

Bis[2-(2-pyridylmethyleneamino)-benzenesulfonato- κ^3N,N',O]-manganese(II) dihydrate

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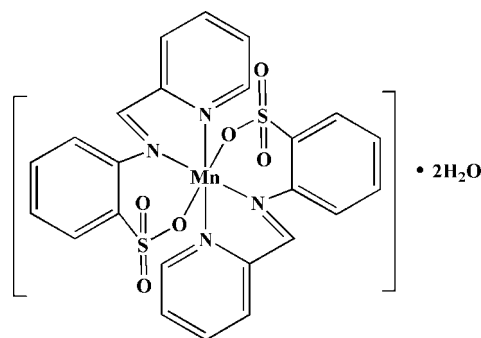
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(C-C) = 0.004$ Å; R factor = 0.032; wR factor = 0.092; data-to-parameter ratio = 13.1.

The title complex, $[Mn(C_{12}H_9N_2O_3S)_2] \cdot 2H_2O$, is isotypic with the previously reported Zn^{II} and Cd^{II} species. The complex was prepared by the reaction of the potassium salt of 2-(2-pyridylmethyleneamino)benzenesulfonic acid with $MnCl_2 \cdot 6H_2O$ in methanol. The complex displays twofold symmetry, with the ligands coordinated in a tridentate meridional-like arrangement through pyridyl N, imine N, and sulfonate O atoms. The metal center has a strongly distorted octahedral coordination geometry. The uncoordinated water molecules and the complexes participate in a hydrogen-bonding network, forming a two-dimensional structure parallel to the ab plane.

Related literature

For the synthesis of the ligand, see: Casella & Gullotti (1986). For the structures of the Zn^{II} and Cd^{II} analogues, see: Cai *et al.* (2008); Ou-Yang *et al.* (2008). For related Schiff bases complexes, see: Correia *et al.* (2003); Li *et al.* (2007, 2008); Ou-Yang *et al.* (2009); Zhang *et al.* (2005).



Experimental

Crystal data

$[Mn(C_{12}H_9N_2O_3S)_2] \cdot 2H_2O$

$M_r = 613.52$

Orthorhombic, $Pbcn$

$a = 20.041$ (10) Å

$b = 7.918$ (4) Å

$c = 16.409$ (8) Å

$V = 2604$ (2) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 0.72$ mm⁻¹

$T = 296$ K

$0.49 \times 0.34 \times 0.21$ mm

Data collection

SMART APEX CCD

diffractometer

Absorption correction: none

13313 measured reflections

2320 independent reflections

1950 reflections with $I > 2\sigma(I)$

$R_{int} = 0.028$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.092$

$S = 1.01$

2320 reflections

177 parameters

H-atom parameters constrained

$\Delta\rho_{max} = 0.47$ e Å⁻³

$\Delta\rho_{min} = -0.41$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O4-H1W \cdots O3^i$	0.85	2.14	2.993 (3)	179
$O4-H2W \cdots O2$	0.84	2.11	2.866 (3)	151

Symmetry code: (i) $-x + \frac{3}{2}, y - \frac{1}{2}, z$.

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINTE* (Bruker, 2004); data reduction: *SAINTE*; 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: BH2236).

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supplementary materials

Acta Cryst. (2009). E65, m1055-m1056 [doi:10.1107/S1600536809030670]

Bis[2-(2-pyridylmethyleneamino)benzenesulfonato- κ^3N,N',O]manganese(II) dihydrate

C.-Q. Zhao, M. Ou-Yang, X.-R. Huang and Y.-M. Jiang

Comment

In the past decades, Schiff-base complexes containing sulfonate have received much attention owing to the diverse coordination modes and bridging ability (Zhang *et al.*, 2005; Li *et al.*, 2007; Ou-Yang *et al.*, 2009). Herein, we prepared a mononuclear Mn(II) complex, which is isostructural with [Zn(Paba)₂].2 H₂O and [Cd(Paba)₂].2 H₂O, whose structures were described in details (Cai *et al.*, 2008; Ou-Yang *et al.*, 2008). The six-coordinated Mn^{II} ion lies on a twofold axis, and two deprotonated PabaH anions coordinate to Mn in a *meridional* arrangement as *N,N',O*-tridentate donor ligands. The coordination mode of the complex is similar to that observed in other complexes with *N,N',O*-tridentate donor ligands (Li *et al.*, 2007, 2008; Correia *et al.*, 2003).

