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6,6′-Dimethoxy-2,2′-[*m*-phenylenebis(nitrilomethylidyne)]diphenol

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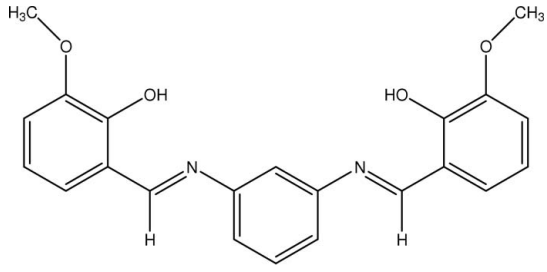
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.048; wR factor = 0.159; data-to-parameter ratio = 20.9.

In the title compound, $C_{22}H_{20}N_2O_4$, the central benzene ring forms dihedral angles of 33.58 (7) and 35.27 (7)° with the terminal benzene rings. The molecular conformation is stabilized by two intramolecular O—H···N hydrogen bonds. In the crystal structure, the molecules are linked by intermolecular C—H···O interactions to form sheets parallel to the *bc* plane. The sheets are then stacked along the *a* axis. In addition, the crystal structure is stabilized by weak C—H···π interactions.

Related literature

For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For bond length data, see: Allen *et al.* (1987). For related literature, see: Baseer *et al.* (2000); Bedioui *et al.* (1999); Campos *et al.* (1999); Desai *et al.* (2001); El-Masry *et al.* (2000); Eltayeb *et al.* (2007); Jarrahpour *et al.* (2004); Jarrahpour & Zarei (2004); Kabeer *et al.* (2001); Kuz'min, *et al.* (2000); More *et al.* (2001); Naeimi *et al.* (2007); Park & Kim (2000); Singh & Dash (1988); So *et al.* (2007); Trevin *et al.* (1997); Vigato & Tamburini (2004).



Experimental

Crystal data

$C_{22}H_{20}N_2O_4$
M_r = 376.40
Monoclinic, $C2/c$
a = 19.5677 (7) Å
b = 6.8591 (3) Å
c = 29.3903 (11) Å
β = 109.131 (2)°
V = 3726.8 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100.0 (1) K
0.80 × 0.41 × 0.23 mm

Data collection

Bruker SMART APEX II CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.893, T_{max} = 0.979
20168 measured reflections
5487 independent reflections
3714 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.159
S = 1.07
5487 reflections
263 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

*Cg*1 and *Cg*2 are centroids of the C8–C13 and C15–C20 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2*A*···N1	0.98 (2)	1.67 (2)	2.590 (2)	155 (2)
O4—H4*B*···N2	0.91 (2)	1.75 (2)	2.592 (2)	151 (2)
C4—H4*A*···O1^{i}	0.93	2.50	3.340 (2)	151
C5—H5*A*···O2^{i}	0.93	2.45	3.296 (2)	151
C22—H22*A*···O4^{ii}	0.96	2.60	3.502 (2)	157
C9—H9*A*···*Cg*1^{iii}	0.93	2.86	3.709 (2)	152
C11—H11*A*···*Cg*2^{i}	0.93	2.91	3.351 (2)	111

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2439).

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supplementary materials

Acta Cryst. (2007). E**63**, o3570-o3571 [**doi:10.1107/S1600536807034812**]

