Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-*N*′-(2-Hydroxy-3,5-diiodobenzylidene)-2-nitrobenzohydrazide methanol solvate

Heng-Yu Qian[a]* and Da-Ping Qu[b]

[a]Key Laboratory of Surface and Interface Science of Henan, School of Material & Chemical Engineering, Zhengzhou University of Light Industry, Zhengzhou 450002, People's Republic of China, and [b]Department of Chemistry, Dalian Teacher College, Dalian 116000, People's Republic of China
Correspondence e-mail: hengyu_qian@126.com

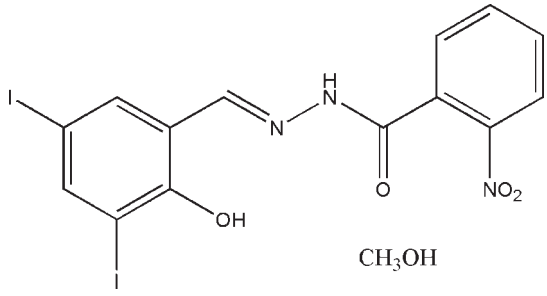
Received 19 August 2009; accepted 19 August 2009

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.028; wR factor = 0.068; data-to-parameter ratio = 17.3.

In the title compound, $C_{14}H_9I_2N_3O_4\cdot CH_3OH$, the Schiff base molecule adopts an E geometry with respect to the C=N bond and the dihedral angle between the benzene rings is 45.0 (2)°; an intramolecular O—H···N hydrogen bond is present. In the crystal, adjacent Schiff base molecules are linked by methanol solvent molecules through intermolecular N—H···O and O—H···O hydrogen bonds, forming dimers.

Related literature

For a related structure and background, see: Qian & Qu (2009).



Experimental

Crystal data

$C_{14}H_9I_2N_3O_4\cdot CH_4O$
M_r = 569.08
Monoclinic, $C2/c$
a = 19.5041 (12) Å
b = 10.2306 (7) Å
c = 19.9474 (14) Å
β = 111.764 (4)°
V = 3696.6 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 3.43 mm^{-1}
T = 298 K
0.20 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.547, T_{max} = 0.577
11057 measured reflections
4015 independent reflections
3394 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.068
S = 1.11
4015 reflections
232 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −1.02 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.88	2.599 (3)	146
O5—H5···O2^{i}	0.82	1.91	2.711 (4)	164
N2—H2···O5^{ii}	0.891 (10)	1.985 (12)	2.870 (3)	172 (4)

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5054).

References

Bruker (2001). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Qian, H.-Y. & Qu, D.-P. (2009). *Acta Cryst.* E**65**, o2237.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2009). E**65**, o2238 [doi:10.1107/S1600536809033091]

(*E*)-*N*′-(2-Hydroxy-3,5-diiodobenzylidene)-2-nitrobenzohydrazide methanol solvate

Heng-Yu Qian and Da-Ping Qu

S1. Comment

As part of our ongoing studies of Schiff bases (Qian & Qu, 2009), we now report the synthsis and structure of the title compound, (I), (Fig. 1).

The Schiff base molecule adopts an *E* geometry with respect to the C=N bond, and there forms an intramolecular O—H···N hydrogen bond. The two benzene rings forms a dihedral angle of 45.0 (2)°. The dihedral angle between the O3/N3/O4 plane and the C9—C14 benzene ring is 39.2 (2)°. In the crystal structure, the adjacent two Schiff base molecules are linked by a methanol molecule through intermolecular N—H···O and O—H···O hydrogen bonds (Table 1) to form a dimer (Fig. 2).

S2. Experimental

2-Nitrobenzohydrazide (1 mmol, 0.181 g) and 3,5-diiodosalicylaldehyde (1 mmol, 0.374 g) were dissolved in anhydrous methanol (15 ml). The mixture was stirred for several minutes at room temperature. The product was isolated and recrystallized from methanol, colorless blocks of (I) were obtained after 3 days.

