

## Structure of Solidagenone

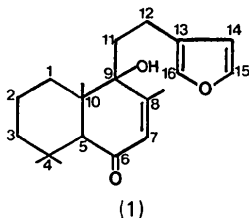
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**Abstract.**  $C_{20}H_{28}O_3$ ,  $M_r = 316.2$ , hexagonal,  $P6_1$ ,  $a = 13.535$  (2),  $c = 34.010$  (5) Å,  $V = 5396$  (3) Å<sup>3</sup>,  $Z = 12$ ,  $D_x = 1.2$  (1) Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 5.31$  mm<sup>-1</sup>,  $F(000) = 2028$ ,  $T = 294$  K,  $R = 0.050$  for 2257 observed reflections with  $I > 3\sigma(I)$ . There are two independent molecules in the asymmetric unit which are almost identical. The *A* rings in both molecules are slightly distorted chairs. The *B* rings have a sofa conformation. The two fused rings show a *cis* form of coupling. Despite the disorder both furan rings are planar. The hydroxyl groups are involved in hydrogen bonds with the carbonyl O atoms, with  $O2 \cdots O1 = 2.934$  (6),  $O2-H = 1.02$  (3),  $H \cdots O1 = 2.02$  (2) Å,  $O2-H \cdots O1 = 148$  (8)° and  $O2 \cdots O1 = 2.985$  (6),  $O2-H = 1.01$  (3),  $H \cdots O1 = 2.02$  (3) Å,  $O2-H \cdots O1 = 159$  (8)°.

**Experimental.** Hexagonal pyramidal crystals of solidagenone (1) 0.5 mm across were used to confirm photographically determined cell dimensions (13 reflections,  $26 < 2\theta < 49^\circ$ ) and for intensity data collection with monochromated Cu *K*α radiation on a Picker computer-controlled four-circle diffractometer; intensity data measured using  $\theta$ - $2\theta$  scans over the  $2\theta$  range 2–120°, scan width  $(1.2 + 0.18 \tan \theta)^\circ$  divided into 25 steps, counting time 2 s step<sup>-1</sup>.



Reflections with  $0 < h < 13$ ,  $0 < k < 13$ ,  $0 < l < 38$  were measured giving 2667 independent reflections, 2257 with  $I > 3\sigma(I)$ .  $R_{\text{int}} = 0.018$ . Reflections 330 and 037 monitored every 60 reflections (intensity variation 3%); data corrected for this variation, no correction for absorption, Lorentz and polarization corrections, atomic scattering factors from

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*SHELX76* (Sheldrick, 1976). The structure was solved with *MULTAN80* (Main *et al.*, 1980). An *E* map generated from the phase set with the highest combined figure of merit located a small fragment of the molecule. Three cycles of tangent refinement showed 35 of the 46 non-H atoms. The remaining non-H atoms were located from a difference Fourier synthesis. The structure was refined on *F* by the full-matrix least-squares method of *SHELX76*. The *z* coordinate of O1 was held constant to define the origin along the *z* axis. Anisotropic thermal parameters were used for non-H atoms. H atoms were included when bonded to an ordered C atom.

In the final refinement cycles the weighting scheme  $w = 5.8[\sigma^2(F_o) + 0.000039F_o^2]$  was applied. Final  $R = 0.050$ ,  $wR = 0.047$ ,  $S = 0.090$ , max. shift/e.s.d = 0.090 (disordered atoms), max. and min. heights in the final difference Fourier map 0.44 and  $-0.49$  e Å<sup>-3</sup> at the disorder regions.

The furan rings in both molecules show disorder in the O atoms and to a lesser extent in the C atoms. The flexibility of the linear chain which links the furan rings to the rest of the molecule results in their large anisotropic thermal motion. Attempts to refine the furan rings as rigid planar groups did not improve the refinement, nor did the generation of one molecule from the other through a pseudosymmetry operation. Final atomic parameters for non-H atoms are given in Table 1.† The bond lengths and angles are listed in Table 2. The structure of the molecule is shown in Fig. 1. The crystal packing of the molecules is shown in Fig. 2.

**Related literature.** Solidagenone is a diterpenoid isolated from the roots of *Solidago canadensis* L. The molecular formula was established by mass spectrometry and the chemical structure proposed from spectroscopic studies (Anthonson, McCabe, McCrindle, Murray & Young, 1970).

