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4-(4-Methylphenyl)-2-(prop-2-yn-1-yl)-phthalazin-1(2*H*)-one

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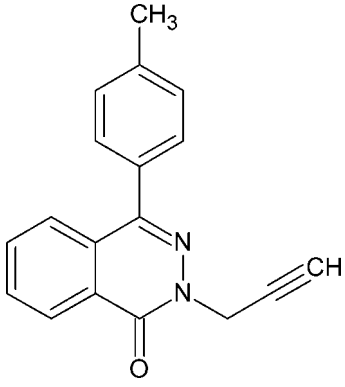
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.044; wR factor = 0.132; data-to-parameter ratio = 12.2.

In the title compound, $C_{18}H_{14}N_2O$, the dihedral angle between the methylphenyl ring and the phthalazone ring system (r.m.s. deviation = 0.034 Å) is 53.93 (9)°. In the crystal, molecules are connected by C—H···O hydrogen bonds, forming chains along [101]. The chains are linked by π–π interactions [centroid–centroid distance 3.6990 (12) Å], forming layers parallel to (10$\overline{1}$).

Related literature

For general background and the biological and pharmacological properties of phthalazine derivatives, see: Abd alla *et al.* (2010); Awadallah *et al.* (2012); Khalil *et al.* (2009); Kim *et al.* (2008); Ryu *et al.* (2007). For a related structure, see: Bausch *et al.* (1997).



Experimental

Crystal data

$C_{18}H_{14}N_2O$
M_r = 274.31
Monoclinic, $P2_1/n$
a = 11.9917 (19) Å
b = 9.7116 (16) Å
c = 12.602 (2) Å
β = 101.285 (7)°
V = 1439.2 (4) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.63 mm^{-1}
T = 296 K
0.23 × 0.20 × 0.19 mm

Data collection

Bruker X8 Proteum diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.864, T_{max} = 0.886
8733 measured reflections
2337 independent reflections
2093 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.132
S = 1.10
2337 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O1^{i}	0.93	2.45	3.322 (2)	157

Symmetry code: (i) $x-\frac{1}{2}, -y-\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5329).

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Acta Cryst. (2014). E**70**, o138 [doi:10.1107/S1600536813034880]

4-(4-Methylphenyl)-2-(prop-2-yn-1-yl)phthalazin-1(2*H*)-one

M. K. Usha, S. Madan Kumar, P. C. Shyma, B. Kalluraya, N. K. Lokanath and D. Revannasiddaiah

S1. Comment

Nitrogen heterocyclic compounds have received a great attention because of their wide applicability in different areas, especially drugs (Kim *et al.*, 2008). Phthalazines are an important class of nitrogen heterocyclic compounds that possess exciting pharmacological and biological properties (Khalil *et al.*, 2009). Phthalazines have been reported to possess antifungal (Ryu *et al.*, 2007), antibacterial (Khalil *et al.*, 2009), cytotoxic (Kim *et al.*, 2008), anti-inflammatory (Abd alla *et al.*, 2010), antihypertensive and vasorelaxant (Awadallah *et al.*, 2012) properties. As part of our studies in this area, herewith we report the structure of the title compound.

The *ORTEP* of the title compound is shown (Fig. 1). The phthalazine ring is nearly planar. The dihedral angle between the methylphenyl ring and the phenyl ring of the phthalazinone moiety is 53.93 (9)°. The bond lengths and bond angles of the title compound are comparable to related structure, 4-(9-fluorenoxy)-2-phenylphthalazin-1(*2H*)-one (Bausch *et al.*, 1997). In the crystal structure, molecules are connected by intermolecular C—H···O hydrogen bonds (Fig. 2). Also, short contacts of the type π–π are observed [minimum centroid-centroid distance 3.6990 (12) Å].

