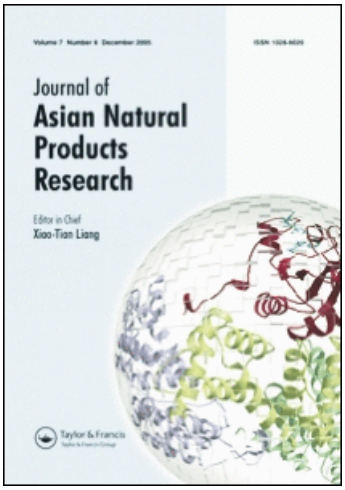

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Ethylenediamine: an effective reagent for deacetylation of natural products

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ORIGINAL ARTICLE

Ethylenediamine: an effective reagent for deacetylation of natural products

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The use of ethylenediamine in methanol is described for the selective cleavage of the acetate group in nimbin (**1**) to 6-deacetyl nimbin (**1a**) under microwave irradiation. This method enables to deacetylate without affecting other functional groups such as α,β-unsaturated ketone, ester, ether, etc. in certain tetranortriterpenoids and other acetate-containing natural compounds.

Keywords: forskolin; ethylenediamine; nimbin; microwave

1. Introduction

Semi-synthetic modification of natural products has gathered momentum in recent times owing to its potential in generating molecules with promising biological activity. Modification of a given structural entity is best achieved by functional group transformation or by introduction of moieties, which can enhance or alter the activity.

2. Results and discussion

During our ongoing investigations to prepare nitrogen analogs of some selected diterpenes and tetranortriterpenoids, we employed ethylenediamine in methanol to produce an imino product with forskolin [1], a compound known to exhibit excellent vasodilating and cardio-stimulating properties.

The NMR spectrum of the product revealed the disappearance of the C═O resonance at 170 ppm (acetate carbonyl), indicating hydrolysis of a 7β-acetoxy moiety. The carbonyl resonance at 203 ppm confirmed the presence of C-11 carbonyl in concurrence with the above-mentioned observation. This prompted us to evaluate the use of ethylenediamine to produce deacetyl products from polyfunctional natural products. The available agents such as NaOH and Na_2CO_3 in alcohol resulted in the formation of de-esterified products along with the deacetylated product in nimbin. The Lewis acids would cleave the epoxide moiety [2] and the use of the other reagents like hydrazine hydrate may result in the cleavage of α,β-epoxy ketones [3]. Ethylenediamine seems to be a superior reagent for deacetylation in comparison with NH_3 in MeOH [4], exclusively yielding one product irrespective of the quantity of amine used. Hence, an attempt has been made to deacetylate

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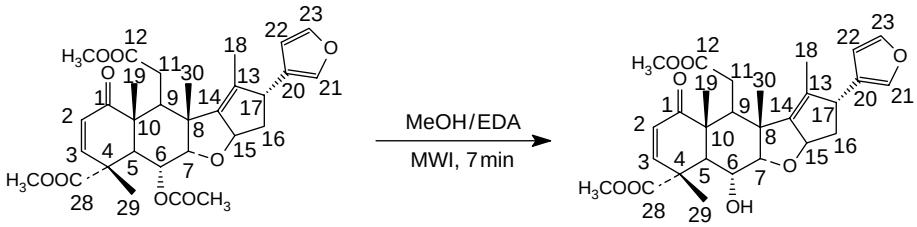


1 1a

Scheme 1. Conversion of nimbin (**1**) to deacetyl nimbin (**1a**).

nimbin using ethylenediamine resulting in deacetyl nimbin (Scheme 1) in 75% yield within 7 min under microwave [5,6] irradiation condition without any side products. The compound was then purified by column chromatography.

It has been observed that this reagent is mild and selectively cleaves the acetate group amid other functional groups. We have demonstrated (Table 1) the selective cleavage of acetate in certain tetranortriterpenoids such as nimbin [7,8], nimonol, salannin, and azadiradione [9–11] with multiple functional groups, and therefore this method has advantages over other methods.

