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Chlorido-(2,2'-(ethane-bis(5-methoxyphenolato))- $\kappa^4 N, N', O, O'$) manganese(III) monohydrate, $C_{19}H_{18}Cl_2CuN_2O_2$

Table 1: Data collection and handling.

Crystal:	Purple block
Size:	0.311 × 0.28 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.83 mm ⁻¹
Diffraction, scan mode:	CCD area detector, φ and ω -scans
$\theta_{\max}$, completeness:	25°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7696, 3476, 0.018
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3081
$N(\text{param})_{\text{refined}}$:	269
Programs:	CrysAlis ^{PRO} [1], OLEX2 [2], SHELX [3, 4]

Source of material

The structure was solved with the OLEX2 program [2] as an interface together with the SHELXT and SHELXL programs [3, 4]. All H atoms were placed in geometrically idealized positions and refined using a riding model, with C–H = 0.98 (methylene), 0.95 Å (benzene), and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ for H atoms on methylene, phenolic hydroxyl and benzene.

Experimental details

Ethylenediamine (0.3005 g, 5 mmol) in ethanol (10 mL) was added dropwise to a DMF (20 mL) solution of 2'-hydroxy-4'-methoxy-acetophenone (1.90 g, 10 mmol) with constant stirring at room temperature and the yellow suspension was then refluxed for 1 h under stirring. When no more liquid was distilled under 78–80 °C the refluxing under 110 °C was continued for an additional hour. The mixture was cooled gradually to room temperature. A amount of pale-yellow product (H_2L) was appeared and isolated by filtration, washed with ethanol rapidly. $MnCl_2 \cdot 4H_2O$ (0.099 g, 0.5 mmol) was dissolved in 4 mL distilled water and added dropwise to a ethanol suspension (40 mL) of H_2L (0.178 g 0.5 mmol). The above mixture was mechanically stirred for 4 h at an ambient temperature and turned to dark brown solution. The obtained filtrate was then sealed with

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Abstract

$C_{19}H_{18}Cl_2CuN_2O_2$, monoclinic, $C2/c$ (No. 15), $a = 21.010(3)$ Å, $b = 11.3886(15)$ Å, $c = 7.9578(10)$ Å, $\beta = 100.725(4)^\circ$, $V = 1870.8(4)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0331$, $wR_{\text{ref}}(F^2) = 0.1077$, $T = 170.0$ K.

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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.