The O—H donor groups from the lattice water molecules and the SO acceptor groups of the Paba ligands participate in the hydrogen bonding through which the complex completes a two-dimensional network parallel to the *ab* plane (Fig. 2).

Experimental

The potassium salt of 2-(2-pyridylmethyleneamino)benzenesulfonic acid (PabaK) was synthesized according to the literature methods (Casella & Gullotti, 1986). The ligand PabaK (1 mmol, 0.30 g) was dissolved in methanol (10 ml). To this solution, MnCl₂·6 H₂O (0.5 mmol, 0.12 g) was added, and the resulting mixture was stirred at 333 K for 4 h. Then the mixture was filtrated and the filtrate was left to stand at room temperature. Yellow crystals suitable for X-ray diffraction were obtained after a week in a yield of 55%. Elemental analysis: found C 46.87, H 4.04, N 9.05, S 10.42%; calc. C 46.94, H 3.59, N 9.13, S 10.43%.

Refinement

C-bonded H atoms were positioned geometrically with C—H distances of 0.93 Å, and treated as riding atoms, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{carrier C})$. Water H atoms were placed in fixed positions and assigned U_{iso} values of 1.5 U_{eq} of the water O atom.

Figures

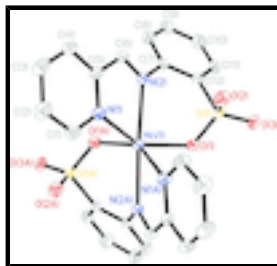


Fig. 1. The molecular structure of the title complex, showing the atom-numbering scheme. The water molecule and all H atoms have been omitted for clarity. Symmetry code for 'A' labelled atoms: 2 - x, y, 1/2 - z.

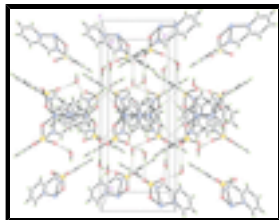


Fig. 2. Packing of the title complex, showing the two-dimensional network in the *ab* plane, linked *via* hydrogen bonds (dashed lines).

Bis[2-(2-pyridylmethyleneamino)benzenesulfonato- κ^3N,N',O]manganese(II) dihydrate

Crystal data

$[\text{Mn}(\text{C}_{12}\text{H}_9\text{N}_2\text{O}_3\text{S})_2] \cdot 2\text{H}_2\text{O}$

$M_r = 613.52$

Orthorhombic, *Pbcn*

Hall symbol: -P 2n 2ab

$a = 20.041(10) \text{ \AA}$

$b = 7.918(4) \text{ \AA}$

$c = 16.409(8) \text{ \AA}$

$V = 2604(2) \text{ \AA}^3$

$Z = 4$

$F_{000} = 1260$

$D_x = 1.565 \text{ Mg m}^{-3}$

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

Cell parameters from 4677 reflections

$\theta = 2.5\text{--}28.5^\circ$

$\mu = 0.72 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Block, yellow

$0.49 \times 0.34 \times 0.21 \text{ mm}$

Data collection

SMART APEX CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 296 \text{ K}$

φ and ω scans

Absorption correction: none

13313 measured reflections

2320 independent reflections

1950 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\text{max}} = 25.1^\circ$

$\theta_{\text{min}} = 2.5^\circ$

$h = -21 \rightarrow 23$

$k = -9 \rightarrow 9$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.092$

$S = 1.01$

2320 reflections

177 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 1.5899P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.47 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.41 \text{ e \AA}^{-3}$

