

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

N'-(3,4-Dihydroxybenzylidene)-acetohydrazide

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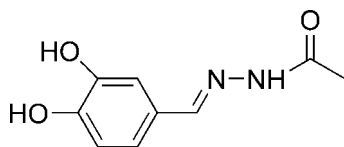
Received 2 August 2009; accepted 2 August 2009

 Key indicators: single-crystal X-ray study; $T = 223$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.037; wR factor = 0.111; data-to-parameter ratio = 16.0.

In the title compound, $\text{C}_9\text{H}_{10}\text{N}_2\text{O}_3$, the Schiff base molecule is approximately planar, the dihedral angle between the benzene ring and the acetohydrazide group (r.m.s. deviation = 0.034 Å) being 8.81 (7)°. An intramolecular O—H...O hydrogen bond is observed. In the crystal, molecules are linked into a three-dimensional network by O—H...O, N—H...O and C—H...O hydrogen bonds.

Related literature

For general background to Schiff bases, see: Cimerman *et al.* (1997); Offe *et al.* (1952); Richardson *et al.* (1988). For related structures, see: Li *et al.* (2008); Tamboura *et al.* (2009).



Experimental

Crystal data

 $\text{C}_9\text{H}_{10}\text{N}_2\text{O}_3$
 $M_r = 194.19$
 Monoclinic, $P2_1/c$
 $a = 10.598$ (2) Å
 $b = 8.5017$ (16) Å

 $c = 10.621$ (2) Å
 $\beta = 107.232$ (7)°
 $V = 913.9$ (3) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

 $\mu = 0.11$ mm⁻¹
 $T = 223$ K

 $0.25 \times 0.24 \times 0.20$ mm

Data collection

 Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\min} = 0.972$, $T_{\max} = 0.980$

 5752 measured reflections
 2066 independent reflections
 1665 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.111$
 $S = 0.94$
 2066 reflections

 129 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.17$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
N2—H2...O1 ⁱ	0.86	2.17	2.9692 (15)	154
O2—H2A...O1 ⁱⁱ	0.82	1.96	2.7206 (13)	154
O3—H3...O2	0.82	2.26	2.7109 (14)	115
O3—H3...O2 ⁱⁱⁱ	0.82	2.14	2.7784 (14)	134
C9—H9C...O3 ^{iv}	0.96	2.51	3.445 (2)	166

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

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); 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.

The authors thank the Science and Technology Project of Zhejiang Province (grant No. 2007 F70077) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2878).

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supplementary materials

Acta Cryst. (2009). E65, o2097 [doi:10.1107/S1600536809030712]

N'-(3,4-Dihydroxybenzylidene)acetohydrazide

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Comment

Schiff bases have attracted much attention due to the possibility of their analytical applications (Cimerman *et al.*, 1997). They are also important ligands, which have been reported to have mild bacteriostatic activity and as potential oral iron-chelating drugs for genetic disorders such as thalassemia (Offe *et al.*, 1952; Richardson *et al.*, 1988). Metal complexes based on Schiff bases have received considerable attention because they can be utilized as model compounds of active centres in various complexes (Tamboura *et al.*, 2009). We report here the crystal structure of the title compound (Fig. 1).

In the Schiff base molecule, the acetohydrazide group (O1/N1/N2/C8/C9) is planar (r.m.s. deviation 0.034 Å) and it forms a dihedral angle of 8.81 (7)° with the benzene (C1-C6) ring. The molecule adopts a *trans* configuration with respect to the C=N bond. Bond lengths and angles are comparable to those observed for *N'*-[1-(4-methoxyphenyl)ethylidene]acetohydrazide (Li *et al.*, 2008). An intramolecular O3—H3···O2 hydrogen bond is observed.

In the crystal, molecules are linked into a three-dimensional network (Fig.2) by O—H···O, N—H···O and C—H···O hydrogen bonds (Table 1).