S2. Experimental

2-(4-Methylbenzoyl)benzoic acid (0.1 mol) is esterified in ethanol (25 ml) in presence of few drops of sulfuric acid. The ethyl 2-(4-methylbenzoyl)benzoate obtained is further refluxed with hydrazine hydrate (5 ml, 98%) in absolute ethanol (50 ml)for 2 hrs. Solid obtained on cooling was filtered off and dried to give 4-(4-methylphenyl)phthalazin-1-ol. *A* mixture of 4-(4-methylphenyl)phthalazin-1-ol (0.015 mol), anhydrous potassium carbonate (3.04 g, 0.022 mol) and propargylbromide (1.78 g, 0.015 mol) in DMF (25 ml) was stirred at 65 °C for 2 h. After completion of reaction, reaction mixture was poured into ice-cold water. The solid product obtained was purified by column chromatography using n-hexane and ethyl acetate as eluent to get pure compound. Further the compound was recrystallized from ethyl acetate to get the yellow crystals (m.p. 168–170 °C).

S3. Refinement

The H atoms were placed in calculated positions (C—H = 0.93–0.97 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl}).

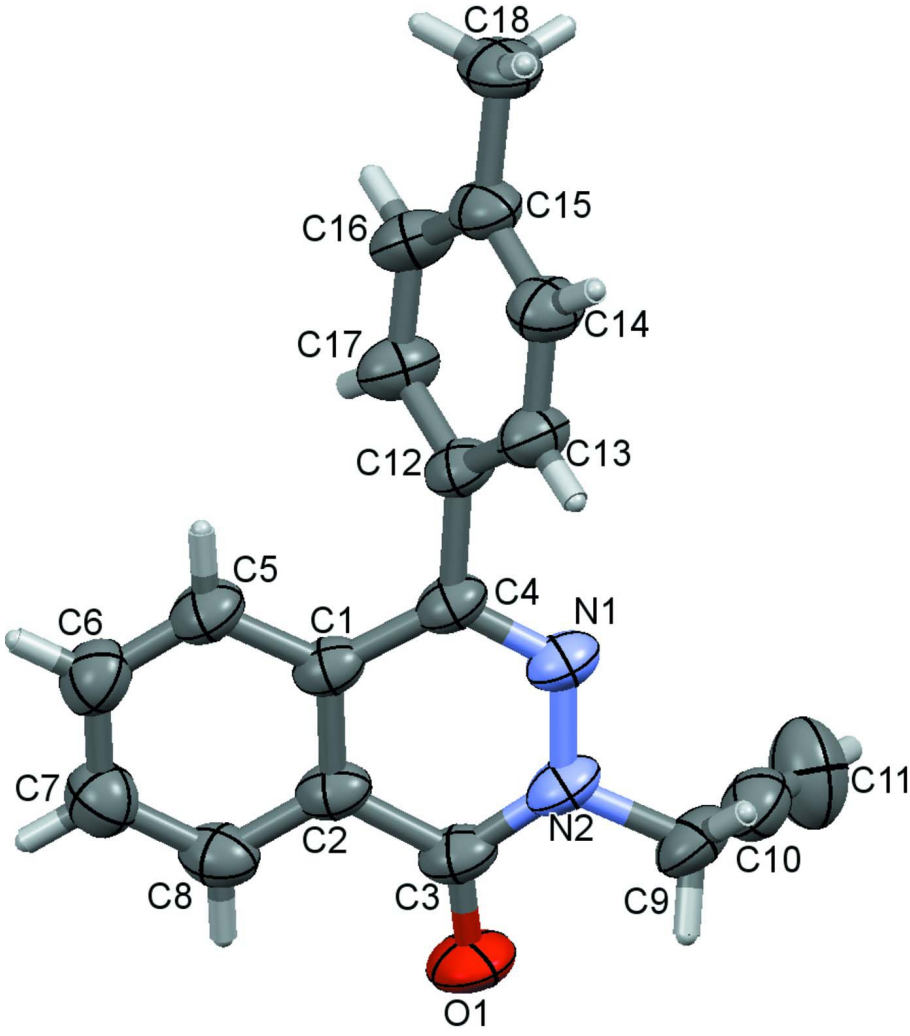

Figure 1

ORTEP diagram of the title compound with 50% probability ellipsoids.

