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

C(1*A*)—C(2*A*)	1·537 (5)	C(1*B*)—C(6*B*)	1·525 (5)
C(2*A*)—C(3*A*)	1·525 (5)	C(3*B*)—C(4*B*)	1·527 (5)
C(4*A*)—C(11*A*)	1·509 (5)	C(4*B*)—O(12*B*)	1·438 (5)
C(7*A*)—C(8*A*)	1·525 (6)	C(7*B*)—C(9*B*)	1·514 (6)
C(1*B*)—C(2*B*)	1·543 (5)	C(1*A*)—C(7*A*)	1·554 (5)
C(2*B*)—C(3*B*)	1·513 (5)	C(4*A*)—C(5*A*)	1·544 (6)
C(4*B*)—C(11*B*)	1·513 (6)	C(5*A*)—C(6*A*)	1·530 (6)
C(7*B*)—C(8*B*)	1·520 (6)	C(7*A*)—O(10*A*)	1·463 (5)
C(1*A*)—C(6*A*)	1·520 (6)	C(1*B*)—C(7*B*)	1·543 (5)
C(3*A*)—C(4*A*)	1·521 (5)	C(4*B*)—C(5*B*)	1·531 (6)
C(4*A*)—O(12*A*)	1·427 (5)	C(5*B*)—C(6*B*)	1·534 (6)
C(7*A*)—C(9*A*)	1·504 (6)	C(7*B*)—O(10*B*)	1·458 (4)

C(2*A*)—C(1*A*)—C(6*A*)	109·8 (3)	C(2*A*)—C(1*A*)—C(7*A*)	113·2 (2)
C(6*A*)—C(1*A*)—C(7*A*)	112·6 (3)	C(1*A*)—C(2*A*)—C(3*A*)	110·7 (3)
C(2*A*)—C(3*A*)—C(4*A*)	113·6 (3)	C(3*A*)—C(4*A*)—C(5*A*)	108·9 (3)
C(3*A*)—C(4*A*)—C(11*A*)	111·6 (3)	C(3*A*)—C(4*A*)—O(12*A*)	109·4 (3)
C(5*A*)—C(4*A*)—C(11*A*)	111·3 (3)	C(5*A*)—C(4*A*)—O(12*A*)	106·4 (3)
C(11*A*)—C(4*A*)—O(12*A*)	109·0 (3)	C(4*A*)—C(5*A*)—C(6*A*)	112·0 (3)
C(1*A*)—C(6*A*)—C(5*A*)	112·2 (3)	C(1*A*)—C(7*A*)—C(8*A*)	111·7 (3)
C(1*A*)—C(7*A*)—C(9*A*)	113·9 (3)	C(1*A*)—C(7*A*)—O(10*A*)	108·5 (3)
C(8*A*)—C(7*A*)—C(9*A*)	111·3 (3)	C(8*A*)—C(7*A*)—O(10*A*)	105·9 (3)
C(9*A*)—C(7*A*)—O(10*A*)	105·1 (3)	C(2*B*)—C(1*B*)—C(6*B*)	109·5 (3)
C(2*B*)—C(1*B*)—C(7*B*)	113·2 (3)	C(6*B*)—C(1*B*)—C(7*B*)	113·2 (3)
C(1*B*)—C(2*B*)—C(3*B*)	110·9 (3)	C(2*B*)—C(3*B*)—C(4*B*)	113·9 (3)
C(3*B*)—C(4*B*)—C(5*B*)	109·3 (3)	C(3*B*)—C(4*B*)—C(11*B*)	112·3 (3)
C(3*B*)—C(4*B*)—O(12*B*)	109·2 (3)	C(5*B*)—C(4*B*)—C(11*B*)	111·1 (3)
C(5*B*)—C(4*B*)—O(12*B*)	106·7 (3)	C(11*B*)—C(4*B*)—O(12*B*)	108·1 (3)
C(4*B*)—C(5*B*)—C(6*B*)	111·9 (3)	C(1*B*)—C(6*B*)—C(5*B*)	112·5 (3)
C(1*B*)—C(7*B*)—C(8*B*)	111·2 (3)	C(1*B*)—C(7*B*)—C(9*B*)	114·0 (3)
C(1*B*)—C(7*B*)—O(10*B*)	108·5 (3)	C(8*B*)—C(7*B*)—C(9*B*)	110·1 (3)
C(8*B*)—C(7*B*)—O(10*B*)	106·8 (3)	C(9*B*)—C(7*B*)—O(10*B*)	105·8 (3)

