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2,4-Dichloro-6-(8-quinolylaminomethylene)cyclohexa-2,4-dien-1-one methanol solvate

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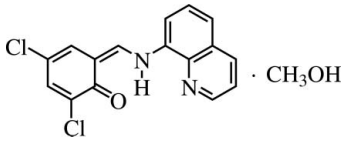
Key indicators: single-crystal X-ray study; T = 93 K; mean σ(C–C) = 0.002 Å; R factor = 0.028; wR factor = 0.078; data-to-parameter ratio = 16.1.

The main molecule of the title methanol solvate, $C_{16}H_{10}Cl_2N_2O\cdot CH_3OH$, exists in the keto form and the C═O and N—H bonds are mutually *cis* in the crystal structure. The dihedral angle between the quinoline and benzene rings is 11.17 (3)°. A bifurcated intramolecular N—H···(O,N) hydrogen bond is present as well as an O—H···O hydrogen bond. In the crystal, C—H···O interactions link the 3,5-dichlorosalicylidene-8-aminoquinoline and methanol molecules.

Related literature

For a related structure, see: Sakane *et al.* (2006).



Experimental

Crystal data

$C_{16}H_{10}Cl_2N_2O\cdot CH_4O$
M_r = 349.22
Triclinic, $P\overline{1}$
a = 7.044 (2) Å
b = 8.139 (3) Å
c = 13.935 (5) Å
α = 88.030 (11)°
β = 80.205 (9)°
γ = 73.611 (7)°
V = 755.2 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.44 mm^{-1}
T = 93 K
0.71 × 0.24 × 0.18 mm

Data collection

Rigaku Mercury diffractometer
Absorption correction: multi-scan (*REQAB*; Jacobson, 1998)
T_{min} = 0.745, T_{max} = 0.925
7177 measured reflections
3357 independent reflections
3262 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.078
S = 1.01
3357 reflections
209 parameters
H-atom parameters not refined
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H14···O1	0.82	1.95	2.7694 (14)	179
N2—H7···O1	0.86	1.92	2.6094 (12)	137
N2—H7···N1	0.86	2.28	2.6758 (13)	108
C8—H6···O2^{i}	0.97	2.70	3.6425 (14)	166
C10—H8···O2^{i}	0.98	2.27	3.2026 (13)	160

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

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2007); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CrystalStructure*; software used to prepare material for publication: *CrystalStructure*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2250).

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supplementary materials

Acta Cryst. (2010). E**66**, o429 [**doi:10.1107/S1600536810002345**]

2,4-Dichloro-6-(8-quinolylaminomethylene)cyclohexa-2,4-dien-1-one methanol solvate

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Comment

In the present work, the title compound, (I), was prepared and its crystal structure determined, to explore the modification effect of Schiff base ligand on the fluorescence of metal complexes of 2-hydroxy-1-naphthaldehydene-8-aminoquinoline ($C_{20}H_{14}N_2O$), (II) (Sakane *et al.*, 2006). The molecule of (I) (Fig. 1) exists in the keto form and the C═O and N—H bonds are mutually *cis* which is similar to that found in the structure of (II). In the structure of (I), *N*—*H*···O carbonyl and *N*—*H*···N pyridine intramolecular hydrogen bonds exist (Table 1). In addition, there is a formal intermolecular hydrogen-bonding association between the molecules of 3,5-dichlorosalicylidene-8-aminoquinoline and methanol solvate (Table 1 and Fig. 2).

Experimental

Refluxing a suspension of 8-aminoquinoline (145 mg, 1.0 mmol) and 3,5-dichloro-salicylaldehyde (191 mg, 1.0 mmol) in methanol (3 ml) at 338 K for one hour gave vivid red powder. Slow evaporation of the supernatant solution gave vivid red plate like crystals of $C_{16}H_{10}Cl_2N_2O$ (I).CH_3OH. Yield 302 mg (95%). Anal. Found: C, 58.03; H, 3.73; N, 8.21%. Calcd for $C_{17}H_{14}N_2O_2$: C, 58.47; H, 4.04; N, 8.02%.

