Table 3. *Hydrogen-bonding geometry (Å, °) for (1)*

D—H···A		D—H	H···A	D···A	D—H···A
C5—H5···O1^{i}	(300 K)	1.00 (3)	2.25 (3)	3.048 (4)	136 (2)
	(90 K)	0.97 (3)	2.21 (3)	3.009 (4)	139 (3)

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

Compound (1) at 90 K

Crystal data

$C_6H_7N_3O_3$
M_r = 169.15
Orthorhombic
Pbca
a = 12.756 (3) Å
b = 6.870 (1) Å
c = 15.740 (3) Å
V = 1379.4 (5) Å^3
Z = 8
D_x = 1.629 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 28 reflections
θ = 18–23°
μ = 0.133 mm^{-1}
T = 90.0 (1) K
Plate
0.48 × 0.40 × 0.36 mm
Light yellow

Data collection

Kuma KM-4 diffractometer
ω–θ scans
Absorption correction: none
1757 measured reflections
1757 independent reflections
1183 reflections with $I > 2\sigma(I)$

θ_{max} = 30.87°
$h = 0 \rightarrow 17$
$k = -9 \rightarrow 0$
$l = -20 \rightarrow 0$
2 standard reflections every 50 reflections intensity decay: 0.32%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.146
S = 1.153
1757 reflections
137 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0066P)^2 + 6.7646P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.423 e Å^{-3}
$\Delta\rho_{min}$ = −0.386 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 4. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (1) at 90 K*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a^i a^j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	0.37956 (17)	0.2110 (3)	0.51814 (15)	0.0171 (5)
N1	0.2860 (2)	0.2321 (4)	0.55015 (16)	0.0130 (5)
C2	0.2743 (2)	0.3028 (4)	0.6305 (2)	0.0129 (6)
C3	0.1766 (2)	0.3244 (4)	0.6659 (2)	0.0134 (6)
C4	0.0887 (2)	0.2740 (4)	0.6184 (2)	0.0117 (6)
C5	0.1008 (2)	0.2037 (4)	0.5367 (2)	0.0127 (6)
C6	0.2001 (2)	0.1838 (4)	0.5036 (2)	0.0134 (6)
N7	−0.0131 (2)	0.3087 (4)	0.65405 (17)	0.0128 (5)
N8	−0.0798 (2)	0.1559 (4)	0.65894 (17)	0.0136 (5)
O9	−0.04386 (18)	−0.0094 (3)	0.64919 (15)	0.0169 (5)
O10	−0.17175 (17)	0.1924 (4)	0.67684 (15)	0.0184 (5)
C11	−0.0585 (3)	0.5049 (5)	0.6559 (2)	0.0160 (6)

For both compounds, cell refinement: *Kuma Diffraction Software* (Kuma, 1997); data reduction: *Kuma Diffraction Software*; program(s) used to solve structures: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structures: *SHELXL97* (Sheldrick, 1997*b*); molecular graphics: *SHELXTL* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1027). Services for accessing these data are described at the back of the journal.

References

Bujak, M., Ejsmont, K., Kyzioł, J. B., Daszkiewicz, Z. & Zaleski, J. (1998). *Acta Cryst.* C**54**, 1945–1948.

Cameron, T. S., Cordes, R. E., Morris, D. G. & Murray, A. M. (1979). *J. Chem. Soc. Perkin Trans.* 2, pp. 300–302.

Coppens, P. & Lehman, M. S. (1976). *Acta Cryst.* B**32**, 1777–1784.

Daszkiewicz, Z., Domański, A. & Kyzioł, J. B. (1994). *Org. Prep. Proced. Int.* **26**, 337–341.

Kuma (1997). *Kuma Diffraction Software.* Version KM4*b*8. Kuma Diffraction, Wrocław, Poland.

Nakai, H., Saito, T. & Yamakawa, M. (1988). *Acta Cryst.* C**44**, 533–535.

Sheldrick, G. M. (1990). *SHELXTL.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1997*a*). *SHELXS97. Program for the Solution of Crystal Structures.* University of Göttingen, Germany.

Sheldrick, G. M. (1997*b*). *SHELXL97. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Wang, Y., Blessing, R. H., Ross, F. K. & Coppens, P. (1976). *Acta Cryst.* B**32**, 572–578.

Zaleski, J., Daszkiewicz, Z. & Kyzioł, J. (1999). *Acta Cryst.* C**55**, 691–693.

Acta Cryst. (1999). C**55**, 1295–1297

(1*R*,2*R*,3*R*,3′*R*,4*R*)-3-[(2-Furyl)hydroxymethyl]-1,7,7-trimethylbicyclo[2.2.1]heptan-2-ol, a new camphor-based chiral auxiliary

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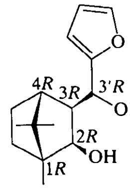
Abstract

Camphor chiral derivatives have proven to be very powerful chiral adjuvants in organic synthesis. The absolute molecular configuration of the title compound, $C_{15}H_{22}O_3$, has been determined due to the chiral

character of the (1*R*)-(+)-camphor. An infinite one-dimensional hydrogen-bonding chain along the [010] direction [O···O 2.747 (3) Å] and an intramolecular hydrogen bond [O···O 2.770 (3) Å] have been found.