Extinction correction: none

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Mn1	1.0000	0.18060 (6)	0.2500	0.02835 (15)
S1	0.87380 (3)	0.32706 (7)	0.33859 (3)	0.03333 (17)
O1	0.94725 (8)	0.3323 (2)	0.33645 (9)	0.0381 (4)
O2	0.84921 (9)	0.1575 (2)	0.32866 (10)	0.0508 (5)
O3	0.84826 (8)	0.4155 (2)	0.40879 (9)	0.0466 (4)
N1	0.99221 (9)	-0.0275 (2)	0.15711 (11)	0.0352 (4)
N2	0.90694 (8)	0.2347 (2)	0.16781 (10)	0.0303 (4)
C1	1.03597 (13)	-0.1521 (3)	0.14852 (14)	0.0444 (6)
H1	1.0708	-0.1601	0.1858	0.053*
C2	1.03222 (15)	-0.2704 (3)	0.08686 (15)	0.0495 (6)
H2	1.0641	-0.3553	0.0826	0.059*
C3	0.98074 (14)	-0.2607 (3)	0.03207 (16)	0.0503 (6)
H3	0.9767	-0.3399	-0.0095	0.060*
C4	0.93496 (13)	-0.1310 (3)	0.03971 (14)	0.0440 (6)
H4	0.8998	-0.1210	0.0031	0.053*
C5	0.94222 (11)	-0.0165 (3)	0.10256 (12)	0.0329 (5)
C6	0.89698 (11)	0.1272 (3)	0.11156 (13)	0.0360 (5)
H6	0.8610	0.1400	0.0762	0.043*
C7	0.86610 (10)	0.3806 (3)	0.17345 (12)	0.0321 (5)
C8	0.84533 (12)	0.4702 (3)	0.10524 (14)	0.0440 (6)
H8	0.8560	0.4311	0.0534	0.053*
C9	0.80902 (13)	0.6167 (4)	0.11399 (15)	0.0536 (7)
H9	0.7950	0.6754	0.0680	0.064*
C10	0.79326 (14)	0.6770 (4)	0.19056 (17)	0.0542 (7)
H10	0.7695	0.7772	0.1960	0.065*
C11	0.81279 (12)	0.5887 (3)	0.25907 (14)	0.0432 (6)
H11	0.8014	0.6283	0.3106	0.052*
C12	0.84920 (10)	0.4420 (3)	0.25118 (11)	0.0319 (5)
O4	0.79877 (12)	-0.0889 (4)	0.44087 (15)	0.1140 (12)
H1W	0.7570	-0.0869	0.4320	0.171*
H2W	0.8166	-0.0498	0.3988	0.171*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0279 (3)	0.0336 (3)	0.0235 (2)	0.000	-0.00482 (17)	0.000
S1	0.0326 (3)	0.0437 (3)	0.0236 (3)	0.0002 (2)	0.0001 (2)	0.0008 (2)
O1	0.0316 (8)	0.0540 (10)	0.0287 (8)	0.0050 (7)	-0.0044 (6)	-0.0070 (7)
O2	0.0606 (12)	0.0486 (11)	0.0431 (10)	-0.0120 (9)	-0.0038 (8)	0.0083 (8)
O3	0.0434 (9)	0.0699 (12)	0.0267 (8)	0.0085 (8)	0.0051 (7)	-0.0043 (8)
N1	0.0409 (10)	0.0350 (10)	0.0297 (9)	0.0024 (8)	-0.0067 (8)	-0.0015 (8)
N2	0.0280 (9)	0.0384 (10)	0.0245 (8)	0.0003 (8)	-0.0007 (7)	-0.0004 (7)
C1	0.0503 (15)	0.0428 (14)	0.0401 (13)	0.0087 (12)	-0.0108 (11)	-0.0008 (10)
C2	0.0684 (17)	0.0356 (13)	0.0445 (14)	0.0116 (12)	-0.0024 (13)	-0.0030 (11)

