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Aquaazido{2,2′-[*o*-phenylenebis(nitrilomethylidyne)]diphenolato}manganese(III) hemihydrate

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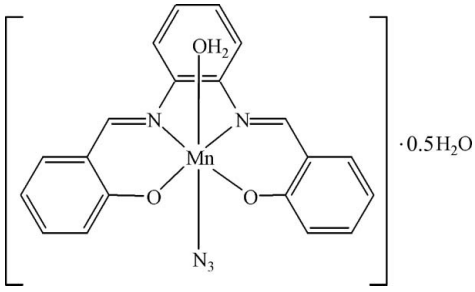
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.086; data-to-parameter ratio = 11.3.

In the title compound, $[Mn(C_{20}H_{14}N_2O_2)(N_3)(H_2O)]\cdot 0.5H_2O$, the Mn^{III} ion is chelated by the N,N',O,O'-tetradentate Schiff base ligand and further coordinated by one azide ion and one water molecule in *trans* positions, resulting in a distorted *fac*-MnN_3O_3 octahedral arrangement. The O atom of the uncoordinated water molecule lies on a crystallographic twofold axis. In the crystal, O—H···O and O—H···N hydrogen bonds help to establish the packing.

Related literature

For background to salicylaldehyde complexes, see: Alam *et al.* (2003); Zelewsky & von Knof (1999).



Experimental

Crystal data

$[Mn(C_{20}H_{14}N_2O_2)(N_3)(H_2O)]\cdot 0.5H_2O$
M_r = 438.33
Monoclinic, $C2/c$
a = 25.100 (10) Å
b = 11.478 (5) Å
c = 12.599 (5) Å
β = 94.175 (3)°
V = 3620 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.77 mm^{-1}
T = 293 K
0.12 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.914, T_{max} = 0.941
11927 measured reflections
3162 independent reflections
2371 reflections with $I > 2\sigma(I)$
R_{int} = 0.082

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.086
S = 1.00
3162 reflections
280 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Selected bond lengths (Å).

Mn1—O1	1.8636 (18)	Mn1—N1	1.988 (2)
Mn1—O2	1.8844 (18)	Mn1—N3	2.306 (2)
Mn1—N2	1.986 (2)	Mn1—O1*W*	2.321 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H2*W*···N3^{i}	0.82 (2)	2.12 (2)	2.937 (3)	176 (3)
O1*W*—H1*W*···O2^{ii}	0.820 (11)	2.076 (6)	2.885 (3)	169 (2)
O2*W*—H3*W*···N5	0.82 (3)	2.18 (3)	3.000 (3)	173 (4)

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: HB2977).

References

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supporting information

Acta Cryst. (2009). E**65**, m707 [doi:10.1107/S1600536809020200]

Aquaazido{2,2′-[*o*-phenylenebis(nitrilomethylidyne)]diphenolato}manganese(III) hemihydrate

Xiutang Zhang, Peihai Wei, Bin Li, Chunyong Wu and Bo Hu

S1. Comment

The synthesis of complexes consisting of salicylaldehyde ligand has attracted continuous research interest not only because of their appealing structural and topological novelty, but also due to their unusual optical, electronic, magnetic, and catalytic properties, as well as their potential medical application (Alam *et al.*, 2003; Zelewsky & von Knof, 1999). In the present paper, we describe the synthesis and structural characterizations of the title compound, (I),

As shown in Fig. 1, each Mn(III) atom is chelated by Schiff base ligand *via* two N and two O atoms and is additionally coordinated by one azide and a water molecule, forming a distorted octahedral geometry (Table 1) in which, the Schiff base lies in the equatorial plane, and the azide and aqua ligands lie in the axial coordination sites.

With O—H···O and O—H···N hydrogen bonds (Table 2), a three-dimensional network is formed as shown in Fig. 2.

