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Supporting Information

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Total Synthesis and Stereochemical Reassignment of (+)-Neopeltolide

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The following data are included as supporting information:

S1: ^{1}H NMR data comparison of natural and synthetic neopeltolide **1a** and **1b**
S2: ^{13}C NMR data comparison of natural and synthetic neopeltolide **1a** and **1b**
S3: ^{1}H NMR spectrum of synthetic neopeltolide **1b**
S4: Expansion of ^{1}H NMR spectrum of synthetic neopeltolide **1b**
S5: ^{13}C NMR spectrum of synthetic neopeltolide **1b**
S6: Expansion of ^{13}C NMR spectrum of synthetic neopeltolide **1b**
S7: ^{1}H NMR spectrum of synthetic neopeltolide **1a**
S8: Expansion of ^{1}H NMR spectrum of synthetic neopeltolide **1a**
S9: ^{13}C NMR spectrum of synthetic neopeltolide **1a**
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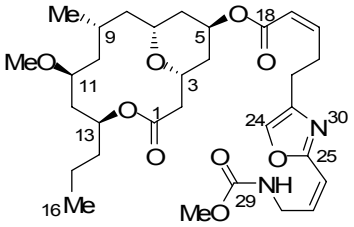
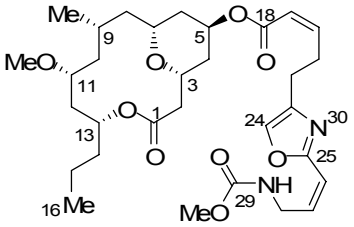
S11: Expansion of ^{1}H NMR spectrum and assignment of synthetic neopeltolide **1b**
S12: Expansion of ^{13}C NMR spectrum and assignment of synthetic neopeltolide **1b**
S13: Expansion of ^{1}H NMR spectrum and assignment of synthetic neopeltolide **1a**
S14: Expansion of ^{13}C NMR spectrum and assignment of synthetic neopeltolide **1a**

^{1}H and ^{13}C NMR spectra were taken in CD_3OD at 400 MHz and 100 MHz, respectively. Chemical shifts are reported in parts per million relative to CD_3OD (^{1}H, δ 3.285; ^{13}C, δ 49.0). Coupling constants (*J* values) are given in Hertz (Hz). ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d= doublet, t = triplet, q = quartet, sept = septuplet, m = multiplet, br = broad), coupling constant, integration.

S1. ^{1}H NMR data for natural and synthetic neopeltolide (reassigned **1b**, original proposed **1a**) (CD_3OD)[a]



neopeltolide, **1b**
11*S*, 13*S*

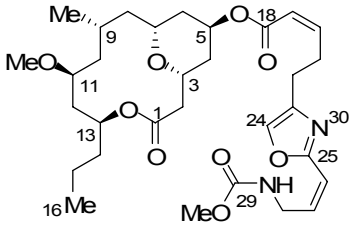


original proposed neopeltolide, **1a**
11*R*, 13*R*

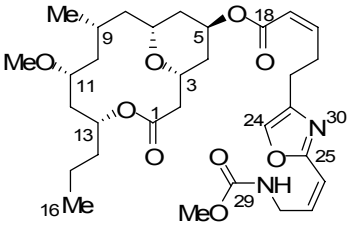
Proton No.	natural neopeltolide[b,c]	synthetic neopeltolide, 1b reassigned[d]	synthetic neopeltolide, 1a original proposed[d]
2a	2.66, dd (15.1, 4.1)	2.68, m	2.40, m
2b	2.26, dd (15.1,11.0)	2.29, dd (14.5, 10.9)	2.26, m
3	4.04, ddt (4.1, 2.1 11.7)	4.07, dddd (4.1, 2.3, 11.2)	4.02 ddt (4.9, 2.5, 11.5)
4a	1.78, m	1.78, dd (14.1, 2.3)	1.79, m
4b	1.46, m	1.46, m	1.45, m
5	5.17, m	5.20, m	5.19, m
6a	1.64, m	1.64, m	1.69, m
6b	1.49, m	1.49, m	1.30, m
7	3.55, bt (10.3)	3.56, t (10.2)	3.69, bt (8.5)
8a	1.36, m	1.36, m	1.36, m
8b	1.25, m	1.25, m	1.25, m
9	1.38, m	1.38, m	1.24, m
10a	1.54, m	1.54, m	2.28, m
10b	1.08, m	1.08, m	1.08, m
11	3.64, m	3.66, m	3.23, m
OCH_3-11	3.23, s	3.27, s	3.30, s
12a	1.83, m	1.83, m	1.83, m
12b	1.28, m	1.28, m	1.49, m
13	5.14, dt (4, 8, 9.6)	5.15, m	5.24, m
14a	1.68, m	1.68, m	1.55, bddd (2.2, 4.2, 14.0)
14b	1.48, m	1.48, m	1.48, m
15	1.33, m	1.33, m	1.33, m
16	0.92, t (7.6)	0.92, t (7.4)	0.92, t (7.4)
17	0.94, d (6.9)	0.94, d (7.2)	0.94, d (7.2)
19	5.86, dt (11.7, 1.4)	5.87, dt (11.3, 1.76)	5.86, dt (11.6, 1.5)
20	6.33, dt (11.7, 7.6)	6.36, dt (11.6, 7.8)	6.37, dt (11.2, 7.6)
21	2.98, m	3.00, m	2.99, dddd (1.5, 7.5, 7.9, 15.5)
22	2.68, dd (7.6, 7.6)	2.70, m	2.69, t (7.5)
24	7.64, s	7.67, s	7.67, s
26	6.24, dt (11.7, 2.1)	6.27, ddd (11.9, 2.15, 1.95)	6.26, dt (11.6, 1.6)
27	6.02, dt, (11.7, 6.2)	6.04, dt (12.0, 5.9)	6.03, dt (12, 6.4)
28	4.287, bd, (4.8)	4.31, dd (5.86, 1.2)	4.29, dd (5.9, 1.5)
OCH_3-29	3.62, s	3.65, s	3.64, s

[a] Data recorded in order: chemical shift (δ in ppm), (multiplicity, coupling constant in Hertz). [b] A. E. Wright, J. C. Botelho, E. Guzmán, D. Harmody, P. Linley, P. J. McCarthy, T. P. Pitts, S. A. Pomponi, J. K. Reed, *J. Nat. Prod*. **2007**, *70*, 412-416. [c] Recorded at 600 MHz. [d] Recorded at 400 MHz.

