Supporting Information

NMR Data (400 MHz, basic frequency, $CDCl_3$) for the paper:

Twilight Zone Sponges from Guam Yield Theonellin Isocyanate and Psammaplysins I and J

By

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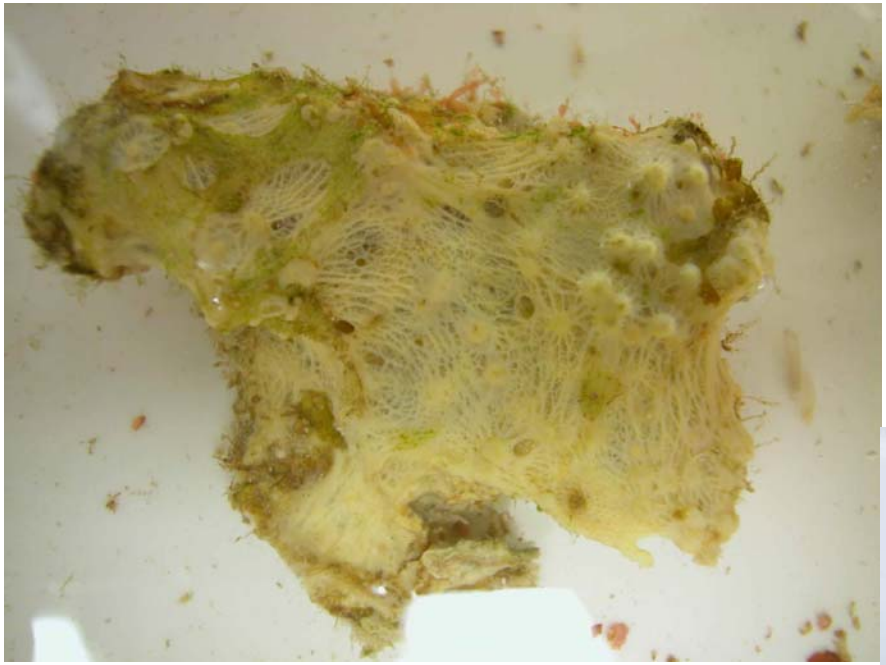
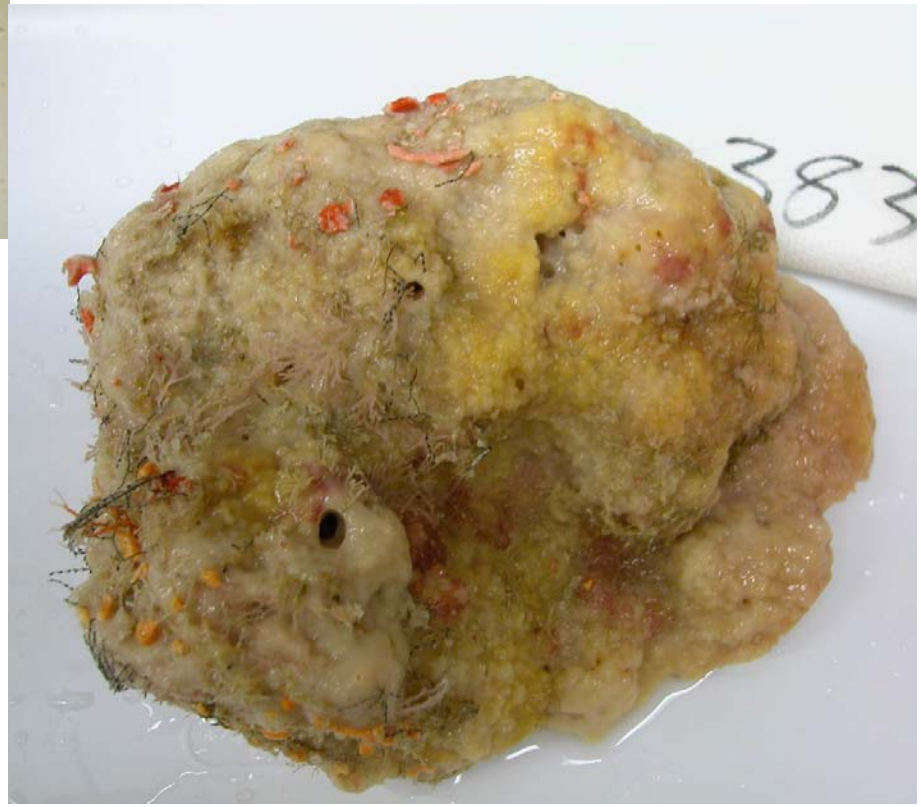
Supporting Information: List of Contents:



***Suberea* sp.**



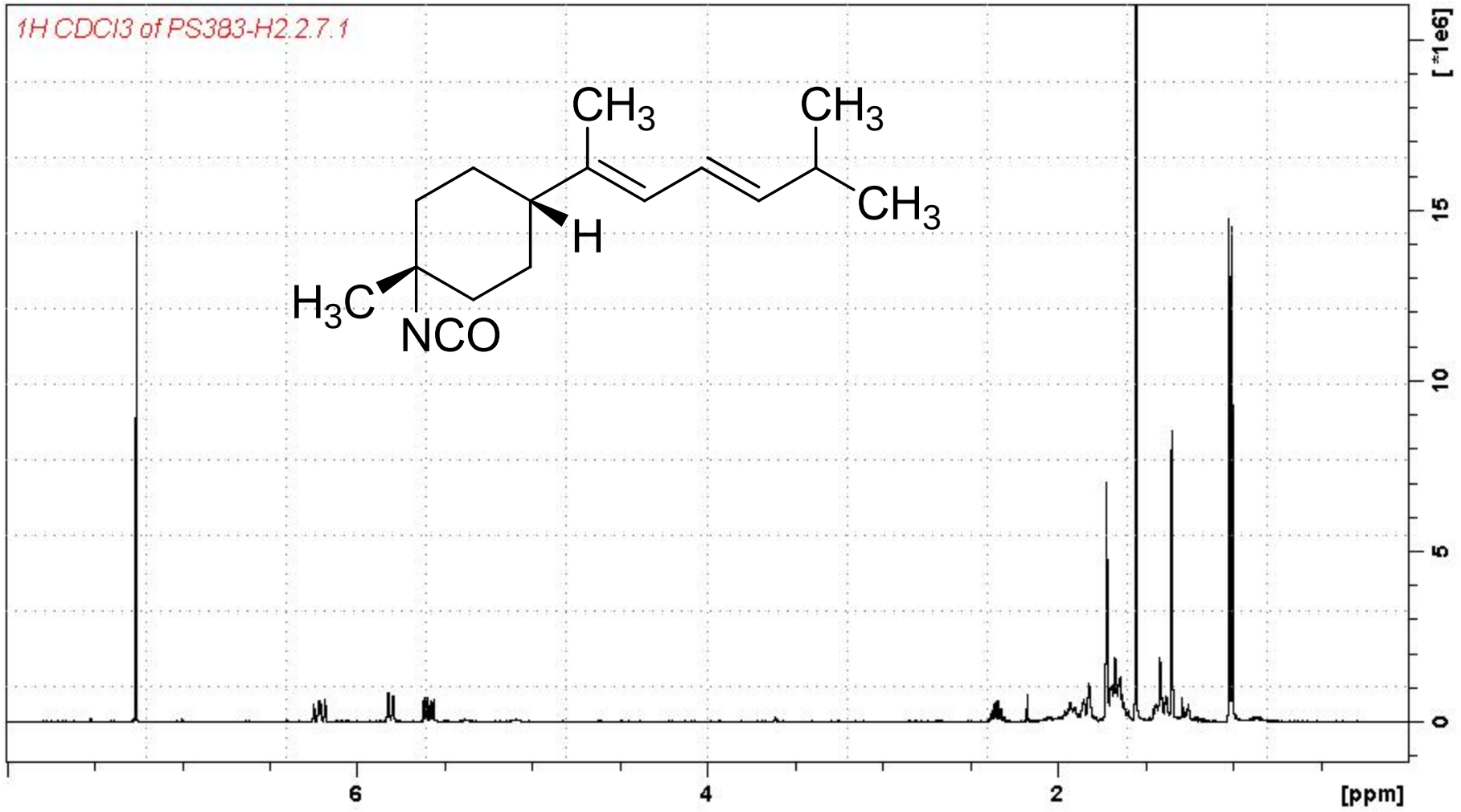
***Rhaphoxya* sp.**

Table S1. ^{1}H (400 MHz $CDCl_3$) and ^{13}C NMR(100 MHz $CDCl_3$) Spectroscopic Data for **3**, **4** and **10**[a]

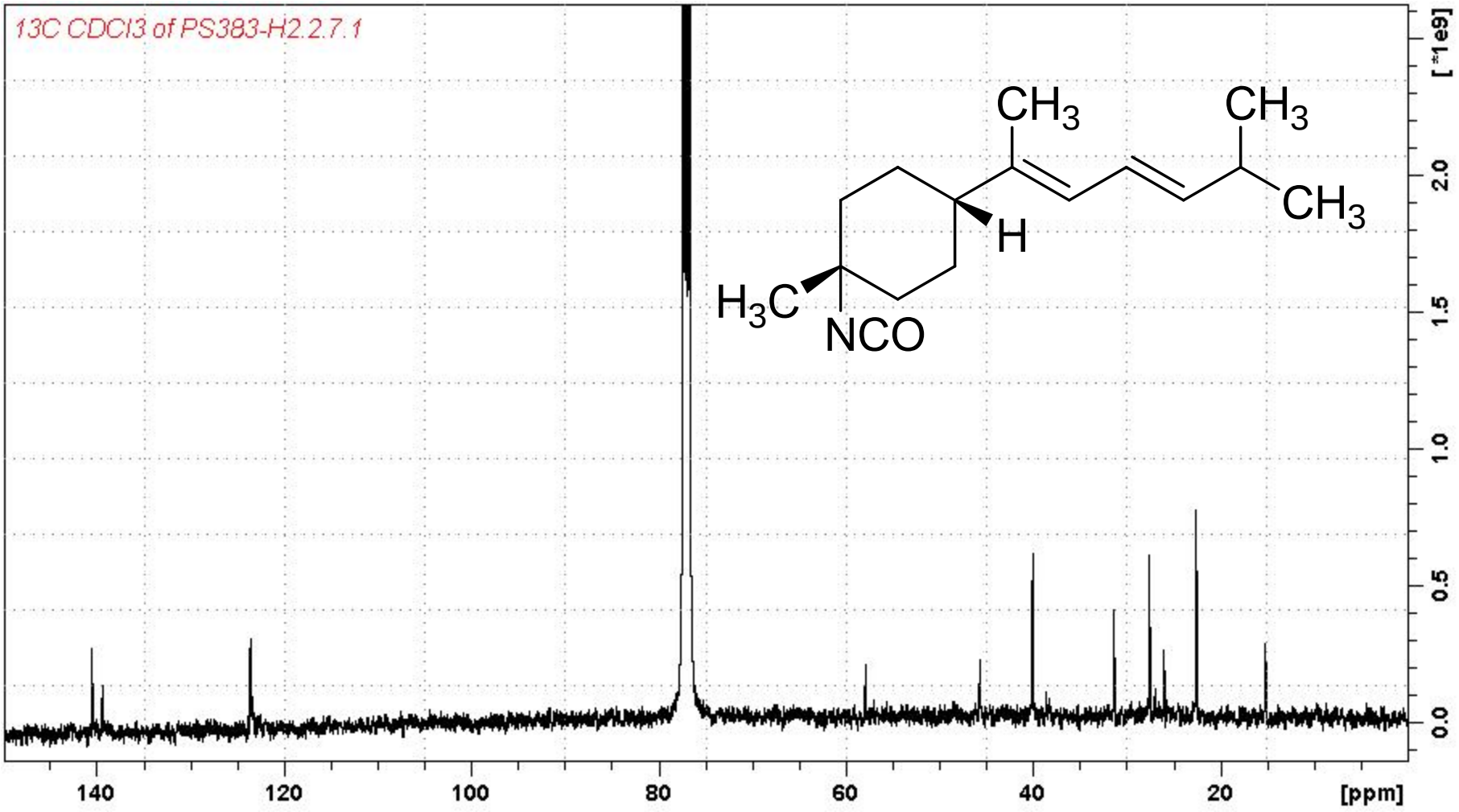
No.	δ_C, mult. (**3**)	δ_H (*J* in Hz) (**3**)	δ_C, mult. (**4**)	δ_H (*J* in Hz) (**4**)	δ_C, mult. (**10**)	δ_H (*J* in Hz) (**10**)
1	26.4, CH_2	1.42, m; 1.71, m	24.1, CH_2	1.30, m; 1.83, m	145.4, CH	7.00, s
2	38.2, CH_2	1.81, m; 1.91, m	31.0, CH_2	2.02, m	103.5, C	
3	56.7, C, t, 4.0 Hz		134.2 C		148.5, C	
4	38.2, CH_2	1.81, m; 1.91, m	120.1, CH	5.38, brm	104.6, C	
5	26.4, CH_2	1.42, m; 1.71, m	27.1, CH_2	1.92, m; 2.11, m	37.1, CH_2	3.09, d, (16.0) 3.38, d, (16.0)
6	44.7, CH	1.97, m	43.4, CH	1.72, m	121.3, C	
7	138.6, C		67.1, C		78.8, CH	5.03, s
8	123.8, CH	5.79, brd. (10.7)	39.3, CH_2	1.60, m; 1.70, m	156.7, C	
9	123.4, CH	6.20, ddd, (1.3, 10.7, 15.2)	23.0, CH_2	2.09, m	158.8, C	
10	140.7, CH	5.59, dd, (6.9, 15.2)	123.2, CH	5.09, brt, (6.7)	37.0, CH_2	3.68, m
11	31.4, CH	2.34, dqq, (6.9, 6.7, 6.7)	132.8, C		29.3, CH_2	2.08, m
12	22.5, CH_3	1.00, d, (6.7)	26.1, CH_3	1.70, brs	70.8, CH_2	4.05, t, (5.5)
13	25.1, CH_3	1.43, brs	23.5, CH_3	1.65, brs	151.0, C	
14	15.2, CH_3	1.71, brs	23.7, CH_3	1.35, s	118.0, C	
15	22.5, CH_3	1.01, d, (6.7)	18.0, CH_3	1.63, brs	132.8, CH	
16	152.1, NC		129.7, NCS		138.6, C	
17					132.8, CH	7.34, s
18					118.0, C	
19					34.3, CH_2	2.73, t, (7.0)
20					52.1, CH_2	2.82, t, (7.0)
21					59.0, CH_3	3.67, s
NCH_3					57.0, CH_3	2.44, s
CONH						7.26, s
NH						3.68, s
OH						4.10, s

[a]All assignments are based on interpretation of extensive 1D and 2D NMR measurements

