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7-Benzyloxy-1,4-dihydro-6-methoxyisocoumarin*

BY K. SIVAKUMAR, K. CHINNAKALI, K. SUBRAMANIAN AND S. NATARAJAN

Department of Physics, Anna University, Madras 600 025, *India*

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Abstract. $C_{17}H_{16}O_4$, $M_r = 284{\cdot}3$, monoclinic, $P2_1/c$, $a = 10{\cdot}822$ (1), $b = 13{\cdot}961$ (1), $c = 10{\cdot}047$ (1) Å, $\beta = 109{\cdot}57$ (1)°, $V = 1430{\cdot}3$ (2) Å^3, $Z = 4$, $D_m = 1{\cdot}33$ (2), $D_x = 1{\cdot}32$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 7{\cdot}3$ cm^{-1}, $F(000) = 600$, $T = 294$ K, $R = 0{\cdot}056$ for 2034 observed reflections. The dihydropyran ring is in the boat conformation. The phenyl ring is planar and perpendicular to the plane of the isocoumarin skeleton. The crystal packing is stabilized by van der Waals interactions.

Introduction. Many coumarin and isocoumarin derivatives are of biological importance (Michel & Durant, 1976; Schmalle, Jarchow, Hausen & Schulz, 1982). The crystal structure of the title compound has been determined as part of our program on the crystal-structure analysis of these derivatives.

Experimental. Crystals from methanol–chloroform mixture, density measured by flotation. CAD-4 diffractometer, crystal dimensions 0·20 × 0·20 × 0·15 mm, monochromatized Cu $K\alpha$ radiation, cell parameters by least squares for 21 reflections with $25 \le \theta \le 35°$, intensity data for $0 < \theta < 60°$, $\omega/2\theta$ scan, two standard reflections for every 100 observations, Lp but not absorption, correction; 2242 reflections (h 0→11, k 0→14, l −11→11), 2034 with $I > 3\sigma(I)$. Direct methods with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). An E map, computed from the set of phases with the largest combined figure of merit, revealed the structure. Full-matrix least-squares refinement on F; anisotropic temperature factors for C and O atoms, isotropic for H atoms. H-atom positions from difference map. $w = [\sigma^2(F_o) + 0{\cdot}0315F_o^2]^{-1}$, final $R = 0{\cdot}056$, $wR = 0{\cdot}064$ for 2034 observed reflections; $S = 1{\cdot}39$, $(\Delta/\sigma)_{max} = 0{\cdot}047$, final F map featureless; excursions −0·27, 0·25 e Å^{-3}, no corrections for secondary extinction; scattering factors as in *SHELX*76 (Sheldrick, 1976). The geometrical and crystal packing were computed by the program *PARST* (Nardelli, 1983). Calculations were performed on an IBM 360/44 computer.

Discussion. Final atomic parameters are given in Table 1.† Bond distances and angles are given in Figs. 1(*a*) and 1(*b*) respectively. A thermal-ellipsoid plot of the molecule is shown in Fig. 2.

* 7-Benzyloxy-6-methoxy-3-isochromanone.

† Lists of the structure factors, anisotropic thermal parameters, H-atom coordinates, distances and angles involving H atoms, torsion angles, least-squares planes and intermolecular distances less than 3·5 Å have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51625 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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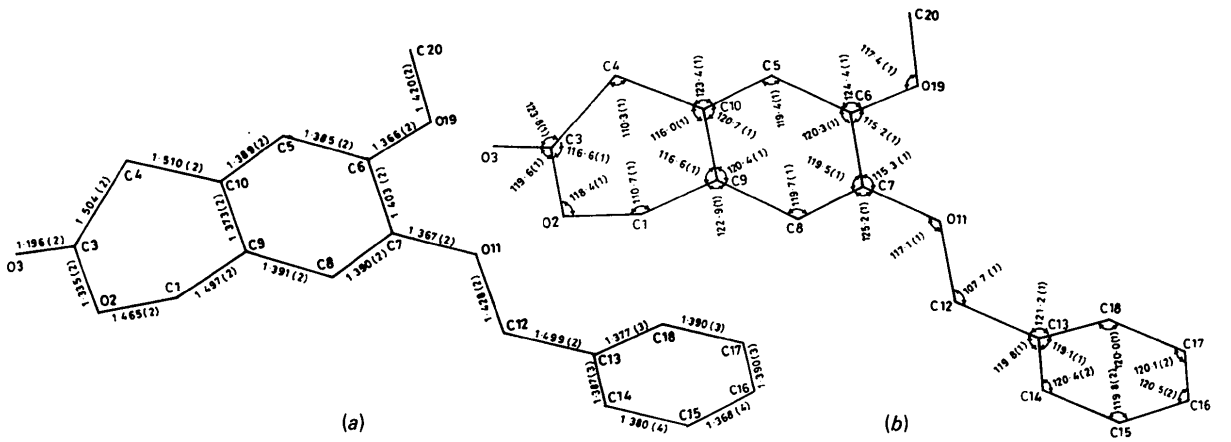

Fig. 1. (*a*) Atom numbering and bond lengths (Å) and (*b*) bond angles (°), with e.s.d.'s in parentheses.

