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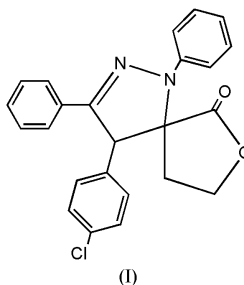
Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.054
 wR factor = 0.131
Data-to-parameter ratio = 15.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.4-(4-Chlorophenyl)-1,3-diphenyl-7-oxa-
1,2-diazaspiro[4.4]non-2-en-6-one

The title compound, $\text{C}_{24}\text{H}_{19}\text{ClN}_2\text{O}_2$, contains a spiro moiety, consisting of a pyrazole ring and a dihydrofuran ring. The pyrazole and dihydrofuran rings adopt envelope conformations. Inversion-related molecules exist as dimers in the crystalline state.

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, 4-(4-chlorophenyl)-1,3-diphenyl-7-oxa-1,2-diazaspiro[4.4]non-2-en-6-one, (I), is reported.



The structure of (I) contains a spiro moiety, consisting of a pyrazole ring and a dihydrofuran ring (Fig. 1). The pyrazole ring adopts an envelope conformation, with the spiro-atom C2 deviating from the N1/N2/C6/C5 plane by 0.412 (4) Å. The dihedral angle between the C5/C2/N1 and N1/N2/C6/C5 mean planes is 25.0 (1)°. The dihydrofuran ring also adopts an envelope conformation, with atom C3 lying 0.343 (5) Å from the C2/C1/O2/C4 plane. The dihedral angle between the C2/C3/C4 and C2/C1/O2/C4 planes is 21.6 (3)°. Phenyl rings *A* (C7–C12) and *C* (C19–C24) are nearly coplanar with the N1/N2/C6/C5 plane (r.m.s. deviation = 0.041 Å). The other phenyl ring *B* (C13–C18) forms a dihedral angle of 68.31 (8)° with the N1/N2/C6/C5 plane. Inversion-related molecules are linked by C11–H11...O1(1 - *x*, 1 - *y*, 1 - *z*) interactions to form dimers in the crystalline state.

Experimental

To a solution of 3-(4-chlorobenzylidene)dihydrofuran-2-one (10 mmol) and *N*-phenylbenzhydrazidoyl chloride (10 mmol) in dry benzene (30 ml) was added triethylamine (10 mmol), and the reaction mixture was stirred under reflux until the disappearance of the starting material. After the reaction was complete, the solution was filtered to remove triethylamine hydrochloride. After evaporation of

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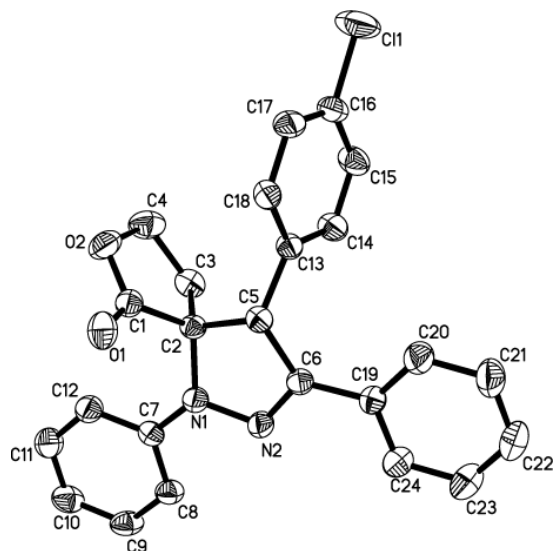


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. For clarity, H atoms have been omitted.

