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N-(1-Cyano-1,2-dimethylpropyl)-5-methylisoxazole-4-carboxamide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.056
wR factor = 0.144
Data-to-parameter ratio = 18.0

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

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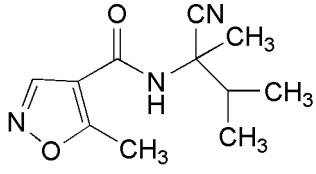
The title compound, $C_{11}H_{15}N_3O_2$, is a potent new herbicide containing a planar 5-methylisoxazole-4-carboxamide ring system. N—H···N hydrogen bonds link symmetry-related molecules into chains along the *c* axis.

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). Likewise, aminoacetonitrile derivatives exhibit various biological activities, *e.g.* insecticidal (Andoh *et al.*, 1999; Ducray *et al.*, 2003; Ducray *et al.*, 2003), fungicidal (Walker & Baker, 1979; Kim *et al.*, 2004) and herbicidal (Berliner & Richter, 1971). In view of these facts and in continuation of our interest in the chemistry of isoxazoles, we attempted to synthesize a series of 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. To the best of our knowledge, this is the first reported crystal structure determination of a molecule with a 5-methylisoxazole-4-carboxamide ring system.



(I)

The molecular structure of (I) is shown in Fig. 1. The X-ray analysis reveals that the isoxazole ring is planar, with an r.m.s deviation of 0.001 Å. The carboxamide moiety is coplanar with the isoxazole ring [dihedral angle 3.7 (3)°]. The C5═O2 distance of 1.236 (2) Å is comparable to the mean value of the C═O distance [1.231 (12) Å] in amides. In the crystal structure, glide-related molecules are linked by weak N—H···N hydrogen-bonding interactions, forming chains along the *c* axis (Fig. 2, Table 2).

Experimental

The title compound was prepared according to the reported procedure of Liu *et al.* (2004). Colourless single crystals suitable for X-ray diffraction analysis were obtained by recrystallization from a mixture of ethyl acetate and petroleum ether (1:3).

Crystal data

$C_{11}H_{15}N_3O_2$
$M_r = 221.26$
Monoclinic, $P2_1/c$
$a = 10.738\ (8)$ Å
$b = 10.608\ (8)$ Å
$c = 11.623\ (8)$ Å
$\beta = 111.442\ (12)^\circ$
$V = 1232.3\ (16)$ Å^3
$Z = 4$
$D_x = 1.193$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 725 reflections
$\theta = 3.8$–24.5°
$\mu = 0.08$ mm^{-1}
$T = 293\ (2)$ K
Prism, colourless
0.30 × 0.26 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.960$, $T_{max} = 0.982$
7272 measured reflections
2748 independent reflections
1795 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 27.4^\circ$
$h = -13 \rightarrow 8$
$k = -13 \rightarrow 12$
$l = -12 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.144$
$S = 1.02$
2748 reflections
153 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0609P)^2 + 0.3201P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.357 (3)	C2—C3	1.366 (3)
O1—N1	1.424 (3)	C3—C4	1.426 (3)
O2—C5	1.236 (2)	C3—C5	1.483 (3)
N1—C4	1.304 (3)	C6—C7	1.503 (3)
N2—C5	1.364 (2)	C6—C8	1.553 (3)
N2—C6	1.472 (2)	C6—C9	1.556 (3)
N3—C7	1.146 (2)	C9—C10	1.534 (3)
C1—C2	1.490 (3)	C9—C11	1.540 (3)
C2—O1—N1	109.59 (15)	C2—C3—C5	125.09 (18)
C4—N1—O1	104.27 (17)	C4—C3—C5	130.80 (17)
C5—N2—C6	123.06 (16)	N1—C4—C3	113.1 (2)
O1—C2—C3	108.96 (18)	O2—C5—N2	121.81 (18)
O1—C2—C1	116.50 (18)	O2—C5—C3	122.09 (17)
C3—C2—C1	134.54 (19)	N2—C5—C3	116.07 (16)
C2—C3—C4	104.06 (18)	N3—C7—C6	173.1 (2)
C5—C3—C4—N1	177.8 (2)	C5—N2—C6—C7	46.0 (3)
C6—N2—C5—O2	−3.3 (3)	C5—N2—C6—C8	−74.0 (2)
C2—C3—C5—O2	1.5 (3)	C5—N2—C6—C9	163.10 (17)
C4—C3—C5—O2	−175.6 (2)	N2—C6—C9—C10	−53.4 (2)
C4—C3—C5—N2	2.6 (3)	C7—C6—C9—C11	−60.2 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···N3^{i}	0.85 (2)	2.33 (2)	3.169 (3)	168 (2)

