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(6*E*,7*E*)-*N*,*N*′-Bis(2-hydroxy-3-methoxybenzylidene)benzene-1,4-diamine

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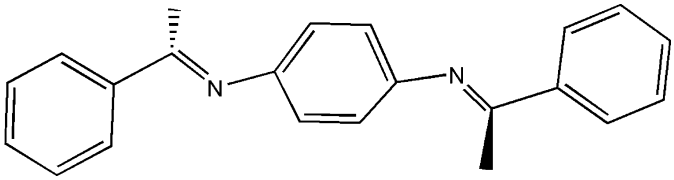
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.051; wR factor = 0.144; data-to-parameter ratio = 14.2.

The title compound, $C_{22}H_{20}N_2$, consists of two hypnone groups attached to a benzene ring *via* two azomethine linkages and crystallizes with an inversion centre at the mid-point of the central benzene ring. Molecules are linked into a chain by C—H···N hydrogen bonds along the *b*-axis direction. Neighbouring chains are linked by C—H···π hydrogen bonds into sheets.

Related literature

For related literature, see: Allen *et al.* (1987); Bernstein *et al.* (1995); Hodnett & Dunn (1970); Liu *et al.* (2006); Modi *et al.* (1970); Xia *et al.* (2007).



Experimental

Crystal data

$C_{22}H_{20}N_2$
M_r = 312.40
Monoclinic, $P2_1/c$
a = 5.9718 (11) Å
b = 6.8701 (15) Å
c = 21.713 (2) Å
β = 90.720 (2)°
V = 890.8 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 298 (2) K
0.45 × 0.42 × 0.19 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.970, T_{max} = 0.987
3502 measured reflections
1560 independent reflections
879 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.144
S = 1.02
1560 reflections
110 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

*Cg*1 is the centroid of ring C3–C8.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···N1	0.93	2.51	2.792 (3)	98
C11—H11···N1^{i}	0.93	2.80	3.715 (3)	166
C6—H6···*Cg*1^{ii}	0.93	2.86	3.707 (5)	152

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1996); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2308).

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supplementary materials

Acta Cryst. (2007). E**63**, o3560 [**doi:10.1107/S1600536807027754**]

(6*E*,7*E*)-*N*,*N*'-Bis(2-hydroxy-3-methoxybenzylidene)benzene-1,4-diamine

Y.-F. Liu, H.-T. Xia, S.-P. Yang and D.-Q. Wang

Comment

As part of our investigation of the reactions between hypnone with diamines, we report here the crystal structure of a hypnone Schiff base, *N*,*N*'-bis(3-methoxy-2-hydroxybenzylidene) benzene-1, 4-diamie, (I) and (Fig. 1).

The asymmetric unit consists of one half-molecule. The molecule have an inversion centre at the mid-point of the central benzen ring. The dihedral angle between the neighbouring benzen rings in the molecule is 74.10 (10) °. The bond lengths and angles are normal (Allen *et al.*, 1987) and are in agreement with those in two similar compounds (Xia *et al.*., 2007; Liu *et al.*, 2006). The molecules are linked into a chain of ring by C—H···N hydrogen bonds. Atom C11 in the molecule (*x*, *y*, *z*) act as hydrogen-bond donor to atom N1 in the molecule (− *x*, − *y*, 1 − *z*), generating a chain of $R_2^2(8)$ rings (Bernstein *et al.*, 1995) along the *b*-axisdirection (Fig. 2 and Table 1). Neighbouring chains are linked by C—H···π hydrogen bonds into the sheets (Fig. 3) and there are no direction-specific interactions between adjacent sheets.

Experimental

Solutions of hypnone (20 mmol) and benzene-1,2-diamie (10 mmol) in benzene (30 ml) was stirred for 6 h and then mixtures was filtered, and then solution was left to produce crystals of (I) slowly.

Refinement

All H atoms were located in difference Fourier maps. H atoms were treated as riding atoms, with C—H distances of 0.93 Å (aryl), 0.96 Å (methyl), and U_{iso}(H) = 1.2U_{eq}(C) for aryl or 1.5 U_{eq}(C) for methyl.

Figures

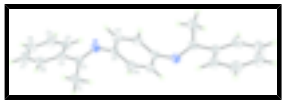

Fig. 1. The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are at the 30% probability level. Unlabelled atoms in the molecular are related to labelled atoma by (−*x*, 1 − *y*, 1 − *z*).

