Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Two new conformationally restricted 4,5-dihydroxynorvaline analogues with a norbornane skeleton

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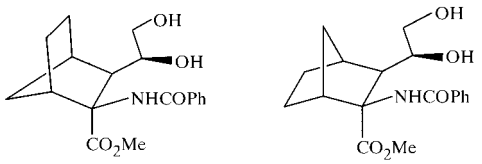
Received 1 October 1999
Accepted 1 February 2000

The structures of two conformationally restricted 4,5-dihydroxynorvaline analogues with a norbornane skeleton, namely methyl (1*S*,2*S*,3*R*,4*R*)-2-benzamido-3-(1,2-dihydroxyethyl)bicyclo[2.2.1]heptane-2-carboxylate, $C_{18}H_{23}NO_5$, and methyl (1*R*,2*S*,3*R*,4*S*)-2-benzamido-3-(1,2-dihydroxyethyl)bicyclo[2.2.1]heptane-2-carboxylate, $C_{18}H_{23}NO_5$, exhibit a conformation in the helical region of the φ,ψ map but their handedness is opposite. In both cases, the torsion angles ($\chi^{1,1}$) giving the relative orientation of the 1,2-dihydroxyethyl group of the amino acid side chain and the benzamide group of the peptide chain indicate that these groups adopt a nearly eclipsed conformation. Both compounds show a complex hydrogen-bonding pattern.

Comment

Amino acids containing one or more hydroxy groups form an important class of naturally occurring compounds (Hunt, 1985). Such compounds, whether of natural or synthetic origin, are useful precursors in the synthesis of β-lactams (Miller, 1986). An important example is (2*S*,4*S*)-4,5-dihydroxynorvaline (Liao & Zhou, 1998; Girard *et al.*, 1998), which is a key intermediate in the synthesis of the antibiotic clavalanine.

For some years, we have been focusing our attention on those amino acids that possess specific conformational and topographical modifications on their side-chain and, in particular, cyclization of the side-chain atoms with the main-chain atoms. This structural modification can result in significant changes in potency, receptor selectivity and biostability when incorporated into bioactive compounds. Recently, we reported our findings on the asymmetric Diels–Alder reaction between (*Z*)-2-phenyl-4-[(*S*)-2,2-dimethyl-1,3-dioxolan-4-ylmethylene]-5-(4*H*)-oxazolone and cyclopentadiene. The corresponding *exo*/*endo* adducts were isolated in enantiomerically pure form and transformed into two new conformationally constrained 4,5-dihydroxynorvaline analogues with a norbornane skeleton (Buñuel, Cativiela & Díaz-de-Villegas, 1996), methyl (1*S*,2*S*,3*R*,4*R*)-2-benz-amido-3-(1,2-dihydroxyethyl)bicyclo[2.2.1]heptane-2-carboxylate, *exo*-(I), and methyl (1*R*,2*S*,3*R*,4*S*)-2-benzamido-3-(1,2-dihydroxyethyl)bicyclo-[2.2.1]heptane-2-carboxylate, *endo*-(I). We describe here the crystal and molecular structures of these two new diastereomeric amino acids.



exo-(I) *endo*-(I)

Compound *exo*-(I) crystallizes with two molecules (*A* and *B*) in the asymmetric unit (Fig. 1). The numbering of the atoms runs from 1 to 18 for the first molecule and from 19 to 36 for the second. These two independent molecules differ slightly in their conformation. The main difference between them is the orientation of the phenyl group, with N1—C10—C11—C12 and O3—C10—C11—C16 torsion angles of 7.7 (5) and 3.6 (5)° for molecule *A* and N2—C28—C29—C30 and O8—C28—C29—C34 torsion angles of −28.6 (5) and −30.5 (6)° for molecule *B*. The configuration at the chiral atoms C1, C2, C3, C4 and C17 for the first conformer and at chiral atoms C19, C20, C21, C22 and C35 for the second are *S*, *S*, *R*, *R* and *S*, respectively. The bond lengths and angles show small variations from reported values for other norbornane amino acid derivatives (Apgar & Ludwig, 1972; Glass *et al.*, 1990; Buñuel,

