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2-Amino-*N*′-phenylbenzohydrazide

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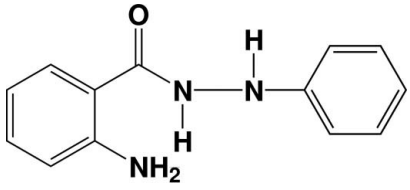
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.068; wR factor = 0.163; data-to-parameter ratio = 18.0.

In the title compound, $C_{13}H_{13}N_3O$, the NNCO unit forms dihedral angles of 35.8 (1) and 84.0 (1)° with the benzene and phenyl rings, respectively. The dihedral angles between the aromatic rings is 61.2 (1)°. An intramolecular N—H···O hydrogen bond occurs. In the crystal, molecules are linked by weak N—H···O hydrogen bonds into $C(4)$ chains parallel to the c axis. Neighbouring chains are linked by weak N—H···N hydrogen bonds, forming $R_4^4(20)$ rings, and resulting in the formation of a two-dimensional network lying parallel to (010). The packing also features π–π stacking interactions between phenyl rings [centroid–centroid distance = 3.803 (2) Å].

Related literature

For the pharmacological activity of quinazolinones, see: Kamal *et al.* (2010) and of benzotriazepinones, see: Filippakopoulos *et al.* (2012); Spencer *et al.* (2008). For the synthesis of the starting material 1*H*-benzo[*d*][1,3]oxazine-2,4-dione, see: Iwakura *et al.* (1976); Leiby & Heindel (1976). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{13}H_{13}N_3O$
M_r = 227.26
Monoclinic, $P2_1/c$
a = 6.1190 (12) Å
b = 19.921 (4) Å
c = 9.6490 (19) Å
β = 94.08 (3)°
V = 1173.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.40 × 0.21 × 0.10 mm

Data collection

Nonius KappaCCD area-detector diffractometer
17096 measured reflections
2923 independent reflections
2330 reflections with $I > 2\sigma(I)$
R_{int} = 0.092

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.163
S = 1.09
2923 reflections
162 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	2.07	2.903 (2)	162
N3—H3A···O1	0.86 (3)	2.21 (3)	2.845 (2)	131 (2)
N2—H2···N3^{ii}	0.86	2.54	3.126 (3)	126

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

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *publCIF* (Westrip, 2010).

We are grateful to the Consejo Superior de Investigaciones Científicas (CSIC) of Spain for the award of a licence for the use of the Cambridge Structural Database (CSD).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DS2196).

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supplementary materials

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2-Amino-*N*′-phenylbenzohydrazide

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Comment

The 2-amino-*N*′-phenylbenzohydrazide **2** is a key intermediate to obtain quinazolinones and benzotriazepines derivatives. The quinazolinone nucleous and its derivatives have been extensively studied because of their wide range of pharmacological activities, including antiviral, antibacterial, antifungal, antimalarial, anticancer, antihypertensive, diuretic, anticonvulsant and anti-inflammatory (Kamal *et al.*, 2010). On the other hand, the benzotriazepinones have been described as efficient enzymatic inhibitors (Filippakopoulos *et al.*, 2012; Spencer *et al.*, 2008). We report herein on the synthesis and crystal structure of the title compound, a member of this important family of compounds. In the title molecule, Fig. 1, the NNCO moiety form a dihedral angle of 35.8 (1)° and 84.0 (1)° with benzene and phenyl rings respectively. The dihedral angles between the aromatic rings is 61.2 (1)°. In the crystal the molecules are packed *via* π–π stacking interaction [centroid–centroid distance 3.803 (2) Å] and linked by N1—H1···O1(x, $-y + 3/2$, $z + 1/2$) weak hydrogen bond to form a C(4) chain running parallel to the *c* axis, which are linked to neighboring chains by N2—H3···N3($x - 1$, y, z) weak hydrogen bond to form $R^4_4(20)$ centrosymmetric rings (Bernstein *et al.*, 1995). One intramolecular N—H···O hydrogen bond is observed too, Fig.2, Table1.

Experimental

The synthesis of the 2-amino-*N*′-phenylbenzohydrazide **2** was done starting of isatoic anhydride (1*H*-benzo[*d*][1,3]oxazine-2,4-dione) (Leiby & Heindel 1976; Iwakura *et al.*, 1976), which was treated with phenyl hydrazine in DMF at reflux by 2 h to give an 82% yield. The product was crystallized in ethyl acetate with melting point 227–228 °C. UV λ(MeOH) 310, 280 and 225 nm, λ(MeONa) 340, 265 and 225 nm. IR cm^{-1}, (Nujol), 3300 (NH), 1670 (carbonyl).

