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(*E*)-2-{[(5-Chloro-2-methoxyphenyl)imino]methyl}-4-nitrophenol

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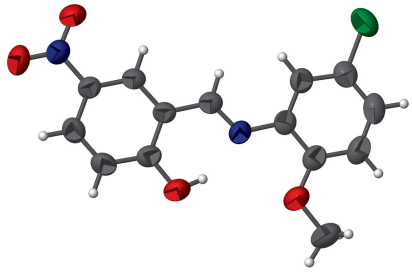
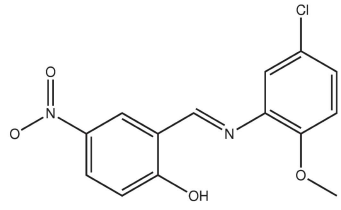
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{14}H_{11}ClN_2O_4$, is a Schiff base. Its molecule is approximatelly planar, with a maximum deviation of 0.096 (4) Å from planarity for the methyl C atom of the methoxy group. The dihedral angle between the 5-chloro-2-methoxyphenyl ring and the phenol ring is 2.40 (10)°. In the crystal structure, intermolecular C—H···O hydrogen bonds and π–π stacking interactions consolidate the crystal packing.

3D view



Chemical scheme



Structure description

Many Schiff bases are biologically active, making this class of compounds important for many different disciplines in chemistry. Moreover, some Schiff bases show photochromism which can be used for radiation intensity measurements, display systems or optical devices (Yıldız *et al.*, 2005; Hadjoudis *et al.*, 1987). Apart from these areas, Schiff base are versatile complexing agents, with the products known to show antifungal, antibacterial, herbicidal, anticancer, antiviral, anticonvulsant, diuretic or cytotoxic properties (Cozzi & Alesi, 2004; Shebl & Khalil, 2015; Tarafder *et al.*, 2002). As another class of compounds, nitroaromatics are common components of explosives, dyes and pesticides and have manifold use in organic synthesis as starting materials or intermediates (Yan *et al.*, 2006; Soojhawon *et al.*, 2005). Aromatic compounds containing multiple nitro substituents are known to resist electrophilic attack by oxgenases (Hallas & Alexander, 1983). On the other hand, nitroaromatics are industrial waste, directly polluting the environment due to their moderate solubility in water, hence poisoning rivers, ponds and soil (Yan *et al.*, 2006; Soojhawon *et al.*, 2005). We have synthesized a Schiff base with an aromatic nitro substituent and report herein on its crystal structure.

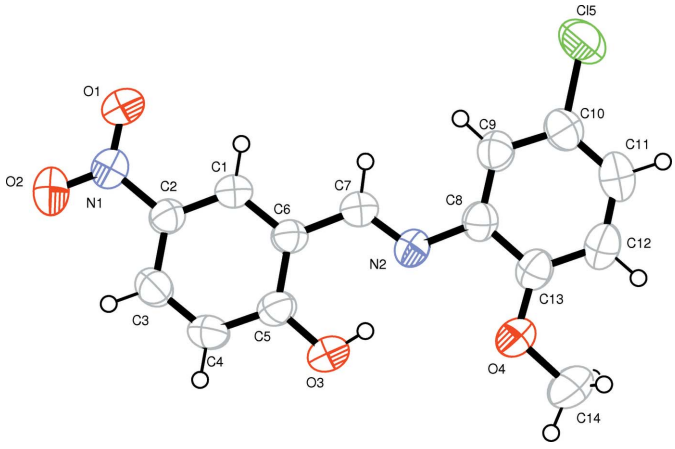


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

The title molecule (Fig. 1) is essentially planar, with a maximum deviation of 0.096 (4) Å from planarity for the methyl C atom (C14) of the methoxy group. The C7—N2 bond length of 1.270 (3) Å and the C5—O3 bond lengths of 1.328 (3) Å are consistent with a double bond and a single bond, respectively, and are comparable with those of related structures (Kılıç *et al.*, 2009). The entities *A* (phenol ring; C1–C6/O3), *B* (nitro group; O1/O2/N1) and *C* (5-chloro-2-methoxyphenyl ring; C8–C13/O4/ C14/CI1) are inclined by dihedral angles of *A*/*B* = 3.1 (3)°, *A*/*C* = 2.40 (10)° and (*A*+*B*)/*C* = 2.15 (9)°. An intramolecular O—H···N hydrogen bond stabilizes the molecular conformation whereas intermolecular