Refinement

The positions of all H atoms were located from difference maps and refined with restrained distances (N–H = 0.86 Å; C–H = 0.92-1.00 Å). The isotropic displacement parameters for H atoms were fixed at $1.2U_{eq}$ of their carrier atoms.

Figures

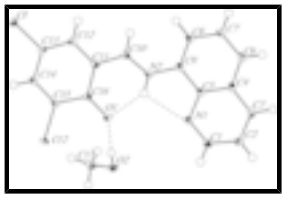

Fig. 1. Molecular configuration and atom-numbering scheme for (I).CH_3OH with displacement ellipsoids drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

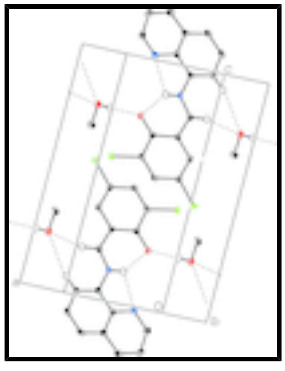

Fig. 2. Molecular packing of (I).CH_3OH in the unit cell.

2,4-Dichloro-6-(8-quinolylaminomethylene)cyclohexa-2,4-dien-1-one methanol solvate

Crystal data

$C_{16}H_{10}Cl_2N_2O\cdot CH_4O$	$Z = 2$
$M_r = 349.22$	$F(000) = 360.00$
Triclinic, $P\overline{1}$	$D_x = 1.536$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71070$ Å
$a = 7.044$ (2) Å	Cell parameters from 1999 reflections
$b = 8.139$ (3) Å	$\theta = 5.6$–$27.5°$
$c = 13.935$ (5) Å	$\mu = 0.44$ mm^{-1}
$\alpha = 88.030$ (11)°	$T = 93$ K
$\beta = 80.205$ (9)°	Platelet, red
$\gamma = 73.611$ (7)°	0.71 × 0.24 × 0.18 mm
$V = 755.2$ (4) Å^3	

Data collection

Rigaku Mercury diffractometer	3357 independent reflections
Radiation source: Mo Kα	3262 reflections with $F^2 > 2\sigma(F^2)$
Detector resolution: 14.63 pixels mm^{-1}	$R_{int} = 0.022$
ω scans	$\theta_{max} = 27.5°$
Absorption correction: multi-scan (REQAB; Jacobson, 1998)	$h = -8\rightarrow9$
$T_{min} = 0.745$, $T_{max} = 0.925$	$k = -10\rightarrow10$
7177 measured reflections	$l = -18\rightarrow17$

Refinement

Refinement on F^2	H-atom parameters not refined
$R[F^2 > 2\sigma(F^2)] = 0.028$	$w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 0.4083P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.078$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.01$	$\Delta\rho_{max} = 0.42$ e Å^{-3}
3357 reflections	$\Delta\rho_{min} = -0.28$ e Å^{-3}
209 parameters	

Special details

Geometry. The dihedral angle between the quinoline (C1~C9, N1) and the benzene rings (C11~C16) is 11.17 (3)°: Mean deviations of the atoms from the former and latter planes are 0.014 and 0.004 Å, respectively.

