

6-[(*R*)-(2-Hydroxy-1-phenylethyl)aminomethylidene]-4-(2-phenyldiazen-1-yl)cyclohexa-2,4-dien-1-one

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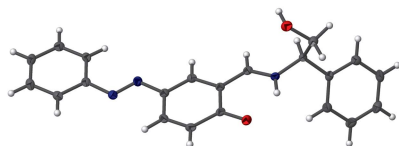
Keywords: crystal structure; Schiff base; azobenzene; photochromic.

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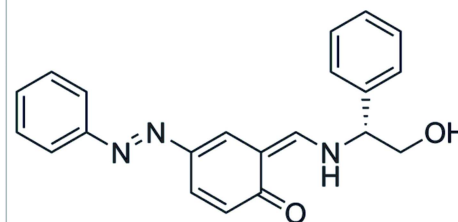
Structural data: full structural data are available from iucrdata.iucr.org

The title chiral photochromic Schiff base compound, C₂₁H₁₉N₃O₂, was synthesized from (*R*)-(-)-2-phenylglycinol and salicylaldehyde of azobenzene derivative. The molecule exhibits the keto–amine tautomeric form and displays characteristic features of azobenzene derivatives. The diazenyl group adopts a *trans* (*E*) conformation, with N=N bond length of 1.260 (2) Å. The hydroxy group is involved in an intermolecular O—H···O hydrogen bond.

3D view



Chemical scheme



Structure description

Schiff bases with an azobenzene moiety are well known in the literature (Miura *et al.*, 2009; Aritake *et al.*, 2010; Moriwaki & Akitsu, 2015). Schiff base ligands are known to perform as photochromic, thermochromic, or fluorescent substances (Akitsu *et al.*, 2004; Hadjoudis & Mavridis, 2004; Moustakali-Mavridis *et al.*, 1978; Akitsu & Einaga, 2006*b*). Schiff base complexes have also been investigated regarding changes of chiral conformation in solutions induced by photochromic solutes (Akitsu & Einaga, 2005*a,b*, 2006*a*; Akitsu, 2007) and their optical anisotropy as a composite in polymer films has been also reported (Labarthe *et al.*, 1999). Here we report the crystal structure of the title compound (Fig. 1), a new chiral photochromic dye of a keto–amine tautomer.

Schiff bases display two possible tautomeric forms, namely, phenol–imine and keto–amine. In the solid state, the keto–amine tautomer has been found in naphthaldimine (Hökelek *et al.*, 2000; Ünver *et al.*, 2002), while the phenol–imine tautomer is found in salicylaldimine Schiff bases (Elerman *et al.*, 1998; Dey *et al.*, 2001; Yang & Vittal, 2003). The title molecule (Fig. 1) has a chiral C atom (C9) with an *R* configuration. The C17=O2, C8–N3 and C7–C8 bond lengths of 1.285 (2), 1.299 (2) and 1.420 Å, respectively, are in good agreement with the corresponding distances observed in 4-[(3-chlorophenyl)diazenyl]-2-[[tris(hydroxymethyl)methyl]aminomethylene]cyclohexa-3,5-dien-1(2*H*)-one [1.285 (3), 1.414 (2) and 1.411 (3) Å, respectively; Odabasoglu *et al.*,

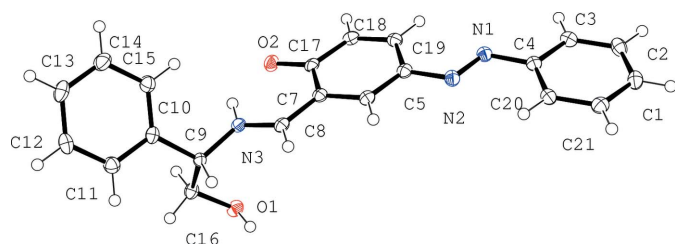


Figure 1
The molecular structure of the title compound (50% probability displacement ellipsoids).

