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N′-(5-Chloro-2-hydroxybenzylidene)-3,4,5-trihydroxybenzohydrazide dihydrate

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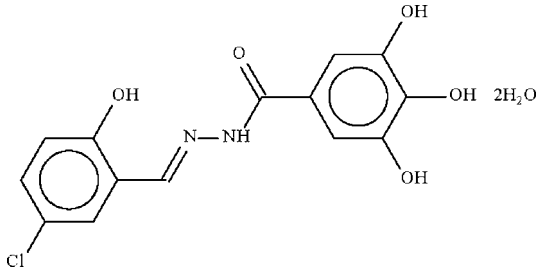
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.007 Å; R factor = 0.071; wR factor = 0.181; data-to-parameter ratio = 11.9.

The benzohydrazide molecule in the title dihydrate, $C_{14}H_{11}ClN_2O_5\cdot 2H_2O$, is non-planar, with the two aromatic rings at either side of the –C(=O)—NH—N=CH– unit forming a dihedral angle of 29.7 (2)°. The benzohydrazide molecule is linked to the water molecules by O—H···O and N—H···O hydrogen bonds, with other O—H···O hydrogen bonds leading to a layer structure.

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

$C_{14}H_{11}ClN_2O_5\cdot 2H_2O$
M_r = 358.73
Monoclinic, $P2_1/c$
a = 30.5627 (12) Å
b = 3.7539 (2) Å
c = 12.8882 (5) Å
β = 90.450 (3)°
V = 1478.61 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 100 K
0.36 × 0.04 × 0.04 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.899, T_{max} = 0.988
10104 measured reflections
2623 independent reflections
1801 reflections with $I > 2\sigma(I)$
R_{int} = 0.097

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.181
S = 1.07
2623 reflections
221 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.84	1.89	2.631 (5)	146
O3—H3···O1w^{i}	0.84	1.96	2.737 (6)	153
O4—H4···O1w^{ii}	0.84	1.80	2.599 (7)	158
O5—H5···O2^{ii}	0.84	1.93	2.765 (5)	171
O1w—H11···O3	0.83	2.28	2.969 (6)	140
O1w—H12···O4^{iii}	0.84	2.07	2.900 (7)	170
O2w—H21···O1	0.84	2.15	2.946 (5)	157
O2w—H22···O2^{iv}	0.84	1.97	2.808 (5)	172
N2—H2···O2w^{ii}	0.88	2.03	2.882 (5)	162

Symmetry codes: (i) $-x+2, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (iii) $x, -y+\frac{5}{2}, z+\frac{1}{2}$; (iv) $x, y-1, z$.

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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2403).

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supplementary materials

Acta Cryst. (2009). E**65**, o910 [**doi:10.1107/S1600536809010812**]

N'-(5-Chloro-2-hydroxybenzylidene)-3,4,5-trihydroxybenzohydrazide dihydrate

A. A. Abdul Alhadi, H. M. Ali and S. W. Ng

Comment

(type here to add)

Experimental

5-Chloro-2-hydroxybenzaldehyde (0.31 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.

Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 Å, *U*(H) = 1.2*U*(C)], and were included in the refinement in the riding model approximation. The amino (0.88 Å) and hydroxy H-atoms (0.84 Å) were similarly generated with U_{iso} = 1.2U_{eq}(carrier atom) for N-H and U_{iso} = 1.5U_{eq}(carrier atom) for O-H. The water H-atoms were placed in chemically sensible positions on the basis of possible hydrogen bonds, but were not refined; U_{iso} = 1.5U_{eq}(O).

