Supporting information for

Oxysterols from Free Radical Chain Oxidation of 7-Dehydrocholesterol: Product and Mechanistic studies

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Table S1. ^{1}H-NMR chemical shifts of compounds **1** to **6**.[a]

Proton	**1**[b]	**2a**[b]	**2b**[c]	**3**[d]	**4**[d]	**5**[b]	**6a**[b]	**6b**[b]
3	3.93 (m)	3.87 (m)	4.01 (m)	3.99 (m)	4.11 (m)	4.32 (m)	3.91 (m)	3.90 (m)
4	2.25 (dd, 13.3, 11.8), 1.40 (m)	2.59 (ddd, 14.4, 4.6, 2.2), 1.32 (m)		2.21 (ddd, 14.3, 4.9, 1.7), 1.66 (m)	1.87 (m), 1.70 (m)	5.42 (s, br)	2.13 (dd, 13.0, 11.7), 1.42 (m)	2.18 (dd, 13.3, 11.7), 1.44 (m)
6	3.16 (d, 4.2)	3.97 (d, 11.9)	3.77 (d, br, 2.8)	5.88 (d, 9.5)	5.50 (d, 9.8)	5.87 (d, 9.7)	3.14 (d, 3.6)	3.00 (d, 2.5)
7	5.54 (dd, 4.3, 2.9)	5.30 (t, 2.2)	5.50 (dd, 4.4, 2.9)	5.56 (d, 9.5)	6.21 (d, 9.8)	6.19 (d, 9.7)	4.43 (dd, 9.3, 3.2)	4.54 (t, br, 2.3)
9					2.41 (m)		2.34 (m)	2.22 (m)
14	2.38 (m)	2.21 (m)	2.22 (m)					
15						2.45 (m), 2.32 (m)	2.64 (m), 2.29 (m)	2.39 (t, 14.5), 2.25 (m)
18	0.56 (s)	0.57 (s)	0.62 (s)	0.90 (s)	0.90 (s)	0.92 (s)	0.85 (s)	0.88 (s)
19	1.08 (s)	1.19 (s)	1.26 (s)	1.16 (s)	0.77 (s)	0.90 (s)	0.86 (s)	1.02 (s)
21	0.91 (d, 6.5)	0.90 (d, 6.4)	0.91 (d, 6.5)	0.89 (d, 6.6)	0.95 (d, 6.6)	0.95 (d, 6.7)	0.92 (d, 6.6)	0.92 (d, 6.7)
26	0.85 (d, 3.0)	0.85 (d, 2.0)	0.86 (d, 2.6)	0.85 (d, 2.2)	0.86 (d, 1.1)	0.86 (d, 2.2)	0.86 (d, 2.8)	0.86 (d, 2.6)
27	0.86 (d, 3.0)	0.87 (d, 2.0)	0.87 (d, 2.6)	0.86 (d, 2.2)	0.87 (d, 1.1)	0.87 (d, 2.2)	0.87 (d, 2.8)	0.87 (d, 2.6)

[a] All in $CDCl_3$. [b] 600 MHz. [c] 500 MHz. [d] 400 MHz

Table S2. ^{1}H-NMR chemical shifts of compounds **7** to **12**.[a,b]

Proton	**7**	**8**	**9**	**10**	**11**	**12a**	**12b**
1		2.34 (dt, 13.7, 3.6), 1.53 (m)			2.33 (dt, 17.5, 3.8), 1.55 (m)	2.08 (dt, 13.8, 3.7), 1.22 (m)	2.05 (dt, 13.9, 3.5), 1.15 (m)
3	4.00 (m)	4.06 (m)	4.08 (m)	4.03 (m)	4.09 (m)	3.68 (m)	3.77 (m)
4	2.22 (ddd, 14.0, 4.8, 2.0), 1.60 (dd, 14.0, 11.0)	2.08 (dd, 14.4, 3.2), 1.79 (dd, 14.2, 11.6)	2.18 (ddd, 13.9, 4.8, 1.7), 1.65 (d, t, br, 12.6)	2.12 (m), 1.74 (m)	2.14 (ddd, 14.3, 4.9, 2.0), 1.74 (dd, 14.3, 11.5)	1.96 (dd, 13.6, 4.7), 1.28 (m)	1.92 (dd, 13.3, 11.6), 1.47 (m)
6	5.54 (d, 9.4)					3.72 (d, br, 7.2)	3.50 (m)
7	6.36 (d, 9.4)	5.68 (d, br, 1.4)	5.68 (s)	5.66 (s, br)	6.04 (s)	4.89 (s, br)	5.13 (dd, 5.0, 1.9)
9				2.51 (ddd, 11.8, 7.1, 2.4)			
11			6.06 (dt, 6.9, 1.9)				
12			2.54 (m), 2.26 (d, 17.9)				
14		2.73 (dd, 10.8, 6.9)	2.52 (m)			2.40 (m)	2.37 (m)
15	2.46 (m), 2.36(m)				6.09 (t, 2.5)		
16					2.49 (ddd, 17.0, 7.6, 3.6), 2.08 (dd, 17.0, 10.8)		
18	0.87 (s)	0.61 (s)	0.61 (s)	0.60 (s)	0.91 (s)	0.51 (s)	0.53 (s)
19	0.99 (s)	1.03 (s)	1.13 (s)	0.95 (s)	1.05 (s)	0.90 (s)	0.95 (s)
21	0.93 (d, 6.7)	0.93 (d, 5.5)	0.93 (d, 6.2)	0.94 (d, 6.5)	0.95 (d, 6.5)	0.89 (d, 6.5)	0.89 (d, 6.4)
26	0.86 (d, 2.5)	0.86 (d, 2.8)	0.87 (d, 2.9)	0.86 (d, 2.9)	0.87 (d, 1.5)	0.84 (d, 3.0)	0.83 (d, 2.4)
27	0.87 (d, 2.5)	0.87 (d, 2.7)	0.88 (d, 2.9)	0.87 (d, 2.9)	0.88 (d, 1.5)	0.85 (d, 3.0)	0.85 (d, 2,4)

[a] **7-11** in $CDCl_3$, **12a** and **12b** in DMSO-d6. [b] 600 MHz.

Table S3. ^{13}C-NMR chemical shifts of compounds **1** to **12**.[a]

Carbon	**1**	**2a**	**2b**	**3**	**4**	**5**	**6a**	**6b**	**7**	**8**	**9**	**10**	**11**	**12a**	**12b**
1	25.9	29.1	28.8	27.7	29.3	33.8	32.3	33.1	27.6	25.6	29.1	30.5	25.2	26.5	26.9
2	31.0	30.9	31.8	30.9	31.0	29.4	31.3	31.4	31.5	30.3	30.3	30.4	30.3	30.6	31.0
3	68.5	66.7	66.9	66.1	67.2	68.5	68.8	68.8	66.4	67.4	67.5	67.6	67.3	65.4	65.8
4	40.1	34.2	34.9	35.8	41.8	125.9	39.7	39.7	36.0	37.5	35.0	36.7	37.1	40.0	40.2
5	68.4	88.4	86.7	85.9	74.1	145.5	67.9	63.7	85.4	79.9	77.6	78.0	79.6	76.8	77.3
6	55.8	73.2	72.3	135.7	130.2	125.8	61.5	60.4	129.6	197.5	198.1	198.4	197.3	69.1	71.6
7	116.9	123.3	122.7	128.8	128.0	126.4	65.3	65.1	128.5	120.1	118.4	119.8	119.1	121.1	120.4
8	151.8	141.8	142.0	63.9	124.6	124.9	125.2	126.8	126.6	164.2	155.0	165.5	154.1	141.0	141.2
9	73.6	84.3	84.8	87.0	39.8	45.6	38.8	40.6	86.5	74.8	140.4	44.1	74.9	73.4	73.7
10	38.7	54.3	51.0	50.6	39.1	35.9	36.0	35.0	51.0	42.0	42.1	40.6	42.1	40.4	40.1
11	27.8	22.8	23.3	19.9	19.8	19.3	19.2	19.5	23.4	29.0	132.3	22.1	27.8	26.8	27.5
12	35.7	36.8	36.8	33.6	36.9	36.6	36.8	37.4	33.7	35.1	42.7	39.1	34.6	34.9	35.1
13	43.0	42.3	42.2	40.6	44.2	43.9	43.3	44.0	43.9	45.6	43.6	45.1	47.1	43.4	43.4
14	51.8	52.1	52.1	75.3	151.7	150.1	152.8	151.9	152.4	51.9	52.4	55.9	148.8	49.8	50.1
15	23.2	23.2	23.3	26.6	25.1	25.1	25.1	26.0	25.7	22.6	22.9	22.7	131.4	22.4	22.6
16	27.9	28.1	28.1	27.7	27.4	27.4	26.8	27.6	27.3	27.8	28.2	27.8	36.3	27.6	27.8
17	55.9	55.8	55.8	55.7	55.8	56.0	56.9	55.4	55.8	56.4	56.4	56.4	58.9	55.5	55.7
18	11.7	11.4	11.6	15.5	15.9	19.1	18.1	19.3	17.0	12.1	11.6	12.6	17.2	11.2	11.4
19	20.4	16.3	17.4	15.7	19.1	18.5	16.7	17.1	16.4	20.7	24.2	16.6	20.4	20.1	21.5
20	36.2	36.1	36.2	34.3	34.9	34.8	34.7	34.8	34.6	36.1	36.0	36.1	34.0	35.6	35.7
21	18.9	18.9	18.9	18.9	19.0	19.2	19.1	19.0	19.0	18.9	18.6	18.9	19.0	18.7	18.7
22	36.1	36.2	36.1	33.6	36.0	36.1	36.0	36.0	36.0	36.1	36.0	36.1	36.1	35.5	35.6
23	24.0	24.0	24.0	23.7	23.8	23.9	23.8	23.8	23.8	24.0	24.0	24.2	23.8	23.2	23.3
24	39.6	39.6	39.6	39.6	39.7	39.7	39.7	39.6	39.6	39.6	39.6	39.6	39.6	38.9	39.0
25	28.1	28.1	28.1	28.1	28.2	28.2	28.2	28.2	28.2	28.2	28.2	28.1	28.2	27.4	27.5
26	22.7	22.7	22.7	22.7	22.7	22.7	22.7	22.7	22.7	22.7	22.7	22.7	22.7	22.4	22.5
27	23.0	22.9	23.0	22.9	22.9	23.0	23.0	22.9	22.9	23.0	23.0	23.0	22.9	22.7	22.7

[a] **1-11** in $CDCl_3$, **12a** and **12b** in DMSO-d6

UV and HRMS of isolated oxysterols:

5α,6α-Epoxycholesta-7-en-3β,9α-diol (1). Small impurity (< 15%) cannot be separated with either normal phase or reverse phase HPLC. UV, λ_{max} = 222 nm (10% 2-propanol in hexanes). HRMS (ESI) $[M+Na]^+$, calculated for $C_{27}H_{44}O_3Li$, 423.3451, found, 423.3454.

5α,9α-Epidioxycholesta-7-en-3β,6α-diol (2a). HRMS (ESI) $[M+Na]^+$, calculated for $C_{27}H_{44}O_4Na$, 455.3137, found, 455.3127.

5α,9α-Epidioxycholesta-7-en-3β,6β-diol (2b). HRMS (ESI) $[M+Na]^+$, calculated for $C_{27}H_{44}O_4Na$, 455.3137, found, 455.3155.

5α,9α-Epidioxy-8α,14α-epoxycholesta-6-en-3β-ol (3). HRMS (ESI) $[M+Li]^+$, calculated for $C_{27}H_{42}O_4Li$, 453.3243, found, 453.3226.

Cholesta-6,8(14)-dien-3β,5α-diol (4).[1] UV: λ_{max} = 252 nm (10% 2-propanol in hexanes). HRMS (ESI) $[M+Li]^+$, calculated for $C_{27}H_{44}O_2Li$, 407.3501, found, 407.3492.

Cholesta-4,6,8(14)-trien-3β-ol (5).[1] UV: λ_{max} = 285 nm (2-propanol); 283 nm (10% 2-propanol in hexanes). HRMS (API) $[M+H^+-H_2O]^+$, calculated for $C_{27}H_{41}$, 365.3208, found, 365.3218.

5α,6α-Epoxycholesta-8(14)-en-3β,7α-diol (6a). HRMS (ESI) $[M+Na]^+$, calculated for $C_{27}H_{44}O_3Na$, 439.3188, found, 439.3179.

5α,6α-Epoxycholesta-8(14)-en-3β,7β-diol (6b). HRMS (ESI) $[M+Li]^+$, calculated for $C_{27}H_{44}O_3Li$, 423.3451, found, 423.3460.

5α,9α-Epidioxycholesta-6,8(14)-dien-3β-ol (7). UV: λ_{max} = 255 nm (2-propanol); 252 nm (10% 2-propanol in hexanes). HRMS (ESI) $[M+Na]^+$, calculated for $C_{27}H_{42}O_3Na$, 437.3032, found, 439.3032.

3β,5α,9α-Trihydroxycholesta-7-en-6-one (8). UV, λ_{max} = 238 nm (2-propanol); 237 nm (10% 2-propanol in hexanes); 238 nm (acetonitrile/methanol = 7/3). HRMS (ESI) $[M+Na]^+$, calculated for $C_{27}H_{44}O_4Na$, 455.3137, found, 455.3123.

3β,5α-Dihydroxycholesta-7,9(11)-dien-6-one (9).[2] UV, λ_{max} = 296 nm (2-propanol); 292 nm (10% 2-propanol in hexanes); 293 nm (acetonitrile/methanol = 7/3). HRMS (ESI) $[M+Li]^+$, calculated for $C_{27}H_{42}O_3Li$, 421.3294, found, 421.3290.

3β,5α-Dihydroxycholesta-7-en-6-one (10). UV, λ_{max} = 247 nm (2-propanol); 246 nm (10% 2-propanol in hexanes). HRMS (ESI) $[M+Na]^+$, calculated for $C_{27}H_{44}O_3Na$, 439.3188, found, 439.3192.

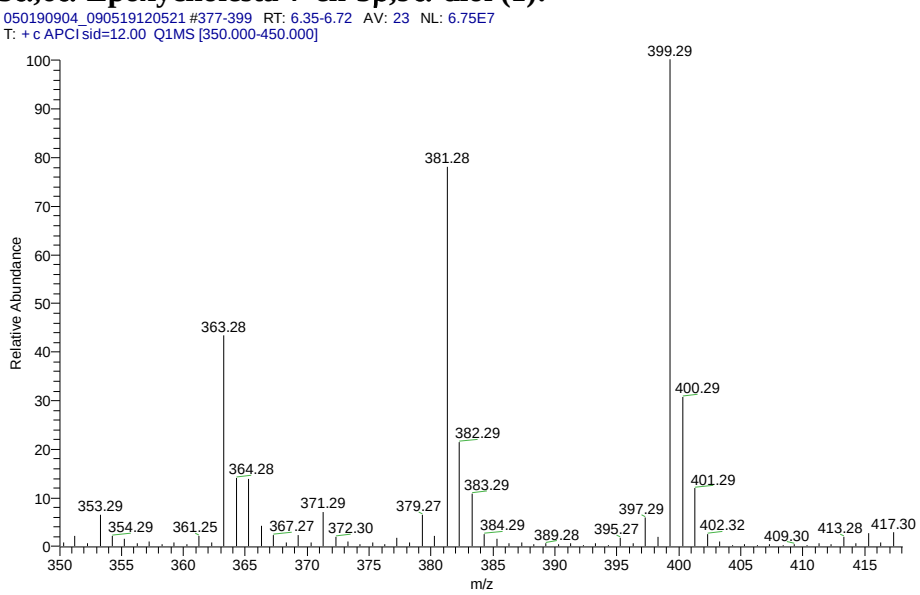
3β,5α,9α-Trihydroxycholesta-7,14-dien-6-one (11). UV, λ_{max} = 301 nm (2-propanol); 296 nm (10% 2-propanol in hexanes); 298 nm (acetonitrile/methanol = 7/3). HRMS (ESI) $[M+Li]^+$, calculated for $C_{27}H_{42}O_4Li$, 437.3243, found, 437.3235.

Cholesta-7-en-3β,6α,5α,9α-tetraol (12a). HRMS (ESI) $[M+Li]^+$, calculated for $C_{27}H_{46}O_4Li$, 441.3556, found, 441.3569.

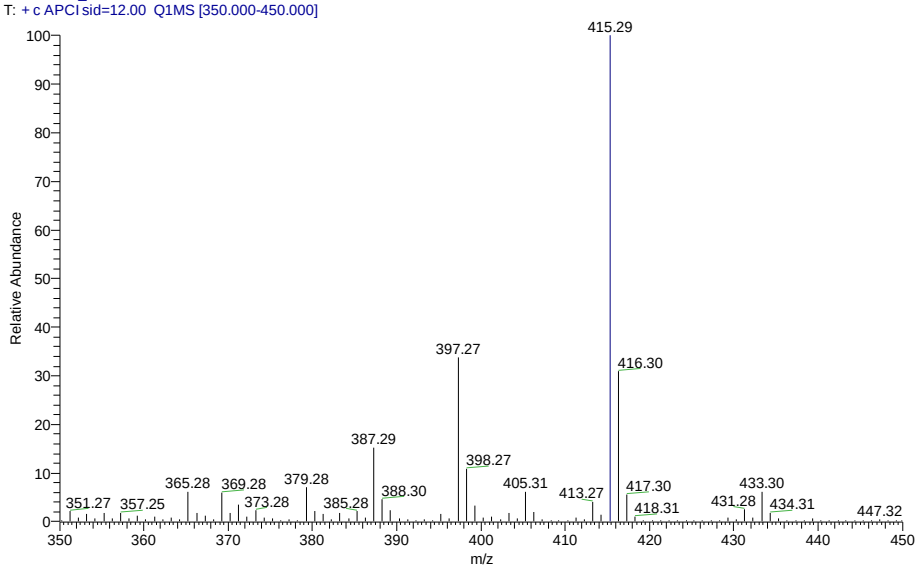
Cholesta-7-en-3β,6β,5α,9α-tetraol (12b). HRMS (ESI) $[M+Li]^+$, calculated for $C_{27}H_{46}O_4Li$, 441.3556, found, 441.3548.

Mass Spectra (APCI) of isolated oxysterols:

5α,6α-Epoxycholesta-7-en-3β,9α-diol (1):



5α,9α-Epidioxycholesta-7-en-3β,6α-diol (2a):



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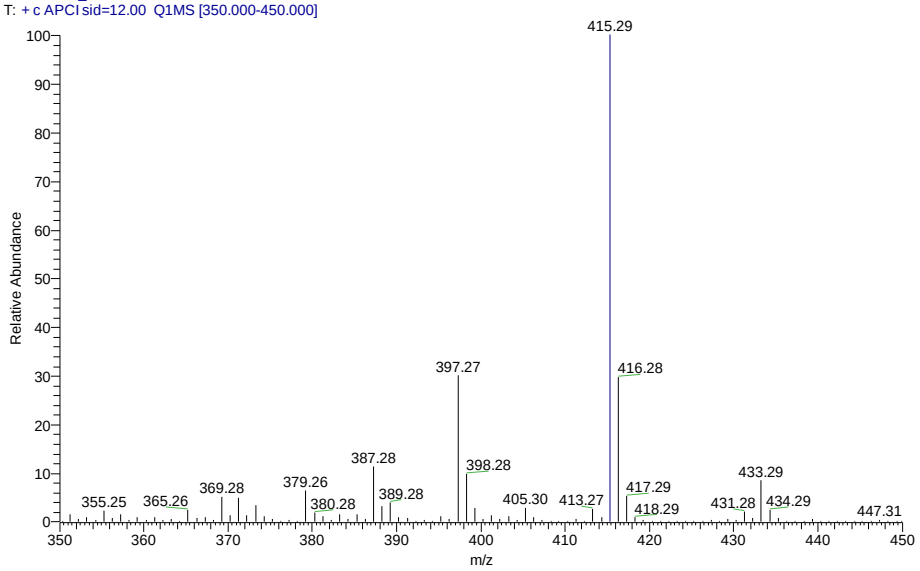

5α,9α-Epidioxy-8α,14α-epoxycholesta-6-en-3β-ol (3):

050190903_090519112334 #342-358 RT: 5.76-6.03 AV: 17 NL: 1.27E8
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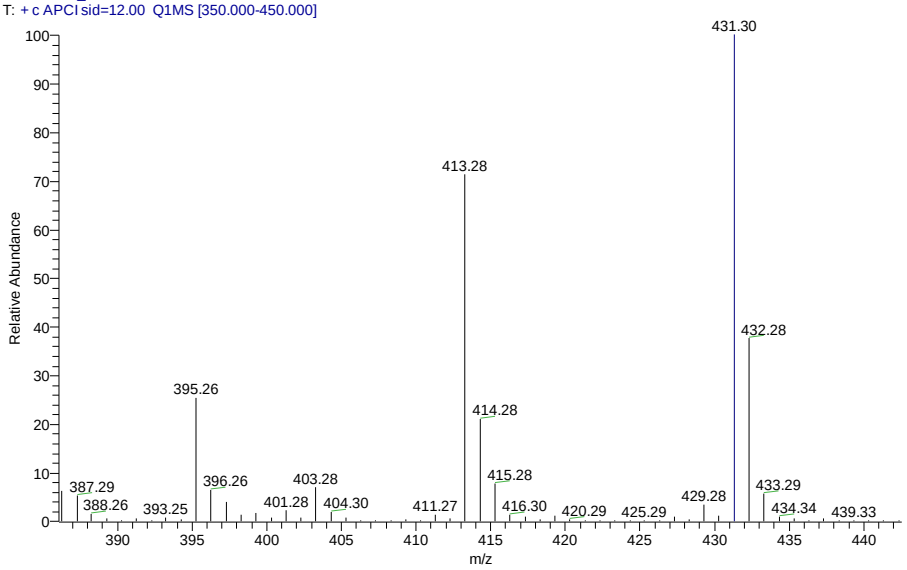

Cholesta-6,8(14)-dien-3β,5α-diol (4):

050190904_090519120521 #422-435 RT: 7.11-7.33 AV: 14 NL: 1.54E8
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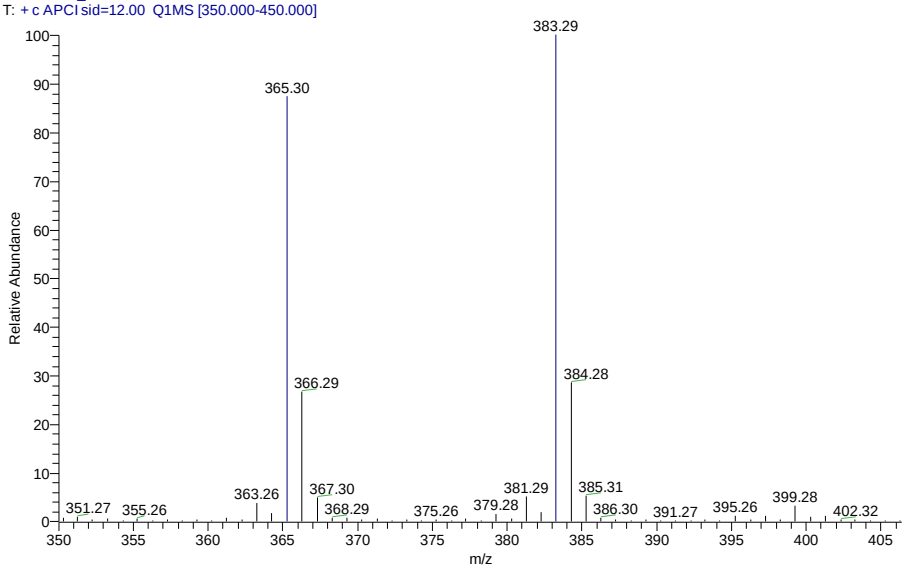

Cholesta-4,6,8(14)-trien-3β-ol (5):

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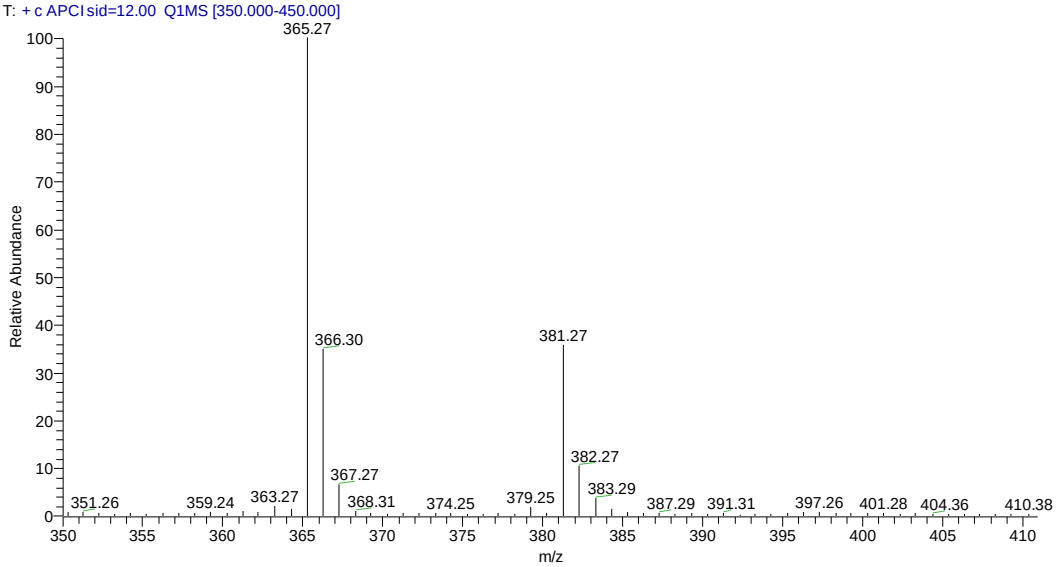
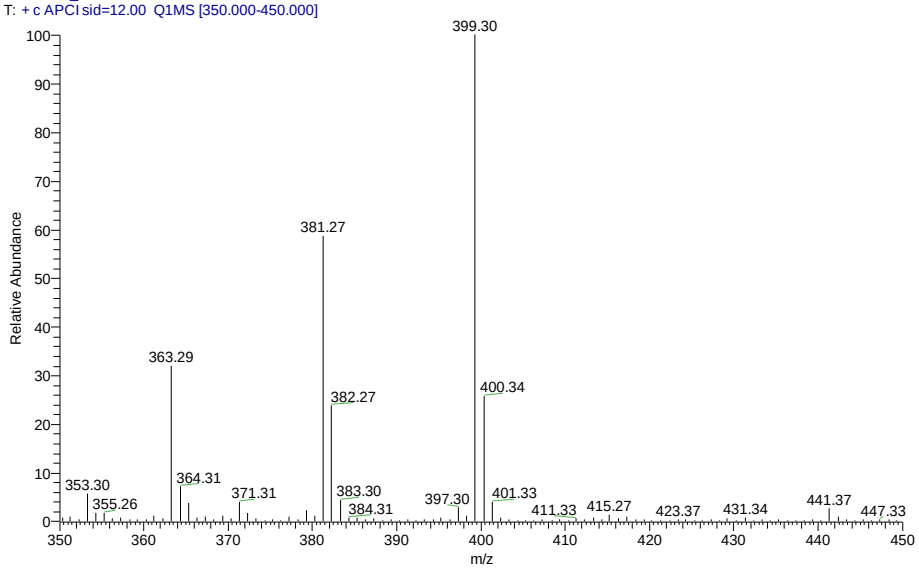
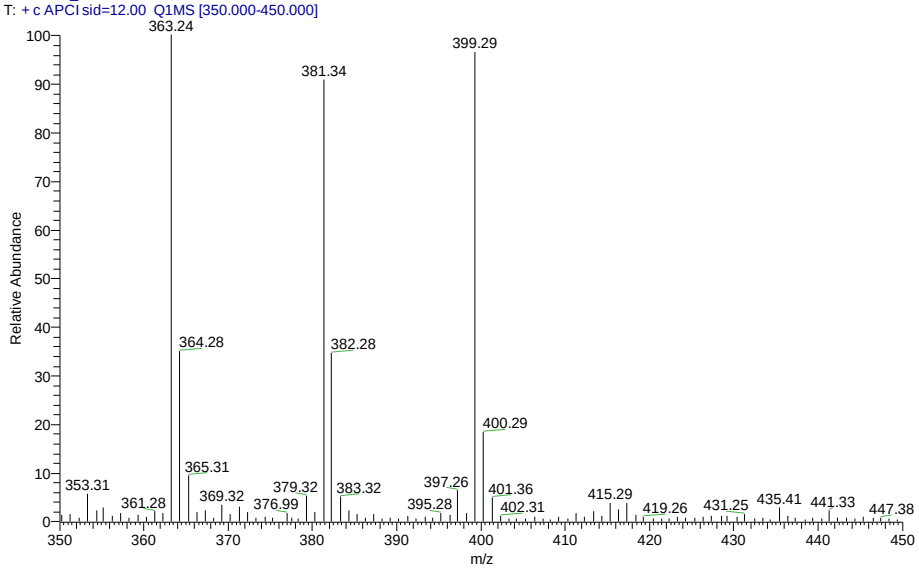


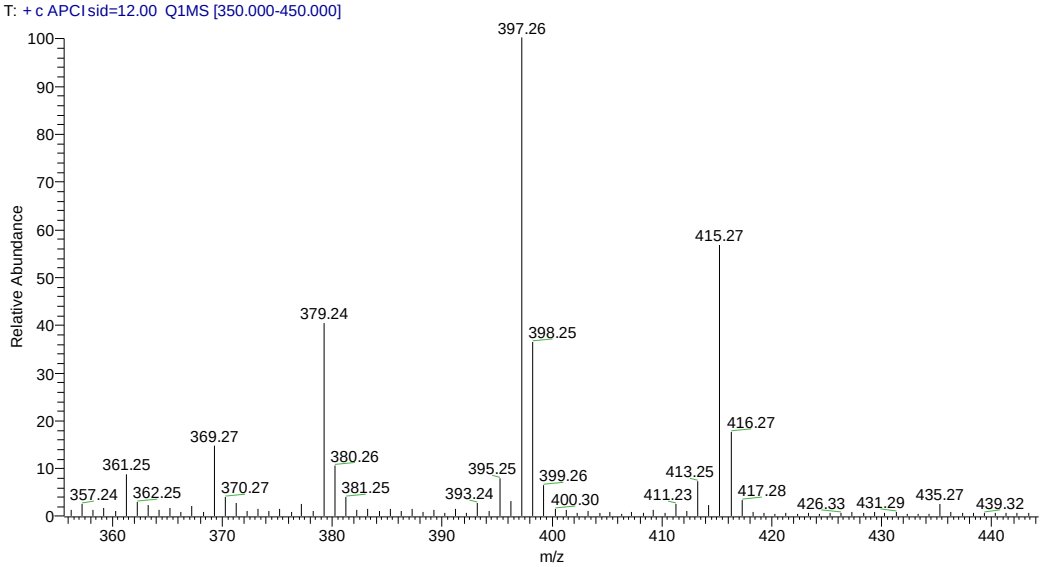
5α,6α-Epoxycholesta-8(14)-en-3β,7α-diol (6a):



5α,6α-Epoxycholesta-8(14)-en-3β,7β-diol (6b):



5α,9α-Epidioxycholesta-6,8(14)-dien-3β-ol (7):



3β,5α,9α-Trihydroxycholesta-7-en-6-one (8):

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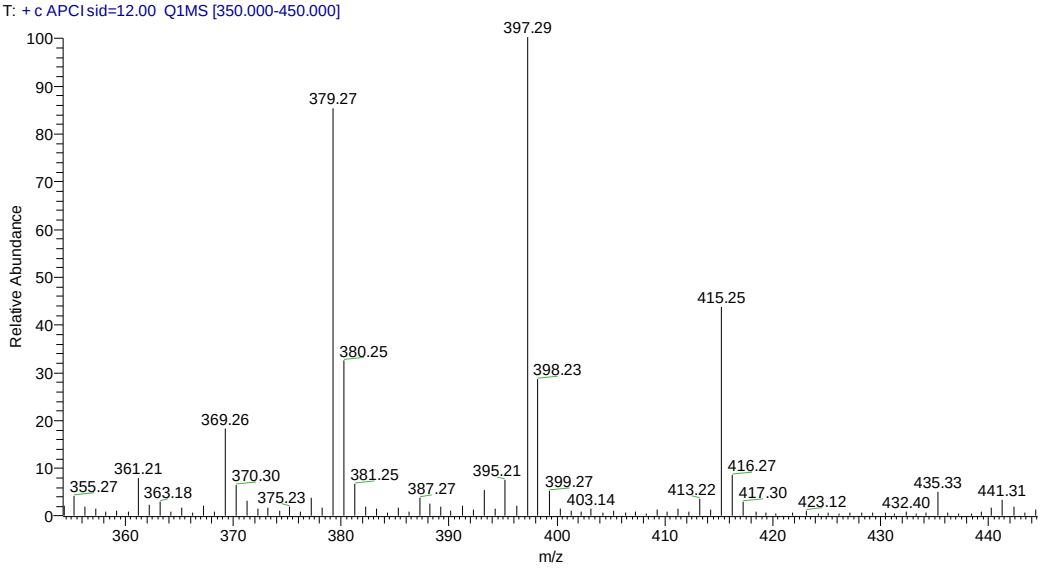

3β,5α-Dihydroxycholesta-7,9(11)-dien-6-one (9):

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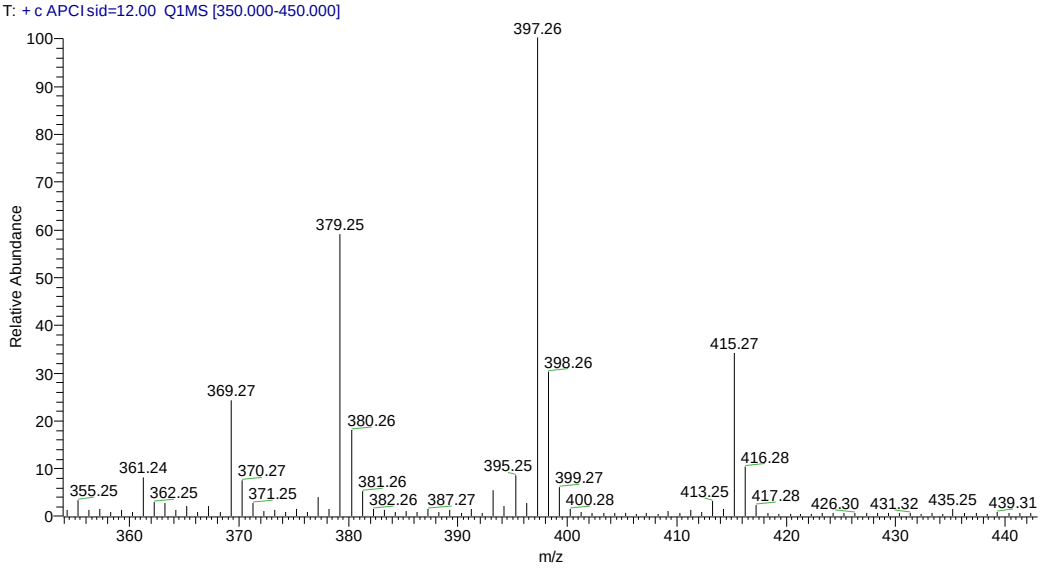

3β,5α-Dihydroxycholesta-7-en-6-one (10):

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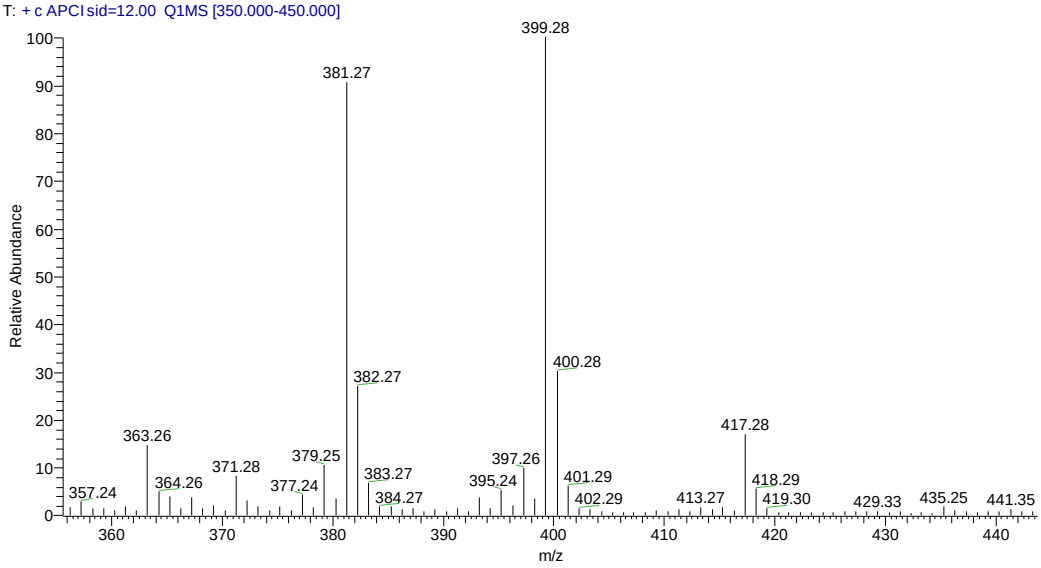