2003]. The π -conjugated system around the imine group is substantially planar as shown by the C7–C8–N3–C9 torsion angle of 172.05 (15)°. The N=N double bond is 1.260 (2) Å and adopts an *E* conformation. All of the geometrical parameters agree with those in related compounds adopting the phenol-imine form, for example the corresponding torsion angle C4–N1–N2–C5 of 176.27 (16)° (Moriwaki & Akitsu, 2015).

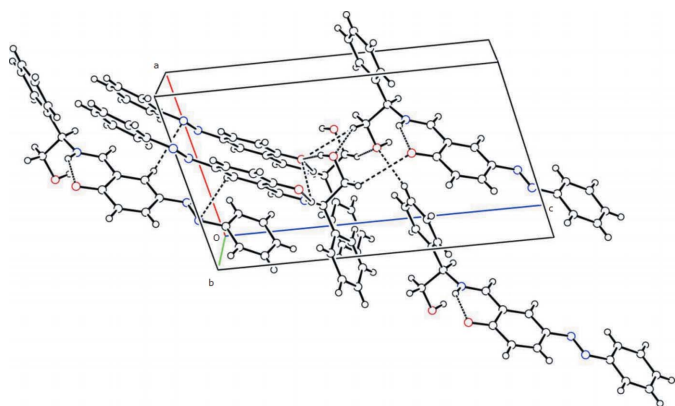


Figure 2
A view of the various N–H...O and O–H...O hydrogen bonds (blue dashed lines) present in the crystal of the title compound.

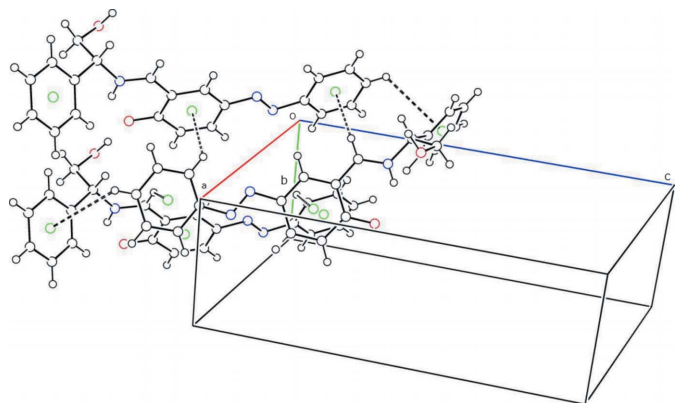


Figure 3
A view of the various C–H... π interactions (blue dashed lines) present in the crystal of the title compound.

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

Cg1 and Cg2 are the centroids of the C1–C4/C20/C21 and C5–C7/C17–C19 rings, respectively.

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
N3–H3...O2	0.86	1.92	2.587 (2)	134
O1–H8...O2 ⁱ	0.93 (3)	1.79 (3)	2.708 (2)	168 (2)
C16–H9...O1 ⁱⁱ	0.97	2.45	3.355 (2)	156
C9–H11...O2 ⁱ	0.98	2.62	3.294 (2)	127
C8–H12...Cg1 ⁱⁱⁱ	0.93	2.75	3.458 (2)	134
C20–H16...Cg2 ⁱⁱⁱ	0.93	2.89	3.480 (2)	122
C3–H18...Cg1 ^{iv}	0.93	3.02	3.711 (2)	132

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

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₂₁ H ₁₉ N ₃ O ₂
<i>M_r</i>	345.39
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁
Temperature (K)	103
<i>a</i> , <i>b</i> , <i>c</i> (Å)	9.0503 (7), 5.9762 (5), 16.3508 (12)
β (°)	102.732 (1)
<i>V</i> (Å ³)	862.61 (12)
<i>Z</i>	2
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.09
Crystal size (mm)	0.15 × 0.09 × 0.08
Data collection	
Diffractometer	Bruker APEXII
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2014)
<i>T_{min}</i> , <i>T_{max}</i>	0.987, 0.993
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	4779, 3542, 3408
<i>R_{int}</i>	0.014
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.653
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.031, 0.074, 1.04
No. of reflections	3542
No. of parameters	238
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.24, -0.20
Absolute structure	Flack parameter not reliable here

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXT* (Sheldrick, 2015a), *SHELXL2014* (Sheldrick, 2015b), *ORTEP-3 for Windows* (Farrugia, 2012) and *SHELXTL* (Sheldrick, 2008).