The basicity of ethylenediamine is exploited in deacetylation without affecting other functional groups. The ^{1}H and ^{13}C NMR spectra of this compound showed a similar characteristic to that of nimbin, except for the acetate group at C-7. It was clearly evident from the disappearance of the carbonyl group of acetate at 170.3 ppm and the methyl group signal at 20.8 ppm. The formation of the hydroxyl group at C-7 was confirmed by the appearance of the signal at 86.9 ppm, which in turn showed the connectivity to the proton at 3.76 ppm. The assigned structure for the product obtained is 6-deacetyl nimbin, which is in agreement with the mass 498 $[M]^{+}$.

In summary, we have developed a simple, selective, and mild procedure for the cleavage of the acetate group in natural products, which will have a wide scope in view of the usefulness of ethylenediamine [12,13] in organic synthesis.

3. Experimental

3.1 Procedure

Various tetranortriterpenoids such as nimbin, salannin, azadiradione, nimonol, etc. were obtained in the pure form after being subjected to repeated purification by column chromatography from the oil, fruit coat, and leaves of *Azadirachta indica*. Similarly, forskolin was obtained from *Coleus forskohlii* and cedrelone from the barks of *Toona ciliata*. The β-amyrin and pregnenolone were obtained from SD Fine Chemicals, Worli, Mumbai, India.

NMR spectra were recorded on a Bruker 200 MHz instrument using TMS as the internal reference for both ^{1}H and ^{13}C NMR experiments. $CDCl_3$ was used as the solvent. Chemical shifts are given in terms of parts per million (δ scale). MS analyses were recorded on a Shimadzu QP 5000 instrument. IR spectra (cm^{-1}) were recorded on a Perkin-Elmer RX1 FT-IR spectrophotometer. Precoated thin layer chromatography plates (E-Merck, Darmstadt, Germany; Kieselgel 60 F254, 0.2 mm thickness, coated on aluminum sheets) were used. Column chromatography was performed using silica gel (60–120 mesh and 230–400 mesh).

3.2 Deacetylation of nimbin

Nimbin (108 mg, 5 mmol) was added to a solution of ethylenediamine (2 ml in 2 ml

Table 1. Deacetylation of natural products with ethylenediamine under modified microwave irradiation.

