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ORGANOTELLURIUM(IV) COMPLEXES: SYNTHESIS AND MOLECULAR STRUCTURE OF 2,6-DIACETYLPYRIDINE(C,N,O) TELLURIUM(IV) TRICHLORIDE

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Summary

The reaction of $TeCl_4$ with 2,6-diacetylpyridine in methylene chloride or tetrahydrofuran gives a new type of organotellurium (IV) compound. An X-ray structure determination showed that the organic radical bonds to the tellurium as a tridentate ligand via a methylene carbon of one of the acetyl groups, the pyridine nitrogen, and the carbonyl oxygen of the second acetyl group. Analogous organotellurium trichloride complexes involving C,O coordination have been formulated for the condensation products of $TeCl_4$ with 2-acetylcyclohexanone and 3-acetyl-7-methoxycoumarin, while C,N coordination occurs in the condensation product of $TeCl_4$ and 2-acetylpyridine.

Introduction

The organometallic chemistry of tellurium [1-4] has been the subject of increasing interest in recent years, a variety of derivatives of the type TeR_nX_{4-n} (R = alkyl, aryl; X = halide, pseudohalide, carboxylate; n = 1-4) having been described. A number of derivatives of this type have been obtained from condensation reactions of $TeCl_4$ with various aliphatic and aromatic ketones [e.g., $TeCl_2(CH_2COC_6H_5)_2$] [5]. Such condensation reactions with acetylacetone and its substituted derivatives have recently been shown by X-ray crystallographic studies to result in the formation of products in which the terminal carbon atoms bond to tellurium to give six-membered heterocyclic products [6-8]

In this paper we describe a new type of organotellurium compound resulting from the condensation of $TeCl_4$ with ketones having basic sites available for coordination to the tellurium. A single-crystal X-ray diffraction study of the product obtained from the condensation of $TeCl_4$ with 2,6-diacetylpyridine, $TeCl_3(-2-CH_2CO(6-CH_3CO-C_5H_3N)$, in which the organic radical functions as a tridentate (C,N,O) ligand, is also reported.

Results and discussion

In contrast to the reactions of $TeCl_4$ with simple ketones such as acetophenone [5] and its ring-substituted derivatives [6] and various diketones [7-10] which readily eliminate 2 mol of HCl with the formation of $TeCl_2R_2$-type derivatives, the condensation reactions with polyfunctional ketones (e.g., 2,6-diacetylpyridine, 2-acetylcyclohexanone, 3-acetyl-7-methoxycoumarin) proceed with the elimination of only 1 mol of HCl and the formation of a new type of organotellurium complex, $TeCl_3R$, in which the organic radical functions as a bi- or tridentate ligand. Surprisingly, even when the condensation reactions are carried out under forcing conditions with a twofold molar excess of ketone, the 1:1 products (i.e., organotellurium trichlorides) are isolated. The stability of these complexes is in marked contrast to the facile aerial decomposition of previously reported alkyl tellurium trichlorides.

Methyl tellurium trichloride [11] and the condensation products of $TeCl_4$ with a number of ketones [12] are very susceptible to aerial hydrolysis. Attempts to isolate such derivatives from the reactions of $TeCl_4$ with acetophenone (i.e.,

$Cl_3TeCH_2COC_6H_5$) and benzylmercuric chloride (i.e., $Cl_3TeCH_2C_6H_5$) gave products which readily decomposed in air at room temperature. However, the reaction of tellurium tetrachloride with 2,6-diacetylpyridine in a 1:1 molar ratio in refluxing methylene chloride gave, upon concentration of the reaction solution, a tan powder from which pale yellow needles were obtained by recrystallization from methylene chloride/ether.