In the crystal, the molecules are connected through intermolecular hydrogen bonds (O1–H8...O2), forming a sheet arrangement (Table 1, Fig. 2). In addition, weak supra-molecular C–H... π interactions such as C8–H12...Cg1, C3–H18...Cg1, C14–H15...Cg2 and C20–H16...Cg2 are also found in the crystal structure (Table 1, Fig. 3).

Synthesis and crystallization

Treatment of aniline (0.951 g, 10.0 mmol) in 15 ml of 6 *M* HCl and NaNO₂ (0.690 g, 10 mmol) in 15 ml of H₂O for 30 min at

278 K gave rise to a yellow precursor. Treatment of the precursor and salicylaldehyde (1.22 g 10.0 mmol) in 30 ml of 10% NaOH aqueous solution for 1 h at 278 K gave an orange precipitate, which was filtrated and washed with water and ethanol, and dried in a desiccator for several days. Treatment of the brown precipitate (0.678 g, 3.00 mmol) and (*R*)-(-)-2-phenylglycinol (0.4116 g, 3.00 mmol) in 30 ml of toluene for 5 h at 393 K gave rise to an orange compound after evaporation (yield 0.9243 g, 89%). This crude orange compound was filtered and recrystallized by slow evaporation of an acetone solution to give orange prismatic single crystals. IR (KBr, cm^{-1}): 1405 (N=N), 1635 (C=N), 3445 (O-H). ^1H NMR (300 MHz, DMSO) δ (p.p.m.): 3.70 (*m*, 2H), 4.61 (*m*, 1H), 5.26 (*t*, 1H), 7.02 (*d*, 1H), 7.41 (*m*, 9H), 7.83 (*d*, 2H), 7.96 (*dd*, 1H), 8.14 (*d*, 1H), 8.83 (*s*, 1H).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2017). **2**, x170979 [https://doi.org/10.1107/S2414314617009798]

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6-[(*R*)-(2-Hydroxy-1-phenylethyl)aminomethylidene]-4-(2-phenyldiazen-1-yl)cyclohexa-2,4-dien-1-one

Crystal data

$C_{21}H_{19}N_3O_2$
 $M_r = 345.39$
 Monoclinic, $P2_1$
 $a = 9.0503$ (7) Å
 $b = 5.9762$ (5) Å
 $c = 16.3508$ (12) Å
 $\beta = 102.732$ (1)°
 $V = 862.61$ (12) Å³
 $Z = 2$

$F(000) = 364$
 $D_x = 1.330$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 3069 reflections
 $\theta = 2.3$ – 27.7°
 $\mu = 0.09$ mm⁻¹
 $T = 103$ K
 Prism, orange
 $0.15 \times 0.09 \times 0.08$ mm

Data collection

Bruker APEXII
 diffractometer
 Radiation source: fine-focus sealed tube
 Detector resolution: 8.3333 pixels mm⁻¹
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2014)
 $T_{\min} = 0.987$, $T_{\max} = 0.993$

4779 measured reflections
 3542 independent reflections
 3408 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.014$
 $\theta_{\max} = 27.7^\circ$, $\theta_{\min} = 2.3^\circ$
 $h = -6 \rightarrow 11$
 $k = -7 \rightarrow 7$
 $l = -20 \rightarrow 13$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.074$
 $S = 1.04$
 3542 reflections
 238 parameters
 1 restraint
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: mixed
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0306P)^2 + 0.2577P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.24$ e Å⁻³
 $\Delta\rho_{\min} = -0.20$ e Å⁻³
 Absolute structure: Flack parameter not reliable
 here