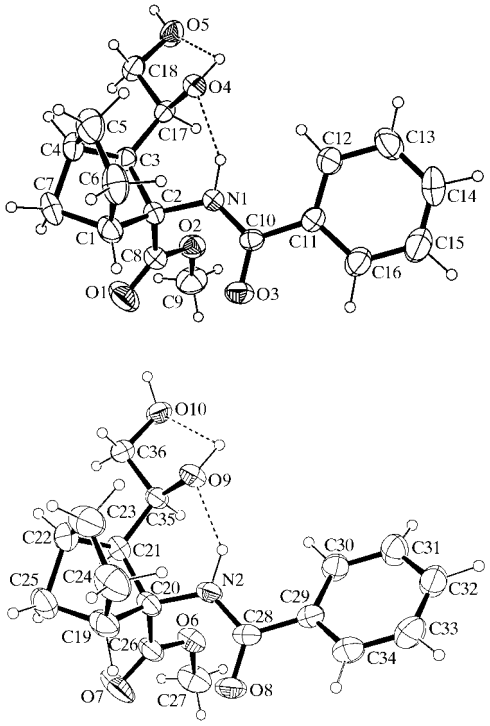


Figure 1
The molecular structures of the two conformers forming the asymmetric unit of *exo*-(I). Displacement ellipsoids are drawn at the 30% probability level and intramolecular hydrogen bonds are indicated by dashed lines. H atoms are shown as spheres of arbitrary radii.

Cativiela, Díaz-de-Villegas & Gálvez, 1996; Buñuel *et al.*, 1997). The C4—C7—C1 and C19—C25—C22 angles of 94.2 (3) and 93.3 (3)°, respectively, are significantly contracted with respect to the regular tetrahedral value, while the endocyclic angles of the five-membered norbornane rings are in the range 98.7 (4)–105.7 (5)°. The values for the conformationally sensitive N—C^{α}—C′ (τ) bond angles [N1—C2—C8 = 109.5 (3)° and N2—C20—C26 = 108.3 (3)°], which are external to the cyclic system, are very close to the tetrahedral value, as would be expected for the $C^{\alpha,\alpha}$-dialkylated glycines that form regular helices (Benedetti *et al.*, 1997).

In the bicyclo[2.2.1]heptane (norbornane) unit of each independent molecule, the two five-membered rings are in envelope conformations, while the six-membered ring adopts an approximate boat conformation. The slightly distorted boat conformation of the norbornane six-membered rings, C1–C6 and C19–C24, is evidenced by the puckering parameters (Cremer & Pople, 1975): q_2 = 0.919 (4) Å, q_3 = 0.022 (4) Å, φ_2 = 2.7 (3)°, θ_2 = 88.6(2)° and Q_T = 0.919 (4) Å, and q_2 = 0.928 (5) Å, q_3 = 0.026 (5) Å, φ_2 = 4.2 (3)°, θ_2 = 88.4 (3)° and Q_T = 0.929 (5) Å, respectively. In both independent molecules, the substitution of the norbornane ring produces a twist of type *S*-(+,+) (Altona & Sundaralingam, 1970) about the C1···C4 or C19···C22 vectors. The twisting can be seen from the C1—C2—C3—C4 and C4—C5—C6—C1 torsion angles of 4.5 (3) and 0.8 (4)°, respectively, for molecule *A*, and from the C19—C20—C21—C22 and C22—C23—C24—C19 torsion angles of 6.5 (4) and 2.6 (5)°, respectively, for molecule *B*.

