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4,4′-Dimethoxy-2,2′-[1,2-phenylenebis(nitrilomethylidyne)]diphenol

Naser Eltaher Eltayeb,[a]‡ Siang Guan Teoh,[a] Suchada Chantrapromma,[b]§ Hoong-Kun Fun[c]* and Kamarulazizi Ibrahim[d]

[a]School of Chemical Science, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, [b]Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, [c]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [d]School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: hkfun@usm.my

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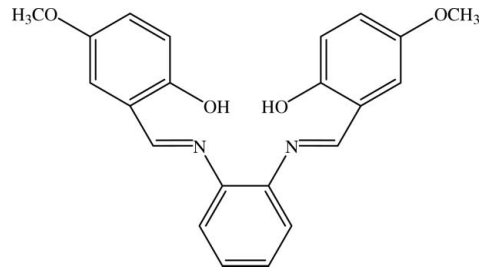
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.053; wR factor = 0.154; data-to-parameter ratio = 29.4.

The title compound, $C_{22}H_{20}N_2O_4$, was synthesized by the reaction of *o*-phenylenediamine with 5-methoxysalicylaldehyde, as an extension of our investigations of Schiff base ligands and complexes. The central ring makes dihedral angles of 36.35 (5) and 17.81 (5)° with the two phenol rings. Each methoxy group is coplanar with the attached phenol ring. There are two O—H···N intramolecular hydrogen bonds involving the two hydroxy groups, which generate S(6) ring motifs. In the crystal structure, the molecules are arranged into chains along the c axis. These chains form molecular sheets parallel to the ac plane. The crystal is stabilized by intramolecular O—H···N hydrogen bonds and C—H···π interactions.

Related literature

For related literature on values of bond lengths, see: Allen *et al.* (1987). For related literature on hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related structures, see, for example: Eltayeb, Teoh, Chantrapromma *et al.* (2007); Eltayeb, Teoh, Teh *et al.* (2007*a*,*b*). For related literature on pharmacological activities and applications, see, for example: Dao *et al.* (2000); Karthikeyan *et al.* (2006); Sriram *et al.* (2006); Eltayeb & Ahmed (2005*a*,*b*).



Experimental

Crystal data

$C_{22}H_{20}N_2O_4$
M_r = 376.40
Monoclinic, $P2_1/c$
a = 11.6772 (3) Å
b = 13.8721 (3) Å
c = 13.1182 (3) Å
β = 119.875 (2)°
V = 1842.60 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100.0 (1) K
0.57 × 0.47 × 0.12 mm

Data collection

Bruker SMART APEX II CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.948, T_{max} = 0.989
36710 measured reflections
8095 independent reflections
6228 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.154
S = 1.04
8095 reflections
275 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O1···N1	0.86	1.78	2.5811 (13)	153
O3—H1O3···N2	0.90	1.82	2.6285 (14)	148
C7—H7*A*···*Cg*2^{i}	0.93	2.82	3.2893 (11)	112
C22—H22*C*···*Cg*1^{i}	0.96	2.84	3.7312 (12)	155

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

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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‡ On study leave from International University of Africa, Sudan. E-mail: nasertaha90@hotmail.com.
§ Additional correspondence author, email: suchada.c@psu.ac.th.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2174).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bruker (2005). *APEX2* (Version 1.27), *SAINT* (Version 7.12A) and *SADABS* (Version 2004/1). Bruker AXS Inc., Madison, Wisconsin, USA.

Dao, V.-T., Gaspard, C., Mayer, M., Werner, G. H., Nguyen, S. N. & Michelot, R. J. (2000). *Eur. J. Med. Chem.* **35**, 805–813.

Eltayeb, N. E. & Ahmed, T. A. (2005*a*). *J. Sci. Technol.* **6**, 51–59.

Eltayeb, N. E. & Ahmed, T. A. (2005*b*). *Sudan J. Basic Sci.* **7**, 97–108.

Eltayeb, N. E., Teoh, S. G., Chantrapromma, S., Fun, H.-K. & Ibrahim, K. (2007). *Acta Cryst.* E**63**, m1672–m1673.

