Supplementary Information

One-Step Isolation of Monoterpene Indole Alkaloids from *Psychotria leiocarpa* Leaves and Their Antiviral Activity on Dengue Virus Type-2

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Isolation of *N*,β-D-glucopyranosyl vincosamide (**1**) used as reference compound

338 mg of the MeOH extract from *Psychotria leiocarpa* leaves were suspended in 50 mL of HCl 0.1 M, kept by 5 min in an ultrasound bath and then partitioned with EtOAc (3 × 50 mL). The aqueous fraction was treated with NH_4OH conc. (pH 9-10), partitioned with EtOAc (4 × 50 mL), the remaining aqueous fraction was neutralized with HCl conc. and partitioned with BuOH (3 × 50 mL). The BuOH fraction (93.3 mg) was submitted to a Sephadex LH-20 CC (phase height 6.0 cm, external diameter 2 cm) using the solvent systems: hexane/CH_2Cl_2 4:1, CH_2Cl_2/acetone 4:1, acetone/MeOH 1:1 and MeOH as mobile phases. The sub-fraction 5 (58.6 mg) eluted with acetone/MeOH 1:1 revealed by thin layer chromatography (TLC) three major spots. It was sequentially submitted to silica gel C18 (40-63 μm) CC (phase height 17.0 cm, e.d. 2 cm) using a gradient solvent system from MeOH/H_2O 3:7 to $CHCl_3$ as mobile phase. Compound **1** was isolated from the sub-fraction 5.7 (10.1 mg) eluted with MeOH/H_2O 1:1. Its chemical structure was characterized on the basis of 1D and 2D NMR, UV and comparison to literature data.

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Table S1. Amounts of the combined fractions yielded from the semi-prep SPE experiments from the bioactive extract of *Psychotria leiocarpa*

SPE combined fraction	Amount / mg
E1.1-E1.2	314.6
E1.3	5.0
E2.1	8.6
E2.2-E2.3 + E3.1	56.5
E3.2-E3.3	40.7
E4	7.2
E5	7.9
E6	18.4
E7	81.6

