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13-Benzyl-3,10-bis(2,6-dichlorophenyl)-4,11-diphenyl-1,8-dioxa-2,9,13-triazadispiro[4.1.4.3]tetradeca-2,9-dien-6-one chloroform solvate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
Disorder in solvent or counterion
R factor = 0.064
wR factor = 0.196
Data-to-parameter ratio = 14.0

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

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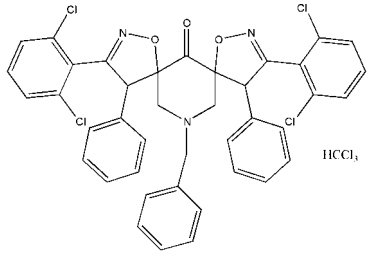
The title compound, $C_{40}H_{29}C_{l4}N_3O_3.CHCl_3$, was synthesized by the intermolecular [3 + 2]-cycloaddition of 2,6-dichlorobenzonitrile oxide and 1-benzyl-3,5-dibenzylidenepiperidin-4-one. There are three rings linked by two spiro-C atoms. The piperidin-4-one ring adopts a chair conformation and the two five-membered isoxazoline rings are envelopes.

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 piperidin-4-one ring and two isoxazoline rings. The six-membered piperidin-4-one ring has a chair conformation. Attached to the isoxazoline rings are phenyl and 2,6-dichlorophenyl substituents.

The two isoxazoline rings (*A* and *B*) are non-planar, with envelope conformations. O2/N2/C7/C6 (ring *A*) and O3/N3/C9/C8 (ring *B*) form nearly planar arrangements, with mean deviations of 0.0088 and 0.0060 Å, respectively. The spiro-atom C2 lies 0.3436 (3) Å out from the plane of ring *A* and C5 is −0.3772 (3) Å out from the plane of ring *B*, forming the flaps of the envelopes. The dihedral angle between the C6/C2/O2 and O2/N2/C7/C6 mean planes is 21.9 (4)°. The corresponding angle between the C8/C5/O3 and O3/N3/C9/C8 mean planes is 24.1 (4)°. The dihedral angle between the two aryl rings on ring *A* is 97.7 (3)°, while that between the two aryl rings on ring *B* is 78.8 (3)°.

Experimental

A mixture of 2,6-dichlorobenzonitrile oxide (3 mmol) and 1-benzyl-3,5-dibenzylidenepiperidin-4-one (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. 442–443 K; IR (KBr): 1736 (C═O), 1602, 1580 (C═N, C═C)

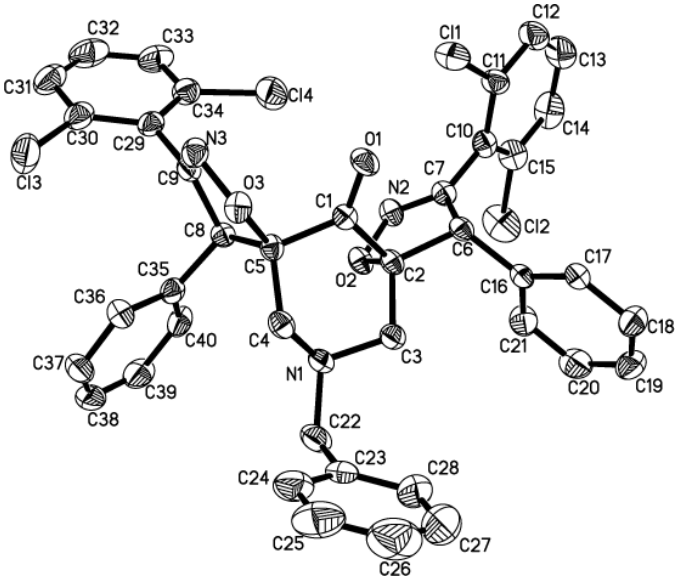


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms and the $CHCl_3$ molecule have been omitted for clarity.

cm^{-1}; ^1H NMR ($CDCl_3$, p.p.m.): 2.42 (2H, *d*), 2.79 (2H, *d*), 3.10 (2H, *m*), 6.13 (2H, *s*), 6.94–7.37 (21H, *m*). 20 mg of (I) was dissolved in 15 ml chloroform; the solution was kept at room temperature for 10 d and natural evaporation gave colorless single crystals of (I) suitable for X-ray analysis.

Crystal data

$C_{40}H_{29}Cl_4N_3O_3\cdot CHCl_3$	$Z = 2$
$M_r = 860.83$	$D_x = 1.414$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 11.905$ (18) Å	Cell parameters from 688 reflections
$b = 12.046$ (17) Å	$\theta = 2.1$–$19.7°$
$c = 15.17$ (2) Å	$\mu = 0.53$ mm^{-1}
$\alpha = 87.89$ (3)°	$T = 293$ (2) K
$\beta = 81.20$ (3)°	Block, colorless
$\gamma = 70.20$ (3)°	0.22 × 0.20 × 0.18 mm
$V = 2022$ (5) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	7038 independent reflections
	3463 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.039$
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)	$\theta_{max} = 25.0°$
	$h = -14 \rightarrow 13$
$T_{min} = 0.697$, $T_{max} = 0.909$	$k = -14 \rightarrow 10$
10516 measured reflections	$l = -18 \rightarrow 16$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.064$	$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.196$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.07$	$\Delta\rho_{max} = 0.35$ e Å^{-3}
7038 reflections	$\Delta\rho_{min} = -0.31$ e Å^{-3}
504 parameters	

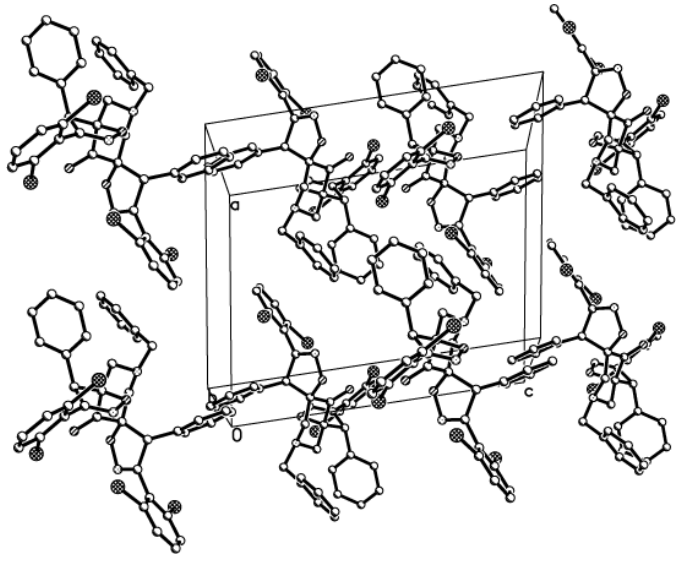


Figure 2
The crystal packing of (I), viewed along the *b* axis, with $CHCl_3$ molecules omitted.

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.196 (5)	N1—C4	1.453 (6)
O2—N2	1.407 (5)	N2—C7	1.276 (5)
O3—N3	1.413 (5)	N3—C9	1.280 (6)
N2—O2—C2	108.6 (3)	C7—N2—O2	108.8 (3)
N3—O3—C5	108.8 (3)	C9—N3—O3	107.8 (4)
C4—N1—C3	112.0 (4)		
C2—O2—N2—C7	12.5 (5)	O1—C1—C2—C6	−1.8 (6)
C5—O3—N3—C9	14.6 (4)		

H atoms were positioned geometrically and refined with riding-model constraints.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* and *SHELXTL* (Bruker, 1998); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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