Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-(3-Bromo-5-chloro-2-hydroxybenzylidene)-3,4,5-trihydroxybenzohydrazide methanol solvate

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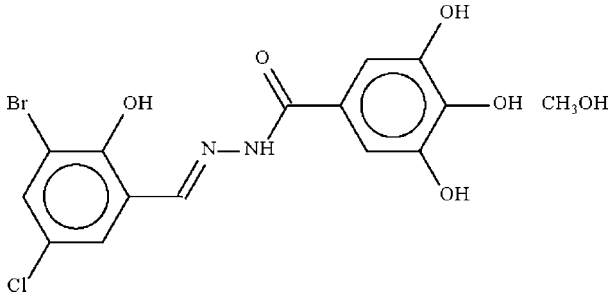
Received 18 March 2009; accepted 23 March 2009

Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.002 Å; R factor = 0.023; wR factor = 0.068; data-to-parameter ratio = 14.8.

The benzohydrazide molecule of the title compound, $C_{14}H_{10}BrClN_2O_5\cdot CH_3OH$, is non-planar, the two aromatic rings at either side of the –C(=O)–NH–N=CH– unit being twisted by 5.9 (1)°. The benzohydrazide molecule is linked to the solvent molecule by an O—H···O hydrogen bond. Molecules are connected by further O—H···O hydrogen bonds and an N—H···O link into a two-dimensional array.

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

$C_{14}H_{10}BrClN_2O_5\cdot CH_4O$
M_r = 433.64
Monoclinic, $C2/c$
a = 21.6157 (3) Å
b = 12.7408 (2) Å
c = 17.0803 (2) Å
β = 136.641 (1)°
V = 3229.57 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 2.75 mm^{-1}
T = 123 K
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.610, T_{max} = 0.771
15301 measured reflections
3714 independent reflections
3407 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.068
S = 1.03
3714 reflections
251 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O5^{i}	0.83 (1)	2.03 (3)	2.607 (2)	126 (3)
O3—H3···O6^{ii}	0.83 (1)	2.05 (1)	2.856 (2)	165 (3)
O4—H4···O1^{iii}	0.83 (1)	2.20 (1)	3.027 (2)	173 (2)
O5—H5···O2^{iii}	0.82 (1)	1.79 (1)	2.608 (2)	176 (2)
O6—H6···O2	0.83 (1)	2.04 (1)	2.843 (2)	164 (2)
N2—H2···O6^{iii}	0.87 (1)	2.25 (1)	3.112 (2)	169 (2)

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

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: TK2400).

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supporting information

Acta Cryst. (2009). E**65**, o909 [doi:10.1107/S1600536809010575]

N′-(3-Bromo-5-chloro-2-hydroxybenzylidene)-3,4,5-trihydroxybenzohydrazide methanol solvate

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

S1. Experimental

3-Bromo-5-chloro-2-hydroxybenzaldehyde (0.47 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 methanol.

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.

