Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# *N*′-(5-Chloro-2-hydroxybenzylidene)-4-hydroxybenzohydrazide

**Xiao-Yang Qiu**

Department of Chemistry, Shangqiu Normal University, Shangqiu 476000, People's Republic of China
Correspondence e-mail: xiaoyang_qiu@126.com

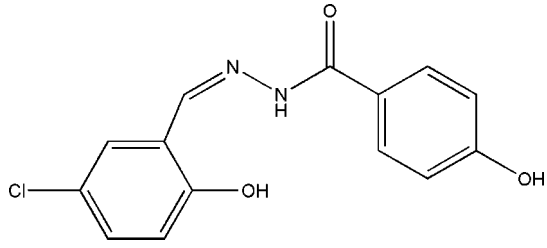
Received 30 March 2009; accepted 1 April 2009

Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.026; $wR$ factor = 0.071; data-to-parameter ratio = 11.9.

The title Schiff base compound, $C_{14}H_{11}ClN_2O_3$, was prepared by the reaction of 5-chlorosalicylaldehyde and 4-hydroxybenzohydrazide. The molecule exists in a *trans* configuration with respect to the methylidene group. The dihedral angle between the two benzene rings is 40.1 (2)°. An intramolecular O—H···N hydrogen bond helps to stabilize the molecular conformation. In the crystal structure, molecules are linked into a three-dimensional network by intermolecular N—H···O and O—H···O hydrogen bonds.

## Related literature

For the biological properties of hydrazone compounds, see: Bedia *et al.* (2006); Rollas *et al.* (2002); Fun *et al.* (2008). For the structures of hydrazone compounds we have reported previously, see: Qiu, Fang *et al.* (2006); Qiu, Luo *et al.*, (2006*a*,*b*); Qiu, Xu *et al.* (2006). For bond-length data, see: Allen *et al.* (1987). For related structures see: Singh *et al.* (2007); Narayana *et al.* (2007); Cui *et al.* (2007); Diao *et al.* (2008).




## Experimental

### *Crystal data*

$C_{14}H_{11}ClN_2O_3$
$M_r$ = 290.70
Orthorhombic, $Pna2_1$
$a$ = 9.423 (1) Å
$b$ = 9.839 (1) Å
$c$ = 13.770 (1) Å
$V$ = 1276.7 (2) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.31 mm$^{-1}$
$T$ = 298 K
0.17 × 0.15 × 0.15 mm

### *Data collection*

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.950, $T_{max}$ = 0.955
7398 measured reflections
2231 independent reflections
2144 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.022

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.071
$S$ = 1.06
2231 reflections
187 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.32 e Å$^{-3}$
Absolute structure: Flack (1983), 784 Friedel pairs
Flack parameter: −0.01 (6)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2···O2$^{i}$ | 0.892 (10) | 2.121 (11) | 3.0065 (18) | 172 (3) |
| O3—H3···O2$^{ii}$ | 0.82 | 1.98 | 2.7479 (19) | 157 |
| O1—H1···N1 | 0.82 | 1.89 | 2.6057 (19) | 145 |

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); 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*.

The author acknowledges the Natural Science Foundation of the Education Office of Anhui Province (Project No. 2009 A150020).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2605).

## References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bedia, K.-K., Elcin, O., Seda, U., Fatma, K., Nathaly, S., Sevim, R. & Dimoglo, A. (2006). *Eur. J. Med. Chem.* **41**, 1253-1261.
Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cui, J., Yin, H. & Qiao, Y. (2007). *Acta Cryst.* E**63**, o3548.
Diao, Y.-P., Zhen, Y.-H., Han, X. & Deng, S. (2008). *Acta Cryst.* E**64**, o101.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Fun, H.-K., Patil, P. S., Jebas, S. R., Sujith, K. V. & Kalluraya, B. (2008). *Acta Cryst.* E**64**, o1594–o1595.
Narayana, B., Siddaraju, B. P., Raju, C. R., Yathirajan, H. S. & Bolte, M. (2007). *Acta Cryst.* E**63**, o3522.
Qiu, X.-Y., Fang, X.-N., Liu, W.-S. & Zhu, H.-L. (2006). *Acta Cryst.* E**62**, o2685–o2686.
Qiu, X.-Y., Luo, Q.-Y., Yang, S.-L. & Liu, W.-S. (2006*a*). *Acta Cryst.* E**62**, o4291–o4292.
Qiu, X.-Y., Luo, Z.-G., Yang, S.-L. & Liu, W.-S. (2006*b*). *Acta Cryst.* E**62**, o3531–o3532.
Qiu, X.-Y., Xu, H.-J., Liu, W.-S. & Zhu, H.-L. (2006). *Acta Cryst.* E**62**, o2304–o2305.
Rollas, S., Gülerman, N. & Erdeniz, H. (2002). *Farmaco*, **57**, 171–174.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Singh, N. K., Singh, M., Srivastava, A. K., Shrivastav, A. & Sharma, R. K. (2007). *Acta Cryst.* E**63**, o4895.

