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# (*Z*)-Ethyl 2-[2-(4-acetylphenyl)hydrazono]-4-chloro-3-oxobutanoate

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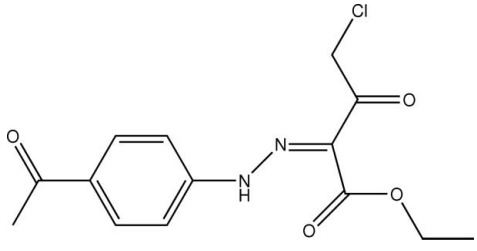
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.044; $wR$ factor = 0.130; data-to-parameter ratio = 15.0.

The title compound, $C_{14}H_{15}ClN_2O_4$, adopts a keto–hydrazo tautomeric form stabilized by an intramolecular N—H···O hydrogen bond. The configuration around the N—N bond is *trans*. The dihedral angle between the aromatic ring and the plane of the aliphatic chain attached to the hydrazone group is 10.5 (1)°. In the solid state, molecules exist as centrosymmetrically related C—H···O hydrogen-bonded $R_2^2(24)$ dimers.

## Related literature

For ring motif details, see: Bernstein *et al.* (1995). For related literature, see: Harada *et al.* (1997). For preparation details, see: Odabaşoğlu *et al.* (2005).




## Experimental

### *Crystal data*

$C_{14}H_{15}ClN_2O_4$
$M_r$ = 310.73
Monoclinic, $P2_1/c$
$a$ = 8.6018 (5) Å
$b$ = 18.9829 (16) Å
$c$ = 9.1035 (6) Å
$\beta$ = 93.591 (5)°
$V$ = 1483.57 (18) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.27 mm$^{-1}$
$T$ = 296 (2) K
0.80 × 0.53 × 0.35 mm

### *Data collection*

Stoe IPDSII diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min}$ = 0.854, $T_{max}$ = 0.914
15055 measured reflections
2916 independent reflections
2277 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.130
$S$ = 1.05
2916 reflections
195 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.31 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O3 | 0.82 (2) | 1.90 (2) | 2.573 (2) | 139 (2) |
| C11—H11$B$···O1$^{i}$ | 0.97 | 2.47 | 3.334 (3) | 149 |

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2369).

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# supplementary materials

*Acta Cryst.* (2007). E**63**, o2746 [ **doi:10.1107/S1600536807019617** ]

# (*Z*)-Ethyl 2-[2-(4-acetylphenyl)hydrazono]-4-chloro-3-oxobutanoate

**G. Alpaslan, Ö. Özdamar, M. Odabasoglu, N. Ocak İskeleli and A. Erdönmez**

## Comment

As part of our project to study the crystal structures of a series of phenylhydrazones and their stereochemistry, the crystal structure of the title compound, (I), has been determined.

The molecular structure and atom-labelling scheme of (I) are shown in Fig. 1. There is significant elongation of the N1—N2 bond (1.304 (2) Å) and contraction of the C1—N1 bond (1.402 (2) Å) in comparison with azo compounds. For example, the N═N and C—N distances in azobenzene are 1.249 (4) and 1.431 (4) Å, respectively (Harada *et al.*, 1997).

The molecule is approximately planar with the dihedral angle between the aromatic C1—C6 ring and the mean plane of the C9—C14/O2—O4/Cl1 aliphatic chain being 10.5 (1)°.

An intramolecular N—H···O hydrogen bond generates an S(6) ring motif (Bernstein *et al.*, 1995). In the crystal structure of (I), the molecules are linked into centrosymmetric $R_2^2(24)$ dimers by C—H···O intermolecular hydrogen bonds (Fig.2 and Table 1).

## Experimental

The title compound was prepared as described by Odabaşoğlu *et al.* (2005), using *m*-bromoaniline and ethyl 4-chloroaceto-acetate as starting materials (yield 91%, m.p. 415–417 K). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an absolute acetic acid solution at room temperature.

