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## Three new phytoconstituents from *Lepidium sativum*

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Three new phytoconstituents, namely lepidiumsesterterpenol, lepidiumterpenoid and lepidiumterpenyl ester were isolated from *Lepidium sativum*. The structures have been established as 3,7,11,15,19-pentamethyleicos-5-en-8, 19-diol, 15-(17,21,21-trimethylcyclohex-16-ene-yl)-4,8,12-trimethylpentadec-14-ene-9-one-11-ol and 2,6,10-trimethyl tridecan-13-oic acid-1-hept-6'-ene-1'-oate, on the basis of spectral data analyses and chemical reactions.

### 1. Introduction

*Lepidium sativum* L. commonly called 'garden cress' is a polymorphic species. In spite of great medicinal values, *L. sativum* has not received the attention it deserves. So far there are few reports on systematic investigation on its biochemistry. Pharmacological studies reveal its cardiovascular and diuretic properties. The plant is used as anti-fertility and antiovarulatory drug.

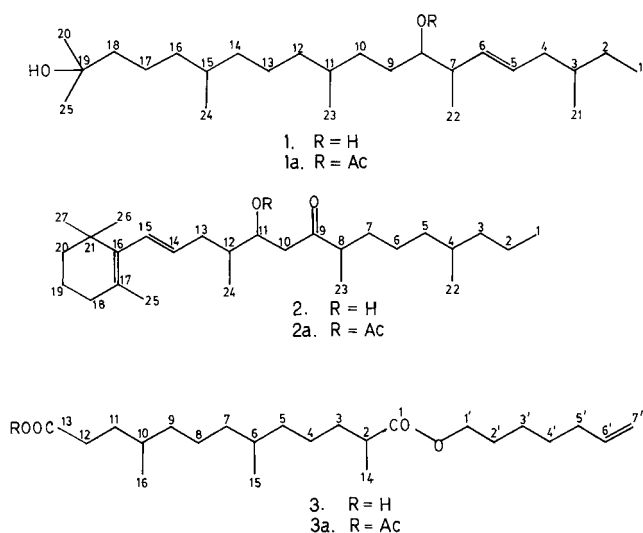
Variable proportions of benzyl cyanide and benzyl isocyanate are reported from this plant which contribute towards its activity against *Bacillus subtilis* and *Micrococcus pyogenes* [1, 2]. Despite its potential therapeutic values no serious attempt has been made to work out its phytochemistry. This paper describes the isolation and characterization of three terpenic constituents from the aerial parts of the plant.

