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O—H...O, C—H...O and C—H...
 π_{arene} intermolecular interactions
in (2*S*)-2-(1-oxo-1*H*-2,3-dihydro-
isoindol-2-yl)pentanoic acid and
(2*S*)-3-methyl-2-(1-oxo-1*H*-2,3-di-
hydroisoindol-2-yl)butanoic acid

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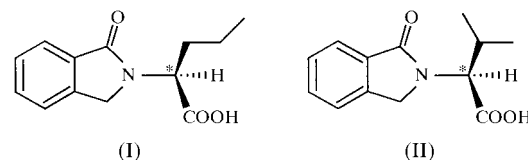
In the first of the title compounds, (2*S*)-2-(1-oxo-1*H*-2,3-dihydroisoindol-2-yl)pentanoic acid, C₁₃H₁₅NO₃, prepared from *L*-norvaline, a hydrogen-bonded network is formed in the solid state through O—H...O=C, C—H...O=C and C—H... π_{arene} intermolecular interactions, with shortest O...O, C...O and C...centroid distances of 2.582 (13), 3.231 (11) and 3.466 (3) Å, respectively. In the *L*-valine derivative, (2*S*)-3-methyl-2-(1-oxo-1*H*-2,3-dihydroisoindol-2-yl)butanoic acid, C₁₃H₁₅NO₃, O—H...O=C and C_{arene}—H...O=C intermolecular interactions generate a cyclic R₂²(9) motif through cooperativity, with shortest O...O and C...O distances of 2.634 (3) and 3.529 (5) Å, respectively. Methylene C—H...O=C_{indole} interactions complete the hydrogen bonding, with C...O distances ranging from 3.283 (4) to 3.477 (4) Å.

Comment

Phthalimidines (isoindolin-1-ones) often display biological activity as potential anti-inflammatory agents and anti-psychotics (Mukherjee *et al.*, 2000), and most of the structurally determined systems are either *N*-substituted or have a hydroxy substituent at the 3-position (McNab *et al.*, 1997; Kundu *et al.*, 1999). Amino acids constitute a fundamental building block in biological compounds and valine derivatives have been utilized in the formation of chiral host lattices (Weigand *et al.*, 1998). The title compounds, (2*S*)-2-(1-oxo-1*H*-2,3-dihydroisoindol-2-yl)pentanoic acid, (I), and (2*S*)-3-methyl-2-(1-oxo-1*H*-2,3-dihydroisoindol-2-yl)butanoic acid, (II), derived from *L*-norvaline and *L*-valine, respectively, form part of a systematic study of hydrogen-bonding interactions in a series of amino acid derivatives (Brady *et al.*, 1998; Dalton *et al.*, 1999; Gallagher & Murphy, 1999; Gallagher *et al.*, 2000).

Compound (I) crystallizes in space group *P*2₁2₁ with one molecule in the asymmetric unit and a view of (I) with the

atomic numbering scheme is given in Fig. 1, with selected dimensions in Table 1. The bond lengths and angles in the heterocyclic ring are similar to those reported previously (McNab *et al.*, 1997; Brady *et al.*, 1998; Gallagher & Murphy, 1999) and are in agreement with expected values (Orpen *et al.*, 1994). The carboxylic acid group exhibits rotational disorder, with site occupancies of 0.55 (4) and 0.45 (4) for the major and



minor orientations, respectively. The angle between the CO₂ planes is 31 (3)° and the major CO₂ orientation is at an angle of 67 (2)° to the C₄N ring plane [87.1 (16)° for the minor site]. The angle between the five- and six-membered rings of the isoindole system is 1.37 (17)° and the maximum deviation from planarity for an atom in either ring plane is 0.0084 (16) Å for C9 (C₆ ring), with the carbonyl O3 atom 0.026 (3) Å from the C₄N ring plane. The *n*-propyl chain adopts two conformations, with site occupancies of 0.519 (11) and 0.481 (11); details are given in the *Experimental* section.