# supplementary materials

*Acta Cryst.* (2009). E**65**, o975 [ **doi:10.1107/S160053680901215X** ]

# *N*'-(5-Chloro-2-hydroxybenzylidene)-4-hydroxybenzohydrazide

**X.-Y. Qiu**

## Comment

Hyrazone compounds, derived from the reaction of aldehydes with hydrazides, have been widely studied due to their excellent biological properties (Bedia *et al.*, 2006; Rollas *et al.*, 2002; Fun *et al.*, 2008). Recently, we have reported several Schiff base hydrazone compounds (Qiu, Fang *et al.*, 2006; Qiu, Luo *et al.*, 2006*a*,b; Qiu, Xu *et al.*, 2006), and we report herein the crystal structure of the new title compound, (I), Fig. 1.

The molecule in (I) exists in a *trans* configuration with respect to the methylidene group. The dihedral angle between the two benzene rings is 40.1 (2)°. The bond lengths in (I) are found to have normal values (Allen *et al.*, 1987) and are comparable to the values found in similar compounds (Singh *et al.*, 2007; Narayana *et al.*, 2007; Cui *et al.*, 2007; Diao *et al.*, 2008).

An intramolecular O–H···N hydrogen bond (Table 1) helps to stabilize the molecular conformation. In the crystal structure, molecules are linked into a three-dimensional network by intermolecular N–H···O and O–H···O hydrogen bonds (Table 1 and Fig. 2).

## Experimental

The title compound was prepared by the Schiff base condensation of equimolar amounts (0.5 mmol each) of 5-chlorosalicylaldehyde and 4-hydroxybenzohydrazide in methanol (20 ml). Excess methanol was removed from the reaction mixture by distillation. The colourless solid was filtered and dried in air. Colourless block-shaped crystals suitable for X-ray diffraction were obtained from a methanol solution.

## Refinement

The imino H atoms were located in a difference map and refined with N–H distances restrained to 0.90 (1) Å. The remaining H atoms were positioned geometrically [C–H = 0.93 Å, O–H = 0.82 Å] and refined using a riding model, with $U_{iso}$(H) = 1.2$U_{eq}$(C) and 1.5$U_{eq}$(O).

## Figures

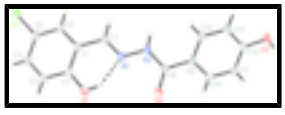


Fig. 1. The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms.

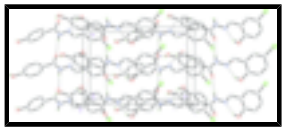


Fig. 2. The crystal packing of (I), viewed along the *b* axis with hydrogen bonds drawn as dashed lines.