Eltayeb, N. E., Teoh, S. G., Teh, J. B.-J., Fun, H.-K. & Ibrahim, K. (2007*a*). *Acta Cryst.* E**63**, o695–o696.

Eltayeb, N. E., Teoh, S. G., Teh, J. B.-J., Fun, H.-K. & Ibrahim, K. (2007*b*). *Acta Cryst.* E**63**, o766–o767.

Karthikeyan, M. S., Prasad, D. J., Poojary, B., Bhat, K. S., Holla, B. S. & Kumari, N. S. (2006). *Bioorg. Med. Chem.* **14**, 7482–7489.

Sheldrick, G. M. (1998). *SHELXTL.* Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Sriram, D., Yogeeswari, P., Myneedu, N. S. & Saraswat, V. (2006). *Bioorg. Med. Chem. Lett.* **16**, 2127–2129.

supplementary materials

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4,4'-Dimethoxy-2,2'-[1,2-phenylenebis(nitrilomethylidyne)]diphenol

N. E. Eltayeb, S. G. Teoh, S. Chantrapromma, H.-K. Fun and K. Ibrahim

Comment

Schiff base compounds have received much attention because of their potential applications. Some of these compounds exhibit various pharmacological activities, such as anticancer (Dao *et al.*, 2000), anti-HIV (Sriram *et al.*, 2006), antibacterial and antifungal (Karthikeyan *et al.*, 2006) properties. In addition, some of them may be used as analytical reagents for the determination of trace elements (Eltayeb & Ahmed, 2005*a*,b). We have previously reported the crystal structures of Schiff base ligands which are 2,2'-[1,2-phenylenebis(nitrilomethylidyne)]bis(5-methylphenol) (Eltayeb *et al.*, 2007*a*), 6,6'-dimethyl-2,2'-[1,2-phenylenebis(nitrilomethylidyne)]diphenol (Eltayeb *et al.*, 2007*b*) and 5,5'-dimethoxy-2,2'-[1,2-phenylenebis(nitrilomethylidyne)]diphenol (Eltayeb *et al.*, 2007). As an extension of our investigations of Schiff base ligands and complexes, the title compound, (I), was synthesized by the reaction of *o*-phenylenediamine with 5-methoxysalicylaldehyde, and its crystal structure is reported here.

The structure of the title compound is not planar. The orientations of the C1–C6 and C15–C20 benzene rings respect to the *o*-phenylenediamine unit are indicated by the dihedral angles of 36.35 (5) and 17.81 (5)°, respectively. The C1–C6 and C15–C20 benzene rings makes the dihedral angle of 43.94 (5)°. The two methoxy groups are planarly attached to the C1–C6 and C15–C20 benzene rings. The C8/N1/C7/C6 unit is not planar as indicated by the torsion angles C8/N1/C7/C6 = −171.09 (8)° while the C13/N2/C14/C15 unit is planarly attached with the torsion angle of 178.91 (8)°. The orientations of these units with respect to the *o*-phenylenediamine ring are shown by the torsion angles C7/N1/C8/C9 = 31.92 (13)° and C14/N2/C13/C12 = −22.97 (14)°.

The two intramolecular hydrogen bonds, O1—H1O1···N1 and O3—H1O3···N2 generate S(6) ring motifs (Bernstein *et al.*, 1995). Bond lengths and angles in (I) are in normal ranges (Allen *et al.*, 1987) and comparable to those in related structures (Eltayeb *et al.*, 2007; Eltayeb *et al.*, 2007*a*,b).

In the crystal, the molecules are arranged into chains along the *c* axis. These chains form molecular sheets parallel to the *ac* plane (Fig. 2). The crystal is stabilized by O—H···N intramolecular hydrogen bonds (Table 1) and further stabilized by C—H···π interactions (Table 1); Cg_1 and Cg_2 are the centroids of C8–C13 and C15–C20 benzene rings, respectively.

Experimental

The title compound was synthesized by adding 5-methoxysalicylaldehyde (0.4 ml, 4 mmol) into a solution of *o*-phenylenediamine (0.216 g, 2 mmol) in ethanol 95% (20 ml). The mixture was refluxed with stirring for half an hour. The resultant red solution was filtered. Red plate-shaped single crystals of (I) suitable for X-ray structure determination were formed after two days of slow evaporation of the solvent at room temperature.