Special details

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

Refinement. All H atoms were located on difference Fourier maps but C-bound and N-bound H atoms were constrained using a riding model [C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic H atoms, C—H = 0.98 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for the methine H atom, and N—H = 0.86 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$]. The coordinates of the hydroxy H atom were freely refined but its isotropic displacement parameter was considered as $1.5U_{\text{eq}}(\text{O})$.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.52800 (14)	−0.2877 (3)	0.43795 (9)	0.0208 (3)
H8	0.522 (3)	−0.406 (5)	0.4000 (16)	0.031*
O2	0.47218 (16)	0.3639 (2)	0.32957 (8)	0.0208 (3)
N1	0.74880 (17)	0.2803 (3)	0.01627 (9)	0.0173 (3)
N2	0.66505 (18)	0.1391 (3)	0.04115 (9)	0.0184 (3)
N3	0.34194 (17)	−0.0234 (3)	0.32052 (9)	0.0158 (3)
H3	0.3524	0.1072	0.3431	0.019*
C1	0.9597 (2)	0.0884 (4)	−0.17739 (12)	0.0210 (4)
H1	1.01	0.048	−0.219	0.025*
C2	0.9847 (2)	0.2973 (4)	−0.13999 (12)	0.0218 (4)
H19	1.0508	0.3976	−0.1568	0.026*
C3	0.9102 (2)	0.3558 (3)	−0.07716 (11)	0.0183 (4)
H18	0.9263	0.4958	−0.0519	0.022*
C4	0.81138 (19)	0.2051 (3)	−0.05196 (11)	0.0163 (4)
C5	0.6113 (2)	0.2097 (3)	0.11202 (11)	0.0170 (4)
C6	0.5257 (2)	0.0588 (3)	0.14488 (11)	0.0166 (4)
H15	0.5008	−0.0779	0.1182	0.02*
C7	0.4750 (2)	0.1084 (3)	0.21870 (11)	0.0158 (4)
C8	0.3900 (2)	−0.0539 (3)	0.25210 (11)	0.0161 (4)
H12	0.3677	−0.1886	0.2236	0.019*
C9	0.27104 (19)	−0.1981 (3)	0.36140 (11)	0.0150 (4)
H11	0.2624	−0.3331	0.3266	0.018*
C10	0.1132 (2)	−0.1343 (3)	0.37080 (11)	0.0166 (4)
C11	0.0294 (2)	−0.2918 (4)	0.40452 (12)	0.0210 (4)
H7	0.0719	−0.4302	0.422	0.025*
C12	−0.1174 (2)	−0.2432 (4)	0.41213 (12)	0.0235 (4)
H6	−0.1721	−0.3486	0.4351	0.028*
C13	−0.1823 (2)	−0.0378 (4)	0.38554 (12)	0.0238 (4)
H2	−0.281	−0.0063	0.3897	0.029*
C14	−0.0994 (2)	0.1195 (4)	0.35286 (13)	0.0245 (4)
H5	−0.1423	0.2578	0.3355	0.029*
C15	0.0482 (2)	0.0720 (4)	0.34567 (12)	0.0210 (4)
H4	0.1034	0.1792	0.3239	0.025*
C16	0.3771 (2)	−0.2510 (3)	0.44649 (11)	0.0187 (4)
H9	0.3756	−0.1272	0.4847	0.022*

