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Bis(dimethylammonium) tetrabromidomanganate(II)

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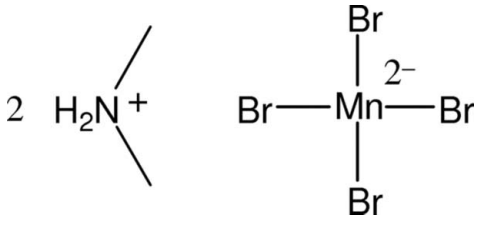
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–N) = 0.011 Å; R factor = 0.050; wR factor = 0.113; data-to-parameter ratio = 26.7.

The title compound, $(C_2H_{10}N)_2[MnBr_4]$, consists of discrete dimethylammonium cations and tetrabromidomanganate(II) anions which are held together in the crystal structure by N—H···Br hydrogen bonds.

Related literature

For related literature, see: Daoud (1976); Kückmann (2007); Lerner *et al.* (2005); Pabst *et al.* (1990); Waskowska (1994); Williams *et al.* (1992); Salah *et al.* (1982).



Experimental

Crystal data

$(C_2H_8N)_2[MnBr_4]$
M_r = 466.77
Monoclinic, $P2_1/n$
a = 8.1854 (7) Å
b = 11.7492 (9) Å
c = 15.1444 (11) Å
β = 95.087 (6)°
V = 1450.7 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 11.89 mm^{-1}
T = 173 (2) K
0.22 × 0.21 × 0.18 mm

Data collection

Stoe IPDSII two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.090, T_{max} = 0.114
12230 measured reflections
2693 independent reflections
2105 reflections with $I > 2\sigma(I)$
R_{int} = 0.079

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.113
S = 1.06
2693 reflections
101 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.85 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···Br1	0.92	2.47	3.377 (6)	167
N1—H1A···Br2^{i}	0.92	2.89	3.492 (7)	125
N1—H1A···Br4^{i}	0.92	2.78	3.466 (6)	132
N2—H2A···Br4^{i}	0.92	2.56	3.401 (7)	153
N2—H2B···Br3^{ii}	0.92	2.49	3.346 (7)	155

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x, -y+1, -z+1$.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2275).

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supplementary materials

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Bis(dimethylammonium) tetrabromidomanganate(II)

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Comment

Recently we have found that the manganese complex $[Mn(CO)_5Br]$ can easily transformed into Mn(II) and $[Mn(CO)_5]_2$ in the presence of strong nucleophiles such as $NaSSitBu_3$ or $Na_2PSitBu_3$ (Kückmann, 2007; Lerner *et al.*, 2005). In attempting to synthesize the 1,4-phenylene-brigded Mn(I) scorpionate (I) from the corresponding lithium scorpionate and $[Mn(CO)_5Br]$ in the presence of $[NMe_2H_2][Br]$, we obtained $[NMe_2H_2]_2[MnBr_4]$ as a side-product. X-ray quality crystals of $[NMe_2H_2]_2[MnBr_4]$ were grown by diffusion of hexane into a solution of $[NMe_2H_2]_2[MnBr_4]$ in tetrahydrofuran at ambient temperature.

The title compound, $2(C_2H_{10}N)^+.MnBr_4^{2-}$, consists of discrete dimethylammonium cations und tetrabromo-manganese anions which are held together in the crystal by N—H···Br hydrogen bonds. The title compound is isostructural with $2(C_2H_{10}N)^+\cdot HgBr_4^{2-}$ (Pabst *et al.*, 1990) $2(C_2H_{10}N)^+\cdot CdBr_4^{2-}$ (Daoud, 1976; Waskowska, 1994), $2(C_2H_{10}N)^+\cdot HgCl_4^{2-}$ (Salah *et al.*, 1982) and $2(C_2H_{10}N)^+\cdot CoCl_4^{2-}$ (Williams *et al.*, 1992).

Experimental

By the reaction of the 1,4-phenylene-brigded Li scorpionate (I) (0.59 g, 1.10 mmol) with $[Mn(CO)_5Br]$ (0.64 g, 2.31 mmol) and $[NMe_2H_2][Br]$ (ca. 0.2 mmol) in 30 ml THF $[NMe_2H_2]_2[MnBr_4]$ was obtained as a side-product. X-ray quality crystals of $[NMe_2H_2]_2[MnBr_4]$ were grown by diffusion of hexane into a solution of $[NMe_2H_2]_2[MnBr_4]$ in tetrahydrofuran at ambient temperature.