In *exo*-(I), the amino acid residue adopts a folded conformation. The values of the backbone torsion angles φ [C10—N1—C2—C8 = −52.3 (5)° for molecule *A* and C28—N2—C20—C26 = −52.4 (5)° for molecule *B*] and ψ [N1—C2—C8—O2 = −47.1 (4)° for molecule *A* and N2—C20—C26—O6 = −42.7 (4)° for molecule *B*] fall in the *A* region of the conformational map (Zimmerman *et al.*, 1977). These values differ by less than 20° from those pertaining to the ideal 3_{10}-helix (60, 30°) or α-helix (55, 45°). The torsion angles ω [C11—C10—N1—C2 = 177.3 (3)° and C29—C28—N2—C20 = 176.1 (3)°] indicate that the amide linkage adopts the usual *trans* conformation in both conformers. The spatial arrangement of the 1,2-dihydroxyethyl group of the amino acid side-chain with respect to the peptide chain is defined by the torsion angles $\chi^{1,1}$ [N1—C2—C3—C17 = 15.8 (4)° for molecule *A* and N2—C20—C21—C35 = 16.9 (5)° for molecule *B*]. These values indicate that the benzamide and 1,2-dihydroxyethyl groups are in a nearly eclipsed conformation. In addition, both groups are involved in an N—H···O intramolecular hydrogen bond with an N1···O4 distance of 2.712 (4) Å for the first independent molecule and an N2···O9 distance of 2.698 (4) Å for the second. Another short intramolecular contact is observed between the two hydroxy groups [O4···O5 2.857 (4) and O9···O10 2.874 (4) Å].

In the crystals of *exo*-(I), the two independent molecules of the asymmetric unit form dimers in which two hydrogen bonds are observed between the O4—H4O donor of molecule *A* and the O10 acceptor of molecule *B*, with a distance of 2.714 (4) Å, and between the O9—H9O donor of molecule *B* and the O5 acceptor of molecule *A*, with a distance of 2.783 (4) Å. Moreover, the crystal structure is stabilized by two additional O—H···O intermolecular hydrogen bonds, involving the terminal hydroxy group of each independent molecule and the benzamide group of a symmetry-related molecule of the same type (*A* or *B*) [O5···O3 2.739 (4) Å and O10···O8 2.673 (4) Å]. Both hydroxy groups function as hydrogen-bond donors, whereby the molecules are linked into sheets parallel to the *z* axis (Fig. 2). Some short intra- and intermolecular C—H···O contacts (Table 2) are also found, which can be

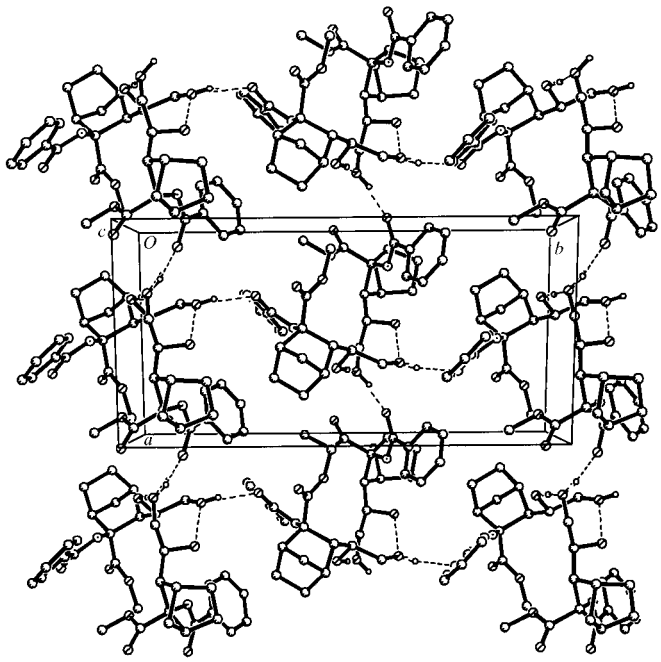


Figure 2
Packing diagram for *exo*-(I) viewed parallel to the *z* axis. H atoms not involved in hydrogen bonds have been omitted for clarity. Hydrogen bonds are indicated by dashed bonds.

