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3-(2-Chlorophenyl)-*N*-methylisoxazole-5-carboxamide

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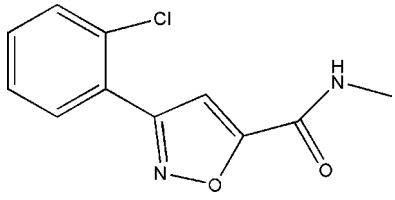
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.082; data-to-parameter ratio = 11.2.

In the title molecule, $C_{11}H_9ClN_2O_2$, the isoxazole and benzene rings make a dihedral angle of 36.80 (2)°. Intermolecular N—H···O and C—H···O hydrogen bonds link the molecules into zigzag chains running in the [101] direction. The crystal packing exhibits weak π–π stacking interactions [short distances of 3.675 (2) and 3.801 (3) Å between the centroids of the benzene and isoxazole rings (at $\frac{1}{2}-x$, $-y$, $-\frac{1}{2}+z$) and (at $\frac{1}{2}-x$, $-y$, $\frac{1}{2}+z$), respectively], which form stacks of molecules extending along the c axis.

Related literature

For the crystal structure of the related compound 3-(4-chlorophenyl)-*N*-methylisoxazole-5-carbaldehyde, see: Zhang *et al.* (2006). For details of the pharmacological properties of isoxazolyl carboxamide derivatives, see: Lee *et al.* (2006); Xin *et al.* (2005).



Experimental

Crystal data

$C_{11}H_9ClN_2O_2$
M_r = 236.65
Orthorhombic, *Fdd*2
a = 18.722 (4) Å
b = 31.454 (6) Å
c = 7.2137 (14) Å
V = 4248.1 (14) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 298 (2) K
0.51 × 0.40 × 0.39 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.844, T_{max} = 0.877
5233 measured reflections
1630 independent reflections
1563 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.082
S = 1.05
1630 reflections
146 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Absolute structure: Flack (1983), with 737 Friedel pairs
Flack parameter: 0.05 (7)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O2^{i}	0.86	2.14	2.983 (3)	165
C8—H8A···O2^{i}	0.93	2.33	3.173 (3)	151

Symmetry code: (i) $x+\frac{1}{4}, -y+\frac{1}{4}, z+\frac{1}{4}$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2248).

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supplementary materials

Acta Cryst. (2007). E**63**, o3074 [**doi:10.1107/S1600536807025895**]

3-(2-Chlorophenyl)-*N*-methylisoxazole-5-carboxamide

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Comment

The derivatives of isoxazolyl carboxamides have been reported to possess various chemical and biological activities (Lee *et al.*, 2006; Xin *et al.*, 2005). In connection with our study of the design and synthesis of new aryl-substituted isoxazole, we prepared 3-(2-chlorophenyl)-*N*-methylisoxazole-5-carboxamide, (I), by a convenient method from isoxazole-5-carboxyl acid. Here, we report the crystal structure of (I).

In (I) (Fig. 1), all bond lengths and angles are normal and in a good agreement with those reported recently for 3-(4-chlorophenyl)-*N*-methylisoxazole-5-carbaldehyde (Zhang *et al.*, 2006). Atoms C11/C12/N2/O2 lies in the isoxazole ring (C7/C8/C9/N1/O1) plane, and the deviations from the least-squares plane through the ring atoms are all smaller than 0.024 (3) Å. The dihedral angle between the plane of the isoxazole and benzene (C1/C2/C3/C4/C5/C6) rings is 36.80 (2)°. The relatively short distances between the centroids of benzene (*Cg*1) and isoxazole (*Cg*2) rings from the neighbouring molecules - $Cg1\cdots Cg2^{i}$ 3.675 (2) Å and $Cg2\cdots Cg1^{i}$ 3.801 (3) Å indicates a presence of weak π-π interactions, which form stacks of the molecules extended along the *c* axis. Intermolecular N—H···O and C—H···O hydrogen bonds link the molecules into zigzag chains running in the direction [101].

Experimental

A mixture of 3-(2-chlorophenyl)-isoxazole-5-carboxyl acid (8 mmol) and $SOCl_2$ (10 ml) was heated under reflux for 7 h. The excess $SOCl_2$ was removed on a water vacuum pump and the residue was distilled *in vacuo* to give carbonyl chlorides (over 85% yield). The product was dissolved in 20 ml of dry acetone, which was added to excessive 30% methylamine anhydrous solution below −5° C. After stirring vigorously at the same temperature for 1 h, the mixture was extracted with CH_2Cl_2 and the organic layer was washed with $NaHCO_3$ solution, H_3PO_4 solution, and finally with water. The solution was dried and evaporated at 308 K (15 mm H g) to yield pale powder, which was recrystallized from ethyl acetate to obtain the product (74% yield).

Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a solution of (I) in a hexane–dichloromethane mixture (1:1 *v*/*v*) at room temperature over a period of one week.

Refinement

All H atoms were geometrically positioned (N—H 0.86 Å, C—H 0.93–0.96 Å) and refined as riding with U_{iso}(H) = 1.2–1.5U_{eq}(parent atom).

Figures

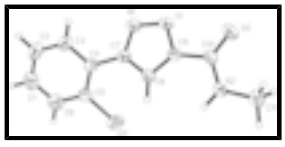

Fig. 1. The molecular structure of (I), with atom labels and 40% probability displacement ellipsoids for non-H atoms.

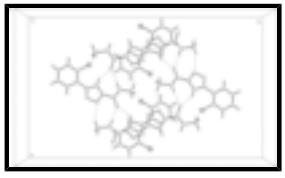

Fig. 2. The packing of (I), viewed down the *c* axis, showing the molecules connected by N—H···O and C—H···O hydrogen bonds (dashed lines).

3-(2-Chlorophenyl)-*N*-methylisoxazole-5-carboxamide

Crystal data

$C_{11}H_9ClN_2O_2$	F_{000} = 1952
M_r = 236.65	D_x = 1.480 Mg m^{-3}
Orthorhombic, *Fdd*2	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: F 2 -2d	Cell parameters from 622 reflections
a = 18.722 (4) Å	θ = 2.7–20.3º
b = 31.454 (6) Å	μ = 0.34 mm^{-1}
c = 7.2137 (14) Å	*T* = 298 (2) K
V = 4248.1 (14) Å^3	Block, colourless
Z = 16	0.51 × 0.40 × 0.39 mm

Data collection

Bruker SMART CCD area-detector diffractometer	1630 independent reflections
Radiation source: fine-focus sealed tube	1563 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.024
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	*h* = −15→22
T_{min} = 0.844, T_{max} = 0.877	*k* = −37→37
5233 measured reflections	*l* = −7→8

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.032	$w = 1/[\sigma^2(F_o^2) + (0.0452P)^2 + 3.3758P]$ where $P = (F_o^2 + 2F_c^2)/3$

$wR(F^2) = 0.082$	$(\Delta/\sigma)_{max} = 0.002$
$S = 1.05$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
1630 reflections	$\Delta\rho_{min} = -0.15$ e Å^{-3}
146 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), with 737 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.05 (7)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.40451 (3)	0.01932 (2)	0.63140 (14)	0.0677 (3)
O1	0.15158 (7)	0.05205 (5)	0.7033 (3)	0.0466 (4)
C4	0.28830 (11)	−0.02604 (7)	0.7458 (3)	0.0365 (5)
C10	0.18338 (12)	0.12278 (7)	0.7981 (3)	0.0397 (5)
O2	0.12321 (9)	0.13506 (6)	0.7591 (3)	0.0578 (5)
N2	0.23485 (9)	0.14753 (6)	0.8611 (3)	0.0445 (5)
H2A	0.2762	0.1366	0.8827	0.053*
N1	0.17918 (10)	0.01105 (6)	0.6847 (3)	0.0452 (5)
C7	0.24522 (12)	0.01304 (7)	0.7449 (3)	0.0349 (5)
C8	0.26279 (11)	0.05475 (7)	0.8031 (3)	0.0367 (5)
H8A	0.3061	0.0643	0.8506	0.044*
C9	0.20348 (11)	0.07743 (7)	0.7747 (3)	0.0362 (5)
C5	0.36072 (12)	−0.02668 (8)	0.7037 (4)	0.0428 (6)
C6	0.39989 (14)	−0.06374 (9)	0.7098 (4)	0.0579 (8)
H6A	0.4482	−0.0633	0.6804	0.070*
C3	0.25671 (14)	−0.06454 (8)	0.7907 (4)	0.0462 (6)
H3A	0.2081	−0.0653	0.8169	0.055*
C2	0.29559 (15)	−0.10164 (9)	0.7974 (4)	0.0570 (7)
H2B	0.2731	−0.1271	0.8278	0.068*
C11	0.22384 (14)	0.19281 (8)	0.8955 (5)	0.0608 (8)
H11A	0.2622	0.2036	0.9702	0.091*
H11B	0.1794	0.1969	0.9593	0.091*
H11C	0.2227	0.2078	0.7795	0.091*