H10	0.3409	-0.3832	0.4703	0.022*
C17	0.5143 (2)	0.3177 (3)	0.26138 (11)	0.0164 (4)
C18	0.6022 (2)	0.4711 (3)	0.22420 (11)	0.0182 (4)
H14	0.6292	0.6084	0.2499	0.022*
C19	0.6472 (2)	0.4208 (3)	0.15215 (11)	0.0174 (4)
H13	0.702	0.5254	0.1289	0.021*
C20	0.7848 (2)	-0.0038 (3)	-0.09025 (11)	0.0180 (4)
H16	0.7177	-0.1037	-0.0741	0.022*
C21	0.8596 (2)	-0.0612 (3)	-0.15289 (11)	0.0203 (4)
H17	0.8428	-0.2005	-0.1786	0.024*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0164 (6)	0.0203 (7)	0.0247 (7)	0.0002 (6)	0.0027 (5)	-0.0014 (6)
O2	0.0251 (7)	0.0181 (7)	0.0216 (7)	-0.0001 (6)	0.0100 (6)	-0.0037 (5)
N1	0.0174 (7)	0.0173 (8)	0.0167 (7)	0.0003 (7)	0.0028 (6)	0.0009 (6)
N2	0.0185 (7)	0.0203 (9)	0.0166 (7)	0.0007 (7)	0.0041 (6)	0.0013 (6)
N3	0.0159 (7)	0.0126 (8)	0.0198 (7)	-0.0002 (6)	0.0056 (6)	-0.0016 (6)
C1	0.0166 (9)	0.0301 (11)	0.0164 (8)	0.0030 (8)	0.0040 (7)	0.0016 (8)
C2	0.0176 (9)	0.0277 (11)	0.0201 (9)	-0.0037 (8)	0.0040 (7)	0.0038 (8)
C3	0.0173 (9)	0.0175 (10)	0.0186 (9)	-0.0024 (8)	0.0004 (7)	0.0011 (7)
C4	0.0136 (8)	0.0190 (9)	0.0152 (8)	0.0015 (7)	0.0008 (7)	0.0023 (7)
C5	0.0154 (8)	0.0179 (9)	0.0171 (8)	0.0020 (8)	0.0022 (7)	0.0001 (7)
C6	0.0162 (8)	0.0160 (9)	0.0171 (8)	0.0002 (7)	0.0022 (7)	-0.0010 (7)
C7	0.0140 (8)	0.0157 (9)	0.0169 (8)	0.0019 (7)	0.0020 (7)	0.0015 (7)
C8	0.0136 (8)	0.0173 (9)	0.0164 (8)	0.0024 (7)	0.0012 (6)	-0.0007 (7)
C9	0.0158 (8)	0.0133 (9)	0.0170 (8)	-0.0002 (7)	0.0060 (7)	-0.0003 (7)
C10	0.0159 (8)	0.0205 (9)	0.0138 (8)	-0.0008 (7)	0.0041 (7)	-0.0029 (7)
C11	0.0219 (9)	0.0193 (9)	0.0221 (9)	-0.0006 (8)	0.0055 (7)	0.0009 (8)
C12	0.0220 (9)	0.0276 (12)	0.0229 (10)	-0.0067 (9)	0.0090 (8)	0.0000 (8)
C13	0.0155 (9)	0.0345 (12)	0.0216 (9)	0.0011 (9)	0.0050 (7)	-0.0042 (9)
C14	0.0208 (9)	0.0248 (11)	0.0278 (10)	0.0053 (9)	0.0055 (8)	0.0006 (9)
C15	0.0204 (9)	0.0211 (10)	0.0222 (9)	0.0007 (8)	0.0064 (7)	0.0020 (8)
C16	0.0175 (8)	0.0205 (10)	0.0179 (9)	0.0005 (8)	0.0038 (7)	-0.0013 (7)
C17	0.0133 (8)	0.0170 (10)	0.0184 (8)	0.0039 (7)	0.0026 (7)	0.0012 (7)
C18	0.0173 (9)	0.0135 (9)	0.0228 (9)	0.0005 (8)	0.0025 (7)	-0.0015 (8)
C19	0.0155 (8)	0.0157 (9)	0.0208 (9)	-0.0004 (7)	0.0037 (7)	0.0029 (7)
C20	0.0165 (9)	0.0195 (10)	0.0176 (8)	-0.0008 (8)	0.0029 (7)	0.0028 (7)
C21	0.0212 (9)	0.0201 (10)	0.0184 (9)	0.0020 (8)	0.0016 (7)	-0.0001 (8)

