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2-Amino-3-[(*E*)-(2-hydroxy-3-methylbenzylidene)amino]but-2-enedinitrile

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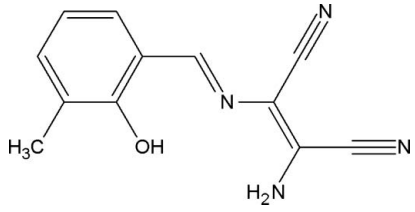
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.059; wR factor = 0.132; data-to-parameter ratio = 13.9.

The title compound, $C_{12}H_{10}N_4O$, is a Schiff base obtained from the condensation of diaminomaleonitrile and 2-hydroxy-3-methylbenzaldehyde. The molecule is roughly planar, with an r.m.s. deviation of 0.0354 Å, and adopts the phenol–imine tautomeric form. An intramolecular O—H···N hydrogen bond involving the O—H group and the azomethine N atom generates an S(6) ring. In the crystal, there are two N—H···N hydrogen bonds.

Related literature

For the biological properties of Schiff bases see: Da Silva *et al.* (2011) and for their use in coordination chemistry, see: Aazam *et al.* (2011); Kargar *et al.* (2009); Yeap *et al.* (2009). For graph-set notation, see: Bernstein *et al.*, (1995). For related structures, see: Aazam & Büyükgüngör (2010); Hökelek *et al.* (2000); Odabaşoğlu *et al.* (2005); Rivera *et al.* (2006).



Experimental

Crystal data

$C_{12}H_{10}N_4O$
M_r = 226.24
Monoclinic, $P2_1/c$
a = 6.9041 (6) Å
b = 11.8791 (7) Å
c = 14.0282 (11) Å
β = 101.600 (7)°
V = 1127.02 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 K
0.76 × 0.48 × 0.03 mm

Data collection

Stoe IPDS 2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.949, T_{max} = 0.996
16504 measured reflections
2336 independent reflections
1700 reflections with $I > 2\sigma(I)$
R_{int} = 0.054

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.132
S = 1.14
2336 reflections
168 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.88 (3)	1.83 (3)	2.643 (2)	153 (3)
N2—H2A···N4^{i}	0.89 (3)	2.40 (3)	3.156 (3)	142 (2)
N2—H2B···N3^{ii}	0.88 (3)	2.26 (3)	3.098 (3)	159 (2)

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GO2051).

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supporting information

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2-Amino-3-[(*E*)-(2-hydroxy-3-methylbenzylidene)amino]but-2-enedinitrile

Elham S. Aazam and Orhan Büyükgüngör

S1. Comment

Tetrameric HCN (diaminomaleonitrile, DAMN) is one of the most versatile reagents in organic chemistry. It has been used as a precursor for producing nucleotides and for synthesizing a wide variety of heterocyclic compounds. These compounds are important as synthetic intermediates and they are also used in pharmacology (Da Silva *et al.*, 2011; Rivera *et al.*, 2006).

Schiff bases derived from DAMN have also been used as versatile ligands in coordination chemistry (Aazam *et al.*, 2011; Kargar *et al.*, 2009; Yeap *et al.*, 2009). There are two types of intra-molecular hydrogen bonds in Schiff bases, which may be stabilized either in keto-amine (N—H···O hydrogen bond) (Hökelek *et al.*, 2000) or phenol-imine (N···H—O hydrogen bond) tautomeric forms (Odabaşoğlu *et al.*, 2005; Aazam & Büyükgüngör, 2010). The present X-ray investigation shows that the title compound is a Schiff base and exists in the phenol-imine form in the solid-state.

The molecular structure of the title compound is shown in Figure 1. An intramolecular O1—H1···N1 hydrogen bond, a characteristic hydrogen bond for Schiff bases, leads to the formation of a S(6) six-membered ring (Figure 1) (Bernstein *et al.*, 1995).

