

Fig. 2. The molecular packing: N—H...S bridges are denoted by dotted lines.

only the electron density of H1 was localized at 0.85 (3) Å from the indolyl N atom.

The bond distances and uniformly small angles of the indolyl unit show the delocalization of π electrons over the whole indolyl moiety; the bond C4—C9 [1.417 (3) Å] is elongated in comparison with a typical benzene bond, the bonds C3—C4 [1.424 (3) Å], N1—C2 [1.369 (2) Å] are shorter than expected for single bonds, and C(2)—C(3) [1.392 (2) Å] cannot be considered as a double bond.

No conjugation between the indolyl and pyridyl ring can be assumed because of a high torsion angle around the C3—N11 bond; C2—C3—N11—C12 is 55.4° and C4—C3—N11—C16 58.3°. The C3—N11

bond deviates slightly from the indolyl plane; the torsion angles are C5—C4—C3—N11 -2.7° and S10—C2—C3—N11 6.5° .

The packing of atoms in the unit cell is shown in Fig. 2. The molecules are paired by two symmetrically equivalent hydrogen bridges N—H...Sⁱ, thus forming dimers around the screw axis: N1—H1 0.85 (2), N1...Sⁱ 3.366 (2), H1...Sⁱ 2.57 (2) Å, angle N1—H1...Sⁱ 157 (1)° [(i) $-x, y-\frac{1}{2}, \frac{3}{2}-z$].

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Structure of Karanjin*

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Abstract. C₁₈H₁₂O₄, $M_r = 292.3$, monoclinic, $P2_1/n$, $a = 7.196$ (3), $b = 11.275$ (3), $c = 17.495$ (3) Å, $\beta = 100.70$ (2)°, $V = 1394.8$ (7) Å³, $D_m = 1.38$ (2), $D_x = 1.39$ g cm⁻³, $Z = 4$, $\lambda(\text{Cu K}\alpha) = 1.5418$ Å, $\mu = 7.2$ cm⁻¹, $T = 293$ K, $F(000) = 608$, $R = 0.058$, $wR =$

0.066 for 1453 reflections with $I \geq 2\sigma(I)$. The furan ring is planar while the pyran ring is distorted from planarity. The phenyl ring makes an angle of 28.7 (2)° with the pyran ring.

Introduction. The title compound, a naturally occurring furoflavone, extracted from the seed oil of the Indian beech *Pongamia glabra* (Aneja, Khanna & Seshadri, 1963), was obtained from commercial sour-

* 3-Methoxy-2-phenyl-4H-furo[2,3-h][1]benzopyran-4-one.

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Table 1. *Positional parameters and equivalent isotropic temperature factors of non-hydrogen atoms with e.s.d.'s in parentheses*

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	$U_{eq}(\text{\AA}^2)$
O1	0.2530 (5)	-0.2718 (3)	0.4546 (2)	0.073 (2)
O2	0.2582 (4)	0.1191 (3)	0.5300 (2)	0.050 (2)
O3	0.3171 (6)	0.2426 (4)	0.3174 (2)	0.076 (2)
O4	0.3064 (5)	0.3995 (3)	0.4361 (2)	0.066 (2)
C1	0.2306 (7)	-0.1487 (4)	0.5539 (3)	0.059 (3)
C2	0.2333 (8)	-0.2616 (5)	0.5311 (4)	0.071 (4)
C3	0.2621 (7)	-0.1590 (5)	0.4288 (3)	0.058 (3)
C4	0.2844 (9)	-0.1269 (6)	0.3538 (3)	0.072 (4)
C5	0.2928 (8)	-0.0085 (6)	0.3392 (3)	0.067 (3)
C6	0.2823 (7)	0.0764 (4)	0.3969 (2)	0.053 (2)
C7	0.2611 (6)	0.0424 (4)	0.4708 (2)	0.046 (2)
C8	0.2485 (6)	-0.0796 (5)	0.4871 (3)	0.050 (2)
C9	0.2951 (7)	0.2036 (4)	0.3813 (3)	0.056 (3)
C10	0.2880 (7)	0.2804 (4)	0.4467 (3)	0.050 (3)
C11	0.2753 (6)	0.2379 (4)	0.5179 (2)	0.047 (2)
C12	0.1423 (11)	0.4532 (6)	0.3874 (5)	0.077 (4)
C13	0.2780 (6)	0.3057 (4)	0.5901 (3)	0.048 (2)
C14	0.3738 (8)	0.4135 (5)	0.6030 (3)	0.059 (3)
C15	0.3803 (9)	0.4756 (5)	0.6720 (3)	0.066 (3)
C16	0.2837 (10)	0.4312 (6)	0.7276 (4)	0.080 (4)
C17	0.1899 (10)	0.3255 (6)	0.7155 (3)	0.078 (4)
C18	0.1887 (8)	0.2610 (5)	0.6479 (3)	0.063 (3)

