4-Phenyl-1-(prop-2-yn-1-yl)-1H-1,5-benzodiazepin-2(3H)-one

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4-Phenyl-1H-1,5-benzodiazepin-2(3H)-one reacts in the presence of a concentrated aqueous solution of sodium hydroxide and a quaternary ammonium salt (as catalyst) in benzene (phase transfer catalysis) with propargyl bromide, affording the title benzodiazepine derivative, $C_{18}H_{14}N_2O$. In the molecule, the mean plane of the propargyl substituent is almost perpendicular with that of the amide group [dihedral angle = 87.81 (8)°]. In the crystal, the molecules are linked by C—H⋯O and C—H⋯N interactions.

Related literature

For general background to applications of benzodiazepines, see: Ahmed *et al.* (1983); Bird (1996); Di Braccio *et al.* (1990, 2001); Goetzke *et al.* (1983); Kavita *et al.* (1988); Sieghart & Schuster (1984); Wolff (1996). For examples of benzodiazepines used as medicine, see: Wolff (1996). For the pharmacological effects of benzodiazepines, see: Meldrum & Chapman (1986). For examples of synthetic pathways of new benzodiazepines, see: Aatif *et al.* (2000); Baouid *et al.* (2001); Boudina *et al.* (2007); Nardi *et al.* (1973). For previous work from our groups on organic crystals, see: Fernandes *et al.* (2011); Amarante, Figueiredo *et al.* (2009); Amarante, Gonçalves & Almeida Paz (2009); Paz & Klinowski (2003); Paz *et al.* (2002).
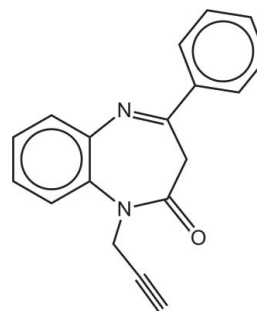
Experimental

Crystal data

$C_{18}H_{14}N_2O$	$V = 1387.1$ (4) Å^3
$M_r = 274.31$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 8.2574$ (14) Å	$\mu = 0.08$ mm^{-1}
$b = 18.961$ (3) Å	$T = 150$ K
$c = 9.0914$ (15) Å	$0.12 \times 0.08 \times 0.04$ mm
$\beta = 102.962$ (4)°	

Data collection

Bruker X8 Kappa CCD APEX II diffractometer	11049 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)	5228 independent reflections
$T_{min} = 0.990$, $T_{max} = 0.997$	3621 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$	190 parameters
$wR(F^2) = 0.143$	H-atom parameters constrained
$S = 1.05$	$\Delta\rho_{max} = 0.45$ e Å^{-3}
5228 reflections	$\Delta\rho_{min} = -0.25$ e Å^{-3}

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1A⋯O1^i	0.99	2.14	3.1074 (15)	166
C3—H3⋯N2^{ii}	0.95	2.58	3.4269 (18)	149

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT-Plus* (Bruker, 2005); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 2009); software used to prepare material for publication: *SHELXTL*.

organic compounds

References

Aatif, A., Baouid, A., Hasnaoui, A. & Pierrot, M. (2000). *Acta Cryst.* C**56**, e459–e460.

Ahmed, F., Rittmeyer, G., Goetzke, E. & Koster, J. (1983). *Brit. J. Clin. Pharmacol.* **16**, 419S–423S.

Amarante, T. R., Figueiredo, S., Lopes, A. D., Gonçalves, I. S. & Almeida Paz, F. A. (2009). *Acta Cryst.* E**65**, o2047.

Amarante, T. R., Gonçalves, I. S. & Almeida Paz, F. A. (2009). *Acta Cryst.* E**65**, o1962–o1963.

Baouid, A., Elhazazi, S., Hasnaoui, A., Compain, P., Lavergne, J. P. & Huet, F. (2001). *New J. Chem.* **25**, 1479–1481.

Bird, C. W. (1996). *Comprehensive Heterocyclic Chemistry*. Oxford: Pergamon.

Boudina, A., Baouid, A., Hasnaoui, A., Aatif, A., Eddike, D. & Tillard, M. (2007). *Acta Cryst.* E**63**, o1544–o1545.

Brandenburg, K. (2009). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.

Bruker (2005). *SAINT-Plus*. Bruker AXS, Inc. Madison, Wisconsin, USA.