## Refinement

The H atom bonded to N1 was refined freely. All other H atoms were placed in calculated positions and constrained to ride on their parent atoms, with C—H = 0.93–0.97 Å and $U_{iso}$(H) = 1.2 $U_{eq}$(C) or 1.5$U_{eq}$(methyl C).

## Figures

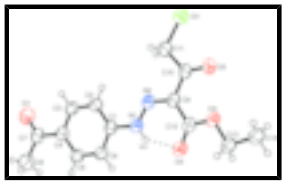

Fig. 1. The molecular structure of (I), showing the atom-numbering scheme and the intramolecular N—H···O hydrogen bond (dashed line). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

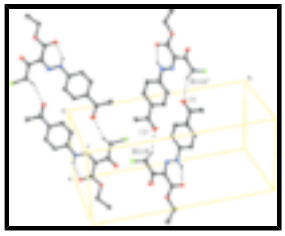

Fig. 2. Part of the crystal structure of (I), showing the formation of a hydrogen-bonded (dashed line) $R_2^2(24)$ dimers. H atoms not involved in the interactions have been omitted for clarity [Symmetry code: (i) -x, -y + 1, -z+1]

**(*Z*)-ethyl 2-(2-(4-acetylphenyl)hydrazono)-4-chloro-3-oxobutanoate**

*Crystal data*

| | |
|---|---|
| $C_{14}H_{15}ClN_2O_4$ | $F_{000}$ = 648 |
| $M_r$ = 310.73 | $D_x$ = 1.391 Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| Hall symbol: -P 2ybc | Cell parameters from 20436 reflections |
| $a$ = 8.6018 (5) Å | θ = 2.2–27.1º |
| $b$ = 18.9829 (16) Å | μ = 0.27 mm$^{-1}$ |
| $c$ = 9.1035 (6) Å | $T$ = 296 (2) K |
| β = 93.591 (5)º | Prism, yellow |
| $V$ = 1483.57 (18) Å$^3$ | 0.80 × 0.53 × 0.35 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Stoe IPDSII diffractometer | 2916 independent reflections |
| Radiation source: fine-focus sealed tube | 2277 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.032 |
| Detector resolution: 6.67 pixels mm$^{-1}$ | $\theta_{max}$ = 26.0º |
| $T$ = 296(2) K | $\theta_{min}$ = 2.2º |
| ω scans | $h$ = −10→10 |
| Absorption correction: integration (X-RED32; Stoe & Cie, 2002) | $k$ = −23→23 |
| $T_{min}$ = 0.854, $T_{max}$ = 0.914 | $l$ = −11→11 |
| 15055 measured reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | H atoms treated by a mixture of independent and constrained refinement |
| Least-squares matrix: full | $w = 1/[\sigma^2(F_o^2) + (0.0701P)^2 + 0.264P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)]$ = 0.044 | $(\Delta/\sigma)_{max}$ = 0.001 |
| $wR(F^2)$ = 0.130 | $\Delta\rho_{max}$ = 0.21 e Å$^{-3}$ |
| $S$ = 1.05 | $\Delta\rho_{min}$ = −0.31 e Å$^{-3}$ |

2916 reflections
195 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
Extinction correction: none

