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).

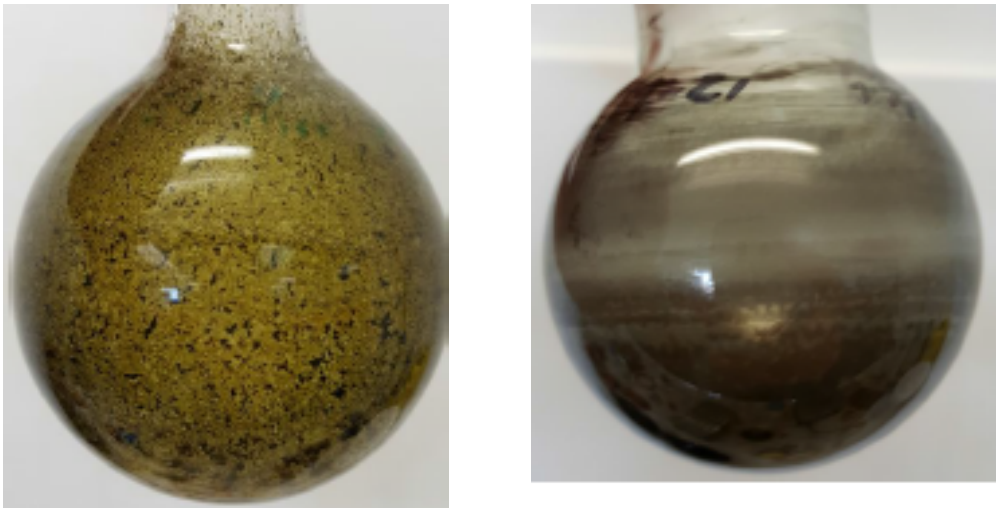


Figure S2. Maceration extract (left) and polygodial isolated after chromatography from maceration (right) (Table 1, entry 7).

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

Yield, % w/w[a]		EtOH/H_2O (% 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_2O (% 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.

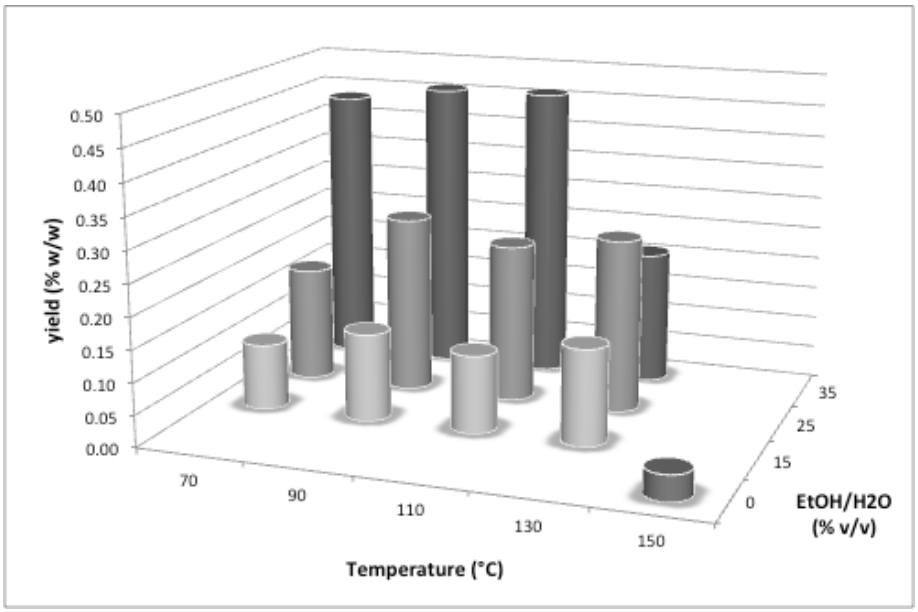
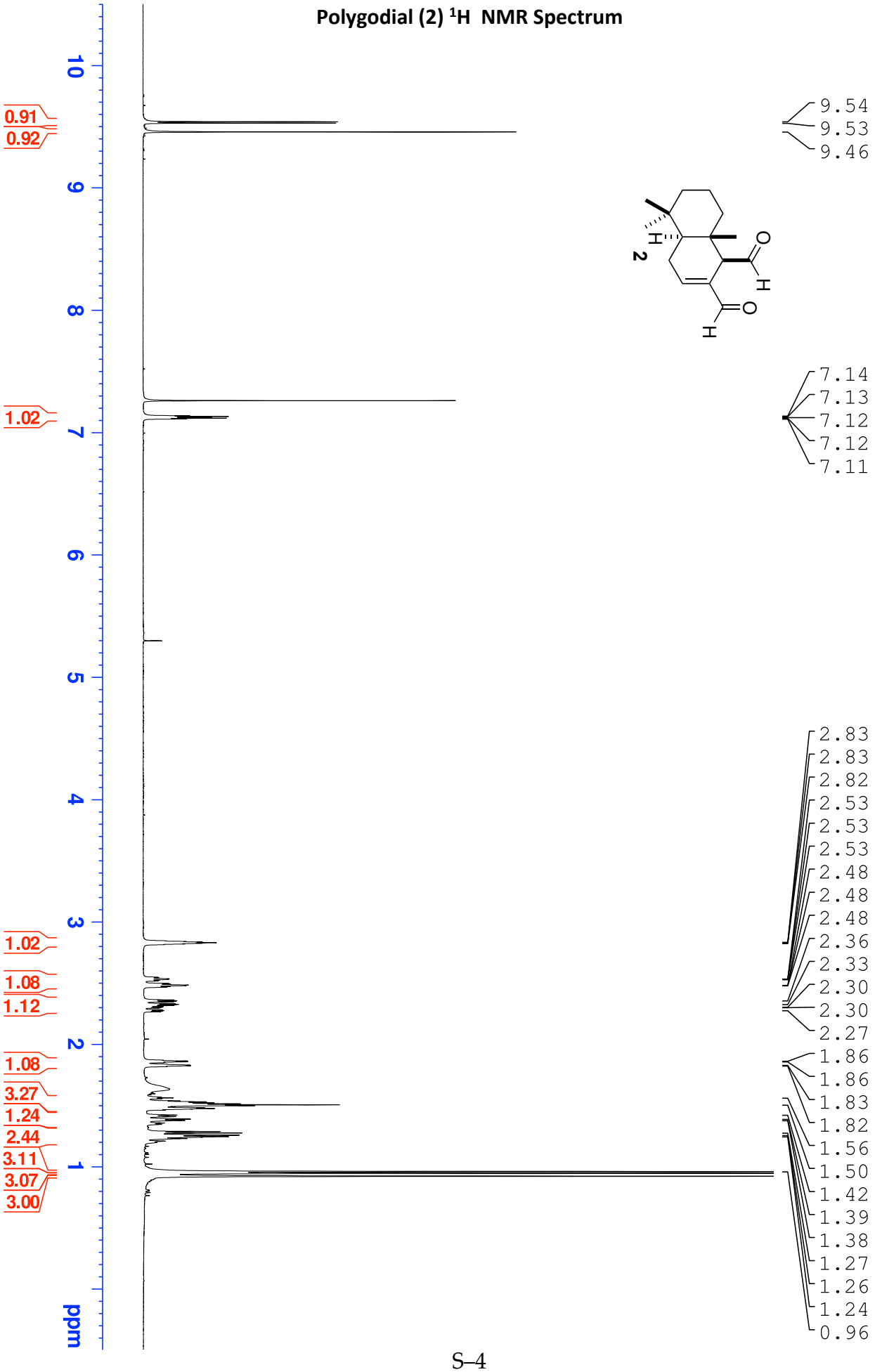
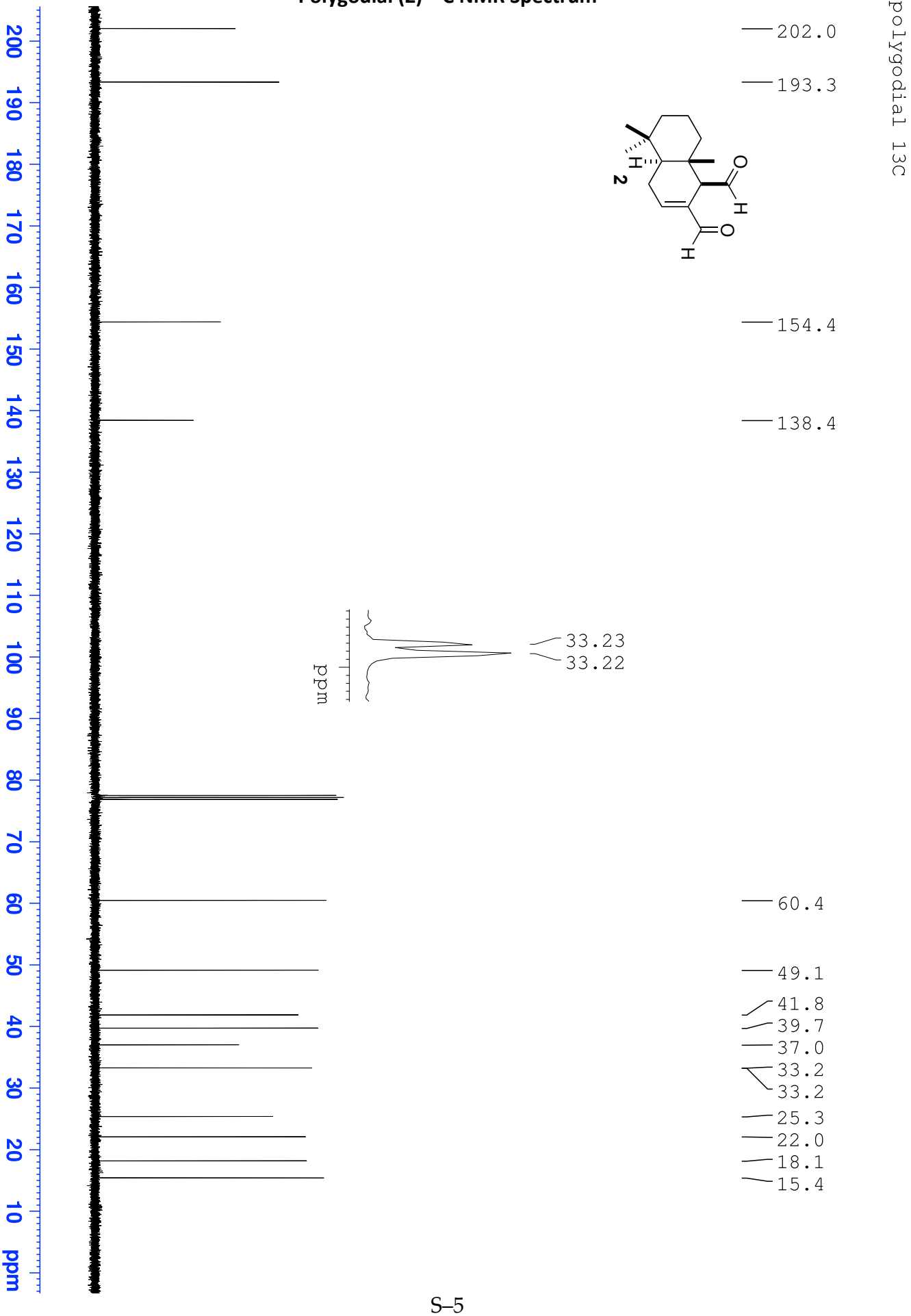


Figure S3. Effect of temperature and solvent composition on polygodial yield. Experiments were performed using a Dionex ASE200 at 1500 psi (~35 bar). Yields were determined *via* ^{1}H NMR spectroscopy with the aid of an internal standard.