The N2—H2A···N4^{i} (i; -*x*, *y* - 1/2, -*z* + 1/2) and N2—H2B···N3^{ii} (ii; *x*, -*y* + 3/2, *z* - 1/2) hydrogen bonds generate a C(5) zigzag chain running parallel to the *b* axis and a C(6) chain running parallel to the *c* axis, respectively, (Figure 2–3). The intersection of the C(5) and C(6) chains produce alternating $R_4^4(18)$ and $R_4^4(22)$ ring motives, (Figure 4), (Bernstein *et al.*, 1995) which link the molecules into corrugated sheets which lie in the *b,c* plane. Details of the hydrogen bonds are given in Table 1.

S2. Refinement

The H atoms bonded to oxygen and nitrogen atoms were located in Fourier map and refined isotropically. Other hydrogen atoms were positioned geometrically and treated using a riding model, fixing bond lengths at 0.93 and 0.96 Å for CH (aromatic) and CH_3, respectively. The displacement parameters of the H atoms were constrained with U_{iso}(H) = 1.2U_{eq} (aromatic and methyl C).

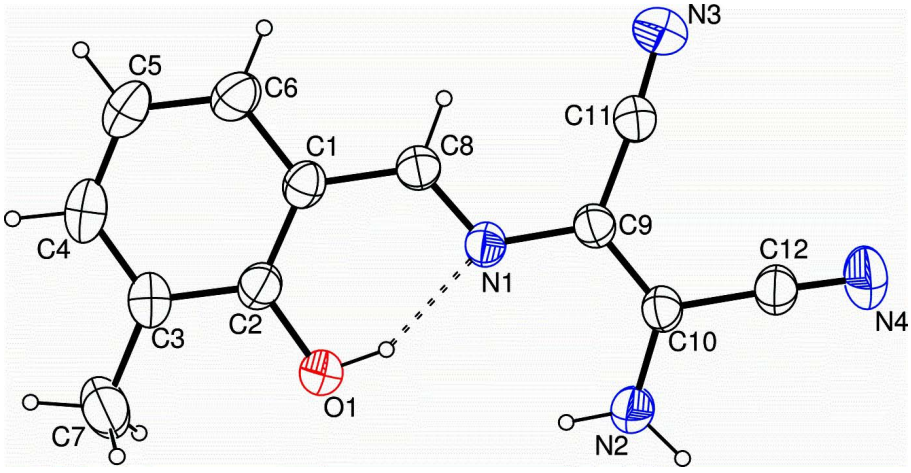

Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids and atomic numbering.

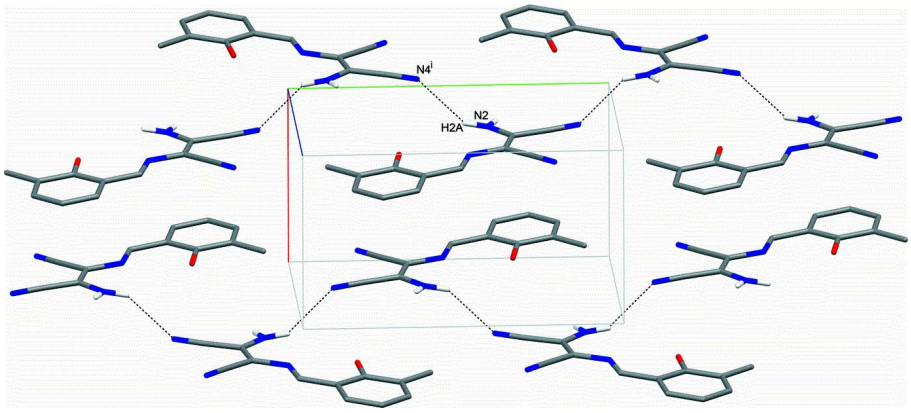

Figure 2

Part of the crystal structure of the title compound, showing the formation of C(5) chains parallel to the *b* axis (i; -*x*, *y* - 1/2, -*z* + 1/2). Hydrogen bonds are indicated by dashed lines.

