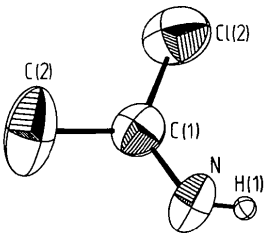

Fig. 1. Das Chloracetimin-Molekül mit Ellipsoiden der thermischen Schwingung (50% Aufenthaltswahrscheinlichkeit).

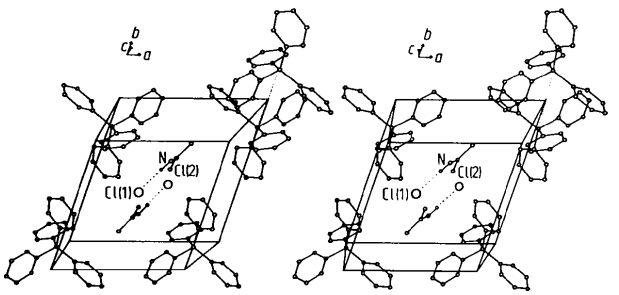

Fig. 2. Stereoskopische Ansicht der Elementarzelle. Punktiert: zwei über ein Inversionszentrum einander gegenüberstehende PPh_4^+-Ionen aus einer Zickzacklinie, die längs [001] verläuft.

Reflexe gemessen, davon 3852 unabhängig, R_{int} = 0,006, 274 mit $F < 3\sigma(F)$ als unbeobachtet gewertet. 'Empirische' Absorptionskorrektur nach ψ-scans, relative Transmissionsfaktoren 0,96 bis 1,00. Strukturbestimmung durch 'direkte Methode'. Verfeinerung durch Minimieren von $\sum w(|F_o| - |F_c|)^2$, $w = 1/\sigma^2(F)$. Phenyl- und Methyl-H-Atome aus modellmäßig berechneten Positionen angenommen, Imin-H-Atom aus Differenz-Fourier Synthese. Alle H-Atomlagen verfeinert mit individuellen isotropen Temperaturfaktoren, dabei Methylgruppe als starre Gruppe angenommen. $\Delta/\sigma_{max} < 0{,}03$, Restelektronendichte $-0{,}4 < \Delta\rho < 0{,}6$ e Å^{-3}. Keine Extinktionskorrektur. Atomformfaktoren: Cromer & Mann (1968). f', f'': Cromer & Liberman (1970). Rechenprogramme: Sheldrick (1976, 1986), Johnson (1965). $R = 0{,}051$, $wR = 0{,}048$. Die Atomparameter sind in Tabelle 1, die wichtigsten Atomabstände und -winkel in Tabelle 2 aufgeführt.* Fig. 1 zeigt das Chloracetimin-Molekül mit Ellipsoiden der thermischen Schwingung und Fig. 2 eine stereoskopische Ansicht der Elementarzelle.

Verwandte Literatur. $CH_3CClNH_2^+.Cl^-$: Williams, Peterson & Brown (1968). Ähnliche Packung der PPh_4^+-Ionen zu Zickzacklinien im β-$(AsPh_4)_2$-$[UCl_6].2CH_2Cl_2$: Müller, Klingelhöfer, Eicher & Bohrer (1984) und Conradi, Bohrer & Müller (1987).

* Die H-Atomkoordinaten, die Parameter für den anisotropen Temperaturfaktor und die Liste der beobachteten und berechneten Strukturfaktoren sind beim British Library Document Supply Centre (Supplementary Publication No. SUP 53014: 16 pp.) hinterlegt. Kopien sind erhältlich durch: The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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4′,5-Dihydroxy-7-methoxyflavanone

BY M. SHOJA

Chemistry Department, Fordham University, Bronx, NY 10458, *USA*

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Abstract. 5-Hydroxy-2-(4-hydroxyphenyl)-7-methoxy-4-chromanone, $C_{16}H_{14}O_5$, $M_r = 286{\cdot}28$, monoclinic, $P2_1/c$, $a = 13{\cdot}172\ (1)$, $b = 5{\cdot}660\ (1)$, $c = 18{\cdot}101\ (2)$ Å, $\beta = 97{\cdot}28\ (2)°$, $V = 1338{\cdot}6\ (4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}421$ g cm^{-3}, Cu $K\alpha$ radiation, $\lambda = 1{\cdot}5418$ Å, $\mu = 9{\cdot}0$ cm^{-1}, $F(000) = 600$, $T = 293$ K. Final $R = 0{\cdot}045$ for 1422 observed reflections. The hydroxy group at C(5) forms an intramolecular hydrogen

