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1,3-Bis(prop-2-ynyl)-1*H*-1,3-benzimidazol-2(3*H*)-one

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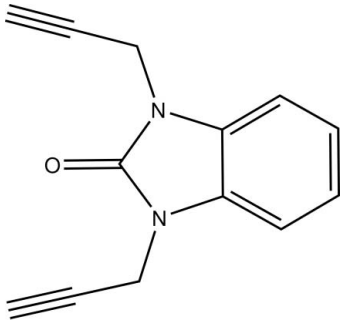
Key indicators: single-crystal X-ray study; T = 170 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.104; data-to-parameter ratio = 18.0.

In the title compound, $C_{13}H_{10}N_2O$, the fused-ring system is essentially planar, the largest deviation from the mean plane being 0.015 (1) Å. The two propynyl groups are nearly perpendicular to the benzimidazole plane, making dihedral angles of 85 (3) and 80 (2) °, and point in opposite directions. There are two short intermolecular C—H···O contacts to the carbonyl O atom, one involving the acetylenic H atom and the other a H atom of the methylene group.

Related literature

For applications of benzimidazole compounds, see: Gravatt *et al.* (1994); Horton *et al.* (2003); Kim *et al.* (1996); Roth *et al.* (1997); Ouzidan *et al.* (2011*a*,*b*).



Experimental

Crystal data

$C_{13}H_{10}N_2O$
M_r = 210.23
Monoclinic, $P2_1/c$
a = 7.7398 (4) Å
b = 17.1869 (9) Å
c = 8.4856 (5) Å
β = 101.459 (6)°
V = 1106.28 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 170 K
0.42 × 0.41 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur E Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
T_{min} = 0.966, T_{max} = 0.984
5295 measured reflections
2631 independent reflections
2244 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.104
S = 1.05
2631 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8A···O1^{i}	0.99	2.42	3.3096 (15)	149
C13—H13···O1^{ii}	0.95	2.34	3.2252 (17)	156

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2361).

References

Gravatt, G. L., Baguley, B. C., Wilson, W. R. & Denny, W. A. (1994). *J. Med. Chem.* **37**, 4338–4345.
Horton, D. A., Bourne, G. T. & Smythe, M. L. (2003). *Chem. Rev.* **103**, 893–930.
Kim, J. S., Gatto, B., Yu, C., Liu, A., Liu, L. F. & La Voie, E. J. (1996). *J. Med. Chem.* **39**, 992–998.
Ouzidan, Y., Kandri Rodi, Y., Butcher, R. J., Essassi, E. M. & El Ammari, L. (2011*a*). *Acta Cryst.* E**67**, o283.
Ouzidan, Y., Kandri Rodi, Y., Fronczek, F. R., Venkatraman, R., El Ammari, L. & Essassi, E. M. (2011*b*). *Acta Cryst.* E**67**, o362–o363.
Oxford Diffraction (2009). *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, England.
Roth, T., Morningstar, M. L., Boyer, P. L., Hughes, S. H., Buckheit, R. W. & Michejda, C. J. (1997). *J. Med. Chem.* **40**, 4199–4207.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2011). E**67**, o1091 [doi:10.1107/S1600536811012578]

1,3-Bis(prop-2-ynyl)-1*H*-1,3-benzimidazol-2(3*H*)-one

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S1. Comment

Benzimidazoles are very useful intermediates/subunits for the development of molecules of pharmaceutical or biological interest. Benzimidazole and its derivatives are an important class of bioactive molecules in the field of drugs and pharmaceuticals.

Benzimidazole derivatives have found applications in diverse therapeutic areas including anti-ulcers, anti-hypertensives, anti-virals, anti-fungals, anti-cancers (Gravatt *et al.*, 1994; Horton *et al.*, 2003; Kim *et al.*, 1996; Roth *et al.*, 1997).

As a continuation of our research works devoted to the development benzimidazol-2-one derivatives (Ouzidan *et al.*, 2011*a*,*b*), we report in this paper the synthesis of a new benzimidazol-2-one derivative prepared by action of propargyl bromide on 1*H*-benzimidazol-2(3*H*)-one in the presence of a catalytic quantity of tetra-n-butylammonium bromide under mild conditions to furnish the title compound (Scheme 1).

