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Spiro[3-(4-chlorophenyl)-4-(4-methylphenyl)-4,5-dihydroisoxazole-5,3′-flavan-4′-one]

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.047
wR factor = 0.148
Data-to-parameter ratio = 15.4

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

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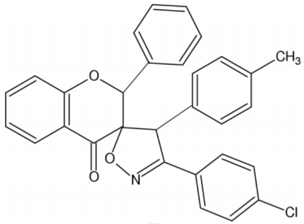
The pyran ring of the flavanone moiety in the title compound, $C_{30}H_{22}ClNO_3$, is puckered due to the saturation of a bond and this causes the ring to adopt a sofa conformation. The spiroisoxazoline ring adopts an envelope conformation. The phenyl rings on the isoxazoline ring are perpendicular to each other. The structure is stabilized by intermolecular C—H···O and C—H···N hydrogen bonds.

Comment

Flavanones are widely distributed and they form a part of our human diet, owing to their abundance in edible plants. The different properties and activities possessed by these compounds are discussed in our previous article (Jeyabharathi *et al.*, 2003). To understand the structure and conformation, a crystallographic study of the title compound, (I), was undertaken.



(I)

The geometry of the flavanone moiety in (I) agrees with reported values (Tomlin & Cantrell, 1990). As shown in Fig. 1, the pyran ring is planar, with the fused phenyl ring making a dihedral angle of 3.9 (1)°, larger than the reported value (Kendi & Ozbey, 1995). This may be due to the substitution at C5. The mean plane of the benzopyran ring is perpendicular to the isoxazoline ring plane. A study of torsion angles and asymmetry parameters (Cremer & Pople, 1975) reveals that the pyran ring adopts a slightly distorted sofa conformation [$q_2 = 0.343$ (2) Å, $q_3 = -0.263$ (2) Å, $Q_T = 0.432$ (2) Å and $\varphi_2 = -107.3$ (4)°]. For the isoxazoline ring, $q_2 = 0.146$ (2) Å and $\varphi_2 = -41.3$ (9)°, which confirms its envelope conformation. Apart from van der Waals interactions, the packing of the molecules in the crystal structure is stabilized by intermolecular C—H···O, C—H···N and C—H···π interactions. Thus C19—H19 points towards the centroid of the C22–C27($x - \frac{1}{2}$, $-\frac{1}{2} - y$, $z - \frac{3}{2}$) phenyl ring, *Cg*(1), suggesting a C—H···π intermolecular interaction (Table 2). The geometry of these interactions is comparable with others reported in the literature (Abdul Ajees *et al.*, 2001; Gallagher *et al.*, 2000; Kooijman *et al.*, 2000).

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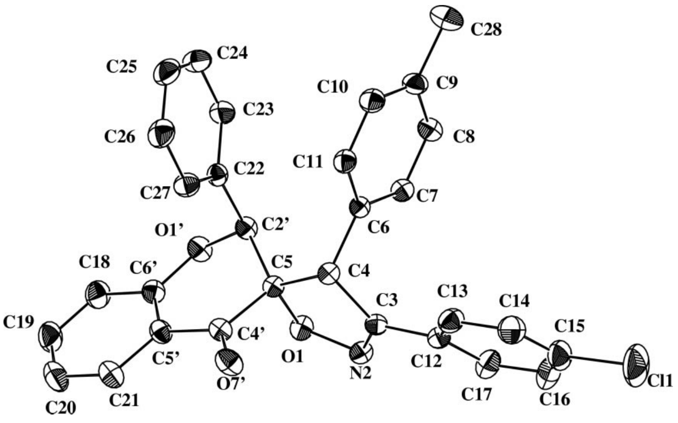

Figure 1
A view of the molecular structure of (I), showing ellipsoids at the 40% probability level. H atoms have been omitted.

Experimental

To a stirred solution of 3-*p*-methylbenzylidene-4-flavanone (3 mmol) and *N*-(*p*-chlorobenzhydroxyiminoyl chloride (3 mmol) in dry $CHCl_3$ (5 ml), 3.3 mmol of triethylamine was added. The reaction was monitored by TLC. After completion of the reaction, water was added to remove triethylamine hydrochloride and the resulting solution extracted with $CHCl_3$. The extracts were combined and dried using $MgSO_4$ and the product was purified by column chromatography (hexane/ethylacetate 9:1). The title compound, (I), was recrystallized from ethylacetate/hexane.

Crystal data

$C_{30}H_{22}ClNO_3$
$M_r = 479.94$
Monoclinic, $P2_1/n$
$a = 14.139\ (2)$ Å
$b = 10.939\ (2)$ Å
$c = 16.139\ (3)$ Å
$\beta = 99.809\ (13)°$
$V = 2459.8\ (8)$ Å^3
$Z = 4$

$D_x = 1.296$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 14–25°$
$\mu = 1.63$ mm^{-1}
$T = 293\ (2)$ K
Plate, colourless
$0.18 \times 0.17 \times 0.10$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.711$, $T_{max} = 0.849$
5076 measured reflections
4874 independent reflections
2453 reflections with $I > 2\sigma(I)$

$R_{int} = 0.028$
$\theta_{max} = 72.4°$
$h = 0 \rightarrow 17$
$k = 0 \rightarrow 13$
$l = -19 \rightarrow 19$
3 standard reflections every 200 reflections frequency: 120 min intensity decay: <0.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.148$
$S = 0.98$
4874 reflections
317 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0767P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.00123 (18)

Table 1
Selected geometric parameters (Å, °).

O1—N2	1.415 (2)	O1′—C2′	1.456 (3)
O1—C5	1.469 (3)	C4′—O7′	1.221 (3)
N2—C3	1.283 (3)	C4′—C5′	1.468 (3)
C3—C4	1.514 (3)	C5′—C6′	1.387 (4)
C5—C4′	1.522 (3)	C8—C9	1.391 (4)
C5—C2′	1.527 (3)		
O1′—C6′	1.366 (3)		
C5—O1—N2—C3	−8.5 (3)	N2—O1—C5—C4	14.4 (2)
O1—N2—C3—C4	−1.6 (3)	C3—C4—C5—O1	−13.9 (2)
N2—C3—C4—C5	10.1 (3)	C5—C2′—C22—C23	148.6 (2)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O7′	0.98	2.43	2.824 (3)	103
C7—H7···O1′^{i}	0.93	2.72	3.610 (3)	160
C2′—H2′···N2^{i}	0.98	2.66	3.482 (3)	142
C4—H4···O7′^{ii}	0.98	2.64	3.535 (3)	153
C19—H19···*Cg*(1^{iii})	0.93	2.75	3.597	153

Symmetry codes: (i) $2 - x, -y, -z$; (ii) $2 - x, 1 - y, -z$; (iii) $x - \frac{1}{2}, -\frac{1}{2} - y, z - \frac{3}{2}$.

After checking their presence in a difference map, all H atoms were fixed geometrically and allowed to ride on their parent C atoms and refined isotropically.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *SDP* (Frenz, 1978); data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *PARST*97 (Nardelli, 1995) and *PLATON* (Spek, 1998).

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