

Practical Isolation of Polygodial from *Tasmannia lanceolata*: a Viable Scaffold for Synthesis

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Supporting Information

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I. Supplementary Figures and Tables



Figure S1. PHWE extract (left) and isolated polygodial (right).

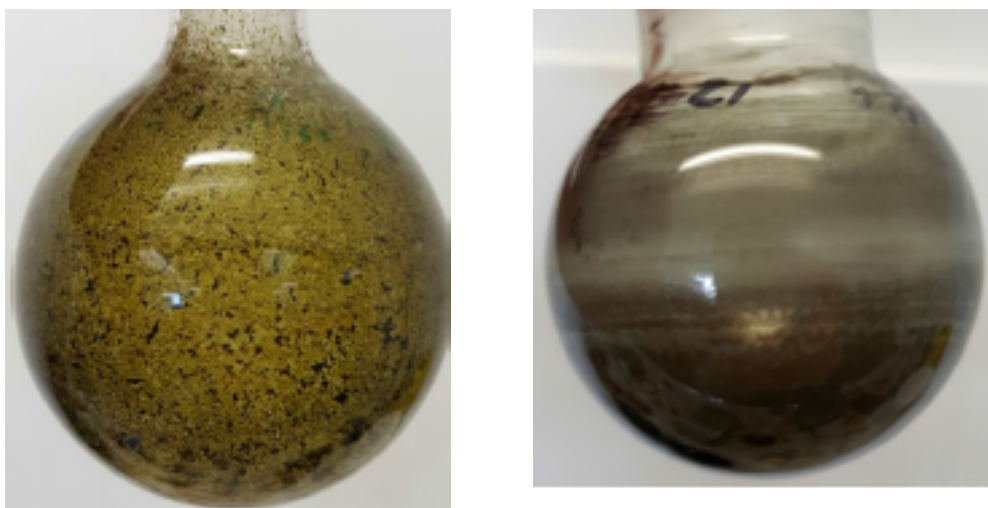


Figure S2. Maceration extract (left) and polygodial isolated after chromatography from maceration (right) (Table 1, entry 7).

Table S1. Effect of temperature and solvent composition on polygodial yield (500 psi).

Yield, % w/w ^[a]		EtOH/H ₂ O (% v/v)		
		15	25	35
Temp. (°C)	70	0.11 (0.12)	0.19 (0.20)	0.42 (0.45)
	90	0.15 (0.17)	0.29 (0.31)	0.46 (0.49)
	110	0.13 (0.17)	0.30 (0.35)	0.42 (0.46)
	130	0.15 (0.24)	0.18 (0.25)	0.36 (0.46)

[a] Value in parentheses refers to the combined yield of polygodial and epi-polygodial.

Table S2. Effect of temperature and solvent composition on polygodial yield (1500 psi).

Yield, % w/w ^[a]		EtOH/H ₂ O (% v/v)			
		0	15	25	35
Temp. (°C)	70	-	0.10 (0.11)	0.18 (0.19)	0.43 (0.46)
	90	-	0.14 (0.15)	0.28 (0.30)	0.46 (0.49)
	110	-	0.12 (0.15)	0.25 (0.29)	0.46 (0.51)
	130	-	0.15 (0.23)	0.27 (0.37)	0.21 (0.26)
	150	0.04 (0.08)	-	-	-

[a] Value in parentheses refers to the combined yield of polygodial and epi-polygodial.

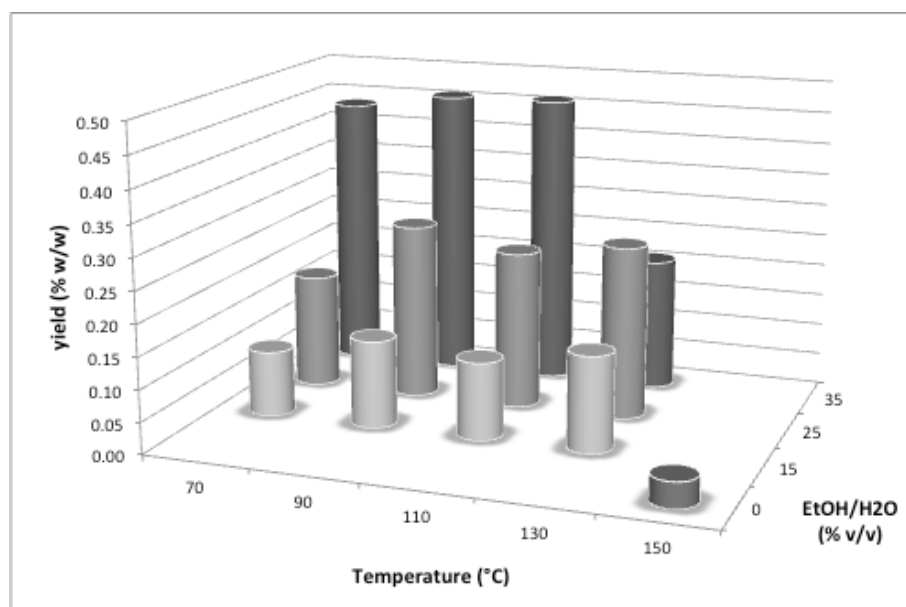
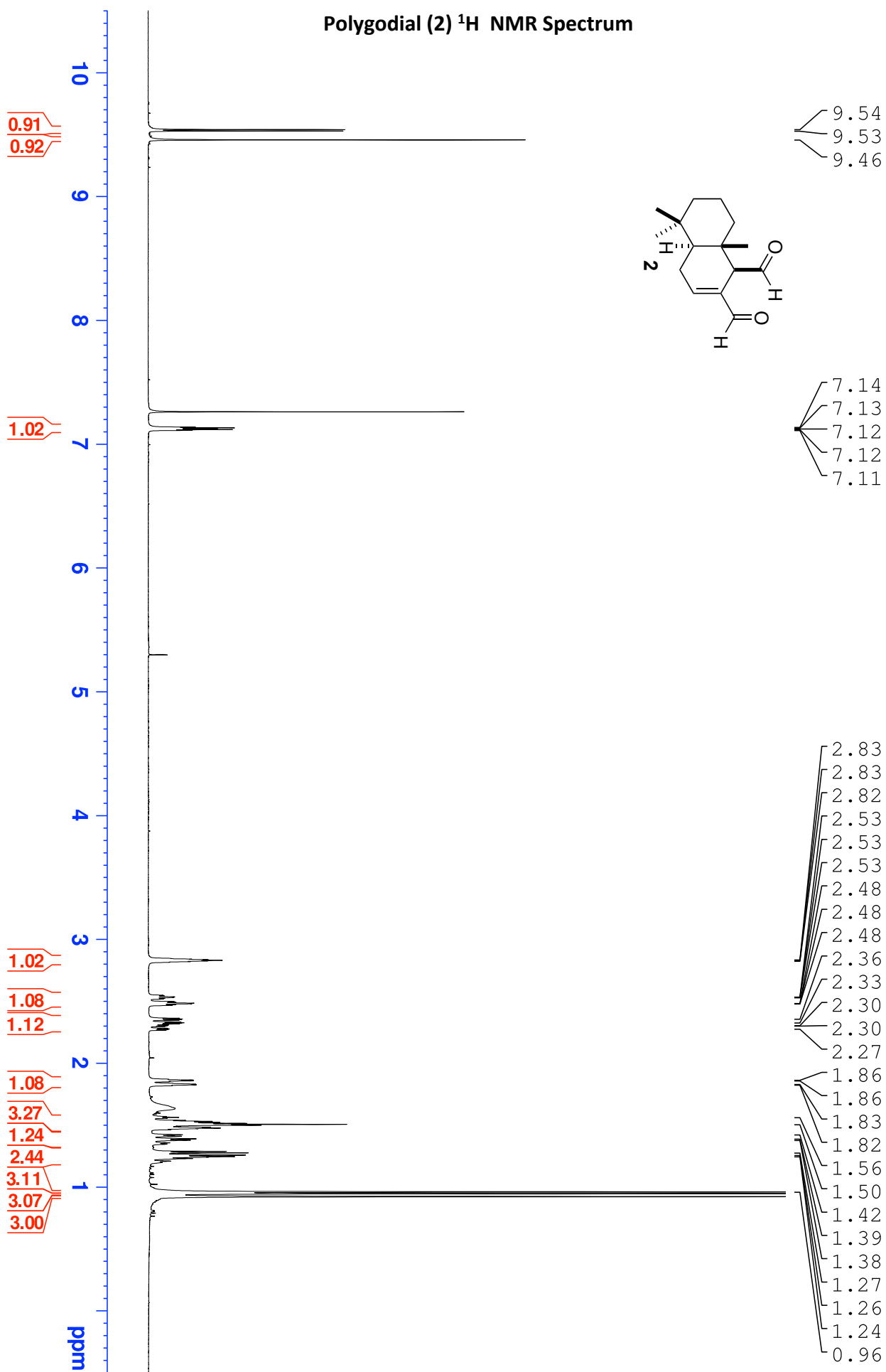
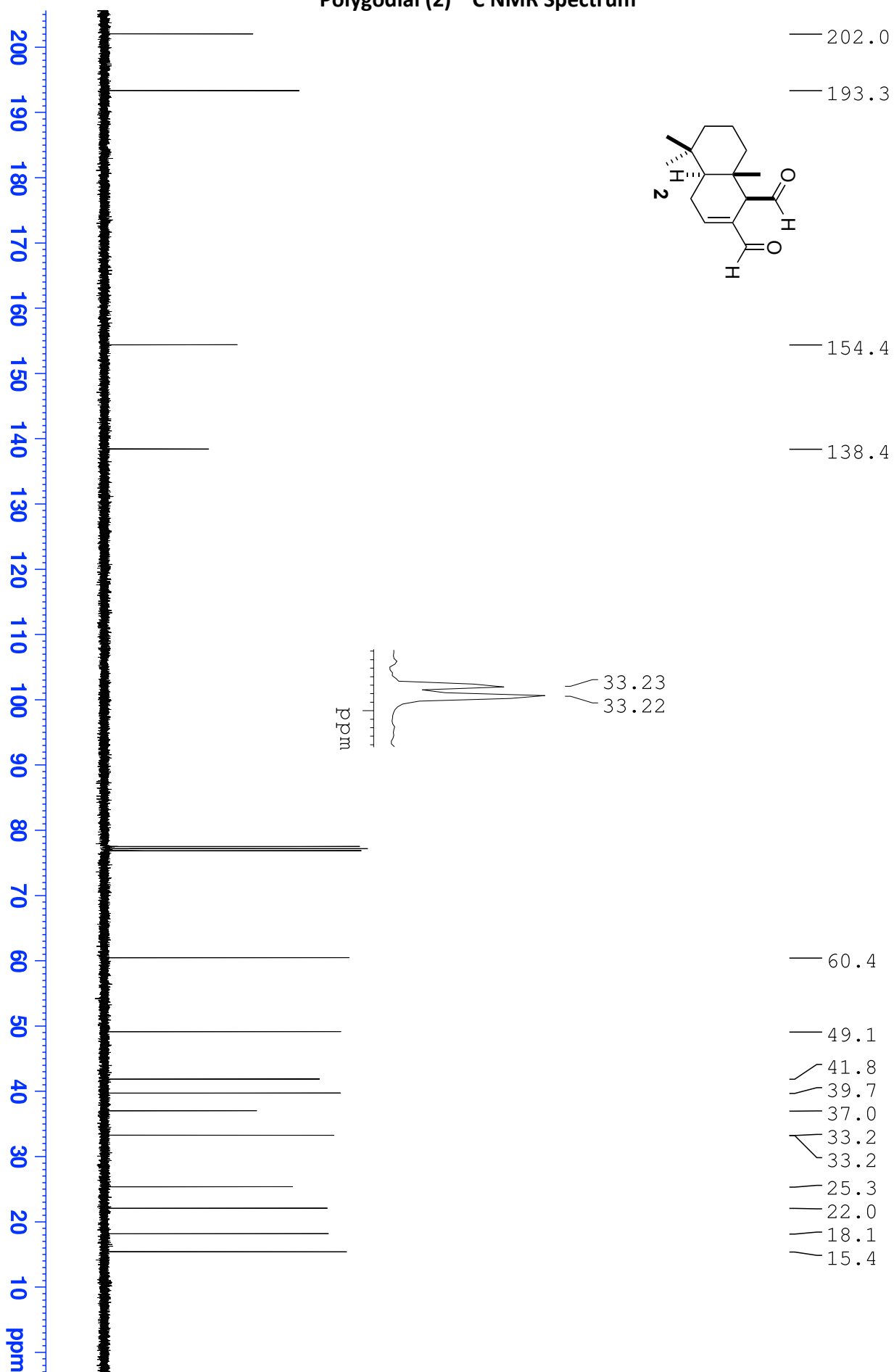


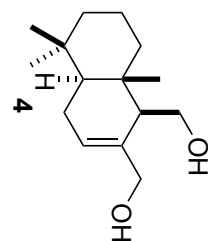
Figure S3. Effect of temperature and solvent composition on polygodial yield. Experiments were performed using a Dionex ASE200 at 1500 psi (~35 bar). Yields were determined *via* ¹H NMR spectroscopy with the aid of an internal standard.

