Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2-[(2-Aminophenylimino)(phenyl)methyl]-4,6-dichlorophenol

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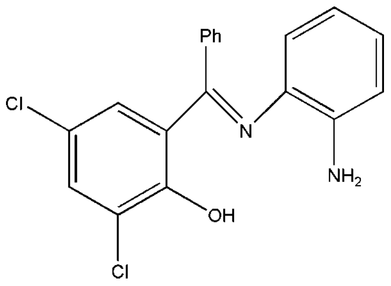
Received 28 August 2007; accepted 29 August 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.054; wR factor = 0.171; data-to-parameter ratio = 16.7.

The title compound, $C_{19}H_{14}Cl_2N_2O$, a tridentate Schiff base, has been synthesized and structurally characterized by X-ray structure analysis. The hydroxy and imino groups are involved in a resonance-assisted O—H···N hydrogen bond [O···N = 2.502 (3) Å]. Intermolecular N—H···O hydrogen bonds further stabilize the crystal packing.

Related literature

For related literature, see: Ai *et al.* (2006); Allen *et al.* (1987); Gilli *et al.* (2000).



Experimental

Crystal data

$C_{19}H_{14}Cl_2N_2O$
M_r = 357.22
Triclinic, $P\bar{1}$
a = 8.9833 (18) Å
b = 9.4191 (19) Å
c = 11.361 (2) Å
α = 66.61 (3)°
β = 72.87 (3)°
γ = 72.38 (3)°
V = 824.0 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 293 (2) K
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{\min}$ = 0.901, $T_{\max}$ = 0.963
3854 measured reflections
3625 independent reflections
2801 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.012

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.171
S = 1.13
3625 reflections
217 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.31 e Å^{-3}
$\Delta\rho_{\min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N2	0.85	1.87	2.502 (3)	130
N1—H1A···O1$^{\rm i}$	0.86	2.53	3.161 (4)	131
N1—H1B···N2	0.86	2.45	2.765 (4)	103

Symmetry code: (i) $-x+2, -y, -z+1$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

The authors gratefully acknowledge the support of the China Postdoctoral Science Foundation and the Knowledge Innovation Project of the Chinese Academy of Science (grant No. KZXCX2-YW-210).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2286).

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doi:10.1107/S160053680704233X

supplementary materials

Acta Cryst. (2007). E**63**, o3944 [**doi:10.1107/S160053680704233X**]

2-[(2-Aminophenylimino)(phenyl)methyl]-4,6-dichlorophenol

W.-H. Li, F.-Q. Liu, X.-D. Zhao, J.-Y. Deng and B.-R. Hou

Comment

In the title compound, (I), (Fig. 1), the atom N2 lies in the planes of imino-phenyl and hydroxy-phenyl groups. The dihedral angle formed by the two phenyls is 43.51 (7)°. The C7═N2 (1.298 (3) Å), C7—C14 (1.475 (4) Å) and O1—C19 (1.330 (3) Å) bond lengths are shorter than normal C═N (1.32 Å), C—C (1.54 Å) and O—C (1.44 Å) bonds (Allen *et al.*, 1987). The C14—C19 bond length (1.424 (4) Å) is longer than a normal C═C bond (1.40 Å in benzene). These differences in bond lengths are related to a resonance-assisted hydrogen bond (RAHBA) acting between molecules with conjugated multiple π bonds (Gilli *et al.*, 2000). Intermolecular N—H···O hydrogen bonds further stabilize the crystal packing. In the related 4-chlorophenyl compound similar resonance-assisted O—H···N hydrogen bonding was reported (Ai *et al.*, 2006).

