Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-(3,4-Dihydroxybenzylidene)-acetohydrazide monohydrate

Lu-Ping Lv,[a] Tie-Ming Yu,[a] Wen-Bo Yu,[a] Wei-Wei Li[a] and Xian-Chao Hu[b]*

[a]Department of Chemical Engineering, Hangzhou Vocational and Technical College, Hangzhou 310018, People's Republic of China, and [b]Research Center for Analysis and Measurement, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China
Correspondence e-mail: zgdhxc@126.com

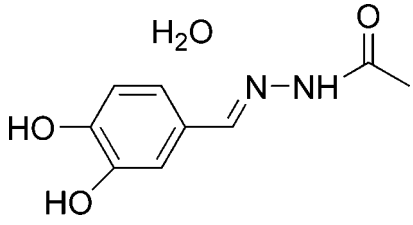
Received 10 July 2009; accepted 11 July 2009

Key indicators: single-crystal X-ray study; T = 223 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.102; data-to-parameter ratio = 11.9.

In the title compound, $C_9H_{10}N_2O_3\cdot H_2O$, the Schiff base molecule is approximately planar, the dihedral angle between the benzene and acetohydrazide planes being 5.40 (7)°. An intramolecular O—H···O hydrogen bond is observed. In the crystal, molecules are linked into a two-dimensional network parallel to (100) by O—H···O, N—H···O, O—H···N and C—H···O hydrogen bonds, and by π–π interactions between symmetry-related benzene rings [centroid–centroid distance = 3.543 (2) Å].

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 & Jian (2008); Tamboura *et al.* (2009).



Experimental

Crystal data

$C_9H_{10}N_2O_3\cdot H_2O$
M_r = 212.21
Monoclinic, $P2_1/c$
a = 9.325 (4) Å
b = 13.877 (7) Å
c = 8.210 (4) Å
β = 106.435 (5)°
V = 1019.0 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 223 K
0.25 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.977, T_{max} = 0.979
5060 measured reflections
1765 independent reflections
1640 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.102
S = 1.03
1765 reflections
148 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2	0.82	2.22	2.6694 (18)	115
O1—H1···O1W^{i}	0.82	2.11	2.8529 (18)	151
O1W—H1F···O3^{ii}	0.80 (3)	2.31 (3)	3.031 (2)	152 (3)
O1W—H1F···N1^{ii}	0.80 (3)	2.48 (3)	3.101 (2)	135 (2)
O2—H2···O1W	0.82	1.96	2.7736 (18)	171
N2—H2A···O3^{iii}	0.86	2.09	2.9110 (19)	160
C7—H7···O3^{iii}	0.93	2.53	3.311 (2)	142

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

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: CI2851).

References

Bruker (2002). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cimerman, Z., Galic, N. & Bosner, B. (1997). *Anal. Chim. Acta*, **343**, 145–153.
Li, Y.-F. & Jian, F.-F. (2008). *Acta Cryst.* E**64**, o2409.
Offe, H. A., Siefen, W. & Domagk, G. (1952). *Z. Naturforsch. Teil B*, **7**, 446–447.
Richardson, D., Baker, E., Ponka, P., Wilairat, P., Vitolo, M. L. & Webb, J. (1988). *Thalassemia: Pathophysiology and Management*, Part B, p. 81. New York: Alan R. Liss Inc.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tamboura, F. B., Gaye, M., Sall, A. S., Barry, A. H. & Bah, Y. (2009). *Acta Cryst.* E**65**, m160–m161.

supporting information

Acta Cryst. (2009). E**65**, o1897 [doi:10.1107/S1600536809027299]

N′-(3,4-Dihydroxybenzylidene)acetohydrazide monohydrate

Lu-Ping Lv, Tie-Ming Yu, Wen-Bo Yu, Wei-Wei Li and Xian-Chao Hu

S1. 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 is planar and it forms a dihedral angle of 5.40 (7)° with the benzene 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 O1—H1···O2 hydrogen bond is observed.

In the crystal, the Schiff base and water molecules are linked into a two-dimensional network by O—H···O, N—H···O, O—H···N and C—H···O hydrogen bonds (Table 1). In the network, an intermolecular π-π interaction is also observed between the benzene rings of the molecules at (x, y, z) and (1-x, 1-y, -z), with a centroid-to-centroid distance of 3.543 (2) Å.

S2. Experimental

3,4-Dihydroxybenzaldehyde (1.38 g, 0.01 mol) and acetohydrazide (0.74 g, 0.01 mol) were dissolved in stirred methanol (15 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 95% yield. Single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution at room temperature (m.p. 460–462 K).

