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

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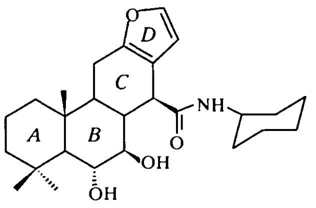
Abstract

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.

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As expected, CHAD has a skeletal conformation (Fig. 1) with bond distances and angles similar to those of ADV (Ruggiero *et al.*, 1997). A least-squares superposition of the title compound and ADV resulted in an r.m.s. deviation of 0.072 Å. A comparison realised with only the furanditerpene skeleton of both molecules resulted in an r.m.s. deviation of 0.042 Å. The bond distances and angles are within normally expected ranges. According to puckering parameters (Cremer & Pople, 1975) and ring torsion angles, cyclohexane rings *A* [q_2 = 0.084 (5) Å, q_3 = 0.537 (5) Å, Q = 0.543 (5) Å, θ = 8.9 (5)° and φ = 255 (3)°] and *B* [q_2 = 0.091 (4) Å, q_3 = 0.608 (4) Å, Q = 0.615 (4) Å, θ = 8.5 (3)° and φ = 311 (2)°] form a slightly distorted double-chair. Ring *C* has a distorted half-chair character [q_2 = 0.376 (4) Å, q_3 = 0.294 (4) Å, Q = 0.477 (4) Å, θ = 52.0 (4)° and φ = 38.7 (5)°]. The conformation of the cyclohexane ring on the amide group is almost a pure chair [q_2 = 0.013 (7) Å, q_3 = −0.561 (7) Å, Q = 0.561 (7) Å, θ = 178.7 (7)° and φ = 210 (28)°]. The furan ring is planar, with a maximum deviation from the least-squares plane of 0.002 Å [$13.63 (4)x - 0.423 (17)y + 7.073 (12)z = 12.126 (7)$]. The bond lengths in the furan ring and the amide group indicate some π-bond character. As in normal diterpenes, the junction between the three six-membered carbon rings is *trans*. The torsion angles C3—C4—C5—C6, C2—C1—C10—C9 and C6—C7—C8—C14 have values of 174.0 (4), 169.9 (4) and −176.0 (3)°, respectively (the expected value is 180°). The C1—C10—C9—C11 torsion angle is 56.3 (4)° (the expected value is 60°).

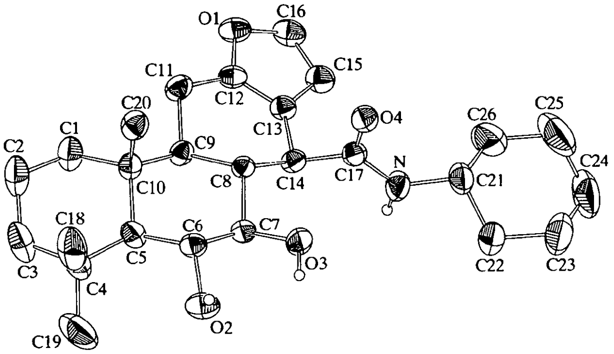

Fig. 1. *ZORTEP* (Zsolnai, 1994) view of the title compound with the crystallographic numbering scheme, showing 40% probability ellipsoids. All H atoms, except those attached to O and N atoms, have been omitted for clarity.

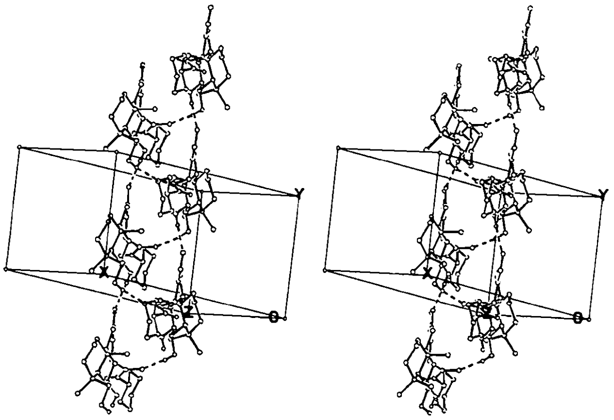

Fig. 2. Stereoview of the two one-dimensional chains along the *b* axis. Hydrogen bonds are illustrated by dotted lines (Zsolnai, 1994).

