Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,2-Bis[(2-hydroxy-3-methoxyphenyl)-methylamino]benzene

Yi-Feng Chen[a] and Seik Weng Ng[b]*

[a]College of Chemistry, Central China Normal University, Wuhan 430079, People's Republic of China, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

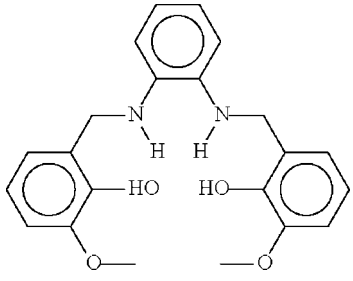
Received 17 May 2007; accepted 18 May 2007

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.132; data-to-parameter ratio = 16.4.

The title compound, $C_{22}H_{24}N_2O_4$, is a disubstituted α,α'-*o*-phenylenediamine whose hydroxyl groups engage in intermolecular O—H···N hydrogen bonding to form a chain. The amino groups have pyramidal configurations; their H atoms are not involved in any strong hydrogen-bonding interactions.

Related literature

For related structures and background, see: Bi *et al.* (2007); Przychodzeń *et al.* (2005); Wu *et al.* (2006) and Salmon *et al.* (2006).



Experimental

Crystal data

$C_{22}H_{24}N_2O_4$
M_r = 380.43
Orthorhombic, *Pbca*
a = 10.4665 (7) Å
b = 15.778 (1) Å
c = 23.439 (2) Å
V = 3870.8 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 295 (2) K
0.37 × 0.27 × 0.18 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: none
21495 measured reflections
4411 independent reflections
2372 reflections with $I > 2\sigma(I)$
R_{int} = 0.052

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.132
S = 0.99
4411 reflections
269 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2*o*···N1^{i}	0.86 (1)	2.08 (2)	2.834 (2)	146 (3)
O3—H3*o*···N2^{ii}	0.86 (1)	2.20 (1)	3.035 (2)	163 (2)
N1—H1*n*···O2	0.86 (1)	2.56 (2)	2.945 (2)	108 (2)
N2—H2*n*···O3	0.86 (1)	2.55 (2)	3.080 (2)	121 (2)
N2—H2*n*···O4^{i}	0.86 (1)	2.57 (2)	3.055 (2)	117 (2)

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

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

The authors thank Central China Normal University and the University of Malaya for generously supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2420).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bi, W.-Y., Lü, X.-Q., Cai, W.-L., Song, J.-R. & Ng, S. W. (2007). *Acta Cryst.* E**63**, o1615–o1616.
Bruker (2003). *SMART* (Version 6.28a) and *SAINT* (Version 6.28a). Bruker AXS Inc., Madison, Wisconsin, USA.
Przychodzeń, P., Rams, M., Guyard-Duhayon, C. & Sieklucka, B. (2005). *Inorg. Chem. Commun.* **8**, 350–354.
Salmon, L., Thuery, P. & Ephritikhine, M. (2006). Private Communication to the Cambridge Structural Database. (Deposition No. 296460).
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Westrip, S. P. (2007). *publCIF*. In preparation.
Wu, L.-B., Hu, Z.-Q. & Lai, G. Q. (2006). *Chin. J. Struct. Chem.* **25**, 567–571.

  doi:10.1107/S160053680702452X

supplementary materials

Acta Cryst. (2007). E**63**, o2988 [**doi:10.1107/S160053680702452X**]

1,2-Bis[(2-hydroxy-3-methoxyphenyl)methylamino]benzene

Y.-F. Chen and S. W. Ng

Comment

In this study, we synthesized the Schiff base, 1,2-bis(2-hydroxy-3-methoxybenzylideneamino)benzene, whose constitution is similar to that of the heterocyclic analog, 3,4-bis(2-hydroxy-3-methoxybenzylideneamino)pyridine, and whose structure we have recently reported (Bi *et al.*, 2007). We then reduced the carbon–nitrogen double-bond of the Schiff base to form the title double-amine, which should function as a flexible tetradentate chelate in the formation of metal complexes.

For 1,2-bis(2-hydroxy-3-methoxybenzylideneamino)benzene Schiff base itself, the rigid nature is seen is the strained bond dimensions found in, for example, the manganese (Przychodzeń *et al.*, 2005), cobalt (Wu *et al.*, 2006) and copper (Salmon *et al.*, 2005) complexes.

