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N,*N*-Dimethyl-3-phenylisoxazole-5-carboxamide

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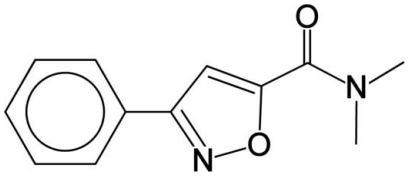
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.128; data-to-parameter ratio = 13.4.

In the title compound, $C_{12}H_{12}N_2O_2$, synthesized by ammonolysis of 3-phenylisoxazole-5-carbonyl chloride in dichloromethane, the dihedral angle between the isoxazole ring and the phenyl ring is 14.05 (7)°. In the crystal, centrosymmetrically related molecules are linked into dimers by pairs of C—H···O hydrogen bonds, generating rings of graph-set motif $R_2^2(10)$.

Related literature

For the biological activity of isoxazole derivatives, see: Lopes *et al.* (2011). For the synthesis and structure of a related compound, see: Wang *et al.* (2013).



Experimental

Crystal data

$C_{12}H_{12}N_2O_2$
M_r = 216.24
Monoclinic, $P2_1/c$
a = 7.596 (3) Å
b = 12.377 (6) Å
c = 12.123 (6) Å
β = 102.964 (8)°
V = 1110.7 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 K
0.36 × 0.25 × 0.13 mm

Data collection

Bruker APEXII CCD area detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.970, T_{max} = 0.989
5434 measured reflections
1977 independent reflections
1373 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.128
S = 1.03
1977 reflections
148 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O2^{i}	0.93	2.43	3.340 (3)	165

Symmetry code: (i) $-x, -y, -z$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ5100).

References

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supporting information

Acta Cryst. (2014). E**70**, o94 [https://doi.org/10.1107/S1600536813033199]

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S1. Comment

Isoxazoles are reactants or intermediates in the synthesis of compounds that have attracted chemists, biologists and pharmacologists interest. Isoxazole derivatives have been used as semiconductors and corrosion inhibitors. They also show widespread biological activities, and are employed as anthelmintics, antiparasitic, herbicidal, pesticides, fungicide and antiviral drugs (Lopes *et al.*, 2011).

In the molecule of the title compound (Fig. 1), the dihedral angle between the phenyl and the isoxazole rings is 14.05 (7)°, which is bigger than that of 7.37 (19)° observed in the related compound isopropyl 3-phenylisoxazole-5-carboxylate (Wang *et al.*, 2013). The bond lengths within the isoxazole ring are in agreement with those reported for isopropyl 3-phenylisoxazole-5-carboxylate. In the crystal, centrosymmetrically related molecules are linked into dimers by C—H···O hydrogen bonds (Table 1), generating a ring of graph-set motif $R^2_2(10)$.

S2. Experimental

3-Phenylisoxazole-5-carboxylic acid (10 mmol, 1.95 g; Wang *et al.*, 2013) was dissolved in 100 ml dichloromethane, then thionyl chloride (12 mmol, 1.43 g) was dropped into the solution and stirred for 20 minutes in ice bath. The solvent was removed under reduced pressure and the mixture was used for the next step without further purification. Dimethylamine (20 mmol, 0.9 g) was added subsequently and the mixture stirred for 6 h at room temperature. The resulting residue was purified as a white solid (1.82 g, 84% yield). Recrystallization in dichloromethane gave fine colourless crystals suitable for X-ray study. All chemicals were purchased by Sigma Aldrich Germany.

S3. Refinement

All H atoms were placed in idealized positions and allowed to ride on the respective parent atom, with C—H = 0.93–0.96 Å and with U_{iso}(H) = 1.2 U_{eq}(C) or 1.5U_{eq}(C) for methyl H atoms.

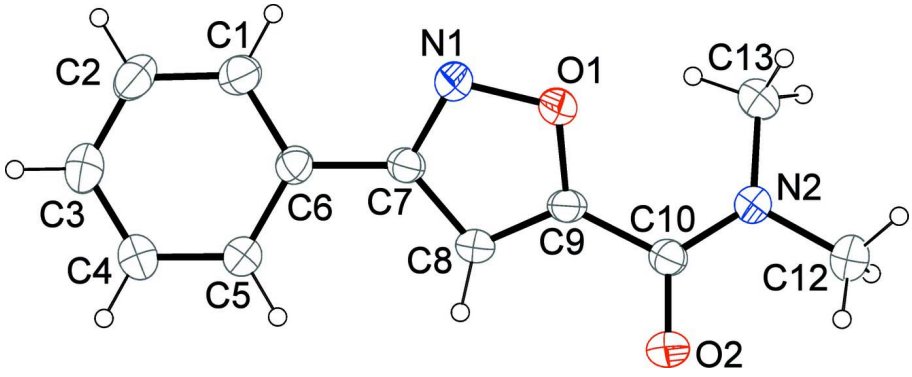

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

N,N-Dimethyl-3-phenylisoxazole-5-carboxamide

Crystal data

$C_{12}H_{12}N_2O_2$
M_r = 216.24
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 7.596 (3) Å
b = 12.377 (6) Å
c = 12.123 (6) Å
β = 102.964 (8)°
V = 1110.7 (9) Å^3
Z = 4
$F(000)$ = 456
D_x = 1.287 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
θ = 2.4–25.1°
μ = 0.09 mm^{-1}
T = 296 K
Block, colourless
0.36 × 0.25 × 0.13 mm

