C(2)—B(6)	1.715 (8)	B(7)—B(12)	1.78 (1)
C(2)—B(7)	1.711 (7)	B(8)—B(9)	1.76 (1)
C(2)—B(11)	1.696 (8)	B(8)—B(12)	1.78 (1)
C(2)—C(13)	1.513 (8)	B(9)—B(10)	1.788 (8)
B(3)—B(4)	1.775 (7)	B(9)—B(12)	1.759 (7)
B(3)—B(7)	1.762 (8)	B(10)—B(11)	1.752 (8)
B(3)—B(8)	1.760 (7)	B(10)—B(12)	1.772 (9)
B(4)—B(5)	1.771 (7)	B(11)—B(12)	1.767 (7)
C(1)—P—C(14)	102.9 (2)	B(6)—C(2)—C(13)	117.7 (5)
C(1)—P—C(20)	105.1 (2)	B(7)—C(2)—C(13)	119.8 (4)
C(14)—P—C(20)	104.6 (2)	B(11)—C(2)—C(13)	120.8 (4)
P—C(1)—C(2)	113.1 (3)	P—C(14)—C(15)	126.5 (3)
P—C(1)—B(3)	112.0 (3)	P—C(14)—C(19)	115.0 (4)
P—C(1)—B(4)	123.4 (3)	C(15)—C(14)—C(19)	118.5 (4)
P—C(1)—B(5)	129.0 (3)	P—C(20)—C(21)	114.1 (3)
P—C(1)—B(6)	119.2 (3)	P—C(20)—C(25)	128.2 (4)
C(1)—C(2)—C(13)	119.3 (4)	C(21)—C(20)—C(25)	117.6 (4)
B(3)—C(2)—C(13)	117.5 (5)		
P—C(1)—C(2)—C(13)	−4.4 (7)	C(20)—P—C(1)—B(4)	−19.7 (4)
C(14)—P—C(1)—C(2)	95.8 (3)	C(20)—P—C(1)—B(5)	60.2 (4)
C(14)—P—C(1)—B(5)	−49.1 (4)	C(20)—P—C(14)—C(15)	−34.7 (5)
C(14)—P—C(1)—B(6)	28.1 (4)	C(14)—P—C(20)—C(25)	65.2 (4)
C(20)—P—C(1)—C(2)	−154.9 (3)		

Cell refinement, data collection and data reduction: Rigaku AFC-5*S* software. Program used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program used to refine structure: *Xtal*3.0 (Hall & Stewart, 1990). Refinement of the enantiomeric model did not change the *R* factors. Molecular graphics: *ORTEP* (Johnson, 1965). Programs used to prepare material for publication: *BONDLA* and *ATABLE* from *Xtal*3.0.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1140). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-Methyl-5-(2,6,6-trimethyl-1-cyclohexenyl)-2(5*H*)-furanone

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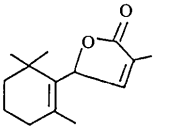
Abstract

The title compound, $C_{14}H_{20}O_2$, can be regarded as a synthetic derivative resembling tobacco isoprenoids and is structurally related to primary precursor α- and β-ionones. The double bond distance C(3)=C(4) in the furanone ring is 1.329 (8) Å and the double bond distance C(1′)=C(2′) in the cyclohexene ring is 1.331 (6) Å. The bond between the rings, C(5)—C(2′), is 1.513 (7) Å and the bond torsion angle O(1)—C(5)—C(2′)—C(1′), which describes the relative twist of the rings, is ±51.3 (7)°.

Comment

The title compound (I) was isolated as a side product in phase-transfer dihalocarbene addition to β-ionone and was a result of chemoselective adduct formation from a dichloro ether intermediate hydrolysed to the γ lactone (Díaz, Alvarez, Toscano, Shoolery & Jankowski, 1990). A structure determination was undertaken in order to confirm the identity of the compound and establish the relative positions of the rings.



(I)

The cyclohexene ring adopts a half-chair conformation [Cremer & Pople (1975) parameters: Q = 0.408 (7) Å, θ = 51.7 (8)° and φ = −151 (1)°] while the methyl benzofuranone ring is almost planar. The

dihedral angle between the best least-squares planes of the two rings is 99.8 (2)°. In the crystal, the molecules are held together by van der Waals interactions.