The compound, which decomposes with darkening above 165°C and gives a murky black melt at 205°C, is very soluble in acetone and DMF, moderately soluble in CH_2Cl_2, and only slightly soluble in $CHCl_3$ and ethyl acetate. The isolation of such well-formed crystals was itself unusual since all of the other organotellurium derivatives resulting from condensation reactions between $TeCl_4$ and simple acetophenone derivatives [e.g., $CH_3COC_6H_4X$ (X = H, p-CH_3, o-CH_3, p-CH_3O, o-CH_3O); $CH_3COC_6F_5$] [13] were obtained as white powders even after repeated recrystallizations from various solvents.

The infrared spectrum of the product also supported an unusual bonding mode of the potentially ambidentate 2,6-diacetylpyridine radical. The pyridine ring breathing band is shifted from 997 cm^{-1} in 2,6-diacetylpyridine to 1013 cm^{-1} in the tellurium compound. Such a shift to higher energy in metal compounds of pyridine derivatives generally indicates coordination to the pyridyl nitrogen [14]. Other infrared absorptions also support coordination of the ring nitrogen to tellurium [14]: (a) the two ring vibrations at 1410 and 1578 cm^{-1} are shifted to higher energy in the tellurium compound (e.g., 1418 and 1592 cm^{-1}) and (b) the weak band at 1568 cm^{-1} in free 2,6-diacetylpyridine is reinforced in the tellurium compound.

Two carbonyl stretching bands were observed (1696 and 1712 cm^{-1}), free 2,6-diacetylpyridine exhibiting one band at 1707 cm^{-1}. The carbonyl stretching frequencies of several acetophenone condensation products $[Cl_2Te(CH_2COAr)_2]$ were generally unchanged from those of the free ketone [13]. The shift to lower frequency of one of the carbonyl bands in this product is consistent with some interaction of the carbonyl oxygen with the tellurium. The relatively small decrease (11 cm^{-1}), however, suggests that the interaction is weak. Strong carbonyl interactions with metals have been shown to result in shifts of up to 215 cm^{-1} in the carbonyl stretching frequency [15] [e.g., $NbCl_5$·acetophenone, $\nu_{C=O}$ = 1542 cm^{-1} (a decrease of 143 cm^{-1} from acetophenone)].

The 90 MHz ^1H NMR spectrum in acetone-d^6 (vs. TMS) is consistent with the proposed six-coordinate organotellurium compound involving a tridentate C,N,O bonding mode for the 2,6-diacetylpyridine radical [δ_{CH_3} = 2.85 ppm (1); δ_{CH_2} = 2.94 ppm (0.6); $\delta_{arom.}$ = 8.32 ppm (1)].

A single-crystal X-ray structure determination of the title compound confirmed the structural features as shown in the stereographic view [16] in Fig. 1. The coordination geometry can be described as a distorted pentagonal bipyramid with two axial Te-Cl bonds and the equatorial positions occupied by a Cl, the three atoms of the tridentate organic radical, and a stereochemically active lone pair of electrons. Such a geometry

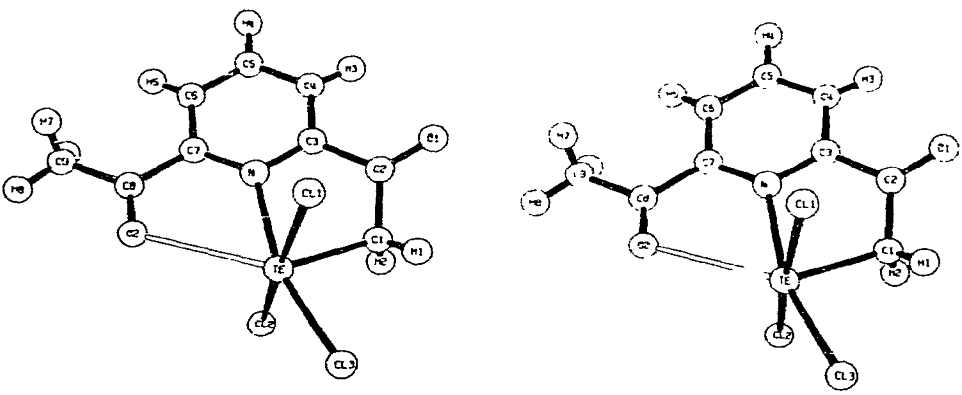


Fig. 1. Stereographic view of $TeCl_3(C_9H_8NO_2)$. Atom H6 is partly hidden behind atom C9.