polygodial CDCl₃

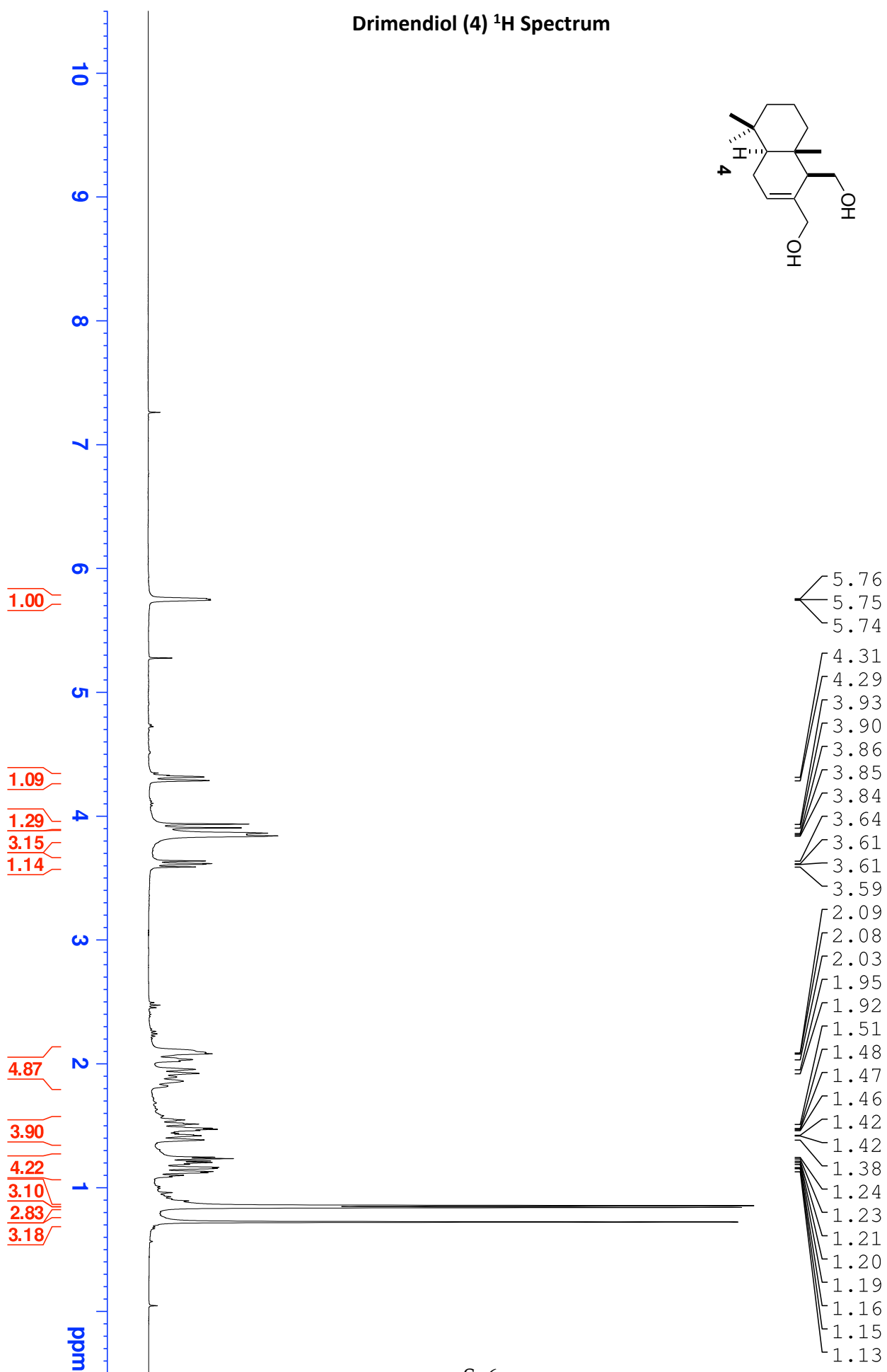


Polygodial (2) ¹³C NMR Spectrum

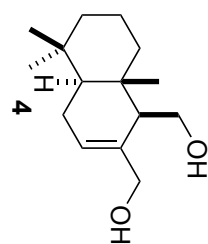
drimendiol CDCl₃



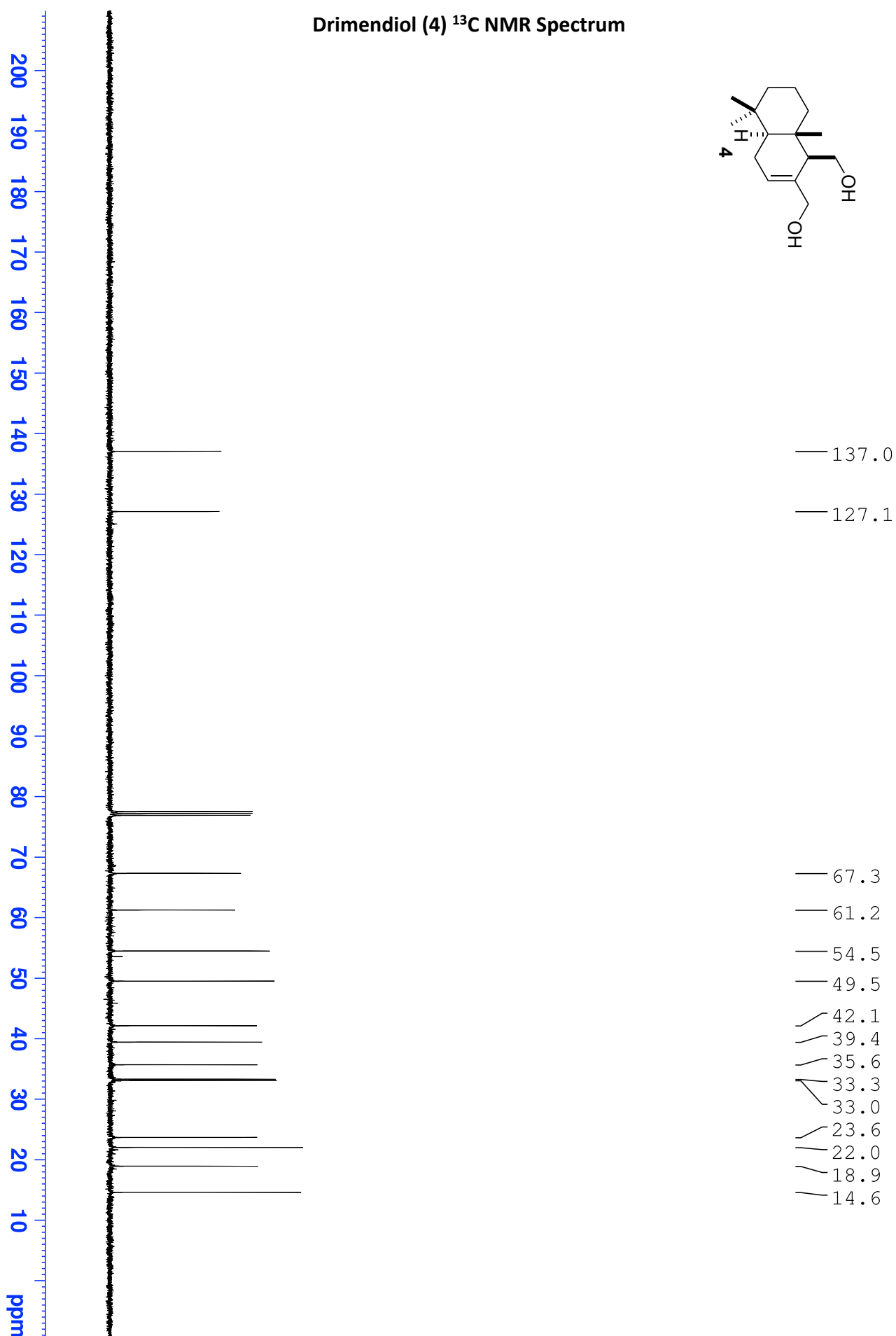
Drimendiol (4) ¹H Spectrum



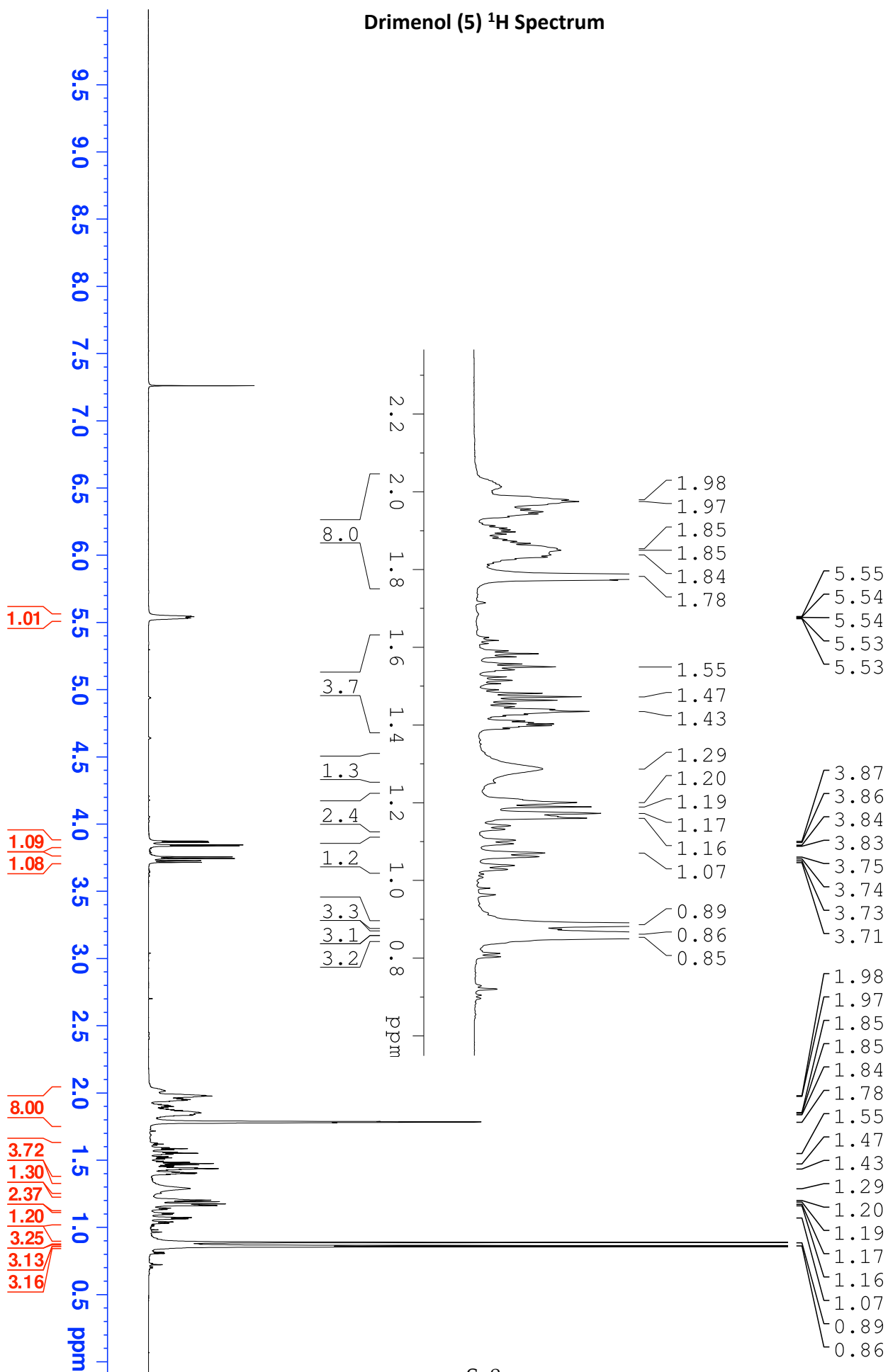
drimendiol 13C CDCl3



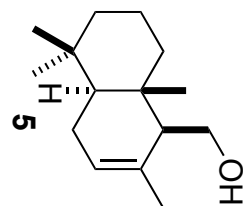
Drimendiol (4) ^{13}C NMR Spectrum



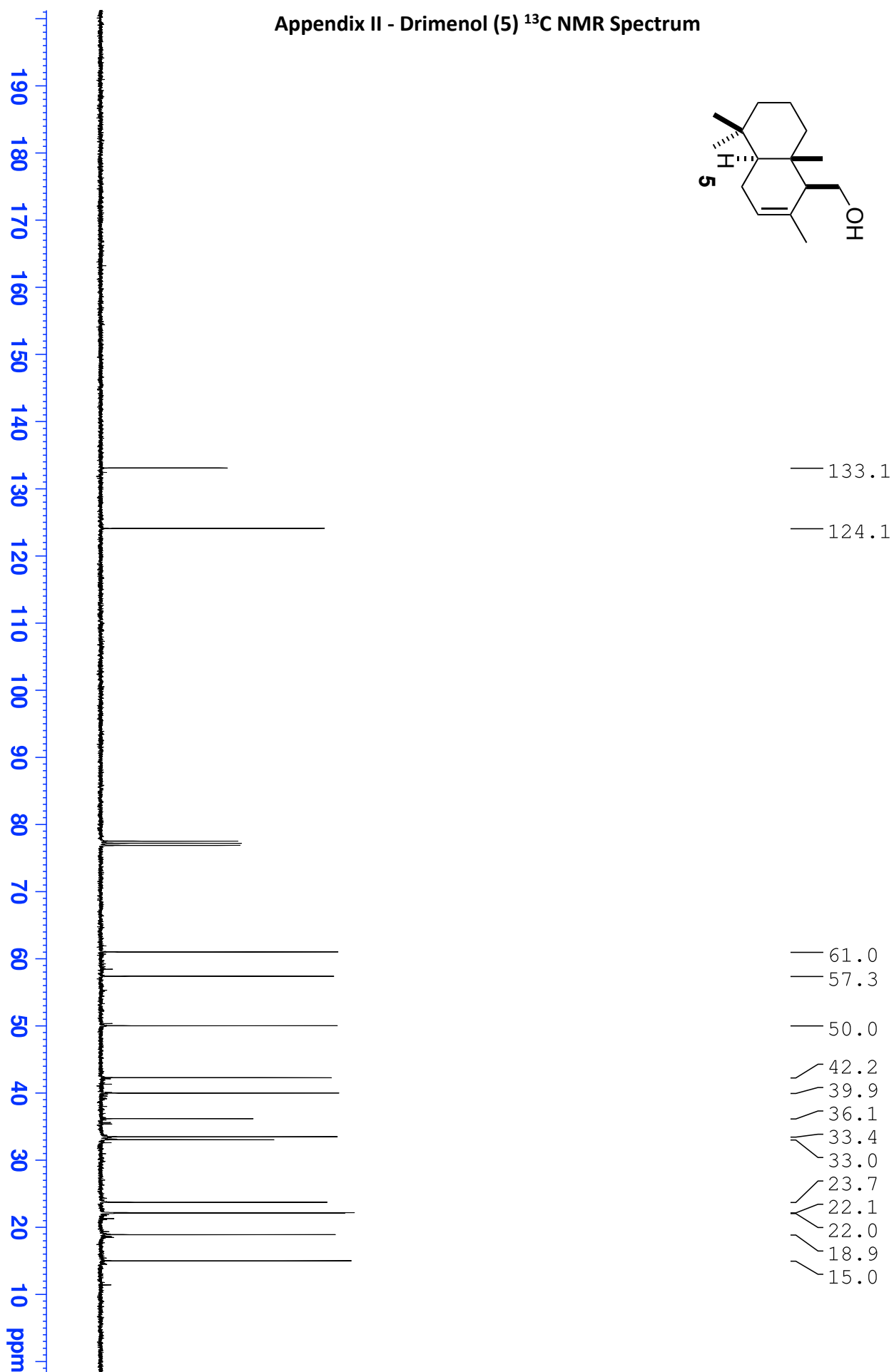
Drimenol (5) ¹H Spectrum



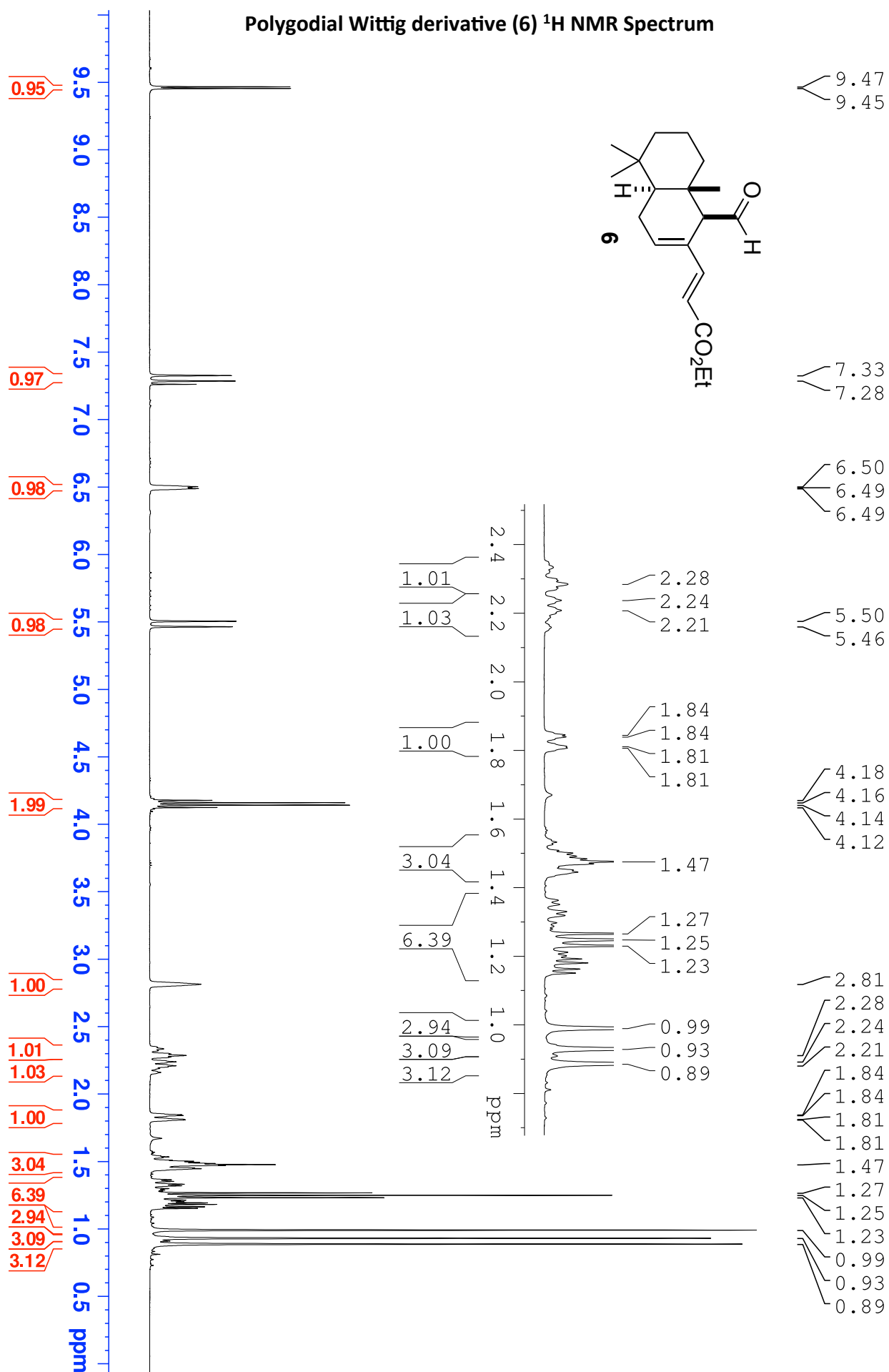
drimenol
13C CDCl3



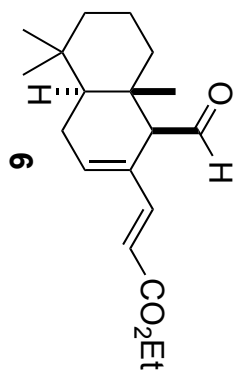
Appendix II - Drimenol (5) ¹³C NMR Spectrum



