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2-{(*E*)-[(2-Chloro-5-nitrophenyl)imino]-methyl}-5-(diethylamino)phenol

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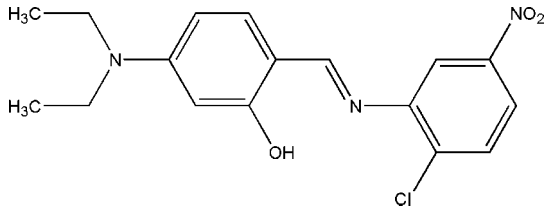
Key indicators: single-crystal X-ray study; T = 203 K; mean σ(C–C) = 0.002 Å; R factor = 0.056; wR factor = 0.162; data-to-parameter ratio = 24.4.

In the title molecule, $C_{17}H_{18}ClN_3O_3$, the phenol and 2-chloro-5-nitrophenyl groups are coplanar with each other and also with the imine linkage. Crystal packing is stabilized by intermolecular C—H···O hydrogen bonds between a chlorophenyl H atom and a nitro O atom which link the molecules into a chain along the c axis of the unit cell. Intramolecular O—H···N hydrogen bonding also occurs between the hydroxyl H atom and the imine N atom.

Related literature

For related structures, see: Odabaşoğlu *et al.* (2007); Yathirajan *et al.* (2007). For related literature, see: Hodnett & Dunn (1970); Misra *et al.* (1981); Agarwal *et al.* (1983); Varma *et al.* (1986); Singh & Dash (1988).



Experimental

Crystal data

$C_{17}H_{18}ClN_3O_3$
M_r = 347.79
Monoclinic, $P2_1/c$
a = 7.4956 (3) Å
b = 23.3915 (7) Å
c = 10.0552 (5) Å
β = 111.008 (5)°
V = 1645.82 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 203 (2) K
0.49 × 0.45 × 0.25 mm

Data collection

Oxford Diffraction Gemini R diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
T_{min} = 0.940, T_{max} = 1.000 (expected range = 0.882–0.939)
17009 measured reflections
5364 independent reflections
3914 reflections with $I > 2\sigma(I)$
R_{int} = 0.054

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.162
S = 1.10
5364 reflections
220 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···O2^{i}	0.94	2.41	3.291 (2)	157
O3—H3···N2	0.83	1.90	2.6356 (18)	147

Symmetry code: (i) $x+1, y, z$.

Data collection: *CrysAlisPro* (Oxford Diffraction, 2007); cell refinement: *CrysAlisPro*; data reduction: *CrysAlisPro*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2365).

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supplementary materials

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2-{(*E*)-[(2-Chloro-5-nitrophenyl)imino]methyl}-5-(diethylamino)phenol

R. J. Butcher, J. P. Jasinski, H. S. Yathirajan, A. M. Vijesh and B. Narayana

Comment

Schiff bases are used as substrates in the preparation of number of industrial and biologically active compounds *via* ring closure, cycloaddition and replacement reactions. Some Schiff base derivatives were reported to possess antimicrobial, anti-inflammatory and central nervous system activities. Moreover, Schiff bases are also known to have biological activities such as antimicrobial, antifungal, antitumor, and as herbicides. A new Schiff base, (I), $C_{17}H_{18}ClN_3O_3$ has been synthesized and herein its crystal structure is reported.

The phenol and 2-chloro-5-nitrophenyl groups of the title molecule (I) (Fig. 1) are coplanar with each other [dihedral angle = 5.1 (4)°] and also with the imine linkage, forming torsion angles C7—N2—C6—C5 and N2—C7—C8—C9 of 5.0 (2)° and 0.2 (2)(18)°, respectively.

Intermolecular C—H···O hydrogen bonding between a chlorophenyl hydrogen (C2–H2A) and O2 from the nitro group link the molecules into a chain along the *c* axis of the unit cell and stabilize crystal packing (Fig. 2). Intramolecular O—H···N hydrogen bonding also occurs between the hydroxyl hydrogen (H3) and the imine nitrogen (N2) within the asymmetric unit (Fig. 1, Tablo 1).

Experimental

A mixture of 2-chloro-5-nitroaniline (0.345 g, 0.002 mol) and 4-(diethylamino)-2-hydroxybenzaldehyde (0.386 g, 0.002 mol) in 15 ml of absolute ethanol containing 2 drops of 4 *M* sulfuric acid was refluxed for about 3 h. On cooling, the solid separated, was filtered and recrystallized from acetone (m.p.: 441–445 K). Analysis found: C 58.59, H 5.16, N 12.01%; $C_{17}H_{18}ClN_3O_3$ requires: C 58.71, H 5.22, N 12.08%.

