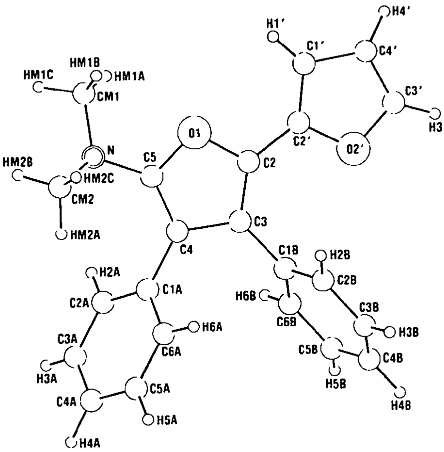

Fig. 2. Structure (II) with atom numbering.

Related literature. Details of the syntheses, reactions, and mechanisms are given by Yamashita, Toy, Watt & Muchmore (1988). The only other related compound reported that has a long C—C distance (1·58 Å) is a tetracyclone–dimethyl phosphonate adduct (Iball, Kaye & Miller, 1974). Another bifuran structure was published by Still & Romero (1986).

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6-Methoxy-2-(2,3,4-trimethoxyphenyl)-4*H*-1-benzopyran-4-one (6,2′,3′,4′-Tetramethoxyflavone)

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Abstract. $C_{19}H_{18}O_6$, $M_r = 342{\cdot}35$, monoclinic, $P2_1/a$, $a = 22{\cdot}341\,(9)$, $b = 16{\cdot}489\,(9)$, $c = 4{\cdot}477\,(1)$ Å, $\beta = 91{\cdot}35\,(3)°$, $V = 1649\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}38$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 8{\cdot}71$ cm^{-1}, $F(000) = 720$, $T = 291$ K, $R = 0{\cdot}064$ for 2560 observed reflections. The three rings are planar within experimental error (maximum deviation from mean planes less than 0·02 Å). The dihedral angle between the mean planes of rings *A* (O1—C2—C3—C4—C9—C10) and *B* (C5—C6—C7—C8—C9—C10) is 2 (1)°, and that between rings *A* and *C* (C1′—C2′—C3′—C4′—C5′—C6′) is 45 (1)°. The two methoxy groups bound to C2′ and C3′ have an out-of-plane conformation due to steric inhibition: C3′—C2′—O14—C15 = − 108·7 (6), C4′—C3′—O16—C17 = − 83·2 (6)°. The two other methoxy groups are only slightly out of plane with C5′—C4′—O18—C19 = 6·6 (6) and C7—C6—O12—C13 = 12·2 (6)°.

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Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}(Å^2)
O1	0·1235 (1)	0·0577 (1)	0·1218 (3)	3·71 (3)
C2	0·1535 (1)	0·1167 (1)	0·2733 (5)	3·56 (3)
C3	0·2042 (1)	0·1018 (1)	0·4354 (6)	4·25 (4)
C4	0·2300 (1)	0·0221 (1)	0·4543 (6)	4·38 (4)
C5	0·2135 (1)	−0·1224 (1)	0·3035 (5)	3·87 (4)
C6	0·1796 (1)	−0·1811 (1)	0·1563 (5)	3·62 (3)
C7	0·1274 (1)	−0·1587 (1)	−0·0033 (5)	3·88 (4)
C8	0·1100 (1)	−0·0784 (1)	−0·0103 (5)	3·86 (4)
C9	0·1440 (1)	−0·0204 (1)	0·1382 (4)	3·32 (3)
C10	0·1962 (1)	−0·0415 (1)	0·2973 (5)	3·54 (3)
C1′	0·1268 (1)	0·1971 (1)	0·2322 (5)	3·56 (3)
C2′	0·0653 (1)	0·2122 (1)	0·2531 (4)	3·17 (3)
C3′	0·0430 (1)	0·2899 (1)	0·2124 (4)	3·22 (3)
C4′	0·0816 (1)	0·3537 (1)	0·1406 (5)	3·82 (4)
C5′	0·1421 (1)	0·3405 (1)	0·1266 (7)	4·62 (4)
C6′	0·1641 (1)	0·2625 (1)	0·1712 (6)	4·65 (4)
O11	0·2777 (1)	0·0078 (1)	0·5925 (6)	7·15 (5)
O12	0·1995 (1)	−0·2585 (1)	0·1816 (4)	4·65 (3)
C13	0·1727 (1)	−0·3188 (1)	−0·0083 (6)	4·61 (4)
O14	0·0250 (1)	0·1517 (1)	0·3003 (3)	3·74 (3)
C15	0·0236 (2)	0·1190 (2)	0·5869 (6)	6·44 (6)
O16	−0·0174 (1)	0·3042 (1)	0·2268 (3)	3·74 (3)
C17	−0·0355 (1)	0·3367 (2)	0·5030 (5)	6·11 (6)
O18	0·0544 (1)	0·4265 (1)	0·0961 (4)	4·66 (3)
C19	0·0910 (1)	0·4921 (2)	−0·0016 (8)	5·37 (5)

