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tert-Butyl *N*′-[4-(2-pyridyl)benzylidene]-hydrazinecarboxylate

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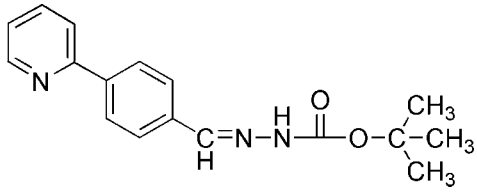
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.007 Å; R factor = 0.051; wR factor = 0.170; data-to-parameter ratio = 7.9.

In the molecule of the title compound, $C_{17}H_{19}N_3O_2$, the aromatic rings are oriented at a dihedral angle of 3.68 (3)°. In the crystal structure, intermolecular N—H···O hydrogen bonds link the molecules into chains along the *a* axis. A weak C—H···π interaction is also present.

Related literature

For a related structure, see: Sugi *et al.* (2002). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{17}H_{19}N_3O_2$
M_r = 297.35
Monoclinic, $P2_1$
a = 5.3080 (11) Å
b = 6.3010 (13) Å
c = 23.459 (5) Å
β = 91.01 (3)°
V = 784.5 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 294 K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.975, T_{max} = 0.992
1743 measured reflections
1566 independent reflections
1249 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.170
S = 1.00
1566 reflections
199 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^{i}	0.86	2.30	3.113 (5)	158
C16—H16A···$Cg2^{ii}$	0.93	2.80	3.588 (4)	144

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, y-\frac{1}{2}, -z$. $Cg2$ is the centroid of the N3/C13–C17 ring.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2661).

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Sugi, K., Matsui, K., Shintaku, T. & Itaya, N. (2002). US Patent No. 6 376 678.

supporting information

Acta Cryst. (2009). E**65**, o1127 [doi:10.1107/S1600536809012835]

tert-Butyl *N*′-[4-(2-pyridyl)benzylidene]hydrazinecarboxylate

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S1. Comment

The title compound is an important intermediate in the syntheses of medicines. We report herein its crystal structure.

In the molecule of the title compound (Fig 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Rings A (C7-C12) and B (N3/C13-C17) are, of course, planar, and they are oriented at a dihedral angle of 3.68 (3)°. So, they are nearly coplanar.

In the crystal structure, intermolecular N-H···O hydrogen bonds (Table 1) link the molecules into chains along the a axis, in which they may be effective in the stabilization of the structure. There also exists a weak C—H···π interaction (Table 1).

S2. Experimental

The title compound was prepared according to a literature method (Sugi *et al.*, 2002). Crystals suitable for X-ray analysis were obtained by dissolving the title compound (1.5 g) in methanol (25 ml) and evaporating the solvent slowly at room temperature for about 5 d.

S3. Refinement

H atoms were positioned geometrically, with N-H = 0.86 Å (for NH) and C-H = 0.93 and 0.96 Å for aromatic and methyl H, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,N), where x = 1.5 for methyl H and x = 1.2 for all other H atoms. The absolute structure could not be determined reliably, and 338 Friedel pairs were averaged before the last cycle of refinement.

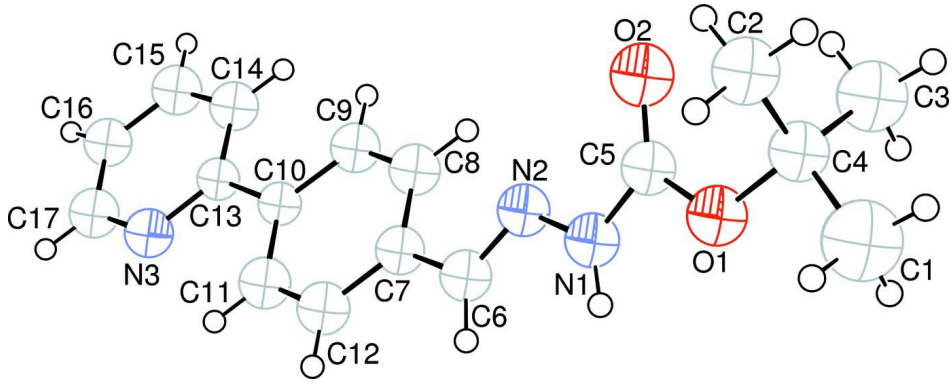

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme.