The hydrogen bonding in (I) is dominated by O—H...O=C, C—H...O=C and C_{arene}—H... π_{arene} intermolecular interactions (Table 2 and Fig. 2). Conventional O—H...O hydrogen bonding is not observed, either between pairs of carboxylic acid groups [graph set R₂²(8); Ferguson *et al.*, 1995] or through interaction of the COOH group with a C—H/C=O pair from an isoindolin-1-one system [compound (III); graph set R₂²(9); Brady *et al.*, 1998]. Carboxylic acid O—H...O=C hydrogen bonds are formed with the heterocyclic ring C=O group O1A/O1B...O3ⁱ = 2.582 (13)/2.640 (12) Å [symmetry code: (i) 1 - x, y - 1/2, 1/2 - z], where B/A are the major/minor carboxylate sites. A C_{arene}—H...O interaction involving the carboxylic acid C=O moiety as C7—H7...O2Aⁱⁱ/O2Bⁱⁱ, with C...O = 3.231 (11)/3.49 (2) Å [symmetry code: (ii) 1/2 + x, 1/2 - y, 1 - z], generates a chain of C_{arene}^{iv}—H...O=C—O—H...O=Cⁱ_{indole} hydrogen bonds,

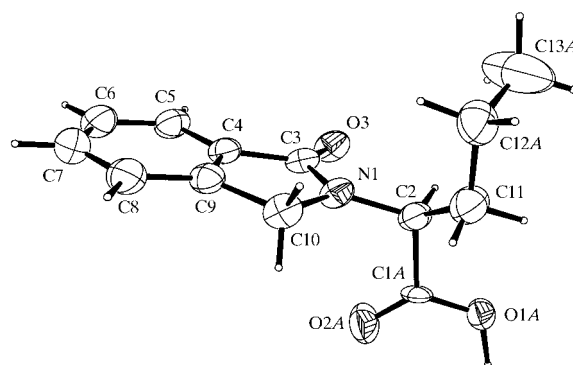


Figure 1

A view of (I) with the atomic numbering scheme. Atom labels with the suffix *A* indicate one of the disordered conformations of the carboxylic acid and *n*-propyl groups. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

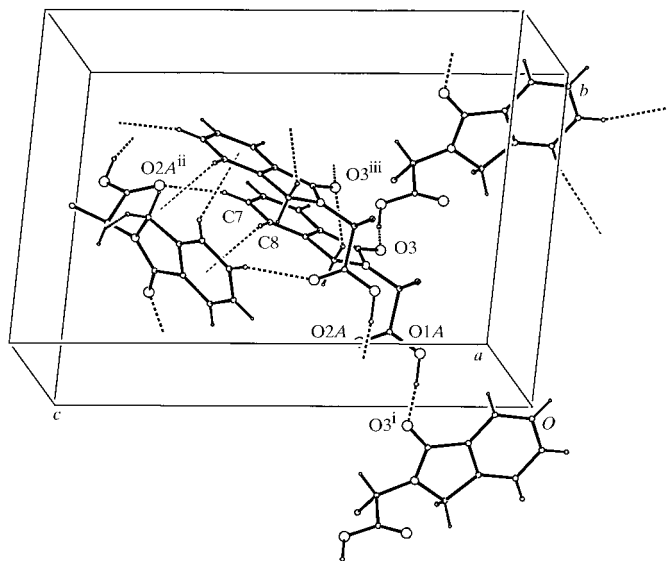


Figure 2
A view of the intermolecular interactions of (I) with the major conformation only; symmetry codes are as given in Table 2.

thus preventing the formation of a cyclic $R_2^2(9)$ system [Brady *et al.*, 1998; symmetry code: (iv) $-\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$]. Further association of (I) through $C8-H8 \cdots Cg1^{ii}$ interactions [$C8 \cdots Cg1^{ii} = 3.466(3) \text{ \AA}$, where $Cg1$ is the $C4-C9$ ring centroid] and $C10_{\text{methylene}}-H10A \cdots O3^{iii}$ interactions [$C \cdots O^{iii} = 3.335(3) \text{ \AA}$], complete the hydrogen-bonding network [symmetry code: (iii) $1 + x, y, z$]. The orientation of the isoindole ring defined by $C3-N1-C2-C1A$ is $-95.3(9)^\circ$, which is greater than the values of $-85.2(2)^\circ$ in a related 3-phenylpropanoic acid derivative, (III) (Brady *et al.*, 1998), or $-86.6(2)^\circ$ in a *meta*-tyrosine derivative, (IV) (Gallagher & Murphy, 1999), but smaller than the values of $-104.5(3)$ and $-112.29(14)^\circ$ in the chiral, (V), and racemic forms, (VI), of related threonine structures (Gallagher *et al.*, 2000).