Polygodial wittig product
¹H CDCl₃



Polygodial wittig product
¹³C CDCl₃



— 205.4

— 167.1

— 146.4

— 141.5

— 130.5

— 116.7

— 62.9

— 60.5

— 48.8

— 41.9

— 40.3

— 37.5

— 33.3

— 33.2

— 25.0

— 22.4

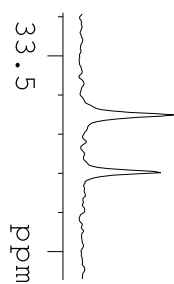
— 18.2

— 15.6

— 14.4

— 33.35

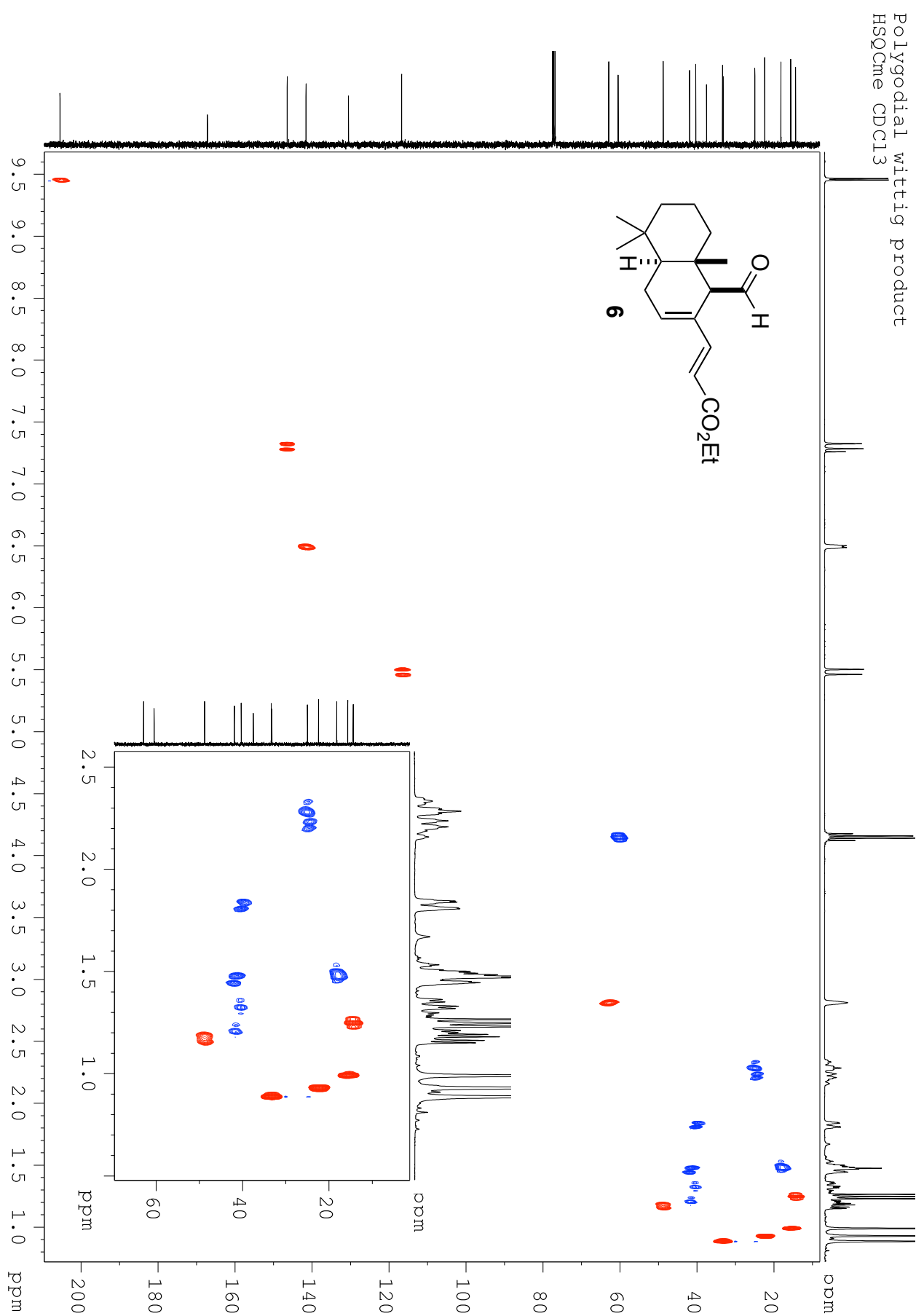
— 33.20



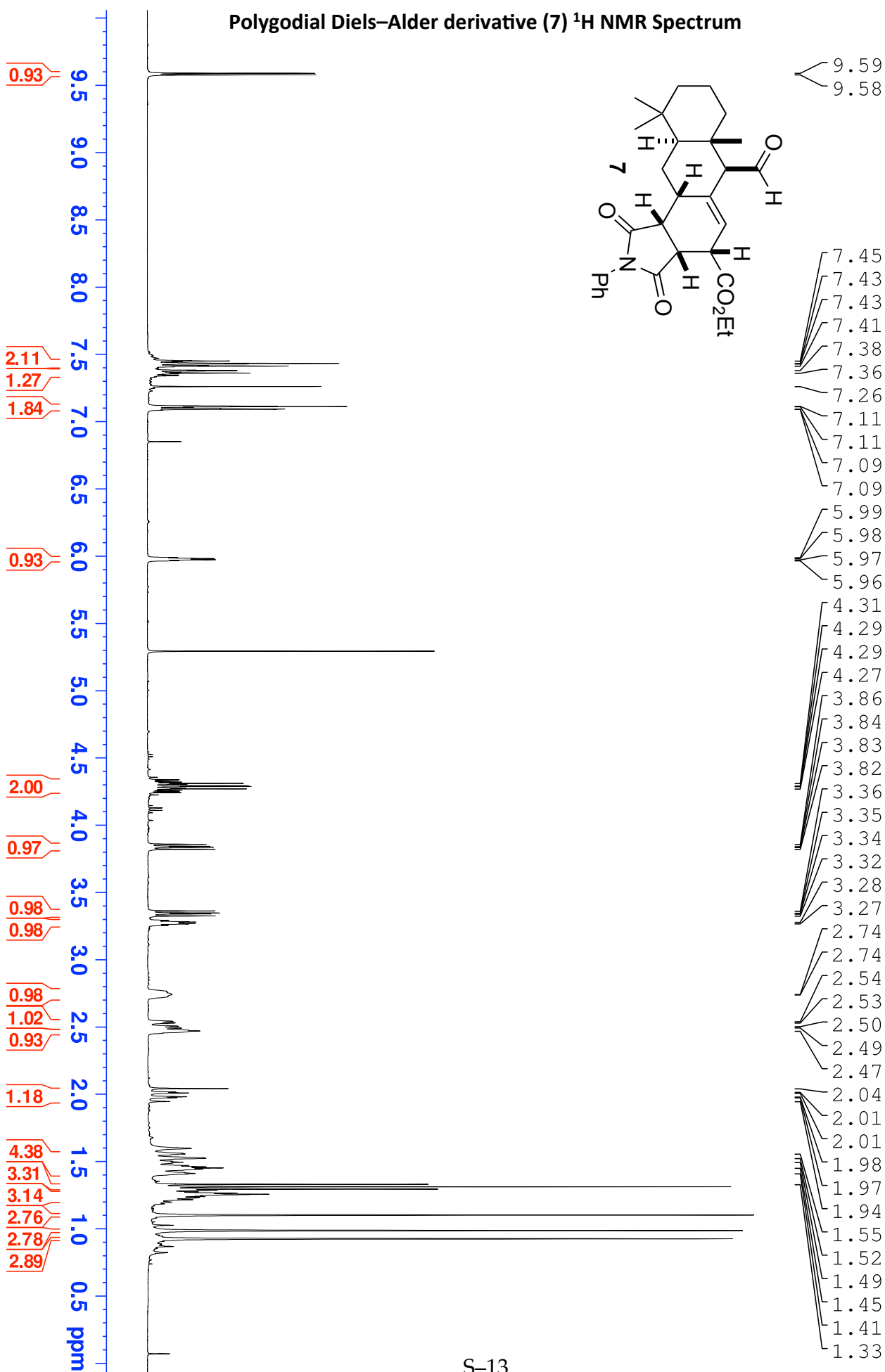
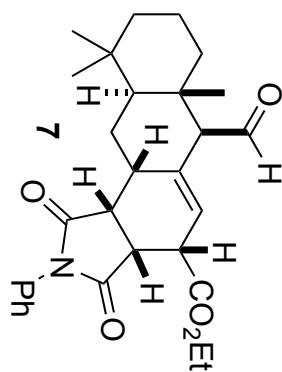
Polygodial Wittig derivative (6) ¹³C NMR Spectrum

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Polygodial Wittig derivative (6) ^1H - ^{13}C HSQCme NMR Spectrum

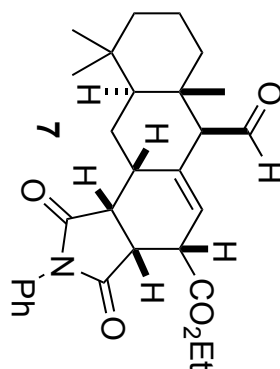


Diels-Alder product
¹H CDCl₃



Diels-Alder product
¹³C CDCl₃

Polygodial Diels-Alder derivative (7) ¹³C NMR Spectrum



— 202.5

— 176.6

— 175.7

— 170.2

— 137.1

— 131.6

— 129.4

— 129.0

— 126.7

— 123.5

— 67.3

— 61.6

— 48.5

— 45.7

— 43.6

— 42.1

— 41.8

— 40.1

— 36.8

— 33.7

— 33.6

— 32.7

— 22.4

— 21.9

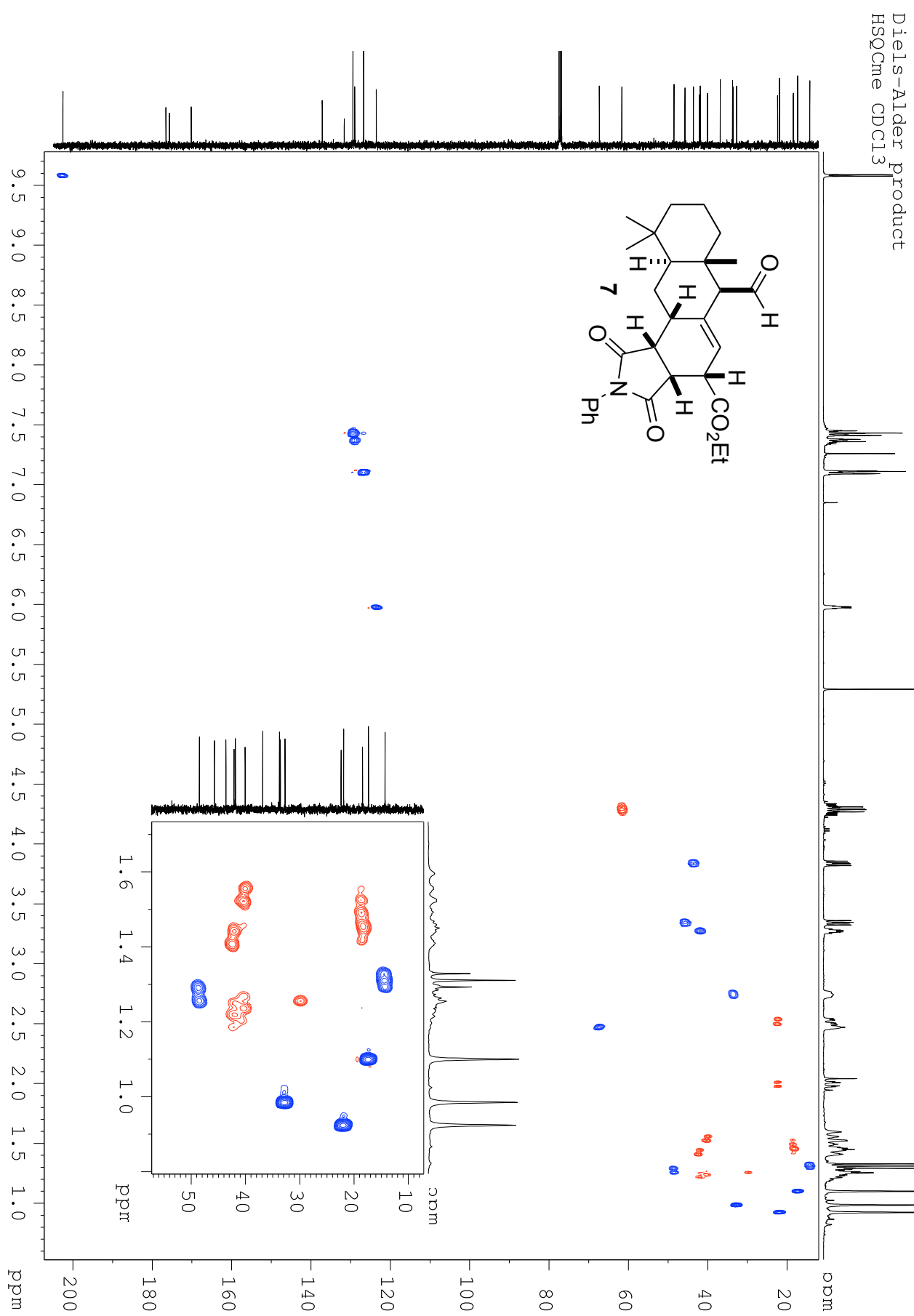
— 18.4

— 17.3

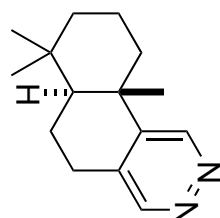
— 14.3

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Polygodial Diels–Alder derivative (7) ^1H - ^{13}C HSQCme NMR Spectrum



polygodial pyridazine derivative
¹H CDCl₃



8.99
 8.80

2.94
 2.92
 2.90
 2.88
 2.83
 2.81
 2.80
 2.78
 2.76
 2.76
 2.74
 2.35
 2.32
 2.02
 2.00
 1.99
 1.97
 1.80
 1.76
 1.69
 1.55
 1.51
 1.44
 1.43
 1.41
 1.40
 1.38
 1.37

2.94
 2.92
 2.90
 2.88
 2.83
 2.81
 2.80
 2.78
 2.76
 2.76
 2.74
 2.35
 2.32
 2.02
 2.00
 1.99
 1.97
 1.80
 1.76
 1.69
 1.55
 1.51
 1.44
 1.43
 1.41
 1.40
 1.38
 1.37
 1.29
 1.28
 1.26
 1.25
 1.21
 0.97
 0.94

