Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6,6′-Dimethoxy-2,2′-(*o*-phenylenediimino)diphenol

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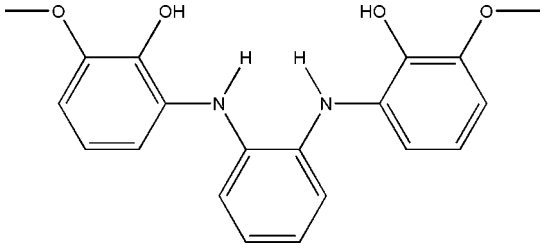
Received 4 June 2007; accepted 6 June 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.047; wR factor = 0.096; data-to-parameter ratio = 12.8.

In the title compound, $C_{22}H_{24}N_2O_4$, the dihedral angles between the central benzene ring and the other two benzene rings are 70.26 (8) and 84.40 (8)°. The molecules are linked into chains running parallel to the [001] direction by O—H···N and N—H···O hydrogen bonds, and adjacent chains are linked into sheets parallel to the (001) plane by C—H···π hydrogen bonds.

Related literature

For related literature, see: Allen *et al.* (1987); Bernstein *et al.* (1995); Liu *et al.* (2007); Xia *et al.* (2007).



Experimental

Crystal data

$C_{22}H_{24}N_2O_4$
M_r = 380.43
Orthorhombic, *Pbca*
a = 10.4331 (14) Å
b = 15.7674 (16) Å
c = 23.398 (2) Å
V = 3849.1 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.38 × 0.21 × 0.10 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.966, T_{max} = 0.991
14963 measured reflections
3383 independent reflections
1530 reflections with $I > 2\sigma(I)$
R_{int} = 0.091

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.096
S = 0.81
3383 reflections
265 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

*Cg*1 is the centroid of ring C16–C21.

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N1^{i}	0.87 (3)	2.04 (3)	2.821 (3)	149 (3)
O3—H3···N2^{ii}	0.88 (3)	2.18 (3)	3.038 (3)	166 (2)
N2—H2···O4^{i}	0.91 (2)	2.55 (2)	3.052 (3)	115.5 (17)
N1—H1···O1	0.89 (2)	2.50 (2)	2.940 (3)	111.3 (18)
N2—H2···O3	0.91 (2)	2.50 (2)	3.065 (3)	120.1 (18)
N2—H2···N1	0.91 (2)	2.28 (2)	2.694 (3)	107.4 (17)
N1—H1···N2	0.89 (2)	2.40 (2)	2.694 (3)	99.6 (16)
O1—H1A···O2	0.87 (3)	2.29 (3)	2.659 (2)	106 (2)
O3—H3···O4	0.88 (3)	2.27 (3)	2.665 (3)	107 (2)
C7—H7B···O1	0.97	2.52	2.696 (3)	90
C6—H6···*Cg*1^{iii}	0.93	3.23	4.087 (9)	155

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

The authors acknowledge the financial support of the Huaihai Institute of Technology Science Foundation.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2321).

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  doi:10.1107/S1600536807027778

supplementary materials

Acta Cryst. (2007). E**63**, o3562 [**doi:10.1107/S1600536807027778**]

6,6'-Dimethoxy-2,2'-(*o*-phenylenediimino)diphenol

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Comment

As part of our investigation of *o*-vanillin diamine derivatives, we have recently reported the crystal structure of *N*,*N*'-(2-hydroxy-3-methoxy benzyl benzene-1,4-diamine (II) (Xia *et al.*, 2007) and *N*,*N*'-(2-hydroxy-3-methoxy benzyl) benzene-1,3-diamine (III) (Liu *et al.,* 2007), we report here the their isocompound, crystal structure of *N*,*N*'-(2-hydroxy-3-methoxy benzyl benzene-1,2-diamine, (I).