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with O—H distances of 0.86–0.90 Å and C—H distances in the range 0.93–0.96 Å. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups.

Figures

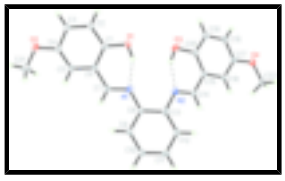

Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. Hydrogen bonds were drawn as dash lines.

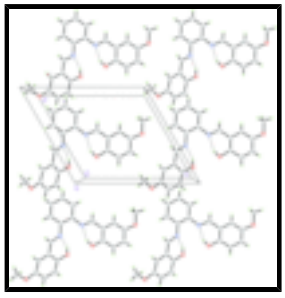

Fig. 2. The crystal packing of (I), viewed along the *b* axis. Hydrogen bonds were drawn as dash lines.

4,4'-Dimethoxy-2,2'-[1,2-phenylenebis(nitrilomethylidyne)]diphenol

Crystal data

$C_{22}H_{20}N_2O_4$	F_{000} = 792
M_r = 376.40	D_x = 1.357 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 8095 reflections
a = 11.6772 (3) Å	θ = 2.3–35.0º
b = 13.8721 (3) Å	μ = 0.09 mm^{-1}
c = 13.1182 (3) Å	T = 100.0 (1) K
β = 119.875 (2)º	Plate, red
V = 1842.60 (8) Å^3	0.57 × 0.47 × 0.12 mm
Z = 4	

Data collection

Bruker SMART APEX II CCD area-detector diffractometer	8095 independent reflections
Radiation source: fine-focus sealed tube	6228 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.049
Detector resolution: 8.33 pixels mm^{-1}	θ_{max} = 35.0º

T = 100.0(1) K	θ_{min} = 2.3°
ω scans	h = −18→18
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	k = −22→22
T_{min} = 0.948, T_{max} = 0.989	l = −21→21
36710 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.053	H-atom parameters constrained
$wR(F^2)$ = 0.154	$w = 1/[\sigma^2(F_o^2) + (0.0831P)^2 + 0.3111P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.001
8095 reflections	$\Delta\rho_{max}$ = 0.41 e Å^{-3}
275 parameters	$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. The low-temparture 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	0.83325 (7)	0.46826 (6)	0.48148 (6)	0.02043 (15)
H1O1	0.7502	0.4544	0.4461	0.062 (6)*
O2	1.06862 (7)	0.28976 (6)	0.91533 (7)	0.02480 (16)
O3	0.72475 (7)	0.41856 (6)	0.21130 (6)	0.02213 (15)
H1O3	0.6644	0.4180	0.2348	0.075 (7)*
O4	0.48112 (7)	0.34127 (6)	−0.26517 (6)	0.02223 (15)
N1	0.60911 (8)	0.39635 (6)	0.43686 (7)	0.01623 (15)
N2	0.49232 (8)	0.40442 (6)	0.19622 (7)	0.01599 (15)
C1	0.88570 (9)	0.42265 (7)	0.58656 (8)	0.01575 (16)
C2	1.01832 (9)	0.43830 (7)	0.66858 (8)	0.01768 (17)
H2A	1.0690	0.4789	0.6503	0.024 (4)*

