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Crystal structure of bis(phenanthroline-κ^2N,N')propanedionato-κ^2O,O'-manganese(II) ethanol solvate, $C_{27}H_{18}N_4O_4Mn \cdot CH_3CH_2OH$

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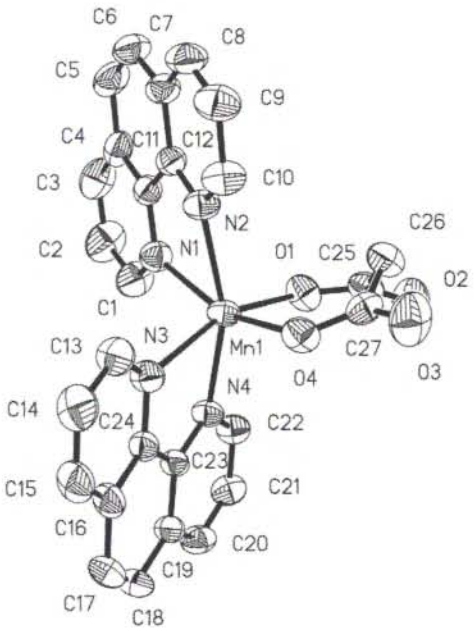

Abstract

$C_{29}H_{24}MnN_4O_5$, triclinic, $P\bar{1}$ (No. 2), a = 9.5971(6) Å, b = 11.4837(7) Å, c = 12.8316(9) Å, α = 87.408(5)°, β = 83.313(5)°, γ = 66.373(4)°, V = 1286.8 Å^3, Z = 2, $R_{gt}(F)$ = 0.042, $wR_{ref}(F^2)$ = 0.118, T = 295 K.

Source of material

The reaction of manganese(II) acetate tetrahydrate (2 mmol, 0.490 g) and 2 eqivalent of 1,10-phenanthroline (4 mmol, 0.792 g) in 10 ml absolute ethanol afforded a pale yellow solution. Propanedionic acid (0.208 g, 2 mmol) in 10 ml absolute ethanol was added. A yellow precipitate formed immediately upon addition. After the addition was complete, the mixture was stirred for an additional hour, then filtered. Suitable crystals were obtained by slow vapour diffusion of diethyl ether in the filtrate. The structure proposed is consistent with elemental analysis: calculated C 61.81, H 4.29, N 9.95; found C 62.03, H 4.35, N 9.74.

Discussion

In the complex, the coordination geometry of the manganese atom is a distorted octahedron, with the propanedionic acid ligand coordinated through two oxygen atoms, and the four nitrogen atoms from two phenanthroline ligands occupying the remnant sites. The stability of the six-membered ring formed by Mn(II) and propanedionic acid in the structure is the reason that propanedionic acid coordinates only to one metal atom and does not act as a bridge between different metal atoms. And there is one ethanol molecule disorderly arranged in the complex. The dihedral angle between the two phenanthroline planes is 56.7°. The Mn ion is six-coordinated of a bidentate propanedionic acid and two phenantroline ligands which are coordinated to the manganese atom in an bidentate manner in the opposite site.

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.25 × 0.35 × 0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	5.61 cm^{-1}
Diffractometer, scan mode:	Bruker P4, $\omega/2\theta$
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5374, 4497
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3745
$N(param)_{refined}$:	352
Program:	SHELXTL [1]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(1A)	2*i*		0.5066	0.8861	0.8881	0.070
H(2A)	2*i*		0.2792	1.0621	0.8850	0.083
H(3A)	2*i*		0.1676	1.1174	0.7313	0.076
H(5A)	2*i*		0.1746	1.0717	0.5387	0.069
H(6A)	2*i*		0.3000	0.9547	0.3948	0.068
H(8A)	2*i*		0.5328	0.7700	0.3090	0.065
H(9A)	2*i*		0.7635	0.6047	0.3272	0.068
H(10A)	2*i*		0.8531	0.5619	0.4896	0.059
H(13A)	2*i*		0.9831	0.7216	0.5718	0.059
H(14A)	2*i*		1.2235	0.7248	0.5683	0.069
H(15A)	2*i*		1.3346	0.6989	0.7214	0.067
H(17A)	2*i*		1.3135	0.6734	0.9197	0.065
H(18A)	2*i*		1.1734	0.6611	1.0697	0.062
H(20A)	2*i*		0.9303	0.6452	1.1627	0.055
H(21A)	2*i*		0.6993	0.6347	1.1488	0.055
H(22A)	2*i*		0.6192	0.6402	0.9851	0.050
H(26A)	2*i*		0.6538	0.4388	0.6152	0.060
H(26B)	2*i*		0.6998	0.3031	0.6645	0.060
C(2'A)	2*i*	0.36(2)	–0.105(2)	1.025(1)	0.887(2)	0.099(7)
C(2'B)	2*i*	0.64	–0.115(1)	1.0402(9)	0.816(1)	0.110(4)
O(1'A)	2*i*	0.47(2)	–0.379(1)	1.1323(8)	0.899(1)	0.113(4)
O(1'B)	2*i*	0.53	–0.3722(7)	1.1280(6)	0.8330(8)	0.093(3)