Experimental

3,4-Dihydroxybenzaldehyde (1.38 g, 0.01 mol) and acetohydrazide (0.74 g, 0.01 mol) were dissolved in stirred methanol (20 ml) and left for 2.5 h at room temperature. The resulting solid was filtered off and recrystallized from ethanol to give the title compound in 85% yield. Single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution at room temperature (m.p. 475–477 K).

Refinement

H atoms were positioned geometrically (N-H = 0.86 Å, O-H = 0.82 Å and C-H = 0.93 or 0.96 Å) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ and $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$.

Figures

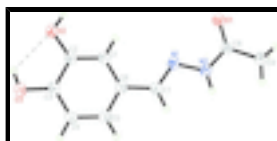


Fig. 1. The molecular structure of the title compound. Displacement ellipsoids are drawn at the 40% probability level. The dashed line indicates a hydrogen bond.

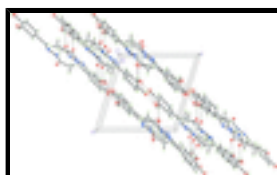


Fig. 2. Part of the crystal packing of the title compound. Hydrogen bonds are shown as dashed lines.

N'-(3,4-Dihydroxybenzylidene)acetohydrazide

Crystal data

$C_9H_{10}N_2O_3$	$F_{000} = 408$
$M_r = 194.19$	$D_x = 1.411 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2ybc	Cell parameters from 2066 reflections
$a = 10.598 (2) \text{ \AA}$	$\theta = 2.0\text{--}27.4^\circ$
$b = 8.5017 (16) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$c = 10.621 (2) \text{ \AA}$	$T = 223 \text{ K}$
$\beta = 107.232 (7)^\circ$	Block, colourless
$V = 913.9 (3) \text{ \AA}^3$	$0.25 \times 0.24 \times 0.20 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	2066 independent reflections
Radiation source: fine-focus sealed tube	1665 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.023$
$T = 223 \text{ K}$	$\theta_{\text{max}} = 27.4^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.0^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2002)	$h = -13 \rightarrow 13$
$T_{\text{min}} = 0.972$, $T_{\text{max}} = 0.980$	$k = -10 \rightarrow 10$
5752 measured reflections	$l = -13 \rightarrow 12$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.037$	$w = 1/[\sigma^2(F_o^2) + (0.0674P)^2 + 0.2108P]$
$wR(F^2) = 0.111$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.94$	$(\Delta/\sigma)_{\text{max}} = 0.009$
2066 reflections	$\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$
129 parameters	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kF_c[1 + 0.001xF_c^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.019 (4)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N2	1.00361 (10)	0.13127 (13)	0.68707 (10)	0.0357 (3)
H2	0.9880	0.0352	0.7029	0.043*
O3	0.47733 (10)	0.25734 (12)	0.01091 (10)	0.0522 (3)
H3	0.4848	0.3492	-0.0093	0.078*
O2	0.64301 (9)	0.48688 (11)	0.14025 (10)	0.0424 (3)
H2A	0.6973	0.5473	0.1870	0.064*
O1	1.13124 (9)	0.34588 (11)	0.75557 (10)	0.0445 (3)
N1	0.93041 (10)	0.20010 (13)	0.56882 (11)	0.0362 (3)
C6	0.75370 (12)	0.15246 (15)	0.36828 (13)	0.0345 (3)
C3	0.56800 (13)	0.22557 (16)	0.12889 (13)	0.0374 (3)
C1	0.74814 (12)	0.30536 (15)	0.31694 (13)	0.0338 (3)
H1	0.8063	0.3821	0.3628	0.041*
C2	0.65615 (12)	0.34155 (15)	0.19818 (12)	0.0333 (3)
C5	0.66449 (13)	0.03947 (16)	0.29930 (14)	0.0397 (3)
H5	0.6673	-0.0617	0.3334	0.048*
C8	1.09751 (12)	0.21065 (15)	0.77666 (13)	0.0336 (3)
C9	1.15560 (15)	0.12587 (18)	0.90450 (13)	0.0440 (3)
H9A	1.1378	0.0153	0.8919	0.066*
H9B	1.1169	0.1653	0.9693	0.066*
H9C	1.2494	0.1427	0.9342	0.066*
C7	0.84748 (13)	0.10590 (15)	0.49377 (13)	0.0359 (3)
H7	0.8468	0.0018	0.5205	0.043*
C4	0.57178 (14)	0.07602 (16)	0.18054 (14)	0.0412 (3)
H4	0.5122	0.0000	0.1357	0.049*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.0383 (6)	0.0271 (6)	0.0362 (6)	-0.0028 (4)	0.0024 (5)	0.0041 (4)
O3	0.0513 (6)	0.0409 (6)	0.0465 (6)	-0.0082 (4)	-0.0132 (5)	0.0037 (4)
O2	0.0409 (5)	0.0333 (5)	0.0426 (6)	-0.0053 (4)	-0.0036 (4)	0.0054 (4)
O1	0.0454 (5)	0.0301 (5)	0.0501 (6)	-0.0069 (4)	0.0019 (4)	0.0001 (4)