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.3964 (2) | 0.53258 (10) | 0.63530 (19) | 0.0532 (4) |
| C2 | 0.2686 (2) | 0.49933 (11) | 0.5648 (2) | 0.0599 (5) |
| H2 | 0.2413 | 0.4538 | 0.5905 | 0.072* |
| C3 | 0.1830 (2) | 0.53539 (11) | 0.4558 (2) | 0.0607 (5) |
| H3 | 0.0978 | 0.5134 | 0.4074 | 0.073* |
| C4 | 0.2205 (2) | 0.60356 (10) | 0.4163 (2) | 0.0558 (4) |
| C5 | 0.3510 (2) | 0.63541 (10) | 0.4873 (2) | 0.0593 (5) |
| H5 | 0.3796 | 0.6807 | 0.4608 | 0.071* |
| C6 | 0.4373 (2) | 0.60000 (10) | 0.5962 (2) | 0.0599 (5) |
| H6 | 0.5235 | 0.6216 | 0.6436 | 0.072* |
| C7 | 0.1191 (2) | 0.64027 (12) | 0.3018 (2) | 0.0663 (5) |
| C8 | 0.1670 (3) | 0.71088 (12) | 0.2475 (3) | 0.0872 (7) |
| H8A | 0.0867 | 0.7291 | 0.1800 | 0.131* |
| H8B | 0.2620 | 0.7063 | 0.1983 | 0.131* |
| H8C | 0.1830 | 0.7425 | 0.3293 | 0.131* |
| C9 | 0.5394 (2) | 0.40843 (10) | 0.9062 (2) | 0.0533 (4) |
| C10 | 0.4718 (2) | 0.34275 (11) | 0.9629 (2) | 0.0579 (4) |
| C11 | 0.3336 (3) | 0.31351 (13) | 0.8727 (3) | 0.0784 (6) |
| H11A | 0.3654 | 0.3009 | 0.7758 | 0.094* |
| H11B | 0.2540 | 0.3496 | 0.8605 | 0.094* |
| C12 | 0.6893 (2) | 0.43994 (11) | 0.9652 (2) | 0.0568 (4) |
| C13 | 0.9253 (2) | 0.42970 (12) | 1.1078 (3) | 0.0720 (6) |
| H13A | 0.9878 | 0.4393 | 1.0250 | 0.086* |
| H13B | 0.9092 | 0.4736 | 1.1591 | 0.086* |
| C14 | 1.0054 (3) | 0.37880 (13) | 1.2077 (3) | 0.0890 (8) |
| H14A | 1.1045 | 0.3976 | 1.2428 | 0.134* |