S2. ^{13}C NMR data for natural and synthetic neopeltolide (reassigned **1b**, original proposed **1a**) (CD_3OD)[a]



neopeltolide, **1b**
11*S*, 13*S*

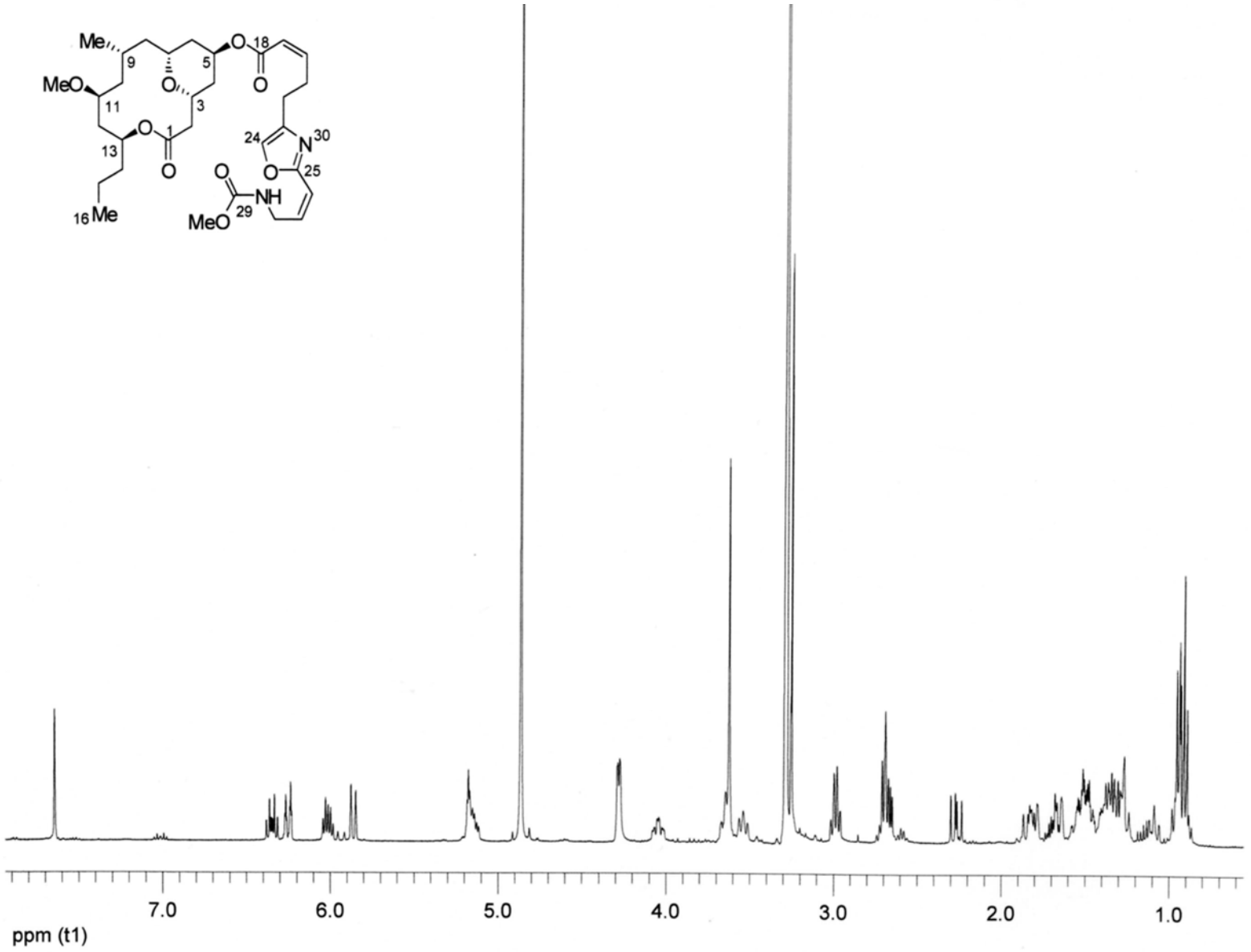


original proposed neopeltolide, **1a**
11*R*, 13*R*

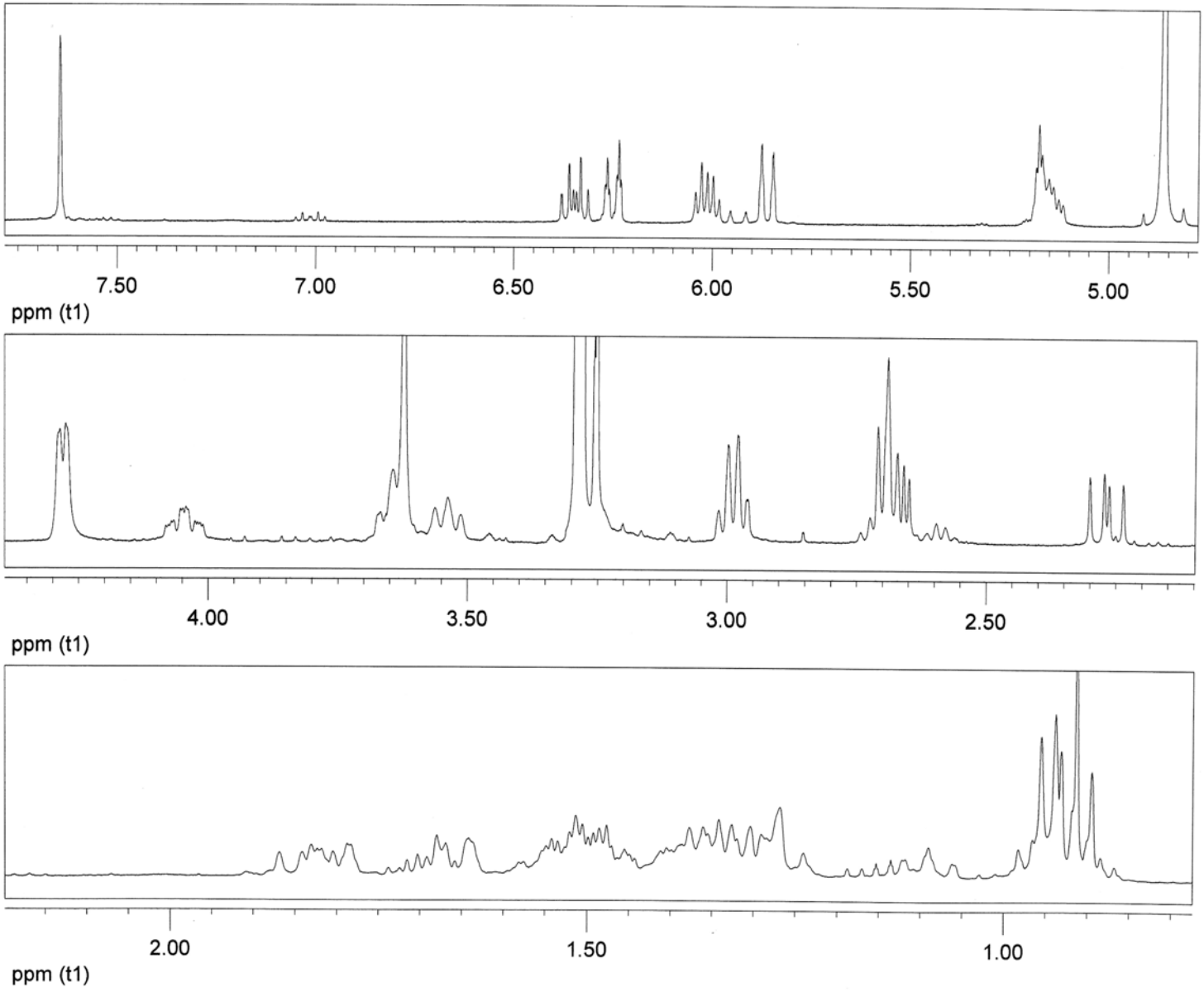
Carbon No.	natural neopeltolide[b,c]	synthetic neopeltolide 1b reassigned[d]	synthetic neopeltolide 1a original proposed[d]
1	173.0	173.0	174.7
2	43.2	43.2	43.2
3	71.3	71.3	72.8
4	36.2	36.2	36.2
5	69.2	69.2	69.1
6	37.4	37.4	38.8
7	77.0	77.0	76.5
8	45.2	45.2	45.6
9	32.6	32.6	35.6
10	43.5	43.5	46.0
11	77.1	77.1	84.5
OCH_3-11	56.4	56.4	56.6
12	41.0	41.0	41.3
13	73.9	73.9	75.7
14	37.9	37.9	37.1
15	20.0	20.0	19.7
16	14.1	14.1	14.2
17	26.0	26.0	25.2