In the molecule, the dihedral angle between the certral benzen ring and another two benzen rings are 70.26(0.08)°(C9—C13 benzen ring) and 84.40(0.08)°(C16—C21 benzen ring), respectively (Fig. 1). Its bond lengths and angles are similar with compounds (II), (III) and are normal (Allen *et al.*, 1987). The principal difference between compounds (I) and (II), (III) concerns the intermoleculae aggregation. In (I), The molecular are linked into chains running parallel to the [001] directions by O—H···N and N—H···O hydrogen bonds. Atoms O1 and O3 in the molecule (*x*, *y*, *z*) act as hydrogen-bond donors to atoms N1 in the molecule (−1/2 + *x*, *y*, 3/2 − *z*) and N2 in the molecule (1/2 + *x*, *y*, 3/2 − *z*), respectively, in addition, atom N2 in the molecule (*x*, *y*, *z*) act as hydrogen-bond donor to atom O4 in the molecule (−1/2 + *x*, *y*, 3/2 − *z*), forming a chain of $R_2^2(7)$ ring (Bernstein *et al.*, 1995) running parallel to the [001] direction (Fig. 2 and Table 1), adjacent chains are linked into sheets parallel to the [001] plane by C—H···π hydrogen bonds (Fig. 3). There are no direction-specific interactions between adjacent sheets and are in agreement with their isocompound. By contrast, in (II), the molecule are linked into sheets by means of N—H···O and O—H···N hydrogen-bonds and in (III), the molecule are linked into sheets by means of O—H···O and C—H···π hydrogen-bonds, they are absent from the structure of (I).

Experimental

Solutions of *N*,*N*'-bis(2-hydroxy-3-methoxy benzylene)benzene-1,2-diamine (20 mmol) in methanol–chloroform (*v*/*v* = 1/1) (40 ml) and $NaBH_4$ (80 mmol) were mixed, the mixture solution was stirred under room temperature for 48 h and then mixtures was filtered, and then solution was left to produce crystals of (I) slowly.

Refinement

H atoms bonded to O and N atoms were found a difference Fourier map and refined freely. All other H atoms were place in geometrically calculated positions and allowed to ride on their respective parent atoms, with C—H distances of 0.93 Å (aryl), 0.96 Å (methyl), 0.97 Å (methylene), U_{iso}(H) = 1.2 ($C_{aryl,\ methylene}$) or 1.5 U_{eq}(C_{methyl}).

Figures

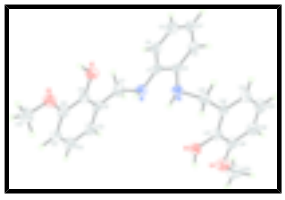

Fig. 1. The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are at the 30% probability level.

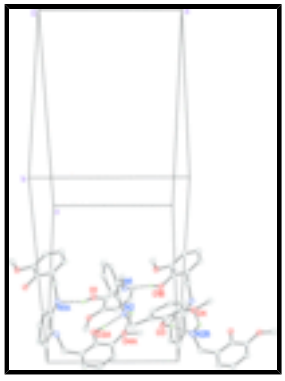

Fig. 2. A larger portion of the crystal structure of (I), showing the formation of a hydrogen-bonded chains built from O—H···N and N—H···O. For clarity, H atoms not involved in the hydrogen bonding have been omitted. Dashed lines indicate hydrogen bonds. [Symmetry code: (A) −1/2 + *x*, *y*, 3/2 − *z*; (B) 1/2 + *x*, *y*, 3/2 − *z*].

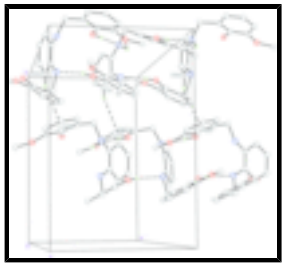

Fig. 3. A larger portion of the crystal structure of (I), showing the formation of a hydrogen-bonded sheets built from C—H···π. For clarity, H atoms not involved in the hydrogen bonding have been omitted. Dashed lines indicate hydrogen bonds. [Symmetry code: (A) −1/2 + *x*, *y*, 3/2 − *z*; (B) 1/2 + *x*, *y*, 3/2 − *z*; (C) 3/2 − *x*, −1/2 + *y*, *z*; (D) 2 − *x*, −1/2 + *y*, 3/2 − *z*; (E) 1 − *x*, −1/2 + *y*, 3/2 − *z*].

