snoutanone, the nonan-9-one derivative, has been reported (Mehta, Srikrishna & Suri, 1980). The structures of 5-bromohomocubane-4-carboxyclic acid (Smits, Beurskens, Klunder & Van der Loop, 1986) and homocubane-9-carboxylic acid (Dauben, Schallhorn & Whalen, 1971) have been reported. The molecular mechanics program *MM*2 was used for estimating the heat of formation and strain energies (Allinger & Yu, 1980).

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5-Hydroxy-3,7,4′-trimethoxyflavone

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Abstract. 5-Hydroxy-3,7-dimethoxy-2-(4-methoxyphenyl)chroman-4-one, $C_{18}H_{16}O_6$, $M_r = 328{\cdot}32$, triclinic, $P\overline{1}$, $a = 5{\cdot}2678\,(14)$, $b = 11{\cdot}2895\,(6)$, $c = 12{\cdot}704$ Å, $\alpha = 96{\cdot}29\,(1)$, $\beta = 99{\cdot}62\,(2)$, $\gamma = 94{\cdot}61\,(1)°$, $V = 736{\cdot}60\,(2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}480\,(1)$ g cm^{-3}, monochromatized Mo $K\alpha$ ($\lambda = 0{\cdot}71073$ Å), $\mu = 1{\cdot}045$ cm^{-1}, $F(000) = 688$, $T = 110$ K, final $R = 0{\cdot}037$ for 4284 unique reflections. The 4′-methoxyphenyl ring forms a torsion angle of 27·1 (2)° with the benzopyranone ring. The compound forms a strong intramolecular hydrogen bond.

Experimental. The petroleum ether extract of the leaves of the previously uninvestigated *Haplopappus sonorensis* Compositae yielded the yellow flavonoid which was recrystallized from acetone. Since the positions of the substituents in this biologically active compound cold not easily be determined using spectroscopic methods, a crystal structure investigation was undertaken.

The diffraction data were collected on an Enraf–Nonius CAD-4 diffractometer using $\omega/2\theta$ scans. The crystal was cooled down to 110 ± 0·8 K in a stream of N_2 gas. The cell dimensions were determined from a least-squares fit of 25 reflections, well distributed in reciprocal space, with θ values in the range 16–20°. The maximum scan time was 60 s. Three reflections were measured every 2·8 h to check for decay of the crystal which, however, did not appear to take place. Crystallographic data are given in Table 1. No correction was made for absorption.

The structure was solved using *SHELXS*86 (Sheldrick, 1985), and refined with *SDP* (Enraf–Nonius, 1981). Atomic scattering factors were taken from Cromer & Waber (1974), except that for hydrogen which was taken from Stewart, Davidson & Simpson

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Table 1. *Experimental data*

Crystal description	Yellow, tabular
Dimensions (mm)	0·10 × 0·35 × 0·50
Corrections	Lorentz–polarization
Maximum 2θ (°)	60·0
hkl range	h 0→10 k −15→15 l −17→17
No. of refl. measured	4716
No. of refl. measured once	3852
Reflection averaging	$R_{\text{int}}(I)$ = 1·9%
No. of independent refl.	4284
Reflections included	2196 with $F_o^2 > 3{\cdot}0\sigma(F_o^2)$
Minimization function	$\sum w(\lvert F_o\rvert - \lvert F_c\rvert)^2$
H atoms	Located and refined isotropically
Parameters refined	281
Unweighted agreement factor, *R*	0·037
Weighted agreement factor, *wR*	0·043
Goodness of fit, *S*	1·33
w	$[\sigma^2(F) + 0{\cdot}005F^2]^{-1}$
High peak in final diff. map. (e Å^{-3})	0·30 (3)
Lowest peak in final diff. map (e Å^{-3})	−0·29 (3)
Final Δ/σ	<0·01

