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(*Z*)-4-[(*E*)-(4-Butylphenyl)diazenyl]-6-{[1,3-dihydroxy-2-(hydroxymethyl)propan-2-ylamino]methylene}-2-methoxycyclohexa-2,4-dienone

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.083
wR factor = 0.250
Data-to-parameter ratio = 16.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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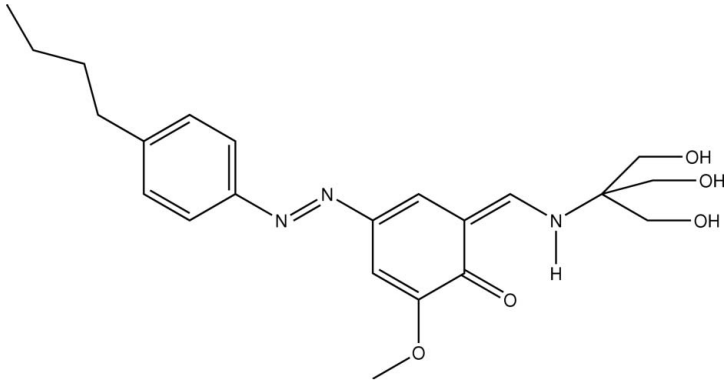
The title compound, $C_{22}H_{29}N_3O_5$, is approximately planar with the aromatic rings in a *trans* configuration with respect to the azo double bond. The eneamine portion of the molecule exists as the keto–amine tautomer, stabilized by a strong intramolecular N—H···O hydrogen bond. Intramolecular N—H···O and O—H···O and intermolecular O—H···O and C—H···O interactions influence the conformation of the molecules and the crystal packing.

Comment

Azo compounds are the most widely used class of dyes, owing to their versatile application in various fields, such as dyeing and colouring, and high-technology areas, such as electro-optical devices and ink-jet printers (Peters & Freeman, 1991). There is interest in Schiff base ligands and their complexes with regard to their impressive antitumour activities (Zhou *et al.*, 2000). Schiff base compounds can be classified by their photochromic and thermochromic characteristics (Cohen *et al.*, 1964; Moustakali *et al.*, 1978; Hadjoudis *et al.*, 1987). On the basis of studies of some thermochromic and photochromic Schiff base compounds, it has been proposed that molecules exhibiting thermochromism are planar, while those exhibiting photochromism are non-planar (Moustakali *et al.*, 1978).



(I)

We report here the structure of the Schiff base compound, (I) (Fig. 1 and Table 1). There are two possible tautomers that this molecule could adopt, *viz*. the keto–amine and enol–imine tautomeric forms. The structural data clearly show that (I) adopts the keto–amine form, stabilized by the formation of an N—H···O hydrogen bond. Similar *o*-hydroxy Schiff bases have been found to adopt the keto (Ersanlı *et al.*, 2003; Ersanlı, Albayrak, Odabaşoğlu, Thöne & Erdönmez, 2004; Ersanlı *et al.*, 2005), enol (Ersanlı, Albayrak, Odabaşoğlu & Erdönmez, 2004; Ersanlı, Odabaşoğlu *et al.*, 2004) or enol/keto

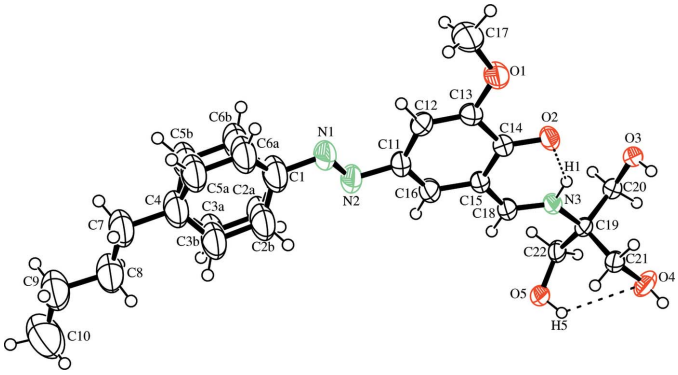


Figure 1
A view of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids. There is orientational disorder in the C1–C6 benzene ring; occupancy factors for atoms C2a/C3a/C5a/C6a and C2b/C3b/C5b/C6b are 0.617 (6) and 0.383 (6), respectively. Hydrogen bonds are shown as dashed lines.