6,6'-Dimethoxy-2,2'-(*o*-phenylenediimino)diphenol

Crystal data

$C_{22}H_{24}N_2O_4$	F_{000} = 1616
M_r = 380.43	D_x = 1.313 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 1282 reflections
a = 10.4331 (14) Å	θ = 2.6–18.5º
b = 15.7674 (16) Å	μ = 0.09 mm^{-1}
c = 23.398 (2) Å	*T* = 298 (2) K
V = 3849.1 (7) Å^3	Acerate, red
Z = 8	0.38 × 0.21 × 0.10 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer	3383 independent reflections
Radiation source: fine-focus sealed tube	1530 reflections with $I > 2\sigma(I)$

Monochromator: graphite	R_{int} = 0.091
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −12→12
T_{min} = 0.966, T_{max} = 0.991	k = −18→9
14963 measured reflections	l = −27→27

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.047	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.096	$w = 1/[\sigma^2(F_o^2) + (0.0315P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.81	$(\Delta/\sigma)_{max} < 0.001$
3383 reflections	$\Delta\rho_{max}$ = 0.12 e Å^{-3}
265 parameters	$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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}
N1	0.5932 (2)	0.84158 (13)	0.71070 (9)	0.0485 (6)
H1	0.566 (2)	0.8767 (14)	0.7378 (9)	0.058*
N2	0.5906 (2)	1.00932 (13)	0.68887 (9)	0.0456 (6)
H2	0.644 (2)	0.9819 (14)	0.7137 (9)	0.055*
O1	0.36186 (18)	0.82490 (13)	0.78154 (8)	0.0624 (6)
H1A	0.288 (3)	0.8358 (19)	0.7969 (12)	0.094*
O2	0.32169 (19)	0.83823 (12)	0.89338 (7)	0.0701 (6)
O3	0.86629 (16)	1.03619 (11)	0.73032 (7)	0.0534 (5)
H3	0.938 (3)	1.0251 (17)	0.7482 (11)	0.080*
O4	1.08934 (18)	0.99872 (13)	0.68090 (8)	0.0696 (6)

C1	0.5414 (2)	0.86734 (17)	0.65774 (10)	0.0450 (7)
C2	0.5445 (2)	0.95466 (16)	0.64562 (10)	0.0416 (6)
C3	0.4963 (2)	0.98313 (18)	0.59464 (10)	0.0536 (7)
H3A	0.4980	1.0408	0.5864	0.064*
C4	0.4451 (3)	0.9273 (2)	0.55528 (12)	0.0678 (9)
H4	0.4110	0.9476	0.5212	0.081*
C5	0.4446 (3)	0.8420 (2)	0.56652 (12)	0.0756 (10)
H5	0.4114	0.8043	0.5398	0.091*
C6	0.4933 (3)	0.81202 (18)	0.61745 (12)	0.0617 (8)
H6	0.4937	0.7540	0.6247	0.074*
C7	0.5696 (3)	0.75466 (15)	0.73205 (11)	0.0571 (8)
H7A	0.6398	0.7178	0.7210	0.069*
H7B	0.4912	0.7325	0.7156	0.069*
C8	0.5585 (3)	0.75709 (16)	0.79603 (12)	0.0493 (7)
C9	0.4505 (3)	0.79434 (16)	0.81874 (11)	0.0466 (7)
C10	0.4332 (3)	0.80035 (17)	0.87713 (12)	0.0530 (7)
C11	0.5251 (3)	0.76689 (17)	0.91359 (12)	0.0621 (8)
H11	0.5141	0.7699	0.9530	0.075*
C12	0.6334 (3)	0.72896 (19)	0.89072 (14)	0.0706 (9)
H12	0.6952	0.7064	0.9150	0.085*
C13	0.6505 (3)	0.72439 (17)	0.83255 (14)	0.0649 (9)
H13	0.7239	0.6993	0.8177	0.078*
C14	0.2953 (3)	0.84900 (19)	0.95184 (11)	0.0816 (10)
H14A	0.2140	0.8767	0.9563	0.122*
H14B	0.3612	0.8831	0.9689	0.122*
H14C	0.2928	0.7946	0.9702	0.122*
C15	0.6505 (2)	1.09019 (15)	0.67237 (10)	0.0491 (7)
H15A	0.6634	1.1243	0.7064	0.059*
H15B	0.5924	1.1208	0.6474	0.059*
C16	0.7768 (3)	1.07911 (15)	0.64244 (11)	0.0434 (7)
C17	0.8811 (3)	1.04933 (15)	0.67309 (11)	0.0446 (7)
C18	0.9967 (3)	1.03145 (17)	0.64600 (11)	0.0536 (7)
C19	1.0088 (3)	1.04800 (19)	0.58878 (12)	0.0713 (9)
H19	1.0863	1.0375	0.5705	0.086*
C20	0.9071 (3)	1.08003 (19)	0.55801 (12)	0.0741 (10)
H20	0.9163	1.0914	0.5192	0.089*
C21	0.7921 (3)	1.09514 (16)	0.58479 (12)	0.0615 (8)
H21	0.7237	1.1165	0.5638	0.074*
C22	1.2059 (3)	0.9721 (2)	0.65500 (12)	0.0846 (10)
H22A	1.2626	0.9502	0.6838	0.127*
H22B	1.2457	1.0195	0.6363	0.127*
H22C	1.1883	0.9286	0.6274	0.127*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0537 (16)	0.0395 (14)	0.0523 (15)	−0.0008 (12)	0.0054 (13)	−0.0033 (11)
N2	0.0469 (15)	0.0448 (14)	0.0452 (14)	0.0015 (12)	−0.0027 (11)	0.0015 (12)

