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2-[(*E*)-(2,4,6-Trichlorophenyl)imino-methyl]phenolHoong-Kun Fun,^{a,*} Ching Kheng Quah,^{a,§} S. Viveka,^b D. J. Madhukumar^b and G. K. Nagaraja^b^aX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and ^bDepartment of Chemistry, Mangalore University, Karnataka, India

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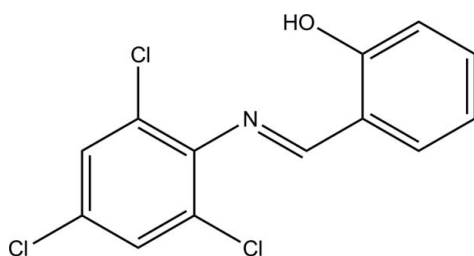
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.043; wR factor = 0.124; data-to-parameter ratio = 22.6.

The title molecule, $\text{C}_{13}\text{H}_8\text{Cl}_3\text{NO}$, exists in a *trans* configuration with respect to the $\text{C}=\text{N}$ bond [1.278 (2) Å]. The benzene rings form a dihedral angle of 24.64 (11)°. The molecular structure is stabilized by an intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond, which generates an $S(6)$ ring motif. In the crystal, $\pi-\pi$ stacking interactions [centroid-centroid distances = 3.6893 (14) Å] are observed.

Related literature

For general background to and the pharmacological activity of Schiff base compounds, see: Shapiro (1998); Villar *et al.* (2004); Venugopal & Jayashree (2008); Pandey *et al.* (2003); Bhat *et al.* (2005); Wadher *et al.* (2009). For related structures, see: Fun *et al.* (2011*a,b*). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For standard bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_{13}\text{H}_8\text{Cl}_3\text{NO}$
 $M_r = 300.55$
 Monoclinic, $P2_1/c$
 $a = 12.8847$ (16) Å

$b = 6.9505$ (9) Å
 $c = 14.4265$ (18) Å
 $\beta = 96.612$ (2)°
 $V = 1283.4$ (3) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.70$ mm⁻¹

$T = 296$ K
 $0.36 \times 0.19 \times 0.14$ mm

Data collection

Bruker SMART APEXII DUO
 CCD area-detector
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2009)
 $T_{\min} = 0.785$, $T_{\max} = 0.908$

10136 measured reflections
 3782 independent reflections
 2586 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.124$
 $S = 1.02$
 3782 reflections
 167 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.45$ e Å⁻³
 $\Delta\rho_{\min} = -0.44$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1O1}\cdots\text{N1}$	0.79 (3)	1.94 (3)	2.633 (2)	146 (3)

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); 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 and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5276).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
 Bernstein, J., Davis, R. E., Shimon, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
 Bhat, M. A., Imran, M., Khan, S. A. & Siddiqui, N. (2005). *J. Pharm. Sci.* **67**, 151–159.
 Bruker (2009). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
 Fun, H.-K., Quah, C. K., Viveka, S., Madhukumar, D. J. & Nagaraja, G. K. (2011*a*). *Acta Cryst.* **E67**, o1933.
 Fun, H.-K., Quah, C. K., Viveka, S., Madhukumar, D. J. & Prasad, D. J. (2011*b*). *Acta Cryst.* **E67**, o1932.
 Pandey, S. N., Lakshmi, V. S. & Pandey, A. (2003). *Indian J. Pharm. Sci.* **65**, 213–222.
 Shapiro, H. K. (1998). *Am. J. Ther.* **5**, 323–353.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
 Venugopal, K. N. & Jayashree, B. S. (2008). *Indian J. Pharm. Sci.* **70**, 88–91.
 Villar, R., Encio, I., Migliaccio, M., Gil, M. G. & Martinez-Merino, V. (2004). *Bioorg. Med. Chem.* **12**, 963–968.
 Wadher, S. J., Puranik, M. P., Karande, N. A. & Yeole, P. G. (2009). *Int. J. PharmTech Res.* **1**, 22–33.

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supporting information

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2-[(*E*)-(2,4,6-Trichlorophenyl)iminomethyl]phenol

Hoong-Kun Fun, Ching Kheng Quah, S. Viveka, D. J. Madhukumar and G. K. Nagaraja

S1. Comment

Schiff bases are the important compound owing to their wide range of biological activities and industrial application. The synthesis and structural research of Schiff bases are derived from aldehydes and amines bearing various alkyl and aryl *N*-substituents. Schiff base ligands may contain a variety of substituents with different electron-donating or electron-withdrawing groups and therefore may have interesting chemical properties. They have attracted particular interest due to their biological activities (Shapiro, 1998). They have been found to possess the pharmacological activities such as antimalarial, anticancer (Villar *et al.*, 2004) antibacterial (Venugopal & Jayashree, 2008) antifungal (Pandey *et al.*, 2003) antitubercular (Bhat *et al.*, 2005), anti-inflammatory and antimicrobial (Wadher *et al.*, 2009) properties.

