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A LUPENEDIOL FROM *EUONYMUS REVOLUTUS*

N. SAVITRI KUMAR, P. MANGALA MUTHUKUDA and MOHAMED I. M. WAZEER*

Department of Chemistry, University of Peradeniya, Sri Lanka; *Department of Chemistry, University of Petroleum & Minerals, Dhahran, Saudi Arabia

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Key Word Index—*Euonymus revolutus*; Celastraceae; triterpenes; 2α,3α-dihydroxy-lup-20(29)-en-28-oic acid.

Abstract—A new lupane derivative isolated from *Euonymus revolutus* (Celastraceae) has been established to be 2α,3α-dihydroxy-lup-20(29)-en-28-oic acid on the basis of chemical interconversions and spectroscopic data.

INTRODUCTION

Three species of *Euonymus*, *E. revolutus*, *E. thwaitesii* and *E. walkeri* belonging to the family Celastraceae are endemic to Sri Lanka. *E. revolutus* Wight is a tree of moderate size found in the upper montane forests of Sri Lanka [1]. Species of *Euonymus* have found many medicinal applications [2]. We have recently reported the isolation of three new D:A-friedooleanane derivatives from the stem bark extract of *E. revolutus* [3]. In this paper we report the structure elucidation of a new lupene, 2α,3α-dihydroxy-lup-20(29)-en-28-oic acid isolated from the same extract. A D:A-friedooleanane-2,3-diol isolated from *Mortonia greggi* [4] is the only vicinal diol which has thus far been reported from the Celastraceae. Lupene-2,3-diols have also been isolated from *Pterocarpus santalinus* (Leguminosae) [5]. This paper is the first report of the occurrence of lupanes in the genus *Euonymus*.

RESULTS

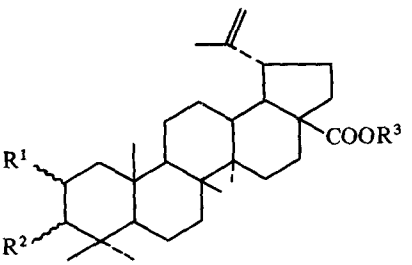
Column chromatography and preparative TLC of a highly polar fraction from the cold CH_2Cl_2 extract of the stem bark of *Euonymus revolutus* yielded a triterpene diol (**1**). The IR spectrum of compound **1** indicated the presence of hydroxy groups and a carboxylic acid group (ν_{max} 3600–3100, 3400, 1690 cm^{-1}). Treatment of compound **1** with diazomethane yielded the methyl ester (**2**) ($C_{31}H_{50}O_4$) confirming the presence of a carboxylic acid group. Acetylation of **1** gave a diacetate (**3**) [^{1}H NMR: δ5.21 (1H, *m*), 4.95 (1H, *d*), 2.12 (3H, *s*) and 1.95 (3H, *s*)] indicating that there were two secondary hydroxy groups in **1**.

Two doublets in the ^{1}H NMR spectrum at δ4.72 and 4.60 due to a single proton each and a broad singlet at 1.69 due to a vinylic methyl suggested that triterpene **1** had a lup-20(29)-ene system [6]. The half height width of the multiplet at δ3.92 ($W_{1/2}$ = 22 Hz) due to a carbinol methine proton indicated that this proton had an axial orientation. Hence the hydroxy group at this position was equatorial. The signal due to the second carbinol methine proton at δ3.40 was a doublet with J = 4 Hz, showing the presence of an axial hydroxy group. High resolution mass spectroscopy of the diacetate **3** gave a molecular formula $C_{34}H_{52}O_6$. The fragment at m/z 189 also supported the presence of a lup-20(29)-ene derivative [7]. The intense peak at m/z 233 suggested that the two hydroxy groups were at rings A and/or B.

