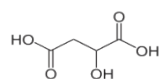
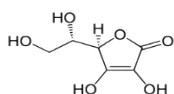


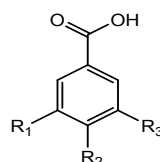
Supplementary Materials



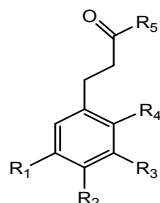
Malic acid (1)



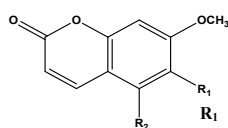
Ascorbic acid (4)



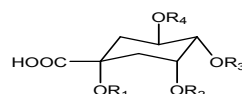
R ₁	R ₂	R ₃	Compound
OH	H	OH	3, 5-Dihydroxybenzoic acid (3)
H	OH	H	<i>p</i> -Hydroxybenzoic acid (8)
OMe	OH	H	Vanillic acid (5, 9)
OMe	OH	OMe	Syringic acid (13)



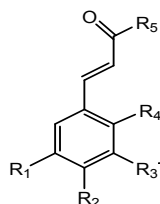
R ₁	R ₂	R ₃	R ₄	R ₅	Compound
OMe	OH	OMe	H	OH	Dihydrosinapic acid (6)
OMe	OH	OMe	H	O-glc	Dihydrosinapoyl- <i>O</i> -glucoside (25)
OMe	OH	H	H	O-glc-HMG	Dihydroferulic acid-HMG-glucoside (26)



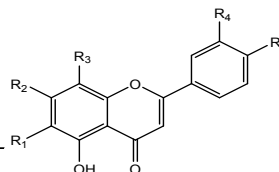
R ₁	R ₂	Compound
H		5-Geranyloxy-7-methoxy coumarin (2)
	H	Citribuntin (14)



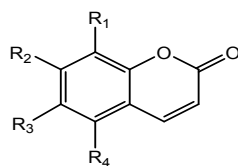
R ₁	R ₂	R ₃	R ₄	Compound
H	H	H	F	5- <i>O</i> -Feruloylquinic acid (7)
H	F	H	H	3- <i>O</i> -Feruloylquinic acid (10)
H	H	F	H	4- <i>O</i> -Feruloylquinic acid (12)



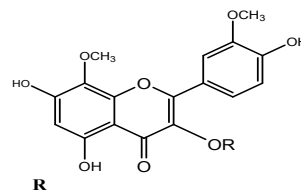
R ₁	R ₂	R ₃	R ₄	R ₅	Compound
OMe	OH	H	H	OH	Ferulic acid (19)
H	H	H	OH	O-Quin.	5- <i>p</i> -Coumaroyl quinic acid (11)
OH	OMe	H	H	OH	Isoferulic acid (20)
H	O-glc	H	H	OH	<i>p</i> -Coumaroyl-6'-glc Ester (21)
OMe	OH	OMe	H	OH	Sinapic acid (22)
OMe	O-glc	H	H	OH	Feruloyl-6'-glc Ester (28)



R	Compound
O-glc-HMG	Limocitrol- <i>O</i> -glucoside- HMG (42)



R ₁	R ₂	R ₃	R ₄	Compound
	OMe	H	H	Meranzine hydrate (17)



R	Compound
O-glc-HMG	Limocitrin - <i>O</i> -glucoside- HMG (38)
O-glc-HMG-HMG-Caf.	Limocitrin - <i>O</i> -glucoside-HMG-HMG-Caf. (69)

Fig. S1A Metabolites identified in Murcott dichloromethane (DCM) and ethyl acetate (ET) fractions using HPLC-PDA-ESI- MS in negative ion mode.

glc= Glucose; Rut. = Rutinosyl; Neoh. = Neohesperidosyl; Caf. = Caffeoyl; HMG= 3- Hydroxy-3-methyl glutaryl; F= Feruloyl; Quin. = Quinoyl.

	R1	R2	R3	R4	R5	R6	Compound
	H	(2'''-O-Xylosyl) Glc.	OH	H	H	OH	Apigenin-6-C-glucosyl-2'''-O-xyloside (33)
	H	H	OH	(2'''-O-Xylosyl) glc	H	OH	Apigenin-8-C-glucosyl-2'''-O-xyloside (35)
	H	H	OH	H	OH	OMe	Diosmetin (75)
	H	H	O-Rut.	H	OH	OMe	Diosmin (53)
	O-Rut.	H	OH	H	OH	OH	Rutin (62)
	OMe	H	OMe	H	H	OMe	5-Hydroxy- 3,4', 7-trimethoxy flavone (70)
	O-HMG-glc	H	OH	OMe	OH	OH	8-Methoxyquercetin-3-O-[6''-HMG]-glucoside (27)
	O- glc	H	OH	OMe	OH	OH	8-Methoxyquercetin-3-O-glucoside (56)
	O- Rut.	H	O-glc	H	OH	OH	Quercetin-3-O-rutinosyl-7-O- glucoside (68)
	O-Rut.	H	OH	H	OMe	OH	Isorhamnetin-3-O-rutinoside (64)
	OMe	H	OH	H	OMe	OH	Quercetin dimethyl ether (74)
	OMe	OMe	OH	H	OMe	OH	Jaceidin (73)
	R1	R2	R3	R4	R5	Compound	
	glc	OH	glc	OMe	OH	Stellarin-2 (30)	
	glc	OH	glc	OH	OMe	Lucenin-2 4'-methyl ether (31)	
	glc	OH	glc	H	OH	Vicenin-2 (32)	
	glc	OH	H	OH	OH	Isoorientin (34)	
	H	OH	glc	OH	OH	Orientin (36)	
	H	OH	glc	OMe	OH	Scoparin (37)	
H	OH	glc	H	OH	Vitexin (43)		
glc	OH	H	H	OH	Isovitexin (44)		
H	OH	glc	OH	OMe	Orientin-4'-methyl ether (45)		
glc	OH	H	OMe	OH	Isoscoparin (49)		
H	O-Rut.	H	OH	OH	Luteolin-7-O-rutnoside (50)		
H	O-glc	H	OMe	OH	Chrysoeriol-7-O-glucoside (63)		
glc	OH	H	OH	OMe	Isoorientin-4'-methyl ether (65)		
H	OH	H	OMe	OH	Chrysoeriol (72)		
H	O-Neoh.	H	OH	OH	Luteolin-7-O-neohesperidoside		
H	OH	H	OH	OH	Luteolin (89)		
	R1	R2	R3	R4	R5	Compound	
	glc	OH	glc	OMe	OH	Stellarin-2 (30)	
	glc	OH	glc	OH	OMe	Lucenin-2 4'-methyl ether (31)	
	glc	OH	glc	H	OH	Vicenin-2 (32)	
	glc	OH	H	OH	OH	Isoorientin (34)	
	H	OH	glc	OH	OH	Orientin (36)	
	H	OH	glc	OMe	OH	Scoparin (37)	
	H	OH	glc	H	OH	Vitexin (43)	
	glc	OH	H	H	OH	Isovitexin (44)	
	H	OH	glc	OH	OMe	Orientin-4'-methyl ether (45)	

Fig. S1B Metabolites identified in Murcott dichloromethane (DCM) and ethyl acetate (ET) fractions using HPLC-PDA-ESI- MS in negative ion mode.

glc= Glucose; Rut. = Rutinosyl; Neoh. = Neohesperidosyl; Caf. = Caffeoyl; HMG= 3- Hydroxy-3-methyl glutaryl; F= Feruloyl; Quin. = Quinoyl.

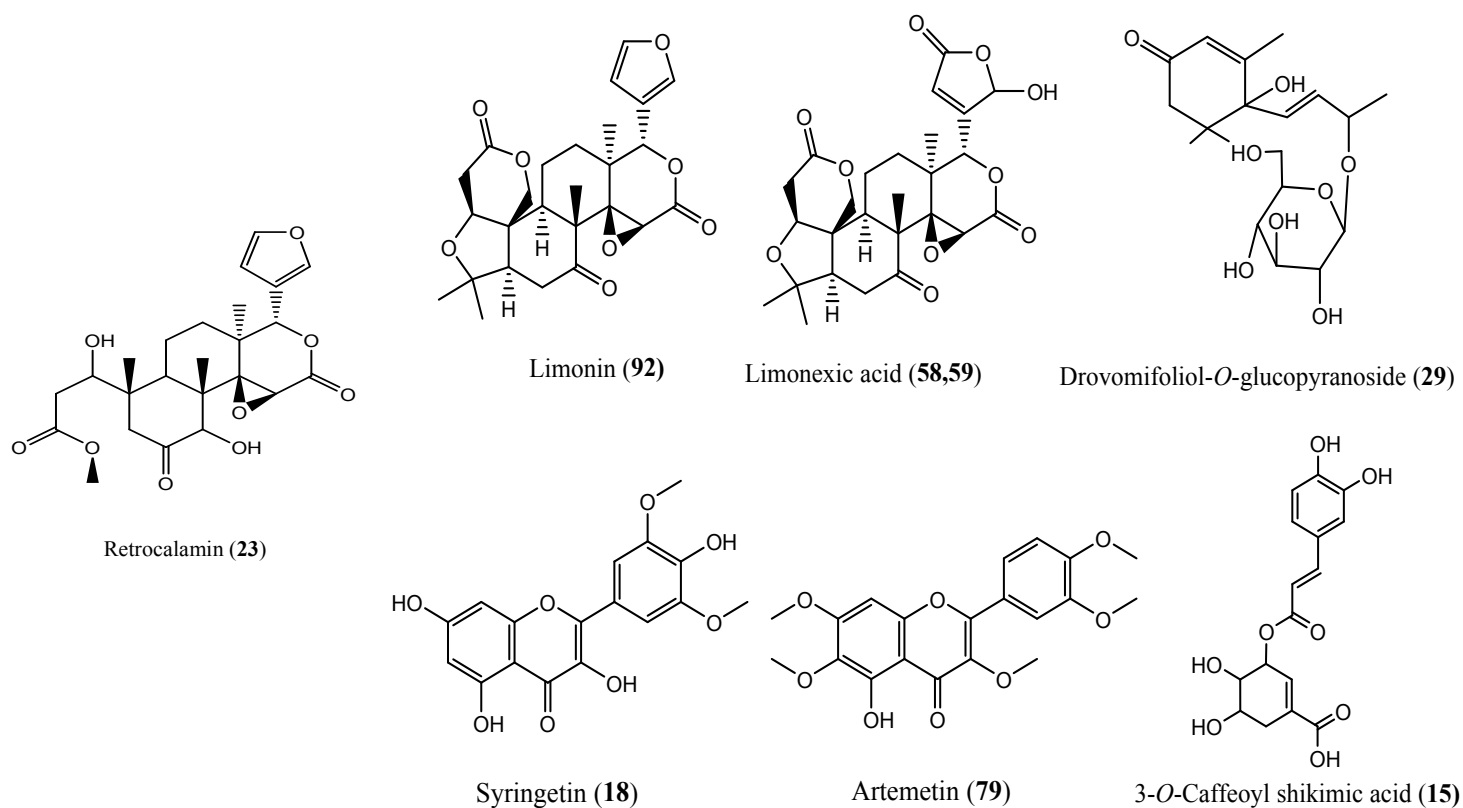


Fig. S1C Metabolites identified in Murcott dichloromethane (DCM) and ethyl acetate (ET) fractions using HPLC-PDA-ESI- MS in negative ion mode.

glc= Glucose; Rut. = Rutinosyl; Neoh. = Neohesperidosoyl; Caf. = Caffeyoyl; HMG= 3- Hydroxy-3-methyl glutaryl; F= Feruloyl; Quin. = Quinoyl.

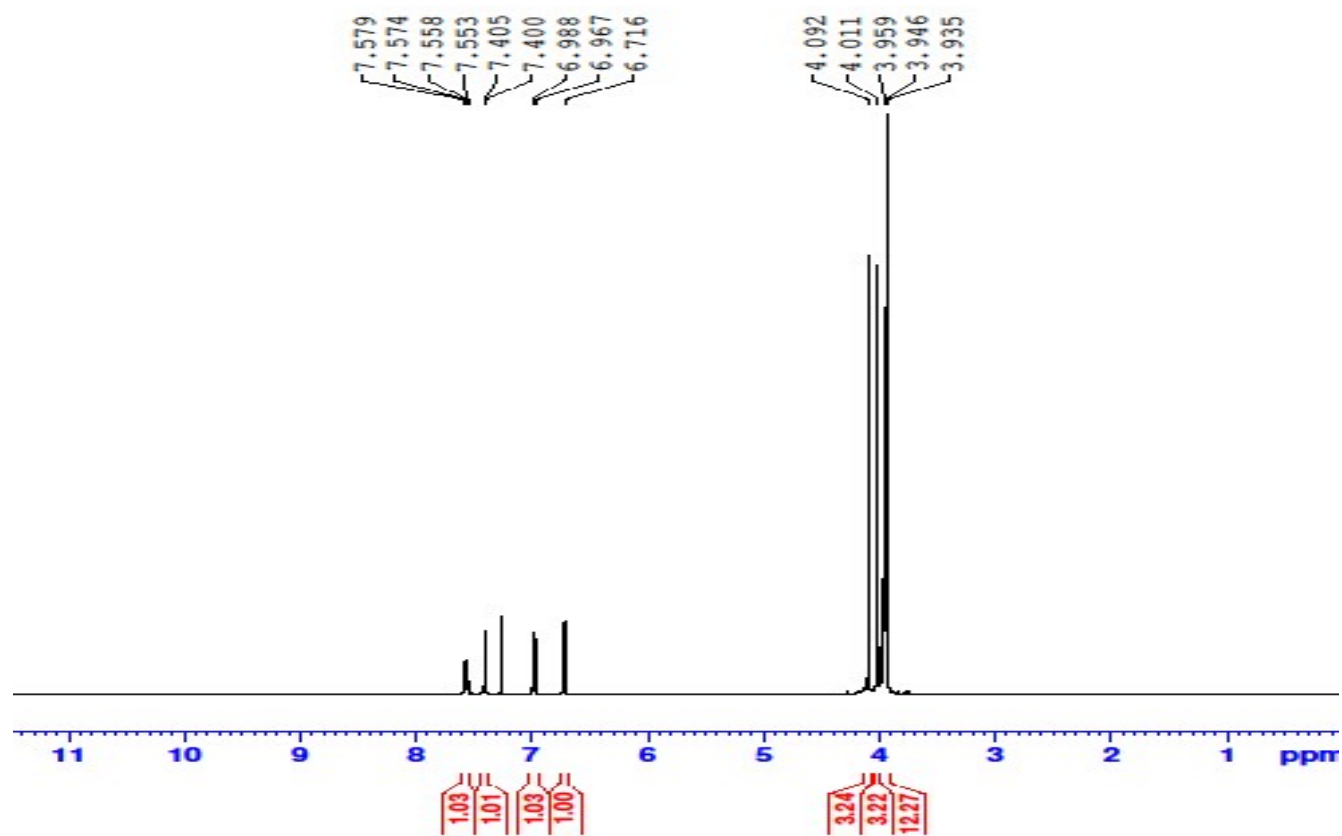


Fig. S2 ^1H -NMR spectrum of compound **C1** (Nobiletin)