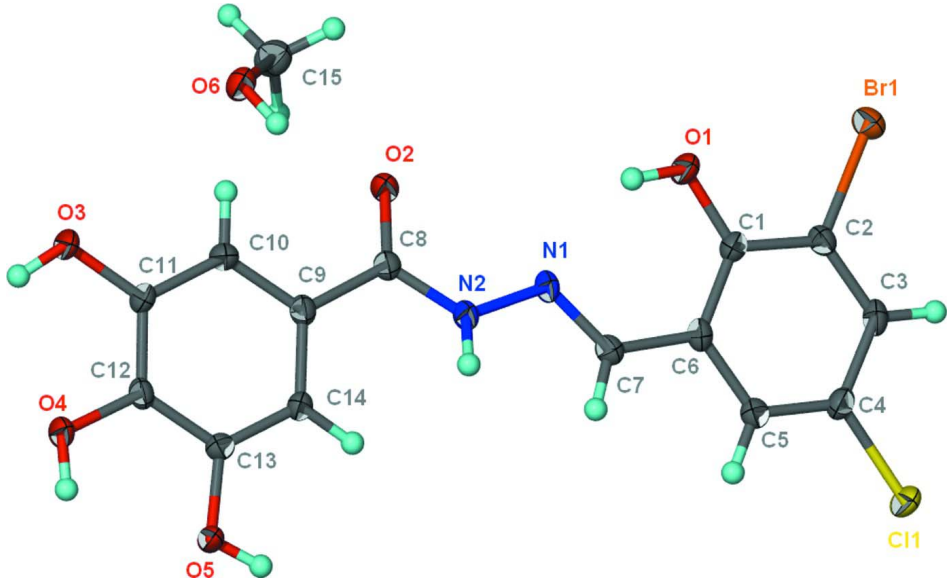

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{14}H_{10}BrClNO_5{\cdot}CH_3OH$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

N′-(3-Bromo-5-chloro-2-hydroxybenzylidene)-3,4,5-trihydroxybenzohydrazide methanol solvate

Crystal data

$C_{14}H_{10}BrClN_2O_5{\cdot}CH_4O$
M_r = 433.64

Monoclinic, $C2/c$
Hall symbol: -C 2yc

a = 21.6157 (3) Å
b = 12.7408 (2) Å
c = 17.0803 (2) Å
β = 136.641 (1)°
V = 3229.57 (8) Å^3
Z = 8
F(000) = 1744
D_x = 1.784 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8789 reflections
θ = 2.7–28.3°
μ = 2.75 mm^{-1}
T = 123 K
Polyhedral, tan
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.610, T_{max} = 0.771

15301 measured reflections
3714 independent reflections
3407 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 27.5°, θ_{min} = 2.0°
h = −28→26
k = −16→16
l = −22→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.068
S = 1.03
3714 reflections
251 parameters
6 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
$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 3.4447P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
Br1	0.044783 (12)	0.783092 (13)	0.137916 (14)	0.02172 (7)
Cl1	0.01420 (3)	0.35175 (3)	0.10946 (4)	0.02369 (10)
N1	0.18779 (9)	0.61375 (11)	0.54542 (11)	0.0148 (3)
N2	0.23179 (9)	0.58418 (11)	0.65407 (11)	0.0154 (3)
O1	0.13116 (9)	0.74001 (9)	0.37133 (11)	0.0211 (3)
O2	0.20939 (8)	0.74133 (9)	0.69379 (10)	0.0187 (2)
O3	0.36037 (9)	0.69205 (10)	1.09583 (10)	0.0219 (3)
O4	0.37603 (8)	0.47725 (9)	1.12740 (10)	0.0189 (2)
O5	0.31633 (9)	0.34724 (9)	0.95863 (10)	0.0205 (3)
O6	0.15053 (8)	0.88367 (9)	0.75713 (10)	0.0196 (2)
C1	0.10878 (11)	0.64859 (12)	0.31698 (13)	0.0155 (3)
C2	0.06644 (11)	0.65171 (12)	0.20499 (14)	0.0156 (3)
C3	0.03858 (11)	0.56163 (13)	0.14173 (13)	0.0172 (3)
H3A	0.0088	0.5655	0.0652	0.021*
C4	0.05469 (11)	0.46540 (12)	0.19171 (14)	0.0168 (3)
C5	0.09940 (10)	0.45814 (12)	0.30364 (13)	0.0158 (3)
H5A	0.1117	0.3914	0.3373	0.019*

