

Characterization of flavonoid and quinic acid derivatives from *Calystegia soldanella* with anti-oxidant activities

Ji-Yul Kim^{1,†,*}, Gun-Woo Oh^{1,†}, Dae-Cheol Choi², Kyung Lee¹, Seok-Chun Ko¹, Mi-Jin Yim¹, Jeong Min Lee¹, Dae-Won Ki², Kyung Woo Kim¹, Chul Hwan Kim¹, Moongeun Yoon¹ and Dae-Sung Lee^{1,*}

¹National Marine Biodiversity Institute of Korea, Seochon, Republic of Korea

²Division of Biotechnology and Advanced Institute of Environmental and Bioscience, College of Environmental and Bioresource Sciences, Jeonbuk National University, Iksan-si, Republic of Korea

* Authors to whom correspondence should be addressed.

†These authors equally contributed to this work.

Corresponding authors:

National Marine Biodiversity Institute of Korea, Seochon, Republic of Korea.

E-mail: jiyul2224@mabik.re.kr, daesung@mabik.re.kr

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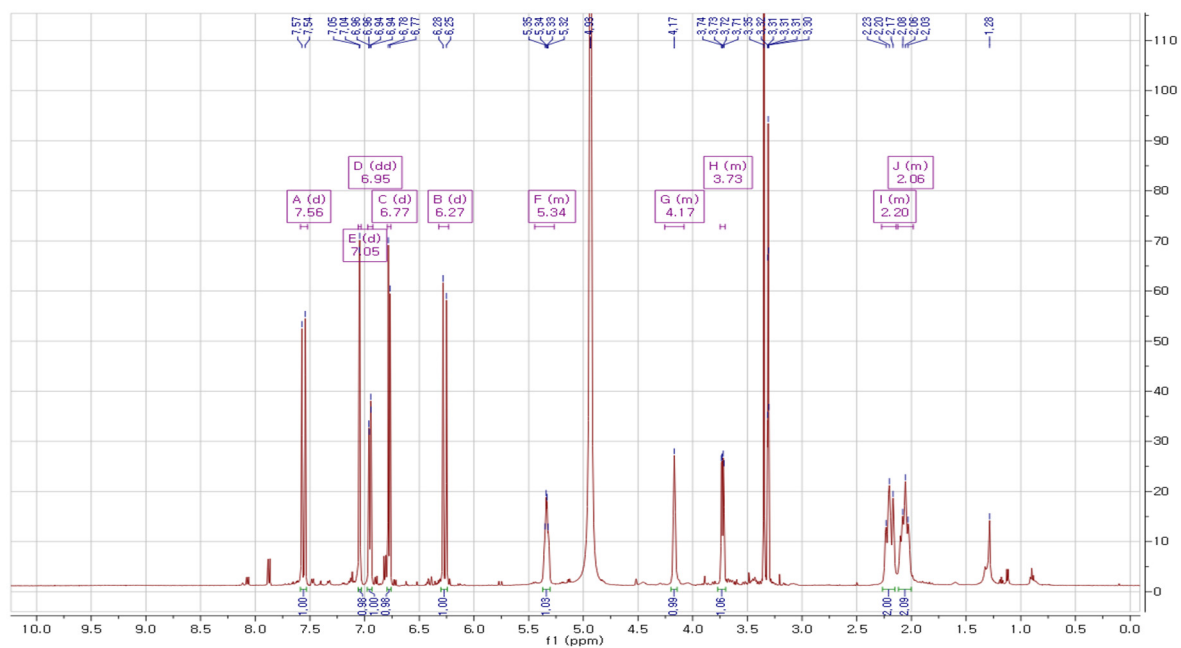


Figure S1. ^1H NMR spectrum of compound 1

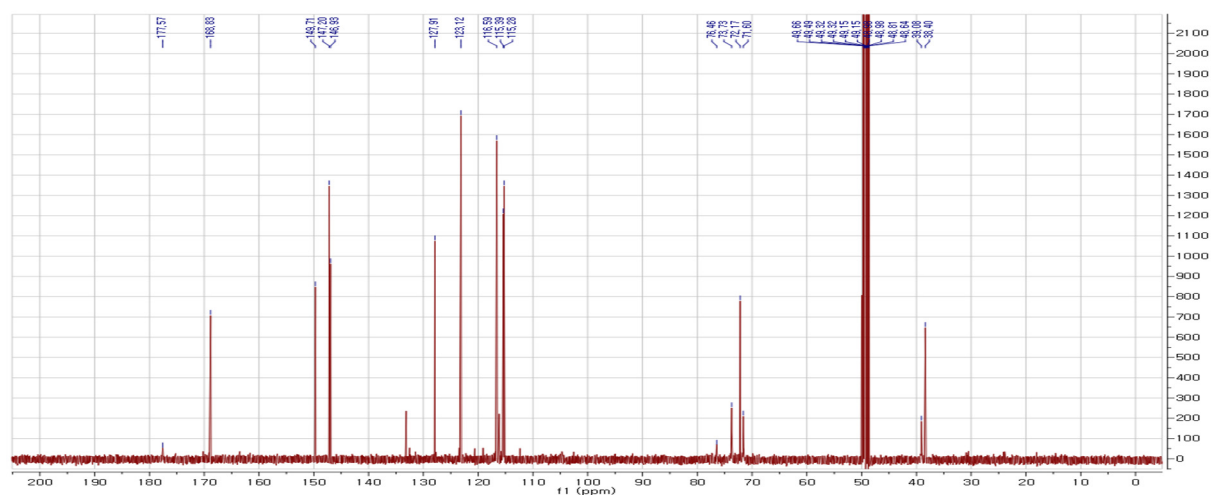
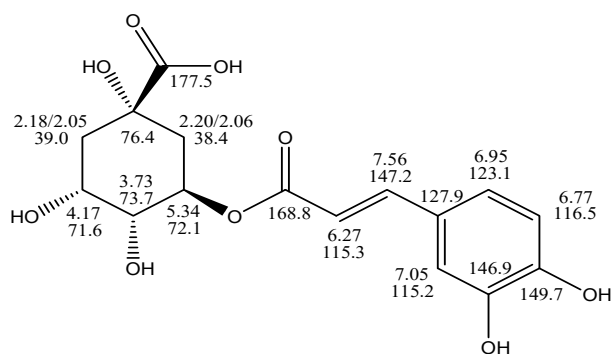