Figures

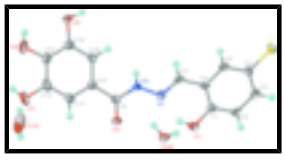


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{14}H_{11}ClN_2O_5{\cdot}2H_2O$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

N'-(5-Chloro-2-hydroxybenzylidene)-3,4,5-trihydroxybenzohydrazide dihydrate

Crystal data

$C_{14}H_{11}ClN_2O_5{\cdot}2H_2O$	F_{000} = 744
M_r = 358.73	D_x = 1.611 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1505 reflections
a = 30.5627 (12) Å	θ = 2.7–24.3°
b = 3.7539 (2) Å	μ = 0.30 mm^{-1}
c = 12.8882 (5) Å	T = 100 K
β = 90.450 (3)°	Prism, yellow
V = 1478.61 (11) Å^3	0.36 × 0.04 × 0.04 mm
Z = 4	

Data collection

Bruker SMART APEX diffractometer	2623 independent reflections
Radiation source: fine-focus sealed tube	1801 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.097$
$T = 100$ K	$\theta_{max} = 25.0^{o}$
ω scans	$\theta_{min} = 0.7^{o}$
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)	$h = -36\rightarrow36$
$T_{min} = 0.899$, $T_{max} = 0.988$	$k = -4\rightarrow4$
10104 measured reflections	$l = -15\rightarrow14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.071$	H-atom parameters constrained
$wR(F^2) = 0.181$	$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 5.5995P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} = 0.001$
2623 reflections	$\Delta\rho_{max} = 0.38$ e Å^{-3}
221 parameters	$\Delta\rho_{min} = -0.49$ 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}
Cl1	0.53161 (4)	0.3168 (4)	0.59750 (10)	0.0244 (3)
O1	0.67086 (11)	0.6471 (10)	0.8994 (2)	0.0243 (9)
H1	0.6933	0.7236	0.8694	0.036*
O2	0.79657 (11)	0.9733 (10)	0.8420 (2)	0.0232 (9)
O3	0.94472 (12)	1.3696 (12)	0.7043 (3)	0.0349 (10)
H3	0.9663	1.3279	0.6661	0.052*
O4	0.94729 (12)	1.0933 (13)	0.5106 (3)	0.0395 (12)
H4	0.9481	0.9332	0.4651	0.059*
O5	0.87480 (11)	0.7706 (11)	0.4244 (2)	0.0245 (9)
H5	0.8498	0.7191	0.4013	0.037*
O1w	0.97110 (13)	0.8480 (13)	0.8663 (4)	0.0542 (14)
H11	0.9676	0.9037	0.8040	0.081*
H12	0.9666	1.0253	0.9042	0.081*
O2w	0.73755 (11)	0.1847 (10)	0.9961 (2)	0.0239 (8)
H21	0.7141	0.2633	0.9700	0.036*
H22	0.7554	0.1438	0.9482	0.036*
N1	0.71793 (13)	0.8677 (12)	0.7419 (3)	0.0194 (10)