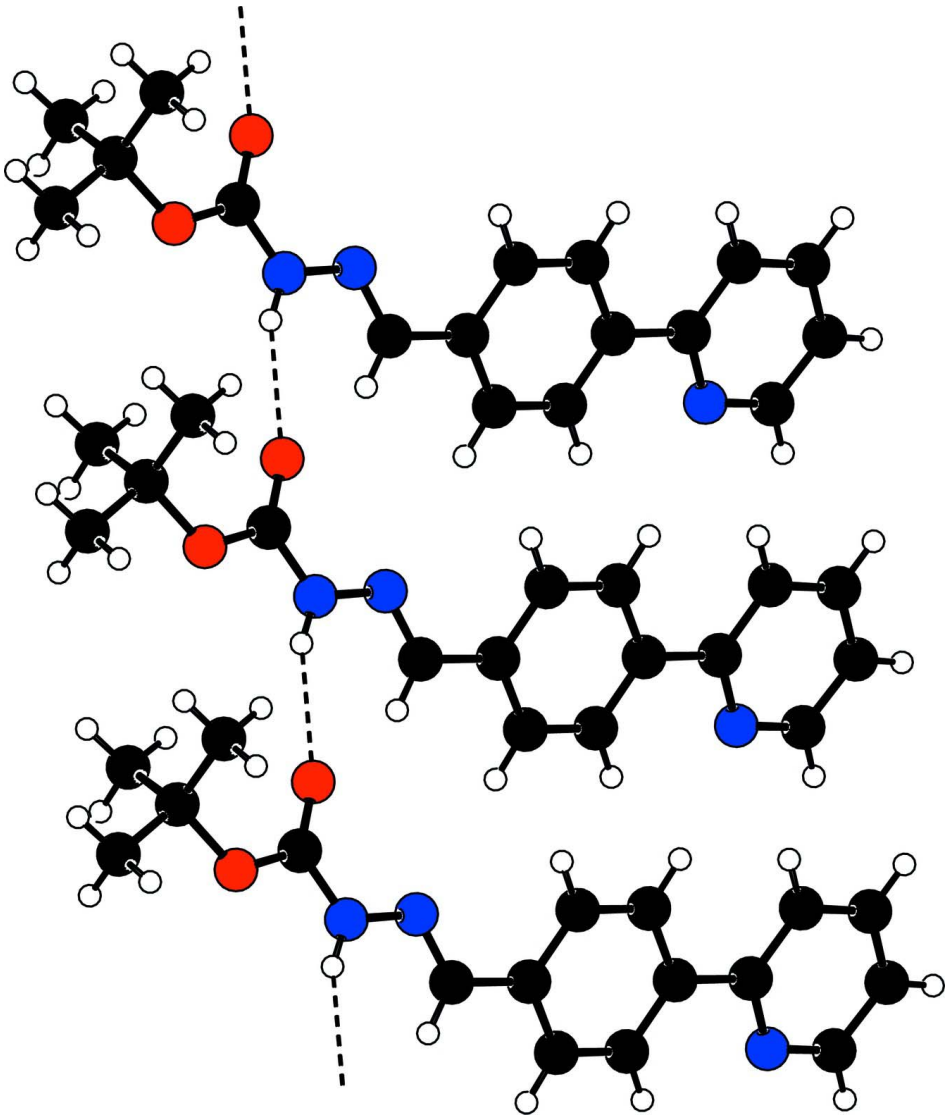

Figure 2

A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

tert-Butyl *N*′-[4-(2-pyridyl)benzylidene]hydrazinecarboxylate

Crystal data

$C_{17}H_{19}N_3O_2$
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Hall symbol: P 2yb
$a = 5.3080\ (11)$ Å
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$c = 23.459\ (5)$ Å
$\beta = 91.01\ (3)°$
$V = 784.5\ (3)$ Å^3
$Z = 2$
$F(000) = 316$
$D_x = 1.259$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9–13°$
$\mu = 0.08$ mm^{-1}
$T = 294$ K
Needle, colorless
0.30 × 0.20 × 0.10 mm