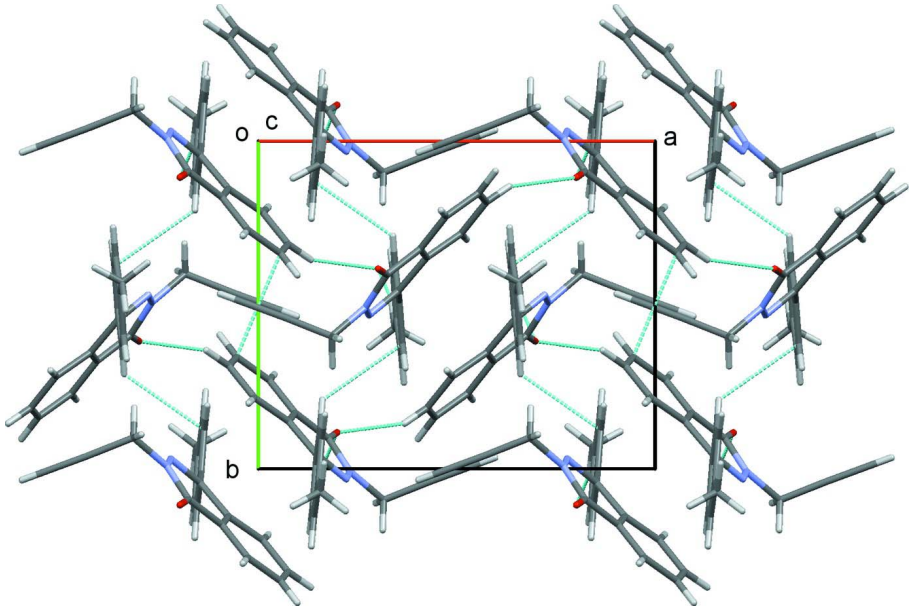

Figure 2

Packing diagram of the title compound, viewed along the *c* axis.

4-(4-Methylphenyl)-2-(prop-2-yn-1-yl)phthalazin-1(2*H*)-one

Crystal data

$C_{18}H_{14}N_2O$
$M_r = 274.31$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 11.9917\ (19)$ Å
$b = 9.7116\ (16)$ Å
$c = 12.602\ (2)$ Å
$\beta = 101.285\ (7)°$
$V = 1439.2\ (4)$ Å^3
$Z = 4$
$F(000) = 576$
$D_x = 1.266$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 2337 reflections
$\theta = 4.7–64.6°$
$\mu = 0.63$ mm^{-1}
$T = 296$ K
Block, yellow
0.23 × 0.20 × 0.19 mm