6,6'-Dimethoxy-2,2'-[*m*-phenylenebis(nitrilomethylidyne)]diphenol

M. H. Al-Douh, S. A. Hamid, H. Osman, S.-L. Ng and H.-K. Fun

Comment

Schiff bases rank among the most versatile synthetic organic intermediates. They are reported to show a variety of biological activities including antifungal (Singh & Dash, 1988; More *et al.*, 2001), antibacterial (Baseer *et al.*, 2000; El-Masry *et al.*, 2000; Kabeer *et al.*, 2001) and anticancer (Kuz'min *et al.*, 2000; Desai *et al.*, 2001) among others. They are also employed as receptors to the neutral guest molecules (Park & Kim, 2000) and found many applications in bioorganic catalysis, separation process and environmental chemistry (Trevin *et al.*, 1997; Bedioui *et al.*, 1999; Campos *et al.*, 1999). The imines formed from the reaction of the aldehydes and amines also proved to be the source of versatile ligands for many transition metals where they act as ligand donor groups to bind to the metal ions (Vigato & Tamburini, 2004). Schiff bases are also used to produce azo dyes (Jarrahpour & Zarei, 2004; Naeimi *et al.*, 2007) and they also show moderate activity against *Staphylococcus aureus and Bacillus subtilis* (Jarrahpour *et al.*, 2004). In another application, So *et al.* synthesized and characterized a series of Schiff base derivatives, which exhibited liquid crystal properties (So *et al.*, 2007). Given its importance in many areas of synthetic chemistry, we have synthesized a new symmetric Schiff base by the condensation of *o*-vanillin with *m*-phenylene diamine and its X-ray crystal structure is presented here.

The bond lengths and angles in (I) have normal values (Allen *et al.*, 1987) and are comparable with those in the related structures (Eltayeb *et al.*, 2007). The dihedral angles between the central benzene ring (C8—C13) and the terminal benzene rings [(C1—C6) and (C15—C20)] are 33.58 (7) and 35.27 (7)°, respectively. The methoxy group at C2 is slightly twisted from the attached benzene ring [C21—O1—C2—C3 = −11.33 (18)°] whereas the methoxy group at C19 is almost coplanar with the attached benzene rings with torsion angle of C22—O3—C19—C18 = 4.0 (2)°.

Intramolecular O2—H2A···N1 and O4—H4B···N2 interactions generate S(6) ring motifs (Table 1 and Figure 1) (Bernstein *et al.*, 1995). In the crystal structure, the molecules are linked by intermolecular C4—H4A···O1, C5—H5A···O2 and C22—H22A···O4 interactions to form sheets parallel to the *bc* plane. These sheets are then stacked along *a* axis. In addition, the crystal structure is further stabilized by C—H···π interactions involving C8—C13 (centroid *Cg*1) and C15—C20 (centroid *Cg*2) (Table 1).

Experimental

A 100 ml three-necked round-bottomed flask was equipped with an argon inlet adapter, rubber septum, glass stopper and a magnetic stirring bar. The flask was filled with 5 ml of dichloromethane and *o*-vanillin (608.61 mg, 0.004 mol), and was then cooled in an ice-water bath while a solution of *m*-phenylene diamine (216.29 mg, 0.002 mol) in 5 ml of dichloromethane was added dropwise *via* syringe over 15 min. After 30 min, 10 g of anhydrous magnesium sulfate was added in one portion. The ice-water bath was removed, and the reaction mixture was stirred at room temperature for 2 h. The resulting mixture was then filtered through a sintered glass funnel with an aid of two 10 ml portions of dichloromehtane, and the filtrate was concentrated at reduced pressure by rotary evaporation at room temperature to afford an orange powder. This material was dissolved in 150 ml of ethanol heated in an 353 K water bath while 270 ml of hot water was added with stirring. The resulting solution was allowed to cool to room temperature and was then further cooled in an ice-water bath for 2 h. Filtration

provided the 6,6'-dimethoxy-2,2'-[*m*-phenylenebis(nitrilomethylidyne)]diphenol. The crude product was then purified by column chromatography with *n*-hexane–diethyl ether 1:4. The product was dissolved in chloroform, and single crystals suitable for X-ray diffraction were obtained by evaporating the solvent at room temperature.

Refinement

O-bound H atoms were located in a difference map and refined isotropically. The remaining H atoms were positional geometrically and refined as riding, with C—H distances in the range 0.93 − 0.96 Å. The U_{iso} values were constrained to be 1.5 U_{eq} of the carrier atom for methyl H atoms and 1.2 U_{eq} for the remaining H atoms. The methyl groups were allowed to rotate but not to tip.

Figures

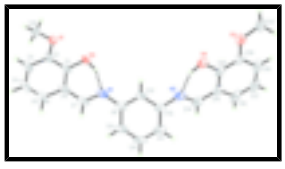

Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. The dashed lines indicate intramolecular hydrogen bonds.

