

Biological Evaluations, NMR Analyses, Molecular Modelling Studies, and Overview of the Synthesis of the Marine Natural Product (–)-Mucosin

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Innhold

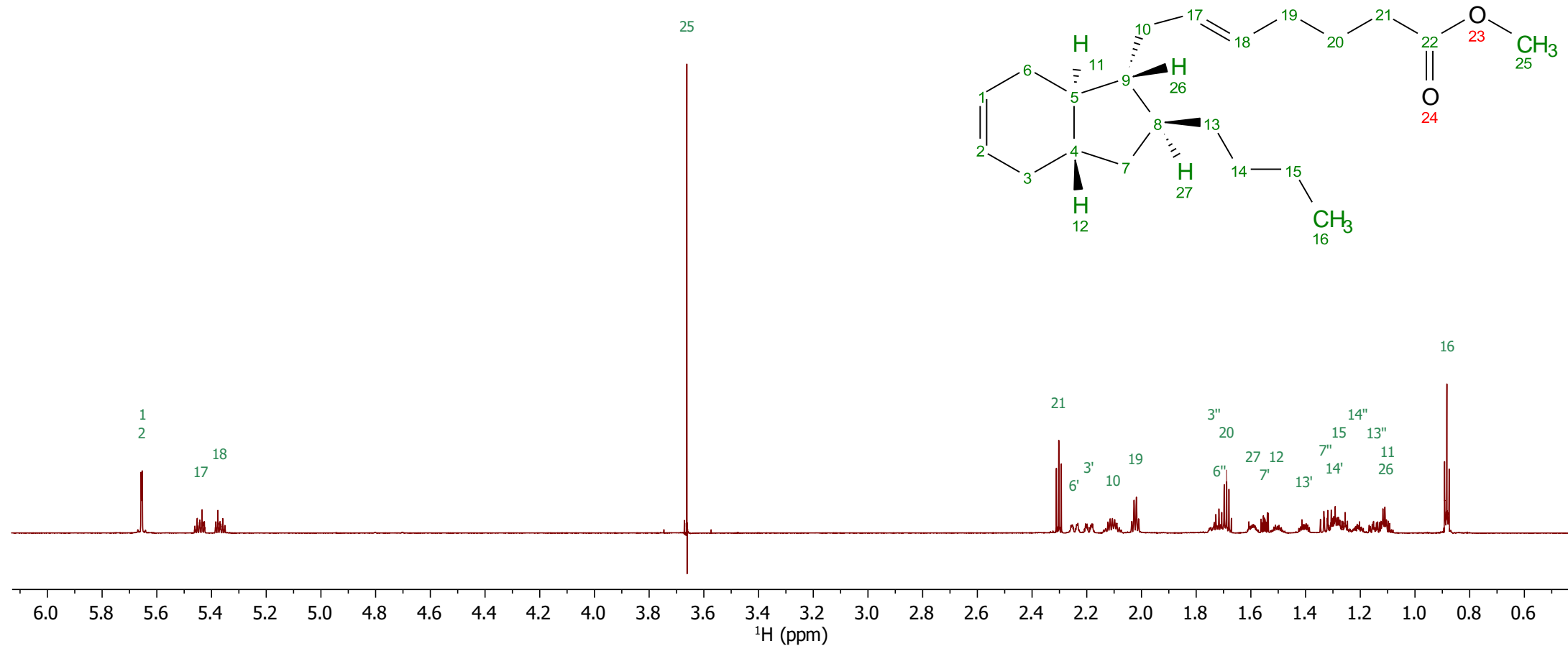
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General Information

The stated yields are based on isolated material.

The NMR spectra were acquired at 298 K on an 850 MHz Bruker AVANCE III HD equipped with a TCI CryoProbe (Bruker BioSpin, Billerica, MA, USA). Coupling constants (*J*) are reported in hertz and chemical shifts (δ) are reported in parts per million (ppm), referenced to the residual solvent signal (7.26 ppm for ¹H and 77.16 ppm for ¹³C).

