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N′-[(*E*)-1-(3,5-Dichloro-2-hydroxyphenyl)ethylidene]-4-methoxybenzohydrazide monohydrate

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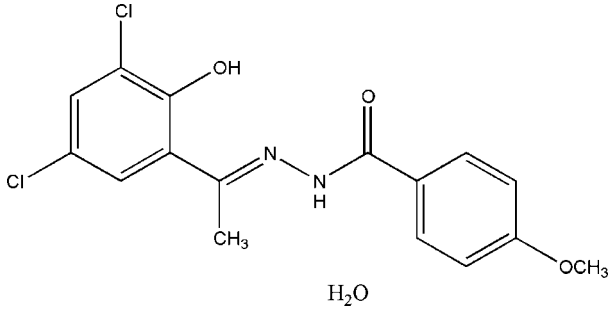
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.066; wR factor = 0.170; data-to-parameter ratio = 13.3.

The title compound, $C_{16}H_{14}Cl_2N_2O_3\cdot H_2O$, displays a *trans* conformation with respect to the C=N double bond. The dihedral angle between the two benzene rings is 4.98 (12)°. Intramolecular O—H···N and O—H···O hydrogen bonds occur. The crystal structure is stabilized by intermolecular O—H···O and N—H···O hydrogen bonds. In addition, there are π–π interactions between the chemically distinct benzene rings of inversion-related molecules [centroid–centroid separation = 3.715 (1) Å].

Related literature

For further details of the chemistry of the title compound, see: Carcelli *et al.* (1995); Salem (1998). For a related stucture, see: Chang *et al.* (2007).



Experimental

Crystal data

$C_{16}H_{14}Cl_2N_2O_3\cdot H_2O$
M_r = 371.21
Triclinic, $P\overline{1}$
a = 7.033 (5) Å
b = 7.516 (7) Å
c = 16.647 (10) Å
α = 85.105 (10)°
β = 81.386 (12)°
γ = 79.414 (10)°
V = 853.7 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 298 K
0.30 × 0.23 × 0.16 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.906, T_{max} = 0.946
4423 measured reflections
2936 independent reflections
1997 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.170
S = 1.00
2936 reflections
220 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O4	0.86	2.15	2.926 (5)	150
O1—H1···O2	0.82	2.58	3.287 (5)	146
O1—H1···N1	0.82	1.77	2.484 (5)	145
O4—H16···O1^{i}	0.85	2.09	2.887 (5)	156
O4—H15···O2^{ii}	0.85	1.88	2.726 (5)	176

Symmetry codes: (i) $-x+2, -y, -z+1$; (ii) $x, y+1, z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2270).

References

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supplementary materials

Acta Cryst. (2010). E**66**, o2667 [**doi:10.1107/S1600536810038328**]

N'-[(*E*)-1-(3,5-Dichloro-2-hydroxyphenyl)ethylidene]-4-methoxybenzohydrazide monohydrate

C.-H. He, J.-P. Zhang and J.-G. Chang

Comment

The chemistry of aroylhydrazones continues to attract much attention due to their coordination ability to metal ions and their biological activity (Carcelli *et al.*, 1995; Salem, 1998; Chang *et al.*, 2007). As an extension of work on the structural characterization of aroylhydrazone derivatives, the title compound, was synthesized and its crystal structure is reported here.

The title molecule displays a *trans* conformation with respect to the C7=N1 double bond (Fig. 1). The dihedral angle between the two benzene rings is 4.98 (12) °. The crystal structure is stabilized by intramolecular O—H···N, O—H···O and intermolecular O—H···O, N—H···O hydrogen bonds. (Table. 1, Figs. 1 and 2). There are π - π interactions between the chemically distinct benzene rings on inversion related molecules [*Cg*···*Cg* = 3.715 (1) Å; *Cg* represents a ring centroid].

Experimental

4-methoxybenzohydrazide (0.01 mol,1.66 g) was dissolved in anhydrous ethanol (50 ml), and 1-(3,5-dichloro-2-hydroxyphenyl)ethanone (0.01 mol, 2.05 g) was added. The reaction mixture was refluxed for 5 h with stirring, then the resulting precipitate was collected by filtration, washed several times with ethanol and dried *in vacuo* (yield 78%). The compound (1.0 mmol,0.35 g) was dissolved in dimethylformamide (30 ml) and kept at room temperature for 20d to obtain yellow single crystals suitable for X-ray diffraction.

