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(1-{(*E*)-2-[(2*E*,3*Z*)-4-Oxidopent-3-en-2-ylideneamino]ethyliminomethyl}-naphthalen-2-olato)manganese(II) methanol solvate

Bin Xu,[a] Jing-Wen Ran[a] and Ya-Hong Li[a,b]*

[a]Institute of Salt Lakes, Chinese Academy of Sciences, Xining City 810008, People's Republic of China, and [b]Key Laboratory of Organic Synthesis of Jiangsu Province, School of Chemistry and Chemical Engineering, Suzhou University, Suzhou 215123, People's Republic of China
Correspondence e-mail: liyahong@isl.ac.cn

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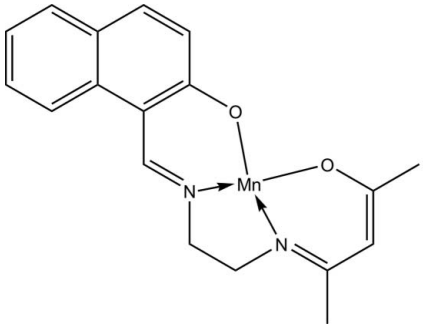
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.011 Å; R factor = 0.050; wR factor = 0.152; data-to-parameter ratio = 13.8.

In the title compound, $[Mn(C_{18}H_{18}N_2O_2)]\cdot CH_3OH$, the Mn atom is coordinated by two N atoms and two O atoms from the asymmetrical Schiff base ligand 1-[2-(4-oxidopent-3-en-2-ylideneamino)ethyliminomethyl]naphthalen-2-olate in an approximately square-planar configuration. There is an O—H···O hydrogen-bond interaction between the complex and the methanol solvent molecule.

Related literature

Complexes with a similar ligand were reported by Yan *et al.* (2006); in those complexes the ligand was synthesized from the reaction of ethylenediamine, acetylacetone and salicylaldehyde.



· CH_3OH

Experimental

Crystal data

$[Mn(C_{18}H_{18}N_2O_2)]\cdot CH_4O$
M_r = 381.33
Orthorhombic, $P2_12_12_1$
a = 7.3800 (16) Å
b = 10.8935 (14) Å
c = 22.01 (2) Å
V = 1769.6 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.77 mm^{-1}
T = 298 (2) K
0.48 × 0.32 × 0.29 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.709, T_{max} = 0.808
9257 measured reflections
3122 independent reflections
1863 reflections with $I > 2\sigma(I)$
R_{int} = 0.081

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.152
S = 0.91
3122 reflections
227 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Absolute structure: Flack (1983), 1306 Friedel pairs
Flack parameter: −0.14 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H4···O2	0.82	2.20	3.008 (9)	171

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); 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: XU2325).

References

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Yan, X. Y., Wang, B., Ma, H. Z. & Gao, F. Q. (2006). *Chem. Res. Appl.* **18**, 261–264.

supplementary materials

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(1-{(*E*)-2-[(2*E*,3*Z*)-4-Oxidopent-3-en-2-ylideneamino]ethyliminomethyl}naphthalen-2-olato)manganese(II) methanol solvate

B. Xu, J.-W. Ran and Y.-H. Li

Comment

Complexes synthesized from manganese and Schiff base ligand have been of great interest for many years. They are very important in the development of coordination chemistry. As an extension of the research on the structural characterization of Mn compounds, we report here the crystal structure of a new mononuclear manganese(II) complex.

The title compound is an electronically neutral mononuclear manganese(II) complex. The Mn^{II} ion in the compound is four coordinated by two N atoms and two O atoms from the asymmetrical tetradentate Schiff base ligand H_2hemn (H_2hemn= 1-((*E*)-(2-((*E*)-((*Z*)-4-hydroxypent-3-en-2-ylidene)amino)\ ethylimino)methyl)naphthalen-2-ol) in an approximately square-planar geometry. The Mn—N2 bond distance (1.812 (6) Å) is shorter than Mn—N1(1.836 (5) Å) and the other two Mn—O bonds distance (Mn—O1=1.835 (5) Å, Mn—O2=1.829 (4) Å). There is O—H···O hydrogen bond interaction between the complex and the methanol solvent molecule (Table 1).