Bruker (2006). *APEX2*. Bruker AXS, Delft, The Netherlands.

Di Braccio, M., Grossi, G., Roma, G., Vargiu, L., Mura, M. & Marongiu, M. E. (2001). *Eur. J. Med. Chem.* **36**, 935–949.

Di Braccio, M., Roma, G., Grossi, G. C., Ghima, M. & Mereto, E. (1990). *Eur. J. Med. Chem.* **25**, 681–687.

Fernandes, J. A., Almeida Paz, F. A., Marques, J., Marques, M. P. M. & Braga, S. S. (2011). *Acta Cryst.* C**67**, o57–o59.

Goetzke, E., Findeisen, P., Welbers, I. B. & Koster, J. (1983). *Brit. J. Clin. Pharmacol.* **16**, 397S–402S.

Kavita, D. T., Achaiah, G. & Reddy, V. M. (1988). *J. Indian Chem. Soc.* **65**, 567–570.

Meldrum, B. S. & Chapman, A. G. (1986). *Epilepsia*, **27 (suppl. 1)**, S3–S13.

Nardi, D., Tajana, A. & Rossi, S. (1973). *J. Heterocycl. Chem.* **10**, 815–819.

Paz, F. A. A., Bond, A. D., Khimyak, Y. Z. & Klinowski, J. (2002). *New J. Chem.* **26**, 381–383.

Paz, F. A. A. & Klinowski, J. (2003). *CrystEngComm* **5**, 238–244.

Sheldrick, G. M. (1997). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sieghart, W. & Schuster, A. (1984). *Biochem. Pharmacol.* **33**, 4033–4038.

Wolff, M. E. (1996). *Burger's Medicinal Chemistry and Drug Discovery*. 5th ed. New York: John Wiley & Sons.

supporting information

4-Phenyl-1-(prop-2-yn-1-yl)-1*H*-1,5-benzodiazepin-2(3*H*)-one

Mohamed Loughzail, José A. Fernandes, Abdesselam Baouid, Mohamed Essaber, José A. S. Cavaleiro and Filipe A. Almeida Paz

S1. Comment

Benzodiazepine derivatives are an important class of heterocyclic compounds in the field of drugs, pharmaceuticals and synthetic organic chemistry (Bird, 1996; Wolff, 1996), as they show antiviral (Di Braccio *et al.*, 2001), analgesic (Di Braccio *et al.*, 1990), and antipsychotic (Kavita *et al.*, 1988) activities. These compounds are used worldwide as anticonvulsant agents (Sieghart & Schuster, 1984) or as sedative or hypnotics (Goetzke *et al.*, 1983; Ahmed *et al.*, 1983). Examples of well known diazepines are Alprazolam, Diazepam and Flunitrazepam (Wolff, 1996). Their pharmacological effects come from the activation of the benzodiazepine receptor which interacts with the GABA recognition site (Meldrum & Chapman, 1986). Research in this area is highly active being directed towards the synthesis of compounds with enhanced pharmacological activity. Following the research efforts from some of us concerning novel synthetic pathways of new benzodiazepines (Aatif *et al.*, 2000; Baouid *et al.*, 2001; Boudina *et al.*, 2007), and our interest on the structural features of organic crystals (Fernandes *et al.*, 2011; Amarante, Figueiredo *et al.*, 2009; Amarante, Gonçalves & Almeida Paz , 2009; Paz & Klinowski, 2003; Paz *et al.*, 2002), here we wish to report the synthesis *via* phase transfer catalysis and the crystallographic studies of the title compound (I).

The asymmetric unit is composed of a whole molecular moiety of I (Fig. 1). All atoms are distributed over four medium planes (see Table 1 for details), which converge in the diazepine ring. The plane of the substituent aromatic ring is extended to the imine group from the diazepine moiety (plane A) and subtends an angle of 71.78 (4)° with the amide plane (C). The plane of the benzo ring (B) subtends, on the other hand, two almost similar angles with the previously described planes [41.76 (4)° with plane A and 40.75 (4)° with plane C]. The plane of the propargyl substituent (D) is almost perpendicular with that of the amide group [87.81 (8)°].

The crystal packing (Fig. 2) features weak supramolecular interactions (see Table 2 for details), namely the C—H and CH$_2$ groups of the propargyl moiety interact with N2 from the imine and O1 from the amide of neighbouring molecules, respectively.

