Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and torsion angles have been deposited with the IUCr (Reference: CR1103). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C**50**, 2032–2033

Anticancer Agent Chloroquinoxaline Sulfonamide

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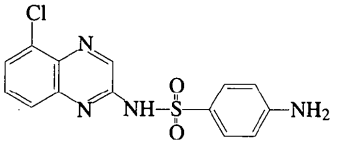
(*Received 11 October 1993; accepted 29 June 1994*)

Abstract

The crystal structure of the antitumor agent 4-amino-*N*-(5-chloro-2-quinoxalinyl)benzenesulfonamide, $C_{14}H_{11}ClN_4O_2S$, has been determined by X-ray diffraction methods. The geometry around the S atom is distorted tetrahedral. The quinoxaline ring system is almost planar with a dihedral angle of 84.4 (2)° between the phenyl ring of the sulfonamide group and the quinoxaline ring. There is an intermolecular N—H···O bond of 2.966 (6) Å.

Comment

Chloroquinoxaline sulfonamide (I) is a new agent with *in vitro* antitumor activity against human lung, breast, melanoma and ovarian cancers (Pinedo, Longo & Chabner, 1992). The mechanism of the antitumor activity is unknown.



(I)

An *ORTEP*II drawing (Johnson, 1976) of the structure is shown in Fig. 1. The S=O distances [1.416 (4) and 1.436 (4) Å] are within the range observed for other sulfanalide drugs (Chaterjee, Dattagupta & Saha, 1981). The S—C distance of 1.729 (6) Å is shorter than normally observed in sulfonamides (Urbanczyk-Lipkowska, Krajewski, Gluzinski & Stadnicka, 1982). In addition, the C—Cl bond distance [1.704 (6) Å] is somewhat shorter than that observed in 6-chloro-3-ethoxycarbonyl-2-methylquinoxaline 1,4-dioxide (Macdonald & Arora, 1981). The dihedral angle between the chloro-substituted benzene ring and the heterocyclic ring is 1.52 (5)°, indicating that the quinoxaline part is planar. The structure of monoclinic crystals of the acetonitrile solvate of chloroquinoxaline sulfonamide has been reported (Deutsch, Van Derveer & Zalkow, 1985). Bond distances and angles are similar to those observed here but the molecular conformation is slightly different; the torsion angle C9—S—N2—C2 is −63.2 (6) here and 55.6 (6)° in the monoclinic form.

There is an intermolecular hydrogen bond between the amino N atom and atom O2 of the sulfoxide [N2—H···O2($2-x, -y, 2-z$) 2.966 (6) Å]. Other short contacts (< 3.5 Å) are N12···O1($1+x, y, z$) 3.248 (7), N12···N1($x, 1+y, z$) 3.208 (7) and N12···O2($2-x, 1-y, 2-z$) 3.300 (6) Å.

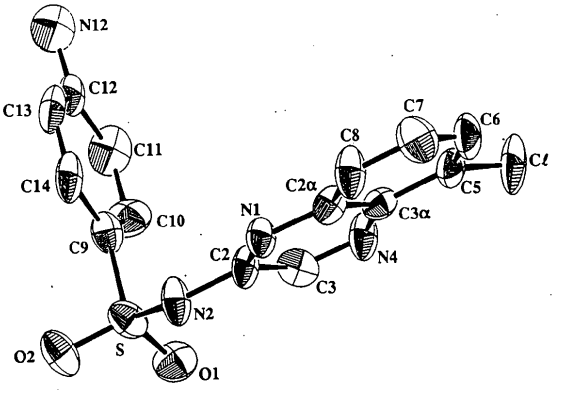


Fig. 1. Displacement ellipsoid plot (Johnson, 1976) of the title molecule. Ellipsoids are drawn at the 50% probability level.

Experimental

Crystal data

$C_{14}H_{11}ClN_4O_2S$
$M_r = 334.8$
Triclinic
$P\bar{1}$
$a = 7.924$ (4) Å
$b = 9.211$ (3) Å
$c = 11.188$ (4) Å
$\alpha = 77.36$ (3)°
$\beta = 85.04$ (3)°
$\gamma = 64.43$ (3)°
$V = 718.5$ (5) Å^3
$Z = 2$
$D_x = 1.547$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 15.4$–17.2°
$\mu = 0.414$ mm^{-1}
$T = 291$ K
Diamond plate
0.2 × 0.2 × 0.125 mm
Light yellow
Crystal source: hot methanol solution of drug from National Cancer Institute

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.007$
$\omega/2\theta$ scans	$\theta_{max} = 30°$
Absorption correction: none	$h = -11 \rightarrow 11$
4426 measured reflections	$k = 0 \rightarrow 12$
4179 independent reflections	$l = -15 \rightarrow 15$
1929 observed reflections $[F > 3\sigma(F)]$	3 standard reflections frequency: 100 min intensity variation: 4%

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.003$
$R = 0.057$	$\Delta\rho_{max} = 0.251$ e Å^{-3}
$wR = 0.057$	$\Delta\rho_{min} = -0.51$ e Å^{-3}
$S = 1.56$	Extinction correction: none
1929 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
232 parameters	
Only coordinates of H atoms refined	
$w = 1/[\sigma^2(F) + (0.02F_o^2)^2]$	

