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Ethyl 4-chloro-2-[(4-nitrophenyl)hydrazono]-3-oxobutyrate

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The title compound, $C_{12}H_{12}N_3O_5$, adopts a keto-hydrazo tautomeric form stabilized by an intramolecular hydrogen bond. The aromatic ring and aliphatic chain, which adopt a *trans* configuration about the N—N bond, are nearly coplanar, with a dihedral angle of 18.30 (6)° between them. The molecules pack *via* weak intermolecular C—H···O hydrogen bonds which, together with an intramolecular N—H···O bond, form an $S(6)C(13)$ motif. The structure is further stabilized by C—H···π and π–π interactions.

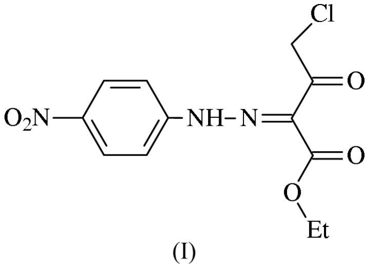
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.032
wR factor = 0.083
Data-to-parameter ratio = 11.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The chemical background of the title compound, (I), was described in the previous paper (Odabaşoğlu *et al.*, 2005). The molecular structure and the atom-labelling scheme are shown in Fig. 1. Selected bond lengths and angles are listed in Table 1.



(I)

The molecule adopts a (Z) configuration with respect to the N1—N2 bond, and the dihedral angle between the aromatic

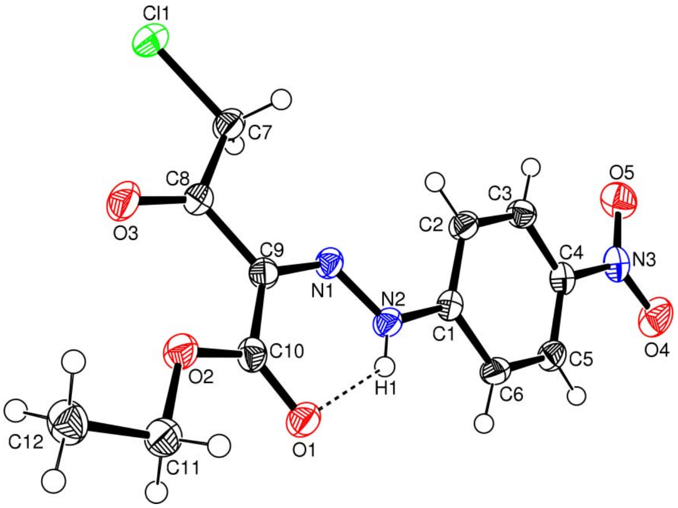


Figure 1
The structure of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids. The intramolecular hydrogen bond is drawn as a dashed line.

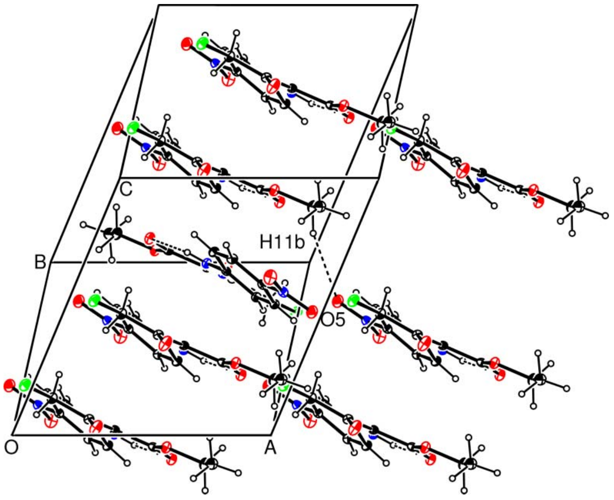


Figure 2
A view of the packing of (I). Hydrogen bonds are drawn as dashed lines.

C1–C6 ring (*A*) and the plane of the C7/C8/C9/C10/O3 aliphatic chain is 18.30 (6)°. The dihedral angles between the H1/N2/N1/C9/C10/O1 ring formed through intramolecular hydrogen bonding, the –NO_2 and C7/C8/C9/C10/O1/O3/Cl1 planes and ring *A* are 16.48 (5), 6.60 (6) and 20.63 (1)°, respectively.