Refinement. Refinement was performed using all reflections. The weighted *R*-factor (*wR*) and goodness of fit (*S*) are based on F^2. *R*-factor (gt) are based on *F*. The threshold expression of $F^2 > 2.0\ \sigma(F^2)$ is used only for calculating *R*-factor (gt).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl(2)	1.24813 (4)	0.00187 (3)	0.581434 (19)	0.01543 (8)
Cl(1)	1.28180 (4)	0.62464 (3)	0.452063 (19)	0.01392 (8)
O(1)	1.03744 (12)	0.19010 (10)	0.76600 (6)	0.01351 (16)
O(2)	0.81786 (14)	−0.04501 (11)	0.77301 (6)	0.01778 (18)
N(1)	0.83476 (14)	0.24199 (12)	1.01752 (7)	0.01204 (18)
N(2)	0.88903 (14)	0.46400 (12)	0.87656 (7)	0.01041 (17)
C(1)	0.80423 (17)	0.13097 (15)	1.08523 (9)	0.0146 (2)
C(2)	0.68177 (18)	0.17741 (16)	1.17744 (8)	0.0161 (2)
C(3)	0.59125 (17)	0.34651 (16)	1.19956 (8)	0.0149 (2)
C(4)	0.62125 (16)	0.47193 (15)	1.12960 (8)	0.0119 (2)
C(5)	0.74388 (16)	0.41182 (14)	1.03902 (8)	0.0105 (2)
C(6)	0.53722 (17)	0.65040 (15)	1.14652 (8)	0.0146 (2)
C(7)	0.57568 (17)	0.76438 (14)	1.07635 (9)	0.0150 (2)
C(8)	0.69577 (17)	0.70710 (14)	0.98534 (8)	0.0128 (2)
C(9)	0.77592 (16)	0.53372 (14)	0.96660 (8)	0.0104 (2)
C(10)	0.94877 (16)	0.54862 (14)	0.80119 (8)	0.0111 (2)
C(11)	1.05243 (16)	0.46773 (14)	0.71172 (8)	0.0109 (2)
C(16)	1.09174 (16)	0.28530 (14)	0.69891 (8)	0.0106 (2)
C(15)	1.19770 (17)	0.21966 (14)	0.60378 (8)	0.0118 (2)
C(14)	1.25470 (16)	0.32146 (14)	0.53118 (8)	0.0118 (2)
C(13)	1.20940 (16)	0.49959 (14)	0.54743 (8)	0.0114 (2)
C(12)	1.11163 (16)	0.57250 (14)	0.63530 (8)	0.0114 (2)
C(17)	0.72759 (19)	−0.02464 (16)	0.68811 (9)	0.0184 (2)
H(1)	0.8725	0.0177	1.0721	0.018*
H(2)	0.6749	0.0946	1.2238	0.019*
H(3)	0.5055	0.3897	1.2617	0.018*
H(4)	0.4564	0.6860	1.2066	0.018*
H(5)	0.5203	0.8855	1.0900	0.018*
H(6)	0.7140	0.7905	0.9363	0.015*
H(7)	0.9237	0.3548	0.8700	0.012*
H(8)	0.9221	0.6724	0.8071	0.013*
H(10)	1.3178	0.2802	0.4703	0.014*
H(9)	1.0851	0.6895	0.6459	0.014*
H(11)	0.6033	0.0671	0.6913	0.022*
H(12)	0.6853	−0.1289	0.6783	0.022*
H(13)	0.8203	−0.0129	0.6303	0.022*
H(14)	0.8844	0.0234	0.7709	0.021*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl(2)	0.02066 (16)	0.00883 (14)	0.01551 (14)	−0.00396 (10)	0.00090 (10)	−0.00332 (10)
Cl(1)	0.01419 (14)	0.01408 (14)	0.01329 (14)	−0.00503 (10)	−0.00052 (10)	0.00295 (10)
O(1)	0.0180 (4)	0.0099 (3)	0.0130 (3)	−0.0053 (3)	−0.0011 (3)	−0.0001 (2)

