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Dimethyl(2-trimethylsilylethyl)[(2-trimethylsilylethyl)dimethylammonio]ammonium tetrakis[3,5-bis(trifluoromethyl)phenyl]borate at 130 K

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The crystal structure of the title compound, $C_{14}H_{39}N_2Si_2^+\cdot C_{32}H_{12}F_{24}B^-$, reveals a centrosymmetric cation consisting of two 2-(trimethylsilylethyl)dimethylamine molecules held together by a symmetrical N···H···N hydrogen bond. The N—H distance is 1.347 (2) Å, the N···N distance is 2.695 (3) Å and the N···H···N angle is 180°. The tetrakis[3,5-bis(trifluoromethyl)phenyl]borate counter-ion lies on a crystallographic twofold axis of symmetry. The trifluoromethyl groups are disordered and were refined with partial occupancies.

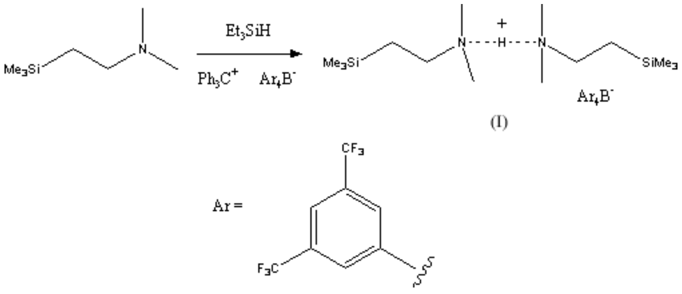
Key indicators

Single-crystal X-ray study
T = 130 K
Mean σ(C–C) = 0.003 Å
Disorder in solvent or counterion
R factor = 0.046
wR factor = 0.126
Data-to-parameter ratio = 11.9

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

Comment

The structure of the title compound, (I) (Fig. 1), consists of (2-trimethylsilylethyl)dimethylamine hydrogen-bonded to its ammonium ion, the combined cation lying on a crystallographic inversion centre. The staggered N···H···N hydrogen bond is characterized by an N—H distance of 1.347 (2) Å and an N···N distance of 2.695 (3) Å. This compares with the quinicludine–quinuclidinium cation, which has an N···N separation of 2.689 Å (Mareque Rivas & Brammer, 1999). The counter-ion lies on a twofold axis of symmetry.



Experimental

The title compound, (I), was isolated as a by-product from the reaction of a catalytic quantity of triphenylcarbenium tetrakis[3,5-bis(trifluoromethyl)phenyl]borate, (II), with (2-trimethylsilylethyl)dimethylamine, (III), in triethylsilane, and presumably arises as a result of the presence of adventitious water in the reaction medium.

Crystal data

$C_{14}H_{39}N_2Si_2^+\cdot C_{32}H_{12}F_{24}B^-$
$M_r = 1154.88$
Orthorhombic, *Pbcn*
$a = 17.1047 (11)$ Å
$b = 16.8652 (11)$ Å
$c = 18.9828 (12)$ Å
$V = 5476.0 (6)$ Å^3
$Z = 4$
$D_x = 1.401$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 6546 reflections
$\theta = 2.4$–$24.8°$
$\mu = 0.18$ mm^{-1}
$T = 130.0 (2)$ K
Block, colourless

0.30 × 0.30 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.776$, $T_{\max} = 0.970$
29606 measured reflections
4835 independent reflections
3972 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.038$
$\theta_{\max} = 25.0°$
$h = -19 \rightarrow 20$
$k = -20 \rightarrow 19$
$l = -12 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.126$
$S = 1.04$
4835 reflections
407 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0645P)^2 + 2.2182P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.010$
$\Delta\rho_{\max} = 0.30$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}

H atoms, other than the one at the center of the cation, were positioned geometrically and refined as riding, with C—H distances of 0.93–0.97 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. Each of the four CF_3 groups was refined as disordered over two orientations, with occupancies 0.932:0.068(5), 0.935:0.065 (4), 0.633:0.367 (13) and 0.958:0.042 (4). Isotropic displacement parameters were refined for the F atoms with low site-occupancy. 'Same distance' constraints were applied to the disordered CF_3 groups to keep geometries reasonable.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

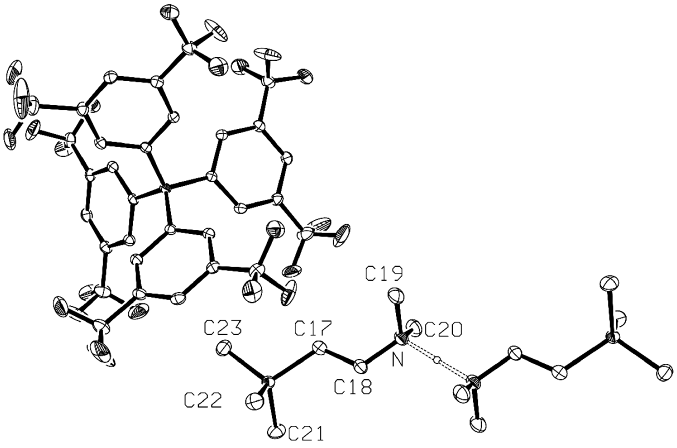


Figure 1
Displacement ellipsoid plot of (I), ellipsoids are at the 20% probability level. Hydrogen atoms have been omitted.

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