

**Ethyl 3-[4,5-Dimethoxy-2-(4-methyl-2-pyridylsulphamoyl)phenyl]propionate,  
C<sub>19</sub>H<sub>24</sub>N<sub>2</sub>O<sub>6</sub>S**

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**Abstract.**  $M_r = 408.47$ ,  $P\bar{1}$ ,  $a = 9.473$  (1),  $b = 10.281$  (1),  $c = 11.174$  (1) Å,  $\alpha = 88.648$  (7),  $\beta = 72.011$  (9),  $\gamma = 79.526$  (7)°,  $V = 1017.23$  Å<sup>3</sup>,  $Z = 2$ ,  $D_m = 1.34$ ,  $D_x = 1.33$  Mg m<sup>-3</sup>,  $F(000) = 432$ , Ni-filtered Cu K $\alpha$ ,  $\lambda = 1.5418$  Å,  $\mu = 1.632$  mm<sup>-1</sup>,  $T = 293$  K,  $R = 0.084$  for 2966 observed reflections. The methoxy groups are nearly coplanar with the phenyl ring [maximum deviation from plane 0.18 (18) Å], and pairs of molecules are connected by N–H...N hydrogen bonds across a centre of symmetry.

**Introduction.** Derivatives of benzothiazinone have anti-inflammatory activity (Lombardino & Wiseman, 1971) and are known to affect the central nervous system (Sianesi, Redaelli, Magistretti & Massarani, 1973).

Sulphonamides, similar in structure to benzothiazinone, have been synthesized (Camoutsis & Catsoulakos, 1976, 1980; Catsoulakos & Camoutsis, 1980) and are being tested for pharmacological properties.

Crystallographic studies of a series of these compounds are being carried out (Hamodrakas & Filippakis, 1977; Hamodrakas, Filippakis & Sheldrick, 1977; Hamodrakas, Akrigg, Sheldrick & Camoutsis, 1978; Perdikatsis, Catsoulakos & Filippakis, 1979; Panagiotopoulos & Filippakis, 1980; Panagiotopoulos, Filippakis & Catsoulakos, 1980) in attempts to relate structure and activity. The title compound is a member of this series.

**Experimental.** Crystal 0.25 × 0.32 × 1.00 mm, 7715 reflections collected, Enraf–Nonius CAD-4 diffractometer, moving-crystal/moving-counter technique, 2° < 2 $\theta$  ≤ 140°, –11 to 11 ( $h$ ), –12 to 12 ( $h$ ), –13 to 13 ( $l$ ). 24 reflections were used to measure the lattice parameters;  $\bar{2}04$  used as intensity standard, average count 12 039 with  $\sigma = 2426$ . After merging ( $R = 0.10$ ), 2994 were non-zero and with  $I > 2.2 \sigma(I)$ . No absorption corrections were applied. The structure was solved in  $P\bar{1}$  by direct methods using *MULTAN* (Declercq, Germain, Main & Woolfson, 1973). Isotropic least-squares refinement and further refinement with

anisotropic temperature factors, refining one molecule at a time (eight cycles) using *SHELX 76* (Sheldrick, 1976), gave  $R = 0.096$  (maximum atom coordinate  $\sigma = 0.0015$  Å). Further refinement with anisotropic overall scale factor (Shakked & Rabinovich, 1977) using  $P\bar{1}$  symmetry and omitting 28 reflections with  $(F_c - F_o)/\sigma > 4.2$  (poor agreement probably due to absorption or extinction) gave final  $R = 0.084$  (maximum atom coordinate  $\sigma = 0.0012$  Å). In final cycle, maximum shift/ $\sigma$  0.092, average shift/ $\sigma$  0.022. 18 of the H atoms were found using difference Fourier syntheses and the remaining six were calculated. The latter included the imide H, for which the electron peak was not very clear. The final difference Fourier map showed residual electron density of 0.22 (max.) and –0.30 e Å<sup>-3</sup> (min.).

**Discussion.** Final atomic coordinates are in Table 1\* and bond lengths and angles in Table 2. Fig. 1 is a view of the molecule showing the atom numbering.