is consistent with valence shell electron pair repulsion theory [17] and the empirical rules of Wynne [18] for lone-pair stereochemical activity. An alternative bonding scheme involving three 3-center bonds with an inert electron pair has been suggested by Zingaro et al. [19] for six-coordinated Te structures where distortions from octahedral geometry are attributed to steric effects. The angles involved in the Te coordination geometry are given in Table 1. We believe the distortions from ideal values, particularly N-Te-Cl(3) (163.37°) and O(2)-Te-Cl(3) (135.56°) are better explained from the view of a lone pair of electrons occupying an equatorial position between O(2) and Cl(3). That the C(1)-Te-Cl(3) angle (88.4°) is not smaller is probably due to the Cl(3)....H(1) contact (d = 2.88 Å), about the sum of the van der Waals radii [20].

Table 1. Angles of the Te coordination geometry.[a]

C(1)-Te-N	75.1(1)°	Cl(2)-Te-C(1)	88.8(1)
C(1)-Te-Cl(3)	88.4(1)	Cl(2)-Te-N	88.61(7)
N-Te-O(2)	61.02(8)	Cl(2)-Te-O(2)	81.37(7)
O(2)-Te-Cl(3)	135.56(6)	Cl(2)-Te-Cl(3)	92.76(5)
Cl(1)-Te-C(1)	84.9(1)	Cl(1)-Te-Cl(2)	171.28(4)
Cl(1)-Te-N	85.00(7)	C(1)-Te-O(2)	134.8(1)
Cl(1)-Te-O(2)	100.59(6)	N-Te-Cl(3)	163.37(5)
Cl(1)-Te-Cl(3)	91.69(4)		

[a] Estimated standard deviations are given in parentheses in all tables.

The intramolecular bond lengths are given in Table 2. The Te-C(1) bond length (2.129 Å) is in good agreement with the sum of covalent radii (2.14 Å) [20] and is comparable to values commonly found for Te(IV)-C_{sp^2} bonds [19]. The Te-N bond length (2.402 Å) is considerably longer than the sum of covalent radii (2.07 Å). The Te-O(2) bond (2.878 Å) is much longer than the sum

Table 2. Bond lengths.

Te-C(1)	2.129(3) Å	C(3)-C(4)	1.386(4)
Te-N	2.402(3)	C(3)-N	1.335(3)
Te-O(2)	2.878(3)	C(4)-C(5)	1.378(4)
Te-Cl(1)	2.500(1)	C(5)-C(6)	1.381(5)
Te-Cl(2)	2.491(1)	C(6)-C(7)	1.388(4)
Te-Cl(3)	2.438(2)	C(7)-C(8)	1.497(4)
C(1)-C(2)	1.498(5)	C(7)-N	1.339(3)
C(2)-C(3)	1.495(4)	C(8)-C(9)	1.487(4)
C(2)-O(1)	1.207(4)	C(8)-O(2)	1.213(4)

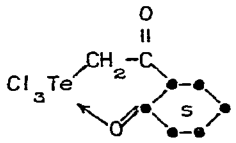
of covalent radii (2.03 Å) but much less than the sum of the van der Waals radii (3.60 Å), indicating a weak bond. Long Te-O (carbonyl) bonds have been reported in *o*-formylphenyltellurenyl(II) bromide [21] (2.31 Å) and in 9-telluro-1-formyl-3,4,5,6,7,8-hexahydro-2H-anthracene [22] (2.574, 2.577 Å). Interestingly, the IR carbonyl stretching frequency is 110 cm^{-1} lower in the former compound [23] and 92.5 cm^{-1} lower in the latter compound [24] compared to the corresponding aldehydes, but we find a decrease of 11 cm^{-1} due to the weaker Te-O interaction in this complex.

