3-Phenylisoxazolin-5-one: a redetermination

Abdelhanine Essaghouani,[a]* Mohammed Boulhaoua,[a] Latifa El Ouasif,[a] Nada Kheira Sebbar,[a] El Mokhtar Essassi[a] and Joel T. Mague[b]

[a]Laboratoire de Chimie Organique Hétérocyclique, URAC 21, Pôle de Compétence Pharmacochimie, Av Ibn Battouta, BP 1014, Faculté des Sciences, Université Mohammed V, Rabat, Morocco, and [b]Department of Chemistry, Tulane University, New Orleans, LA 70118, USA. *Correspondence e-mail: essaghouani.hanine@gmail.com

The structure of the title molecule, $C_9H_7NO_2$, has been redetermined to improved precision and the H atoms located [Cannas *et al.* (1969). *Acta Cryst.* **B25**, 1050]. The five-membered ring is almost planar (r.m.s. deviation = 0.006 Å) and subtends a dihedral angle of 2.45 (6)° with the benzene ring. In the crystal, molecules form ribbons running parallel to the *a*-axis direction through a combination of C—H···N and C—H···O hydrogen bonds. 'Stair-step' offset π–π stacking interactions are also observed.

3D view

Chemical scheme
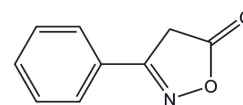
Structure description

Isoxazole derivatives are employed in different areas of pharmaceuticals such as anti-fungal (Mares, *et al.*, 2002), antibacterial (Kwon, *et al.*, 1995), and anti-inflammatory agents (Panda, *et al.*, 2009).

In an attempt to prepare a different compound (shown in Fig. 1), the title molecule (Fig. 2) was obtained instead. Noting that the original structure determination (Cannas *et al.*, 1969) cited in the Cambridge Crystallographic Database was performed at room temperature with film data (R_1 = 0.108), we felt that a detailed report of the low temperature structure (R_1 = 0.0338) of the title molecule was warranted. The present determination decreases the s.u.s on the bond distances and bond angles to about one fourth to one sixth of those in the original determination as well as unambiguously locating and refining the hydrogen atoms. The molecule is twisted about the C1···C4 axis by 2.45 (6)°, which is almost identical to the degree of twist found previously (2.45°).

In the crystal, a combination of pairwise C5—H5···N1[ii] [symmetry code: (ii) $1 - x$, $-y$, $1 - z$] and single C8—H8···O2[iii] [symmetry code: (iii) $-1 + x$, y, z] hydrogen bonds (Table 1 and Fig. 3) form ribbons running parallel to the *a* direction and alternately inclined at 32.7 (1) and −32.7 (1)° to (001). This motif was noted in the earlier report but
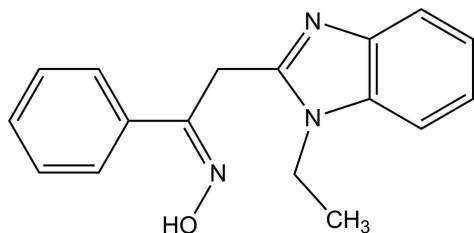
Figure 1
The intended compound.

we find, in addition, that the ribbons are formed into 'stair-step' stacks through complementary, offset π–π-stacking interactions between centrosymmetrically related six-membered and five-membered rings [centroid–centroid separation = 3.812 (1) Å, dihedral angle = 2.45 (6)°] (Fig. 2).

Synthesis and crystallization

A mixture of 4-phenyl-1,5-benzodiazepin-2-one (1.18 g, 5.0 mmol) and hydroxylamine hydrochloride (0.86 g, 12.5 mmol) in anhydrous ethanol (40 ml) was stirred at room temperature for 24 h. The solvent was evaporated under reduced pressure. The resulted solid residue was recrystallized from ethanol solution to afford the title compound as orange crystals (yield: 65%).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.
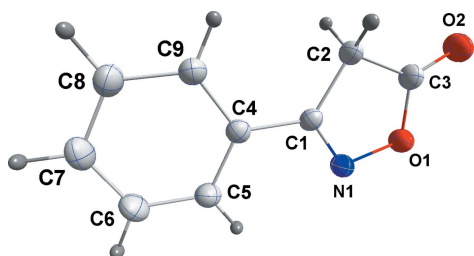