S2. Experimental

A mixture of manganese(III) acetylacetonate (1 mmol) and *N*,*N*′-bis(2-hydroxy-5-bromobenzyl)1,2-diaminopropane (1 mmol), and dipotassium nickel tetracyanide (1 mmol) in 20 ml methanol was refluxed for several hours. The above cooled solution was filtered and the filtrate was kept in an ice box. One week later, brown blocks of (I) were obtained with a yield of 5%. Anal. Calc. for $C_{40}H_{34}Mn_2N_{10}O_7$: C 54.75, H 3.88, N 15.97%; Found: C 54.71, H 3.75, N 15.82.

S3. Refinement

All H atoms were placed in calculated positions with C—H = 0.93Å and refined as riding with U_{iso}(H) = 1.2U_{eq}(carrier). H atom on aqua were located from difference density maps and were refined with distance restraints of O–H = 0.82 (1) Å.

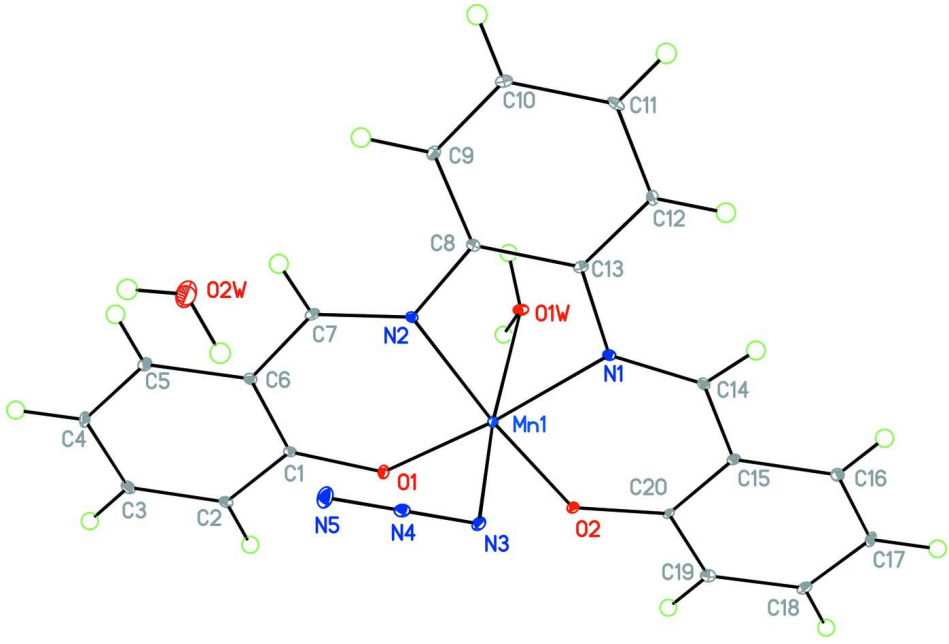

Figure 1

The molecular structure of (I), drawn with 30% probability displacement ellipsoids for the non-hydrogen atoms.