There are two factors possibly contributing to the long Te-O and Te-N bonds. The steric requirements of the 5-membered rings prevent a close approach of all three coordinating atoms in the tridentate ligand. It is significant that the Te-O bond is much stronger in the Te-coumarin derivative complex where a 6-membered ring is formed. The strong Te-C bond has been shown to have a *trans* effect in Te(II) compounds [25] in which the bond *trans* to the strong bond is weakened. This effect has been used to explain bond lengthening in several Te(IV) compounds [26].

The Te-Cl bonds (2.438 Å, equatorial; 2.491 and 2.500 Å, axial) are all longer than the sum of covalent radii (2.36 Å) but agree well with values commonly found for organotellurium halides [19]. Significantly, the equatorial Te-Cl(3) bond *trans* to the long Te-N bond is shorter than the axial bonds. The bond lengths in the diacetylpyridine ligand all have normal values and indicate that coordination to Te has not perturbed the bonding within the ligand.

The reaction of $TeCl_4$ with 2-acetylpyridine gave a drastically different product. Here a brick-red product precipitated from the reaction solution wich was insoluble in all common organic solvents except DMF. The infrared spectrum, however, exhibited a shift of the pyridine ring breathing band characteristic of coordination of the pyridine nitrogen to a metal (i.e., 1011 vs. 991 cm^{-1} in free 2-acetylpyridine) and no shift of the carbonyl band. The solubility properties and infrared spectrum of this compound suggest a polymeric formulation with an intermolecular coordination of the pyridine nitrogen rather than intramolecular as in the 2,6-diacetylpyridine analog. It has not been possible to obtain suitable crystals of this material for a single-crystal X-ray diffraction study.

Reaction of $TeCl_4$ with 2 equivalents of 2-acetylcyclohexanone in refluxing chloroform for 0.5 h gave a product (30% yield) as formulated below:

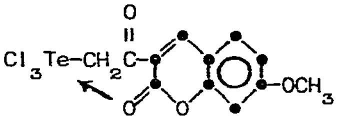


$\nu_{CO} = 1570, 1540\ cm^{-1}$

(ν_{CO} of free 2-acetylcyclohexanone = 1605 cm^{-1}, br)

The condensation reaction of $TeCl_4$ with 3-acetyl-7-

methoxycoumarin in methylene chloride gave the green trichloride:



ν_{CO} = 1669, 1635 cm^{-1}

(ν_{CO} of free coumarin = 1672, 1735 cm^{-1})

M.W. = 452 (field desorption mass spectroscopy; ^{130}Te, ^{35}Cl)

By analogy with the other ketone derivatives, the band at 1669 cm^{-1} is assigned to the carbonyl adjacent to the $Te-CH_2$ and the band at 1635 cm^{-1} is assigned to the other carbonyl stretching frequency, the decrease of ca. 100 cm^{-1} vs. the corresponding value for the free coumarin indicating a rather strong Te....O interaction. The field desorption spectrum of the compound, in addition to the parent ion, showed a M-Cl peak.

Reaction of $TeCl_4$ with o-lithiodimethylbenzylamine in THF at -78°C resulted in decomposition to elemental tellurium on attempted workup. A number of transition-metal organometallic chelates of this radical have been reported [27], these compounds in general exhibiting considerably enhanced thermal stability compared to the simple metal aryl analogs.

The condensation reaction with 2-acetylthiophene gave the $TeCl_2R_2$ product with no evidence for interaction of the ring sulfur with tellurium.

Experimental

2-Acetylpyridine, 2,6-diacetylpyridine, and 2-acetylcyclohexanone were obtained from Aldrich Chemical Co. $TeCl_4$ was obtained from Alfa Products. 3-Acetyl-7-methoxycoumarin was prepared as described in the literature [28]. All other chemicals were obtained from Eastman Organic Chemicals.

$CHCl_3$ and CH_2Cl_2 were purified by washing three times with water (20% by volume), drying over anhydrous $MgSO_4$, and then distilling under Ar from P_4O_{10}.