3.2
 3.0
 2.8
 2.6
 2.4
 2.2
 2.0
 1.8
 1.6
 1.4
 1.2
 1.0
 0.8
 ppm

0.96
 1.00
 1.00
 1.03
 3.24
 1.13
 1.18
 5.30
 2.94
 2.90

Polygodial pyridazine derivative (8) ¹H NMR Spectrum

0.89
 0.88

0.96
 1.00

1.00

1.03

3.24

1.13

1.18

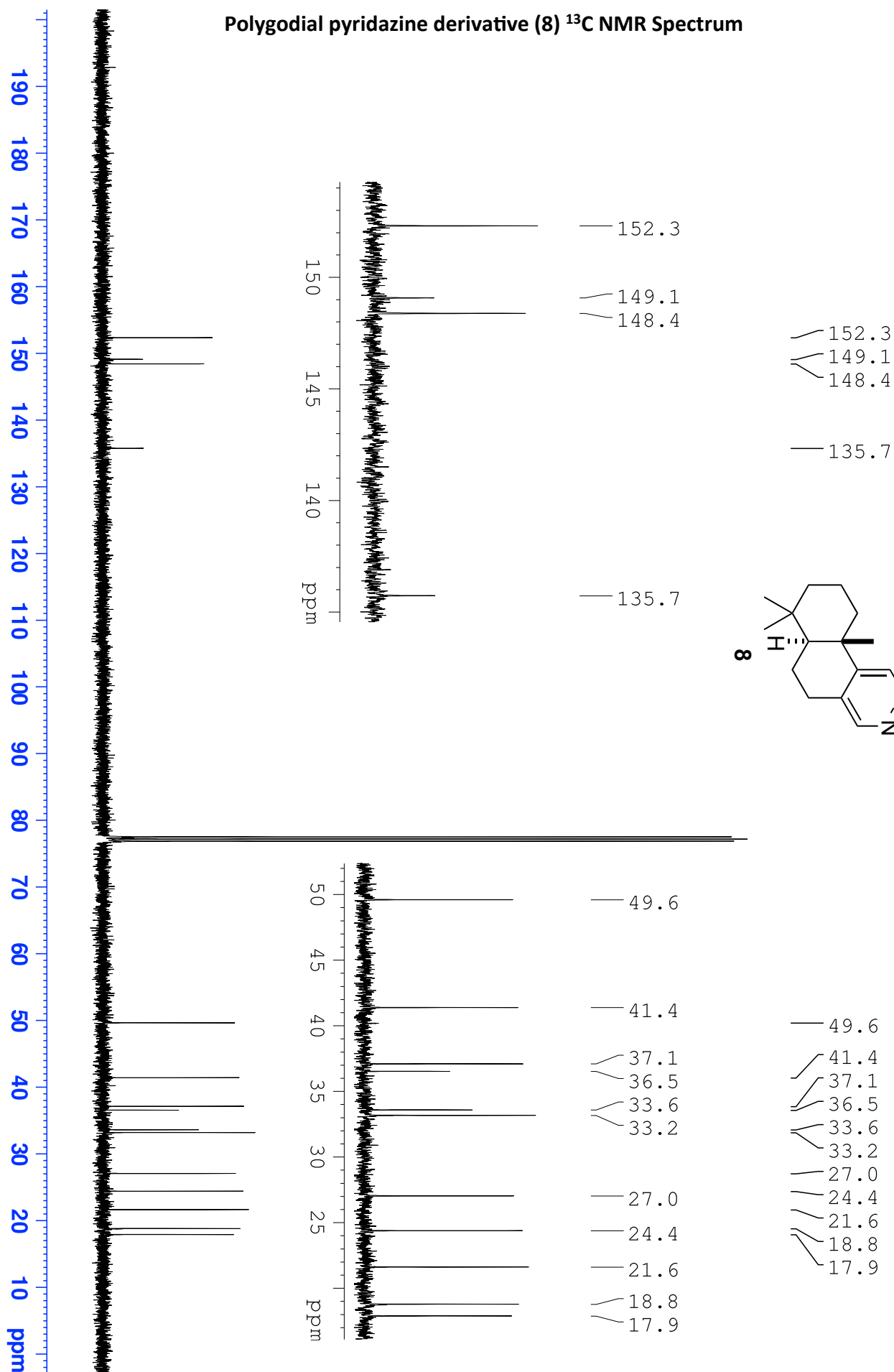
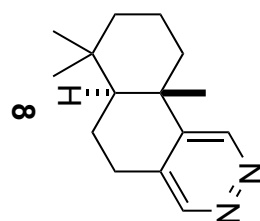
5.30

2.94

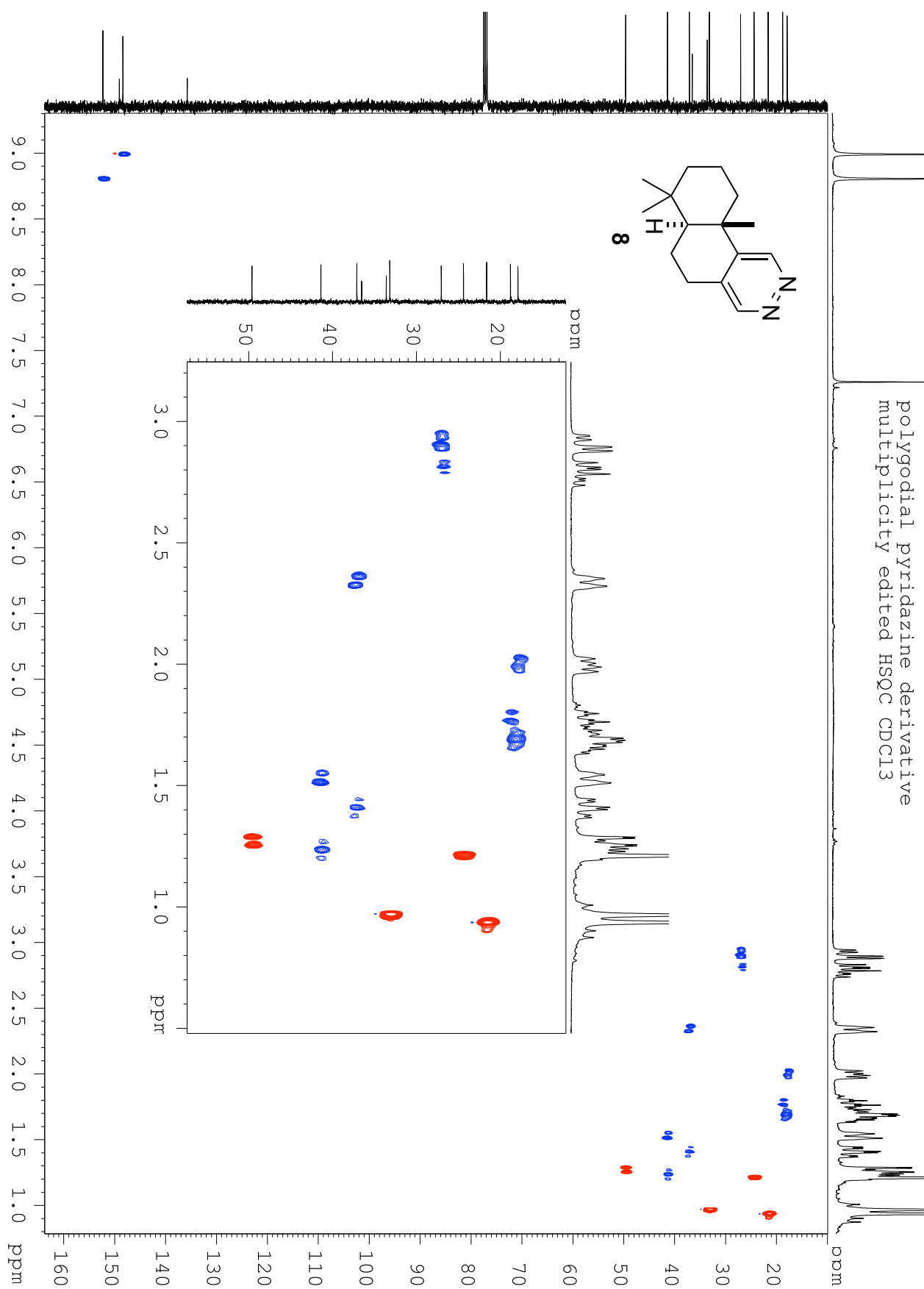
2.90

9.5
 9.0
 8.5
 8.0
 7.5
 7.0
 6.5
 6.0
 5.5
 5.0
 4.5
 4.0
 3.5
 3.0
 2.5
 2.0
 1.5
 1.0
 0.5 ppm

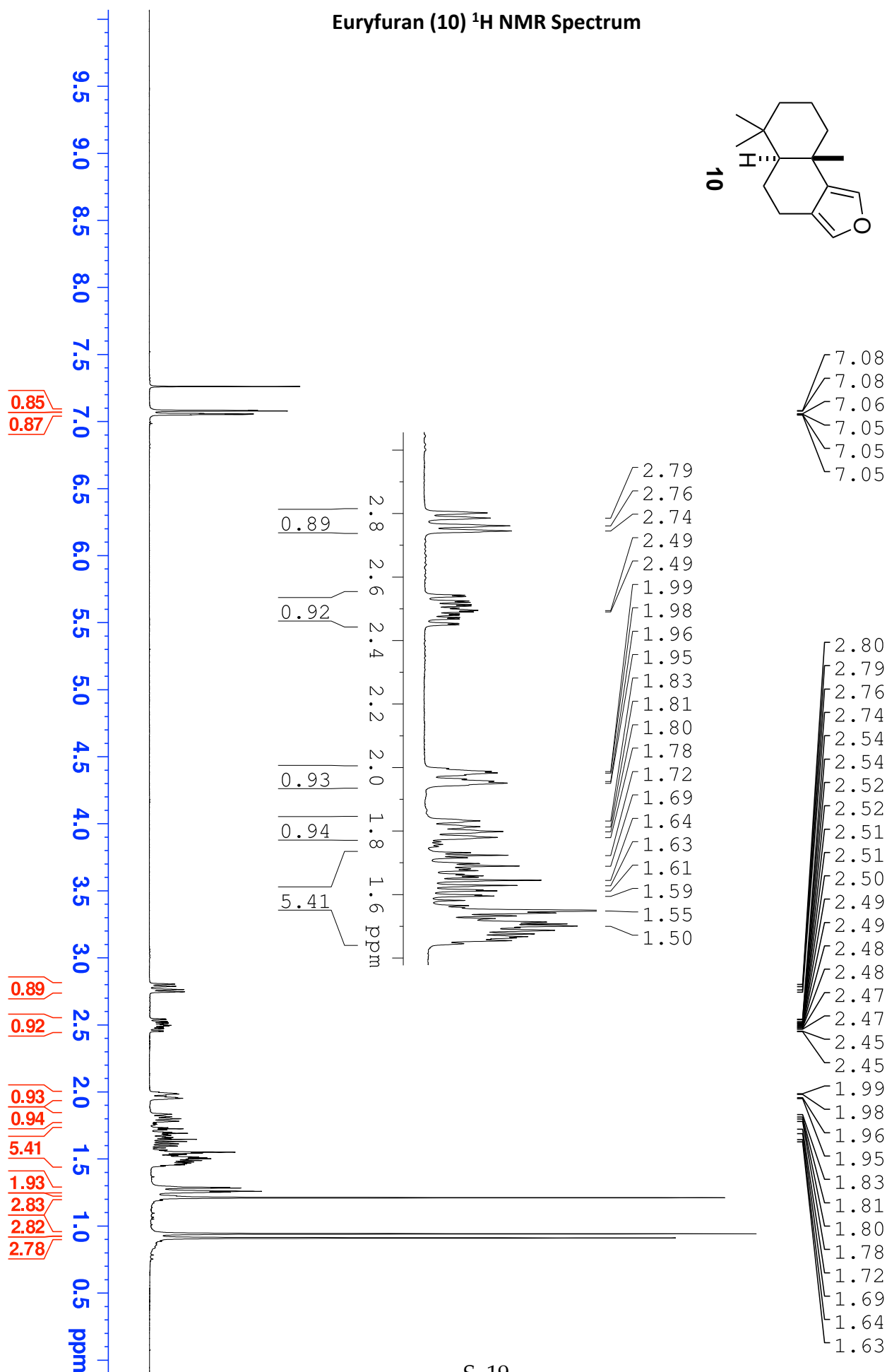
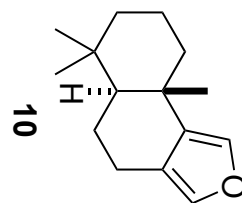
polygodial pyridazine derivative
 ^{13}C CDCl₃



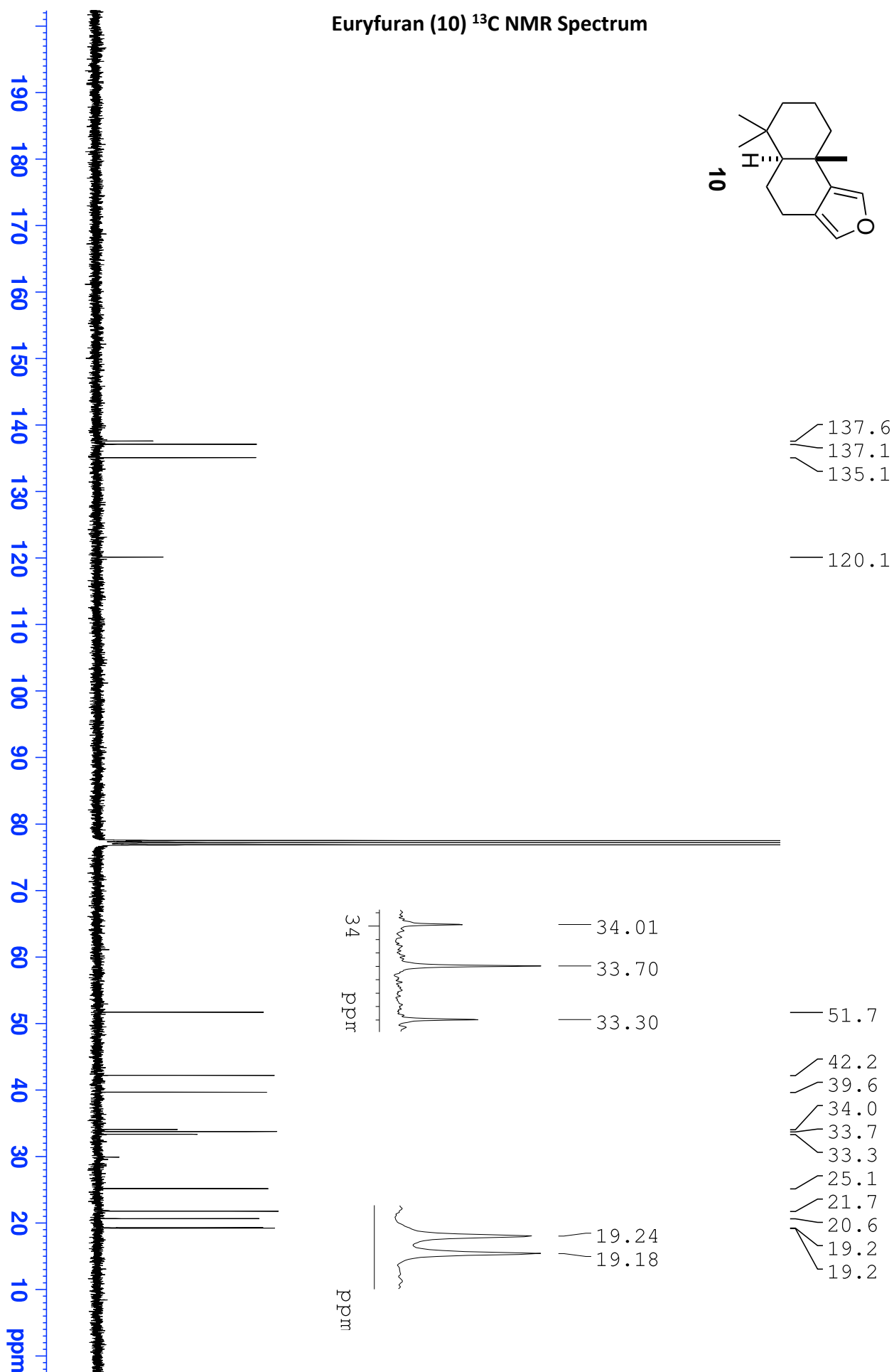
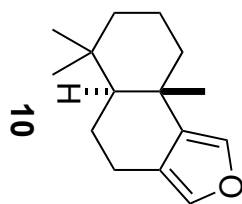
Polygodial pyridazine derivative (8) HSQCme NMR Spectrum



