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

Hongchang Qu, Michael D. Carducci, Gary S. Nichol* and Victor J. Hruby

Department of Chemistry, The University of Arizona, 1306 E University Boulevard, Tucson, AZ 85721, USA

Correspondence e-mail: gsnichol@email.arizona.edu

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The title compound, $C_{25}H_{32}N_2O_3$, 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.

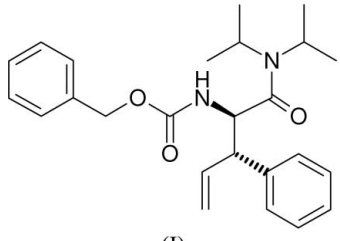
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.049
wR factor = 0.143
Data-to-parameter ratio = 15.8

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

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.



(I)

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

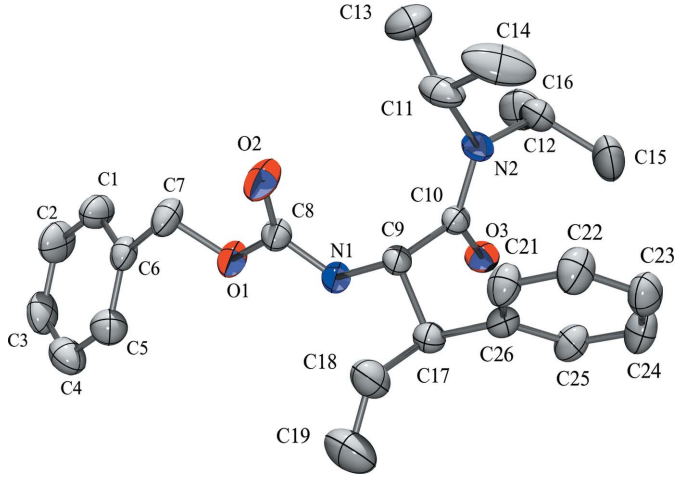


Figure 1
The molecular structure of (I), with displacement ellipsoids at the 30% probability level. H atoms have been omitted for clarity.

and the β-phenyl group. Crystal packing in this compound is determined by a single N—H···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

$C_{25}H_{32}N_2O_3$
M_r = 408.53
Triclinic, $P\overline{1}$
a = 10.3807 (12) Å
b = 11.2172 (14) Å
c = 11.7700 (14) Å
α = 112.085 (7)°
β = 108.206 (7)°
γ = 93.159 (7)°
V = 1183.0 (2) Å^3
Z = 2
D_x = 1.147 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 (2) K
Block, colourless
0.40 × 0.30 × 0.20 mm

Data collection

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

Refinement

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

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O3^{i}	0.82 (2)	2.14 (2)	2.9470 (19)	169.1 (19)

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

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_{iso}(H) = 1.5U_{eq}(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_{iso}(H) = 1.2U_{eq}(C).

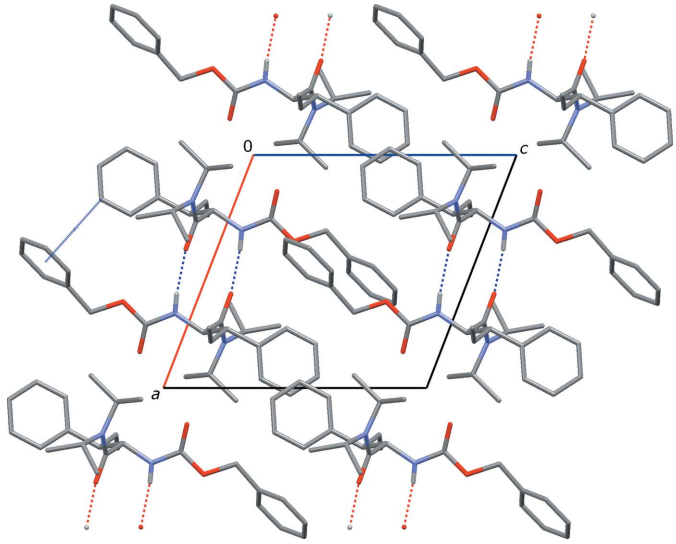


Figure 2
A *b*-axis projection of the crystal packing of (I). C—H H atoms have been omitted. Hydrogen bonding is indicated by dashed lines; a solid line also indicates the type of edge–face aromatic stacking interaction found in this structure. The distance H24···Cg^{i} is 2.74 Å [Cg is the centroid of atoms C1–C6; symmetry code: (i) $1-x, -y, -z$].

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*, publCIF (Westrip, 2006) and local programs.

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