

Bis(trimethylammonium) Hexachlorotellurate(IV)

BY M. H. BEN GHOZLEN* AND J. W. BATS

Institut für Kristallographie der Universität, Senckenberganlage 30, Postfach 11 19 32,
6000 Frankfurt/Main 11, Federal Republic of Germany

(Received 8 September 1981; accepted 23 November 1981)

Abstract. $[\text{NH}(\text{CH}_3)_3]_2[\text{TeCl}_6]$, cubic, $Pa\bar{3}$, $Z = 4$, $a = 12.269$ (2) Å, $V = 1846.8$ (9) Å³, $D_c = 1.656$, $D_m = 1.653$ (5) Mg m⁻³, $\mu(\text{Mo } K\alpha) = 2.57$ mm⁻¹, 570 non-equivalent diffractometer data up to $\sin \theta/\lambda = 0.65$ Å⁻¹, final $R(F) = 0.050$, $R_w(F) = 0.023$. The structure contains $[\text{TeCl}_6]^{2-}$ octahedra, with a Te–Cl distance, corrected for libration, of 2.546 (1) Å. Each octahedron is bonded to two $[\text{NH}(\text{CH}_3)_3]^+$ groups *via* weak trifurcated hydrogen bonds.

Introduction. The title compound was obtained by mixing stoichiometric amounts of concentrated solutions of tellurium dioxide and trimethylamine in hydrochloric acid. Yellow crystals precipitated on cooling. They were purified by recrystallization from a hot dilute acid solution. The compound is unstable in pure water but is soluble in dilute acid without decomposition.

A cube, 0.25 × 0.32 × 0.36 mm, was selected for the experiments. Precession photographs showed a cubic lattice with space group $Pa\bar{3}$. Data were collected in a quadrant of reciprocal space up to $\sin \theta/\lambda = 0.65$ Å⁻¹ on a Syntex $P2_1$ diffractometer with Nb-filtered Mo $K\alpha$ radiation. A total of 4288 reflections were obtained of which 660 were independent. Background corrections were made by profile analysis (Blessing, Coppens & Becker, 1974). Three standard reflections remeasured after every 60 reflections showed long-term fluctuations up to about 5%. The data were rescaled with respect to the standards. An absorption correction gave transmission factors ranging from 0.46 to 0.56. A weight was assigned to each reflection according to $w(I) = \{\sigma^2(I)_{\text{counting}} + (0.03I)^2\}^{-1}$. The equivalent reflections were averaged [$R(I) = \sum |I - \langle I \rangle| / \sum I = 0.025$].

570 of the resulting reflections had $I > 0$ and were used in the analysis. The structure was determined from a Patterson synthesis and refined by least-squares methods. A difference synthesis revealed the positions of the H atoms. They were included in the refinement, but their isotropic thermal parameters were not varied.

* Permanent address: Laboratoire de Chimie Minérale, Faculté des Sciences et Techniques de Sfax, Sfax, Tunisia.

Table 1. Positional parameters and equivalent values of the isotropic thermal parameters

	$U_{\text{eq}} = \frac{1}{3} \text{trace } U.$			
	x	y	z	U_{eq} (Å ²)
Te	0.0	0.0	0.0	0.0453 (2)
Cl	0.03456 (7)	-0.02627 (7)	0.20209 (6)	0.0624 (5)
N	0.2056 (2)	0.2056 (2)	0.2056 (2)	0.062 (1)
C	0.1520 (4)	0.2111 (4)	0.3139 (4)	0.096 (4)
H(1)	0.166 (2)	0.166 (2)	0.166 (2)	0.080*
H(2)	0.124 (3)	0.344 (3)	0.150 (3)	0.095*
H(3)	0.237 (3)	0.293 (3)	0.089 (3)	0.095*
H(4)	0.267 (3)	0.350 (3)	0.205 (2)	0.095*

* Not refined.

Table 2. Bond lengths (Å) and angles (°)

6 × Te–Cl	2.536 (1)	C–H(3)	0.87 (4)
3 × N–C	1.484 (5)	C–H(4)	1.05 (3)
N–H(1)	0.84 (2)	3 × N...Cl	3.536 (2)
C–H(2)	1.13 (3)	3 × H(1)...Cl	2.89 (2)
6 × Cl–Te–Cl	88.97 (3)	N–C–H(2)	105 (2)
6 × Cl–Te–Cl	91.03 (3)	N–C–H(3)	98 (3)
3 × Cl–Te–Cl	180.00	N–C–H(4)	98 (2)
3 × H(1)–N–C	107 (1)	3 × N–H(1)...Cl	135 (1)
3 × C–N–C	112.1 (3)		

Scattering factors were from *International Tables for X-ray Crystallography* (1974), except for H (Stewart, Davidson & Simpson, 1965). Anomalous-dispersion factors were applied to Te and Cl (Cromer & Liberman, 1970). Secondary extinction was found to be negligible. A final difference synthesis was featureless. The structure refined to $R(F) = 0.050$, $R_w(F) = 0.023$ and $S = \{\sum w(F_o - F_c)^2 / (NO - NV)\}^{1/2} = 1.79$. The $R(F)$ value is larger than $R_w(F)$ as a considerable number of reflections at larger diffraction angles were rather weak. The positional parameters of the atoms are reported in Table 1, † bond lengths and angles in Table 2.