ces. It is widely used as a drug in Indian medicines for treating scabies, herpes and leucoderma. It is also used in the treatment of dyspepsia with sluggish liver, diarrhoea, cough, leprosy and beri-beri (Krishnamurthy, 1969; Nadkarni & Chopra, 1976). This paper describes the molecular structure of karanjin obtained by X-ray diffraction techniques.

Experimental. Colourless needle-shaped single crystals by slow evaporation from methanol, $0.22 \times 0.17 \times 0.12$ mm, D_m by flotation, three-dimensional intensity data from Enraf-Nonius CAD-4 automatic diffractometer using graphite-monochromated Cu $K\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$). Cell constants from 25 strong reflections in the range $34 \leq 2\theta \leq 84^\circ$, $\omega/2\theta$ scan mode, 2416 reflections collected in the range $4 \leq 2\theta \leq 130^\circ$, $0 \leq h \leq 8$, $0 \leq k \leq 13$, $-20 \leq l \leq 20$. Two standard reflections (251, $\bar{2}$ 34) showed 3% average fluctuation, Lorentz and polarization corrections but no absorption correction ($\mu t < 1$). 1453 reflections with $I \geq 2\sigma(I)$, structure solution by direct methods using MULTAN80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). H-atom positions from ΔF map, full-matrix least-squares refinement with anisotropic temperature factors for non-hydrogen atoms and isotropic for hydrogens using SHELX76 (Sheldrick, 1976). Final $R = 0.058$ and $wR = 0.066$, individual weighting scheme where $w = 0.005/[\sigma^2(F_o^2) + 0.034F_o^2]$, goodness of fit S for 247 parameters = 0.023, final (shift/e.s.d.) $_{\max} = 0.504$ and (shift/e.s.d.) $_{\text{av}} = 0.106$. Final ΔF map showed no peaks $> 0.25 \text{ e \AA}^{-3}$. The scattering factors are as in *International Tables for X-ray Crystallography* (1974).

Discussion. The final positional parameters with equivalent isotropic temperature factors of non-hydrogen atoms are given in Table 1. The bond lengths and angles involving all the non-hydrogen atoms are shown in Figs. 1 and 2 respectively.*

In all essential details, the geometry of the molecules in terms of bond lengths and angles shows normal values.

* 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 52299 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

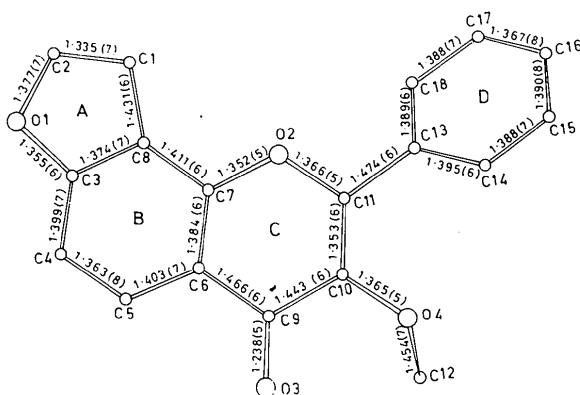


Fig. 1. Bond lengths (\AA) involving non-hydrogen atoms.

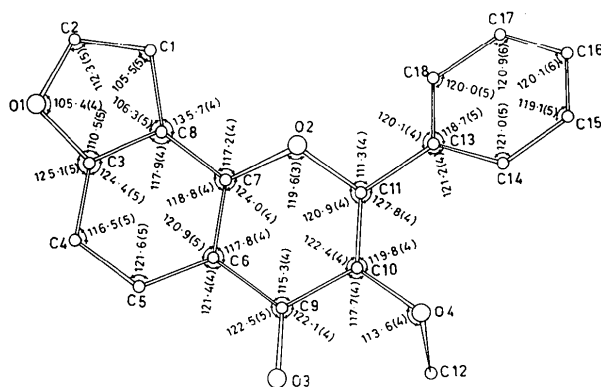


Fig. 2. Bond angles ($^\circ$) involving non-hydrogen atoms.

