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2,2′-[Ethylenebis(azanediylmethylene)]-diphenol

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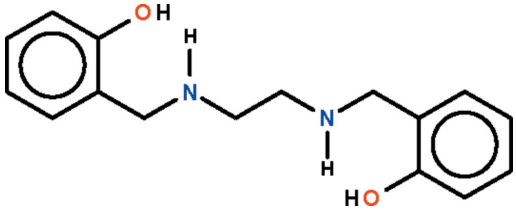
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.052; wR factor = 0.176; data-to-parameter ratio = 16.5.

In the title compound, $C_{16}H_{20}N_2O_4$, the molecule features a zigzag –CH_2–NH–CH_2–CH_2–NH–CH_2– chain whose ends are connected to the hydroxyphenyl rings. The molecules lies about a center of inversion. The imino group is a hydrogen-bond donor for the hydroxy group, which is a hydrogen-bond donor for the imino group of an adjacent molecule. This latter intermolecular hydrogen bonding leads to a layer structure.

Related literature

The title compound was doubly-deprotonated, forming several tetradentate chelated metal complexes. For their crystal structures, see: Atwood *et al.* (1995, 1996); Borer *et al.* (1983); Bottcher *et al.* (1994); García-Zarracino *et al.* (2002); Henrick *et al.* (1984); Viswanathan *et al.* (1998); Xie *et al.* (2006); Yang *et al.* (2007).



Experimental

Crystal data

$C_{16}H_{20}N_2O_2$
M_r = 272.34
Monoclinic, $P2_1/c$
a = 15.263 (2) Å
b = 4.860 (1) Å
c = 9.770 (1) Å
β = 96.318 (3)°
V = 720.3 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 K
0.31 × 0.27 × 0.25 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.975, T_{max} = 0.979
6726 measured reflections
1635 independent reflections
912 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.176
S = 1.09
1635 reflections
99 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1o···N1^{i}	0.86 (1)	1.89 (1)	2.721 (2)	165 (3)
N1—H1n···O1	0.86 (1)	2.23 (2)	2.884 (2)	133 (2)

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalClear* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2679).

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supporting information

Acta Cryst. (2009). E**65**, o3151 [doi:10.1107/S1600536809048831]

2,2′-[Ethylenebis(azanediylmethylene)]diphenol

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S1. Experimental

To a solution of salicylaldehyde (2.44 g, 20 mmol) in methanol was added a solution of ethylenediamine (0.6 ml, 10 mmol) in methanol. The solution was heated for two hours. The yellow Schiff base that was isolated upon evaporation of the solvent was reduced in absolute methanol by sodium borohydride. Colorless prismatic crystals were grown from a solution of the diamine in methanol.

S2. Refinement

Carbon-bound H-atoms generated geometrically (0.93–0.97 Å, U_{iso}(H) = 1.2U_{eq}(C)). The nitrogen- and oxygen-bound H-atoms were refined with a distance restraint of N–H = O–H = 0.85±0.01 Å; their temperature factors were refined.

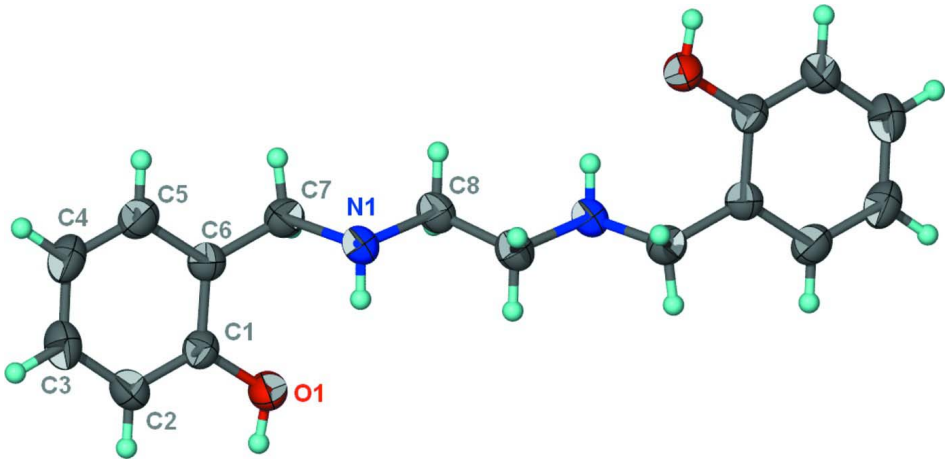

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of the molecule of $C_{16}H_{20}N_2O_2$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

2,2′-[Ethylenebis(azanediylmethylene)]diphenol

Crystal data

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M_r = 272.34
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 15.263 (2) Å
b = 4.860 (1) Å
c = 9.770 (1) Å
β = 96.318 (3)°
V = 720.3 (2) Å^3
Z = 2
F(000) = 292
D_x = 1.256 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3415 reflections
θ = 4.0–27.4°
μ = 0.08 mm^{-1}

T = 293 K
Prism, colorless

0.31 × 0.27 × 0.25 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.975, T_{max} = 0.979

