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3,4,5-Trihydroxy-*N*′-(2-hydroxy-5-nitrobenzylidene)benzohydrazide monohydrate

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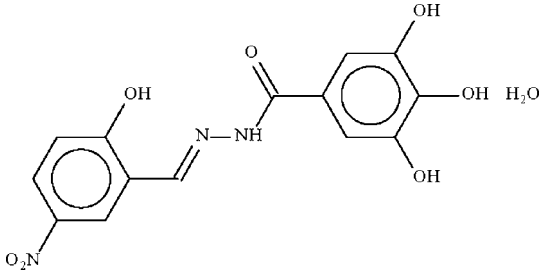
Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.112; data-to-parameter ratio = 12.6.

The benzohydrazide molecule of the title compound, $C_{14}H_{11}N_3O_7\cdot H_2O$, is planar (r.m.s. deviation = 0.068 Å). The benzohydrazide molecule and the uncoordinated water molecule interact through O—H···O hydrogen bonds; these together with O—H···N and N—H···O hydrogen bonds form a three-dimensional network.

Related literature

For the the parent *N*′-(2-hydroxybenzylidene)benzohydrazide, see: Lyubchova *et al.* (1995). For other *N*′-(2-hydroxy-5-nitrobenzylidene)benzohydrazides, see: Ali *et al.* (2005); Lyubchova *et al.* (1995); Xu & Liu (2006).



Experimental

Crystal data

$C_{14}H_{11}N_3O_7\cdot H_2O$
M_r = 351.27
Triclinic, $P\overline{1}$
a = 7.0097 (2) Å
b = 7.8380 (2) Å
c = 13.2953 (3) Å
α = 75.597 (1)°
β = 88.826 (2)°
γ = 81.929 (2)°
V = 700.42 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 123 K
0.15 × 0.10 × 0.02 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: none
6629 measured reflections
3209 independent reflections
2373 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.112
S = 1.03
3209 reflections
254 parameters
7 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N2	0.84 (1)	1.81 (1)	2.581 (2)	152 (2)
O5—H5···O6^{i}	0.84 (1)	2.16 (2)	2.847 (2)	139 (2)
O6—H6···O1w^{ii}	0.84 (1)	1.81 (1)	2.630 (2)	162 (2)
O7—H7···O4^{iii}	0.84 (1)	1.87 (1)	2.715 (2)	177 (2)
O1w—H11···O5	0.84 (1)	2.13 (1)	2.918 (2)	156 (2)
O1w—H12···O1^{iv}	0.84 (1)	2.13 (1)	2.962 (2)	169 (2)
N3—H3···O3^{v}	0.88 (1)	2.07 (1)	2.890 (2)	155 (2)

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); 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 supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2399).

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supporting information

Acta Cryst. (2009). E**65**, o908 [doi:10.1107/S1600536809010563]

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Abeer A. Abdul Alhadi, Hapipah Mohd. Ali and Seik Weng Ng

S1. Experimental

5-Nitro-2-hydroxybenzaldehyde (0.33 g, 2 mmol) and 3,4,5-trihydroxybenzoylhydrazide (0.36 g, 2 mmol) were heated in ethanol (50 ml) for several hours. The solvent was removed and the product recrystallized from DMSO.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 Å, U(H) = 1.2U(C)], and were included in the refinement in the riding model approximation.

The amino and hydroxy H-atoms were located in a difference Fourier map, and were refined with distance restraints of N–H 0.88±0.01 Å and O–H 0.84±0.01 Å, respectively; their temperature factors were refined isotropically.