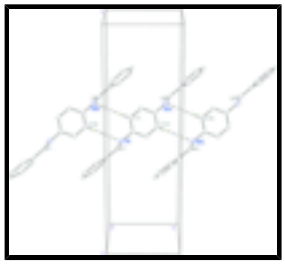

Fig. 2. A larger portion of the crystal struture of (I), the formation of a hydrogen-bonded chain built from C—H···N. For clarity, H atoms not involved in the hydrogen bonding have been omitted. Dashed lines indicate hydrogen bonds. [Symmetry codes: (A) −*x*, 1 − *y*, 1 − *z*; (B) − *x*, − *y*, 1 − *z*;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

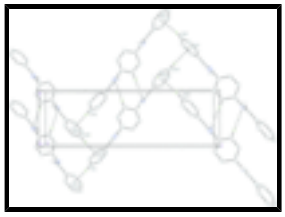

Fig. 3. A larger portion of the crystal struture of (I), the formation of a hydrogen-bonded sheet built from C—H···π. For clarity, H atoms not involved in the hydrogen bonding have been omitted. Dashed lines indicate hydrogen bonds. [Symmetry codes: (A) −*x*, 1 − *y*, 1 − *z*; (B) − *x*, − *y*, 1 − *z*;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

(6E,7E)-*N*,*N*'-Bis(2-hydroxy-3-methoxybenzylidene)benzene-1,4-diamine

Crystal data

$C_{22}H_{20}N_2$	F_{000} = 332
M_r = 312.40	D_x = 1.165 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 770 reflections
a = 5.9718 (11) Å	θ = 3.1–23.0º
b = 6.8701 (15) Å	μ = 0.07 mm^{-1}
c = 21.713 (2) Å	*T* = 298 (2) K
β = 90.720 (2)º	Block, colourless
V = 890.8 (3) Å^3	0.45 × 0.42 × 0.19 mm
Z = 2	

Data collection

Siemens SMART 1000 CCD area-detector diffractometer	1560 independent reflections
Radiation source: fine-focus sealed tube	879 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.046
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −7→6
T_{min} = 0.970, T_{max} = 0.987	*k* = −7→8
3502 measured reflections	*l* = −25→19

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.051	$w = 1/[\sigma^2(F_o^2) + (0.0578P)^2 + 0.0928P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.144	$(\Delta/\sigma)_{max} < 0.001$
S = 1.02	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
1560 reflections	$\Delta\rho_{min}$ = −0.15 e Å^{-3}

110 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.035 (5)
Secondary atom site location: difference Fourier map	

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	0.1054 (3)	0.1888 (3)	0.42130 (8)	0.0520 (6)
C1	0.5090 (4)	0.2304 (5)	0.43650 (14)	0.0942 (11)
H1A	0.4661	0.3410	0.4606	0.141*
H1B	0.6053	0.2719	0.4039	0.141*
H1C	0.5871	0.1386	0.4623	0.141*
C2	0.3038 (4)	0.1361 (3)	0.40941 (10)	0.0466 (6)
C3	0.3318 (4)	−0.0279 (3)	0.36540 (10)	0.0467 (6)
C4	0.1630 (4)	−0.0695 (4)	0.32348 (11)	0.0582 (7)
H4	0.0326	0.0045	0.3234	0.070*
C5	0.1853 (5)	−0.2190 (5)	0.28178 (12)	0.0774 (9)
H5	0.0716	−0.2433	0.2531	0.093*
C6	0.3754 (6)	−0.3328 (5)	0.28231 (14)	0.0844 (10)
H6	0.3896	−0.4346	0.2544	0.101*
C7	0.5421 (6)	−0.2951 (5)	0.32399 (15)	0.0838 (10)
H7	0.6700	−0.3723	0.3249	0.101*
C8	0.5219 (4)	−0.1423 (4)	0.36498 (12)	0.0671 (8)
H8	0.6380	−0.1161	0.3927	0.080*
C9	0.0613 (3)	0.3471 (3)	0.46147 (10)	0.0448 (6)
C10	0.0549 (4)	0.5353 (4)	0.43993 (11)	0.0532 (7)
H10	0.0903	0.5604	0.3991	0.064*
C11	0.0035 (4)	0.3121 (4)	0.52201 (10)	0.0523 (7)
H11	0.0039	0.1855	0.5371	0.063*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0428 (12)	0.0496 (14)	0.0636 (13)	−0.0032 (10)	0.0030 (10)	−0.0110 (10)
C1	0.0482 (17)	0.105 (3)	0.129 (3)	−0.0022 (18)	−0.0095 (17)	−0.047 (2)