Experimental

4,6-Dichloro-2-hydroxybenzophenone was prepared *via* the Fries rearrangement of 3,5-dichlorophenyl benzoate. Then this ketone (26.7 g, 0.1 mol), *o*-phenylenediamine (10.8 g, 0.1 mol), piperidine (9.4 g, 0.11 mol), and triethylorthoformate (16 ml) were refluxed in anhydrous ethanol (100 ml) to give a red product of the mono-condensed Schiff base 2-[(2-aminophenylimino)-phenylmethyl]-4,6-dichlorophenol. The precipitated solid was collected by filtration and washed twice with hot methanol. Single crystals suitable for X-ray measurements were obtained by slow evaporation of absolute ethanol and acetic ether (2:1 *v*/*v*) at room temperature.

Refinement

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with N—H = 0.86 Å, C—H = 0.93 Å and O—H = 0.85 Å, and with U_{iso}(H) = 1.2U_{eq}(C) for the aromatic H atoms or 1.2U_{eq}(parent) for the other atoms. H atoms were positioned geometrically (C—H = 0.93 Å) and allowed to ride on their parent atoms with U_{iso}(H) = 1.2 times U_{eq}(C).

Figures

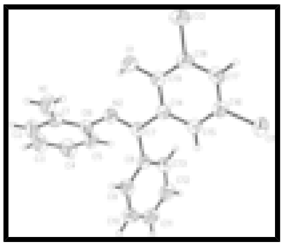

Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

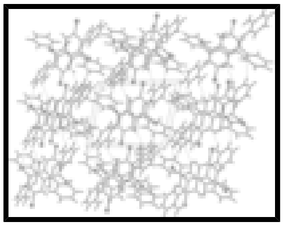

Fig. 2. The packing of (I), viewed down the *b* axis.

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Crystal data

$C_{19}H_{14}Cl_2N_2O$

$M_r = 357.22$

Triclinic, $P\overline{1}$

Hall symbol: -P 1

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$b = 9.4191$ (19) Å

$c = 11.361$ (2) Å

$\alpha = 66.61$ (3)°

$\beta = 72.87$ (3)°

$\gamma = 72.38$ (3)°

$V = 824.0$ (4) Å^3

$Z = 2$

$F_{000} = 368$

$D_x = 1.440$ Mg m^{-3}

Mo *K*α radiation
$\lambda = 0.71073$ Å

Cell parameters from 2933 reflections

$\theta = 2.5$–26.0°

$\mu = 0.40$ mm^{-1}

$T = 293$ (2) K

Block, colourless

0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293(2)$ K

thin–slice ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 2004)

$T_{\min} = 0.901$, $T_{\max} = 0.963$

3854 measured reflections

3625 independent reflections

2801 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.012$

$\theta_{\max} = 27.1$°

$\theta_{\min} = 2.0$°

$h = -10 \rightarrow 11$

$k = -11 \rightarrow 9$

$l = -14 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.054$

$wR(F^2) = 0.171$

$S = 1.13$

3625 reflections

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0797P)^2 + 0.556P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.31$ e Å^{-3}

