acetate is gt. The dihedral angle between the plane formed by O(5),C(2),C(3),C(5) and the acetyl group formed by O(4),O(6),C(7),C(8) is 67 (1)°.

The methoxycarbonylmethyl substituent is in an equatorial position. Its conformation is described by C(2)–C(3)–C(11)–C(12) = $-179\cdot6$ (3), C(4)–C(3)–C(11)–C(12) = $56\cdot3$ (4) and C(3)–C(11)–C(12)–O(2) = $39\cdot3$ (5)°. The group of atoms O(2),O(3),-C(12),C(13) is planar and is inclined at 68 (1)° from the plane formed by O(5),C(2),C(3),C(5).

The ethenyl group is attached at C(4) in an axial position. The atoms C(4),C(9),C(10),H(9),H(101) and H(102) are coplanar. This plane is at 93 (2)° from the pyranose mean plane [O(5),C(2),C(3),C(5)].

In the crystal the molecules are held by hydrogen bonds between the anomeric hydroxyl O(1)–H(O1) and the ring oxygen O(5) where O(1)–H(O1) = $0\cdot81$ (4), H(O1)$\cdots$O(5') = $2\cdot02$ (4), O(1)$\cdots$O(5') = $2\cdot823$ (3) Å, O(1)–H(O1)$\cdots$O(5') = 174 (4)°, and O(5') is related to O(5) by ($\frac{1}{2}-x$, $-\frac{1}{2}-y$, $2-z$). This hydrogen bond is comparable to those observed in α-DL-fucopyranose, $2\cdot804$ (3) Å (Longchambon & Gillier-Pandraud, 1977), α-D-glycopyranose, $2\cdot849$ (2) Å (Brown & Levy, 1979), and α-D-talopyranose, $2\cdot844$ (2) Å (Ohanessian, Avenel, Kanters & Smits, 1977).

References

AHMED, F. R., HALL, S. R., PIPPY, M. E. & HUBER, C. P. (1973). NRC Crystallographic Computer Programs for the IBM/360. Accession Nos. 133–147 in *J. Appl. Cryst.* **6**, 309–346.

ARNOTT, S. & SCOTT, W. E. (1972). *J. Chem. Soc. Perkin Trans.* 2, pp. 324–335.

BROWN, G. M. & LEVY, H. A. (1979). *Acta Cryst.* B35, 656–659.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A24, 321–324.

DOEDENS, R. J. & IBERS, J. A. (1967). *Inorg. Chem.* **6**, 204–210.

HANESSIAN, S. (1983). *Total Synthesis of Natural Products: The 'Chiron' Approach*, edited by J. E. BALDWIN. Oxford: Pergamon Press.

HANESSIAN, S. & LÉGER, S. (1983). Unpublished results.

JEFFREY, G. A., POPLE, J. A. & RADOM, L. (1974). *Carbohydr. Res.* **38**, 81–95.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

LIPSHUTZ, B. H., WILHELM, R. S. & FLOYD, D. M. (1981). *J. Am. Chem. Soc.* **103**, 7672–7674.

LONGCHAMBON, F. & GILLIER-PANDRAUD, H. (1977). *Acta Cryst.* B33, 2094–2097.

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.

OHANESSIAN, J., AVENEL, D., KANTERS, J. A. & SMITS, D. (1977). *Acta Cryst.* B33, 1063–1066.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

SUNDARALINGAM, M. (1968). *Biopolymers*, **6**, 189–213.

5,7-Dimethoxy-1-indanone, $C_{11}H_{12}O_3$

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Abstract. $M_r = 190\cdot2$, monoclinic, $P2_1/n$, $a = 8\cdot695$ (2), $b = 9\cdot782$ (1), $c = 11\cdot189$ (2) Å, $\beta = 90\cdot68$ (3)°, $V = 951\cdot6$ (5) Å³, $Z = 4$, $D_m = 1\cdot34$, $D_x = 1\cdot327$ Mg m⁻³, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 0\cdot105$ mm⁻¹, $F(000) = 400$, room temperature, $R_w = 0\cdot042$ for 1417 observed [$I > 2\sigma(I)$] reflections. The molecular geometry is normal with the aromatic ring quite planar, but not the five-membered ring. The latter is close to a C_s conformation with the pseudo-mirror plane through C=O and the middle of the C(3)–C(9) bond. The fragments H₃C–O–C(aryl)–C(aryl) have a staggered synperiplanar conformation.

