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7a-Phenyltetrahydropyrrolo[2,1-*b*]oxazol-5(6*H*)-one

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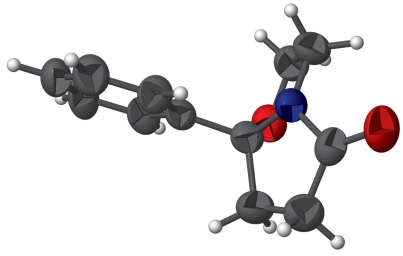
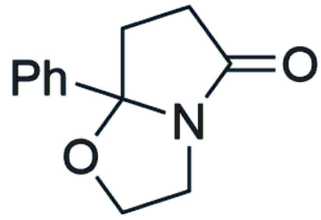
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{12}H_{13}NO_2$, the pyrrolidinone moiety is almost flat while the oxazole ring adopts an envelope conformation with the carbon atom bearing the phenyl substituent as the flap: the angle between the mean planes of the fused heterocyclic rings is 45.47 (19)°. In the crystal, C—H···O and C—H···π contacts link the molecules into infinite [010] chains.

3D view



Chemical scheme



Structure description

The title compound, $C_{12}H_{13}NO_2$, has been reported in the literature several times (Aeberli & Houlihan, 1969; Aeberli *et al.*, 1976; Amal'chieva & Egorova, 2006). It has been also reported for its anti-depressant (Aeberli *et al.*, 1976) and anti-convulsant activities (Trapani *et al.*, 1996) as well as the synthetic potential to obtain 4,5-dihydro-2*H*-pyridazin-3-ones (Lim *et al.*, 2003). We now describe its crystal structure.

Molecules of title compound consist of pyrrolidinone and oxazole rings fused *via* the C3—N1 edge into a bicyclic system (Fig. 1). The pyrrolidinone moiety is almost flat (r.m.s. deviation = 0.054 Å) with a maximum torsion angle N1—C6—C5—C4 of 13.4 (5)°, whereas the minimum torsion angle C5—C4—C3—N1 is 2.7 (5)°. The oxazole ring is more twisted and adopts an envelope conformation with atom C3 as the flap and a maximum torsion angle C2—O1—C3—N1 of −35.7 (4)°. The heterocyclic rings are fused with a dihedral angle between their mean planes of 45.47 (19)°. The phenyl substituent is located orthogonally to the mean plane of the whole bicycle [dihedral angle = 89.28 (14)°].

In contrast to closely related pyrrolopyrimidinones (Grinev *et al.*, 2020), there is no classical hydrogen bonding in the crystal of the title molecule, obviously due to the

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

*Cg*3 is the centroid of the C7–C12 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5*A*···O2^{i}	0.97	2.58	3.346 (6)	136
C10—H10···*Cg*3^{ii}	0.93	2.88	3.734 (6)	154

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

absence of NH groups (Fig. 2). The molecules are connected *via* weak C5—H5*A*···O2 links (Table 1) to generate infinite chains directed along [010]. The H5*A*···O2 distance of 2.58 Å is significantly longer than the corresponding distance in pyrrolopyrimidinones [2.28 (5)–2.306 (18) Å]. Moreover, there are C10—H10···π contacts to an adjacent phenyl ring (Fig. 3), which reinforce the [010] chains.

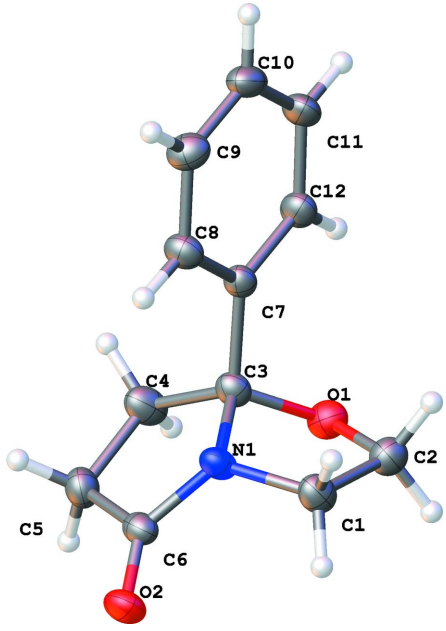


Figure 1
The molecular structure of the title compound showing 50% displacement ellipsoids.

