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4,11-Bis(4-chlorophenyl)-3,10-bis(2,6-dichlorophenyl)-1,8-dioxa-2,9-diaza-dispiro[4.1.4.2]trideca-2,9-dien-6-one–chloroform–methanol (1/1/0.75)

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The title compound, $C_{32}H_{20}Cl_6N_2O_3\cdot CHCl_3\cdot 0.75CH_4O$, was synthesized by the intermolecular [3 + 2]-cycloaddition of 2,6-dichlorobenzonitrile oxide and 2,5-bis(4-chlorobenzylidene)-cyclopentanone. There are three five-membered rings linked by two spiro-C atoms, with approximate C_2 symmetry. The cyclopentane ring has a twist conformation and the two isoxazoline rings are envelopes.

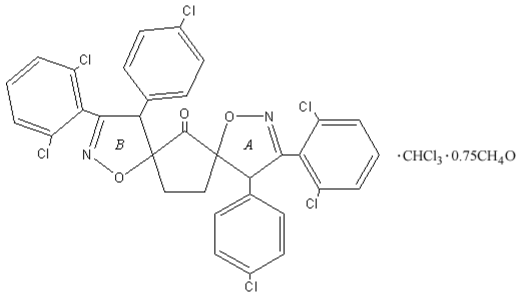
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
H-atom completeness 88%
Disorder in solvent or counterion
R factor = 0.056
wR factor = 0.195
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.

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 provide an important route for the construction of spiro-compounds (Caramella & Grunanger, 1984).



(I)

Compound (I) contains three spiro-linked rings, namely a cyclopentanone ring and two isoxazoline rings. The five-membered cyclopentanone ring has a twist conformation. The two isoxazoline rings are attached to this central ring through spiro-C atoms, giving 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—C7—C6 (ring A) and O3—N2—C21—C20 (ring B) form almost exactly planar arrangements; the torsion angles O2—N1—C7—C6 of −2.9 (5)° and O3—N2—C21—C20 of −3.0 (5)° are different from that of −0.9 (5)° in a related structure, *viz.* 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 0.75-hydrate (Li *et al.*, 2003). Spiro-atom C2 lies 0.168 (3) Å from the O2/N1/C7/C6 plane in ring A and C5 is 0.345 (3) Å from the O3/N2/C21/C20 plane in ring B, these two atoms forming the flaps of the envelopes.

The bond lengths O2—N1 and O3—N2 are both 1.410 (4) Å, comparable to 1.413 (2) Å in a related structure, *viz.* 8-methyl-3-phenyl-1-oxa-2,8-diaza-spiro[4.5]dec-2-ene (Feng *et al.*, 1997).

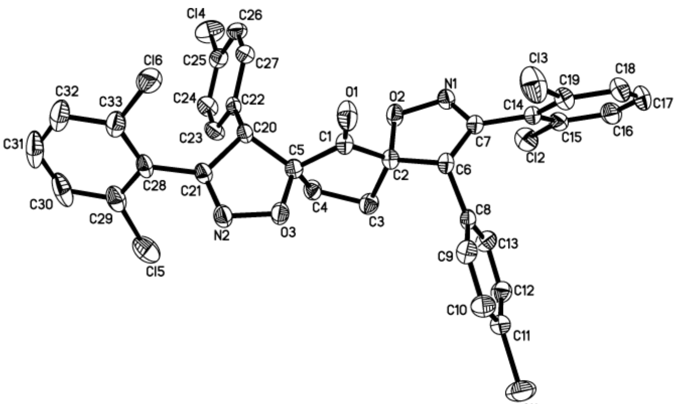


Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids.

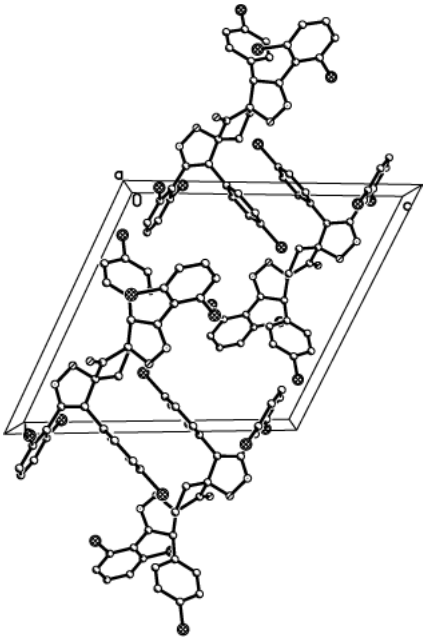


Figure 2
The crystal structure of (I), viewed along the *a* axis, omitting the solvent molecules.

The dihedral angle between the two substituted phenyl rings on ring *A* is 94.9 (3)°, while that between the two substituted phenyl rings on ring *B* is 74.8 (3)°. The dihedral angle between the two isoxazoline ring envelope flaps (C6/C2/O2 and C20/C5/O3), which form the spiro linkages with the central ring, is 44.5 (3)°.

Experimental

A mixture of 2,6-dichlorobenzonitrile oxide (3 mmol) and 2,5-bis(4-chlorobenzylidene)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. 535–536 K; IR (KBr): 1765 (C=O), 1595, 1581 (C=N and C=C) cm^{-1}; ^{1}H NMR (CDCl$_3$, p.p.m.): 1.77–1.81 (2H, *m*), 1.99–2.02 (2H, *m*), 5.62 (2H, *s*), 7.18–7.37 (14H, *m*); 20 mg of (I) was dissolved in a mixture of 15 ml chloroform and methanol, and the solution was kept at room temperature for 10 d, to give colorless single crystals of (I) on evaporation.

Crystal data

$C_{33}H_{20}Cl_6N_2O_3 \cdot CHCl_3 \cdot 0.75CH_4O$
$M_r = 848.61$
Triclinic, $P\overline{1}$
$a = 9.896$ (3) Å
$b = 14.724$ (4) Å
$c = 15.550$ (5) Å
$\alpha = 111.815$ (5)°
$\beta = 101.814$ (5)°
$\gamma = 103.510$ (5)°
$V = 1935.5$ (10) Å^3
$Z = 2$
$D_x = 1.456$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 983 reflections
$\theta = 3.2$–25.4°
$\mu = 0.69$ mm^{-1}
$T = 293$ (2) K
Plate, colorless
0.28 × 0.18 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{\min} = 0.804$, $T_{\max} = 0.908$
10032 measured reflections
6787 independent reflections
4617 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.020$
$\theta_{\max} = 25.0°$
$h = -11 \rightarrow 11$
$k = -16 \rightarrow 17$
$l = -18 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.195$
$S = 1.10$
6787 reflections
467 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.87$ e Å^{-3}
$\Delta\rho_{\min} = -0.39$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C11	1.742 (5)	O2—N1	1.410 (4)
O1—C1	1.192 (5)	O3—N2	1.410 (4)
N1—O2—C2	109.9 (3)	O2—C2—C1	103.3 (3)
N2—O3—C5	108.9 (3)	O2—C2—C6	104.5 (3)
C7—N1—O2	109.6 (3)	O3—C5—C20	103.7 (3)
C21—N2—O3	109.1 (3)	C7—C6—C2	100.1 (3)
O1—C1—C5	125.8 (4)		

H atoms were included in the riding-model approximation with displacement parameters related to the atoms to which they were bonded. H atoms were not included for disordered methanol molecules.

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*.

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