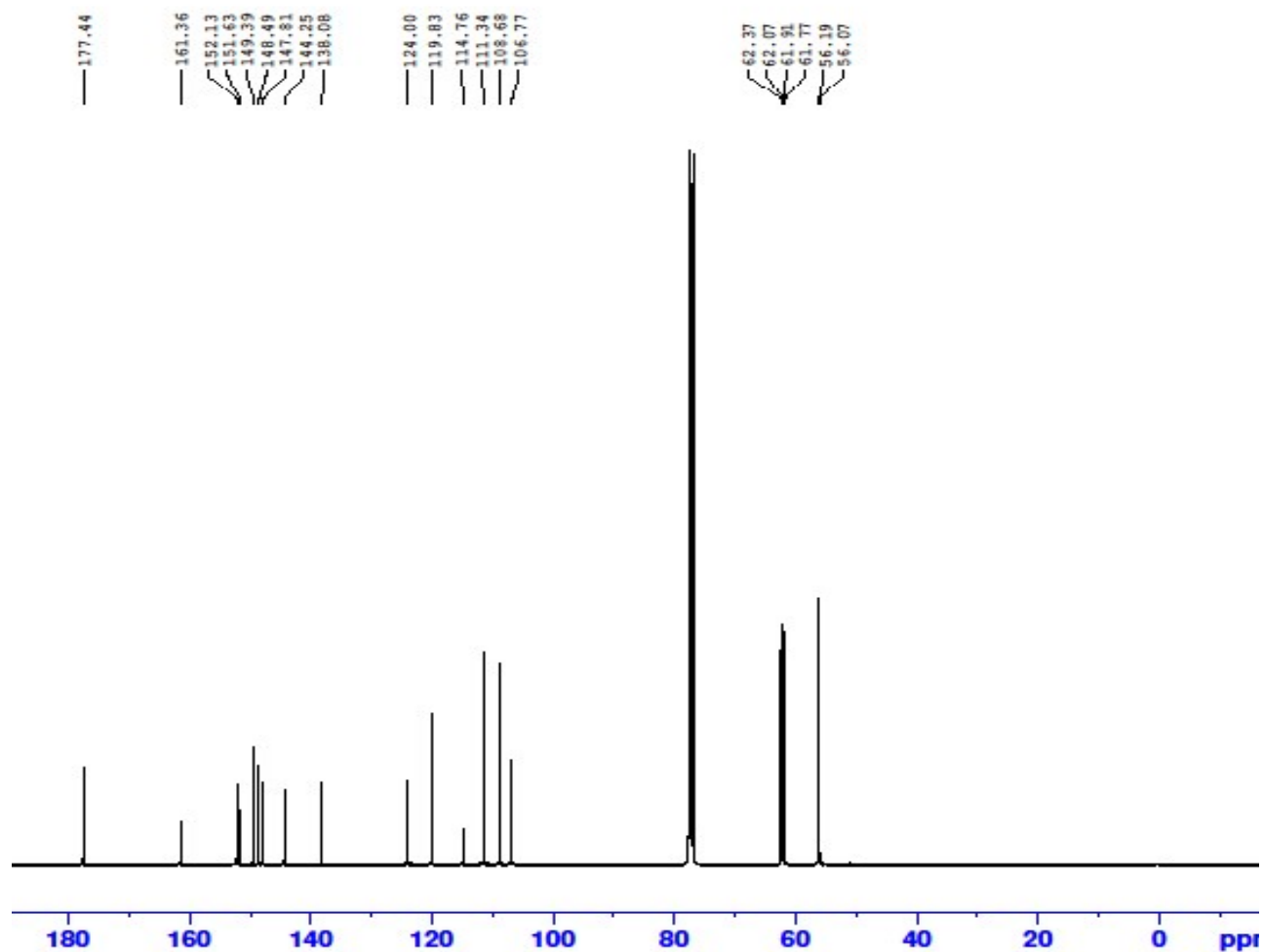


Fig. S3 ^{13}C -NMR spectrum of compound **C1** (Nobiletin)

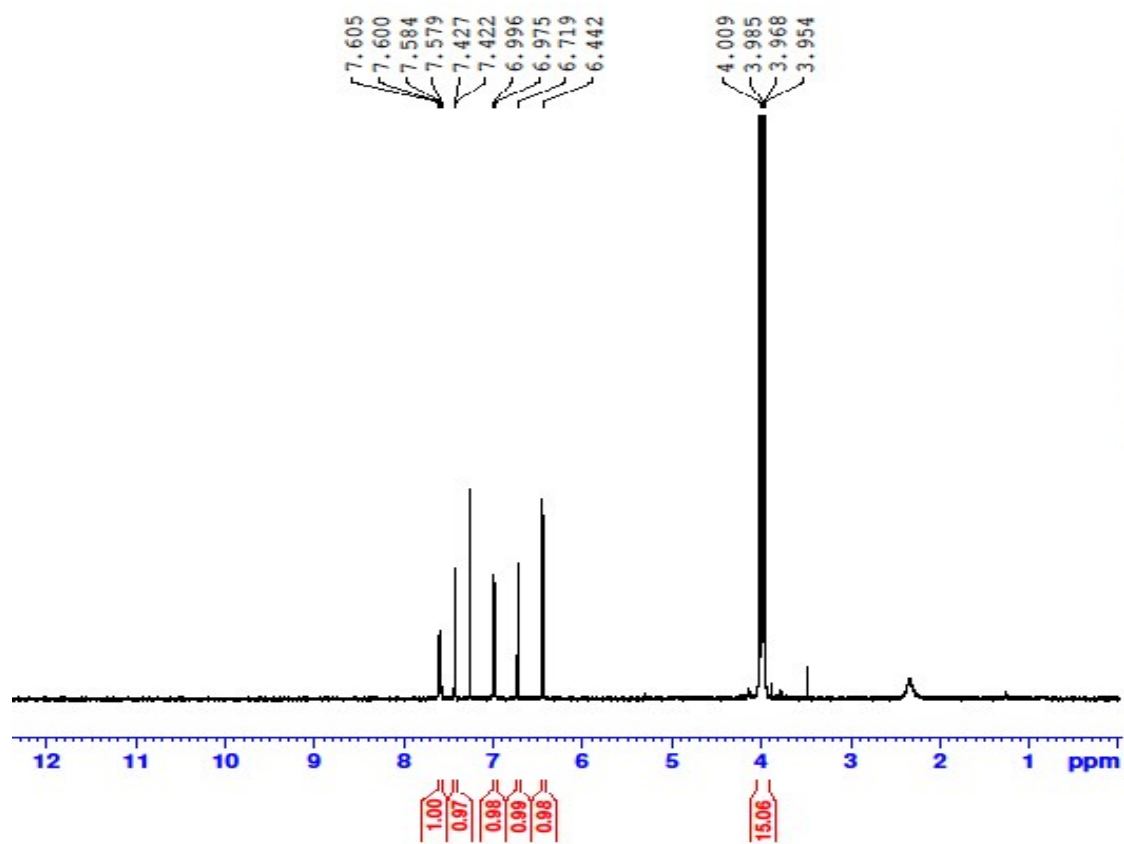


Fig. S4 ^1H -NMR spectrum of compound **C2** (Isosinensetin)

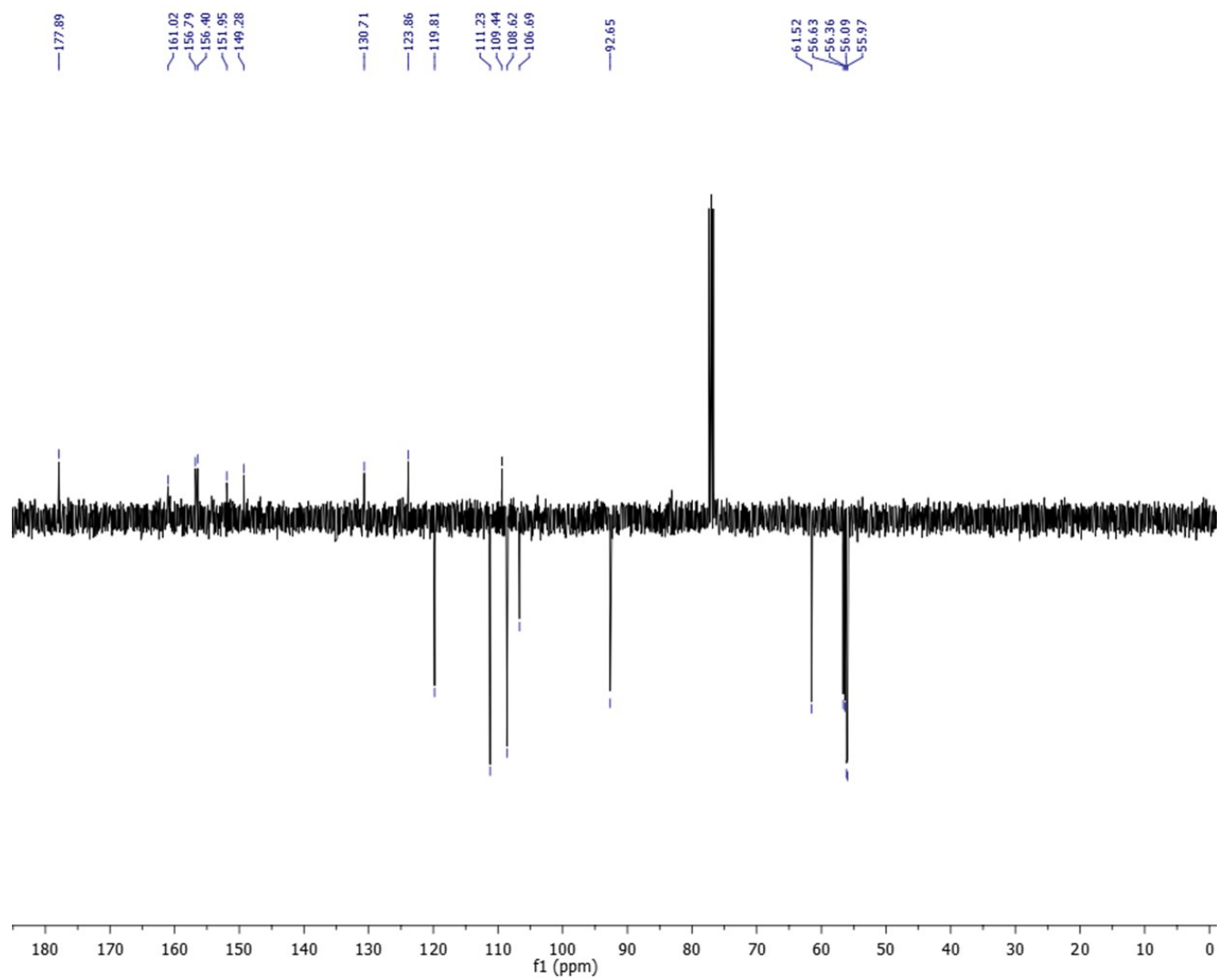


Fig. S5 ^{13}C -NMR spectrum of compound **C2** (Isosinensetin)

