Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-4-Hydroxy-*N*′-(2-hydroxy-4-methoxybenzylidene)benzohydrazide monohydrate

Nooraziah Mohd Lair, Hapipah Mohd Ali and Seik Weng Ng*

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

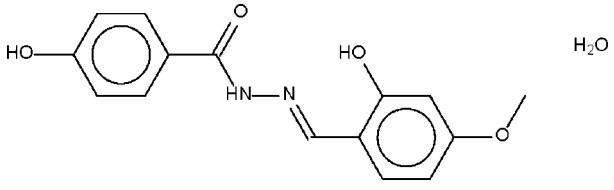
Received 15 December 2008; accepted 16 December 2008

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.060; wR factor = 0.180; data-to-parameter ratio = 16.6.

The Schiff base molecule of the title compound, $C_{15}H_{14}N_2O_4\cdot H_2O$, adopts a *trans* configuration with respect to the C=N double bond; the Schiff base itself is almost planar (r.m.s. deviation for all non-H atoms = 0.040 Å). The amido N atom is the hydrogen-bond donor to the water molecule, which is the hydrogen-bond donor to the hydroxy groups of two neighboring molecules. One of the hydroxyl groups acts as an intramolecular and the other as an intermolecular hydrogen-bond donor.

Related literature

For the structure of (*E*)-4-chloro-*N*′-(2-hydroxy-3-methoxybenzylidene)benzohydrazide, which crystallizes as a monohydrate, see: Cui *et al.* (2007). For a series of similar compounds, see: Lu *et al.* (2008*a*,*b*,*c*). For this and other compounds with antimalarial properties, see: Melnyk *et al.* (2006).



Experimental

Crystal data

$C_{15}H_{14}N_2O_4\cdot H_2O$
M_r = 304.30
Monoclinic, $P2_1/n$
a = 7.1763 (2) Å
b = 16.6507 (5) Å
c = 12.1828 (4) Å
β = 98.022 (2)°
V = 1441.48 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 100 (2) K
0.16 × 0.04 × 0.04 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: none
13327 measured reflections
3315 independent reflections
1903 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.180
S = 1.05
3315 reflections
200 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.84	1.86	2.621 (3)	150
O3—H3···N2	0.84	1.94	2.575 (3)	132
O5—H51···O1^{ii}	0.84	2.03	2.833 (3)	160
O5—H52···O3^{iii}	0.84	2.27	3.070 (4)	160
N1—H11···O5	0.88	2.05	2.883 (3)	158

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

We thank the University of Malaya for funding this study (Science Fund grants 12–02-03–2031, 12–02-03–2051).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2836).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cui, J., Yin, H. & Qiao, Y. (2007). *Acta Cryst.* E**63**, o3548.
Lu, J.-F., Min, S.-T., Ji, X.-H. & Dang, Z.-H. (2008*a*). *Acta Cryst.* E**64**, o1693.
Lu, J.-F., Min, S.-T., Ji, X.-H. & Dang, Z.-H. (2008*b*). *Acta Cryst.* E**64**, o1694.
Lu, J.-F., Min, S.-T., Ji, X.-H. & Dang, Z.-H. (2008*c*). *Acta Cryst.* E**64**, o1695.
Melnyk, P., Leroux, V., Sergheraert, C. & Grellier, P. (2006). *Bioorg. & Med. Chem. Lett.* **16**, 31–35.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2009). *publCIF*. In preparation.

supplementary materials

Acta Cryst. (2009). E**65**, o189 [**doi:10.1107/S1600536808042888**]

(*E*)-4-Hydroxy-*N*'-(2-hydroxy-4-methoxybenzylidene)benzohydrazide monohydrate

N. Mohd Lair, H. Mohd Ali and S. W. Ng

Comment

(type here to add)

Experimental

2-Hydroxy-3-methoxybenzaldehyde (0.30 g, 2 mmol) and 4-hydroxybenzohydrazide (0.30 g, 2 mmol) were heated in an ethanol-methanol mixture (50 ml) for 2 h. The solvent was removed and the resulting compound recrystallized from ethanol.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95–0.98 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2–1.5*U*(C). The oxygen- and nitrogen-bound ones were located in a difference Fourier map, and were refined with distance restraints (O–H 0.84±0.01, N–H 0.88±0.01 Å); their isotropic displacement parameters were freely refined.