supplementary materials

N1	0.0360 (6)	0.0332 (6)	0.0354 (6)	0.0007 (4)	0.0045 (5)	0.0048 (4)
C6	0.0347 (6)	0.0349 (7)	0.0326 (7)	-0.0013 (5)	0.0081 (5)	0.0000 (5)
C3	0.0341 (7)	0.0385 (7)	0.0347 (7)	-0.0021 (5)	0.0024 (5)	-0.0019 (5)
C1	0.0322 (6)	0.0320 (7)	0.0345 (7)	-0.0046 (5)	0.0060 (5)	-0.0027 (5)
C2	0.0326 (6)	0.0304 (6)	0.0350 (7)	-0.0018 (5)	0.0070 (5)	-0.0001 (5)
C5	0.0436 (7)	0.0323 (7)	0.0402 (7)	-0.0048 (5)	0.0079 (6)	0.0020 (5)
C8	0.0332 (6)	0.0288 (6)	0.0376 (7)	0.0014 (5)	0.0086 (5)	-0.0027 (5)
C9	0.0470 (8)	0.0416 (8)	0.0376 (8)	0.0004 (6)	0.0038 (6)	0.0022 (6)
C7	0.0384 (7)	0.0315 (7)	0.0363 (7)	-0.0022 (5)	0.0087 (5)	0.0024 (5)
C4	0.0406 (7)	0.0355 (7)	0.0416 (8)	-0.0097 (5)	0.0031 (6)	-0.0047 (6)

Geometric parameters (Å, °)