O(2)	0.0235 (4)	0.0142 (4)	0.0197 (4)	−0.0104 (3)	−0.0062 (3)	0.0020 (3)
N(1)	0.0115 (4)	0.0102 (4)	0.0138 (4)	−0.0022 (3)	−0.0018 (3)	−0.0008 (3)
N(2)	0.0114 (4)	0.0082 (4)	0.0114 (4)	−0.0023 (3)	−0.0015 (3)	−0.0019 (3)
C(1)	0.0127 (5)	0.0123 (5)	0.0171 (5)	−0.0012 (4)	−0.0018 (4)	0.0009 (4)
C(2)	0.0153 (5)	0.0181 (5)	0.0141 (5)	−0.0044 (4)	−0.0017 (4)	0.0046 (4)
C(3)	0.0126 (5)	0.0200 (5)	0.0117 (5)	−0.0043 (4)	−0.0011 (4)	0.0001 (4)
C(4)	0.0102 (4)	0.0148 (5)	0.0114 (4)	−0.0038 (4)	−0.0026 (3)	−0.0019 (4)
C(5)	0.0089 (4)	0.0113 (5)	0.0117 (4)	−0.0027 (3)	−0.0029 (3)	−0.0015 (3)
C(6)	0.0131 (5)	0.0164 (5)	0.0136 (5)	−0.0031 (4)	−0.0004 (4)	−0.0057 (4)
C(7)	0.0148 (5)	0.0110 (5)	0.0187 (5)	−0.0025 (4)	−0.0018 (4)	−0.0054 (4)
C(8)	0.0131 (4)	0.0114 (5)	0.0145 (5)	−0.0041 (4)	−0.0026 (4)	−0.0012 (3)
C(9)	0.0087 (4)	0.0118 (5)	0.0111 (4)	−0.0030 (3)	−0.0019 (3)	−0.0021 (3)
C(10)	0.0108 (4)	0.0099 (4)	0.0135 (4)	−0.0031 (3)	−0.0034 (3)	−0.0010 (3)
C(11)	0.0107 (4)	0.0101 (4)	0.0123 (4)	−0.0030 (3)	−0.0028 (3)	−0.0006 (3)
C(16)	0.0104 (4)	0.0104 (4)	0.0116 (4)	−0.0032 (3)	−0.0029 (3)	−0.0004 (3)
C(15)	0.0121 (4)	0.0090 (4)	0.0144 (5)	−0.0028 (3)	−0.0024 (3)	−0.0027 (3)
C(14)	0.0107 (4)	0.0132 (5)	0.0115 (4)	−0.0031 (4)	−0.0015 (3)	−0.0024 (3)
C(13)	0.0109 (4)	0.0121 (4)	0.0119 (4)	−0.0043 (3)	−0.0026 (3)	0.0027 (3)
C(12)	0.0113 (4)	0.0088 (4)	0.0145 (5)	−0.0030 (3)	−0.0032 (3)	−0.0001 (3)
C(17)	0.0188 (5)	0.0170 (5)	0.0199 (5)	−0.0049 (4)	−0.0043 (4)	−0.0017 (4)

Geometric parameters (Å, °)

Cl(2)—C(15)	1.7350 (11)	C(11)—C(12)	1.4220 (15)
Cl(1)—C(13)	1.7411 (11)	C(16)—C(15)	1.4446 (14)
O(1)—C(16)	1.2714 (14)	C(15)—C(14)	1.3650 (16)
O(2)—C(17)	1.4193 (16)	C(14)—C(13)	1.4114 (15)
N(1)—C(1)	1.3172 (15)	C(13)—C(12)	1.3631 (14)
N(1)—C(5)	1.3690 (13)	O(2)—H(14)	0.820
N(2)—C(9)	1.4088 (13)	N(2)—H(7)	0.856
N(2)—C(10)	1.3107 (14)	C(1)—H(1)	0.920
C(1)—C(2)	1.4179 (15)	C(2)—H(2)	0.923
C(2)—C(3)	1.3649 (16)	C(3)—H(3)	0.980
C(3)—C(4)	1.4214 (16)	C(6)—H(4)	0.933
C(4)—C(5)	1.4184 (14)	C(7)—H(5)	0.965
C(4)—C(6)	1.4166 (15)	C(8)—H(6)	0.965
C(5)—C(9)	1.4261 (15)	C(10)—H(8)	0.975
C(6)—C(7)	1.3709 (16)	C(14)—H(10)	0.915
C(7)—C(8)	1.4122 (15)	C(12)—H(9)	0.929
C(8)—C(9)	1.3798 (15)	C(17)—H(11)	0.973
C(10)—C(11)	1.4130 (14)	C(17)—H(12)	0.996
C(11)—C(16)	1.4431 (16)	C(17)—H(13)	0.967
C(1)—N(1)—C(5)	117.32 (9)	C(14)—C(13)—C(12)	121.14 (10)
C(9)—N(2)—C(10)	126.88 (9)	C(11)—C(12)—C(13)	119.52 (10)
N(1)—C(1)—C(2)	123.85 (10)	C(17)—O(2)—H(14)	108.2
C(1)—C(2)—C(3)	119.18 (11)	C(9)—N(2)—H(7)	117.1
C(2)—C(3)—C(4)	119.30 (9)	C(10)—N(2)—H(7)	116.0
C(3)—C(4)—C(5)	117.05 (9)	N(1)—C(1)—H(1)	117.0
C(3)—C(4)—C(6)	123.46 (9)	C(2)—C(1)—H(1)	119.1

