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3-Phenyl-4*H*,6*H*-1,2,4-oxadiazol-5-one

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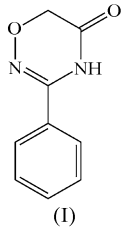
Molecules of the title compound, $C_9H_8N_2O_2$, are linked into complex sheets by a combination of N—H···O, C—H···N and C—H···O hydrogen bonds.

Comment

The title compound, (I) (Fig. 1), is a lactam which was obtained on the attempted recrystallization of $H_2N(Ph)C{=}N\text{-}OCH_2COOH$ from hot water.



(I)

The bond distances within the heterocyclic ring of (I) (Table 1) show very strong bond fixation in the C—N bonds. Thus, N2—C3 is very much shorter than C3—N4, which is rather long for its type (Allen *et al.*, 1987). These values effectively preclude any electron delocalization in the N2—C3—N4 fragment. The ring puckering parameters (Cremer & Pople, 1975) of θ = 66.5 (2)° and φ = 335.9 (2)° for the atom sequence O1—N2—C3—N4—C5—C6 indicate a screw-boat conformation for the heterocyclic ring (Evans & Boeyens, 1989). The torsion angles within this ring (Table 1) indicate the near-planarity of the O1—N2—C3—N4 fragment containing the N2=C3 double bond, and of the *cis*-amidic fragment C3—N4—C5—C6.

The molecules of (I) are linked into a sheet of some complexity *via* a combination of N—H···O, C—H···O and C—H···N hydrogen bonds (Table 2). The formation of the sheet can most readily be analysed and described in terms of two one-dimensional substructures.

Atom N4 in the molecule at (x, y, z) acts as hydrogen-bond donor to carbonyl atom O5 in the molecule at ($\frac{1}{2}-x$, $-\frac{1}{2}-y$, $1-z$), so generating a centrosymmetric $R_2^2(8)$ (Bernstein *et al.*, 1995) dimer centred at ($\frac{1}{4}$, $-\frac{1}{4}$, $\frac{1}{2}$) (Fig. 2). The formation of this dimer is reinforced by the C—H···O hydrogen bond which links the same two molecules in an $R_2^2(14)$ motif, in which the $R_2^2(8)$ ring is embedded, so producing two additional rings, of $R_2^1(7)$ type (Fig. 2).

These dimers are linked into [130] chains by one of the C—H···N hydrogen bonds. Aryl atom C36 in the molecule at (x, y, z) acts as donor to ring atom N2 in the molecule at ($1-x$, $1-y$, $1-z$), so generating a centrosymmetric $R_2^2(10)$ motif,

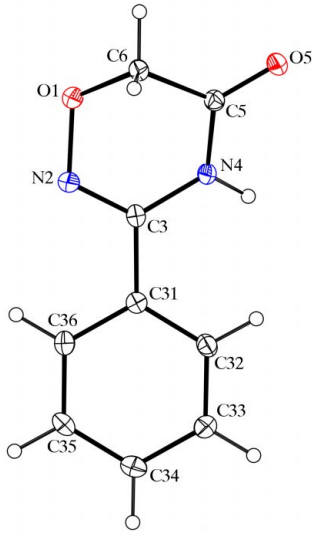


Figure 1
The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

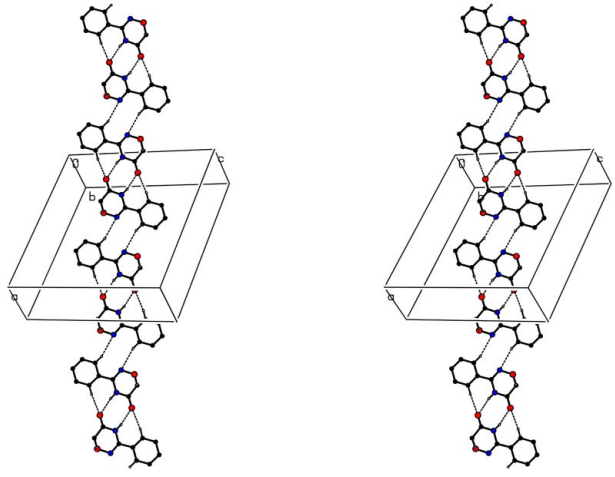


Figure 2
A stereoview of part of the crystal structure of (I), showing the formation of a [130] chain of edge-fused rings. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

