Isolation of Taste-Active Triterpenoids from *Quercus robur*, Sensory Assessment and Identification in Wines and Spirit

Marine Gammacurta,[†] Pierre Waffo-Teguo,[†] Delphine Winstel,[†] Denis Dubourdieu,[†] and Axel Marchal[*,†]

[†] Univ. Bordeaux, Unité de recherche Œnologie, EA 4577, USC 1366 INRA, ISVV, 33882 Villenave d'Ornon cedex, France

SUPPORTING INFORMATION

Table S1. ^{1}H NMR (600MHz, CD_3OD) spectroscopic data for compounds **7**−**13**

Table S2. ^{13}C NMR (150MHz) spectroscopic data for compounds **7**−**13**

S1. HRMS (top) and HCD-HRMS (bottom) spectra of compound **1**

S2. ^{1}H NMR (600MHz, CD_3OD) (top), 2D COSY (CD_3OD), ^{1}H (600MHz) (middle), and 2D ROESY (CD_3OD), ^{1}H (600MHz) (bottom) spectra of compound **1**

S3. 2D HSQC (CD_3OD), ^{1}H (600MHz) and ^{13}C (150MHz) (top) and 2D HMBC (CD_3OD), ^{1}H (600MHz) and ^{13}C (150MHz) (bottom) spectra of compound **1**

S4. HRMS (top) and HCD-HRMS (bottom) spectra of compound **2**

S5. ^{1}H NMR (600MHz, CD_3OD) (top), 2D COSY (CD_3OD), ^{1}H (600MHz) (middle), and 2D ROESY (CD_3OD), ^{1}H (600MHz) (bottom) spectra of compound **2**

S6. 2D HSQC (CD_3OD), ^{1}H (600MHz) and ^{13}C (150MHz) (top) and 2D HMBC (CD_3OD), ^{1}H (600MHz) and ^{13}C (150MHz) (bottom) spectra of compound **2**

S7. HRMS (top) and HCD-HRMS (bottom) spectra of compound **3**

S8. ^{1}H NMR (600MHz, CD_3OD) (top), 2D COSY (CD_3OD), ^{1}H (600MHz) (middle), and 2D ROESY (CD_3OD), ^{1}H (600MHz) (bottom) spectra of compound **3**

S9. 2D HSQC (CD_3OD), ^{1}H (600MHz) and ^{13}C (150MHz) (top) and 2D HMBC (CD_3OD), ^{1}H (600MHz) and ^{13}C (150MHz) (bottom) spectra of compound **3**

S10. HRMS (top) and HCD-HRMS (bottom) spectra of compound **4**

S11. ^{1}H NMR (600MHz, CD_3OD) (top), 2D COSY (CD_3OD), ^{1}H (600MHz) (middle), and 2D ROESY (CD_3OD), ^{1}H (600MHz) (bottom) spectra of compound **4**

S12. 2D HSQC (CD_3OD), ^{1}H (600MHz) and ^{13}C (150MHz) (top) and 2D HMBC (CD_3OD), ^{1}H (600MHz) and ^{13}C (150MHz) (bottom) spectra of compound **4**

S13. HRMS (top) and HCD-HRMS (bottom) spectra of compound **5**

S14. ^{1}H NMR (600MHz, CD_3OD) (top), 2D COSY (CD_3OD), ^{1}H (600MHz) (middle), and 2D ROESY (CD_3OD), ^{1}H (600MHz) (bottom) spectra of compound **5**

S15. 2D HSQC (CD_3OD), ^{1}H (600MHz) and ^{13}C (150MHz) (top) and 2D HMBC (CD_3OD), ^{1}H (600MHz) and ^{13}C (150MHz) (bottom) spectra of compound **5**

S16. HRMS (top) and HCD-HRMS (bottom) spectra of compound **6**

S17. ^{1}H NMR (600MHz, CD_3OD) (top), 2D COSY (CD_3OD), ^{1}H (600MHz) (middle), and 2D ROESY (CD_3OD), ^{1}H (600MHz) (bottom) spectra of compound **6**

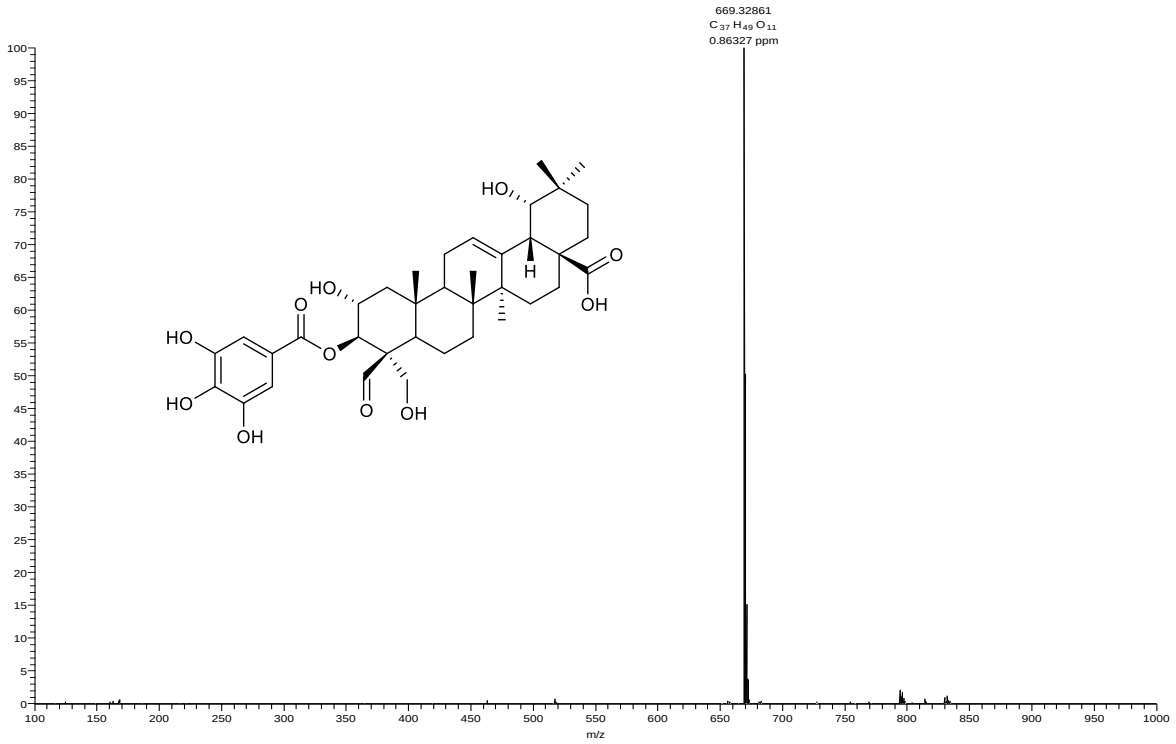
Table S1. ^{1}H NMR (600MHz, CD_3OD) spectroscopic data for compounds **7**−**13**

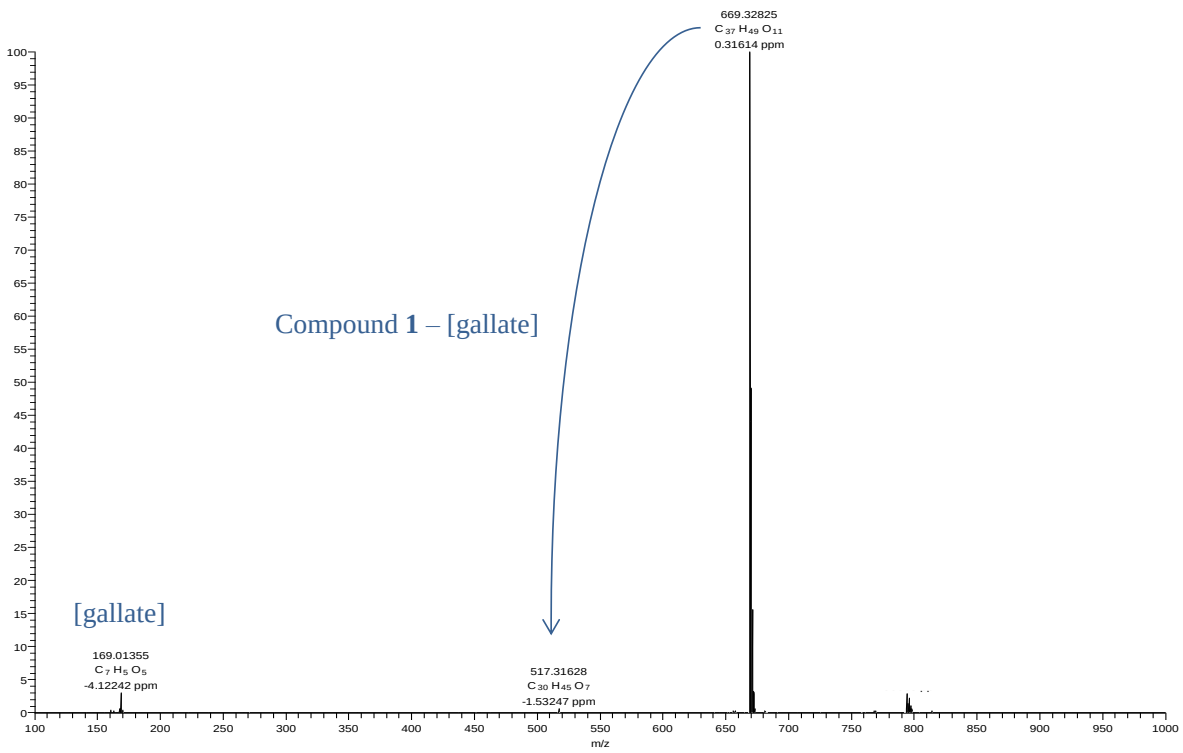
	7	**8**	**9**	**10**	**11**	**12**	**13**
position	*δH (J in Hz)*	δ_H (*J* in Hz)	δ_H (*J* in Hz)	δ_H (*J* in Hz)	δ_H (*J* in Hz)	δ_H (*J* in Hz)	δ_H (*J* in Hz)
1*α*	1.26 m	0.92 m	0.90 m	1.07 m	1.11 m	1.08 m	1.11 m
1*β*	1.63 brd	2.00 brd	1.98 brd	2.10 m	2.15 dd (12.3, 5.2)	2.09 dd (12.6, 4.9)	2.13 dd (12.4, 5.1)
2	4.15 td (12.1, 3.4)	4.10 td (10.5, 4.8)	4.23 td (10.7, 4.8)	4.20 td (10.7, 4.4)	5.69 td (10.5, 4.9)	4.61 dd (9.9, 4.9)	5.67 td (10.8, 4.9)
3	3.96 d (2.5)	2.90 d (9.8)	2.99 d (9.7)	3.66 d (9.7)	3.23 d (10)	4.75 d (9.9)	3.24 d (10.0)
4							
5	1.46 m	1.05 m	1.16 m	1.60 m	1.11 m	1.24 m	1.14 brd
6*α*	1.72 m	1.51 m	1.78 m	1.31 m	1.48 m	1.71 m	1.50 m
6*β*	1.87 m	1.81 m	1.82 m	1.67 m	1.85 m	1.85 m	1.85 m
7*α*	1.35 m	1.36 m	1.38 m	1.26 m	1.40 m	1.39 m	1.36 m
7*β*	1.53 m	1.52 m	1.47 m	1.37 m	1.54 m	1.53 td (13.2, 3.5)	1.51 m
8							
9	1.87 m	1.78 m	1.98 m	1.90 m	1.82 m	1.84 m	1.80 m
10							
11*α*	1.95 m	1.96 m	1.99 m	1.97 m	1.97 m	2.04 m	1.94 m
11*β*	2.02 m	2.02 m	2.02 m	2.02 m	2.04 m		2.0 m
12	5.35 t (3.6)	5.35 t (3.9)	5.34 t (3.5)	5.32 brd	5.35 t (3.1)	5.36 t (3.3)	5.33 t (3.4)
13							
14							
15*α*	1.03 m	1.03 m	1.02 m	0.97 m	1.05 m	1.04 m	1.04 m
15*β*	1.77 m	1.77 m	1.76 m	1.58 m	1.65 m	1.69 m	1.64 m
16*α*	1.64 m	1.63 m	1.68 m	1.57 m	1.64 m	1.75 m	1.72 m
16*β*	2.30 m	2.30 td (13.5, 3.5)	2.34 brd	2.24 brd	2.32 brd	2.35 td (13.5, 3.5)	2.33 td (13.0, 4.0)
17							
18	3.07 m	3.07 m	3.08 d (3.5)	3.09 brd	3.08 brd	3.08 d (3.7)	3.06 m
19	3.27 m	3.27 d (3.5)	3.27 d (3.5)	3.27 d (3.7)	3.27 d (3.7)	3.29 d (3.7)	3.27 d (3.9)
20							
21*α*	1.05 m	1.00 m	1.02 m	1.00 m	1.04 m	1.06 m	1.02 m
21*β*	1.78 m	1.76 m	1.72 m	1.75 m	1.78 m	1.78 m	1.80 m
22*α*	1.65 m	1.62 m	1.65 m	1.60 m	1.65 m	1.69 m	1.66 m
22*β*	1.78 m	1.76 m	1.79 m	1.75 m	1.77 m	1.79 m	1.79 m
23*α*		1.45 s		4.33 d (11.1)	1.47 s	1.25 s	1.46 s
23*β*				4.80 d (11.1)			
24	1.33 s		1.52 s	10.0 s			
25	0.88 s	0.96 s	0.97 s	0.97 s	1.10 s	1.04 s	1.05 s
26	0.79 s	0.80 s	0.78 s	0.78 s	0.82 s	0.78 s	0.79 s
27	1.35 s	1.32 s	1.33 s	1.19 s	1.35 s	1.34 s	1.30 s
28							
29	0.98 s	0.98 s	0.94 s	0.97 s	0.96 s	0.96 s	0.94 s
30	0.95 s	0.95 s	0.97 s	0.99 s	0.98 s	0.97 s	0.95 s
1′			5.39 d (8.1)			5.39 d (8.2)	5.39 d (7.8)
2′			3.33 m			3.33 m	3.32 m
3′			3.41 m			3.41 m	3.40 m
4′			3.35 m			3.36 m	3.34 m
5′			3.32 m			3.36 m	3.35 m
6′*α*			3.69 brd			3.69 dd (12.2,4.1)	3.68 dd (12.1, 4.6)
6′*β*			3.81 brd			3.84 m	3.82 m
1‴							
2‴, 6‴				7.09 s	7.10 s	7.13 s	7.09 s
3‴, 5‴							
4‴							
7‴							