18	166.9	166.8	166.9
19	121.7	121.7	121.7
20	150.0	150.0	150.1
21	29.0	29.0	29.0
22	26.4	26.4	26.4
23	142.3	142.3	142.3
24	135.9	135.9	136.0
25	161.9	161.9	161.9
26	115.7	115.9	116.0
27	139.2	139.2	139.2
28	41.0	41.0	41.0
29	159.6	159.6	159.5
OCH_3-29	52.6	52.6	52.6

[a] Data recorded: chemical shift (δ in ppm). [b] A. E. Wright, J. C. Botelho, E. Guzmán, D. Harmody, P. Linley, P. J. McCarthy, T. P. Pitts, S. A. Pomponi, J. K. Reed, *J. Nat. Prod*. **2007**, *70*, 412-416. [c] Recorded at 150 MHz. [d] Recorded at 100 MHz.

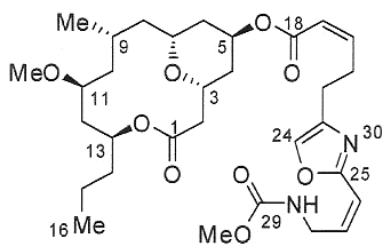
S3. ^{1}H NMR spectrum of synthetic neopeltolide (**1b**), 400 MHz, CD_3OD



S4. Expansion of ^{1}H NMR spectrum of synthetic neopeltolide (**1b**), 400 MHz, CD_3OD