3β,5α,9α-Trihydroxycholesta-7,14-dien-6-one (11):

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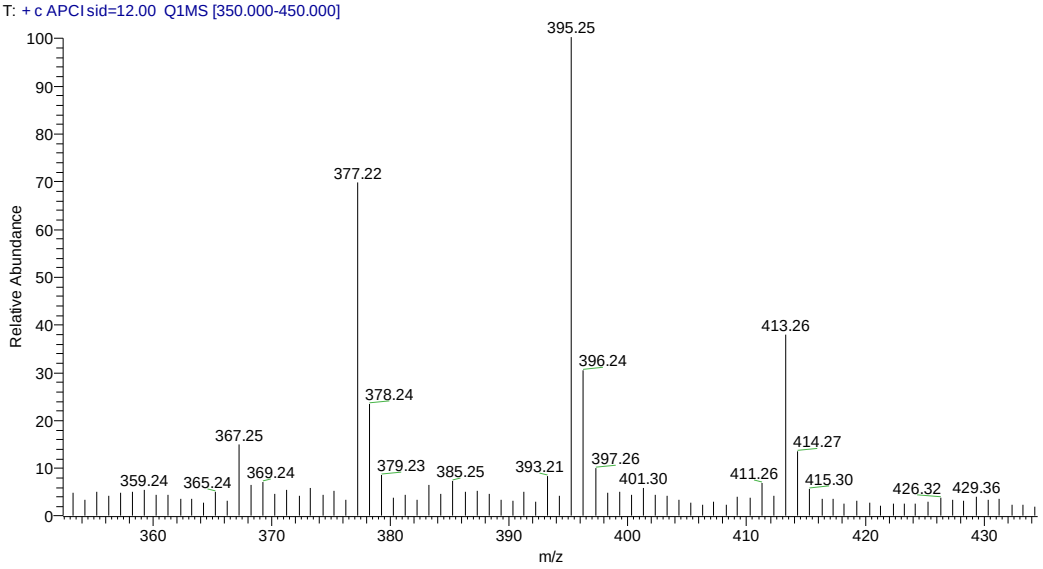

Cholesta-7-en-3β,6α,5α,9α-tetraol (12a):

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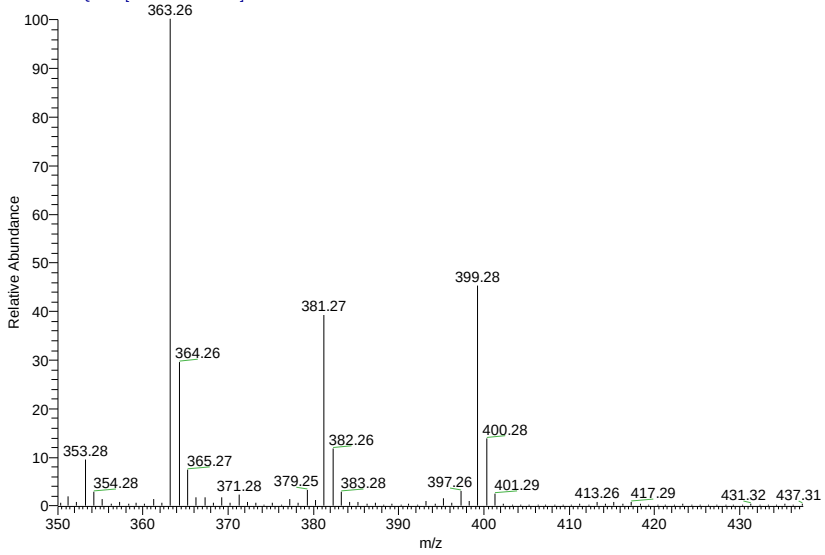
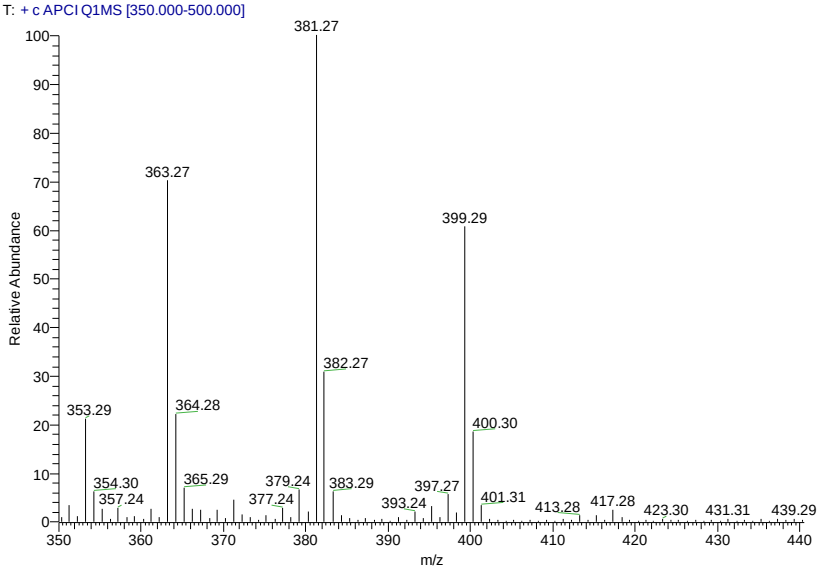


Cholesta-7-en-3β,6β,5α,9α-tetraol (12b):

11200803 #1812-1910 RT: 30.63-32.29 AV: 99 NL: 3.66E6
T: + c APCI Q1MS [350.000-500.000]



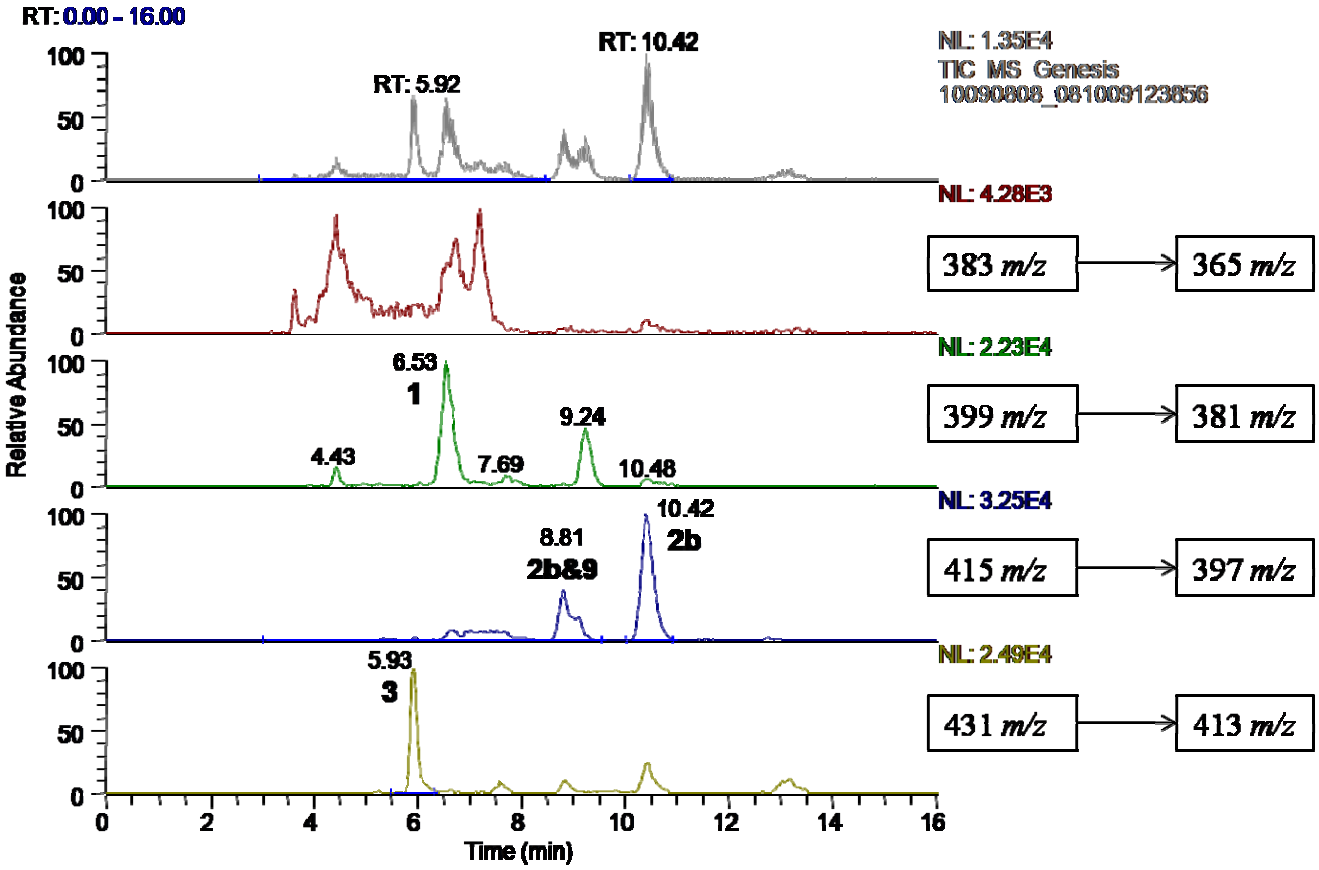


Figure S1. HPLC-APCI-MS-MS chromatogram of the reaction mixture of free radical oxidation of 7-hydroperoxycholesta-5,8(9)-dien-3β-ol (**32**) after two hours of reaction and PPh_3 reduction (see Scheme 6).

References

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(S2) Migliuolo, A.; Notaro, G.; Piccialli, V.; Sica, D. *Steroids* 1991, *56*, 154-158.

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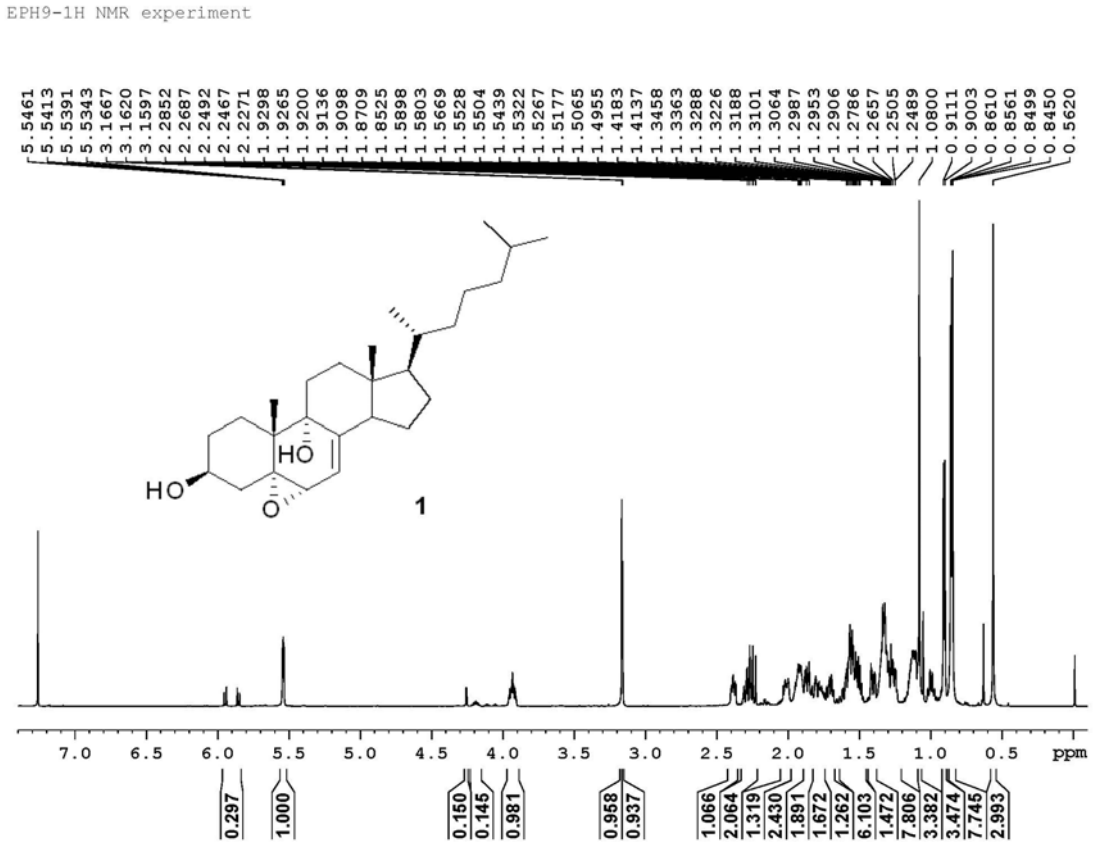
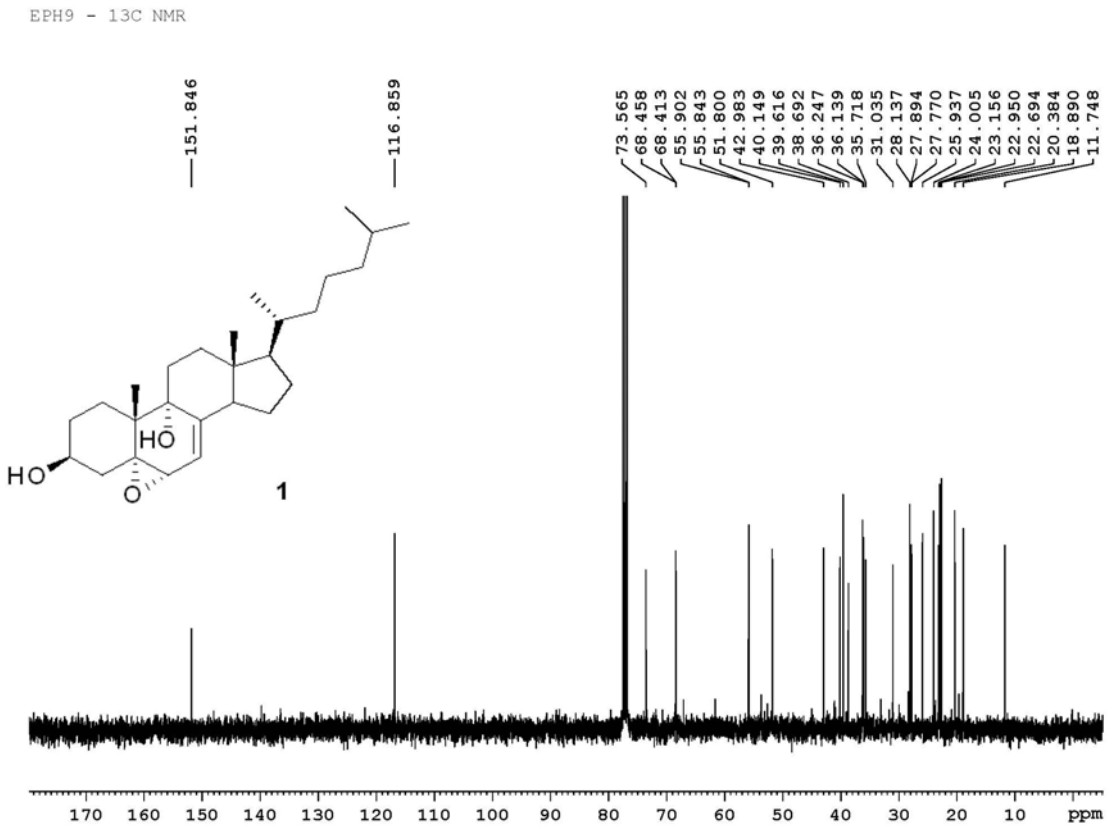
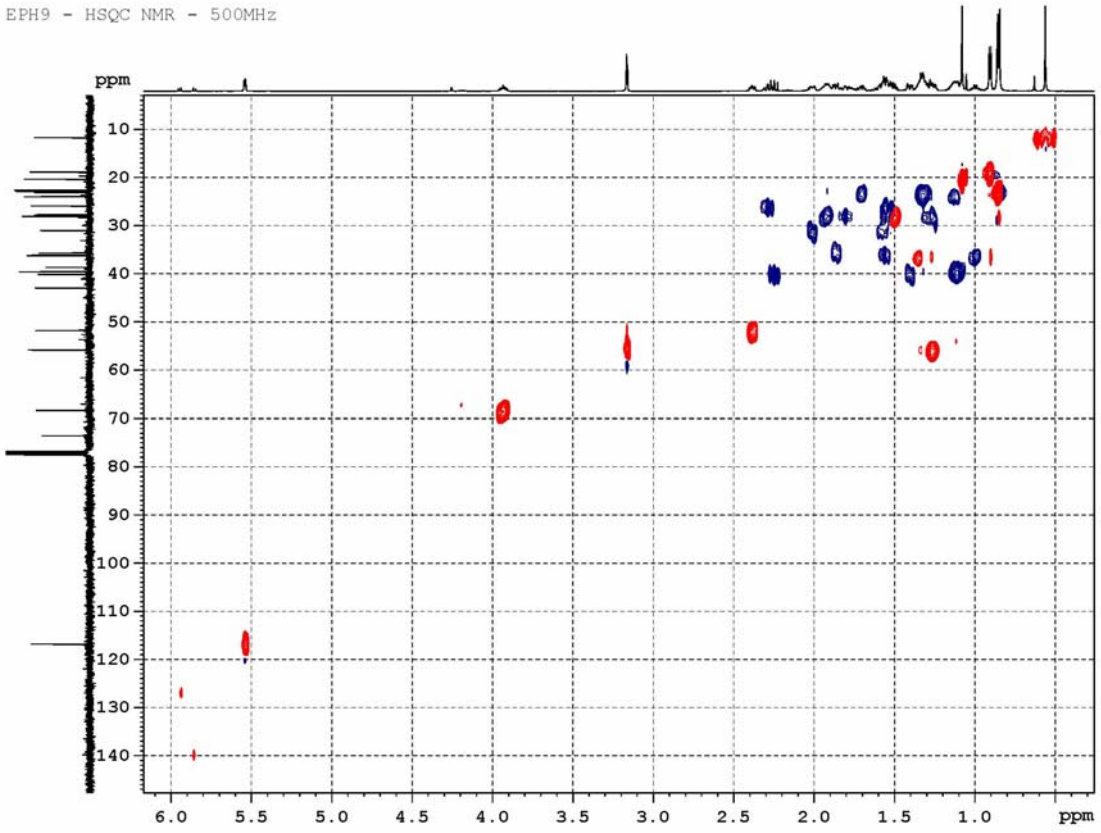
NMR Spectra:
5α,6α-Epoxycholesta-7-en-3β,9α-diol (1).



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80
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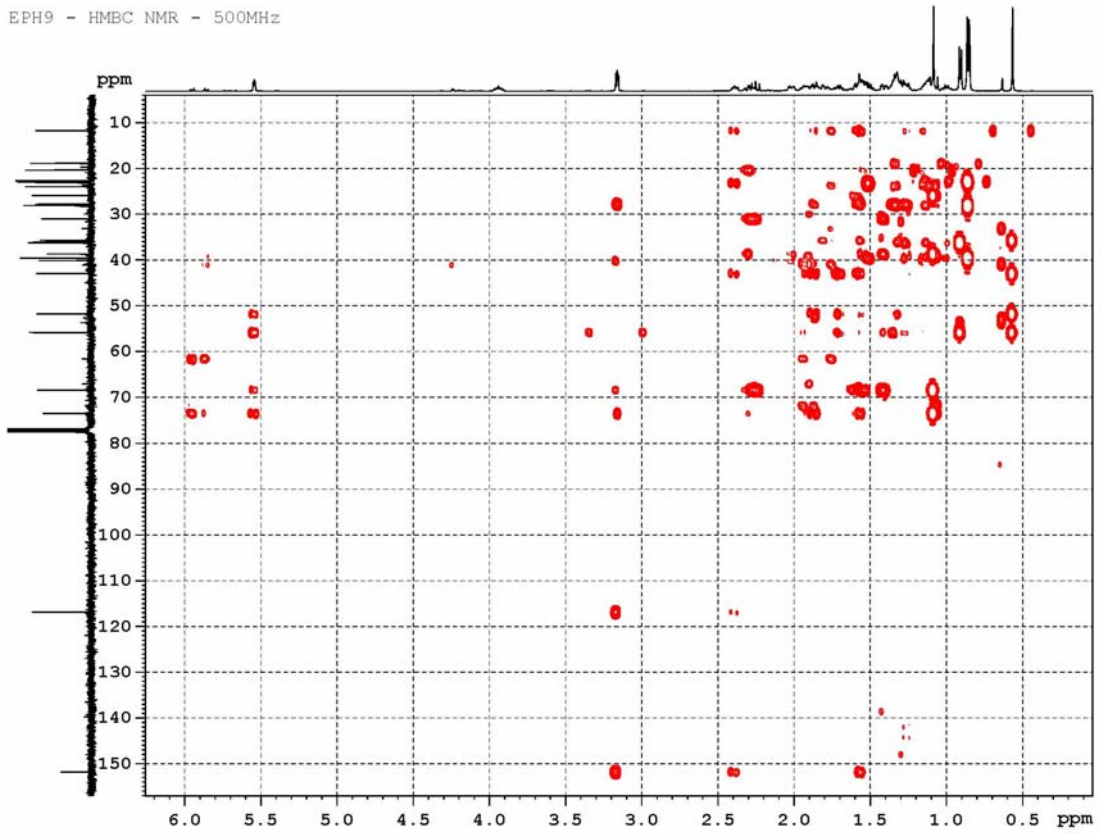
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm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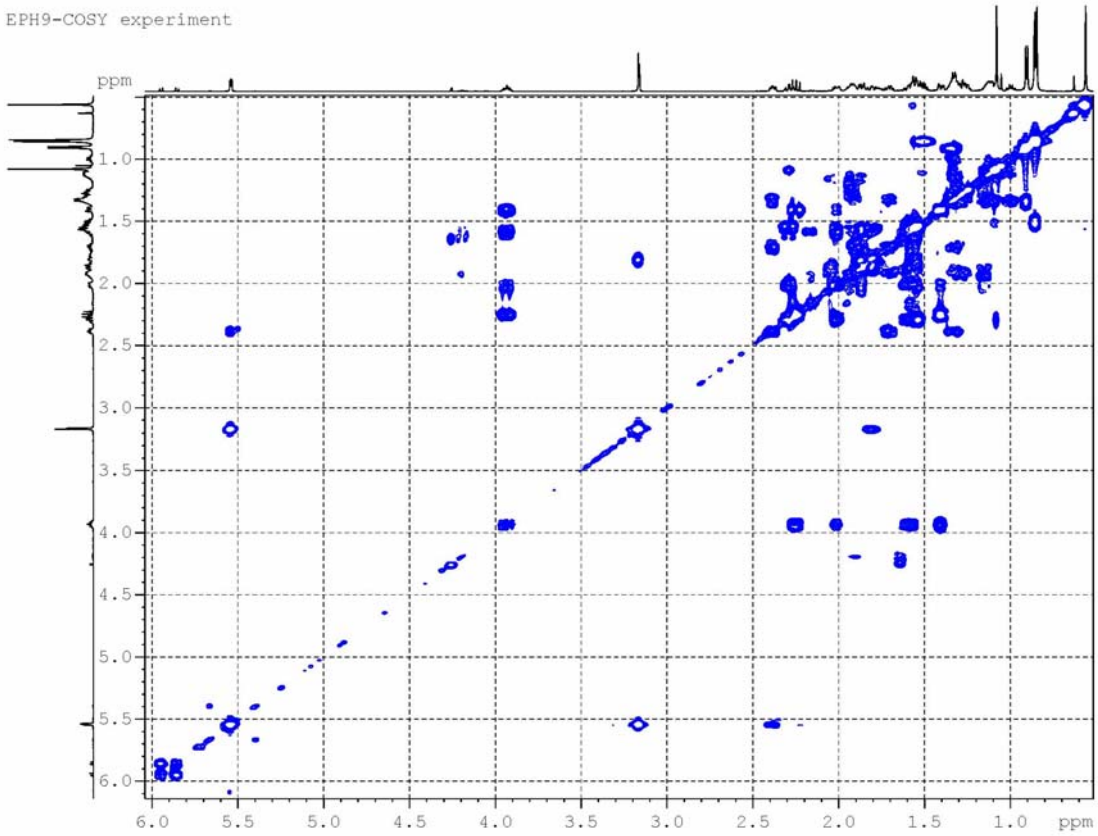
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm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3.5
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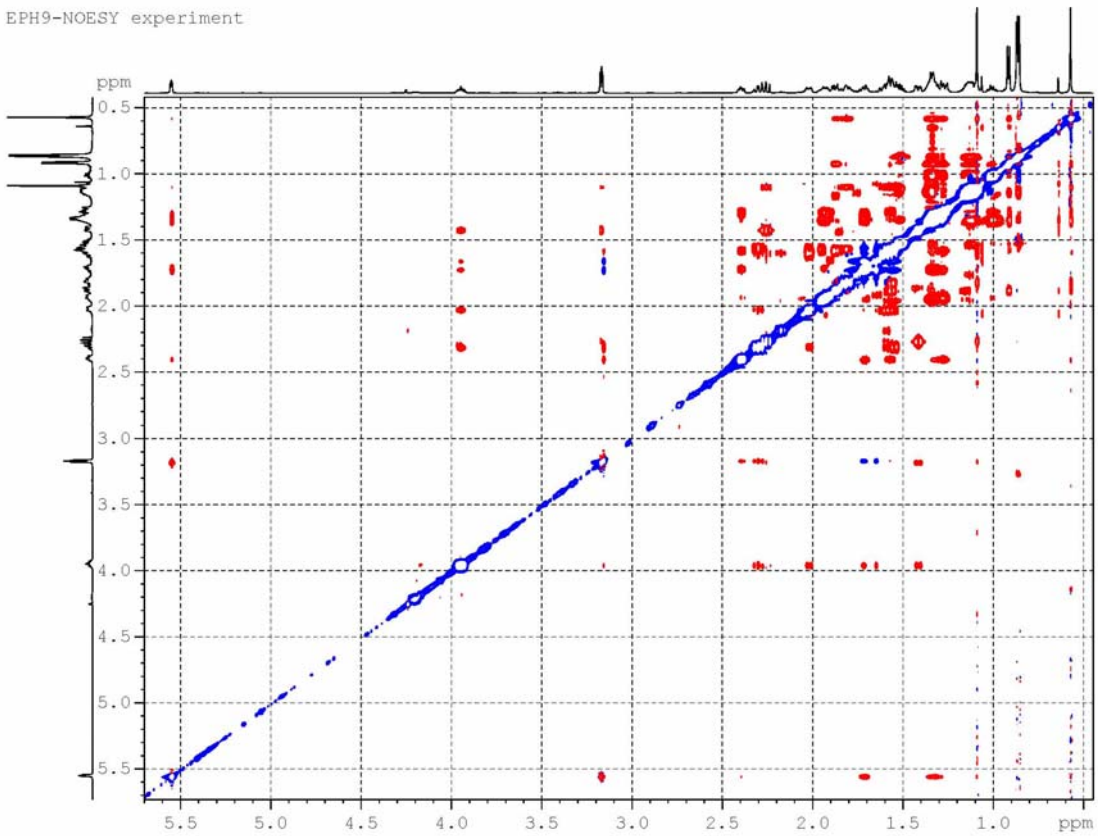
6.0
6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm


EPH9-NOESY experiment
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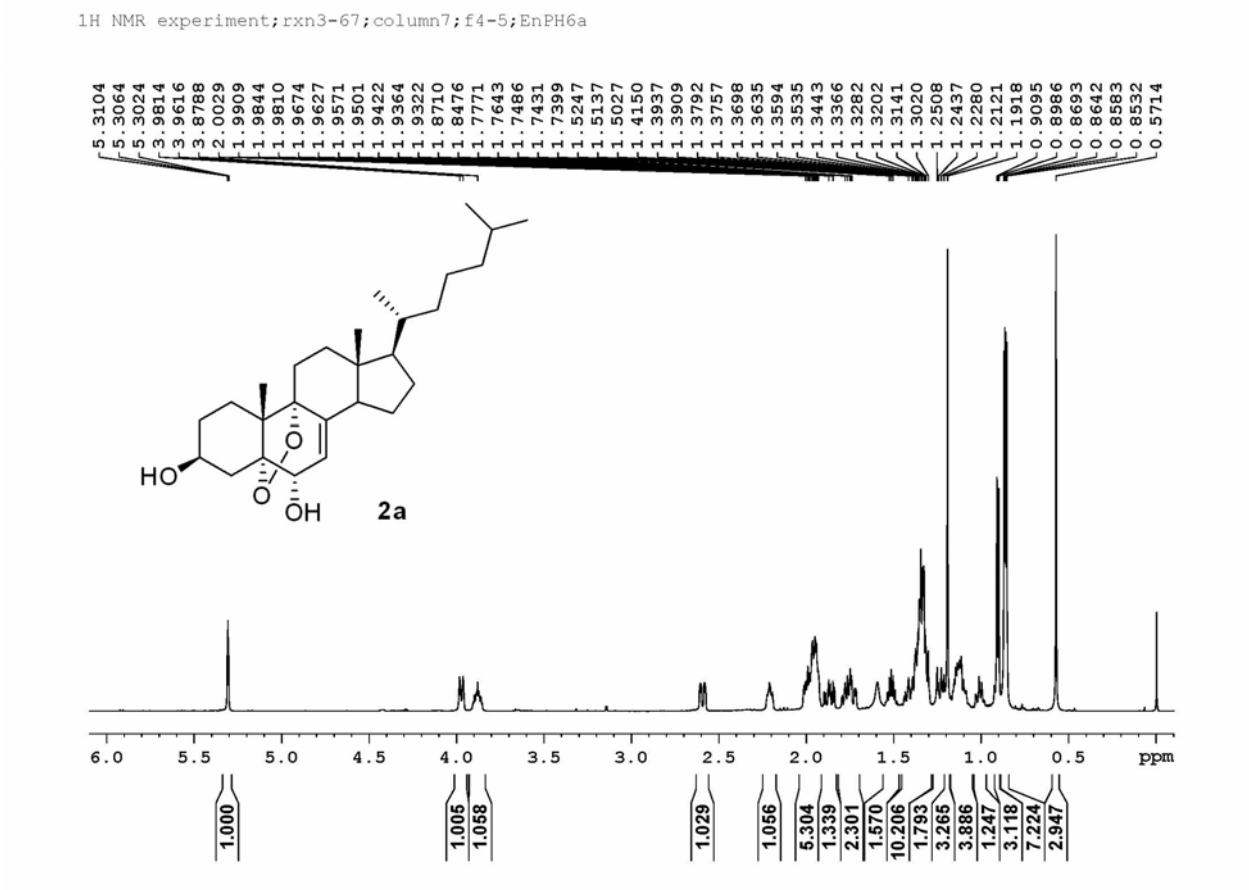
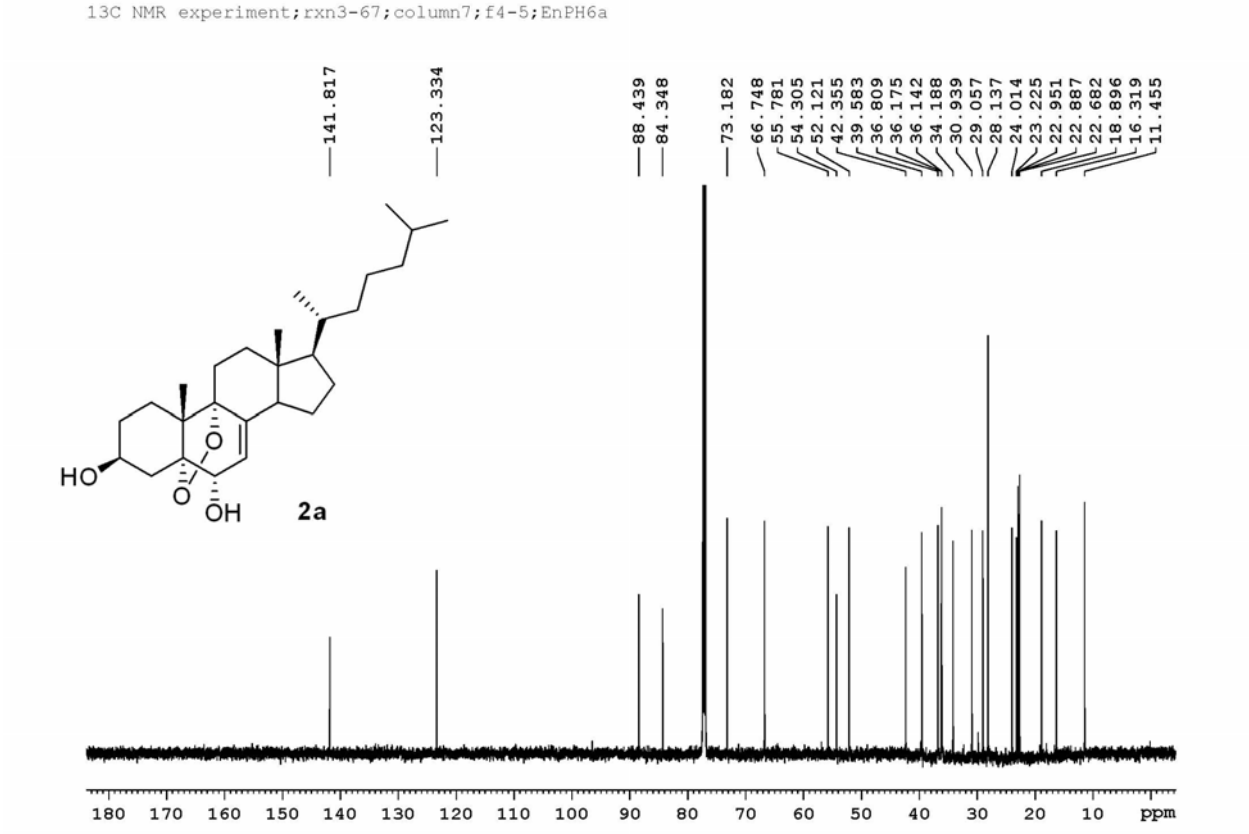
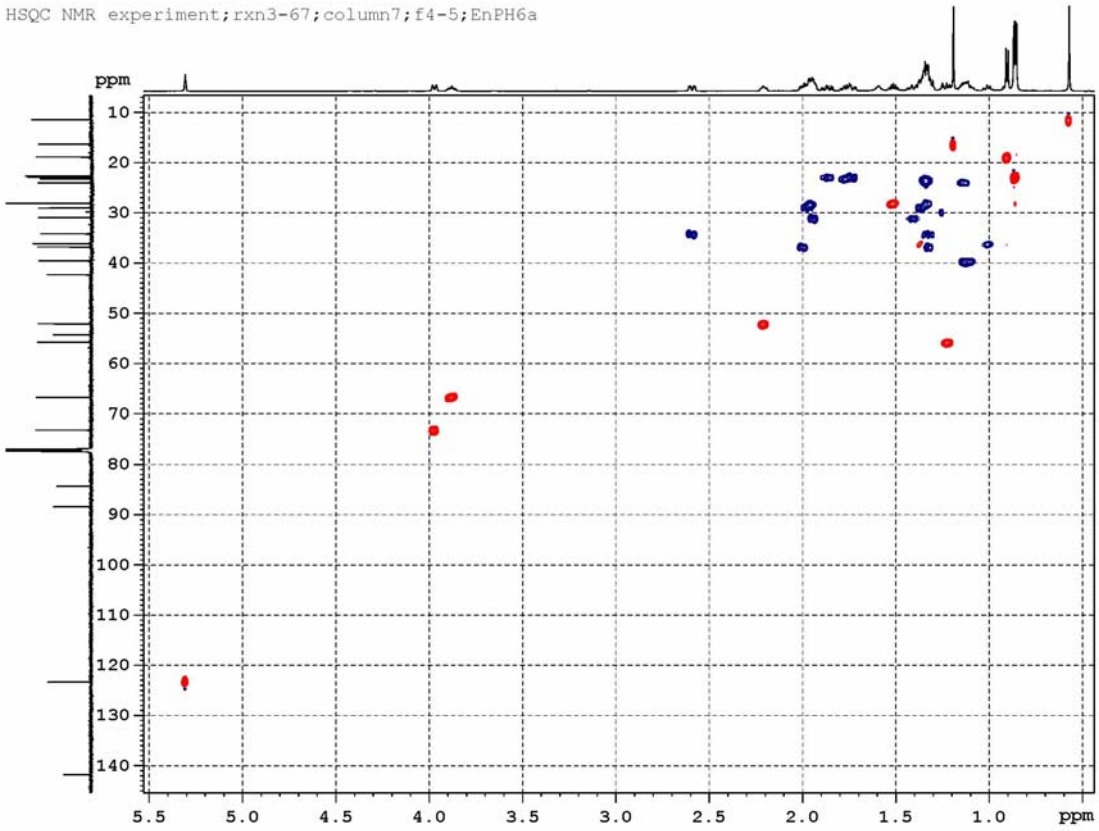
4.0
4.5
5.0
5.5
5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm

5α,9α-Epidioxycholesta-7-en-3β,6α-diol (2a).



