

Supporting Information

Acetophenone Monomers from *Acronychia Trifoliolata*

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Figure S1. NCI-60 Human tumor cell line assay data for the crude organic extract of *A. trifoliolata*.

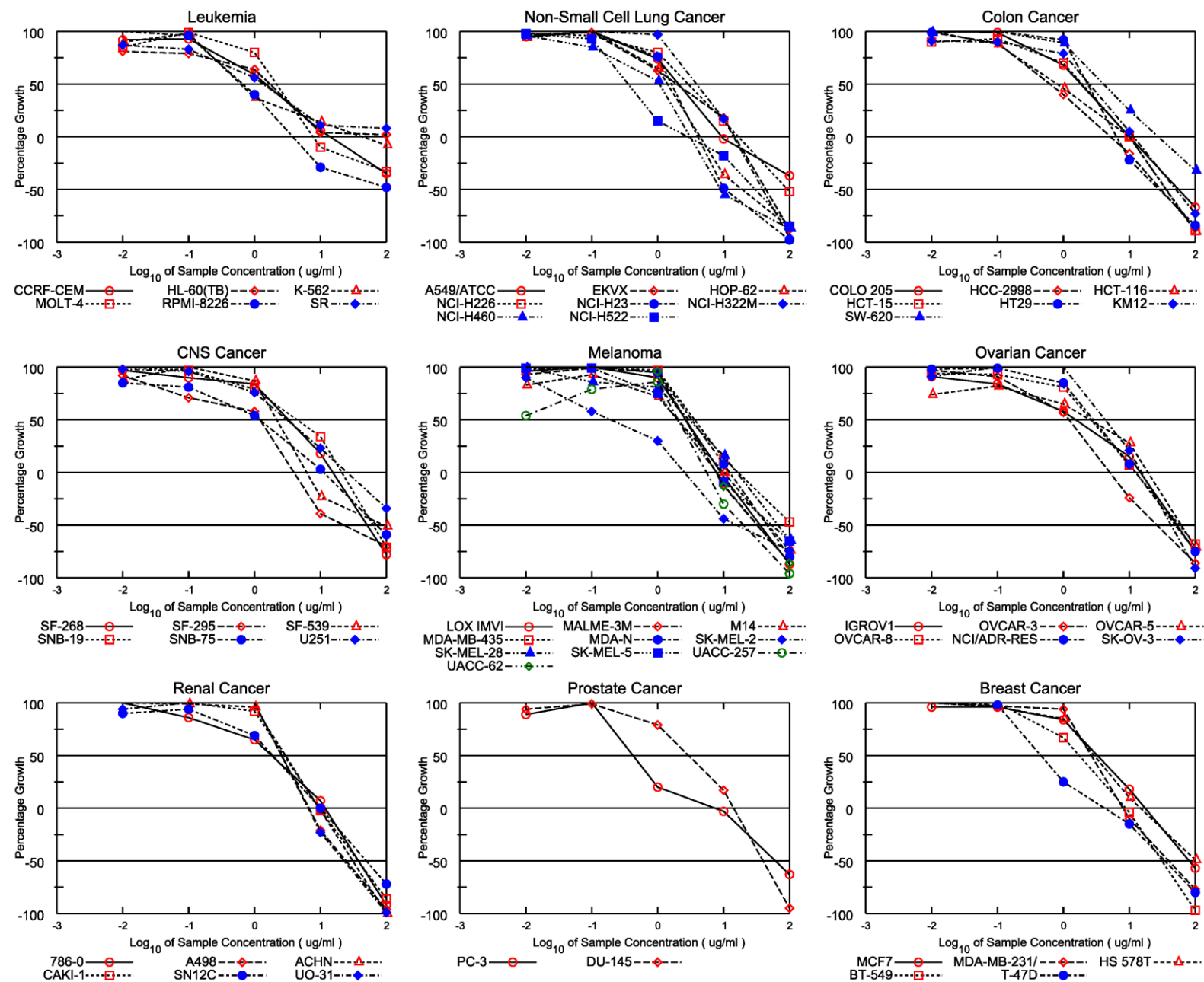


Figure S2. ^1H NMR spectrum of **1** (600 MHz, in CDCl_3).

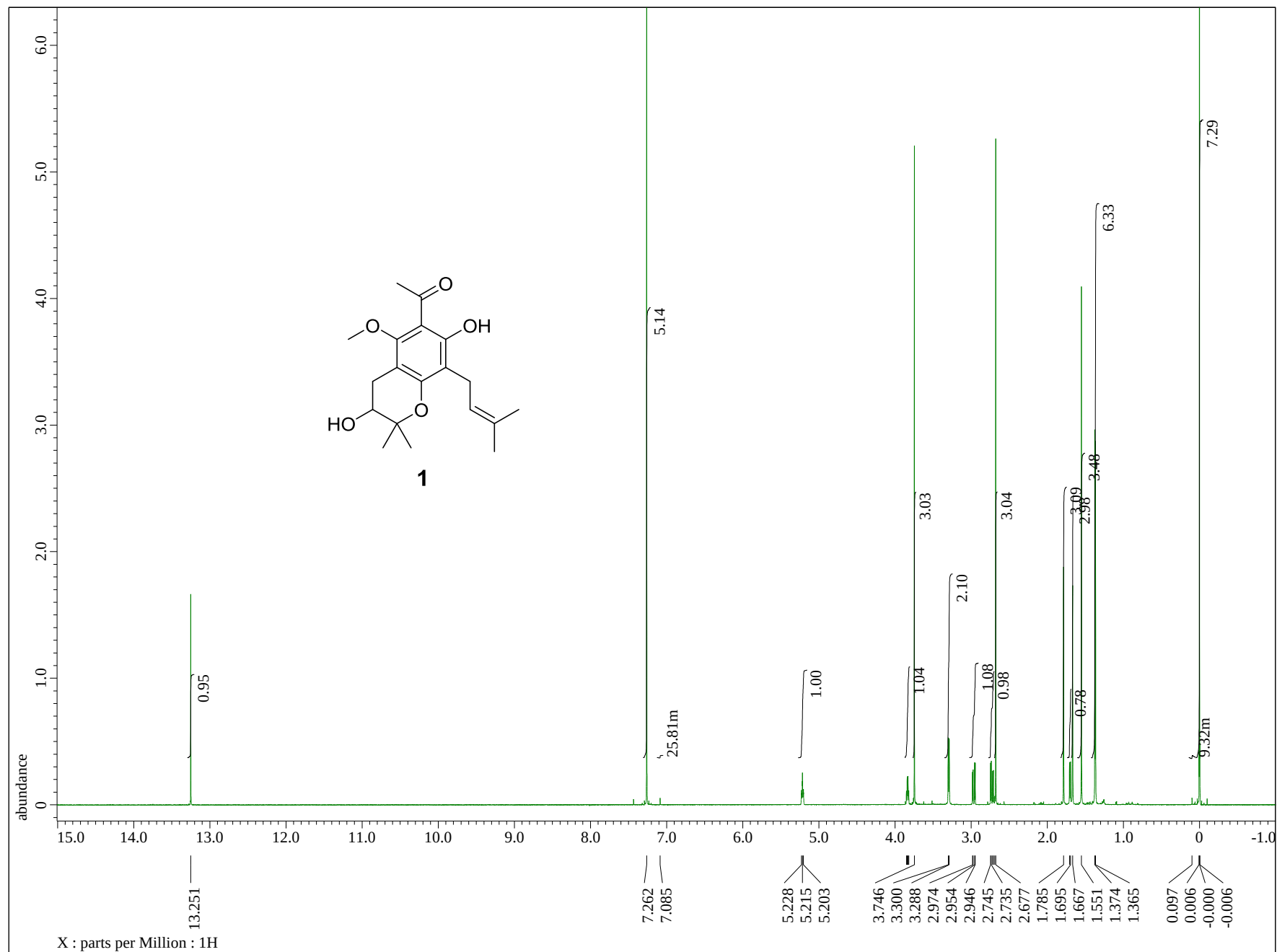


Figure S3. ^{13}C NMR spectrum of **1** (150 MHz, in CDCl_3).

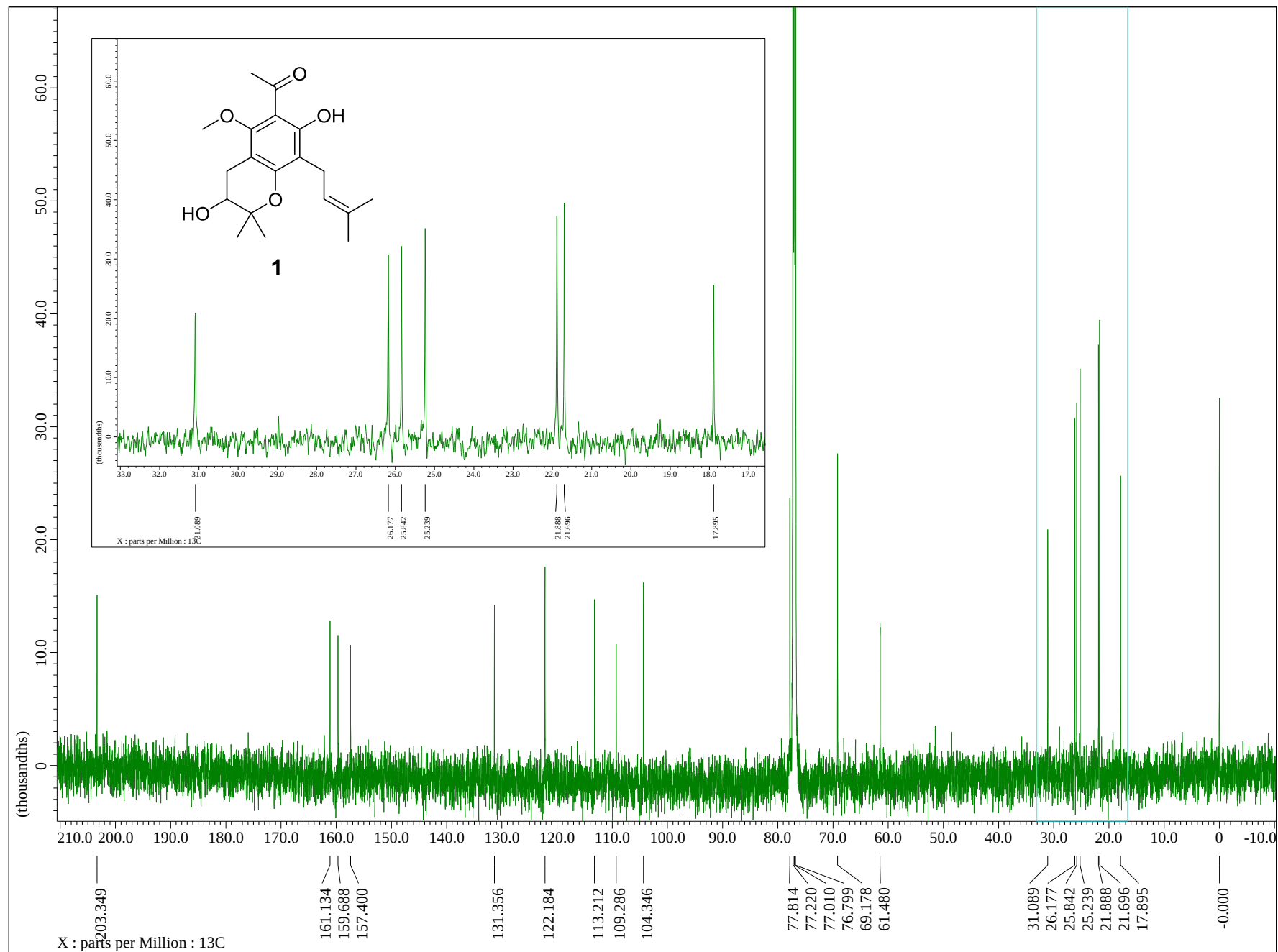


Figure S4. H-H COSY experiment of **1** (in CDCl₃, 400 MHz).

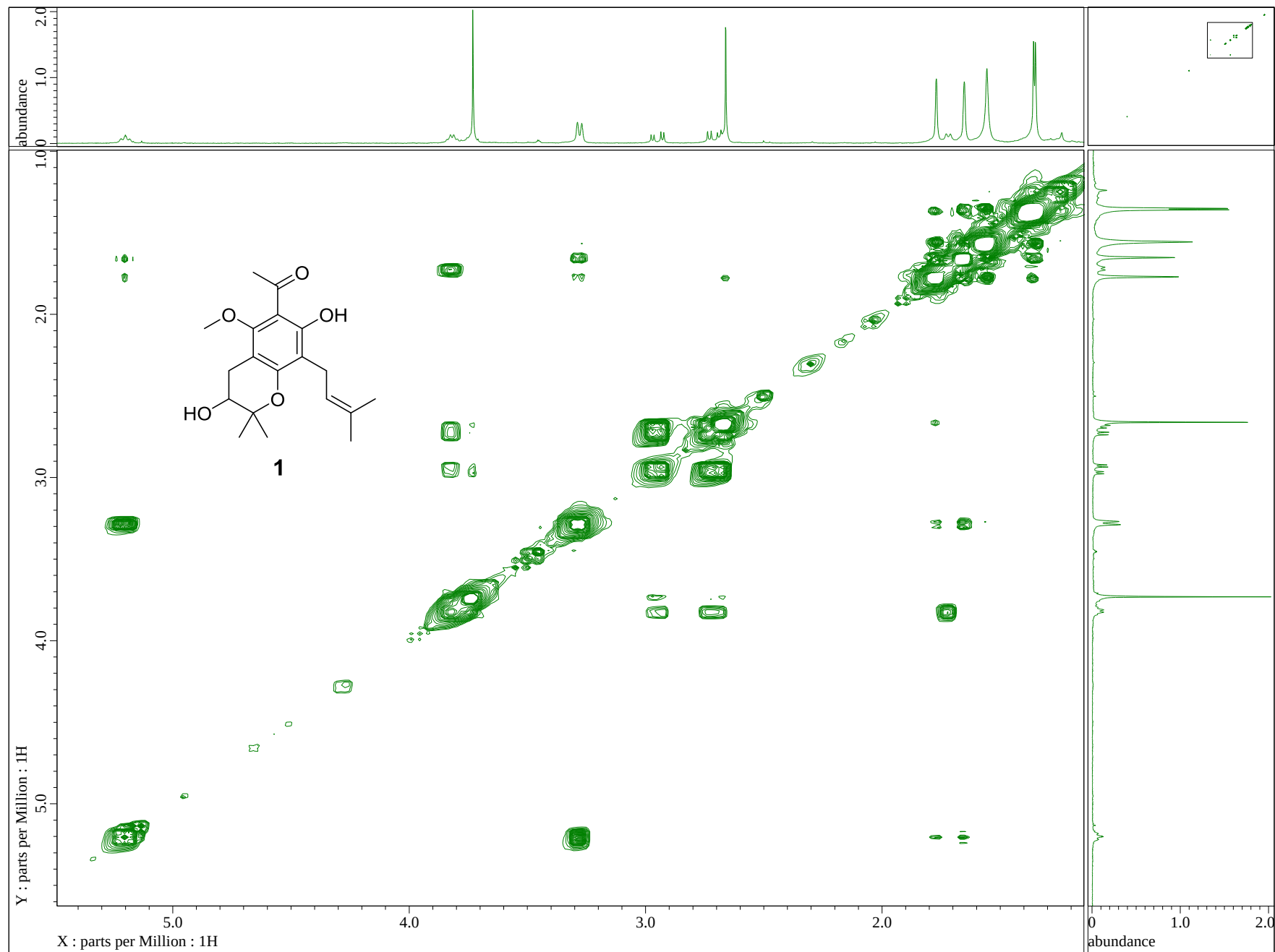


Figure S5. NOESY experiment of **1** (in CDCl₃).

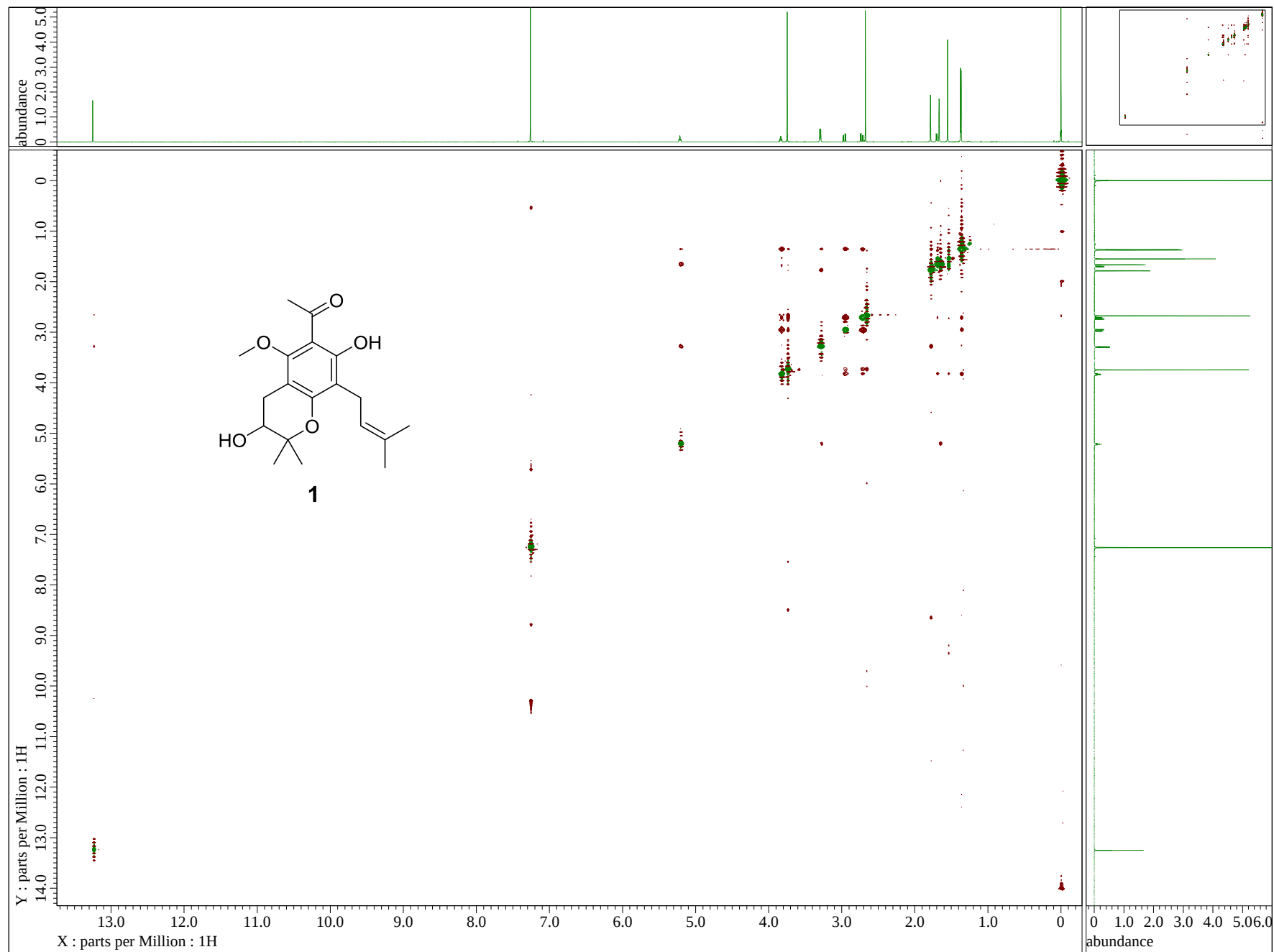


Figure S6. NOESY experiment of **1** (in CDCl₃).