Figure S2. ^{13}C NMR spectrum of compound 1



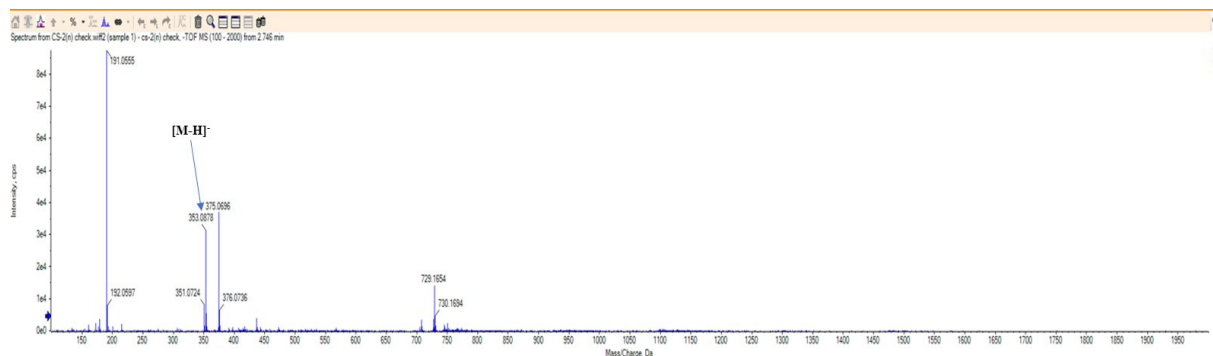


Figure S3. HR-ESI-MS spectrum of compound **1**

Chlorogenic acid (**1**) : HR-ESI-MS (negative) m/z 353.0878 $[M-H]^-$, calcd for $C_{16}H_{17}O_9$ 353.0873; 1H NMR (500 MHz, CD_3OD): δ 7.56 (1H, d, $J = 15.9$ Hz, H-7'), 7.05 (1H, d, $J = 1.8$ Hz, H-2'), 6.95 (1H, dd, $J = 8.2, 1.8$ Hz, H-6'), 6.77 (1H, d, $J = 8.2$ Hz, H-5'), 6.27 (1H, d, $J = 15.9$ Hz, H-8'), 5.34 (1H, m, H-3), 4.17 (1H, m, H-5), 3.73 (1H, m, H-4), 2.20/2.06 (2H, m, H-2), 2.18/2.05 (2H, m, H-6); ^{13}C NMR (125 MHz, CD_3OD): δ 177.5 (C-7), 168.8 (C-9'), 149.7 (C-4'), 147.2 (C-7'), 146.9 (C-3'), 127.9 (C-1'), 123.1 (C-6'), 116.5 (C-5'), 115.3 (C-8'), 115.2 (C-2'), 76.4 (C-1), 73.7 (C-4), 72.1 (C-3), 71.6 (C-5), 39.0 (C-6), 38.4 (C-2).

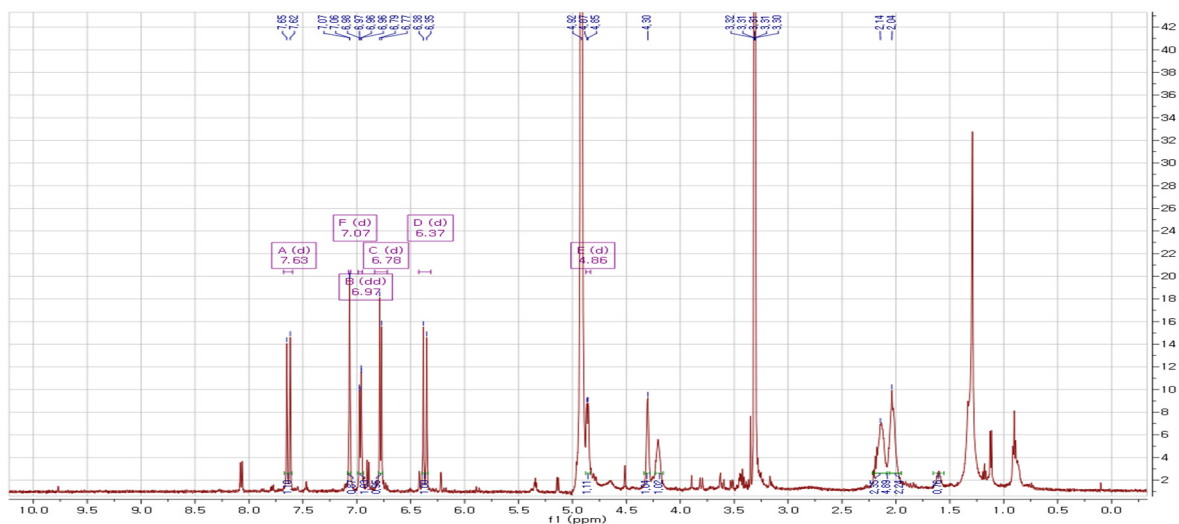


Figure S4. ^1H NMR spectrum of compound **2**

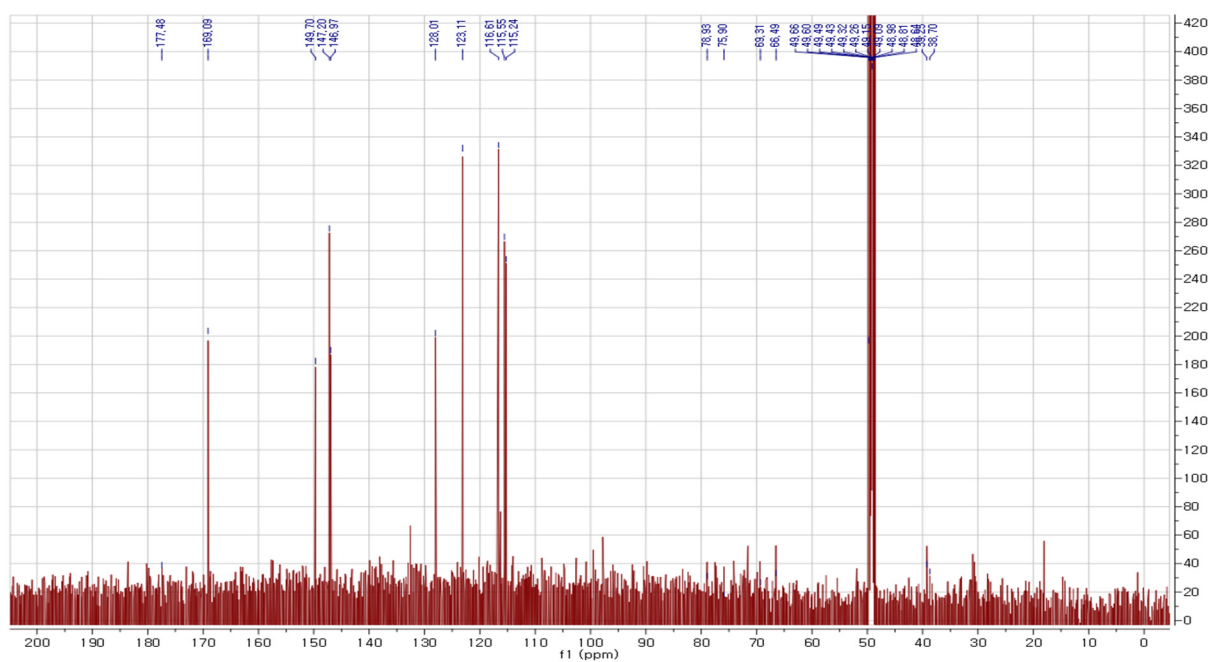
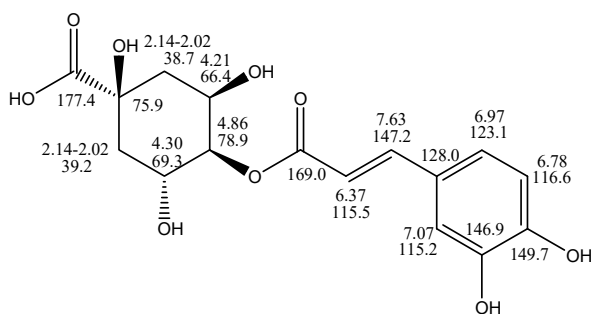


