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(*E*)-*N*′-(5-Bromo-2-hydroxybenzylidene)-4-hydroxybenzohydrazide ethanol solvate

Zuo-Liang Jing,* Ming Yu and Xin Chen

College of Sciences, Tianjin University of Science and Technology, Tianjin 300457, People's Republic of China
Correspondence e-mail: jzl74@tust.edu.cn

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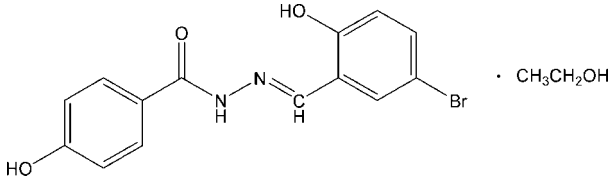
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.004 Å; R factor = 0.034; wR factor = 0.086; data-to-parameter ratio = 12.8.

In the Schiff base molecule of the title compound, $C_{14}H_{11}BrN_2O_3\cdot C_2H_6O$, the dihedral angle between the two benzene planes is 6.10 (2)°. An intramolecular O—H···N hydrogen bond stabilizes the molecular structure. The Schiff base and ethanol molecules are linked *via* weak intermolecular O—H···O and N—H···O hydrogen bonds, forming a three-dimensional network.

Related literature

For general background, see: Belloni *et al.* (2005); Kahwa *et al.* (1986); Parashar *et al.* (1988); Santos *et al.* (2001); Tynan *et al.* (2005).



Experimental

Crystal data

$C_{14}H_{11}BrN_2O_3\cdot C_2H_6O$
M_r = 381.23
Monoclinic, $C2/c$
a = 17.873 (4) Å
b = 17.834 (4) Å
c = 12.955 (3) Å
β = 129.48 (3)°
V = 3187.2 (19) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 2.60 mm^{-1}
T = 113 (2) K
0.14 × 0.10 × 0.04 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.712, T_{max} = 0.903
9688 measured reflections
2806 independent reflections
2367 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.087
S = 1.00
2806 reflections
219 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.86 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
O4—H4···O1^{i}	0.84	2.55	3.085 (3)	123
O4—H4···O3^{ii}	0.84	2.21	2.983 (3)	153
N2—H2···O4^{iii}	0.76 (3)	2.20 (3)	2.932 (3)	163 (3)
O3—H3···O2^{iii}	0.78 (3)	1.86 (3)	2.639 (3)	174 (3)
O1—H1···N1	0.77 (3)	1.96 (3)	2.645 (3)	148 (3)

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CrystalStructure* (Rigaku/MSC, 2005); software used to prepare material for publication: *CrystalStructure*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2249).

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supplementary materials

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(*E*)-*N*'-(5-Bromo-2-hydroxybenzylidene)-4-hydroxybenzohydrazide ethanol solvate

Z.-L. Jing, M. Yu and X. Chen

Comment

In order to establish control over the preparation of crystalline solid materials so that their architecture and properties are predictable (Belloni *et al.*, 2005; Tynan *et al.*, 2005; Parashar *et al.*, 1988), the synthesis of new and designed crystal structures has become a major strand of modern chemistry. Metal complexes based on Schiff bases have attracted much attention because they can be utilized as model compounds of the active centres in various proteins and enzymes (Kahwa *et al.*, 1986; Santos *et al.*, 2001). As part of an investigation of the coordination properties of Shiff bases functioning as ligands, we report the synthesis and crystal structure of the title compound, (I).

In the molecular structure of the compound, (I), the geometric parameters are normal (Fig. 1). The dihedral angle between two benzene planes is 6.10 (2)°. An intramolecular O—H···N hydrogen bond (Table 1) stabilizes the molecular structure. The Schiff base and ethanol molecules are linked *via* weak intermolecular O—H···O and N—H···O hydrogen bonds (Table 1), forming a three-dimensional framework, as illustrated in Fig. 2.

Experimental

An anhydrous ethanol solution (50 ml) of 5-bromo-2-hydroxybenzaldehyde (1.99 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of 4-methoxybenzohydrazide (1.66 g, 10 mmol), and the mixture was stirred at 350 K for 6 h under N_2, whereupon a red precipitate appeared. The product was isolated, recrystallized from anhydrous ethanol and then dried *in vacuo* to give pure compound (I) in 94% yield. Red single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an anhydrous ethanol solution.

