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4,11-Bis(4-chlorophenyl)-3,10-bis(2,6-dichlorophenyl)-1,8-dioxa-2,9-diaza-dispiro[4.1.4.4]pentadeca-2,9-dien-6-one

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Key indicators

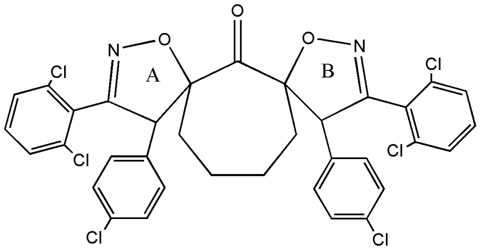
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.065
wR factor = 0.099
Data-to-parameter ratio = 14.5

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

The title compound, $C_{35}H_{24}Cl_6N_2O_3$, which was synthesized by the [3 + 2]-cycloaddition of 2,6-dichlorobenzonitrile oxide and 2,7-bis(4-chlorobenzylidene)cycloheptanone, contains two independent molecules in the asymmetric unit. Three rings are linked by two spiro-C atoms. The cycloheptanone ring adopts a chair conformation and the two five-membered isoxazoline rings have envelope conformations.

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 structure of the title compound, (I), is reported here.



(I)

The molecular structure of (I) is illustrated in Fig. 1. (I) contains three spiro-linked rings, *viz.* a cycloheptanone ring and two isoxazoline rings. The seven-membered cycloheptanone ring has a chair conformation. The two isoxazoline rings are attached to this central ring through spiro-C atoms; this is different from the similar structure in the literature (Li *et al.*, 2003), which has approximate C_2 symmetry. Attached to the isoxazoline rings are 4-chlorophenyl and 2,6-dichlorophenyl substituents. The two isoxazoline rings (*A* and *B*) are non-planar, with envelope conformations. O2/N1/C8/C9 (in ring *A*) and O3/N2/C10/C11 (in ring *B*) form nearly planar arrangements. The spiro-atom C2 lies 0.440 (3) Å from the O2/N1/C8/C9 plane in ring *A* and C7 lies 0.194 (3) Å from the O3/N2/C10/C11 plane in ring *B*, forming the flaps of the envelopes. The dihedral angle between the C9/C2/O2 plane and the O2/N1/C8/C9 mean plane is 28.6 (4)°, and that between the C11/C7/O3 plane and the O3/N2/C10/C11 mean plane is 12.2 (4)°, similar to 12.0 (4) and 26.7 (4)° reported in the literature (Li *et al.*, 2003). Similar values are obtained for the second molecule in the asymmetric unit.

The dihedral angles between the two substituted phenyl rings on rings *A* and *B* are 90.4 (3) and 104.2 (3)°, respectively. The dihedral angle between the two isoxazoline ring envelope

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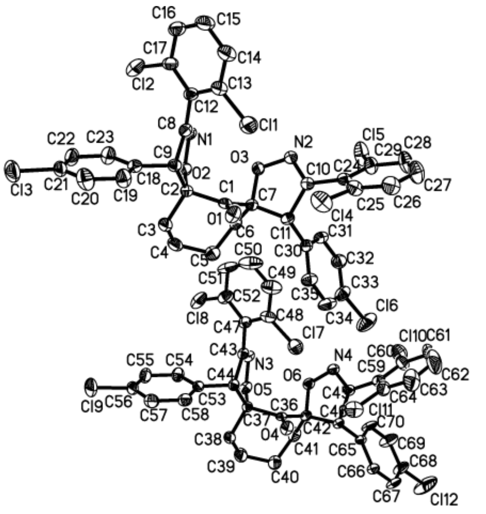

Figure 1
The asymmetric unit of (I), with 30% probability displacement ellipsoids and the atom-numbering scheme.

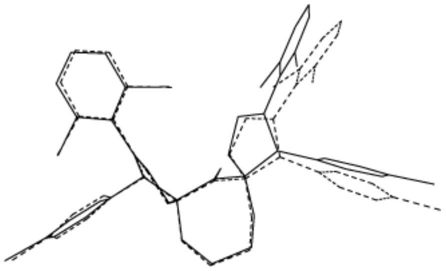

Figure 2
Least-squeares superposition of the two independent molecules in the asymmetric unit.

flaps (C9/C2/O2 and C11/C7/O3), which form the spiro junctions with the central ring, is 86.7 (3)°. Similar values are obtained for the second molecule in the asymmetric unit.

The differences between the two independent molecules in the asymmetric unit are shown in Fig. 2 and Table 1.

Experimental

A mixture of 2,6-dichlorobenzonitrile oxide (3 mmol) and 2,7-bis(4-chlorobenzylidene)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. 535–536 K; IR (KBr): 1718 (C=O), 1598, 1582 (C=N, C=C) cm^{-1}; ^{1}H NMR (CDCl$_3$, p.p.m.): 1.10–1.61 (4H, *m*), 1.71–2.22 (4H, *m*), 5.68 (1H, *s*), 5.91 (1H, *s*), 7.15–7.35 (14H, *m*); 20 mg of (I) was dissolved in 15 ml chloroform, and the solution was kept at room temperature for 10 d, to give colorless single crystals of (I) by evaporation.

Crystal data

$C_{35}H_{24}Cl_6N_2O_3$
M_r = 733.26
Monoclinic, $C2/c$
a = 33.510 (9) Å
b = 12.419 (5) Å
c = 32.946 (9) Å
β = 95.152 (8)°
V = 13655 (8) Å^3
Z = 16

D_x = 1.427 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 914 reflections
θ = 3.3–23.7°
μ = 0.54 mm^{-1}
T = 293 (2) K
Plate, colorless
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{\min}$ = 0.835, $T_{\max}$ = 0.947
27390 measured reflections

11983 independent reflections
5317 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.099
$\theta_{\max}$ = 25.0°
$h = -39 \rightarrow 39$
$k = -9 \rightarrow 14$
$l = -39 \rightarrow 35$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.099
S = 0.97
11983 reflections
829 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
$\Delta\rho_{\max}$ = 0.25 e Å^{-3}
$\Delta\rho_{\min}$ = −0.36 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—O2	1.423 (4)	N3—O5	1.418 (3)
N2—O3	1.400 (4)	N4—O6	1.409 (4)
N2—O3—C7	109.5 (3)	N4—O6—C42	108.7 (3)
N2—O3—C7—C11	−12.4 (4)	N4—O6—C42—C46	−22.3 (3)
O2—N1—C8—C9	−3.3 (5)	O5—N3—C43—C47	179.1 (3)
O3—N2—C10—C24	177.9 (3)	O5—N3—C43—C44	−2.8 (4)
O3—N2—C10—C11	−0.5 (5)	O6—N4—C45—C59	177.0 (3)
N4—O6—C42—C41	−148.7 (3)	O6—N4—C45—C46	3.0 (5)

H atoms were refined as riding, with C—H distances of 0.93 or 0.97 Å.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* and *SHELXTL* (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*.

References

Bruker (1998). *SADABS, SMART* (Version 5.10), *SAINT* (Version 4.0) and *SHELXTL*. 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. & Mikamiy, Y. (1991). *Tetrahedron*, **47**, 6617–6622.

Li, X. F., Feng, Y. Q., Zhuang, J. P. & Hu, X. F. (2003). *Acta Cryst.* E**59**, o247–o249.

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