PROTON



C13

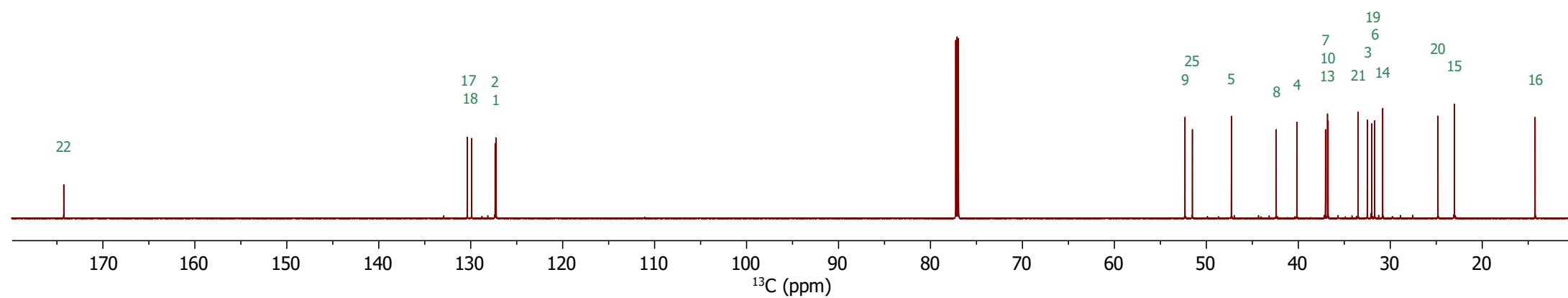


Figure S1: ^1H and ^{13}C NMR of **7** with assignment

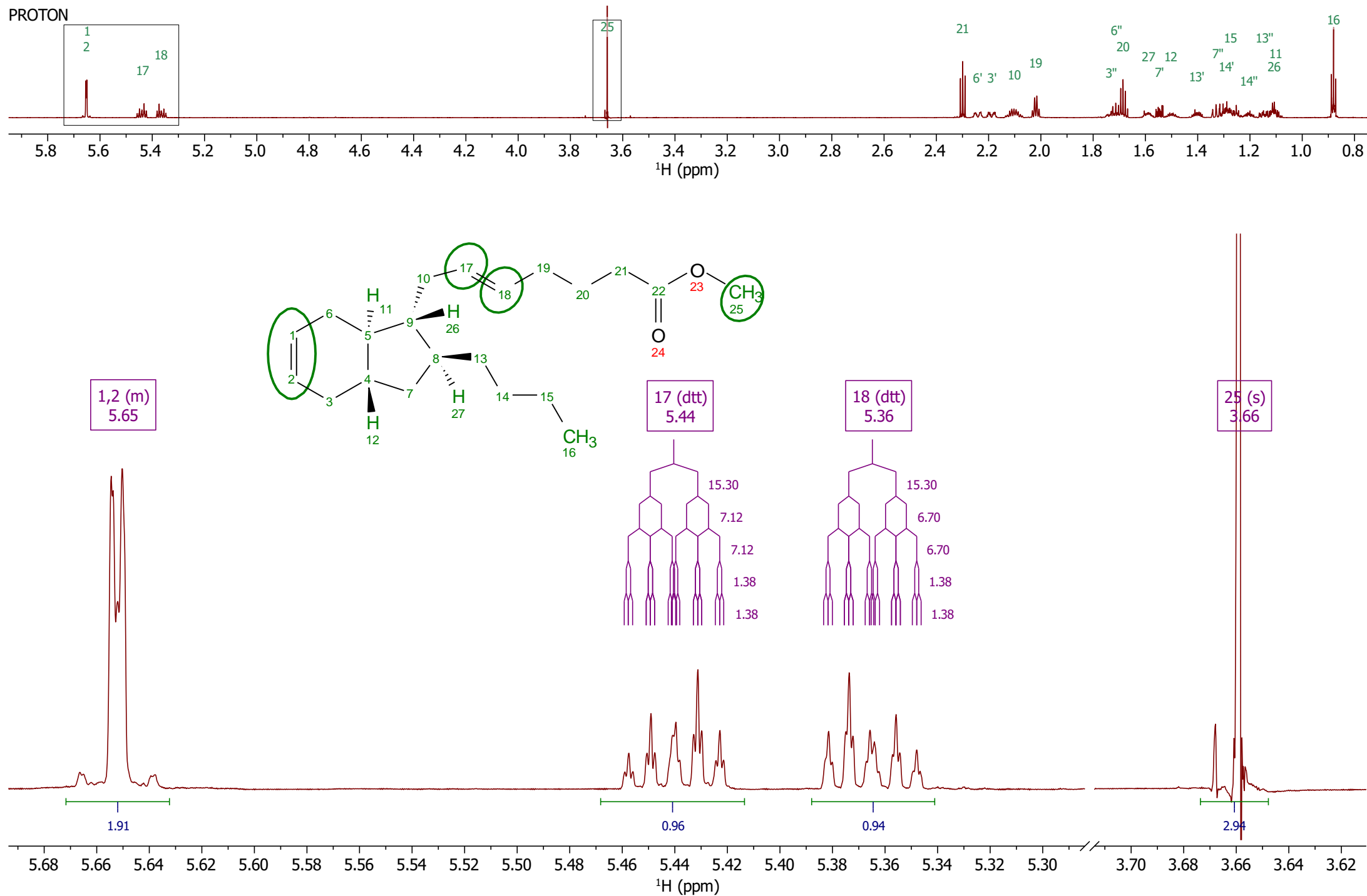