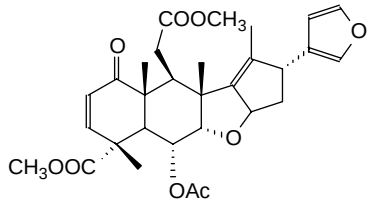
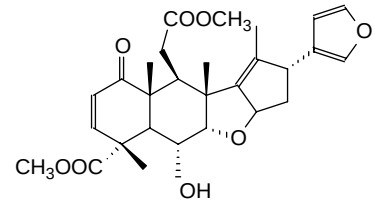
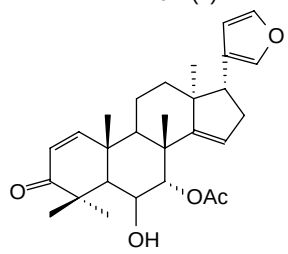
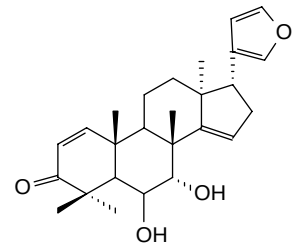
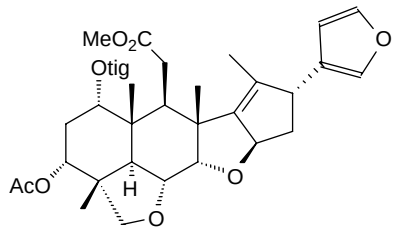
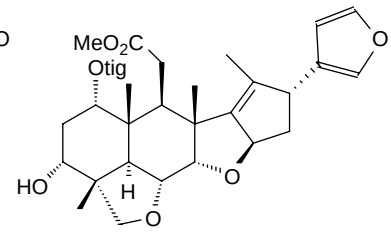
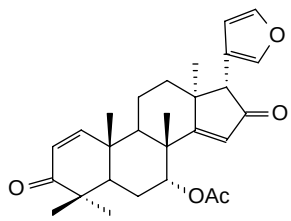
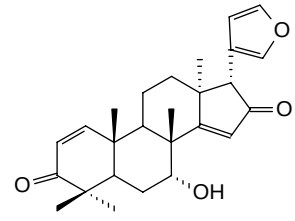
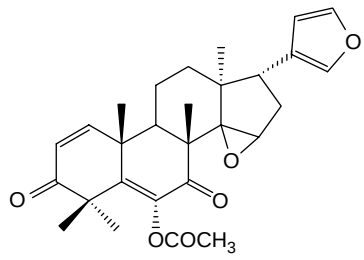
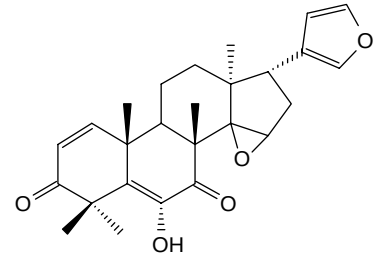
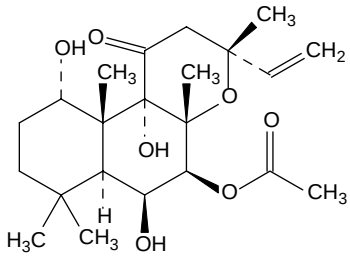
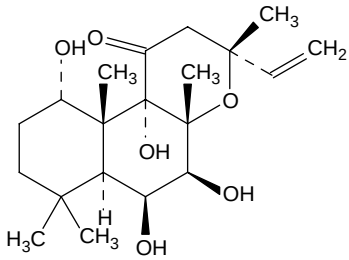
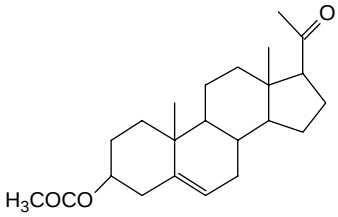
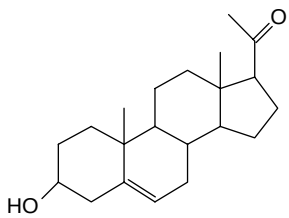
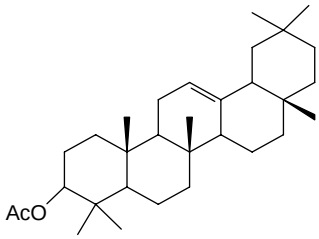
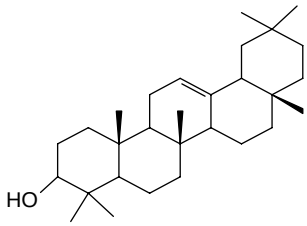
Entry	Substrate	Product	Time (min)	Isolated yield (%)
1	 Nimbin (**1**)	 Deacetyl nimbin (**1a**)	7	75
2	 Nimonol	 Deacetyl nimonol	6	88
3	 Salannin	 Deacetyl salannin	12	76
4	 Azadiradione	 Deacetyl azadiradione	10	78
5	 Cedrelone acetate	 Cedrelone	5	82

Table 1 – *continued*

Entry	Substrate	Product	Time (min)	Isolated yield (%)
6	 Forskolin	 Deacetyl forskolin	5	80
7	 Pregnenolone acetate	 Pregnenolone	4	90
8	 Amyrin acetate	 Amyrin	4	94

Note: Yields were calculated after purification by column chromatography.

of methanol) and irradiated under microwave with stirring for 7 min. The reaction was monitored by TLC (70:30; ethyl acetate–hexane). The solvent was removed under reduced pressure. The reaction mixture was poured into ice-cold water and extracted with ethyl acetate to afford a white powder. The yield obtained after purification by column chromatography was 81 mg (75%). A series of deacetyl products are given in Table 1. The selected ^{1}H and ^{13}C NMR spectral data of both the starting material and the product are shown in Tables 2–6.