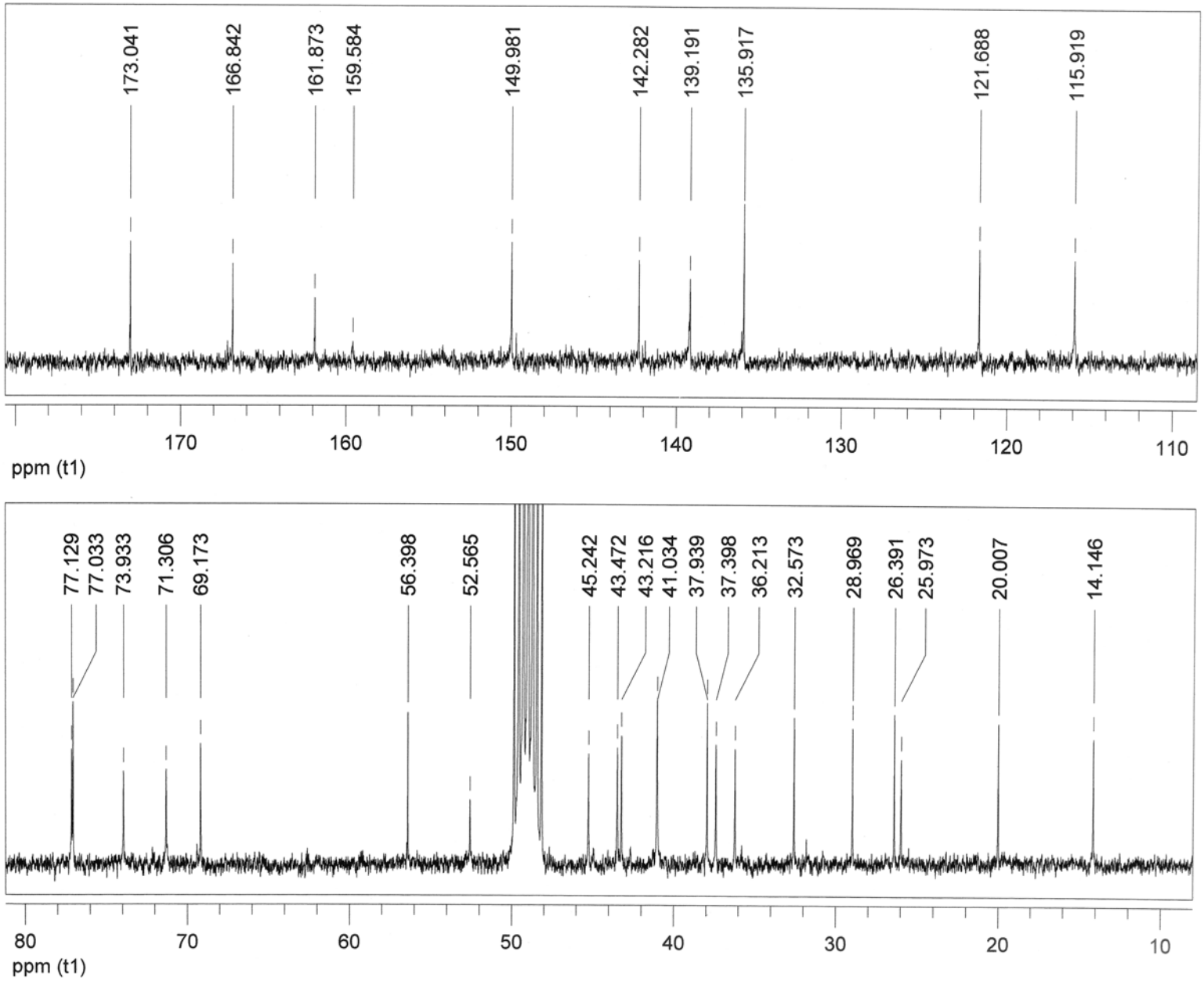


S5. ^{13}C NMR spectrum of synthetic neopeltolide (**1b**), 100 MHz, CD_3OD

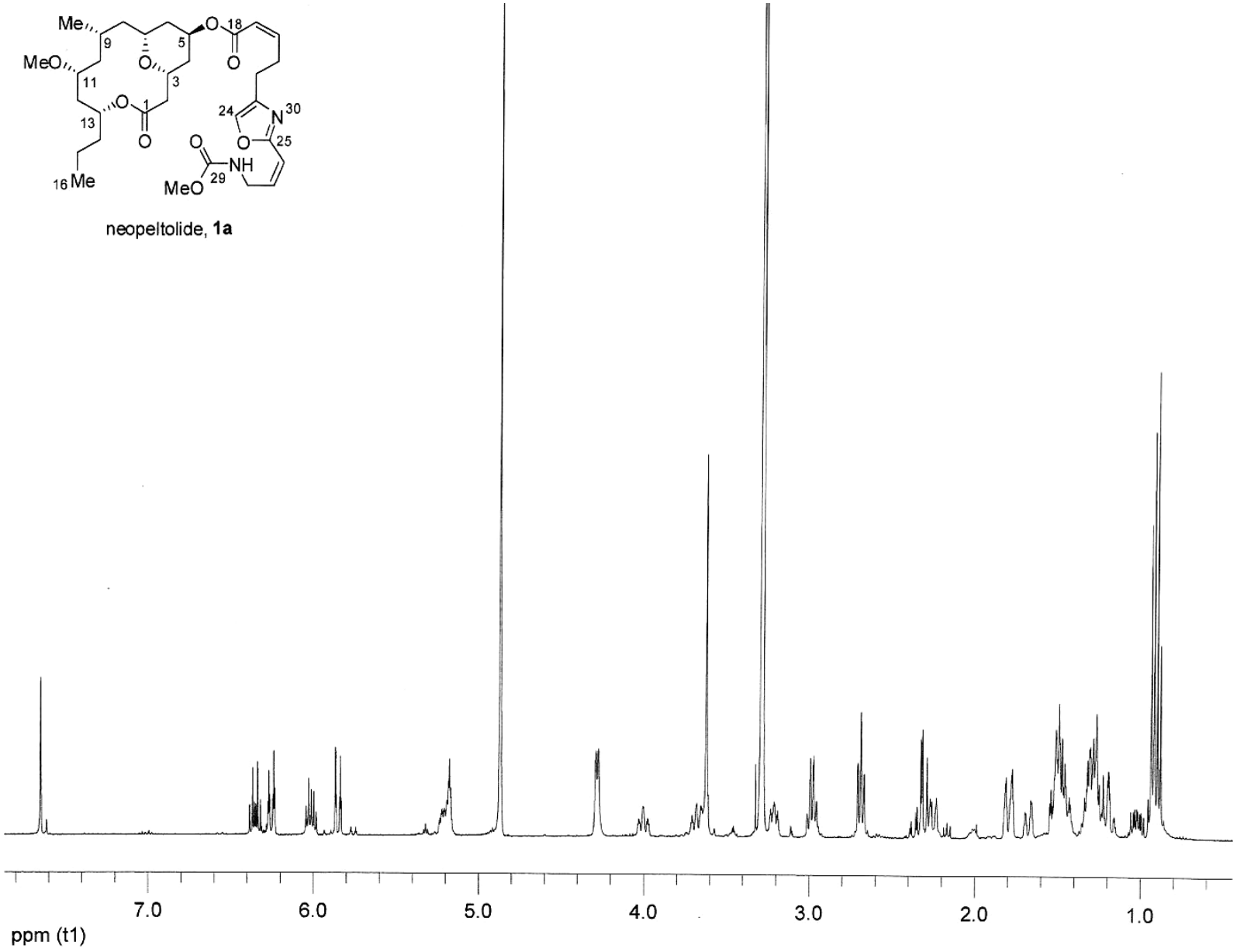


neopeltolide, **1b**

S6. Expansion of ^{13}C NMR spectrum of synthetic neopeltolide (**1b**), 100 MHz, CD_3OD

