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Dihydridotetrakis(4-picoline-*N*)silicon dichloride chloroform hexasolvate

Thorsten Stumpf[a] and Michael Bolte[b]*

[a]Forschungszentrum Rossendorf, Institut für Radiochemie, PO Box 510119, 01324 Dresden, Germany, and [b]Institut für Organische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany

Correspondence e-mail:
bolte@chemie.uni-frankfurt.de

The title compound, $[SiH_2(C_6H_7N)_4]Cl_2\cdot6CHCl_3$ or $C_{24}H_{30}N_4Si^{2+}\cdot2Cl^-\cdot6CHCl_3$, 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 4-picoline ligands in the equatorial plane and the two H atoms occupying axial positions. The title compound is isomorphous with its analogue where the Cl ions are substituted by Br ions.

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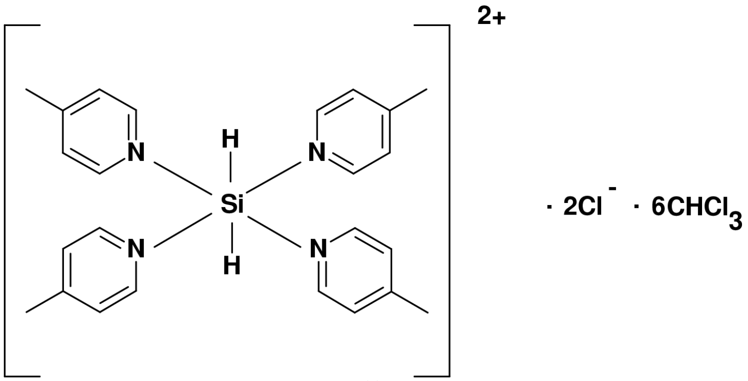
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.009 Å
R factor = 0.069
wR factor = 0.181
Data-to-parameter ratio = 17.6

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

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.*, 1988, 1997; Chuit *et al.*, 1993). In addition to $[H_2Si(3\text{-picoline})_4]Cl_2\cdot4CHCl_3$ (Fleischer *et al.*, 1996), $[H_2Si(\text{pyridine})_4]Cl_2\cdot4CHCl_3$ (Hensen *et al.*, 1998), $[H_2Si(N\text{-methylimidazole})_4]Cl_2\cdot3CHCl_3$ (Hensen, Mayr-Stein *et al.*, 2000), $[H_2Si(3,4\text{-lutidine})_4]Cl_2\cdot6CHCl_3$ (Hensen, Kettner *et al.*, 2000) and $[H_2Si(3,5\text{-lutidine})_4]Br_2\cdot2CH_3CN$ (Bolte *et al.*, 2000), the crystal structure of the title compound, (I), is a further example with the structural element SiH_2. The title compound is an ionic complex with the Si atom located on a crystallographic centre of inversion and with half a molecule in the asymmetric unit. The coordination of the Si atom can be described as a slightly distorted octahedron, with the picoline ligands in the equatorial plane and the two H atoms occupying axial positions. Two Cl^- ions are displaced from the first sphere of coordination. The title compound is isomorphous with its analogue where the Cl^- ions are substituted by Br^- ions (Bolte & Faber, 2001).



(I)

The title compound was prepared in two different ways. Firstly, the reaction of 4-picoline with bis(dichlorosilyl)-methylamine affords directly the crystals of the title compound. A second way to synthesize (I) is the reaction of

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liquid dichlorosilane with 4-picoline to form H_2SiCl_2(4-picoline)$_2$. With an excess of base in chloroform this adduct forms again the title compound.

Experimental

The title compound was prepared in two different ways: (i) 0.60 ml (3.46 mmol) bis(dichlorosilyl)methylamine, prepared and purified according to Fleischer *et al.* (1995), was dissolved in 5.0 ml chloroform. Then 1.0 ml (10.24 mmol) 4-picoline was added in portions of 0.2 ml. After several days, colourless crystals had formed. They were isolated, washed with *n*-hexane, and a single-crystal was selected for the diffraction experiment. (ii) 1.0 ml (13.9 mmol) H_2SiCl_2 (liquid) was condensed into 50 ml *n*-hexane and 6.0 ml (66.4 mmol) 4-picoline was added within 30 min whilst the solution was stirred and kept at 213 K. The solution was then allowed to warm to room temperature. The formation of a white precipitate was observed. This precipitate was washed with *n*-hexane and dried *in vacuo*. Yield: 100%; $C_{12}H_{16}Cl_2N_2Si$ (287.3), calculated: C 50.2, H 5.6, Cl 24.7, N 9.8%; found: C 48.3, H 5.7, Cl 25.4, N9.0%; IR (Nujol, NaCl, cm^{-1}): 2922.1 (*vs*), 2855.3 (*vs*), 2165.5 (*br,s*), 1626.8 (*s*), 1581.1 (*s*), 1459.6 (*vs*), 1376.2 (*vs*), 1315.0 (*m*), 1203.7 (m), 997.8 (*s*), 992.6 (*s*), 825.4 (*m*). Single crystals were grown from a solution of 150 mg (0.52 mmol) H_2SiCl_2(4-picoline)$_2$ and 0.2 ml (2.0 mmol) 4-picoline in 3.0 ml $CHCl_3$ at 277 K within 5 d. After cell determination, the selected crystal turned out to contain the identical structure as the former crystal, which was used for data collection. All procedures were carried out under argon (dried with 4 Å molecular sieves and Sicapent) either on a vacuum line or in a glove-box. 4–Picoline was distilled from CaH_2 and sealed in ampules. H_2SiCl_2 (Aldrich, 99.99%) was used without further purification.

