

(E)-1-(4-Chlorophenyl)ethanone semicarbazone

Hoong-Kun Fun,^{a*‡} Ching Kheng Quah,^{a§} Mahesh Padaki,^b Shridhar Malladi^b and Arun M. Isloor^b

^aX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and ^bDepartment of Chemistry, National Institute of Technology—Karnataka, Surathkal, Mangalore 575 025, India
Correspondence e-mail: hkfun@usm.my

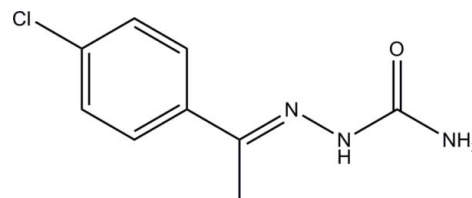
Received 11 June 2009; accepted 13 June 2009

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.039; wR factor = 0.101; data-to-parameter ratio = 21.2.

In the title compound, $\text{C}_9\text{H}_{10}\text{ClN}_3\text{O}$, the semicarbazone group is approximately planar, with an r.m.s. deviation from the mean plane of 0.054 (1) Å. The dihedral angle between the least-squares planes through the semicarbazone group and the benzene ring is 30.46 (5)°. In the solid state, molecules are linked *via* intermolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds, generating $R_2^2(9)$ ring motifs which, together with $R_2^2(8)$ ring motifs formed by pairs of intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, lead to the formation of a seldom-observed molecular trimer. Furthermore, $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds form $R_2^1(7)$ ring motifs with $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, further consolidating the crystal structure. Molecules are linked by these intermolecular interactions, forming two-dimensional networks parallel to (100).

Related literature

For the synthetic utility and applications of semicarbazone derivatives, see: Warren *et al.* (1977); Chandra & Gupta (2005); Jain *et al.* (2002); Pilgram (1978); Yogeewari *et al.* (2004). For a related structure, see: Fun *et al.* (2009). For the preparation, see: Furniss *et al.* (1978). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For bond-length data, see: Allen *et al.* (1987). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$\text{C}_9\text{H}_{10}\text{ClN}_3\text{O}$
 $M_r = 211.65$
Monoclinic, $C2/c$
 $a = 21.8191$ (4) Å
 $b = 7.0484$ (1) Å
 $c = 13.7249$ (2) Å
 $\beta = 109.633$ (1)°

$V = 1988.04$ (6) Å³
 $Z = 8$
Mo $K\alpha$ radiation
 $\mu = 0.35$ mm⁻¹
 $T = 100$ K
 $0.41 \times 0.20 \times 0.03$ mm

Data collection

Bruker SMART APEXII CCD
area-detector diffractometer
Absorption correction: multi-scan
(*SADABS*; Bruker, 2005)
 $T_{\text{min}} = 0.867$, $T_{\text{max}} = 0.991$

28636 measured reflections
3539 independent reflections
2912 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.052$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.101$
 $S = 1.04$
3539 reflections
167 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.45$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.27$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|------------|-------------|-------------|---------------|
| $\text{N2}-\text{H1N2}\cdots\text{O1}^{\text{i}}$ | 0.884 (19) | 2.007 (19) | 2.8866 (12) | 173.3 (19) |
| $\text{N3}-\text{H1N3}\cdots\text{N1}^{\text{ii}}$ | 0.835 (18) | 2.264 (18) | 3.0904 (14) | 170.5 (16) |
| $\text{N3}-\text{H2N3}\cdots\text{O1}^{\text{iii}}$ | 0.826 (17) | 2.316 (17) | 3.0499 (13) | 148.4 (15) |
| $\text{C9}-\text{H9C}\cdots\text{O1}^{\text{i}}$ | 0.94 (2) | 2.55 (2) | 3.2162 (16) | 128.1 (16) |

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINTE* (Bruker, 2005); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

HKF and CKQ thank Universiti Sains Malaysia (USM) for the Research University Golden Goose Grant (1001/PFIZIK/811012). CKQ thanks USM for a Research Fellowship. AMI is grateful to the Head of the Department of Chemistry and the Director, NITK, Surathkal, India, for providing research facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2631).

‡ Thomson Reuters ResearcherID: A-3561-2009.

§ Thomson Reuters ResearcherID: A-5525-2009.