S2. Experimental

Melting points were taken in an open capillary tube on a Buchi 510 apparatus and are uncorrected. The FT—IR spectrum was obtained from KBr pellets using a Bruker Tensor 27 spectrophotometer. NMR Spectra were recorded with the following instruments: ^{1}H, Bruker AC-300; ^{13}C, Bruker AC-75. TMS was used as an internal reference. Mass spectra were recorded using a Jeol JMS DX 300 instrument. Column chromatography was carried out using E-Merck silica gel 60F$_{254}$. All reagents were purchased from commercial sources and were used without further purification.

The precursor, 4-phenyl-2,3-dihydro-1*H*-1,5-benzodiazepin-2-one (II), was prepared following literature procedures (Nardi *et al.*, 1973) by refluxing *o*-phenylenediamine and ethyl benzoylacetate for 2 h in xylene.

A mixture of 1 g (4.6 mmol) of II, 0.43 g (2.3 mmol) of benzyltriethylammonium chloride (TBA-Cl) and 3 ml of a 50% sodium hydroxide aqueous solution in benzene (25 ml) was stirred at ambient temperature. After 15 min, propargyl bromide was added slowly. After 6 h of stirring at 298 K, the reaction mixture was diluted with water (30 ml). The organic layer was extracted with benzene (3 × 10 ml), dried over anhydrous sodium sulfate and evaporated under vacuum. The title compound was isolated by column chromatography on silica gel using hexane/ethyl acetate as eluent. The solid product was recrystallized in dichloromethane to give yellow crystals of I. Yield: 96%. Melting point: 438–440 K.

FT–IR (KBr): 3259(m), 3060(w), 2984(m), 1659(vs), 1602(s), 1586(w), 1570(m), 1496(w), 1479(s), 1452(s), 1431(m), 1379(s), 1362(w), 1321(w), 1307(m), 1293(w), 1279(m), 1262(m), 1211(m), 1162(w), 1014(m), 958(m), 774(s), 688(m), 662(w), 639(w), 598(m), 484(w), 426(w) cm^{-1}. ^{1}H NMR (300 MHz, CDCl$_3$): 7.25-8.14 (9H, Ar-H), 4.19 and 4.27 (AB system, d, J= 17.7 Hz, 2H, N-CH$_2$-C), 3.04 and 4.76 (AB system, d, J=12 Hz, 2H, CH$_2$-CO-N), 2.32 (t, J= 2.25 Hz, 1H, HC≡C) ppm. ^{13}C NMR (75 MHz, CDCl$_3$): 165 (1C, **C**O), 160.0 (1C, Ph-**C**=N), 140.9, 136.9, 133.3, 130.5, 128.1, 127.1, 126.7, 125.7, 125.1, 120.9 (12C, Ar-**C**), 78.5 (1C, H**C**≡ C), 71.9 (1C, HC≡**C**), 39.1 (1C, **C**H$_2$-CO-N), 36.9 (1C, N-**C**H$_2$-C) ppm. MS (EI, m/z): 275 [M+H]$^+$.