C6	0.12644 (10)	0.54979 (12)	0.36718 (13)	0.0151 (3)
C7	0.17192 (10)	0.53678 (12)	0.48399 (13)	0.0155 (3)
H7	0.1907	0.4684	0.5163	0.019*
C8	0.23645 (10)	0.64897 (12)	0.72065 (13)	0.0147 (3)
C9	0.27436 (10)	0.60244 (12)	0.82779 (13)	0.0143 (3)
C10	0.30257 (11)	0.66835 (12)	0.91445 (13)	0.0161 (3)
H10	0.2997	0.7424	0.9056	0.019*
C11	0.33473 (11)	0.62583 (12)	1.01356 (13)	0.0156 (3)
C12	0.34227 (10)	0.51690 (12)	1.02835 (13)	0.0146 (3)
C13	0.31234 (10)	0.45163 (12)	0.94055 (13)	0.0147 (3)
C14	0.27745 (10)	0.49359 (12)	0.84020 (13)	0.0146 (3)
H14	0.2557	0.4486	0.7800	0.018*
C15	0.06333 (12)	0.92150 (14)	0.65359 (15)	0.0237 (4)
H15A	0.0445	0.9781	0.6715	0.036*
H15B	0.0196	0.8640	0.6160	0.036*
H15C	0.0664	0.9482	0.6027	0.036*
H1	0.154 (2)	0.731 (3)	0.4365 (15)	0.087 (13)*
H3	0.3635 (18)	0.6613 (19)	1.1413 (18)	0.044 (7)*
H4	0.3783 (17)	0.4119 (8)	1.129 (2)	0.036 (7)*
H5	0.3065 (15)	0.3125 (16)	0.9101 (15)	0.030 (6)*
H6	0.1634 (16)	0.8331 (14)	0.741 (2)	0.037 (7)*
H2	0.2604 (14)	0.5242 (11)	0.6799 (18)	0.029 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02904 (11)	0.01747 (10)	0.01845 (10)	0.00031 (6)	0.01720 (9)	0.00285 (6)
Cl1	0.0373 (2)	0.01580 (19)	0.01895 (19)	−0.00382 (16)	0.02079 (19)	−0.00522 (14)
N1	0.0169 (6)	0.0172 (6)	0.0109 (6)	0.0006 (5)	0.0103 (5)	0.0013 (5)
N2	0.0205 (6)	0.0142 (6)	0.0119 (6)	0.0032 (5)	0.0120 (6)	0.0031 (5)
O1	0.0321 (7)	0.0152 (5)	0.0161 (6)	0.0006 (5)	0.0175 (6)	−0.0016 (5)
O2	0.0293 (6)	0.0135 (5)	0.0186 (6)	0.0039 (5)	0.0191 (5)	0.0034 (4)
O3	0.0374 (7)	0.0154 (5)	0.0171 (6)	−0.0039 (5)	0.0212 (6)	−0.0038 (5)
O4	0.0294 (6)	0.0154 (6)	0.0142 (5)	0.0022 (5)	0.0166 (5)	0.0019 (4)
O5	0.0378 (7)	0.0109 (5)	0.0182 (6)	0.0028 (5)	0.0221 (6)	0.0017 (4)
O6	0.0251 (6)	0.0178 (6)	0.0186 (6)	0.0016 (5)	0.0168 (5)	−0.0013 (5)
C1	0.0184 (7)	0.0151 (7)	0.0154 (7)	−0.0006 (6)	0.0131 (7)	−0.0020 (6)
C2	0.0186 (7)	0.0153 (7)	0.0153 (7)	0.0015 (6)	0.0130 (7)	0.0026 (6)
C3	0.0185 (7)	0.0226 (8)	0.0135 (7)	−0.0012 (6)	0.0126 (7)	−0.0011 (6)
C4	0.0202 (7)	0.0170 (7)	0.0169 (7)	−0.0017 (6)	0.0147 (7)	−0.0037 (6)
C5	0.0184 (7)	0.0153 (7)	0.0164 (7)	0.0005 (6)	0.0136 (6)	0.0007 (6)
C6	0.0164 (7)	0.0182 (7)	0.0129 (7)	0.0004 (6)	0.0113 (6)	0.0003 (6)
C7	0.0173 (7)	0.0156 (7)	0.0135 (7)	0.0006 (6)	0.0112 (6)	0.0017 (6)
C8	0.0165 (7)	0.0151 (7)	0.0138 (7)	−0.0016 (6)	0.0114 (6)	−0.0007 (5)
C9	0.0158 (7)	0.0158 (7)	0.0126 (7)	0.0010 (6)	0.0107 (6)	0.0009 (6)
C10	0.0212 (8)	0.0123 (7)	0.0166 (8)	−0.0005 (6)	0.0144 (7)	−0.0003 (6)
C11	0.0198 (7)	0.0148 (7)	0.0140 (7)	−0.0021 (6)	0.0129 (6)	−0.0030 (6)
C12	0.0168 (7)	0.0167 (7)	0.0123 (7)	0.0009 (6)	0.0112 (6)	0.0013 (6)

