Crystal Structure of 2-Se-(2-methyl-2-propenyl)-1-benzoic Acid

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$C_{11}H_{12}O_2Se$ is triclinic, $P\bar{1}$. Unit-cell dimensions at 293 K are $a = 5.8450(10)$, $b = 8.1490(10)$, $c = 11.4620(10)$ Å, $\alpha = 97.050(10)^\circ$, $\beta = 90.140(10)^\circ$, $\gamma = 90.120(10)^\circ$, $V = 541.81(12)$ Å³, $D_x = 1.564$ g/cm³, and $Z = 2$. The R value is 0.047 for 1388 observed reflections. The dihedral angle between the phenyl ring and the isobutenyl group is $72.3(2)^\circ$. There is an intermolecular hydrogen bond between two symmetry-related carbonyl groups with an O1...O2 distance of $2.669(6)$ Å. The molecules in the crystal are packed at normal van der Waals distances.

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Selenium is well recognized as an important "dietary" antioxidant.¹ It is recognized as being an essential component of the active sites of a number of enzymes, including the glutathione peroxidase selenoenzyme family, which scavenges hydroperoxides to prevent cellular damage.² Dietary selenium deficiency has been linked to diseases as diverse as cancer, heart disease, arthritis and AIDS.² Thus, the pharmacology, biology and biochemistry of selenium metabolism have become of considerable interest, which are spurring efforts to develop synthetic selenium-containing compounds as possible therapeutic agents.² We are currently working on the synthesis of a new series of agents containing selenium, such as 2-Se-(2-methyl-2-propenyl)-1-benzoic acid (Fig. 1). In this paper, we present the crystal structure of (1).

The title compound was prepared by a method described in the literature.³ A detailed description of the experimental

synthesis will be reported elsewhere. The obtained material was recrystallized from chloroform:isopropyl ether (1:1) to give a colorless crystal (m.p. 133 – 135°C); IR (1672 cm⁻¹, CO); NMR (1.93 ppm, C11-CH₃; 3.6 ppm, 8-CH₂; 5 ppm, 10-CH₂; 7.2 – 8.2

Table 1 Crystal and experimental data

Formula: $C_{11}H_{12}O_2Se$
Formula weight = 255.17
Crystal system: triclinic
Space group: $P\bar{1}$ $Z = 2$
$a = 5.8450(10)$ Å
$b = 8.1490(10)$ Å
$c = 11.4620(10)$ Å
$\alpha = 97.050(10)^\circ$
$\beta = 90.140(10)^\circ$
$\gamma = 90.120(10)^\circ$
$V = 541.81(12)$ Å ³
$D_x = 1.564$ g/cm ³
$D_m = 1.566$ g/cm ³ (measured by flotation)
$\mu(Mo K\alpha) = 3.436$ mm ⁻¹
$T = 293$ K
Colorless
$F(000) = 256$
$0.32 \times 0.20 \times 0.16$ mm
Radiation: Mo $K\alpha$
$2\theta/\theta$ scans with $2\theta_{max} = 50^\circ$
$R = 0.0468$ (on F^2)
$R_w = 0.088$ (on F^2)
$(\Delta\sigma)_{max} = 0.086$
$(\Delta\rho)_{max} = 0.318$ eÅ ⁻³
$(\Delta\rho)_{min} = -0.348$ eÅ ⁻³
No. of reflections measured = 1893
No. of reflections used = 1388
No. of parameters = 131
Goodness-of-fit = 1.026
Measurement: Siemens P4/PC diffractometer
Program system: SHELXL-Plus
Structure determination: direct methods (SHELXS-97)
Refinement: full matrix least-squares (SHELXL-97)

Fig. 1 Molecular structure of the title compound with atom labeling. Thermal ellipsoids are drawn at the 50% probability level.