Figure S2: Extract of ^1H NMR of **7** with assignment

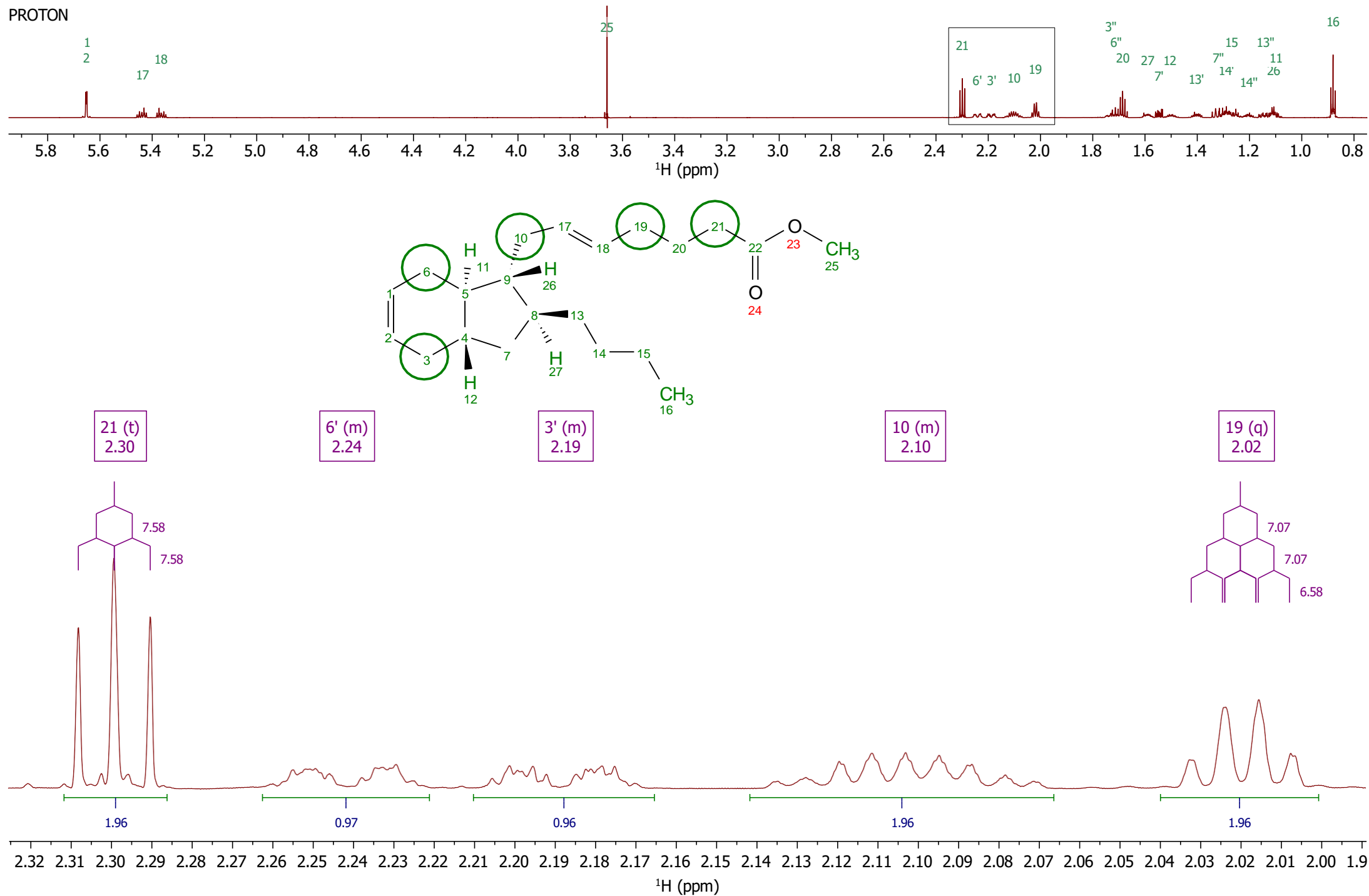
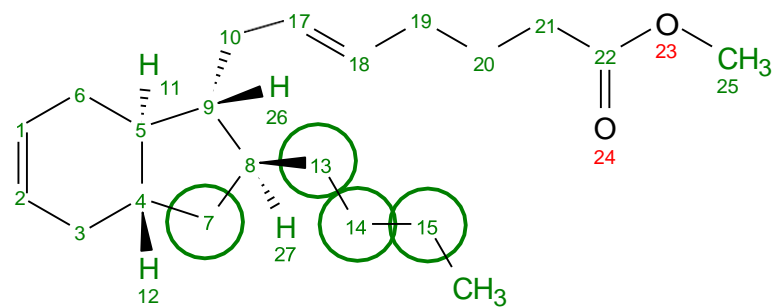
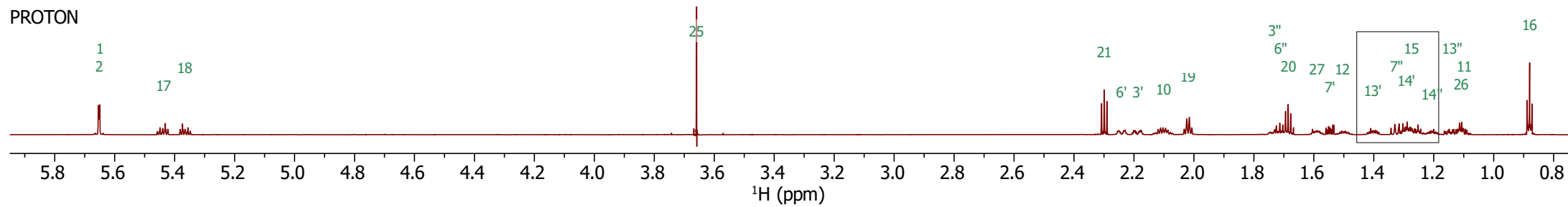