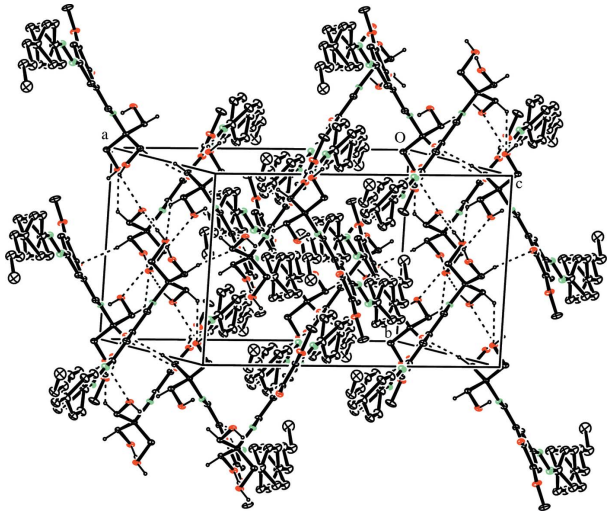


Figure 2
Packing diagram for (I), with hydrogen bonds drawn as dashed lines. For the sake of clarity, all H atoms, except for H3, H4, H5, H20*B* and H22*B*, have been omitted.

forms (Nazır *et al.*, 2000). The C14—O2 and C18—N3 bond distances (Table 1) are reasonably similar, a characteristic of systems in the keto–amine form, and comparable to those found in 4-[(2-chlorophenyl)diazenyl]-6-methoxy-2-{[tris-(hydroxymethyl)methyl]aminomethylene}cyclohexa-3,5-dien-1(2*H*)-one [1.287 (2) and 1.294 (2) Å; Ersanlı, Albayrak, Odabaşoğlu, Thöne & Erdönmez, 2004]. In contrast, the corresponding distances are significantly different in systems known to adopt the enol-imine tautomeric form, with a much longer C—O bond, for example, 1.354 (2) and 1.275 (2) Å in 2-[2-(hydroxymethyl)phenyliminomethyl]phenol (Ersanlı, Odabaşoğlu *et al.*, 2004), and 1.355 (2) and 1.278 (2) Å in 4-(2-hydroxyphenyliminomethylene)phenol (Ersanlı, Albayrak, Odabaşoğlu & Erdönmez, 2004).

Intramolecular N3—H1···O2 and C22—O5—H5···O4 hydrogen bonds influence the conformation of the molecule (Table 2). The packing is also stabilized by a number of intermolecular O—H···O and C—H···O interactions (Fig. 2).

Experimental

The title compound, (I), was prepared as previously described (Ersanlı, Albayrak, Odabaşoğlu, Thöne & Erdönmez, 2004) The product was recrystallized from ethanol and well shaped crystals were obtained by slow evaporation of an acetonitrile solution (m.p. 461–463 K).

Crystal data

$C_{22}H_{29}N_3O_5$
M_r = 415.48
Monoclinic, $P2_1/c$
a = 18.3934 (13) Å
b = 10.6960 (7) Å
c = 10.8516 (8) Å
β = 95.488 (6)°
V = 2125.1 (3) Å^3
Z = 4
D_x = 1.299 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 17039 reflections
θ = 1.9–27.9°
μ = 0.09 mm^{-1}
T = 296 (2) K
Plate, orange
0.30 × 0.20 × 0.05 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.975, T_{max} = 0.996
23762 measured reflections
4173 independent reflections
2194 reflections with $I > 2\sigma(I)$
R_{int} = 0.097
θ_{max} = 26.0°
$h = -22 \rightarrow 22$
$k = -13 \rightarrow 13$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.083
$wR(F^2)$ = 0.250
S = 1.02
4173 reflections
252 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1495P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.427 (7)	C18—N3	1.289 (5)
C4—C7	1.525 (8)	C20—O3	1.422 (5)
C11—N2	1.406 (6)	C21—O4	1.416 (5)
C13—O1	1.363 (5)	C22—O5	1.416 (5)
C14—O2	1.276 (5)	N1—N2	1.238 (5)
C17—O1	1.423 (6)		
C16—C11—N2	116.6 (4)	O2—C14—C13	120.3 (3)
N2—C11—C12	123.9 (4)	N3—C18—C15	122.7 (4)
C12—C13—O1	125.9 (4)	N1—N2—C11	115.5 (4)
O2—C14—C15	123.2 (3)	C18—N3—C19	129.2 (3)
C1—N1—N2—C11	−175.6 (5)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O3^{i}	0.82	2.03	2.769 (4)	150
O3—H3···O4^{i}	0.82	2.00	2.769 (4)	155
O5—H5···O3^{ii}	0.82	2.37	2.771 (3)	111
C20—H20*B*···O4^{iii}	0.97	2.55	3.310 (5)	135
C22—H22*B*···O2^{ii}	0.97	2.42	3.375 (5)	169
N3—H1···O2	0.88 (5)	1.88 (5)	2.603 (4)	138 (4)
O5—H5···O4	0.82	2.56	3.088 (4)	124