Refinement

The N-bound H atom was located in a difference Fourier map and its positional parameters were refined, with U_{iso}(H) = 1.2U_{eq}(N). C-bound H atoms were included in calculated positions, with C—H = 0.93 (aromatic) or 0.96 Å (methyl), and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) for aromatic H or 1.5U_{eq}(C) for methyl H atoms.

Figures

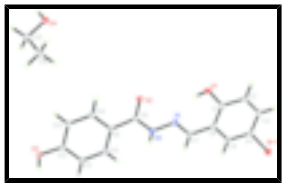

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

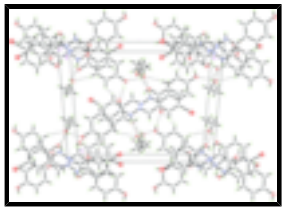

Fig. 2. The crystal packing of (I), viwed down the *c* axis. Hydrogen bonds are indicated by dashed lines.

(*E*)—*N*'-(5-Bromo-2-hydroxybenzylidene)-4-hydroxybenzohydrazide ethanol solvate

Crystal data

$C_{14}H_{11}BrN_2O_3 \cdot C_2H_6O$	F_{000} = 1552
M_r = 381.23	D_x = 1.589 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 2324 reflections
a = 17.873 (4) Å	θ = 2.3–25.0º
b = 17.834 (4) Å	μ = 2.60 mm^{-1}
c = 12.955 (3) Å	*T* = 113 (2) K
β = 129.48 (3)º	Block, red
V = 3187.2 (19) Å^3	0.14 × 0.10 × 0.04 mm
Z = 8	

Data collection

Rigaku Saturn diffractometer	2806 independent reflections
Radiation source: rotating anode	2367 reflections with $I > 2\sigma(I)$
Monochromator: confocal	R_{int} = 0.047
T = 113(2) K	θ_{max} = 25.0º
ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)	*h* = −21→19
T_{min} = 0.712, T_{max} = 0.903	*k* = −21→18
9688 measured reflections	*l* = −14→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.034	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.087	$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max}$ = 0.002
2806 reflections	$\Delta\rho_{max}$ = 0.86 e Å^{-3}

219 parameters	$\Delta\rho_{min} = -0.49$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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}
Br1	0.55893 (2)	0.853491 (14)	0.65804 (3)	0.02871 (13)
O1	0.34688 (14)	0.55776 (11)	0.50624 (18)	0.0256 (5)
H1	0.383 (2)	0.5253 (18)	0.538 (3)	0.038*
O2	0.45039 (13)	0.35423 (9)	0.59475 (17)	0.0204 (4)
O3	0.77352 (14)	0.10340 (10)	0.88829 (18)	0.0239 (4)
H3	0.827 (2)	0.1135 (18)	0.948 (3)	0.036*
N1	0.51873 (15)	0.49290 (11)	0.64199 (19)	0.0190 (5)
N2	0.57836 (16)	0.43189 (12)	0.6910 (2)	0.0185 (5)
H2	0.632 (2)	0.4377 (15)	0.731 (3)	0.022*
C1	0.3970 (2)	0.62352 (15)	0.5420 (2)	0.0213 (6)
C2	0.3441 (2)	0.69029 (15)	0.4984 (3)	0.0245 (6)
H2A	0.2756	0.6888	0.4450	0.029*
C3	0.3913 (2)	0.75889 (15)	0.5328 (3)	0.0239 (6)
H3A	0.3553	0.8043	0.5034	0.029*
C4	0.4911 (2)	0.76069 (14)	0.6103 (2)	0.0224 (6)
C5	0.54505 (19)	0.69547 (15)	0.6545 (2)	0.0205 (6)
H5	0.6135	0.6978	0.7077	0.025*
C6	0.4982 (2)	0.62563 (14)	0.6204 (2)	0.0199 (6)
C7	0.55798 (18)	0.55823 (15)	0.6696 (2)	0.0201 (6)
H7	0.6263	0.5626	0.7224	0.024*
C8	0.53862 (19)	0.36222 (13)	0.6657 (2)	0.0171 (6)
C9	0.60542 (18)	0.29712 (14)	0.7271 (2)	0.0174 (5)
C10	0.70658 (19)	0.30213 (14)	0.8167 (2)	0.0219 (6)
H10	0.7365	0.3501	0.8402	0.026*
C11	0.76376 (19)	0.23865 (14)	0.8716 (2)	0.0213 (6)
H11	0.8324	0.2431	0.9323	0.026*
C12	0.72042 (19)	0.16788 (14)	0.8377 (2)	0.0195 (6)
C13	0.61988 (19)	0.16194 (14)	0.7488 (3)	0.0207 (6)
H13	0.5901	0.1140	0.7256	0.025*
C14	0.56334 (18)	0.22577 (14)	0.6941 (2)	0.0183 (5)