C(5)—C(4)—C(6)	119.49 (10)	C(1)—C(2)—H(2)	119.7
N(1)—C(5)—C(4)	123.28 (10)	C(3)—C(2)—H(2)	120.8
N(1)—C(5)—C(9)	118.02 (9)	C(2)—C(3)—H(3)	124.5
C(4)—C(5)—C(9)	118.70 (9)	C(4)—C(3)—H(3)	116.2
C(4)—C(6)—C(7)	120.33 (9)	C(4)—C(6)—H(4)	117.5
C(6)—C(7)—C(8)	121.05 (9)	C(7)—C(6)—H(4)	122.2
C(7)—C(8)—C(9)	119.58 (10)	C(6)—C(7)—H(5)	119.1
N(2)—C(9)—C(5)	115.40 (9)	C(8)—C(7)—H(5)	119.9
N(2)—C(9)—C(8)	123.77 (10)	C(7)—C(8)—H(6)	118.9
C(5)—C(9)—C(8)	120.81 (9)	C(9)—C(8)—H(6)	121.4
N(2)—C(10)—C(11)	122.74 (10)	N(2)—C(10)—H(8)	118.8
C(10)—C(11)—C(16)	120.40 (10)	C(11)—C(10)—H(8)	118.4
C(10)—C(11)—C(12)	117.76 (10)	C(15)—C(14)—H(10)	122.9
C(16)—C(11)—C(12)	121.83 (9)	C(13)—C(14)—H(10)	117.2
O(1)—C(16)—C(11)	122.66 (9)	C(11)—C(12)—H(9)	119.8
O(1)—C(16)—C(15)	122.74 (10)	C(13)—C(12)—H(9)	120.6
C(11)—C(16)—C(15)	114.60 (9)	O(2)—C(17)—H(11)	115.5
Cl(2)—C(15)—C(16)	117.70 (8)	O(2)—C(17)—H(12)	108.9
Cl(2)—C(15)—C(14)	119.29 (8)	O(2)—C(17)—H(13)	112.0
C(16)—C(15)—C(14)	123.00 (10)	H(11)—C(17)—H(12)	103.0
C(15)—C(14)—C(13)	119.89 (9)	H(11)—C(17)—H(13)	109.7
Cl(1)—C(13)—C(14)	118.23 (7)	H(12)—C(17)—H(13)	107.0
Cl(1)—C(13)—C(12)	120.63 (8)		
C(1)—N(1)—C(5)—C(4)	0.26 (18)	C(6)—C(7)—C(8)—C(9)	−0.04 (15)
C(1)—N(1)—C(5)—C(9)	179.81 (11)	C(7)—C(8)—C(9)—N(2)	176.74 (11)
C(5)—N(1)—C(1)—C(2)	0.84 (19)	C(7)—C(8)—C(9)—C(5)	−1.86 (19)
C(9)—N(2)—C(10)—C(11)	−176.33 (11)	N(2)—C(10)—C(11)—C(16)	1.95 (18)
C(10)—N(2)—C(9)—C(5)	−175.47 (11)	N(2)—C(10)—C(11)—C(12)	−179.38 (11)
C(10)—N(2)—C(9)—C(8)	5.9 (2)	C(10)—C(11)—C(16)—O(1)	−0.39 (18)
N(1)—C(1)—C(2)—C(3)	−1.1 (2)	C(10)—C(11)—C(16)—C(15)	179.62 (10)
C(1)—C(2)—C(3)—C(4)	0.22 (19)	C(10)—C(11)—C(12)—C(13)	−179.06 (11)
C(2)—C(3)—C(4)—C(5)	0.77 (18)	C(16)—C(11)—C(12)—C(13)	−0.41 (17)
C(2)—C(3)—C(4)—C(6)	−178.65 (12)	C(12)—C(11)—C(16)—O(1)	−179.01 (11)
C(3)—C(4)—C(5)—N(1)	−1.05 (18)	C(12)—C(11)—C(16)—C(15)	1.00 (16)
C(3)—C(4)—C(5)—C(9)	179.40 (11)	O(1)—C(16)—C(15)—Cl(2)	0.90 (16)
C(3)—C(4)—C(6)—C(7)	178.72 (12)	O(1)—C(16)—C(15)—C(14)	179.36 (11)
C(5)—C(4)—C(6)—C(7)	−0.68 (19)	C(11)—C(16)—C(15)—Cl(2)	−179.10 (8)
C(6)—C(4)—C(5)—N(1)	178.39 (11)	C(11)—C(16)—C(15)—C(14)	−0.64 (17)
C(6)—C(4)—C(5)—C(9)	−1.16 (18)	Cl(2)—C(15)—C(14)—C(13)	178.14 (9)
N(1)—C(5)—C(9)—N(2)	4.16 (16)	C(16)—C(15)—C(14)—C(13)	−0.30 (18)
N(1)—C(5)—C(9)—C(8)	−177.13 (11)	C(15)—C(14)—C(13)—Cl(1)	−179.04 (9)
C(4)—C(5)—C(9)—N(2)	−176.26 (11)	C(15)—C(14)—C(13)—C(12)	0.97 (18)
C(4)—C(5)—C(9)—C(8)	2.45 (18)	Cl(1)—C(13)—C(12)—C(11)	179.40 (9)
C(4)—C(6)—C(7)—C(8)	1.31 (19)	C(14)—C(13)—C(12)—C(11)	−0.61 (17)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H14···O1	0.820	1.950	2.7694 (14)	179

