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Crystal structure of *trans*-dichlorido-bis(2,6-pyridinediyl-bis(3-pyridinyl) methanone)-κ^2N,N'-manganese(II), $C_{34}H_{22}Cl_2MnN_6O_4$

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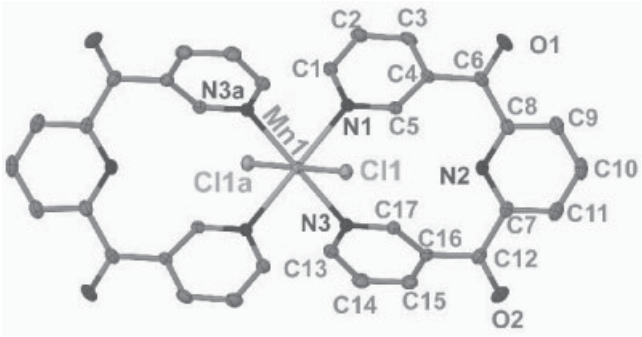

Abstract

$C_{17}H_{11}ClMn_{0.50}N_3O_2$, monoclinic, $P2_1/c$ (no. 14), a = 8.542(1) Å, b = 16.186(2) Å, c = 11.652(2) Å, β = 103.160(3)°, V = 1568.6 Å^3, Z = 2, $R_{gt}(F)$ = 0.0415, $wR_{ref}(F^2)$ = 0.1069, T = 293 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.40×0.44×0.56 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	6.41 cm^{-1}
Diffractometer, scan mode:	Bruker APEXII CCD area detector, ω
$2\theta_{max}$:	56.68°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10814, 3925
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2702
$N(param)_{refined}$:	214
Programs:	SHELX [7]

Source of material

All reagents and solvents from commercial sources were used without further purification. 2,6-pyridinediyl-bis(3-pyridinyl)-methanone was obtained via the methodology according to the literature [1]. Reaction of the 2,6-pyridinediylbis(3-pyridinyl)-methanone (58 mg, 0.2 mmol) with $MnCl_2$ (13 mg, 0.05 mmol) succeeded in a mixed solvent of 3 mL acetonitrile and 2 mL methanol with stirring at room temperature. After two hours, the clear solution was filtrated and the filtration was left in air for about one week. *trans*-[$Mn(C_{17}H_{11}N_3O_2)_2Cl_2$] was obtained as block-shaped crystals (yield 28.9 mg, 41% based on ligand).

Discussion

The coordination chemistry of di-2-pyridinylmethanone (common name di-2-pyridylketone, also abbreviated as DPK) [2] and its the positional isomer [3, 4] have attracted wide attention because these ligands can exist in their neat, alcoholated hemiketal and hydrated gem-diol forms in either uncharged or deprotonated state in coordination to metal centers. In comparison, the coordination chemistry of analogous oligo-pyridyl ketone remains little explored, several copper(II) and silver(I) complexes of 2,6-pyridinediylbis(3-pyridinyl)methanone [1, 5] and 2,6-pyridinediylbis(4-pyridinyl)methanone [6] in their neat forms were recently reported. Herein, we report one new Mn(II) complex with 2,6-pyridinediylbis(3-pyridinyl)methanone. In the mononuclear complex, the Mn(II) located on an inversion center is surround by four 3-pyridyl N atoms from two 2,6-pyridinediylbis(3-pyridinyl)methanone and two Cl^- ligands. The four N atoms are in the equatorial sites, while the two chlorido ligands are in axial postions, as shown in the Figure. (symmetry code a: $-x$, $-y$+1, $-z$+1). For the C–H···π interaction occurred between C14–H and the N1a-containing π system (symmetry code $-x$+1, $-y$+1, $-z$+1), the C···centroid is 3.556(3) Å, and the C–H···centroid angle equals 143.9°.

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

Atom	Site	x	y	z	U_{iso}
H(1A)	4*e*	0.0276	0.3083	0.4394	0.048
H(2A)	4*e*	0.1002	0.2184	0.3089	0.056
H(3A)	4*e*	0.1679	0.2695	0.1414	0.053
H(5A)	4*e*	0.0673	0.4960	0.2393	0.039
H(9A)	4*e*	0.2445	0.4974	–0.1148	0.064
H(10A)	4*e*	0.3665	0.6242	–0.1243	0.085
H(11A)	4*e*	0.4413	0.7028	0.0452	0.080
H(13A)	4*e*	0.3457	0.5493	0.6548	0.049
H(14A)	4*e*	0.5891	0.6052	0.6454	0.055
H(15A)	4*e*	0.6251	0.6581	0.4684	0.052
H(17A)	4*e*	0.1642	0.6087	0.3285	0.041