In the crystal structure, molecules of (I) are linked through C11—H11*B*···O5 hydrogen bonds, forming chains along the *a* axis (Table 2 and Fig. 2). The hydrogen-bonded molecules are arranged in an *S*(6)*C*(13) motif (Bernstein *et al.*, 1995).

The packing is further stabilized by C—H···π and π–π interactions. There is a C—H···π interaction between the ethyl group and the aromatic ring. For the C12–H12*B*···π contact, the distance between atom H12*B* and the aromatic ring centroid is 2.82 (3) Å [symmetry code $(1-x, 1-x, 1-z)$], with an angle of 158 (2)°. There is also π–π stacking between adjacent molecules, with N1···C10^{i} and N1···O1^{i} distances of 3.216 (2) and 3.415 (2) Å, respectively (Fig. 2) [symmetry code: (i) $1-x, 1-y, 1-z$].

Experimental

Compound (I) was prepared as the 4-chloro-2-[(4-nitrophenyl)-hydrazono]-3-oxobutyric acid ethyl ester (Odabaşoğlu *et al.*, 2005), using 4-nitroaniline and ethyl 4-chloroacetoacetate as starting materials (yield 91%; m.p. 419–421 K). The compound was recrystallized from glacial acetic acid.

Crystal data

$C_{12}H_{12}ClN_3O_5$
$M_r = 313.70$
Triclinic, $P\bar{1}$
$a = 9.0131$ (8) Å
$b = 9.4161$ (9) Å
$c = 9.8932$ (9) Å
$\alpha = 62.458$ (7)°
$\beta = 66.520$ (7)°
$\gamma = 86.438$ (7)°
$V = 675.08$ (13) Å^3
$Z = 2$
$D_x = 1.543$ Mg m^{-3}
Mo *K*α radiation
Cell parameters from 12102 reflections
$\theta = 3.2$–26.0°
$\mu = 0.31$ mm^{-1}
$T = 296$ (2) K
Irregular fragment, dark red
0.42 × 0.33 × 0.18 mm

Data collection

Stoe IPDS 2 diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min} = 0.884$, $T_{max} = 0.946$
12102 measured reflections
2654 independent reflections
2336 reflections with $I > 2\sigma(I)$
$R_{int} = 0.063$
$\theta_{max} = 25.6°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.083$
$S = 1.04$
2654 reflections
238 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0411P)^2 + 0.167P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N2	1.396 (2)	C8—C9	1.491 (2)
C4—N3	1.460 (2)	C9—N1	1.306 (2)
C7—C8	1.515 (2)	C9—C10	1.481 (2)
C7—Cl1	1.773 (1)	C10—O1	1.223 (2)
C8—O3	1.203 (2)	N1—N2	1.315 (2)
O3—C8—C9	123.11 (13)	C9—N1—N2	122.43 (13)
C10—C9—C8	123.84 (12)	N1—N2—C1	118.68 (12)
O1—C10—C9	121.80 (13)		
O3—C8—C9—N1	168.59 (14)	C9—N1—N2—C1	−179.88 (12)
N1—C9—C10—O1	−5.5 (2)	C3—C4—N3—O5	−6.66 (19)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1···O1	0.78 (2)	2.02 (2)	2.6193 (17)	134 (2)
C11—H11*B*···O5^{i}	0.99 (2)	2.51 (2)	3.441 (2)	157 (2)

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

All H atoms were refined freely, with C—H distances in the range 0.87 (2)–0.99 (2) Å and an N—H distance of 0.78 (2) Å, and with U_{iso}(H) values in the range 0.026 (4)–0.062 (7) Å^2.

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: *ORTEP3* for Windows (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.*. **32**, 837–838.
Odabaşoğlu, M., Özdamar, Ö. & Büyükgüngör O. (2005). *Acta Cryst.* E**61**, o2065–o2067..

Acta Cryst. (2005). E**61**, o2068–o2070 Odabaşoğlu *et al.* • $C_{12}H_{12}ClN_3O_5$ **o2069**

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.

o2070 Odabaşoğlu *et al.* • $C_{12}H_{12}ClN_3O_5$ *Acta Cryst.* (2005). E**61**, o2068–o2070