Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *DATAP* (Coppens, Leiserowitz & Rabinovich, 1965). Molecular graphics: *ORTEPII* (Johnson, 1976).

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precursors of vegetal origin, in view of the diminishing availability of whales. The (+)-isomer of the title compound, (1), has proved to be a powerful synthetic tool for the preparation of *trans*-decalin derivatives with a strong ambergris odor (Gaitán, 1996). The title compound can be isolated by chemical resolution of the racemic mixture, which is obtained by cross-sectioning the trunk of *Eperua purpurea* (De Santis & Medina, 1981). The (−)-enantiomeric constituent and the (+)-isomer are expected to have the same structural characteristics, except for their configuration. This paper reports the results of an X-ray crystal structure study of (1), carried out in order to determine the conformation and crystal packing of the molecule.

(1)
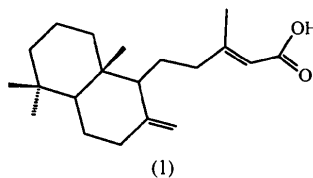
A view of the molecular structure is shown in Fig. 1. The (−)-enantiomer was chosen in accord with the high negative value for the specific optical rotation of the ethanolic solution of the crystals obtained after resolution. The C—C bond distances of the *trans*-decalinic fragment of (1) are similar to those found (single-bond range 1.497–1.575 Å) in two related *trans*-decalinic molecular fragments (Singh, Gray, Skelton, Waterman & White, 1991; Grant, Hanton & Wong, 1995), while the bond lengths C8—C17 [1.324 (5) Å]

(13*E*)-(−)-Labda-8(17),13-dien-15-oic Acid

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Abstract

In the title compound, $C_{20}H_{32}O_2$, the two six-membered rings have a $^1C_4$ chair conformation and the rings are *trans*-fused. The crystal structure is stabilized by approximately symmetric $R_2^2(8)$-type hydrogen bonds.

Comment

As part of our efforts towards the introduction of new starting materials for the synthesis of ambergris odorants, we have directed our attention to synthetic
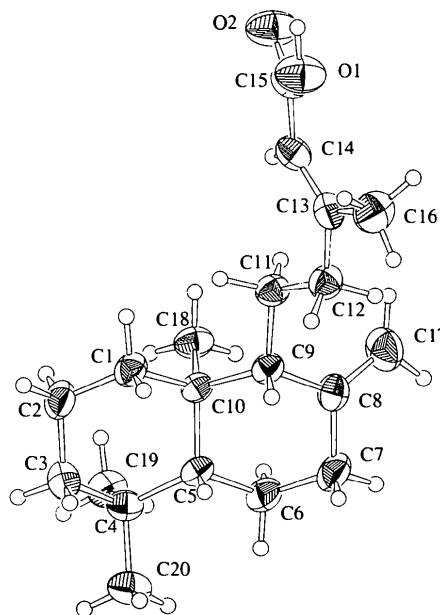

Fig. 1. Molecular diagram of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 35% probability level.

and C13—C14 [1.328 (5) Å] show the expected double-bond character and compare well with previous values (average 1.322 Å).

The six-membered rings are *trans*-fused. According to the puckering parameters [for C1, C10, C5, C4, C3, C2: $\psi^2 = -41$ (6), $\theta^2 = 2.9$ (4)°, $Q = 0.545$ (3) Å; for C9, C8, C7, C6, C5, C10: $\psi^2 = -52$ (4), $\theta^2 = 4.5$ (4)°, $Q = 0.597$ (4) Å] (Cremer & Pople, 1975), the conformations of both rings can be described as the chair form ($^1C_4$) (Boeyens, 1978).

