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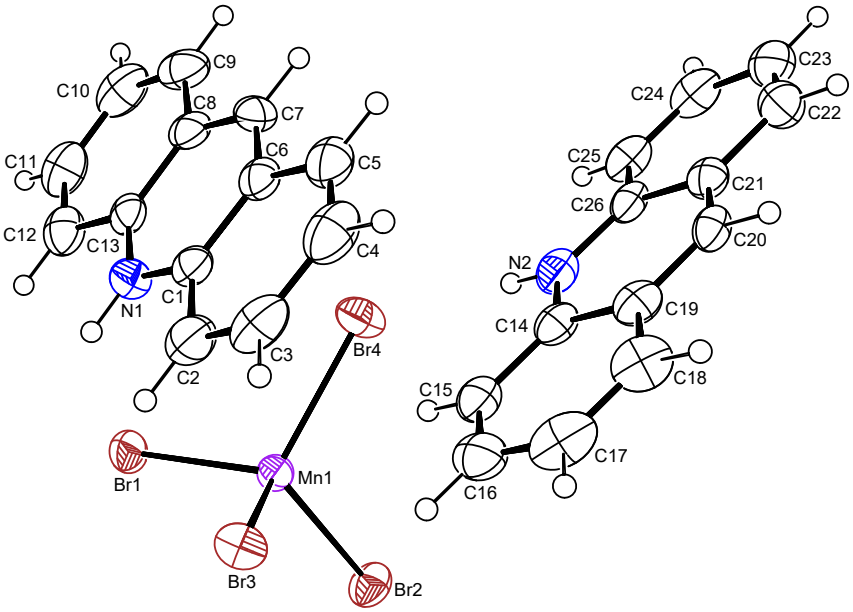
Crystal structure of bis(acridinium) tetrabromidomanganate(II), $C_{26}H_{20}Br_4MnN_2$



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Abstract

$C_{26}H_{20}Br_4MnN_2$, triclinic, $P\bar{1}$ (no. 2), a = 9.5638(11) Å, b = 10.2013(11) Å, c = 14.6758(16) Å, α = 97.534(4)°, β = 93.522(4)°, γ = 113.149(4)°, V = 1295.1(3) Å^3, Z = 2, $R_{gt}(F)$ = 0.0389, $wR_{ref}(F^2)$ = 0.0953, T = 223 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.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.26 × 0.16 × 0.08 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	6.70 mm^{-1}
Diffractometer, scan mode:	PHOTON II M14, φ and ω
θ_{max}, completeness:	26.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	30,570, 5112, 0.056
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3919
$N(param)_{refined}$:	298
Programs:	Bruker [1], SHELX [2], ORTEP-3 [3], PLATON [4]

Source of materials

The solution of $MnBr_2{\cdot}4H_2O$ (0.5733 g, 1.999 mmol) and acridine (0.3586 g, 2.001 mmol) in EtOH (30 mL) was refluxed for 3 h. After removal of the undissolved, the solvent was evaporated under vacuum and the residue was washed with ether, and dried at 50 °C, to give a yellow powder (0.6889 g). Crystals were obtained by slow evaporation from a CH_3NO_2 solution at room temperature.

Experimental details

Hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms with d(C–H) = 0.94 Å, d(N–H) = 0.87 Å and U_{iso}(H) = 1.2U_{eq}(C, N) with the help of the SHELXL program