In the title compound (Fig. 1), the benzimidazole ring system is essentially planar with a maximum deviation of 0.015 (1) Å for C1 atom. The two propynyl chains are almost perpendicular to the benzimidazole mean plane but oriented one above and one below the plane. The molecular conformation is also characterized by the following torsion angles: C1-N1-C8-C9 = 93.5 (2) ° and C1-N2-C11-C12 = 105.9 (2) °. In the crystal structure, molecules are linked by weak intermolecular C—H···O no classic hydrogen bonds as shown in Fig. 2 and Table 2.

S2. Experimental

To a mixture of 1*H*-benzimidazol-2(3*H*)-one (0.2 g, 1.5 mmol), potassium carbonate (0.45 g, 3.2 mmol), tetra-n-butyl-ammonium bromide (0.1 g, 0.2 mmol) in DMF (15 ml) was added propargyl bromide (0.28 ml, 3.2 mmol). Stirring was continued at room temperature for 6 h. The salt was removed by filtration and the filtrate concentrated under reduced pressure. The product was purified by recrystallization from dichloromethane to give colourless crystals (m.p. 425 K).

S3. Refinement

H atoms were located in a difference map and treated as riding with C—H = 0.95 Å or 0.99 Å with U_{iso}(H) = 1.2 U_{eq} (C).

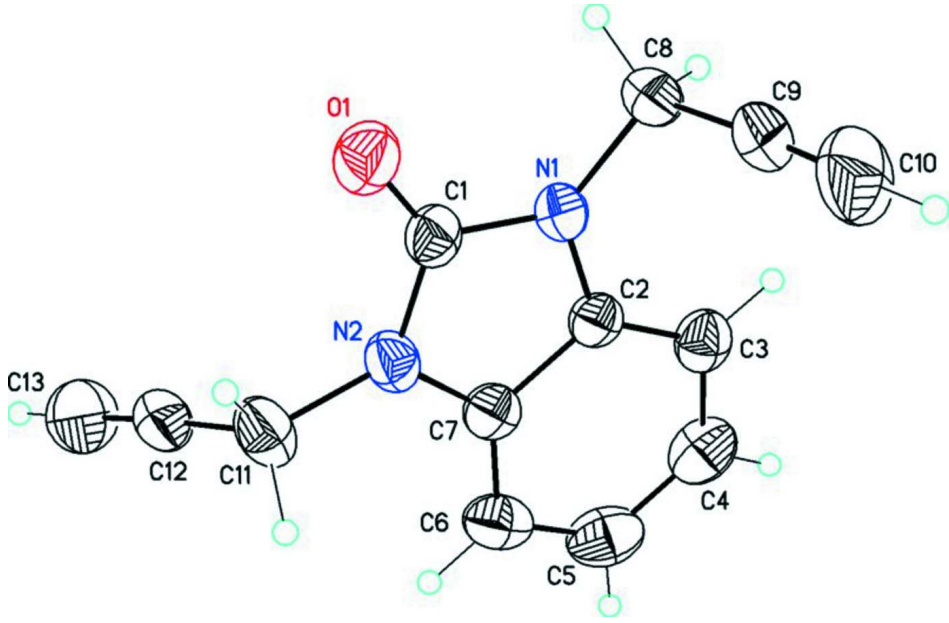

Figure 1

Molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are represented as small circles.