Refinement

All H atoms were refined using a riding model with O—H = 0.83 Å and C—H = 0.94–0.97 Å, and with U_{iso}(H) = 1.19–1.50U_{eq}(C, O).

Figures

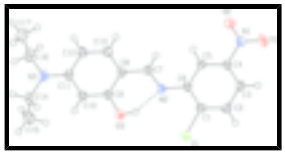

Fig. 1. Molecular structure of the title compound (I), showing atom labelling and 50% probability displacement ellipsoids. Dashed lines indicate N—H···O intramolecular hydrogen bonds.

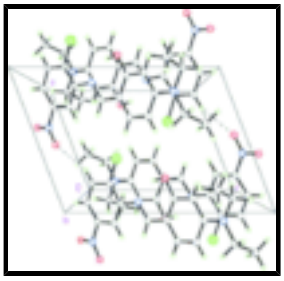

Fig. 2. Packing diagram of (I), viewed down the *b* axis. Dashed lines indicate C—H···O intermolecular hydrogen bonds.

2-{(*E*)-[(2-Chloro-5-nitrophenyl)imino]methyl}-5-(diethylamino)phenol

Crystal data

$C_{17}H_{18}ClN_3O_3$	F_{000} = 728
M_r = 347.79	D_x = 1.404 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 9461 reflections
a = 7.4956 (3) Å	θ = 4.9–32.3º
b = 23.3915 (7) Å	μ = 0.25 mm^{-1}
c = 10.0552 (5) Å	T = 203 (2) K
β = 111.008 (5)º	Plate, yellow
V = 1645.82 (12) Å^3	0.49 × 0.45 × 0.25 mm
Z = 4	

Data collection

Oxford Diffraction Gemini R diffractometer	3914 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.054
Monochromator: graphite	θ_{max} = 32.3º
T = 203 K	θ_{min} = 4.9º
φ and ω scans	h = −11→11
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2007)	k = −34→33
T_{min} = 0.940, T_{max} = 1.000	l = −15→14
17009 measured reflections	Standard reflections: ?
5364 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.056	H-atom parameters constrained
$wR(F^2)$ = 0.162	$w = 1/[\sigma^2(F_o^2) + (0.0722P)^2 + 0.8483P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.10	$(\Delta/\sigma)_{max}$ = 0.001

5364 reflections	$\Delta\rho_{max} = 0.41$ e Å^{-3}
220 parameters	$\Delta\rho_{min} = -0.31$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl	1.34352 (6)	−0.00398 (2)	0.80924 (6)	0.03748 (14)
O1	0.6102 (2)	0.14212 (7)	0.38511 (19)	0.0513 (4)
O2	0.45852 (19)	0.08230 (6)	0.46828 (16)	0.0390 (3)
O3	1.19131 (17)	−0.09886 (5)	1.04607 (14)	0.0301 (3)
H3	1.1681	−0.0748	0.9815	0.036*
N1	0.6067 (2)	0.10152 (6)	0.46062 (16)	0.0273 (3)
N2	0.97949 (19)	−0.03280 (5)	0.83664 (15)	0.0230 (3)
N3	0.8752 (2)	−0.22171 (6)	1.26954 (17)	0.0307 (3)
C1	1.1265 (2)	0.02557 (7)	0.70757 (18)	0.0230 (3)
C2	1.1238 (2)	0.06546 (7)	0.60541 (19)	0.0272 (3)
H2A	1.2378	0.0753	0.5920	0.033*
C3	0.9525 (2)	0.09101 (7)	0.52241 (18)	0.0265 (3)
H3A	0.9478	0.1184	0.4528	0.032*
C4	0.7889 (2)	0.07449 (6)	0.54647 (17)	0.0218 (3)
C5	0.7879 (2)	0.03435 (6)	0.64667 (17)	0.0213 (3)
H5A	0.6728	0.0245	0.6583	0.026*
C6	0.9605 (2)	0.00830 (6)	0.73132 (17)	0.0204 (3)
C7	0.8315 (2)	−0.05334 (6)	0.85754 (17)	0.0231 (3)
H7A	0.7096	−0.0400	0.8009	0.028*
C8	0.8467 (2)	−0.09556 (6)	0.96367 (17)	0.0218 (3)
C9	1.0257 (2)	−0.11792 (6)	1.05438 (17)	0.0217 (3)
C10	1.0346 (2)	−0.15990 (6)	1.15374 (18)	0.0243 (3)
H10A	1.1540	−0.1748	1.2107	0.029*
C11	0.8672 (2)	−0.18063 (6)	1.17092 (17)	0.0237 (3)
C12	0.6883 (2)	−0.15741 (7)	1.08184 (19)	0.0268 (3)
H12A	0.5746	−0.1699	1.0919	0.032*
C13	0.6820 (2)	−0.11718 (7)	0.98224 (18)	0.0265 (3)
H13A	0.5621	−0.1032	0.9232	0.032*

