# SUPPLEMENTARY DATA

## A new flavonoid glycoside from the aerial parts of *Diplotaxis erucoides* (L.) DC. growing in Algeria

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**Abstract**

The phytochemical study of the 70% ethanol extract of the aerial parts of *Diplotaxis erucoides* afforded one new flavonoid glycoside, namely kaempferol-3-*O*-[*α*-L-rhamnopyranosyl-(1→2)-*β*-D-xylopyranoside]-7-*O*-*α*-L-rhamnopyranoside (**1**), named diploerucoside A and seven known compounds including one flavonoid (**2**), one phenolic glycoside (**3**), one monoterpene (**4**), one triterpene (**5**), one sitosterol (**6**) and two monoglycerolipids (**7**, **8**). Their structures were established by extensive spectroscopic analysis including 1D- and 2D-NMR ($^{1}$H, $^{13}$C, $^{1}$H-$^{1}$H COSY, HSQC and HMBC), mass spectrometry (HR-ESI-MS) and by comparison with the data reported in the literature.

**Keywords:** Brassicaceae, *Diplotaxis erucoides*, Kaempferol-3-*O*-[*α*-L-rhamnopyranosyl-(1→2)-*β*-D-xylopyranoside]-7-*O*-*α*-L-rhamnopyranoside, flavonoids, NMR

## Appendix A. Supplementary data

## 1. MS, 1D and 2D NMR spectra of compound 1

**Kaempferol 3-*O*-[*α*-L-rhamnopyranosyl-(1→2)-*β*-D-xylopyranoside]-7-*O*-*α*-L-rhamnopyranoside (1):** Yellow crystals; mp 252-253 °C; $[\alpha]_D^{20}$ – 21.6 (*c* 0.15, MeOH); $^1$H and $^{13}$C see **Table S1**.**HR-ESI-MS *m/z***: 709.1985 $[M – H]^-$ (calcd $[C_{32}H_{37}O_{18}]^-$, 709.1985) with molecular formula $C_{32}H_{38}O_{18}$

