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N′-[(*E*)-2-Hydroxy-3,5-diiodobenzylidene]cyclohexane-1-carbohydrazide

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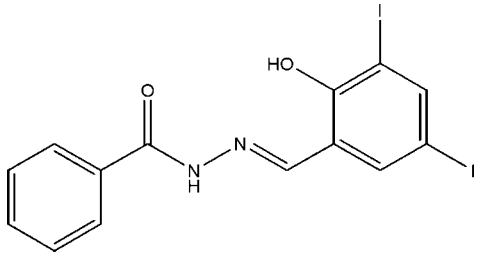
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.018 Å; R factor = 0.058; wR factor = 0.172; data-to-parameter ratio = 14.9.

In the title compound, $C_{14}H_{10}I_2N_2O_2$, the two aromatic rings are inclined at a dihedral angle of 16.72 (33)°. The molecular structure is stabilized by an intramolecular O—H···N hydrogen bond. In the crystal, intermolecular N—H···O interactions link the molecules into chains running along the c axis. C—H···O interactions also occur. The crystal used for the structure determination was a non-merohedral twin with a domain ratio of 0.972 (2):0.028 (2).

Related literature

For the biological activity of Schiff base derivatives, see: Daier *et al.* (2004); Golcu *et al.* (2005); Liu & Yang (2010); Zgierski & Grabowska (2000). For related structures, see: Manvizhi *et al.* (2011); Thirugnanasundar *et al.* (2011).



Experimental

Crystal data

$C_{14}H_{10}I_2N_2O_2$
M_r = 492.04
Monoclinic, $P2_1/c$
a = 17.7495 (13) Å
b = 9.4273 (6) Å
c = 9.4684 (7) Å
β = 103.052 (3)°
V = 1543.42 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.08 mm^{-1}
T = 295 K
0.20 × 0.10 × 0.10 mm

Data collection

Bruker Kappa APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.496, T_{max} = 0.686
13146 measured reflections
2708 independent reflections
2349 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.172
S = 1.11
2708 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.48 e Å^{-3}
$\Delta\rho_{min}$ = −1.52 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···N2	0.85	1.84	2.586 (13)	145
N1—H1···O1^{i}	0.86	2.02	2.824 (14)	155
C3—H3···O2^{ii}	0.93	2.53	3.365 (19)	150

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

The authors wish to acknowledge the SAIF, IIT, Madras, for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5637).

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supporting information

Acta Cryst. (2011). E**67**, o2620 [https://doi.org/10.1107/S1600536811036488]

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S1. Comment

Schiff base ligands play an important role in the development of coordination chemistry and possess important properties such as biological activity (Golcu *et al.*, 2005; Liu & Yang, 2010), catalytic activity (Daier *et al.*, 2004) and photochromic properties (Zgierski & Grabowska, 2000).

The goemetric parameters of the title compound (Fig. 1) agree well with related structures (Manvizhi *et al.*, 2011; Thirugnanasundar *et al.*, 2011). The two aromatic rings are inclined at an angle of 16.72 (33)°.

The molecular structure is stabilized by an intramolecular O-H···N hydrogen bond. The crystal structure is controlled by intermolecular N-H···O and C—H···O (Fig. 2 & Table 1) interactions.

S2. Experimental

A methanolic solution (10 ml) of benzoicacid hydrazide (5 mmol) was magnetically stirred in a round bottom flask followed by drop wise addition of methanolic solution of 3,5-diiodosalicylaldehyde (5 mmol). The reaction mixture was then refluxed for three hours and upon cooling to 278 K, a yellow crystalline solid precipitates from the mixture. The solid which is separated out was filtered washed with ice cold ethanol and dried in vaccuo over anhydrous $CaCl_2$. Single crystals suitable for the X-ray diffraction were obtained by slow evaporation of a solution of the title compound in DMF at room temperature. Melting Point : 485 K.