Data collection

Bruker APEXII CCD area detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.970, T_{max} = 0.989
5434 measured reflections
1977 independent reflections
1373 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 25.1°, θ_{min} = 2.4°
h = −5→9
k = −14→14
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.128
S = 1.03
1977 reflections
148 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2 + 0.0693P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.007 (2)

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.4029 (3)	0.01587 (14)	0.33843 (15)	0.0628 (6)
O1	0.3706 (2)	0.11427 (10)	0.27668 (12)	0.0616 (5)
O2	0.1335 (2)	0.15233 (11)	−0.00766 (12)	0.0643 (5)
C1	0.3700 (3)	−0.19723 (17)	0.43043 (18)	0.0575 (6)
H1	0.4227	−0.1441	0.4815	0.069*
C2	0.3675 (3)	−0.30437 (19)	0.4660 (2)	0.0659 (7)
H2	0.4206	−0.3224	0.5405	0.079*
C3	0.2870 (3)	−0.38412 (18)	0.3915 (2)	0.0654 (7)
H3	0.2847	−0.4552	0.4160	0.078*
C4	0.2103 (3)	−0.35728 (18)	0.2807 (2)	0.0654 (7)
H4	0.1563	−0.4108	0.2305	0.079*
C5	0.2126 (3)	−0.25108 (17)	0.24309 (18)	0.0538 (6)
H5	0.1606	−0.2341	0.1681	0.065*
C6	0.2930 (3)	−0.16985 (15)	0.31781 (16)	0.0448 (5)
C7	0.2953 (3)	−0.05662 (15)	0.27653 (16)	0.0431 (5)
C8	0.1948 (3)	−0.00969 (15)	0.17427 (16)	0.0470 (5)
H8	0.1123	−0.0440	0.1166	0.056*
C9	0.2449 (3)	0.09524 (15)	0.17905 (16)	0.0440 (5)
C10	0.1868 (3)	0.18267 (15)	0.09211 (17)	0.0460 (5)
N2	0.1934 (2)	0.28752 (12)	0.12311 (13)	0.0493 (5)
C12	0.1405 (4)	0.36960 (17)	0.03430 (19)	0.0701 (7)
H12A	0.1469	0.3393	−0.0376	0.105*
H12B	0.2209	0.4303	0.0506	0.105*
H12C	0.0192	0.3929	0.0319	0.105*
C13	0.2324 (3)	0.32936 (17)	0.23967 (17)	0.0613 (6)
H13A	0.2315	0.2708	0.2915	0.092*
H13B	0.1421	0.3814	0.2472	0.092*
H13C	0.3491	0.3632	0.2564	0.092*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0738 (13)	0.0460 (10)	0.0568 (12)	−0.0028 (9)	−0.0101 (10)	0.0042 (8)
O1	0.0719 (10)	0.0442 (8)	0.0564 (9)	−0.0073 (7)	−0.0118 (8)	−0.0004 (7)
O2	0.0872 (12)	0.0580 (9)	0.0411 (9)	−0.0080 (8)	0.0000 (8)	−0.0026 (7)
C1	0.0584 (14)	0.0616 (13)	0.0488 (13)	−0.0027 (11)	0.0040 (11)	0.0037 (11)
C2	0.0668 (15)	0.0713 (16)	0.0552 (14)	0.0036 (12)	0.0048 (12)	0.0208 (12)
C3	0.0724 (16)	0.0536 (14)	0.0705 (17)	0.0020 (12)	0.0169 (13)	0.0148 (12)
C4	0.0806 (17)	0.0477 (13)	0.0657 (16)	−0.0039 (11)	0.0117 (13)	−0.0004 (11)
C5	0.0626 (14)	0.0483 (12)	0.0475 (12)	0.0016 (10)	0.0060 (11)	0.0026 (10)
C6	0.0430 (11)	0.0442 (11)	0.0458 (11)	0.0019 (9)	0.0073 (9)	0.0020 (9)
C7	0.0422 (11)	0.0431 (11)	0.0421 (12)	0.0000 (9)	0.0052 (9)	−0.0049 (9)
C8	0.0517 (12)	0.0461 (12)	0.0394 (11)	−0.0049 (9)	0.0017 (10)	−0.0041 (9)
C9	0.0457 (11)	0.0465 (11)	0.0368 (11)	−0.0007 (9)	0.0031 (9)	−0.0055 (9)
C10	0.0497 (12)	0.0449 (11)	0.0425 (12)	−0.0056 (9)	0.0087 (10)	−0.0027 (9)

