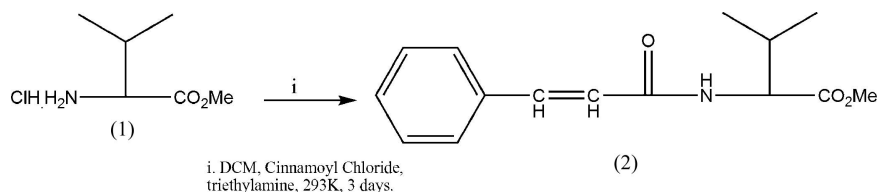


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Key indicators

Single-crystal X-ray study
 $T = 295\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.008\text{ \AA}$
 R factor = 0.048
 wR factor = 0.160
Data-to-parameter ratio = 8.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.*N*-Cinnamoyl-L-valine methyl esterThe crystal structure of the title compound, $\text{C}_{15}\text{H}_{19}\text{NO}_3$, shows that the overall molecular conformation, which is approximately planar, is stabilized by intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen-bonding interactions.Received 21 November 2006
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Comment

We have recently demonstrated the cross-metathesis reaction of derivatized amino acids to generate dynamic combinatorial libraries (Poulsen & Bornaghi, 2006) using the Grubbs carbene ruthenium complex as catalyst (Grubbs & Chang, 1998, 2004). This dynamic combinatorial library was targeted towards the enzyme carbonic anhydrase II, and proved a useful way to generate and identify potent enzyme inhibitors. As part of this investigation, we synthesized a range of *N*-substituted amino acids as library building blocks (Poulsen *et al.*, 2003; Bornaghi *et al.*, 2004). In the present communication, we report the structure of one of these building blocks, *N*-cinnamoyl-L-valine methyl ester, (2).The molecular structure of (2) is shown in Fig. 1. The bond lengths and angles for (2) are in accord with conventional values (Allen *et al.*, 1987). The cinnamoylmethyl ester portion of the molecule is approximately planar, with torsion angles $\text{C}6-\text{C}1-\text{C}7-\text{C}8 = -21.0(7)^\circ$, $\text{C}7-\text{C}8-\text{C}9-\text{O}1 = -16.4(7)^\circ$, $\text{C}10-\text{N}1-\text{C}9-\text{O}1 = 2.3(8)^\circ$ and $\text{N}1-\text{C}10-\text{C}14-\text{O}2 = -12.4(6)^\circ$. The isopropyl group lies above this plane. The crystal structure is stabilized by intermolecular $\text{N}-\text{H}\cdots\text{O}$ interactions (Fig. 2), together with a number of weaker intra- and intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions (Table 1).

Experimental

Triethylamine (5.41 g, 53 mmol) was added dropwise to a solution of L-valine methyl ester hydrochloride, (1) (1 g, 5.96 mmol), and cinnamoyl chloride (994 mg, 5.96 mmol) in anhydrous dichloromethane (100 ml). The reaction mixture was stirred at room temperature (298 K) for 3 d before being washed with 2 M HCl ($2 \times 100\text{ ml}$) and saturated brine solution (100 ml), then dried over MgSO_4 . The volatiles were removed under reduced pressure to give a clear solid residue (1.9 g). The title compound, (2), was obtained in 94% yield after crystallization using a 1:4 mixture of ethyl acetate and hexane. $^1\text{H NMR}$ (CDCl_3 , 300 MHz): δ 0.98 (d, 3H, $J = 6.9\text{ Hz}$, δCH_3),

1.01 (*d*, 3H, $J = 6.9$ Hz, $\delta^{\text{C}}\text{CH}_3$), 2.20–2.31 (*m*, 1H, βCH), 3.79 (*s*, 3H, OCH_3), 4.75 (*t*, 1H, $J = 7.3$ Hz, αCH), 6.12 (*br d*, 1H, NH), 6.49 (*d*, 1H, $J = 15.6$ Hz, $=\text{CHCO}$), 7.38–7.43 (*m*, 3H, ArH), 7.52–7.56 (*m*, 2H, ArH), 7.67 (*d*, 1H, $J = 15.6$ Hz, $=\text{CHPh}$); ^{13}C NMR (CDCl_3 , 75 MHz): δ 17.9 and 19.0 (δCH_3), 31.6 (βCH), 52.3 (OCH_3), 57.1 (αCH), 120.1 ($=\text{CHCO}$), 127.9, 128.8 and 129.8 (CH from Ar), 135.6 (C from Ar), 141.8 ($=\text{CHPh}$), 167.4 (CONH), 171.5 (COOCH_3); MS (LRMSES): m/z 262.3 $[\text{M}+\text{H}]^+$, 284.3 $[\text{M}+\text{Na}]^+$; m.p. 362 K.