N2	0.75565 (13)	0.9864 (12)	0.6939 (3)	0.0187 (9)
H2	0.7545	1.0529	0.6284	0.022*
C1	0.63896 (16)	0.5781 (13)	0.8275 (4)	0.0179 (11)
C2	0.60017 (17)	0.4266 (14)	0.8603 (4)	0.0209 (12)
H2A	0.5965	0.3740	0.9318	0.025*
C3	0.56699 (17)	0.3513 (14)	0.7916 (4)	0.0233 (12)
H3A	0.5403	0.2508	0.8151	0.028*
C4	0.57295 (16)	0.4243 (13)	0.6865 (4)	0.0189 (11)
C5	0.61125 (16)	0.5725 (13)	0.6514 (4)	0.0193 (11)
H5A	0.6148	0.6213	0.5797	0.023*
C6	0.64514 (16)	0.6516 (14)	0.7219 (4)	0.0179 (11)
C7	0.68550 (15)	0.7976 (13)	0.6815 (4)	0.0172 (11)
H7	0.6880	0.8421	0.6092	0.021*
C9	0.79416 (16)	1.0018 (14)	0.7457 (4)	0.0181 (11)
C10	0.83308 (16)	1.0434 (14)	0.6800 (4)	0.0183 (11)
C11	0.87050 (16)	1.1993 (15)	0.7207 (4)	0.0214 (12)
H11A	0.8704	1.2939	0.7890	0.026*
C12	0.90791 (16)	1.2173 (16)	0.6620 (4)	0.0266 (13)
C13	0.90870 (16)	1.0734 (15)	0.5623 (4)	0.0241 (13)
C14	0.87108 (16)	0.9157 (15)	0.5212 (4)	0.0219 (12)
C15	0.83293 (16)	0.9046 (14)	0.5784 (4)	0.0189 (11)
H15	0.8070	0.8046	0.5496	0.023*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0204 (7)	0.0272 (7)	0.0257 (7)	−0.0018 (6)	−0.0025 (5)	−0.0039 (6)
O1	0.026 (2)	0.031 (2)	0.0151 (17)	−0.0024 (18)	−0.0023 (15)	−0.0001 (17)
O2	0.0211 (19)	0.036 (2)	0.0122 (18)	0.0034 (17)	−0.0014 (14)	−0.0031 (16)
O3	0.022 (2)	0.049 (3)	0.034 (2)	−0.014 (2)	−0.0055 (17)	0.006 (2)
O4	0.021 (2)	0.070 (4)	0.028 (2)	−0.009 (2)	0.0042 (17)	0.011 (2)
O5	0.0199 (18)	0.042 (2)	0.0118 (17)	−0.0017 (18)	0.0023 (14)	−0.0004 (17)
O1w	0.027 (2)	0.056 (3)	0.079 (3)	0.013 (2)	0.012 (2)	0.017 (3)
O2w	0.0223 (18)	0.035 (2)	0.0139 (17)	0.0042 (17)	−0.0002 (14)	−0.0012 (17)
N1	0.015 (2)	0.024 (2)	0.019 (2)	0.0020 (18)	−0.0001 (17)	0.0006 (19)
N2	0.018 (2)	0.026 (2)	0.0121 (19)	−0.0012 (19)	0.0017 (17)	−0.0016 (19)
C1	0.023 (3)	0.016 (3)	0.014 (2)	0.002 (2)	−0.001 (2)	0.002 (2)
C2	0.027 (3)	0.022 (3)	0.013 (2)	0.002 (2)	0.005 (2)	0.000 (2)
C3	0.022 (3)	0.023 (3)	0.026 (3)	0.000 (2)	0.009 (2)	0.000 (2)
C4	0.017 (3)	0.017 (3)	0.023 (3)	0.001 (2)	0.000 (2)	−0.003 (2)
C5	0.025 (3)	0.020 (3)	0.013 (2)	0.004 (2)	0.001 (2)	0.000 (2)
C6	0.022 (3)	0.018 (3)	0.014 (2)	0.002 (2)	0.002 (2)	−0.001 (2)
C7	0.024 (3)	0.016 (3)	0.011 (2)	0.005 (2)	0.003 (2)	0.001 (2)
C9	0.021 (3)	0.018 (3)	0.015 (3)	0.001 (2)	−0.001 (2)	0.002 (2)
C10	0.019 (3)	0.023 (3)	0.013 (2)	0.003 (2)	−0.003 (2)	0.006 (2)
C11	0.022 (3)	0.024 (3)	0.018 (3)	0.001 (2)	−0.004 (2)	0.001 (2)
C12	0.019 (3)	0.036 (3)	0.025 (3)	−0.007 (2)	−0.006 (2)	0.013 (3)
C13	0.017 (3)	0.035 (3)	0.020 (3)	−0.001 (2)	0.000 (2)	0.009 (2)

C14	0.024 (3)	0.029 (3)	0.013 (2)	0.000 (2)	−0.002 (2)	0.007 (2)
C15	0.018 (3)	0.022 (3)	0.017 (2)	−0.001 (2)	−0.002 (2)	0.002 (2)

Geometric parameters (Å, °)