Table S2. ^{13}C NMR (150MHz) spectroscopic data for compounds **7**−**13**

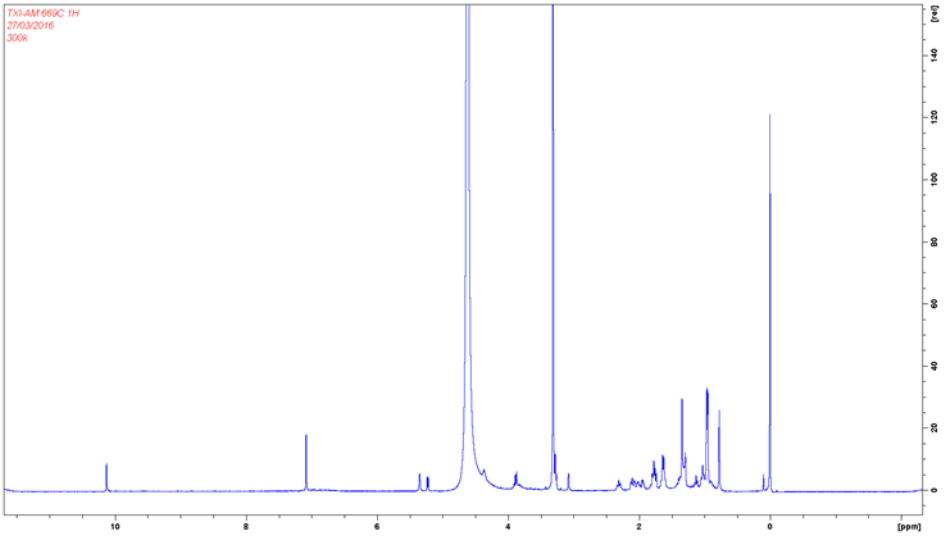
	7	**8**	**9**	**10**	**11**	**12**	**13**
position	δ_C, type	δ_C, type	δ_C, type	δ_C, type	δ_C, type	δ_C, type	δ_C, type
1*α*	41.6, CH_2	46.8, CH_2	46.7, CH_2	47.5, CH_2	44.2, CH_2	47.4, CH_2	43.7, CH_2
1*β*							
2	65.8, CH	67.8, CH	67.5, CH	69.7, CH	72.4, CH	65.6, CH	71.4, CH
3	73.7, CH	82.9, CH	83.2, CH	77.1, CH	80.2, CH	82.6, CH	80.6, CH
4	50.6, C	49.3, C	51.4, C	59.8, C	49.8, C	48.9, C	50.0, C
5	48.0, CH	56.1, CH	56.6, CH	51.8, CH	56.1, CH	55.5, CH	55.5, CH
6*α*	19.6, CH_2	20.1, CH_2	20.6, CH_2	20.2, CH_2	23.6, CH_2	20.0, CH_2	22.7, CH_2
6*β*							
7*α*	32.6, CH_2	32.7, CH_2	32.6, CH_2	33.6, CH_2	32.8, CH_2	32.9, CH_2	31.9, CH_2
7*β*							
8	39.1, C	38.8, C	39.3, C	40.9, C	38.9, C	39.2, C	38.9, C
9	46.8, CH	47.4, CH	46.8, CH	48.8, CH	47.3, CH	47.5, CH	47.2, CH
10	38.6, C	38.3, C	38.3, C	39.5, C	38.4, C	38.3, C	37.5, C
11*α*	23.5, CH_2	23.5, CH_2	23.3, CH_2	25.5, CH_2	23.5, CH_2	23.5, CH_2	22.8, CH_2
11*β*							
12	124.0, CH	123.2, CH	123.5, CH	124.8, CH	123.5, CH	123.2, CH	123.0, CH
13	143.4, C	143.2, C	142.9, C	144.9, C	143.2, C	143.3, C	142.2, C
14	41.9, C	41.3, C	40.6, C	43.1, C	41.3, C	40.7, C	40.2, C
15*α*	28.4, CH_2	28.2, CH_2	28.3, CH_2	29.6, CH_2	28.1, CH_2	27.8, CH_2	27.3, CH_2
15*β*							
16*α*	27.3, CH_2	27.3, CH_2	27.2, CH_2	29.0, CH_2	27.2, CH_2	26.8, CH_2	26.6, CH_2
16*β*							
17	45.1, C	45.3, C	45.8, C	46.9, C	45.2, C	45.5, C	45.6, C
18	43.9, CH	43.8, CH	43.3, CH	45.8, CH	44.0, CH	43.6, CH	43.0, CH
19	81.1, CH	80.8, CH	80.5, CH	82.9, CH	81.1, CH	80.9, CH	80.8, CH
20	34.7, C	34.5, C	34.9, C	36.5, C	34.6, C	34.5, C	34.4, C
21*α*	27.9, CH_2	28.1, CH_2	27.8, CH_2	29.7, CH_2	28.3, CH_2	28.0, CH_2	27.2, CH_2
21*β*							
22*α*	32.6, CH_2	32.7, CH_2	31.8, CH_2	34.1, CH_2	32.8, CH_2	31.7, CH_2	31.4, CH_2
22*β*							
23*α*	176.0, C	23.5, CH	175.7, C	62.7, CH_2	23.6, CH	23.1, CH	23.3, CH
23*β*							
24	23.6, CH	178.9, C	24.1, CH	205.9, C	180.1, C	175.8, C	179.2, C
25	13.3, CH	13.7, CH	13.6, CH	18.4, CH	13.5, CH	13.4, CH	13.0, CH
26	16.2, CH	16.1, CH	16.4, CH	18.2, CH	16.1, CH	16.0, CH	15.2, CH
27	29.1, CH	23.5, CH	23.2, CH	25.2, CH	23.6, CH	23.4, CH	22.7, CH
28	179.6, C	180.8, C	177.2, C	182.0, C	177.1, C	177.1, C	176.5, C
29	28.2, CH	27.5, CH	26.7, CH	29.1, CH	27.4, CH	27.1, CH	26.6, CH
30	23.7, CH	23.7, CH	23.3, CH	25.4, CH	23.9, CH	23.6, CH	23.1, CH
1′			94.1, CH			94.4, CH	94.0, CH
2′			72.2, CH			72.5, CH	72.2, CH
3′			76.8, CH			76.9, CH	76.5, CH
4′			69.0, CH			69.6, CH	69.3, CH
5′			76.6, CH			77.3, CH	76.8, CH
6′*α*			60.6, CH_2			60.9, CH_2	60.6, CH_2
6′*β*							
1‴				118.9, C	121.2, C	120.4, C	120.5, C
2‴, 6‴				110.4, CH	108.9, CH	109.0, CH	107.9, CH
3‴, 5‴				147.1, C	145.3, C	145.0, C	145.6, C
4‴				140.5, C	138.8, C	138.1, C	137.0, C
7‴				168.5, C	167.7, C	167.6, C	166.4, C

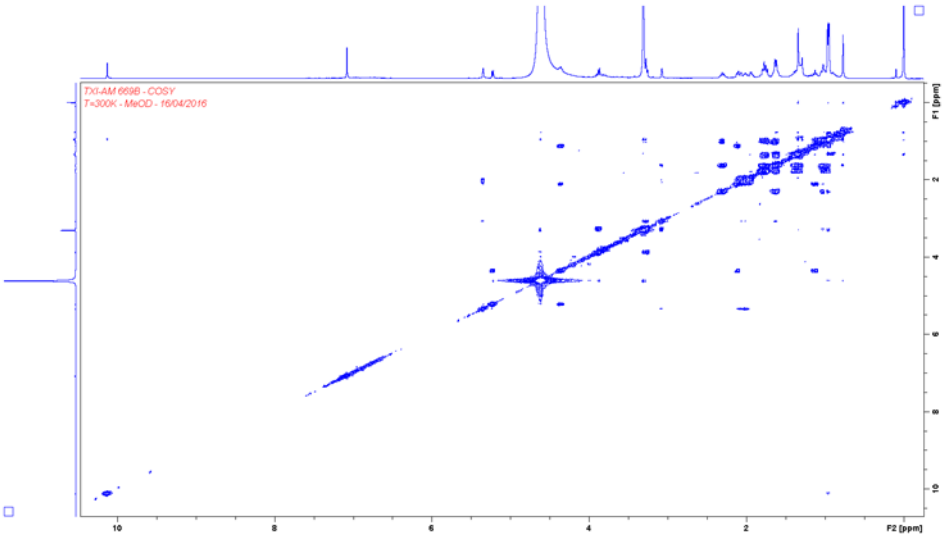
S1. HRMS (top) and HCD-HRMS (bottom) spectra of compound **1**

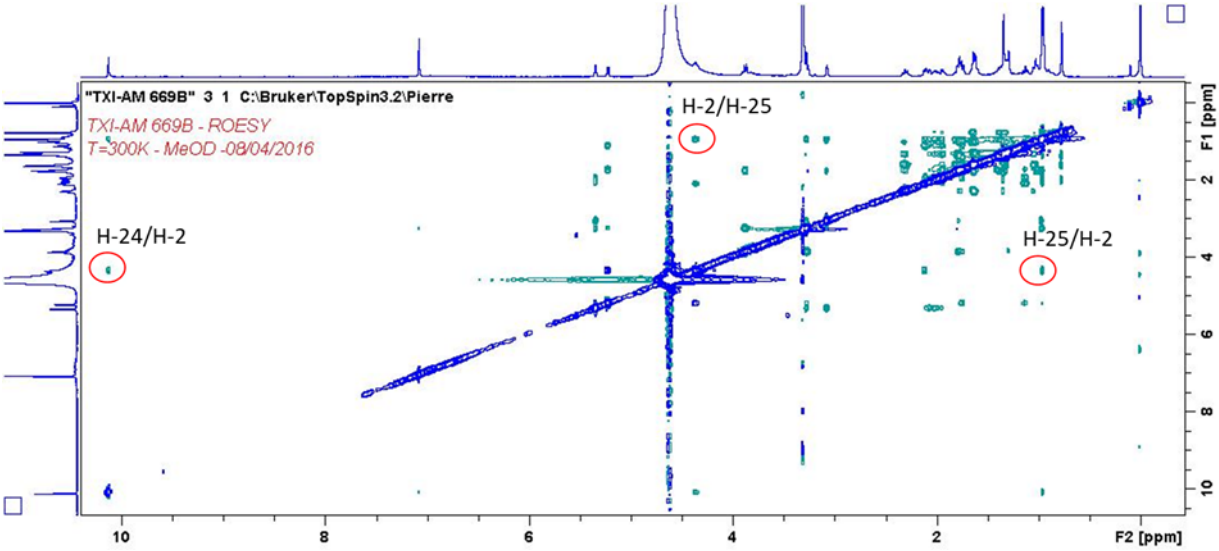




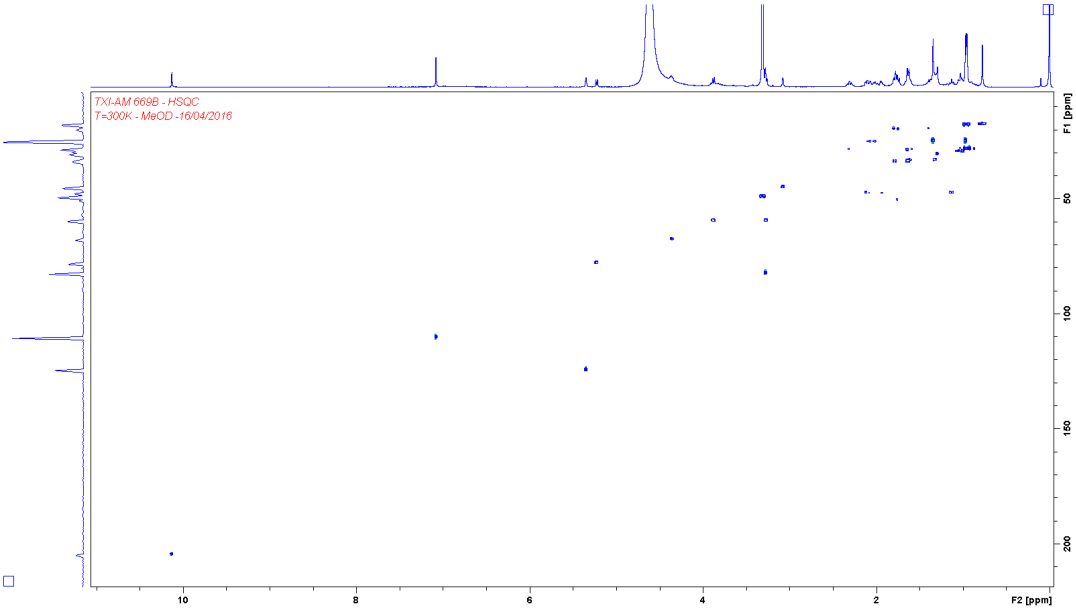
S2. ^{1}H NMR (600MHz, CD_3OD) (top), 2D COSY (CD_3OD), ^{1}H (600MHz) (middle), and 2D ROESY (CD_3OD), ^{1}H (600MHz) (bottom) spectra of compound **1**

