Acta Cryst. (1982). B**38**, 2947–2948

6,7-Dimethoxy-2,3-dimethyl-1(2*H*)-isoquinolinone

BY M. P. GUPTA AND KRISHNA BANERJEE

Department of Physics, Ranchi University, Ranchi-834008, *India*

(*Received* 9 *February* 1982; *accepted* 7 *June* 1982)

Abstract. $C_{13}H_{15}NO_3$, monoclinic, $P2_1/c$, a = 10·663 (11), b = 13·809 (5), c = 8·206 (9) Å, β = 110·95 (7)° (with λ = 0·7107 Å), Z = 4, D_m = 1·36, D_x = 1·37 g cm^{-3}. R = 0·080, R_w = 0·081 for 1983 reflexions. The central aromatic ring atoms can be divided into two parts [C(1), N, C(3), C(4), C(10), C(9) and C(5), C(6), C(7), C(8), C(9), C(10)] and the two rings subtend an angle of 2·1 (3)°.

Introduction. The intensity data were collected at room temperature (297–299 K) on a CAD-4 (Enraf–Nonius) single-crystal diffractometer using Mo $K\alpha$ radiation and the $\omega/2\theta$ scan technique. Out of 1983 independent reflexions collected, 1306 were considered useful for crystal structure work with $I > 3\sigma(I)$. The structure was solved by direct determination of phases using the program system *MULTAN* (Germain, Main & Woolfson, 1971). A three-dimensional E map gave three aromatic rings, while two were expected. With peaks of doubtful value discarded and a plausible stereochemistry for the molecule assumed, a weighted Fourier synthesis gave all the atoms in the molecule, except the H atoms. Full-matrix least-squares refinement with isotropic thermal parameters and block-diagonal least-squares refinement with anisotropic thermal parameters, and with weights $w = 1/(\Delta F)^2$ where $\Delta F = ||F_o| - |F_c||$, brought the conventional unweighted and weighted R factors to 0·080 and 0·081 respectively for all reflexions. The rather high R factor is a consequence of poor-quality crystals. The H atoms were located from a difference Fourier synthesis.

The atomic positions are given in Table 1.*

Table 1. *Final atomic coordinates* (×10^5 *for non-hydrogen atoms*, ×10^3 *for hydrogen atoms*) *and isotropic temperature factors* (Å^2) *with estimated standard deviations in parentheses*

	x	y	z	B_{eq}† or B_{iso}
O(1)	22848 (22)	7240 (18)	8835 (28)	5·3
O(2)	73797 (19)	9832 (16)	42675 (22)	4·1
O(3)	83846 (19)	15213 (16)	19954 (23)	4·3
C(1)	28325 (28)	9690 (21)	−1427 (35)	3·7
C(3)	26178 (30)	14184 (20)	−31259 (34)	3·7
C(4)	39311 (30)	15922 (21)	−26310 (34)	3·7
C(5)	61984 (28)	15879 (20)	−3199 (32)	3·5
C(6)	70277 (28)	14207 (20)	13750 (32)	3·5
C(7)	64477 (29)	11209 (19)	26086 (32)	3·5
C(8)	51053 (27)	9896 (21)	21162 (33)	3·6
C(9)	42591 (28)	11405 (19)	3633 (32)	3·3
C(10)	48134 (28)	14456 (19)	−8590 (31)	3·3
C(11)	16798 (33)	15474 (25)	−49816 (38)	4·8
C(12)	6290 (34)	8946 (27)	−24262 (45)	5·3
C(13)	89994 (31)	16417 (26)	7151 (39)	4·8
C(14)	68411 (34)	7068 (28)	55581 (38)	5·2
N	20621 (22)	10973 (17)	−19062 (28)	3·7
H(4)	428 (3)	182 (2)	−345 (4)	2·9 (9)
H(5)	660 (3)	183 (2)	−106 (4)	3·3 (10)
H(8)	463 (3)	76 (3)	286 (5)	3·8 (10)
H(11)	122 (4)	92 (3)	−558 (5)	4·8 (12)
H(11′)	219 (4)	175 (3)	−564 (5)	5·4 (12)
H(11″)	98 (3)	201 (2)	−511 (5)	3·3 (10)
H(12)	39 (5)	44 (4)	−340 (7)	10·5 (20)
H(12′)	6 (5)	143 (3)	−280 (6)	6·8 (14)
H(12″)	38 (4)	49 (3)	−168 (5)	4·7 (11)
H(13)	871 (3)	229 (2)	14 (4)	2·6 (10)
H(13′)	871 (5)	102 (4)	−6 (7)	9·0 (20)
H(13″)	1002 (4)	164 (3)	137 (5)	5·1 (12)
H(14)	622 (4)	6 (3)	525 (5)	4·7 (11)
H(14′)	611 (4)	123 (3)	570 (5)	5·0 (12)
H(14″)	753 (3)	61 (2)	653 (5)	3·7 (12)

† $B_{eq} = \frac{4}{3}(B_{11}a^2 + B_{22}b^2 + B_{33}c^2 + B_{13}ac\cos\beta)$.