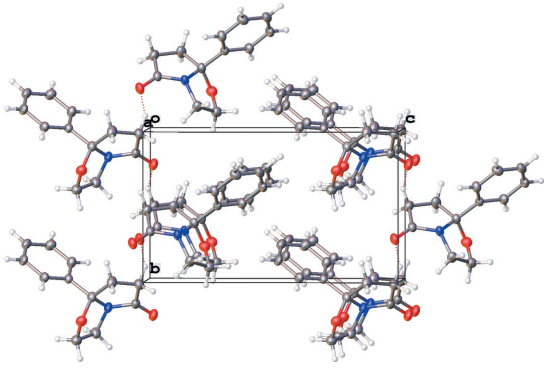


Figure 2
The packing of the title compound viewed down [100] showing hydrogen bonds as red dashed lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_{13}NO_2$
M_r	203.23
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	295
a, b, c (Å)	5.7173 (17), 7.346 (3), 12.436 (4)
β (°)	93.07 (3)
V (Å^3)	521.5 (3)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.55 × 0.1 × 0.08
Data collection	
Diffractometer	Agilent Technologies New Xcalibur, Ruby
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.217, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5059, 2406, 1462
R_{int}	0.048
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.691
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.054, 0.160, 1.06
No. of reflections	2406
No. of parameters	137
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.16, −0.15

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXS* (Sheldrick, 2008), *SHELXL* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

Synthesis and crystallization

5-Phenylfuran-2(3*H*)-one (1 g, 6 mmol) and ethanolamine (0.34 g, 6 mmol) were placed in a round-bottomed flask equipped with Dean–Stark apparatus. Dry benzene (30 ml) was added and the reaction mixture refluxed for 3–4 h. After

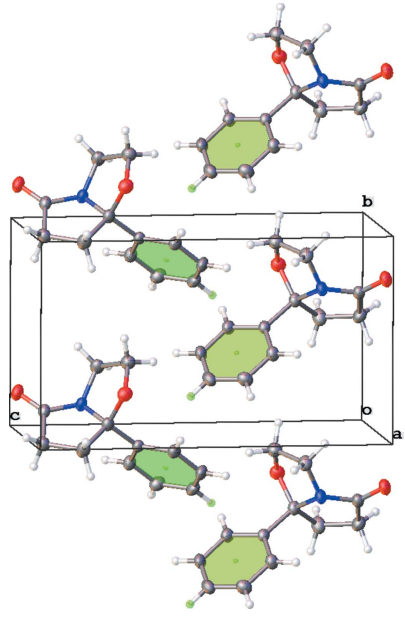


Figure 3
The packing of the title compound showing C—H···π interactions.

being left to stand overnight, the separated crystals and precipitate were washed with benzene and acetone and the solid placed in a vacuum desiccator for drying (yield 0.91 g, 75%; m.p. 65–67°C). The single crystal used for data collection was obtained directly from the cooled reaction mixture.

Refinement

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

Funding information

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References

Aeberli, P., Gogerty, J. H., Houlihan, W. J. & Iorio, L. C. (1976). *J. Med. Chem.* **19**, 436–438.
Aeberli, P. & Houlihan, W. J. (1969). *J. Org. Chem.* **34**, 165–170.
Agilent (2014). *CrysAlis PRO*. Agilent Technologies, USA.
Amal'chieva, O. A. & Egorova, A. Yu. (2006). *Russ. J. Org. Chem.* **42**, 1340–1343.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Grinev, V. S., Linkova, E. I., Krainov, M. N., Dmitriev, M. V. & Yegorova, A. Y. (2020). *Acta Cryst.* C**76**, 483–489.
Lim, Y. J., Angela, M. & Buonora, P. T. (2003). *Tetrahedron Lett.* **44**, 7799–7801.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Trapani, G., Franco, M., Latrofa, A., Carotti, A., Cellamare, S., Serra, M., Ghiani, C. A., Tuligi, G., Biggio, G. & Liso, G. (1996). *J. Pharm. Pharmacol.* **48**, 834–840.

full crystallographic data

IUCrData (2020). **5**, x200919 [https://doi.org/10.1107/S2414314620009190]

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Crystal data

$C_{12}H_{13}NO_2$
M_r = 203.23
Monoclinic, $P2_1$
a = 5.7173 (17) Å
b = 7.346 (3) Å
c = 12.436 (4) Å
β = 93.07 (3)°
V = 521.5 (3) Å^3
Z = 2
$F(000)$ = 216
D_x = 1.294 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1357 reflections
θ = 3.5–22.6°
μ = 0.09 mm^{-1}
T = 295 K
Needle, clear colourless
0.55 × 0.1 × 0.08 mm