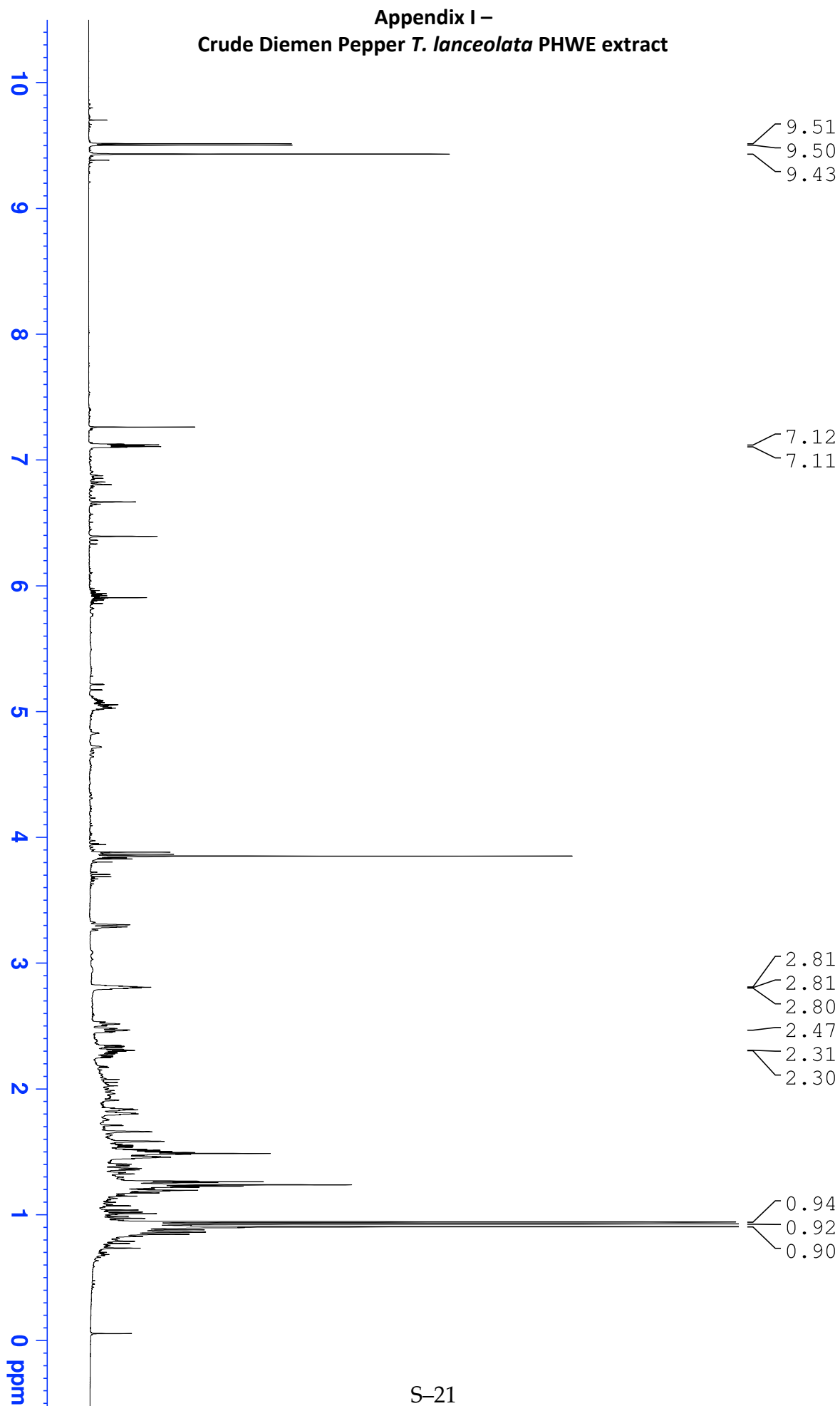
euryfuran
¹H CDCl₃



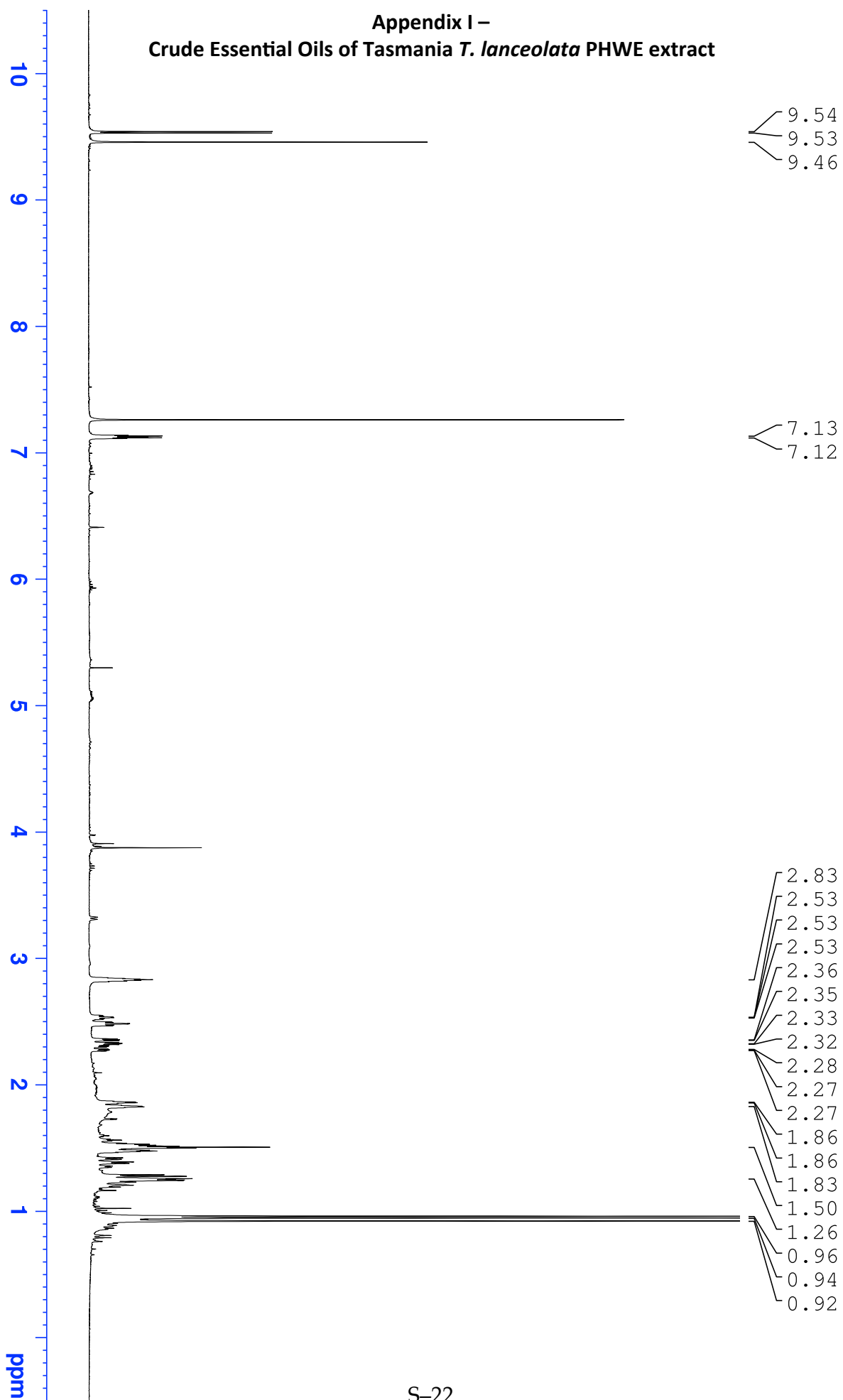
euryfuran
 ^{13}C CDCl_3



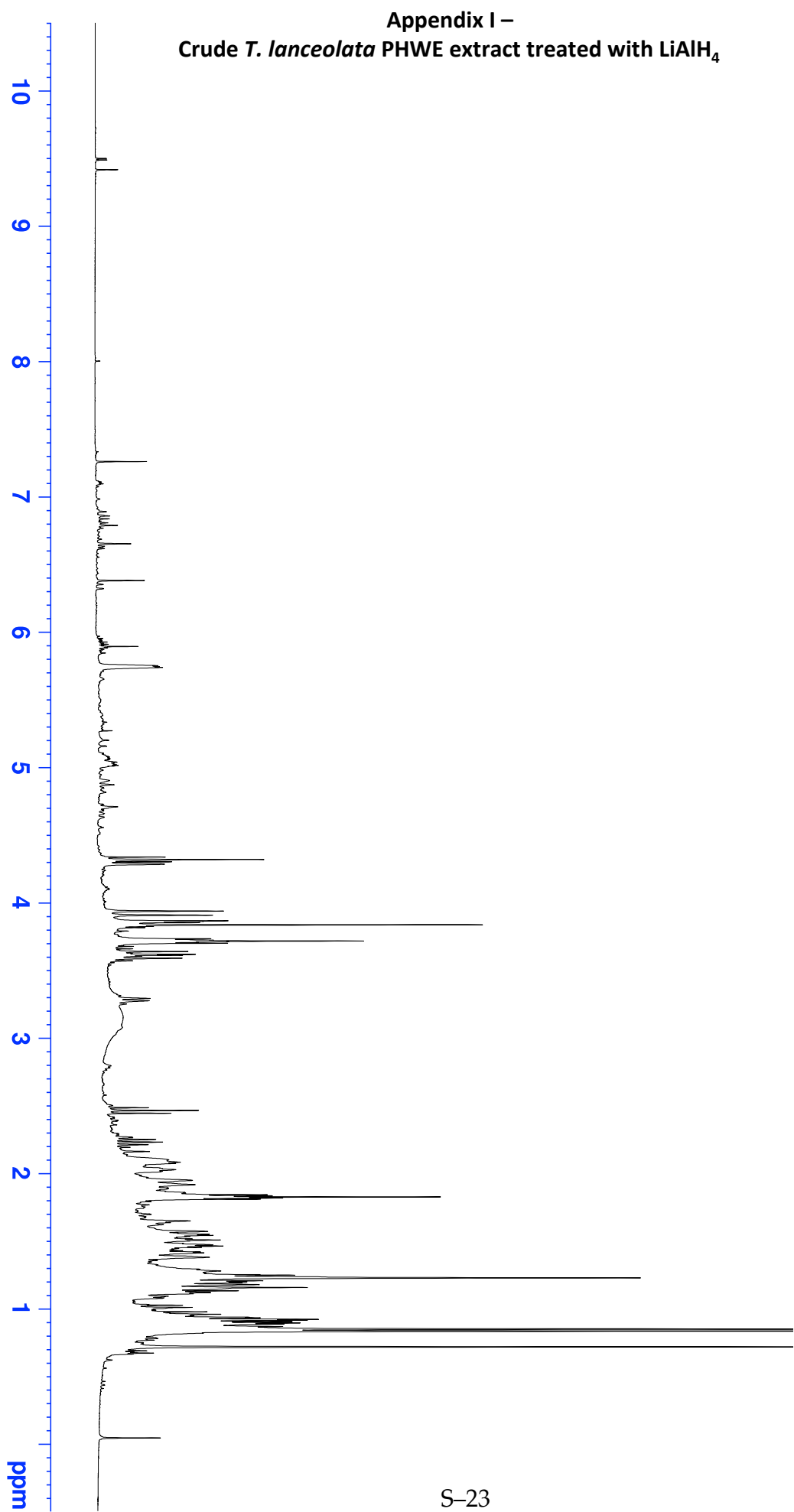
Low yielding *T. lanceolata* from Diemen Pepper
Crude extract CDCl₃