Figures

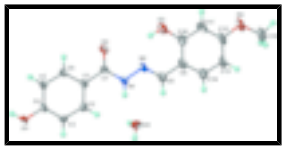


Fig. 1. Anisotropic displacement ellipsoid plot (Barbour, 2001) of $C_{15}H_{14}N_2O_4{\cdot}H_2O$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

(*E*)-4-Hydroxy-*N*'-(2-hydroxy-4-methoxybenzylidene)benzohydrazide monohydrate

Crystal data

$C_{15}H_{14}N_2O_4{\cdot}H_2O$	F_{000} = 640
M_r = 304.30	D_x = 1.402 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 1918 reflections
a = 7.1763 (2) Å	θ = 2.4–27.3°
b = 16.6507 (5) Å	μ = 0.11 mm^{-1}
c = 12.1828 (4) Å	T = 100 (2) K
β = 98.022 (2)°	Prism, yellow
V = 1441.48 (8) Å^3	0.16 × 0.04 × 0.04 mm
Z = 4	

Data collection

Bruker SMART APEX diffractometer	1903 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.053$
Monochromator: graphite	$\theta_{max} = 27.5^{o}$
$T = 100(2)$ K	$\theta_{min} = 2.1^{o}$
ω scans	$h = -9\rightarrow9$
Absorption correction: None	$k = -21\rightarrow21$
13327 measured reflections	$l = -15\rightarrow15$
3315 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.060$	H-atom parameters constrained
$wR(F^2) = 0.180$	$w = 1/[\sigma^2(F_o^2) + (0.0687P)^2 + 1.4764P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.001$
3315 reflections	$\Delta\rho_{max} = 0.66$ e Å^{-3}
200 parameters	$\Delta\rho_{min} = -0.43$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.7450 (3)	0.31702 (12)	1.09538 (15)	0.0271 (5)
H1	0.7745	0.2698	1.1150	0.041*
O2	0.3884 (3)	0.32742 (11)	0.58919 (15)	0.0246 (5)
O3	0.1758 (3)	0.41831 (13)	0.32215 (18)	0.0444 (6)
H3	0.2262	0.4067	0.3866	0.067*
O4	−0.1014 (3)	0.59721 (14)	0.05179 (16)	0.0359 (6)
O5	0.4249 (4)	0.61425 (14)	0.73043 (18)	0.0495 (7)
H51	0.4027	0.6363	0.7892	0.074*
H52	0.5420	0.6108	0.7317	0.074*
N1	0.3620 (3)	0.45875 (14)	0.62786 (18)	0.0208 (5)
H11	0.3800	0.4987	0.6754	0.025*
N2	0.2800 (3)	0.47111 (14)	0.51985 (17)	0.0218 (5)
C1	0.4997 (3)	0.36829 (15)	0.7739 (2)	0.0174 (5)
C2	0.5351 (4)	0.42869 (16)	0.8536 (2)	0.0204 (6)
H2	0.5012	0.4826	0.8345	0.024*
C3	0.6192 (4)	0.41082 (16)	0.9602 (2)	0.0216 (6)
H3A	0.6449	0.4524	1.0135	0.026*
C4	0.6653 (3)	0.33229 (16)	0.9886 (2)	0.0197 (6)