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.
- Bernstein, J., Davis, R. E., Shimon, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Bruker (2005). *APEX2, SAINT and SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Chandra, S. & Gupta, L. K. (2005). *Spectrochim Acta Part A*, **62**, 1089–94.
- Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
- Fun, H.-K., Yeap, C. S., Padaki, M., Malladi, S. & Isloor, A. M. (2009). *Acta Cryst.* **E65**, o1619–o1620.
- Furniss, B. S., Hannaford, A. J., Rogers, V., Smith, P. W. G. & Tatchell, A. R. (1978). *Vogel's Textbook of Practical Organic Chemistry*, 4th ed., p. 1112. London:ELBS.
- Jain, V. K., Handa, A., Pandya, R., Shrivastav, P. & Agrawal, Y. K. (2002). *React. Funct. Polym.* **51**, 101–110.
- Pilgram, K. H. G. (1978). US Patent No. 4 108 399.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Warren, J. D., Woodward, D. L. & Hargreaves, R. T. (1977). *J. Med. Chem.* **20**, 1520–1521.
- Yogeeswari, P., Sriram, D., Pandeya, S. N. & Stables, J. P. (2004). *Farmaco*, **59**, 609–613.

supplementary materials

Acta Cryst. (2009). E65, o1634-o1635 [doi:10.1107/S160053680902279X]

(E)-1-(4-Chlorophenyl)ethanone semicarbazone

H.-K. Fun, C. K. Quah, M. Padaki, S. Malladi and A. M. Isloor

Comment

In organic chemistry, a semicarbazone is a derivative of an aldehyde or ketone formed by a condensation between a ketone or aldehyde and semicarbazide. Semicarbazones find numerous applications in the field of synthetic chemistry, such as medicinal chemistry (Warren *et al.*, 1977), organometalics (Chandra & Gupta, 2005), polymers (Jain *et al.*, 2002) and herbicides (Pilgram, 1978). 4-Sulphamoylphenyl semicarbazones were synthesized and were found to possess anticonvulsant activity (Yogeeswari *et al.*, 2004). Herein we report the crystal structure of the title semicarbazone which may have commercial and synthetic importance.

The bond lengths (Allen *et al.*, 1987) and angles in the molecule (Fig. 1) are within normal ranges, and are comparable to those observed in a closely related structure (Fun *et al.*, 2009). The semicarbazone group (C9/C6/C7/N1/N2/C8/O1/N3) is approximately planar, with an r.m.s. deviation of 0.054 (1) Å for atom N2 while the dihedral angle between the least-squares planes through the semicarbazone group and the benzene ring is 30.46 (5)°.

In the solid state, the molecules are linked *via* intermolecular N3—H2N3···O1 and N3—H1N3···N1 hydrogen bonds to generate $R_2^2(9)$ ring motifs which, together with the $R_2^2(8)$ ring motifs formed by pairs of intermolecular N2—H1N2···O1 hydrogen bonds, lead to the formation of a seldom-observed molecular trimer (Fig. 2). Furthermore, N2—H1N2···O1 hydrogen bonds form $R_2^1(7)$ ring motifs (Fig. 2) with C9—H9C···O1 hydrogen bonds to further consolidated the crystal structure. The molecules are linked by these intermolecular interactions to form two-dimensional networks parallel to the (1 0 0) plane.

Experimental

0.780 g (7.0 mmol) of semicarbazide hydrochloride and 0.698 g (8.5 mmol) of crystallized sodium acetate was dissolved in 10 ml of water (Furniss *et al.*, 1978). The reaction mixture was stirred at room temperature for 10 minutes. To this (1 g, 6.5 mmol) of 4-choloacetophenone was added and shaken well. A little alcohol was added to dissolve the turbidity. It was shaken for 10 more minutes and allowed to stand. The semicarbazone crystallizes on standing for 6 h. The separated crystals were filtered, washed with cold water and recrystallized from alcohol. Yield was found to be 1.1 g, 80.35%. *M.p.* 478–479 K.

Refinement

All hydrogen atoms were located from the difference Fourier map and refined freely.

Figures

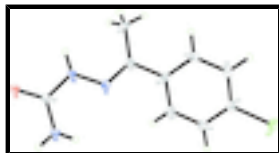


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

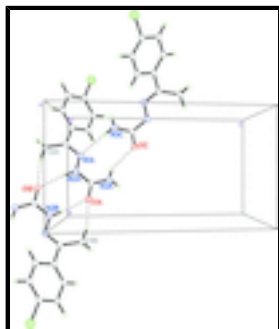


Fig. 2. Part of the crystal packing of the title compound, viewed along the *a* axis, showing the formation of a molecular trimer. Atom-numbering is shown for those non-H atoms involved in hydrogen bonds and intermolecular interactions are shown as dashed lines. Molecule A is related to molecules B and C via symmetry codes of $-x + 1/2, -y + 1/2, -z$ and $-x + 1/2, y + 1/2, -z + 1/2$, respectively.