### 2. Investigations, results and discussion

Compound **1** named lepidiumsesterterpenol, was obtained as colorless flakes from the chloroform eluents. Its IR spectrum demonstrated the presence of a characteristic absorption band for hydroxyl groups ( $3414\text{ cm}^{-1}$ ) and the MS exhibited a molecular ion peak at  $m/z$  382 corresponding to a sesterterpenoid molecule  $\text{C}_{25}\text{H}_{50}\text{O}_2$ . It indicated one double bond equivalent which was adjusted in an olefinic linkage. The presence of one hydroxyl group at C-19 was established by appearing important peaks at  $m/z$  59 [C<sub>18</sub>-C<sub>19</sub> fission, ion a]<sup>+</sup>, 101 [C<sub>15</sub>-C<sub>16</sub> fission, ion b]<sup>+</sup>, 281 [M-ion b]<sup>+</sup>, 129 [C<sub>14</sub>-C<sub>15</sub> fission, ion d]<sup>+</sup>, 41 [ion a-H<sub>2</sub>O]<sup>+</sup>, 55 [ion b-2-CH<sub>2</sub>-H<sub>2</sub>O]<sup>+</sup>, 69 [ion b-CH<sub>2</sub>-H<sub>2</sub>O]<sup>+</sup> and 111 [ion d-H<sub>2</sub>O]<sup>+</sup>. The other ion peaks generating at  $m/z$  171 [C<sub>11</sub>-C<sub>12</sub> fission, ion 3]<sup>+</sup>, 211 [M-ion e, ion f]<sup>+</sup>, 199 [C<sub>10</sub>-C<sub>11</sub> fission, ion g]<sup>+</sup>, 183 [M-ion g, ion h]<sup>+</sup>, 257 [C<sub>7</sub>-C<sub>8</sub> fission, ion i]<sup>+</sup>, 125 [M-ion i, ion j]<sup>+</sup>, 227 [ion i-CHOH]<sup>+</sup>, 139 [ion e-CH<sub>2</sub>-H<sub>2</sub>O]<sup>+</sup>, 153 [ion e-H<sub>2</sub>O]<sup>+</sup>, 181 [ion g-H<sub>2</sub>O]<sup>+</sup>, 195 [213-H<sub>2</sub>O]<sup>+</sup> support the presence of a secondary hydroxyl group at C-8. The location of an olefinic linkage at C-5 (6) was established from the ion fragments arising at  $m/z$  285 [C<sub>6</sub>-C<sub>7</sub> fission, ion k]<sup>+</sup>, 97 [M-ion k, ion l]<sup>+</sup>, 311 [C<sub>4</sub>-C<sub>5</sub> fission, ion m]<sup>+</sup>, 71 [M-ion m, ion n]<sup>+</sup> 57 [C<sub>3</sub>-C<sub>4</sub> fission, ion p]<sup>+</sup> and 325 [M-ion] [3,4]. The <sup>1</sup>H NMR spectrum (Table) of **1** displayed a multiplet at  $\delta$  5.29, integrated for two protons, assignable to H-5 and H-6 and a one proton broad singlet at  $\delta$  3.66 ascribed to C-8 carbinol protons. Another broad singlet at  $\delta$  1.20, integrated for six protons, was accounted to C-20 and C-25 methyls attached to C-19 carbinol carbon. A three proton doublet at  $\delta$  1.04 with coupling interaction of six Hz was associated with C-22 secondary methyl function. A nine proton broad singlet at

$\delta$  0.87 was due to C-21, C-23 and C-24 methyl protons. The C-1 primary methyl function resonated as a three proton triplet  $\delta$  0.81 ( $J = 6.5\text{ Hz}$ ). The remaining methylene and methine protons appeared in the range of  $\delta$  2.80–1.25. The <sup>13</sup>C NMR spectrum (Table) of **1** disclosed the presence of two carbinol carbons ( $\delta$  66.20, C-8; 68.18, C-19) and two unsaturated carbons ( $\delta$  130.91, C-5; 128.80, C-6) [5, 6]. Multiplicities of the <sup>13</sup>C NMR signals were determined by DEPT experiments. The assignments of the carbons bearing hydrogens were established by the <sup>1</sup>H-<sup>13</sup>C COSY spectrum. The analysis of the <sup>1</sup>H-<sup>1</sup>H COSY spectrum showed the proton connectivities of H-2/H-6/H-10, H-6/H-5a and H-1a/H-2a. Compound **1** yielded a monoacetyl product **1a** on treatment with acetic anhydride and pyridine, supporting the presence of one tertiary and one acetylatable hydroxyl group in the molecule. Based on these spectral data analyses and chemical reactions, the structure of the new natural lepidiumsesterterpenol (**1**) was identified as 3,7,11,15,19 pentamethyleicos-5-en-8,19-diol.

This unknown and rare compound is being reported for the first time from any natural or synthetic source. Compound **2**, named lepidiumterpenoid, was obtained as colorless flakes from chloroform eluents. Its IR spectrum demonstrated the presence of hydroxyl ( $3415\text{ cm}^{-1}$ ) and carbonyl ( $1716\text{ cm}^{-1}$ ) groups. Compound **2** had a molecular ion peak at  $m/z$  404 in its MS consistent with the molecular formula of a monocyclic homosesterterpenoid  $\text{C}_{27}\text{H}_{48}\text{O}_2$ . It indicated ion peaks at  $m/z$  123 [C<sub>15</sub>-C<sub>16</sub> fission, ion a]<sup>+</sup>, 281 [M-ion a, ion b]<sup>+</sup>, 149 [C<sub>13</sub>-C<sub>14</sub> fission, ion c]<sup>+</sup>, 225 [M-ion c, ion d]<sup>+</sup>, 164 [C<sub>12</sub>-C<sub>13</sub> fission, ion e]<sup>+</sup>, 108 [ion a-Me]<sup>+</sup>, 263 [ion b-H<sub>2</sub>O]<sup>+</sup> and 134 [ion c-Me]<sup>+</sup> suggesting the presence of a trimethyl