C14	1.0565 (3)	−0.24648 (8)	1.36135 (19)	0.0336 (4)
H14A	1.0431	−0.2600	1.4496	0.040*
H14B	1.1550	−0.2167	1.3867	0.040*
C15	1.1208 (4)	−0.29595 (9)	1.2914 (3)	0.0470 (5)
H15A	1.2391	−0.3116	1.3576	0.070*
H15B	1.1412	−0.2824	1.2068	0.070*
H15C	1.0232	−0.3254	1.2650	0.070*
C16	0.7024 (3)	−0.24621 (8)	1.2819 (2)	0.0365 (4)
H16A	0.7309	−0.2848	1.3218	0.044*
H16B	0.6040	−0.2495	1.1866	0.044*
C17	0.6245 (4)	−0.21056 (10)	1.3756 (3)	0.0480 (5)
H17A	0.5152	−0.2298	1.3854	0.072*
H17B	0.5855	−0.1734	1.3322	0.072*
H17C	0.7230	−0.2058	1.4689	0.072*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0244 (2)	0.0427 (3)	0.0451 (3)	0.00914 (16)	0.01210 (19)	0.00777 (19)
O1	0.0425 (8)	0.0488 (9)	0.0589 (10)	0.0079 (7)	0.0136 (7)	0.0329 (8)
O2	0.0258 (6)	0.0482 (8)	0.0409 (8)	0.0009 (5)	0.0093 (6)	0.0095 (6)
O3	0.0257 (6)	0.0334 (6)	0.0320 (7)	0.0020 (5)	0.0114 (5)	0.0096 (5)
N1	0.0294 (7)	0.0263 (6)	0.0250 (7)	0.0028 (5)	0.0082 (6)	0.0048 (5)
N2	0.0259 (6)	0.0210 (6)	0.0221 (6)	0.0019 (5)	0.0089 (5)	0.0038 (5)
N3	0.0401 (8)	0.0247 (6)	0.0304 (8)	0.0011 (6)	0.0164 (7)	0.0075 (6)
C1	0.0206 (7)	0.0225 (7)	0.0264 (8)	0.0021 (5)	0.0090 (6)	−0.0006 (6)
C2	0.0261 (7)	0.0285 (7)	0.0320 (9)	−0.0031 (6)	0.0166 (7)	0.0013 (6)
C3	0.0305 (8)	0.0259 (7)	0.0262 (8)	−0.0017 (6)	0.0139 (7)	0.0048 (6)
C4	0.0244 (7)	0.0199 (6)	0.0210 (7)	0.0002 (5)	0.0081 (6)	0.0008 (5)
C5	0.0206 (6)	0.0206 (6)	0.0234 (7)	−0.0007 (5)	0.0086 (6)	0.0014 (5)
C6	0.0237 (7)	0.0177 (6)	0.0208 (7)	0.0001 (5)	0.0090 (6)	0.0006 (5)
C7	0.0249 (7)	0.0217 (7)	0.0228 (7)	0.0023 (5)	0.0087 (6)	0.0031 (6)
C8	0.0261 (7)	0.0186 (6)	0.0205 (7)	0.0007 (5)	0.0081 (6)	0.0021 (5)
C9	0.0251 (7)	0.0205 (6)	0.0210 (7)	0.0007 (5)	0.0099 (6)	−0.0001 (5)
C10	0.0289 (7)	0.0201 (6)	0.0239 (7)	0.0039 (5)	0.0096 (6)	0.0022 (6)
C11	0.0331 (8)	0.0166 (6)	0.0229 (7)	−0.0003 (5)	0.0119 (6)	0.0002 (5)
C12	0.0280 (7)	0.0259 (7)	0.0275 (8)	−0.0033 (6)	0.0112 (6)	0.0025 (6)
C13	0.0255 (7)	0.0264 (7)	0.0264 (8)	0.0004 (6)	0.0079 (6)	0.0031 (6)
C14	0.0484 (11)	0.0286 (8)	0.0227 (8)	0.0032 (7)	0.0115 (8)	0.0083 (6)
C15	0.0610 (14)	0.0342 (10)	0.0476 (13)	0.0148 (9)	0.0217 (11)	0.0096 (9)
C16	0.0481 (11)	0.0279 (8)	0.0370 (10)	−0.0063 (7)	0.0197 (9)	0.0065 (7)
C17	0.0527 (13)	0.0499 (12)	0.0525 (13)	−0.0034 (10)	0.0324 (11)	0.0039 (10)