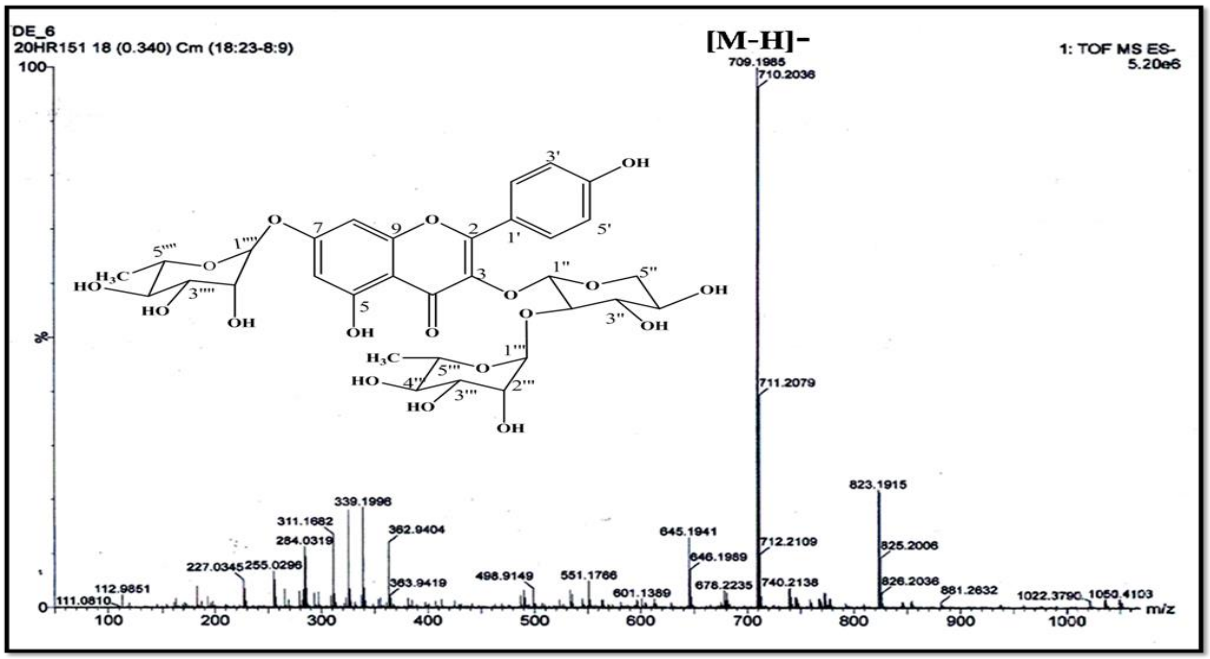


**Figure S1. HR-ESI-MS of compound 1**

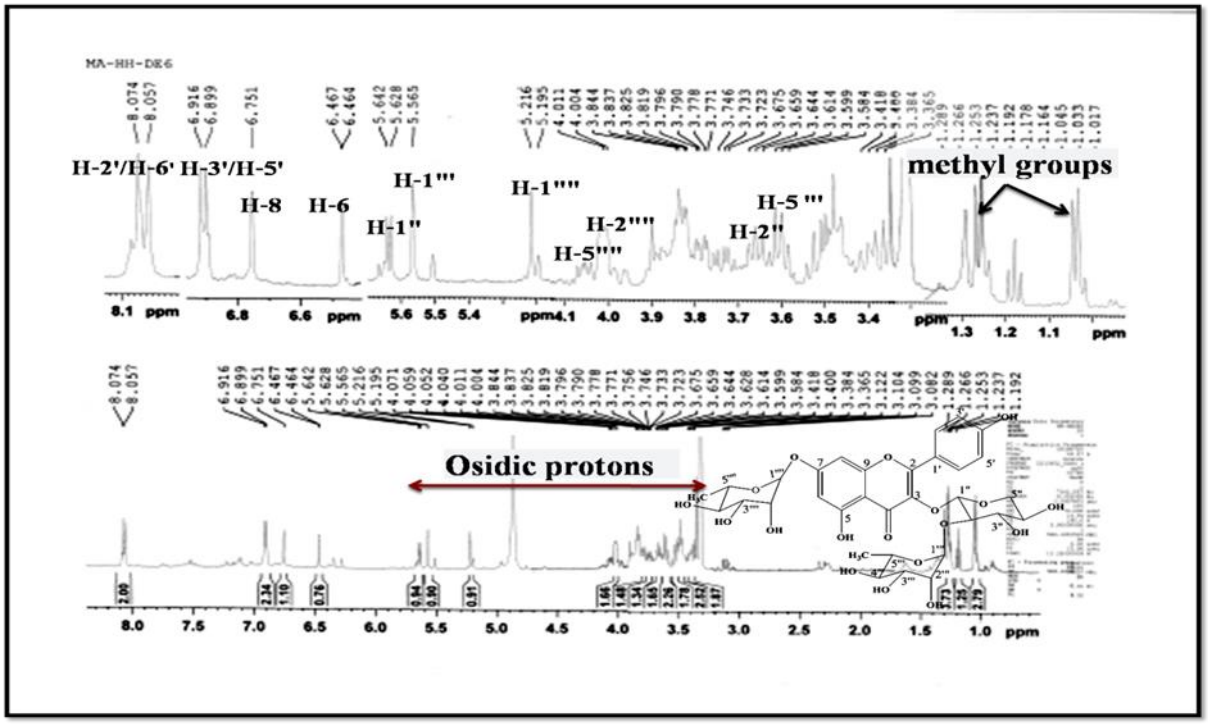


**Figure S2. $^1$H NMR (500 MHz, $CD_3OD$) spectrum of compound 1**

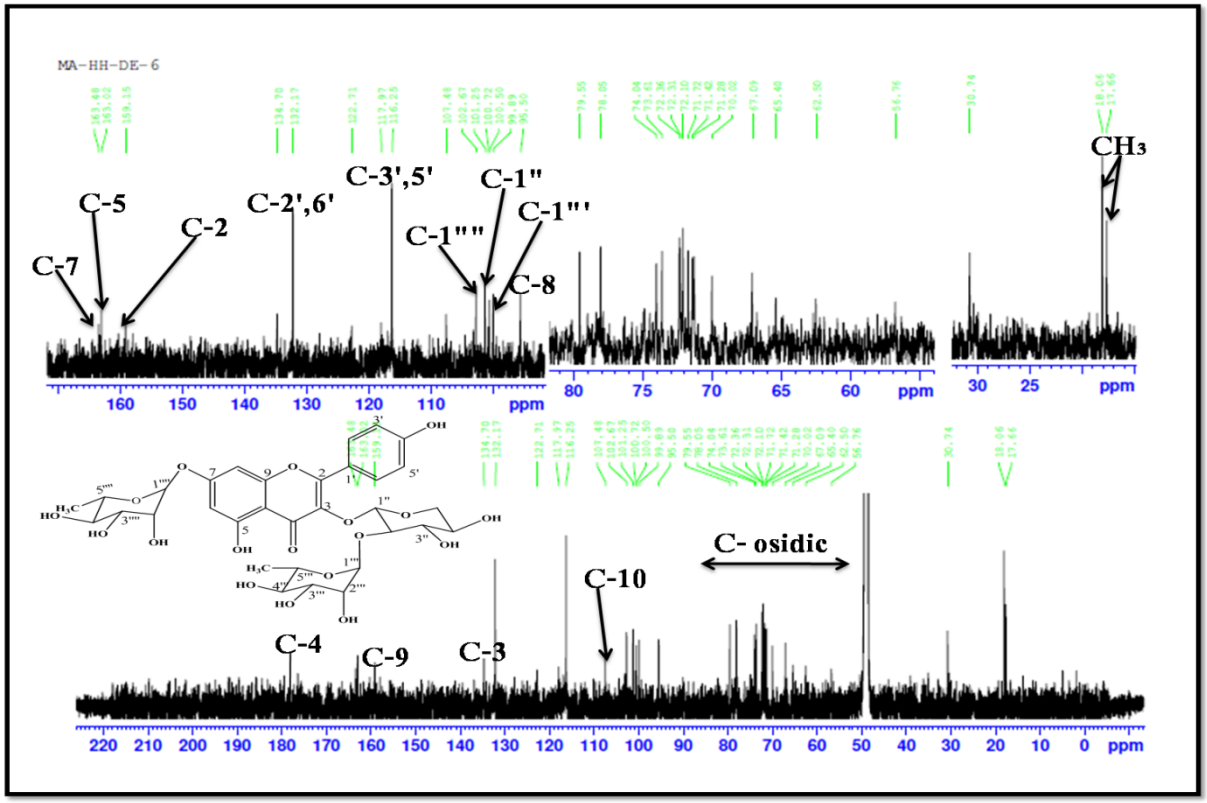


**Figure S3. $^{13}$C-NMR (125 MHz, $CD_3OD$) spectrum of compound 1**

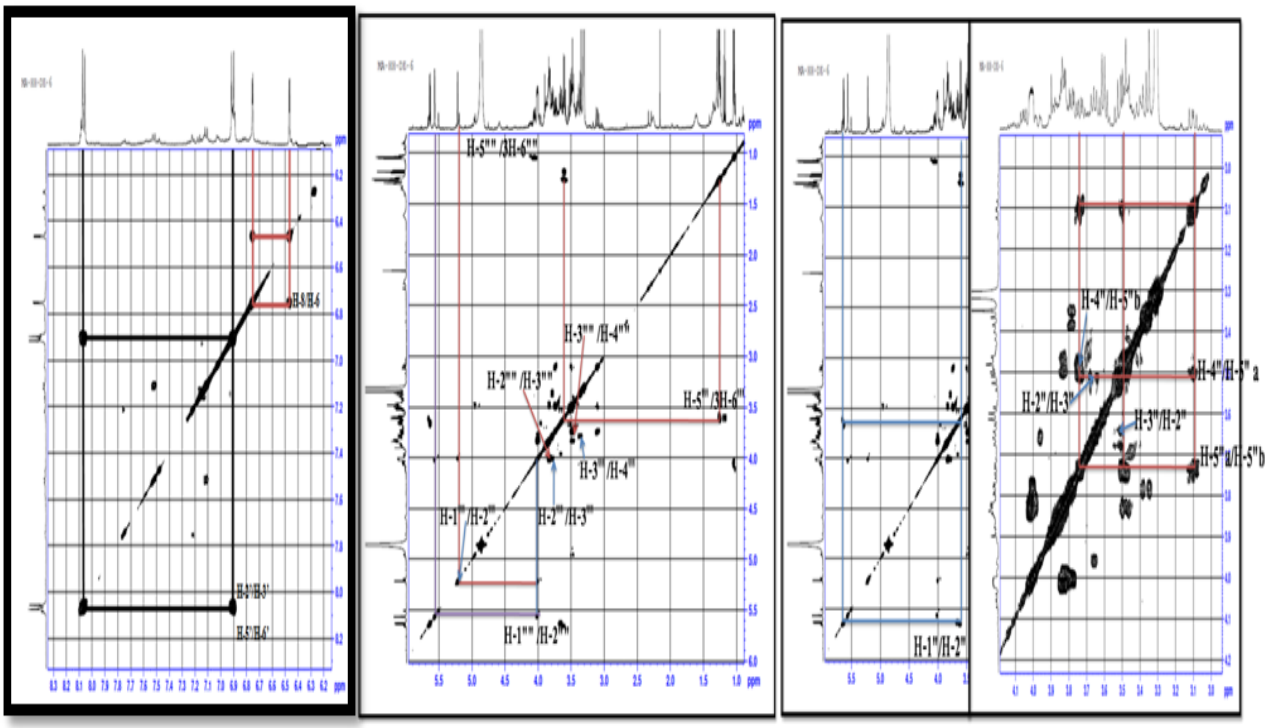


**Figure S4. COSY spectrum of compound 1**

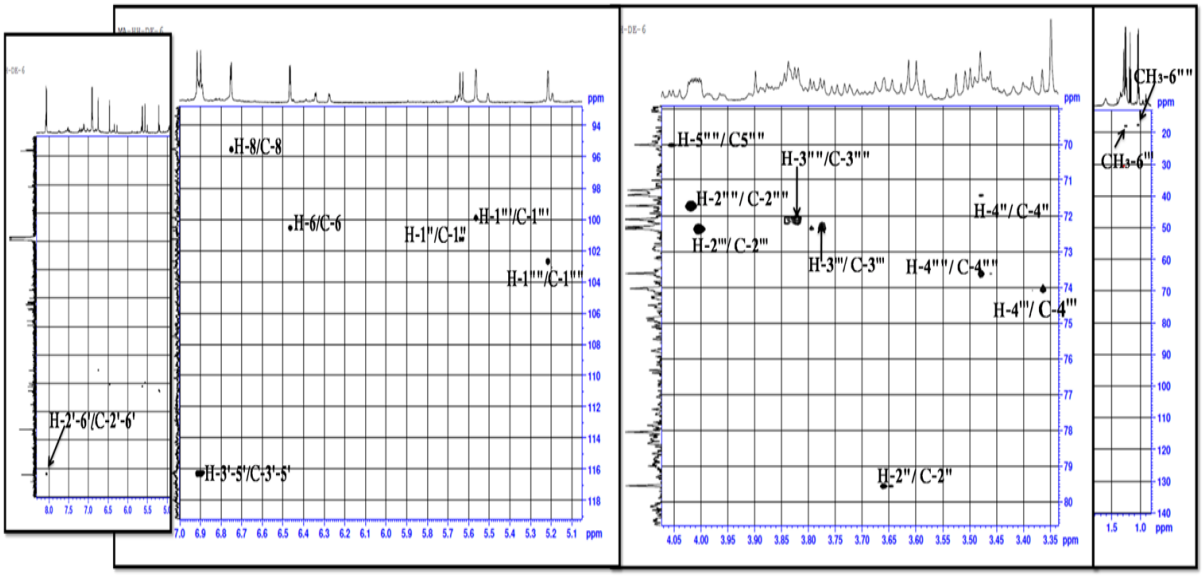


**Figure S5. HSQC spectrum of compound 1**

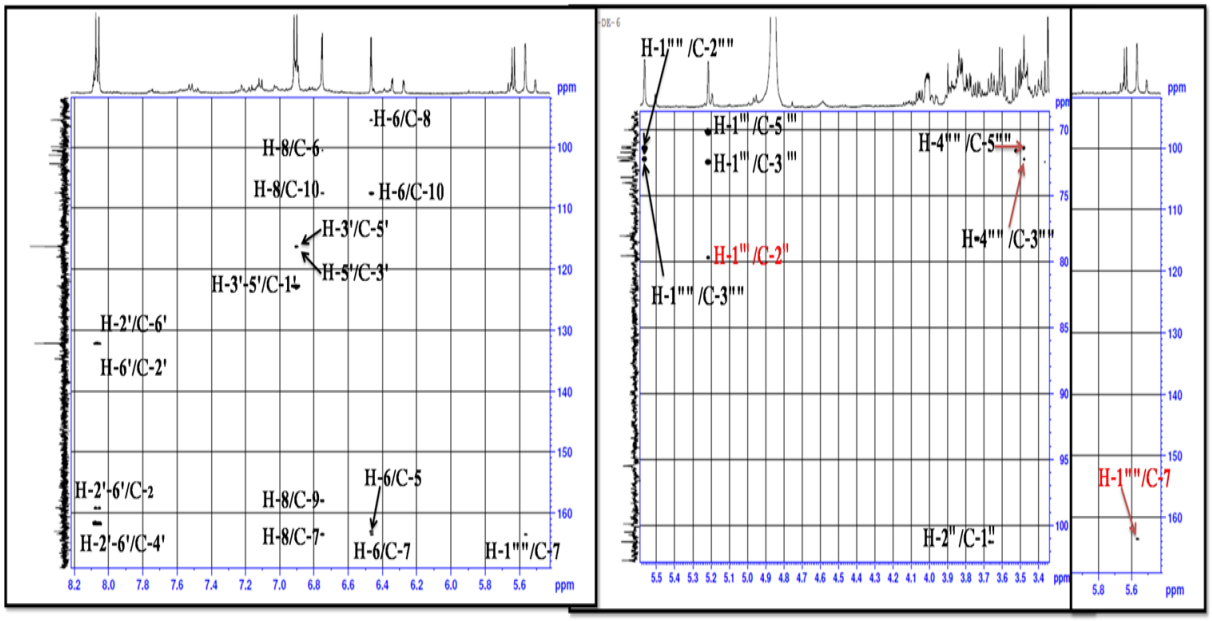


**Figure S6. HMBC spectrum of compound 1**

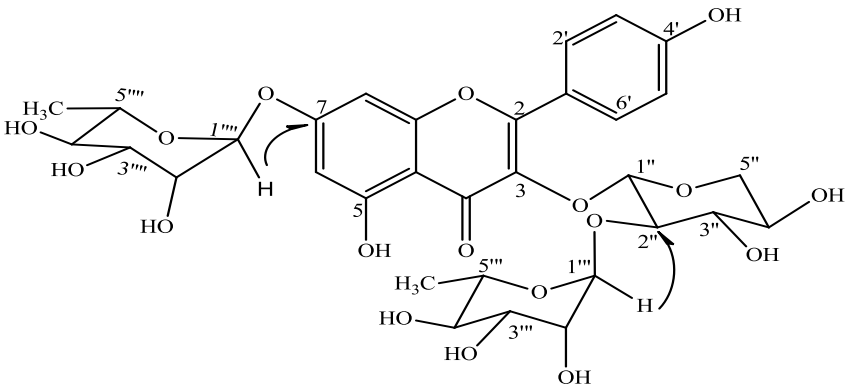


**Figure S7. HMBC (H-C) correlations for compound 1**

## 2. MS, 1D NMR spectra of compound 2

**Astragalin (2):** Yellow powder; $^{1}H$ and $^{13}C$ see **Table S1. ESI-MS *m/z*** : 447.0926 $[M-H]^-$ with molecular formula $C_{21}H_{20}O_{11}$