substituted cyclohexane moiety at one of the terminal carbons of the molecule and an olefinic linkage at  $\Delta^{14}$  position. The existence of a hydroxyl group at C-11 and a carbonyl group at C-9 was inferred from the ion peaks generated at  $m/z$  191 [ $C_{11}$ - $C_{12}$  fission, ion f] $^+$ , 213 [M-ion f, ion g] $^+$ , 221 [ $C_{10}$ - $C_{11}$  fission, ion h] $^+$ , 183 [M-ion h, ion i] $^+$ , 263 [ $C_8$ - $C_9$  fission, ion j] $^+$ , 141 [M-ion j, ion k] $^+$ , 291 [ $C_7$ - $C_8$  fission, ion l] $^+$  and 223 [M-ion e- $H_2O$ ] $^+$ . The other important peaks arose at  $m/z$  389 [M-Me] $^+$ , 113 [M-ion l, ion m] $^+$ , 99 [113- $CH_2$ ] $^+$ , 301 [319- $H_2O$ ] $^+$ , 85 [99- $CH_2$ ] $^+$ , 71 [85- $CH_2$ ] $^+$  and 43 [ $C_3H_7$ ] $^+$ . These data suggested the monocyclic nature of the homosesterterpene possessing one hydroxyl and one carbonyl group. The  $^1H$  NMR spectrum (Table) of **2** displayed a downfield two proton multiplet at  $\delta$  5.34 assignable to vinylic H-14 and H-15. A one proton multiplet at  $\delta$  3.66 was attributed to the C-11 carbinol proton. A three proton broad singlet at  $\delta$  1.71 was ascribed to the C-25 methyl group attached at C-17 unsaturated carbon. Another three upfield broad singlets at  $\delta$  1.20 (3H) 1.16 (3H) and 0.88 (6H) were ascribed correspondingly to C-26, C-27 and C-1, C-23 methyl protons. The C-24 and C-22 methyl functionalities resonated as three protons, each doublets, at  $\delta$  0.96 ( $J = 6.0$  Hz), 0.80 ( $J = 6.0$  Hz), respectively. The remaining methine and methylene protons appeared between  $\delta$  2.00–1.25. The  $^{13}C$  NMR spectrum (Table) of **2** showed [7] the presence of 27 carbon atoms along with a carbinol carbon ( $\delta$  70.08). Except for the quaternary one, each carbon signal was assigned based on the DEPT spectral data. The correlations between  $^1H$  and  $^{13}C$  were established by the  $^1H$ - $^{13}C$  COSY spectrum. The analysis of the  $^1H$ - $^1H$  COSY spectrum of **2** showed connectivities of H-4/H-8/H-12 and H-11/H-10<sub>a</sub>. Treatment of **2** with acetic anhydride and pyridine yielded a monoacetyl product **2a**. On the basis of these studies the structure of lepidiumterpe-

niod (**2**) has been elucidated as 15-(17,21,21-trimethylcyclohex-16-ene-yl)-4,8,12-trimethylpentadec-14-ene-9-one-11-ol. This is a new member of the sesterterpenoid class and is being reported for the first time from a species of Brassicaceae.