Compound (II) crystallizes in space group $P1$, with two independent molecules, *A* and *B*, in the asymmetric unit, which differ slightly in conformation but retain the same configuration (*S*) at the chiral centre. A view of the asymmetric unit with the atomic numbering scheme is given in Fig. 3 and selected dimensions are given in Table 3. Bond lengths and angles are in accord with anticipated values (Orpen *et al.*, 1994). The r.m.s. deviation for the superposition of the non-H atoms in both molecules is 0.39 \AA (Spek, 1998). The angles between the five- and six-membered rings of the isoindole system are $1.0(2)$ (*A*) and $1.7(2)$ (*B*), and the maximum deviation from planarity for an atom in either indole ring is $0.010(3) \text{ \AA}$ for $C7A$, with the carbonyl $O3$ atom $0.039(5)$ (*A*) and $0.036(5) \text{ \AA}$ (*B*) from the C_4N ring plane. The angles between the CCO_2 group and the C_4N ring planes are $77.51(11)$ and $79.28(11)^\circ$ in molecules *A* and *B*, respectively. Torsion angle differences are evident from $N1-C2-C11-C12$ with values of $-43.6(4)$ (*A*) and $-52.0(3)^\circ$ (*B*) (Table 3). The orientations of the isoindole rings defined by $C3-N1-$

$C2-C1$ are $57.3(4)$ (*A*) and $60.7(4)^\circ$ (*B*), and these values are opposite to those in structures (I)–(VI), presumably due to steric hindrance of the isopropyl group in (II) compared with the *n*-propyl group in (I).

The hydrogen bonding in (II) is dominated by $O-H \cdots O=C$, $C-H \cdots O=C$ and $Csp^3-H \cdots O$ intermolecular interactions (Table 4 and Fig. 4). Hydrogen-bonded rings with graph set $R_2^2(9)$ are formed from the combination of acid $O1_{A/B}-H1 \cdots O3_{B/A}$ interactions with the heterocyclic ring $C=O$ group [$2.642(3)$ and $2.634(3) \text{ \AA}$, respectively] and arene $C5_{B/A}-H5 \cdots O2_{A/B}$ contacts with the carboxylic acid $C=O$ group [$3.529(5)$ and $3.714(5) \text{ \AA}$, respectively]. The $R_2^2(9)$ motif is present in a related 3-phenylpropanoic acid system, (III) (Brady *et al.*, 1998). This cooperativity generates a hydrogen-bonded zigzag chain in the direction of the *a* and *b* axes. The hydrogen-bonded network is completed by $C10-H10 \cdots O3$ interactions in which all four methylene H atoms, $H10A$ and $H10B$ in *A*, and $H10C$ and $H10D$ in *B*, participate. The $C5-H5 \cdots O2$ distances are longer in (II) than those in (III), although the $O \cdots O$ distances are similar. This $C \cdots O$ difference may be due to the weak intramolecular $C13-H13 \cdots O2$ contacts present in both molecules of (II).

The hydrogen bonding in (I) and (II) is similar in terms of hydrogen-bond numbers and associated distances per molecule, with one $O-H \cdots O$, two $C-H \cdots O$ and a $C-H \cdots \pi_{\text{arene}}$ interaction in (I), comparable with the $O-H \cdots O$ and three $C-H \cdots O$ interactions per molecule in (II). The unit-cell volumes of 1265.3 \AA^3 in (I) and 584.6 \AA^3 in (II) show a difference of 24 \AA^3 per molecule [316 \AA^3 in (I) and 292 \AA^3 in (II)], which can be accounted for by the carboxylic acid and *n*-propyl-group disorder in (I). The rotational disorder of the carboxylic acid group is assisted by the looser interactions involving the carboxylate $O2$ atom in (I). Examination of (II) and the major conformation of (I) with *PLATON* (Spek,