C13	0.0180 (7)	0.0121 (7)	0.0168 (7)	0.0016 (6)	0.0136 (6)	0.0006 (6)
C14	0.0189 (7)	0.0136 (7)	0.0144 (7)	0.0005 (6)	0.0131 (6)	−0.0005 (5)
C15	0.0247 (8)	0.0219 (8)	0.0214 (8)	0.0000 (7)	0.0158 (7)	−0.0015 (7)

Geometric parameters (Å, º)

Br1—C2	1.8852 (15)	C3—C4	1.386 (2)
Cl1—C4	1.7460 (16)	C3—H3A	0.9500
N1—C7	1.287 (2)	C4—C5	1.382 (2)
N1—N2	1.3870 (18)	C5—C6	1.400 (2)
N2—C8	1.348 (2)	C5—H5A	0.9500
N2—H2	0.874 (10)	C6—C7	1.457 (2)
O1—C1	1.3415 (19)	C7—H7	0.9500
O1—H1	0.832 (10)	C8—C9	1.480 (2)
O2—C8	1.2438 (19)	C9—C14	1.397 (2)
O3—C11	1.3642 (19)	C9—C10	1.398 (2)
O3—H3	0.828 (10)	C10—C11	1.390 (2)
O4—C12	1.3619 (18)	C10—H10	0.9500
O4—H4	0.833 (10)	C11—C12	1.399 (2)
O5—C13	1.3538 (18)	C12—C13	1.396 (2)
O5—H5	0.820 (10)	C13—C14	1.383 (2)
O6—C15	1.437 (2)	C14—H14	0.9500
O6—H6	0.828 (10)	C15—H15A	0.9800
C1—C2	1.397 (2)	C15—H15B	0.9800
C1—C6	1.410 (2)	C15—H15C	0.9800
C2—C3	1.379 (2)		
C7—N1—N2	113.63 (13)	C6—C7—H7	118.5
C8—N2—N1	121.35 (13)	O2—C8—N2	122.77 (14)
C8—N2—H2	121.2 (15)	O2—C8—C9	121.61 (14)
N1—N2—H2	117.2 (15)	N2—C8—C9	115.61 (13)
C1—O1—H1	112 (3)	C14—C9—C10	119.97 (14)
C11—O3—H3	111.6 (19)	C14—C9—C8	120.49 (14)
C12—O4—H4	112.6 (18)	C10—C9—C8	119.44 (14)
C13—O5—H5	111.9 (17)	C11—C10—C9	120.15 (14)
C15—O6—H6	107.8 (17)	C11—C10—H10	119.9
O1—C1—C2	118.05 (14)	C9—C10—H10	119.9
O1—C1—C6	123.52 (14)	O3—C11—C10	118.85 (14)
C2—C1—C6	118.42 (14)	O3—C11—C12	121.39 (14)
C3—C2—C1	121.82 (14)	C10—C11—C12	119.75 (14)
C3—C2—Br1	119.27 (12)	O4—C12—C13	121.65 (14)
C1—C2—Br1	118.90 (12)	O4—C12—C11	118.63 (14)
C2—C3—C4	118.86 (14)	C13—C12—C11	119.67 (14)
C2—C3—H3A	120.6	O5—C13—C14	123.32 (14)
C4—C3—H3A	120.6	O5—C13—C12	115.94 (14)
C5—C4—C3	121.40 (14)	C14—C13—C12	120.69 (14)
C5—C4—Cl1	119.65 (12)	C13—C14—C9	119.64 (14)
C3—C4—Cl1	118.92 (12)	C13—C14—H14	120.2

