Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,2′-[(*E*,*E*)-*cis*-(Cyclohexane-1,4-diyl)bis(nitrilomethanylylidene)]diphenol

Shaaban K. Mohamed,[a] Mehmet Akkurt,[b]* Muhammad N. Tahir[c] and Antar A. Abdelhamid[a]

[a]Chemistry and Environmental Division, Manchester Metropolitan University, Manchester M1 5GD, England, [b]Department of Physics, Faculty of Sciences, Erciyes University, 38039 Kayseri, Turkey, and [c]University of Sargodha, Department of Physics, Sargodha, Pakistan
Correspondence e-mail: akkurt@erciyes.edu.tr

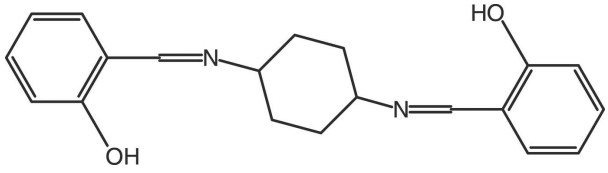
Received 21 May 2012; accepted 22 May 2012

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.050; wR factor = 0.140; data-to-parameter ratio = 15.7.

In the title compound, $C_{20}H_{22}N_2O_2$, the asymmetric unit contains two independent half-molecules, which are both completed by crystallographic inversion symmetry. The cyclohexane rings of both molecules adopt chair conformations; the N atoms are in equatorial orientations in one molecule and in axial orientations in the other. Both molecules feature two intramolecular O—H···N hydrogen bonds, which generate S(6) rings.

Related literature

For background to Schiff bases as ligands, see: Li & Zhang (2004).



Experimental

Crystal data

$C_{20}H_{22}N_2O_2$
M_r = 322.40
Monoclinic, $P2_1/n$
a = 16.2979 (11) Å
b = 6.1103 (4) Å
c = 18.2336 (12) Å
β = 104.975 (4)°
V = 1754.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 296 K
0.32 × 0.28 × 0.25 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.975, T_{max} = 0.980
12904 measured reflections
3428 independent reflections
1641 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.140
S = 1.01
3428 reflections
219 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.85	2.579 (2)	148
O2—H2A···N2	0.82	1.86	2.593 (3)	148

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

This project was sponsored by the General Association of Scholarships in Egypt. The University of Sargodha is gratefully acknowledged for The X-ray difraction measurements and the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6813).

References

Bruker (2005). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *APEX2* and S*AINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Li, Z.-X. & Zhang, X.-L. (2004). *Acta Cryst.* E**60**, m1017–m1019.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supplementary materials

Acta Cryst. (2012). E**68**, o1905 [doi:10.1107/S1600536812023367]

2,2′-[(*E*,*E*)-*cis*-(Cyclohexane-1,4-diyl)bis(nitrilomethanylylidene)]diphenol

Shaaban K. Mohamed, Mehmet Akkurt, Muhammad N. Tahir and Antar A. Abdelhamid

Comment

Schiff base compounds have been reported as excelent substrates in the development of coordination chemistry (e.g. Li & Zhang, 2004), In this study we report the synthesis and crystal structure of the title compound (I).

As shown in Fig. 1, there are two independent half molecules A (with C1) and B (with C11) in the asymmetric unit of the title compound. They are centrosymmetric and the centres of symmetry are lied on the centroids of their cyclohexane rings. The cyclohexane rings of them adopt chair conformations Molecular conformation of the title compound is stabilized by intramolecular O—H···N hydrogen bonds, generating an S(6) ring motif (Table 1, Fig. 2).

Experimental

The title compound arose as a bi-product from heating a reaction mixture of 114 mg (1 mmol) cyclohexane-1,4-diamine, 112 mg (1 mmol) cyclohexane-1,3-dione and 122 mg (1 mmol) salicylaldehyde in 50 ml e thanol under reflux for 6 h. The reaction mixture was concentrated under vacuum then left to cool at ambient temperature. The obtained solid was collected by Buckner funnel, washed with water then ethanol, dried in desiccator and crystallized from ethanol (m.p. 451 K). Yellow prisms were grown from ethanol solution by slow evaporation over two days.