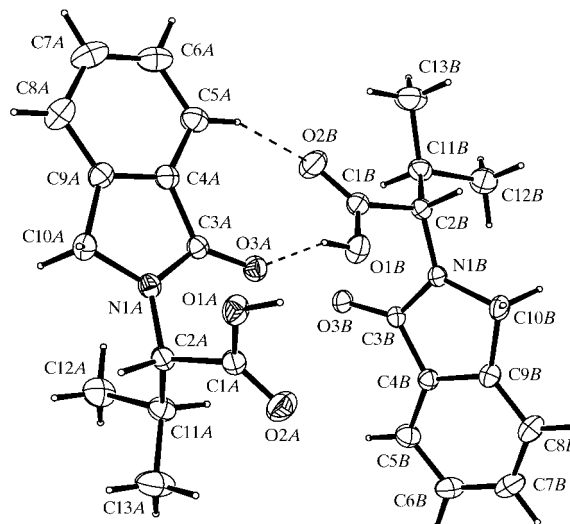


Figure 3
A view of the two independent molecules in the asymmetric unit of (II) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

1998) showed that there were no solvent-accessible voids in either crystal lattice. The hydrogen bonding in (II) can be compared with that of the two independent molecules which differ slightly in conformation in *N,N'*-dicyclohexyl-*N*-(3-pyridylcarbonyl)urea (Gallagher *et al.*, 1999). The overall crystal structure of (II) may be facilitated through hydrogen-bonded oligomeric units crystallizing from solution to produce the primary $[A \cdots B \cdots]_n$ hydrogen-bonded chain.

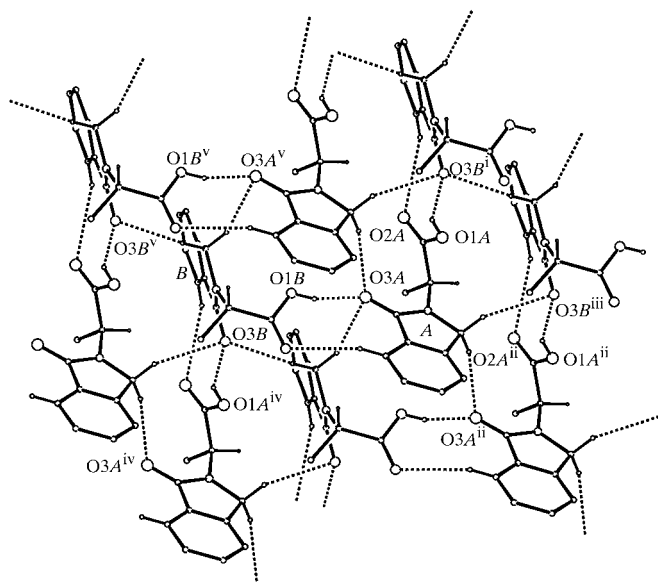


Figure 4

A view of the intermolecular interactions in (II); symmetry codes are as given in Table 4.

Experimental

The title compounds, (I) and (II), were prepared by the overnight reaction of *o*-phthalaldehyde with L-norvaline and L-valine, respectively, in refluxing CH_3CN under N_2 (Allin *et al.*, 1996). Filtration of the hot solution and subsequent slow cooling of the filtrate allowed the isolation of large colourless crystals from CH_3CN . Spectroscopic analysis for (I), m.p. 467–468 K (uncorrected): IR ($\nu_{\text{C=O}}$, cm^{-1}): 1730, 1649 (KBr); ^1H NMR (400 MHz, δ , d_6 -DMSO): 0.89 (*t*, 3H, CH_3), 1.29 (*br m*, 2H, CH_2), 1.90 (*br m*, 2H, CH_2), 4.49 (*q*, 2H, CH_2), 4.77 (*m*, 1H, CH), 7.48–7.51, 7.69–7.72 (*m*, 4H, C_6H_4). Spectroscopic analysis for (II), m.p. 436–438 K (uncorrected): IR ($\nu_{\text{C=O}}$, cm^{-1}): 1734, 1647, 1634 (KBr); ^1H NMR (400 MHz, δ , d_6 -DMSO): 0.84 (*d*, 3H, CH_3), 1.02 (*d*, 3H, CH_3), 2.29 (*br m*, 1H, CH), 4.53 (*m*, 2H, CH_2), 4.63 (*m*, 1H, CH), 7.30–7.37, 7.69–7.72 (*m*, 4H, C_6H_4).

