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N′-[(1*E*)-(5-Chloro-2-hydroxyphenyl)(phenyl)methylene]-4-methoxybenzohydrazide

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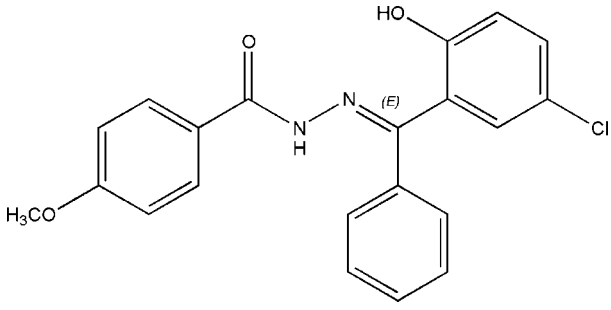
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.150; data-to-parameter ratio = 13.2.

The title compound, $C_{21}H_{17}ClN_2O_3$, displays a *trans* configuration with respect to the C=N double bond. The crystal structure is stabilized by intramolecular O—H···N and intermolecular C—H···O hydrogen bonds.

Related literature

For further details of the chemistry of the title compound, see: Carcelli *et al.* (1995); Salem (1998); Singh *et al.* (1982).



Experimental

Crystal data

$C_{21}H_{17}ClN_2O_3$
M_r = 380.82
Monoclinic, $P2_1/n$
a = 9.2861 (19) Å
b = 13.026 (3) Å
c = 15.722 (3) Å
β = 102.089 (8)°
V = 1859.6 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 273 (2) K
0.33 × 0.24 × 0.13 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.945, T_{max} = 0.966
16781 measured reflections
3259 independent reflections
2262 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.150
S = 1.00
3259 reflections
247 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.87	2.589 (2)	146
C11—H11···O2^{i}	0.93	2.60	3.287 (3)	130

Symmetry code: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); 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: PK2040).

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supplementary materials

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N'-[(1*E*)-(5-Chloro-2-hydroxyphenyl)(phenyl)methylene]-4-methoxybenzohydrazide

J.-G. Chang and R.-D. Yang

Comment

The chemistry of aroylhydrazones continues to attract much attention due to their coordination ability to metal ions (Singh *et al.*, 1982; Salem, 1998) and their biological activity (Singh *et al.*, 1982; Carcelli *et al.*, 1995). 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 three benzene rings, C1—C6 (A), C8—C13 (B) and C15—C20 (C), make dihedral angles of 74.21 (7) ° (A/B), 79.58 (7) ° (B/C) and 16.85 (12) ° (A/C). The crystal structure is stabilized by intramolecular N—H···O and intermolecular C—H···O hydrogen bonds (Table 1. and Fig. 2).

Experimental

4-methoxybenzohydrazide (0.01 mol,1.66 g) was dissolved in anhydrous ethanol (50 ml), and (5-chloro-2-hydroxyphenyl)(phenyl)methanone (0.01 mol, 2.32 g) was added. The reaction mixture was refluxed for 6 h with stirring, then the resulting precipitate was collected by filtration, washed several times with ethanol and dried *in vacuo* (yield 81%). The compound (1.0 mmol,0.38 g) was dissolved in dimethylformamide (20 ml) and kept at room temperature for 45 d 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_{Me}—H = 0.96 Å, C_{Ar}—H = 0.93 Å, O—H = 0.82 Å, and N—H = 0.86 Å and with U_{iso}(H) = 1.5U_{eq}(C_{Me}, O) and 1.2U_{eq}(C_{Ar}, N).

Figures

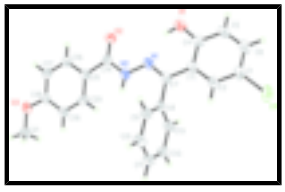

Fig. 1. The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

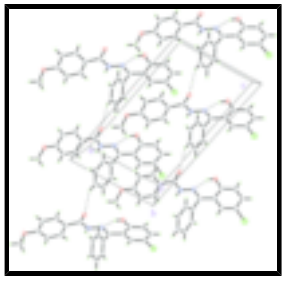

Fig. 2. Crystal packing of the title compound, viewed roughly along the *b* axis. Dashed lines show intra-and intermolecular hydrogen bonds.