THF was initially dried over NaOH and then distilled from Na/benzophenone.

Bis(2-acetylthiophene)tellurium dichloride

2-Acetylthiophene (25.2 g, 0.2 mol) was added to a solution of $TeCl_4$ (26.94 g, 0.1 mol) in 250 ml of chloroform. The reaction solution was heated at reflux for 0.5 h, cooled to room temperature, and filtered. The filtrate was concentrated under vacuum to ca. 100 ml and diluted with hexane to give a dark oil which solidified on cooling at -10°C. The resulting brown solid was recrystallized from chloroform/ether (1:1) to yield 3.1 g (7%) of a white solid, m.p. 180-182°C dec. Anal.: Found: C, 31.9; H, 2.4; Cl, 16.1; Te, 27.8. $C_{12}H_{10}Cl_2O_2S_2Te$ (448.8) calcd.: C, 32.11; H, 2.25; Cl, 15.80; Te, 28.43%.

2-Acetylcyclohexanone tellurium trichloride

2-Acetylcyclohexanone (14.02 g, 0.1 mol) was added to a suspension of tellurium tetrachloride (13.45 g, 0.05 mol) in 150 ml of chloroform. The solution was heated at reflux for 30 min, cooled to room temperature, and filtered. The volume of the filtrate was reduced to 100 ml, and 150 ml of ether was added. Cooling overnight yielded a cream-colored solid, which was filtered and dried. Concentration of the mother liquor and cooling yielded another 1.1 g of product. Total yield 5.6 g (30%), m.p. darkens over 105°C, black melt 129-131°C. Anal.: Found: C, 25.7; H, 3.0; Cl, 29.0; Te, 34.2. $C_8H_{11}Cl_3O_2Te$ (373.11) calcd.: C, 25.75; H, 2.97; Cl, 28.50; Te, 34.20%.

2,6-Diacetylpyridine tellurium trichloride

To a solution of $TeCl_4$ (25 g, 92.8 mmol) in 400 ml of $CHCl_3$ under an Ar atmosphere was added 15.2 g (93 mmol) of 2,6-diacetylpyridine. The reaction solution, which did not evolve HCl at room temperature, was refluxed 24 h under the inert atmosphere, then cooled to room temperature and diluted with 1ℓ of ethyl ether to give a brown precipitate. This crude product was filtered, washed well with ether, and air dried [21.7 g; 59% based on $Cl_3Te(C_9H_8NO_2)$]. Recrystallization of this crude product from 1300 ml of hot CH_2Cl_2 (and activated carbon) gave 12.6 g of bright yellow crystals (overall yield, 33.5%). Anal.: Found: C, 27.2; H, 2.2; N, 3.7; Cl, 26.6; Te, 32.5. $C_9H_8Cl_3NO_2Te$ (396.13) calcd.: C, 27.29; H, 2.03; N, 3.54; Cl, 26.84; Te, 32.21%.

3-Acetyl-7-methoxycoumarin tellurium trichloride

To a solution of the coumarin (3.85 g, 18.1 mmol) in 200 ml of CH_2Cl_2 was added 5 g (18.6 mmol) of $TeCl_4$. The reaction solution was stirred for 0.5 h at room temperature and then refluxed for 1.25 h. After cooling to room temperature the reaction solution was diluted to 1ℓ with ether. The resulting green precipitate was filtered, air dried (5.2 g), and recrystallized from 700 ml of hot dichloroethane; 3.1 g, m.p. dec. above 165°C. Anal.: Found: C, 31.8; H, 2.0; Cl, 23.7; Te, 28.4. $C_{12}H_9Cl_3O_4Te$ (451.14) calcd.: C, 31.95; H, 2.01; Cl, 23.57; Te, 28.28%.

Physical measurements

Microanalyses were done by the Analytical Sciences Division of the Kodak Research Laboratories. Infrared spectra were recorded on a Nicolet series 7000 Fourier-transform infrared spectrometer. Proton NMR spectra were recorded on a Bruker model HX-90 spectrometer. Field desorption mass spectra were recorded on a Varian MAT 731 mass spectrometer.

