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4-(Diethylamino)salicylaldehyde azine

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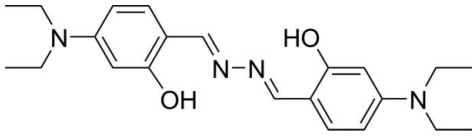
Key indicators: single-crystal X-ray study; T = 290 K; mean σ(C–C) = 0.004 Å; R factor = 0.074; wR factor = 0.228; data-to-parameter ratio = 18.8.

The title compound, $C_{22}H_{30}N_4O_2$, has a crystallographic inversion center located at the mid-point of the N—N single bond. Apart from the four ethyl C atoms, the non-H atoms are nearly coplanar with a mean deviation of 0.0596 (2) Å. An intramolecular O—H···N hydrogen bond occurs. In the crystal, weak intermolecular C—H···O hydrogen bonds link the molecules into layers parallel to (100).

Related literature

For the synthesis, see Tang *et al.* (2009). For a related structure, see Gil *et al.* (2010). For applications of photochromic aromatic Schiff base molecules as molecular memories and switches, see Sliwa *et al.* (2005).



Experimental

Crystal data

$C_{22}H_{30}N_4O_2$
M_r = 382.50
Monoclinic, $P2_1/c$
a = 8.736 (5) Å
b = 7.809 (5) Å
c = 16.122 (10) Å
β = 103.57 (2)°
V = 1069.1 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 290 K
0.15 × 0.14 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.988, T_{max} = 0.991
9903 measured reflections
2431 independent reflections
1227 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.228
S = 1.10
2431 reflections
129 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8B···O1^{i}	0.97	2.64	3.481 (5)	145
O1—H1···N1	0.85	1.88	2.640 (3)	149

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

Data collection: *RAPID-AUTO* (Rigaku Corporation, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC and Rigaku Corporation, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5147).

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supplementary materials

Acta Cryst. (2011). E**67**, o1092 [**doi:10.1107/S1600536811013237**]

4-(Diethylamino)salicylaldehyde azine

J.-B. Qiu and B.-Z. Yin

Comment

Salicylaldehyde azine belongs to the photochromic aromatic schiff base molecules with two intramolecular hydrogen bonds (Gil *et al.*, 2010). The photochromism of the molecules, owing to enol-keto intramolecular tautomerism, attracts much interest because of possible applications, for example, in molecular memories and switches (Sliwa *et al.*, 2005). Herein, we report the crystal structure of the title compound.

The title compound, as shown in Fig. 1, all bond lengths and angles are in the normal ranges. Except for four carbon atoms, all the other non-hydrogen atoms nearly lie on the same plane. The intramolecular O—H···N and intermolecular C—H···O hydrogen bonds (Table 1) link the molecules into layers prallel to (100).

Experimental

The title compound was prepared according to the literature (Tang *et al.*, 2009). Single crystals suitable for X-ray diffraction were prepared by slow evaporation a mixture of dichloromethane and petroleum (60–90 °C) at room temperature.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 to 0.97 Å) and were included in the refinement in the riding model with U_{iso}(H) = 1.2 or 1.5 U_{eq}(C). The hydroxy H atom was located in a difference Fourier map and treated as riding on its parent O atom with U_{iso}(H) = 1.5 U_{eq}(O). The distance of O1 and H1 was restricted to 0.85 Å with *DFIX* command.

Figures

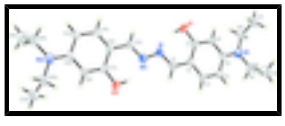


Fig. 1. The crystal structure of the title compound, with the atom numbering. Displacement ellipsoids of non-H atoms are drawn at the 30% probalility level. [Symmetry code: A: 1 - *x*, 1 - *y*, 1 - *z*]

4-(Diethylamino)-2-hydroxybenzaldehyde azine

Crystal data

$C_{22}H_{30}N_4O_2$	$F(000)$ = 412
M_r = 382.50	D_x = 1.188 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5162 reflections
a = 8.736 (5) Å	θ = 3.1–27.7°

b = 7.809 (5) Å	μ = 0.08 mm^{-1}
c = 16.122 (10) Å	T = 290 K
β = 103.57 (2)°	Block, yellow
V = 1069.1 (11) Å^3	0.15 × 0.14 × 0.12 mm
Z = 2	

Data collection

Rigaku R-AXIS RAPID diffractometer	2431 independent reflections
Radiation source: fine-focus sealed tube	1227 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.046
ω scans	θ_{max} = 27.5°, θ_{min} = 3.1°
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	h = −11→11
T_{min} = 0.988, T_{max} = 0.991	k = −10→10
9903 measured reflections	l = −20→20