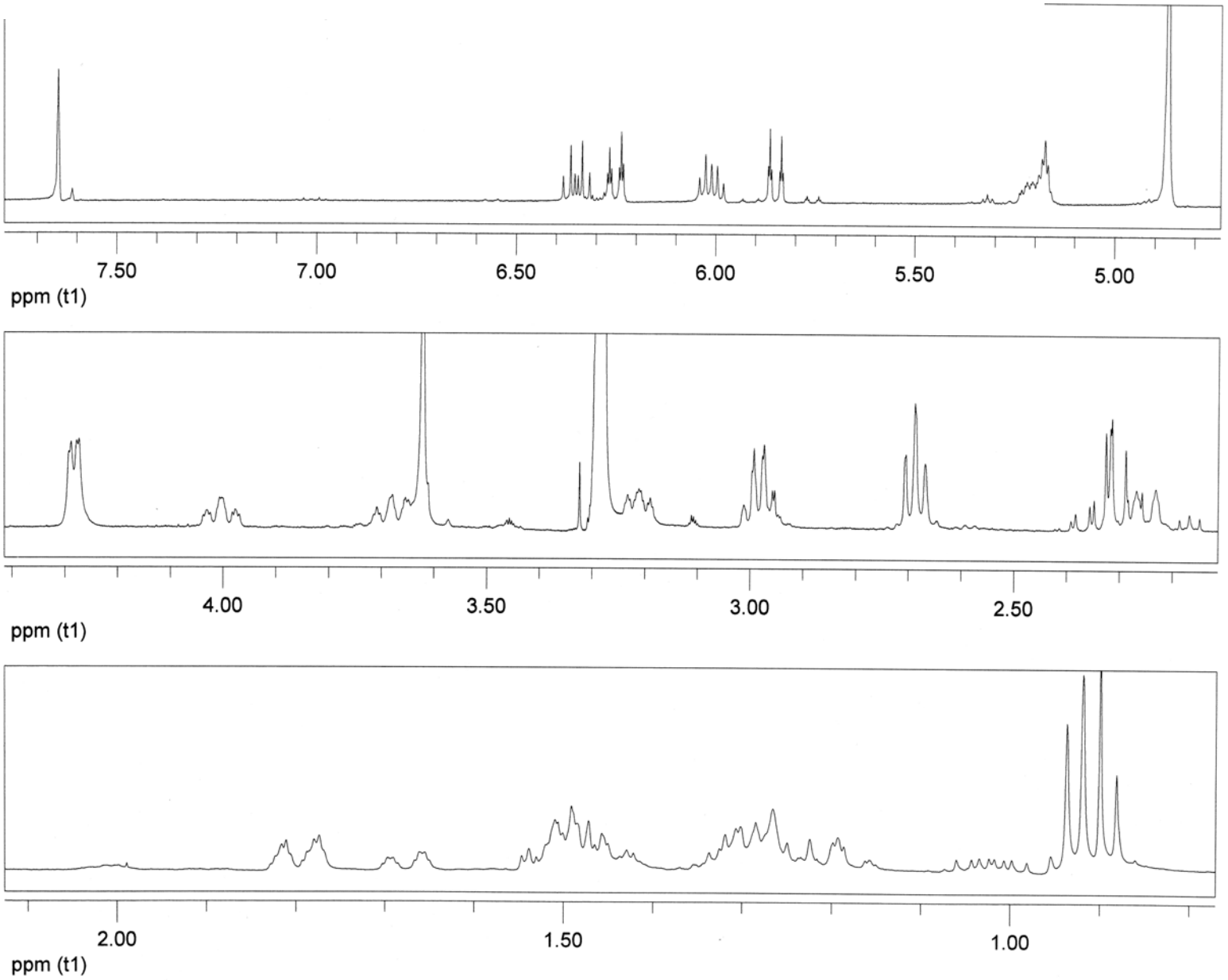


S7. ^{1}H NMR spectrum of synthetic neopeltolide (**1a**), 400 MHz, CD_3OD

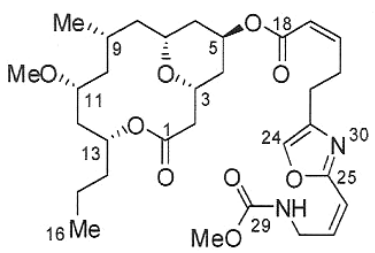


neopeltolide, **1a**

S8. Expansion of ^{1}H NMR spectrum of synthetic neopeltolide (**1a**), 400 MHz, CD_3OD