(E)-1-(4-Chlorophenyl)ethanone semicarbazone

Crystal data

$C_9H_{10}ClN_3O$

$M_r = 211.65$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 21.8191\ (4)\ \text{\AA}$

$b = 7.0484\ (1)\ \text{\AA}$

$c = 13.7249\ (2)\ \text{\AA}$

$\beta = 109.633\ (1)^\circ$

$V = 1988.04\ (6)\ \text{\AA}^3$

$Z = 8$

$F_{000} = 880$

$D_x = 1.414\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 6351 reflections

$\theta = 3.1\text{--}32.1^\circ$

$\mu = 0.35\ \text{mm}^{-1}$

$T = 100\ \text{K}$

Plate, colourless

$0.41 \times 0.20 \times 0.03\ \text{mm}$

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 100\ \text{K}$

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

$T_{\min} = 0.867, T_{\max} = 0.991$

28636 measured reflections

3539 independent reflections

2912 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.052$

$\theta_{\max} = 32.3^\circ$

$\theta_{\min} = 3.1^\circ$

$h = -32 \rightarrow 32$

$k = -10 \rightarrow 10$

$l = -20 \rightarrow 20$

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.039$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.101$ | $w = 1/[\sigma^2(F_o^2) + (0.049P)^2 + 1.2937P]$ |
| $S = 1.04$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 3539 reflections | $(\Delta/\sigma)_{\max} = 0.001$ |
| 167 parameters | $\Delta\rho_{\max} = 0.45 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$ |
| | Extinction correction: none |

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cyrosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|--------------|----------------------------------|
| Cl1 | 0.023375 (16) | -0.37949 (4) | 0.13113 (2) | 0.02330 (9) |
| O1 | 0.28038 (4) | 0.80096 (12) | 0.13075 (6) | 0.01675 (17) |
| N1 | 0.18652 (5) | 0.38677 (13) | 0.08164 (7) | 0.01332 (18) |
| N2 | 0.22047 (5) | 0.53956 (13) | 0.06342 (7) | 0.01416 (18) |
| N3 | 0.24395 (6) | 0.61942 (14) | 0.23627 (8) | 0.0176 (2) |
| C1 | 0.11370 (6) | -0.05633 (16) | -0.01844 (9) | 0.0167 (2) |
| C2 | 0.08397 (6) | -0.21004 (16) | 0.01146 (10) | 0.0178 (2) |
| C3 | 0.06045 (6) | -0.18847 (16) | 0.09274 (9) | 0.0172 (2) |
| C4 | 0.06590 (6) | -0.01713 (17) | 0.14505 (9) | 0.0184 (2) |
| C5 | 0.09612 (6) | 0.13476 (16) | 0.11541 (9) | 0.0167 (2) |
| C6 | 0.12052 (6) | 0.11705 (15) | 0.03359 (9) | 0.0136 (2) |
| C7 | 0.15469 (6) | 0.27915 (15) | 0.00516 (9) | 0.0137 (2) |
| C8 | 0.24965 (6) | 0.65995 (15) | 0.14448 (8) | 0.0136 (2) |
| C9 | 0.15172 (7) | 0.30648 (18) | -0.10430 (9) | 0.0189 (2) |
| H1 | 0.1299 (8) | -0.071 (2) | -0.0751 (13) | 0.022 (4)* |

