Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Triaqua[2-(carboxylatomethylimino-methyl)-4-formylphenolato-κ^3O,N,O']-manganese(II) monohydrate

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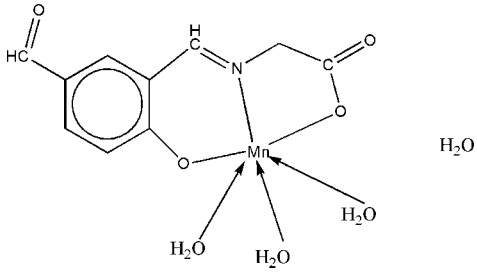
Received 2 May 2007; accepted 3 May 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.039; wR factor = 0.100; data-to-parameter ratio = 14.6.

The Mn atom in the title compound, $[Mn(C_{10}H_7NO_4)(H_2O)_3]\cdot H_2O$, adopts an octahedral geometry owing to N,O,O'-tridentate chelation by the planar dianionic ligand. Intermolecular hydrogen bonds form a three-dimensional framework.

Related literature

For metal complexes of Schiff bases derived from 5-formylsalicylaldehyde, see: Zeng *et al.* (2003); Liu *et al.* (2006); Cai *et al.* (2006*a*,*b*). For related literature, see: Reddy *et al.* (2004); Wang *et al.* (1999).



Experimental

Crystal data

$[Mn(C_{10}H_7NO_4)(H_2O)_3]\cdot H_2O$
M_r = 332.17
Orthorhombic, *Pbca*
a = 11.208 (5) Å
b = 7.890 (3) Å
c = 31.212 (13) Å
V = 2760 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.99 mm^{-1}
T = 293 (2) K
0.20 × 0.15 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.836, T_{max} = 0.952
12431 measured reflections
3016 independent reflections
2197 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.100
S = 1.04
2999 reflections
205 parameters
12 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7A···O3^{i}	0.836 (9)	1.928 (11)	2.758 (3)	172 (3)
O6—H6B···O4^{ii}	0.840 (9)	2.281 (17)	3.014 (3)	146 (2)
O6—H6A···O4^{i}	0.847 (9)	1.831 (10)	2.677 (3)	177 (3)
O5—H5B···O1^{iii}	0.843 (10)	1.886 (11)	2.723 (3)	172 (2)
O8—H8B···O2^{iv}	0.844 (10)	1.997 (13)	2.793 (3)	157 (3)
O5—H5A···O8^{v}	0.847 (10)	1.833 (10)	2.669 (3)	169 (3)
O7—H7B···O4^{vi}	0.848 (10)	1.904 (11)	2.746 (3)	172 (3)
O8—H8A···O7	0.843 (10)	2.087 (13)	2.900 (3)	162 (3)

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x-\frac{1}{2}, y, -z+\frac{1}{2}$; (iii) $-x+\frac{1}{2}, y-\frac{1}{2}, z$; (iv) $-x+1, -y+1, -z+1$; (v) $x, y-1, z$; (vi) $-x+\frac{3}{2}, y+\frac{1}{2}, z$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

This work was supported by the Ministry of Education Foundation of the Guangxi Chuang Autonomous Region of the People's Republic of China. We also thank Hechi University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2264).

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 doi:10.1107/S1600536807021824

supplementary materials

Acta Cryst. (2007). E**63**, m1631 [**doi:10.1107/S1600536807021824**]

Triaqua[2-(carboxylatomethyliminomethyl)-4-formylphenolato-κ^3O,N,O']manganese(II) monohydrate

J.-H. Cai

Comment

Several crystal structures of metal complexes of salicylaldehyde–amino acids have reported (Wang *et al.*,1999; Reddy *et al.*, 2004). The present study follows studies on the complexes of the Schiff bases derived from 5-formylsalicylaldehyde derivative (Liu *et al.*, 2006; Cai *et al.*, 2006*a* 2006*b*).