S3. Refinement

All H atoms were positioned geometrically with C—H = 0.93Å, O—H = 0.82 Å and N—H = 0.86 Å and allowed to ride on their parent atoms, with Uiso(H) = 1.5 Ueq(O), 1.2 Ueq(N) and 1.2 Ueq(C). Initial checkCIF/PLATON results indicated possible twinning; introduction of the suggested command TWIN 1 0 0.85 0 -1 0 0 0 -1 2 during refinement gave a very modest decrease in the R-factor, from 0.0743 to 0.0584, with BASF = 0.028 (2).

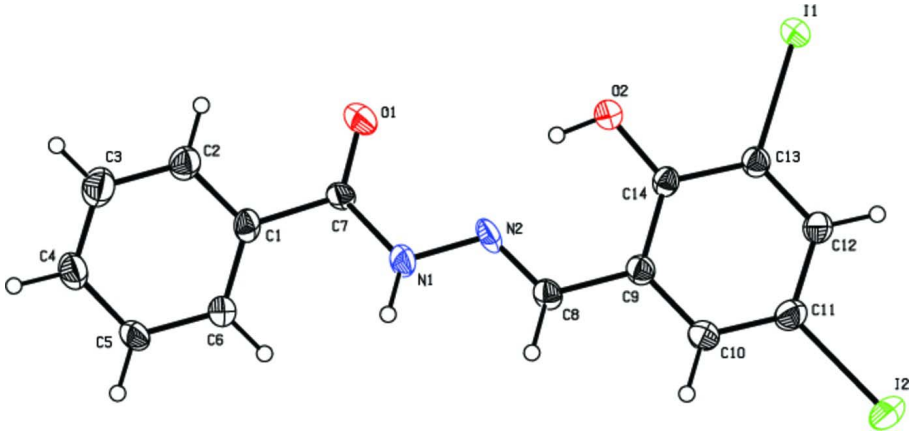

Figure 1

The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms.

