Crystal structure of tetraaqua-di(1-carboxamide-1,1-dicyanomethanide)-manganese(II) dihydrate, $Mn[C(CN)_2CONH_2]_2(H_2O)_4 \cdot 2H_2O$

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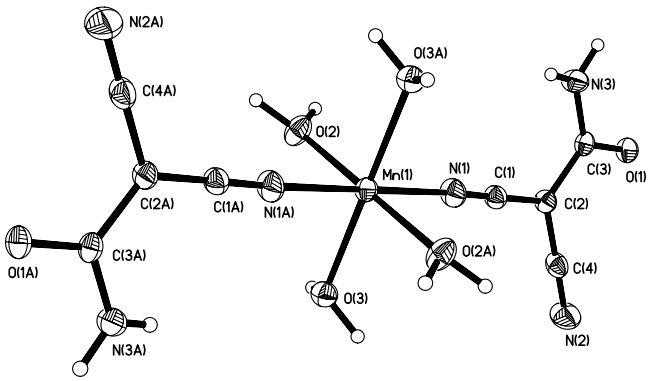

Abstract

$C_8H_{16}MnN_6O_8$, monoclinic, $P12_1/n1$ (No. 14), $a = 9.516(1)$ Å, $b = 7.4299(8)$ Å, $c = 12.187(1)$ Å, $\beta = 108.749(2)°$, $V = 815.9$ Å^3, $Z = 2$, $R_{gt}(F) = 0.026$, $wR_{ref}(F^2) = 0.072$, $T = 293$ K.

Source of material

A solution of sodium carbamyldicyanomethanide 0.3767 g (2.87 mmol, in 15 ml H_2O) was added to a solution of $Mn(ClO_4)_2 \cdot 6H_2O$ 0.5193 g (1.44 mmol, in 20 ml H_2O) and then the mixed solution was stirred for a few minutes. The transparent colorless single crystal was obtained after the mixed solution was allowed to stand at room temperature for a few of days.

Discussion

The structure of the title complex consists of a manganese cation coordinated by two carbamyldicyanomethanide anions and four water molecules. The divalent manganese cation is coordinated by two N atoms of the –CN radicals and four O atoms of H_2O molecules. The bond distance of Mn—N is 2.201(2) Å and the Mn—O bond distances are 2.160(2) Å and 2.206(1) Å. The angle of N–Mn–N is 180.0° and the angles dealing with N–Mn–O and O–Mn–O are in the range of 87.43(6)° to 92.57(6)°. The bond distances and the angles indicate that the divalent manganese cation has a little distorted octahedral environment. The complex is similar to that of the divalent nickel cation and the divalent cobalt cation [1,2]. The neutral complexes connect to each other through the hydrogen bonds to form a one-dimensional chain and the one-dimensional chains connect through the hydrogen bonds to form the countless microporosities along the *a* axis. In addition, there exist four non-coordinated H_2O molecules in each unit cell.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.15 × 0.20 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	8.58 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	50.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4181, 1435
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1177
$N(param)_{refined}$:	139
Programs:	SHELXS-97 [3], SHELXL-97 [4]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*e*	0.940(2)	0.509(3)	0.370(2)	0.037(6)
H(2)	4*e*	0.842(3)	0.396(3)	0.407(2)	0.050(7)
H(3)	4*e*	0.376(3)	0.175(3)	0.285(2)	0.10(1)
H(4)	4*e*	0.252(2)	0.156(3)	0.332(2)	0.084(9)
H(5)	4*e*	0.451(4)	–0.264(4)	0.335(1)	0.11(1)
H(6)	4*e*	0.554(3)	–0.348(4)	0.450(4)	0.17(2)
H(7)	4*e*	0.225(2)	0.419(3)	0.423(2)	0.073(8)
H(8)	4*e*	0.330(5)	0.588(4)	0.449(4)	0.19(2)

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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*a*	1/2	0	1/2	0.0276(2)	0.0310(3)	0.0370(3)	–0.0002(2)	0.0177(2)	0.0016(2)
N(1)	4*e*	0.6856(2)	0.0436(2)	0.4323(2)	0.038(1)	0.047(1)	0.053(1)	–0.0056(8)	0.028(1)	–0.0005(8)
N(2)	4*e*	1.0240(2)	–0.2014(3)	0.3071(2)	0.061(1)	0.046(1)	0.083(2)	–0.001(1)	0.037(1)	–0.019(1)
N(3)	4*e*	0.9058(2)	0.4011(3)	0.3768(2)	0.043(1)	0.033(1)	0.064(1)	–0.0002(8)	0.032(1)	–0.0018(9)
O(1)	4*e*	1.0786(1)	0.2696(2)	0.3152(1)	0.0306(7)	0.0420(8)	0.0420(8)	–0.0026(6)	0.0207(7)	0.0014(6)
O(2)	4*e*	0.3491(2)	0.1074(2)	0.3410(1)	0.0364(8)	0.058(1)	0.0451(9)	0.0109(7)	0.0209(7)	0.0145(8)
O(3)	4*e*	0.4719(2)	–0.2676(2)	0.4172(1)	0.0411(8)	0.0361(8)	0.0392(9)	–0.0007(6)	0.0162(7)	–0.0002(6)
O(4)	4*e*	0.2878(2)	0.4920(2)	0.4814(1)	0.0382(8)	0.0375(9)	0.057(1)	–0.0043(7)	0.0204(8)	–0.0044(8)
C(1)	4*e*	0.7841(2)	0.0597(3)	0.3980(2)	0.032(1)	0.033(1)	0.037(1)	–0.0027(8)	0.014(1)	–0.0006(8)
C(2)	4*e*	0.9053(2)	0.0812(3)	0.3579(2)	0.030(1)	0.034(1)	0.037(1)	–0.0018(8)	0.0188(9)	–0.0026(9)
C(3)	4*e*	0.9668(2)	0.2534(3)	0.3493(2)	0.025(1)	0.039(1)	0.027(1)	0.0002(8)	0.0101(8)	0.0008(8)
C(4)	4*e*	0.9711(2)	–0.0739(3)	0.3300(2)	0.034(1)	0.040(1)	0.046(1)	–0.0083(9)	0.020(1)	–0.005(1)

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