for trivalent phosphorus in 2-[$Ph_2P(CH_2)_3N$=CH]C_6H_4OH the comparable P—C(phenyl) bonds are longer at 1.828 (7) and 1.842 (6) Å (Banbery, Hussain, Hamor, Jones & McCleverty, 1990). The difference between the C—P—C angles (mean 106.4°) and the O—P—C angles [mean 112.4 (5)°] is presumably a result of the repulsive effect of the short electron-rich P—O double bond. The phenyl C—C bonds adjacent to the P—C bond tend to be longer than those away from the centre of the molecule. A similar, but smaller effect was noted by Brock *et al.* (1985) and was considered to be a genuine molecular phenomenon, rather than an artefact caused by molecular libration. Our results would, however, indicate that thermal libration is probably the prime cause for the apparent bond-length variation.

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5,7-Dihydroxy-2-(2,3,4-trimethoxyphenyl)-4*H*-1-benzopyran-4-one (5,7-Dihydroxy-2′,3′,4′-trimethoxyflavone)

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Abstract. $C_{18}H_{16}O_7$, $M_r = 344.32$, orthorhombic, $Pna2_1$, $a = 7.768$ (1), $b = 26.621$ (3), $c = 7.467$ (1) Å, $V = 1544.1$ (6) Å^3, $Z = 4$, $D_x = 1.481$ g cm^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 9.26$ cm^{-1}, $F(000) = 720$, $T = 291$ K, $R = 0.044$ for 1551 observed reflections. The molecule is not planar, the dihedral angle between the phenyl ring and the γ-benzopyrone portion is O(1)—C(2)—C(1′)—C(6′) = −34.5 (5)°. There is an intramolecular hydrogen bond between the carbonyl O atom and the O atom of the 5-hydroxy group, O(4)···O(3) = 2.586 Å. This carbonyl O atom can form a second intermolecular hydrogen bond with the O atom of the 7-hydroxy group O(2)···O(4) = 2.759 (5) Å. The two *ortho*-substituted methoxy groups have an out-of-plane conformation C(3′)—C(2′)—O(5)—C(15) = 70.6 (5)°, C(4′)—C(3′)—O(6)—C(14) = 73.4 (5)°. The third methoxy group is in the plane of the phenyl ring, C(5′)—C(4′)—O(7)—C(13) = 4.8 (6)°.

Experimental. The title compound (m.p. 428 K) was synthesized from 2,3,4-trimethoxybenzoic acid and 2,4,6-trihydroxyacetophenone (Gaydou & Bianchini, 1978). Good-quality crystals were obtained by slow evaporation from ethanol solution. D_m not measured. Parallelepiped crystal with approximate dimensions 0.2 × 0.3 × 0.3 mm. Lattice parameters refined using 25 reflections in the range $25.35 \leq \theta \leq 33.55°$. Enraf–Nonius CAD-4 diffractometer, Ni-filtered Cu $K\alpha$ radiation, $\omega/2\theta$ scan. 3329 hkl measured reflections with $\sin\theta/\lambda \leq 0.61$ Å^{-1}, $0 \leq h \leq 9$, $0 \leq k \leq 32$, $0 \leq l \leq 9$ and $I \geq 3\sigma(I)$. Five reflections checked every 300 reflections, no significant deviation. Structure solved by

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10^2$) *with e.s.d.'s in parentheses for the title compound*

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

	x	y	z	B_{eq}
C(8)	3549 (4)	2341 (1)	3451	429 (8)
O(1)	2983 (3)	1633 (1)	1674 (6)	420 (6)
C(7)	3494 (4)	2860 (1)	3677 (7)	438 (8)
C(6)	2817 (5)	3175 (1)	2375 (8)	469 (9)
C(10)	2184 (4)	2446 (1)	526 (7)	391 (7)
C(9)	2893 (4)	2149 (1)	1865 (7)	394 (7)
C(4′)	3405 (4)	−163 (1)	68 (7)	413 (7)
O(7)	3636 (3)	−669 (1)	−112 (6)	496 (6)
C(5)	2151 (4)	2973 (1)	821 (7)	440 (8)
C(3)	1714 (4)	1689 (1)	−1209 (7)	443 (8)
O(2)	4098 (4)	3078 (1)	5168 (6)	578 (7)
O(4)	852 (3)	2479 (1)	−2305 (6)	522 (6)
O(3)	1469 (4)	3274 (1)	−424 (7)	563 (7)
C(5′)	4520 (4)	162 (1)	969 (7)	421 (8)
C(2′)	1612 (4)	540 (1)	−762 (7)	402 (7)
C(4)	1531 (4)	2225 (1)	−1078 (7)	417 (8)
O(6)	908 (3)	−278 (1)	−1801 (6)	521 (6)
C(6′)	4172 (4)	674 (1)	989 (7)	409 (7)
C(3′)	1930 (4)	28 (1)	−790 (7)	401 (7)
C(2)	2421 (4)	1417 (1)	135 (7)	401 (7)
C(1′)	2705 (4)	870 (1)	137 (7)	386 (7)
O(5)	212 (3)	727 (1)	−1659 (7)	579 (7)
C(13)	5071 (5)	−896 (1)	803 (8)	576 (11)
C(15)	−1459 (5)	626 (2)	−971 (9)	770 (15)
C(14)	−155 (5)	−627 (2)	−837 (8)	583 (11)