217 parameters	$\Delta\rho_{min}$ = −0.30 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 > 2sigma(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}
Cl1	0.02655 (9)	0.32831 (12)	0.43610 (8)	0.0610 (3)
Cl2	0.62563 (10)	0.17475 (13)	0.18588 (8)	0.0667 (3)
O1	0.7144 (2)	0.1622 (3)	0.4170 (2)	0.0484 (5)
H1	0.7376	0.2060	0.4599	0.058*
N1	0.9269 (3)	−0.0348 (3)	0.6999 (3)	0.0603 (7)
H1A	1.0099	−0.1070	0.7198	0.072*
H1B	0.8726	−0.0410	0.6518	0.072*
N2	0.6616 (3)	0.1981 (3)	0.6334 (2)	0.0402 (5)
C1	0.8814 (3)	0.0902 (4)	0.7449 (3)	0.0465 (7)
C2	0.9686 (4)	0.1012 (5)	0.8227 (3)	0.0604 (9)
H2B	1.0592	0.0237	0.8426	0.072*
C3	0.9244 (5)	0.2225 (5)	0.8704 (4)	0.0690 (11)
H3A	0.9853	0.2263	0.9221	0.083*
C4	0.7907 (4)	0.3402 (4)	0.8434 (3)	0.0585 (9)
H4A	0.7613	0.4231	0.8760	0.070*
C5	0.7012 (4)	0.3318 (4)	0.7666 (3)	0.0486 (7)
H5A	0.6120	0.4111	0.7461	0.058*
C6	0.7427 (3)	0.2074 (4)	0.7199 (3)	0.0399 (6)
C7	0.5084 (3)	0.2380 (3)	0.6407 (3)	0.0374 (6)
C8	0.3940 (3)	0.2836 (3)	0.7510 (3)	0.0361 (6)
C9	0.3843 (3)	0.1752 (4)	0.8777 (3)	0.0430 (6)
H9A	0.4420	0.0716	0.8924	0.052*
C10	0.2890 (4)	0.2213 (4)	0.9816 (3)	0.0529 (8)
H10A	0.2812	0.1483	1.0661	0.063*
C11	0.2051 (4)	0.3761 (5)	0.9603 (3)	0.0561 (8)
H11A	0.1441	0.4080	1.0308	0.067*
C12	0.2117 (4)	0.4822 (4)	0.8356 (3)	0.0538 (8)
H12A	0.1528	0.5854	0.8215	0.065*
C13	0.3049 (3)	0.4373 (4)	0.7306 (3)	0.0469 (7)
H13A	0.3083	0.5099	0.6460	0.056*

C14	0.4484 (3)	0.2356 (3)	0.5341 (3)	0.0367 (6)
C15	0.2839 (3)	0.2715 (4)	0.5370 (3)	0.0424 (6)
H15A	0.2117	0.2914	0.6093	0.051*
C16	0.2297 (3)	0.2773 (4)	0.4349 (3)	0.0444 (7)
C17	0.3345 (3)	0.2457 (4)	0.3270 (3)	0.0444 (7)
H17A	0.2962	0.2488	0.2582	0.053*
C18	0.4950 (3)	0.2097 (4)	0.3227 (3)	0.0427 (6)
C19	0.5575 (3)	0.2001 (3)	0.4248 (3)	0.0398 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0374 (4)	0.0925 (7)	0.0603 (5)	−0.0136 (4)	−0.0155 (3)	−0.0289 (5)
Cl2	0.0573 (5)	0.1012 (8)	0.0515 (5)	−0.0093 (5)	−0.0022 (4)	−0.0475 (5)
O1	0.0349 (10)	0.0656 (14)	0.0490 (11)	−0.0030 (9)	−0.0081 (9)	−0.0294 (10)
N1	0.0488 (16)	0.0594 (17)	0.0722 (19)	0.0062 (13)	−0.0190 (14)	−0.0297 (15)
N2	0.0346 (12)	0.0504 (14)	0.0392 (12)	−0.0050 (10)	−0.0101 (9)	−0.0196 (11)
C1	0.0353 (14)	0.0550 (18)	0.0408 (15)	−0.0114 (13)	−0.0053 (12)	−0.0077 (13)
C2	0.0439 (18)	0.069 (2)	0.063 (2)	−0.0079 (16)	−0.0256 (16)	−0.0095 (18)
C3	0.064 (2)	0.090 (3)	0.063 (2)	−0.032 (2)	−0.0342 (18)	−0.009 (2)
C4	0.064 (2)	0.067 (2)	0.060 (2)	−0.0212 (17)	−0.0187 (17)	−0.0274 (17)
C5	0.0432 (16)	0.0547 (18)	0.0518 (17)	−0.0080 (13)	−0.0137 (13)	−0.0206 (15)
C6	0.0327 (13)	0.0526 (16)	0.0330 (13)	−0.0101 (12)	−0.0069 (11)	−0.0119 (12)
C7	0.0357 (14)	0.0412 (15)	0.0340 (13)	−0.0052 (11)	−0.0093 (11)	−0.0117 (11)
C8	0.0317 (13)	0.0468 (15)	0.0352 (13)	−0.0097 (11)	−0.0090 (10)	−0.0168 (12)
C9	0.0442 (15)	0.0472 (16)	0.0425 (15)	−0.0092 (12)	−0.0128 (12)	−0.0176 (13)
C10	0.0483 (17)	0.078 (2)	0.0351 (14)	−0.0207 (16)	−0.0063 (13)	−0.0179 (15)
C11	0.0426 (17)	0.085 (3)	0.0525 (18)	−0.0109 (16)	−0.0032 (14)	−0.0417 (18)
C12	0.0418 (16)	0.0587 (19)	0.069 (2)	−0.0003 (14)	−0.0115 (15)	−0.0374 (17)
C13	0.0403 (15)	0.0511 (17)	0.0438 (15)	−0.0037 (13)	−0.0096 (12)	−0.0139 (13)
C14	0.0350 (13)	0.0438 (15)	0.0338 (13)	−0.0083 (11)	−0.0084 (11)	−0.0144 (11)
C15	0.0330 (13)	0.0555 (17)	0.0352 (13)	−0.0079 (12)	−0.0052 (11)	−0.0140 (13)
C16	0.0350 (14)	0.0544 (17)	0.0448 (15)	−0.0132 (12)	−0.0115 (12)	−0.0126 (13)
C17	0.0470 (16)	0.0543 (17)	0.0360 (14)	−0.0116 (13)	−0.0126 (12)	−0.0158 (13)
C18	0.0438 (15)	0.0499 (16)	0.0363 (14)	−0.0092 (12)	−0.0040 (12)	−0.0198 (13)
C19	0.0344 (14)	0.0439 (15)	0.0371 (14)	−0.0052 (11)	−0.0068 (11)	−0.0121 (12)

