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Tetraaquabis[2-(thiosemicarbazonomethyl)benzenesulfonato]manganese(II)

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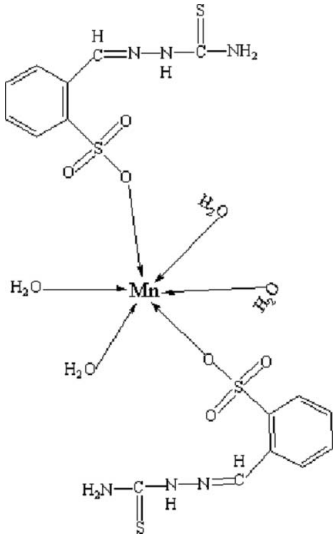
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.028; wR factor = 0.077; data-to-parameter ratio = 13.0.

In the title compound, $[Mn(C_8H_8N_3O_3S_2)_2(H_2O)_4]$, the Mn^{II} atom (site symmetry $\overline{1}$) adopts a slightly distorted octahedral MnO_6 geometry. The molecular conformation is supported by N—H···N and O—H···O hydrogen bonds. In the crystal, molecules interact by O—H···O, O—H···S, N—H···O and N—H···S hydrogen bonds, thereby forming (011) sheets.

Related literature

For background to coordination networks, see: Ranford *et al.* (1998).



Experimental

Crystal data

$[Mn(C_8H_8N_3O_3S_2)_2(H_2O)_4]$
M_r = 643.59
Triclinic, $P\overline{1}$
a = 6.8096 (6) Å
b = 9.5498 (8) Å
c = 10.7898 (9) Å
α = 64.386 (1)°
β = 88.495 (1)°
γ = 83.791 (1)°
V = 628.83 (9) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.92 mm^{-1}
T = 273 K
0.19 × 0.14 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.845, T_{max} = 0.898
3329 measured reflections
2207 independent reflections
2005 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.077
S = 1.07
2207 reflections
170 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Selected bond lengths (Å).

Mn1—O4	2.1369 (17)	Mn1—O1	2.2166 (14)
Mn1—O5	2.1495 (16)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N3	0.86	2.29	2.638 (3)	104
O4—H10···O3	0.85	2.02	2.761 (3)	146
N1—H1B···O3^{i}	0.86	2.33	2.986 (3)	134
N2—H2···S2^{ii}	0.86	2.57	3.4231 (19)	170
O4—H9···S2^{iii}	0.85	2.36	3.182 (2)	162
O5—H11···S2^{iv}	0.85	2.46	3.2603 (19)	156
O5—H12···O2^{v}	0.85	1.87	2.712 (3)	172

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; 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: HB5192).

References

Bruker (2000). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Ranford, J. D., Vittal, J. J. & Wang, Y. M. (1998). *Inorg. Chem.* **37**, 1226–1231.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2009). E**65**, m1491 [https://doi.org/10.1107/S160053680904495X]

Tetraaquabis[2-(thiosemicarbazonomethyl)benzenesulfonato]manganese(II)

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S1. Experimental

A solution of 1.0 mmol 2-formyl-benzenesulfonate-thiosemicarbazide was added to a solution of 0.5 mmol $MnCl_2.4H_2O$ in 5 ml ethanol at room temperature. The mixture was refluxed for 4 h with stirring, then the resulting precipitate was filtered, washed, and dried *in vacuo* over P_4O_{10} for 48 h. Pink blocks of (I) were obtained by slowly evaporating from methanol at room temperature.

S2. Refinement

The H atoms were positioned geometrically (C—H = 0.93, N—H = 0.86, O—H = 0.85Å) and refined as riding with U_{iso}(H)= 1.2 U_{eq}(C, N) or 1.5U_{eq}(O).

