

3-(*p*-Chlorophenyl)-4-phenyl-4,5-dihydroisoxazole-5-spiro-2'-1',2',3',4'-tetrahydronaphthalen-1'-one

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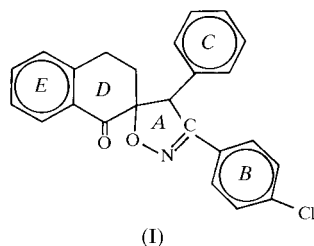
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In the title compound, C₂₄H₁₈ClNO₂, the phenyl ring and the tetralone moiety are approximately orthogonal to the isoxazoline ring. The isoxazoline ring adopts an envelope conformation, while the cyclohexenone ring of the tetralone moiety has an intermediate sofa/half-chair conformation. In this structure, one C—H···N intermolecular and two C—H···O intramolecular hydrogen bonds occur; the H···A distances are 2.60, and 2.35 and 2.57 Å, respectively. The molecules are held together by an intermolecular C—H···N hydrogen bond, forming a one-dimensional chain along the [100] direction.

Comment

Isoxazoline derivatives have been used in many natural-product syntheses and have also proved to be efficient precursors for many synthetic intermediates, including γ -amino alcohols, β -hydroxy ketones, *etc.* (Kozikowski, 1984; Kanemasa & Tsuge, 1990). Spiro-isoxazoline compounds display interesting biological properties, such as herbicidal,



plant-growth regulatory and antitumour activities (Howe & Shelton, 1990; De Amici *et al.*, 1990; Smietana *et al.*, 1999). We report here the structure of 3-(*p*-chlorophenyl)-4-phenyl-4,5-dihydroisoxazole-5-spiro-2'-1',2',3',4'-tetrahydronaphthalen-1'-one, (I).

The structure of (I) (Fig. 1) consists of an isoxazoline ring (A) connected to a *p*-chlorophenyl ring (B) at C2, a phenyl ring (C) at C3 and a tetralone moiety [D (cyclohexenone) and E (phenyl)] at C1. Rings A and B are approximately coplanar, forming a dihedral angle of 1.2 (1)°. Phenyl ring C and tetralone ring D are approximately orthogonal to ring A, forming dihedral angles of 77.5 (1) and 88.5 (1)°, respectively. There is a slight folding of rings D and E of the tetralone substituent, the rings forming a dihedral angle of 4.9 (1)° with one another.

The bond lengths O1—N1 and N1=C2, and the angles N1—O1—C1 and O1—N1—C2 in ring A are comparable with the corresponding values of two other structures (Mackay *et al.*, 1994; Pereira *et al.*, 1993). Also, bond lengths C11—C16 and C12=O2 are in good agreement with the values found in the literature (Allen *et al.*, 1987). Selected bond lengths and angles are given in Table 1.

The puckering (Cremer & Pople, 1975) and asymmetry parameters (Nardelli, 1983a) calculated for ring D are $Q_T = 0.450$ (4) Å, $\Delta C_s(C4) = 0.052$ (2)° and $\Delta C_2(C1-C4) = 0.060$ (2)°, and for ring A are $Q_T = 0.146$ (4) Å and $\Delta C_s(C1) = 0.003$ (2)°, which correspond to an intermediate sofa/half-chair conformation for ring D and an envelope conformation for ring A.

The N atom of ring A is involved in a C—H···N intermolecular hydrogen bond. The O atom of ring A and the carbonyl O atom of ring D are involved in two C—H···O short contacts (Table 2). The C3—H3···O2 and C5—