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C3	0.0721 (18)	0.0382 (13)	0.0406 (13)	0.0020 (13)	-0.0054 (13)	-0.0095 (11)
C4	0.0510 (15)	0.0466 (14)	0.0345 (12)	-0.0044 (11)	-0.0110 (11)	-0.0076 (10)
C5	0.0350 (11)	0.0369 (12)	0.0269 (10)	-0.0033 (9)	-0.0028 (9)	-0.0003 (9)
C6	0.0318 (11)	0.0488 (13)	0.0273 (11)	-0.0008 (10)	-0.0067 (9)	-0.0017 (10)
C7	0.0243 (11)	0.0435 (13)	0.0284 (10)	0.0018 (9)	-0.0006 (8)	0.0017 (9)
C8	0.0398 (13)	0.0621 (16)	0.0302 (11)	0.0098 (12)	0.0019 (10)	0.0066 (11)
C9	0.0495 (15)	0.0690 (18)	0.0421 (14)	0.0200 (14)	0.0001 (12)	0.0159 (13)
C10	0.0497 (16)	0.0556 (17)	0.0574 (16)	0.0213 (13)	0.0015 (13)	0.0089 (13)
C11	0.0380 (13)	0.0517 (15)	0.0398 (13)	0.0107 (11)	0.0045 (10)	-0.0024 (11)
C12	0.0257 (11)	0.0415 (12)	0.0284 (11)	-0.0003 (9)	0.0002 (8)	0.0014 (9)
O4	0.0640 (15)	0.182 (3)	0.0956 (19)	-0.0353 (17)	-0.0233 (13)	0.075 (2)

Geometric parameters (\AA , $^\circ$)

Mn1—O1	2.1382 (16)	C3—C4	1.383 (4)
Mn1—O1 ⁱ	2.1382 (16)	C3—H3	0.9300
Mn1—N1	2.250 (2)	C4—C5	1.381 (3)
Mn1—N1 ⁱ	2.250 (2)	C4—H4	0.9300
Mn1—N2 ⁱ	2.3412 (19)	C5—C6	1.463 (3)
Mn1—N2	2.3412 (19)	C6—H6	0.9300
S1—O2	1.4395 (19)	C7—C8	1.389 (3)
S1—O3	1.4420 (17)	C7—C12	1.406 (3)
S1—O1	1.4729 (18)	C8—C9	1.376 (4)
S1—C12	1.769 (2)	C8—H8	0.9300
N1—C1	1.327 (3)	C9—C10	1.381 (4)
N1—C5	1.346 (3)	C9—H9	0.9300
N2—C6	1.271 (3)	C10—C11	1.380 (3)
N2—C7	1.418 (3)	C10—H10	0.9300
C1—C2	1.381 (3)	C11—C12	1.378 (3)
C1—H1	0.9300	C11—H11	0.9300
C2—C3	1.371 (4)	O4—H1W	0.8495
C2—H2	0.9300	O4—H2W	0.8364
O1—Mn1—O1 ⁱ	111.67 (10)	C1—C2—H2	120.6
O1—Mn1—N1	145.73 (6)	C2—C3—C4	118.8 (2)
O1 ⁱ —Mn1—N1	89.79 (7)	C2—C3—H3	120.6
O1—Mn1—N1 ⁱ	89.79 (7)	C4—C3—H3	120.6
O1 ⁱ —Mn1—N1 ⁱ	145.73 (6)	C5—C4—C3	119.0 (2)
N1—Mn1—N1 ⁱ	85.83 (10)	C5—C4—H4	120.5
O1—Mn1—N2 ⁱ	84.77 (6)	C3—C4—H4	120.5
O1 ⁱ —Mn1—N2 ⁱ	83.42 (7)	N1—C5—C4	122.2 (2)
N1—Mn1—N2 ⁱ	125.42 (7)	N1—C5—C6	116.40 (18)
N1 ⁱ —Mn1—N2 ⁱ	71.86 (7)	C4—C5—C6	121.4 (2)
O1—Mn1—N2	83.42 (6)	N2—C6—C5	119.80 (19)
O1 ⁱ —Mn1—N2	84.77 (6)	N2—C6—H6	120.1
N1—Mn1—N2	71.86 (7)	C5—C6—H6	120.1
N1 ⁱ —Mn1—N2	125.42 (7)	C8—C7—C12	118.8 (2)