Figure S3: Extract of ^1H NMR of **7** with assignment



13' (dddd)
1.40

7'' (ddd)
1.32

14' (m)
1.29

15 (m)
1.27

14'' (m)
1.21

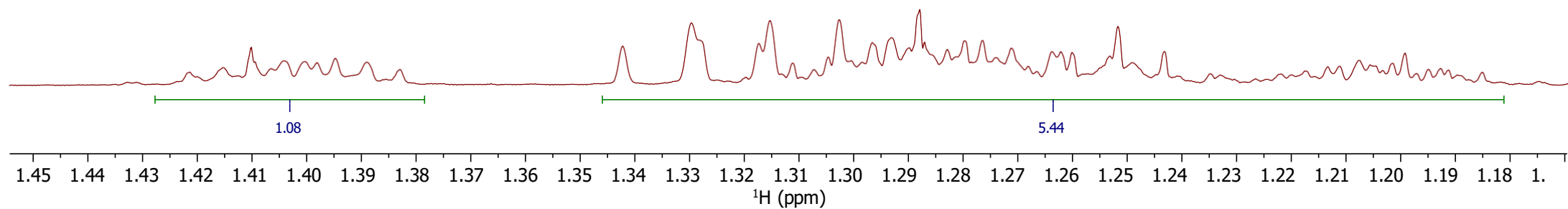
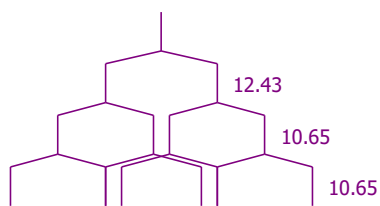
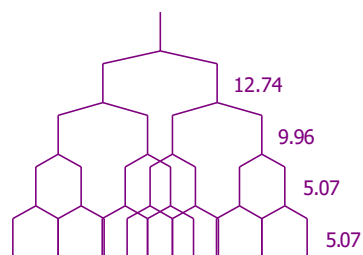