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.074	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.228	H-atom parameters constrained
S = 1.10	$w = 1/[\sigma^2(F_o^2) + (0.0919P)^2 + 0.3133P]$ where $P = (F_o^2 + 2F_c^2)/3$
2431 reflections	$(\Delta/\sigma)_{max}$ = 0.003
129 parameters	$\Delta\rho_{max}$ = 0.45 e Å^{-3}
1 restraint	$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Special details

Experimental. (See detailed section in the paper)

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}

O1	0.2021 (2)	0.6394 (3)	0.57469 (13)	0.0809 (8)
H1	0.2663	0.6244	0.5433	0.121*
C1	0.4983 (3)	0.4599 (4)	0.60317 (18)	0.0556 (7)
H1A	0.5914	0.3972	0.6165	0.067*
C2	0.4129 (3)	0.4858 (3)	0.66828 (16)	0.0489 (7)
C3	0.4698 (3)	0.4235 (4)	0.75059 (18)	0.0602 (8)
H3	0.5638	0.3624	0.7625	0.072*
C4	0.3937 (3)	0.4480 (4)	0.81492 (18)	0.0653 (9)
H4	0.4360	0.4031	0.8688	0.078*
C5	0.2503 (3)	0.5418 (4)	0.79966 (18)	0.0575 (7)
C6	0.1898 (3)	0.5998 (4)	0.71712 (17)	0.0558 (7)
H6	0.0939	0.6573	0.7048	0.067*
C7	0.2678 (3)	0.5745 (4)	0.65257 (17)	0.0537 (7)
C8	0.2343 (5)	0.5030 (6)	0.9510 (2)	0.0878 (11)
H8A	0.3481	0.5128	0.9679	0.105*
H8B	0.1906	0.5672	0.9915	0.105*
C9	0.1893 (5)	0.3226 (6)	0.9530 (3)	0.1028 (14)
H9A	0.0770	0.3142	0.9441	0.154*
H9B	0.2387	0.2742	1.0074	0.154*
H9C	0.2226	0.2614	0.9086	0.154*
C10	0.0357 (4)	0.6857 (5)	0.8511 (2)	0.0735 (9)
H10A	0.0461	0.7776	0.8123	0.088*
H10B	0.0312	0.7373	0.9052	0.088*
C11	−0.1157 (4)	0.5939 (5)	0.8161 (2)	0.0858 (11)
H11A	−0.1144	0.5460	0.7615	0.129*
H11B	−0.2018	0.6729	0.8100	0.129*
H11C	−0.1283	0.5038	0.8545	0.129*
N1	0.4511 (3)	0.5198 (3)	0.52728 (15)	0.0589 (7)
N2	0.1766 (3)	0.5765 (4)	0.86429 (15)	0.0752 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0839 (14)	0.1066 (19)	0.0616 (13)	0.0420 (13)	0.0362 (11)	0.0333 (12)
C1	0.0554 (15)	0.0532 (17)	0.0651 (18)	0.0019 (13)	0.0280 (13)	0.0001 (13)
C2	0.0482 (14)	0.0504 (16)	0.0521 (15)	−0.0016 (12)	0.0197 (11)	0.0012 (12)
C3	0.0454 (14)	0.076 (2)	0.0619 (18)	0.0117 (13)	0.0182 (13)	0.0085 (15)
C4	0.0519 (15)	0.097 (2)	0.0484 (16)	0.0105 (16)	0.0142 (12)	0.0101 (15)
C5	0.0505 (14)	0.0707 (19)	0.0569 (17)	0.0039 (13)	0.0242 (13)	0.0042 (14)
C6	0.0525 (14)	0.0620 (18)	0.0581 (16)	0.0110 (13)	0.0235 (13)	0.0091 (14)
C7	0.0560 (15)	0.0550 (17)	0.0549 (16)	0.0075 (13)	0.0227 (13)	0.0111 (13)
C8	0.084 (2)	0.115 (3)	0.074 (2)	0.002 (2)	0.0365 (19)	−0.009 (2)
C9	0.101 (3)	0.114 (4)	0.103 (3)	0.019 (3)	0.043 (2)	0.009 (2)
C10	0.075 (2)	0.083 (2)	0.072 (2)	0.0104 (18)	0.0374 (17)	−0.0018 (17)
C11	0.079 (2)	0.091 (3)	0.094 (3)	0.009 (2)	0.0336 (19)	0.007 (2)
N1	0.0643 (14)	0.0606 (15)	0.0616 (15)	0.0027 (12)	0.0345 (11)	0.0032 (12)
N2	0.0708 (16)	0.108 (2)	0.0548 (15)	0.0222 (15)	0.0301 (12)	0.0097 (14)