Crystal data

$\text{C}_{15}\text{H}_{19}\text{NO}_3$
 $M_r = 261.31$
 Orthorhombic, $P2_12_12_1$
 $a = 9.979$ (2) Å
 $b = 17.899$ (3) Å
 $c = 8.2712$ (14) Å
 $V = 1477.4$ (5) Å³

$Z = 4$
 $D_x = 1.175$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 295$ K
 Prism, colorless
 $0.35 \times 0.35 \times 0.25$ mm

Data collection

Rigaku AFC-7R diffractometer
 ω - 2θ scans
 Absorption correction: none
 1950 measured reflections
 1510 independent reflections
 871 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$
 $\theta_{\text{max}} = 25.0^\circ$
 3 standard reflections
 every 150 reflections
 intensity decay: 0.3%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.160$
 $S = 1.01$
 1510 reflections
 172 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0838P)^2 + 0.1628P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.16$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.24$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O1 ⁱ	0.96	2.22	3.136 (4)	158
C7—H7...O1	0.95	2.50	2.837 (6)	101
C7—H7...O2 ⁱⁱ	0.95	2.53	3.422 (5)	158
C8—H8...O1 ⁱ	0.96	2.58	3.358 (5)	138
C10—H10...O1	0.94	2.37	2.781 (6)	106
C12—H12C...O1	0.96	2.53	3.361 (9)	145
C12—H12C...N1	0.96	2.47	2.940 (9)	110

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

The carbon-bound H atoms were constrained as riding atoms, with $\text{C—H} = 0.94\text{--}0.96$ Å. $U_{\text{iso}}(\text{H})$ values were set at $1.2U_{\text{eq}}$ of the parent atom. In the absence of significant anomalous scattering effects, 102 Friedel pairs were merged. The absolute configuration of (2) was assigned on the basis of the known configuration of the starting material.

Data collection: *MSC/AFC7 Diffractometer Control Software* (Molecular Structure Corporation, 1999); cell refinement: *MSC/AFC7 Diffractometer Control Software*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 2001); program(s) used to solve structure: *TEXSAN for Windows*; program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *TEXSAN for Windows* and *PLATON*.

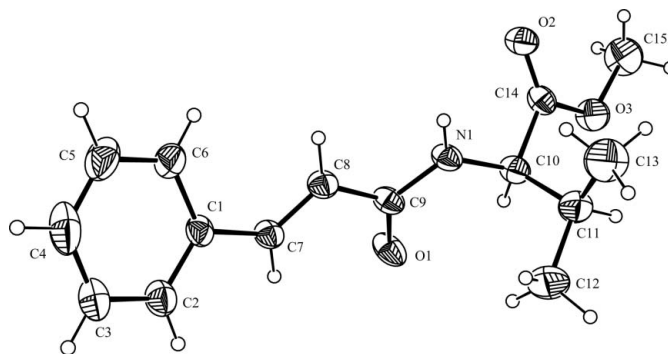


Figure 1

The molecular structure of (2), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

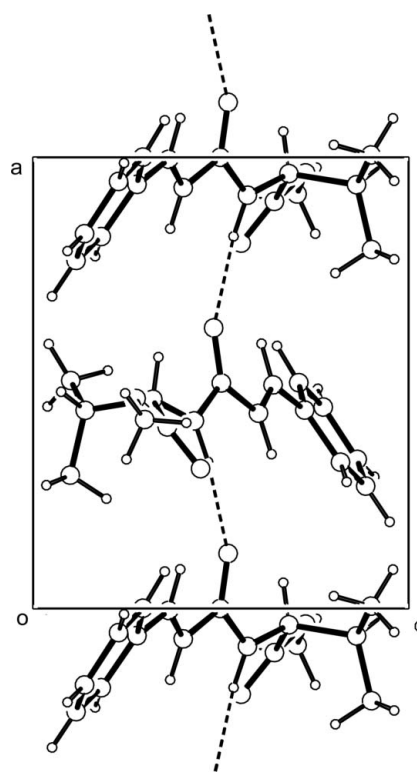


Figure 2

A view of the crystal packing projected on to the *ac* plane. Hydrogen bonds are shown as dashed lines.

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