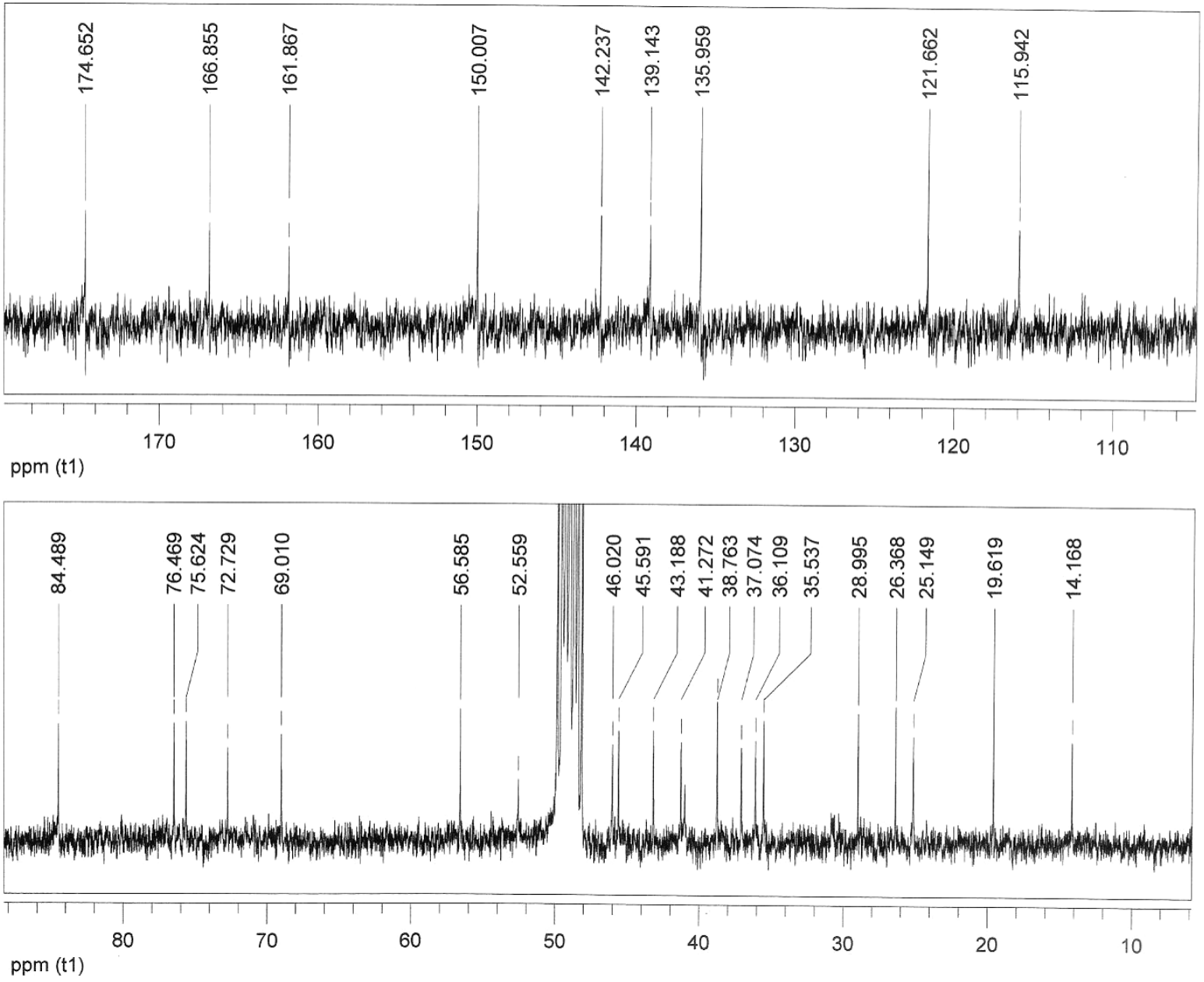


S9. ^{13}C NMR spectrum of synthetic neopeltolide (**1a**), 100 MHz, CD_3OD

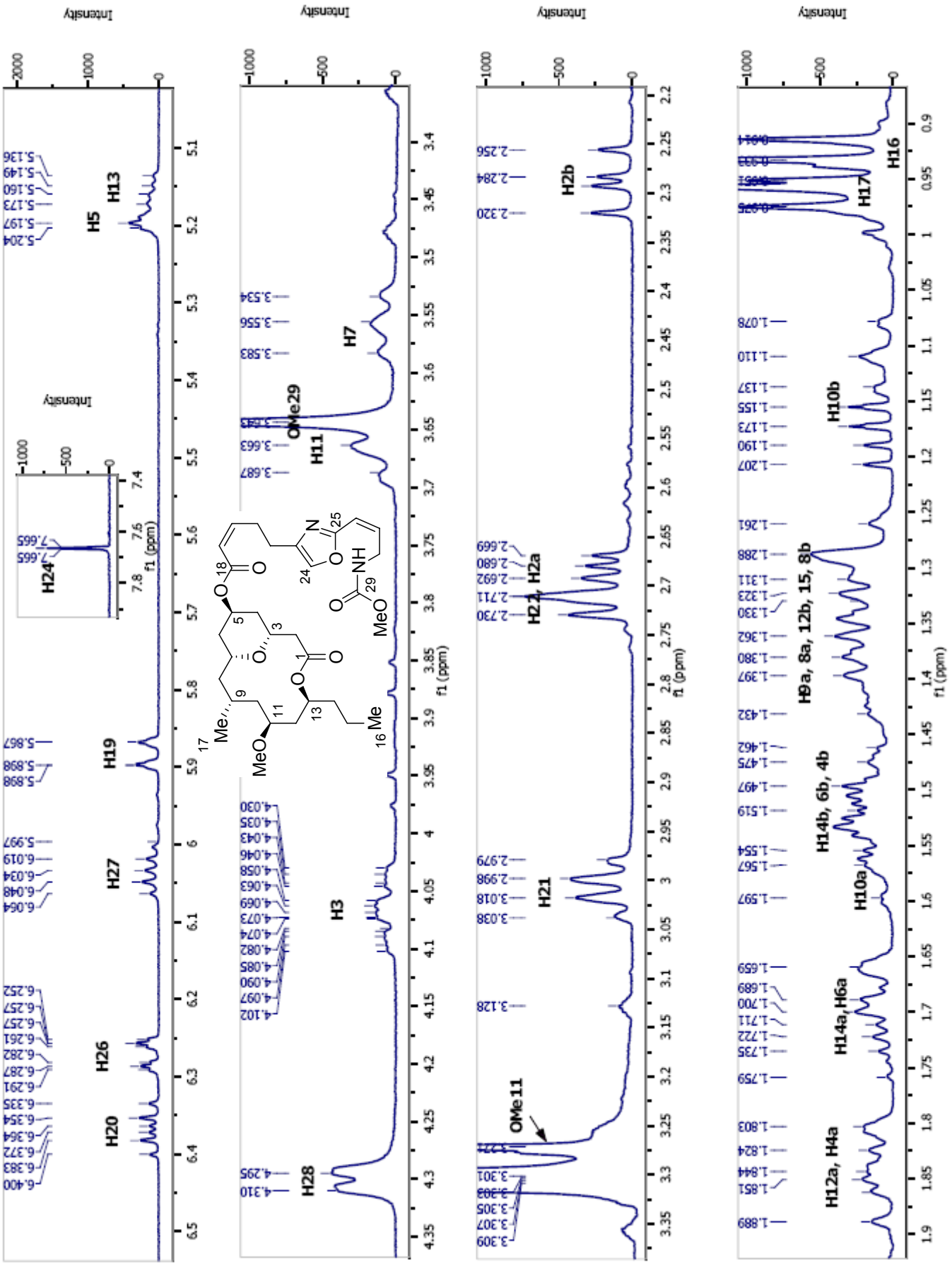


