The tetrahydropyran ring has a chair conformation and the oxacyclodecane part has a distorted boat–chair–boat conformation.

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Structure of (*Z*)-1-*p*-Methoxyphenyltelluro-2-phenylethene

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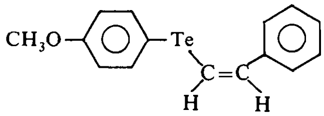
Abstract. $C_{15}H_{14}OTe$, $M_r = 337{\cdot}9$, triclinic, $P\bar{1}$, $a = 7{\cdot}878\,(3)$, $b = 5{\cdot}757\,(2)$, $c = 14{\cdot}853\,(5)$ Å, $\alpha = 90{\cdot}28\,(3)$, $\beta = 92{\cdot}60\,(3)$, $\gamma = 91{\cdot}08\,(3)°$, $V = 672{\cdot}8\,(4)$ Å^3, $Z = 2$, $D_x = 1{\cdot}67$ Mg m^{-3}, λ(Mo $K\alpha$) = $0{\cdot}71073$ Å, $\mu = 2{\cdot}27$ mm^{-1}, $F(000) = 328$, $T = 296$ K, final $R = 0{\cdot}060$ for 2156 unique observed reflections. The average Te—C bond distance is 2·103 (11) Å and the C=C bond distance is 1·317 (7) Å. The Te—C=C—phenyl grouping is nearly planar and rotated approximately 66° from the planar *p*-methoxyphenyl group. Steric effects in the molecule are apparent, with Te—C=C = 130·9 (4)° and C=C—phenyl = 130·4 (5)°. There is a short intermolecular Te···Te′ distance, 3·839 (1) Å.

Introduction. We have determined the structure of a molecule in which Se^{II} is directly bonded to an acetylenic C and to a phenyl C (Husebye, Meyers, Zingaro, Braga, Comasseto & Petragnani, 1986). In that structure, the bond from Se to the acetylenic C is 0·079 Å shorter than that from Se to the phenyl C. In addition, there seems to be some slight indication of secondary bonding between Se atoms, linking the molecules into zigzag chains. We wished to explore the possibility of similar structural features in molecules which have Te attached to phenyl groups and to unsaturated C atoms and have determined the present structure (I) in order to do so.



(I)

Experimental. The preparation of the title compound has been reported previously (Dabdoub, Dabdoub, Comasseto & Petragnani, 1986). The material was recrystallized from ethanol as very-pale-yellow plates. X-ray data were collected by the Molecular Structure Corporation, College Station, Texas. A plate 0·40 × 0·20 × 0·05 mm was used for intensity measurements with graphite-monochromatized Mo $K\alpha$ radiation on an Enraf–Nonius CAD-4 diffractometer. Cell constants were obtained from least-squares refinement of 2θ for 25 reflections in the range $5 < \theta < 12°$. The crystals are triclinic, $P1$ or $P\bar{1}$. At $T = 296$ K, 3097 independent reflections were examined and the intensities were measured ($\theta/2\theta$ scan mode) for 2464 of them for which $I > 0{\cdot}5\sigma(I)$ and $2\theta < 55°$ $(0 \le h \le 10$, $-6 \le k \le 6$,

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Table 1. *Fractional coordinates and equivalent isotropic temperature factors* (Å²) *for non*-H *atoms*

$$U_{\rm eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*(\mathbf{a}_i.\mathbf{a}_j).$$

	x	y	z	$U_{\rm eq}$
Te	0·72044 (7)	0·42082 (9)	0·46065 (3)	0·0577 (2)
C1	0·8284 (9)	0·1272 (12)	0·4047 (5)	0·0544 (26)
C2	0·8581 (9)	0·0750 (12)	0·3205 (5)	0·0532 (25)
C3	0·8287 (9)	0·2071 (12)	0·2385 (5)	0·0481 (23)
C4	0·8953 (9)	0·1267 (14)	0·1586 (5)	0·0581 (27)
C5	0·8723 (11)	0·2435 (16)	0·0787 (5)	0·0687 (32)
C6	0·7799 (13)	0·4408 (16)	0·0736 (6)	0·0754 (35)
C7	0·7081 (12)	0·5245 (14)	0·1499 (6)	0·0685 (31)
C8	0·7314 (11)	0·4092 (13)	0·2314 (5)	0·0598 (27)
C9	0·7284 (9)	0·3034 (12)	0·5954 (5)	0·0502 (23)
C10	0·8082 (9)	0·4400 (12)	0·6623 (5)	0·0537 (25)
C11	0·8019 (10)	0·3794 (13)	0·7511 (5)	0·0562 (26)
C12	0·7117 (9)	0·1786 (12)	0·7758 (5)	0·0484 (23)
C13	0·6331 (10)	0·0399 (14)	0·7093 (5)	0·0597 (27)
C14	0·6424 (11)	0·1061 (13)	0·6197 (5)	0·0603 (27)
C15	0·6260 (12)	−0·0732 (16)	0·8930 (5)	0·0720 (33)
O	0·7098 (7)	0·1341 (10)	0·8655 (3)	0·0641 (19)