| | | | | |
|---|---|---|---|---|
| H14B | 1.0210 | 0.3356 | 1.1560 | 0.134* |
| H14C | 0.9432 | 0.3700 | 1.2897 | 0.134* |
| Cl1 | 0.25437 (7) | 0.23854 (3) | 0.95607 (8) | 0.0870 (2) |
| N1 | 0.48968 (19) | 0.49975 (9) | 0.74712 (18) | 0.0576 (4) |
| N2 | 0.45181 (16) | 0.43895 (8) | 0.80112 (16) | 0.0544 (4) |
| O1 | −0.0001 (2) | 0.61212 (11) | 0.2525 (2) | 0.0970 (6) |
| O2 | 0.5234 (2) | 0.31394 (10) | 1.07369 (18) | 0.0892 (5) |
| O3 | 0.73029 (17) | 0.49863 (8) | 0.92995 (17) | 0.0716 (4) |
| O4 | 0.77406 (15) | 0.39936 (8) | 1.05545 (15) | 0.0648 (4) |
| H1 | 0.565 (3) | 0.5192 (13) | 0.787 (3) | 0.079 (7)* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0474 (9) | 0.0614 (10) | 0.0506 (10) | 0.0050 (8) | 0.0004 (7) | −0.0017 (8) |
| C2 | 0.0522 (10) | 0.0628 (11) | 0.0641 (12) | −0.0038 (8) | −0.0004 (8) | 0.0047 (8) |
| C3 | 0.0462 (9) | 0.0723 (13) | 0.0626 (12) | −0.0054 (8) | −0.0042 (8) | −0.0001 (9) |
| C4 | 0.0498 (9) | 0.0622 (11) | 0.0551 (10) | 0.0078 (8) | 0.0008 (8) | −0.0027 (8) |
| C5 | 0.0597 (10) | 0.0533 (10) | 0.0643 (11) | 0.0018 (8) | −0.0024 (9) | −0.0036 (8) |
| C6 | 0.0554 (10) | 0.0613 (11) | 0.0615 (11) | −0.0035 (8) | −0.0072 (8) | −0.0048 (8) |
| C7 | 0.0592 (11) | 0.0736 (12) | 0.0651 (12) | 0.0135 (9) | −0.0043 (9) | −0.0014 (10) |
| C8 | 0.0988 (17) | 0.0635 (13) | 0.0960 (18) | 0.0180 (12) | −0.0197 (14) | 0.0059 (12) |
| C9 | 0.0469 (9) | 0.0629 (11) | 0.0497 (9) | 0.0053 (7) | −0.0010 (7) | −0.0039 (8) |
| C10 | 0.0512 (9) | 0.0664 (11) | 0.0549 (10) | 0.0048 (8) | −0.0064 (8) | −0.0001 (8) |
| C11 | 0.0664 (12) | 0.0841 (15) | 0.0815 (15) | −0.0147 (11) | −0.0198 (11) | 0.0172 (12) |
| C12 | 0.0532 (10) | 0.0659 (12) | 0.0505 (10) | 0.0054 (8) | −0.0028 (8) | −0.0054 (8) |
| C13 | 0.0593 (11) | 0.0729 (13) | 0.0809 (14) | −0.0110 (9) | −0.0186 (10) | 0.0019 (11) |
| C14 | 0.0744 (14) | 0.0777 (15) | 0.1103 (19) | −0.0128 (11) | −0.0308 (13) | 0.0152 (13) |
| Cl1 | 0.0707 (4) | 0.0790 (4) | 0.1079 (5) | −0.0144 (3) | −0.0203 (3) | 0.0178 (3) |
| N1 | 0.0505 (9) | 0.0621 (10) | 0.0591 (10) | 0.0010 (7) | −0.0054 (7) | 0.0022 (7) |
| N2 | 0.0470 (8) | 0.0632 (9) | 0.0528 (8) | 0.0058 (6) | 0.0009 (6) | −0.0007 (7) |
| O1 | 0.0690 (10) | 0.1116 (13) | 0.1057 (13) | −0.0014 (9) | −0.0305 (9) | 0.0230 (10) |
| O2 | 0.0887 (11) | 0.0929 (11) | 0.0812 (10) | −0.0223 (9) | −0.0319 (9) | 0.0260 (9) |
| O3 | 0.0642 (8) | 0.0690 (9) | 0.0792 (10) | −0.0077 (7) | −0.0154 (7) | 0.0044 (7) |
| O4 | 0.0525 (7) | 0.0696 (8) | 0.0702 (9) | −0.0038 (6) | −0.0134 (6) | 0.0051 (6) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—C6 | 1.380 (3) | C9—C10 | 1.482 (3) |
| C1—C2 | 1.390 (3) | C9—C12 | 1.490 (3) |
| C1—N1 | 1.402 (2) | C10—O2 | 1.207 (2) |
| C2—C3 | 1.379 (3) | C10—C11 | 1.508 (3) |
| C2—H2 | 0.93 | C11—Cl1 | 1.769 (2) |
| C3—C4 | 1.387 (3) | C11—H11A | 0.97 |
| C3—H3 | 0.93 | C11—H11B | 0.97 |
| C4—C5 | 1.397 (3) | C12—O3 | 1.218 (2) |
| C4—C7 | 1.490 (3) | C12—O4 | 1.313 (2) |
| C5—C6 | 1.376 (3) | C13—C14 | 1.469 (3) |
| C5—H5 | 0.93 | C13—O4 | 1.474 (2) |