S3. Refinement

H atoms of the water molecule were located in a difference map and were refined freely. All other H atoms were positioned geometrically (N-H = 0.86 Å, O-H = 0.82Å and C-H = 0.93 or 0.96Å) and refined using a riding model, with U_{iso}(H) =1.2U_{eq}(C,N) and 1.5U_{eq}(C_{methyl}).

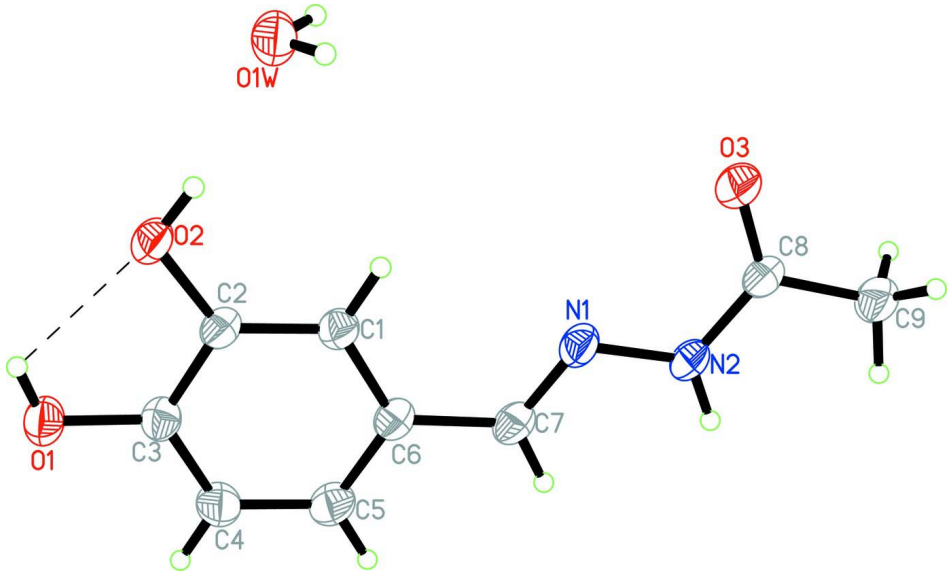


Figure 1

The asymmetric unit of the title compound. Displacement ellipsoids are drawn at the 40% probability level. Dashed lines indicate hydrogen bonds.

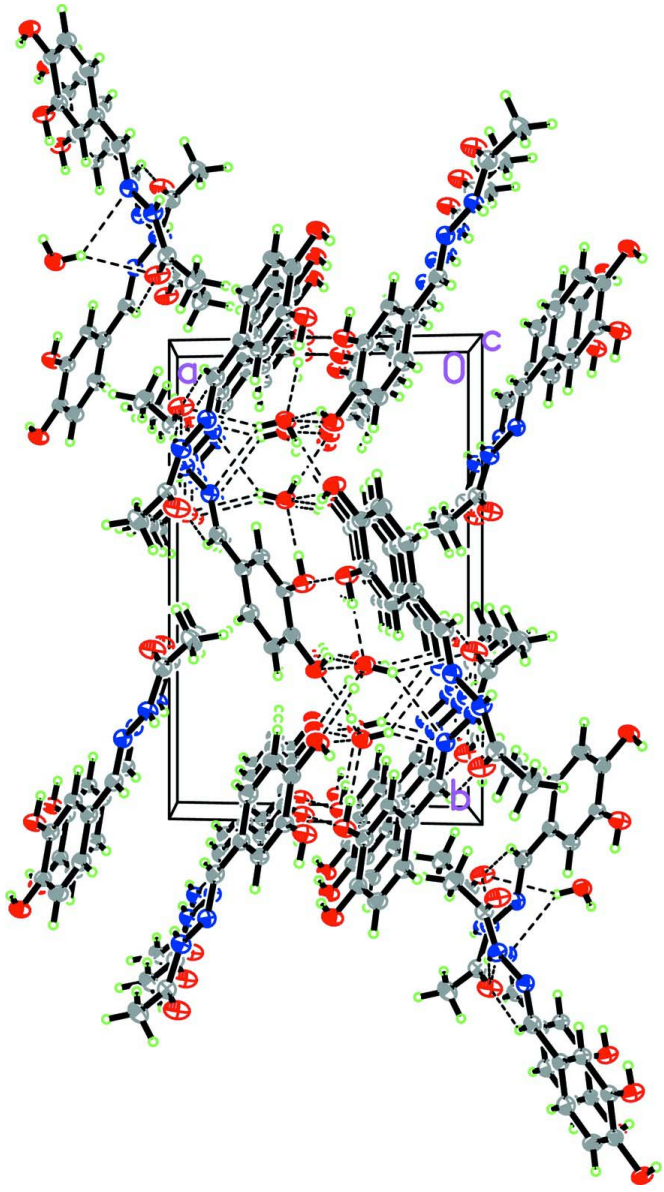


Figure 2

Crystal packing of the title compound. Hydrogen bonds are shown as dashed lines.

