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The crystal structure of bis(2-oxidopyridin-1-ium-3-carboxylato- $\kappa^2 O, O'$)-(phenantroline- $\kappa^2 N, N'$) manganese(II) - methanol (1/3), $C_{27}H_{28}N_4O_9Mn$

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Abstract

$C_{27}H_{28}N_4O_9Mn$, monoclinic, $P2_1/n$ (no. 14), $a = 10.0269(7)$ Å, $b = 14.1702(10)$ Å, $c = 20.0750(14)$ Å, $\beta = 100.298(1)^\circ$, $V = 2806.4(3)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0403$, $wR_{ref}(F^2) = 0.0985$, $T = 296.15$ 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 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.15 × 0.15 × 0.15 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.530 mm ⁻¹
Diffraction, scan mode:	Bruker Smart APEX-II, φ and ω
θ_{max} , completeness:	27.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	19,168, 6216, 0.041
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 4175
$N(param)_{refined}$:	376
Programs:	Bruker [1], Olex2 [2]

Source of material

2-Hydroxypyridine-3-carboxylic acid (2.0 mmol) was dissolved in 20 mL of methanol. $Mn(CH_3COO)_2 \cdot 4H_2O$ (1.0 mmol) dissolved in 20 mL of methanol was added dropwise to the above 2-Hydroxypyridine-3-carboxylic acid solution and stirred for 4 h at 55 °C. Then 1,10-phenanthroline (1.0 mmol) was dissolved in 10 mL of methanol was added dropwise to the above solution and stirred for 4 h at 55 °C cooled and filtered. The filtrate was left for slow evaporation at room temperature. The yellow block crystals were formed 16 days later. Yield: 41.6%. **Anal. Calcd.** for $C_{27}H_{28}N_4O_9Mn$: C, 53.38; H, 4.65; N, 9.22; found: C, 53.60; H, 4.63; N, 9.21.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

Complexes with heterocyclic ligands are worth for many reasons, chief among them are their biological activities [3–5]. Pyridine ring drugs have the ability to enhance the binding of drugs to biomacromolecules and resist cancer [6]. As a part of our current research on the biological activity of heterocyclic transition metal complexes, we report herein a new manganese complex.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	x	y	z	U_{iso}^*/U_{eq}
Mn1	0.48376 (3)	0.73084 (2)	0.16374 (2)	0.03437 (11)
O1	0.3942 (2)	0.75381 (13)	0.24930 (9)	0.0634 (5)
O2	0.26414 (18)	0.80029 (13)	0.31965 (8)	0.0582 (5)
O3	0.36297 (16)	0.85036 (11)	0.12491 (8)	0.0451 (4)
O4	0.66095 (16)	0.81310 (10)	0.18994 (8)	0.0473 (4)
O5	0.83807 (15)	0.86849 (9)	0.26015 (9)	0.0496 (4)
O6	0.61517 (14)	0.61866 (9)	0.20869 (8)	0.0375 (4)
O7	0.4710 (2)	0.74745 (18)	0.41799 (11)	0.0867 (7)
H7	0.413018	0.761581	0.385299	0.130*
O8	0.4176 (3)	0.67300 (16)	0.53196 (11)	0.0910 (7)
H8	0.428318	0.697868	0.496405	0.136*
O9	0.2685 (3)	0.5143 (2)	0.51818 (14)	0.1296 (11)
H9	0.312762	0.561086	0.532397	0.194*
N1	0.20155 (18)	0.96047 (12)	0.11013 (10)	0.0439 (5)
H1	0.220988	0.970154	0.070666	0.053*
N2	0.80018 (17)	0.53735 (11)	0.25538 (9)	0.0349 (4)
H2	0.751267	0.487145	0.249876	0.042*
N3	0.32079 (18)	0.62228 (12)	0.12078 (10)	0.0409 (5)
N4	0.50555 (18)	0.70051 (12)	0.05504 (9)	0.0377 (4)
C1	0.3022 (2)	0.80537 (16)	0.26436 (12)	0.0394 (5)
C2	0.2352 (2)	0.87733 (14)	0.21450 (11)	0.0346 (5)
C3	0.2732 (2)	0.89168 (14)	0.14943 (11)	0.0338 (5)
C4	0.1031 (3)	1.01403 (17)	0.12861 (14)	0.0543 (7)
H4	0.059626	1.059501	0.099039	0.065*
C5	0.0675 (3)	1.00217 (18)	0.18914 (15)	0.0584 (7)
H5	0.000343	1.039150	0.202496	0.070*
C6	0.1342 (2)	0.93276 (16)	0.23155 (13)	0.0459 (6)
H6	0.109203	0.923493	0.273493	0.055*
C7	0.7696 (2)	0.80239 (14)	0.23157 (11)	0.0327 (5)
C8	0.8230 (2)	0.70368 (13)	0.24784 (10)	0.0296 (4)
C9	0.7382 (2)	0.62157 (13)	0.23500 (10)	0.0285 (4)
C10	0.9318 (2)	0.52763 (15)	0.28331 (13)	0.0445 (6)
H10	0.966347	0.468012	0.295673	0.053*
C11	1.0141 (2)	0.60370 (15)	0.29357 (13)	0.0501 (6)
H11	1.105764	0.597434	0.311630	0.060*
C12	0.9564 (2)	0.69249 (15)	0.27603 (12)	0.0413 (5)
H12	1.011182	0.745756	0.283884	0.050*
C13	0.2287 (2)	0.58494 (18)	0.15313 (14)	0.0558 (7)
H13	0.224509	0.606717	0.196418	0.067*
C14	0.1379 (3)	0.5147 (2)	0.12534 (18)	0.0686 (9)
H14	0.075034	0.490223	0.149604	0.082*
C15	0.1434 (3)	0.48300 (19)	0.06220 (19)	0.0705 (9)
H15	0.085165	0.435225	0.043263	0.085*
C16	0.2366 (3)	0.52174 (16)	0.02507 (14)	0.0533 (7)
C17	0.3238 (2)	0.59197 (15)	0.05696 (12)	0.0406 (5)
C18	0.4214 (2)	0.63399 (14)	0.02179 (11)	0.0374 (5)
C19	0.4266 (3)	0.60586 (17)	−0.04461 (13)	0.0503 (6)
C20	0.5214 (3)	0.6509 (2)	−0.07692 (14)	0.0664 (8)
H20	0.527881	0.634613	−0.121097	0.080*
C21	0.6036 (3)	0.7181 (2)	−0.04423 (15)	0.0651 (8)
H21	0.666086	0.748859	−0.065680	0.078*
C22	0.5932 (3)	0.74039 (17)	0.02208 (13)	0.0519 (6)
H22	0.651238	0.786011	0.044428	0.062*
C23	0.2474 (3)	0.49314 (19)	−0.04183 (16)	0.0680 (9)
H23	0.190969	0.445737	−0.062895	0.082*