Refinement

All H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H (methyl) = 0.96 Å, C—H (aromatic) = 0.93 Å, O—H = 0.82 Å, N—H = 0.86 Å and with U_{iso}(H) = 1.5U_{eq}(C_{methyl}, O and 1.2U_{eq}($C_{aromatic}$, $C_{methylene}$, N).

Figures

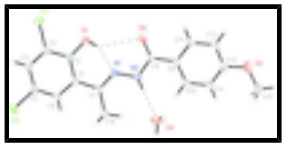

Fig. 1. The molecular structure of compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30° probability level. Dashed lines show intramolecular O—H···N, O—H···O and intermolecular N—H···O hydrogen bonds.

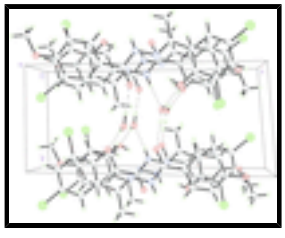

Fig. 2. Packing diagram of compound, Showing intermolecular O—H···O and N—H···O hydrogen bonds (dashed lines).

N'-[(*E*)-1-(3,5-Dichloro-2-hydroxyphenyl)ethylidene]-4- methoxybenzohydrazide monohydrate

Crystal data

$C_{16}H_{14}Cl_2N_2O_3 \cdot H_2O$	$Z = 2$
$M_r = 371.21$	$F(000) = 384$
Triclinic, $P\overline{1}$	$D_x = 1.444$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.033$ (5) Å	Cell parameters from 1429 reflections
$b = 7.516$ (7) Å	$\theta = 3.0$–$25.5°$
$c = 16.647$ (10) Å	$\mu = 0.40$ mm^{-1}
$\alpha = 85.105$ (10)°	$T = 298$ K
$\beta = 81.386$ (12)°	Plate, yellow
$\gamma = 79.414$ (10)°	0.30 × 0.23 × 0.16 mm
$V = 853.7$ (11) Å^3	

Data collection

Bruker APEXII CCD area-detector diffractometer	2936 independent reflections
Radiation source: fine-focus sealed tube	1997 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.025$
φ and ω scans	$\theta_{max} = 25.1°$, $\theta_{min} = 2.5°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$h = -8 \rightarrow 8$
$T_{min} = 0.906$, $T_{max} = 0.946$	$k = -6 \rightarrow 8$
4423 measured reflections	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.066$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.170$	H-atom parameters constrained
$S = 1.00$	$w = 1/[\sigma^2(F_o^2) + (0.0727P)^2 + 0.8688P]$ where $P = (F_o^2 + 2F_c^2)/3$
2936 reflections	$(\Delta/\sigma)_{max} < 0.001$
220 parameters	$\Delta\rho_{max} = 0.56$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.32$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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}
Cl1	1.0655 (2)	−0.37144 (18)	0.19059 (9)	0.0675 (5)
Cl2	0.8926 (3)	0.3075 (2)	0.05875 (8)	0.0774 (6)
O1	0.9149 (5)	−0.2090 (4)	0.34325 (19)	0.0462 (8)
H1	0.8675	−0.1599	0.3856	0.069*
O2	0.7613 (6)	−0.2102 (4)	0.5397 (2)	0.0611 (10)
O3	0.5111 (6)	0.0452 (6)	0.8985 (2)	0.0714 (12)
O4	0.7853 (6)	0.4252 (4)	0.5716 (3)	0.0710 (12)
N1	0.7805 (5)	0.0497 (5)	0.4303 (2)	0.0367 (9)
N2	0.7259 (5)	0.0879 (5)	0.5112 (2)	0.0392 (9)
H2	0.6983	0.1973	0.5264	0.047*
C1	0.8427 (6)	0.1022 (6)	0.2902 (3)	0.0360 (9)
C2	0.9122 (6)	−0.0844 (6)	0.2809 (3)	0.0374 (10)
C3	0.9789 (7)	−0.1428 (6)	0.2023 (3)	0.0442 (11)
C4	0.9757 (7)	−0.0251 (7)	0.1341 (3)	0.0488 (11)
H4	1.0221	−0.0668	0.0826	0.059*
C5	0.9020 (7)	0.1566 (7)	0.1441 (3)	0.0448 (11)
C6	0.8384 (7)	0.2200 (6)	0.2203 (3)	0.0419 (10)
H6	0.7918	0.3432	0.2255	0.050*
C7	0.7781 (6)	0.1720 (6)	0.3720 (3)	0.0357 (9)
C8	0.7186 (6)	−0.0562 (6)	0.5645 (3)	0.0387 (10)
C9	0.6577 (6)	−0.0211 (6)	0.6511 (3)	0.0387 (10)
C10	0.5662 (7)	0.1480 (7)	0.6775 (3)	0.0452 (11)
H10	0.5393	0.2442	0.6396	0.054*
C11	0.5141 (7)	0.1750 (7)	0.7606 (3)	0.0508 (12)
H11	0.4542	0.2887	0.7780	0.061*
C12	0.5526 (7)	0.0310 (7)	0.8165 (3)	0.0483 (11)
C13	0.6383 (7)	−0.1378 (7)	0.7914 (3)	0.0517 (12)
H13	0.6614	−0.2346	0.8294	0.062*
C14	0.6904 (7)	−0.1634 (7)	0.7088 (3)	0.0457 (11)
H14	0.7484	−0.2780	0.6919	0.055*
C15	0.4310 (10)	0.2206 (10)	0.9292 (4)	0.0819 (19)
H15A	0.5202	0.3029	0.9109	0.123*
H15B	0.4107	0.2103	0.9876	0.123*
H15C	0.3086	0.2657	0.9095	0.123*
C16	0.7148 (6)	0.3729 (4)	0.3805 (3)	0.0605 (15)
H16A	0.6850	0.3969	0.4371	0.091*
H16B	0.6008	0.4148	0.3542	0.091*
H16C	0.8183	0.4351	0.3555	0.091*