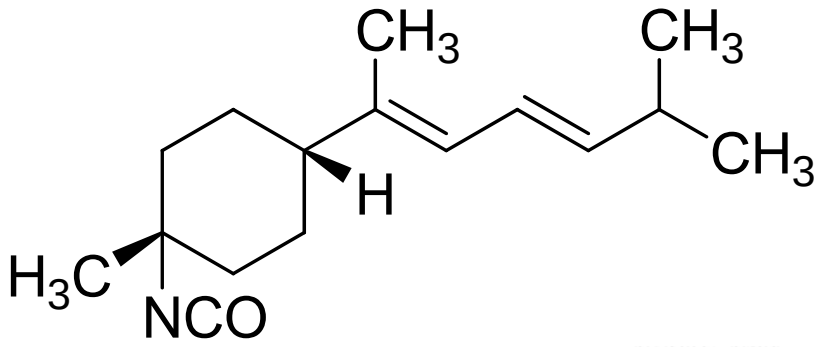
^{1}H-NMR spectrum of **1**

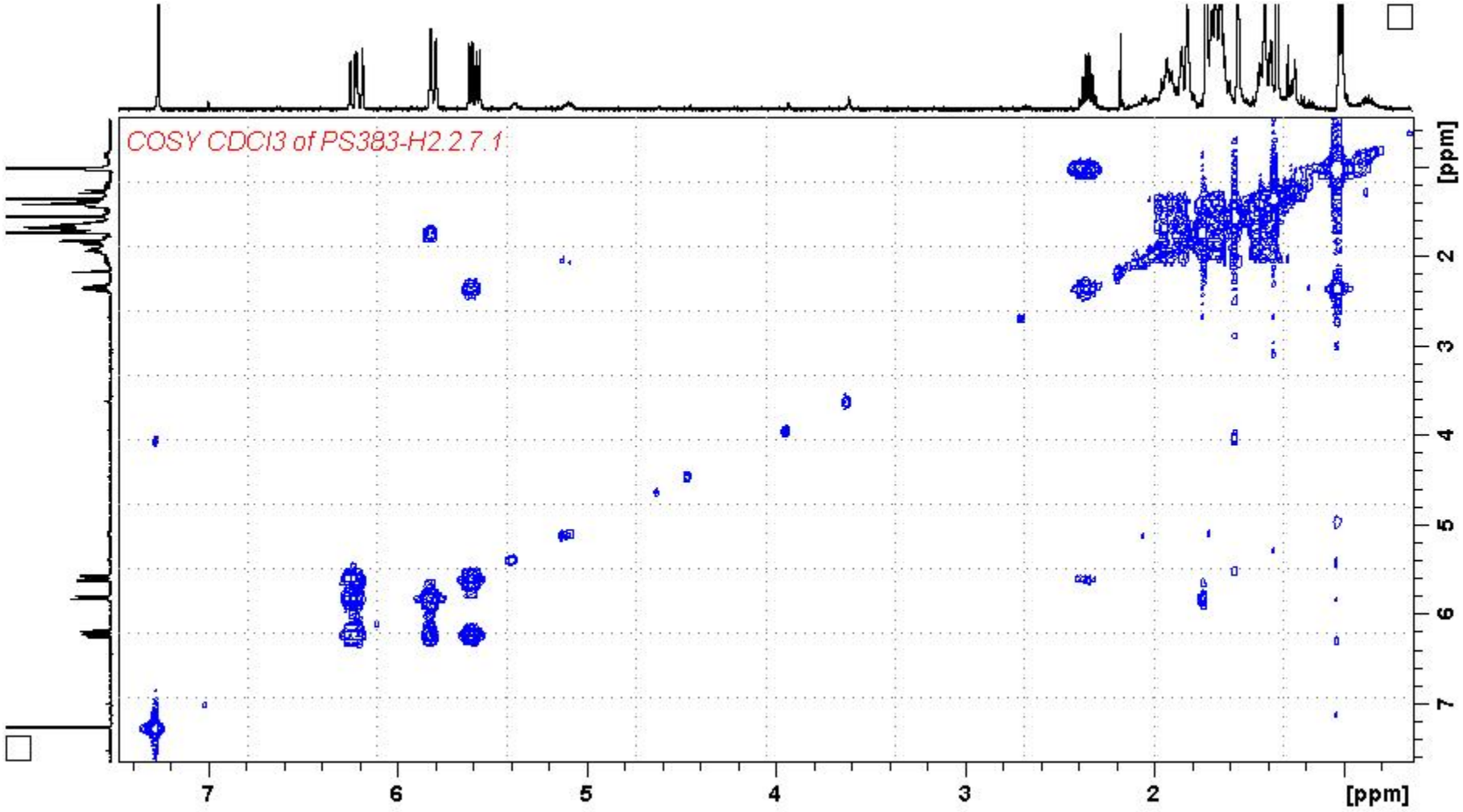


^{13}C-NMR spectrum of **1**



COSY-NMR spectrum of **1**





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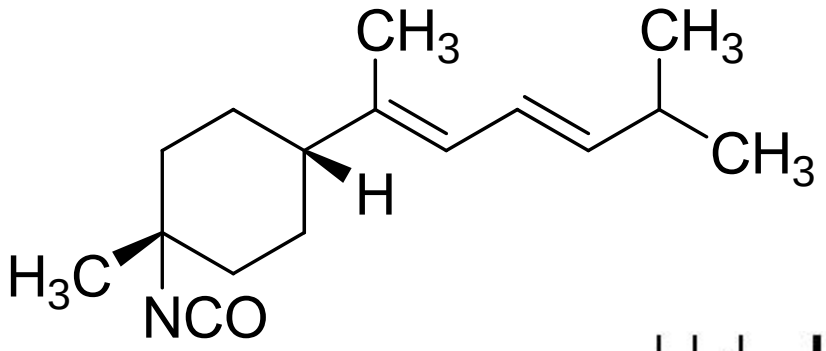
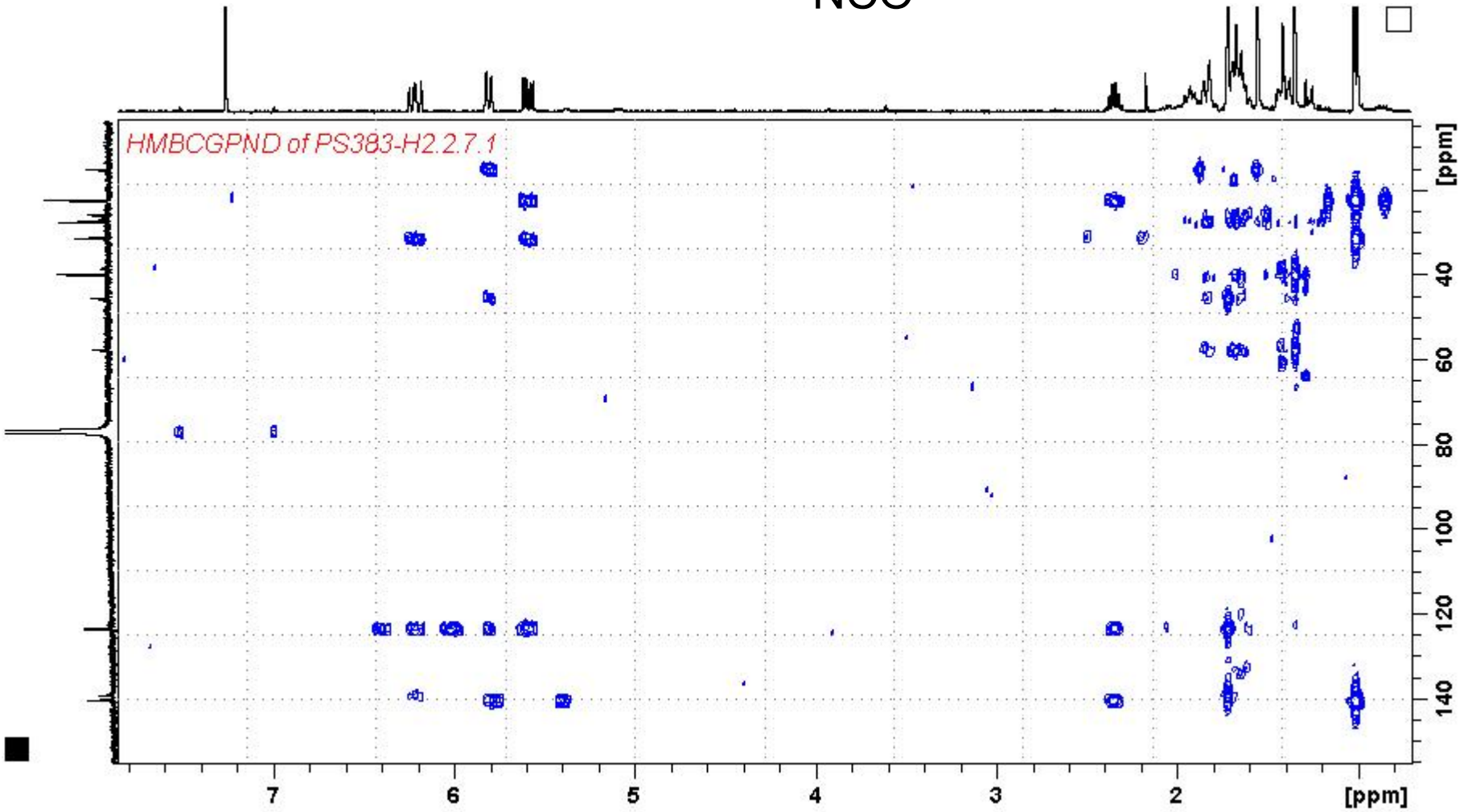
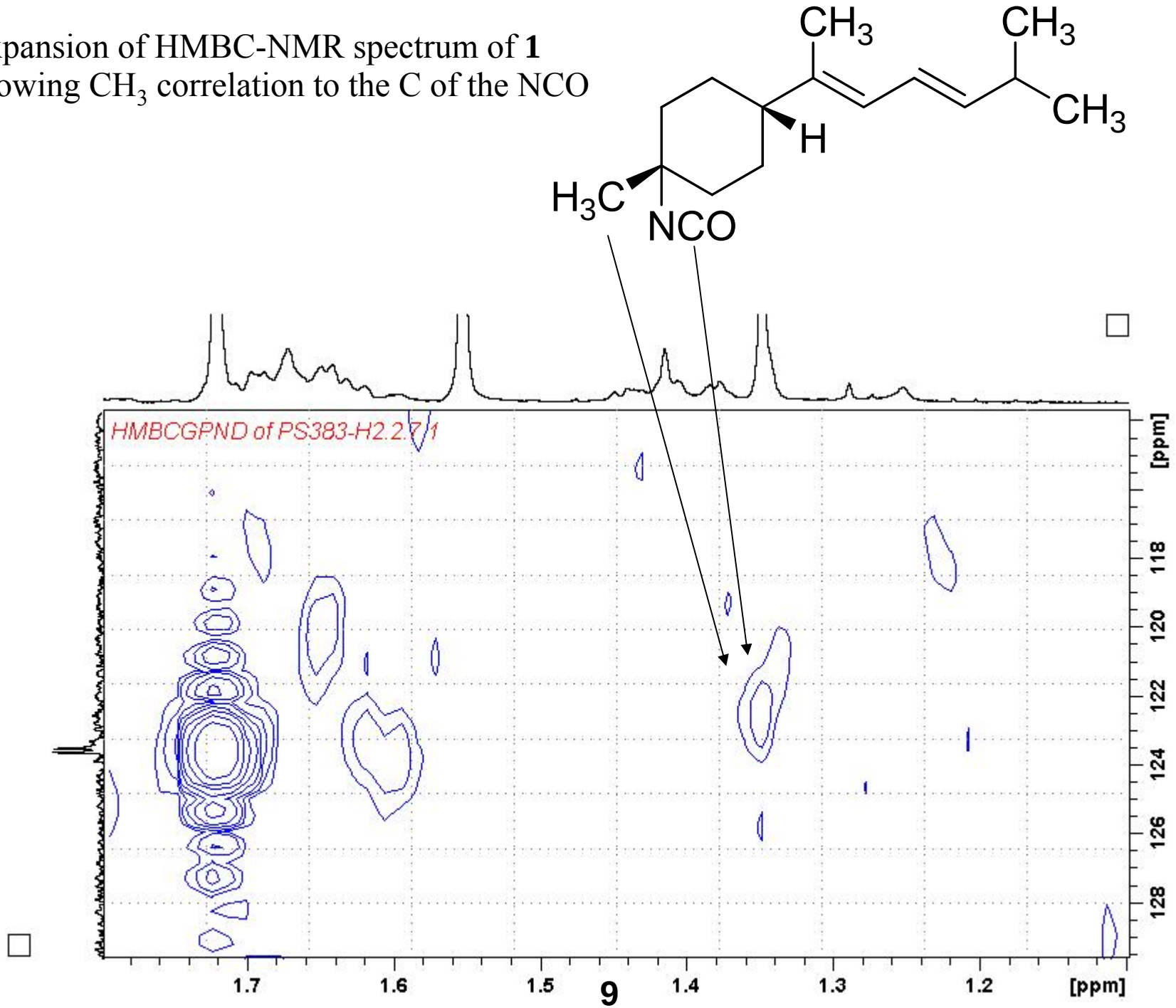
HMBC-NMR spectrum of **1**





Expansion of HMBC-NMR spectrum of **1** showing CH_3 correlation to the C of the NCO