Symmetry code: (i) $x, \frac{3}{2} - y, z - \frac{1}{2}$.

The amine H atom was located in a difference map and was refined isotropically [N—H = 0.85 (2) Å]. All other H atoms were placed in calculated positions (C—H = 0.93 or 0.96 Å) and included in the refinement using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$.

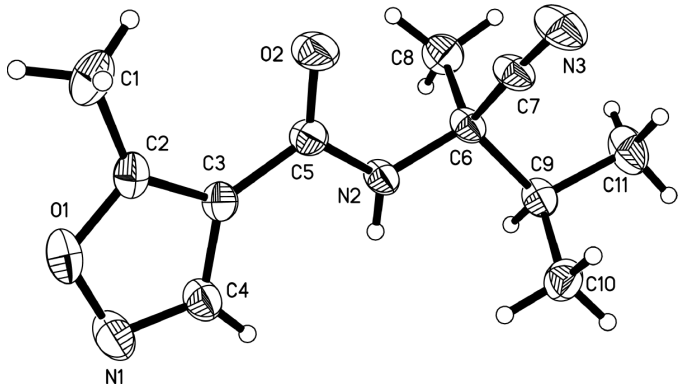


Figure 1
View of the title compound (I), with the atomic numbering. Displacement ellipsoids are drawn at the 40% probability level.

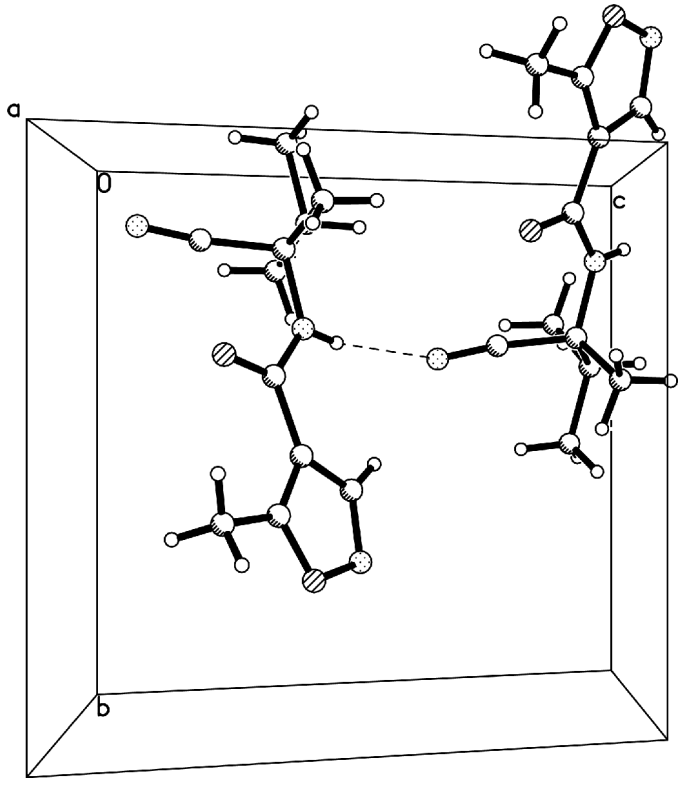


Figure 2
The N—H···N hydrogen bonds (shown as dashed lines) in (I), viewed along the *a* axis.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXL*97.

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