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3,10-Bis(2,6-dichlorophenyl)-4,11-diphenyl-1,8-dioxa-2,9-diazadispiro[4.1.4.3]tetradeca-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 = 0.041
wR factor = 0.108
Data-to-parameter ratio = 15.8

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

The title compound, $C_{34}H_{24}Cl_4N_2O_3$, was synthesized by the intermolecular [3+2]-cycloaddition of 2,6-dichlorobenzonitrile oxide and 2,6-bis(benzylidene)cyclohexanone. Three of the rings are linked by two spiro–C atoms. The cyclohexane ring has a chair conformation and the two five-membered isoxazoline rings are envelopes. The crystal packing of the title compound is stabilized by weak C—H···O and C—H···π interactions.

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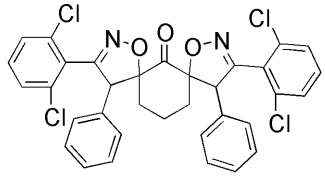
Comment

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 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 2,6-bis(benzylidene)cyclohexanone. The molecular structure of (I) is illustrated in Fig. 1. There are two spiro junctions in the

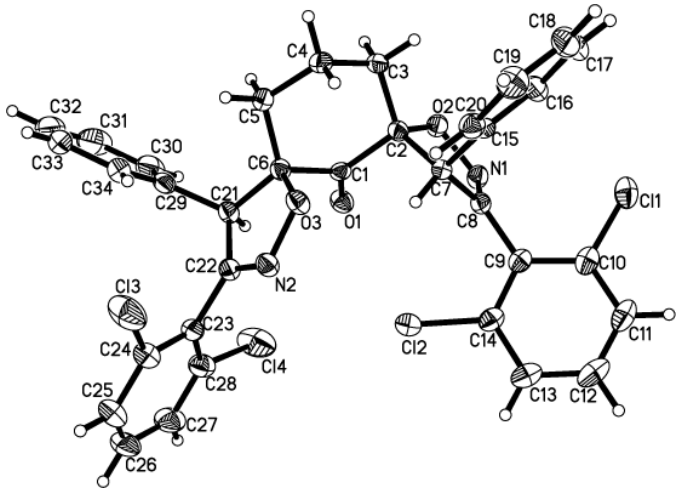


Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids.

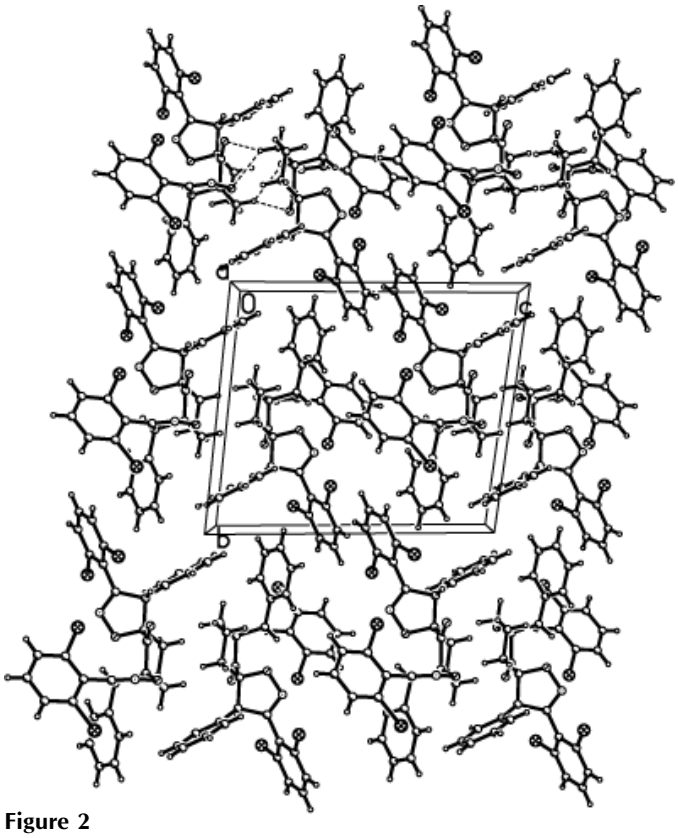


Figure 2
The crystal structure of (I), viewed along the *a* axis. Dashed lines indicate C—H···O interactions.

molecule, which contains two isoxazoline rings and a cyclohexane ring. The two isoxazoline rings adopt envelope conformations. The cyclohexane ring has a chair conformation. The structure of 4,11-bis(4-chlorophenyl)-3,10-bis(2,6-dichlorophenyl)-1,8-dioxa-2,9-diazadispiro[4.1.4.3]tetradeca-2,9-dien-6-one hydrate has been reported previously (Li *et al.*, 2003). The crystal packing of the title compound is stabilized by weak C—H···O and C—H···π interactions (Fig. 2).

Experimental

A mixture of 2,6-dichlorobenzonitrile oxide (3 mmol) and 2,6-bis-(benzylidene)cyclohexanone (1.5 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 title compound, (I). M.p. 589–590 K; IR (KBr): 1734.1 (C═O), 1601.1, 1581.7 (C═N, C═C) cm^{-1}; ^1H NMR ($CDCl_3$, p.p.m.): 1.61 (4H, *m*), 1.95 (2H, *m*), 6.23 (2H, *s*), 7.14–7.44 (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 days, yielding colorless single crystals of (I) by evaporation.

Crystal data

$C_{34}H_{24}Cl_4N_2O_3$
M_r = 650.35
Triclinic, $P\overline{1}$
a = 8.764 (3) Å
b = 12.872 (4) Å
c = 14.284 (5) Å
α = 94.490 (6)°
β = 96.563 (6)°
γ = 108.739 (5)°
V = 1504.5 (9) Å^3
Z = 2
D_x = 1.436 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 996 reflections
θ = 2.4–26.3°
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} = 0.864, T_{max} = 0.917
12 694 measured reflections
6118 independent reflections
4357 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 26.5°
$h = -10 \rightarrow 10$
$k = -16 \rightarrow 16$
$l = -16 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.108
S = 1.03
6118 reflections
388 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
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 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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