Figure S5: Extract of ^1H NMR of **7** with assignment

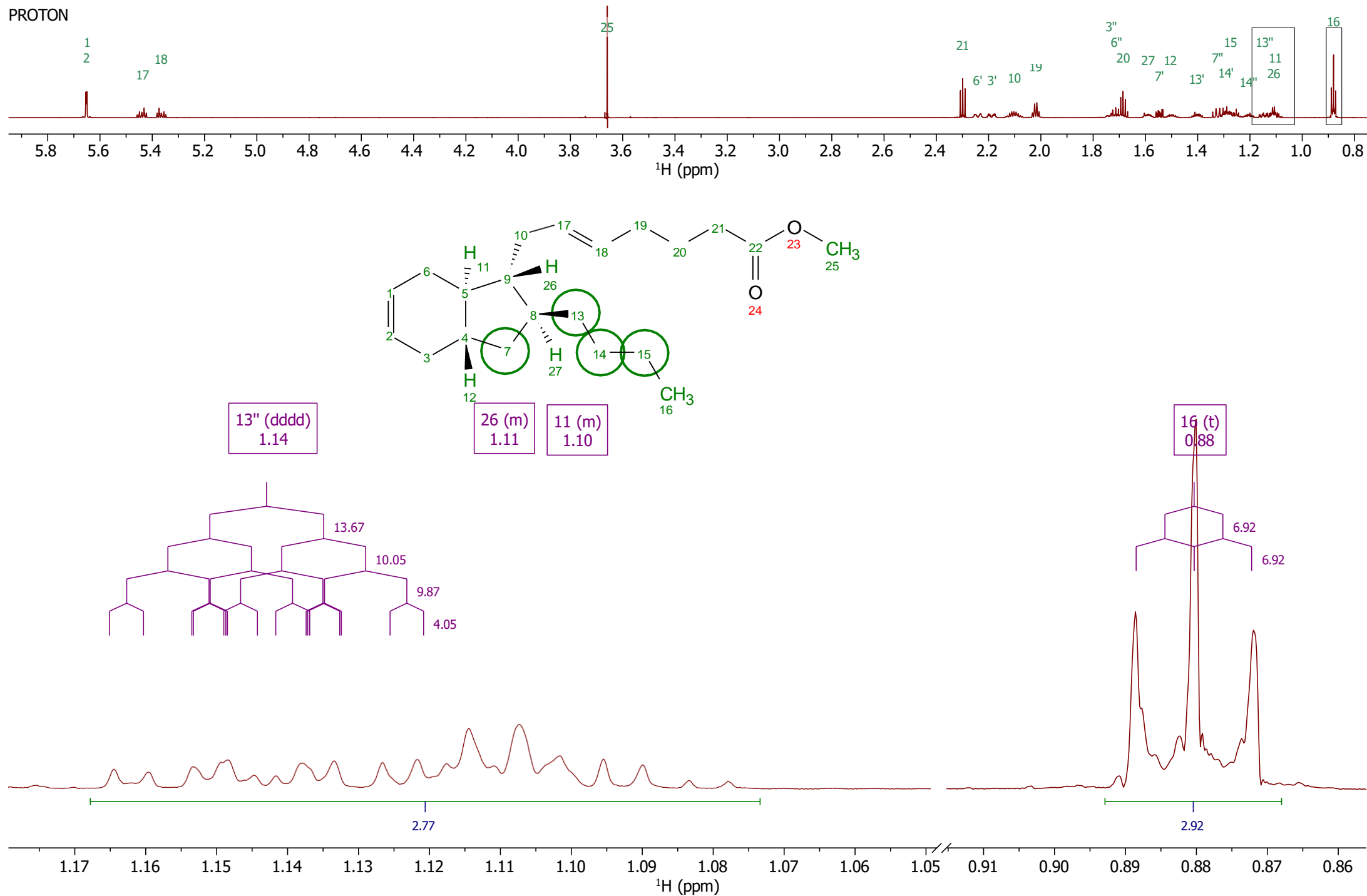


Figure S6: Extract