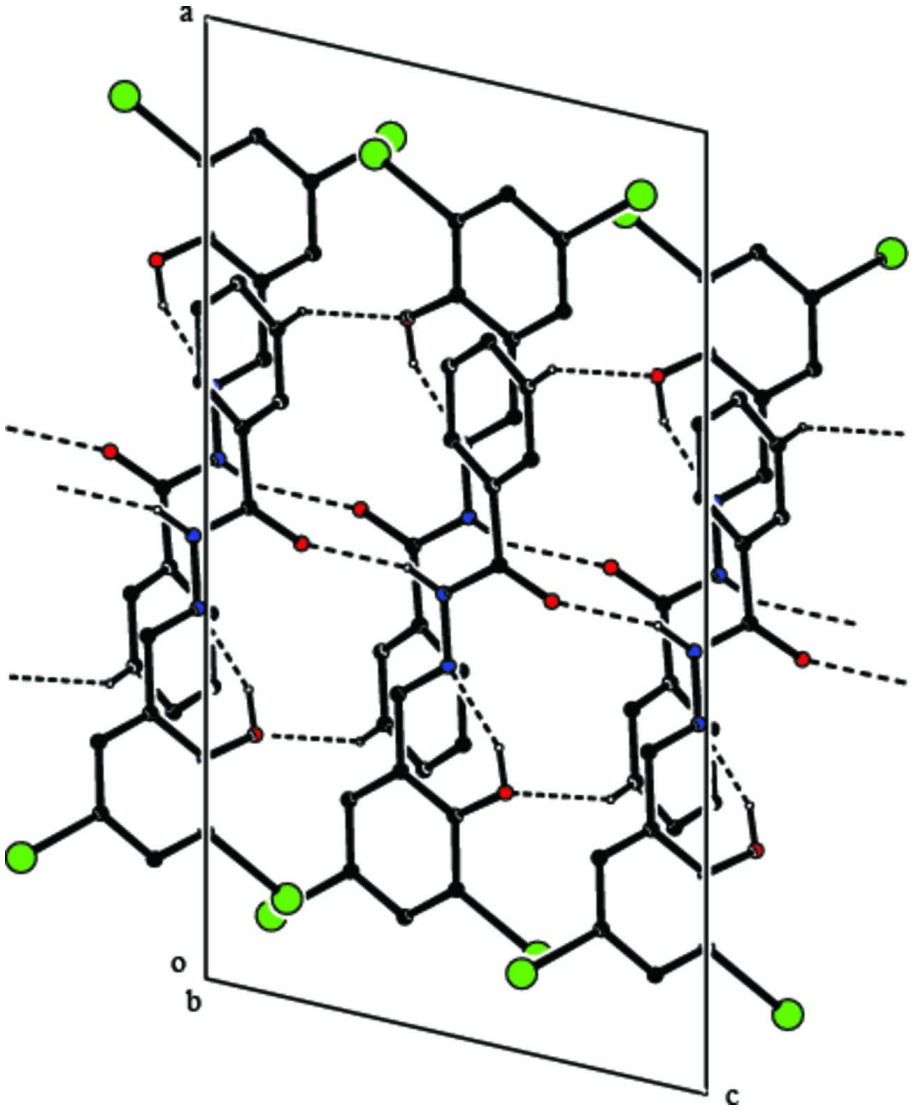

Figure 2

The packing of (I), viewed down *b* axis. Intermolecular Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

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Hall symbol: -P 2ybc
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$c = 9.4684\ (7)$ Å
$\beta = 103.052\ (3)°$
$V = 1543.42\ (19)$ Å^3
$Z = 4$
$F(000) = 920$
$D_x = 2.118$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 13146 reflections
$\theta = 2.2–25.0°$
$\mu = 4.08$ mm^{-1}
$T = 295$ K
Block, yellow
0.20 × 0.10 × 0.10 mm

Data collection

Bruker Kappa APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.496, T_{max} = 0.686
13146 measured reflections
2708 independent reflections
2349 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 25.0°, θ_{min} = 2.2°
h = −21→21
k = −11→8
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.172
S = 1.11
2708 reflections
182 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0412P)^2 + 42.3729P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.48 e Å^{-3}
$\Delta\rho_{min}$ = −1.52 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.89740 (5)	0.16968 (10)	0.83785 (10)	0.0458 (3)
I2	0.91863 (7)	−0.18253 (14)	1.36905 (13)	0.0777 (5)
O1	0.5247 (5)	0.3352 (11)	0.8089 (10)	0.052 (3)
O2	0.7346 (5)	0.2255 (10)	0.9018 (10)	0.044 (2)
H2A	0.6895	0.2404	0.9145	0.065*
N1	0.5428 (6)	0.2182 (13)	1.0250 (11)	0.043 (3)
H1	0.5244	0.1906	1.0969	0.052*
N2	0.6169 (6)	0.1863 (12)	1.0158 (11)	0.040 (3)
C1	0.4189 (7)	0.3296 (14)	0.9272 (12)	0.036 (3)
C2	0.3821 (7)	0.4436 (15)	0.8509 (13)	0.043 (3)
H2	0.4075	0.4988	0.7950	0.052*
C3	0.3079 (9)	0.4755 (18)	0.8577 (16)	0.057 (4)
H3	0.2836	0.5541	0.8082	0.068*
C4	0.2694 (8)	0.3939 (16)	0.9356 (15)	0.047 (3)
H4	0.2185	0.4156	0.9369	0.057*
C5	0.3052 (7)	0.2777 (15)	1.0138 (13)	0.041 (3)
H5	0.2790	0.2217	1.0678	0.049*
C6	0.3807 (7)	0.2479 (15)	1.0092 (14)	0.041 (3)
H6	0.4060	0.1719	1.0621	0.049*
C7	0.4996 (7)	0.2964 (14)	0.9137 (11)	0.035 (3)
C8	0.6579 (7)	0.1120 (14)	1.1159 (12)	0.036 (3)
H8	0.6375	0.0800	1.1924	0.043*
C9	0.7375 (7)	0.0770 (13)	1.1107 (13)	0.034 (3)
C10	0.7797 (7)	−0.0131 (15)	1.2142 (14)	0.042 (3)
H10	0.7576	−0.0486	1.2872	0.051*