Crystal structure determination of $TeCl_3(C_9H_8NO_2)$

Large, yellow, acicular (c) crystals were obtained by recrystallization from a hot methylene chloride/ether solution. A crystal 0.28 x 0.38 x 0.43 mm was cemented onto a glass rod and used for intensity-data collection. Precession photographs gave preliminary lattice parameters and systematic absences.

Least-squares refinement of the positions of 17 reflections at moderately high Bragg angles (Mo $K\alpha_1$, $\lambda = 0.70926$) accurately centered on a Picker four-circle diffractometer gave the lattice constants shown below.

Crystal data. $TeCl_3C_9H_8NO_2$, M = 396.13, Monoclinic, *a* 13.662(6), *b* 10.900(6), *c* 8.726(6) Å, β 102.62(1)°, V 1268(1) $Å^3$, Z = 4, F(000) = 752, $D_c = 2.075$, $D_{obs} = 2.11$ g cm^{-3}, μ(Mo $K\alpha$) 29.8 cm^{-1}, absent spectra: *h0ℓ* for *h* odd, *0k0* for *k* odd, space group $P2_1/a$.

Data collection and reduction. Intensity data were collected with Zr-filtered Mo radiation by the θ-2θ scan technique at 1° min^{-1} with 20 sec background counts at each end of the scan. The (545) reflection was remeasured after every 50th reflection and used to correct all intensities for a small but constant decrease in intensity (maximum of 1.8%) over the data-collection period. This loss is probably due to radiation damage, as the crystal moderately darkened on prolonged exposure. The intensities were corrected for background, Lorentz, and polarization factors but not absorption. Of the 3734 reflections with $2\theta \leqslant 60°$, 3528 had $I > \sigma(I)$ and were considered observed. Unobserved reflections were excluded from further calculations.

Structure determination and refinement. The structure was solved by the heavy-atom method and refined by full-matrix least squares using program ORFLS [29]. The function minimized was $\Sigma w(|Fo|-K|Fc|)^2$. The weighting scheme of Killean and Lawrence [30] was used with $w^{-1} = \sigma^2(Fo) + (0.022\ Fo)^2 + 1.2$, where $\sigma(Fo) = [(I + \sigma(I))/Lp]^{1/2} - Fo$. Atomic scattering factors for all atoms and the anomalous dispersion corrections for Te and Cl were obtained from the International Tables [31].

A sharpened Patterson map gave the Te position which was used to phase all reflections and calculate an electron density map. This map yielded the positions of all nonhydrogen atoms.

Table 3. Final atomic parameters for the nonhydrogen atoms. The anisotropic thermal parameters are in the form exp $[-0.25(h^2a^{*2}B_{11} + ...2k\ell b^*c^*B_{23})]$.