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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}
Mn1	0.61043 (4)	0.46999 (4)	0.15145 (3)	0.03365 (14)
Cl1	0.82675 (7)	0.34055 (7)	0.29444 (6)	0.04820 (19)
O1	0.63686 (18)	0.3611 (2)	0.04537 (17)	0.0419 (4)
O2	0.49358 (18)	0.35532 (18)	0.23948 (16)	0.0387 (4)
O3	0.9190 (2)	0.0606 (2)	−0.14917 (17)	0.0460 (4)
O4	0.2012 (2)	0.1500 (2)	0.53852 (17)	0.0544 (5)
N1	0.6914 (2)	0.6230 (2)	0.03383 (18)	0.0344 (4)
N2	0.5307 (2)	0.6293 (2)	0.22250 (18)	0.0349 (4)
C1	0.8200 (2)	0.4588 (3)	−0.0683 (2)	0.0324 (5)
C2	0.7478 (2)	0.3454 (3)	−0.0213 (2)	0.0314 (5)
C3	0.7875 (2)	0.2140 (3)	−0.0498 (2)	0.0339 (5)
H3	0.7407	0.1397	−0.0176	0.041*
C4	0.8948 (2)	0.1917 (3)	−0.1251 (2)	0.0348 (5)
C5	0.9682 (3)	0.3004 (3)	−0.1718 (2)	0.0409 (6)
H5	1.0422	0.2850	−0.2209	0.049*
C6	0.9299 (3)	0.4304 (3)	−0.1442 (2)	0.0382 (5)
H6	0.9783	0.5031	−0.1768	0.046*
C7	0.7761 (2)	0.6040 (3)	−0.0497 (2)	0.0328 (5)
C8	0.6365 (3)	0.7728 (3)	0.0462 (3)	0.0463 (6)
H8A	0.5510	0.8335	−0.0103	0.056*
H8B	0.7080	0.8273	0.0258	0.056*
C9	0.6017 (3)	0.7505 (3)	0.1760 (3)	0.0498 (7)
H9A	0.6893	0.7228	0.2284	0.060*
H9B	0.5393	0.8455	0.1786	0.060*
C10	0.4414 (2)	0.6296 (3)	0.3057 (2)	0.0335 (5)
C11	0.3788 (2)	0.5043 (3)	0.3604 (2)	0.0334 (5)
C12	0.2822 (3)	0.5080 (3)	0.4511 (2)	0.0420 (6)
H12	0.2581	0.5927	0.4743	0.050*
C13	0.2228 (3)	0.3932 (3)	0.5058 (2)	0.0473 (6)
H13	0.1580	0.4013	0.5638	0.057*
C14	0.2594 (3)	0.2635 (3)	0.4748 (2)	0.0395 (6)
C15	0.3526 (3)	0.2536 (3)	0.3868 (2)	0.0356 (5)
H15	0.3772	0.1664	0.3669	0.043*
C16	0.4110 (2)	0.3734 (3)	0.3268 (2)	0.0303 (5)
C17	0.3990 (3)	0.7628 (3)	0.3492 (3)	0.0463 (6)
H17A	0.4770	0.8061	0.3439	0.070*
H17B	0.3768	0.7276	0.4330	0.070*
H17C	0.3163	0.8392	0.2981	0.070*
C18	0.2130 (4)	0.0283 (4)	0.4947 (3)	0.0684 (9)
H18A	0.1684	0.0696	0.4118	0.103*
H18B	0.1662	−0.0419	0.5465	0.103*
H18C	0.3125	−0.0243	0.4964	0.103*
C19	1.0224 (3)	0.0368 (4)	−0.2334 (3)	0.0558 (7)
H19A	0.9916	0.1173	−0.3126	0.084*
H19B	1.0315	−0.0599	−0.2408	0.084*
H19C	1.1135	0.0372	−0.2033	0.084*
C21	0.8274 (3)	0.7355 (3)	−0.1294 (2)	0.0460 (6)
H21A	0.7466	0.8246	−0.1654	0.069*
H21B	0.8772	0.7093	−0.1935	0.069*
H21C	0.8911	0.7565	−0.0800	0.069*
O5	0.5833 (3)	1.0512 (2)	0.2290 (2)	0.0701 (6)
H5A	0.5555	1.1483	0.2050	0.105*
H5B	0.6463	1.0152	0.2904	0.105*

parafilm. The brown black crystals of the title compound were obtained after 1 week by slow evaporation.

Comment

Paeonol is a class of methyl ketone compound with relatively small steric hindrance, and the carbonyl group tends to condensed to generate Schiff base derivatives. That is, the reaction of paeonol (−CO−CH₃) with amine can produces the compounds containing the Schiff base characteristic imine group (−RC=N−) [5, 6]. Although these compounds belong to ketone Schiff base, their reactivities are very high and can act as a multi-dentate ligand to create complexes with various structures and properties [7, 8]. However, related studies on complexes constructed from the paeonol Schiff base ligand is still largely unexplored. In order to explore the regulating effect of different types of Schiff base ligands on the structure of transition metal complexes, we report herein a new manganese complex based on the paeonol Schiff base ligand.

Single-crystal X-ray diffraction analyses results reveal that the asymmetric unit is formed by a mononuclear Mn(III) complex plus a water molecule. The coordination environment of Mn(III) is defined by N₂O₂ at the equatorial plane. The bond lengths distributions for Mn–O and Mn–N are in the range of 1.8667(15) and 1.8732(17) Å, 1.9813(19) and 1.9871(19) Å, respectively, suggesting the bond length of Mn–N is longer than that of Mn–O. The apical positions of the title compound are occupied by one chlorido ligand Cl(1) and one oxygen atom from the other halves of paeonol Schiff base ligand, which bond length of Mn(1)–O(1)*i* and Mn(1)–Cl(1) is 3.002(3) and 2.4338(8) Å, respectively (*i*: 1 − *x*, 1 − *y*, −*z*). We can clearly see the Jahn–Teller effect of the high-spin *d*⁴ metal center. The hexa-coordinate Mn(III) center possesses an elongated axis, furnishing a common distorted octahedral geometric structure. Besides, the bond angles around Mn(III) centers were also found to be similar with previously reported structures [9, 10].

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