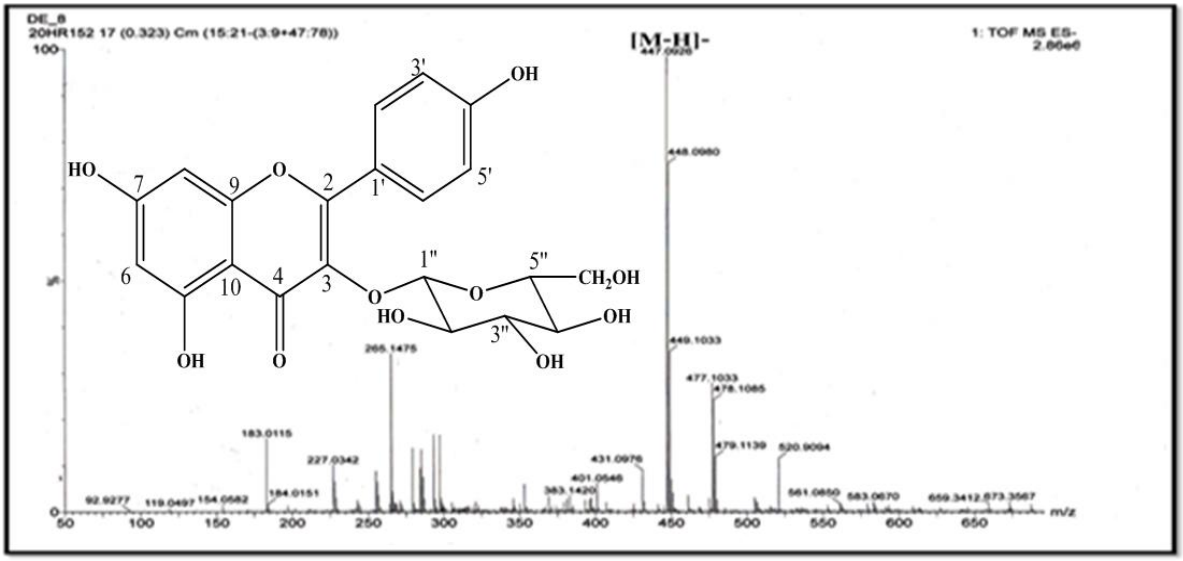


**Figure S8. HR-ESI-MS of compound 2**

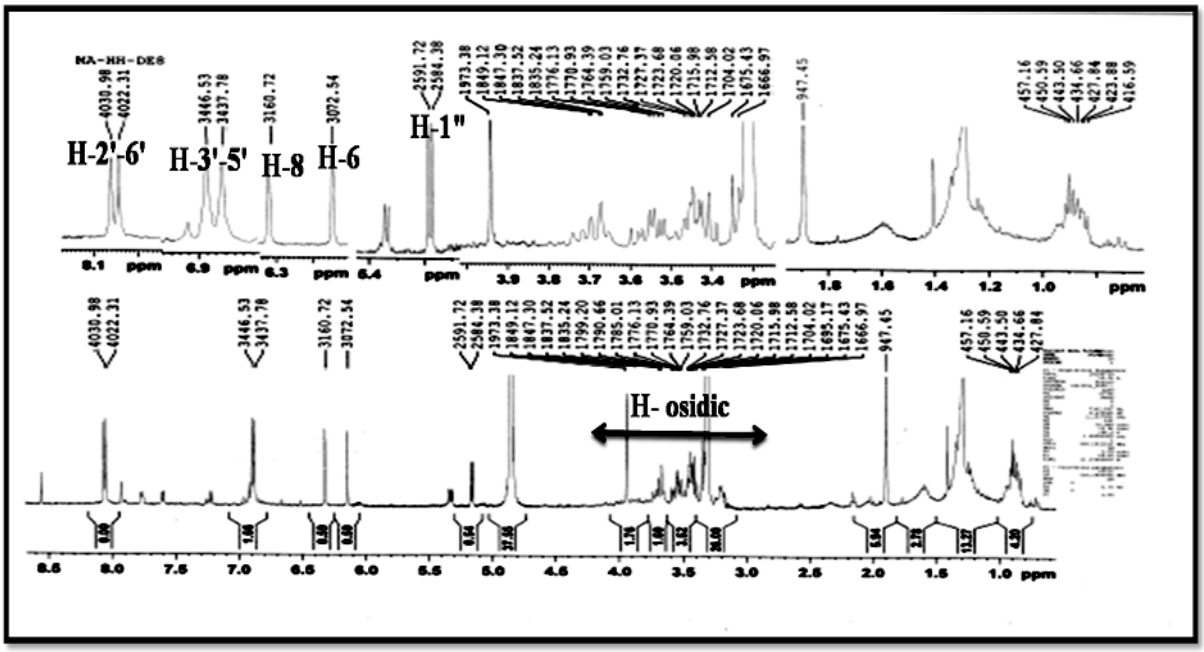


**Figure S9. $^{1}H$ NMR (500 MHz, $CD_3OD$) spectrum of compound 2**

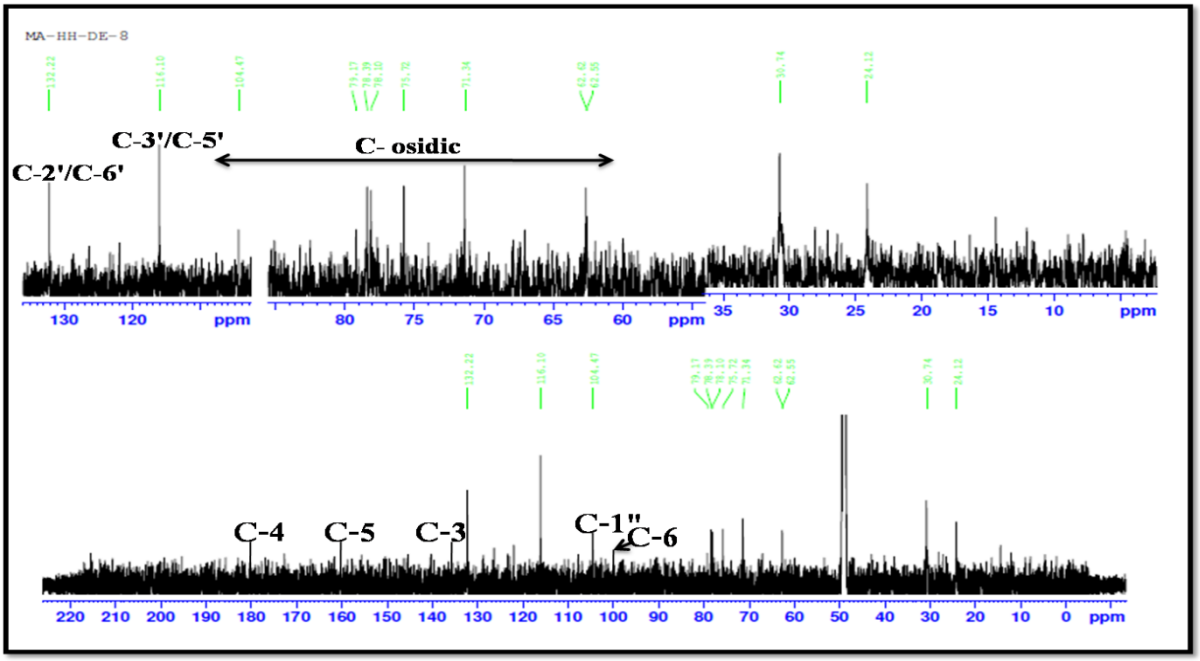


**Figure S10. $^{13}$C-NMR (125 MHz, $CD_3OD$) spectrum of compound 2**

## 3. MS, 1D NMR spectra of compound 3

**1-*O*-*p*-coumaroyl-*β*-D-glucose (3):** Yellow crystals;$^{1}$H and $^{13}$C see **Table S2. HR-ESI-MS** ***m/z***: 325.0924 $[M-H]^-$ with molecular formula $C_{15}H_{18}O_8$