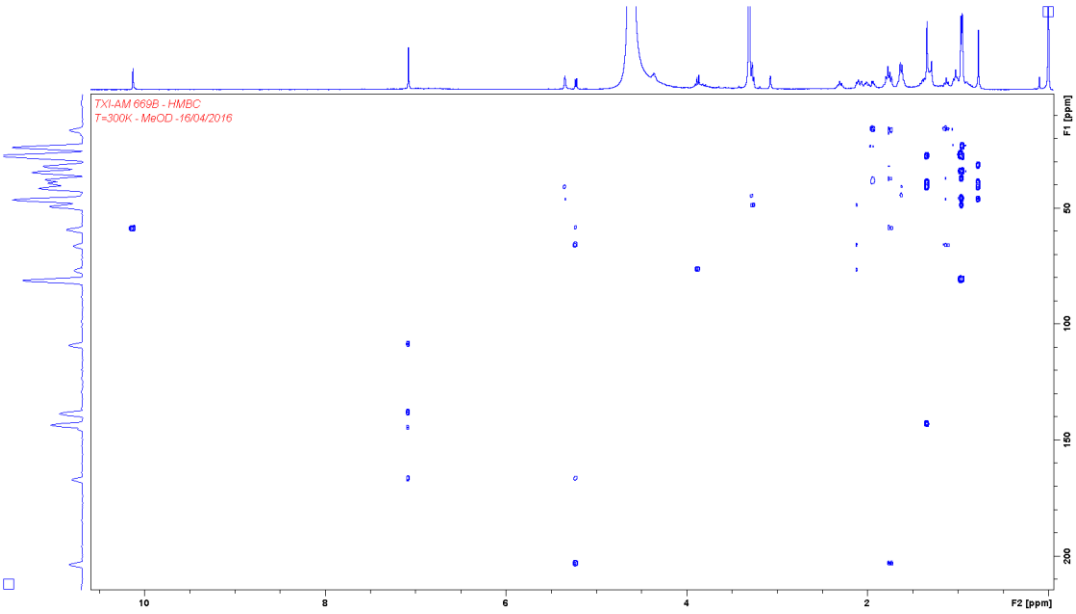




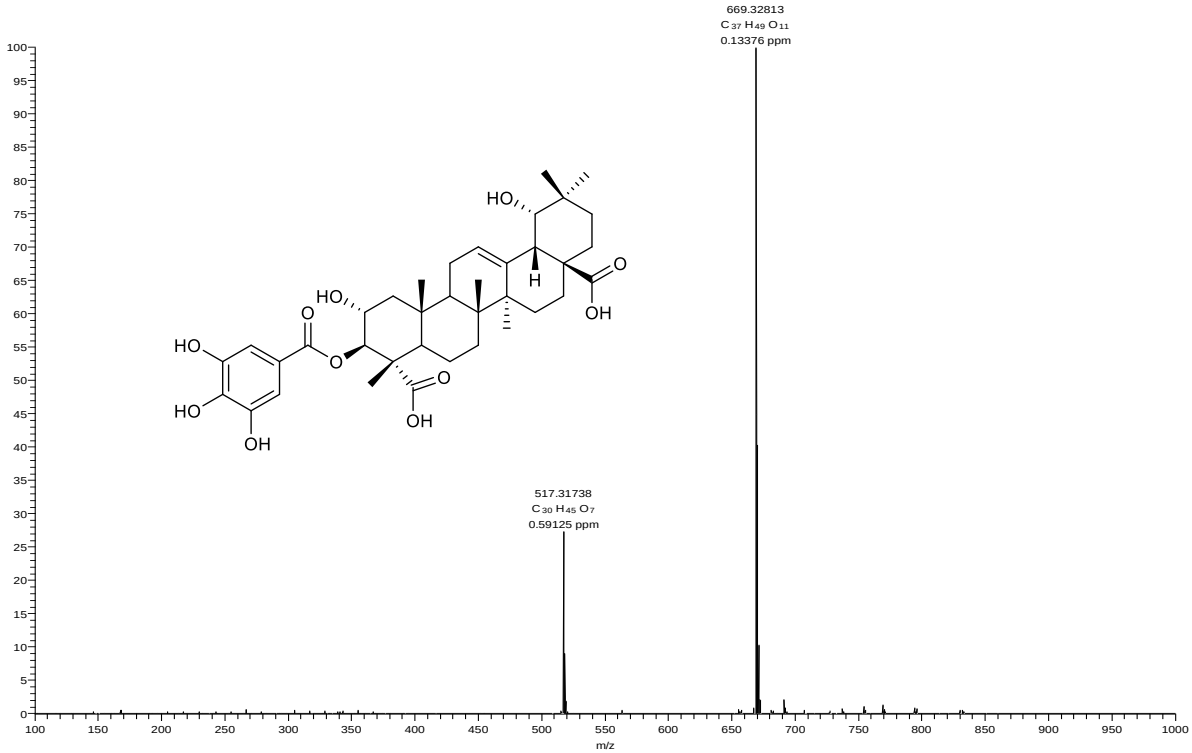


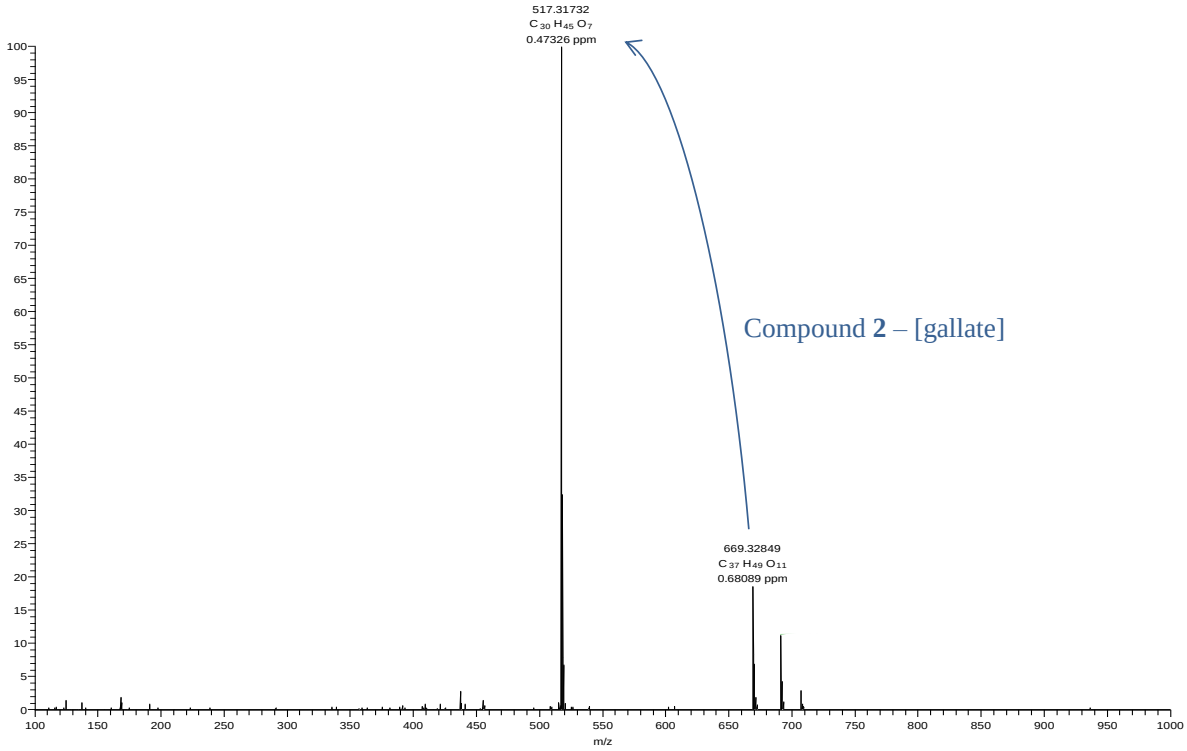
S3. 2D HSQC (CD_3OD), ^1H (600MHz) and ^{13}C (150MHz) (top) and 2D HMBC (CD_3OD), ^1H (600MHz) and ^{13}C (150MHz) (bottom) spectra of compound **1**



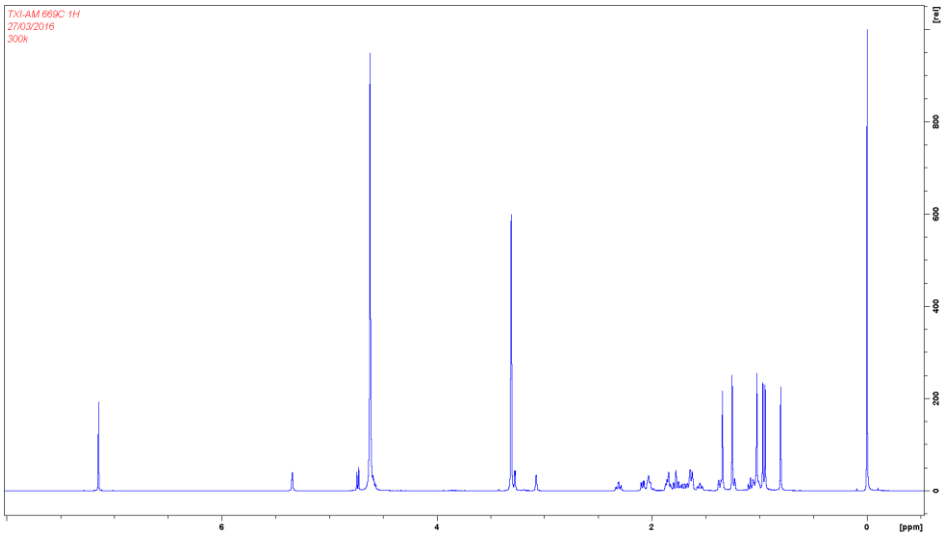


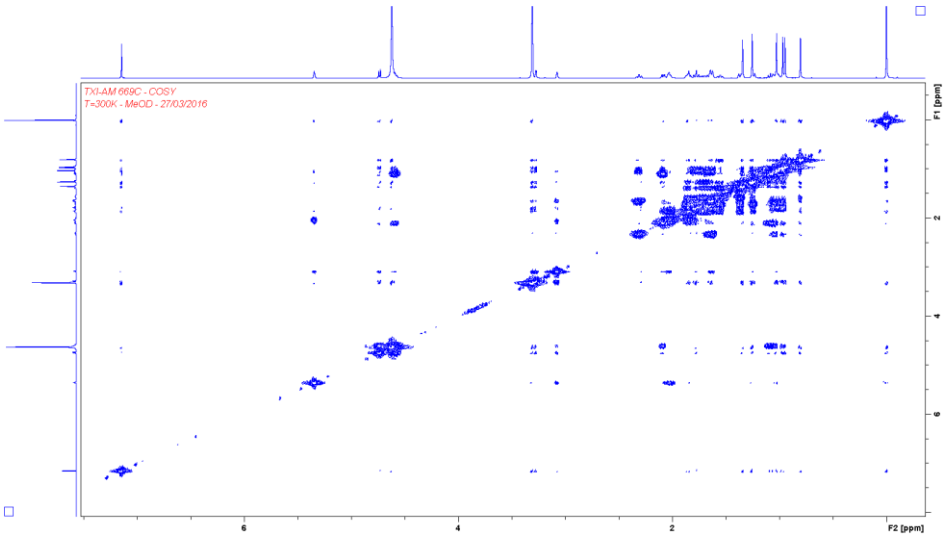
S4. HRMS (top) and HCD-HRMS (bottom) spectra of compound **2**

