## Structure Reports

Online
ISSN 1600-5368

Poulsen, Peter C. Healy and Alan R. White*

Eskitis Institute for Cell and Molecular Therapies, Griffith University, Nathan, Brisbane 4111, Australia

Correspondence e-mail:
Alan.White@griffith.edu.au

## Key indicators

Single-crystal X-ray study
$T=295 \mathrm{~K}$
Mean $\sigma(\mathrm{C}-\mathrm{C})=0.008 \AA$
$R$ factor $=0.048$
$w R$ factor $=0.160$
Data-to-parameter ratio $=8.8$
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

[^0]
## $N$-Cinnamoyl-L-valine methyl ester

The crystal structure of the title compound, $\mathrm{C}_{15} \mathrm{H}_{19} \mathrm{NO}_{3}$, shows that the overall molecular conformation, which is approximately planar, is stabilized by intermolecular $\mathrm{N}-\mathrm{H} \cdots \mathrm{O}$ 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).

i. DCM, Cinnamoyl Chloride
(2) triethylamine, 293 K , 3 days.

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 $\mathrm{C} 6-\mathrm{C} 1-\mathrm{C} 7-\mathrm{C} 8=-21.0(7)^{\circ}, \quad \mathrm{C} 7-\mathrm{C} 8-\mathrm{C} 9-\mathrm{O} 1=$ $-16.4(7)^{\circ}, \mathrm{C} 10-\mathrm{N} 1-\mathrm{C} 9-\mathrm{O} 1=2.3(8)^{\circ}$ and $\mathrm{N} 1-\mathrm{C} 10-$ $\mathrm{C} 14-\mathrm{O} 2=-12.4(6)^{\circ}$. The isopropyl group lies above this plane. The crystal structure is stabilized by intermolecular $\mathrm{N}-$ H...O interactions (Fig. 2), together with a number of weaker intra- and intermolecular $\mathrm{C}-\mathrm{H} \cdots \mathrm{O}$ interactions (Table 1).

## Experimental

Triethylamine ( $5.41 \mathrm{~g}, 53 \mathrm{mmol}$ ) was added dropwise to a solution of l-valine methyl ester hydrochloride, (1) ( $1 \mathrm{~g}, 5.96 \mathrm{mmol}$ ), and cinnamoyl chloride ( $994 \mathrm{mg}, 5.96 \mathrm{mmol}$ ) in anhydrous dichloromethane ( 100 ml ). The reaction mixture was stirred at room temperature ( 298 K ) for 3 d before being washed with $2 M \mathrm{HCl}(2 \times$ 100 ml ) and saturated brine solution ( 100 ml ), then dried over $\mathrm{MgSO}_{4}$. 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. ${ }^{1} \mathrm{H}$ NMR $\left(\mathrm{CDCl}_{3}, 300 \mathrm{MHz}\right): \delta 0.98\left(d, 3 \mathrm{H}, J=6.9 \mathrm{~Hz}, \delta \mathrm{CH}_{3}\right)$,
$1.01\left(d, 3 \mathrm{H}, J=6.9 \mathrm{~Hz}, \delta^{\prime} \mathrm{CH}_{3}\right), 2.20-2.31(m, 1 \mathrm{H}, \beta \mathrm{CH}), 3.79(s, 3 \mathrm{H}$, $\left.\mathrm{OCH}_{3}\right), 4.75(t, 1 \mathrm{H}, J=7.3 \mathrm{~Hz}, \alpha \mathrm{CH}), 6.12(b r d, 1 \mathrm{H}, \mathrm{NH}), 6.49(d, 1 \mathrm{H}$, $J=15.6 \mathrm{~Hz},=\mathrm{CHCO}), 7.38-7.43(m, 3 \mathrm{H}, \mathrm{ArH}), 7.52-7.56(m, 2 \mathrm{H}$, ArH), $7.67(d, 1 \mathrm{H}, J=15.6 \mathrm{~Hz},=\mathrm{CHPh}) ;{ }^{13} \mathrm{C} \mathrm{NMR}\left(\mathrm{CDCl}_{3}\right.$, $75 \mathrm{MHz}): \delta 17.9$ and $19.0\left(\delta \mathrm{CH}_{3}\right), 31.6(\beta \mathrm{CH})$, $52.3\left(\mathrm{OCH}_{3}\right), 57.1$ $(\alpha \mathrm{CH}), 120.1(=\mathrm{CHCO}), 127.9,128.8$ and $129.8(\mathrm{CH}$ from Ar$), 135.6$ $\left(\mathrm{C}\right.$ from Ar), $141.8(=\mathrm{CHPh}), 167.4(\mathrm{CONH}), 171.5\left(\mathrm{COOCH}_{3}\right)$; MS (LRMSES): $m / z 262.3[M+\mathrm{H}]^{+}, 284.3[M+\mathrm{Na}]^{+} ;$m.p. 362 K.