O1	0.0496 (13)	0.0782 (14)	0.0594 (13)	0.0092 (12)	0.0015 (11)	0.0044 (11)
O2	0.0665 (15)	0.0902 (15)	0.0536 (13)	0.0108 (12)	0.0058 (11)	−0.0026 (12)
O3	0.0457 (13)	0.0683 (12)	0.0462 (12)	0.0048 (11)	0.0029 (9)	0.0050 (10)
O4	0.0449 (13)	0.1012 (16)	0.0627 (13)	0.0104 (12)	0.0079 (11)	−0.0100 (12)
C1	0.0378 (17)	0.0527 (19)	0.0443 (16)	−0.0038 (14)	0.0055 (14)	−0.0055 (15)
C2	0.0323 (16)	0.0504 (17)	0.0421 (16)	−0.0033 (13)	0.0033 (14)	−0.0040 (14)
C3	0.0459 (18)	0.0672 (19)	0.0477 (17)	−0.0066 (15)	−0.0029 (15)	0.0022 (16)
C4	0.055 (2)	0.102 (3)	0.0469 (18)	−0.012 (2)	−0.0022 (15)	0.003 (2)
C5	0.080 (3)	0.096 (3)	0.051 (2)	−0.028 (2)	0.0026 (18)	−0.019 (2)
C6	0.067 (2)	0.0619 (19)	0.0558 (19)	−0.0155 (17)	0.0093 (18)	−0.0101 (17)
C7	0.059 (2)	0.0410 (16)	0.072 (2)	−0.0004 (15)	0.0108 (16)	0.0000 (15)
C8	0.0458 (19)	0.0385 (16)	0.0636 (19)	−0.0048 (15)	0.0064 (17)	0.0065 (15)
C9	0.048 (2)	0.0407 (16)	0.0509 (18)	−0.0022 (15)	−0.0034 (17)	0.0045 (14)
C10	0.047 (2)	0.0532 (18)	0.059 (2)	−0.0049 (16)	−0.0007 (17)	0.0014 (16)
C11	0.061 (2)	0.070 (2)	0.0558 (18)	−0.0108 (18)	−0.0051 (19)	0.0066 (17)
C12	0.056 (2)	0.080 (2)	0.076 (2)	−0.0001 (18)	−0.0115 (19)	0.0193 (19)
C13	0.050 (2)	0.060 (2)	0.085 (2)	0.0046 (16)	0.0033 (19)	0.0113 (18)
C14	0.089 (3)	0.098 (3)	0.059 (2)	0.010 (2)	0.0088 (18)	−0.0121 (19)
C15	0.057 (2)	0.0383 (16)	0.0518 (16)	0.0008 (15)	−0.0050 (15)	0.0010 (14)
C16	0.0483 (18)	0.0363 (15)	0.0456 (16)	−0.0060 (14)	0.0047 (15)	0.0006 (13)
C17	0.053 (2)	0.0412 (16)	0.0399 (16)	−0.0052 (14)	0.0068 (15)	−0.0047 (13)
C18	0.0483 (19)	0.0637 (19)	0.0488 (17)	−0.0034 (16)	0.0086 (17)	−0.0065 (16)
C19	0.060 (2)	0.094 (2)	0.060 (2)	−0.011 (2)	0.0150 (19)	−0.0087 (19)
C20	0.083 (3)	0.094 (2)	0.0453 (18)	−0.020 (2)	0.014 (2)	0.0037 (18)
C21	0.068 (2)	0.0632 (19)	0.0531 (19)	−0.0116 (18)	−0.0052 (17)	0.0103 (16)
C22	0.051 (2)	0.117 (3)	0.085 (2)	0.009 (2)	0.0109 (19)	−0.022 (2)