3.2.1 *Deacetyl nimbin*

$C_{28}H_{34}O_8$; mp 208°C; UV λ_{max} (MeOH): 209 nm; IR (KBr) ν_{max}: 3574, 3640, 2940, 2855, 1455, 1390, 1370, 1355, 1275, 1250, 1140, 1112, 1090, 1060, 1028, 878, 783, 744 cm^{-1}; ^{1}H NMR (δ, ppm): 7.32 (m, 1H), 7.24 (m, 1H), 6.41 (d, 1H, $J = 10.1$ Hz), 6.33 (m, 1H), 5.84 (d, 1H, $J = 10.1$ Hz), 5.55 (m, 1H), 4.01 (d, 1H, $J = 3.3$ Hz), 3.91 (dd, 1H, $J = 11.4$, 3.3 Hz), 3.70 (s, 3H), 3.65 (s, 3H), 3.60 (m, 1H), 3.40 (d, 1H, $J = 11.7$ Hz), 2.92 (m, 1H), 2.77 (m, 1H), 2.25 (m, 1H), 2.20 (m, 1H), 2.06 (m, 1H), 1.21–1.69

Table 2. The selected ^{1}H and ^{13}C NMR spectral data of nimbin (**1**) and 6-deacetyl nimbin (**1a**).

	^{1}H NMR (δ ppm)		^{13}C NMR (δ ppm)	
Carbon no.	**1**	**1a**	**1**	**1a**
5	3.70 (m, 1H)	3.40 (d, 1H, $J = 11.7$ Hz)	41.5	43.6
6	5.25 (dd, 1H, $J = 12.3$, 3.0 Hz)	3.91 (dd, 1H, $J = 11.7$, 3.3 Hz)	68.6	66.1
7	4.05 (d, 1H, $J = 3.0$ Hz)	4.01 (d, 1H, $J = 3.3$ Hz)	84.5	86.9
8	–	–	47.1	47.3
9	2.85 (dd, 1H, $J = 5.5$, 3.0 Hz)	2.77 (m, 1H)	38.5	39.0
10	–	–	47.9	47.4

Table 3. The selected ^{1}H and ^{13}C NMR spectral data of nimonol (**2**) and 7-deacetyl nimonol (**2a**).

	^{1}H NMR (δ ppm)		^{13}C NMR (δ ppm)	
Carbon no.	**2**	**2a**	**2**	**2a**
5	2.23 (m, 1H)	2.45 (m, 1H)	49.9	47.2
6	4.37 (m, 1H)	4.22 (m, 1H)	68.1	68.4
7	5.37 (m, 1H)	3.49 (m, 1H)	79.1	75.9
8	–	–	45.5	44.4
9	2.44 (m, 1H)	2.87 (m, 1H)	36.9	35.6
10	–	–	43.3	45.6

Table 4. The selected ^{1}H and ^{13}C NMR spectral data of salannin (**3**) and 3-deacetyl salannin (**3a**).

	^{1}H NMR (δ ppm)		^{13}C NMR (δ ppm)	
Carbon no.	**3**	**3a**	**3**	**3a**
1	4.78 (t, 1H, $J = 2.7$ Hz)	5.30 (m, 1H)	71.3	72.8
2	2.04–2.37 (m, 2H)	2.05–2.35 (m, 2H)	27.5	30.5
3	4.96 (t, 1H, $J = 2.8$ Hz)	3.87 (m, 1H)	71.3	70.8
4	–	–	42.6	44.2
5	2.81 (d, 1H, $J = 12.5$ Hz)	2.63 (d, 1H, $J = 12.3$ Hz)	40.5	38.8
6	4.01 (dd, 1H, $J = 12.5$, 3.3 Hz)	4.08 (dd, 1H, $J = 12.3$, 3.3 Hz)	72.5	72.4

Table 5. The selected ^{1}H and ^{13}C NMR spectral data of azadiradione (**4**) and 7-deacetyl azadiradione (**4a**).

