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Acta Cryst. (1983). C**39**, 620–622

Structure of Diphenacyl Selenide, $C_{16}H_{14}O_2Se$

BY G. V. N. APPA RAO AND M. SESHASAYEE

Department of Physics, Indian Institute of Technology, Madras–600 036, *India*

AND G. ARAVAMUDAN AND S. SOWRIRAJAN

Department of Chemistry, Indian Institute of Technology, Madras–600 036, *India*

(*Received* 12 *July* 1982; *accepted* 1 *February* 1983)

Abstract. $M_r = 317{\cdot}2$, monoclinic, $P2_1/c$, $a = 5{\cdot}618$ (3), $b = 9{\cdot}963$ (5), $c = 25{\cdot}235$ (7) Å, $\beta = 95{\cdot}08$ (4)°, $V = 1406{\cdot}9$ Å^3, $Z = 4$, $D_x = 1{\cdot}50$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 3{\cdot}326$ mm^{-1}, $F(000) = 640$. Final $R = 0{\cdot}071$ for 1932 independent reflections. Selenium forms two primary Se–C bonds and one secondary Se–O intramolecular contact.

Introduction. Diphenacyl selenide (R_2Se) is a symmetric selenide often encountered as an intermediate in SeO_2 oxidation and related reactions. Such carbon-bonded Se molecules play important roles in the metabolism and toxicity of selenium.

Experimental. R_2Se prepared by reduction of dichlorodiphenacylselenium(IV) by thiourea in acetone medium. Slow evaporation of ethyl acetate solution containing R_2Se yielded colourless, needle-shaped crystals; Weissenberg and precession photographs gave preliminary unit-cell dimensions and symmetry information; systematic absences fixed the space group to be $P2_1/c$; cell parameters were obtained by least-squares refinement of θ values of 25 high-angle reflections; crystal dimensions 0·26 × 0·25 × 0·08 mm, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Cu $K\alpha$ radiation, $\omega/2\theta$ scan mode, $2 < \theta < 78°$; 3350 reflections collected, out of which 1932

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had $I > 3\sigma(I)$ and considered observed; no absorption correction was applied.

Three-dimensional Patterson maps gave the positions of the Se atoms. Refined selenium position parameters with isotropic temperature factors gave an R factor of 0·37. Selenium-phased Fourier maps were unable to develop the molecule further. At this stage, it was decided to try direct methods for solving the structure and hence the program *MULTAN* (Germain, Main & Woolfson, 1971) was used with $P2_1/c$ as the space group, but the E maps failed to reveal the structure. *MULTAN* was again tried with Pc as the space group (two molecules per asymmetric unit). 14 peaks from the resulting E map were chosen for the initial model and were subsequently developed into the full structure with the help of difference Fourier maps. The positions of the atoms agreed with the space group $P2_1/c$ and hence refinements were done in that space group.

Final difference Fourier maps were featureless and did not reveal positions of the H atoms, which were fixed geometrically; weighting scheme $w = 0{\cdot}01/[\sigma^2(F_o) + 0{\cdot}54|F_o|^2]$ employed in the minimization function $\sum w(|F_o| - |F_c|)^2$; final R and R_w values 0·071 and 0·076; atomic scattering factors for non-H atoms taken from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965); anomalous-dispersion correction factors of Cromer & Liberman (1970) used for non-H atoms.

Discussion. Fractional coordinates of the atoms are listed in Table 1.* The molecule of R_2Se is depicted in Fig. 1. Bond lengths and angles are shown in Table 2.

Selenium, in the present compound, can be regarded as in oxidation state +2. It forms two Se–C linkages [1·952 (6) and 1·950 (6) Å], the C–Se–C angle being 96·3 (3)°. These values agree with those reported for 2-dimethylaminoethyl selenobenzoate (Dexter, 1972), namely 1·945 (3) Å and 96·4 (3)°. The observed intramolecular Se···O distance of 2·874 (8) Å is longer than the covalent distance of 1·83 Å and significantly shorter than the van der Waals sum of 3·4 Å. Such Se···O distances in the range 2·6–3·1 Å have been reported in solid selenium oxide (McCullough, 1937), in $KH_3(SeO_3)_2$ (Hansen, Hazell & Rasmussen, 1969) and in $SbCl_5.SeOCl_2.N(CH_3)_4Cl.5SeOCl_2$ (Hermodsson, 1967*a,b*). Hence the central Se atom can be regarded to be three-coordinated to C(1), O(1) and C(9) and deviates by 0·429 (5) Å from the plane formed by them.