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4.5
4.0
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1.5
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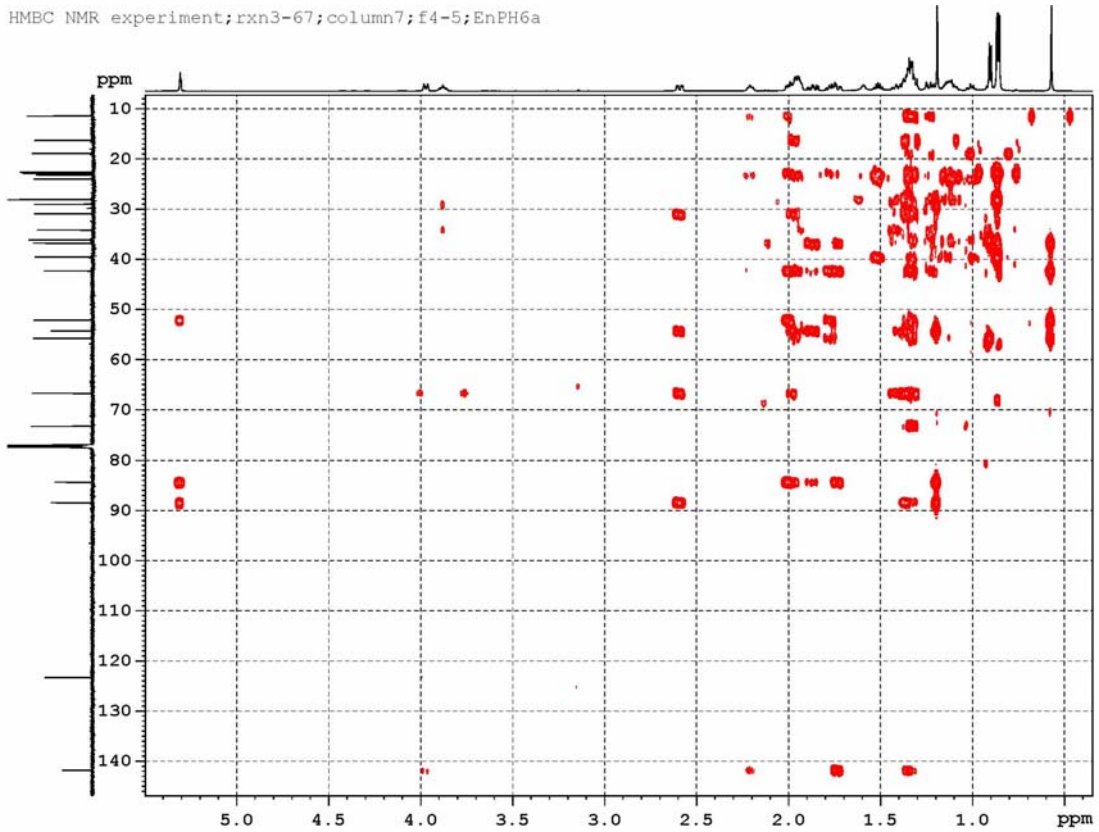
ppm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4.0
3.5
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2.5
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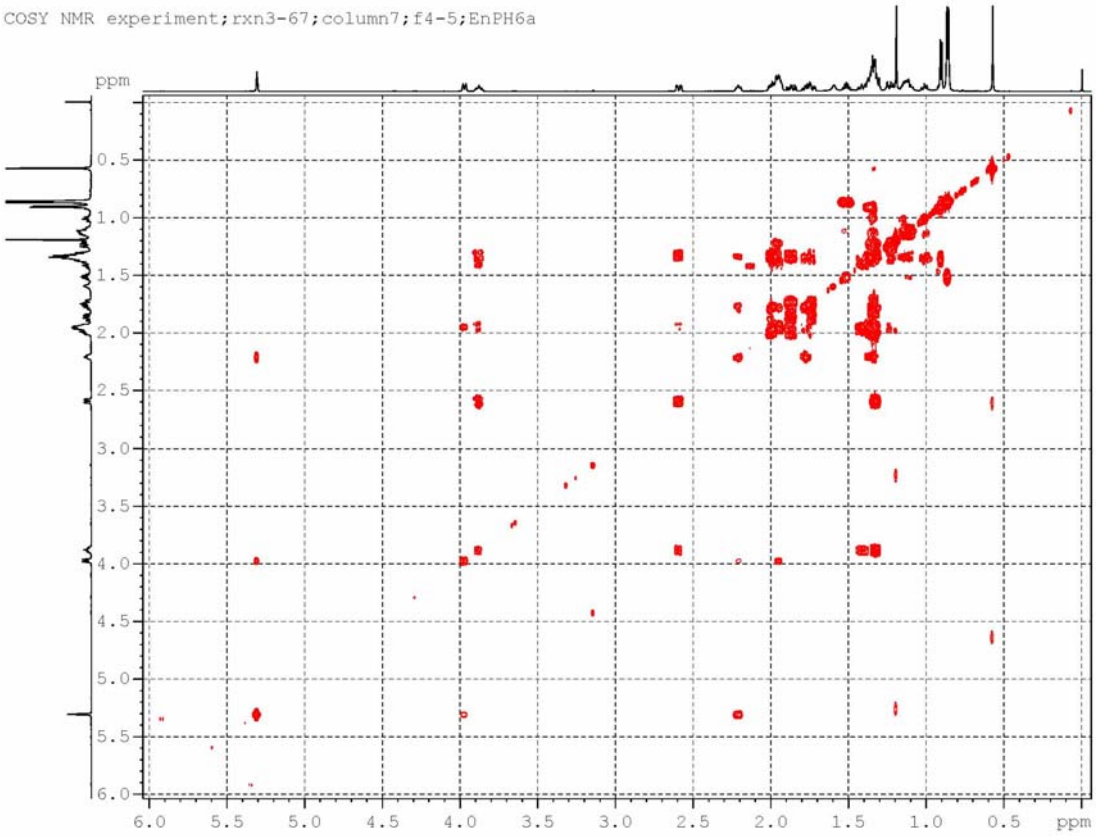
1.5
1.0
ppm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4.0
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6.0
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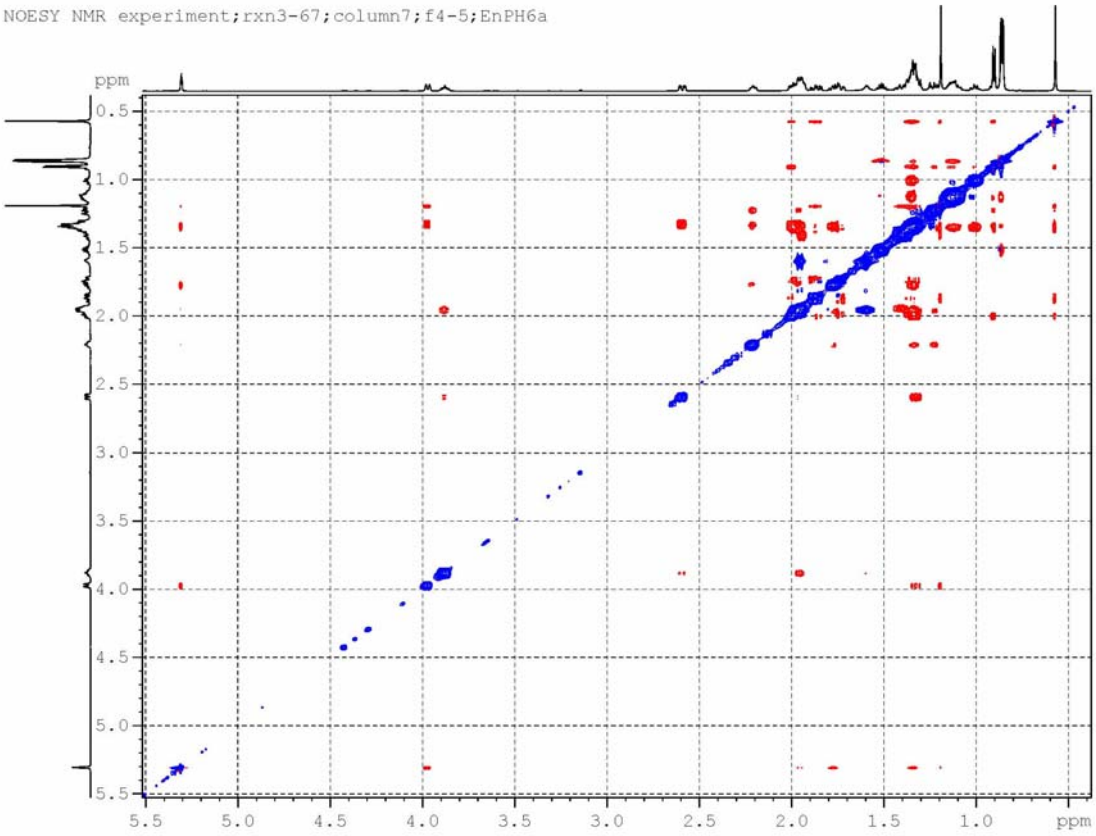
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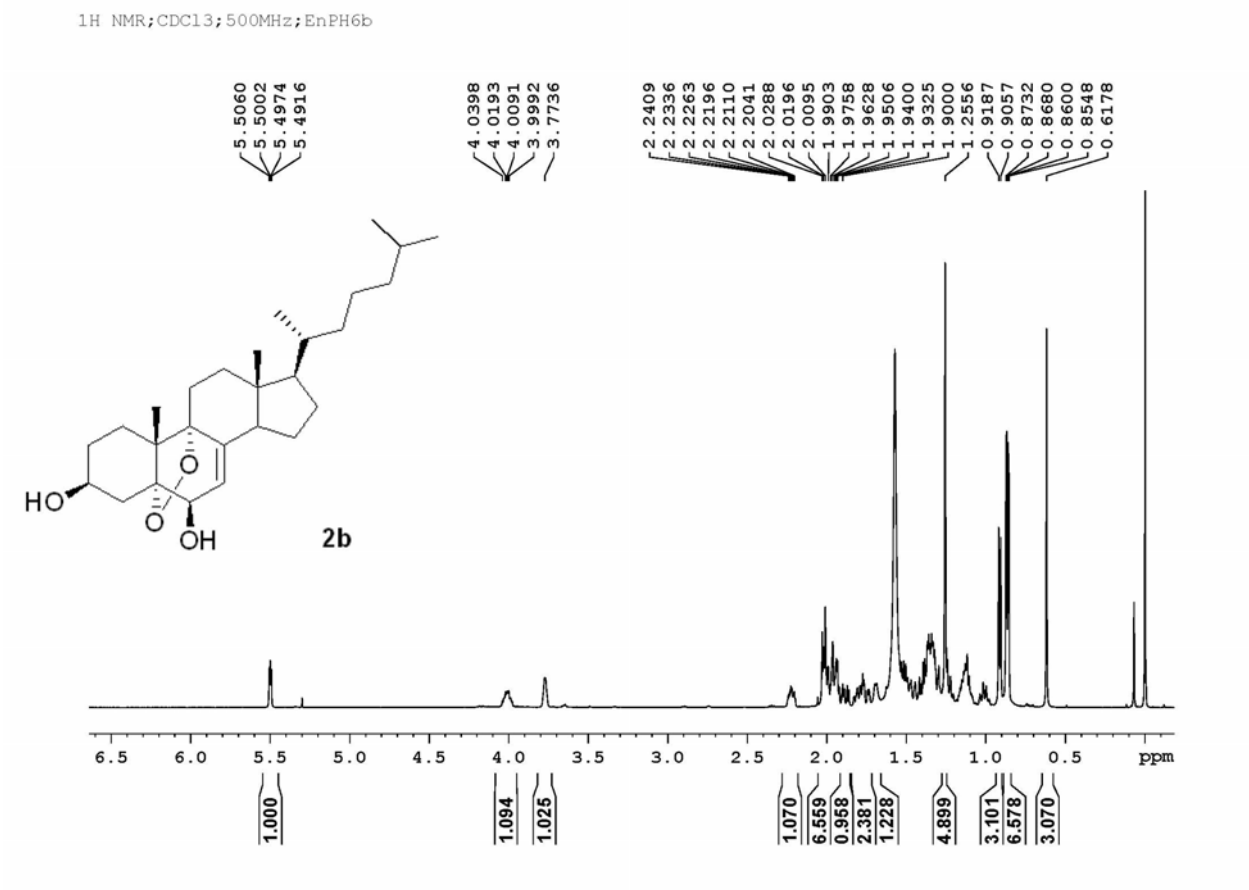
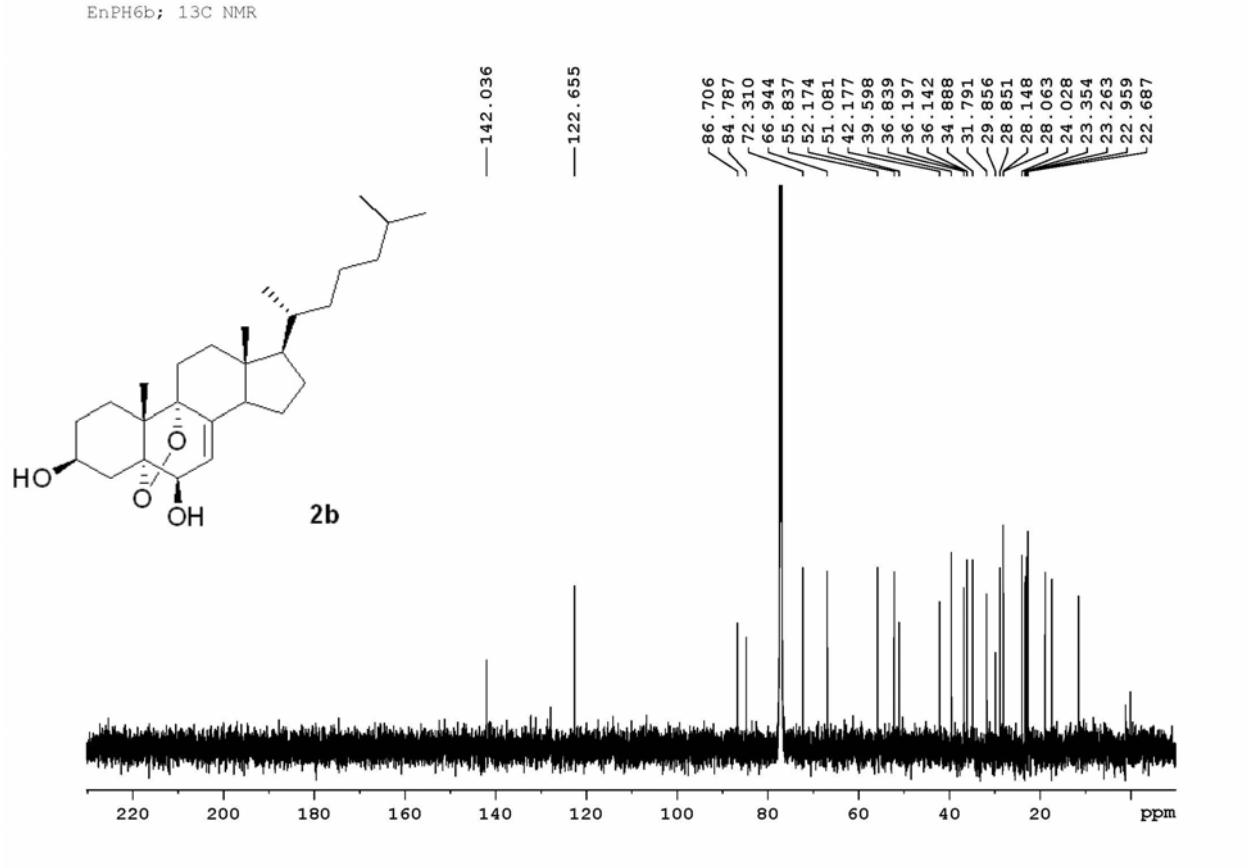
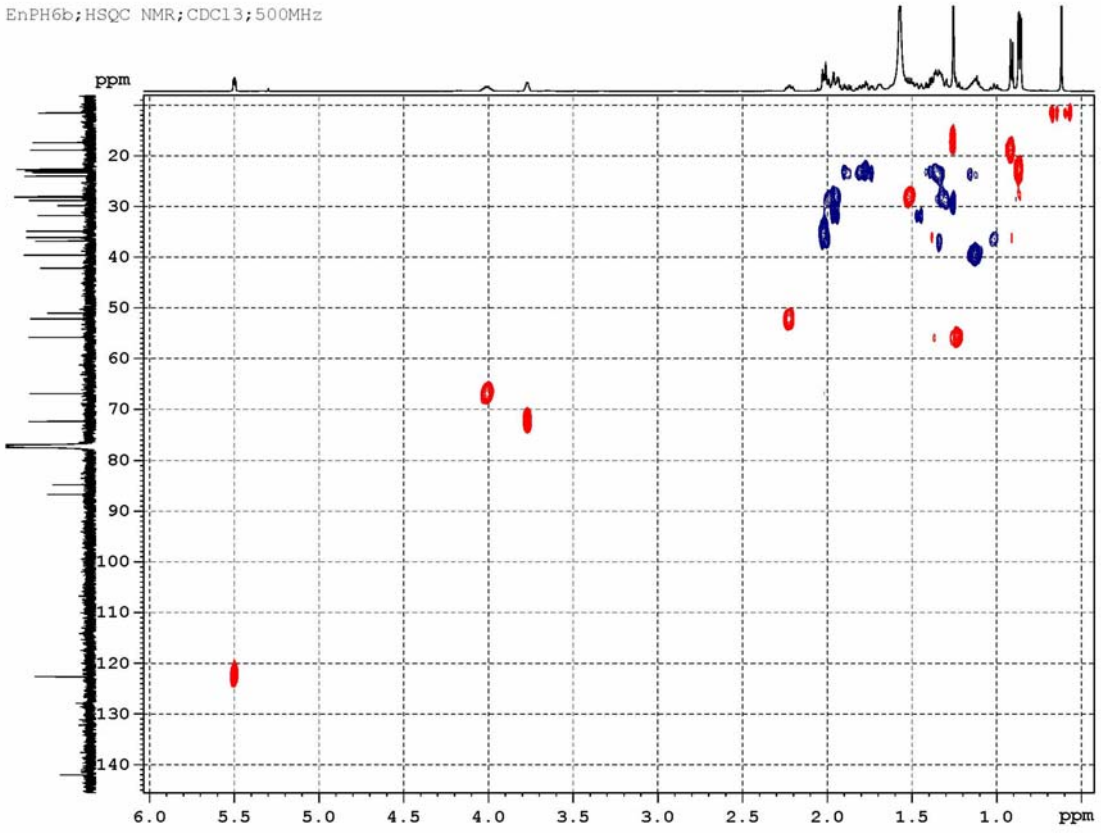
4.0
4.5
5.0
5.5
5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm

5α,9α-Epidioxycholesta-7-en-3β,6β-diol (2b).



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120
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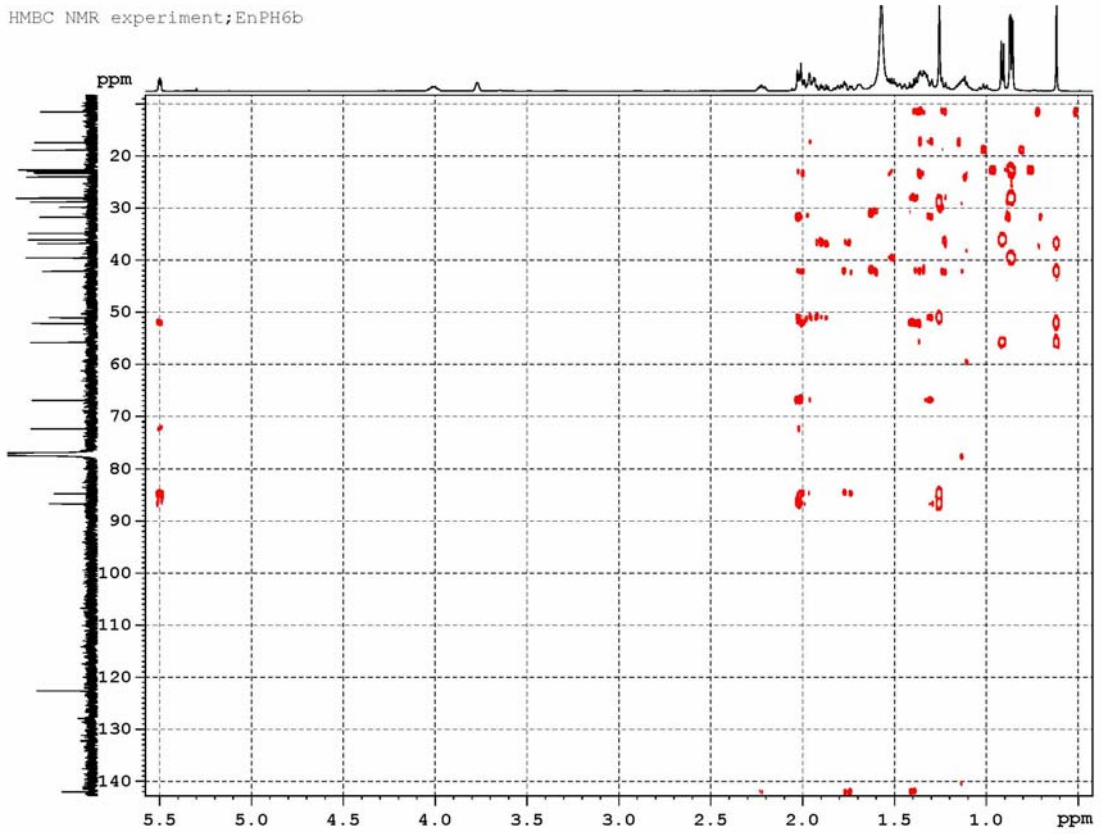
ppm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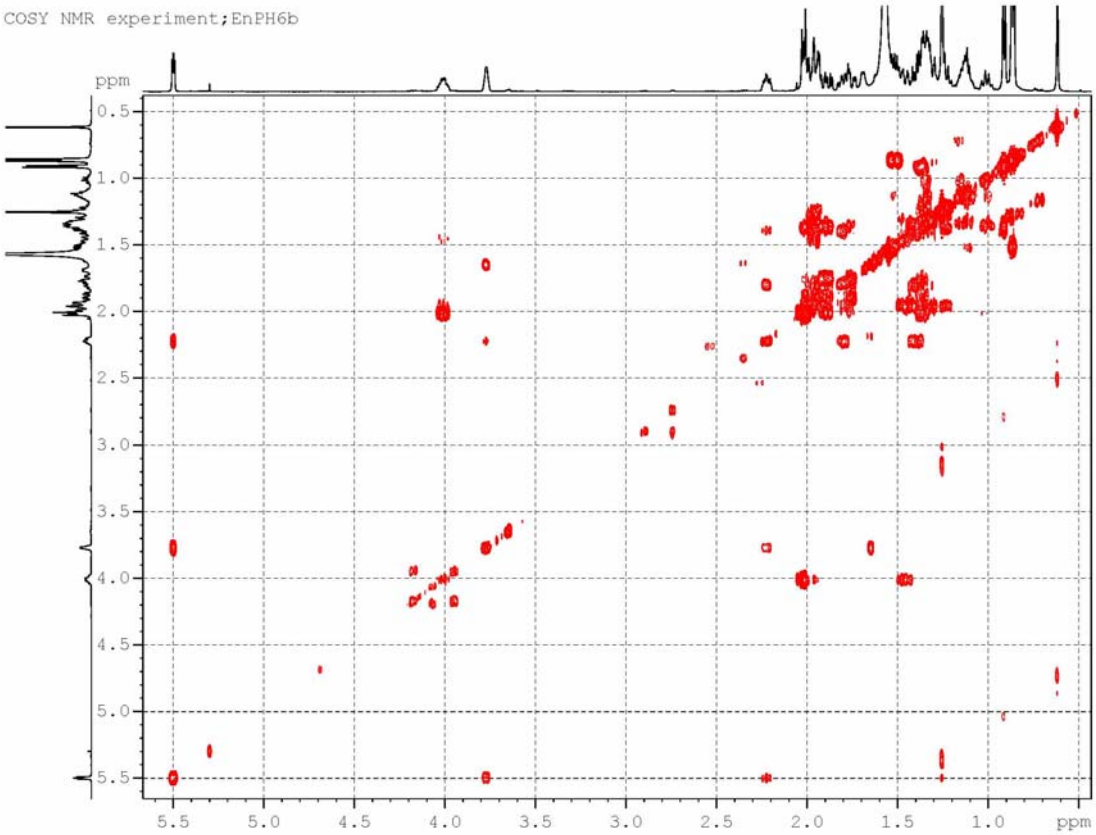
1.5
1.0
ppm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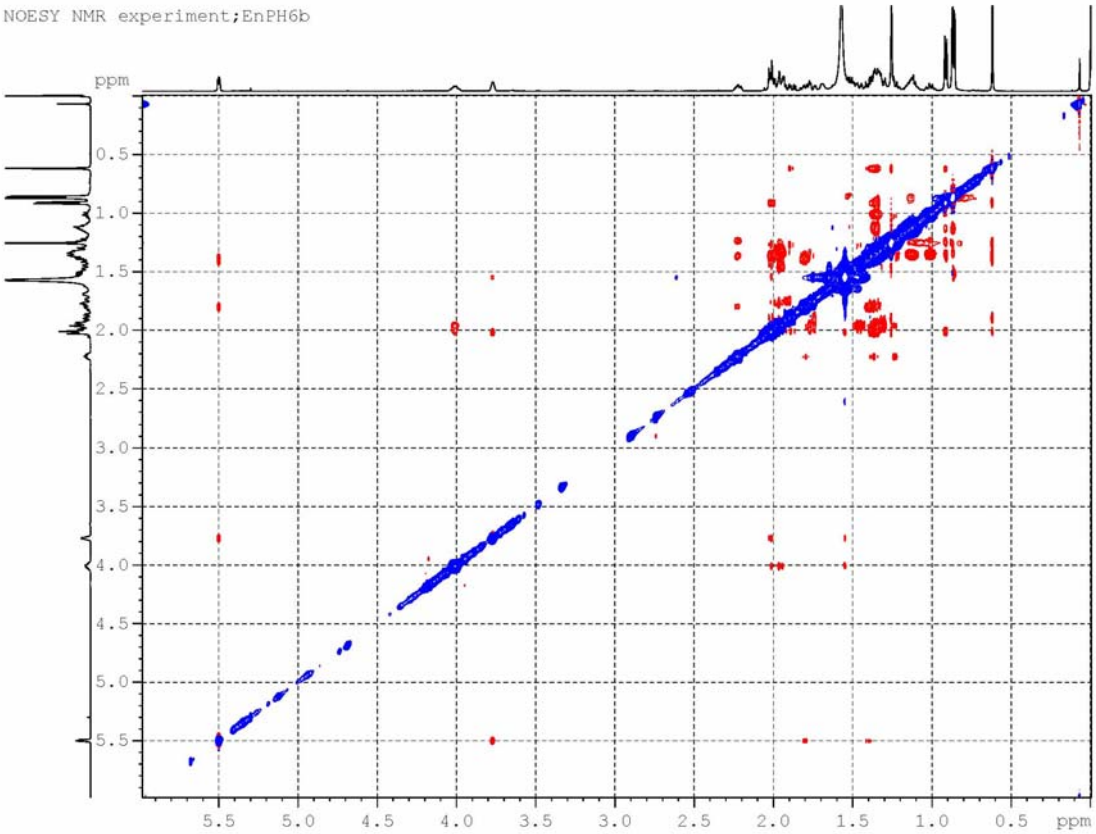
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4.5
4.0
3.5
3.0
2.5
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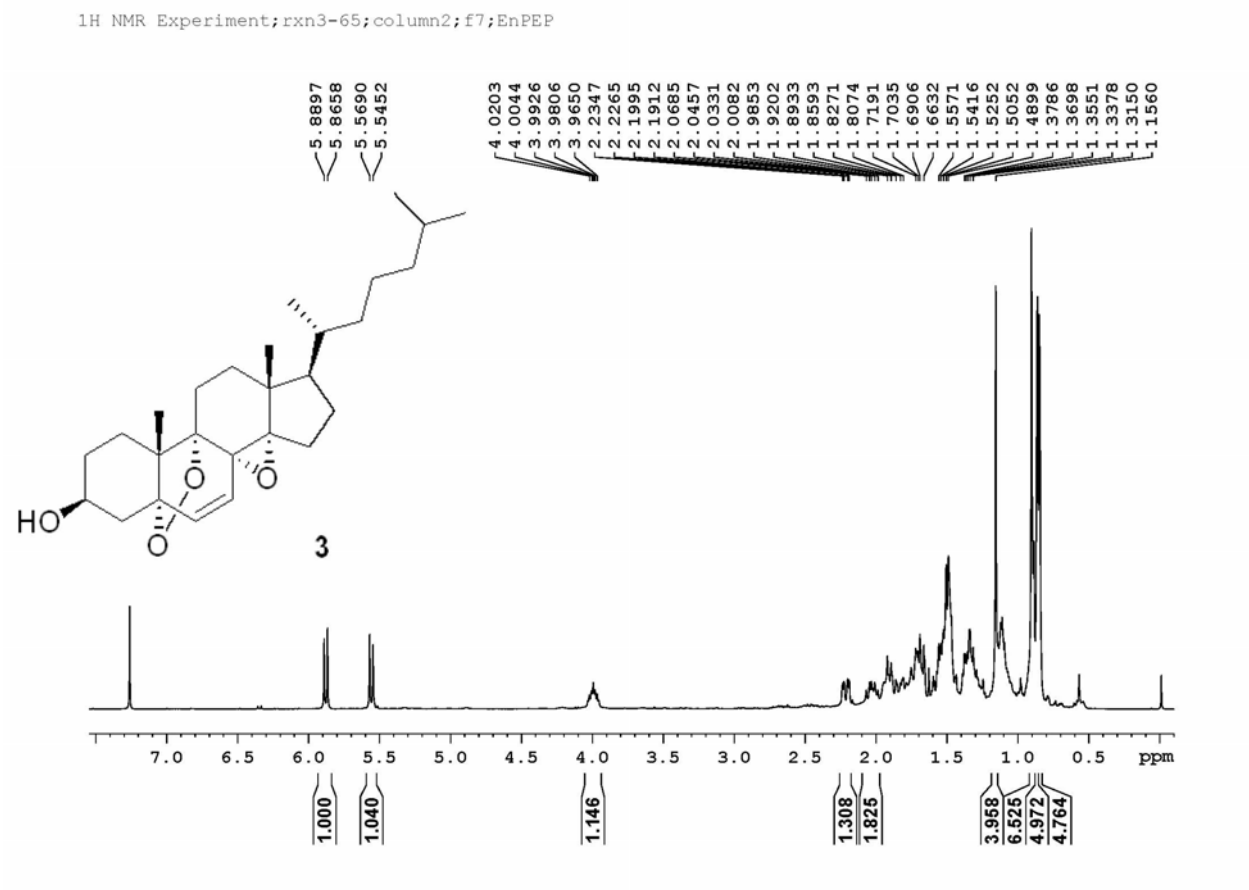
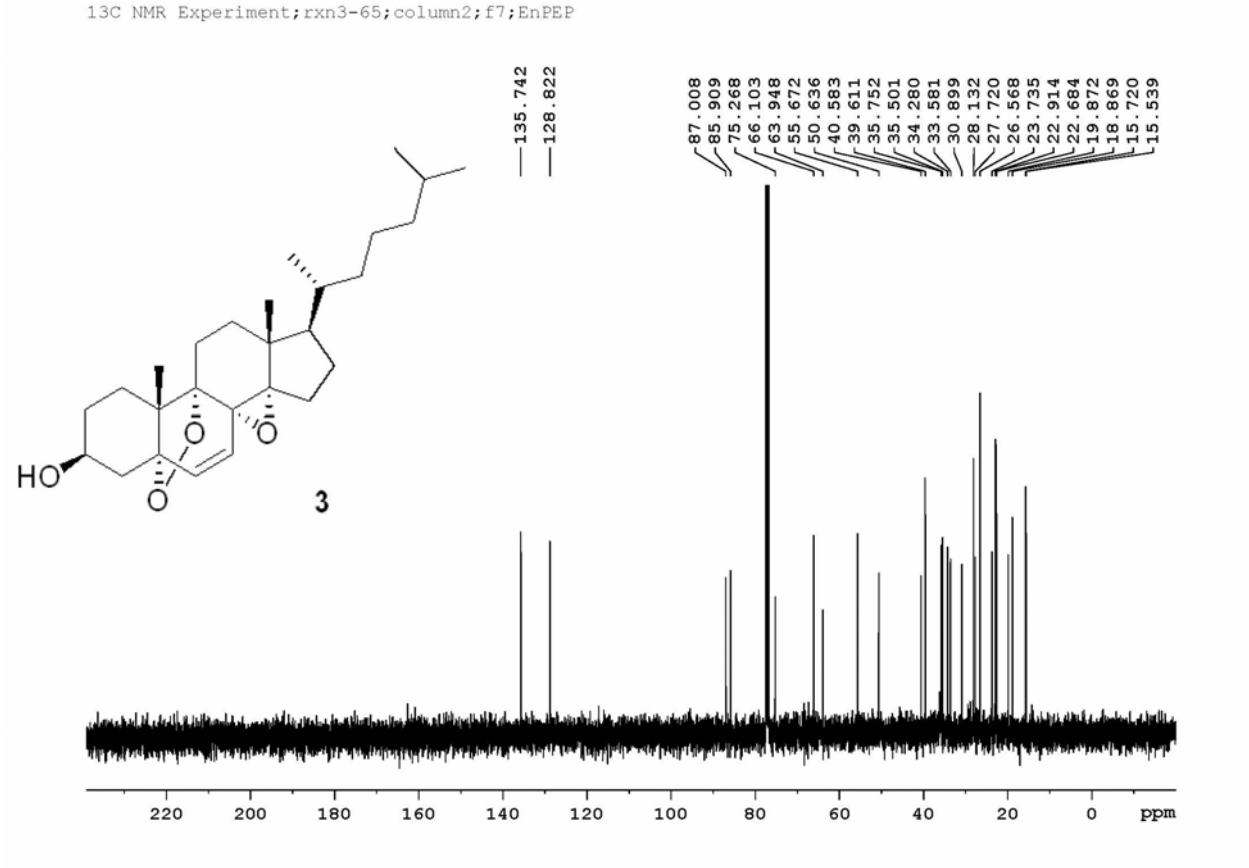
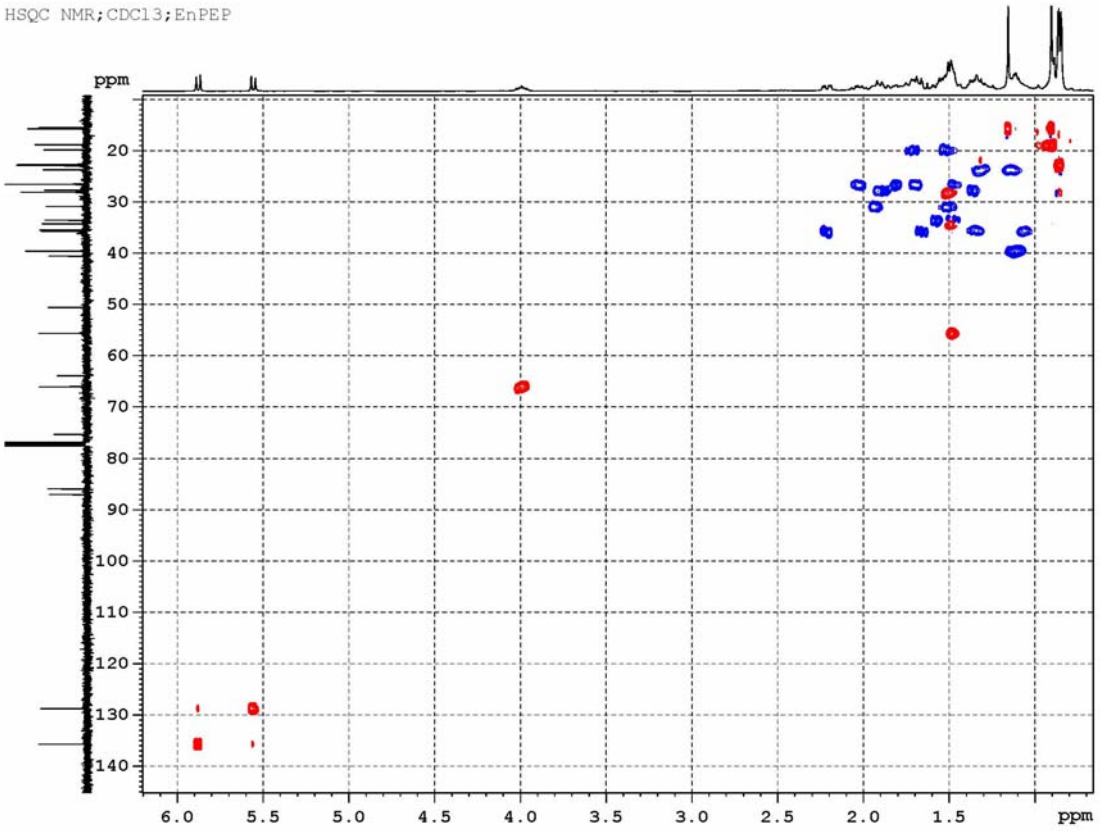
1.0
0.5
ppm

5α,9α-Epidioxy-8α,14α-epoxycholesta-6-en-3β-ol (3).