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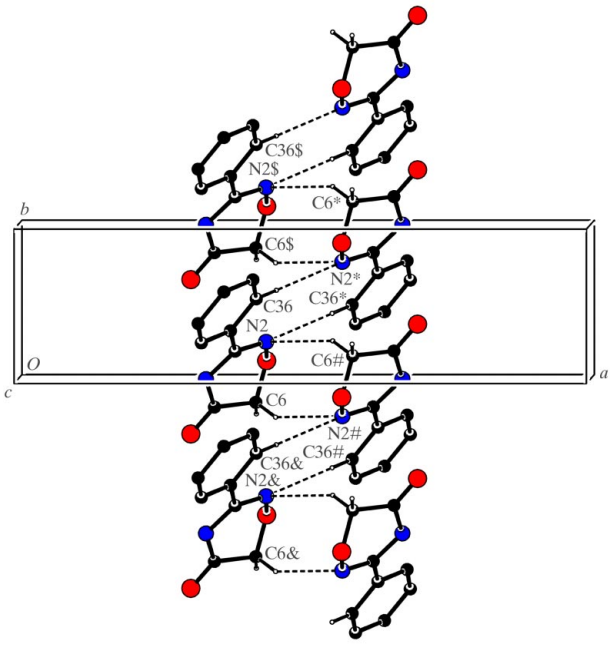

Figure 3
Part of the crystal structure of (I), showing the formation of an [010] chain of spiro-fused rings. For the sake of clarity, H atoms not involved in the motifs shown have been omitted. Atoms marked with an asterisk (*), hash (#), dollar sign ($) or ampersand (&) are at the symmetry positions $(1-x, 1-y, 1-z)$, $(1-x, -y, 1-z)$ $(x, 1+y, z)$ and $(x, y-1, z)$, respectively.

centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$, and linking pairs of the $R_2^2(8)$ dimers into a chain of centrosymmetric rings running parallel to the [130] direction (Fig. 2).

A second chain motif is generated by the combined action of the two C—H···N hydrogen bonds, which unexpectedly have the same acceptor, *viz*. ring atom N2. Ring atom C6 in the molecule at (x, y, z) acts as hydrogen-bond donor, *via* the axial atom H6*A*, to atom N2 in the molecule at $(1-x, -y, 1-z)$, so generating a centrosymmetric $R_2^2(8)$ ring centred at $(\frac{1}{2}, 0, \frac{1}{2})$. This motif, in combination with the $R_2^2(10)$ motif, also generated by paired C—H···N hydrogen bonds, produces a chain of spiro-fused rings running parallel to the [010] direction (Fig. 3). The combination of the [130] and [010] chains generates an (001) sheet, but there are no direction-specific interactions between adjacent sheets.

Experimental

The title compound was obtained on the attempted recrystallization of $H_2N(Ph)C{=}NOCH_2COOH$ (Forrester *et al.*, 1979) from hot water.

Crystal data

$C_9H_8N_2O_2$
$M_r = 176.17$
Monoclinic, $C2/c$
$a = 18.9100\ (10)$ Å
$b = 5.1093\ (2)$ Å
$c = 17.0632\ (9)$ Å
$\beta = 90.885\ (3)^\circ$
$V = 1648.40\ (14)$ Å^3
$Z = 8$
$D_x = 1.420$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1903 reflections
$\theta = 3.2$–27.6°
$\mu = 0.10$ mm^{-1}
$T = 120\ (2)$ K
Lath, colourless
0.55 × 0.12 × 0.03 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.954$, $T_{max} = 0.997$
10 146 measured reflections
1903 independent reflections
1200 reflections with $I > 2\sigma(I)$
$R_{int} = 0.066$
$\theta_{max} = 27.6^\circ$
$h = -24 \rightarrow 24$
$k = -6 \rightarrow 6$
$l = -20 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.136$
$S = 0.98$
1903 reflections
118 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0779P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—N2	1.4320 (18)	N4—C5	1.361 (2)
N2—C3	1.287 (2)	C5—C6	1.493 (2)
C3—N4	1.396 (2)	C6—O1	1.426 (2)
O1—N2—C3—N4	3.4 (2)	N4—C5—C6—O1	−33.9 (2)
N2—C3—N4—C5	21.4 (2)	C5—C6—O1—N2	58.6 (2)
C3—N4—C5—C6	−4.0 (2)	C6—O1—N2—C3	−43.56 (19)

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···O5^{i}	0.85	2.06	2.886 (2)	165
C32—H32···O5^{i}	0.95	2.39	3.296 (2)	159
C6—H6*A*···N2^{ii}	0.99	2.59	3.475 (2)	148
C36—H36···N2^{iii}	0.95	2.56	3.476 (2)	161

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

The systematic absences permitted *Cc* and *C*2/*c* as possible space groups; *C*2/*c* was selected and confirmed by the subsequent structure analysis. All H atoms were located from difference maps and then treated as riding, with C—H distances of 0.95 (aromatic) or 0.99 Å (CH_2) and an N—H distance of 0.85 Å, and with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PRPKAPPA* (Ferguson, 1999).