Data collection

Bruker X8 Proteum diffractometer
Radiation source: Bruker MicroStar microfocus rotating anode
Helios multilayer optics monochromator
Detector resolution: 10.7 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
$T_{min} = 0.864$, $T_{max} = 0.886$
8733 measured reflections
2337 independent reflections
2093 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 64.6°$, $\theta_{min} = 4.7°$
$h = -13\rightarrow12$
$k = -10\rightarrow11$
$l = -8\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.132$
$S = 1.10$
2337 reflections
192 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
$w = 1/[\sigma^2(F_o^2) + (0.0698P)^2 + 0.2752P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}
Extinction correction: *SHELXL*, $FC^*=KFC[1+0.001XFC^2\Lambda^3/SIN(2\Theta)]^{-1/4}$
Extinction coefficient: 0.0090 (9)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.19809 (12)	−0.11059 (16)	1.21105 (8)	0.0755 (5)
N1	0.22126 (12)	0.03101 (14)	0.95663 (10)	0.0524 (4)
N2	0.23069 (12)	0.00807 (15)	1.06549 (10)	0.0548 (5)
C1	0.07580 (12)	−0.14488 (16)	0.92636 (11)	0.0464 (5)
C2	0.09215 (13)	−0.17108 (17)	1.03779 (11)	0.0486 (5)
C3	0.17640 (14)	−0.09248 (18)	1.11276 (12)	0.0536 (5)
C4	0.14781 (13)	−0.04216 (16)	0.88990 (11)	0.0468 (5)
C5	−0.01049 (14)	−0.21765 (18)	0.85760 (13)	0.0562 (5)
C6	−0.07462 (15)	−0.3140 (2)	0.89874 (15)	0.0645 (6)
C7	−0.05524 (16)	−0.3416 (2)	1.00855 (15)	0.0647 (6)
C8	0.02715 (15)	−0.27061 (19)	1.07770 (14)	0.0588 (6)
C9	0.31525 (16)	0.0952 (2)	1.13355 (14)	0.0646 (6)
C10	0.43037 (18)	0.0402 (2)	1.14605 (14)	0.0653 (7)
C11	0.5226 (2)	−0.0013 (3)	1.1548 (2)	0.0997 (10)
C12	0.14520 (13)	−0.01379 (17)	0.77355 (12)	0.0473 (5)
C13	0.13700 (15)	0.11933 (18)	0.73343 (13)	0.0554 (6)
C14	0.13974 (16)	0.1456 (2)	0.62603 (13)	0.0604 (6)
C15	0.15141 (14)	0.0409 (2)	0.55518 (12)	0.0567 (6)
C16	0.15944 (16)	−0.0918 (2)	0.59542 (13)	0.0638 (6)
C17	0.15584 (16)	−0.11952 (19)	0.70219 (13)	0.0583 (6)
C18	0.15649 (17)	0.0690 (2)	0.43701 (13)	0.0708 (7)
H5	−0.02440	−0.20060	0.78360	0.0670*
H6	−0.13170	−0.36110	0.85220	0.0770*
H7	−0.09810	−0.40830	1.03530	0.0780*
H8	0.03990	−0.28870	1.15150	0.0710*
H9A	0.31310	0.18630	1.10180	0.0770*
H9B	0.29570	0.10410	1.20440	0.0770*
H11	0.59650	−0.03440	1.16180	0.1200*
H13	0.12950	0.19220	0.77940	0.0660*