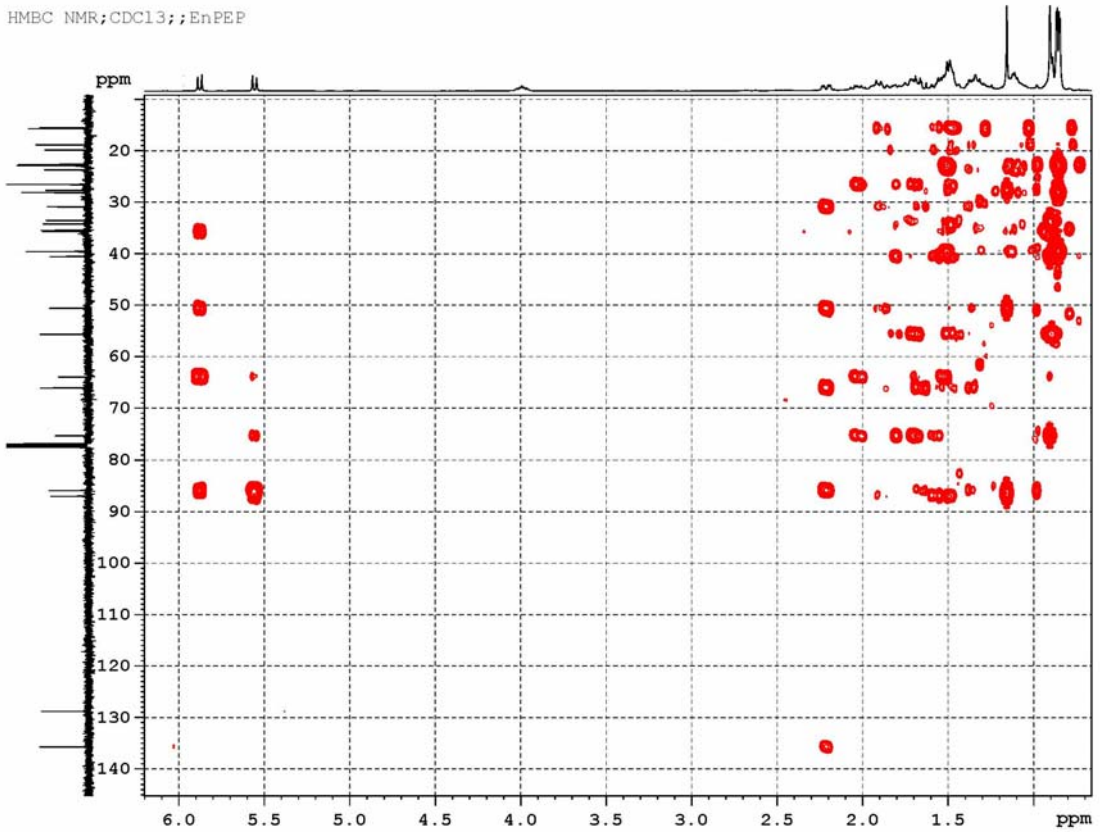




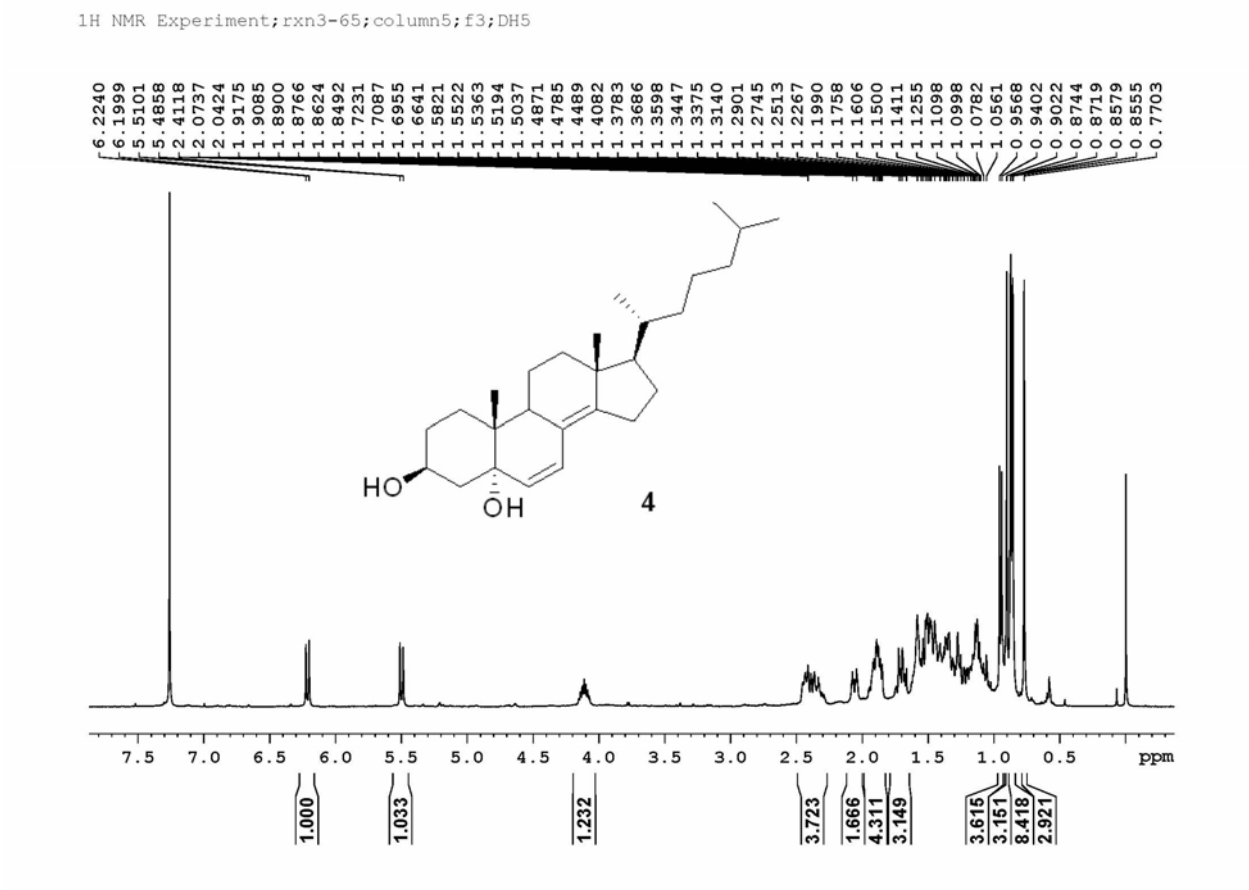
HSQC NMR;CDCl3;EnPEP

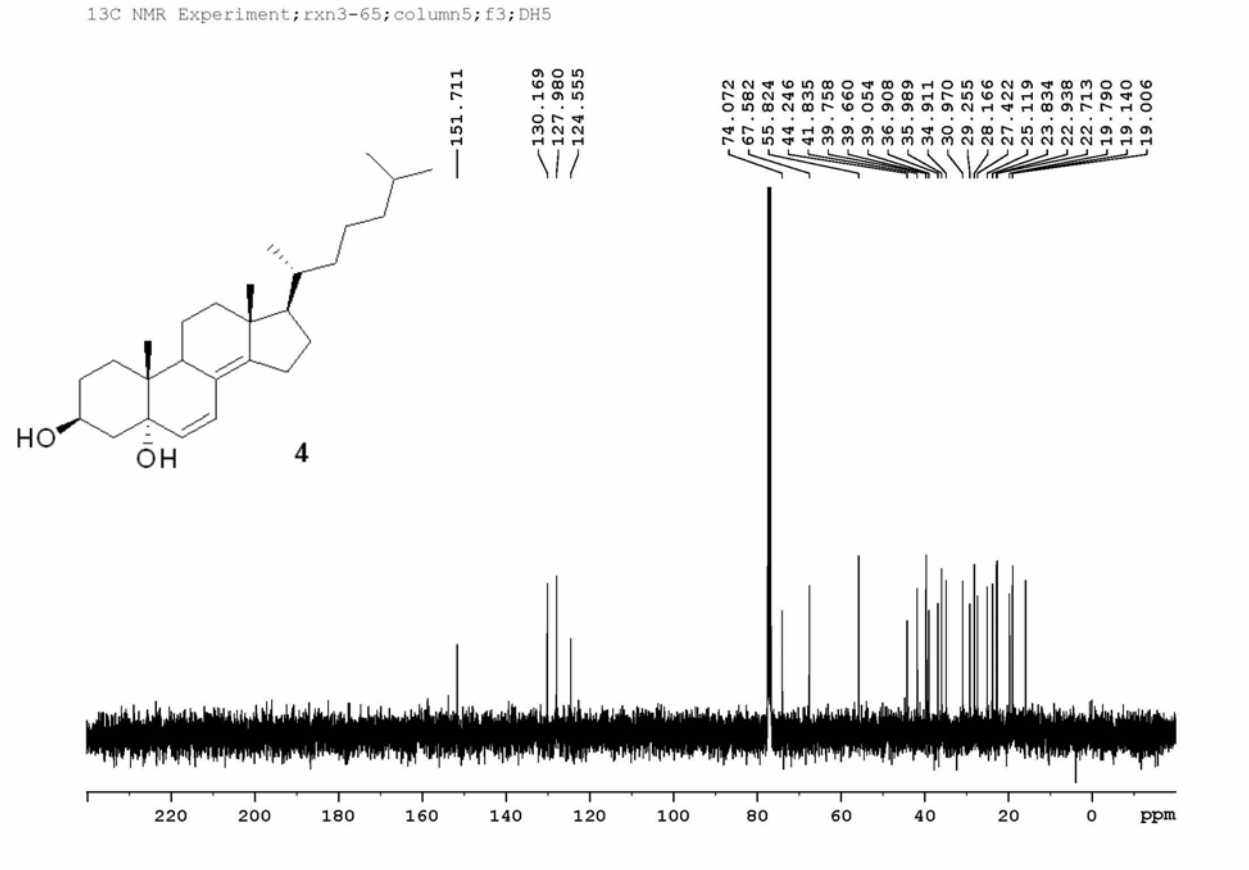


HMBC NMR;CDCl3;;EnPEP

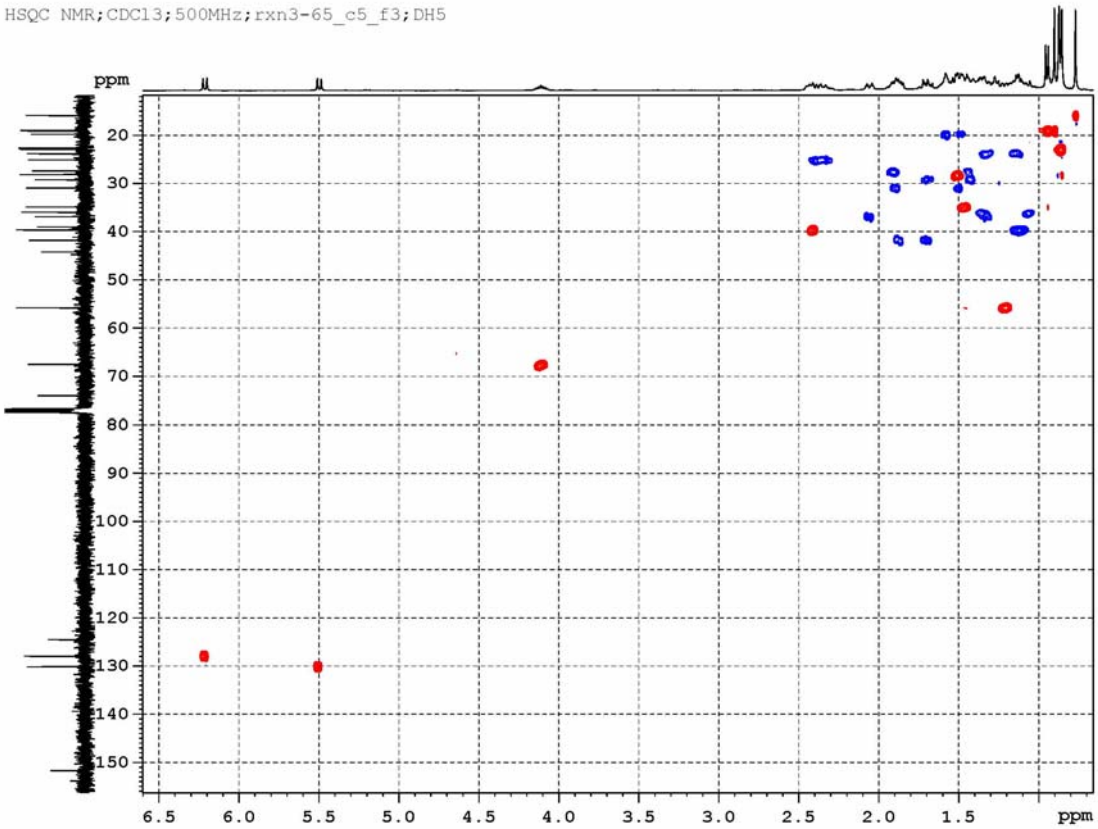


Cholesta-6,8(14)-dien-3β,5α-diol (4).

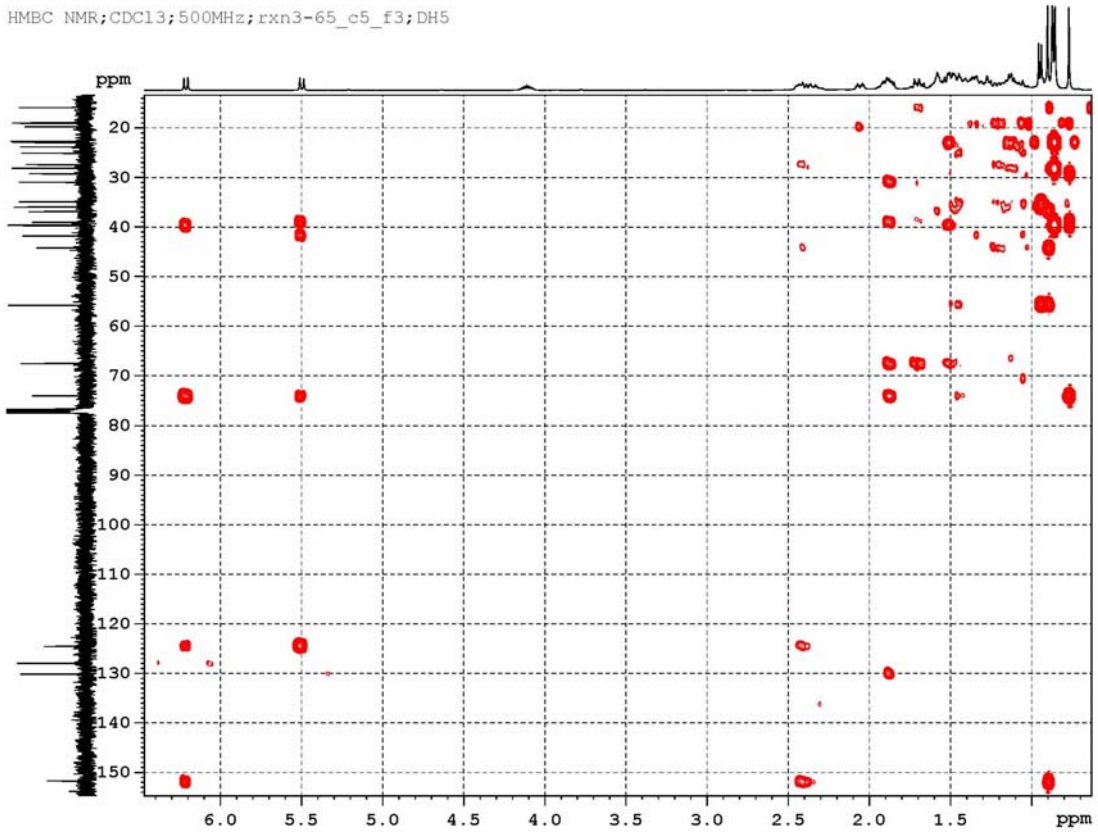


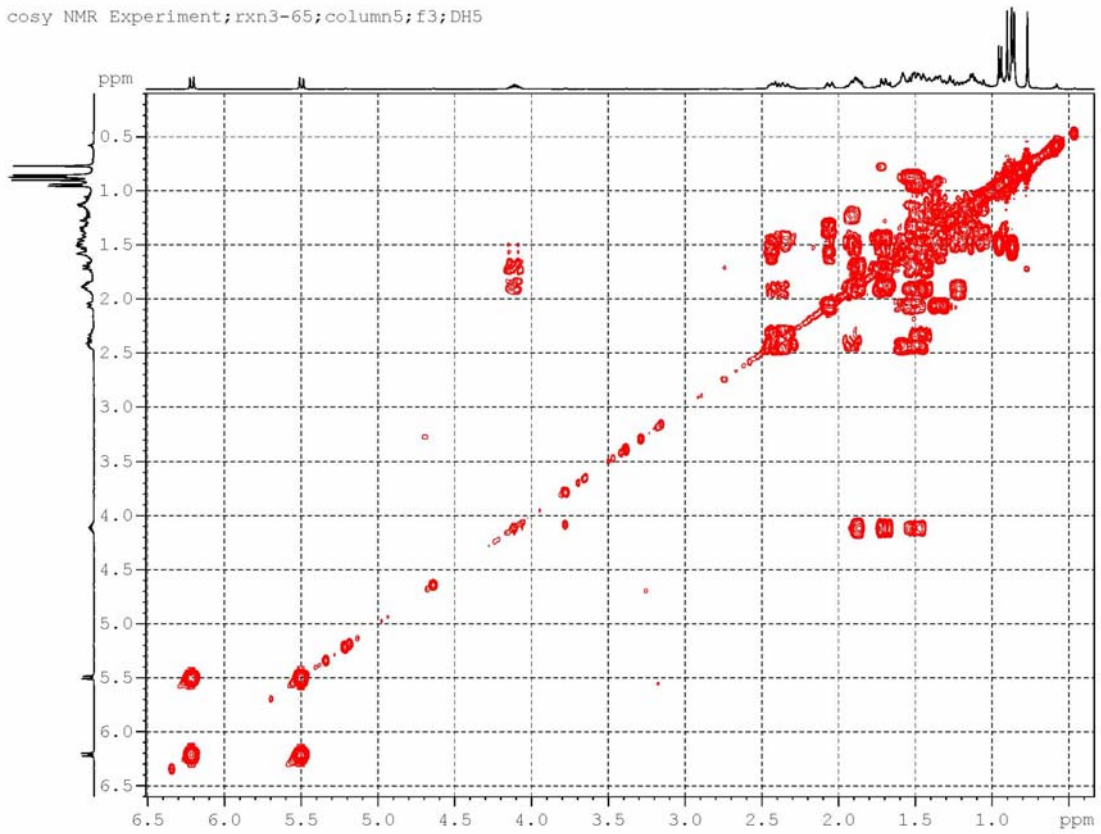


HSQC NMR;CDCl3;500MHz;rxn3-65_c5_f3;DH5

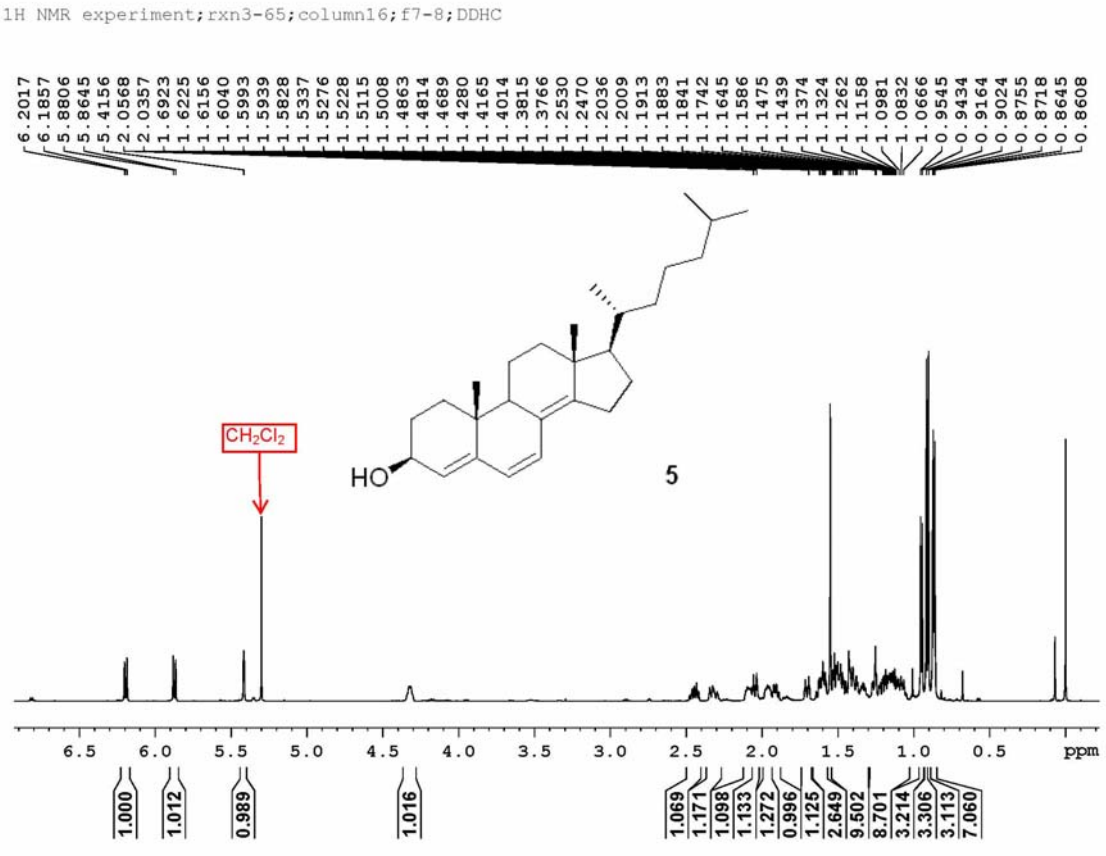


HMBC NMR;CDCl3;500MHz;rxn3-65_c5_f3;DH5

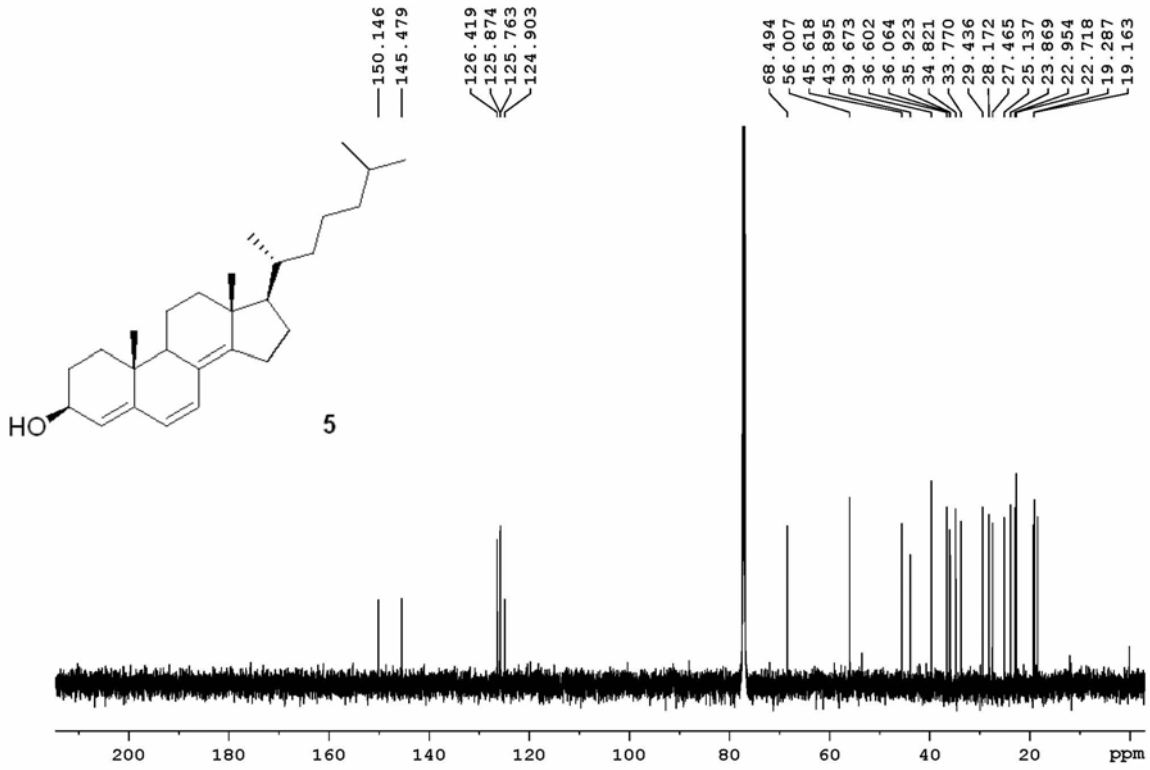




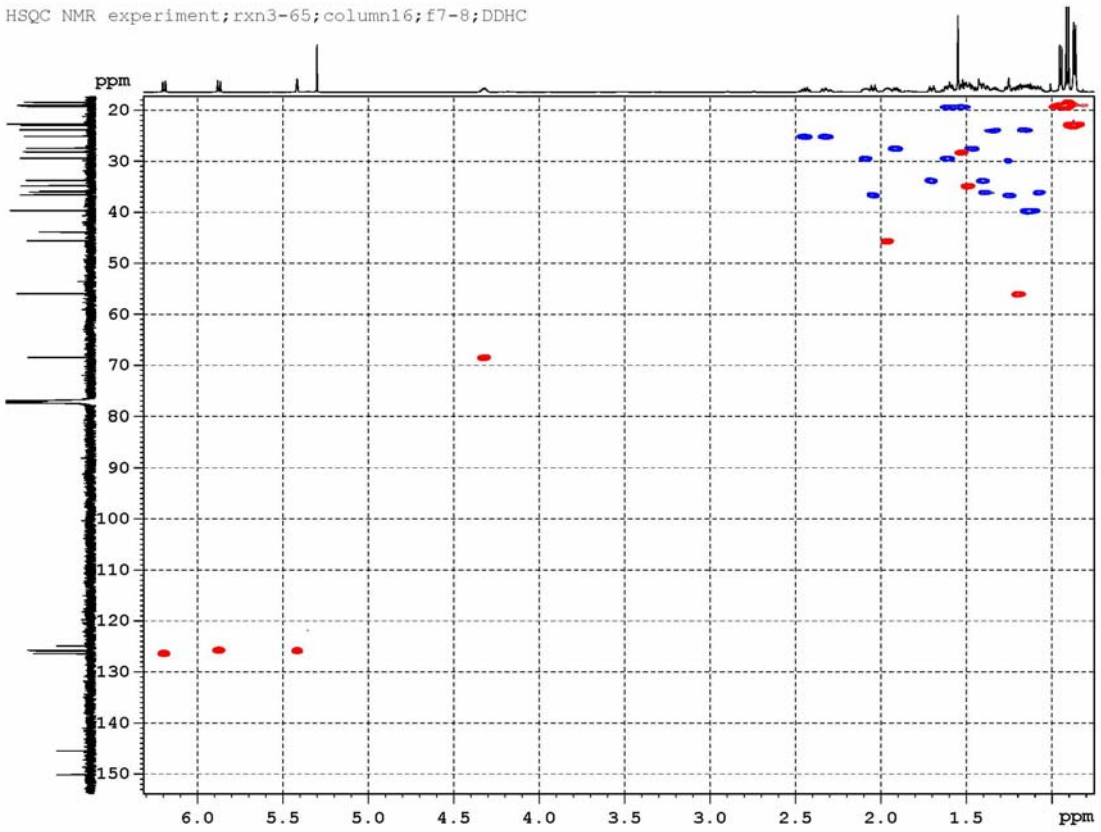
Cholesta-4,6,8(14)-trien-3β-ol (5).

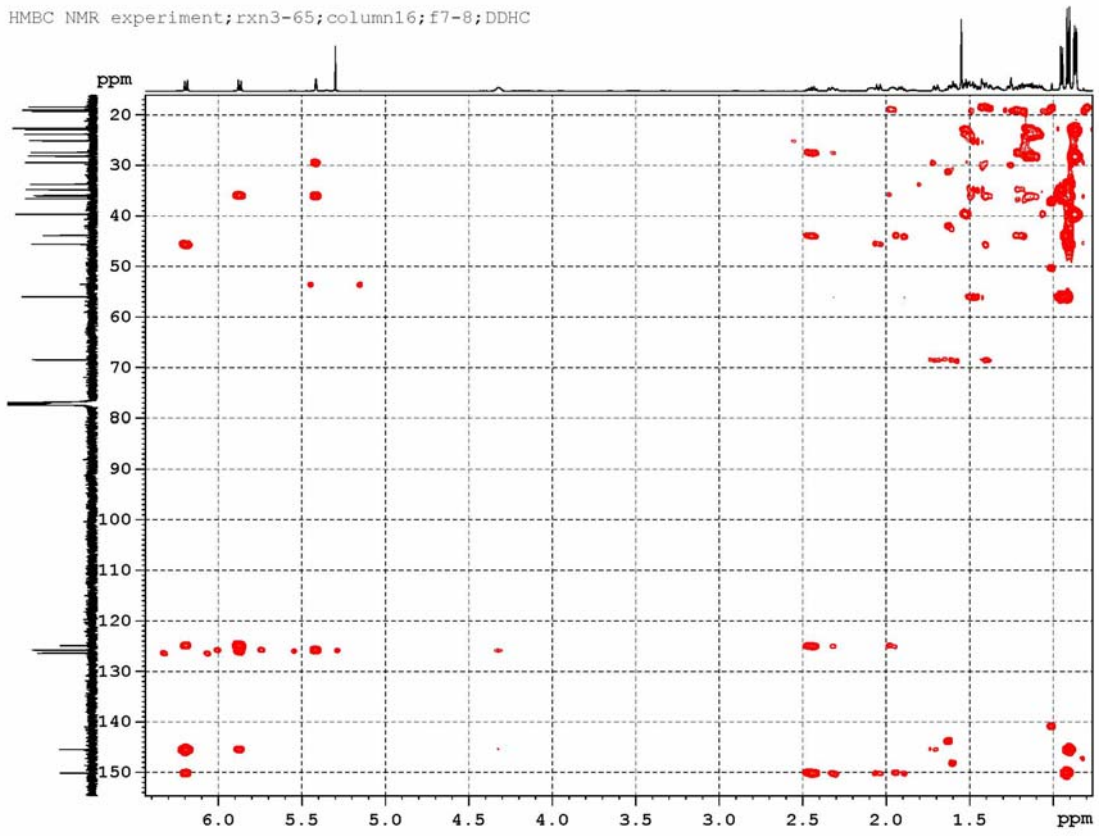


13C NMR experiment;rxn3-65;column16;f7-8;DDHC

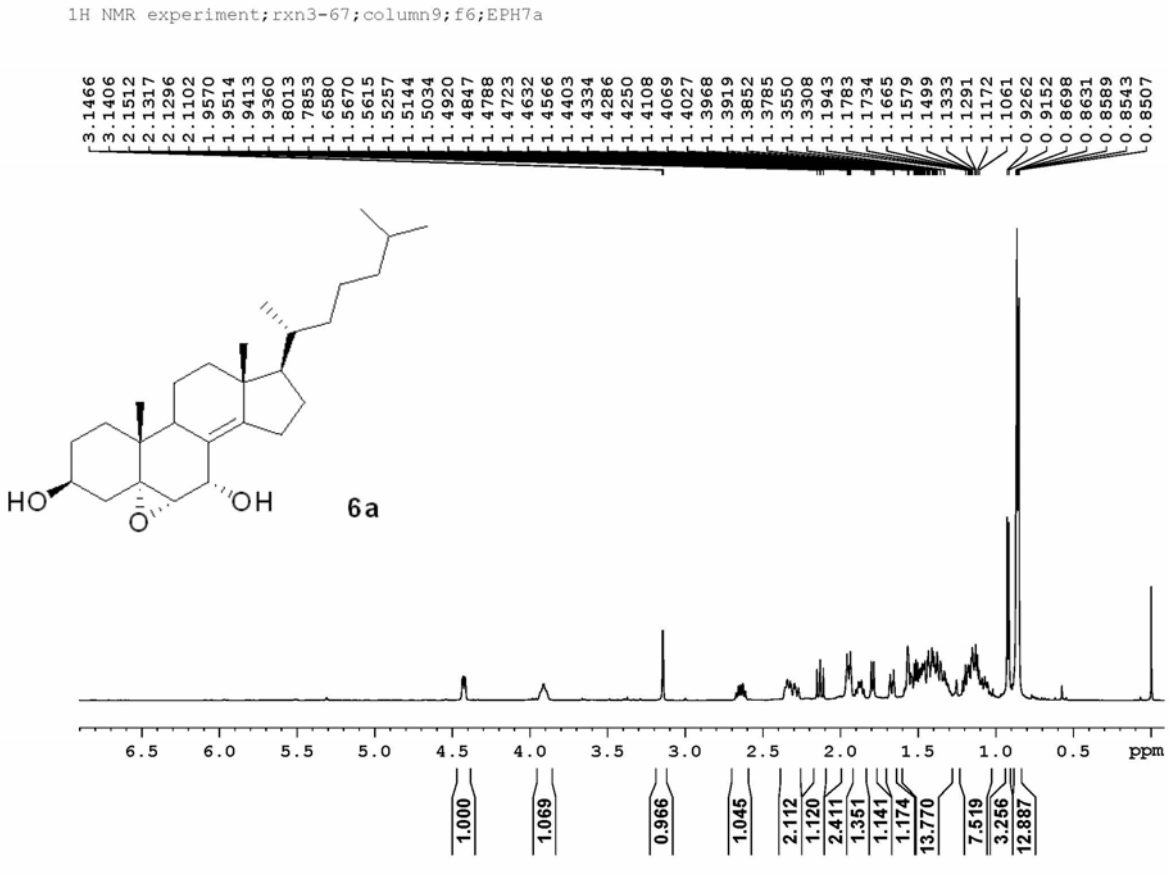


HSQC NMR experiment;rxn3-65;column16;f7-8;DDHC

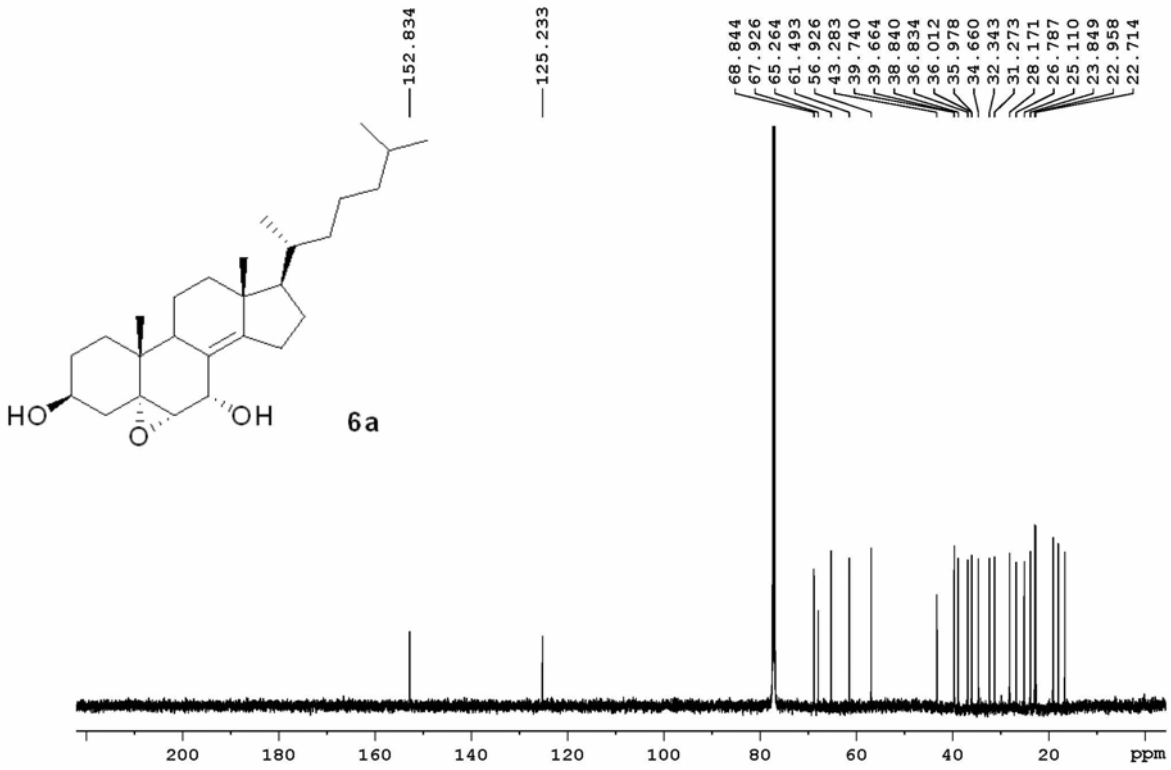




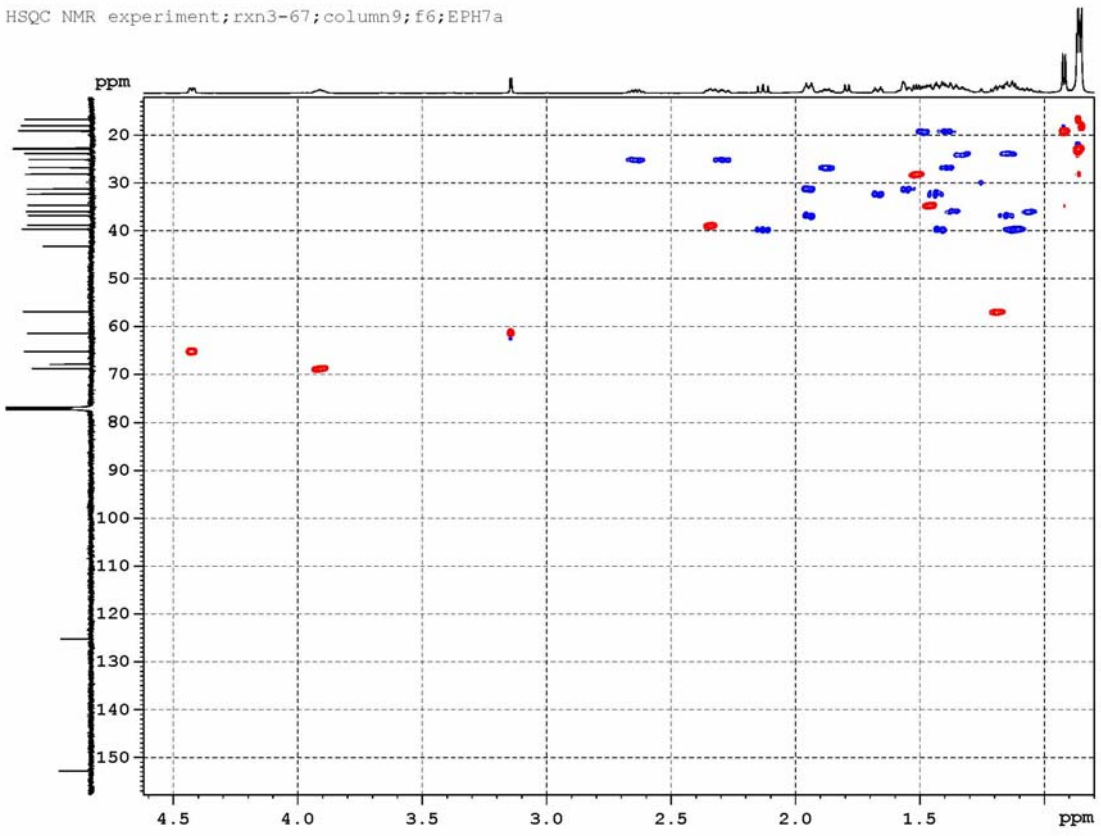
5α,6α-Epoxycholesta-8(14)-en-3β,7α-diol (6a).