Experimental

1. Synthesis of the ligand H_2hemn

To a 250 ml 3-neck round-bottom flask containing a solution of ethylenediamine (0.1 mol, 6.01 g) in ethanol (60 ml) at 50 °C, was added dropwise a solution of acetylacetone (0.1 mol, 10.01 g) in ethanol (60 ml). After the mixture was stirred at 50 °C for 4 h. A suspension of 2-hydroxy-1-naphthaldehyde (0.1 mol, 17.22 g) in ethanol (50 ml) was added into the flask. The resulted mixture was continued being stirred for another 4 h and then cooled down and the crude product was precipitated. The crude product was collected by filtration, washed with ethanol and vacuum dried overnight. The brown product H_2hemn was used without further purification.

2. Synthesis of the complex

To a solution of $MnCl_2{\cdot}4H_2O$ (1 mmol, 197 mg) in methanol (40 ml) was added ligand H_2hemn (1 mmol, 298 mg). After the resulted brown mixture was stirred at room temperature for 48 h, a brown turbid solution was obtained. The solution was filtered and slow evaporation of the solvent from the filtrate afforded dark brown crystals after 30 d.

Refinement

Methyl H atoms and hydroxyl H atom were placed in calculated positions with C—H = 0.96 Å and O—H = 0.82 Å, and torsion angles were refined, U_{iso}(H) = 1.5U_{eq}(C,*O*). Other H atoms were placed in calculated positions with C—H = 0.93 (aromatic) or 0.97 Å (methylene) and refined in riding mode, with U_{iso}(H) = 1.2U_{eq}(C).

Figures

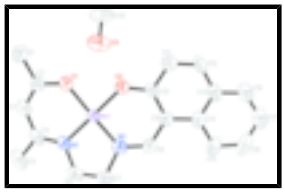

Fig. 1. The atom-numbering scheme of the title complex. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted.

Fig. 2. The packed diagram for the title compound, viewed down the *b* axis with hydrogen bonds drawn as dashed lines. H atoms have been omitted.

(1-{(*E*)-2-[(2E,3*Z*)-4-Oxidopent-3-en-2- ylideneamino]ethyliminomethyl}naphthalen-2-olato)manganese(II) methanol solvate

Crystal data

$[Mn(C_{18}H_{18}N_2O_2)]\cdot CH_4O$	F_{000} = 796
M_r = 381.33	D_x = 1.431 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	θ = 1.9–25.0º
a = 7.3800 (16) Å	μ = 0.77 mm^{-1}
b = 10.8935 (14) Å	*T* = 298 (2) K
c = 22.01 (2) Å	Block, dark brown
V = 1769.6 (18) Å^3	0.48 × 0.32 × 0.29 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3122 independent reflections
Radiation source: fine-focus sealed tube	1863 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.081
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −8→8
T_{min} = 0.709, T_{max} = 0.808	*k* = −12→12
9257 measured reflections	*l* = −12→26

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$w = 1/[\sigma^2(F_o^2) + (0.0806P)^2]$

	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.152$	$(\Delta/\sigma)_{max} = 0.001$
$S = 0.91$	$\Delta\rho_{max} = 0.29$ e Å^{-3}
3122 reflections	$\Delta\rho_{min} = -0.29$ e Å^{-3}
227 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1306 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: −0.14 (4)

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 $F^2 > 2\sigma(F^2)$ 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	0.56596 (12)	0.66361 (7)	0.47664 (4)	0.0444 (3)
N1	0.5557 (7)	0.8306 (5)	0.4659 (3)	0.0621 (13)
N2	0.5466 (8)	0.6932 (5)	0.5574 (3)	0.0653 (15)
O1	0.6034 (6)	0.6254 (4)	0.3964 (2)	0.0703 (14)
C1	0.6364 (10)	0.6470 (7)	0.2897 (3)	0.091 (2)
H1A	0.7642	0.6325	0.2856	0.136*
H1B	0.5972	0.7025	0.2584	0.136*
H1C	0.5723	0.5707	0.2858	0.136*
O2	0.5492 (7)	0.4972 (4)	0.48610 (18)	0.0685 (12)
O3	0.3061 (10)	0.4431 (6)	0.3797 (3)	0.140 (3)
H4	0.3692	0.4501	0.4102	0.210*
C2	0.5976 (10)	0.7030 (7)	0.3516 (3)	0.070 (2)
C3	0.5641 (10)	0.8231 (6)	0.3573 (3)	0.0754 (19)
H2	0.5551	0.8686	0.3216	0.090*
C4	0.5406 (9)	0.8882 (6)	0.4136 (4)	0.070 (2)
C5	0.4973 (10)	1.0246 (6)	0.4112 (4)	0.104 (3)
H4A	0.5880	1.0694	0.4332	0.155*
H4B	0.3808	1.0389	0.4292	0.155*
H4C	0.4958	1.0514	0.3697	0.155*
C6	0.5291 (10)	0.9018 (6)	0.5220 (4)	0.077 (2)
H5A	0.4023	0.9231	0.5269	0.093*
H5B	0.5997	0.9768	0.5207	0.093*
C7	0.5913 (10)	0.8210 (6)	0.5734 (3)	0.076 (2)
H6A	0.7209	0.8297	0.5794	0.091*