Table 2: (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C24	0.3373 (3)	0.5332 (2)	−0.07487 (15)	0.0676 (8)
H24	0.341783	0.513322	−0.118564	0.081*
C25	0.5989 (4)	0.7519 (3)	0.3999 (2)	0.1020 (12)
H25A	0.613324	0.814063	0.383456	0.153*
H25B	0.604237	0.706480	0.364995	0.153*
H25C	0.667059	0.738365	0.438711	0.153*
C26	0.4659 (4)	0.7328 (2)	0.58586 (17)	0.0874 (10)
H26A	0.554225	0.755548	0.581601	0.131*
H26B	0.471690	0.698946	0.627681	0.131*
H26C	0.405212	0.785294	0.585468	0.131*
C27	0.1883 (5)	0.4896 (3)	0.5621 (2)	0.1377 (17)
H27A	0.234153	0.502156	0.607448	0.207*
H27B	0.167776	0.423467	0.557373	0.207*
H27C	0.105777	0.525319	0.552964	0.207*

Single crystal X-ray diffraction analysis demonstrates that the asymmetric unit of the title structure contains one manganese(II) cation, two 2-oxidopyridin-1-ium-3-carboxylate ligands, one 1,10-phenanthroline ligand and three methanol molecules (see the Figure). The bond distances of Mn–N are 2.2727(18)–2.2971(19) Å, bond lengths of Mn–O are 2.1010(17) and 2.1568(14) Å, respectively, which are similar to the reference [7–9]. In the title complex, the bond angles of O(3)–Mn(1)–O(6), O(1)–Mn(1)–N(4) and O(4)–Mn(1)–N(3), are 175.20(6), 160.52(7), 166.83(7)° respectively. The Mn (II) is six coordinated by four oxygen and two nitrogen atoms from the two 2-oxidopyridin-1-ium-3-carboxylate ligands and 1, 10-phenanthroline to furnish a distorted octahedron geometry [10]. In addition, there are three solvent methanol molecules not coordinating with metal ions in the complex.

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