H15	0.7839	0.5382	0.5611	0.14 (3)*
H16	0.8905	0.3884	0.5919	0.09 (2)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0958 (12)	0.0380 (7)	0.0647 (9)	−0.0048 (7)	−0.0002 (8)	−0.0142 (6)
Cl2	0.1369 (15)	0.0568 (9)	0.0331 (7)	−0.0125 (9)	−0.0067 (8)	0.0091 (6)
O1	0.067 (2)	0.0312 (17)	0.0383 (18)	−0.0054 (15)	−0.0049 (16)	0.0012 (14)
O2	0.104 (3)	0.0296 (18)	0.046 (2)	−0.0090 (18)	−0.0052 (19)	0.0065 (15)
O3	0.089 (3)	0.084 (3)	0.034 (2)	−0.001 (2)	−0.0038 (19)	0.0013 (19)
O4	0.090 (3)	0.037 (2)	0.096 (3)	−0.0169 (19)	−0.044 (3)	0.007 (2)
N1	0.050 (2)	0.031 (2)	0.0279 (19)	−0.0080 (16)	−0.0040 (16)	0.0009 (15)
N2	0.053 (2)	0.0283 (19)	0.035 (2)	−0.0060 (16)	−0.0037 (17)	−0.0006 (16)
C1	0.038 (2)	0.039 (2)	0.032 (2)	−0.0095 (18)	−0.0057 (17)	−0.0004 (17)
C2	0.041 (2)	0.033 (2)	0.038 (2)	−0.0108 (18)	−0.0052 (18)	0.0006 (18)
C3	0.048 (2)	0.041 (2)	0.045 (2)	−0.0085 (19)	−0.005 (2)	−0.007 (2)
C4	0.057 (3)	0.049 (3)	0.040 (2)	−0.013 (2)	−0.003 (2)	−0.006 (2)
C5	0.058 (3)	0.043 (2)	0.034 (2)	−0.013 (2)	−0.007 (2)	0.0028 (19)
C6	0.048 (2)	0.038 (2)	0.038 (2)	−0.0076 (19)	−0.0049 (19)	0.0003 (19)
C7	0.043 (2)	0.031 (2)	0.033 (2)	−0.0059 (17)	−0.0058 (18)	0.0025 (17)
C8	0.047 (2)	0.029 (2)	0.039 (2)	−0.0067 (18)	−0.0073 (19)	0.0035 (18)
C9	0.040 (2)	0.040 (2)	0.037 (2)	−0.0101 (18)	−0.0062 (18)	0.0037 (18)
C10	0.051 (3)	0.043 (2)	0.039 (2)	−0.005 (2)	−0.005 (2)	0.002 (2)
C11	0.057 (3)	0.047 (3)	0.046 (3)	−0.006 (2)	−0.002 (2)	−0.002 (2)
C12	0.048 (3)	0.059 (3)	0.037 (2)	−0.008 (2)	−0.005 (2)	0.001 (2)
C13	0.051 (3)	0.058 (3)	0.043 (2)	−0.006 (2)	−0.008 (2)	0.011 (2)
C14	0.050 (3)	0.044 (2)	0.041 (2)	−0.006 (2)	−0.005 (2)	0.006 (2)
C15	0.100 (5)	0.092 (5)	0.048 (3)	−0.004 (4)	0.003 (3)	−0.018 (3)
C16	0.098 (4)	0.035 (3)	0.041 (3)	−0.001 (3)	−0.001 (3)	−0.001 (2)

