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Dihydridotetrakis(3-picoline)silicon dibromide chloroform tetrasolvate

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.013 Å
R factor = 0.093
wR factor = 0.268
Data-to-parameter ratio = 23.3

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

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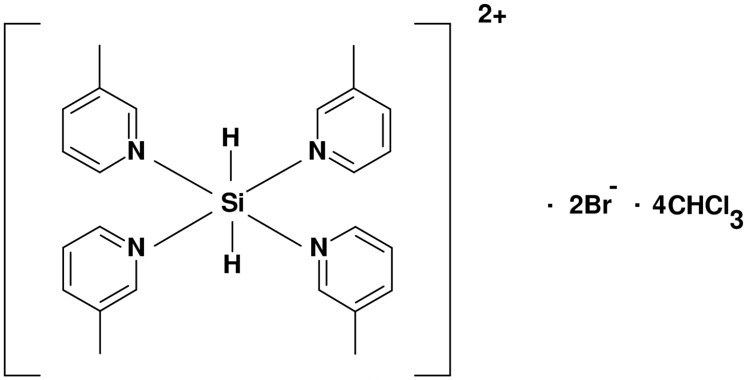
The title compound, $C_{24}H_{30}N_4Si^{2+}\cdot 2Br^-\cdot 4CHCl_3$, (I), contains a hexacoordinated Si atom located on a crystallographic centre of inversion. The coordination of the Si atom can be described as a slightly distorted octahedron, with the 3-picoline ligands in the equatorial plane and the two H atoms occupying axial positions. It is remarkable that (I) is not isomorphous with its analogue where the Br^- ions are substituted by Cl^- ions.

Comment

Lewis acid–base complexes between silicon compounds and nitrogen bases have in recent years been the subject of many structural studies (Hensen *et al.*, 1997, 1988; Chuit *et al.*, 1993). Apart from $[H_2Si(3\text{-picoline})_4]^{2+}\cdot 2Cl^-\cdot 4CHCl_3$ (Fleischer *et al.*, 1996), $[H_2Si(\text{pyridine})_4]^{2+}\cdot 2Cl^-\cdot 4CHCl_3$ (Hensen *et al.*, 1998), $[H_2Si(3,4\text{-lutidine})_4]^{2+}\cdot 2Cl^-\cdot 6CHCl_3$ (Hensen *et al.*, 2000), $[H_2Si(3,5\text{-lutidine})_4]^{2+}\cdot 2Br^-\cdot 2CH_3CN$ (Bolte *et al.*, 2000), $[H_2Si(4\text{-picoline})_4]^{2+}\cdot 2Cl^-\cdot 6CHCl_3$ (Stumpf & Bolte, 2001) and $[H_2Si(4\text{-picoline})_4]^{2+}\cdot 2Br^-\cdot 6CHCl_3$ (Bolte & Faber, 2001), the crystal structure of the title compound, (I), is a further example with the structural element $H_2Si(\text{pyridine})_4$.



(I)

The coordination of the Si atom can be described as a slightly distorted octahedron, with the 3-picoline ligands in the equatorial plane and the two H atoms occupying axial positions. The two Br^- ions are displaced from the first sphere of coordination. It is remarkable that (I) is not isomorphous with its analogue in which the Br^- ions are substituted by Cl^- ions (Fleischer *et al.*, 1996). A least-squares fit of all non-H atoms of the cations of both structures (r.m.s. deviation 0.259 Å) shows that there are only minor differences between them, *e.g.* the angle between the aromatic rings is 90.0 (3)° in (I) and 96.9° in the structure containing the Cl^- ions. However, the