S3. Refinement

The imino H atom was located in a difference map and its positional parameters were refined with a fixed isotropic thermal parameter of 0.08 Å^2. Other H atoms were positioned geometrically and refined as riding with C—H = 0.93 Å (aromatic) and 0.96 Å (methyl), O—H = 0.82 å, and with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(C15 and O).

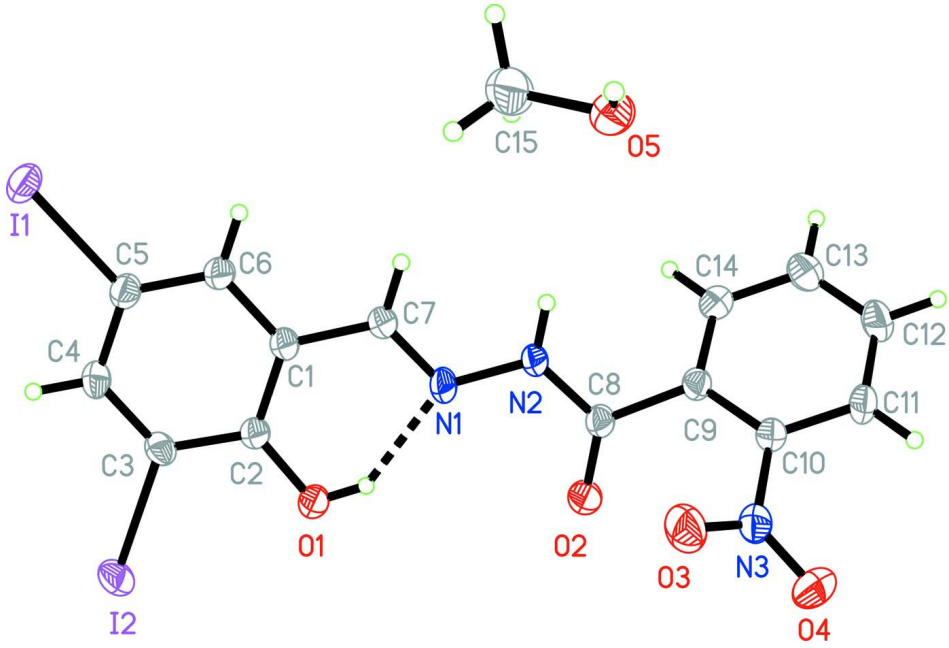


Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. Hydrogen bonding is shown by dashed lines.

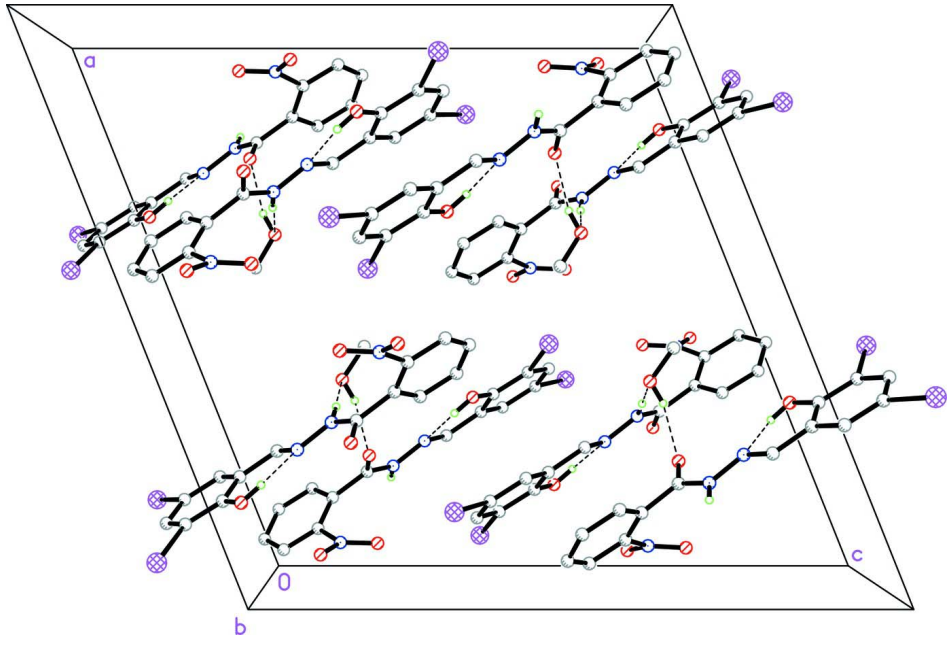


Figure 2

The molecular packing of the title compound, viewed along the *b* axis. Hydrogen bonding is shown in dashed lines.