Table 2. *Positional and displacement parameters* (Å^2)

	x	y	z	B_{eq}*
O(1)	0·4722 (2)	0·4052 (1)	0·7855 (1)	1·13 (2)
O(7′)	1·3293 (3)	0·2250 (1)	1·1063 (1)	1·34 (3)
O(11)	0·3260 (3)	0·0828 (1)	0·7264 (1)	1·17 (2)
O(13)	−0·0834 (3)	0·1657 (1)	0·5954 (1)	1·31 (3)
O(14)	−0·3354 (3)	0·3436 (1)	0·5388 (1)	1·49 (3)
O(15)	0·0550 (3)	0·7360 (1)	0·6917 (1)	1·49 (3)
C(1′)	0·7115 (3)	0·2634 (2)	0·8655 (1)	0·99 (3)
C(2)	0·4911 (3)	0·2848 (2)	0·7840 (2)	1·00 (3)
C(2′)	0·7912 (4)	0·3485 (2)	0·9570 (2)	1·17 (3)
C(3)	0·3142 (4)	0·2030 (2)	0·7188 (1)	1·02 (3)
C(3′)	0·9962 (4)	0·3325 (2)	1·0352 (2)	1·25 (3)
C(4)	0·0896 (3)	0·2404 (2)	0·6522 (1)	1·04 (3)
C(4′)	1·1295 (4)	0·2315 (2)	1·0239 (2)	1·06 (3)
C(5)	−0·1404 (3)	0·4171 (2)	0·6027 (1)	1·14 (3)
C(5′)	1·0560 (4)	0·1467 (2)	0·9335 (2)	1·08 (3)
C(6)	−0·1540 (3)	0·5395 (2)	0·6115 (2)	1·15 (3)
C(6′)	0·8485 (4)	0·1634 (2)	0·8556 (1)	1·02 (3)
C(7)	0·0486 (4)	0·6145 (2)	0·6772 (2)	1·12 (3)
C(8)	0·2619 (4)	0·5697 (2)	0·7343 (2)	1·19 (3)
C(8′)	1·4850 (4)	0·1272 (2)	1·0960 (2)	1·38 (4)
C(9)	0·2666 (3)	0·4472 (2)	0·7245 (1)	1·00 (3)
C(10)	0·0728 (3)	0·3675 (2)	0·6587 (1)	0·96 (3)
C(12)	0·3643 (4)	0·0134 (2)	0·6286 (2)	1·38 (4)
C(16)	−0·1688 (4)	0·7891 (2)	0·6449 (2)	1·55 (4)
H(2′1)	0·697 (4)	0·418 (2)	0·966 (2)	1·7 (5)
H(3′1)	1·049 (4)	0·390 (2)	1·097 (2)	1·3 (4)
H(5′1)	1·150 (4)	0·076 (2)	0·926 (2)	1·3 (4)
H(61)	−0·290 (4)	0·570 (2)	0·574 (2)	1·6 (5)
H(6′1)	0·806 (4)	0·107 (2)	0·795 (2)	1·2 (4)
H(81)	0·401 (4)	0·622 (2)	0·783 (2)	2·1 (5)
H(8′1)	1·558 (4)	0·126 (2)	1·028 (2)	1·5 (4)
H(8′2)	1·620 (4)	0·142 (2)	1·159 (2)	1·4 (4)
H(8′3)	1·382 (4)	0·051 (2)	1·095 (2)	1·6 (4)
H(121)	0·229 (4)	0·023 (2)	0·570 (2)	1·1 (4)
H(122)	0·534 (4)	0·041 (2)	0·613 (2)	2·2 (5)
H(123)	0·357 (4)	−0·069 (2)	0·643 (2)	1·6 (4)
H(141)	−0·295 (5)	0·266 (2)	0·545 (2)	3·6 (6)
H(161)	−0·130 (4)	0·873 (2)	0·673 (2)	1·7 (5)
H(162)	−0·199 (4)	0·773 (2)	0·567 (2)	1·6 (5)
H(163)	−0·326 (4)	0·756 (2)	0·674 (2)	2·4 (5)

* Non-H atoms were refined anisotropic. Anisotropically refined displacement parameters are given in the isotropic equivalent form defined as: $B_{\text{eq}} = \frac{4}{3}(a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + 2ab(\cos\gamma)\beta_{12} + 2ac(\cos\beta)\beta_{13} + 2bc(\cos\alpha)\beta_{23})$.