Experimental

The precursor bis(3-methoxysalicylaldimine)-*o*-phenylene was prepared by condensing 3-methoxysalicylaldehyde with *o*-phenylenediamine. To a solution of the Schiff base (0.38 g, 1 mol) in methanol (80 ml) at room temperature was added solid potassium borohydride (0.27 g, 5 mol). The solution was stirred for several hours until the yellow color had disappeared. The solvent was removed and dichloromethane (50 ml) was added. A small amount of anhydrous sodium sulfate was added to remove traces of water. The colorless solution was dried to obtain a brown solid. This was purified by recrystallization from anhydrous methanol to give brown crystals of (I) in about 60% yield. CH&N elemental analysis. Calculated for $C_{22}H_{24}N_2O_4$: C 69.46, H 6.36, N 7.36; found: C 69.43, H 6.44, N 7.29%.

Refinement

Carbon-bound hydrogen atoms were placed in calculated positions (C—H 0.93–0.96 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

The amino and hydroxy H-atoms were located in a difference Fourier map, and were refined with a distance restraint of N—H = O—H = 0.85±0.01 Å; their U_{iso} vaules were freely refined.

Figures

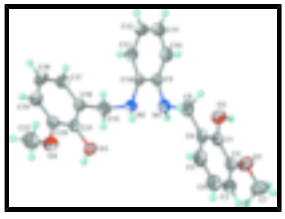


Fig. 1. View of (I) as a displacement ellipsoid plot (50% probability). Hydrogen atoms are drawn as spheres of arbitrary radius.

1,2-Bis[(2-hydroxy-3-methoxyphenyl)methylamino]benzene

Crystal data

$C_{22}H_{24}N_2O_4$	F_{000} = 1616
M_r = 380.43	D_x = 1.306 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 3418 reflections
a = 10.4665 (7) Å	θ = 2.6–21.5º
b = 15.778 (1) Å	μ = 0.09 mm^{-1}
c = 23.439 (2) Å	*T* = 295 (2) K
V = 3870.8 (4) Å^3	Block, brown
Z = 8	0.37 × 0.27 × 0.18 mm

Data collection

Bruker APEX CCD area-detector diffractometer	2372 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.052
Monochromator: graphite	θ_{max} = 27.5º
T = 295(2) K	θ_{min} = 2.5º
φ and ω scans	*h* = −13→13
Absorption correction: none	*k* = −20→19
21495 measured reflections	*l* = −30→22
4411 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.045	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.132	$w = 1/[\sigma^2(F_o^2) + (0.0522P)^2 + 0.9447P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.99	$(\Delta/\sigma)_{max}$ = 0.001
4411 reflections	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
269 parameters	$\Delta\rho_{min}$ = −0.14 e Å^{-3}
4 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.32120 (15)	0.66198 (11)	0.60670 (6)	0.0675 (5)
O2	0.36109 (14)	0.67543 (11)	0.71842 (7)	0.0606 (4)
O3	0.86678 (13)	0.46432 (10)	0.76967 (6)	0.0504 (4)
O4	1.08944 (13)	0.50169 (11)	0.81913 (6)	0.0674 (5)
N1	0.59204 (16)	0.65856 (11)	0.78926 (7)	0.0459 (4)
N2	0.59019 (15)	0.49037 (11)	0.81097 (7)	0.0426 (4)
C1	0.44971 (19)	0.70552 (13)	0.68120 (9)	0.0467 (5)
C2	0.4333 (2)	0.70033 (13)	0.62250 (9)	0.0504 (5)
C3	0.5247 (2)	0.73288 (15)	0.58635 (10)	0.0622 (6)
H3	0.5144	0.7293	0.5470	0.075*
C4	0.6333 (2)	0.77142 (17)	0.60961 (11)	0.0719 (7)
H4	0.6950	0.7942	0.5855	0.086*
C5	0.6496 (2)	0.77591 (15)	0.66762 (11)	0.0645 (7)
H5	0.7226	0.8012	0.6825	0.077*
C6	0.55805 (18)	0.74309 (13)	0.70423 (9)	0.0480 (5)
C7	0.2956 (3)	0.65090 (18)	0.54835 (10)	0.0794 (8)
H7A	0.2146	0.6231	0.5438	0.119*
H7B	0.3615	0.6168	0.5315	0.119*
H7C	0.2931	0.7052	0.5299	0.119*
C8	0.5692 (2)	0.74491 (13)	0.76782 (9)	0.0549 (6)
H8A	0.6393	0.7816	0.7789	0.066*
H8B	0.4912	0.7673	0.7843	0.066*
C9	0.54105 (17)	0.63253 (13)	0.84202 (8)	0.0439 (5)
C10	0.4937 (2)	0.68866 (16)	0.88249 (9)	0.0625 (6)
H10	0.4947	0.7467	0.8755	0.075*
C11	0.4449 (2)	0.65783 (19)	0.93341 (10)	0.0743 (8)
H11	0.4115	0.6954	0.9601	0.089*
C12	0.4452 (2)	0.57282 (19)	0.94485 (9)	0.0687 (7)
H12	0.4114	0.5527	0.9790	0.082*
C13	0.49600 (19)	0.51689 (15)	0.90553 (8)	0.0531 (5)
H13	0.4975	0.4592	0.9138	0.064*
C14	0.54468 (17)	0.54540 (13)	0.85409 (8)	0.0405 (5)
C15	0.65038 (19)	0.40989 (13)	0.82723 (9)	0.0483 (5)
H15A	0.6634	0.3760	0.7932	0.058*
H15B	0.5928	0.3790	0.8521	0.058*
C16	0.77694 (19)	0.42103 (12)	0.85726 (8)	0.0447 (5)
C17	0.7925 (2)	0.40482 (14)	0.91521 (9)	0.0590 (6)
H17	0.7243	0.3835	0.9362	0.071*
C18	0.9069 (3)	0.41991 (17)	0.94171 (10)	0.0749 (8)
H18	0.9159	0.4084	0.9804	0.090*
C19	1.0091 (2)	0.45205 (16)	0.91134 (10)	0.0684 (7)
H19	1.0864	0.4625	0.9297	0.082*
C20	0.9966 (2)	0.46862 (14)	0.85399 (9)	0.0524 (5)

