compound is long compared with that in 1-dimethylamino-4-nitrobenzene, 1.358 Å (Mak & Trotter, 1965) and in *p*-dimethylaminobenzaldehyde oxime, 1.380 Å (Bachechi & Zambonelli, 1972). Therefore, the degree of the double-bond character in the title compound is relatively small.

The torsion angles C(12)-C(11)-C(13)-O(1), C(11) - C(12) - C(16) - O(3), $-154.2(4)^{\circ}$, and $-142.9(3)^{\circ}$, show that the two carbonyl groups twist from the mean plane of the triazole ring on opposite sides. These directions differ notably in the case of diisopropyl 1-(1-naphthyl)-1H-1,2,3-triazole-4,5-dicarboxylate (Nagawa, Goto, Honda & Nakanishi, 1988), in which the carbonyl group at the 4-position exists in the triazole plane and at the 5-position it twists from the triazole plane. The results reflect the requirements of intramolecular contacts of the extra dimethylamino group. The larger temperature factors of C(15) and O(1) atoms might be due to the disorder.

The packing of the molecule in the crystal is shown in Fig. 2. The crystal structure is stabilized mainly by van der Waals forces; the shortest intermolecular distance is 3.315(5) Å for C(19) $(x, y, z) \cdots N(3) (1-x, -y, 1-z)$.

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Acta Cryst. (1990). C46, 435-437

Structure of 3-(1-Pyridinio)indole-2-thiolate

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(Received 13 February 1989; accepted 10 May 1989)

Abstract. $C_{13}H_{10}N_2S$, $M_r = 226\cdot3$, monoclinic, $P2_1/c$, $a = 10\cdot037$ (4), $b = 8\cdot076$ (3), $c = 14\cdot124$ (4) Å, $\beta = 104\cdot59$ (3)°, $V = 1107\cdot9$ (6) Å³, Z = 4, $D_m = 1\cdot34$ (1), $D_x = 1\cdot36$ g cm⁻³, λ (Mo $K\alpha$) = 0.71069 Å, $\mu = 2\cdot6$ cm⁻¹, F(000) = 472, T = 295 K, $R = 0\cdot039$ for 2057 observed reflexions. Sulfur forms a hydrogen bond to the NH group of the neighbouring molecule symmetrically related by the screw axis along b. The phenyl ring is rotated out of the indolyl plane by 57°.

Introduction. X-ray structure analysis of the title compound has been used to confirm the chemical constitution of the product of a new synthetic method for the preparation of indolyl-based com-

0108-2701/90/030435-03\$03.00

pounds (Gonda, Kristian & Imrich, 1987; Gonda & Kristian, 1988) and to obtain information about the contributions of the resonance states (I)–(III) to the average crystal structure. This paper is part of a general project on the development of biologically efficient indolyl compounds substituted by different function groups.





NI

C2

C3 C4 C5

C6

C7 C8

C9

S10 N11

C12

C13

C14 C15

C16

Experimental. Crystals of 3-(1-pyridinio)indole-2thiolate (m.p. 480-482 K) were prepared by Gonda, Kristian & Imrich (1987). A suitable crystal (dimensions $0.6 \times 0.4 \times 0.4$ mm) was cut from a thick darkblue slab. The crystal density was measured pycnometrically in toluene-CCl₄. The intensities were measured with a Syntex $P2_1$ diffractometer using θ -2 θ scans at room temperature. 2057 of 2559 independent reflexions $[(\sin \theta)/\lambda < 0.651 \text{ Å}^{-1}, h 0/13,$ k 0/10, l - 18/18 were classified as observed with F_o $> 3.92\sigma_F$. Space group $P2_1/c$ was confirmed by Weissenberg photographs and by scanning of systematically extinct reflexions. Unit-cell parameters were determined from 17 reflexions with $15 < 2\theta <$ 28°. Three reference intensities measured at intervals of 30 reflexions were stable within 2% during data collection. Other measurement details are as given in Hašek (1985). No corrections for absorption or extinction were made. The structure was solved by MULTAN80 (Main et al., 1980) and refined by SHELX76 (Sheldrick, 1976). Atomic scattering factors were taken from International Tables for X-ray Crystallography (1974). $\sum w(|F_o| - |F_c|)^2$ minimized with $w = |\sigma_F^2 + 0.015|F_o|^2]^{-1}$; N1–C16 refined with anisotropic temperature parameters and H1-H16 localized from difference map with isotropic temperature parameters until all the parameter shifts dropped below 0.2 of the respective e.s.d.'s. The resulting R = 0.039, wR = 0.052 for 2057 independent reflexions and 185 refined parameters. The highest peak on the final difference map is $0.2 \text{ e} \text{ Å}^{-3}$ and the minimum height is $-0.32 \text{ e} \text{ Å}^{-3}$. Atomic coordinates and the U_{eq} 's for non-H atoms are given in Table 1.* Bond lengths and bond angles are given in Fig. 1. The structure geometry was calculated using XANADU (Roberts & Sheldrick, 1976), PARST (Nardelli, 1983) and IMC (Hašek, 1981).