(1965). The atomic parameters derived are listed in Table 2.*

Table 3. *Selected bond distances* (Å), *bond angles* (°) *and torsion angles* (°)

O(1)—C(2)	1·368 (2)	C(2)—C(3)	1·359 (2)
O(1)—C(9)	1·372 (2)	C(2′)—C(3′)	1·378 (3)
O(7′)—C(4′)	1·367 (2)	C(3)—C(4)	1·455 (3)
O(7′)—C(8′)	1·435 (2)	C(3′)—C(4′)	1·394 (3)
O(11)—C(3)	1·377 (2)	C(4)—C(10)	1·439 (3)
O(11)—C(12)	1·448 (2)	C(4′)—C(5′)	1·391 (2)
O(13)—C(4)	1·259 (2)	C(5)—C(6)	1·383 (3)
O(14)—C(5)	1·356 (2)	C(5)—C(10)	1·415 (3)
O(15)—C(7)	1·361 (2)	C(5′)—C(6′)	1·386 (2)
O(15)—C(16)	1·435 (2)	C(6)—C(7)	1·398 (2)
C(1′)—C(2)	1·475 (2)	C(7)—C(8)	1·395 (3)
C(1′)—C(2′)	1·406 (2)	C(8)—C(9)	1·377 (3)
C(1′)—C(6′)	1·394 (3)	C(9)—C(10)	1·398 (2)

C(2)—O(1)—C(9)	120·9 (1)	O(7′)—C(4′)—C(5′)	125·0 (2)
C(4′)—O(7′)—C(8′)	117·6 (1)	C(3′)—C(4′)—C(5′)	120·0 (2)
C(3)—O(11)—C(12)	114·1 (1)	O(14)—C(5)—C(6)	119·1 (2)
C(7)—O(15)—C(16)	118·1 (1)	O(14)—C(5)—C(10)	119·7 (2)
C(2)—C(1′)—C(2′)	118·8 (2)	C(6)—C(5)—C(10)	121·3 (2)
C(2)—C(1′)—C(6′)	123·2 (2)	C(4′)—C(5′)—C(6′)	119·5 (2)
C(2′)—C(1′)—C(6′)	118·0 (2)	C(5)—C(6)—C(7)	118·6 (2)
O(1)—C(2)—C(1′)	110·1 (1)	C(1′)—C(6′)—C(5′)	121·6 (2)
O(1)—C(2)—C(3)	121·4 (2)	O(15)—C(7)—C(6)	123·5 (2)
C(1′)—C(2)—C(3)	128·4 (2)	O(15)—C(7)—C(8)	114·4 (1)
C(1′)—C(2′)—C(3′)	121·0 (2)	C(6)—C(7)—C(8)	122·2 (2)
O(11)—C(3)—C(2)	119·8 (2)	C(7)—C(8)—C(9)	117·5 (2)
O(11)—C(3)—C(4)	118·7 (1)	O(1)—C(9)—C(8)	116·3 (1)
C(2)—C(3)—C(4)	120·9 (2)	O(1)—C(9)—C(10)	120·5 (2)
C(2′)—C(3′)—C(4′)	120·0 (2)	C(8)—C(9)—C(10)	123·3 (2)
O(13)—C(4)—C(3)	121·9 (2)	C(4)—C(10)—C(5)	122·3 (1)
O(13)—C(4)—C(10)	122·2 (2)	C(4)—C(10)—C(9)	120·4 (2)
C(3)—C(4)—C(10)	115·8 (1)	C(5)—C(10)—C(9)	117·3 (2)
O(7′)—C(4′)—C(3′)	115·0 (1)		

O(13)—O(14)	2·595 (2)
O(14)—H(141)	0·93 (3)
O(13)—H(141)	1·75 (3)

O(14)—H(141)—O(13)	151 (2)

O(1)—C(2)—C(3)—C(4)	−3·4 (3)	C(16)—O(15)—C(7)—C(6)	5·8 (3)
O(1)—C(2)—C(1′)—C(2′)	27·1 (2)	O(13)—C(4)—C(10)—C(5)	0·7 (3)
C(8′)—O(7′)—C(4′)—C(3′)	−175·9 (2)	H(141)—O(14)—C(5)—C(6)	−178 (2)
C(12)—O(11)—C(3)—C(2)	−119·2 (2)		

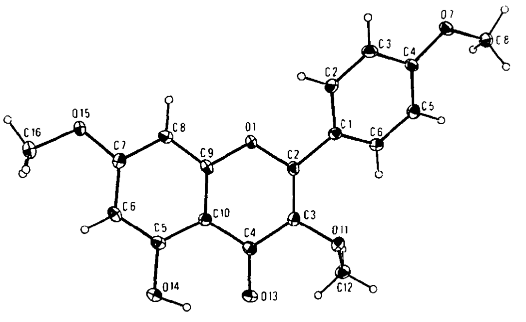

Fig. 1. *ORTEP* drawing of 5-hydroxy-3,7,4′-trimethoxyflavone. Thermal ellipsoids are drawn at the 50% probability level.