Compound **3**, designated as lepidiumterpenyl ester, was obtained as granular crystallized mass from chloroform eluents. The compound gave effervescence with sodium bicarbonate solution suggesting a carboxylic nature of the molecule. Its IR spectrum showed characteristic absorption bands for carboxylic groups (3413, 1700  $cm^{-1}$ ) and ester linkage (1739  $cm^{-1}$ ). The MS of **3** exhibited a molecular ion peak at  $m/z$  382, corresponding to the homosequiterpene ester formula  $C_{23}H_{42}O_3$ . Lack of the  $C_nH_{2n}$  and  $C_nH_{2n-1}$  ion peaks ruled out an aliphatic nature of the molecule [3, 4]. The appearance of important ion peaks at  $m/z$  73 [ $C_{10}$ - $C_{11}$  fission, ion a] $^+$ , 309 [M-ion a, ion b] $^+$ , 101 [ $C_9$ - $C_{10}$  fission, ion c] $^+$ , 281 [M-ion c, ion d] $^+$ , 143 [ $C_5$ - $C_6$  fission, ion e] $^+$ , 171 [ $C_5$ - $C_6$  fission, ion g] $^+$  213 [ $C_2$ - $C_3$  fission, ion i] $^+$  and 241 [ $C_1$ - $C_2$  fission, ion k] $^+$  supported the presence of a carboxylic group at one of the terminal carbons and the saturated nature of the sesquiterpene moiety [8]. The ester linkage of an unsaturated heptyl alcohol at another terminal carbon was deduced from the ion fragments generated at  $m/z$  141 [M-ion K, ion l] $^+$ , 269 [CO-C fission, ion m] $^+$ , 113 [ $C_2$ - $C_3$  fission, ion q] $^+$ , 69 [M-ion q, ion r] $^+$ , 55 [ion r- $CH_2$ ] $^+$  and 41 [ion r-2  $\times$   $CH_2$ ] $^+$ . The  $^1H$  NMR spectrum of **3** (Table) displayed three one-proton each down field signals at  $\delta$  5.36 (m), 5.26 (brs) and 5.20 (brs) assigned to olefinic H-6' and the terminal H-7'a and H-7'b methylene protons. A two-proton broad signal at  $\delta$  3.66 was associated with an oxygen substituted methylene group. A three proton doublet at  $\delta$  0.98 ( $J = 6.0$  Hz) was ascribed to a C-14 secondary methyl group. The C-15 and C-16 methyl functionalities

**Table:**  $^1H$  and  $^{13}C$  NMR spectral data of compounds **1**, **2** and **3** ( $CDCl_3$ )

| Position | $^1H$ NMR              |                      |                      | $^{13}C$ NMR |        |        |
|----------|------------------------|----------------------|----------------------|--------------|--------|--------|
|          | 1                      | 2                    | 3                    | 1            | 2      | 3      |
| 1        | 0.81 t ( $J = 6.5$ Hz) | 0.88 brs             | –                    | 14.04        | 14.01  | 179.31 |
| 2        | 1.25 brs               | 1.25 brs             | –                    | 22.54        | 25.60  | 52.26  |
| 3        | 2.80 m                 | 1.25 brs             | 2.78 m               | 35.77        | 26.43  | 26.71  |
| 4        | 2.22 m                 | 1.55 m               | 1.36 brs             | 38.78        | 44.30  | 29.34  |
| 5        | 5.29 m                 | 1.25 brs             | 1.36 brs             | 130.91       | 37.30  | 29.34  |
| 6        | 5.29 m                 | 1.25 brs             | 1.36 brs             | 128.80       | 37.36  | 31.92  |
| 7        | 2.80 m                 | 1.25 brs             | 2.30 brs             | 46.63        | 40.18  | 29.34  |
| 8        | 3.66 brs               | 2.00 m               | 1.36 brs             | 66.20        | 46.31  | 29.34  |
| 9        | 1.25 brs               | –                    | 1.36 brs             | 31.93        | 201.01 | 29.34  |
| 10       | 1.25 brs               | 2.00                 | 2.00 brs             | 28.95        | 29.61  | 31.41  |
| 11       | 2.36 m                 | 3.66                 | 1.36                 | 37.01        | 70.08  | 26.25  |
| 12       | 1.25 brs               | 1.55                 | 1.60                 | 29.71        | 52.62  | 29.75  |
| 13       | 1.25 brs               | 1.68 m               | –                    | 26.71        | 31.44  | 163.57 |
| 14       | 1.25 brs               | 5.36 m               | 0.98 d ( $J = 6$ Hz) | 26.55        | 129.70 | 15.33  |
| 15       | 2.33 m                 | 5.36 m               | 0.97 brs             | 36.33        | 129.93 | 14.40  |
| 16       | 1.76 m                 | –                    | 0.94 brs             | 23.79        | 144.90 | 14.13  |
| 17       | 1.76 m                 | –                    | –                    | 22.99        | 138.91 | –      |
| 18       | 2.03 m                 | 1.68 m               | –                    | 30.41        | 31.92  | –      |
| 19       | –                      | 1.25 brs             | –                    | 68.18        | 29.45  | –      |
| 20       | 1.20 brs               | 1.25 brs             | –                    | 18.17        | 29.32  | –      |
| 21       | 0.87 brs               | –                    | 3.66 brs             | 11.79        | 55.49  | –      |
| 22       | 1.04 d ( $J = 6$ Hz)   | 0.80 d ( $J = 6$ Hz) | 1.54 brs             | 22.30        | 15.31  | 27.23  |
| 23       | 0.87 brs               | 0.88 brs             | 1.36 brs             | 15.31        | 21.08  | 29.28  |
| 24       | 0.87 brs               | 0.96 d ( $J = 6$ Hz) | 1.36 brs             | 13.10        | 22.63  | 22.75  |
| 25       | 1.20 brs               | 1.71 brs             | 2.00 m               | 16.28        | 24.95  | 30.23  |
| 26       | –                      | 1.20 brs             | 5.36 m               | –            | 20.02  | 128.68 |
| 27       | –                      | 1.16 brs             | 5.26 brs<br>5.20 brs | –            | 20.11  | 109.35 |