N'-[(1E)-(5-chloro-2-hydroxyphenyl)(phenyl)methylene]-4- methoxybenzohydrazide

Crystal data

$C_{21}H_{17}ClN_2O_3$	F_{000} = 792
M_r = 380.82	D_x = 1.360 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 3423 reflections
a = 9.2861 (19) Å	θ = 2.4–21.3º
b = 13.026 (3) Å	μ = 0.23 mm^{-1}
c = 15.722 (3) Å	T = 273 (2) K
β = 102.089 (8)º	Plate, yellow
V = 1859.6 (7) Å^3	0.33 × 0.24 × 0.13 mm
Z = 4	

Data collection

Bruker APEX II CCD area-detector diffractometer	3259 independent reflections
Radiation source: fine-focus sealed tube	2262 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.045
T = 273(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.1º
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	h = −10→11
T_{min} = 0.945, T_{max} = 0.966	k = −15→15
16781 measured reflections	l = −18→18

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.043	$w = 1/[\sigma^2(F_o^2) + (0.0962P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.150	$(\Delta/\sigma)_{max} < 0.001$
S = 1.00	$\Delta\rho_{max}$ = 0.20 e Å^{-3}

3259 reflections	$\Delta\rho_{min} = -0.26$ e Å^{-3}
247 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.021 (3)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\sigma(F^2)$ 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	0.13352 (7)	0.88139 (5)	0.18144 (5)	0.0801 (3)
O1	0.6354 (2)	0.60874 (13)	0.30330 (11)	0.0705 (5)
H1	0.6975	0.6134	0.2733	0.106*
O2	0.99011 (18)	0.55710 (13)	0.21902 (10)	0.0674 (5)
O3	1.35103 (16)	0.55585 (13)	−0.08113 (10)	0.0647 (5)
N1	0.74992 (19)	0.67796 (14)	0.17801 (11)	0.0530 (5)
N2	0.85754 (19)	0.67454 (14)	0.13064 (12)	0.0566 (5)
H21	0.8526	0.7146	0.0866	0.068*
C1	0.2845 (2)	0.80152 (17)	0.21698 (15)	0.0570 (6)
C2	0.3986 (2)	0.80150 (16)	0.17394 (14)	0.0533 (5)
H2	0.3948	0.8449	0.1266	0.064*
C3	0.5204 (2)	0.73755 (15)	0.19997 (13)	0.0471 (5)
C4	0.5222 (3)	0.67327 (16)	0.27222 (14)	0.0538 (5)
C5	0.4053 (3)	0.67538 (19)	0.31437 (15)	0.0643 (7)
H5	0.4074	0.6327	0.3620	0.077*
C6	0.2874 (3)	0.7384 (2)	0.28777 (15)	0.0664 (7)
H6	0.2100	0.7388	0.3169	0.080*
C7	0.6397 (2)	0.73889 (16)	0.15167 (13)	0.0473 (5)
C8	0.6309 (2)	0.80770 (15)	0.07446 (13)	0.0458 (5)
C9	0.7175 (2)	0.89464 (16)	0.07990 (14)	0.0557 (6)
H9	0.7789	0.9121	0.1325	0.067*
C10	0.7133 (3)	0.95584 (18)	0.00764 (15)	0.0643 (6)
H10	0.7722	1.0141	0.0119	0.077*
C11	0.6227 (3)	0.93109 (18)	−0.07028 (15)	0.0612 (6)
H11	0.6207	0.9719	−0.1190	0.073*
C12	0.5354 (3)	0.8458 (2)	−0.07564 (15)	0.0643 (6)
H12	0.4728	0.8294	−0.1282	0.077*