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mn(1)	2*i*	0.74087(4)	0.64502(4)	0.73655(3)	0.0262(2)	0.0440(2)	0.0346(2)	–0.0122(2)	–0.0060(2)	–0.0008(2)
C(1)	2*i*	0.4631(4)	0.9084(3)	0.8253(3)	0.063(2)	0.049(2)	0.051(2)	–0.010(2)	–0.005(2)	–0.007(1)
C(2)	2*i*	0.3254(4)	1.0144(3)	0.8243(3)	0.072(2)	0.050(2)	0.063(2)	–0.002(2)	0.004(2)	–0.011(2)
C(3)	2*i*	0.2596(4)	1.0470(3)	0.7332(3)	0.052(2)	0.040(2)	0.079(2)	–0.000(1)	0.000(2)	0.001(2)
C(4)	2*i*	0.3299(3)	0.9753(3)	0.6423(2)	0.038(2)	0.036(1)	0.064(2)	–0.014(1)	–0.005(1)	0.008(1)
C(5)	2*i*	0.2680(4)	1.0037(3)	0.5437(3)	0.043(2)	0.049(2)	0.074(2)	–0.010(1)	–0.020(2)	0.019(2)
C(6)	2*i*	0.3423(4)	0.9337(3)	0.4582(3)	0.054(2)	0.062(2)	0.060(2)	–0.025(2)	–0.027(2)	0.019(2)
C(7)	2*i*	0.4859(3)	0.8269(3)	0.4624(2)	0.040(2)	0.055(2)	0.045(2)	–0.024(1)	–0.014(1)	0.012(1)
C(8)	2*i*	0.5703(4)	0.7522(3)	0.3741(2)	0.060(2)	0.075(2)	0.036(2)	–0.033(2)	–0.014(1)	0.010(1)
C(9)	2*i*	0.7065(4)	0.6543(3)	0.3848(2)	0.056(2)	0.078(2)	0.037(2)	–0.028(2)	–0.002(1)	–0.004(2)
C(10)	2*i*	0.7595(3)	0.6292(3)	0.4832(2)	0.039(2)	0.065(2)	0.038(2)	–0.014(1)	–0.001(1)	–0.001(1)
C(11)	2*i*	0.4699(3)	0.8709(2)	0.6502(2)	0.033(1)	0.037(1)	0.046(2)	–0.016(1)	–0.007(1)	0.006(1)
C(12)	2*i*	0.5485(3)	0.7955(2)	0.5582(2)	0.032(1)	0.044(1)	0.040(1)	–0.020(1)	–0.008(1)	0.008(1)
C(13)	2*i*	1.0267(3)	0.7120(3)	0.6343(2)	0.050(2)	0.060(2)	0.041(2)	–0.028(2)	0.000(1)	0.005(1)
C(14)	2*i*	1.1721(4)	0.7139(3)	0.6315(3)	0.053(2)	0.070(2)	0.057(2)	–0.036(2)	0.009(2)	0.009(2)
C(15)	2*i*	1.2372(3)	0.6997(3)	0.7226(3)	0.039(2)	0.059(2)	0.078(2)	–0.030(2)	–0.003(2)	0.006(2)
C(16)	2*i*	1.1581(3)	0.6863(3)	0.8177(2)	0.034(1)	0.044(2)	0.059(2)	–0.021(1)	–0.006(1)	0.002(1)
C(17)	2*i*	1.2165(3)	0.6747(3)	0.9170(3)	0.041(2)	0.062(2)	0.073(2)	–0.030(2)	–0.022(2)	0.007(2)
C(18)	2*i*	1.1338(3)	0.6657(3)	1.0061(3)	0.049(2)	0.054(2)	0.059(2)	–0.023(1)	–0.027(2)	0.003(1)