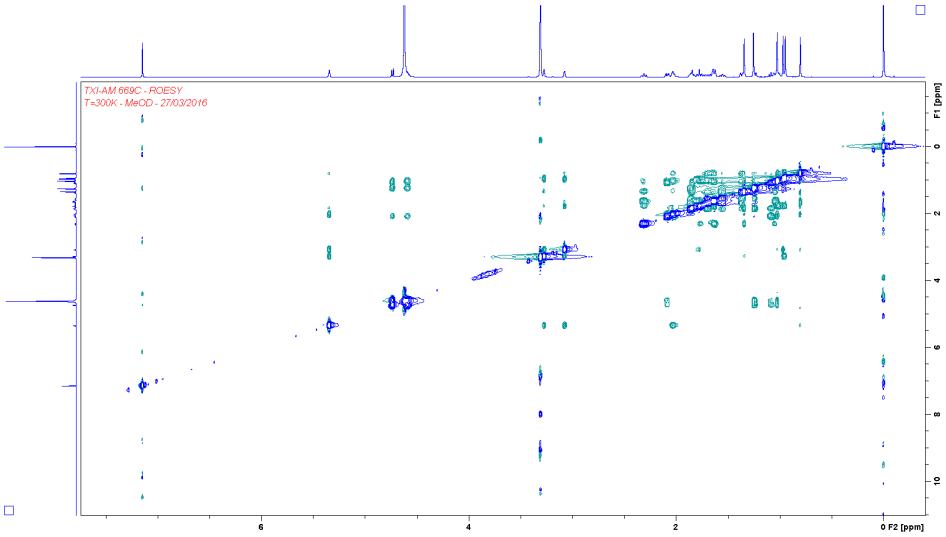




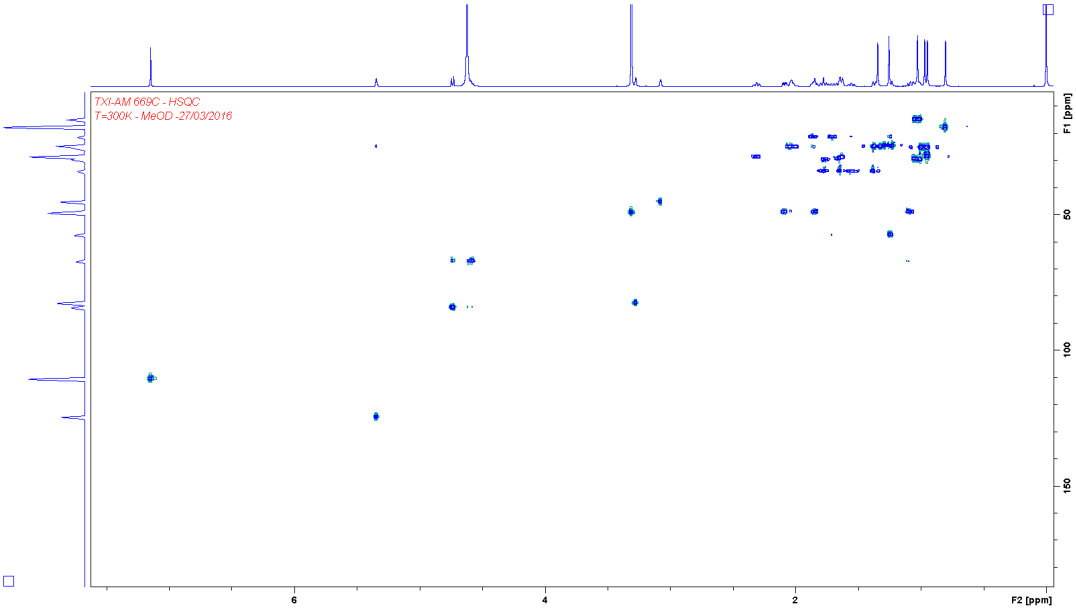
S5. ^{1}H NMR (600MHz, CD_3OD) (top), 2D COSY (CD_3OD), ^{1}H (600MHz) (middle), and 2D ROESY (CD_3OD), ^{1}H (600MHz) (bottom) spectra of compound **2**

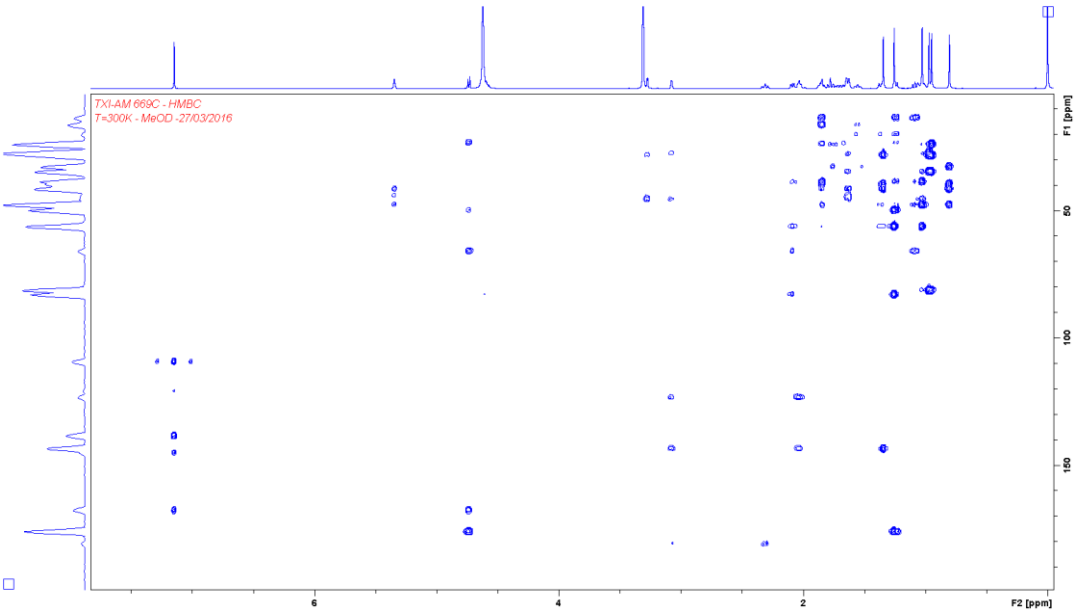




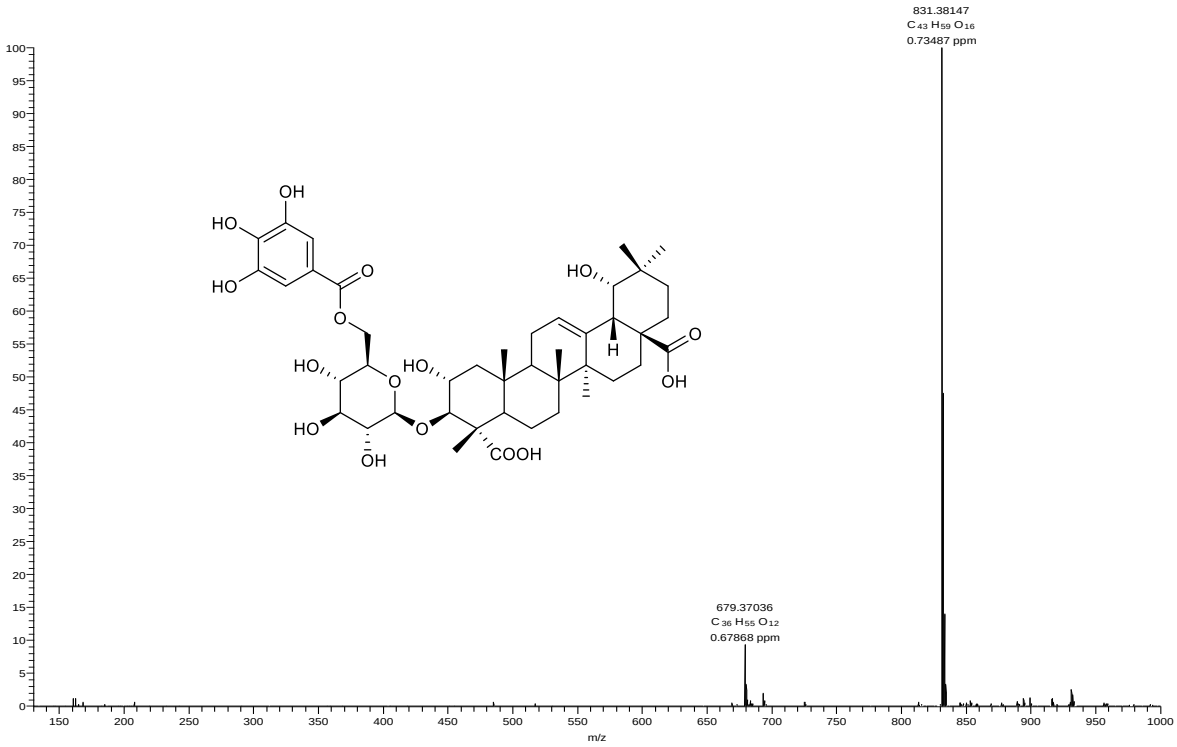


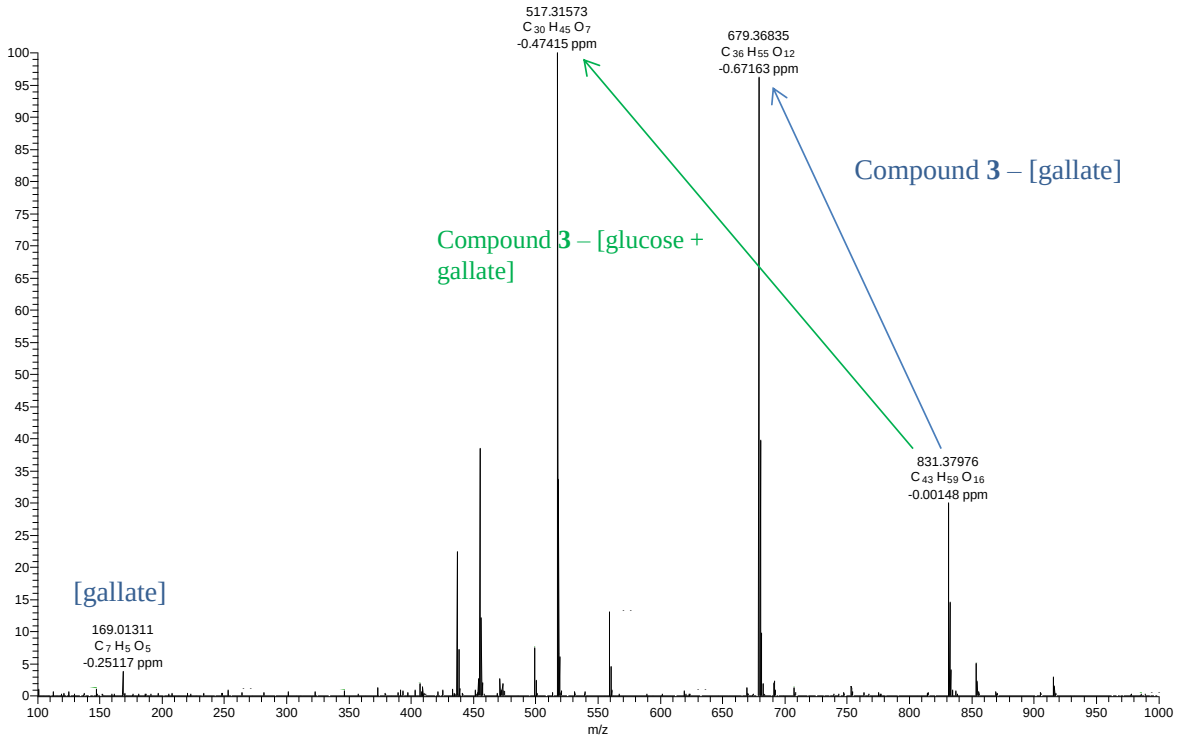
S6. 2D HSQC (CD_3OD), ^1H (600MHz) and ^{13}C (150MHz) (top) and 2D HMBC (CD_3OD), ^1H (600MHz) and ^{13}C (150MHz) (bottom) spectra of compound **2**



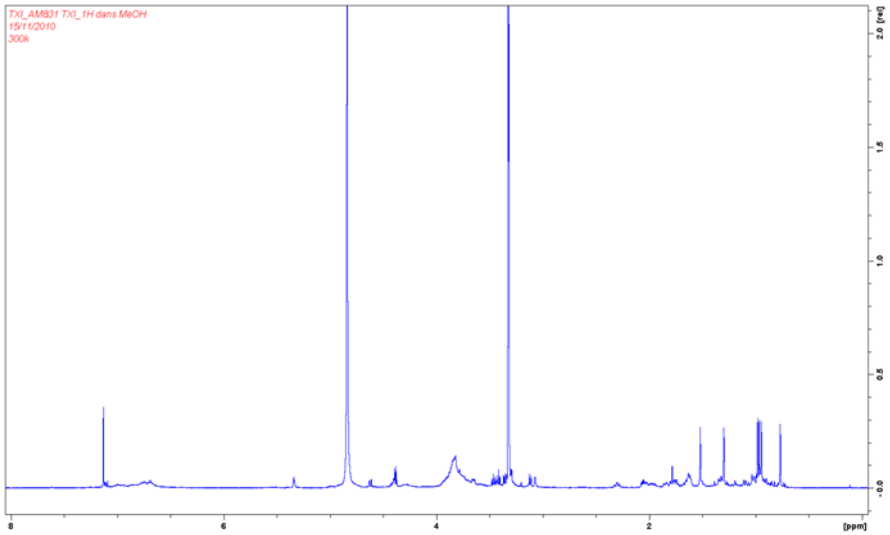


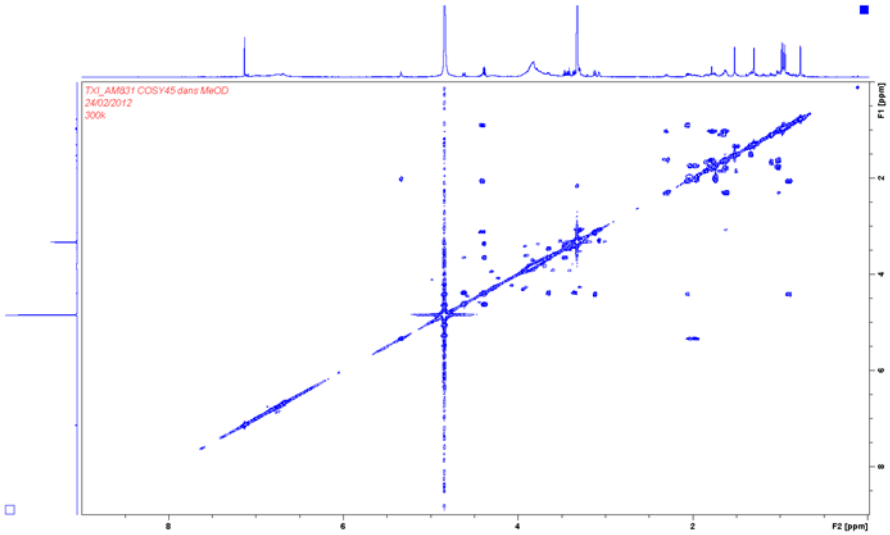
S7. HRMS (top) and HCD-HRMS (bottom) spectra of compound **3**