The title manganese complex (I) is chelated by the 5-formysalicylideneglycinate anion; it is also coordinated by three water molecules. The mononuclear molecule interacts with the lattice water molecule through hydrongen bonds (Table 1) to give rise to a three-dimensional, hydrogen-bonded network.

Experimental

5-Formylsalicylaldehyde (0.2 mmol, 0.268 g), glycine (0.2 mmol, 0.15 g) and potassium hydroxide (0.2 mmol, 0.112 g) were dissolved in aqueous methanol (80% 15 ml) to give a clear yellow solution. To the solution was added an aqueous solution (10 ml) of nanganese sulfate heptahydrate (1 mmol, 0.28 g). The mixture was heated at 323 K for 2 h. Brown crystals separated from the solution after several days.

Refinement

Water H atoms were located in a difference Fourier map and refined with O–H distance restraints of 0.85 (1) Å, and with U_{iso}(H) = 1.5U_{eq}(O). Other H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, and refined in the riding-model approximation with U_{iso}(H) = 1.2U_{eq}(C).

Figures

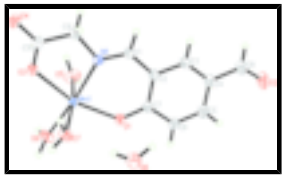

Fig. 1. The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

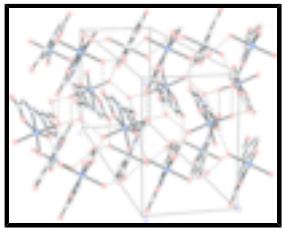

Fig. 2. Packing of (I). Hydrogen bonds are shown as dotted lines.

Triaqua[2-(carboxylatomethyliminomethyl)-4-formylphenolato- κ^3O,N,O']manganese(II) monohydrate

Crystal data

$[Mn(C_{10}H_7NO_4)(H_2O)_3]\cdot H_2O$	F_{000} = 1368
M_r = 332.17	D_x = 1.599 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 887 reflections
a = 11.208 (5) Å	θ = 3.2–25.8º
b = 7.890 (3) Å	μ = 0.99 mm^{-1}
c = 31.212 (13) Å	*T* = 293 (2) K
V = 2760 (2) Å^3	Layer, brown
Z = 8	0.20 × 0.15 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer	3016 independent reflections
Radiation source: fine-focus sealed tube	2197 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.050
T = 293(2) K	θ_{max} = 27.0º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Bruker, 1998)	*h* = −5→14
T_{min} = 0.836, T_{max} = 0.952	*k* = −9→10
12431 measured reflections	*l* = −39→38