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36571 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. The structure consists of $[\text{TeCl}_6]^{2-}$ octahedra and $[\text{NH}(\text{CH}_3)_3]^+$ groups. The six equivalent Te—Cl bonds have a length of 2.536 (1) Å without a libration correction and 2.546 (1) Å after a correction for librational motion has been made (Schomaker & Trueblood, 1968). They agree very well with the values of 2.541 (7) and 2.541 (5) Å respectively in $(\text{NH}_4)_2\text{TeCl}_6$ (Hazell, 1966) and Rb_2TeCl_6 (Webster & Collins, 1973). Each $[\text{TeCl}_6]^{2-}$ octahedron is connected by hydrogen bonding to two $[\text{NH}(\text{CH}_3)_3]^+$ groups. A stereoscopic view of an $[\text{NH}(\text{CH}_3)_3]_2[\text{TeCl}_6]$ unit is shown in Fig. 1.

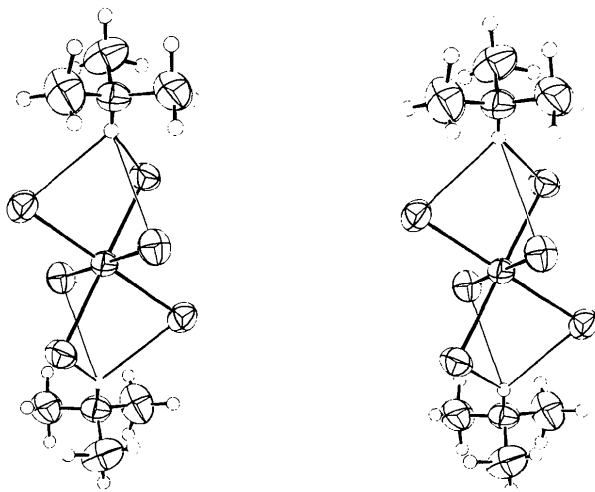


Fig. 1. A stereoscopic view of the structure. The thermal ellipsoids are the 50% probability surfaces; the H atoms are on an arbitrary scale.

The threefold symmetry of the structure would lead to a trifurcated hydrogen bond between N—H(1) and three related Cl atoms (Table 2). Such a feature is rather unusual in crystal structures. Moreover, the observed $\text{H}(1)\cdots\text{Cl}$ distance of 2.89 (2) Å corresponds to the sum of van der Waals radii of H and Cl, indicating that there is no significant hydrogen-bond interaction. However, the observed N—H(1) distance of 0.84 (2) Å is obviously too short. If the N—H(1) distance were assumed to be 1.03 Å, as commonly found in ammonium groups, the $\text{H}(1)\cdots\text{Cl}$ distance would be 2.76 Å. Consequently the hydrogen bond would be very weak.

One of us (MHBG) gratefully acknowledges financial support from the Deutscher Akademischer Austauschdienst.

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Acta Cryst. (1982). **B38**, 1309–1312

Structure of the Potassium Thiocyanate Complex of 1,4,7,10,13,16-Hexaoxacyclooctadecane-2,6-dione (2,6-Diketo-18-crown-6)

BY S. B. LARSON

Department of Chemistry, University of Texas at Austin, Austin, TX 78712, USA

AND N. K. DALLEY

Department of Chemistry (and Contribution No. 256 from the Thermochemical Institute), Brigham Young University, Provo, UT 84602, USA

(Received 15 June 1981; accepted 25 November 1981)

Abstract. $\text{C}_{12}\text{H}_{20}\text{O}_8 \cdot \text{KSCN}$, monoclinic, $P2_1/c$, $M_r = 389.45$, $a = 12.517$ (5), $b = 10.943$ (3), $c = 15.145$ (2) Å, $\beta = 119.975$ (24)°, $V = 1796.7$ (11) Å³, $Z = 4$, $D_x = 1.44$ Mg m⁻³, colorless, m.p. 442–442.5 K. The cation is situated in the cavity of the polyether ring, coordinated to the six O atoms in the ring and to two anions. Resolution of the disorder in the anion and a

portion of the polyether gave $R = 0.046$ and $R_w = 0.016$ for 2675 reflections.

Introduction. The complex was synthesized by G. Maas (Maas, Bradshaw, Izatt & Christensen, 1977). Approximate cell parameters and the space group were determined from precession photographs. 2θ angles of