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with O—H = 0.82 Å and C—H = 0.93 Å (aromatic), 0.97 Å (methylene) and 0.98 Å (methine), with U_{iso}(H) = 1.5U_{eq}(O) for OH groups and U_{iso}(H) = 1.2U_{eq}(C) for others.

Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

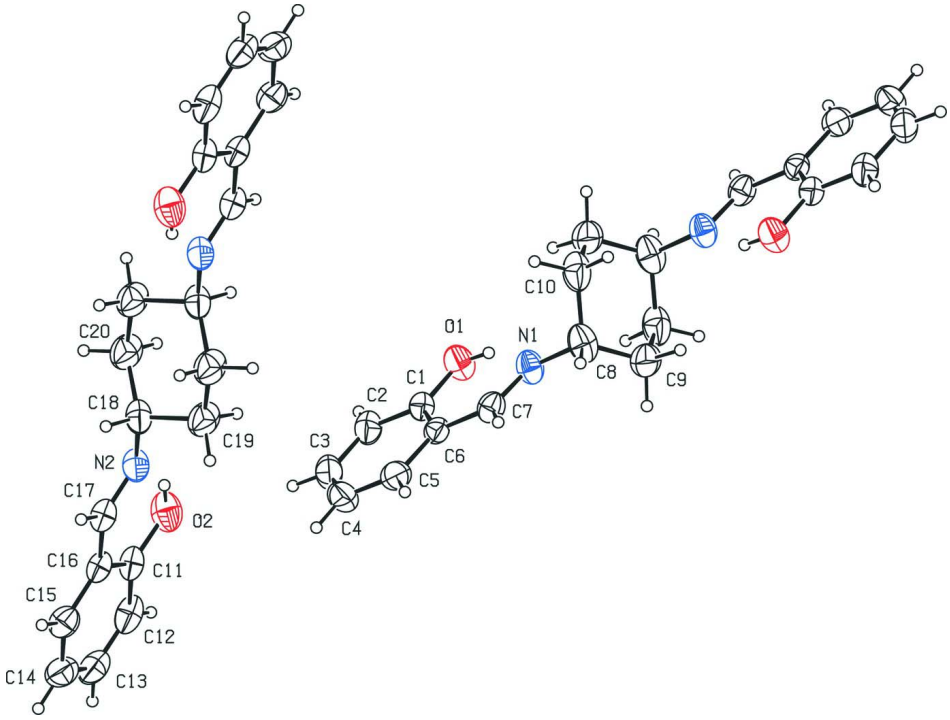


Figure 1

The molecular structure of the title compound, showing 30% probability ellipsoids.