H14	0.13360	0.23590	0.60110	0.0720*
H16	0.16750	−0.16440	0.54940	0.0760*
H17	0.16060	−0.21010	0.72650	0.0700*
H18A	0.12210	0.15650	0.41580	0.1060*
H18B	0.11620	−0.00210	0.39220	0.1060*
H18C	0.23440	0.07020	0.42870	0.1060*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0829 (9)	0.1064 (11)	0.0305 (6)	−0.0022 (8)	−0.0054 (6)	0.0047 (6)
N1	0.0592 (8)	0.0597 (8)	0.0341 (7)	0.0033 (6)	−0.0011 (6)	−0.0022 (6)
N2	0.0602 (8)	0.0656 (9)	0.0324 (7)	0.0008 (7)	−0.0062 (6)	−0.0062 (6)
C1	0.0452 (8)	0.0546 (9)	0.0356 (8)	0.0106 (7)	−0.0011 (6)	−0.0001 (6)
C2	0.0463 (8)	0.0616 (10)	0.0347 (8)	0.0120 (7)	0.0004 (6)	0.0012 (7)
C3	0.0546 (9)	0.0684 (11)	0.0338 (8)	0.0099 (8)	−0.0010 (7)	−0.0002 (7)
C4	0.0485 (8)	0.0534 (9)	0.0340 (8)	0.0077 (7)	−0.0029 (6)	−0.0022 (6)
C5	0.0558 (9)	0.0676 (11)	0.0398 (8)	0.0020 (8)	−0.0041 (7)	−0.0023 (7)
C6	0.0544 (10)	0.0732 (12)	0.0611 (11)	−0.0037 (9)	−0.0001 (8)	−0.0053 (9)
C7	0.0560 (10)	0.0724 (12)	0.0646 (11)	−0.0008 (9)	0.0089 (8)	0.0059 (9)
C8	0.0555 (9)	0.0757 (12)	0.0446 (9)	0.0087 (8)	0.0084 (7)	0.0102 (8)
C9	0.0735 (12)	0.0688 (12)	0.0429 (9)	−0.0040 (9)	−0.0094 (8)	−0.0117 (8)
C10	0.0669 (12)	0.0716 (12)	0.0519 (10)	−0.0129 (9)	−0.0015 (8)	−0.0003 (8)
C11	0.0672 (15)	0.0930 (17)	0.135 (2)	−0.0067 (13)	0.0102 (14)	0.0109 (15)
C12	0.0463 (8)	0.0578 (9)	0.0338 (8)	0.0043 (7)	−0.0016 (6)	−0.0019 (6)
C13	0.0683 (10)	0.0552 (10)	0.0416 (9)	−0.0007 (8)	0.0081 (7)	−0.0051 (7)
C14	0.0725 (11)	0.0613 (10)	0.0462 (9)	−0.0006 (8)	0.0089 (8)	0.0060 (8)
C15	0.0498 (9)	0.0820 (12)	0.0360 (8)	−0.0015 (8)	0.0027 (7)	−0.0015 (8)
C16	0.0757 (12)	0.0726 (12)	0.0424 (9)	0.0067 (9)	0.0102 (8)	−0.0125 (8)
C17	0.0726 (11)	0.0572 (10)	0.0434 (9)	0.0116 (8)	0.0075 (8)	−0.0021 (7)
C18	0.0664 (11)	0.1084 (16)	0.0362 (9)	−0.0050 (11)	0.0064 (8)	0.0095 (9)

Geometric parameters (Å, º)

O1—C3	1.2275 (18)	C14—C15	1.378 (3)
N1—N2	1.3725 (18)	C15—C16	1.381 (3)
N1—C4	1.303 (2)	C15—C18	1.527 (2)
N2—C3	1.373 (2)	C16—C17	1.381 (2)
N2—C9	1.463 (2)	C5—H5	0.9300
C1—C2	1.4026 (19)	C6—H6	0.9300
C1—C4	1.452 (2)	C7—H7	0.9300
C1—C5	1.404 (2)	C8—H8	0.9300
C2—C3	1.457 (2)	C9—H9A	0.9700
C2—C8	1.396 (2)	C9—H9B	0.9700
C4—C12	1.486 (2)	C11—H11	0.9300
C5—C6	1.375 (3)	C13—H13	0.9300
C6—C7	1.384 (3)	C14—H14	0.9300
C7—C8	1.369 (3)	C16—H16	0.9300

C9—C10	1.460 (3)	C17—H17	0.9300
C10—C11	1.162 (3)	C18—H18A	0.9600
C12—C13	1.385 (2)	C18—H18B	0.9600
C12—C17	1.387 (2)	C18—H18C	0.9600
C13—C14	1.384 (2)		