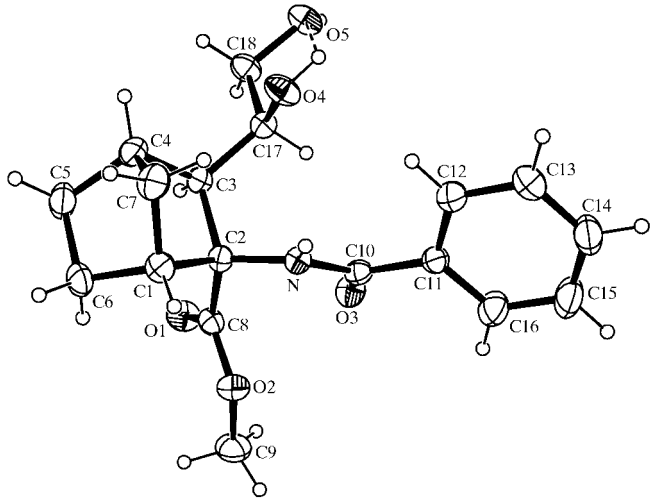


Figure 3
The molecular structure of *endo*-(I). Displacement ellipsoids are drawn at the 30% probability level and the intramolecular hydrogen bond is indicated by a dashed line. H atoms are shown as spheres of arbitrary radii.

described as weak hydrogen bonds (Steiner, 1997; Desiraju, 1996).

Diastereoisomer *endo*-(I) crystallizes with one molecule in the asymmetry unit (Fig. 3). The stereochemistry at the chiral C1, C2, C3, C4 and C17 atoms is *R*, *S*, *R*, *S* and S, respectively. Comparison of the bond distances and angles in *endo*-(I) with those determined for other norbornane amino acids, and in particular with *exo*-(I), reveals no strikingly unusual features and these parameters lie within the expected ranges. The C4—C7—C1 angle is 94.5 (2)° and the endocyclic angles of the five-membered norbornane rings vary from 101.2 (2) to 103.2 (2)°, which are all substantially less than the regular tetrahedral value of 109.5°. The critical intra-ring bond angle τ [N—C2—C8 = 109.48 (17)°] is comparable with that in *exo*-(I).

The two five-membered norbornane rings of *endo*-(I) are in envelope conformations, while the six-membered norbornane ring is in an almost perfect boat conformation [puckering parameters are q_2 = 0.958 (9) Å, q_3 = 0.005 (3) Å, φ_2 = 179.51 (17)°, θ_2 = 89.69 (15)° and Q_T = 0.958 (9) Å]. The norbornane system shows a weak distortion from C_{2v} symmetry, although it is less important than in *exo*-(I). The twisting can be seen from the C1—C2—C3—C4 and C4—C5—C6—C1 torsion angles of 1.4 (2) and 0.0 (3)°, respectively.

The amino acid residue in *endo*-(I) exhibits a conformation in the helical region of the φ,ψ map, but it has opposite handedness to that shown by *exo*-(I): the values of the backbone torsion angles φ [C10—N—C2—C8 = 42.8 (3)°] and ψ [N—C2—C8—O2 = 50.1 (2)°] fall in the A^* region of the conformational map (Zimmerman *et al.*, 1977). The torsion angle ω (C11—C10—N—C2) differs by less than 10° from 180°, the ideal value of the *trans* planar amide unit. In *endo*-(I), the torsion angle $\chi^{1,1}$ [N—C2—C3—C17 = 9.8 (3)°] also shows that the 1,2-dihydroxyethyl and benzamide groups are in a nearly eclipsed conformation. However, in contrast with *exo*-(I), no intramolecular hydrogen bond is observed between these two groups, whereas the short intramolecular contact involving both hydroxy groups observed in *exo*-(I) is also present in *endo*-(I) [O4···O5 2.787 (3) Å].