Figure S5. ^{13}C NMR spectrum of compound **2**



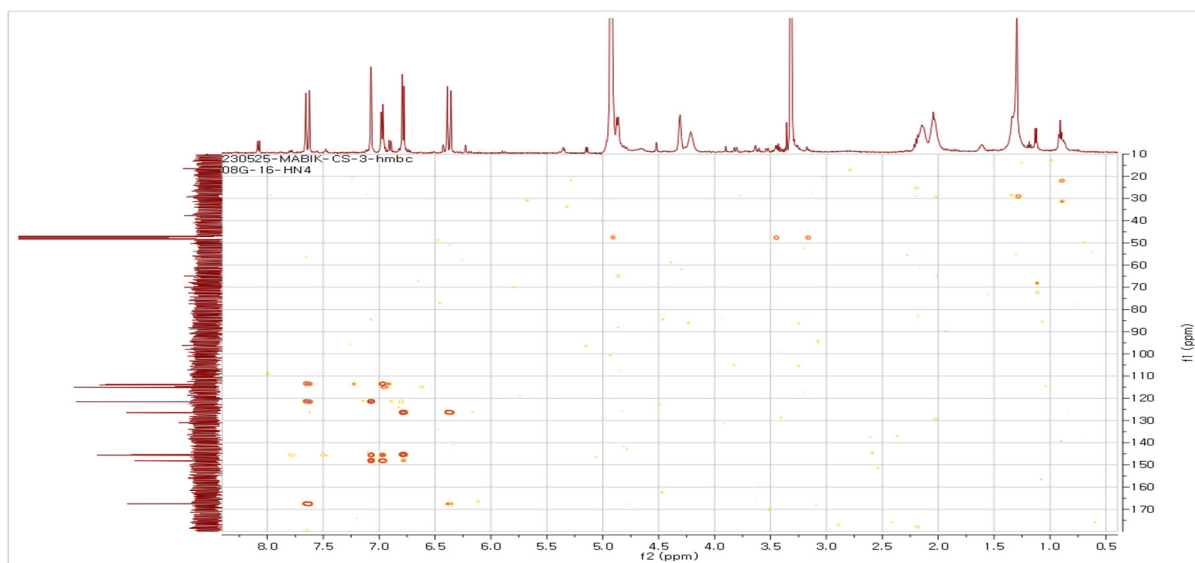


Figure S6. HMBC spectrum of compound **2**

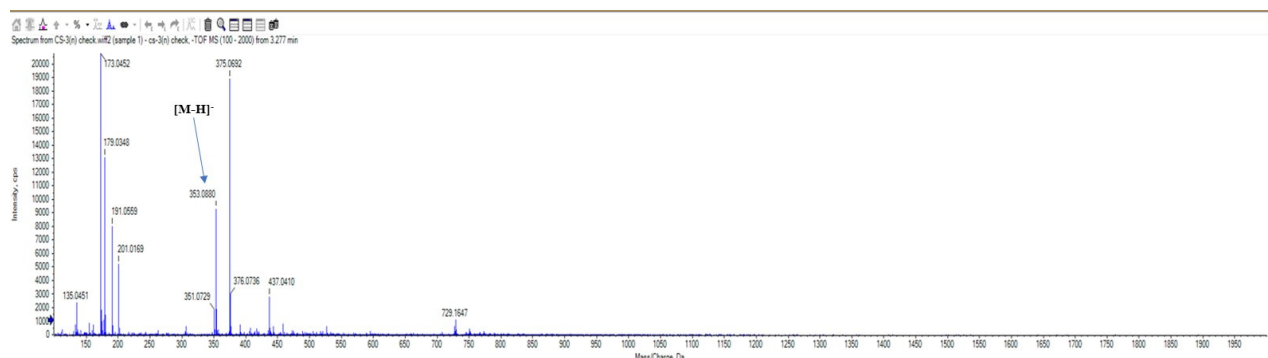


Figure S7. HR-ESI-MS spectrum of compound **2**

Cryptochlorogenic acid (**2**) : HR-ESI-MS (negative) m/z 353.0880 $[M-H]^-$, calcd for $C_{16}H_{17}O_9$ 353.0873; 1H NMR (500 MHz, CD_3OD): δ 7.63 (1H, d, $J = 15.9$ Hz, H-7'), 7.07 (1H, d, $J = 1.7$ Hz, H-2'), 6.97 (1H, dd, $J = 8.1, 1.7$ Hz, H-6'), 6.78 (1H, d, $J = 8.1$ Hz, H-5'), 6.37 (1H, d, $J = 15.9$ Hz, H-8'), 4.86 (1H, m, overlap, H-3), 4.30 (1H, m, H-4), 4.21 (1H, m, H-2), 2.14-2.02 (4H, m, H-1 and 5); ^{13}C NMR (125 MHz, CD_3OD): δ 177.4 (C-7), 169.0 (C-9'), 149.7 (C-4'), 147.2 (C-7'), 146.9 (C-3'), 128.0 (C-1'), 123.1 (C-6'), 116.6 (C-5'), 115.5 (C-8'), 115.2 (C-2'), 78.9 (C-3), 75.9 (C-6), 69.3 (C-4), 66.4 (C-2), 39.2 (C-5), 38.7 (C-1).