Geometric parameters (Å, °)

N1—C1	1.412 (3)	C8—C9	1.377 (3)
N1—C7	1.479 (3)	C8—C13	1.384 (3)
N1—H1	0.89 (2)	C9—C10	1.381 (3)
N2—C2	1.413 (3)	C10—C11	1.387 (3)
N2—C15	1.472 (3)	C11—C12	1.386 (4)
N2—H2	0.91 (2)	C11—H11	0.9300
O1—C9	1.358 (3)	C12—C13	1.375 (4)
O1—H1A	0.87 (3)	C12—H12	0.9300
O2—C10	1.362 (3)	C13—H13	0.9300
O2—C14	1.405 (3)	C14—H14A	0.9600
O3—C17	1.364 (3)	C14—H14B	0.9600
O3—H3	0.88 (3)	C14—H14C	0.9600
O4—C18	1.366 (3)	C15—C16	1.503 (3)
O4—C22	1.423 (3)	C15—H15A	0.9700
C1—C6	1.379 (3)	C15—H15B	0.9700
C1—C2	1.406 (3)	C16—C21	1.382 (3)
C2—C3	1.370 (3)	C16—C17	1.385 (3)
C3—C4	1.381 (3)	C17—C18	1.392 (3)
C3—H3A	0.9300	C18—C19	1.370 (3)
C4—C5	1.371 (4)	C19—C20	1.378 (4)