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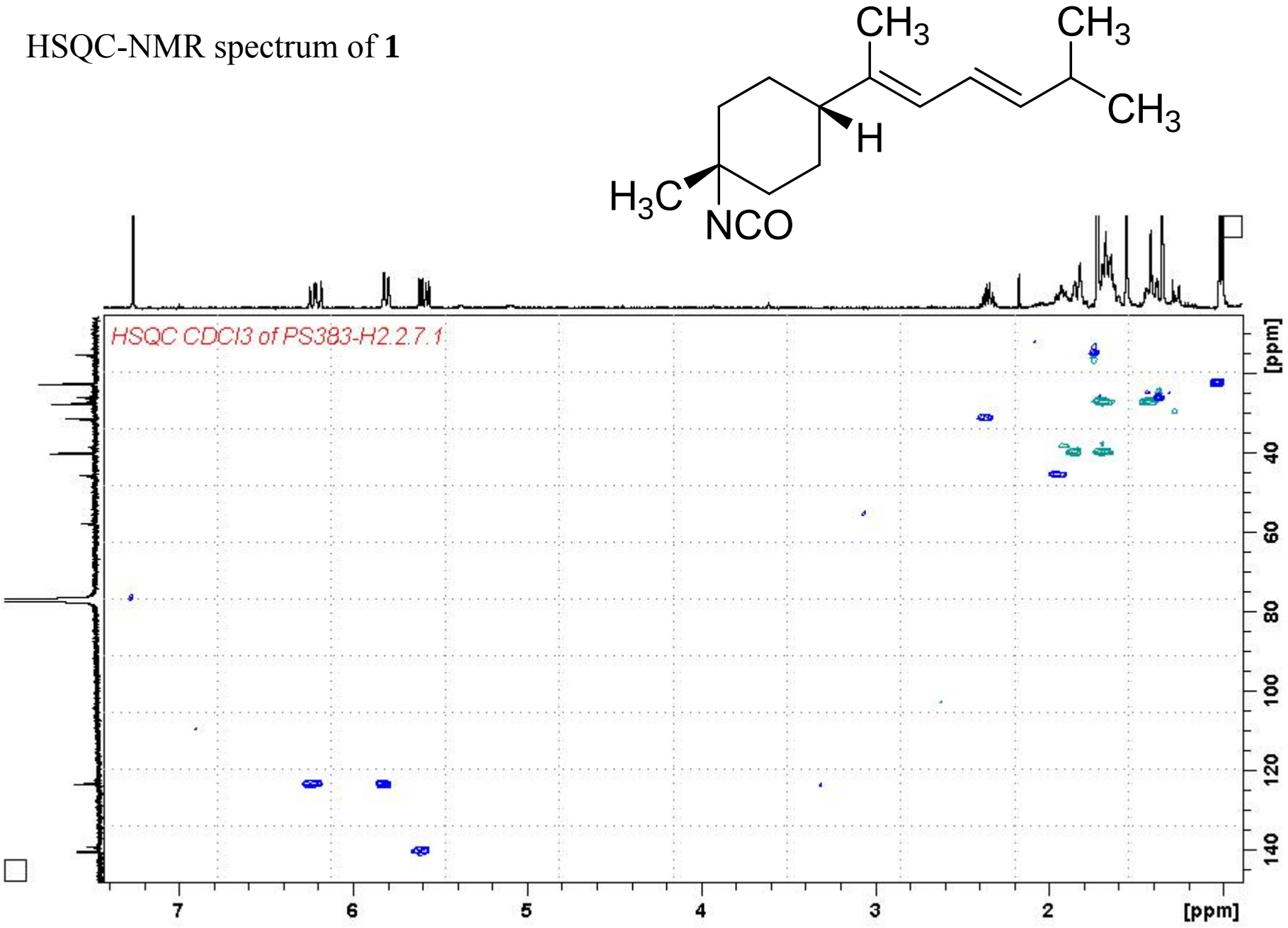
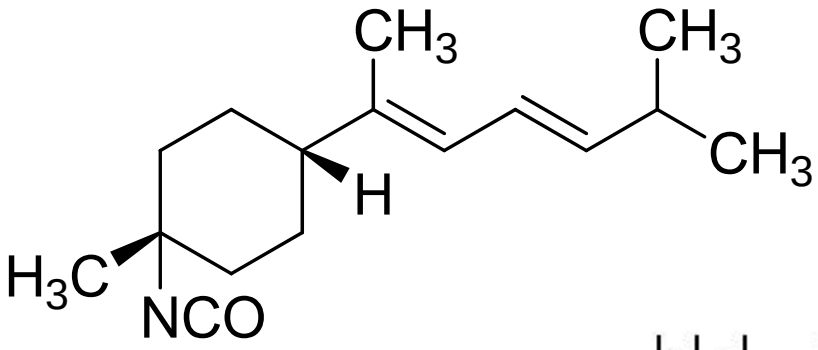
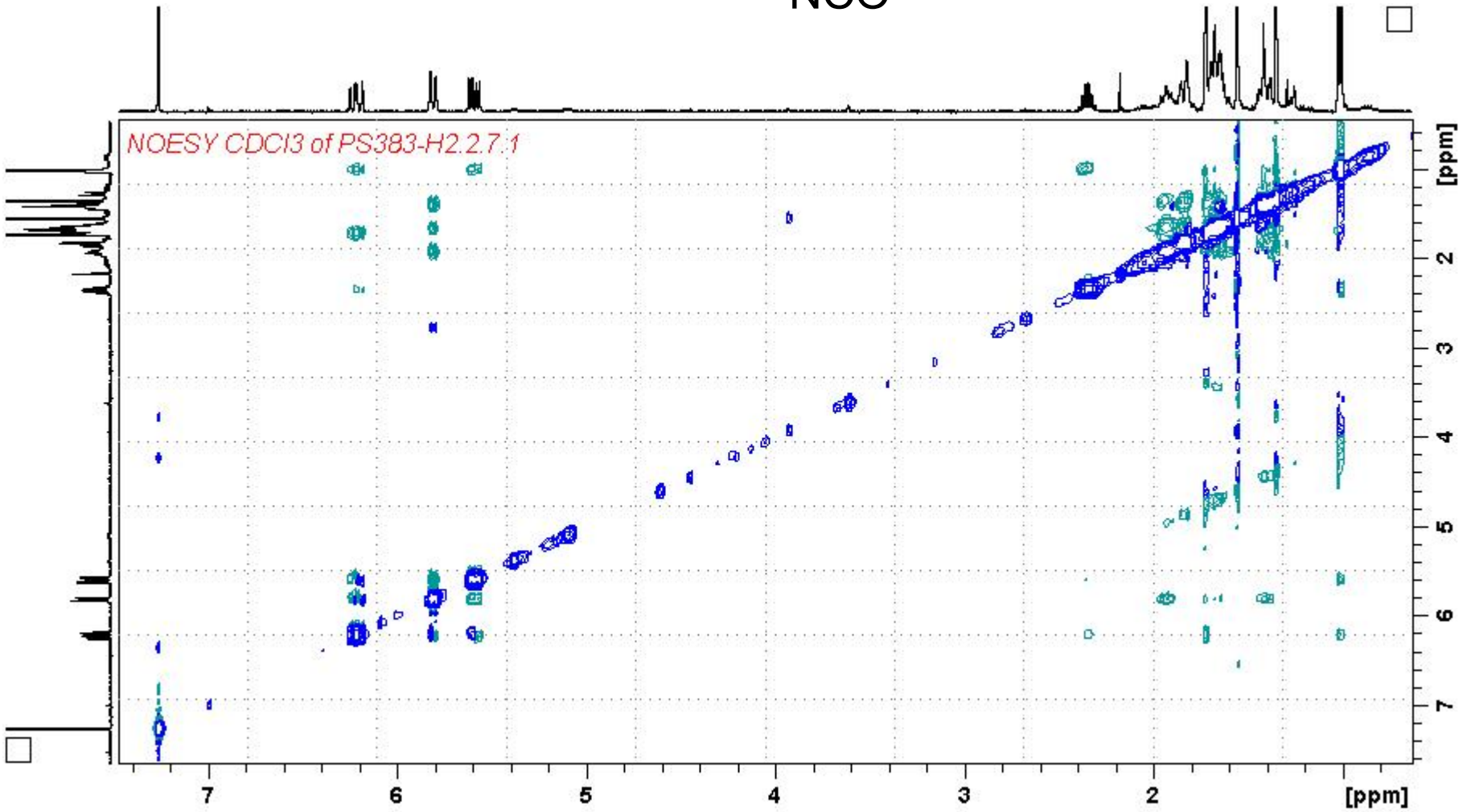
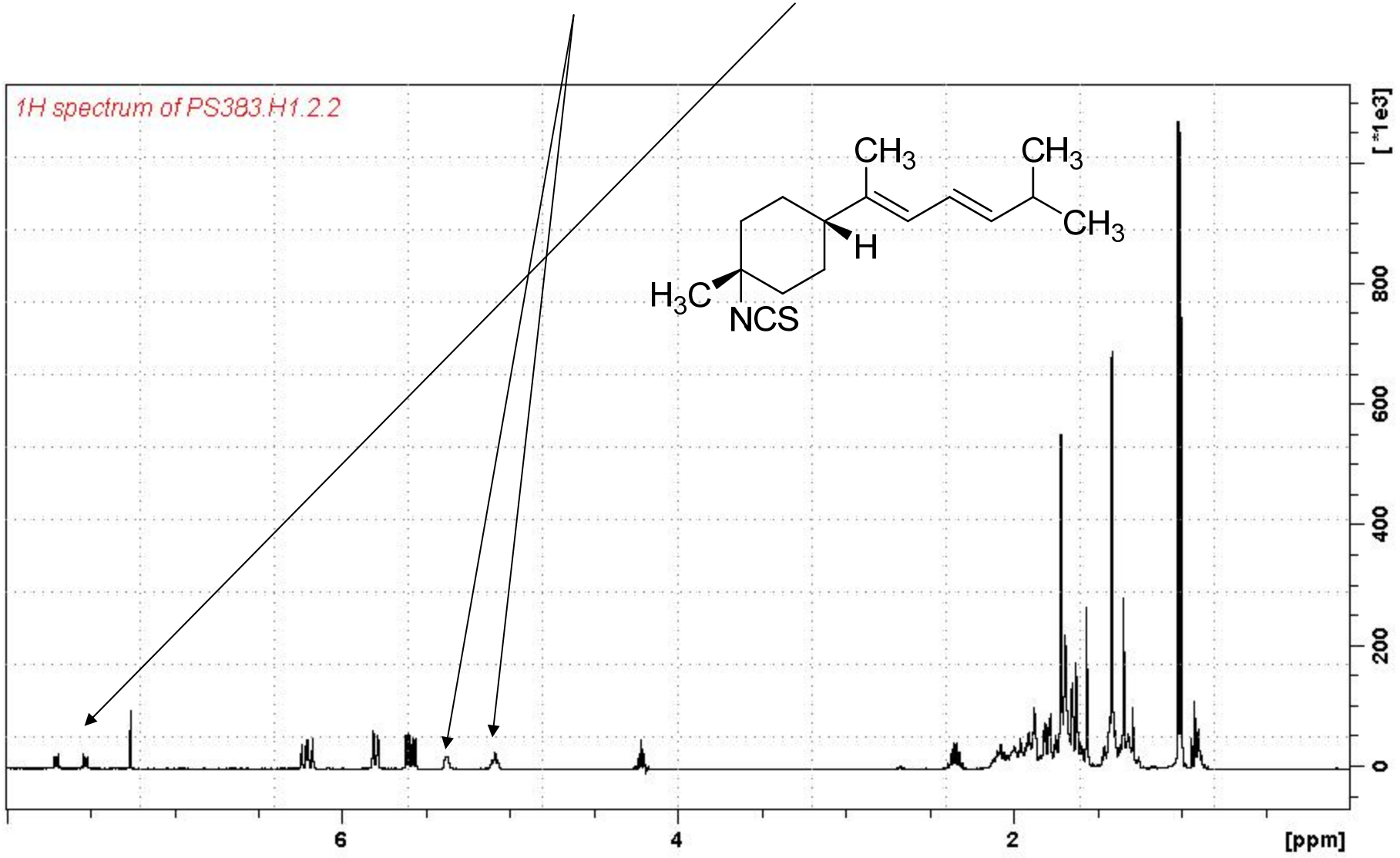


NOESY-NMR spectrum of **1**

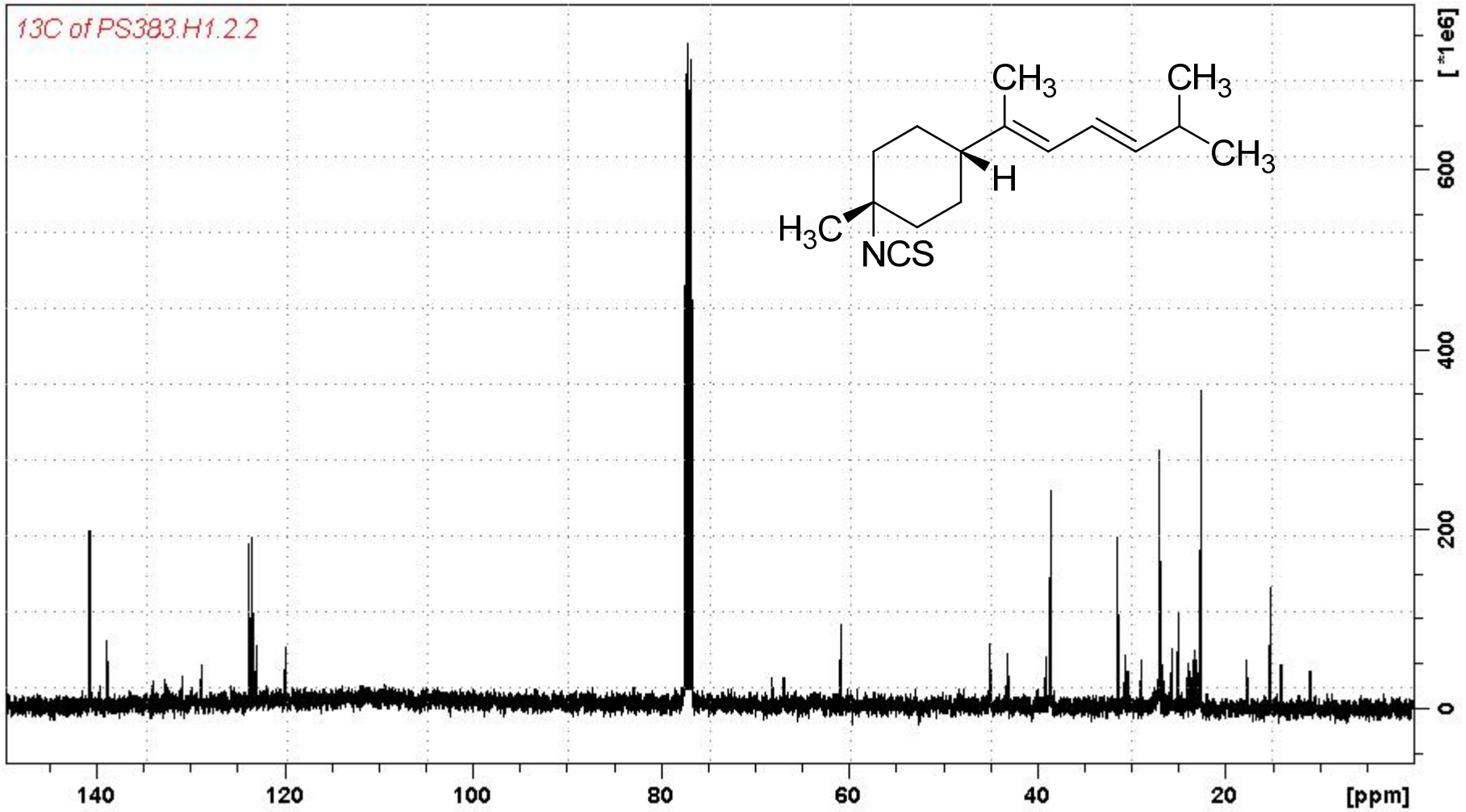




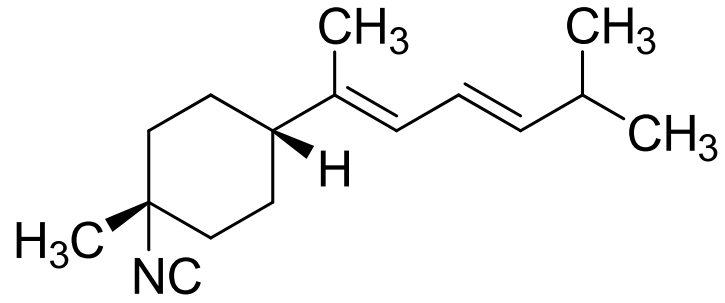
^{1}H-NMR spectrum of **2** with traces of **4** and some phthalate