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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*	0	$\frac{1}{2}$	$\frac{1}{2}$	0.0355(3)	0.0249(2)	0.0268(3)	–0.0001(2)	0.0124(2)	–0.0003(2)
Cl(1)	4*e*	–0.15642(7)	0.58913(3)	0.34257(5)	0.0453(3)	0.0311(3)	0.0349(3)	0.0060(2)	0.0132(3)	0.0063(2)
C(1)	4*e*	0.0516(3)	0.3292(1)	0.3710(2)	0.058(2)	0.030(1)	0.032(1)	–0.001(1)	0.011(1)	0.000(1)
C(2)	4*e*	0.0967(3)	0.2749(2)	0.2939(2)	0.069(2)	0.026(1)	0.045(2)	0.002(1)	0.011(1)	–0.004(1)
C(3)	4*e*	0.1366(3)	0.3053(2)	0.1944(2)	0.057(2)	0.038(1)	0.039(2)	0.002(1)	0.013(1)	–0.015(1)
C(4)	4*e*	0.1298(3)	0.3896(1)	0.1737(2)	0.039(1)	0.035(1)	0.028(1)	–0.0034(9)	0.008(1)	–0.0068(9)
C(5)	4*e*	0.0773(3)	0.4396(1)	0.2544(2)	0.038(1)	0.027(1)	0.033(1)	–0.0006(9)	0.010(1)	–0.0027(9)
N(1)	4*e*	0.0406(2)	0.4111(1)	0.3522(2)	0.041(1)	0.0278(9)	0.031(1)	–0.0006(8)	0.0118(8)	–0.0020(8)
C(6)	4*e*	0.1712(4)	0.4216(2)	0.0642(2)	0.076(2)	0.053(2)	0.036(2)	–0.013(1)	0.024(1)	–0.014(1)
O(1)	4*e*	0.1414(5)	0.3795(2)	–0.0230(2)	0.229(4)	0.095(2)	0.053(2)	–0.077(2)	0.073(2)	–0.042(1)
N(2)	4*e*	0.2963(2)	0.5473(1)	0.1595(2)	0.045(1)	0.041(1)	0.034(1)	–0.0036(9)	0.0166(9)	0.0006(9)
C(8)	4*e*	0.2504(3)	0.5038(2)	0.0596(2)	0.046(1)	0.049(1)	0.033(1)	–0.000(1)	0.014(1)	–0.003(1)
C(9)	4*e*	0.2759(4)	0.5300(2)	–0.0478(2)	0.065(2)	0.065(2)	0.033(2)	–0.004(2)	0.016(1)	–0.000(1)
C(10)	4*e*	0.3482(5)	0.6050(2)	–0.0533(3)	0.104(3)	0.077(2)	0.040(2)	–0.011(2)	0.032(2)	0.013(2)
C(11)	4*e*	0.3932(4)	0.6514(2)	0.0472(3)	0.095(2)	0.058(2)	0.053(2)	–0.021(2)	0.030(2)	0.010(2)
C(7)	4*e*	0.3661(3)	0.6206(2)	0.1522(2)	0.051(2)	0.043(1)	0.040(2)	–0.007(1)	0.018(1)	0.004(1)
C(12)	4*e*	0.4198(3)	0.6701(2)	0.2630(2)	0.048(1)	0.042(1)	0.051(2)	–0.009(1)	0.020(1)	0.003(1)
C(13)	4*e*	0.3574(3)	0.5728(2)	0.5843(2)	0.048(1)	0.041(1)	0.034(1)	–0.007(1)	0.007(1)	–0.004(1)
C(14)	4*e*	0.5049(3)	0.6049(2)	0.5789(2)	0.039(1)	0.052(2)	0.042(2)	–0.006(1)	–0.001(1)	–0.007(1)
C(15)	4*e*	0.5262(3)	0.6367(2)	0.4741(2)	0.036(1)	0.043(1)	0.052(2)	–0.008(1)	0.013(1)	–0.011(1)
C(16)	4*e*	0.3982(3)	0.6362(1)	0.3777(2)	0.042(1)	0.029(1)	0.039(1)	–0.0060(9)	0.017(1)	–0.006(1)
C(17)	4*e*	0.2522(3)	0.6062(1)	0.3923(2)	0.037(1)	0.033(1)	0.033(1)	–0.0047(9)	0.009(1)	–0.003(1)
N(3)	4*e*	0.2307(2)	0.5736(1)	0.4930(2)	0.038(1)	0.034(1)	0.032(1)	–0.0053(8)	0.0102(9)	–0.0026(8)
O(2)	4*e*	0.4852(3)	0.7365(1)	0.2609(2)	0.091(2)	0.050(1)	0.070(2)	–0.033(1)	0.031(1)	0.000(1)

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