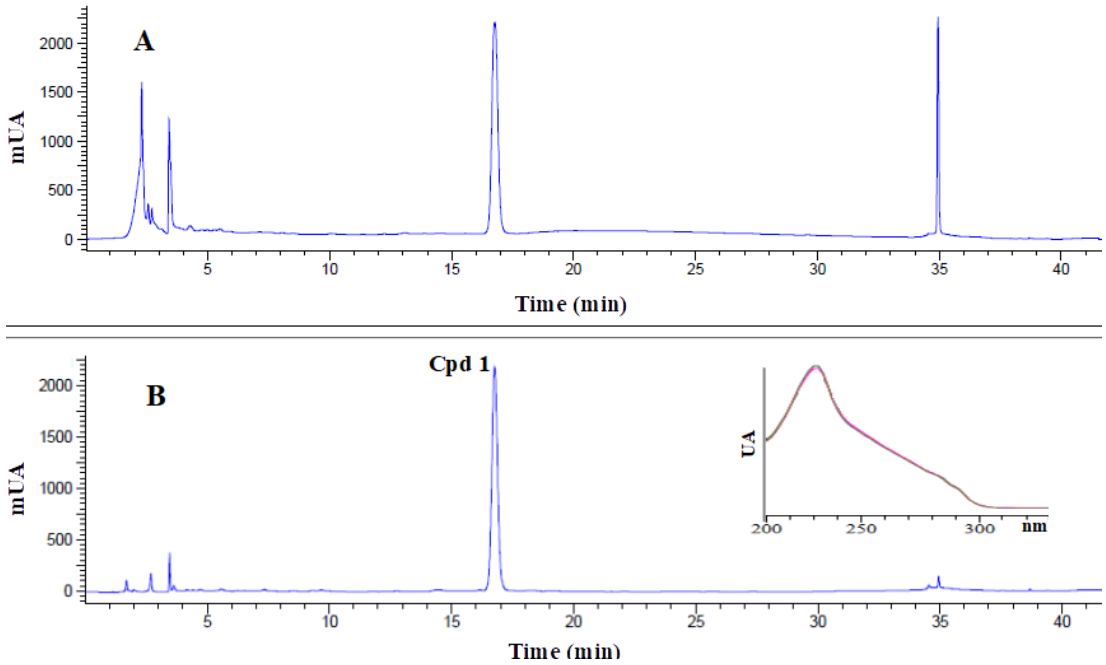

Figure S1. HPLC-DAD profiles at 225 nm of: (a) defatted MeOH extract from *Psychotria leiocarpa* leaves; (b) *N*,β-D-glycopyranosylvincosamide (**1**) (t_R = 16.8 min) and its UV spectrum. Lichrocart Lichrospher RP-18 (250 × 4.6 mm; 5 µm) column coupled to a precolumn SupelguardTM LC-18 (20 × 4.0 mm, 5 µm); mobile phase: ultrapurified water, adjusted to pH 3 with HCOOH (A) and ACN (B): 10-20% B, 0-5 min; 20-22% B, 5-10 min; 22-24% B, 10-15 min; 24-26% B, 15-20 min; 26-28% B, 20-25 min; 28-30% B, 25-30 min, and finally, 30-100% B, 30-35 min. Flow rate: 0.8 mL min^{-1}; temperature: 40 °C; c_A = 20 mg mL^{-1}; c_B = 2 mg mL^{-1}; injection volume = 10 µL.

Data of the isolated alkaloids

N-Glucopyranosyl vincosamide (**1**)

UV λ_{MAX} / nm (log ε) 225 (4.36); HR-ESI-TOF-MS *m/z*, calcd. for $C_{32}H_{41}N_2O_{13}$ $[M + H]^+$: 661.2609, found: 661.2943; ^{1}H NMR (500 MHz, CD_3OD) δ 5.05 (1H, m, H-3), 2.78 (1H, br t, *J* 12.5, 3.4 Hz, H-5a), 4.99 (1H, br dd, *J* 12.4, 3.3 Hz, H-5b), 2.59 (1H, m, H-6a), 2.76 (1H, m, H6-b), 7.38 (1H, d, *J* 7.7 Hz, H-9), 7.00 (1H, br t, *J* 7.4 Hz, H-10), 7.05 (1H, br t, *J* 7.2 Hz, H-11), 7.56 (1H, d, *J* 8.1 Hz, H-12), 1.31 (1H, br dd, *J* 13.5, 11.5 Hz, H-14a), 2.27 (1H, br d, *J* 13.0 Hz, H-14b), 3.24 (1H, m, H-15), 7.41 (1H, d, *J* 2.3 Hz, H-17), 5.18 (1H, dd, *J* 10.5, 1.5 Hz, H-18a), 5.21 (1H, dd, *J* 17.1, 1.4 Hz, H-18b), 5.41 (1H, m, H-19), 2.66 (1H, m, H-20), 5.46 (1H, d, *J* 1.5 Hz, H-21), 4.65 (1H, d, *J* 7.9 Hz, H-1'), 3.17 (1H, d, *J* 8.0 Hz, H-2'), 3.33 (1H, m, H-3'), 3.56 (1H, m, H-4'), 3.26 (1H, m, H-5'), 3.61 (1H, dd, *J* 12.0, 5.5 Hz, H-6'a), 3.89 (1H, dd, *J* 11.9, 1.6 Hz, H-6'b), 5.03 (1H, m, H-1"), 4.04 (1H, m, H-2"), 3.47 (1H, m, H-3"), 3.54 (2H, m, H-4''and H-5''), 3.72 (1H, dd, *J* 12.2, 5.9 Hz, H-6"a), 3.90 (1H, m, H-6"b); ^{13}C NMR (125 MHz, CD_3OD) δ 136.1 (C-2), 54.5 (C-3), 40.7 (C-5), 22.3 (C-6), 111.5 (C-7), 129.5 (C-8), 119.3 (C-9), 121.3 (C-10), 122.9 (C-11), 114.3 (C-12), 137.7 (C-13), 34.6 (C-14), 27.9 (C-15), 109.1 (C-16), 149.2 (C-17), 120.7 (C-18), 133.3 (C-19), 44.3 (C-20), 97.5 (C-21), 166.2 (C-22), 99.6 (C-1'), 74.7 (C-2'), 77.9 (C-3'), 71.5 (C-4'), 78.5 (C-5'), 62.7 (C-6'), 87.5 (C-1"), 71.9 (C-2"), 79.1 (C-3"), 71.6 (C-4"), 81.2 (C-5"), 62.9 (C-6").