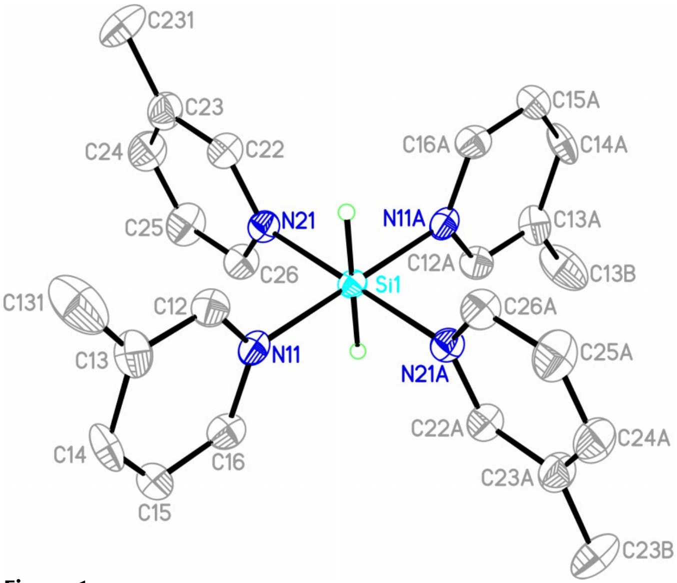


Figure 1
A perspective view of the cation of (I) with the atom-numbering scheme. Displacement ellipsoids are at the 50% probability level. H atoms, except those attached to Si, have been omitted for clarity.

packing patterns of the two structures are completely different.

Experimental

The title compound was prepared as described by Faber (2000).

Crystal data

$C_{28}H_{34}Br_2Cl_{12}N_4Si$
$M_r = 1039.90$
Triclinic, $P\bar{1}$
$a = 8.840$ (1) Å
$b = 10.059$ (1) Å
$c = 13.330$ (1) Å
$\alpha = 97.65$ (1)°
$\beta = 90.68$ (1)°
$\gamma = 108.63$ (1)°
$V = 1111.35$ (19) Å^3
$Z = 1$
$D_x = 1.554$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7621 reflections
$\theta = 1$–25°
$\mu = 2.60$ mm^{-1}
$T = 173$ (2) K
Block, colourless
0.40 × 0.35 × 0.20 mm

Data collection

Siemens CCD three-circle diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.37$, $T_{\max} = 0.60$
22 862 measured reflections
5095 independent reflections
3406 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.087$
$\theta_{\max} = 27.5°$
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = -17 \rightarrow 17$
160 standard reflections
frequency: 1200 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.093$
$wR(F^2) = 0.268$
$S = 1.11$
5095 reflections
219 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0856P)^2 + 12.6187P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 2.39$ e Å^{-3}
$\Delta\rho_{\min} = -0.85$ e Å^{-3}

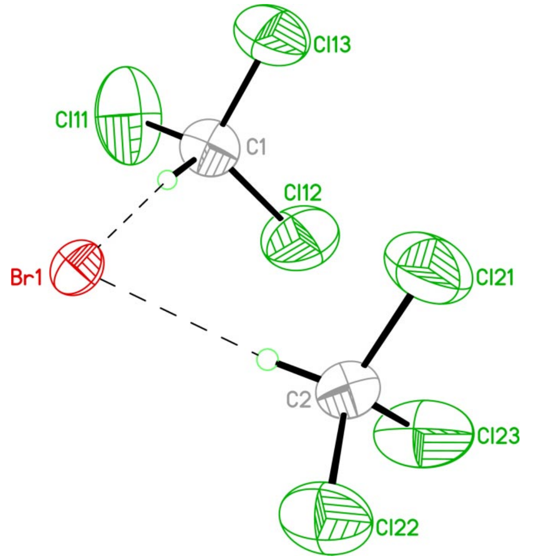


Figure 2
A perspective view of the anion and the solvent molecules of (I) with the atom-numbering scheme. Displacement ellipsoids are at the 50% probability level and H atoms are drawn as small spheres of arbitrary radii.

Table 1
Selected geometric parameters (Å, °).

Si1—N11	1.990 (7)	Si1—N21	1.966 (6)
N21—Si1—N11	89.8 (3)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H11···Br1$^{\rm i}$	1.00	2.58	3.565 (12)	170
C2—H21···Br1$^{\rm ii}$	1.00	2.52	3.519 (11)	174

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

All H atoms were located by difference Fourier synthesis and refined with fixed individual displacement parameters [U(H) = 1.5 $U_{\rm eq}$[C(methyl)] or U(H) = 1.2$U_{\rm eq}$(C)] using a riding model with C—H(aromatic) = 0.95, C—H(methyl) = 0.98, or C—H(tertiary) = 1.00 Å. The H atom bonded to Si was refined isotropically applying a restraint of 1.40 (1) Å to the Si—H distance The methyl groups attached to the aromatic rings were allowed to rotate about their local threefold axis.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); 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).

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