C13	0.5392 (2)	0.78388 (18)	−0.00424 (13)	0.0585 (6)
H13	0.4798	0.7258	−0.0090	0.070*
C14	0.9726 (2)	0.60810 (16)	0.15275 (13)	0.0492 (5)
C15	1.0706 (2)	0.60199 (15)	0.08952 (13)	0.0472 (5)
C16	1.1987 (2)	0.54446 (18)	0.11171 (15)	0.0592 (6)
H16	1.2230	0.5141	0.1664	0.071*
C17	1.2902 (2)	0.53153 (19)	0.05433 (15)	0.0626 (6)
H17	1.3764	0.4936	0.0707	0.075*
C18	1.2549 (2)	0.57464 (17)	−0.02765 (13)	0.0504 (5)
C19	1.1298 (2)	0.63223 (18)	−0.05077 (14)	0.0603 (6)
H19	1.1059	0.6625	−0.1055	0.072*
C20	1.0394 (3)	0.64517 (19)	0.00765 (15)	0.0605 (6)
H20	0.9544	0.6844	−0.0087	0.073*
C21	1.3176 (3)	0.5988 (2)	−0.16640 (15)	0.0705 (7)
H21A	1.2245	0.5729	−0.1973	0.106*
H21B	1.3931	0.5803	−0.1969	0.106*
H21C	1.3126	0.6722	−0.1624	0.106*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0543 (4)	0.0815 (5)	0.1113 (6)	0.0072 (3)	0.0332 (4)	−0.0043 (4)
O1	0.0758 (12)	0.0759 (12)	0.0649 (11)	0.0089 (9)	0.0260 (9)	0.0189 (8)
O2	0.0651 (10)	0.0873 (12)	0.0523 (9)	0.0141 (9)	0.0184 (7)	0.0135 (8)
O3	0.0530 (9)	0.0840 (12)	0.0633 (10)	0.0063 (8)	0.0263 (8)	−0.0002 (8)
N1	0.0519 (10)	0.0576 (11)	0.0548 (10)	0.0021 (9)	0.0232 (8)	−0.0006 (8)
N2	0.0537 (11)	0.0623 (12)	0.0605 (11)	0.0105 (9)	0.0269 (9)	0.0088 (9)
C1	0.0490 (13)	0.0548 (13)	0.0726 (15)	−0.0083 (10)	0.0250 (11)	−0.0100 (11)
C2	0.0540 (13)	0.0506 (13)	0.0604 (13)	−0.0049 (10)	0.0240 (10)	−0.0018 (10)
C3	0.0490 (12)	0.0449 (11)	0.0510 (11)	−0.0057 (9)	0.0186 (9)	−0.0040 (9)
C4	0.0599 (13)	0.0514 (13)	0.0534 (12)	−0.0057 (11)	0.0194 (10)	−0.0013 (10)
C5	0.0748 (16)	0.0673 (16)	0.0582 (14)	−0.0141 (13)	0.0310 (12)	0.0019 (11)
C6	0.0611 (15)	0.0736 (16)	0.0737 (16)	−0.0130 (13)	0.0355 (12)	−0.0107 (13)
C7	0.0471 (12)	0.0481 (12)	0.0497 (11)	−0.0017 (10)	0.0166 (9)	−0.0045 (9)
C8	0.0418 (11)	0.0497 (12)	0.0508 (12)	0.0022 (9)	0.0212 (9)	−0.0018 (9)
C9	0.0558 (13)	0.0575 (14)	0.0541 (13)	−0.0076 (11)	0.0125 (10)	−0.0019 (10)
C10	0.0713 (16)	0.0550 (14)	0.0705 (16)	−0.0094 (12)	0.0237 (12)	0.0038 (11)
C11	0.0741 (16)	0.0592 (14)	0.0554 (13)	0.0080 (13)	0.0252 (12)	0.0065 (11)
C12	0.0650 (15)	0.0789 (16)	0.0495 (13)	−0.0035 (13)	0.0131 (11)	−0.0033 (11)
C13	0.0601 (14)	0.0641 (14)	0.0528 (13)	−0.0151 (12)	0.0155 (11)	−0.0056 (11)
C14	0.0452 (12)	0.0560 (13)	0.0465 (12)	−0.0004 (10)	0.0094 (9)	−0.0022 (9)
C15	0.0423 (11)	0.0502 (12)	0.0497 (12)	−0.0001 (9)	0.0107 (9)	−0.0020 (9)
C16	0.0525 (13)	0.0745 (15)	0.0511 (12)	0.0092 (12)	0.0122 (10)	0.0092 (11)
C17	0.0461 (12)	0.0785 (16)	0.0637 (14)	0.0166 (12)	0.0123 (10)	0.0070 (12)
C18	0.0432 (12)	0.0554 (13)	0.0547 (13)	−0.0034 (10)	0.0153 (10)	−0.0045 (10)
C19	0.0583 (14)	0.0733 (15)	0.0533 (13)	0.0133 (12)	0.0205 (11)	0.0111 (11)
C20	0.0563 (13)	0.0699 (15)	0.0580 (13)	0.0215 (12)	0.0180 (11)	0.0119 (11)
C21	0.0718 (16)	0.0882 (18)	0.0580 (15)	−0.0049 (14)	0.0285 (12)	−0.0033 (12)

Geometric parameters (Å, °)