neopeltolide, **1a**

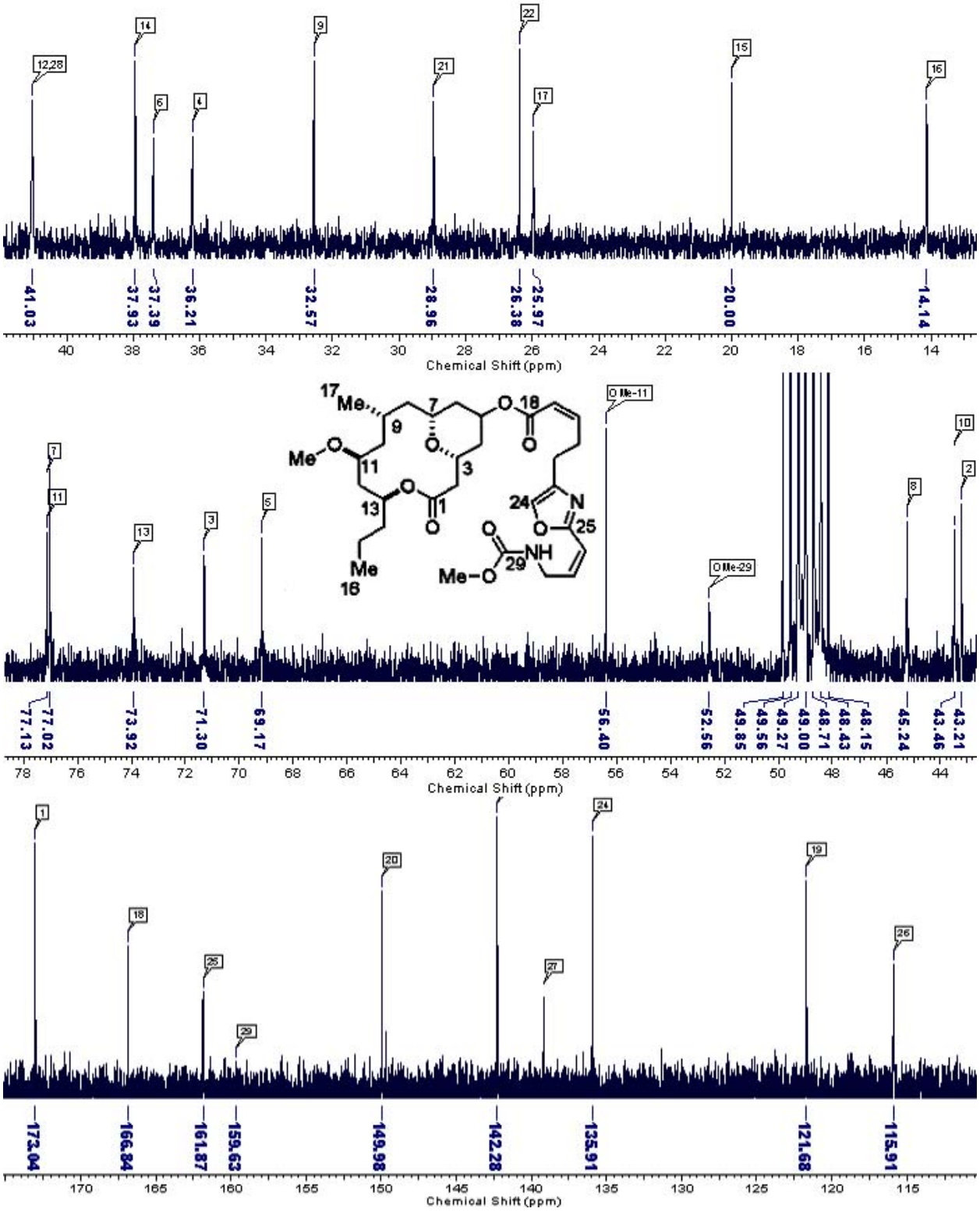
S10. Expansion of ^{13}C NMR spectrum of synthetic neopeltolide (**1a**), 100 MHz, CD_3OD



