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1-[(*E*)-Anthracen-9-ylmethylidene]-2-(2,4-dinitrophenyl)hydrazine

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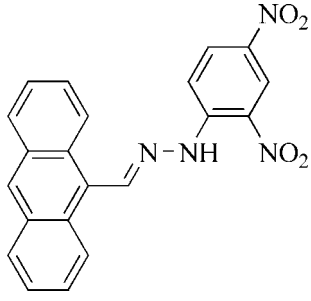
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.009 Å; R factor = 0.074; wR factor = 0.242; data-to-parameter ratio = 14.1.

In the title Schiff base, $C_{21}H_{14}N_4O_4$, the dihedral angle between the two nitro groups and the central benzene ring are 83.6 (5) and 2.6 (6)°. The anthracene ring system and the benzene ring make a dihedral angle of 0.7 (2)°. Intramolecular N—H···O and C—H···N hydrogen bonds occur. In the crystal, C—H···O hydrogen bonds link the molecules, forming chains along the *b*-axis direction.

Related literature

For general background to hydrazone derivatives, see: Kahwa *et al.* (1986). For the structures of 2,4-dinitrophenylhydrazine and 9-anthraldehyde, see: Okabe *et al.* (1993) and Trotter (1959), respectively.



Experimental

Crystal data

$C_{21}H_{14}N_4O_4$ M_r = 386.36
Orthorhombic, $P2_12_12_1$
a = 5.6355 (4) Å
b = 8.1597 (5) Å
c = 36.794 (2) Å
V = 1691.95 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.08 × 0.02 × 0.01 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
T_{min} = 0.764, T_{max} = 0.999
18174 measured reflections
3708 independent reflections
1466 reflections with $I > 2\sigma(I)$
R_{int} = 0.132

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.242
S = 0.88
3708 reflections
263 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H03···O4	0.86	1.99	2.617 (7)	129
C11—H11···O4^{i}	0.93	2.47	3.251 (8)	142
C20—H20···N4	0.93	2.25	2.894 (8)	126

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

Data collection: *APEX2* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 2012).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT6891).

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supporting information

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1-[(*E*)-Anthracen-9-ylmethylidene]-2-(2,4-dinitrophenyl)hydrazine

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S1. Comment

The title compound was synthesized as part of an investigation of the coordination properties of Schiff bases functioning as ligands. Metal complexes based on Schiff bases have been developed in biology and macromolecular chemistry in the last years (Kahwa *et al.*, 1986).

The three dimensional arrangement of the molecules is held together by weak hydrogen bonds interactions between C—H and nitro-oxygen atoms.

Each unit is almost planar with a maximum deviation of 0.179 (6) A for O2, bond lengths varying in the ranges of [1.331 (9)–1.463 (8), 1.215 (7)–1.242 (6), 1.294 (7)–1.461 (8) and 1.389 (7) A for C—C, N—O, C—N and N—N respectively] and bond angles agreeing with those for the initial ligands. Molecules grow along the *a*-axis giving layers in the plane *bc* with an ABAB disposition, as well as each A and B layers are actually an alternating double layer. Two neighbor units of compound **1** create an angle of 68.92 (3)° between them along the *c*-axis.

The angle between the two nitro groups and the central benzene ring by 83.6 (5) and 2.6 (6)°, and the angle between these two nitro groups is 11.1 (7)°. Dihedral angle between the two aromatic parts of the molecule are 179.7 (6) and -171.7 (6)°, for C8—C7—N4—N3 and C7—N4—N3—C1 respectively.

S2. Experimental

All reagents were obtained from commercial sources and used wirh no further purifications.

The compound was obtained when 1 g of (2,4-dinitrophenyl) hydrazine was dissolved in 5 mL of concentrated H_2SO_4.7.5 mL of water where added very slowly to the solution, after this were also added 25 mL of ethanol. In other flask, 4 mL of ethanol 0.05 g of anthracene-9-carbaldehyde where dissolved, and then, 1.80 mL of (2,4-dinitrophenyl)-hydrazine was added to the solution. The two solutions were mixed and left to stand, at room temperature, for 24 h and then the solid compound was filtered., 049 g (52,7%)of the final product were obtained.

S3. Refinement

All H atoms could be located in a difference Fourier synthesis but were placed in calculated positions and refined as riding on their parent atoms, using *SHELXL97* (Sheldrick, 2008) defaults. Due to the absence of anomalous scatterers, the absolute structure could not be determined.