appeared as a six proton broad singlet at  $\delta$  0.94. The remaining methylene and methine protons resonated in between  $\delta$  2.78–1.36. The existence of all the methyls at  $\delta$  0.98 and 0.94 suggested their attachment to the saturated carbons. The carbon skeleton of **3** (acyclic homosesquiterpenoid esterified with heptenyl alcohol) was supported by analysis of the  $^{13}\text{C}$  NMR spectrum [9–11]. The presence of the ester and carboxylic groups was indicated by signals at  $\delta$  179.31 and 163.57, respectively (Table). The olefinic carbons appeared at  $\delta$  128.68 (C-6') and 10.35 (C-7'). DEPT experiments were carried out to assign the nature of  $-\text{CH}$ ,  $-\text{CH}_2$ ,  $-\text{CH}_3$  and olefinic carbon resonances. The connectivities of 2-H, 6-H and 10-H were established by  $^1\text{H}-^1\text{H}$  and  $^1\text{H}-^{13}\text{C}$  experiments which coupled with the methyl signals attached to these positions. Methylation of **3** with diazomethane yielded a monomethyl ester **3a**. Based on these evidences the structure of lepidiumterpenyl ester **3b** was formulated as 2,6,10-trimethyl tridecan 13-oic acid-1-hept-6'-ene-1'-oate. This is a new sesquiterpenic ester of natural origin and is being reported for the first time from *L. sativum*.

### 3. Experimental

Melting points were determined on a perfit melting point apparatus and are uncorrected. UV spectra were recorded on Beckmann DU-64. IR spectra on a Shimadzu 8201 PC FTIR spectrophotometer,  $^1\text{H}$  NMR on a Bruker WM-400 (400 MHz FT) instrument,  $^{13}\text{C}$  NMR spectra at 100 MHz and MS on a Jeol D-30 spectrometer.

#### 3.1. Plant material

The aerial parts of *L. sativum* were collected from the Herbal Garden at Jamia Hamdard and identified by Dr. M. P. Sharma (Taxonomist), Department of Botany. A voucher specimen is preserved in the Herbarium of the Department of Botany, Faculty of Science, Jamia Hamdard.

#### 3.2. Extraction

The dried and coarsely-powdered plant material (1 kg) was defatted with petroleum ether and Soxhlet-extracted with methanol. The concentrated brown colored mass (60 g) was fractionated into 0.3 M HCl-soluble and insoluble portions. The HCl soluble portion was extracted with  $\text{CHCl}_3$  ( $3 \times 100$  ml), basified with  $\text{NH}_3$  solution (pH 9) and re-extracted with  $\text{CHCl}_3$  ( $5 \times 100$  ml). The later combined extract was dried ( $\text{Na}_2\text{SO}_4$ ), concentrated under reduced pressure to get a dark brown viscous semi-solid mass. It was then transferred to china dish and adsorbed in basic alumina (60–120 mesh) with constant stirring until dried. This was left for 2–3 days for drying before packing in the column.

