Acta Cryst. (1989). C**45**, 828–829

5-Hydroxy-7-methoxyflavone*

BY M. SHOJA

Chemistry Department, Fordham University, Bronx, NY 10458, *USA*

(*Received* 13 *September* 1988; *accepted* 14 *November* 1988)

Abstract. $C_{16}H_{12}O_4$, $M_r = 268{\cdot}27$, monoclinic, $P2_1/c$, $a = 10{\cdot}127\ (2)$, $b = 15{\cdot}270\ (3)$, $c = 8{\cdot}226\ (2)$ Å, $\beta = 92{\cdot}42\ (3)°$, $V = 1270{\cdot}9\ (5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}407$ g cm^{-3}, Cu $K\alpha$ radiation, $\lambda = 1{\cdot}5418$ Å, $\mu = 8{\cdot}5$ cm^{-1}, $F(000) = 560$, $T = 293$ K. Final $R = 0{\cdot}039$ for 979 observed reflections. The dihedral angle between the planar phenyl ring and the nonplanar γ-benzopyrone portion of the molecule is 24·8 (2)°. The methoxy group is oriented out of the plane of the benzene ring, with the torsion angle C(11)–O(4)–C(7)–C(8) −5·0 (6)°. The torsion angle O(1)–C(2)–C(1′)–C(2′) is 21·8 (5)°.

Experimental. The title compound (Fig. 1) was purchased from the Indofine Chemical Company. Crystals grown from acetonitrile solution. Data collected on an Enraf–Nonius CAD-4 diffractometer, graphite monochromator. Crystal 0·2 × 0·2 × 0·3 mm. Cell parameters measured on the diffractometer using 25 reflections in the 2θ range 20–40°. Range of indices $0 < h < 11$, $0 < k < 17$, $-9 < l < 9$ ($\theta < 60°$). Three standards 150, 35$\bar{2}$, 23$\bar{1}$, measured after every 200

* Flavone is 2-phenyl-4*H*-1-benzopyran-4-one.

Table 1. *Final fractional coordinates and equivalent isotropic temperature factors for non-*H *atoms with e.s.d.'s in parentheses*

$B_{eq} = \frac{4}{3}\sum_i\sum_j B_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}(Å^2)
O(1)	0·3371 (3)	0·1269 (2)	0·3320 (3)	2·9 (1)
O(2)	0·1795 (3)	−0·0601 (2)	0·6205 (4)	4·5 (1)
O(3)	0·3741 (3)	−0·0357 (2)	0·8294 (4)	4·5 (1)
O(4)	0·6817 (3)	0·1833 (2)	0·7291 (4)	4·1 (1)
C(1′)	0·1708 (4)	0·1160 (3)	0·1248 (5)	3·0 (1)
C(2)	0·2209 (4)	0·0861 (3)	0·2878 (5)	3·0 (1)
C(2′)	0·2555 (4)	0·1529 (3)	0·0159 (5)	3·3 (1)
C(3)	0·1653 (4)	0·0257 (3)	0·3804 (5)	3·5 (1)
C(3′)	0·2081 (5)	0·1788 (3)	−0·1375 (5)	4·0 (1)
C(4)	0·2270 (4)	−0·0007 (3)	0·5342 (5)	3·3 (1)
C(4′)	0·0753 (5)	0·1681 (3)	−0·1812 (6)	4·4 (1)
C(5)	0·4184 (4)	0·0276 (3)	0·7306 (5)	3·3 (1)
C(5′)	−0·0092 (4)	0·1325 (3)	−0·0735 (6)	4·4 (1)
C(6′)	0·0373 (4)	0·1064 (3)	0·0808 (6)	3·6 (1)
C(6)	0·5282 (4)	0·0746 (3)	0·7750 (5)	3·3 (1)
C(7)	0·5724 (4)	0·1389 (3)	0·6716 (5)	3·1 (1)
C(8)	0·5098 (4)	0·1577 (3)	0·5210 (5)	3·1 (1)
C(9)	0·3977 (4)	0·1079 (3)	0·4823 (5)	2·7 (1)
C(10)	0·3473 (4)	0·0443 (3)	0·5810 (5)	2·7 (1)
C(11)	0·7401 (5)	0·2466 (3)	0·6261 (6)	4·8 (1)

Table 2. *Bond lengths* (Å), *angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