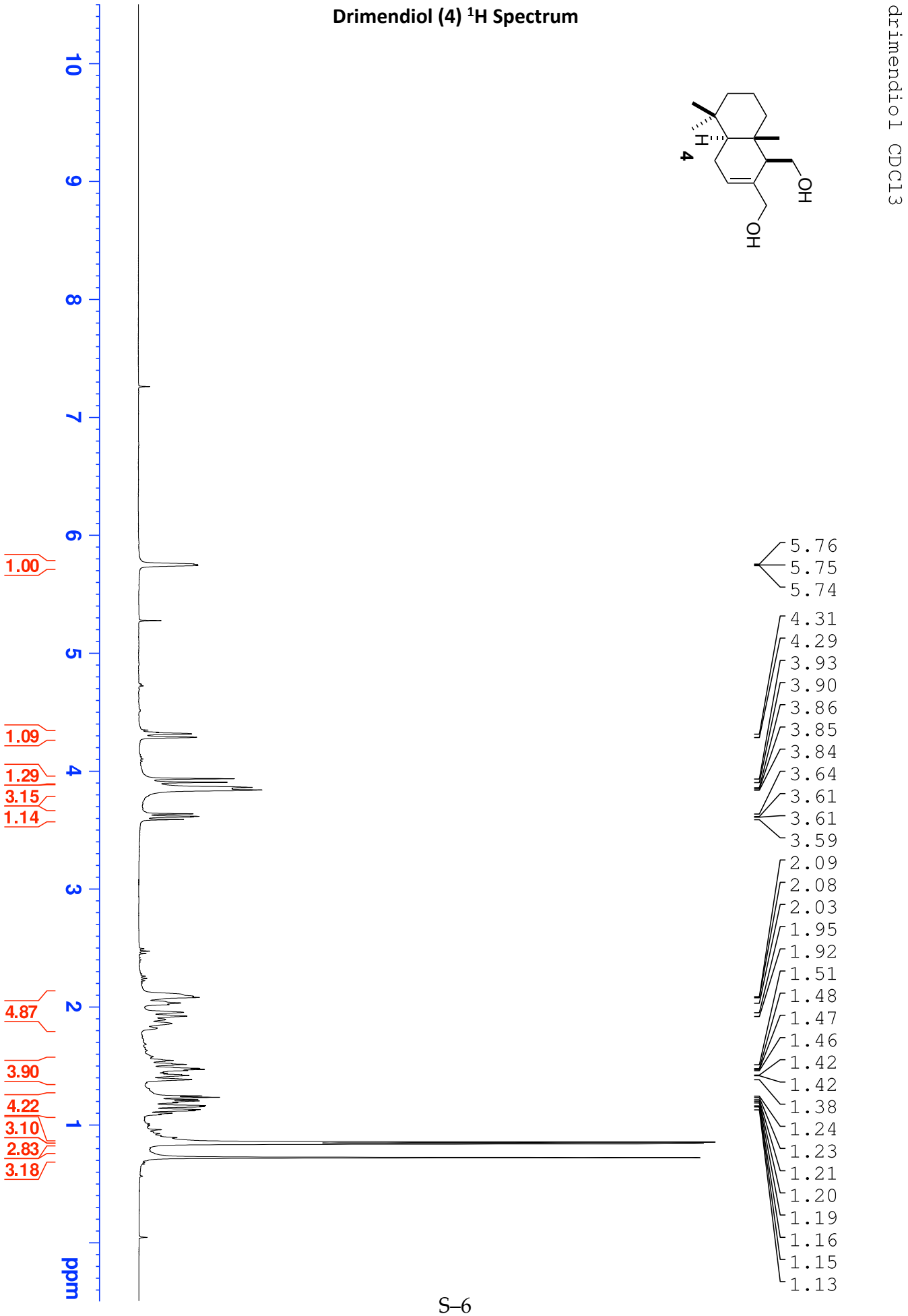
Polygodial (2) ^{1}H NMR Spectrum



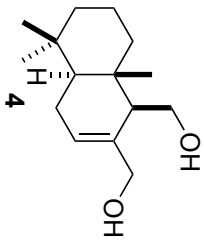
Polygodial (2) ^{13}C NMR Spectrum



Drimendiol (4) ^{1}H Spectrum

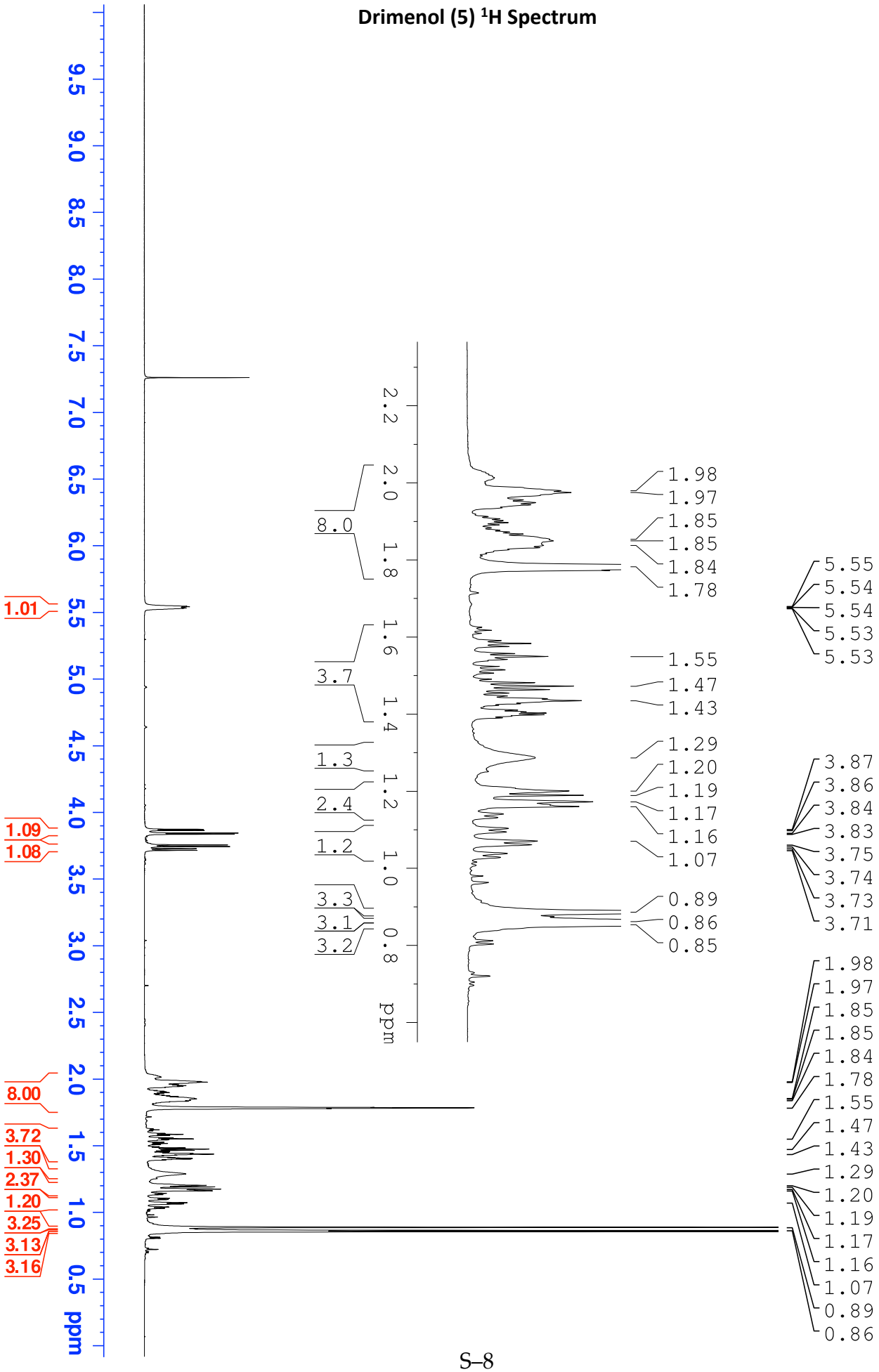


Drimendiol (4) ^{13}C NMR Spectrum

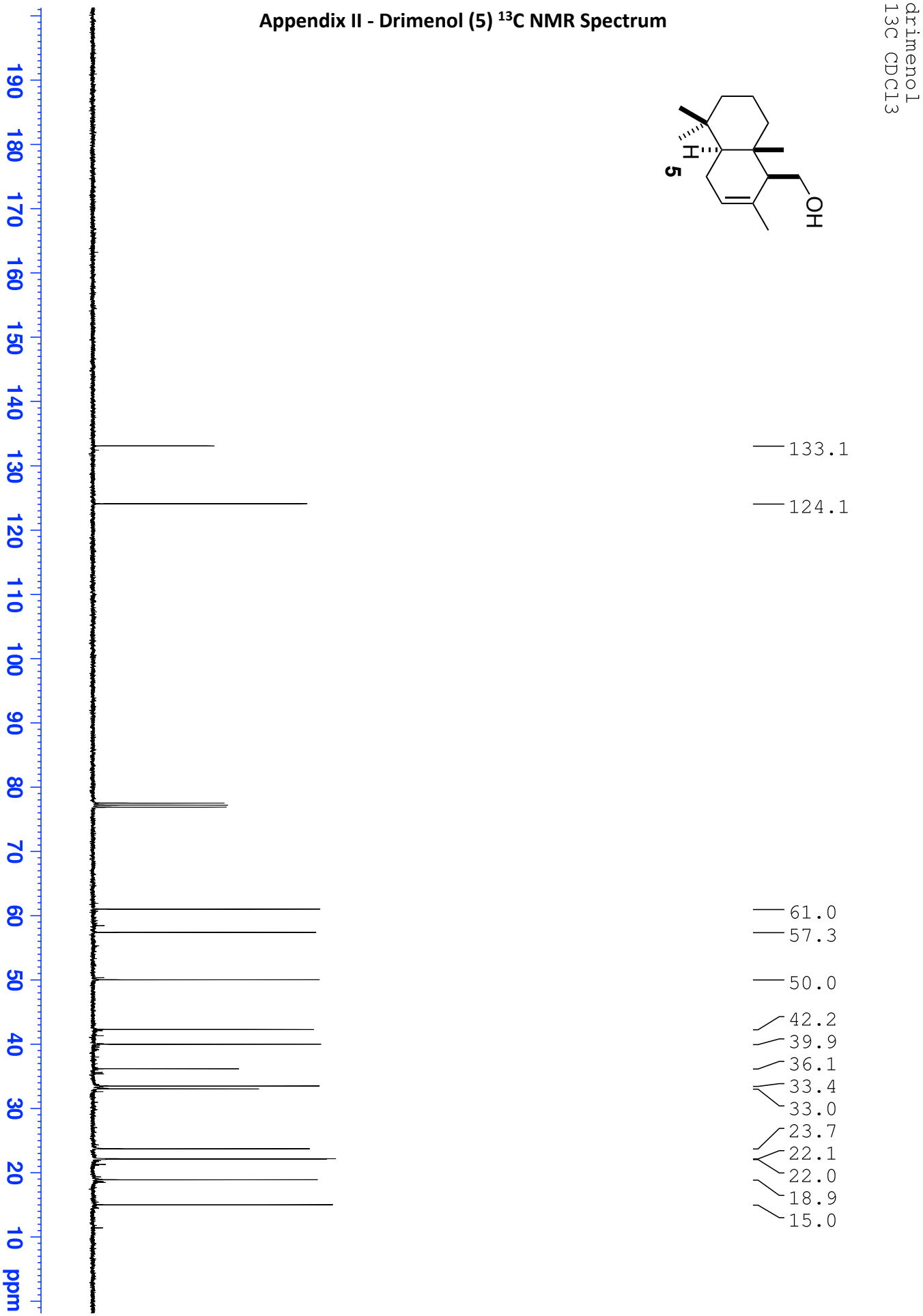


drimendiol 13C CDC13

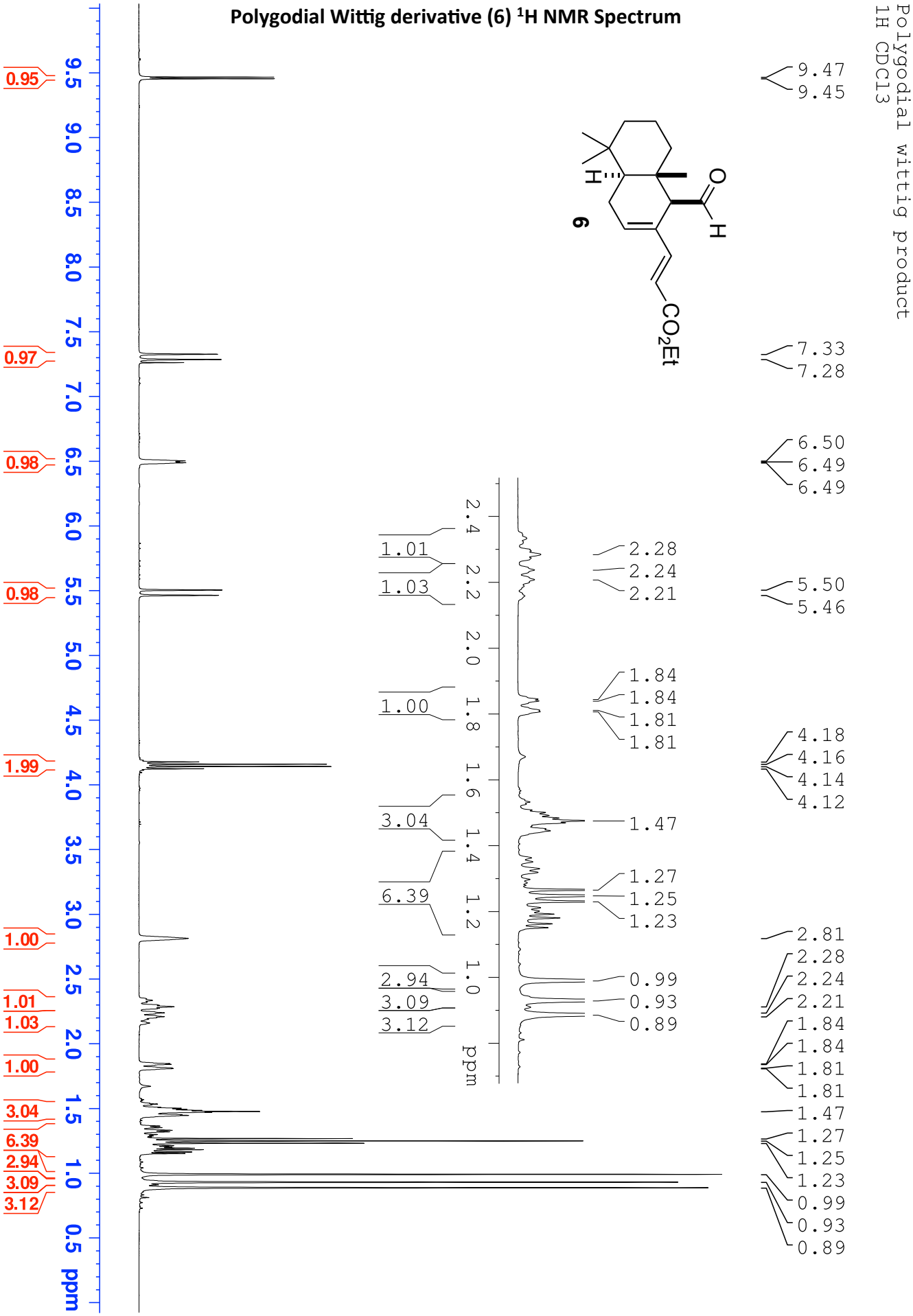
Drimenol (5) ^{1}H Spectrum



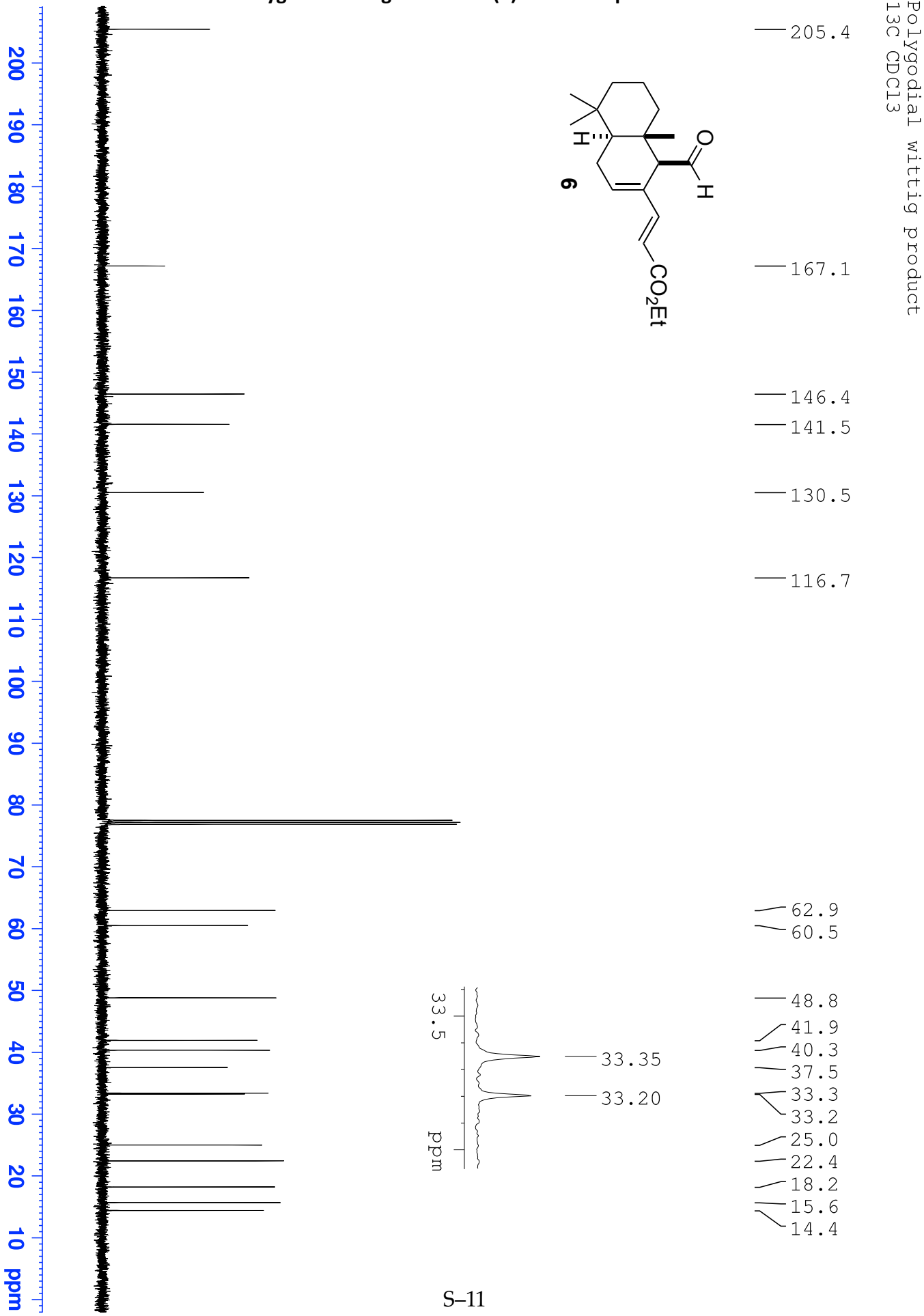
Appendix II - Drimenol (5) ^{13}C NMR Spectrum