H6B	0.5305	0.8441	0.6108	0.091*
C8	0.5105 (8)	0.6148 (6)	0.6014 (3)	0.0626 (19)
H7	0.5063	0.6450	0.6409	0.075*
C9	0.4781 (8)	0.4893 (6)	0.5928 (3)	0.0618 (18)
C10	0.4983 (9)	0.4373 (6)	0.5344 (3)	0.067 (2)
C11	0.4626 (12)	0.3081 (6)	0.5271 (3)	0.090 (2)
H10	0.4787	0.2719	0.4893	0.108*
C12	0.4054 (13)	0.2374 (6)	0.5749 (4)	0.092 (3)
H11	0.3757	0.1555	0.5683	0.111*
C13	0.3906 (11)	0.2869 (7)	0.6343 (4)	0.078 (2)
C14	0.4268 (10)	0.4126 (7)	0.6430 (3)	0.0694 (18)
C15	0.4008 (10)	0.4593 (8)	0.7031 (3)	0.087 (2)
H14	0.4212	0.5417	0.7119	0.105*
C16	0.3433 (11)	0.3769 (11)	0.7489 (4)	0.110 (3)
H15	0.3262	0.4074	0.7879	0.132*
C17	0.3113 (14)	0.2542 (10)	0.7389 (5)	0.110 (3)
H16	0.2712	0.2031	0.7700	0.132*
C18	0.3400 (11)	0.2108 (8)	0.6825 (5)	0.097 (3)
H17	0.3256	0.1273	0.6753	0.116*
C19	0.3463 (13)	0.3403 (9)	0.3519 (5)	0.148 (4)
H18A	0.3735	0.3571	0.3100	0.222*
H18B	0.2451	0.2852	0.3542	0.222*
H18C	0.4498	0.3034	0.3710	0.222*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0396 (4)	0.0422 (4)	0.0515 (5)	−0.0011 (4)	0.0017 (5)	−0.0022 (5)
N1	0.047 (3)	0.056 (3)	0.083 (4)	−0.006 (3)	0.003 (4)	−0.002 (3)
N2	0.053 (3)	0.068 (4)	0.075 (4)	−0.004 (3)	0.000 (3)	−0.008 (3)
O1	0.081 (4)	0.059 (3)	0.071 (3)	−0.004 (2)	0.004 (3)	0.006 (2)
C1	0.094 (6)	0.097 (6)	0.081 (5)	0.010 (5)	0.016 (4)	0.011 (5)
O2	0.088 (3)	0.054 (2)	0.064 (3)	0.005 (3)	0.011 (3)	−0.002 (2)
O3	0.144 (6)	0.110 (5)	0.166 (6)	0.018 (4)	−0.034 (5)	−0.036 (5)
C2	0.062 (5)	0.074 (5)	0.075 (5)	−0.009 (4)	0.009 (4)	0.004 (4)
C3	0.073 (5)	0.057 (4)	0.097 (5)	−0.003 (5)	0.000 (5)	0.012 (4)
C4	0.041 (4)	0.058 (4)	0.111 (6)	−0.008 (3)	0.003 (5)	0.014 (4)
C5	0.075 (6)	0.064 (5)	0.172 (9)	0.000 (4)	0.011 (6)	0.028 (6)
C6	0.062 (5)	0.056 (4)	0.113 (6)	−0.008 (3)	0.015 (5)	−0.017 (5)
C7	0.066 (5)	0.075 (5)	0.086 (5)	−0.015 (5)	0.006 (4)	−0.024 (4)
C8	0.051 (4)	0.077 (5)	0.060 (4)	0.000 (3)	−0.002 (3)	0.002 (4)
C9	0.056 (5)	0.063 (4)	0.067 (4)	0.006 (3)	0.004 (3)	0.004 (4)
C10	0.077 (5)	0.047 (4)	0.077 (5)	0.008 (3)	−0.002 (4)	0.004 (4)
C11	0.112 (7)	0.076 (5)	0.083 (5)	0.008 (4)	0.014 (6)	−0.004 (4)
C12	0.111 (7)	0.055 (4)	0.111 (7)	0.001 (5)	0.023 (6)	0.023 (5)
C13	0.070 (6)	0.074 (5)	0.090 (6)	0.012 (4)	0.016 (4)	0.019 (5)
C14	0.054 (4)	0.082 (5)	0.073 (5)	0.011 (5)	0.004 (4)	0.012 (4)
C15	0.072 (6)	0.118 (7)	0.071 (5)	0.010 (5)	−0.004 (4)	0.014 (5)