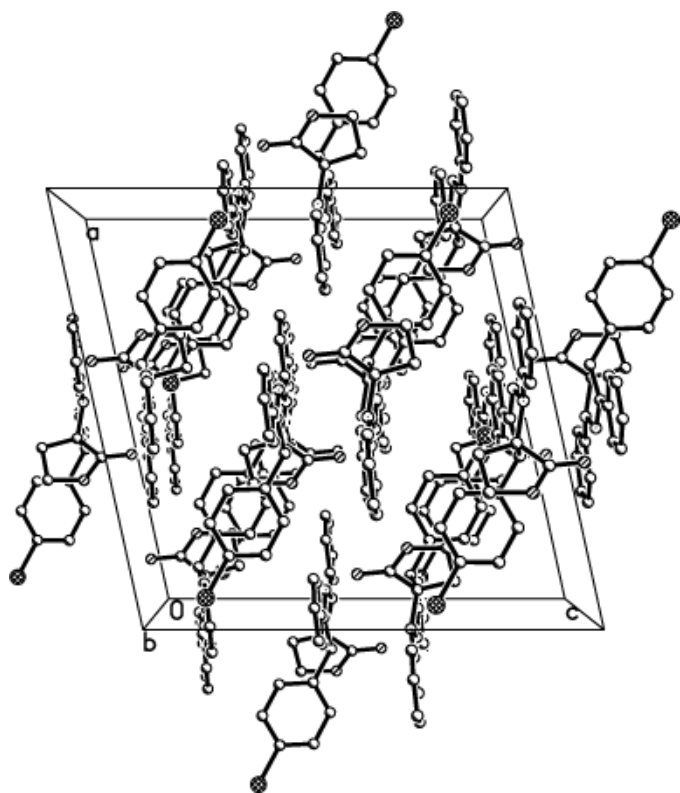


Figure 2
The crystal structure of (I), viewed along the *b* axis

the solvent of the filtrate, the residue was separated by column chromatography (silica gel, petroleum ether/ethyl acetate, 10:1) to give the title compound, (I). M.p. 439–440 K; IR (KBr): 1775 (C=O), 1595 (C=N, C=C) cm^{-1} ; ^1H NMR (CDCl_3 , p.p.m.): 1.95–2.04 (1H, *m*), 2.53–2.63 (1H, *m*), 3.61–3.70 (1H, *m*), 4.11–4.19 (1H, *m*), 5.15 (1H, *s*), 7.03–7.53 (14H, *m*); 20 mg of (I) was dissolved in 15 ml chloroform and the solution was kept at room temperature for 15 d to give colorless single crystals of (I) by natural evaporation.

Crystal data

$\text{C}_{24}\text{H}_{19}\text{ClN}_2\text{O}_2$
 $M_r = 402.86$
Monoclinic, $C2/c$
 $a = 16.013$ (6) Å
 $b = 15.627$ (6) Å
 $c = 16.317$ (6) Å
 $\beta = 102.325$ (7)°
 $V = 3989$ (3) Å³
 $Z = 8$

$D_x = 1.342$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 907 reflections
 $\theta = 2.4$ – 22.8°
 $\mu = 0.22$ mm⁻¹
 $T = 293$ (2) K
Block, colorless
0.30 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.940$, $T_{\max} = 0.958$
9301 measured reflections

4039 independent reflections
2154 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.039$
 $\theta_{\max} = 26.4^\circ$
 $h = -19 \rightarrow 7$
 $k = -19 \rightarrow 19$
 $l = -18 \rightarrow 20$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.131$
 $S = 1.00$
4039 reflections
262 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.32$ e Å⁻³
 $\Delta\rho_{\min} = -0.41$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

N1–N2	1.382 (3)	O2–C4	1.453 (3)
N1–C7	1.410 (3)	C1–C2	1.526 (3)
N1–C2	1.466 (3)	C2–C3	1.530 (3)
N2–C6	1.293 (3)	C2–C5	1.566 (3)
O1–C1	1.196 (3)	C3–C4	1.510 (4)
O2–C1	1.332 (3)		
N2–N1–C7	118.04 (19)	C7–N1–C2	126.1 (2)
N2–N1–C2	110.80 (18)	C6–N2–N1	109.99 (19)
N2–N1–C7–C12	−174.5 (2)	N2–C6–C19–C20	−173.1 (3)

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
C8–H8...N2	0.93	2.43	2.758 (4)	101
C11–H11...O1 ⁱ	0.93	2.51	3.369 (4)	154

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

The H atoms were geometrically positioned and were treated as riding on the parent C atoms, with C–H distances of 0.93, 0.97 or 0.98 Å, and $U_{\text{iso}}(\text{H}) = 1.2$ – $1.5U_{\text{eq}}(\text{C})$.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Bruker, 1997); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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