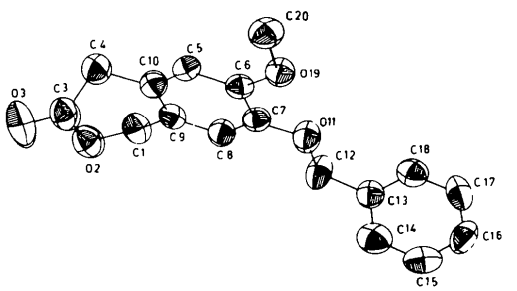

Fig. 2. *ORTEP* drawing (Johnson, 1976) of the title compound. Thermal ellipsoids are at the 50% probability level.

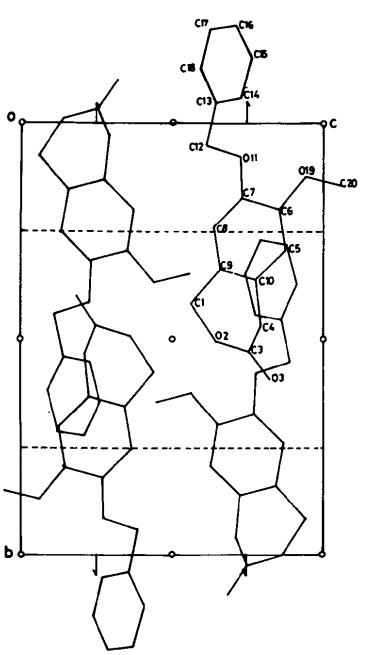

Fig. 3. Molecular packing of the title compound viewed along the *a* axis.

Table 1. *Positional and isotropic thermal parameters with e.s.d.'s in parentheses*

$B_{eq} = \frac{8}{3}\pi^2(U_{11} + U_{22} + U_{33})$.

	x	*y*	*z*	B_{eq} (Å^2)
C(1)	0·2331 (2)	0·4201 (1)	0·5592 (2)	4·98 (5)
O(2)	0·2160 (1)	0·5040 (1)	0·6388 (2)	5·56 (6)
C(3)	0·3159 (2)	0·5306 (1)	0·7524 (2)	5·29 (5)
O(3)	0·3040 (2)	0·5986 (1)	0·8195 (2)	7·81 (7)
C(4)	0·4377 (2)	0·4700 (1)	0·7898 (2)	5·03 (5)
C(5)	0·4676 (2)	0·2960 (1)	0·8723 (2)	3·91 (5)
C(6)	0·4280 (1)	0·2013 (1)	0·8498 (2)	3·47 (5)
C(7)	0·3205 (1)	0·1757 (1)	0·7315 (2)	3·72 (5)
C(8)	0·2570 (2)	0·2456 (1)	0·6341 (2)	4·09 (6)
C(9)	0·2983 (2)	0·3404 (1)	0·6572 (2)	4·06 (6)
C(10)	0·4019 (2)	0·3651 (1)	0·7748 (2)	3·93 (5)
O(11)	0·2849 (1)	0·0814 (1)	0·7246 (1)	4·44 (6)
C(12)	0·1692 (2)	0·0540 (1)	0·6124 (2)	5·58 (5)
C(13)	0·1326 (1)	−0·0449 (1)	0·6439 (2)	4·22 (5)
C(14)	0·0562 (2)	−0·0569 (2)	0·7293 (2)	5·24 (5)
C(15)	0·0239 (2)	−0·1476 (2)	0·7614 (2)	6·12 (7)
C(16)	0·0680 (2)	−0·2261 (2)	0·7093 (3)	5·97 (5)
C(17)	0·1412 (2)	−0·2156 (1)	0·6234 (2)	5·65 (5)
C(18)	0·1743 (2)	−0·1246 (2)	0·5903 (2)	4·95 (5)
O(19)	0·4860 (1)	0·1274 (1)	0·9377 (1)	4·15 (5)
C(20)	0·5978 (2)	0·1492 (2)	1·0567 (2)	4·83 (5)