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.100	$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.4197P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.001
2999 reflections	$\Delta\rho_{max}$ = 0.34 e Å^{-3}
205 parameters	$\Delta\rho_{min}$ = −0.52 e Å^{-3}
12 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Mn1	0.43496 (3)	0.08299 (5)	0.321939 (11)	0.02955 (13)
N1	0.58315 (17)	0.0511 (3)	0.36816 (6)	0.0309 (5)
O1	0.36278 (15)	0.2373 (2)	0.37017 (5)	0.0396 (4)
O2	0.3893 (2)	0.4050 (3)	0.56880 (7)	0.0679 (7)
O3	0.56775 (15)	−0.0691 (2)	0.28804 (5)	0.0370 (4)
O4	0.74624 (15)	−0.1884 (3)	0.28876 (5)	0.0437 (5)
O5	0.35268 (18)	−0.1504 (3)	0.34151 (8)	0.0572 (6)
O6	0.2977 (2)	0.0909 (3)	0.27446 (6)	0.0543 (6)
O7	0.50884 (16)	0.3129 (2)	0.29047 (5)	0.0393 (4)
O8	0.5092 (2)	0.5967 (3)	0.34947 (7)	0.0630 (6)
C1	0.5816 (2)	0.0875 (3)	0.40768 (8)	0.0343 (6)
H1	0.6456	0.0493	0.4240	0.041*
C2	0.4904 (2)	0.1827 (3)	0.43006 (7)	0.0339 (6)
C3	0.5064 (2)	0.2088 (4)	0.47405 (8)	0.0412 (6)
H3	0.5726	0.1604	0.4872	0.049*
C4	0.4289 (2)	0.3029 (4)	0.49883 (8)	0.0419 (7)
C5	0.3320 (3)	0.3787 (4)	0.47888 (8)	0.0458 (7)
H5	0.2802	0.4456	0.4949	0.055*
C6	0.3115 (2)	0.3566 (4)	0.43600 (9)	0.0447 (7)
H6	0.2458	0.4089	0.4235	0.054*
C7	0.3878 (2)	0.2563 (3)	0.41017 (8)	0.0334 (6)
C8	0.4519 (3)	0.3259 (4)	0.54424 (9)	0.0512 (8)
H8	0.5203	0.2758	0.5554	0.061*
C9	0.6851 (2)	−0.0454 (3)	0.35156 (8)	0.0365 (6)
H9A	0.6995	−0.1423	0.3700	0.044*
H9B	0.7557	0.0256	0.3522	0.044*
C10	0.6639 (2)	−0.1067 (3)	0.30606 (8)	0.0322 (5)
H5A	0.3944 (18)	−0.240 (2)	0.3436 (9)	0.048*
H5B	0.2833 (11)	−0.177 (3)	0.3495 (9)	0.048*
H6A	0.287 (2)	0.161 (2)	0.2543 (6)	0.048*
H6B	0.268 (2)	−0.0012 (18)	0.2664 (7)	0.048*
H7A	0.4847 (19)	0.338 (4)	0.2659 (5)	0.048*
H7B	0.5840 (9)	0.303 (4)	0.2890 (8)	0.048*
H8A	0.498 (3)	0.505 (2)	0.3363 (7)	0.048*
H8B	0.527 (3)	0.573 (3)	0.3750 (4)	0.048*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0266 (2)	0.0350 (2)	0.0270 (2)	0.00148 (16)	−0.00204 (15)	−0.00171 (16)
N1	0.0250 (10)	0.0373 (12)	0.0304 (11)	0.0034 (9)	−0.0014 (8)	−0.0044 (9)
O1	0.0365 (10)	0.0508 (11)	0.0316 (9)	0.0126 (9)	−0.0046 (7)	−0.0073 (8)
O2	0.0706 (15)	0.0929 (18)	0.0402 (12)	0.0036 (13)	0.0077 (11)	−0.0221 (12)
O3	0.0323 (9)	0.0491 (11)	0.0297 (9)	0.0066 (8)	−0.0034 (7)	−0.0042 (8)

O4	0.0293 (9)	0.0582 (13)	0.0436 (10)	0.0083 (9)	0.0021 (8)	−0.0170 (9)
O5	0.0363 (11)	0.0437 (12)	0.0916 (16)	−0.0060 (10)	0.0136 (12)	0.0115 (11)
O6	0.0614 (14)	0.0505 (13)	0.0509 (12)	−0.0136 (11)	−0.0297 (10)	0.0098 (10)
O7	0.0332 (10)	0.0478 (12)	0.0368 (10)	−0.0038 (9)	−0.0013 (8)	0.0088 (9)
O8	0.0815 (17)	0.0497 (13)	0.0577 (14)	0.0077 (12)	−0.0120 (13)	−0.0068 (11)
C1	0.0308 (13)	0.0387 (14)	0.0334 (13)	0.0039 (11)	−0.0066 (10)	−0.0031 (11)
C2	0.0339 (13)	0.0374 (14)	0.0303 (13)	0.0016 (11)	0.0000 (11)	−0.0047 (11)
C3	0.0436 (15)	0.0496 (17)	0.0305 (13)	0.0065 (14)	−0.0049 (11)	−0.0021 (12)
C4	0.0445 (15)	0.0485 (17)	0.0327 (14)	−0.0023 (13)	0.0037 (12)	−0.0056 (12)
C5	0.0405 (15)	0.0565 (18)	0.0403 (15)	0.0018 (14)	0.0094 (12)	−0.0130 (13)
C6	0.0351 (14)	0.0521 (17)	0.0468 (16)	0.0111 (13)	0.0011 (12)	−0.0100 (13)
C7	0.0323 (13)	0.0350 (14)	0.0328 (13)	−0.0020 (11)	0.0014 (10)	−0.0026 (11)
C8	0.0561 (18)	0.064 (2)	0.0334 (15)	−0.0015 (16)	0.0039 (13)	−0.0096 (14)
C9	0.0294 (13)	0.0443 (15)	0.0357 (13)	0.0062 (12)	−0.0038 (11)	−0.0061 (11)
C10	0.0306 (13)	0.0311 (13)	0.0347 (13)	−0.0055 (11)	0.0036 (10)	−0.0028 (10)

