

## Structure of Dichloro[(4,4-dimethyl-5-oxo-2,3,4,5-tetrahydro-2-furyl)methyl]- (4-methoxyphenyl)tellurium(IV)

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**Abstract.**  $[\text{TeCl}_2(\text{C}_7\text{H}_7\text{O})(\text{C}_7\text{H}_{11}\text{O}_2)]$ ,  $M_r = 432.8$ , monoclinic,  $P2_1/m$ ,  $a = 11.077(4)$ ,  $b = 9.472(2)$ ,  $c = 16.222(4)$  Å,  $\beta = 101.70(3)^\circ$ ,  $V = 1667(2)$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.73$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 2.17$  mm<sup>-1</sup>,  $F(000) = 848$ ,  $T = 296$  K,  $R = 0.036$  for 1627 observed reflections. The Te atom is in a pseudo trigonal-bipyramidal configuration with the lone pair at one of the equatorial positions. The lactone moiety is almost planar and the furan ring has the typical envelope conformation. Distances and angles are: Te–Cl = 2.490(2), 2.507(3) Å; Te–C = 2.094(7), 2.119(8) Å; Cl–Te–Cl = 174.97(8)°; Cl–Te–C = 89.3(2), 88.5(2), 88.9(2), 87.1(2)°; C–Te–C = 98.3(3)°.

**Introduction.** The development of methods of organic synthesis based on tellurium reagents has received much attention in the last few years. Because it is of interest in this field of organic chemistry, a new cyclofunctionalization reaction was developed using tellurium methodology (Comassetto & Petragnani, 1983) and a series of *p*-methoxyphenyltellurium dichloride lactones were prepared and characterized by <sup>1</sup>H NMR and IR spectra and elemental analysis. The title compound was crystallized and its crystal structure determination was undertaken in order to confirm the reaction product.

**Experimental.** Crystals from chloroform at 277 K, 0.20 × 0.22 × 0.33 mm; Nonius CAD-4 diffractometer; graphite-monochromated Mo *K*α; cell parameters by least squares on setting angles of 21 reflections, 16° < 2θ < 38°; ω–2θ scans, scan width (0.80 + 0.35 tan θ)°, max. scan speed 6.7° min<sup>-1</sup>;

range of *hkl*:  $-12 < h < 12$ ,  $k < 10$ ,  $l < 17$ ,  $\theta_{\text{max}} = 23^\circ$ ; standards 2,  $\bar{1}$ , 10, 622 varied ±2% of mean intensities over data collection; 2400 reflections measured, 2310 unique,  $R_{\text{int}} = 0.024$ , 1627 observed above 2σ(*I*); Lp corrections. Structure solved by direct methods. In final cycles of least-squares refinement all non-hydrogen atoms anisotropic. H atoms included at constrained positions [C–H = 1.01(1) Å] based on those found in difference synthesis, all with fixed isotropic  $U = 0.06$  Å<sup>2</sup>; methyl groups refined as rigid groups. Function minimized  $\sum w(|F_o| - |F_c|)^2$  with  $w = [\sigma^2(F_o) + 0.0042 F_o^2]^{-1}$ , 218 parameters refined; excluding unobserved reflections  $R = 0.036$ ,  $wR = 0.040$ ; inspection of  $F_c$  and  $F_o$  values indicated a correction for secondary extinction required; † max.  $\Delta/\sigma = 0.005$ ,  $\Delta\rho$  excursions within  $-0.54$  and  $0.34$  e Å<sup>-3</sup>; scattering factors for non-H atoms from Cromer & Mann (1968) with corrections for anomalous dispersion from Cromer & Liberman (1970), for H from Stewart, Davidson & Simpson (1965). Programs used: *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), *SHELX76* (Sheldrick, 1976) and *ORTEP* (Johnson, 1965).