Refinement

The H-atoms could be located in difference Fourier maps. H3A and H3B atoms parameters were freely refined. The remaining H atoms, were positioned geometrically and treated using a riding model with N—H = 0.86 Å, C—H = 0.93 with U_{iso}(H) = k × U_{eq}(N,*C*), where k = 1.2 for both atoms.

Computing details

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *publCIF* (Westrip, 2010).

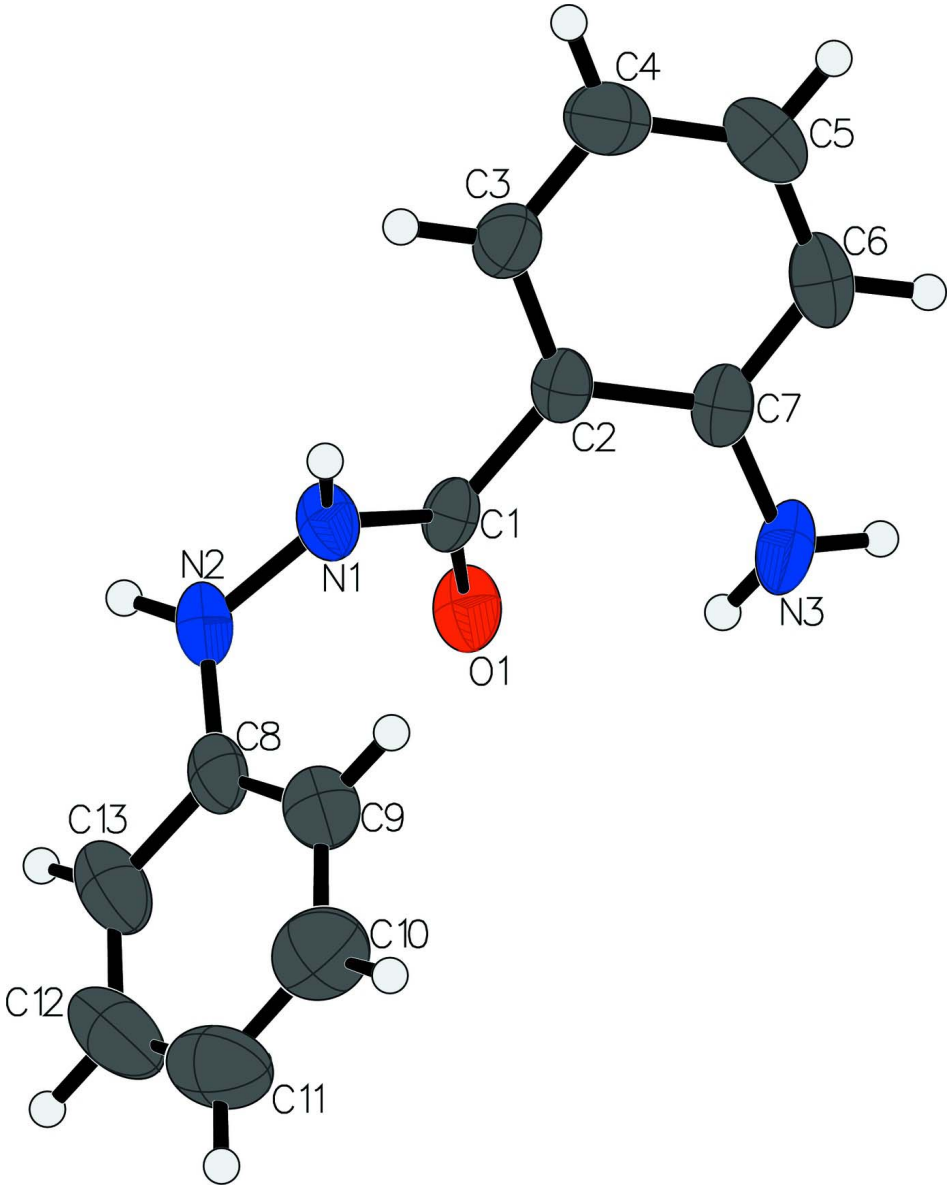

Figure 1

A view of the molecular structure of the title molecule, with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level.