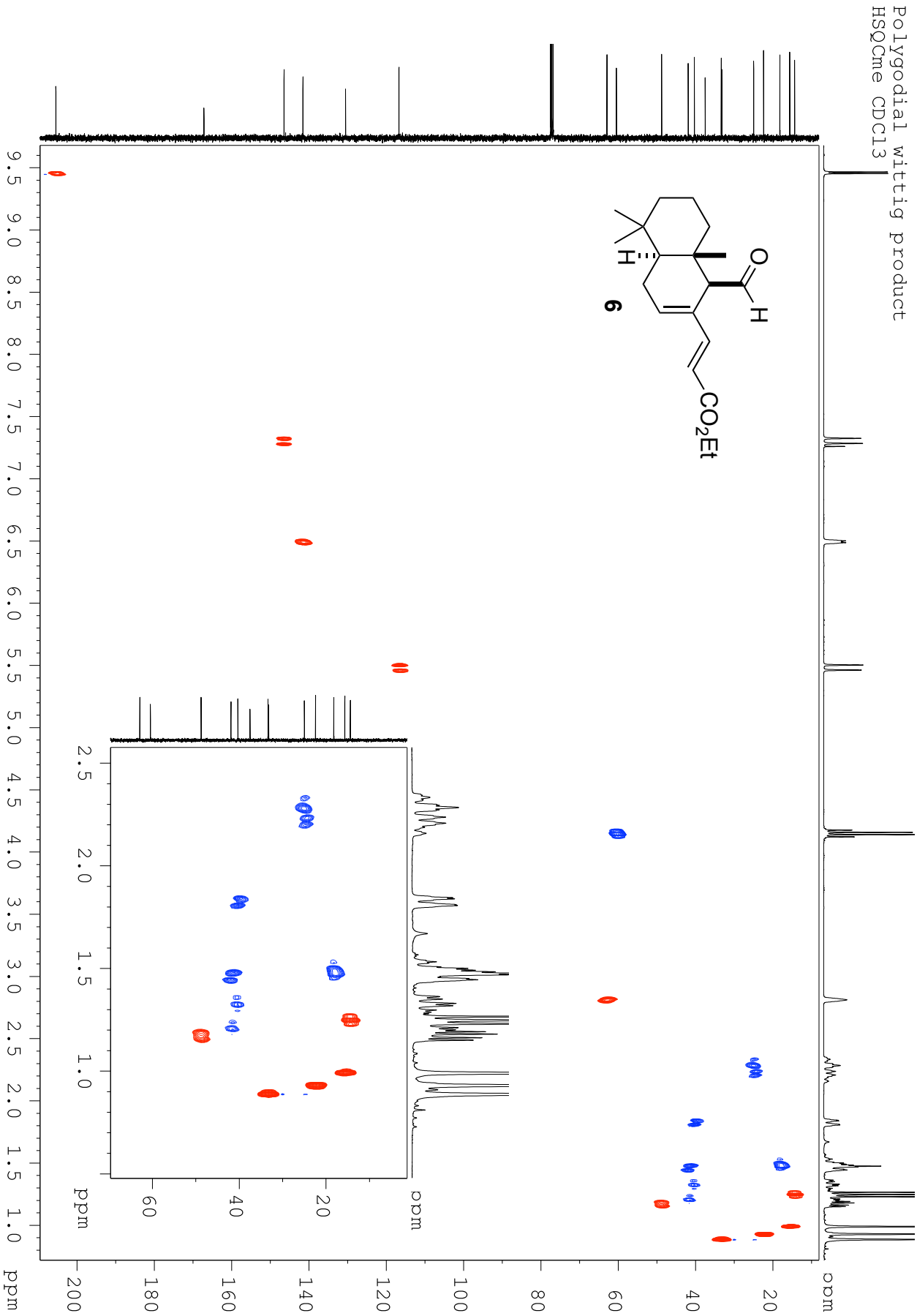
Polygodial Wittig derivative (6) ^{1}H NMR Spectrum



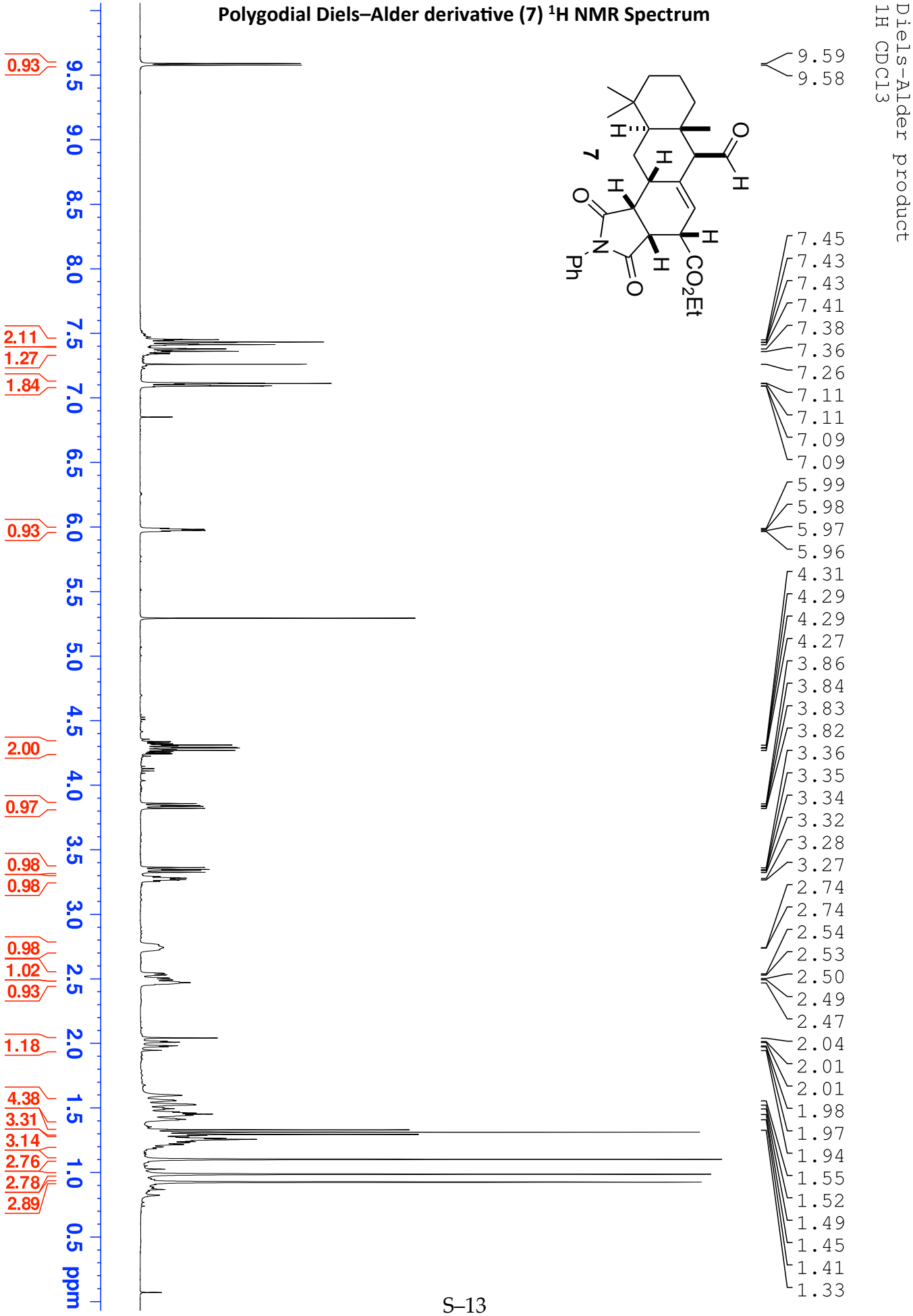
Polygodial Wittig derivative (6) ^{13}C NMR Spectrum



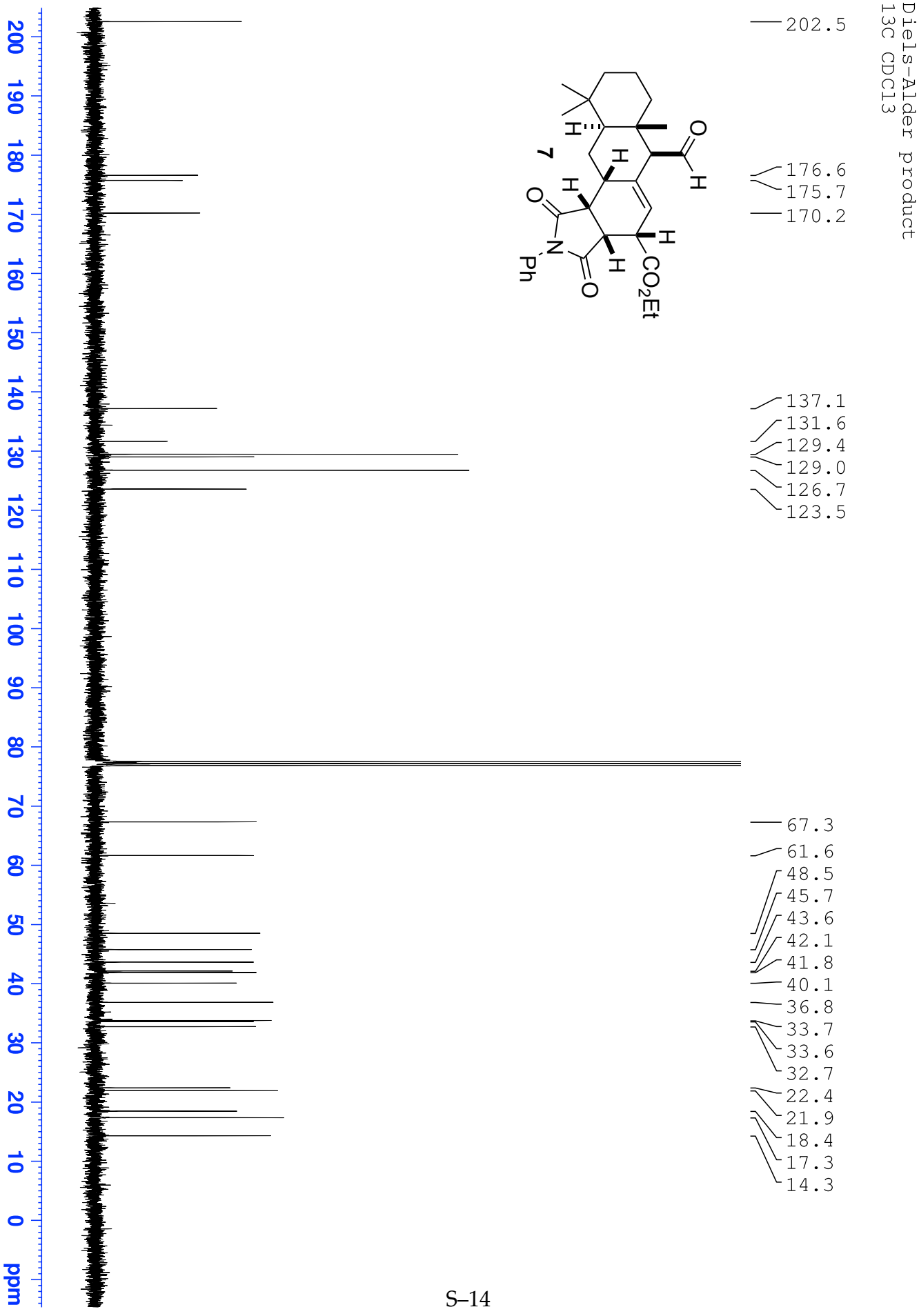
Polygodial Wittig derivative (6) ^{1}H-^{13}C HSQCme NMR Spectrum



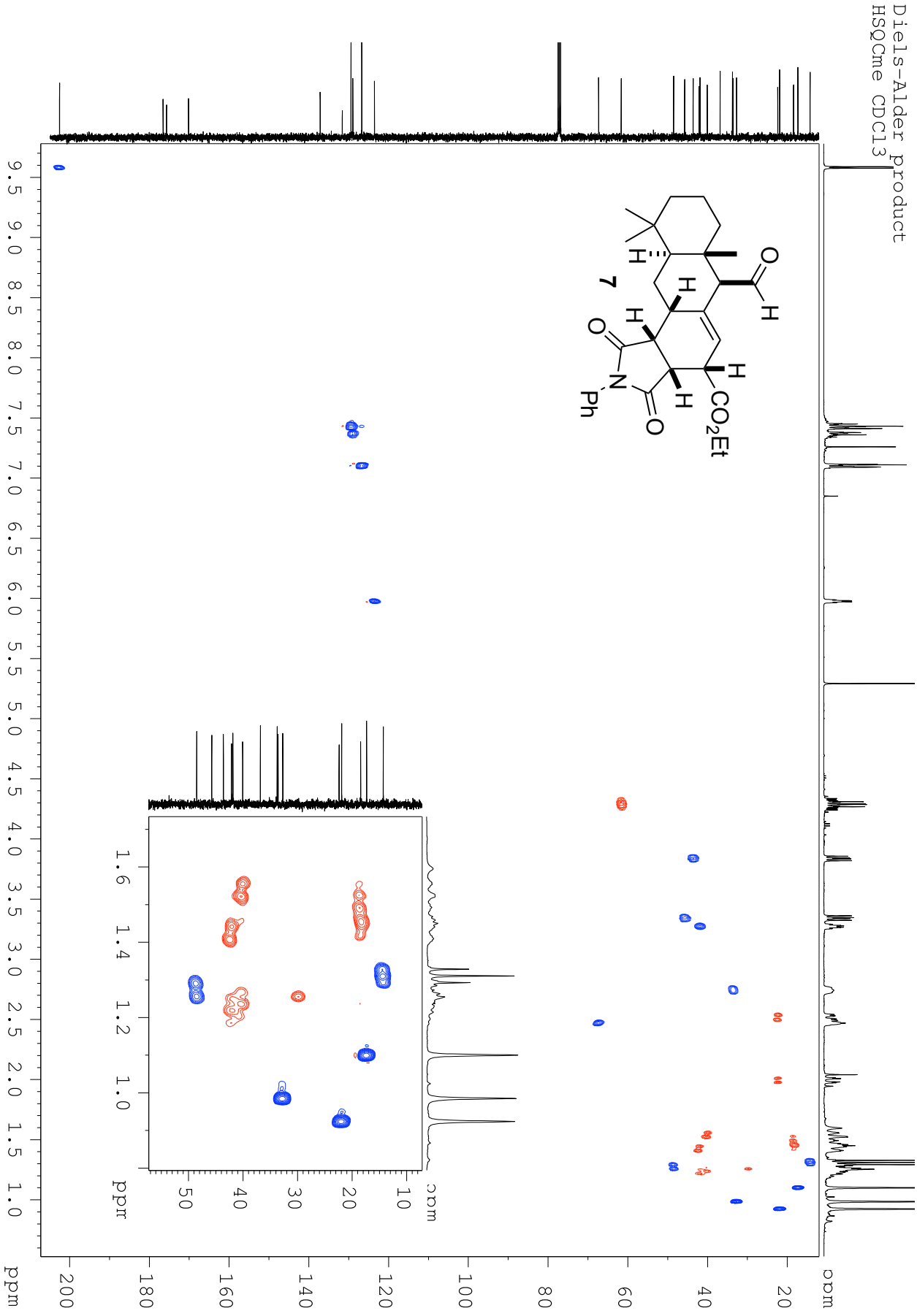
Polygodial Diels–Alder derivative (7) ^{1}H NMR Spectrum