Table 3. *The* O···O *distances* (Å) *in the unit cell*

O(10*A*)—O(13*A*)	2·861 (4)	O(12*A*)—O(10*B*)	2·725 (4)
O(10*A*)—O(12*A*)	2·727 (4)	O(13*A*)—O(10*A*)	2·861 (4)
O(10*A*)—O(13*B*)	2·770 (4)	O(13*A*)—O(12*A*)	2·727 (4)
O(12*A*)—O(13*A*)	2·727 (4)	O(13*A*)—O(10*B*)	2·798 (4)
O(10*B*)—O(12*A*)	2·725 (4)	O(12*B*)—O(13*B*)	2·722 (4)
O(10*B*)—O(13*A*)	2·798 (4)	O(13*B*)—O(10*A*)	2·770 (4)
O(10*B*)—O(13*B*)	2·872 (4)	O(13*B*)—O(10*B*)	2·872 (4)
O(12*B*)—O(10*A*)	2·727 (4)	O(13*B*)—O(12*B*)	2·722 (4)

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Structure of *p*-Chlorophenyl(phenylseleno)acetylene

By S. Husebye, E. A. Meyers and R. A. Zingaro

Department of Chemistry, Texas A&M University, College Station, Texas 77843, *USA*

and A. L. Braga, J. V. Comasseto and N. Petragnani

Instituto de Química, Universidade de São Paulo, Caixa Postal 20.780, *São Paulo, Brazil*

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Abstract. $C_{14}H_9ClSe$, $M_r = 291\cdot6$, monoclinic, $P2_1$, $a = 7\cdot270\,(2)$, $b = 5\cdot835\,(3)$; $c = 14\cdot550\,(6)$ Å, $\beta = 100\cdot83\,(3)°$, $V = 606\cdot3$ Å^3, $Z = 2$, $D_x = 1\cdot597$ Mg m^{-3}, $\lambda(\text{Mo}\,K\alpha) = 0\cdot71073$ Å, $\mu = 3\cdot25$ mm^{-1}, $F(000) = 288$, $T = 296$ K, final $R = 0\cdot048$ for 1309 unique observed reflections. The structure consists of molecules of *p*-ClPhC≡CSePh possibly connected by very weak, secondary Se···Cl bonds of 3·704 (2) Å into zigzag chains parallel to the *ab* plane. In the nearly linear C—C≡C—Se— sequence, the bond lengths are 1·446 (9), 1·174 (10) and 1·836 (7) Å, respectively, while Se—C(Ph) = 1·915 (5) Å and angle C—Se—C = 98·4 (3)°.

Introduction. We recently determined the structure of a Wittig-reaction intermediate, phenacyl-α-(phenylseleno)triphenylphosphorane (Husebye, Meyers, Zingaro, Braga, Comasseto & Petragnani, 1986). Heating this compound under vacuum results in the formation of a phenylselenoacetylene and triphenylphosphine oxide (Braga, Comasseto & Petragnani, 1984). The present study was undertaken in order to learn something about the structure of molecules in which Se is bonded directly to an acetylenic C atom.

Experimental. The preparation of *p*-chlorophenyl(phenylseleno)acetylene has previously been reported

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(Braga, Comasseto & Petragnani, 1984). X-ray data were collected by the Molecular Structure Corporation, College Station, Texas. A pale yellow prism $0{\cdot}30 \times 0{\cdot}20 \times 0{\cdot}20$ mm was used for intensity measurements with graphite-monochromatized Mo $K\alpha$ radiation. Cell constants were obtained by a least-squares refinement of 2θ values for 25 reflections in the range $3 < \theta < 11^\circ$. From systematic absences, $0k0 = 2n + 1$, the monoclinic crystals have the space group $P2_1$ or $P2_1/m$. Intensities of 1925 independent reflections with $2\theta < 60^\circ$ were measured at 296 ± 1 K with an Enraf–Nonius CAD-4 diffractometer operated in the ω–2θ scan mode ($0 \le h \le 10$, $0 \le k \le 8$, $-20 \le l \le 20$). No significant decline in the intensities of three standard reflections was observed. Of the reflections collected, 421 had $I < 0{\cdot}5\sigma(I)$ and were regarded as unobserved. These were rejected along with an additional 195 reflections which had $F < \sigma(F)$. Corrections were applied for Lorentz and polarization effects and for absorption (empirical, min. transmission coefficient = 0·884, max. = 0·999). *SHELX76* (Sheldrick, 1976) modified for a PDP 1144 computer was used for the structure determination. The structure was solved in $P2_1$ by Patterson and Fourier methods and refined by successive, experimentally weighted least-squares interactions. Positions of H atoms were calculated for C—H = 1·08 Å, and initially assigned the same isotropic temperature factors as the C atoms to which they belong. These H-atom parameters were adjusted once before the final least-squares iterations. The anisotropic refinement of non-H-atom parameters was then continued and terminated when all parameter shifts were less than $0{\cdot}07\sigma$. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$; $R = 0{\cdot}0477$, $wR = 0{\cdot}0453$, $S = 1{\cdot}080$, $w = 1/\sigma^2(F)$, $\sigma(I) = [\sigma^2 + (0{\cdot}05I)^2]^{1/2}$ with σ calculated from counting statistics. A refinement of the inverse structure gave $R = 0{\cdot}0525$. This was rejected according to Hamilton's test (Hamilton, 1965). A final difference electron density map ranged from +0·6 to −0·4 e Å^3. Scattering factors used were taken from Cromer & Liberman (1970), Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965).*