C16	0.077 (6)	0.184 (11)	0.069 (6)	0.024 (7)	0.004 (5)	0.031 (7)
C17	0.105 (8)	0.106 (8)	0.120 (9)	0.017 (6)	0.008 (7)	0.039 (7)
C18	0.086 (6)	0.085 (6)	0.118 (7)	0.013 (5)	0.013 (6)	0.029 (6)
C19	0.152 (10)	0.120 (8)	0.172 (9)	0.031 (8)	0.003 (8)	−0.078 (8)

Geometric parameters (Å, °)

Mn1—N2	1.812 (6)	C6—H5B	0.9700
Mn1—O2	1.829 (4)	C7—H6A	0.9700
Mn1—O1	1.835 (5)	C7—H6B	0.9700
Mn1—N1	1.836 (5)	C8—C9	1.401 (8)
N1—C4	1.316 (8)	C8—H7	0.9300
N1—C6	1.470 (8)	C9—C10	1.412 (9)
N2—C8	1.319 (7)	C9—C14	1.436 (8)
N2—C7	1.475 (8)	C10—C11	1.441 (9)
O1—C2	1.300 (7)	C11—C12	1.370 (9)
C1—C2	1.521 (10)	C11—H10	0.9300
C1—H1A	0.9600	C12—C13	1.418 (10)
C1—H1B	0.9600	C12—H11	0.9300
C1—H1C	0.9600	C13—C18	1.396 (10)
O2—C10	1.303 (7)	C13—C14	1.408 (10)
O3—C19	1.311 (9)	C14—C15	1.432 (9)
O3—H4	0.8200	C15—C16	1.414 (10)
C2—C3	1.338 (8)	C15—H14	0.9300
C3—C4	1.439 (9)	C16—C17	1.375 (11)
C3—H2	0.9300	C16—H15	0.9300
C4—C5	1.520 (8)	C17—C18	1.346 (11)
C5—H4A	0.9600	C17—H16	0.9300
C5—H4B	0.9600	C18—H17	0.9300
C5—H4C	0.9600	C19—H18A	0.9600
C6—C7	1.506 (9)	C19—H18B	0.9600
C6—H5A	0.9700	C19—H18C	0.9600
N2—Mn1—O2	93.4 (2)	C6—C7—H6A	110.2
N2—Mn1—O1	174.9 (2)	N2—C7—H6B	110.2
O2—Mn1—O1	83.98 (18)	C6—C7—H6B	110.2
N2—Mn1—N1	86.9 (2)	H6A—C7—H6B	108.5
O2—Mn1—N1	173.7 (2)	N2—C8—C9	124.5 (6)
O1—Mn1—N1	96.2 (2)	N2—C8—H7	117.8
C4—N1—C6	118.1 (6)	C9—C8—H7	117.8
C4—N1—Mn1	126.0 (5)	C8—C9—C10	119.8 (6)
C6—N1—Mn1	114.8 (4)	C8—C9—C14	120.6 (6)
C8—N2—C7	118.7 (6)	C10—C9—C14	119.6 (6)
C8—N2—Mn1	128.5 (4)	O2—C10—C9	124.9 (6)
C7—N2—Mn1	112.7 (4)	O2—C10—C11	116.8 (6)
C2—O1—Mn1	125.3 (4)	C9—C10—C11	118.3 (7)
C2—C1—H1A	109.5	C12—C11—C10	121.3 (7)
C2—C1—H1B	109.5	C12—C11—H10	119.3
H1A—C1—H1B	109.5	C10—C11—H10	119.3
C2—C1—H1C	109.5	C11—C12—C13	121.2 (7)