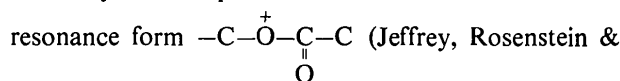
**Discussion.** Fractional atomic coordinates and  $B_{\text{eq}}$  values are listed in Table 1. ‡ The atom numbering and a view of the molecule are given in Fig. 1. Bond lengths and angles are given in Table 2. The main result of this

†  $F_{\text{corr}} = F_o / [1.0 - 10^{-4} \chi F_o^2 / \sin \theta]$ , where  $\chi$  refined to 0.0029 in the final run.

‡ Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42488 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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communication is the confirmation of the molecular formula previously proposed. The phenyl ring is planar, to within experimental accuracy [ $\sigma_{av}$ , defined as  $(\sum d_i^2/N-3)^{1/2}$ , is 0.01 Å]. As observed in other compounds (Harlow & Simonsen, 1976; Bocelli & Grenier-Loustalot, 1981), the lactone moiety is nearly planar [ $\sigma_{av}$  on C(8), O(2), C(11), O(3), C(10) is 0.04 Å] while the ring shows a typical furan-envelope conformation with C(9) 0.38 (1) Å out of the mean plane. The C=O bond [C(11)–O(3) = 1.20 (1) Å] is in good agreement with the accepted value [1.215 (5) Å; Sutton, 1965]. The two C–O bonds are different [1.34 (1), 1.45 (1) Å]; this is often observed in lactone derivatives and may be interpreted as due to the valence-bond



The largest internal angles of the lactone ring are at the carbonyl carbon [110.6 (7)°] and at the oxygen [111.0 (6)°].

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å<sup>2</sup>) with *e.s.d.*'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> <sub>eq</sub> <sup>*</sup>
Te	0.7029 (1)	0.2580 (1)	1.0724 (1)	3.41 (2)
Cl(1)	0.5550 (2)	0.1729 (2)	0.9456 (1)	4.95 (7)
Cl(2)	0.8453 (2)	0.3264 (3)	1.2071 (2)	5.45 (8)
O(1)	0.2658 (5)	0.4744 (6)	1.2249 (4)	4.9 (2)
O(2)	0.8392 (5)	−0.0131 (6)	1.0177 (3)	4.4 (2)
O(3)	0.9060 (6)	−0.1400 (7)	0.9215 (4)	5.4 (2)
C(1)	0.5537 (7)	0.3218 (8)	1.1247 (5)	3.3 (2)
C(2)	0.5457 (7)	0.4619 (8)	1.1460 (6)	4.2 (3)
C(3)	0.4481 (8)	0.5104 (8)	1.1780 (6)	4.7 (3)
C(4)	0.3587 (7)	0.4166 (9)	1.1915 (5)	3.8 (3)
C(5)	0.3652 (7)	0.2764 (9)	1.1715 (5)	3.9 (3)
C(6)	0.4634 (8)	0.2295 (8)	1.1373 (6)	4.3 (3)
C(7)	0.1723 (8)	0.385 (1)	1.2435 (6)	5.1 (3)
C(8)	0.8328 (8)	−0.0237 (9)	1.1056 (6)	4.6 (3)
C(9)	0.8375 (9)	−0.1798 (9)	1.1219 (6)	4.9 (3)
C(10)	0.9078 (7)	−0.2415 (8)	1.0603 (5)	3.6 (2)
C(11)	0.8865 (7)	−0.1311 (9)	0.9913 (5)	3.9 (3)
C(12)	0.7179 (8)	0.0475 (8)	1.1176 (6)	4.3 (3)
C(13)	1.0445 (9)	−0.250 (1)	1.0933 (6)	5.9 (4)
C(14)	0.857 (1)	−0.3848 (9)	1.0248 (7)	6.1 (4)

$$* B_{eq} = \frac{4}{3} \sum_{ij} T_{ij} B_{ij} \quad (\text{Hamilton, 1959}).$$

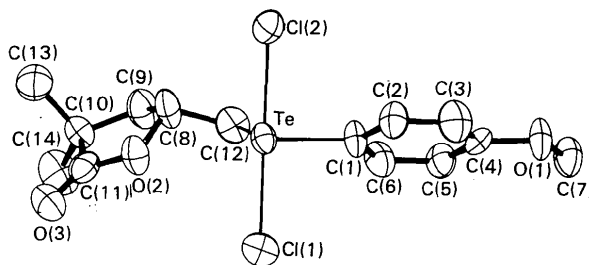


Fig. 1. A view of the molecule with the crystallographic numbering scheme (thermal ellipsoids are drawn at the 50% probability level).