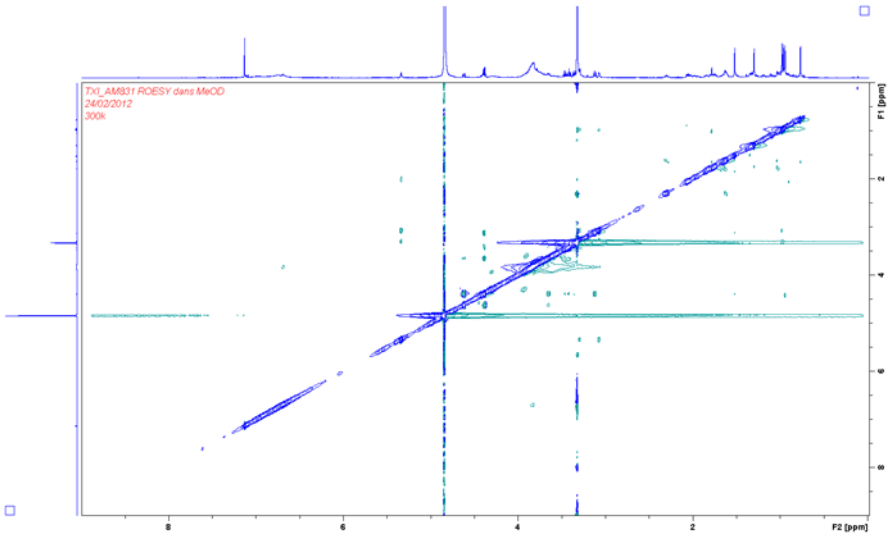




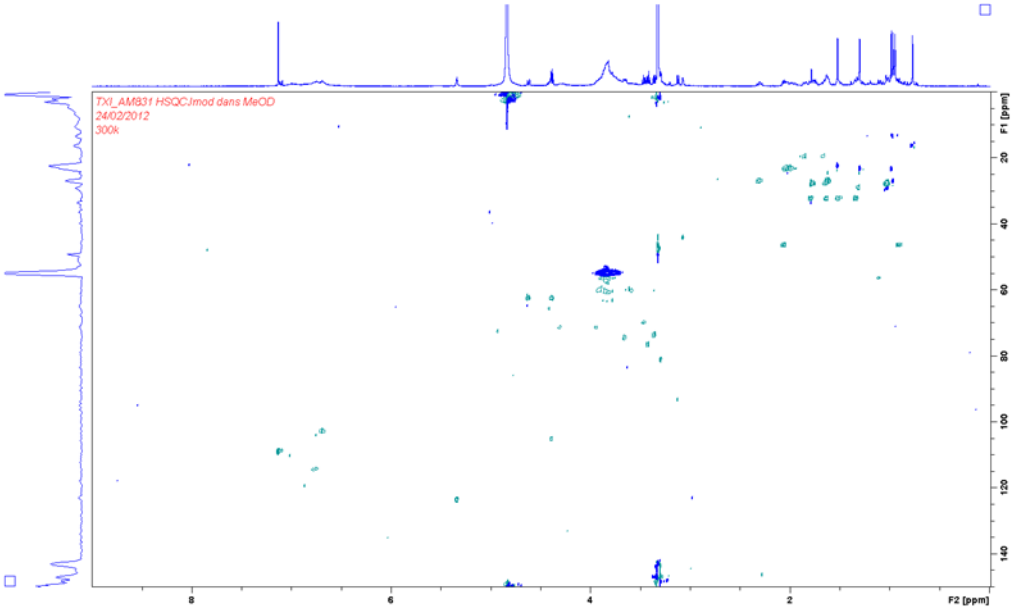
S8. ^{1}H NMR (600MHz, CD_3OD) (top), 2D COSY (CD_3OD), ^{1}H (600MHz) (middle), and 2D ROESY (CD_3OD), ^{1}H (600MHz) (bottom) spectra of compound **3**

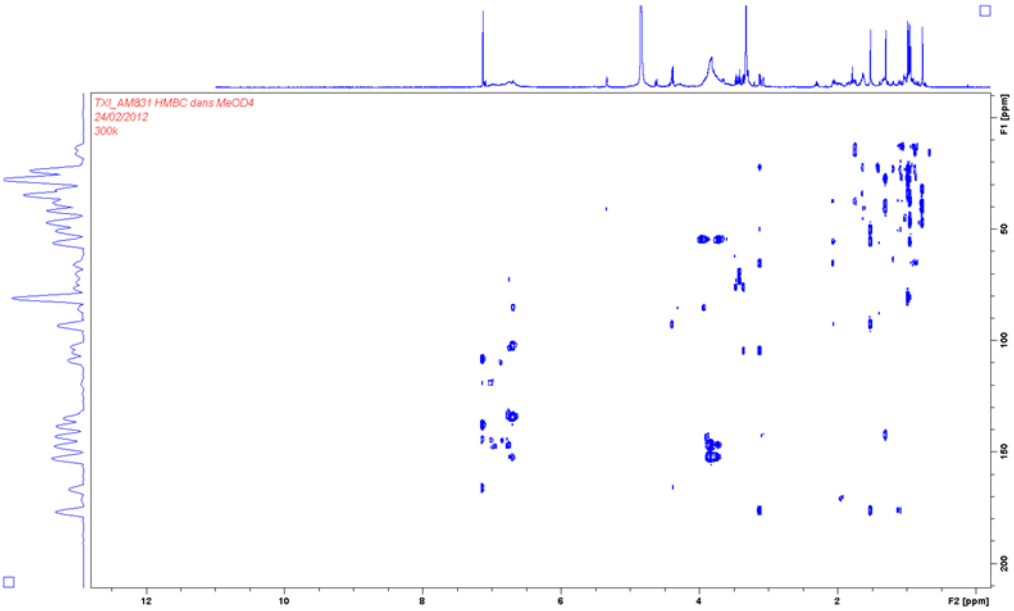




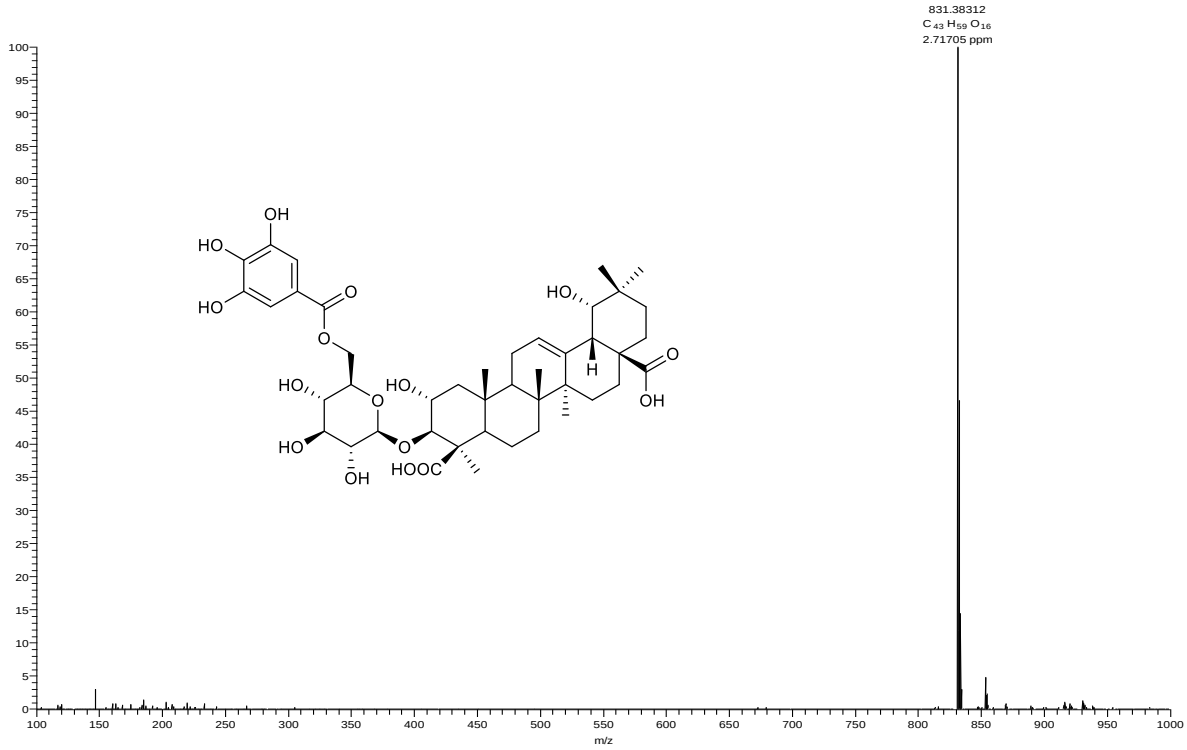


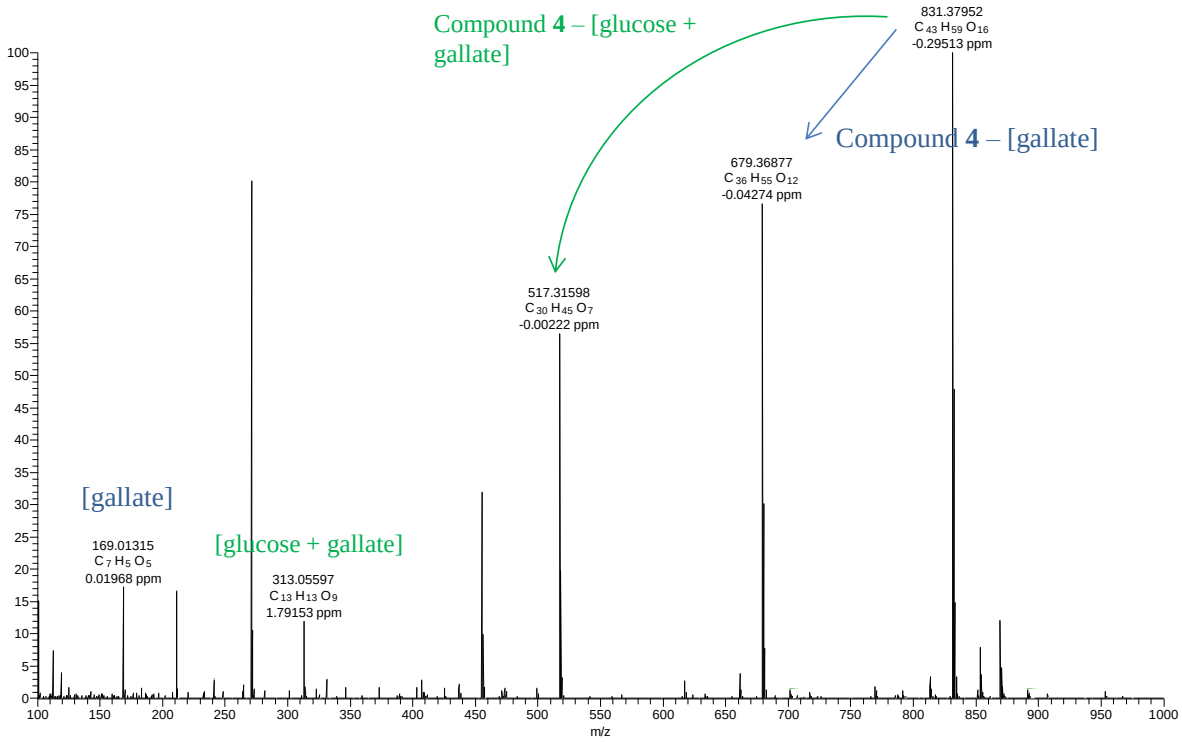
S9. 2D HSQC (CD_3OD), ^1H (600MHz) and ^{13}C (150MHz) (top) and 2D HMBC (CD_3OD), ^1H (600MHz) and ^{13}C (150MHz) (bottom) spectra of compound **3**