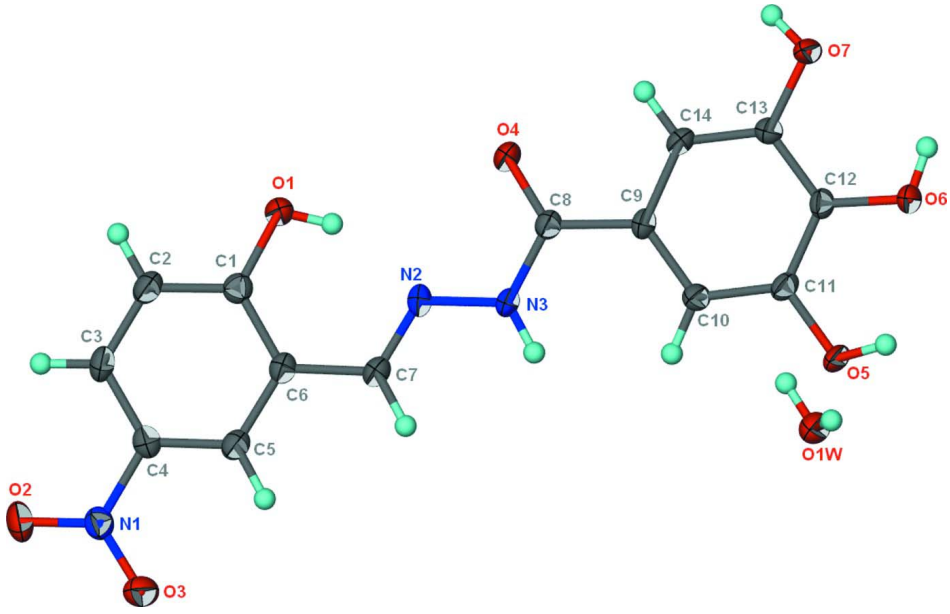

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{14}H_{11}N_3O_7{\cdot}H_2O$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

3,4,5-Trihydroxy-*N*′-(2-hydroxy-5-nitrobenzylidene)benzohydrazide monohydrate

Crystal data

$C_{14}H_{11}N_3O_7 \cdot H_2O$
$M_r = 351.27$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.0097$ (2) Å
$b = 7.8380$ (2) Å
$c = 13.2953$ (3) Å
$\alpha = 75.597$ (1)°
$\beta = 88.826$ (2)°
$\gamma = 81.929$ (2)°
$V = 700.42$ (3) Å^3
$Z = 2$
$F(000) = 364$
$D_x = 1.666$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2084 reflections
$\theta = 2.7$–$28.3°$
$\mu = 0.14$ mm^{-1}
$T = 123$ K
Plate, yellow
0.15 × 0.10 × 0.02 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
6629 measured reflections
3209 independent reflections
2373 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.6°$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -17 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.112$
$S = 1.03$
3209 reflections
254 parameters
7 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
$w = 1/[\sigma^2(F_o^2) + (0.0583P)^2 + 0.1907P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.45792 (16)	0.65420 (15)	0.74975 (9)	0.0199 (3)
O2	−0.28084 (18)	0.28619 (17)	0.85473 (9)	0.0280 (3)
O3	−0.29787 (16)	0.35460 (16)	0.68665 (9)	0.0235 (3)
O4	0.75724 (17)	0.84619 (16)	0.53567 (9)	0.0241 (3)
O5	0.70666 (16)	1.00100 (17)	0.07165 (8)	0.0216 (3)
O6	1.05293 (16)	1.11263 (16)	0.08126 (8)	0.0195 (3)
O7	1.21628 (16)	1.10003 (17)	0.27137 (9)	0.0216 (3)
O1W	0.36719 (17)	1.26409 (18)	0.06982 (9)	0.0236 (3)
N1	−0.21856 (19)	0.35371 (18)	0.76939 (10)	0.0188 (3)
N2	0.43352 (19)	0.71596 (18)	0.55014 (10)	0.0174 (3)
N3	0.50987 (19)	0.77875 (19)	0.45456 (10)	0.0182 (3)
C1	0.2933 (2)	0.5835 (2)	0.75146 (12)	0.0170 (3)

