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2,2′-(1,1′-Azinodiethylidyne)diphenol

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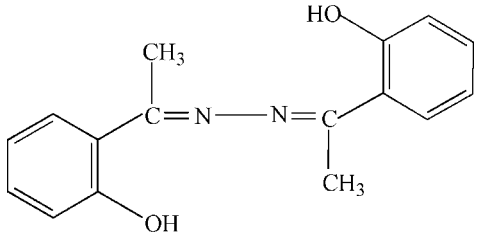
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.043; wR factor = 0.128; data-to-parameter ratio = 7.9.

In the title molecule, $C_{16}H_{16}N_2O_2$, the C—N bond lengths are 1.295 (5) and 1.300 (5) Å, which suggests that they are double bonds. The structure is stabilized by intramolecular O—H···N and C—H···N, and intermolecular C—H···O hydrogen-bond interactions.

Related literature

For related literature, see: Tai *et al.* (2003).



Experimental

Crystal data

$C_{16}H_{16}N_2O_2$
M_r = 268.31
Orthorhombic, $P2_12_12_1$
a = 6.3358 (8) Å
b = 13.5625 (10) Å
c = 15.9956 (15) Å
V = 1374.5 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.38 × 0.15 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.968, T_{max} = 0.988
7170 measured reflections
1422 independent reflections
849 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.128
S = 1.08
1422 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 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
O1—H1···N1	0.82	1.80	2.529 (5)	146
O2—H2···N2	0.82	1.80	2.529 (4)	147
C1—H1A···N2	0.96	2.32	2.739 (5)	106
C5—H5···O2^{i}	0.93	2.59	3.403 (6)	147
C9—H9A···N1	0.96	2.30	2.724 (6)	106

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: AT2562).

References

Bruker (2000). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tai, X.-S., Yin, X.-H., Tan, M.-Y. & Li, Y.-Z. (2003). *Acta Cryst.* E**59**, o681–o682.

supporting information

Acta Cryst. (2008). E**64**, o905 [doi:10.1107/S1600536808011318]

2,2′-(1,1′-Azinodiethylidyne)diphenol

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S1. Comment

As part of our ongoing studies of the coordination chemistry of Schiffbase ligands (Tai *et al.*, 2003), we now report the synthesis and structure of the title compound, (I), (Fig. 1).

In the molecule of (I), both C2—N1 [1.295 (5) Å], and C10—N2 [1.300 (5) Å] are close to double-bond separations, indicating that the Lewis structure shown in the scheme is only an approximation to the electron distribution in the molecule. Otherwise, the geometrical parameters for (I) are normal. The structure is stabilized by intramolecular O—H···N and C—H···N, and intermolecular C—H···O hydrogen bonding interactions.

S2. Experimental

2 mmol of 2′-Hhydroxyacetophenone (2 mmol) was added to a solution of hydrazide (1 mmol) in 10 ml of 95% ethanol. The mixture was continuously stirred for 3 h at refluxing temperature, evaporating some ethanol, then, upon cooling, the solid product was collected by filtration and dried *in vacuo* (yield 58%). Clear blocks of (I) were obtained by evaporation from a methanol solution after 6 days.

S3. Refinement

The H atoms were placed geometrically (C—H = 0.93–0.96 Å, O—H = 0.82 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(aromatic C) or 1.5U_{eq}(methyl C, hydroxyl O).

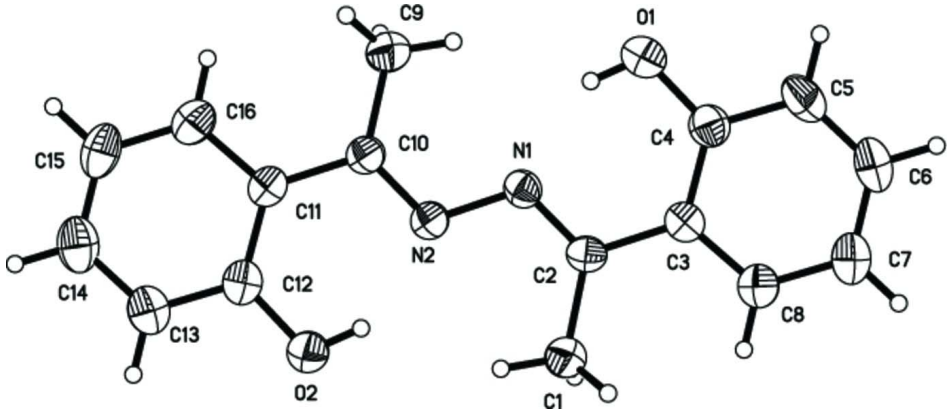

Figure 1

The molecular structure of (I) showing 30% displacement ellipsoids.

