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

Xiao-Fang Li,* Ya-Qing Feng, Bo Gao and Nan Li

School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, People's Republic of China

Correspondence e-mail: lxf7212@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.048
wR factor = 0.132
Data-to-parameter ratio = 14.2

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

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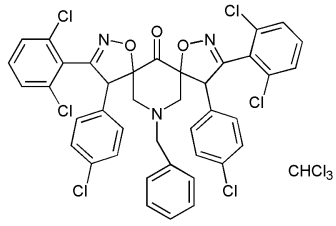
The title compound, $C_{40}H_{27}Cl_6N_3O_3\cdot CHCl_3$, was synthesized by the intermolecular [3 + 2]-cycloaddition of 2,6-dichlorobenzonitrile oxide and 1-benzyl-3,5-bis(4-chlorobenzylidene)-piperidin-4-one. There are three rings linked by two spiro-C atoms. The piperidin-4-one ring adopts a chair 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). 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 1-benzyl-3,5-bis(4-chlorobenzylidene)piperidin-4-one. The molecular structure of (I) is illustrated in Fig. 1. It is very similar to the structure of 13-benzyl-3,10-bis(2,6-dichlorophenyl)-4,11-diphenyl-1,8-dioxa-2,9,13-triaza-dispiro[4.1.4.3]-tetradeca-2,9-dien-6-one, (II), reported earlier as the chloroform solvate (Li *et al.*, 2003). Compound (II) has unsubstituted phenyl groups at the 4- and 11- positions, in contrast to the title compound, which has 4-chlorophenyl substituents in the same positions.

Molecule (I) contains three spiro-linked rings, *viz*. two isoxazoline rings and a piperidin-4-one ring. The two isoxazoline rings adopt envelope conformations. The piperidin-4-one ring has a chair conformation. 4-Chlorophenyl and 2,6-dichlorophenyl substituents are attached to the isoxazoline rings.

The two isoxazoline rings (*A* and *B*) in molecule (I) are non-planar and have envelope conformations similar to those observed in (II). The O2/N2/C7/C6 (ring *A*) and O3/N3/C21/C20 (ring *B*) fragments form nearly planar arrangments, with mean deviations of 0.0100 and 0.0051 Å, respectively. Spiro

atoms C2 and C5 are displaced by 0.4235 (3) and 0.3380 (4) Å from the *A* and *B* planes, respectively, thus forming the flaps of the envelopes. The dihedral angle between the C6/C2/O2 and O2/N2/C7/C6 mean planes is 27.3 (4)°; the corresponding angle between the C20/C5/O3 and O3/N3/C21/C20 mean planes is 21.5 (3)°.

The cell dimensions and molecular packing in the crystal structure of (I) (Fig. 2) are also strikingly similar to those of crystal (II).

Experimental

A mixture of 2,6-dichlorobenzonitrile oxide (3 mmol) and 1-benzyl-3,5-bis-(4-chlorobenzylidene)piperidin-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. 441–442 K; IR (KBr): 1735 (C═O), 1602, 1583 (C═N, C═C) cm^{-1}; ^1H NMR ($CDCl_3$, p.p.m.): 2.46 (2H, *d*), 2.79 (2H, *d*), 3.17 (2H, *m*), 6.10 (2H, *s*), 6.92–7.29 (19H, *m*). 20 mg of (I) was dissolved in 15 ml of 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

$C_{40}H_{27}Cl_6N_3O_3\cdot CHCl_3$	$Z = 2$
$M_r = 929.71$	$D_x = 1.484$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 11.948$ (4) Å	Cell parameters from 866 reflections
$b = 12.066$ (4) Å	$\theta = 3.4$–$25.2°$
$c = 15.674$ (5) Å	$\mu = 0.65$ mm^{-1}
$\alpha = 97.542$ (5)°	$T = 293$ (2) K
$\beta = 92.617$ (5)°	Block, colorless
$\gamma = 111.000$ (5)°	0.26 × 0.22 × 0.20 mm
$V = 2080.8$ (11) $Å^3$	