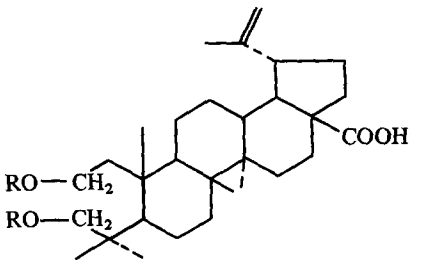
The lupene **1** was oxidized readily by periodic acid suggesting that the compound was a vicinal diol. The periodate oxidation product was reduced with sodium borohydride to the seco diol **4** which was then acetylated to give the secodiacetate **5** [$(C_{34}H_{54}O_6)$, ν_{max} 1780, 1690 and 1235 cm^{-1}]. The ^{1}H NMR spectrum of compound **5** showed two one proton doublets at δ4.04 and 3.76 (J = 12 Hz) due to the protons at C-3. The protons at C-2 appeared as one proton multiplets at δ4.22 and 4.10 respectively, which collapsed to two doublets (J = 11 Hz) on irradiation at δ1.90. The *cis*-vicinal nature of the diol system was confirmed by formation of the isopropylidene derivative **6** ($C_{33}H_{52}O_4$) from the lupene **1**. The two methyl groups of the isopropylidene residue appeared as singlets at δ1.48 and 1.29 respectively in the ^{1}H NMR spectrum of compound **6**. A one proton multiplet at δ4.15 and a doublet at 3.69 due to the protons at C-2 and C-3 respectively, were also observed in addition to the signals of the lup-20(29)-ene system.

Partial acetylation of the methyl ester **2** was effected by treating **2** with acetic anhydride–pyridine at room temperature for 2 hr. The monoacetate **7** ($C_{33}H_{52}O_5$) was obtained. The ^{1}H NMR spectrum of compound **7** showed that the hydroxy group with the equatorial orientation had been preferentially acetylated. The carbinol methine proton of compound **1** which appeared as a multiplet at δ3.92 in the ^{1}H NMR spectrum was shifted downfield to give a multiplet at δ5.23 ($W_{1/2}$ = 20 Hz) in the ^{1}H NMR spectrum of **7**, while the chemical shift of the second carbinol methine proton remained unaltered.

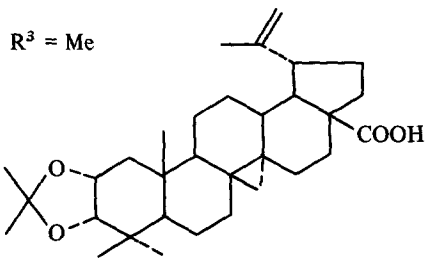
The monoacetate **7** was oxidized with CrO_3 in pyridine to give the keto-lupene **8** ($C_{33}H_{50}O_5$). IR spectral evidence supported the presence of an α-acetoxy keto system (ν_{max} 1735, 1725 cm^{-1}). The double doublet at δ5.58 (J = 14 Hz and 6 Hz) in the ^{1}H NMR spectrum of compound **8** due to the acetoxy methylene protons indicated that it was adjacent to a $-CH_2-$ group. Deacetoxylation of the keto-lupene **8** was effected with difficulty after refluxing with zinc and glacial acetic acid [8] for 12 hr to give lupene **9** (45%) and unchanged starting material (47%). We have previously reported [3] that the deacetoxylation of an axial acetoxy group situated α- to a keto group in a D:A-friedooleanane derivative was complete in less than one hour. Hence these observations regarding deacetoxy-



1 $R^1 = R^2 = \alpha$-OH, β-H; R^3 = H
2 $R^1 = R^2 = \alpha$-OH, β-H, R^3 = Me
3 $R^1 = R^2 = \alpha$-OAc, β-H; R^3 = H
7 $R^1 = \alpha$-OAc, β-H; $R^2 = \alpha$-OH, β-H; R^3 = Me
8 $R^1 = \alpha$-OAc, β-H; R^2 = O; R^3 = Me
9 R^1 = H; R^2 = O; R^3 = Me
10 $R^1 = R^3$ = H; $R^2 = \beta$-OH, α-H
15 R^1 = H, $R^2 = \beta$-OH, α-H; R^3 = Me



4 R = H
5 R = Ac



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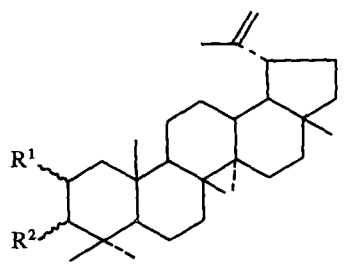