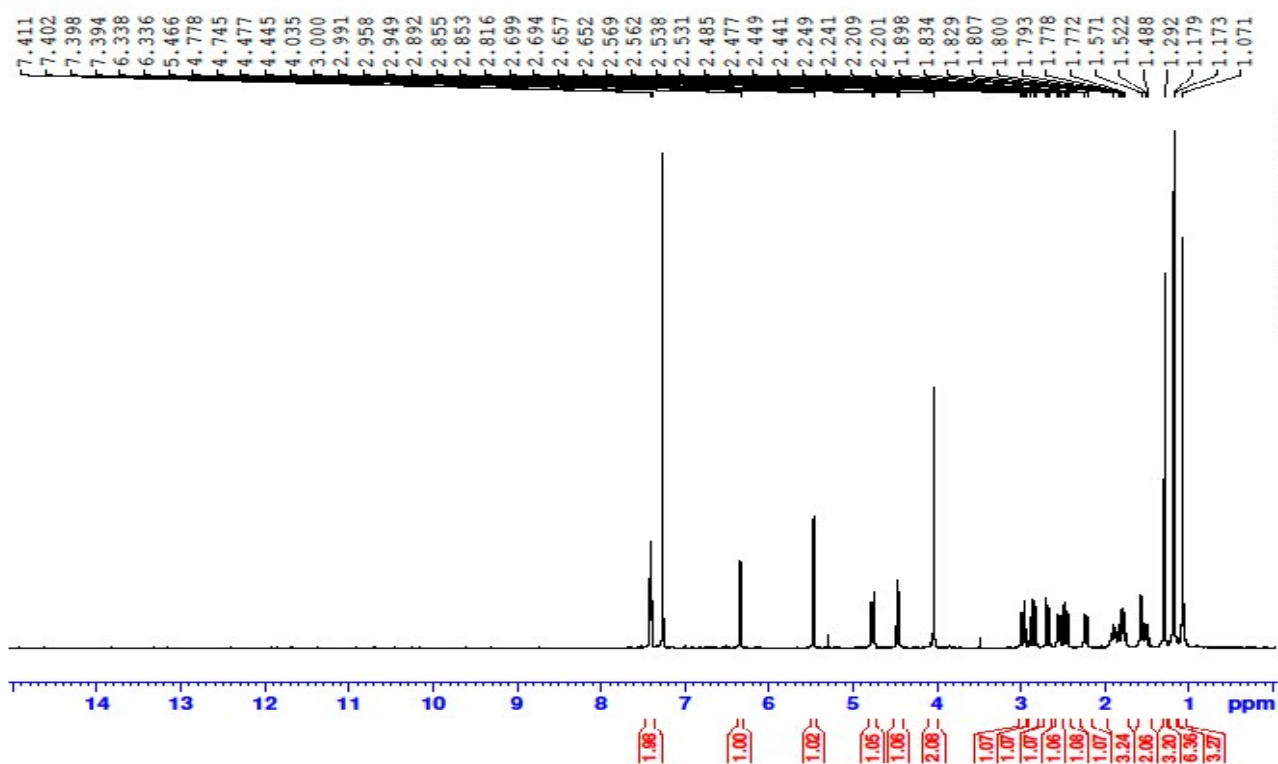


Fig. S6 ^1H -NMR spectrum of compound **C3** (Limonin)

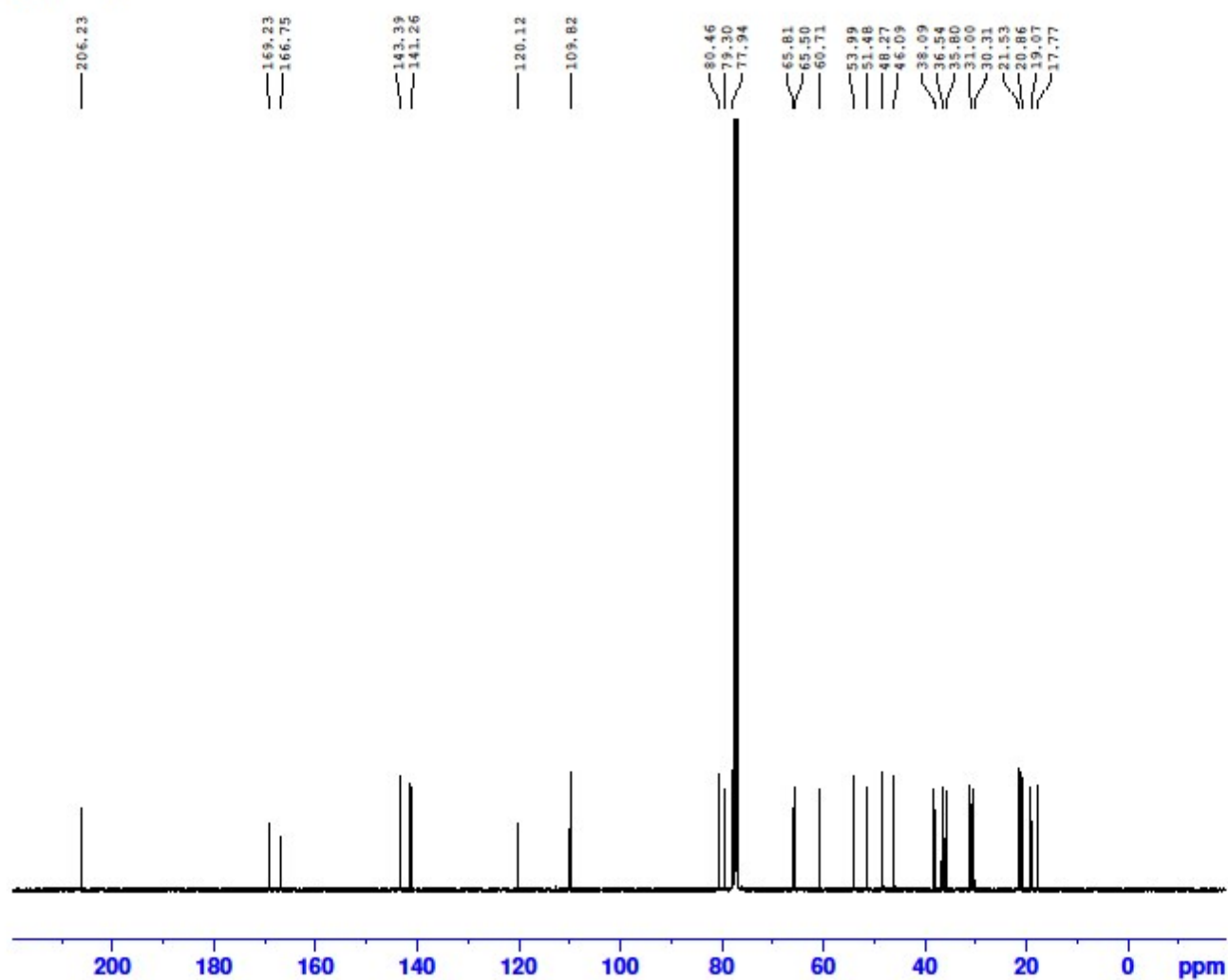


Fig. S7 ^{13}C -NMR spectrum of compound **C3** (Limonin)

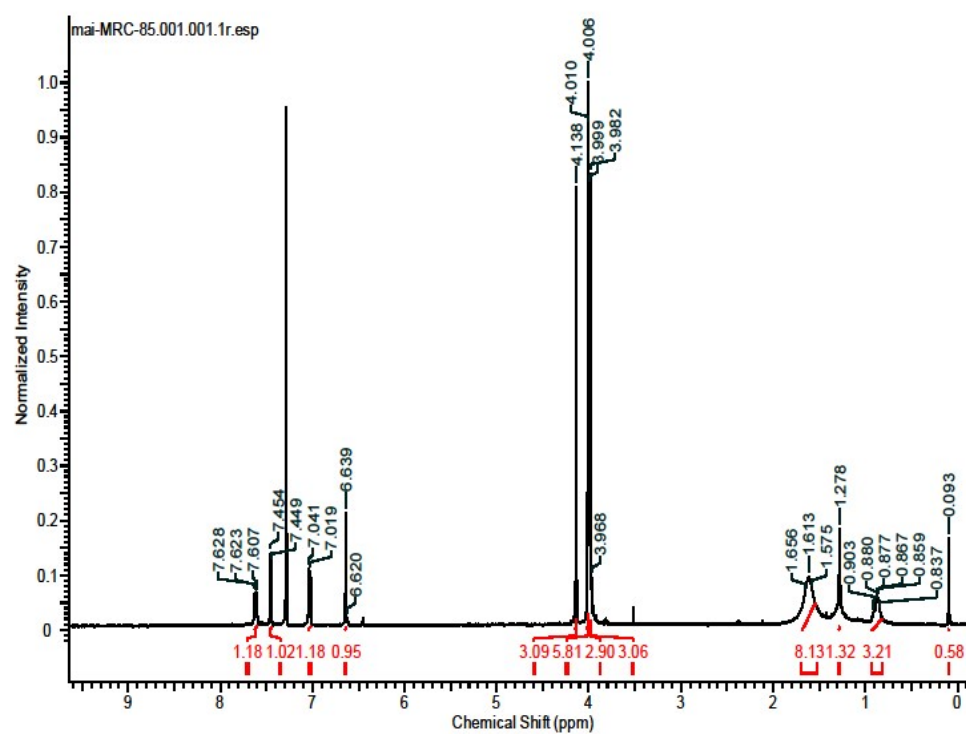


Fig. S8 ^1H -NMR spectrum of compound C4 (4-Demethylnobiletin)

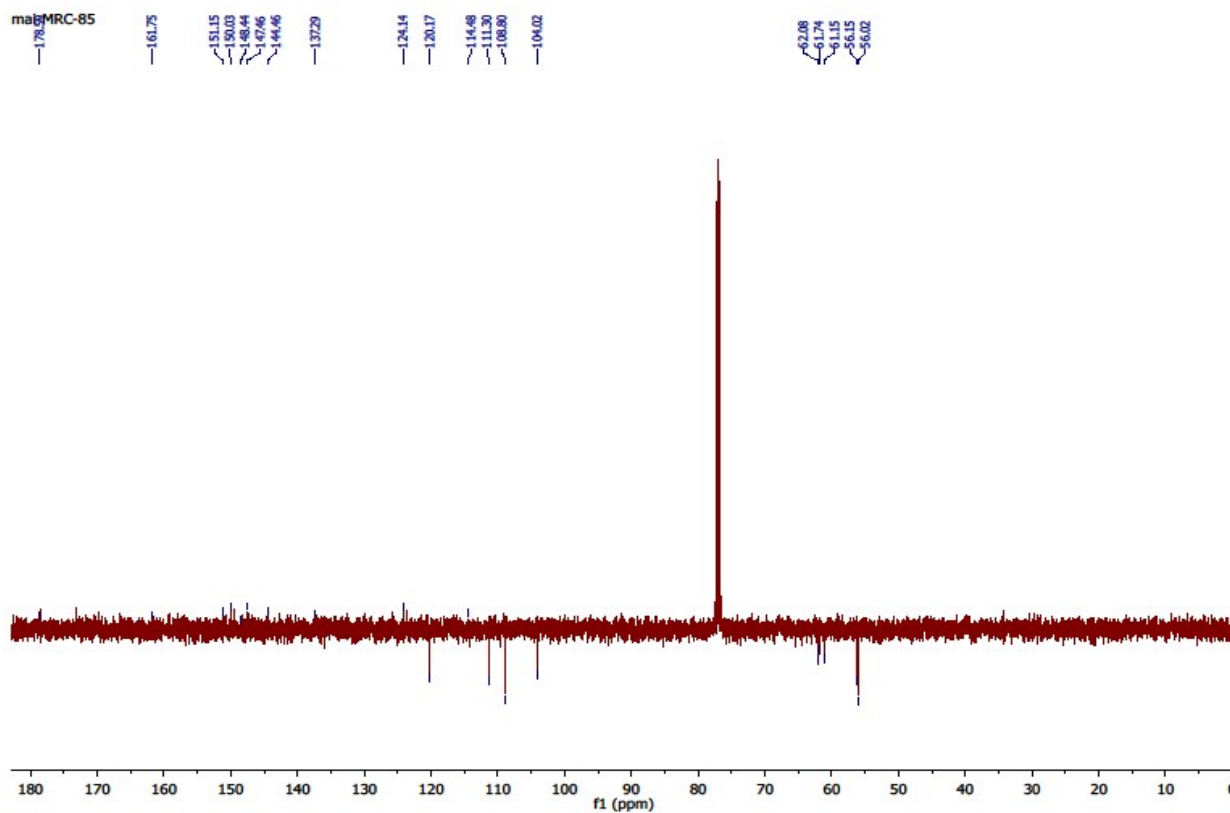


Fig. S9 ^{13}C -NMR spectrum of compound **C4** (4 β -Demethylnobiletin)

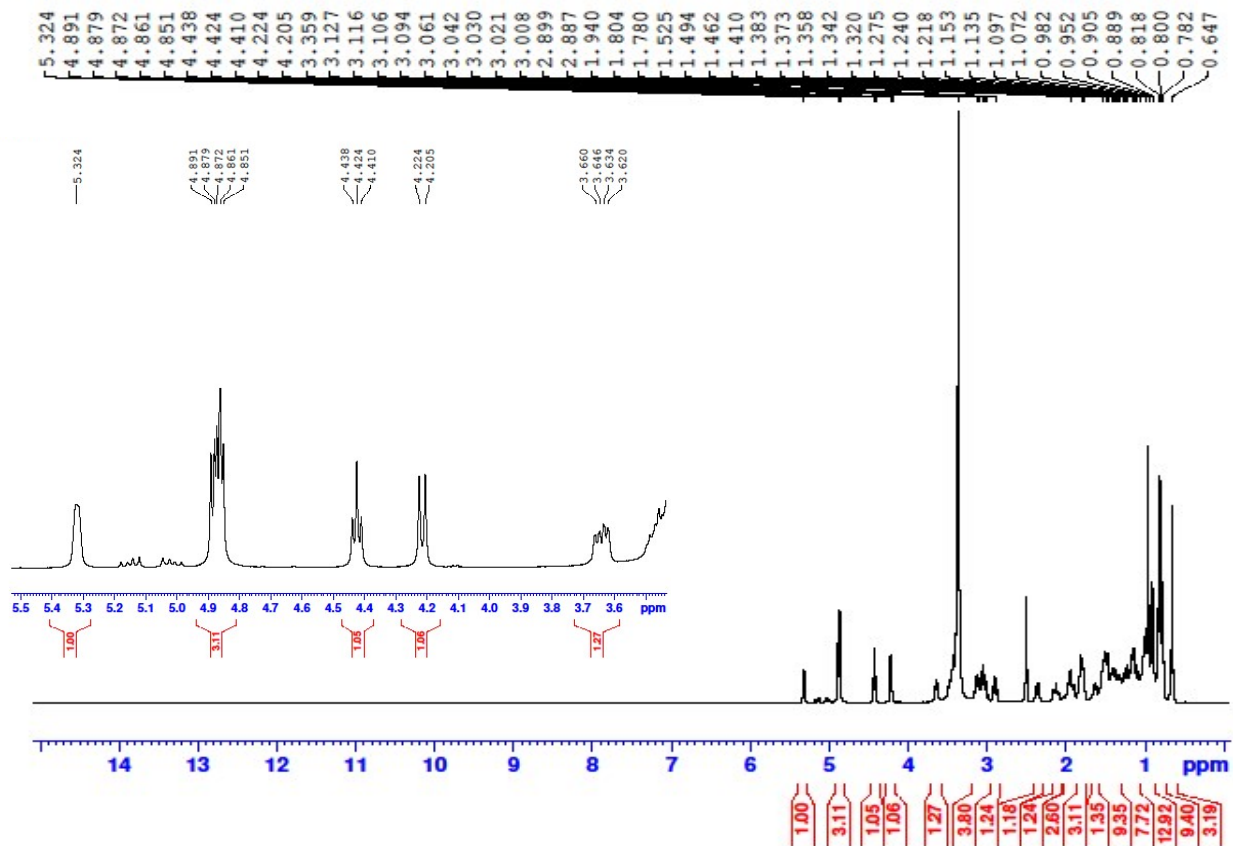


Fig. S10 ^1H -NMR spectrum of compound **C5** (Stigmasterol-O- β -D-glucoside)