C(19)	2*i*	0.9857(3)	0.6631(2)	1.0060(2)	0.038(1)	0.036(1)	0.043(1)	–0.011(1)	–0.014(1)	–0.002(1)
C(20)	2*i*	0.8961(3)	0.6495(3)	1.0971(2)	0.050(2)	0.048(2)	0.035(1)	–0.011(1)	–0.012(1)	–0.004(1)
C(21)	2*i*	0.7598(3)	0.6426(3)	1.0890(2)	0.045(2)	0.052(2)	0.033(1)	–0.013(1)	–0.001(1)	–0.001(1)
C(22)	2*i*	0.7115(3)	0.6474(3)	0.9898(2)	0.032(1)	0.051(2)	0.040(1)	–0.014(1)	–0.002(1)	0.001(1)
C(23)	2*i*	0.9261(3)	0.6707(2)	0.9098(2)	0.030(1)	0.033(1)	0.039(1)	–0.011(1)	–0.008(1)	–0.001(1)
C(24)	2*i*	1.0124(3)	0.6848(2)	0.8140(2)	0.031(1)	0.034(1)	0.043(1)	–0.013(1)	–0.007(1)	0.001(1)
C(25)	2*i*	0.6118(3)	0.4442(3)	0.7756(2)	0.026(1)	0.056(2)	0.053(2)	–0.018(1)	–0.005(1)	–0.002(1)
C(26)	2*i*	0.7030(3)	0.3856(3)	0.6722(2)	0.046(2)	0.055(2)	0.052(2)	–0.022(1)	–0.009(1)	–0.007(1)
C(27)	2*i*	0.8703(3)	0.3691(3)	0.6620(2)	0.038(2)	0.041(2)	0.038(1)	–0.007(1)	–0.003(1)	0.003(1)
N(1)	2*i*	0.5352(3)	0.8377(2)	0.7413(2)	0.040(1)	0.043(1)	0.042(1)	–0.013(1)	–0.006(1)	–0.001(1)
N(2)	2*i*	0.6842(2)	0.6958(2)	0.5680(2)	0.032(1)	0.050(1)	0.037(1)	–0.014(1)	–0.0049(9)	0.002(1)
N(3)	2*i*	0.9488(2)	0.6969(2)	0.7224(2)	0.035(1)	0.048(1)	0.038(1)	–0.019(1)	–0.0031(9)	0.003(1)
N(4)	2*i*	0.7906(2)	0.6616(2)	0.9021(2)	0.028(1)	0.045(1)	0.034(1)	–0.0147(9)	–0.0049(9)	0.0003(9)
O(1)	2*i*	0.5908(2)	0.5585(2)	0.7935(2)	0.034(1)	0.054(1)	0.057(1)	–0.0197(9)	0.0042(9)	–0.0103(9)
O(2)	2*i*	0.5636(3)	0.3790(2)	0.8366(2)	0.067(2)	0.069(2)	0.080(2)	–0.038(1)	0.014(1)	0.002(1)
O(3)	2*i*	0.9673(3)	0.2679(2)	0.6267(2)	0.057(1)	0.049(1)	0.103(2)	–0.006(1)	0.008(1)	–0.014(1)
O(4)	2*i*	0.8990(2)	0.4619(2)	0.6884(2)	0.0273(9)	0.050(1)	0.058(1)	–0.0105(8)	–0.0038(8)	–0.0071(9)
C(1′)	2*i*	–0.2416(9)	1.0399(7)	0.8737(8)	0.106(5)	0.121(5)	0.26(1)	–0.020(4)	–0.014(6)	0.032(6)

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Reference

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