	^{1}H NMR (δ ppm)		^{13}C NMR (δ ppm)	
Carbon no.	**4**	**4a**	**4**	**4a**
5	2.20 (dd, 1H, $J = 3.7$, 11.6 Hz)	2.50 (dd, 1H, $J = 12.8$ Hz)	46.1	36.5
6	1.88 (m, 2H)	1.88 (m, 2H)	23.3	27.1
7	5.32 (t, 1H, $J = 2.7$ Hz)	3.99 (m, 1H)	73.8	71.7
8	–	–	44.4	44.2
9	2.50 (m, 1H)	2.55 (m, 1H)	38.1	44.5
10	–	–	39.9	40.1

Table 6. The selected ^{1}H and ^{13}C NMR spectral data of forskolin (**6**) and 7-deacetyl forskolin (**6a**).

	^{1}H NMR (δ ppm)		^{13}C NMR (δ ppm)	
Carbon no.	**6**	**6a**	**6**	**6a**
5	1.05 (m, 1H)	1.07 (m, 1H)	41.8	42.9
6	4.57 (m, 1H)	4.48 (m, 1H)	74.0	70.5
7	5.48 (d, 1H, $J = 4.3$ Hz)	4.62 (m, 1H)	73.2	74.7
8	–	–	68.8	82.2
9	–	–	80.3	82.2
10	–	–	41.7	42.7

(br s, 4 × CH_3); ^{13}C NMR (δ, ppm): 202.9, 175.5, 173.7, 148.1, 146.9, 143.0, 139.0, 134.9, 126.8, 126.4, 110.4, 87.4, 86.9, 66.2, 53.0, 51.6, 49.6, 47.8, 47.5, 43.6, 47.3, 41.4, 39.0, 34.4. MS *m/z*: 498 $[M]^{+}$.

3.2.2 *Deacetyl salannin*

$C_{32}H_{42}O_8$; mp 213–216°C; UV λ_{max} (MeOH): 210 nm; IR (KBr) cm^{-1}: 3430, 2993, 1698, 1662, 1593, 1460, 1387, 1151, 1030, 752, 602; ^{1}H NMR (δ, ppm): 7.31 (m, 1H), 7.25 (m, 1H), 6.92 (m, 1H), 6.27 (m, 1H), 5.38 (m, 1H), 5.30 (m, 1H), 4.18 (d, 1H, $J = 3.3$ Hz), 4.08 (dd, 1H, $J = 12.3$, 3.3 Hz), 3.87 (m, 1H), 3.87 (m, 1H), 3.62 (m, 1H), 3.62 (m, 1H), 3.19 (s, 3H), 2.67 (m, 1H), 2.50–2.75 (d, 1H, $J = 4.5$ Hz), 2.05–2.35 (m, 2H), 2.05–2.35 (m, 1H), 2.05–2.35 (m, 1H), 2.05–2.35 (m, 2H), 1.85 (s, 3H), 0.96–1.69 (br s, 4 × CH_3); ^{13}C NMR (δ, ppm): 172.6, 166.3, 146.4, 142.8, 138.7, 138.1, 134.9, 128.6, 127.1, 110.6, 87.8, 85.8, 77.8, 72.8, 72.4, 70.8, 51.4, 49.3, 48.9, 44.2, 41.2, 40.9, 39.4, 38.8, 30.5, 30.5, 19.8, 16.8, 15.1, 14.5, 13.1, 12.1. MS *m/z*: 554 $[M]^{+}$.