## Crystal data

$$
\begin{aligned}
& \mathrm{C}_{15} \mathrm{H}_{19} \mathrm{NO}_{3} \\
& M_{r}=261.31 \\
& \text { Orthorhombic, } P 2_{1} 2_{1} 2_{1} \\
& a=9.979(2) \AA \AA \\
& b=17.899(3) \AA \\
& c=8.2712(14) \AA \\
& V=1477.4(5) \AA
\end{aligned}
$$

$$
Z=4
$$

$$
\begin{aligned}
& Z=4 \\
& D_{x}=1.175 \mathrm{Mg} \mathrm{~m}^{-3}
\end{aligned}
$$

$$
\text { Mo } K \alpha \text { radiation }
$$

$$
\mu=0.08 \mathrm{~mm}^{-1}
$$

$$
T=295 \mathrm{~K}
$$

Prism, colorless $0.35 \times 0.35 \times 0.25 \mathrm{~mm}$

## Data collection

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

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

## Refinement

Refinement on $F^{2}$

$$
\begin{gathered}
w=1 /\left[\sigma^{2}\left(F_{\mathrm{o}}^{2}\right)+(0.0838 P)^{2}\right. \\
\quad+0.1628 P] \\
\text { where } P=\left(F_{\mathrm{o}}^{2}+2 F_{\mathrm{c}}^{2}\right) / 3 \\
(\Delta / \sigma)_{\max }<0.001 \\
\Delta \rho_{\max }=0.16 \mathrm{e} \AA^{-3} \\
\Delta \rho_{\min }=-0.24 \mathrm{e} \AA^{-3}
\end{gathered}
$$

$w R\left(F^{2}\right)=0.160$
$S=1.01$
1510 reflections
172 parameters
H -atom parameters constrained

Table 1
Hydrogen-bond geometry ( $\left(\AA^{\circ}{ }^{\circ}\right.$ ).

| $D-\mathrm{H} \cdots A$ | $D-\mathrm{H}$ | $\mathrm{H} \cdots A$ | $D \cdots A$ | $D-\mathrm{H} \cdots A$ |
| :--- | :--- | :--- | :--- | :--- |
| $\mathrm{~N} 1-\mathrm{H} 1 \cdots \mathrm{O}^{\mathrm{i}}$ | 0.96 | 2.22 | $3.136(4)$ | 158 |
| $\mathrm{C} 7-\mathrm{H} 7 \cdots \mathrm{O}^{\mathrm{i}}$ | 0.95 | 2.50 | $2.837(6)$ | 101 |
| $\mathrm{C} 7-\mathrm{H} 7 \cdots \mathrm{O}^{2 i}$ | 0.95 | 2.53 | $3.422(5)$ | 158 |
| $\mathrm{C} 8-\mathrm{H} 8 \cdots \mathrm{O}^{\mathrm{i}}$ | 0.96 | 2.58 | $3.358(5)$ | 138 |
| $\mathrm{C} 10-\mathrm{H} 10 \cdots \mathrm{O} 1$ | 0.94 | 2.37 | $2.781(6)$ | 106 |
| $\mathrm{C} 12-\mathrm{H} 12 C \cdots \mathrm{O} 1$ | 0.96 | 2.53 | $3.361(9)$ | 145 |
| C12-H12C $\cdots \mathrm{N} 1$ | 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 $\mathrm{C}-\mathrm{H}=0.94-0.96 \AA . U_{\text {iso }}(\mathrm{H})$ values were set at $1.2 U_{\text {eq }}$ 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.

We acknowledge financial support of this work by Griffith University and the Eskitis Institute for Cell and Molecular Therapies.

## References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. \& Taylor, R. (1987). J. Chem. Soc. Perkin Trans. 2, pp. S1-19.

Bornaghi, L. F., Poulsen, S.-A. \& Healy, P. C. (2004). Acta Cryst. E60, o383o385.
Farrugia, L. J. (1997). J. Appl. Cryst. 30, 565.
Grubbs, R. H. \& Chang, S. (1998). Tetrahedron, 54, 4413-4450.
Grubbs, R. H. \& Chang, S. (2004). Tetrahedron, 60, 7117-7140.
Molecular Structure Corporation (1999). MSC/AFC7 Diffractometer Control Software for Windows. Version 1.02. MSC, The Woodlands, Texas, USA.

## organic papers

Molecular Structure Corporation (2001). TEXSAN for Windows. Version 1.06. MSC, The Woodlands, Texas, USA
Poulsen, S.-A. \& Bornaghi, L. F. (2006). Bioorg. Med. Chem. 14, 3275-3284

Poulsen, S.-A., Noack, C. L. \& Healy, P. C. (2003). Acta Cryst. E59, o967-o968. Sheldrick, G. M. (1997). SHELXL97. University of Göttingen, Germany. Spek, A. L. (2003). J. Appl. Cryst. 36, 7-13.


[^0]:    © 2007 International Union of Crystallography All rights reserved

