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(*E*)-4-Iodo-2-[(phenylimino)methyl]phenol

G. Swetha,[a] R. Ida Malarselvi,[b] C. Ramachandra Raja,[a] A. Thiruvalluvar[c]* and J. Priscilla[d]

[a]Postgraduate and Research Department of Physics, Government Arts College (Autonomous), Kumbakonam 612 001, Tamilnadu, India, [b]Postgraduate and Research Department of Physics, D.G. Government Arts College for Women, Mayiladuthurai 609 001, Tamilnadu, India, [c]Principal, Kunthavai Naacchiyaar Government Arts College for Women (Autonomous), Thanjavur 613 007, Tamilnadu, India, and [d]Postgraduate Department of Physics, A.D.M. College for Women (Autonomous), Nagapattinam 611 001, Tamilnadu, India. *Correspondence e-mail: thiruvalluvar.a@gmail.com

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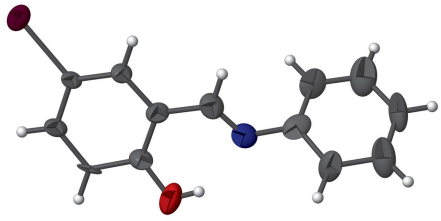
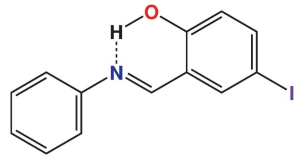
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{13}H_{10}INO$, is not planar as the dihedral angle between the planes of the two aryl rings is 44.5 (9)°. The configuration about the central C=N bond is *E*, and there is an intramolecular O—H···N hydrogen bond which generates an *S*(6) ring. The molecular packing is stabilized by weak C—H···π interactions. The structure was refined as a two-component inversion twin.

3D view



Chemical scheme



Structure description

We report here, as part of our on-going research (Ida Malarselvi *et al.*, 2016; Swetha *et al.*, 2017, 2018), the synthesis and X-ray crystal structure determination of the title iodinated Schiff base compound, Fig. 1, which was synthesized from the condensation reaction of equimolar amounts of 5-iodosalicylaldehyde and aniline in DMSO.

The benzene and phenyl rings deviate from co-planarity with the dihedral angle between the two ring being 44.5 (9)°. The molecule has an *E* configuration about the C=N bond, and the C1—C7=N1—C8 torsion angle is 169.5 (17)°. There is a strong intra-molecular O1—H1···N1 hydrogen bond, Table 1, with an H···N separation of 1.94 Å which leads to an *S*(6) ring. The crystal structure (Fig. 2) is stabilized by three weak C—H···π interactions, see Table 1.

Yan *et al.* (2014) have reported the crystal structure determination of 4-bromo-2-[(phenylimino)methyl]phenol, in which the molecule is essentially planar (r.m.s. deviation = 0.026 Å), a result in contrast to our present study.

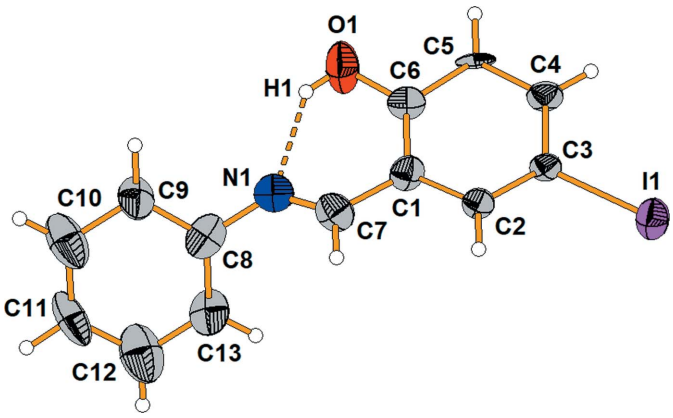


Figure 1
A view of the molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level and showing the atom-numbering scheme. Dashed lines indicate the intramolecular hydrogen-bonding interaction (Table 1).

Synthesis and crystallization

5-Iodosalicylaldehyde (0.3 g) was dissolved in DMSO (15 ml). To this solution, aniline (0.2 g) was added dropwise with constant stirring for 1 h at 50°C. During this time, the solution turned light yellow. On standing for 1 month with slow evaporation of the solvent, light-orange crystals of the title compound suitable for the X-ray study were obtained.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The structure was refined as a two-component inversion twin.