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

The crystals were weakly diffracting but attempts to find one of better quality were not successful. Furthermore, high displacement parameters for atoms in the benzene ring suggested possible disorder. This was resolved by refining alternative positions for the unsubstituted atoms C2, C3, C5 and C6 of the benzene ring. Their occupancies refined to 0.617 (6) and 0.383 (6), respectively. All H atoms bound to C atoms were refined using a riding model [C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic C atoms, C—H = 0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for methylene C atoms, and C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl C atoms]. The H atoms of the hydroxy O atoms were refined with O—H constrained to 0.82 Å and with $U_{iso}(H) = 1.5U_{eq}(O)$. The H atom bonded to N was refined freely, with $U_{iso}(H) = 1.2U_{eq}(N)$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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supporting information

Acta Cryst. (2005). E**61**, o4139–o4141 [https://doi.org/10.1107/S1600536805037177]

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(*Z*)-4-((*E*)-(4-butylphenyl)diazenyl)-6-((1,3-dihydroxy-2-(hydroxymethyl)p ropan- 2-ylamino)methylene)-2-methoxycyclohexa-2,4-dienone

Crystal data

$C_{22}H_{29}N_3O_5$
$M_r = 415.48$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 18.3934\ (13)$ Å
$b = 10.6960\ (7)$ Å
$c = 10.8516\ (8)$ Å
$\beta = 95.488\ (6)°$
$V = 2125.1\ (3)$ Å^3
$Z = 4$
$F(000) = 888$
$D_x = 1.299$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 17039 reflections
$\theta = 1.9–27.9°$
$\mu = 0.09$ mm^{-1}
$T = 296$ K
Plate, orange
0.30 × 0.20 × 0.05 mm