N2—H7···O1	0.856	1.916	2.6094 (12)	137
N2—H7···N1	0.856	2.284	2.6758 (13)	108
C8—H6···O2^{i}	0.965	2.699	3.6425 (14)	166
C10—H8···O2^{i}	0.975	2.268	3.2026 (13)	160

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

Fig. 1

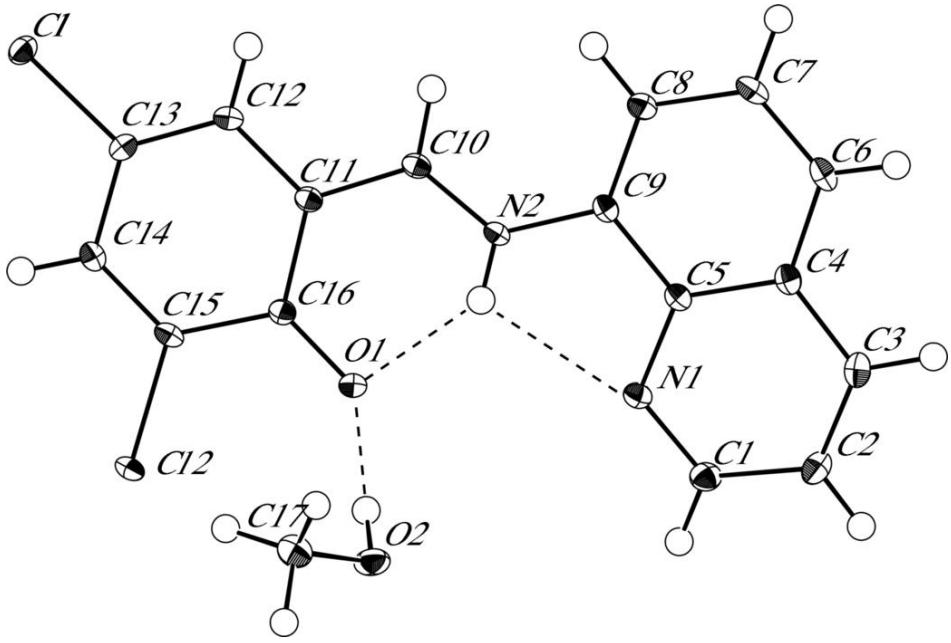
Cl
C13
C12
C11
C10
C8
C7
C6
C9
N2
C14
C5
C4
C15
C16
O1
C3
N1
Cl2
C1
C2
C17
O2

Fig. 2