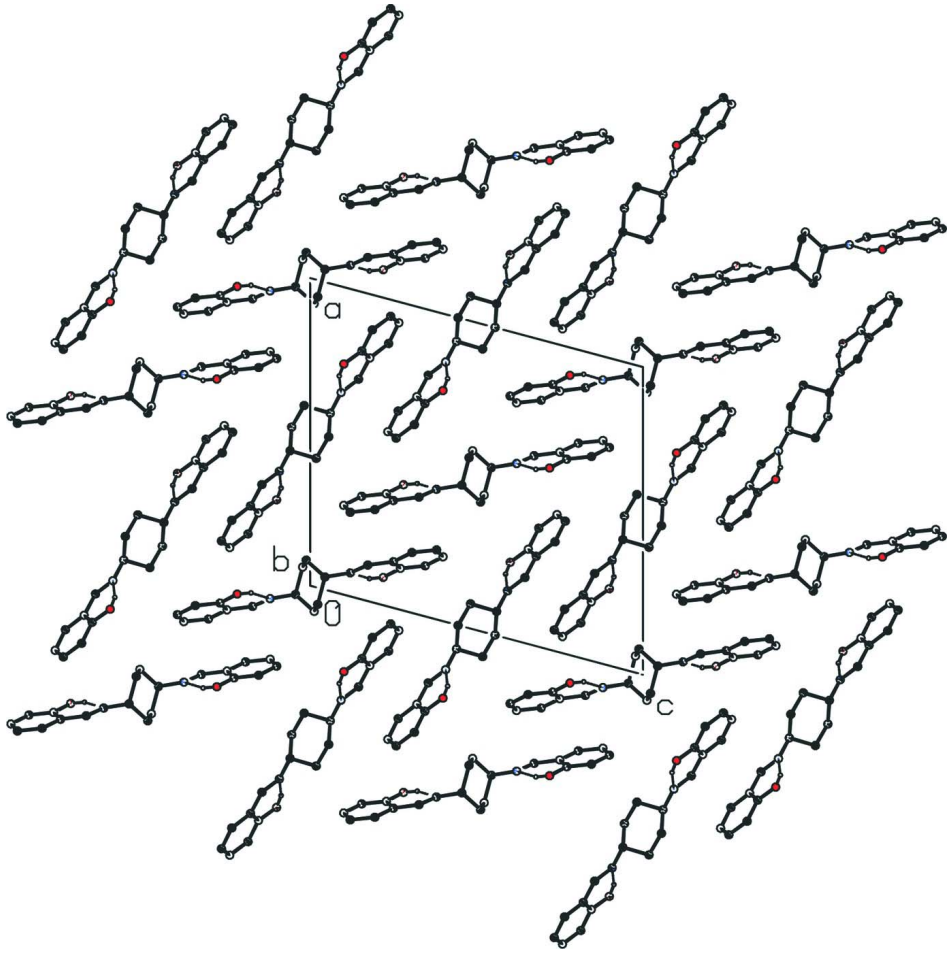


Figure 2

The crystal packing of the title compound, viewing along the *b* axis.

2,2′-[(*E,E*)-*cis*-(Cyclohexane-1,4- diyl)bis(nitrilomethanylylidene)]diphenol

Crystal data

$C_{20}H_{22}N_2O_2$
$M_r = 322.40$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 16.2979$ (11) Å
$b = 6.1103$ (4) Å
$c = 18.2336$ (12) Å
$\beta = 104.975$ (4)°
$V = 1754.1$ (2) Å^3
$Z = 4$
$F(000) = 688$
$D_x = 1.221$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 355 reflections
$\theta = 3.5$–18°
$\mu = 0.08$ mm^{-1}
$T = 296$ K
Prism, light yellow
0.32 × 0.28 × 0.25 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0.81 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.975$, $T_{max} = 0.980$
12904 measured reflections

3428 independent reflections
1641 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.3°$
$h = -20 \rightarrow 17$
$k = -7 \rightarrow 7$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.140$
$S = 1.01$
3428 reflections
219 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
$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 0.1881P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}

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	0.09064 (11)	−0.2099 (2)	0.21955 (9)	0.0798 (7)
N1	0.07675 (13)	0.0983 (3)	0.12044 (10)	0.0689 (8)
C1	0.13461 (14)	−0.0662 (4)	0.27110 (13)	0.0590 (9)
C2	0.16439 (15)	−0.1325 (4)	0.34525 (13)	0.0737 (10)
C3	0.20939 (17)	0.0070 (6)	0.39911 (15)	0.0845 (11)
C4	0.22627 (17)	0.2171 (6)	0.37967 (16)	0.0878 (14)
C5	0.19641 (15)	0.2847 (4)	0.30586 (15)	0.0732 (10)
C6	0.15061 (13)	0.1469 (4)	0.25003 (12)	0.0540 (8)
C7	0.11732 (14)	0.2225 (4)	0.17308 (13)	0.0629 (9)
C8	0.0412 (2)	0.1898 (4)	0.04433 (14)	0.0798 (12)
C9	0.07984 (17)	0.0747 (5)	−0.01193 (15)	0.0893 (13)
C10	−0.05416 (19)	0.1620 (5)	0.02264 (14)	0.0895 (13)
O2	0.24999 (12)	−0.0079 (3)	0.60105 (10)	0.0906 (8)
N2	0.15493 (13)	0.3363 (3)	0.59279 (11)	0.0689 (8)
C11	0.30811 (17)	0.0985 (4)	0.65534 (13)	0.0658 (10)
C12	0.3858 (2)	−0.0030 (4)	0.68695 (16)	0.0789 (11)
C13	0.44577 (18)	0.1016 (5)	0.74179 (17)	0.0835 (12)
C14	0.43165 (18)	0.3068 (5)	0.76653 (15)	0.0827 (12)
C15	0.35547 (17)	0.4075 (4)	0.73567 (14)	0.0726 (10)
C16	0.29237 (15)	0.3078 (4)	0.67939 (13)	0.0594 (9)
C17	0.21310 (16)	0.4199 (4)	0.64521 (13)	0.0639 (9)
C18	0.07848 (17)	0.4641 (4)	0.56020 (12)	0.0707 (10)