C2	0.2175 (2)	0.5116 (2)	0.84825 (12)	0.0206 (3)
H2	0.2824	0.5136	0.9099	0.025*
C3	0.0492 (2)	0.4379 (2)	0.85482 (12)	0.0196 (3)
H3A	−0.0027	0.3890	0.9206	0.024*
C4	−0.0440 (2)	0.4361 (2)	0.76366 (12)	0.0173 (3)
C5	0.0277 (2)	0.5064 (2)	0.66704 (12)	0.0172 (3)
H5A	−0.0392	0.5042	0.6060	0.021*
C6	0.1980 (2)	0.5805 (2)	0.65926 (12)	0.0166 (3)
C7	0.2744 (2)	0.6509 (2)	0.55666 (12)	0.0179 (3)
H7A	0.2075	0.6485	0.4957	0.021*
C8	0.6794 (2)	0.8441 (2)	0.45388 (12)	0.0158 (3)
C9	0.7687 (2)	0.9115 (2)	0.35203 (11)	0.0152 (3)
C10	0.6888 (2)	0.9198 (2)	0.25525 (12)	0.0157 (3)
H10	0.5683	0.8798	0.2504	0.019*
C11	0.7882 (2)	0.9876 (2)	0.16644 (11)	0.0160 (3)
C12	0.9662 (2)	1.0473 (2)	0.17210 (12)	0.0154 (3)
C13	1.0437 (2)	1.0389 (2)	0.26925 (12)	0.0159 (3)
C14	0.9458 (2)	0.9706 (2)	0.35795 (12)	0.0164 (3)
H14	0.9998	0.9637	0.4240	0.020*
H1	0.482 (3)	0.691 (3)	0.6866 (9)	0.043 (7)*
H5	0.789 (3)	1.020 (3)	0.0256 (16)	0.060 (8)*
H6	1.151 (2)	1.158 (3)	0.0912 (18)	0.045 (7)*
H7	1.228 (3)	1.117 (3)	0.3308 (10)	0.045 (7)*
H11	0.452 (3)	1.191 (3)	0.0523 (19)	0.047 (7)*
H12	0.408 (3)	1.278 (3)	0.1260 (12)	0.055 (8)*
H3	0.453 (3)	0.769 (2)	0.3981 (10)	0.022 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0212 (6)	0.0250 (6)	0.0149 (6)	−0.0097 (5)	−0.0003 (5)	−0.0041 (5)
O2	0.0256 (6)	0.0346 (7)	0.0205 (6)	−0.0096 (5)	0.0064 (5)	0.0017 (5)
O3	0.0212 (6)	0.0307 (7)	0.0219 (6)	−0.0077 (5)	0.0003 (5)	−0.0102 (5)
O4	0.0268 (6)	0.0361 (7)	0.0116 (5)	−0.0148 (5)	0.0000 (5)	−0.0040 (5)
O5	0.0172 (6)	0.0399 (7)	0.0094 (5)	−0.0089 (5)	0.0009 (4)	−0.0067 (5)
O6	0.0184 (6)	0.0301 (7)	0.0117 (5)	−0.0098 (5)	0.0032 (4)	−0.0053 (5)
O7	0.0186 (6)	0.0368 (7)	0.0134 (6)	−0.0132 (5)	0.0016 (5)	−0.0087 (5)
O1W	0.0215 (6)	0.0334 (7)	0.0169 (6)	−0.0077 (5)	0.0004 (5)	−0.0060 (5)
N1	0.0174 (7)	0.0206 (7)	0.0181 (7)	−0.0030 (5)	0.0028 (5)	−0.0041 (6)
N2	0.0194 (7)	0.0200 (7)	0.0123 (6)	−0.0046 (5)	0.0033 (5)	−0.0023 (5)
N3	0.0186 (7)	0.0271 (8)	0.0095 (6)	−0.0077 (6)	0.0015 (5)	−0.0034 (5)
C1	0.0184 (7)	0.0166 (8)	0.0163 (8)	−0.0028 (6)	0.0003 (6)	−0.0045 (6)
C2	0.0251 (8)	0.0233 (9)	0.0133 (8)	−0.0045 (7)	−0.0013 (6)	−0.0038 (6)
C3	0.0241 (8)	0.0213 (8)	0.0124 (7)	−0.0042 (7)	0.0032 (6)	−0.0016 (6)
C4	0.0176 (7)	0.0166 (8)	0.0173 (8)	−0.0031 (6)	0.0027 (6)	−0.0036 (6)
C5	0.0185 (7)	0.0192 (8)	0.0135 (7)	−0.0019 (6)	−0.0007 (6)	−0.0036 (6)
C6	0.0181 (8)	0.0178 (8)	0.0134 (7)	−0.0024 (6)	0.0009 (6)	−0.0032 (6)
C7	0.0202 (8)	0.0215 (8)	0.0124 (7)	−0.0043 (6)	−0.0001 (6)	−0.0044 (6)

