

# 3'''-(2,6-Dichlorophenyl)-1'-methyl-4',4'''-diphenyl-4''',5'''-dihydro-indole-3-spiro-2'-pyrrolidine-3'-spiro-1''-cyclopentane-3''-spiro-5'''-[1,2]oxazole-2(3H),2''-dione

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## Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$

$R$  factor = 0.061

$wR$  factor = 0.126

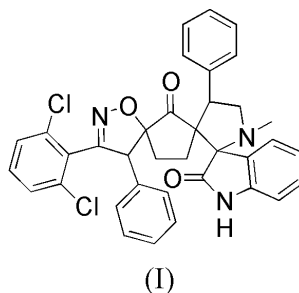
Data-to-parameter ratio = 15.3

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

The title compound,  $\text{C}_{36}\text{H}_{29}\text{Cl}_2\text{N}_3\text{O}_3$ , contains a planar 2-oxindole ring, an envelope pyrrolidine ring, a planar isoxazoline ring and a twist-shaped cyclopentane ring. There are three spiro junctions in the molecule. The molecules form dimers, joined by two  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds.

## 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 title compound, (I), was synthesized by a double dipolar cycloaddition. Azomethine ylide, derived from isatin and sarcosine by a decarboxylative route, was first reacted with 2,5-dibenzylidenecyclopentanone. The resulting cycloaddition product was then reacted with 2,6-dichlorobenzonitrile oxide to give (I).



The molecular structure of (I) (Fig. 1) revealed the presence of a ring system with spiro junctions at atoms C2, C5 and C20. It contains of a planar 2-oxindole ring, an envelope pyrrolidine ring, a planar isoxazoline ring and a twist-shaped cyclopentane ring. The molecules are arranged as dimers, connected by two  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds (Fig. 2), with  $\text{N}2\cdots\text{O}3^i = 2.833(2)\text{ \AA}$  and  $\text{N}-\text{H}\cdots\text{O} = 159.41(3)^\circ$  [symmetry code: (i)  $2-x, 1-y, 1-z$ ]. The structure of 1-methylspiro[2.3']-oxindolespiro[3.2'']-5',6''-dihydroimidazo[2'',1''-b]thiazol-3''-one-4-(2-benzo[1,3]dioxol-5-yl)pyrrolidine has been determined previously (Li *et al.*, 2003). That molecule also forms dimers in the solid state, but *via*  $\text{N}-\text{H}\cdots\text{N}$  hydrogen bonds.

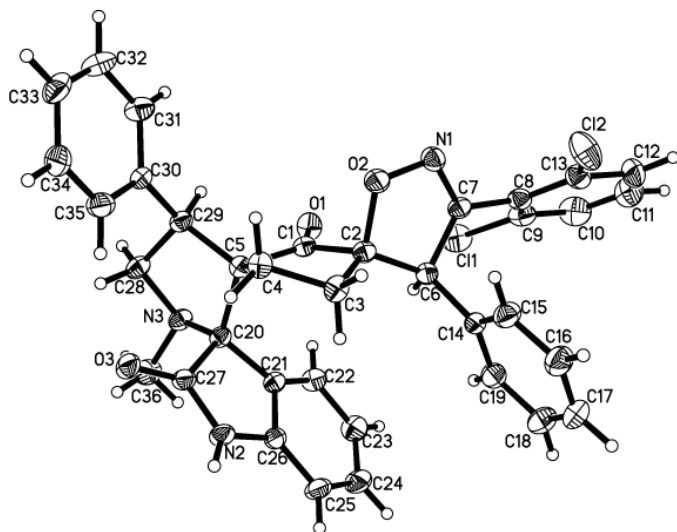
## Experimental

A mixture of 2,5-dibenzylidenecyclopentanone (2 mmol), isatin (2 mmol) and sarcosine (2 mmol) was refluxed in methanol (80 ml) until the starting material had disappeared, as evidenced by thin-layer chromatography. When the reaction was complete, the solvent was removed *in vacuo* and the residue separated by column

