

4,11-Bis(4-methoxyphenyl)-3,10-bis(2,6-dichlorophenyl)-1,8-dioxo-2,9-diazadispiro[4.1.4.3]tetradeca-2,9-dien-6-one–chloroform–methanol (2/2/1)**Xiao-Fang Li,* Ya-Qing Feng, Bo Gao and Nan Li**

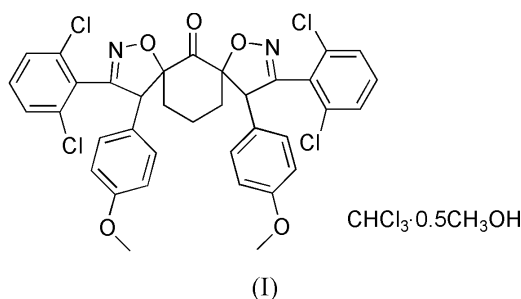
School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, People's Republic of China

Correspondence e-mail:
lxf7212@yahoo.com.cn**Key indicators**Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$
Disorder in solvent or counterion
 R factor = 0.068
 wR factor = 0.232
Data-to-parameter ratio = 14.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, $\text{C}_{36}\text{H}_{28}\text{Cl}_4\text{N}_2\text{O}_5 \cdot \text{CHCl}_3 \cdot 0.5\text{CH}_4\text{O}$, was synthesized by the intermolecular [3+2]-cycloaddition of 2,6-dichlorobenzonitrile oxide and 2,6-bis(4-methoxybenzylidene)cyclohexanone. There are three rings linked by two spiro-C atoms. The cyclohexane ring has a chair conformation and the two five-membered isoxazoline rings have envelope conformations.

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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



Compound (I) was synthesized by the intermolecular [3+2]-cycloaddition of 2,6-dichlorobenzonitrile oxide and 2,6-bis(4-methoxybenzylidene)cyclohexanone. The molecular structure of the main residue of (I) is illustrated in Fig. 1. There are two spiro junctions in the molecule, which contains two isoxazoline rings and a cyclohexane ring. The two isoxazoline rings adopt envelope conformations, while the cyclohexane ring has a chair conformation. The structure of 4,11-bis(4-chlorophenyl)-3,10-bis(2,6-dichlorophenyl)-1,8-dioxo-2,9-diazadispiro[4.1.4.3]tetradeca-2,9-dien-6-one 0.75-hydrate has been reported previously (Li *et al.*, 2003).

Experimental

A mixture of 2,6-dichlorobenzonitrile oxide (3 mmol) and 2,6-bis(4-methoxybenzylidene)cyclohexanone (1.5 mmol) in dry benzene (30 ml) was heated under reflux for 36 h. After evaporation of the solvent, the residue was separated by column chromatography (silica

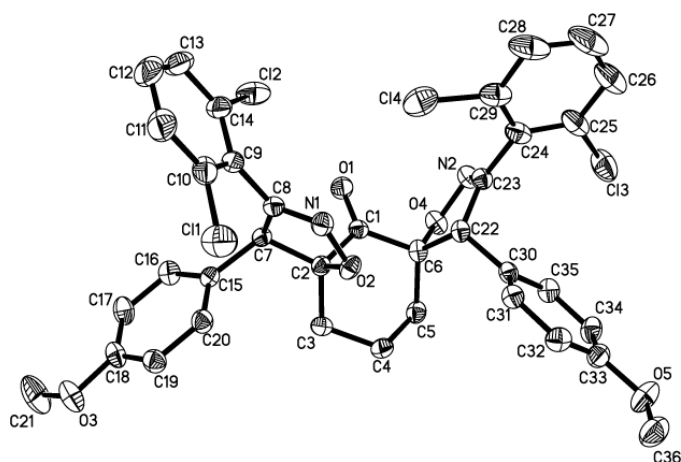


Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids. H atoms have been omitted for clarity.

gel, petroleum ether–ethyl acetate, 5:1) to give the title compound, (I) (m.p. 521–522 K). IR (KBr, cm^{-1}): 1734 (C=O), 1610, 1581 (C=N, C=C); ^1H NMR (CDCl_3 , p.p.m.): 1.26 (4H, *m*), 1.61 (2H, *m*), 3.73 (6H, *s*), 6.18 (2H, *s*), 6.81–7.35 (14H, *m*). Compound (I) (20 mg) was dissolved in a mixture of chloroform and methanol (15 ml), and the resulting solution was kept at room temperature for 10 d, giving colorless single crystals of (I) by evaporation.

Crystal data

$\text{C}_{36}\text{H}_{28}\text{Cl}_4\text{N}_2\text{O}_5 \cdot \text{CHCl}_3 \cdot 0.5\text{CH}_4\text{O}$
 $M_r = 845.79$
 Triclinic, $P\bar{1}$
 $a = 11.293$ (3) Å
 $b = 12.955$ (4) Å
 $c = 15.027$ (4) Å
 $\alpha = 114.946$ (4)°
 $\beta = 96.953$ (5)°
 $\gamma = 96.393$ (5)°
 $V = 1946.2$ (10) Å³

$Z = 2$
 $D_x = 1.443$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 956 reflections
 $\theta = 3.5$ – 23.5 °
 $\mu = 0.56$ mm⁻¹
 $T = 293$ (2) K
 Block, colorless
 $0.38 \times 0.36 \times 0.28$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	6824 independent reflections
φ and ω scans	4415 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$R_{\text{int}} = 0.019$
$T_{\text{min}} = 0.732$, $T_{\text{max}} = 0.856$	$\theta_{\text{max}} = 25.0^\circ$
10 073 measured reflections	$h = -13 \rightarrow 13$
	$k = -13 \rightarrow 15$
	$l = -17 \rightarrow 10$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.068$	$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$
$wR(F^2) = 0.232$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{\text{max}} < 0.001$
6824 reflections	$\Delta\rho_{\text{max}} = 0.64 \text{ e \AA}^{-3}$
480 parameters	$\Delta\rho_{\text{min}} = -0.77 \text{ e \AA}^{-3}$

The positions of the C and O atoms of the methanol molecule were found in a difference Fourier map and refined with occupancies fixed at 0.5. H atoms were positioned geometrically with C–H = 0.93–0.98 Å and refined in a riding model 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*, *SAINT* 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.
- Li, X.-F., Feng, Y.-Q., Zhuang, J.-P. & Hu, X.-F. (2003). *Acta Cryst.* **E59**, o247–o249.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.