3.2.3 *Deacetyl azadiradione*

$C_{26}H_{32}O_4$; mp 208°C; UV λ_{max} (MeOH) 237 nm; IR (KBr) cm^{-1}: 3409, 2923, 2826, 1728, 1649, 1435, 1387, 1259, 1155, 1037, 983, 786, 732, 599; ^{1}H NMR (δ, ppm): 7.43 (m, 1H), 7.34 (m, 1H), 7.10 (d, 1H, $J = 10.2$ Hz), 6.10 (s, 1H), 6.06 (m, 1H), 5.89 (d, 1H, $J = 10.2$ Hz), 3.99 (m, 1H), 3.50 (s, 1H), 2.55 (m, 1H), 2.50 (dd, 1H, $J = 12.8$ Hz), 2.08 (m, 1H), 2.04 (m, 1H), 1.88 (m, 2H), 1.86 (m, 1H), 1.84 (m, 1H), 1.42 (s, 3H), 1.27 (s, 3H), 1.12 (s, 3H), 1.12 (s, 3H), 0.96 (s, 3H); ^{13}C NMR (δ, ppm): 204.6, 201.8, 193.7, 157.1, 142.8, 141.7, 125.9, 123.4, 118.4, 111.1, 71.7, 60.9, 48.3, 44.6, 44.2, 40.2, 36.6, 30.5, 27.1, 26.4, 26.0, 25.4, 21.44, 19.1, 15.7. MS *m/z*: 408 $[M]^{+}$.

3.2.4 *Deacetyl nimonol*

$C_{26}H_{34}O_4$; mp 194–198°C; UV λ_{max} (MeOH): 223 nm; IR ν_{max} (MeOH) cm^{-1}: 3500, 2900, 2875, 1658, 1460, 1382, 1224, 1157, 1103, 1028, 931, 794, 597, 466; ^{1}H NMR (δ, ppm): 7.38 (m, 1H), 7.25 (m, 1H), 7.07 (d, 1H, $J = 10.2$ Hz), 6.29 (m, 1H), 5.86 (d, 1H, $J = 10.2$ Hz), 5.58 (d, 1H, $J = 10.2$ Hz), 4.22 (m, 1H), 3.49 (m, 1H), 2.87 (m, 1H), 2.45 (m, 1H), 2.45 (m, 1H), 2.16–2.37 (m, 2H), 2.16–2.37 (m, 2H), 1.95 (m, 2H), 1.11–1.42 (s, 3H), 1.11–1.42 (s, 3H), 1.11–1.42 (s, 3H), 1.11–1.42 (s, 3H); ^{13}C NMR (δ, ppm): 206.1, 161.0, 157.3, 142.7, 139.8, 126.2, 124.2, 120.0, 111.0, 76.0, 68.4, 51.6, 49.5, 47.2, 45.6, 44.4, 40.8, 35.6, 34.4, 32.3, 29.7, 26.9, 20.6, 20.3, 16.2. MS *m/z*: 410 $[M]^{+}$.

3.2.5 *Cedrelone*

$C_{26}H_{32}O_4$; mp 208°C; UV λ_{max} (MeOH): 237 nm; IR (KBr) cm^{-1}: 3392, 3118, 3033, 2933, 2866, 1676, 1618, 1448, 1384, 1340, 1251, 1153, 1060, 1031, 981, 947,

918, 877, 813, 779, 752, 715, 669, 634, 596; ^{1}H NMR (δ, ppm): 7.37 (m, 1H), 7.15 (m, 1H), 6.90 (d, 1H, $J = 9.7$ Hz), 6.44 (bs, D_2O exchangeable), 6.18 (m, 1H), 5.89 (d, 1H, $J = 9.7$ Hz), 3.80 (s, 1H), 2.30 (m, 1H), 2.75 (m, 1H), 2.67 (m, 1H), 2.07 (m, 1H), 1.94 (m, 1H), 1.85 (m, 1H), 1.69 (m, 1H), 0.75–1.57 (br s, × CH_3); ^{13}C NMR (δ, ppm): 203.7, 197.9, 152.4, 143.0, 141.1, 139.3, 134.8, 127.2, 123.1, 110.6, 69.8, 55.0, 46.8, 48.5, 43.1, 42.1, 42.0, 41.7, 40.2, 35.1, 26.7, 23.9, 23.0, 21.1, 20.2, 19.4. MS *m/z*: 422 $[M]^+$.

