C11	117.32 (18)
C3—C2—C1	119.3 (2)	C15—O4—Mn1	123.20 (14)
C3—C2—H2A	120.4	C15—O4—H4B	109.7
C1—C2—H2A	120.4	Mn1—O4—H4B	109.9
C4—C3—C2	121.6 (2)	C16—O5—Mn1	127.52 (13)
C4—C3—H3A	119.2	C16—O5—H5B	106.9
C2—C3—H3A	119.2	Mn1—O5—H5B	106.9
C3—C4—C5	119.2 (2)	O4—C15—H15A	109.5
C3—C4—H4A	120.4	O4—C15—H15B	109.5
C5—C4—H4A	120.4	H15A—C15—H15B	109.5

C4—C5—C6	121.6 (2)	O4—C15—H15C	109.5
C4—C5—H5A	119.2	H15A—C15—H15C	109.5
C6—C5—H5A	119.2	H15B—C15—H15C	109.5
C5—C6—C1	118.4 (2)	O5—C16—H16A	109.5
C5—C6—C7	120.2 (2)	O5—C16—H16B	109.5
C1—C6—C7	121.4 (2)	H16A—C16—H16B	109.5
N1—C7—C6	122.4 (2)	O5—C16—H16C	109.5
N1—C7—H7A	118.8	H16A—C16—H16C	109.5
C6—C7—H7A	118.8	H16B—C16—H16C	109.5
O5 ⁱ —Mn1—O3—C14	-173.33 (18)	C8—C9—C10—C11	1.0 (3)
O5—Mn1—O3—C14	6.67 (18)	C9—C10—C11—C12	0.3 (3)
O4—Mn1—O3—C14	94.64 (19)	C9—C10—C11—C14	-179.90 (18)
O4 ⁱ —Mn1—O3—C14	-85.36 (19)	C10—C11—C12—C13	-1.0 (3)
O1—C1—C2—C3	178.3 (2)	C14—C11—C12—C13	179.17 (18)
C6—C1—C2—C3	-0.6 (3)	C11—C12—C13—C8	0.4 (3)
C1—C2—C3—C4	-0.5 (4)	C9—C8—C13—C12	0.8 (3)
C2—C3—C4—C5	1.0 (4)	N1—C8—C13—C12	176.55 (18)
C3—C4—C5—C6	-0.2 (3)	Mn1—O3—C14—O2	-1.2 (3)
C4—C5—C6—C1	-0.9 (3)	Mn1—O3—C14—C11	-179.75 (12)
C4—C5—C6—C7	176.7 (2)	C10—C11—C14—O2	10.7 (3)
O1—C1—C6—C5	-177.6 (2)	C12—C11—C14—O2	-169.4 (2)
C2—C1—C6—C5	1.3 (3)	C10—C11—C14—O3	-170.6 (2)
O1—C1—C6—C7	4.8 (3)	C12—C11—C14—O3	9.2 (3)
C2—C1—C6—C7	-176.2 (2)	O3—Mn1—O4—C15	-41.12 (17)
C8—N1—C7—C6	174.53 (19)	O3 ⁱ —Mn1—O4—C15	138.88 (17)
C5—C6—C7—N1	-174.7 (2)	O5 ⁱ —Mn1—O4—C15	-132.49 (17)
C1—C6—C7—N1	2.8 (3)	O5—Mn1—O4—C15	47.51 (17)
C7—N1—C8—C13	157.1 (2)	O3—Mn1—O5—C16	-142.12 (16)
C7—N1—C8—C9	-27.4 (3)	O3 ⁱ —Mn1—O5—C16	37.88 (16)
C13—C8—C9—C10	-1.5 (3)	O4—Mn1—O5—C16	128.49 (16)
N1—C8—C9—C10	-176.92 (19)	O4 ⁱ —Mn1—O5—C16	-51.51 (16)

Symmetry code: (i) $-x+1, -y, -z$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1A \cdots N1	0.85	1.83	2.619 (2)	153
O4—H4B \cdots O2 ⁱⁱ	0.85	1.84	2.621 (2)	151
O5—H5B \cdots O2	0.85	1.83	2.618 (2)	153

Symmetry code: (ii) $-x+1, y+1/2, -z+1/2$.