Table 2. *Bond distances* (Å) *and angles* (°)

C2—O1	1·355 (2)	C9—O1	1·368 (2)
C3—C2	1·353 (3)	C1′—C2	1·464 (3)
C4—C3	1·437 (3)	C10—C4	1·462 (3)
O11—C4	1·243 (3)	C6—C5	1·385 (3)
C10—C5	1·390 (3)	C7—C6	1·402 (3)
O12—C6	1·356 (2)	C8—C7	1·381 (3)
C9—C8	1·382 (3)	C10—C9	1·396 (3)
C2′—C1′	1·401 (3)	C6′—C1′	1·393 (3)
C3′—C2′	1·385 (3)	O14—C2′	1·364 (2)
C4′—C3′	1·402 (3)	O16—C3′	1·371 (2)
C5′—C4′	1·373 (3)	O18—C4′	1·358 (2)
C6′—C5′	1·389 (3)	C13—O12	1·430 (3)
C15—O14	1·393 (3)	C17—O16	1·416 (3)
C19—O18	1·430 (3)		

C9—O1—C2	119·2 (2)	C3—C2—O1	122·4 (2)
C1′—C2—O1	113·1 (2)	C1′—C2—C3	124·4 (2)
C4—C3—C2	121·8 (2)	C10—C4—C3	115·2 (2)
O11—C4—C3	122·8 (2)	O11—C4—C10	122·0 (2)
C10—C5—C6	120·8 (2)	C7—C6—C5	119·9 (2)
O12—C6—C5	116·3 (2)	O12—C6—C7	123·8 (2)
C8—C7—C6	119·6 (2)	C9—C8—C7	120·1 (2)
C8—C9—O1	116·4 (2)	C10—C9—O1	122·5 (2)
C10—C9—C8	121·1 (2)	C5—C10—C4	122·6 (2)
C9—C10—C4	118·8 (2)	C9—C10—C5	118·5 (2)
C2′—C1′—C2	123·4 (2)	C6′—C1′—C2	118·8 (2)
C6′—C1′—C2′	117·8 (2)	C3′—C2′—C1′	120·5 (2)
O14—C2′—C1′	122·1 (2)	O14—C2′—C3′	117·4 (2)
C4′—C3′—C2′	120·1 (2)	O16—C3′—C2′	120·3 (2)
O16—C3′—C4′	119·5 (2)	C5′—C4′—C3′	120·3 (2)
O18—C4′—C3′	114·9 (2)	O18—C4′—C5′	124·8 (2)
C6′—C5′—C4′	119·0 (2)	C5′—C6′—C1′	122·3 (2)
C13—O12—C6	118·3 (2)	C15—O14—C2′	117·1 (2)
C17—O16—C3′	114·1 (2)	C19—O18—C4′	117·2 (2)

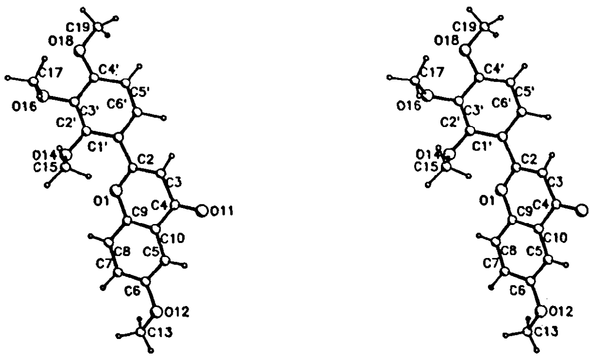

Fig. 1. Stereoscopic view of the molecule.

Experimental. The title compound (m.p. 398–399 K) was synthesized from 2,3,4-trimethoxybenzoic acid and 2,5-dihydroxyacetophenone (Gaydou & Bianchini, 1978). Good quality crystals were obtained by slow evaporation from an ethanol solution. D_m not measured. Parallelepiped crystal with approximate dimensions 0·20 × 0·24 × 0·40 mm. Lattice parameters refined using 15 reflections in the range $3 \leq 2\theta \leq 30°$. Huber four-circle diffractometer, graphite-monochromated Cu $K\alpha$ radiation. 2991 $h, k, \pm l$ measured reflections with $(\sin\theta)/\lambda \leq 0{\cdot}60$ Å^{-1}; $0 \leq h \leq 26$, $0 \leq k \leq 19$, $-5 \leq l \leq 5$, 2560 with $I \geq 2{\cdot}5\sigma(I)$. Standard reflection ($\bar{4}10$) checked every 50 reflections: no significant deviation. Structure solved by *SHELXS*86 (Sheldrick, 1985). H atoms from difference Fourier synthesis except those of the methoxy groups C15 and C17 calculated with C—H = 1·08 Å and H—C—H = 109·5°. Anisotropic least-squares refinement (*SHELX*76, Sheldrick, 1976) using F; H isotropic with common refined temperature factor. $w = 1/(\sigma^2 + 0{\cdot}11975F^2)$, $R = 0{\cdot}064$, $wR = 0{\cdot}079$ for 2560 observed reflections. Final maximum $\Delta/\sigma = 0{\cdot}30$ (z of atom C13). $s = 0{\cdot}35$. Maximum and minimum heights in final difference Fourier synthesis = 0·33 and −0·40 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The atomic parameters are given in Table 1.* Fig. 1 is a stereoscopic view of the molecule, showing the numbering of the atoms (program *PLUTO*, Motherwell & Clegg, 1978). Bond distances and angles are given in Table 2.