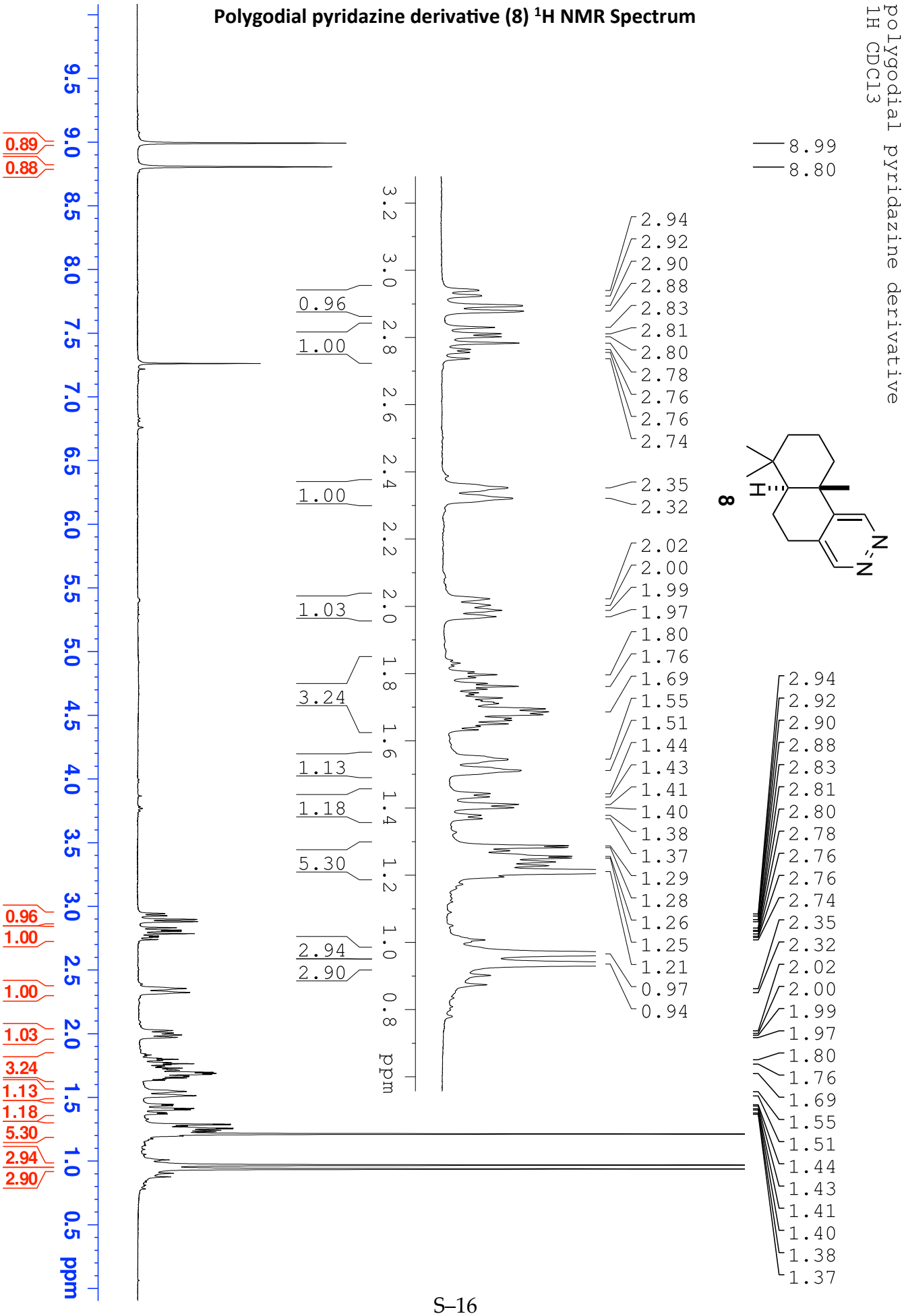
Polygodial Diels–Alder derivative (7) ^{13}C NMR Spectrum



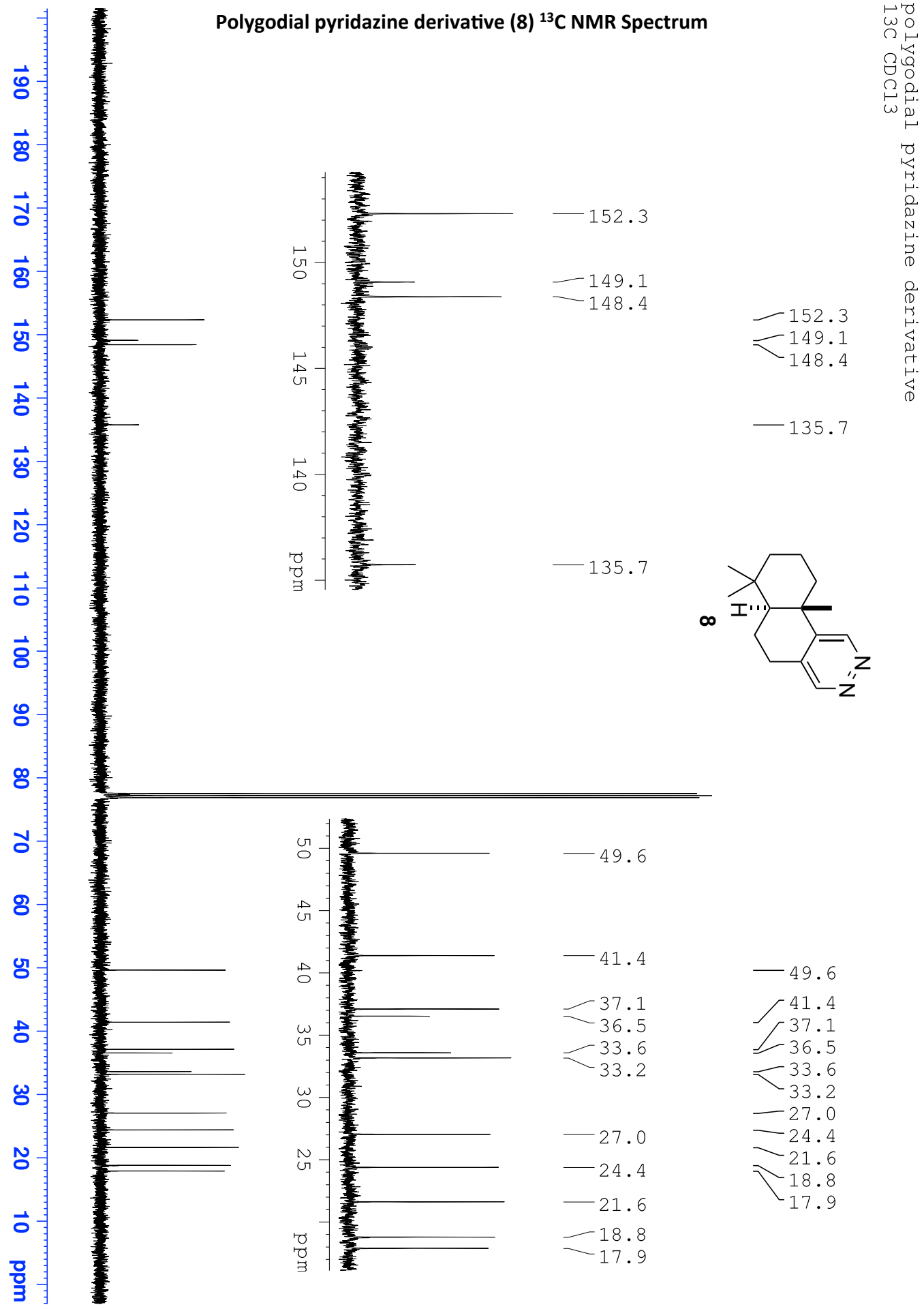
Polygodial Diels–Alder derivative (7) ^{1}H-^{13}C HSQCme NMR Spectrum



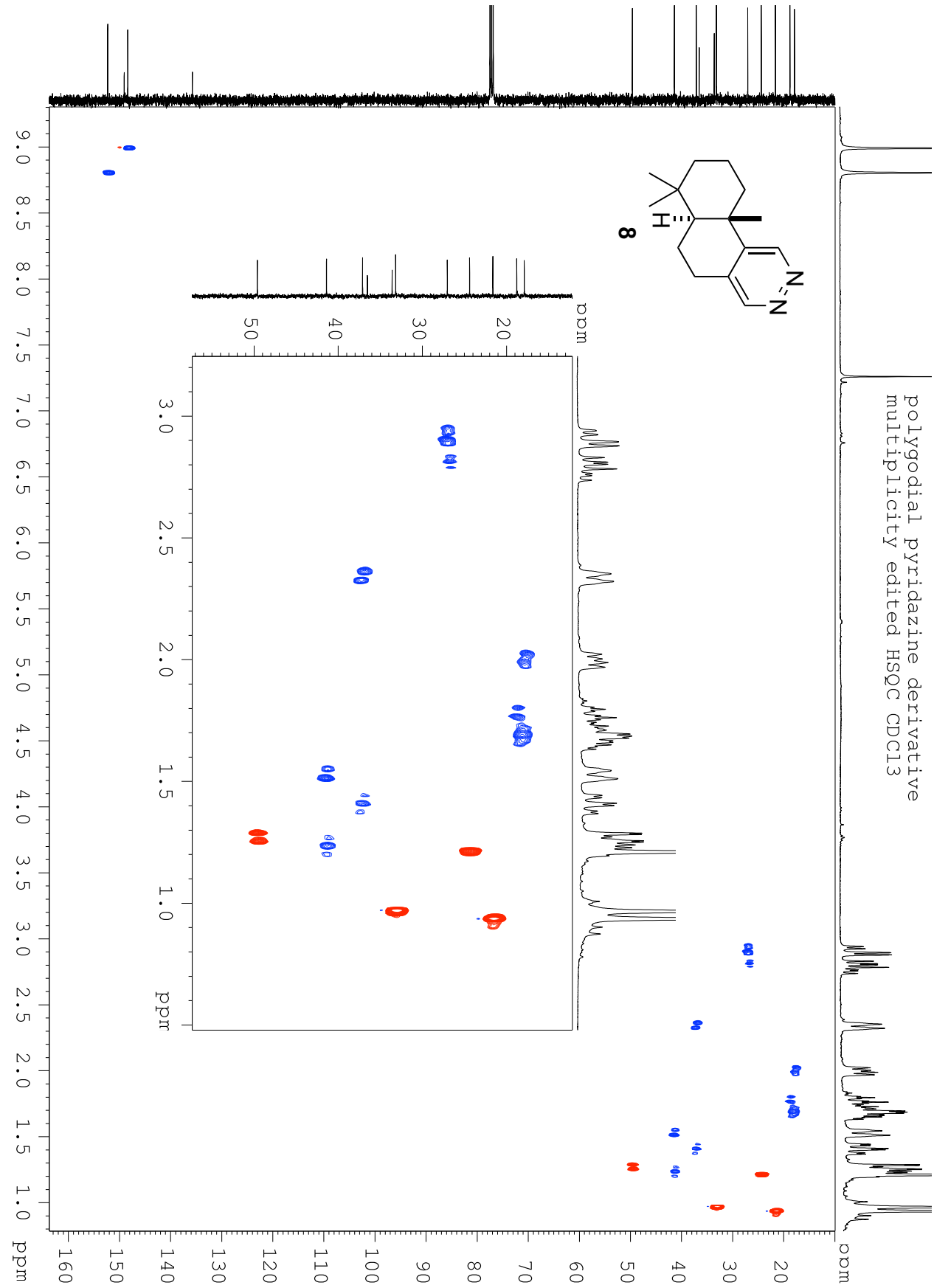
Polygodial pyridazine derivative (8) ^{1}H NMR Spectrum



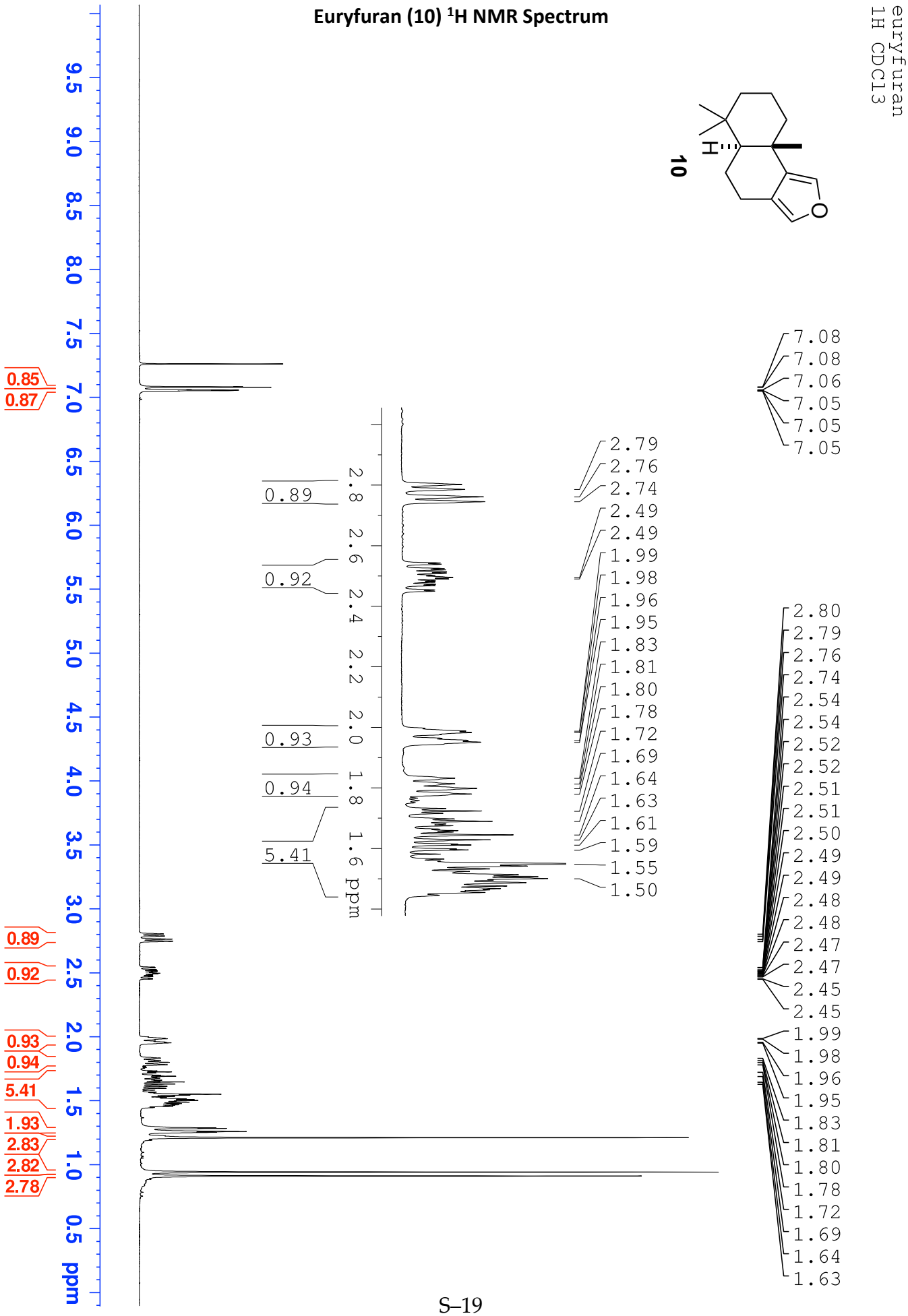
Polygodial pyridazine derivative (8) ^{13}C NMR Spectrum