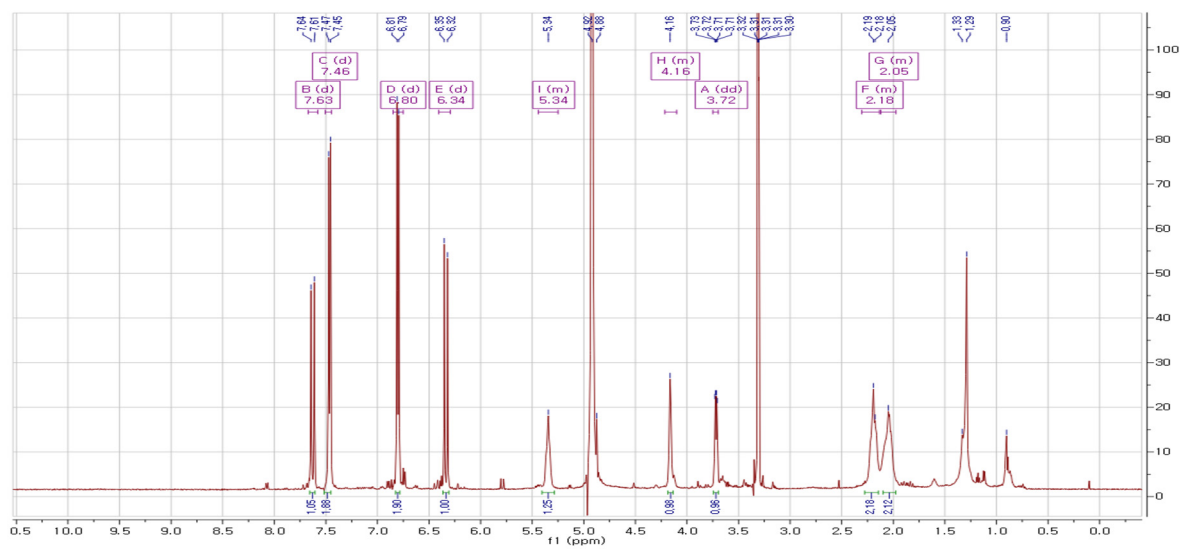


Figure S8. ^1H NMR spectrum of compound **3**

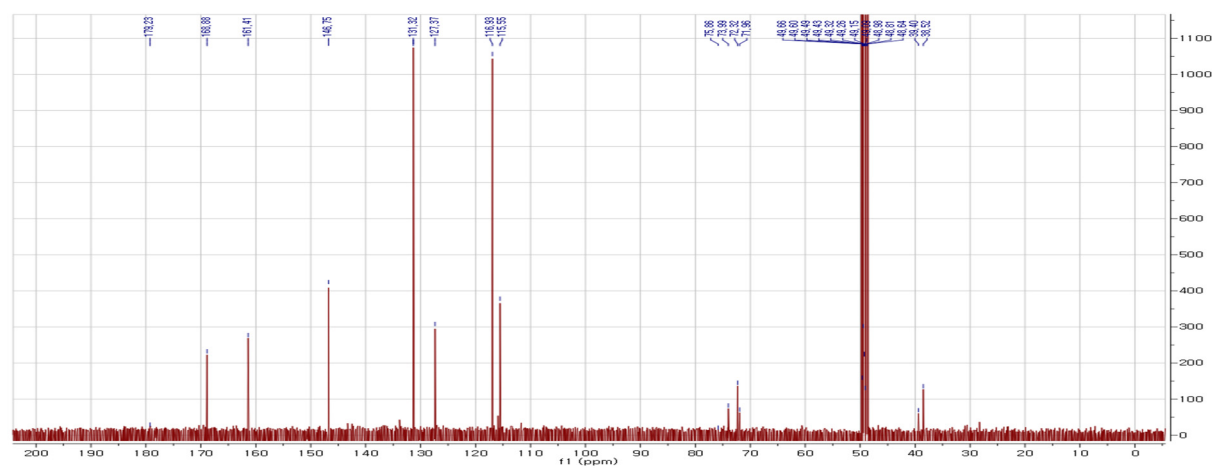
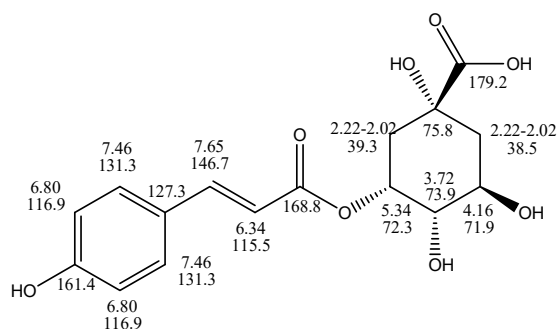


Figure S9. ^{13}C NMR spectrum of compound **3**



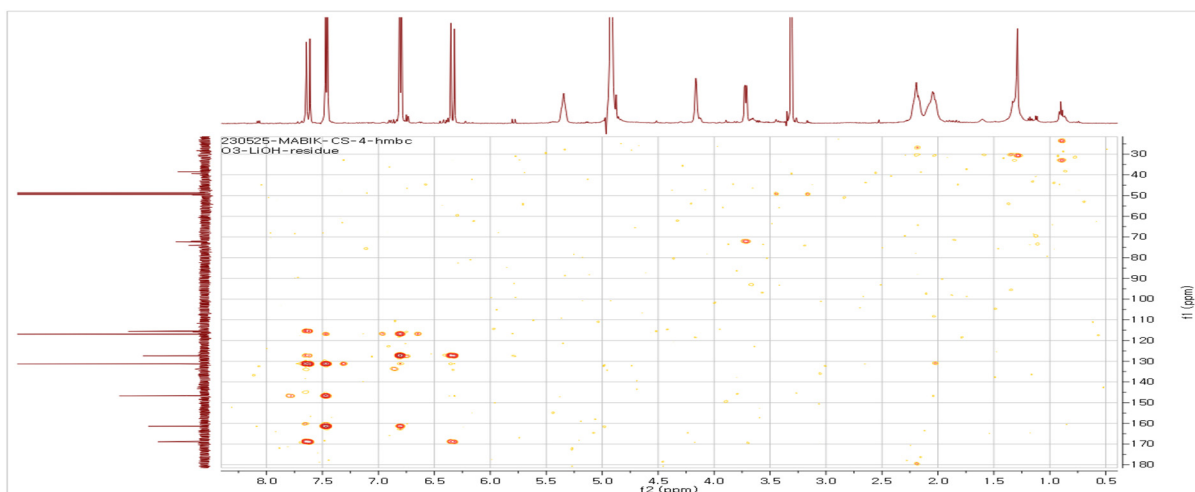


Figure S10. HMBC spectrum of compound **3**

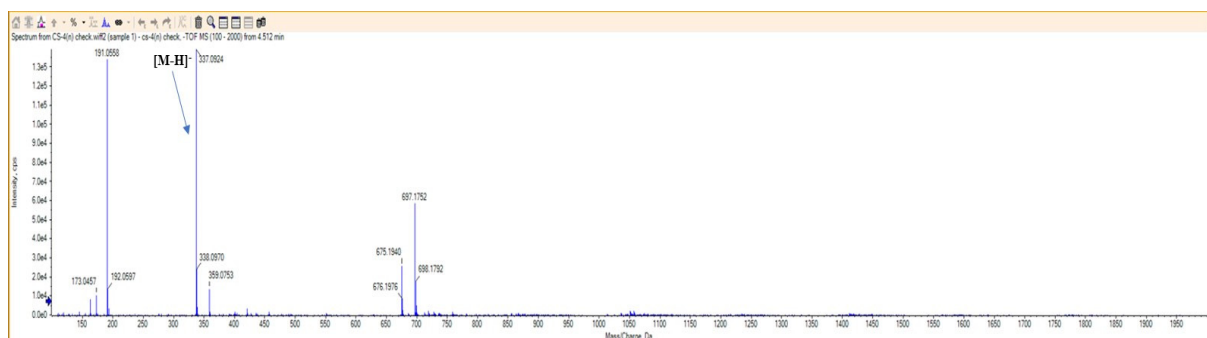
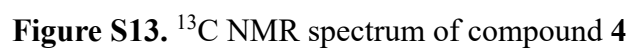
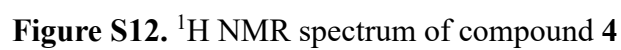


Figure S11. HR-ESI-MS spectrum of compound **3**

5-O-(*E*)-*p*-coumaroylquinic acid (**3**) : HR-ESI-MS (negative) m/z 337.0924 $[M-H]^-$, calcd for $C_{16}H_{17}O_8$ 337.0923; 1H NMR (500 MHz, CD_3OD): δ 7.63 (1H, d, $J = 15.9$ Hz, H-7'), 7.46 (2H, d, $J = 8.5$ Hz, H-2' and H-6'), 6.80 (2H, d, $J = 8.5$ Hz, H-3' and H-5'), 6.34 (1H, d, $J = 15.9$ Hz, H-8'), 5.34 (1H, m, H-3), 4.16 (1H, m, H-5), 3.72 (1H, dd, $J = 8.5, 2.6$ Hz), 2.22-2.02 (4H, m, H-2 and H-6); ^{13}C NMR (125 MHz, CD_3OD): δ 179.2 (C-7) 168.8 (C-9'), 161.4 (C-4'), 146.7 (C-7'), 131.3 (C-2' and 6'), 127.3 (C-1'), 116.9 (C-3' and 5'), 115.5 (C-8'), 75.8 (C-1), 73.9 (C-4), 72.3 (C-3), 71.9 (C-5), 39.3 (C-2), 38.5 (C-6).