In the title molecule (Fig. 1), the benzene rings (C1-C6 and C8-C13) form a dihedral angle of 24.64 (11)°. The title molecule exists in *trans* configuration with respect to the C7=N1 bond [C7=N1 = 1.278 (2) Å]. Bond lengths (Allen *et al.*, 1987) and angles are within normal ranges and are comparable to related structures (Fun *et al.*, 2011*a,b*). The molecular structure is stabilized by an intramolecular O1–H1O1···N1 hydrogen bond (Table 1) which generates a *S*(6) ring motif (Fig. 1, Bernstein *et al.*, 1995).

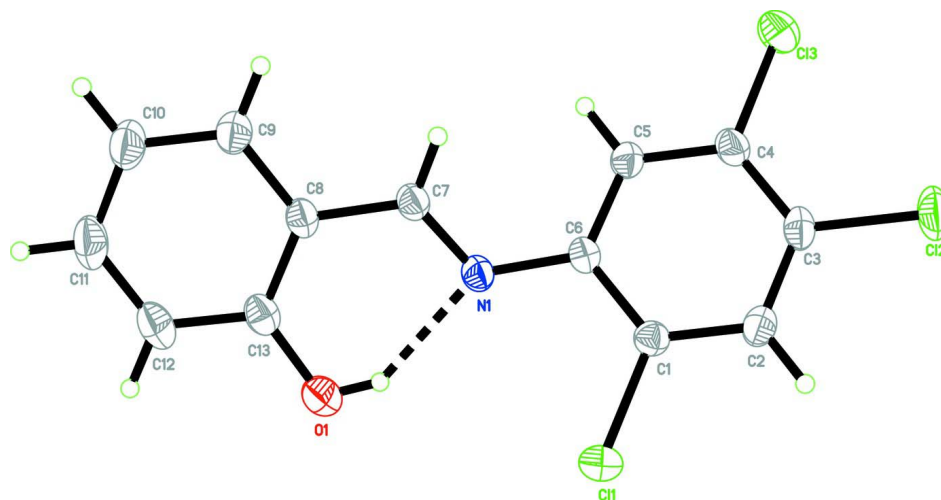
In the crystal packing, π - π stacking interactions between the centroid of C1-C6 (Cg1) and C8-C13 (Cg2) benzene rings, with Cg1···Cg2ⁱ distance of 3.6893 (14) Å [symmetry code: (i) -x, -1/2+y, 3/2-z] are observed. No significant intermolecular hydrogen bonds are observed.

S2. Experimental

A mixture of salicylaldehyde (0.01 mol) and 2,4,6-trichloroaniline (0.01 mol) was dissolved in a minimum amount of ethanol, followed by addition of 2 mL glacial acetic acid. The mixture was refluxed gently for 4-5 h. The reaction was monitored by TLC. After completion of the reaction, the mixture was poured into a beaker containing crushed ice. The precipitate obtained was filtered, dried and recrystallized from ethanol. Yield: 68%, *m.p.* 425-426 K.

S3. Refinement

H1O1 atom was located in a difference Fourier map and refined freely [O1–H1O1 = 0.79 (3) Å]. The remaining H atoms were positioned geometrically and refined using a riding model with C–H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$. The highest residual electron density peak and the deepest hole are located at 0.93 and 0.85 Å from C13, respectively.

**Figure 1**

The molecular structure of the title compound showing 30% probability displacement ellipsoids for non-H atoms. An intramolecular hydrogen bond is shown as a dashed line.

2-[(*E*)-(2,4,6-Trichlorophenyl)iminomethyl]phenol

Crystal data

$C_{13}H_8Cl_3NO$

$M_r = 300.55$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 12.8847\ (16)\ \text{\AA}$

$b = 6.9505\ (9)\ \text{\AA}$

$c = 14.4265\ (18)\ \text{\AA}$

$\beta = 96.612\ (2)^\circ$

$V = 1283.4\ (3)\ \text{\AA}^3$

$Z = 4$

$F(000) = 608$

$D_x = 1.556\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 3223 reflections

$\theta = 2.9\text{--}30.0^\circ$

$\mu = 0.70\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Block, yellow

$0.36 \times 0.19 \times 0.14\ \text{mm}$

Data collection

Bruker SMART APEXII DUO CCD area-detector

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2009)

$T_{\min} = 0.785$, $T_{\max} = 0.908$

10136 measured reflections

3782 independent reflections

2586 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 30.2^\circ$, $\theta_{\min} = 3.1^\circ$

$h = -18 \rightarrow 18$

$k = -9 \rightarrow 9$

$l = -20 \rightarrow 20$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.124$