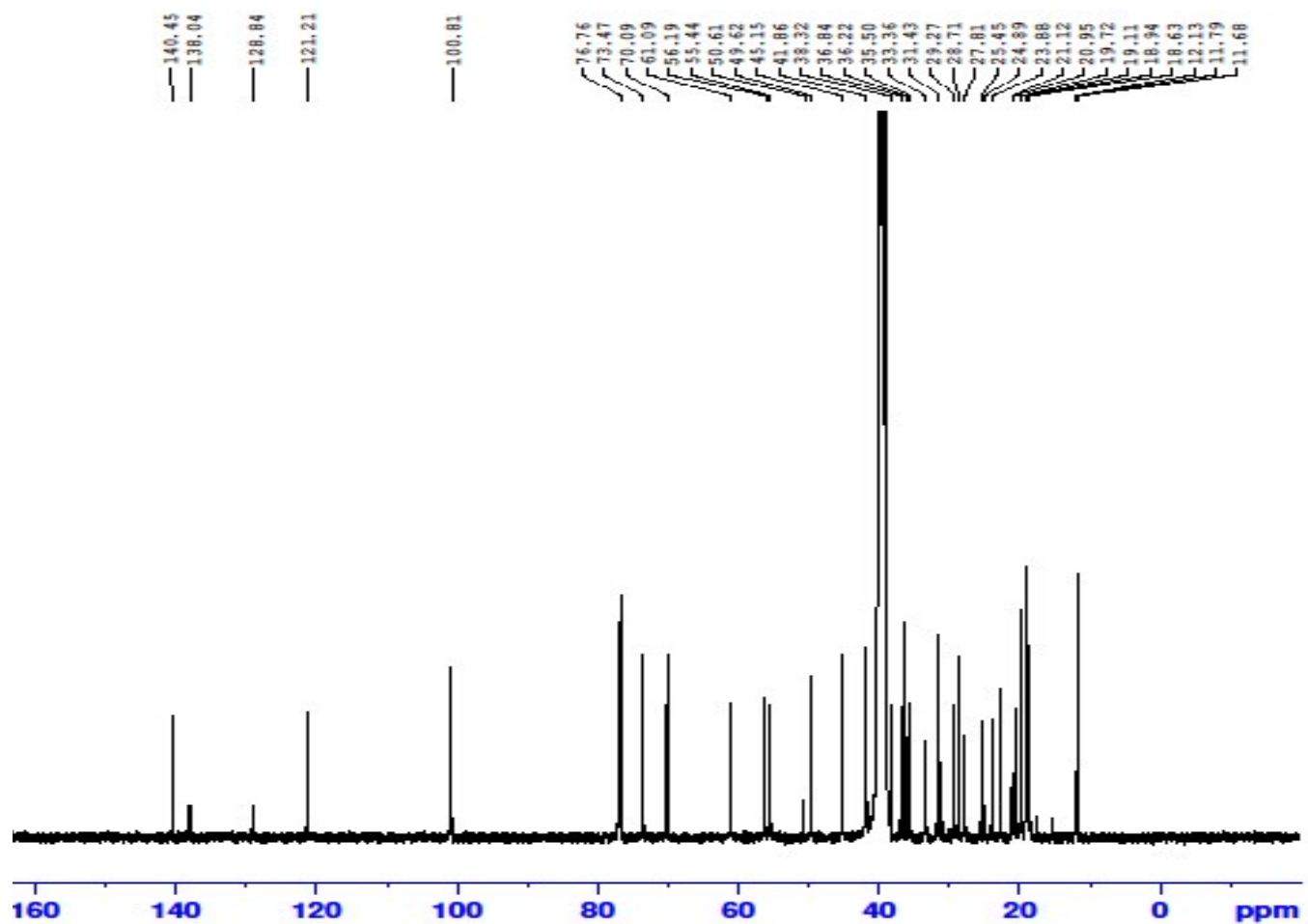


Fig. S11 ^{13}C -NMR spectrum of compound **C5** (Stigmasterol-O- β -D-glucoside)

MRC-173-HSQC

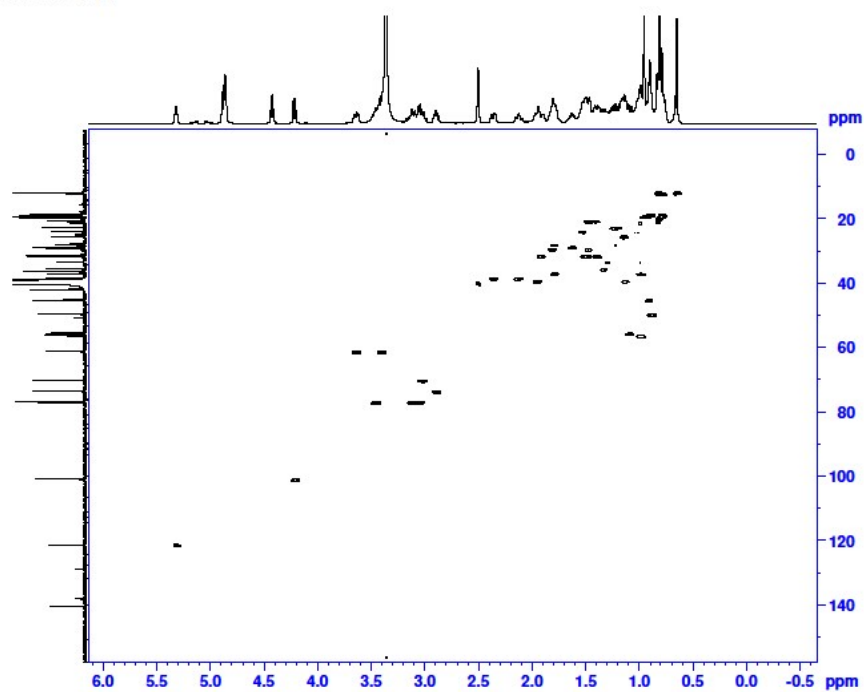


Fig. S12 HSQC spectrum of compound **C5** (Stigmasterol-O- β -D-glucoside)

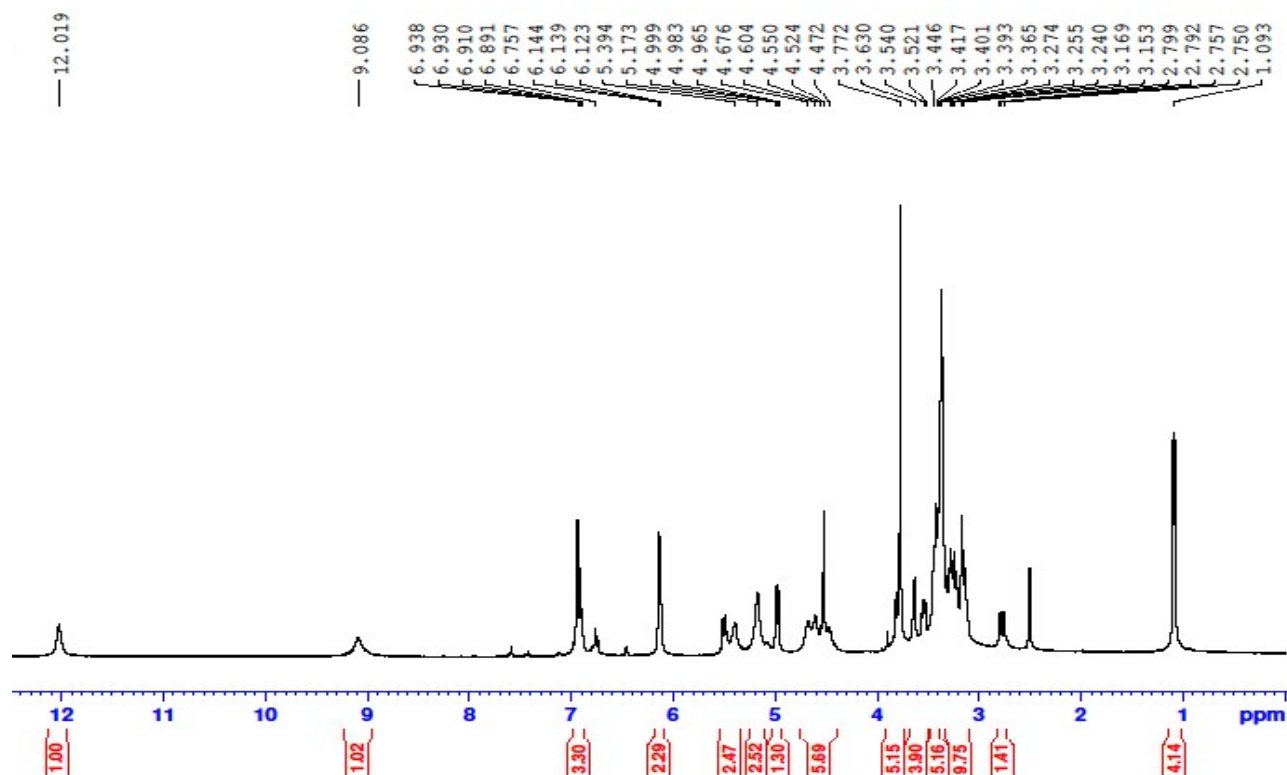


Fig. S13 ^1H -NMR spectrum of compound C6 (Hesperidin)

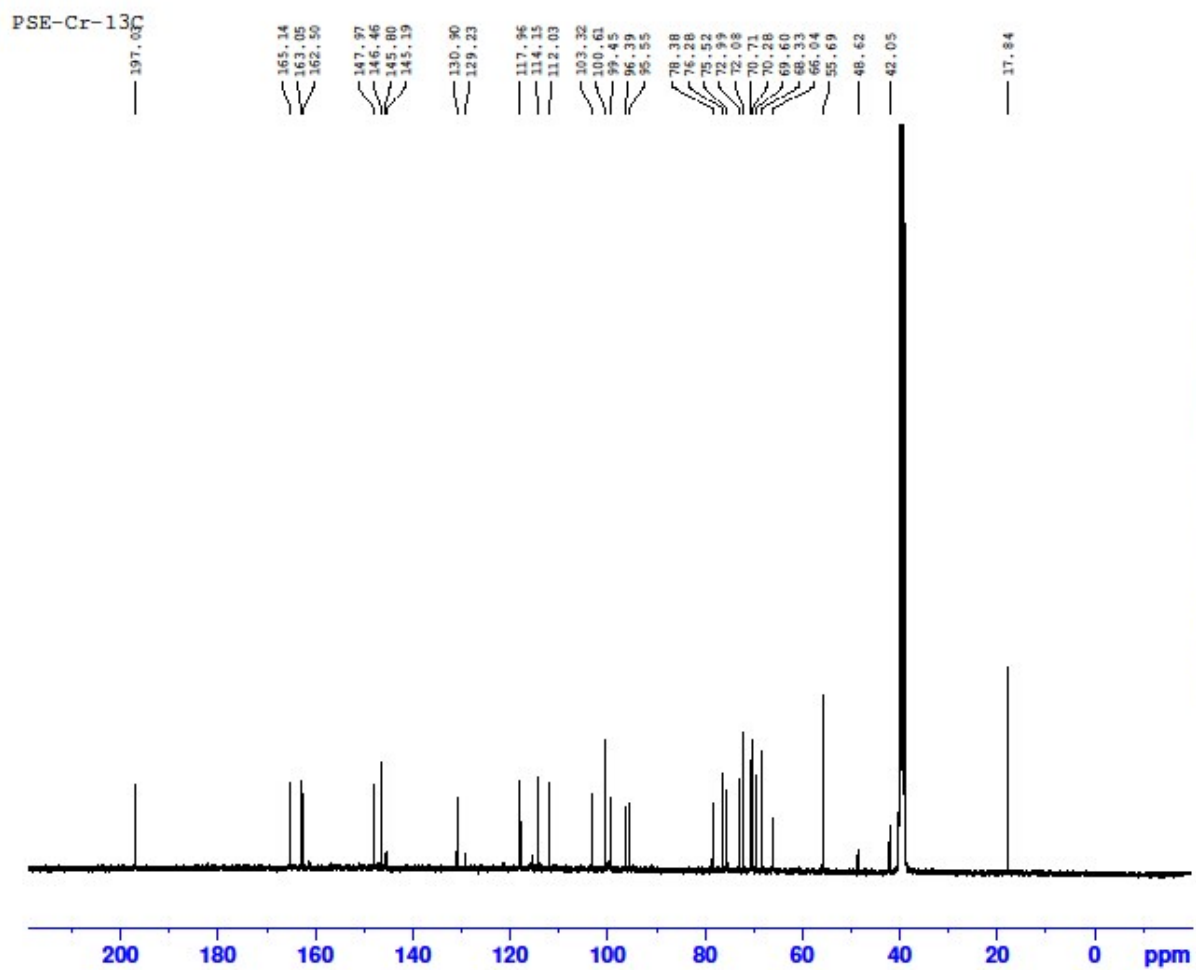


Fig. S14 ^{13}C -NMR spectrum of compound C6 (Hesperidin)

