Instrumental Achievements

Crystal Structure of *ent*-3β,7α-Dihydroxy-18-acetoxykaur-16-ene (linearol)

Tuncer HÖKELEK*†, Emine KILIÇ and Atilla ÖKTEMER****

**Hacettepe University, Department of Physics, 06532 Beytepe, Ankara, Turkey*
***Ankara University, Department of Chemistry, 06130 Tandoğan, Ankara, Turkey*

ent-Kaurene diterpenes have been studied in microbiological transformation reactions[1,2], the identification of new anti-HIV and antitumor agents[3] and biological-activity research.[4]

Sideritis sipylea Boiss is distributed mainly in western and southern Turkey. The extract of this plant with light petroleum (40 – 60°C) was chromatographed [silica gel (Merck, 70 – 230 mesh)] with a light petroleum–diethyl ether–methanol (1:1:1) mixture and the known diterpenes; siderol, linearol, epicandicandiol and foliol were eluted. The physical and spectroscopic properties of these compounds were identical with those reported in the literature.[5]

The structure of linearol (Fig. 1) [$C_{22}H_{34}O_4$, m.p. 492 K, $[\alpha]_D^{20}$=–67.0° (*c*=1.0 ethanol)] was clarified to be (–)-18-acetoxy-kaur-16-ene by means of an X-ray crystallographic analysis (Fig. 2) and its optical rotation. The torsional angles for rings A, B and C are shown in Fig. 3. Rings A, B and C have chair conformations while the conformation of ring D is a half-chair. Ring C is especially distorted. The puckering parameter of the bridging D ring, the angle between planes C14–C13–C16–C15 and C14–C8–C15, is 40.6(3)°.

Ring junction A–B is *trans*, while B–C is *cis*. The nearest distances between the moieties are C18···C19=2.427(5), O3···C19=2.784(6), C19···C20=3.244(5),

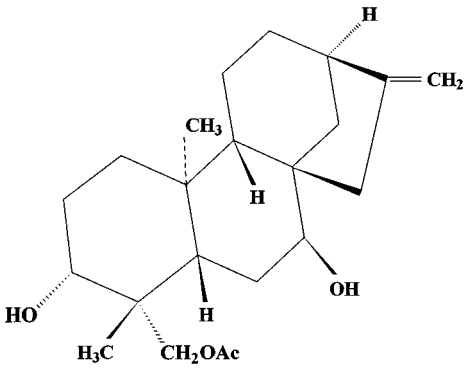

Fig. 1 Chemical diagram.

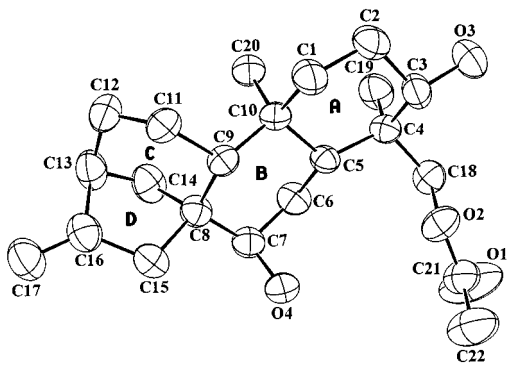

Fig. 2 Molecular structure of the title compound with the atom-numbering scheme. The thermal ellipsoids are drawn at the 50% probability level.

Table 1 Crystal and experimental data

Formula: $C_{22}H_{34}O_4$
Formula weight=362.51
Crystal system: monoclinic
Space group: $P2_1$ Z=2
a=7.785(1)Å
b=11.670(1)Å
c=11.799(2)Å
β=107.35(1)°
V=1023.2(2)Å^3
D_x=1.176 g/cm^3
μ(Mo K$_\alpha$)=0.07 mm^{-1}
T=293 K
F(0 0 0)=396
Colorless
Cyrstal size: 0.20×0.25×0.30 mm
λ(Mo K$_\alpha$)=0.71073Å
R=0.0631
R_W=0.0837
No. of reflections used=2003
No. of parameters=235
Goodness-of-fit=1.21
$(\Delta/\sigma)_{max}$=0.01
$\Delta\rho_{max}$=0.44 e/Å^3
$\Delta\rho_{min}$=–0.38 e/Å^3
Measurements: Enraf-Nonius CAD-4 diffractometer
Program system: CAD-4 EXPRESS Software
Structure determination: MolEN
Treatment of hydrogen atoms: geometric calculation
Refinement: full matrix least-squares (MolEN)

† To whom correspondence should be addressed.

