

**Related literature.** Final atomic parameters are listed in Table 1.\* The bond lengths and angles are listed in Table 2. Fig. 1 shows a thermal-ellipsoid plot (Johnson, 1965) of the molecule with atom labelling and Fig. 2 displays the hydrogen-bond network in the crystal.

The title compound is one of the ligands used to model the active centre of the metalloprotein of Fe<sup>II</sup> called the ferroquinone complex (Boinnard, Cassoux, Petrouleas, Savariault & Tuchagues, 1990). It contains two imidazole rings and a carboxylic group and represents a simplification of the fundamental structural elements that are believed to make up the ferroquinone complex. The HBIP molecule is a zwitterion, the predominant form for the amino acids. The two imidazole rings of the same molecule make a dihedral angle of 66.7 (1)°; the relative spatial positions of the N atoms and the carboxylic group are such that the molecule is expected to act as a tridentate ligand after removal of a proton and yield

\* Lists of structure factors, H-atom parameters, anisotropic thermal parameters and least-squares-planes equations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54980 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA0265]

neutral  $M^{II}(\text{BIP})_2$  complexes containing  $M^{II}\text{N}_4\text{O}_2$  chromophores.

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## Structure of *tert*-Butylammonium Hexachlorotellurate(IV)

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**Abstract.**  $[(\text{CH}_3)_3\text{CNH}_3]_2\text{TeCl}_6$ ,  $M_r = 488.6$ , monoclinic,  $P2_1/a$ ,  $a = 12.620$  (3),  $b = 15.719$  (5),  $c = 9.945$  (2) Å,  $\beta = 90.14$  (2)°,  $V = 1973.0$  (9) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.645$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 2.318$  mm<sup>-1</sup>,  $F(000) = 960$ ,  $T = 289$  K, final  $R = 0.051$  for 2630 unique reflections with  $I > 3\sigma(I)$ . The structure may be regarded as having monoclinically distorted *anti*-CdI<sub>2</sub>-type packing of the composite ions forming puckered layers parallel to the *ab* plane. Each complex anion is surrounded by six cations, while each cation has three anion neighbours with Cl...N distances in the range 3.233 (7)–3.584 (8) Å. The C—N axes of the cations are almost parallel to

the *c* axis, the N atoms pointing in the layers. The  $\text{TeCl}_6^{2-}$  octahedron is distorted and noncentrosymmetric.

**Experimental.** Crystals of  $[(\text{CH}_3)_3\text{CNH}_3]_2\text{TeCl}_6$  were grown by slow evaporation of a concentrated HCl solution. A yellow prismatic crystal with dimensions 0.35 × 0.30 × 0.40 mm was mounted in a glass capillary. Measurements were made on a Rigaku AFC-5R diffractometer with graphite-monochromated Mo  $K\alpha$  radiation (40 kV, 200 mA) at the X-ray Laboratory of Okayama University. Cell constants were obtained from a least-squares refinement

using the setting angles of 25 reflections in the range  $21 < 2\theta < 22^\circ$ . Based on the systematic absences of  $h0l$ ,  $h = 2n + 1$  and  $0k0$ ,  $k = 2n + 1$ , the space group was determined to be  $P2_1/a$ . The intensity data were collected using the  $\omega$ - $2\theta$  scan technique to a maximum  $2\theta$  of  $55.0^\circ$  ( $-16 \leq h \leq 16$ ,  $0 \leq k \leq 20$ ,  $-1 \leq l \leq 12$ ). Of the 5513 reflections collected, 4705 were unique ( $R_{int} = 0.045$ ). The intensities of three representative reflections (403, 403 and 044) which were measured after every 97 reflections remained constant throughout data collection. An absorption correction based on azimuthal scans of three reflections was applied, the transmission factors ranging from 0.77 to 1.00. The data were corrected for Lorentz and polarization effects.

The structure was solved by Patterson method using *PHASE* (Calbrese, 1972) and *DIRDIF* (Beurskens, 1984). The non-H atoms were refined anisotropically. No H atoms were located in the difference Fourier map and their parameters were not determined. The final cycle of the full-matrix least-squares refinement, minimizing  $\sum w(|F_o| - |F_c|)^2$  [ $w = 4F_o^2/\sigma^2(F_o^2)$ ], was based on 2630 observed reflections [ $I > 3\sigma(I)$ ] and 154 variable parameters and converged to  $R = 0.051$ ,  $wR = 0.052$ ,  $(\Delta/\sigma)_{max} = 0.82$  and  $S = 1.92$ . The maximum and minimum  $\Delta\rho$  in the final difference Fourier map were 0.65 and  $-0.72 \text{ e } \text{\AA}^{-3}$ . Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed on a VAX 3100 computer using *TEXSAN* (Molecular Structure Corporation, 1989).

The atomic parameters are given in Table 1.\* Bond distances and angles are given in Table 2. The crystal structure is shown in Fig. 1.