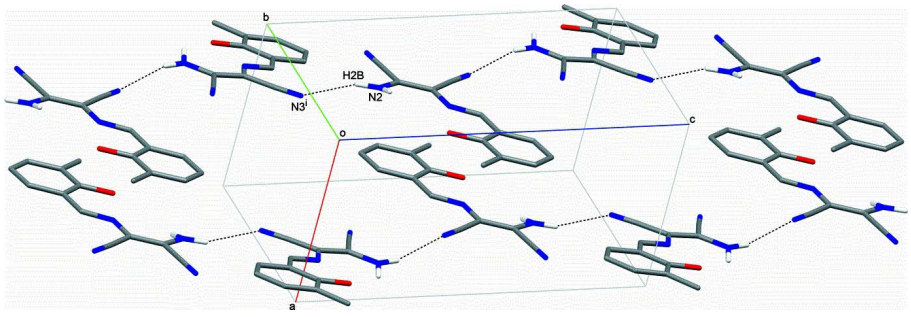

Figure 3

Part of the crystal structure of the title compound, showing the formation of C(6) chains parallel to the *c* axis (i; *x*, -*y* + 3/2, *z* - 1/2). Hydrogen bonds are indicated by dashed lines.

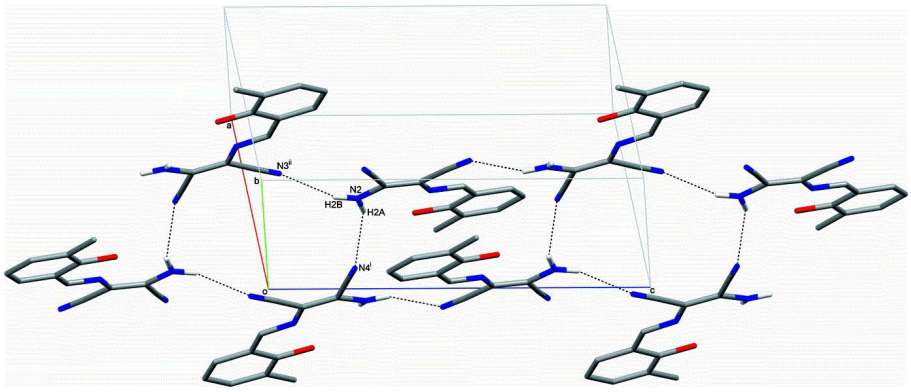

Figure 4

Part of the crystal structure of the title compound, showing the formation of $R^4_4(18)$ and $R^4_4(22)$ rings. Hydrogen bonds are indicated by dashed lines. (Symmetry codes as in Table 1)

2-Amino-3-[(*E*)-(2-hydroxy-3-methylbenzylidene)amino]but-2-enedinitrile

Crystal data

$C_{12}H_{10}N_4O$
$M_r = 226.24$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 6.9041\ (6)$ Å
$b = 11.8791\ (7)$ Å
$c = 14.0282\ (11)$ Å
$\beta = 101.600\ (7)°$
$V = 1127.02\ (15)$ Å^3
$Z = 4$
$F(000) = 472$
$D_x = 1.333$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 16504 reflections
$\theta = 2.3–28.0°$
$\mu = 0.09$ mm^{-1}
$T = 296$ K
Plate, brown
0.76 × 0.48 × 0.03 mm