Apart from the spatial arrangement of the methyl ester and the benzamido and 1,2-dihydroxyethyl groups relative to the norbornane ring, the main difference between the 4,5-dihydroxynorvaline analogues described here, *i.e.* *endo*-(I) and *exo*-(I), involves the intermolecular hydrogen-bond pattern. In the crystals of *endo*-(I) three intermolecular hydrogen bonds are seen: one weak O(hydroxy)—H···O/N(amide/methyl ester) three-centre hydrogen bond [O4···N 3.239 (3) Å and O4···O2 3.026 (2) Å], one O(hydroxy)—H···O=C(amide) two-centre hydrogen bond [O5···O3 2.728 (3) Å] and one N(amide)—H···O(hydroxy) two-centre hydrogen bond [N···O5 2.917 (3) Å]. Both OH and NH groups function as hydrogen-bond donors, whereby the molecules are linked into sheets parallel to the *z* axis (Fig. 4). As in the diastereoisomer *exo*-(I), some possible borderline cases of C—H···O hydrogen bonds are also present in *endo*-(I) (Table 4).

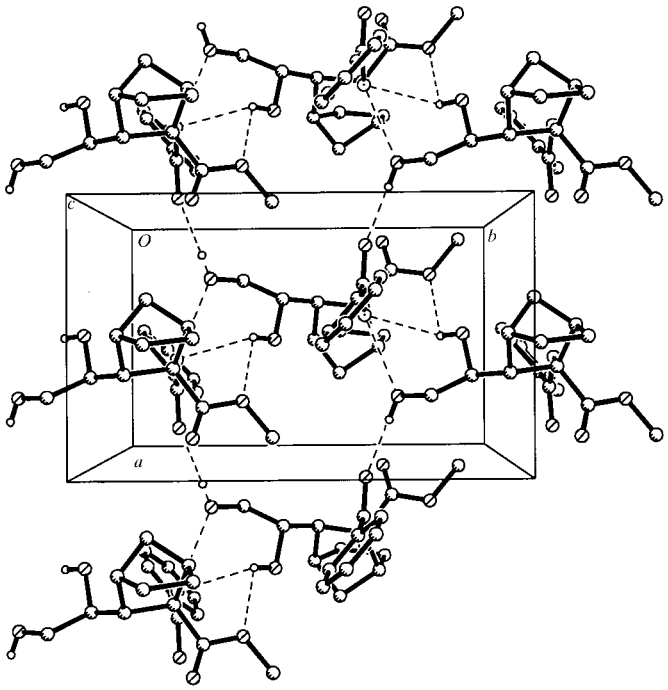


Figure 4
Packing diagram for *endo*-(I) viewed parallel to the *z* axis. H atoms not involved in hydrogen bonds have been omitted for clarity. Hydrogen bonds are indicated by dashed bonds.

Experimental

Compounds *exo*-(I) and *endo*-(I) were prepared according to the procedure previously described by Buñuel, Cativiela & Díaz-de-Villegas (1996). Crystals were obtained by slow evaporation of methanol solutions.

Compound *exo*-(I)

Crystal data

$C_{18}H_{23}NO_5$
M_r = 333.37
Monoclinic, $P2_1$
a = 9.323 (5) Å
b = 18.038 (5) Å
c = 10.968 (5) Å
β = 106.570 (5)°
V = 1767.9 (13) Å^3
Z = 4
D_x = 1.253 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 38 reflections
θ = 7.19–12.47°
μ = 0.091 mm^{-1}
T = 293 (2) K
Prism, colourless
0.64 × 0.32 × 0.10 mm

Data collection

Siemens *P*4 diffractometer
$\omega/2\theta$ scans
7359 measured reflections
3213 independent reflections
2274 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 25°
$h = -1 \rightarrow 11$
$k = -21 \rightarrow 21$
$l = -13 \rightarrow 12$
3 standard reflections every 97 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.093
S = 1.053
3213 reflections
459 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0422P)^2 + 0.0448P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Selected geometric parameters (Å, °) for *exo*-(I).

