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Methyl 2-(2-methoxycarbonylacetamido)-3-methylbutyrate

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Key indicators

Single-crystal X-ray study
T = 230 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.048
wR factor = 0.147
Data-to-parameter ratio = 9.4

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

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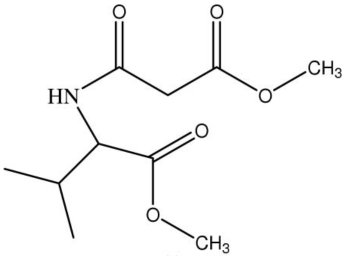
In the title compound, $C_{10}H_{17}NO_5$, an intramolecular C—H···O interaction generates an *S*(5) ring motif, whereas intermolecular N—H—O and C—H···O interactions form an $R_2^1(6)$ ring, linking the molecules into a tape along the *a* axis.

Comment

Naturally occurring heterocyclic compounds containing a pyrrolidinone unit have been studied extensively due to their varied structures and diverse bioactivities. The title compound, (I), can act as an essential intermediate in the synthesis of such a pyrrolidinone unit, which eventually can be used as a template in multi-step syntheses of diverse natural products such as janolusimide, a tripeptide neurotoxin, isolated from the marine microorganism *Janolus sp* (Giodarno *et al.*, 2000; Sodano & Spinella, 1986).



(I)

The bond lengths and angles in (I) show normal values (Allen *et al.*, 1987). The C1—C2—C3—N1 and N1—C4—C8—O5 torsion angles are 104.9 (3) and −142.0 (2)°, respectively. An intramolecular C4—H4*A*···O3 interaction generates an *S*(5) ring motif (Bernstein *et al.*, 1995) (Fig. 1). In the crystal structure, intermolecular N1—H1*A*···O3^{i} and C2—H2*B*···O3^{i} hydrogen bonds (symmetry code as in Table 1) form an $R_2^1(6)$ ring, linking the molecules along the *a* axis (Fig. 2). In addition, the crystal structure is further stabilized by a weak intermolecular C6*B*—H6B*C*···O2*B*^{i} interaction (Table 1).

Experimental

The title compound, (I), was synthesized *via* condensation between an equimolar amount of valine methyl ester (5.03 g, 30 mmol) and methyl malonate potassium salt (4.68 g, 30 mmol) in acetonitrile/water (70/20 ml) at 273 K. The mixture was stirred for 2 h in the presence of dicyclohexylcarbodiimide, which acted as a catalyst and a peptide-coupling agent. The white precipitate formed during the reaction was filtered and washed thoroughly with dichloromethane. The filtrate and the dichloromethane were combined and evaporated. The resultant crude product was partitioned between water and

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dichloromethane, and the dichloromethane extract was dried over anhydrous magnesium sulfate to afford (I) as a white powder (yield 6.26 g, 90%). Single crystals suitable for X-ray investigation were obtained by slow evaporation of an acetone–petroleum ether (4:1 *v*/*v*) solution. Analysis found: C 51.88, H 7.48, N 6.12, O 34.52%; calculated for $C_{10}H_{17}NO_5$: C 51.94, H 7.41, N 6.06, O 34.59%.

Crystal data

$C_{10}H_{17}NO_5$
$M_r = 231.25$
Orthorhombic, $P2_12_12_1$
$a = 9.2644$ (2) Å
$b = 10.7251$ (2) Å
$c = 12.9015$ (2) Å
$V = 1281.91$ (4) Å^3

$Z = 4$
$D_x = 1.198$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 230.0$ (1) K
Block, colourless
0.33 × 0.26 × 0.21 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{\min} = 0.969$, $T_{\max} = 0.981$

15828 measured reflections
1700 independent reflections
1267 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.032$
$\theta_{\max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.147$
$S = 1.08$
1700 reflections
181 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0816P)^2 + 0.1264P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4*A*···O3	0.98	2.36	2.783 (3)	105
N1—H1*A*···O3$^{\rm i}$	0.86	2.01	2.837 (3)	162
C2—H2*B*···O3$^{\rm i}$	0.97	2.30	3.115 (3)	142
C6*B*—H6*BC*···O2*B*$^{\rm i}$	0.96	2.15	3.063 (17)	160

Symmetry code: (i) $x - \frac{1}{2}, -y + \frac{3}{2}, -z + 1$.