Cl1—C1	1.741 (2)	C9—C10	1.382 (3)
O1—C4	1.355 (3)	C9—H9	0.9300
O1—H1	0.8200	C10—C11	1.371 (3)
O2—C14	1.217 (2)	C10—H10	0.9300
O3—C18	1.371 (3)	C11—C12	1.368 (3)
O3—C21	1.426 (3)	C11—H11	0.9300
N1—C7	1.293 (3)	C12—C13	1.377 (3)
N1—N2	1.367 (2)	C12—H12	0.9300
N2—C14	1.362 (3)	C13—H13	0.9300
N2—H21	0.8600	C14—C15	1.484 (3)
C1—C2	1.372 (3)	C15—C20	1.379 (3)
C1—C6	1.379 (3)	C15—C16	1.387 (3)
C2—C3	1.395 (3)	C16—C17	1.373 (3)
C2—H2	0.9300	C16—H16	0.9300
C3—C4	1.409 (3)	C17—C18	1.381 (3)
C3—C7	1.468 (3)	C17—H17	0.9300
C4—C5	1.385 (3)	C18—C19	1.366 (3)
C5—C6	1.362 (3)	C19—C20	1.378 (3)
C5—H5	0.9300	C19—H19	0.9300
C6—H6	0.9300	C20—H20	0.9300
C7—C8	1.497 (3)	C21—H21A	0.9600
C8—C9	1.381 (3)	C21—H21B	0.9600
C8—C13	1.382 (3)	C21—H21C	0.9600
C4—O1—H1	109.5	C12—C11—C10	119.4 (2)
C18—O3—C21	117.72 (18)	C12—C11—H11	120.3
C7—N1—N2	117.75 (17)	C10—C11—H11	120.3
C14—N2—N1	120.38 (17)	C11—C12—C13	120.8 (2)
C14—N2—H21	119.8	C11—C12—H12	119.6
N1—N2—H21	119.8	C13—C12—H12	119.6
C2—C1—C6	120.7 (2)	C12—C13—C8	120.3 (2)
C2—C1—Cl1	119.68 (19)	C12—C13—H13	119.9
C6—C1—Cl1	119.60 (17)	C8—C13—H13	119.9
C1—C2—C3	121.2 (2)	O2—C14—N2	121.46 (19)
C1—C2—H2	119.4	O2—C14—C15	123.61 (19)
C3—C2—H2	119.4	N2—C14—C15	114.92 (18)
C2—C3—C4	117.59 (19)	C20—C15—C16	117.21 (19)
C2—C3—C7	119.61 (18)	C20—C15—C14	124.38 (19)
C4—C3—C7	122.79 (19)	C16—C15—C14	118.31 (18)
O1—C4—C5	117.8 (2)	C17—C16—C15	121.1 (2)
O1—C4—C3	122.5 (2)	C17—C16—H16	119.4
C5—C4—C3	119.7 (2)	C15—C16—H16	119.4
C6—C5—C4	121.7 (2)	C16—C17—C18	120.3 (2)
C6—C5—H5	119.2	C16—C17—H17	119.8
C4—C5—H5	119.2	C18—C17—H17	119.8
C5—C6—C1	119.1 (2)	C19—C18—O3	124.16 (19)
C5—C6—H6	120.5	C19—C18—C17	119.6 (2)