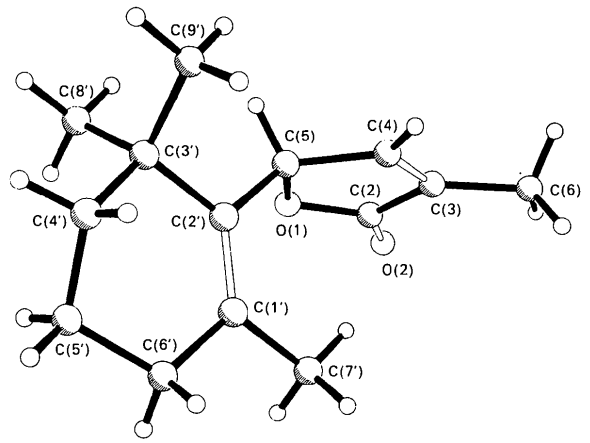


Fig. 1. Perspective view of the title molecule with atomic numbering scheme.

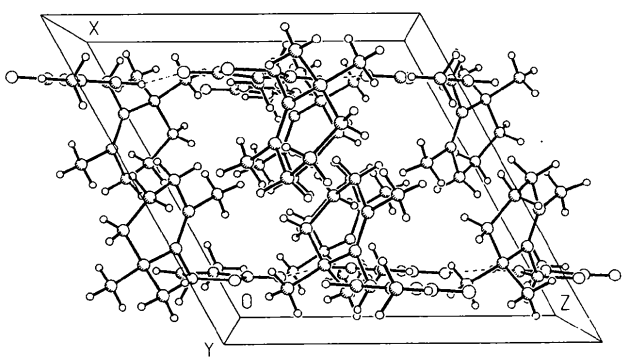


Fig. 2. Crystal packing of the title molecule.

Experimental

Crystal data

$C_{14}H_{20}O_2$
$M_r = 220.3$
Monoclinic
$P2_1/c$
$a = 11.828$ (2) Å
$b = 10.284$ (2) Å
$c = 11.790$ (2) Å
$\beta = 118.3$ (3)°
$V = 1262.72$ Å^3
$Z = 4$
$D_x = 1.159$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 6.06$–$12.05°$
$\mu = 0.60$ mm^{-1}
$T = 297.0$ K
Prism
0.38 × 0.32 × 0.30 mm
Colorless
Crystal source: evaporation of a mixture of acetone and isopropyl ether

Data collection

Siemens *P3/F* diffractometer
$2\theta/\theta$ scans
Absorption correction: none
1488 measured reflections
1302 independent reflections
1233 observed reflections $[F_o > 3\sigma(F_o)]$
$R_{int} = 0.019$
$\theta_{max} = 50°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 10$
$l = -11 \rightarrow 11$
2 standard reflections monitored every 50 reflections
intensity variation: ±3%

Refinement

Refinement on F
$R = 0.081$
$wR = 0.081$
$S = 1.14$
1233 reflections
145 parameters
H-atom parameters not refined
Unit weights applied
$(\Delta/\sigma)_{max} = 0.006$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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}
O(1)	0.8352 (3)	0.0102 (3)	0.4146 (3)	0.054 (2)
O(2)	0.8354 (4)	−0.1504 (4)	0.2873 (4)	0.081 (2)
C(2)	0.8309 (5)	−0.1170 (5)	0.3813 (5)	0.056 (3)
C(3)	0.8203 (5)	−0.1959 (5)	0.4802 (5)	0.051 (3)
C(4)	0.8178 (5)	−0.1165 (5)	0.5679 (5)	0.047 (2)
C(5)	0.8281 (5)	0.0219 (5)	0.5335 (4)	0.045 (2)
C(6)	0.8122 (6)	−0.3408 (5)	0.4713 (6)	0.073 (3)
C(1′)	0.6040 (5)	0.1079 (4)	0.4289 (4)	0.043 (2)
C(2′)	0.7248 (5)	0.1155 (4)	0.5227 (4)	0.040 (2)
C(3′)	0.7719 (5)	0.2210 (5)	0.6266 (5)	0.050 (2)
C(4′)	0.6595 (6)	0.2940 (6)	0.6225 (7)	0.090 (4)
C(5′)	0.5568 (7)	0.3241 (6)	0.4971 (7)	0.100 (4)
C(6′)	0.5026 (5)	0.2057 (5)	0.4133 (5)	0.066 (3)
C(7′)	0.5509 (5)	0.0046 (5)	0.3267 (5)	0.056 (2)
C(8′)	0.8649 (5)	0.3125 (5)	0.6108 (5)	0.067 (3)
C(9′)	0.8427 (7)	0.1593 (6)	0.7602 (5)	0.087 (4)