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Te	0.32362(1)	0.58939(1)	0.20799(2)	3.31(1)	2.27(1)	2.26(1)	-0.11(1)	0.63(0)	0.30(0)
Cl(1)	0.26723(8)	0.79155(8)	0.29307(10)	6.77(4)	3.38(3)	3.64(3)	1.12(3)	1.92(3)	0.14(2)
Cl(2)	0.40007(9)	0.40613(7)	0.10994(15)	6.30(5)	3.16(3)	6.25(5)	0.35(3)	2.59(4)	-0.50(3)
Cl(3)	0.35117(7)	0.50839(8)	0.47444(9)	5.45(4)	4.38(3)	2.91(3)	0.21(3)	0.77(2)	1.43(2)
O(1)	0.5316(2)	0.8647(2)	0.2219(3)	4.61(11)	3.41(10)	4.75(11)	-1.36(8)	-0.16(9)	0.06(8)
O(2)	0.1772(2)	0.5519(3)	-0.0799(3)	4.23(10)	4.78(10)	3.24(8)	-1.60(9)	0.51(7)	0.52(8)
N	0.3421(2)	0.6976(2)	-0.0238(2)	2.98(8)	2.14(7)	2.37(7)	0.16(6)	0.78(6)	0.11(6)
C(1)	0.4699(2)	0.6672(3)	0.2672(4)	3.69(12)	3.17(11)	3.67(12)	-0.54(10)	-0.32(10)	0.92(9)
C(2)	0.4787(2)	0.7788(3)	0.1709(4)	3.21(10)	2.63(10)	3.56(11)	-0.03(8)	0.54(8)	0.09(8)
C(3)	0.4183(2)	0.7770(2)	0.0058(3)	3.08(9)	2.19(8)	2.90(9)	0.08(7)	0.94(7)	0.19(7)
C(4)	0.4395(2)	0.8554(3)	-0.1078(4)	3.91(12)	2.67(10)	3.58(11)	-0.36(9)	1.32(9)	0.35(9)
C(5)	0.3802(3)	0.8488(3)	-0.2572(4)	4.99(15)	3.63(12)	3.35(11)	0.03(11)	1.79(11)	1.01(10)
C(6)	0.3020(3)	0.7657(3)	-0.2877(3)	4.21(12)	3.47(11)	2.57(10)	0.32(10)	1.18(9)	0.43(8)
C(7)	0.2830(2)	0.6919(2)	-0.1680(3)	3.10(9)	2.53(9)	2.34(8)	0.32(7)	0.82(7)	-0.03(7)
C(8)	0.1941(2)	0.6081(3)	-0.1914(3)	3.08(10)	3.24(10)	2.80(10)	-0.11(8)	0.49(8)	-0.17(8)
C(9)	0.1269(3)	0.5988(4)	-0.3499(4)	4.05(14)	5.61(19)	2.94(12)	-0.72(12)	-0.09(10)	-0.18(11)

Three cycles of refinement with isotropic thermal parameters and two cycles with anisotropic thermal parameters gave R_1 = 0.039. A difference electron density map gave the positions of all hydrogen atoms, which when included in the refinement reduced R_1 to 0.036. The largest structure factors were affected by extinction so the 432 largest F's were used with unit weighting to calculate the extinction parameter [32], $g = 1.87(7) \times 10^{-6}$. Three cycles of least squares with g held constant concluded refinement and gave R_1 = 0.031 and R_2 = 0.040. The final scale factor was 2.660(4). Only 14 of 177 parameter shifts were >0.1σ with a maximum shift of 0.25σ on the final cycle. The final atomic positional parameters are given in Tables 3 and 4.*

A final difference electron density map contained considerable detail around the heavy atoms with residual peaks of +1.0, +1.3, -1.3, and -1.2 $e/\mathring{A}^3$ around Te and peaks between ±0.4 $e/\mathring{A}^3$ around the chlorine atoms. Elsewhere, residual density was between +0.3 and -0.7 $e/\mathring{A}^3$.

Table 4. Atomic parameters and bond lengths (r) for the hydrogen atoms.

Atom	x	y	z	B(Å)^2	r(Å)
H(1)	0.482(3)	0.688(4)	0.380(5)	4.4(8)	0.99(4)
H(2)	0.512(3)	0.605(4)	0.250(5)	4.2(9)	0.92(4)
H(3)	0.497(3)	0.904(4)	-0.082(5)	4.4(9)	0.94(4)
H(4)	0.392(4)	0.914(4)	-0.340(6)	5.5(11)	1.05(5)
H(5)	0.266(2)	0.761(3)	-0.388(4)	2.6(6)	0.91(3)
H(6)	0.166(4)	0.580(4)	-0.432(6)	5.8(12)	1.01(6)
H(7)	0.096(4)	0.667(5)	-0.395(6)	6.3(11)	0.91(5)
H(8)	0.066(4)	0.539(5)	-0.355(5)	5.7(10)	1.05(5)

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*The table of structure factors can be obtained from the authors on request.

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