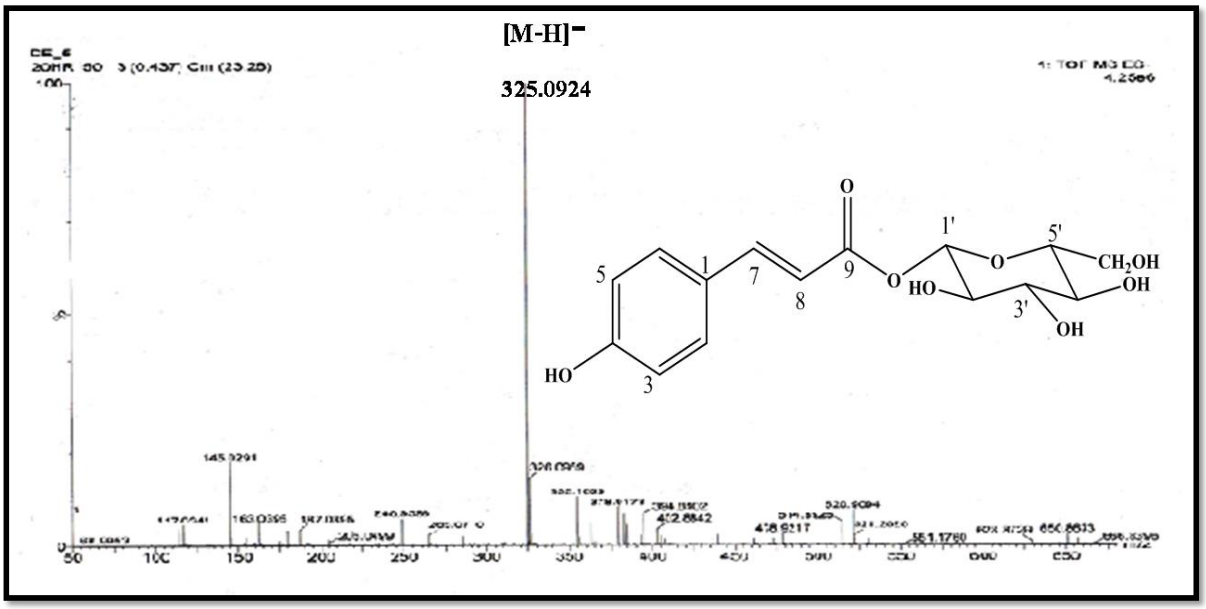


**Figure S11. HR-ESI-MS of compound 3**

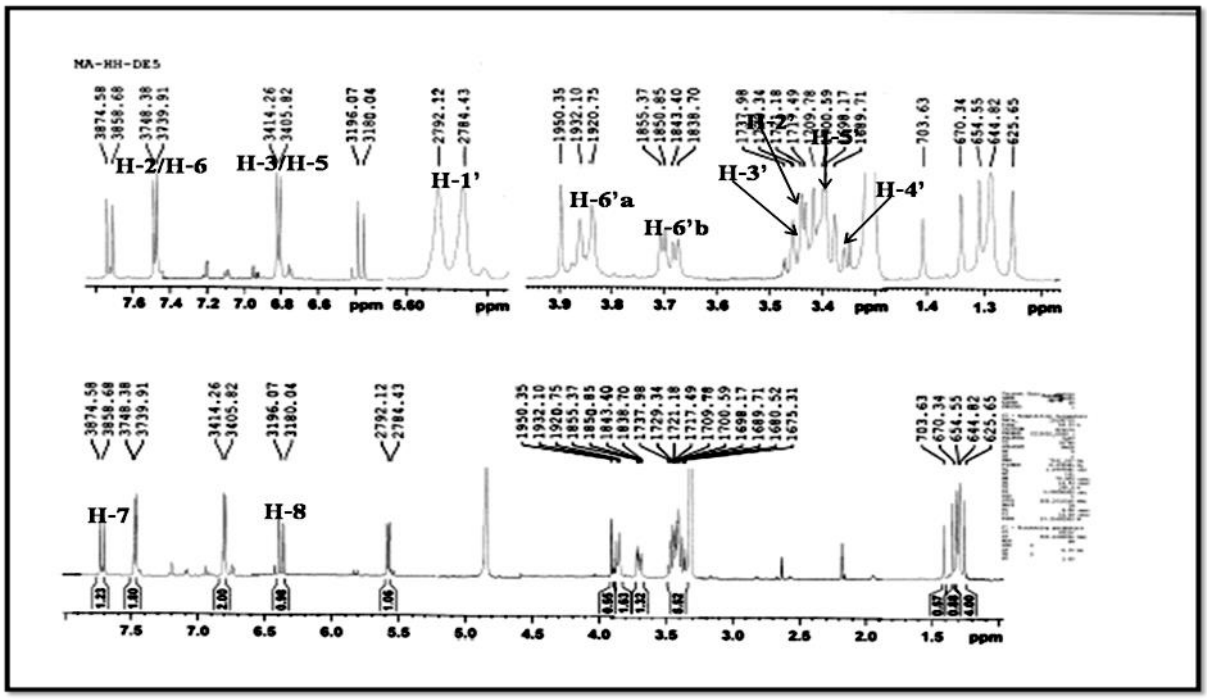


**Figure S12. $^{1}$H NMR (500 MHz, $CD_3OD$) spectrum of compound 3**

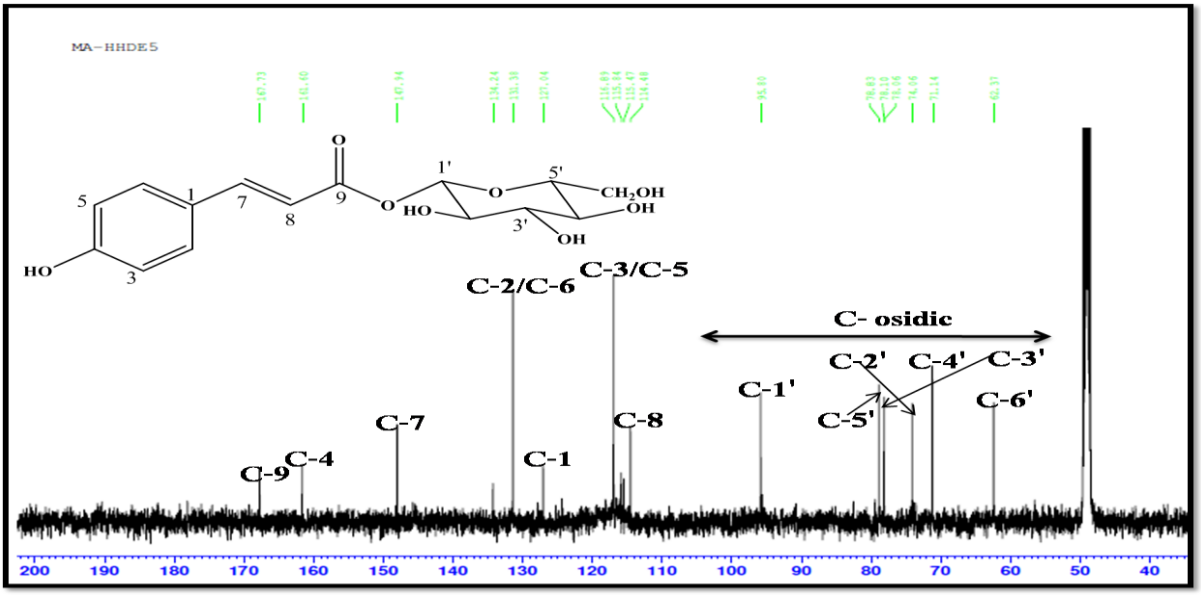


**Figure S13. $^{13}$C-NMR (125 MHz, $CD_3OD$) spectrum of compound 3**

## 4. MS, 1D NMR spectra of compound 4

**Loliolide (4):** Colorless crystals; $[\alpha]_D^{20}$ − 41° (*c* 0.06, $CHCl_3$).$^{1}$H and $^{13}$C see **Table S3. HR-ESI-MS *m/z*** : 219.0998 $[M+Na]^+$, 415.3031 $[2M+Na]^+$ with molecular formula $C_{11}H_{16}O_3$.

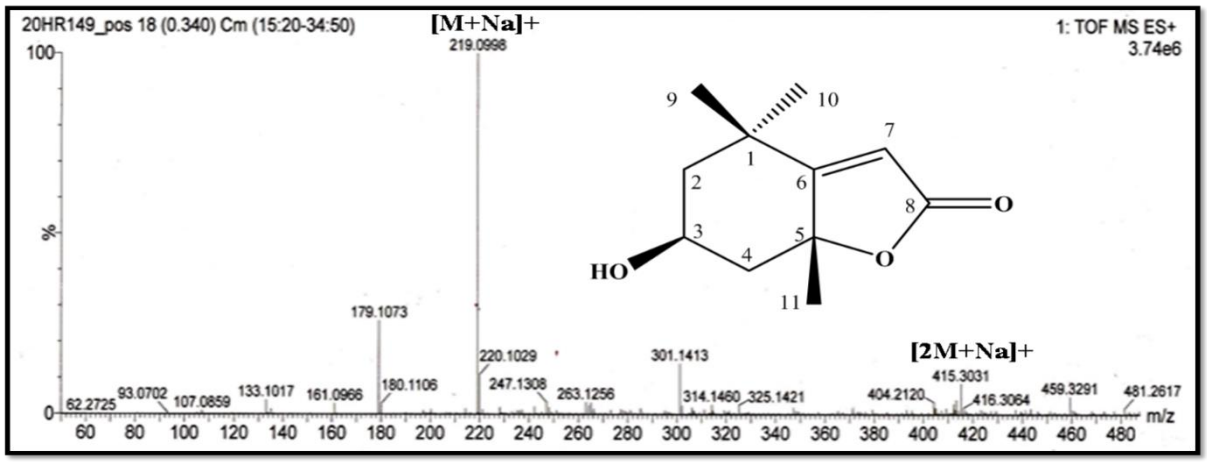


**Figure S14. HR-ESI-MS of compound 4**

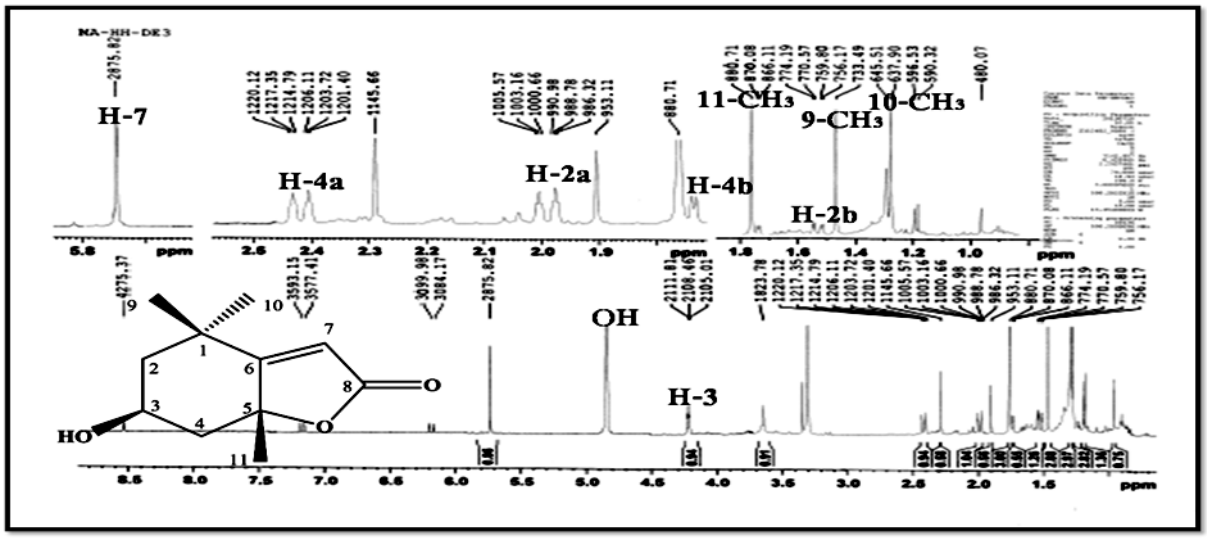


**Figure S15. $^{1}$H NMR (500 MHz, $CD_3OD$) spectrum of compound 4**

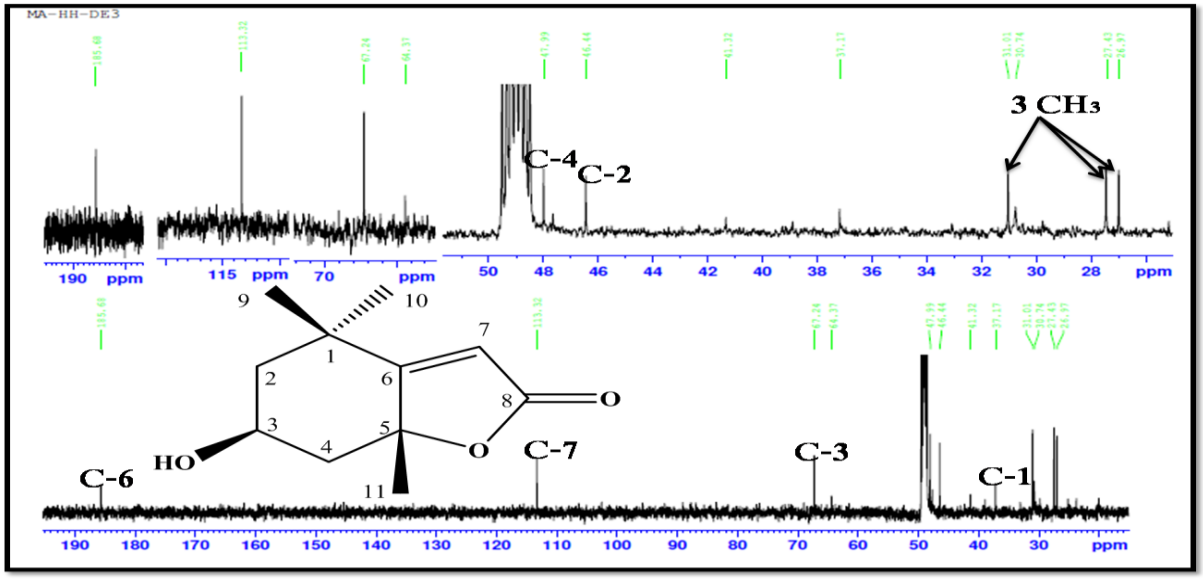


**Figure S16. $^{13}$C-NMR (125 MHz, $CD_3OD$) spectrum of compound 4**

## 5. MS, 1D NMR spectra of compound 5

**Lupeol (5) :** White powder; $[\alpha]_D^{20}$ +22.4° (*c* 0.18, MeOH).$^1$H and $^{13}$C see **Table S4. ESI-MS** ***m/z*** : 449 $[M+Na]^+$, 876 $[2M+Na]^+$ with molecular formula $C_{30}H_{50}O$.