supplementary materials

| | | | | |
|------|-------------|------------|--------------|------------|
| H2 | 0.0807 (8) | -0.332 (3) | -0.0235 (13) | 0.029 (4)* |
| H4 | 0.0472 (8) | -0.007 (2) | 0.2047 (13) | 0.026 (4)* |
| H5 | 0.0998 (8) | 0.254 (2) | 0.1502 (13) | 0.024 (4)* |
| H9A | 0.1211 (11) | 0.237 (3) | -0.1478 (18) | 0.053 (6)* |
| H9B | 0.1901 (12) | 0.271 (3) | -0.1107 (18) | 0.064 (7)* |
| H9C | 0.1416 (9) | 0.433 (3) | -0.1261 (15) | 0.036 (5)* |
| H1N2 | 0.2197 (8) | 0.579 (3) | 0.0019 (14) | 0.028 (4)* |
| H1N3 | 0.2668 (8) | 0.684 (2) | 0.2863 (13) | 0.022 (4)* |
| H2N3 | 0.2271 (8) | 0.522 (2) | 0.2487 (12) | 0.020 (4)* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|---------------|--------------|--------------|
| C11 | 0.02608 (17) | 0.01993 (15) | 0.02367 (16) | -0.00699 (11) | 0.00806 (12) | 0.00422 (10) |
| O1 | 0.0227 (4) | 0.0154 (4) | 0.0128 (4) | -0.0054 (3) | 0.0067 (3) | -0.0010 (3) |
| N1 | 0.0157 (4) | 0.0121 (4) | 0.0120 (4) | -0.0013 (3) | 0.0044 (3) | 0.0007 (3) |
| N2 | 0.0202 (5) | 0.0127 (4) | 0.0100 (4) | -0.0042 (3) | 0.0056 (4) | -0.0007 (3) |
| N3 | 0.0272 (6) | 0.0163 (4) | 0.0102 (4) | -0.0065 (4) | 0.0076 (4) | -0.0015 (3) |
| C1 | 0.0176 (5) | 0.0165 (5) | 0.0163 (5) | -0.0024 (4) | 0.0064 (4) | -0.0024 (4) |
| C2 | 0.0183 (6) | 0.0141 (5) | 0.0204 (6) | -0.0024 (4) | 0.0055 (4) | -0.0019 (4) |
| C3 | 0.0165 (5) | 0.0148 (5) | 0.0188 (5) | -0.0024 (4) | 0.0039 (4) | 0.0037 (4) |
| C4 | 0.0200 (6) | 0.0194 (5) | 0.0166 (5) | -0.0019 (4) | 0.0074 (4) | 0.0013 (4) |
| C5 | 0.0200 (6) | 0.0151 (5) | 0.0158 (5) | -0.0013 (4) | 0.0073 (4) | -0.0010 (4) |
| C6 | 0.0138 (5) | 0.0142 (5) | 0.0121 (5) | -0.0004 (4) | 0.0034 (4) | 0.0013 (4) |
| C7 | 0.0158 (5) | 0.0127 (4) | 0.0121 (5) | -0.0006 (4) | 0.0040 (4) | 0.0003 (4) |
| C8 | 0.0164 (5) | 0.0129 (4) | 0.0109 (5) | -0.0002 (4) | 0.0040 (4) | -0.0005 (4) |
| C9 | 0.0266 (7) | 0.0185 (5) | 0.0124 (5) | -0.0052 (5) | 0.0077 (5) | -0.0008 (4) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|------------|-------------|----------|-------------|
| C11—C3 | 1.7410 (12) | C2—C3 | 1.3842 (18) |
| O1—C8 | 1.2481 (13) | C2—H2 | 0.977 (18) |
| N1—C7 | 1.2922 (14) | C3—C4 | 1.3895 (17) |
| N1—N2 | 1.3768 (13) | C4—C5 | 1.3882 (16) |
| N2—C8 | 1.3737 (14) | C4—H4 | 1.033 (17) |
| N2—H1N2 | 0.883 (18) | C5—C6 | 1.4006 (16) |
| N3—C8 | 1.3378 (14) | C5—H5 | 0.958 (17) |
| N3—H1N3 | 0.835 (18) | C6—C7 | 1.4863 (15) |
| N3—H2N3 | 0.826 (17) | C7—C9 | 1.4943 (16) |
| C1—C2 | 1.3935 (16) | C9—H9A | 0.88 (2) |
| C1—C6 | 1.3979 (15) | C9—H9B | 0.91 (2) |
| C1—H1 | 0.963 (16) | C9—H9C | 0.94 (2) |
| C7—N1—N2 | 119.12 (9) | C4—C5—C6 | 120.81 (11) |
| C8—N2—N1 | 117.86 (9) | C4—C5—H5 | 120.0 (10) |
| C8—N2—H1N2 | 115.9 (12) | C6—C5—H5 | 119.1 (10) |
| N1—N2—H1N2 | 125.4 (12) | C1—C6—C5 | 118.92 (10) |
| C8—N3—H1N3 | 116.1 (12) | C1—C6—C7 | 120.97 (10) |
| C8—N3—H2N3 | 124.2 (11) | C5—C6—C7 | 120.08 (10) |

