

Gökhan Alpaslan,<sup>a</sup> Özgür  
Özdamar,<sup>b</sup> Mustafa  
Odabaşoğlu,<sup>b</sup> Orhan  
Büyükgüngör<sup>a</sup> and Ahmet  
Erdönmez<sup>a\*</sup><sup>a</sup>Department of Physics, Faculty of Arts & Science, Ondokuz Mayıs University, TR-55139 Kurupelit-Samsun, Turkey, and <sup>b</sup>Department of Chemistry, Faculty of Arts & Science, Ondokuz Mayıs University, TR-55139 Kurupelit-Samsun, Turkey

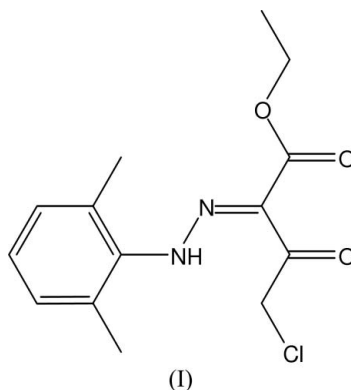
Correspondence e-mail: gokhana@omu.edu.tr

## Key indicators

Single-crystal X-ray study  
 $T = 296$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.044  
 $wR$  factor = 0.126  
Data-to-parameter ratio = 15.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**(E)-Ethyl 4-chloro-2-[2-(2,6-dimethylphenyl)hydrazono]-3-oxobutanoate**The title compound,  $\text{C}_{14}\text{H}_{17}\text{ClN}_2\text{O}_3$ , adopts a keto–hydrazo tautomeric form stabilized by an intramolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond. The molecule is roughly planar.Received 7 June 2006  
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## 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.

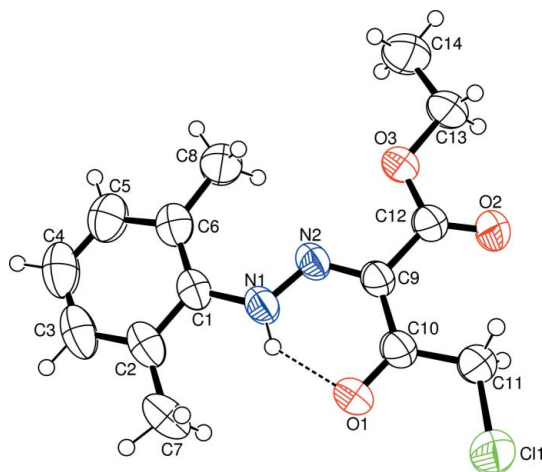


The molecular structure and atom-labelling scheme are shown in Fig. 1. The whole molecule is roughly planar (r.m.s. deviation = 0.075 Å), the largest deviation from the mean plane being 0.2075 (13) Å for atom Cl1. Bond lengths and angles in (I) show normal values and are comparable with those observed in ethyl 4-chloro-3-oxo-2-(phenylhydrazono)butyrate, (II) (Alpaslan *et al.*, 2005a), (*E*)-ethyl 4-chloro-3-[2-(2-fluorophenyl)hydrazono]butanoate, (III) (Alpaslan *et al.*, 2005b) and ethyl 4-chloro-2-[(2-nitrophenyl)hydrazono]-3-oxobutyrate (IV) (Odabaşoğlu *et al.*, 2005).

Our investigations show that, in the solid state, the molecular structure of (I) adopts the keto–hydrazo tautomeric form with an intramolecular hydrogen bond (Fig. 1). The bond lengths (Table 1) show that there is significant elongation of the N1–N2 bond and contraction of the C1–N1 bond in comparison with azo compounds. For example, the N=N and C–N bonds in azobenzene are 1.249 (4) and 1.431 (4) Å, respectively (Harada *et al.*, 1997).

The N1–N2 single bond of 1.293 (3) Å is shorter than that observed in 1,2-bis[1-(3-pyridyl)-3-methyltriazene-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.

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, 2004; Alpaslan *et al.*, 2005a,b).



**Figure 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.

## Experimental

The title compound was prepared as described by Odabaşoğlu *et al.* (2005), using *o*-methylaniline and ethyl 4-chloroacetate as starting materials (yield 90%, m.p. 397–399 K). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an absolute ethanol solution at room temperature.

### Crystal data

$C_{14}H_{17}ClN_2O_3$   
 $M_r = 296.75$   
 Triclinic,  $P\bar{1}$   
 $a = 7.0423$  (7) Å  
 $b = 9.3029$  (9) Å  
 $c = 11.8285$  (11) Å  
 $\alpha = 93.707$  (8)°  
 $\beta = 100.515$  (8)°  
 $\gamma = 103.75$  (8)°

$V = 735.3$  (3) Å<sup>3</sup>  
 $Z = 2$   
 $D_x = 1.340$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 $\mu = 0.27$  mm<sup>-1</sup>  
 $T = 296$  (2) K  
 Prismatic rod, yellow  
 $0.68 \times 0.36 \times 0.20$  mm

### Data collection

Stoe IPDS-II diffractometer  
 $\omega$  scans  
 Absorption correction: integration  
 (*X-RED32*; Stoe & Cie, 2002)  
 $T_{\min} = 0.880$ ,  $T_{\max} = 0.952$

12526 measured reflections  
 2892 independent reflections  
 2019 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.046$   
 $\theta_{\text{max}} = 26.0^\circ$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.044$   
 $wR(F^2) = 0.126$   
 $S = 1.04$   
 2892 reflections  
 187 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.072P)^2 + 0.0348P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.24$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.22$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

C1—N1	1.402 (2)	C12—O2	1.204 (2)
C9—N2	1.319 (2)	C12—O3	1.322 (2)
C10—O1	1.226 (2)	C13—O3	1.452 (2)
C11—C11	1.762 (2)	N1—N2	1.293 (2)
N1—C1—C2	114.95 (19)	O2—C12—O3	122.89 (18)
N1—C1—C6	123.46 (17)	O2—C12—C9	124.41 (18)
N2—C9—C10	123.70 (16)	O3—C12—C9	112.69 (16)
N2—C9—C12	114.52 (17)	O3—C13—C14	107.81 (19)
O1—C10—C9	120.40 (18)	N2—N1—C1	123.73 (18)
O1—C10—C11	120.33 (19)	N1—N2—C9	121.41 (17)
C10—C11—C11	112.55 (14)	C12—O3—C13	115.88 (16)

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1...O1	0.86 (3)	1.86 (2)	2.575 (2)	138 (2)

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

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).

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