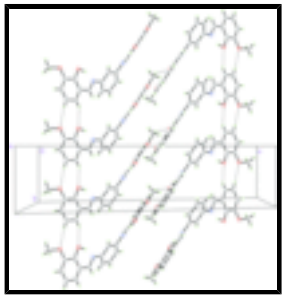

Fig. 2. The crystal packing of (I), viewed down the *a* axis. The intermolecular C—H···O hydrogen bonds are shown as dashed lines.

6,6'-Dimethoxy-2,2'-[*m*-phenylenebis(nitrilomethylidyne)]diphenol

Crystal data

$C_{22}H_{20}N_2O_4$	F_{000} = 1584
M_r = 376.40	D_x = 1.342 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 5349 reflections
a = 19.5677 (7) Å	θ = 1.5–30.3º
b = 6.8591 (3) Å	μ = 0.09 mm^{-1}
c = 29.3903 (11) Å	*T* = 100.0 (1) K
β = 109.131 (2)º	Block, orange
V = 3726.8 (3) Å^3	0.80 × 0.41 × 0.23 mm
Z = 8	

Data collection

Bruker SMART APEX II CCD area-detector	5487 independent reflections

diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 8.33 pixels mm^{-1}

T = 100.0(1) K

ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

T_{min} = 0.893, T_{max} = 0.979

20168 measured reflections

3714 reflections with $I > 2\sigma(I)$

R_{int} = 0.031

θ_{max} = 30.3º

θ_{min} = 1.5º

h = −27→27

k = −8→9

l = −41→29

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.048

$wR(F^2)$ = 0.159

S = 1.07

5487 reflections

263 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0877P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max}$ = 0.25 e $Å^{-3}$

$\Delta\rho_{min}$ = −0.20 e $Å^{-3}$

Extinction correction: none

Special details

Experimental. The data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.48169 (5)	0.71031 (13)	0.12357 (4)	0.0339 (2)
O2	0.42622 (5)	0.70622 (12)	0.19363 (4)	0.0337 (3)
O3	0.41256 (5)	−0.02808 (14)	0.47976 (3)	0.0360 (3)
O4	0.39662 (5)	0.28910 (15)	0.42757 (4)	0.0345 (2)
N1	0.37688 (6)	0.89553 (15)	0.25253 (4)	0.0332 (3)
N2	0.31250 (6)	0.55350 (17)	0.37499 (4)	0.0342 (3)