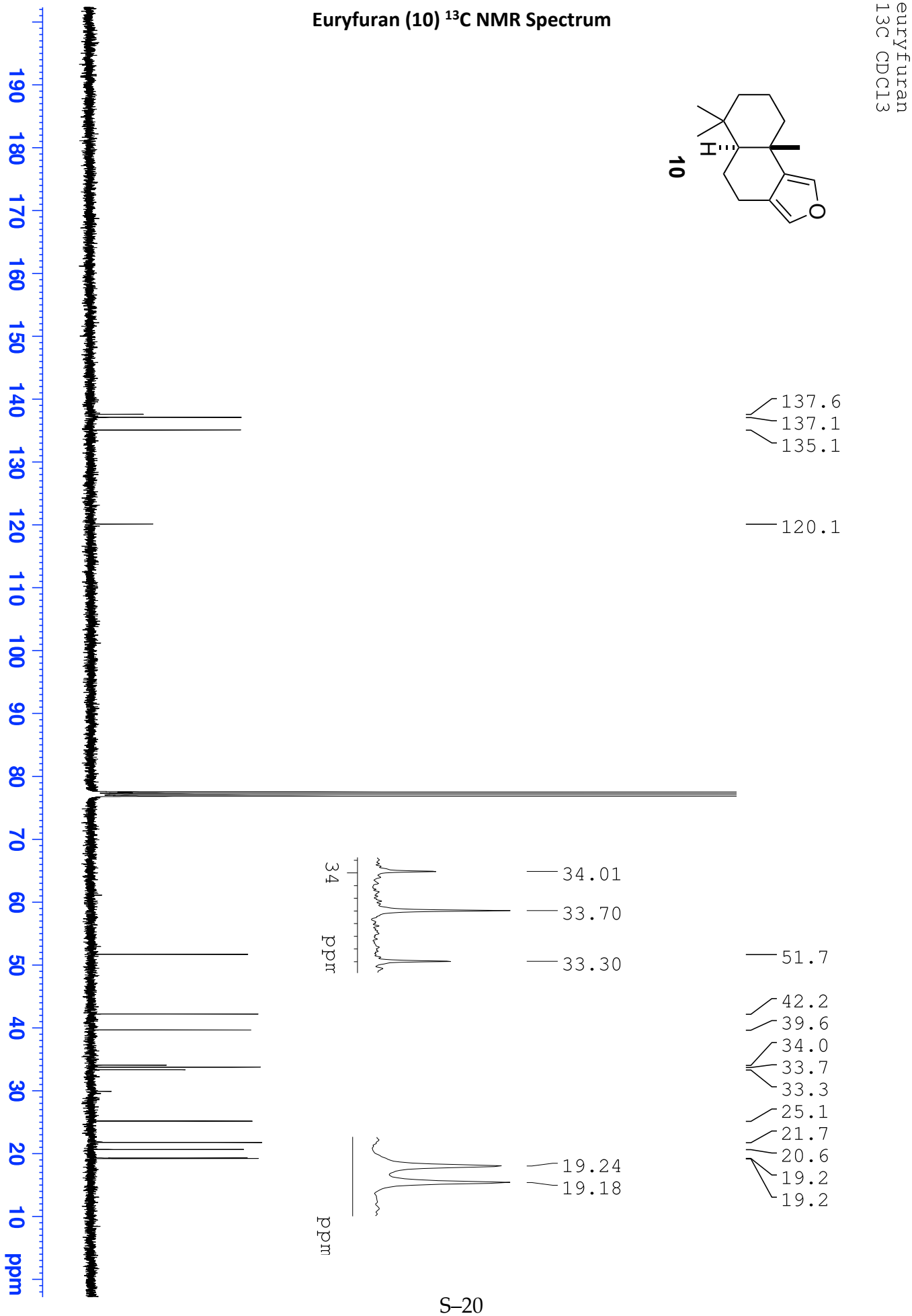
Polygodial pyridazine derivative (8) HSQCme NMR Spectrum