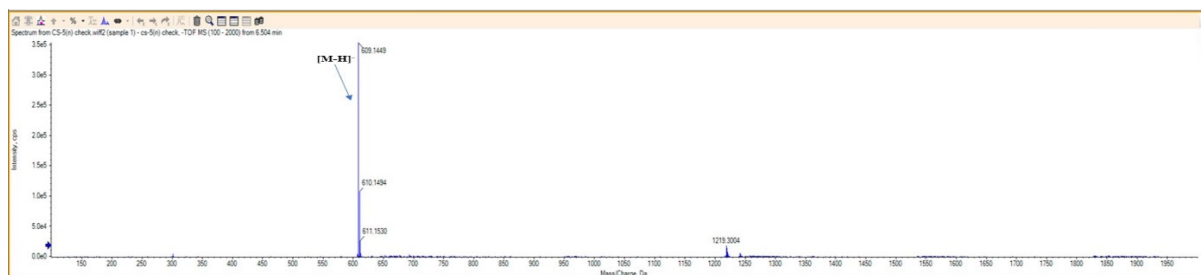


Figure S14. HR-ESI-MS spectrum of compound **4**

Rutin (**4**) : HR-ESI-MS (negative) m/z 609.1449 $[M-H]^-$, calcd for $C_{27}H_{29}O_{16}$ 609.1456; 1H NMR (500 MHz, CD_3OD): δ 7.67 (1H, d, $J = 1.9$ Hz, H-2'), 7.63 (1H, dd, $J = 8.4, 2.0$ Hz, H-6'), 6.87 (1H, d, $J = 8.4$ Hz, H-5'), 6.38 (1H, d, $J = 1.8$ Hz, H-8), 6.20 (1H, d, $J = 1.8$ Hz, H-6), 5.11 (1H, d, $J = 7.6$ Hz, H-1''), 4.52 (1H, d, $J = 1.0$ Hz, H-1'''), 3.81/3.39 (2H, m, H-5''), 3.64 (1H, m, H-2'''), 3.55 (1H, m, H-3'''), 3.47 (1H, m, H-2''), 3.45 (1H, m, H-5'''), 3.44 (1H, m, H-3''), 3.33 (1H, m, H-5''), 3.30 (1H, m, H-4'''), 3.29 (1H, m, H-4''), 1.12 (3H, d, $J = 6.2$ Hz, H-6'''); ^{13}C NMR (125 MHz, CD_3OD): δ 179.5 (C-4), 166.1 (C-7), 163.0 (C-5), 159.4 (C-9), 158.6 (C-2), 149.9 (C-4'), 145.9 (C-3'), 135.7 (C-3), 123.6 (C-6'), 123.2 (C-1'), 117.8 (C-2''), 116.1 (C-5''), 105.7 (C-10), 104.8 (C-1''), 102.5 (C-1'''), 100.0 (C-6), 94.9 (C-8), 78.2 (C-3''), 77.3 (C-5''), 75.8 (C-2''), 74.0 (C-4'''), 72.3 (H-3'''), 72.2 (H-2'''), 71.5 (H-4''), 69.8 (H-5'''), 68.6 (H-6''), 18.0 (H-6''').