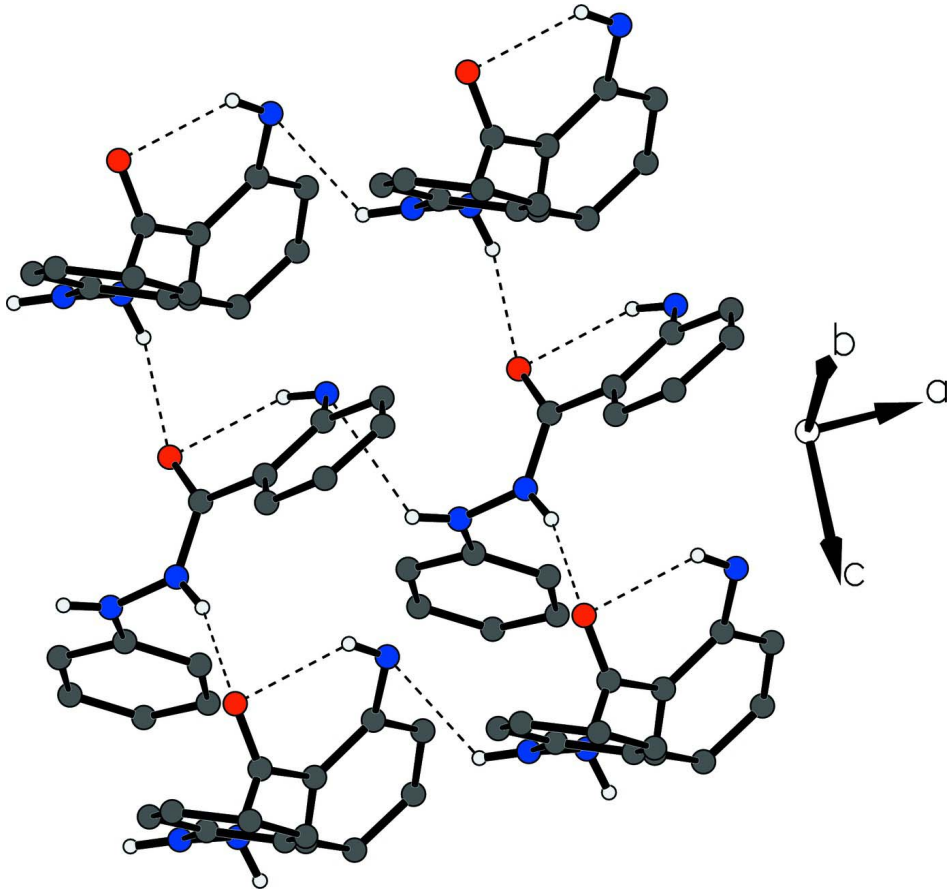

Figure 2

A partial view along the *c* axis of the crystal packing of the title compound, showing the formation of the N—H··· O hydrogen bonded chain and the centrosymmetric R^4_4 (20) rings [see Table 1 for details; the H-atoms not involved in hydrogen-bonding have been omitted for clarity]

2-Amino-*N*′-phenylbenzohydrazide

Crystal data

$C_{13}H_{13}N_3O$
$M_r = 227.26$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 6.1190\ (12)$ Å
$b = 19.921\ (4)$ Å
$c = 9.6490\ (19)$ Å
$\beta = 94.08\ (3)°$
$V = 1173.2\ (4)$ Å^3
$Z = 4$

$F(000) = 480$
$D_x = 1.287$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2923 reflections
$\theta = 3.9–28.6°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Block, colourless
0.40 × 0.21 × 0.10 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans with κ offsets

17096 measured reflections
2923 independent reflections
2330 reflections with $I > 2\sigma(I)$
$R_{int} = 0.092$
$\theta_{max} = 28.6°$, $\theta_{min} = 3.9°$