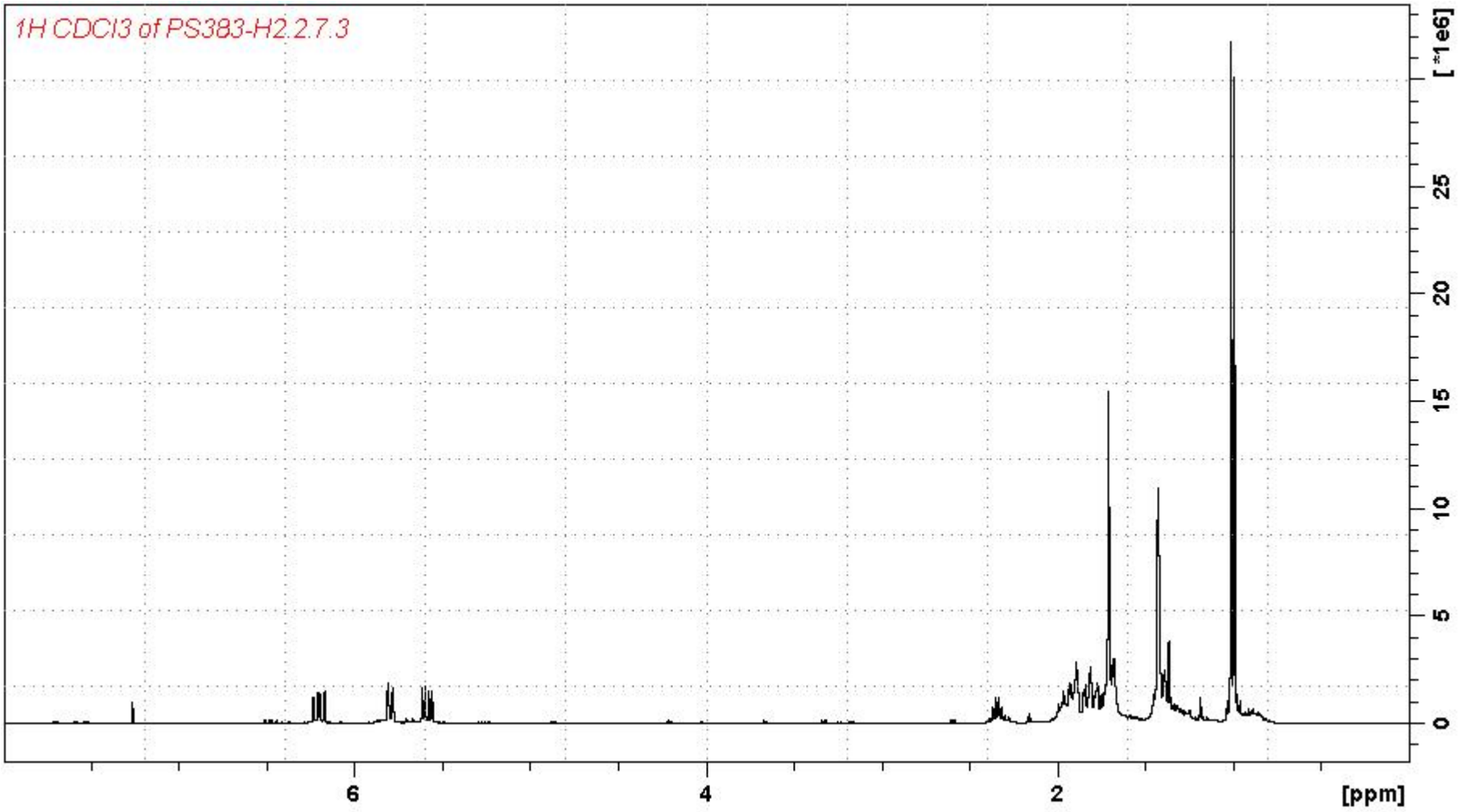


^{13}C-NMR spectrum of **2** with traces of **4** and some phthalate

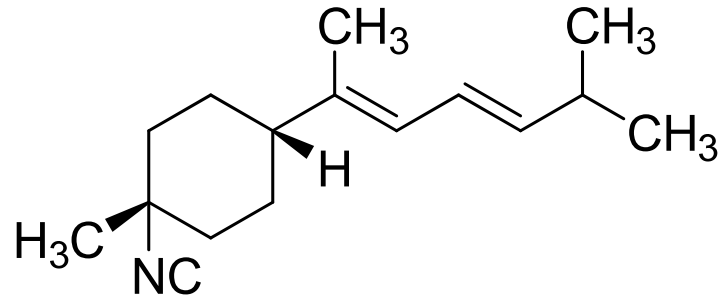


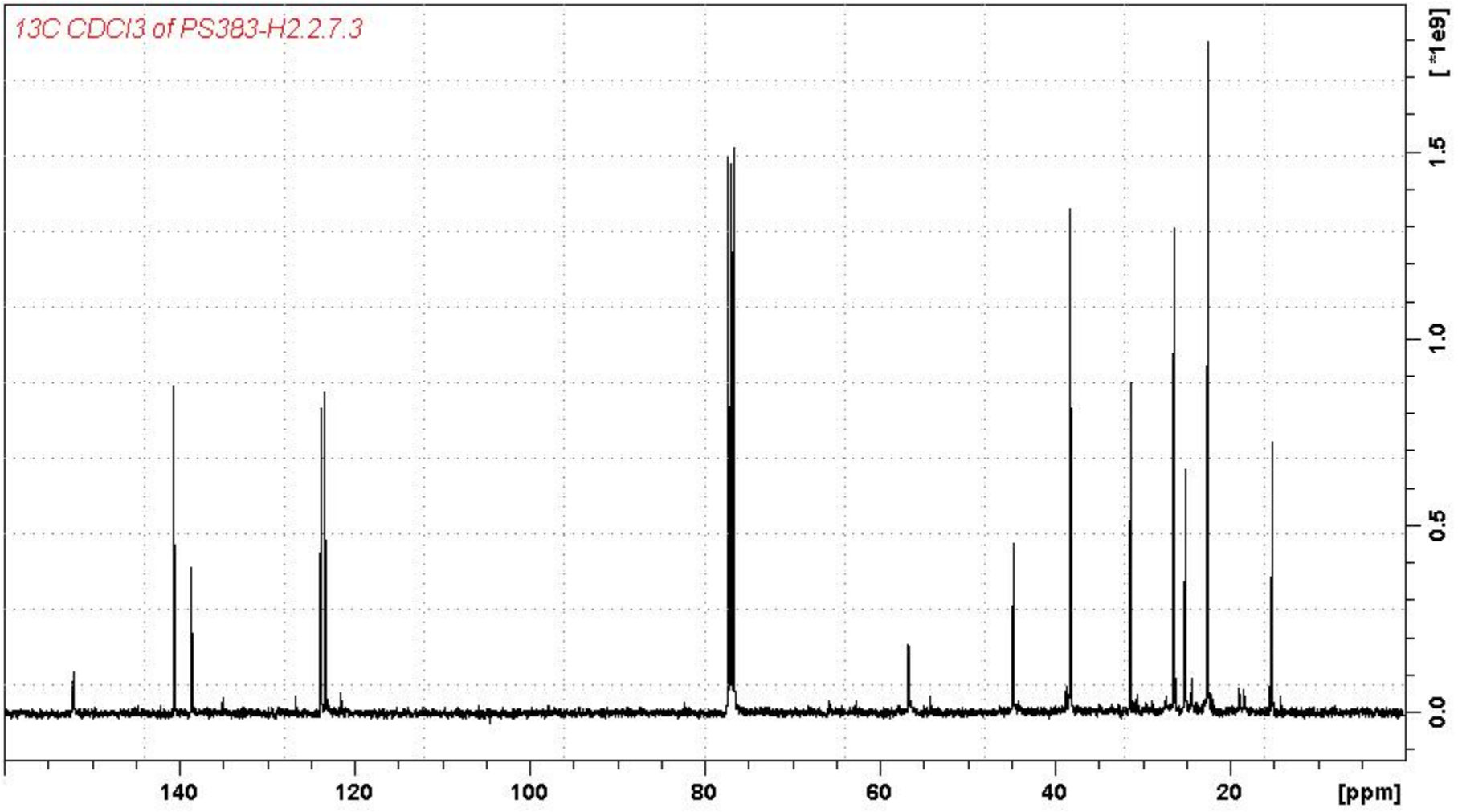
^{1}H-NMR spectrum of **3**



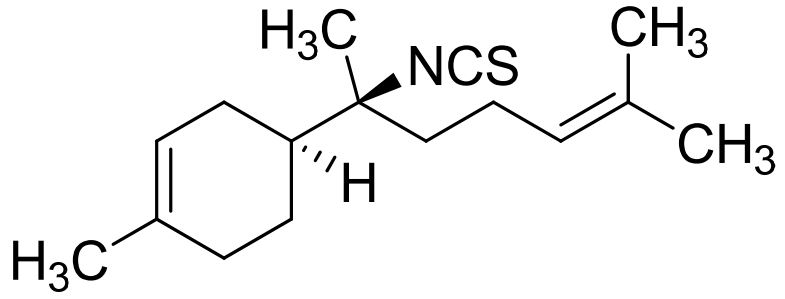


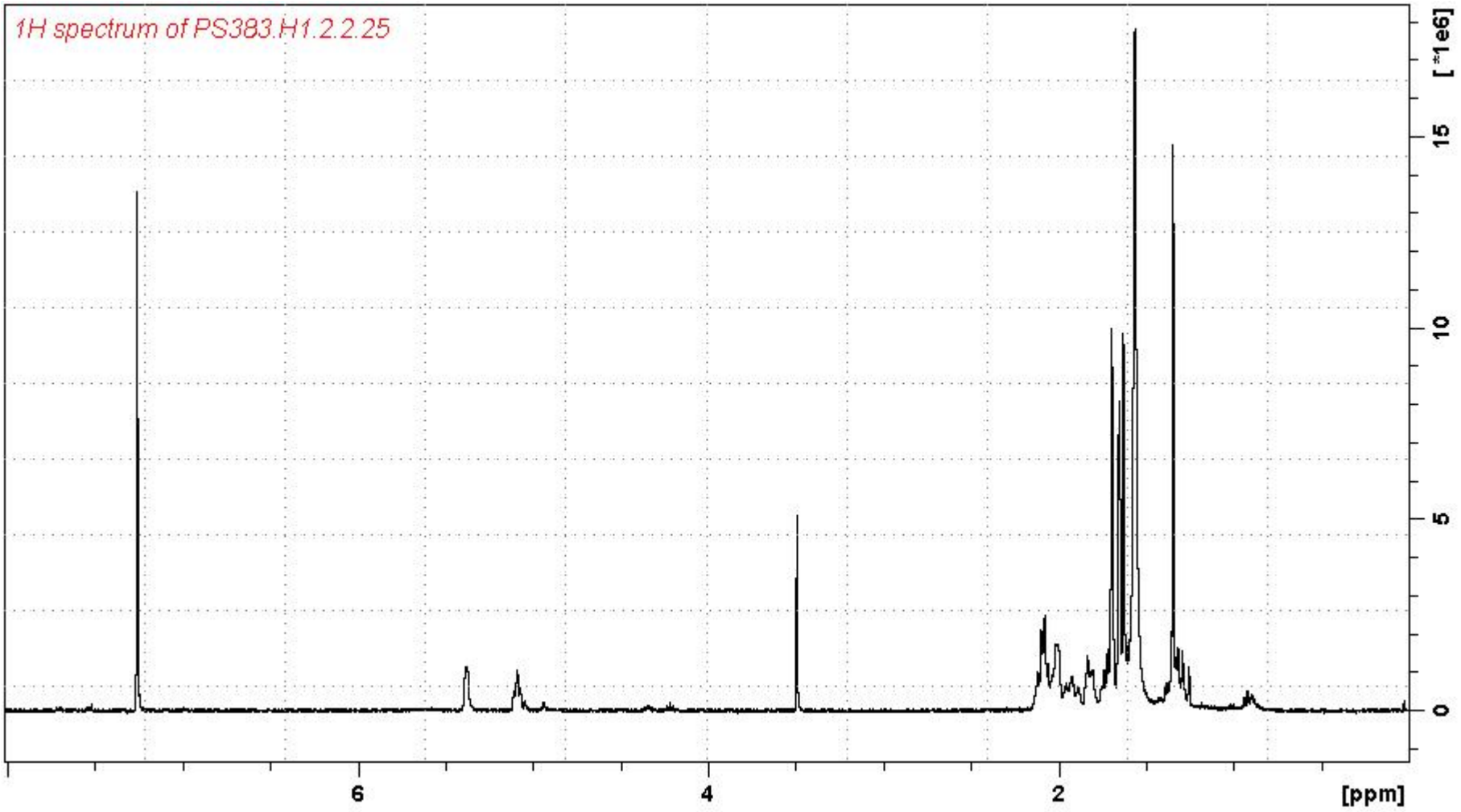
^{13}C-NMR spectrum of **3**





^{1}H-NMR spectrum of **4**





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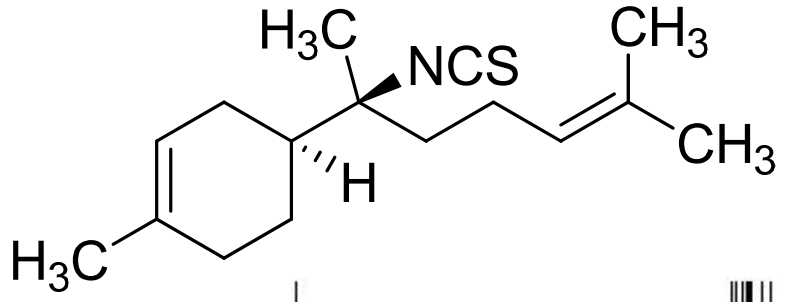
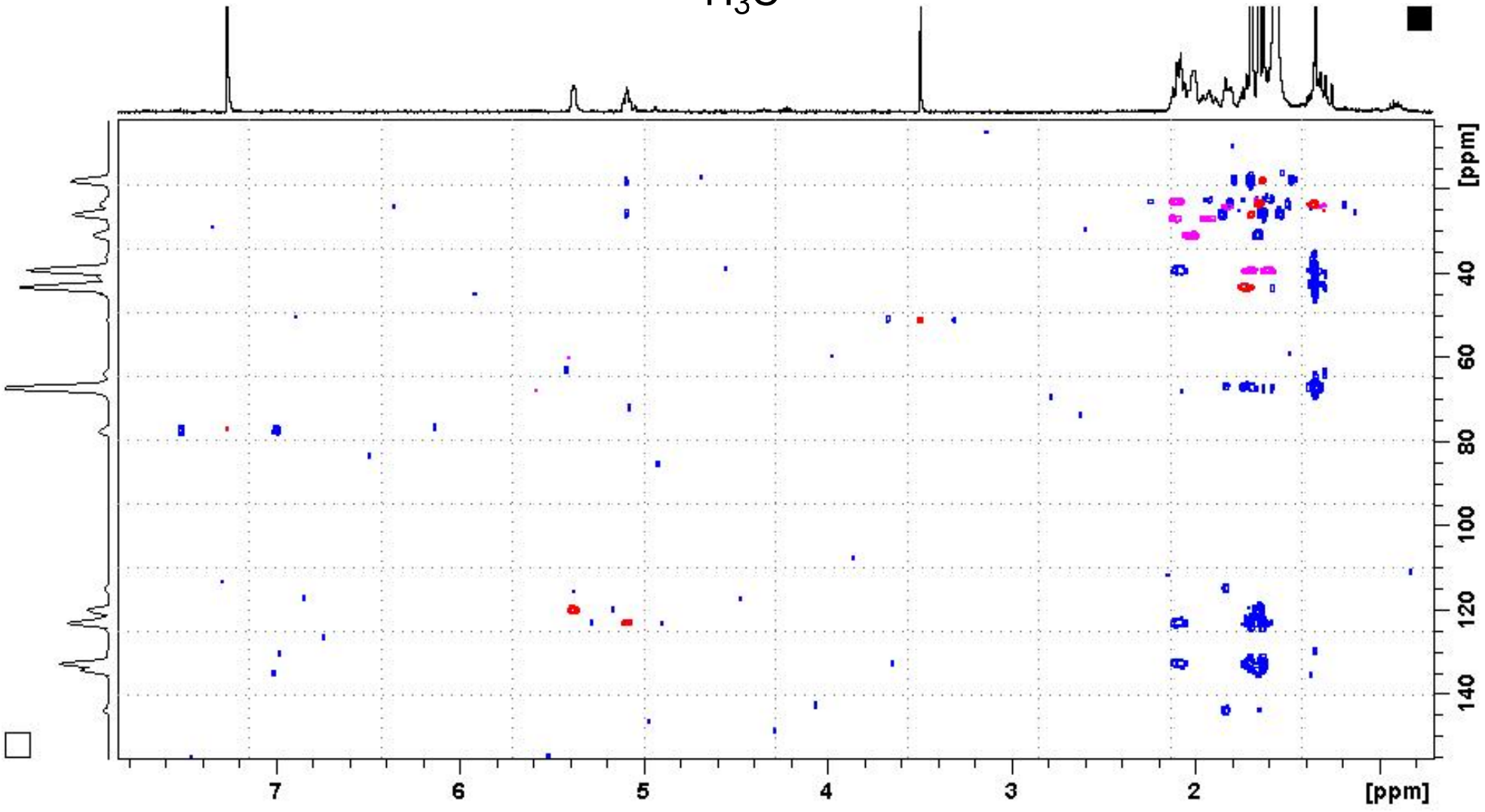
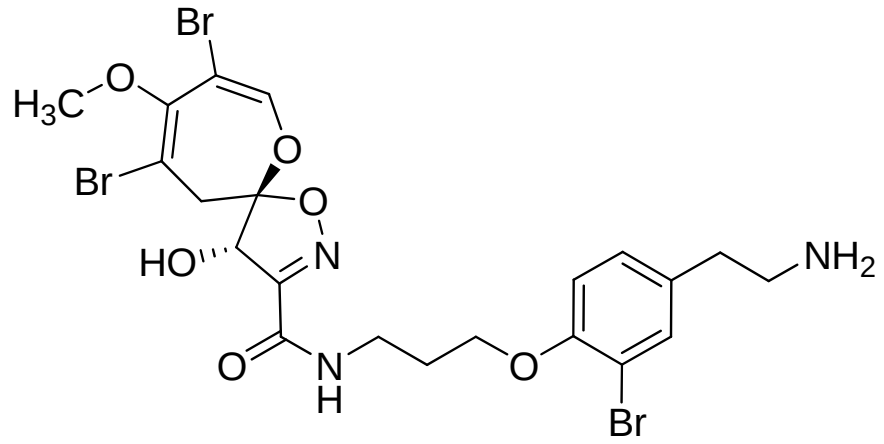
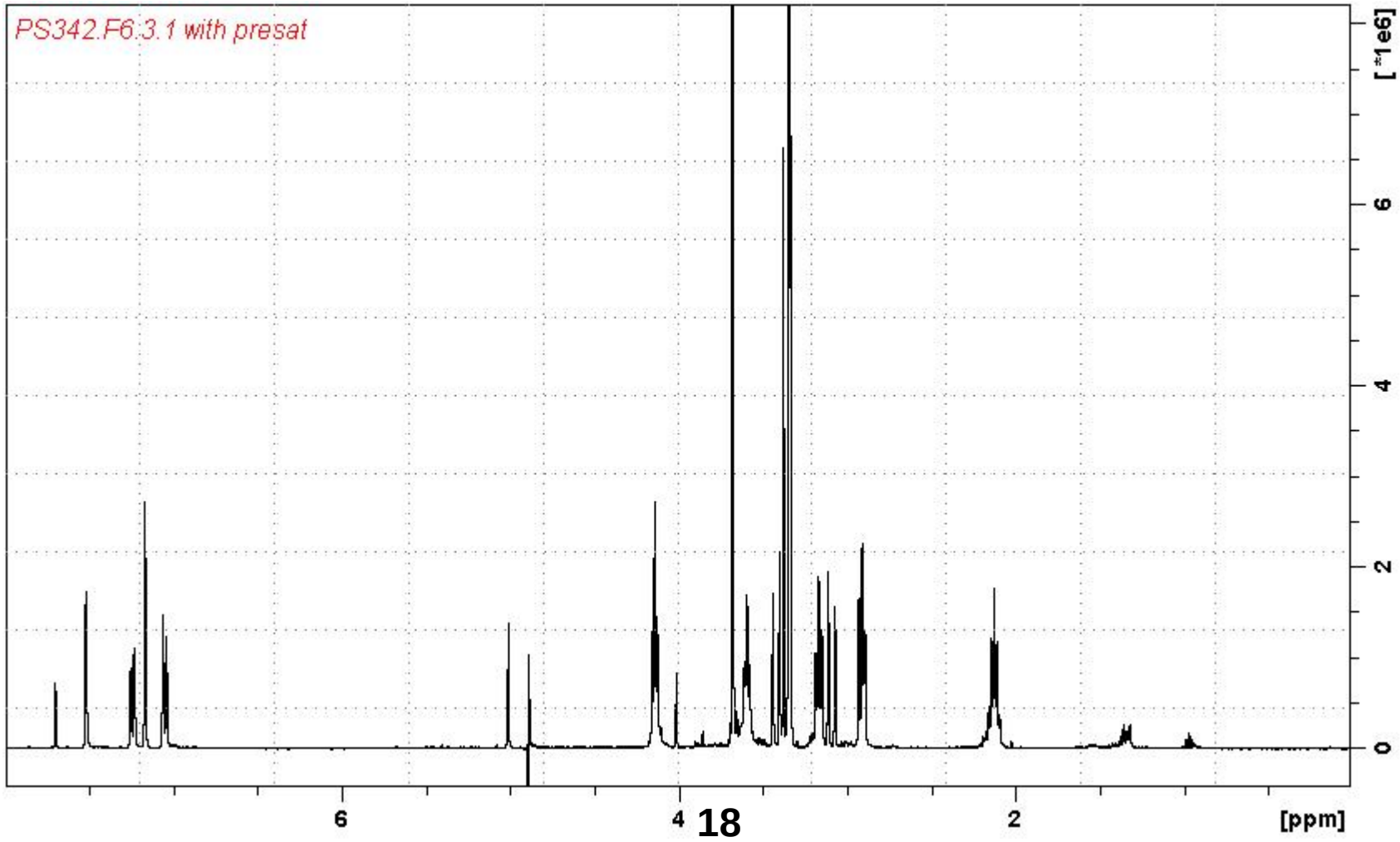
Combined HSQC (Red and purple) and HMBC (Blue)-NMR spectra of **4**