In the crystal structure, each pair of molecules is linked by an approximately symmetric $R_2^2(8)$-type hydrogen bond (Etter, 1990) to form dimeric species, where the mean plane containing all atoms of the intermolecular $R_2^2(8)$ hydrogen bond is approximately perpendicular to the direction of the *a* axis (Fig. 2). In this carboxylic acid dimer, the C—O distances [C15—O1 1.259 (4) and C15—O2 1.270 (5) Å] are equal and the carboxylic groups are related by a twofold axis, where the corresponding H atoms, located in the latest difference Fourier maps, are both on the twofold axis. The average O—H distance is 1.32 (10) Å. The O1···O1(−x+2, y, −z+2) and O2···O2(−x+2, y, −z+2) intermolecular distances are 2.605 (4) and 2.648 (4) Å, respectively.
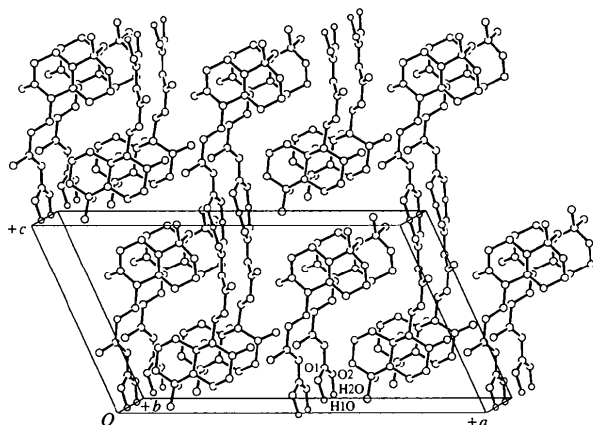

Fig. 2. A perspective drawing of the crystal packing showing the $R_2^2(8)$ hydrogen bond between carboxylic acid groups.

Experimental

Resolution was achieved by treatment of a hot ethanolic solution of the racemate (1.0 mmol) with (*R*)-(+)-α-methyl-benzylamine (0.5 mmol, Aldrich) and heating the mixture for 3 min. The solvent was evaporated to dryness and the colorless oil obtained redissolved in hexane, to give fine white needles immediately. Left in the refrigerator overnight, the crystals were filtered and recrystallized twice from hexane. The salt obtained had $[\alpha]_D^{25}$ −29° (c = 2.0, EtOH). The acid was recovered in the usual manner and crystallized from ethanol solution by slow evaporation of the solvent at room temperature, to give colorless crystals of the title compound with $[\alpha]_D^{25}$ −47.68° (c = 2.0, EtOH). A crystal suitable for X-ray diffraction was mounted on a glass fiber.

Crystal data

$C_{20}H_{32}O_2$	Mo $K\alpha$ radiation
$M_r = 304.46$	$\lambda = 0.71069$ Å
Monoclinic	Cell parameters from 25
$C2$	reflections
$a = 24.967$ (3) Å	$\theta = 37.92–41.33°$
$b = 6.075$ (2) Å	$\mu = 0.068$ mm^{-1}
$c = 13.232$ (2) Å	$T = 293$ (2) K
$\beta = 113.134$ (11)°	Rod
$V = 1845.7$ (6) Å^3	0.69 × 0.36 × 0.30 mm
$Z = 4$	Colorless
$D_x = 1.096$ Mg m^{-3}	
D_m not measured	

Data collection

Rigaku AFC-7S diffractom-eter	1525 reflections with $I > 2\sigma(I)$
$\omega–2\theta$ scans	$R_{int} = 0.0063$
Absorption correction:	$\theta_{max} = 25°$
ψ scan (North, Phillips & Mathews, 1968)	$h = 0 \rightarrow 29$
	$k = 0 \rightarrow 7$
$T_{min} = 0.94$, $T_{max} = 1.00$	$l = -15 \rightarrow 14$
1842 measured reflections	3 standard reflections
1796 independent reflections	every 150 reflections intensity decay: 0.74%

Refinement

Refinement on F^2	Extinction correction:
$R[F^2 > 2\sigma(F^2)] = 0.044$	SHELXL93 (Sheldrick,
$wR(F^2) = 0.163$	1993)
$S = 1.108$	Extinction coefficient:
1730 reflections	0.012 (3)
205 parameters	Scattering factors from
H atoms: see below	International Tables for
$w = 1/[\sigma^2(F_o^2) + (0.071P)^2 + 0.3371P]$	Crystallography (Vol. C)
where $P = (F_o^2 + 2F_c^2)/3$	Absolute configuration:
$(\Delta/\sigma)_{max} = 0.005$	Flack (1983)
$\Delta\rho_{max} = 0.153$ e Å^{-3}	Flack parameter = 0 (3)
$\Delta\rho_{min} = -0.169$ e Å^{-3}	

