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Key indicators

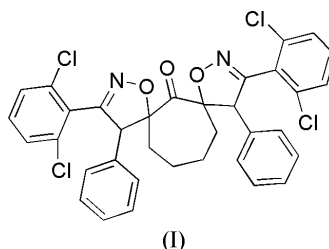
Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
 R factor = 0.048
 wR factor = 0.135
Data-to-parameter ratio = 16.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.3,10-Bis(2,6-dichlorophenyl)-4,11-diphenyl-
1,8-dioxo-2,9-diazadispiro[4.1.4.4]pentadeca-
2,9-dien-6-one

The title compound, $\text{C}_{35}\text{H}_{26}\text{Cl}_4\text{N}_2\text{O}_3$, was synthesized by the intermolecular [3 + 2]-cycloaddition of 2,6-dichlorobenzonitrile oxide and 2,7-bis(benzylidene)cycloheptanone. Three of the rings are linked by two spiro-C atoms. The cycloheptanone ring adopts a chair conformation and the two five-membered isoxazoline rings are envelopes.

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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).



The title compound, (I), was synthesized by the intermolecular [3 + 2]-cycloaddition of 2,6-dichlorobenzonitrile oxide and 2,7-bis(benzylidene)cycloheptanone. The molecular structure, illustrated in Fig. 1, contains three spiro-linked rings, *viz.* a cycloheptanone ring and two isoxazoline rings. The

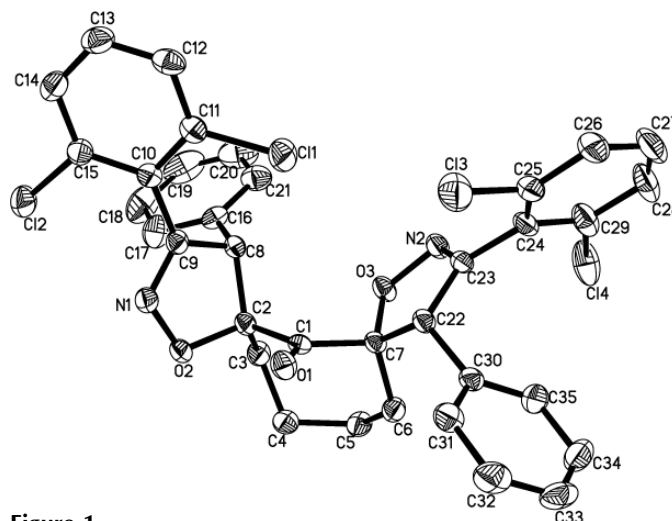


Figure 1

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

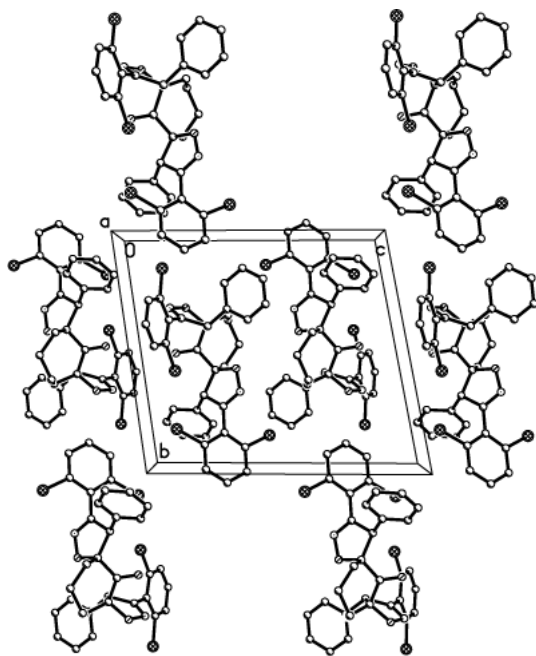


Figure 2
The crystal structure of (I), viewed along the *a* axis.

seven-membered cycloheptanone ring has a chair conformation. Attached to the isoxazoline rings are phenyl and 2,6-dichlorophenyl substituents. The two isoxazoline rings have envelope conformations.

Experimental

A mixture of 2,6-dichlorobenzonitrile oxide (3 mmol) and 2,7-bis-(benzylidene)cycloheptanone (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. 517–518 K; IR (KBr): 1720.6 (C=O), 1601.0, 1579.8 (C=N and C=C) cm^{-1} . ^1H NMR (CDCl_3 , p.p.m.): 1.10–1.61 (4H, *m*, $-\text{CH}_2$), 1.71–2.22 (4H, *m*, $-\text{CH}_2$), 5.74 (1H, *s*, $-\text{CH}$), 5.97 (1H, *s*, $-\text{CH}$), 7.09–7.42 (16H, *m*, ArH). 20 mg of (I) was dissolved in 15 ml chloroform and the solution kept at room temperature for 10 d to give colorless single crystals of (I) by evaporation.

Crystal data

$\text{C}_{35}\text{H}_{26}\text{Cl}_4\text{N}_2\text{O}_3$
 $M_r = 664.38$
Triclinic, $P\bar{1}$
 $a = 9.095$ (3) Å
 $b = 13.010$ (4) Å
 $c = 14.078$ (4) Å
 $\alpha = 79.674$ (5)°
 $\beta = 86.207$ (5)°
 $\gamma = 72.793$ (4)°
 $V = 1565.3$ (8) Å³

$Z = 2$
 $D_x = 1.410$ Mg m⁻³
Cell parameters from 954 reflections
 $\theta = 3.5$ – 26.1 °
 $\mu = 0.42$ mm⁻¹
 $T = 293$ (2) K
Block, colorless
 $0.28 \times 0.22 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 1997)
 $T_{\text{min}} = 0.834$, $T_{\text{max}} = 0.960$
9083 measured reflections

6351 independent reflections
3949 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$
 $\theta_{\text{max}} = 26.4$ °
 $h = -11 \rightarrow 10$
 $k = -16 \rightarrow 16$
 $l = -17 \rightarrow 8$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.135$
 $S = 1.06$
6351 reflections
397 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.40$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.40$ e Å⁻³

H atoms were positioned geometrically, with C–H = 0.93–0.98 Å, and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$.

Data collection: SMART (Bruker, 1997); cell refinement: SMART; data reduction: SAINT (Bruker, 1997); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1997); software used to prepare material for publication: SHELXTL.

References

- Bruker (1997). *SADABS*, *SMART*, *SAINTE* and *SHELXTL*. Versions 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Caramella, P. & Grunanger, P. (1984). *1,3-Dipolar Cycloaddition Chemistry*, Vol. 1, edited by A. Padwa, pp. 291–312. New York: Wiley.
James, D. M., Kunze, H. B. & Faulkner, D. J. (1991). *J. Nat. Prod.* **54**, 1137–1140.
Kobayashi, J., Tsuda, M., Agemi, K., Shigemori, H., Ishibashi, M., Sasaki, T. & Mikami Y. (1991). *Tetrahedron*, **47**, 6617–6622.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.