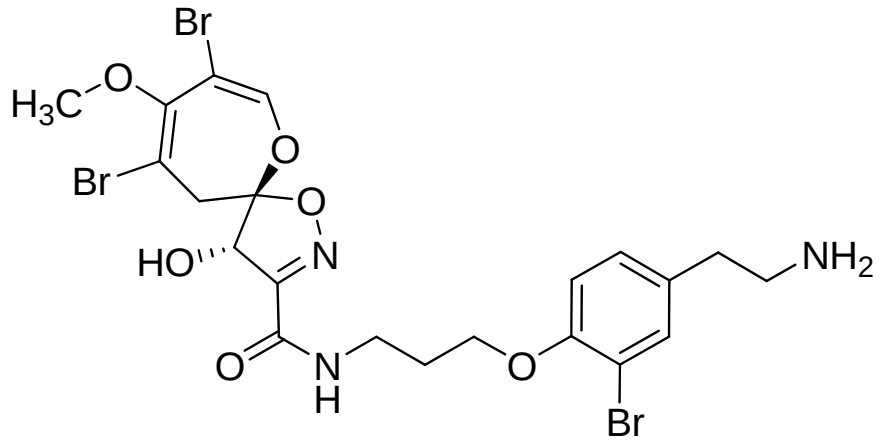


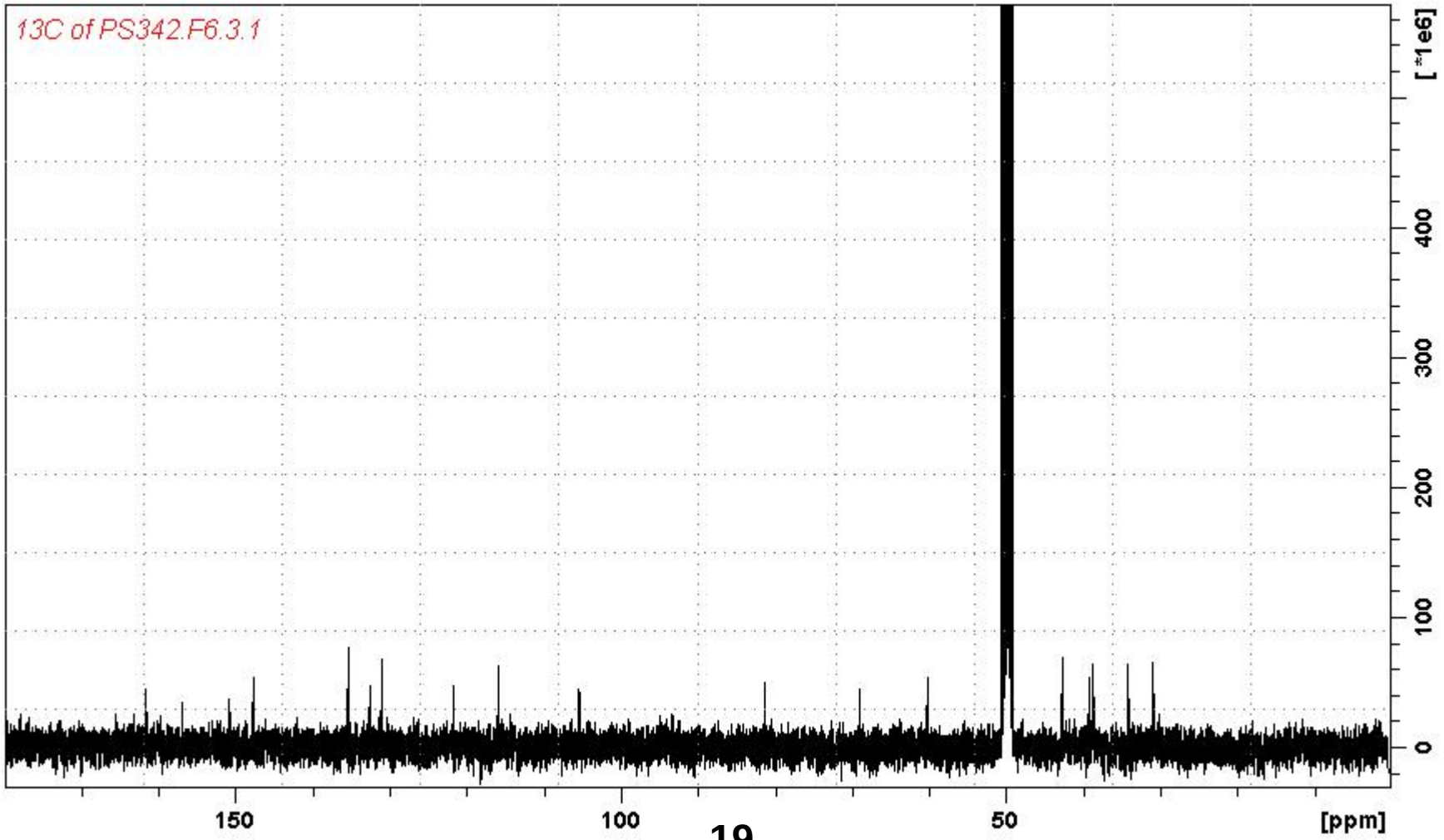
^{1}H-NMR spectrum of **5**



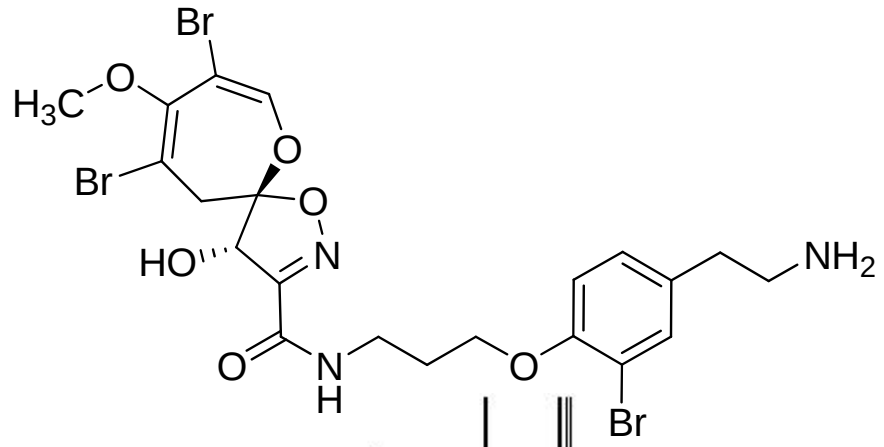


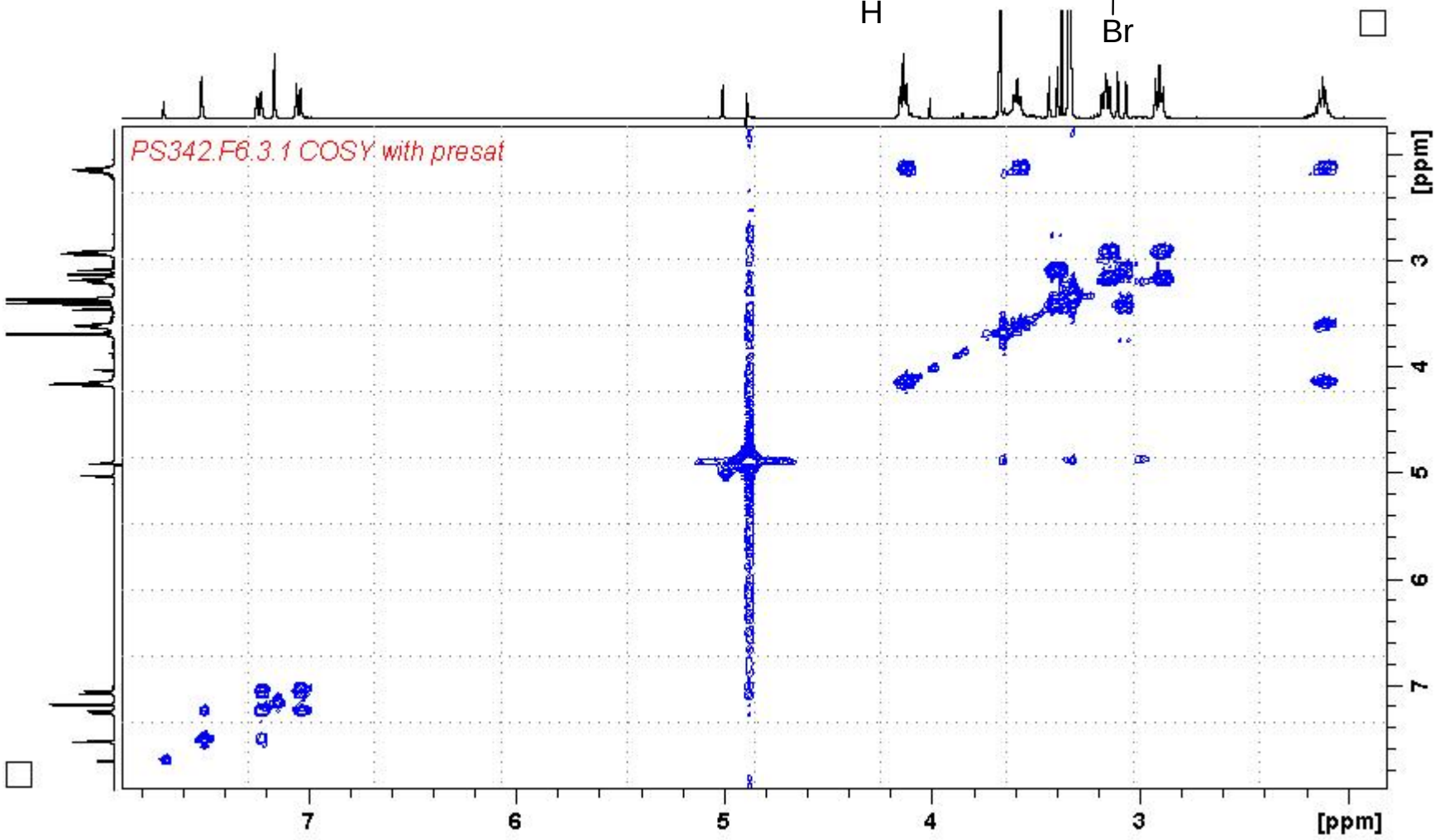
^{13}C-NMR spectrum of **5**





COSY-NMR spectrum of **5**





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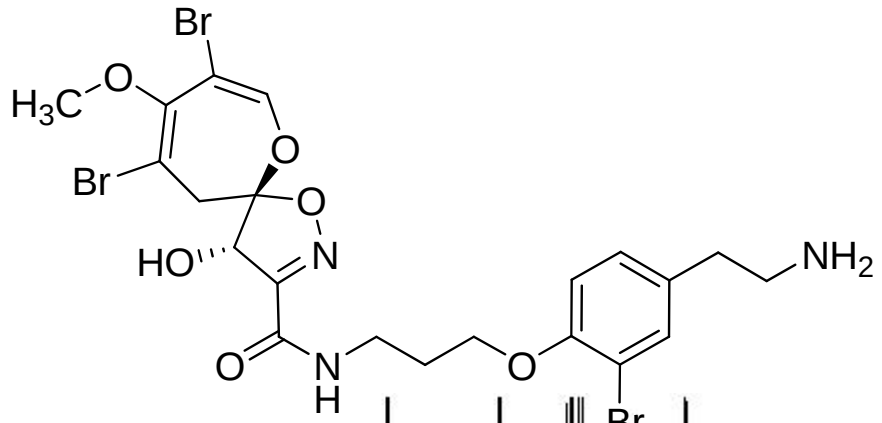
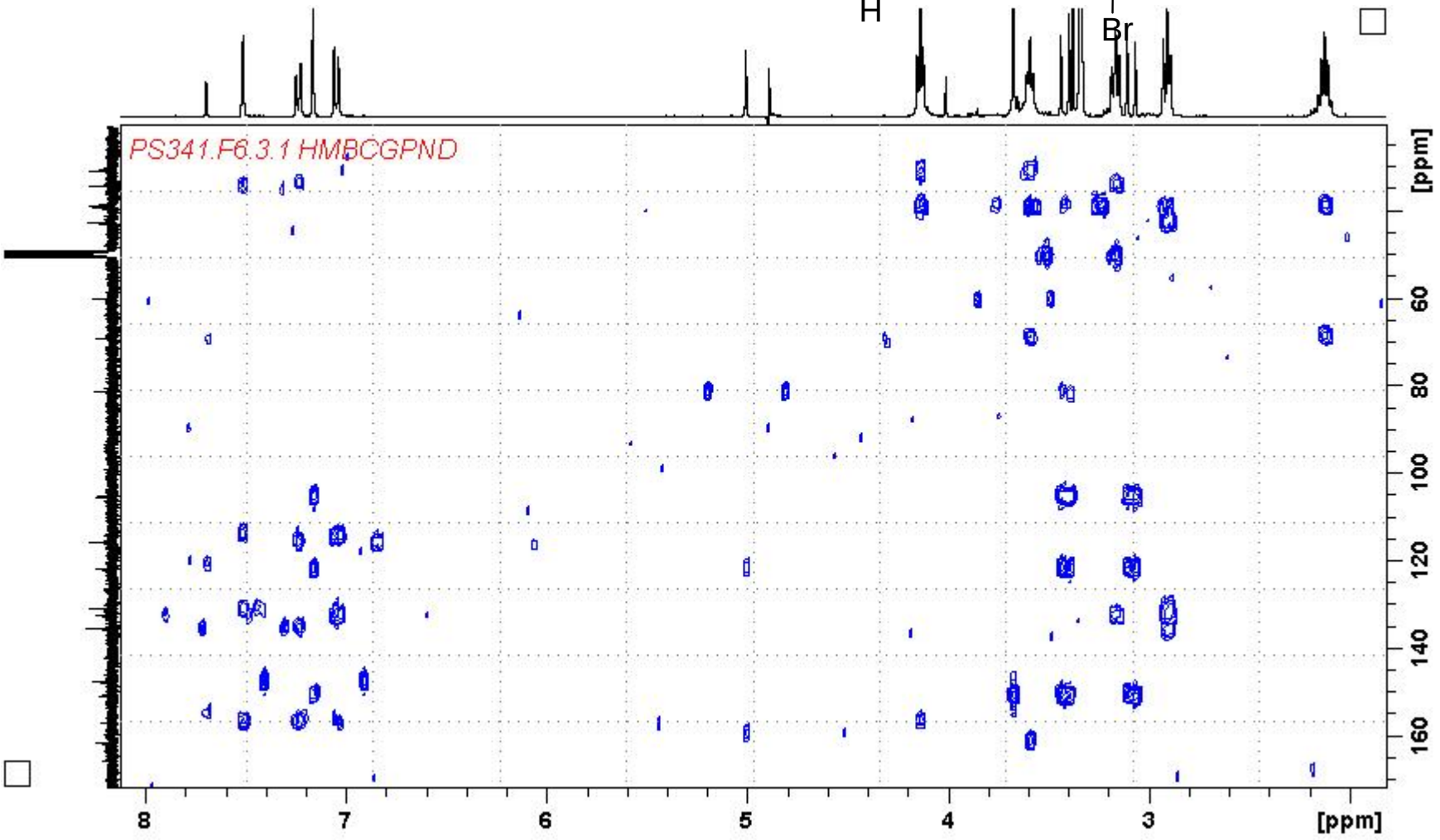
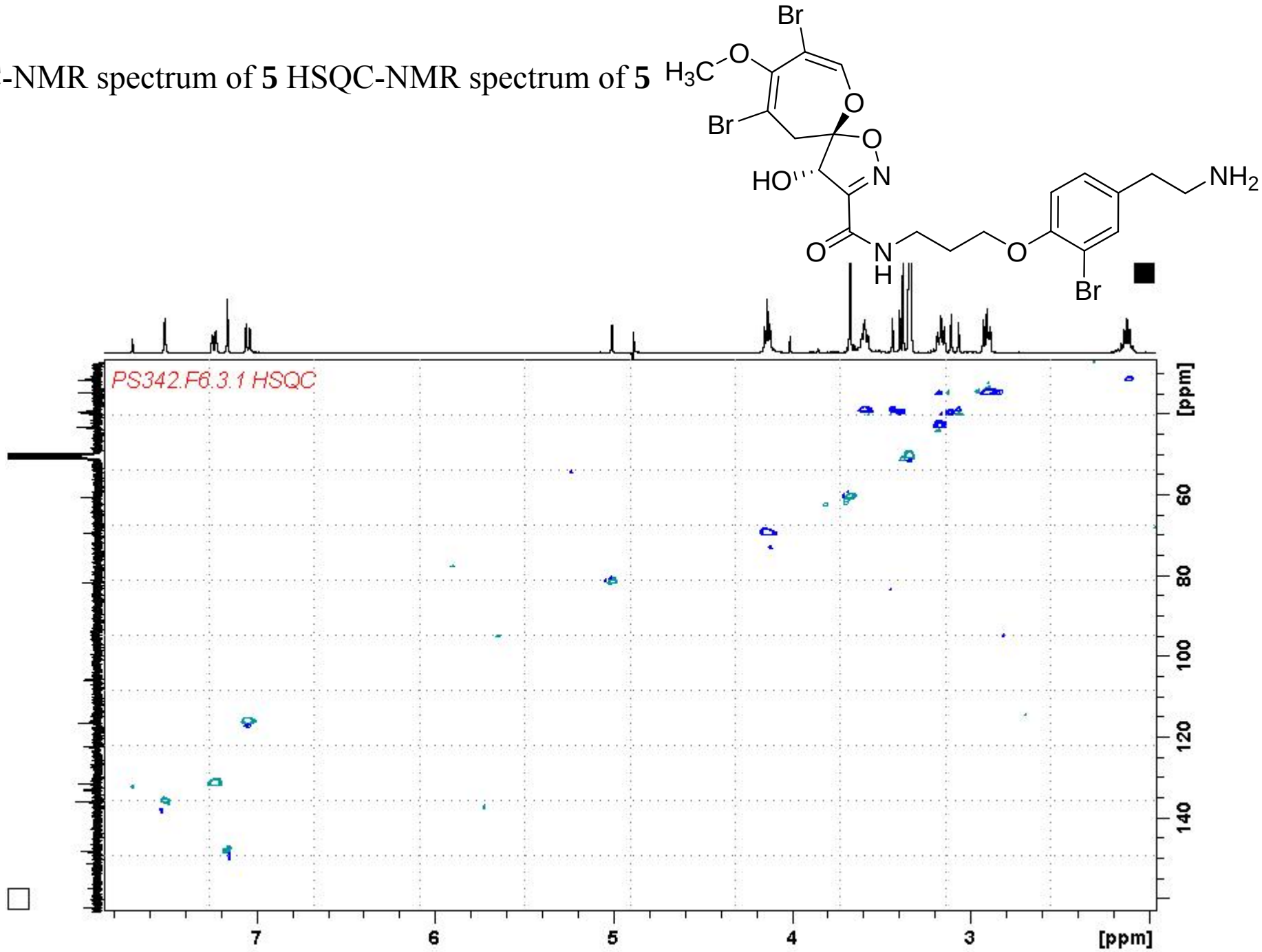
HMBC-NMR spectrum of **5**