High yielding *T. lanceolata* from EOT
Crude extract CDCl₃

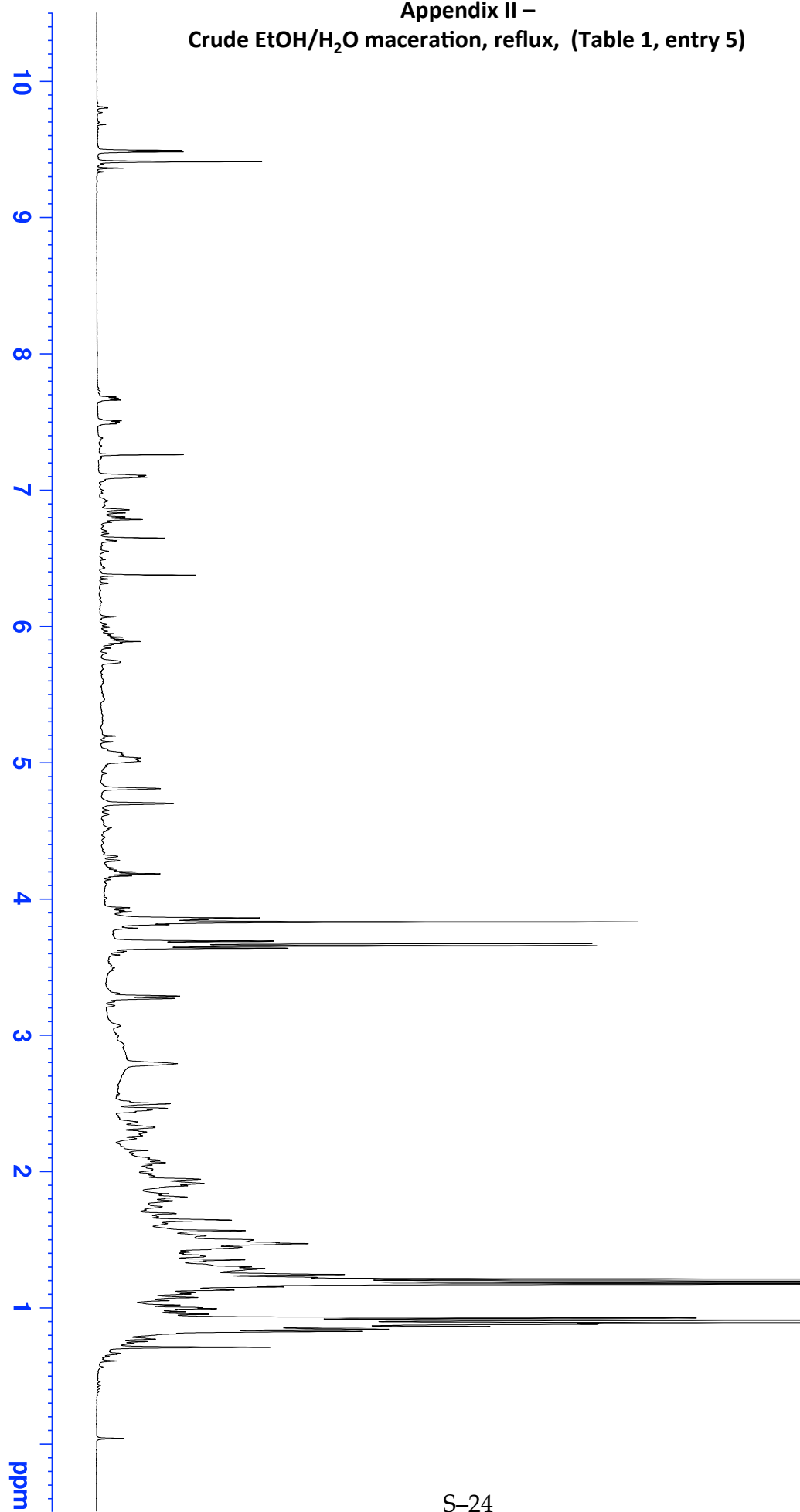


crude *T. lanceolata* extract treated with LiAlH₄
CDCl₃



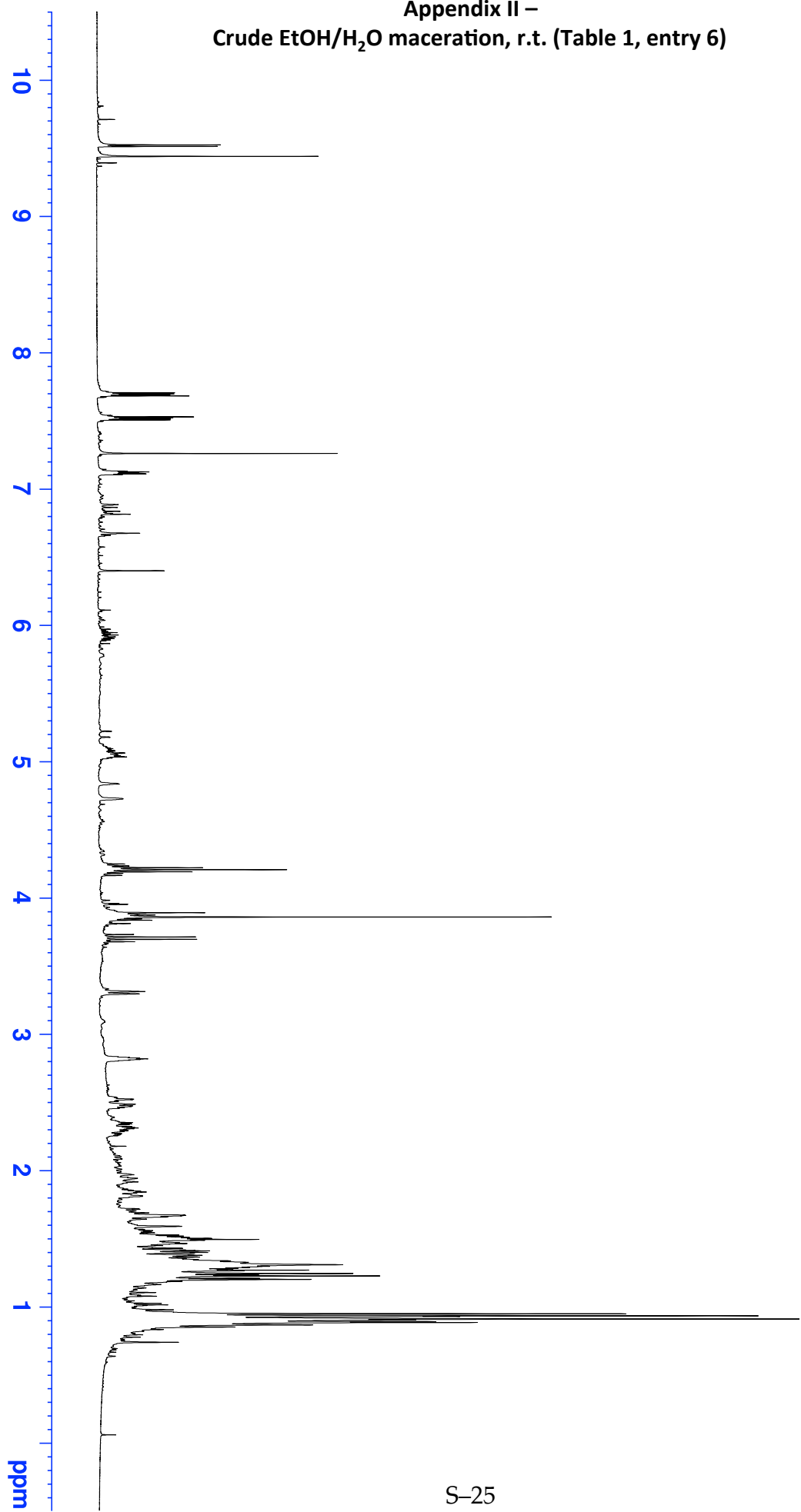
reflux maceration 1hr 35%EtOH/H₂O
CDCl₃

Appendix II –
Crude EtOH/H₂O maceration, reflux, (Table 1, entry 5)



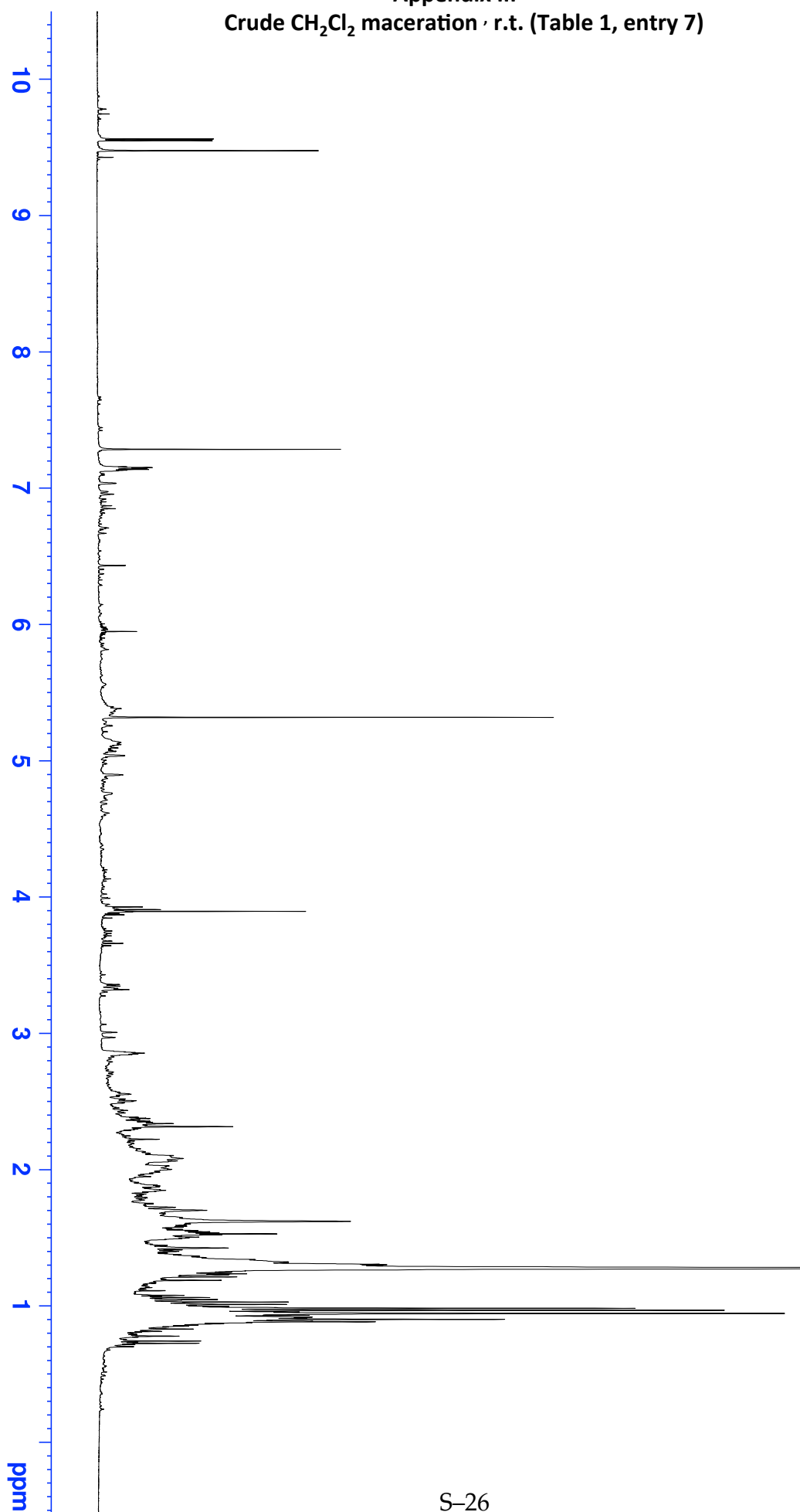
room temp maceration 24hr 35%EtOH/H₂O
CDCl₃

Appendix II –
Crude EtOH/H₂O maceration, r.t. (Table 1, entry 6)

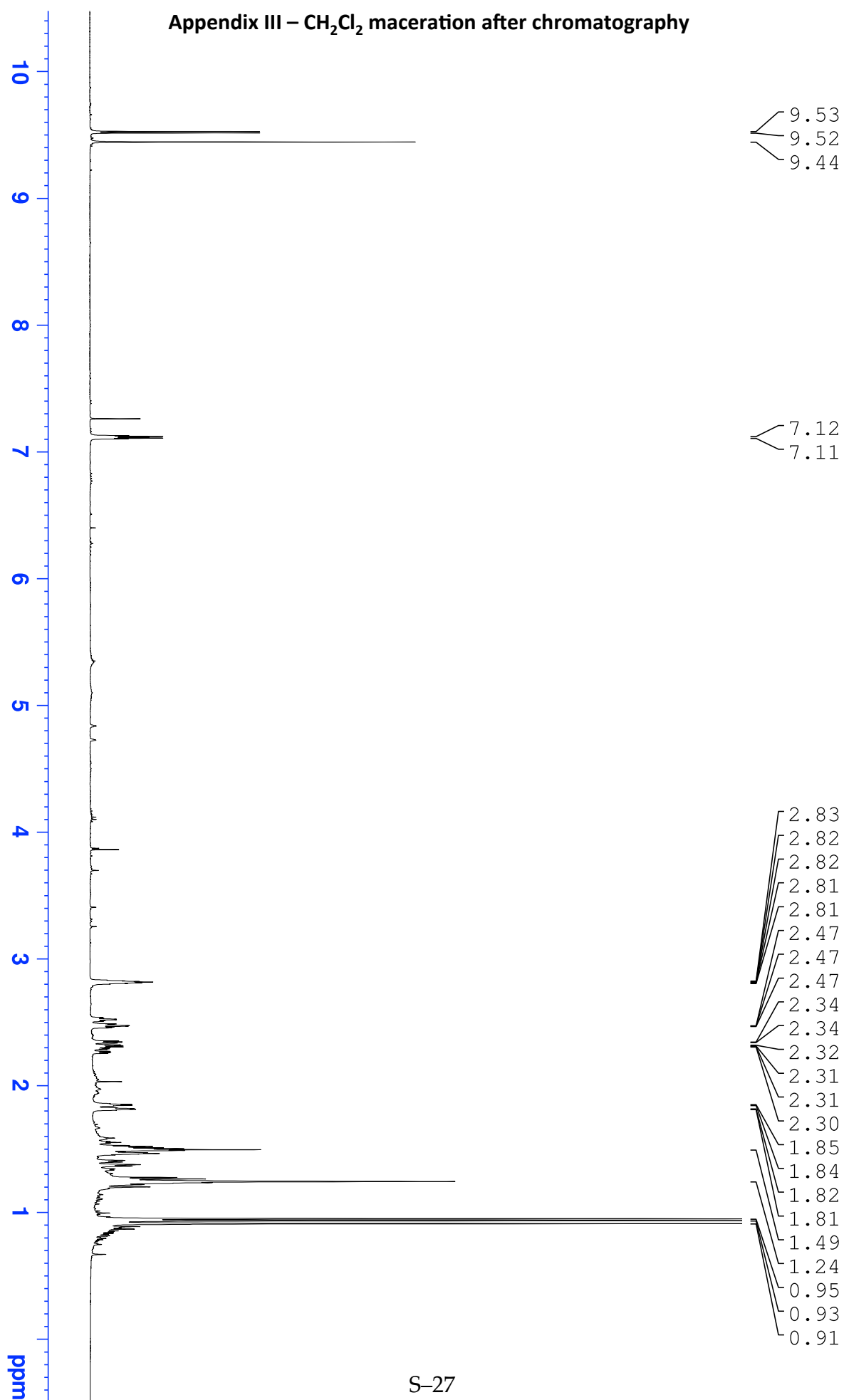


room temp maceration 24hr CH₂Cl₂
CDCl₃

Appendix III –
Crude CH₂Cl₂ maceration ' r.t. (Table 1, entry 7)

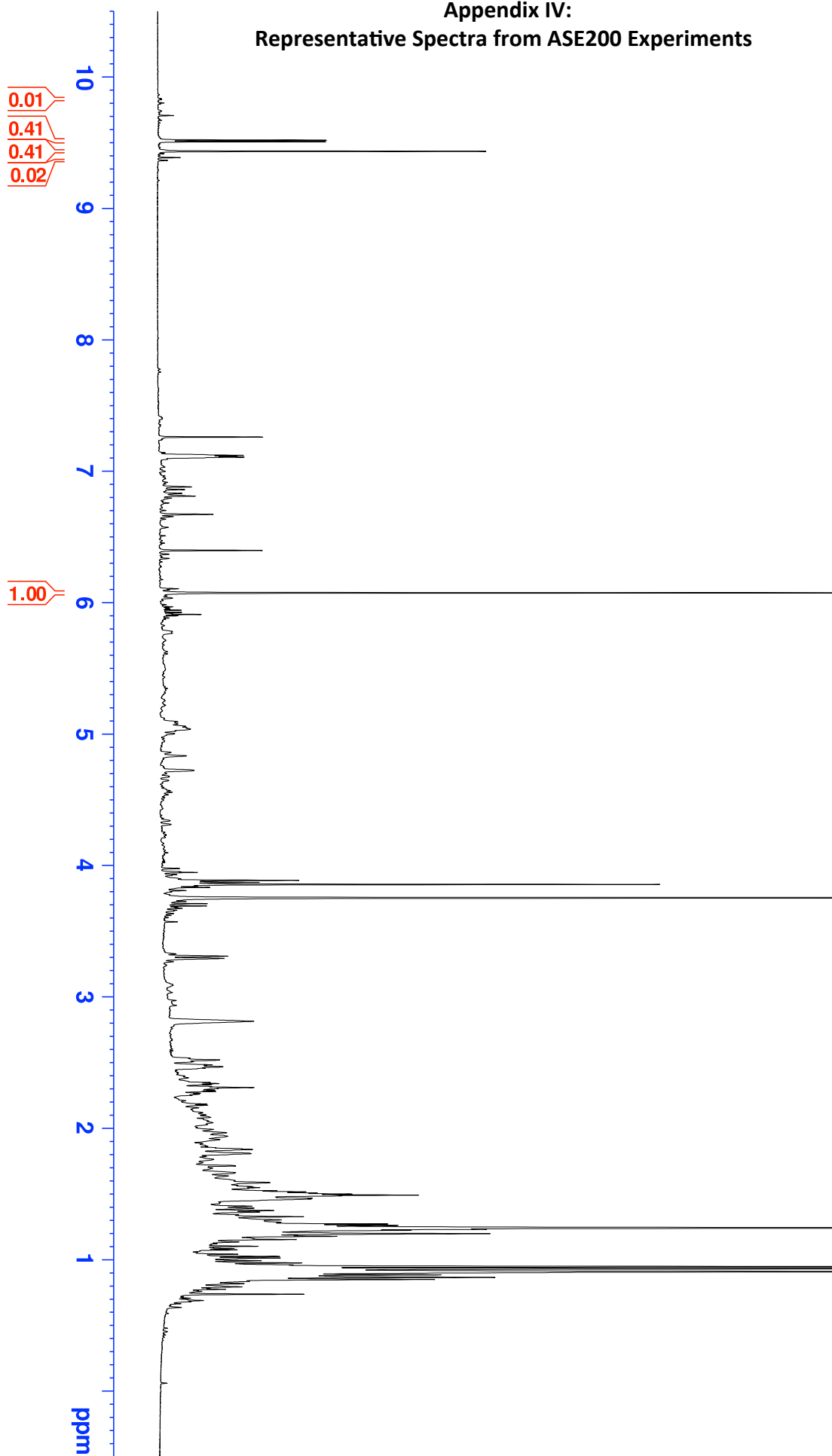


room temp maceration 24hr CH₂Cl₂ F19-21
CDCl₃

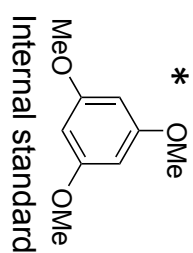
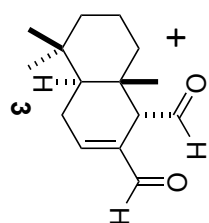
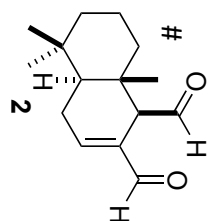