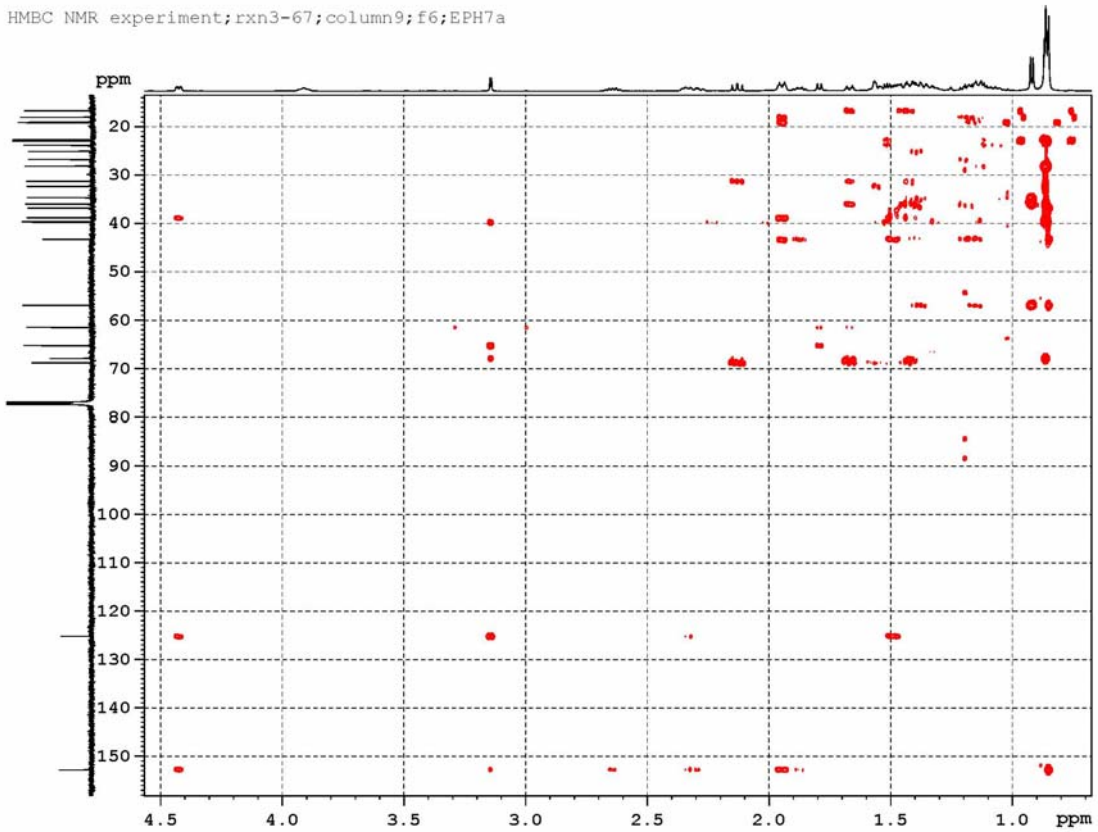
13C NMR experiment;rxn3-67;column9;f6;EPH7a



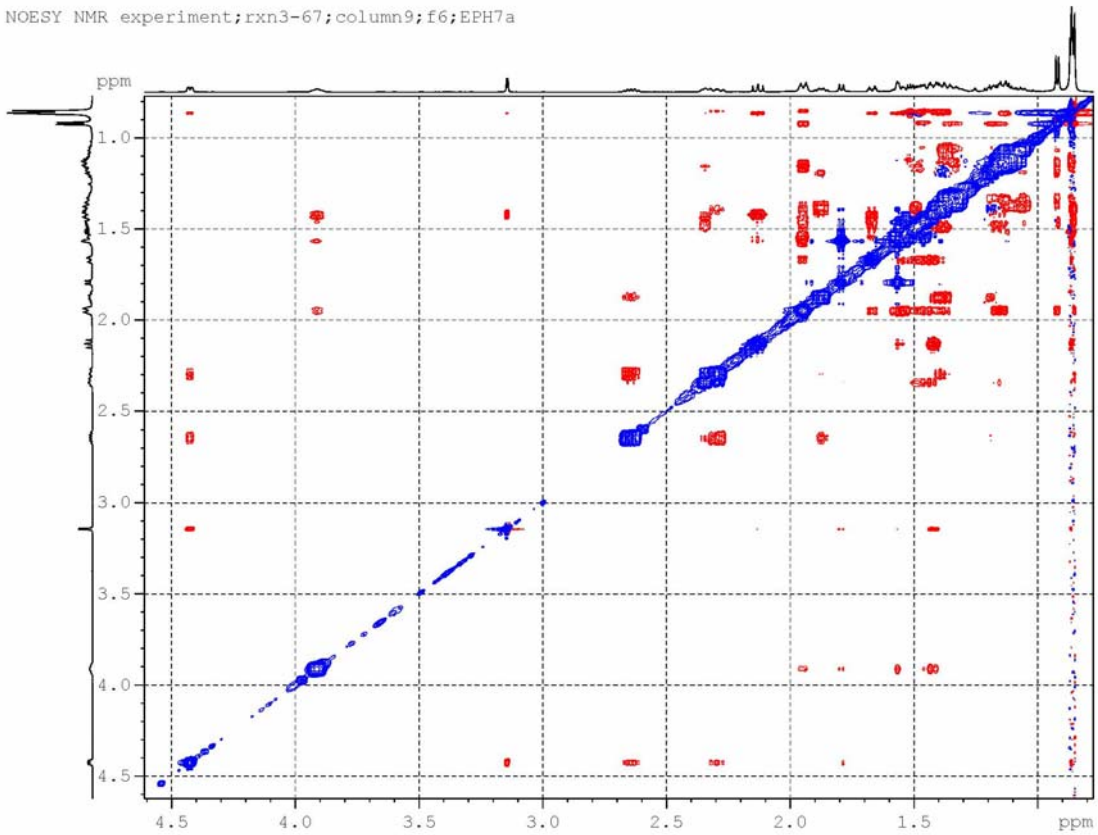
HSQC NMR experiment;rxn3-67;column9;f6;EPH7a



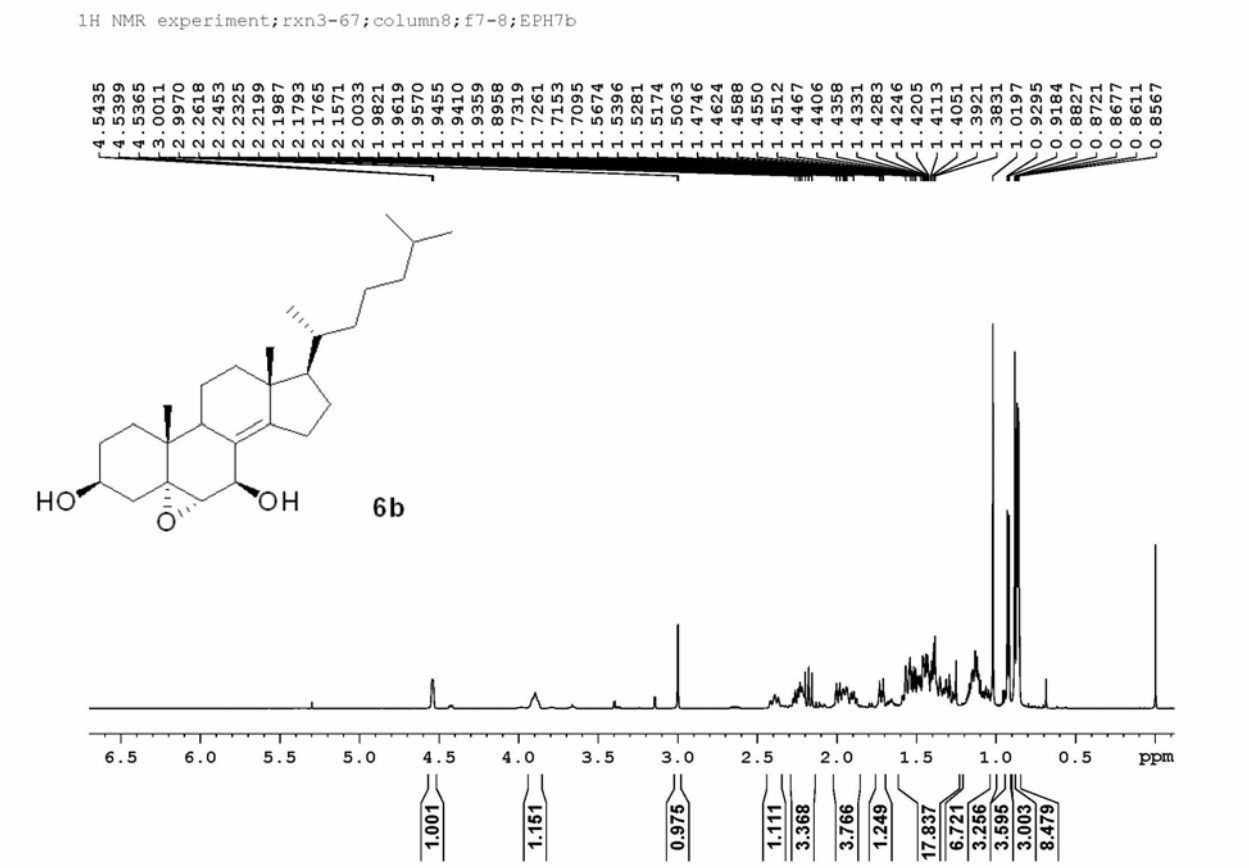
HMBC NMR experiment;rxn3-67;column9;f6;EPH7a

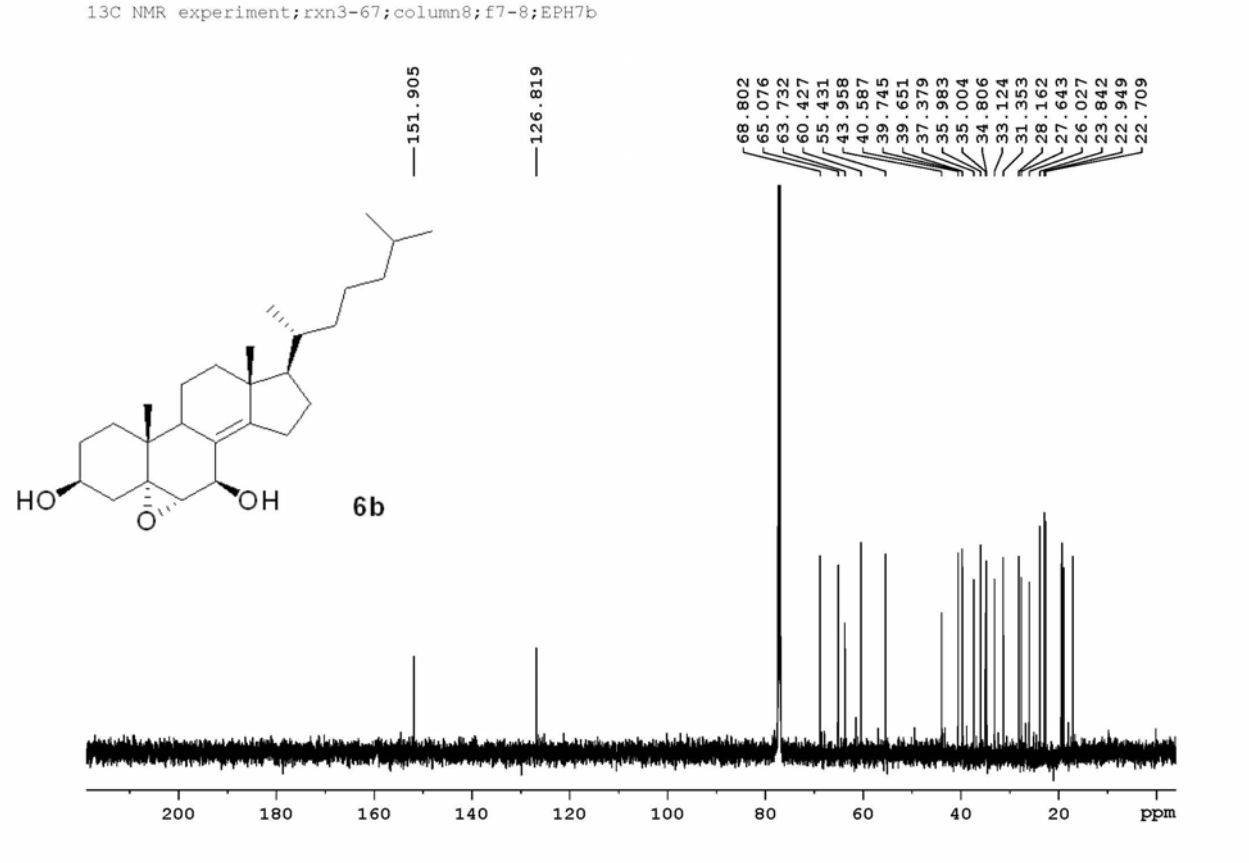


NOESY NMR experiment;rxn3-67;column9;f6;EPH7a

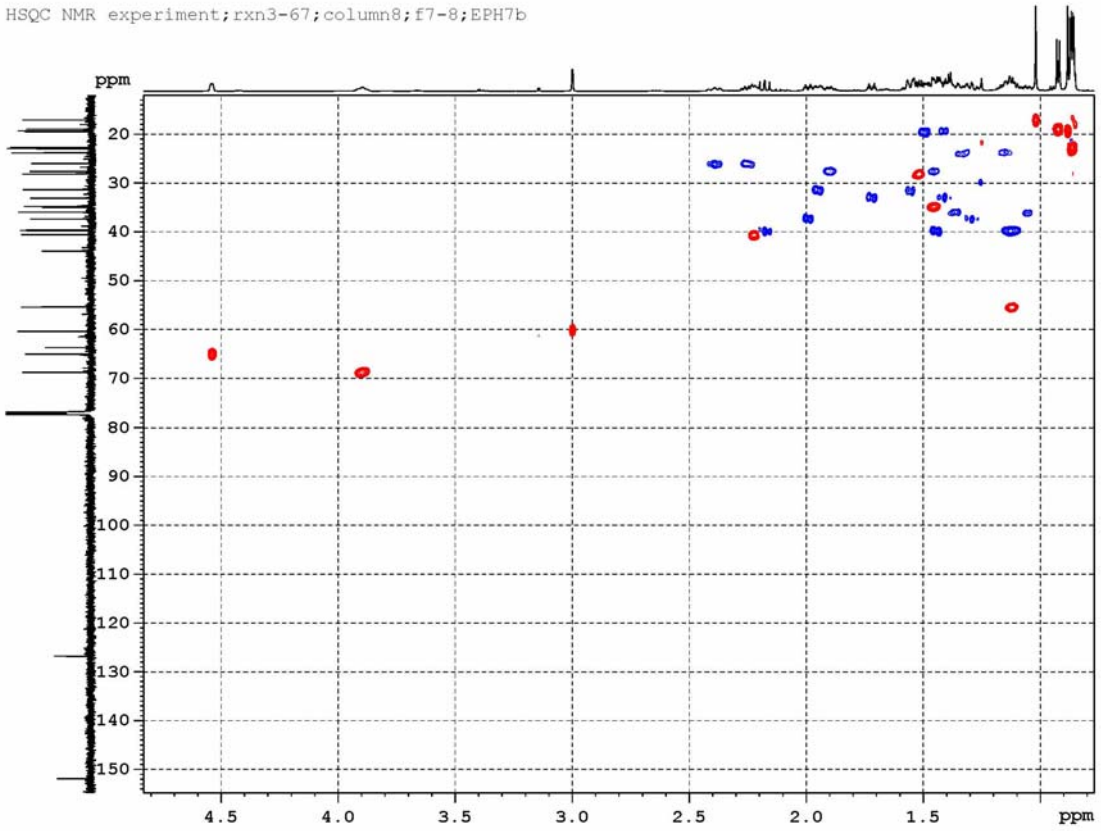


5α,6α-Epoxycholesta-8(14)-en-3β,7β-diol (6b).

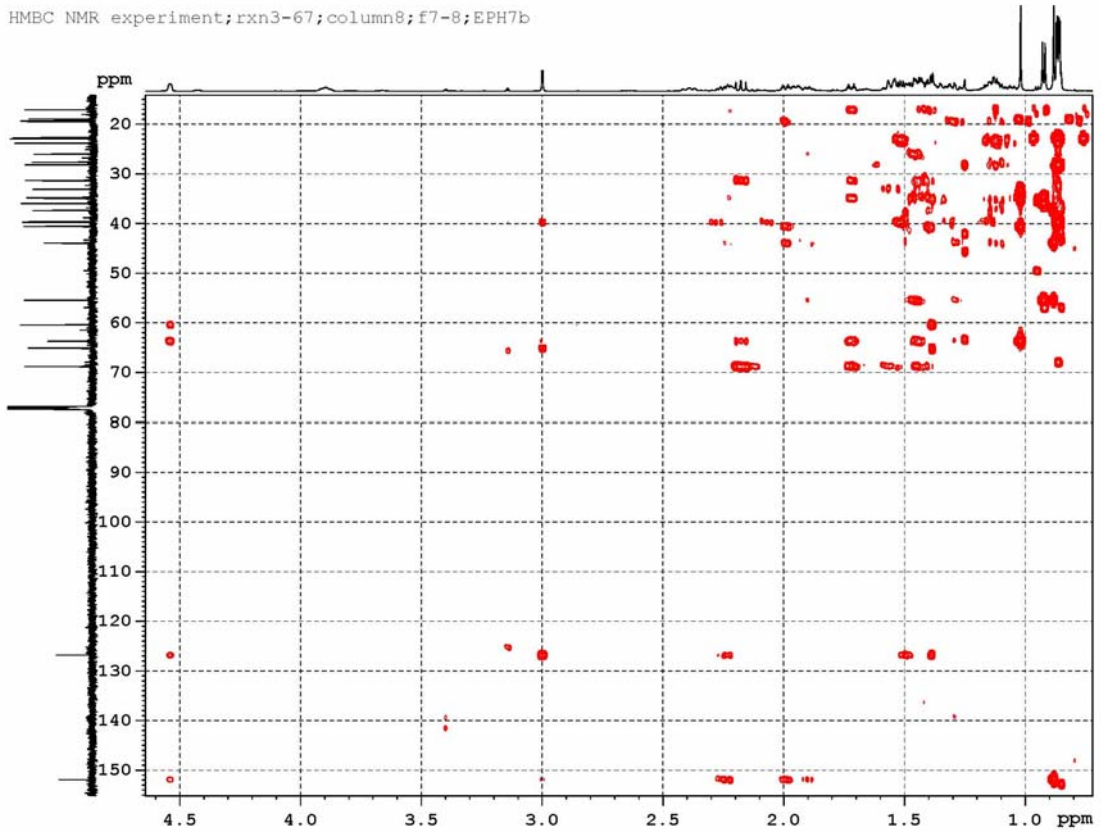


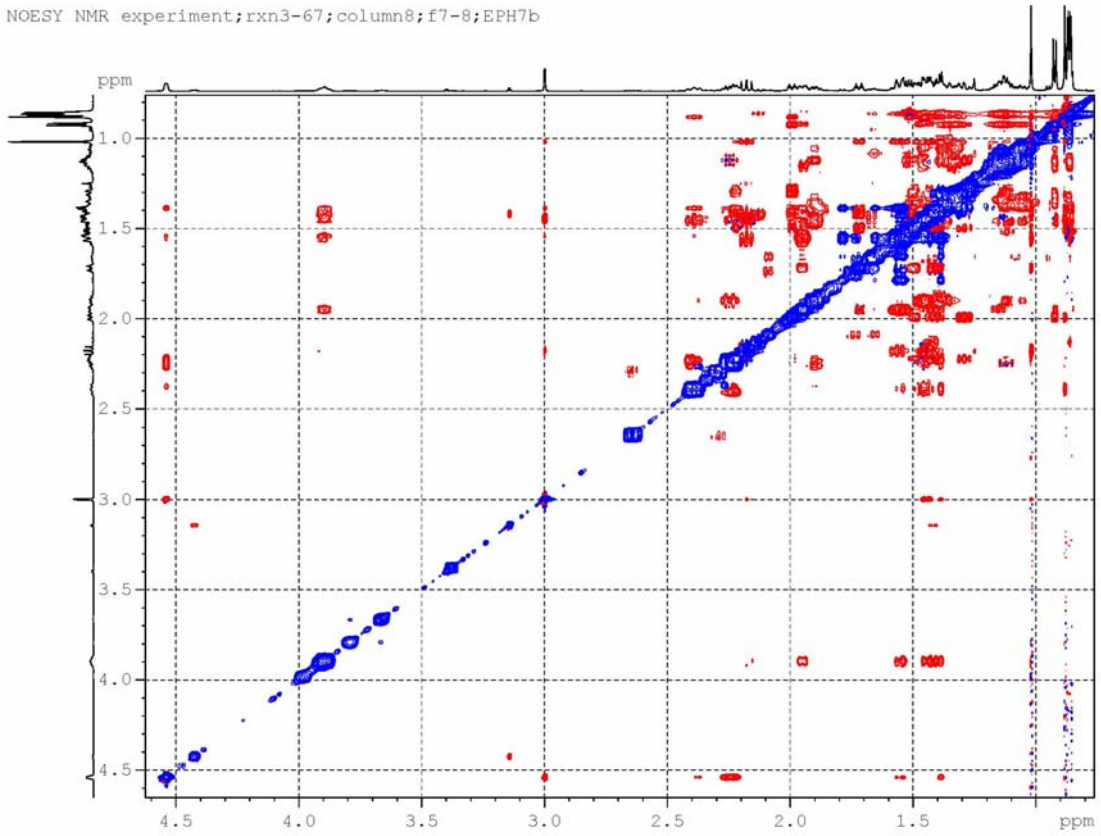


HSQC NMR experiment;rxn3-67;column8;f7-8;EPH7b

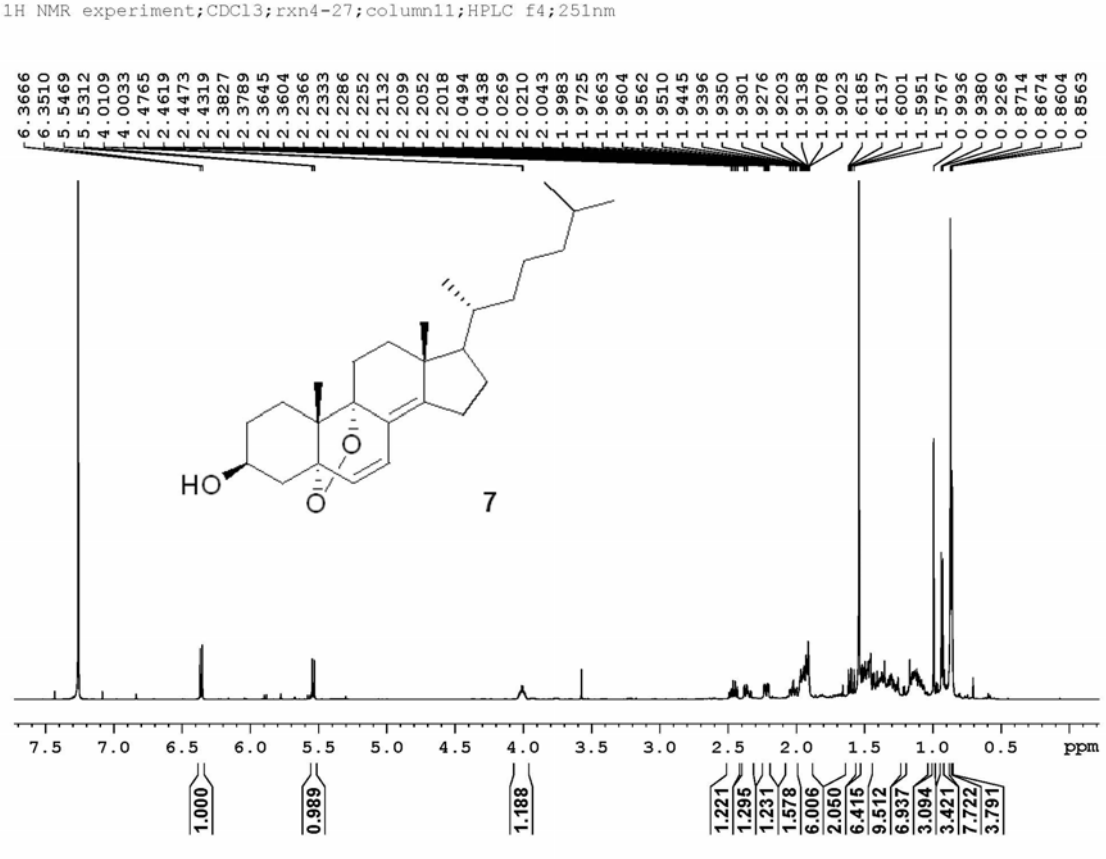


HMBC NMR experiment;rxn3-67;column8;f7-8;EPH7b

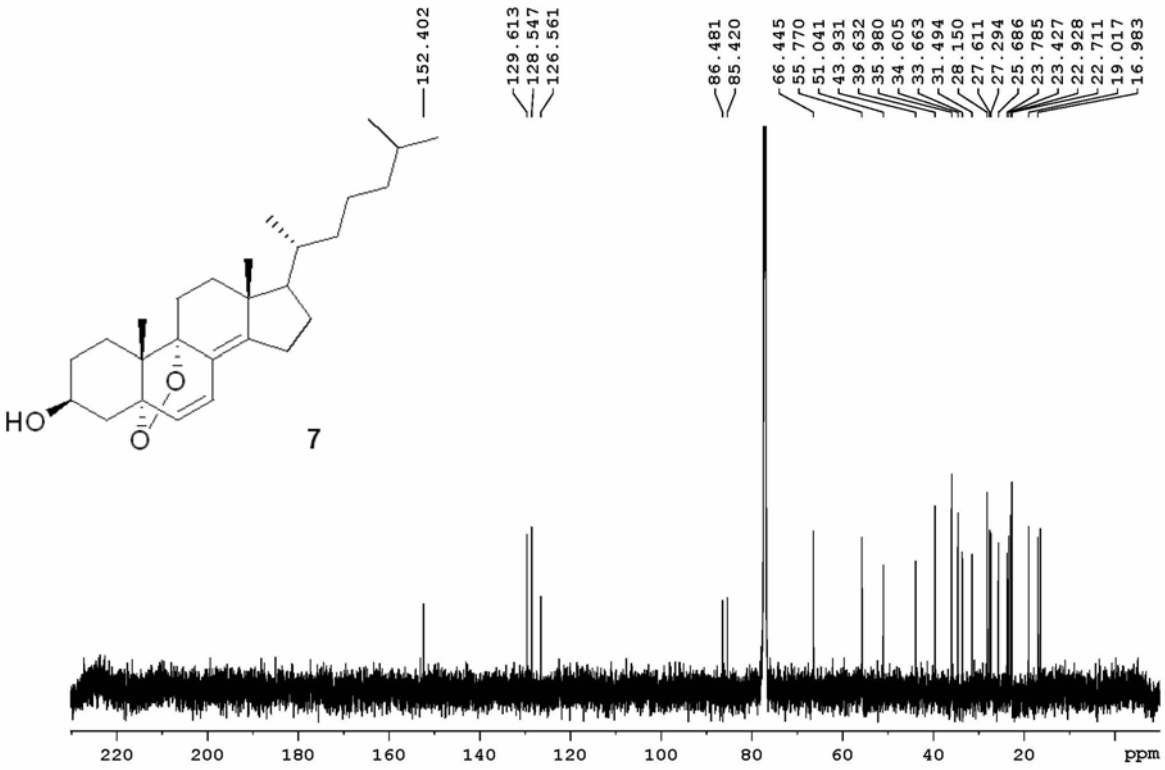




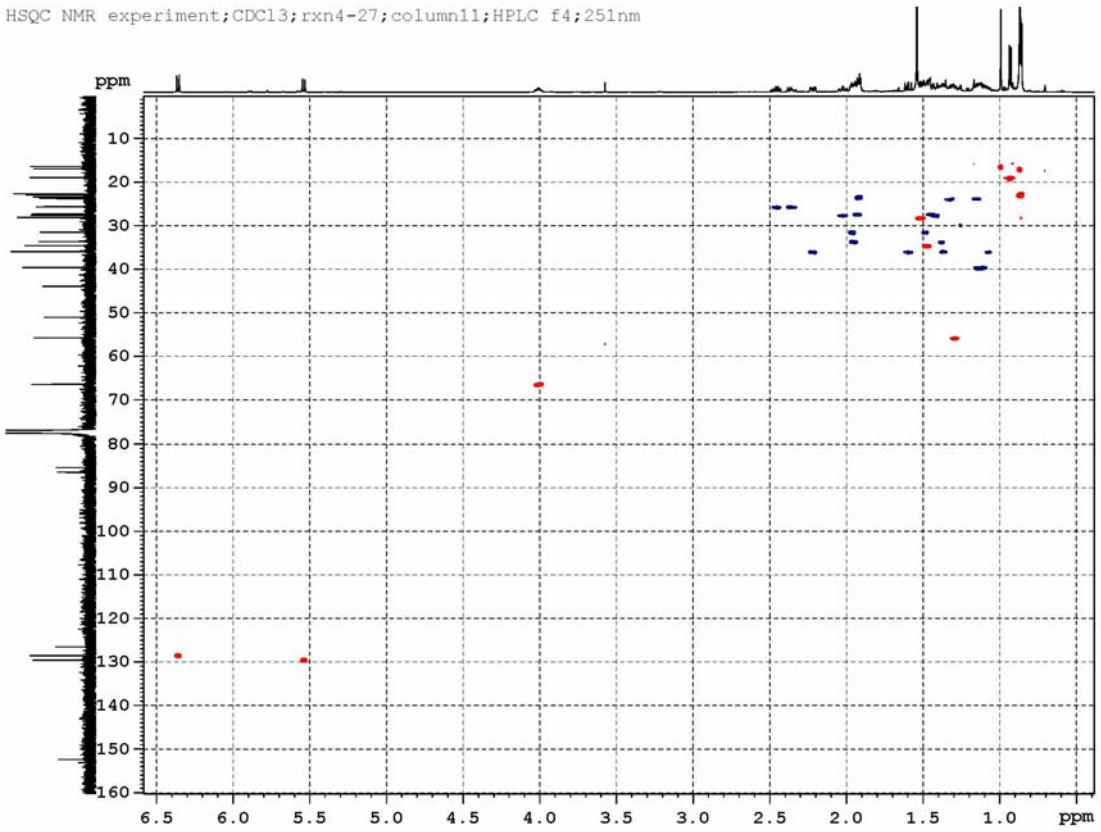
5α,9α-Epidioxycholesta-6,8(14)-dien-3β-ol (7).