Acknowledgements

The authors are grateful to the Sophisticated Analytical Instrument Facility (SAIF), IITM, Chennai 600 036, Tamilnadu, India, for the single-crystal X-ray diffraction data.

References

Brandenburg, K. & Putz, H. (2018). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.

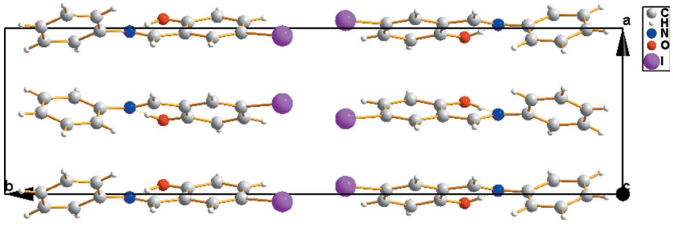


Figure 2
The molecular packing, viewed along the crystallographic *c* axis.

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

*Cg*1 and *Cg*2 are the centroids of the C1–C6 benzene ring and the C8–C13 phenyl ring, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.94	2.64 (2)	143
C5—H5···*Cg*1^{i}	0.93	2.86	3.476 (15)	125
C9—H9···*Cg*2^{ii}	0.93	2.81	3.48 (2)	129
C12—H12···*Cg*2^{iii}	0.93	2.82	3.55 (3)	136

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{10}INO$
M_r	323.12
Crystal system, space group	Orthorhombic, $Pca2_1$
Temperature (K)	296
a, *b*, *c* (Å)	7.0848 (8), 26.422 (3), 6.2664 (7)
V (Å^3)	1173.1 (2)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	2.71
Crystal size (mm)	0.30 × 0.25 × 0.15
Data collection	
Diffractometer	Bruker Kappa APEX3 CMOS
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.287, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13051, 2057, 2043
R_{int}	0.055
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.080, 0.223, 1.18
No. of reflections	2057
No. of parameters	146
No. of restraints	148
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	2.90, −1.22
Absolute structure	Refined as an inversion twin.
Absolute structure parameter	0.53 (13)

Computer programs: *APEX3*, *SAINT* and *XPREP* (Bruker, 2016), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2018), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

Bruker (2016). *APEX3*, *SAINT*, *XPREP* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Ida Malarselvi, R., Ramachandra Raja, C., Thiruvalluvar, A., Priscilla, J. & Panneer Selvam, K. (2016). *IUCrData*, **1**, x161595.
Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Swetha, G., Ida Malarselvi, R., Ramachandra Raja, C., Thiruvalluvar, A. & Priscilla, J. (2017). *IUCrData*, **2**, x171671.
Swetha, G., Ida Malarselvi, R., Ramachandra Raja, C., Thiruvalluvar, A. & Priscilla, J. (2018). *IUCrData*, **3**, x180464.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Yan, X.-X., Lu, L.-P. & Zhu, M.-L. (2014). *Acta Cryst.* E**70**, o853.

full crystallographic data

IUCrData (2019). **4**, x190788 [https://doi.org/10.1107/S2414314619007880]

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(*E*)-4-Iodo-2-[(phenylimino)methyl]phenol

Crystal data

$C_{13}H_{10}INO$
$M_r = 323.12$
Orthorhombic, $Pca2_1$
$a = 7.0848$ (8) Å
$b = 26.422$ (3) Å
$c = 6.2664$ (7) Å
$V = 1173.1$ (2) Å^3
$Z = 4$
$F(000) = 624$
$D_x = 1.830$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9900 reflections
$\theta = 2.9$–$30.8°$
$\mu = 2.71$ mm^{-1}
$T = 296$ K
Plate, orange
0.30 × 0.25 × 0.15 mm