C5	0.6322 (4)	0.27144 (16)	0.9108 (2)	0.0214 (6)
H5A	0.6655	0.2176	0.9305	0.026*
C6	0.5504 (4)	0.28976 (16)	0.8043 (2)	0.0208 (6)
H6	0.5284	0.2481	0.7508	0.025*
C7	0.4134 (3)	0.38292 (16)	0.6579 (2)	0.0191 (6)
C8	0.2221 (4)	0.54217 (18)	0.4913 (2)	0.0231 (6)
H8	0.2362	0.5848	0.5436	0.028*
C9	0.1350 (4)	0.55631 (18)	0.3780 (2)	0.0235 (6)
C10	0.1117 (4)	0.49462 (18)	0.2982 (2)	0.0289 (7)
C11	0.0295 (4)	0.5107 (2)	0.1905 (2)	0.0329 (7)
H11A	0.0125	0.4689	0.1371	0.040*
C12	−0.0278 (4)	0.5882 (2)	0.1614 (2)	0.0281 (7)
C13	−0.0097 (4)	0.64983 (19)	0.2380 (2)	0.0290 (7)
H13	−0.0519	0.7025	0.2177	0.035*
C14	0.0714 (4)	0.63284 (18)	0.3450 (2)	0.0270 (6)
H14	0.0843	0.6749	0.3982	0.032*
C15	−0.1581 (4)	0.6759 (2)	0.0151 (3)	0.0385 (8)
H15A	−0.2026	0.6749	−0.0647	0.058*
H15B	−0.0508	0.7127	0.0299	0.058*
H15C	−0.2598	0.6944	0.0549	0.058*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0379 (11)	0.0266 (11)	0.0146 (9)	0.0096 (9)	−0.0039 (8)	0.0015 (8)
O2	0.0309 (10)	0.0230 (10)	0.0188 (10)	−0.0048 (8)	−0.0008 (8)	−0.0005 (8)
O3	0.0701 (17)	0.0318 (13)	0.0264 (12)	−0.0012 (12)	−0.0107 (11)	0.0029 (10)
O4	0.0320 (12)	0.0538 (15)	0.0203 (10)	0.0041 (10)	−0.0018 (9)	0.0118 (10)
O5	0.0772 (18)	0.0431 (14)	0.0259 (12)	0.0265 (13)	−0.0009 (12)	−0.0119 (11)
N1	0.0228 (12)	0.0233 (12)	0.0152 (11)	0.0005 (9)	−0.0016 (9)	0.0003 (9)
N2	0.0209 (11)	0.0306 (13)	0.0132 (11)	−0.0014 (10)	0.0004 (9)	0.0039 (9)
C1	0.0136 (12)	0.0223 (13)	0.0164 (13)	−0.0033 (10)	0.0020 (10)	−0.0001 (11)
C2	0.0201 (13)	0.0213 (14)	0.0197 (13)	0.0023 (10)	0.0027 (10)	0.0031 (11)
C3	0.0260 (14)	0.0207 (14)	0.0172 (13)	0.0012 (11)	0.0001 (10)	−0.0035 (11)
C4	0.0192 (13)	0.0249 (14)	0.0144 (12)	0.0032 (11)	0.0007 (10)	0.0034 (11)
C5	0.0221 (13)	0.0201 (14)	0.0214 (13)	0.0022 (11)	0.0006 (11)	0.0011 (11)
C6	0.0217 (13)	0.0228 (14)	0.0170 (13)	−0.0005 (11)	−0.0004 (11)	−0.0033 (11)
C7	0.0173 (13)	0.0225 (14)	0.0176 (13)	−0.0027 (10)	0.0029 (10)	0.0000 (11)
C8	0.0197 (13)	0.0303 (15)	0.0195 (14)	0.0004 (11)	0.0035 (11)	0.0013 (12)
C9	0.0184 (13)	0.0327 (16)	0.0198 (14)	−0.0033 (11)	0.0043 (11)	0.0066 (12)
C10	0.0333 (16)	0.0286 (16)	0.0242 (14)	−0.0018 (13)	0.0013 (12)	0.0074 (13)
C11	0.0371 (17)	0.0391 (18)	0.0211 (15)	−0.0053 (14)	−0.0013 (12)	0.0008 (13)
C12	0.0192 (14)	0.0471 (19)	0.0177 (14)	−0.0008 (13)	0.0011 (11)	0.0108 (13)
C13	0.0237 (14)	0.0358 (17)	0.0269 (16)	0.0055 (12)	0.0018 (12)	0.0123 (13)
C14	0.0235 (14)	0.0327 (16)	0.0249 (15)	0.0039 (12)	0.0043 (12)	0.0033 (12)
C15	0.0314 (17)	0.057 (2)	0.0263 (16)	0.0062 (16)	0.0018 (13)	0.0173 (16)