Geometric parameters (Å, °)

Cl—C1	1.7251 (16)	C8—C13	1.407 (2)
O1—N1	1.222 (2)	C8—C9	1.424 (2)
O2—N1	1.2258 (19)	C9—C10	1.385 (2)
O3—C9	1.3495 (19)	C10—C11	1.412 (2)

O3—H3	0.8300	C10—H10A	0.9400
N1—C4	1.472 (2)	C11—C12	1.426 (2)
N2—C7	1.293 (2)	C12—C13	1.363 (2)
N2—C6	1.3993 (19)	C12—H12A	0.9400
N3—C11	1.367 (2)	C13—H13A	0.9400
N3—C14	1.461 (2)	C14—C15	1.520 (3)
N3—C16	1.462 (2)	C14—H14A	0.9800
C1—C2	1.382 (2)	C14—H14B	0.9800
C1—C6	1.408 (2)	C15—H15A	0.9700
C2—C3	1.391 (2)	C15—H15B	0.9700
C2—H2A	0.9400	C15—H15C	0.9700
C3—C4	1.387 (2)	C16—C17	1.523 (3)
C3—H3A	0.9400	C16—H16A	0.9800
C4—C5	1.379 (2)	C16—H16B	0.9800
C5—C6	1.407 (2)	C17—H17A	0.9700
C5—H5A	0.9400	C17—H17B	0.9700
C7—C8	1.428 (2)	C17—H17C	0.9700
C7—H7A	0.9400		
C9—O3—H3	109.5	C9—C10—H10A	119.5
O1—N1—O2	123.10 (15)	C11—C10—H10A	119.5
O1—N1—C4	118.42 (14)	N3—C11—C10	121.36 (15)
O2—N1—C4	118.48 (14)	N3—C11—C12	120.67 (15)
C7—N2—C6	121.17 (14)	C10—C11—C12	117.97 (14)
C11—N3—C14	121.75 (15)	C13—C12—C11	120.09 (15)
C11—N3—C16	121.84 (15)	C13—C12—H12A	120.0
C14—N3—C16	116.27 (14)	C11—C12—H12A	120.0
C2—C1—C6	122.75 (14)	C12—C13—C8	123.02 (15)
C2—C1—Cl	118.06 (12)	C12—C13—H13A	118.5
C6—C1—Cl	119.18 (12)	C8—C13—H13A	118.5
C1—C2—C3	120.04 (14)	N3—C14—C15	112.76 (16)
C1—C2—H2A	120.0	N3—C14—H14A	109.0
C3—C2—H2A	120.0	C15—C14—H14A	109.0
C4—C3—C2	117.34 (14)	N3—C14—H14B	109.0
C4—C3—H3A	121.3	C15—C14—H14B	109.0
C2—C3—H3A	121.3	H14A—C14—H14B	107.8
C5—C4—C3	123.63 (14)	C14—C15—H15A	109.5
C5—C4—N1	118.29 (13)	C14—C15—H15B	109.5
C3—C4—N1	118.09 (14)	H15A—C15—H15B	109.5
C4—C5—C6	119.49 (13)	C14—C15—H15C	109.5
C4—C5—H5A	120.3	H15A—C15—H15C	109.5
C6—C5—H5A	120.3	H15B—C15—H15C	109.5
N2—C6—C5	125.29 (13)	N3—C16—C17	112.71 (16)
N2—C6—C1	117.97 (14)	N3—C16—H16A	109.1
C5—C6—C1	116.74 (14)	C17—C16—H16A	109.1
N2—C7—C8	122.36 (14)	N3—C16—H16B	109.1
N2—C7—H7A	118.8	C17—C16—H16B	109.1
C8—C7—H7A	118.8	H16A—C16—H16B	107.8
C13—C8—C9	116.94 (14)	C16—C17—H17A	109.5
C13—C8—C7	120.60 (14)	C16—C17—H17B	109.5