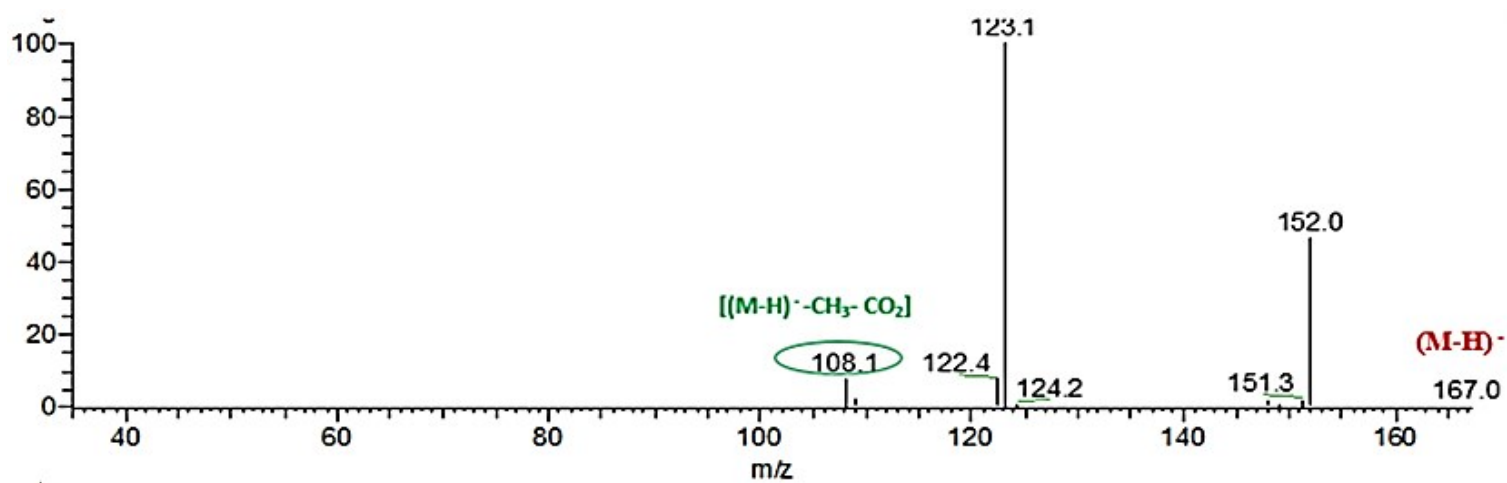


Fig. S15 MS-MS spectrum of vanillic acid (5)

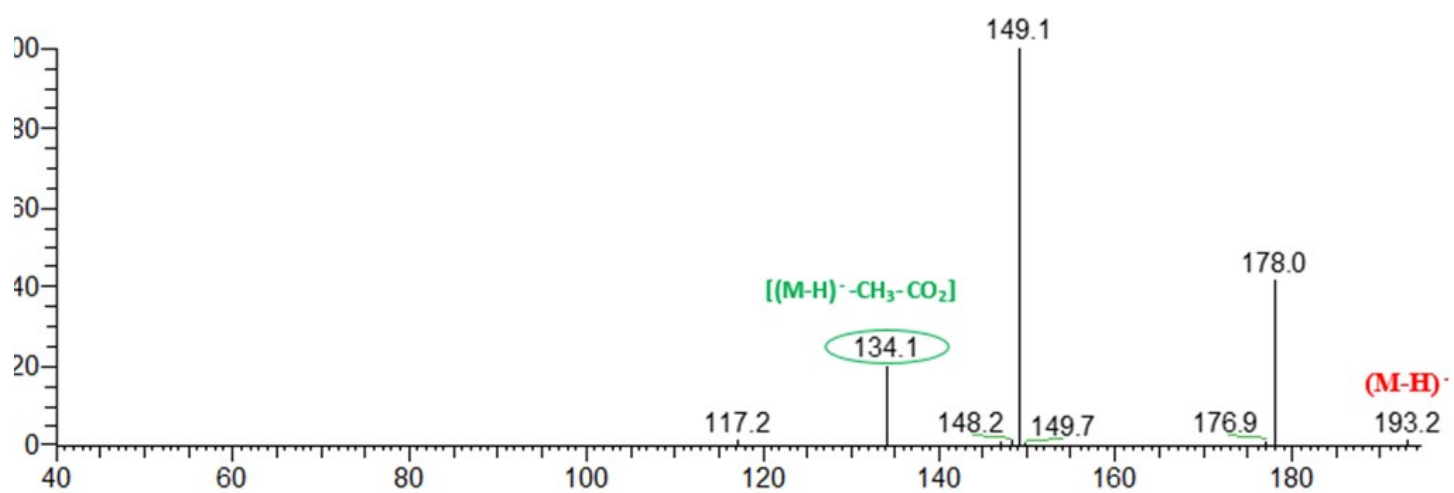


Fig. S16 MS-MS spectrum of ferulic acid (19)

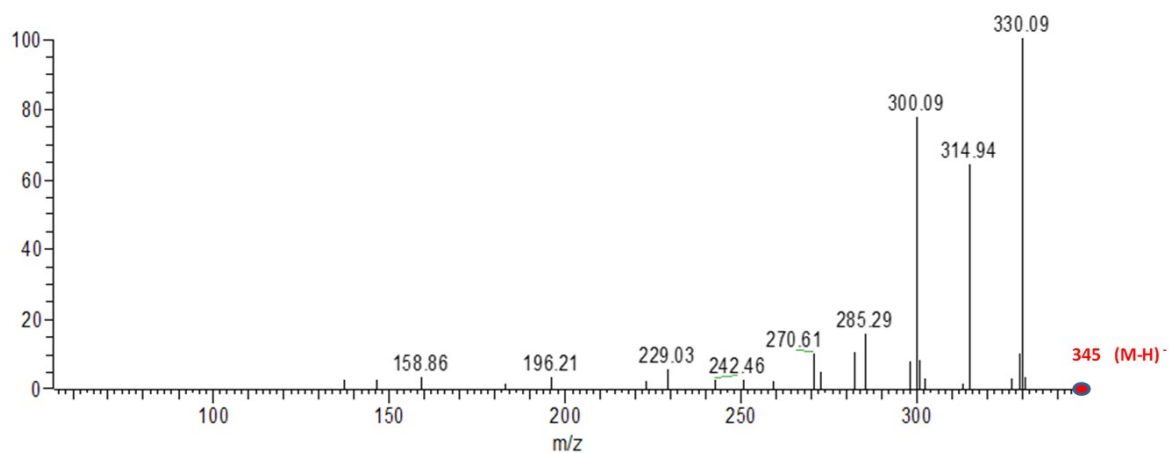


Fig. S17 MS-MS spectrum of syringetin (**18**)

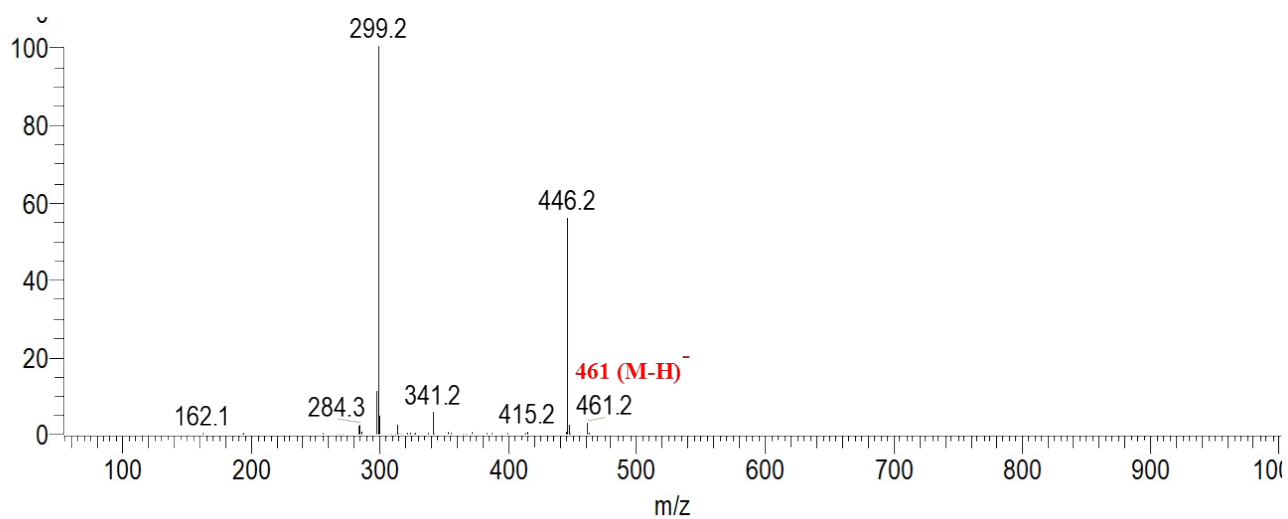


Fig. S18 MS-MS spectrum of chryseriol-7-O-glucoside (**63**)

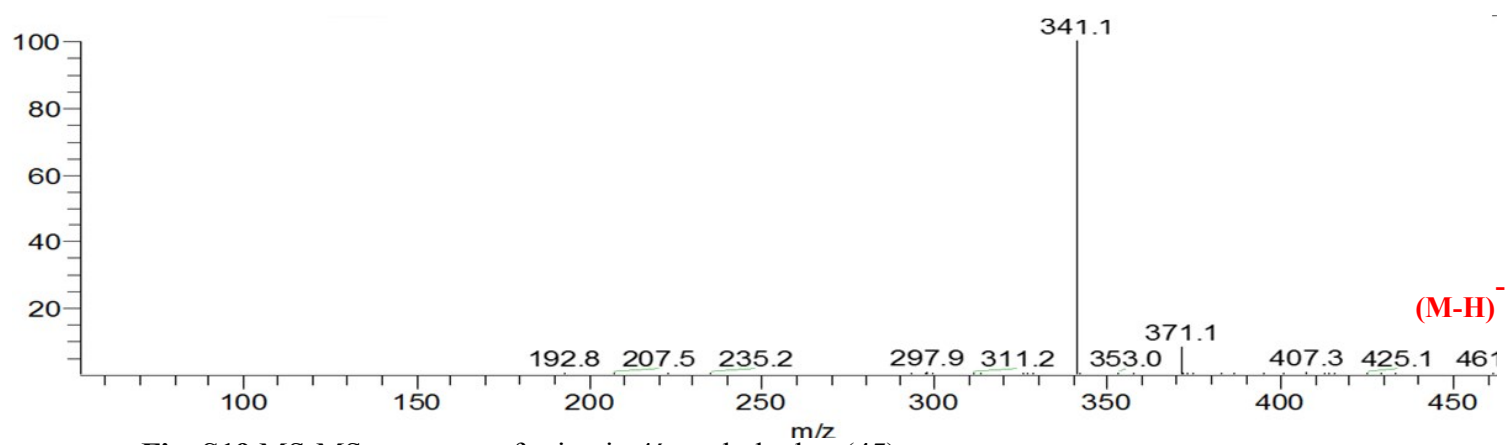


Fig. S19 MS-MS spectrum of orientin 4'-methyl ether (45)