(*E*)-*N*′-(2-Hydroxy-3,5-diiodobenzylidene)-2-nitrobenzohydrazide methanol solvate

Crystal data

$C_{14}H_9I_2N_3O_4 \cdot CH_4O$
$M_r = 569.08$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 19.5041\ (12)$ Å
$b = 10.2306\ (7)$ Å
$c = 19.9474\ (14)$ Å
$\beta = 111.764\ (4)°$
$V = 3696.6\ (4)$ Å^3
$Z = 8$
$F(000) = 2160$
$D_x = 2.045$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5751 reflections
$\theta = 2.5–30.0°$
$\mu = 3.43$ mm^{-1}
$T = 298$ K
Block, colorless
0.20 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.547$, $T_{max} = 0.577$
11057 measured reflections
4015 independent reflections
3394 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.2°$
$h = -24 \rightarrow 22$
$k = -13 \rightarrow 10$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.068$
$S = 1.11$
4015 reflections
232 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0318P)^2 + 0.9087P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -1.02$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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}
I1	0.143857 (13)	0.30042 (2)	−0.138167 (11)	0.05559 (9)
I2	0.067514 (12)	0.86426 (2)	−0.123056 (11)	0.05414 (9)
N1	0.25171 (13)	0.6839 (2)	0.15211 (12)	0.0419 (6)

N2	0.29927 (14)	0.6742 (2)	0.22329 (12)	0.0418 (6)
N3	0.42504 (16)	0.9532 (3)	0.35060 (14)	0.0540 (7)
O1	0.16625 (13)	0.8109 (2)	0.03922 (11)	0.0512 (6)
H1	0.1961	0.8028	0.0807	0.077*
O2	0.27120 (13)	0.8790 (2)	0.24931 (11)	0.0546 (6)
O3	0.42749 (16)	0.9390 (3)	0.29089 (12)	0.0736 (8)
O4	0.4402 (2)	1.0549 (3)	0.38515 (16)	0.0940 (10)
O5	0.63153 (13)	0.4225 (2)	0.24221 (12)	0.0578 (6)
H5	0.6700	0.4074	0.2360	0.087*
C1	0.19975 (15)	0.5847 (3)	0.03648 (14)	0.0380 (6)
C2	0.16300 (16)	0.6994 (3)	0.00277 (14)	0.0369 (6)
C3	0.12219 (15)	0.6956 (3)	−0.07147 (14)	0.0385 (6)
C4	0.11739 (16)	0.5834 (3)	−0.11097 (14)	0.0413 (7)
H4	0.0900	0.5829	−0.1603	0.050*
C5	0.15337 (16)	0.4712 (3)	−0.07701 (15)	0.0425 (7)
C6	0.19422 (16)	0.4716 (3)	−0.00401 (14)	0.0417 (7)
H6	0.2183	0.3959	0.0184	0.050*
C7	0.24533 (16)	0.5819 (3)	0.11310 (14)	0.0415 (7)
H7	0.2700	0.5056	0.1338	0.050*
C8	0.30522 (15)	0.7759 (3)	0.26755 (14)	0.0385 (6)
C9	0.35442 (16)	0.7502 (3)	0.34453 (14)	0.0377 (6)
C10	0.40679 (17)	0.8399 (3)	0.38486 (15)	0.0410 (6)
C11	0.4469 (2)	0.8220 (3)	0.45769 (16)	0.0533 (8)
H11	0.4814	0.8838	0.4839	0.064*
C12	0.4346 (2)	0.7113 (4)	0.49031 (17)	0.0621 (10)
H12	0.4614	0.6974	0.5391	0.075*
C13	0.3837 (2)	0.6215 (4)	0.45209 (17)	0.0640 (10)
H13	0.3760	0.5469	0.4750	0.077*
C14	0.34329 (19)	0.6404 (3)	0.37922 (16)	0.0491 (8)
H14	0.3085	0.5786	0.3536	0.059*
C15	0.5718 (2)	0.3661 (4)	0.1857 (2)	0.0800 (13)
H15A	0.5270	0.3806	0.1940	0.120*
H15B	0.5800	0.2739	0.1838	0.120*
H15C	0.5678	0.4054	0.1407	0.120*
H2	0.324 (2)	0.600 (2)	0.237 (2)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.06140 (16)	0.04922 (14)	0.04793 (13)	−0.00025 (10)	0.01070 (11)	−0.01635 (9)
I2	0.05556 (15)	0.05314 (15)	0.04538 (13)	0.01490 (9)	0.00901 (10)	0.01138 (9)
N1	0.0382 (13)	0.0469 (14)	0.0291 (11)	0.0020 (10)	−0.0010 (10)	−0.0033 (10)
N2	0.0445 (14)	0.0384 (13)	0.0290 (11)	0.0081 (11)	−0.0021 (10)	−0.0019 (10)
N3	0.0567 (17)	0.0532 (17)	0.0405 (14)	−0.0102 (13)	0.0046 (12)	0.0001 (12)
O1	0.0615 (15)	0.0400 (12)	0.0383 (11)	0.0108 (10)	0.0024 (10)	−0.0057 (9)
O2	0.0569 (14)	0.0436 (13)	0.0481 (12)	0.0149 (10)	0.0019 (10)	−0.0024 (10)
O3	0.089 (2)	0.0819 (19)	0.0490 (14)	−0.0147 (15)	0.0246 (14)	0.0089 (13)
O4	0.139 (3)	0.0533 (17)	0.0708 (17)	−0.0332 (17)	0.0174 (18)	−0.0079 (14)