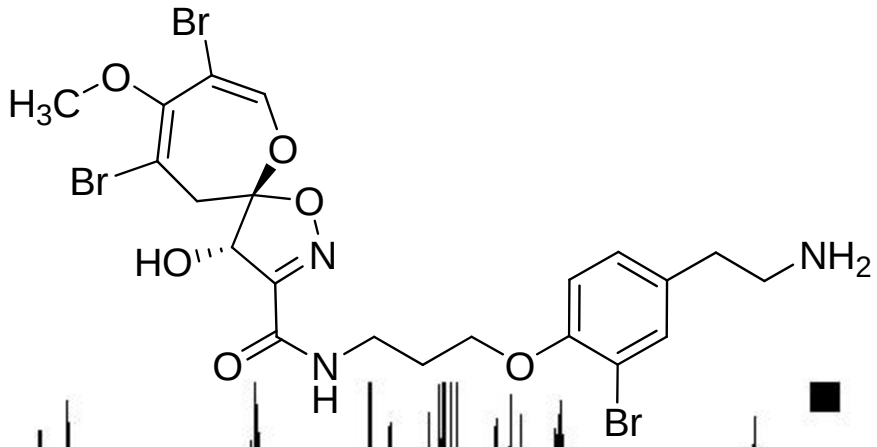


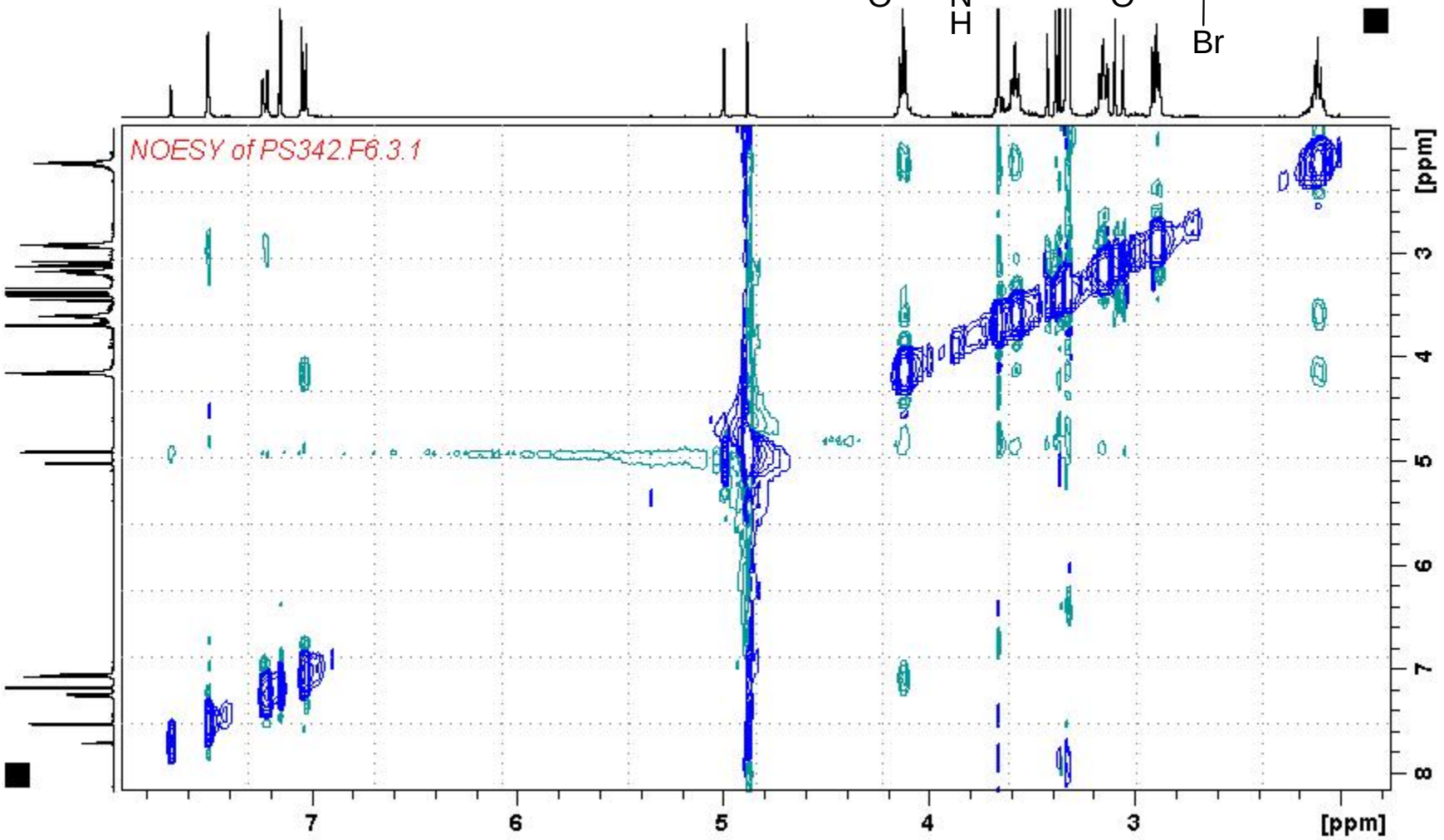


HSQC-NMR spectrum of **5** HSQC-NMR spectrum of **5**



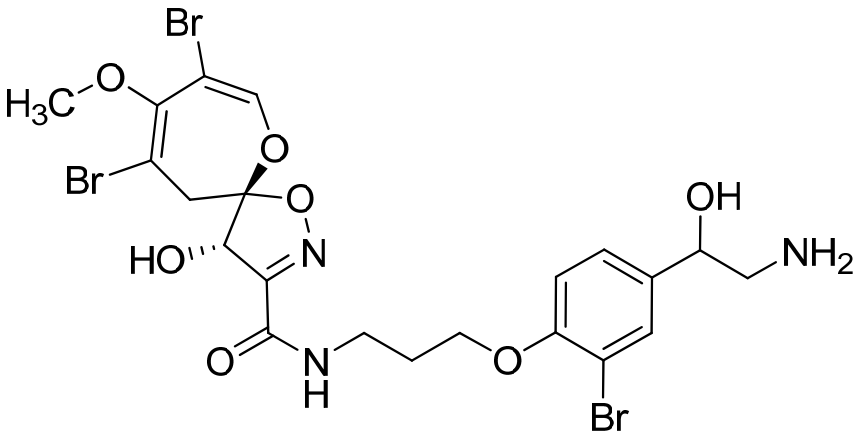
NOESY-NMR spectrum of **5**

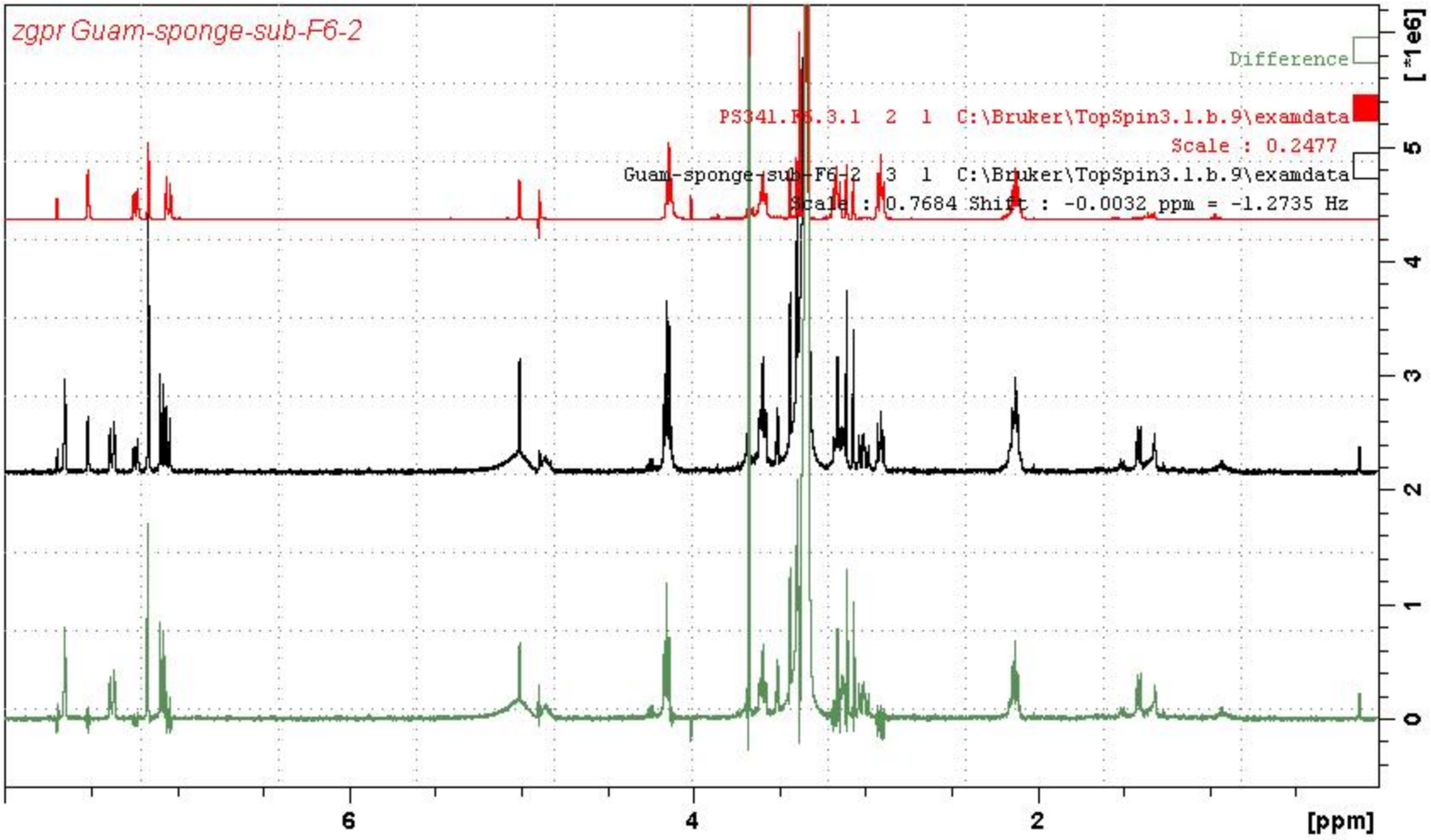




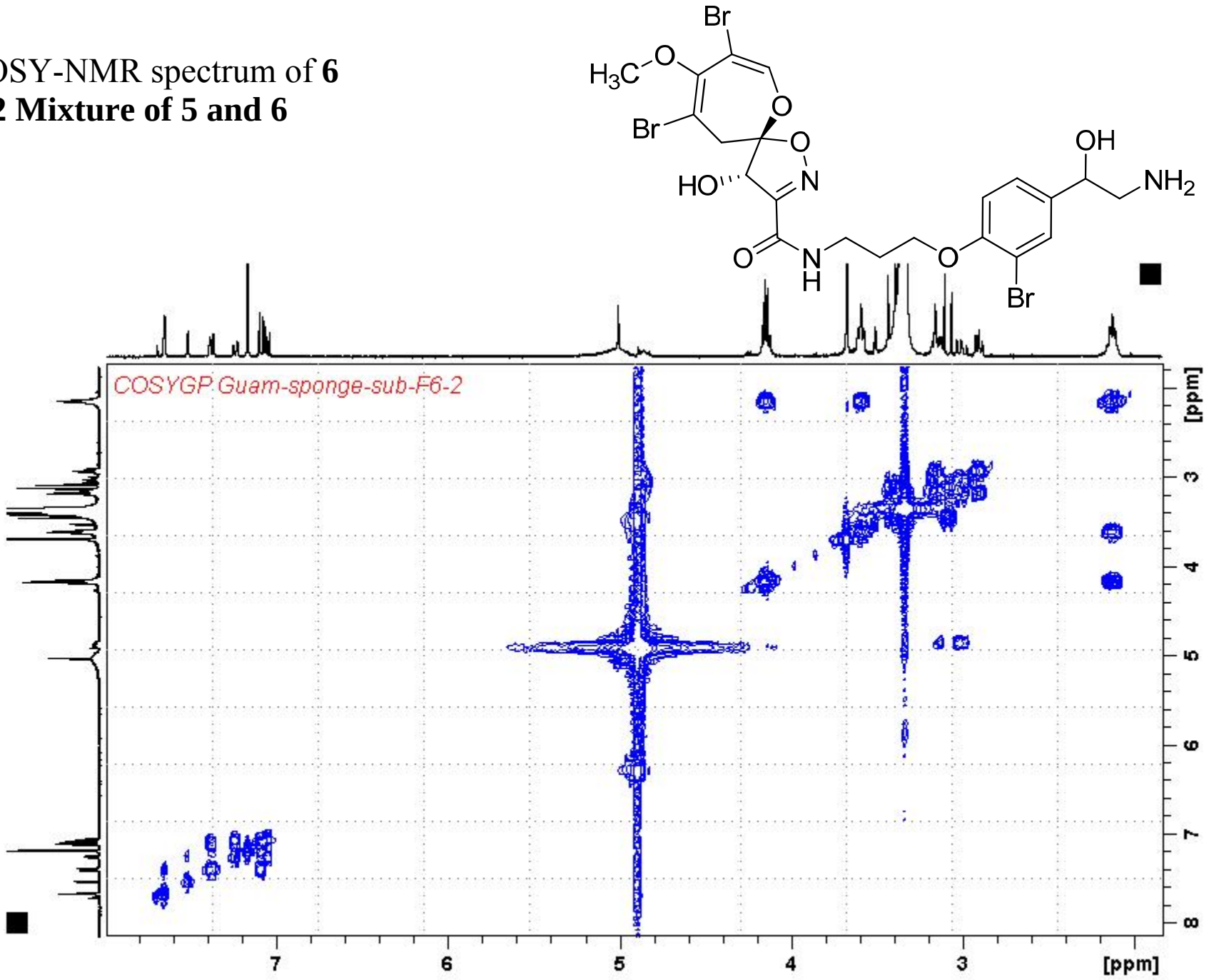
^{1}H-NMR spectra of 3:2 mixture of **5** and **6** (Black), ^{1}H-NMR of **5** (Red), and