S3. Refinement

Hydrogen atoms bound to carbon were placed at their idealized positions and were included in the final structural model in riding-motion approximation with C—H = 0.95 Å (aromatic and acetylenic), and C—H = 0.99 Å (aliphatic —CH$_2$—). The isotropic thermal displacement parameters for these atoms were fixed at $1.2×U_{eq}$ of the respective parent carbon atom.
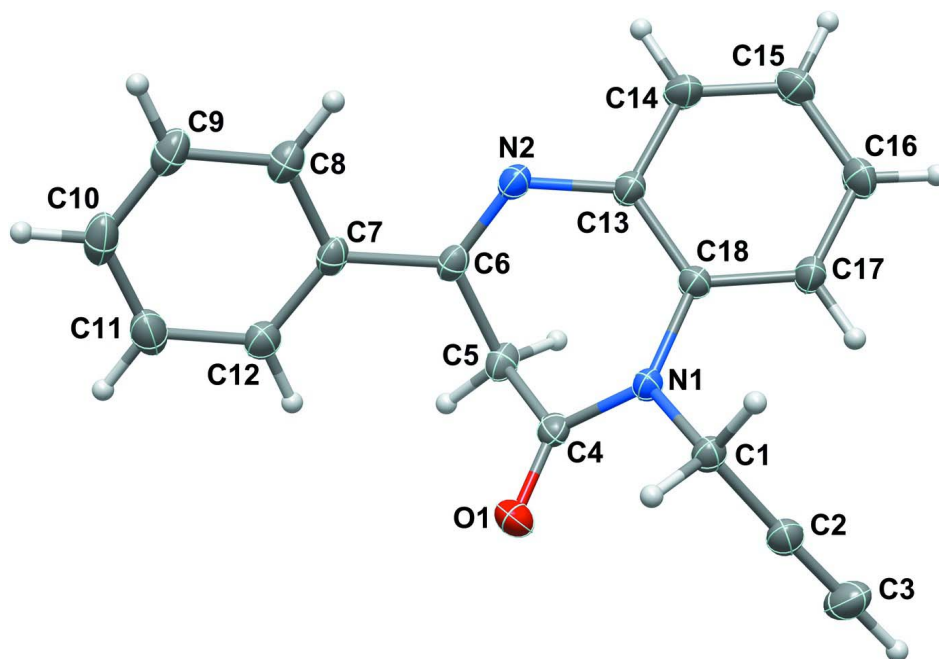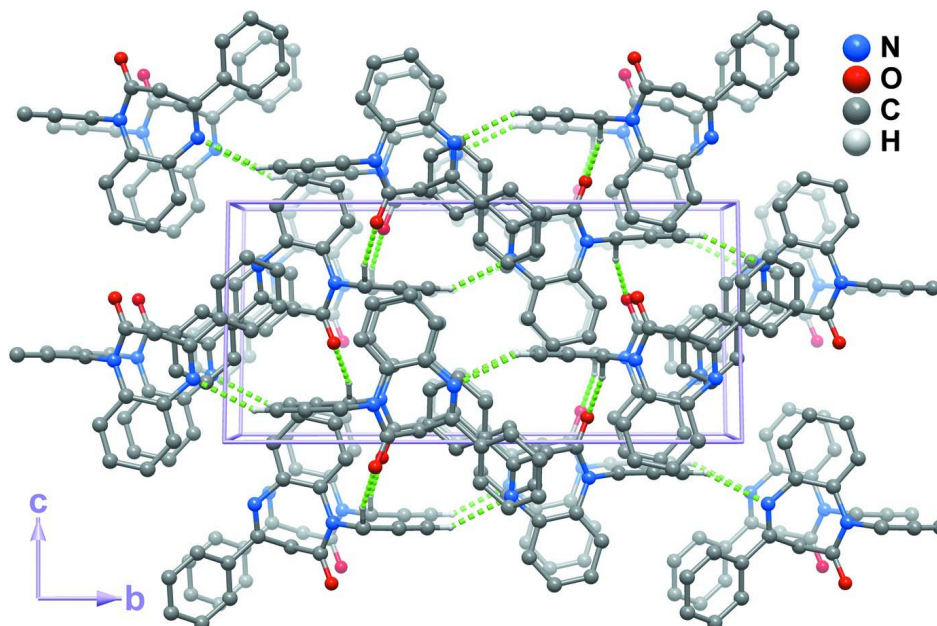

Figure 1

Asymmetric unit of the title compound showing all non-hydrogen atoms represented as thermal ellipsoids drawn at the 50% probability level and hydrogen atoms as small spheres with arbitrary radii.

Figure 2

Crystal packing of the title compound viewed in perspective along the [100] direction of the unit cell. C—H⋯(N,O) weak hydrogen bonds are represented as dashed green lines. H-atoms not involved in hydrogen bonding interactions have been omitted for clarity.

