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Structure of Dichloro(*p*-methoxyphenyl)(2-oxocyclohexyl)tellurium(IV)

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Abstract. $[TeCl_2(CH_3OC_6H_4)(C_6H_9O)]$, $M_r = 402{\cdot}78$, monoclinic, $P2_1/n$, $a = 7{\cdot}435$ (3), $b = 18{\cdot}982$ (2), $c = 21{\cdot}252$ (4) Å, $\beta = 90{\cdot}66$ (2)°, $V = 2999$ (2) Å^3, $Z = 8$, $D_x = 1{\cdot}78$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 2{\cdot}16$ mm^{-1}, $F(000) = 1568$, $T = 296$ K, final $R = 0{\cdot}068$ for 1577 observed reflections. There are two essentially identical independent molecules in the asymmetric unit. In both molecules the Te atom is bonded to the *p*-methoxyphenyl group, to the cyclohexanone and to the Cl atoms in a pseudo-trigonal-bipyramidal arrangement with axial halogens and with the lone pair at one of the equatorial positions. Coordination distances and angles: Te—Cl = 2·542 (9), 2·47 (1), Te—C(cyclohexanone) = 2·16 (3) and Te—C = 2·10 (3) Å; Cl—Te—C = 86·4 (8), 86·6 (7), 88·3 (9), 92·3 (7), Cl—Te—Cl = 174·4 (3), C—Te—C = 99 (1)° for one molecule and Te′—Cl′ = 2·536 (9), 2·487 (9), Te′—C′(cyclohexanone) = 2·23 (3), Te′—C′ = 2·10 (3) Å; Cl′—Te′—C′ = 88·7 (8), 86·7 (9), 91·0 (8), 89·5 (9), Cl′—Te—Cl′ = 176·1 (3), C′—Te′—C′ = 99 (1)° for the other. Short intramolecular Te···O contacts of 2·93 (1) Å for one molecule and 2·94 (2) Å for the other were found.

Introduction. Methods of organic synthesis based on tellurium reagents have received much attention in the last few years (Petragnani & Comasseto, 1986). α-Telluroketones are among the potential intermediates for this purpose, because of their analogy with the widely used α-selenoketones (Liotta, 1986). Recently, (dichloro)aryltelluroketones were systematically studied for the first time (Stefani, Comasseto & Petragnani, 1987). Compound (I) is one of several α-(dichloro)arytelluroketones synthesized.

To learn about the stereochemistry of this class of compounds, and more specifically about the possible

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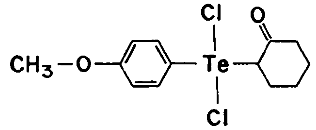
interaction between Te^{IV} and the carbonyl oxygen, a crystal structure determination of (I) was undertaken.



(I)

Experimental. The preparation of the title compound was reported by Stefani, Comasseto & Petragnani (1987). The crystal data were collected with an Enraf–Nonius CAD-4 diffractometer. A prismatic yellow crystal with dimensions 0·15 × 0·04 × 0·02 mm was used with graphite-monochromated Mo $K\alpha$ radiation. 25 reflections ($7{\cdot}6 < \theta < 15{\cdot}5°$) were used to determine cell parameters. Intensities were measured with the ω–2θ scan technique, with scan width $(0{\cdot}80 + 0{\cdot}35\tan\theta)°$, range of hkl: $-7 \leq h \leq 7$, $k \leq 19$, $l \leq 22$, $\theta_{\max} = 22°$; no significant variation was found in the intensities of two standard reflections. 3277 reflections measured, 3118 unique, $R_{\rm sym} = 0{\cdot}019$ from 159 Laue equivalents, after absorption correction; 1579 observed above $3\sigma(I)$; Lp and absorption corrections (max. and min. transmission factors 0·9032 and 0·7222). The structure was solved by Patterson and difference Fourier methods. In final cycles of least-squares refinement only the Te and Cl atoms anisotropic. H atoms were not found and were not included in the model. Function minimized $\sum w(|F_o| - |F_c|)^2$ using unit weights; 175 parameters refined; excluding unobserved and two reflections (204, 114) $R = 0{\cdot}068$; max. $\Delta/\sigma = 0{\cdot}002$; excursions within 1·17 and $-0{\cdot}97$ e Å^{-3}, the highest peaks were found at about 1·1 Å from the Te atoms. Scattering factors from Cromer & Mann (1968) with corrections for anomalous dispersion from Cromer & Liberman (1970). Programs used: *SHELX76* (Sheldrick, 1976) and *ORTEP* (Johnson, 1965). Most of the calculations were performed on a VAX11/780 computer.