| | | | |
|---|---|---|---|
| C6—H6 | 0.93 | C13—H13A | 0.97 |
| C7—O1 | 1.217 (3) | C13—H13B | 0.97 |
| C7—C8 | 1.495 (3) | C14—H14A | 0.96 |
| C8—H8A | 0.96 | C14—H14B | 0.96 |
| C8—H8B | 0.96 | C14—H14C | 0.96 |
| C8—H8C | 0.96 | N1—N2 | 1.304 (2) |
| C9—N2 | 1.315 (2) | N1—H1 | 0.82 (2) |
| C6—C1—C2 | 120.60 (17) | O2—C10—C9 | 122.72 (17) |
| C6—C1—N1 | 117.13 (16) | O2—C10—C11 | 121.60 (19) |
| C2—C1—N1 | 122.27 (17) | C9—C10—C11 | 115.68 (16) |
| C3—C2—C1 | 118.56 (18) | C10—C11—Cl1 | 111.99 (15) |
| C3—C2—H2 | 120.7 | C10—C11—H11A | 109.2 |
| C1—C2—H2 | 120.7 | Cl1—C11—H11A | 109.2 |
| C2—C3—C4 | 121.82 (17) | C10—C11—H11B | 109.2 |
| C2—C3—H3 | 119.1 | Cl1—C11—H11B | 109.2 |
| C4—C3—H3 | 119.1 | H11A—C11—H11B | 107.9 |
| C3—C4—C5 | 118.49 (17) | O3—C12—O4 | 122.89 (17) |
| C3—C4—C7 | 118.85 (17) | O3—C12—C9 | 122.02 (17) |
| C5—C4—C7 | 122.65 (18) | O4—C12—C9 | 115.08 (18) |
| C6—C5—C4 | 120.22 (19) | C14—C13—O4 | 108.28 (17) |
| C6—C5—H5 | 119.9 | C14—C13—H13A | 110.0 |
| C4—C5—H5 | 119.9 | O4—C13—H13A | 110.0 |
| C5—C6—C1 | 120.29 (17) | C14—C13—H13B | 110.0 |
| C5—C6—H6 | 119.9 | O4—C13—H13B | 110.0 |
| C1—C6—H6 | 119.9 | H13A—C13—H13B | 108.4 |
| O1—C7—C4 | 119.7 (2) | C13—C14—H14A | 109.5 |
| O1—C7—C8 | 121.0 (2) | C13—C14—H14B | 109.5 |
| C4—C7—C8 | 119.31 (19) | H14A—C14—H14B | 109.5 |
| C7—C8—H8A | 109.5 | C13—C14—H14C | 109.5 |
| C7—C8—H8B | 109.5 | H14A—C14—H14C | 109.5 |
| H8A—C8—H8B | 109.5 | H14B—C14—H14C | 109.5 |
| C7—C8—H8C | 109.5 | N2—N1—C1 | 121.46 (16) |
| H8A—C8—H8C | 109.5 | N2—N1—H1 | 116.2 (17) |
| H8B—C8—H8C | 109.5 | C1—N1—H1 | 122.1 (17) |
| N2—C9—C10 | 113.87 (15) | N1—N2—C9 | 121.27 (16) |
| N2—C9—C12 | 121.93 (17) | C12—O4—C13 | 114.65 (16) |
| C10—C9—C12 | 124.15 (16) | | |
| C6—C1—C2—C3 | −0.5 (3) | N2—C9—C10—C11 | 12.1 (2) |
| N1—C1—C2—C3 | −179.95 (17) | C12—C9—C10—C11 | −170.44 (19) |
| C1—C2—C3—C4 | −0.5 (3) | O2—C10—C11—Cl1 | 4.1 (3) |
| C2—C3—C4—C5 | 1.5 (3) | C9—C10—C11—Cl1 | −176.18 (15) |
| C2—C3—C4—C7 | −177.58 (18) | N2—C9—C12—O3 | 7.6 (3) |
| C3—C4—C5—C6 | −1.5 (3) | C10—C9—C12—O3 | −169.67 (18) |
| C7—C4—C5—C6 | 177.56 (18) | N2—C9—C12—O4 | −171.31 (16) |
| C4—C5—C6—C1 | 0.5 (3) | C10—C9—C12—O4 | 11.4 (3) |
| C2—C1—C6—C5 | 0.5 (3) | C6—C1—N1—N2 | 173.36 (17) |
| N1—C1—C6—C5 | 179.98 (17) | C2—C1—N1—N2 | −7.2 (3) |
| C3—C4—C7—O1 | 5.2 (3) | C1—N1—N2—C9 | −179.82 (16) |