Geometric parameters (Å, °)

O1—C4	1.370 (3)	C3—H3A	0.9500
O1—H1	0.8400	C4—C5	1.385 (4)
O2—C7	1.243 (3)	C5—C6	1.382 (4)
O3—C10	1.369 (4)	C5—H5A	0.9500
O3—H3	0.8400	C6—H6	0.9500
O4—C12	1.374 (3)	C8—C9	1.452 (4)
O4—C15	1.426 (4)	C8—H8	0.9500
O5—H51	0.8400	C9—C14	1.394 (4)
O5—H52	0.8400	C9—C10	1.408 (4)
N1—C7	1.351 (3)	C10—C11	1.387 (4)
N1—N2	1.380 (3)	C11—C12	1.385 (4)
N1—H11	0.8800	C11—H11A	0.9500
N2—C8	1.286 (4)	C12—C13	1.381 (4)
C1—C6	1.393 (4)	C13—C14	1.381 (4)
C1—C2	1.396 (4)	C13—H13	0.9500
C1—C7	1.482 (3)	C14—H14	0.9500
C2—C3	1.386 (4)	C15—H15A	0.9800
C2—H2	0.9500	C15—H15B	0.9800
C3—C4	1.381 (4)	C15—H15C	0.9800
C4—O1—H1	119.9	N1—C7—C1	118.3 (2)
C10—O3—H3	120.0	N2—C8—C9	119.1 (3)
C12—O4—C15	117.3 (3)	N2—C8—H8	120.5
H51—O5—H52	108.8	C9—C8—H8	120.5
C7—N1—N2	117.5 (2)	C14—C9—C10	117.7 (3)
C7—N1—H11	121.3	C14—C9—C8	120.2 (3)
N2—N1—H11	121.3	C10—C9—C8	122.1 (3)
C8—N2—N1	118.3 (2)	O3—C10—C11	117.8 (3)
C6—C1—C2	118.4 (2)	O3—C10—C9	121.7 (3)
C6—C1—C7	117.8 (2)	C11—C10—C9	120.4 (3)
C2—C1—C7	123.8 (2)	C12—C11—C10	119.6 (3)
C3—C2—C1	120.7 (2)	C12—C11—H11A	120.2
C3—C2—H2	119.7	C10—C11—H11A	120.2
C1—C2—H2	119.7	O4—C12—C13	124.3 (3)
C4—C3—C2	119.8 (2)	O4—C12—C11	114.2 (3)
C4—C3—H3A	120.1	C13—C12—C11	121.5 (3)
C2—C3—H3A	120.1	C12—C13—C14	118.3 (3)
O1—C4—C3	117.9 (2)	C12—C13—H13	120.9
O1—C4—C5	121.5 (2)	C14—C13—H13	120.9
C3—C4—C5	120.5 (2)	C13—C14—C9	122.5 (3)
C6—C5—C4	119.4 (2)	C13—C14—H14	118.7
C6—C5—H5A	120.3	C9—C14—H14	118.7
C4—C5—H5A	120.3	O4—C15—H15A	109.5
C5—C6—C1	121.2 (2)	O4—C15—H15B	109.5
C5—C6—H6	119.4	H15A—C15—H15B	109.5
C1—C6—H6	119.4	O4—C15—H15C	109.5
O2—C7—N1	120.3 (2)	H15A—C15—H15C	109.5