Geometric parameters (Å, °)

Cl1—C3	1.731 (5)	C5—C6	1.376 (7)
Cl2—C5	1.742 (5)	C6—H6	0.9300
O1—C2	1.338 (5)	C7—C16	1.506 (5)
O1—H1	0.8200	C8—C9	1.474 (6)
O2—C8	1.231 (5)	C9—C14	1.385 (6)
O3—C12	1.362 (6)	C9—C10	1.391 (6)
O3—C15	1.440 (7)	C10—C11	1.399 (7)
O4—H15	0.8511	C10—H10	0.9300
O4—H16	0.8496	C11—C12	1.381 (7)
N1—C7	1.279 (5)	C11—H11	0.9300
N1—N2	1.383 (5)	C12—C13	1.372 (7)
N2—C8	1.344 (5)	C13—C14	1.389 (7)
N2—H2	0.8600	C13—H13	0.9300
C1—C6	1.401 (6)	C14—H14	0.9300
C1—C2	1.412 (6)	C15—H15A	0.9600
C1—C7	1.477 (6)	C15—H15B	0.9600

C2—C3	1.401 (6)	C15—H15C	0.9600
C3—C4	1.378 (7)	C16—H16A	0.9600
C4—C5	1.383 (7)	C16—H16B	0.9600
C4—H4	0.9300	C16—H16C	0.9600
C2—O1—H1	109.5	C14—C9—C10	118.5 (4)
C12—O3—C15	118.7 (5)	C14—C9—C8	118.6 (4)
H15—O4—H16	104.1	C10—C9—C8	122.9 (4)
C7—N1—N2	123.2 (4)	C9—C10—C11	120.7 (5)
C8—N2—N1	115.9 (4)	C9—C10—H10	119.7
C8—N2—H2	122.0	C11—C10—H10	119.7
N1—N2—H2	122.0	C12—C11—C10	119.3 (5)
C6—C1—C2	118.5 (4)	C12—C11—H11	120.4
C6—C1—C7	120.8 (4)	C10—C11—H11	120.4
C2—C1—C7	120.7 (4)	O3—C12—C13	115.8 (5)
O1—C2—C3	118.2 (4)	O3—C12—C11	123.4 (5)
O1—C2—C1	123.3 (4)	C13—C12—C11	120.8 (5)
C3—C2—C1	118.5 (4)	C12—C13—C14	119.6 (5)
C4—C3—C2	122.3 (4)	C12—C13—H13	120.2
C4—C3—Cl1	119.1 (4)	C14—C13—H13	120.2
C2—C3—Cl1	118.6 (4)	C9—C14—C13	121.2 (5)
C3—C4—C5	118.5 (4)	C9—C14—H14	119.4
C3—C4—H4	120.8	C13—C14—H14	119.4
C5—C4—H4	120.8	O3—C15—H15A	109.5
C6—C5—C4	121.1 (4)	O3—C15—H15B	109.5
C6—C5—Cl2	119.6 (4)	H15A—C15—H15B	109.5
C4—C5—Cl2	119.3 (4)	O3—C15—H15C	109.5
C5—C6—C1	121.1 (4)	H15A—C15—H15C	109.5
C5—C6—H6	119.5	H15B—C15—H15C	109.5
C1—C6—H6	119.5	C7—C16—H16A	109.5
N1—C7—C1	114.5 (4)	C7—C16—H16B	109.5
N1—C7—C16	125.9 (4)	H16A—C16—H16B	109.5
C1—C7—C16	119.6 (4)	C7—C16—H16C	109.5
O2—C8—N2	119.6 (4)	H16A—C16—H16C	109.5
O2—C8—C9	122.8 (4)	H16B—C16—H16C	109.5
N2—C8—C9	117.6 (4)		
C7—N1—N2—C8	173.8 (4)	C6—C1—C7—C16	−2.6 (6)
C6—C1—C2—O1	−177.2 (4)	C2—C1—C7—C16	176.7 (4)
C7—C1—C2—O1	3.5 (6)	N1—N2—C8—O2	0.7 (6)
C6—C1—C2—C3	1.7 (6)	N1—N2—C8—C9	−179.0 (4)
C7—C1—C2—C3	−177.5 (4)	O2—C8—C9—C14	14.7 (7)
O1—C2—C3—C4	177.9 (4)	N2—C8—C9—C14	−165.6 (4)
C1—C2—C3—C4	−1.1 (7)	O2—C8—C9—C10	−164.4 (5)
O1—C2—C3—Cl1	−1.4 (6)	N2—C8—C9—C10	15.3 (6)
C1—C2—C3—Cl1	179.7 (3)	C14—C9—C10—C11	2.0 (7)
C2—C3—C4—C5	−0.7 (7)	C8—C9—C10—C11	−178.9 (4)
Cl1—C3—C4—C5	178.5 (4)	C9—C10—C11—C12	−0.7 (7)
C3—C4—C5—C6	1.9 (7)	C15—O3—C12—C13	176.6 (5)
C3—C4—C5—Cl2	−178.9 (4)	C15—O3—C12—C11	−2.9 (8)