Figure 2
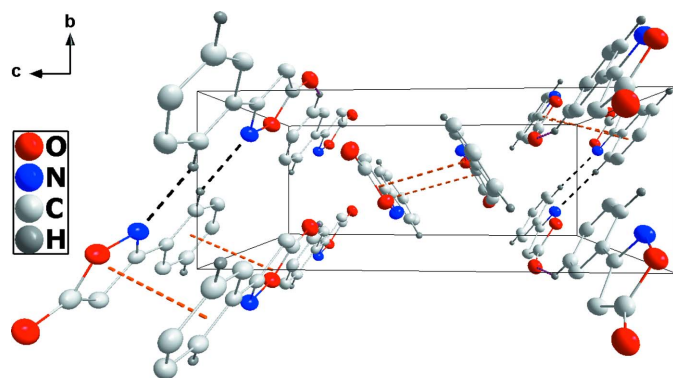The title molecule with 50% probability displacement ellipsoids.

Figure 3
The packing viewed along the *a* axis with C—H···N and C—H···O hydrogen bonds and π–π stacking interactions shown, respectively, as black, purple and orange dotted lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···N1^{i}	0.995 (16)	2.602 (16)	3.5525 (17)	160.0 (12)
C5—H5···N1^{ii}	0.991 (17)	2.55 (2)	3.438 (2)	149 (1)
C8—H8···O2^{iii}	0.972 (18)	2.57 (2)	3.306 (2)	133 (1)

Symmetry codes: (i) $x, y + 1, z$; (ii) $-x + 1, -y, -z + 1$; (iii) $x - 1, y, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_9H_7NO_2$
M_r	161.16
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	150
a, b, c (Å)	9.9869 (6), 5.3008 (3), 13.9874 (9)
β (°)	93.106 (2)
V (Å^3)	739.39 (8)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.86
Crystal size (mm)	0.19 × 0.12 × 0.06
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.83, 0.95
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5465, 1433, 1282
R_{int}	0.029
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.034, 0.089, 1.10
No. of reflections	1433
No. of parameters	138
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.14

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015a), *SHELXL2014* (Sheldrick, 2015b), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

References

Brandenburg, K. & Putz, H. (2012). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2016). *APEX3*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cannas, M., Biagini, S. & Marongiu, G. (1969). *Acta Cryst.* B**25**, 1050–1056.
Kwon, T., Heiman, A. S., Oriaku, E. T., Yoon, K. & Lee, H. J. (1995). *J. Med. Chem.* **38**, 1048–1051.
Mares, D., Romagnoli, C., Tosi, B., Benvegnù, R., Bruni, A. & Vicentini, C. B. (2002). *Fungal Genet. Biol.* **36**, 47–57.
Panda, S. S., Chowdary, P. V. R. & Jayashree, B. S. (2009). *Indian J. Pharm. Sci.* **71**, 684–687.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015a). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015b). *Acta Cryst.* C**71**, 3–8.

full crystallographic data

3-Phenylisoxazolin-5-one: a redetermination

Abdelhanine Essaghouani, Mohammed Boulhaoua, Latifa El Ouasif, Nada Kheira Sebbar, El Mokhtar Essassi and Joel T. Mague