H14	0.4947	0.2212	0.6331	0.022*
O4	0.27282 (13)	0.02334 (10)	0.29428 (17)	0.0264 (4)
H4	0.2566	−0.0001	0.2266	0.040*
C16	0.3665 (2)	0.02035 (17)	0.5312 (3)	0.0368 (7)
H16A	0.3933	0.0700	0.5395	0.055*
H16B	0.4141	−0.0089	0.6121	0.055*
H16C	0.3071	0.0259	0.5200	0.055*
C15	0.3436 (2)	−0.01922 (16)	0.4121 (3)	0.0353 (8)
H15A	0.4032	−0.0245	0.4224	0.042*
H15B	0.3181	−0.0700	0.4045	0.042*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0351 (2)	0.01993 (19)	0.0299 (2)	−0.00043 (11)	0.02010 (16)	0.00053 (10)
O1	0.0235 (11)	0.0244 (11)	0.0217 (10)	−0.0003 (8)	0.0109 (9)	0.0005 (8)
O2	0.0182 (11)	0.0245 (10)	0.0174 (10)	0.0002 (7)	0.0107 (9)	0.0006 (7)
O3	0.0185 (10)	0.0210 (10)	0.0198 (10)	0.0011 (8)	0.0064 (8)	−0.0010 (8)
N1	0.0241 (12)	0.0200 (12)	0.0159 (11)	0.0024 (9)	0.0141 (10)	0.0023 (9)
N2	0.0178 (11)	0.0177 (12)	0.0205 (12)	−0.0002 (9)	0.0125 (10)	0.0001 (9)
C1	0.0262 (15)	0.0247 (14)	0.0163 (14)	−0.0029 (11)	0.0151 (13)	−0.0010 (10)
C2	0.0226 (15)	0.0330 (16)	0.0180 (13)	0.0029 (11)	0.0129 (12)	0.0030 (11)
C3	0.0330 (16)	0.0218 (14)	0.0204 (14)	0.0064 (11)	0.0186 (13)	0.0050 (11)
C4	0.0312 (16)	0.0198 (14)	0.0195 (14)	−0.0012 (11)	0.0177 (13)	−0.0007 (10)
C5	0.0213 (14)	0.0268 (15)	0.0146 (13)	0.0001 (10)	0.0119 (12)	−0.0007 (10)
C6	0.0281 (16)	0.0238 (14)	0.0130 (13)	0.0013 (11)	0.0155 (12)	0.0006 (10)
C7	0.0211 (14)	0.0253 (15)	0.0149 (13)	0.0006 (10)	0.0118 (12)	0.0004 (10)
C8	0.0187 (15)	0.0240 (15)	0.0105 (13)	−0.0005 (10)	0.0101 (12)	−0.0006 (10)
C9	0.0181 (14)	0.0230 (14)	0.0116 (12)	0.0009 (10)	0.0096 (11)	0.0010 (10)
C10	0.0250 (15)	0.0188 (13)	0.0198 (13)	−0.0029 (10)	0.0133 (12)	−0.0016 (10)
C11	0.0153 (13)	0.0249 (14)	0.0178 (13)	−0.0012 (10)	0.0078 (11)	−0.0019 (10)
C12	0.0204 (14)	0.0224 (14)	0.0166 (13)	0.0025 (10)	0.0122 (12)	0.0011 (10)
C13	0.0228 (15)	0.0189 (13)	0.0203 (14)	−0.0031 (10)	0.0135 (13)	−0.0025 (10)
C14	0.0173 (14)	0.0233 (14)	0.0142 (12)	−0.0016 (10)	0.0100 (11)	−0.0006 (10)
O4	0.0260 (11)	0.0288 (10)	0.0183 (10)	0.0027 (8)	0.0112 (9)	−0.0029 (8)
C16	0.0380 (18)	0.0439 (18)	0.0231 (15)	0.0096 (14)	0.0169 (14)	0.0026 (13)
C15	0.0336 (18)	0.0304 (16)	0.0251 (16)	0.0104 (13)	0.0108 (14)	−0.0008 (12)