11 $R^1 = R^2 = \alpha$-OH, β-H
12 $R^1 = \alpha$-OH, β-H; $R^2 = \beta$-OH, α-H
13 $R^1 = R^2 = \alpha$-OAc, β-H
14 $R^1 = \alpha$-OAc, β-H; $R^2 = \beta$-OAc, α-H
16 $R^1 = R^2$ = H

lation of the keto-lupene **8** provided additional evidence for the equatorial orientation of the acetoxy group as equatorial acetoxy groups are known to undergo such deacetoxylation with difficulty [9]. The lupene **9** was found to be identical (co-TLC, mmp, IR and ^{1}H NMR) with methyl betulonate (**9**) prepared by oxidation followed by esterification of betulinic acid (**10**). Hence, this confirmed that the carbonyl group in compound **8** and therefore the axial hydroxy group in the lupene **1** was at C-3, that the equatorial hydroxy group must therefore be at C-2 and that the carboxylic acid group was at C-28. The chemical shifts of the carbinol methine protons reported in the literature [5] for the lup-20(29)-ene-2,3-diols **11** and **12**, and their diacetates **13** and **14** also supported the position and configuration assigned to these protons. The structure of lupene **1** was thus established to be 2α,3α-dihydroxylup-20(29)-en-28-oic acid.

The ^{13}C NMR chemical shifts of compound **2** were assigned by comparison with the reported shifts of methyl betulinate (**15**) and lup-20(29)-ene (**16**) [10], and are shown in Table 1. The doublet signals at δ66.6 and 78.9 confirm the presence of two secondary hydroxy groups. The ^{13}C NMR data also provide evidence for the axial nature of the hydroxy group at C-3 which gives rise to a γ-gauche interaction [11] with the C-5 carbon causing a shielding of 4 ppm compared to the corresponding carbon in compound **11** where the hydroxy group is equatorial and such an interaction is absent.

EXPERIMENTAL

Mps were determined on a Kofler hot stage apparatus and are uncorr. Identities of compounds were established by mmp, co-TLC, IR and ^{1}H NMR comparison. Petrol refers to the fraction having boiling range 60–80°, PLC was carried out on Merck-kieselgel 60 $PF_{254+365}$. Optical rotations were measured at 25° in $CHCl_3$. IR spectra were recorded for KBr discs. ^{1}H NMR spectra were recorded unless otherwise stated at 60 MHz in $CDCl_3$ with TMS as internal standard. 200 MHz ^{1}H spectra and 50.5 MHz ^{13}C spectra were recorded on a Varian XL-200 spectrometer in $CDCl_3$. Usual work up refers to dilution with ice cold water, acidification with dil. HCl, extraction with Et_2O, drying the Et_2O with dry $MgSO_4$ and evaporation to dryness. Mass spectra were recorded at the Research School of Chemistry, The Australian National University. Low resolution EI mass spectra were recorded at 70 eV on a VG 7070F instrument using the direct insertion probe. High resolution (~ 10000) accurate mass measurements were carried out on an MS-902 under the same operating conditions.

Extraction of E. revolutus. Dried and powdered stem bark (5 kg) was extracted with cold CH_2Cl_2. The extract was evaporated to dryness (111 g) and re-extracted with hot MeOH to remove gutta percha. The dried MeOH extract (70 g) was chromatographed on silica gel (C_6H_6–EtOAc–MeOH).

Isolation of 2α,3α-*dihydroxy-lup*-20(29)-*en*-28-*oic acid* (**1**). Elution of the column with EtOAc–5% MeOH gave a white solid which on preparative TLC followed by crystallization from

Table 1. ^{13}C NMR chemical shifts of some lupenes (ppm)