† Lists of structure factors, anisotropic thermal parameters for non-H atoms and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54768 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates ( $\times 10^4$ ) and equivalent isotropic temperature factors for non-H atoms with e.s.d.'s in parentheses

$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$B_{\text{eq}}$ ( $\text{\AA}^2$ )
<b>Molecule 1</b>				
C1	9021 (4)	5323 (4)	4244 (2)	5.08 (38)
C2	0256 (5)	6166 (4)	4373 (2)	6.72 (48)
C3	1116 (5)	6028 (4)	4127 (2)	6.12 (43)
C4	0983 (4)	4854 (4)	4143 (2)	5.04 (38)
C5	9724 (4)	3938 (4)	4058 (2)	4.02 (32)
C6	9430 (4)	2710 (4)	4076 (2)	4.01 (33)
C7	8260 (4)	1859 (4)	4017 (2)	4.32 (35)
C8	7386 (4)	2073 (4)	3998 (2)	4.22 (34)
C9	7612 (4)	3280 (4)	4051 (2)	4.55 (36)
C10	8756 (4)	4063 (4)	4270 (2)	3.96 (32)
C11	6619 (4)	3302 (5)	4258 (2)	5.80 (29)
C12	5639 (5)	3186 (5)	4012 (2)	7.74 (39)
C13	4710 (5)	3218 (5)	4247 (2)	7.67 (39)
C14	3726 (6)	3132 (6)	4062 (3)	10.48 (54)
C15	3122 (8)	3225 (8)	4380 (4)	10.23 (61)
C16	4687 (7)	3338 (7)	4638 (3)	10.79 (54)
C17	1434 (5)	4721 (5)	4542 (2)	6.51 (32)
C18	1749 (4)	4765 (5)	3825 (2)	5.66 (30)
C19	6190 (5)	1105 (5)	3929 (2)	6.94 (36)
C20	8638 (4)	3659 (5)	4698 (2)	5.42 (29)
O1	0152 (3)	2414 (3)	4124 (1)	4.91 (18)
O2	-2341 (3)	3691 (3)	3650 (1)	5.57 (19)
O3	3614 (6)	3333 (6)	4713 (3)	13.67 (52)
<b>Molecule 2</b>				
C1	8136 (4)	-2446 (4)	4037 (2)	4.75 (28)
C2	7072 (5)	-3527 (4)	3915 (2)	5.60 (29)
C3	6169 (5)	-3248 (4)	3793 (2)	5.51 (30)
C4	5838 (4)	-2687 (4)	4114 (2)	4.56 (27)
C5	6928 (4)	-1646 (4)	4283 (2)	3.71 (22)
C6	6758 (5)	-1018 (4)	4615 (2)	4.51 (27)
C7	7786 (5)	-0091 (4)	4798 (2)	4.72 (28)
C8	8839 (5)	0097 (4)	4746 (1)	4.36 (26)
C9	9067 (4)	-0618 (4)	4438 (2)	4.00 (24)
C10	7957 (4)	-1824 (4)	4384 (2)	4.04 (24)
C11	0091 (4)	-0752 (4)	4550 (2)	5.13 (27)
C12	1270 (4)	0189 (5)	4410 (2)	7.84 (38)
C13	2149 (4)	9821 (5)	4440 (2)	7.55 (39)
C14	2592 (7)	9643 (9)	4766 (4)	15.40 (77)
C15	3374 (9)	9399 (9)	4711 (5)	15.96 (89)
C16	2633 (6)	9574 (8)	4143 (3)	12.92 (68)
C17	5085 (5)	-3583 (4)	4424 (2)	6.46 (33)
C18	5101 (4)	-2252 (5)	3917 (2)	5.47 (30)
C19	9828 (6)	1050 (5)	4948 (2)	6.17 (31)
C20	7750 (4)	-2509 (4)	4767 (2)	4.89 (26)
O1	5813 (3)	-1188 (3)	4722 (1)	6.44 (23)
O2	9270 (3)	-0050 (3)	4064 (1)	4.08 (16)
O3	3376 (5)	9261 (6)	4285 (4)	16.40 (65)

Table 2 (cont.)