$S = 1.02$

3782 reflections

167 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 atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0489P)^2 + 0.6136P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.45 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.44 \text{ e } \text{\AA}^{-3}$$

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. 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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.05175 (4)	0.34050 (9)	0.92951 (3)	0.05264 (16)
Cl2	0.44455 (4)	0.27949 (11)	0.84597 (5)	0.0669 (2)
Cl3	0.35845 (4)	0.16620 (13)	0.63858 (4)	0.0723 (2)
O1	-0.20343 (13)	0.2343 (3)	0.77771 (11)	0.0562 (4)
N1	-0.01212 (11)	0.2621 (2)	0.73161 (11)	0.0370 (3)
C1	0.13702 (14)	0.3001 (3)	0.84736 (13)	0.0368 (4)
C2	0.24358 (14)	0.3061 (3)	0.87539 (14)	0.0427 (4)
H2A	0.2685	0.3344	0.9369	0.051*
C3	0.31237 (14)	0.2693 (3)	0.81056 (15)	0.0428 (4)
C4	0.27450 (14)	0.2244 (3)	0.71929 (14)	0.0427 (4)
C5	0.16785 (14)	0.2223 (3)	0.69162 (13)	0.0408 (4)
H5A	0.1432	0.1931	0.6301	0.049*
C6	0.09726 (13)	0.2636 (3)	0.75513 (12)	0.0346 (4)
C7	-0.05235 (13)	0.3079 (3)	0.64944 (13)	0.0372 (4)
H7A	-0.0084	0.3437	0.6056	0.045*
C8	-0.16440 (14)	0.3060 (3)	0.62241 (14)	0.0387 (4)
C9	-0.20251 (16)	0.3408 (4)	0.52901 (15)	0.0514 (5)
H9A	-0.1558	0.3640	0.4857	0.062*
C10	-0.30845 (17)	0.3411 (4)	0.50043 (18)	0.0628 (7)
H10A	-0.3333	0.3623	0.4382	0.075*
C11	-0.37712 (17)	0.3093 (4)	0.56579 (19)	0.0638 (7)
H11A	-0.4486	0.3109	0.5471	0.077*
C12	-0.34176 (16)	0.2754 (4)	0.65778 (19)	0.0561 (6)
H12A	-0.3893	0.2549	0.7006	0.067*
C13	-0.23490 (15)	0.2716 (3)	0.68736 (15)	0.0430 (4)
H101	-0.142 (2)	0.238 (5)	0.787 (2)	0.077 (10)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0521 (3)	0.0677 (4)	0.0399 (2)	0.0004 (3)	0.0130 (2)	-0.0002 (2)
Cl2	0.0322 (2)	0.0865 (5)	0.0786 (4)	-0.0046 (3)	-0.0083 (2)	0.0036 (3)

C13	0.0415 (3)	0.1152 (6)	0.0624 (4)	0.0192 (3)	0.0154 (2)	-0.0023 (4)
O1	0.0452 (8)	0.0715 (12)	0.0537 (9)	0.0035 (8)	0.0128 (7)	0.0064 (8)
N1	0.0303 (7)	0.0408 (9)	0.0400 (8)	0.0040 (6)	0.0037 (6)	-0.0019 (7)
C1	0.0366 (8)	0.0376 (10)	0.0365 (8)	-0.0005 (8)	0.0054 (7)	0.0016 (8)
C2	0.0388 (9)	0.0464 (12)	0.0407 (9)	-0.0037 (8)	-0.0044 (7)	0.0007 (8)
C3	0.0300 (8)	0.0448 (11)	0.0518 (11)	-0.0015 (8)	-0.0036 (7)	0.0060 (9)
C4	0.0319 (8)	0.0509 (12)	0.0459 (10)	0.0077 (8)	0.0070 (7)	0.0037 (9)
C5	0.0337 (8)	0.0502 (12)	0.0379 (9)	0.0065 (8)	0.0012 (7)	-0.0009 (8)
C6	0.0291 (7)	0.0349 (9)	0.0396 (9)	0.0009 (7)	0.0032 (6)	0.0000 (7)
C7	0.0309 (8)	0.0399 (10)	0.0411 (9)	0.0031 (7)	0.0049 (7)	-0.0029 (8)
C8	0.0305 (8)	0.0391 (10)	0.0460 (9)	0.0031 (7)	0.0020 (7)	-0.0049 (8)
C9	0.0404 (10)	0.0649 (15)	0.0473 (10)	0.0039 (10)	-0.0016 (8)	-0.0046 (10)
C10	0.0443 (11)	0.0808 (19)	0.0592 (13)	0.0061 (11)	-0.0114 (10)	-0.0079 (13)
C11	0.0352 (10)	0.0735 (18)	0.0790 (17)	0.0022 (11)	-0.0098 (10)	-0.0134 (14)
C12	0.0346 (9)	0.0597 (15)	0.0752 (15)	-0.0016 (10)	0.0109 (10)	-0.0075 (12)
C13	0.0360 (9)	0.0391 (11)	0.0544 (11)	0.0025 (8)	0.0072 (8)	-0.0047 (9)