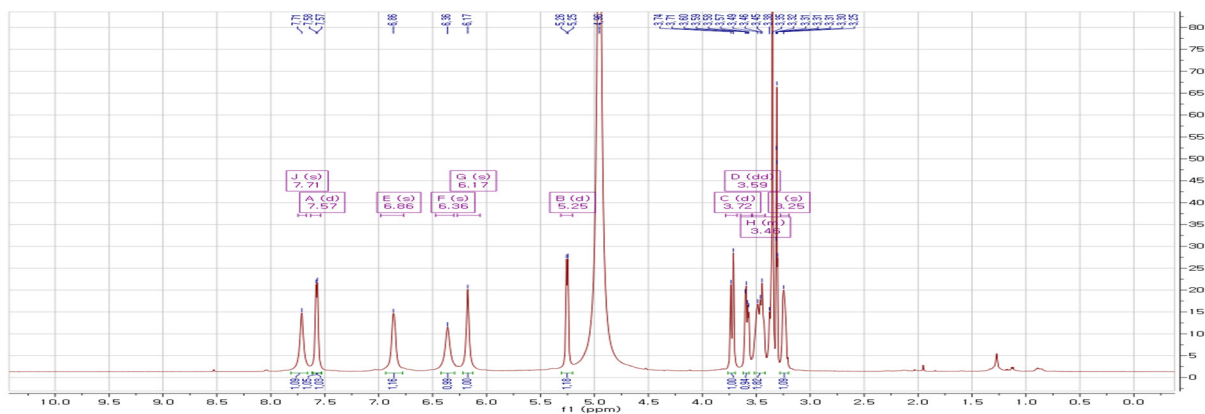


Figure S15. ^1H NMR spectrum of compound **5**

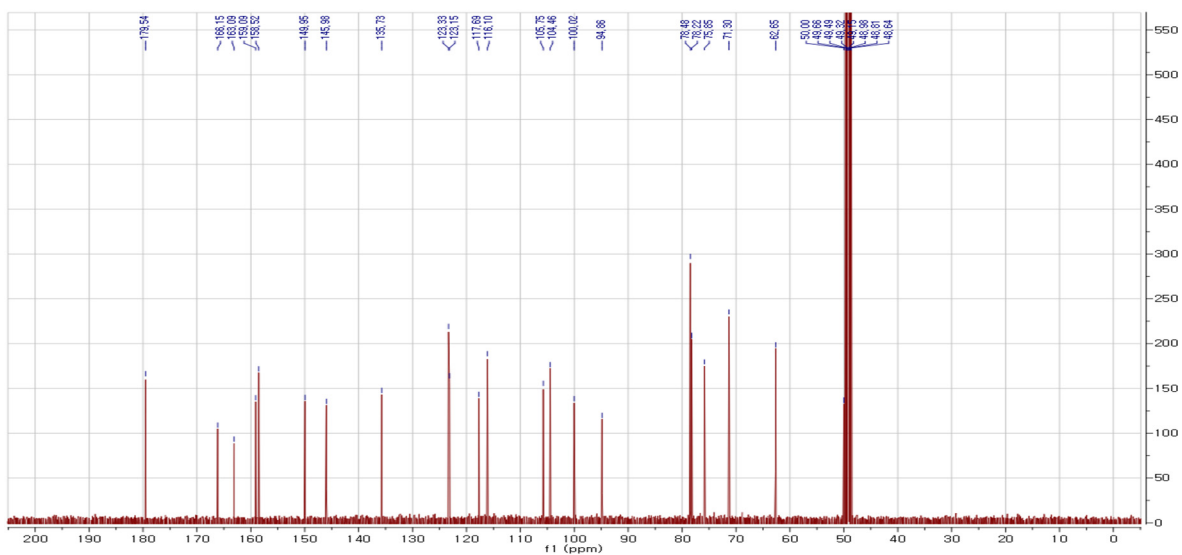
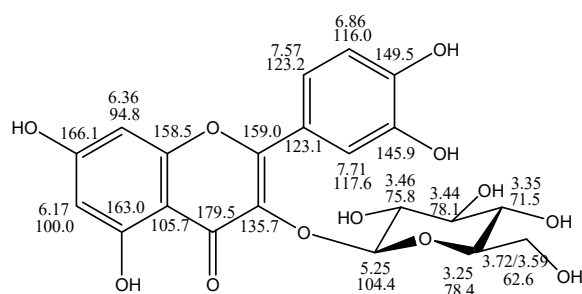


Figure S16. ^{13}C NMR spectrum of compound **5**



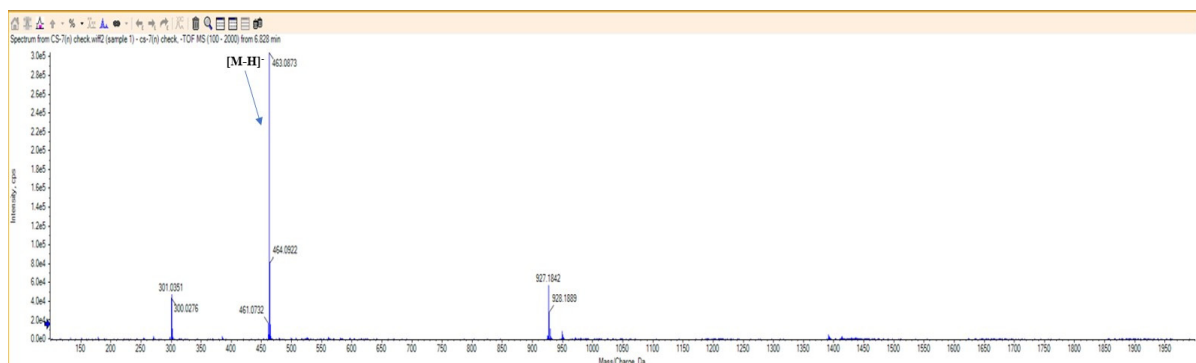


Figure S17. HR-ESI-MS spectrum of compound **5**

Isoquercetin (**5**) : HR-ESI-MS (negative) m/z 463.0873 [M-H]⁻, calcd for C₂₁H₁₉O₁₂ 463.0877; ¹H NMR (500 MHz, CD₃OD): δ 7.71 (1H, brs, H-2'), 7.57 (1H, d, J = 7.3 Hz, H-6'), 6.86 (1H, d, J = 7.3 Hz, H-5'), 6.36 (1H, brs, H-8), 6.17 (1H, brs, H-6), 5.25 (1H, d, J = 6.6 Hz), 3.72/3.59 (2H, m, H-6''), 3.46 (1H, m, H-2''), 3.44 (1H, m, H-3''), 3.35 (1H, m, H-4''), 3.25 (1H, m, H-5''); ¹³C NMR (125 MHz, CD₃OD): δ 179.5 (C-4), 166.1 (C-7), 163.0 (C-5), 159.0 (C-2), 158.5 (C-9), 149.5 (C-4'), 145.9 (C-3'), 135.7 (C-3), 123.2 (C-6'), 123.1 (C-1'), 117.6 (C-2'), 116.0 (C-5'), 105.7 (C-10), 104.4 (C-1''), 100.0 (C-6), 94.8 (C-8), 78.4 (C-5''), 78.1 (C-3''), 75.8 (C-2''), 71.5 (C-4''), 62.6 (C-6'').