The X-ray data were collected at the EPSRC X-ray Crystallographic Service, University of Southampton, England; the authors thank the staff for all their help and advice. JNL thanks NCR Self-Service, Dundee, for grants which have provided computing facilities for this work. JLW thanks CNPq and FAPERJ for financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1774). Services for accessing these data are described at the back of the journal.

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supporting information

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3-Phenyl-4*H*,6*H*-1,2,4-oxadiazol-5-one

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Computing details

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

3-Phenyl-4H,6*H*-1,2,4-oxadiazolin-5-one

Crystal data

$C_9H_8N_2O_2$
$M_r = 176.17$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 18.910\ (1)$ Å
$b = 5.1093\ (2)$ Å
$c = 17.0632\ (9)$ Å
$\beta = 90.885\ (3)°$
$V = 1648.40\ (14)$ Å^3
$Z = 8$
$F(000) = 736$
$D_x = 1.420$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1903 reflections
$\theta = 3.2–27.6°$
$\mu = 0.10$ mm^{-1}
$T = 120$ K
Lath, colourless
0.55 × 0.12 × 0.03 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: Bruker-Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.954$, $T_{max} = 0.997$
10146 measured reflections
1903 independent reflections
1200 reflections with $I > 2\sigma(I)$
$R_{int} = 0.066$
$\theta_{max} = 27.6°$, $\theta_{min} = 3.2°$
$h = -24\rightarrow24$
$k = -6\rightarrow6$
$l = -20\rightarrow22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.136$
$S = 0.98$
1903 reflections
118 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
$w = 1/[\sigma^2(F_o^2) + (0.0779P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.43308 (6)	0.1136 (2)	0.40375 (7)	0.0266 (4)
O5	0.30092 (6)	−0.3600 (2)	0.42484 (7)	0.0272 (4)
N2	0.43312 (8)	0.2398 (3)	0.47870 (9)	0.0240 (4)
N4	0.32837 (7)	−0.0004 (3)	0.49774 (8)	0.0222 (4)
C3	0.37966 (9)	0.1813 (3)	0.52107 (11)	0.0200 (4)
C5	0.34240 (9)	−0.1851 (3)	0.44265 (10)	0.0222 (4)
C6	0.41412 (9)	−0.1560 (3)	0.40829 (11)	0.0243 (5)
C31	0.37249 (9)	0.3179 (3)	0.59706 (10)	0.0207 (4)
C32	0.32239 (9)	0.2428 (4)	0.65152 (11)	0.0253 (5)
C33	0.31777 (10)	0.3726 (4)	0.72243 (11)	0.0280 (5)
C34	0.36309 (10)	0.5784 (4)	0.74019 (11)	0.0284 (5)
C35	0.41289 (10)	0.6536 (4)	0.68611 (11)	0.0272 (5)
C36	0.41785 (10)	0.5258 (3)	0.61516 (11)	0.0246 (5)
H4	0.2896	−0.0106	0.5221	0.027*
H6A	0.4493	−0.2506	0.4412	0.029*
H6B	0.4143	−0.2341	0.3552	0.029*
H32	0.2912	0.1018	0.6400	0.030*
H33	0.2833	0.3204	0.7592	0.034*
H34	0.3600	0.6668	0.7890	0.034*
H35	0.4440	0.7947	0.6979	0.033*
H36	0.4523	0.5794	0.5785	0.030*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0312 (7)	0.0264 (7)	0.0223 (7)	−0.0068 (6)	0.0039 (6)	−0.0006 (6)
O5	0.0238 (7)	0.0293 (8)	0.0286 (8)	−0.0070 (6)	0.0019 (6)	−0.0062 (6)
N2	0.0265 (9)	0.0240 (9)	0.0215 (9)	−0.0030 (7)	0.0016 (7)	−0.0024 (7)
N4	0.0175 (8)	0.0256 (9)	0.0237 (9)	−0.0035 (6)	0.0033 (7)	−0.0030 (7)
C3	0.0176 (9)	0.0181 (9)	0.0244 (10)	−0.0002 (7)	−0.0012 (8)	0.0026 (8)
C5	0.0221 (10)	0.0234 (10)	0.0209 (10)	−0.0024 (8)	−0.0011 (8)	0.0007 (8)
C6	0.0245 (10)	0.0226 (10)	0.0258 (11)	−0.0049 (8)	0.0033 (8)	−0.0034 (8)
C31	0.0201 (9)	0.0200 (10)	0.0219 (10)	0.0016 (7)	−0.0011 (8)	0.0011 (8)
C32	0.0228 (10)	0.0247 (10)	0.0284 (11)	−0.0041 (8)	−0.0011 (8)	−0.0024 (9)
C33	0.0270 (10)	0.0318 (11)	0.0254 (11)	−0.0022 (8)	0.0036 (9)	−0.0011 (9)
C34	0.0310 (11)	0.0269 (11)	0.0271 (11)	0.0025 (9)	−0.0031 (9)	−0.0062 (9)
C35	0.0277 (11)	0.0239 (10)	0.0298 (11)	−0.0047 (8)	−0.0051 (9)	−0.0043 (9)
C36	0.0232 (9)	0.0225 (10)	0.0282 (11)	−0.0019 (8)	0.0008 (8)	0.0028 (8)