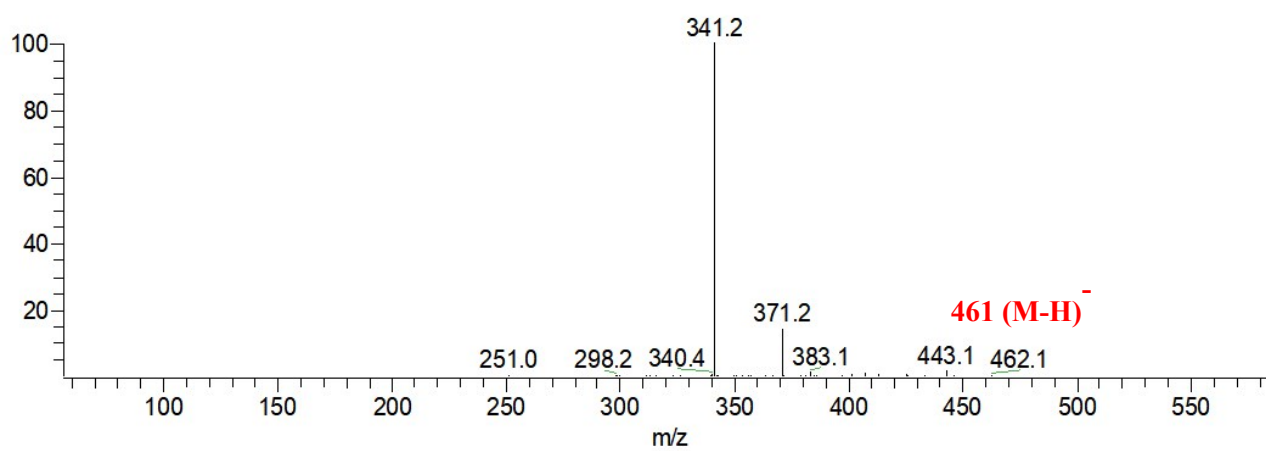
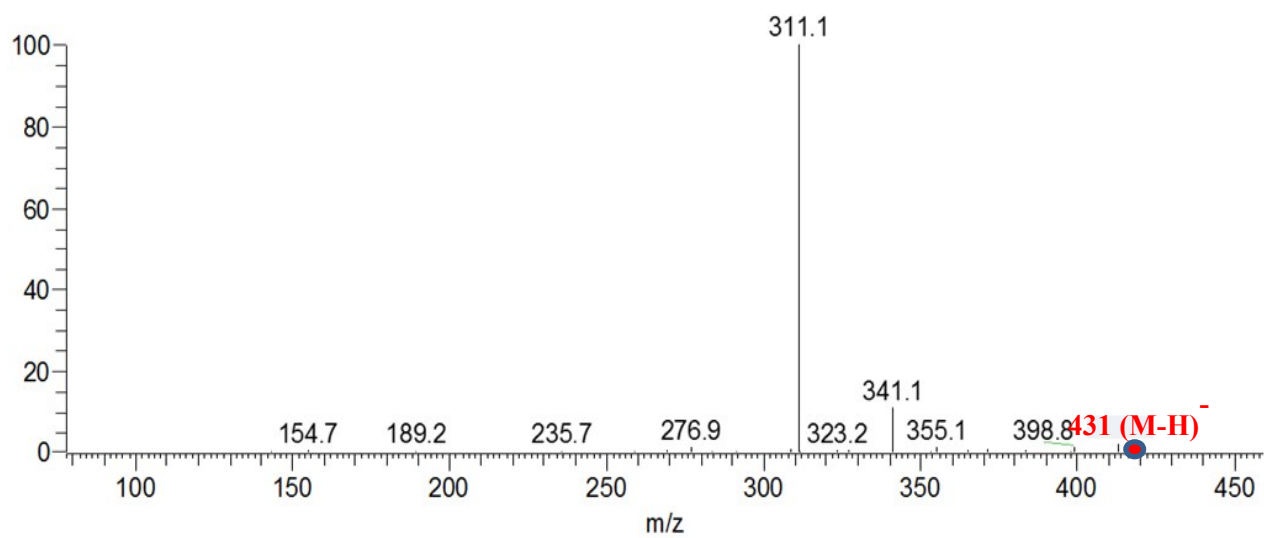


Fig. S20 MS-MS spectrum of scoparin (37)



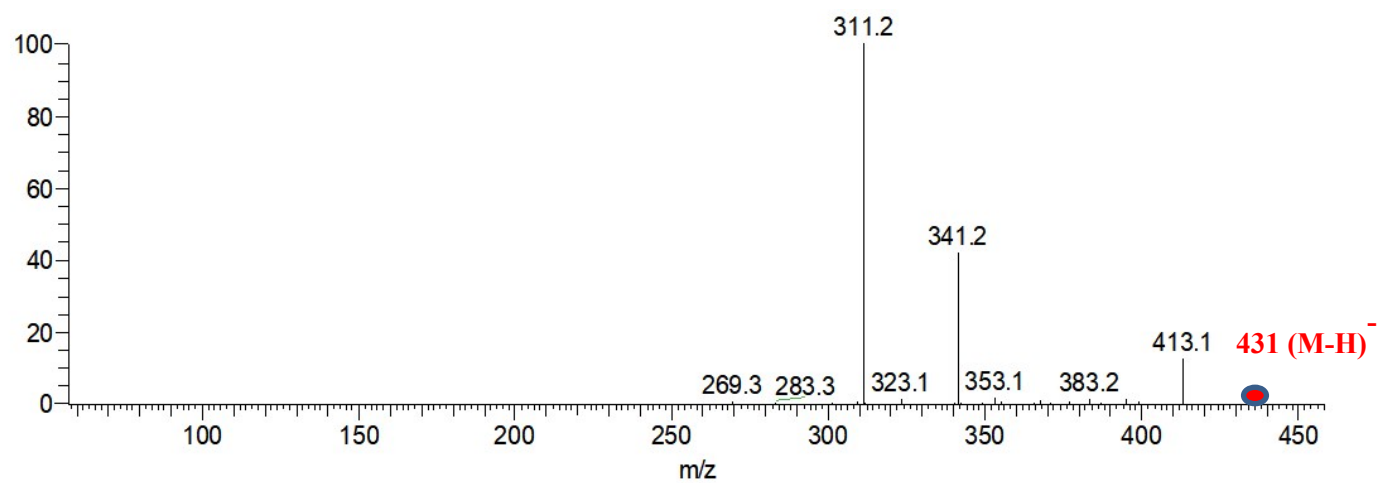


Fig. S22 MS-MS spectrum of isovitexin (**44**)

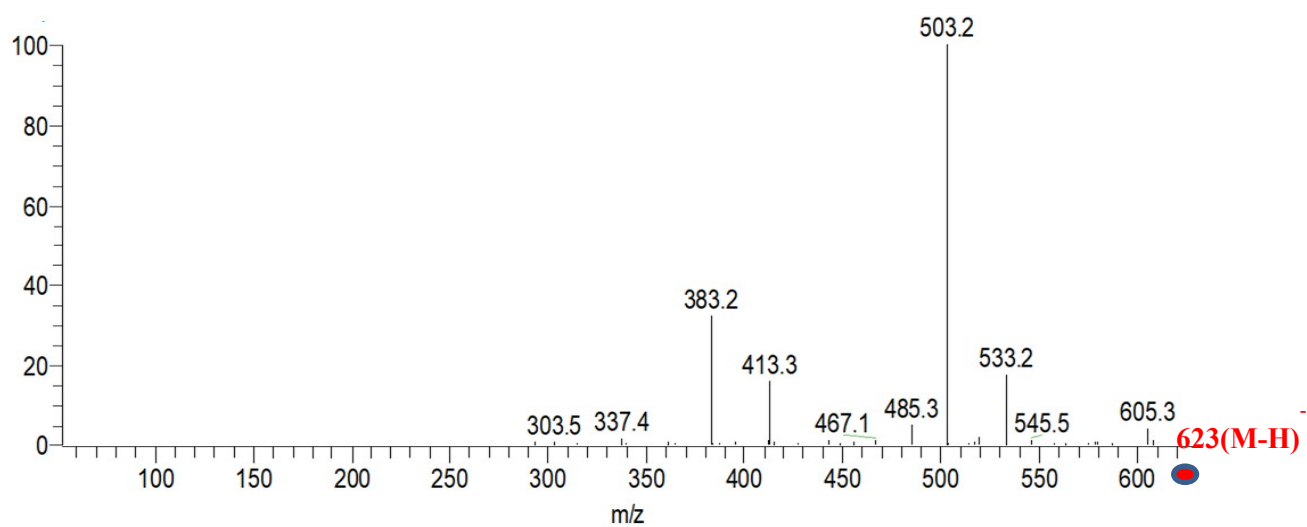


Fig. 23 MS-MS spectrum of lucenin-2 4'-methyl ether (**31**)

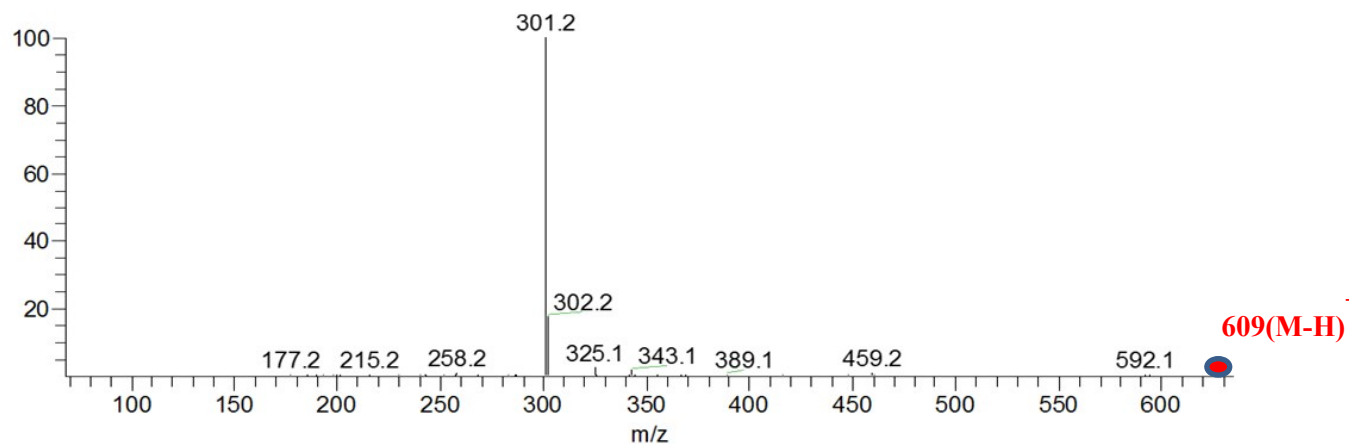


Fig. S24 MS-MS spectrum of hesperidin (**54**)

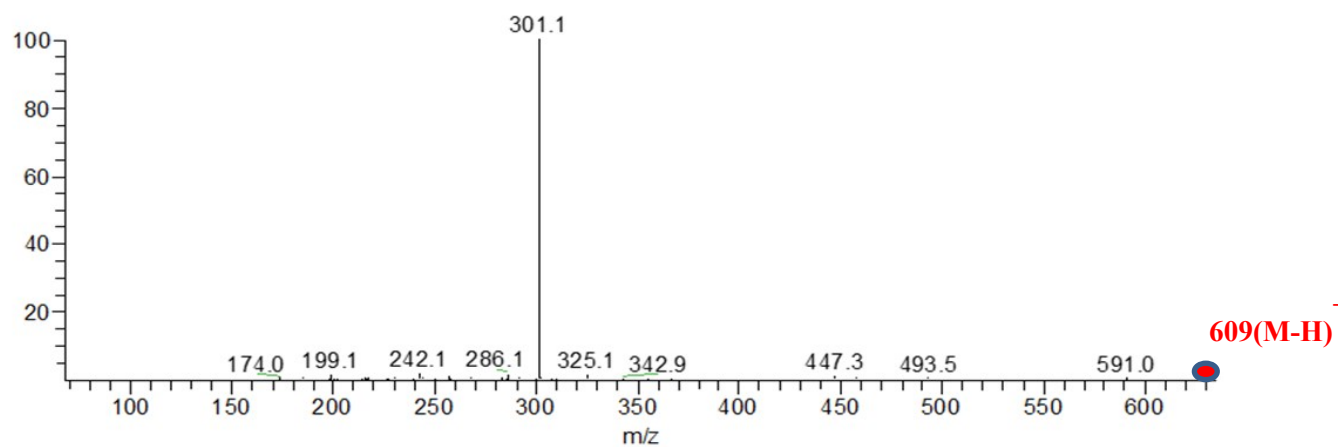


Fig. S25 MS-MS spectrum of neohesperidin (**61**)

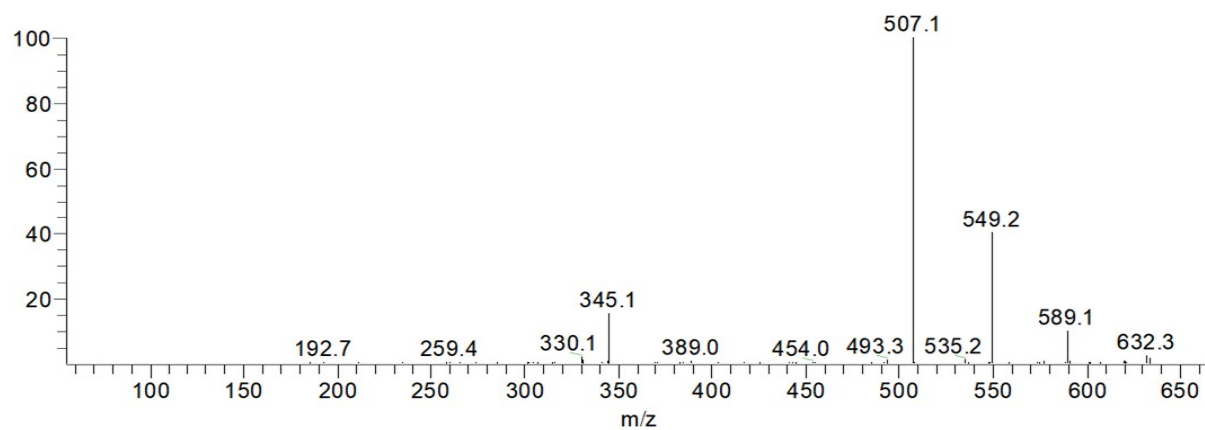


Fig. S26 MS-MS spectrum of limocitrin-O-Glc. HMG (**38**)