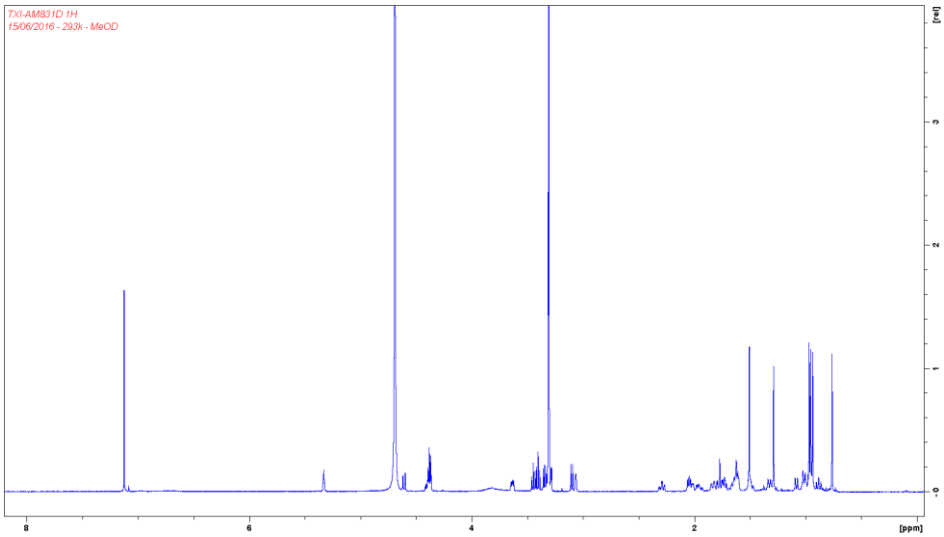


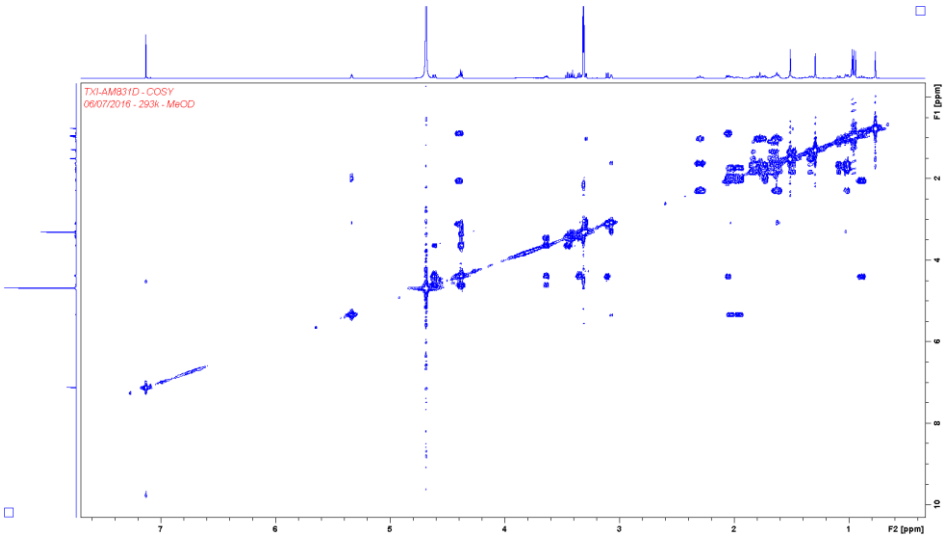
S10. HRMS (top) and HCD-HRMS (bottom) spectra of compound **4**

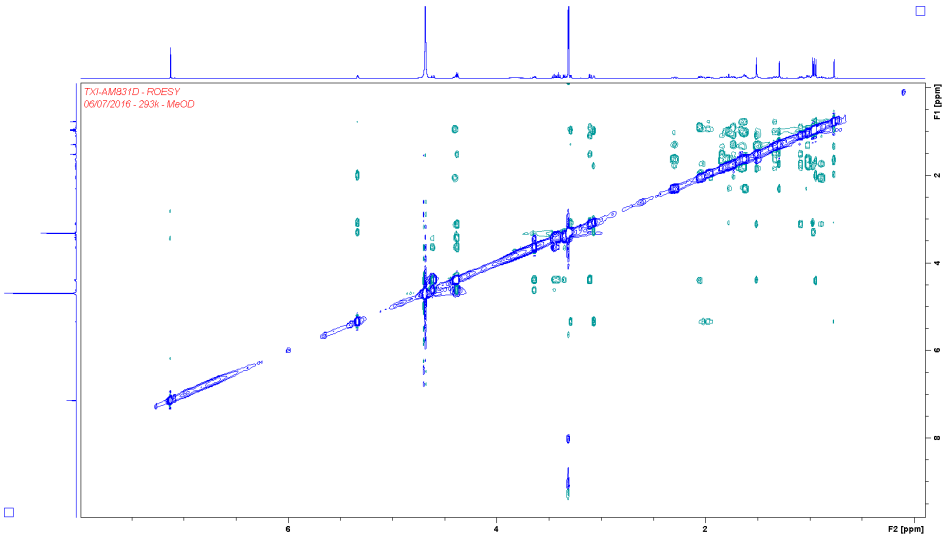




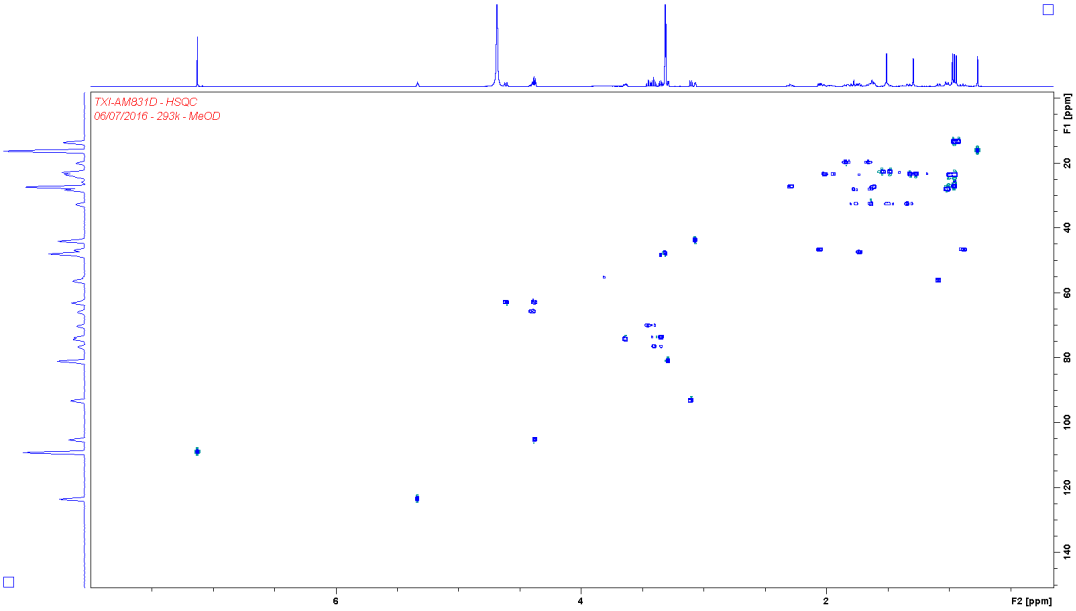
S11. ^{1}H NMR (600MHz, CD_3OD) (top), 2D COSY (CD_3OD), ^{1}H (600MHz) (middle), and 2D ROESY (CD_3OD), ^{1}H (600MHz) (bottom) spectra of compound **4**

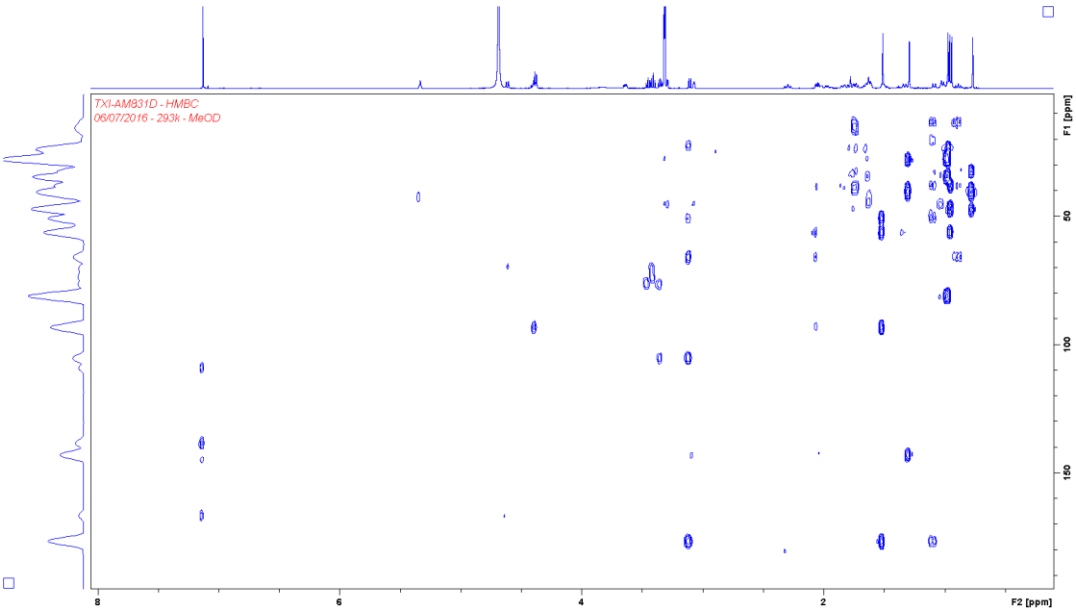




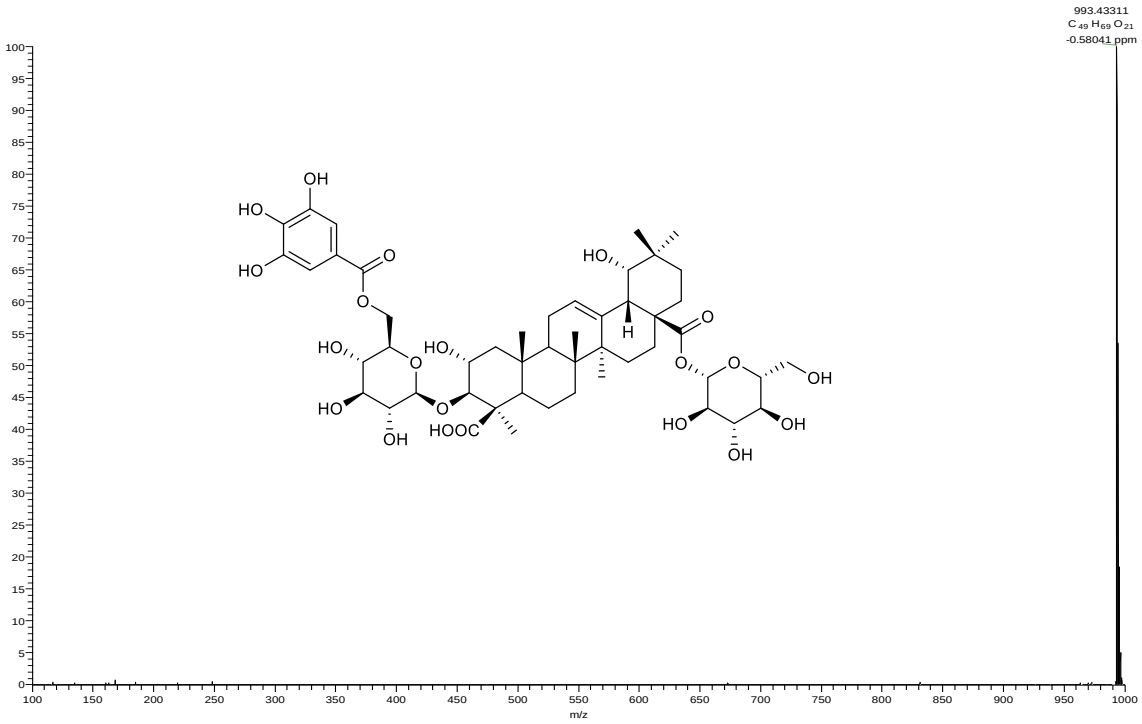


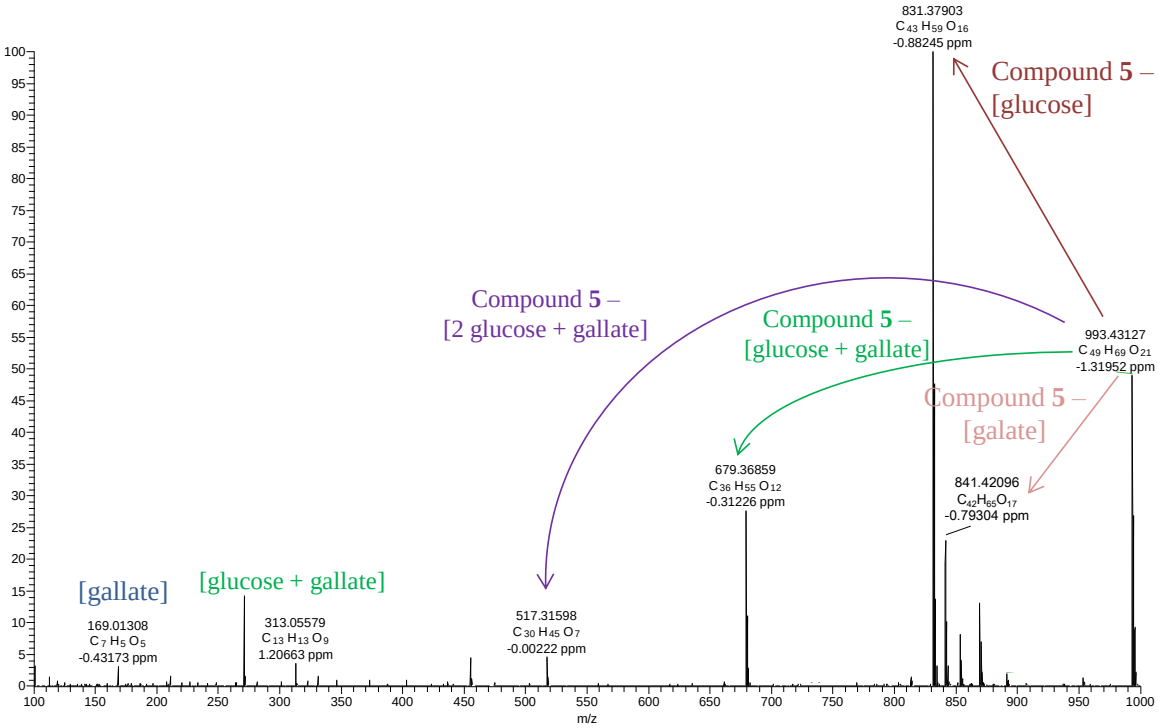
S12. 2D HSQC (CD_3OD), ^1H (600MHz) and ^{13}C (150MHz) (top) and 2D HMBC (CD_3OD), ^1H (600MHz) and ^{13}C (150MHz) (bottom) spectra of compound **4**