N2	0.0597 (11)	0.0432 (9)	0.0425 (10)	−0.0016 (8)	0.0060 (8)	0.0020 (8)
C12	0.0889 (18)	0.0525 (13)	0.0637 (16)	−0.0034 (12)	0.0059 (14)	0.0114 (11)
C13	0.0781 (16)	0.0486 (12)	0.0551 (14)	−0.0018 (11)	0.0107 (12)	−0.0078 (11)

Geometric parameters (Å, º)

N1—C7	1.327 (2)	C6—C7	1.490 (3)
N1—O1	1.422 (2)	C7—C8	1.425 (3)
O1—C9	1.364 (2)	C8—C9	1.351 (3)
O2—C10	1.244 (2)	C8—H8	0.9300
C1—C2	1.396 (3)	C9—C10	1.506 (3)
C1—C6	1.401 (3)	C10—N2	1.349 (2)
C1—H1	0.9300	N2—C12	1.469 (2)
C2—C3	1.384 (3)	N2—C13	1.471 (2)
C2—H2	0.9300	C12—H12A	0.9600
C3—C4	1.380 (3)	C12—H12B	0.9600
C3—H3	0.9300	C12—H12C	0.9600
C4—C5	1.393 (3)	C13—H13A	0.9600
C4—H4	0.9300	C13—H13B	0.9600
C5—C6	1.399 (3)	C13—H13C	0.9600
C5—H5	0.9300		
C7—N1—O1	105.64 (16)	C9—C8—H8	127.4
C9—O1—N1	108.28 (14)	C7—C8—H8	127.4
C2—C1—C6	119.9 (2)	C8—C9—O1	109.81 (16)
C2—C1—H1	120.0	C8—C9—C10	128.71 (18)
C6—C1—H1	120.0	O1—C9—C10	121.42 (16)
C3—C2—C1	120.7 (2)	O2—C10—N2	123.01 (18)
C3—C2—H2	119.6	O2—C10—C9	116.34 (17)
C1—C2—H2	119.6	N2—C10—C9	120.64 (17)
C4—C3—C2	119.4 (2)	C10—N2—C12	118.31 (17)
C4—C3—H3	120.3	C10—N2—C13	126.38 (16)
C2—C3—H3	120.3	C12—N2—C13	115.05 (16)
C3—C4—C5	120.8 (2)	N2—C12—H12A	109.5
C3—C4—H4	119.6	N2—C12—H12B	109.5
C5—C4—H4	119.6	H12A—C12—H12B	109.5
C4—C5—C6	120.2 (2)	N2—C12—H12C	109.5
C4—C5—H5	119.9	H12A—C12—H12C	109.5
C6—C5—H5	119.9	H12B—C12—H12C	109.5
C5—C6—C1	118.91 (18)	N2—C13—H13A	109.5
C5—C6—C7	119.67 (18)	N2—C13—H13B	109.5
C1—C6—C7	121.42 (18)	H13A—C13—H13B	109.5
N1—C7—C8	110.98 (17)	N2—C13—H13C	109.5
N1—C7—C6	119.88 (17)	H13A—C13—H13C	109.5
C8—C7—C6	129.14 (17)	H13B—C13—H13C	109.5
C9—C8—C7	105.29 (17)		
C7—N1—O1—C9	−0.6 (2)	N1—C7—C8—C9	−1.2 (2)

C6—C1—C2—C3	−1.1 (3)	C6—C7—C8—C9	179.07 (19)
C1—C2—C3—C4	0.7 (4)	C7—C8—C9—O1	0.8 (2)
C2—C3—C4—C5	−0.1 (4)	C7—C8—C9—C10	177.71 (19)
C3—C4—C5—C6	−0.1 (3)	N1—O1—C9—C8	−0.1 (2)
C4—C5—C6—C1	−0.2 (3)	N1—O1—C9—C10	−177.31 (16)
C4—C5—C6—C7	179.77 (19)	C8—C9—C10—O2	−24.5 (3)
C2—C1—C6—C5	0.8 (3)	O1—C9—C10—O2	152.16 (19)
C2—C1—C6—C7	−179.19 (19)	C8—C9—C10—N2	155.4 (2)
O1—N1—C7—C8	1.1 (2)	O1—C9—C10—N2	−27.9 (3)
O1—N1—C7—C6	−179.12 (15)	O2—C10—N2—C12	−2.0 (3)
C5—C6—C7—N1	−165.58 (19)	C9—C10—N2—C12	178.11 (18)
C1—C6—C7—N1	14.4 (3)	O2—C10—N2—C13	171.7 (2)
C5—C6—C7—C8	14.1 (3)	C9—C10—N2—C13	−8.2 (3)
C1—C6—C7—C8	−165.9 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O2^{i}	0.93	2.43	3.340 (3)	165

Symmetry code: (i) −*x*, −*y*, −*z*.