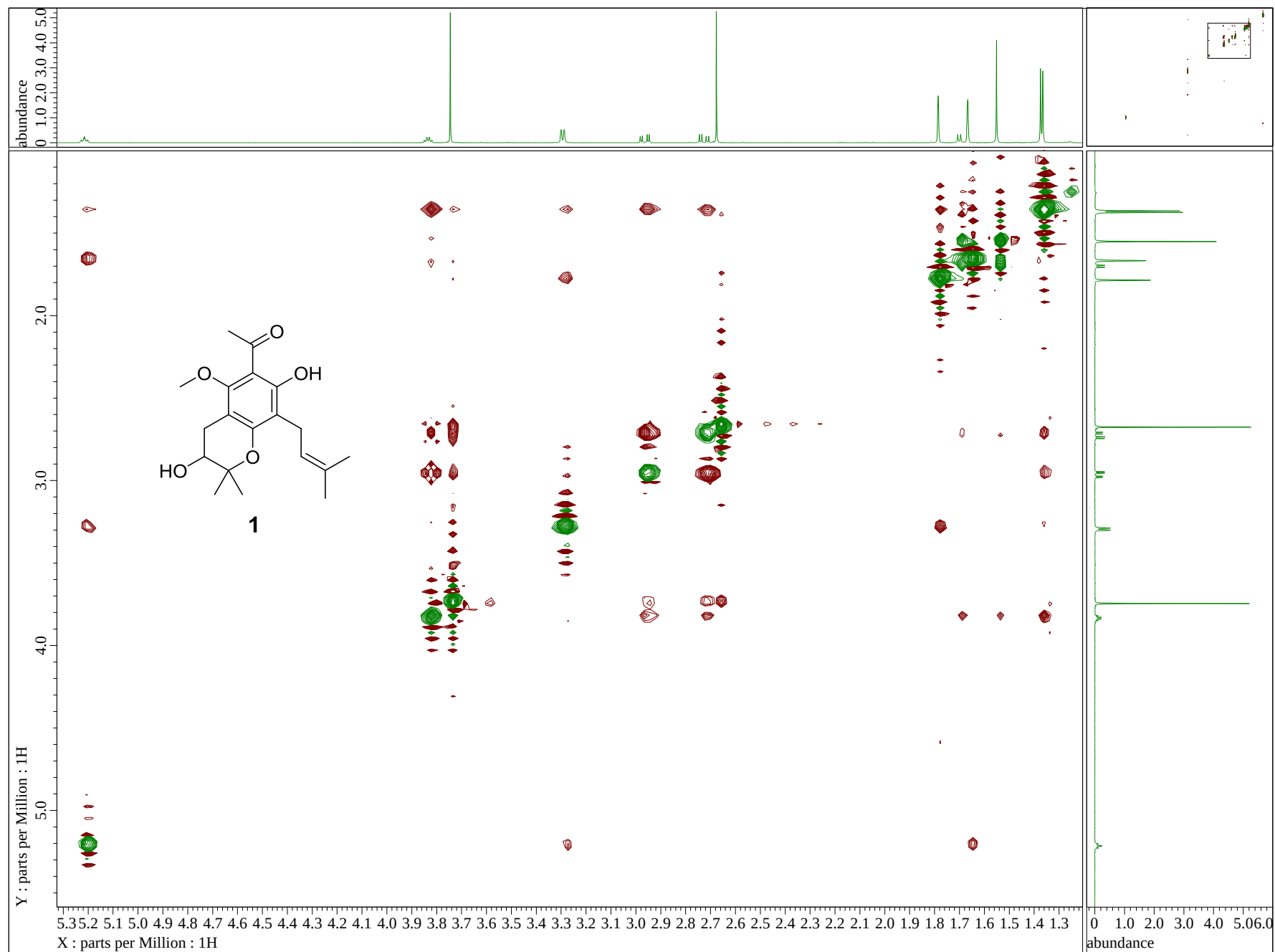


Figure S7. HMQC experiment of 1 (in CDCl₃).

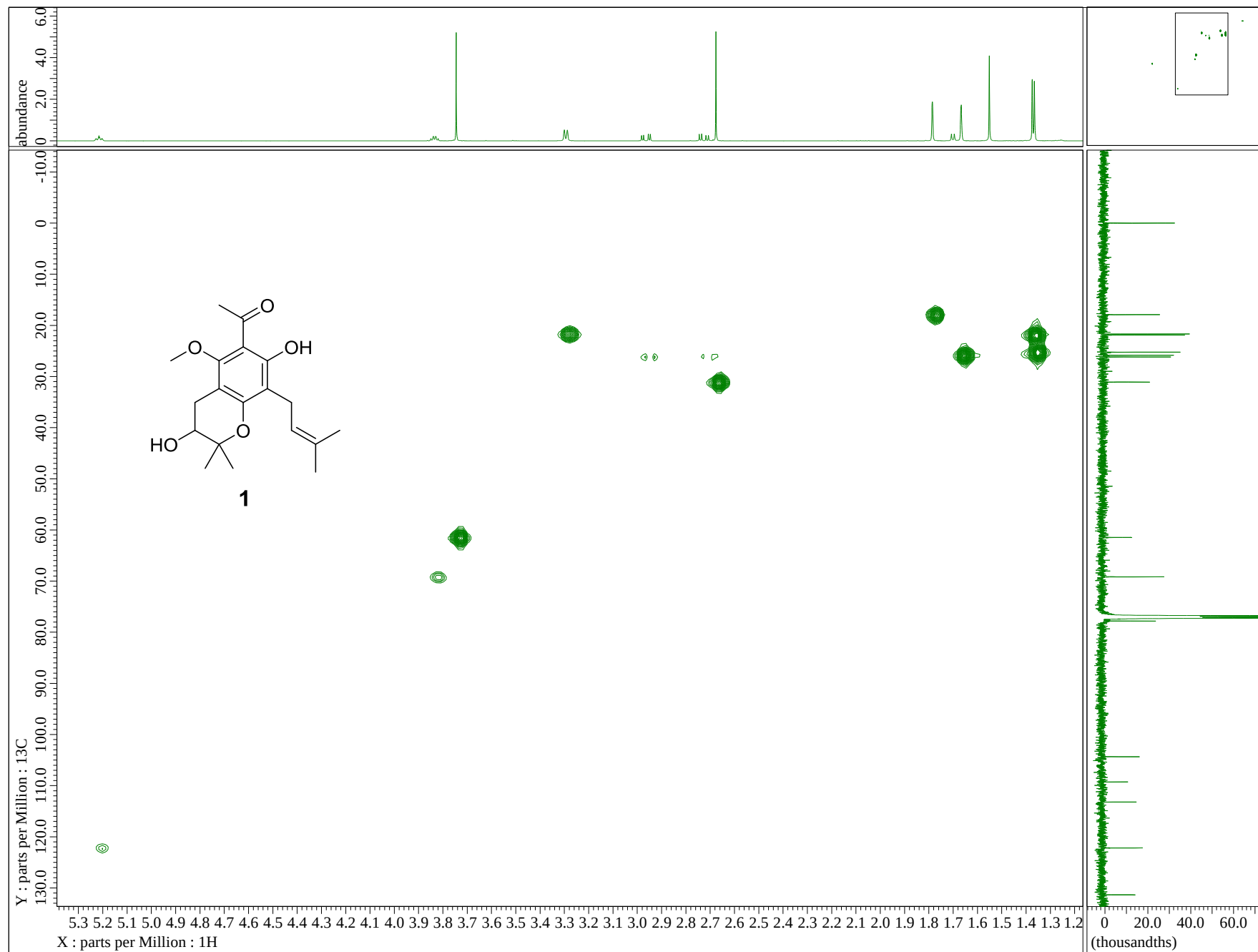


Figure S8. HMBC experiment of 1 (in CDCl₃).

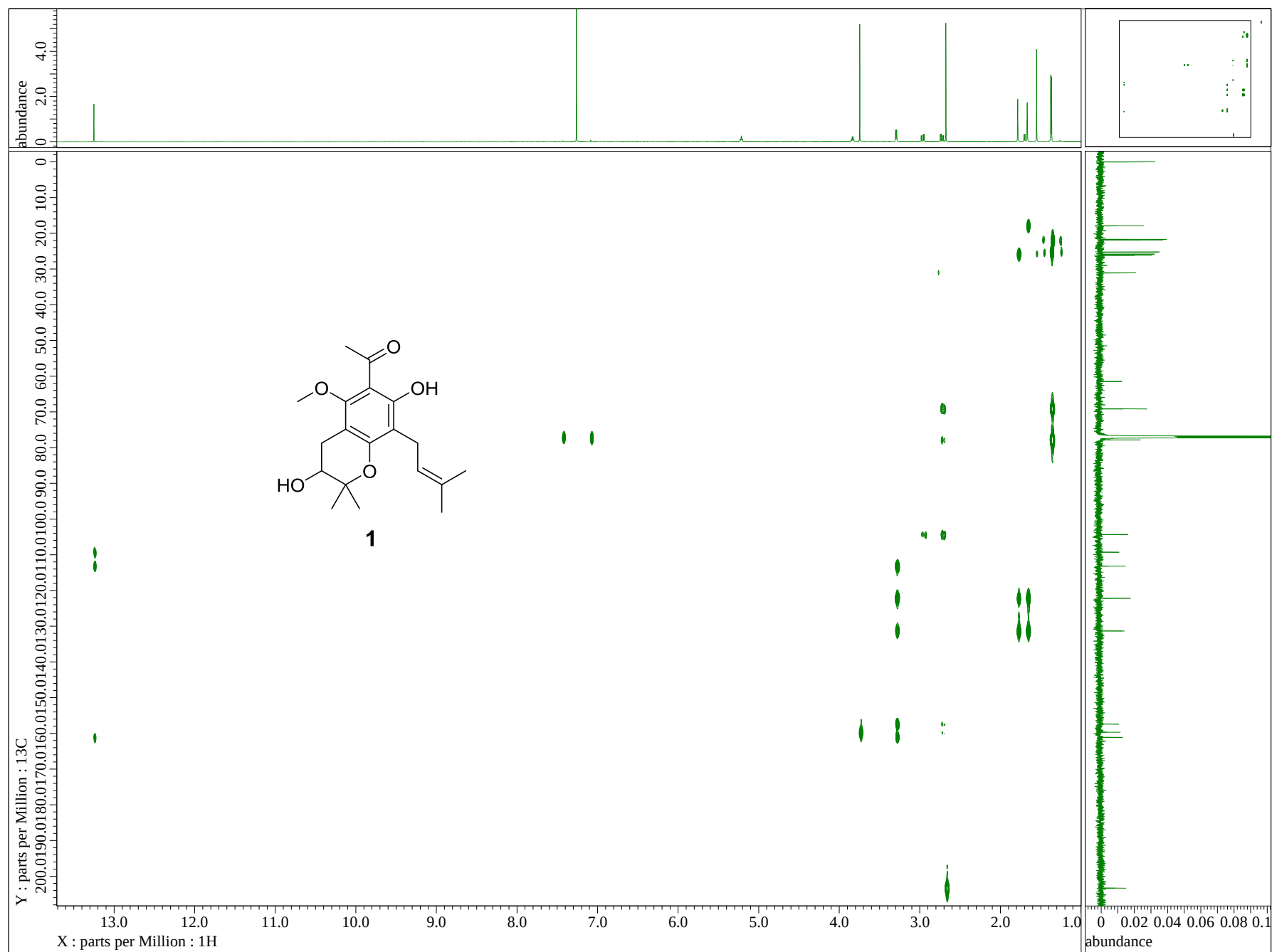


Figure S9. HMBC experiment of 1 (in CDCl₃).

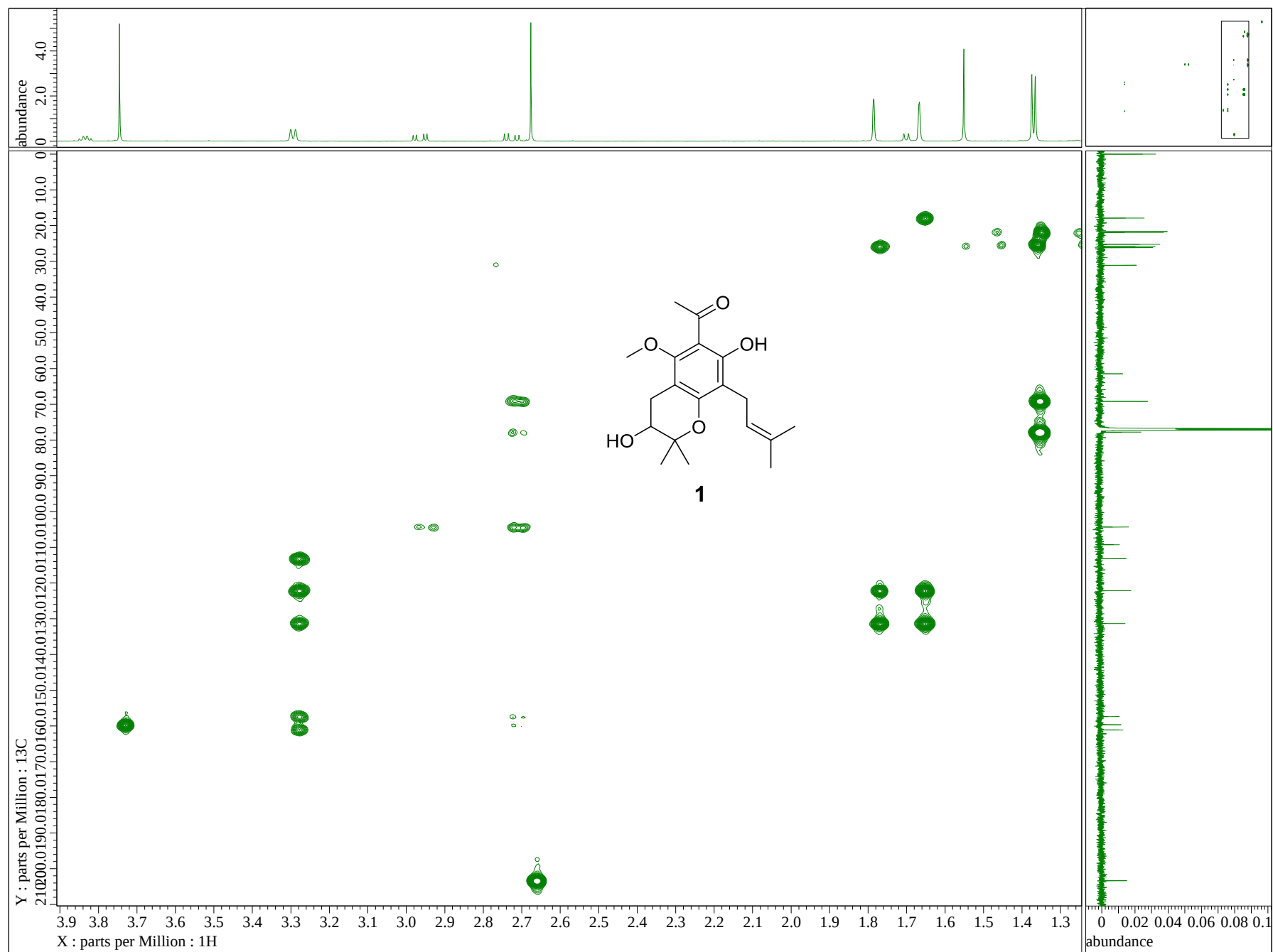


Figure S10. ^1H NMR spectrum of **2** (600 MHz, in CDCl_3).

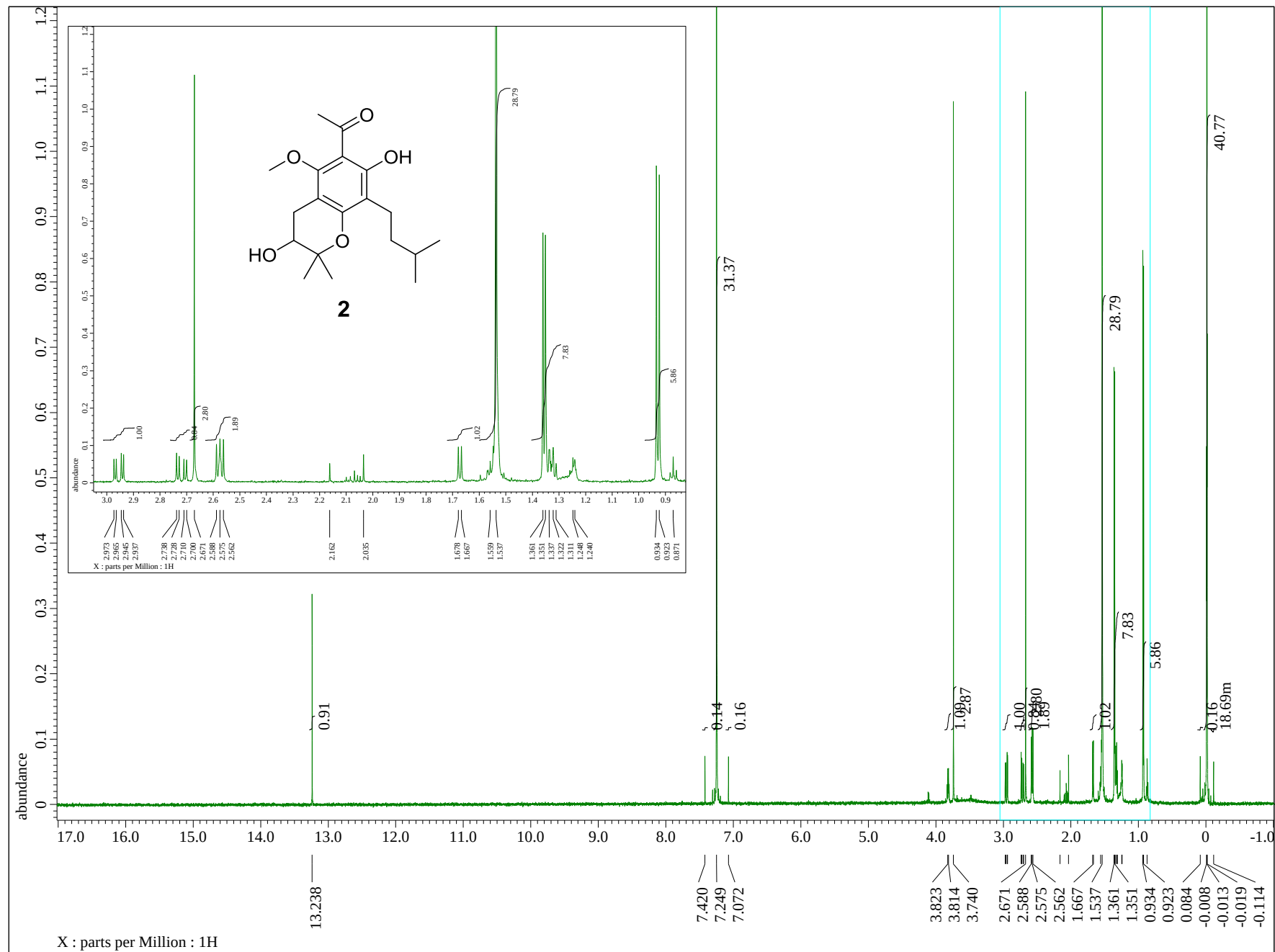


Figure S11. H-H COSY experiment of **2** (in CDCl₃).