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

Te–Cl(1)	2.490 (2)	Cl(1)–Te–Cl(2)	174.97 (8)
Te–Cl(2)	2.507 (3)	Cl(1)–Te–C(1)	89.3 (2)
Te–C(1)	2.094 (7)	Cl(1)–Te–C(12)	88.5 (2)
Te–C(12)	2.119 (8)	Cl(2)–Te–C(1)	88.9 (2)
		Cl(2)–Te–C(12)	87.1 (2)
		C(1)–Te–C(12)	98.3 (3)
C(1)–C(2)	1.38 (1)	C(2)–C(1)–C(6)	119.3 (7)
C(1)–C(6)	1.37 (1)	C(1)–C(2)–C(3)	120.9 (8)
C(2)–C(3)	1.37 (1)	C(2)–C(3)–C(4)	119.4 (8)
C(3)–C(4)	1.38 (1)	C(3)–C(4)–C(5)	120.8 (8)
C(4)–C(5)	1.37 (1)	C(4)–C(5)–C(6)	119.1 (8)
C(5)–C(6)	1.39 (1)	C(1)–C(6)–C(5)	120.5 (8)
C(4)–O(1)	1.37 (1)	C(3)–C(4)–O(1)	115.2 (7)
C(7)–O(1)	1.42 (1)	C(5)–C(4)–O(1)	124.0 (7)
		C(4)–O(1)–C(7)	119.0 (6)
C(8)–C(9)	1.50 (1)	C(9)–C(8)–O(2)	103.6 (7)
C(8)–O(2)	1.45 (1)	C(9)–C(8)–C(12)	115.1 (8)
C(8)–C(12)	1.49 (1)	O(2)–C(8)–C(12)	108.2 (7)
C(9)–C(10)	1.50 (1)	C(8)–C(9)–C(10)	105.7 (7)
C(10)–C(11)	1.51 (1)	C(9)–C(10)–C(11)	101.5 (7)
C(10)–C(13)	1.50 (1)	C(9)–C(10)–C(13)	113.8 (7)
C(10)–C(14)	1.54 (1)	C(9)–C(10)–C(14)	113.1 (7)
C(11)–O(2)	1.34 (1)	C(11)–C(10)–C(13)	107.2 (7)
C(11)–O(3)	1.20 (1)	C(11)–C(10)–C(14)	110.0 (7)
		C(13)–C(10)–C(14)	110.6 (7)
		C(10)–C(11)–O(2)	110.6 (7)
		C(10)–C(11)–O(3)	128.2 (8)
		O(2)–C(11)–O(3)	121.2 (8)
		C(8)–O(2)–C(11)	111.0 (6)

The coordination around Te<sup>IV</sup> 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(1) and C(12) while the chlorines occupy the axial positions. The angle between the normal to the equatorial plane and the line through the Cl atoms is 180° and Cl–Te–Cl is 174.97 (8)°. The Te–C bond lengths are, as in other R<sub>2</sub>TeCl<sub>2</sub> molecules (Chadha, Drake & Kahn, 1983; Chadha, Drake & Hencher, 1983; Ziolo & Troup, 1983), close to that predicted from the sum of the single-bond covalent radii. The C–Te–C angle is also within the range found for these species. This angle is less than the ideal of 120° and the distortion has been explained on the basis of lone-pair–bond-pair repulsions being greater than bond-pair–bond-pair repulsions (Chadha, Drake & Khan, 1983). The shortest non-bonded Te...Cl distance [4.956 (2) Å] is greater than the sum of van der Waals radii (4.0 Å). The ratio Te...Cl/Te–Cl is 2.0 and so it must be presumed that the secondary Te...Cl interactions are insignificant in this compound. The intramolecular Te...O(2) distance [3.194 (5) Å] is shorter than the sum of van der Waals radii (3.60 Å).

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