Discussion. Final parameters are listed in Table 1, interatomic distances and angles are listed in Table 2, and an *ORTEP* drawing of the molecule is shown in Fig. 1. In the title compound, the central acetylene group and the atoms bonded to it form an almost linear four-atom sequence C(3)—C(2)—C(1)—Se. Angles C(3)—C(2)—C(1) and C(2)—C(1)—Se are 176·6 (6) and 179·3 (6)°, respectively, while the bond lengths are C(3)—C(2) 1·446 (9), C(2)—C(1) 1·174 (10) and C(1)—Se 1·836 (7) Å. The acetylenic bond lengths of 1·174 (10) Å may be compared with 1·2033 (2) Å found in acetylene itself (Fast & Welsh, 1972; van Nes & van Bolhuis, 1979) and to 1·20 (3) Å found for most acetylenic triple bonds listed in *Landolt–Börnstein* (1976). However, these latter bond lengths are determined in the gas phase, mostly by electron diffraction and microwave methods. X-ray diffraction results give bond lengths at the lower end of the above range, mostly at 1·17–1·20 Å (Ferguson & Islam, 1966; Majesté & Meyers, 1972; Cotrait, Destrade & Gasparoux, 1977; Mavridis & Moustakali-Mavridis, 1977; Jungk & Schmidt, 1971; Irngartinger, Leiserowitz & Schmidt, 1970).

Table 1. *Fractional coordinates and equivalent isotropic temperature factors* (Å^2) *for non-*H *atoms*

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

	x	y	z	$U_{\rm eq}$
Se	0·38200 (8)	0·5	0·19947 (4)	0·05447 (0)
Cl	−0·40649 (2)	1·38381 (4)	0·44707 (1)	0·05967 (1)
C(1)	0·2300 (9)	0·6816 (13)	0·2563 (4)	0·0551 (2)
C(2)	0·1334 (8)	0·7964 (14)	0·2936 (4)	0·0517 (2)
C(3)	0·0060 (8)	0·9367 (10)	0·3345 (3)	0·0410 (2)
C(4)	−0·1784 (8)	0·8640 (12)	0·3310 (4)	0·0471 (2)
C(5)	−0·3054 (7)	1·0008 (18)	0·3661 (3)	0·0483 (2)
C(6)	−0·2464 (8)	1·2110 (12)	0·4047 (4)	0·0423 (2)
C(7)	−0·0657 (8)	1·2867 (12)	0·4098 (4)	0·0467 (2)
C(8)	0·0620 (7)	1·1506 (12)	0·3743 (4)	0·0446 (2)
C(9)	0·3003 (7)	0·6022 (11)	0·0733 (4)	0·0401 (2)
C(10)	0·3364 (8)	0·4577 (11)	0·0028 (4)	0·0482 (2)
C(11)	0·2840 (9)	0·5132 (19)	−0·0887 (4)	0·0647 (2)
C(12)	0·1973 (9)	0·7264 (15)	−0·1125 (4)	0·0620 (3)
C(13)	0·1611 (8)	0·8740 (14)	−0·0427 (5)	0·0624 (3)
C(14)	0·2160 (8)	0·8146 (12)	0·0496 (4)	0·0492 (2)

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

Se—C(1)	1·836 (7)	C(7)—C(8)	1·393 (9)
Se—C(9)	1·915 (5)	C(6)—Cl	1·738 (6)
C(1)—C(2)	1·174 (10)	C(9)—C(10)	1·390 (9)
C(2)—C(3)	1·446 (9)	C(9)—C(14)	1·397 (9)
C(3)—C(4)	1·398· (8)	C(10)—C(11)	1·353 (8)
C(3)—C(8)	1·404 (9)	C(11)—C(12)	1·408 (13)
C(4)—C(5)	1·388 (10)	C(12)—C(13)	1·394 (11)
C(5)—C(6)	1·384 (11)	C(13)—C(14)	1·372 (9)
C(6)—C(7)	1·374 (8)	Se···Cl$^{\rm i}$	3·704 (2)