Data collection

Nonius–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.975, T_{max} = 0.992
1743 measured reflections
1566 independent reflections
1249 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 25.3°, θ_{min} = 1.7°
h = 0→6
k = 0→7
l = −28→28
3 standard reflections every 120 min
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.170
S = 1.00
1566 reflections
199 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.33P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.5696 (5)	1.5114 (6)	0.37089 (14)	0.0589 (9)
O2	0.8570 (6)	1.3033 (6)	0.32757 (16)	0.0688 (10)
N1	0.4371 (6)	1.2553 (6)	0.31524 (16)	0.0524 (9)
H1A	0.2891	1.3001	0.3235	0.063*
N2	0.4651 (7)	1.0839 (6)	0.27985 (16)	0.0507 (9)
N3	0.1499 (6)	0.2707 (7)	0.06762 (16)	0.0545 (10)
C1	0.5928 (11)	1.7923 (13)	0.4341 (3)	0.099 (2)
H1B	0.5025	1.8791	0.4071	0.149*
H1C	0.6959	1.8808	0.4583	0.149*
H1D	0.4751	1.7155	0.4569	0.149*
C2	0.9263 (10)	1.7590 (10)	0.3637 (2)	0.0684 (14)
H2B	0.8254	1.8437	0.3381	0.103*
H2C	1.0257	1.6612	0.3423	0.103*
H2D	1.0356	1.8496	0.3859	0.103*
C3	0.9018 (11)	1.4977 (13)	0.4441 (2)	0.0815 (18)

H3A	1.0048	1.4006	0.4233	0.122*
H3B	0.7854	1.4195	0.4669	0.122*
H3C	1.0069	1.5841	0.4684	0.122*
C4	0.7575 (8)	1.6377 (9)	0.40286 (19)	0.0569 (12)
C5	0.6416 (8)	1.3524 (7)	0.3368 (2)	0.0506 (10)
C6	0.2709 (8)	1.0167 (8)	0.25407 (19)	0.0505 (10)
H6A	0.1184	1.0872	0.2581	0.061*
C7	0.2841 (8)	0.8282 (8)	0.21781 (17)	0.0476 (10)
C8	0.4830 (8)	0.6875 (9)	0.2220 (2)	0.0544 (12)
H8A	0.6092	0.7110	0.2493	0.065*
C9	0.4994 (8)	0.5144 (8)	0.18695 (19)	0.0532 (11)
H9A	0.6369	0.4236	0.1905	0.064*
C10	0.3124 (7)	0.4726 (7)	0.14596 (16)	0.0410 (9)
C11	0.1129 (8)	0.6120 (8)	0.1429 (2)	0.0567 (12)
H11A	−0.0145	0.5891	0.1159	0.068*
C12	0.0966 (8)	0.7830 (9)	0.17841 (19)	0.0566 (12)
H12A	−0.0441	0.8706	0.1759	0.068*
C13	0.3321 (7)	0.2892 (7)	0.10648 (16)	0.0412 (9)
C14	0.5270 (9)	0.1443 (8)	0.1101 (2)	0.0564 (12)
H14A	0.6522	0.1580	0.1381	0.068*
C15	0.5332 (9)	−0.0208 (9)	0.0717 (2)	0.0612 (13)
H15A	0.6635	−0.1194	0.0735	0.073*
C16	0.3498 (9)	−0.0394 (8)	0.0314 (2)	0.0566 (12)
H16A	0.3513	−0.1501	0.0052	0.068*
C17	0.1603 (9)	0.1099 (9)	0.0301 (2)	0.0590 (12)
H17A	0.0346	0.0991	0.0021	0.071*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0443 (15)	0.052 (2)	0.081 (2)	−0.0021 (15)	0.0055 (14)	−0.0195 (18)
O2	0.0480 (17)	0.057 (2)	0.102 (2)	0.0030 (18)	0.0129 (16)	−0.022 (2)
N1	0.0446 (18)	0.042 (2)	0.070 (2)	0.0005 (18)	0.0065 (15)	−0.015 (2)
N2	0.057 (2)	0.036 (2)	0.059 (2)	0.0007 (18)	0.0094 (16)	−0.0084 (18)
N3	0.0498 (19)	0.047 (2)	0.066 (2)	−0.001 (2)	−0.0026 (16)	−0.010 (2)
C1	0.070 (3)	0.100 (6)	0.129 (5)	−0.010 (4)	0.012 (3)	−0.068 (5)
C2	0.072 (3)	0.055 (3)	0.078 (3)	−0.008 (3)	−0.005 (2)	0.007 (3)
C3	0.081 (3)	0.096 (5)	0.066 (3)	−0.007 (4)	−0.020 (2)	0.018 (4)
C4	0.050 (2)	0.055 (3)	0.065 (3)	−0.004 (2)	0.003 (2)	−0.011 (3)
C5	0.049 (2)	0.035 (2)	0.067 (3)	0.003 (2)	0.0086 (19)	−0.008 (2)
C6	0.046 (2)	0.044 (2)	0.061 (2)	−0.001 (2)	0.0100 (18)	−0.006 (2)
C7	0.048 (2)	0.044 (2)	0.051 (2)	−0.003 (2)	0.0120 (17)	−0.001 (2)
C8	0.052 (2)	0.049 (3)	0.062 (3)	0.010 (2)	−0.0083 (19)	−0.011 (2)
C9	0.049 (2)	0.042 (2)	0.068 (3)	0.010 (2)	−0.0021 (19)	−0.004 (2)
C10	0.0413 (19)	0.041 (2)	0.0405 (18)	−0.0016 (19)	0.0030 (14)	0.0036 (18)
C11	0.046 (2)	0.050 (3)	0.074 (3)	0.003 (2)	−0.008 (2)	−0.010 (3)
C12	0.042 (2)	0.056 (3)	0.072 (3)	0.014 (2)	−0.0091 (19)	−0.010 (3)
C13	0.0380 (18)	0.040 (2)	0.0461 (19)	−0.004 (2)	0.0050 (15)	−0.0028 (19)