C21	0.88098 (19)	0.45128 (12)	0.82675 (8)	0.0428 (5)
C22	1.2061 (2)	0.52865 (19)	0.84492 (11)	0.0809 (8)
H22A	1.2624	0.5504	0.8161	0.121*
H22B	1.2458	0.4814	0.8637	0.121*
H22C	1.1885	0.5723	0.8723	0.121*
H2O	0.2899 (16)	0.6617 (18)	0.7028 (11)	0.111 (11)*
H3O	0.9387 (14)	0.4747 (16)	0.7532 (9)	0.084 (9)*
H1N	0.5700 (19)	0.6231 (11)	0.7634 (7)	0.053 (6)*
H2N	0.6401 (15)	0.5163 (11)	0.7879 (7)	0.042 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0620 (10)	0.0901 (13)	0.0504 (9)	−0.0097 (9)	−0.0049 (8)	−0.0025 (8)
O2	0.0473 (9)	0.0791 (12)	0.0555 (10)	−0.0105 (8)	0.0002 (8)	0.0063 (8)
O3	0.0445 (8)	0.0652 (10)	0.0414 (8)	−0.0049 (8)	−0.0054 (7)	0.0027 (7)
O4	0.0427 (8)	0.1014 (14)	0.0581 (10)	−0.0082 (9)	−0.0091 (7)	−0.0113 (9)
N1	0.0495 (10)	0.0381 (10)	0.0501 (11)	0.0026 (8)	−0.0049 (8)	−0.0011 (8)
N2	0.0432 (9)	0.0437 (10)	0.0411 (10)	0.0003 (8)	0.0025 (8)	−0.0007 (8)
C1	0.0440 (11)	0.0421 (12)	0.0540 (13)	0.0062 (10)	0.0009 (10)	0.0054 (9)
C2	0.0465 (12)	0.0496 (13)	0.0552 (13)	0.0064 (10)	−0.0020 (10)	0.0029 (10)
C3	0.0634 (15)	0.0695 (17)	0.0539 (14)	0.0108 (13)	0.0049 (12)	0.0087 (12)
C4	0.0569 (14)	0.0809 (19)	0.0779 (18)	−0.0008 (13)	0.0124 (13)	0.0230 (14)
C5	0.0486 (13)	0.0611 (16)	0.0839 (18)	−0.0048 (11)	−0.0050 (12)	0.0166 (13)
C6	0.0447 (11)	0.0366 (11)	0.0627 (13)	0.0052 (9)	−0.0059 (10)	0.0077 (10)
C7	0.0802 (18)	0.105 (2)	0.0529 (15)	−0.0062 (16)	−0.0019 (13)	−0.0120 (14)
C8	0.0557 (13)	0.0400 (12)	0.0689 (15)	0.0010 (11)	−0.0137 (11)	−0.0005 (10)
C9	0.0380 (10)	0.0528 (13)	0.0409 (11)	0.0045 (9)	−0.0087 (9)	−0.0052 (9)
C10	0.0689 (15)	0.0640 (16)	0.0544 (14)	0.0181 (12)	−0.0107 (12)	−0.0135 (11)
C11	0.0770 (17)	0.096 (2)	0.0496 (15)	0.0328 (16)	−0.0064 (12)	−0.0218 (14)
C12	0.0580 (14)	0.107 (2)	0.0413 (13)	0.0199 (15)	0.0027 (11)	−0.0002 (13)
C13	0.0435 (11)	0.0702 (15)	0.0455 (12)	0.0044 (11)	−0.0001 (10)	0.0032 (11)
C14	0.0315 (9)	0.0523 (13)	0.0378 (10)	0.0039 (9)	−0.0067 (8)	−0.0036 (9)
C15	0.0513 (12)	0.0391 (12)	0.0546 (13)	−0.0032 (10)	0.0028 (10)	−0.0029 (9)
C16	0.0523 (12)	0.0354 (11)	0.0463 (12)	0.0069 (9)	−0.0020 (10)	−0.0015 (9)
C17	0.0664 (15)	0.0620 (15)	0.0487 (13)	0.0086 (12)	0.0052 (12)	0.0084 (11)
C18	0.0862 (19)	0.094 (2)	0.0442 (14)	0.0218 (16)	−0.0122 (14)	0.0050 (13)
C19	0.0602 (14)	0.0934 (19)	0.0515 (14)	0.0155 (14)	−0.0145 (12)	−0.0066 (13)
C20	0.0477 (12)	0.0617 (15)	0.0478 (12)	0.0077 (11)	−0.0072 (10)	−0.0092 (10)
C21	0.0482 (12)	0.0391 (11)	0.0409 (11)	0.0071 (9)	−0.0078 (9)	−0.0031 (9)
C22	0.0468 (13)	0.113 (2)	0.0827 (18)	−0.0067 (14)	−0.0139 (13)	−0.0261 (16)