	Molecule 1	Molecule 2
C5—C10—C1	110.0 (4)	108.1 (4)
C10—C1—C2	111.3 (6)	113.9 (5)
C4—C5—C6	116.9 (5)	116.3 (4)
C5—C6—C7	117.3 (6)	117.1 (5)
C6—C7—C8	124.6 (5)	125.1 (6)
C7—C8—C9	119.9 (4)	119.8 (4)
C8—C9—C10	112.7 (5)	109.7 (4)
C9—C10—C5	107.8 (4)	109.0 (4)
C10—C5—C6	109.0 (4)	108.9 (4)
C9—C10—C1	108.9 (5)	109.0 (4)
C3—C4—C17	108.2 (3)	109.9 (5)
C3—C4—C18	108.9 (2)	106.8 (5)
C5—C4—C17	114.5 (2)	114.4 (5)
C5—C4—C18	108.5 (2)	108.5 (5)
C17—C4—C18	106.8 (4)	107.3 (5)
C5—C6—O1	123.2 (1)	123.7 (4)
C7—C6—O1	119.5 (5)	119.0 (6)
C7—C8—C19	119.7 (3)	121.3 (6)
C9—C8—C19	120.4 (3)	118.6 (5)
C8—C9—O2	104.2 (2)	107.9 (4)
C10—C9—O2	110.3 (1)	106.1 (4)
C8—C9—C11	111.7 (2)	112.2 (4)
C10—C9—C11	110.5 (2)	110.8 (5)
C9—C11—C12	118.6 (5)	116.8 (5)
C11—C12—C13	114.0 (6)	111.7 (6)
C9—C10—C20	108.6 (2)	108.5 (4)
C1—C10—C20	111.6 (2)	110.6 (5)
C5—C10—C20	109.8 (3)	111.6 (4)
C11—C9—O2	107.1 (3)	109.9 (4)

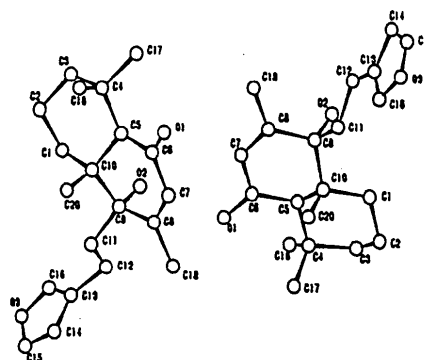


Fig. 1. Atomic numbering and molecular structure of solidagenone.

Table 2. Selected bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ )

	Molecule 1	Molecule 2
C1—C2	1.544 (7)	1.510 (6)
C2—C3	1.518 (9)	1.507 (10)
C3—C4	1.508 (9)	1.520 (9)
C4—C5	1.554 (6)	1.554 (6)
C5—C10	1.576 (8)	1.565 (8)
C10—C1	1.559 (8)	1.539 (8)
C5—C6	1.504 (8)	1.498 (8)
C6—C7	1.432 (6)	1.467 (7)
C7—C8	1.353 (10)	1.328 (10)
C8—C9	1.515 (8)	1.557 (8)
C9—C10	1.559 (7)	1.581 (5)
C4—C17	1.532 (7)	1.544 (8)
C4—C18	1.545 (7)	1.542 (10)
C6—O1	1.239 (5)	1.236 (8)
C9—C11	1.530 (6)	1.534 (9)
C11—C12	1.508 (9)	1.536 (7)
C12—C13	1.507 (11)	1.505 (11)
C16—C13	1.344 (12)	1.334 (14)
C9—O2	1.461 (4)	1.440 (6)
C10—C20	1.537 (6)	1.542 (8)
<b>Angles (<math>^\circ</math>)</b>		
C1—C2—C3	111.5 (5)	109.4 (5)
C2—C3—C4	114.5 (5)	114.0 (5)
C3—C4—C5	109.8 (5)	109.6 (4)
C4—C5—C10	117.9 (4)	117.2 (5)

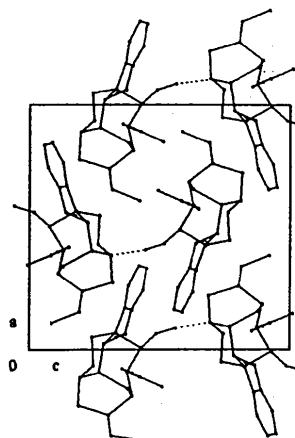


Fig. 2. Packing of the molecules in the crystal structure.