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

$$B_{eq} = (4/3)\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
O(1)	0·7846 (1)	0·2542 (3)	0·3121 (1)	3·53 (3)
O(2)	0·6924 (1)	0·1630 (3)	0·5171 (1)	4·42 (3)
O(3)	0·8064 (1)	0·5258 (3)	0·5656 (1)	4·92 (4)
O(4)	1·0105 (1)	0·8699 (3)	0·4047 (1)	5·38 (4)
O(5)	0·5575 (1)	−0·3092 (3)	0·0471 (1)	4·89 (4)
C(1′)	0·6460 (1)	0·0378 (4)	0·2468 (1)	3·24 (4)
C(2)	0·6799 (1)	0·1741 (4)	0·3165 (1)	3·41 (4)
C(2′)	0·5704 (2)	0·1273 (4)	0·1941 (1)	3·62 (4)
C(3′)	0·5415 (2)	0·0090 (4)	0·1281 (1)	3·83 (4)
C(3)	0·6764 (2)	0·0449 (4)	0·3891 (1)	3·71 (4)
C(4′)	0·5881 (1)	−0·2026 (4)	0·1136 (1)	3·39 (4)
C(4)	0·7182 (1)	0·1963 (4)	0·4542 (1)	3·35 (4)
C(5′)	0·6625 (1)	−0·2982 (4)	0·1659 (1)	3·53 (4)
C(5)	0·8342 (1)	0·5332 (4)	0·4965 (1)	3·55 (4)
C(6′)	0·6912 (2)	−0·1762 (4)	0·2318 (1)	3·78 (4)
C(6)	0·9056 (2)	0·6970 (4)	0·4824 (1)	3·99 (5)
C(7)	0·9392 (1)	0·7025 (4)	0·4128 (1)	3·66 (4)
C(8)	0·8999 (1)	0·5506 (4)	0·3560 (1)	3·53 (4)
C(9)	0·8247 (1)	0·3937 (4)	0·3701 (1)	3·05 (4)
C(10)	0·7905 (1)	0·3761 (4)	0·4403 (1)	3·13 (4)
C(11)	1·0458 (2)	0·8984 (6)	0·3340 (1)	6·33 (6)

Table 2. *Bond lengths* (Å), *angles* (°) *and selected torsion angles* (°); *e.s.d.'s in parentheses*

O(1)—C(2)	1·463 (2)	C(3′)—C(4′)	1·385 (3)
O(1)—C(9)	1·365 (2)	C(3)—C(4)	1·503 (3)
O(2)—C(4)	1·244 (2)	C(4′)—C(5′)	1·383 (2)
O(3)—C(5)	1·348 (2)	C(4)—C(10)	1·438 (3)
O(4)—C(7)	1·355 (3)	C(5′)—C(6′)	1·389 (3)
O(4)—C(11)	1·425 (3)	C(5)—C(6)	1·368 (3)
O(5)—C(4′)	1·361 (2)	C(5)—C(10)	1·417 (3)
C(1′)—C(2)	1·497 (3)	C(6)—C(7)	1·387 (3)
C(1′)—C(2′)	1·385 (3)	C(7)—C(8)	1·389 (3)
C(1′)—C(6′)	1·391 (3)	C(8)—C(9)	1·378 (3)
C(2)—C(3)	1·511 (3)	C(9)—C(10)	1·405 (2)
C(2′)—C(3′)	1·381 (3)		

C(2)—O(1)—C(9)	114·4 (1)	C(4′)—C(5′)—C(6′)	119·1 (2)
C(7)—O(4)—C(11)	119·1 (2)	O(3)—C(5)—C(6)	118·0 (2)
C(2)—C(1′)—C(2′)	120·2 (2)	O(3)—C(5)—C(10)	120·9 (2)
C(2)—C(1′)—C(6′)	121·6 (2)	C(6)—C(5)—C(10)	121·1 (2)
C(2′)—C(1′)—C(6′)	118·2 (2)	C(1′)—C(6′)—C(5′)	121·5 (2)
O(1)—C(2)—C(1′)	107·2 (1)	C(5)—C(6)—C(7)	119·4 (2)
O(1)—C(2)—C(3)	109·3 (1)	O(4)—C(7)—C(6)	114·8 (2)
C(1′)—C(2)—C(3)	116·5 (2)	O(4)—C(7)—C(8)	123·4 (2)
C(1′)—C(2′)—C(3′)	120·9 (2)	C(6)—C(7)—C(8)	121·8 (2)
C(2′)—C(3′)—C(4′)	120·2 (2)	C(7)—C(8)—C(9)	118·1 (2)
C(2)—C(3)—C(4)	111·0 (2)	O(1)—C(9)—C(8)	116·6 (2)
O(5)—C(4′)—C(3′)	117·5 (2)	O(1)—C(9)—C10)	121·2 (2)
O(5)—C(4′)—C(5′)	122·4 (2)	C(8)—C(9)—C(10)	122·2 (2)
C(3′)—C(4′)—C(5′)	120·1 (2)	C(4)—C(10)—C(5)	122·4 (2)
O(2)—C(4)—C(3)	121·1 (2)	C(4)—C(10)—C(9)	120·3 (2)
O(2)—C(4)—C(10)	122·1 (2)	C(5)—C(10)—C(9)	117·2 (2)
C(3)—C(4)—C(10)	116·8 (2)		

C(2)—O(1)—C(9)—C(10)	26·5 (2)	O(1)—C(9)—C(10)—C(4)	5·2 (3)
C(9)—C(10)—C(4)—C(3)	−4·5 (3)	C(10)—C(4)—C(3)—C(2)	−25·8 (2)
C(4)—C(3)—C(2)—O(1)	54·9 (2)	C(3)—C(2)—O(1)—C(9)	−56·5 (2)

bond with the carbonyl group resulting in a six-membered ring. It also exhibits intermolecular hydrogen bonds with molecules related by the screw axis. The methoxy group is slightly twisted out of the plane of the flavanone ring. The torsion angle for C(11)—O(4)—C(7)—C(8) is 1·8 (3)°.