The results of this study are in close agreement with those of related compounds (Hamodrakas *et al.*, 1978; Hamodrakas *et al.*, 1977; Hamodrakas & Filippakis, 1977). The ester group, C(13), C(14), O(3), O(4), C(15), C(16), is almost planar [maximum deviation from the plane 0.13 (1) Å] with dimensions similar to other observations (Hamodrakas *et al.*, 1978).

The propionate side chain, C(12), C(13), C(14), O(4), O(3), is nearly perpendicular to the phenyl ring [torsion angle C(1)–C(2)–C(12)–C(13) = 93.3 (6)°]. This conformation has also been observed previously (Hamodrakas *et al.*, 1978). The high temperature factors of O(3) and C(16) probably indicate that this side chain is involved in some disorder. The methoxy groups O(5)–C(17) and O(6)–C(18) are almost coplanar with the phenyl ring [torsion angles of –5.7 (7) and –4.5 (8)°] with C(17) and C(18) being 0.18 (18) and 0.13 (18) Å from the plane of the ring.

\* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38392 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates ( $\times 10^4$ ) for non-hydrogen atoms and equivalent isotropic temperature factors ( $\text{\AA}^2 \times 10^4$ )

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j \quad (\text{Hamilton, 1959}).$$

	x	y	z	$U_{eq}$
S(1)	3988 (1)	-2642 (1)	1372 (1)	361
N(1)	4931 (4)	-1613 (4)	538 (4)	375
N(2)	6898 (4)	-1040 (4)	-1020 (4)	394
O(1)	3859 (4)	-3712 (3)	625 (3)	483
O(2)	2592 (4)	-1835 (3)	2110 (3)	428
O(3)	2146 (9)	1197 (5)	4789 (5)	1149
O(4)	1689 (5)	229 (4)	6614 (4)	558
O(5)	7440 (4)	-5350 (4)	4596 (4)	628
O(6)	7068 (5)	-6661 (4)	2826 (4)	638
C(1)	4958 (5)	-3376 (4)	2423 (4)	357
C(2)	5081 (5)	-2656 (5)	3418 (5)	408
C(3)	5919 (6)	-3333 (5)	4164 (5)	490
C(4)	6590 (5)	-4638 (5)	3930 (5)	463
C(5)	6401 (6)	-5363 (5)	2956 (5)	438
C(6)	5606 (6)	-4719 (5)	2195 (5)	422
C(7)	6291 (5)	-2032 (5)	-340 (4)	352
C(8)	7145 (6)	-3321 (5)	-631 (5)	421
C(9)	8556 (6)	-3551 (5)	-1511 (5)	427
C(10)	9134 (6)	-2478 (5)	-2169 (5)	479
C(11)	8270 (6)	-1252 (5)	-1921 (5)	484
C(12)	4323 (6)	-1232 (5)	3791 (5)	514
C(13)	2799 (7)	-1161 (5)	4814 (5)	537
C(14)	2155 (8)	209 (6)	5386 (6)	599
C(15)	1057 (6)	1498 (6)	7287 (6)	542
C(16)	978 (12)	1318 (8)	8602 (8)	1075
C(17)	7769 (7)	-4656 (7)	5547 (6)	784
C(18)	6852 (11)	-7449 (7)	1890 (7)	1004
C(19)	9480 (6)	-4919 (6)	-1757 (6)	529

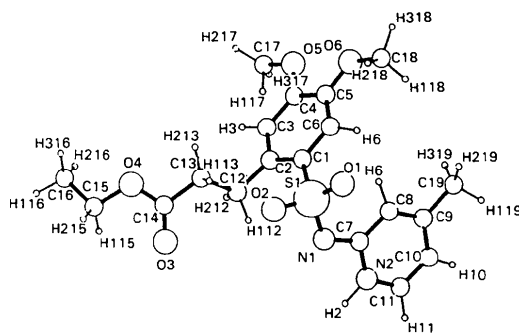


Fig. 1. PLUTO 78 (Motherwell, 1978) drawing of the molecule showing numbering of the atoms.