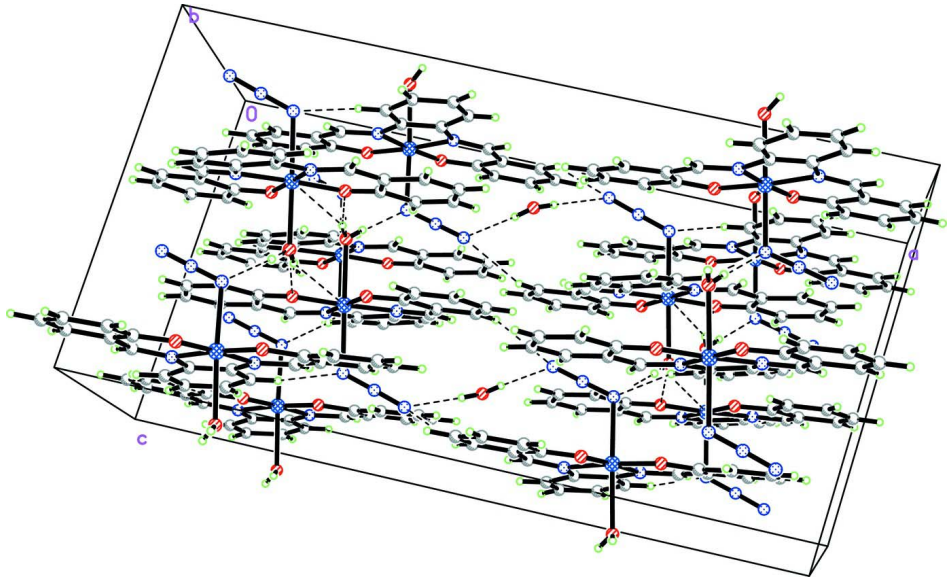

Figure 2

Three-dimensional network formed by hydrogen bonds (dashed lines).

Aquaazido{2,2′-[*o*- phenylenebis(nitrilomethylidyne)]diphenolato}manganese(III) hemihydrate

Crystal data

$[Mn(C_{20}H_{14}N_2O_2)(N_3)(H_2O)]\cdot 0.5H_2O$
$M_r = 438.33$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 25.10\ (1)$ Å
$b = 11.478\ (5)$ Å
$c = 12.599\ (5)$ Å
$\beta = 94.175\ (3)°$
$V = 3620\ (3)$ Å^3
$Z = 8$

$F(000) = 1808$
$D_x = 1.612$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71070$ Å
Cell parameters from 3162 reflections
$\theta = 3.0–25.0°$
$\mu = 0.77$ mm^{-1}
$T = 293$ K
Block, pink
0.12 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min} = 0.914$, $T_{max} = 0.941$
11927 measured reflections
3162 independent reflections
2371 reflections with $I > 2\sigma(I)$
$R_{int} = 0.082$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.0°$
$h = -27\rightarrow29$
$k = -13\rightarrow13$
$l = -14\rightarrow13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.086$
$S = 1.00$
3162 reflections
280 parameters
4 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
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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}
Mn1	0.204685 (15)	0.09791 (3)	0.87813 (3)	0.00903 (13)
C1	0.11631 (10)	0.2543 (2)	0.90445 (18)	0.0102 (6)
C2	0.09780 (10)	0.3688 (2)	0.90882 (19)	0.0124 (6)
H2	0.1219	0.4301	0.9061	0.015*
C3	0.04451 (10)	0.3928 (2)	0.91706 (19)	0.0156 (6)
H3	0.0332	0.4699	0.9196	0.019*
C4	0.00719 (10)	0.3029 (2)	0.9217 (2)	0.0180 (6)
H4	−0.0288	0.3197	0.9260	0.022*
C5	0.02444 (10)	0.1897 (2)	0.9198 (2)	0.0161 (6)
H5	−0.0002	0.1297	0.9238	0.019*
C6	0.07880 (10)	0.1623 (2)	0.91183 (18)	0.0110 (6)