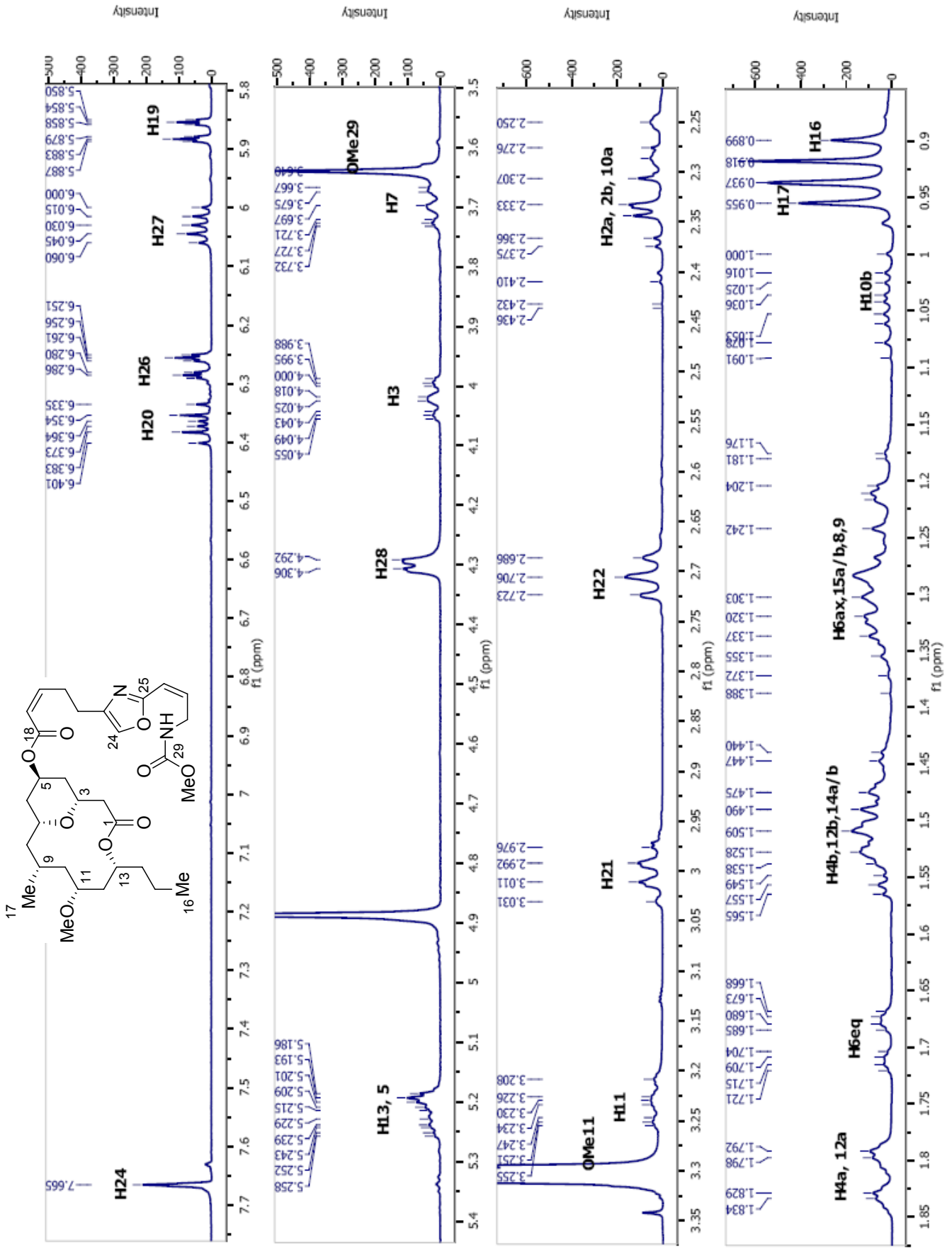
S11. Expansion and assignment of ^{1}H NMR of synthetic neopeltolide **1b**, 400 MHz, CD_3OD



S12. Expansion and assignment of ^{13}C NMR of synthetic neopeltolide **1b**, 100 MHz, CD_3OD



S13. Expansion and assignment of ^{1}H NMR of synthetic neopeltolide **1a**, 400 MHz, CD_3OD



S14. Expansion and assignment of ^{13}C NMR of synthetic neopeltolide **1a**, 100 MHz, CD_3OD