2,2′-(1,1′-Azinodiethylidyne)diphenol

Crystal data

$C_{16}H_{16}N_2O_2$
$M_r = 268.31$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 6.3358$ (8) Å
$b = 13.5625$ (10) Å
$c = 15.9956$ (15) Å
$V = 1374.5$ (2) Å^3
$Z = 4$
$F(000) = 568$
$D_x = 1.297$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1489 reflections
$\theta = 2.9$–$20.4°$
$\mu = 0.09$ mm^{-1}
$T = 298$ K
Block, colourless
0.38 × 0.15 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.968$, $T_{max} = 0.988$
7170 measured reflections
1422 independent reflections
849 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.0°$
$h = -7 \rightarrow 7$
$k = -16 \rightarrow 13$
$l = -17 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.128$
$S = 1.08$
1422 reflections
181 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.4138P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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	0.5862 (6)	0.4828 (2)	0.5963 (2)	0.0489 (9)
N2	0.4075 (6)	0.4685 (2)	0.6456 (2)	0.0486 (9)
O1	0.8168 (6)	0.4367 (2)	0.47328 (19)	0.0824 (11)
H1	0.7174	0.4320	0.5059	0.124*
O2	0.1570 (5)	0.5228 (2)	0.75976 (17)	0.0670 (9)

H2	0.2602	0.5259	0.7290	0.101*
C1	0.6514 (8)	0.6288 (3)	0.6846 (3)	0.0719 (14)
H1A	0.5090	0.6183	0.7031	0.108*
H1B	0.6665	0.6953	0.6649	0.108*
H1C	0.7468	0.6177	0.7302	0.108*
C2	0.7010 (7)	0.5586 (3)	0.6150 (2)	0.0472 (10)
C3	0.8877 (7)	0.5753 (3)	0.5630 (2)	0.0487 (11)
C4	0.9357 (8)	0.5152 (3)	0.4941 (3)	0.0605 (13)
C5	1.1111 (9)	0.5351 (4)	0.4453 (3)	0.0779 (16)
H5	1.1417	0.4951	0.3996	0.093*
C6	1.2395 (9)	0.6130 (4)	0.4636 (3)	0.0778 (15)
H6	1.3570	0.6254	0.4304	0.093*
C7	1.1973 (8)	0.6727 (4)	0.5301 (3)	0.0698 (14)
H7	1.2848	0.7259	0.5422	0.084*
C8	1.0243 (7)	0.6535 (3)	0.5790 (3)	0.0589 (12)
H8	0.9973	0.6942	0.6246	0.071*
C9	0.3515 (10)	0.3201 (3)	0.5599 (3)	0.0861 (17)
H9A	0.4546	0.3497	0.5237	0.129*
H9B	0.2270	0.3046	0.5283	0.129*
H9C	0.4084	0.2608	0.5836	0.129*
C10	0.2963 (7)	0.3905 (3)	0.6284 (2)	0.0502 (11)
C11	0.1087 (7)	0.3741 (3)	0.6796 (3)	0.0500 (11)
C12	0.0460 (7)	0.4402 (3)	0.7423 (3)	0.0542 (11)
C13	−0.1329 (8)	0.4228 (4)	0.7893 (3)	0.0651 (13)
H13	−0.1705	0.4669	0.8312	0.078*
C14	−0.2557 (9)	0.3416 (4)	0.7750 (3)	0.0766 (15)
H14	−0.3769	0.3310	0.8065	0.092*
C15	−0.1990 (9)	0.2764 (3)	0.7141 (3)	0.0762 (15)
H15	−0.2824	0.2212	0.7041	0.091*
C16	−0.0201 (8)	0.2914 (3)	0.6674 (3)	0.0668 (13)
H16	0.0165	0.2456	0.6267	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.044 (2)	0.052 (2)	0.051 (2)	0.0052 (18)	0.0000 (18)	−0.0005 (16)
N2	0.046 (2)	0.047 (2)	0.053 (2)	0.0031 (18)	−0.0032 (18)	0.0000 (16)
O1	0.093 (3)	0.084 (2)	0.070 (2)	−0.013 (2)	0.021 (2)	−0.0175 (17)
O2	0.070 (2)	0.0691 (19)	0.0615 (19)	−0.0107 (19)	0.0074 (18)	−0.0083 (15)
C1	0.062 (3)	0.067 (3)	0.087 (3)	−0.006 (3)	0.012 (3)	−0.017 (3)
C2	0.049 (3)	0.044 (2)	0.049 (2)	0.010 (2)	−0.004 (2)	−0.0004 (18)
C3	0.048 (3)	0.052 (2)	0.047 (2)	0.009 (2)	−0.005 (2)	0.006 (2)
C4	0.068 (4)	0.062 (3)	0.052 (3)	−0.001 (3)	0.001 (3)	0.005 (2)
C5	0.087 (4)	0.092 (4)	0.054 (3)	0.005 (4)	0.020 (3)	0.004 (3)
C6	0.063 (3)	0.094 (4)	0.076 (4)	0.000 (3)	0.019 (3)	0.019 (3)
C7	0.056 (3)	0.076 (3)	0.076 (3)	−0.003 (3)	0.003 (3)	0.017 (3)
C8	0.053 (3)	0.063 (3)	0.061 (3)	0.003 (3)	−0.004 (3)	0.005 (2)
C9	0.079 (4)	0.072 (3)	0.107 (4)	−0.013 (3)	0.020 (3)	−0.035 (3)