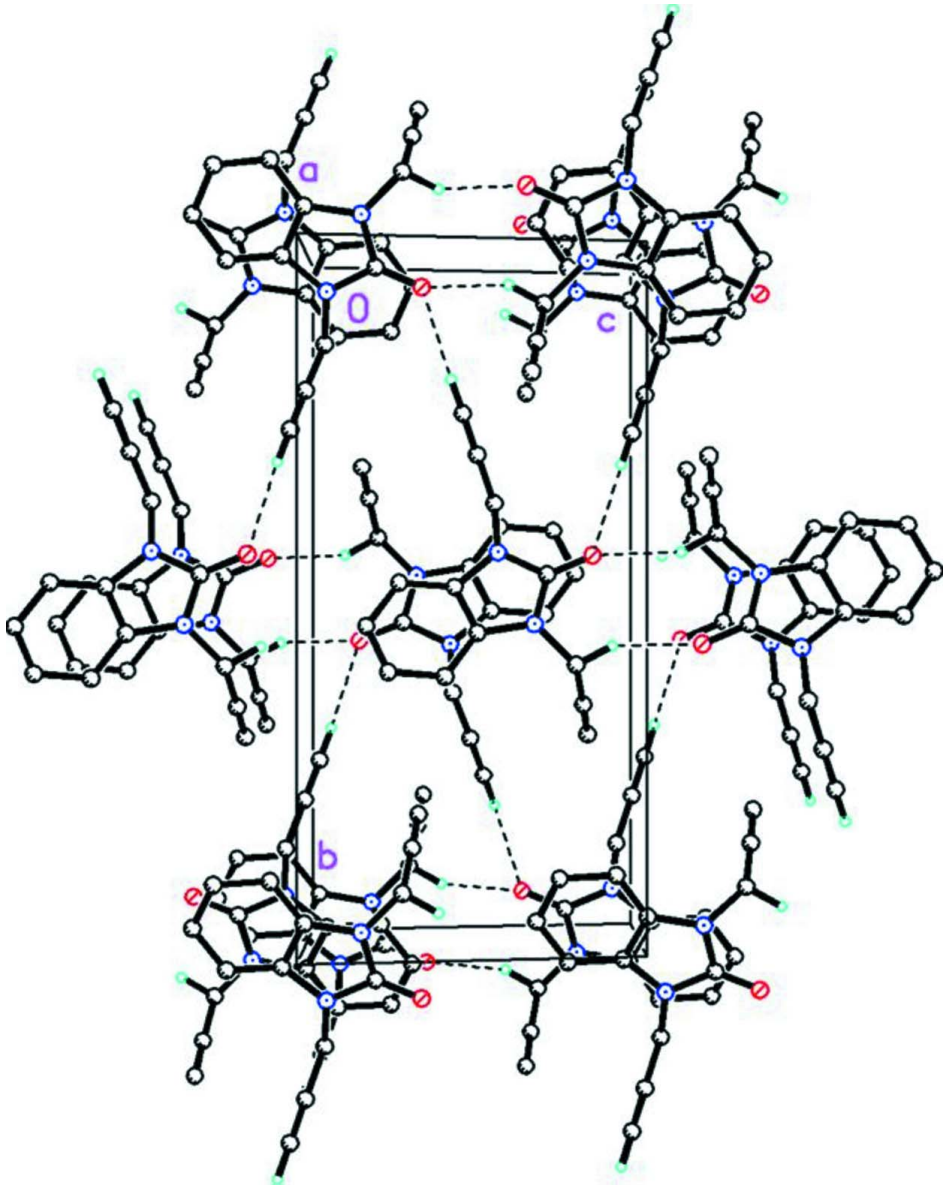

Figure 2

Partial packing view showing the C—H···O interactions (dashed lines).

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Crystal data

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$M_r = 210.23$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.7398$ (4) Å
$b = 17.1869$ (9) Å
$c = 8.4856$ (5) Å
$\beta = 101.459$ (6)°
$V = 1106.28$ (10) Å^3
$Z = 4$

$F(000) = 440$
$D_x = 1.262$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3141 reflections
$\theta = 3.4$–32.2°
$\mu = 0.08$ mm^{-1}
$T = 170$ K
Block, colorless
0.42 × 0.41 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur E Gemini diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1500 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
T_{min} = 0.966, T_{max} = 0.984
5295 measured reflections
2631 independent reflections
2244 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 27.9°, θ_{min} = 3.4°
h = −10→10
k = −20→22
l = −11→4

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.104
S = 1.05
2631 reflections
146 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
$w = 1/[\sigma^2(F_o^2) + (0.0447P)^2 + 0.2172P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.042 (4)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.19931 (12)	0.56190 (5)	0.15005 (11)	0.0468 (2)
N1	0.13961 (12)	0.46462 (5)	0.32281 (11)	0.0338 (2)
N2	0.29126 (12)	0.56670 (5)	0.42832 (12)	0.0356 (2)
C1	0.20862 (14)	0.53446 (6)	0.28398 (14)	0.0346 (3)
C2	0.18290 (13)	0.45232 (6)	0.48810 (13)	0.0319 (2)
C3	0.14722 (15)	0.39128 (7)	0.58201 (15)	0.0400 (3)
H3A	0.0826	0.3470	0.5362	0.048*
C4	0.21003 (18)	0.39733 (9)	0.74692 (16)	0.0493 (3)
H4A	0.1871	0.3565	0.8153	0.059*
C5	0.30518 (18)	0.46160 (9)	0.81362 (16)	0.0524 (4)
H5A	0.3463	0.4638	0.9267	0.063*
C6	0.34172 (16)	0.52287 (8)	0.71876 (15)	0.0447 (3)
H6A	0.4073	0.5669	0.7646	0.054*
C7	0.27878 (14)	0.51721 (6)	0.55512 (14)	0.0337 (3)

