Crystal structure of bis(acridinium)tetrachloromanganate(II), $[(C_{13}H_{10}N)_2][MnCl_4]$

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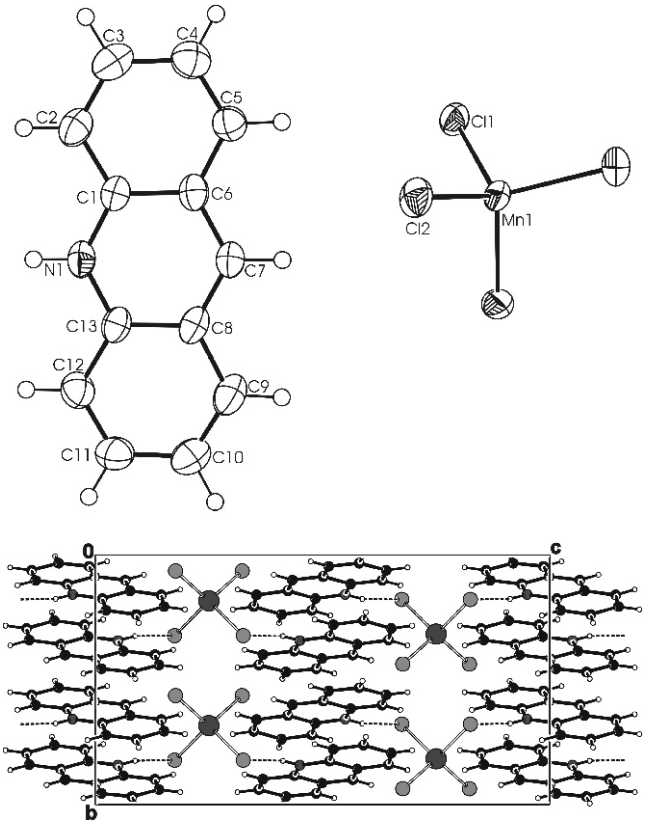

Abstract

$C_{26}H_{20}Cl_4MnN_2$, monoclinic, *C*2/*c* (no. 15), a = 12.838(1) Å, b = 10.2435(9) Å, c = 18.730(2) Å, β = 92.553(2)°, V = 2460.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.055, $wR_{ref}(F^2)$ = 0.139, T = 200 K.

Source of material

The title compound was unexpectedly obtained as a byproduct from the reaction of $MnCl_2 \cdot 4H_2O$ (0.1982 g, 1.001 mmol) with acridine (0.3538 g, 1.974 mmol) and pyridine-2-carboxylic acid (0.2464 g, 2.001 mmol) in EtOH (20 ml). After reflux of the reaction mixture for 3 h, the formed white and yellow precipitates were separated by filtration, washed with EtOH and ether, to give the main product as a white powder (0.2321 g). The yellow byproduct (0.0672 g) was obtained from the mixture of filtrate and washing solution by recrystallizing at -85 °C. Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a CH_3NO_2 solution of the byproduct.

Experimental details

Hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms with d(C—H) = 0.95 Å, d(N—H) = 0.88 Å and U_{iso}(H) = 1.2 U_{eq}(C,N). The highest peak (0.51 e Å^{-3}) and the deepest hole (–0.55 e Å^{-3}) in the difference Fourier map are located 1.13 Å and 1.76 Å from the atoms Cl1 and Cl2, respectively.

