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The crystal structure of ((*E*)-2,4-dibromo-6-(((5-(nitro)-2-oxidophenyl)imino)methyl)phenolato- $\kappa^3 N, O, O'$)tris(pyridine- κN)manganese(II), $C_{28}H_{21}Br_2MnN_5O_4$

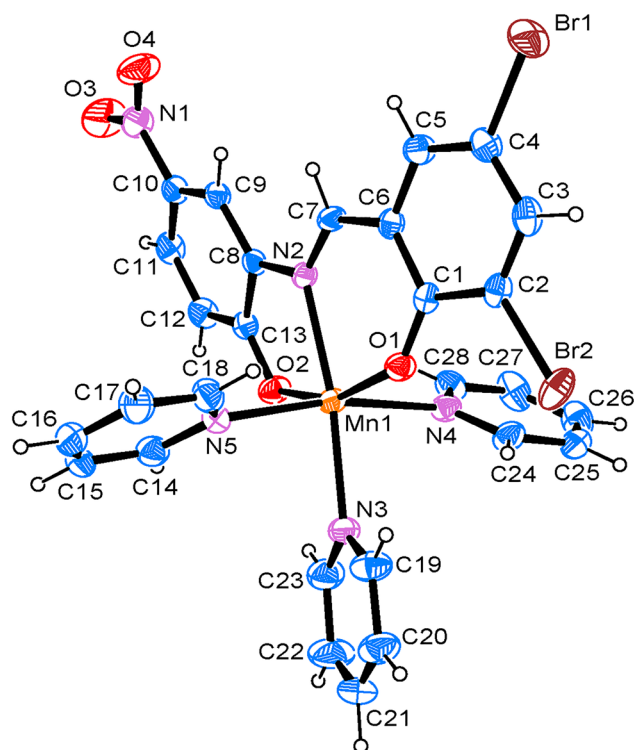


Table 1: Data collection and handling.

Crystal:	Brown block
Size:	0.22 × 0.21 × 0.18 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	3.33 mm ⁻¹
Diffractometer, scan mode:	BRUKER APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.1°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	15,027, 5015, 0.026
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 3905
$N(\text{param})_{\text{refined}}$:	361
Programs:	BRUKER [1], SHELX [2, 3], WINGX/ORTEP [4]

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

All chemicals were purchased from commercial sources and used as received. A mixture of 2-amino-4-nitrophenol (10 mmol), 3,5-dibromo-2-hydroxybenzaldehyde (10 mmol) and CH₃OH (50 mL) was added in a round-bottomed flask (100 mL), and refluxed with agitating for 4.0 h. The solution was cooled, and then the insoluble matter is removed by filtration. The ligand 2,4-dibromo-6-(((2-hydroxy-5-nitrophenyl)imino)methyl)phenol was obtained. 1 mmol 2,4-dibromo-6-(((2-hydroxy-5-nitrophenyl)imino)methyl)-phenol and 1 mmol manganese(II) acetate were added in CH₃OH (50 mL) and refluxed with agitating for 4 h. Then, most of the methanol was evaporated, followed by adding pyridine dropwise until it just dissolved. The reaction solution continued to reflux for 2.5 h and filtered afterwards. Finally, the title crystal was precipitated by controlling solvent volatilization.