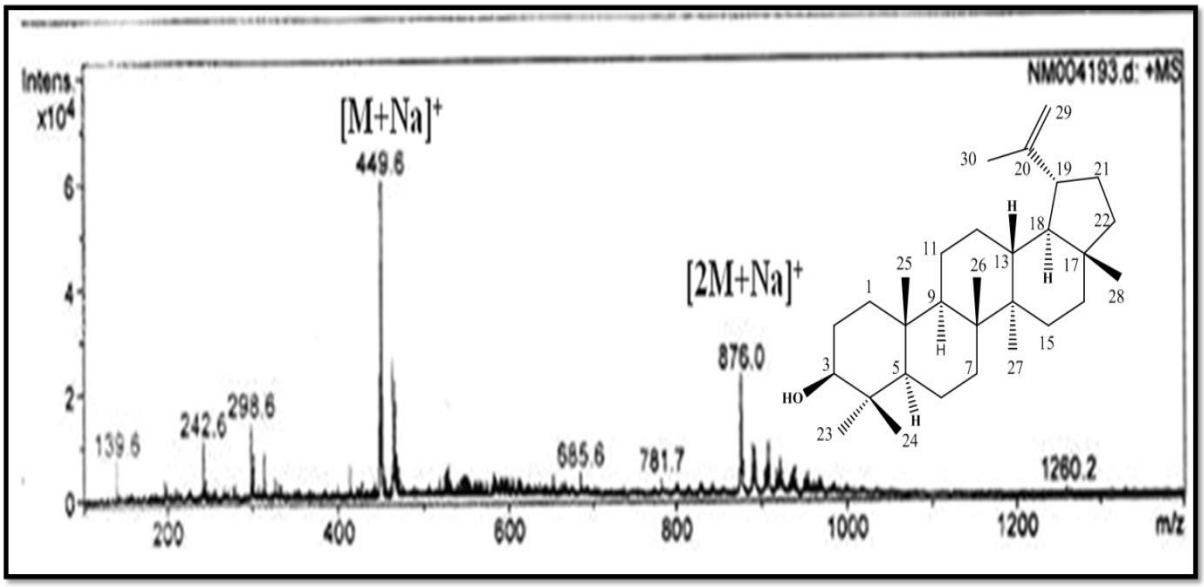


**Figure S17. ESI-MS of compound 5**

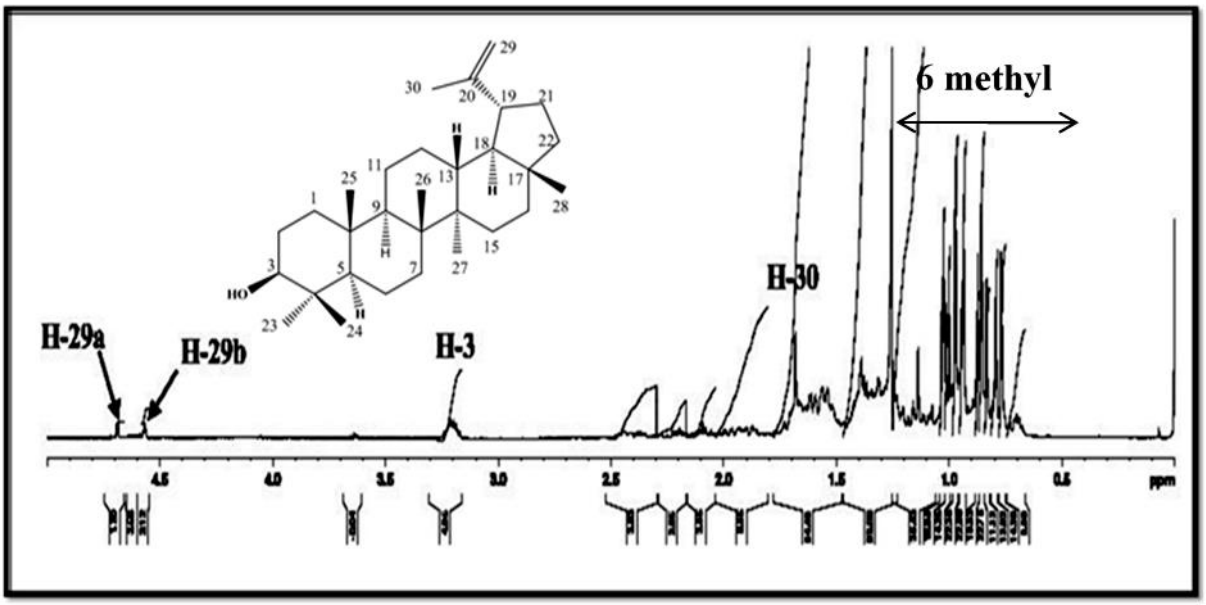


**Figure S18. $^1$H NMR (500 MHz, $CDCl_3$) spectrum of compound 5**

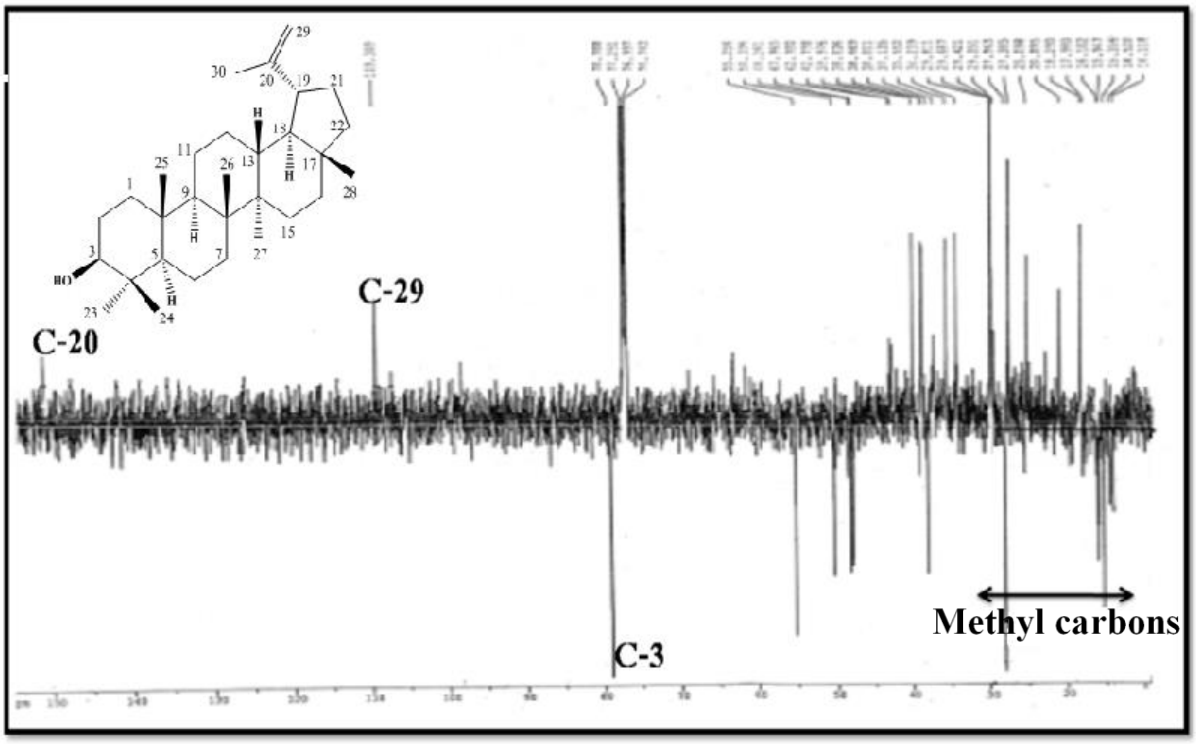


**Figure S19. $^{13}$C-NMR (125 MHz, $CDCl_3$) spectrum of compound 5**

## 6. MS, 1D NMR spectra of compound 6

***β*-sitosterol (6):** Colorless crystals**;** $[\alpha]_D^{20}$−30 (*c* 0,8, $CHCl_3$).$^1$H and $^{13}$C see **Table S4. ESI-MS** ***m/z*** : 413.27 $[M-H]^-$ with molecular formula $C_{29}H_{50}O$