Compound (I)

Crystal data

$\text{C}_{13}\text{H}_{15}\text{NO}_3$
 $M_r = 233.26$
 Orthorhombic, $P2_12_12_1$
 $a = 5.9384$ (4) Å
 $b = 12.3808$ (9) Å
 $c = 17.2097$ (14) Å
 $V = 1265.29$ (16) Å³
 $Z = 4$
 $D_x = 1.224$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 9.49$ – 19.63°
 $\mu = 0.087$ mm⁻¹
 $T = 294$ (1) K
 Block, colourless
 $0.48 \times 0.20 \times 0.18$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 4433 measured reflections
 1313 independent reflections
 1001 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.011$

$\theta_{\text{max}} = 25^\circ$
 $h = 0 \rightarrow 7$
 $k = -14 \rightarrow 14$
 $l = -20 \rightarrow 20$
 3 standard reflections
 frequency: 120 min
 intensity decay: <1%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.076$
 $S = 1.020$
 1313 reflections
 202 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0451P)^2 + 0.0395P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.09$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.10$ e Å⁻³
 Extinction correction: SHELXL97 (Sheldrick, 1997)
 Extinction coefficient: 0.047 (5)

Table 1

Selected geometric parameters (Å, °) for (I).

O3–C3	1.243 (3)	C2–C11	1.516 (4)
N1–C3	1.343 (3)	C3–C4	1.467 (3)
N1–C2	1.444 (3)	C9–C10	1.490 (3)
N1–C10	1.461 (3)		
C2–N1–C3	122.8 (2)	N1–C3–C4	107.5 (2)
C2–N1–C10	124.3 (2)	C3–C4–C5	130.5 (2)
C3–N1–C10	112.6 (2)	C3–C4–C9	108.0 (2)
N1–C2–C11	112.9 (2)	C4–C9–C10	109.5 (2)
O3–C3–N1	123.9 (2)	C8–C9–C10	129.9 (2)
O3–C3–C4	128.5 (2)		
C3–N1–C2–C1A	–95.3 (9)	C3–N1–C2–C1B	–95.8 (11)

Table 2

Hydrogen-bonding geometry (Å, °) for (I).

Cg is the centroid of the C4–C9 ring.

D–H \cdots A	D–H	H \cdots A	D \cdots A	D–H \cdots A
O1A–H1A \cdots O3 ⁱ	1.06	1.54	2.582 (13)	165
O1B–H1B \cdots O3 ⁱ	0.98	1.74	2.640 (12)	152
C7–H7 \cdots O2A ⁱⁱ	0.93	2.49	3.231 (11)	136
C7–H7 \cdots O2B ⁱⁱ	0.93	2.66	3.49 (2)	149
C8–H8 \cdots Cg ¹ⁱⁱ	0.93	2.74	3.466 (3)	136
C10–H10A \cdots O3 ⁱⁱⁱ	0.97	2.49	3.335 (3)	146

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

Compound (II)

Crystal data

$\text{C}_{13}\text{H}_{15}\text{NO}_3$
 $M_r = 233.26$
 Triclinic, $P1$
 $a = 5.8767$ (6) Å
 $b = 9.9036$ (13) Å
 $c = 10.4818$ (15) Å
 $\alpha = 103.332$ (13) $^\circ$
 $\beta = 99.759$ (11) $^\circ$
 $\gamma = 89.792$ (11) $^\circ$
 $V = 584.62$ (13) Å³

$Z = 2$
 $D_x = 1.325$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 9.48$ – 19.91°
 $\mu = 0.094$ mm⁻¹
 $T = 294$ (1) K
 Plate, colourless
 $0.45 \times 0.35 \times 0.14$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 5259 measured reflections
 2575 independent reflections
 2059 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.014$

$\theta_{\text{max}} = 27^\circ$
 $h = -7 \rightarrow 7$
 $k = -12 \rightarrow 12$
 $l = -13 \rightarrow 13$
 3 standard reflections
 frequency: 120 min
 intensity decay: <1%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.112$
 $S = 1.016$
 2575 reflections
 310 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0711P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.36 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.31 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXL97*
 (Sheldrick, 1997)
 Extinction coefficient: 0.022 (8)

Table 3

Selected geometric parameters (\AA , $^\circ$) for (II).