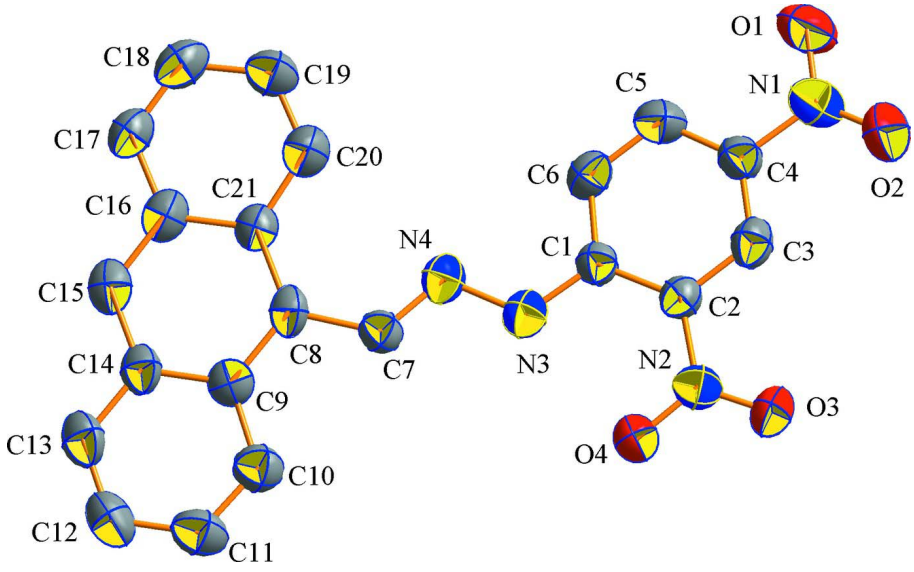

Figure 1

Asymmetric unit of the title compound, showing the atom-labelling scheme and displacement ellipsoids drawn at the 50% probability level.

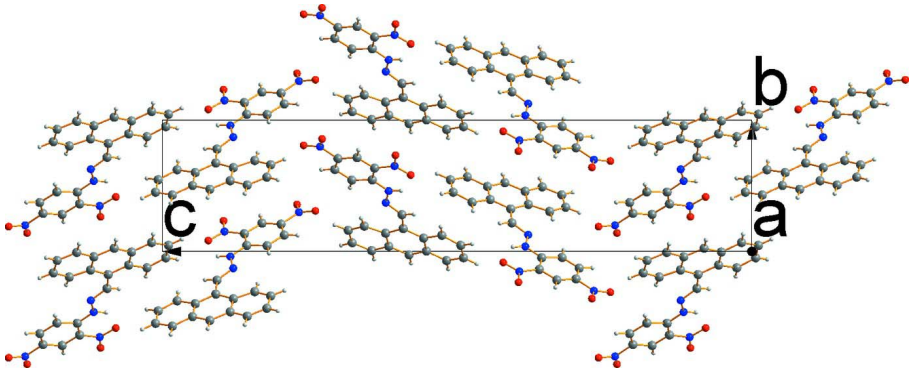

Figure 2

View of the crystal packing of the title compound, projected along *c*.

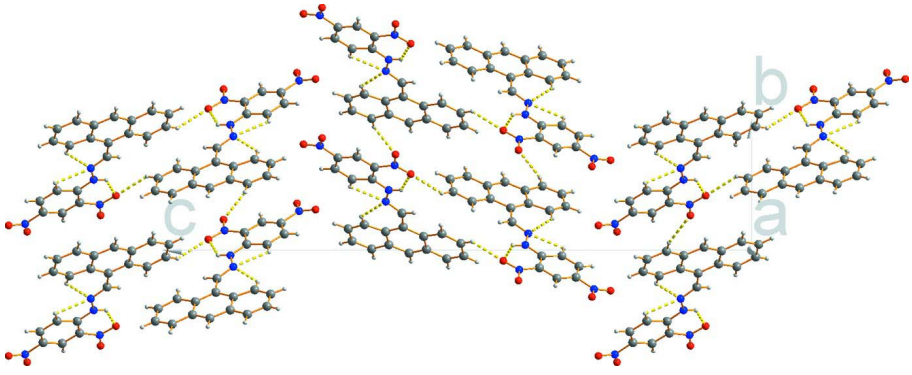

Figure 3

A view showing part of the three-dimensional supramolecular network linked by weak hydrogen-bond interactions (*yellow dotted lines*).