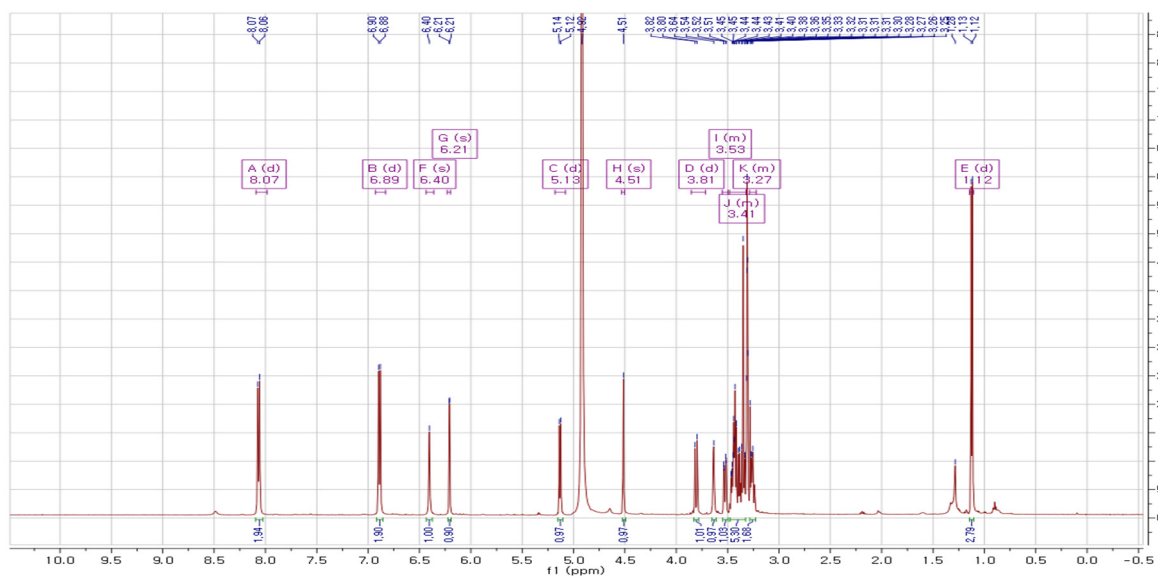


Figure S18. ^1H NMR spectrum of compound **6**

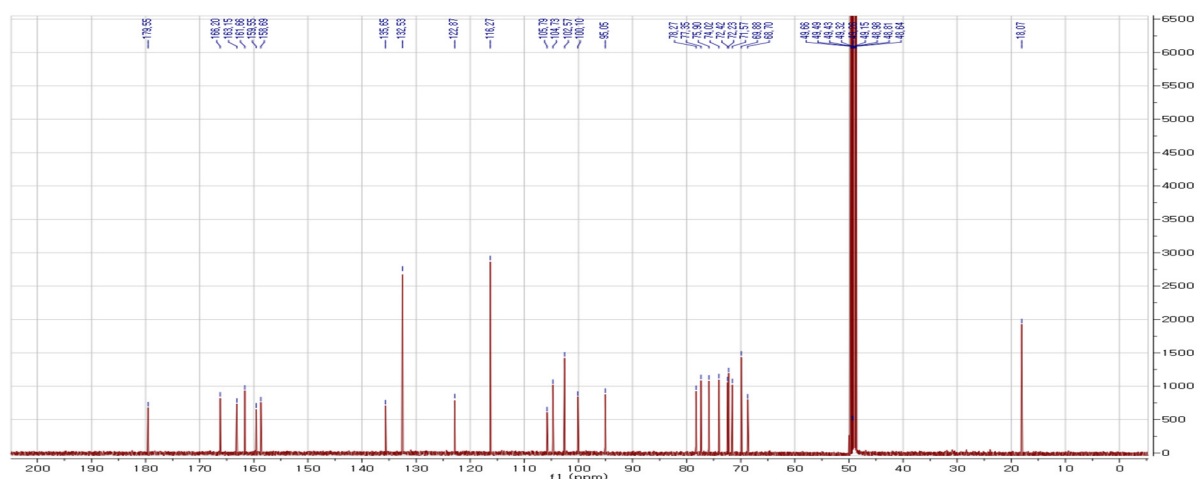
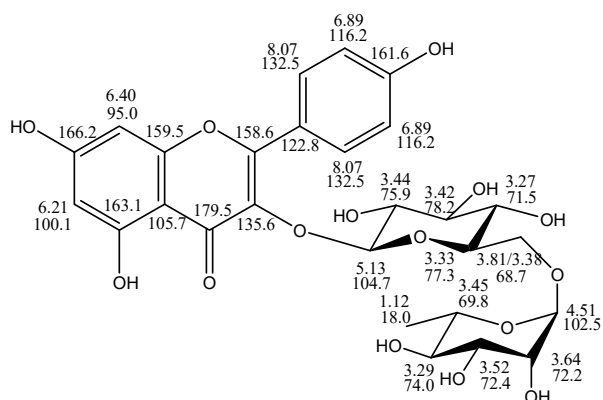


Figure S19. ^{13}C NMR spectrum of compound **6**



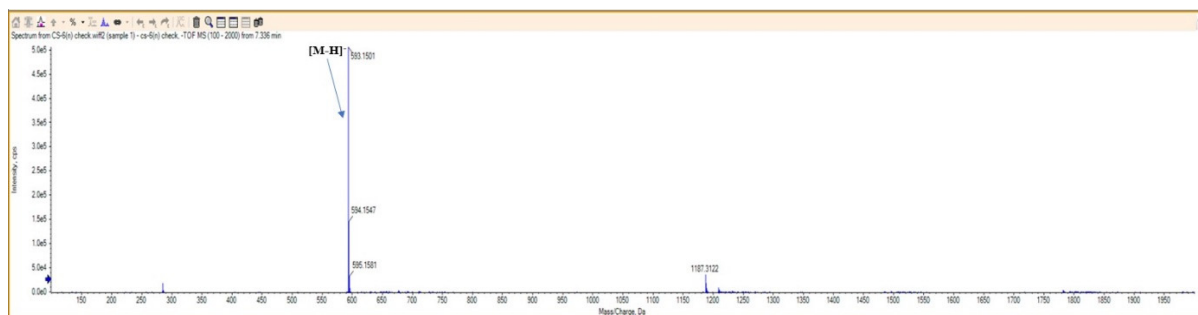
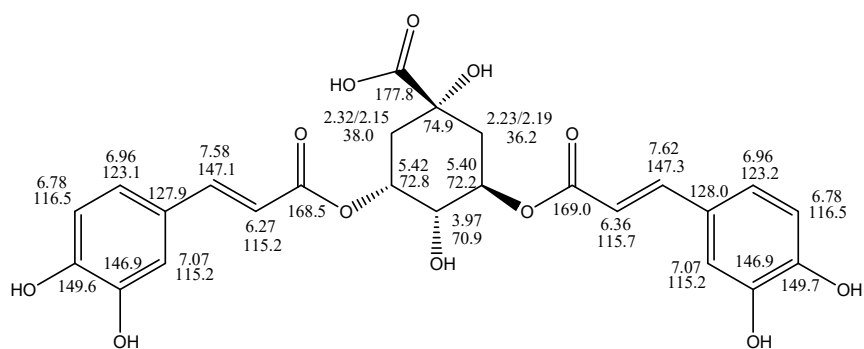
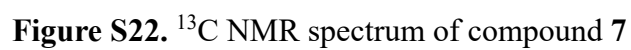
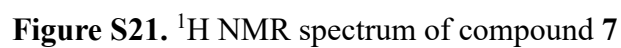


Figure S20. HR-ESI-MS spectrum of compound **6**

Nicotiflorin (**6**) : HR-ESI-MS (negative) m/z 593.1501 $[M-H]^-$, calcd for $C_{27}H_{29}O_{15}$ 593.1507; 1H NMR (500 MHz, CD_3OD): δ 8.07 (2H, d, $J = 8.7$ Hz, H-2' and H-6'), 6.89 (2H, d, $J = 8.7$ Hz, H-3' and H-5'), 6.40 (1H, brs, H-8), 6.21 (1H, brs, H-6), 5.13 (1H, d, $J = 7.4$ Hz, H-1''), 4.51 (1H, brs, H-1'''), 3.81/3.38 (2H, m, H-6''), 3.64 (1H, m, H-2'''), 3.52 (1H, m, H-3'''), 3.45 (1H, m, H-5'''), 3.44 (1H, m, H-2''), 3.42 (1H, m, H-3''), 3.33 (1H, m, H-5'') 3.29 (1H, m, H-4'''), 3.27 (1H, m, H-4''), 1.12 (3H, d, $J = 6.2$ Hz); ^{13}C NMR (125 MHz, CD_3OD): δ 179.5 (C-4), 166.2 (C-7), 163.1 (C-5), 161.6 (C-4'), 159.5 (C-9), 158.6 (C-2), 135.6 (C-3), 132.5 (C-2' and C-6'), 122.8 (C-1'), 116.2 (C-3' and C-5'), 105.7 (C-10), 104.7 (C-1''), 102.5 (C-1'''), 100.1 (C-6), 95.0 (C-8), 78.2 (C-3''), 77.3 (C-5''), 75.9 (C-2''), 74.0 (C-4'''), 72.4 (C-3'''), 72.2 (C-2'''), 71.5 (C-4''), 69.8 (C-5'''), 68.7 (C-6''), 18.0 (C-6''').