C10	0.045 (3)	0.047 (2)	0.058 (3)	0.004 (2)	−0.004 (2)	−0.002 (2)
C11	0.049 (3)	0.045 (2)	0.056 (3)	0.001 (2)	−0.009 (2)	0.006 (2)
C12	0.055 (3)	0.054 (3)	0.054 (3)	0.001 (2)	−0.006 (2)	0.015 (2)
C13	0.063 (3)	0.070 (3)	0.063 (3)	0.005 (3)	0.002 (3)	0.015 (3)
C14	0.068 (3)	0.081 (3)	0.081 (4)	−0.002 (3)	0.009 (3)	0.031 (3)
C15	0.067 (4)	0.068 (3)	0.094 (4)	−0.019 (3)	−0.004 (3)	0.023 (3)
C16	0.068 (3)	0.057 (3)	0.075 (3)	−0.005 (3)	−0.009 (3)	0.003 (2)

Geometric parameters (Å, º)

N1—C2	1.295 (5)	C7—C8	1.371 (6)
N1—N2	1.394 (4)	C7—H7	0.9300
N2—C10	1.300 (5)	C8—H8	0.9300
O1—C4	1.346 (5)	C9—C10	1.495 (5)
O1—H1	0.8200	C9—H9A	0.9600
O2—C12	1.352 (5)	C9—H9B	0.9600
O2—H2	0.8200	C9—H9C	0.9600
C1—C2	1.497 (5)	C10—C11	1.460 (6)
C1—H1A	0.9600	C11—C16	1.401 (5)
C1—H1B	0.9600	C11—C12	1.403 (6)
C1—H1C	0.9600	C12—C13	1.380 (6)
C2—C3	1.464 (5)	C13—C14	1.368 (6)
C3—C8	1.392 (5)	C13—H13	0.9300
C3—C4	1.405 (6)	C14—C15	1.364 (6)
C4—C5	1.385 (7)	C14—H14	0.9300
C5—C6	1.365 (7)	C15—C16	1.372 (7)
C5—H5	0.9300	C15—H15	0.9300
C6—C7	1.364 (6)	C16—H16	0.9300
C6—H6	0.9300		
C2—N1—N2	115.8 (3)	C7—C8—H8	118.8
C10—N2—N1	115.7 (3)	C3—C8—H8	118.8
C4—O1—H1	109.5	C10—C9—H9A	109.5
C12—O2—H2	109.5	C10—C9—H9B	109.5
C2—C1—H1A	109.5	H9A—C9—H9B	109.5
C2—C1—H1B	109.5	C10—C9—H9C	109.5
H1A—C1—H1B	109.5	H9A—C9—H9C	109.5
C2—C1—H1C	109.5	H9B—C9—H9C	109.5
H1A—C1—H1C	109.5	N2—C10—C11	116.5 (4)
H1B—C1—H1C	109.5	N2—C10—C9	123.2 (4)
N1—C2—C3	116.5 (4)	C11—C10—C9	120.3 (4)
N1—C2—C1	124.0 (4)	C16—C11—C12	116.5 (4)
C3—C2—C1	119.6 (4)	C16—C11—C10	121.2 (4)
C8—C3—C4	116.8 (4)	C12—C11—C10	122.3 (4)
C8—C3—C2	121.1 (4)	O2—C12—C13	117.1 (4)
C4—C3—C2	122.1 (4)	O2—C12—C11	122.0 (4)
O1—C4—C5	117.6 (4)	C13—C12—C11	120.9 (4)
O1—C4—C3	122.1 (4)	C14—C13—C12	120.9 (5)

