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Diethyl (*Z*,*Z*)-3,3′-(ethane-1,2-diyldiimino)dibut-2-enoate

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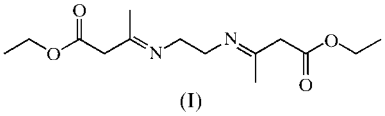
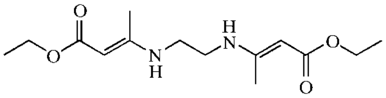
The title compound, $C_{14}H_{24}N_2O_4$, is an intermediate in the synthesis of the corresponding heterocyclic compounds. The molecule lies about a twofold axis and has a 'twist' conformation; it adopts the *Z*,*Z* configuration, which is shown to be governed mainly by intramolecular hydrogen bonds.

Comment

The condensation reaction of ethylenediamine with ethyl acetoacetate, catalyzed by various Friedel–Crafts-type catalysts, gives the open-chain compound (I) (Moazzam *et al.*, 1988). However, we found that the reaction occurs readily without any catalyst and yields a crystalline product that was originally thought to have the structure of (I). However, ^{1}H NMR suggested that there are two vinyl H atoms (δ = 4.43 p.p.m., *s*) and two H atoms on the N atoms (δ = 8.59 p.p.m., *s*), and that the structure is in fact (II) (Costes & Laurent, 1988).



(I)



(II)

Although structure (II) is strongly supported by ^{1}H NMR spectroscopy, there are still some questions regarding its conformation and configuration. In addition to verifying structure (II), X-ray analysis of the compound has now been undertaken and the results are presented here (Fig. 1).

The ethylenediamine structural unit in this molecule adopts a low-energy *gauche* conformation with respect to the central C7—C7*A* bond, which is very similar to 1,2-ethanediamine (Burgess *et al.*, 1976; Marstokk & Møllendal, 1978). The molecule adopts a 'twist' conformation, with an N1—C7—C7*A*—N1$^{\rm i}$ torsion angle of 66°, and the C4=C5 bond is in the *Z*,*Z* configuration [symmetry code: (i) $-x, y, -z + \frac{1}{2}$].

The N1—C5 bond length of 1.342 (4) Å is shorter than the standard N—C experimental bond length of 1.469 Å (Lice, 1957). This large difference is considered to be the result of π–π conjugation between the N atom and the C=C bond. Conversely, the C3=O2 bond [1.222 (4) Å] is longer than that in other derivative compounds [*e.g.* 1.199 (3) Å; Balsamini *et al.*, 1992]. The O2—C3—C4—C5 torsion angle is −1.7°, indicating that the π–π conjugation is spread between the C=C bond and the C=O (ester) bond.

Another interesting characteristic of (II) is its *Z*,*Z* configuration, which is probably caused and stabilized by intramolecular N—H⋯O=C hydrogen bonds (Fig. 2 and Table 2) (Jeffrey, 1997).

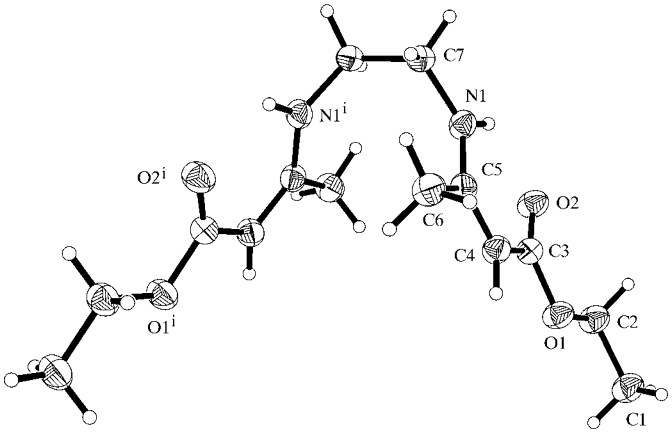


Figure 1
The molecular structure of (II), showing the atom-labelling scheme and 40% probability displacement ellipsoids. H atoms are shown as small spheres of arbitrary radii. [Symmetry code: (i) $-x, y, -z + \frac{1}{2}$.]

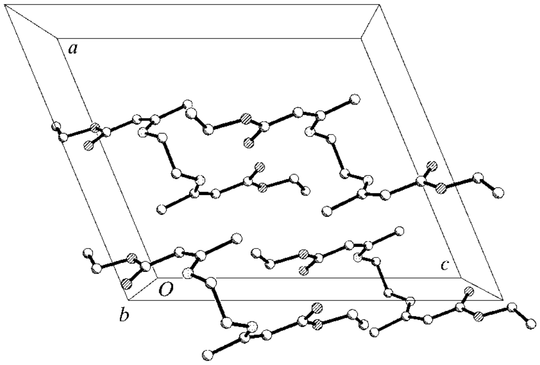


Figure 2
A packing diagram for (II), viewed along the *b* axis.

Experimental

Diethyl (*Z*,*Z*)-3,3′-(ethane-1,2-diyldinitrilo)dibut-2-enoate was prepared by the condensation of 1,2-diaminoethane and ethyl acetoacetate (molar ratio 1:2) in alcohol at room temperature. The product was recrystallized from dichloromethane (yield 92%, m.p. 399–

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401 K). Spectroscopic analysis, ^{1}H NMR (300 MHz, CDCl$_3$, δ): 8.59 (*s*, 2H, NH), 4.43 (*s*, 2H, =CH), 4.02 (*q*, J = 15 Hz, 4H, ethyl CH$_2$), 3.31 (*d*, J = 6 Hz, 4H, ethylene CH$_2$), 1.86 (*s*, 6H, CH$_3$), 1.19 (*t*, J = 15 Hz, 6H, ethyl CH$_3$).

Crystal data

$C_{14}H_{24}N_2O_4$
$M_r = 284.35$
Monoclinic, $C2/c$
$a = 11.505\ (2)$ Å
$b = 10.540\ (2)$ Å
$c = 13.471\ (3)$ Å
$\beta = 112.66\ (3)^\circ$
$V = 1507.4\ (6)$ Å^3
$Z = 4$
$D_x = 1.253$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1735 reflections
$\theta = 2.7$–25.8°
$\mu = 0.09$ mm^{-1}
$T = 293\ (2)$ K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.973$, $T_{\max} = 0.982$
3026 measured reflections
1325 independent reflections
1147 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.032$
$\theta_{\max} = 25.0^\circ$
$h = -10 \rightarrow 13$
$k = -12 \rightarrow 11$
$l = -15 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.076$
$wR(F^2) = 0.206$
$S = 1.07$
1325 reflections
91 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0925P)^2 + 4.2899P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.50$ e Å^{-3}
$\Delta\rho_{\min} = -0.49$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.362 (4)	C3—C4	1.439 (4)
O2—C3	1.222 (4)	C4—C5	1.370 (4)
N1—C5	1.342 (4)		
C5—N1—C7	126.2 (3)	N1—C5—C4	122.9 (3)
O2—C3—C4	127.4 (3)	N1—C5—C6	118.5 (3)
O1—C3—C4	110.7 (3)	C4—C5—C6	118.5 (3)
C5—C4—C3	123.7 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2	0.86	2.14	2.787 (3)	132

H atoms were constrained to their parent atoms using a riding model, with C—H distances in the range 0.93–0.96 Å and N—H distances of 0.86 Å, and with $U_{\rm iso}$(H) = 1.2 or 1.5 times $U_{\rm eq}$ of the parent atom.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TY1002). Services for accessing these data are described at the back of the journal.

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