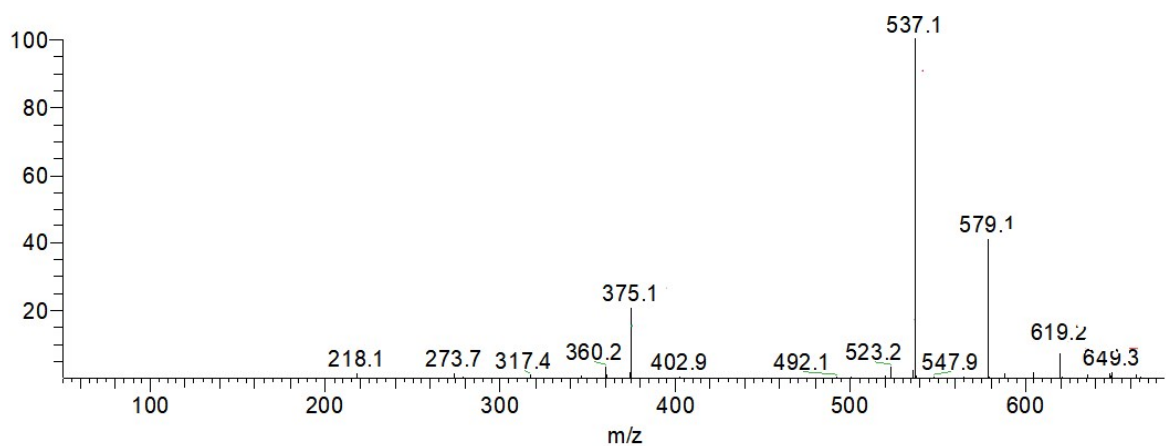
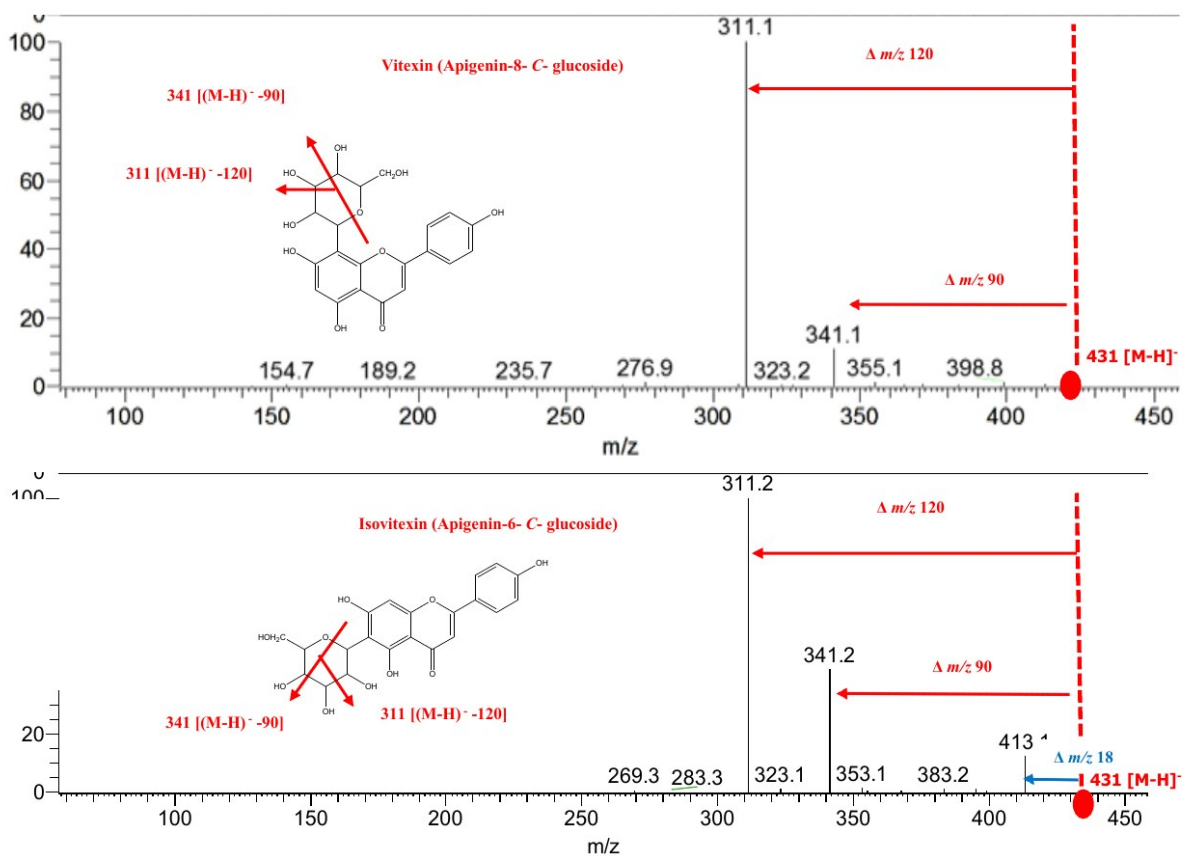
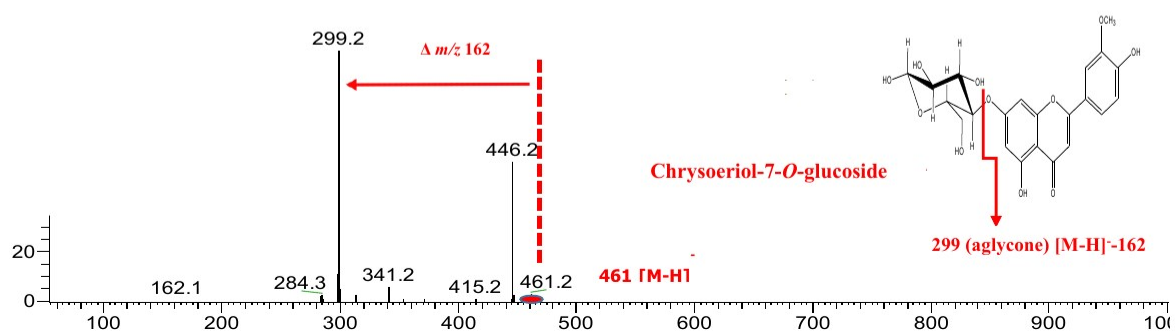
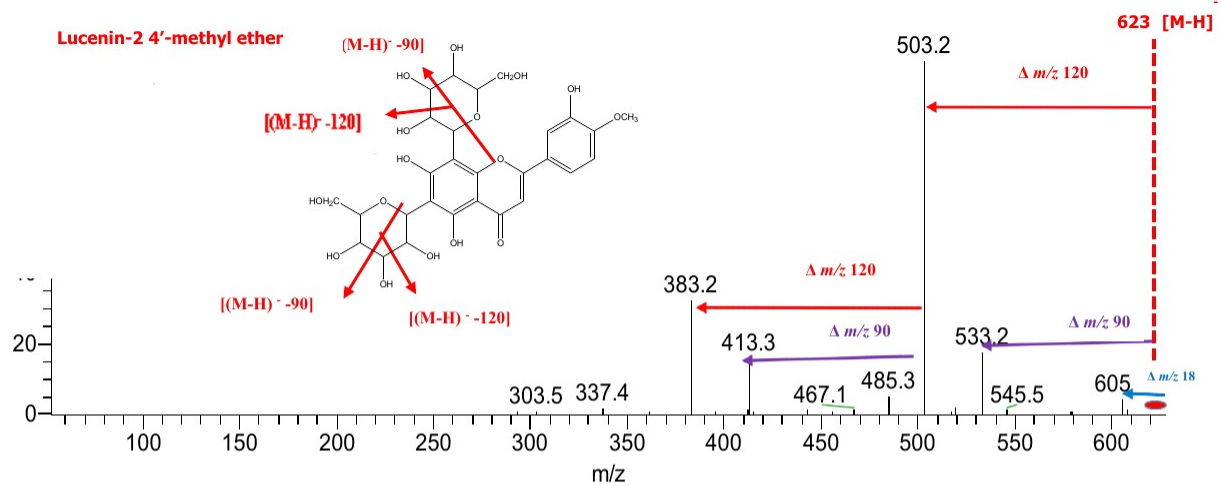


Fig. S27 MS- MS spectrum of limocitrol-O-Glc. HMG (42)





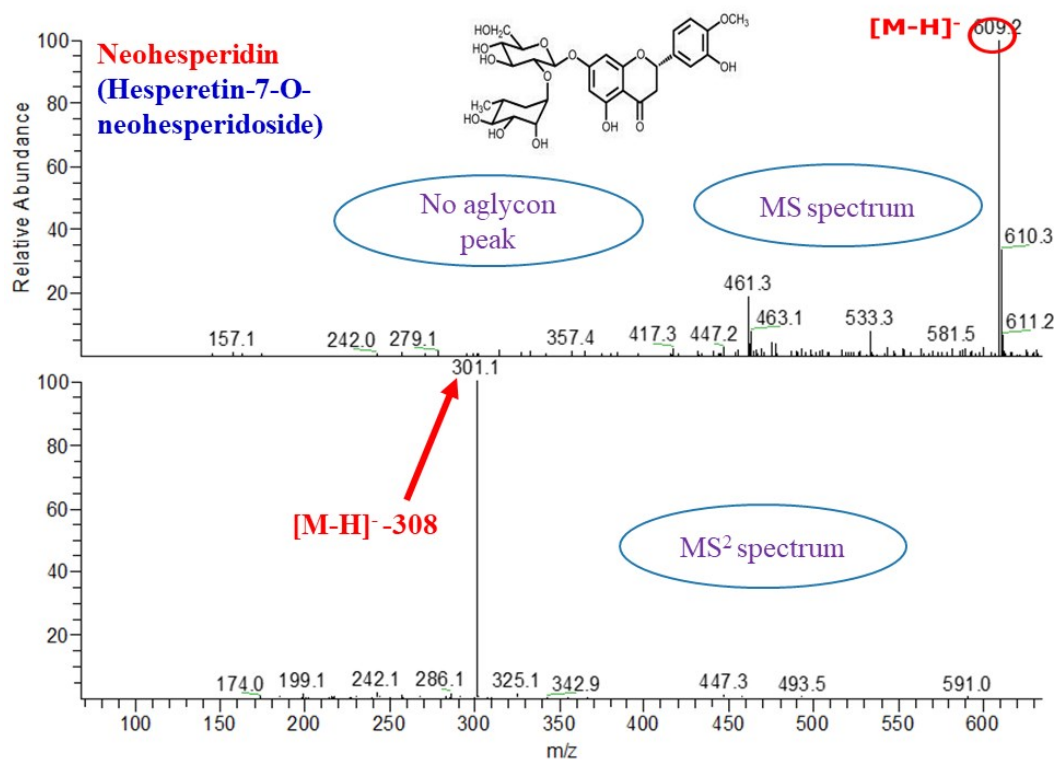
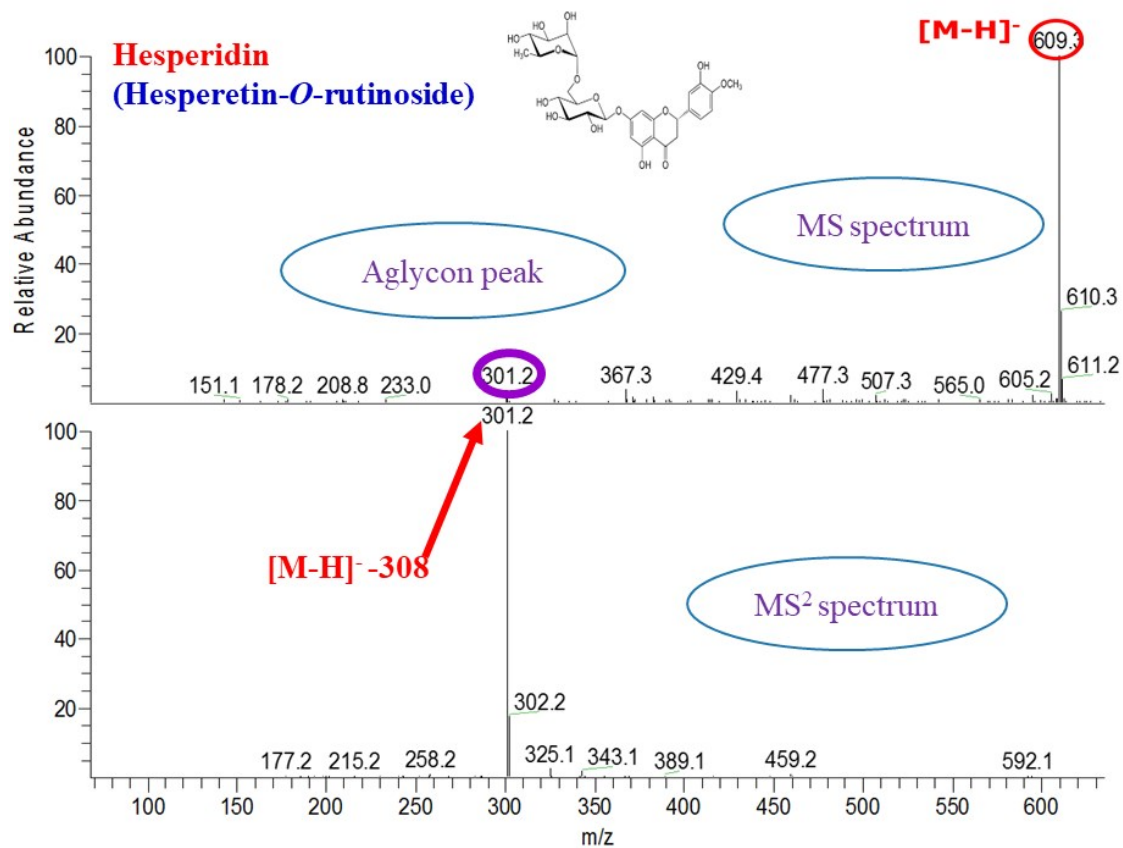