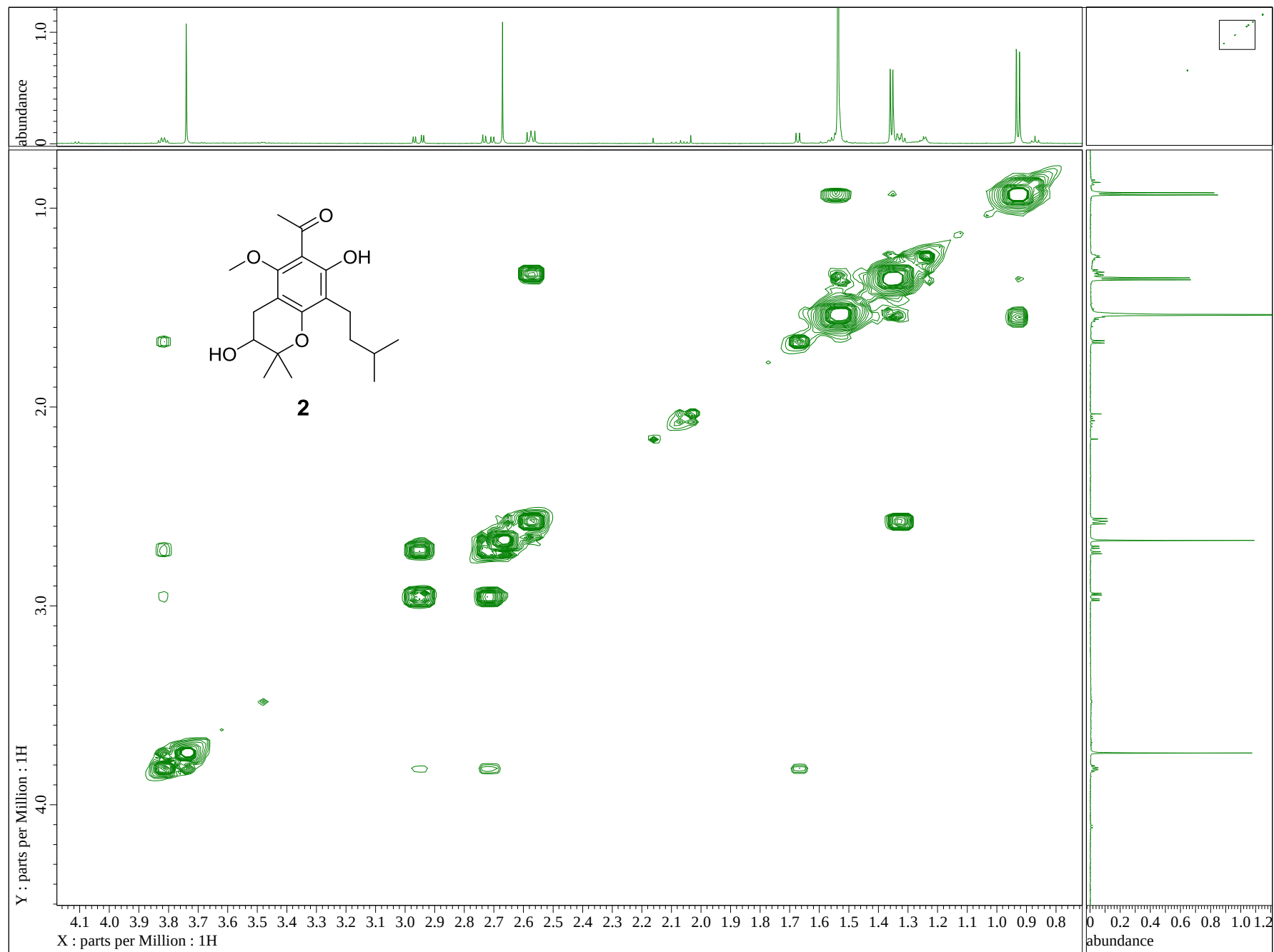


Figure S12. NOESY experiment of **2** (in CDCl₃).

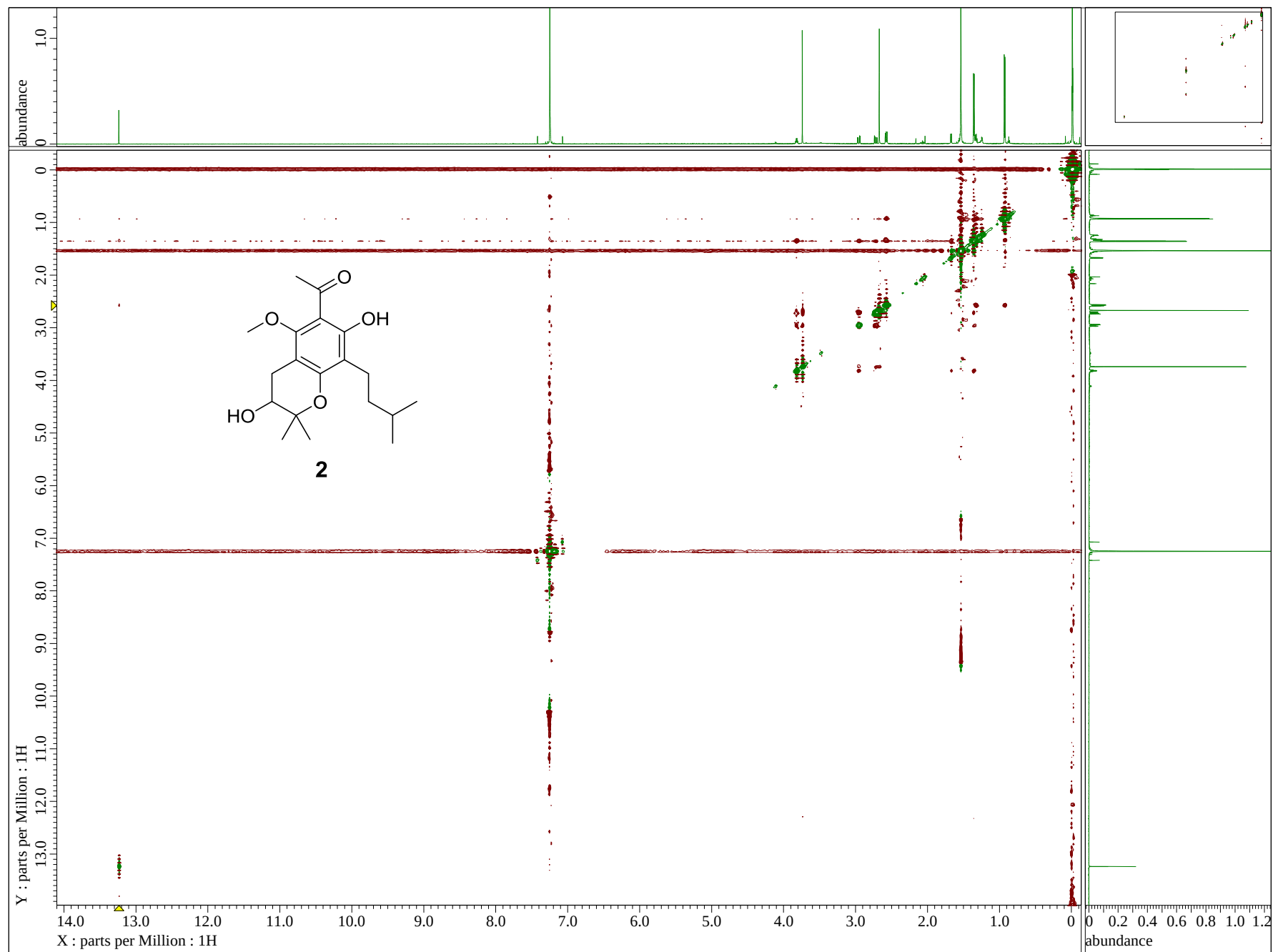


Figure S13. NOESY experiment of **2** (in CDCl₃).

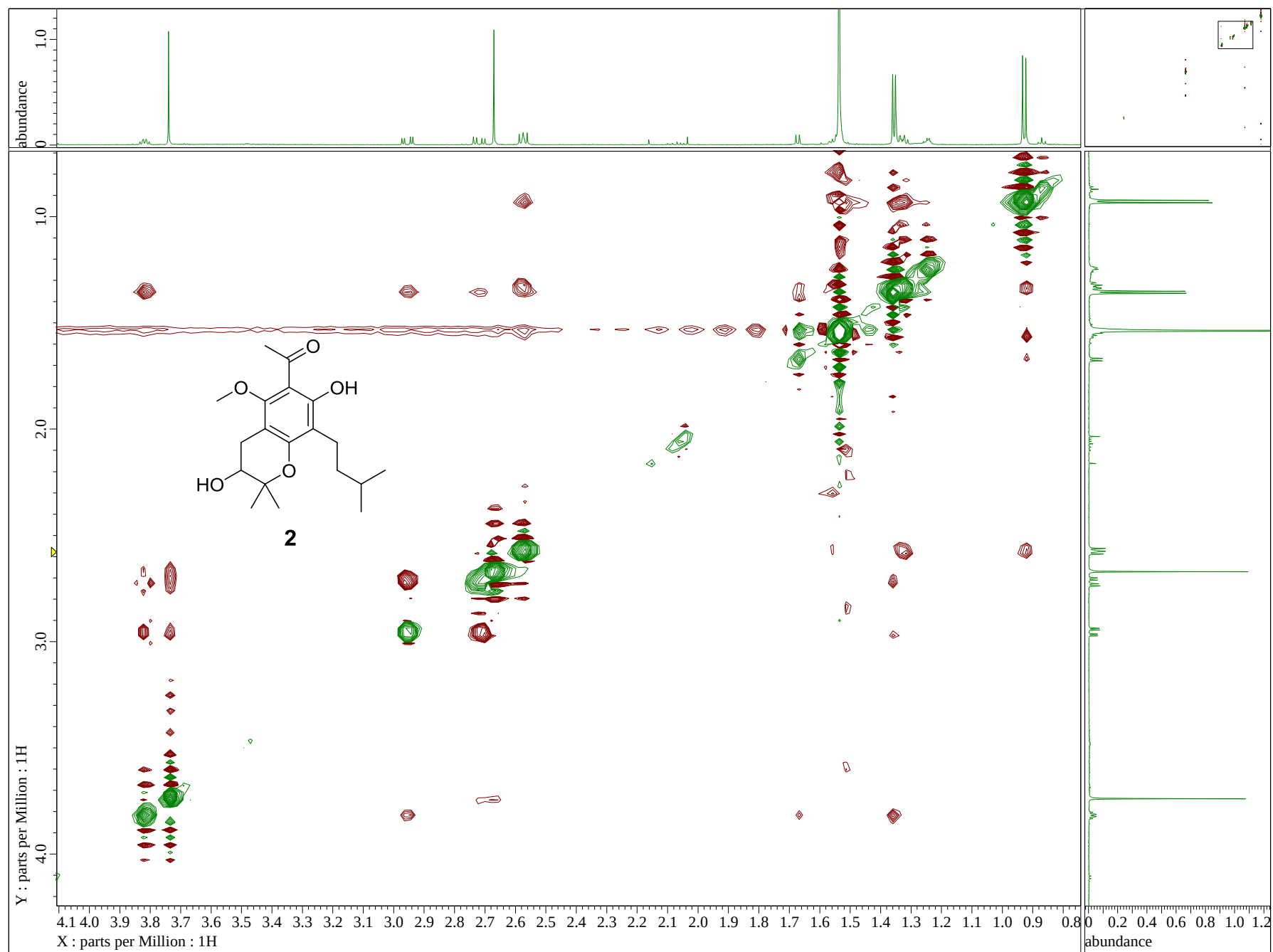


Figure S14. ^1H NMR spectrum of **3** (600 MHz, in CDCl_3).

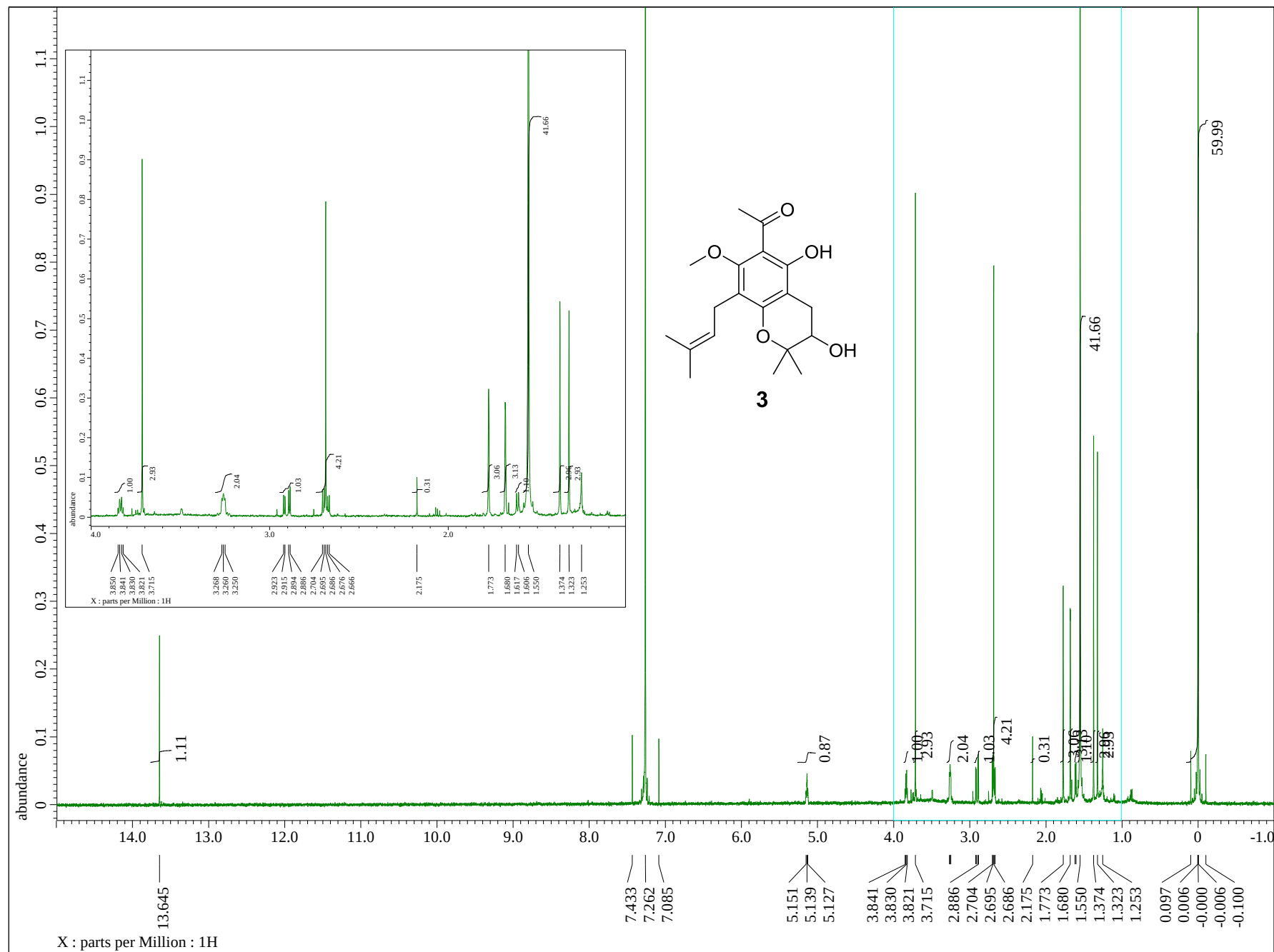


Figure S15. H-H COSY experiment of **3** (in CDCl₃).

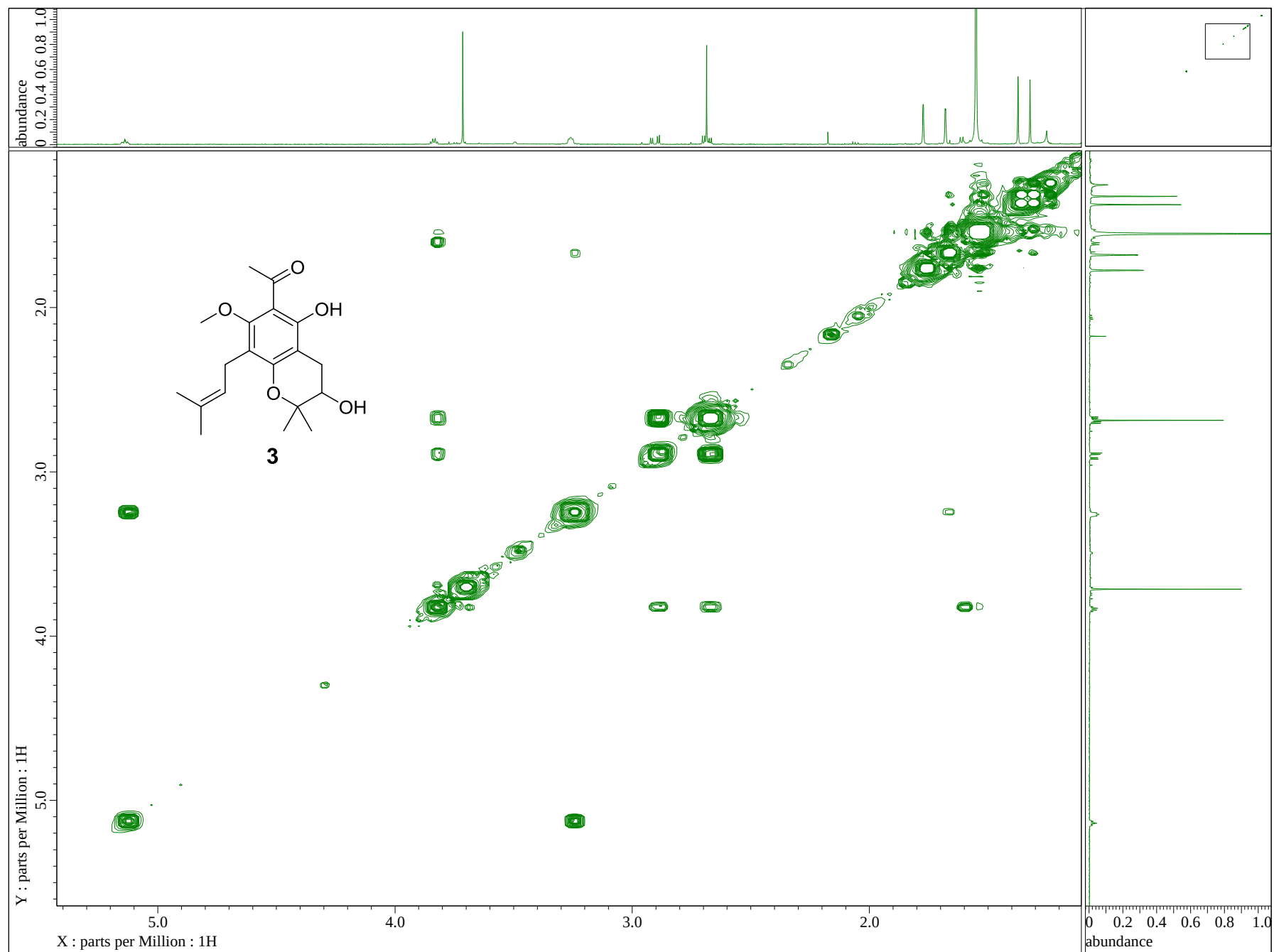
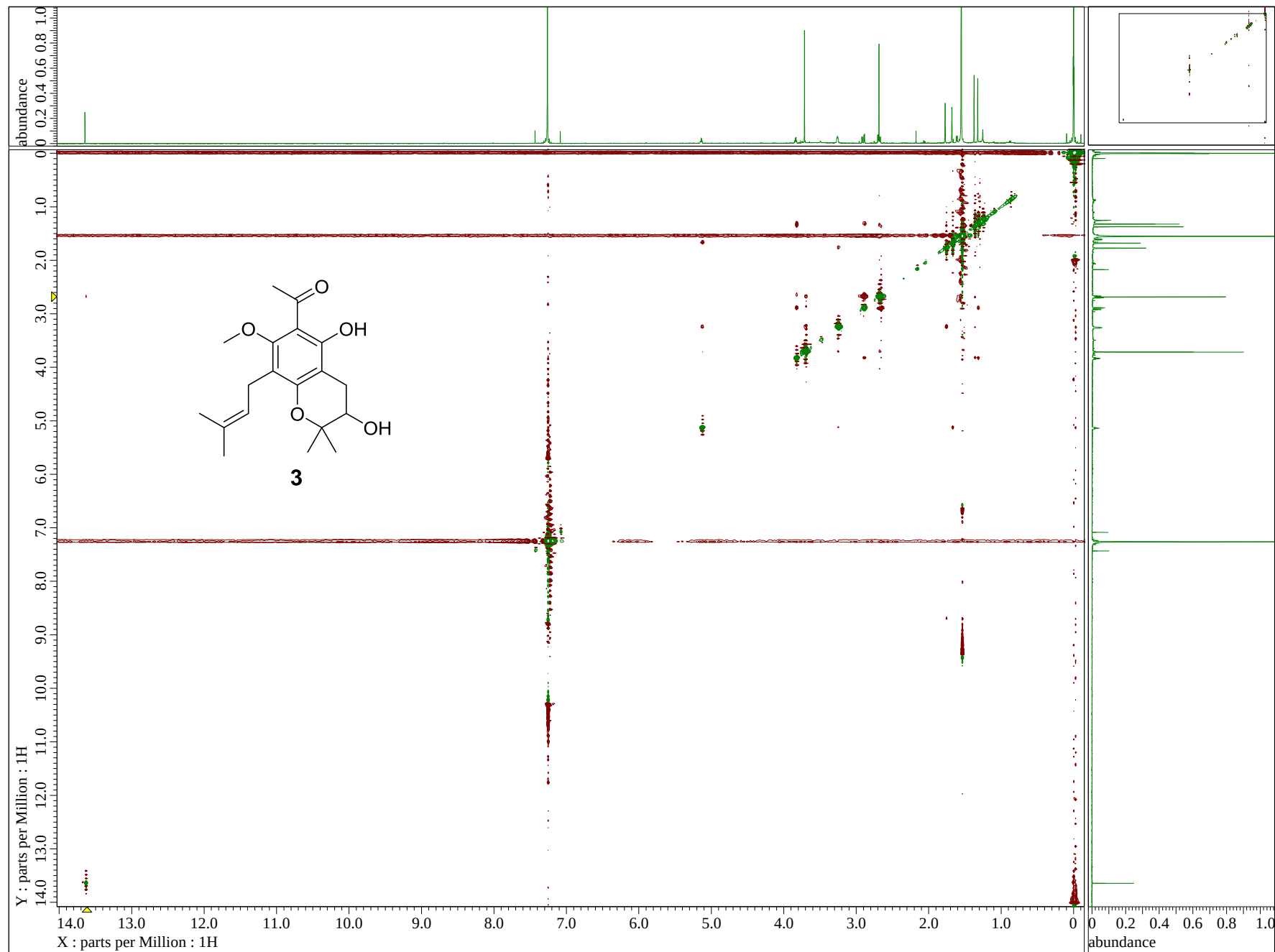


Figure S16. NOESY experiment of **3** (in CDCl₃).