3-Phenylisoxazolin-5-one

Crystal data

$C_9H_7NO_2$	$F(000) = 336$
$M_r = 161.16$	$D_x = 1.448$ Mg m^{-3}
Monoclinic, $P2_1/n$	Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
$a = 9.9869$ (6) Å	Cell parameters from 4379 reflections
$b = 5.3008$ (3) Å	$\theta = 5.6$–$72.1°$
$c = 13.9874$ (9) Å	$\mu = 0.86$ mm^{-1}
$\beta = 93.106$ (2)°	$T = 150$ K
$V = 739.39$ (8) Å^3	Plate, orange
$Z = 4$	$0.19 \times 0.12 \times 0.06$ mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer	$T_{min} = 0.83$, $T_{max} = 0.95$
Radiation source: INCOATEC IμS micro-focus source	5465 measured reflections
	1433 independent reflections
Mirror monochromator	1282 reflections with $I > 2\sigma(I)$
Detector resolution: 10.4167 pixels mm^{-1}	$R_{int} = 0.029$
ω scans	$\theta_{max} = 72.1°$, $\theta_{min} = 5.3°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)	$h = -12 \rightarrow 12$
	$k = -6 \rightarrow 5$
	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.034$	Hydrogen site location: difference Fourier map
$wR(F^2) = 0.089$	All H-atom parameters refined
$S = 1.10$	$w = 1/[\sigma^2(F_o^2) + (0.0441P)^2 + 0.1913P]$ where $P = (F_o^2 + 2F_c^2)/3$
1433 reflections	
138 parameters	$(\Delta/\sigma)_{max} < 0.001$
0 restraints	$\Delta\rho_{max} = 0.26$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{min} = -0.14$ e Å^{-3}
	Extinction correction: *SHELXL2014* (Sheldrick, 2015*b*), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
	Extinction coefficient: 0.0097 (11)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
O1	0.72988 (8)	0.35740 (17)	0.61102 (6)	0.0291 (3)
O2	0.83139 (9)	0.67872 (19)	0.68908 (7)	0.0345 (3)
N1	0.59421 (10)	0.2725 (2)	0.59036 (8)	0.0276 (3)
C1	0.51416 (12)	0.4307 (2)	0.62725 (8)	0.0219 (3)
C2	0.58547 (12)	0.6434 (2)	0.67731 (9)	0.0254 (3)
H2A	0.5637 (15)	0.811 (3)	0.6485 (11)	0.033 (4)*
H2B	0.5708 (16)	0.650 (3)	0.7461 (12)	0.036 (4)*
C3	0.72877 (12)	0.5762 (2)	0.66339 (9)	0.0260 (3)
C4	0.36809 (12)	0.3949 (2)	0.61691 (8)	0.0223 (3)
C5	0.31305 (13)	0.1913 (2)	0.56468 (9)	0.0270 (3)
H5	0.3730 (16)	0.070 (3)	0.5343 (11)	0.037 (4)*
C6	0.17523 (13)	0.1648 (3)	0.55302 (9)	0.0308 (3)
H6	0.1396 (18)	0.020 (4)	0.5152 (13)	0.050 (5)*
C7	0.09063 (13)	0.3393 (3)	0.59296 (9)	0.0297 (3)
H7	−0.0084 (17)	0.321 (3)	0.5830 (11)	0.035 (4)*
C8	0.14499 (13)	0.5399 (3)	0.64548 (9)	0.0293 (3)
H8	0.0864 (18)	0.661 (3)	0.6743 (12)	0.043 (4)*
C9	0.28324 (12)	0.5680 (2)	0.65752 (9)	0.0258 (3)
H9	0.3184 (16)	0.708 (3)	0.6934 (11)	0.037 (4)*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0220 (4)	0.0294 (5)	0.0360 (5)	0.0020 (3)	0.0029 (4)	−0.0048 (4)
O2	0.0227 (5)	0.0418 (6)	0.0390 (5)	−0.0033 (4)	0.0001 (4)	−0.0054 (4)
N1	0.0234 (5)	0.0263 (6)	0.0329 (6)	0.0000 (4)	0.0018 (4)	−0.0038 (4)
C1	0.0237 (6)	0.0192 (6)	0.0228 (5)	0.0013 (4)	0.0010 (4)	0.0023 (5)
C2	0.0218 (6)	0.0232 (6)	0.0311 (6)	0.0008 (5)	0.0001 (5)	−0.0028 (5)
C3	0.0240 (6)	0.0280 (6)	0.0259 (6)	0.0007 (5)	0.0014 (5)	0.0009 (5)
C4	0.0242 (6)	0.0204 (6)	0.0221 (6)	−0.0006 (4)	0.0001 (4)	0.0028 (5)
C5	0.0294 (6)	0.0230 (6)	0.0285 (6)	−0.0005 (5)	0.0004 (5)	−0.0008 (5)
C6	0.0315 (7)	0.0289 (7)	0.0313 (7)	−0.0070 (5)	−0.0036 (5)	−0.0008 (5)
C7	0.0247 (6)	0.0337 (7)	0.0305 (6)	−0.0036 (5)	−0.0014 (5)	0.0060 (5)
C8	0.0248 (6)	0.0306 (7)	0.0328 (7)	0.0023 (5)	0.0033 (5)	0.0018 (6)
C9	0.0252 (6)	0.0238 (6)	0.0284 (6)	0.0003 (5)	0.0011 (5)	−0.0012 (5)