C1	0.36764 (16)	−0.10126 (9)	0.7592 (5)	0.0611 (8)
H1B	0.3942	−0.1262	0.7669	0.073*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0399 (3)	0.0536 (4)	0.1097 (6)	−0.0067 (3)	0.0177 (4)	0.0027 (4)
O1	0.0300 (8)	0.0444 (9)	0.0654 (12)	−0.0012 (7)	−0.0097 (8)	0.0011 (8)
C4	0.0362 (11)	0.0388 (12)	0.0343 (13)	−0.0019 (9)	−0.0011 (9)	−0.0029 (10)
C10	0.0305 (11)	0.0451 (13)	0.0433 (13)	0.0060 (9)	−0.0012 (9)	0.0059 (11)
O2	0.0368 (9)	0.0509 (10)	0.0857 (14)	0.0104 (8)	−0.0167 (9)	−0.0006 (10)
N2	0.0298 (9)	0.0379 (10)	0.0659 (14)	0.0076 (7)	−0.0068 (10)	−0.0005 (10)
N1	0.0337 (10)	0.0418 (11)	0.0602 (14)	−0.0029 (8)	−0.0036 (9)	−0.0025 (10)
C7	0.0311 (11)	0.0394 (12)	0.0343 (12)	−0.0035 (9)	−0.0017 (8)	0.0009 (10)
C8	0.0284 (11)	0.0383 (12)	0.0433 (13)	0.0007 (9)	−0.0043 (9)	−0.0020 (10)
C9	0.0288 (10)	0.0409 (12)	0.0389 (12)	−0.0025 (9)	−0.0008 (9)	0.0036 (11)
C5	0.0357 (11)	0.0432 (13)	0.0495 (15)	−0.0019 (10)	0.0001 (10)	−0.0055 (11)
C6	0.0440 (14)	0.0522 (16)	0.078 (2)	0.0093 (11)	0.0085 (13)	−0.0110 (15)
C3	0.0475 (13)	0.0405 (12)	0.0507 (16)	−0.0059 (10)	0.0026 (11)	−0.0019 (11)
C2	0.0723 (18)	0.0367 (13)	0.0621 (18)	−0.0026 (12)	0.0087 (15)	0.0014 (12)
C11	0.0513 (14)	0.0430 (14)	0.088 (2)	0.0085 (11)	−0.0083 (15)	−0.0117 (16)
C1	0.0654 (17)	0.0452 (15)	0.073 (2)	0.0159 (13)	0.0044 (15)	−0.0035 (15)

Geometric parameters (Å, °)

Cl1—C5	1.743 (3)	C8—C9	1.336 (3)
O1—C9	1.359 (3)	C8—H8A	0.9300
O1—N1	1.396 (3)	C5—C6	1.378 (3)
C4—C3	1.386 (3)	C6—C1	1.373 (4)
C4—C5	1.390 (3)	C6—H6A	0.9300
C4—C7	1.470 (3)	C3—C2	1.376 (4)
C10—O2	1.224 (3)	C3—H3A	0.9300
C10—N2	1.320 (3)	C2—C1	1.377 (4)
C10—C9	1.485 (3)	C2—H2B	0.9300
N2—C11	1.460 (3)	C11—H11A	0.9600
N2—H2A	0.8600	C11—H11B	0.9600
N1—C7	1.312 (3)	C11—H11C	0.9600
C7—C8	1.416 (3)	C1—H1B	0.9300
C9—O1—N1	108.32 (16)	C6—C5—Cl1	117.47 (19)
C3—C4—C5	117.0 (2)	C4—C5—Cl1	120.82 (17)
C3—C4—C7	119.8 (2)	C1—C6—C5	120.1 (2)
C5—C4—C7	123.1 (2)	C1—C6—H6A	120.0
O2—C10—N2	124.4 (2)	C5—C6—H6A	120.0
O2—C10—C9	120.7 (2)	C2—C3—C4	121.6 (2)
N2—C10—C9	114.88 (19)	C2—C3—H3A	119.2
C10—N2—C11	122.0 (2)	C4—C3—H3A	119.2
C10—N2—H2A	119.0	C3—C2—C1	120.3 (3)
C11—N2—H2A	119.0	C3—C2—H2B	119.9