Introduction. The crystal structure analysis of the title compound (Fig. 1) was undertaken to establish the disposition and conformation of the two methoxy groups *vis à vis* the aromatic ring. A private communication from the Cambridge Structural Database had indicated an earlier determination (Gupta & Ram, 1979) to be incorrect, inasmuch as the packing of the molecules showed very short contacts of the nature of polymeric linkages and this is chemically unacceptable. The present work is, therefore, a new determination.

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Experimental. Compound synthesized according to Chatterjee, Mukherjee & Sengupta (1975), recrystallized from methanol, m.p. 373–374 K, previously reported by Haisgen, Seidl & Wimmer (1964), m.p. 371·6–372·6 K. D_m by flotation in $CHBr_3$–toluene. Cell dimensions from setting angles of 25 reflections. Three intensity and orientation control reflections measured every 120 min and 200 reflections, no significant drift. 1897 independent reflections, $\theta_{max} = 27°$, Enraf–Nonius CAD-4 diffractometer, Zr-filtered Mo radiation, pure ω scan, 1417 reflections with $I > 2\sigma(I)$ used in analysis; h 0–10, k 0–11, l −13–12. Low absorption coefficient and size of crystal (0·5 × 0·2 × 0·2 mm) made absorption corrections unnecessary. Structure solved by *MULTAN* (Germain, Main & Woolfson, 1971); the most likely E map showed all non-hydrogen atoms. Subsequent difference Fourier synthesis, with carbon and oxygen atom contributions subtracted, revealed positions of all H atoms. Least-squares refinement on F, Gauss–Seidel block method (Sparks, 1974) and Enraf–Nonius *SDP* package (Frenz, 1978), each reflection given a weight based on counting statistics; Debye–Waller temperature parameters fixed at 4·0 Å^2 for all H atoms; no extinction correction. $R = 0·044$, $R_w = 0·042$, $S = 4·35$. Max. $\Delta/\sigma = 0·05$; max. peak in final difference map 0·10 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Refined atomic coordinates are listed in Table 1, the atoms being numbered as in Fig. 1.*

Bond lengths and valence angles, given in Table 2, are normal. The aromatic ring is planar within the limits of error, the greatest deviation of atoms from the least-squares fitted plane being 0·005 (2) Å. Atoms C(2) and C(3) are almost in the plane, but C(1) is 0·039 (2) Å out of this plane. Consequently, the five-membered ring has a slightly puckered envelope conformation $(C_s$ symmetry) with the pseudo-mirror plane passing through C=O and the middle of the C(3)–C(9) bond. Relevant torsion angles as well as Cremer & Pople (1975) ring parameters are presented in Table 2.

C(10), O(2), C(11) and O(3) are approximately coplanar with the aromatic ring, while C(10) and C(11) are both turned towards C(6) (see Fig. 1). The torsion angles C(10)O(2)C(5)C(6) and C(11)O(3)C(7)C(6) are 1·1 (4) and −0·3 (4)°, respectively. H atoms in the methyl groups are staggered with respect to their O–C(aryl) bonds. This seems to be the normal orientation of an OCH_3 adjacent to a double bond.

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* Lists of structure factors, hydrogen positions, CH lengths, CCH and OCH valence angles and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39406 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional parameters in fractions of cell edges with e.s.d.'s in parentheses*

Equivalent isotropic temperature factors (Å^2) of non-hydrogen atoms are calculated from anisotropic temperature parameters assuming equal volume of the 50% probability region; $B_{eq} = 8\pi^2(U_{11}^o U_{22}^o U_{33}^o)^{1/3}$ was calculated according to Lipson & Cochran (1966). All anisotropic thermal parameters were physically acceptable.

	x	y	z	B_{eq}(Å^2)
O(1)	0·1045 (2)	0·1321 (2)	−0·2352 (1)	4·61
O(2)	0·4450 (2)	−0·0571 (2)	0·2585 (1)	4·21
O(3)	0·2803 (2)	0·2678 (1)	−0·0414 (1)	3·54
C(1)	0·1261 (2)	0·0369 (2)	−0·1674 (2)	3·44
C(2)	0·0650 (3)	−0·1071 (2)	−0·1874 (2)	3·88
C(3)	0·1265 (3)	−0·1940 (2)	−0·0850 (2)	3·84
C(4)	0·2936 (3)	−0·1264 (2)	0·0973 (2)	3·58
C(5)	0·3707 (2)	−0·0211 (2)	0·1557 (2)	3·28
C(6)	0·3693 (2)	0·1119 (2)	0·1119 (2)	3·11
C(7)	0·2903 (2)	0·1402 (2)	0·0067 (2)	2·99
C(8)	0·2135 (2)	0·0350 (2)	−0·0544 (2)	2·91
C(9)	0·2164 (2)	−0·0965 (2)	−0·0071 (2)	3·15
C(10)	0·5215 (3)	0·0467 (2)	0·3243 (2)	4·39
C(11)	0·3552 (3)	0·3760 (2)	0·0217 (2)	4·31