Data collection

STOE IPDS-II diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 6.67 pixels mm^{-1}
ω scans
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)
$T_{min} = 0.975$, $T_{max} = 0.996$
23762 measured reflections
4173 independent reflections
2194 reflections with $I > 2\sigma(I)$
$R_{int} = 0.097$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.2°$
$h = -22\rightarrow22$
$k = -13\rightarrow13$
$l = -13\rightarrow13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.083$
$wR(F^2) = 0.250$
$S = 1.02$
4173 reflections
252 parameters
8 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
$w = 1/[\sigma^2(F_o^2) + (0.1495P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.52$ e Å^{-3}
$\Delta\rho_{min} = -0.52$ 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}	Occ. (<1)
C1	0.5721 (3)	0.6142 (6)	0.4063 (6)	0.0827 (9)	
C4	0.4512 (3)	0.6396 (6)	0.2310 (6)	0.0827 (9)	
C2B	0.5346 (6)	0.5174 (9)	0.3560 (10)	0.0827 (9)	0.617 (6)
H2B	0.5499	0.4376	0.3806	0.099*	0.617 (6)
C2A	0.5653 (9)	0.5243 (15)	0.3030 (16)	0.0827 (9)	0.383 (6)
H2A	0.6002	0.4625	0.2958	0.099*	0.383 (6)
C3B	0.4738 (6)	0.5274 (10)	0.2690 (10)	0.0827 (9)	0.617 (6)
H3B	0.4495	0.4561	0.2382	0.099*	0.617 (6)
C3A	0.5053 (9)	0.5352 (13)	0.2173 (16)	0.0827 (9)	0.383 (6)
H3A	0.4982	0.4790	0.1519	0.099*	0.383 (6)
C5A	0.4606 (6)	0.7126 (9)	0.3305 (10)	0.0827 (9)	0.617 (6)
H5A	0.4256	0.7733	0.3417	0.099*	0.617 (6)
C5B	0.4909 (9)	0.7377 (14)	0.2644 (16)	0.0827 (9)	0.383 (6)
H5B	0.4799	0.8145	0.2271	0.099*	0.383 (6)
C6A	0.5206 (5)	0.7023 (9)	0.4190 (10)	0.0827 (9)	0.617 (6)
H6A	0.5251	0.7560	0.4868	0.099*	0.617 (6)
C6B	0.5514 (8)	0.7278 (13)	0.3583 (14)	0.0827 (9)	0.383 (6)
H6B	0.5764	0.7994	0.3864	0.099*	0.383 (6)
C7	0.3844 (3)	0.6545 (6)	0.1375 (6)	0.0827 (9)	
H7A	0.3412	0.6573	0.1822	0.099*	
H7B	0.3879	0.7343	0.0958	0.099*	
C8	0.3742 (4)	0.5531 (7)	0.0407 (6)	0.092 (2)	
H8A	0.3682	0.4738	0.0819	0.110*	
H8B	0.4182	0.5477	−0.0017	0.110*	
C9	0.3096 (4)	0.5723 (7)	−0.0549 (7)	0.099 (2)	
H9A	0.2664	0.5864	−0.0120	0.119*	
H9B	0.3180	0.6473	−0.1017	0.119*	
C10	0.2947 (6)	0.4670 (10)	−0.1432 (10)	0.143 (4)	
H10A	0.2531	0.4869	−0.2001	0.215*	
H10B	0.2849	0.3925	−0.0983	0.215*	
H10C	0.3365	0.4537	−0.1883	0.215*	
C11	0.7252 (2)	0.4940 (4)	0.5897 (4)	0.0476 (10)	
C12	0.7383 (2)	0.5769 (3)	0.6916 (4)	0.0465 (10)	
H12	0.7119	0.6509	0.6936	0.056*	
C13	0.7892 (2)	0.5484 (3)	0.7860 (4)	0.0436 (10)	

C14	0.8290 (2)	0.4321 (3)	0.7903 (4)	0.0386 (9)
C15	0.8195 (2)	0.3556 (3)	0.6825 (4)	0.0388 (9)
C16	0.7672 (3)	0.3887 (4)	0.5841 (4)	0.0490 (11)
H16	0.7613	0.3381	0.5142	0.059*
C17	0.7787 (4)	0.7442 (5)	0.8885 (6)	0.092 (2)
H17A	0.7963	0.7852	0.9642	0.138*
H17B	0.7263	0.7393	0.8830	0.138*
H17C	0.7933	0.7909	0.8195	0.138*
C18	0.8620 (2)	0.2461 (3)	0.6740 (4)	0.0426 (10)
H18	0.8552	0.1981	0.6023	0.051*
C19	0.9597 (2)	0.1020 (3)	0.7650 (3)	0.0341 (8)
C20	1.0352 (2)	0.1500 (3)	0.8124 (4)	0.0387 (9)
H20A	1.0673	0.0793	0.8320	0.046*
H20B	1.0551	0.1978	0.7475	0.046*
C21	0.9295 (2)	0.0091 (3)	0.8547 (4)	0.0401 (9)
H21A	0.9281	0.0470	0.9356	0.048*
H21B	0.8803	−0.0159	0.8244	0.048*
C22	0.9662 (2)	0.0438 (3)	0.6383 (3)	0.0394 (9)
H22A	0.9687	0.1103	0.5780	0.047*
H22B	1.0115	−0.0030	0.6412	0.047*
N1	0.6307 (2)	0.6045 (4)	0.5019 (4)	0.0621 (11)
N2	0.6700 (2)	0.5117 (3)	0.4923 (4)	0.0574 (10)
N3	0.90972 (19)	0.2101 (3)	0.7615 (3)	0.0379 (8)
O1	0.8084 (2)	0.6214 (3)	0.8869 (3)	0.0635 (10)
O2	0.87152 (16)	0.4034 (2)	0.8860 (2)	0.0436 (7)
O3	1.03352 (16)	0.2263 (2)	0.9193 (2)	0.0434 (7)
H3	1.0175	0.1858	0.9748	0.065*
O4	0.97655 (19)	−0.0960 (2)	0.8625 (3)	0.0549 (9)
H4	0.9617	−0.1475	0.9101	0.082*
O5	0.90756 (17)	−0.0366 (2)	0.5983 (2)	0.0453 (7)
H5	0.9057	−0.0942	0.6478	0.068*
H1	0.915 (2)	0.259 (4)	0.827 (4)	0.048 (12)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.072 (2)	0.0913 (17)	0.079 (2)	0.0263 (15)	−0.0246 (15)	−0.0076 (15)
C4	0.072 (2)	0.0913 (17)	0.079 (2)	0.0263 (15)	−0.0246 (15)	−0.0076 (15)
C2B	0.072 (2)	0.0913 (17)	0.079 (2)	0.0263 (15)	−0.0246 (15)	−0.0076 (15)
C2A	0.072 (2)	0.0913 (17)	0.079 (2)	0.0263 (15)	−0.0246 (15)	−0.0076 (15)
C3B	0.072 (2)	0.0913 (17)	0.079 (2)	0.0263 (15)	−0.0246 (15)	−0.0076 (15)
C3A	0.072 (2)	0.0913 (17)	0.079 (2)	0.0263 (15)	−0.0246 (15)	−0.0076 (15)
C5A	0.072 (2)	0.0913 (17)	0.079 (2)	0.0263 (15)	−0.0246 (15)	−0.0076 (15)
C5B	0.072 (2)	0.0913 (17)	0.079 (2)	0.0263 (15)	−0.0246 (15)	−0.0076 (15)
C6A	0.072 (2)	0.0913 (17)	0.079 (2)	0.0263 (15)	−0.0246 (15)	−0.0076 (15)
C6B	0.072 (2)	0.0913 (17)	0.079 (2)	0.0263 (15)	−0.0246 (15)	−0.0076 (15)
C7	0.072 (2)	0.0913 (17)	0.079 (2)	0.0263 (15)	−0.0246 (15)	−0.0076 (15)
C8	0.080 (4)	0.109 (5)	0.083 (4)	0.017 (4)	−0.019 (4)	0.008 (4)