O1—C8	1.192 (5)	C2—C3	1.577 (5)
O4—C17	1.422 (4)	C3—C17	1.524 (5)
O5—C18	1.412 (5)	C3—C4	1.541 (5)
N1—C2	1.463 (4)	C4—C5	1.504 (6)
C1—C6	1.514 (6)	C4—C7	1.524 (6)
C1—C2	1.549 (5)	C5—C6	1.514 (7)
C1—C7	1.552 (6)	C17—C18	1.513 (5)
C2—C8	1.520 (5)		
C6—C1—C2	109.0 (3)	C5—C4—C3	111.9 (3)
C6—C1—C7	99.2 (4)	C7—C4—C3	100.3 (3)
C2—C1—C7	101.5 (3)	C4—C5—C6	103.3 (4)
N1—C2—C1	114.8 (3)	C1—C6—C5	105.1 (4)
C8—C2—C1	111.4 (3)	O1—C8—C2	124.4 (4)
N1—C2—C3	110.3 (3)	O2—C8—C2	112.0 (3)
C8—C2—C3	108.2 (3)	N1—C10—C11	117.6 (3)
C1—C2—C3	102.4 (3)	O4—C17—C18	110.9 (3)
C17—C3—C4	119.8 (3)	O4—C17—C3	111.1 (3)
C17—C3—C2	117.6 (3)	C18—C17—C3	111.5 (3)
C4—C3—C2	102.8 (3)	O5—C18—C17	108.3 (3)
C5—C4—C7	101.4 (4)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4O···O10	0.81 (4)	1.96 (4)	2.714 (4)	155 (4)
O5—H5O···O3^{i}	0.81 (6)	1.96 (6)	2.739 (4)	161 (5)
N1—H1*A*···O4	0.85 (3)	1.97 (4)	2.712 (4)	146 (3)
O9—H9O···O5	0.92 (6)	1.94 (6)	2.783 (4)	153 (5)
O10—H10O···O8^{ii}	0.86 (4)	1.86 (4)	2.673 (4)	159 (4)
N2—H2*A*···O9	0.82 (3)	1.98 (3)	2.698 (4)	146 (3)
O4—H4O···O5	0.81 (4)	2.49 (4)	2.857 (4)	109 (3)
O9—H9O···O10	0.92 (6)	2.48 (6)	2.874 (4)	106 (5)
C5—H5*B*···O4	0.97	2.30	3.005 (5)	129
C23—H23*B*···O9	0.97	2.35	3.067 (6)	130
C33—H33···O3^{iii}	0.93	2.44	3.230 (6)	143

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

Compound *endo*-(I)

Crystal data

$C_{18}H_{23}NO_5$
M_r = 333.37
Orthorhombic, $P2_12_12_1$
a = 7.187 (5) Å
b = 11.720 (5) Å
c = 20.258 (5) Å
V = 1706.4 (15) Å^3
Z = 4
D_x = 1.298 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 28 reflections
θ = 7.57–12.46°
μ = 0.095 mm^{-1}
T = 293 (2) K
Prism, colourless
0.40 × 0.36 × 0.30 mm

Data collection

Siemens *P*4 diffractometer
$\omega/2\theta$ scans
3472 measured reflections
1748 independent reflections
1525 reflections with $I > 2\sigma(I)$
R_{int} = 0.013
θ_{max} = 25°
$h = -8 \rightarrow 8$
$k = -13 \rightarrow 13$
$l = -24 \rightarrow 24$
3 standard reflections
every 97 reflections
intensity decay: none

Table 3
Selected geometric parameters (Å, °) for *endo*-(I).