N2—N1—C4	118.01 (13)	C15—C16—C17	121.75 (17)
N1—N2—C3	126.58 (13)	C12—C17—C16	120.78 (17)
N1—N2—C9	113.85 (13)	C1—C5—H5	120.00
C3—N2—C9	119.36 (13)	C6—C5—H5	120.00
C2—C1—C4	117.79 (13)	C5—C6—H6	120.00
C2—C1—C5	117.93 (14)	C7—C6—H6	120.00
C4—C1—C5	124.26 (13)	C6—C7—H7	120.00
C1—C2—C3	119.84 (14)	C8—C7—H7	120.00
C1—C2—C8	120.52 (14)	C2—C8—H8	120.00
C3—C2—C8	119.64 (13)	C7—C8—H8	120.00
O1—C3—N2	121.00 (16)	N2—C9—H9A	109.00
O1—C3—C2	124.23 (16)	N2—C9—H9B	109.00
N2—C3—C2	114.77 (13)	C10—C9—H9A	109.00
N1—C4—C1	122.64 (13)	C10—C9—H9B	109.00
N1—C4—C12	114.62 (14)	H9A—C9—H9B	108.00
C1—C4—C12	122.73 (13)	C10—C11—H11	180.00
C1—C5—C6	120.59 (15)	C12—C13—H13	119.00
C5—C6—C7	120.79 (17)	C14—C13—H13	120.00
C6—C7—C8	119.87 (18)	C13—C14—H14	119.00
C2—C8—C7	120.25 (16)	C15—C14—H14	119.00
N2—C9—C10	112.59 (16)	C15—C16—H16	119.00
C9—C10—C11	178.6 (2)	C17—C16—H16	119.00
C4—C12—C13	121.31 (14)	C12—C17—H17	120.00
C4—C12—C17	121.03 (15)	C16—C17—H17	120.00
C13—C12—C17	117.61 (14)	C15—C18—H18A	109.00
C12—C13—C14	121.04 (16)	C15—C18—H18B	109.00
C13—C14—C15	121.49 (17)	C15—C18—H18C	110.00
C14—C15—C16	117.33 (15)	H18A—C18—H18B	109.00
C14—C15—C18	121.90 (17)	H18A—C18—H18C	109.00
C16—C15—C18	120.77 (16)	H18B—C18—H18C	109.00

C4—N1—N2—C3	5.4 (2)	C8—C2—C3—O1	2.6 (3)
C4—N1—N2—C9	−179.97 (16)	C8—C2—C3—N2	−176.34 (16)
N2—N1—C4—C1	0.6 (2)	C1—C2—C8—C7	−1.5 (3)
N2—N1—C4—C12	−178.36 (14)	C3—C2—C8—C7	177.55 (17)
N1—N2—C3—O1	174.12 (16)	N1—C4—C12—C13	−50.0 (2)
N1—N2—C3—C2	−6.9 (2)	N1—C4—C12—C17	127.20 (17)
C9—N2—C3—O1	−0.3 (3)	C1—C4—C12—C13	131.06 (17)
C9—N2—C3—C2	178.71 (15)	C1—C4—C12—C17	−51.7 (2)
N1—N2—C9—C10	−83.42 (18)	C1—C5—C6—C7	−0.2 (3)
C3—N2—C9—C10	91.67 (19)	C5—C6—C7—C8	1.3 (3)
C4—C1—C2—C3	2.2 (2)	C6—C7—C8—C2	−0.4 (3)

C4—C1—C2—C8	−178.72 (15)	C4—C12—C13—C14	177.07 (16)
C5—C1—C2—C3	−176.58 (15)	C17—C12—C13—C14	−0.2 (3)
C5—C1—C2—C8	2.5 (2)	C4—C12—C17—C16	−176.53 (17)
C2—C1—C4—N1	−4.1 (2)	C13—C12—C17—C16	0.8 (3)
C2—C1—C4—C12	174.76 (15)	C12—C13—C14—C15	−0.4 (3)
C5—C1—C4—N1	174.61 (16)	C13—C14—C15—C16	0.5 (3)
C5—C1—C4—C12	−6.5 (2)	C13—C14—C15—C18	−178.98 (17)
C2—C1—C5—C6	−1.6 (2)	C14—C15—C16—C17	0.1 (3)
C4—C1—C5—C6	179.67 (16)	C18—C15—C16—C17	179.55 (18)
C1—C2—C3—O1	−178.30 (17)	C15—C16—C17—C12	−0.7 (3)
C1—C2—C3—N2	2.7 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O1^{i}	0.93	2.45	3.322 (2)	157

Symmetry code: (i) *x*−1/2, −*y*−1/2, *z*−1/2.