Table 2. *Torsion angles* (°) *for the dihydropyran ring*

O(2)–C(3)–C(4)–C(10)	−42·5 (2)
C(3)–C(4)–C(10)–C(9)	42·6 (2)
C(4)–C(10)–C(9)–C(1)	−0·4 (2)
C(10)–C(9)–C(1)–O(2)	−42·8 (2)
C(9)–C(1)–O(2)–C(3)	44·0 (2)
C(1)–O(2)–C(3)–C(4)	−1·0 (2)

The dihydropyran ring is in the boat conformation (see Table 2); the asymmetry parameter C_s (C1) = 1·07 (Duax, Weeks & Rohrer, 1976). The C—O distances C(1)–O(2) 1·465 (2) and C(3)–O(2) 1·335 (2) Å are asymmetrical. The benzene ring of the isocoumarin skeleton is essentially planar with maximum deviations at C(6) and C(7) [0·009 (1) and −0·009 (1) Å respectively]. The methoxy group O(19)–C(20) lies in

the plane of the benzene ring [torsion angle C(5)–C(6)–O(19)–C(20) = −2·8 (2)°]; this coplanarity results in a close approach between C(20) and C(5) [2·805 (3) Å] which causes expansion of the angle C(5)–C(6)–O(19) [124·4 (1)°] and contraction of the angle C(7)–C(6)–O(19) [115·2 (1)°] (Sheldrick, Akrigg & Geddes, 1980; Koetzle & Williams, 1976). The shortened distance C(7)–O(11) 1·367 (2) Å and angle C(7)–O(11)–C(12) 117·1 (1)° are, perhaps, indicative of some sp^2 character of the atom O(11) (Durant, Bufkens, Lefeore, Evrard & Michel, 1985). The phenyl ring is planar ($\chi^2 = 37{\cdot}4$) and is nearly perpendicular to the least-squares plane of the isocoumarin skeleton [dihedral angle 91·7 (5)°].

The molecular packing viewed along **a** is shown in Fig. 3. The molecules are held in the crystal by van der Waals interactions.

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Acta Cryst. (1989). C**45**, 810–811

Structure of Rubidium Uranyl(VI) Trinitrate

BY ALLAN ZALKIN, LIESELOTTE K. TEMPLETON AND DAVID H. TEMPLETON

Materials and Chemical Sciences Division, Lawrence Berkeley Laboratory, 1 Cyclotron Road, Berkeley, CA 94720, USA, and Department of Chemistry, University of California, Berkeley, CA 94720, USA

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Abstract. $RbUO_2(NO_3)_3$, $M_r = 541{\cdot}51$, hexagonal, $R\bar{3}c$, $a = 9{\cdot}384$ (4), $c = 18{\cdot}899$ (6) Å, $V = 1441{\cdot}3$ (14) Å^3, $Z = 6$, $D_m = 3{\cdot}72$, $D_x = 3{\cdot}743$ (4) g cm^{-3}, Mo $K\alpha$, $\lambda(\alpha_1) = 0{\cdot}70930$ Å, $\mu = 209{\cdot}7$ cm^{-1}, $F(000) = 1428$, $T = 296$ K, $R = 0{\cdot}014$ for 261 independent reflections with $I > 2\sigma(I)$. The uranyl ion is coordinated in bidentate fashion by three nitrate ions in its equatorial plane. Bond lengths are U–O = 1·746 (4) (uranyl), 2·474 (3) (nitrate), N–O = 1·205 (6) (terminal), 1·268 (4) Å (bridge). The anomalous-scattering term f' for U measured at 0·71 Å is −10·7 (2).

Experimental. Crystals were prepared by slow evaporation of an aqueous solution of rubidium nitrate, uranyl nitrate and nitric acid. The measured density is quoted from Hoard & Stroupe (1943). A prismatic crystal 0·038 × 0·063 × 0·16 mm (8 faces, elongated on **c**) was glued to a glass fiber and mounted on a Picker diffractometer equipped with a graphite monochromator. Cell dimensions were derived from 14 reflections in the range $13 < \theta < 27°$. Integrated intensities were measured by θ–2θ scan for 1295 reflections permitted by the space group in the ranges: θ up to 25° for h −11 to 0, k 0 to 10, l 0 to 22 and up to 27·5° for h −12 to 0, k 0 to 11, l −24 to 0. After analytical correction for absorption ($2{\cdot}09 < A < 3{\cdot}53$) and adjustments of up to 2% based on variation of two intensity standards, reflections which were equivalent were averaged giving 378 unique ones and 261 with

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