Geometric parameters (Å, °)

Cl1—C16	1.738 (3)	C7—C8	1.487 (4)
Cl2—C18	1.732 (3)	C8—C13	1.390 (4)
O1—C19	1.330 (3)	C8—C9	1.391 (4)
O1—H1	0.8500	C9—C10	1.379 (4)
N1—C1	1.370 (4)	C9—H9A	0.9300
N1—H1A	0.8600	C10—C11	1.384 (5)
N1—H1B	0.8600	C10—H10A	0.9300
N2—C7	1.298 (3)	C11—C12	1.368 (5)
N2—C6	1.426 (3)	C11—H11A	0.9300
C1—C2	1.392 (5)	C12—C13	1.379 (4)

C1—C6	1.407 (4)	C12—H12A	0.9300
C2—C3	1.358 (6)	C13—H13A	0.9300
C2—H2B	0.9300	C14—C15	1.405 (4)
C3—C4	1.381 (5)	C14—C19	1.424 (4)
C3—H3A	0.9300	C15—C16	1.362 (4)
C4—C5	1.385 (4)	C15—H15A	0.9300
C4—H4A	0.9300	C16—C17	1.385 (4)
C5—C6	1.380 (4)	C17—C18	1.368 (4)
C5—H5A	0.9300	C17—H17A	0.9300
C7—C14	1.475 (4)	C18—C19	1.393 (4)