Comment

Due to their great structural variety and availability, terpenes, as the readily available (1*R*)-(+)-camphor and its derivatives, might be used as a natural source for the synthesis of chiral compounds (Oppolzer, 1987). Much effort has gone into seeking simple and efficient synthesis routes to inexpensive chiral auxiliaries (Vasconcellos *et al.*, 1991, 1992; Cabral *et al.*, 1996; Costa *et al.*, 1997; Dumas *et al.*, 1997; Almquist *et al.*, 1997). This effort has resulted in the synthesis of new 1,3-diols (Mellão & Vasconcellos, 1996; Vasconcellos *et al.*, 1998). In order to improve the study of new chiral tridentate ligand systems, the title compound, (I), was synthesized from (1*R*)-(+)-camphor (Vasconcellos *et al.*, 1998) and its crystal structure determined.



(I)

The title compound (Fig. 1) adopts a conformation in which the angle between the least-squares plane through C2—C6—C5—C3 of the bicyclic ring [$5.25(1)x - 3.68(2)y - 8.68(1)z = 2.62(2)$; r.m.s. deviation = 0.02 Å] and the least-squares plane of the furan ring [$-0.22(1)x + 0.65(2)y + 11.06(1)z = 1.25(1)$; r.m.s. deviation = 0.004 Å] is 50.7 (1)°; the key torsion angles are given in Table 1. The bond distances and angles are within the normally expected ranges; the bond lengths in the furan ring show some π-bond character.

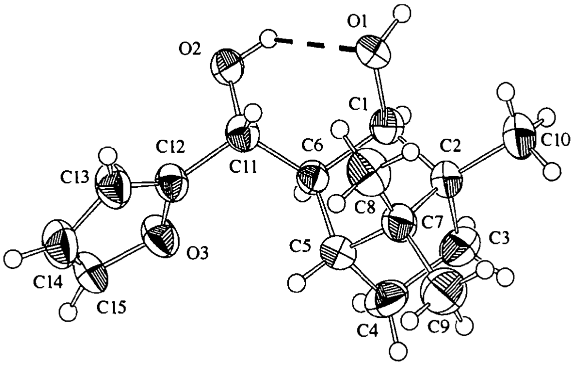


Fig. 1. *ZORTEP* (Zsolnai, 1994) view of the title compound with the crystallographic numbering scheme, showing 40% probability ellipsoids. The intramolecular hydrogen bond is shown with a dashed line.

In the title compound, an intermolecular O1···O2^{i} hydrogen bond [2.747 (3) Å; symmetry code: (i) $2-x, \frac{1}{2}+y, -z$] forms a one-dimensional infinite zigzag chain along the [010] direction, similar to the hydrogen-bonding packing for (1*R*)-(+)-camphor semicarbazone (Robinson *et al.*, 1994). The intramolecular O1···O2 hydrogen bond [2.770 (3) Å] forms a pseudo-six-membered ring. The geometric hydrogen-bonding parameters are given in Table 2.

Experimental

Details of the synthesis of (I) have been published elsewhere (Vasconcellos *et al.*, 1998). Single crystals suitable for X-ray analysis were obtained by slow evaporation from a hexane solution.

Crystal data

$C_{15}H_{22}O_3$
$M_r = 250.33$
Monoclinic
$P2_1$
$a = 7.581(1)$ Å
$b = 8.663(1)$ Å
$c = 11.475(1)$ Å
$\beta = 106.44(1)°$
$V = 722.8(1)$ Å^3
$Z = 2$
$D_x = 1.150$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 19 reflections
$\theta = 9.81–18.59°$
$\mu = 0.079$ mm^{-1}
$T = 293(2)$ K
Prism
0.15 × 0.10 × 0.08 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1934 measured reflections
1859 independent reflections
966 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.013$
$\theta_{\rm max} = 28.97°$
$h = -9 \rightarrow 10$
$k = 0 \rightarrow 11$
$l = -15 \rightarrow 0$
3 standard reflections
frequency: 120 min
intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.122$
$S = 0.905$
1859 reflections
163 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0616P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.15$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.19$ e Å^{-3}

Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: correct enantiomer chosen on the basis of the known absolute configuration of the (1*R*)-(+)-camphor used in the synthesis