Discussion. Fig. 1 shows the labelling of the atoms and the intramolecular bond lengths and angles. The distances C(3)–C(4), C(5)–C(6), C(7)–C(8) and C(9)–C(10) show double-bond character whereas the other C–C distances in the isoquinolinoid nucleus are long. This type of short–long pattern is also observed in some other isoquinolinone derivatives (Ammon & Wheeler, 1974). The two C–N distances of 1·397 and 1·405 Å in the pyridine ring are very close to similar bonds in 2-methyl-1-phenyl-3-isoquinolone (Ammon & Wheeler, 1974) but longer than the 1·340 Å C–N distance found in pyridine itself (Bak, Hansen-Nygaard & Rastrup-Andersen, 1958). This type of lengthening of C–N distances is probably caused by the methyl substituent with a C–N distance of 1·459 Å, which is close to that found in 2-methyl-1-phenyl-3-isoquinolone (Ammon & Wheeler, 1974). The other C–O and C–C distances for the methoxy and methyl groups are close to the expected values.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36988 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0567-7408/82/112947-02$01.00

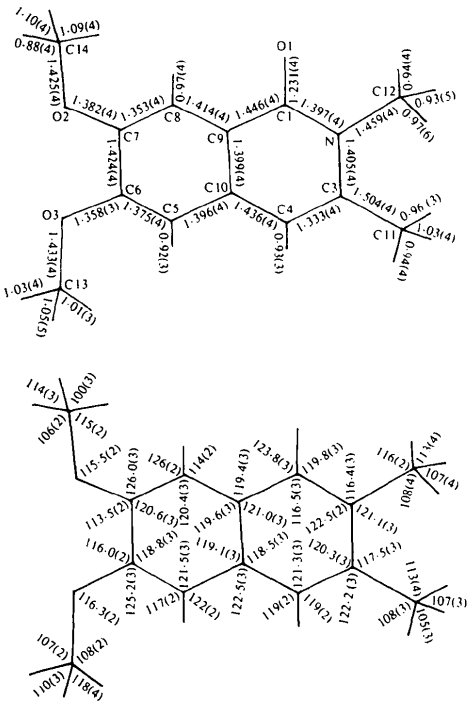

Fig. 1. Bond lengths (Å) and angles (°) in the title compound, with e.s.d.'s in parentheses.

The planes through atoms C(5), O(3), C(13) and through C(7), O(2), C(14) are inclined at 11·4 (3) and 3·1 (3)° respectively to the mean plane through the ring atoms. The molecule has a nearly planar quinolinone system in which the two six-membered rings are inclined at 2·1 (3)° – as was also found in 2-(2,6-dichlorobenzyl)-1-isoquinolone (Ammon & Wheeler, 1974).

The molecules in the crystal lie nearly parallel to the (010) plane. Molecules in adjacent layers are oppositely oriented in parallel planes with their methoxy groups facing each other as a consequence of the centre of symmetry and are separated by a distance of $b/4$. The typical nature of the layer structure explains the very strong reflexion 040.

In terms of molecular packing the structure is not a strongly bonded one. This explains the easy deformation in the crystal, with soft texture and also large thermal motion.

The authors gratefully acknowledge the award of a Teacher Fellowship to KB by the University Grants Commission of India and the gift of the crystal by Professor J. N. Chatterjee, Department of Chemistry, Patna University, Patna, India.

References

AMMON, H. L. & WHEELER, G. L. (1974). *Acta Cryst.* B**30**, 1146–1154.

BAK, B., HANSEN-NYGAARD, L. & RASTRUP-ANDERSEN, J. (1958). *J. Mol. Spectrosc.* **2**, 361–368.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

Acta Cryst. (1982). B**38**, 2948–2951

Structure of the Anti-inflammatory Drug 4-Hydroxy-2-methyl-*N*-2-pyridyl-2*H*-1λ⁶,2-benzothiazine-3-carboxamide 1,1-Dioxide (Piroxicam)

BY BISERKA KOJIĆ-PRODIĆ AND ŽIVA RUŽIĆ-TOROŠ

'Rudjer Bošković' Institute, PO Box 1016, 41001 *Zagreb, Yugoslavia*

(*Received* 20 *April* 1982; *accepted* 7 *June* 1982)

Abstract. $C_{15}H_{13}N_3O_4S$, M_r = 331·35, monoclinic, $P2_1/c$, a = 7·127 (2), b = 15·136 (7), c = 13·949 (6) Å, β = 97·35 (4)°, Z = 4, U = 1491·15 Å^3, D_x = 1·481 Mg m^{-3}, Mo $K\alpha$ (λ = 0·7107 Å, μ = 0·244 mm^{-1}); final R = 0·050 for 2289 observed reflexions [$I > 2\sigma(I)$]. Bond lengths and angles are in agreement with expected values. The thiazine ring exhibits a half-chair conformation. An amide group is involved in an intramolecular hydrogen bond to the hydroxy group [O(17)–H(17)···O(15) [2·561 (3) Å]. It also forms an intermolecular hydrogen bond to an O atom bonded to the S atom [N(16)–H(16)···O(11), 3·053 (3) Å], connecting piroxicam molecules in an infinite chain along **b**. The molecular packing is also influenced by van der Waals interactions.

Introduction. Piroxicam (adopted name in USA) (CP-16171) or felden (Pfizer) is an effective analgesic and anti-inflammatory agent in rheumatoid arthritis, osteoarthritis, ankylosing spondylitis and acute pain in

0567-7408/82/112948-04$01.00