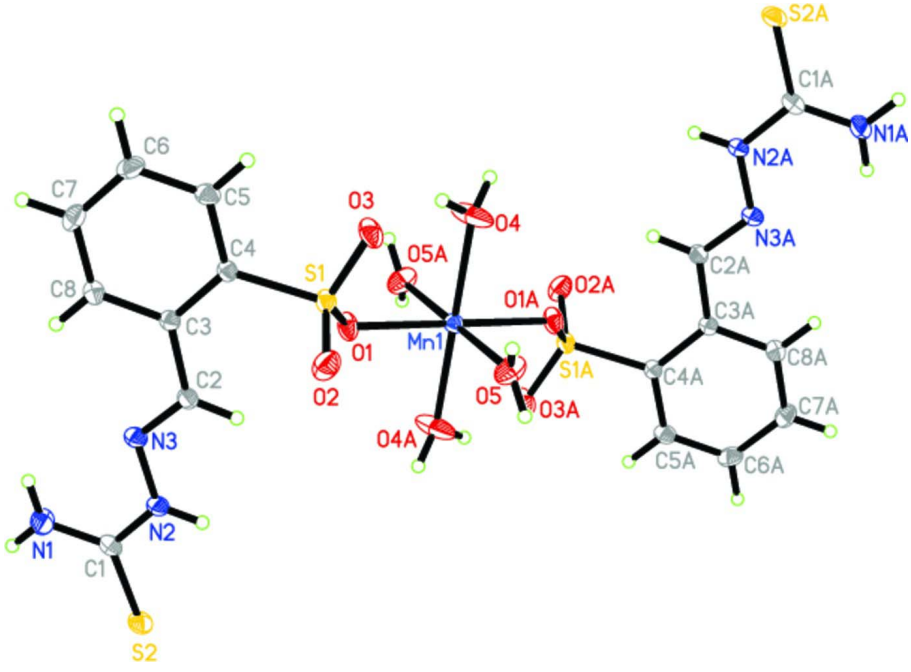

Figure 1

The molecular structure of (I) showing 30% displacement ellipsoids. Atoms with the suffix A are generated by the symmetry operation (1-x, 1-y, -z).

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$\alpha = 64.386$ (1)°
$\beta = 88.495$ (1)°
$\gamma = 83.791$ (1)°
$V = 628.83$ (9) Å^3
$Z = 1$
$F(000) = 331$
$D_x = 1.700$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2123 reflections
$\theta = 2.4$–$28.2°$
$\mu = 0.92$ mm^{-1}
$T = 273$ K
Block, pink
0.19 × 0.14 × 0.12 mm

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Bruker SMART CCD diffractometer
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ω scans
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$T_{min} = 0.845$, $T_{max} = 0.898$
3329 measured reflections
2207 independent reflections
2005 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 25.1°$, $\theta_{min} = 2.1°$
$h = -8 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -9 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.077$
$S = 1.07$
2207 reflections
170 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
$w = 1/[\sigma^2(F_o^2) + (0.0384P)^2 + 0.3206P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.049 (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	0.5000	0.5000	0.0000	0.02389 (16)
S1	0.73935 (8)	0.67535 (6)	0.15413 (5)	0.02518 (16)