C7	0.09346 (10)	0.0426 (2)	0.91314 (18)	0.0114 (6)
H7	0.0662	−0.0115	0.9188	0.014*
C8	0.15275 (10)	−0.1198 (2)	0.91053 (18)	0.0092 (6)
C9	0.11626 (10)	−0.2052 (2)	0.93648 (19)	0.0123 (6)
H9	0.0819	−0.1844	0.9525	0.015*
C10	0.13142 (10)	−0.3207 (2)	0.93819 (18)	0.0123 (6)
H10	0.1071	−0.3777	0.9553	0.015*
C11	0.18272 (10)	−0.3529 (2)	0.91454 (18)	0.0124 (6)
H11	0.1925	−0.4311	0.9157	0.015*
C12	0.21902 (10)	−0.2690 (2)	0.88938 (18)	0.0109 (6)
H12	0.2533	−0.2906	0.8736	0.013*
C13	0.20450 (10)	−0.1518 (2)	0.88751 (18)	0.0096 (5)
C14	0.28922 (10)	−0.0719 (2)	0.84750 (19)	0.0110 (6)
H14	0.3007	−0.1480	0.8385	0.013*
C15	0.32741 (10)	0.0188 (2)	0.83746 (18)	0.0111 (6)
C16	0.38034 (10)	−0.0164 (2)	0.82023 (18)	0.0145 (6)
H16	0.3878	−0.0954	0.8143	0.017*
C17	0.42059 (10)	0.0627 (2)	0.81212 (19)	0.0155 (6)
H17	0.4548	0.0377	0.7998	0.019*
C18	0.40976 (10)	0.1809 (2)	0.82255 (18)	0.0142 (6)
H18	0.4371	0.2350	0.8181	0.017*
C19	0.35893 (10)	0.2184 (2)	0.83938 (19)	0.0136 (6)
H19	0.3525	0.2978	0.8462	0.016*
C20	0.31675 (10)	0.1395 (2)	0.84649 (18)	0.0094 (6)
N1	0.23943 (8)	−0.05699 (18)	0.86829 (15)	0.0098 (5)
N2	0.14151 (8)	0.00196 (18)	0.90708 (15)	0.0092 (5)
N3	0.17896 (8)	0.07150 (19)	0.70029 (16)	0.0131 (5)
N4	0.13255 (9)	0.05323 (19)	0.67637 (16)	0.0136 (5)
N5	0.08768 (9)	0.0341 (2)	0.65186 (17)	0.0234 (6)
O1	0.16772 (7)	0.23708 (15)	0.89293 (13)	0.0127 (4)
O2	0.26797 (6)	0.18073 (15)	0.85790 (12)	0.0113 (4)
O1W	0.23229 (7)	0.08568 (17)	1.05761 (13)	0.0138 (4)
O2W	0.0000	−0.0960 (3)	0.7500	0.0383 (8)
H1W	0.2366 (10)	0.1525 (7)	1.0796 (16)	0.023 (9)*
H2W	0.2189 (11)	0.0413 (15)	1.0991 (14)	0.037 (10)*
H3W	0.0224 (11)	−0.056 (3)	0.723 (3)	0.064 (13)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0082 (2)	0.0069 (2)	0.0122 (2)	−0.00022 (17)	0.00244 (16)	−0.00015 (16)
C1	0.0133 (14)	0.0136 (14)	0.0037 (13)	0.0011 (11)	0.0004 (10)	0.0013 (11)
C2	0.0163 (14)	0.0085 (14)	0.0124 (14)	0.0000 (11)	0.0008 (11)	0.0017 (11)
C3	0.0190 (15)	0.0117 (15)	0.0162 (15)	0.0046 (12)	0.0017 (12)	0.0029 (11)
C4	0.0105 (14)	0.0172 (16)	0.0271 (16)	0.0050 (12)	0.0059 (12)	0.0031 (13)
C5	0.0130 (14)	0.0117 (15)	0.0239 (16)	−0.0027 (11)	0.0035 (12)	0.0033 (12)
C6	0.0149 (14)	0.0083 (14)	0.0101 (14)	0.0023 (11)	0.0031 (11)	0.0004 (11)
C7	0.0121 (14)	0.0118 (15)	0.0105 (14)	−0.0045 (11)	0.0024 (11)	−0.0004 (11)