Fig. S28: MS/MS fragmentation pathways of some selected compounds

Supplementary Texts: Characterization of the isolated compounds

Text S1: Identification of compound C1 (Nobiletin): white flakes (55 mg) with R_f 0.5 and 0.69 (S2 and S3, respectively). UV: $\lambda_{\max}$ (MeOH) nm: 248 (sh), 270 (sh), 333; (+NaOMe): 248 (sh), 270 (sh), 332; (+ AlCl_3): 245 (sh), 269 (sh), 331; (+ AlCl_3 + HCl): 247 (sh), 269 (sh), 332; (+ NaOAc): 248 (sh), 270 (sh), 332; (+ NaOAc+Boric acid): 248 (sh), 270 (sh), 332. EI-MS: m/z (relative abundance %): 402 (M^+ , 27), 387 (100), 371 (6.3), 359 (6.1), 344 (13), 182 (6.3), 162 (4.0), 153 (2.3), 147 (2.0), 119 (1.8), 91 (2.8) and 83 (6.7). ^1H -NMR (400 MHz, CDCl_3): δ ppm 6.72 (1H, *s*, H-3), 7.40 (1H, *d*, $J=2$, H-2'), 6.98 (1H, *d*, $J=8.4$, H-5'), 7.57 (1H, *dd*, $J=2, 8.4$, H-6'), 4.90 (3H, *s*, 5-OCH₃), 4.01 (3H, *s*, 6-OCH₃), 3.96 (3H, *s*, 7-OCH₃), 3.95 (3H, *s*, 8-OCH₃), 3.94 (3H, *s*, 3'-OCH₃) and 3.94 (3H, *s*, 4'-OCH₃). ^{13}C -NMR (100 MHz, CDCl_3): δ ppm 161.36 (C-2), 106.77 (C-3), 177.44 (C-4), 144.25 (C-5), 136.08 (C-6), 151.63 (C-7), 147.81 (C-8), 148.49 (C-9), 114.76 (C-10), 124.00 (C-1'), 108.68 (C-2'), 149.39 (C-3'), 152.13 (C-4'), 111.34 (C-5'), 119.83 (C-6'), 61.77 (5-OCH₃), 62.37 (6-OCH₃), 62.07 (7-OCH₃), 61.91 (8-OCH₃), 56.19 (3'-OCH₃) and 56.07 (4'-OCH₃). Spectral data of UV, EI-MS and NMR of this compound were found to be identical to nobiletin upon comparison with the reported data.¹

Text S2: Identification of compound C2 (Isosinensetin): White needles (75 mg) with R_f 0.61 (S3). UV: $\lambda_{\max}$ (MeOH) nm: 248 (sh), 270 (sh), 338; (+ NaOMe): 247 (sh), 269 (sh), 340; (+ AlCl_3): 240 (sh), 270 (sh), 342; (+ AlCl_3 + HCl): 239 (sh), 270 (sh), 345; (+ NaOAc): 248 (sh), 270 (sh), 338; (+ NaOAc+ Boric acid): 248 (sh), 270 (sh), 339. EI-MS m/z (relative abundance %): 372 (M^+ , 74.66), 357 (100), 342 (17.43), 329 (23.6), 328 (32.4), 327 (23.3), 313 (6.3), 312 (2.0), 299 (12.8), 298 (5.2), 297 (1.4), 283 (1.6), 167 (1.0) and 162 (1.1). ^1H -NMR (400 MHz, CDCl_3): δ

ppm 6.72 (1H, *s*, H-3), 6.44 (1H, *s*, H-6), 7.42 (1H, *d*, *J*=2, H-2'), 6.99 (1H, *d*, *J*=8.4, H-5'), 7.59 (1H, *dd*, *J*=2, 8.2, H-6'), 4.01 (3H, *s*, 5-OCH₃), 3.99 (3H, *s*, 7-OCH₃), 3.97 (3H, *s*, 8-OCH₃), 3.95 (3H, *s*, 3'-OCH₃) and 3.95 (3H, *s*, 4'-OCH₃). ¹³C-NMR-APT (100 MHz, CDCl₃): δ ppm 161.02 (C-2), 106.69 (C-3), 177.89 (C-4), 156.40 (C-5), 92.65 (C-6), 156.79 (C-7), 130.71 (C-8), 151.95 (C-9), 109.44 (C-10), 123.86 (C-1'), 108.62 (C-2'), 149.28 (C-3'), 151.95 (C-4'), 111.23 (C-5'), 119.81 (C-6'), 56.36 (5-OCH₃), 56.63 (7-OCH₃), 61.52 (8-OCH₃), 56.09 (3'-OCH₃) and 55.97 (4'-OCH₃). The above spectral data are identical for isosinensetin with the reported one.²

Text S3: Identification of compound C3 (Limonin): White needles (45 mg) with R_f value 0.74 (S3). EI-MS, *m/z* (relative abundance %): 413 (M⁺-57, 1.82), 347 (100), 329 (8.16), 287 (6.22), 241 (3.83), 201 (4.91), 187 (5.58), 147 (6.46), 136 (9.10), 135 (19.08), 108 (19.94), 95 (39.22), 69 (18.33) and 43 (27.15). ¹H-NMR (400 MHz, DMSO-*d*₆): δ ppm 4.04 (1H, *br. s*, H-1), 2.23 (1H, *dd*, *J*=3.2, 16, H-2a), 2.68 (1H, *dd*, *J*=2, 16.8 H-2b), 2.46 (1H, *dd*, *J*=3.2, 14.4, H-5), 2.98 (1H, *dd*, *J*=3.6, 16.8, H-6a), 2.85 (1H, *t*, *J*=15.2, H-6b), 2.55 (1H, *dd*, *J*=2.8, 12.6, H-9), 1.85 (1H, *m*, H-11a), 1.91 (1H, *m*, H-11b), 1.50 (1H, *m*, H-12a), 1.77 (1H, *m*, H-12b), 4.04 (1H, *br. s*, H-15), 5.47 (1H, *s*, H-17), 1.18 (3H, *s*, H-18), 4.46 (1H, *d*, *J*=12.8, H-19a), 4.75 (1H, *d*, *J*=13.2, H-19b), 7.41 (1H, *br. s*, H-21), 6.34 (1H, *br. s*, H-22), 7.40 (1H, *br. s*, H-23), 1.07 (3H, *s*, H-24), 1.29 (3H, *s*, H-25) and 1.17 (3H, *s*, H-26). ¹³C-NMR (100 MHz, DMSO-*d*₆): δ ppm 79.3 (C-1), 35.8 (C-2), 169.23 (C-3), 80.46 (C-4), 61.71 (C-5), 36.54 (C-6), 206.23 (C-7), 51.48 (C-8), 48.27 (C-9), 46.09 (C-10), 19.07 (C-11), 30.31 (C-12), 38.09 (C-13), 65.81 (C-14), 53.99 (C-15), 166.75 (C-16), 77.94 (C-17), 17.77 (C-18), 65.50 (C-19), 120.12 (C-20), 143.39 (C-21), 109.82 (C-22), 141.26 (C-23), 20.86 (C-24), 31.00 (C-25) and 21.53 (C-26). This compound was confirmed upon comparison of their spectral data with the available literature¹).

Text S4: Identification of compound C4 (4- α -Demethylnobiletin): yellow crystals (48 mg) with R_f value 0.9 (S2). UV: $\lambda_{\max}$ (MeOH) nm: 252 (sh), 278, 340; (+NaOMe): 291, 313, 391 (sh); (+ AlCl₃): 281, 354; (+ AlCl₃+ HCl): 283, 353; (+ NaOAc): 252 (sh), 280, 345; (+ NaOAc+Boric acid): 283, 336. EI-MS, m/z (relative abundance %): 388 (M⁺, 13.0), 373 (25.1), 358 (35.5), 357 (22.3), 344 (20.6), 343 (100), 328 (13.3), 313 (7.2), 225 (0.9), 197 (1.1), 151 (2.6) and 148 (3.2). ¹H-NMR (400 MHz, CDCl₃): δ ppm 6.64 (1H, *s*, H-3), 7.45 (1H, *d*, J=2, H-2'), 7.03 (1H, *d*, J=8, H-5'), 7.61 (1H, *dd*, J=8, 2, H-6'), 4.14 (3H, *s*, O-CH₃), 4.01 (3H, *s*, O-CH₃), 4.01 (3H, *s*, O-CH₃), 4.00 (3H, *s*, O-CH₃), and 3.98 (3H, *s*, O-CH₃). ¹³C-NMR-APT (100 MHz, CDCl₃): δ ppm 161.75 (C-2), 111 104.02 (C-3), 178.60 (C-4), 144.46 (C-5), 137.29 (C-6), 151.15 (C-7), 147.46 (C-8), 148.44 (C-9), 114.48 (C-10), 124.14 (C-1'), 108.80 (C-2'), 150.03 (C-3'), 151.15 (C-4'), 111.30 (C-5'), 120.17 (C-6'), 61.74 (5-OCH₃), 56.02 (6-OCH₃), 62.08 (7-OCH₃), 56.15 (8-OCH₃) and 61.15 (3-OCH₃). These spectral data are matching with the previously reported ones¹).

Text S5: Identification of compound C5 (Stigmasterol-O- β -D-glucoside): yellowish white amorphous powder (39 mg) with R_f 0.6 (S3). EI-MS, m/z (relative abundance %): 412 [M⁺ - sugar] (1), 397 (64.2), 396 (100), 394 (23.5), 382 (17.4), 381 (13.5), 329 (1.1), 303 (1.2), 255 (25.3), 213 (16.4), 189 (5.8), 173 (10.9), 157 (8.2), 145 (37.6), 131 (14.6), 117 (8.4), 105 (21.7), 93 (20.6), 81 (29.7), 69 (27.9), and 55 (19). ¹H- NMR (400 MHz; DMSO-*d*₆): δ (ppm): 3.64 (1H, *m*, H-3), 5.32 (1H, *br s.*, H-6), 0.66 (3H, *s*, H-18), 0.98 (3H, *s*, H-19), 0.90 (3H, *d*, J = 6.4 Hz, H-21), 4.87 (1H, *m*, H-22), 4.42 (1H, *t*, J = 5.6 Hz, H-23), 0.79 (3H, *d*, J = 7.2 Hz, H-26), 0.79 (3H,

d, *J* = 6.8 Hz, H-27), 0.83 (3H, *d*, *J* = 6.8 Hz, H-29), 4.22 (1H, *d*, *J* = 7.6 Hz, H-1'), 3- 3.6 (4H, *m*, sugar protons 2', 3', 4', 5'), 2.89 (1H, *m*, H-6'α), 2.4 (1H, *m*, H-6'β) and 1 – 2.9 (16H, CH₂ of steroid nucleus and side chain). ¹³C-NMR (100 MHz, DMSO-*d*₆): δ ppm 36.84 (C-1), 31.43 (C-2), 73.47 (C-3), 41.86 (C-4), 140.45 (C-5), 121.21 (C-6), 31.36 (C-7), 31.39 (C-8), 49.62 (C-9), 36.22 (C-10), 19.72 (C-11), 38.32 (C-12), 45.15 (C-13), 56.19 (C-14), 23.88 (C-15), 28.71 (C-16), 55.44 (C-17), 11.7 (C-18), 18.63 (C-19), 35.5 (C-20), 20.95 (C-21), 138.04 (C-22), 128.84 (C-23), 50.61 (C-24), 28.7 (C-25), 19.11 (C-26), 18.94 (C-27), 25.45 (C-28), 11.68 (C-29), 100.81 (C-1□), 76.95 (C-2□), 76.76 (C-3□), 70.09 (C-4□), 76.74 (C-5□) and 61.09 (C-6□). The characterization of this compound depends on comparison with literature.^{3,4}

Text S6: Identification of compound C6 (Hesperidin): Buff amorphous powder (830 mg) with R_f 0.5 (S3). λ_{max} (MeOH) nm: 283, 324 (sh.); (+NaOMe): 242, 287, 361; (+ AlCl₃): 306, 380 (sh); (+ AlCl₃+ HCl): 305, 381; (+ NaOAc): 283, 325 (sh); (+ NaOAc+Boric acid): 283, 325 (sh). EI-MS: *m/z* (relative abundance %): 302 [M⁺ - 308] (28.48), 301 (12.93), 286 (10.41), 285 (8.95), 271 (4.89), 259 (3.66), 179 (32.23), 165 (10.65), 152 (13.76), 150 (64.78), 137 (100), 135 (64.61), 129 (15.72), 124 (24.51), 111 (10.85), 107 (20.83), 84 (37.13), 78 (10.31), 77 (21.76), 73 (32.65), 71 (39.32), 69 (41.87), 60 (42.58), 57 (32.52) and 43 (40.68). ¹H-NMR (400 MHz, DMSO-*d*₆): δ ppm 5.50 (1H, *dd*, *J*=4, 12.4, H-2), 3.27 (1H, *m*, H-3a), 2.78 (1H, *dd*, *J*= 2.6, 17.2, H-3b), 6.12 (1H, *d*, *J*=1.2, H-6), 6.14 (1H, *d*, *J*=1.2, H-8), 6.94 (1H, *d*, *J*=1.6, H-2'), 6.96 (1H, *d*, *J*=8, H-5'), 6.92 (1H, *dd*, *J*=1.6, 11.6, H-6'), 4.97 (1H, *d*, *J*=6.8, H-1''), 4.52 (1H, *br.s*, H-1'''), 3.14- 3.63 (10 H, *m*, sugar protons), 1.08 (3H, *d*, *J*=5.6, CH₃), 12.02 (1H, *s*, 5-OH), 9.1 (1H, *s*, 3□-OH) and 3.77 (3H, *s*, O-CH₃). ¹³C-NMR (100 MHz, DMSO-*d*₆): δ ppm 78.38 (C-2), 42.02 (C-3), 197.04 (C-4), 163.04 (C-5), 96.38 (C-6), 165.14 (C-7), 95.55 (C-8), 162.50 (C-9), 103.32

(C-10), 130.90 (C-1'), 114.15 (C-2'), 146.46 (C-3'), 147.97 (C-4'), 112.03 (C-5'), 117.97 (C-6'), 99.45 (C-1''), 70.27 (C-2''), 75.52 (C-3''), 68.32 (C-4''), 76.27 (C-5''), 66.02 (C-6''), 100.60 (C-1'''), 69.60 (C-2'''), 70.27 (C-3'''), 72.99 (C-4'''), 75.52 (C-5''') 17.84 (CH₃) and 55.69 (O-CH₃).

Hesperidin was identified by comparison their spectral data with the available literature and Co-TLC with authentic sample.¹

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