Data collection

Bruker Kappa APEX3 CMOS diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scan
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)
$T_{min} = 0.287$, $T_{max} = 0.746$
13051 measured reflections
2057 independent reflections
2043 reflections with $I > 2\sigma(I)$
$R_{int} = 0.055$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.0°$
$h = -8 \rightarrow 8$
$k = -31 \rightarrow 31$
$l = -7 \rightarrow 7$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.080$
$wR(F^2) = 0.223$
$S = 1.18$
2057 reflections
146 parameters
148 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1256P)^2 + 10.5304P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 2.90$ e Å^{-3}
$\Delta\rho_{min} = -1.22$ e Å^{-3}
Absolute structure: Refined as an inversion twin.
Absolute structure parameter: 0.53 (13)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refined as a two-component inversion twin. C-bound H atoms were placed in geometrically idealized positions with C—H = 0.93 Å. The OH H1 atom was placed geometrically with O—H = 0.82 Å.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.528 (2)	0.7086 (7)	0.466 (3)	0.036 (3)
C2	0.557 (2)	0.6640 (6)	0.586 (3)	0.031 (3)
H2	0.598694	0.666661	0.725680	0.038*
C3	0.526 (2)	0.6174 (6)	0.503 (2)	0.027 (3)
C4	0.461 (2)	0.6133 (7)	0.289 (3)	0.035 (4)
H4	0.439214	0.581274	0.232509	0.042*
C5	0.429 (2)	0.6565 (6)	0.1556 (19)	0.034 (4)
H5	0.388597	0.653661	0.015024	0.040*
C6	0.465 (2)	0.7045 (7)	0.255 (2)	0.035 (3)
C7	0.551 (2)	0.7588 (8)	0.554 (4)	0.044 (4)
H7	0.595451	0.760351	0.693338	0.053*
N1	0.5206 (18)	0.7982 (6)	0.470 (3)	0.038 (3)
O1	0.445 (2)	0.7439 (6)	0.126 (3)	0.063 (5)
H1	0.478128	0.769632	0.188944	0.095*
C8	0.516 (2)	0.8464 (8)	0.565 (3)	0.043 (4)
C9	0.582 (3)	0.8877 (6)	0.444 (4)	0.052 (5)
H9	0.627478	0.883081	0.305929	0.062*
C10	0.577 (4)	0.9366 (9)	0.538 (5)	0.069 (7)
H10	0.628761	0.964479	0.468623	0.083*
C11	0.491 (3)	0.9414 (7)	0.738 (4)	0.063 (6)
H11	0.482026	0.973329	0.799836	0.076*
C12	0.420 (4)	0.9000 (8)	0.847 (5)	0.077 (7)
H12	0.363078	0.904559	0.979138	0.092*
C13	0.431 (2)	0.8520 (7)	0.760 (3)	0.049 (5)
H13	0.382080	0.824147	0.831719	0.059*
I1	0.54634 (16)	0.55120 (4)	0.6950 (5)	0.0477 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.036 (8)	0.039 (6)	0.034 (7)	−0.001 (5)	−0.011 (6)	0.005 (5)
C2	0.033 (8)	0.034 (6)	0.028 (7)	−0.005 (5)	−0.009 (6)	0.000 (5)
C3	0.026 (7)	0.034 (6)	0.021 (6)	−0.007 (5)	−0.008 (5)	−0.001 (5)
C4	0.038 (9)	0.043 (8)	0.024 (6)	−0.004 (6)	−0.009 (6)	−0.001 (6)
C5	0.044 (8)	0.052 (7)	0.004 (8)	0.000 (6)	−0.005 (5)	−0.001 (5)
C6	0.027 (7)	0.045 (7)	0.032 (7)	0.002 (6)	−0.008 (5)	0.000 (5)
C7	0.038 (10)	0.047 (6)	0.047 (10)	−0.003 (6)	−0.010 (7)	−0.006 (5)
N1	0.019 (6)	0.051 (7)	0.044 (8)	−0.002 (6)	−0.008 (6)	−0.001 (6)
O1	0.074 (11)	0.034 (8)	0.082 (14)	0.002 (7)	−0.015 (8)	0.005 (7)
C8	0.019 (7)	0.054 (9)	0.056 (10)	0.004 (7)	−0.009 (7)	0.015 (7)
C9	0.043 (10)	0.039 (7)	0.074 (13)	0.008 (7)	0.028 (9)	−0.002 (7)
C10	0.045 (11)	0.052 (9)	0.111 (17)	0.002 (10)	0.009 (12)	−0.027 (10)
C11	0.044 (9)	0.039 (8)	0.106 (18)	0.008 (7)	0.003 (12)	−0.031 (9)
C12	0.052 (12)	0.058 (9)	0.120 (18)	0.009 (9)	0.009 (12)	−0.012 (10)
C13	0.020 (7)	0.052 (8)	0.074 (13)	−0.009 (7)	0.014 (7)	0.003 (7)