H1A—C1—H1C	109.5	C11—C12—H11	119.4
H1B—C1—H1C	109.5	C13—C12—H11	119.4
C10—O2—Mn1	127.5 (4)	C18—C13—C14	121.6 (8)
C19—O3—H4	109.5	C18—C13—C12	119.7 (8)
O1—C2—C3	124.8 (6)	C14—C13—C12	118.7 (7)
O1—C2—C1	114.5 (6)	C13—C14—C15	116.5 (7)
C3—C2—C1	120.7 (7)	C13—C14—C9	120.7 (7)
C2—C3—C4	125.8 (7)	C15—C14—C9	122.7 (7)
C2—C3—H2	117.1	C16—C15—C14	118.2 (8)
C4—C3—H2	117.1	C16—C15—H14	120.9
N1—C4—C3	120.6 (6)	C14—C15—H14	120.9
N1—C4—C5	120.9 (8)	C17—C16—C15	123.7 (9)
C3—C4—C5	118.4 (7)	C17—C16—H15	118.2
C4—C5—H4A	109.5	C15—C16—H15	118.2
C4—C5—H4B	109.5	C18—C17—C16	117.5 (10)
H4A—C5—H4B	109.5	C18—C17—H16	121.2
C4—C5—H4C	109.5	C16—C17—H16	121.2
H4A—C5—H4C	109.5	C17—C18—C13	122.3 (9)
H4B—C5—H4C	109.5	C17—C18—H17	118.8
N1—C6—C7	106.4 (5)	C13—C18—H17	118.8
N1—C6—H5A	110.4	O3—C19—H18A	109.5
C7—C6—H5A	110.4	O3—C19—H18B	109.5
N1—C6—H5B	110.4	H18A—C19—H18B	109.5
C7—C6—H5B	110.4	O3—C19—H18C	109.5
H5A—C6—H5B	108.6	H18A—C19—H18C	109.5
N2—C7—C6	107.6 (6)	H18B—C19—H18C	109.5
N2—C7—H6A	110.2		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H4···O2	0.82	2.20	3.008 (9)	171

Fig. 1

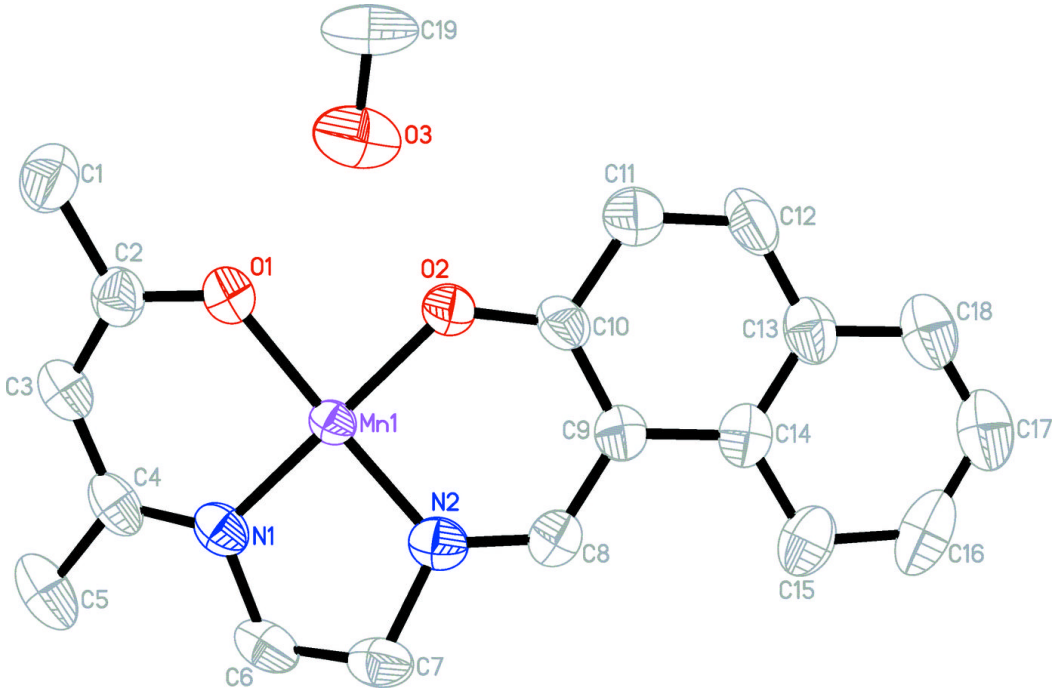
C19
O3
C1
C2
O1
O2
C11
C12
C10
C13
C18
C3
Mn1
C9
C14
C17
C4
N2
N1
C8
C16
C5
C15
C6
C7

Fig. 2