Vincosamide (**2**)

UV λ_{MAX} / nm (log ε) 230 (4.45); HR-ESI-TOF-MS *m/z*, calcd. for $C_{26}H_{31}N_2O_8$ $[M + H]^+$: 499.2080, found: 499.2083; ^{1}H NMR (500 MHz, CD_3OD) δ 4.97 (1H, d, *J* 11.2 Hz, H-3), 2.98 (1H, td, *J* 12.5, 4.5 Hz, H-5a), 5.11 (1H, m, H-5b), 2.80 (2H, m, H-6), 7.46 (1H, br d, *J* 7.8 Hz, H-9), 7.04 (1H, br t, 7.8 Hz, H-10), 7.12 (1H, br t, *J* 8.1 Hz, H-11), 7.35 (1H, d, *J* 8.1 Hz, H-12), 1.50 (1H, dd, *J* 13.2, 11.8 Hz, H-14a), 2.51 (1H, dt, *J* 12.9, 3.8 Hz, H-14b), 3.28 (1H, m, H-15), 7.50 (1H, d, *J* 2.4 Hz, H-17), 5.23 (1H, dd, *J* 10.3, 1.9 Hz, H-18a), 5.34 (1H, dd, 17.1, 1.8 Hz, H-18b), 5.58 (1H, m, H-19), 2.82 (1H, m, H-20), 5.55 (1H, d, *J* 1.8 Hz, H-21), 4.75 (1H, d, *J* 7.9 Hz, H-1'), 3.27 (1H, m, H-2'), 3.40 (2H, m, H-3' and H-5'), 3.30 (1H, m, H-4'), 3.73 (1H, dd, *J* 12.0, 5.6 Hz, H-6'a), 3.96 (1H, dd, *J* 10.0, 1.9 Hz, H-6'b); ^{13}C NMR (125 MHz, CD_3OD) δ 134.5 (C-2), 54.8 (C-3), 41.2 (C-5), 22.0 (C-6), 108.9 (C-7), 127.7 (C-8), 118.9 (C-9), 119.9 (C-10), 122.4 (C-11), 111.9 (C-12), 138.0 (C-13), 32.6 (C-14), 27.3 (C-15), 109.1 (C-16), 148.9 (C-17), 120.5 (C-18), 133.9 (C-19), 44.5 (C-20), 97.3 (C-21), 166.0 (C-22), 99,6 (C-1'), 74.8 (C-2'), 78.3 (C-3'), 71.4 (C-4'), 77.9 (C-5'), 62.5 (C-6').

Strictosidinic acid (**3**)

UV λ_{MAX} / nm (log ε) 221 (4.35); HR-ESI-TOF-MS *m/z*, calcd. for $C_{26}H_{33}N_2O_9$ $[M + H]^+$: 517.2193, found: 517.2186; ^{1}H NMR (500 MHz, CD_3OD) δ 4.54 (1H, bs, H-3), 3.39 (2H, m, H-5), 2.93 (2H, m, H-6), 7.37 (1H, d, *J* 7.7 Hz, H-9), 7.05 (1H, d, *J* 7.5 Hz, H-9), 6.95 (1H, t, *J* 7.6 Hz, H-11), 7.28 (1H, d, *J* 8.1 Hz, H-12), 1.89 (1H, m, H-14a), 2.38 (1H, m, H-14b), 2.86 (1H, m, H-15), 7.20 (1H, br s, H-17), 5.30 (1H, d, *J* 10.3 Hz, H-18a), 5.32 (1H, d, *J* 16.9 Hz, H-18b), 6.01 (1H, ddd, *J* 17.2, 10.3, 8.4 Hz, H-19), 2.66 (1H, m, H-20), 5.46 (1H, d, *J* 7.9 Hz, H-21), 4.68 (1H, d, *J* 7.9 Hz, H-1'), 3.16 (1H, d, *J* 8.0 Hz, H-2'), 3.30 (1H, m, H-3'), 3.24 (1H, m, H-4'), 3.33 (1H, m, H-5'), 3.58 (1H, m, H-6'a), 3.86 (1H, dd, *J* 11.8, 2.0 Hz, H-6'b); ^{13}C NMR (125 MHz, CD_3OD) δ 53.4 (C-3), 39.5 (C-5), 18.0 (C-6), 106.0 (C-7), 126.0 (C-8), 117.5 (C-9), 121.9 (C-10), 119.0 (C-11), 110.8 (C-12), 136.9 (C-13), 33.9 (C-14), 32.9 (C-15), 114.8 (C-16), 149.0 (C-17), 118.6 (C-18), 134.5 (C-19), 44.7 (C-20), 95.6 (C-21), 173.3 (C-22), 98.7 (C-1'), 73.3 (C-2'), 76.6 (C-3'), 70.3 (C-4'), 77.1 (C-5'), 61.3 (C-6').