O(1)–C(2)	1·367 (5)	C(3)–C(4)	1·444 (6)
O(1)–C(9)	1·387 (5)	C(3′)–C(4′)	1·388 (7)
O(2)–C(4)	1·259 (5)	C(4)–C(10)	1·437 (6)
O(3)–C(5)	1·352 (5)	C(4′)–C(5′)	1·370 (7)
O(4)–C(7)	1·365 (5)	C(5)–C(6)	1·360 (6)
O(4)–C(11)	1·429 (6)	C(5)–C(10)	1·422 (6)
C(1′)–C(2)	1·485 (6)	C(5′)–C(6′)	1·392 (6)
C(1′)–C(2′)	1·386 (6)	C(6)–C(7)	1·386 (6)
C(1′)–C(6′)	1·393 (6)	C(7)–C(8)	1·397 (6)
C(2)–C(3)	1·336 (6)	C(8)–C(9)	1·392 (6)
C(2′)–C(3′)	1·388 (6)	C(9)–C(10)	1·378 (6)

C(2)–O(1)–C(9)	119·1 (3)	O(3)–C(5)–C(10)	118·6 (4)
C(7)–O(4)–C(11)	118·7 (3)	C(6)–C(5)–C(10)	121·1 (4)
C(2)–C(1′)–C(2′)	120·7 (4)	C(4′)–C(5′)–C(6′)	120·4 (4)
C(2)–C(1′)–C(6′)	119·6 (4)	C(1′)–C(6′)–C(5′)	119·6 (4)
C(2′)–C(1′)–C(6′)	119·7 (4)	C(5)–C(6)–C(7)	119·4 (4)
O(1)–C(2)–C(1′)	111·0 (4)	O(4)–C(7)–C(6)	114·7 (4)
O(1)–C(2)–C(3)	122·8 (4)	O(4)–C(7)–C(8)	122·4 (4)
C(1′)–C(2)–C(3)	126·2 (4)	C(6)–C(7)–C(8)	122·9 (4)
C(1′)–C(2′)–C(3′)	120·2 (4)	C(7)–C(8)–C(9)	115·2 (4)
C(2)–C(3)–C(4)	121·0 (4)	O(1)–C(9)–C(8)	114·6 (3)
C(2′)–C(3′)–C(4′)	119·8 (4)	O(1)–C(9)–C(10)	120·7 (3)
O(2)–C(4)–C(3)	122·2 (4)	C(8)–C(9)–C(10)	124·7 (4)
O(2)–C(4)–C(10)	122·3 (4)	C(4)–C(10)–C(5)	122·6 (4)
C(3)–C(4)–C(10)	115·5 (4)	C(4)–C(10)–C(9)	120·7 (4)
C(3′)–C(4′)–C(5′)	120·3 (4)	C(5)–C(10)–C(9)	116·7 (4)
O(3)–C(5)–C(6)	120·3 (4)		

O(1)–C(2)–C(3)–C(4)	−0·6 (6)	C(2)–C(3)–C(4)–C(10)	2·1 (6)
C(3)–C(4)–C(10)–C(5)	178·4 (4)	C(3)–C(4)–C(10)–C(9)	−0·2 (6)
C(4)–C(10)–C(9)–O(1)	−3·1 (6)	C(4)–C(10)–C(5)–C(6)	−176·6 (4)
C(10)–C(9)–O(1)–C(2)	4·6 (5)	C(10)–C(5)–C(6)–C(7)	−1·0 (6)
C(5)–C(6)–C(7)–C(8)	−0·6 (7)	C(6)–C(7)–C(8)–C(9)	0·9 (6)
C(7)–C(8)–C(9)–C(10)	0·5 (6)	C(7)–C(8)–C(9)–O(1)	−180·0 (3)
C(8)–C(9)–C(10)–C(4)	176·8 (4)	C(8)–C(9)–C(10)–C(5)	−1·9 (6)

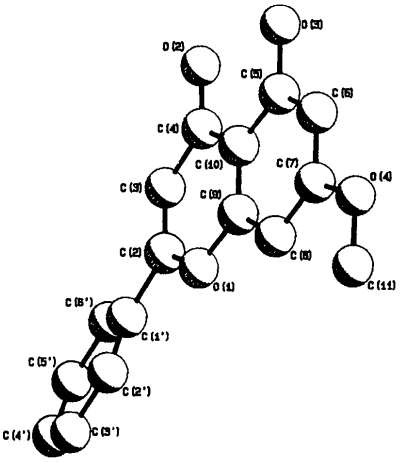

Fig. 1. Numbering of atoms and conformation of the molecule.

0108-2701/89/050828-02$03.00

reflections, showed a variation of 0·2%. No absorption corrections. Lorentz and polarization corrections. 1884 unique reflections measured. 979 observed reflections with $I > 3{\cdot}0\sigma(I)$. Direct methods (*MULTAN*82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) used for structure determination. H atoms located by difference Fourier synthesis. Anisotropic full-matrix least-squares refinement (on F) for non-H atoms, isotropic for H atoms. $\sum w(|F_o| - |F_c|)^2$ minimized. $R = 0{\cdot}039$, $wR = 0{\cdot}040$, max. $\Delta/\sigma = 0{\cdot}89$. Max. peak height in the final difference Fourier map 0·34 e Å^{-3}, $S = 0{\cdot}94$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Enraf–Nonius *SDP* (Frenz, 1984).

Atomic parameters are given in Table 1,* bond distances, bond angles, and relevant torsion angles are presented in Table 2. Atomic numbering is shown in Fig. 1 and molecular packing in Fig. 2.

Related literature. The title compound is the first reported structure in the systematic study of the conformational preferences of methoxy derivatives of flavone, isoflavone and flavanone which is now being undertaken in our laboratory. Probably because of the difficulty in growing good quality crystals only a few structure determinations have been made. In 3′,5,5′,6-tetramethoxyflavone the dihedral angle between the phenyl ring and the benzopyrone portion of the molecule is 28° (Ting & Watson, 1972), whereas in 4′,5,7-trihydroxyisoflavone the reported dihedral angle is 53·2° (Breton, Precigoux, Courseille & Hospital, 1975). In 5,7,4′-trimethoxyflavanone the value for the dihedral angle is 70·8° (Mariezcurrena, 1978).

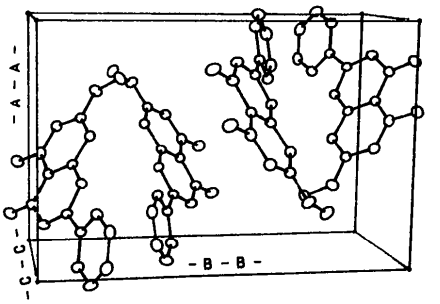

Fig. 2. Molecular packing diagram.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51595 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

BRETON, P. M., PRECIGOUX, G., COURSEILLE, C. & HOSPITAL, M. (1975). *Acta Cryst.* B**31**, 921–923.

FRENZ, B. A. (1984). *Structure Determination Package.* College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.

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., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MARIEZCURRENA, R. A. (1978). *Acta Cryst.* B**34**, 2322–2324.

TING, H. & WATSON, W. H. (1972). *Acta Cryst.* B**28**, 1046–1051.

Acta Cryst. (1989). C**45**, 829–831

4,*N*-Dimethyl-2-nitrosoaniline

BY R. G. PRITCHARD, G. S. HEATON AND I. M. EL-NAHHAL

Department of Chemistry, University of Manchester Institute of Science and Technology, PO Box 88, *Manchester M*60 1*QD, England*

(*Received* 30 *September* 1988; *accepted* 10 *November* 1988)

Abstract. $C_8H_{10}N_2O$, $M_r = 150{\cdot}18$, triclinic, $P\bar{1}$, $a = 8{\cdot}636$ (3), $b = 8{\cdot}631$ (3), $c = 11{\cdot}101$ (2) Å, $\alpha = 92{\cdot}57$ (2), $\beta = 94{\cdot}12$ (2), $\gamma = 105{\cdot}02$ (2)°, $V = 795$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}25$ Mg m^{-3}, $F(000) = 320$, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}05$ mm^{-1}, $T = 293$ K, $R = 0{\cdot}043$ for 2170 observed reflexions [$F \geq 3\sigma(F)$]. The crystal contains two crystallographically distinct but similarly conformed molecules. Both are planar and the configuration facilitates weak intramolecular hydrogen bonding between the nitroso oxygen and amine hydrogen atom [N···O 2·619 (5), 2·592 (5); O··· H 2·01 (4), 1·97 (4) Å; N–H···O 128 (1), 128 (1)°]. The considerable 3,5-diene character within the rings and short extra-annular C–N bonds indicate a dipole, whose positive and negative poles are focused at the amine and nitrosyl substituents respectively.

Experimental. Green crystals suitable for X-ray work were prepared by nitrosating 2-acetoxymercurio-4,*N*-dimethylaniline with sodium nitrite in concentrated

0108-2701/89/050829-03$03.00