

1980). Atomic parameters are given in Table 1;\* the bond distances, bond angles, and relevant torsion angles are presented in Table 2. Atomic numbering is given in Fig. 1, and the packing diagram, with inter- and intramolecular hydrogen bonding, is shown in Fig. 2.

**Related literature.** The chroman ring in this structure has the sofa conformation. The O(1), C(3) and C(4) atoms are coplanar with the aromatic nucleus. The planar phenyl ring is in the equatorial position, C(4') shows the maximum deviation of 0.008 (2) Å. The dihedral angle between this plane and that of the chroman ring is 94.52 (4)°. The twist of the exocyclic phenyl ring relative to the rest of the molecule as characterized by the C(3)—C(2)—C(1')—C(2') torsion angle is 125.9 (2)°, which is significantly higher than the range of -50 to 110° reported for

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52775 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

other flavone structures (Cody, 1988). The C(2)—C(1') bond distance of 1.497 (3) Å (Table 2) is shorter than the assigned 1.536 (9) Å in 5-hydroxy-7-methoxyflavanone (Shoja, 1989), possibly due to stabilization of the negative charge on the exocyclic O atom of the chroman ring system (Rossi, Rickles & Halpin, 1986).

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*Acta Cryst.* (1990). **C46**, 1971–1973

## Methyl (4*R*,9*R*,10*S*)-7-Isopropyl-4-methyl-5,8-dioxo-1,6-tricyclo[8.3.0.0<sup>4,9</sup>]tridecadiene-9-carboxylate

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(Received 5 February 1990; accepted 6 April 1990)

**Abstract.** C<sub>19</sub>H<sub>24</sub>O<sub>4</sub>, *M<sub>r</sub>* = 316.40, orthorhombic, *Pbca*, *a* = 12.444 (1), *b* = 14.615 (3), *c* = 18.797 (5) Å, *V* = 3419 (2) Å<sup>3</sup>, *Z* = 8, *D<sub>x</sub>* = 1.229 g cm<sup>-3</sup>, λ(Mo *K*α) = 0.71073 Å, μ = 0.79 cm<sup>-1</sup>, *F*(000) = 1360, *T* = 298 K, *R* = 0.050 for 1060 observed reflections. The five-membered ring is in an envelope conformation, the cyclohexene ring in a half-chair conformation and the cyclohexenedione in an envelope conformation with C(8)—O(1) out of the plane. The molecule adopts an overall hemispherical conformation.

**Experimental.** The data collection and refinement parameters for compound (1) are summarized in Table 1.

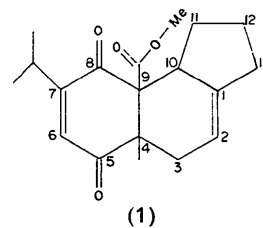


Table 1. Crystallographic summary for (1)

(a) Data collection <sup>i,ii</sup>	
Mode	$\omega$ -2 $\theta$
Scan rate (° min <sup>-1</sup> )	1.9-6.7
$\theta$ range (°)	3-25
Range of $hkl$	$0 \leq h \leq 14, 0 \leq k \leq 17, 0 \leq l \leq 22$
Total reflections measured	2340
Unique reflections	2147
$R_{int}$	0.02
Crystal dimensions (mm)	0.13 × 0.18 × 0.30
(b) Structure refinement <sup>iii</sup>	
Reflections used [ $I > 3\sigma(I)$ ]	1063
No. of variables	209
$R, wR$	0.050, 0.044
Max. shift/e.s.d.	0.004
Max., min. density in final difference map (e Å <sup>-3</sup> )	0.21, -0.16

Notes: (i) Unit-cell parameters by least-squares refinement of the setting angles of 25 reflections with  $9 < \theta < 20^\circ$ . (ii) Enraf-Nonius CAD-4 diffractometer with a graphite monochromator. Two standard reflections (604 and 218) measured every hour showed no significant variation. (iii) Function minimized was  $\sum w(|F_o| - |F_c|)^2$ , where  $w^{-1} = [\sigma^2(F_o) + 0.0001F_o^2]$ .

