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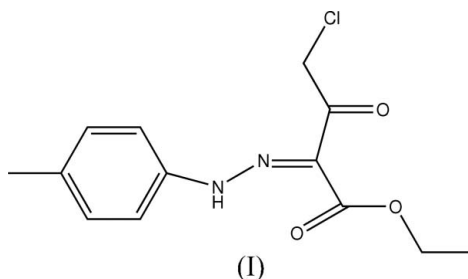
## Key indicators

Single-crystal X-ray study  
 $T = 296$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å  
 $R$  factor = 0.034  
 $wR$  factor = 0.097  
Data-to-parameter ratio = 15.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**(Z)-Ethyl 4-chloro-3-oxo-2-(2-*p*-tolyl-hydrazono)butanoate**The title compound,  $\text{C}_{13}\text{H}_{15}\text{ClN}_2\text{O}_3$ , adopts a keto-hydrazo tautomeric form stabilized by an intramolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond. The configuration around the  $\text{N}-\text{N}$  bond is *trans*. The dihedral angle between the aromatic ring and the plane of the aliphatic chain is  $3.18(4)^\circ$ .

Received 17 October 2006

Accepted 26 October 2006

## Comment

The chemistry of hydrazones has been intensively investigated in recent years, owing to their coordination capability, pharmacological activity, and antibacterial and antifungal properties, and their use in analytical chemistry as highly selective extractants (Domiano *et al.*, 1984; Sakamoto *et al.*, 1993; Li *et al.*, 1988). 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 are shown in Fig. 1. The molecule is approximately planar with the dihedral angle between the aromatic  $\text{C}1-\text{C}6$  ring and the plane of the  $\text{C}8-\text{C}13/\text{O}1-\text{O}3/\text{Cl}1$  aliphatic chain being  $3.18(4)^\circ$ .In (I), the imino group adopts a (*Z*) configuration. The bond lengths (Table 1) show that there is significant elongation of the  $\text{N}1-\text{N}2$  bond and contraction of the  $\text{C}1-\text{N}1$  bond in comparison with azo compounds. For example, the  $\text{N}=\text{N}$  and  $\text{C}-\text{N}$  bonds in azobenzene are  $1.249(4)$  and  $1.431(4)$  Å, respectively (Harada *et al.*, 1997), while the  $\text{C}=\text{N}$  double bonds in imino compounds are in the range  $1.272(2)$ – $1.303(3)$  Å (Galić *et al.*, 2000; Baughman *et al.*, 2004; Ersanlı, Albayrak *et al.*, 2004; Ersanlı, Odabaşoğlu *et al.*, 2004; Butcher *et al.*, 2005).Atom H1 was located on N1, confirming a preference for the keto-hydrazo tautomer in the solid state. There is a strong intramolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond in (I) (Table 2), which is a common feature of similar systems (Ersanlı *et al.*, 2003; Ersanlı, Albayrak *et al.*, 2004; Alpaslan *et al.*, 2005a,b).The  $\text{N}1-\text{N}2$  single bond of  $1.3057(17)$  Å is shorter than that observed in 1,2-bis-[1-(3-pyridyl)3-methyltriazen-3-

yl]ethane [1.326 (3) Å; Vaughan *et al.*, 2004]. This shortening in the hydrazone group is consistent with the resonance structures, which give the N1–N2 bond some double-bond character.

## Experimental

The title compound was prepared as described by Odabaşoğlu *et al.* (2005), using *p*-methylaniline and ethyl 4-chloroacetoacetate as starting materials (yield 84%, m.p. 368–370 K). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an absolute acetic acid solution at room temperature.

### Crystal data

$C_{13}H_{15}ClN_2O_3$	$Z = 4$
$M_r = 282.72$	$D_x = 1.341 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 7.2025 (4) \text{ \AA}$	$\mu = 0.28 \text{ mm}^{-1}$
$b = 19.3625 (14) \text{ \AA}$	$T = 296 (2) \text{ K}$
$c = 10.6093 (6) \text{ \AA}$	Prism, brown
$\beta = 108.852 (4)^\circ$	$0.63 \times 0.48 \times 0.23 \text{ mm}$
$V = 1400.19 (15) \text{ \AA}^3$	

### Data collection

Stoe IPDS-II diffractometer	24033 measured reflections
$\omega$ scans	2743 independent reflections
Absorption correction: integration ( <i>X-RED32</i> ; Stoe & Cie, 2002)	2060 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.869$ , $T_{\max} = 0.933$	$R_{\text{int}} = 0.053$
	$\theta_{\text{max}} = 26.0^\circ$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 0.0706P]$
$R[F^2 > 2\sigma(F^2)] = 0.034$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.097$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.16 \text{ e \AA}^{-3}$
2743 reflections	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$
178 parameters	Extinction correction: <i>SHELXL97</i>
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.0090 (17)

**Table 1**

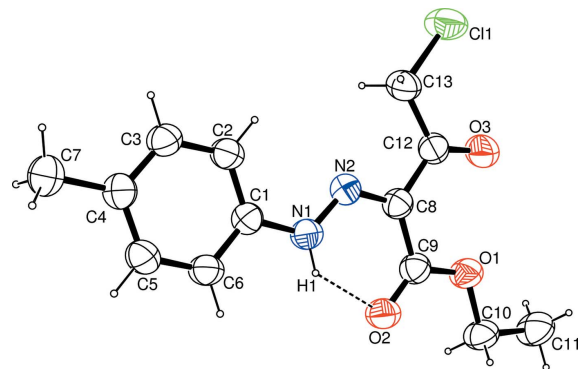
Selected geometric parameters (Å, °).

C1–N1	1.4045 (18)	C10–O1	1.4575 (18)
C8–N2	1.3135 (18)	C12–O3	1.2069 (18)
C9–O2	1.2175 (16)	C13–Cl1	1.7626 (16)
C9–O1	1.3225 (17)	N1–N2	1.3057 (17)
C6–C1–N1	118.08 (13)	O3–C12–C8	124.23 (14)
C2–C1–N1	122.32 (13)	C8–C12–C13	114.80 (12)
N2–C8–C12	113.13 (12)	C12–C13–Cl1	112.17 (11)
C9–C8–C12	124.55 (13)	N2–N1–C1	121.07 (12)
O2–C9–O1	122.77 (13)	N1–N2–C8	121.49 (12)
O1–C9–C8	114.95 (13)	C9–O1–C10	115.17 (12)
O1–C10–C11	106.73 (13)		

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1–H1 $\cdots$ O2	0.836 (18)	1.933 (18)	2.5762 (16)	133 (2)



**Figure 1**

The molecular structure of (I), showing the atom-numbering scheme and the intramolecular N–H $\cdots$ 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.

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  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$  or  $1.5 U_{\text{eq}}(\text{methyl C})$ .

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).

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS-II diffractometer (purchased under grant No. F279 of the University Research Fund).

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