Geometric parameters (Å, °)

Mn1—O1	2.0982 (18)	O8—H8A	0.843 (10)
Mn1—O6	2.1367 (19)	O8—H8B	0.844 (10)
Mn1—O5	2.148 (2)	C1—C2	1.448 (3)
Mn1—O3	2.1851 (18)	C1—H1	0.9300
Mn1—N1	2.215 (2)	C2—C3	1.400 (3)
Mn1—O7	2.2227 (19)	C2—C7	1.431 (4)
N1—C1	1.267 (3)	C3—C4	1.380 (4)
N1—C9	1.467 (3)	C3—H3	0.9300
O1—C7	1.288 (3)	C4—C5	1.387 (4)
O2—C8	1.213 (4)	C4—C8	1.452 (4)
O3—C10	1.252 (3)	C5—C6	1.369 (4)
O4—C10	1.249 (3)	C5—H5	0.9300
O5—H5A	0.847 (10)	C6—C7	1.417 (4)
O5—H5B	0.843 (10)	C6—H6	0.9300
O6—H6A	0.847 (9)	C8—H8	0.9300
O6—H6B	0.840 (9)	C9—C10	1.519 (3)
O7—H7A	0.836 (9)	C9—H9A	0.9700
O7—H7B	0.848 (10)	C9—H9B	0.9700
O1—Mn1—O6	101.72 (9)	C2—C1—H1	116.7
O1—Mn1—O5	97.34 (9)	C3—C2—C7	118.0 (2)
O6—Mn1—O5	85.02 (9)	C3—C2—C1	117.3 (2)
O1—Mn1—O3	158.23 (6)	C7—C2—C1	124.7 (2)
O6—Mn1—O3	99.81 (8)	C4—C3—C2	123.2 (3)
O5—Mn1—O3	87.67 (8)	C4—C3—H3	118.4
O1—Mn1—N1	83.55 (7)	C2—C3—H3	118.4
O6—Mn1—N1	174.32 (8)	C3—C4—C5	118.3 (2)
O5—Mn1—N1	92.25 (9)	C3—C4—C8	120.1 (3)
O3—Mn1—N1	75.06 (7)	C5—C4—C8	121.6 (3)
O1—Mn1—O7	89.27 (8)	C6—C5—C4	121.0 (3)
O6—Mn1—O7	86.44 (8)	C6—C5—H5	119.5
O5—Mn1—O7	170.11 (8)	C4—C5—H5	119.5