Chemical structure of compound **3** is shown in the top left. The 2D COSY NMR spectrum displays correlations between protons. The x-axis is labeled "X : parts per Million : ^1H " and the y-axis is labeled "Y : parts per Million : ^1H ". The spectrum shows correlations between protons, with 1D ^1H NMR spectra projected on the top and right. A small inset in the top right shows the ^{13}C NMR spectrum.

Figure S18. ^1H NMR spectrum of **4** (600 MHz, in CDCl_3).

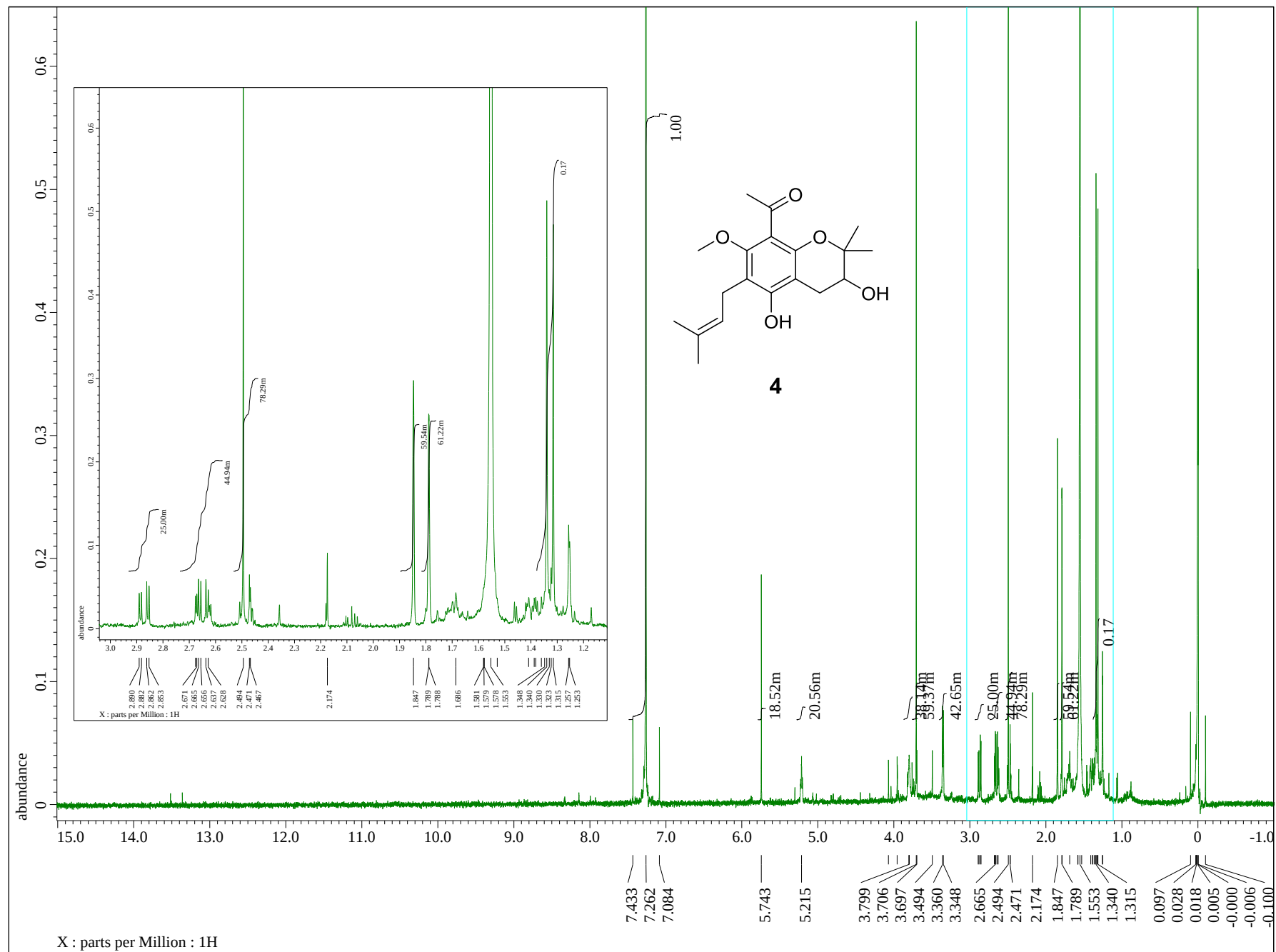


Figure S19. H-H COSY experiment of **4** (in CDCl₃).

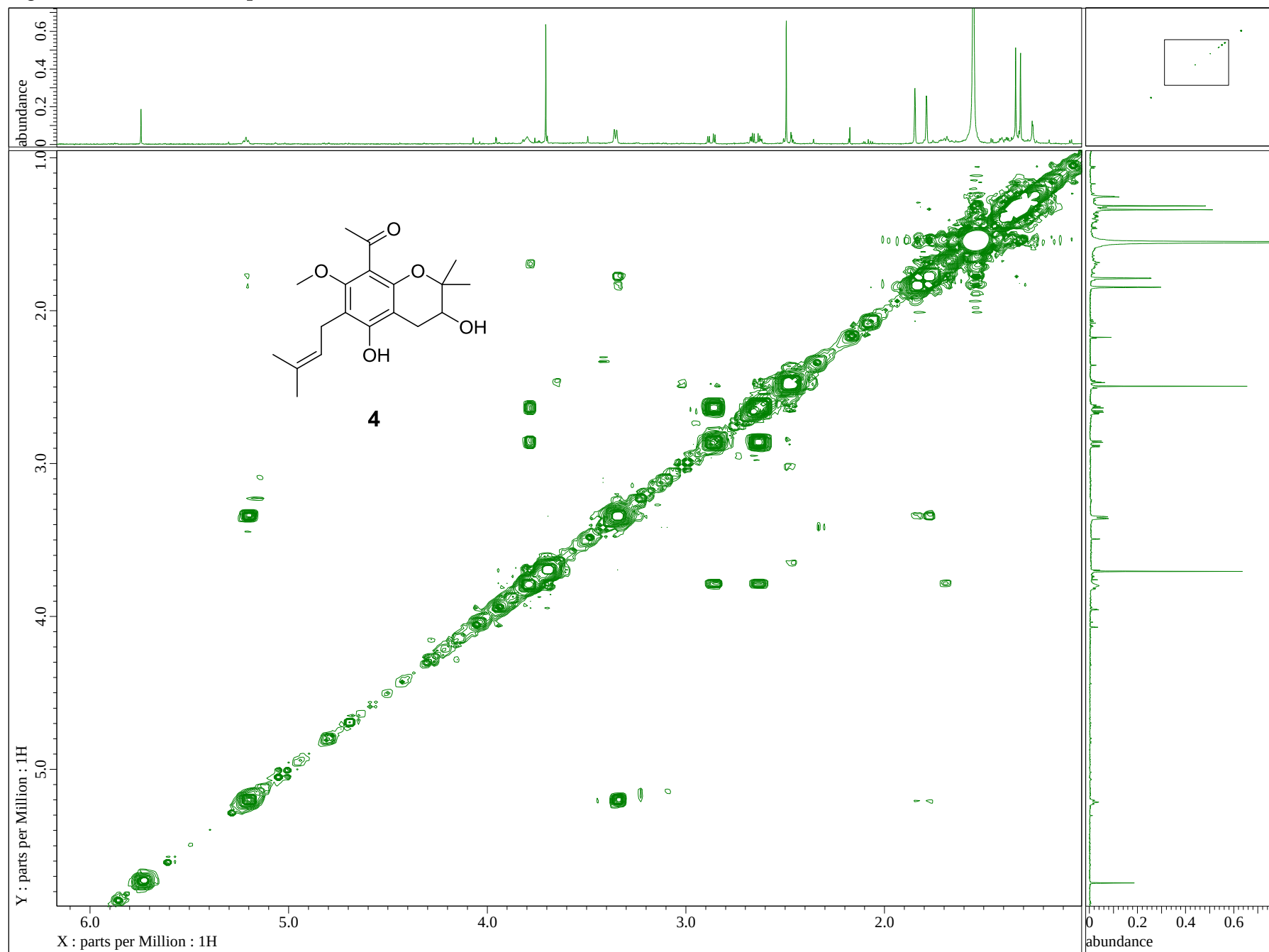


Figure S20. NOESY experiment of **4** (in CDCl₃).

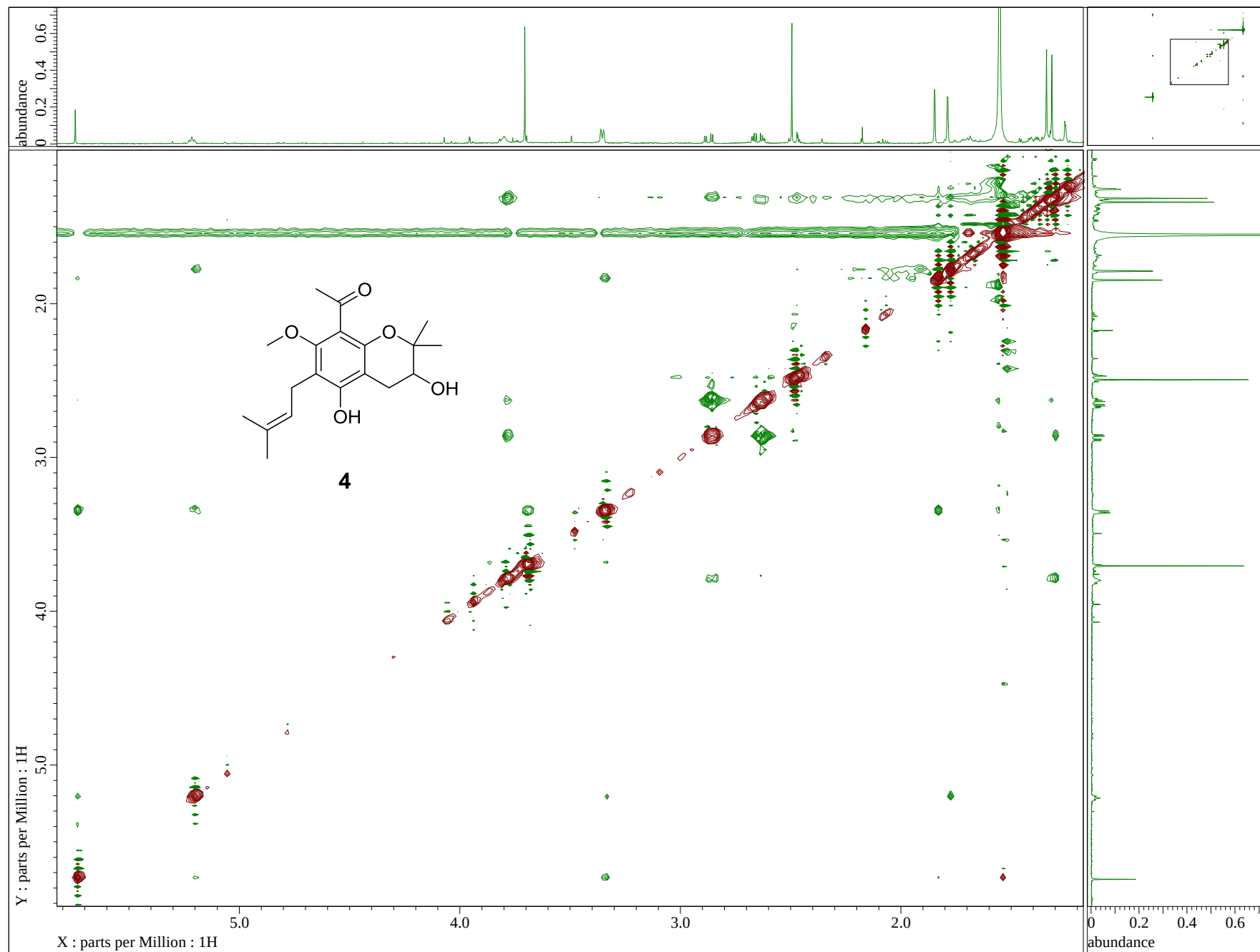


Figure S21. ^1H NMR spectrum of **5** (600 MHz, in CDCl_3).

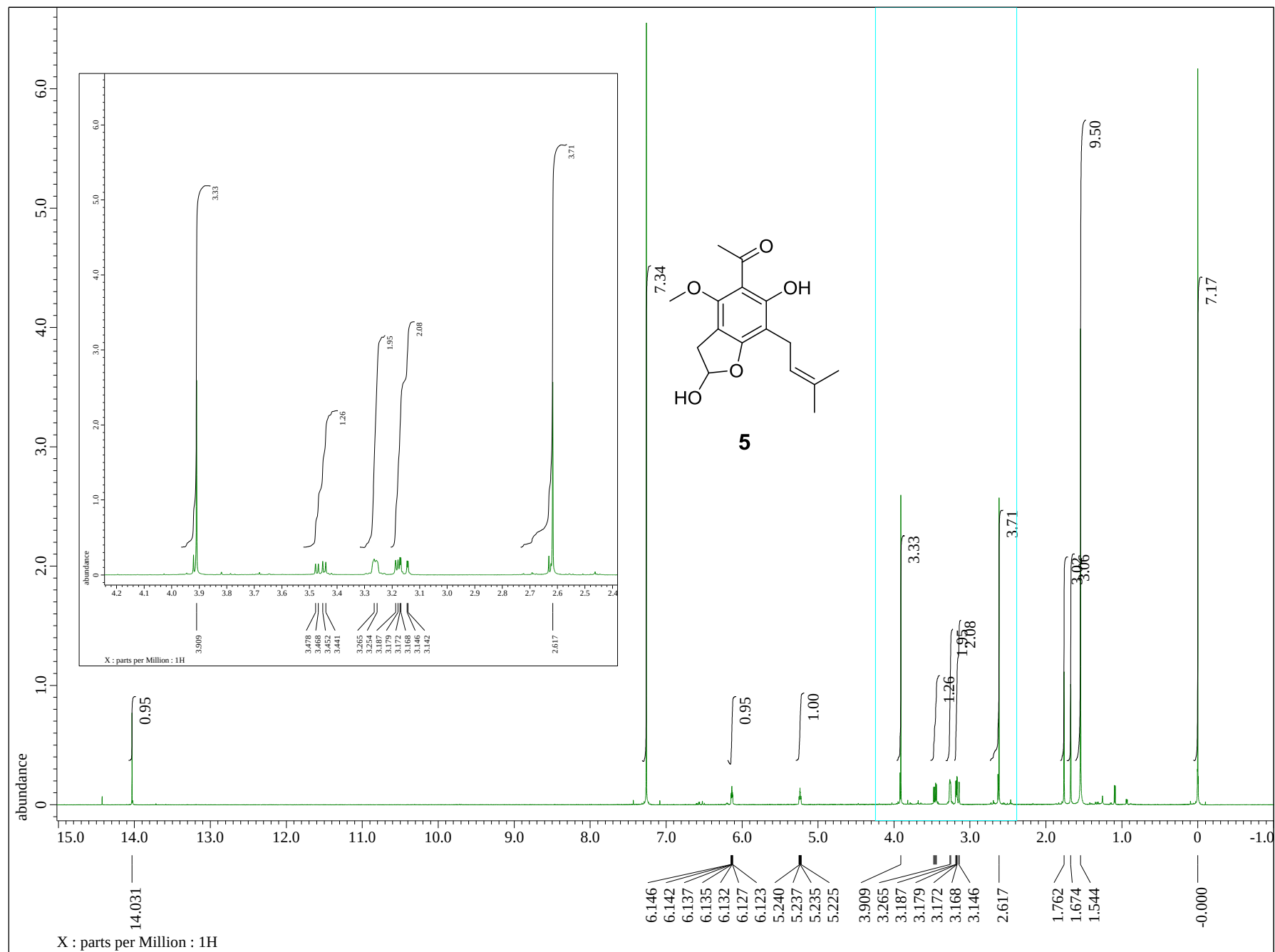


Figure S22. ^{13}C NMR spectrum of **5** (150 MHz, in CDCl_3).

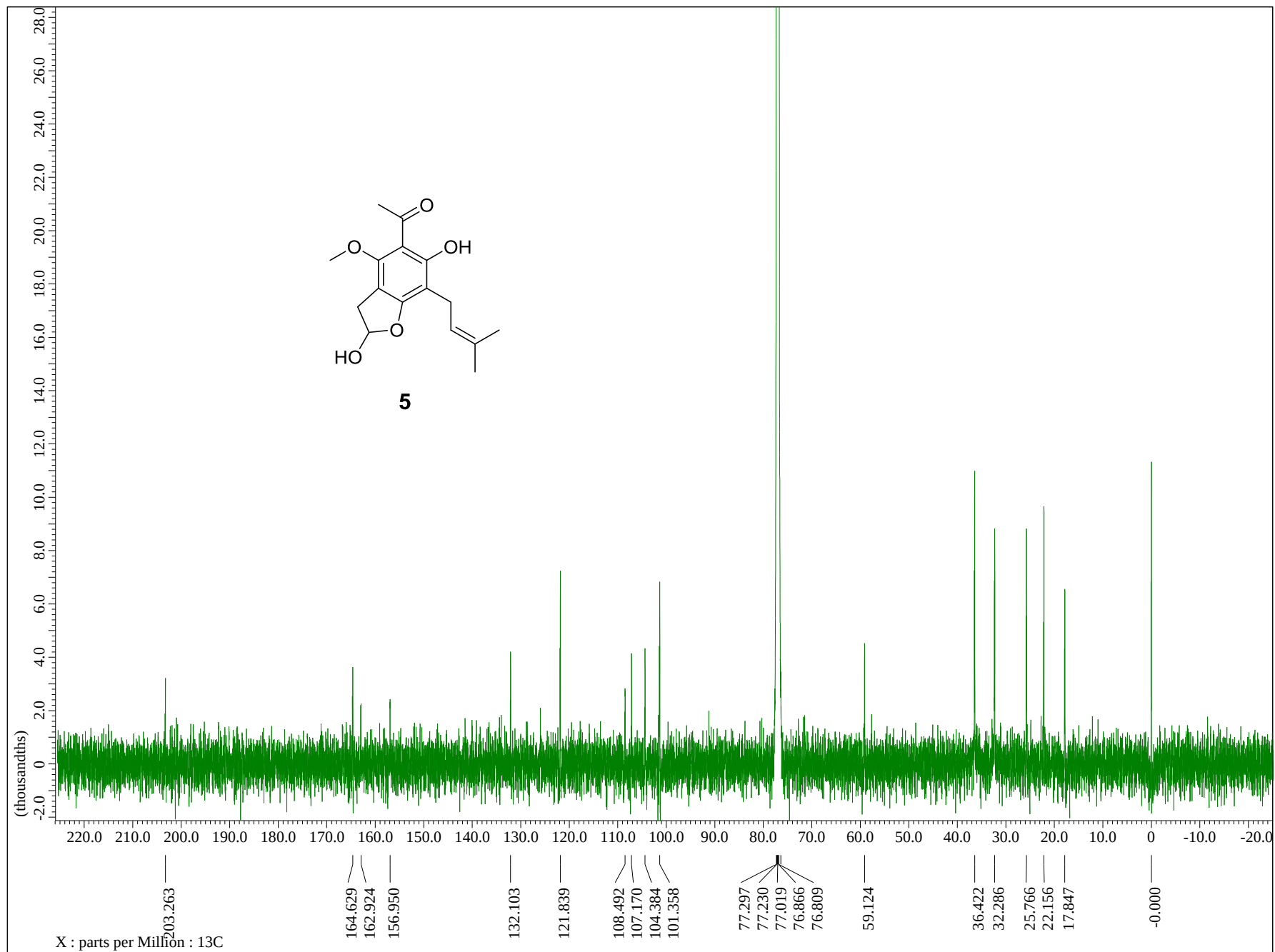


Figure S23. H-H COSY experiment of **5** (in CDCl₃).