| | | | |
|---|---|---|---|
| C5—C4—C7—O1 | −173.9 (2) | C10—C9—N2—N1 | 175.03 (16) |
| C3—C4—C7—C8 | −173.91 (19) | C12—C9—N2—N1 | −2.5 (3) |
| C5—C4—C7—C8 | 7.0 (3) | O3—C12—O4—C13 | −1.6 (3) |
| N2—C9—C10—O2 | −168.1 (2) | C9—C12—O4—C13 | 177.30 (16) |
| C12—C9—C10—O2 | 9.3 (3) | C14—C13—O4—C12 | 179.58 (19) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O3 | 0.82 (2) | 1.90 (2) | 2.573 (2) | 139 (2) |
| C11—H11B···O1$^{i}$ | 0.97 | 2.47 | 3.334 (3) | 149 |

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

**Fig. 1**

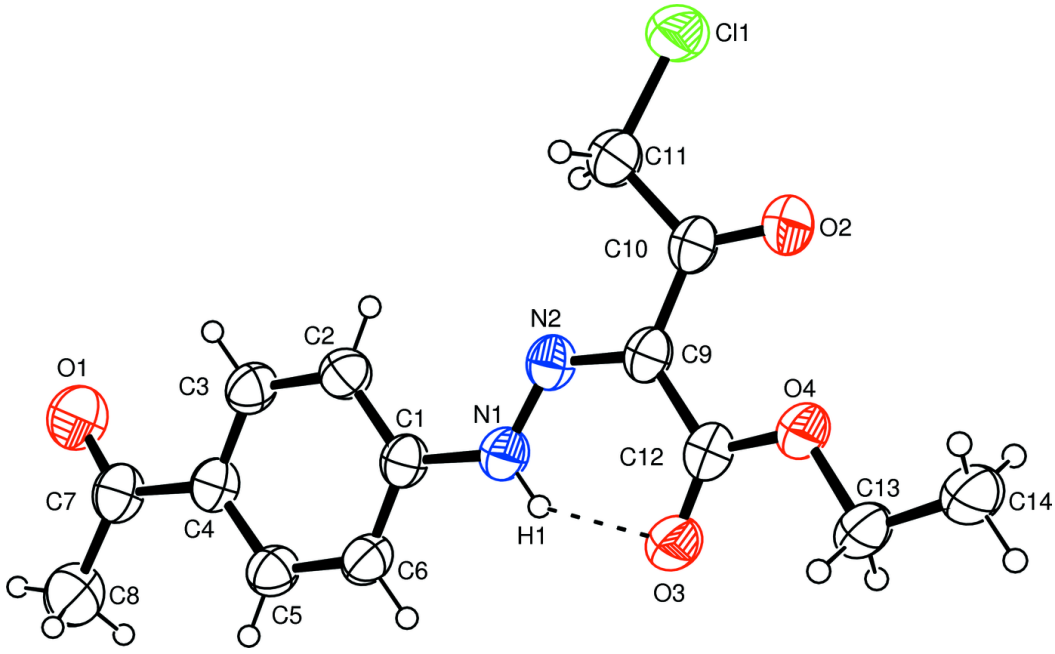
Cl1
C11
O2
C10
N2
C9
O4
C12
C13
C14
O1
C3
C2
C1
N1
C7
C4
H1
O3
C8
C5
C6

**Fig. 2**