O5	0.0566 (14)	0.0457 (13)	0.0594 (13)	−0.0042 (11)	0.0080 (11)	−0.0033 (11)
C1	0.0361 (15)	0.0392 (15)	0.0316 (13)	0.0014 (12)	0.0042 (11)	−0.0010 (11)
C2	0.0370 (15)	0.0358 (15)	0.0341 (13)	0.0011 (11)	0.0087 (12)	−0.0009 (11)
C3	0.0348 (15)	0.0436 (16)	0.0343 (13)	0.0068 (12)	0.0095 (12)	0.0049 (11)
C4	0.0363 (15)	0.0511 (18)	0.0315 (13)	−0.0004 (12)	0.0068 (11)	−0.0037 (12)
C5	0.0426 (16)	0.0430 (16)	0.0368 (14)	−0.0011 (13)	0.0088 (12)	−0.0069 (12)
C6	0.0419 (16)	0.0368 (15)	0.0379 (14)	0.0019 (12)	0.0051 (12)	−0.0026 (12)
C7	0.0436 (16)	0.0370 (15)	0.0339 (14)	0.0039 (12)	0.0029 (12)	−0.0002 (12)
C8	0.0338 (14)	0.0400 (16)	0.0343 (13)	0.0025 (12)	0.0041 (11)	−0.0033 (11)
C9	0.0405 (15)	0.0387 (15)	0.0316 (13)	0.0051 (12)	0.0107 (11)	−0.0038 (12)
C10	0.0454 (17)	0.0392 (16)	0.0336 (13)	0.0016 (12)	0.0090 (12)	−0.0024 (12)
C11	0.061 (2)	0.0546 (19)	0.0320 (14)	0.0020 (16)	0.0024 (14)	−0.0109 (14)
C12	0.082 (3)	0.066 (2)	0.0297 (15)	0.0082 (19)	0.0103 (16)	0.0026 (15)
C13	0.090 (3)	0.057 (2)	0.0406 (17)	−0.0001 (19)	0.0190 (18)	0.0088 (15)
C14	0.059 (2)	0.0411 (17)	0.0441 (16)	−0.0029 (14)	0.0157 (15)	−0.0033 (13)
C15	0.074 (3)	0.058 (2)	0.074 (3)	−0.0091 (19)	−0.012 (2)	0.0031 (19)