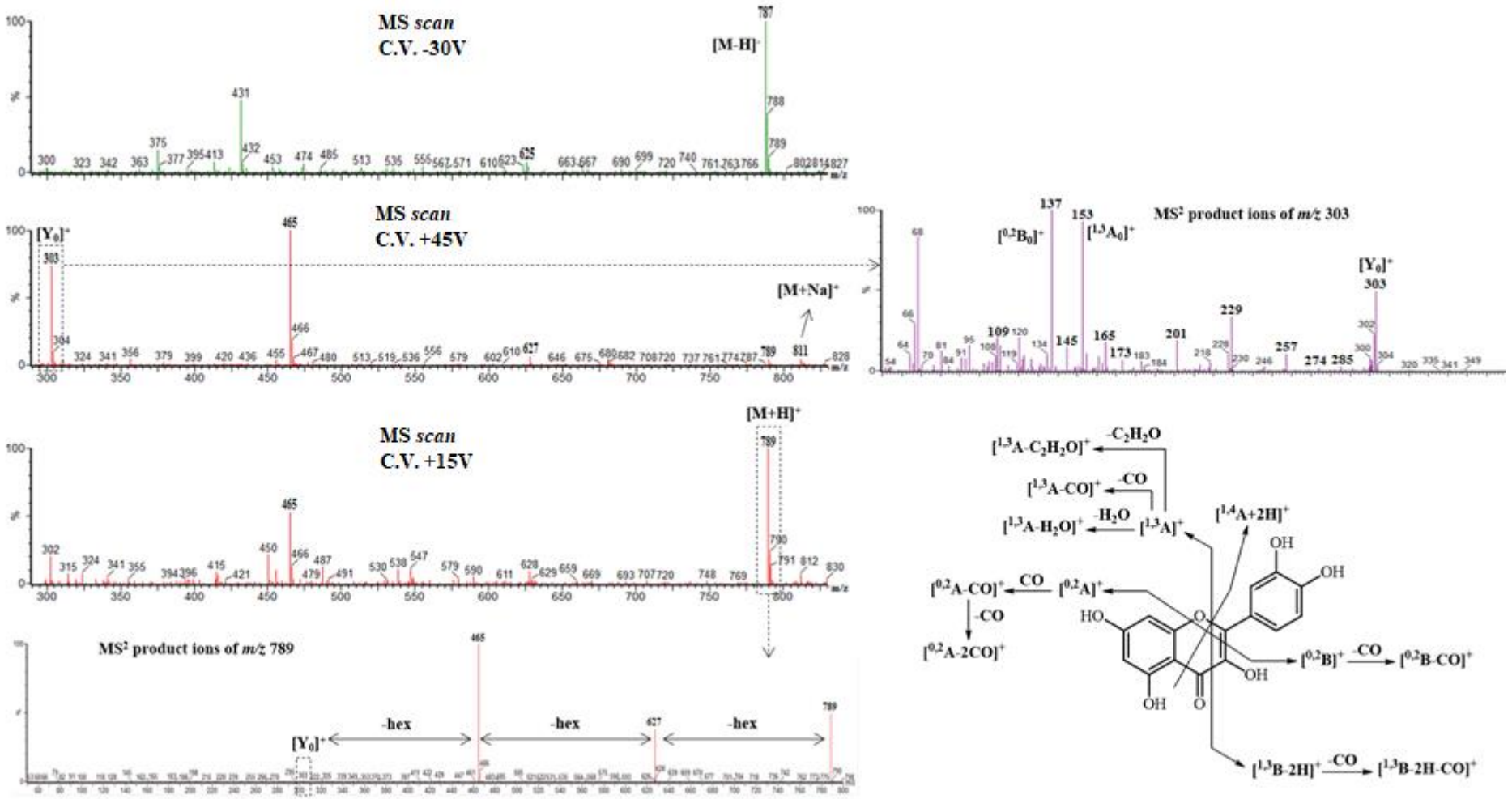

Figure S2. MS^2 ions from $[Y_0]^+$ of the polyphenol chromatographic peak with t_R = 20.15 min (UV λ_{MAX} = 253, 267, 353 nm). C.V.: cone voltage.