C8	0.0177 (7)	0.0169 (8)	0.0127 (7)	−0.0029 (6)	0.0005 (6)	−0.0034 (6)
C9	0.0174 (7)	0.0170 (8)	0.0110 (7)	−0.0024 (6)	0.0022 (6)	−0.0034 (6)
C10	0.0131 (7)	0.0202 (8)	0.0145 (7)	−0.0031 (6)	0.0008 (6)	−0.0051 (6)
C11	0.0166 (7)	0.0207 (8)	0.0112 (7)	−0.0014 (6)	−0.0002 (6)	−0.0056 (6)
C12	0.0154 (7)	0.0183 (8)	0.0121 (7)	−0.0024 (6)	0.0028 (6)	−0.0034 (6)
C13	0.0143 (7)	0.0196 (8)	0.0147 (7)	−0.0045 (6)	0.0004 (6)	−0.0048 (6)
C14	0.0185 (7)	0.0198 (8)	0.0116 (7)	−0.0039 (6)	−0.0018 (6)	−0.0043 (6)

Geometric parameters (Å, º)

O1—C1	1.3456 (19)	C1—C6	1.415 (2)
O1—H1	0.840 (10)	C2—C3	1.377 (2)
O2—N1	1.2263 (17)	C2—H2	0.9500
O3—N1	1.2408 (17)	C3—C4	1.393 (2)
O4—C8	1.2313 (18)	C3—H3A	0.9500
O5—C11	1.3684 (18)	C4—C5	1.380 (2)
O5—H5	0.835 (10)	C5—C6	1.391 (2)
O6—C12	1.3567 (18)	C5—H5A	0.9500
O6—H6	0.844 (10)	C6—C7	1.460 (2)
O7—C13	1.3654 (18)	C7—H7A	0.9500
O7—H7	0.842 (10)	C8—C9	1.486 (2)
O1W—H11	0.837 (10)	C9—C14	1.394 (2)
O1W—H12	0.843 (10)	C9—C10	1.397 (2)
N1—C4	1.454 (2)	C10—C11	1.386 (2)
N2—C7	1.283 (2)	C10—H10	0.9500
N2—N3	1.3705 (18)	C11—C12	1.402 (2)
N3—C8	1.358 (2)	C12—C13	1.394 (2)
N3—H3	0.882 (9)	C13—C14	1.380 (2)
C1—C2	1.397 (2)	C14—H14	0.9500
C1—O1—H1	105.4 (16)	C5—C6—C1	118.80 (14)
C11—O5—H5	109.5 (19)	C5—C6—C7	119.23 (14)
C12—O6—H6	111.2 (16)	C1—C6—C7	121.96 (14)
C13—O7—H7	106.8 (16)	N2—C7—C6	118.88 (14)
H11—O1W—H12	105 (2)	N2—C7—H7A	120.6
O2—N1—O3	122.81 (13)	C6—C7—H7A	120.6
O2—N1—C4	119.27 (13)	O4—C8—N3	120.72 (14)
O3—N1—C4	117.92 (13)	O4—C8—C9	121.07 (14)
C7—N2—N3	119.72 (13)	N3—C8—C9	118.21 (13)
C8—N3—N2	116.36 (13)	C14—C9—C10	119.94 (13)
C8—N3—H3	123.5 (13)	C14—C9—C8	114.80 (13)
N2—N3—H3	120.0 (13)	C10—C9—C8	125.25 (14)
O1—C1—C2	117.78 (14)	C11—C10—C9	118.85 (14)
O1—C1—C6	122.00 (14)	C11—C10—H10	120.6
C2—C1—C6	120.21 (14)	C9—C10—H10	120.6
C3—C2—C1	120.36 (15)	O5—C11—C10	118.74 (13)
C3—C2—H2	119.8	O5—C11—C12	119.86 (13)
C1—C2—H2	119.8	C10—C11—C12	121.36 (14)