4-Phenyl-1-(prop-2-yn-1-yl)-1*H***-1,5-benzodiazepin-2(3***H***)-one**

Crystal data

C$_{18}$H$_{14}$N$_2$O

$M_r = 274.31$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 8.2574$ (14) Å

$b = 18.961$ (3) Å

$c = 9.0914$ (15) Å

$\beta = 102.962$ (4)°

$V = 1387.1$ (4) Å^3

$Z = 4$

$F(000) = 576$

$D_x = 1.314$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2335 reflections

$\theta = 2.5$–32.7°

$\mu = 0.08$ mm^{-1}

$T = 150$ K

Block, yellow

$0.12 \times 0.08 \times 0.04$ mm

Data collection

Bruker X8 Kappa CCD APEX II
   diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω / φ scans

Absorption correction: multi-scan
   (*SADABS*; Sheldrick, 1997)

$T_{min} = 0.990$, $T_{max} = 0.997$

11049 measured reflections

5228 independent reflections

3621 reflections with $I > 2\sigma(I)$

$R_{int} = 0.034$

$\theta_{max} = 33.1°$, $\theta_{min} = 3.7°$

$h = -12 \rightarrow 11$

$k = -24 \rightarrow 29$

$l = -10 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier
Least-squares matrix: full	map
$R[F^2 > 2\sigma(F^2)] = 0.052$	Hydrogen site location: inferred from
$wR(F^2) = 0.143$	neighbouring sites
$S = 1.05$	H-atom parameters constrained
5228 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0689P)^2 + 0.095P]$
190 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant	$\Delta\rho_{max} = 0.45$ e Å^{-3}
direct methods	$\Delta\rho_{min} = -0.25$ e Å^{-3}

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}
N1	0.81747 (12)	0.20787 (5)	0.66080 (10)	0.01806 (19)
N2	0.80660 (13)	0.05512 (5)	0.73647 (10)	0.0212 (2)
O1	0.73372 (11)	0.20477 (5)	0.40627 (9)	0.0263 (2)
C1	0.92473 (14)	0.26835 (6)	0.64614 (12)	0.0203 (2)
H1A	1.0213	0.2687	0.7333	0.024*
H1B	0.9675	0.2624	0.5535	0.024*
C2	0.83874 (16)	0.33625 (6)	0.63915 (12)	0.0247 (2)
C3	0.7724 (2)	0.39109 (7)	0.63764 (16)	0.0338 (3)
H3	0.7187	0.4355	0.6364	0.041*
C4	0.72159 (14)	0.18209 (6)	0.52947 (11)	0.0191 (2)
C5	0.60888 (14)	0.12224 (6)	0.54917 (13)	0.0218 (2)
H5A	0.5294	0.1118	0.4527	0.026*
H5B	0.5453	0.1343	0.6260	0.026*
C6	0.72038 (14)	0.05952 (6)	0.59981 (12)	0.0197 (2)
C7	0.73684 (14)	0.00310 (6)	0.49079 (12)	0.0203 (2)
C8	0.84549 (16)	−0.05308 (6)	0.53927 (14)	0.0250 (2)
H8	0.9068	−0.0547	0.6410	0.030*
C9	0.86457 (17)	−0.10638 (7)	0.44028 (15)	0.0289 (3)
H9	0.9375	−0.1446	0.4751	0.035*
C10	0.77807 (19)	−0.10440 (7)	0.29068 (15)	0.0318 (3)
H10	0.7916	−0.1410	0.2231	0.038*
C11	0.67212 (19)	−0.04871 (7)	0.24087 (14)	0.0318 (3)
H11	0.6135	−0.0468	0.1383	0.038*
C12	0.65061 (17)	0.00474 (7)	0.34008 (13)	0.0263 (3)
H12	0.5767	0.0426	0.3048	0.032*