C11	0.8534 (8)	−0.0503 (15)	1.2102 (14)	0.043 (3)
C12	0.8881 (7)	0.0029 (13)	1.1027 (14)	0.040 (3)
H12	0.9385	−0.0225	1.1010	0.048*
C13	0.8471 (7)	0.0932 (13)	0.9997 (13)	0.034 (3)
C14	0.7710 (6)	0.1333 (13)	1.0022 (12)	0.031 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0346 (4)	0.0620 (6)	0.0442 (5)	0.0048 (4)	0.0159 (4)	0.0154 (4)
I2	0.0733 (8)	0.0933 (9)	0.0714 (8)	0.0406 (7)	0.0266 (6)	0.0484 (7)
O1	0.041 (5)	0.074 (7)	0.047 (6)	0.002 (5)	0.021 (4)	0.010 (5)
O2	0.034 (4)	0.048 (5)	0.051 (5)	0.007 (4)	0.015 (4)	0.015 (4)
N1	0.032 (5)	0.064 (7)	0.036 (6)	0.015 (5)	0.015 (4)	0.005 (5)
N2	0.024 (5)	0.058 (7)	0.042 (6)	0.002 (5)	0.017 (4)	−0.006 (5)
C1	0.026 (6)	0.053 (8)	0.025 (6)	0.003 (5)	−0.001 (5)	−0.006 (5)
C2	0.041 (7)	0.054 (8)	0.034 (7)	0.006 (6)	0.006 (6)	0.000 (6)
C3	0.052 (9)	0.066 (10)	0.051 (8)	0.018 (8)	0.010 (7)	0.002 (8)
C4	0.036 (7)	0.055 (9)	0.054 (8)	0.007 (6)	0.016 (6)	−0.002 (7)
C5	0.035 (6)	0.059 (8)	0.033 (6)	0.003 (6)	0.016 (5)	0.003 (6)
C6	0.034 (6)	0.046 (8)	0.046 (7)	0.008 (6)	0.013 (6)	0.010 (6)
C7	0.031 (6)	0.057 (8)	0.017 (5)	0.001 (5)	0.007 (4)	0.001 (5)
C8	0.034 (6)	0.052 (8)	0.023 (6)	0.005 (6)	0.011 (5)	−0.005 (5)
C9	0.034 (6)	0.039 (7)	0.031 (6)	0.003 (5)	0.010 (5)	−0.002 (5)
C10	0.042 (7)	0.053 (8)	0.036 (7)	0.007 (6)	0.016 (6)	0.006 (6)
C11	0.046 (7)	0.048 (8)	0.034 (6)	0.007 (6)	0.008 (6)	0.006 (6)
C12	0.042 (7)	0.037 (7)	0.044 (7)	0.011 (6)	0.016 (6)	0.004 (5)
C13	0.033 (6)	0.037 (7)	0.032 (6)	0.003 (5)	0.008 (5)	−0.002 (5)
C14	0.031 (6)	0.033 (6)	0.028 (6)	0.006 (5)	0.006 (5)	−0.003 (5)

Geometric parameters (Å, º)

I1—C13	2.070 (12)	C4—C5	1.393 (19)
I2—C11	2.090 (13)	C4—H4	0.9300
O1—C7	1.231 (14)	C5—C6	1.379 (17)
O2—C14	1.342 (14)	C5—H5	0.9300
O2—H2A	0.8480	C6—H6	0.9300
N1—C7	1.370 (15)	C8—C9	1.463 (16)
N1—N2	1.370 (13)	C8—H8	0.9300
N1—H1	0.8600	C9—C10	1.382 (17)
N2—C8	1.268 (16)	C9—C14	1.402 (16)
C1—C2	1.375 (18)	C10—C11	1.363 (18)
C1—C6	1.376 (18)	C10—H10	0.9300
C1—C7	1.500 (16)	C11—C12	1.397 (18)
C2—C3	1.366 (19)	C12—C13	1.373 (17)
C2—H2	0.9300	C12—H12	0.9300
C3—C4	1.35 (2)	C13—C14	1.409 (16)
C3—H3	0.9300		

