

3-(2,6-Dichlorophenyl)-8-ethyl-4-phenyl-1-oxa-6-thia-2,8-diazaspiro[4.4]non-2-ene-7,9-dione

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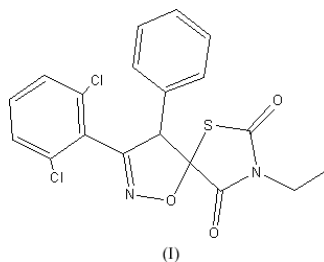
Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
 R factor = 0.052
 wR factor = 0.110
Data-to-parameter ratio = 13.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $\text{C}_{19}\text{H}_{14}\text{Cl}_2\text{N}_2\text{O}_3\text{S}$, which was synthesized by the intermolecular [3 + 2]-cycloaddition of 2,6-dichlorobenzonitrile oxide and 5-benzylidene-3-ethyl-thiazolidine-2,4-dione, there is a spiro connection between an isoxazoline ring and a thiazolidine ring. The thiazolidine ring is essentially planar, but the isoxazoline ring has a mean deviation from the plane of 0.0177 (3) Å. The dihedral angle between the mean planes of the rings is 88.5 (4)°.

Comment

Spiro compounds represent an important class of naturally occurring substances characterized by pronounced biological properties (Kobayashi *et al.*, 1991; James *et al.*, 1991). 1,3-Dipolar cycloaddition reactions are important processes for the construction of spiro compounds (Caramella & Grunanger, 1984). The structure of the title compound, (I), is reported here.



The molecular structure of (I) is illustrated in Fig. 1. A spiro-C atom is shared by an isoxazoline and a thiazolidine ring. The isoxazoline ring (O3/N2/C5/C4/C3) is almost planar, with a mean deviation of 0.0177 (3) Å. This is different from a previously reported structure, in which the isoxazoline ring is moderately puckered (Feng *et al.*, 1997). The O3–N2, C5–N2 and O3–C3 bond lengths and O3–N2–C5 and C3–O3–N2 angles (Table 1) can be compared with respective values of 1.413 (2), 1.281 (3) and 1.472 (2) Å, and 109.5 (2) and 108.6 (1)°, in the structure of Feng *et al.* (1997). The dihedral angle between the isoxazoline ring mean plane and the substituted phenyl-ring plane (C14–C19) is 65.9 (4)°, and that between the isoxazoline ring mean plane and the unsubstituted phenyl-ring plane (C8–C13) is 93.6 (4)°. The thiazolidine ring (S1/C1/N1/C2/C3) is essentially planar, with atoms O1 and O2 lying 0.0248 (2) and 0.0446 (2) Å, respectively, from this mean plane. The S1–C1 and S1–C3 bond lengths (Table 1) are different from the values of 1.774 (2) and 1.741 (2) Å reported for a related compound (Bozdag-Dundar *et al.*, 1999). The O1–C1 and N1–C1 bond lengths and C1–S1–C3, S1–C1–N1 and C2–N1–C1 angles can be compared with the values of 1.207 (3) and 1.370 (3) Å, and 91.77 (10), 110.80 (14) and 116.2 (2)°, respectively, reported by

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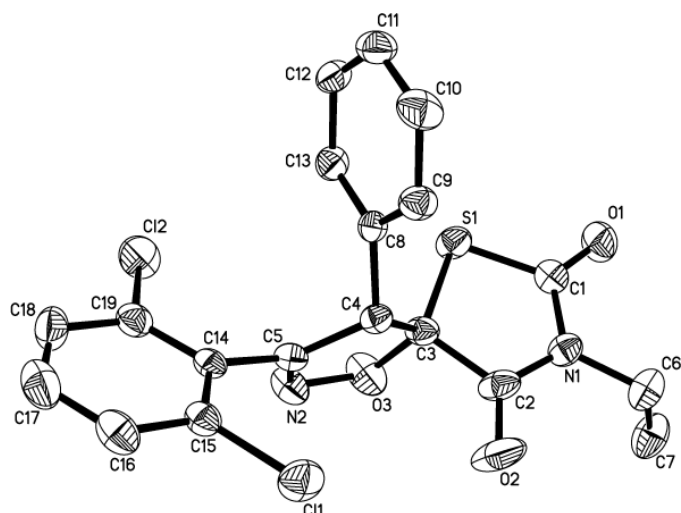


Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids. H atoms have been omitted.