C9	0.099 (5)	0.109 (5)	0.082 (4)	0.027 (4)	−0.028 (4)	−0.011 (4)
C10	0.129 (8)	0.172 (9)	0.120 (7)	−0.003 (7)	−0.038 (6)	−0.024 (6)
C11	0.048 (3)	0.046 (2)	0.046 (2)	0.0056 (18)	−0.013 (2)	0.0023 (17)
C12	0.050 (3)	0.0343 (18)	0.053 (3)	0.0098 (17)	−0.006 (2)	−0.0011 (17)
C13	0.047 (2)	0.0365 (18)	0.046 (2)	0.0023 (17)	−0.004 (2)	−0.0054 (17)
C14	0.040 (2)	0.0366 (18)	0.038 (2)	−0.0015 (16)	−0.0045 (18)	0.0018 (15)
C15	0.043 (2)	0.0349 (17)	0.037 (2)	0.0026 (16)	−0.0079 (18)	−0.0012 (15)
C16	0.059 (3)	0.042 (2)	0.043 (2)	0.0067 (19)	−0.012 (2)	−0.0054 (17)
C17	0.107 (5)	0.055 (3)	0.104 (5)	0.030 (3)	−0.036 (4)	−0.034 (3)
C18	0.052 (3)	0.0365 (19)	0.037 (2)	0.0027 (17)	−0.0098 (19)	−0.0012 (16)
C19	0.043 (2)	0.0245 (15)	0.0335 (19)	0.0048 (14)	−0.0051 (17)	0.0005 (13)
C20	0.044 (2)	0.0326 (17)	0.038 (2)	0.0016 (16)	−0.0045 (18)	0.0032 (15)
C21	0.054 (3)	0.0327 (17)	0.033 (2)	0.0017 (17)	−0.0035 (19)	0.0010 (14)
C22	0.053 (2)	0.0358 (17)	0.0282 (19)	0.0069 (17)	−0.0043 (18)	−0.0001 (14)
N1	0.055 (2)	0.058 (2)	0.069 (3)	0.0148 (19)	−0.018 (2)	0.0006 (19)
N2	0.059 (2)	0.053 (2)	0.056 (2)	0.0144 (18)	−0.014 (2)	0.0025 (17)
N3	0.045 (2)	0.0312 (15)	0.0348 (18)	0.0046 (13)	−0.0078 (16)	−0.0035 (13)
O1	0.082 (2)	0.0449 (16)	0.058 (2)	0.0206 (15)	−0.0205 (18)	−0.0186 (14)
O2	0.0495 (17)	0.0423 (14)	0.0358 (15)	0.0062 (12)	−0.0120 (13)	−0.0053 (11)
O3	0.0602 (19)	0.0335 (12)	0.0342 (14)	−0.0064 (12)	−0.0086 (13)	0.0022 (10)
O4	0.086 (2)	0.0323 (13)	0.0466 (17)	0.0128 (14)	0.0056 (16)	0.0103 (12)
O5	0.065 (2)	0.0318 (12)	0.0366 (15)	−0.0006 (12)	−0.0104 (14)	0.0001 (10)