C13	0.80668 (15)	0.11000 (6)	0.83976 (12)	0.0206 (2)
C14	0.81461 (17)	0.09009 (6)	0.99013 (13)	0.0265 (3)
H14	0.8139	0.0414	1.0143	0.032*
C15	0.82341 (17)	0.13908 (7)	1.10369 (13)	0.0285 (3)
H15	0.8261	0.1241	1.2039	0.034*
C16	0.82834 (16)	0.21074 (7)	1.07035 (13)	0.0259 (2)
H16	0.8353	0.2449	1.1480	0.031*
C17	0.82299 (15)	0.23177 (6)	0.92394 (12)	0.0221 (2)
H17	0.8267	0.2806	0.9020	0.026*
C18	0.81219 (14)	0.18262 (6)	0.80699 (11)	0.0187 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0243 (5)	0.0140 (4)	0.0163 (4)	−0.0031 (4)	0.0055 (3)	−0.0011 (3)
N2	0.0281 (5)	0.0145 (4)	0.0223 (4)	0.0000 (4)	0.0083 (4)	−0.0004 (3)
O1	0.0319 (5)	0.0288 (5)	0.0172 (3)	−0.0007 (4)	0.0036 (3)	0.0012 (3)
C1	0.0248 (5)	0.0163 (5)	0.0207 (4)	−0.0036 (4)	0.0071 (4)	−0.0004 (4)
C2	0.0341 (6)	0.0196 (5)	0.0213 (5)	−0.0056 (5)	0.0083 (4)	0.0002 (4)
C3	0.0461 (8)	0.0222 (6)	0.0355 (6)	0.0045 (6)	0.0145 (6)	0.0060 (5)
C4	0.0209 (5)	0.0165 (5)	0.0192 (4)	0.0034 (4)	0.0033 (4)	−0.0012 (4)
C5	0.0213 (5)	0.0176 (5)	0.0258 (5)	−0.0010 (4)	0.0038 (4)	−0.0033 (4)
C6	0.0227 (5)	0.0138 (5)	0.0241 (5)	−0.0023 (4)	0.0082 (4)	−0.0015 (4)
C7	0.0241 (5)	0.0146 (5)	0.0242 (5)	−0.0042 (4)	0.0092 (4)	−0.0029 (4)
C8	0.0265 (6)	0.0189 (5)	0.0310 (5)	0.0004 (4)	0.0094 (4)	−0.0032 (5)
C9	0.0323 (6)	0.0204 (6)	0.0379 (6)	0.0021 (5)	0.0162 (5)	−0.0042 (5)
C10	0.0422 (8)	0.0240 (6)	0.0355 (6)	−0.0056 (6)	0.0224 (6)	−0.0095 (5)
C11	0.0438 (8)	0.0296 (7)	0.0243 (5)	−0.0063 (6)	0.0126 (5)	−0.0053 (5)
C12	0.0350 (7)	0.0198 (5)	0.0252 (5)	−0.0019 (5)	0.0088 (5)	−0.0005 (4)
C13	0.0263 (5)	0.0158 (5)	0.0208 (4)	0.0005 (4)	0.0074 (4)	−0.0009 (4)
C14	0.0387 (7)	0.0197 (5)	0.0229 (5)	0.0008 (5)	0.0105 (5)	0.0036 (4)
C15	0.0397 (7)	0.0290 (6)	0.0190 (5)	0.0015 (5)	0.0110 (4)	0.0023 (5)
C16	0.0346 (6)	0.0254 (6)	0.0194 (4)	0.0000 (5)	0.0099 (4)	−0.0038 (4)
C17	0.0299 (6)	0.0170 (5)	0.0204 (4)	−0.0007 (4)	0.0078 (4)	−0.0025 (4)
C18	0.0224 (5)	0.0174 (5)	0.0169 (4)	−0.0002 (4)	0.0060 (4)	0.0001 (4)

Geometric parameters (Å, º)

N1—C4	1.3665 (13)	C8—H8	0.9500
N1—C18	1.4226 (13)	C9—C10	1.388 (2)
N1—C1	1.4731 (14)	C9—H9	0.9500
N2—C6	1.2886 (14)	C10—C11	1.381 (2)
N2—C13	1.4017 (14)	C10—H10	0.9500
O1—C4	1.2246 (13)	C11—C12	1.3942 (17)
C1—C2	1.4648 (17)	C11—H11	0.9500
C1—H1A	0.9900	C12—H12	0.9500
C1—H1B	0.9900	C13—C14	1.4056 (16)
C2—C3	1.1739 (19)	C13—C18	1.4115 (16)