Geometric parameters (Å, °)

O1—C16	1.420 (2)	C9—C10	1.519 (2)
O1—H8	0.93 (3)	C9—C16	1.539 (2)
O2—C17	1.285 (2)	C9—H11	0.98
N1—N2	1.260 (2)	C10—C15	1.388 (3)
N1—C4	1.430 (2)	C10—C11	1.397 (3)

N2—C5	1.416 (2)	C11—C12	1.392 (3)
N3—C8	1.299 (2)	C11—H7	0.93
N3—C9	1.462 (2)	C12—C13	1.389 (3)
N3—H3	0.86	C12—H6	0.93
C1—C2	1.387 (3)	C13—C14	1.381 (3)
C1—C21	1.393 (3)	C13—H2	0.93
C1—H1	0.93	C14—C15	1.396 (3)
C2—C3	1.392 (3)	C14—H5	0.93
C2—H19	0.93	C15—H4	0.93
C3—C4	1.394 (3)	C16—H9	0.97
C3—H18	0.93	C16—H10	0.97
C4—C20	1.394 (3)	C17—C18	1.434 (3)
C5—C6	1.373 (3)	C18—C19	1.362 (3)
C5—C19	1.426 (3)	C18—H14	0.93
C6—C7	1.414 (2)	C19—H13	0.93
C6—H15	0.93	C20—C21	1.390 (3)
C7—C8	1.420 (3)	C20—H16	0.93
C7—C17	1.438 (3)	C21—H17	0.93
C8—H12	0.93		
C16—O1—H8	105.7 (16)	C11—C10—C9	118.38 (17)
N2—N1—C4	114.28 (15)	C12—C11—C10	120.44 (19)
N1—N2—C5	113.82 (15)	C12—C11—H7	119.8
C8—N3—C9	123.89 (16)	C10—C11—H7	119.8
C8—N3—H3	118.1	C13—C12—C11	120.19 (19)
C9—N3—H3	118.1	C13—C12—H6	119.9
C2—C1—C21	120.29 (18)	C11—C12—H6	119.9
C2—C1—H1	119.9	C14—C13—C12	119.62 (18)
C21—C1—H1	119.9	C14—C13—H2	120.2
C1—C2—C3	119.54 (19)	C12—C13—H2	120.2
C1—C2—H19	120.2	C13—C14—C15	120.4 (2)
C3—C2—H19	120.2	C13—C14—H5	119.8
C2—C3—C4	120.17 (19)	C15—C14—H5	119.8
C2—C3—H18	119.9	C10—C15—C14	120.42 (19)
C4—C3—H18	119.9	C10—C15—H4	119.8
C3—C4—C20	120.31 (17)	C14—C15—H4	119.8
C3—C4—N1	114.70 (17)	O1—C16—C9	111.39 (14)
C20—C4—N1	124.95 (16)	O1—C16—H9	109.4
C6—C5—N2	116.70 (17)	C9—C16—H9	109.4
C6—C5—C19	119.47 (17)	O1—C16—H10	109.4
N2—C5—C19	123.76 (17)	C9—C16—H10	109.4
C5—C6—C7	120.89 (18)	H9—C16—H10	108.0
C5—C6—H15	119.6	O2—C17—C18	121.93 (17)
C7—C6—H15	119.6	O2—C17—C7	121.24 (17)
C6—C7—C8	119.36 (17)	C18—C17—C7	116.84 (16)
C6—C7—C17	120.28 (17)	C19—C18—C17	121.69 (18)
C8—C7—C17	120.31 (16)	C19—C18—H14	119.2
N3—C8—C7	123.23 (18)	C17—C18—H14	119.2

