culations utilized *CALC* (Gould & Taylor, 1985). Program used to solve and refine structure: *SHELX*76 (Sheldrick, 1976).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71184 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1042]

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8-(*N*,*N*-Di-*p*-toluenesulfonylamino)quinoline

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Abstract

The S atoms exist in distorted tetrahedral geometries with an average S—N bond distance of 1.672 (5) Å. The coordination about the amido N atom is trigonal planar indicating delocalization of π-electron density around this centre.

Comment

The near trigonal planar geometry about the N(8) atom [this atom lies 0.120 (6) Å out of the plane defined by atoms S(1), S(2) and C(8)] is shown in the three trigonal angles of 117.5 (7), 118.6 (7) and 122.2 (5)°. The equivalence of the S—N bond distances [average 1.672 (5) Å], coupled with the planar N(8) centre, suggest delocalization of π-electron density over this chromophore. The elongation of the S(2)—O(3) bond distance to 1.431 (7) Å may indicate the participation of this bond in conjugation; the S(1)—N(8)—S(2)—O(3) torsion angle is 179.4 (6)°. The remaining S—N—S—O torsion angles [50.8 (7), 144.1 (6) and 15.2 (7)°] suggest little participation in conjugation of the other S=O groups [range of S=O distances 1.408 (8)-1.415 (7) Å]. The S(1)—N(8)—C(8)—C(7) torsion angle of −82 (1)° also indicates that the quinoline residue is not involved in conjugation to the O=S—N—S moiety.

The structure is molecular with the closest intermolecular contact (2.381 Å) occurring between H(17) and H(17′) (symmetry operation: $-x$, $1-y$, $-z$). There are some weaker contacts involving the O atoms, *i.e.* O(3)· · ·H(4″) 2.389 Å (symmetry operation: $0.5-x$, $1-y$, $0.5+z$) and O(1)· · ·H(11‴) 2.482 Å (symmetry operation: x, $0.5-y$, $-0.5+z$); it is therefore unlikely that the presence of intermolecular contacts accounts for the elongation of the S(2)=O(3) bond distance relative to the remaining S=O distances.

Geometric parameters of the the title compound and of the parent compound, *i.e.* 8-toluenesulfonamido-

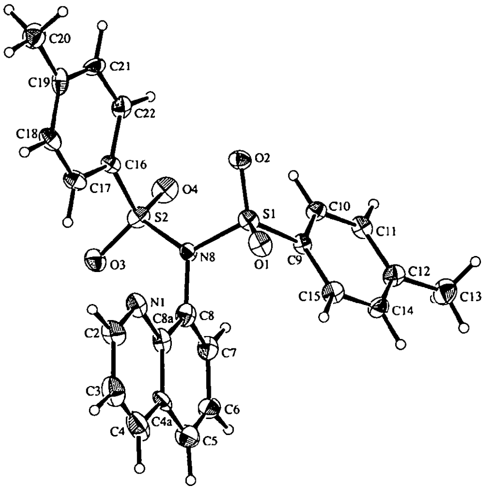

Fig. 1. Molecular structure of the title compound showing the crystallographic numbering scheme; diagram drawn at 20% probability levels (Johnson, 1976).

quinoline (Germain, Declercq, Castresana, Elizalde & Arrieta, 1983) are difficult to compare as the errors associated with the derived parameters are quite high.

Experimental

Crystal data

$C_{23}H_{20}N_2O_4S_2$
$M_r = 452.5$
Orthorhombic
Pbca
$a = 13.784\ (4)$ Å
$b = 22.510\ (4)$ Å
$c = 13.409\ (3)$ Å
$V = 4160\ (1)$ Å^3
$Z = 8$
$D_x = 1.445$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 23.7\text{–}33.9°$
$\mu = 2.902$ cm^{-1}
$T = 295$ K
Multifaceted
0.07 × 0.26 × 0.26 mm
Colourless

Data collection

AFC-6*R* diffractometer
$\omega/2\text{-}\theta$ scans
Absorption correction: refined from ΔF (Walker & Stuart, 1983)
$T_{\min} = 0.803$, $T_{\max} = 1.099$
4278 measured reflections
3813 independent reflections
1272 observed reflections [$I \geq 3.0\sigma(I)$]

$\theta_{\max} = 25.0°$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 27$
$l = 0 \rightarrow 16$
3 standard reflections monitored every 400 reflections
intensity variation: 0.23%

Refinement

Refinement on *F*
Final $R = 0.0550$
$wR = 0.0560$
$S = 3.020$
1272 reflections
280 parameters
H atoms located from difference maps; not refined
Weighting scheme based on measured e.s.d.'s

$(\Delta/\sigma)_{\max} = 0.0000$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.24$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1986). Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN FINISH*.