O1A—C1A	1.317 (4)	O1B—C1B	1.311 (4)
O2A—C1A	1.194 (4)	O2B—C1B	1.202 (4)
O3A—C3A	1.229 (4)	O3B—C3B	1.235 (3)
N1A—C2A	1.460 (4)	N1B—C2B	1.456 (4)
N1A—C3A	1.344 (4)	N1B—C3B	1.348 (4)
N1A—C10A	1.456 (4)	N1B—C10B	1.463 (4)
C1A—C2A	1.540 (4)	C1B—C2B	1.530 (4)
C2A—C11A	1.535 (5)	C2B—C11B	1.537 (4)
C3A—C4A	1.472 (4)	C3B—C4B	1.465 (4)
C9A—C10A	1.497 (5)	C9B—C10B	1.489 (5)
C2A—N1A—C3A	124.0 (3)	C2B—N1B—C3B	124.2 (2)
C2A—N1A—C10A	122.3 (3)	C2B—N1B—C10B	122.7 (2)
C3A—N1A—C10A	113.3 (3)	C3B—N1B—C10B	112.8 (2)
O1A—C1A—O2A	124.2 (3)	O1B—C1B—O2B	123.1 (3)
O1A—C1A—C2A	110.7 (3)	O1B—C1B—C2B	111.4 (3)
O2A—C1A—C2A	125.1 (3)	O2B—C1B—C2B	125.5 (3)
N1A—C2A—C1A	108.6 (2)	N1B—C2B—C1B	108.9 (2)
O3A—C3A—N1A	124.4 (3)	O3B—C3B—N1B	123.9 (3)
O3A—C3A—C4A	129.2 (3)	O3B—C3B—C4B	129.1 (3)
N1A—C10A—C9A	102.0 (3)	N1B—C10B—C9B	102.5 (2)
O1A—C1A—C2A—C11A	172.8 (3)	O1B—C1B—C2B—C11B	168.6 (3)
N1A—C2A—C11A—C12A	−43.6 (4)	N1B—C2B—C11B—C12B	−52.0 (3)
C1A—C2A—C11A—C13A	69.1 (4)	C1B—C2B—C11B—C13B	59.2 (4)
C3A—N1A—C2A—C1A	57.3 (4)	C3B—N1B—C2B—C1B	60.7 (4)

Table 4

Hydrogen-bonding geometry (\AA , $^\circ$) for (II).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1A—H1A \cdots O3B ⁱ	0.82	1.93	2.642 (3)	145
C5A—H5A \cdots O2B	0.93	2.68	3.529 (5)	152
C10A—H10B \cdots O3A ⁱⁱ	0.97	2.43	3.314 (4)	151
C10A—H10A \cdots O3B ⁱⁱⁱ	0.97	2.58	3.477 (4)	153
O1B—H1B \cdots O3A	0.82	1.87	2.634 (3)	154
C5B—H5B \cdots O2A ^{iv}	0.93	2.86	3.714 (5)	153
C10B—H10C \cdots O3A ^v	0.97	2.56	3.455 (4)	154
C10B—H10D \cdots O3B ^v	0.97	2.46	3.283 (4)	143

Symmetry codes: (i) $x, 1 + y, z$; (ii) $x - 1, y, z$; (iii) $x - 1, 1 + y, z$; (iv) $x, y - 1, z$; (v) $1 + x, y, z$.

For both compounds, all atoms bound to C were treated as riding, with the *SHELXL97* (Sheldrick, 1997) defaults for C—H distances and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2U_{\text{eq}}(\text{C})$ for the remainder. For (I), the H-atom sites of O—H were located from difference Fourier maps in the penultimate stages of refinement and were included at these positions in the calculations, with O—H distances of 1.07 and 0.98 \AA , while for (II), the H atom bound to O was located from a difference Fourier map and subsequently treated as a rigid rotating group, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. The absolute structures of (I) and (II) were not reliably determined by our X-ray analysis, but they can be inferred from the known absolute configurations of the L-norvaline and L-valine used in the synthesis of (I) and (II), respectively.

For both compounds, data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992); cell refinement: *CAD-4-PC Software*; data reduction: *NRCVAX96* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *NRCVAX96* and *SHELXL97*; molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEX* (McArdle, 1995) and *PLATON* (Spek, 1998); software used to prepare material for publication: *NRCVAX96*, *SHELXL97* and *PREP8* (Ferguson, 1998).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1085). Additional figures are also available. Services for accessing these data are described at the back of the journal.

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