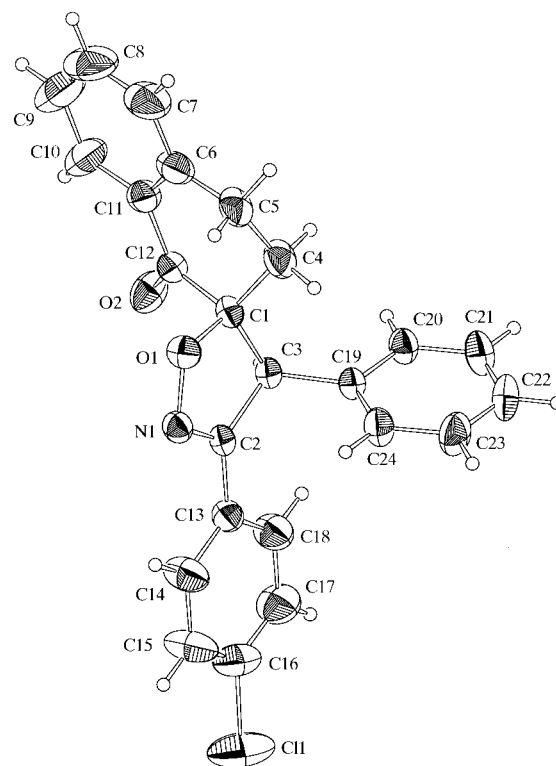


Figure 1
The molecular structure of the title compound (ZORTEP; Zsolnai, 1997) shown with 50% probability displacement ellipsoids. H atoms are displayed as small circles of arbitrary radii.

H5A...O1 intramolecular interactions are a result of the configurations at C3 and C1, respectively, while the intermolecular C22—H22...N1ⁱ interaction joins the molecules in chains running along the [100] direction [symmetry code: (i) 1 + x, y, z].

Experimental

To a well stirred solution of 2-arylidine-1-tetralone (3 mmol) in CHCl₃ (10 ml), *N-p*-chlorobenzhydroxyiminoyl chloride was added (3 mmol), followed by 3.3 mmol of triethylamine. The reaction was monitored *via* thin-layer chromatography until the starting material had disappeared, and was then quenched with water, extracted with CHCl₃, dried with anhydrous MgSO₄, column chromatographed using hexane/ethyl acetate (4:1) and recrystallized from hexane/ethyl acetate (3:1).

Crystal data

C ₂₄ H ₁₈ ClNO ₂	Z = 2
M _r = 387.84	D _x = 1.299 Mg m ⁻³
Triclinic, P $\bar{1}$	Mo K α radiation
a = 9.2559 (8) Å	Cell parameters from 2150 reflections
b = 10.9527 (9) Å	θ = 2.9–25.0°
c = 11.6466 (10) Å	μ = 0.21 mm ⁻¹
α = 62.867 (2)°	T = 293 (2) K
β = 71.010 (2)°	Rectangular, colourless
γ = 78.653 (2)°	0.42 × 0.16 × 0.14 mm
V = 991.93 (15) Å ³	

Data collection

Siemens SMART CCD area-detector	R _{int} = 0.027
ω scans	θ_{\max} = 25.0°
5316 measured reflections	h = -10 → 10
3387 independent reflections	k = -11 → 13
2158 reflections with I > 2 σ (I)	l = -10 → 13
	Intensity decay: 1%

Refinement

Refinement on F ²	w = 1/[$\sigma^2(F_o^2) + (0.0861P)^2 + 0.4816P$]
R[F ² > 2 σ (F ²)] = 0.065	where P = (F _o ² + 2F _c ²)/3
wR(F ²) = 0.191	(Δ/σ) _{max} < 0.001
S = 1.03	$\Delta\rho_{\max}$ = 0.38 e Å ⁻³
3387 reflections	$\Delta\rho_{\min}$ = -0.46 e Å ⁻³
254 parameters	Extinction correction: SHELXL97
H-atom parameters constrained	Extinction coefficient: 0.020 (5)

The range of constrained C—H distances was 0.93–0.98 Å.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ZORTEP (Zsolnai, 1997); software used to prepare material for publication: SHELXL97, PARST (Nardelli, 1983b, 1995) and PLATON (Spek, 1990).

Table 1

Selected geometric parameters (Å, °).

Cl1—C16	1.741 (4)	O2—C12	1.218 (4)
O1—N1	1.407 (3)	N1—C2	1.281 (4)
O1—C1	1.459 (4)		
N1—O1—C1	108.8 (2)	N1—C2—C13	119.7 (3)
C2—N1—O1	110.0 (2)	N1—C2—C3	114.0 (3)
O1—C1—C4	107.3 (3)	O2—C12—C11	122.4 (3)
O1—C1—C3	105.2 (2)	O2—C12—C1	120.1 (3)
O1—C1—C12	103.5 (2)		

Table 2

Hydrogen-bonding and short-contact geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
C5—H5A...O1	0.97	2.57	2.831 (6)	95
C3—H3...O2	0.98	2.35	2.777 (4)	105
C22—H22...N1 ⁱ	0.93	2.60	3.531 (5)	175

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

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1498). Services for accessing these data are described at the back of the journal.

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