Table 2. Final fractional atomic coordinates and equivalent isotropic temperature factors (Å<sup>2</sup>) with e.s.d.'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> <sub>iso</sub> <sup>*</sup>
O(1)	0.3594 (3)	0.6191 (2)	0.7689 (2)	4.6 (1)
O(2)	0.4138 (4)	0.3598 (2)	0.9538 (2)	6.5 (2)
O(3)	0.6351 (3)	0.4866 (2)	0.8854 (1)	4.5 (1)
O(4)	0.6039 (3)	0.6186 (2)	0.8302 (2)	4.4 (1)
C(1)	0.4209 (5)	0.4451 (3)	0.6848 (2)	4.0 (2)
C(2)	0.3407 (5)	0.4017 (3)	0.7150 (3)	4.7 (3)
C(3)	0.3321 (4)	0.3873 (3)	0.7949 (3)	4.8 (2)
C(4)	0.4396 (4)	0.4085 (3)	0.8337 (2)	3.7 (2)
C(5)	0.4124 (4)	0.4222 (3)	0.9116 (3)	4.2 (2)
C(6)	0.3761 (4)	0.5134 (3)	0.9352 (2)	3.9 (2)
C(7)	0.3654 (4)	0.5853 (3)	0.8916 (2)	3.2 (2)
C(8)	0.3997 (4)	0.5732 (3)	0.8155 (2)	3.1 (2)
C(9)	0.4850 (4)	0.5007 (3)	0.8029 (2)	2.7 (2)
C(10)	0.5153 (4)	0.4848 (3)	0.7246 (2)	3.3 (2)
C(11)	0.5522 (4)	0.5655 (3)	0.6783 (2)	4.4 (2)
C(12)	0.5299 (6)	0.5292 (4)	0.6022 (3)	7.2 (3)
C(13)	0.4323 (5)	0.4700 (3)	0.6061 (3)	5.5 (2)
C(14)	0.5148 (4)	0.3255 (3)	0.8254 (3)	5.1 (2)
C(15)	0.5835 (4)	0.5313 (3)	0.8449 (2)	3.0 (2)
C(16)	0.6936 (4)	0.6611 (3)	0.8676 (2)	5.4 (2)
C(17)	0.3205 (4)	0.6770 (3)	0.9122 (2)	3.9 (2)
C(18)	0.4059 (5)	0.7504 (3)	0.9078 (3)	6.2 (2)
C(19)	0.2669 (5)	0.6770 (3)	0.9852 (3)	6.2 (2)

$$*B_{iso} = \frac{1}{3} \sum_i \sum_j B_{ij}(\mathbf{a}_i \cdot \mathbf{a}_j).$$

The structure was solved using standard direct methods and difference-Fourier techniques. In the final cycles of full-matrix least-squares refinement all non-H atoms were treated anisotropically. H atoms were included at positions found in a difference synthesis, all with a common isotropic temperature factor that refined to  $U = 0.12 \text{ \AA}^2$ . Scattering factors for non-H atoms were taken from Cromer & Mann (1968) with corrections for anomalous dispersion from Cromer & Liberman (1970); for H atoms from Stewart, Davidson & Simpson (1965). Programs

Table 3. Interatomic bond distances (Å) and angles (°)

O(1)—C(8)	1.212 (6)	C(5)—C(6)	1.476 (7)
O(2)—C(5)	1.209 (6)	C(6)—C(7)	1.339 (6)
O(3)—C(15)	1.191 (5)	C(7)—C(8)	1.503 (6)
O(4)—C(15)	1.330 (5)	C(7)—C(17)	1.503 (6)
O(4)—C(16)	1.458 (6)	C(8)—C(9)	1.518 (6)
C(1)—C(2)	1.312 (8)	C(9)—C(10)	1.537 (6)
C(1)—C(10)	1.509 (7)	C(9)—C(15)	1.525 (6)
C(1)—C(13)	1.530 (7)	C(10)—C(11)	1.536 (6)
C(2)—C(3)	1.520 (7)	C(11)—C(12)	1.551 (7)
C(3)—C(4)	1.555 (7)	C(12)—C(13)	1.493 (9)
C(4)—C(5)	1.516 (7)	C(17)—C(18)	1.512 (7)
C(4)—C(9)	1.572 (6)	C(17)—C(19)	1.526 (7)
C(4)—C(14)	1.540 (6)		
C(15)—O(4)—C(16)	117.0 (3)	O(1)—C(8)—C(9)	124.2 (4)
C(2)—C(1)—C(10)	124.3 (5)	C(7)—C(8)—C(9)	115.4 (4)
C(2)—C(1)—C(13)	127.2 (5)	C(4)—C(9)—C(8)	106.8 (4)
C(10)—C(1)—C(13)	108.4 (4)	C(4)—C(9)—C(10)	108.1 (4)
C(1)—C(2)—C(3)	123.2 (5)	C(4)—C(9)—C(15)	110.5 (4)
C(2)—C(3)—C(4)	112.0 (4)	C(8)—C(9)—C(10)	115.2 (4)
C(3)—C(4)—C(5)	106.7 (4)	C(8)—C(9)—C(15)	106.1 (4)
C(3)—C(4)—C(9)	107.9 (4)	C(10)—C(9)—C(15)	110.1 (4)
C(3)—C(4)—C(14)	108.6 (4)	C(1)—C(10)—C(9)	110.0 (4)
C(5)—C(4)—C(9)	108.8 (4)	C(1)—C(10)—C(11)	104.3 (4)
C(5)—C(4)—C(14)	109.7 (4)	C(9)—C(10)—C(11)	120.0 (4)
C(9)—C(4)—C(14)	114.8 (4)	C(10)—C(11)—C(12)	101.9 (4)
O(2)—C(5)—C(4)	122.1 (4)	C(11)—C(12)—C(13)	107.4 (5)
O(2)—C(5)—C(6)	119.2 (5)	C(1)—C(13)—C(12)	105.1 (4)
C(4)—C(5)—C(6)	118.6 (4)	O(3)—C(15)—O(4)	123.8 (4)
C(5)—C(6)—C(7)	123.7 (4)	O(3)—C(15)—C(9)	127.1 (4)
C(6)—C(7)—C(8)	117.5 (4)	O(4)—C(15)—C(9)	109.1 (3)
C(6)—C(7)—C(17)	125.4 (4)	C(7)—C(17)—C(18)	110.9 (4)
C(8)—C(7)—C(17)	117.1 (4)	C(7)—C(17)—C(19)	113.2 (4)
O(1)—C(8)—C(7)	120.3 (4)	C(18)—C(17)—C(19)	110.9 (4)