C19	0.08057 (16)	0.5442 (5)	0.48189 (14)	0.0877 (11)
C20	0.00025 (17)	0.3308 (5)	0.55562 (15)	0.0893 (11)
H1	0.07470	−0.14950	0.17810	0.0960*
H2	0.15370	−0.27440	0.35880	0.0880*
H3	0.22880	−0.03980	0.44920	0.1020*
H4	0.25760	0.31200	0.41620	0.1050*
H5	0.20730	0.42720	0.29300	0.0880*
H7	0.12590	0.36770	0.16170	0.0760*
H8	0.05480	0.34610	0.04490	0.0960*
H9A	0.06250	0.14910	−0.06050	0.1070*
H9B	0.14120	0.08380	0.00550	0.1070*
H10A	−0.07710	0.22480	0.06200	0.1070*
H10B	−0.07820	0.24110	−0.02410	0.1070*
H2A	0.20730	0.06790	0.58720	0.1090*
H12	0.39670	−0.14170	0.67070	0.0950*
H13	0.49730	0.03250	0.76290	0.1000*
H14	0.47330	0.37660	0.80380	0.0990*
H15	0.34570	0.54600	0.75280	0.0870*
H17	0.20470	0.55910	0.66260	0.0770*
H18	0.07770	0.59130	0.59280	0.0850*
H19A	0.08650	0.42010	0.45050	0.1050*
H19B	0.12940	0.63890	0.48620	0.1050*
H20A	−0.00150	0.28800	0.60640	0.1070*
H20B	0.00280	0.19850	0.52690	0.1070*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1064 (14)	0.0639 (10)	0.0648 (11)	−0.0200 (10)	0.0146 (10)	0.0018 (8)
N1	0.0927 (15)	0.0592 (12)	0.0533 (12)	−0.0029 (11)	0.0160 (10)	0.0030 (10)
C1	0.0618 (15)	0.0655 (16)	0.0526 (14)	−0.0007 (13)	0.0202 (11)	−0.0056 (12)
C2	0.0842 (19)	0.0826 (18)	0.0583 (16)	0.0068 (15)	0.0257 (13)	0.0046 (14)
C3	0.084 (2)	0.114 (2)	0.0565 (17)	0.0167 (18)	0.0201 (14)	−0.0041 (17)
C4	0.0716 (19)	0.114 (3)	0.073 (2)	−0.0034 (17)	0.0099 (15)	−0.0310 (18)
C5	0.0669 (17)	0.0738 (17)	0.0834 (19)	−0.0097 (13)	0.0274 (14)	−0.0203 (15)
C6	0.0537 (14)	0.0567 (14)	0.0558 (14)	−0.0014 (11)	0.0217 (11)	−0.0057 (12)
C7	0.0736 (17)	0.0543 (14)	0.0672 (16)	−0.0008 (12)	0.0296 (13)	0.0013 (13)
C8	0.120 (3)	0.0562 (15)	0.0589 (16)	0.0001 (16)	0.0152 (16)	0.0100 (13)
C9	0.083 (2)	0.120 (3)	0.0661 (18)	−0.0034 (18)	0.0212 (14)	0.0239 (17)
C10	0.107 (3)	0.100 (2)	0.0625 (17)	0.0366 (19)	0.0238 (16)	−0.0014 (15)
O2	0.1192 (16)	0.0754 (12)	0.0774 (13)	0.0202 (11)	0.0259 (11)	−0.0102 (10)
N2	0.0781 (15)	0.0755 (14)	0.0541 (12)	0.0147 (12)	0.0189 (10)	0.0012 (11)
C11	0.085 (2)	0.0656 (17)	0.0548 (15)	0.0085 (15)	0.0327 (14)	0.0045 (13)
C12	0.101 (2)	0.0716 (18)	0.0787 (19)	0.0263 (18)	0.0497 (17)	0.0179 (15)
C13	0.073 (2)	0.104 (2)	0.084 (2)	0.0205 (18)	0.0391 (17)	0.0301 (18)
C14	0.067 (2)	0.097 (2)	0.088 (2)	−0.0028 (17)	0.0271 (15)	0.0135 (17)
C15	0.0768 (19)	0.0668 (16)	0.0809 (18)	−0.0025 (15)	0.0324 (15)	0.0048 (14)
C16	0.0693 (17)	0.0588 (15)	0.0594 (15)	0.0058 (13)	0.0335 (13)	0.0077 (12)
C17	0.0772 (18)	0.0605 (15)	0.0622 (16)	0.0092 (14)	0.0328 (13)	0.0061 (13)
C18	0.0829 (19)	0.0775 (17)	0.0530 (15)	0.0188 (16)	0.0202 (12)	0.0007 (13)

