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***trans*-Tetrachlorobis(4-methylpyridine)germanium(IV)**

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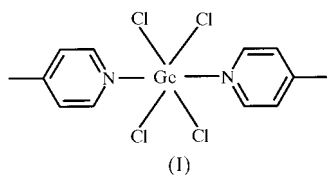
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The structure of the title compound, [GeCl₄(C₆H₇N)₂], (I), is the first example of an addition compound of GeCl₄ with two aromatic nitrogen bases. The molecule, with essential *D*_{2h} symmetry, has crystallographic *C*_{2h} symmetry. The environment around the Ge atom can be described as a slightly distorted octahedron with the 4-methylpyridine ligands occupying axial positions and the four chloro ligands in the equatorial plane. The structure of (I) is isomorphous with the corresponding silicon halides.

Comment

The tetrahalides of germanium, as an element of the fourth period, form interesting Lewis-acid–base complexes. Addition of a Lewis base enlarges the coordination number of the germanium centre from four to five (Bilton & Webster, 1972) or six (Abel, 1958; Fergusson *et al.*, 1959; Hulme *et al.*, 1960; Muetterties, 1960). A similar reaction is shown by the tetrahalides of silicon (Wannagat *et al.*, 1954, 1968; Beattie *et al.*, 1964; Mayr-Stein, 1998; Spangenberg, 1999). We present here the first structure, (I), of an addition compound of GeCl₄ with aromatic nitrogen bases as part of our studies on the structure and properties of germanium tetrahalides and tertiary amines.



Compound (I) belongs essentially to the symmetry point group *D*_{2h}, with the Ge atom on a special position of site symmetry *2/m* and the 4-methylpyridine moieties located on a crystallographic mirror plane. Only the Cl atoms and two H atoms of each methyl group occupy a general position. The Ge centre appears in a nearly ideal octahedral environment,

where the two 4-methylpyridine ligands occupy axial positions and the four chloro ligands lie in the equatorial plane. There are only minor deviations from the perfect octahedral coordination. The structure of (I) is isomorphous with the corresponding silicon halides (Hensen *et al.*, 2000).

Experimental

Compound (I) was prepared according to the method of Faber (2000). Single crystals were obtained from a chloroform solution.

Crystal data

[GeCl₄(C₆H₇N)₂]
M_r = 400.64
 Orthorhombic, *Pmna*
a = 7.154 (2) Å
b = 7.933 (2) Å
c = 13.731 (3) Å
V = 779.3 (3) Å³
Z = 2
D_x = 1.707 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 2407 reflections
 θ = 1–25°
 μ = 2.638 mm⁻¹
T = 173 (2) K
 Block, colourless
 0.60 × 0.20 × 0.20 mm

Data collection

Siemens CCD three-circle diffractometer
 ω scans
 Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
 T_{\min} = 0.301, T_{\max} = 0.621
 3722 measured reflections
 1036 independent reflections
 895 reflections with $I > 2\sigma(I)$

R_{int} = 0.031
 θ_{max} = 28.25°
 h = –9 → 5
 k = –10 → 8
 l = –17 → 16
 10 standard reflections
 frequency: 300 min
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)]$ = 0.026
 $wR(F^2)$ = 0.062
 S = 1.064
 1036 reflections
 57 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0334P)^2 + 0.1042P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.41 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.51 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0063 (14)

All H atoms were located by difference Fourier synthesis and refined with fixed individual displacement parameters [$U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $U(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{Me}})$] using a riding model with aromatic C–H = 0.95 or methyl C–H = 0.98 Å. The H atoms of the methyl groups are disordered. Two orientations differing by a 60° rotation about the C_{Py}–C_{Me} bond could be identified.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *SHELXL97*.

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