Data collection

Agilent Technologies New Xcalibur, Ruby diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 10.4752 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlisPro; Agilent, 2014)
T_{min} = 0.217, T_{max} = 1.000
5059 measured reflections
2406 independent reflections
1462 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 29.4°, θ_{min} = 3.2°
h = −7→7
k = −9→9
l = −16→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.160
S = 1.06
2406 reflections
137 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0638P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: SHELXL2018/1 (Sheldrick 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.094 (18)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4191 (7)	0.8471 (7)	0.1967 (3)	0.0669 (12)
H1A	0.440537	0.941850	0.143766	0.080*
H1B	0.563590	0.831027	0.240152	0.080*
N1	0.3418 (5)	0.6763 (5)	0.1455 (2)	0.0560 (9)
O1	0.0340 (4)	0.7581 (5)	0.2424 (2)	0.0666 (9)
C2	0.2140 (7)	0.8897 (6)	0.2663 (3)	0.0694 (13)
H2A	0.264333	0.883883	0.341924	0.083*
H2B	0.155301	1.011334	0.250689	0.083*
O2	0.4289 (5)	0.7275 (5)	−0.0288 (2)	0.0743 (10)
C3	0.1537 (6)	0.6008 (6)	0.2049 (3)	0.0545 (10)
C4	−0.0033 (7)	0.4983 (8)	0.1217 (3)	0.0750 (13)
H4A	−0.017904	0.371365	0.141669	0.090*
H4B	−0.158290	0.552282	0.115247	0.090*
C5	0.1181 (7)	0.5165 (7)	0.0181 (3)	0.0686 (12)
H5A	0.180468	0.399755	−0.003026	0.082*
H5B	0.009114	0.559003	−0.038927	0.082*
C6	0.3109 (7)	0.6501 (6)	0.0374 (3)	0.0559 (10)
C7	0.2432 (6)	0.4828 (5)	0.2990 (3)	0.0489 (9)
C8	0.4440 (6)	0.3789 (6)	0.2926 (3)	0.0622 (11)
H8	0.529967	0.384189	0.231306	0.075*
C9	0.5168 (7)	0.2675 (6)	0.3775 (4)	0.0712 (12)
H9	0.653085	0.199387	0.373066	0.085*
C10	0.3912 (8)	0.2559 (7)	0.4680 (4)	0.0724 (12)
H10	0.441108	0.179917	0.524518	0.087*
C11	0.1929 (9)	0.3565 (8)	0.4745 (3)	0.0746 (13)
H11	0.107507	0.349304	0.535934	0.090*
C12	0.1163 (7)	0.4699 (6)	0.3904 (3)	0.0633 (11)
H12	−0.020343	0.537312	0.395607	0.076*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.068 (2)	0.072 (3)	0.061 (3)	−0.019 (3)	0.005 (2)	−0.004 (2)
N1	0.0587 (17)	0.066 (2)	0.0439 (19)	−0.0115 (16)	0.0075 (13)	0.0058 (16)
O1	0.0574 (14)	0.0675 (18)	0.076 (2)	0.0055 (15)	0.0118 (13)	0.0046 (16)
C2	0.075 (3)	0.063 (3)	0.070 (3)	−0.005 (2)	0.005 (2)	0.000 (2)
O2	0.090 (2)	0.082 (2)	0.0525 (18)	−0.0011 (18)	0.0205 (15)	0.0124 (16)
C3	0.0511 (19)	0.063 (2)	0.050 (2)	−0.0077 (18)	0.0097 (16)	0.001 (2)
C4	0.075 (3)	0.088 (4)	0.061 (3)	−0.026 (3)	−0.002 (2)	0.003 (3)
C5	0.078 (2)	0.068 (3)	0.060 (2)	−0.003 (2)	0.006 (2)	−0.012 (2)
C6	0.061 (2)	0.060 (3)	0.047 (2)	0.008 (2)	0.0061 (16)	0.005 (2)
C7	0.0494 (19)	0.051 (2)	0.047 (2)	−0.0065 (17)	0.0098 (14)	0.0002 (19)
C8	0.060 (2)	0.073 (3)	0.055 (2)	−0.001 (2)	0.0119 (18)	0.006 (2)
C9	0.064 (2)	0.068 (3)	0.082 (3)	0.005 (2)	0.007 (2)	0.012 (3)
C10	0.098 (3)	0.062 (3)	0.057 (3)	−0.011 (3)	−0.002 (2)	0.013 (2)

C11	0.106 (3)	0.069 (3)	0.052 (2)	−0.003 (3)	0.028 (2)	0.009 (2)
C12	0.070 (2)	0.062 (3)	0.059 (2)	−0.003 (2)	0.0219 (18)	0.002 (2)

Geometric parameters (Å, º)