O1—C8	1.197 (3)	C2—C3	1.578 (3)
O4—C17	1.416 (3)	C3—C17	1.527 (3)
O5—C18	1.424 (3)	C3—C4	1.538 (3)
N—C2	1.465 (3)	C4—C7	1.529 (4)
C1—C7	1.521 (4)	C4—C5	1.538 (4)
C1—C6	1.542 (4)	C5—C6	1.542 (4)
C1—C2	1.555 (3)	C17—C18	1.508 (3)
C2—C8	1.523 (3)		
C7—C1—C6	101.2 (2)	C7—C4—C5	101.2 (2)
C7—C1—C2	101.65 (18)	C3—C4—C5	108.6 (2)
C6—C1—C2	109.2 (2)	C4—C5—C6	103.2 (2)
N—C2—C1	108.91 (18)	C5—C6—C1	103.1 (2)
C8—C2—C1	110.10 (17)	O1—C8—C2	125.8 (2)
N—C2—C3	112.92 (18)	O2—C8—C2	110.29 (19)
C8—C2—C3	112.82 (18)	N—C10—C11	116.71 (19)
C1—C2—C3	102.34 (18)	O4—C17—C18	110.30 (19)
C17—C3—C4	114.78 (19)	O4—C17—C3	109.21 (18)
C17—C3—C2	115.21 (18)	C18—C17—C3	112.21 (19)
C4—C3—C2	102.40 (17)	O5—C18—C17	109.7 (2)
C7—C4—C3	102.71 (18)		

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.070
S = 1.058
1748 reflections
231 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0310P)^2 + 0.3022P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.054 (2)

Table 4
Hydrogen-bonding geometry (Å, °) for *endo*-(I).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4O···N^{i}	0.87 (3)	2.50 (3)	3.239 (3)	143 (3)
O4—H4O···O2^{i}	0.87 (3)	2.55 (3)	3.026 (2)	116 (3)
O5—H5O···O3^{ii}	0.80 (3)	1.93 (3)	2.728 (3)	175 (3)
N—H···O5^{iii}	0.86 (3)	2.17 (3)	2.917 (3)	146 (2)
O4—H4O···O5	0.87 (3)	2.39 (3)	2.787 (3)	109 (2)
C3—H3···O1	0.98	2.39	2.882 (3)	111
C7—H7*A*···O4	0.97	2.36	2.960 (3)	120
C7—H7*B*···O1^{iv}	0.97	2.57	3.313 (4)	134

Symmetry codes: (i) $-x, \frac{1}{2}+y, \frac{3}{2}-z$; (ii) $-1-x, \frac{1}{2}+y, \frac{3}{2}-z$; (iii) $-x, y-\frac{1}{2}, \frac{3}{2}-z$; (iv) $1+x, y, z$.

The absolute configurations of compounds *exo*-(I) and *endo*-(I) were deduced from the known stereochemistry of the chiral centre at C17 [for conformer *A* of *exo*-(I) and for *endo*-(I)] or C35 [for conformer *B* of *exo*-(I)], which was derived from the (*R*)-*O*,*O*-isopropylideneglyceraldehyde starting material. H atoms bonded to N or O were located from Fourier syntheses and refined freely, methyl group H atoms were refined as rigid groups (initial position taken from Fourier syntheses and H atoms allowed to rotate but not tip) and the remaining H atoms were treated as riding. All H atoms bonded to C were refined with fixed individual displacement parameters [U_{iso}(H) = 1.5U_{eq}-(C_{methyl}) or 1.2U_{eq}(C)]. Molecular geometry calculations were performed using *PARST* (Nardelli, 1983).

For both compounds, data collection: *XSCANS* (Siemens, 1993); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1989); software used to prepare material for publication: *SHELXL*97.

This work was supported by the Dirección General de Investigación Científica y Técnica (project number PB97-0998-C02-01) and Diputación General de Aragón (project number P22/98).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1375). Services for accessing these data are described at the back of the journal.

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