C3—H3	0.9500	C14—C15	1.3785 (17)
C4—C5	1.5037 (16)	C14—H14	0.9500
C5—C6	1.5112 (16)	C15—C16	1.3947 (18)
C5—H5A	0.9900	C15—H15	0.9500
C5—H5B	0.9900	C16—C17	1.3807 (16)
C6—C7	1.4851 (15)	C16—H16	0.9500
C7—C12	1.3956 (16)	C17—C18	1.4015 (15)
C7—C8	1.3988 (17)	C17—H17	0.9500
C8—C9	1.3855 (17)		
C4—N1—C18	124.24 (10)	C8—C9—C10	120.56 (12)
C4—N1—C1	116.21 (9)	C8—C9—H9	119.7
C18—N1—C1	119.45 (9)	C10—C9—H9	119.7
C6—N2—C13	120.99 (10)	C11—C10—C9	119.40 (12)
C2—C1—N1	113.12 (10)	C11—C10—H10	120.3
C2—C1—H1A	109.0	C9—C10—H10	120.3
N1—C1—H1A	109.0	C10—C11—C12	120.45 (12)
C2—C1—H1B	109.0	C10—C11—H11	119.8
N1—C1—H1B	109.0	C12—C11—H11	119.8
H1A—C1—H1B	107.8	C11—C12—C7	120.53 (12)
C3—C2—C1	178.08 (13)	C11—C12—H12	119.7
C2—C3—H3	180.0	C7—C12—H12	119.7
O1—C4—N1	121.55 (11)	N2—C13—C14	116.43 (10)
O1—C4—C5	123.62 (10)	N2—C13—C18	125.31 (10)
N1—C4—C5	114.75 (9)	C14—C13—C18	118.07 (10)
C4—C5—C6	106.21 (9)	C15—C14—C13	122.04 (11)
C4—C5—H5A	110.5	C15—C14—H14	119.0
C6—C5—H5A	110.5	C13—C14—H14	119.0
C4—C5—H5B	110.5	C14—C15—C16	119.54 (11)
C6—C5—H5B	110.5	C14—C15—H15	120.2
H5A—C5—H5B	108.7	C16—C15—H15	120.2
N2—C6—C7	118.90 (10)	C17—C16—C15	119.64 (11)
N2—C6—C5	120.78 (10)	C17—C16—H16	120.2
C7—C6—C5	120.28 (9)	C15—C16—H16	120.2
C12—C7—C8	118.44 (11)	C16—C17—C18	121.48 (11)
C12—C7—C6	122.43 (11)	C16—C17—H17	119.3
C8—C7—C6	119.11 (10)	C18—C17—H17	119.3
C9—C8—C7	120.60 (12)	C17—C18—C13	119.22 (10)
C9—C8—H8	119.7	C17—C18—N1	118.34 (10)
C7—C8—H8	119.7	C13—C18—N1	122.33 (10)
C4—N1—C1—C2	84.64 (12)	C10—C11—C12—C7	0.6 (2)
C18—N1—C1—C2	−91.95 (12)	C8—C7—C12—C11	0.32 (18)
C18—N1—C4—O1	−178.39 (11)	C6—C7—C12—C11	178.80 (11)
C1—N1—C4—O1	5.21 (16)	C6—N2—C13—C14	144.00 (12)
C18—N1—C4—C5	−1.33 (15)	C6—N2—C13—C18	−41.08 (17)
C1—N1—C4—C5	−177.73 (9)	N2—C13—C14—C15	176.89 (12)
O1—C4—C5—C6	106.80 (12)	C18—C13—C14—C15	1.6 (2)

N1—C4—C5—C6	−70.19 (12)	C13—C14—C15—C16	−1.4 (2)
C13—N2—C6—C7	174.26 (10)	C14—C15—C16—C17	0.5 (2)
C13—N2—C6—C5	−3.54 (17)	C15—C16—C17—C18	0.18 (19)
C4—C5—C6—N2	75.83 (13)	C16—C17—C18—C13	−0.01 (18)
C4—C5—C6—C7	−101.94 (11)	C16—C17—C18—N1	−176.21 (11)
N2—C6—C7—C12	−178.58 (11)	N2—C13—C18—C17	−175.69 (11)
C5—C6—C7—C12	−0.77 (17)	C14—C13—C18—C17	−0.85 (17)
N2—C6—C7—C8	−0.12 (16)	N2—C13—C18—N1	0.36 (18)
C5—C6—C7—C8	177.70 (11)	C14—C13—C18—N1	175.20 (11)
C12—C7—C8—C9	−1.09 (18)	C4—N1—C18—C17	−140.25 (11)
C6—C7—C8—C9	−179.61 (11)	C1—N1—C18—C17	36.05 (15)
C7—C8—C9—C10	0.98 (19)	C4—N1—C18—C13	43.68 (17)
C8—C9—C10—C11	−0.1 (2)	C1—N1—C18—C13	−140.03 (11)
C9—C10—C11—C12	−0.7 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O1[i]	0.99	2.14	3.1074 (15)	166
C3—H3···N2[ii]	0.95	2.58	3.4269 (18)	149

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