réflexions observées, les coordonnées des atomes d'hydrogène n'ont pas été affinées à l'exception de celles des atomes H(3) et H(6) qui participent aux liaisons hydrogène.

Les listes des facteurs de structure, des facteurs d'agitation thermique anisotrope, des coordonnées des atomes d'hydrogène, des distances C—H et N—H, des distances interatomiques intermoléculaires, des distances des atomes aux plans moyens et des angles de torsion ont été déposées au dépôt d'archives de la British Library Document Supply Centre (Supplementary Publication No. SUP 71335: 11 pp.). On peut en obtenir des copies en s'adressant à: The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre. [Référence de CIF: PA1049]

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6-(2-Methylpropylthio)-2-pyridyl Methanesulfonate

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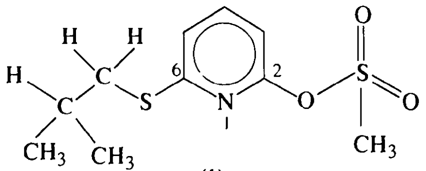
Abstract

The title compound, $C_{10}H_{15}NO_3S_2$, is an acetylcholinesterase inhibitor when bioactivated to the sulfoxide or sulfone. The pyridine ring atoms are coplanar to within experimental error and the 2-methylpropylthio substituent is extended with atoms C6, S6, C8 and C9 lying close to the ring plane [torsion angle 177.0 (4)°]. The orientation of the methanesulfonate moiety to the pyridine ring is given by the torsion angle C2—O2—S2—C7 = 84.4 (4)°.

Comment

The structure determination was carried out to define the conformation of the title compound (1) for use in theoretical studies aiming to define the relationships between molecular structure and biological activity. As far as we are aware, this is the first reported X-ray structure of a 2-pyridyl sulfonate. Moreover, a search of the Cambridge Structural Database (Allen, Kennard & Taylor, 1983) did not reveal any structural moiety containing a pyridine ring substituted with O and S atoms at the 2 and 6 positions, respectively.



(1)

A perspective view of (1) is shown in Fig. 1. It is interesting to note a significant difference in the two C—N bond lengths, *cf.* N1—C2 = 1.302 (6) and N1—C6 = 1.349 (5) Å, and in the two endocyclic bond angles, *cf.* N1—C2—C3 = 127.6 (3) and N1—C6—C5 = 122.2 (3)°. The endocyclic angle subtended at the N atom is 116.4 (3)°. The distortion of the tetrahedral array about the sulfonate S atom is reflected in the variation of the bond lengths and interbond angles involving S2. The S—C bond length is 1.754 (5) Å and the S—O and S=O bonds are 1.598 (3) and 1.423 (2) Å (mean value), respectively. The O=S=O angle, 120.1 (2)°, is much larger than the other angles subtended at S2, which range in value from 103.2 (3) (O2—S2—O3) to 110.7 (2)° (O4—S2—C7). These dimensions agree with those reported for other methanesulfonate moieties. The C6—S6 bond

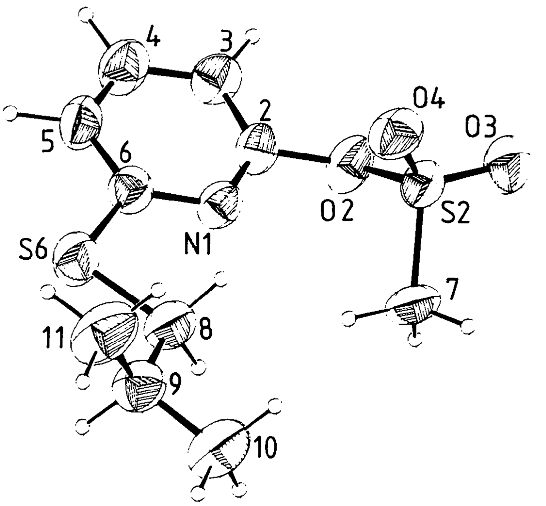


Fig. 1. The molecular structure with thermal ellipsoids scaled to 50% probability. The C symbol is omitted and the H atoms are denoted by spheres of arbitrary radii.

length in the methylpropylthio moiety is 1.745 (5) Å, while the S6—C8 bond, as expected, has the significantly larger value of 1.827 (5) Å with the C6—S6—C8 angle of 102.4 (2)°. There are no abnormally short intermolecular contacts in the crystal structure and the molecules are stabilized by van der Waals interactions.