C2	0.0404 (14)	0.0492 (16)	0.0502 (13)	−0.0039 (12)	0.0011 (10)	−0.0013 (11)
C3	0.0453 (15)	0.0469 (16)	0.0481 (13)	0.0004 (12)	0.0090 (11)	0.0030 (11)
C4	0.0625 (17)	0.0571 (18)	0.0550 (15)	−0.0008 (14)	0.0014 (12)	−0.0043 (13)
C5	0.092 (2)	0.076 (2)	0.0647 (18)	−0.0074 (19)	0.0038 (15)	−0.0183 (16)
C6	0.105 (3)	0.074 (2)	0.075 (2)	−0.001 (2)	0.0305 (19)	−0.0197 (17)
C7	0.084 (2)	0.075 (2)	0.093 (2)	0.0217 (19)	0.0296 (19)	−0.0062 (19)
C8	0.0564 (17)	0.073 (2)	0.0722 (18)	0.0092 (16)	0.0067 (13)	−0.0050 (15)
C9	0.0363 (13)	0.0415 (15)	0.0566 (14)	−0.0056 (11)	−0.0001 (10)	−0.0094 (12)
C10	0.0581 (16)	0.0508 (17)	0.0510 (14)	−0.0081 (13)	0.0068 (12)	−0.0020 (13)
C11	0.0541 (15)	0.0444 (15)	0.0586 (16)	−0.0040 (13)	0.0012 (11)	0.0009 (12)

Geometric parameters (Å, °)

N1—C2	1.268 (3)	C5—H5	0.9300
N1—C9	1.421 (3)	C6—C7	1.362 (4)
C1—C2	1.500 (3)	C6—H6	0.9300
C1—H1A	0.9600	C7—C8	1.382 (4)
C1—H1B	0.9600	C7—H7	0.9300
C1—H1C	0.9600	C8—H8	0.9300
C2—C3	1.489 (3)	C9—C10	1.376 (3)
C3—C4	1.379 (3)	C9—C11	1.384 (3)
C3—C8	1.381 (3)	C10—C11^{i}	1.382 (3)
C4—C5	1.377 (3)	C10—H10	0.9300
C4—H4	0.9300	C11—C10^{i}	1.382 (3)
C5—C6	1.378 (4)	C11—H11	0.9300
C2—N1—C9	121.6 (2)	C7—C6—C5	119.5 (3)
C2—C1—H1A	109.5	C7—C6—H6	120.2
C2—C1—H1B	109.5	C5—C6—H6	120.2
H1A—C1—H1B	109.5	C6—C7—C8	120.2 (3)
C2—C1—H1C	109.5	C6—C7—H7	119.9
H1A—C1—H1C	109.5	C8—C7—H7	119.9
H1B—C1—H1C	109.5	C3—C8—C7	121.0 (3)
N1—C2—C3	117.3 (2)	C3—C8—H8	119.5
N1—C2—C1	123.9 (2)	C7—C8—H8	119.5
C3—C2—C1	118.7 (2)	C10—C9—C11	118.7 (2)
C4—C3—C8	118.2 (2)	C10—C9—N1	121.0 (2)
C4—C3—C2	119.6 (2)	C11—C9—N1	120.0 (2)
C8—C3—C2	122.3 (2)	C9—C10—C11^{i}	121.0 (2)
C5—C4—C3	120.8 (3)	C9—C10—H10	119.5
C5—C4—H4	119.6	C11^{i}—C10—H10	119.5
C3—C4—H4	119.6	C10^{i}—C11—C9	120.3 (2)
C4—C5—C6	120.3 (3)	C10^{i}—C11—H11	119.9
C4—C5—H5	119.9	C9—C11—H11	119.9
C6—C5—H5	119.9		
C9—N1—C2—C3	−178.4 (2)	C5—C6—C7—C8	−0.7 (5)
C9—N1—C2—C1	1.0 (4)	C4—C3—C8—C7	−0.4 (4)
N1—C2—C3—C4	23.3 (3)	C2—C3—C8—C7	179.3 (2)

C1—C2—C3—C4	−156.2 (2)	C6—C7—C8—C3	1.3 (4)
N1—C2—C3—C8	−156.4 (2)	C2—N1—C9—C10	86.1 (3)
C1—C2—C3—C8	24.1 (3)	C2—N1—C9—C11	−99.9 (3)
C8—C3—C4—C5	−1.0 (4)	C11—C9—C10—C11^{i}	1.3 (4)
C2—C3—C4—C5	179.3 (2)	N1—C9—C10—C11^{i}	175.4 (2)
C3—C4—C5—C6	1.5 (4)	C10—C9—C11—C10^{i}	−1.3 (4)
C4—C5—C6—C7	−0.7 (4)	N1—C9—C11—C10^{i}	−175.4 (2)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···N1	0.93	2.51	2.792 (3)	98
C11—H11···N1^{ii}	0.93	2.80	3.715 (3)	166

Symmetry codes: (ii) −*x*, −*y*, −*z*+1.