C8	0.0129 (14)	0.0080 (14)	0.0064 (13)	−0.0001 (11)	−0.0005 (10)	−0.0019 (10)
C9	0.0119 (14)	0.0114 (15)	0.0138 (14)	−0.0013 (11)	0.0035 (11)	−0.0016 (11)
C10	0.0159 (14)	0.0119 (15)	0.0092 (14)	−0.0046 (11)	0.0014 (11)	0.0001 (11)
C11	0.0205 (15)	0.0067 (14)	0.0093 (13)	0.0021 (11)	−0.0028 (11)	0.0002 (11)
C12	0.0129 (14)	0.0145 (15)	0.0052 (13)	0.0043 (11)	0.0005 (10)	−0.0027 (10)
C13	0.0124 (14)	0.0120 (14)	0.0042 (13)	−0.0028 (11)	−0.0007 (10)	−0.0015 (11)
C14	0.0142 (14)	0.0106 (15)	0.0082 (13)	0.0035 (11)	0.0011 (11)	0.0006 (10)
C15	0.0128 (14)	0.0144 (14)	0.0062 (13)	−0.0007 (11)	0.0016 (10)	0.0010 (11)
C16	0.0170 (15)	0.0162 (15)	0.0104 (14)	0.0038 (12)	0.0021 (11)	0.0012 (11)
C17	0.0069 (14)	0.0278 (17)	0.0119 (14)	0.0028 (12)	0.0011 (11)	0.0015 (12)
C18	0.0107 (14)	0.0240 (17)	0.0077 (14)	−0.0048 (12)	−0.0008 (11)	0.0009 (12)
C19	0.0193 (15)	0.0119 (15)	0.0095 (14)	−0.0023 (12)	−0.0006 (11)	−0.0023 (11)
C20	0.0082 (13)	0.0175 (15)	0.0026 (12)	0.0006 (11)	0.0005 (10)	0.0020 (11)
N1	0.0135 (12)	0.0086 (12)	0.0073 (11)	0.0005 (9)	0.0014 (9)	0.0004 (9)
N2	0.0118 (11)	0.0070 (12)	0.0088 (11)	0.0010 (9)	0.0018 (9)	0.0002 (9)
N3	0.0097 (12)	0.0190 (14)	0.0108 (12)	−0.0020 (9)	0.0013 (9)	0.0014 (9)
N4	0.0198 (14)	0.0138 (13)	0.0078 (12)	0.0027 (10)	0.0047 (10)	0.0002 (9)
N5	0.0122 (13)	0.0395 (17)	0.0183 (13)	0.0007 (12)	−0.0002 (10)	0.0000 (11)
O1	0.0099 (9)	0.0074 (10)	0.0213 (10)	−0.0001 (7)	0.0038 (7)	−0.0005 (8)
O2	0.0114 (9)	0.0097 (10)	0.0133 (10)	−0.0007 (8)	0.0037 (7)	0.0011 (8)
O1W	0.0192 (11)	0.0090 (11)	0.0133 (10)	−0.0039 (8)	0.0027 (8)	−0.0004 (9)
O2W	0.025 (2)	0.028 (2)	0.063 (2)	0.000	0.0130 (18)	0.000

Geometric parameters (Å, º)

Mn1—O1	1.8636 (18)	C10—H10	0.9300
Mn1—O2	1.8844 (18)	C11—C12	1.379 (3)
Mn1—N2	1.986 (2)	C11—H11	0.9300
Mn1—N1	1.988 (2)	C12—C13	1.393 (3)
Mn1—N3	2.306 (2)	C12—H12	0.9300
Mn1—O1W	2.321 (2)	C13—N1	1.430 (3)
C1—O1	1.324 (3)	C14—N1	1.307 (3)
C1—C2	1.397 (3)	C14—C15	1.427 (3)
C1—C6	1.422 (3)	C14—H14	0.9300
C2—C3	1.377 (3)	C15—C20	1.418 (4)
C2—H2	0.9300	C15—C16	1.420 (3)
C3—C4	1.398 (4)	C16—C17	1.367 (4)
C3—H3	0.9300	C16—H16	0.9300
C4—C5	1.371 (4)	C17—C18	1.392 (4)
C4—H4	0.9300	C17—H17	0.9300
C5—C6	1.411 (3)	C18—C19	1.378 (3)
C5—H5	0.9300	C18—H18	0.9300
C6—C7	1.422 (4)	C19—C20	1.401 (3)
C7—N2	1.301 (3)	C19—H19	0.9300
C7—H7	0.9300	C20—O2	1.330 (3)
C8—C9	1.397 (3)	N3—N4	1.200 (3)
C8—C13	1.401 (3)	N4—N5	1.167 (3)
C8—N2	1.425 (3)	O1W—H1W	0.820 (11)

C9—C10	1.379 (4)	O1W—H2W	0.82 (2)
C9—H9	0.9300	O2W—H3W	0.82 (3)
C10—C11	1.393 (3)		