**Related literature.** The synthesis of  $[(\text{CH}_3)_3\text{CNH}_3]_2\text{-TeCl}_6$  has been reported (Ishida, Inada, Hayama, Nakamura & Ikeda, 1991). To our knowledge, the compound represents the first structural analysis of a *tert*-butylammonium hexahalometallate(IV) complex. The crystal data of the compounds containing *tert*-butylammonium ions are also very few (Trueblood, 1987). Of the  $[(\text{CH}_3)_n\text{NH}_{4-n}]_2\text{MCl}_6$  compounds ( $n = 1-4$ ;  $M = \text{Sn, Te, Pt}$ ) which are known to have an antiferrotype structure at room temperature (Knop, Cameron & James, 1983; Wyckoff, 1928; Ben Ghazlen, Daoud & Bats, 1981; Ishida, Ikeda & Nakamura, 1984; Wyckoff & Corey, 1929; Ikeda, Kadel, Weiss, Ishida & Nakamura, 1982; Berg & S\o\tofte, 1978; Mackenzie, Berg & Pawley, 1980),

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54989 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0574]

Table 1. Positional parameters and equivalent isotropic thermal parameters ( $\text{\AA}^2$ ) with e.s.d.'s in parentheses

$$B_{eq} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	$B_{eq}$
Te	0.22715 (5)	0.12633 (4)	0.15023 (5)	5.10 (3)
Cl(1)	0.0696 (2)	0.1188 (2)	-0.0228 (2)	6.7 (1)
Cl(2)	0.2941 (2)	0.2640 (1)	0.0301 (2)	6.7 (1)
Cl(3)	0.3503 (2)	0.0298 (2)	-0.0057 (2)	7.0 (1)
Cl(4)	0.1565 (3)	-0.0056 (2)	0.2469 (2)	8.8 (2)
Cl(5)	0.3644 (3)	0.1361 (2)	0.3207 (3)	11.1 (2)
Cl(6)	0.1116 (3)	0.2109 (2)	0.2894 (3)	12.3 (2)
N(1)	0.3639 (6)	0.4368 (4)	0.1828 (6)	5.8 (4)
N(2)	0.5431 (5)	0.1693 (4)	-0.1023 (6)	5.9 (4)
C(1)	0.3284 (8)	0.4512 (5)	0.3292 (8)	5.3 (5)
C(2)	0.388 (1)	0.3887 (7)	0.417 (1)	8.7 (7)
C(3)	0.350 (1)	0.5427 (6)	0.364 (1)	8.9 (7)
C(4)	0.2066 (8)	0.4318 (8)	0.332 (1)	9.0 (7)
C(5)	0.5393 (7)	0.1749 (6)	-0.2576 (8)	5.4 (5)
C(6)	0.528 (1)	0.0842 (6)	-0.314 (1)	8.5 (7)
C(7)	0.6452 (8)	0.2145 (6)	-0.303 (1)	7.9 (6)
C(8)	0.4412 (9)	0.2292 (8)	-0.292 (1)	9.9 (7)

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

Te-Cl(1)	2.629 (3)	N(1)-C(1)	1.542 (9)
Te-Cl(2)	2.614 (2)	N(2)-C(5)	1.548 (9)
Te-Cl(3)	2.671 (2)	C(1)-C(2)	1.51 (1)
Te-Cl(4)	2.454 (3)	C(1)-C(3)	1.51 (1)
Te-Cl(5)	2.425 (3)	C(1)-C(4)	1.57 (1)
Te-Cl(6)	2.413 (3)	C(5)-C(6)	1.54 (1)
		C(5)-C(7)	1.54 (1)
		C(5)-C(8)	1.54 (1)
Cl(1)-Te-Cl(2)	89.02 (8)	N(1)-C(1)-C(2)	107.9 (7)
Cl(1)-Te-Cl(3)	91.96 (8)	N(1)-C(1)-C(3)	108.0 (7)
Cl(1)-Te-Cl(4)	86.74 (9)	N(1)-C(1)-C(4)	106.1 (7)
Cl(1)-Te-Cl(5)	176.3 (1)	C(2)-C(1)-C(3)	113.6 (9)
Cl(1)-Te-Cl(6)	86.8 (1)	C(2)-C(1)-C(4)	110.2 (8)
Cl(2)-Te-Cl(3)	90.91 (8)	C(3)-C(1)-C(4)	110.8 (9)
Cl(2)-Te-Cl(4)	175.50 (9)	N(2)-C(5)-C(6)	108.4 (7)
Cl(2)-Te-Cl(5)	92.1 (1)	N(2)-C(5)-C(7)	106.8 (7)
Cl(2)-Te-Cl(6)	90.1 (1)	N(2)-C(5)-C(8)	106.0 (7)
Cl(3)-Te-Cl(4)	87.71 (9)	C(6)-C(5)-C(7)	110.2 (8)
Cl(3)-Te-Cl(5)	91.5 (1)	C(6)-C(5)-C(8)	111.1 (9)
Cl(3)-Te-Cl(6)	178.3 (1)	C(7)-C(5)-C(8)	114.0 (9)
Cl(4)-Te-Cl(5)	92.3 (1)		
Cl(4)-Te-Cl(6)	91.2 (1)		
Cl(5)-Te-Cl(6)	89.8 (1)		