C7—N1—O1	105.85 (18)	C1—C2—H2B	119.9
N1—C7—C8	111.17 (19)	N2—C11—H11A	109.5
N1—C7—C4	118.6 (2)	N2—C11—H11B	109.5
C8—C7—C4	130.2 (2)	H11A—C11—H11B	109.5
C9—C8—C7	104.85 (19)	N2—C11—H11C	109.5
C9—C8—H8A	127.6	H11A—C11—H11C	109.5
C7—C8—H8A	127.6	H11B—C11—H11C	109.5
C8—C9—O1	109.8 (2)	C6—C1—C2	119.4 (2)
C8—C9—C10	134.9 (2)	C6—C1—H1B	120.3
O1—C9—C10	115.23 (18)	C2—C1—H1B	120.3
C6—C5—C4	121.7 (2)		
O2—C10—N2—C11	1.6 (5)	O2—C10—C9—C8	179.7 (3)
C9—C10—N2—C11	−178.9 (2)	N2—C10—C9—C8	0.2 (4)
C9—O1—N1—C7	0.2 (2)	O2—C10—C9—O1	1.9 (3)
O1—N1—C7—C8	−0.3 (3)	N2—C10—C9—O1	−177.7 (2)
O1—N1—C7—C4	178.9 (2)	C3—C4—C5—C6	−1.3 (4)
C3—C4—C7—N1	−37.5 (3)	C7—C4—C5—C6	178.6 (2)
C5—C4—C7—N1	142.7 (2)	C3—C4—C5—Cl1	175.96 (18)
C3—C4—C7—C8	141.5 (2)	C7—C4—C5—Cl1	−4.2 (3)
C5—C4—C7—C8	−38.4 (4)	C4—C5—C6—C1	−0.3 (4)
N1—C7—C8—C9	0.2 (3)	Cl1—C5—C6—C1	−177.7 (2)
C4—C7—C8—C9	−178.8 (2)	C5—C4—C3—C2	1.4 (4)
C7—C8—C9—O1	0.0 (3)	C7—C4—C3—C2	−178.4 (2)
C7—C8—C9—C10	−178.0 (3)	C4—C3—C2—C1	0.0 (4)
N1—O1—C9—C8	−0.1 (3)	C5—C6—C1—C2	1.8 (5)
N1—O1—C9—C10	178.25 (19)	C3—C2—C1—C6	−1.7 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O2^{i}	0.86	2.14	2.983 (3)	165
C8—H8A···O2^{i}	0.93	2.33	3.173 (3)	151

Symmetry codes: (i) *x*+1/4, −*y*+1/4, *z*+1/4.

Fig. 1

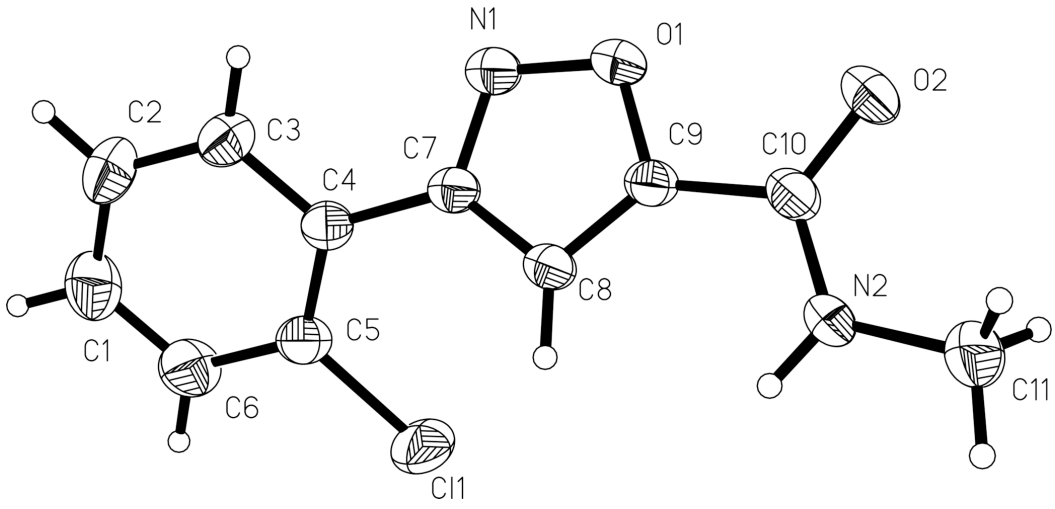

Fig. 2