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

O(1)—C(2)	1.360 (6)	O(1)—C(5)	1.449 (7)
O(2)—C(2)	1.185 (9)	C(2)—C(3)	1.474 (9)
C(3)—C(4)	1.329 (8)	C(3)—C(6)	1.494 (7)
C(4)—C(5)	1.501 (7)	C(5)—C(2′)	1.513 (7)
C(1′)—C(2′)	1.331 (6)	C(1′)—C(6′)	1.508 (8)
C(1′)—C(7′)	1.502 (7)	C(2′)—C(3′)	1.530 (6)
C(3′)—C(4′)	1.51 (1)	C(3′)—C(8′)	1.524 (9)
C(3′)—C(9′)	1.528 (7)	C(4′)—C(5′)	1.434 (8)
C(5′)—C(6′)	1.507 (8)		
C(2)—O(1)—C(5)	110.4 (4)	O(1)—C(2)—O(2)	122.4 (6)
O(1)—C(2)—C(3)	107.9 (5)	O(2)—C(2)—C(3)	129.7 (5)
C(2)—C(3)—C(4)	108.6 (5)	C(2)—C(3)—C(6)	121.4 (6)
C(4)—C(3)—C(6)	130.0 (6)	C(3)—C(4)—C(5)	109.8 (5)
O(1)—C(5)—C(4)	103.4 (4)	O(1)—C(5)—C(2′)	112.7 (3)
C(4)—C(5)—C(2′)	117.4 (5)	C(2′)—C(1′)—C(6′)	122.7 (4)
C(2′)—C(1′)—C(7′)	125.7 (5)	C(6′)—C(1′)—C(7′)	111.6 (4)
C(5)—C(2′)—C(1′)	122.6 (4)	C(5)—C(2′)—C(3′)	114.2 (4)
C(1′)—C(2′)—C(3′)	123.2 (5)	C(2′)—C(3′)—C(4′)	110.2 (4)
C(2′)—C(3′)—C(8′)	110.5 (5)	C(4′)—C(3′)—C(8′)	111.4 (5)
C(2′)—C(3′)—C(9′)	110.2 (4)	C(4′)—C(3′)—C(9′)	106.5 (6)
C(8′)—C(3′)—C(9′)	107.9 (4)	C(3′)—C(4′)—C(5′)	116.4 (7)
C(4′)—C(5′)—C(6′)	113.0 (5)	C(1′)—C(6′)—C(5′)	113.1 (4)

Program used to solve and refine structure: *SHELXTL* (Sheldrick, 1981).

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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Anticancer Agent Chloroquinoxaline Sulfonamide

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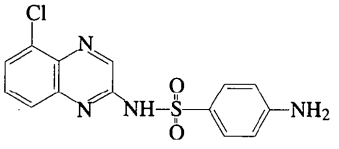
(*Received 11 October 1993; accepted 29 June 1994*)

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.

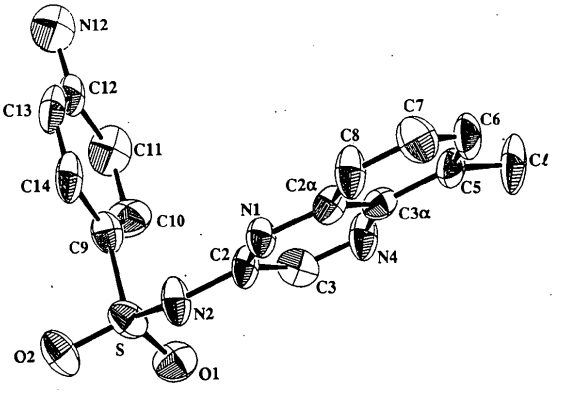


Fig. 1. Displacement ellipsoid plot (Johnson, 1976) of the title molecule. Ellipsoids are drawn at the 50% probability level.

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