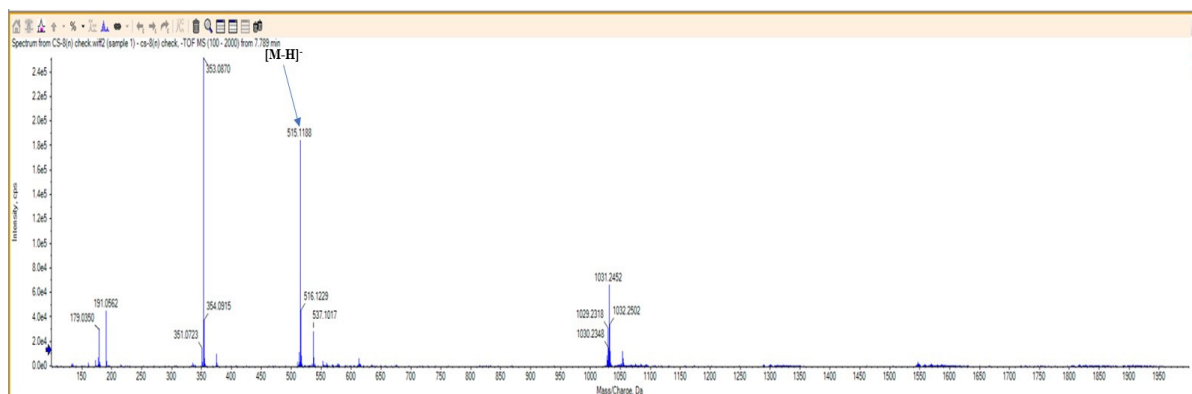
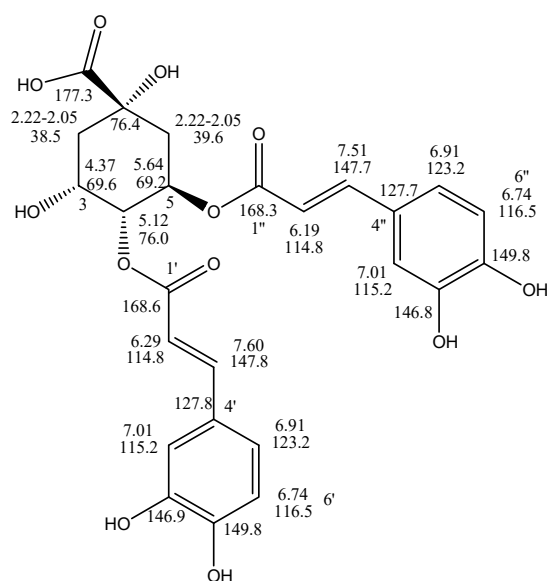
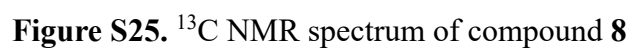
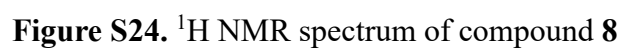


Figure S23. HR-ESI-MS spectrum of compound **7**

Isochlorogenic acid A (**7**) : HR-ESI-MS (negative) m/z 515.1188 $[M-H]^-$, calcd for $C_{25}H_{23}O_{12}$ 515.1190; 1H NMR (500 MHz, CD_3OD): δ 7.62 (1H, d, $J = 16.0$ Hz, H-3'), 7.58 (1H, d, $J = 16.0$ Hz, H-3''), 7.07 (2H, s, H-9' and H-9''), 6.96 (2H, m, H-5' and H-5''), 6.78 (2H, d, $J = 8.0$ Hz, H-6' and H-6''), 6.36 (1H, d, $J = 16.0$ Hz, H-2'), 6.27 (1H, d, $J = 16.0$ Hz, H-2''), 5.42 (1H, m, H-3), 5.40 (1H, m, H-5), 3.97 (1H, m, H-4), 2.32/2.15 (2H, m, H-2), 2.23/2.19 (2H, m, H-6); ^{13}C NMR (125 MHz, CD_3OD): δ 177.8 (C-7), 169.0 (C-1'), 168.5 (C-1''), 149.7 (C-7'), 149.6 (C-7''), 147.3 (C-3'), 147.1 (C-3''), 146.9 (C-8' and C-8''), 128.0 (C-4'), 127.9 (C-4''), 123.2 (C-5'), 123.1 (C-5''), 116.5 (C-6' and C-6''), 115.7 (C-2'), 115.3 (C-2''), 115.2 (C-9' and C-9''), 74.9 (C-1), 72.8 (C-3), 72.2 (C-5), 70.9 (C-4), 38.0 (C-2), 36.2 (C-6).



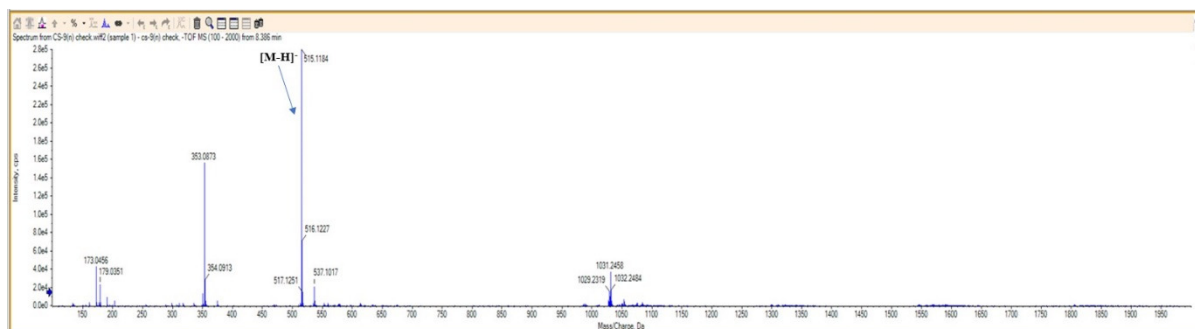


Figure S26. HR-ESI-MS spectrum of compound **8**

Isochlorogenic acid C (**8**) : HR-ESI-MS (negative) m/z 515.1184 $[M-H]^-$, calcd for $C_{25}H_{23}O_{12}$ 515.1190; 1H NMR (500 MHz, CD_3OD): δ 7.60 (1H, d, $J = 15.8$ Hz, H-3'), 7.51 (1H, d, $J = 15.8$ Hz, H-3''), 7.01 (2H, d, $J = 1.8$ Hz, H-9' and H-9''), 6.91 (2H, brt, H-5' and H-5''), 6.74 (2H, d, $J = 1.8$ Hz, H-6' and H-6''), 6.29 (1H, d, $J = 15.8$ Hz, H-2'), 6.19 (1H, d, $J = 15.8$ Hz, H-2''), 5.62 (1H, m, H-5), 5.12 (1H, dd, $J = 9.0, 2.8$ Hz, H-4), 4.37 (1H, m, H-3), 2.22-2.05 (4H, m, H-2 and H-6); ^{13}C NMR (125 MHz, CD_3OD): δ 177.3 (C-7), 168.6 (C-1'), 168.3 (C-1''), 149.8 (C-7' and C-7''), 147.8 (C-3'), 147.7 (C-3''), 146.9 (C-8' and C-8''), 127.8 (C-4'), 127.7 (C-4''), 123.2 (C-5' and C-5''), 116.5 (C-6' and C-6''), 115.2 (C-9' and C-9''), 114.8 (C-2' and C-2''), 76.4 (C-1), 76.0 (C-4), 69.6 (C-3), 69.2 (C-5), 39.6 (C-6), 38.5 (C-2).