The oxygen–oxygen dipole interaction causes the two O atoms to stay apart, thereby producing the Se···O intramolecular short contact and the shortening of the distance between the phenyl carbon and the

* 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 38344 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional* (× 10^5 *for* Se, × 10^4 *for other atoms) and equivalent isotropic thermal parameters* (× 10^3) *for non-*H *atoms with e.s.d.'s in parentheses*

$U_{eq} = \frac{1}{3}\sum_i U_{ii}$.

	x	y	z	U_{eq}(Å^2)
Se	12335 (12)	44221 (8)	19340 (2)	65 (1)
O(1)	−1002 (10)	3247 (5)	2804 (2)	87 (4)
O(2)	−3123 (10)	5560 (5)	967 (2)	79 (4)
C(1)	−1316 (12)	5247 (6)	2299 (2)	59 (4)
C(2)	−1981 (11)	4339 (5)	2737 (2)	54 (3)
C(3)	−3795 (10)	4804 (5)	3079 (2)	50 (3)
C(4)	−4760 (14)	3875 (7)	3420 (3)	72 (4)
C(5)	−6535 (16)	4257 (9)	3740 (3)	86 (5)
C(6)	−7289 (16)	5580 (10)	3734 (3)	86 (6)
C(7)	−6380 (16)	6477 (8)	3403 (3)	85 (6)
C(8)	−4646 (14)	6115 (6)	3071 (3)	68 (4)
C(9)	906 (12)	5586 (6)	1311 (3)	63 (4)
C(10)	−1155 (11)	5077 (6)	945 (2)	55 (3)
C(11)	−792 (10)	3926 (6)	583 (2)	55 (3)
C(12)	−2608 (12)	3588 (7)	205 (3)	69 (4)
C(13)	−2415 (15)	2548 (9)	−161 (3)	83 (5)
C(14)	−304 (16)	1817 (8)	−120 (3)	91 (6)
C(15)	1510 (14)	2121 (8)	252 (3)	85 (5)
C(16)	1309 (11)	3182 (7)	612 (3)	69 (4)

Table 2. *Bond lengths* (Å) *and angles* (°)

Se–C(1)	1·952 (6)	Se–C(9)	1·950 (6)
C(1)–C(2)	1·500 (8)	C(9)–C(10)	1·503 (9)
C(2)–O(1)	1·225 (7)	C(10)–O(2)	1·212 (8)
C(2)–C(3)	1·468 (8)	C(10)–C(11)	1·493 (8)
C(3)–C(4)	1·405 (9)	C(11)–C(12)	1·375 (9)
C(4)–C(5)	1·391 (12)	C(12)–C(13)	1·399 (11)
C(5)–C(6)	1·385 (13)	C(13)–C(14)	1·389 (12)
C(6)–C(7)	1·353 (12)	C(14)–C(15)	1·357 (11)
C(7)–C(8)	1·388 (12)	C(15)–C(16)	1·406 (11)
C(3)–C(8)	1·391 (8)	C(11)–C(16)	1·391 (9)
Se···O(1)	2·874 (1)	Se···O(2)	3·491 (1)
C(1)–Se–C(9)	96·3 (3)		
Se–C(1)–C(2)	109·6 (4)	Se–C(9)–C(10)	107·8 (4)
C(1)–C(2)–O(1)	119·9 (6)	C(9)–C(10)–O(2)	119·7 (6)
C(1)–C(2)–C(3)	118·3 (5)	C(9)–C(10)–C(11)	119·7 (5)
O(1)–C(2)–C(3)	121·7 (5)	O(2)–C(10)–C(11)	120·5 (5)
C(2)–C(3)–C(4)	118·6 (5)	C(10)–C(11)–C(12)	118·7 (5)
C(2)–C(3)–C(8)	122·9 (5)	C(10)–C(11)–C(16)	122·6 (5)
C(8)–C(3)–C(4)	118·5 (6)	C(16)–C(11)–C(12)	118·7 (6)
C(3)–C(4)–C(5)	120·8 (6)	C(11)–C(12)–C(13)	122·9 (6)
C(4)–C(5)–C(6)	119·2 (8)	C(12)–C(13)–C(14)	117·1 (7)
C(6)–C(7)–C(8)	121·7 (8)	C(14)–C(15)–C(16)	121·1 (7)
C(5)–C(6)–C(7)	120·2 (8)	C(13)–C(14)–C(15)	121·2 (7)
C(3)–C(8)–C(7)	119·6 (6)	C(11)–C(16)–C(15)	118·9 (6)