C1	0.44357 (7)	0.88164 (17)	0.17962 (5)	0.0307 (3)
C2	0.47494 (7)	0.88652 (18)	0.14290 (5)	0.0325 (3)
C3	0.49608 (8)	1.0641 (2)	0.12906 (6)	0.0410 (4)
H3A	0.5184	1.0676	0.1056	0.049*
C4	0.48410 (10)	1.2363 (2)	0.15010 (6)	0.0483 (4)
H4A	0.4987	1.3543	0.1407	0.058*
C5	0.45103 (9)	1.2344 (2)	0.18464 (6)	0.0459 (4)
H5A	0.4422	1.3512	0.1978	0.055*
C6	0.43032 (7)	1.05683 (19)	0.20024 (5)	0.0349 (3)
C7	0.39534 (7)	1.05516 (19)	0.23658 (5)	0.0367 (3)
H7A	0.3858	1.1732	0.2489	0.044*
C8	0.33731 (7)	0.89671 (18)	0.28512 (5)	0.0334 (3)
C9	0.28861 (8)	1.0459 (2)	0.28590 (6)	0.0405 (4)
H9A	0.2836	1.1543	0.2661	0.049*
C10	0.24824 (8)	1.0302 (2)	0.31643 (6)	0.0445 (4)
H10A	0.2163	1.1298	0.3171	0.053*
C11	0.25398 (7)	0.8711 (2)	0.34595 (6)	0.0413 (4)
H11A	0.2261	0.8632	0.3662	0.050*
C12	0.30233 (7)	0.7206 (2)	0.34523 (5)	0.0343 (3)
C13	0.34366 (7)	0.73609 (19)	0.31490 (5)	0.0328 (3)
H13A	0.3761	0.6373	0.3146	0.039*
C14	0.26012 (7)	0.4806 (2)	0.38664 (5)	0.0371 (3)
H14A	0.2148	0.5389	0.3751	0.045*
C15	0.26938 (7)	0.3105 (2)	0.41726 (5)	0.0376 (3)
C16	0.20960 (8)	0.2280 (3)	0.42651 (6)	0.0516 (5)
H16A	0.1644	0.2863	0.4140	0.062*
C17	0.21716 (8)	0.0632 (3)	0.45369 (6)	0.0579 (5)
H17A	0.1772	0.0106	0.4597	0.069*
C18	0.28455 (8)	−0.0272 (3)	0.47251 (6)	0.0491 (4)
H18A	0.2893	−0.1394	0.4910	0.059*
C19	0.34440 (7)	0.0503 (2)	0.46362 (5)	0.0361 (3)
C20	0.33712 (7)	0.2194 (2)	0.43601 (5)	0.0325 (3)
C21	0.50031 (8)	0.7139 (2)	0.08047 (6)	0.0399 (4)
H21A	0.4984	0.5840	0.0680	0.060*
H21B	0.5483	0.7649	0.0874	0.060*
H21C	0.4667	0.7953	0.0570	0.060*
C22	0.42160 (8)	−0.2084 (2)	0.50499 (5)	0.0415 (4)
H22A	0.4715	−0.2466	0.5147	0.062*
H22B	0.3924	−0.3066	0.4843	0.062*
H22C	0.4070	−0.1937	0.5329	0.062*
H2A	0.4091 (9)	0.743 (3)	0.2201 (7)	0.054 (5)*
H4B	0.3807 (11)	0.392 (3)	0.4071 (8)	0.071 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0321 (5)	0.0275 (5)	0.0410 (6)	0.0002 (4)	0.0107 (4)	0.0058 (4)
O2	0.0350 (5)	0.0188 (5)	0.0480 (7)	−0.0009 (4)	0.0145 (5)	0.0020 (4)

O3	0.0245 (5)	0.0483 (6)	0.0332 (5)	−0.0027 (4)	0.0065 (4)	0.0031 (4)
O4	0.0207 (4)	0.0419 (6)	0.0408 (6)	0.0003 (4)	0.0099 (4)	−0.0004 (5)
N1	0.0276 (5)	0.0248 (6)	0.0384 (7)	0.0034 (4)	−0.0013 (5)	−0.0030 (5)
N2	0.0254 (5)	0.0406 (7)	0.0330 (6)	0.0049 (5)	0.0047 (5)	−0.0097 (5)
C1	0.0245 (6)	0.0198 (6)	0.0385 (8)	−0.0009 (4)	−0.0020 (5)	0.0055 (5)
C2	0.0246 (6)	0.0247 (7)	0.0393 (8)	−0.0014 (5)	−0.0015 (6)	0.0072 (5)
C3	0.0387 (8)	0.0327 (8)	0.0403 (9)	−0.0068 (6)	−0.0022 (6)	0.0144 (6)
C4	0.0577 (10)	0.0262 (7)	0.0462 (10)	−0.0104 (6)	−0.0034 (8)	0.0127 (7)
C5	0.0525 (9)	0.0177 (7)	0.0488 (10)	−0.0014 (6)	−0.0088 (8)	0.0030 (6)
C6	0.0316 (7)	0.0219 (6)	0.0385 (8)	0.0016 (5)	−0.0058 (6)	0.0023 (5)
C7	0.0327 (7)	0.0223 (6)	0.0408 (8)	0.0053 (5)	−0.0075 (6)	−0.0056 (6)
C8	0.0249 (6)	0.0296 (7)	0.0359 (8)	0.0039 (5)	−0.0032 (6)	−0.0103 (6)
C9	0.0320 (7)	0.0321 (7)	0.0443 (9)	0.0105 (6)	−0.0052 (6)	−0.0082 (6)
C10	0.0334 (7)	0.0411 (8)	0.0483 (9)	0.0173 (6)	−0.0013 (7)	−0.0141 (7)
C11	0.0274 (7)	0.0472 (9)	0.0417 (8)	0.0109 (6)	0.0010 (6)	−0.0152 (7)
C12	0.0238 (6)	0.0372 (8)	0.0338 (8)	0.0057 (5)	−0.0016 (5)	−0.0113 (6)
C13	0.0230 (6)	0.0291 (7)	0.0392 (8)	0.0067 (5)	0.0003 (6)	−0.0095 (6)
C14	0.0215 (6)	0.0564 (9)	0.0301 (7)	0.0070 (6)	0.0038 (6)	−0.0090 (6)
C15	0.0223 (6)	0.0623 (10)	0.0269 (7)	0.0011 (6)	0.0063 (5)	−0.0054 (6)
C16	0.0208 (7)	0.0974 (14)	0.0370 (9)	0.0051 (7)	0.0101 (6)	0.0044 (9)
C17	0.0258 (7)	0.1075 (15)	0.0429 (10)	−0.0062 (8)	0.0147 (7)	0.0139 (10)
C18	0.0299 (7)	0.0806 (12)	0.0361 (9)	−0.0060 (7)	0.0099 (7)	0.0115 (8)
C19	0.0226 (6)	0.0594 (9)	0.0247 (7)	−0.0022 (6)	0.0058 (5)	−0.0027 (6)
C20	0.0207 (6)	0.0510 (9)	0.0255 (7)	−0.0023 (5)	0.0072 (5)	−0.0072 (6)
C21	0.0355 (7)	0.0430 (8)	0.0390 (8)	0.0019 (6)	0.0092 (6)	0.0109 (7)
C22	0.0342 (7)	0.0578 (10)	0.0287 (7)	−0.0065 (7)	0.0053 (6)	0.0059 (7)