Discussion. Fractional atomic coordinates and equivalent and isotropic temperature factors are listed in Table 1.* The crystallographic atom numbering and a view of the molecule are shown in Fig. 1. Bond lengths and angles are given in Table 2. There are two independent molecules in the asymmetric unit which are very nearly enantiomorphous, as indicated by the fact that a least-squares fit (Kabsch, 1976) gives a root-mean-square deviation between homologous atoms of 0·14 Å and a maximum deviation of 0·28 Å between O(2) and O(2′), with a determinant of the orthogonal transformation equal to minus one.

Table 1. *Fractional atomic coordinates and isotropic temperature factors* (Å^2)

$$B_{\rm eq} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}/B_{\rm iso}$
Te(1)	−0·0327 (3)	0·5861 (1)	0·8580 (1)	3·73 (7)
Cl(1)	−0·223 (1)	0·5049 (5)	0·7893 (4)	5·0 (4)
Cl(2)	0·172 (2)	0·6660 (6)	0·9158 (4)	6·9 (4)
O(1)	0·230 (3)	0·713 (1)	0·605 (1)	6·2 (6)
O(2)	−0·096 (3)	0·465 (1)	0·9404 (9)	4·3 (5)
C(1)	0·294 (6)	0·670 (2)	0·555 (2)	8· (1)
C(2)	0·181 (4)	0·682 (2)	0·660 (2)	4·7 (7)
C(3)	0·121 (4)	0·723 (2)	0·710 (1)	3·8 (7)
C(4)	0·066 (4)	0·697 (2)	0·767 (1)	3·7 (6)
C(5)	0·074 (4)	0·625 (2)	0·774 (1)	3·9 (7)
C(6)	0·137 (4)	0·580 (2)	0·729 (1)	4·0 (7)
C(7)	0·196 (5)	0·605 (2)	0·668 (2)	5·8 (9)
C(8)	0·145 (4)	0·497 (1)	0·872 (1)	2·8 (6)
C(9)	0·047 (5)	0·453 (2)	0·921 (2)	4·5 (7)
C(10)	0·148 (5)	0·387 (2)	0·941 (2)	6·3 (9)
C(11)	0·356 (6)	0·403 (2)	0·960 (2)	8· (1)
C(12)	0·430 (5)	0·440 (2)	0·901 (2)	5·2 (8)
C(13)	0·339 (5)	0·513 (2)	0·885 (2)	6·2 (9)
Te(1′)	0·0356 (3)	0·3052 (1)	0·6626 (1)	3·31 (7)
Cl(1′)	0·223 (1)	0·3878 (4)	0·7304 (4)	4·9 (3)
Cl(2′)	−0·165 (1)	0·2303 (5)	0·5963 (4)	5·6 (4)
O(1′)	−0·255 (3)	0·174 (1)	0·911 (1)	5·5 (5)
O(2′)	0·072 (3)	0·418 (1)	0·569 (1)	6·3 (6)
C(1′)	−0·303 (5)	0·219 (2)	0·968 (2)	5·5 (8)
C(2′)	−0·195 (4)	0·207 (2)	0·858 (1)	3·7 (6)
C(3′)	−0·152 (4)	0·164 (2)	0·809 (1)	3·9 (7)
C(4′)	−0·089 (4)	0·191 (2)	0·750 (1)	4·3 (7)
C(5′)	−0·077 (4)	0·264 (2)	0·745 (1)	3·1 (6)
C(6′)	−0·136 (4)	0·309 (2)	0·792 (1)	4·1 (6)
C(7′)	−0·196 (4)	0·280 (2)	0·848 (1)	3·2 (6)
C(8′)	−0·150 (5)	0·396 (2)	0·647 (2)	4·9 (8)
C(9′)	−0·070 (4)	−0·438 (2)	0·595 (1)	3·4 (6)
C(10′)	−0·150 (5)	0·504 (2)	0·582 (2)	5·8 (9)
C(11′)	−0·356 (5)	0·490 (2)	0·575 (2)	5·0 (8)
C(12′)	−0·437 (5)	0·454 (2)	0·627 (2)	5·3 (8)
C(13′)	−0·347 (5)	0·382 (2)	0·640 (2)	6·0 (9)