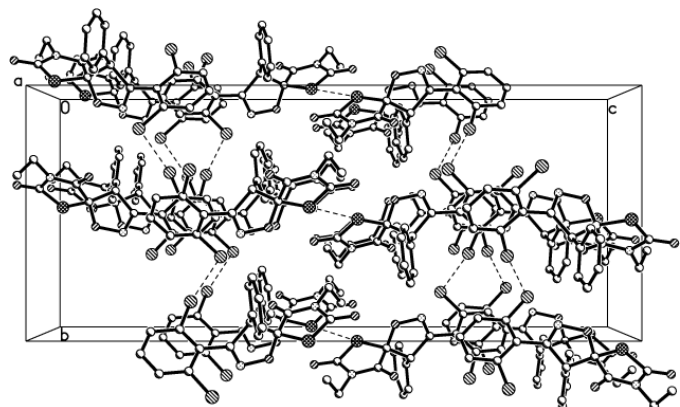


Figure 2
The crystal structure of (I), viewed along the *a* axis. Short intermolecular contacts are shown with dashed lines.

Bozdog-Dundar *et al.* (1999). The dihedral angle between the isoxazoline and thiazolidine rings is $88.5(4)^\circ$. The Cl—C bond lengths in the substituted phenyl ring are in good agreement with values reported in the literature (Busetti *et al.*, 1980; Sutherland & Ali-Adib, 1987). There are short intermolecular contacts of $3.481(2) \text{ \AA}$ for $\text{Cl1} \cdots \text{Cl2}(\frac{1}{2} - x, -\frac{1}{2} + y, z)$ and its symmetry-equivalent $\text{Cl2} \cdots \text{Cl1}(\frac{1}{2} - x, \frac{1}{2} + y, z)$, as shown in Fig. 2.

Experimental

A mixture of 2,6-dichlorobenzonitrile oxide (4 mmol) and 5-benzylidene-3-ethylthiazolidine-2,4-dione (2 mmol) in dry chloroform (30 ml) was heated under reflux for 4 d. After evaporation of the solvent, the residue was separated by column chromatography (silica gel, petroleum ether/ethyl acetate = 10:1) to give the title compound, (I). M.p. 432–433 K; IR (KBr): 2920 (CH_2CH_3), 1763, 1703 ($\text{C}=\text{O}$), 1602, 1580 ($\text{C}=\text{N}$, $\text{C}=\text{C}$) cm^{-1} ; ^1H NMR (CDCl_3 , p.p.m.): 1.27 (3H, *t*), 3.76 (2H, *m*), 6.09 (1H, *s*), 7.16–7.38 (8H, *m*). Compound (I) (20 mg) was dissolved in 15 ml chloroform, and the solution was kept

at room temperature for 10 d, yielding colorless single crystals of (I) suitable for X-ray analysis by evaporation of the solvent.

Crystal data

$\text{C}_{19}\text{H}_{14}\text{Cl}_2\text{N}_2\text{O}_3\text{S}$
 $M_r = 421.28$
Orthorhombic, *Pbca*
 $a = 10.0339(9) \text{ \AA}$
 $b = 12.5618(12) \text{ \AA}$
 $c = 30.220(3) \text{ \AA}$
 $V = 3809.1(7) \text{ \AA}^3$
 $Z = 8$
 $D_x = 1.469 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation
Cell parameters from 864 reflections
 $\theta = 2.7\text{--}21.2^\circ$
 $\mu = 0.47 \text{ mm}^{-1}$
 $T = 293(2) \text{ K}$
Plate, colorless
 $0.26 \times 0.24 \times 0.16 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 1998)
 $T_{\text{min}} = 0.825$, $T_{\text{max}} = 0.927$
13516 measured reflections

3335 independent reflections
2148 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.057$
 $\theta_{\text{max}} = 25.0^\circ$
 $h = -9 \rightarrow 11$
 $k = -14 \rightarrow 14$
 $l = -35 \rightarrow 33$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.110$
 $S = 1.07$
3335 reflections
245 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.36 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.33 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

| | | | |
|-----------|------------|----------|-----------|
| S1—C1 | 1.754 (4) | N1—C1 | 1.387 (4) |
| S1—C3 | 1.811 (3) | N1—C6 | 1.481 (4) |
| Cl1—C15 | 1.737 (4) | N2—C5 | 1.268 (4) |
| Cl2—C19 | 1.726 (4) | N2—O3 | 1.425 (4) |
| N1—C2 | 1.360 (5) | O3—C3 | 1.440 (4) |
| Cl1—S1—C3 | 93.10 (17) | N1—C1—S1 | 111.5 (3) |
| C2—N1—C1 | 116.5 (3) | N1—C2—C3 | 113.2 (3) |
| C5—N2—O3 | 109.0 (3) | O3—C3—C4 | 105.2 (3) |
| N2—O3—C3 | 110.5 (2) | C2—C3—S1 | 105.6 (2) |

H atoms were placed geometrically and refined with riding-model constraints.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINTE* (Bruker, 1998); program(s) used to solve structure: *SHELXTL* (Bruker, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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