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Abstract

$C_{28}H_{21}Br_2MnN_5O_4$, triclinic, $P\bar{1}$ (no. 2), $a = 8.9193(14)$ Å, $b = 9.2271(15)$ Å, $c = 17.933(3)$ Å, $\alpha = 103.574(2)^\circ$, $\beta = 91.518(2)^\circ$, $\gamma = 98.762(2)^\circ$, $V = 1415.0(4)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0347$, $wR_{\text{ref}}(F^2) = 0.0864$, $T = 296(2)$ K.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
Br1	0.18458 (5)	0.46796 (6)	0.35855 (3)	0.08344 (16)
Br2	0.77624 (5)	0.75514 (5)	0.47505 (2)	0.07265 (14)
Mn1	0.90399 (5)	0.87393 (5)	0.22980 (3)	0.04434 (13)
O1	0.8114 (2)	0.7609 (2)	0.30946 (12)	0.0489 (5)
O2	0.9105 (2)	0.9820 (3)	0.13740 (13)	0.0552 (6)
O4	0.3667 (4)	0.6314 (4)	−0.08933 (17)	0.1019 (11)
O3	0.4608 (4)	0.7898 (5)	−0.15187 (17)	0.1136 (12)
N1	0.4602 (4)	0.7393 (5)	−0.09467 (17)	0.0734 (9)
N2	0.6833 (3)	0.7760 (3)	0.16347 (13)	0.0394 (5)
N5	1.0041 (3)	0.6661 (3)	0.16103 (15)	0.0492 (6)
N3	1.1405 (3)	0.9492 (3)	0.28493 (16)	0.0531 (7)
N4	0.8498 (3)	1.0919 (3)	0.31044 (16)	0.0498 (6)
C4	0.3819 (4)	0.5607 (4)	0.3421 (2)	0.0552 (8)
C3	0.4903 (4)	0.6107 (4)	0.40302 (19)	0.0558 (9)
H3	0.468446	0.596070	0.451395	0.067*
C2	0.6302 (4)	0.6823 (4)	0.39079 (17)	0.0472 (7)
C1	0.6748 (3)	0.7048 (3)	0.31819 (16)	0.0410 (7)
C13	0.8015 (3)	0.9307 (3)	0.08481 (18)	0.0445 (7)
C12	0.7961 (4)	0.9844 (4)	0.0171 (2)	0.0556 (8)
H12	0.871043	1.062302	0.011526	0.067*
C11	0.6845 (4)	0.9254 (4)	−0.04019 (19)	0.0561 (9)
H11	0.682861	0.964016	−0.083633	0.067*
C10	0.5737 (4)	0.8079 (4)	−0.03316 (17)	0.0515 (8)
C9	0.5705 (3)	0.7536 (4)	0.03294 (17)	0.0462 (7)
H9	0.494949	0.674957	0.037038	0.055*
C8	0.6791 (3)	0.8162 (3)	0.09219 (16)	0.0394 (7)
C7	0.5693 (3)	0.6984 (4)	0.18504 (17)	0.0461 (7)
H7	0.485962	0.662393	0.149704	0.055*
C6	0.5558 (3)	0.6605 (3)	0.25846 (16)	0.0426 (7)
C5	0.4134 (4)	0.5870 (4)	0.27136 (19)	0.0535 (8)
H5	0.338477	0.555193	0.231284	0.064*
C14	1.0981 (4)	0.6768 (5)	0.1050 (2)	0.0621 (9)
H14	1.125151	0.770358	0.093742	0.075*
C15	1.1564 (4)	0.5551 (6)	0.0633 (2)	0.0723 (12)
H15	1.221043	0.566425	0.024457	0.087*
C16	1.1183 (4)	0.4182 (5)	0.0796 (2)	0.0729 (11)
H16	1.157246	0.334790	0.052449	0.087*
C17	1.0225 (5)	0.4046 (5)	0.1361 (2)	0.0717 (11)
H17	0.994554	0.312004	0.148196	0.086*
C18	0.9682 (4)	0.5303 (4)	0.1747 (2)	0.0601 (9)
H18	0.901962	0.519804	0.213013	0.072*
C19	1.1891 (4)	0.8904 (4)	0.3400 (2)	0.0700 (11)
H19	1.122857	0.816830	0.355638	0.084*
C20	1.3336 (5)	0.9338 (5)	0.3749 (3)	0.0807 (13)
H20	1.364187	0.891356	0.413793	0.097*
C21	1.4301 (5)	1.0397 (6)	0.3514 (3)	0.0939 (15)
H21	1.529065	1.069086	0.373140	0.113*
C22	1.3820 (5)	1.1026 (7)	0.2960 (3)	0.1124 (19)
H22	1.446324	1.176900	0.279906	0.135*
C23	1.2353 (4)	1.0538 (5)	0.2641 (2)	0.0790 (12)
H23	1.202022	1.096949	0.226001	0.095*
C24	0.8710 (4)	1.1121 (4)	0.3860 (2)	0.0599 (9)
H24	0.905788	1.036435	0.404584	0.072*
C25	0.8445 (4)	1.2384 (4)	0.4377 (2)	0.0658 (10)
H25	0.861636	1.248558	0.490237	0.079*