C(1)—Se—C(9)	98·4 (3)	C(5)—C(6)—Cl	118·8 (4)
Se—C(1)—C(2)	179·3 (6)	C(7)—C(6)—Cl	119·4 (5)
C(1)—C(2)—C(3)	176·6 (6)	Se—C(9)—C(10)	116·8 (4)
C(2)—C(3)—C(4)	119·8 (5)	Se—C(9)—C(14)	123·7 (4)
C(2)—C(3)—C(8)	120·9 (5)	C(9)—C(10)—C(11)	121·6 (7)
C(3)—C(4)—C(5)	120·7 (6)	C(10)—C(11)—C(12)	118·9 (7)
C(4)—C(5)—C(6)	118·9 (5)	C(11)—C(12)—C(13)	120·3 (6)
C(5)—C(6)—C(7)	121·8 (6)	C(12)—C(13)—C(14)	119·8 (7)
C(6)—C(7)—C(8)	119·6 (6)	C(13)—C(14)—C(9)	119·9 (6)
C(7)—C(8)—C(3)	119·8 (5)	C(14)—C(9)—C(10)	119·4 (5)
C(8)—C(3)—C(4)	119·2 (5)		

Symmetry code: (i) $1 + x, -1 + y, z$.

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* Lists of anisotropic temperature factors for non-H atoms, H-atom coordinates, shortest intermolecular distances, least-squares planes and structure amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43193 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The same trend in bond lengths is not repeated for the C(Ph)–C(acetylene) bonds. *Landolt–Börnstein* (1976) and the X-ray structure just cited yield such bond lengths predominantly in the range 1·44 (1) Å, in excellent agreement with the present investigation (1·446 Å) and with the sum of the covalent radii (1·44 Å) for C_{sp} and C_{sp^2} (Bastiensen & Trætteberg, 1962).

There is a great asymmetry in the two Se—C bond lengths. The bond to acetylenic C is 0·079 Å shorter than that to the phenyl group. This is about twice the difference expected based on the difference in the radius of C_{sp^2} and C_{sp} (Bastiansen & Trætteberg, 1962). While the Se—C(9) bond of 1·915 (3) Å has the bond length expected for a bond between Se and aromatic C (Husebye *et al.*, 1986), the Se—C(1) bond, 1·836 (7) Å, has about the same length as Se—CN bonds found in selenocyanates and related compounds (Hauge, 1975; Marsden & Sheldrick, 1971; Maartmann-Moe, Sanderud & Songstad, 1984). The shortening of *X*—CN bonds (*X* = Te, Se, S) in pseudohalides was interpreted by the latter authors in terms of double-bond character in the *X*—C bonds. For comparison, the Se—C double bond found in CSSe has a length of 1·693 (2) Å (Hirose & Curl, 1971) which indicates that the π-bond character of the Se—C(1) bond is not very great. Also, halogen atoms bonded to acetylene show a similar shortening in their bonds to acetylenic C atoms (Bjorseth, Kloster-Jensen, Marstokk & Molendal, 1970).

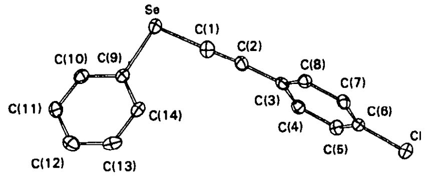

Fig. 1 *ORTEP* (Johnson, 1965) drawing of $C_{14}H_9ClSe$ (H atoms not shown).

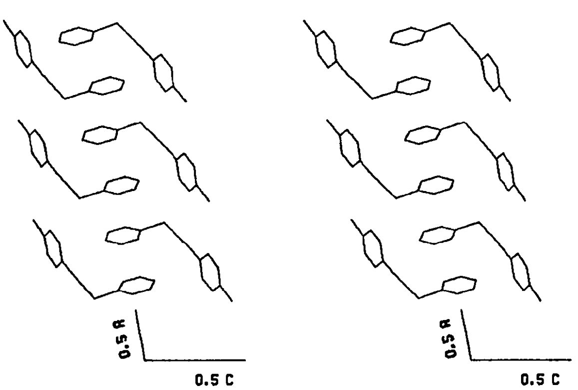

Fig. 2. Stereodiagram of $C_{14}H_9$ ClSe as viewed along the *b* axis.