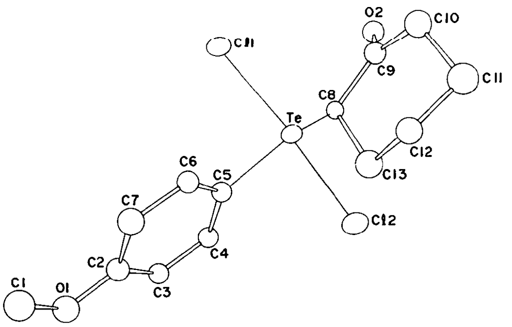


Fig. 1. *ORTEP* drawing of $[TeCl_2(CH_3OC_6H_4)(C_6H_9O)]$ (Johnson, 1965).

The coordination around Te^{IV} may be interpreted by postulating the presence of a stereoactive lone pair on the Te atom giving rise to a pseudo-trigonal-bipyramidal configuration. As has been pointed out (Cotton & Wilkinson, 1972) the siting of this lone pair is hard to predict. In the present case it turns out to be in the equatorial plane together with C(5) and C(8) while the Cl atoms occupy the axial positions. The

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51185 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Interatomic bond distances* (Å) *and bond angles* (°)

Te(1)—Cl(1)	2·542 (9)	Te(1′)—Cl(1′)	2·536 (9)
Te(1)—Cl(2)	2·47 (1)	Te(1′)—Cl(2′)	2·487 (9)
Te(1)—C(5)	2·10 (3)	Te(1′)—C(5′)	2·10 (3)
Te(1)—C(8)	2·16 (3)	Te(1′)—C(8′)	2·23 (3)
O(1)—C(1)	1·43 (5)	O(1′)—C(1′)	1·53 (4)
O(1)—C(2)	1·36 (4)	O(1′)—C(2′)	1·37 (4)
O(2)—C(9)	1·17 (4)	O(2′)—C(9′)	1·26 (4)
C(2)—C(3)	1·39 (4)	C(2′)—C(3′)	1·36 (4)
C(2)—C(7)	1·48 (5)	C(2′)—C(7′)	1·40 (4)
C(3)—C(4)	1·37 (4)	C(3′)—C(4′)	1·44 (4)
C(4)—C(5)	1·38 (4)	C(4′)—C(5′)	1·39 (4)
C(5)—C(6)	1·37 (4)	C(5′)—C(6′)	1·39 (4)
C(6)—C(7)	1·45 (5)	C(6′)—C(7′)	1·39 (4)
C(8)—C(9)	1·53 (4)	C(8′)—C(9′)	1·49 (4)
C(8)—C(13)	1·50 (5)	C(8′)—C(13′)	1·49 (5)
C(9)—C(10)	1·52 (5)	C(9′)—C(10′)	1·41 (5)
C(10)—C(11)	1·62 (6)	C(10′)—C(11′)	1·56 (5)
C(11)—C(12)	1·54 (5)	C(11′)—C(12′)	1·44 (5)
C(12)—C(13)	1·58 (5)	C(12′)—C(13′)	1·55 (5)