Related literature. A recent review indicates that this molecule has never previously been synthesized (Iinuma & Mizuno, 1989). This study forms part of our work on polymethoxylated flavones. As the biochemical activity of flavonoids is conformation dependent (Cody, 1988), we have undertaken an analysis of the effects of steric hindrance on the orientation of the phenyl ring towards the benzo-

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* 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 52583 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

pyrone moiety. In this context, we have already determined several structures of flavones (Wallet, Gaydou, Fadlane & Baldy, 1988; Wallet, Gaydou & Baldy, 1989). The observed bond lengths are similar to the values reported for 5,6-benzoflavone (Rossi, Cantrell, Farber, Dyott, Carrell & Glusker, 1980) and 5-hydroxy-6,7,8,3′,4′,5′-hexamethoxyflavone (Vijayalakshmi, Rajan & Srinivasan, 1987), particularly the C2—C1′ bond linking the phenyl ring and the γ-benzopyrone moiety at 1·460 (5) and 1·468 (5) Å respectively.

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Structure of Racemic 4-Isopropenyl-1-cyclohexene-1-carbaldehyde Oxime (Perillartine)

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Abstract. $C_{10}H_{15}NO$, M_r = 165·23, triclinic, $P\bar{1}$, a = 7·3217 (6), b = 8·1899 (6), c = 8·8402 (8) Å, α = 104·551 (1), β = 96·215 (7), γ = 104·591 (1)°, V = 488·14 (7) Å^3, Z = 2, D_x = 1·124 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 0·7 cm^{-1}, F(000) = 180, T = 298 K, R = 0·049 for 1275 unique observed diffractometer data [$I \geq 2·5\sigma(I)$]. A partial substitutional disorder of enantiomers was observed. The molecules form O—H···N-type cyclic hydrogen-bonded dimers across inversion centers. A systematic search for the so-called AH—B moieties which are thought to be responsible for the sweet taste revealed a CH—O moiety with a C···O distance of 2·22 Å as the only candidate.

Experimental. The title compound has an intensely sweet taste (Beets, 1978; van der Heijden, van der Wel & Peer, 1985) and a structure determination was undertaken to elucidate the molecular conformation in order to identify the origin of the sweet taste in terms of the so-called AH—B moiety (Shallenberger & Acree, 1967).

The title compound was recrystallized by sitting-drop vapor diffusion (McPherson, 1976) using 2-propanol as solvent and water as precipitant. Two sets of data were collected (November 1985 and August 1989) on a CAD-4F diffractometer at ambient temperature for two transparent, colorless crystals (0·2 × 0·3 × 0·3 mm, 0·4 × 0·2 × 0·2 mm), mounted on a glass fiber. Collection of data set 1: 876 reflections were scanned [h: −8, 0; $h = 2n$; k: −9, 9; l: −10, 10; $\theta \leq$ 24·99°; $\omega/2\theta$-scan mode; $\Delta\omega$ = (0·60 + 0·35tanθ)°; Zr-filtered Mo $K\alpha$ radiation]. During the measurement three reference reflections ($\bar{2}00$, 010, 002) showed fluctuations of 3%, and negligible decay during 35 h of X-ray exposure time. The data were corrected for Lp but not for absorption. $\sigma^2(I) = \sigma^2_{cs}(I) + (pI)^2$ (McCandlish, Stout & Andrews, 1975) with p = 0·02. Collection of data set 2: 3243 reflections were scanned [h: −9, 0; k: −10, 10; l: −11, 11; $\theta \leq$ 28·47°; $\omega/2\theta$-scan mode; $\Delta\omega$ = (0·65 + 0·35tanθ)°; Zr-filtered Mo $K\alpha$ radiation]. During the measurement three reference reflections (012, $\bar{2}04$, $\bar{3}30$) showed fluctuations of 4%, and a decay of about 20% during the 43 h of X-ray exposure time, for which a linear correction was

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