AST exp 24
500psi
70 degrees
35% EtOH

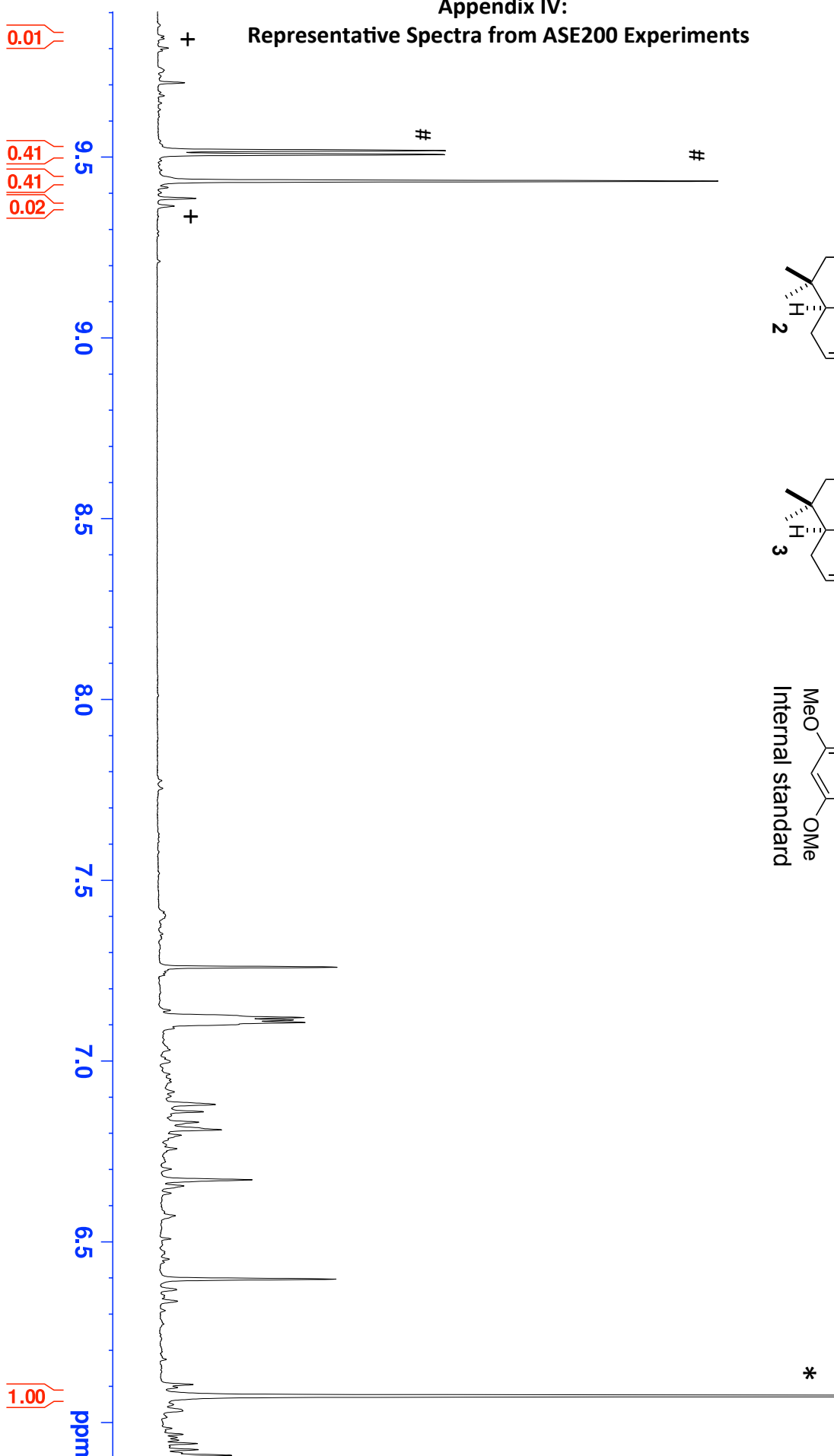
Appendix IV: Representative Spectra from ASE200 Experiments



AST exp 24
500psi
70 degrees
35% EtOH

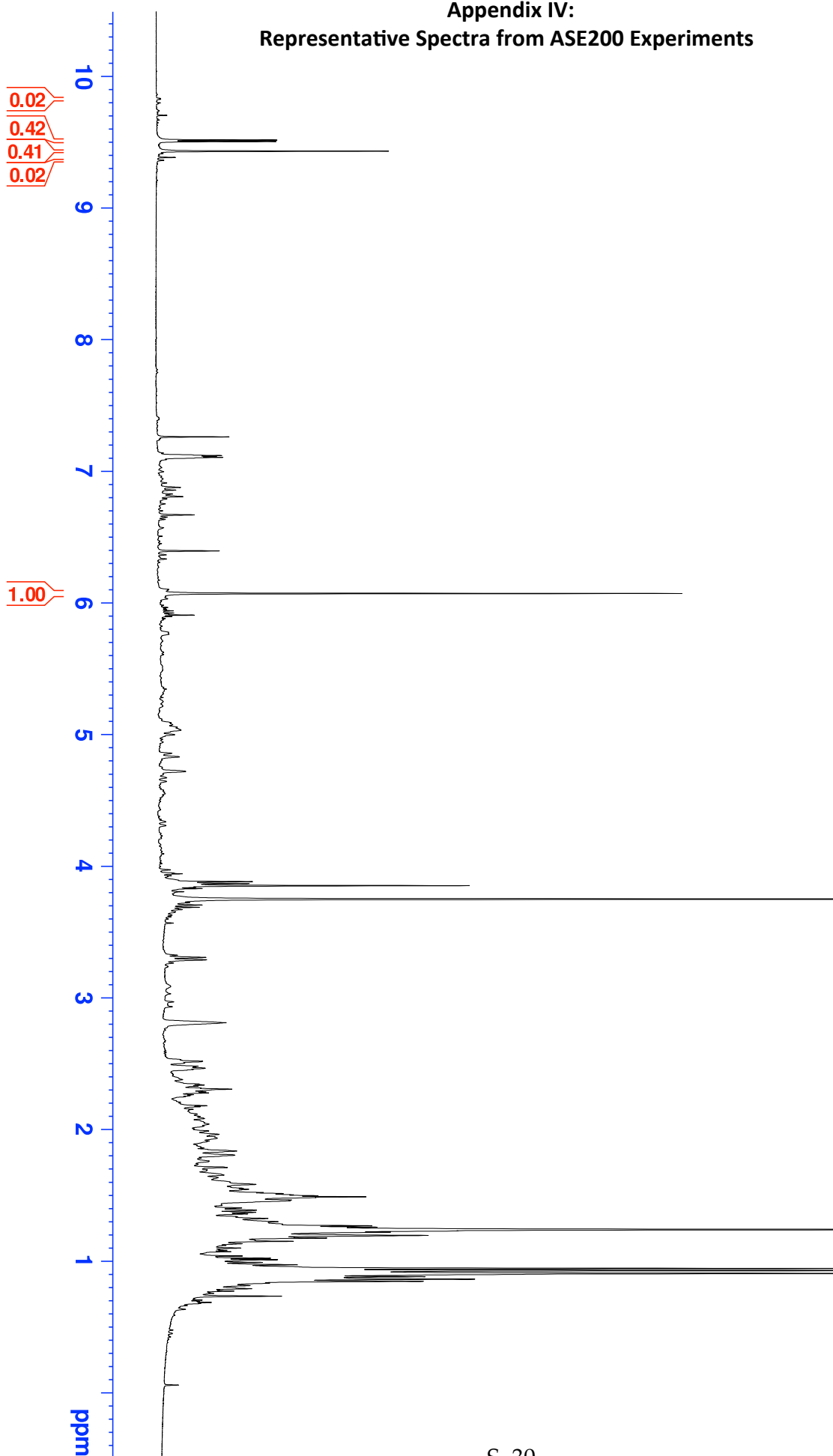


Appendix IV: Representative Spectra from ASE200 Experiments

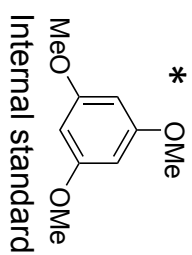
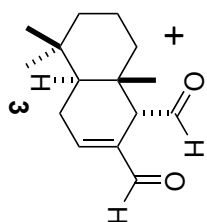
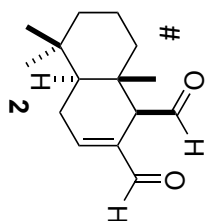


AST exp 18
500psi
90 degrees
35% EtOH

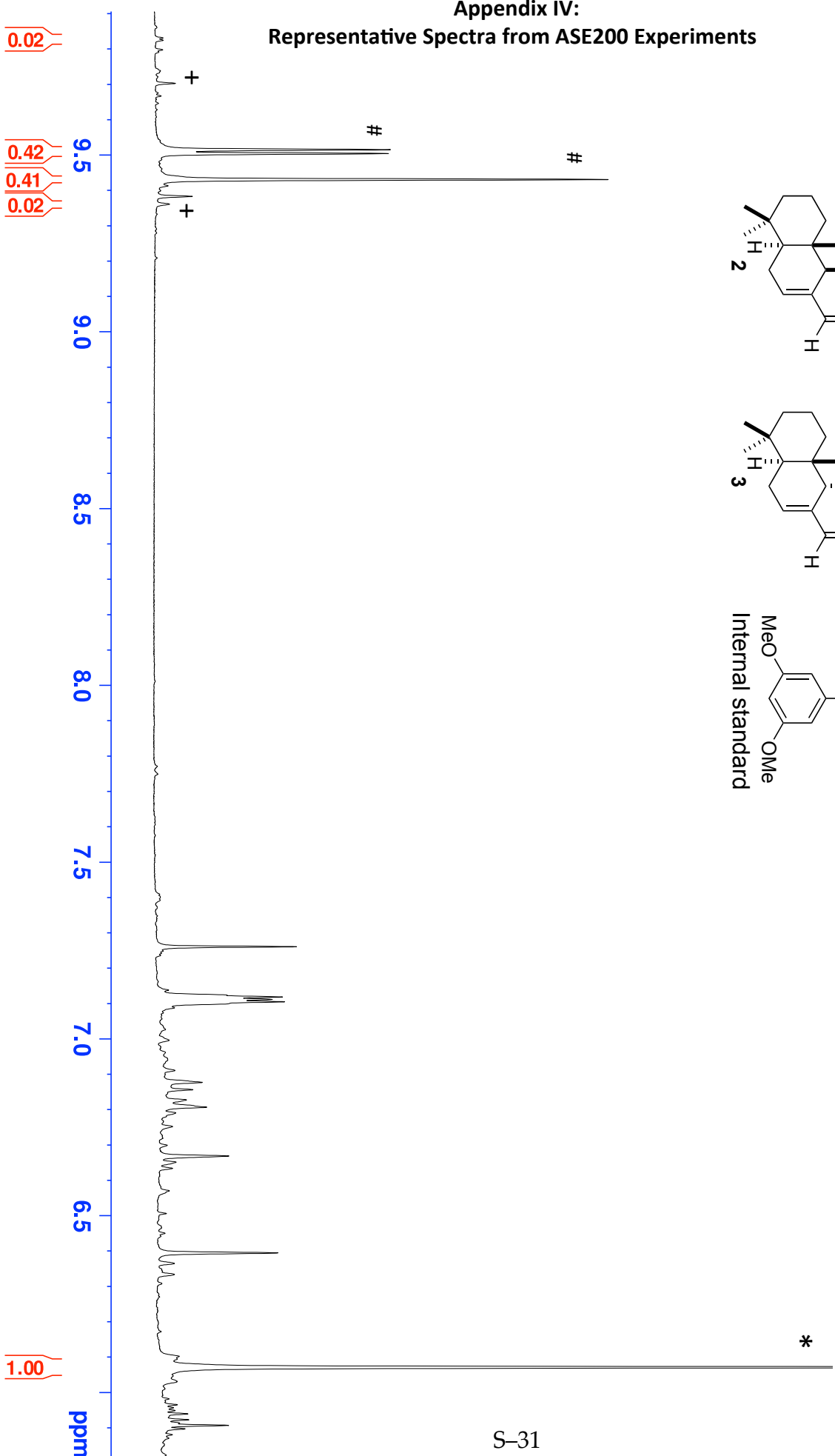
Appendix IV: Representative Spectra from ASE200 Experiments



AST exp 18
500psi
90 degrees
35% EtOH

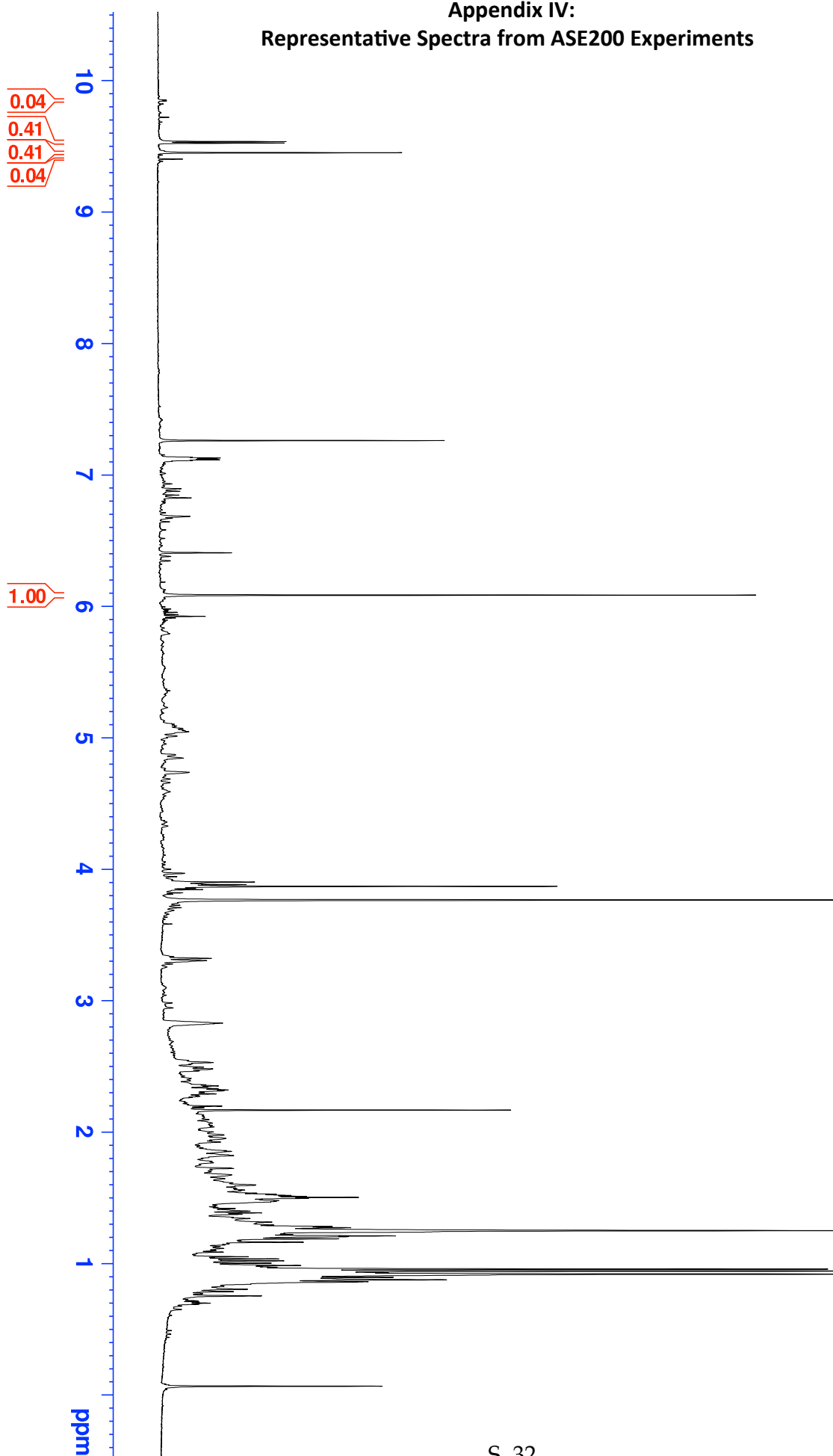


Appendix IV: Representative Spectra from ASE200 Experiments

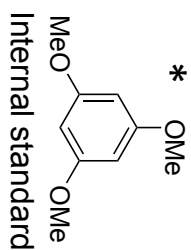
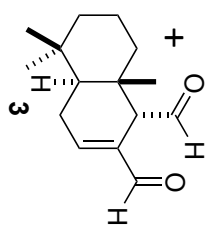
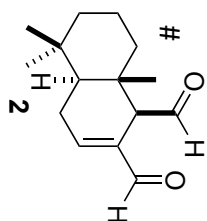