## *N*′-(5-Chloro-2-hydroxybenzylidene)-4-hydroxybenzohydrazide

*Crystal data*

| | |
|---|---|
| $C_{14}H_{11}ClN_2O_3$ | $F_{000} = 600$ |
| $M_r = 290.70$ | $D_x = 1.513$ Mg m$^{-3}$ |
| Orthorhombic, *Pna*2$_1$ | Mo *K*α radiation<br>λ = 0.71073 Å |
| Hall symbol: P 2c -2n | Cell parameters from 4763 reflections |
| *a* = 9.423 (1) Å | θ = 2.5–30.6º |
| *b* = 9.839 (1) Å | μ = 0.31 mm$^{-1}$ |
| *c* = 13.770 (1) Å | *T* = 298 K |
| *V* = 1276.7 (2) Å$^3$ | Block, colourless |
| *Z* = 4 | 0.17 × 0.15 × 0.15 mm |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD diffractometer | 2231 independent reflections |
| Radiation source: fine-focus sealed tube | 2144 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.022$ |
| *T* = 298 K | $\theta_{max} = 27.0$º |
| ω scans | $\theta_{min} = 2.5$º |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | *h* = −12→11 |
| $T_{min} = 0.950$, $T_{max} = 0.955$ | *k* = −12→12 |
| 7398 measured reflections | *l* = −17→11 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H atoms treated by a mixture of independent and constrained refinement |
| $R[F^2 > 2\sigma(F^2)] = 0.026$ | $w = 1/[\sigma^2(F_o^2) + (0.0409P)^2 + 0.1827P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.071$ | $(\Delta/\sigma)_{max} = 0.001$ |
| *S* = 1.06 | $\Delta\rho_{max} = 0.21$ e Å$^{-3}$ |
| 2231 reflections | $\Delta\rho_{min} = -0.32$ e Å$^{-3}$ |
| 187 parameters | Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| 2 restraints | Extinction coefficient: 0.034 (3) |
| Primary atom site location: structure-invariant direct methods | Absolute structure: Flack (1983), 784 Friedel pairs |
| Secondary atom site location: difference Fourier map | Flack parameter: −0.01 (6) |