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## Structure of 1,2,4,5-Tetrakis(trifluoromethyl)benzene

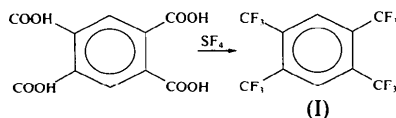
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**Abstract.**  $C_{10}H_2F_{12}$ ,  $M_r = 350.11$ , orthorhombic, *Pbca*,  $a = 9.203$  (3),  $b = 9.811$  (3),  $c = 12.705$  (6) Å,  $V = 1147.0$  (7) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 2.03$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 2.470$  cm<sup>-1</sup>,  $F(000) = 680$ ,  $T = 173$  K,  $R = 0.0513$  for 828 reflections [ $F_o \geq 6\sigma(F_o)$ ]. The molecule lies around an inversion center at  $0, \frac{1}{2}, 0$ . Steric crowding caused by adjacent CF<sub>3</sub> groups is reflected in the exocyclic bond angles at C1 and C2. The angles for C4—C1—C2 and C5—C2—C1 are 123.5 (3) and 123.4 (3)°, respectively, while those for C5—C2—C3 and C4—C1—C3' are 117.8 (3) and 117.2 (3)°, respectively. The C—F bond lengths are normal and average 1.330 (2) Å.

**Experimental.** The title compound (I) was synthesized by the action of SF<sub>4</sub> on benzene tetracarboxylic acid (Hasek, Smith & Engelhardt, 1960). In a typical reaction, 5 g of the tetraacid was placed into an 80 cm<sup>3</sup>, stainless-steel high-pressure cylinder which



was then fitted with a high-pressure valve and evacuated. A 50% stoichiometric excess of SF<sub>4</sub> was condensed into the cylinder at 77 K and subsequently heated to 393 K for 12 h. After heating, the cylinder was allowed to cool to room temperature and the volatile components were removed *in vacuo*. The solid residue was placed in a sublimator. Large well formed colorless crystals of (I) were obtained by sublimation onto a 'cold finger' at 273 K at approxi-

mately 10 mm Hg in a matter of a few hours. The data crystal was a colourless needle of approximate dimensions 0.27 × 0.35 × 0.83 mm. The data were collected on a Nicolet R3 diffractometer using a graphite monochromator and a Nicolet LT2 low-temperature delivery system. Lattice parameters were obtained from the least-squares refinement of 32 reflections with  $18.4 < 2\theta < 22.0^\circ$ . The data were collected using the  $\omega$ -scan technique, with a  $1.2^\circ$   $\omega$  scan at  $8\text{--}12^\circ \text{min}^{-1}$  and a  $2\theta$  range of  $4.0\text{--}50.0^\circ$  ( $h = 10 \rightarrow 10$ ,  $k = 0 \rightarrow 11$ ,  $l = 0 \rightarrow 15$ ). A total of 2248 reflections was collected, of which 1009 were unique, with  $R_{\text{int}} = 0.013$ . Three reflections ( $23\bar{3}$ ,  $4\bar{2}2$ ,  $4\bar{2}\bar{2}$ ) were remeasured every 97 reflections to monitor instrument and crystal stability. A smoothed curve of the intensities of these check reflections was used to scale the data. The scaling factor range was 0.948–1.00. The data were also corrected for Lp effects but not for absorption. Data reduction and decay correction were performed using *SHELXTL-Plus* (Sheldrick, 1989). Reflections having  $F_o < 6\sigma(F_o)$  were considered unobserved (411 reflections). The structure was solved by direct methods and refined by full-matrix least squares (Sheldrick, 1989) on  $F$ . In all, 105 parameters were refined. The non-H atoms were refined with anisotropic thermal parameters. The H-atom position was obtained from a  $\Delta F$  map and refined with an isotropic thermal parameter. The function  $\sum w(|F_o| - |F_c|)^2$  was minimized, where  $w = 1/[\sigma(F_o)]^2$  and  $\sigma(F_o) = 0.5kI^{-1/2}\{[\sigma(I)]^2 + (0.02I)^2\}^{1/2}$ ; the intensity,  $I$ , is given by  $(I_{\text{peak}} - I_{\text{background}})(\text{scan rate})$ , 0.02 is a factor to downweight intense reflections and to account for instrument instability, and  $k$  is the correction for Lp effects and decay.  $\sigma(I)$  was