N′-(3,4-Dihydroxybenzylidene)acetohydrazide monohydrate

Crystal data

$C_9H_{10}N_2O_3·H_2O$
$M_r = 212.21$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.325$ (4) Å
$b = 13.877$ (7) Å
$c = 8.210$ (4) Å
$\beta = 106.435$ (5)°
$V = 1019.0$ (8) Å^3
$Z = 4$
$F(000) = 448$
$D_x = 1.383$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1765 reflections
$\theta = 2.3$–25.0°
$\mu = 0.11$ mm^{-1}
$T = 223$ K
Block, colourless
0.25 × 0.22 × 0.20 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, 2002)
T_{min} = 0.977, T_{max} = 0.979
5060 measured reflections
1765 independent reflections
1640 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 25.0°, θ_{min} = 2.3°
h = −10→11
k = −16→15
l = −9→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.102
S = 1.03
1765 reflections
148 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
$w = 1/[\sigma^2(F_o^2) + (0.061P)^2 + 0.2305P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.048 (5)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
H1E	0.587 (2)	0.2853 (17)	0.445 (3)	0.074 (7)*
H1F	0.708 (3)	0.3160 (19)	0.554 (4)	0.098 (9)*
O1	0.52471 (13)	0.68267 (7)	0.22048 (14)	0.0454 (3)
H1	0.4899	0.6582	0.2919	0.068*
O2	0.56486 (12)	0.50802 (7)	0.36246 (13)	0.0457 (3)
H2	0.5820	0.4528	0.3981	0.068*
O3	0.95803 (14)	0.14282 (8)	0.12355 (14)	0.0552 (4)
C1	0.70188 (15)	0.45382 (10)	0.16721 (16)	0.0341 (3)
H1A	0.7158	0.3923	0.2140	0.041*
C6	0.75933 (14)	0.47698 (10)	0.03162 (16)	0.0338 (3)
C2	0.62483 (15)	0.52199 (9)	0.23148 (16)	0.0328 (3)
C3	0.60346 (15)	0.61461 (10)	0.16033 (17)	0.0345 (3)
C7	0.84173 (15)	0.40745 (10)	−0.04047 (17)	0.0366 (3)
H7	0.8769	0.4262	−0.1309	0.044*
C8	0.99310 (16)	0.17592 (10)	0.00126 (17)	0.0375 (3)

C4	0.66068 (17)	0.63811 (11)	0.02822 (19)	0.0418 (4)
H4	0.6473	0.6998	−0.0179	0.050*
C5	0.73807 (16)	0.56971 (11)	−0.03575 (18)	0.0412 (4)
H5	0.7765	0.5859	−0.1251	0.049*
N1	0.86712 (13)	0.32131 (8)	0.01702 (14)	0.0364 (3)
N2	0.94966 (13)	0.26400 (9)	−0.06162 (14)	0.0380 (3)
H2A	0.9728	0.2846	−0.1497	0.046*
C9	1.08651 (18)	0.12110 (13)	−0.08811 (19)	0.0483 (4)
H9A	1.1843	0.1107	−0.0117	0.072*
H9B	1.0949	0.1573	−0.1846	0.072*
H9C	1.0403	0.0601	−0.1254	0.072*
O1W	0.62190 (16)	0.32929 (8)	0.51868 (17)	0.0472 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0601 (7)	0.0354 (6)	0.0490 (6)	0.0077 (5)	0.0288 (5)	0.0011 (4)
O2	0.0665 (7)	0.0386 (6)	0.0438 (6)	0.0082 (5)	0.0350 (5)	0.0048 (4)
O3	0.0887 (9)	0.0445 (6)	0.0447 (6)	0.0080 (6)	0.0390 (6)	0.0052 (5)
C1	0.0409 (7)	0.0310 (7)	0.0331 (7)	0.0005 (5)	0.0148 (6)	0.0008 (5)
C6	0.0349 (7)	0.0369 (7)	0.0319 (7)	−0.0028 (5)	0.0133 (5)	−0.0022 (5)
C2	0.0366 (7)	0.0349 (7)	0.0295 (6)	−0.0026 (5)	0.0138 (5)	−0.0019 (5)
C3	0.0365 (7)	0.0322 (7)	0.0362 (7)	−0.0008 (5)	0.0127 (5)	−0.0031 (5)
C7	0.0399 (7)	0.0420 (8)	0.0324 (7)	−0.0034 (6)	0.0175 (6)	−0.0014 (6)
C8	0.0438 (8)	0.0430 (8)	0.0273 (6)	0.0011 (6)	0.0126 (6)	−0.0035 (5)
C4	0.0499 (8)	0.0342 (8)	0.0461 (8)	0.0014 (6)	0.0213 (7)	0.0073 (6)
C5	0.0457 (8)	0.0443 (8)	0.0401 (8)	−0.0023 (6)	0.0229 (6)	0.0055 (6)
N1	0.0408 (6)	0.0401 (7)	0.0336 (6)	0.0012 (5)	0.0191 (5)	−0.0028 (5)
N2	0.0478 (7)	0.0425 (7)	0.0317 (6)	0.0049 (5)	0.0242 (5)	0.0012 (5)
C9	0.0533 (9)	0.0556 (10)	0.0388 (8)	0.0155 (7)	0.0177 (7)	0.0000 (7)
O1W	0.0532 (7)	0.0412 (6)	0.0542 (7)	0.0047 (5)	0.0265 (6)	−0.0013 (5)