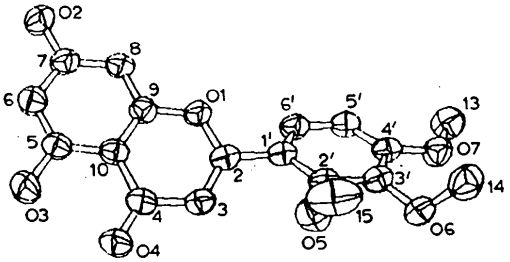

Fig. 1. View of the title compound showing the atom-numbering scheme.

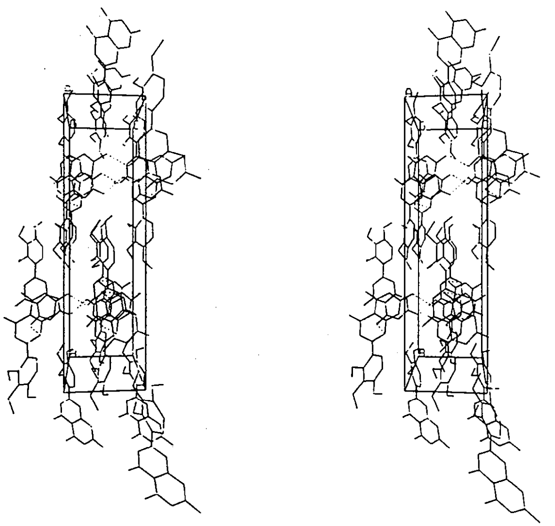

Fig. 2. Stereoscopic view of the unit cell with hydrogen bonds as dashed lines.

Table 2. *Bond distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses for the title compound*

C(8)—C(7)	1.393 (5)	C(6)—C(5)	1.380 (7)
O(7)—C(13)	1.441 (5)	C(5′)—C(6′)	1.391 (4)
C(6′)—C(1′)	1.405 (5)	C(10)—C(9)	1.389 (6)
C(8)—C(9)	1.386 (5)	C(10)—C(5)	1.418 (4)
C(5)—O(3)	1.338 (6)	C(2′)—C(3′)	1.387 (5)
C(3)—C(4)	1.437 (5)	C(10)—C(4)	1.427 (7)
C(2)—C(1′)	1.472 (4)	C(2′)—C(1′)	1.394 (5)
O(1)—C(9)	1.385 (4)	C(4′)—O(7)	1.363 (4)
C(3)—C(2)	1.355 (6)	C(2′)—O(5)	1.371 (5)
O(5)—C(15)	1.422 (6)	C(4′)—C(5′)	1.397 (5)
O(1)—C(2)	1.357 (6)	O(6)—C(3′)	1.365 (5)
C(7)—C(6)	1.388 (6)	C(4′)—C(3′)	1.407 (5)
C(7)—O(2)	1.340 (6)	O(6)—C(14)	1.437 (6)
O(4)—C(4)	1.255 (6)		

C(7)—C(8)—C(9)	117.2 (2)	C(10)—C(4)—O(4)	122.7 (3)
C(4)—C(3)—C(2)	121.4 (3)	C(8)—C(9)—O(1)	115.8 (3)
C(3)—C(2)—C(1′)	126.2 (3)	C(3)—C(4)—O(4)	121.8 (3)
C(2′)—C(1′)—C(6′)	118.6 (3)	C(8)—C(9)—C(10)	123.5 (3)
C(2′)—C(1′)—C(2)	122.1 (3)	C(3′)—O(6)—C(14)	116.3 (3)
C(9)—O(1)—C(2)	119.5 (3)	O(1)—C(9)—C(10)	120.8 (3)
C(6′)—C(1′)—C(2)	119.2 (3)	C(5′)—C(6′)—C(1′)	121.1 (3)
C(8)—C(7)—C(6)	121.8 (3)	O(7)—C(4′)—C(5′)	125.2 (3)
C(2′)—O(5)—C(15)	118.6 (3)	O(7)—C(4′)—C(3′)	114.8 (3)
C(8)—C(7)—O(2)	121.3 (3)	C(5′)—C(4′)—C(3′)	120.0 (3)
C(4′)—C(5′)—C(6′)	119.5 (3)	C(4′)—C(3′)—C(2′)	119.6 (3)
C(6)—C(7)—O(2)	117.0 (3)	C(4′)—O(7)—C(13)	118.0 (3)
C(7)—C(6)—C(5)	119.7 (3)	C(4′)—C(3′)—O(6)	120.7 (3)
C(3′)—C(2′)—C(1′)	121.2 (3)	C(6)—C(5)—C(10)	120.7 (3)
C(3′)—C(2′)—O(5)	119.5 (3)	C(2′)—C(3′)—O(6)	119.4 (3)
C(9)—C(10)—C(5)	117.2 (3)	C(6)—C(5)—O(3)	119.9 (3)
C(1′)—C(2′)—O(5)	119.3 (3)	O(1)—C(2)—C(3)	122.1 (3)
C(9)—C(10)—C(4)	120.7 (3)	C(10)—C(5)—O(3)	119.5 (3)
C(10)—C(4)—C(3)	115.6 (3)	O(1)—C(2)—C(1′)	111.7 (3)
C(5)—C(10)—C(4)	122.1 (3)		

*MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Refinement with anisotropic thermal parameters for all non-H atoms. The H atoms were placed in geometrically calculated positions assuming 1.08 Å for C—H. Only the H atoms of hydroxy groups involved in hydrogen bonding were located from a difference Fourier map and refined isotropically; $w = 1/\sigma^2$, $R = 0.044$, $wR = 0.062$ for 1551 observed reflections, 226 parameters. Final max. $(\Delta/\sigma) = 0.02$; $S = 3.88$. Maximum and minimum heights in final difference Fourier synthesis $= 0.28$ and $-0.33\ \text{e Å}^{-3}$. All calculations were carried out on a VAX8600 computer. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). The atomic parameters are given in Table 1.* The structure with the atom-numbering scheme is shown in Fig. 1. Bond distances and angles are given in Table 2. Fig. 2 represents a stereoscopic view of the unit cell with hydrogen bonds as dashed lines.

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* Lists of anisotropic displacement parameters, torsion angles, H-atom positions and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55527 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1011]

Related literature. The structure determination of the title compound was undertaken to elucidate its molecular conformation in order to help explain why this compound inhibits xanthine oxidase (Henichart, Pesando, Breittmayer, Wallet, Gaydou & Puiseux-Dao, 1991). A similar compound which is not a xanthine oxidase inhibitor has already been described (Wallet, Gaydou, Tinant, Declercq, Baldy & Bonifassi, 1990). 5,7-Dihydroxy-4′-methoxyflavone presents the same hydrogen-bond pattern (Cantrel, 1986). A survey of the structures of flavones and flavanones has been reported (Cody, 1988).

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Structure of 1,3-Di-*tert*-butyl-2,4-dichloro-2,4-bis[1-(trimethylsilyl)vinyl]-1,3-diaza-2,4-disilacyclobutane*

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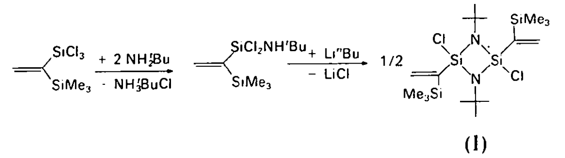
Abstract. $C_{18}H_{40}Cl_2N_2Si_4$, $M_r = 467.8$, monoclinic, $P2_1/c$, $a = 10.580\ (5)$, $b = 9.803\ (4)$, $c = 13.610\ (7)$ Å, $\beta = 104.57\ (4)°$, $V = 1366\ (1)$ Å^3, $Z = 2$, $D_x = 1.137$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0.71073$ Å, $\mu = 4.2$ cm^{-1}, $F(000) = 504$, $T = 223$ K, $R = 0.041$, $wR = 0.038$, for 2287 observed reflections. The central four-membered ring, Si_2N_2, shows a crystallographic centre of symmetry. Surrounded by very bulky ligands, the exocylic C(1)═C(2) double bond is preserved, and is therefore not easily accessible for further reaction.

Experimental. The title compound (I) was prepared in a multiple-step procedure (Penzenstadler, 1991). Recrystallization from *n*-hexane yielded colourless crystals suitable for X-ray crystallography. Elemental analysis: calculated C 46.20, H 8.62, N 5.99, Si 24.03, Cl 15.16%; found C 45.52, H 8.82, N 5.59, Si 23.90, Cl 14.96%. A well shaped colourless prism of approximate dimensions 0.51 × 0.31 × 0.31 mm was randomly mounted on a glass fibre. Preliminary examinations and data collection were carried out with Mo *Kα* radiation on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator. The observed extinctions ($h0l$: $l = 2n + 1$; $0k0$: $k = 2n + 1$) together with the monoclinic crystal system confirmed the space group $P2_1/c$. Final cell constants were obtained by least-squares refinement of 23 automatically centred high-angle reflections ($39.9 < 2\theta < 49.1°$). Data were collected using ω scans. Each reflection in the hemisphere h 12, k 11, l ±16 and $\theta_{\max} = 25.0°$ was measured with a maximum scan time of 90 s. Orientation control reflections were monitored every 100 reflections. No loss of intensity of three stand-



(I)

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* Silaheterocycles. XIX. Part XVIII: Auner & Penzenstadler (1992).

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