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4-(2-Chlorophenyl)-3-(2,6-dichlorophenyl)-spiroisoxazoline-5,2′-benzo[4,5]imidazo[2,1-*b*]-thiazol-3′-one dioxane hemisolvate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.049
wR factor = 0.142
Data-to-parameter ratio = 12.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{23}H_{12}Cl_3N_3O_2S\cdot0.5C_4H_8O_2$, was synthesized by the intermolecular [3+2]-cycloaddition of 2,6-dichlorobenzonitrile oxide and 2-(2-chlorobenzylidene)-benzo[4,5]imidazo[2,1-*b*]thiazol-3-one. The dioxane molecule of solvation is located about a centre of inversion. A spiro junction links an isoxazoline ring and a planar benzo[4,5]-imidazo[2,1-*b*]thiazol-3-one ring.

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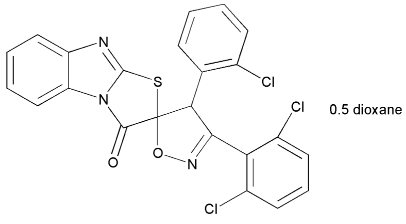
Comment

Spiro-compounds represent an important class of naturally occurring substances, characterized by highly pronounced biological properties (Kobayashi *et al.*, 1991; James *et al.*, 1991). 1,3-Dipolar cycloaddition reactions are important for the construction of spiro-compounds (Caramella & Grunanger, 1984). In this paper, the structure of 4-(2-chlorophenyl)-3-(2,6-dichlorophenyl)spiroisoxazoline-5,2′-benzo-[4,5]imidazo[2,1-*b*]thiazol-3′-one dioxane hemisolvate, (I), is reported.



(I)

The molecule features a spiro junction (Fig. 1) linking a benzo[4,5]imidazo[2,1-*b*]thiazol-3-one ring and an isoxazoline ring. The benzo[4,5]imidazo[2,1-*b*]thiazol-3-one ring is nearly planar, with the mean deviation of the atoms comprising this plane being 0.029 (3) Å. The bond lengths for N1—C7, N2—

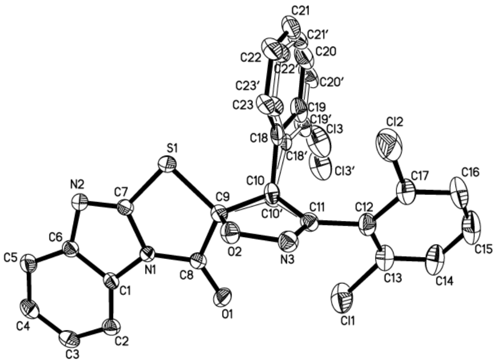


Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids. H atoms and the solvent molecule have been omitted for clarity. Primed atom labels denote atoms of the minor disorder component, which is shown with hollow bonds.

C7 and S1—C7 are 1.379 (3), 1.282 (3) and 1.740 (2) Å, and angles N1—C7—N2 and S1—C7—N1 are 115.1 (2) and 113.03 (18)°, respectively. These compare with 1.294 (6), 1.384 (6) and 1.714 (5) Å, and 115.8 (2) and 131.9 (4)°, respectively, reported for 6-(4-chlorobenzylidene)-2,3-dihydroimidazo[2,1-*b*]thiazol-5(6*H*)-one (Karolak-Wojciechowska, 1991). The isoxazoline ring is moderately puckered, the torsion angle for C11—N3—O2—C9 being 9.5 (3) Å. In between centrosymmetrically related pairs of (I) are centrosymmetric molecules of dioxane. The dioxane molecule serves to link molecules of (I) *via* O3···C8 contacts of 2.895 (3) Å.

Experimental

A mixture of 2,6-dichlorobenzonitrile oxide (2 mmol) and 2-(2-chlorobenzylidene)benzo[4,5]imidazo[2,1-*b*]thiazol-3-one (2 mmol) in dry benzene (30 ml) was heated under reflux for 36 h. After evaporation of the solvent, the residue was separated by column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give the spiro compound (m.p. 470–471 K). IR (KBr, cm^{-1}): 1751.5 (νC=O), 1612.6 (νC=N). ^{1}H NMR (CDCl$_3$, p.p.m.): 6.97 (1H, *s*), 7.24–7.99 (11H, *m*). The spiro compound (20 mg) was dissolved in dioxane (15 ml) and the solution was kept at room temperature for 10 d. Natural evaporation gave colorless single crystals of (I), suitable for X-ray analysis.

Crystal data

$C_{23}H_{12}Cl_3N_3O_2S\cdot0.5C_4H_8O_2$
$M_r = 544.82$
Triclinic, $P\overline{1}$
$a = 7.904$ (2) Å
$b = 9.166$ (3) Å
$c = 16.963$ (5) Å
$\alpha = 102.180$ (5)°
$\beta = 93.697$ (5)°
$\gamma = 94.789$ (5)°
$V = 1192.8$ (6) Å^3
$Z = 2$
$D_x = 1.517$ Mg m^{-3}
Mo *K*α radiation
Cell parameters from 881 reflections
$\theta = 3.4$–26.1°
$\mu = 0.51$ mm^{-1}
$T = 293$ (2) K
Plate, colorless
0.24 × 0.22 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997; Blessing, 1995)
$T_{min} = 0.862$, $T_{max} = 1.000$
9829 measured reflections
4851 independent reflections
3444 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 26.4$°
$h = -9 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.142$
$S = 1.08$
4851 reflections
377 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.68$ e Å^{-3}
$\Delta\rho_{min} = -0.56$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C8	1.373 (3)	N2—C6	1.417 (3)
N1—C7	1.379 (3)	S1—C7	1.740 (2)
N1—C1	1.406 (3)	S1—C9	1.832 (3)
N2—C7	1.282 (3)		
C8—N1—C7	117.87 (19)	N2—C7—N1	115.1 (2)
N3—O2—C9	109.15 (18)	N2—C7—S1	131.81 (19)
C7—S1—C9	91.46 (11)	N1—C7—S1	113.03 (18)
C11—N3—O2—C9	9.5 (3)	N1—C1—C6—N2	0.2 (3)
C2—C1—C6—N2	179.9 (2)	C8—N1—C7—S1	−3.1 (3)

H atoms were positioned geometrically and refined in the riding-model approximation [C—H = 0.93–0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$]. Atom C10 and the atoms of the attached 2-chlorophenyl ring are disordered over two sites. The ratio of site occupancies, from the refinement, was 0.55:0.45 (17).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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