Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Amino-3-(4-hydroxybenzylideneamino)-butenedinitrile monohydrate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.050
wR factor = 0.137
Data-to-parameter ratio = 15.1

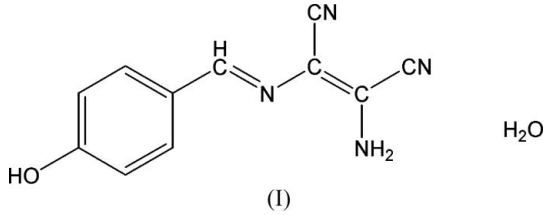
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 3 January 2006
Accepted 24 January 2006

In the crystal structure of the title compound, $C_{11}H_8N_4O\cdot H_2O$, the molecules are held together by O—H···O, O—H···N and N—H···N hydrogen bonds. The compound exhibits strong fluorescent emission in the solid state.

Comment

2-Amino-3-(4-hydroxybenzylidene)aminobutenedinitrile, (I), is a useful compound in the dye and medicine synthesis fields (Begland *et al.*, 1974; Begland & Del, 1975), but its crystal structure is unknown to date. The crystal structures of the related compounds 2-amino-3-(2-hydroxybenzylideneamino)-2,3-butenedinitrile and 2-amino-3-[4-(diethylamino)-2-hydroxybenzylideneamino]-2,3-butenedinitrile have been reported by MacLachlan *et al.* (1996) and Costes *et al.* (2005), respectively. As part of an investigation of its physical properties, we report the molecular structure of the title compound, (I) (Fig. 1).



(I)

The butenedinitrile molecule is approximately planar, with a dihedral angle of 7.73 (3)° between the planes of the hydroxylbenzylidene and diaminomaleonitrile groups. The bond lengths imply that all non-H atoms of the molecule are involved in a conjugated system. Hydrogen bonds (Table 1) connect the water molecule and the hydroxy group, the water molecule and the cyano group, and the amino and cyano groups. The title compound exhibits a strong fluorescence spectrum with a maximum emission peak at 564 nm when the excitation wavelength is selected at 320 nm.

Experimental

4-Hydroxybenzaldehyde (3.5 g, 0.0287 mol) and diaminomaleonitrile (3.1 g, 0.0287 mol) were added to ethanol (60 ml) and the mixture was refluxed for 30 min. The crude product was filtered off and washed with ethanol. Yellow crystals were obtained from an acetonitrile solution over a period of two weeks at room temperature. Analysis calculated for $C_{11}H_{10}N_4O_2$: C 57.38, H 4.38, N 24.34%; found: C 57.43, H 4.61, N 24.05%. The IR spectrum contains strong peaks at 2200 and 1611 cm^{-1} for the vibrations of cyano groups and the C═N bond, respectively.

Crystal data

$C_{11}H_{10}N_4O_2$
M_r = 230.23
Monoclinic, $P2_1/n$
a = 9.848 (2) Å
b = 7.0388 (15) Å
c = 16.473 (3) Å
β = 90.604 (3)°
V = 1141.9 (4) Å^3
Z = 4
D_x = 1.339 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2655 reflections
θ = 2.4–27.6°
μ = 0.10 mm^{-1}
T = 298 (2) K
Prism, yellow
0.23 × 0.21 × 0.19 mm

Data collection

Bruker SMART APEX CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.978, T_{max} = 0.982
6301 measured reflections
2358 independent reflections
1943 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 26.5°
$h = -11 \rightarrow 12$
$k = -8 \rightarrow 8$
$l = -20 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.137
S = 1.03
2358 reflections
156 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0884P)^2 + 0.0552P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.024 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H4···O2^{i}	0.82	1.87	2.6848 (16)	169
O2—H7···O1^{ii}	0.88	2.04	2.8283 (15)	148
O2—H8···N2^{iii}	0.89	2.26	3.115 (2)	161
N4—H4*A*···N2^{iii}	0.86	2.42	3.1691 (19)	146
N4—H4*B*···N3^{iv}	0.86	2.15	3.0053 (18)	171

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

The H atoms of the water molecule were located in a difference Fourier map and refined as riding, with O—H = 0.88 and 0.89 Å and U_{iso}(H) = 1.5U_{eq}(O). Other H atoms were placed in calculated positions, with C—H = 0.93 Å, O—H = 0.82 Å and N—H = 0.93 Å, and were refined as riding, with U_{iso}(H) = 1.2U_{eq}(C,*N*) and 1.5U_{eq}(O).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

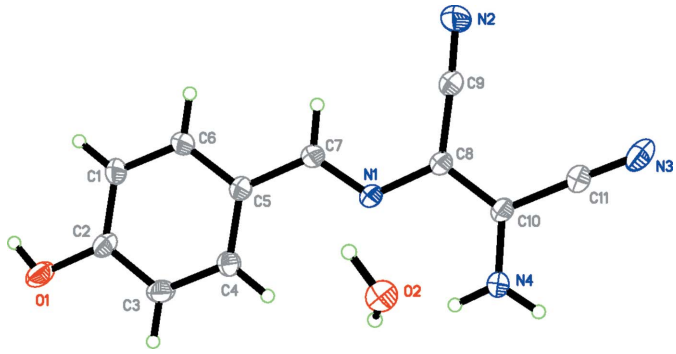


Figure 1
The molecular structure of (I), showing the atom-numbering scheme, with displacement ellipsoids drawn at the 30% probability level.

The authors thank the Natural Science Foundation of China (grant No. 20271043).

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