Cl1—C4	1.747 (5)	C1—C6	1.403 (6)
O1—C1	1.365 (6)	C2—C3	1.371 (7)
O1—H1	0.8400	C2—H2A	0.9500
O2—C9	1.247 (6)	C3—C4	1.395 (7)
O3—C12	1.371 (6)	C3—H3A	0.9500
O3—H3	0.8400	C4—C5	1.376 (7)
O4—C13	1.361 (6)	C5—C6	1.403 (7)
O4—H4	0.8400	C5—H5A	0.9500
O5—C14	1.366 (6)	C6—C7	1.450 (7)
O5—H5	0.8400	C7—H7	0.9500
O1w—H11	0.8347	C9—C10	1.474 (7)
O1w—H12	0.8378	C10—C11	1.384 (7)
O2w—H21	0.8421	C10—C15	1.408 (7)
O2w—H22	0.8400	C11—C12	1.377 (7)
N1—C7	1.283 (6)	C11—H11A	0.9500
N1—N2	1.386 (6)	C12—C13	1.395 (8)
N2—C9	1.350 (6)	C13—C14	1.394 (7)
N2—H2	0.8800	C14—C15	1.385 (7)
C1—C2	1.384 (7)	C15—H15	0.9500
C1—O1—H1	109.5	C1—C6—C7	122.9 (4)
C12—O3—H3	109.5	C5—C6—C7	118.3 (4)
C13—O4—H4	109.5	N1—C7—C6	121.0 (4)
C14—O5—H5	109.5	N1—C7—H7	119.5
H11—O1w—H12	110.0	C6—C7—H7	119.5
H21—O2w—H22	109.0	O2—C9—N2	122.2 (4)
C7—N1—N2	115.9 (4)	O2—C9—C10	122.6 (4)
C9—N2—N1	121.2 (4)	N2—C9—C10	115.1 (4)
C9—N2—H2	119.4	C11—C10—C15	120.3 (5)
N1—N2—H2	119.4	C11—C10—C9	119.7 (4)
O1—C1—C2	118.6 (4)	C15—C10—C9	119.9 (4)
O1—C1—C6	121.4 (4)	C12—C11—C10	120.0 (5)
C2—C1—C6	120.0 (4)	C12—C11—H11A	120.0
C3—C2—C1	121.2 (5)	C10—C11—H11A	120.0
C3—C2—H2A	119.4	O3—C12—C11	119.0 (5)
C1—C2—H2A	119.4	O3—C12—C13	120.5 (5)
C2—C3—C4	119.0 (5)	C11—C12—C13	120.5 (5)
C2—C3—H3A	120.5	O4—C13—C14	123.5 (5)
C4—C3—H3A	120.5	O4—C13—C12	116.8 (5)
C5—C4—C3	121.1 (5)	C14—C13—C12	119.7 (5)
C5—C4—Cl1	119.4 (4)	O5—C14—C15	123.4 (4)
C3—C4—Cl1	119.5 (4)	O5—C14—C13	116.2 (4)
C4—C5—C6	119.9 (4)	C15—C14—C13	120.3 (5)
C4—C5—H5A	120.0	C14—C15—C10	119.2 (4)
C6—C5—H5A	120.0	C14—C15—H15	120.4