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Crystal data

$C_{21}H_{14}N_4O_4$
$M_r = 386.36$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 5.6355$ (4) Å
$b = 8.1597$ (5) Å
$c = 36.794$ (2) Å
$V = 1691.95$ (19) Å^3
$Z = 4$
$F(000) = 146$
$D_x = 1.517$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1164 reflections
$\theta = 3.0$–$18.8°$
$\mu = 0.11$ mm^{-1}
$T = 293$ K
Needle, colorless
0.08 × 0.02 × 0.01 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
$T_{min} = 0.764$, $T_{max} = 0.999$
18174 measured reflections
3708 independent reflections
1466 reflections with $I > 2\sigma(I)$
$R_{int} = 0.132$
$\theta_{max} = 27.3°$, $\theta_{min} = 3.3°$
$h = -7 \rightarrow 7$
$k = -10 \rightarrow 9$
$l = -46 \rightarrow 46$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.074$
$wR(F^2) = 0.242$
$S = 0.88$
3708 reflections
263 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
$w = 1/[\sigma^2(F_o^2) + (0.1212P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.036 (5)

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	0.2253 (10)	0.1891 (7)	0.26020 (13)	0.0951 (18)
O2	−0.0875 (10)	0.1412 (7)	0.22882 (14)	0.0932 (17)
O3	−0.1033 (8)	0.2666 (5)	0.10426 (11)	0.0669 (13)

O4	0.1605 (8)	0.4177 (5)	0.07843 (12)	0.0703 (13)
N1	0.1114 (12)	0.2008 (7)	0.23234 (16)	0.0725 (16)
N2	0.0789 (10)	0.3493 (6)	0.10576 (14)	0.0558 (14)
N3	0.5138 (9)	0.5396 (6)	0.11586 (14)	0.0559 (14)
H03	0.4470	0.5385	0.0948	0.067*
N4	0.7204 (9)	0.6275 (6)	0.12078 (13)	0.0561 (14)
C1	0.4148 (10)	0.4550 (7)	0.14367 (16)	0.0475 (14)
C2	0.2018 (10)	0.3647 (7)	0.13979 (15)	0.0481 (15)
C3	0.1020 (12)	0.2848 (7)	0.16947 (17)	0.0554 (16)
H3	−0.0398	0.2275	0.1671	0.066*
C4	0.2148 (11)	0.2921 (8)	0.20181 (16)	0.0536 (16)
C5	0.4246 (11)	0.3756 (8)	0.20701 (17)	0.0582 (17)
H5	0.4983	0.3771	0.2296	0.070*
C6	0.5215 (11)	0.4563 (8)	0.17795 (17)	0.0562 (16)
H6	0.6626	0.5137	0.1811	0.067*
C7	0.7784 (10)	0.7172 (7)	0.09321 (16)	0.0497 (15)
H7	0.6795	0.7136	0.0730	0.060*
C8	0.9857 (10)	0.8234 (7)	0.09117 (16)	0.0490 (15)
C9	1.0328 (10)	0.8946 (7)	0.05641 (16)	0.0482 (15)
C10	0.8823 (13)	0.8738 (8)	0.02552 (16)	0.0609 (17)
H10	0.7477	0.8083	0.0274	0.073*
C11	0.9330 (12)	0.9482 (8)	−0.00647 (18)	0.0667 (19)
H11	0.8285	0.9355	−0.0258	0.080*
C12	1.1380 (13)	1.0440 (8)	−0.0115 (2)	0.070 (2)
H12	1.1724	1.0905	−0.0339	0.084*
C13	1.2834 (13)	1.0664 (7)	0.01730 (19)	0.0652 (18)
H13	1.4182	1.1309	0.0145	0.078*
C14	1.2364 (12)	0.9944 (7)	0.05161 (17)	0.0519 (15)
C15	1.3859 (11)	1.0226 (7)	0.08107 (18)	0.0583 (17)
H15	1.5209	1.0862	0.0777	0.070*
C21	1.1340 (10)	0.8580 (7)	0.12098 (15)	0.0466 (14)
C16	1.3403 (10)	0.9587 (7)	0.11560 (17)	0.0525 (16)
C17	1.4973 (12)	0.9911 (8)	0.14472 (18)	0.0591 (17)
H17	1.6304	1.0560	0.1407	0.071*
C18	1.4578 (12)	0.9301 (8)	0.1779 (2)	0.0660 (19)
H18	1.5653	0.9491	0.1966	0.079*
C19	1.2511 (12)	0.8366 (8)	0.18424 (18)	0.0635 (18)
H19	1.2202	0.7970	0.2075	0.076*
C20	1.0978 (12)	0.8040 (8)	0.15704 (17)	0.0604 (17)
H20	0.9623	0.7433	0.1622	0.073*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.097 (4)	0.130 (5)	0.059 (3)	−0.006 (4)	−0.007 (3)	0.025 (3)
O2	0.080 (4)	0.121 (4)	0.079 (3)	−0.027 (4)	0.010 (3)	0.016 (3)
O3	0.056 (3)	0.074 (3)	0.070 (3)	−0.017 (3)	−0.005 (2)	0.000 (2)
O4	0.069 (3)	0.085 (3)	0.057 (3)	−0.020 (3)	0.000 (2)	0.010 (2)