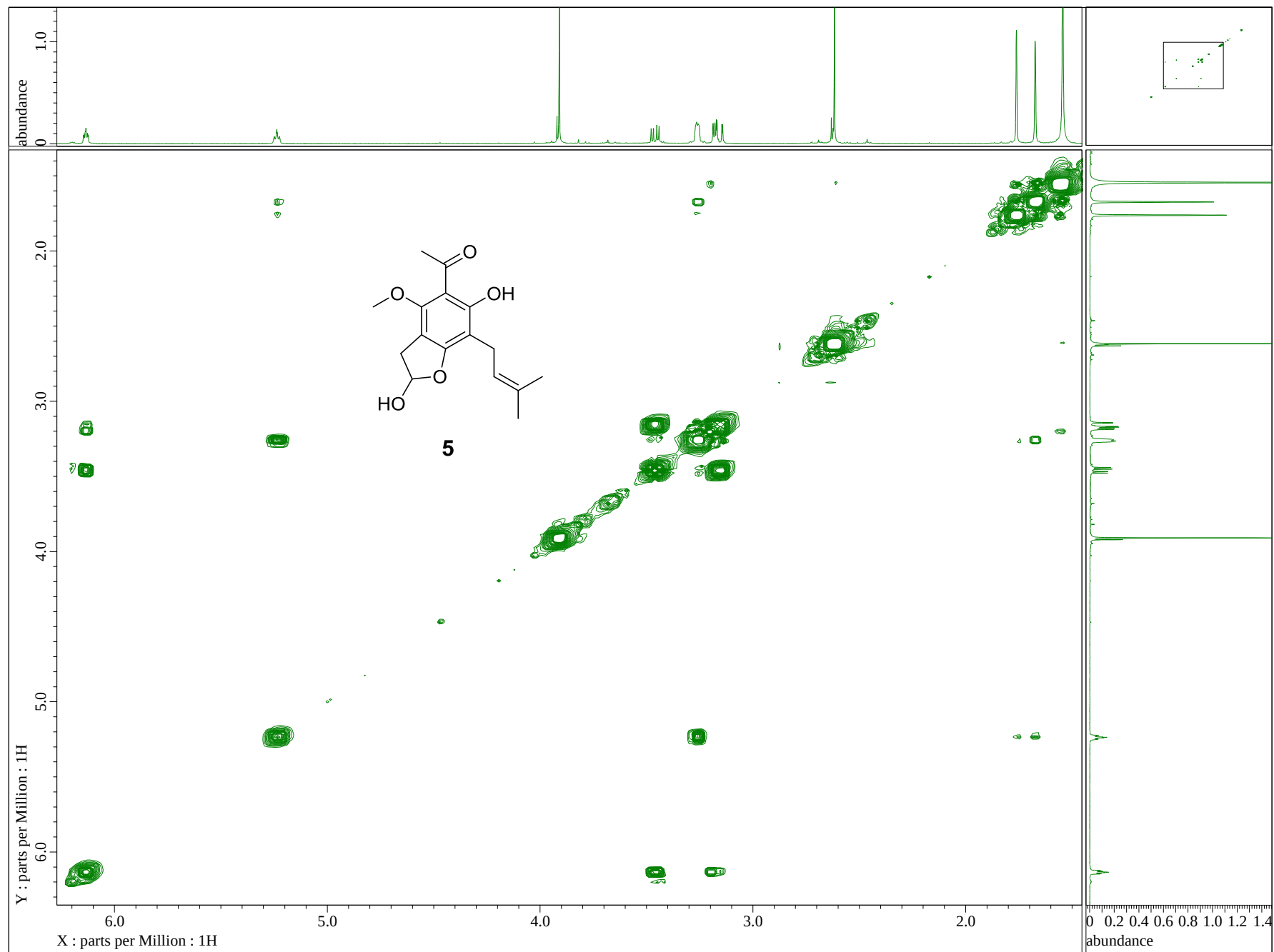


Figure S24. NOESY experiment of **5** (in CDCl₃).

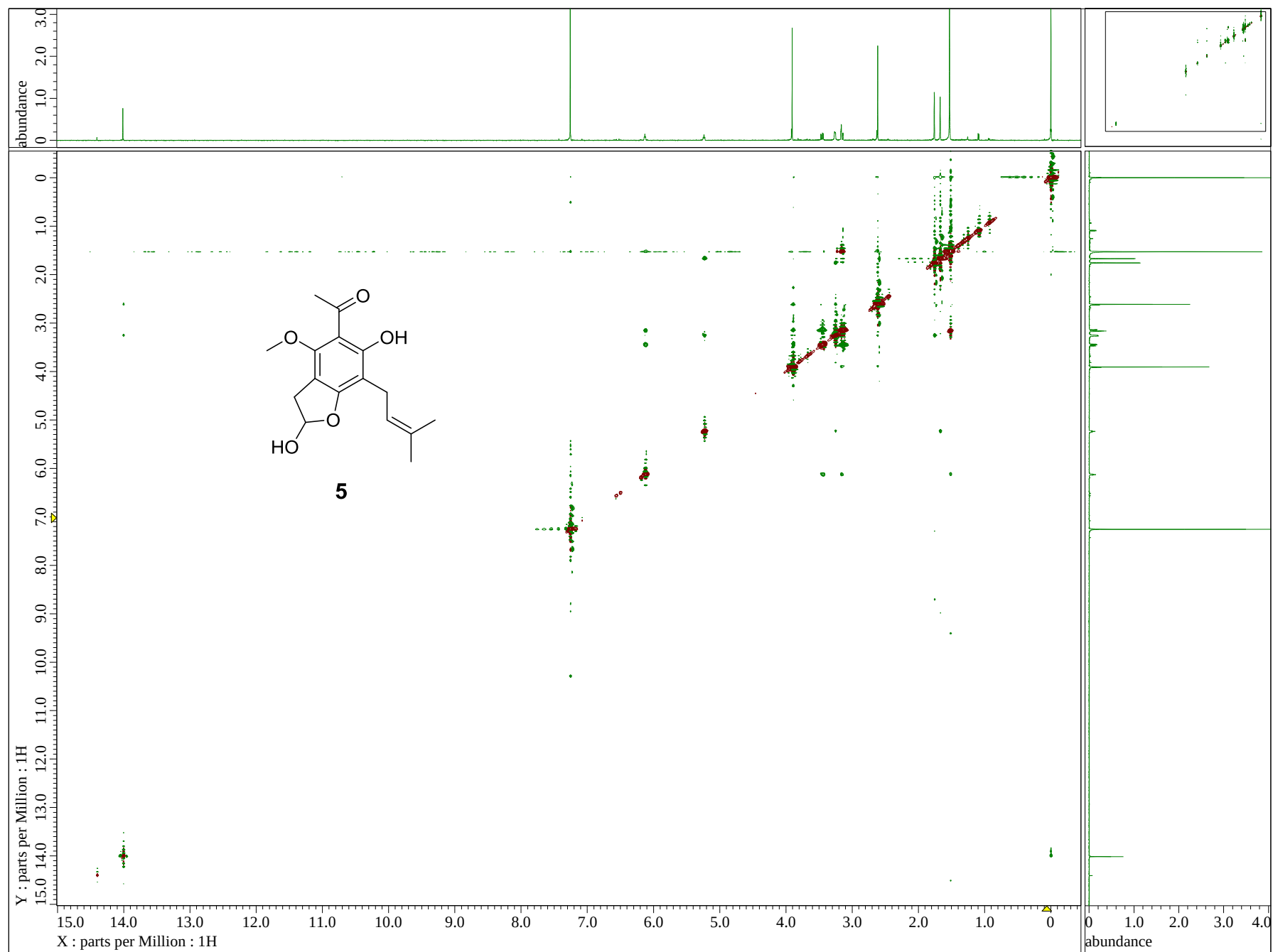


Figure S25. NOESY experiment of **5** (in CDCl₃).

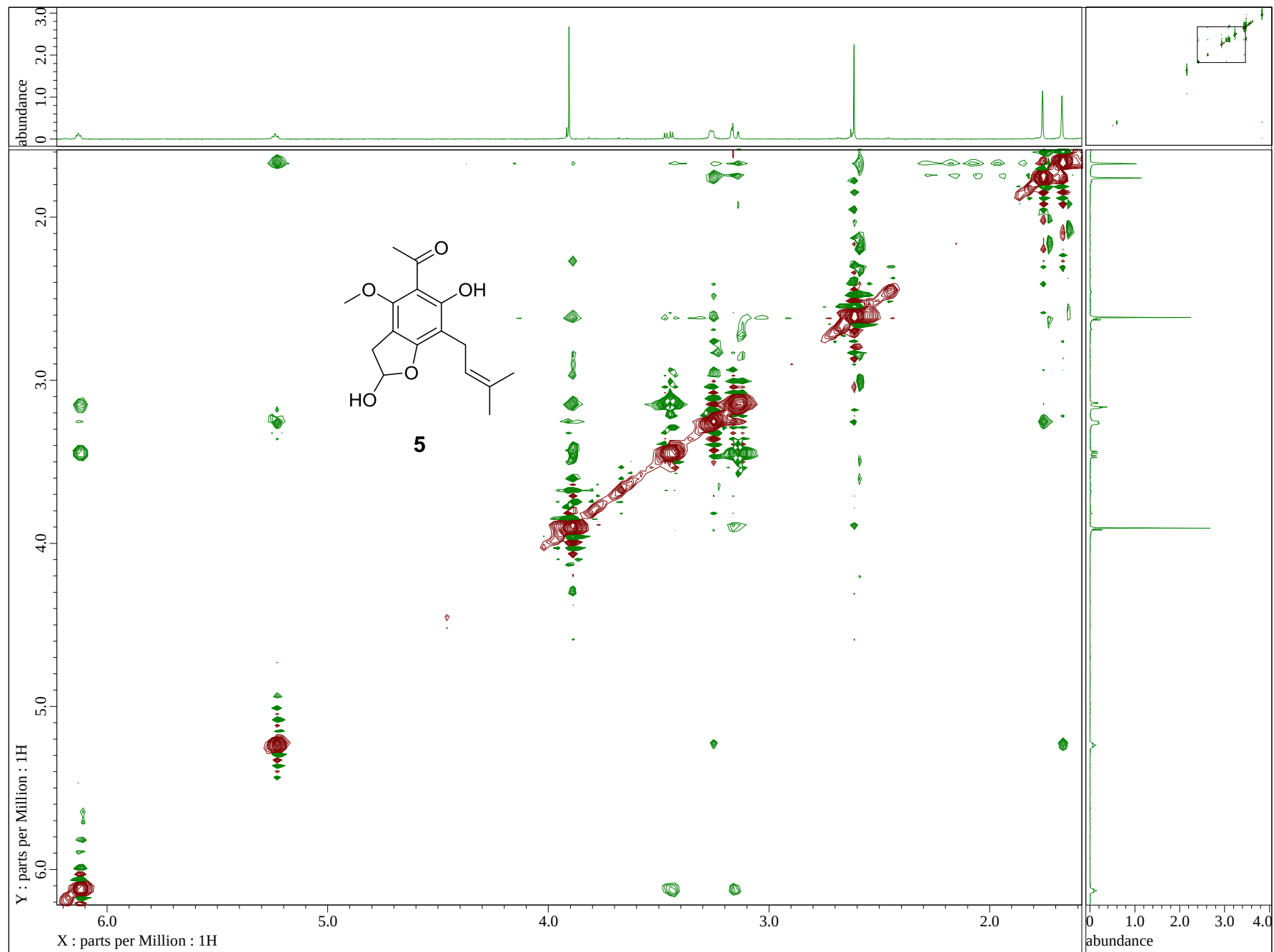


Figure S26. HMQC experiment of 5 (in CDCl₃).

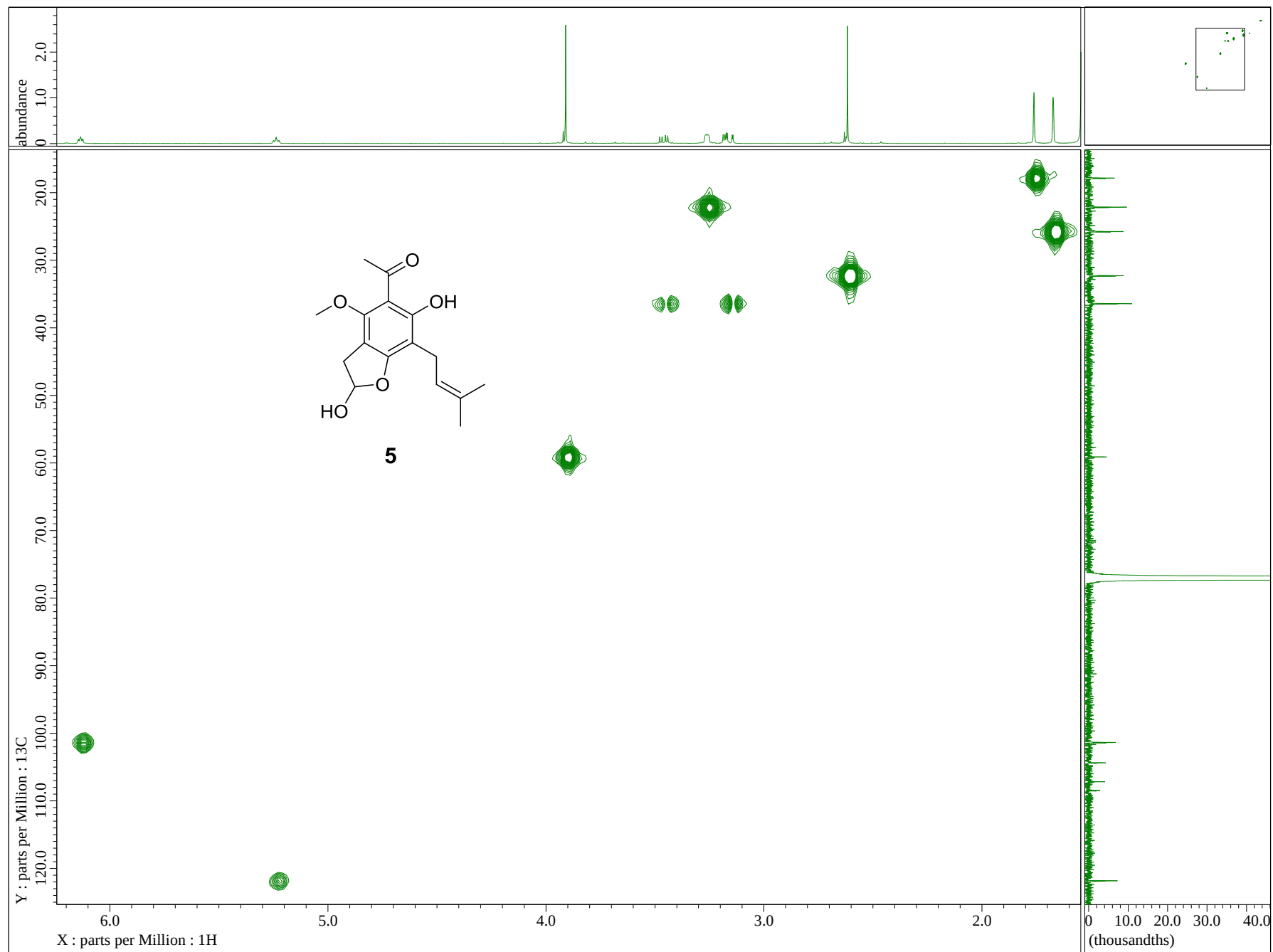


Figure S27. HMBC experiment of 5 (in CDCl₃).

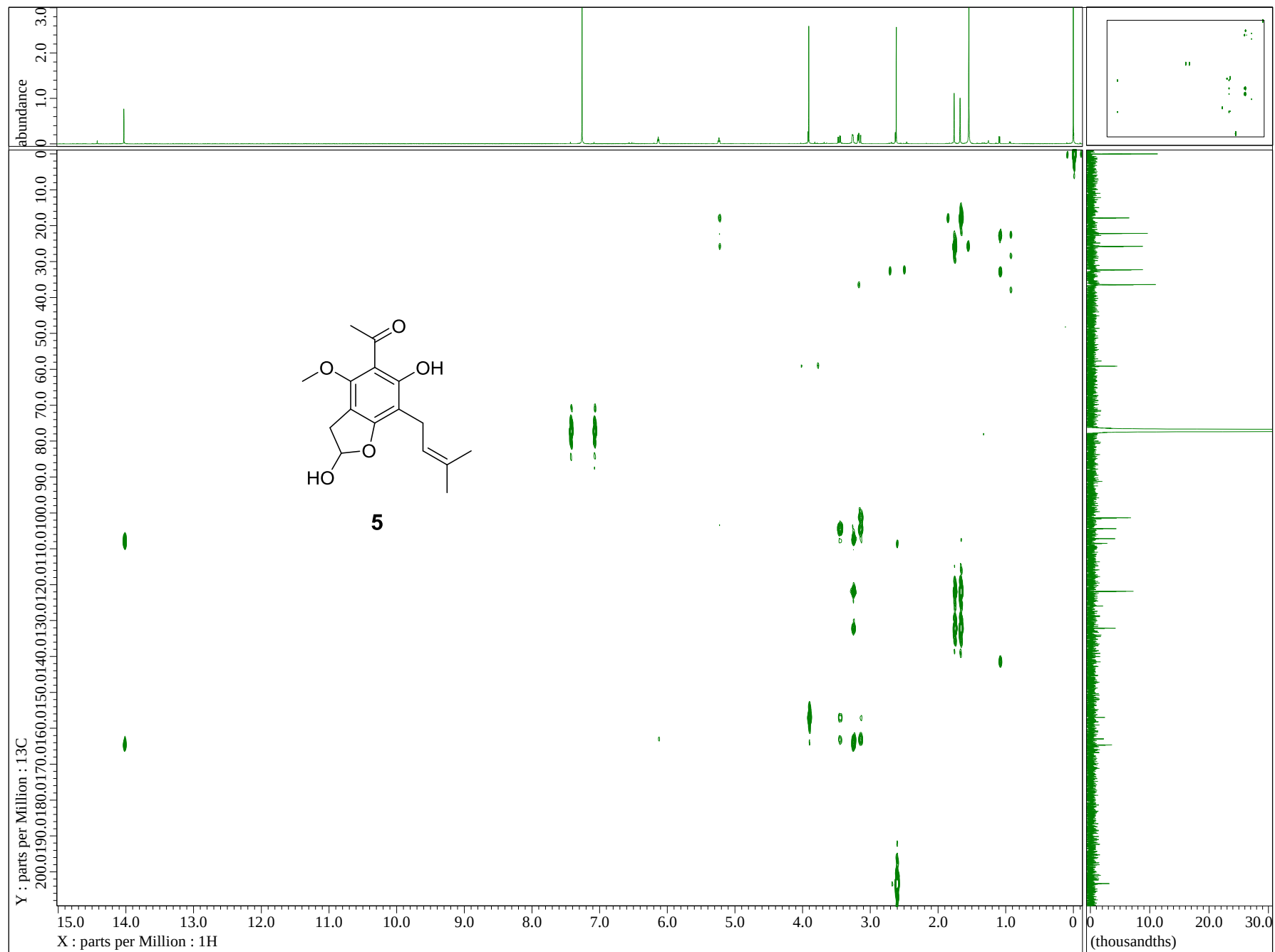


Figure S28. HMBC experiment of **5** (in CDCl₃).