C14—O2—H2A	108.9	O1—C7—C1	122.2 (11)
C7—N1—N2	116.6 (10)	N1—C7—C1	115.2 (10)
C7—N1—H1	121.7	N2—C8—C9	119.7 (11)
N2—N1—H1	121.7	N2—C8—H8	120.1
C8—N2—N1	118.1 (10)	C9—C8—H8	120.1
C2—C1—C6	119.8 (11)	C10—C9—C14	120.1 (11)
C2—C1—C7	117.9 (12)	C10—C9—C8	119.2 (11)
C6—C1—C7	122.2 (11)	C14—C9—C8	120.8 (11)
C3—C2—C1	119.8 (14)	C11—C10—C9	120.5 (12)
C3—C2—H2	120.1	C11—C10—H10	119.7
C1—C2—H2	120.1	C9—C10—H10	119.7
C4—C3—C2	120.7 (14)	C10—C11—C12	120.8 (12)
C4—C3—H3	119.6	C10—C11—I2	120.9 (10)
C2—C3—H3	119.6	C12—C11—I2	118.3 (9)
C3—C4—C5	120.7 (13)	C13—C12—C11	119.4 (11)
C3—C4—H4	119.6	C13—C12—H12	120.3
C5—C4—H4	119.6	C11—C12—H12	120.3
C6—C5—C4	118.3 (12)	C12—C13—C14	120.7 (11)
C6—C5—H5	120.9	C12—C13—I1	119.6 (9)
C4—C5—H5	120.9	C14—C13—I1	119.7 (9)
C1—C6—C5	120.7 (12)	O2—C14—C9	123.3 (10)
C1—C6—H6	119.7	O2—C14—C13	118.1 (10)
C5—C6—H6	119.7	C9—C14—C13	118.5 (11)
O1—C7—N1	122.6 (11)		

C7—N1—N2—C8	−178.8 (12)	N2—C8—C9—C14	4.9 (19)
C6—C1—C2—C3	0 (2)	C14—C9—C10—C11	−1 (2)
C7—C1—C2—C3	178.5 (12)	C8—C9—C10—C11	178.5 (13)
C1—C2—C3—C4	−2 (2)	C9—C10—C11—C12	1 (2)
C2—C3—C4—C5	2 (2)	C9—C10—C11—I2	178.5 (10)
C3—C4—C5—C6	0 (2)	C10—C11—C12—C13	0 (2)
C2—C1—C6—C5	1 (2)	I2—C11—C12—C13	−178.1 (10)
C7—C1—C6—C5	−177.0 (12)	C11—C12—C13—C14	0.5 (19)
C4—C5—C6—C1	−1 (2)	C11—C12—C13—I1	−178.9 (10)
N2—N1—C7—O1	1.0 (19)	C10—C9—C14—O2	−177.3 (12)
N2—N1—C7—C1	−179.9 (11)	C8—C9—C14—O2	2.8 (18)
C2—C1—C7—O1	−22.7 (18)	C10—C9—C14—C13	1.5 (18)
C6—C1—C7—O1	155.4 (13)	C8—C9—C14—C13	−178.4 (11)
C2—C1—C7—N1	158.2 (12)	C12—C13—C14—O2	177.8 (11)
C6—C1—C7—N1	−23.7 (18)	I1—C13—C14—O2	−2.8 (15)
N1—N2—C8—C9	−179.4 (11)	C12—C13—C14—C9	−1.1 (18)
N2—C8—C9—C10	−175.0 (13)	I1—C13—C14—C9	178.3 (9)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···N2	0.85	1.84	2.586 (13)	145

N1—H1···O1^{i}	0.86	2.02	2.824 (14)	155
C3—H3···O2^{ii}	0.93	2.53	3.365 (19)	150

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