N2 ⁱ —Mn1—N2	158.90 (9)	C8—C7—N2	122.43 (19)
O2—S1—O3	114.95 (11)	C12—C7—N2	118.65 (18)
O2—S1—O1	111.44 (11)	C9—C8—C7	120.3 (2)
O3—S1—O1	111.11 (9)	C9—C8—H8	119.8
O2—S1—C12	107.02 (10)	C7—C8—H8	119.8
O3—S1—C12	107.40 (11)	C8—C9—C10	120.5 (2)
O1—S1—C12	104.18 (9)	C8—C9—H9	119.8
S1—O1—Mn1	119.62 (9)	C10—C9—H9	119.8
C1—N1—C5	117.98 (19)	C11—C10—C9	120.1 (2)
C1—N1—Mn1	124.77 (15)	C11—C10—H10	120.0
C5—N1—Mn1	117.04 (14)	C9—C10—H10	120.0
C6—N2—C7	120.13 (18)	C12—C11—C10	120.0 (2)
C6—N2—Mn1	114.88 (15)	C12—C11—H11	120.0
C7—N2—Mn1	124.83 (13)	C10—C11—H11	120.0
N1—C1—C2	123.1 (2)	C11—C12—C7	120.3 (2)
N1—C1—H1	118.5	C11—C12—S1	120.34 (16)
C2—C1—H1	118.5	C7—C12—S1	119.38 (17)
C3—C2—C1	118.9 (2)	H1W—O4—H2W	105.8
C3—C2—H2	120.6		
O2—S1—O1—Mn1	41.05 (13)	C2—C3—C4—C5	-0.5 (4)
O3—S1—O1—Mn1	170.64 (10)	C1—N1—C5—C4	0.9 (3)
C12—S1—O1—Mn1	-74.01 (13)	Mn1—N1—C5—C4	175.90 (17)
O1 ⁱ —Mn1—O1—S1	115.14 (11)	C1—N1—C5—C6	-177.0 (2)
N1—Mn1—O1—S1	-10.02 (18)	Mn1—N1—C5—C6	-2.0 (2)
N1 ⁱ —Mn1—O1—S1	-92.28 (11)	C3—C4—C5—N1	-0.5 (4)
N2 ⁱ —Mn1—O1—S1	-164.08 (11)	C3—C4—C5—C6	177.4 (2)
N2—Mn1—O1—S1	33.44 (10)	C7—N2—C6—C5	175.63 (19)
O1—Mn1—N1—C1	-137.89 (18)	Mn1—N2—C6—C5	0.1 (3)
O1 ⁱ —Mn1—N1—C1	91.6 (2)	N1—C5—C6—N2	1.3 (3)
N1 ⁱ —Mn1—N1—C1	-54.42 (17)	C4—C5—C6—N2	-176.7 (2)
N2 ⁱ —Mn1—N1—C1	9.8 (2)	C6—N2—C7—C8	-40.0 (3)
N2—Mn1—N1—C1	176.1 (2)	Mn1—N2—C7—C8	135.02 (19)
O1—Mn1—N1—C5	47.5 (2)	C6—N2—C7—C12	143.7 (2)
O1 ⁱ —Mn1—N1—C5	-83.05 (16)	Mn1—N2—C7—C12	-41.3 (3)
N1 ⁱ —Mn1—N1—C5	130.97 (18)	C12—C7—C8—C9	0.2 (4)
N2 ⁱ —Mn1—N1—C5	-164.81 (14)	N2—C7—C8—C9	-176.1 (2)
N2—Mn1—N1—C5	1.52 (15)	C7—C8—C9—C10	0.5 (4)
O1—Mn1—N2—C6	-156.78 (16)	C8—C9—C10—C11	-1.3 (4)
O1 ⁱ —Mn1—N2—C6	90.65 (16)	C9—C10—C11—C12	1.3 (4)
N1—Mn1—N2—C6	-0.84 (15)	C10—C11—C12—C7	-0.6 (4)
N1 ⁱ —Mn1—N2—C6	-71.75 (17)	C10—C11—C12—S1	-179.4 (2)
N2 ⁱ —Mn1—N2—C6	146.83 (16)	C8—C7—C12—C11	-0.1 (3)
O1—Mn1—N2—C7	27.95 (16)	N2—C7—C12—C11	176.3 (2)
O1 ⁱ —Mn1—N2—C7	-84.62 (16)	C8—C7—C12—S1	178.73 (17)
N1—Mn1—N2—C7	-176.11 (17)	N2—C7—C12—S1	-4.8 (3)
N1 ⁱ —Mn1—N2—C7	112.98 (16)	O2—S1—C12—C11	124.5 (2)