C1—H1A	0.9700	C4—C5	1.502 (6)
C1—H1B	0.9700	C5—H5A	0.9700
C1—N1	1.464 (6)	C5—H5B	0.9700
C1—C2	1.527 (6)	C5—C6	1.486 (6)
N1—C3	1.447 (5)	C7—C8	1.384 (5)
N1—C6	1.361 (5)	C7—C12	1.385 (5)
O1—C2	1.432 (5)	C8—H8	0.9300
O1—C3	1.434 (5)	C8—C9	1.382 (6)
C2—H2A	0.9700	C9—H9	0.9300
C2—H2B	0.9700	C9—C10	1.370 (6)
O2—C6	1.230 (5)	C10—H10	0.9300
C3—C4	1.531 (5)	C10—C11	1.359 (7)
C3—C7	1.523 (5)	C11—H11	0.9300
C4—H4A	0.9700	C11—C12	1.389 (6)
C4—H4B	0.9700	C12—H12	0.9300
H1A—C1—H1B	109.3	C4—C5—H5A	110.3
N1—C1—H1A	111.5	C4—C5—H5B	110.3
N1—C1—H1B	111.5	H5A—C5—H5B	108.6
N1—C1—C2	101.5 (3)	C6—C5—C4	107.0 (4)
C2—C1—H1A	111.5	C6—C5—H5A	110.3
C2—C1—H1B	111.5	C6—C5—H5B	110.3
C3—N1—C1	108.8 (3)	N1—C6—C5	108.0 (4)
C6—N1—C1	124.8 (4)	O2—C6—N1	123.3 (4)
C6—N1—C3	112.9 (3)	O2—C6—C5	128.7 (4)
C2—O1—C3	105.2 (3)	C8—C7—C3	121.1 (3)
C1—C2—H2A	110.1	C8—C7—C12	118.8 (4)
C1—C2—H2B	110.1	C12—C7—C3	120.0 (3)
O1—C2—C1	108.0 (3)	C7—C8—H8	120.0
O1—C2—H2A	110.1	C9—C8—C7	120.0 (4)
O1—C2—H2B	110.1	C9—C8—H8	120.0
H2A—C2—H2B	108.4	C8—C9—H9	119.5
N1—C3—C4	105.6 (3)	C10—C9—C8	121.0 (4)
N1—C3—C7	112.5 (3)	C10—C9—H9	119.5
O1—C3—N1	103.8 (3)	C9—C10—H10	120.3
O1—C3—C4	110.1 (3)	C11—C10—C9	119.4 (4)
O1—C3—C7	110.8 (3)	C11—C10—H10	120.3
C7—C3—C4	113.6 (4)	C10—C11—H11	119.6
C3—C4—H4A	110.8	C10—C11—C12	120.8 (4)
C3—C4—H4B	110.8	C12—C11—H11	119.6
H4A—C4—H4B	108.9	C7—C12—C11	120.1 (4)
C5—C4—C3	104.8 (3)	C7—C12—H12	120.0
C5—C4—H4A	110.8	C11—C12—H12	120.0

C5—C4—H4B	110.8		
C1—N1—C3—O1	33.1 (3)	C3—O1—C2—C1	25.9 (4)
C1—N1—C3—C4	148.9 (4)	C3—C4—C5—C6	−9.5 (5)
C1—N1—C3—C7	−86.7 (4)	C3—C7—C8—C9	−177.5 (4)
C1—N1—C6—O2	32.0 (6)	C3—C7—C12—C11	177.4 (4)
C1—N1—C6—C5	−148.2 (4)	C4—C3—C7—C8	85.8 (4)
N1—C1—C2—O1	−5.7 (4)	C4—C3—C7—C12	−90.5 (4)
N1—C3—C4—C5	2.7 (5)	C4—C5—C6—N1	13.4 (5)
N1—C3—C7—C8	−34.1 (5)	C4—C5—C6—O2	−166.8 (4)
N1—C3—C7—C12	149.6 (4)	C6—N1—C3—O1	−109.9 (4)
O1—C3—C4—C5	114.1 (4)	C6—N1—C3—C4	5.9 (5)
O1—C3—C7—C8	−149.7 (3)	C6—N1—C3—C7	130.3 (4)
O1—C3—C7—C12	34.0 (5)	C7—C3—C4—C5	−121.0 (4)
C2—C1—N1—C3	−16.7 (4)	C7—C8—C9—C10	0.9 (6)
C2—C1—N1—C6	120.9 (4)	C8—C7—C12—C11	1.0 (6)
C2—O1—C3—N1	−35.7 (4)	C8—C9—C10—C11	−0.4 (7)
C2—O1—C3—C4	−148.3 (3)	C9—C10—C11—C12	0.2 (7)
C2—O1—C3—C7	85.2 (3)	C10—C11—C12—C7	−0.5 (7)
C3—N1—C6—O2	168.1 (4)	C12—C7—C8—C9	−1.1 (5)
C3—N1—C6—C5	−12.2 (5)		

Hydrogen-bond geometry (Å, º)

*Cg*3 is the centroid of the C7–C12 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5*A*···O2^{i}	0.97	2.58	3.346 (6)	136
C10—H10···*Cg*3^{ii}	0.93	2.88	3.734 (6)	154

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*; (ii) −*x*+1, *y*−1/2, −*z*+1.