H atoms were placed in calculated positions and constrained to ride on their carrier atoms, with C—H distances in the range 0.96–0.98 Å and N—H = 0.86 Å. The $U_{\rm iso}$(H) values were set equal to $1.5U_{\rm eq}$ of the carrier atom for methyl H atoms and $1.2U_{\rm eq}$ for the remaining H atoms. The ratio of the refined occupancies for the major and minor components of the disordered oxygen and methoxy groups are 0.60 (7):0.40 (7) and 0.584 (7):0.416 (7), respectively. In the final refinement, both occupancy ratios were fixed at 0.6:0.4. In the absence of significant anomalous dispersion effects, Friedel pairs were merged before the final refinement.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

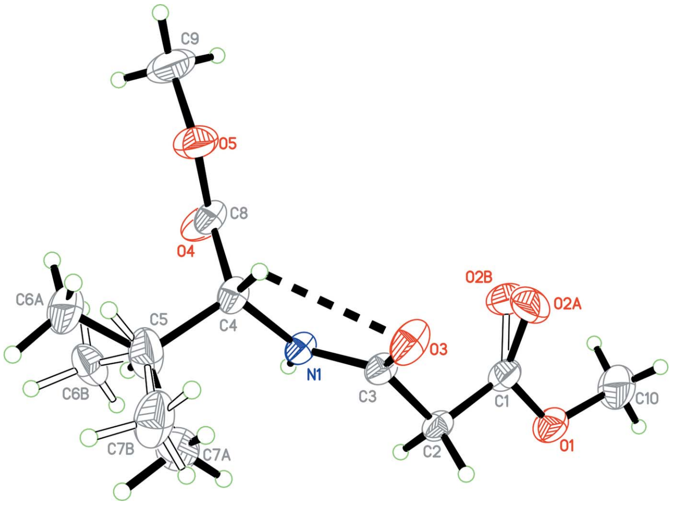

Figure 1
The molecular structure of (I), showing 20% probability displacement ellipsoids and the atomic numbering. The dashed line indicates the intramolecular hydrogen bond. Both disorder components are shown.

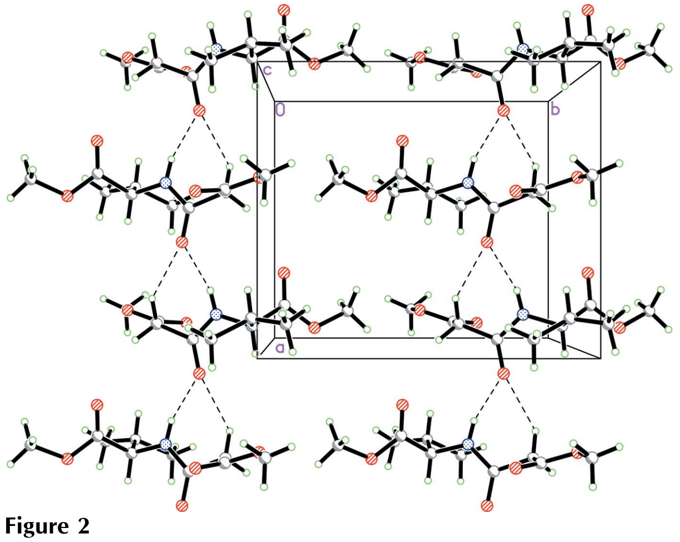

Figure 2
Packing diagram of the major disorder component of (I), viewed down the *c* axis. The dashed lines indicate intermolecular C—H···O and N—H···O interactions. The disordered atoms of the minor component have been omitted for clarity.

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