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C26	0.7921 (5)	1.3497 (4)	0.4109 (3)	0.0763 (12)
H26	0.772808	1.436926	0.444761	0.092*
C27	0.7686 (5)	1.3300 (4)	0.3332 (3)	0.0753 (12)
H27	0.732729	1.403805	0.313461	0.090*
C28	0.7985 (4)	1.2010 (4)	0.2851 (2)	0.0574 (9)
H28	0.782454	1.188760	0.232403	0.069*

Experimental details

All H-atoms were placed geometrically and refined using a riding model with common isotropic displacement factors $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5 U_{\text{eq}}(\text{parent C-atom})$.

Comment

Schiff bases are versatile compounds, which have been synthesized from the condensation of carbonyl compounds with amino compounds and exhibit a broad range of applications [5]. The metal complexes with Schiff bases have been widely investigated for their versatile structures and potential applications in many fields [6, 7]. At present, the Schiff base derivatives of salicylaldehyde are mostly studied. Such Schiff bases have multiple coordination sites and can form stable metal complexes with most metal ions [8]. Furthermore, manganese is one of the most important transition metal ions in biological systems and plays an important role in activation of enzymes and sugar metabolism [9, 10].

The ORTEP diagram is presented in the Figure. Bond lengths and angles are all in the expected ranges. The ligand of the title molecule exhibits an *E* configuration. The bond length of C7=N2 is 1.275(4) Å, which is similar to those reported in the literature [11]. Mn1 forms a distorted octahedral coordination geometry and it is surrounded by two oxygen atoms (O1 and O2) and one nitrogen atom (N2) from the ligand, three nitrogen atoms (N3, N4 and N5) from three different pyridine ligands. The axial positions were occupied by two nitrogen atoms (N4 and N5) from two pyridine ligands, respectively. The angle of N4–Mn1–N5 is 169.09(9)° which obviously deviates from linear angle 180°. The four atoms (O1, O2, N2 and N3) were placed in equatorial sites, the sum of the angles [O2–Mn1–N2 = 76.44(8)°, O1–Mn1–N2 = 83.74(8)°, O1–Mn1–N3 = 98.41(10)°, O2–Mn1–N3 = 102.38(10)°] is 360.97°, which shows that the four

atoms are basically planar, but the bond lengths [Mn1–O1 = 2.076(2) Å, Mn1–O2 = 2.123(2) Å, Mn1–N2 = 2.222(2) Å, Mn1–N3 = 2.243(3) Å] are unequal. It indicated that the central Mn atom is six-coordinate in a distorted pentagonal pyramid geometry, and the coordination mode is similar to that of previously reported complexes [12, 13].

Intermolecular hydrogen bonding exists in the crystal structure of the title compound. The carbon atom C15 provides one intermolecular hydrogen bond to O4' of another molecule ($C15 \cdots O4 = 3.504(5)$ Å; $' = x + 1, y, z$). The carbon atom C22 provides one intermolecular hydrogen bond to O3'' of another molecule ($C22 \cdots O3'' = 3.257(6)$ Å; $'' = -x + 2, -y + 2, -z$). The carbon atom C28 provides one intermolecular hydrogen bond to O3''' of another molecule ($C28 \cdots O3''' = 3.306(5)$ Å; $''' = -x + 1, -y + 2, -z$).

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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