Data collection

Stoe IPDS 2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
rotation method scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min} = 0.949$, $T_{max} = 0.996$
16504 measured reflections
2336 independent reflections
1700 reflections with $I > 2\sigma(I)$
$R_{int} = 0.054$
$\theta_{max} = 26.5°$, $\theta_{min} = 2.3°$
$h = -8\rightarrow8$
$k = -14\rightarrow14$
$l = -17\rightarrow17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.132$
$S = 1.14$
2336 reflections
168 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0644P)^2 + 0.0085P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.008 (2)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2927 (3)	0.39788 (16)	0.57527 (14)	0.0412 (5)
C2	0.2741 (3)	0.30861 (18)	0.50843 (15)	0.0451 (5)
C3	0.2980 (3)	0.19643 (18)	0.53978 (17)	0.0507 (5)
C4	0.3421 (3)	0.1774 (2)	0.63866 (19)	0.0579 (6)
H4	0.3581	0.1036	0.6609	0.069*
C5	0.3637 (3)	0.2639 (2)	0.70634 (17)	0.0597 (6)
H5	0.3951	0.2480	0.7726	0.072*
C6	0.3382 (3)	0.3731 (2)	0.67447 (16)	0.0518 (5)
H6	0.3514	0.4313	0.7196	0.062*
C7	0.2734 (4)	0.1024 (2)	0.4671 (2)	0.0730 (7)
H7A	0.3735	0.1081	0.4287	0.088*
H7B	0.1452	0.1073	0.4254	0.088*
H7C	0.2860	0.0315	0.5006	0.088*
C8	0.2653 (3)	0.51404 (16)	0.54458 (14)	0.0426 (5)
H8	0.2785	0.5698	0.5920	0.051*
C9	0.2013 (3)	0.65633 (15)	0.42752 (14)	0.0393 (5)
C10	0.1644 (3)	0.68409 (16)	0.33180 (14)	0.0405 (5)
C11	0.2178 (3)	0.74210 (17)	0.50067 (15)	0.0461 (5)
C12	0.1464 (3)	0.80158 (18)	0.30383 (15)	0.0482 (5)
N1	0.2236 (2)	0.54359 (13)	0.45456 (11)	0.0405 (4)
N2	0.1385 (3)	0.61018 (17)	0.25795 (15)	0.0563 (5)
H2A	0.112 (4)	0.538 (3)	0.2695 (19)	0.077 (8)*
H2B	0.130 (4)	0.637 (2)	0.199 (2)	0.076 (8)*
N3	0.2305 (3)	0.80465 (17)	0.56309 (15)	0.0664 (6)
N4	0.1280 (3)	0.89245 (17)	0.27921 (17)	0.0709 (6)
O1	0.2304 (3)	0.32719 (15)	0.41171 (12)	0.0668 (5)
H1	0.217 (4)	0.400 (3)	0.405 (2)	0.081 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0386 (10)	0.0447 (11)	0.0419 (11)	0.0035 (8)	0.0116 (8)	0.0071 (9)
C2	0.0471 (11)	0.0469 (12)	0.0413 (12)	0.0045 (9)	0.0090 (9)	0.0061 (9)
C3	0.0485 (12)	0.0429 (12)	0.0594 (14)	0.0028 (9)	0.0076 (10)	0.0095 (10)
C4	0.0520 (13)	0.0504 (13)	0.0705 (17)	0.0023 (10)	0.0108 (11)	0.0236 (12)
C5	0.0649 (14)	0.0679 (16)	0.0466 (13)	0.0040 (11)	0.0120 (11)	0.0215 (12)

C6	0.0563 (13)	0.0597 (14)	0.0417 (12)	0.0015 (10)	0.0149 (10)	0.0064 (10)
C7	0.0825 (17)	0.0444 (13)	0.0863 (19)	0.0090 (12)	0.0027 (14)	−0.0023 (13)
C8	0.0473 (11)	0.0408 (11)	0.0415 (12)	0.0017 (8)	0.0135 (8)	0.0006 (9)
C9	0.0416 (10)	0.0355 (10)	0.0421 (11)	−0.0004 (8)	0.0113 (8)	0.0004 (8)
C10	0.0430 (10)	0.0345 (10)	0.0436 (12)	−0.0030 (8)	0.0077 (8)	0.0026 (8)
C11	0.0566 (13)	0.0382 (11)	0.0446 (12)	0.0035 (9)	0.0127 (10)	0.0029 (10)
C12	0.0548 (12)	0.0404 (12)	0.0482 (12)	−0.0029 (9)	0.0072 (9)	0.0055 (10)
N1	0.0450 (9)	0.0367 (8)	0.0411 (10)	0.0015 (7)	0.0114 (7)	0.0039 (7)
N2	0.0836 (14)	0.0420 (11)	0.0416 (11)	−0.0110 (10)	0.0084 (9)	0.0022 (9)
N3	0.0917 (15)	0.0538 (12)	0.0540 (12)	0.0047 (10)	0.0151 (11)	−0.0096 (10)
N4	0.0901 (15)	0.0433 (11)	0.0779 (15)	0.0048 (10)	0.0134 (12)	0.0155 (10)
O1	0.1120 (14)	0.0444 (9)	0.0415 (10)	0.0164 (9)	0.0098 (9)	0.0023 (7)

