The crystal structure of the title compound, C_{15}H_{19}NO_{3}, shows that the overall molecular conformation, which is approximately planar, is stabilized by intermolecular N—H⋯O hydrogen-bonding interactions.

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 C6—C1—C7—C8 = −21.0 (7)°, C7—C8—C9—O1 = −16.4 (7)°, C10—N1—C9—O1 = 2.3 (8)° and N1—C10—C14—O2 = −12.4 (6)°. The isopropyl group lies above this plane. The crystal structure is stabilized by intermolecular N—H⋯O interactions (Fig. 2), together with a number of weaker intra- and intermolecular C—H⋯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 × 100 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 1:4 mixture of ethyl acetate and hexane. 1H NMR (CDCl_{3}, 300 MHz): δ 0.98 (d, 3H, J = 6.9 Hz, δCH_{3}),
1.01 (d, 3H, J = 6.9 Hz, 8CH3), 2.20–2.31 (m, 1H, βCH), 3.79 (s, 3H, OCH3), 4.75 (t, 1H, J = 7.3 Hz, αCH), 6.12 (br d, 1H, NH), 6.49 (d, 1H, J = 15.6 Hz, CHCO), 7.38–7.43 (m, 3H, ArH), 7.52–7.56 (m, 2H, ArH), 7.67 (d, 1H, J = 15.6 Hz, CHPh); 13C NMR (CDCl3, CHPh), 167.4 (CONH), 171.5 (COOCH3); MS (LRMSES): m/z 262.3 [M+H]+, 284.3 [M+Na]+; m.p. 362 K.

Crystal data

C13H19NO3
M_r = 261.31
Orthorhombic, P212121
a = 9.979 (2) Å
b = 17.899 (3) Å
c = 8.2712 (14) Å
V = 1477.4 (5) Å³
Z = 4
D_g = 1.175 Mg m⁻³
Mo Kα radiation
μ = 0.08 mm⁻¹
T = 295 K
Prism, colorless
0.35 × 0.35 × 0.25 mm

Data collection

Rigaku AFC-7R diffractometer
ω–2θ scans
Absorption correction: none
3 standard reflections every 150 reflections
3050 measured reflections
1510 independent reflections
1510 reflections with I > 2σ(I)
Refinement on F²
wR(F²) = 0.048
S = 1.01
1510 reflections
172 parameters
H-atom parameters constrained

Table 1

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<th>D—H—A</th>
<th>D—H</th>
<th>H···A</th>
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<td>N1—H1</td>
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<td>C7—H7</td>
<td>0.95</td>
<td>2.50</td>
<td>2.837 (6)</td>
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<tr>
<td>C7—H7</td>
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<td>2.53</td>
<td>3.422 (5)</td>
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<tr>
<td>C8—H8</td>
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<td>C10—H10</td>
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<tr>
<td>C12—H12</td>
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<td>3.361 (9)</td>
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<tr>
<td>C12—H12</td>
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<tr>
<td>Symmetry codes: (i) x, y, z + 1; (ii) x, y, z + 1</td>
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</table>

The carbon-bound H atoms were constrained as riding atoms, with C—H = 0.94–0.96 Å. Uiso(H) values were set at 1.2Ueq 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.

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References


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.
