7-Hydroxy-4'-methoxyisoflavanquinone (Claussequinone), $C_{16}H_{14}O_5$

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Abstract. $M_r = 286.2$, tetragonal, $P4_1$, $a = 6.740$ (1), $c = 29.411$ (2) Å, $V = 1336$ (4) Å^3, $Z = 4$, $D_m = 1.41$ (flotation), $D_x = 1.42$ Mg m^{-3}, $\lambda(Cu\,K\alpha) = 1.54018$ Å, $\mu(Cu\,K\alpha) = 0.54$ mm^{-1}, $F(000) = 600$, room temperature, final $R = 0.064$ for 1237 observations. The quinone ring is linked in an equatorial position to the chroman ring. The extended structure shows a helical arrangement of the molecules along the c axis.

Introduction. The title compound is one of the components of the extract from the heartwood of *Cyclobium claussene* Benth., a tree originating from the region of Pedro Leopoldo, State of Minas Gerais, Brazil. The main component of its benzenic extract is an orange pigment that was named claussequinone in view of its quinonic character. The proton NMR and mass spectroscopy analysis of this compound disclosed its isoflavanquinonic skeleton and pattern of substituents. ORD studies of this compound and of a synthetic compound allowed the determination of the configuration shown in Fig. 1 (Gottlieb, Oliveira, Gonçalves, Oliveira & Pereira, 1975). This X-ray diffraction study was carried out to elucidate the precise molecular conformation of the heterocyclic ring, and to look for any hydrogen bond present in the crystalline structure.

Experimental. Red-orange single crystals, tetragonal-pyramidal habit, obtained by slow evaporation of ethanol–chloroform solution, single-crystal dimension about 0.4 mm. Cell dimensions from setting angles of 25 high-angle reflections, CAD–4 automatic diffractometer, room temperature, $\theta \leq 70°$, θ–2θ scan mode, graphite-monochromatized Cu $K\alpha$ radiation, Lp corrections, absorption and extinction corrections ignored. 1537 measured unique reflexions, 1237 observed $[F^2 > 2\sigma(F^2)]$; structure solved by direct methods (Germain, Main & Woolfson, 1971) and refined by

full-matrix least-squares calculations by minimization of $\sum w(k|F_o| - |F_c|)^2$, where $w = [\sigma(F_o)^2 + (0.05F_o)^2]^{-1}$ for observed and $w = 0$ for unobserved reflexions, until all the atomic-parameter shifts were smaller than their standard deviations. The final unweighted R factor, omitting unobserved reflexions, was 0.064 (including them 0.067).* Anisotropic temperature factors were assigned to all non-hydrogen atoms. Neutral complex scattering factors were used (*International Tables for X-ray Crystallography*, 1974). Final positional parameters for the H atoms were calculated assuming a C—H bond length of 1.0 Å and the expected geometry, with isotropic temperature factors assumed to be 6.0 Å^2.

Discussion. Table 1 gives the atomic parameters for the C and O atoms. The atomic notation, and interatomic distances and bond angles are shown in Fig. 2. There are no unusual bond lengths in the structure. The bond distances and angles in the chroman ring agree well with those found in two adducts of 4-(p-hydroxyphenyl)-2,2,4-trimethylchroman with ethanol and chloroform (Flippen, Karle & Karle, 1970). The benzene and the quinone rings are planar [average distances of atoms from the planes are 0.011 (4) and 0.019 (8) Å respectively] and the dihedral angle between the best least-squares plane through these rings is 26.9°. A distorted half-chair conformation of the dihydropyran ring can be observed from the deviations of the atoms O(4), C(2), C(3) and C(4) from the best least-squares plane through the benzene ring [0.027 (3), 0.439 (4), −0.350 (3) and 0.052 (4) Å respectively] and from the torsion angle of the bond O(4)—C(2)—C(3)—C(4), 66.1°. The quinone ring is linked to the chroman ring by C(3) in an equatorial position. Table 2 shows some torsion angles to characterize the conformation of the molecule.

The packing of the molecules, shown in Fig. 3, is stabilized by a system of hydrogen bonds which generates a helical arrangement along the c axis. The
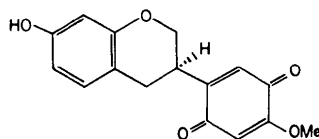

Fig. 1. Molecular structure as disclosed by proton NMR and ORD.

* Lists of structure factors, anisotropic thermal parameters, and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38343 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

molecules are linked head to tail, by a hydrogen bond of 2·822 (4) Å between O(1) of the methoxy group of one and O(5) of the hydroxyl group of another which is related to the former by the symmetry operation $(x, y, \frac{1}{2} + z)$. The O(5)—H and O(1)···H distances are 1·017 (3) and 1·805 (4) Å respectively and the angle formed is 178·47 (4)°.