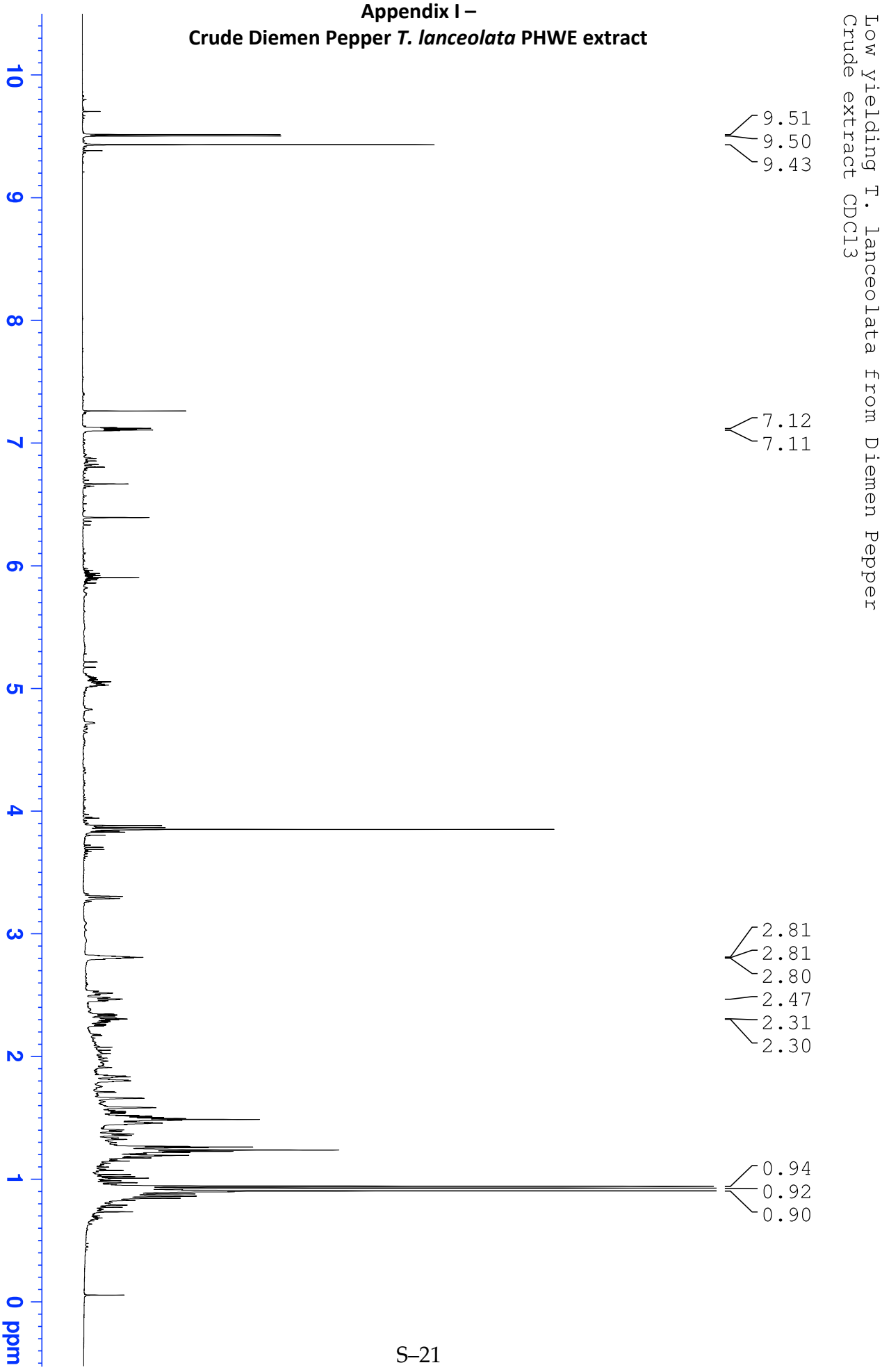
Euryfuran (10) ^{1}H NMR Spectrum



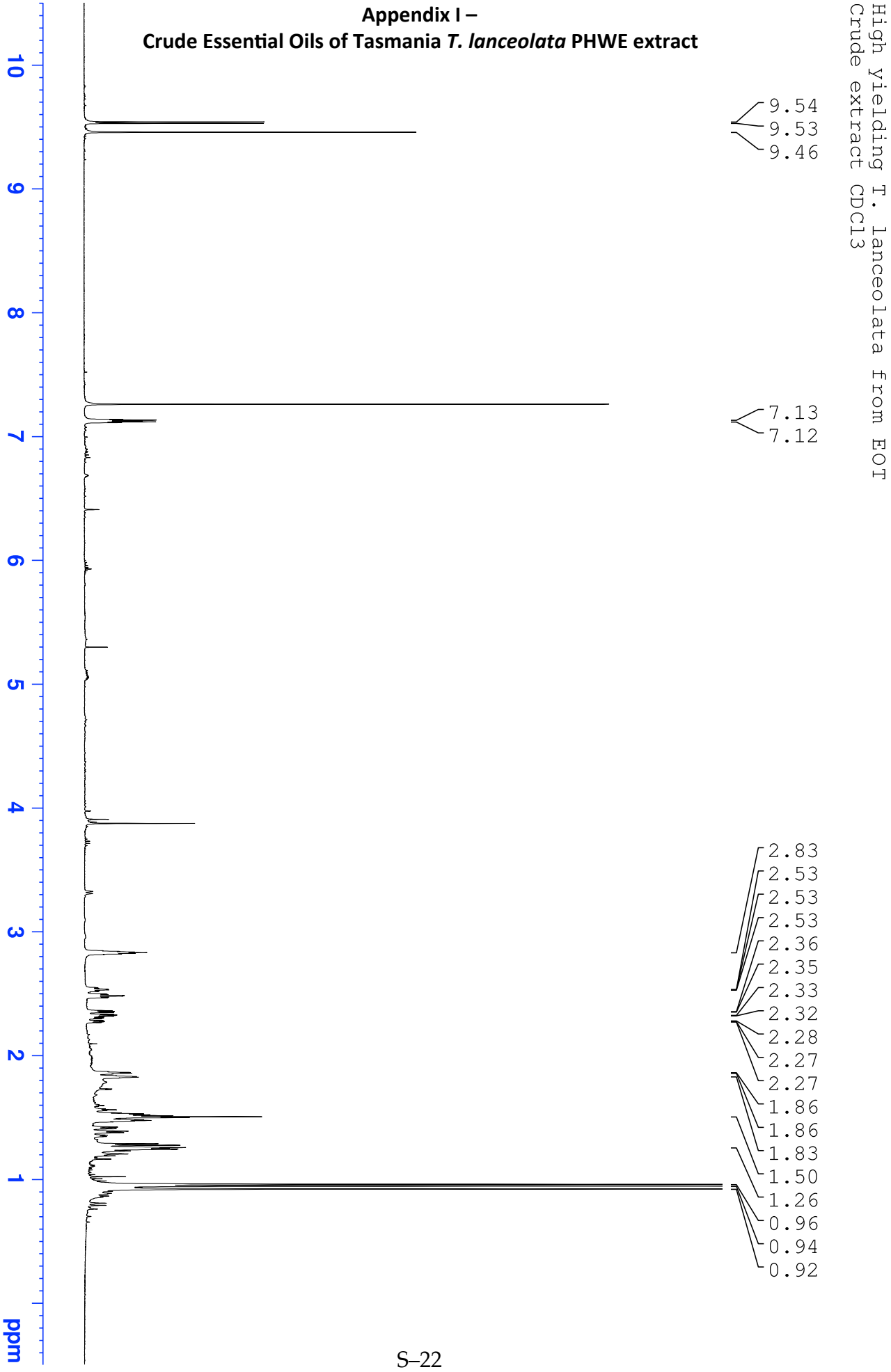
Euryfuran (10) ^{13}C NMR Spectrum



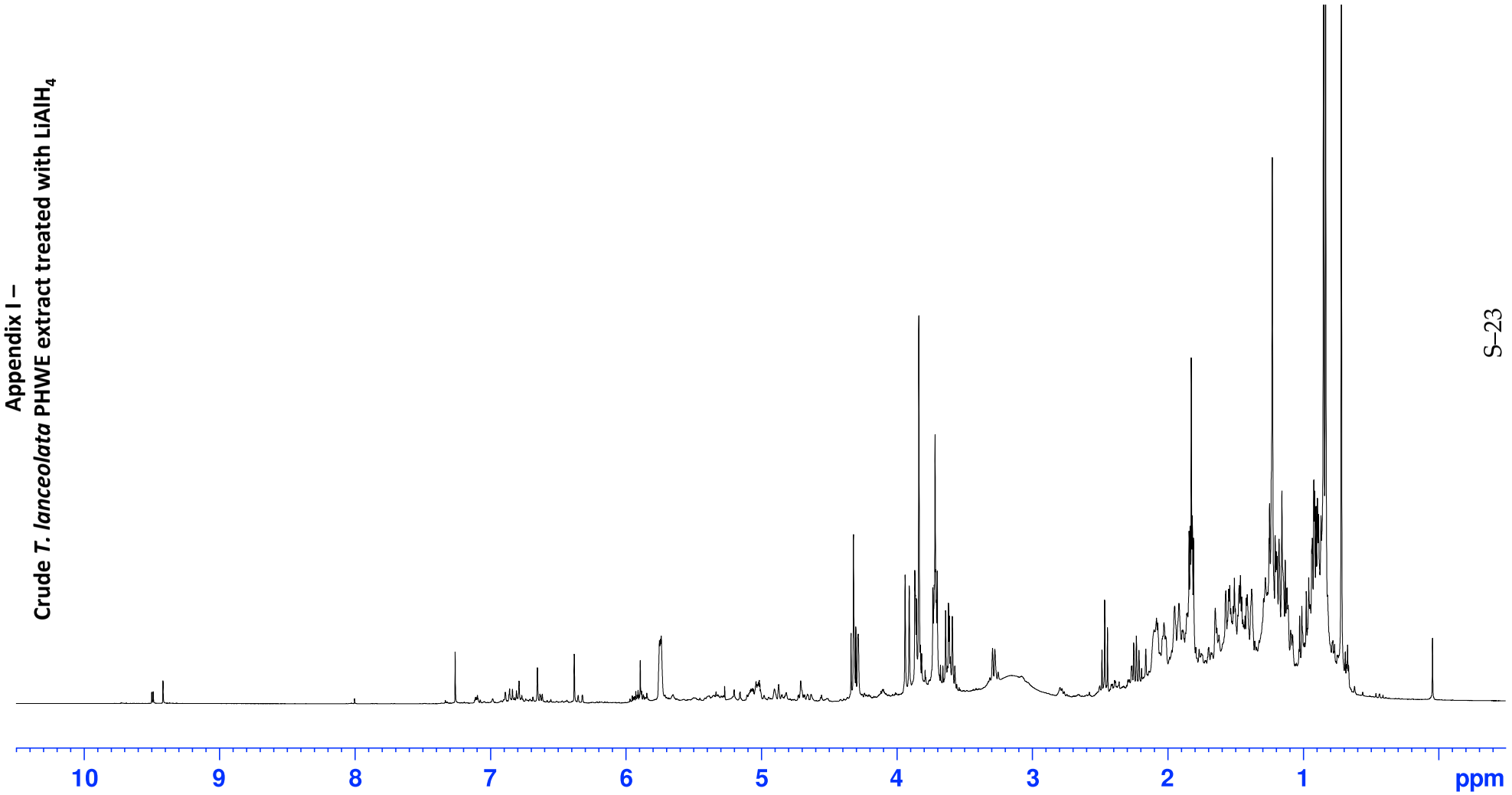
Appendix I –
Crude Diemen Pepper *T. lanceolata* PHWE extract



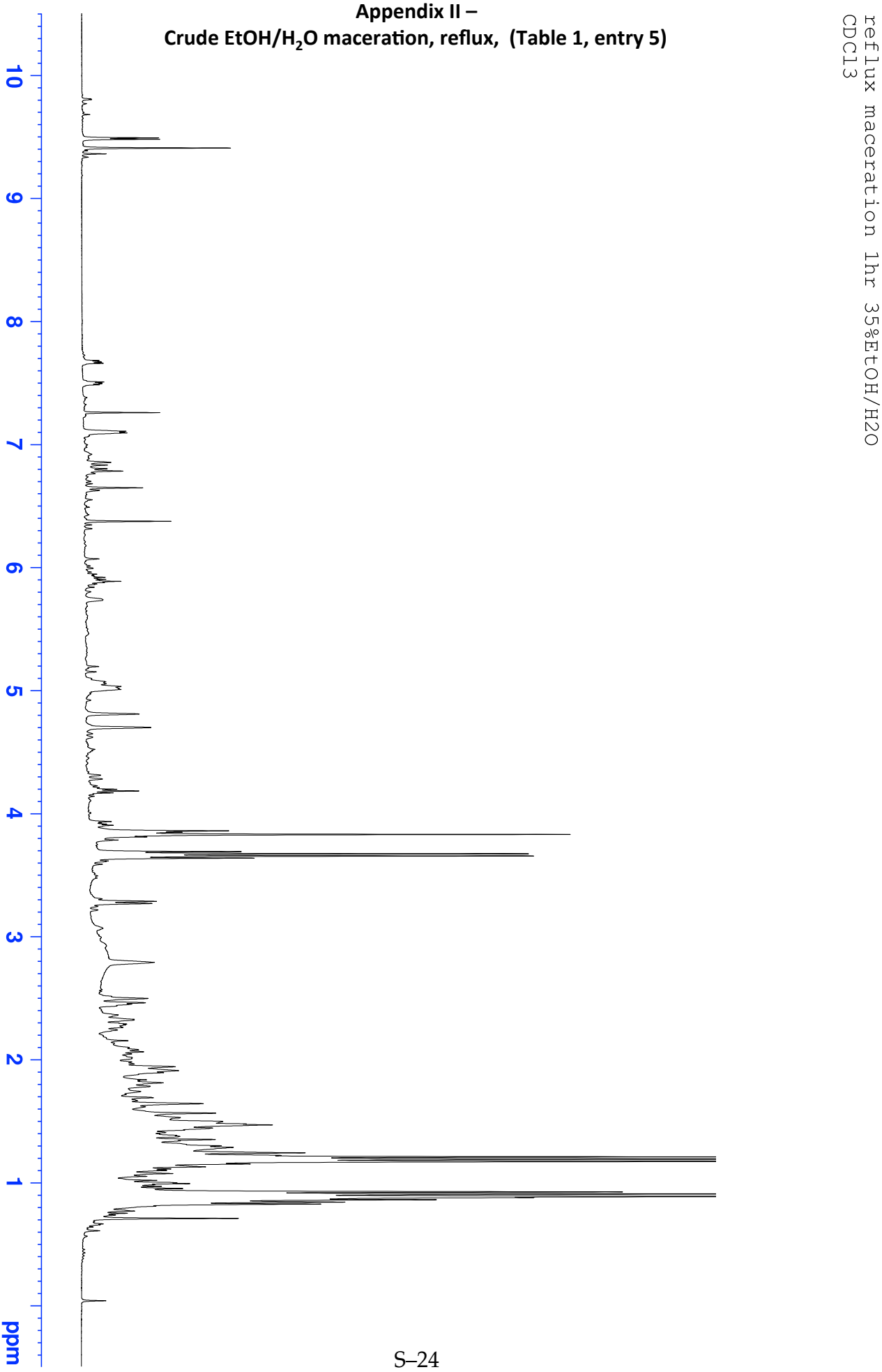
Appendix I –
Crude Essential Oils of Tasmania *T. lanceolata* PHWE extract



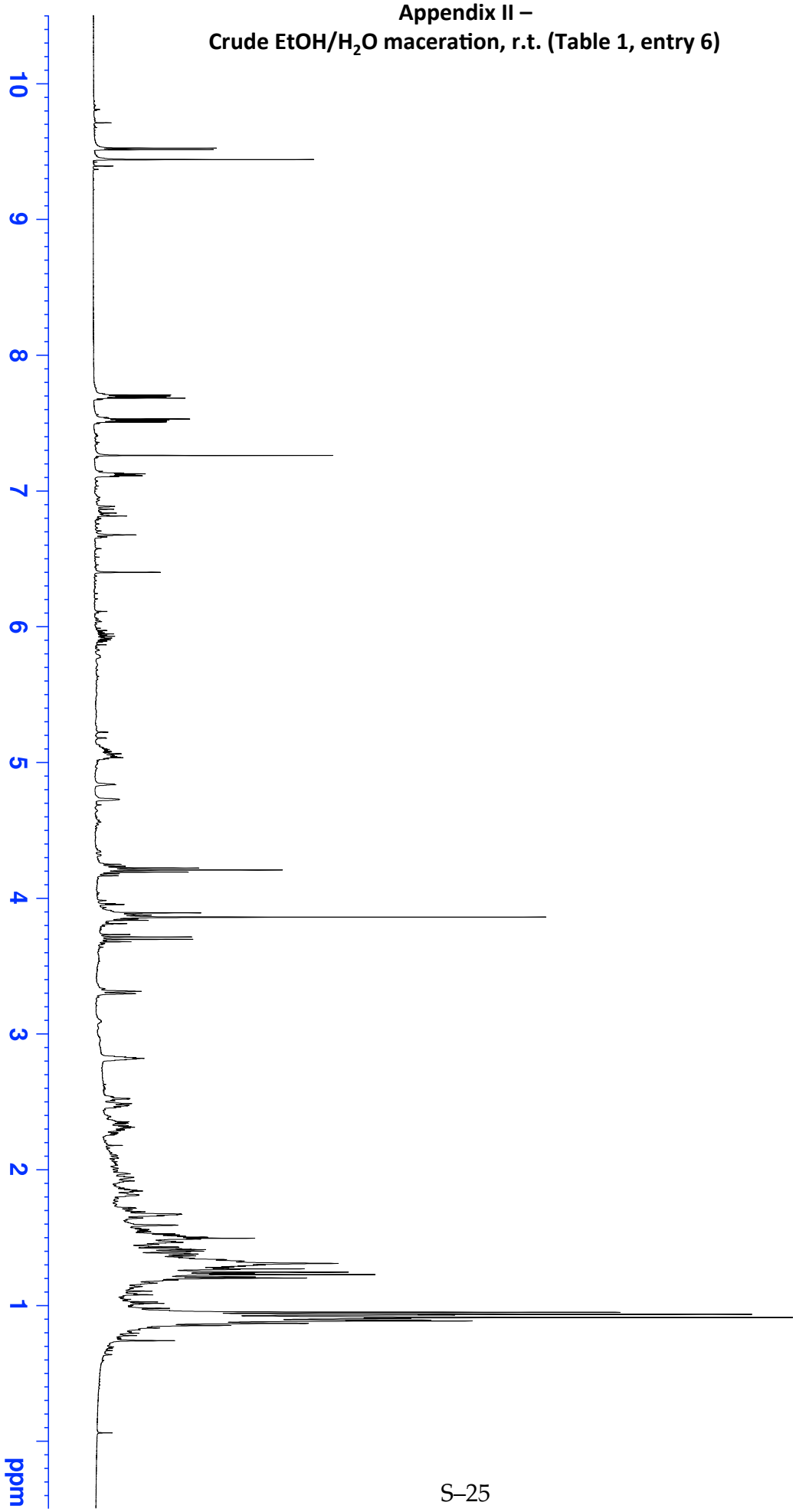
crude T. lanceolata extract treated wih LiAlH4
CDCl3



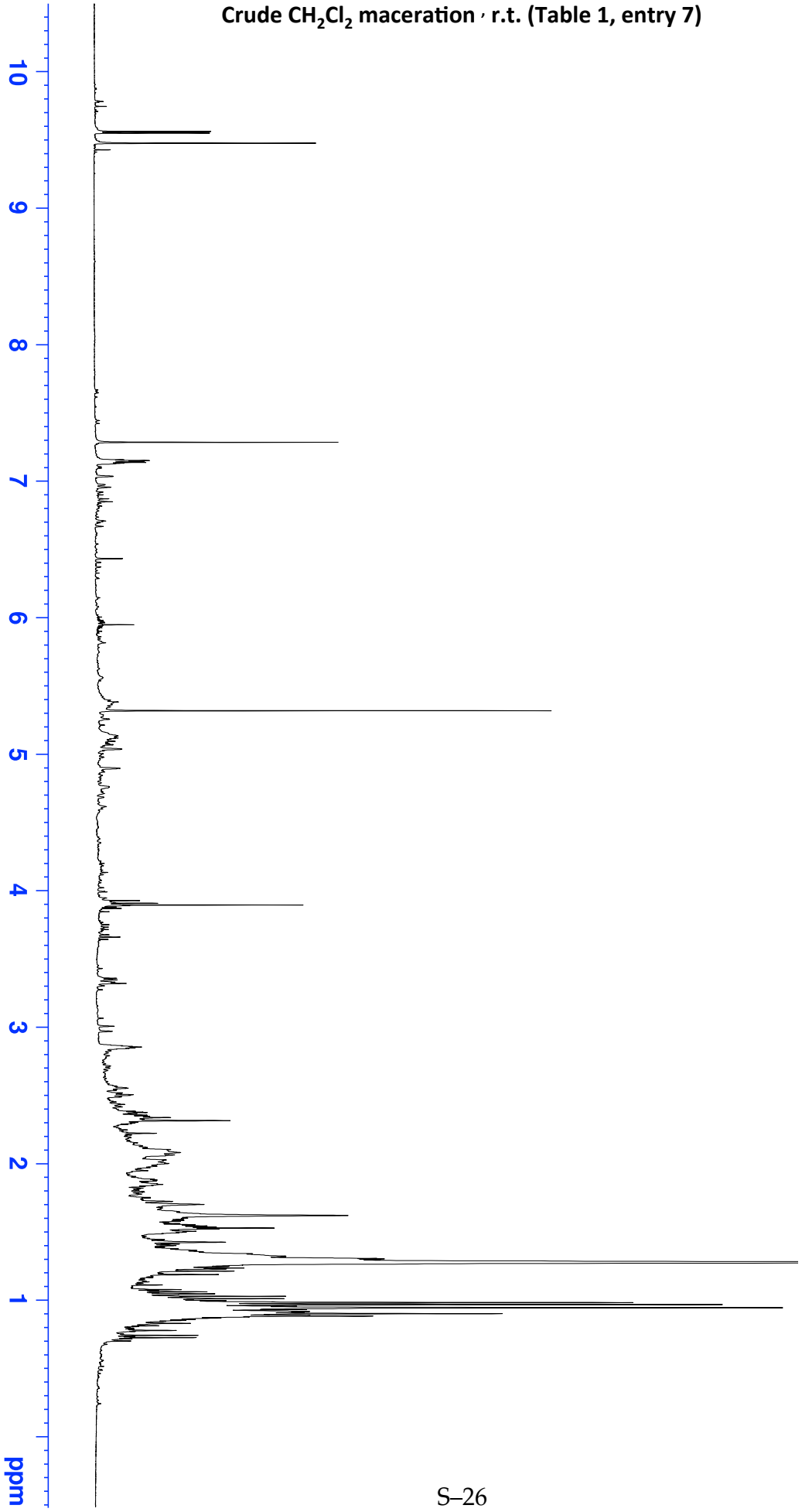
Appendix II –
Crude EtOH/H_2O maceration, reflux, (Table 1, entry 5)