Geometric parameters (Å, º)

O1—N2	1.4320 (18)	C33—C34	1.387 (3)
N2—C3	1.287 (2)	C33—H33	0.95
C3—N4	1.396 (2)	C34—C35	1.383 (3)
N4—C5	1.361 (2)	C34—H34	0.95

C5—C6	1.493 (2)	C35—C36	1.380 (3)
C6—O1	1.426 (2)	C35—H35	0.95
C3—C31	1.481 (3)	C36—H36	0.95
C31—C32	1.391 (2)	N4—H4	0.85
C31—C36	1.397 (2)	C5—O5	1.2243 (19)
C32—C33	1.384 (3)	C6—H6A	0.99
C32—H32	0.95	C6—H6B	0.99

C6—O1—N2	112.52 (13)	C36—C35—H35	119.7
C3—N2—O1	114.05 (14)	C34—C35—H35	119.7
N2—C3—N4	122.85 (17)	C35—C36—C31	120.26 (17)
N2—C3—C31	117.77 (16)	C35—C36—H36	119.9
N4—C3—C31	119.38 (16)	C31—C36—H36	119.9
C32—C31—C36	118.96 (17)	C5—N4—C3	121.07 (15)
C32—C31—C3	121.84 (16)	C5—N4—H4	118.3
C36—C31—C3	119.19 (16)	C3—N4—H4	120.1
C33—C32—C31	120.32 (17)	O5—C5—N4	123.13 (16)
C33—C32—H32	119.8	O5—C5—C6	123.88 (16)
C31—C32—H32	119.8	N4—C5—C6	112.97 (15)
C32—C33—C34	120.48 (18)	O1—C6—C5	110.36 (14)
C32—C33—H33	119.8	O1—C6—H6A	109.6
C34—C33—H33	119.8	C5—C6—H6A	109.6
C35—C34—C33	119.31 (18)	O1—C6—H6B	109.6
C35—C34—H34	120.3	C5—C6—H6B	109.6
C33—C34—H34	120.3	H6A—C6—H6B	108.1
C36—C35—C34	120.67 (17)		

O1—N2—C3—N4	3.4 (2)	C3—C31—C32—C33	179.12 (16)
N2—C3—N4—C5	21.4 (2)	C31—C32—C33—C34	−0.2 (3)
C3—N4—C5—C6	−4.0 (2)	C32—C33—C34—C35	0.3 (3)
N4—C5—C6—O1	−33.9 (2)	C33—C34—C35—C36	−0.1 (3)
C5—C6—O1—N2	58.6 (2)	C34—C35—C36—C31	0.0 (3)
C6—O1—N2—C3	−43.56 (19)	C32—C31—C36—C35	0.1 (3)
O1—N2—C3—C31	−175.50 (13)	C3—C31—C36—C35	−179.03 (16)
N2—C3—C31—C32	−170.97 (16)	C31—C3—N4—C5	−159.71 (15)
N4—C3—C31—C32	10.1 (2)	C3—N4—C5—O5	174.28 (15)
N2—C3—C31—C36	8.2 (2)	N2—O1—C6—C5	58.59 (19)
N4—C3—C31—C36	−170.81 (15)	O5—C5—C6—O1	147.78 (16)
C36—C31—C32—C33	0.0 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···O5^{i}	0.85	2.06	2.886 (2)	165
C32—H32···O5^{i}	0.95	2.39	3.296 (2)	159

C6—H6*A*···N2^{ii}	0.99	2.59	3.475 (2)	148
C36—H36···N2^{iii}	0.95	2.56	3.476 (2)	161

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