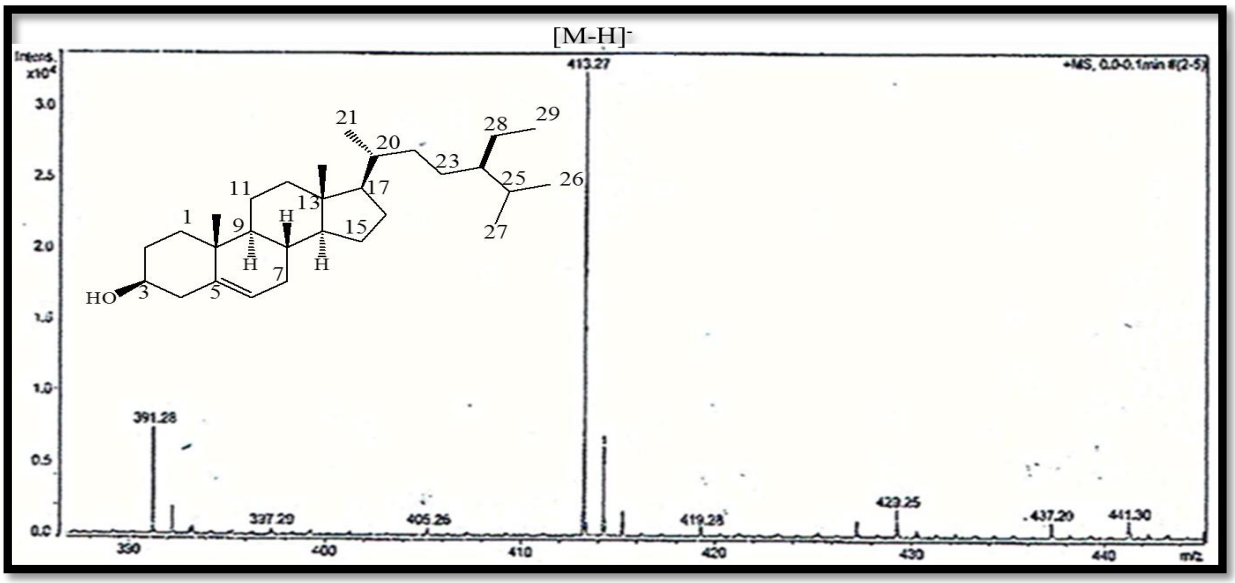


**Figure S20. ESI-MS spectrum of compound 6**

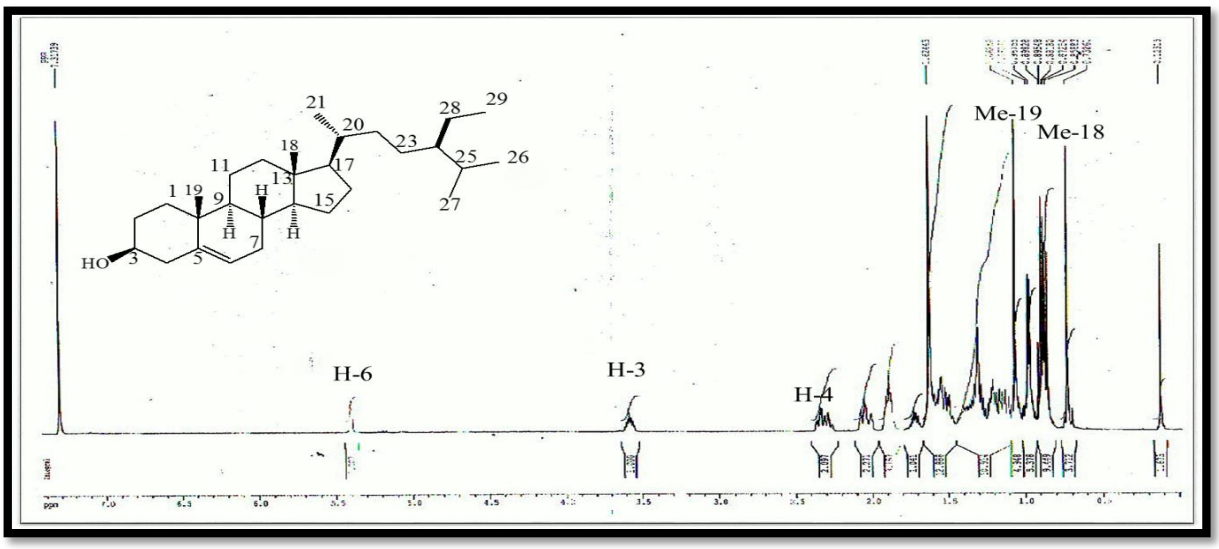


**Figure S21. $^{1}$H NMR (500 MHz, $CDCl_3$) spectrum of compound 6**

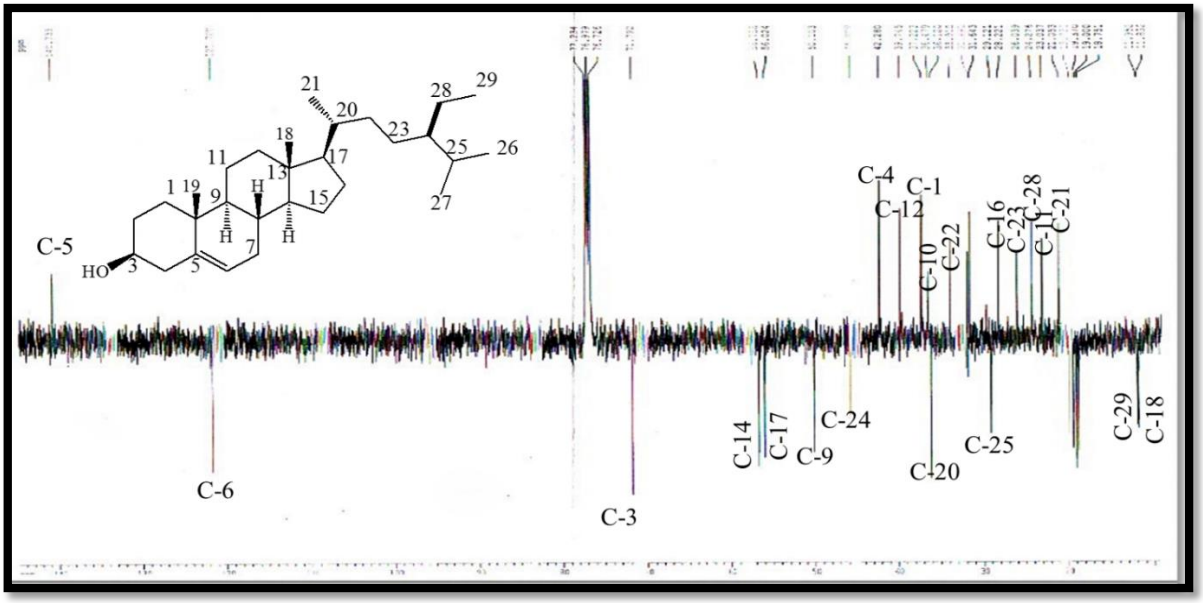


**Figure S22. $^{13}$C-NMR (125 MHz, $CDCl_3$) spectrum of compound 6**

## 7. MS, 1D NMR spectra of compound 7

**(10*E*,12*Z*)-2,3-dihydroxypropyl octadeca-10,12-dienoate (7):** Yellow powder;$^{1}$H and $^{13}$C see **Table S5. ESI-MS *m/z*** : 279 uma $[M-H-C_3H_7O_2]^-$ with molecular formula $C_{18}H_{31}O_2$