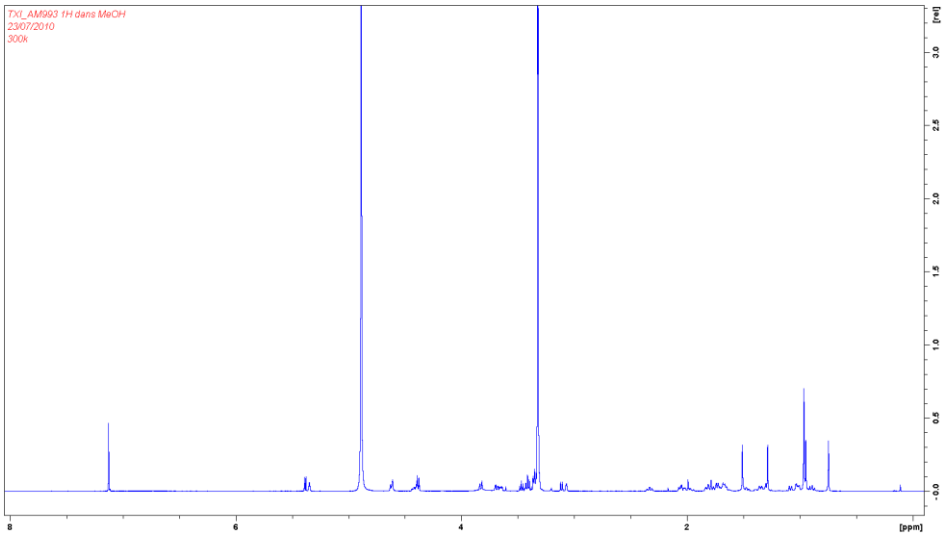


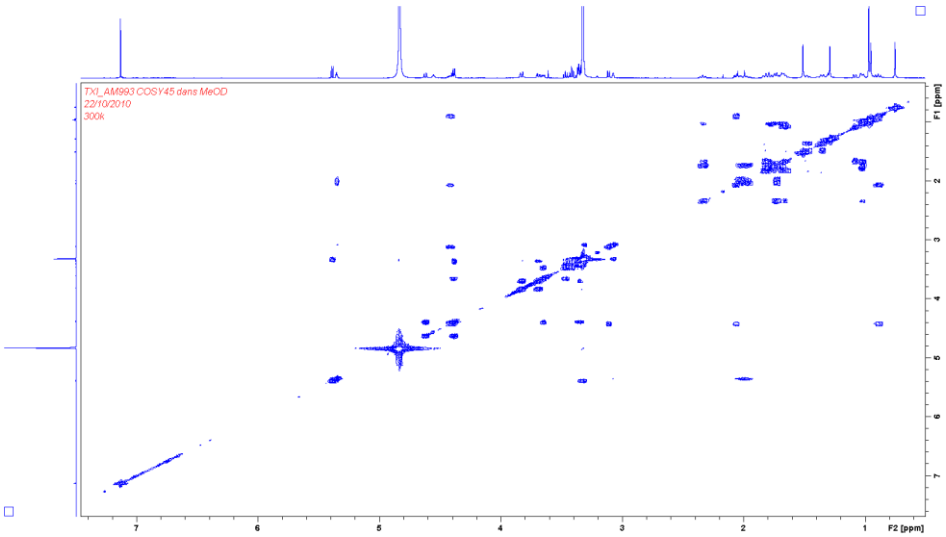
S13. HRMS (top) and HCD-HRMS (bottom) spectra of compound **5**

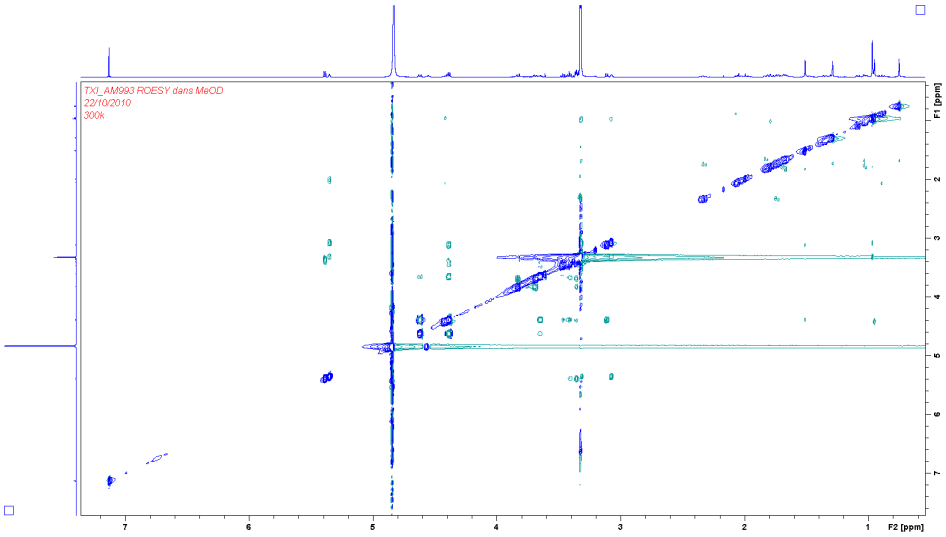




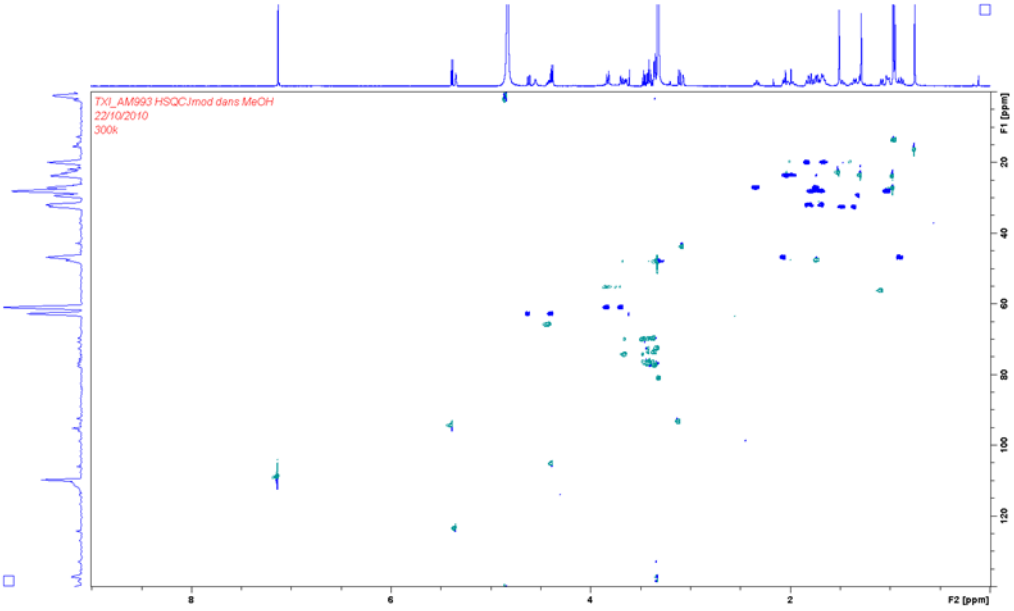
S14. ^{1}H NMR (600MHz, CD_3OD) (top), 2D COSY (CD_3OD), ^{1}H (600MHz) (middle), and 2D ROESY (CD_3OD), ^{1}H (600MHz) (bottom) spectra of compound **5**