C9—C8—C7	122.46 (14)	H17A—C17—H17B	109.5
O3—C9—C10	118.11 (14)	C16—C17—H17C	109.5
O3—C9—C8	121.03 (14)	H17A—C17—H17C	109.5
C10—C9—C8	120.86 (14)	H17B—C17—H17C	109.5
C9—C10—C11	121.09 (15)		
C6—C1—C2—C3	1.0 (3)	C13—C8—C9—O3	−178.67 (15)
Cl—C1—C2—C3	−179.07 (13)	C7—C8—C9—O3	1.6 (2)
C1—C2—C3—C4	−0.4 (2)	C13—C8—C9—C10	1.3 (2)
C2—C3—C4—C5	−0.3 (2)	C7—C8—C9—C10	−178.35 (15)
C2—C3—C4—N1	179.38 (15)	O3—C9—C10—C11	178.20 (14)
O1—N1—C4—C5	170.73 (17)	C8—C9—C10—C11	−1.8 (2)
O2—N1—C4—C5	−9.2 (2)	C14—N3—C11—C10	−0.4 (2)
O1—N1—C4—C3	−9.0 (2)	C16—N3—C11—C10	−175.88 (16)
O2—N1—C4—C3	171.05 (16)	C14—N3—C11—C12	179.56 (15)
C3—C4—C5—C6	0.4 (2)	C16—N3—C11—C12	4.1 (2)
N1—C4—C5—C6	−179.30 (13)	C9—C10—C11—N3	−179.47 (15)
C7—N2—C6—C5	5.0 (2)	C9—C10—C11—C12	0.6 (2)
C7—N2—C6—C1	−175.37 (15)	N3—C11—C12—C13	−178.87 (16)
C4—C5—C6—N2	179.86 (14)	C10—C11—C12—C13	1.1 (2)
C4—C5—C6—C1	0.2 (2)	C11—C12—C13—C8	−1.6 (3)
C2—C1—C6—N2	179.40 (15)	C9—C8—C13—C12	0.4 (2)
Cl—C1—C6—N2	−0.5 (2)	C7—C8—C13—C12	−179.95 (16)
C2—C1—C6—C5	−0.9 (2)	C11—N3—C14—C15	−85.2 (2)
Cl—C1—C6—C5	179.17 (11)	C16—N3—C14—C15	90.5 (2)
C6—N2—C7—C8	179.62 (14)	C11—N3—C16—C17	−85.1 (2)
N2—C7—C8—C13	−179.46 (15)	C14—N3—C16—C17	99.1 (2)
N2—C7—C8—C9	0.2 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2A···O2^{i}	0.94	2.41	3.291 (2)	157
O3—H3···N2	0.83	1.90	2.6356 (18)	147

Symmetry codes: (i) *x*+1, *y*, *z*.

Fig. 1

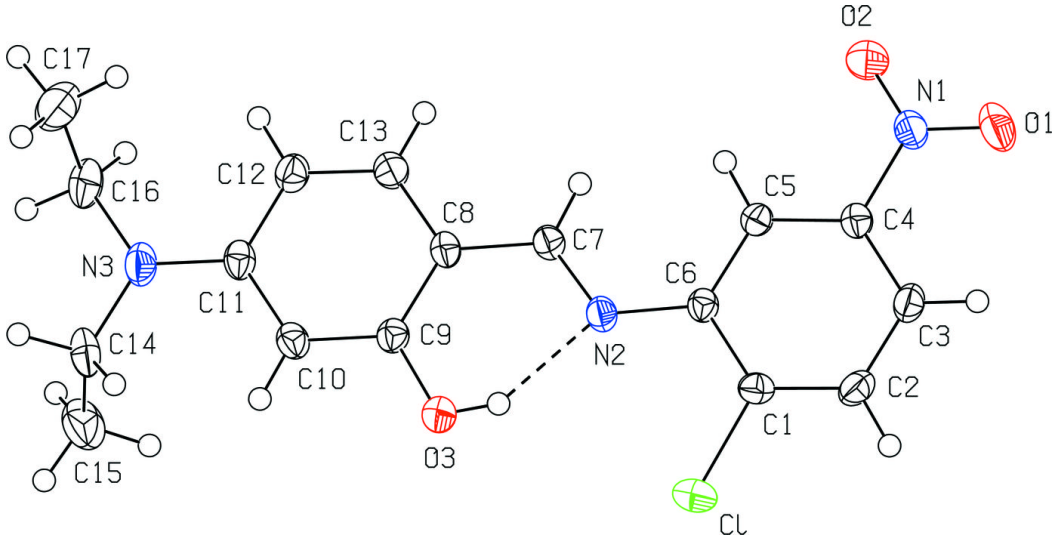
O2
N1
O1
C17
C13
C12
C16
C8
C7
C5
C4
N3
C11
C6
C14
C9
N2
C3
C10
C2
C1
O3
C15
Cl

Fig. 2