C2—C3—C4	119.02 (14)	O6—C12—C13	123.45 (13)
C2—C3—H3A	120.5	O6—C12—C11	117.42 (13)
C4—C3—H3A	120.5	C13—C12—C11	119.13 (13)
C5—C4—C3	121.78 (14)	O7—C13—C14	122.96 (14)
C5—C4—N1	118.55 (14)	O7—C13—C12	117.31 (13)
C3—C4—N1	119.64 (14)	C14—C13—C12	119.73 (14)
C4—C5—C6	119.81 (14)	C13—C14—C9	120.99 (14)
C4—C5—H5A	120.1	C13—C14—H14	119.5
C6—C5—H5A	120.1	C9—C14—H14	119.5
C7—N2—N3—C8	−179.41 (15)	N2—N3—C8—C9	179.23 (13)
O1—C1—C2—C3	179.85 (15)	O4—C8—C9—C14	1.7 (2)
C6—C1—C2—C3	−0.3 (2)	N3—C8—C9—C14	−177.85 (14)
C1—C2—C3—C4	0.0 (3)	O4—C8—C9—C10	−177.86 (16)
C2—C3—C4—C5	−0.1 (3)	N3—C8—C9—C10	2.6 (2)
C2—C3—C4—N1	178.09 (15)	C14—C9—C10—C11	−0.1 (2)
O2—N1—C4—C5	177.95 (14)	C8—C9—C10—C11	179.45 (15)
O3—N1—C4—C5	−1.8 (2)	C9—C10—C11—O5	−177.89 (14)
O2—N1—C4—C3	−0.3 (2)	C9—C10—C11—C12	0.0 (2)
O3—N1—C4—C3	179.95 (14)	O5—C11—C12—O6	−1.7 (2)
C3—C4—C5—C6	0.4 (2)	C10—C11—C12—O6	−179.59 (14)
N1—C4—C5—C6	−177.79 (14)	O5—C11—C12—C13	177.58 (14)
C4—C5—C6—C1	−0.7 (2)	C10—C11—C12—C13	−0.3 (2)
C4—C5—C6—C7	178.65 (14)	O6—C12—C13—O7	−0.1 (2)
O1—C1—C6—C5	−179.54 (14)	C11—C12—C13—O7	−179.35 (14)
C2—C1—C6—C5	0.6 (2)	O6—C12—C13—C14	179.91 (15)
O1—C1—C6—C7	1.2 (2)	C11—C12—C13—C14	0.7 (2)
C2—C1—C6—C7	−178.71 (15)	O7—C13—C14—C9	179.26 (15)
N3—N2—C7—C6	178.44 (14)	C12—C13—C14—C9	−0.8 (2)
C5—C6—C7—N2	−178.70 (15)	C10—C9—C14—C13	0.5 (2)
C1—C6—C7—N2	0.6 (2)	C8—C9—C14—C13	−179.11 (14)
N2—N3—C8—O4	−0.3 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N2	0.84 (1)	1.81 (1)	2.581 (2)	152 (2)
O5—H5···O6^{i}	0.84 (1)	2.16 (2)	2.847 (2)	139 (2)
O6—H6···O1w^{ii}	0.84 (1)	1.81 (1)	2.630 (2)	162 (2)
O7—H7···O4^{iii}	0.84 (1)	1.87 (1)	2.715 (2)	177 (2)
O1w—H11···O5	0.84 (1)	2.13 (1)	2.918 (2)	156 (2)
O1w—H12···O1^{iv}	0.84 (1)	2.13 (1)	2.962 (2)	169 (2)
N3—H3···O3^{v}	0.88 (1)	2.07 (1)	2.890 (2)	155 (2)

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