Geometric parameters (Å, °)

C11—C1	1.7292 (19)	C5—H5A	0.9300
C12—C3	1.7222 (18)	C7—C8	1.452 (2)
C13—C4	1.726 (2)	C7—H7A	0.9300
O1—C13	1.345 (3)	C8—C13	1.399 (3)
O1—H1O1	0.79 (3)	C8—C9	1.401 (3)
N1—C7	1.278 (2)	C9—C10	1.380 (3)
N1—C6	1.411 (2)	C9—H9A	0.9300
C1—C2	1.387 (2)	C10—C11	1.383 (4)
C1—C6	1.393 (2)	C10—H10A	0.9300
C2—C3	1.384 (3)	C11—C12	1.373 (4)
C2—H2A	0.9300	C11—H11A	0.9300
C3—C4	1.386 (3)	C12—C13	1.394 (3)
C4—C5	1.386 (2)	C12—H12A	0.9300
C5—C6	1.393 (2)		
C13—O1—H1O1	110 (2)	N1—C7—H7A	119.0
C7—N1—C6	120.55 (16)	C8—C7—H7A	119.0
C2—C1—C6	121.80 (17)	C13—C8—C9	119.42 (18)
C2—C1—C11	118.76 (14)	C13—C8—C7	121.61 (18)
C6—C1—C11	119.44 (13)	C9—C8—C7	118.97 (18)
C3—C2—C1	119.14 (17)	C10—C9—C8	120.8 (2)
C3—C2—H2A	120.4	C10—C9—H9A	119.6
C1—C2—H2A	120.4	C8—C9—H9A	119.6
C2—C3—C4	120.04 (17)	C9—C10—C11	119.0 (2)
C2—C3—C12	118.70 (15)	C9—C10—H10A	120.5
C4—C3—C12	121.26 (16)	C11—C10—H10A	120.5
C3—C4—C5	120.32 (18)	C12—C11—C10	121.3 (2)
C3—C4—C13	120.94 (15)	C12—C11—H11A	119.4
C5—C4—C13	118.73 (15)	C10—C11—H11A	119.4

C4—C5—C6	120.60 (17)	C11—C12—C13	120.4 (2)
C4—C5—H5A	119.7	C11—C12—H12A	119.8
C6—C5—H5A	119.7	C13—C12—H12A	119.8
C1—C6—C5	118.01 (16)	O1—C13—C12	118.5 (2)
C1—C6—N1	118.57 (16)	O1—C13—C8	122.39 (18)
C5—C6—N1	123.37 (16)	C12—C13—C8	119.1 (2)
N1—C7—C8	122.10 (17)		
C6—C1—C2—C3	-1.9 (3)	C7—N1—C6—C1	151.17 (19)
C11—C1—C2—C3	178.45 (16)	C7—N1—C6—C5	-31.6 (3)
C1—C2—C3—C4	-0.9 (3)	C6—N1—C7—C8	179.28 (17)
C1—C2—C3—C12	179.37 (16)	N1—C7—C8—C13	6.2 (3)
C2—C3—C4—C5	2.1 (3)	N1—C7—C8—C9	-174.2 (2)
C12—C3—C4—C5	-178.18 (17)	C13—C8—C9—C10	-0.2 (4)
C2—C3—C4—C13	-176.72 (17)	C7—C8—C9—C10	-179.7 (2)
C12—C3—C4—C13	3.0 (3)	C8—C9—C10—C11	1.0 (4)
C3—C4—C5—C6	-0.5 (3)	C9—C10—C11—C12	-0.8 (4)
C13—C4—C5—C6	178.34 (16)	C10—C11—C12—C13	-0.3 (4)
C2—C1—C6—C5	3.4 (3)	C11—C12—C13—O1	-178.8 (2)
C11—C1—C6—C5	-176.92 (15)	C11—C12—C13—C8	1.1 (4)
C2—C1—C6—N1	-179.22 (18)	C9—C8—C13—O1	179.0 (2)
C11—C1—C6—N1	0.5 (3)	C7—C8—C13—O1	-1.5 (3)
C4—C5—C6—C1	-2.2 (3)	C9—C8—C13—C12	-0.9 (3)
C4—C5—C6—N1	-179.41 (19)	C7—C8—C13—C12	178.7 (2)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1—H1O1...N1	0.79 (3)	1.94 (3)	2.633 (2)	146 (3)