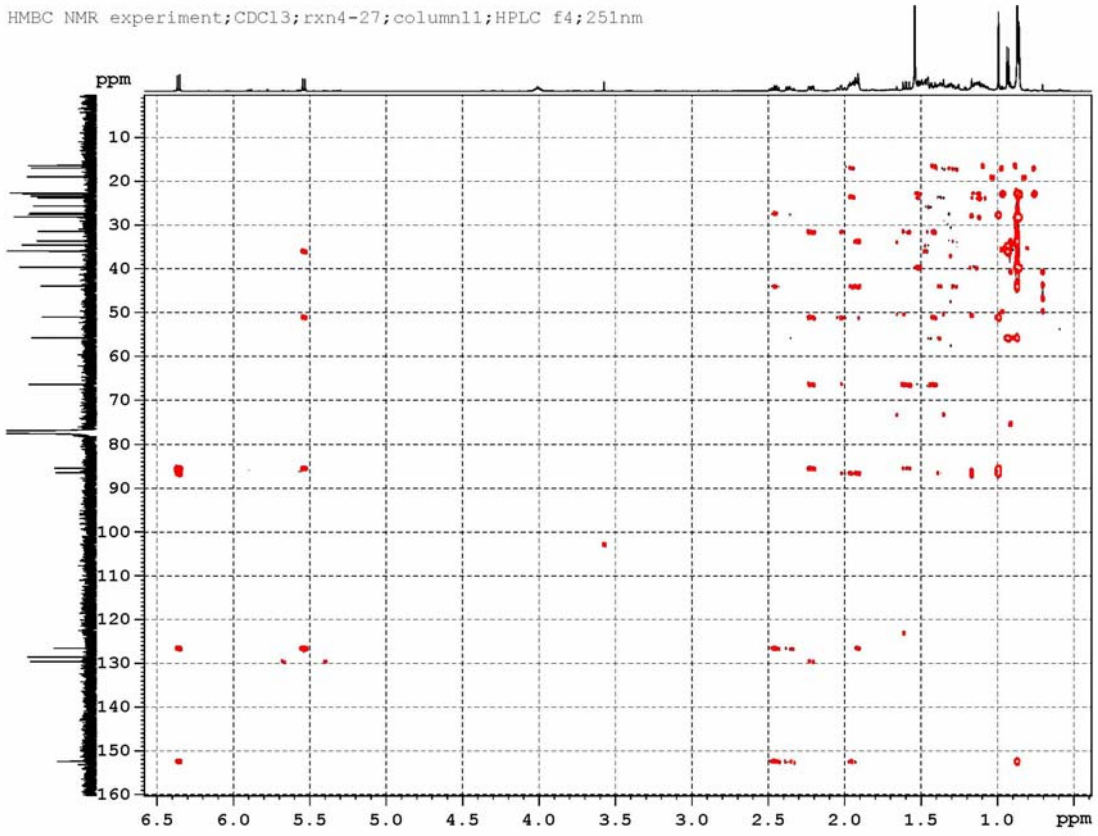


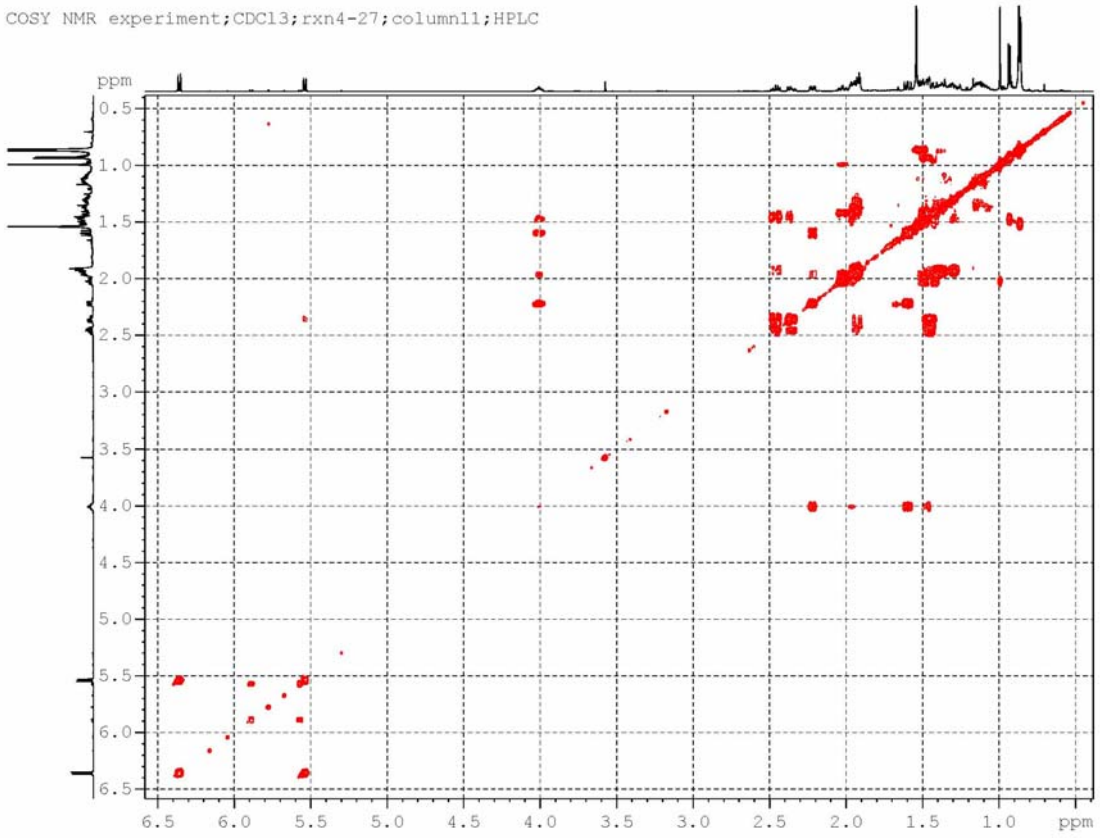
13C NMR experiment;CDCl3;rxn4-27;column11;HPLC f4;251nm

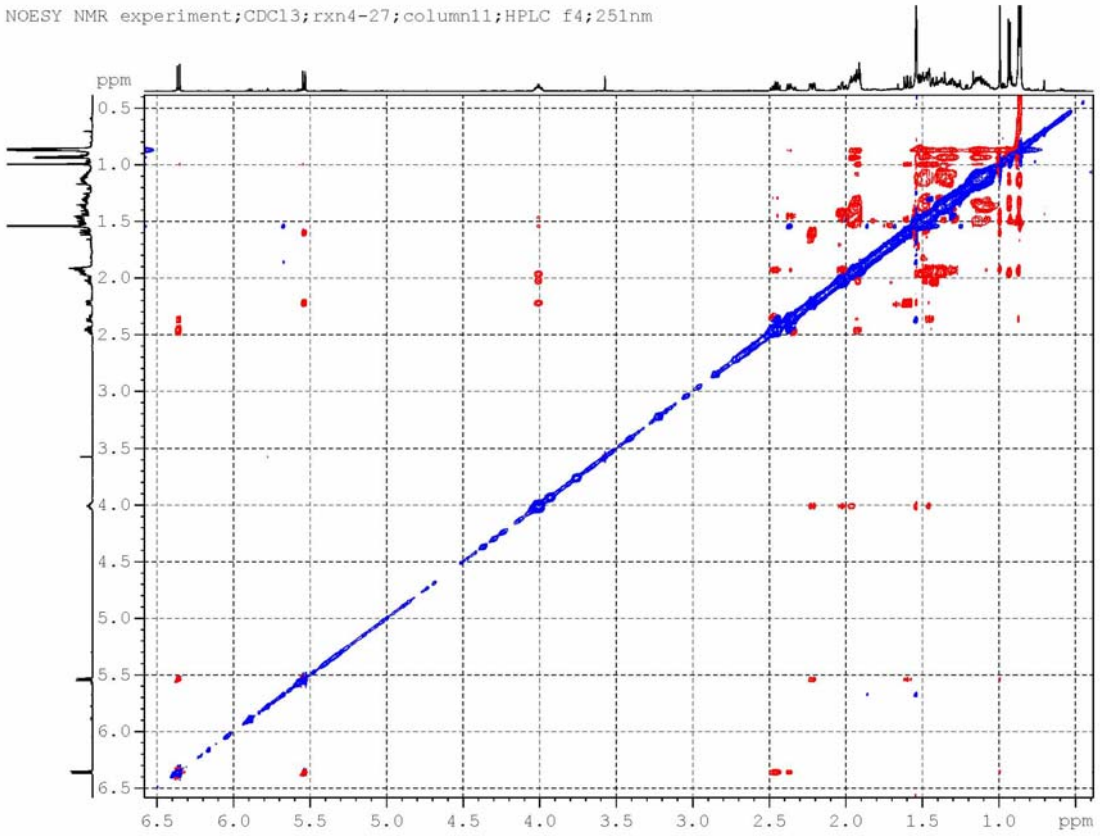


HSQC NMR experiment;CDCl3;rxn4-27;column11;HPLC f4;251nm

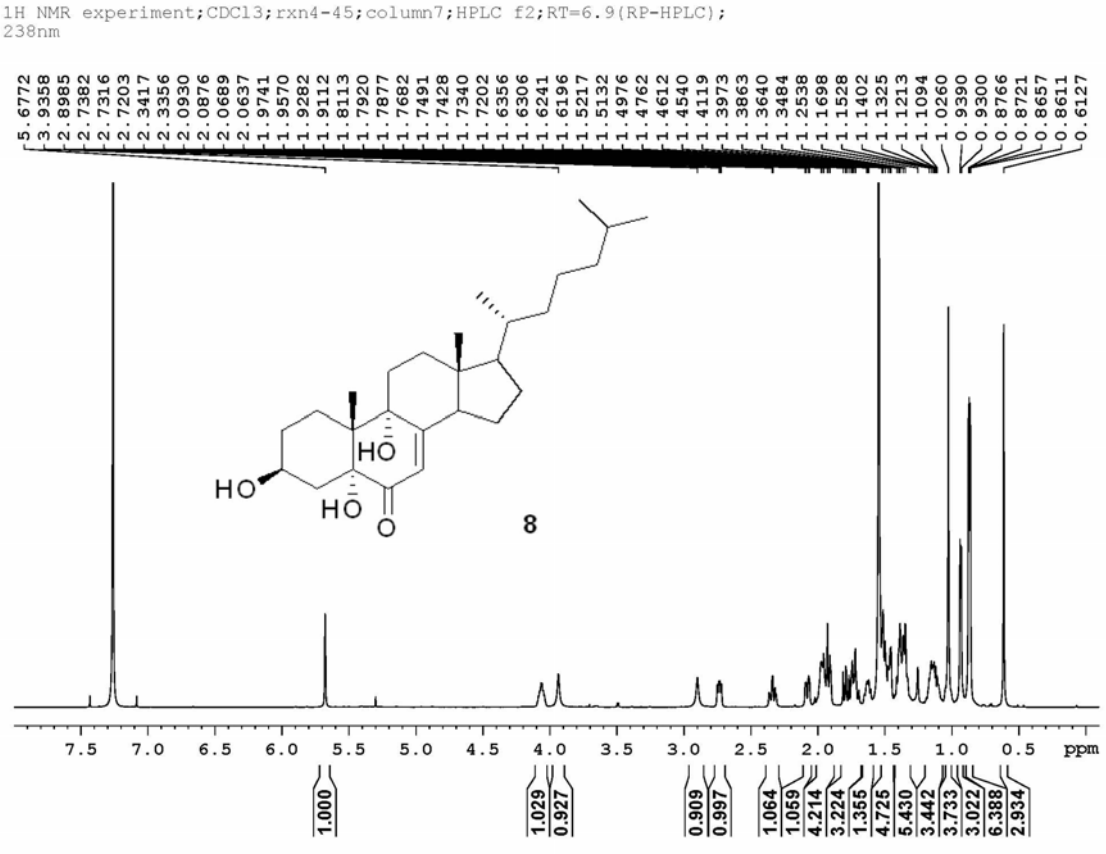



HMBC NMR experiment;CDCl3;rxn4-27;column11;HPLC f4;251nm
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

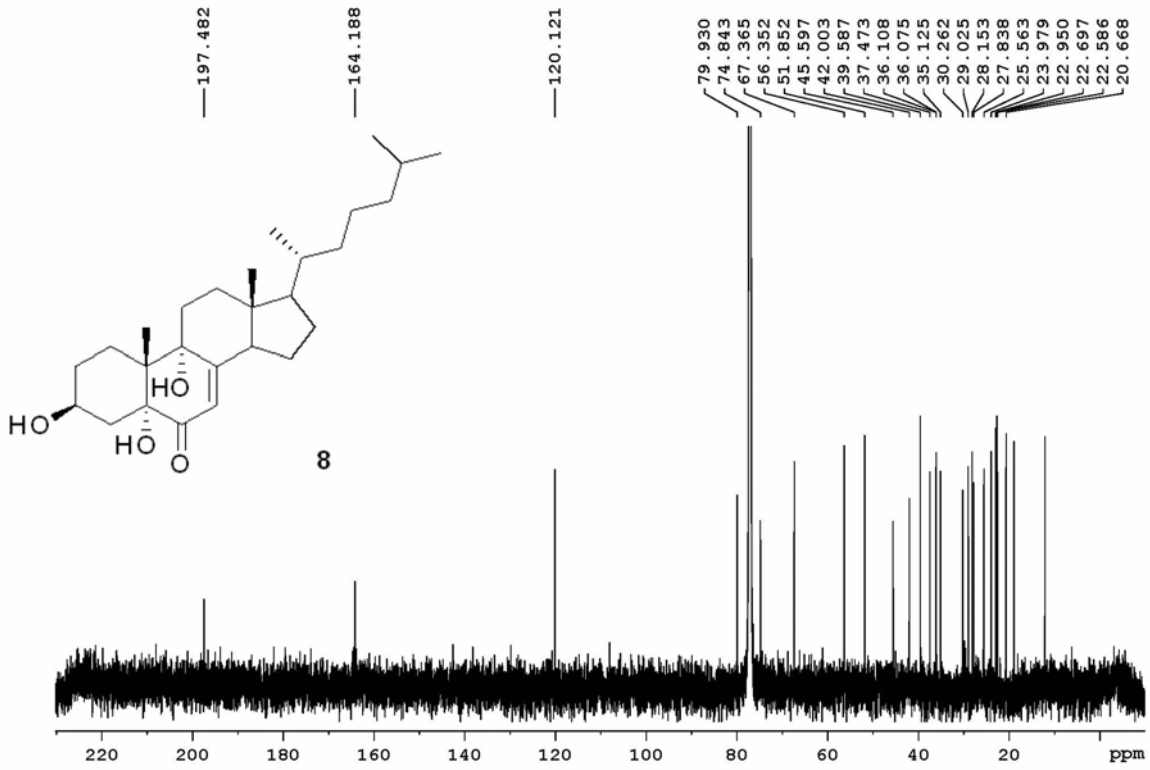

COSY NMR experiment;CDCl3;rxn4-27;column11;HPLC
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm



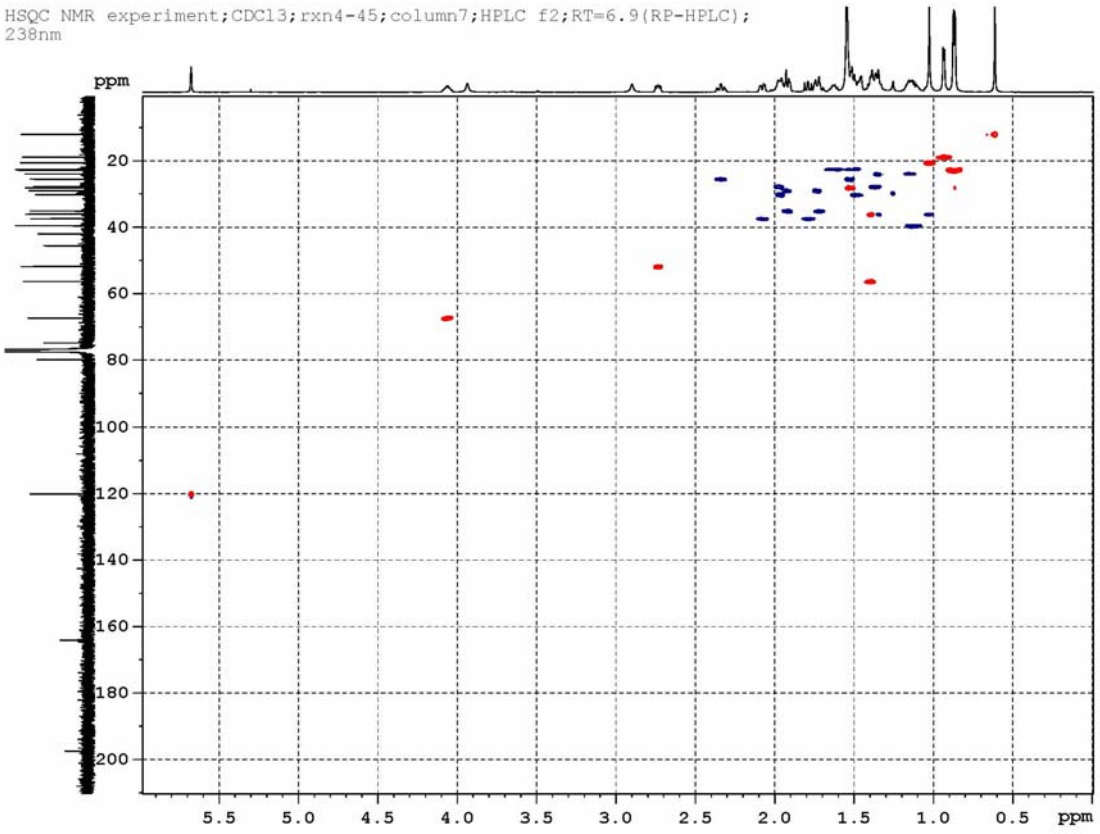
3β,5α,9α-Trihydroxycholesta-7-en-6-one (8).

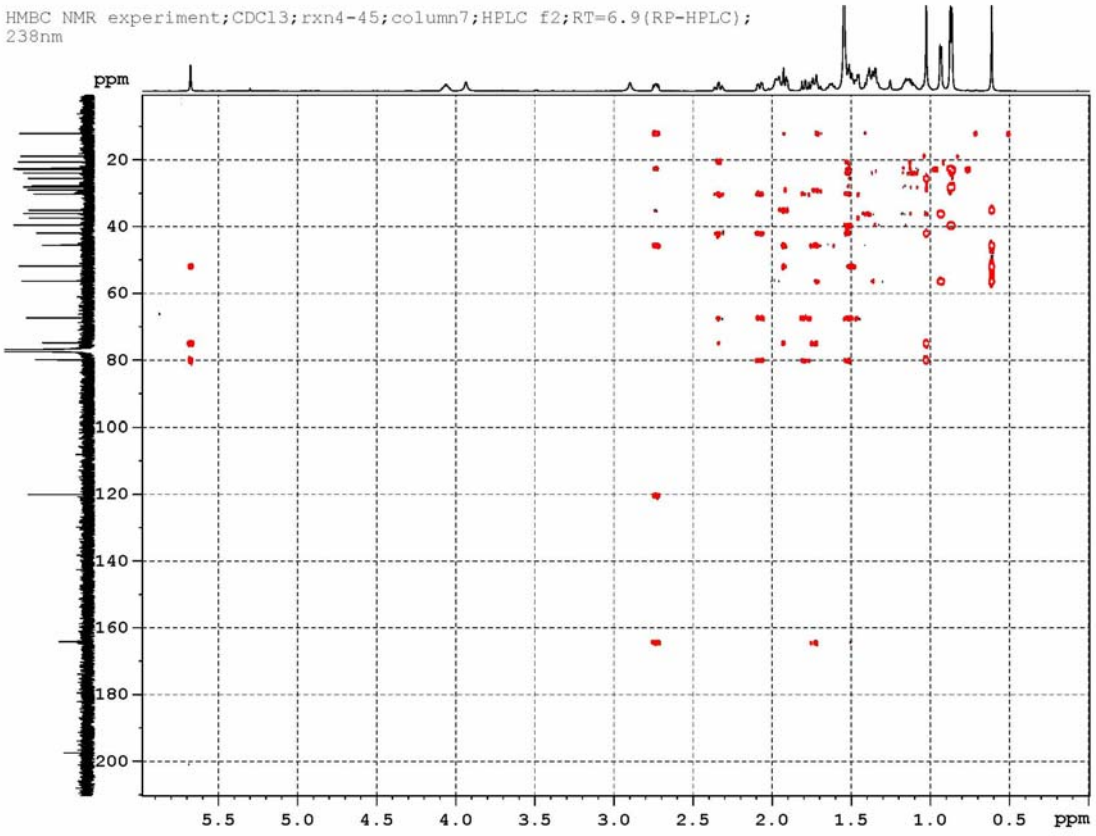


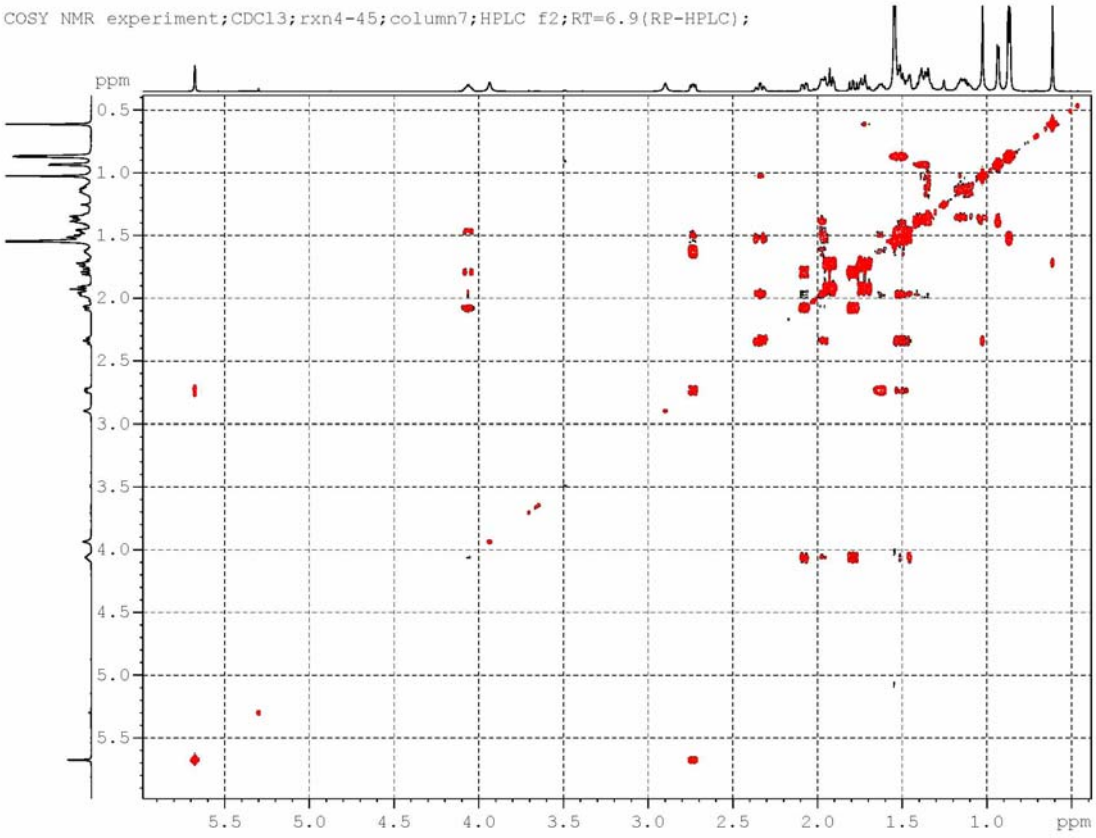
13C NMR experiment;CDCl3;rxn4-45;column7;HPLC f2;RT=6.9(10%IPA);238nm

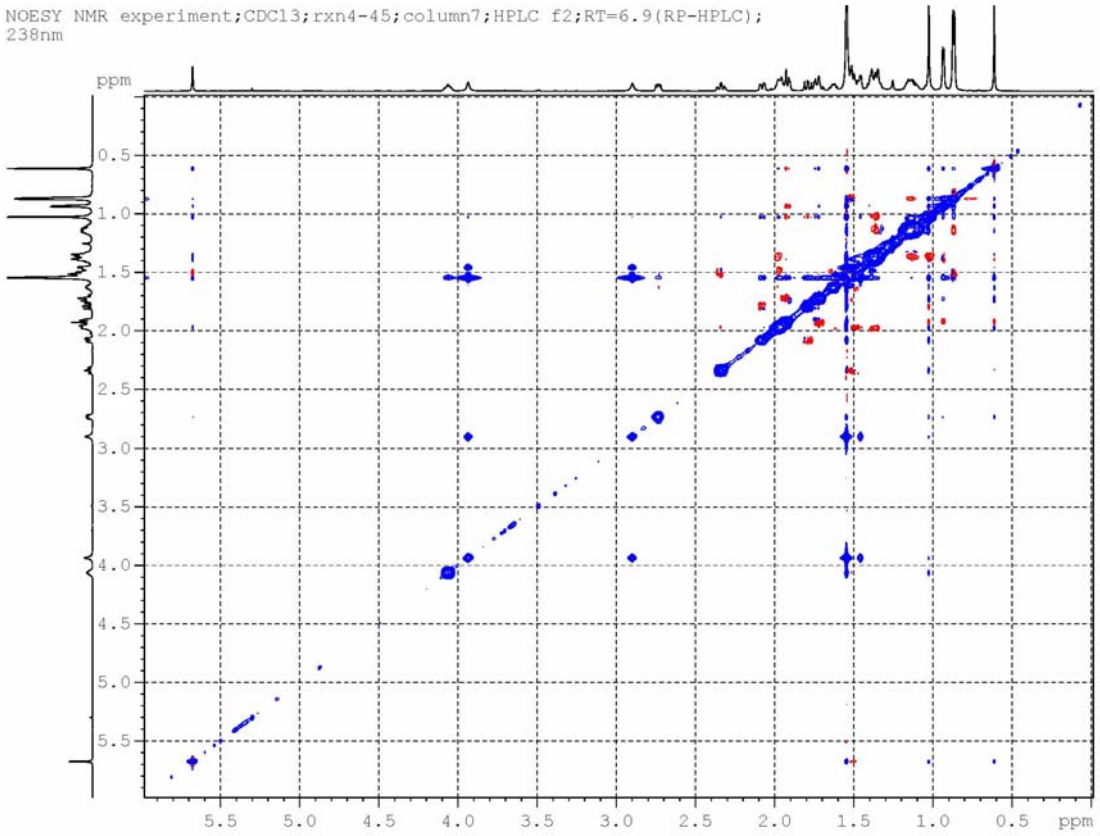


HSQC NMR experiment;CDCl3;rxn4-45;column7;HPLC f2;RT=6.9(RP-HPLC);
238nm

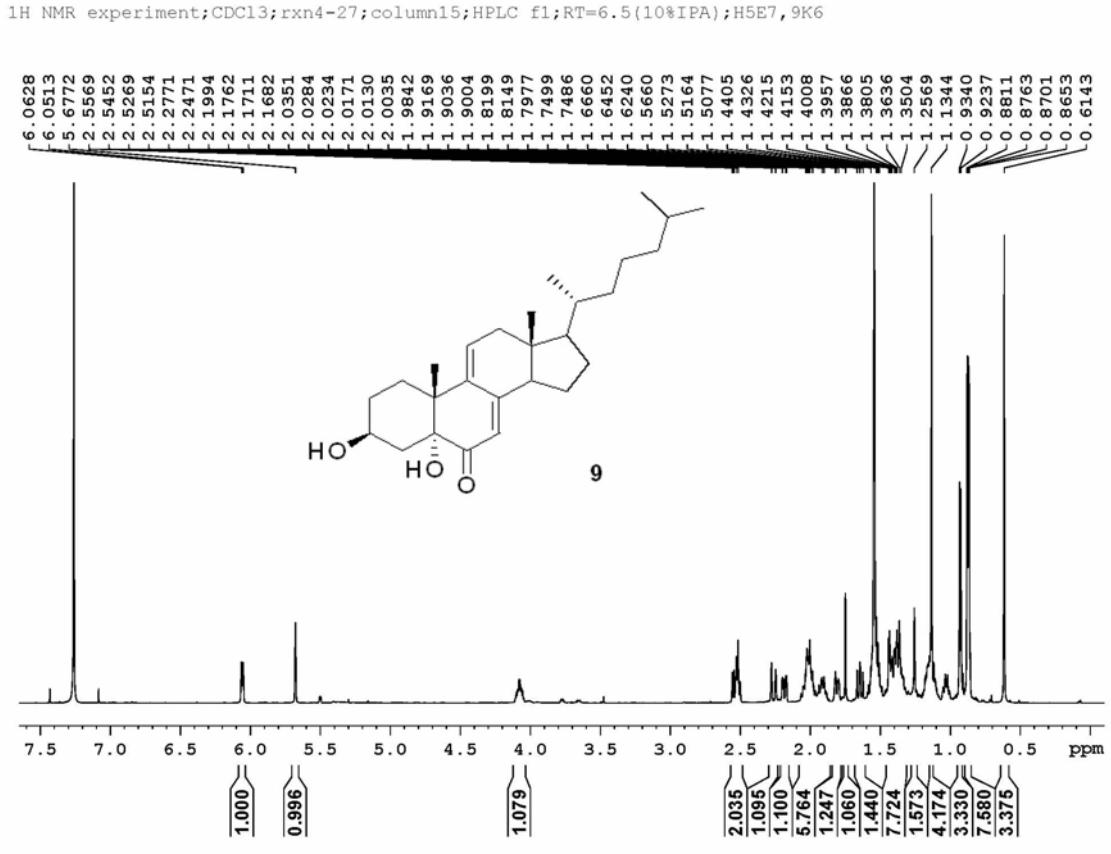



HMBC NMR experiment;CDCl3;rxn4-45;column7;HPLC f2;RT=6.9(RP-HPLC);
238nm
ppm
20
40
60
80
100
120
140
160
180
200
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

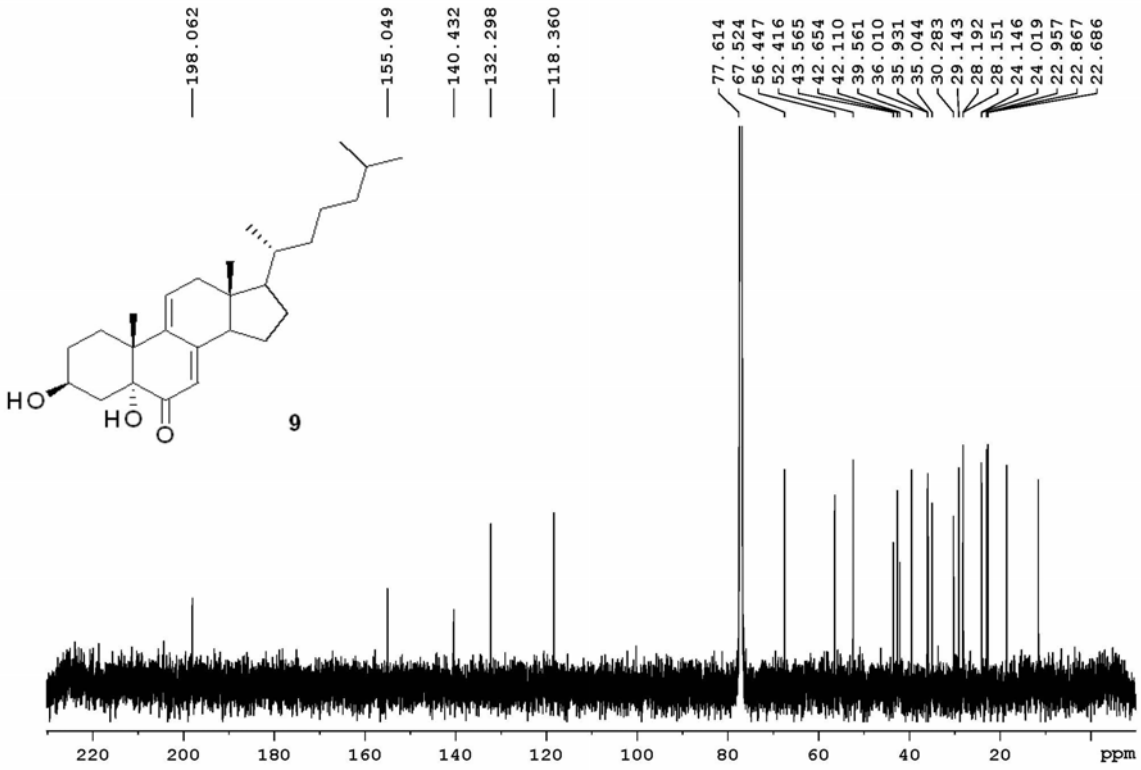

COSY NMR experiment;CDCl3;rxn4-45;column7;HPLC f2;RT=6.9(RP-HPLC);
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm



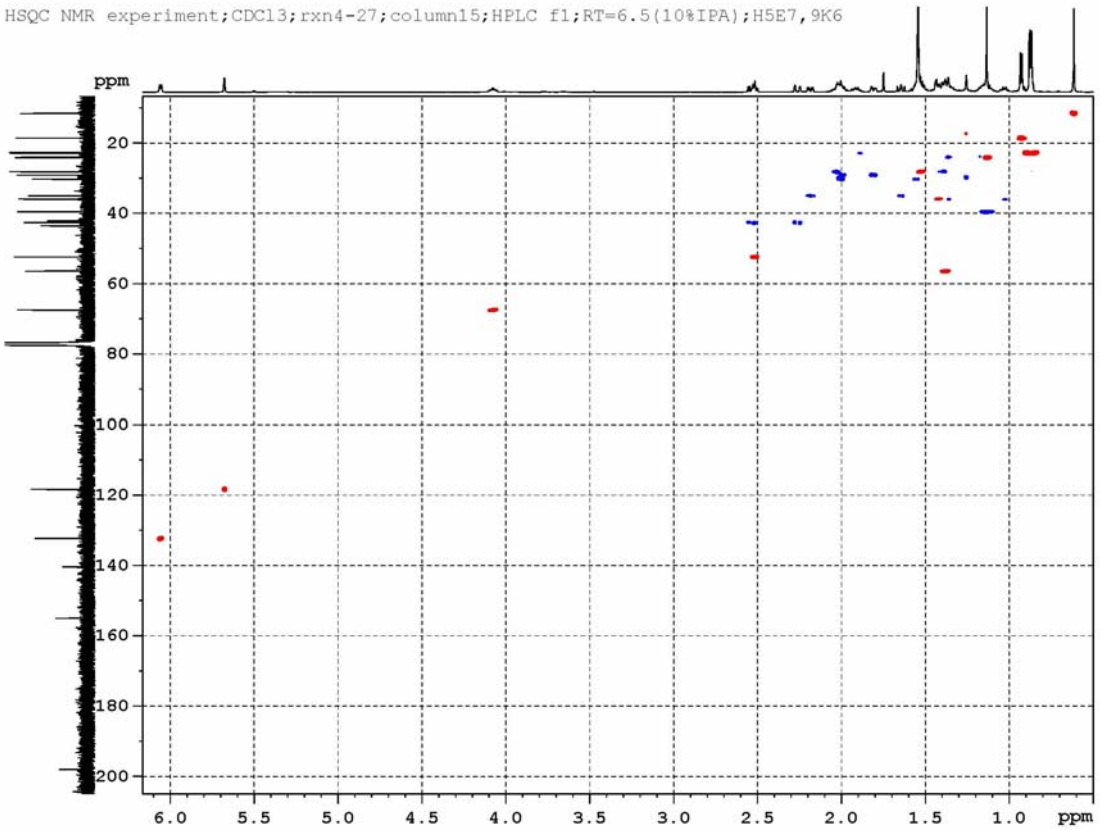
3β,5α-Dihydroxycholesta-7,9(11)-dien-6-one (9).