C14	0.059 (3)	0.046 (3)	0.064 (3)	0.009 (2)	0.003 (2)	−0.001 (2)
C15	0.061 (3)	0.049 (3)	0.073 (3)	0.011 (3)	0.009 (2)	−0.003 (3)
C16	0.062 (3)	0.046 (3)	0.062 (3)	−0.004 (2)	0.008 (2)	−0.013 (2)
C17	0.061 (3)	0.055 (3)	0.061 (3)	−0.008 (3)	−0.002 (2)	−0.013 (3)

Geometric parameters (Å, º)

O1—C5	1.341 (5)	C6—C7	1.463 (6)
O1—C4	1.471 (5)	C6—H6A	0.9300
O2—C5	1.208 (5)	C7—C12	1.376 (6)
N1—C5	1.338 (6)	C7—C8	1.381 (6)
N1—N2	1.372 (5)	C8—C9	1.369 (7)
N1—H1A	0.8600	C8—H8A	0.9300
N2—C6	1.259 (6)	C9—C10	1.395 (6)
N3—C13	1.322 (5)	C9—H9A	0.9300
N3—C17	1.344 (6)	C10—C11	1.377 (6)
C1—C4	1.508 (8)	C10—C13	1.486 (6)
C1—H1B	0.9600	C11—C12	1.365 (7)
C1—H1C	0.9600	C11—H11A	0.9300
C1—H1D	0.9600	C12—H12A	0.9300
C2—C4	1.503 (7)	C13—C14	1.381 (6)
C2—H2B	0.9600	C14—C15	1.377 (7)
C2—H2C	0.9600	C14—H14A	0.9300
C2—H2D	0.9600	C15—C16	1.351 (7)
C3—C4	1.508 (8)	C15—H15A	0.9300
C3—H3A	0.9600	C16—C17	1.377 (7)
C3—H3B	0.9600	C16—H16A	0.9300
C3—H3C	0.9600	C17—H17A	0.9300
C5—O1—C4	120.6 (3)	C7—C6—H6A	119.8
C5—N1—N2	119.6 (3)	C12—C7—C8	117.2 (4)
C5—N1—H1A	120.2	C12—C7—C6	121.2 (4)
N2—N1—H1A	120.2	C8—C7—C6	121.6 (4)
C6—N2—N1	117.3 (4)	C9—C8—C7	121.7 (4)
C13—N3—C17	118.7 (4)	C9—C8—H8A	119.1
C4—C1—H1B	109.5	C7—C8—H8A	119.1
C4—C1—H1C	109.5	C8—C9—C10	120.8 (4)
H1B—C1—H1C	109.5	C8—C9—H9A	119.6
C4—C1—H1D	109.5	C10—C9—H9A	119.6
H1B—C1—H1D	109.5	C11—C10—C9	116.9 (4)
H1C—C1—H1D	109.5	C11—C10—C13	121.8 (4)
C4—C2—H2B	109.5	C9—C10—C13	121.3 (4)
C4—C2—H2C	109.5	C12—C11—C10	121.9 (4)
H2B—C2—H2C	109.5	C12—C11—H11A	119.0
C4—C2—H2D	109.5	C10—C11—H11A	119.0
H2B—C2—H2D	109.5	C11—C12—C7	121.4 (4)
H2C—C2—H2D	109.5	C11—C12—H12A	119.3
C4—C3—H3A	109.5	C7—C12—H12A	119.3