Geometric parameters (Å, °)

O1—C2	1.372 (3)	C8—H8A	0.9700
O1—C7	1.405 (3)	C8—H8B	0.9700
O2—C1	1.359 (2)	C9—C10	1.389 (3)
O2—H2O	0.858 (10)	C9—C14	1.404 (3)
O3—C21	1.362 (2)	C10—C11	1.387 (3)

O3—H3O	0.862 (10)	C10—H10	0.9300
O4—C20	1.373 (3)	C11—C12	1.368 (4)
O4—C22	1.427 (2)	C11—H11	0.9300
N1—C9	1.408 (2)	C12—C13	1.382 (3)
N1—C8	1.472 (3)	C12—H12	0.9300
N1—H1N	0.857 (9)	C13—C14	1.384 (3)
N2—C14	1.415 (2)	C13—H13	0.9300
N2—C15	1.468 (3)	C15—C16	1.510 (3)
N2—H2N	0.856 (9)	C15—H15A	0.9700
C1—C6	1.389 (3)	C15—H15B	0.9700
C1—C2	1.389 (3)	C16—C21	1.387 (3)
C2—C3	1.377 (3)	C16—C17	1.392 (3)
C3—C4	1.400 (3)	C17—C18	1.370 (3)
C3—H3	0.9300	C17—H17	0.9300
C4—C5	1.372 (3)	C18—C19	1.381 (3)
C4—H4	0.9300	C18—H18	0.9300
C5—C6	1.387 (3)	C19—C20	1.376 (3)
C5—H5	0.9300	C19—H19	0.9300
C6—C8	1.495 (3)	C20—C21	1.395 (3)
C7—H7A	0.9600	C22—H22A	0.9600
C7—H7B	0.9600	C22—H22B	0.9600
C7—H7C	0.9600	C22—H22C	0.9600
C2—O1—C7	118.78 (18)	C11—C10—H10	120.1
C1—O2—H2O	114 (2)	C9—C10—H10	120.1
C21—O3—H3O	111.9 (17)	C12—C11—C10	120.8 (2)
C20—O4—C22	117.84 (18)	C12—C11—H11	119.6
C9—N1—C8	120.56 (17)	C10—C11—H11	119.6
C9—N1—H1N	109.2 (14)	C11—C12—C13	119.8 (2)
C8—N1—H1N	108.6 (14)	C11—C12—H12	120.1
C14—N2—C15	119.34 (16)	C13—C12—H12	120.1
C14—N2—H2N	111.3 (13)	C12—C13—C14	121.0 (2)
C15—N2—H2N	108.4 (13)	C12—C13—H13	119.5
O2—C1—C6	117.19 (18)	C14—C13—H13	119.5
O2—C1—C2	122.08 (19)	C13—C14—C9	118.94 (18)
C6—C1—C2	120.7 (2)	C13—C14—N2	123.13 (19)
O1—C2—C3	126.3 (2)	C9—C14—N2	117.78 (17)
O1—C2—C1	113.52 (18)	N2—C15—C16	113.37 (16)
C3—C2—C1	120.1 (2)	N2—C15—H15A	108.9
C2—C3—C4	119.1 (2)	C16—C15—H15A	108.9
C2—C3—H3	120.5	N2—C15—H15B	108.9
C4—C3—H3	120.5	C16—C15—H15B	108.9
C5—C4—C3	120.6 (2)	H15A—C15—H15B	107.7
C5—C4—H4	119.7	C21—C16—C17	118.33 (19)
C3—C4—H4	119.7	C21—C16—C15	119.22 (17)
C4—C5—C6	120.6 (2)	C17—C16—C15	122.41 (19)
C4—C5—H5	119.7	C18—C17—C16	120.8 (2)
C6—C5—H5	119.7	C18—C17—H17	119.6
C5—C6—C1	118.9 (2)	C16—C17—H17	119.6
C5—C6—C8	123.8 (2)	C17—C18—C19	120.5 (2)