C19—O1—H1	109.1	C8—C9—H9A	120.0
C1—N1—H1A	120.0	C9—C10—C11	120.1 (3)
C1—N1—H1B	120.0	C9—C10—H10A	120.0
H1A—N1—H1B	120.0	C11—C10—H10A	120.0
C7—N2—C6	125.4 (2)	C12—C11—C10	120.1 (3)
N1—C1—C2	120.4 (3)	C12—C11—H11A	120.0
N1—C1—C6	122.1 (3)	C10—C11—H11A	120.0
C2—C1—C6	117.4 (3)	C11—C12—C13	120.5 (3)
C3—C2—C1	121.6 (3)	C11—C12—H12A	119.8
C3—C2—H2B	119.2	C13—C12—H12A	119.8
C1—C2—H2B	119.2	C12—C13—C8	120.0 (3)
C2—C3—C4	121.2 (3)	C12—C13—H13A	120.0
C2—C3—H3A	119.4	C8—C13—H13A	120.0
C4—C3—H3A	119.4	C15—C14—C19	119.2 (2)
C3—C4—C5	118.4 (3)	C15—C14—C7	120.8 (2)
C3—C4—H4A	120.8	C19—C14—C7	120.0 (2)
C5—C4—H4A	120.8	C16—C15—C14	120.5 (3)
C6—C5—C4	121.0 (3)	C16—C15—H15A	119.8
C6—C5—H5A	119.5	C14—C15—H15A	119.8
C4—C5—H5A	119.5	C15—C16—C17	121.0 (3)
C5—C6—C1	120.3 (3)	C15—C16—Cl1	120.5 (2)
C5—C6—N2	123.4 (3)	C17—C16—Cl1	118.6 (2)
C1—C6—N2	116.0 (3)	C18—C17—C16	119.4 (3)
N2—C7—C14	117.3 (2)	C18—C17—H17A	120.3
N2—C7—C8	123.0 (2)	C16—C17—H17A	120.3
C14—C7—C8	119.7 (2)	C17—C18—C19	122.2 (3)
C13—C8—C9	119.3 (3)	C17—C18—Cl2	119.1 (2)
C13—C8—C7	120.7 (2)	C19—C18—Cl2	118.6 (2)
C9—C8—C7	119.9 (2)	O1—C19—C18	120.0 (2)
C10—C9—C8	120.0 (3)	O1—C19—C14	122.2 (2)
C10—C9—H9A	120.0	C18—C19—C14	117.7 (2)

N1—C1—C2—C3	−178.9 (3)	C11—C12—C13—C8	−0.4 (5)
C6—C1—C2—C3	−1.6 (5)	C9—C8—C13—C12	1.8 (4)
C1—C2—C3—C4	0.0 (6)	C7—C8—C13—C12	−173.3 (3)
C2—C3—C4—C5	0.3 (6)	N2—C7—C14—C15	177.6 (3)
C3—C4—C5—C6	1.2 (5)	C8—C7—C14—C15	−1.8 (4)
C4—C5—C6—C1	−2.9 (5)	N2—C7—C14—C19	−3.8 (4)
C4—C5—C6—N2	−175.6 (3)	C8—C7—C14—C19	176.8 (2)

N1—C1—C6—C5	−179.8 (3)	C19—C14—C15—C16	−2.2 (4)
C2—C1—C6—C5	3.1 (4)	C7—C14—C15—C16	176.5 (3)
N1—C1—C6—N2	−6.5 (4)	C14—C15—C16—C17	1.1 (5)
C2—C1—C6—N2	176.3 (3)	C14—C15—C16—Cl1	−177.8 (2)
C7—N2—C6—C5	−40.2 (4)	C15—C16—C17—C18	−0.7 (5)
C7—N2—C6—C1	146.7 (3)	Cl1—C16—C17—C18	178.2 (2)
C6—N2—C7—C14	173.7 (2)	C16—C17—C18—C19	1.5 (5)
C6—N2—C7—C8	−6.8 (4)	C16—C17—C18—Cl2	−178.5 (2)
N2—C7—C8—C13	113.4 (3)	C17—C18—C19—O1	178.9 (3)
C14—C7—C8—C13	−67.2 (4)	Cl2—C18—C19—O1	−1.1 (4)
N2—C7—C8—C9	−61.6 (4)	C17—C18—C19—C14	−2.6 (4)
C14—C7—C8—C9	117.8 (3)	Cl2—C18—C19—C14	177.5 (2)
C13—C8—C9—C10	−1.0 (4)	C15—C14—C19—O1	−178.7 (3)
C7—C8—C9—C10	174.1 (3)	C7—C14—C19—O1	2.7 (4)
C8—C9—C10—C11	−1.1 (4)	C15—C14—C19—C18	2.8 (4)
C9—C10—C11—C12	2.4 (5)	C7—C14—C19—C18	−175.8 (3)
C10—C11—C12—C13	−1.7 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N2	0.85	1.87	2.502 (3)	130
N1—H1A···O1^{i}	0.86	2.53	3.161 (4)	131
N1—H1B···N2	0.86	2.45	2.765 (4)	103