6726 measured reflections
1635 independent reflections
912 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 27.4°, θ_{min} = 4.0°
h = −19→19
k = −6→6
l = −12→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.176
S = 1.09
1635 reflections
99 parameters
2 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0749P)^2 + 0.1302P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.34270 (10)	0.3118 (4)	0.13029 (16)	0.0555 (5)
N1	0.38397 (11)	0.4899 (4)	0.41165 (18)	0.0451 (5)
C1	0.25836 (14)	0.3821 (5)	0.1507 (2)	0.0439 (6)
C2	0.18572 (15)	0.2708 (5)	0.0744 (2)	0.0552 (6)
H2A	0.1932	0.1404	0.0070	0.066*
C3	0.10190 (15)	0.3510 (6)	0.0972 (3)	0.0622 (7)
H3	0.0532	0.2732	0.0460	0.075*
C4	0.09036 (16)	0.5466 (6)	0.1958 (3)	0.0624 (7)
H4	0.0340	0.6040	0.2103	0.075*
C5	0.16325 (16)	0.6564 (5)	0.2727 (2)	0.0564 (7)
H5	0.1552	0.7871	0.3397	0.068*
C6	0.24808 (13)	0.5773 (5)	0.2530 (2)	0.0443 (6)
C7	0.32748 (15)	0.6983 (5)	0.3364 (2)	0.0524 (6)
H7A	0.3621	0.7987	0.2754	0.063*
H7B	0.3079	0.8285	0.4019	0.063*
C8	0.46843 (13)	0.6100 (5)	0.4701 (2)	0.0497 (6)
H8A	0.4579	0.7403	0.5418	0.060*
H8B	0.4947	0.7091	0.3988	0.060*
H1O	0.3458 (18)	0.209 (5)	0.0600 (19)	0.081 (10)*
H1N	0.3943 (15)	0.370 (4)	0.3504 (19)	0.060 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0458 (10)	0.0730 (13)	0.0477 (10)	0.0062 (8)	0.0060 (7)	−0.0106 (8)
N1	0.0441 (10)	0.0469 (12)	0.0432 (10)	−0.0043 (8)	0.0005 (8)	−0.0013 (9)
C1	0.0434 (12)	0.0475 (13)	0.0409 (11)	0.0033 (10)	0.0055 (9)	0.0053 (10)
C2	0.0546 (14)	0.0616 (16)	0.0482 (13)	−0.0008 (11)	−0.0005 (11)	−0.0064 (12)
C3	0.0457 (14)	0.0746 (19)	0.0642 (16)	−0.0037 (12)	−0.0036 (12)	0.0023 (14)
C4	0.0465 (13)	0.0746 (19)	0.0660 (16)	0.0126 (13)	0.0065 (12)	0.0082 (14)
C5	0.0565 (14)	0.0592 (16)	0.0536 (14)	0.0127 (12)	0.0074 (11)	0.0000 (11)
C6	0.0489 (13)	0.0424 (13)	0.0418 (11)	0.0012 (9)	0.0050 (10)	0.0039 (9)
C7	0.0570 (14)	0.0458 (14)	0.0532 (13)	0.0055 (11)	0.0014 (11)	0.0000 (11)
C8	0.0480 (13)	0.0493 (15)	0.0510 (13)	−0.0079 (10)	0.0009 (10)	−0.0009 (11)

Geometric parameters (Å, º)

O1—C1	1.367 (2)	C4—C5	1.380 (4)
O1—H1O	0.86 (1)	C4—H4	0.9300
N1—C8	1.472 (3)	C5—C6	1.384 (3)
N1—C7	1.473 (3)	C5—H5	0.9300
N1—H1N	0.86 (1)	C6—C7	1.504 (3)
C1—C2	1.377 (3)	C7—H7A	0.9700
C1—C6	1.399 (3)	C7—H7B	0.9700
C2—C3	1.379 (3)	C8—C8^{i}	1.513 (4)
C2—H2A	0.9300	C8—H8A	0.9700
C3—C4	1.379 (4)	C8—H8B	0.9700
C3—H3	0.9300		
C1—O1—H1O	113.5 (19)	C4—C5—H5	119.1
C8—N1—C7	111.12 (17)	C6—C5—H5	119.1
C8—N1—H1N	108.6 (16)	C5—C6—C1	117.9 (2)
C7—N1—H1N	105.1 (16)	C5—C6—C7	121.7 (2)
O1—C1—C2	122.6 (2)	C1—C6—C7	120.34 (19)
O1—C1—C6	117.04 (19)	N1—C7—C6	113.21 (18)
C2—C1—C6	120.4 (2)	N1—C7—H7A	108.9
C1—C2—C3	120.5 (2)	C6—C7—H7A	108.9
C1—C2—H2A	119.7	N1—C7—H7B	108.9
C3—C2—H2A	119.7	C6—C7—H7B	108.9
C2—C3—C4	120.0 (2)	H7A—C7—H7B	107.8
C2—C3—H3	120.0	N1—C8—C8^{i}	111.3 (2)
C4—C3—H3	120.0	N1—C8—H8A	109.4
C3—C4—C5	119.4 (2)	C8^{i}—C8—H8A	109.4
C3—C4—H4	120.3	N1—C8—H8B	109.4
C5—C4—H4	120.3	C8^{i}—C8—H8B	109.4
C4—C5—C6	121.8 (2)	H8A—C8—H8B	108.0
O1—C1—C2—C3	−179.0 (2)	C2—C1—C6—C5	−1.0 (3)
C6—C1—C2—C3	0.4 (4)	O1—C1—C6—C7	−0.7 (3)

C1—C2—C3—C4	0.8 (4)	C2—C1—C6—C7	179.9 (2)
C2—C3—C4—C5	−1.2 (4)	C8—N1—C7—C6	169.35 (18)
C3—C4—C5—C6	0.6 (4)	C5—C6—C7—N1	122.4 (2)
C4—C5—C6—C1	0.6 (4)	C1—C6—C7—N1	−58.5 (3)
C4—C5—C6—C7	179.7 (2)	C7—N1—C8—C8^{i}	−171.9 (2)
O1—C1—C6—C5	178.39 (19)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1o···N1^{ii}	0.86 (1)	1.89 (1)	2.721 (2)	165 (3)
N1—H1n···O1	0.86 (1)	2.23 (2)	2.884 (2)	133 (2)

Symmetry code: (ii) *x*, −*y*+1/2, *z*−1/2.