C3	1.07443 (9)	0.39357 (7)	0.77689 (8)	0.01846 (17)
H3A	1.1626	0.4052	0.8317	0.028 (4)*
C4	1.00057 (9)	0.33070 (7)	0.80566 (8)	0.01765 (17)
C5	0.86873 (9)	0.31521 (7)	0.72544 (8)	0.01713 (16)
H5A	0.8192	0.2738	0.7441	0.025 (4)*
C6	0.80918 (9)	0.36209 (7)	0.61532 (8)	0.01528 (16)
C7	0.66841 (9)	0.35170 (7)	0.53648 (8)	0.01638 (16)
H7A	0.6197	0.3121	0.5580	0.019 (3)*
C8	0.47016 (9)	0.39602 (7)	0.36932 (8)	0.01505 (15)
C9	0.39089 (10)	0.39297 (7)	0.42200 (9)	0.01845 (17)
H9A	0.4305	0.3887	0.5033	0.032 (4)*
C10	0.25443 (10)	0.39636 (8)	0.35414 (9)	0.02039 (18)
H10A	0.2029	0.3916	0.3897	0.034 (4)*
C11	0.19468 (10)	0.40685 (8)	0.23291 (9)	0.02124 (19)
H11A	0.1032	0.4106	0.1876	0.029 (4)*
C12	0.27157 (9)	0.41177 (8)	0.17945 (9)	0.01916 (18)
H12A	0.2312	0.4200	0.0985	0.027 (4)*
C13	0.40965 (9)	0.40438 (7)	0.24637 (8)	0.01541 (16)
C14	0.44488 (9)	0.37959 (7)	0.08798 (8)	0.01615 (16)
H14A	0.3568	0.3609	0.0448	0.028 (4)*
C15	0.52455 (9)	0.37990 (7)	0.03136 (8)	0.01512 (15)
C16	0.46282 (9)	0.35957 (7)	−0.08930 (8)	0.01622 (16)
H16A	0.3727	0.3469	−0.1309	0.023 (3)*
C17	0.53497 (9)	0.35827 (7)	−0.14717 (8)	0.01730 (16)
C18	0.67094 (10)	0.37513 (8)	−0.08346 (9)	0.01983 (18)
H18A	0.7201	0.3728	−0.1215	0.038 (4)*
C19	0.73312 (10)	0.39529 (7)	0.03560 (9)	0.01973 (18)
H19A	0.8236	0.4066	0.0768	0.034 (4)*
C20	0.66080 (9)	0.39875 (7)	0.09450 (8)	0.01706 (16)
C21	0.99719 (12)	0.22443 (10)	0.94684 (11)	0.0321 (3)
H21A	1.0553	0.1983	1.0234	0.047 (5)*
H21B	0.9623	0.1731	0.8904	0.041 (5)*
H21C	0.9259	0.2580	0.9480	0.038 (4)*
C22	0.34072 (10)	0.33517 (8)	−0.33058 (9)	0.02211 (19)
H22A	0.3125	0.3243	−0.4120	0.034 (4)*
H22B	0.3033	0.3943	−0.3226	0.038 (4)*
H22C	0.3117	0.2827	−0.3013	0.022 (3)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0168 (3)	0.0278 (4)	0.0151 (3)	−0.0011 (3)	0.0067 (2)	0.0054 (3)
O2	0.0181 (3)	0.0329 (4)	0.0166 (3)	−0.0015 (3)	0.0035 (3)	0.0090 (3)
O3	0.0155 (3)	0.0301 (4)	0.0173 (3)	−0.0036 (3)	0.0055 (3)	−0.0050 (3)
O4	0.0211 (3)	0.0304 (4)	0.0165 (3)	0.0008 (3)	0.0104 (3)	−0.0006 (3)
N1	0.0136 (3)	0.0192 (3)	0.0134 (3)	−0.0008 (3)	0.0049 (3)	−0.0004 (3)
N2	0.0141 (3)	0.0180 (3)	0.0152 (3)	0.0007 (3)	0.0068 (3)	0.0005 (3)
C1	0.0157 (4)	0.0182 (4)	0.0137 (3)	0.0000 (3)	0.0076 (3)	0.0007 (3)