Experimental

Crystal data

$C_{10}H_{15}NO_3S_2$
$M_r = 261.4$
Monoclinic
$P2_1/c$
$a = 13.655$ (1) Å
$b = 5.562$ (1) Å
$c = 17.100$ (2) Å
$\beta = 98.82$ (1)°
$V = 1283.4$ (4) Å^3
$Z = 4$
$D_x = 1.353$ Mg m^{-3}
D_m(flotation) = 1.34 (1) Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 20$–$31°$
$\mu = 3.66$ mm^{-1}
$T = 291$ (1) K
Platelet
0.10 × 0.26 × 0.41 mm
Colourless

Data collection

Rigaku AFC diffractometer
$\omega/2\theta$ scans
Absorption correction: Gaussian (*SHELX*76; Sheldrick, 1976)
$T_{\min} = 0.303$, $T_{\max} = 0.708$
2138 measured reflections
2082 independent reflections
1152 observed reflections [$F > 4.0\sigma(F)$]

$R_{\rm int} = 0.012$
$\theta_{\max} = 65.0°$
$h = -16 \rightarrow 16$
$k = 0 \rightarrow 6$
$l = 0 \rightarrow 20$
3 standard reflections monitored every 50 reflections intensity variation: none

Refinement

Refinement on F
Final $R = 0.041$
$wR = 0.059$
$S = 1.00$
1152 reflections
161 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F) + 0.0025F^2]$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

Extinction correction: isotropic; $F^* = F(1 - xF^2/\sin\theta)$
Extinction coefficient: $x = 1.88$ (14) × 10^{-6}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

Data collection: Rigaku AFC software. Cell refinement: Rigaku AFC software. Data reduction: Rigaku AFC software. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1986). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *Xtal*3.0 (Hall & Stewart, 1990). Program used to prepare figures: *ORTEP*II (Johnson, 1976). Calculations were performed on a VAX8800 computer. The structure was solved by direct methods and refined by full-matrix least-squares techniques. The H-atom coordinates were calculated and the atoms given individual isotropic temperature factors.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
N1	0.2422 (2)	0.0246 (6)	0.1648 (2)	3.7 (1)
C2	0.2006 (3)	0.1762 (8)	0.2075 (3)	3.8 (1)
C3	0.2447 (3)	0.3638 (8)	0.2519 (3)	4.3 (1)
C4	0.3451 (3)	0.3913 (9)	0.2501 (3)	5.2 (1)
C5	0.3933 (3)	0.2402 (9)	0.2042 (3)	4.8 (1)
C6	0.3394 (3)	0.0590 (8)	0.1620 (3)	3.8 (1)
O2	0.0994 (2)	0.1404 (6)	0.2117 (2)	4.6 (1)
S2	0.0214 (1)	0.0813 (2)	0.1345 (1)	4.04 (3)
O3	−0.0714 (2)	0.1382 (6)	0.1583 (2)	5.9 (1)
O4	0.0525 (2)	0.2036 (6)	0.0696 (2)	5.1 (1)
C7	0.0308 (4)	−0.2310 (8)	0.1233 (3)	4.6 (1)
S6	0.3995 (1)	−0.1272 (2)	0.1023 (1)	5.21 (3)
C8	0.2973 (3)	−0.3049 (9)	0.0501 (3)	4.8 (10
C9	0.3308 (4)	−0.4705 (9)	−0.0083 (3)	4.9 (1)
C10	0.2444 (4)	−0.6348 (10)	−0.0418 (4)	6.3 (2)
C11	0.3701 (5)	−0.3395 (12)	−0.0755 (4)	7.0 (2)

Table 2. *Geometric parameters* (Å, °)

N1—C2	1.302 (6)	O2—S2	1.598 (3)
N1—C6	1.349 (5)	S2—O3	1.425 (3)
C2—C3	1.374 (6)	S2—O4	1.421 (4)
C2—O2	1.409 (5)	S2—C7	1.754 (5)
C3—C4	1.384 (6)	S6—C8	1.827 (5)
C4—C5	1.383 (7)	C8—C9	1.482 (7)
C5—C6	1.384 (6)	C9—C10	1.532 (7)
C6—S6	1.745 (5)	C9—C11	1.526 (9)
C2—N1—C6	116.4 (3)	O2—S2—O3	103.2 (2)
N1—C2—C3	127.6 (3)	O2—S2—O4	107.8 (2)
N1—C2—O2	116.9 (3)	O2—S2—C7	104.1 (2)
C3—C2—O2	115.4 (3)	O3—S2—O4	120.1 (2)
C2—C3—C4	115.0 (3)	O3—S2—C7	109.5 (2)
C3—C4—C5	120.3 (4)	O4—S2—C7	110.7 (2)
C4—C5—C6	118.5 (3)	C6—S6—C8	102.4 (2)
N1—C6—C5	122.2 (3)	S6—C8—C9	112.0 (3)
N1—C6—S6	119.3 (3)	C8—C9—C10	108.9 (4)
C5—C6—S6	118.5 (3)	C8—C9—C11	113.0 (4)
C2—O2—S2	121.4 (2)	C10—C9—C11	110.0 (4)

The compound was synthesized by a method adapted from that reported by Kato, Masui & Ishida (1989) and crystals suitable for an X-ray study were grown from petroleum ether (b.p. 333–353 K).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry and stereoview of the crystal packing have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71384 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HL1033]

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