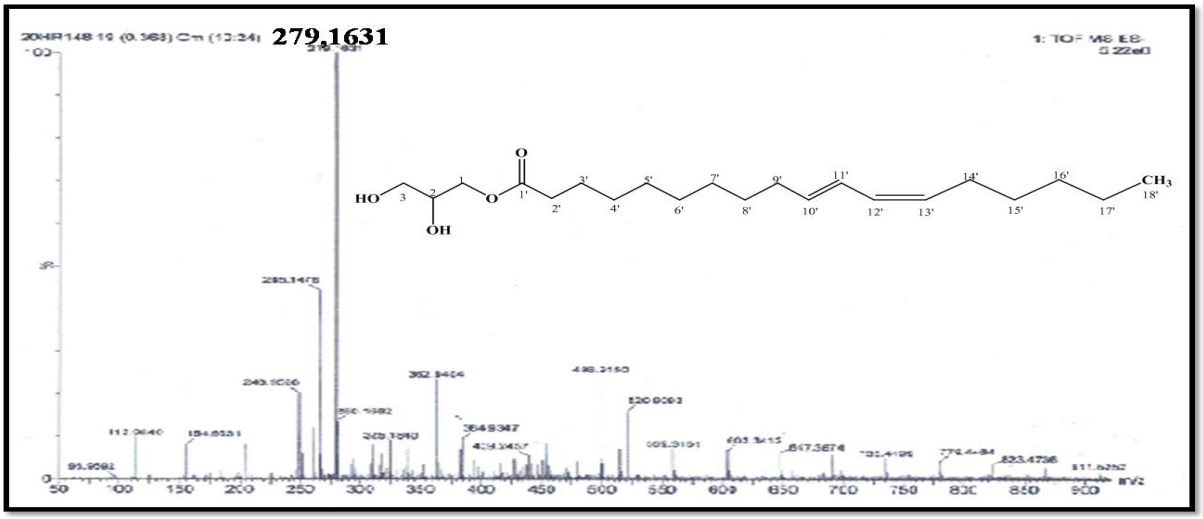


**Figure S23. ESI-MS of compound 7**

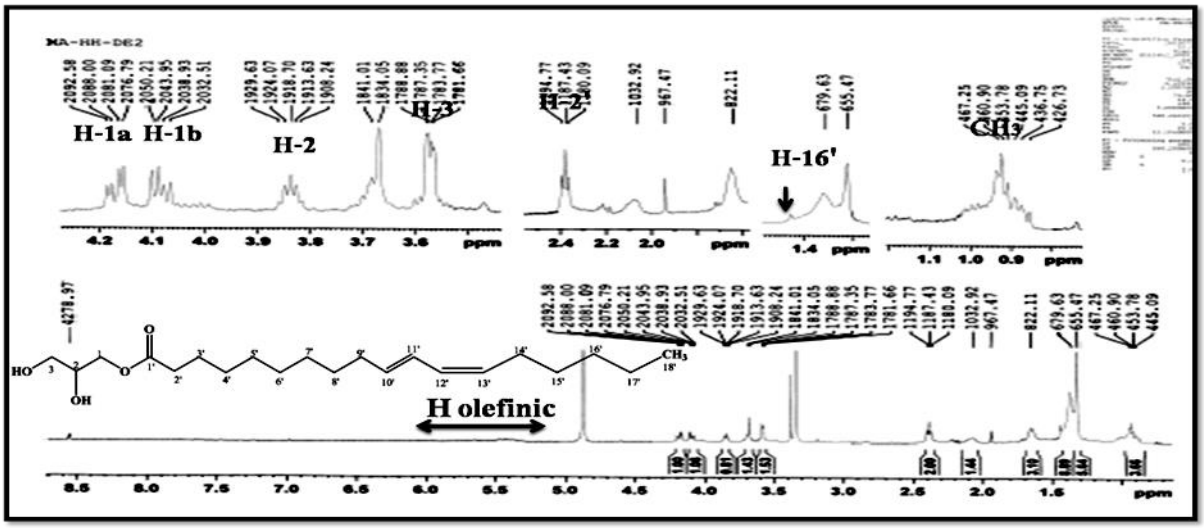


**Figure S24. $^{1}$H NMR (500 MHz, $CD_3OD$) spectrum of compound 7**

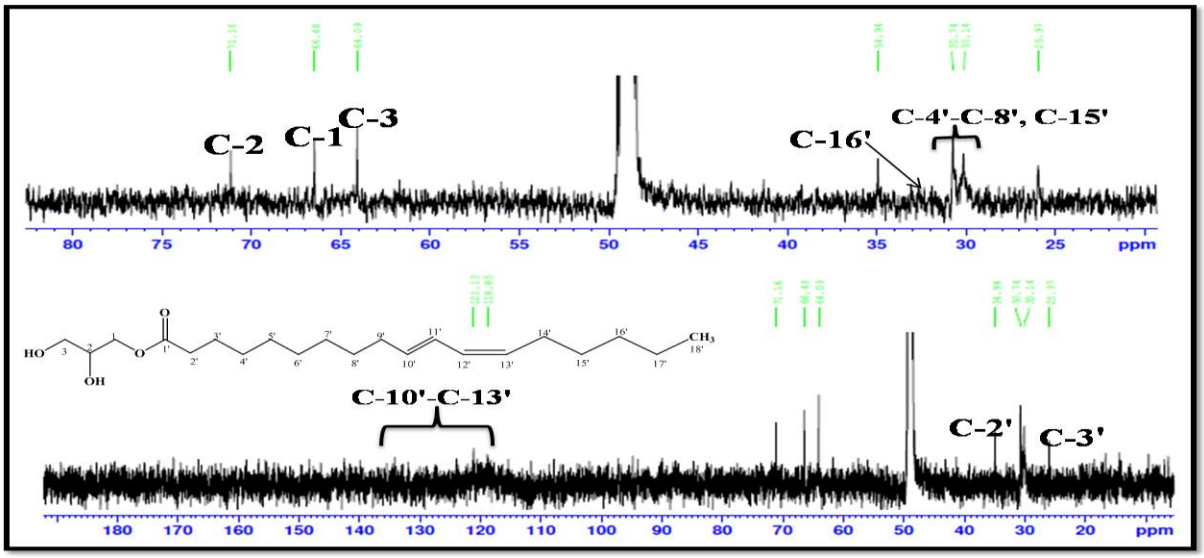


**Figure S25. $^{13}$C-NMR (125 MHz, $CD_3OD$) spectrum of compound 7**

## 8. MS, 1D NMR spectra of compound 8

**(9*Z*,11*E*)-2,3-dihydroxypropyl octadeca-9,11-dienoate (8):** Yellow powder; $^{1}H$ and $^{13}C$ NMR data, see **Table S6. HR-ESI-MS *m/z***: 355.0703 $[M+H]^{+}$. With molecular formula $C_{18}H_{31}O_2$

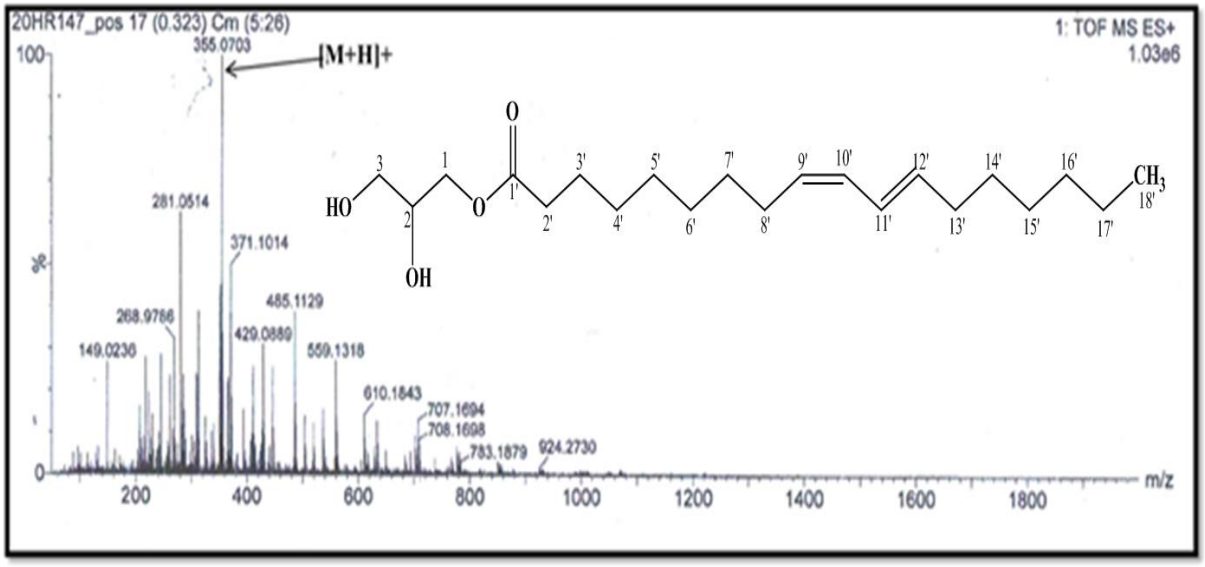


**Figure S26. ESI-MS of compound 8**

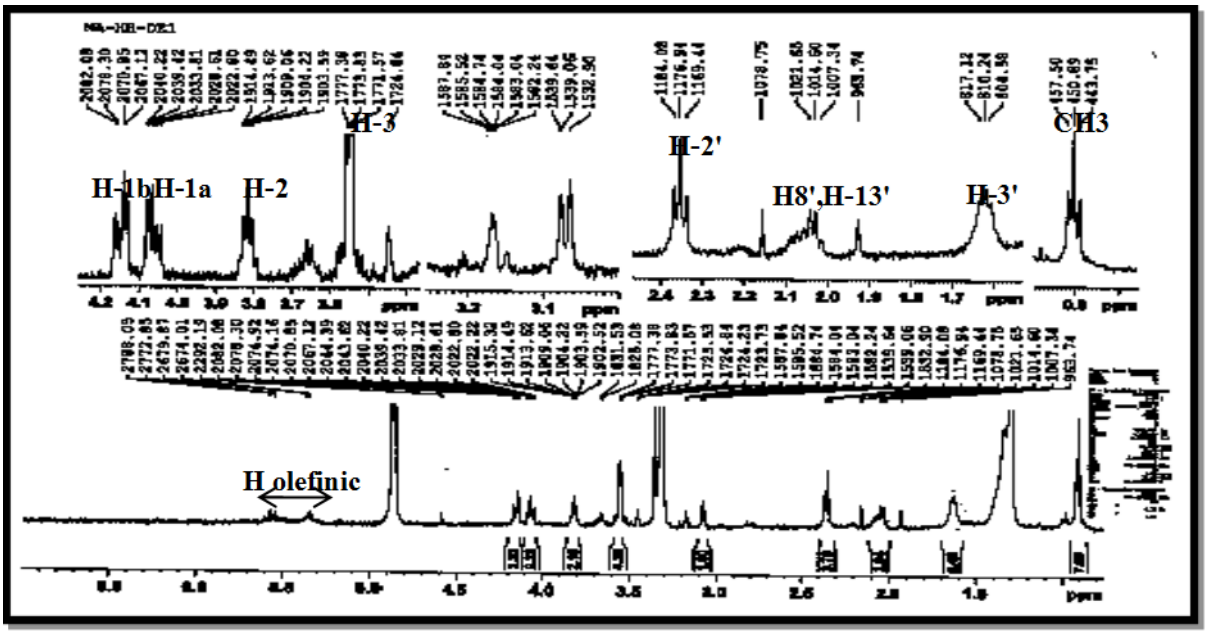


**Figure S27. $^{1}H$ NMR (500 MHz, $CD_3OD$) spectrum of compound 8**

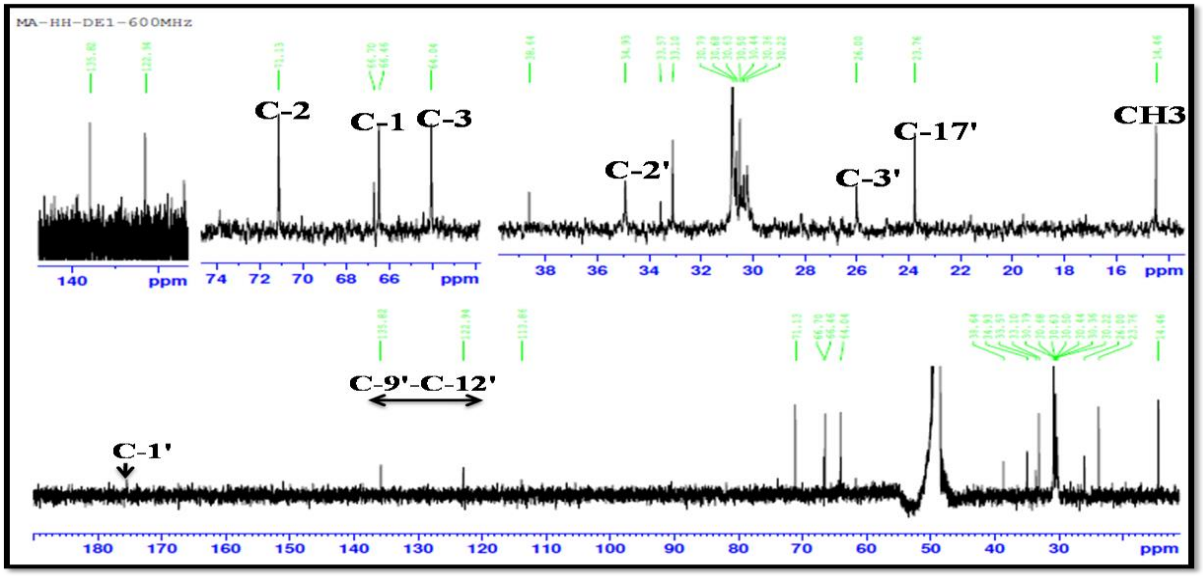


**Figure S28. $^{13}$C-NMR (125 MHz, $CD_3OD$) spectrum of compound 8**

## 9. NMR data of compounds 1-8

**Table S1.** $^{1}$H NMR (500 MHz) and $^{13}$C NMR (125 MHz) data of compounds **1** and **2** in $CD_3OD$ ($\delta$ in ppm, $J$ in Hz).