Table 2. *Bond lengths (Å), angles (°) and selected torsion angles (°)*

E.s.d.'s are given in parentheses for bond lengths and angles; e.s.d.'s are ~0·4° for torsion angles.

The Cremer & Pople (1975) ring-shape parameters of the five-membered ring [C(1)C(2)C(3)C(9)C(8)] are given; e.s.d.'s are calculated according to Norrestam (1981).

C(1)–O(1)	1·214 (1)		C(5)–C(6)	1·391 (1)
C(1)–C(2)	1·521 (2)		C(6)–C(7)	1·385 (1)
C(1)–C(8)	1·467 (1)		C(7)–O(3)	1·361 (1)
C(2)–C(3)	1·518 (2)		C(7)–C(8)	1·400 (1)
C(3)–C(9)	1·505 (1)		C(8)–C(9)	1·392 (1)
C(4)–C(5)	1·388 (1)		C(10)–O(2)	1·415 (1)
C(4)–C(9)	1·372 (1)		C(11)–O(3)	1·392 (1)
C(5)–O(2)	1·359 (1)			

O(1)–C(1)–C(2)	124·6 (1)	O(3)–C(7)–C(8)	116·9 (1)
O(1)–C(1)–C(8)	128·5 (1)	C(6)–C(7)–C(8)	119·8 (1)
C(2)–C(1)–C(8)	106·9 (1)	C(1)–C(8)–C(7)	130·6 (1)
C(1)–C(2)–C(3)	106·8 (1)	C(1)–C(8)–C(9)	110·3 (1)
C(2)–C(3)–C(9)	105·0 (1)	C(7)–C(8)–C(9)	119·2 (1)
C(5)–C(4)–C(9)	118·1 (1)	C(3)–C(9)–C(4)	127·2 (1)
O(2)–C(5)–C(4)	115·4 (1)	C(3)–C(9)–C(8)	111·0 (1)
O(2)–C(5)–C(6)	122·9 (1)	C(4)–C(9)–C(8)	121·8 (1)
C(4)–C(5)–C(6)	121·8 (1)	C(5)–O(2)–C(10)	118·0 (1)
C(5)–C(6)–C(7)	119·3 (1)	C(7)–O(3)–C(11)	117·3 (1)
O(3)–C(7)–C(6)	123·2 (1)		

C(8)–C(1)–C(2)–C(3)	−2·7
C(1)–C(2)–C(3)–C(9)	1·9
C(2)–C(3)–C(9)–C(8)	−0·5
C(3)–C(9)–C(8)–C(1)	−1·2
C(9)–C(8)–C(1)–C(2)	2·4

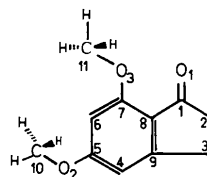|	
$Q(2)$	0·025 (5) Å
$\varphi(2)$	12 (11)°

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

Similar staggered synperiplanar conformations were observed in, for example, solid 4-*tert*-butyl-3,5-dinitro-anisole (Van Havere, Lenstra & Geise, 1982), solid 5-*endo*-(bromomethyl)-1,2,4-trichloro-3,7,7-trimethoxy-norborn-2-ene (Van Hemelrijk, Lenstra & Geise, 1982) and gaseous methyl vinyl ether (Durig & Compton, 1978; Pyckhout, Van Nuffel, Van Alsenoy, Van den Enden & Geise, 1983).

One of us (MPG) is grateful to the Board of the University of Antwerp (UIA) and to the Belgian National Science Foundation (NFWO) for a visiting professorship, during the tenure of which the above work was done.

References

CHATTERJEE, J. N., MUKHERJEE, J. & SENGUPTA, S. C. (1975). *Indian J. Chem.* **13**, 859–860.

CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

DURIG, J. R. & COMPTON, D. A. (1978). *J. Chem. Phys.* **69**(5), 2028–2035.