Carbon No.	(2)	(11)	(12)
1	42.1 *t*	38.7	40.3
2	66.6 *d*	27.3	18.7
3	78.9 *d*	78.6	42.1
4	38.3 *s*	38.7	33.2
5	51.2 *d*	55.2	56.3
6	17.9 *t*	18.2	18.7
7	34.0 *t*	34.2	34.3
8	40.8 *s*	40.5	41.0
9	49.4 *d*	50.4	50.5
10	38.5 *s*	37.0	37.5
11	20.8 *t*	20.8	20.8
12	25.8 *t*	25.4	25.2
13	38.1 *d*	38.1	38.0
14	42.4 *s*	42.2	42.8
15	29.6 *t*	29.6	27.4
16	32.1 *t*	32.0	35.6
17	56.6 *s*	56.4	43.0
18	48.1 *d*	49.3	48.3
19	46.9 *d*	46.8	47.9
20	150.5 *s*	150.1	150.6
21	30.5 *t*	30.5	29.9
22	36.9 *t*	36.8	40.0
23	28.4 *q*	27.9	33.4
24	21.6 *q*	15.3	21.6
25	17.1 *q*	16.0	16.1
26	15.9 *q*	15.9	16.1
27	14.7 *q*	14.6	14.6
28	176.6 *s*	176.2	18.0
29	109.6 *t*	109.3	109.2
30	19.3 *q*	19.3	19.3

$CHCl_3$–MeOH yielded 2α,3α-dihydroxy-lup-20(29)-en-28-oic acid (**1**; 120 mg), mp 298–300°; (Found: M^+ 472.3546; calc. for $C_{30}H_{48}O_4$, 472.3552); IR ν_{max} cm^{-1}: 3600–3100, 3400, 1690; ^1H NMR ($CDCl_3$–CD_3OD): δ4.72, 4.60 (each 1H, *m*, C=CH_2), 3.92 (1H, *m*, $W_{1/2}$ = 22 Hz, 2β-H), 3.40 (1H, *d*, *J* = 4 Hz, 3β-H), 1.69 (3H, *s*, Me–C), 1.00–0.83 (5 × t-Me). MS *m/z* (rel. int.): 472 (29) $[M]^+$, 454 (25), 439 (34), 426 (51), 408 (60), 393 (21), 355 (10), 302 (13), 248 (52), 235 (39), 223 (41), 205 (97), 189 (100), 175 (47).

Methylation of compound **1**. Compound **1** (50 mg) was suspended in MeOH (1 ml) and CH_2N_2–Et_2O added dropwise until a yellow colour persisted. Evaporation of the solvent followed by crystallization yielded methyl-2α,3α-dihydroxy-lup-20(29)-en-28-oate (**2**; 50 mg) mp 195–197°, $[\alpha]_D$ −6.0 (*c* 1.0) (Found M^+ 486.3703; calc. for $C_{31}H_{50}O_4$, 486.3709); IR ν_{max} cm^{-1}: 3400, 1730, 880; ^1H NMR: δ4.72, 4.60 (1H each, *m*, C=CH_2), 3.93 (1H, *m*, $W_{1/2}$ = 22 Hz, 2β-H), 3.62 (3H, *s*, MeO), 3.41 (1H, *d*, *J* = 3 Hz, 3β-H), 1.66 (3H, *s*, Me–C=), 0.95–0.83 (5 × t-Me); MS (rel. int.): 486 (27) $[M]^+$, 468 (8), 453 (10), 426 (19), 408 (13), 273 (6), 262 (53), 236 (20), 223 (38), 203 (54), 189 (100), 175 (38).

Acetylation of compound **1**. Compound **1** (20 mg) was treated with Ac_2O–pyridine (1:1) (5 ml) at 27° for 24 hr and usual work up followed by preparative TLC (5% MeOH and $CHCl_3$) afforded a white solid which on crystallization gave 2α,3α-bis(acetyloxy)-lup-20(29)-en-28-oic acid (**3**; 18 mg), mp 155–156°; $[\alpha]_D$ −3.6° (*c* 1.0). (Found M^+ 556.3739; calc. for $C_{34}H_{52}O_6$, 556.3764); IR ν_{max} cm^{-1}: 3500–3100, 1740, 1690, 1235, 880; ^1H NMR: δ5.21 (1H, *m*, $W_{1/2}$ = 23 Hz, 2β-H), 4.95 (1H, *d*, *J* = 3 Hz, 3β-H), 4.75, 4.63 (each 1H, *m*, C=CH_2), 2.12 (3H, *s*, Ac), 1.95 (3H, *s*, Ac), 1.69 (3H, *s*, Me–C=), 1.03–0.86 (5 × t-Me); MS *m/z* (rel. int.): 510 (9) $[M]^+$, 496 (7), 454 (11), 436 (34), 223 (19), 273 (3), 205 (32), 204 (18), 203 (50), 191 (19), 189 (93), 175 (38).