S2	1.00275 (9)	−0.13908 (6)	0.71686 (6)	0.03340 (18)
O1	0.6334 (2)	0.54631 (17)	0.16197 (15)	0.0333 (4)
O2	0.9517 (2)	0.6407 (2)	0.15616 (17)	0.0417 (4)
O3	0.6673 (3)	0.82183 (18)	0.04065 (15)	0.0386 (4)
O4	0.3965 (3)	0.7431 (2)	−0.0981 (2)	0.0652 (6)
H9	0.2884	0.7905	−0.1404	0.098*
H10	0.4415	0.7985	−0.0637	0.098*
O5	0.7698 (2)	0.5559 (2)	−0.11007 (18)	0.0433 (4)
H12	0.8605	0.4911	−0.1165	0.065*
H11	0.8157	0.6433	−0.1363	0.065*
N1	0.8828 (3)	0.0620 (2)	0.81969 (19)	0.0400 (5)
H1A	0.8452	0.1545	0.8118	0.048*
H1B	0.8919	−0.0160	0.8996	0.048*
N2	0.9104 (3)	0.16402 (19)	0.58726 (18)	0.0271 (4)
H2	0.9474	0.1543	0.5143	0.033*
N3	0.8342 (2)	0.30737 (19)	0.57900 (17)	0.0257 (4)
C1	0.9263 (3)	0.0400 (2)	0.7097 (2)	0.0269 (5)
C2	0.8091 (3)	0.4167 (2)	0.4577 (2)	0.0242 (4)
H2A	0.8437	0.3975	0.3821	0.029*
C3	0.7252 (3)	0.5734 (2)	0.4382 (2)	0.0216 (4)
C4	0.6849 (3)	0.6967 (2)	0.3074 (2)	0.0212 (4)
C5	0.6053 (3)	0.8429 (2)	0.2938 (2)	0.0284 (5)
H5A	0.5780	0.9236	0.2066	0.034*
C6	0.5664 (3)	0.8694 (3)	0.4078 (2)	0.0345 (5)
H6	0.5140	0.9679	0.3979	0.041*
C7	0.6056 (3)	0.7491 (3)	0.5373 (2)	0.0329 (5)
H7	0.5791	0.7667	0.6147	0.040*
C8	0.6835 (3)	0.6034 (3)	0.5523 (2)	0.0283 (5)
H8	0.7090	0.5235	0.6401	0.034*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0270 (3)	0.0197 (2)	0.0237 (3)	−0.00156 (17)	−0.00312 (17)	−0.00820 (19)
S1	0.0331 (3)	0.0220 (3)	0.0199 (3)	−0.0055 (2)	0.0001 (2)	−0.0080 (2)
S2	0.0437 (3)	0.0198 (3)	0.0306 (3)	0.0006 (2)	−0.0044 (2)	−0.0057 (2)
O1	0.0521 (10)	0.0266 (8)	0.0230 (8)	−0.0120 (7)	−0.0042 (7)	−0.0104 (6)
O2	0.0352 (9)	0.0540 (11)	0.0448 (10)	−0.0042 (8)	0.0067 (7)	−0.0301 (9)
O3	0.0603 (11)	0.0259 (8)	0.0221 (8)	−0.0100 (7)	−0.0032 (7)	−0.0020 (7)
O4	0.0675 (13)	0.0263 (9)	0.0932 (16)	0.0101 (9)	−0.0507 (12)	−0.0182 (10)
O5	0.0405 (10)	0.0403 (10)	0.0559 (11)	−0.0125 (8)	0.0177 (8)	−0.0262 (9)
N1	0.0639 (14)	0.0245 (10)	0.0246 (10)	−0.0001 (9)	0.0020 (9)	−0.0051 (8)
N2	0.0313 (9)	0.0206 (9)	0.0239 (9)	0.0028 (7)	−0.0010 (7)	−0.0056 (7)
N3	0.0269 (9)	0.0203 (9)	0.0267 (10)	−0.0011 (7)	−0.0023 (7)	−0.0074 (8)
C1	0.0250 (11)	0.0235 (11)	0.0261 (11)	−0.0033 (8)	−0.0033 (8)	−0.0047 (9)
C2	0.0263 (10)	0.0228 (10)	0.0225 (10)	−0.0017 (8)	−0.0020 (8)	−0.0091 (9)
C3	0.0206 (10)	0.0211 (10)	0.0238 (10)	−0.0031 (8)	−0.0027 (8)	−0.0100 (8)
C4	0.0217 (10)	0.0212 (10)	0.0216 (10)	−0.0043 (8)	−0.0016 (8)	−0.0095 (8)

C5	0.0320 (11)	0.0194 (10)	0.0308 (12)	−0.0016 (9)	−0.0024 (9)	−0.0080 (9)
C6	0.0375 (13)	0.0260 (12)	0.0450 (14)	0.0009 (10)	−0.0004 (10)	−0.0210 (11)
C7	0.0362 (12)	0.0367 (13)	0.0361 (12)	−0.0053 (10)	0.0022 (10)	−0.0250 (11)
C8	0.0306 (11)	0.0297 (11)	0.0240 (11)	−0.0039 (9)	−0.0013 (9)	−0.0108 (9)

Geometric parameters (Å, º)