*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 | 0.74914 (6) | 0.47741 (5) | 1.21657 (5) | 0.05224 (15) |
| N1 | 0.99352 (15) | 0.30628 (14) | 0.80967 (10) | 0.0316 (3) |
| N2 | 0.94949 (14) | 0.28112 (13) | 0.71621 (12) | 0.0319 (3) |
| O1 | 1.19292 (14) | 0.32175 (17) | 0.94031 (11) | 0.0504 (4) |
| H1 | 1.1598 | 0.3057 | 0.8865 | 0.076* |
| O2 | 1.14143 (12) | 0.14658 (12) | 0.69247 (10) | 0.0370 (3) |
| O3 | 0.84616 (17) | 0.10538 (15) | 0.27816 (10) | 0.0505 (4) |
| H3 | 0.8733 | 0.0312 | 0.2583 | 0.076* |
| C1 | 0.94953 (17) | 0.38640 (15) | 0.96863 (13) | 0.0312 (3) |
| C2 | 1.08639 (18) | 0.35624 (17) | 1.00215 (14) | 0.0346 (4) |
| C3 | 1.1165 (2) | 0.36019 (19) | 1.10078 (15) | 0.0403 (4) |
| H3A | 1.2072 | 0.3386 | 1.1225 | 0.048* |
| C4 | 1.0133 (2) | 0.39569 (17) | 1.16663 (14) | 0.0391 (4) |
| H4 | 1.0332 | 0.3960 | 1.2328 | 0.047* |
| C5 | 0.87956 (19) | 0.43088 (17) | 1.13363 (14) | 0.0362 (4) |
| C6 | 0.84744 (18) | 0.42738 (18) | 1.03619 (14) | 0.0352 (4) |
| H6 | 0.7573 | 0.4524 | 1.0152 | 0.042* |
| C7 | 0.90835 (18) | 0.36642 (16) | 0.86788 (13) | 0.0325 (3) |
| H7 | 0.8207 | 0.3972 | 0.8459 | 0.039* |
| C8 | 1.03211 (17) | 0.20101 (16) | 0.65983 (13) | 0.0292 (3) |
| C9 | 0.98366 (16) | 0.17900 (16) | 0.55911 (12) | 0.0296 (3) |
| C10 | 1.03520 (17) | 0.06631 (17) | 0.50845 (14) | 0.0335 (4) |
| H10 | 1.0998 | 0.0084 | 0.5384 | 0.040* |
| C11 | 0.99177 (18) | 0.03955 (17) | 0.41486 (14) | 0.0350 (4) |
| H11 | 1.0265 | −0.0361 | 0.3821 | 0.042* |
| C12 | 0.89604 (19) | 0.12597 (18) | 0.36973 (13) | 0.0352 (4) |
| C13 | 0.8457 (2) | 0.2394 (2) | 0.41833 (15) | 0.0450 (5) |
| H13 | 0.7826 | 0.2980 | 0.3876 | 0.054* |
| C14 | 0.8889 (2) | 0.26545 (18) | 0.51195 (14) | 0.0392 (4) |
| H14 | 0.8544 | 0.3417 | 0.5441 | 0.047* |
| H2 | 0.8598 (14) | 0.303 (3) | 0.703 (2) | 0.080* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0578 (3) | 0.0676 (3) | 0.0313 (2) | 0.0148 (2) | 0.0054 (2) | −0.0062 (3) |
| N1 | 0.0343 (7) | 0.0371 (7) | 0.0235 (8) | −0.0016 (5) | −0.0046 (6) | −0.0003 (6) |
| N2 | 0.0319 (7) | 0.0408 (7) | 0.0229 (7) | 0.0001 (5) | −0.0045 (7) | −0.0009 (6) |
| O1 | 0.0365 (7) | 0.0791 (10) | 0.0354 (8) | 0.0131 (7) | −0.0033 (6) | −0.0060 (7) |
| O2 | 0.0323 (6) | 0.0466 (6) | 0.0322 (7) | 0.0039 (5) | −0.0061 (5) | 0.0025 (5) |
| O3 | 0.0679 (10) | 0.0545 (8) | 0.0289 (8) | 0.0089 (6) | −0.0118 (7) | −0.0097 (6) |
| C1 | 0.0344 (8) | 0.0322 (8) | 0.0271 (9) | −0.0001 (6) | −0.0049 (7) | −0.0006 (6) |
| C2 | 0.0352 (8) | 0.0376 (8) | 0.0311 (9) | 0.0014 (6) | −0.0033 (8) | −0.0024 (7) |
| C3 | 0.0400 (10) | 0.0450 (9) | 0.0358 (11) | 0.0036 (7) | −0.0127 (8) | −0.0025 (8) |
| C4 | 0.0531 (11) | 0.0398 (8) | 0.0244 (9) | 0.0025 (7) | −0.0103 (8) | −0.0032 (7) |
| C5 | 0.0444 (9) | 0.0359 (8) | 0.0283 (9) | 0.0031 (7) | 0.0005 (7) | −0.0044 (7) |
| C6 | 0.0350 (8) | 0.0406 (8) | 0.0300 (9) | 0.0035 (7) | −0.0046 (7) | −0.0008 (7) |
| C7 | 0.0317 (8) | 0.0386 (8) | 0.0271 (9) | 0.0007 (6) | −0.0056 (7) | 0.0011 (7) |
| C8 | 0.0298 (8) | 0.0318 (7) | 0.0261 (9) | −0.0043 (6) | −0.0007 (6) | 0.0027 (6) |
| C9 | 0.0304 (7) | 0.0342 (7) | 0.0243 (9) | −0.0013 (6) | 0.0003 (6) | 0.0019 (6) |
| C10 | 0.0309 (8) | 0.0371 (8) | 0.0327 (10) | 0.0039 (6) | −0.0011 (7) | −0.0011 (7) |
| C11 | 0.0358 (9) | 0.0370 (8) | 0.0321 (10) | 0.0015 (6) | 0.0030 (7) | −0.0058 (7) |
| C12 | 0.0392 (9) | 0.0426 (9) | 0.0239 (9) | −0.0033 (7) | −0.0011 (7) | −0.0004 (7) |
| C13 | 0.0593 (12) | 0.0446 (9) | 0.0311 (10) | 0.0158 (8) | −0.0110 (9) | 0.0004 (8) |
| C14 | 0.0527 (10) | 0.0360 (8) | 0.0290 (9) | 0.0111 (7) | −0.0052 (8) | −0.0040 (7) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Cl1—C5 | 1.7391 (19) | C4—C5 | 1.384 (3) |
| N1—C7 | 1.279 (2) | C4—H4 | 0.9300 |
| N1—N2 | 1.375 (2) | C5—C6 | 1.376 (3) |
| N2—C8 | 1.353 (2) | C6—H6 | 0.9300 |
| N2—H2 | 0.892 (10) | C7—H7 | 0.9300 |
| O1—C2 | 1.359 (2) | C8—C9 | 1.476 (2) |
| O1—H1 | 0.8200 | C9—C14 | 1.394 (2) |
| O2—C8 | 1.245 (2) | C9—C10 | 1.397 (2) |
| O3—C12 | 1.361 (2) | C10—C11 | 1.378 (3) |
| O3—H3 | 0.8200 | C10—H10 | 0.9300 |
| C1—C6 | 1.398 (2) | C11—C12 | 1.387 (2) |
| C1—C2 | 1.402 (2) | C11—H11 | 0.9300 |
| C1—C7 | 1.454 (2) | C12—C13 | 1.385 (3) |
| C2—C3 | 1.388 (3) | C13—C14 | 1.376 (3) |
| C3—C4 | 1.374 (3) | C13—H13 | 0.9300 |
| C3—H3A | 0.9300 | C14—H14 | 0.9300 |
| C7—N1—N2 | 118.72 (14) | N1—C7—C1 | 119.54 (15) |
| C8—N2—N1 | 117.94 (13) | N1—C7—H7 | 120.2 |
| C8—N2—H2 | 125 (2) | C1—C7—H7 | 120.2 |
| N1—N2—H2 | 116 (2) | O2—C8—N2 | 121.31 (16) |
| C2—O1—H1 | 109.5 | O2—C8—C9 | 122.14 (15) |