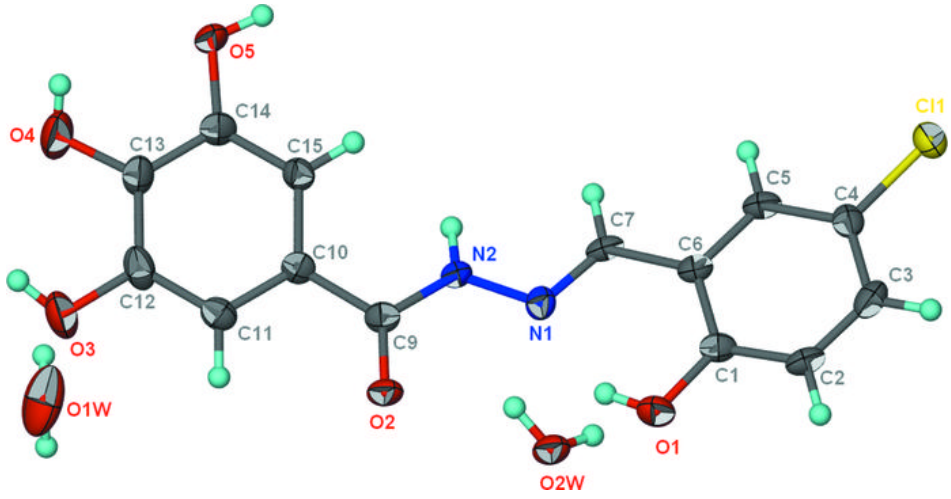
C1—C6—C5	118.8 (5)	C10—C15—H15	120.4
C7—N1—N2—C9	−166.9 (5)	N2—C9—C10—C11	154.5 (5)
O1—C1—C2—C3	−180.0 (5)	O2—C9—C10—C15	148.7 (5)
C6—C1—C2—C3	−1.1 (8)	N2—C9—C10—C15	−29.5 (7)
C1—C2—C3—C4	1.0 (8)	C15—C10—C11—C12	−0.4 (8)
C2—C3—C4—C5	−0.6 (8)	C9—C10—C11—C12	175.6 (5)
C2—C3—C4—Cl1	178.0 (4)	C10—C11—C12—O3	−179.3 (5)
C3—C4—C5—C6	0.2 (8)	C10—C11—C12—C13	−1.2 (8)
Cl1—C4—C5—C6	−178.4 (4)	O3—C12—C13—O4	−0.1 (8)
O1—C1—C6—C5	179.6 (5)	C11—C12—C13—O4	−178.2 (5)
C2—C1—C6—C5	0.7 (7)	O3—C12—C13—C14	179.2 (5)
O1—C1—C6—C7	1.2 (8)	C11—C12—C13—C14	1.1 (8)
C2—C1—C6—C7	−177.7 (5)	O4—C13—C14—O5	1.3 (8)
C4—C5—C6—C1	−0.3 (7)	C12—C13—C14—O5	−178.0 (5)
C4—C5—C6—C7	178.2 (5)	O4—C13—C14—C15	179.9 (5)
N2—N1—C7—C6	176.3 (4)	C12—C13—C14—C15	0.6 (8)
C1—C6—C7—N1	−0.1 (8)	O5—C14—C15—C10	176.3 (5)
C5—C6—C7—N1	−178.5 (5)	C13—C14—C15—C10	−2.2 (8)
N1—N2—C9—O2	−13.1 (8)	C11—C10—C15—C14	2.1 (7)
N1—N2—C9—C10	165.1 (4)	C9—C10—C15—C14	−173.9 (5)
O2—C9—C10—C11	−27.4 (8)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.84	1.89	2.631 (5)	146
O3—H3···O1w^{i}	0.84	1.96	2.737 (6)	153
O4—H4···O1w^{ii}	0.84	1.80	2.599 (7)	158
O5—H5···O2^{ii}	0.84	1.93	2.765 (5)	171
O1w—H11···O3	0.83	2.28	2.969 (6)	140
O1w—H12···O4^{iii}	0.84	2.07	2.900 (7)	170
O2w—H21···O1	0.84	2.15	2.946 (5)	157
O2w—H22···O2^{iv}	0.84	1.97	2.808 (5)	172
N2—H2···O2w^{ii}	0.88	2.03	2.882 (5)	162

Symmetry codes: (i) −*x*+2, *y*+1/2, −*z*+3/2; (ii) *x*, −*y*+3/2, *z*−1/2; (iii) *x*, −*y*+5/2, *z*+1/2; (iv) *x*, *y*−1, *z*.

Fig. 1


O5
C14
O4
C13
C15
C10
N2
C7
C6
C5
C4
Cl1
C12
C11
C9
N1
C3
O3
C1
C2
O1W
O2
O1
O2W