Refinement

H atoms were refined with fixed individual displacement parameters [$U_{iso}(H) = 1.2\ U_{eq}(C, N)$] using a riding model with N—H = 0.92Å or C—H = 0.98 Å.

Figures

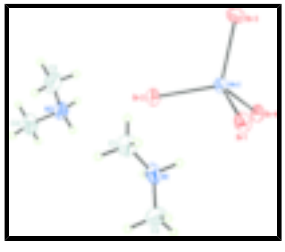

Fig. 1. Perspective view of the title compound, with the atom numbering scheme; displacement ellipsoids are at the 50% probability level; H atoms are drawn as small spheres of arbitrary radii.

Bis(dimethylammonium) tetrabromidoomanganese(II)

Crystal data

$(C_2H_8N)_2[MnBr_4]$	F_{000} = 876
M_r = 466.77	D_x = 2.137 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 10816 reflections
a = 8.1854 (7) Å	θ = 3.5–25.4º
b = 11.7492 (9) Å	μ = 11.89 mm^{-1}
c = 15.1444 (11) Å	T = 173 (2) K
β = 95.087 (6)º	Block, yellow
V = 1450.7 (2) Å^3	0.22 × 0.21 × 0.18 mm
Z = 4	

Data collection

Stoe IPDSII two-circle diffractometer	2693 independent reflections
Radiation source: fine-focus sealed tube	2105 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.079
T = 173(2) K	θ_{max} = 25.6º
ω scans	θ_{min} = 3.4º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	h = −9→9
T_{min} = 0.090, T_{max} = 0.114	k = −14→14
12230 measured reflections	l = −18→16

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0617P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.113	$\Delta\rho_{max}$ = 1.57 e Å^{-3}
S = 1.06	$\Delta\rho_{min}$ = −0.85 e Å^{-3}
2693 reflections	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
101 parameters	Extinction coefficient: 0.0058 (5)
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Mn1	0.28760 (13)	0.70597 (8)	0.54751 (8)	0.0256 (3)
Br1	0.53567 (9)	0.78194 (6)	0.63851 (6)	0.0345 (2)
Br2	0.28514 (9)	0.49320 (6)	0.55910 (6)	0.0334 (2)
Br3	0.03107 (10)	0.79225 (7)	0.59464 (6)	0.0391 (2)
Br4	0.32489 (10)	0.76510 (6)	0.38986 (5)	0.0325 (2)
N1	0.6983 (9)	0.5194 (5)	0.6703 (5)	0.0348 (15)
H1A	0.6640	0.4705	0.6250	0.042*
H1B	0.6635	0.5913	0.6536	0.042*
C1	0.6192 (11)	0.4864 (7)	0.7498 (6)	0.0393 (19)
H1C	0.4998	0.4886	0.7369	0.059*
H1D	0.6518	0.5395	0.7981	0.059*
H1E	0.6533	0.4091	0.7675	0.059*
C2	0.8784 (10)	0.5188 (8)	0.6814 (7)	0.046 (2)
H2C	0.9215	0.5413	0.6257	0.068*
H2D	0.9173	0.4421	0.6978	0.068*
H2E	0.9168	0.5725	0.7283	0.068*
N2	0.2655 (9)	0.1858 (5)	0.5713 (5)	0.0383 (16)
H2A	0.3660	0.2205	0.5698	0.046*
H2B	0.1967	0.2153	0.5257	0.046*
C3	0.2002 (14)	0.2138 (8)	0.6547 (8)	0.055 (3)
H3A	0.1902	0.2966	0.6599	0.082*
H3B	0.2744	0.1849	0.7040	0.082*
H3C	0.0919	0.1787	0.6566	0.082*
C4	0.2855 (13)	0.0612 (8)	0.5559 (9)	0.061 (3)
H4A	0.3293	0.0493	0.4984	0.092*
H4B	0.1787	0.0235	0.5560	0.092*
H4C	0.3615	0.0292	0.6030	0.092*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0270 (6)	0.0230 (5)	0.0264 (6)	0.0006 (4)	−0.0006 (4)	0.0000 (4)