All computer calculations were performed using the Enraf–Nonius structure determination package.

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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *for non-hydrogen atoms with e.s.d.'s in parentheses*

The $B_{eq}(\text{Å}^2)$ values are the equivalent isotropic temperature factors (Hamilton, 1959).

	x	y	z	B_{eq}
O(1)	4908 (4)	2121 (4)	6795 (1)	3·4
O(2)	−887 (5)	4521 (4)	6075 (1)	4·6
O(3)	3567 (4)	−1462 (4)	6630 (1)	3·9
O(4)	−4224 (4)	1714 (4)	5143 (1)	3·9
O(5)	−9632 (5)	−2006 (5)	4603 (1)	4·9
C(1)	5627 (7)	4083 (6)	6894 (2)	4·0
C(2)	−2258 (6)	1665 (5)	5323 (1)	3·2
C(3)	−2282 (5)	789 (5)	5804 (1)	2·7
C(4)	−2919 (6)	−1390 (5)	5767 (1)	3·1
C(5)	−5919 (6)	−3262 (6)	5470 (1)	3·3
C(6)	−7561 (6)	−3475 (6)	5182 (2)	3·7
C(7)	−8019 (6)	−1927 (6)	4894 (2)	3·4
C(8)	−6944 (6)	−183 (6)	4893 (2)	3·5
C(9)	−5267 (6)	−34 (5)	5177 (1)	3·0
C(10)	−4727 (5)	−1557 (5)	5468 (1)	2·9
C(1')	−345 (5)	1061 (5)	6050 (1)	2·5
C(2')	237 (6)	3128 (5)	6171 (2)	3·0
C(3')	2072 (6)	3520 (5)	6421 (1)	3·1
C(4')	3191 (5)	1993 (5)	6560 (1)	2·7
C(5')	2609 (5)	−100 (5)	6468 (1)	2·7
C(6')	838 (6)	−440 (5)	6190 (1)	3·0

Table 2. *Torsion angles* (°)

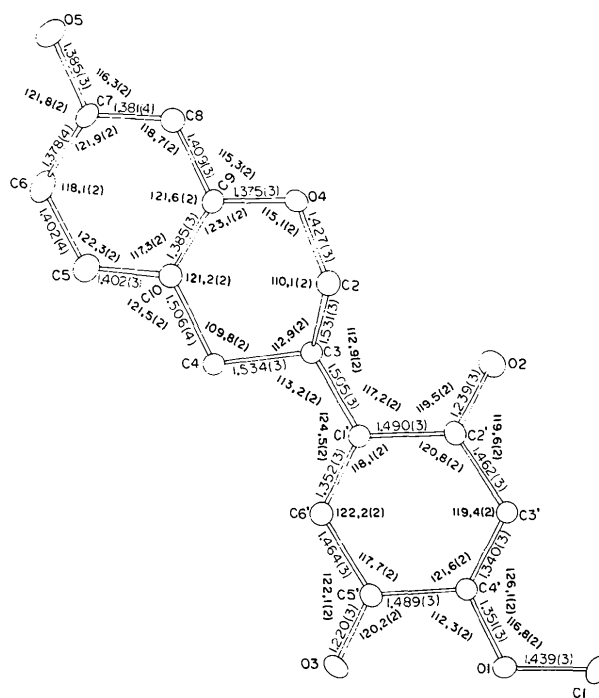|	
C(9)—O(4)—C(2)—C(3)	50·2 (4)
O(4)—C(2)—C(3)—C(4)	−66·1 (4)
C(2)—C(3)—C(4)—C(10)	47·6 (4)
C(3')—C(4')—O(1)—C(1)	1·9 (4)
C(5')—C(4')—O(1)—C(1)	−178·8 (4)
C(2)—C(3)—C(1')—C(2')	−67·2 (4)
C(4)—C(3)—C(1')—C(6')	−6·0 (4)
C(8)—C(9)—O(4)—C(2)	162·6 (4)
C(5)—C(10)—C(4)—C(3)	163·0 (4)

Fig. 2. *ORTEP* (Johnson, 1965) representation of the molecule showing atom numbering, bond lengths (Å) and bond angles (°).
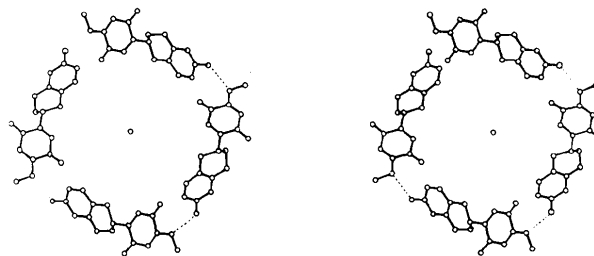

Fig. 3. Stereoscopic view of the helical arrangement of the molecules along the *c* axis.

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