Table 1. *Selected geometric parameters (Å, °)*

O1—C15	1.259 (4)	C6—C7	1.534 (5)
O1—H1O	1.31 (13)	C7—C8	1.495 (4)
O2—C15	1.270 (5)	C8—C17	1.324 (5)
O2—H2O	1.33 (14)	C8—C9	1.520 (4)
C1—C2	1.516 (4)	C9—C11	1.537 (3)
C1—C10	1.545 (4)	C9—C10	1.562 (4)
C2—C3	1.502 (4)	C10—C18	1.524 (4)
C3—C4	1.525 (4)	C11—C12	1.518 (4)
C4—C19	1.534 (5)	C12—C13	1.501 (4)
C4—C20	1.547 (4)	C13—C14	1.328 (5)
C4—C5	1.563 (4)	C13—C16	1.496 (5)
C5—C6	1.528 (4)	C14—C15	1.465 (4)
C5—C10	1.558 (3)		
C15—O1—H1O	118 (5)	C8—C9—C11	113.9 (2)
C15—O2—H2O	115 (6)	C8—C9—C10	109.8 (2)
C2—C1—C10	113.5 (2)	C11—C9—C10	114.9 (2)

C3—C2—C1	111.4 (2)	C18—C10—C1	109.8 (2)
C2—C3—C4	114.2 (3)	C18—C10—C5	114.4 (2)
C3—C4—C19	110.7 (3)	C1—C10—C5	107.6 (2)
C3—C4—C20	107.0 (3)	C18—C10—C9	108.8 (2)
C19—C4—C20	106.9 (3)	C1—C10—C9	109.2 (2)
C3—C4—C5	108.2 (2)	C5—C10—C9	107.0 (2)
C19—C4—C5	114.3 (3)	C12—C11—C9	112.3 (2)
C20—C4—C5	109.5 (2)	C13—C12—C11	117.8 (3)
C6—C5—C10	111.6 (2)	C14—C13—C16	124.7 (3)
C6—C5—C4	113.5 (2)	C14—C13—C12	121.6 (3)
C10—C5—C4	116.7 (2)	C16—C13—C12	113.6 (3)
C7—C6—C5	110.8 (2)	C13—C14—C15	126.7 (3)
C8—C7—C6	110.6 (2)	O1—C15—O2	122.0 (3)
C17—C8—C7	121.2 (3)	O1—C15—C14	120.7 (3)
C17—C8—C9	125.8 (3)	O2—C15—C14	117.2 (3)
C7—C8—C9	112.9 (3)		

Intensity data were corrected for Lorentz and polarization effects. A semi-empirical absorption correction (North, Phillips & Mathews, 1968) based on azimuthal scans of several reflections was applied. The intensities of three reflections were measured after every 150 reflections; no variation was observed. The crystal structure was solved by using direct methods followed by conventional Fourier techniques and subsequent least-squares refinement. All non-H atoms were refined anisotropically. The H atoms were included in their found positions, except for those of the C3, C7 (C—H = 0.97 Å) and C5 (C—H = 0.98 Å) atoms, and those of the methyl group (C—H = 0.96 Å), which were included in calculated positions. All these H atoms were refined riding on their C atoms with a common isotropic displacement parameter. The two H atoms of the symmetrically hydrogen-bonded carboxylic dimer occupy two positions on a twofold axis. Thus, both positions were included in the last cycles and their y coordinates and isotropic displacement parameters were successfully refined.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1024). Services for accessing these data are described at the back of the journal.

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Hydrogen-Bonding Networks Involving Water in Amphiphilic N-Dodecanoyl-L-serine Monohydrate

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Abstract

The amphiphilic title compound, $C_{15}H_{29}NO_4.H_2O$, forms a head-to-tail bilayer packing arrangement in the crystal. The crystal structure shows an entangled network of hydrogen bonds in which the water molecule has a central position. A rarely observed methine donor hydrogen bridge was found.

Comment

Many crystal structure investigations aim to examine the intra- and intermolecular interactions of biologically relevant compounds. Among these, amino acids, peptides and their derivatives are of special interest. To overcome the unfavourable ionic interactions due to the zwitterionic appearance of most of the amino acid compounds in the crystal, we linked L-serine with lauric acid to give N-dodecanoyl-L-serine (Fig. 1). Thus, the amino acid retains its normal properties in peptides without any ammonium-to-carboxylate attractions. We present here the crystal structure of this amino acid derivative, (I), which contains a network of hydrogen bonds. Spe-