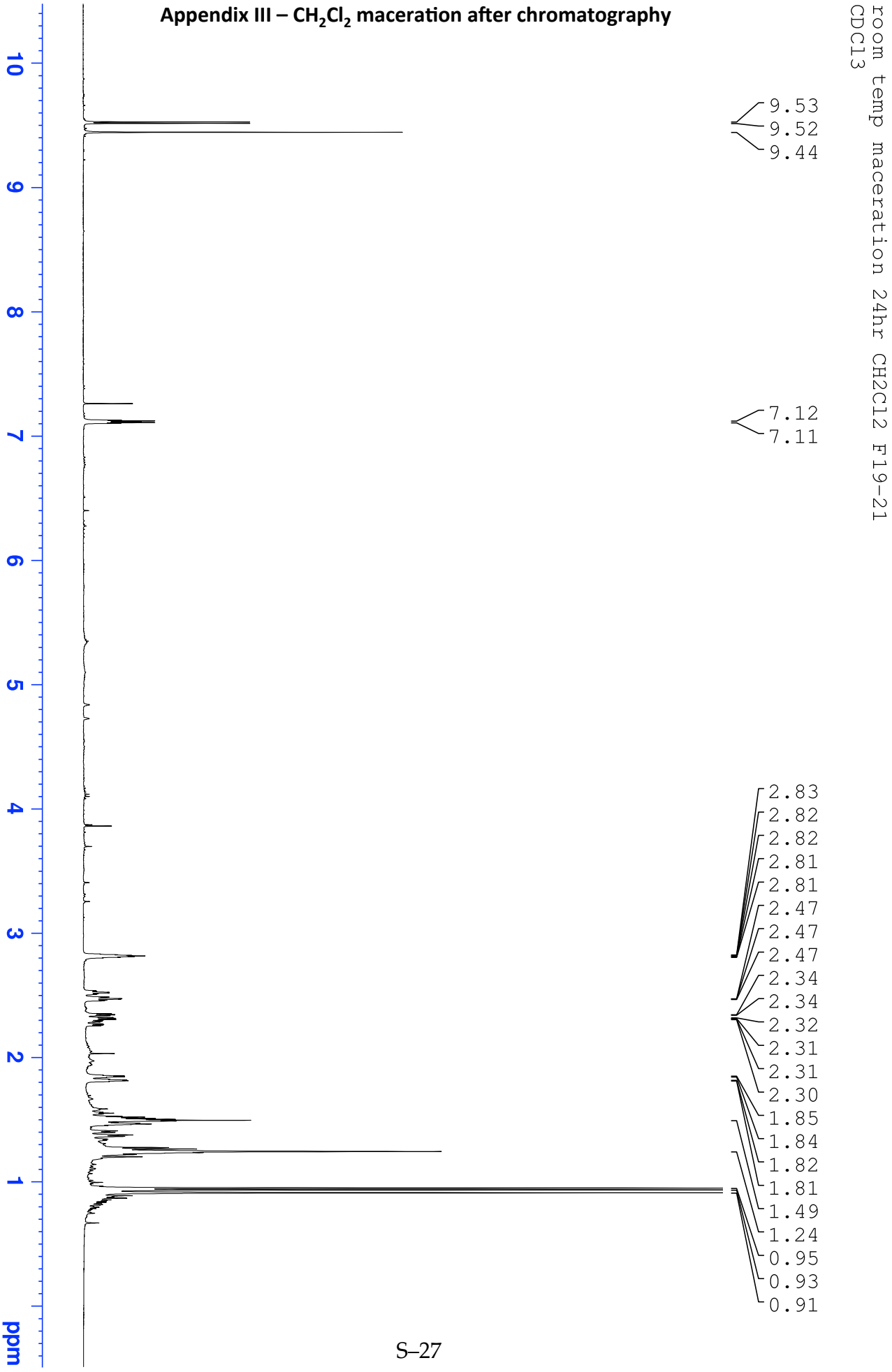
Appendix II –
Crude EtOH/H_2O maceration, r.t. (Table 1, entry 6)



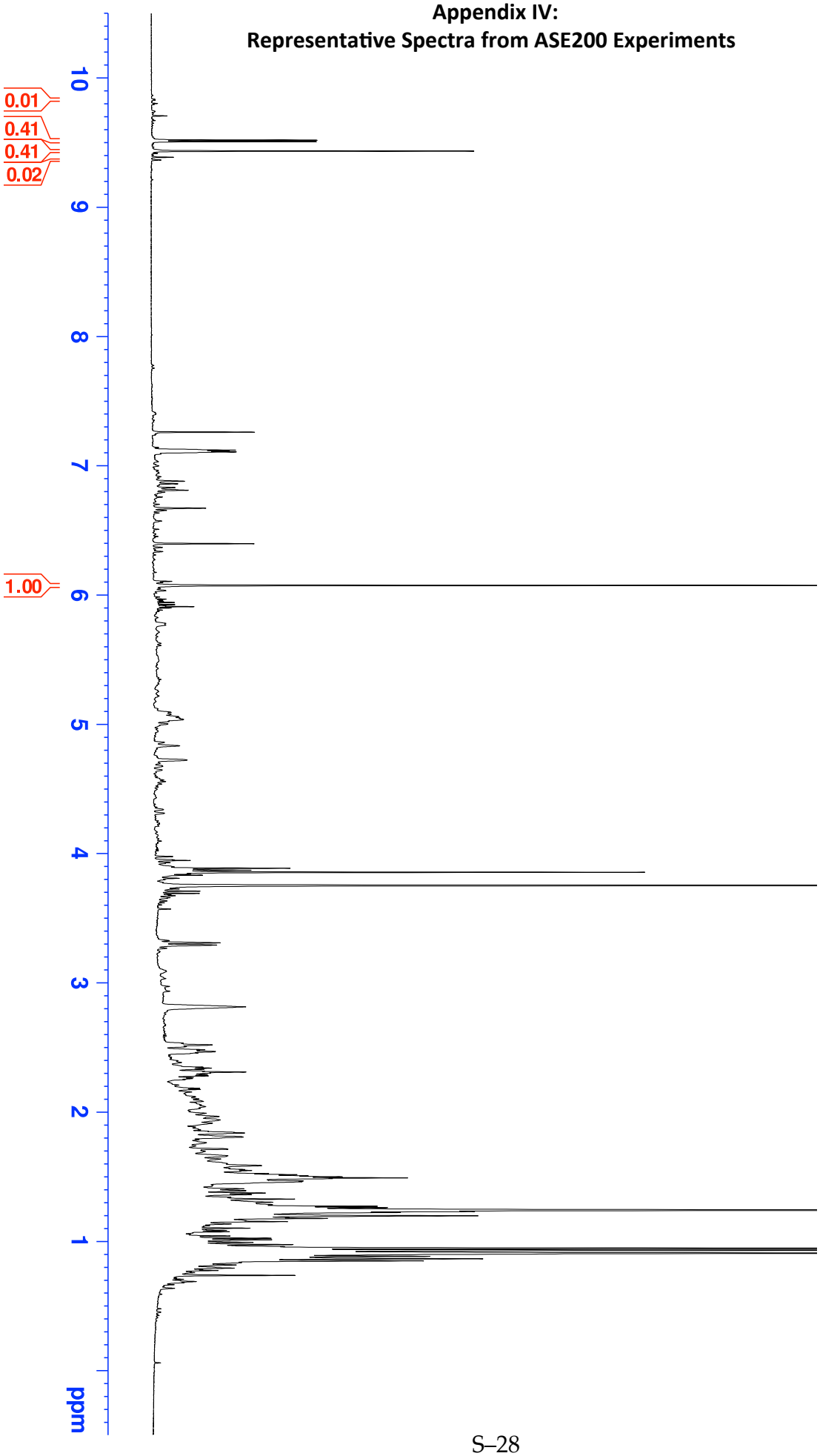
Appendix III –
Crude CH_2Cl_2 maceration , r.t. (Table 1, entry 7)



Appendix III – CH_2Cl_2 maceration after chromatography



Appendix IV:
Representative Spectra from ASE200 Experiments



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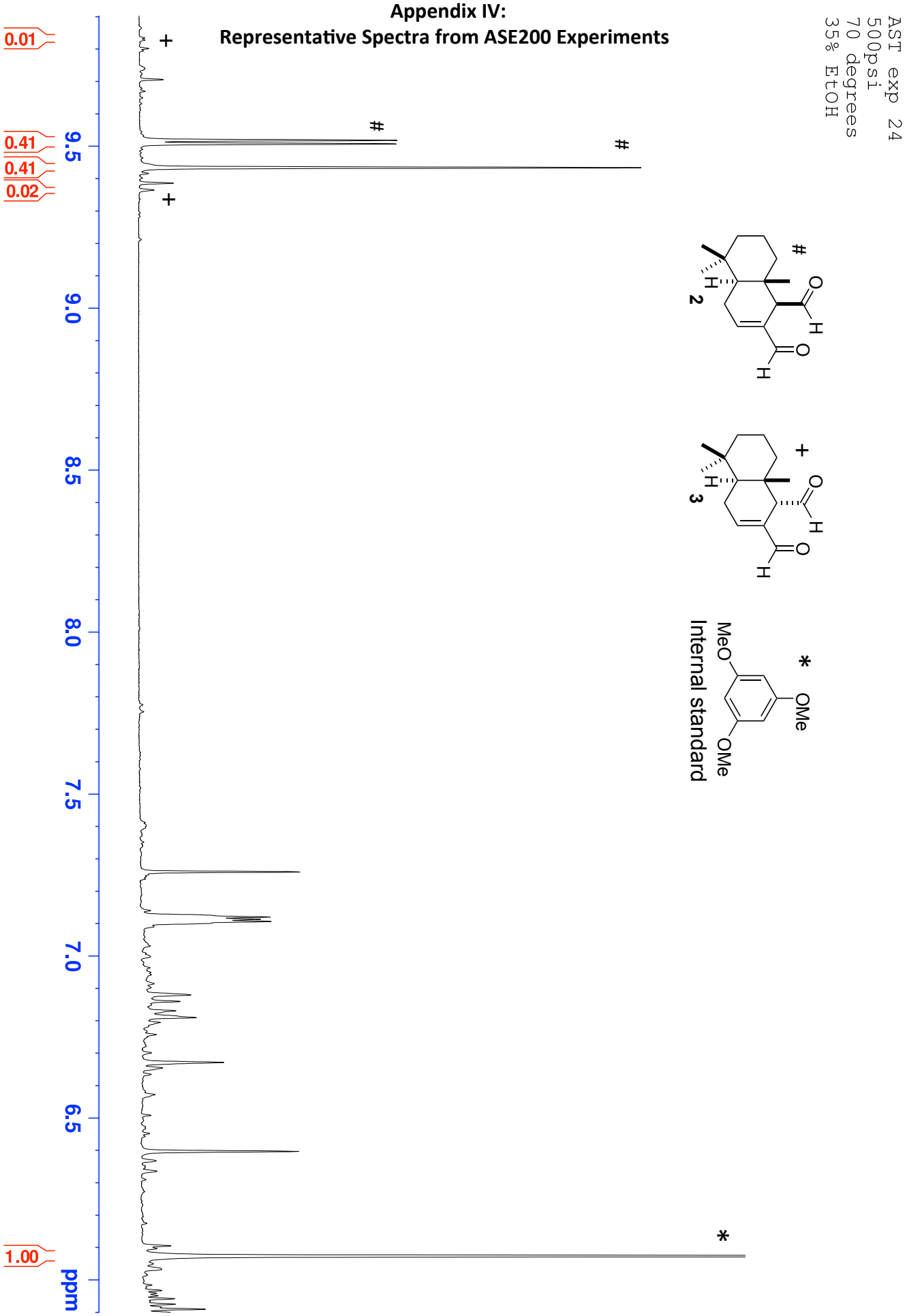

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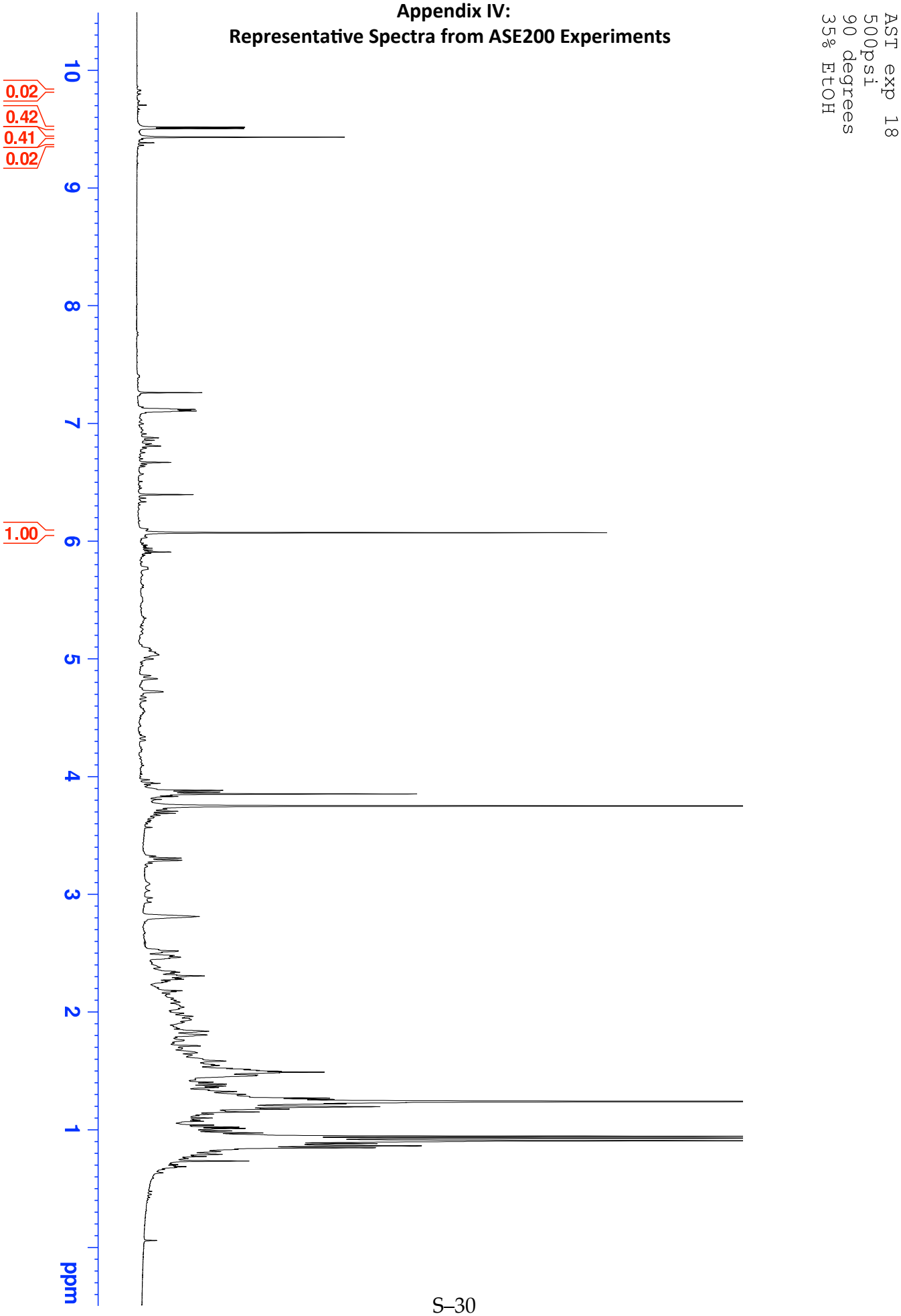