their difference spectra (Green). Hence the green spectrum is the ^{1}H-NMR spectrum of **6**

All sprectra were recorded with presat of the HOD peak at about δ4.9

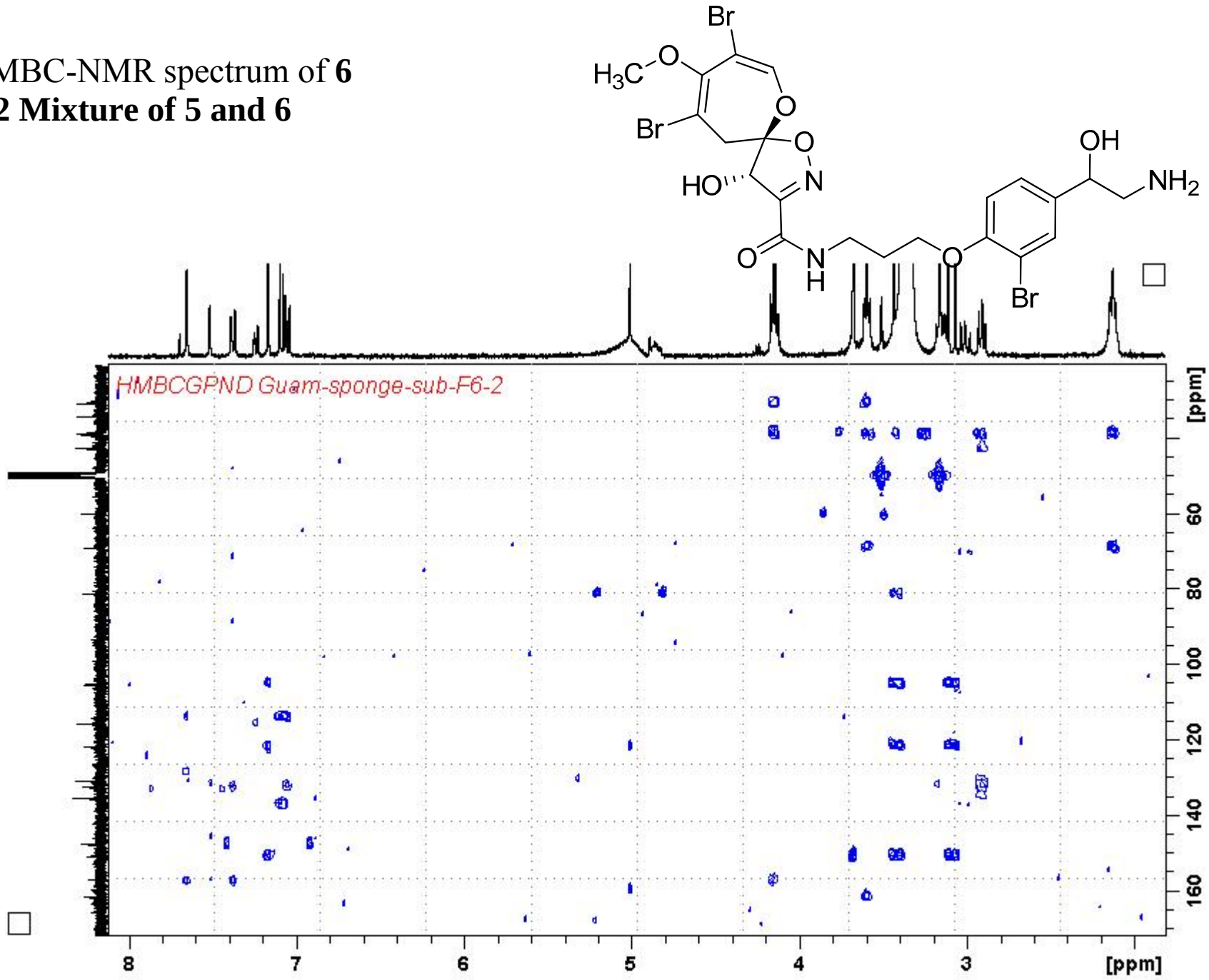




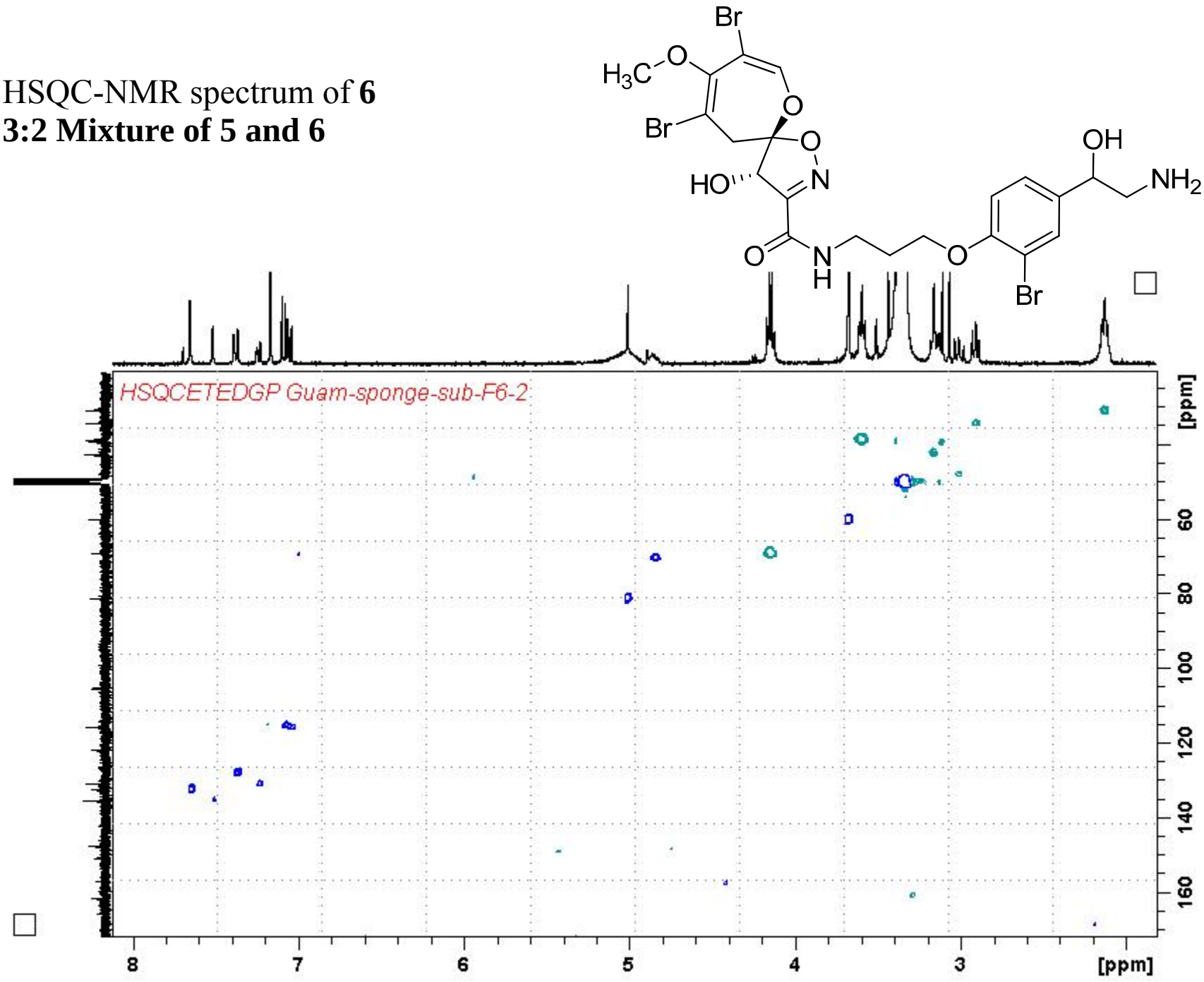
COSY-NMR spectrum of **6**
3:2 Mixture of 5 and 6



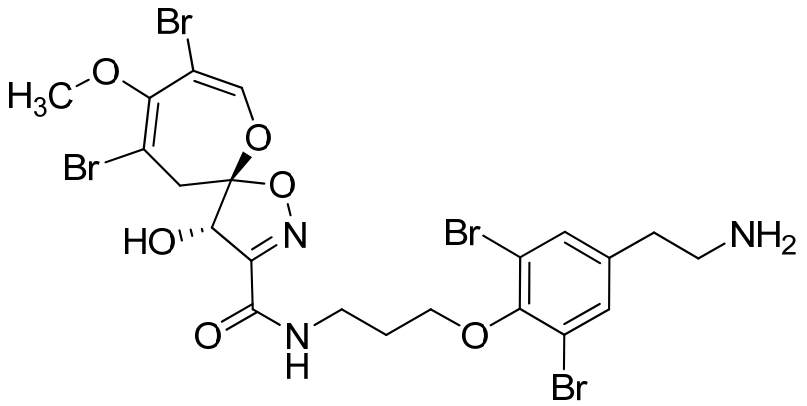
HMBC-NMR spectrum of **6**
3:2 Mixture of 5 and 6