of ^1H NMR of **7** with assignment

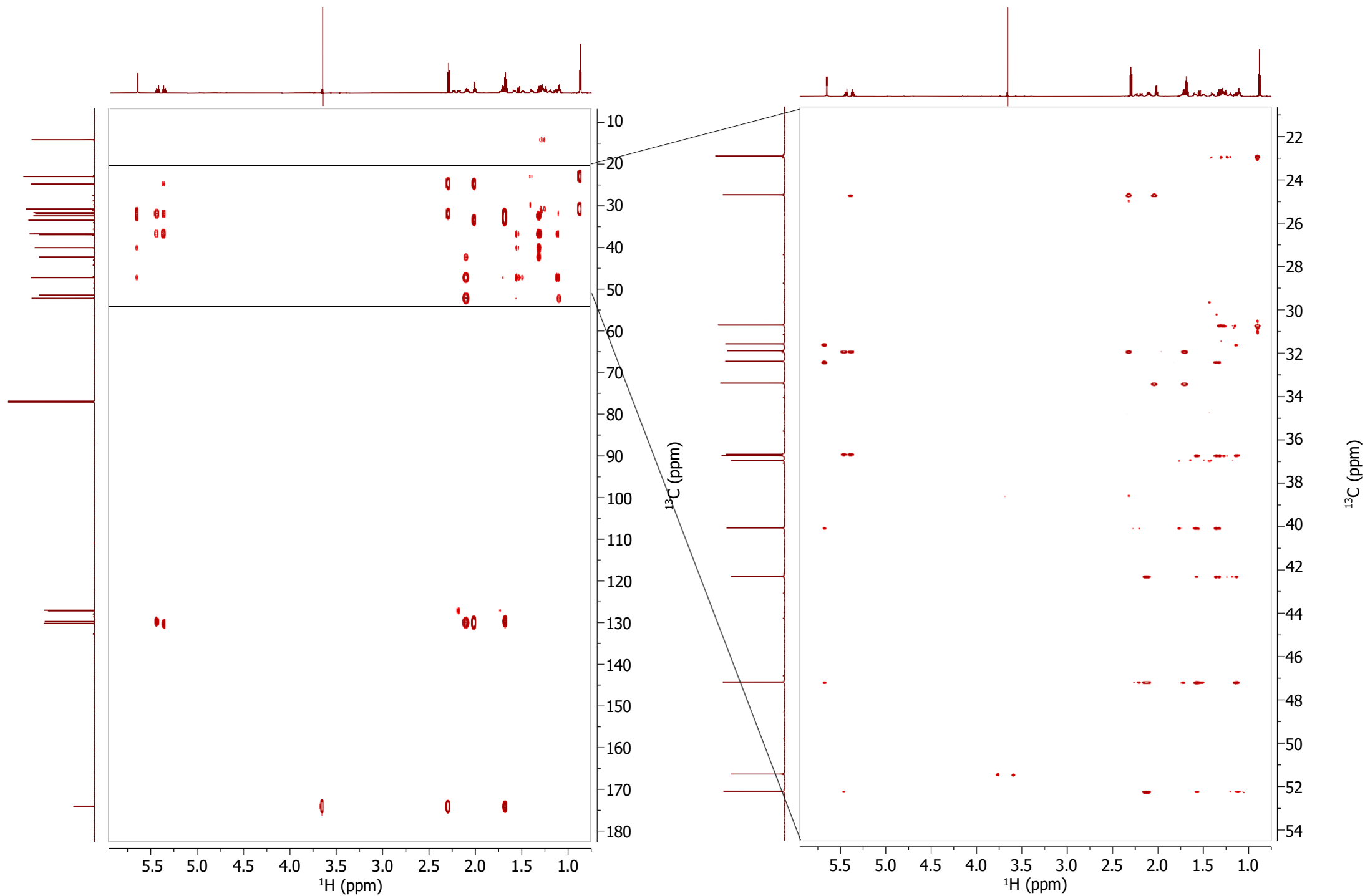
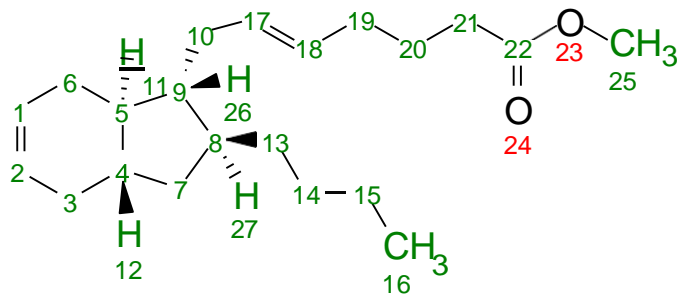