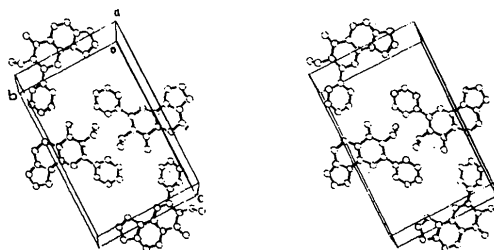


Fig. 3. Stereoview of the packing of the molecules in the unit cell.

The C—O bond lengths in the pyrone ring are symmetrical with C7—O2 = 1.352 (5) and C11—O2 = 1.366 (5) Å.

The widening of the angle O2—C7—C6, 124.0 (4)°, and the narrowing of the angle C6—C9—C10, 115.3 (4)°, in ring *C* may be attributed to the ring strain caused by the neighbouring Csp^2 — Csp^2 atoms and has been observed in ethyl 1-cyano-1,2,3,4-tetrahydro-2-isoquinolinecarboxylate (Gzella, Jaskólski, Rychlewska & Kosturkiewicz, 1984).

The methoxy group attached to ring *C* is rotated from the plane of the ring about the bond C10—O4 by an angle of 71.6 (6)°. The deviation of C12 from the plane of the ring is 1.226 (8) Å. A similar rotation of the methoxy group has been observed in various structures: celebixanthone, 72.8° (Stout, Stout & Welsh, 1963), 2-hydroxy-1,3,4,7-tetramethoxyxanthone, 72.0, 58.1, 83.0° (Stout, Shunlin & Singh, 1969), xanthone B, 64.0° (Ravikumar, Rajan & Padmanabhan, 1987).

The rings *A*, *B* and *D* are planar ($\chi^2 = 1.0, 8.7$ and 29.6 respectively). The pyran ring *C* is distorted ($\chi^2 = 68.1$). The angles between *A* and *B*, *A* and *C*, and *B* and *C* are 1.1 (2), 3.0 (2) and 2.4 (2)° respectively, showing that the rings *A*, *B* and *C* are coplanar. Similar coplanarity of the phenyl ring *B* and the pyrone ring *C* has been observed in 3',5,5',6-tetramethoxyflavone (Ting, Watson & Dominguez, 1972). The rotation of the phenyl ring *D* through 28.7 (2)° from the plane of the pyran ring may be to minimize the steric interaction between the oxygen atom O2 in the pyrone ring and O4 of the methoxy group with the phenyl ring *D*. This is substantiated by the distances HC18...O2 and HC14...O4, 2.36 (6) and 2.36 (5) Å respectively, which are less than 2.62 Å, the sum of the van der Waals radii of the H and O atoms (Srinivasan, Meena Hariharan & Vijayalakshmi, 1987; Nyburg & Faerman, 1985).

A stereoview of the unit-cell packing (Motherwell, 1978) is shown in Fig. 3. The molecules are stacked

along the *a* axis with C7...C8 ($1-x, -y, 1-z$) being 3.494 (7) Å. The structure is stabilized by C—H...O-type contacts and stacking forces. C14 is linked by a hydrogen bond to O4 ($1-x, 1-y, 1-z$) with C14...O4 3.285 (7), C14—HC14 1.01 (6) Å and angle C14—HC14...O4 137 (4)°. There is another C...O contact, between C4 and O3 ($\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$), with C4...O3 3.294 (7) Å. The molecules lie approximately in the *bc* plane.

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Structure of Dicyclopenta[*ef,k*]heptalene (Azupyrene)

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Abstract. C₁₆H₁₀, *M_r* = 202.255, monoclinic, *P*2₁/*c*, *a* = 8.356 (1), *b* = 11.076 (3), *c* = 11.446 (2) Å, β = 104.91 (1)°, *V* = 1023.8 (7) Å³, *Z* = 4, *D_x* = 1.312 g cm⁻³, λ(Mo *K*α) = 0.71073 Å, μ =

0.692 cm⁻¹, *F*(000) = 424, *T* = 298 (1) K, *R* = 0.055 for 961 observed reflections. The molecule is planar with slight perimeter bond-length alternation, primarily in the five-carbon-ring moieties. The lengths of