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Onur Şahin,^{a*} Orhan
Büyükgüngör,^a Selami Şaşmaz,^b
Nurhan Gümrükçüoğlu^c and
Cihan Kantar^b

^aDepartment of Physics, Ondokuz Mayıs
University, TR-55139 Samsun, Turkey,

^bDepartment of Chemistry, Rize University,
Turkey, and ^cDepartment of Chemistry, Giresun
University, Turkey

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

Key indicators

Single-crystal X-ray study

$T = 296$ K

Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å

Disorder in main residue

R factor = 0.045

wR factor = 0.120

Data-to-parameter ratio = 16.1

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

2'-[Ethoxy(*p*-tolyl)methylene]acetohydrazide

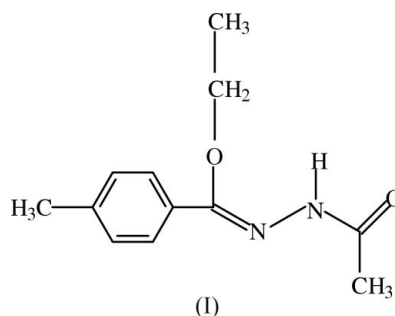
Molecules of the title compound, $\text{C}_{12}\text{H}_{16}\text{N}_2\text{O}_2$, are linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds [$\text{N}\cdots\text{O} = 2.971(2)$ Å, $\text{H}\cdots\text{O} = 2.17(2)$ Å and $\text{N}-\text{H}\cdots\text{O} = 162(2)^\circ$] into cyclic centrosymmetric $R_2^2(8)$ dimers.

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Comment

Hydrazone is formed when hydrazines condense with aldehydes and ketones, *e.g.* by the condensation of aldehydes (or substituted aldehydes) with phenylhydrazine at a pH of 4–5 (McMurry, 1999), and typically are crystalline compounds with sharp melting points. Hydrazone is frequently more efficient than oximes in this reaction since their greater molecular weight causes a lower solubility in most solvents and they can, therefore, often be more easily isolated and recrystallized. Hydrazone has been widely studied as chelating ligands for the spectrophotometric and fluorimetric determination of trace metal ions (Katyal & Dutt, 1975; Galiano-Roth & Collum, 1988). Many of these compounds have found widespread application in medicine, technology and analytical chemistry (Kitaev, 1977). In addition to the X-ray crystal structure determination reported here, the title compound, (I), has also been characterized by IR, ^1H NMR and ^{13}C NMR spectroscopy and by elemental analysis.



The molecular structure and atom-numbering scheme are shown in Fig. 1. The dihedral angle between the aromatic C1–C6 ring and the plane of the C8/N1/N2/C11/C12/O2 chain is $12.7(1)^\circ$. The plane of the C8/O1/C9/C10 chain forms a dihedral angle of $84.6(1)^\circ$ with the C1–C6 ring. The N1–N2 and C11=O2 bond lengths are in agreement with those reported for 2,4-difluorobenzaldehyde isonicotinoylhydrazone and 2,3-dichlorobenzaldehyde isonicotinoylhydrazone (Wardell *et al.*, 2005).

The molecules are linked by a pair of $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 2) into a centrosymmetric dimer with an $R_2^2(8)$ ring (Bernstein *et al.*, 1995) (Fig. 2).

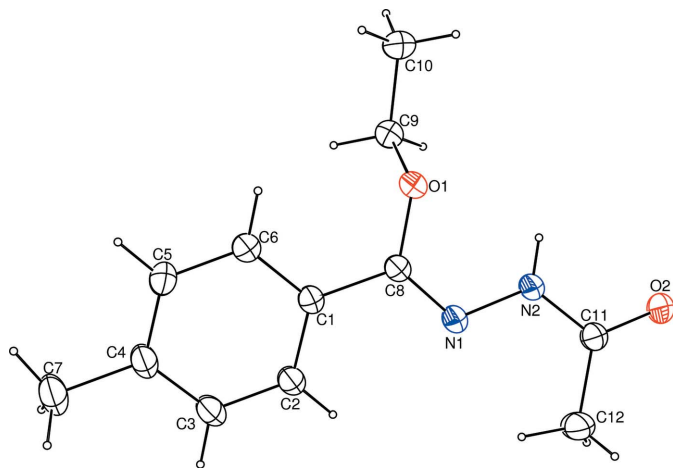


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level.