C4—C5—C6	119.62 (14)	C9—C14—H14	120.2
C4—C5—H5A	120.2	O6—C15—H15A	109.5
C6—C5—H5A	120.2	O6—C15—H15B	109.5
C5—C6—C1	119.81 (14)	H15A—C15—H15B	109.5
C5—C6—C7	116.89 (14)	O6—C15—H15C	109.5
C1—C6—C7	123.30 (14)	H15A—C15—H15C	109.5
N1—C7—C6	122.97 (14)	H15B—C15—H15C	109.5
N1—C7—H7	118.5		

C7—N1—N2—C8	−165.52 (15)	N1—N2—C8—C9	172.04 (14)
O1—C1—C2—C3	−177.88 (15)	O2—C8—C9—C14	159.38 (15)
C6—C1—C2—C3	2.7 (2)	N2—C8—C9—C14	−19.4 (2)
O1—C1—C2—Br1	0.8 (2)	O2—C8—C9—C10	−17.1 (2)
C6—C1—C2—Br1	−178.63 (11)	N2—C8—C9—C10	164.18 (15)
C1—C2—C3—C4	−1.2 (2)	C14—C9—C10—C11	0.8 (2)
Br1—C2—C3—C4	−179.92 (12)	C8—C9—C10—C11	177.25 (14)
C2—C3—C4—C5	−1.3 (2)	C9—C10—C11—O3	−178.92 (15)
C2—C3—C4—Cl1	176.64 (12)	C9—C10—C11—C12	2.6 (2)
C3—C4—C5—C6	2.2 (2)	O3—C11—C12—O4	0.0 (2)
Cl1—C4—C5—C6	−175.66 (12)	C10—C11—C12—O4	178.42 (14)
C4—C5—C6—C1	−0.7 (2)	O3—C11—C12—C13	177.72 (14)
C4—C5—C6—C7	179.02 (14)	C10—C11—C12—C13	−3.9 (2)
O1—C1—C6—C5	178.92 (15)	O4—C12—C13—O5	2.0 (2)
C2—C1—C6—C5	−1.7 (2)	C11—C12—C13—O5	−175.63 (14)
O1—C1—C6—C7	−0.8 (2)	O4—C12—C13—C14	179.35 (14)
C2—C1—C6—C7	178.61 (14)	C11—C12—C13—C14	1.7 (2)
N2—N1—C7—C6	−179.99 (14)	O5—C13—C14—C9	178.84 (15)
C5—C6—C7—N1	−169.07 (15)	C12—C13—C14—C9	1.7 (2)
C1—C6—C7—N1	10.7 (2)	C10—C9—C14—C13	−3.0 (2)
N1—N2—C8—O2	−6.7 (2)	C8—C9—C14—C13	−179.38 (14)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O5^{i}	0.83 (1)	2.03 (3)	2.607 (2)	126 (3)
O3—H3···O6^{ii}	0.83 (1)	2.05 (1)	2.856 (2)	165 (3)
O4—H4···O1^{iii}	0.83 (1)	2.20 (1)	3.027 (2)	173 (2)
O5—H5···O2^{iii}	0.82 (1)	1.79 (1)	2.608 (2)	176 (2)
O6—H6···O2	0.83 (1)	2.04 (1)	2.843 (2)	164 (2)
N2—H2···O6^{iii}	0.87 (1)	2.25 (1)	3.112 (2)	169 (2)

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