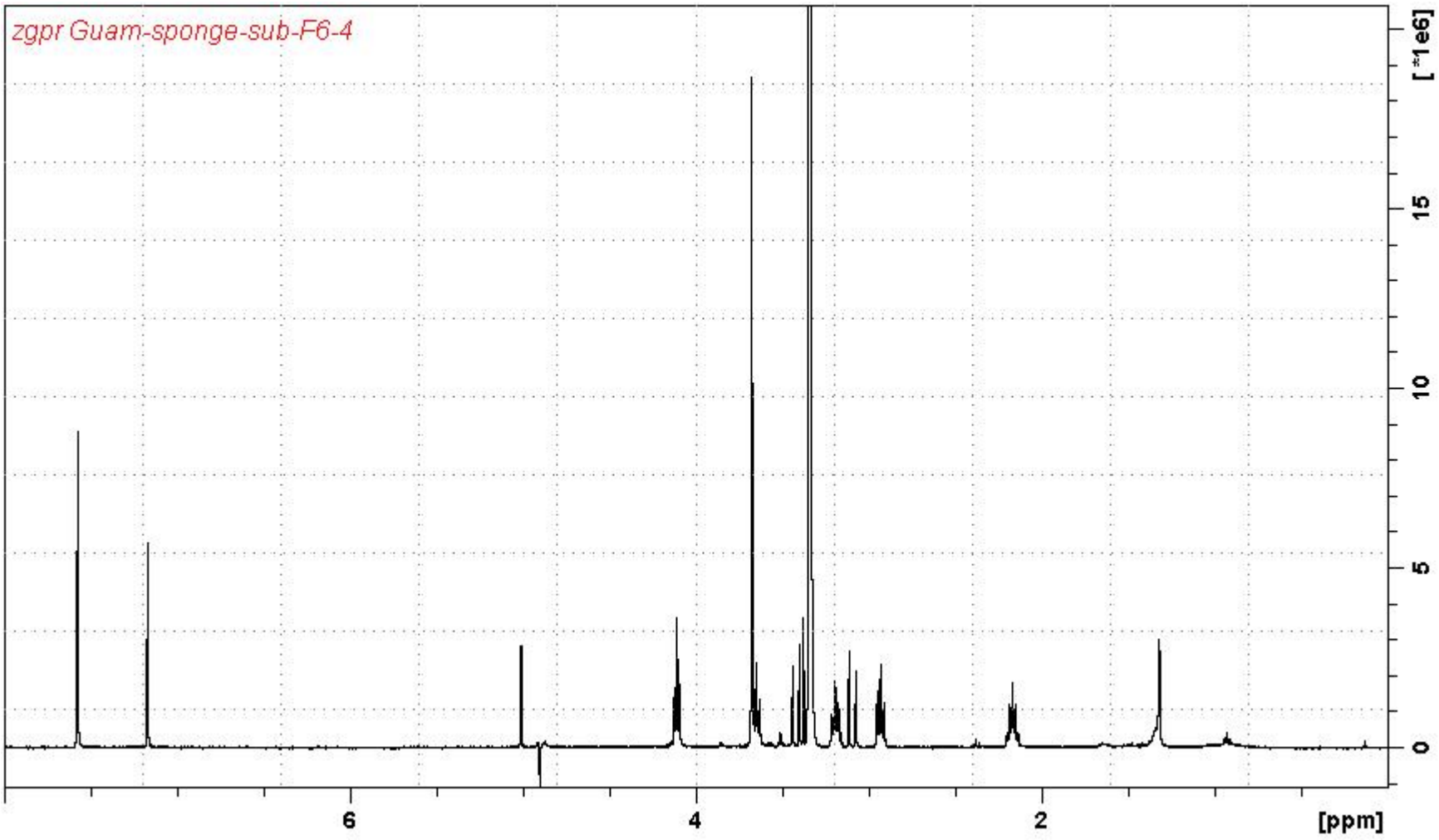


HSQC-NMR spectrum of **6**
3:2 Mixture of 5 and 6

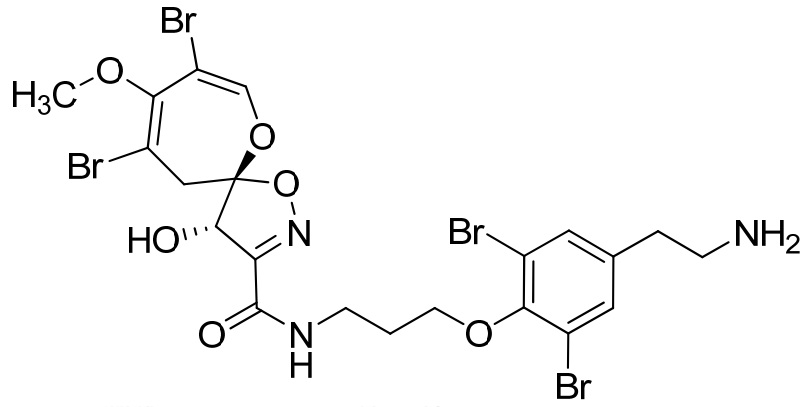


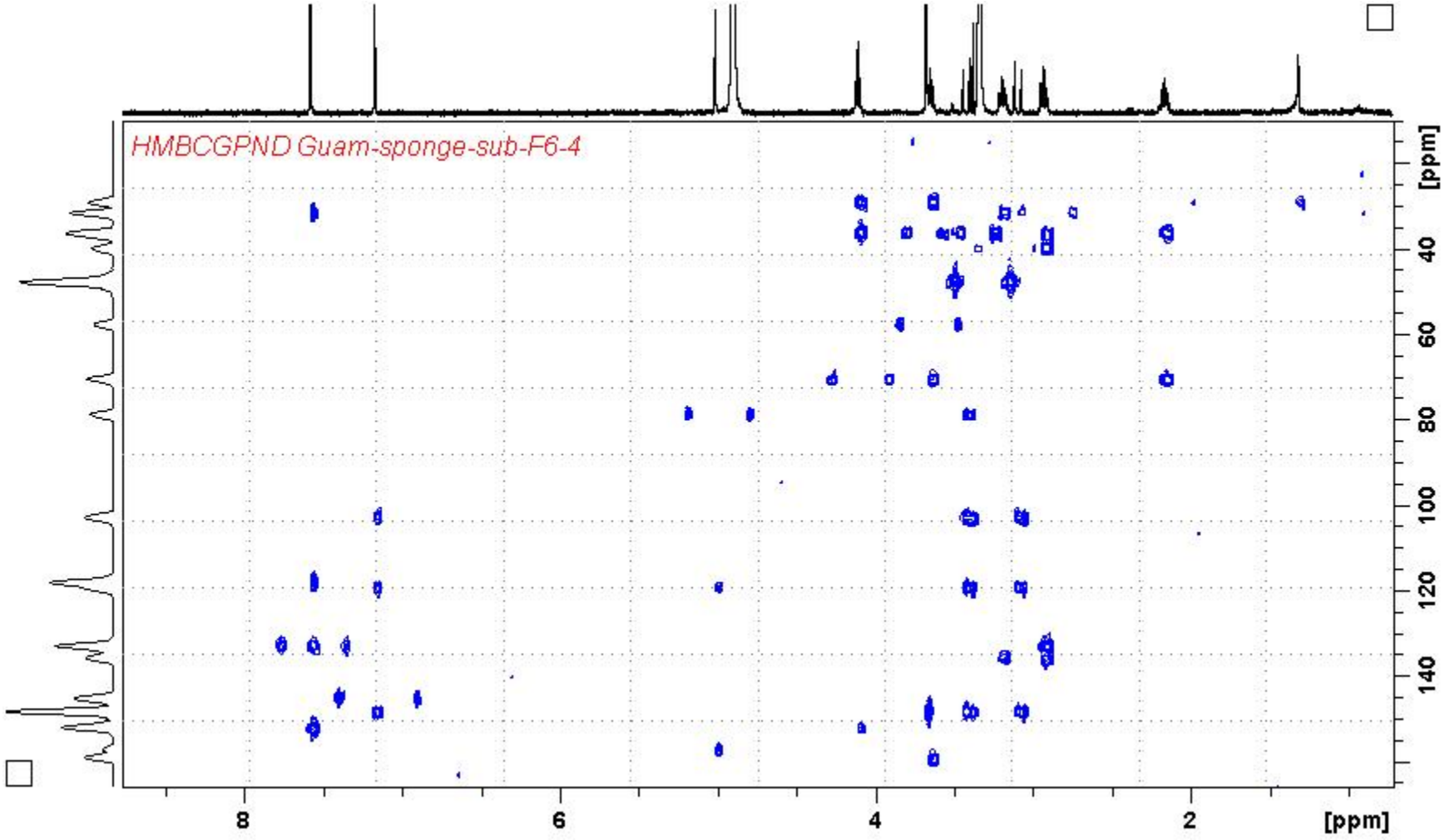
^{1}H-NMR spectrum of **7**



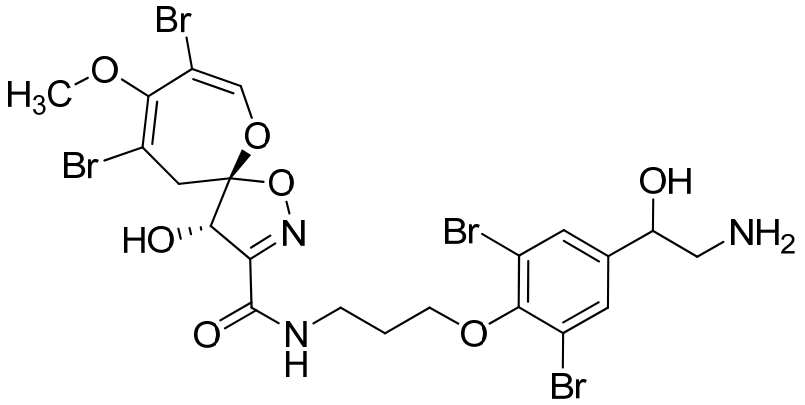


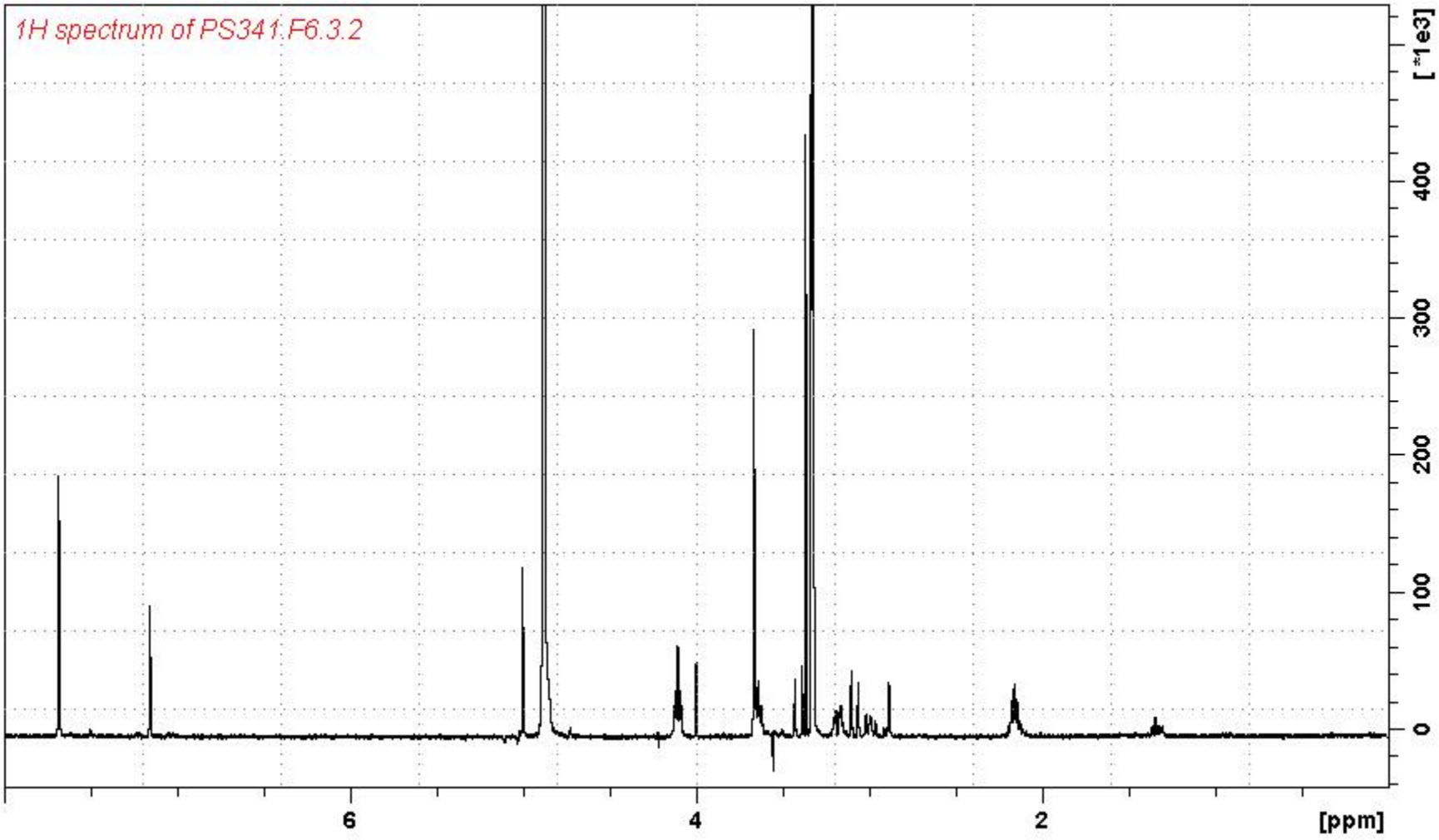
HMBC-NMR spectrum of **7**



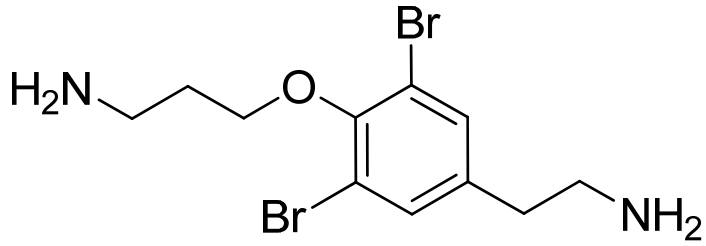


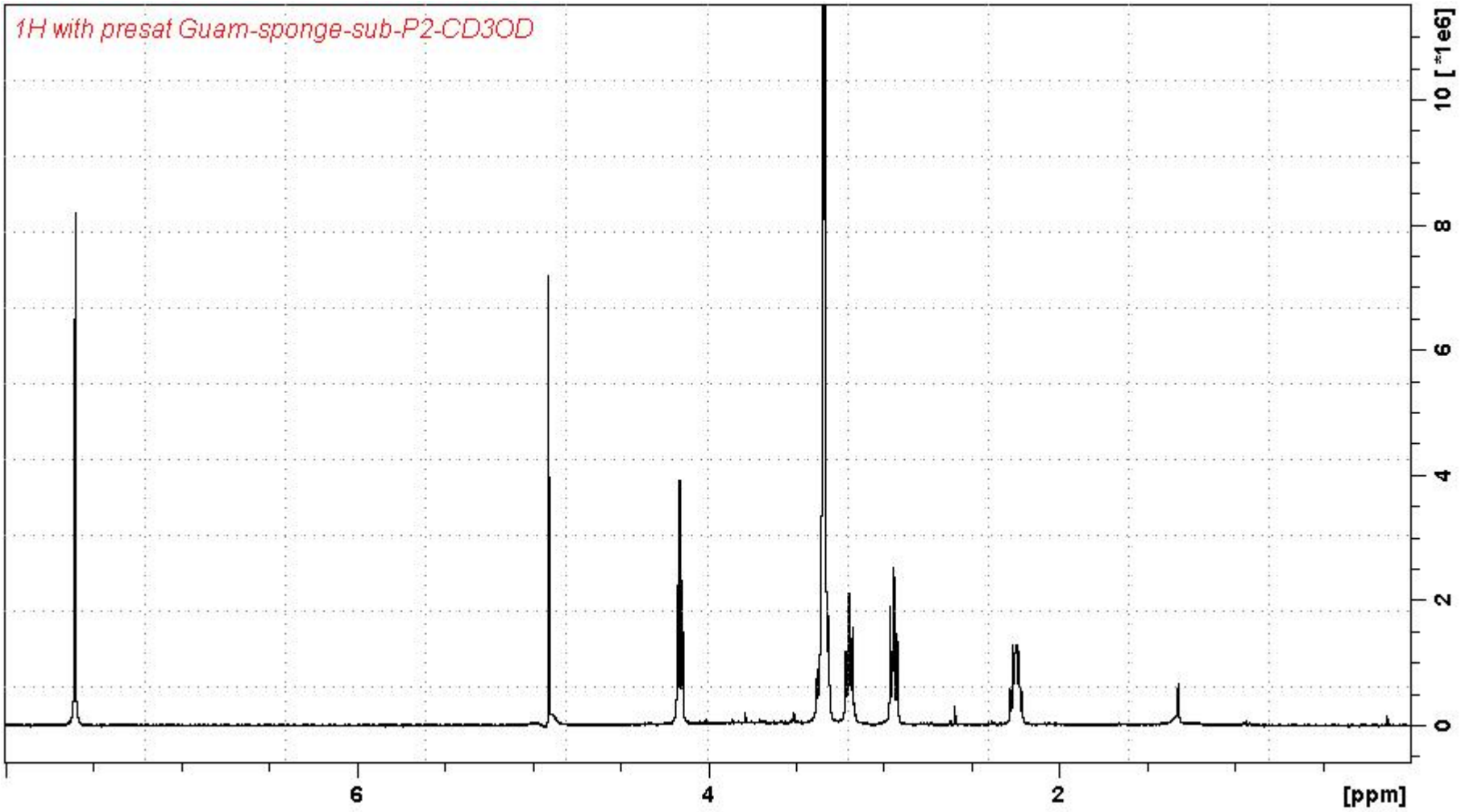
^{1}H-NMR spectrum of **8**





^{1}H-NMR spectrum of **9**





HMBC-NMR spectrum of **9**