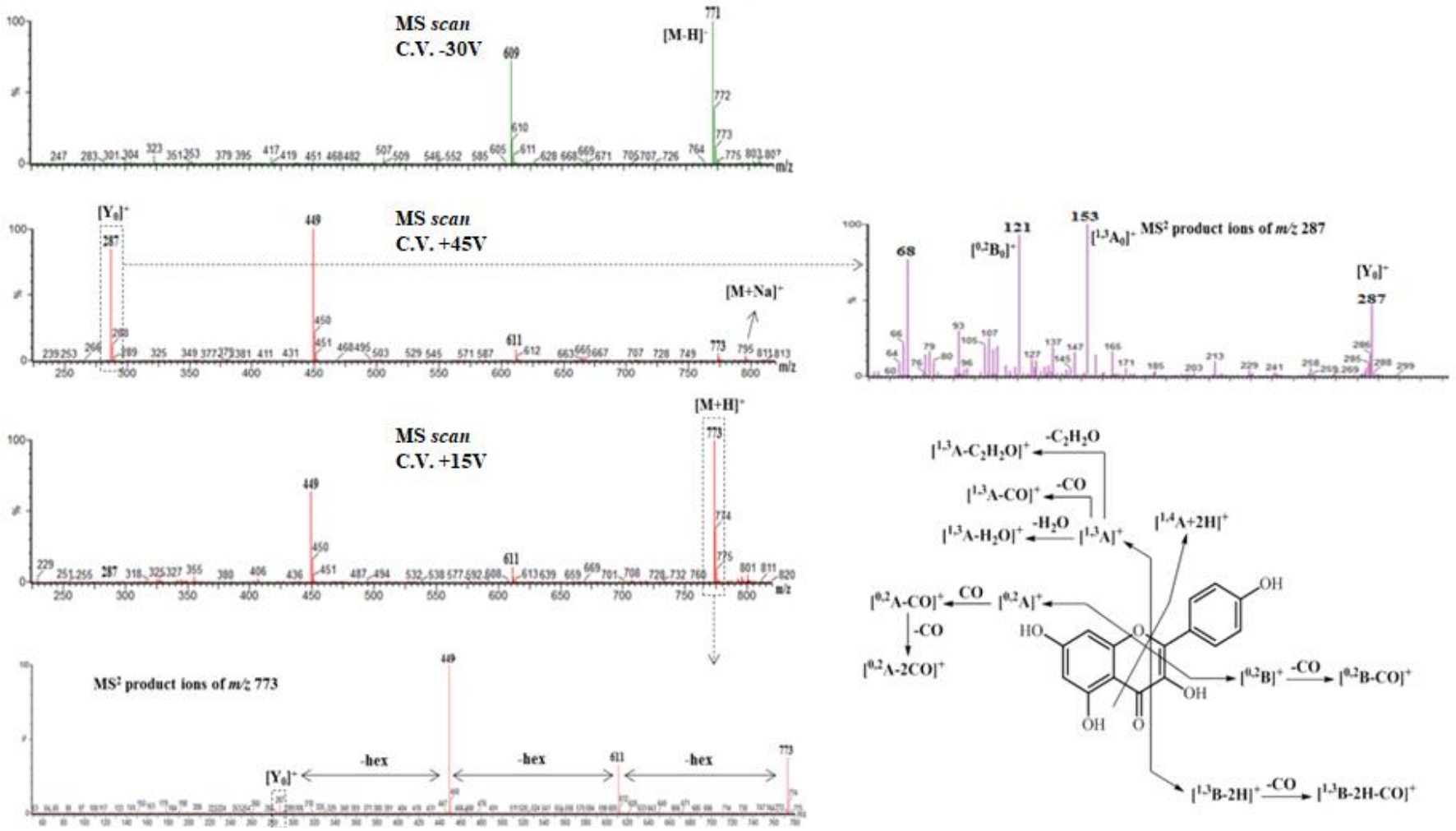

Figure S3. MS^2 ions from $[Y_0]^+$ of the polyphenol chromatographic peak with t_R = 22.97 min, (UV λ_{MAX} = 265, 346 nm). C.V.: cone voltage.