Geometric parameters (Å, º)

C1—C2B	1.332 (11)	C11—N2	1.406 (6)
C1—C6A	1.354 (11)	C11—C12	1.420 (6)
C1—C6B	1.363 (15)	C12—C13	1.355 (6)
C1—N1	1.427 (7)	C12—H12	0.9300
C1—C2A	1.473 (18)	C13—O1	1.363 (5)
C4—C5B	1.310 (17)	C13—C14	1.442 (5)
C4—C3B	1.323 (11)	C14—O2	1.276 (5)
C4—C5A	1.331 (11)	C14—C15	1.425 (5)
C4—C3A	1.512 (16)	C15—C16	1.411 (6)
C4—C7	1.525 (8)	C15—C18	1.417 (5)
C2B—C3B	1.397 (13)	C16—H16	0.9300
C2B—H2B	0.9300	C17—O1	1.423 (6)
C2A—C3A	1.38 (2)	C17—H17A	0.9600
C2A—H2A	0.9300	C17—H17B	0.9600
C3B—H3B	0.9300	C17—H17C	0.9600
C3A—H3A	0.9300	C18—N3	1.289 (5)
C5A—C6A	1.395 (13)	C18—H18	0.9300
C5A—H5A	0.9300	C19—N3	1.476 (5)
C5B—C6B	1.44 (2)	C19—C20	1.523 (6)
C5B—H5B	0.9300	C19—C22	1.524 (5)
C6A—H6A	0.9300	C19—C21	1.532 (5)
C6B—H6B	0.9300	C20—O3	1.422 (5)
C7—C8	1.509 (9)	C20—H20A	0.9700

C7—H7A	0.9700	C20—H20B	0.9700
C7—H7B	0.9700	C21—O4	1.416 (5)
C8—C9	1.513 (9)	C21—H21A	0.9700
C8—H8A	0.9700	C21—H21B	0.9700
C8—H8B	0.9700	C22—O5	1.416 (5)
C9—C10	1.487 (11)	C22—H22A	0.9700
C9—H9A	0.9700	C22—H22B	0.9700
C9—H9B	0.9700	N1—N2	1.238 (5)
C10—H10A	0.9600	N3—H1	0.88 (5)
C10—H10B	0.9600	O3—H3	0.8200
C10—H10C	0.9600	O4—H4	0.8200
C11—C16	1.372 (6)	O5—H5	0.8200