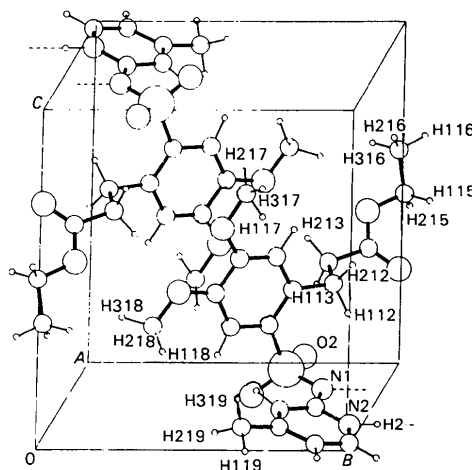


Fig. 2. x-axis projection packing diagram drawn by PLUTO 78. Postulated hydrogen bonds are indicated with dashed lines.

Table 2. Bond lengths ( $\text{\AA}$ ) and bond angles ( $^\circ$ ) with e.s.d.'s in parentheses

S(1)—N(1)	1.603 (4)	C(1)—C(6)	1.400 (6)
S(1)—O(1)	1.441 (4)	C(2)—C(3)	1.411 (7)
S(1)—O(2)	1.441 (3)	C(2)—C(12)	1.514 (7)
S(1)—C(1)	1.779 (5)	C(3)—C(4)	1.370 (7)
N(1)—C(7)	1.359 (6)	C(4)—C(5)	1.407 (8)
N(2)—C(7)	1.362 (6)	C(5)—C(6)	1.384 (7)
N(2)—C(11)	1.360 (6)	C(7)—C(8)	1.409 (6)
O(3)—C(14)	1.203 (7)	C(8)—C(9)	1.376 (7)
O(4)—C(14)	1.304 (7)	C(9)—C(10)	1.411 (7)
O(4)—C(15)	1.453 (6)	C(9)—C(19)	1.497 (7)
O(5)—C(4)	1.366 (6)	C(10)—C(11)	1.356 (7)
O(5)—C(17)	1.435 (8)	C(12)—C(13)	1.530 (8)
O(6)—C(5)	1.359 (6)	C(13)—C(14)	1.501 (8)
O(6)—C(18)	1.422 (8)	C(15)—C(16)	1.457 (10)
C(1)—C(2)	1.396 (7)		
N(1)—S(1)—O(1)	112.6 (2)	O(5)—C(4)—C(3)	125.3 (5)
N(1)—S(1)—O(2)	104.5 (2)	O(6)—C(5)—C(4)	115.7 (4)
N(1)—S(1)—C(1)	108.7 (2)	O(6)—C(5)—C(6)	125.2 (5)
O(1)—S(1)—O(2)	116.3 (2)	C(4)—C(5)—C(6)	119.1 (5)
O(1)—S(1)—C(1)	106.5 (2)	C(5)—C(6)—C(1)	120.2 (5)
O(2)—S(1)—C(1)	108.0 (2)	N(1)—C(7)—N(2)	113.9 (4)
S(1)—N(1)—C(7)	121.3 (3)	N(1)—C(7)—C(8)	129.5 (4)
C(15)—O(4)—C(14)	118.5 (5)	N(2)—C(7)—C(8)	116.6 (4)
C(4)—O(5)—C(17)	117.8 (5)	C(7)—C(8)—C(9)	121.4 (5)
C(5)—O(6)—C(18)	117.8 (4)	C(8)—C(9)—C(10)	119.2 (5)
S(1)—C(1)—C(2)	122.1 (4)	C(8)—C(9)—C(19)	120.6 (5)
S(1)—C(1)—C(6)	116.4 (4)	C(10)—C(9)—C(19)	120.2 (5)
C(2)—C(1)—C(6)	121.5 (4)	C(9)—C(10)—C(11)	118.7 (5)
C(1)—C(2)—C(3)	117.0 (5)	N(2)—C(11)—C(10)	121.1 (5)
C(1)—C(2)—C(12)	125.0 (4)	C(2)—C(12)—C(13)	110.9 (4)
C(3)—C(2)—C(12)	117.9 (5)	C(12)—C(13)—C(14)	112.4 (5)
C(2)—C(3)—C(4)	122.0 (5)	O(4)—C(14)—O(3)	122.9 (6)
C(5)—C(4)—O(5)	114.6 (5)	O(3)—C(14)—C(13)	124.1 (6)
C(5)—C(4)—C(3)	120.1 (5)	O(4)—C(14)—C(13)	112.9 (5)
		O(4)—C(15)—C(16)	108.3 (5)