C8	0.06303 (16)	0.40760 (7)	0.20295 (15)	0.0398 (3)
H8A	0.0159	0.4343	0.1000	0.048*
H8B	−0.0362	0.3810	0.2383	0.048*
C9	0.19408 (17)	0.34996 (7)	0.17828 (16)	0.0438 (3)
C10	0.3035 (2)	0.30587 (10)	0.1615 (2)	0.0748 (5)
H10	0.3927	0.2699	0.1479	0.090*
C11	0.39412 (16)	0.63759 (7)	0.43751 (18)	0.0443 (3)
H11A	0.4208	0.6484	0.3302	0.053*
H11B	0.5076	0.6293	0.5131	0.053*
C12	0.30770 (17)	0.70582 (7)	0.48968 (16)	0.0454 (3)
C13	0.2472 (2)	0.76260 (9)	0.5312 (2)	0.0678 (5)
H13	0.1980	0.8088	0.5649	0.081*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0561 (5)	0.0414 (5)	0.0426 (5)	0.0004 (4)	0.0090 (4)	0.0060 (4)
N1	0.0375 (5)	0.0275 (4)	0.0351 (5)	0.0017 (4)	0.0043 (4)	−0.0037 (4)
N2	0.0349 (5)	0.0290 (5)	0.0430 (5)	−0.0006 (4)	0.0080 (4)	−0.0051 (4)
C1	0.0339 (5)	0.0297 (5)	0.0405 (6)	0.0054 (4)	0.0079 (5)	−0.0013 (4)
C2	0.0276 (5)	0.0317 (5)	0.0363 (6)	0.0059 (4)	0.0060 (4)	−0.0026 (4)
C3	0.0359 (6)	0.0374 (6)	0.0481 (7)	0.0031 (5)	0.0118 (5)	0.0028 (5)
C4	0.0470 (7)	0.0577 (8)	0.0457 (7)	0.0070 (6)	0.0150 (6)	0.0114 (6)
C5	0.0489 (7)	0.0727 (9)	0.0351 (6)	0.0086 (7)	0.0068 (5)	0.0004 (6)
C6	0.0367 (6)	0.0538 (7)	0.0419 (7)	0.0025 (5)	0.0034 (5)	−0.0114 (6)
C7	0.0278 (5)	0.0348 (5)	0.0390 (6)	0.0055 (4)	0.0077 (4)	−0.0044 (4)
C8	0.0396 (6)	0.0349 (6)	0.0413 (6)	−0.0008 (5)	−0.0003 (5)	−0.0066 (5)
C9	0.0525 (7)	0.0328 (6)	0.0440 (7)	−0.0025 (5)	0.0043 (6)	−0.0082 (5)
C10	0.0733 (11)	0.0515 (9)	0.0976 (14)	0.0171 (8)	0.0124 (10)	−0.0250 (9)
C11	0.0358 (6)	0.0355 (6)	0.0628 (8)	−0.0057 (5)	0.0128 (6)	−0.0079 (6)
C12	0.0447 (7)	0.0340 (6)	0.0536 (8)	−0.0026 (5)	0.0002 (6)	−0.0054 (5)
C13	0.0771 (11)	0.0436 (8)	0.0749 (11)	0.0136 (7)	−0.0038 (9)	−0.0170 (7)

Geometric parameters (Å, º)