Figure S8: HMBC (left) and selective HMBC (right) of **7**

No	Atom Type	δ_{H} (Multiplicity, J , nH)	δ_{C}
1	CH	5.65 (m, 1H)	127.2
2	CH	5.65 (m, 1H)	127.3
17	CH	5.44 (dtt, 15.3, 7.1, 1.4 Hz, 1H)	130.2
18	CH	5.37	130.0
25	CH ₃	3.66 (s, 3H)	51.4
21	CH ₂	2.30 (t, 7.6 Hz, 2H)	33.4
6'	CH ₂	2.24 (m, 1H)	31.6
3'	CH ₂	2.19 (m, 1H)	32.4
10	CH ₂	2.10 (m, 2H)	36.7
19	CH ₂	2.02 (q, 7.1 Hz, 2H)	31.9
3"	CH ₂	1.73 (m, 1H)	32.4
6"	CH ₂	1.71 (m, 1H)	31.6
20	CH ₂	1.69 (p, 7.5 Hz, 2H)	24.7
8/27	CH	1.59 (m, 1H)	42.3
7'	CH ₂	1.55 (ddd, 12.2, 7.3, 2.9 Hz, 1H)	37.0
4/12	CH	1.50 (m, 1H)	40.1
13'	CH ₂	1.40 (dddd, 12.7, 10.0, 5.1, 5.1 Hz, 1H)	36.7
7"	CH ₂	1.32 (ddd, 12.4, 10.7, 10.7 Hz, 1H)	37.0
14'	CH ₂	1.29 (m, 1H)	30.7
15	CH ₂	1.27 (m, 2H)	22.9
14"	CH ₂	1.21 (m, 1H)	30.7
13"	CH ₂	1.14 (dddd, 13.7, 10.0, 9.9, 4.0 Hz, 1H)	36.7
9/26	CH	1.11 (m, 1H)	52.2
5/11	CH	1.10 (m, 1H)	47.3
16	CH ₃	0.88 (t, 6.9 Hz, 3H)	14.2
22	C	-	174.2



¹H NMR (850 MHz, CDCl₃) δ 5.68 – 5.63 (m, 2H), 5.44 (dtt, J = 15.3, 7.1, 1.4 Hz, 1H), 5.36 (dtt, J = 15.3, 6.7, 1.4 Hz, 1H), 3.66 (s, 3H), 2.30 (t, J = 7.6 Hz, 2H), 2.26 – 2.22 (m, 1H), 2.21 – 2.16 (m, 1H), 2.15 – 2.06 (m, 2H), 2.02 (q, J = 7.1 Hz, 2H), 1.76 – 1.71 (m, 1H), 1.72 – 1.69 (m, 1H), 1.69 (p, J = 7.5 Hz, 2H), 1.61 – 1.57 (m, 1H), 1.55 (ddd, J = 12.2, 7.3, 2.9 Hz, 1H), 1.53 – 1.47 (m, 1H), 1.40 (dddd, J = 12.7, 10.0, 5.1, 5.1 Hz, 1H), 1.32 (ddd, J = 12.4, 10.7, 10.7 Hz, 1H), 1.30 – 1.28 (m, 1H), 1.28 – 1.23 (m, 2H), 1.23 – 1.18 (m, 1H), 1.14 (dddd, J = 13.7, 10.0, 9.9, 4.0 Hz, 1H), 1.12 – 1.10 (m, 1H), 1.12 – 1.08 (m, 1H), 0.88 (t, J = 6.9 Hz, 3H).

¹³C NMR (214 MHz, CDCl₃) δ 174.16, 130.29, 129.80, 127.25, 127.13, 52.23, 51.43, 47.18, 42.31, 40.07, 36.95, 36.73, 36.70, 33.41, 32.40, 31.92, 31.60, 30.73, 24.72, 22.93, 14.15.

Figure S9: Full assignment of **7**