Geometric parameters (Å, °)

Br1—C4	1.907 (3)	C7—H7	0.9500
O1—C1	1.366 (3)	C8—C9	1.483 (3)
O1—H1	0.77 (3)	C9—C10	1.398 (4)
O2—C8	1.228 (3)	C9—C14	1.400 (3)
O3—C12	1.364 (3)	C10—C11	1.381 (3)
O3—H3	0.78 (3)	C10—H10	0.9500
N1—C7	1.288 (3)	C11—C12	1.398 (4)
N1—N2	1.365 (3)	C11—H11	0.9500
N2—C8	1.364 (3)	C12—C13	1.391 (4)

N2—H2	0.76 (3)	C13—C14	1.381 (4)
C1—C2	1.397 (4)	C13—H13	0.9500
C1—C6	1.402 (4)	C14—H14	0.9500
C2—C3	1.389 (4)	O4—C15	1.432 (3)
C2—H2A	0.9500	O4—H4	0.8400
C3—C4	1.383 (4)	C16—C15	1.497 (4)
C3—H3A	0.9500	C16—H16A	0.9800
C4—C5	1.381 (4)	C16—H16B	0.9800
C5—C6	1.406 (4)	C16—H16C	0.9800
C5—H5	0.9500	C15—H15A	0.9900
C6—C7	1.459 (4)	C15—H15B	0.9900
C1—O1—H1	108 (3)	C10—C9—C8	124.8 (2)
C12—O3—H3	109 (2)	C14—C9—C8	117.1 (2)
C7—N1—N2	117.8 (2)	C11—C10—C9	121.2 (2)
C8—N2—N1	118.8 (2)	C11—C10—H10	119.4
C8—N2—H2	122 (2)	C9—C10—H10	119.4
N1—N2—H2	119 (2)	C10—C11—C12	119.8 (2)
O1—C1—C2	117.7 (2)	C10—C11—H11	120.1
O1—C1—C6	122.3 (2)	C12—C11—H11	120.1
C2—C1—C6	120.0 (2)	O3—C12—C13	118.1 (2)
C3—C2—C1	120.3 (3)	O3—C12—C11	122.2 (2)
C3—C2—H2A	119.9	C13—C12—C11	119.7 (2)
C1—C2—H2A	119.9	C14—C13—C12	120.0 (2)
C4—C3—C2	119.5 (2)	C14—C13—H13	120.0
C4—C3—H3A	120.2	C12—C13—H13	120.0
C2—C3—H3A	120.2	C13—C14—C9	121.1 (2)
C5—C4—C3	121.3 (2)	C13—C14—H14	119.4
C5—C4—Br1	117.7 (2)	C9—C14—H14	119.4
C3—C4—Br1	121.04 (19)	C15—O4—H4	109.5
C4—C5—C6	119.7 (2)	C15—C16—H16A	109.5
C4—C5—H5	120.1	C15—C16—H16B	109.5
C6—C5—H5	120.1	H16A—C16—H16B	109.5
C1—C6—C5	119.2 (2)	C15—C16—H16C	109.5
C1—C6—C7	122.9 (2)	H16A—C16—H16C	109.5
C5—C6—C7	117.9 (2)	H16B—C16—H16C	109.5
N1—C7—C6	120.4 (2)	O4—C15—C16	109.0 (2)
N1—C7—H7	119.8	O4—C15—H15A	109.9
C6—C7—H7	119.8	C16—C15—H15A	109.9
O2—C8—N2	120.7 (2)	O4—C15—H15B	109.9
O2—C8—C9	121.6 (2)	C16—C15—H15B	109.9
N2—C8—C9	117.8 (2)	H15A—C15—H15B	108.3
C10—C9—C14	118.1 (2)		
C7—N1—N2—C8	178.4 (2)	N1—N2—C8—O2	3.5 (4)
O1—C1—C2—C3	179.3 (2)	N1—N2—C8—C9	−176.2 (2)
C6—C1—C2—C3	−0.2 (4)	O2—C8—C9—C10	−175.0 (2)
C1—C2—C3—C4	0.2 (4)	N2—C8—C9—C10	4.7 (4)
C2—C3—C4—C5	−0.1 (4)	O2—C8—C9—C14	3.9 (4)
C2—C3—C4—Br1	179.13 (19)	N2—C8—C9—C14	−176.4 (2)