Geometric parameters (Å, º)

I1—C5	2.099 (3)	C4—C5	1.384 (4)
I2—C3	2.088 (3)	C4—H4	0.9300
N1—C7	1.280 (4)	C5—C6	1.377 (4)
N1—N2	1.382 (3)	C6—H6	0.9300
N2—C8	1.341 (4)	C7—H7	0.9300
N2—H2	0.891 (10)	C8—C9	1.502 (4)
N3—O3	1.218 (3)	C9—C14	1.378 (4)
N3—O4	1.222 (4)	C9—C10	1.386 (4)
N3—C10	1.456 (4)	C10—C11	1.383 (4)
O1—C2	1.341 (3)	C11—C12	1.371 (5)
O1—H1	0.8200	C11—H11	0.9300
O2—C8	1.227 (3)	C12—C13	1.360 (5)
O5—C15	1.410 (4)	C12—H12	0.9300
O5—H5	0.8200	C13—C14	1.386 (4)
C1—C6	1.392 (4)	C13—H13	0.9300
C1—C2	1.409 (4)	C14—H14	0.9300
C1—C7	1.456 (4)	C15—H15A	0.9600
C2—C3	1.398 (4)	C15—H15B	0.9600
C3—C4	1.376 (4)	C15—H15C	0.9600
C7—N1—N2	116.3 (2)	C1—C7—H7	119.8
C8—N2—N1	118.8 (2)	O2—C8—N2	124.5 (3)
C8—N2—H2	124 (3)	O2—C8—C9	121.6 (3)
N1—N2—H2	117 (3)	N2—C8—C9	113.8 (2)
O3—N3—O4	124.4 (3)	C14—C9—C10	117.8 (3)
O3—N3—C10	117.9 (3)	C14—C9—C8	119.7 (3)
O4—N3—C10	117.6 (3)	C10—C9—C8	122.1 (3)
C2—O1—H1	109.5	C11—C10—C9	122.0 (3)
C15—O5—H5	109.5	C11—C10—N3	117.2 (3)

C6—C1—C2	119.9 (2)	C9—C10—N3	120.7 (2)
C6—C1—C7	118.6 (3)	C12—C11—C10	118.5 (3)
C2—C1—C7	121.5 (3)	C12—C11—H11	120.8
O1—C2—C3	119.5 (2)	C10—C11—H11	120.8
O1—C2—C1	122.4 (2)	C13—C12—C11	120.8 (3)
C3—C2—C1	118.1 (3)	C13—C12—H12	119.6
C4—C3—C2	121.4 (3)	C11—C12—H12	119.6
C4—C3—I2	119.6 (2)	C12—C13—C14	120.4 (3)
C2—C3—I2	119.0 (2)	C12—C13—H13	119.8
C3—C4—C5	119.8 (2)	C14—C13—H13	119.8
C3—C4—H4	120.1	C9—C14—C13	120.4 (3)
C5—C4—H4	120.1	C9—C14—H14	119.8
C6—C5—C4	120.3 (3)	C13—C14—H14	119.8
C6—C5—I1	120.6 (2)	O5—C15—H15A	109.5
C4—C5—I1	119.09 (19)	O5—C15—H15B	109.5
C5—C6—C1	120.4 (3)	H15A—C15—H15B	109.5
C5—C6—H6	119.8	O5—C15—H15C	109.5
C1—C6—H6	119.8	H15A—C15—H15C	109.5
N1—C7—C1	120.4 (3)	H15B—C15—H15C	109.5
N1—C7—H7	119.8		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O2^{i}	0.82	1.91	2.711 (4)	164
O1—H1···N1	0.82	1.88	2.599 (3)	146
N2—H2···O5^{ii}	0.89 (1)	1.99 (1)	2.870 (3)	172 (4)

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