N1	0.071 (4)	0.086 (4)	0.061 (4)	0.004 (4)	0.007 (4)	0.004 (3)
N2	0.054 (3)	0.057 (3)	0.057 (3)	0.005 (3)	−0.004 (3)	−0.002 (3)
N3	0.053 (3)	0.055 (3)	0.059 (3)	−0.004 (3)	0.002 (3)	−0.001 (3)
N4	0.045 (3)	0.059 (3)	0.065 (3)	−0.001 (3)	0.002 (3)	0.001 (3)
C1	0.043 (3)	0.050 (3)	0.050 (4)	0.000 (3)	0.000 (3)	0.001 (3)
C2	0.044 (4)	0.052 (4)	0.048 (3)	−0.003 (3)	−0.002 (3)	0.002 (3)
C3	0.047 (3)	0.054 (4)	0.066 (4)	−0.002 (3)	0.008 (3)	−0.008 (3)
C4	0.052 (4)	0.065 (4)	0.045 (4)	−0.002 (4)	0.003 (3)	0.004 (3)
C5	0.056 (4)	0.068 (4)	0.051 (4)	0.006 (4)	−0.005 (3)	−0.004 (3)
C6	0.050 (4)	0.058 (4)	0.061 (4)	−0.004 (3)	−0.005 (3)	−0.001 (3)
C7	0.049 (4)	0.051 (4)	0.049 (3)	−0.001 (3)	0.003 (3)	−0.002 (3)
C8	0.044 (3)	0.039 (3)	0.064 (4)	−0.002 (3)	0.006 (3)	−0.001 (3)
C9	0.045 (3)	0.042 (3)	0.059 (4)	0.006 (3)	0.001 (3)	−0.001 (3)
C10	0.063 (4)	0.063 (4)	0.056 (4)	0.001 (4)	−0.002 (4)	0.002 (3)
C11	0.064 (5)	0.076 (5)	0.060 (4)	0.006 (4)	−0.004 (4)	0.005 (4)
C12	0.084 (5)	0.062 (4)	0.065 (4)	0.003 (4)	0.014 (4)	0.011 (4)
C13	0.072 (4)	0.045 (4)	0.079 (5)	−0.007 (4)	0.014 (4)	0.007 (3)
C14	0.058 (4)	0.040 (3)	0.058 (4)	−0.002 (3)	0.002 (3)	−0.001 (3)
C15	0.053 (4)	0.046 (4)	0.076 (5)	0.000 (3)	0.013 (4)	−0.002 (3)
C21	0.044 (3)	0.040 (3)	0.055 (4)	0.001 (3)	0.002 (3)	−0.003 (3)
C16	0.043 (4)	0.045 (3)	0.069 (4)	0.009 (3)	0.000 (3)	−0.007 (3)
C17	0.046 (4)	0.060 (4)	0.072 (5)	−0.002 (3)	−0.004 (4)	−0.006 (4)
C18	0.057 (4)	0.070 (5)	0.071 (5)	0.007 (4)	−0.010 (4)	−0.006 (4)
C19	0.067 (5)	0.065 (4)	0.059 (4)	0.007 (4)	−0.001 (4)	0.006 (3)
C20	0.057 (4)	0.058 (4)	0.067 (4)	−0.006 (4)	0.000 (4)	−0.001 (3)

Geometric parameters (Å, º)