C2B—C1—C6A	104.0 (7)	H10A—C10—H10C	109.5
C2B—C1—C6B	114.9 (9)	H10B—C10—H10C	109.5
C2B—C1—N1	124.6 (6)	C16—C11—N2	116.6 (4)
C6A—C1—N1	117.6 (6)	C16—C11—C12	119.5 (4)
C6B—C1—N1	120.5 (8)	N2—C11—C12	123.9 (4)
C6A—C1—C2A	121.5 (9)	C13—C12—C11	120.2 (4)
C6B—C1—C2A	106.9 (9)	C13—C12—H12	119.9
N1—C1—C2A	120.7 (7)	C11—C12—H12	119.9
C5B—C4—C3B	119.3 (9)	C12—C13—O1	125.9 (4)
C3B—C4—C5A	105.4 (7)	C12—C13—C14	122.0 (4)
C5B—C4—C3A	105.3 (9)	O1—C13—C14	112.1 (3)
C5A—C4—C3A	118.5 (9)	O2—C14—C15	123.2 (3)
C5B—C4—C7	119.5 (8)	O2—C14—C13	120.3 (3)
C3B—C4—C7	120.8 (7)	C15—C14—C13	116.5 (3)
C5A—C4—C7	120.8 (6)	C16—C15—C18	119.9 (3)
C3A—C4—C7	120.6 (8)	C16—C15—C14	120.0 (3)
C1—C2B—C3B	124.6 (9)	C18—C15—C14	120.2 (3)
C1—C2B—H2B	117.7	C11—C16—C15	121.3 (4)
C3B—C2B—H2B	117.7	C11—C16—H16	119.4
C3A—C2A—C1	117.5 (13)	C15—C16—H16	119.4
C3A—C2A—H2A	121.2	O1—C17—H17A	109.5
C1—C2A—H2A	121.2	O1—C17—H17B	109.5
C4—C3B—C2B	119.2 (10)	H17A—C17—H17B	109.5
C4—C3B—H3B	120.4	O1—C17—H17C	109.5
C2B—C3B—H3B	120.4	H17A—C17—H17C	109.5
C2A—C3A—C4	119.3 (13)	H17B—C17—H17C	109.5
C2A—C3A—H3A	120.4	N3—C18—C15	122.7 (4)
C4—C3A—H3A	120.4	N3—C18—H18	118.6
C4—C5A—C6A	123.1 (9)	C15—C18—H18	118.6
C4—C5A—H5A	118.5	N3—C19—C20	106.9 (3)
C6A—C5A—H5A	118.5	N3—C19—C22	113.5 (3)
C4—C5B—C6B	120.7 (13)	C20—C19—C22	107.1 (3)
C4—C5B—H5B	119.6	N3—C19—C21	105.2 (3)
C6B—C5B—H5B	119.6	C20—C19—C21	112.3 (3)
C1—C6A—C5A	120.0 (9)	C22—C19—C21	111.9 (3)

C1—C6A—H6A	120.0	O3—C20—C19	112.2 (3)
C5A—C6A—H6A	120.0	O3—C20—H20A	109.2
C1—C6B—C5B	120.3 (13)	C19—C20—H20A	109.2
C1—C6B—H6B	119.8	O3—C20—H20B	109.2
C5B—C6B—H6B	119.8	C19—C20—H20B	109.2
C8—C7—C4	115.2 (5)	H20A—C20—H20B	107.9
C8—C7—H7A	108.5	O4—C21—C19	107.2 (3)
C4—C7—H7A	108.5	O4—C21—H21A	110.3
C8—C7—H7B	108.5	C19—C21—H21A	110.3
C4—C7—H7B	108.5	O4—C21—H21B	110.3
H7A—C7—H7B	107.5	C19—C21—H21B	110.3
C7—C8—C9	114.6 (5)	H21A—C21—H21B	108.5
C7—C8—H8A	108.6	O5—C22—C19	113.7 (3)
C9—C8—H8A	108.6	O5—C22—H22A	108.8
C7—C8—H8B	108.6	C19—C22—H22A	108.8
C9—C8—H8B	108.6	O5—C22—H22B	108.8
H8A—C8—H8B	107.6	C19—C22—H22B	108.8
C10—C9—C8	115.1 (7)	H22A—C22—H22B	107.7
C10—C9—H9A	108.5	N2—N1—C1	113.8 (4)
C8—C9—H9A	108.5	N1—N2—C11	115.5 (4)
C10—C9—H9B	108.5	C18—N3—C19	129.2 (3)
C8—C9—H9B	108.5	C18—N3—H1	116 (3)
H9A—C9—H9B	107.5	C19—N3—H1	115 (3)
C9—C10—H10A	109.5	C13—O1—C17	117.9 (4)
C9—C10—H10B	109.5	C20—O3—H3	109.5
H10A—C10—H10B	109.5	C21—O4—H4	109.5
C9—C10—H10C	109.5	C22—O5—H5	109.5