Experimental. The title compound (Fig. 1) was purchased from the Indofine Chemical Company. Pale yellow needles grown from methanol solution. Data collected on Enraf–Nonius CAD-4 diffractometer, graphite monochromator. Crystal dimensions 0·20 × 0·20 × 0·35 mm. Cell parameters measured on the diffractometer using 25 reflections in the 2θ range 20–40°. Range of indices $0 \le h \le 14$, $0 \le k \le 6$, $-20 \le l \le 20$ ($\theta \le 60°$). Three standards 404, 111, 322, measured after every 200 reflections showed a variation of 0·2%. No absorption corrections. Lorentz and polarization corrections. 1992 unique reflections measured. 1422 observed reflections with $|F_o| > 3\sigma(|F_o|)$. Direct methods, (*MULTAN*82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) used for structure determination. H atoms located by difference Fourier synthesis. Anisotropic full-matrix least-squares refinement (on F) for non-H atoms, isotropic for H atoms. $\sum w(|F_o| - |F_c|)^2$ minimized. The non-Poisson $w = 4F_o^2/[\sigma^2(I) + (pF_o^2)^2]$, $p = 0{\cdot}04$. $wR = 0{\cdot}045$, max. $\Delta/\sigma = 0{\cdot}06$. Max. peak height in the final difference Fourier map 0·21 e Å^{-3}, $S = 1{\cdot}450$, for 247 variables. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Enraf–Nonius *SDP* (Frenz,

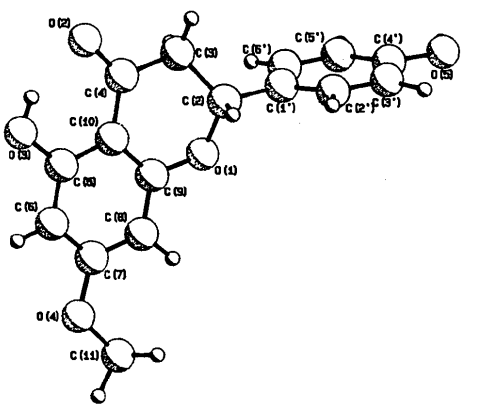

Fig. 1. Numbering of atoms and conformation of the molecule.

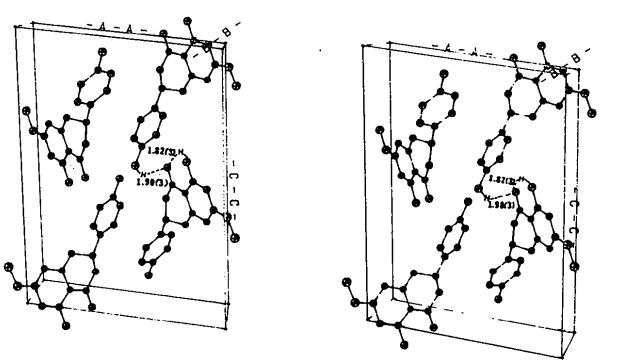

Fig. 2. Stereoview of the unit cell. Intra- and intermolecular hydrogen bonding shown as dashed lines.

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 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).

* 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.

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Methyl (4*R*,9*R*,10*S*)-7-Isopropyl-4-methyl-5,8-dioxo-1,6-tricyclo[8.3.0.0^{4,9}]tridecadiene-9-carboxylate

BY J. ZUKERMAN-SCHPECTOR, E. E. CASTELLANO AND C. A. DE SIMONE

Instituto de Física e Química de São Carlos, Universidade de São Paulo, Caixa Postal 369, 13560 *São Carlos, SP, Brazil*

AND TIMOTHY J. BROCKSOM AND V. CATANI

Departamento de Química, Universidade Federal de São Carlos, Caixa Postal 676, 13560 *São Carlos, SP, Brazil*

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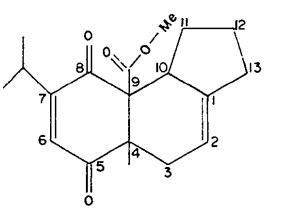
Abstract. $C_{19}H_{24}O_4$, $M_r = 316{\cdot}40$, orthorhombic, *Pbca*, $a = 12{\cdot}444$ (1), $b = 14{\cdot}615$ (3), $c = 18{\cdot}797$ (5) Å, $V = 3419$ (2) Å^3, $Z = 8$, $D_x = 1{\cdot}229$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}79$ cm^{-1}, $F(000) = 1360$, $T = 298$ K, $R = 0{\cdot}050$ 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.



(1)

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