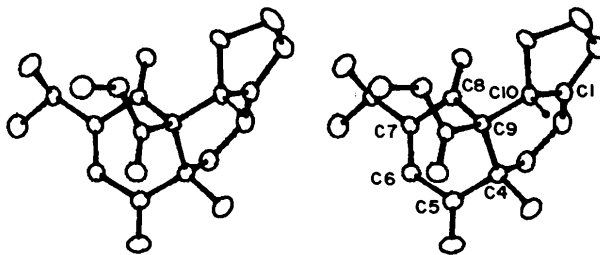


Fig. 1. Stereoscopic projection of the molecule.

used: *SHELX76* (Sheldrick, 1976), *ORTEP* (Johnson, 1965).

Atomic coordinates are listed in Table 2,\* bond lengths and bond angles in Table 3. Shortest intermolecular distances: O(1)⋯C(16) ( $x - 0.5, y, 1.5 - z$ ) = 3.349 (6), O(3)⋯C(6) ( $1 - x, 1 - y, 2 - z$ ) = 3.375 (5) Å. Fig. 1 is a stereoscopic projection of the molecule.

**Related literature.** The title compound was prepared by a Diels-Alder reaction (Catani & Brocksom, 1989) leading to an all-*cis* relationship between the

\* Lists of H-atom positions, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52961 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

substituents at C(4), C(9) and C(10) and therefore a hemispherical conformation for the entire molecule. This result allows prediction of the steric course of subsequent reactions (Trost, 1983) and prediction of the orientation of similar Diels–Alder reactions (Woodward & Hoffmann, 1970).

This work has received partial support from CAPES, CNPq, FAPESP and FINEP. One of us (JZS) thanks the Departamento de Química Inorganica, Facultad de Farmacia, Universidad de Santiago de Compostela, Spain, for the use of the X-ray diffraction equipment.

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*Acta Cryst.* (1990). **C46**, 1973–1975

## Structure of 9,9-Dimethyl-8-oxatricyclo[4.3.0.0<sup>1,3</sup>]nonan-6-ol

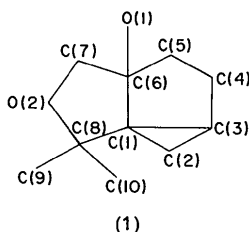
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(Received 22 February 1990; accepted 6 April 1990)

**Abstract.** C<sub>10</sub>H<sub>16</sub>O<sub>2</sub>,  $M_r = 168.24$ , monoclinic,  $P2_1$ ,  $a = 8.390$  (2),  $b = 10.052$  (3),  $c = 12.163$  (2) Å,  $\beta = 108.65$  (2)°,  $V = 971.9$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.150$  g cm<sup>-3</sup>,  $\lambda = 0.71073$  Å,  $\mu(\text{Mo } K\alpha) = 0.73$  cm<sup>-1</sup>,  $F(000) = 368$ ,  $T = 298$  K,  $R = 0.045$  for 1333 observed reflections. There are two independent molecules in the asymmetric unit which are essentially identical. They are hydrogen bonded to each other and to neighbouring symmetry-related ones, giving rise to infinite helical chains of molecules along the direction of the twofold screw axis.

**Experimental.** The synthesis of compound (1) has been reported previously (Marsaioli, Nurnberg, Sarragiotto & Castellano, 1989).



The data collection and refinement parameters are summarized in Table 1. The structure was solved using standard direct-methods and difference-

Fourier techniques. In the final cycles of least-squares refinement, all non-H atoms were treated anisotropically. The origin in the polar axis was fixed by splitting the normal matrix into two blocks, one for each of the independent molecules. H atoms were included as fixed contributors at positions found in difference syntheses, slightly modified on stereochemical grounds, all with a common isotropic temperature factor that refined to  $U = 0.070$  Å<sup>2</sup>. Scattering factors for non-H atoms were taken from Cromer & Mann (1968) with corrections for anomalous dispersion from Cromer & Liberman (1970), for the H atoms from Stewart, Davidson & Simpson (1965). Programs used were *SHELX76* (Sheldrick, 1976) and *ORTEP* (Johnson, 1965). All calculations were performed on a VAX 11/780 computer.

Atomic coordinates are listed in Table 2\* and bond lengths and angles in Table 3. Fig. 1 is a stereoscopic projection of the unit-cell contents.

**Related literature.** Crystals were obtained as previously described (Marsaioli, Nurnberg, Sarragiotto

\* 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 52968 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.