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Dimethylethylammonium bromide

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(N–C) = 0.008 Å
Disorder in main residue
R factor = 0.033
wR factor = 0.072
Data-to-parameter ratio = 21.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_4H_{12}N^+\cdot Br^-$, is a hydrolysis product of the 1-(dibromoboryl)ferrocene–dimethylethylamine (1/1) adduct. The metric symmetry is apparently orthorhombic *C*-centred, but the intensity statistics indicate unambigously the correct Laue symmetry, *viz*. primitive monoclinic. The ethyl group is disordered over two sites. In addition to the hydrogen bond from the NH group to the Br^- ion, the packing is stabilized by several weak C—H···Br hydrogen bonds.

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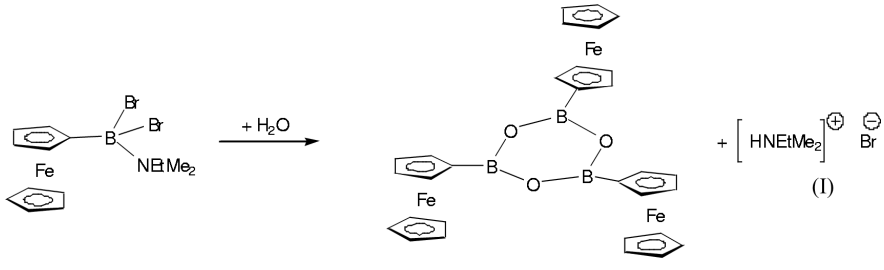
Comment

Recently, our group reported the properties and X-ray crystal structure determination of triferrocenylboroxine (Ma *et al.*, 2002). We report here the hydrolysis of the 1-(dibromoboryl)ferrocene–dimethylethylamine (1/1) adduct and the X-ray crystal structure analysis of the resulting dimethylethylammonium bromide, $[HNEtMe_2]Br$. The synthesis of triferrocenylboroxine and of the title compound, (I), was achieved by the hydrolysis of the 1-(dibromoboryl)ferrocene–dimethylethylammonium (1/1) adduct, as indicated in the reaction scheme below.



There is a strong hydrogen bond linking the NH group and the Br^- ion. Furthermore, the packing is stabilized by several weak C—H···Br hydrogen bonds.

Experimental

The title compound, (I), was obtained by stirring a solution of 0.107 g 1-(dibromoboryl)ferrocene–dimethylethylamine (1/1) in 10 ml CH_2Cl_2 in the presence of 0.05 ml H_2O at ambient temperature. The solution was subjected to gas-phase diffusion of diethyl ether, to yield colourless crystals of $[HNEtMe_2]Br$. The NMR spectra were recorded on a Bruker AM 250 spectrometer. $[HNEtMe_2]Br$: ^1H NMR ($CDCl_3$, internal TMS): δ 1.45 (*t*, CH_3, ^3J = 7.2 Hz), 2.78 (*d*, $2NCH_3$, ^3J = 5.3 Hz), 3.14 (*dq*, NCH_2, ^3J = 7.2 Hz, ^3J = 5.3 Hz), 11.26 (*m*, NH). $^{13}C\{^1H\}$ NMR ($CDCl_3$, internal TMS): δ 10.0 (*s*, CH_3), 42.6 (*s*, 2 NCH_3), 53.3 (*s*, NCH_2).

Crystal data

$C_4H_{12}N^+\cdot Br^-$
M_r = 154.06
Monoclinic, $P2_1/c$
a = 7.0797 (8) Å
b = 7.3352 (9) Å
c = 14.1594 (15) Å
β = 100.030 (9)°
V = 724.07 (14) Å^3
Z = 4
D_x = 1.413 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1423 reflections
θ = 3.5–24.2°
μ = 5.57 mm^{-1}
T = 173 (2) K
Block, colourless
0.19 × 0.17 × 0.13 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 1990; Blessing, 1995)
T_{min} = 0.381, T_{max} = 0.485
5255 measured reflections
1675 independent reflections
1171 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 27.8°
$h = -9 \rightarrow 9$
$k = -8 \rightarrow 9$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.072
S = 0.85
1675 reflections
78 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.032P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.029
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Br1	0.85 (6)	2.37 (6)	3.217 (3)	173 (5)
C2—H2A···Br1^{i}	0.98	2.90	3.819 (5)	156
C1—H1A···Br1^{ii}	0.98	2.88	3.849 (4)	171
C2—H2B···Br1^{iii}	0.98	3.03	3.913 (5)	151
C2—H2C···Br1^{iv}	0.98	3.05	3.931 (5)	150
C1—H1C···Br1^{iii}	0.98	3.00	3.895 (5)	152

Symmetry codes: (i) $-x, y-\frac{1}{2}, \frac{1}{2}-z$; (ii) $1-x, 1-y, 1-z$; (iii) $-x, 1-y, 1-z$; (iv) $x, y-1, z$.

The initial cell determination yielded an orthorhombic *C*-centred cell with a = 18.197 Å, b = 21.698 Å, c = 7.335 Å and V = 2896.1 Å^3. After data collection, the R_{int} value for the orthorhombic setting (0.474) indicated that the symmetry should be lowered to monoclinic *P* with an R_{int} of 0.063. Structure solution and refinement was successful in $P2_1/c$. The ethyl group is disorded over two sites. The site-occupation factors refined to 0.54 (1):0.46 (1). H atoms bonded to C atoms were refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl})], using a riding model, with C—H = 0.99 Å and methyl C—H = 0.98 Å. The H atom bonded to the N atom was freely refined.

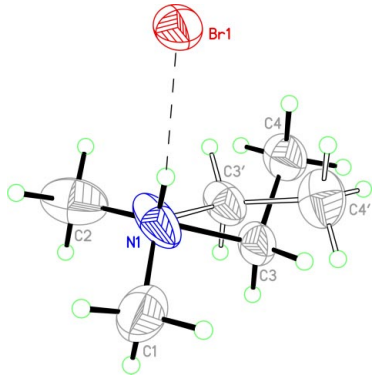


Figure 1
Perspective view of the title compound with the atom numbering; displacement ellipsoids are drawn at the 50% probability level. The major component of the disordered ethyl group (C3 and C4) is drawn with full bonds, the minor component (C3′ and C4′) with open bonds.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97.

References

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Ma, K., Lerner, H.-W., Scholz, S., Bats, J., Bolte, M. & Wagner, M. (2002). *J. Organomet. Chem.* **664**, 94–105.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1991). *SHELXTL-Plus.* Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.