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(1*R*,2*S*)-Benzyl *N*-[3-(diisopropylaminocarbonyl)-2-phenylprop-3-enyl]carbamate

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Abstract

The title compound, C₂₅H₃₂N₂O₃, was synthesized as part of a series of related compounds using a modified Eschenmoser–Claisen rearrangement reaction. The compound is racemic and the structure features a centrosymmetric hydrogen-bonded dimerization along with some aromatic stacking stabilization.

Keywords

Single-crystal X-ray study; *T* = 293 K; Mean $\sigma(\text{C}–\text{C}) = 0.004 \text{ \AA}$; *R* factor = 0.049; *wR* factor = 0.143; Data-to-parameter ratio = 15.8

Comment

We recently reported a new synthetic procedure, based upon a modification of the Eschenmoser–Claisen rearrangement reaction, to provide a convenient route to anti- β -substituted γ,δ -unsaturated amino acids (Qu et al., 2006). As part of the study the crystal structure of one of the derivatives, (I), was determined, and is reported here.

The title compound, (I), is racemic and crystallizes with one molecule in the asymmetric unit (Fig. 1). The molecule has an extended conformation and the molecular dimensions are generally unexceptional. The structure shows unambiguously that there is an anti relationship between the α -amino group and the β -phenyl group. Crystal packing in this compound is determined by a single $\text{N}–\text{H}\cdots\text{O}$ hydrogen bond which, because of the crystallographic inversion centre, generates a discrete $R_2^2(10)$ hydrogen-bonded dimer (Bernstein et al., 1995). Aromatic edge-to-face stacking interactions play a secondary stabilization role in this structure, as indicated in Fig. 2.

Experimental

The synthesis of this compound was reported recently (Qu et al., 2006).

Crystal data

$C_{25}H_{32}N_2O_3$	$V = 1183.0 (2) \text{ \AA}^3$
$M_r = 408.53$	$Z = 2$
	$D_x = 1.147 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	
$a = 10.3807 (12) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 11.2172 (14) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$c = 11.7700 (14) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\alpha = 112.085 (7)^\circ$	Block, colourless
$\beta = 108.206 (7)^\circ$	$0.40 \times 0.30 \times 0.20 \text{ mm}$
$\gamma = 93.159(7)^\circ$	

Data collection

Bruker APEXII CCD	32705 measured reflections
diffractometer	4406 independent reflections
ω and ϕ scans	3298 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan	$R_{\text{int}} = 0.032$
(SADABS; Sheldrick, 2004)	$\theta_{\text{max}} = 25.5^\circ$
$T_{\text{min}} = 0.891, T_{\text{max}} = 0.985$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0645P)^2 + 0.2666P]$
$R[F^2 > 2\sigma(F^2)] = 0.049$	
$wR(F^2) = 0.143$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\text{max}} < 0.001$
4406 reflections	$\Delta\rho_{\text{max}} = 0.29 \text{ e \AA}^{-3}$
279 parameters	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$
H atoms treated by a mixture of independent and constrained refinement	

All H atoms were initially located in a difference Fourier map. With the exception of the amino H atom, which was freely refined, H atoms were refined as follows: methyl H atoms were refined as a rotating group with C–H = 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$; methylene H atoms with C–H = 0.97 Å; CH H atoms with C–H = 0.98 Å; and aromatic H atoms with C–H = 0.95 Å. All were refined with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *APEXII* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *ORTEP-3* (Farrugia, 1997) and *MERCURY* (Version 1.4; Macrae et al., 2006); software used to prepare material for publication: *SHELXTL*, *pubCIF* (Westrip, 2006) and local programs.

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Figure 1.
The molecular structure of (I), with displacement ellipsoids at the 30% probability level. H atoms have been omitted for clarity.

Figure 2.

A *b*-axis projection of the crystal packing of (I). C–H H atoms have been omitted. Hydrogen bonding is indicated by dashed lines; a solid line also indicates the type of edge–face aromatic stacking interaction found in this structure. The distance H24...Cgⁱ is 2.74 Å [*Cg* is the centroid of atoms C1–C6; symmetry code: (i) 1 – *x*, –*y*, –*z*].

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> – <i>H</i> ⋯ <i>A</i>	<i>D</i> – <i>H</i>	<i>H</i> ⋯ <i>A</i>	<i>D</i> ⋯ <i>A</i>	<i>D</i> – <i>H</i> ⋯ <i>A</i>
N1–H1N⋯O3 ⁱ	0.82 (2)	2.14 (2)	2.9470 (19)	169.1 (19)

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