The two phenyl groups are essentially planar and their interplanar angle is 93·9°. Average C—C bond lengths are 1·390 Å for the Cl-substituted group and 1·386 Å for the other. Both phenyl groups have average C—C—C angles of 120·0°. The angles at the Cl- and Se-substituted C atoms are 121·8 (6) and 119·4 (5)°, respectively, in qualitative agreement with the values predicted by Domenicano, Vaciago & Coulson (1975). There are zigzag chains of molecules possibly connected by very weak, secondary Se···Cl bonds of 3·704 (2) Å parallel to the *ab* plane (Fig. 2). For comparison, a van der Waals contact between those atoms is expected to be 3·80 Å (Pauling, 1960). The contact is roughly *trans* to the Se—C(9) bond. There are no other especially short intermolecular contacts.

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Structure of Oxodipine:* a New Calcium Antagonist

By I. Fonseca, S. Martínez-Carrera and S. García-Blanco

Departamento de Rayos X, Instituto de Química-Física 'Rocasolano', Serrano 119, 28006 *Madrid, Spain*

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Abstract. $C_{19}H_{21}NO_6$, $M_r = 359·4$, monoclinic, $P2_1/c$, $a = 15·019$ (7), $b = 8·285$ (2), $c = 14·651$ (6) Å, $\beta = 102·6$ (2)°, $V = 1779$ (2) Å^3, $Z = 4$, $D_x = 1·342$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 7·95$ cm^{-1}, $F(000) = 760$, $T = 293$ K, $R = 0·065$ for 2531 observed reflections. The dihydropyridine ring presents a flat-boat conformation. The pentagonal heterocycle has an envelope conformation with the flap at C10. The O—C—C—O part of the five-membered ring is planar and coplanar with the phenyl ring. The methylenedioxyphenyl moiety is nearly perpendicular to the dihydropyridine ring.

Introduction. The vasodilating effect of calcium antagonists finds clinical application, especially in the treatment of oxygen-deficiency diseases of the heart, such as angina pectoris. In recent years, the study of 4-aryldihydropyridinedicarboxylates has undergone a great development; these 4-aryl derivatives are highly active calcium antagonists: oxodipine, studied here, belongs to this group, hence the importance of its structural study.

Experimental. Light-yellow, acicular crystals were kindly supplied by Dr L. Veiga (Departamento de Farmacia Galénica, Universidad Complutense, Madrid). A suitable crystal of approximate size 0·37 × 0·27 × 0·33 mm was mounted on a Philips PW 1100 automatic four-circle diffractometer. Cell dimensions obtained by least-squares refinement from 83 reflections with $12 < 2\theta < 87°$. Intensity data collected for $2 < \theta < 65°$ using graphite-monochromatized Cu $K\alpha$ radiation and ω–2θ-scan technique; two standard reflections (202, $\bar{2}0\bar{2}$) measured every 90 min showed no significant variation in intensity; Lorentz and polarization corrections; no correction for absorption 3385 ($-17 < h < 17$, $0 < k < 10$, $0 < l < 17$) data measured with $\theta_{\max} = 65°$. 2531 observed data with $I > 2\sigma(I)$. Structure determined by direct methods, using *MITHRIL* (Gilmore, 1983); all non-H atoms were located in this way. Successive isotropic refinement cycles (on F) showed large temperature values for atoms C19, C23, C24 and O25, which suggests a certain disorder. However, a careful inspection of the Fourier map revealed no alternative peaks for the atoms in question. The positions of the H atoms associated with the disordered C atoms were calculated geometrically; the remaining H atoms were located from a difference map and included in the refinement with the same isotropic temperature factors as the atoms to which they are bonded. Several cycles of full-matrix, mixed least-squares refinement of all non-H atoms treated anisotropically, H atoms fixed, were performed; at this stage, an empirical weighting scheme (Martínez-Ripoll & Cano, 1975) was applied to give similar values of $w\Delta^2$ over ranges of $\sin\theta/\lambda$ and F_o. Final $R = 0·065$, $wR = 0·083$, $S = 8·5$; $(\Delta/\sigma)_{\max} = 0·1$; max. height in final difference Fourier synthesis 0·74 e Å^{-3}, min. height −0·41 e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Calculations carried out using *XRAY70* (Stewart, Kundell & Baldwin, 1970) and *PARST* (Nardelli, 1983).

Discussion. Final parameters are given in Table 1.† The identification of the atoms and a perspective molecular drawing of oxodipine are shown in Fig. 1. Bond lengths

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* Ethyl methyl 2,6-dimethyl-4-(2,3-methylenedioxyphenyl)-1,4-dihydro-3,5-pyridinedicarboxylate.

† Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43205 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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