

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), $-154.2(4)^\circ$, and C(11)—C(12)—C(16)—O(3), $-142.9(3)^\circ$, 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)-1*H*-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*)...N(3)(1 - *x*, -*y*, 1 - *z*).

Acta Cryst. (1990). **C46**, 435–437

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

BY J. HAŠEK AND K. HUML

Institute of Macromolecular Chemistry, Czechoslovak Academy of Sciences, 162 06 Prague 6, Czechoslovakia

AND L. KUTSCHABSKY

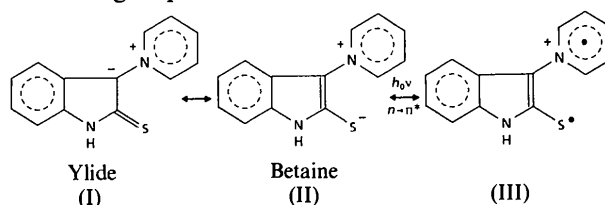
Zentralinstitut für Molekularbiologie der Akademie der Wissenschaften der DDR, Berlin DDR-1115, German Democratic Republic

(Received 13 February 1989; accepted 10 May 1989)

Abstract. C₁₃H₁₀N₂S, *M_r* = 226.3, monoclinic, *P*2₁/*c*, *a* = 10.037(4), *b* = 8.076(3), *c* = 14.124(4) Å, β = 104.59(3)°, *V* = 1107.9(6) Å³, *Z* = 4, *D_m* = 1.34(1), *D_x* = 1.36 g cm⁻³, λ(Mo Kα) = 0.71069 Å, μ = 2.6 cm⁻¹, *F*(000) = 472, *T* = 295 K, *R* = 0.039 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-

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.



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

© 1990 International Union of Crystallography

References

- ALDER, R. W., BOWMAN, P. S., STEELE, W. R. S. & WINTERMAN, D. R. (1968). *J. Chem. Soc. Chem. Commun.* pp. 723–724.
- BACHECHI, F. & ZAMBONELLI, L. (1972). *Acta Cryst.* **B28**, 2489–2492.
- EINSPAHR, H., ROBERT, J. B., MARSH, R. E. & ROBERTS, J. D. (1973). *Acta Cryst.* **B29**, 1611–1616.
- HONDA, K., NAKANISHI, H., NAGAWA, Y. & YABE, A. (1984). *J. Chem. Soc. Chem. Commun.* pp. 450–451.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MAK, T. C. W. & TROTTER, J. (1965). *Acta Cryst.* **18**, 68–74.
- NAGAWA, Y., GOTO, M., HONDA, K. & NAKANISHI, H. (1986). *Acta Cryst.* **C42**, 478–480.
- NAGAWA, Y., GOTO, M., HONDA, K. & NAKANISHI, H. (1987). *Acta Cryst.* **C43**, 147–149.
- NAGAWA, Y., GOTO, M., HONDA, K. & NAKANISHI, H. (1988). *Bull. Chem. Soc. Jpn.* **61**, 3553–3557.
- NAGAWA, Y., HONDA, K. & NAKANISHI, H. (1987). *Synthesis*, pp. 905–906.
- SAKURAI, T. & KOBAYASHI, K. (1979). *Rep. Inst. Phys. Chem. Res.* **55**, 69–77.

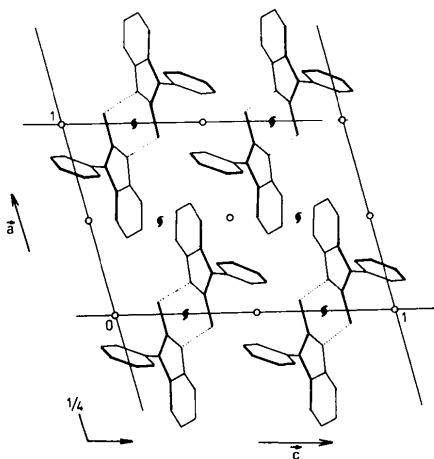


Fig. 2. The molecular packing: N—H...S bridges are denoted by dotted lines.

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.

References

- GONDA, J. & KRISTIAN, P. (1988). *Collect. Czech. Chem. Commun.* **53**, 1761–1769.
- GONDA, J., KRISTIAN, P. & IMRICH, J. (1987). *Collect. Czech. Chem. Commun.* **52**, 2508–2520.
- HAŠEK, J. (1981). *IMC* program package. In *Experimental Techniques in X-ray and Neutron Structure Analysis* (in Czechoslovakian). Institute of Macromolecular Chemistry, Praha, Czechoslovakia.
- HAŠEK, J. (1985). *Acta Cryst.* **C41**, 583–586.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCO, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- NARDELLI, M. (1983). *Comput. Chem.* **3**, 95–98.
- ROBERTS, D. & SHELDRIK, G. M. (1976). *XANADU*. Program for crystallographic calculations. Univ. of Cambridge, England.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1990). **C46**, 437–439

Structure of Karanjin*

BY MEENA HARIHARAN AND S. S. RAJAN†

Department of Biophysics and Crystallography,‡ University of Madras, Guindy Campus, Madras-600 025, India

(Received 13 October 1988; accepted 2 June 1989)

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.

† To whom correspondence should be addressed.

‡ DCB contribution No. 744.