Geometric parameters (Å, °)

O1—C2	1.3602 (16)	C9—C10	1.380 (2)
O1—C21	1.4285 (18)	C9—H9A	0.9300
O2—C1	1.3506 (15)	C10—C11	1.376 (2)
O2—H2A	0.975 (18)	C10—H10A	0.9300
O3—C19	1.3704 (16)	C11—C12	1.4051 (18)
O3—C22	1.4229 (18)	C11—H11A	0.9300
O4—C20	1.3547 (16)	C12—C13	1.390 (2)
O4—H4B	0.91 (2)	C13—H13A	0.9300
N1—C7	1.2884 (18)	C14—C15	1.448 (2)
N1—C8	1.4156 (19)	C14—H14A	0.9300
N2—C14	1.2838 (18)	C15—C16	1.404 (2)
N2—C12	1.4156 (19)	C15—C20	1.4045 (19)
C1—C2	1.406 (2)	C16—C17	1.364 (3)
C1—C6	1.4076 (19)	C16—H16A	0.9300
C2—C3	1.3895 (18)	C17—C18	1.397 (2)
C3—C4	1.388 (2)	C17—H17A	0.9300
C3—H3A	0.9300	C18—C19	1.387 (2)
C4—C5	1.371 (3)	C18—H18A	0.9300
C4—H4A	0.9300	C19—C20	1.396 (2)
C5—C6	1.407 (2)	C21—H21A	0.9600