Geometric parameters (Å, º)

O1—C3	1.3720 (16)	C4—C5	1.3990 (17)
O1—N1	1.4420 (13)	C5—C6	1.3843 (18)
O2—C3	1.1980 (16)	C5—H5	0.991 (17)
N1—C1	1.2857 (16)	C6—C7	1.390 (2)
C1—C4	1.4704 (16)	C6—H6	0.988 (19)
C1—C2	1.4881 (17)	C7—C8	1.386 (2)
C2—C3	1.4977 (16)	C7—H7	0.995 (16)
C2—H2A	0.995 (16)	C8—C9	1.3899 (17)
C2—H2B	0.981 (16)	C8—H8	0.972 (18)
C4—C9	1.3913 (17)	C9—H9	0.952 (17)
C3—O1—N1	109.63 (8)	C5—C4—C1	120.71 (11)
C1—N1—O1	108.30 (10)	C6—C5—C4	119.98 (12)
N1—C1—C4	120.76 (11)	C6—C5—H5	120.2 (9)
N1—C1—C2	113.00 (10)	C4—C5—H5	119.8 (9)
C4—C1—C2	126.23 (10)	C5—C6—C7	120.49 (12)
C1—C2—C3	101.24 (10)	C5—C6—H6	118.0 (11)
C1—C2—H2A	113.4 (9)	C7—C6—H6	121.5 (11)
C3—C2—H2A	110.5 (9)	C8—C7—C6	119.61 (12)
C1—C2—H2B	113.2 (9)	C8—C7—H7	120.3 (9)
C3—C2—H2B	109.3 (9)	C6—C7—H7	120.1 (9)
H2A—C2—H2B	109.0 (13)	C7—C8—C9	120.30 (12)
O2—C3—O1	120.81 (11)	C7—C8—H8	120.1 (10)
O2—C3—C2	131.38 (12)	C9—C8—H8	119.6 (10)
O1—C3—C2	107.81 (10)	C8—C9—C4	120.18 (12)
C9—C4—C5	119.43 (11)	C8—C9—H9	118.9 (9)
C9—C4—C1	119.84 (11)	C4—C9—H9	120.9 (10)
C3—O1—N1—C1	0.43 (13)	N1—C1—C4—C5	−1.60 (17)
O1—N1—C1—C4	179.36 (9)	C2—C1—C4—C5	177.44 (11)
O1—N1—C1—C2	0.20 (14)	C9—C4—C5—C6	0.62 (18)
N1—C1—C2—C3	−0.68 (14)	C1—C4—C5—C6	−177.90 (11)
C4—C1—C2—C3	−179.79 (11)	C4—C5—C6—C7	−0.07 (19)
N1—O1—C3—O2	179.42 (11)	C5—C6—C7—C8	−0.5 (2)
N1—O1—C3—C2	−0.86 (13)	C6—C7—C8—C9	0.56 (19)
C1—C2—C3—O2	−179.41 (14)	C7—C8—C9—C4	−0.01 (19)
C1—C2—C3—O1	0.91 (12)	C5—C4—C9—C8	−0.58 (18)
N1—C1—C4—C9	179.89 (11)	C1—C4—C9—C8	177.95 (11)
C2—C1—C4—C9	−1.07 (18)		

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C2—H2A⋯N1[i]	0.995 (16)	2.602 (16)	3.5525 (17)	160.0 (12)

C5—H5···N1[ii]	0.991 (17)	2.55 (2)	3.438 (2)	149 (1)
C8—H8···O2[iii]	0.972 (18)	2.57 (2)	3.306 (2)	133 (1)

Symmetry codes: (i) x, $y+1$, z; (ii) $-x+1$, $-y$, $-z+1$; (iii) $x-1$, y, z.