Fig. 1

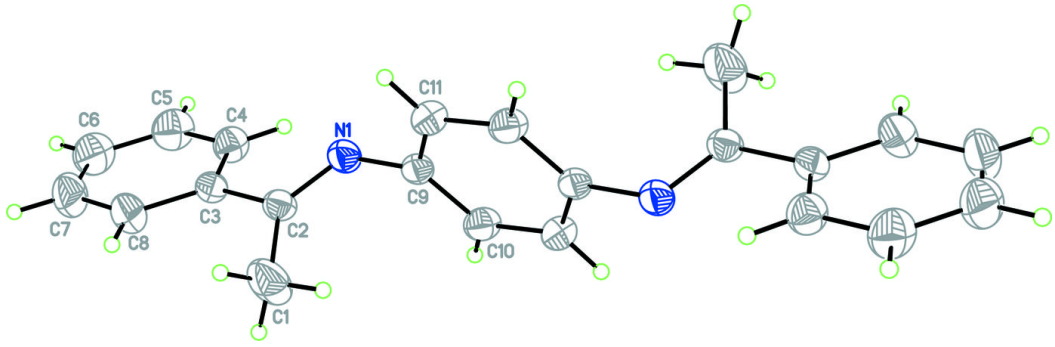

Fig. 2

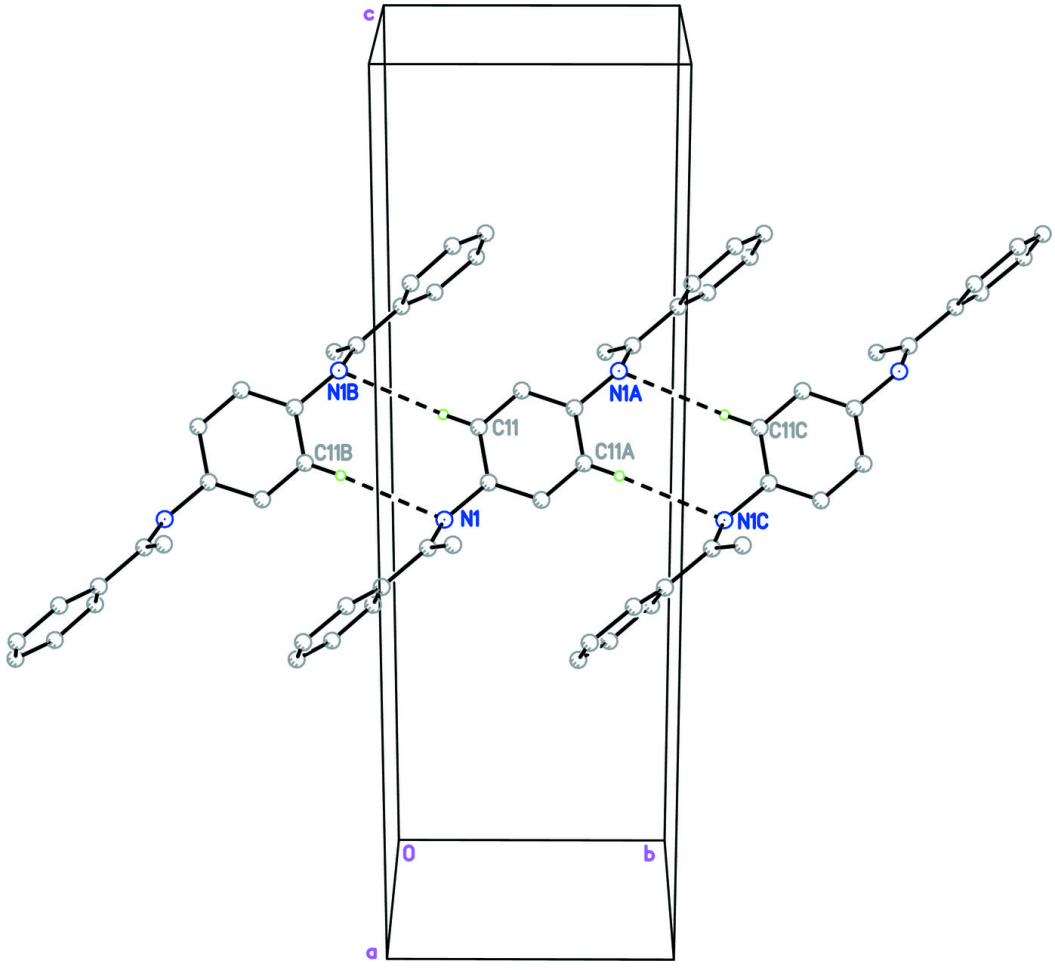

Fig. 3