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Table 2 Final atomic coordinates and equivalent isotropic temperature factors ($\text{\AA}^2$)

Atom	x	y	z	U_{eq}
Se1	0.39723(13)	0.51390(8)	0.73186(6)	0.0474(2)
O1	0.7754(8)	0.4992(5)	0.5884(4)	0.0539(11)
O2	0.9727(8)	0.2804(5)	0.5088(4)	0.0581(13)
C1	0.6630(10)	0.2311(6)	0.6293(5)	0.0396(13)
C2	0.4716(10)	0.2846(6)	0.6980(5)	0.0405(13)
C3	0.3341(11)	0.1638(7)	0.7397(6)	0.0521(16)
C4	0.3902(13)	-0.0004(8)	0.7191(6)	0.0574(17)
C5	0.5801(13)	-0.0537(7)	0.6551(6)	0.0605(18)
C6	0.7146(11)	0.0627(7)	0.6103(6)	0.0523(16)
C7	0.8046(10)	0.3497(7)	0.5747(5)	0.0416(13)
C8	0.1552(11)	0.5040(7)	0.8505(5)	0.0521(16)
C9	0.1231(10)	0.6747(8)	0.9114(6)	0.0496(15)
C10	-0.0523(13)	0.7642(10)	0.8879(7)	0.074(2)
C11	0.2978(14)	0.7361(10)	1.0012(7)	0.079(2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij}(a_i^* a_j^*)(a_i a_j).$$

ppm, H-aromatic; 11 ppm, -COOH).

The X-ray data for crystals of the title compound were collected by graphite-monochromatized Mo K_{α} radiation at 293 K. An analytical face-indexed absorption correction was applied. The structure was solved by direct methods and refined by full-matrix least-squares with anisotropic temperature factors for the non-hydrogen atoms. The hydrogen atoms bonded to the carbon atoms were assigned based on the expected bonding geometry. The hydrogen atom bonded to the hydroxyl group was found in a difference map and its coordinates refined in the final least-squares cycles. The final atomic parameters for hydrogen atoms, and observed and calculated structure factors may be obtained from the second author. The software used to prepare material for publication was PARST97.⁴ Table 1 summarizes the crystal and experimental data. The molecular structure is shown in Fig. 1.

The phenyl ring and the isobutenyl group at Se1 are planar within the experimental error, and the dihedral angle between them is $72.3(2)^{\circ}$. This conformation keeps the Se atom's unshared electron pairs in a more stable configuration with the rest of the molecules. The dihedral angle between the planes of

Table 3 Selected bond distances ($\text{\AA}$) and angles ($^{\circ}$)

Se1 - C2	1.912(5)	Se1 - C8	1.973(5)
O1 - C7	1.221(6)	O2 - C7	1.325(7)
C1 - C7	1.469(7)	C8 - C9	1.490(8)
C9 - C10	1.306(9)	C9 - C11	1.489(10)
C2 - Se1 - C8	100.6(2)	C6 - C1 - C7	119.7(5)
C2 - C1 - C7	120.8(5)	C3 - C2 - Se1	120.6(4)
C1 - C2 - Se1	121.6(4)	C5 - C6 - C1	121.7(6)
O1 - C7 - O2	121.9(5)	O1 - C7 - C1	124.2(5)
O2 - C7 - C1	113.9(5)	C9 - C8 - Se1	107.5(4)
C10 - C9 - C8	121.2(7)	C10 - C9 - C11	122.0(7)
C8 - C9 - C11	116.8(6)		

the carboxyl group at C1 and the isobutenyl moiety is $75.8(3)^{\circ}$. There is an intermolecular hydrogen bond between two symmetry-related carbonyl groups ($-x + 2, -y + 1, -z + 1$) with an O1...O2 distance of $2.669(6)\text{\AA}$. There is one intermolecular contact between C6...O2, $3.481(7)\text{\AA}$ ($-x + 2, -y, -z + 1$). Apart from the intermolecular hydrogen bond, a C...O contact, the packing in the crystal is entirely due to van der Waals forces. The final atomic coordinates and equivalent isotropic temperature factors are given in Table 2. Selected bond distances and angles are listed in Table 3.

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