Selected bond lengths, angles and torsion angles are listed in Table 3. Fig. 1 shows an *ORTEP* (Johnson, 1976) drawing of the molecule and the atomic numbering scheme.

Related literature. *Haplopappus sonorensis* is used in traditional medicine in Mexico as a remedy against, for example, skin ulcers, toothache, tetanus and wounds, and the isolated flavonoid is probably responsible for the registered effects.

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* Lists of anisotropic displacement parameters, bond distances and angles involving H atoms, and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52109 (47 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related structures: 5,7,4′-trimethoxyflavone (Mariezcurrena, 1978), 3-hydroxyflavone (Etter, Urbanczyk-Lipkowska, Baer & Barbara, 1986), 5,4′-dihydroxy-3,6,7,8-tetramethoxyflavone (Vijayalakshmi, Rajan, Srinivasan & Ramachandran, 1986) and 5-hydroxy-4,7′-dimethoxyflavanone (Miles, Main & Nicholson, 1989).

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Structure of *rac*-3β-Hydroxy-18-nor-5α-pregn-13(17)-en-20-one

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Abstract. $C_{20}H_{30}O_2$, $M_r = 302{\cdot}46$, orthorhombic, $Fdd2$, $a = 50{\cdot}270\ (9)$, $b = 18{\cdot}018\ (3)$, $c = 7{\cdot}573\ (1)$ Å, $V = 6859{\cdot}6$ Å^3, $Z = 16$, $D_x = 1{\cdot}172$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 5{\cdot}339$ cm^{-1}, $F(000) = 2656$, $T = 298$ K, $R = 0{\cdot}042$ for 1838 unique observed reflections with $I > 2\sigma(I)$. The title compound, a racemic mixture, was synthesized from achiral starting materials. The C20 carbonyl is observed in an unusual conformation, *trans* to the C16—C17 bond. O3*B* and O20 form an intermolecular hydrogen bond: O···O = 2·86 Å, O—H···O = 177·9°, H···O = 2·04 Å.

Experimental. A thin rectangular plate was grown from ethanol. Crystal size 0·80 × 0·30 × 0·08 mm. Enraf–Nonius CAD-4 diffractometer, cell dimensions and Laue symmetry from 25 centered reflections ($50 < 2\theta < 70°$) checked with oscillation photographs, Cu $K\alpha$ radiation, Ni-filtered, no monochromator, scan width variable, scan speed from 0·625 to 4° min^{-1} in 2θ, scan width ($0{\cdot}80 + 0{\cdot}20 \times \tan\theta$), $2\theta_{\max} = 120°$, $-2 \le h \le 56$, $0 \le k \le 20$, $0 \le l \le 8$, 1900 independent reflections measured using a θ–2θ scan mode, 1838 with $I > 2\sigma(I)$. Four standard reflections (1,11,1, 35,1,1, 19,5,3, 715) were measured every 250 reflections and varied in intensity by ≤ 5% during the data collection.

Direct method using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) revealed positions of all non-H atoms. The positional and anisotropic displacement parameters of all non-H atoms were refined by full-matrix least squares on F using the 1838 reflections for which $I > 2\sigma(I)$. The hydrogen positions were located in a difference map and refined with isotropic temperature parameters. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Final $R = 0{\cdot}042$, $wR = 0{\cdot}055$, $S = 2{\cdot}227$ for observed reflections and $R = 0{\cdot}043$ for all data, $w = 1/\sigma^2$, $(\Delta/\sigma)_{\max} = 0{\cdot}27$. Weighting scheme based on estimates of experimental errors from counting statistics. Final difference map showed maximum positive and negative peaks of +0·10 and −0·04 e Å^{-3}. No corrections for absorption or extinction were made.

Atomic parameters are listed in Table 1. Distances, angles and selected torsion angles are listed in Table 2.* A view of of the molecule showing the atomic numbering and molecular conformation is given in Fig. 1. Fig. 2 shows a stereoview of the molecular packing. The enantiomer chosen for the

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* Lists of structure factors, anisotropic displacement parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52124 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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