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Spiro[3,4-bis(4-chlorophenyl)-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 = 0.044
wR factor = 0.139
Data-to-parameter ratio = 14.4

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

The title compound, $C_{29}H_{19}Cl_2NO_3$, crystallizes with two molecules in the asymmetric unit. The pyran ring of the flavanone moiety is puckered due to the saturation of a bond and this causes the ring to adopt a sofa conformation. The spiroisoxazoline rings adopt envelope conformations. 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.

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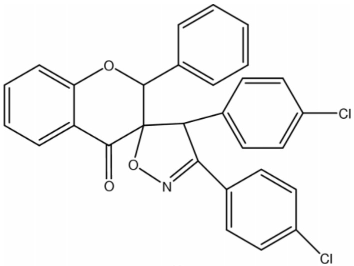
Comment

Flavanones are widely distributed and they form a part of our human diet, owing to their abundance in edible plants. The importance of these compounds is confirmed by their pharmacological activities. They possess therapeutic properties such as antiviral, antibiotic and antitumour (Cody, 1988). Spiroisoxazolines display interesting biological properties, such as herbicidal, plant-growth regulatory and antitumour activities (Howe & Shelton, 1990; Smietana *et al.*, 1999). To understand the structure and conformation, a crystallographic study of the title compound, (I), was undertaken.



(I)

The title compound, $C_{29}H_{19}Cl_2NO_3$, crystallizes with two molecules in the asymmetric unit. 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 3.5 (1)° in molecule *A* and 3.2 (1)° in molecule *B*; these are 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. Conformational parameters (Cremer & Pople, 1975) for the pyran ring are q_2 = 0.360 (3) Å, q_3 = −0.261 (3) Å Q_T = 0.445 (2) Å and φ_2 = −91.8 (4)° in molecule *A*; q_2 = 0.341 (3) Å, q_3 = −0.260 (2) Å, Q_T = 0.428 (2) Å and φ_2 = -95.6 (4)° in molecule *B*. For the isoxazoline ring, q_2 = 0.231 (2) Å and φ_2 = −41.0 (6)° in molecule A; q_2 = 0.211 (2) Å and φ_2 = −38.8 (6)° in molecule *B*, confirming their envelope conformations. Apart from van der Waals interactions, the packing of the molecules in the crystal structure is stabilized by intermolecular C—H···O and C—H···N interactions (Table 2).

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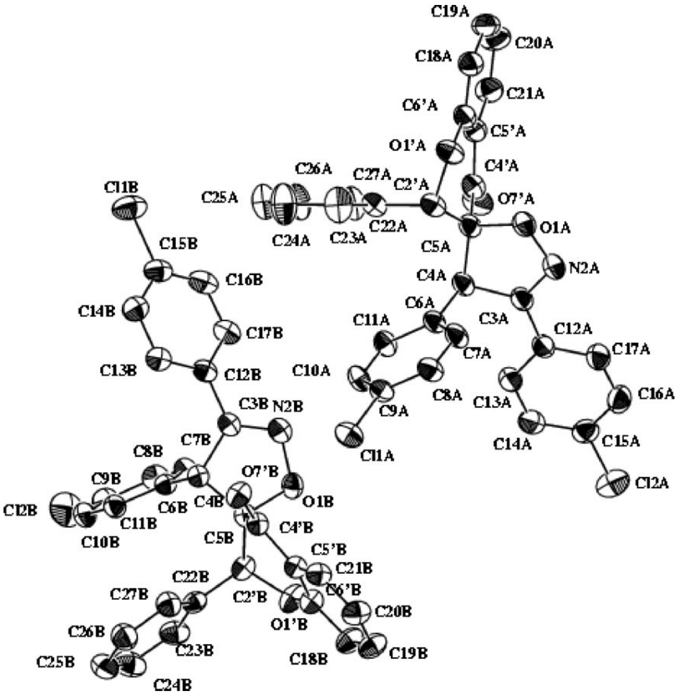

Figure 1
A view of the molecular structure of (I), showing ellipsoids at the 40% probability level. H atoms have been omitted.

Experimental

To a stirred solution of 3-*p*-chlorobenzylidene-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 ethyl acetate/hexane.

Crystal data

$C_{29}H_{19}Cl_2NO_3$
$M_r = 500.35$
Triclinic, $P\bar{1}$
$a = 11.2032\ (10)$ Å
$b = 12.349\ (2)$ Å
$c = 18.973\ (5)$ Å
$\alpha = 108.89\ (1)^\circ$
$\beta = 105.09\ (1)^\circ$
$\gamma = 91.66\ (1)^\circ$
$V = 2379.5\ (8)$ Å^3
$Z = 4$
$D_x = 1.397$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 14$–25°
$\mu = 2.72$ mm^{-1}
$T = 293\ (2)$ K
Plate, colourless
$0.30 \times 0.25 \times 0.10$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.638$, $T_{max} = 0.762$
9478 measured reflections
9032 independent reflections
6262 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 72.0^\circ$
$h = -13 \rightarrow 13$
$k = 0 \rightarrow 15$
$l = -23 \rightarrow 22$
3 standard reflections every 200 reflections frequency: 120 min intensity decay: <0.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.139$
$S = 1.02$
9032 reflections
629 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0735P)^2 + 0.5655P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0021 (2)

Table 1
Selected geometric parameters (Å, °).