C2	0.0146 (4)	0.0206 (4)	0.0178 (4)	−0.0021 (3)	0.0081 (3)	0.0003 (3)
C3	0.0131 (4)	0.0227 (4)	0.0169 (4)	−0.0012 (3)	0.0054 (3)	0.0002 (3)
C4	0.0158 (4)	0.0210 (4)	0.0137 (4)	0.0002 (3)	0.0054 (3)	0.0021 (3)
C5	0.0158 (4)	0.0181 (4)	0.0154 (4)	−0.0018 (3)	0.0062 (3)	0.0018 (3)
C6	0.0134 (4)	0.0165 (4)	0.0139 (3)	−0.0014 (3)	0.0052 (3)	−0.0002 (3)
C7	0.0154 (4)	0.0161 (4)	0.0156 (4)	−0.0021 (3)	0.0062 (3)	0.0000 (3)
C8	0.0131 (4)	0.0156 (4)	0.0149 (4)	−0.0001 (3)	0.0058 (3)	0.0003 (3)
C9	0.0180 (4)	0.0216 (4)	0.0165 (4)	0.0007 (3)	0.0092 (3)	0.0008 (3)
C10	0.0173 (4)	0.0242 (4)	0.0219 (4)	0.0017 (3)	0.0114 (4)	0.0008 (3)
C11	0.0137 (4)	0.0270 (5)	0.0219 (4)	0.0020 (3)	0.0079 (3)	−0.0002 (3)
C12	0.0142 (4)	0.0250 (4)	0.0158 (4)	0.0022 (3)	0.0055 (3)	0.0001 (3)
C13	0.0128 (4)	0.0175 (4)	0.0146 (4)	0.0003 (3)	0.0059 (3)	−0.0001 (3)
C14	0.0139 (4)	0.0184 (4)	0.0150 (4)	0.0003 (3)	0.0064 (3)	0.0005 (3)
C15	0.0136 (4)	0.0156 (4)	0.0153 (4)	0.0005 (3)	0.0066 (3)	0.0006 (3)
C16	0.0139 (4)	0.0182 (4)	0.0158 (4)	0.0006 (3)	0.0068 (3)	0.0008 (3)
C17	0.0181 (4)	0.0178 (4)	0.0170 (4)	0.0014 (3)	0.0095 (3)	0.0009 (3)
C18	0.0177 (4)	0.0210 (4)	0.0232 (4)	0.0010 (3)	0.0120 (4)	0.0002 (3)
C19	0.0144 (4)	0.0220 (4)	0.0227 (4)	−0.0007 (3)	0.0091 (3)	−0.0004 (3)
C20	0.0147 (4)	0.0169 (4)	0.0173 (4)	0.0003 (3)	0.0063 (3)	−0.0004 (3)
C21	0.0220 (5)	0.0418 (7)	0.0282 (5)	0.0008 (5)	0.0092 (4)	0.0185 (5)
C22	0.0219 (5)	0.0267 (5)	0.0157 (4)	0.0015 (4)	0.0079 (3)	0.0021 (3)

Geometric parameters (Å, °)

O1—C1	1.3544 (11)	C9—C10	1.3858 (14)
O1—H1O1	0.8632	C9—H9A	0.9299
O2—C4	1.3729 (11)	C10—C11	1.3898 (14)
O2—C21	1.4253 (14)	C10—H10A	0.9300
O3—C20	1.3567 (12)	C11—C12	1.3893 (14)
O3—H1O3	0.8989	C11—H11A	0.9300
O4—C17	1.3702 (12)	C12—C13	1.4038 (13)
O4—C22	1.4247 (13)	C12—H12A	0.9299
N1—C7	1.2921 (12)	C14—C15	1.4511 (12)
N1—C8	1.4091 (12)	C14—H14A	0.9300
N2—C14	1.2877 (12)	C15—C16	1.4028 (13)
N2—C13	1.4133 (12)	C15—C20	1.4046 (13)
C1—C2	1.3939 (13)	C16—C17	1.3869 (12)
C1—C6	1.4080 (13)	C16—H16A	0.9299
C2—C3	1.3804 (13)	C17—C18	1.3972 (14)
C2—H2A	0.9301	C18—C19	1.3838 (14)
C3—C4	1.4040 (13)	C18—H18A	0.9301
C3—H3A	0.9300	C19—C20	1.4010 (13)
C4—C5	1.3819 (13)	C19—H19A	0.9300
C5—C6	1.4116 (13)	C21—H21A	0.9600
C5—H5A	0.9301	C21—H21B	0.9600
C6—C7	1.4487 (13)	C21—H21C	0.9600
C7—H7A	0.9300	C22—H22A	0.9600
C8—C9	1.4047 (13)	C22—H22B	0.9600
C8—C13	1.4067 (13)	C22—H22C	0.9600