C4—H4	0.9300	C19—H19	0.9300
C5—C6	1.379 (3)	C20—C21	1.374 (4)
C5—H5	0.9300	C20—H20	0.9300
C6—H6	0.9300	C21—H21	0.9300
C7—C8	1.502 (3)	C22—H22A	0.9600
C7—H7A	0.9700	C22—H22B	0.9600
C7—H7B	0.9700	C22—H22C	0.9600
C1—N1—C7	119.9 (2)	C12—C11—H11	120.3
C1—N1—H1	108.8 (15)	C10—C11—H11	120.3
C7—N1—H1	106.4 (15)	C13—C12—C11	120.7 (3)
C2—N2—C15	119.0 (2)	C13—C12—H12	119.6
C2—N2—H2	112.0 (14)	C11—C12—H12	119.6
C15—N2—H2	108.5 (15)	C12—C13—C8	120.1 (3)
C9—O1—H1A	114.3 (19)	C12—C13—H13	119.9
C10—O2—C14	119.5 (2)	C8—C13—H13	119.9
C17—O3—H3	113.7 (18)	O2—C14—H14A	109.5
C18—O4—C22	117.5 (2)	O2—C14—H14B	109.5
C6—C1—C2	119.3 (2)	H14A—C14—H14B	109.5
C6—C1—N1	123.9 (2)	O2—C14—H14C	109.5
C2—C1—N1	116.7 (2)	H14A—C14—H14C	109.5
C3—C2—C1	119.2 (2)	H14B—C14—H14C	109.5
C3—C2—N2	123.3 (2)	N2—C15—C16	113.2 (2)
C1—C2—N2	117.4 (2)	N2—C15—H15A	108.9
C2—C3—C4	120.9 (3)	C16—C15—H15A	108.9
C2—C3—H3A	119.5	N2—C15—H15B	108.9
C4—C3—H3A	119.5	C16—C15—H15B	108.9
C5—C4—C3	119.9 (3)	H15A—C15—H15B	107.7
C5—C4—H4	120.0	C21—C16—C17	118.5 (3)
C3—C4—H4	120.0	C21—C16—C15	122.4 (3)
C4—C5—C6	120.0 (3)	C17—C16—C15	119.1 (2)
C4—C5—H5	120.0	O3—C17—C16	118.1 (2)
C6—C5—H5	120.0	O3—C17—C18	121.0 (3)
C5—C6—C1	120.5 (3)	C16—C17—C18	120.9 (2)
C5—C6—H6	119.7	O4—C18—C19	126.2 (3)
C1—C6—H6	119.7	O4—C18—C17	114.7 (2)
N1—C7—C8	109.0 (2)	C19—C18—C17	119.1 (3)
N1—C7—H7A	109.9	C18—C19—C20	120.7 (3)
C8—C7—H7A	109.9	C18—C19—H19	119.7
N1—C7—H7B	109.9	C20—C19—H19	119.7
C8—C7—H7B	109.9	C21—C20—C19	119.8 (3)
H7A—C7—H7B	108.3	C21—C20—H20	120.1
C9—C8—C13	119.2 (3)	C19—C20—H20	120.1
C9—C8—C7	117.3 (3)	C20—C21—C16	121.0 (3)
C13—C8—C7	123.5 (3)	C20—C21—H21	119.5
O1—C9—C8	117.5 (2)	C16—C21—H21	119.5
O1—C9—C10	121.4 (3)	O4—C22—H22A	109.5
C8—C9—C10	121.2 (3)	O4—C22—H22B	109.5
O2—C10—C9	114.7 (3)	H22A—C22—H22B	109.5
O2—C10—C11	125.8 (3)	O4—C22—H22C	109.5

C9—C10—C11	119.5 (3)	H22A—C22—H22C	109.5
C12—C11—C10	119.3 (3)	H22B—C22—H22C	109.5
C7—N1—C1—C6	−15.2 (4)	C8—C9—C10—C11	−1.2 (4)
C7—N1—C1—C2	167.2 (2)	O2—C10—C11—C12	179.0 (2)
C6—C1—C2—C3	1.8 (4)	C9—C10—C11—C12	0.8 (4)
N1—C1—C2—C3	179.6 (2)	C10—C11—C12—C13	0.1 (4)
C6—C1—C2—N2	177.9 (2)	C11—C12—C13—C8	−0.6 (4)
N1—C1—C2—N2	−4.4 (3)	C9—C8—C13—C12	0.2 (4)
C15—N2—C2—C3	−31.7 (3)	C7—C8—C13—C12	−180.0 (3)
C15—N2—C2—C1	152.4 (2)	C2—N2—C15—C16	−68.3 (3)
C1—C2—C3—C4	−0.1 (4)	N2—C15—C16—C21	109.0 (3)
N2—C2—C3—C4	−175.9 (2)	N2—C15—C16—C17	−69.0 (3)
C2—C3—C4—C5	−1.4 (4)	C21—C16—C17—O3	178.0 (2)
C3—C4—C5—C6	1.0 (5)	C15—C16—C17—O3	−4.0 (3)
C4—C5—C6—C1	0.8 (4)	C21—C16—C17—C18	−3.5 (4)
C2—C1—C6—C5	−2.2 (4)	C15—C16—C17—C18	174.6 (2)
N1—C1—C6—C5	−179.7 (2)	C22—O4—C18—C19	−5.9 (4)
C1—N1—C7—C8	−145.5 (2)	C22—O4—C18—C17	174.6 (2)
N1—C7—C8—C9	70.7 (3)	O3—C17—C18—O4	1.5 (4)
N1—C7—C8—C13	−109.1 (3)	C16—C17—C18—O4	−177.0 (2)
C13—C8—C9—O1	−179.3 (2)	O3—C17—C18—C19	−178.0 (2)
C7—C8—C9—O1	0.9 (3)	C16—C17—C18—C19	3.5 (4)
C13—C8—C9—C10	0.7 (4)	O4—C18—C19—C20	179.0 (3)
C7—C8—C9—C10	−179.1 (2)	C17—C18—C19—C20	−1.5 (4)
C14—O2—C10—C9	−178.9 (2)	C18—C19—C20—C21	−0.4 (5)
C14—O2—C10—C11	2.8 (4)	C19—C20—C21—C16	0.3 (4)
O1—C9—C10—O2	0.4 (4)	C17—C16—C21—C20	1.6 (4)
C8—C9—C10—O2	−179.6 (2)	C15—C16—C21—C20	−176.4 (2)
O1—C9—C10—C11	178.8 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1A···N1^{i}	0.87 (3)	2.04 (3)	2.821 (3)	149 (3)
O3—H3···N2^{ii}	0.88 (3)	2.18 (3)	3.038 (3)	166 (2)
N2—H2···O4^{i}	0.91 (2)	2.55 (2)	3.052 (3)	115.5 (17)
N1—H1···O1	0.89 (2)	2.50 (2)	2.940 (3)	111.3 (18)
N2—H2···O3	0.91 (2)	2.50 (2)	3.065 (3)	120.1 (18)
N2—H2···N1	0.91 (2)	2.28 (2)	2.694 (3)	107.4 (17)
N1—H1···N2	0.89 (2)	2.40 (2)	2.694 (3)	99.6 (16)
O1—H1A···O2	0.87 (3)	2.29 (3)	2.659 (2)	106 (2)
O3—H3···O4	0.88 (3)	2.27 (3)	2.665 (3)	107 (2)
C7—H7B···O1	0.97	2.52	2.696 (3)	90