| | | | |
|---|---|---|---|
| C12—O3—H3 | 109.5 | N2—C8—C9 | 116.53 (14) |
| C6—C1—C2 | 118.37 (16) | C14—C9—C10 | 118.32 (16) |
| C6—C1—C7 | 119.37 (15) | C14—C9—C8 | 123.12 (15) |
| C2—C1—C7 | 122.09 (16) | C10—C9—C8 | 118.56 (14) |
| O1—C2—C3 | 117.99 (16) | C11—C10—C9 | 121.03 (16) |
| O1—C2—C1 | 121.73 (17) | C11—C10—H10 | 119.5 |
| C3—C2—C1 | 120.28 (16) | C9—C10—H10 | 119.5 |
| C4—C3—C2 | 120.55 (16) | C10—C11—C12 | 119.69 (16) |
| C4—C3—H3A | 119.7 | C10—C11—H11 | 120.2 |
| C2—C3—H3A | 119.7 | C12—C11—H11 | 120.2 |
| C3—C4—C5 | 119.41 (17) | O3—C12—C13 | 116.71 (16) |
| C3—C4—H4 | 120.3 | O3—C12—C11 | 123.28 (16) |
| C5—C4—H4 | 120.3 | C13—C12—C11 | 120.01 (17) |
| C6—C5—C4 | 120.95 (18) | C14—C13—C12 | 120.13 (17) |
| C6—C5—Cl1 | 119.44 (14) | C14—C13—H13 | 119.9 |
| C4—C5—Cl1 | 119.59 (15) | C12—C13—H13 | 119.9 |
| C5—C6—C1 | 120.32 (16) | C13—C14—C9 | 120.80 (16) |
| C5—C6—H6 | 119.8 | C13—C14—H14 | 119.6 |
| C1—C6—H6 | 119.8 | C9—C14—H14 | 119.6 |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2···O2$^{i}$ | 0.892 (10) | 2.121 (11) | 3.0065 (18) | 172 (3) |
| O3—H3···O2$^{ii}$ | 0.82 | 1.98 | 2.7479 (19) | 157 |
| O1—H1···N1 | 0.82 | 1.89 | 2.6057 (19) | 145 |

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

**Fig. 1**

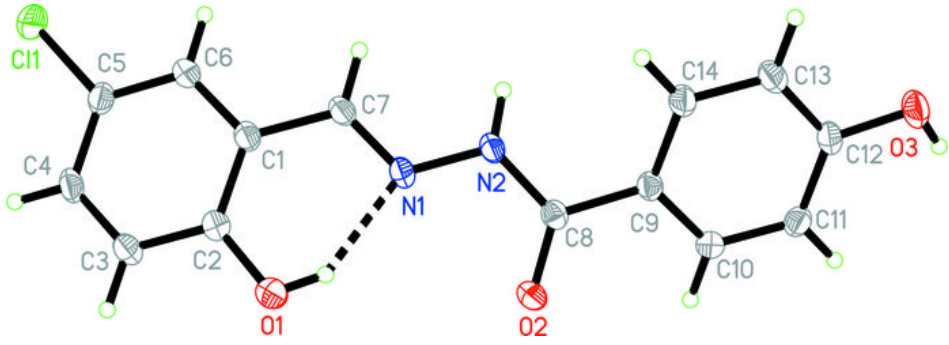


**Fig. 2**