AST exp 12
500psi
110 degrees
35% EtOH

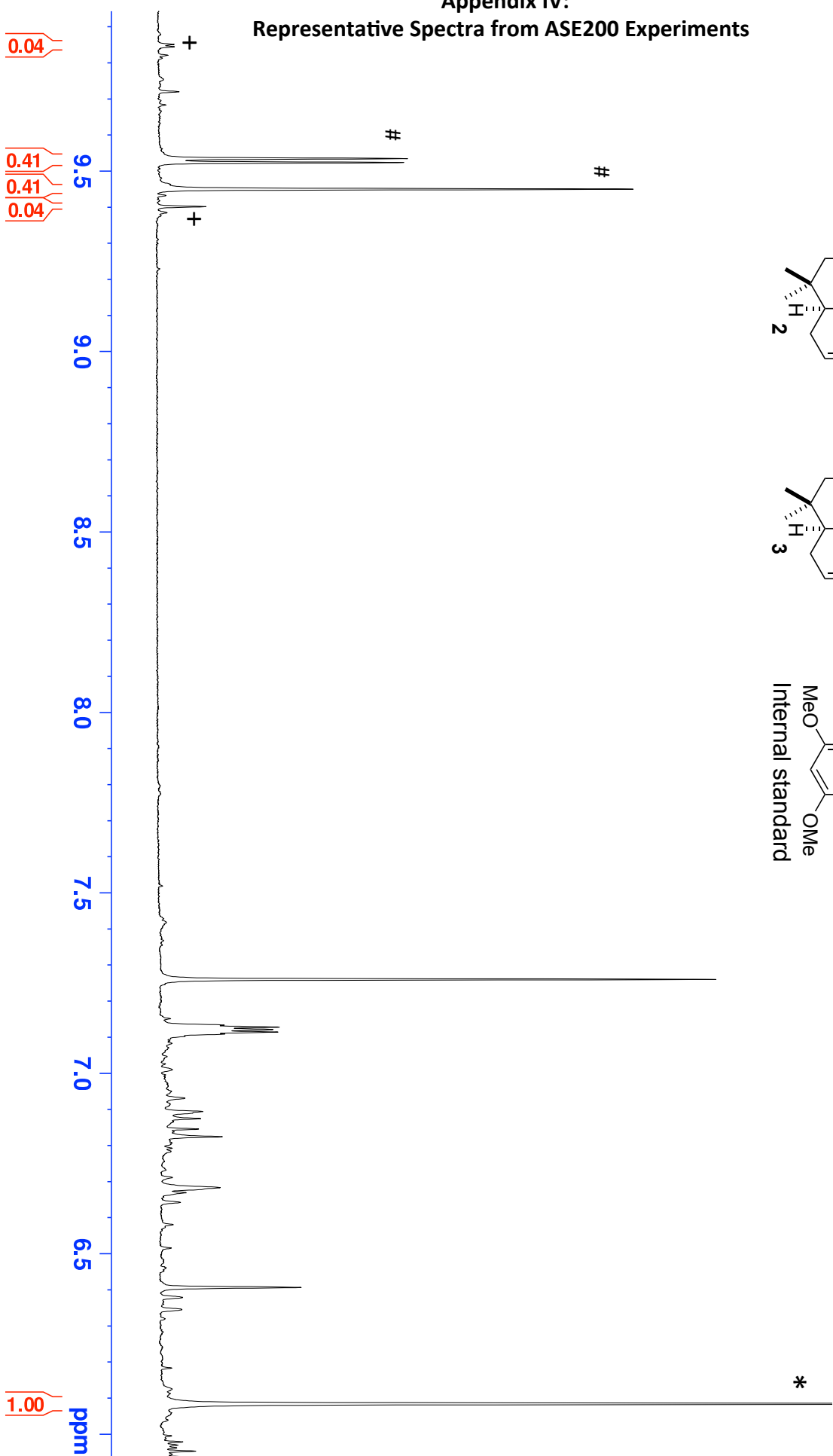
Appendix IV: Representative Spectra from ASE200 Experiments



AST exp 12
500psi
110 degrees
35% EtOH

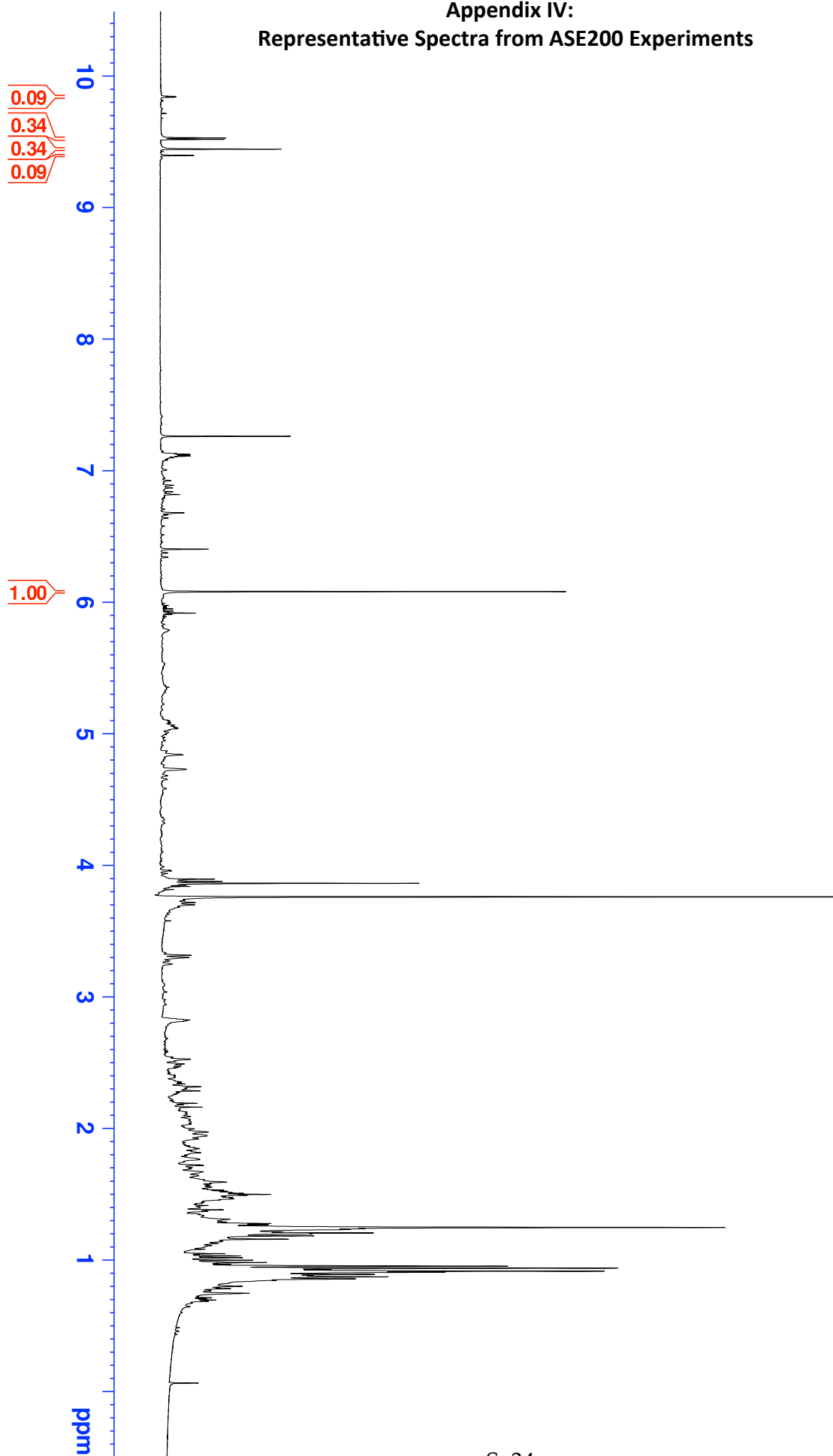


Appendix IV: Representative Spectra from ASE200 Experiments

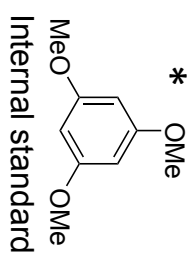
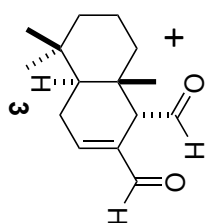
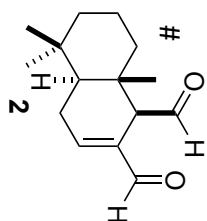


AST exp 6
500psi
130 degrees
35% EtOH

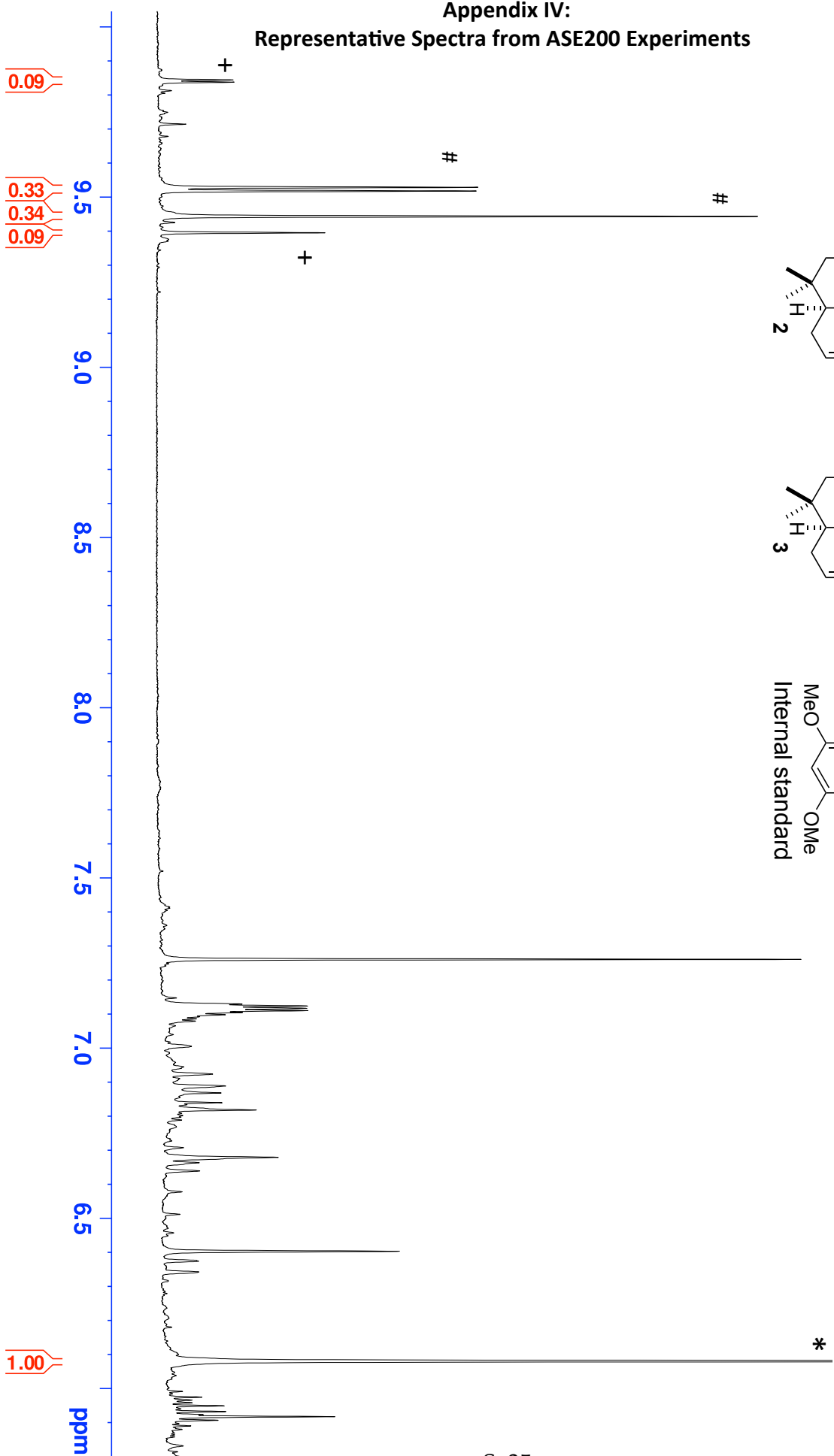
Appendix IV: Representative Spectra from ASE200 Experiments



AST exp 6
500psi
130 degrees
35% EtOH

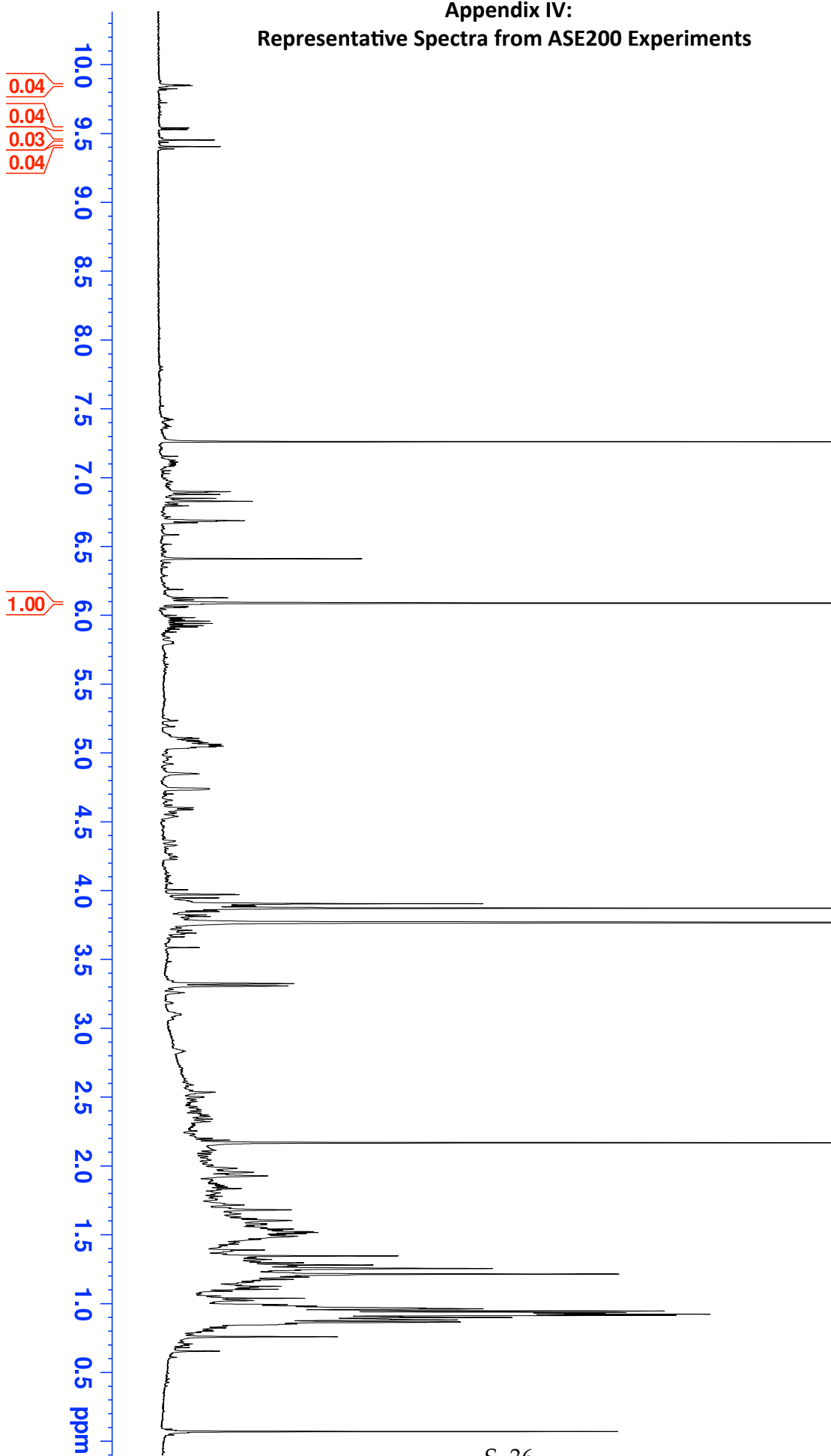


Appendix IV: Representative Spectra from ASE200 Experiments

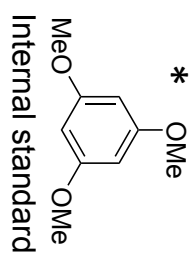
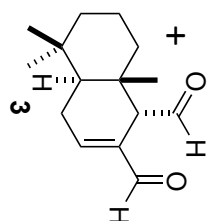
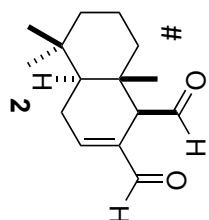


AST exp 1
1500psi
150 degrees
0% EtOH

Appendix IV: Representative Spectra from ASE200 Experiments



AST exp 1
1500psi
150 degrees
0% EtOH



Appendix IV: Representative Spectra from ASE200 Experiments