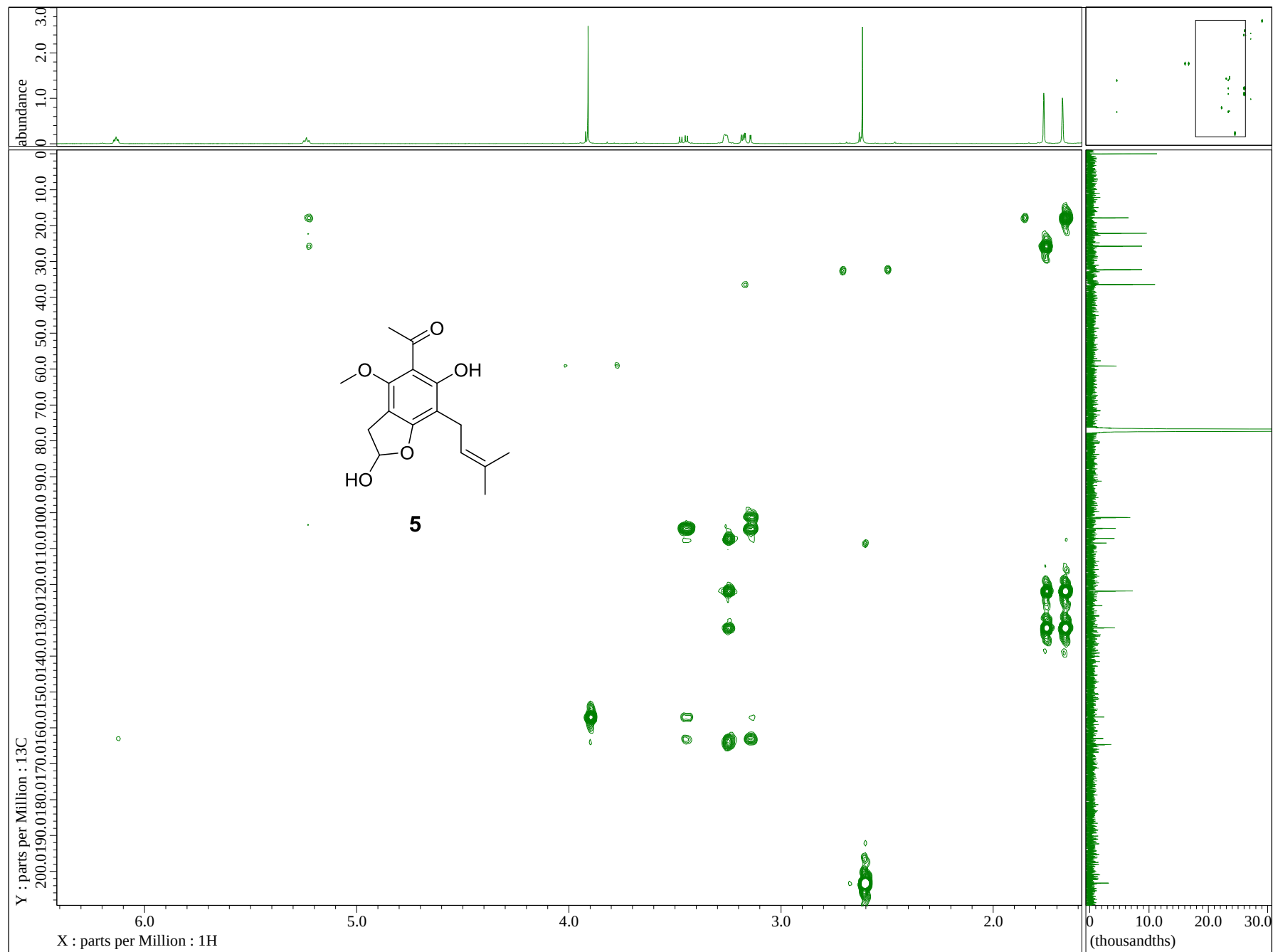
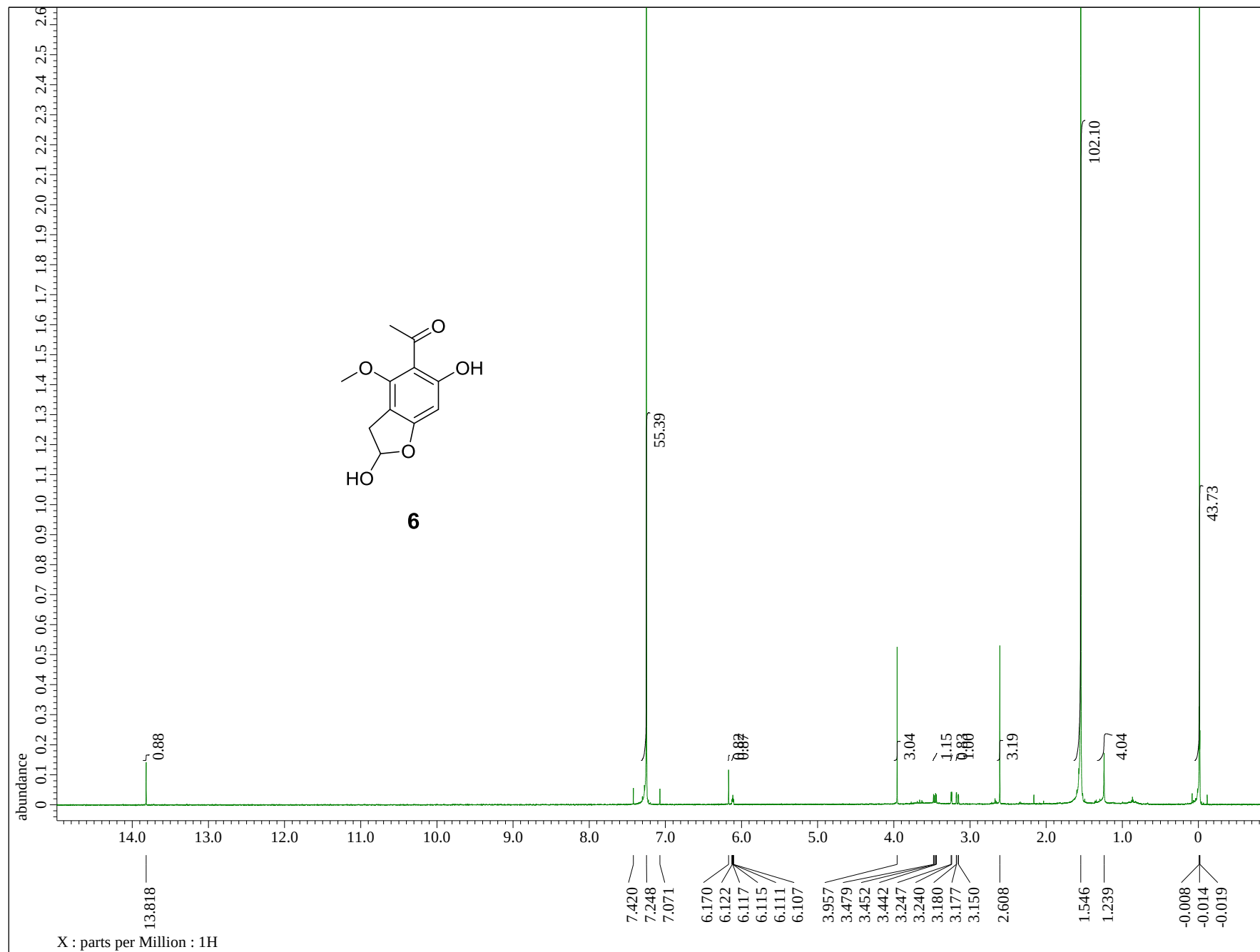


Figure S29. ^1H NMR spectrum of **6** (600 MHz, in CDCl_3).



Chemical structure of compound **6** is shown in the center of the plot:

COc1cc(O)c2c(c1)oc(CO)cc2

The 2D COSY spectrum displays correlations between protons. The x-axis is labeled "X : parts per Million : ^1H " and the y-axis is labeled "Y : parts per Million : ^1H ". Both axes range from 0.0 to 6.5 ppm. The spectrum shows diagonal peaks and off-diagonal cross-peaks. A chemical structure of compound **6** is shown in the center of the plot.

Figure S31. NOESY experiment of **6** (in CDCl₃).

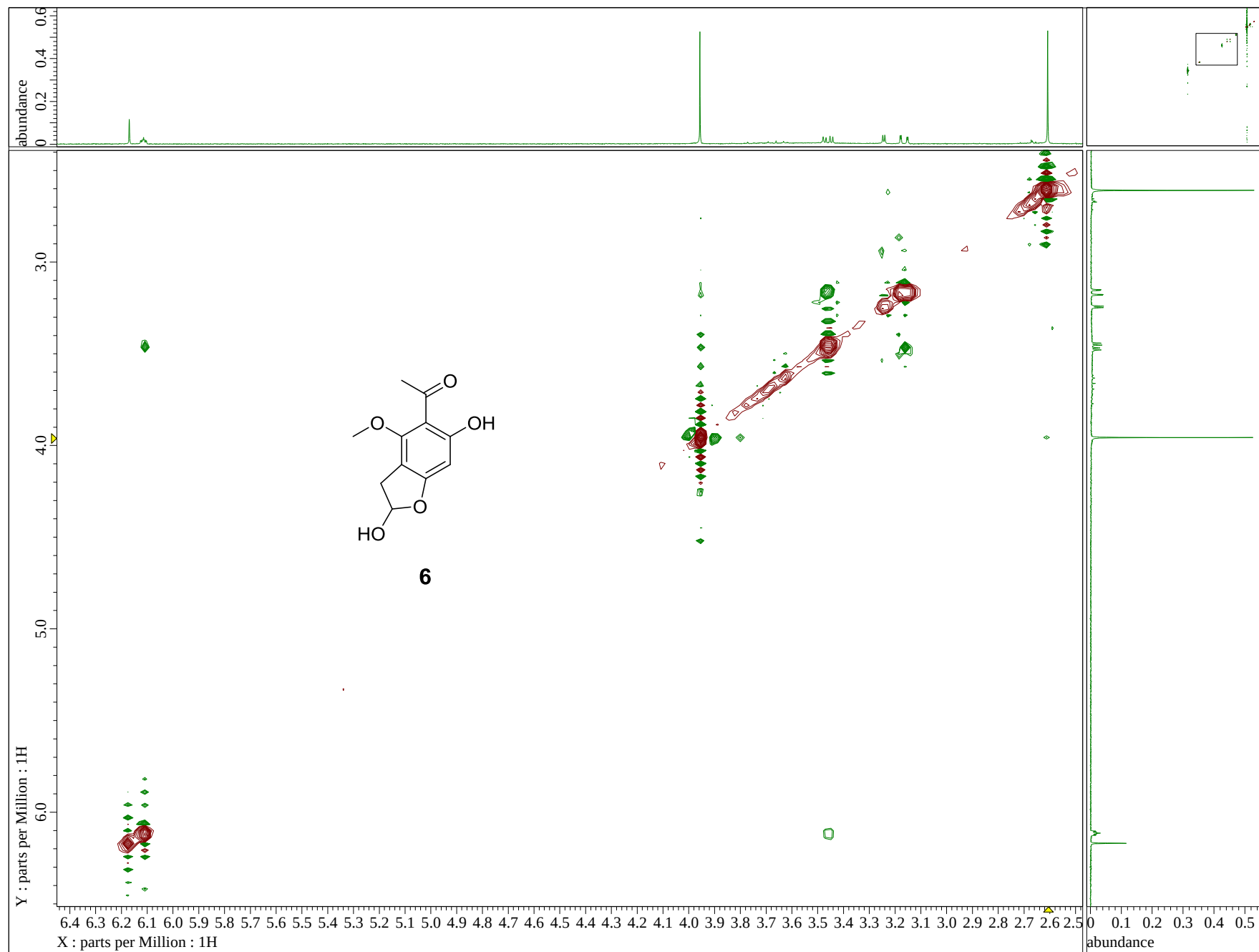


Figure S32. ^1H NMR spectrum of **7** (600 MHz, in CDCl_3).

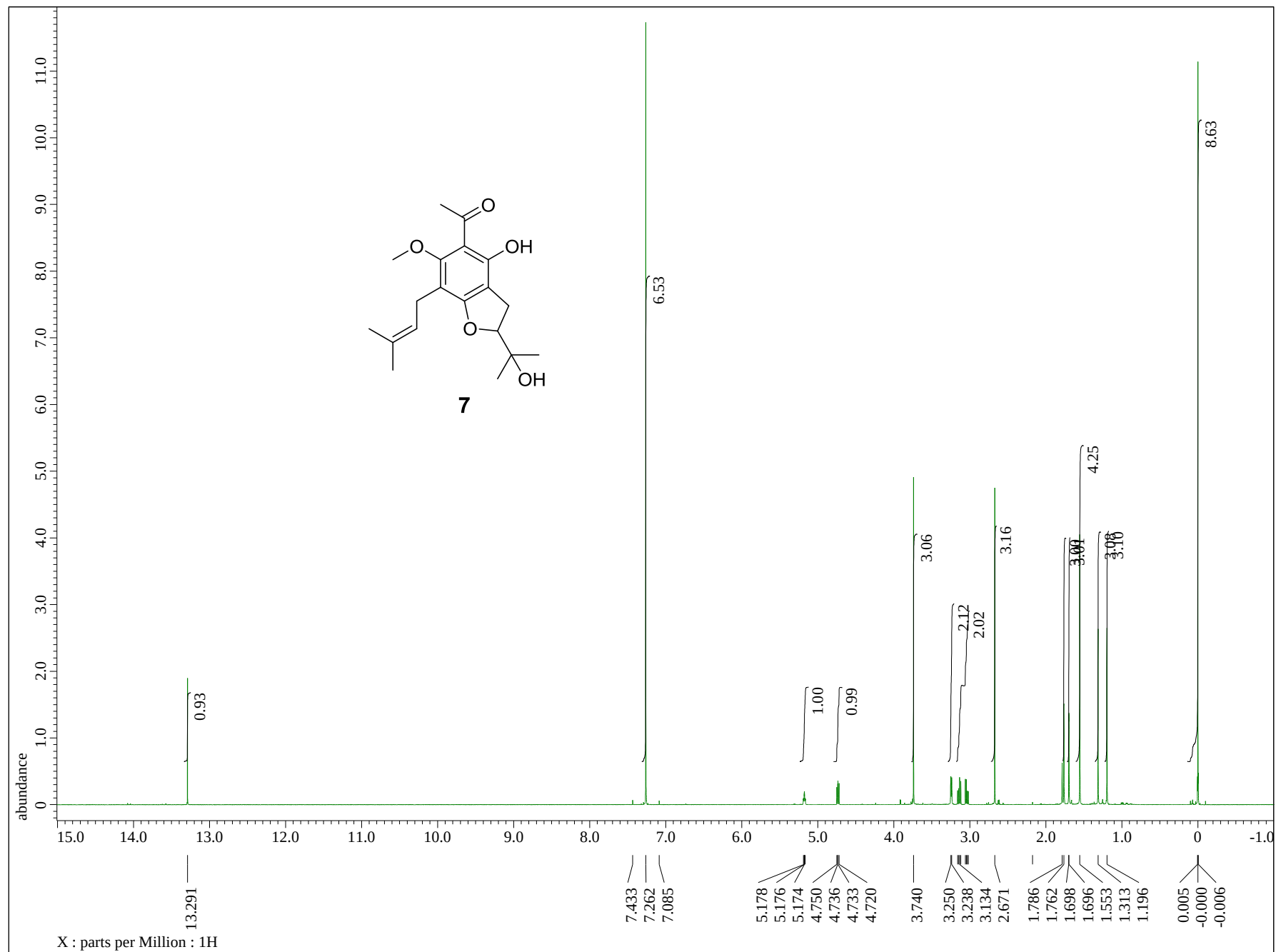


Figure S33. ^{13}C NMR spectrum of **7** (150 MHz, in CDCl_3).

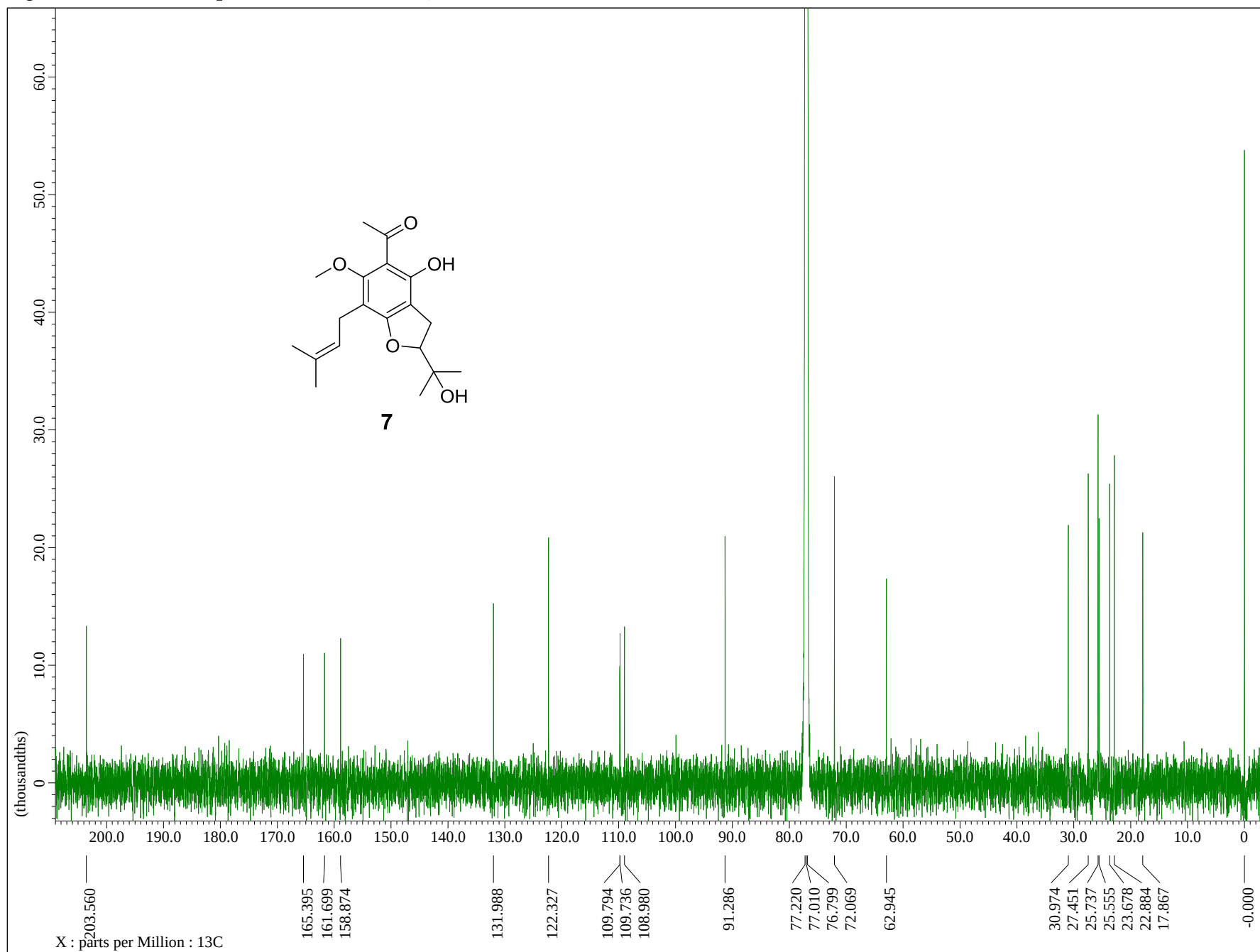


Figure S34. H-H COSY experiment of 7 (in CDCl₃).