C5—C4—C3	120.2 (5)	C14—C13—H13	119.5
C6—C5—C4	120.5 (5)	C12—C13—H13	119.5
C6—C5—H5	119.7	C15—C14—C13	119.5 (5)
C4—C5—H5	119.7	C15—C14—H14	120.3
C7—C6—C5	120.7 (5)	C13—C14—H14	120.3
C7—C6—H6	119.7	C14—C15—C16	120.6 (5)
C5—C6—H6	119.7	C14—C15—H15	119.7
C6—C7—C8	119.3 (5)	C16—C15—H15	119.7
C6—C7—H7	120.4	C15—C16—C11	121.7 (5)
C8—C7—H7	120.4	C15—C16—H16	119.2
C7—C8—C3	122.5 (5)	C11—C16—H16	119.2
C2—N1—N2—C10	−177.9 (4)	N1—N2—C10—C11	179.9 (3)
N2—N1—C2—C3	−179.2 (3)	N1—N2—C10—C9	−0.6 (6)
N2—N1—C2—C1	−0.2 (5)	N2—C10—C11—C16	−178.7 (4)
N1—C2—C3—C8	−178.6 (4)	C9—C10—C11—C16	1.7 (6)
C1—C2—C3—C8	2.3 (5)	N2—C10—C11—C12	2.2 (5)
N1—C2—C3—C4	2.7 (5)	C9—C10—C11—C12	−177.4 (4)
C1—C2—C3—C4	−176.4 (4)	C16—C11—C12—O2	−179.8 (4)
C8—C3—C4—O1	179.0 (4)	C10—C11—C12—O2	−0.7 (6)
C2—C3—C4—O1	−2.3 (6)	C16—C11—C12—C13	0.5 (6)
C8—C3—C4—C5	−0.3 (6)	C10—C11—C12—C13	179.6 (4)
C2—C3—C4—C5	178.4 (4)	O2—C12—C13—C14	179.2 (4)
O1—C4—C5—C6	−179.2 (4)	C11—C12—C13—C14	−1.1 (6)
C3—C4—C5—C6	0.1 (7)	C12—C13—C14—C15	0.8 (7)
C4—C5—C6—C7	−0.1 (8)	C13—C14—C15—C16	0.2 (7)
C5—C6—C7—C8	0.3 (7)	C14—C15—C16—C11	−0.8 (7)
C6—C7—C8—C3	−0.6 (6)	C12—C11—C16—C15	0.5 (6)
C4—C3—C8—C7	0.6 (6)	C10—C11—C16—C15	−178.7 (4)
C2—C3—C8—C7	−178.2 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.80	2.529 (5)	146
O2—H2···N2	0.82	1.80	2.529 (4)	147
C1—H1*A*···N2	0.96	2.32	2.739 (5)	106
C5—H5···O2^{i}	0.93	2.59	3.403 (6)	147
C9—H9*A*···N1	0.96	2.30	2.724 (6)	106

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