Crystal data

$C_{24}H_{30}N_4Si^{2+}\cdot 2Cl^-\cdot 6CHCl_3$
$M_r = 1189.72$
Triclinic, $P\overline{1}$
$a = 9.3854$ (8) Å
$b = 11.0958$ (9) Å
$c = 12.7495$ (11) Å
$\alpha = 93.018$ (5)°
$\beta = 101.092$ (5)°
$\gamma = 97.548$ (4)°
$V = 1287.47$ (19) Å^3

$Z = 1$
$D_x = 1.534$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 35 reflections
$\theta = 2.3$–15.2°
$\mu = 1.11$ mm^{-1}
$T = 150$ (2) K
Plate, colourless
0.4 × 0.3 × 0.2 mm

Data collection

Stoe four-circle diffractometer
ω/θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.665$, $T_{\max} = 0.808$
4775 measured reflections
4518 independent reflections
3349 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.017$
$\theta_{\max} = 25.0°$
$h = -11 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 15$
3 standard reflections
frequency: 120 min
intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.181$
$S = 1.19$
4518 reflections
256 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 3.9420P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.81$ e Å^{-3}
$\Delta\rho_{\min} = -0.56$ e Å^{-3}

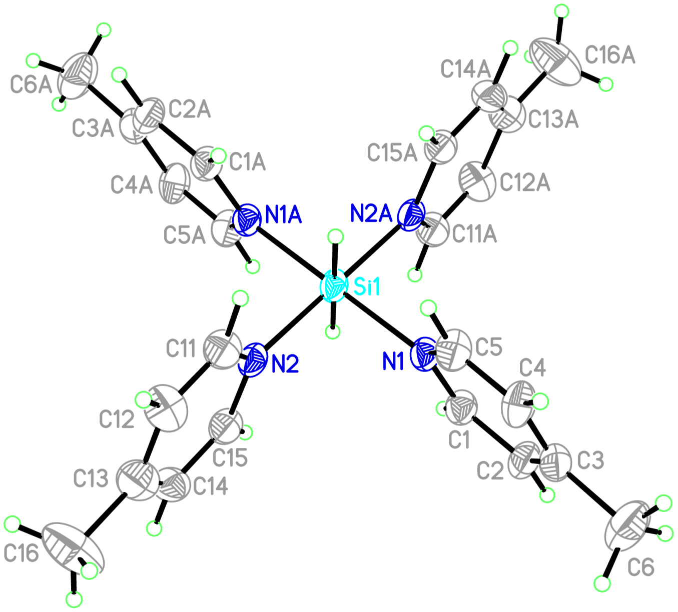

Figure 1

A perspective view of the cation 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.

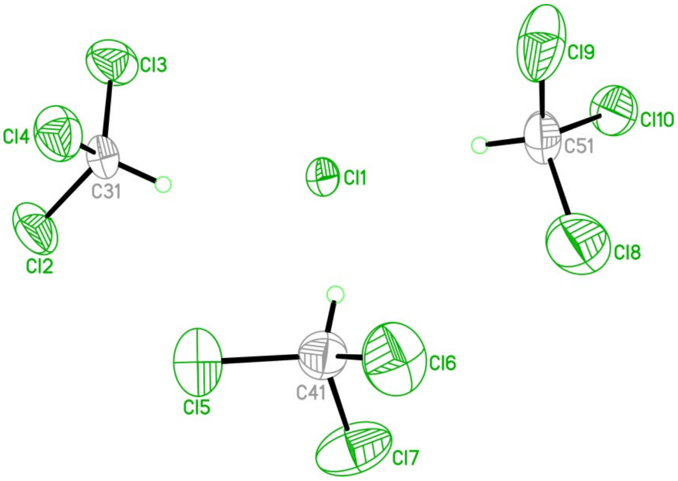

Figure 2

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—N2	1.955 (4)	Si1—H1SI	1.37 (4)
Si1—N1	1.955 (4)		
N2—Si1—N1	89.34 (17)	N1—Si1—H1SI	90.8 (18)
N2—Si1—H1SI	90.4 (18)		

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

Data collection: *DIF*4 (Stoe & Cie, 1992); cell refinement: *DIF*4; data reduction: *REDU*4 (Stoe & Cie, 1992); 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: *CIFTAB* in *SHELXL*97.

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