Table 2. *Interatomic distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Te—C1	2·092 (5)	Te—C9	2·114 (5)
C1—C2	1·317 (7)	C2—C3	1·451 (7)
C3—C4	1·400 (7)	C4—C5	1·373 (8)
C5—C6	1·362 (9)	C6—C7	1·378 (9)
C7—C8	1·390 (8)	C8—C3	1·407 (7)
C9—C10	1·385 (7)	C10—C11	1·370 (7)
C11—C12	1·404 (7)	C12—C13	1·384 (7)
C13—C14	1·391 (7)	C14—C9	1·370 (7)
C12—O	1·358 (6)	C15—O	1·423 (7)
C9—Te—C1	96·7 (2)	Te—C1—C2	130·9 (4)
Te—C9—C10	119·4 (4)	Te—C9—C14	121·5 (4)
C1—C2—C3	130·4 (5)	C2—C3—C4	118·9 (5)
C2—C3—C8	125·0 (5)	C15—O—C12	117·9 (4)
C11—C12—O	116·2 (4)	C13—C12—O	124·5 (5)
C3—C4—C5	121·8 (5)	C4—C5—C6	121·1 (6)
C5—C6—C7	119·4 (6)	C6—C7—C8	120·1 (5)
C7—C8—C3	121·4 (5)	C8—C3—C4	116·1 (5)
C9—C10—C11	120·8 (5)	C10—C11—C12	120·2 (5)
C11—C12—C13	119·3 (5)	C12—C13—C14	119·0 (5)
C13—C14—C9	121·9 (5)	C14—C9—C10	118·7 (5)

$-15 \le l \le 15$). The intensities of three standard reflections were measured periodically and no significant changes in their values were observed. Corrections were applied for Lorentz and polarization effects but not for absorption. Using the data supplied by the Molecular Structure Corporation, the position of Te was found from a Patterson map and the structure was solved and refined in $P\bar{1}$ by means of least squares and Fourier methods using *SHELX*76 (Sheldrick, 1976). H atoms at calculated positions (C—H = 0·96 Å) were assigned the same isotropic temperature factors as the C atoms to which they are bonded. The H-atom parameters were then held fixed while the anisotropic refinement of the other atoms was carried out until all shifts were less than 0·06 e.s.d., using 2156 reflections for which $F_o > \sigma(F_o)$. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$; $R = 0{\cdot}060$, $wR = 0{\cdot}054$, $S = 1{\cdot}59$, $w = \sigma^2(F_o)$, $\sigma^2(I) = \sigma^2 + (0{\cdot}05I)^2$, with σ calculated from counting statistics. The final difference electron density ranged from +0·89 to −1·65 e Å^{-3}. Scattering factors were taken from Cromer & Liberman (1970), Cromer & Mann (1968), and Stewart, Davidson & Simpson (1965).

Discussion. Final parameters for the non-H atoms are listed in Table 1,* interatomic distances and angles are listed in Table 2, and a *SHELXTL-Plus* (Sheldrick, 1987) drawing of the molecule is shown in Fig. 1. The average C—C distance in the two phenyl rings, uncorrected for thermal motion, is 1·386 (4) Å, which is a value typical of that determined in X-ray work (Domenicano, Murray-Rust & Vaciago, 1983). The C1—C2 double-bond distance, 1·317 (7) Å, is only slightly shorter than the expected value of 1·33–1·34 Å and the discrepancy may be due to the same effects that influence the average phenyl-ring distance. The distances C2—C3 = 1·451 (7) Å and O—C12 = 1·358 (6) Å are both considerably shorter than expected from single-bond radii (Pauling, 1960), presumably for the reasons cited above and because of their conjugation with the phenyl rings to which they are attached. They are in reasonable agreement with empirical values proposed by Lide (1962), 1·47–1·48 and 1·34 Å respectively. The distance O—C15 = 1·423 (7) Å is only slightly less than the value of 1·43 Å predicted from Pauling (1960) single-bond radii. Using the Lide (1962) radius for trigonal C and the Pauling (1960) radius for Te, the predicted Te—C distance is (0·74 + 1·37) = 2·11 Å, in good agreement with the values observed, Te—Cl = 2·092 (5), Te—C9 = 2·114 (5) Å.