C5—H5A	0.9300	C21—H21B	0.9600
C6—C7	1.445 (2)	C21—H21C	0.9600
C7—H7A	0.9300	C22—H22A	0.9600
C8—C13	1.387 (2)	C22—H22B	0.9600
C8—C9	1.4036 (18)	C22—H22C	0.9600
C2—O1—C21	116.28 (10)	C13—C12—C11	119.31 (14)
C1—O2—H2A	101.6 (10)	C13—C12—N2	117.69 (11)
C19—O3—C22	117.51 (11)	C11—C12—N2	122.98 (14)
C20—O4—H4B	105.4 (12)	C8—C13—C12	120.98 (12)
C7—N1—C8	121.48 (11)	C8—C13—H13A	119.5
C14—N2—C12	121.16 (11)	C12—C13—H13A	119.5
O2—C1—C2	118.15 (11)	N2—C14—C15	122.10 (12)
O2—C1—C6	121.95 (14)	N2—C14—H14A	118.9
C2—C1—C6	119.90 (12)	C15—C14—H14A	118.9
O1—C2—C3	125.29 (14)	C16—C15—C20	118.94 (15)
O1—C2—C1	115.21 (10)	C16—C15—C14	120.17 (13)
C3—C2—C1	119.50 (13)	C20—C15—C14	120.81 (12)
C4—C3—C2	120.36 (16)	C17—C16—C15	120.66 (14)
C4—C3—H3A	119.8	C17—C16—H16A	119.7
C2—C3—H3A	119.8	C15—C16—H16A	119.7
C5—C4—C3	120.76 (13)	C16—C17—C18	120.46 (14)
C5—C4—H4A	119.6	C16—C17—H17A	119.8
C3—C4—H4A	119.6	C18—C17—H17A	119.8
C4—C5—C6	120.37 (14)	C19—C18—C17	120.09 (17)
C4—C5—H5A	119.8	C19—C18—H18A	120.0
C6—C5—H5A	119.8	C17—C18—H18A	120.0
C5—C6—C1	119.03 (15)	O3—C19—C18	124.81 (15)
C5—C6—C7	120.29 (13)	O3—C19—C20	115.43 (12)
C1—C6—C7	120.67 (12)	C18—C19—C20	119.76 (13)
N1—C7—C6	122.19 (12)	O4—C20—C19	118.02 (12)
N1—C7—H7A	118.9	O4—C20—C15	121.89 (13)
C6—C7—H7A	118.9	C19—C20—C15	120.08 (12)
C13—C8—C9	119.33 (14)	O1—C21—H21A	109.5
C13—C8—N1	117.82 (11)	O1—C21—H21B	109.5
C9—C8—N1	122.69 (13)	H21A—C21—H21B	109.5
C10—C9—C8	119.34 (14)	O1—C21—H21C	109.5
C10—C9—H9A	120.3	H21A—C21—H21C	109.5
C8—C9—H9A	120.3	H21B—C21—H21C	109.5
C11—C10—C9	121.73 (13)	O3—C22—H22A	109.5
C11—C10—H10A	119.1	O3—C22—H22B	109.5
C9—C10—H10A	119.1	H22A—C22—H22B	109.5
C10—C11—C12	119.31 (15)	O3—C22—H22C	109.5
C10—C11—H11A	120.3	H22A—C22—H22C	109.5
C12—C11—H11A	120.3	H22B—C22—H22C	109.5
C21—O1—C2—C3	−11.33 (18)	C10—C11—C12—N2	178.88 (13)
C21—O1—C2—C1	168.48 (11)	C14—N2—C12—C13	−148.15 (13)
O2—C1—C2—O1	2.78 (17)	C14—N2—C12—C11	33.3 (2)
C6—C1—C2—O1	−176.44 (11)	C9—C8—C13—C12	0.31 (19)