$h = 0 \rightarrow 8$
$k = 0 \rightarrow 26$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.163$
$S = 1.09$
2923 reflections
162 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0568P)^2 + 0.7268P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.53$ e Å^{-3}
$\Delta\rho_{min} = -0.49$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.6118 (2)	0.71063 (8)	0.36635 (13)	0.0430 (4)
N1	0.5506 (3)	0.73222 (8)	0.58950 (15)	0.0377 (4)
H1	0.5943	0.7527	0.6648	0.045*
N2	0.3725 (3)	0.68841 (9)	0.59083 (17)	0.0407 (4)
H2	0.2401	0.7021	0.5736	0.049*
N3	1.0637 (3)	0.73656 (12)	0.3383 (2)	0.0499 (5)
H3A	0.956 (5)	0.7108 (13)	0.312 (3)	0.059 (8)*
H3B	1.161 (5)	0.7445 (14)	0.273 (3)	0.073 (8)*
C1	0.6555 (3)	0.74322 (9)	0.47424 (17)	0.0313 (4)
C2	0.8220 (3)	0.79772 (9)	0.48509 (17)	0.0323 (4)
C3	0.7862 (3)	0.85526 (10)	0.5633 (2)	0.0421 (5)
H3	0.6585	0.8587	0.6095	0.051*
C4	0.9359 (4)	0.90690 (12)	0.5732 (3)	0.0560 (6)
H4	0.9095	0.9450	0.6253	0.067*
C5	1.1263 (4)	0.90142 (13)	0.5046 (3)	0.0575 (6)
H5	1.2291	0.9359	0.5115	0.069*
C6	1.1644 (3)	0.84588 (12)	0.4269 (2)	0.0483 (5)
H6	1.2936	0.8431	0.3820	0.058*
C7	1.0137 (3)	0.79316 (10)	0.41334 (18)	0.0364 (4)
C8	0.4207 (3)	0.62058 (11)	0.62176 (19)	0.0387 (4)
C9	0.6191 (4)	0.59962 (12)	0.6849 (2)	0.0484 (5)
H9	0.7320	0.6304	0.7030	0.058*
C10	0.6494 (5)	0.53310 (14)	0.7208 (3)	0.0668 (7)

H10	0.7826	0.5195	0.7642	0.080*
C11	0.4861 (6)	0.48666 (14)	0.6935 (3)	0.0756 (9)
H11	0.5089	0.4417	0.7165	0.091*
C12	0.2896 (6)	0.50745 (15)	0.6322 (3)	0.0744 (9)
H12	0.1780	0.4763	0.6141	0.089*
C13	0.2535 (4)	0.57384 (14)	0.5967 (2)	0.0574 (6)
H13	0.1181	0.5873	0.5562	0.069*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0418 (8)	0.0618 (9)	0.0262 (6)	−0.0055 (7)	0.0065 (5)	−0.0045 (6)
N1	0.0373 (8)	0.0497 (10)	0.0269 (7)	−0.0088 (7)	0.0078 (6)	−0.0030 (6)
N2	0.0271 (8)	0.0565 (11)	0.0392 (9)	−0.0040 (7)	0.0075 (6)	0.0005 (7)
N3	0.0307 (9)	0.0768 (14)	0.0433 (10)	0.0021 (9)	0.0102 (8)	−0.0160 (9)
C1	0.0273 (8)	0.0423 (10)	0.0244 (8)	0.0062 (7)	0.0036 (6)	0.0025 (7)
C2	0.0314 (9)	0.0411 (10)	0.0247 (8)	0.0037 (7)	0.0042 (6)	0.0069 (7)
C3	0.0428 (11)	0.0431 (11)	0.0416 (11)	0.0052 (9)	0.0110 (8)	0.0008 (8)
C4	0.0652 (15)	0.0420 (12)	0.0619 (14)	−0.0052 (11)	0.0122 (12)	−0.0036 (10)
C5	0.0571 (14)	0.0535 (14)	0.0623 (15)	−0.0169 (11)	0.0069 (11)	0.0088 (11)
C6	0.0358 (10)	0.0681 (15)	0.0418 (11)	−0.0061 (10)	0.0080 (8)	0.0098 (10)
C7	0.0312 (9)	0.0518 (11)	0.0264 (8)	0.0050 (8)	0.0029 (7)	0.0054 (7)
C8	0.0369 (10)	0.0527 (12)	0.0277 (9)	−0.0090 (9)	0.0110 (7)	−0.0053 (8)
C9	0.0466 (12)	0.0556 (13)	0.0429 (11)	−0.0047 (10)	0.0032 (9)	−0.0023 (9)
C10	0.0792 (18)	0.0638 (17)	0.0579 (15)	0.0090 (14)	0.0075 (13)	0.0107 (12)
C11	0.115 (3)	0.0522 (16)	0.0625 (17)	−0.0101 (17)	0.0263 (17)	0.0061 (12)
C12	0.094 (2)	0.0721 (19)	0.0589 (16)	−0.0427 (17)	0.0213 (15)	−0.0099 (13)
C13	0.0503 (13)	0.0728 (17)	0.0496 (13)	−0.0242 (12)	0.0069 (10)	−0.0072 (11)

Geometric parameters (Å, º)