O3—Mn1—O7	88.87 (7)	C5—C6—C7	121.8 (3)
N1—Mn1—O7	95.81 (7)	C5—C6—H6	119.1
C1—N1—C9	118.2 (2)	C7—C6—H6	119.1
C1—N1—Mn1	126.74 (17)	O1—C7—C6	119.0 (2)
C9—N1—Mn1	114.38 (14)	O1—C7—C2	123.2 (2)
C7—O1—Mn1	132.77 (16)	C6—C7—C2	117.7 (2)
C10—O3—Mn1	119.94 (16)	O2—C8—C4	125.3 (3)
Mn1—O5—H5A	119.8 (17)	O2—C8—H8	117.3
Mn1—O5—H5B	133.8 (17)	C4—C8—H8	117.3
H5A—O5—H5B	106.4 (15)	N1—C9—C10	111.97 (19)
Mn1—O6—H6A	129.7 (17)	N1—C9—H9A	109.2
Mn1—O6—H6B	118.2 (17)	C10—C9—H9A	109.2
H6A—O6—H6B	106.6 (14)	N1—C9—H9B	109.2
Mn1—O7—H7A	119 (2)	C10—C9—H9B	109.2
Mn1—O7—H7B	108.5 (19)	H9A—C9—H9B	107.9
H7A—O7—H7B	107.1 (15)	O4—C10—O3	124.4 (2)
H8A—O8—H8B	107.6 (15)	O4—C10—C9	117.0 (2)
N1—C1—C2	126.7 (2)	O3—C10—C9	118.6 (2)
N1—C1—H1	116.7		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H7A···O3^{i}	0.836 (9)	1.928 (11)	2.758 (3)	172 (3)
O6—H6B···O4^{ii}	0.840 (9)	2.281 (17)	3.014 (3)	146 (2)
O6—H6A···O4^{i}	0.847 (9)	1.831 (10)	2.677 (3)	177 (3)
O5—H5B···O1^{iii}	0.843 (10)	1.886 (11)	2.723 (3)	172 (2)
O8—H8B···O2^{iv}	0.844 (10)	1.997 (13)	2.793 (3)	157 (3)
O5—H5A···O8^{v}	0.847 (10)	1.833 (10)	2.669 (3)	169 (3)
O7—H7B···O4^{vi}	0.848 (10)	1.904 (11)	2.746 (3)	172 (3)
O8—H8A···O7	0.843 (10)	2.087 (13)	2.900 (3)	162 (3)

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) *x*−1/2, *y*, −*z*+1/2; (iii) −*x*+1/2, *y*−1/2, *z*; (iv) −*x*+1, −*y*+1, −*z*+1; (v) *x*, *y*−1, *z*; (vi) −*x*+3/2, *y*+1/2, *z*.

Fig. 1

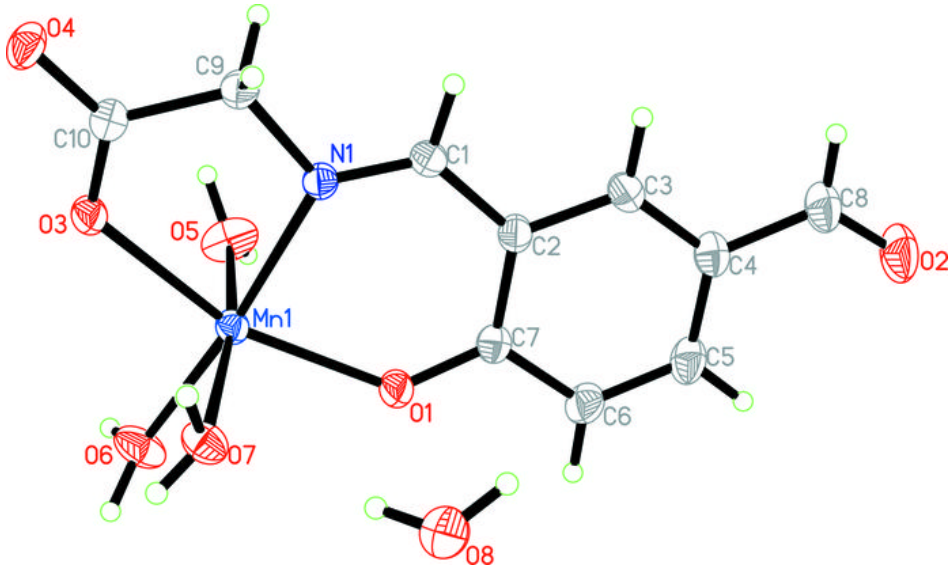

Fig. 2