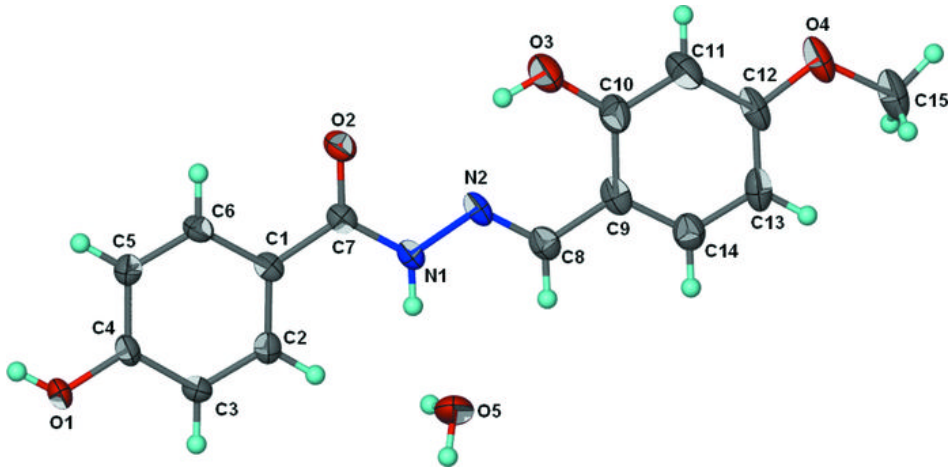
O2—C7—C1	121.4 (2)	H15B—C15—H15C	109.5
C7—N1—N2—C8	176.8 (2)	N2—C8—C9—C14	−179.9 (3)
C6—C1—C2—C3	0.2 (4)	N2—C8—C9—C10	0.0 (4)
C7—C1—C2—C3	−178.9 (2)	C14—C9—C10—O3	177.7 (3)
C1—C2—C3—C4	−1.2 (4)	C8—C9—C10—O3	−2.2 (4)
C2—C3—C4—O1	−178.8 (2)	C14—C9—C10—C11	0.6 (4)
C2—C3—C4—C5	1.5 (4)	C8—C9—C10—C11	−179.3 (3)
O1—C4—C5—C6	179.6 (2)	O3—C10—C11—C12	−176.4 (3)
C3—C4—C5—C6	−0.7 (4)	C9—C10—C11—C12	0.8 (4)
C4—C5—C6—C1	−0.3 (4)	C15—O4—C12—C13	1.6 (4)
C2—C1—C6—C5	0.6 (4)	C15—O4—C12—C11	−178.2 (3)
C7—C1—C6—C5	179.7 (2)	C10—C11—C12—O4	178.0 (3)
N2—N1—C7—O2	0.7 (4)	C10—C11—C12—C13	−1.8 (4)
N2—N1—C7—C1	−179.3 (2)	O4—C12—C13—C14	−178.4 (3)
C6—C1—C7—O2	−0.7 (4)	C11—C12—C13—C14	1.4 (4)
C2—C1—C7—O2	178.4 (2)	C12—C13—C14—C9	0.0 (4)
C6—C1—C7—N1	179.3 (2)	C10—C9—C14—C13	−1.0 (4)
C2—C1—C7—N1	−1.6 (4)	C8—C9—C14—C13	178.9 (3)
N1—N2—C8—C9	−179.8 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.84	1.86	2.621 (3)	150
O3—H3···N2	0.84	1.94	2.575 (3)	132
O5—H51···O1^{ii}	0.84	2.03	2.833 (3)	160
O5—H52···O3^{iii}	0.84	2.27	3.070 (4)	160
N1—H11···O5	0.88	2.05	2.883 (3)	158

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

Fig. 1


O3
C11
O4
C10
C12
C15
O2
N2
C6
C1
C7
C9
C13
C5
N1
C8
C14
C4
C2
O1
C3
O5