Br1	0.0352 (4)	0.0282 (4)	0.0379 (5)	−0.0011 (3)	−0.0093 (3)	−0.0036 (3)
Br2	0.0396 (4)	0.0233 (3)	0.0368 (4)	−0.0012 (3)	0.0013 (3)	0.0034 (3)
Br3	0.0315 (4)	0.0391 (4)	0.0475 (5)	0.0064 (3)	0.0072 (3)	−0.0062 (4)
Br4	0.0392 (4)	0.0292 (4)	0.0283 (4)	−0.0015 (3)	−0.0014 (3)	0.0054 (3)
N1	0.051 (4)	0.026 (3)	0.028 (4)	0.006 (3)	0.001 (3)	0.005 (3)
C1	0.047 (5)	0.037 (4)	0.034 (5)	−0.004 (3)	−0.001 (4)	−0.004 (3)
C2	0.035 (4)	0.052 (5)	0.050 (6)	−0.006 (4)	0.008 (4)	0.004 (4)
N2	0.038 (4)	0.030 (3)	0.045 (5)	−0.006 (3)	−0.001 (3)	0.006 (3)
C3	0.062 (6)	0.049 (5)	0.056 (7)	−0.004 (4)	0.017 (5)	0.010 (5)
C4	0.062 (6)	0.033 (4)	0.089 (9)	−0.004 (4)	0.010 (6)	0.011 (5)

Geometric parameters (Å, °)

Mn1—Br3	2.4924 (13)	C2—H2D	0.9800
Mn1—Br2	2.5062 (12)	C2—H2E	0.9800
Mn1—Br1	2.5145 (13)	N2—C3	1.453 (13)
Mn1—Br4	2.5309 (14)	N2—C4	1.493 (11)
N1—C2	1.469 (11)	N2—H2A	0.9200
N1—C1	1.469 (11)	N2—H2B	0.9200
N1—H1A	0.9200	C3—H3A	0.9800
N1—H1B	0.9200	C3—H3B	0.9800
C1—H1C	0.9800	C3—H3C	0.9800
C1—H1D	0.9800	C4—H4A	0.9800
C1—H1E	0.9800	C4—H4B	0.9800
C2—H2C	0.9800	C4—H4C	0.9800
Br3—Mn1—Br2	111.93 (5)	N1—C2—H2E	109.5
Br3—Mn1—Br1	111.01 (5)	H2C—C2—H2E	109.5
Br2—Mn1—Br1	109.08 (5)	H2D—C2—H2E	109.5
Br3—Mn1—Br4	109.49 (5)	C3—N2—C4	114.3 (8)
Br2—Mn1—Br4	110.02 (5)	C3—N2—H2A	108.7
Br1—Mn1—Br4	105.10 (5)	C4—N2—H2A	108.7
C2—N1—C1	114.5 (7)	C3—N2—H2B	108.7
C2—N1—H1A	108.6	C4—N2—H2B	108.7
C1—N1—H1A	108.6	H2A—N2—H2B	107.6
C2—N1—H1B	108.6	N2—C3—H3A	109.5
C1—N1—H1B	108.6	N2—C3—H3B	109.5
H1A—N1—H1B	107.6	H3A—C3—H3B	109.5
N1—C1—H1C	109.5	N2—C3—H3C	109.5
N1—C1—H1D	109.5	H3A—C3—H3C	109.5
H1C—C1—H1D	109.5	H3B—C3—H3C	109.5
N1—C1—H1E	109.5	N2—C4—H4A	109.5
H1C—C1—H1E	109.5	N2—C4—H4B	109.5
H1D—C1—H1E	109.5	H4A—C4—H4B	109.5
N1—C2—H2C	109.5	N2—C4—H4C	109.5
N1—C2—H2D	109.5	H4A—C4—H4C	109.5
H2C—C2—H2D	109.5	H4B—C4—H4C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1B···Br1	0.92	2.47	3.377 (6)	167
N1—H1A···Br2^{i}	0.92	2.89	3.492 (7)	125
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N2—H2A···Br4^{i}	0.92	2.56	3.401 (7)	153
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Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*, −*y*+1, −*z*+1.

Fig. 1