Geometric parameters (Å, °)

O1—C7	1.351 (3)	C8—C9	1.465 (6)
O1—H1	0.8461	C8—N2	1.486 (4)
C1—N1	1.284 (4)	C8—H8A	0.9700
C1—C2	1.438 (4)	C8—H8B	0.9700
C1—H1A	0.9300	C9—H9A	0.9600
C2—C3	1.391 (4)	C9—H9B	0.9600
C2—C7	1.414 (4)	C9—H9C	0.9600
C3—C4	1.371 (4)	C10—N2	1.471 (4)
C3—H3	0.9300	C10—C11	1.494 (5)
C4—C5	1.422 (4)	C10—H10A	0.9700
C4—H4	0.9300	C10—H10B	0.9700
C5—N2	1.374 (3)	C11—H11A	0.9600
C5—C6	1.387 (4)	C11—H11B	0.9600
C6—C7	1.386 (3)	C11—H11C	0.9600
C6—H6	0.9300	N1—N1^{i}	1.397 (4)
C7—O1—H1	107.9	C9—C8—H8B	109.4
N1—C1—C2	122.6 (3)	N2—C8—H8B	109.4
N1—C1—H1A	118.7	H8A—C8—H8B	108.0
C2—C1—H1A	118.7	C8—C9—H9A	109.5
C3—C2—C7	116.6 (2)	C8—C9—H9B	109.5
C3—C2—C1	121.1 (2)	H9A—C9—H9B	109.5
C7—C2—C1	122.3 (2)	C8—C9—H9C	109.5
C4—C3—C2	123.0 (3)	H9A—C9—H9C	109.5
C4—C3—H3	118.5	H9B—C9—H9C	109.5
C2—C3—H3	118.5	N2—C10—C11	114.4 (3)
C3—C4—C5	120.3 (3)	N2—C10—H10A	108.7
C3—C4—H4	119.8	C11—C10—H10A	108.7
C5—C4—H4	119.8	N2—C10—H10B	108.7
N2—C5—C6	121.5 (2)	C11—C10—H10B	108.7
N2—C5—C4	121.4 (3)	H10A—C10—H10B	107.6
C6—C5—C4	117.1 (2)	C10—C11—H11A	109.5
C7—C6—C5	122.0 (2)	C10—C11—H11B	109.5
C7—C6—H6	119.0	H11A—C11—H11B	109.5
C5—C6—H6	119.0	C10—C11—H11C	109.5
O1—C7—C6	117.9 (2)	H11A—C11—H11C	109.5
O1—C7—C2	121.2 (2)	H11B—C11—H11C	109.5
C6—C7—C2	120.9 (2)	C1—N1—N1^{i}	114.3 (3)
C9—C8—N2	111.0 (3)	C5—N2—C10	122.0 (2)
C9—C8—H8A	109.4	C5—N2—C8	121.4 (3)
N2—C8—H8A	109.4	C10—N2—C8	116.6 (2)

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

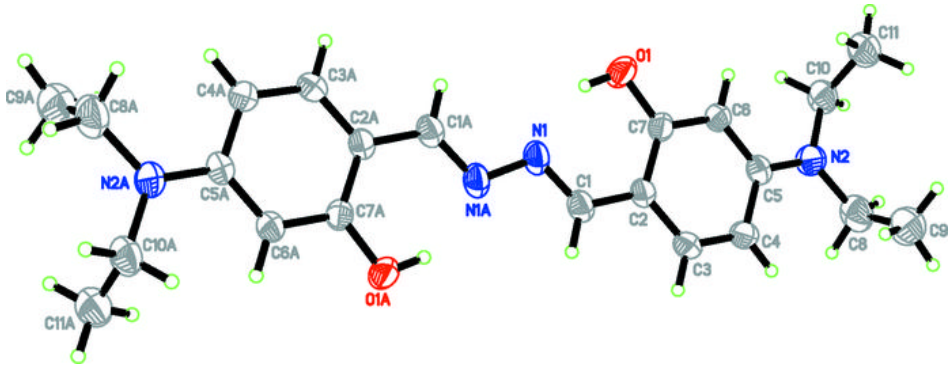
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

C8—H8B···O1^{ii}	0.97	2.64	3.481 (5)	145
O1—H1···N1	0.85	1.88	2.640 (3)	149

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

Fig. 1


C9A
C8A
N2A
C10A
C11A
C4A
C3A
C2A
C1A
C5A
C7A
C6A
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N1
N1A
C1
O1
C7
C6
C11
C10
N2
C5
C2
C4
C3
C8
C9