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

Fig. 1

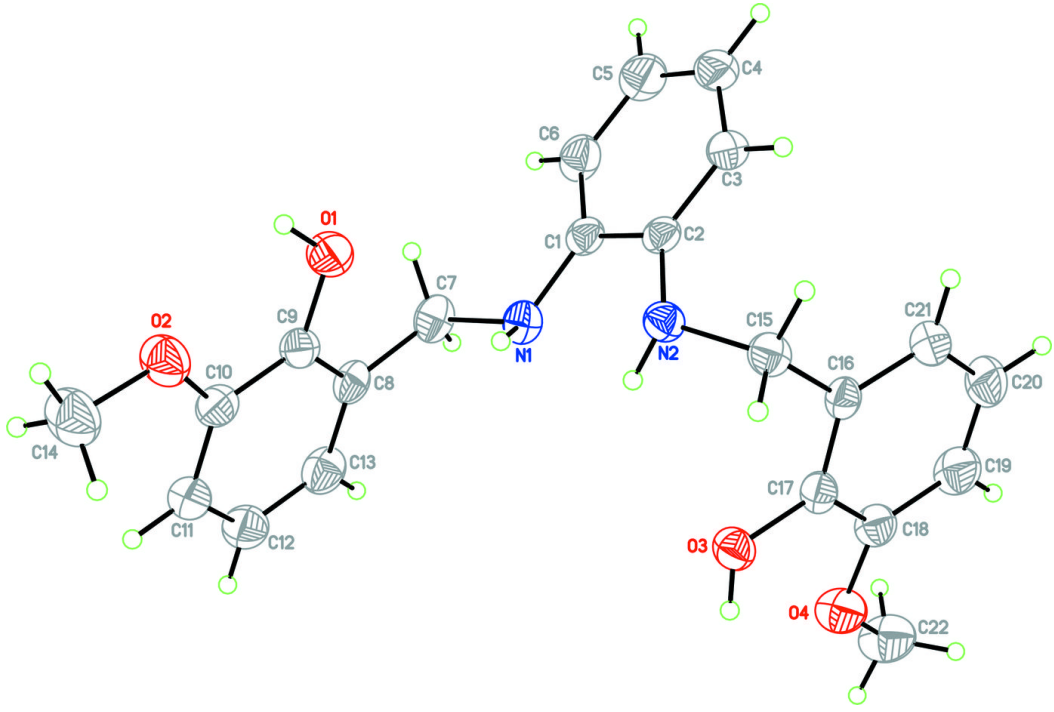
C5
C4
C6
C3
C1
C2
O1
C7
O2
C9
N1
N2
C15
C21
C10
C8
C16
C20
C14
C13
C19
C17
C11
C12
C18
O3
O4
C22

