2′-[Eth-oxy(phen-yl)methyl-ene]acetohydrazide

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2′-[Ethoxy(phenyl)methylene]acetohydrazide

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Key indicators

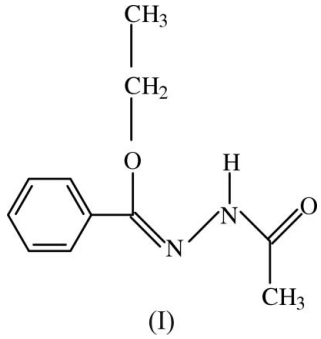
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.103
Data-to-parameter ratio = 15.5

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

The title compound, $C_{11}H_{14}N_2O_2$, contains two independent molecules in the asymmetric unit. Each independent molecule exists as part of an N—H···O hydrogen-bonded centrosymmetric dimer.

Comment

The chemistry of hydrazones has been intensively investigated in recent years, owing to their coordinating capability, pharmacological activity, and antibacterial and antifungal properties, and to their use in analytical chemistry as highly selective extractants (Domiano *et al.*, 1984; Li *et al.*, 1988; Sakamoto *et al.*, 1993). In addition to the X-ray crystal structure determination reported here, the title compound, (I), has also been characterized by IR, ^{1}H NMR and ^{13}C NMR spectroscopy and elemental analysis.



(I)

The asymmetric unit of (I) contains two crystallographically independent molecules (Fig. 1). The corresponding bond lengths (Table 1) of the two molecules agree with each other and are comparable to those of 2′-[ethoxy(*p*-tolyl)methylene]acetohydrazide (Şahin *et al.*, 2007). The torsion angles describing the conformation of the side chains are listed in Table 1.

In the crystal structure of (I), each of the two independent molecules exists as an N—H···O hydrogen-bonded centrosymmetric dimer with an $R_2^2(8)$ ring (Bernstein *et al.*, 1995) (Fig. 2). The geometric parameters of the hydrogen bonds are given in Table 2.

Experimental

A solution of acylhydrazine (0.01 mol) in absolute ethanol (25 ml) was added to a solution of iminoester (*p*-phenylimido)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 40 ml of cold water and 10 g of ice. The precipitate formed

was washed with ice–water (50 ml) and dried. The product was recrystallized from benzene–petroleum ether (1:2) (313–333 K) to give a white product (yield 45%). Single crystals of (I) were obtained by slow evaporation of an ethyl acetate solution at room temperature (m.p. 367–368 K). Analysis calculated for $C_{11}H_{14}N_2O_2$: C 64.06, H 6.84, N 13.58%; found: C 64.00, H 6.77, N 13.45%.

Crystal data

$C_{11}H_{14}N_2O_2$
M_r = 206.24
Monoclinic, $C2/c$
a = 49.228 (3) Å
b = 11.0095 (10) Å
c = 8.4680 (5) Å
β = 99.170 (5)°
V = 4530.8 (6) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 K
0.49 × 0.27 × 0.09 mm

Data collection

Stoe IPDS-II diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.977, T_{max} = 0.993
15347 measured reflections
4373 independent reflections
2164 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.103
S = 0.81
4373 reflections
282 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C7—N1	1.268 (2)	C19—O3	1.4421 (19)
C7—O1	1.3670 (19)	C21—O4	1.2275 (19)
C8—O1	1.437 (2)	C21—N4	1.344 (2)
C10—O2	1.229 (2)	N1—N2	1.3772 (19)
C10—N2	1.340 (2)	N3—N4	1.3823 (18)
C18—O3	1.3626 (18)		
C2—C1—C7—N1	−18.5 (3)	C7—N1—N2—C10	176.94 (18)
C2—C1—C7—O1	167.74 (16)	O3—C18—N3—N4	−4.6 (2)
C17—C12—C18—N3	147.41 (17)	C12—C18—N3—N4	−175.42 (14)
C17—C12—C18—O3	−23.7 (2)	C18—N3—N4—C21	169.63 (16)
O1—C7—N1—N2	−4.7 (3)	C1—C7—O1—C8	−74.4 (2)
C1—C7—N1—N2	−178.27 (15)	C12—C18—O3—C19	−67.1 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O2^{i}	0.90 (2)	2.03 (2)	2.912 (2)	170 (2)
N4—H4A···O4^{ii}	0.90 (2)	2.03 (2)	2.918 (2)	169 (2)

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

The imine H atoms were located in a difference map and refined freely (distances given in Table 2). All other H atoms were placed in calculated positions and constrained to ride on their parent atoms, with C—H = 0.93–0.97Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(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) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

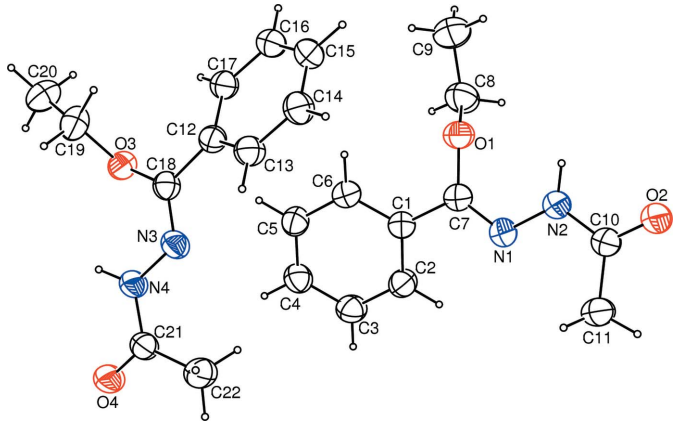


Figure 1
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 40% probability level.

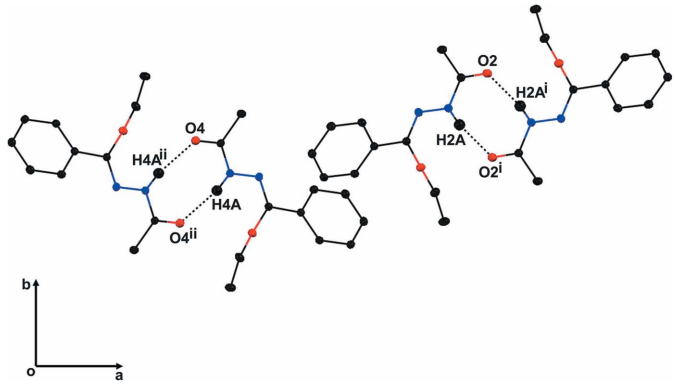


Figure 2
A view of centrosymmetric $R_2^2(8)$ dimers in (I). For the sake of clarity, H atoms bonded to C atoms have been omitted. [Symmetry codes: (i) $1-x$, $1-y$, $1-z$; (ii) $\frac{1}{2}-x$, $\frac{1}{2}-y$, $1-z$.]

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