O1—Mn1—O2	90.68 (8)	C11—C10—H10	119.7
O1—Mn1—N2	92.69 (8)	C12—C11—C10	120.0 (2)
O2—Mn1—N2	175.37 (8)	C12—C11—H11	120.0
O1—Mn1—N1	175.33 (8)	C10—C11—H11	120.0
O2—Mn1—N1	93.71 (8)	C11—C12—C13	120.1 (2)
N2—Mn1—N1	82.83 (9)	C11—C12—H12	119.9
O1—Mn1—N3	95.95 (8)	C13—C12—H12	119.9
O2—Mn1—N3	96.55 (7)	C12—C13—C8	119.7 (2)
N2—Mn1—N3	86.26 (8)	C12—C13—N1	125.1 (2)
N1—Mn1—N3	85.12 (8)	C8—C13—N1	115.1 (2)
O1—Mn1—O1W	94.00 (7)	N1—C14—C15	125.5 (2)
O2—Mn1—O1W	88.14 (7)	N1—C14—H14	117.2
N2—Mn1—O1W	88.47 (7)	C15—C14—H14	117.2
N1—Mn1—O1W	84.58 (7)	C20—C15—C16	118.3 (2)
N3—Mn1—O1W	168.94 (7)	C20—C15—C14	125.0 (2)
O1—C1—C2	118.3 (2)	C16—C15—C14	116.6 (2)
O1—C1—C6	123.5 (2)	C17—C16—C15	121.8 (3)
C2—C1—C6	118.2 (2)	C17—C16—H16	119.1
C3—C2—C1	121.3 (2)	C15—C16—H16	119.1
C3—C2—H2	119.4	C16—C17—C18	119.3 (2)
C1—C2—H2	119.4	C16—C17—H17	120.3
C2—C3—C4	120.9 (3)	C18—C17—H17	120.3
C2—C3—H3	119.6	C19—C18—C17	120.6 (2)
C4—C3—H3	119.6	C19—C18—H18	119.7
C5—C4—C3	119.1 (2)	C17—C18—H18	119.7
C5—C4—H4	120.5	C18—C19—C20	121.3 (3)
C3—C4—H4	120.5	C18—C19—H19	119.3
C4—C5—C6	121.4 (2)	C20—C19—H19	119.3
C4—C5—H5	119.3	O2—C20—C19	118.9 (2)
C6—C5—H5	119.3	O2—C20—C15	122.5 (2)
C5—C6—C1	119.2 (2)	C19—C20—C15	118.6 (2)
C5—C6—C7	117.7 (2)	C14—N1—C13	122.8 (2)
C1—C6—C7	123.1 (2)	C14—N1—Mn1	124.04 (18)
N2—C7—C6	125.9 (2)	C13—N1—Mn1	113.16 (16)
N2—C7—H7	117.1	C7—N2—C8	122.2 (2)
C6—C7—H7	117.1	C7—N2—Mn1	124.61 (18)
C9—C8—C13	119.9 (2)	C8—N2—Mn1	112.96 (15)
C9—C8—N2	124.3 (2)	N4—N3—Mn1	117.69 (16)
C13—C8—N2	115.8 (2)	N5—N4—N3	178.8 (3)
C10—C9—C8	119.6 (2)	C1—O1—Mn1	129.44 (16)
C10—C9—H9	120.2	C20—O2—Mn1	128.82 (16)
C8—C9—H9	120.2	Mn1—O1W—H1W	107.2 (17)
C9—C10—C11	120.7 (2)	Mn1—O1W—H2W	123.5 (18)
C9—C10—H10	119.7	H1W—O1W—H2W	114.6 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H2*W*···N3^{i}	0.82 (2)	2.12 (2)	2.937 (3)	176 (3)
O1*W*—H1*W*···O2^{ii}	0.82 (1)	2.08 (1)	2.885 (3)	169 (2)
O2*W*—H3*W*···N5	0.82 (3)	2.18 (3)	3.000 (3)	173 (4)

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