Geometric parameters (Å, º)

O1—C3	1.3713 (17)	C7—H7	0.93
O1—H1	0.82	C8—N2	1.3439 (19)
O2—C2	1.3591 (17)	C8—C9	1.496 (2)
O2—H2	0.82	C4—C5	1.383 (2)
O3—C8	1.2296 (18)	C4—H4	0.93
C1—C2	1.3799 (19)	C5—H5	0.93
C1—C6	1.4025 (19)	N1—N2	1.3868 (16)
C1—H1A	0.93	N2—H2A	0.86
C6—C5	1.392 (2)	C9—H9A	0.96
C6—C7	1.4592 (19)	C9—H9B	0.96
C2—C3	1.4025 (19)	C9—H9C	0.96
C3—C4	1.377 (2)	O1W—H1E	0.85 (3)
C7—N1	1.2823 (19)	O1W—H1F	0.80 (3)

C3—O1—H1	109.5	N2—C8—C9	115.35 (12)
C2—O2—H2	109.5	C3—C4—C5	119.80 (13)
C2—C1—C6	120.24 (12)	C3—C4—H4	120.1
C2—C1—H1A	119.9	C5—C4—H4	120.1
C6—C1—H1A	119.9	C4—C5—C6	120.95 (13)
C5—C6—C1	118.93 (13)	C4—C5—H5	119.5
C5—C6—C7	118.82 (12)	C6—C5—H5	119.5
C1—C6—C7	122.25 (12)	C7—N1—N2	115.60 (11)
O2—C2—C1	125.46 (12)	C8—N2—N1	119.30 (11)
O2—C2—C3	114.74 (12)	C8—N2—H2A	120.3
C1—C2—C3	119.80 (12)	N1—N2—H2A	120.3
O1—C3—C4	119.15 (12)	C8—C9—H9A	109.5
O1—C3—C2	120.57 (12)	C8—C9—H9B	109.5
C4—C3—C2	120.28 (13)	H9A—C9—H9B	109.5
N1—C7—C6	122.05 (12)	C8—C9—H9C	109.5
N1—C7—H7	119.0	H9A—C9—H9C	109.5
C6—C7—H7	119.0	H9B—C9—H9C	109.5
O3—C8—N2	122.18 (13)	H1E—O1W—H1F	103 (2)
O3—C8—C9	122.47 (14)		

C2—C1—C6—C5	0.4 (2)	O1—C3—C4—C5	−178.86 (13)
C2—C1—C6—C7	179.90 (12)	C2—C3—C4—C5	0.7 (2)
C6—C1—C2—O2	−179.73 (12)	C3—C4—C5—C6	0.0 (2)
C6—C1—C2—C3	0.3 (2)	C1—C6—C5—C4	−0.5 (2)
O2—C2—C3—O1	−1.26 (18)	C7—C6—C5—C4	179.97 (13)
C1—C2—C3—O1	178.75 (12)	C6—C7—N1—N2	−178.62 (11)
O2—C2—C3—C4	179.15 (12)	O3—C8—N2—N1	2.4 (2)
C1—C2—C3—C4	−0.8 (2)	C9—C8—N2—N1	−177.75 (12)
C5—C6—C7—N1	179.01 (13)	C7—N1—N2—C8	173.80 (12)
C1—C6—C7—N1	−0.5 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.82	2.22	2.6694 (18)	115
O1—H1···O1*W*^{i}	0.82	2.11	2.8529 (18)	151
O1*W*—H1*F*···O3^{ii}	0.80 (3)	2.31 (3)	3.031 (2)	152 (3)
O1*W*—H1*F*···N1^{ii}	0.80 (3)	2.48 (3)	3.101 (2)	135 (2)
O2—H2···O1*W*	0.82	1.96	2.7736 (18)	171
N2—H2*A*···O3^{iii}	0.86	2.09	2.9110 (19)	160
C7—H7···O3^{iii}	0.93	2.53	3.311 (2)	142

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