C1—C6—H6	120.5	O3—C18—C17	116.23 (19)
N1—C7—C3	117.34 (18)	C18—C19—C20	119.5 (2)
N1—C7—C8	122.16 (18)	C18—C19—H19	120.2
C3—C7—C8	120.49 (18)	C20—C19—H19	120.2
C9—C8—C13	118.7 (2)	C19—C20—C15	122.2 (2)
C9—C8—C7	120.57 (18)	C19—C20—H20	118.9
C13—C8—C7	120.66 (18)	C15—C20—H20	118.9
C8—C9—C10	120.4 (2)	O3—C21—H21A	109.5
C8—C9—H9	119.8	O3—C21—H21B	109.5
C10—C9—H9	119.8	H21A—C21—H21B	109.5
C11—C10—C9	120.3 (2)	O3—C21—H21C	109.5
C11—C10—H10	119.8	H21A—C21—H21C	109.5
C9—C10—H10	119.8	H21B—C21—H21C	109.5
C7—N1—N2—C14	−176.75 (18)	C7—C8—C9—C10	177.5 (2)
C6—C1—C2—C3	0.4 (3)	C8—C9—C10—C11	0.2 (3)
Cl1—C1—C2—C3	−179.18 (15)	C9—C10—C11—C12	0.6 (4)
C1—C2—C3—C4	−0.2 (3)	C10—C11—C12—C13	−1.0 (4)
C1—C2—C3—C7	179.33 (19)	C11—C12—C13—C8	0.5 (4)
C2—C3—C4—O1	−179.54 (18)	C9—C8—C13—C12	0.3 (3)
C7—C3—C4—O1	0.9 (3)	C7—C8—C13—C12	−177.8 (2)
C2—C3—C4—C5	0.0 (3)	N1—N2—C14—O2	−6.7 (3)
C7—C3—C4—C5	−179.49 (19)	N1—N2—C14—C15	172.13 (17)
O1—C4—C5—C6	179.6 (2)	O2—C14—C15—C20	168.4 (2)
C3—C4—C5—C6	0.0 (3)	N2—C14—C15—C20	−10.5 (3)
C4—C5—C6—C1	0.1 (4)	O2—C14—C15—C16	−7.9 (3)
C2—C1—C6—C5	−0.3 (3)	N2—C14—C15—C16	173.28 (18)
Cl1—C1—C6—C5	179.25 (17)	C20—C15—C16—C17	0.1 (3)
N2—N1—C7—C3	176.48 (17)	C14—C15—C16—C17	176.7 (2)
N2—N1—C7—C8	−2.7 (3)	C15—C16—C17—C18	−1.0 (4)
C2—C3—C7—N1	−179.70 (18)	C21—O3—C18—C19	−0.3 (3)
C4—C3—C7—N1	−0.2 (3)	C21—O3—C18—C17	179.7 (2)
C2—C3—C7—C8	−0.5 (3)	C16—C17—C18—C19	1.4 (4)
C4—C3—C7—C8	179.07 (18)	C16—C17—C18—O3	−178.6 (2)
N1—C7—C8—C9	−74.2 (3)	O3—C18—C19—C20	179.1 (2)
C3—C7—C8—C9	106.6 (2)	C17—C18—C19—C20	−1.0 (3)
N1—C7—C8—C13	103.9 (2)	C18—C19—C20—C15	0.1 (4)
C3—C7—C8—C13	−75.3 (3)	C16—C15—C20—C19	0.3 (4)
C13—C8—C9—C10	−0.7 (3)	C14—C15—C20—C19	−176.0 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.87	2.589 (2)	146
C11—H11···O2^{i}	0.93	2.60	3.287 (3)	130

Symmetry codes: (i) *x*−1/2, −*y*+3/2, *z*−1/2.

Fig. 1

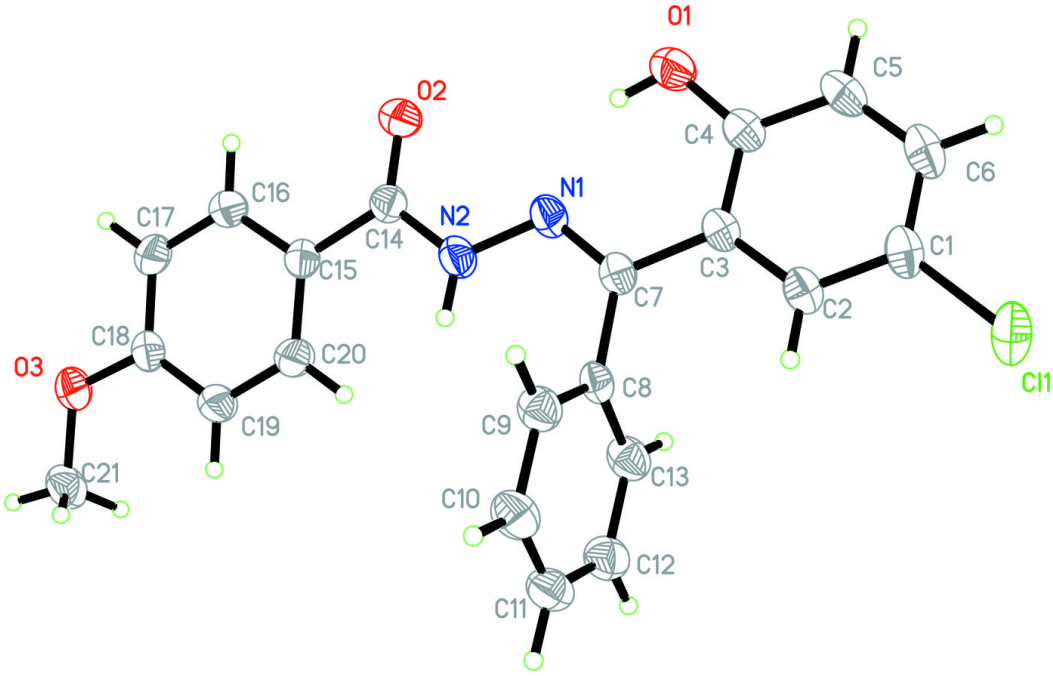
O1
O2
C5
C4
C6
C16
N1
C17
N2
C14
C1
C15
C3
C7
C2
C18
Cl1
O3
C20
C8
C9
C19
C13
C21
C10
C12
C11

Fig. 2