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	$U_{\rm eq}$
S(1)	0.1189 (2)	0.2900 (1)	0.0473 (2)	0.0515
S(2)	0.0525 (2)	0.3961 (2)	0.1534 (2)	0.0549
O(1)	0.1386 (5)	0.2840 (3)	−0.0557 (5)	0.0620
O(2)	0.0294 (5)	0.2703 (3)	0.0869 (5)	0.0572
O(3)	0.0837 (5)	0.4567 (3)	0.1506 (6)	0.0661
O(4)	0.0551 (5)	0.3643 (3)	0.2438 (6)	0.0726
N(1)	0.1208 (8)	0.4243 (4)	−0.1038 (8)	0.0635
N(8)	0.1255 (6)	0.3628 (3)	0.0711 (6)	0.0457
C(2)	0.124 (1)	0.4534 (6)	−0.1887 (10)	0.0723
C(3)	0.205 (1)	0.4803 (7)	−0.223 (1)	0.0910
C(4)	0.290 (1)	0.4787 (7)	−0.175 (1)	0.1091
C(4a)	0.294 (1)	0.4497 (5)	−0.088 (1)	0.0613
C(5)	0.383 (1)	0.4486 (6)	−0.031 (1)	0.0840
C(6)	0.3792 (9)	0.4203 (5)	0.056 (1)	0.0711
C(7)	0.2942 (9)	0.3943 (5)	0.0890 (9)	0.0639
C(8)	0.2118 (9)	0.3943 (5)	0.0372 (10)	0.0599
C(8a)	0.2104 (10)	0.4227 (5)	−0.0522 (9)	0.0516
C(9)	0.2148 (7)	0.2569 (4)	0.1108 (8)	0.0410
C(10)	0.1995 (7)	0.2345 (5)	0.2055 (9)	0.0496
C(11)	0.2729 (8)	0.2044 (5)	0.2521 (8)	0.0499
C(12)	0.3619 (8)	0.1967 (5)	0.2097 (9)	0.0584
C(13)	0.4402 (8)	0.1620 (5)	0.260 (1)	0.0826
C(14)	0.3750 (8)	0.2204 (5)	0.117 (1)	0.0625
C(15)	0.3023 (9)	0.2502 (5)	0.0667 (8)	0.0602
C(16)	−0.0639 (7)	0.3916 (5)	0.1051 (8)	0.0428
C(17)	−0.0877 (9)	0.4268 (5)	0.0258 (9)	0.0572
C(18)	−0.1798 (10)	0.4262 (5)	−0.0105 (9)	0.0588
C(19)	−0.2497 (9)	0.3915 (6)	0.0315 (10)	0.0578
C(20)	−0.3508 (8)	0.3897 (5)	−0.0061 (10)	0.0781
C(21)	−0.2251 (8)	0.3584 (5)	0.110 (1)	0.0603
C(22)	−0.1318 (9)	0.3561 (5)	0.1474 (8)	0.0564

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

S(1)—O(1)	1.415 (8)	C(6)—C(7)	1.38 (2)
S(1)—O(2)	1.414 (7)	C(7)—C(8)	1.33 (2)
S(1)—N(8)	1.672 (8)	C(8)—C(8a)	1.36 (2)
S(1)—C(9)	1.74 (1)	C(9)—C(10)	1.38 (2)
S(2)—O(3)	1.431 (8)	C(9)—C(15)	1.35 (2)
S(2)—O(4)	1.408 (9)	C(10)—C(11)	1.37 (2)
S(2)—N(8)	1.671 (9)	C(11)—C(12)	1.36 (2)
S(2)—C(16)	1.73 (1)	C(12)—C(13)	1.49 (2)
N(1)—C(2)	1.31 (2)	C(12)—C(14)	1.36 (2)
N(1)—C(8a)	1.42 (2)	C(14)—C(15)	1.38 (2)
N(8)—C(8)	1.46 (1)	C(16)—C(17)	1.37 (2)
C(2)—C(3)	1.35 (2)	C(16)—C(22)	1.35 (2)
C(3)—C(4)	1.35 (3)	C(17)—C(18)	1.36 (2)
C(4)—C(4a)	1.34 (2)	C(18)—C(19)	1.36 (2)
C(4a)—C(5)	1.45 (2)	C(19)—C(20)	1.48 (2)
C(4a)—C(8a)	1.38 (2)	C(19)—C(21)	1.33 (2)
C(5)—C(6)	1.33 (2)	C(21)—C(22)	1.38 (2)
O(1)—S(1)—O(2)	120.3 (4)	O(3)—S(2)—C(16)	108.8 (5)
O(1)—S(1)—N(8)	105.6 (4)	O(4)—S(2)—N(8)	109.0 (5)
O(1)—S(1)—C(9)	106.9 (4)	O(4)—S(2)—C(16)	108.4 (5)
O(2)—S(1)—N(8)	106.4 (4)	N(8)—S(2)—C(16)	106.6 (5)
O(2)—S(1)—C(9)	110.2 (5)	C(2)—N(1)—C(8a)	114 (1)
N(8)—S(1)—C(9)	106.6 (4)	S(1)—N(8)—S(2)	122.2 (5)
O(3)—S(2)—O(4)	120.0 (5)	S(1)—N(8)—C(8)	117.5 (7)
O(3)—S(2)—N(8)	103.3 (4)	S(2)—N(8)—C(8)	118.6 (7)