C3—C4—C5—C6	0.0 (4)	C14—C9—C10—C11	−0.2 (4)
Br1—C4—C5—C6	−179.20 (18)	C8—C9—C10—C11	178.7 (2)
O1—C1—C6—C5	−179.4 (2)	C9—C10—C11—C12	0.0 (4)
C2—C1—C6—C5	0.2 (4)	C10—C11—C12—O3	179.5 (2)
O1—C1—C6—C7	0.3 (4)	C10—C11—C12—C13	0.0 (4)
C2—C1—C6—C7	179.8 (2)	O3—C12—C13—C14	−179.3 (2)
C4—C5—C6—C1	−0.1 (3)	C11—C12—C13—C14	0.1 (4)
C4—C5—C6—C7	−179.8 (2)	C12—C13—C14—C9	−0.3 (4)
N2—N1—C7—C6	−179.9 (2)	C10—C9—C14—C13	0.3 (4)
C1—C6—C7—N1	−0.1 (4)	C8—C9—C14—C13	−178.6 (2)
C5—C6—C7—N1	179.6 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O1^{i}	0.84	2.55	3.085 (3)	123
O4—H4···O3^{ii}	0.84	2.21	2.983 (3)	153
N2—H2···O4^{iii}	0.76 (3)	2.20 (3)	2.932 (3)	163 (3)
O3—H3···O2^{iii}	0.78 (3)	1.86 (3)	2.639 (3)	174 (3)
O1—H1···N1	0.77 (3)	1.96 (3)	2.645 (3)	148 (3)

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

Fig. 1

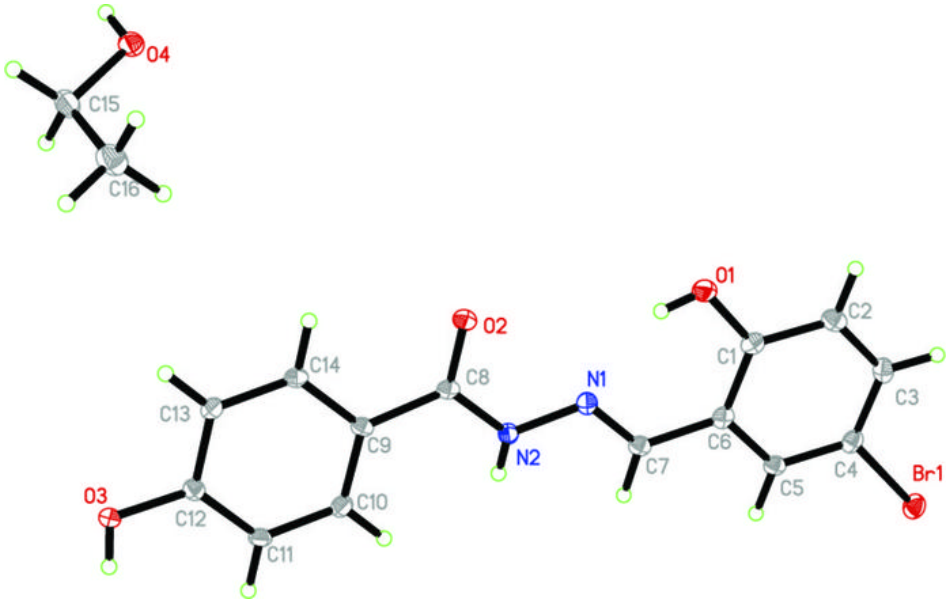
O4
C15
C16
O1
C2
C1
O2
C14
C8
N1
C3
C13
C9
N2
C6
C7
C4
Br1
C5
O3
C12
C10
C11

Fig. 2