O1—N1	1.214 (7)	C9—C14	1.418 (8)
O2—N1	1.229 (7)	C9—C10	1.428 (8)
O3—N2	1.230 (6)	C10—C11	1.355 (8)
O4—N2	1.238 (6)	C10—H10	0.9300
N1—C4	1.469 (8)	C11—C12	1.407 (9)
N2—C2	1.436 (7)	C11—H11	0.9300
N3—C1	1.355 (7)	C12—C13	1.351 (9)
N3—N4	1.379 (6)	C12—H12	0.9300
N3—H03	0.8600	C13—C14	1.418 (8)
N4—C7	1.293 (7)	C13—H13	0.9300
C1—C6	1.397 (8)	C14—C15	1.392 (8)
C1—C2	1.416 (8)	C15—C16	1.397 (8)
C2—C3	1.390 (8)	C15—H15	0.9300
C3—C4	1.350 (8)	C21—C20	1.413 (7)
C3—H3	0.9300	C21—C16	1.437 (8)
C4—C5	1.378 (8)	C16—C17	1.414 (8)
C5—C6	1.369 (8)	C17—C18	1.337 (9)
C5—H5	0.9300	C17—H17	0.9300
C6—H6	0.9300	C18—C19	1.412 (9)
C7—C8	1.456 (7)	C18—H18	0.9300

C7—H7	0.9300	C19—C20	1.348 (8)
C8—C21	1.408 (8)	C19—H19	0.9300
C8—C9	1.430 (8)	C20—H20	0.9300
O1—N1—O2	122.7 (6)	C11—C10—C9	120.8 (7)
O1—N1—C4	118.4 (6)	C11—C10—H10	119.6
O2—N1—C4	118.8 (6)	C9—C10—H10	119.6
O3—N2—O4	121.4 (5)	C10—C11—C12	122.4 (7)
O3—N2—C2	119.3 (5)	C10—C11—H11	118.8
O4—N2—C2	119.3 (5)	C12—C11—H11	118.8
C1—N3—N4	120.9 (5)	C13—C12—C11	118.1 (6)
C1—N3—H03	119.6	C13—C12—H12	121.0
N4—N3—H03	119.6	C11—C12—H12	121.0
C7—N4—N3	113.9 (5)	C12—C13—C14	121.9 (7)
N3—C1—C6	120.0 (6)	C12—C13—H13	119.0
N3—C1—C2	122.6 (6)	C14—C13—H13	119.0
C6—C1—C2	117.4 (5)	C15—C14—C13	120.8 (6)
C3—C2—C1	120.5 (5)	C15—C14—C9	119.2 (6)
C3—C2—N2	116.7 (5)	C13—C14—C9	120.0 (6)
C1—C2—N2	122.8 (5)	C14—C15—C16	122.4 (6)
C4—C3—C2	118.8 (6)	C14—C15—H15	118.8
C4—C3—H3	120.6	C16—C15—H15	118.8
C2—C3—H3	120.6	C8—C21—C20	125.7 (6)
C3—C4—C5	123.2 (6)	C8—C21—C16	119.2 (5)
C3—C4—N1	117.7 (6)	C20—C21—C16	115.1 (6)
C5—C4—N1	119.0 (6)	C15—C16—C17	120.3 (6)
C6—C5—C4	118.1 (6)	C15—C16—C21	119.2 (6)
C6—C5—H5	120.9	C17—C16—C21	120.6 (6)
C4—C5—H5	120.9	C18—C17—C16	121.2 (6)
C5—C6—C1	122.0 (6)	C18—C17—H17	119.4
C5—C6—H6	119.0	C16—C17—H17	119.4
C1—C6—H6	119.0	C17—C18—C19	119.3 (6)
N4—C7—C8	125.5 (6)	C17—C18—H18	120.4
N4—C7—H7	117.3	C19—C18—H18	120.4
C8—C7—H7	117.3	C20—C19—C18	120.8 (6)
C21—C8—C9	120.4 (5)	C20—C19—H19	119.6
C21—C8—C7	123.7 (5)	C18—C19—H19	119.6
C9—C8—C7	115.9 (5)	C19—C20—C21	122.9 (6)
C14—C9—C10	116.7 (5)	C19—C20—H20	118.6
C14—C9—C8	119.7 (5)	C21—C20—H20	118.6
C10—C9—C8	123.6 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H03···O4	0.86	1.99	2.617 (7)	129

C11—H11···O4^{i}	0.93	2.47	3.251 (8)	142
C20—H20···N4	0.93	2.25	2.894 (8)	126

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