Discussion

The asymmetric unit of the title compound contains a protonated acridinium cation and half of an anionic Mn(II) complex (figure, top). In the complex, the Mn(II) ion is four-coordinated by Cl atoms in a tetrahedral environment. The complex is disposed about a twofold rotation axis running in the [010] direction passing through the Mn atom. The distances d(Mn—Cl) are nearly equal (2.339(1) Å and 2.378(1) Å) and ∠Cl–Mn–Cl (106.32(7)° – 116.50(7)°) are close to the tetrahedral angle. These values are similar to those observed in the analogous compound $(C_{10}H_{10}N_3)_2[MnCl_4]$ [1]. Two cations interact with one complex by means of intermolecular N–H···Cl hydrogen bonds with d(N···Cl) = 3.193(4) Å (figure, bottom). Moreover, the cations display numerous intermolecular π-π interactions between adjacent six-membered rings. The shortest distance between Cg1 (the centroid of ring N1–C13) and Cg2^i (ring C1–C6; symmetry code i: ½–x,½–y,–z) is 3.693(2) Å, and the dihedral angle between the ring planes is 1.0(2)°.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.09 × 0.12 × 0.22 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.89 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	56.58°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8973, 3042
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1401
$N(param)_{refined}$:	150
Programs:	SHELXS-97, SHELXL-97 [2], ORTEP-3 [3], PLATON [4]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	8*f*	0.4796	0.1755	–0.0875	0.046
H(2)	8*f*	0.3395	0.1205	–0.1713	0.054
H(3)	8*f*	0.1692	0.0495	–0.1872	0.061
H(4)	8*f*	0.0674	0.0044	–0.0880	0.060
H(5)	8*f*	0.1386	0.0251	0.0258	0.049
H(7)	8*f*	0.2937	0.0785	0.1069	0.042
H(9)	8*f*	0.4485	0.1380	0.1870	0.060
H(10)	8*f*	0.6160	0.2175	0.1973	0.063
H(11)	8*f*	0.7056	0.2714	0.0955	0.059
H(12)	8*f*	0.6318	0.2418	–0.0172	0.053

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)	4*e*	0	0.18329(9)	¼	0.0374(6)	0.0398(6)	0.0251(5)	0	0.0046(4)	0
Cl(1)	8*f*	–0.10435(8)	0.3225(1)	0.17459(6)	0.0403(7)	0.0572(8)	0.0358(7)	0.0050(6)	0.0019(5)	0.0099(5)
Cl(2)	8*f*	0.10760(9)	0.0631(1)	0.17684(6)	0.0506(7)	0.0562(8)	0.0393(7)	0.0061(6)	0.0130(6)	–0.0080(6)
N(1)	8*f*	0.4432(3)	0.1575(3)	–0.0500(2)	0.043(2)	0.040(2)	0.033(2)	–0.004(2)	0.011(2)	0.003(2)
C(1)	8*f*	0.3437(3)	0.1155(4)	–0.0619(2)	0.042(3)	0.035(3)	0.031(3)	0.001(2)	0.006(2)	0.002(2)
C(2)	8*f*	0.2997(4)	0.1014(4)	–0.1311(2)	0.059(3)	0.042(3)	0.034(3)	–0.003(2)	0.004(2)	0.003(2)
C(3)	8*f*	0.1989(4)	0.0598(4)	–0.1402(3)	0.068(4)	0.044(3)	0.041(3)	–0.002(3)	–0.008(3)	–0.001(2)
C(4)	8*f*	0.1377(4)	0.0318(4)	–0.0807(3)	0.050(3)	0.044(3)	0.055(3)	–0.002(2)	0.000(3)	0.001(2)
C(5)	8*f*	0.1798(3)	0.0443(4)	–0.0137(3)	0.039(3)	0.041(3)	0.043(3)	0.002(2)	0.002(2)	0.003(2)
C(6)	8*f*	0.2840(3)	0.0855(4)	–0.0014(2)	0.041(3)	0.029(3)	0.037(3)	0.003(2)	0.011(2)	–0.003(2)
C(7)	8*f*	0.3321(3)	0.0996(4)	0.0662(2)	0.042(3)	0.031(3)	0.033(3)	–0.001(2)	0.009(2)	0.001(2)
C(8)	8*f*	0.4327(4)	0.1427(4)	0.0764(2)	0.053(3)	0.032(3)	0.028(3)	0.002(2)	0.006(2)	–0.001(2)
C(9)	8*f*	0.4840(4)	0.1596(4)	0.1452(3)	0.068(4)	0.050(3)	0.033(3)	–0.004(3)	0.007(3)	–0.003(2)
C(10)	8*f*	0.5830(4)	0.2062(5)	0.1513(3)	0.061(3)	0.056(3)	0.041(3)	–0.002(3)	–0.006(3)	–0.007(2)
C(11)	8*f*	0.6368(4)	0.2377(5)	0.0901(3)	0.045(3)	0.053(3)	0.049(3)	–0.009(2)	–0.005(3)	–0.002(2)
C(12)	8*f*	0.5935(4)	0.2214(4)	0.0236(3)	0.045(3)	0.043(3)	0.045(3)	–0.009(2)	0.007(2)	–0.003(2)
C(13)	8*f*	0.4906(3)	0.1737(4)	0.0155(2)	0.047(3)	0.029(3)	0.030(3)	0.002(2)	0.007(2)	0.000(2)

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