3.2.6 *Deacetyl forskolin*

$C_{20}H_{32}O_6$; mp: 163–166°C; UV λ_{max} (MeOH): 210 nm; IR (KBr) cm^{-1}: 3419, 2939, 1716, 1562, 1402, 1247, 1103, 1060; ^{1}H NMR (δ, ppm): 6.12 (dd, 1H, $J = 17.4$, 10.7 Hz), 5.19 (d, 1H, $J = 17.3$ Hz), 4.98 (d, 1H, $J = 10.8$ Hz), 4.63 (m, 1H), 4.48 (m, 1H), 4.13 (m, 1H), 3.18 (d, 1H, $J = 17.2$ Hz), 2.50 (d, 1H, $J = 17.2$ Hz), 2.35 (m, 1H), 2.20 (m, 1H), 2.09 (m, 1H), 1.75 (m, 1H), 1.65 (s, 3H), 1.43 (s, 3H), 1.41 (s, 3H), 1.27 (s, 3H), 1.06 (s, 3H); ^{13}C NMR (δ, ppm): 205.8, 146.5, 110.5, 82.3, 82.2, 75.2, 74.8, 74.7, 70.5, 48.8, 48.8, 43.0, 42.8, 36.1, 34.3, 33.0, 30.8, 24.2, 23.4, 20.0. MS *m/z*: 368 $[M]^+$.

3.2.7 *Pregnenolone*

$C_{21}H_{32}O_2$; mp 171–173°C; UV λ_{max} (MeOH): 206 nm; IR (KBr) cm^{-1}: 3506, 3352, 3028, 2933, 2835, 1683, 1461, 1433, 1357, 1292, 1224, 1191, 1168, 1134, 1097, 1053, 985, 950, 914, 840, 800, 596, 468; ^{1}H NMR (δ, ppm): 5.36 (m, 1H), 3.53 (m, 1H), 2.54 (t, 1H, $J = 9.0$ Hz), 2.20–2.35 (m, 2H), 2.20–2.35 (m, 2H), 2.13 (s, 3H), 2.06 (m, 1H), 2.04 (m, 2H), 1.40–1.75 (m, 2H), 1.40–1.75 (m, 2H), 1.40–1.75 (m, 1H), 1.40–1.75 (m, 1H), 1.40–1.75 (m, 2H), 1.40–1.75 (m, 1H), 1.00–1.30 (m, 2H), 1.0–1.30 (m, 2H), 1.01 (s, 3H), 0.63 (s, 3H); ^{13}C NMR (δ, ppm): 209.6, 140.8, 121.3, 71.6, 63.7, 56.9, 50.0, 44.0, 42.2, 38.8, 36.4, 36.5, 31.8, 31.7, 31.6, 31.5, 24.5, 22.8, 21.1, 19.4, 13.2. MS *m/z*: 316 $[M]^+$.

3.2.8 *β-Amyrin*

$C_{30}H_{50}O$; mp 196–198°C; UV λ_{max} (MeOH): 206 nm; IR (KBr) cm^{-1}: 3316, 2854, 1643, 1456, 1360, 1195, 1036, 988, 881, 824, 660; ^{1}H NMR (δ, ppm): 4.62 (m, 1H), 3.21 (m, 1H), 1.92–2.10 (m, 2H), 1.65–1.85 (4H, m), 1.40–1.55 (m, 12H), 0.90–1.10 (m, 4H), 1.03 (s, 3H), 0.99 (s, 3H), 0.96 (s, 2H), 0.94 (s, 3H), 0.91 (s, 3H), 0.82 (s, 3H), 0.79 (s, 3H), 0.76 (s, 3H); ^{13}C NMR (δ, ppm): 150.1, 124.4, 79.0, 55.2, 48.0, 48.0, 46.9, 41.5, 40.0, 38.7, 38.7, 37.1, 34.2, 33.3, 32.9, 32.6, 31.3, 28.1, 28.4, 28.0, 27.4, 26.1, 25.1, 23.5, 23.5, 18.9, 16.1, 15.7, 15.4. MS *m/z*: 426 $[M]^+$.

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Supporting Information

Supporting information is available online.

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