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3,10-Bis(2,6-dichlorophenyl)-4,11-diphenyl-1,8-dioxa-2,9-diazadispiro[4.1.4.2]trideca-2,9-dien-6-one

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Key indicators

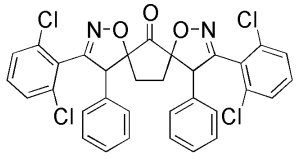
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.048
wR factor = 0.108
Data-to-parameter ratio = 16.2

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

The title compound, $C_{33}H_{22}Cl_4N_2O_3$, was synthesized by the intermolecular [3+2]-cycloaddition of 2,6-dichlorobenzonitrile oxide and 2,5-bis(benzylidene)cyclopentanone. Three five-membered rings are linked by two spiro-C atoms. The cyclopentane ring has a twist conformation and the two isoxazoline rings are envelopes. The crystal packing of the title compound is stabilized by weak C—H···Cl and C—H···π interactions.

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 processes for the construction of spiro-compounds (Caramella & Grunanger, 1984). In this paper, the structure of the title compound, (I), is reported.



(I)

The title compound was synthesized by the intermolecular [3+2]-cycloaddition of 2,6-dichlorobenzonitrile oxide and 2,5-bis(benzylidene)cyclopentanone. The molecular structure of (I) is illustrated in Fig. 1. There are two spiro junctions in the molecule, which contains two isoxazoline rings and a cyclopentane ring. The two isoxazoline rings adopt envelope conformations. The cyclopentane ring has a twist conformation. The structure of 4,11-bis-4-chlorophenyl)-3,10-bis(2,6-dichlorophenyl)-1,8-dioxa-2,9-diazadispiro[4.1.4.2]trideca-2,9-dien-6-one–methanol–chloform (1/0.75/1) has been reported

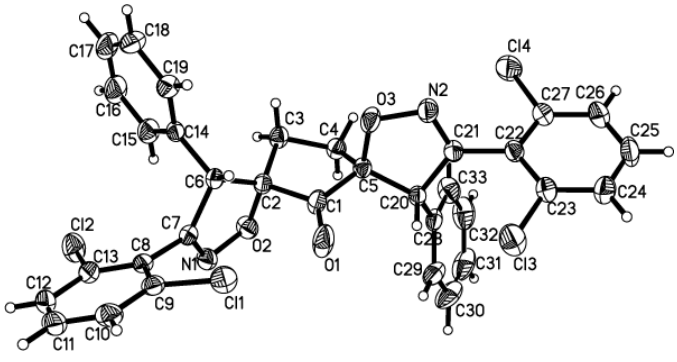


Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids.

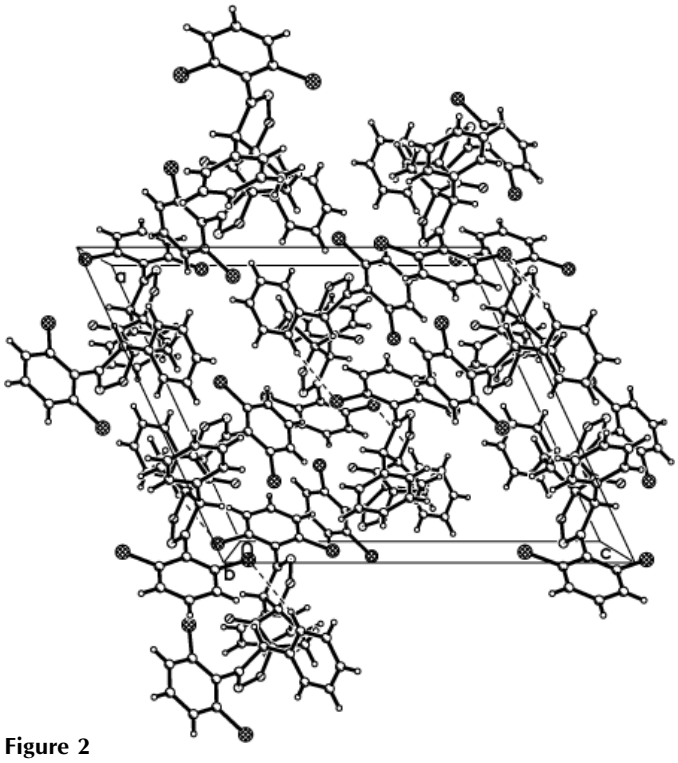


Figure 2
The crystal structure of (I), viewed along the *b* axis. Dashed lines indicate C—H···Cl interactions.

previously (Li *et al.*, 2003). The crystal packing of the title compound is stabilized by weak C—H···Cl and C—H···π interactions (Fig. 2).

Experimental

A mixture of 2,6-dichlorobenzonitrile oxide (3 mmol) and 2,5-bis-(benzylidene)cyclopentanone (1.5 mmol) in dry benzene (30 ml) was heated under reflux for 40 h. After evaporation of the solvent, the residue was separated by column chromatography (silica gel, petroleum ether–ethyl acetate = 5:1) to give the title compound, (I). M.p. 527–529 K; IR (KBr): 1763 (C═O), 1595, 1579 (C═N, C═C) cm^{-1}; ^{1}H NMR ($CDCl_3$, p.p.m.): 1.75–1.85 (2H, *m*), 1.91–2.04 (2H, *m*), 5.65 (2H, *s*), 7.14–7.30 (16H, *m*); 20 mg of (I) was dissolved in 15 ml chloroform and methanol mixed solvent, and the solution was kept at room temperature for 10 d, yielding colorless single crystals of (I) by evaporation.

Crystal data

$C_{33}H_{22}Cl_4N_2O_3$
M_r = 636.33
Monoclinic, $P2_1/n$
a = 14.464 (3) Å
b = 13.377 (3) Å
c = 17.080 (4) Å
β = 114.954 (4)°
V = 2996.2 (13) Å^3
Z = 4
D_x = 1.411 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 950 reflections
θ = 2.9–25.1°
μ = 0.43 mm^{-1}
T = 293 (2) K
Block, colorless
0.28 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.799, T_{max} = 0.917
24 667 measured reflections
6135 independent reflections
3963 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 26.4°
$h = -18 \rightarrow 18$
$k = -16 \rightarrow 16$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.108
S = 1.01
6135 reflections
379 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

H atoms were positioned geometrically, with C—H = 0.93–0.98 Å, and refined in a riding model, with U_{iso}(H) = 1.2U_{eq}(carrier).

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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