C1—C6—C8	117.36 (19)	C17—C18—H18	119.8
O1—C7—H7A	109.5	C19—C18—H18	119.8
O1—C7—H7B	109.5	C20—C19—C18	120.0 (2)
H7A—C7—H7B	109.5	C20—C19—H19	120.0
O1—C7—H7C	109.5	C18—C19—H19	120.0
H7A—C7—H7C	109.5	O4—C20—C19	125.9 (2)
H7B—C7—H7C	109.5	O4—C20—C21	114.60 (18)
N1—C8—C6	109.58 (17)	C19—C20—C21	119.5 (2)
N1—C8—H8A	109.8	O3—C21—C16	118.21 (17)
C6—C8—H8A	109.8	O3—C21—C20	120.97 (19)
N1—C8—H8B	109.8	C16—C21—C20	120.82 (18)
C6—C8—H8B	109.8	O4—C22—H22A	109.5
H8A—C8—H8B	108.2	O4—C22—H22B	109.5
C10—C9—C14	119.76 (19)	H22A—C22—H22B	109.5
C10—C9—N1	123.3 (2)	O4—C22—H22C	109.5
C14—C9—N1	116.89 (16)	H22A—C22—H22C	109.5
C11—C10—C9	119.7 (2)	H22B—C22—H22C	109.5
C7—O1—C2—C3	2.4 (3)	C12—C13—C14—N2	−175.84 (19)
C7—O1—C2—C1	−178.2 (2)	C10—C9—C14—C13	2.5 (3)
O2—C1—C2—O1	−0.4 (3)	N1—C9—C14—C13	179.80 (16)
C6—C1—C2—O1	−179.61 (18)	C10—C9—C14—N2	178.18 (17)
O2—C1—C2—C3	178.97 (19)	N1—C9—C14—N2	−4.5 (2)
C6—C1—C2—C3	−0.2 (3)	C15—N2—C14—C13	−32.3 (3)
O1—C2—C3—C4	178.9 (2)	C15—N2—C14—C9	152.26 (17)
C1—C2—C3—C4	−0.3 (3)	C14—N2—C15—C16	−67.7 (2)
C2—C3—C4—C5	0.8 (4)	N2—C15—C16—C21	−68.9 (2)
C3—C4—C5—C6	−0.6 (4)	N2—C15—C16—C17	108.8 (2)
C4—C5—C6—C1	0.1 (3)	C21—C16—C17—C18	1.2 (3)
C4—C5—C6—C8	179.5 (2)	C15—C16—C17—C18	−176.5 (2)
O2—C1—C6—C5	−178.86 (19)	C16—C17—C18—C19	0.4 (4)
C2—C1—C6—C5	0.4 (3)	C17—C18—C19—C20	−0.4 (4)
O2—C1—C6—C8	1.7 (3)	C22—O4—C20—C19	−6.0 (3)
C2—C1—C6—C8	−179.09 (18)	C22—O4—C20—C21	174.1 (2)
C9—N1—C8—C6	−145.63 (18)	C18—C19—C20—O4	178.9 (2)
C5—C6—C8—N1	−109.3 (2)	C18—C19—C20—C21	−1.2 (3)
C1—C6—C8—N1	70.2 (2)	C17—C16—C21—O3	177.79 (18)
C8—N1—C9—C10	−15.4 (3)	C15—C16—C21—O3	−4.4 (3)
C8—N1—C9—C14	167.46 (17)	C17—C16—C21—C20	−2.8 (3)
C14—C9—C10—C11	−3.0 (3)	C15—C16—C21—C20	174.97 (18)
N1—C9—C10—C11	179.90 (19)	O4—C20—C21—O3	2.1 (3)
C9—C10—C11—C12	1.4 (4)	C19—C20—C21—O3	−177.81 (19)
C10—C11—C12—C13	0.7 (4)	O4—C20—C21—C16	−177.26 (18)
C11—C12—C13—C14	−1.2 (3)	C19—C20—C21—C16	2.8 (3)
C12—C13—C14—C9	−0.4 (3)		