FRENZ, B. A. (1978). *The Enraf–Nonius CAD-4 SDP.* In *Computing in Crystallography*, edited by H. SCHENK, R. OLTHOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI, pp. 64–71. Delft Univ. Press.

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

GUPTA, M. P. & RAM, P. (1979). *Indian J. Phys.* **53A**, 474–476.

HAISGEN, R., SEIDL, G. & WIMMER, I. (1964). *Justus Liebigs Ann. Chem.* **677**, 21.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

LIPSON, H. & COCHRAN, W. (1966). *The Determination of Crystal Structures*, pp. 300 *ff.* London: Bell.

NORRESTAM, P. (1981). *Acta Cryst.* A**37**, 764–765.

PYCKHOUT, W., VAN NUFFEL, P., VAN ALSENOY, K., VAN DEN ENDEN, L. & GEISE, H. J. (1983). *J. Mol. Struct.* **102**, 333–345.

SPARKS, R. A. (1974). *Least-Squares Tutorial.* Proc. Am. Crystallogr. Assoc. Meet., Berkeley, California, pp. 99 *ff.*

VAN HAVERE, W., LENSTRA, A. T. H. & GEISE, H. J. (1982). *Acta Cryst.* B**38**, 3119–3120.

VAN HEMELRIJK, D., LENSTRA, A. T. H. & GEISE, H. J. (1982). *Acta Cryst.* B**38**, 2760–2763.

Structure of 4-Amino-*N*-2-pyridylbenzenesulphonamide (Sulphapyridine), C$_{11}$H$_{11}$N$_3$O$_2$S

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Abstract. $M_r = 249 \cdot 29$, monoclinic, $C2/c$, $a = 12 \cdot 807$ (3), $b = 11 \cdot 711$ (3), $c = 15 \cdot 379$ (2) Å, $\beta = 94 \cdot 07$ (2)°, $V = 2300 \cdot 8$ (9) Å^3, $Z = 8$, $D_m = 1 \cdot 441$, $D_x = 1 \cdot 439$ Mg m^{-3}, $F(000) = 1040$, Mo $K\alpha$, $\lambda = 0 \cdot 7107$ Å, $\mu = 0 \cdot 273$ mm^{-1}, $T = 298$ K, final $R = 0 \cdot 041$ for 2160 observed reflections. The molecule is found to exist as the imido tautomer. Its structural parameters are compared with those of other related molecules. In packing, centrosymmetrically related molecules dimerize through N—H$\cdots$O hydrogen bonds; additional intra- and intermolecular N—H$\cdots$O and N—H$\cdots$N hydrogen bonds stabilize the structure. The pyridine and the phenyl rings stack independently.

Introduction. Sulphapyridine is an N^1-heteroaromatic sulphonamide used as an antibacterial drug in the treatment of pneumonia (Nitya Anand, 1979). The X-ray structural study of the compound has been carried out as part of our studies of N^1-substituted sulphonamides which we have undertaken with the view of correlating the structural features with the anti-bacterial activities (Basak, Mazumdar & Chaudhuri, 1982, 1983, 1984). Our study has shown that in the crystalline state the molecule exists as the imido tautomer $H_2N–C_6H_4SO_2N=C_5H_5N$. In addition, the molecular geometry and the conformation are compared with that of a structurally similar compound, ethyl 3-[4,5-dimethoxy-2-(4-methyl-2-pyridyl-sulphamoyl)phenyl]propionate (Eliopoulos, Sheldrick & Homodrakas, 1983).

Experimental. Crystals obtained from hot methanol and from acetone; crystals from acetone unstable. Density by flotation (KI solution). Space group Cc or $C2/c$ (hkl: $h+k =$ odd absent, $h0l$: l odd absent); ambiguity resolved by $N(z)$ test, E statistics and finally by successful refinement. Crystal $0 \cdot 30 \times 0 \cdot 30 \times 0 \cdot 35$ mm. Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$. Lattice parameters from 25 reflections ($16° \le \theta \le 20°$). 2503 unique reflections ($2° \le \theta \le 27°$, $-16 \le h \le 16$, $0 \le k \le 14$, $0 \le l \le 19$), 2160 observed with $I \ge 3\sigma(I)$. Centring monitored after every 50 reflections (590, 0,2,14 and 6,2,12), intensity monitored after every hour of exposure ($\overline{3},5,11$, 596 and 2,4,13); data corrected for Lp and variation in intensity ($<2\%$), absorption ignored. Direct methods