C4—C3—H3B	109.5	N3—C13—C14	121.5 (4)
H3A—C3—H3B	109.5	N3—C13—C10	116.1 (4)
C4—C3—H3C	109.5	C14—C13—C10	122.5 (3)
H3A—C3—H3C	109.5	C15—C14—C13	119.1 (4)
H3B—C3—H3C	109.5	C15—C14—H14A	120.5
O1—C4—C2	111.7 (4)	C13—C14—H14A	120.5
O1—C4—C3	110.1 (5)	C16—C15—C14	119.9 (5)
C2—C4—C3	112.8 (4)	C16—C15—H15A	120.1
O1—C4—C1	101.7 (4)	C14—C15—H15A	120.1
C2—C4—C1	109.0 (5)	C15—C16—C17	118.3 (5)
C3—C4—C1	111.0 (5)	C15—C16—H16A	120.9
O2—C5—N1	125.4 (4)	C17—C16—H16A	120.9
O2—C5—O1	125.4 (4)	N3—C17—C16	122.6 (4)
N1—C5—O1	109.2 (3)	N3—C17—H17A	118.7
N2—C6—C7	120.3 (4)	C16—C17—H17A	118.7
N2—C6—H6A	119.8		

C5—N1—N2—C6	−170.0 (5)	C13—C10—C11—C12	−178.8 (4)
C5—O1—C4—C2	63.9 (6)	C10—C11—C12—C7	2.3 (7)
C5—O1—C4—C3	−62.3 (5)	C8—C7—C12—C11	−3.3 (7)
C5—O1—C4—C1	−180.0 (5)	C6—C7—C12—C11	177.3 (4)
N2—N1—C5—O2	0.7 (7)	C17—N3—C13—C14	−1.8 (6)
N2—N1—C5—O1	−178.7 (3)	C17—N3—C13—C10	178.8 (4)
C4—O1—C5—O2	−2.8 (7)	C11—C10—C13—N3	1.4 (6)
C4—O1—C5—N1	176.6 (4)	C9—C10—C13—N3	−176.9 (4)
N1—N2—C6—C7	−177.0 (4)	C11—C10—C13—C14	−178.0 (4)
N2—C6—C7—C12	−163.2 (5)	C9—C10—C13—C14	3.7 (6)
N2—C6—C7—C8	17.4 (7)	N3—C13—C14—C15	1.1 (7)
C12—C7—C8—C9	2.5 (7)	C10—C13—C14—C15	−179.6 (4)
C6—C7—C8—C9	−178.1 (4)	C13—C14—C15—C16	−0.2 (7)
C7—C8—C9—C10	−0.8 (7)	C14—C15—C16—C17	0.1 (7)
C8—C9—C10—C11	−0.3 (7)	C13—N3—C17—C16	1.7 (7)
C8—C9—C10—C13	178.0 (4)	C15—C16—C17—N3	−0.8 (7)
C9—C10—C11—C12	−0.4 (7)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2^{i}	0.86	2.30	3.113 (5)	158
C16—H16*A*···*Cg*2^{ii}	0.93	2.80	3.588 (4)	144

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