#### 3.3 Isolation of chemical constituents

The HCl-insoluble portion was adsorbed on alumina (60–120 mesh), the slurry dried and subjected to alumina CC. The column was eluted successively with petroleum ether,  $\text{CHCl}_3$  and MeOH in the order of increasing polarity to isolate the following compounds. Elution with each solvent was combined until a small volume of eluent gave no residue on evaporation. Each fraction was collected separately and subjected to TLC to check homogeneity of various fractions.

##### 3.3.1. Lepidiumsesterterpenol (1)

Elution of the column with  $\text{CHCl}_3$  (fraction 4–16) gave colorless flakes of **1** recrystallized from  $\text{CHCl}_3$ –MeOH (1:1), 130 mg (0.004% yield),  $R_f$  0.2 m.p. 200–201 °C. UV  $\lambda_{\text{max}}$  (MeOH): 205 nm (log  $\epsilon$  1.5) IR  $\nu_{\text{max}}$  (KBr) 3414, 2926, 2852, 1630, 1564, 1450, 1369, 976, 926, 700  $\text{cm}^{-1}$ .  $^1\text{H}$  NMR Table 1. EIMS  $m/z$  (ret int): 382  $[\text{M}]^+$  (1.7), 325 (1.2), 311 (1.3), 297 (2.5), 295 (1.7), 293 (2.8), 285 (1.2), 283 (1.8), 281 (5.8), 297 (3.7), 269 (10.3), 265 (14.8), 257 (6.8), 241 (2.3), 239 (4.0), 227 (5.5), 223 (5.8), 213 (3.7), 211 (2.5), 209 (4.1), 19.9 (6.9), 195 (3.0), 183 (3.5), 181 (5.2), 171 (6.4), 169 (3.9), 157 (3.8), 155 (6.3), 153 (7.2), 149 (22.2), 143 (13.7), 141 (7.3), 139 (9.7), 129 (9.3), 125 (21.6), 113 (9.6), 111 (20.0), 101 (10.1), 99 (24.1), 97 (33.6), 69 (21.2), 59 (24.2), 57 (56.6), 55 (100), 43 (83.6), 41 (95.6).  $^{13}\text{C}$  NMR: Table.

**Acetylation of 1:** Compound **1** (10 mg) was allowed to react with acetic anhydride (3 ml) and pyridine (1 ml) for 24 h.  $\text{H}_2\text{O}$  (10 ml) was added and the reaction mixture was extracted with  $\text{CHCl}_3$  ( $2 \times 10$  ml). The organic phase was washed with  $\text{H}_2\text{O}$  ( $2 \times 5$  ml), dried over  $\text{Na}_2\text{SO}_4$  and then evaporated to obtain the monoacetyl product **1a** m.p. 171–172 °C.  $\nu_{\text{max}}$  3400, 1730  $\text{cm}^{-1}$ .