Preparation of 2,3-*seco diol* **4**. Excess HIO_4 (35 mg) was stirred with THF (2 ml) for 1 hr and the supernatant solution was added to a soln of **1** (20 mg) in THF (2 ml). The product obtained was reduced with $NaBH_4$ (100 mg). Usual work up followed by preparative TLC [(a) 50% EtOAc and toluene (b) 5% MeOH and $CHCl_3$] and crystallization from $CHCl_3$–MeOH yielded 2,3-dihydroxy-2,3-seco-lup-20(29)-en-28-oic acid (**4**; 10 mg), mp 290–291°; IR ν_{max} cm^{-1}: 3580–3100, 3340, 1685, 880; ^1H NMR: δ4.72, 4.63 (1H each, *m*, C=CH_2), 3.70 (1H, *m*), 3.50 (1H, *m*), 3.06 (2H, *br s*), 1.69 (3H, *s*, Me–C=C), 1.03–0.95 (5 × t-Me).

Acetylation of 2,3-*seco diol* **4**. Compound **4** (10 mg) was treated with Ac_2O–pyridine (1:1) (2 ml) for 24 hr at 27°. The usual work up followed by crystallization from $CHCl_3$–MeOH yielded 2,3-bis(acetyloxy)-2,3-seco-lup-20(29)-en-28-oic acid (**5**; 9 mg), mp 200–201°, $[\alpha]_D$ +10° (*c* 0.9) (Found M^+ 558.3925; calc. for $C_{34}H_{54}O_6$, 558.3920); IR ν_{max} cm^{-1}: 3450–3100, 1730, 1690, 1235; ^1H NMR (100 MHz): δ4.74, 4.62 (1H each, *m*, C=CH_2), 4.22 (1H, *m*), 4.10 (1H, *m*) [becomes two doublets (*J* = 11 Hz) when irradiated at 1.90], 4.04 (1H, *d*, *J* = 12 Hz), 3.76 (1H, *d*, *J* = 12 Hz), 2.10 (3H, *s*), 2.04 (3H, *s*), 1.68 (3H, *s*, Me–C=C), 1.13–0.94 (5 × t-Me); MS *m/z* (rel. int.): 558 (4) $[M]^+$, 512 (100), 498 (11), 452 (21), 443 (13), 397 (50), 383 (26), 357 (15), 337 (12), 328 (29), 297 (5), 287 (10), 259 (56), 233 (15), 189 (33), 175 (24).

Preparation of isopropylidene derivative of compound **1**. Compound **1** (30 mg) was dissolved in dry Me_2CO (1 ml) then dry $CuSO_4$ (1 mg) and conc. H_2SO_4 (0.5 ml) were added to the soln with stirring. The reaction mixture was stirred at 27° for 72 hr. Filtering followed by dilution and evaporation of the solvent yielded a dark brown substance which on preparative TLC (10% MeOH and $CHCl_3$) gave a white solid. Crystallization of this solid from $CHCl_3$–MeOH afforded 2α,3α-isopropylidene dioxy-lup-20(29)-en-28-oic acid (**6**; 20 mg) mp 230–231°, $[\alpha]_D$ +33.3° (*c* 1.0) (Found M^+ 512.3885; calc. for $C_{33}H_{52}O_4$, 512.3866); IR ν_{max} cm^{-1}: 3460–3100, 1690; ^1H NMR: δ4.73, 4.63 (1H each, *m*, C=CH_2), 4.15 (1H, *m*, 2-H), 3.69 (1H, *d*, *J* = 4 Hz, 3-H), 1.69 (3H, *s*, Me–C=C), 1.48 (3H, *s*), 1.29 (3H, *s*), 1.06–0.80 (5 × t-Me); MS *m/z* (rel. int.): 512 (4) $[M]^+$, 497 (27), 437 (12), 408 (12), 205 (32), 189 (41), 175 (25).