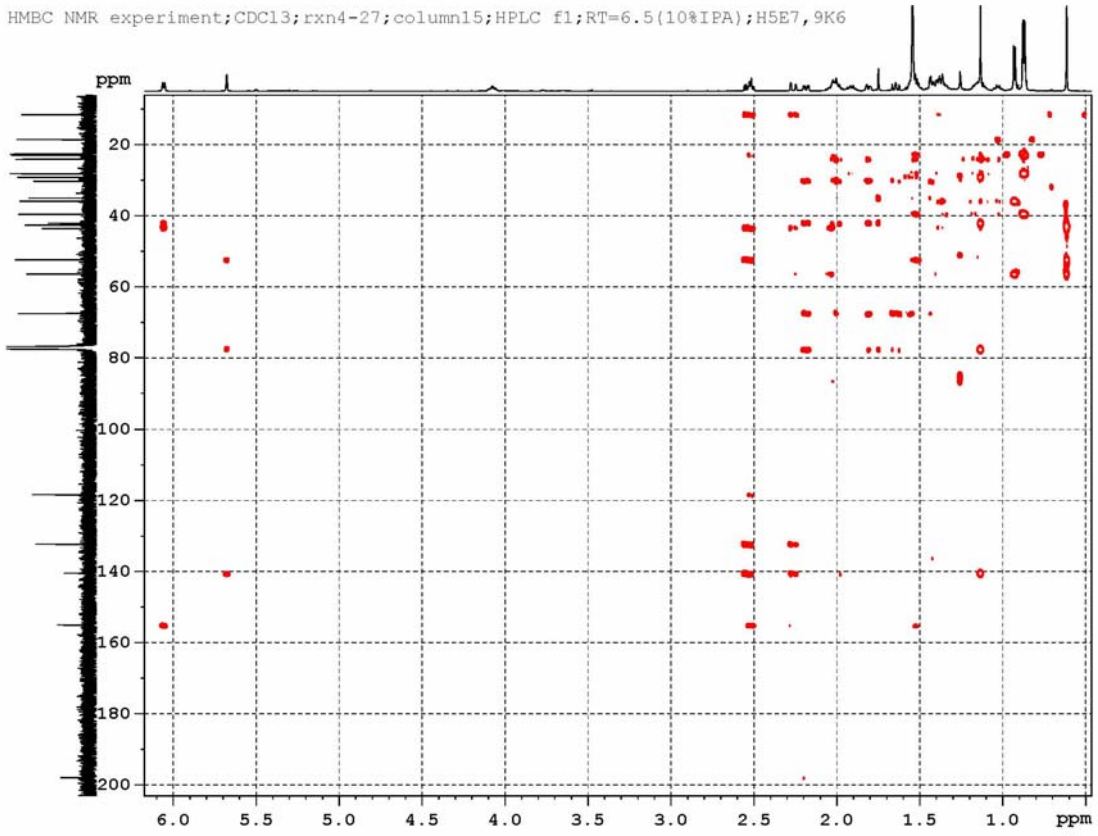


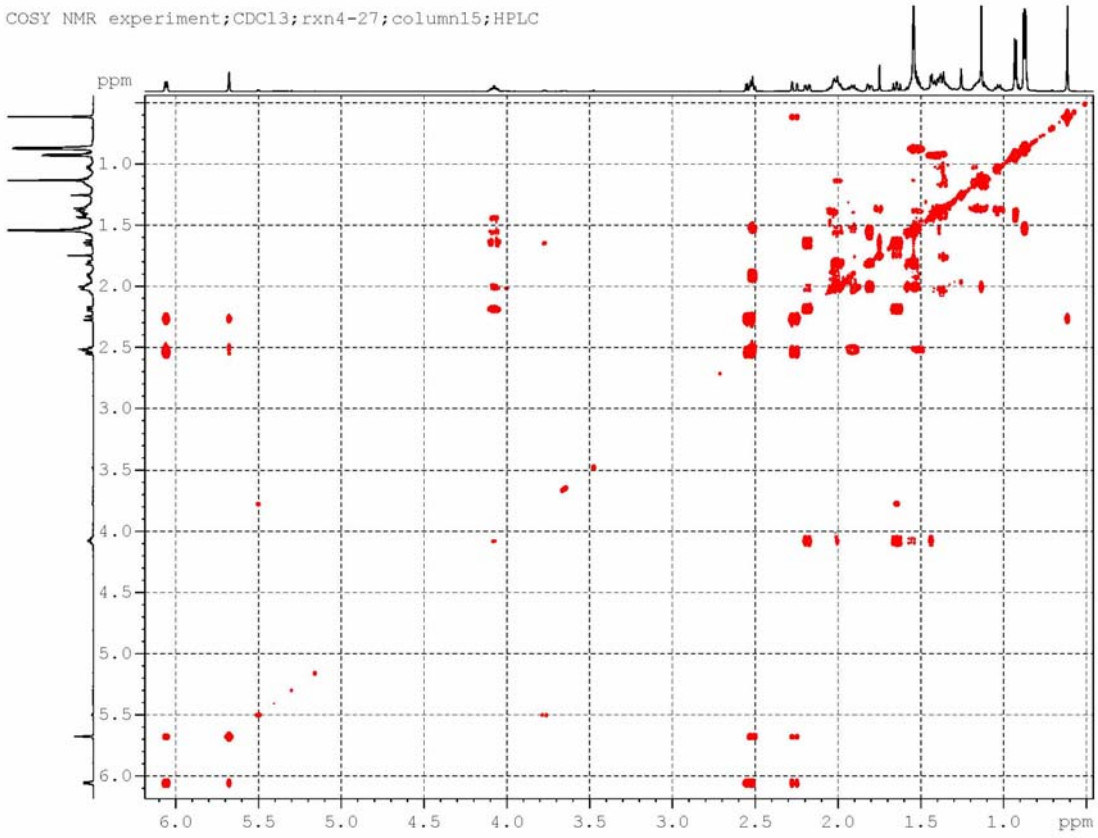
13C NMR experiment;CDCl3;rxn4-27;column15;HPLC f1;RT=6.5(10%IPA);H5E7,9K6

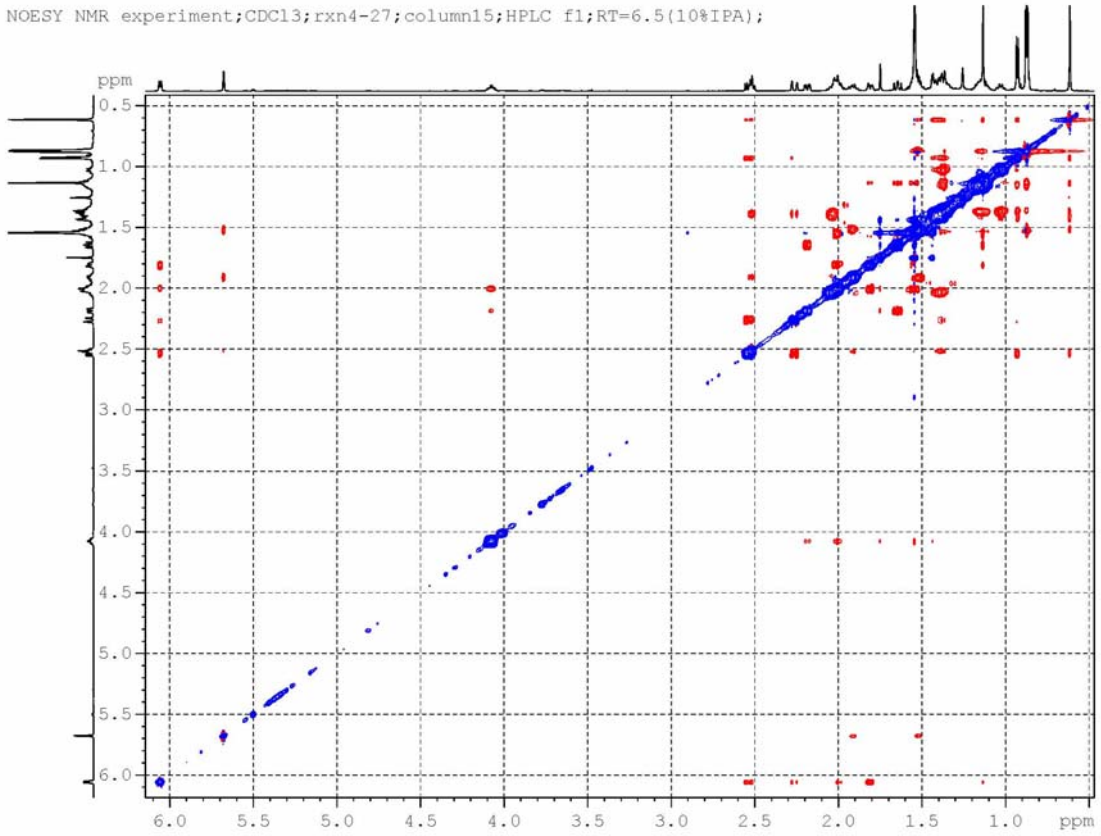


HSQC NMR experiment;CDCl3;rxn4-27;column15;HPLC f1;RT=6.5(10%IPA);H5E7,9K6

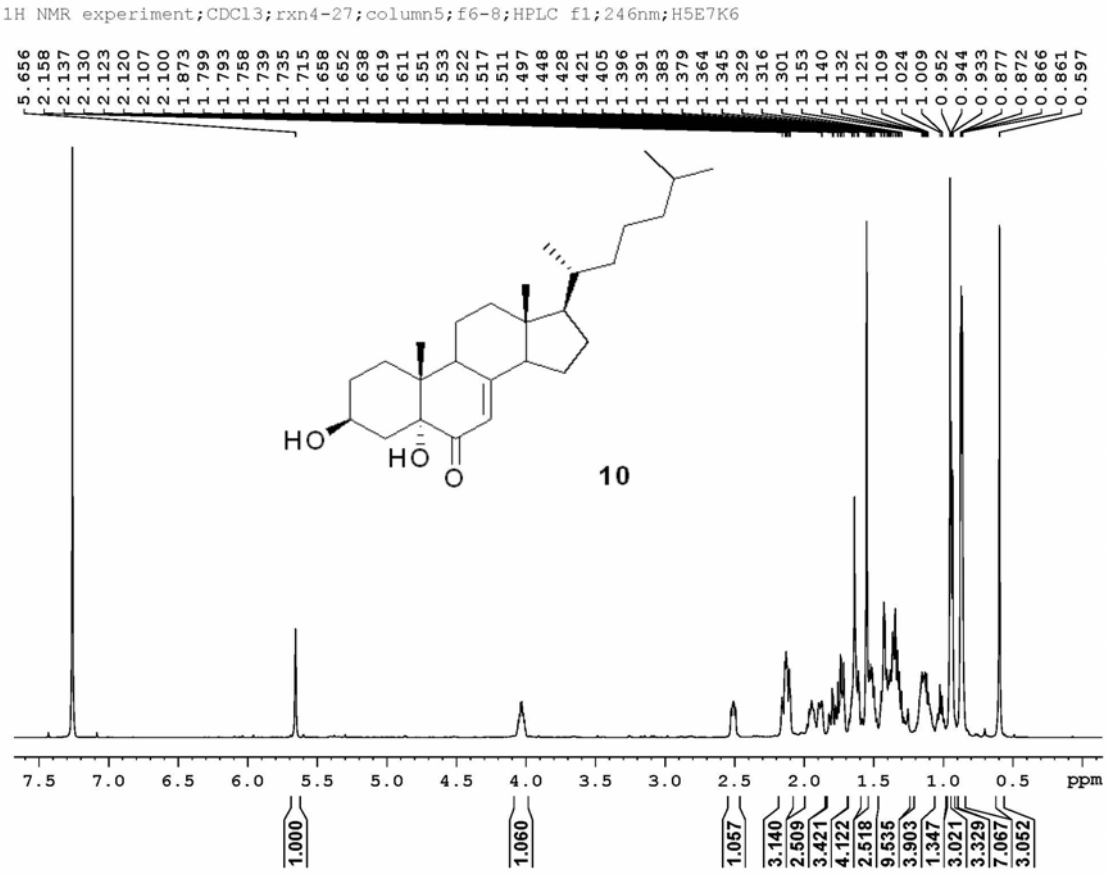



HMBC NMR experiment;CDCl3;rxn4-27;column15;HPLC f1;RT=6.5(10%IPA);H5E7,9K6
ppm
20
40
60
80
100
120
140
160
180
200
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

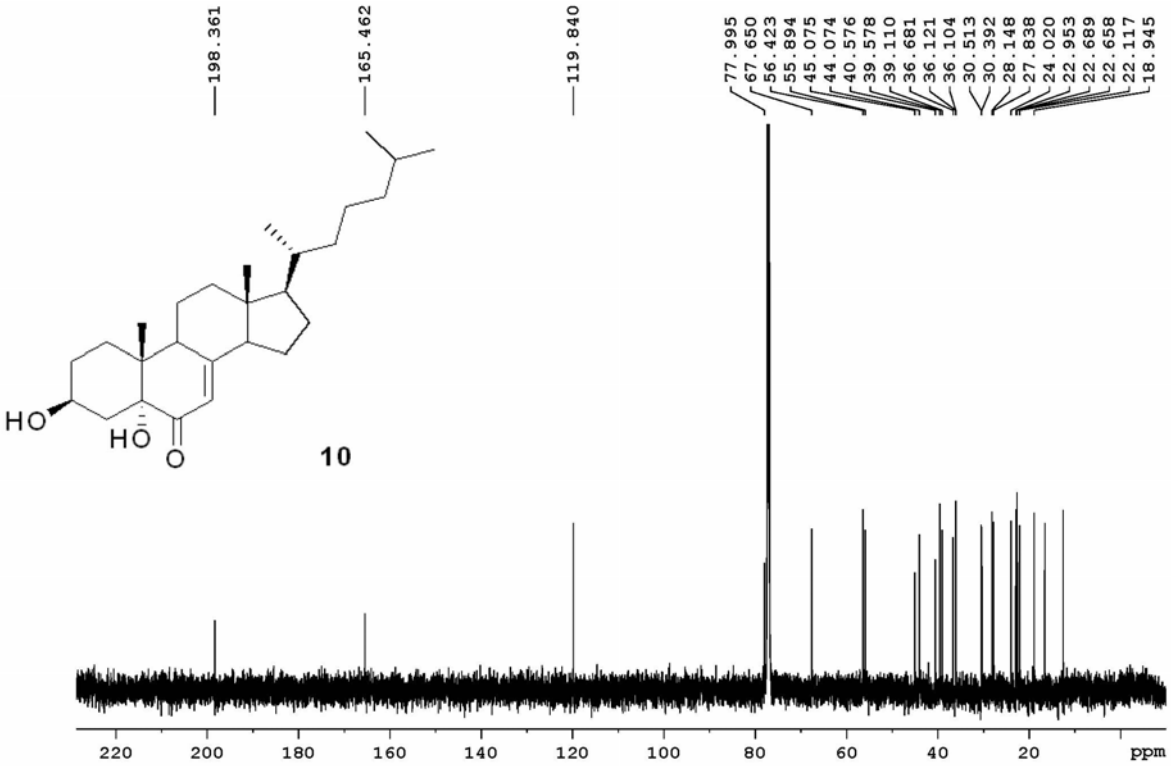

COSY NMR experiment;CDCl3;rxn4-27;column15;HPLC
ppm
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm



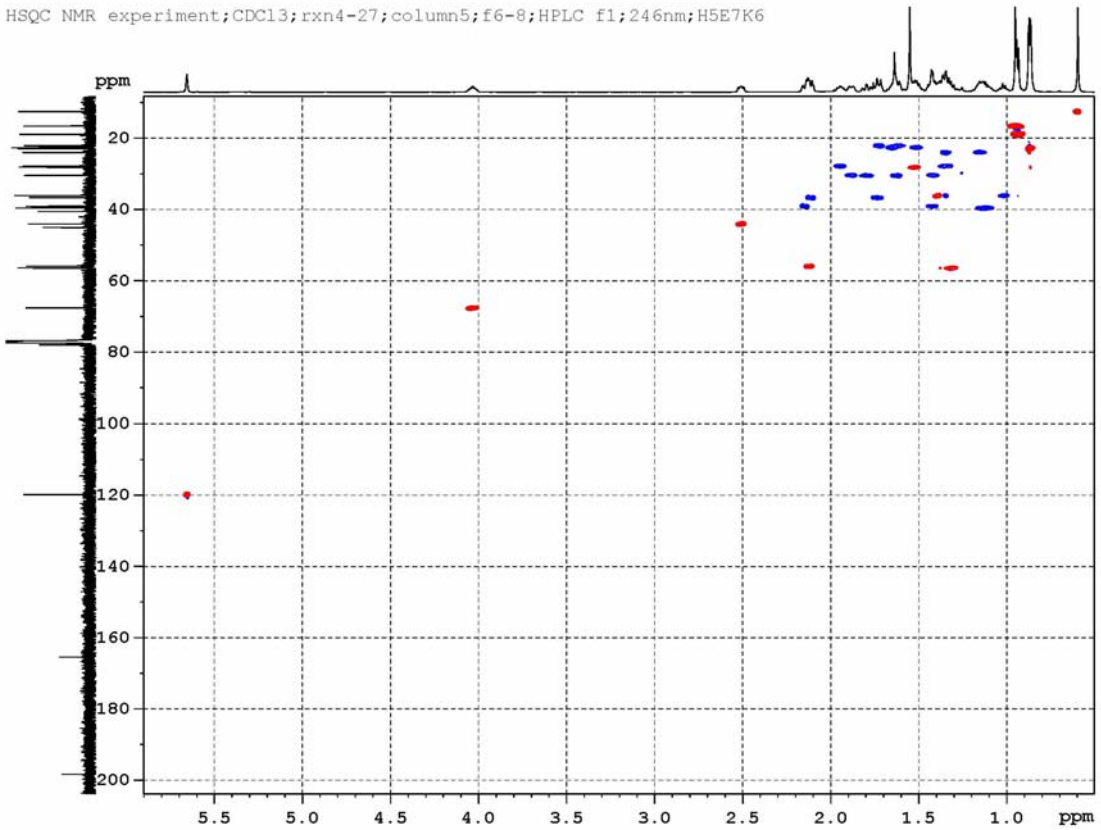
3β,5α-Dihydroxycholesta-7-en-6-one (10).

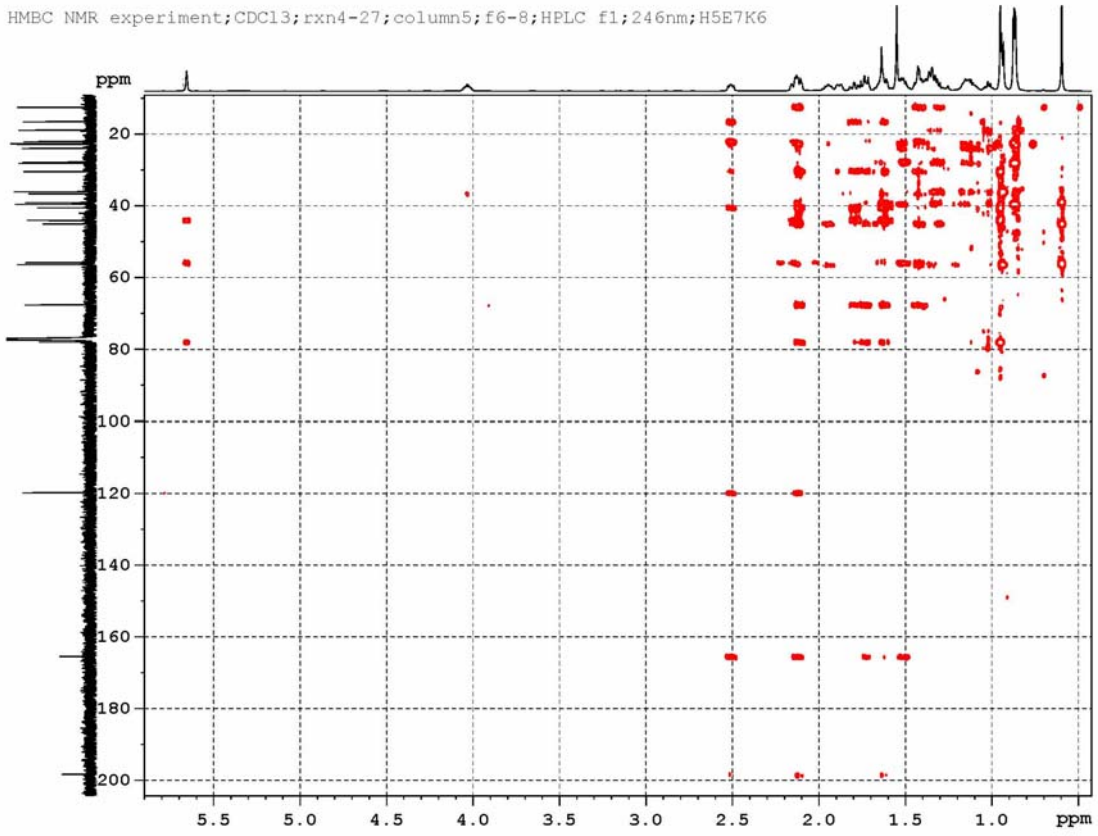


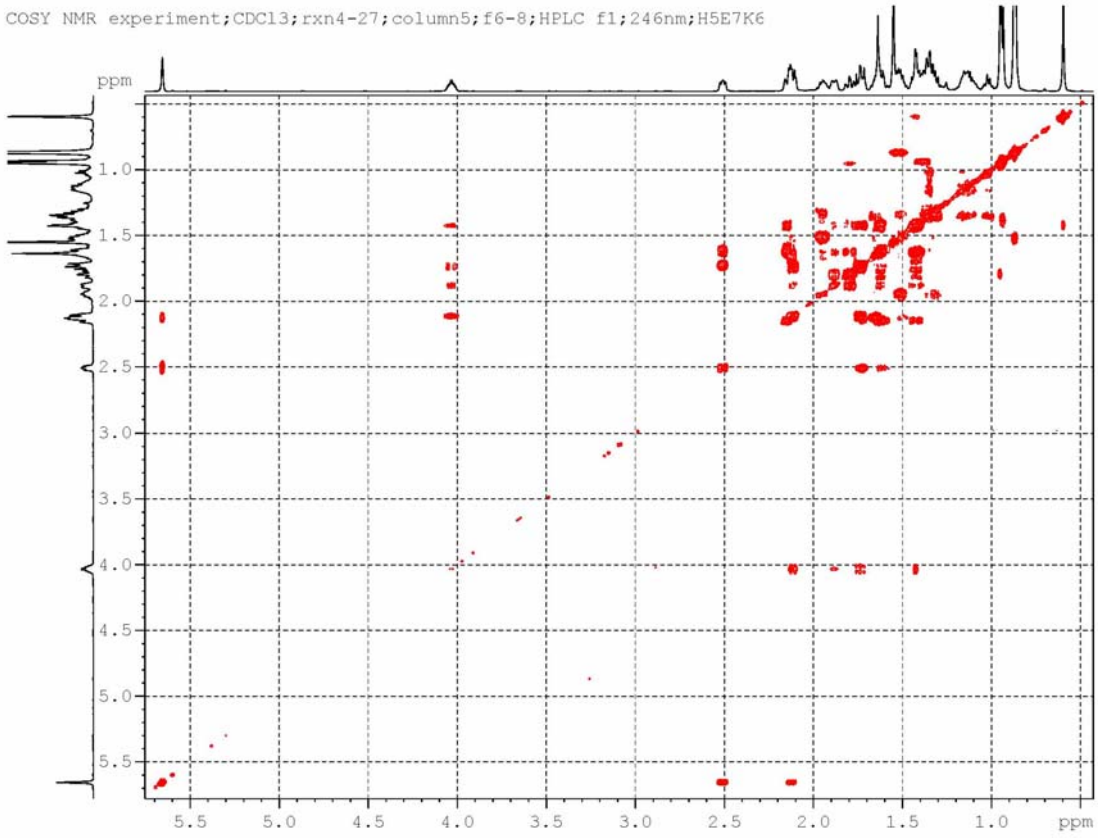
13C NMR experiment;CDCl3;rxn4-27;column5;f6-8;HPLC f1;246nm;H5E7K6

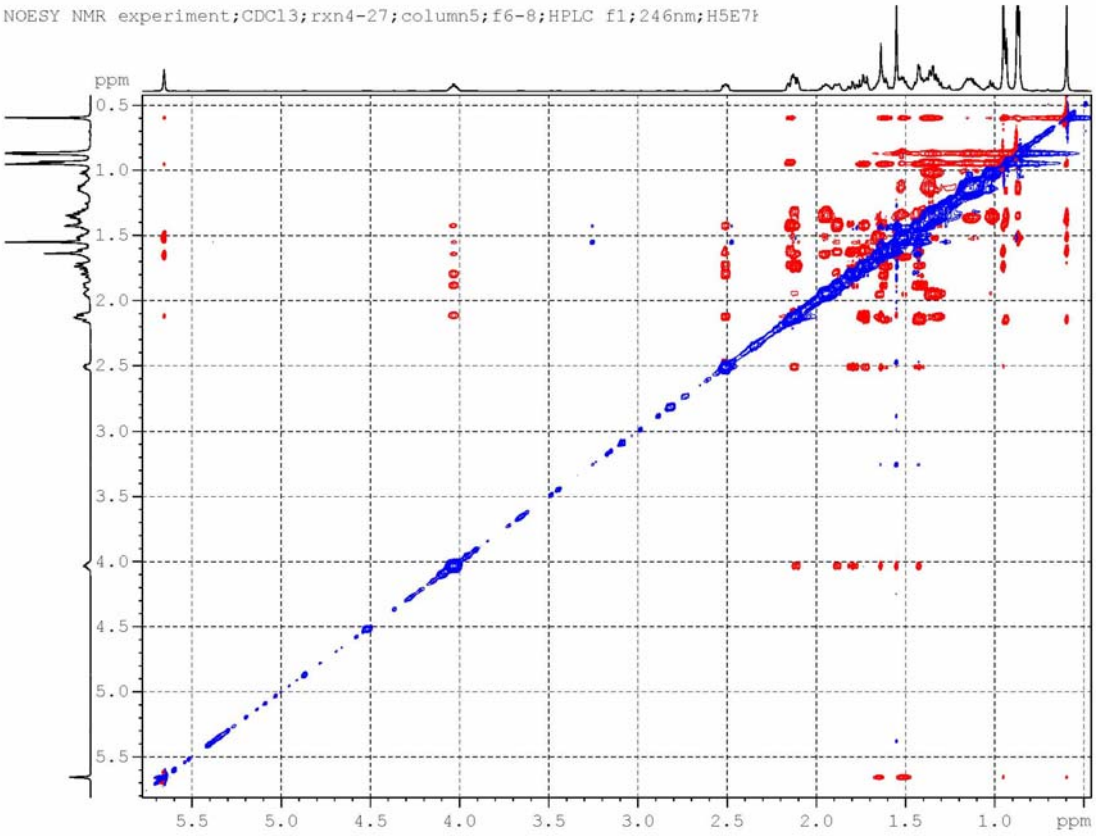


HSQC NMR experiment;CDCl3;rxn4-27;column5;f6-8;HPLC f1;246nm;H5E7K6

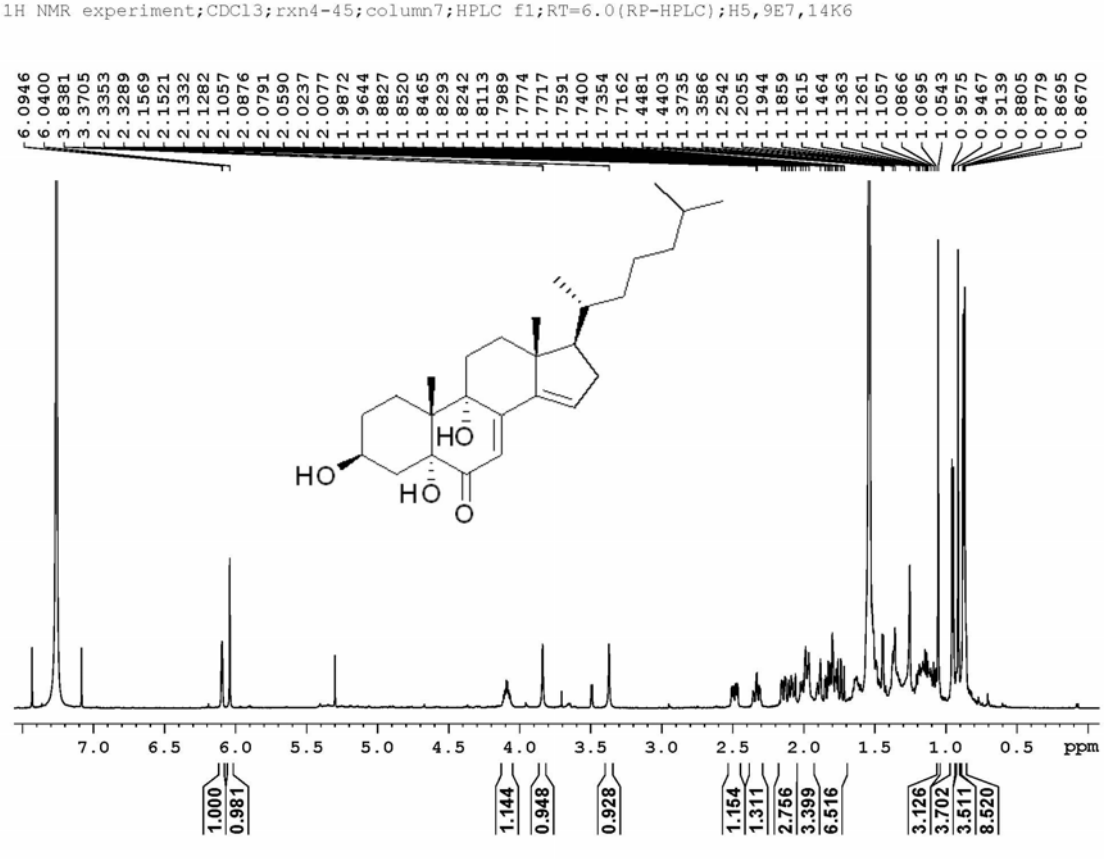


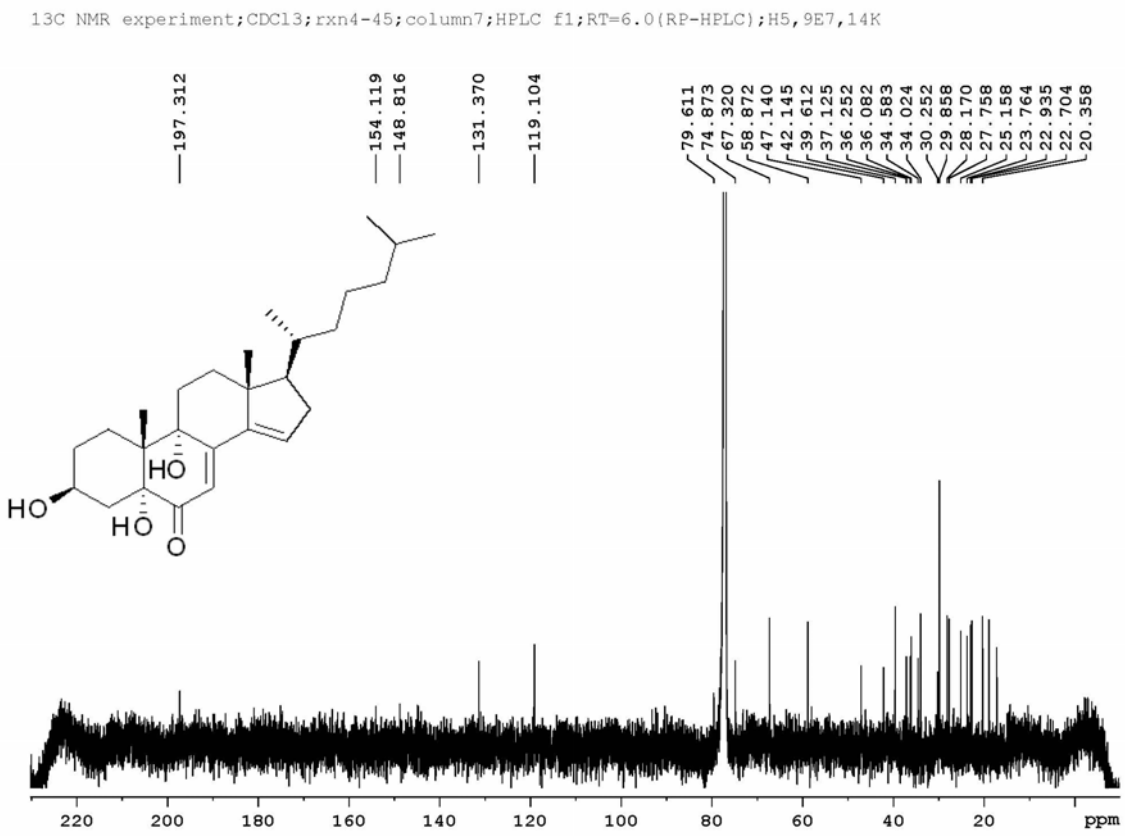

HMBC NMR experiment;CDCl3;rxn4-27;column5;f6-8;HPLC f1;246nm;H5E7K6
ppm
20
40
60
80
100
120
140
160
180
200
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

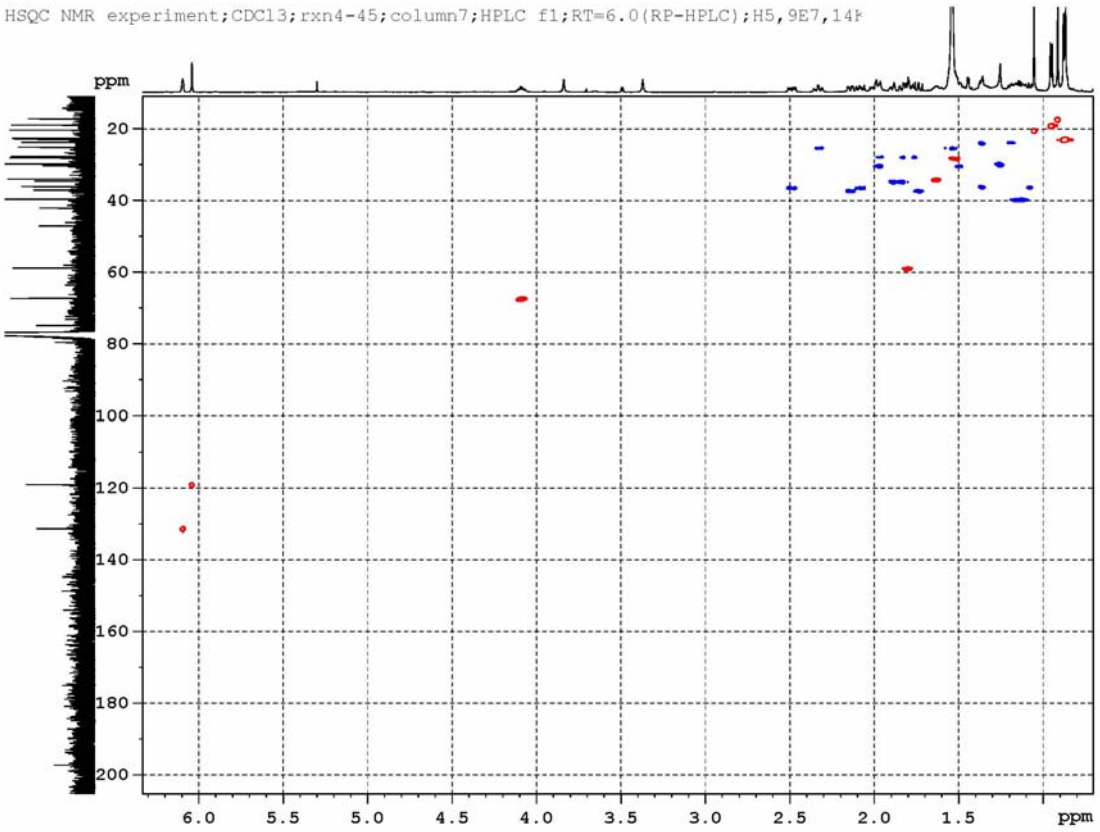

COSY NMR experiment;CDCl3;rxn4-27;column5;f6-8;HPLC f1;246nm;H5E7K6
ppm
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

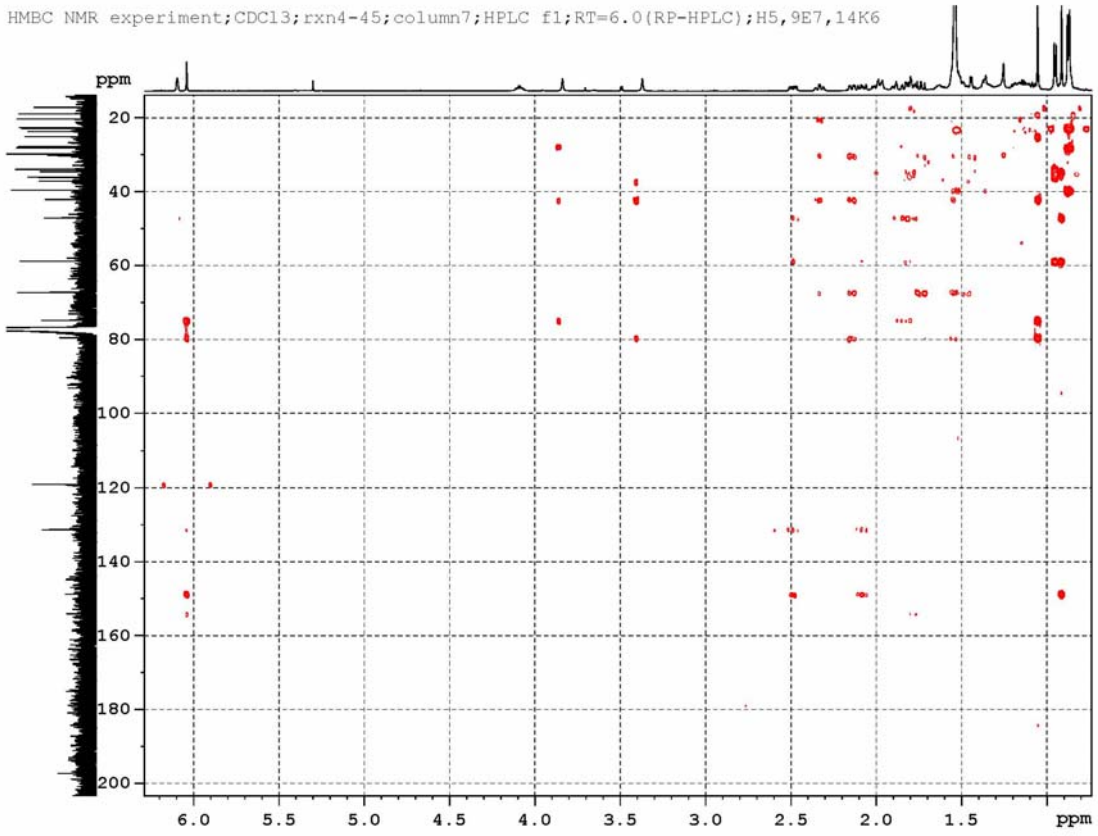


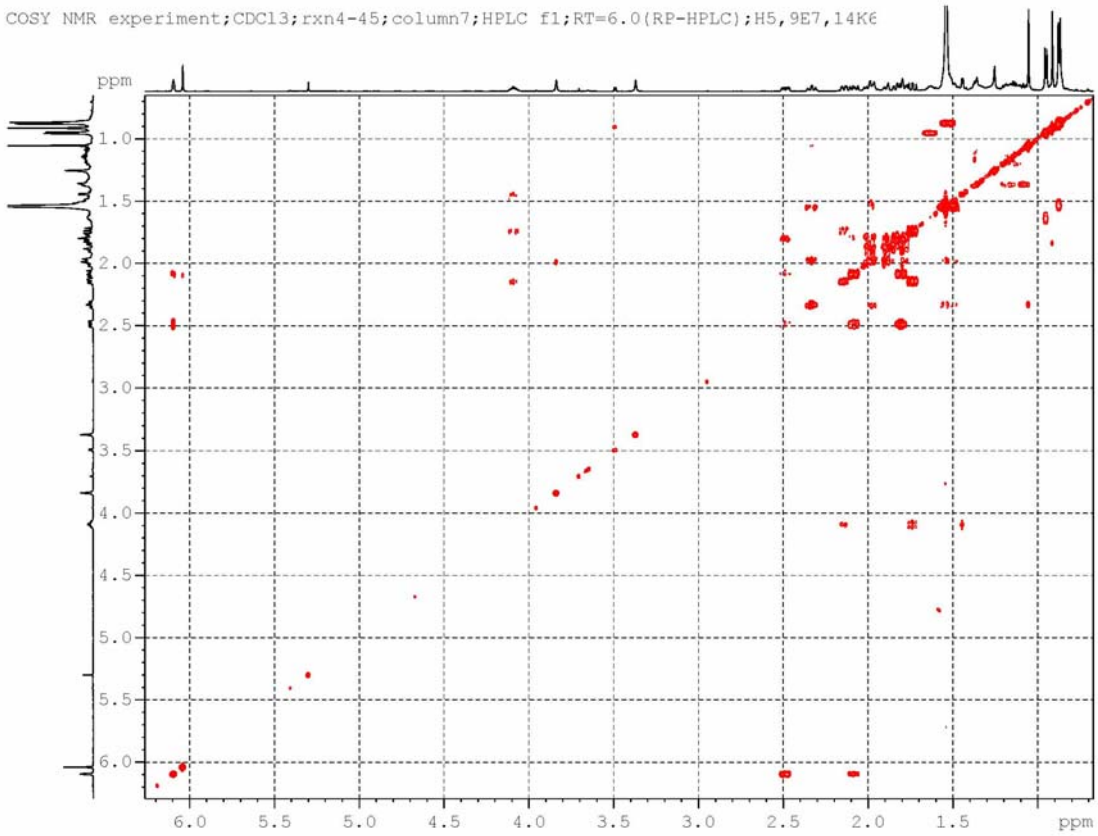
3β,5α,9α-Trihydroxycholesta-7,14-dien-6-one (11).