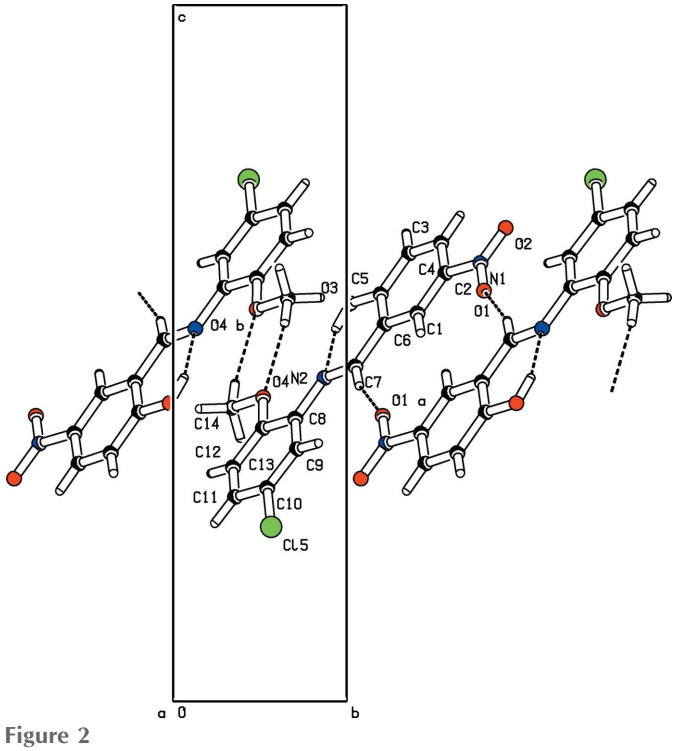


Figure 2
A partial packing view along [100]. Dashed lines indicate hydrogen bonds.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H15···N2	0.84 (3)	1.84 (3)	2.606 (3)	151 (3)
C7—H7···O1^{i}	0.93	2.42	3.325 (3)	163
C14—H14*A*···O4^{ii}	0.96	2.58	3.516 (4)	166

Symmetry codes: (i) $-x+1, -y+3, -z+1$; (ii) $-x, -y+1, -z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{11}ClN_2O_4$
M_r	306.70
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, *b*, *c* (Å)	14.3727 (9), 4.8669 (4), 22.3233 (13)
β (°)	118.859 (9)
V (Å^3)	1367.60 (19)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	0.30
Crystal size (mm)	0.71 × 0.30 × 0.05
Data collection	
Diffractometer	Stoe IPDS 2
Absorption correction	–
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10508, 2679, 1527
R_{int}	0.048
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.042, 0.106, 0.94
No. of reflections	2679
No. of parameters	194
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.13, −0.18

Computer programs: *X-AREA* and *X-RED32* (Stoe & Cie, 2002), *SHELXS97* (Sheldrick, 2008), *SHELXL2016* (Sheldrick, 2015), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012).

(methyl)C—H···O(methoxy) and (imine)C—H···O(nitro) hydrogen bonds lead to the formation of sheets extending parallel to (210) (Fig. 2, Table 1). A plane-to-plane distance of 3.379 (3) Å for parallel-aligned sheets indicates the presence of π–π stacking interactions in the crystal.

Synthesis and crystallization

The title compound was prepared by refluxing a mixture of 2-hydroxy-5-nitrobenzaldehyde (0.0069 g, 0.413 mmol) in 20 ml ethanol and 3-chloro-4-methoxyaniline (0.0065 g, 0.413 mmol) in 20 ml ethanol for one hour. Crystals suitable for X-ray analysis were obtained by slow evaporation of the resulting solution (yield 35%; m.p 450–452 K).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2017). **2**, x170177 [https://doi.org/10.1107/S2414314617001778]

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Crystal data

$C_{14}H_{11}ClN_2O_4$
M_r = 306.70
Monoclinic, $P2_1/c$
a = 14.3727 (9) Å
b = 4.8669 (4) Å
c = 22.3233 (13) Å
β = 118.859 (9)°
V = 1367.60 (19) Å^3
Z = 4
$F(000)$ = 632
D_x = 1.490 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 453 reflections
θ = 2.8–26.5°
μ = 0.30 mm^{-1}
T = 293 K
Stick, orange
0.71 × 0.30 × 0.05 mm

Data collection

Stoe IPDS 2 diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 6.67 pixels mm^{-1}
rotation method scans
10508 measured reflections
2679 independent reflections
1527 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 26.0°, θ_{min} = 1.6°
h = −17→17
k = −5→6
l = −27→27

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.106
S = 0.94
2679 reflections
194 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.52416 (15)	1.7724 (4)	0.62967 (10)	0.0621 (5)
N2	0.21754 (15)	0.8722 (4)	0.46453 (9)	0.0548 (5)
O1	0.55958 (16)	1.7922 (4)	0.58993 (10)	0.0904 (6)
O2	0.55351 (15)	1.9177 (4)	0.68000 (9)	0.0819 (6)
O3	0.20231 (15)	1.0208 (4)	0.57164 (11)	0.0773 (6)
O4	0.06221 (14)	0.5188 (4)	0.43672 (9)	0.0827 (6)
Cl5	0.24175 (6)	0.56490 (18)	0.25064 (4)	0.0910 (3)
C1	0.40681 (17)	1.4090 (5)	0.55759 (11)	0.0532 (6)
H1	0.437436	1.426791	0.529395	0.064*
C2	0.44190 (17)	1.5699 (5)	0.61496 (10)	0.0516 (5)
C3	0.39872 (19)	1.5455 (5)	0.65855 (11)	0.0627 (6)
H3	0.423828	1.653642	0.697654	0.075*
C4	0.3190 (2)	1.3609 (5)	0.64320 (12)	0.0678 (7)
H4	0.289785	1.344033	0.672215	0.081*
C5	0.28050 (19)	1.1971 (5)	0.58499 (12)	0.0572 (6)
C6	0.32592 (17)	1.2200 (5)	0.54146 (10)	0.0504 (5)
C7	0.29030 (18)	1.0508 (5)	0.48111 (11)	0.0567 (6)
H7	0.321734	1.073469	0.453501	0.068*
C8	0.18388 (18)	0.7040 (5)	0.40607 (11)	0.0532 (5)
C9	0.22709 (19)	0.7136 (5)	0.36221 (11)	0.0593 (6)
H9	0.281618	0.836500	0.370621	0.071*
C10	0.18942 (19)	0.5422 (5)	0.30648 (12)	0.0612 (6)
C11	0.1098 (2)	0.3565 (5)	0.29338 (12)	0.0676 (7)
H11	0.085619	0.239788	0.255838	0.081*
C12	0.0665 (2)	0.3450 (5)	0.33613 (12)	0.0675 (7)
H12	0.012689	0.219286	0.327311	0.081*
C13	0.10144 (18)	0.5171 (5)	0.39218 (12)	0.0588 (6)
C14	−0.0258 (3)	0.3385 (8)	0.42157 (18)	0.1160 (13)
H14A	−0.046574	0.357148	0.456270	0.139*
H14B	−0.084445	0.386065	0.377899	0.139*
H14C	−0.004954	0.151962	0.420421	0.139*
H15	0.190 (3)	0.938 (7)	0.5353 (18)	0.110 (12)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0579 (12)	0.0628 (14)	0.0635 (12)	−0.0097 (10)	0.0277 (10)	−0.0060 (11)
N2	0.0572 (11)	0.0488 (12)	0.0588 (11)	−0.0081 (10)	0.0282 (9)	−0.0033 (9)
O1	0.0925 (14)	0.1061 (17)	0.0973 (14)	−0.0438 (12)	0.0655 (12)	−0.0278 (12)
O2	0.0839 (13)	0.0805 (13)	0.0724 (11)	−0.0243 (10)	0.0307 (10)	−0.0242 (11)
O3	0.0841 (13)	0.0825 (15)	0.0841 (13)	−0.0298 (11)	0.0555 (11)	−0.0121 (11)
O4	0.0831 (12)	0.0952 (15)	0.0797 (11)	−0.0399 (11)	0.0470 (10)	−0.0193 (10)
Cl5	0.1001 (6)	0.1078 (6)	0.0782 (4)	0.0006 (5)	0.0534 (4)	−0.0180 (4)
C1	0.0551 (13)	0.0577 (15)	0.0540 (12)	−0.0050 (12)	0.0319 (10)	0.0000 (11)
C2	0.0520 (13)	0.0527 (14)	0.0506 (12)	−0.0054 (11)	0.0252 (10)	0.0012 (11)

C3	0.0777 (17)	0.0616 (17)	0.0530 (13)	−0.0057 (14)	0.0349 (12)	−0.0048 (12)
C4	0.0865 (18)	0.0710 (19)	0.0637 (15)	−0.0118 (15)	0.0504 (14)	−0.0023 (13)
C5	0.0620 (14)	0.0535 (15)	0.0636 (14)	−0.0066 (12)	0.0364 (12)	0.0032 (12)
C6	0.0518 (13)	0.0535 (15)	0.0498 (11)	−0.0027 (11)	0.0276 (10)	0.0022 (11)
C7	0.0593 (14)	0.0601 (15)	0.0565 (13)	−0.0062 (13)	0.0324 (11)	−0.0009 (12)
C8	0.0564 (13)	0.0446 (13)	0.0543 (12)	0.0012 (11)	0.0234 (11)	0.0014 (11)
C9	0.0606 (14)	0.0536 (15)	0.0625 (14)	−0.0020 (12)	0.0288 (12)	−0.0045 (12)
C10	0.0642 (15)	0.0584 (16)	0.0579 (14)	0.0083 (13)	0.0271 (12)	−0.0004 (12)
C11	0.0743 (17)	0.0558 (17)	0.0550 (14)	0.0054 (13)	0.0171 (13)	−0.0070 (12)
C12	0.0689 (16)	0.0529 (16)	0.0669 (16)	−0.0100 (13)	0.0218 (13)	−0.0019 (13)
C13	0.0575 (14)	0.0529 (16)	0.0587 (13)	−0.0042 (12)	0.0224 (12)	0.0032 (11)
C14	0.104 (3)	0.149 (3)	0.113 (2)	−0.071 (2)	0.067 (2)	−0.038 (2)

Geometric parameters (Å, º)

N1—O2	1.218 (2)	C4—H4	0.9300
N1—O1	1.221 (2)	C5—C6	1.413 (3)
N1—C2	1.450 (3)	C6—C7	1.445 (3)
N2—C7	1.270 (3)	C7—H7	0.9300
N2—C8	1.413 (3)	C8—C9	1.391 (3)
O3—C5	1.328 (3)	C8—C13	1.405 (3)
O3—H15	0.84 (3)	C9—C10	1.374 (3)
O4—C13	1.359 (3)	C9—H9	0.9300
O4—C14	1.439 (3)	C10—C11	1.374 (4)
Cl5—C10	1.743 (2)	C11—C12	1.369 (4)
C1—C2	1.373 (3)	C11—H11	0.9300
C1—C6	1.387 (3)	C12—C13	1.383 (3)
C1—H1	0.9300	C12—H12	0.9300
C2—C3	1.389 (3)	C14—H14A	0.9600
C3—C4	1.363 (3)	C14—H14B	0.9600
C3—H3	0.9300	C14—H14C	0.9600
C4—C5	1.391 (3)		

O2—N1—O1	123.0 (2)	C6—C7—H7	118.9
O2—N1—C2	119.0 (2)	C9—C8—C13	118.8 (2)
O1—N1—C2	118.00 (19)	C9—C8—N2	124.1 (2)
C7—N2—C8	122.17 (18)	C13—C8—N2	117.09 (19)
C5—O3—H15	107 (2)	C10—C9—C8	120.2 (2)
C13—O4—C14	116.9 (2)	C10—C9—H9	119.9
C2—C1—C6	120.34 (19)	C8—C9—H9	119.9
C2—C1—H1	119.8	C11—C10—C9	121.1 (2)
C6—C1—H1	119.8	C11—C10—Cl5	119.48 (19)
C1—C2—C3	121.2 (2)	C9—C10—Cl5	119.5 (2)
C1—C2—N1	119.43 (18)	C12—C11—C10	119.4 (2)
C3—C2—N1	119.4 (2)	C12—C11—H11	120.3
C4—C3—C2	119.1 (2)	C10—C11—H11	120.3
C4—C3—H3	120.5	C11—C12—C13	121.1 (2)
C2—C3—H3	120.5	C11—C12—H12	119.4

C3—C4—C5	121.3 (2)	C13—C12—H12	119.4
C3—C4—H4	119.3	O4—C13—C12	124.6 (2)
C5—C4—H4	119.3	O4—C13—C8	116.0 (2)
O3—C5—C4	119.5 (2)	C12—C13—C8	119.4 (2)
O3—C5—C6	121.2 (2)	O4—C14—H14A	109.5
C4—C5—C6	119.3 (2)	O4—C14—H14B	109.5
C1—C6—C5	118.8 (2)	H14A—C14—H14B	109.5
C1—C6—C7	119.69 (18)	O4—C14—H14C	109.5
C5—C6—C7	121.5 (2)	H14A—C14—H14C	109.5
N2—C7—C6	122.21 (19)	H14B—C14—H14C	109.5
N2—C7—H7	118.9		

C6—C1—C2—C3	0.9 (3)	C5—C6—C7—N2	−0.7 (3)
C6—C1—C2—N1	−178.0 (2)	C7—N2—C8—C9	1.0 (3)
O2—N1—C2—C1	178.7 (2)	C7—N2—C8—C13	−178.9 (2)
O1—N1—C2—C1	0.0 (3)	C13—C8—C9—C10	−0.1 (3)
O2—N1—C2—C3	−0.2 (3)	N2—C8—C9—C10	180.0 (2)
O1—N1—C2—C3	−178.9 (2)	C8—C9—C10—C11	−0.8 (4)
C1—C2—C3—C4	−1.0 (4)	C8—C9—C10—Cl5	178.00 (18)
N1—C2—C3—C4	177.9 (2)	C9—C10—C11—C12	0.9 (4)
C2—C3—C4—C5	0.1 (4)	Cl5—C10—C11—C12	−177.96 (19)
C3—C4—C5—O3	−179.6 (2)	C10—C11—C12—C13	0.0 (4)
C3—C4—C5—C6	0.9 (4)	C14—O4—C13—C12	−3.6 (4)
C2—C1—C6—C5	0.1 (3)	C14—O4—C13—C8	177.0 (3)
C2—C1—C6—C7	−179.6 (2)	C11—C12—C13—O4	179.7 (2)
O3—C5—C6—C1	179.5 (2)	C11—C12—C13—C8	−1.0 (4)
C4—C5—C6—C1	−1.0 (3)	C9—C8—C13—O4	−179.6 (2)
O3—C5—C6—C7	−0.8 (4)	N2—C8—C13—O4	0.2 (3)
C4—C5—C6—C7	178.7 (2)	C9—C8—C13—C12	1.0 (3)
C8—N2—C7—C6	−179.0 (2)	N2—C8—C13—C12	−179.1 (2)
C1—C6—C7—N2	179.1 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H15···N2	0.84 (3)	1.84 (3)	2.606 (3)	151 (3)
C7—H7···O1^{i}	0.93	2.42	3.325 (3)	163
C14—H14*A*···O4^{ii}	0.96	2.58	3.516 (4)	166

Symmetry codes: (i) −*x*+1, −*y*+3, −*z*+1; (ii) −*x*, −*y*+1, −*z*+1.