Fig. 2

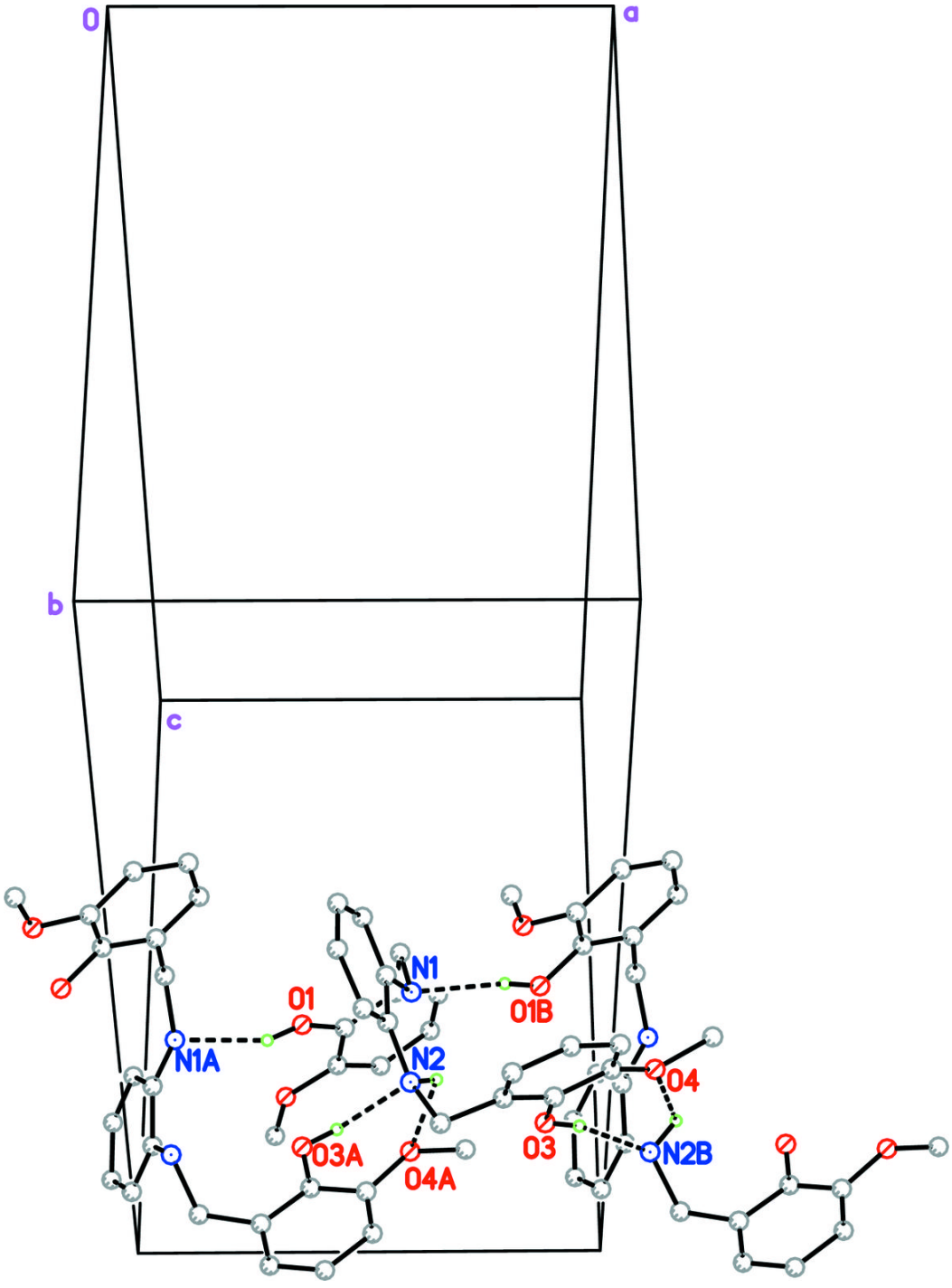

Fig. 3