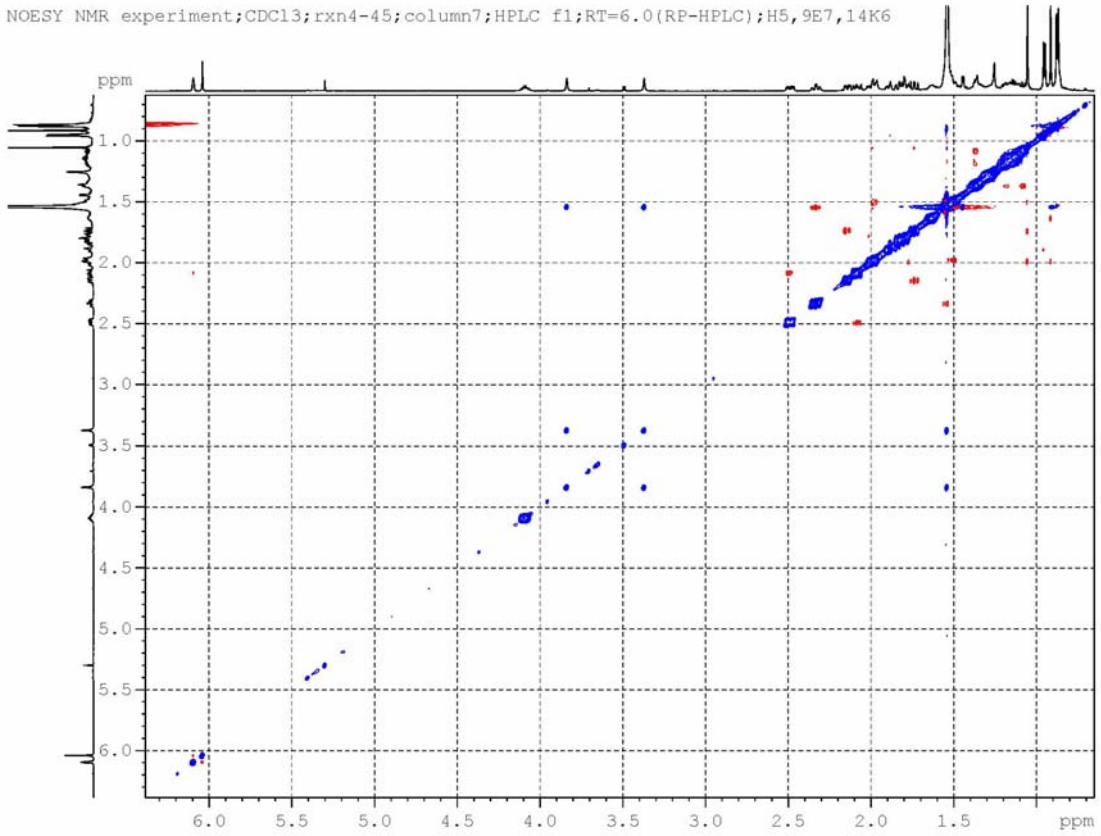



13C NMR experiment;CDCl3;rxn4-45;column7;HPLC f1;RT=6.0(RP-HPLC);H5,9E7,14K
197.312
154.119
148.816
131.370
119.104
79.611
74.873
67.320
58.872
47.140
42.145
39.612
37.125
36.252
36.082
34.583
34.024
30.252
29.858
28.170
27.758
25.158
23.764
22.935
22.704
20.358
HO
HO
HO
O
220
200
180
160
140
120
100
80
60
40
20
ppm

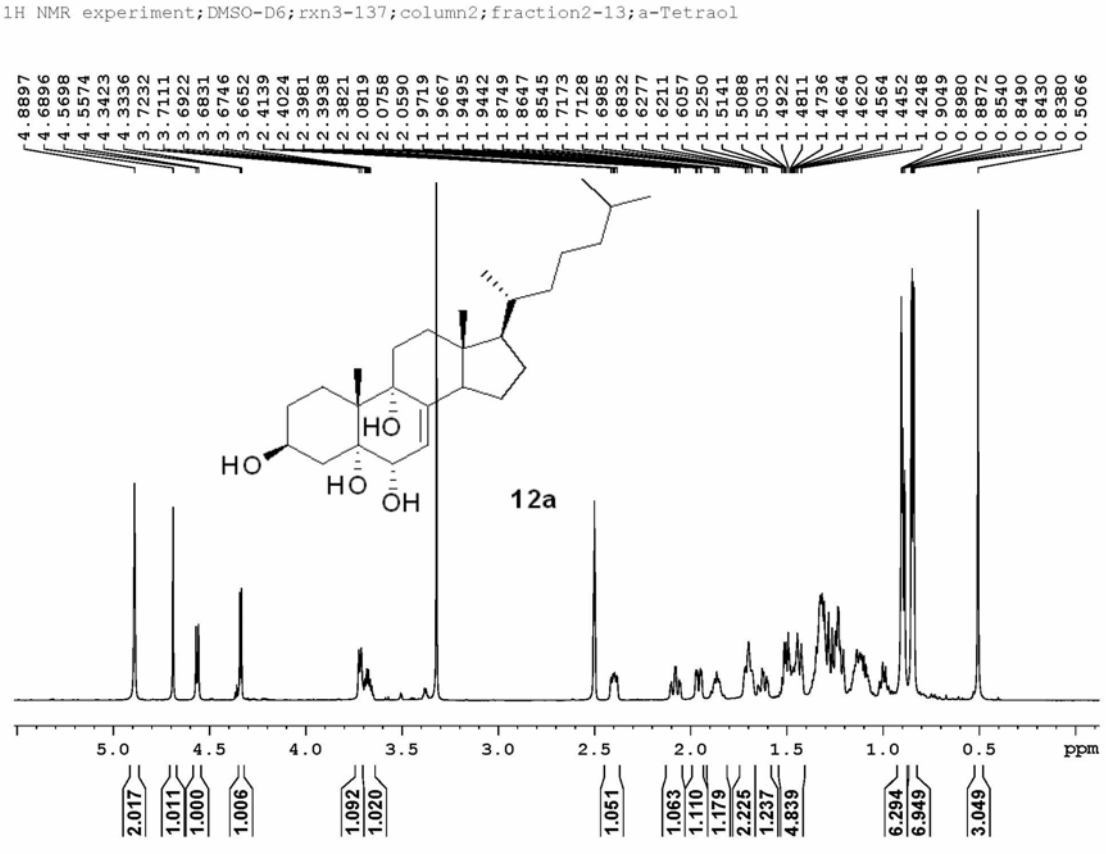

HSQC NMR experiment;CDCl3;rxn4-45;column7;HPLC f1;RT=6.0(RP-HPLC);H5,9E7,14K
ppm
20
40
60
80
100
120
140
160
180
200
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm


HMBC NMR experiment;CDCl3;rxn4-45;column7;HPLC f1;RT=6.0(RP-HPLC);H5,9E7,14K6
ppm
20
40
60
80
100
120
140
160
180
200
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm

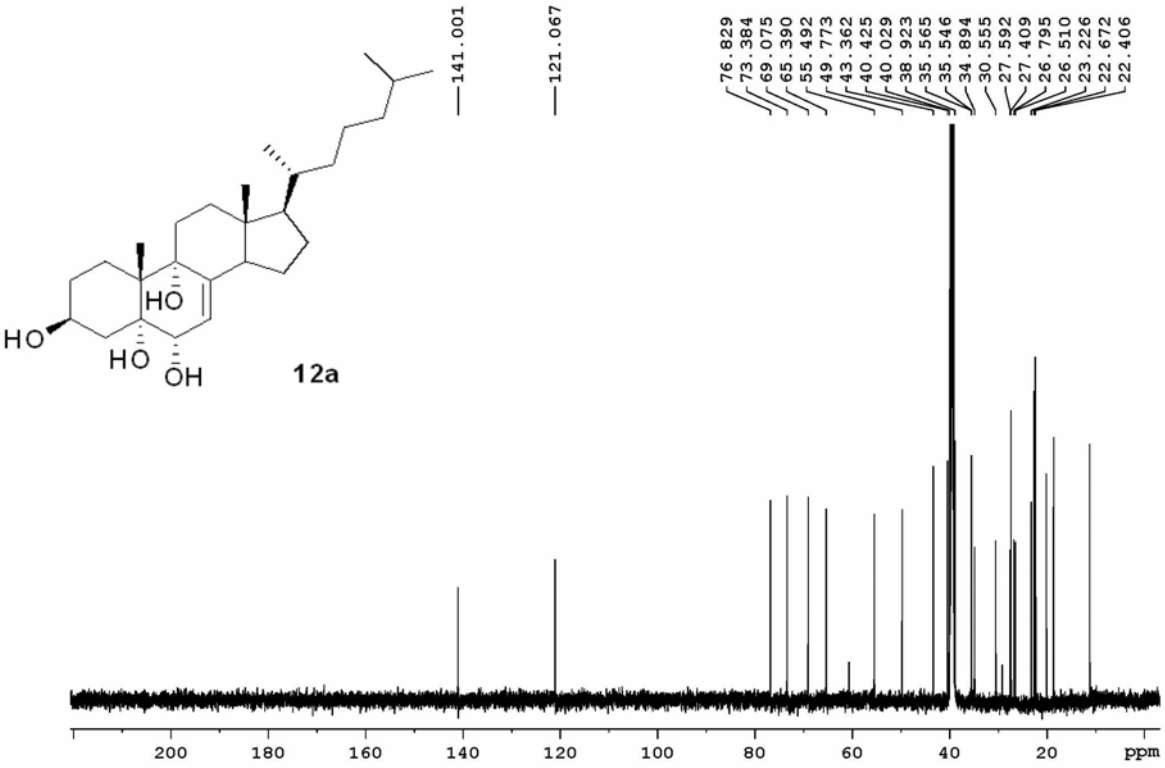

COSY NMR experiment;CDCl3;rxn4-45;column7;HPLC f1;RT=6.0(RP-HPLC);H5,9E7,14K6
ppm
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm



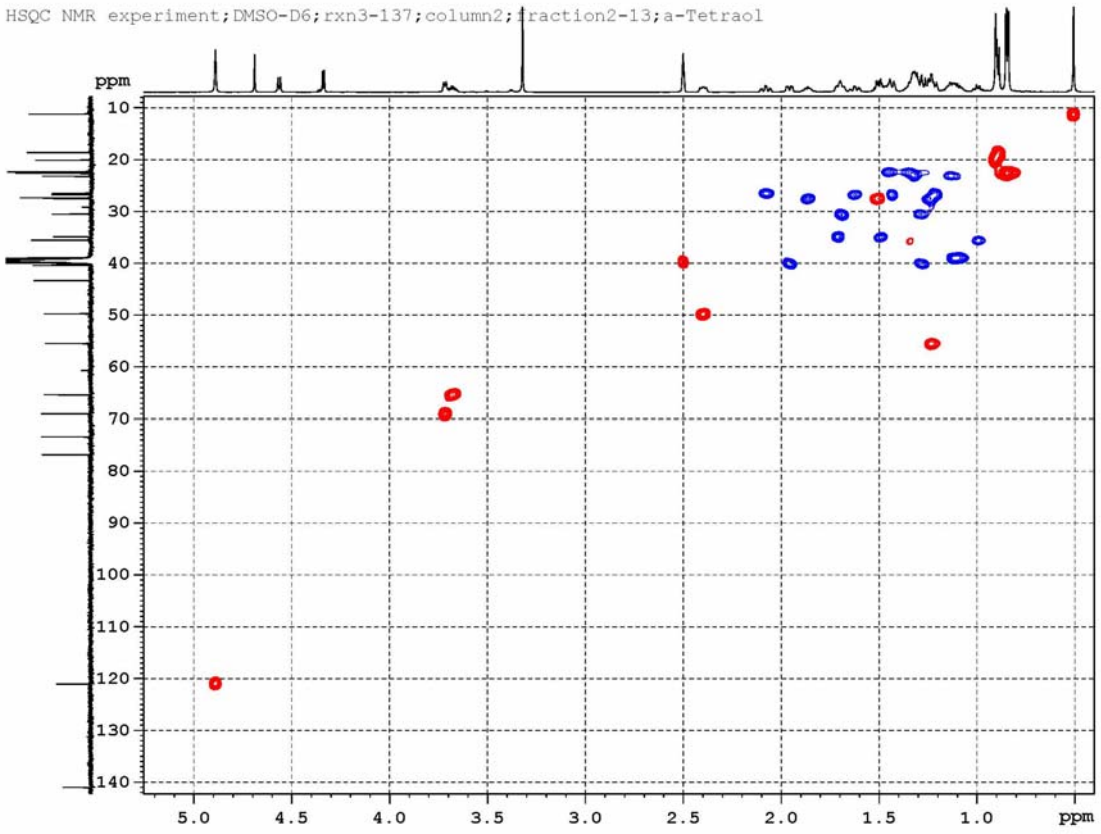
Cholesta-7-en-3β,6α,5α,9α-tetraol (12a).

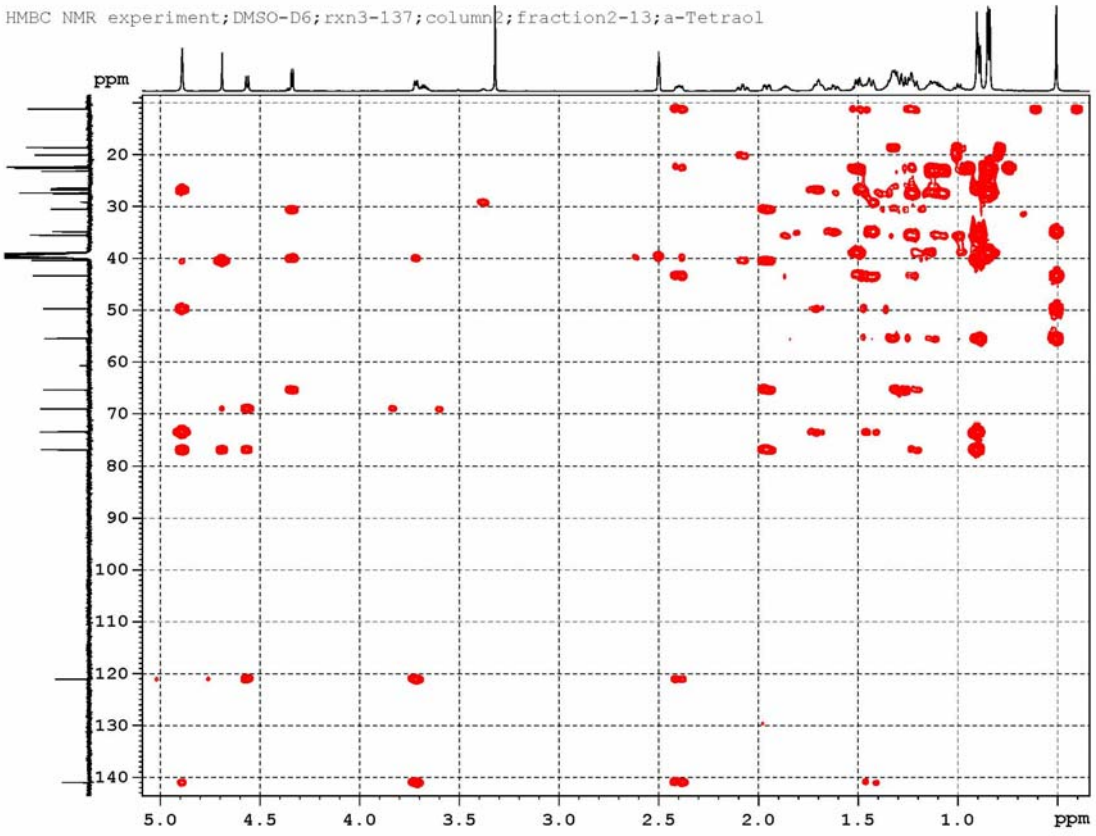


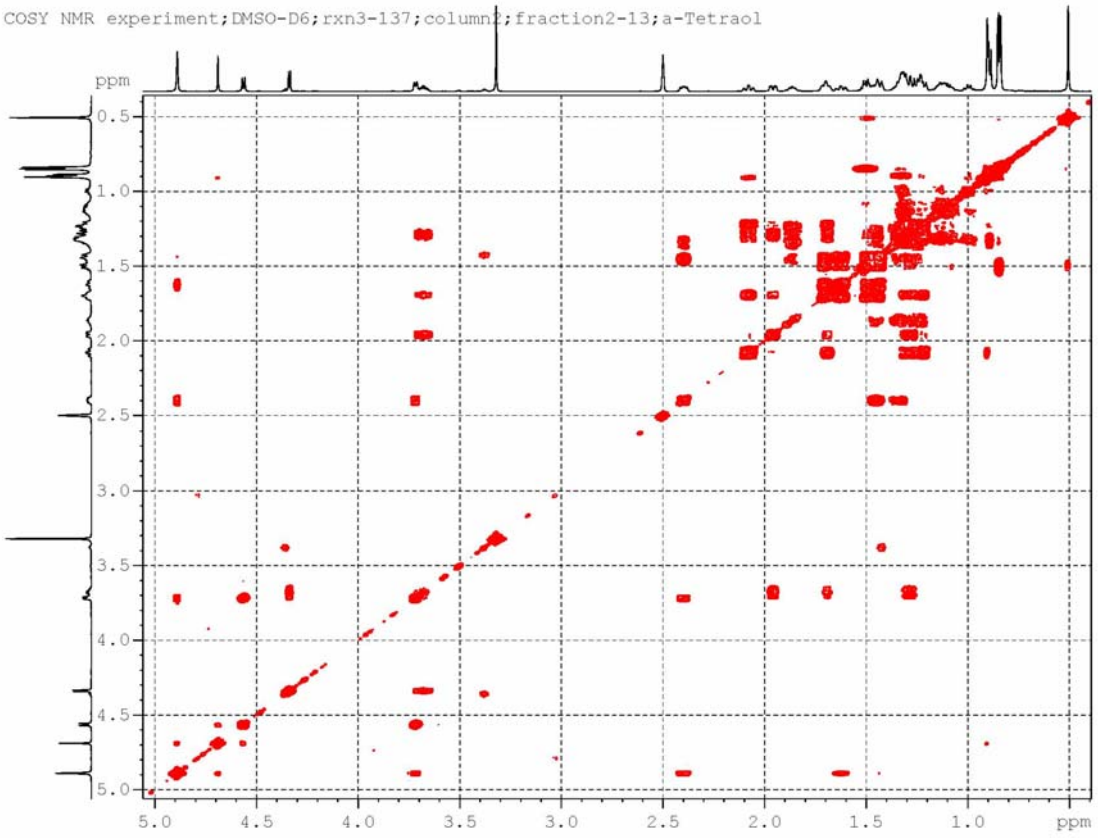
13C NMR experiment;DMSO-D6;rxn3-137;column2;fraction2-13;a-Tetraol

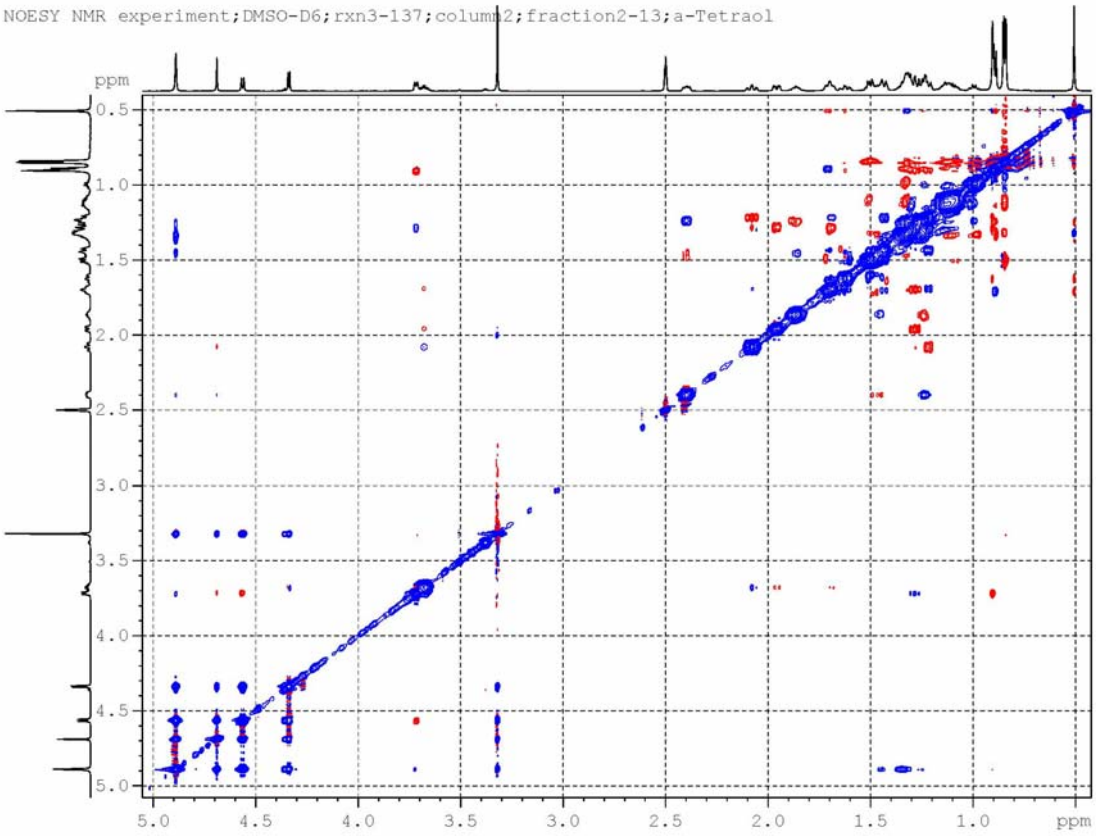


HSQC NMR experiment;DMSO-D6;rxn3-137;column2;fraction2-13;a-Tetraol

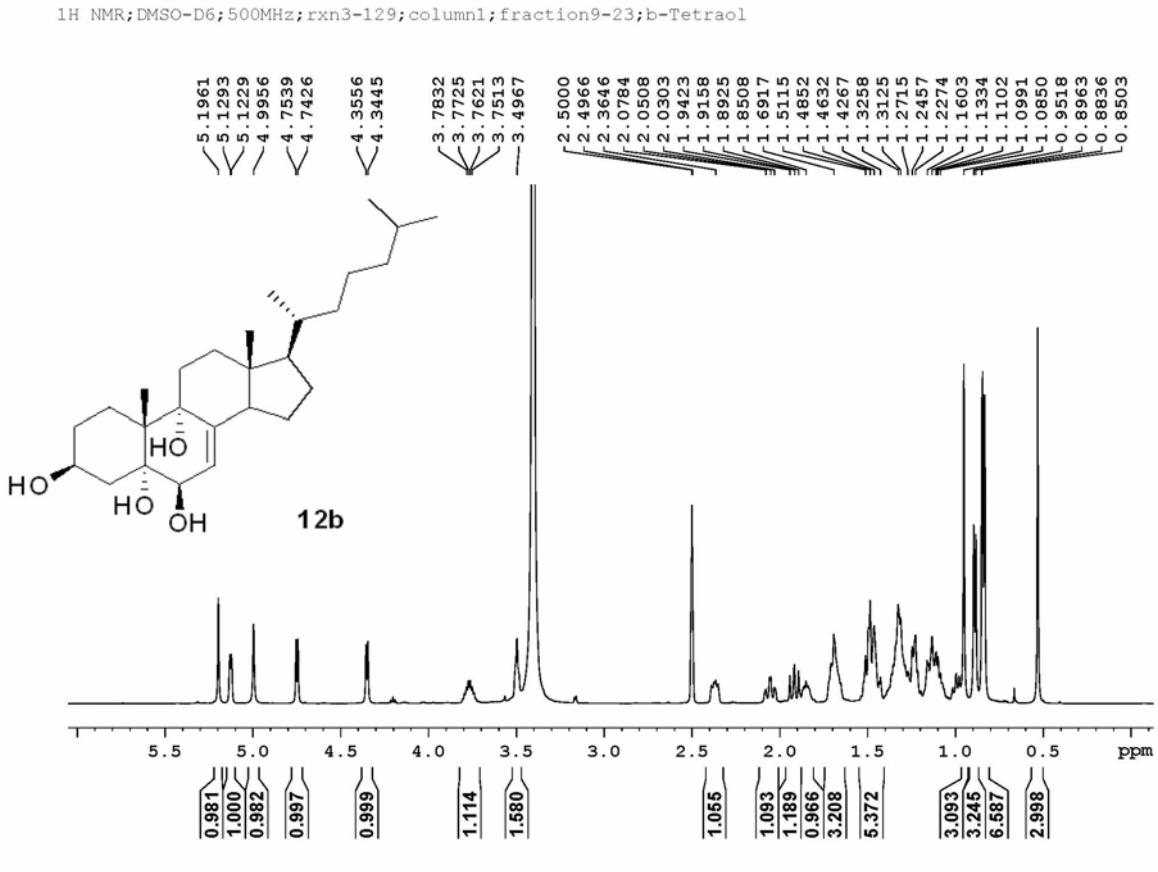



HMBC NMR experiment;DMSO-D6;rxn3-137;column2;fraction2-13;a-Tetraol
ppm
20
30
40
50
60
70
80
90
100
110
120
130
140
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

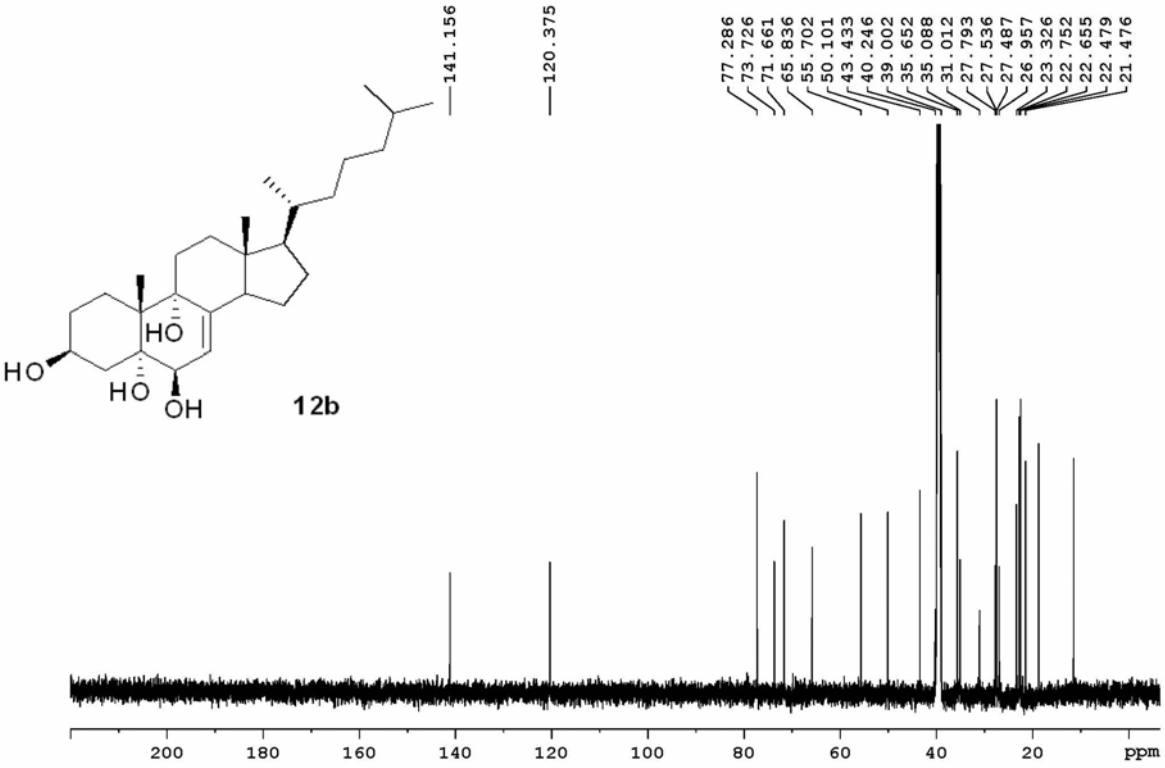

COSY NMR experiment;DMSO-D6;rxn3-137;column2;fraction2-13;a-Tetraol
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm



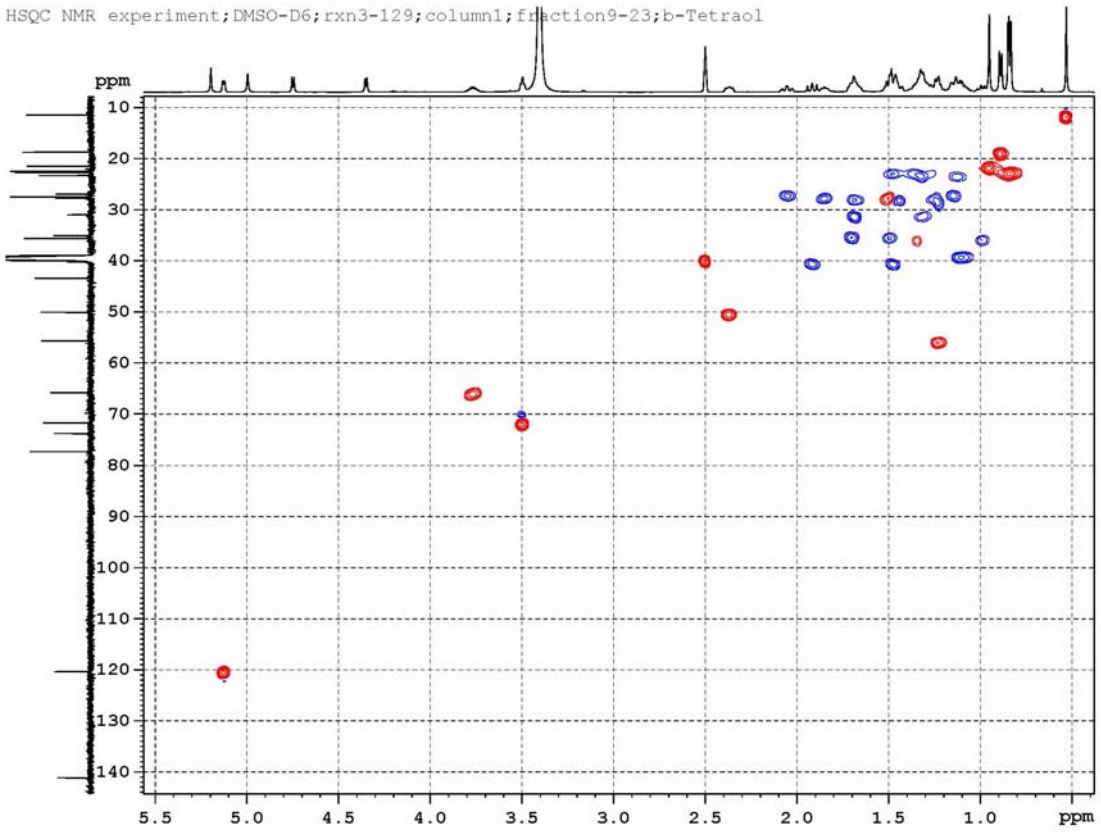
Cholesta-7-en-3β,6β,5α,9α-tetraol (12b).

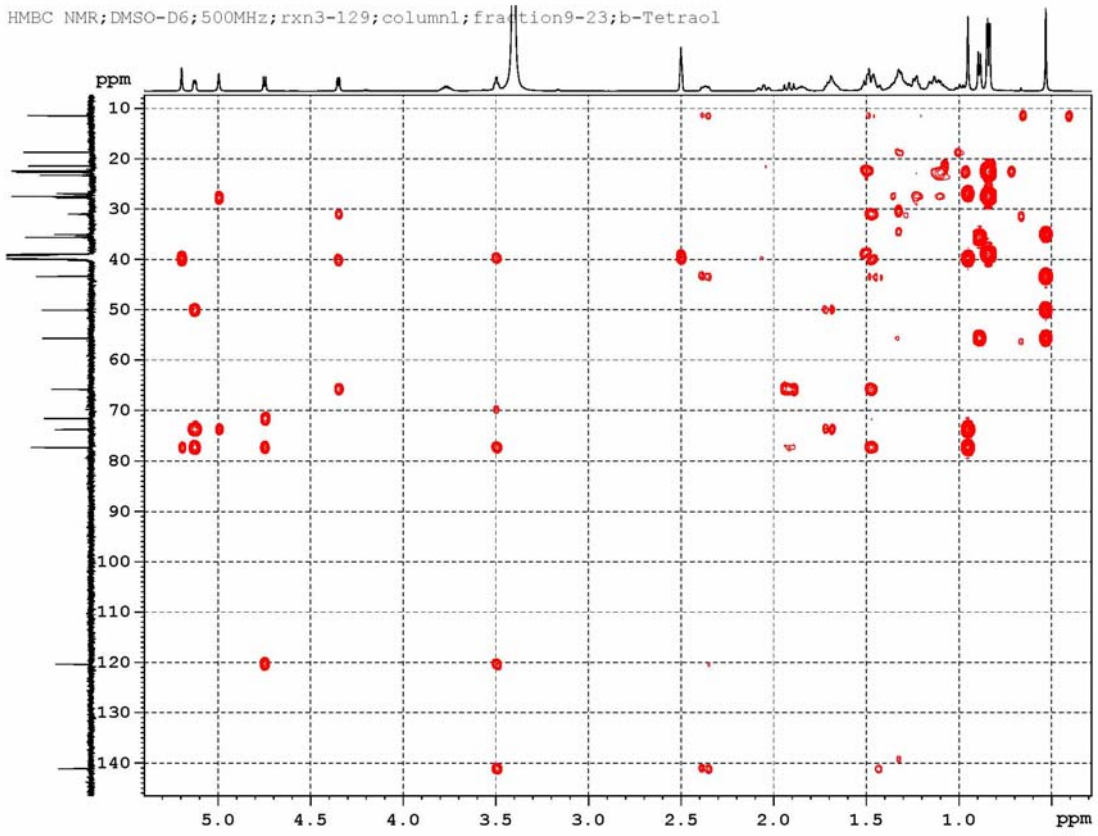


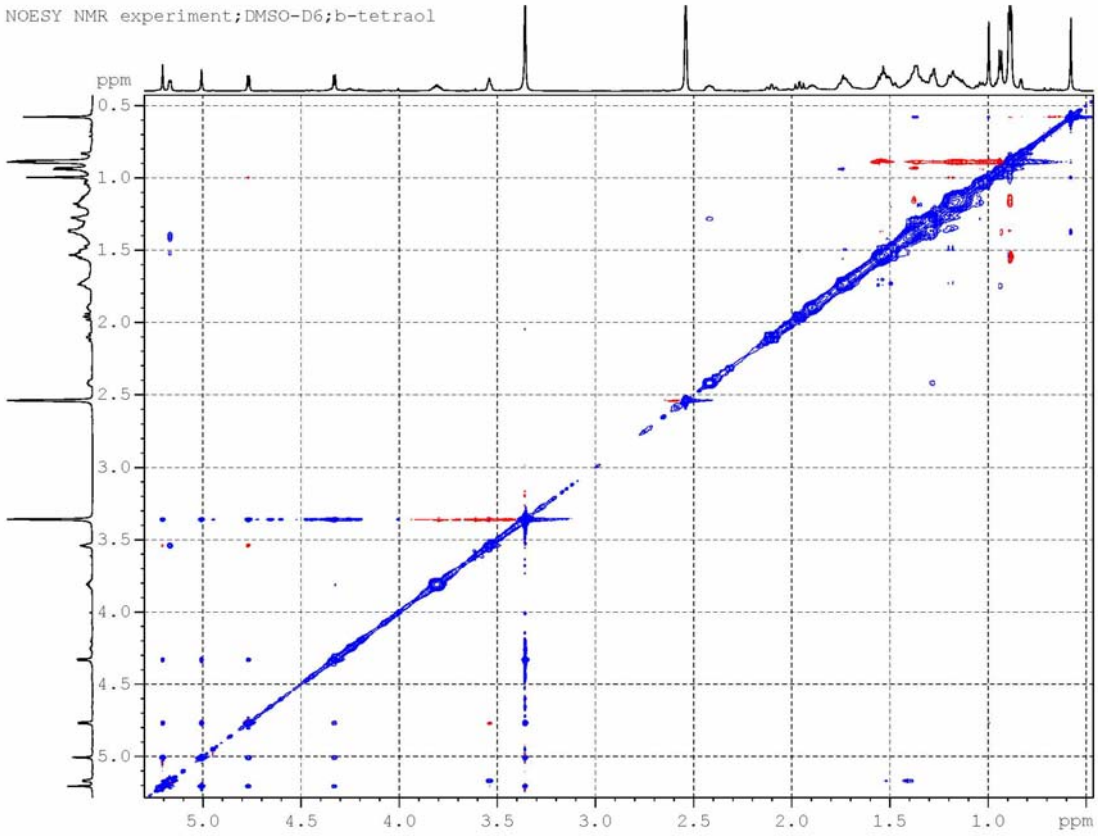
13C NMR experiment;DMSO-D6;rxn3-129;column1;fraction9-23;b-Tetraol



HSQC NMR experiment;DMSO-D6;rxn3-129;column1;fraction9-23;b-Tetraol




HMBC NMR;DMSO-D6;500MHz;rxn3-129;column1;fraction9-23;b-Tetraol
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


NOESY NMR experiment;DMSO-D6;b-tetraol
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm