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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-dihydroisoindol-2-yl)pentanoic acid and (2*S*)-3-methyl-2-(1-oxo-1*H*-2,3-dihydroisoindol-2-yl)butanoic acid

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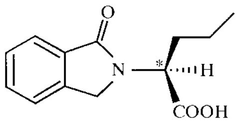
In the first of the title compounds, (2*S*)-2-(1-oxo-1*H*-2,3-dihydroisoindol-2-yl)pentanoic acid, $C_{13}H_{15}NO_3$, prepared from L-norvaline, a hydrogen-bonded network is formed in the solid state through O—H···O═C, C—H···O═C and C—H···π_{arene} intermolecular interactions, with shortest O···O, C···O 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, $C_{13}H_{15}NO_3$, O—H···O═C and C_{arene}—H···O═C intermolecular interactions generate a cyclic $R_2^2(9)$ 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.

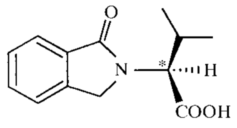
Comment

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 $P2_12_12_1$ 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 CO_2 planes is 31 (3)° and the major CO_2 orientation is at an angle of 67 (2)° to the C_4N 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 (C_6 ring), with the carbonyl O3 atom 0.026 (3) Å from the C_4N 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.



(I)



(II)

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 $R_2^2(8)$; 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 $R_2^2(9)$; Brady *et al.*, 1998]. Carboxylic acid O—H···O═C hydrogen bonds are formed with the heterocyclic ring C═O group O1*A*/O1*B*···O3^{i} = 2.582 (13)/2.640 (12) Å [symmetry code: (i) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$], 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···O2*A*^{ii}/O2*B*^{ii}, with C···O = 3.231 (11)/3.49 (2) Å [symmetry code: (ii) $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$], generates a chain of C^{iv}_{arene}—H···O═C—O—H···O═C^{i}_{indole} hydrogen bonds,

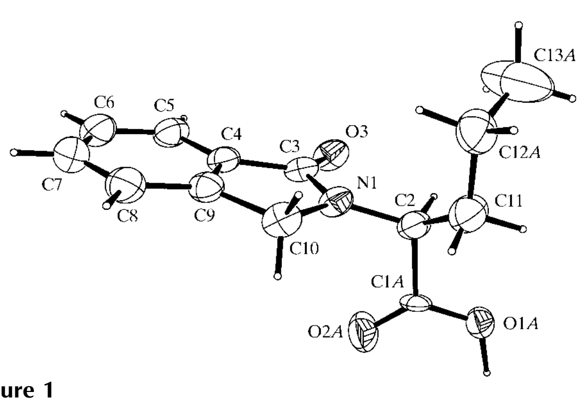


Figure 1
A view of (I) with the atomic numbering scheme. Atom labels with the suffix *A* indicate one of the disordered conformations of the carboxylic acid and *n*-propyl groups. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

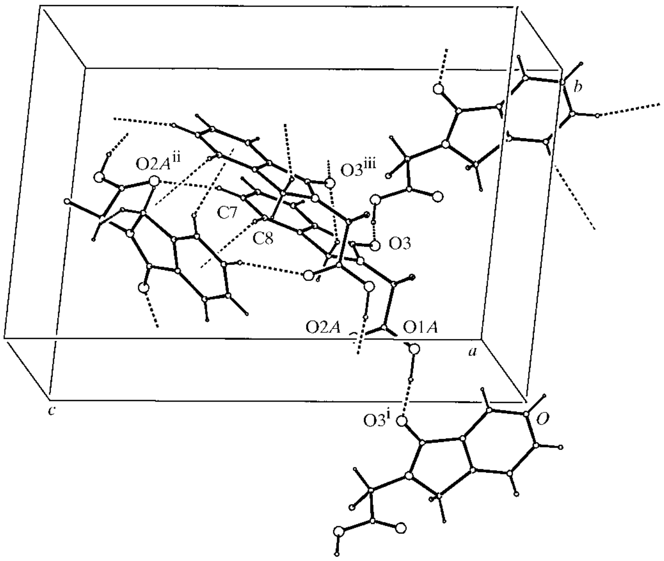


Figure 2
A view of the intermolecular interactions of (I) with the major conformation only; symmetry codes are as given in Table 2.

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) Å, where *Cg*1 is the C4–C9 ring centroid] and $C10_{methylene}-H10A\cdots O3^{iii}$ interactions [$C\cdots O^{iii}$ = 3.335 (3) Å], complete the hydrogen-bonding network [symmetry code: (iii) 1 + *x*, *y*, *z*]. The orientation of the isoindole ring defined by C3—N1—C2—C1*A* is −95.3 (9)°, which is greater than the values of −85.2 (2)° in a related 3-phenylpropanoic acid derivative, (III) (Brady *et al.*, 1998), or −86.6 (2)° in a *meta*-tyrosine derivative, (IV) (Gallagher & Murphy, 1999), but smaller than the values of −104.5 (3) and −112.29 (14)° in the chiral, (V), and racemic forms, (VI), of related threonine structures (Gallagher *et al.*, 2000).

Compound (II) crystallizes in space group *P*1, 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 Å (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) Å for C7*A*, with the carbonyl O3 atom 0.039 (5) (*A*) and 0.036 (5) Å (*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)° 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)° (*B*) (Table 3). The orientations of the isoindole rings defined by C3—N1—C2—C1 are 57.3 (4) (*A*) and 60.7 (4)° (*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) Å, 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) Å, 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, H10*A* and H10*B* in *A*, and H10*C* and H10*D* 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 $C-H\cdots \pi_{arene}$ 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 Å^3 in (I) and 584.6 Å^3 in (II) show a difference of 24 Å^3 per molecule [316 Å^3 in (I) and 292 Å^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,

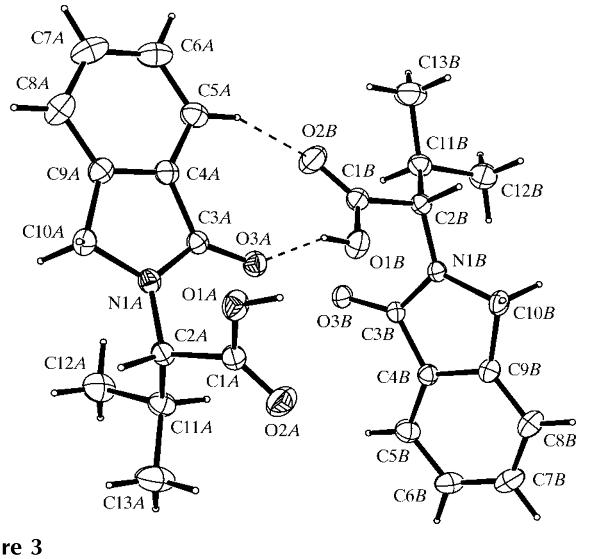


Figure 3
A view of the two independent molecules in the asymmetric unit of (II) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

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.

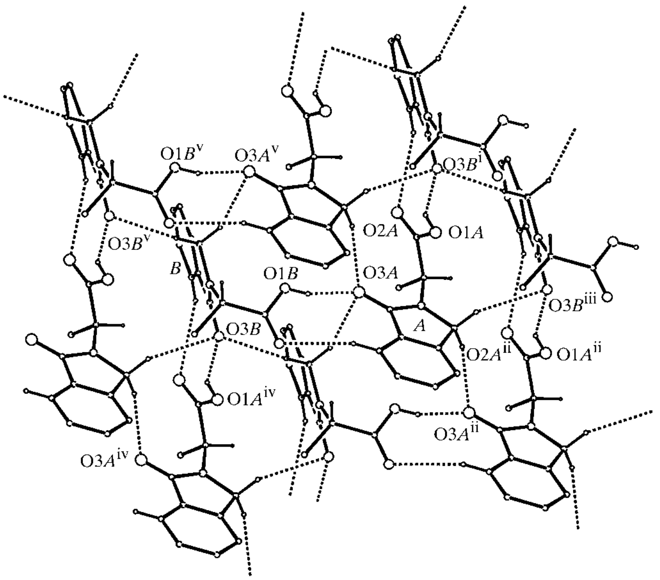


Figure 4
A view of the intermolecular interactions in (II); symmetry codes are as given in Table 4.

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 ($\nu_{C=O}$, cm^{-1}): 1730, 1649 (KBr); ^{1}H 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 ($\nu_{C=O}$ cm^{-1}): 1734, 1647, 1634 (KBr); ^{1}H 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

$C_{13}H_{15}NO_3$
$M_r = 233.26$
Orthorhombic, $P2_12_12_1$
$a = 5.9384\ (4)$ Å
$b = 12.3808\ (9)$ Å
$c = 17.2097\ (14)$ Å
$V = 1265.29\ (16)$ Å^3
$Z = 4$
$D_x = 1.224$ 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 × 0.20 × 0.18 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
4433 measured reflections
1313 independent reflections
1001 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.011$
$\theta_{\rm max} = 25°$
$h = 0 \rightarrow 7$
$k = -14 \rightarrow 14$
$l = -20 \rightarrow 20$
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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.09$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.10$ e Å^{-3}
Extinction correction: *SHELXL*97 (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—C1*A*	−95.3 (9)	C3—N1—C2—C1*B*	−95.8 (11)

Table 2
Hydrogen-bonding geometry (Å, °) for (I).

Cg is the centroid of the C4–C9 ring.

D—H···A	D—H	H···A	D···A	D—H···A
O1*A*—H1*A*···O3$^{\rm i}$	1.06	1.54	2.582 (13)	165
O1*B*—H1*B*···O3$^{\rm i}$	0.98	1.74	2.640 (12)	152
C7—H7···O2*A*$^{\rm ii}$	0.93	2.49	3.231 (11)	136
C7—H7···O2*B*$^{\rm ii}$	0.93	2.66	3.49 (2)	149
C8—H8···*Cg*1$^{\rm ii}$	0.93	2.74	3.466 (3)	136
C10—H10*A*···O3$^{\rm iii}$	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

$C_{13}H_{15}NO_3$
$M_r = 233.26$
Triclinic, $P1$
$a = 5.8767\ (6)$ Å
$b = 9.9036\ (13)$ Å
$c = 10.4818\ (15)$ Å
$\alpha = 103.332\ (13)°$
$\beta = 99.759\ (11)°$
$\gamma = 89.792\ (11)°$
$V = 584.62\ (13)$ Å^3
$Z = 2$
$D_x = 1.325$ 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 × 0.35 × 0.14 mm

Data collection

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

$\theta_{\rm max} = 27°$
$h = -7 \rightarrow 7$
$k = -12 \rightarrow 12$
$l = -13 \rightarrow 13$
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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.36$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.31$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.022 (8)

Table 3
Selected geometric parameters (Å, °) for (II).

O1*A*—C1*A*	1.317 (4)	O1*B*—C1*B*	1.311 (4)
O2*A*—C1*A*	1.194 (4)	O2*B*—C1*B*	1.202 (4)
O3*A*—C3*A*	1.229 (4)	O3*B*—C3*B*	1.235 (3)
N1*A*—C2*A*	1.460 (4)	N1*B*—C2*B*	1.456 (4)
N1*A*—C3*A*	1.344 (4)	N1*B*—C3*B*	1.348 (4)
N1*A*—C10*A*	1.456 (4)	N1*B*—C10*B*	1.463 (4)
C1*A*—C2*A*	1.540 (4)	C1*B*—C2*B*	1.530 (4)
C2*A*—C11*A*	1.535 (5)	C2*B*—C11*B*	1.537 (4)
C3*A*—C4*A*	1.472 (4)	C3*B*—C4*B*	1.465 (4)
C9*A*—C10*A*	1.497 (5)	C9*B*—C10*B*	1.489 (5)
C2*A*—N1*A*—C3*A*	124.0 (3)	C2*B*—N1*B*—C3*B*	124.2 (2)
C2*A*—N1*A*—C10*A*	122.3 (3)	C2*B*—N1*B*—C10*B*	122.7 (2)
C3*A*—N1*A*—C10*A*	113.3 (3)	C3*B*—N1*B*—C10*B*	112.8 (2)
O1*A*—C1*A*—O2*A*	124.2 (3)	O1*B*—C1*B*—O2*B*	123.1 (3)
O1*A*—C1*A*—C2*A*	110.7 (3)	O1*B*—C1*B*—C2*B*	111.4 (3)
O2*A*—C1*A*—C2*A*	125.1 (3)	O2*B*—C1*B*—C2*B*	125.5 (3)
N1*A*—C2*A*—C1*A*	108.6 (2)	N1*B*—C2*B*—C1*B*	108.9 (2)
O3*A*—C3*A*—N1*A*	124.4 (3)	O3*B*—C3*B*—N1*B*	123.9 (3)
O3*A*—C3*A*—C4*A*	129.2 (3)	O3*B*—C3*B*—C4*B*	129.1 (3)
N1*A*—C10*A*—C9*A*	102.0 (3)	N1*B*—C10*B*—C9*B*	102.5 (2)
O1*A*—C1*A*—C2*A*—C11*A*	172.8 (3)	O1*B*—C1*B*—C2*B*—C11*B*	168.6 (3)
N1*A*—C2*A*—C11*A*—C12*A*	−43.6 (4)	N1*B*—C2*B*—C11*B*—C12*B*	−52.0 (3)
C1*A*—C2*A*—C11*A*—C13*A*	69.1 (4)	C1*B*—C2*B*—C11*B*—C13*B*	59.2 (4)
C3*A*—N1*A*—C2*A*—C1*A*	57.3 (4)	C3*B*—N1*B*—C2*B*—C1*B*	60.7 (4)

Table 4
Hydrogen-bonding geometry (Å, °) for (II).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*A*—H1*A*···O3*B*$^{\rm i}$	0.82	1.93	2.642 (3)	145
C5*A*—H5*A*···O2*B*	0.93	2.68	3.529 (5)	152
C10*A*—H10*B*···O3*A*$^{\rm ii}$	0.97	2.43	3.314 (4)	151
C10*A*—H10*A*···O3*B*$^{\rm iii}$	0.97	2.58	3.477 (4)	153
O1*B*—H1*B*···O3*A*	0.82	1.87	2.634 (3)	154
C5*B*—H5*B*···O2*A*$^{\rm iv}$	0.93	2.86	3.714 (5)	153
C10*B*—H10*C*···O3*A*$^{\rm v}$	0.97	2.56	3.455 (4)	154
C10*B*—H10*D*···O3*B*$^{\rm v}$	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 *SHELXL*97 (Sheldrick, 1997) defaults for C—H distances and with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for methyl H atoms and $1.2U_{\rm eq}({\rm C})$ 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 Å, 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 $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm O})$. 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: *NRCVAX*96 (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *NRCVAX*96 and *SHELXL*97; molecular graphics: *ORTEP*III (Burnett & Johnson, 1996), *ORTEX* (McArdle, 1995) and *PLATON* (Spek, 1998); software used to prepare material for publication: *NRCVAX*96, *SHELXL*97 and *PREP*8 (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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