Cl1*A*—C9*A*	1.735 (2)	Cl1*B*—C15*B*	1.730 (3)
Cl2*A*—C15*A*	1.735 (3)	Cl2*B*—C9*B*	1.741 (2)
O1′*A*—C6′*A*	1.354 (3)	O1′*B*—C6′*B*	1.359 (2)
O1′*A*—C2′*A*	1.456 (3)	O1′*B*—C2′*B*	1.448 (3)
C2′*A*—C5*A*	1.509 (3)	C2′*B*—C22*B*	1.506 (3)
C4′*A*—O7′*A*	1.218 (3)	C2′*B*—C5*B*	1.524 (3)
C4′*A*—C5*A*	1.531 (3)	C4′*B*—O7′*B*	1.214 (2)
C5′*A*—C6′*A*	1.394 (3)	C4′*B*—C5*B*	1.533 (3)
O1*A*—N2*A*	1.412 (3)	C5′*B*—C6′*B*	1.397 (3)
O1*A*—C5*A*	1.463 (3)	O1*B*—C5*B*	1.465 (3)
N2*A*—C3*A*	1.275 (3)	N2*B*—C3*B*	1.278 (3)
C3*A*—C4*A*	1.508 (3)	C3*B*—C4*B*	1.505 (3)
C4*A*—C5*A*	1.536 (3)	C4*B*—C5*B*	1.535 (3)
C5*A*—O1*A*—N2*A*—C3*A*	−13.4 (3)	C5*B*—O1*B*—N2*B*—C3*B*	−13.1 (2)
O1*A*—N2*A*—C3*A*—C4*A*	−2.3 (3)	O1*B*—N2*B*—C3*B*—C4*B*	−1.1 (2)
N2*A*—C3*A*—C4*A*—C5*A*	15.8 (3)	N2*B*—C3*B*—C4*B*—C5*B*	13.7 (2)
N2*A*—O1*A*—C5*A*—C4*A*	22.7 (2)	N2*B*—O1*B*—C5*B*—C4*B*	21.11 (19)
C3*A*—C4*A*—C5*A*—O1*A*	−21.9 (2)	C3*B*—C4*B*—C5*B*—O1*B*	−19.88 (18)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4*A*—H4*A*···O7′*A*	0.98	2.42	2.792 (3)	102
C10*A*—H10*A*···N2*B*	0.93	2.67	3.417 (3)	138
C27*A*—H27*A*···O7′*A*	0.93	2.75	3.595 (3)	151
C11*A*—H11*A*···O7′*A*^{i}	0.93	2.72	3.570 (3)	152
C4*A*—H4*A*···O7′*A*^{i}	0.98	2.68	3.619 (3)	161
C17*A*—H17*A*···O1*A*^{ii}	0.93	2.67	3.550 (3)	158
C4*B*—H4*B*···O7′*B*^{iii}	0.98	2.57	3.531 (3)	167

Symmetry codes: (i) $-x, 2-y, -z$; (ii) $-1-x, 2-y, -z$; (iii) $1-x, 2-y, 1-z$.

After checking their presence in a difference map, all H atoms were fixed geometrically and allowed to ride on the 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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References

Cody, V. (1988). *Plant Flavanoids in Biology and Medicine* II: *Biochemical, Cellular and Medicinal Properties*, edited by V. Cody, E. Middleton Jr, J. B. Horborne and A. Beretz, pp. 29–44. New York: Alan R. Liss Inc.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Frenz, B. A. (1978). *The Enraf–Nonius CAD*-4 *SDP – a Real–Time System for Concurrent X-ray Data Collection and Crystal Structure Solution*. *Computing in Crystallography*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld and G. C. Bassi, pp. 64–71. Delft University Press.
Harms, K. & Wocadlo, S. (1995). *XCAD*4. University of Marburg, Germany.
Howe, R. K. & Shelton, B. R. (1990). *J. Org. Chem.* **55**, 4603–4607.
Kendi, E. & Ozbey, S. (1995). *Acta Cryst.* C**51**, 1442–1444.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.
Smietana, M., Gouverneur, V. & Mioskowshi, C. (1999). *Tetrahedron Lett.* **40**, 1291–1294.
Spek, A. L. (1998). *PLATON*. Utrecht University, The Netherlands.
Tomlin, D. W. & Cantrell, J. S. (1990). *Acta Cryst.* C**46**, 519–521.
Zsolnai, L. (1997). *ZORTEP*. University of Heidelberg, Germany.