C4—C5—C6—C1	−1.3 (7)	C10—C11—C12—O3	178.5 (5)
Cl2—C5—C6—C1	179.6 (4)	C10—C11—C12—C13	−1.0 (8)
C2—C1—C6—C5	−0.6 (7)	O3—C12—C13—C14	−178.2 (4)
C7—C1—C6—C5	178.7 (4)	C11—C12—C13—C14	1.3 (8)
N2—N1—C7—C1	179.8 (4)	C10—C9—C14—C13	−1.7 (7)
N2—N1—C7—C16	−0.8 (7)	C8—C9—C14—C13	179.2 (4)
C6—C1—C7—N1	176.9 (4)	C12—C13—C14—C9	0.1 (7)
C2—C1—C7—N1	−3.9 (6)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O4	0.86	2.15	2.926 (5)	150
O1—H1···O2	0.82	2.58	3.287 (5)	146
O1—H1···N1	0.82	1.77	2.484 (5)	145
O4—H16···O1^{i}	0.85	2.09	2.887 (5)	156
O4—H15···O2^{ii}	0.85	1.88	2.726 (5)	176

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

Fig. 1

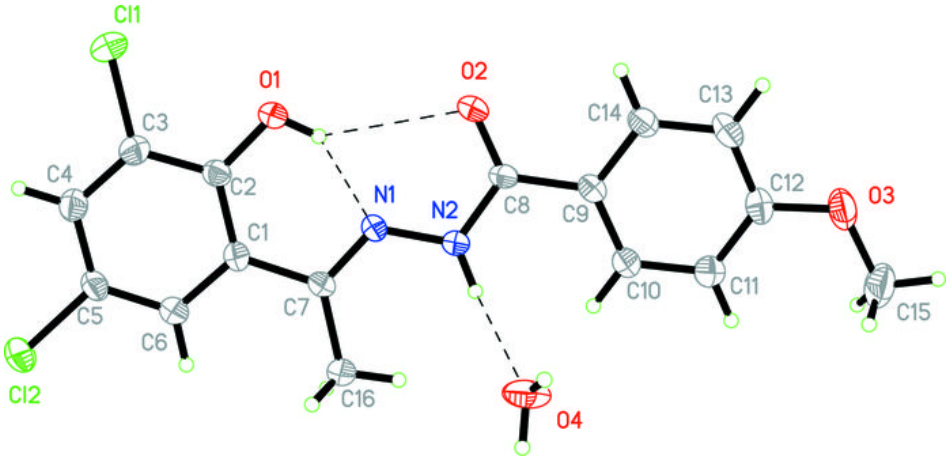
Cl1
O1
O2
C3
C4
C2
N1
N2
C8
C9
C14
C13
C12
O3
C1
C7
C10
C11
C15
C5
C6
Cl2
C16
O4

Fig. 2