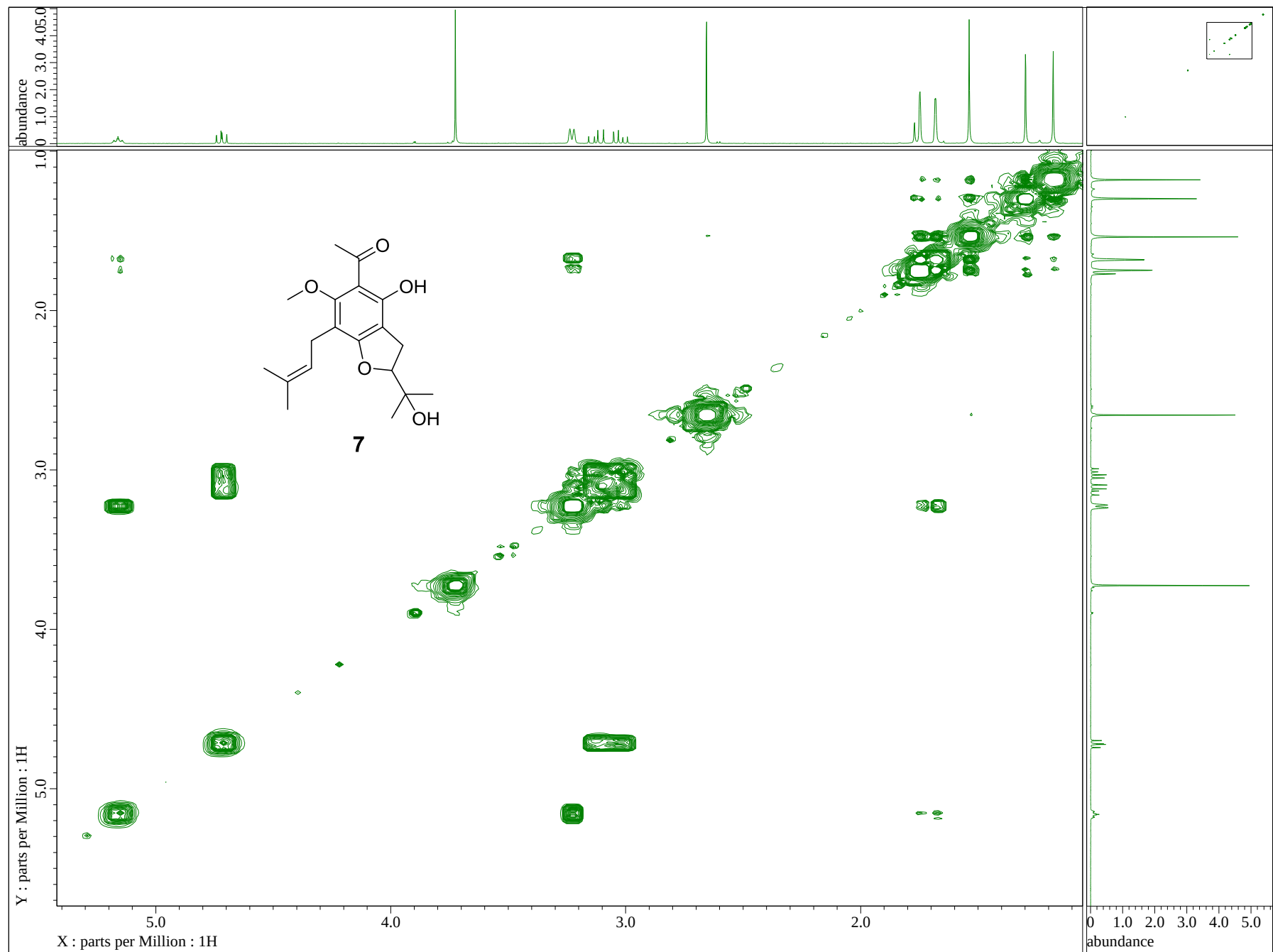


Figure S35. NOESY experiment of **7** (in CDCl₃).

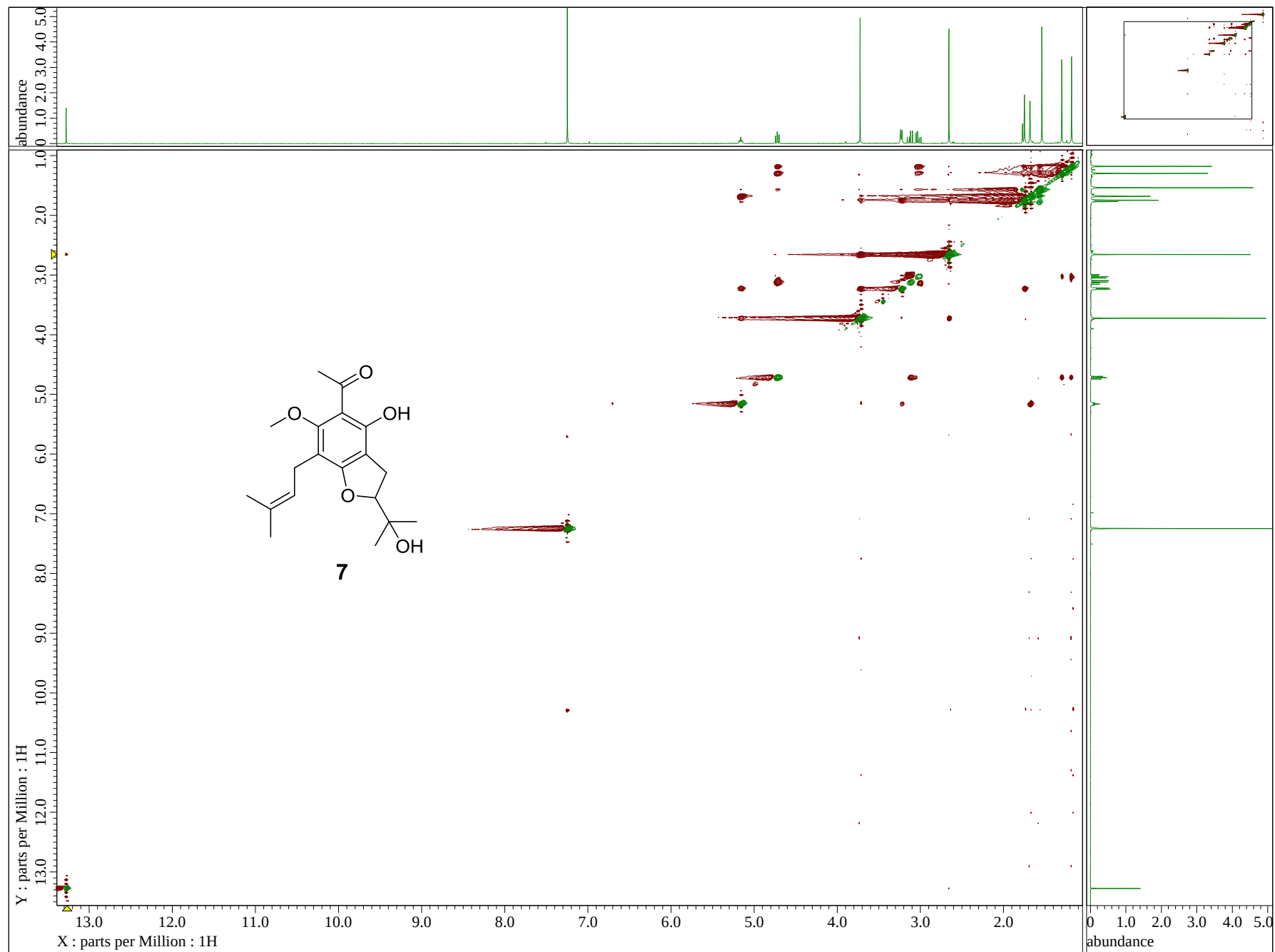


Figure S36. HMQC experiment of **7** (in CDCl₃).

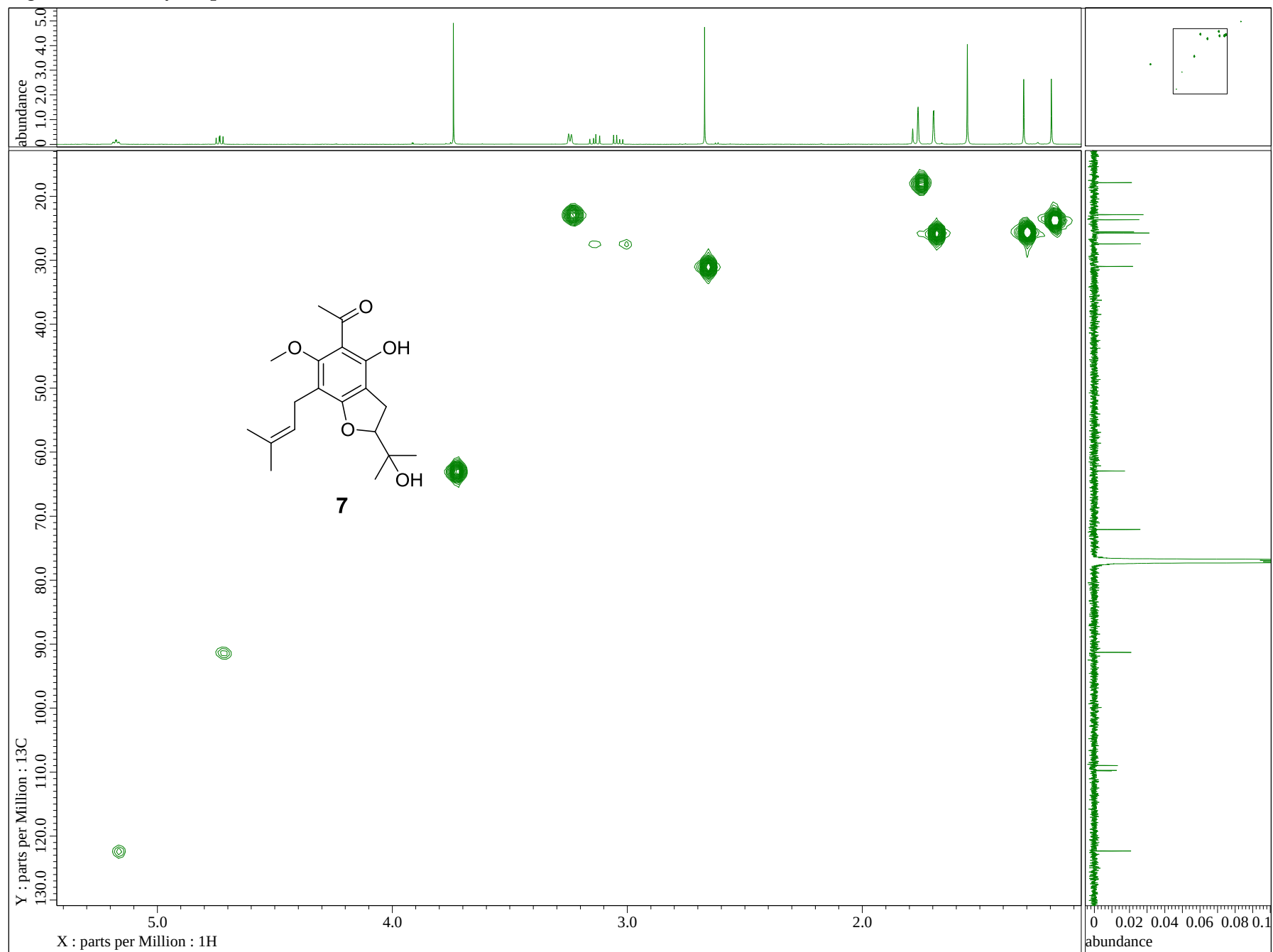


Figure S37. HMBC experiment of 7 (in CDCl₃).

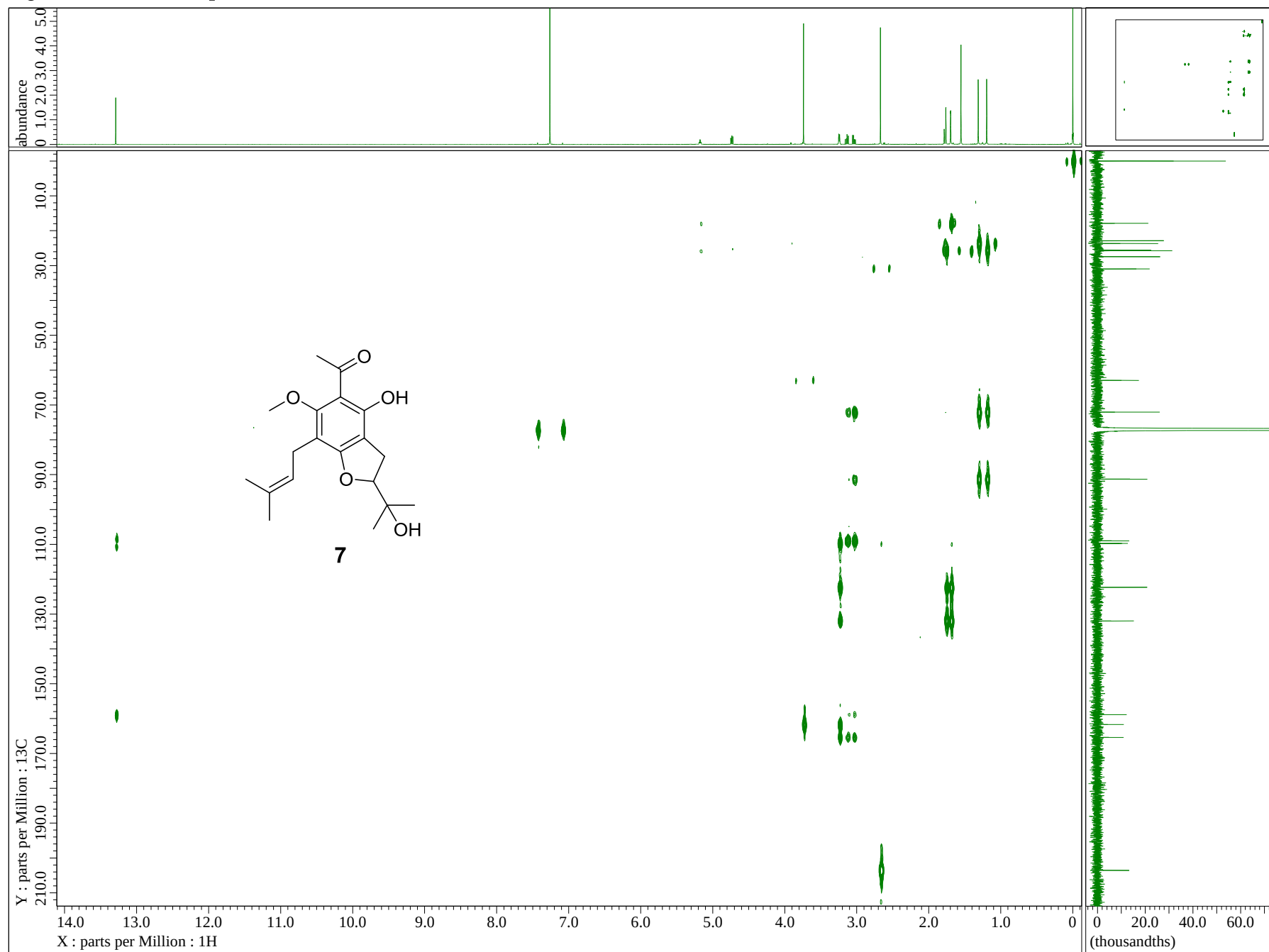


Figure S38. HMBC experiment of 7 (in CDCl₃).

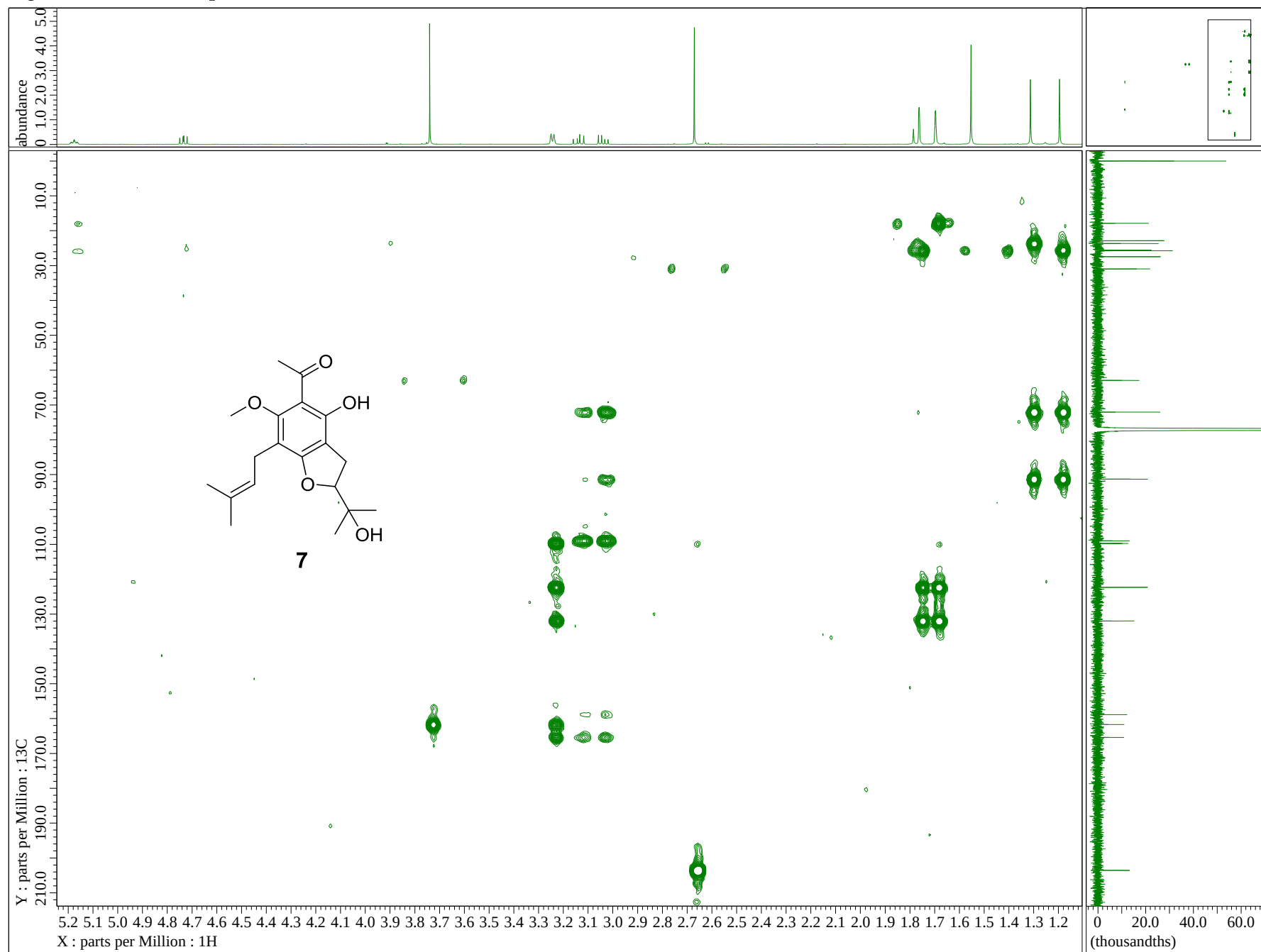


Figure S39. ^1H NMR spectrum of **8** (600 MHz, in acetone- d_6).