I1	0.0529 (8)	0.0357 (7)	0.0545 (8)	0.0010 (4)	−0.0052 (8)	0.0067 (8)

Geometric parameters (Å, º)

C1—C6	1.402 (18)	N1—C8	1.41 (3)
C1—C2	1.412 (19)	O1—H1	0.8200
C1—C7	1.44 (3)	C8—C13	1.37 (2)
C2—C3	1.353 (19)	C8—C9	1.41 (2)
C2—H2	0.9300	C9—C10	1.42 (2)
C3—C4	1.421 (19)	C9—H9	0.9300
C3—I1	2.130 (16)	C10—C11	1.40 (2)
C4—C5	1.431 (19)	C10—H10	0.9300
C4—H4	0.9300	C11—C12	1.38 (2)
C5—C6	1.433 (19)	C11—H11	0.9300
C5—H5	0.9300	C12—C13	1.38 (2)
C6—O1	1.32 (2)	C12—H12	0.9300
C7—N1	1.19 (3)	C13—H13	0.9300
C7—H7	0.9300		
C6—C1—C2	118.9 (17)	C7—N1—C8	127.6 (19)
C6—C1—C7	117.9 (17)	C6—O1—H1	109.5
C2—C1—C7	123.2 (16)	C13—C8—N1	119.1 (17)
C3—C2—C1	122.1 (15)	C13—C8—C9	123 (2)
C3—C2—H2	119.0	N1—C8—C9	117.5 (17)
C1—C2—H2	119.0	C8—C9—C10	118 (2)
C2—C3—C4	119.0 (15)	C8—C9—H9	120.9
C2—C3—I1	121.2 (11)	C10—C9—H9	120.9
C4—C3—I1	119.6 (12)	C11—C10—C9	118 (2)
C3—C4—C5	122.7 (15)	C11—C10—H10	121.1
C3—C4—H4	118.7	C9—C10—H10	121.1
C5—C4—H4	118.7	C12—C11—C10	122 (2)
C4—C5—C6	115.2 (13)	C12—C11—H11	119.1
C4—C5—H5	122.4	C10—C11—H11	119.1
C6—C5—H5	122.4	C13—C12—C11	121 (2)
O1—C6—C1	123.1 (17)	C13—C12—H12	119.7
O1—C6—C5	114.5 (14)	C11—C12—H12	119.7
C1—C6—C5	122.2 (16)	C8—C13—C12	118 (2)
N1—C7—C1	128 (2)	C8—C13—H13	120.8
N1—C7—H7	116.0	C12—C13—H13	120.8
C1—C7—H7	116.0		
C6—C1—C2—C3	0 (3)	C6—C1—C7—N1	1 (3)
C7—C1—C2—C3	176.9 (17)	C2—C1—C7—N1	−175.8 (19)
C1—C2—C3—C4	0 (2)	C1—C7—N1—C8	169.5 (17)
C1—C2—C3—I1	−174.3 (13)	C7—N1—C8—C13	−42 (3)
C2—C3—C4—C5	0 (2)	C7—N1—C8—C9	145 (2)
I1—C3—C4—C5	174.8 (13)	C13—C8—C9—C10	7 (3)
C3—C4—C5—C6	−1 (2)	N1—C8—C9—C10	180 (2)

C2—C1—C6—O1	−175.6 (16)	C8—C9—C10—C11	−6 (4)
C7—C1—C6—O1	7 (3)	C9—C10—C11—C12	2 (4)
C2—C1—C6—C5	0 (3)	C10—C11—C12—C13	0 (4)
C7—C1—C6—C5	−177.3 (16)	N1—C8—C13—C12	−177 (2)
C4—C5—C6—O1	176.3 (16)	C9—C8—C13—C12	−4 (3)
C4—C5—C6—C1	1 (2)	C11—C12—C13—C8	1 (4)

Hydrogen-bond geometry (Å, º)

*Cg*1 and *Cg*2 are the centroids of the C1–C6 benzene ring and the C8–C13 phenyl ring, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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