Table 2 Final atomic coordinates and equivalent isotropic thermal parameters

Atom	*x*	*y*	*z*	B_{eq}/Å^2
O1	0.4648(5)	0.0073(5)	0.3876(4)	10.1(1)
O2	0.2286(4)	0.0310(3)	0.4480(2)	4.66(6)
O3	-0.0119(4)	-0.1888(3)	0.5496(3)	5.27(7)
O4	0.3567(4)	0.2986(3)	0.6133(2)	4.33(6)
C1	-0.0931(5)	0.0830(4)	0.7009(4)	4.56(9)
C2	-0.1098(5)	-0.0430(4)	0.6640(4)	4.96(9)
C3	0.0024(5)	-0.0690(4)	0.5825(4)	4.06(8)
C4	0.2012(5)	-0.0413(3)	0.6356(3)	3.57(7)
C5	0.2155(4)	0.0852(3)	0.6801(3)	3.11(6)
C6	0.4103(5)	0.1294(4)	0.7319(4)	3.98(8)
C7	0.4131(5)	0.2591(4)	0.7355(3)	3.88(8)
C8	0.2954(5)	0.3105(4)	0.8069(3)	3.62(7)
C9	0.1080(4)	0.2526(3)	0.7716(3)	3.19(7)
C10	0.1022(4)	0.1190(4)	0.7639(3)	3.44(7)
C11	-0.0070(5)	0.3062(4)	0.8462(3)	4.29(8)
C12	0.0924(6)	0.3328(5)	0.9749(4)	5.2(1)
C13	0.2744(6)	0.3884(4)	0.9889(4)	4.9(1)
C14	0.3902(6)	0.3069(5)	0.9423(4)	4.81(9)
C15	0.2749(6)	0.4421(4)	0.7905(4)	4.29(8)
C16	0.2531(6)	0.4890(4)	0.9023(4)	4.62(9)
C17	0.2265(7)	0.5977(5)	0.9268(5)	5.8(1)
C18	0.2946(5)	-0.0542(4)	0.5394(3)	4.16(8)
C19	0.2965(7)	-0.1306(4)	0.7311(4)	5.2(1)
C20	0.1630(7)	0.0608(4)	0.8857(3)	4.92(9)
C21	0.3274(6)	0.0547(4)	0.3779(4)	5.11(9)
C22	0.2486(8)	0.1436(6)	0.2891(5)	7.3(1)

$B_{eq}=(8\pi^2/3)\Sigma_i\Sigma_j U_{ij}(a_i^* a_j^*)(\boldsymbol{a}_i\cdot\boldsymbol{a}_j)$.

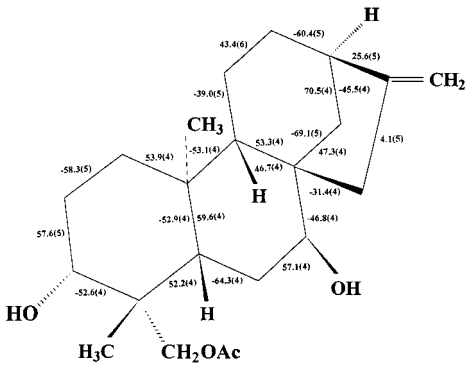

Fig. 3 Torsional angles.

C11···C14=2.959(5) and C14···C20=3.335(6).

The bond lengths and angles (Table 3) are generally in good agreement with the chemically expected values.

Table 3 Bond distances (Å) and angles (°)

C7-C8	1.538(6)	C3-C2	1.509(7)
C8-C9	1.548(5)	C3-O3	1.448(6)
C8-C15	1.549(6)	C2-C1	1.528(7)
C8-C14	1.550(5)	C15-C16	1.484(7)
C7-C6	1.514(6)	C11-C12	1.516(5)
C7-O4	1.453(5)	C18-O2	1.445(5)
C9-C10	1.561(6)	C17-C16	1.331(7)
C9-C11	1.563(6)	C13-C16	1.534(7)
C10-C5	1.558(6)	C5-C6	1.547(5)
C10-C1	1.539(5)	O2-C21	1.314(6)
C10-C20	1.530(6)	C14-C13	1.519(8)
C4-C5	1.560(6)	C4-C18	1.526(6)
C4-C3	1.520(5)	C4-C19	1.553(6)
C21-C22	1.472(7)	C12-C13	1.522(7)
C21-O1	1.180(7)		
C7-C8-C9	110.4(3)	O2-C21-O1	121.5(4)
C7-C8-C15	112.0(4)	C22-C21-O1	124.9(5)
C7-C8-C14	112.1(3)	C11-C12-C13	112.1(4)
C9-C8-C15	110.2(3)	C12-C13-C16	110.0(4)
C9-C8-C14	112.7(3)	C15-C16-C17	127.7(5)
C15-C8-C14	98.9(3)	C15-C16-C13	106.9(4)
C8-C7-C6	113.6(4)	C5-C10-C20	114.2(3)
C8-C7-O4	111.4(3)	C1-C10-C20	108.5(4)
C6-C7-O4	106.9(3)	C5-C4-C3	107.5(3)
C8-C9-C10	117.4(3)	C5-C4-C18	110.0(3)
C8-C9-C11	108.9(3)	C5-C4-C19	114.8(3)
C10-C9-C11	114.9(3)	C3-C4-C18	108.9(3)
C9-C10-C5	106.0(3)	C3-C4-C19	111.4(4)
C9-C10-C1	107.9(3)	C18-C4-C19	104.1(4)
C9-C10-C20	113.2(3)	C10-C5-C4	117.2(3)
C5-C10-C1	106.6(3)	C10-C5-C6	109.7(3)
C4-C3-C2	114.0(3)	C4-C5-C6	114.3(3)
C4-C3-O3	107.6(3)	C7-C6-C5	110.4(3)
C2-C3-O3	110.5(4)	C9-C11-C12	116.1(3)
C3-C2-C1	110.9(4)	C4-C18-O2	110.2(3)
C8-C15-C16	106.9(4)	C14-C13-C12	109.1(4)
C10-C1-C2	112.9(3)	C14-C13-C16	101.3(4)
C18-O2-C21	118.1(3)	O2-C21-C22	113.5(4)
C8-C14-C13	101.8(3)	C17-C16-C13	125.4(5)

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