Each molecule in the crystal is involved in one intramolecular and two intermolecular hydrogen bonds (Table 1). An intermolecular hydrogen bond between the hydroxyl O3 and furan O1 atoms links the molecules in an infinite chain in the [010] direction. Hydrogen bonds between the carbonyl O4 and hydroxyl O3 atoms make a zigzag between the infinite one-dimensional chains (Fig. 2).

Experimental

Good single crystals, suitable for X-ray analysis, were obtained by slow evaporation from an ethanol solution.

Crystal data

$C_{26}H_{39}NO_4$
M_r = 429.58
Monoclinic
$C2$
a = 29.277 (2) Å
b = 8.9805 (8) Å
c = 9.8486 (6) Å
β = 106.2480 (1)°
V = 2486.0 (3) Å^3
Z = 4
D_x = 1.15 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 9.79–18.36°
μ = 0.076 mm^{-1}
T = 293 (2) K
Prism
0.50 × 0.30 × 0.13 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2743 measured reflections
2689 independent reflections
2119 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.011
$\theta_{\rm max}$ = 26.29°
$h = -36 \rightarrow 0$
$k = -11 \rightarrow 0$
$l = -11 \rightarrow 12$
3 standard reflections
frequency: 120 min
intensity decay: 0.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.133
S = 1.145
2689 reflections
280 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 1.2514P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ = 0.012
$\Delta\rho_{\rm max}$ = 0.186 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.258 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—HO3···O2	0.80	2.35	2.728 (4)	110
O2—HO2···O4^{i}	0.89	1.92	2.791 (3)	169
O3—HO3···O1^{ii}	0.80	2.18	2.918 (4)	154

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

The hydroxyl and amide H atoms were determined from a difference Fourier map. All H atoms were refined as riding with a common atomic displacement parameter of 0.076 Å^2.

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: SX1054). Services for accessing these data are described at the back of the journal.

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The twisted structure of 9-(4-cyanophenyl)carbazole

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(Received 13 January 1999; accepted 23 April 1999)

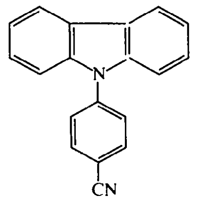
Abstract

The crystal structure determination of the title compound, $C_{19}H_{12}N_2$, has been undertaken with a view to understanding the nature of the fluorescence band of the system. There is a significant twist between the cyanophenyl and carbazolyl moieties.

Comment

A number of electron donor–acceptor (EDA) molecules exhibit, in addition to the locally excited (LE) fluorescence, a second anomalous fluorescence band at longer wavelength that originates from a twisted intramolecular charge-transfer (TICT) state in which the donor and acceptor moieties are mutually orthogonal to each other (Rotkiewicz *et al.*, 1973; Rettig, 1986). An alternative mechanism in terms of a change in the pyramidalization of the amino N atom has been proposed recently to account for this phenomenon (Il'ichev *et al.*, 1998).

The title compound, (I), displays a single fluorescence band that has been tentatively assigned as an emission from the TICT state (Rettig & Zander, 1982). Since the ground-state structure of a system often determines the excited-state conformation of the molecule, we have determined the crystal structure of this system.



(I)

The molecule (I) exhibits a torsion angle C12—N1—C13—C18 of 48.3 (2)° (Fig. 1). This observation suggests that the electron donor (carbazolyl) and acceptor (cyanophenyl) moieties are twisted with respect to each

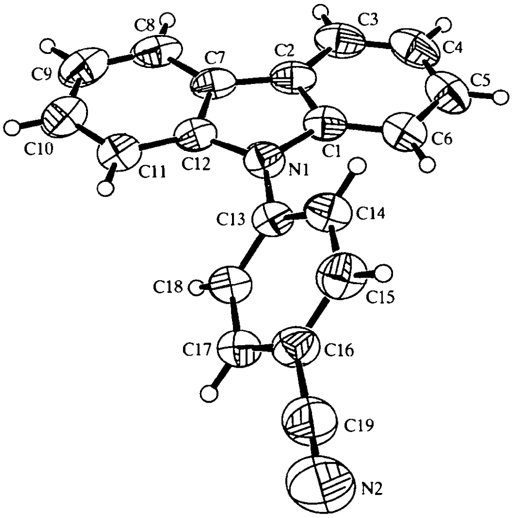


Fig. 1. A view of the molecular structure of (I) showing the atom labels. Displacement ellipsoids are shown at the 40% probability level.

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