Data collection

Bruker SMART CCD area-detector diffractometer	8424 independent reflections
	5461 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.022$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	$\theta_{max} = 26.4°$
	$h = -14 \rightarrow 14$
$T_{min} = 0.759$, $T_{max} = 0.878$	$k = -13 \rightarrow 15$
12075 measured reflections	$l = -19 \rightarrow 17$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.048$	$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.132$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.02$	$\Delta\rho_{max} = 0.40$ e $Å^{-3}$
8424 reflections	$\Delta\rho_{min} = -0.58$ e $Å^{-3}$
594 parameters	

H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and refined in a riding-model approximation [U_{iso}(H) = 1.2U_{eq}(carrier)]. The disordered 2,6-dichlorophenyl group was refined as a rigid group with the fixed geometry of a regular hexagon [C—C = 1.39 Å], with two positions with occupancies of 0.81 (1) and 0.19 (1). The benzyl group is disordered over two positions with occupancies of 0.43 (1) and 0.57 (1). The bond lengths and angles involving the disordered atoms, as well as the U_{ij} values of the disordered atoms, were suitably restrained.

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*.

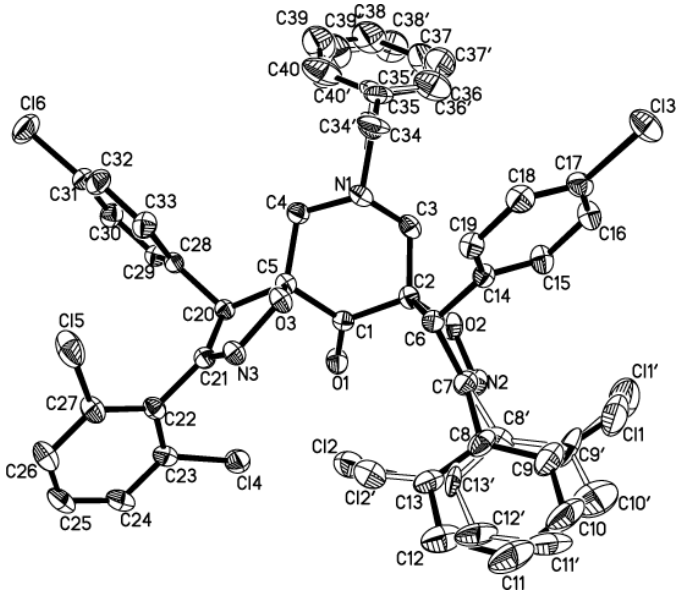


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms and the $CHCl_3$ molecule have been omitted for clarity. Primed atom labels denote atoms of the minor disorder component, which is shown with open bonds.

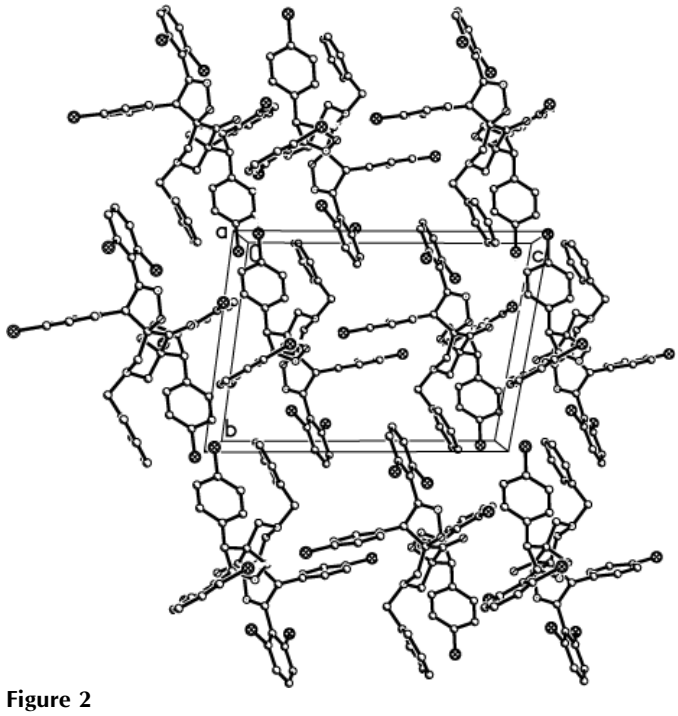


Figure 2
The crystal packing of (I), viewed along the *a* axis. H atoms, the $CHCl_3$ molecule and the minor disorder component have been omitted for clarity.

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