C19	0.0816 (19)	0.109 (2)	0.0774 (19)	0.0142 (17)	0.0296 (14)	0.0299 (16)
C20	0.089 (2)	0.104 (2)	0.0797 (19)	0.0131 (19)	0.0304 (15)	0.0309 (16)

Geometric parameters (Å, º)

O1—C1	1.349 (3)	C9—H9A	0.9700
O1—H1	0.8200	C9—H9B	0.9700
O2—C11	1.347 (3)	C10—H10B	0.9700
O2—H2A	0.8200	C10—H10A	0.9700
N1—C7	1.267 (3)	C11—C16	1.397 (3)
N1—C8	1.469 (3)	C11—C12	1.394 (4)
N2—C18	1.460 (3)	C12—C13	1.363 (4)
N2—C17	1.267 (3)	C13—C14	1.372 (4)
C1—C6	1.401 (3)	C14—C15	1.370 (4)
C1—C2	1.374 (3)	C15—C16	1.392 (4)
C2—C3	1.363 (4)	C16—C17	1.453 (4)
C3—C4	1.378 (5)	C18—C20	1.497 (4)
C4—C5	1.371 (4)	C18—C19	1.518 (3)
C5—C6	1.382 (3)	C19—C20^{ii}	1.523 (4)
C6—C7	1.443 (3)	C12—H12	0.9300
C8—C10	1.511 (5)	C13—H13	0.9300
C8—C9	1.509 (4)	C14—H14	0.9300
C9—C10^{i}	1.504 (4)	C15—H15	0.9300
C2—H2	0.9300	C17—H17	0.9300
C3—H3	0.9300	C18—H18	0.9800
C4—H4	0.9300	C19—H19A	0.9700
C5—H5	0.9300	C19—H19B	0.9700
C7—H7	0.9300	C20—H20A	0.9700
C8—H8	0.9800	C20—H20B	0.9700
C1—O1—H1	109.00	H10A—C10—H10B	108.00
C11—O2—H2A	109.00	C8—C10—H10A	109.00
C7—N1—C8	119.3 (2)	O2—C11—C12	118.7 (2)
C17—N2—C18	119.0 (2)	C12—C11—C16	119.9 (2)
O1—C1—C2	118.9 (2)	O2—C11—C16	121.4 (2)
C2—C1—C6	120.0 (2)	C11—C12—C13	119.8 (2)
O1—C1—C6	121.1 (2)	C12—C13—C14	121.4 (3)
C1—C2—C3	120.8 (2)	C13—C14—C15	119.1 (3)
C2—C3—C4	120.2 (3)	C14—C15—C16	121.7 (2)
C3—C4—C5	119.3 (3)	C11—C16—C15	118.1 (2)
C4—C5—C6	121.8 (3)	C11—C16—C17	120.8 (2)
C1—C6—C5	117.9 (2)	C15—C16—C17	121.1 (2)
C1—C6—C7	120.9 (2)	N2—C17—C16	122.9 (2)
C5—C6—C7	121.1 (2)	N2—C18—C19	109.3 (2)
N1—C7—C6	122.4 (2)	N2—C18—C20	110.9 (2)
N1—C8—C10	109.4 (2)	C19—C18—C20	110.4 (2)
C9—C8—C10	110.6 (2)	C18—C19—C20^{ii}	110.9 (2)
N1—C8—C9	109.4 (2)	C18—C20—C19^{ii}	112.2 (2)
C8—C9—C10^{i}	112.7 (2)	C11—C12—H12	120.00
C8—C10—C9^{i}	112.0 (2)	C13—C12—H12	120.00