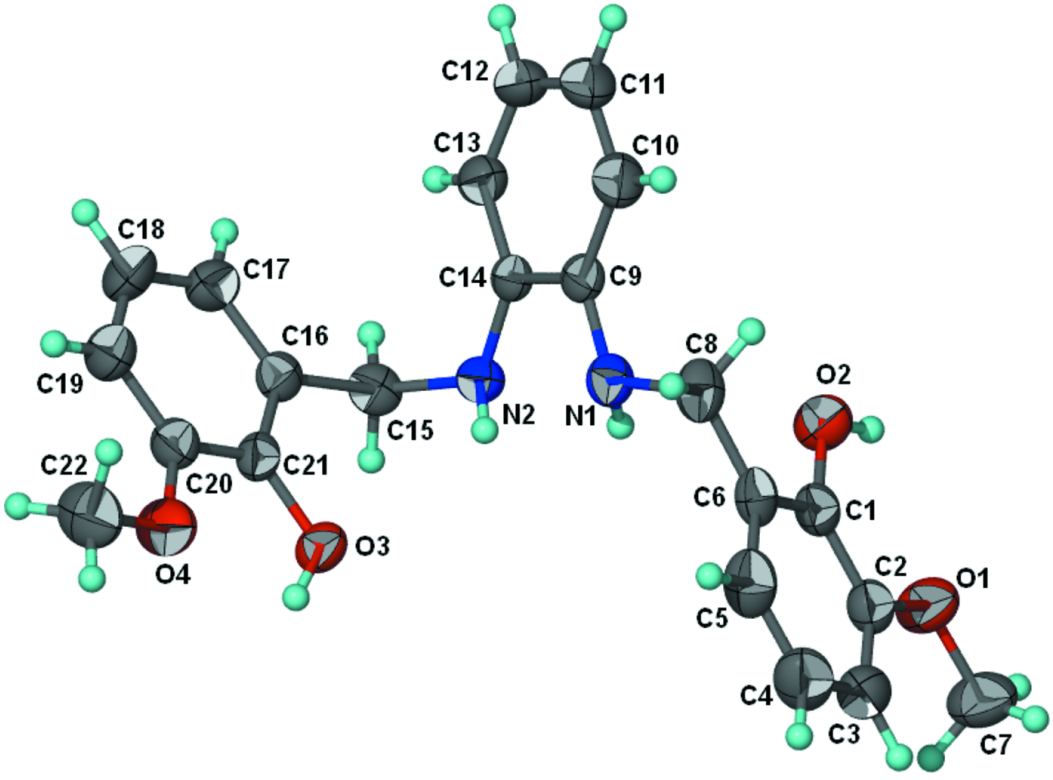
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2O···N1^{i}	0.86 (1)	2.08 (2)	2.834 (2)	146 (3)

O3—H3O···N2^{ii}	0.86 (1)	2.20 (1)	3.035 (2)	163 (2)
N1—H1N···O2	0.86 (1)	2.56 (2)	2.945 (2)	108 (2)
N2—H2N···O3	0.86 (1)	2.55 (2)	3.080 (2)	121 (2)
N2—H2N···O4^{i}	0.86 (1)	2.57 (2)	3.055 (2)	117 (2)

Symmetry codes: (i) $x-1/2, y, -z+3/2$; (ii) $x+1/2, y, -z+3/2$.

Fig. 1


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