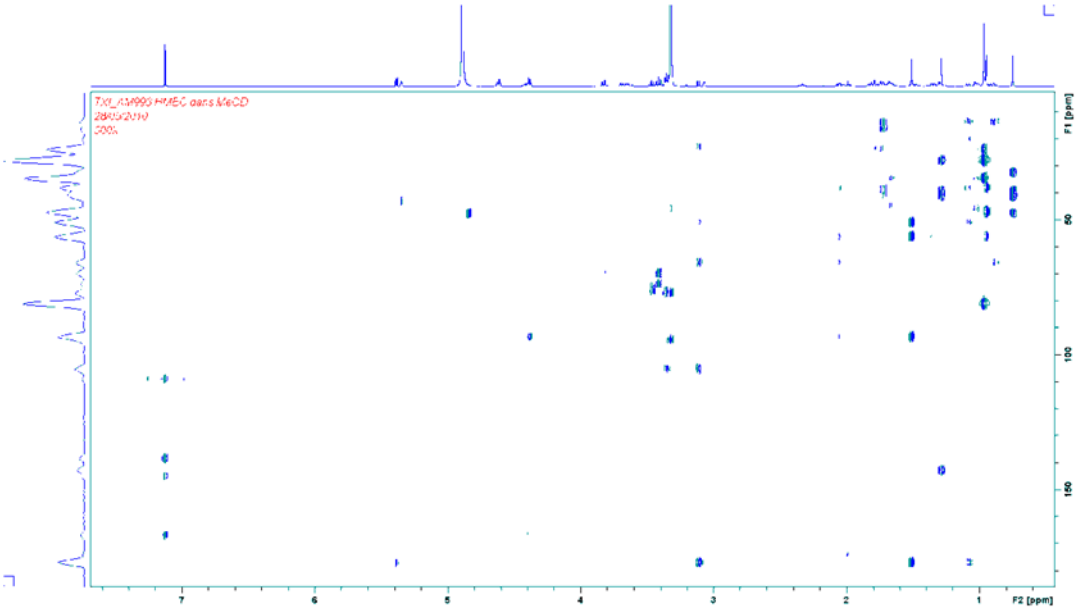




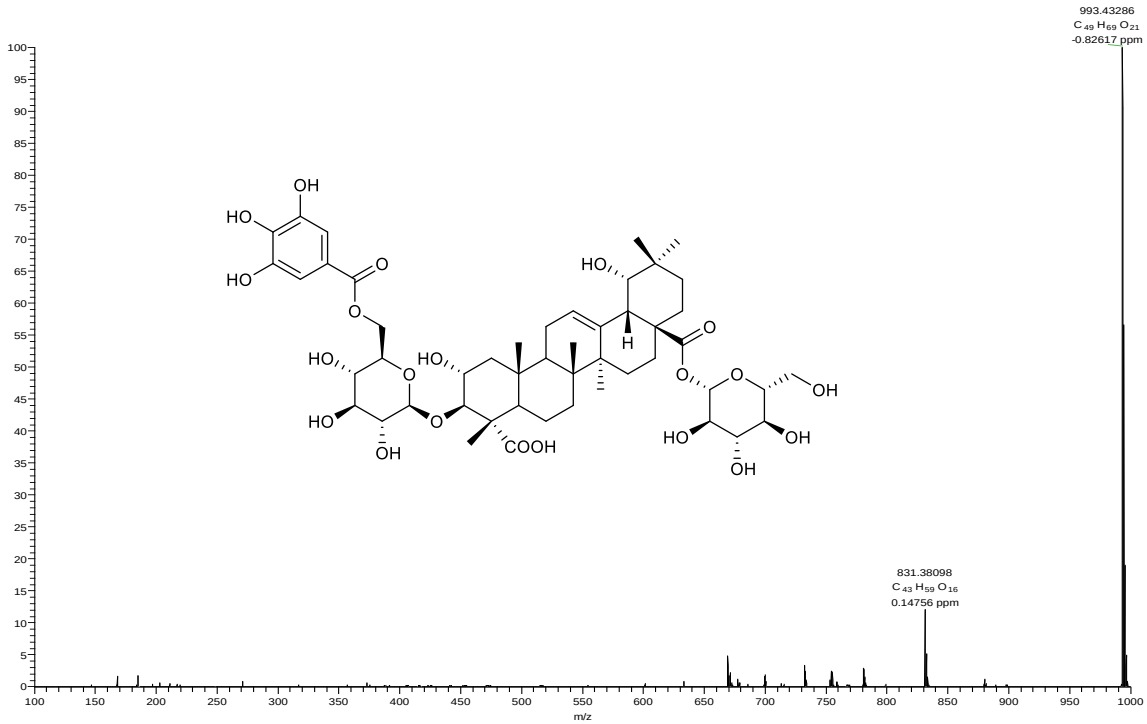


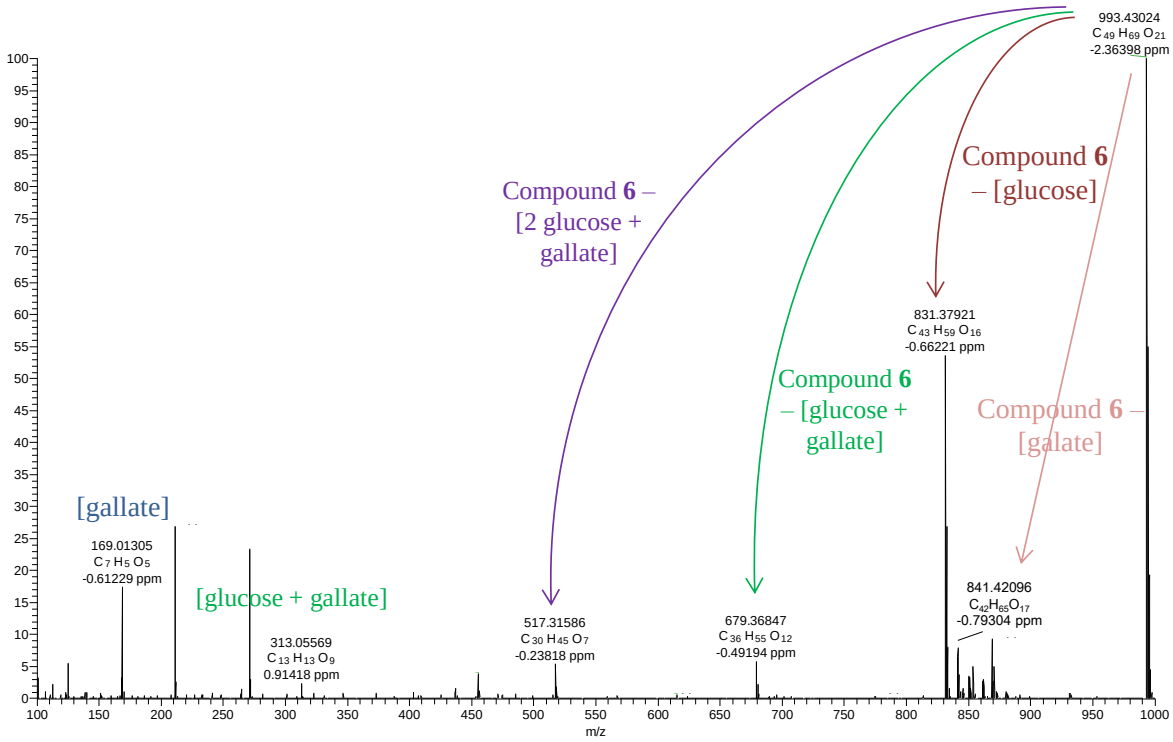
S15. 2D HSQC (CD_3OD), ^1H (600MHz) and ^{13}C (150MHz) (top) and 2D HMBC (CD_3OD), ^1H (600MHz) and ^{13}C (150MHz) (bottom) spectra of compound **5**



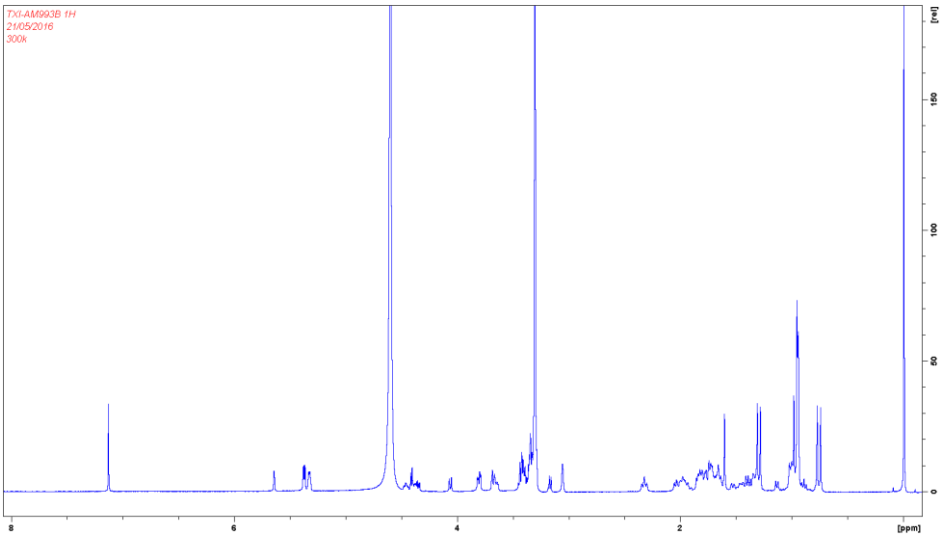


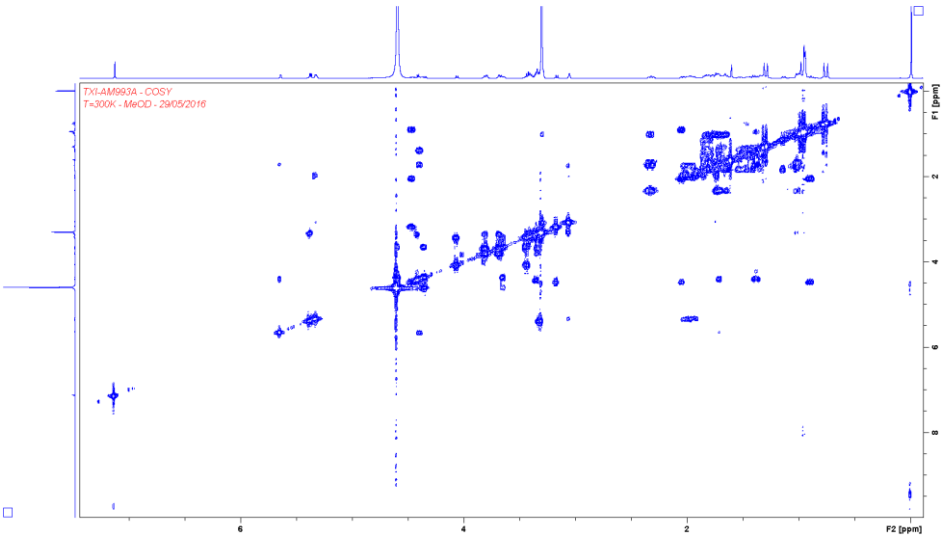
S16. HRMS (top) and HCD-HRMS (bottom) spectra of compound **6**

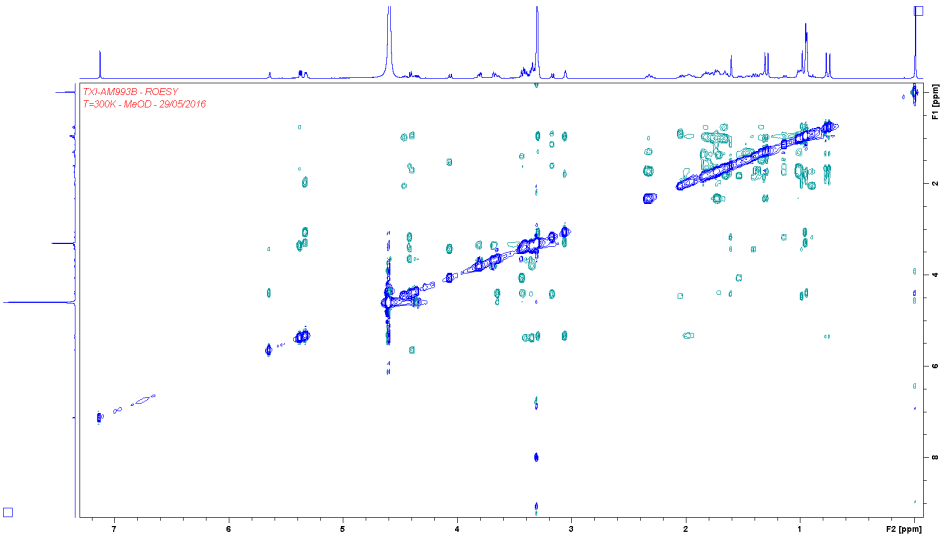
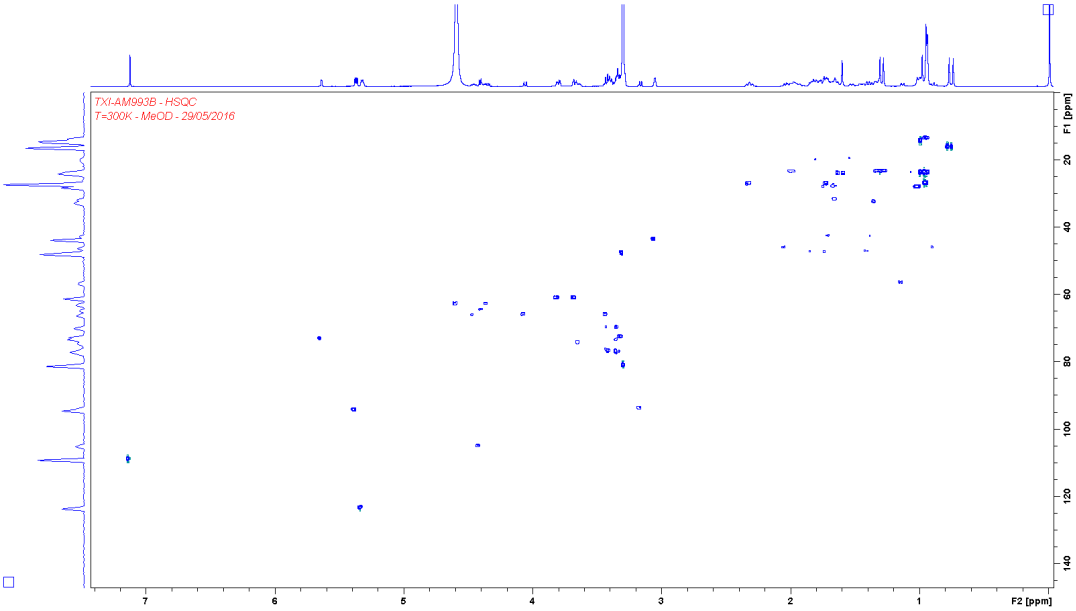
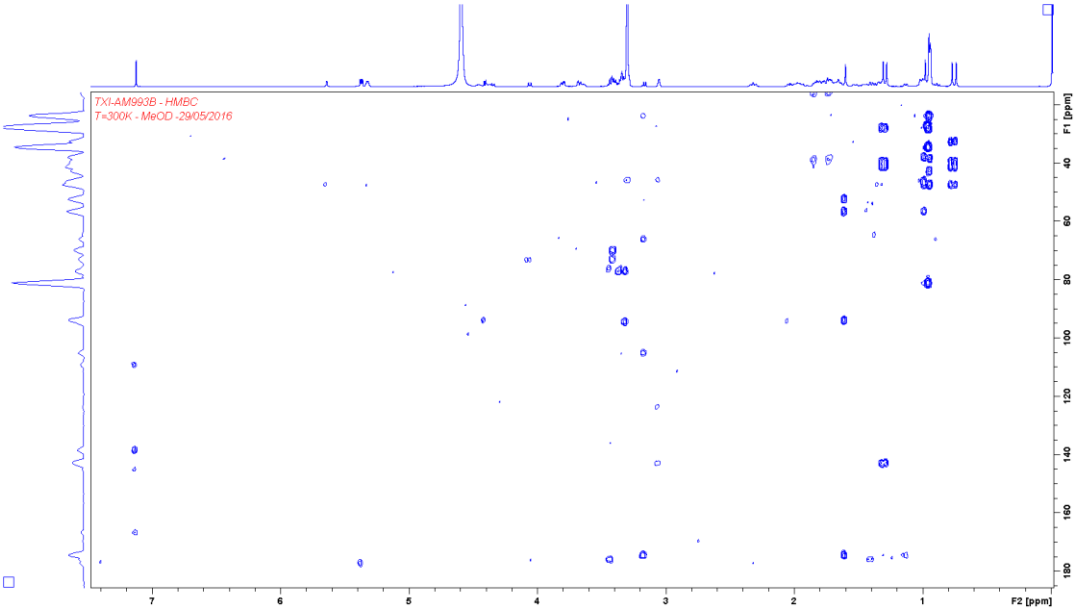




S17. ^{1}H NMR (600MHz, CD_3OD) (top), 2D COSY (CD_3OD), ^{1}H (600MHz) (middle), and 2D ROESY (CD_3OD), ^{1}H (600MHz) (bottom) spectra of compound **6**







S18. 2D HSQC (CD_3OD), ^1H (600MHz) and ^{13}C (150MHz) (top) and 2D HMBC (CD_3OD), ^1H (600MHz) and ^{13}C (150MHz) (bottom) spectra of compound **6**