Symmetry codes: (i) −*x*+2, −*y*, −*z*+1.

Fig. 1

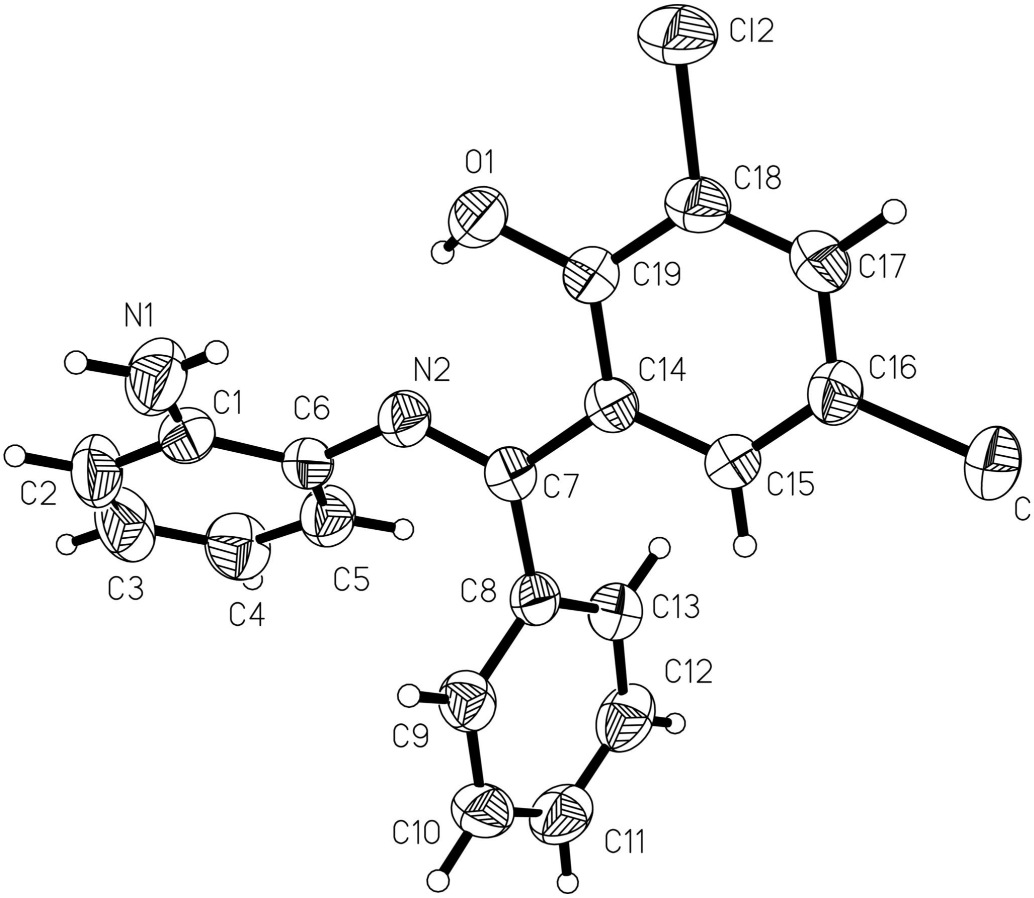
Cl2
O1
C18
C19
C17
N1
N2
C14
C16
C1
C6
C2
C7
C15
Cl1
C5
C3
C4
C8
C13
C12
C9
C10
C11

Fig. 2