The influence of substituents upon phenyl-ring geometry has been discussed at length in a paper by Domenicano, Vaciago & Coulson (1975). In particular, they presented qualitative arguments regarding the effects upon internal ring angles of many different substituent characteristics. In the present molecule, the *ipso* angle at C3 is 116·1 (5)°, in agreement with their predicted closing of this angle because of conjugation with the ring, and the *ipso* angle at C9 is 118·7 (5)°,

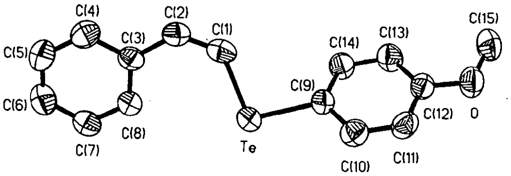

Fig. 1. Drawing of $C_{15}H_{14}OTe$ (Sheldrick, 1987) with H atoms not shown. Thermal ellipsoids at the 50% probability level.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, interplanar angles and shortest intermolecular contact distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44971 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

with its reduced value possibly due to geminal non-bonded interactions of C10 and C14 with the large Te atom to which C9 is attached or to VSEPR effects. Steric effects, whether attributed to non-bonded atom interactions or to electron-pair repulsions, seem to be indicated by the angles C1—C2—C3 = 130·4 (5)° and Te—C1—C2 = 130·9 (4)°. A valuable scheme for the correlation of 1···3 intramolecular distances has been developed by Bartell and his co-workers (Bartell, 1968) and applied to a wide range of inorganic materials (*e.g.* O'Keeffe & Hyde, 1981). The distance C1···C3 = 2·513 Å is in good agreement with the value 2·50 Å predicted from the 1···3 radius for C of 1·25 Å. Since Te···C2 = 3·117 Å, the 1···3 radius for Te may be estimated to be 1·87 Å, compared to the van der Waals radius of 2·20 Å (Pauling, 1960). An earlier application of Bartell's idea to α-TeO_2 gave 1·87 Å for the estimated 1···3 radius for Te (Glidewell, 1979). There is a short Te···Te′ intermolecular contact of 3·84 Å, across the center at $\frac{1}{2},\frac{1}{2},\frac{1}{2}$, but the angles C1—Te—Te′ = 136° and C9—Te—Te′ = 77° do not have any obvious pictorial interpretation. All other intermolecular contact distances are normal.

The atoms C9, Te, C1, C2 and C3 lie close to one plane, with the phenyl ring C3–C8 rotated 10° from it in one direction and the other ring (C9–C14) turned 58° in the opposite sense. The planes O, C12, C15 and C9–C14 are rotated 1·7° from each other.

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Structure of the 4-Phenyl-1,2-dithia-3,5-diazolium Chloride Adduct with Toluene, $C_7H_5N_2S_2^+.Cl^-.\frac{1}{6}C_7H_8$

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Abstract. M_r = 232·07, triclinic, $P\bar{1}$, a = 15·774 (10), b = 7·564 (5), c = 13·787 (9) Å, α = 100·23 (3), β = 109·85 (4), γ = 95·20 (4)°, V = 1502 (2) Å^3, Z = 6, D_x = 1·539 (2) Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 0·734 (1) mm^{-1}, $F(000)$ = 710, room temperature, $R(F)$ = 0·055 for 1523 reflexions [$I > 3\sigma(I)$] and 172 variables. There are three independent phenyl-dithiadiazolium ions in the asymmetric unit. The mean distances in the dithiadiazolium rings are S—S = 1·990 (3), S—N = 1·590 (4), C—N = 1·340 (5) Å. Each of the three independent cations has a Cl$^-$ close to its plane and about 2·9 Å from each S. There are also weak S—Cl interactions linking the disulfide groups and the chloride ions in planes parallel to (10$\bar{1}$). The toluene lies on a symmetry centre and is disordered.

Introduction. Short disulfide-to-anion distances have been observed in several compounds. In the cases where the disulfide group is part of an aromatic ring the anion is close to the plane of the ring and is approximately equidistant from the two S atoms (Andreasen, Hazell & Hazell, 1977, and references therein). The structure of 4-phenyl-1,2-dithia-3,5-diazolium chloride (Alange, Banister, Bell & Millen, 1977) has been determined for comparison with related compounds.

Experimental. The compound, which was provided by Dr A. J. Banister, recrystallized from toluene as orange-yellow needles. A crystal of dimensions 0·10 × 0·06 × 0·75 mm was mounted on a Picker FACS-1 diffractometer and cell dimensions determined from the

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