| No. | 1 | | 2 | |
|---|---|---|---|---|
| | $\delta_C$ | $\delta_H$ | $\delta_C$ | $\delta_H$ |
| 2 | 159.1 | - | 158.0 | - |
| 3 | 134.7 | - | 135.4 | - |
| 4 | 179.0 | - | 179.5 | - |
| 5 | 163.0 | - | 161.0 | - |
| 6 | 100.5 | 6.46 (d, 1.5) | 99.5 | 6.15 (brs) |
| 7 | 163.5 | - | 169.0 | - |
| 8 | 95.5 | 6.75 (d, 1.5) | 95.4 | 6.32 (brs) |
| 9 | 159.0 | - | 158.5 | - |
| 10 | 107.5 | - | 105.0 | - |
| 1′ | 122.7 | - | 123.0 | - |
| 2′ | 132.2 | 8.06 (d, 8.5) | 132.2 | 8.05 (d, 8.7) |
| 3′ | 116.2 | 6.91 (d, 8.5) | 116.1 | 6.88 (d, 8.7) |
| 4′ | 161.7 | - | 161.0 | - |
| 5′ | 116.2 | 6.91 (d, 8.5) | 116.1 | 6.88 (d, 8.7) |
| 6′ | 132.2 | 8.06 (d, 8.5) | 132.2 | 8.05 (d, 8.7) |
| **3-*O*-sugar** | | | | |
| **Xyl** | | | **Glc** | |
| 1″ | 101.2 | 5.63 (d, 7.0) | 104.5 | 5.18 (d, 7.3) |
| 2″ | 79.5 | 3.66 (dd, 9.5, 7.0) | 75.7 | 3.45 (dd, 8.5, 7.3) |
| 3″ | 77.5 | 3.51 (t, 9.5) | 78.1 | 3.42 (t, 8.5) |
| 4″ | 71.4 | 3.49 (t, 9.5) | 71.3 | 3.32 (m) |
| 5″ | 67.2 | 3.74 (dd, 11.5, 5)/3.1 (dd, 11.5, 7.8) | 78.4 | 3.20 (m) |
| 6″ | | | 62.6 | 3.69 (dd, 12.1, 2.3)/3.53 (dd, 11.9, 5.2) |
| **Rha** | | | | |
| 1‴ | 102.7 | 5.22 (brs) | | |
| 2‴ | 72.4 | 4.01 (dd, 3.5, 1.2) | | |
| 3‴ | 72.3 | 3.77 (dd, 9.5, 3.5) | | |
| 4‴ | 74.0 | 3.36 (t, 9.5) | | |
| 5‴ | 70.0 | 4.06 (dd, 9.8, 6.0) | | |
| 6‴ | 17.7 | 1.26 (d, 6.0) | | |
| **7-*O*-sugar** | | | | |
| **Rha** | | | | |
| 1⁗ | 99.9 | 5,56 (brs) | | |
| 2⁗ | 71.7 | 4.02 (dd, 3.4, 1.1) | | |
| 3⁗ | 72.1 | 3.83 (dd, 9.5, 3.4) | | |
| 4⁗ | 73.6 | 3.48 (t, 9.5) | | |
| 5⁗ | 71.3 | 3.60 (dd, 10.2, 6.5) | | |
| 6⁗ | 18.1 | 1.04 (d, 6.5) | | |

**Table S2.** $^{1}$H NMR (500 MHz) and $^{13}$C NMR (125 MHz) data of compound **3** in $CD_3OD$ ($\delta$ in ppm, $J$ in Hz).

| No. | **3** | |
|---|---|---|
| | $\delta_C$ | $\delta_H$ |
| 1 | 127.0 | - |
| 2 | 131.4 | 7.49 (d, 8.5) |
| 3 | 116.9 | 6.82 (d, 8.5) |
| 4 | 161.6 | - |
| 5 | 116.9 | 6.82 (d, 8.5) |
| 6 | 131.4 | 7.49 (d, 8.5) |
| 7 | 147.9 | 7.77 (d, 15.9) |
| 8 | 114.5 | 6.38 (d, 16.0) |
| 9 | 167.7 | - |
| **Glc** | | |
| 1′ | 95.8 | 5.55 (d, 7.7) |
| 2′ | 74.1 | 3.43 (t, 7.7) |
| 3′ | 78.1 | 3.46 (t, 8.6) |
| 4′ | 71.1 | 3.38 (t, 8.4) |
| 5′ | 78.8 | 3.4 (m) |
| 6′ | 62.4 | 3.85 (brd, 11.3)/3.7 (dd,11.9, 4.6) |

**Table S3.** $^{1}$H NMR (500 MHz) and $^{13}$C NMR (125 MHz) data of compound **4** in $CD_3OD$ ($\delta$ in ppm, $J$ in Hz).

| No. | **4** | |
|---|---|---|
| | $\delta_C$ | $\delta_H$ |
| 1 | 37.2 | - |
| 2 | 48.0 | 1.99 (dt, 14.4, 2.3) /1.52 (dd, 14.1, 3.6) |
| 3 | 67.2 | 4.2 (quint, 3.3) |
| 4 | 46.4 | 2.42 (dt, 13.7, 2.4) /1.75 (dd, 13.6, 4.0) |
| 5 | 89.0 | - |
| 6 | 185.7 | - |
| 7 | 113.3 | 5.75 (s) |
| 8 | 172.0 | - |
| 9 | 27.0 | 1.47 (s) |
| 10 | 31.0 | 1.28 (s) |
| 11 | 27.4 | 1.77 (s) |

**Table S4.** $^{1}$H NMR (500 MHz) and $^{13}$C NMR (125 MHz) data of compounds **5** and **6** in $CDCl_3$ ($\delta$ in ppm, $J$ in Hz).

| No. | **5** | | **6** | |
|---|---|---|---|---|
| | $\delta_C$ | $\delta_H$ | $\delta_C$ | $\delta_H$ |
| 1 | 38.7 | 1.72 (m)/ 0.96 (m) | 37.2 | 1.9 (m) / 1.15 (m) |
| 2 | 27.4 | 1.65 (m)/ 1.58 (m) | 31.6 | 1.88 (m) / 1.57 (m) |
| 3 | 79.0 | 3.2 (m) | 71.8 | 3.59 (tt, 11.3, 5.1) |
| 4 | 39.9 | - | 42.3 | 2.36 (dd, 13.1, 5.1) / 2.29 (t, 13.1) |
| 5 | 55.3 | 0.69 (m) | 140.7 | - |
| 6 | 18.3 | 1.56 (m)/ 1.44 (m) | 121.7 | 5.45 (dd, 5.2, 2.3) |
| 7 | 34.2 | 1.48 (m) / 1.42 (m) | 31.8 | 2.08 (m) / 1.56 (m) |
| 8 | 41.0 | - | 31.7 | 1.49 (dd, 11.2, 4.6) |
| 9 | 50.4 | 1.3 (brs) | 50.1 | 0.97 (m) |
| 10 | 37.1 | - | 36.5 | - |
| 11 | 20.9 | 1.47 (m) / 1.28 (m) | 21.1 | 1.55 (m) / 1.53 (m) |
| 12 | 25.1 | 1.73 (m) / 1.13 (m) | 39.7 | 2.08 (m) / 1.22 (m) |
| 13 | 38.0 | 1.68 (m) | 42.2 | - |
| 14 | 42.8 | - | 56.7 | 1.0 (m) |
| 15 | 27.9 | 1.62 (m) / 1.04 (m) | 21.1 | 1.63 (m) / 1.11 (m) |
| 16 | 35.6 | 1.52 (m) / 1.41 (m) | 28.2 | 1.9 (m)/ 1.3 (m) |
| 17 | 43.0 | - | 56.0 | 1.15 (m) |
| 18 | 48.3 | 1.39 (m) | 11.8 | 0.72 (s) |
| 19 | 48.0 | 2.42 (m) | 19.4 | 1.05 (s) |
| 20 | 151.2 | - | 36.1 | 1.4 (m) |
| 21 | 29.8 | 1.97 (m) / 1.27 (m) | 18.8 | 0.97 (d, 6.5) |
| 22 | 40.2 | 1.43 (m)/ 1.24 (m) | 33.9 | 1.48 (m)/ 1.31 (m) |
| 23 | 28.2 | 1.02 (s) | 26.0 | 1.21 (m) |
| 24 | 15.4 | 0.82 (s) | 45.8 | 0.97 (m) |
| 25 | 15.9 | 0.88 (s) | 29.1 | 0.72 (m) |
| 26 | 16.1 | 1.0.8 (s) | 19.8 | 0.88 (d,6.8) |
| 27 | 14.5 | 0.99 (s) | 19.0 | 0.86 (d, 6.8) |
| 28 | 18.1 | 0.83 (s) | 23.0 | 1.32 (m) / 1.29 (m) |
| 29 | 109.3 | 4.68 (brs)/ 4.58 (brs) | 11.9 | 0.9 (t, 6.5) |
| 30 | 19.5 | 1.74(s) | | |

**Table S5.** $^{1}$H NMR (500 MHz) and $^{13}$C NMR (125 MHz) data of compound **7** in $CD_3OD$ ($\delta$ in ppm, $J$ in Hz).

| **No.** | **7** | |
|---|---|---|
| | $\delta_C$ | $\delta_H$ |
| 1 | 66.5 | 4.15 (dd, 11, 5.8) / 4.15 (dd, 11.2, 3.8) |
| 2 | 71.1 | 3.82 (quint, 5.4) |
| 3 | 64.0 | 3.56 (dd, 5.8, 3.5) |
| 1′ | 174.0 | - |
| 2′ | 34.9 | 2.35 (t, 7.2) |
| 3′ | 24.8 | 1.62 (m) |
| 4'-7', 14′, 15' | 28.0-30.5 | 1.25-1.40(m) |
| 8′ | nd | 2.05(m) |
| 9′-12′ | 122.9-135.8 | 5.30-6.30(m) |
| 13′ | 33.6 | 2.05(m) |
| 16′ | 31.5 | 1.25-1.40 (m) |
| 17′ | 23.8 | 1.24-1.43 (m) |
| 18′ | 14.5 | 0.90 (t, 6.9) |

**Table S6.** $^{1}$H NMR (500 MHz) and $^{13}$C NMR (125 MHz) data of compound **8** in $CD_3OD$ ($\delta$ in ppm, $J$ in Hz).

| No. | 8 | |
|---|---|---|
| | $\delta_C$ | $\delta_H$ |
| 1 | 66.5 | 4.16 (dd, 11.5, 4.6) / 4.08 (dd, 11.3, 6.3) |
| 2 | 71.2 | 3.84 (quint, 5.4) |
| 3 | 64.1 | 3.75 (dd,5.1, 1.5) |
| 1′ | 175.0 | - |
| 2′ | 34.9 | 2.38 (t, 7.3) |
| 3′ | 26.0 | 1.65 (m) |
| 4'-8',15' | 28.0-30.5 | 1.24-1.43(m) |
| 9′ | 30.7 | nd |
| 10′-13′ | 121.0-135.0 | 5.3-6.3 (m) |
| 14′ | nd | nd |
| 16′ | 31.5 | 1.42 (m) |
| 17′ | 22.1 | 1.24-1.43 (m) |
| 18′ | 14.0 | 0.92 (t, 6.3) |