Table 1. *Selected torsion angles (°)*

C4—C3—C3′—O2	−177.4 (3)	O2—C3′—C11—C12	−96.7 (5)
C2—C3—C3′—O2	61.4 (3)	C3—C3′—C11—C12	142.5 (4)
C4—C3—C3′—C11	−57.8 (4)	O2—C3′—C11—O3	83.3 (3)
C2—C3—C3′—C11	−179.0 (3)	C3—C3′—C11—O3	−37.6 (4)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—HO2···O1	0.86	2.14	2.770 (3)	130
O1—HO1···O2^i	0.88	1.88	2.747 (3)	171

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

All H atoms have been placed in calculated positions, except for the hydroxyl H atoms which were located from a difference Fourier map. All H atoms were refined using a riding model with U equal to $1.2U_{eq}$ of the parent atom, except for methyl groups ($1.5U_{eq}$). The range of idealized C—H bond distances is 0.93–0.98 Å.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *XCAD*4 (Harms, 1997). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* and *XPMA* (Zsolnai, 1994). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1146). Services for accessing these data are described at the back of the journal.

References

Almquist, F., Torstensson, L., Gudmundsson, A. & Frejd, T. (1997). *Angew. Chem. Int. Ed. Engl.* **36**, 376–377.
Cabral, L. M., Costa, P. R. R., Vasconcellos, M. L. A. A., Barreiro, E. J. & Castro, R. N. (1996). *Synth. Commun.* **26**, 3671–3676.
Costa, P. R. R., Cabral, L. M., Alencar, K., Schmidt, L. L. & Vasconcellos, M. L. A. A. (1997). *Tetrahedron Lett.* **38**, 7021–7024.
Dumas, F., Alencar, K., Mahuteau, J., Barbero, M. J. L., Miet, C., Gérard, F., Vasconcellos, M. L. A. A. & Costa, P. R. R. (1997). *Tetrahedron Asymmetry*, **8**, 579–583.
Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Harms, K. (1997). *XCAD*4. *Program for the Lp Correction of CAD-*4 *Diffractometer Data*. University of Marburg, Germany.
Mellão, M. L. & Vasconcellos, M. L. A. A. (1996). *Tetrahedron Asymmetry*, **7**, 1607–1610.
Oppolzer, W. (1987). *Tetrahedron*, **43**, 1969–2004.
Robinson, P. D., Meyers, C. Y. & Kolb, V. M. (1994). *Acta Cryst.* C**50**, 732–734.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Vasconcellos, M. L. A. A., d'Angelo, J., Desmaele, D., Costa, P. R. R. & Potin, D. (1991). *Tetrahedron Asymmetry*, **2**, 353–356.
Vasconcellos, M. L. A. A., Desmaele, D., Costa, P. R. R. & d'Angelo, J. (1992). *Tetrahedron Lett.* **33**, 4921–4922.
Vasconcellos, M. L. A. A., Mellão, M. L., Barreiros, U. G., Branco, M. C., Gambardella, M. T. P., Ramos, O. F. & Ribeiro, M. (1998). *Synth. Commun.* **28**, 4077–4085.
Zsolnai, L. (1994). *XPMA* and *ZORTEP*. *Molecular Geometry and Graphics Programs*. University of Heidelberg, Germany.

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N-Cyclohexyl-6α,7β-dihydroxyvouacapan-17β-carboxamide†

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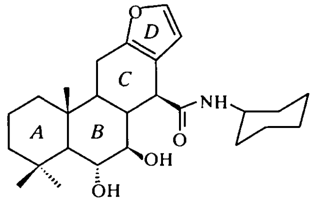
Abstract

The crystal and molecular structures of the title compound (CHAD, $C_{26}H_{39}NO_4$), which is a synthetic derivative of 6α,7β-dihydroxyvouacapan-17β-oic acid (ADV) isolated from *Pterodon polygalaeflorus* Benth, have been determined. *P. polygalaeflorus* Benth is a traditional plant of Brazilian medicinal culture.

Comment

6α,7β-Dihydroxyvouacapan-17β-oic acid (ADV) is a natural furanditerpene extract isolated from the fruits of Sucupira Branca or Faveira (*Pterodon polygalaeflorus* Benth) as reported by Fascio *et al.* (1976). Its structure has been determined previously (Ruggiero *et al.*, 1997). This vouacapan furanditerpene exhibits both anti-inflammatory and analgesic activities. In order to study structure–activity relationships of the vouacapanes, some amides derived from ADV were prepared, including 6α,7β-dihydroxy-*N*-cyclohexylvouacapan-17β-amide (Maltha *et al.*, 1999). The crystallographic study of *N*-cyclohexyl-6α,7β-dihydroxyvouacapan-17β-carboxamide (CHAD) is reported here.



CHAD

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† Alternative name: *N*-cyclohexyl-1,5,5a,6,7,7a,8,9,10,11,11a,11b-dodecahydro-6,7-dihydroxy-8,8,11a-trimethylphenanthro[3,2-*b*]furan-17-carboxamide.