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

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

	x	y	z	U_{eq}
S	0.8820 (2)	0.2169 (2)	0.8417 (1)	0.046 (1)
Cl	1.3380 (2)	0.0960 (2)	0.2376 (1)	0.072 (1)
O1	0.7451 (5)	0.3181 (5)	0.7497 (3)	0.056 (2)
O2	0.8218 (5)	0.1647 (4)	0.9615 (3)	0.058 (2)
N1	1.2440 (6)	−0.1234 (6)	0.6902 (4)	0.042 (2)
C2	1.1075 (8)	0.0202 (7)	0.6893 (5)	0.040 (3)
C2α	1.3390 (8)	−0.1488 (7)	0.5827 (5)	0.039 (3)
N2	1.0112 (7)	0.0425 (5)	0.7985 (4)	0.048 (2)
C3	1.0544 (8)	0.1435 (7)	0.5802 (6)	0.047 (3)
C3α	1.2894 (8)	−0.0233 (7)	0.4760 (5)	0.038 (3)
N4	1.1427 (7)	0.1234 (6)	0.4767 (4)	0.051 (2)
C5	1.3932 (8)	−0.0533 (7)	0.3672 (5)	0.045 (3)
C6	1.5372 (9)	−0.2024 (9)	0.3671 (6)	0.055 (3)
C7	1.5831 (9)	−0.3279 (8)	0.4711 (7)	0.058 (3)
C8	1.4886 (9)	−0.3021 (8)	0.5768 (6)	0.057 (3)
C9	1.0297 (8)	0.3075 (7)	0.8524 (5)	0.049 (3)
C10	1.1863 (9)	0.2284 (8)	0.9262 (5)	0.051 (3)
C11	1.2996 (9)	0.3013 (8)	0.9362 (5)	0.052 (3)
C12	1.2582 (8)	0.4607 (7)	0.8726 (5)	0.043 (3)
N12	1.3696 (9)	0.5354 (7)	0.8849 (5)	0.063 (3)
C13	1.1011 (9)	0.5398 (7)	0.7986 (6)	0.057 (3)
C14	0.9893 (9)	0.4656 (8)	0.7878 (6)	0.057 (3)

Table 2. *Selected geometric parameters* (Å, °)

S—O1	1.416 (4)	C2α—C8	1.409 (9)
S—O2	1.436 (4)	C3α—C5	1.418 (8)
S—N2	1.643 (5)	C5—C6	1.355 (8)
S—C9	1.729 (6)	C6—C7	1.395 (9)
Cl—C5	1.704 (6)	C7—C8	1.353 (9)
N1—C2	1.297 (7)	C9—C10	1.374 (9)
N1—C2α	1.372 (7)	C9—C14	1.386 (9)
C2—C3	1.423 (7)	C10—C11	1.357 (9)
C2—N2	1.390 (7)	C11—C12	1.392 (9)
C3—N4	1.307 (8)	C12—C13	1.377 (8)
N4—C3α	1.351 (8)	C12—N12	1.363 (8)
C2α—C3α	1.414 (8)	C13—C14	1.358 (9)
O1—S—O2	118.8 (2)	C3α—C5—C6	119.3 (5)
O1—S—N2	108.3 (2)	C6—C5—Cl	120.0 (5)
O2—S—N2	102.9 (2)	C5—C6—C7	121.3 (6)
O1—S—C9	109.4 (2)	C6—C7—C8	120.7 (6)
O2—S—C9	110.1 (2)	C7—C8—C2α	120.5 (6)
C2—N1—C2α	117.0 (5)	S—C9—C10	121.7 (5)
N1—C2—C3	121.3 (5)	S—C9—C14	120.2 (5)
N1—C2—N2	116.1 (5)	C10—C9—C14	118.1 (6)
N2—C2—C3	122.5 (5)	S—N2—C2	127.6 (4)
C2—C3—N4	123.2 (6)	C9—C10—C11	121.5 (6)
C3—N4—C3α	116.8 (5)	C10—C11—C12	120.5 (6)
N4—C3α—C2α	120.5 (5)	C11—C12—C13	117.9 (6)
N4—C3α—C5	120.0 (5)	C11—C12—N12	120.6 (6)
N1—C2α—C3α	121.2 (5)	C13—C12—N12	121.6 (6)
N1—C2α—C8	120.1 (5)	C12—C13—C14	121.4 (6)
C3α—C2α—C8	118.7 (5)	C13—C14—C9	120.6 (6)
C2α—C3α—C5	119.5 (5)	N2—S—C9	106.6 (3)
C3α—C5—Cl	120.6 (5)		

Data were collected with the diffractometer mounted on a Rigaku 100 PL with a molybdenum anode operating at 57.5 kV and 90 mA using a 0.5 × 5 mm filament and an Nb filter. The structure was solved by direct methods using the program *SHELXS*86 (Sheldrick, 1985) and refined using *SHELX*76 (Sheldrick, 1976).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: HA1087). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C**50**, 2033–2036

Eupenifeldin

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(*Received* 16 *March* 1993; *accepted* 14 *March* 1994)

Abstract

Eupenifeldin, 4,16,19-trihydroxy-1,6a,10,10,13,18a-hexamethyl-3,6a,7,10,11,11a,12,15,18a,19,20,20a,21-dodecahydro-1*H*-dicyclohepta[*e*:*e*′]cycloundeca[1,2-*b*:5,6-*b*′]dipyran-3,15-dione, $C_{33}H_{40}O_7$, is a novel cytotoxic