supplementary materials

N2 ⁱ —Mn1—N2—C7	-28.44 (15)	O3—S1—C12—C11	0.6 (2)
C5—N1—C1—C2	-0.4 (4)	O1—S1—C12—C11	-117.32 (19)
Mn1—N1—C1—C2	-174.95 (19)	O2—S1—C12—C7	-54.3 (2)
N1—C1—C2—C3	-0.6 (4)	O3—S1—C12—C7	-178.25 (17)
C1—C2—C3—C4	1.0 (4)	O1—S1—C12—C7	63.82 (19)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O4—H1W \cdots O3 ⁱⁱ	0.85	2.14	2.993 (3)	179
O4—H2W \cdots O2	0.84	2.11	2.866 (3)	151

Symmetry codes: (ii) $-x+3/2, y-1/2, z$.

Fig. 1

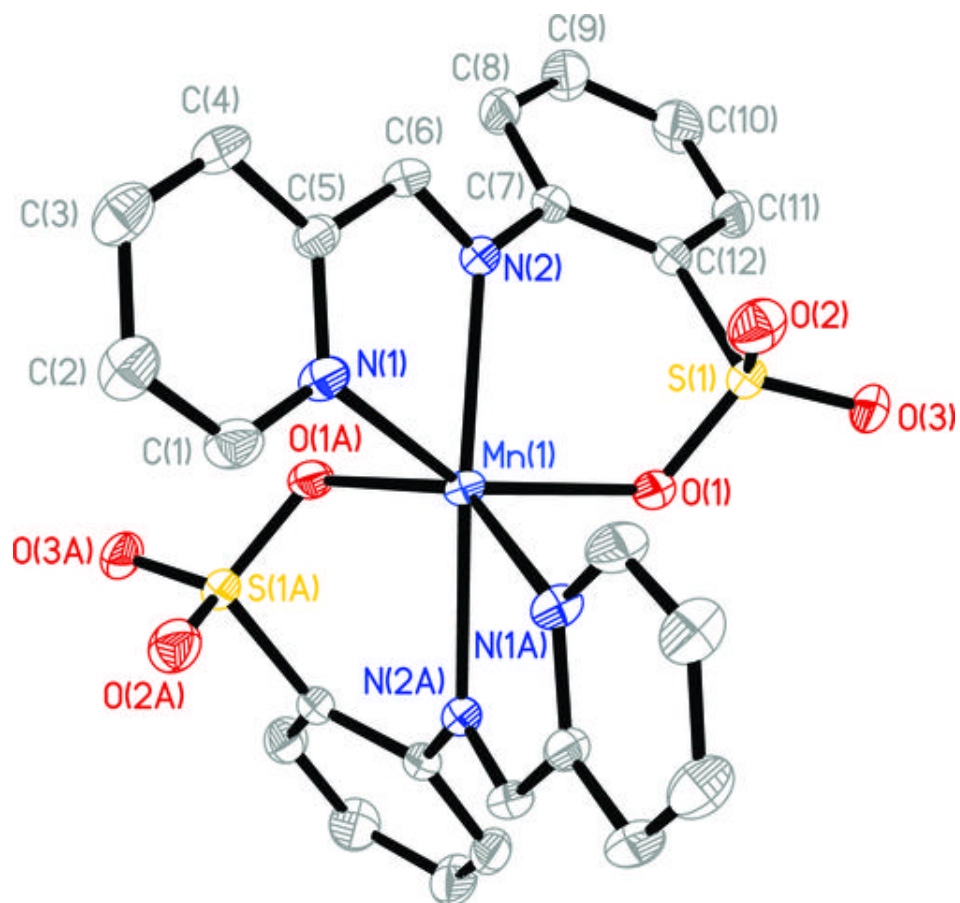


Fig. 2