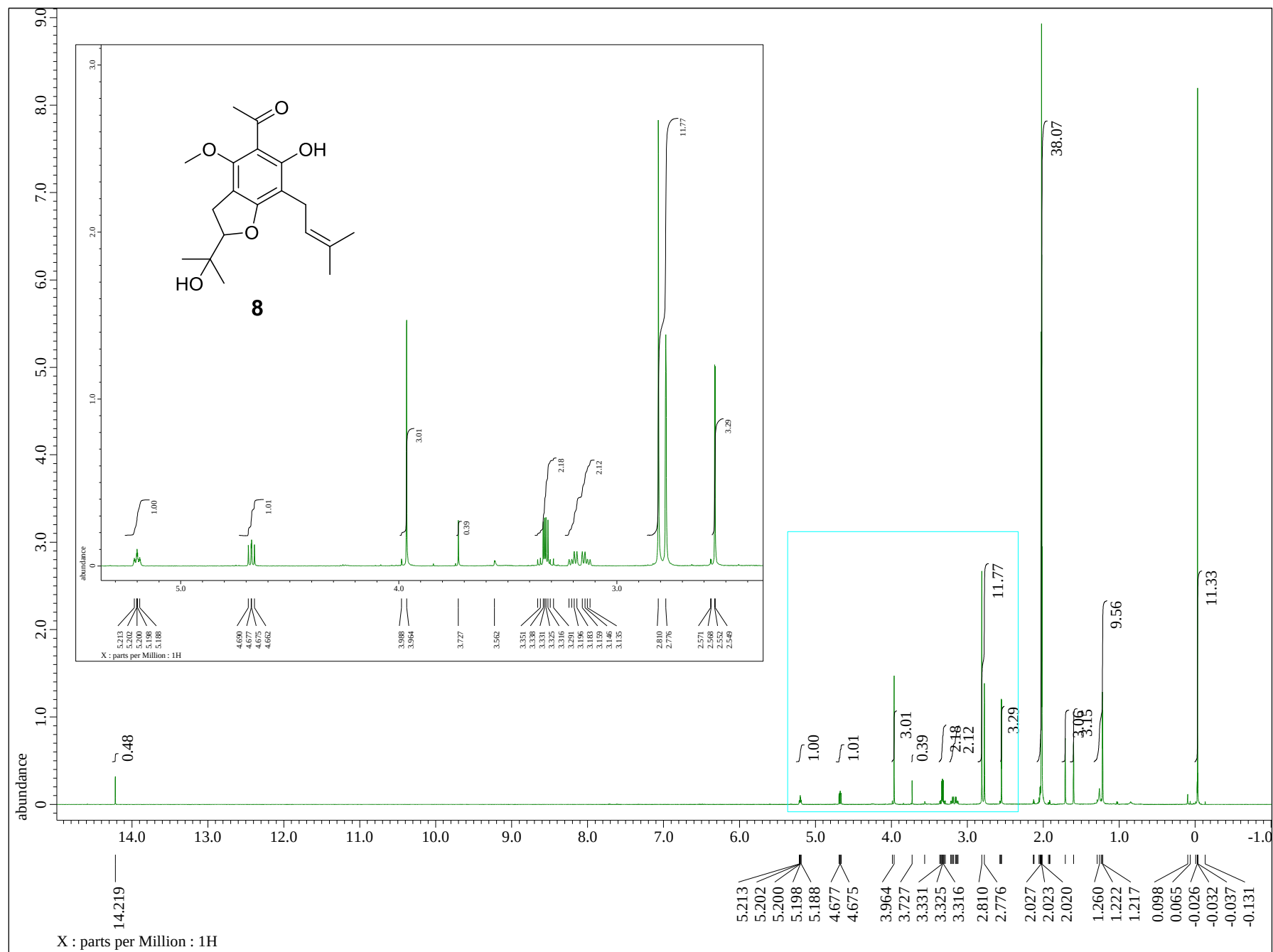


Figure S40. H-H COSY experiment of 8 (in acetone- d_6).

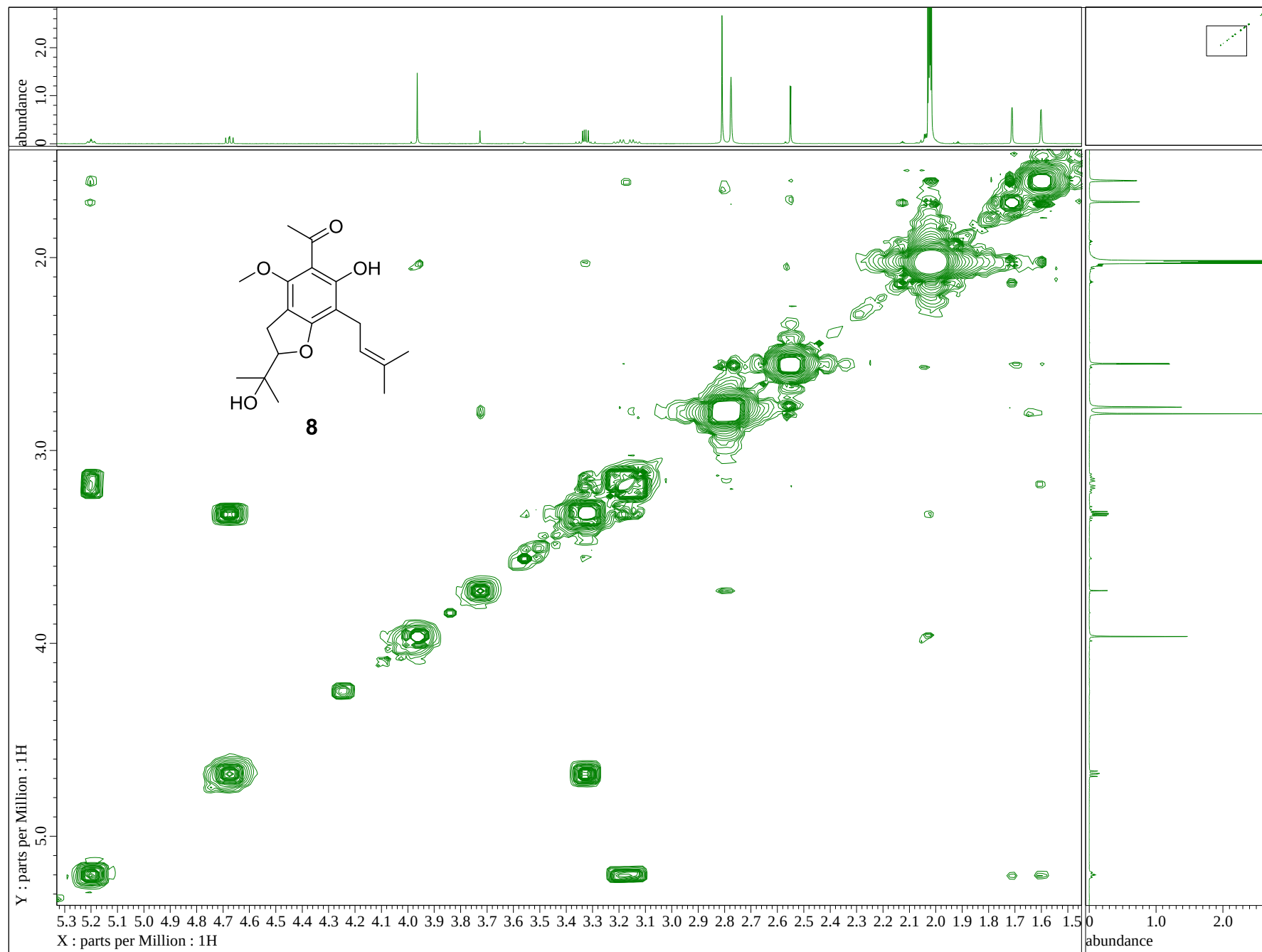


Figure S41. NOESY experiment of 8 (in acetone- d_6).

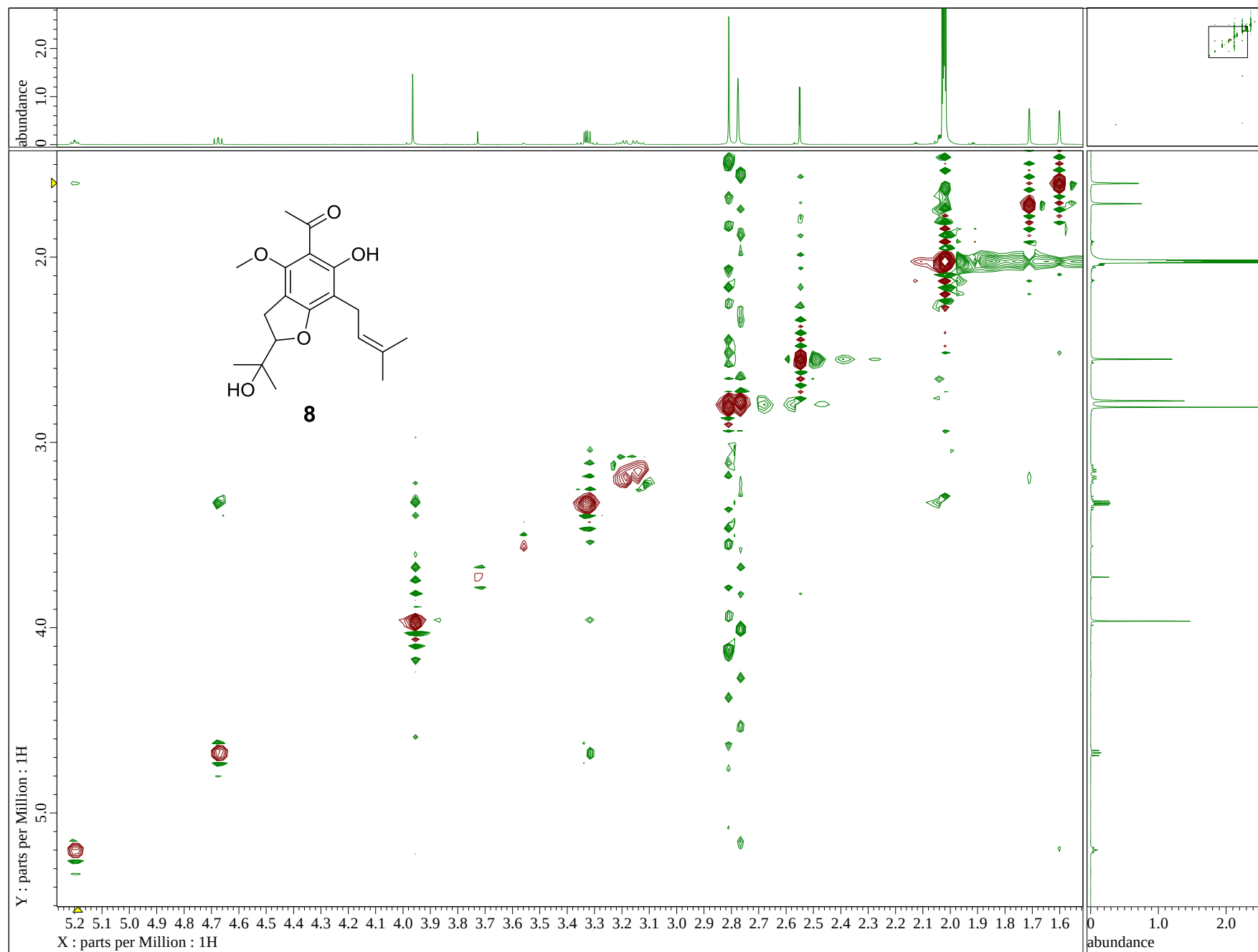


Table S42. ¹H and ¹³C NMR data of 7 and 8.

	7 ^a			8 ^b			8 ^a	
solvent	CDCl ₃			CDCl ₃			acetone- <i>d</i> ₆	
position	δ_{H} (<i>J</i> in Hz)		δ_{C}	δ_{H} (<i>J</i> in Hz)		δ_{C}	δ_{H} (<i>J</i> in Hz)	
1			203.6			203.0		
2	2.67	s	31.0	2.61	s	32.1	2.55	s
1'			109.8			106.5		
2'			158.9			164.7		
3'			109.0			108.2		
4'			165.4			164.8		
5'			109.7			107.1		
6'			161.7			156.7		
1''	3.25	brd (6.4)	22.9	3.26	d (8.0)	22.1	3.31	dd (15.0, 9.0)
							3.34	dd (15.0, 7.2)
2''	5.18	brt (7.6)	122.3	4.64	t (8.0)	90.1	4.68	dd (9.0, 7.8)
3''			132.0			71.8		
4''	1.70	brs	25.7	1.24	s	25.7	1.217	s
5''	1.76	brs	17.9	1.36	s	25.8	1.222	s
1'''	3.04	dd (15.6, 8.0)	27.5	3.22	d (6.6)	28.9	3.14	dd (14.4, 7.8)
	3.14	dd (15.2, 10.0)					3.20	dd (14.4, 7.8)
2'''	4.74	t (10.0)	91.3	5.24	t (6.6)	121.9	5.20	t (7.5)
3'''			72.1			131.7		
4'''	1.20	s	23.7	1.67	s	25.7	1.60	brs
5'''	1.31	s	25.6	1.76	s	17.8	1.71	brs
MeO	3.74	s	62.9	3.90	s	59.1	3.96	s
OH	13.30	s		14.52	s		14.21	s

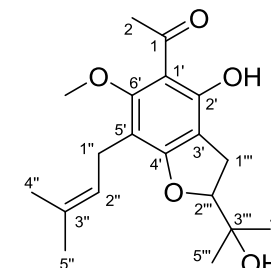
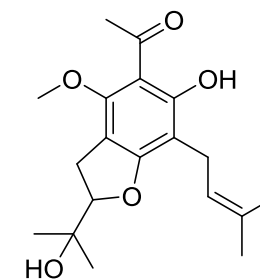
^a Measured at 600 MHz for ¹H NMR and 150 MHz for ¹³C NMR. ^b Ref. 3.**7****8**

Figure S43. HRFABMS data of 1.

Data : 分子生薬学 (後藤) 234 Date : 05-Feb-2016 15:34
Instrument : MStation
Sample : 14AS_mm4
Note : NBA
Inlet : Direct Ion Mode : FAB+
RT : 2.35 min Scan# : 22
Elements : C 19/0, H 27/0, O 5/0
Mass Tolerance : 1000ppm, 5mmu if m/z < 5, 50mmu if m/z > 50
Unsaturation (U.S.) : -0.5 - 20.0

	Observed m/z	Int%				
	335.1845	100.00				
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	O
1	335.1858	-4.0 / -1.3	6.5	19	27	5

Figure S44. HRFABMS data of 2.

Data : 分子生薬学 (後藤) 235 Date : 05-Feb-2016 15:40
Instrument : MStation
Sample : 14AS_mm5
Note : NBA
Inlet : Direct Ion Mode : FAB+
RT : 2.46 min Scan# : 23
Elements : C 19/0, H 29/0, O 5/0
Mass Tolerance : 1000ppm, 5mmu if m/z < 5, 50mmu if m/z > 50
Unsaturation (U.S.) : -0.5 - 20.0

	Observed m/z	Int%				
	337.2012	100.00				
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	O
1	337.2015	-0.9 / -0.3	5.5	19	29	5

Figure S45. HRFABMS data of 3.

Data : 分子生薬学 (後藤) 237 Date : 05-Feb-2016 15:53
Instrument : MStation
Sample : 14AS_mm9
Note : NBA
Inlet : Direct Ion Mode : FAB+
RT : 2.24 min Scan# : 21
Elements : C 19/0, H 27/0, O 5/0
Mass Tolerance : 1000ppm, 5mmu if m/z < 5, 50mmu if m/z > 50
Unsaturation (U.S.) : -0.5 - 20.0

	Observed m/z	Int%					
	335.1850	100.00					
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	O	
1	335.1858	-2.5 / -0.8	6.5	19	27	5	

Figure S46. HRFABMS data of 4.

Data : 分子生薬学 (後藤) 236 Date : 05-Feb-2016 15:47
Instrument : MStation
Sample : 14AS_mm6
Note : NBA
Inlet : Direct Ion Mode : FAB+
RT : 2.02 min Scan# : 19
Elements : C 19/0, H 27/0, O 5/0
Mass Tolerance : 1000ppm, 5mmu if m/z < 5, 50mmu if m/z > 50
Unsaturation (U.S.) : -0.5 - 20.0

	Observed m/z	Int%					
	335.1828	100.00					
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	O	
1	335.1858	-9.1 / -3.0	6.5	19	27	5	

Figure S47. HRFABMS data of 5.

Data : 分子生薬学 (後藤) 238 Date : 05-Feb-2016 16:00
 Instrument : MStation
 Sample : 14AS_mm3
 Note : NBA
 Inlet : Direct Ion Mode : FAB+
 RT : 2.83 min Scan# : 24
 Elements : C 16/0, H 21/0, O 5/0
 Mass Tolerance : 1000ppm, 5mmu if m/z < 5, 50mmu if m/z > 50
 Unsaturation (U.S.) : -0.5 - 20.0

	Observed m/z	Int%				
	293.1392	100.00				
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	O
1	293.1389	+1.0 / +0.3	6.5	16	21	5

Figure S48. HRFABMS data of 6.

Data : 分子生薬学 (後藤) 173 Date : 04-Jun-2015 13:27
 Instrument : MStation
 Sample : 15YK44C
 Note : NBA
 Inlet : Direct Ion Mode : FAB+
 RT : 3.95 min Scan# : 26
 Elements : C 11/0, H 38/0, O 5/0
 Mass Tolerance : 1000ppm, 5mmu if m/z < 5, 50mmu if m/z > 50
 Unsaturation (U.S.) : -0.5 - 10.0

	Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
1	225.0764	100.00	+0.5 / +0.1	5.5	C11 H13 O5

Figure S49. HRFABMS data of 7.

Data : 分子生薬学 (後藤) 233 Date : 05-Feb-2016 15:27
Instrument : MStation
Sample : 14AS_mm2
Note : NBA
Inlet : Direct Ion Mode : FAB+
RT : 2.13 min Scan# : 20
Elements : C 19/0, H 27/0, O 5/0
Mass Tolerance : 1000ppm, 5mmu if m/z < 5, 50mmu if m/z > 50
Unsaturation (U.S.) : -0.5 - 20.0

	Observed m/z	Int%					
	335.1845	100.00					
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	O	
1.	335.1858	-4.0 / -1.3	6.5	19	27	5	

Figure S50. HRFABMS data of 8.

Data : 分子生薬学 (後藤) 240 Date : 24-Feb-2016 13:59
Instrument : MStation
Sample : 14AS_mm1
Note : NBA
Inlet : Direct Ion Mode : FAB+
RT : 2.35 min Scan# : 22
Elements : C 19/0, H 27/0, O 5/0
Mass Tolerance : 1000ppm, 5mmu if m/z < 5, 50mmu if m/z > 50
Unsaturation (U.S.) : -0.5 - 20.0

	Observed m/z	Int%					
	335.1814	100.00					
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	O	
1	335.1858	-13.3 / -4.4	6.5	19	27	5	

Table 51. Optical Rotations of 1–4, 7 and 8

Acronyculatin I (1). $[\alpha]_{\text{D}}^{21} -2.7$ (*c* 0.06, CH₃OH)

Acronyculatin J (2). $[\alpha]_{\text{D}}^{21} +8.4$ (*c* 0.02, CH₃OH).

Acronyculatin K (3). $[\alpha]_{\text{D}}^{21} +2.0$ (*c* 0.03, CH₃OH).

Acronyculatin L (4). $[\alpha]_{\text{D}}^{21} -7.4$ (*c* 0.03, CH₃OH).

Acronyculatin O (7). $[\alpha]_{\text{D}}^{21} +0.7$ (*c* 0.06, CH₃OH).

Acronyculatin B (8). $[\alpha]_{\text{D}}^{21} +2.7$ (*c* 0.04, CH₃OH).