The compound was obtained from the attempted recrystallization of the parent 8-toluenesulfonamidoquinoline from ethanol solution (Billman & Chernin, 1962). Scans of $(1.00 + 0.35\tan\theta)°$ were made at 16° min^{-1} in ω. The ratio of peak to background counting times was 2:1.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71194 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1053]

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N-(2-Chloro-6-methylphenyl)-6-methyl-3-pyridinecarboxamide Hydrate: an Anticonvulsant Drug Exhibiting Solid-State Rotational Disorder

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Abstract

The phenyl ring, the pyridine ring and the carboxamide group are conformationally planar. The Cl atom and the methyl group on the phenyl ring are statistically disordered, by 180° rotation about the N(6)—C(6) linkage, with relative occupancies of 0.7061 (5) and 0.2939 (5) at the major and minor sites, respectively. Hydrogen bonds involving the nitrogen (donor) and the oxygen (acceptor) of the carboxamide moiety with the water oxygen, together with the hydrogen bond between the nitrogen (acceptor) of the pyridine moiety and the water oxygen structurally stabilize the pyridine and carboxamide groups.

Comment

6-Chloro-*N*-(2,6-dimethylphenyl)-3-pyridinecarboxamide is an anticonvulsant agent with a high therapeutic ratio and long half-life. Moreover, it has a lessened propensity to interact with drug metabolizing enzymes compared to other anticonvulsant amides (Robertson, 1990). It is effective against tonic-extensor seizures elicited by maximal electroshock and therefore has potential utility in treating grand-mal, cortical focal, temperoral lobe, focal and post-traumatic seizures. This compound, recrystallized from aqueous methanol, was supplied by Eli Lilly & Co. of Indianapolis, USA. The structure analysis reported here was undertaken as part of a study of convulsant and anticonvulsant drugs being carried out in this department.

The phenyl ring is planar. No significant deviations from the average bond length [1.385 (4) Å] or bond angle [120.0 (3)°] are observed for the phenyl ring. The pyridine ring is also planar, as is the carboxamide moiety. All bond distances are normal and in agreement with those quoted by Allen, Kennard, Watson, Brammer, Orpen & Taylor (1987). The angle between the normal to the least-squares plane through the atoms of the phenyl ring and the least-squares plane through the atoms of the pyrdine ring is 29.5 (9)°; for the phenyl ring plane and the plane through the carboxamide group atoms, the dihedral angle is 68.6 (9)°; and for the phenyl–pyridine ring planes 39.2 (8)°. The Cl atom and the methyl group on the phenyl ring are statistically disordered. Hydrogen bonds involving the

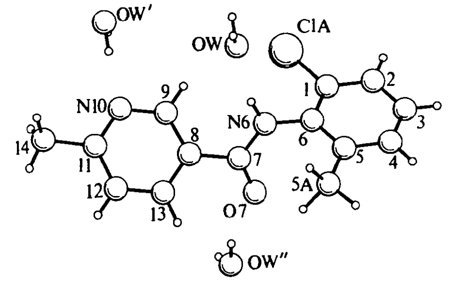

Fig. 1. View of the molecule showing the three hydrogen-bonded symmetry-related water molecules and the numbering scheme.

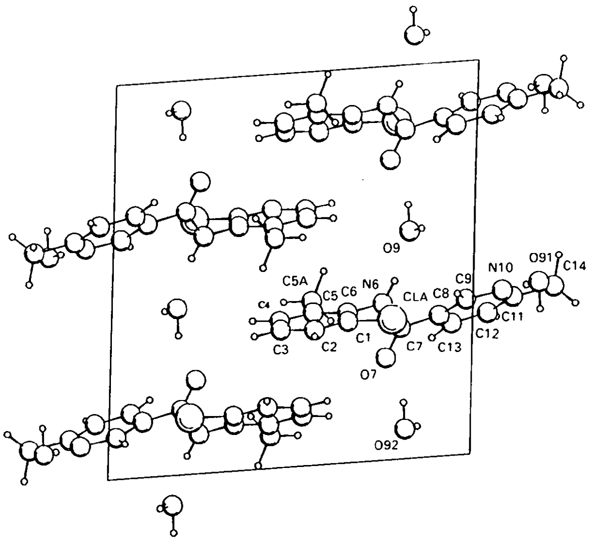

Fig. 2. Crystal packing along **b**.

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