C6A—C1—C2B—C3B	36.2 (13)	C7—C8—C9—C10	−174.2 (8)
C6B—C1—C2B—C3B	−5.8 (15)	C16—C11—C12—C13	−3.8 (7)
N1—C1—C2B—C3B	175.0 (9)	N2—C11—C12—C13	175.2 (4)
C2A—C1—C2B—C3B	−89.7 (18)	C11—C12—C13—O1	177.9 (4)
C2B—C1—C2A—C3A	69.0 (17)	C11—C12—C13—C14	−3.1 (7)
C6A—C1—C2A—C3A	1.8 (18)	C12—C13—C14—O2	−173.4 (4)
C6B—C1—C2A—C3A	−40.5 (17)	O1—C13—C14—O2	5.7 (6)
N1—C1—C2A—C3A	176.7 (12)	C12—C13—C14—C15	8.1 (6)
C5B—C4—C3B—C2B	7.8 (16)	O1—C13—C14—C15	−172.8 (4)
C5A—C4—C3B—C2B	−37.5 (13)	O2—C14—C15—C16	175.0 (4)
C3A—C4—C3B—C2B	80.8 (17)	C13—C14—C15—C16	−6.6 (6)
C7—C4—C3B—C2B	−179.1 (9)	O2—C14—C15—C18	−4.7 (6)
C1—C2B—C3B—C4	0.9 (17)	C13—C14—C15—C18	173.7 (4)
C1—C2A—C3A—C4	2 (2)	N2—C11—C16—C15	−173.9 (4)
C5B—C4—C3A—C2A	40.8 (18)	C12—C11—C16—C15	5.2 (7)
C3B—C4—C3A—C2A	−79.4 (19)	C18—C15—C16—C11	179.9 (4)
C5A—C4—C3A—C2A	−4.6 (19)	C14—C15—C16—C11	0.2 (7)
C7—C4—C3A—C2A	179.8 (12)	C16—C15—C18—N3	−179.0 (4)
C5B—C4—C5A—C6A	−78.4 (15)	C14—C15—C18—N3	0.8 (6)
C3B—C4—C5A—C6A	38.1 (13)	N3—C19—C20—O3	47.2 (4)

C3A—C4—C5A—C6A	4.1 (16)	C22—C19—C20—O3	169.2 (3)
C7—C4—C5A—C6A	179.7 (9)	C21—C19—C20—O3	−67.6 (4)
C3B—C4—C5B—C6B	−10.9 (18)	N3—C19—C21—O4	−179.2 (3)
C5A—C4—C5B—C6B	70.8 (15)	C20—C19—C21—O4	−63.3 (4)
C3A—C4—C5B—C6B	−44.6 (16)	C22—C19—C21—O4	57.2 (4)
C7—C4—C5B—C6B	175.9 (10)	N3—C19—C22—O5	−79.9 (4)
C2B—C1—C6A—C5A	−35.3 (11)	C20—C19—C22—O5	162.3 (3)
C6B—C1—C6A—C5A	76.8 (14)	C21—C19—C22—O5	38.9 (4)
N1—C1—C6A—C5A	−177.6 (8)	C2B—C1—N1—N2	36.9 (10)
C2A—C1—C6A—C5A	−2.5 (13)	C6A—C1—N1—N2	170.7 (6)
C4—C5A—C6A—C1	−0.6 (15)	C6B—C1—N1—N2	−142.3 (8)
C2B—C1—C6B—C5B	2.6 (14)	C2A—C1—N1—N2	−4.4 (10)
C6A—C1—C6B—C5B	−80.0 (16)	C1—N1—N2—C11	−175.6 (5)
N1—C1—C6B—C5B	−178.2 (10)	C16—C11—N2—N1	177.1 (4)
C2A—C1—C6B—C5B	38.9 (14)	C12—C11—N2—N1	−1.9 (7)
C4—C5B—C6B—C1	5.7 (19)	C15—C18—N3—C19	−177.5 (4)
C5B—C4—C7—C8	138.3 (10)	C20—C19—N3—C18	132.3 (4)
C3B—C4—C7—C8	−34.8 (11)	C22—C19—N3—C18	14.4 (6)
C5A—C4—C7—C8	−170.5 (8)	C21—C19—N3—C18	−108.2 (5)
C3A—C4—C7—C8	5.0 (12)	C12—C13—O1—C17	−7.1 (7)
C4—C7—C8—C9	−177.4 (6)	C14—C13—O1—C17	173.8 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O3^{i}	0.82	2.03	2.769 (4)	150
O3—H3···O4^{i}	0.82	2.00	2.769 (4)	155
O5—H5···O3^{ii}	0.82	2.37	2.771 (3)	111
C20—H20*B*···O4^{iii}	0.97	2.55	3.310 (5)	135
C22—H22*B*···O2^{ii}	0.97	2.42	3.375 (5)	169
N3—H1···O2	0.88 (5)	1.88 (5)	2.603 (4)	138 (4)
O5—H5···O4	0.82	2.56	3.088 (4)	124

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