O1—C1	1.240 (2)	C5—C6	1.366 (4)
N1—C1	1.341 (2)	C5—H5	0.9300
N1—N2	1.397 (2)	C6—C7	1.397 (3)
N1—H1	0.8600	C6—H6	0.9300
N2—C8	1.411 (3)	C8—C9	1.384 (3)
N2—H2	0.8600	C8—C13	1.392 (3)
N3—C7	1.386 (3)	C9—C10	1.379 (4)
N3—H3A	0.86 (3)	C9—H9	0.9300
N3—H3B	0.91 (3)	C10—C11	1.374 (4)
C1—C2	1.487 (3)	C10—H10	0.9300
C2—C3	1.398 (3)	C11—C12	1.366 (5)
C2—C7	1.407 (2)	C11—H11	0.9300
C3—C4	1.376 (3)	C12—C13	1.380 (4)
C3—H3	0.9300	C12—H12	0.9300
C4—C5	1.385 (4)	C13—H13	0.9300
C4—H4	0.9300		
C1—N1—N2	121.98 (15)	C5—C6—C7	121.5 (2)
C1—N1—H1	119.0	C5—C6—H6	119.2

N2—N1—H1	119.0	C7—C6—H6	119.2
N1—N2—C8	116.67 (15)	N3—C7—C6	119.45 (18)
N1—N2—H2	121.7	N3—C7—C2	122.17 (19)
C8—N2—H2	121.7	C6—C7—C2	118.23 (18)
C7—N3—H3A	116.4 (18)	C9—C8—C13	119.1 (2)
C7—N3—H3B	113.5 (18)	C9—C8—N2	123.09 (18)
H3A—N3—H3B	115 (3)	C13—C8—N2	117.7 (2)
O1—C1—N1	121.54 (17)	C10—C9—C8	119.9 (2)
O1—C1—C2	123.18 (16)	C10—C9—H9	120.0
N1—C1—C2	115.26 (15)	C8—C9—H9	120.0
C3—C2—C7	119.10 (18)	C11—C10—C9	121.1 (3)
C3—C2—C1	120.27 (16)	C11—C10—H10	119.5
C7—C2—C1	120.60 (17)	C9—C10—H10	119.5
C4—C3—C2	121.46 (19)	C12—C11—C10	119.0 (3)
C4—C3—H3	119.3	C12—C11—H11	120.5
C2—C3—H3	119.3	C10—C11—H11	120.5
C3—C4—C5	119.1 (2)	C11—C12—C13	121.2 (3)
C3—C4—H4	120.5	C11—C12—H12	119.4
C5—C4—H4	120.5	C13—C12—H12	119.4
C6—C5—C4	120.6 (2)	C12—C13—C8	119.7 (3)
C6—C5—H5	119.7	C12—C13—H13	120.2
C4—C5—H5	119.7	C8—C13—H13	120.2
C1—N1—N2—C8	89.2 (2)	C3—C2—C7—N3	177.46 (18)
N2—N1—C1—O1	−6.4 (3)	C1—C2—C7—N3	−4.6 (3)
N2—N1—C1—C2	172.28 (16)	C3—C2—C7—C6	1.9 (3)
O1—C1—C2—C3	142.55 (19)	C1—C2—C7—C6	179.85 (16)
N1—C1—C2—C3	−36.1 (2)	N1—N2—C8—C9	17.7 (3)
O1—C1—C2—C7	−35.3 (3)	N1—N2—C8—C13	−167.37 (17)
N1—C1—C2—C7	146.01 (17)	C13—C8—C9—C10	0.6 (3)
C7—C2—C3—C4	−1.0 (3)	N2—C8—C9—C10	175.5 (2)
C1—C2—C3—C4	−178.95 (19)	C8—C9—C10—C11	0.8 (4)
C2—C3—C4—C5	−0.3 (3)	C9—C10—C11—C12	−1.3 (4)
C3—C4—C5—C6	0.7 (4)	C10—C11—C12—C13	0.4 (4)
C4—C5—C6—C7	0.3 (4)	C11—C12—C13—C8	0.9 (4)
C5—C6—C7—N3	−177.3 (2)	C9—C8—C13—C12	−1.4 (3)
C5—C6—C7—C2	−1.6 (3)	N2—C8—C13—C12	−176.6 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	2.07	2.903 (2)	162
N3—H3*A*···O1	0.86 (3)	2.21 (3)	2.845 (2)	131 (2)
N2—H2···N3^{ii}	0.86	2.54	3.126 (3)	126

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