O1—C1	1.2194 (14)	C5—H5A	0.9500
N1—C1	1.3800 (14)	C6—C7	1.3806 (17)
N1—C2	1.3919 (14)	C6—H6A	0.9500
N1—C8	1.4515 (14)	C8—C9	1.4622 (17)
N2—C1	1.3802 (15)	C8—H8A	0.9900
N2—C7	1.3899 (15)	C8—H8B	0.9900
N2—C11	1.4491 (14)	C9—C10	1.166 (2)
C2—C3	1.3782 (16)	C10—H10	0.9500
C2—C7	1.3965 (15)	C11—C12	1.4615 (17)
C3—C4	1.3911 (19)	C11—H11A	0.9900
C3—H3A	0.9500	C11—H11B	0.9900
C4—C5	1.385 (2)	C12—C13	1.1665 (19)
C4—H4A	0.9500	C13—H13	0.9500

C5—C6	1.388 (2)		
C1—N1—C2	110.22 (9)	C7—C6—H6A	121.4
C1—N1—C8	122.92 (10)	C5—C6—H6A	121.4
C2—N1—C8	125.91 (9)	C6—C7—N2	132.11 (11)
C1—N2—C7	110.49 (9)	C6—C7—C2	121.24 (11)
C1—N2—C11	122.56 (10)	N2—C7—C2	106.65 (10)
C7—N2—C11	126.50 (10)	N1—C8—C9	111.07 (10)
O1—C1—N1	127.23 (11)	N1—C8—H8A	109.4
O1—C1—N2	127.08 (11)	C9—C8—H8A	109.4
N1—C1—N2	105.69 (10)	N1—C8—H8B	109.4
C3—C2—N1	131.49 (11)	C9—C8—H8B	109.4
C3—C2—C7	121.59 (11)	H8A—C8—H8B	108.0
N1—C2—C7	106.92 (9)	C10—C9—C8	177.43 (15)
C2—C3—C4	117.04 (12)	C9—C10—H10	180.0
C2—C3—H3A	121.5	N2—C11—C12	114.29 (10)
C4—C3—H3A	121.5	N2—C11—H11A	108.7
C5—C4—C3	121.47 (12)	C12—C11—H11A	108.7
C5—C4—H4A	119.3	N2—C11—H11B	108.7
C3—C4—H4A	119.3	C12—C11—H11B	108.7
C4—C5—C6	121.41 (13)	H11A—C11—H11B	107.6
C4—C5—H5A	119.3	C13—C12—C11	176.21 (15)
C6—C5—H5A	119.3	C12—C13—H13	180.0
C7—C6—C5	117.25 (12)		
C2—N1—C1—O1	177.95 (11)	C4—C5—C6—C7	0.21 (19)
C8—N1—C1—O1	8.50 (18)	C5—C6—C7—N2	−179.89 (11)
C2—N1—C1—N2	−1.76 (11)	C5—C6—C7—C2	−0.23 (17)
C8—N1—C1—N2	−171.22 (9)	C1—N2—C7—C6	178.83 (11)
C7—N2—C1—O1	−178.10 (11)	C11—N2—C7—C6	6.44 (19)
C11—N2—C1—O1	−5.36 (18)	C1—N2—C7—C2	−0.87 (12)
C7—N2—C1—N1	1.61 (12)	C11—N2—C7—C2	−173.26 (10)
C11—N2—C1—N1	174.35 (9)	C3—C2—C7—C6	−0.08 (16)
C1—N1—C2—C3	−178.62 (11)	N1—C2—C7—C6	−179.97 (10)
C8—N1—C2—C3	−9.55 (18)	C3—C2—C7—N2	179.66 (10)
C1—N1—C2—C7	1.26 (11)	N1—C2—C7—N2	−0.23 (11)
C8—N1—C2—C7	170.33 (10)	C1—N1—C8—C9	93.46 (13)
N1—C2—C3—C4	−179.74 (11)	C2—N1—C8—C9	−74.31 (14)
C7—C2—C3—C4	0.40 (16)	C1—N2—C11—C12	105.88 (13)
C2—C3—C4—C5	−0.42 (18)	C7—N2—C11—C12	−82.58 (15)
C3—C4—C5—C6	0.1 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···O1^{i}	0.99	2.42	3.3096 (15)	149

C13—H13···O1^{ii}	0.95	2.34	3.2252 (17)	156

Symmetry codes: (i) $-x$, $-y+1$, $-z$; (ii) x, $-y+3/2$, $z+1/2$.