N3—C8—H12	118.4	C18—C19—C5	120.78 (17)
C7—C8—H12	118.4	C18—C19—H13	119.6
N3—C9—C10	112.59 (15)	C5—C19—H13	119.6
N3—C9—C16	108.09 (14)	C21—C20—C4	119.22 (18)
C10—C9—C16	111.93 (14)	C21—C20—H16	120.4
N3—C9—H11	108.0	C4—C20—H16	120.4
C10—C9—H11	108.0	C20—C21—C1	120.47 (19)
C16—C9—H11	108.0	C20—C21—H17	119.8
C15—C10—C11	118.95 (17)	C1—C21—H17	119.8
C15—C10—C9	122.66 (17)		
C4—N1—N2—C5	176.27 (14)	C9—C10—C11—C12	-178.17 (17)
C21—C1—C2—C3	0.6 (3)	C10—C11—C12—C13	0.5 (3)
C1—C2—C3—C4	0.1 (3)	C11—C12—C13—C14	-1.1 (3)
C2—C3—C4—C20	-0.9 (3)	C12—C13—C14—C15	0.7 (3)
C2—C3—C4—N1	176.58 (16)	C11—C10—C15—C14	-1.0 (3)
N2—N1—C4—C3	-176.93 (16)	C9—C10—C15—C14	177.65 (18)
N2—N1—C4—C20	0.5 (2)	C13—C14—C15—C10	0.4 (3)
N1—N2—C5—C6	-176.78 (16)	N3—C9—C16—O1	-49.4 (2)
N1—N2—C5—C19	0.2 (2)	C10—C9—C16—O1	-173.93 (16)
N2—C5—C6—C7	175.99 (16)	C6—C7—C17—O2	-178.44 (17)
C19—C5—C6—C7	-1.1 (3)	C8—C7—C17—O2	-1.0 (3)
C5—C6—C7—C8	-178.38 (17)	C6—C7—C17—C18	1.6 (2)
C5—C6—C7—C17	-0.9 (3)	C8—C7—C17—C18	179.09 (17)
C9—N3—C8—C7	-172.05 (16)	O2—C17—C18—C19	179.68 (17)
C6—C7—C8—N3	178.53 (17)	C7—C17—C18—C19	-0.4 (3)
C17—C7—C8—N3	1.0 (3)	C17—C18—C19—C5	-1.6 (3)
C8—N3—C9—C10	-122.26 (18)	C6—C5—C19—C18	2.4 (3)
C8—N3—C9—C16	113.59 (19)	N2—C5—C19—C18	-174.51 (17)
N3—C9—C10—C15	-2.3 (2)	C3—C4—C20—C21	1.0 (3)
C16—C9—C10—C15	119.73 (19)	N1—C4—C20—C21	-176.29 (17)
N3—C9—C10—C11	176.36 (16)	C4—C20—C21—C1	-0.2 (3)
C16—C9—C10—C11	-61.6 (2)	C2—C1—C21—C20	-0.6 (3)
C15—C10—C11—C12	0.5 (3)		

Hydrogen-bond geometry (\AA , $^\circ$)

Cg1 and Cg2 are the centroids of the C1—C4/C20/C21 and C5—C7/C17—C19 rings, respectively.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N3—H3 \cdots O2	0.86	1.92	2.587 (2)	134
O1—H8 \cdots O2 ⁱ	0.93 (3)	1.79 (3)	2.708 (2)	168 (2)
C16—H9 \cdots O1 ⁱⁱ	0.97	2.45	3.355 (2)	156
C9—H11 \cdots O2 ⁱ	0.98	2.62	3.294 (2)	127
C14—H5 \cdots Cg2 ⁱⁱⁱ	0.93	3.20	3.722 (2)	118
C8—H12 \cdots Cg1 ^{iv}	0.93	2.75	3.458 (2)	134

C20—H16...Cg2 ^{iv}	0.93	2.89	3.480 (2)	122
C3—H18...Cg1 ^v	0.93	3.02	3.711 (2)	132

Symmetry codes: (i) $x, y-1, z$; (ii) $-x+1, y+1/2, -z+1$; (iii) $x-1, y, z$; (iv) $-x+1, y-1/2, -z$; (v) $-x+1, y+1/2, -z$.