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3-(2-Chlorophenyl)-*N*-(2-cyano-4-methyl-2-pentyl)-5-methylisoxazole-4-carboxamide

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The title compound, $C_{18}H_{20}ClN_3O_2$, is a potent new herbicide. X-ray analysis reveals that the benzene ring is twisted away from the isoxazole ring by 67.7 (1)°. The crystal packing is stabilized by intermolecular N—H···N and C—H···O hydrogen bonds.

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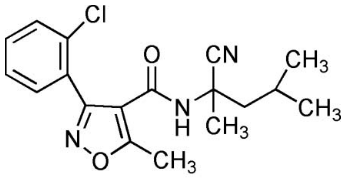
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.150
Data-to-parameter ratio = 17.1

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

Comment

Isoxazole derivatives represent one of the most active classes of compounds, possessing a wide spectrum of biological activity. They are widely used in agrochemicals and pharmaceuticals (He *et al.*, 2000). Similarly, aminoacetonitrile derivatives exhibit various biological activities, *e.g.* insecticidal (Andoh *et al.*, 1999; Ducray, Goebel & Bouvier, 2003; Ducray, Goebel & Fruechtel, 2003), fungicidal (Walker & Baker 1979; Kim *et al.*, 2004) and herbicidal (Berliner & Richter, 1971). In view of these properties and in a continuation of our interest in the chemistry of isoxazoles, we have attempted to synthesize a series of 3-(2-chlorophenyl)-5-methylisoxazole-4-carboxamide derivatives, some of which have comparatively high herbicidal activity. The crystal structure determination of the title compound, (I), was undertaken to investigate the relationship between structure and herbicidal activity.



(I)

The molecular structure of (I) is shown in Fig. 1. The X-ray analysis reveals that the benzene ring is twisted away from the isoxazole ring by 67.7 (1)°. The dihedral angle between the isoxazole ring and the carboxamide N2/O2/C11/C12 plane is 30.2 (1)°. The C11═O2 distance of 1.220 (2) Å is comparable to the mean value of the C═O distance [1.231 (12) Å; Zhong *et al.*, 2004] in amides. In the crystal structure, adjacent molecules are linked by weak N—H···N and C—H···O hydrogen bonds (Table 2).

Experimental

Compound (I) was prepared according to the reported precedure of Liu *et al.* (2004). Single crystals suitable for X-ray diffraction were obtained by recrystallization from a mixture of ethyl acetate and petroleum ether (1:5 *v*/*v*).

Crystal data

$C_{18}H_{20}ClN_3O_2$
$M_r = 345.82$
Monoclinic, $P2_1/n$
$a = 12.340$ (2) Å
$b = 8.7775$ (14) Å
$c = 17.678$ (3) Å
$\beta = 104.861$ (3)°
$V = 1850.7$ (5) Å^3
$Z = 4$
$D_x = 1.241$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2447 reflections
$\theta = 2.6$–22.7°
$\mu = 0.22$ mm^{-1}
$T = 294$ (2) K
Prism, colorless
0.26 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.943$, $T_{\max} = 0.957$
10142 measured reflections
3784 independent reflections
2123 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.032$
$\theta_{\max} = 26.5°$
$h = -15 \rightarrow 15$
$k = -8 \rightarrow 10$
$l = -19 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.150$
$S = 0.94$
3784 reflections
221 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 0.7394P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.014$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C9	1.344 (3)	N1—C1	1.311 (3)
O1—N1	1.407 (3)	N2—C11	1.348 (3)
O2—C11	1.220 (2)	N2—C12	1.463 (3)
O2—C11—C8	122.2 (2)	N2—C11—C8	114.98 (18)
C8—C1—C2—C7	67.4 (4)	C1—C8—C11—N2	29.4 (4)
N1—C1—C2—C3	67.2 (3)	C11—N2—C12—C15	174.5 (2)
C12—N2—C11—O2	−4.8 (4)	C12—C15—C16—C17	170.8 (2)
C9—C8—C11—O2	29.9 (4)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···N1$^{\rm i}$	0.86	2.63	3.187 (3)	124
C10—H10B···O2	0.96	2.53	3.156 (4)	123
C10—H10C···O2$^{\rm ii}$	0.96	2.59	3.407 (4)	143
C13—H13C···O2	0.96	2.54	3.110 (3)	118
C16—H16A···N2	0.98	2.60	2.963 (3)	102

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

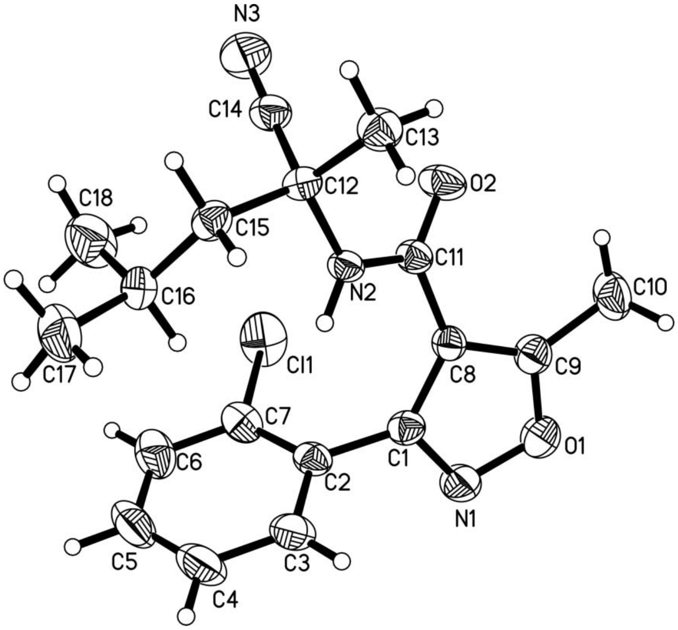


Figure 1
The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at 40% probability level.

The H atoms were placed in calculated positions and allowed to ride on their parent atoms, with an N—H distance of 0.86 Å and C—H distances in the range 0.93–0.98 Å, and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(C/N) and $1.5U_{\rm eq}$(methyl C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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