##### 3.3.2. Lepidiumterpenoid (2)

Further elution of the column with  $\text{CHCl}_3$  (fraction 17–20) afforded colorless flakes of **2**, 100 mg recrystallized from  $\text{CHCl}_3$ –MeOH (1:1) (0.003% yield),  $R_f$  0.1, m.p. 120–121 °C UV  $\lambda_{\text{max}}$ : 204, 263 nm (log 2.8, 0.9), IR  $\nu_{\text{max}}$  (KBr): 3415, 2926, 2852, 1716, 1564, 1452, 1430, 1371, 1277, 1076, 974, 923, 700  $\text{cm}^{-1}$ .  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  5.36 (2H, m, H-14, H-15), 3.66 (1H, m, H-11), 2.00 (3H, m, H-8, H<sub>2</sub>-10), 1.71 (3H, brs, Me-25), 1.68 (4H, m, H<sub>2</sub>-13, H<sub>2</sub>-18), 1.55 (H, m, H-4, H-12), 1.25 (14H, brs,  $7 \times \text{CH}_2$ , 1.20 (3H, brs, Me-26), 1.16 (3H, brs, Me-27), 0.96 (3H, d,  $\tau = 6.0$  Hz, Me-24), 0.88 (6H, brs, Me-1, Me-23), 0.08 (3H, d,  $J = 6.0$  Hz, Me-022). EIMS  $m/z$  (ret. int.): 404  $[\text{M}]^+$  ( $\text{C}_{27}\text{H}_{48}\text{O}_2$ ) (2.4), 389 (3.0), 319 (2.3), 301 (2.4), 291 (3.8), 281 (3.3), 263 (3.4), 255 (3.3), 237 (2.5), 235 (2.5), 223 (13.5), 221 (3.5), 213 (3.4), 191 (3.5), 183 (3.2), 164 (6.4), 149 (17.2), 141 (5.9), 134 (12.8), 123 (15.8), 121 (12.5), 113 (12.4), 108 (31.8), 99 (15.3), 95 (45.3), 85 (32.1), 81 (55.3), 71 (34.3), 67 (83.4), 57 (73.1), 55 (100), 43 (95.0).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  14.01 (C-1), 25.60 (C-2), 26.43 (C-3), 44.3 (C-4), 37.30 (C-5), 37.36 (C-6), 40.18 (C-7), 46.31 (C-8), 201.01 (C-9), 29.61 (C-10), 70.08 (C-11), 52.62 (C-12), 31.44 (C-13), 129.70 (C-14), 129.93 (C-15), 144.90 (C-16), 138.91 (C-17), 31.92 (C-18), 29.45 (C-19), 29.32 (C-20), 55.49 (C-21), 15.31 (C-22), 21.08 (C.23), 22.63 (C-24), 24.95 (C-25), 20.02 (C-26), 20.11 (C-27).

**Acetylation of 2:** Compound **2** (10 mg) was treated with a mixture of acetic anhydride (3 ml) and pyridine (1 ml) for 12 h.  $\text{H}_2\text{O}$  (10 ml) was added and the reaction mixture was extracted with  $\text{CHCl}_3$  ( $3 \times 10$  ml). The organic phase was washed with  $\text{H}_2\text{O}$  ( $2 \times 10$  ml), dried over  $\text{Na}_2\text{SO}_4$  and evaporated to obtain the monoacetyl product **2a** m.p. 98–99 °C.

##### 3.3.3. Lepidium terpenylester (3)

Elution of the column with  $\text{CHCl}_3$  (fraction 21–23) furnished colorless granular crystals of **3**, recrystallized from  $\text{CHCl}_3$ –MeOH (1:1), 135 mg (0.002% yield),  $R_f$  0.25, m.p. 100–101 °C  $[\alpha]_{\text{D}}^{25}$  0°, UV  $\lambda_{\text{max}}$  (MeOH), 205 nm (log  $\epsilon$  1.6), IR  $\nu_{\text{max}}$  (KBr): 3413, 2927, 2856, 1739, 1700, 1564, 1446, 1411, 1255, 1172, 1089, 977  $\text{cm}^{-1}$ .  $^1\text{H}$  NMR: Table. EIMS  $m/z$  (ret. int.): 382  $[\text{M}]^+$  ( $\text{C}_{23}\text{H}_{42}\text{O}_4$ ) (3.2), 355 (2.1), 327 (2.1), 323 (1.1), 313 (2.3), 309 (1.7), 299 (4.0), 291 (8.9), 285 (2.8), 281 (4.8), 269 (12.3), 267 (5.6), 264 (17.9), 241 (2.1), 239 (3.6), 213 (3.8), 211 (1.9), 199 (5.2), 197 (2.1), 185 (5.2), 183 (2.2), 171 (5.4), 169 (3.2), 143 (12.7), 141 (6.8), 129 (9.7), 115 (8.3), 113 (4.3), 101 (12.0), 97 (23.6), 95 (32.4), 87 (52.1), 83 (46.2), 73 (77.1), 69 (68.3), 59 (13.6), 55 (100), 41 (80.1).  $^{13}\text{C}$  NMR: Table.

**Methylation of 3:** Compound **3** (5 mg) was dissolved in solvent ether (5 ml), ethereal solution of diazomethane was added drop-wise and the reaction mixture was left overnight. The solvent was then evaporated to get methyl ester **3a**, m.p. 92–93 °C.

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