| | | | |
|--------------|-------------|-------------|--------------|
| H1N3—N3—H2N3 | 118.0 (16) | N1—C7—C6 | 114.71 (10) |
| C2—C1—C6 | 120.63 (11) | N1—C7—C9 | 124.82 (10) |
| C2—C1—H1 | 119.1 (10) | C6—C7—C9 | 120.46 (10) |
| C6—C1—H1 | 120.3 (10) | O1—C8—N3 | 122.47 (10) |
| C3—C2—C1 | 119.16 (11) | O1—C8—N2 | 119.73 (10) |
| C3—C2—H2 | 120.4 (10) | N3—C8—N2 | 117.80 (10) |
| C1—C2—H2 | 120.4 (10) | C7—C9—H9A | 112.1 (14) |
| C2—C3—C4 | 121.43 (11) | C7—C9—H9B | 109.4 (15) |
| C2—C3—C11 | 119.65 (9) | H9A—C9—H9B | 107 (2) |
| C4—C3—C11 | 118.92 (9) | C7—C9—H9C | 111.6 (11) |
| C5—C4—C3 | 119.04 (11) | H9A—C9—H9C | 105.6 (19) |
| C5—C4—H4 | 122.2 (10) | H9B—C9—H9C | 110.9 (19) |
| C3—C4—H4 | 118.7 (10) | | |
| C7—N1—N2—C8 | 175.03 (10) | C4—C5—C6—C7 | -177.97 (11) |
| C6—C1—C2—C3 | 0.79 (18) | N2—N1—C7—C6 | 179.22 (9) |
| C1—C2—C3—C4 | -0.05 (18) | N2—N1—C7—C9 | 0.30 (17) |
| C1—C2—C3—C11 | -179.84 (9) | C1—C6—C7—N1 | -146.43 (11) |
| C2—C3—C4—C5 | -0.55 (19) | C5—C6—C7—N1 | 31.82 (15) |
| C11—C3—C4—C5 | 179.24 (9) | C1—C6—C7—C9 | 32.55 (16) |
| C3—C4—C5—C6 | 0.41 (18) | C5—C6—C7—C9 | -149.20 (12) |
| C2—C1—C6—C5 | -0.92 (18) | N1—N2—C8—O1 | -179.37 (10) |
| C2—C1—C6—C7 | 177.35 (11) | N1—N2—C8—N3 | 0.92 (16) |
| C4—C5—C6—C1 | 0.32 (17) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|-----------------------------|-------------|---------------|-----------------------|-------------------------|
| N2—H1N2...O1 ⁱ | 0.884 (19) | 2.007 (19) | 2.8866 (12) | 173.3 (19) |
| N3—H1N3...N1 ⁱⁱ | 0.835 (18) | 2.264 (18) | 3.0904 (14) | 170.5 (16) |
| N3—H2N3...O1 ⁱⁱⁱ | 0.826 (17) | 2.316 (17) | 3.0499 (13) | 148.4 (15) |
| C9—H9C...O1 ⁱ | 0.94 (2) | 2.55 (2) | 3.2162 (16) | 128.1 (16) |

Symmetry codes: (i) $-x+1/2, -y+3/2, -z$; (ii) $-x+1/2, y+1/2, -z+1/2$; (iii) $-x+1/2, y-1/2, -z+1/2$.

Fig. 1

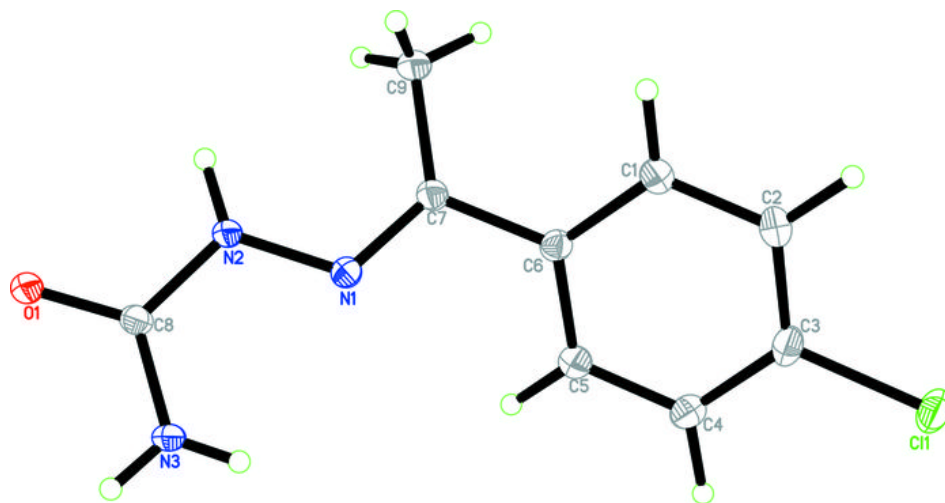


Fig. 2