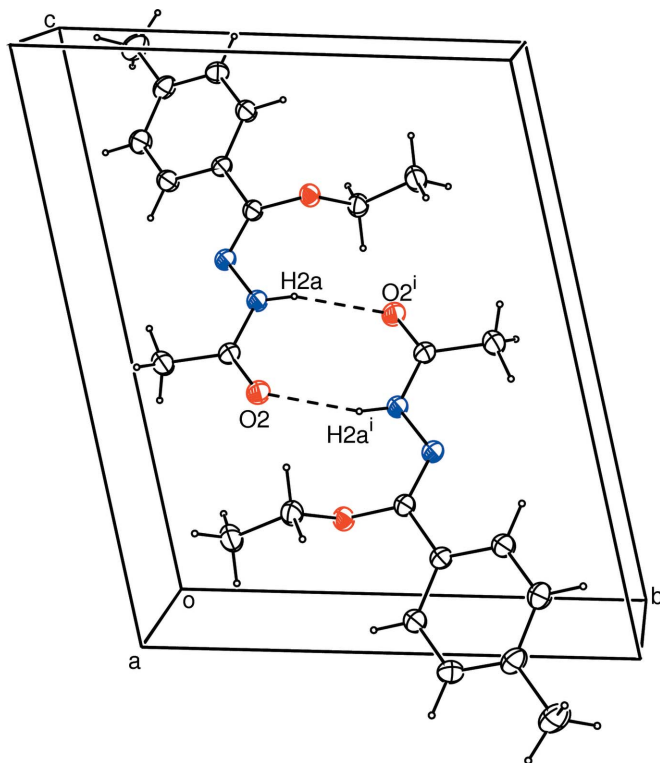


Figure 2
Part of the crystal structure of (I), showing a centrosymmetric $R_2^2(8)$ dimer; dashed lines indicate hydrogen bonds. [Symmetry code: (i) $2 - x, 1 - y, 1 - z$.]

Experimental

A solution of acylhydrazine (0.01 mol) in absolute ethanol (25 ml) was added to a solution of (*p*-methylphenylimido)acetate hydrochloride (0.01 mol) in absolute ethanol (25 ml). The mixture was stirred at 273–278 K for 6 h and subsequently at room temperature for 2 h. The reaction mixture was poured into a beaker containing cold water (40 ml) and ice (10 g). The precipitate formed was washed

with 50 ml of ice water and dried. The product was recrystallized from benzene–petroleum ether (1:2) (313–333 K) to give a white product (yield 50%). Single crystals of (I) were obtained by slow evaporation of an ethyl acetate solution at room temperature (m.p. 375–376 K). IR (KBr, cm^{-1}): 3205 (ν_{NH}), 1669 ($\nu_{\text{C=O}}$), 1618 ($\nu_{\text{C=N}}$); Analysis calculated for $\text{C}_{12}\text{H}_{16}\text{N}_2\text{O}_2$: C 65.43, H 7.32, N 12.72%; found: C 65.10, H 7.12, N 12.45%.

Crystal data

$\text{C}_{12}\text{H}_{16}\text{N}_2\text{O}_2$	$\gamma = 100.950 (8)^\circ$
$M_r = 220.27$	$V = 609.98 (11) \text{ \AA}^3$
Triclinic, $P\bar{1}$	$Z = 2$
$a = 4.6555 (5) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 10.6294 (10) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$c = 13.0612 (13) \text{ \AA}$	$T = 296 \text{ K}$
$\alpha = 103.947 (8)^\circ$	$0.50 \times 0.27 \times 0.09 \text{ mm}$
$\beta = 94.907 (8)^\circ$	

Data collection

Stoe IPDS-II diffractometer	9116 measured reflections
Absorption correction: integration (<i>X-RED32</i> ; Stoe & Cie, 2002)	2401 independent reflections
$T_{\text{min}} = 0.964, T_{\text{max}} = 0.994$	1610 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.050$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.121$	$\Delta\rho_{\text{max}} = 0.14 \text{ e \AA}^{-3}$
$S = 1.02$	$\Delta\rho_{\text{min}} = -0.18 \text{ e \AA}^{-3}$
2401 reflections	
149 parameters	

Table 1

Selected bond lengths (\AA).

C8–O1	1.3676 (19)	C11–N2	1.342 (2)
C9–O1	1.449 (2)	N1–N2	1.3750 (18)
C11–O2	1.222 (2)		

Table 2

Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
$\text{N2---H2a}\cdots\text{O2}^i$	0.83 (2)	2.17 (2)	2.971 (2)	162 (2)

Symmetry code: (i) $-x + 2, -y + 1, -z + 1$.

The H atom bonded to N2 was refined freely. All other H atoms were placed in calculated positions and constrained to ride on their parent atoms, with $\text{C---H} = 0.93\text{--}0.97 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ [$1.5U_{\text{eq}}(\text{disordered methyl})$]. The H atoms of the C7 and C12 methyl groups are disordered over two sites with equal occupancy.

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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