O2—C1—C2—C3	−177.40 (11)	N1—C8—C13—C12	−175.16 (12)
C6—C1—C2—C3	3.38 (19)	C11—C12—C13—C8	−0.6 (2)
O1—C2—C3—C4	177.57 (13)	N2—C12—C13—C8	−179.22 (11)
C1—C2—C3—C4	−2.2 (2)	C12—N2—C14—C15	−179.64 (12)
C2—C3—C4—C5	−0.3 (2)	N2—C14—C15—C16	−175.80 (14)
C3—C4—C5—C6	1.7 (2)	N2—C14—C15—C20	0.8 (2)
C4—C5—C6—C1	−0.5 (2)	C20—C15—C16—C17	0.7 (2)
C4—C5—C6—C7	−179.89 (13)	C14—C15—C16—C17	177.33 (16)
O2—C1—C6—C5	178.77 (12)	C15—C16—C17—C18	−0.5 (3)
C2—C1—C6—C5	−2.04 (19)	C16—C17—C18—C19	−0.1 (3)
O2—C1—C6—C7	−1.82 (19)	C22—O3—C19—C18	4.0 (2)
C2—C1—C6—C7	177.37 (11)	C22—O3—C19—C20	−175.40 (12)
C8—N1—C7—C6	−174.64 (11)	C17—C18—C19—O3	−179.07 (15)
C5—C6—C7—N1	−178.56 (12)	C17—C18—C19—C20	0.3 (3)
C1—C6—C7—N1	2.0 (2)	O3—C19—C20—O4	0.32 (19)
C7—N1—C8—C13	−154.90 (12)	C18—C19—C20—O4	−179.14 (14)
C7—N1—C8—C9	29.79 (19)	O3—C19—C20—C15	179.36 (13)
C13—C8—C9—C10	0.27 (19)	C18—C19—C20—C15	−0.1 (2)
N1—C8—C9—C10	175.51 (13)	C16—C15—C20—O4	178.60 (13)
C8—C9—C10—C11	−0.5 (2)	C14—C15—C20—O4	2.0 (2)
C9—C10—C11—C12	0.2 (2)	C16—C15—C20—C19	−0.4 (2)
C10—C11—C12—C13	0.4 (2)	C14—C15—C20—C19	−177.03 (13)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2A···N1	0.98 (2)	1.67 (2)	2.590 (2)	155 (2)
O4—H4B···N2	0.91 (2)	1.75 (2)	2.592 (2)	151 (2)
C4—H4A···O1^{i}	0.93	2.50	3.340 (2)	151
C5—H5A···O2^{i}	0.93	2.45	3.296 (2)	151
C22—H22A···O4^{ii}	0.96	2.60	3.502 (2)	157
C9—H9A···Cg1^{iii}	0.93	2.86	3.709 (2)	152
C11—H11A···Cg2^{i}	0.93	2.91	3.351 (2)	111

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

Fig. 1

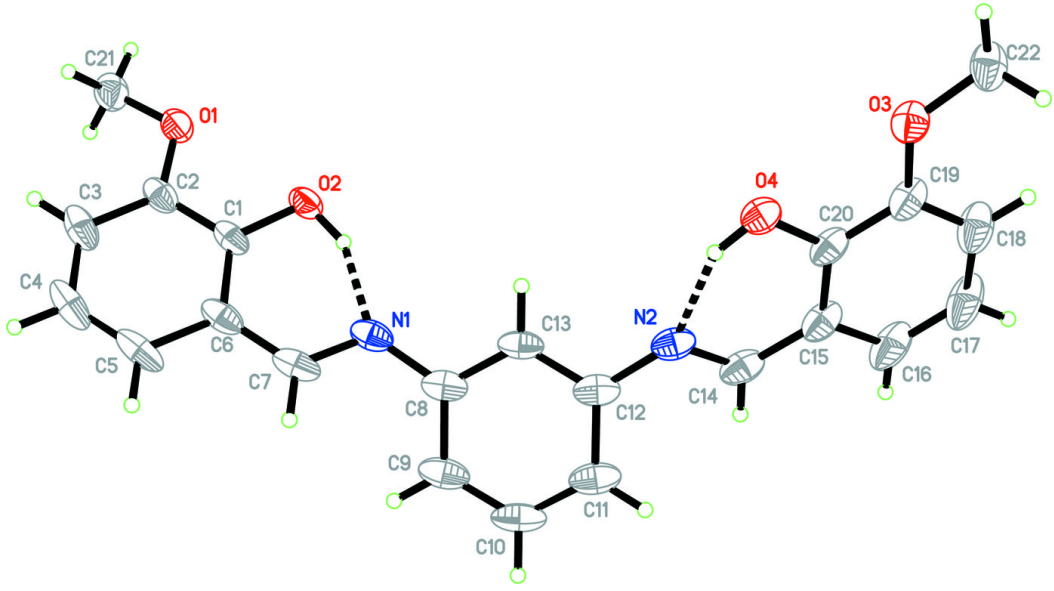
C21
O1
C2
C3
C1
O2
C4
C6
N1
C13
N2
O4
C20
C19
O3
C22
C18
C5
C7
C8
C12
C15
C17
C14
C16
C9
C11
C10

Fig. 2