Partial acetylation of methyl ester **2**. Compound **2** (50 mg) was treated with Ac_2O (1 ml) and NaOAc (20 mg) at 27° for 2 hr. Usual work up followed by crystallisation from $CHCl_3$–MeOH yielded methyl 2α-acetoxy-3α-hydroxy-lup-20(29)-en-28-oate (**7**; 48 mg) mp 90°, $[\alpha]_D$ −6.3° (*c* 1.0) (Found M^+ 528.3828; calc. for $C_{33}H_{52}O_5$, 528.3815); IR ν_{max} cm^{-1}: 3480, 1735, 1720, 1240, 880; ^1H NMR: δ5.23 (1H, *m*, $W_{1/2}$ = 20 Hz, 2β-H), 4.71, 4.60 (each 1H, *m*, C=CH_2), 3.66 (3H, *s*, MeO), 3.49 (1H, *d*, *J* = 4 Hz, 3β-H), 2.04 (3H, *s*, MeO), 1.66 (3H, *s*, Me–C=), 1.00–0.90 (5 × t-Me); MS *m/z* (rel. int.): 528 (3) $[M]^+$, 468 (18), 453 (24), 408 (21), 273 (7), 262 (47), 203 (87), 189 (100), 175 (41).

Oxidation of compound **7**. Compound **7** (48 mg) was treated with CrO_3 (60 mg) in pyridine (2 ml) for 5 hr. Usual work up followed by preparative TLC (2% MeOH and $CHCl_3$) and crystallization from $CHCl_3$–MeOH yielded colourless needles of methyl 2α-acetoxy-3-oxolup-20(29)-en-28-oate (**8**; 40 mg) mp 209–210°, $[\alpha]_D$ +14.1° (*c* 1.0) (Found M^+ 526.3674; calc. for $C_{33}H_{50}O_5$, 526.3658); IR ν_{max} cm^{-1}: 1745, 1725, 880; ^1H NMR: δ5.58 (1H, *dd*, J_{a-a} = 14 Hz, J_{a-e} = 6 Hz), 4.72, 4.63 (1H each, *m*, C=CH_2), 3.69 (3H, *s*, MeO), 2.12 (3H, *s*, Ac), 1.69 (2H, *s*, Me–C=C), 1.20–0.92 (5 × t-Me); MS *m/z* (rel. int.): 526 (5) $[M]^+$, 511 (2), 466 (19), 263 (7), 219 (6), 203 (53), 189 (100), 175 (45).

Deacetoxylation of keto-lupene **8**. Compound **8** (40 mg) was

refluxed with Zn (100 mg) in HOAc (5 ml) for 12 hr. Filtration, followed by dilution and extraction with Et_2O yielded a white solid which on preparative TLC and crystallization from $CHCl_3$–MeOH gave methyl-3-oxolup-20(29)-en-28-oate (**9**; 18 mg), mp 258–259°, $[\alpha]_D$ +31°; IR ν_{max} cm^{-1}: 3440–3100, 1705, 1690, 880; ^1H NMR: δ 4.69, 4.55 (1H each, *m*, C=CH_2), 3.63 (3H, *s*, MeO), 1.66 (3H, *s*, Me–C=), 1.04–0.92 (5 × t-Me) and compound **8** (19 mg), mp 209–210°.

Preparation of methyl betulonate (**9**). An authentic sample of betulinic acid (**10**) (30 mg) in Et_2O was treated with CH_2N_2–Et_2O until a yellow colour persisted. The soln was allowed to stand for 1 hr and evaporated to dryness. The solid was then treated with CrO_3 (50 mg) in pyridine (1 ml) for 3 hr. The usual work up followed by preparative TLC (C_6H_6) and crystallization from $CHCl_3$–MeOH gave colourless crystals of methyl 3-oxolup-20(29)-en-28-oate (**9**; 19 mg), mp 258–259°, $[\alpha]_D$ +37° (*c* 1.0) (lit. [12] mp 252°, $[\alpha]_D$ +31°).

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