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Mn1	0.40020 (8)	0.39511 (8)	0.25612 (5)	0.03692 (17)
Br1	0.43430 (6)	0.61337 (6)	0.37133 (4)	0.05285 (16)
Br2	0.18278 (6)	0.36088 (6)	0.13598 (4)	0.05191 (15)
Br3	0.32849 (6)	0.17663 (6)	0.33136 (4)	0.05208 (15)
Br4	0.63971 (6)	0.44887 (6)	0.18034 (4)	0.05615 (16)
N1	0.7478 (4)	0.3916 (4)	0.4387 (3)	0.0436 (10)
H1N	0.6720	0.3829	0.4707	0.052*
N2	0.7080 (5)	0.1680 (5)	0.0585 (3)	0.0466 (10)
H2N	0.6898	0.2414	0.0826	0.056*
C1	0.7663 (5)	0.2701 (5)	0.4046 (3)	0.0393 (11)
C2	0.6684 (6)	0.1338 (6)	0.4230 (4)	0.0548 (14)
H2	0.5875	0.1250	0.4585	0.066*
C3	0.6934 (7)	0.0144 (6)	0.3882 (4)	0.0629 (16)
H3	0.6300	−0.0766	0.4011	0.075*
C4	0.8119 (8)	0.0254 (7)	0.3335 (4)	0.0655 (17)
H4	0.8256	−0.0582	0.3095	0.079*
C5	0.9064 (7)	0.1555 (7)	0.3152 (4)	0.0570 (15)
H5	0.9857	0.1615	0.2790	0.068*
C6	0.8870 (5)	0.2835 (6)	0.3502 (3)	0.0426 (11)
C7	0.9822 (5)	0.4181 (6)	0.3356 (3)	0.0424 (11)
H7	1.0620	0.4269	0.2993	0.051*
C8	0.9638 (5)	0.5416 (6)	0.3730 (3)	0.0435 (12)
C9	1.0610 (6)	0.6843 (7)	0.3609 (4)	0.0603 (16)
H9	1.1429	0.6982	0.3256	0.072*
C10	1.0355 (8)	0.8009 (7)	0.4001 (4)	0.0663 (17)
H10	1.0996	0.8946	0.3917	0.080*
C11	0.9134 (8)	0.7801 (6)	0.4530 (4)	0.0613 (16)
H11	0.8985	0.8615	0.4803	0.074*
C12	0.8173 (7)	0.6490 (6)	0.4661 (4)	0.0527 (13)
H12	0.7360	0.6385	0.5015	0.063*
C13	0.8408 (5)	0.5260 (5)	0.4256 (3)	0.0389 (11)
C14	0.6397 (5)	0.0397 (5)	0.0898 (3)	0.0402 (11)
C15	0.5394 (6)	0.0240 (6)	0.1585 (3)	0.0491 (12)
H15	0.5195	0.1029	0.1849	0.059*
C16	0.4731 (6)	−0.1046 (7)	0.1853 (4)	0.0586 (15)
H16	0.4064	−0.1154	0.2309	0.070*
C17	0.5020 (7)	−0.2248 (7)	0.1461 (5)	0.0657 (17)
H17	0.4530	−0.3142	0.1657	0.079*
C18	0.5984 (7)	−0.2133 (6)	0.0811 (4)	0.0595 (15)
H18	0.6171	−0.2938	0.0561	0.071*
C19	0.6711 (5)	−0.0788 (5)	0.0511 (3)	0.0420 (11)
C20	0.7688 (5)	−0.0603 (5)	−0.0154 (3)	0.0379 (11)
H20	0.7898	−0.1392	−0.0406	0.046*
C21	0.8367 (5)	0.0676 (5)	−0.0464 (3)	0.0407 (11)
C22	0.9387 (6)	0.0864 (7)	−0.1150 (4)	0.0544 (14)
H22	0.9628	0.0094	−0.1402	0.065*
C23	1.0009 (7)	0.2158 (7)	−0.1439 (4)	0.0623 (16)
H23	1.0699	0.2287	−0.1882	0.075*
C24	0.9632 (7)	0.3319 (6)	−0.1082 (4)	0.0606 (15)
H24	1.0059	0.4201	−0.1301	0.073*
C25	0.8671 (6)	0.3179 (6)	−0.0433 (4)	0.0521 (13)
H25	0.8419	0.3954	−0.0207	0.063*
C26	0.8044 (5)	0.1863 (5)	−0.0094 (3)	0.0401 (11)

(AFIX 43 options) [2]. The highest peak (1.39 e Å^{-3}) and the deepest hole (−0.73 e Å^{-3}) in the difference Fourier map are located 0.62 Å and 0.75 Å from the atoms H10 and Br4, respectively.

Comment

The crystal structure of the related chlorido–Mn complex (H-acr)$_2$[MnCl$_4$] (acr = acridine) was previously determined in the monoclinic space group *C*2/*c* [5].

The asymmetric unit of the title compound contains two protonated acridinium cations and an anionic Mn(II) complex. In the complex, the Mn(II) ion is four-coordinated by Br atoms in a tetrahedral environment. The Mn–Br bond lengths are nearly equal with d(Mn–Br) = 2.4886(9)–2.5242(9) Å and the <Br–Mn–Br bond angles (105.84(3)°–113.38(3)°) are close to the tetrahedral angle. Two cations interact with one complex by means of intermolecular N–H···Br hydrogen bonds with d(N···Br) = 3.372(4) and 3.479(5) Å [4]. The cations are stacked in columns along [100]. In the columns, numerous intermolecular π–π interactions between adjacent six-membered rings are present. The shortest distance between Cg1 (the centroid of ring N1–C13) and Cg2^i (ring C8–C13; symmetry code i: 2 – *x*, 1 – *y*, 1 – *z*) is 3.669(3) Å, and the dihedral angle between the ring planes is 1.0(2)° [4].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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