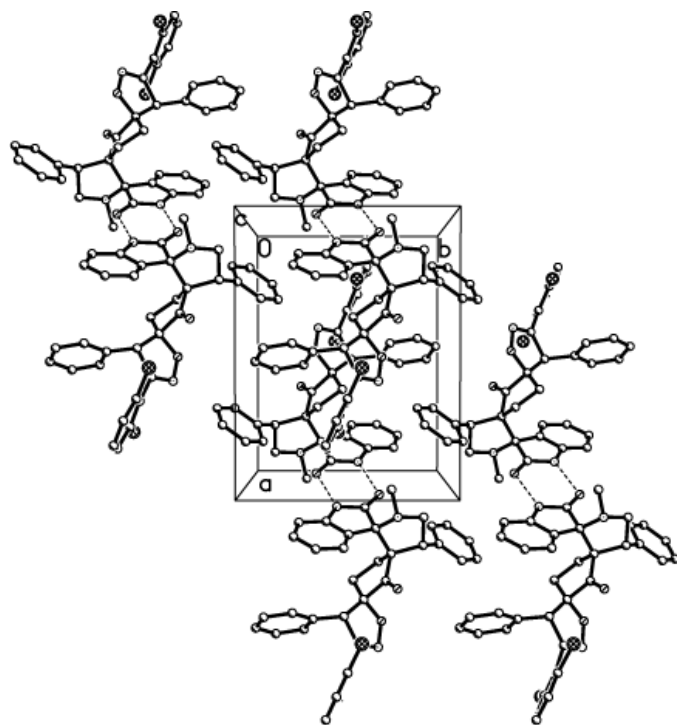
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**Figure 1**  
The molecular structure of (I), drawn with 30% probability ellipsoids.



**Figure 2**  
The crystal structure of (I), viewed along the *a* axis. Hydrogen bonds are indicated by dashed lines.

chromatography (silica gel, petroleum ether–ethyl acetate = 5:1), giving an intermediate compound. This was then reacted with 2,6-dichlorobenzonitrile oxide in refluxing dry benzene for 24 h. The

mixture was then cooled and filtered. Recrystallization from benzene–THF gave the title compound, (I) (m.p 524–525 K). IR (KBr): 3493 (N–H), 1697, 1680 (C=O)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , p.p.m.): 0.93 (1H, *m*), 1.38–1.73 (3H, *m*), 2.15 (3H, *s*), 3.54 (1H, *m*), 3.97 (1H, *m*), 4.20 (1H, *m*), 4.69 (1H, *s*), 6.53–7.50 (17H, *m*), 7.69 (1H, *br*). Colorless crystals, suitable for X-ray analysis, were obtained by slow evaporation of a solution of (I) in chloroform.

#### Crystal data

$\text{C}_{36}\text{H}_{29}\text{Cl}_2\text{N}_3\text{O}_3$   
 $M_r = 622.52$   
Monoclinic,  $P2_1/n$   
 $a = 12.622$  (4) Å  
 $b = 9.644$  (3) Å  
 $c = 24.576$  (9) Å  
 $\beta = 92.958$  (6)°  
 $V = 2987.7$  (17) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.384$  Mg  $\text{m}^{-3}$   
Mo  $K\alpha$  radiation  
Cell parameters from 851 reflections  
 $\theta = 2.7$ – $23.8$ °  
 $\mu = 0.26$   $\text{mm}^{-1}$   
 $T = 293$  (2) K  
Plate, colorless  
 $0.20 \times 0.16 \times 0.10$  mm

#### Data collection

Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
Absorption correction: multi-scan (SADABS; Bruker, 1997)  
 $T_{\min} = 0.873$ ,  $T_{\max} = 0.974$   
16632 measured reflections

6082 independent reflections  
3405 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.059$   
 $\theta_{\max} = 26.4$ °  
 $h = -15 \rightarrow 12$   
 $k = -11 \rightarrow 12$   
 $l = -30 \rightarrow 22$

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.061$   
 $wR(F^2) = 0.126$   
 $S = 1.03$   
6082 reflections  
398 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.34$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.27$  e Å<sup>-3</sup>

H atoms were positioned geometrically, with C–H = 0.93–0.98 Å, and refined as riding with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier atom})$ .

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.

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