Geometric parameters (Å, º)

C1—C6	1.395 (3)	C7—H7C	0.9600
C1—C2	1.404 (3)	C8—N1	1.286 (2)
C1—C8	1.447 (3)	C8—H8	0.9300
C2—O1	1.348 (3)	C9—C10	1.356 (3)
C2—C3	1.403 (3)	C9—N1	1.392 (2)
C3—C4	1.378 (3)	C9—C11	1.434 (3)
C3—C7	1.499 (3)	C10—N2	1.342 (3)
C4—C5	1.386 (4)	C10—C12	1.448 (3)
C4—H4	0.9300	C11—N3	1.138 (3)
C5—C6	1.372 (3)	C12—N4	1.133 (3)
C5—H5	0.9300	N2—H2A	0.89 (3)
C6—H6	0.9300	N2—H2B	0.88 (3)
C7—H7A	0.9600	O1—H1	0.88 (3)
C7—H7B	0.9600		
C6—C1—C2	118.59 (18)	H7A—C7—H7B	109.5
C6—C1—C8	119.22 (19)	C3—C7—H7C	109.5
C2—C1—C8	122.19 (18)	H7A—C7—H7C	109.5
O1—C2—C3	117.37 (19)	H7B—C7—H7C	109.5
O1—C2—C1	121.37 (18)	N1—C8—C1	122.86 (18)
C3—C2—C1	121.26 (19)	N1—C8—H8	118.6
C4—C3—C2	117.4 (2)	C1—C8—H8	118.6
C4—C3—C7	122.3 (2)	C10—C9—N1	119.49 (17)
C2—C3—C7	120.3 (2)	C10—C9—C11	120.52 (17)
C3—C4—C5	122.7 (2)	N1—C9—C11	119.99 (17)
C3—C4—H4	118.7	N2—C10—C9	125.07 (19)
C5—C4—H4	118.7	N2—C10—C12	115.50 (19)
C6—C5—C4	119.2 (2)	C9—C10—C12	119.43 (18)
C6—C5—H5	120.4	N3—C11—C9	175.5 (2)
C4—C5—H5	120.4	N4—C12—C10	177.7 (2)
C5—C6—C1	120.9 (2)	C8—N1—C9	121.38 (16)
C5—C6—H6	119.6	C10—N2—H2A	118.9 (17)
C1—C6—H6	119.6	C10—N2—H2B	117.7 (18)

C3—C7—H7A	109.5	H2A—N2—H2B	123 (2)
C3—C7—H7B	109.5	C2—O1—H1	105.2 (19)
C6—C1—C2—O1	179.99 (18)	C2—C1—C6—C5	−0.2 (3)
C8—C1—C2—O1	0.3 (3)	C8—C1—C6—C5	179.5 (2)
C6—C1—C2—C3	0.8 (3)	C6—C1—C8—N1	179.86 (18)
C8—C1—C2—C3	−178.93 (19)	C2—C1—C8—N1	−0.4 (3)
O1—C2—C3—C4	−179.80 (19)	N1—C9—C10—N2	2.9 (3)
C1—C2—C3—C4	−0.6 (3)	C11—C9—C10—N2	−177.18 (19)
O1—C2—C3—C7	−0.5 (3)	N1—C9—C10—C12	−178.38 (17)
C1—C2—C3—C7	178.8 (2)	C11—C9—C10—C12	1.5 (3)
C2—C3—C4—C5	−0.2 (3)	C1—C8—N1—C9	−178.97 (18)
C7—C3—C4—C5	−179.5 (2)	C10—C9—N1—C8	177.62 (18)
C3—C4—C5—C6	0.8 (3)	C11—C9—N1—C8	−2.3 (3)
C4—C5—C6—C1	−0.5 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.88 (3)	1.83 (3)	2.643 (2)	153 (3)
N2—H2*A*···N4^{i}	0.89 (3)	2.40 (3)	3.156 (3)	142 (2)
N2—H2*B*···N3^{ii}	0.88 (3)	2.26 (3)	3.098 (3)	159 (2)

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