This planar arrangement has been observed in more than 60 structures while the O(5)—C(4) and O(6)—C(5) bond lengths of 1.366 (6) and 1.359 (6)  $\text{\AA}$  indicate partial double-bond character and compare well with the average calculated value of 1.36  $\text{\AA}$  for similar bonds (Hamodrakas, 1974). It would appear that the tendency of the methoxy groups to be coplanar with the phenyl ring has resulted in some close contacts between C(17), C(3) and C(18), C(6) and the H atoms attached to them, which have been relieved by increasing the angles C(3)—C(4)—O(5) and C(6)—C(5)—O(6) from 120 to 125.3 (5) and 125.2 (5) $^\circ$  respectively.

The tetrahedral coordination of the atoms around the S atom is distorted, with the O(1)—S(1)—O(2) angle being 116.3 (2) $^\circ$ , larger than the normal tetrahedral value, possibly due to repulsions between the sulphonamide atoms O(1) and O(2) (Kálmán, Duffin & Kucsman, 1971). The S(1)—O(1) and S(1)—O(2) bond lengths of 1.441 (4) and 1.441 (3)  $\text{\AA}$  indicate  $\pi$ -bond character of over 60% (Cruickshank, 1961), while the S(1)—C(1) and S(1)—N(1) bond lengths of 1.779 (5)

and 1.603 (4) Å are very close to values obtained for similar structures (Hamodrakas *et al.*, 1978). Pairs of molecules are probably held together by pairs of N(1)···H—N(2) hydrogen bonds [N(1)···N(2) = 2.917 (7) Å], as shown in Fig. 2 with possible hydrogen bonds indicated by dashed lines.

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## Structure of 8,5'-Anhydro-8-hydroxy-9-β-D-ribofuranosyladenine (8,5'-O-cyclo-A) Monohydrate, C<sub>10</sub>H<sub>11</sub>N<sub>5</sub>O<sub>4</sub>·H<sub>2</sub>O

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**Abstract.**  $M_r = 283.3$ , monoclinic,  $P2_1$ ,  $a = 32.747$  (6),  $b = 4.975$  (1),  $c = 8.485$  (1) Å,  $\beta = 121.22$  (1)°,  $V = 1182.1$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $D_m$  (floatation in benzene/CCl<sub>4</sub>) = 1.571 (2),  $D_x = 1.592$  Mg m<sup>-3</sup>, graphite-monochromated Cu  $K\alpha$ ,  $\lambda = 1.54173$  Å,  $\mu(\text{Cu } K\alpha) = 1.13$  mm<sup>-1</sup>,  $F(000) = 592$ , final  $R = 0.048$  for 2054 reflections (measured at 293 K). The two independent molecules in the asymmetric unit take very similar conformations: *anti* around the glycosyl bond and C(4')-endo-O(1')-exo sugar puckering. The crystal structure is stabilized by the hydrogen-bonding network.

**Introduction.** The glycosyl torsion angle in cyclonucleosides and cyclonucleotides is determined by a covalent bond between the base and the furanose ring. Such an imposed restriction about the particular bond is suitable for comparison of the molecular conformation in the crystal with that in solution. To this aim, crystal

structures of 8,5'-cyclonucleosides have been solved by X-ray analysis. However, these molecules possessed either an isopropylidene or an ethoxymethylidene group in the sugar moiety. In the present molecule, which was synthesized, such an additional group is not involved and the net effect imposed by the fused C(8)–O(5') linkage could be estimated.

**Experimental.** Colorless prismatic crystals from aqueous solution by slow evaporation at room temperature; cell dimensions by a least-squares refinement of the  $2\theta$  values of 25 reflections with  $2\theta = 27$ –47°; crystal 0.08 × 0.08 × 0.4 mm, Rigaku–Denki AFC-5 automated diffractometer,  $2\theta_{\text{max}} = 128^\circ$ ,  $\theta$ – $2\theta$  scan technique; of 2216 unique reflections,  $-38 \leq h \leq 32$ ,  $0 \leq k \leq 5$  and  $0 \leq l \leq 9$ , 2059 had  $|F_o| > 3\sigma(F_o)$ ; three standard reflections, 10,0,0, 020 and 002, monitored after every 100 reflections, showed less than 2 (1)% intensity fluctuation throughout data collection;