Cl(1)—Te(1)—Cl(2)	174·4 (3)	Cl(1′)—Te(1′)—Cl(2′)	176·1 (3)
Cl(1)—Te(1)—C(5)	86·4 (8)	Cl(1′)—Te(1′)—C(5′)	88·7 (8)
Cl(1)—Te(1)—C(8)	86·6 (7)	Cl(1′)—Te(1′)—C(8′)	86·7 (9)
Cl(2)—Te(1)—C(5)	88·3 (9)	Cl(2′)—Te(1′)—C(5′)	91·0 (8)
Cl(2)—Te(1)—C(8)	92·3 (7)	Cl(2′)—Te(1′)—C(8′)	89·5 (9)
C(5)—Te(1)—C(8)	99 (1)	C(5′)—Te(1′)—C(8′)	99 (1)
C(1)—O(1)—C(2)	119 (1)	C(1′)—O(1′)—C(2′)	119 (1)
O(1)—C(2)—C(3)	120 (1)	O(1′)—C(2′)—C(3′)	116 (1)
O(1)—C(2)—C(7)	120 (1)	O(1′)—C(2′)—C(7′)	125 (1)
C(3)—C(2)—C(7)	119 (1)	C(3′)—C(2′)—C(7′)	118 (1)
C(2)—C(3)—C(4)	125 (1)	C(2′)—C(3′)—C(4′)	122 (1)
C(3)—C(4)—C(5)	116 (1)	C(3′)—C(4′)—C(5′)	116 (1)
C(4)—C(5)—C(6)	124 (1)	C(4′)—C(5′)—C(6′)	122 (1)
C(5)—C(6)—C(7)	122 (1)	C(5′)—C(6′)—C(7′)	119 (1)
C(2)—C(7)—C(6)	114 (1)	C(2′)—C(7′)—C(6′)	121 (1)
C(9)—C(8)—C(13)	117 (1)	C(9′)—C(8′)—C(13′)	115 (1)
O(2)—C(9)—C(8)	125 (1)	O(2′)—C(9′)—C(8′)	121 (1)
O(2)—C(9)—C(10)	121 (1)	O(2′)—C(9′)—C(10′)	122 (1)
C(8)—C(9)—C(10)	114 (1)	C(8′)—C(9′)—C(10′)	117 (1)
C(9)—C(10)—C(11)	112 (1)	C(9′)—C(10′)—C(11′)	106 (1)
C(10)—C(11)—C(12)	103 (1)	C(10′)—C(11′)—C(12′)	115 (1)
C(11)—C(12)—C(13)	115 (1)	C(11′)—C(12′)—C(13′)	112 (1)
C(8)—C(13)—C(12)	106 (1)	C(8′)—C(13′)—C(12′)	106 (1)

Cl—Te—Cl angles are 174·4 (3) and 176·1 (3)° for each independent molecule, respectively. Within the coordination sphere, Te—Cl(1) = 2·542 (9), 2·536 (9); Te—Cl(2) = 2·47 (1), 2·487 (9); Te—C(5) = 2·10 (3), 2·10 (3); Te—C(8) = 2·16 (3), 2·23 (3) Å. The Te—Cl bond lengths are close to those found in other $TeCl_2RR'$ compounds (Ziolo & Troup, 1983; Chadha & Drake, 1984; Castellano, Zukerman-Schpector, Ferreira & Comasseto, 1986). Most of the Te^{IV}—C(aryl) bonds lie near the value 2·12 (2) Å (Chadha & Miller, 1982) in good agreement, to within experimental accuracy, with the values found for Te—C(5). Although 2·23 (3) Å is in the upper range for Te^{IV}—C(alkyl) bonds, several bonds have recently been found near to 2·19 (5) Å (Jones & Hamor, 1984; Blom, Haaland & Seip, 1983; Husebye, Meyers, Zingaro, Comasseto & Petragnani, 1987). In both molecules the Te atom is equatorial, as shown in Fig. 2, which is a Newman projection of the cyclohexanone moiety down the C(8)—C(9) bond. The intramolecular distance Te—O(2) = 2·93 (2), 2·94 (2) Å is shorter than the sum of the van der Waals radii of the Te and O of 3·60 Å (Pauling, 1960), and it is much shorter than the upper range of 3·3–3·4 Å found by Alcock, Harrison & Howes (1984) for weak bonds.

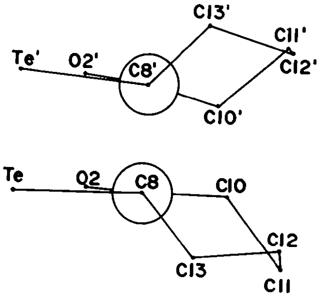

Fig. 2. Newman projection of the cyclohexanone moiety down the C(8)—C(9) bond.

The Cl—Te—Cl angles, 174·4 (3) and 176·1 (3)°, and C—Te—C, 99 (1)° for both molecules, are close to the values found for the corresponding $TeCl_2RR'$, where R is a substituted cyclohexyl group and R' is a tolyl (Husebye, Meyers, Zingaro, Comasseto & Petragnani, 1987; Castellano, Zukerman-Schpector, Ferreira & Comasseto, 1986).

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