Mn1—O4^{i}	2.1369 (17)	N1—H1B	0.8600
Mn1—O4	2.1369 (17)	N2—C1	1.337 (3)
Mn1—O5^{i}	2.1495 (16)	N2—N3	1.376 (2)
Mn1—O5	2.1495 (16)	N2—H2	0.8600
Mn1—O1	2.2166 (14)	N3—C2	1.274 (3)
Mn1—O1^{i}	2.2166 (14)	C2—C3	1.469 (3)
S1—O2	1.4468 (17)	C2—H2A	0.9300
S1—O3	1.4489 (16)	C3—C8	1.396 (3)
S1—O1	1.4647 (15)	C3—C4	1.403 (3)
S1—C4	1.7767 (19)	C4—C5	1.388 (3)
S2—C1	1.702 (2)	C5—C6	1.374 (3)
O4—H9	0.8500	C5—H5A	0.9300
O4—H10	0.8499	C6—C7	1.381 (3)
O5—H12	0.8499	C6—H6	0.9300
O5—H11	0.8499	C7—C8	1.376 (3)
N1—C1	1.313 (3)	C7—H7	0.9300
N1—H1A	0.8600	C8—H8	0.9300
O4^{i}—Mn1—O4	180.0	H1A—N1—H1B	120.0
O4^{i}—Mn1—O5^{i}	88.04 (8)	C1—N2—N3	119.42 (18)
O4—Mn1—O5^{i}	91.96 (8)	C1—N2—H2	120.3
O4^{i}—Mn1—O5	91.96 (8)	N3—N2—H2	120.3
O4—Mn1—O5	88.04 (8)	C2—N3—N2	115.48 (18)
O5^{i}—Mn1—O5	180.0	N1—C1—N2	118.31 (19)
O4^{i}—Mn1—O1	92.72 (7)	N1—C1—S2	122.79 (16)
O4—Mn1—O1	87.28 (7)	N2—C1—S2	118.89 (16)
O5^{i}—Mn1—O1	92.43 (6)	N3—C2—C3	119.54 (19)
O5—Mn1—O1	87.57 (6)	N3—C2—H2A	120.2
O4^{i}—Mn1—O1^{i}	87.28 (7)	C3—C2—H2A	120.2
O4—Mn1—O1^{i}	92.72 (7)	C8—C3—C4	117.81 (18)
O5^{i}—Mn1—O1^{i}	87.57 (6)	C8—C3—C2	119.86 (18)
O5—Mn1—O1^{i}	92.43 (6)	C4—C3—C2	122.33 (18)
O1—Mn1—O1^{i}	180.0	C5—C4—C3	120.37 (18)
O2—S1—O3	113.09 (10)	C5—C4—S1	117.47 (15)
O2—S1—O1	112.81 (10)	C3—C4—S1	122.14 (15)
O3—S1—O1	111.89 (9)	C6—C5—C4	120.6 (2)
O2—S1—C4	105.67 (9)	C6—C5—H5A	119.7
O3—S1—C4	106.74 (9)	C4—C5—H5A	119.7
O1—S1—C4	105.98 (9)	C5—C6—C7	119.6 (2)
S1—O1—Mn1	131.56 (9)	C5—C6—H6	120.2
Mn1—O4—H9	131.3	C7—C6—H6	120.2

Mn1—O4—H10	115.1	C8—C7—C6	120.3 (2)
H9—O4—H10	108.1	C8—C7—H7	119.8
Mn1—O5—H12	126.2	C6—C7—H7	119.8
Mn1—O5—H11	124.0	C7—C8—C3	121.2 (2)
H12—O5—H11	108.1	C7—C8—H8	119.4
C1—N1—H1A	120.0	C3—C8—H8	119.4
C1—N1—H1B	120.0		

O2—S1—O1—Mn1	−96.18 (14)	C8—C3—C4—S1	177.75 (15)
O3—S1—O1—Mn1	32.68 (16)	C2—C3—C4—S1	−2.0 (3)
C4—S1—O1—Mn1	148.66 (12)	O2—S1—C4—C5	115.31 (17)
O4^{i}—Mn1—O1—S1	138.91 (14)	O3—S1—C4—C5	−5.33 (19)
O4—Mn1—O1—S1	−41.09 (14)	O1—S1—C4—C5	−124.75 (17)
O5^{i}—Mn1—O1—S1	−132.94 (13)	O2—S1—C4—C3	−62.83 (18)
O5—Mn1—O1—S1	47.06 (13)	O3—S1—C4—C3	176.53 (15)
O1^{i}—Mn1—O1—S1	−25 (100)	O1—S1—C4—C3	57.11 (18)
C1—N2—N3—C2	−174.55 (18)	C3—C4—C5—C6	0.6 (3)
N3—N2—C1—N1	−5.3 (3)	S1—C4—C5—C6	−177.58 (17)
N3—N2—C1—S2	174.98 (14)	C4—C5—C6—C7	−0.5 (3)
N2—N3—C2—C3	178.94 (17)	C5—C6—C7—C8	0.2 (3)
N3—C2—C3—C8	3.4 (3)	C6—C7—C8—C3	0.1 (3)
N3—C2—C3—C4	−176.81 (18)	C4—C3—C8—C7	0.0 (3)
C8—C3—C4—C5	−0.3 (3)	C2—C3—C8—C7	179.78 (19)
C2—C3—C4—C5	179.89 (19)		

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···N3	0.86	2.29	2.638 (3)	104
O4—H10···O3	0.85	2.02	2.761 (3)	146
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Symmetry codes: (ii) *x*, *y*−1, *z*+1; (iii) −*x*+2, −*y*, −*z*+1; (iv) *x*−1, *y*+1, *z*−1; (v) *x*, *y*+1, *z*−1; (vi) −*x*+2, −*y*+1, −*z*.