N2—C8	1.3380 (16)	C3—C4	1.381 (2)
N2—N1	1.3944 (14)	C3—C2	1.4069 (18)
N2—H2	0.86	C1—C2	1.3811 (18)
O3—C3	1.3612 (16)	C1—H1	0.93
O3—H3	0.82	C5—C4	1.3851 (19)
O2—C2	1.3689 (16)	C5—H5	0.93
O2—H2A	0.82	C8—C9	1.4989 (19)
O1—C8	1.2438 (16)	C9—H9A	0.96
N1—C7	1.2783 (17)	C9—H9B	0.96
C6—C5	1.3939 (18)	C9—H9C	0.96
C6—C1	1.4041 (18)	C7—H7	0.93
C6—C7	1.4615 (18)	C4—H4	0.93
C8—N2—N1	121.80 (11)	C4—C5—C6	120.78 (13)
C8—N2—H2	119.1	C4—C5—H5	119.6
N1—N2—H2	119.1	C6—C5—H5	119.6
C3—O3—H3	109.5	O1—C8—N2	122.07 (12)
C2—O2—H2A	109.5	O1—C8—C9	123.01 (12)
C7—N1—N2	113.26 (11)	N2—C8—C9	114.90 (12)
C5—C6—C1	119.34 (12)	C8—C9—H9A	109.5
C5—C6—C7	117.70 (12)	C8—C9—H9B	109.5
C1—C6—C7	122.94 (12)	H9A—C9—H9B	109.5
O3—C3—C4	118.55 (12)	C8—C9—H9C	109.5
O3—C3—C2	121.38 (12)	H9A—C9—H9C	109.5
C4—C3—C2	120.06 (12)	H9B—C9—H9C	109.5
C2—C1—C6	119.86 (12)	N1—C7—C6	123.72 (12)
C2—C1—H1	120.1	N1—C7—H7	118.1
C6—C1—H1	120.1	C6—C7—H7	118.1
O2—C2—C1	124.19 (11)	C3—C4—C5	119.85 (12)
O2—C2—C3	115.73 (11)	C3—C4—H4	120.1
C1—C2—C3	120.09 (12)	C5—C4—H4	120.1
C8—N2—N1—C7	178.32 (12)	C7—C6—C5—C4	179.18 (13)
C5—C6—C1—C2	-0.99 (19)	N1—N2—C8—O1	-5.29 (19)
C7—C6—C1—C2	-179.44 (12)	N1—N2—C8—C9	173.18 (11)
C6—C1—C2—O2	179.96 (12)	N2—N1—C7—C6	176.10 (11)
C6—C1—C2—C3	0.13 (19)	C5—C6—C7—N1	-176.99 (13)
O3—C3—C2—O2	0.93 (19)	C1—C6—C7—N1	1.5 (2)

C4—C3—C2—O2	-178.75 (12)	O3—C3—C4—C5	178.87 (13)
O3—C3—C2—C1	-179.22 (12)	C2—C3—C4—C5	-1.4 (2)
C4—C3—C2—C1	1.1 (2)	C6—C5—C4—C3	0.6 (2)
C1—C6—C5—C4	0.7 (2)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2 \cdots O1 ⁱ	0.86	2.17	2.9692 (15)	154
O2—H2A \cdots O1 ⁱⁱ	0.82	1.96	2.7206 (13)	154
O3—H3 \cdots O2	0.82	2.26	2.7109 (14)	115
O3—H3 \cdots O2 ⁱⁱⁱ	0.82	2.14	2.7784 (14)	134
C9—H9C \cdots O3 ^{iv}	0.96	2.51	3.445 (2)	166

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

Fig. 1

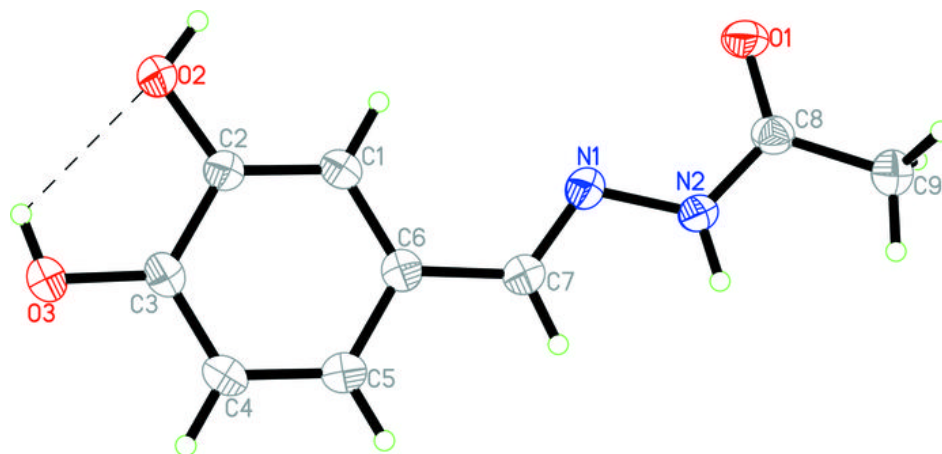


Fig. 2