C1—C2—H2	120.00	C12—C13—H13	119.00
C3—C2—H2	120.00	C14—C13—H13	119.00
C4—C3—H3	120.00	C13—C14—H14	120.00
C2—C3—H3	120.00	C15—C14—H14	120.00
C3—C4—H4	120.00	C14—C15—H15	119.00
C5—C4—H4	120.00	C16—C15—H15	119.00
C6—C5—H5	119.00	N2—C17—H17	119.00
C4—C5—H5	119.00	C16—C17—H17	119.00
C6—C7—H7	119.00	N2—C18—H18	109.00
N1—C7—H7	119.00	C19—C18—H18	109.00
C9—C8—H8	109.00	C20—C18—H18	109.00
C10—C8—H8	109.00	C18—C19—H19A	109.00
N1—C8—H8	109.00	C18—C19—H19B	109.00
C8—C9—H9B	109.00	H19A—C19—H19B	108.00
C8—C9—H9A	109.00	C20^{ii}—C19—H19A	109.00
C10^{i}—C9—H9B	109.00	C20^{ii}—C19—H19B	109.00
H9A—C9—H9B	108.00	C18—C20—H20A	109.00
C10^{i}—C9—H9A	109.00	C18—C20—H20B	109.00
C8—C10—H10B	109.00	H20A—C20—H20B	108.00
C9^{i}—C10—H10A	109.00	C19^{ii}—C20—H20A	109.00
C9^{i}—C10—H10B	109.00	C19^{ii}—C20—H20B	109.00

C8—N1—C7—C6	177.0 (2)	N1—C8—C10—C9^{i}	−67.2 (3)
C7—N1—C8—C9	119.4 (3)	C9—C8—C10—C9^{i}	53.4 (3)
C7—N1—C8—C10	−119.3 (3)	C8—C9—C10^{i}—C8^{i}	54.6 (3)
C18—N2—C17—C16	−178.1 (2)	O2—C11—C12—C13	179.8 (3)
C17—N2—C18—C19	105.8 (3)	C16—C11—C12—C13	0.7 (4)
C17—N2—C18—C20	−132.2 (2)	O2—C11—C16—C15	−179.9 (2)
O1—C1—C2—C3	179.7 (2)	O2—C11—C16—C17	−1.4 (4)
C6—C1—C2—C3	0.2 (4)	C12—C11—C16—C15	−0.9 (4)
O1—C1—C6—C5	−179.7 (2)	C12—C11—C16—C17	177.7 (2)
C2—C1—C6—C7	−177.7 (2)	C11—C12—C13—C14	−0.4 (5)
O1—C1—C6—C7	2.8 (3)	C12—C13—C14—C15	0.4 (4)
C2—C1—C6—C5	−0.2 (3)	C13—C14—C15—C16	−0.6 (4)
C1—C2—C3—C4	−0.5 (4)	C14—C15—C16—C11	0.8 (4)
C2—C3—C4—C5	0.8 (4)	C14—C15—C16—C17	−177.7 (2)
C3—C4—C5—C6	−0.8 (4)	C11—C16—C17—N2	0.0 (4)
C4—C5—C6—C1	0.5 (4)	C15—C16—C17—N2	178.5 (2)
C4—C5—C6—C7	178.0 (2)	N2—C18—C19—C20^{ii}	177.3 (2)
C1—C6—C7—N1	−4.2 (4)	C20—C18—C19—C20^{ii}	55.0 (3)
C5—C6—C7—N1	178.4 (2)	N2—C18—C20—C19^{ii}	−177.1 (2)
C10—C8—C9—C10^{i}	−53.8 (3)	C19—C18—C20—C19^{ii}	−55.7 (3)
N1—C8—C9—C10^{i}	66.8 (3)	C18—C19—C20^{ii}—C18^{ii}	−56.0 (3)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.85	2.579 (2)	148

O2—H2*A*···N2	0.82	1.86	2.593 (3)	148