Discussion. The average bond length in the benzene ring of the indolyl group is as usual $[1\cdot393(1) \text{ Å}]$; however, the C—C bond common to both the fiveand six-membered rings is longer, $1\cdot417(2) \text{ Å}$. The bonds in the pyrrole ring, in the sequence C—N—C—C—C, are $1\cdot382(2)$, $1\cdot369(2)$, $1\cdot392(2)$ and $1\cdot424(2) \text{ Å}$, respectively, and none can be considered to be a single or a double bond. The S atom participating in the hydrogen bridge N—H…S is on the opposite side of the indole to the pyridine ring. The angles are C—C—S $130\cdot8(1)$, N—C—S $123\cdot4(1)^{\circ}$, with the corresponding N—C—C angle $105\cdot8(1)^{\circ}$.

* Lists of H-atom parameters, anisotropic temperature parameters and structure factors, together with a scheme showing torsion angles, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52230 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final coordinates $(\times 10^5)$ and equivalent isotropic thermal parameters $(\times 10^3 \text{ Å}^2)$ for the non-H atoms with e.s.d.'s in parentheses

$\begin{array}{cccccccccccccccccccccccccccccccccccc$	
- 15749 (15) - 9730 (19) 69904 (11) 32 - 10107 (18) 3237 (20) 66021 (12) 33 - 21077 (17) 13527 (21) 61682 (12) 33 - 33685 (19) 6939 (23) 62927 (13) 33	Jeq
$\begin{array}{c ccccc} -10107 (18) & 3237 (20) & 66021 (12) & 3-\\ -21077 (17) & 13527 (21) & 61682 (12) & 3-\\ -33685 (19) & 6939 (23) & 62927 (13) & 3-\\ \end{array}$) (1)
-21077(17) 13527(21) 61682(12) 3 -33685(19) 6039(23) 62977(13) 3	4 (1)
-33685(19) 6030(23) 62027(13) 3	5 (1)
33003(1) $333(23) 02327(13) 35$	3 (1)
-47549 (19) 11401 (29) 60072 (14) 4	3 (1)
- 57183 (20) 895 (34) 62409 (17) 55	3 (1)
-53320(23) $-13723(36)$ $67613(17)$ 65	2 (1)
- 39576 (22) - 18545 (28) 70560 (15) 53	3 (1)
- 29853 (19) 7997 (25) 68166 (13) 4	I (1)
7255 (04) 5239 (06) 67065 (03) 4	I (1)
- 20067 (14) 27997 (17) 56086 (10) 3	5 (1)
-14599 (20) 26815 (24) 48284 (13) 4	3 (1)
- 14173 (23) 40446 (30) 42504 (16) 53	3 (1)
- 19386 (29) 55437 (26) 44688 (18) 5	7(1)
- 24769 (29) 56481 (26) 52675 (18) 65	2 (1)
- 25000 (26) 42670 (26) 58385 (15) 5	l (1)





The mean C—C bond length in the pyridine ring is 1.378(1) Å and the pyridine C—N bonds are 1.355(2) and 1.353(2) Å. The mean endocyclic pyridine angle is $120.0(1)^{\circ}$.

The maxima of electron density of H atoms were refined to the expected distances 0.94-1.06 (3) Å;



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) $-x, y - \frac{1}{2}, \frac{3}{2} - z$].

The authors thank J. Gouda and P. Kristian for preparation of crystals for measurement.

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Acta Cryst. (1990). C46, 437-439

Structure of Karanjin*

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(Received 13 October 1988; accepted 2 June 1989)

Abstract. $C_{18}H_{12}O_4$, $M_r = 292 \cdot 3$, monoclinic, $P2_1/n$, $a = 7 \cdot 196$ (3), $b = 11 \cdot 275$ (3), $c = 17 \cdot 495$ (3) Å, $\beta = 100 \cdot 70$ (2)°, $V = 1394 \cdot 8$ (7) Å³, $D_m = 1 \cdot 38$ (2), $D_x = 1 \cdot 39$ g cm⁻³, Z = 4, λ (Cu $K\alpha$) = 1 · 5418 Å, $\mu = 7 \cdot 2$ cm⁻¹, T = 293 K, F(000) = 608, $R = 0 \cdot 058$, $wR = 100 \cdot 200$

0108-2701/90/030437-03\$03.00

0.066 for 1453 reflections with $I \ge 2\sigma(I)$. 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-

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

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