C1—O1—H1O1	105.1	C11—C12—C13	120.69 (9)
C4—O2—C21	116.75 (8)	C11—C12—H12A	119.7
C20—O3—H1O3	107.5	C13—C12—H12A	119.7
C17—O4—C22	115.70 (7)	C12—C13—C8	119.18 (8)
C7—N1—C8	120.23 (8)	C12—C13—N2	123.15 (8)
C14—N2—C13	119.65 (8)	C8—C13—N2	117.66 (8)
O1—C1—C2	118.57 (8)	N2—C14—C15	122.02 (8)
O1—C1—C6	121.72 (8)	N2—C14—H14A	119.0
C2—C1—C6	119.70 (8)	C15—C14—H14A	119.0
C3—C2—C1	119.98 (8)	C16—C15—C20	119.82 (8)
C3—C2—H2A	120.0	C16—C15—C14	118.48 (8)
C1—C2—H2A	120.0	C20—C15—C14	121.69 (8)
C2—C3—C4	121.04 (9)	C17—C16—C15	120.62 (9)
C2—C3—H3A	119.5	C17—C16—H16A	119.7
C4—C3—H3A	119.5	C15—C16—H16A	119.7
O2—C4—C5	125.26 (9)	O4—C17—C16	123.95 (9)
O2—C4—C3	115.23 (8)	O4—C17—C18	116.73 (8)
C5—C4—C3	119.50 (8)	C16—C17—C18	119.31 (9)
C4—C5—C6	120.11 (8)	C19—C18—C17	120.66 (9)
C4—C5—H5A	119.9	C19—C18—H18A	119.7
C6—C5—H5A	119.9	C17—C18—H18A	119.7
C1—C6—C5	119.61 (8)	C18—C19—C20	120.52 (9)
C1—C6—C7	120.93 (8)	C18—C19—H19A	119.7
C5—C6—C7	119.35 (8)	C20—C19—H19A	119.7
N1—C7—C6	121.35 (8)	O3—C20—C19	119.07 (8)
N1—C7—H7A	119.3	O3—C20—C15	121.89 (8)
C6—C7—H7A	119.3	C19—C20—C15	119.03 (9)
C9—C8—C13	119.26 (8)	O2—C21—H21A	109.5
C9—C8—N1	121.69 (8)	O2—C21—H21B	109.5
C13—C8—N1	118.95 (8)	H21A—C21—H21B	109.5
C10—C9—C8	120.77 (9)	O2—C21—H21C	109.5
C10—C9—H9A	119.6	H21A—C21—H21C	109.5
C8—C9—H9A	119.6	H21B—C21—H21C	109.5
C9—C10—C11	119.99 (9)	O4—C22—H22A	109.5
C9—C10—H10A	120.0	O4—C22—H22B	109.5
C11—C10—H10A	120.0	H22A—C22—H22B	109.5
C12—C11—C10	120.03 (9)	O4—C22—H22C	109.5
C12—C11—H11A	120.0	H22A—C22—H22C	109.5
C10—C11—H11A	120.0	H22B—C22—H22C	109.5
O1—C1—C2—C3	179.42 (9)	C11—C12—C13—N2	176.98 (9)
C6—C1—C2—C3	0.79 (14)	C9—C8—C13—C12	1.49 (14)
C1—C2—C3—C4	1.13 (15)	N1—C8—C13—C12	−175.00 (9)
C21—O2—C4—C5	1.54 (16)	C9—C8—C13—N2	−178.22 (8)
C21—O2—C4—C3	−179.11 (10)	N1—C8—C13—N2	5.30 (13)
C2—C3—C4—O2	178.97 (9)	C14—N2—C13—C12	−22.97 (14)
C2—C3—C4—C5	−1.64 (15)	C14—N2—C13—C8	156.72 (9)
O2—C4—C5—C6	179.54 (9)	C13—N2—C14—C15	178.91 (8)
C3—C4—C5—C6	0.22 (15)	N2—C14—C15—C16	−174.80 (9)
O1—C1—C6—C5	179.24 (9)	N2—C14—C15—C20	5.98 (14)