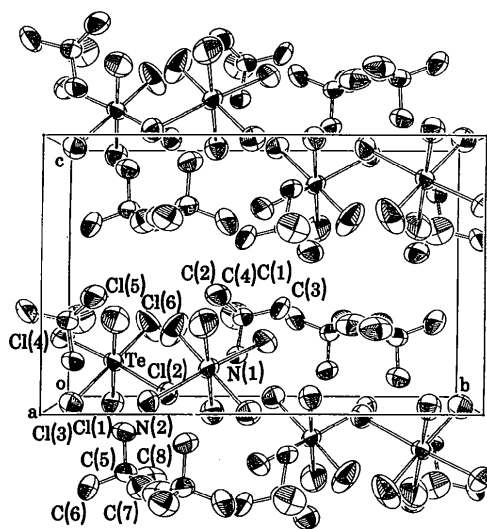


Fig. 1. An ORTEP (Johnson, 1976) perspective view along the *a* axis and atomic numbering. Thermal ellipsoids are drawn at the 50% probability level.

$(\text{CH}_3\text{NH}_3)_2\text{TeCl}_6$  exceptionally forms an *anti*- $\text{CdI}_2$ -type arrangement (Kitahama & Kiriyama, 1978).

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### 1,3,7,9-Tetra-*tert*-butyl-2,2,8,8-tetramethyl-5,10-diphenyl-1,3,5,7,9,10-hexaaza-2,8-disila-4,6-digermadispiro[3.1.3.1]decane

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**Abstract.**  $\text{C}_{32}\text{H}_{58}\text{Ge}_2\text{N}_6\text{Si}_2$ ,  $M_r = 728.20$ , triclinic,  $P\bar{1}$ ,  $a = 9.182$  (11),  $b = 9.774$  (9),  $c = 11.784$  (10) Å,  $\alpha = 107.27$  (7),  $\beta = 100.16$  (8),  $\gamma = 98.05$  (9)°,  $V = 973$  (2) Å<sup>3</sup>,  $Z = 1$ ,  $D_x = 1.243$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 1.61$  mm<sup>-1</sup>,  $F(000) = 384$ ,  $T = 291$  (1) K, final  $R = 0.0443$  for 2186 unique observed [ $F \geq 4.0\sigma(F)$ ] diffractometer data. The crystal consists of discrete molecular units. The central part of the centrosymmetric molecule is a planar four-membered Ge—N—Ge—N— ring [Ge—N 1.851 (4), 1.852 (4); Ge...Ge 2.784 (1); N...N 2.441 (4) Å; Ge—N—Ge 97.5 (1); N—Ge—N 82.5 (1)°]. A phenyl group is bound to each N atom of this ring and each Ge atom of this central ring is also a member of an outer four-membered Ge—N—Si—N— ring [Ge—N 1.829 (4), 1.824 (4); Si—N 1.713 (4), 1.735 (4); Ge...Si 2.608 (4); N...N 2.441 (4) Å; N—Ge—N 82.5 (2); Ge—N—Si 94.8 (3), 94.2 (3); N—Si—N 88.6 (3)°]. The dihedral angle between the plane of the central ring and the least-squares plane through the outer ring is 90.6 (2)°. A distorted tetrahedron [angles between 82.5 (1) and 125.1 (1)°] around Ge is formed by the atoms bound to Ge. The phenyl rings bound to the N atoms of the Ge—N—Ge—N— ring and this central four-membered ring are nearly [4.0 (2)°] coplanar. The coordination around Si is a distorted

tetrahedron with angles between 88.6 (3) and 115.5 (3)°.

**Experimental.** The title compound has been obtained as the major product from the known germylene (Veith & Grosser, 1982) and phenyl azide at 298 K. Colourless crystals were obtained from a saturated THF solution by slow cooling to 253 K. A crystal platelet of approximate size 0.86 × 0.28 × 0.10 mm was used. Its quality was checked with optical polarizing microscopy. The crystal was mounted on the tip of a glass fibre with shellac.  $D_m$  was not determined. Intensity data were collected with  $\omega/2\theta$  scans, variable scan speed 4.5–14.6° min<sup>-1</sup> in  $\theta$ , scan width 1.2° + dispersion. A Nicolet *R3m/V* diffractometer with graphite-monochromated Mo  $K\alpha$  radiation was used for preliminary examinations and data collection. The lattice parameters were determined from a least-squares fit of the angular settings for ten reflections with  $2\theta_{\text{max}} = 24.8^\circ$ .  $\omega$  scans of low-order reflections along the three crystal axes showed acceptable mosaicity. Six standard reflections ( $4\bar{4}2$ ,  $050$ ,  $0\bar{2}7$ ,  $4\bar{4}2$ ,  $050$ ,  $02\bar{7}$ ) were recorded every 300 reflections; only random deviations were detected during 38.4 h of X-ray exposure. 4902 reflections with  $2.0 \leq 2\theta \leq 45.0^\circ$ ,  $-10 \leq h \leq 6$ ,  $-11 \leq k \leq 11$ ,  $-13 \leq l \leq 13$  were measured. The