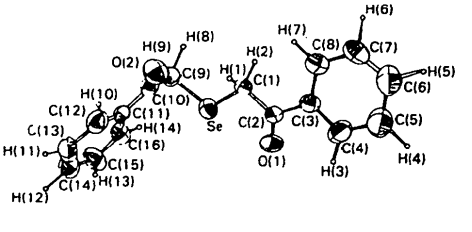

Fig. 1. *ORTEP* plot (Johnson, 1976) of the R_2Se molecule.

carbonyl carbon in the phenacyl moiety [1·468 (8) and 1·493 (8) Å are the observed C—C distances in both the ligands]. The C=O distances, however, remain the same in both ligands [1·225 (7) and 1·212 (8) Å], since the electron drift from C=O to the Se atom is probably compensated by the electron drift towards C=O from the phenyl ring.

The bond lengths and angles around the C atoms have normal values. The C=O bond distance in both ligands is close to the double-bond distance of 1·19 Å. The bond lengths in the phenyl rings range from 1·353 (12) to 1·406 (11) Å. The torsional angles in the rings show the planarity of the rings to be within ± 2·8°.

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Acta Cryst. (1983). C**39**, 622–624

Structure of (1*R**,13*R**,14*S**)-14-Hydroxy-6-methoxy-1,14-dimethyltetracyclo[11.3.1.0^{2,11}.0^{5,10}]heptadeca-2(11),5(10),6,8-tetraen-17-one, $C_{20}H_{24}O_3$

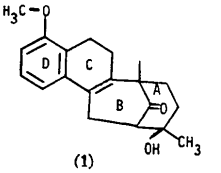
BY N. G. CHARLES, E. A. H. GRIFFITH, G. A. BONITZ, T. A. BRYSON AND E. L. AMMA†

Department of Chemistry, University of South Carolina, Columbia, South Carolina 29208, *USA*

(*Received* 9 *August* 1982; *accepted* 5 *January* 1983)

Abstract. $M_r = 312·4$, triclinic, $P\bar{1}$, $a = 10·710$ (6), $b = 10·905$ (2), $c = 7·609$ (3) Å, $\alpha = 91·70$ (2), $\beta = 93·14$ (4), $\gamma = 68·69$ (4)°, $V = 826·4$ (8) Å^3, $Z = 2$, $D_x = 1·26$, $D_m = 1·24$ (1) g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0·896$ cm^{-1}, $F(000) = 336$, $T = 291$ K. Final $R = 0·046$ with 1850 observed reflections. The structure consists of isolated molecules separated by ordinary van der Waals distances. The *A* ring exists in the chair conformation with the methyl group equatorial and the hydroxy group axial. The *B* ring approximates a sofa conformation. The carbon of the methoxy group is coplanar with the *D* ring.

Introduction. In comparing the activity and structure of natural steroids such as estrone and progesterone to those of bridged homosteroids a well-defined sterochemistry is essential. The subtle differences in these two groups of compounds are well documented in the literature (Lednicer & Mitscher, 1977). To understand better the conformational influences on activity of estrone derivatives (Johnson, David, Dehm, Highet, Warnoff, Wood & Jones, 1958) and the equivalent homologous compounds, one of us (TAB) has begun to investigate several fused and bridging homosteroid structures (Cargill, Bryson, Krueger, Kempf, McKenzie & Bordner, 1976). A classic synthon (1) used for preparing hydrochrysenone compounds (Johnson, Korst, Clement & Dutta, 1960) is an obvious point for initiating such a study since there is no stereochemistry in the *B,C*-ring junction and this compound has a bridging aldol *A* ring. A crystal-structure determination of (1) was considered essential to the futher synthetic development of this ring system.



(1)

Experimental. Title compound synthesized by known literature methods (Johnson *et al.*, 1960) and diffraction-quality crystals grown by slow evaporation of a secondary butanol solution over a period of a week; an approximately parallelepiped-shaped crystal of ~0·70 × 0·35 × 0·63 mm mounted in a glass capillary, faces ($\bar{2}02$), ($\bar{2}0\bar{2}$), ($10\bar{2}$), (102), ($0\bar{1}0$), (101); Enraf–Nonius CAD-4 diffractometer, graphite monochromator, $\theta = 6·1$°; 25 general reflections used in orientation matrix (checked every 24 h) and used for all parameter measurements; absorption corrections made, max. and min. transmission factors 0·983 and 0·957 (Frenz, 1980); variable scan speed with preliminary

† To whom correspondence should be addressed.

0108-2701/83/050622-03$01.50