C2—C1—C6—C5	−2.18 (14)	C20—C15—C16—C17	−0.26 (14)
O1—C1—C6—C7	−4.52 (14)	C14—C15—C16—C17	−179.49 (9)
C2—C1—C6—C7	174.06 (9)	C22—O4—C17—C16	6.82 (14)
C4—C5—C6—C1	1.67 (14)	C22—O4—C17—C18	−173.15 (9)
C4—C5—C6—C7	−174.63 (9)	C15—C16—C17—O4	−178.41 (9)
C8—N1—C7—C6	−171.09 (8)	C15—C16—C17—C18	1.56 (14)
C1—C6—C7—N1	1.04 (14)	O4—C17—C18—C19	178.40 (9)
C5—C6—C7—N1	177.29 (9)	C16—C17—C18—C19	−1.57 (15)
C7—N1—C8—C9	31.92 (13)	C17—C18—C19—C20	0.27 (15)
C7—N1—C8—C13	−151.68 (9)	C18—C19—C20—O3	179.90 (9)
C13—C8—C9—C10	1.17 (14)	C18—C19—C20—C15	1.03 (15)
N1—C8—C9—C10	177.55 (9)	C16—C15—C20—O3	−179.87 (9)
C8—C9—C10—C11	−2.66 (16)	C14—C15—C20—O3	−0.66 (14)
C9—C10—C11—C12	1.44 (16)	C16—C15—C20—C19	−1.03 (14)
C10—C11—C12—C13	1.25 (16)	C14—C15—C20—C19	178.18 (9)
C11—C12—C13—C8	−2.70 (15)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O1···N1	0.86	1.78	2.5811 (13)	153
O3—H1O3···N2	0.90	1.82	2.6285 (14)	148
C7—H7A···Cg2^{i}	0.93	2.82	3.2893 (11)	112
C22—H22C···Cg1^{i}	0.96	2.84	3.7312 (12)	155

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

Fig. 1

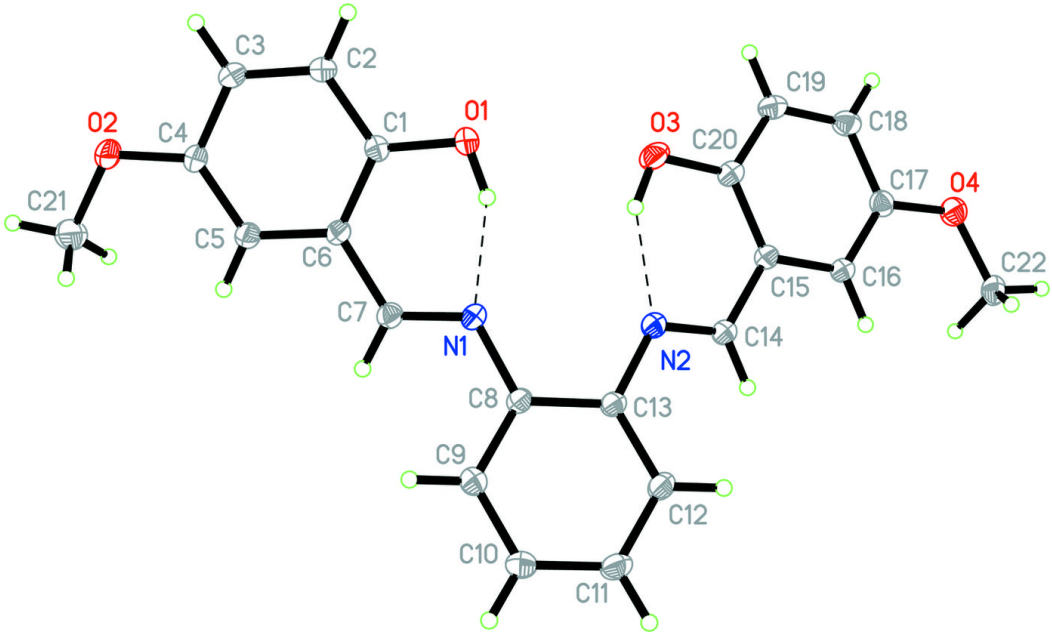
C3
C2
O2
C4
C1
O1
C21
C5
C6
C7
N1
C8
C9
C10
C11
C12
C13
N2
C14
C15
C16
C17
O4
C22
C18
C19
O3
C20

Fig. 2