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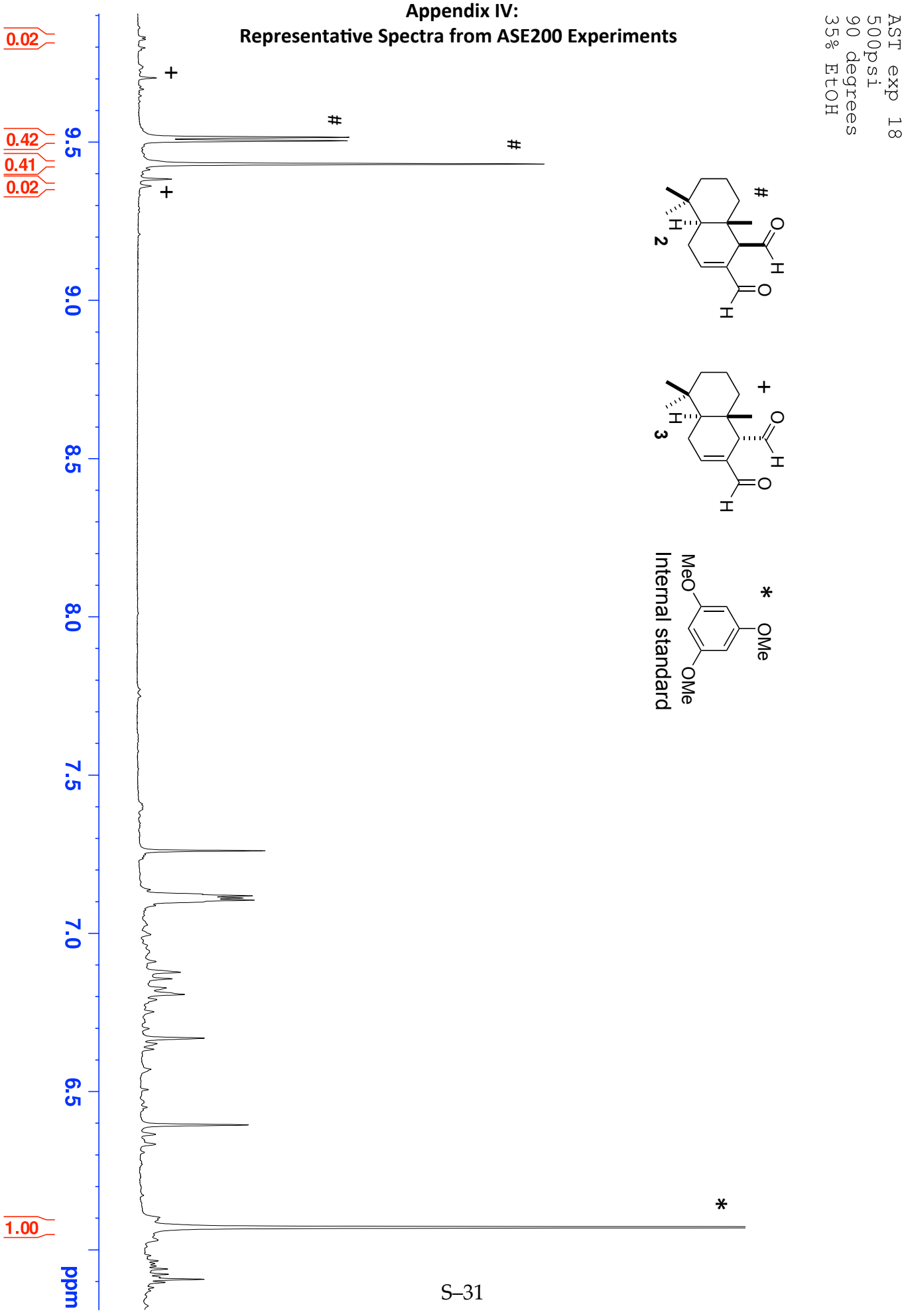

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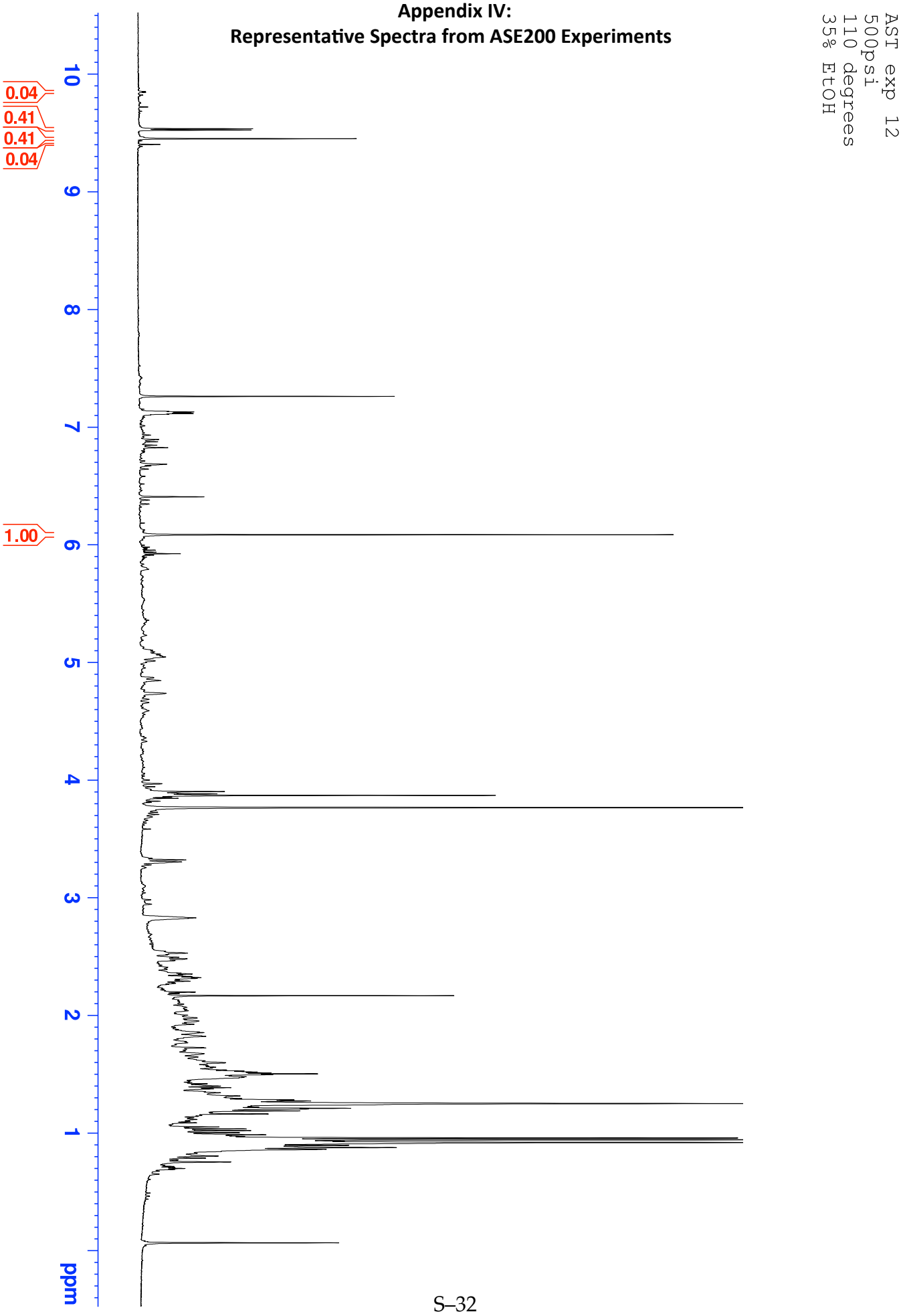

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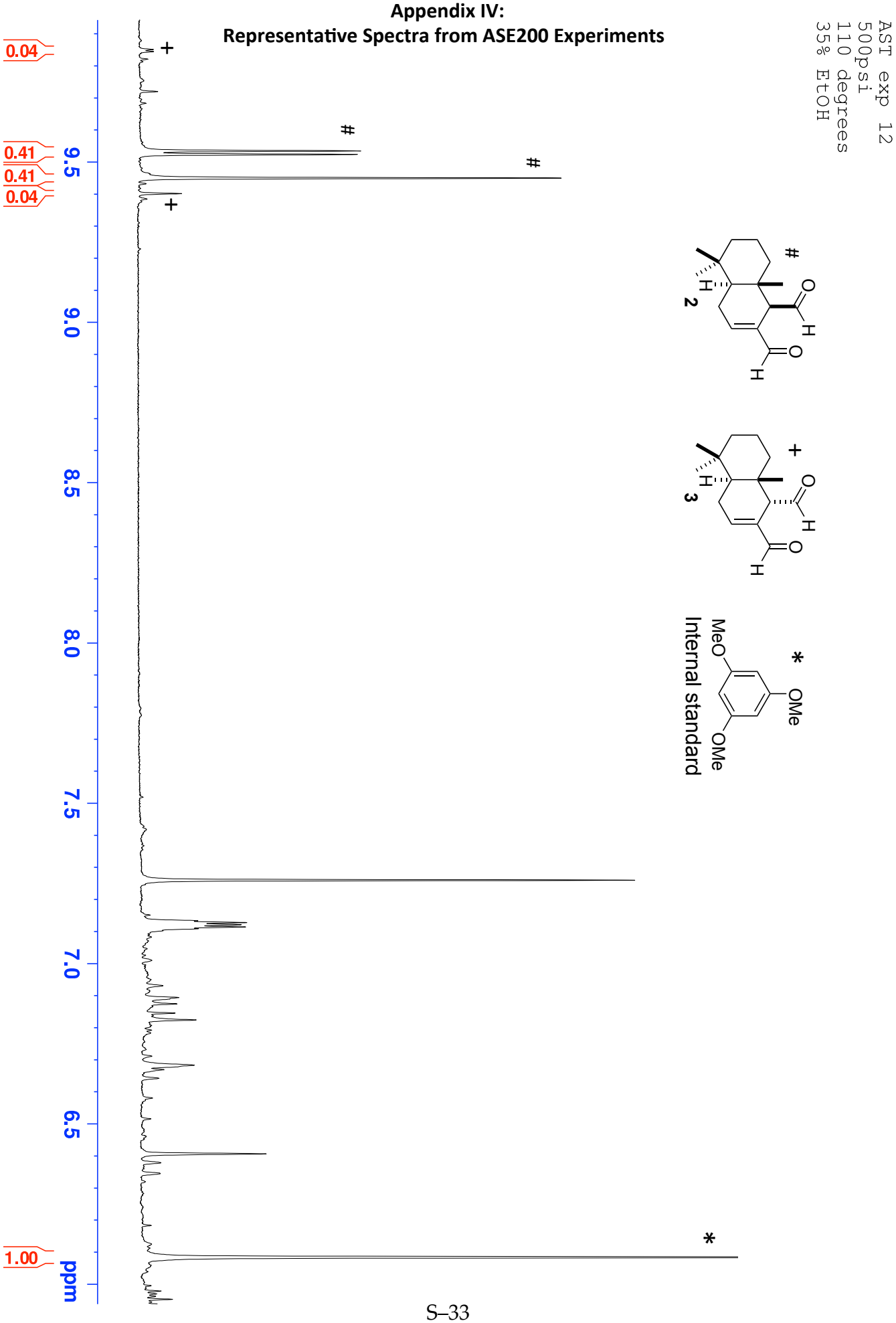

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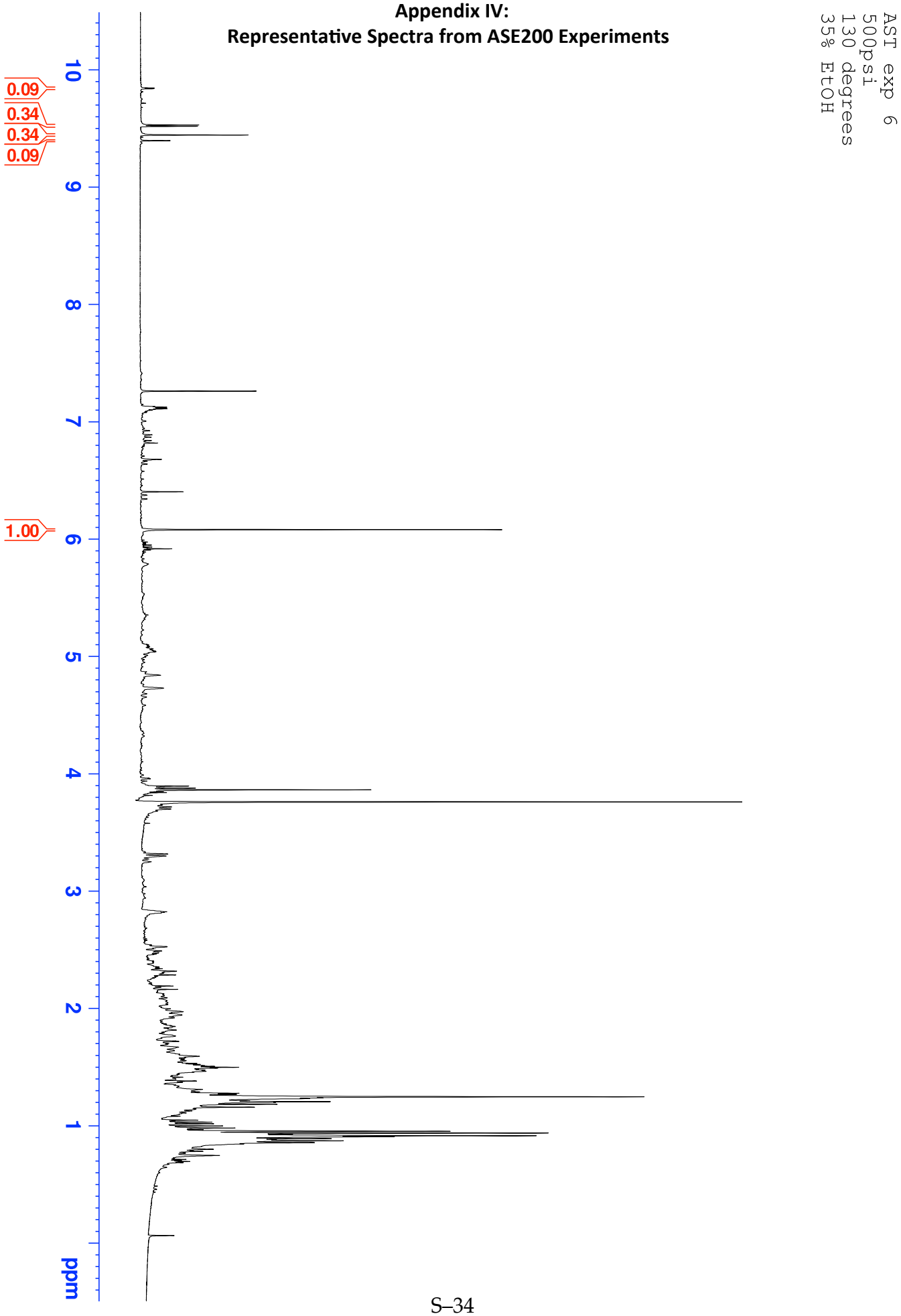

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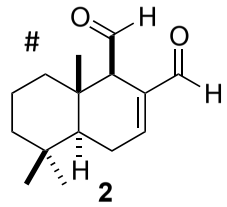
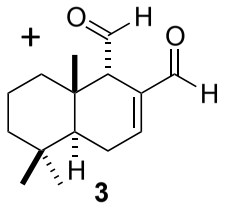
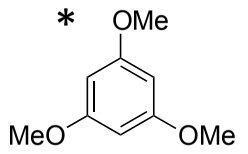
35% EtOH







Internal standard

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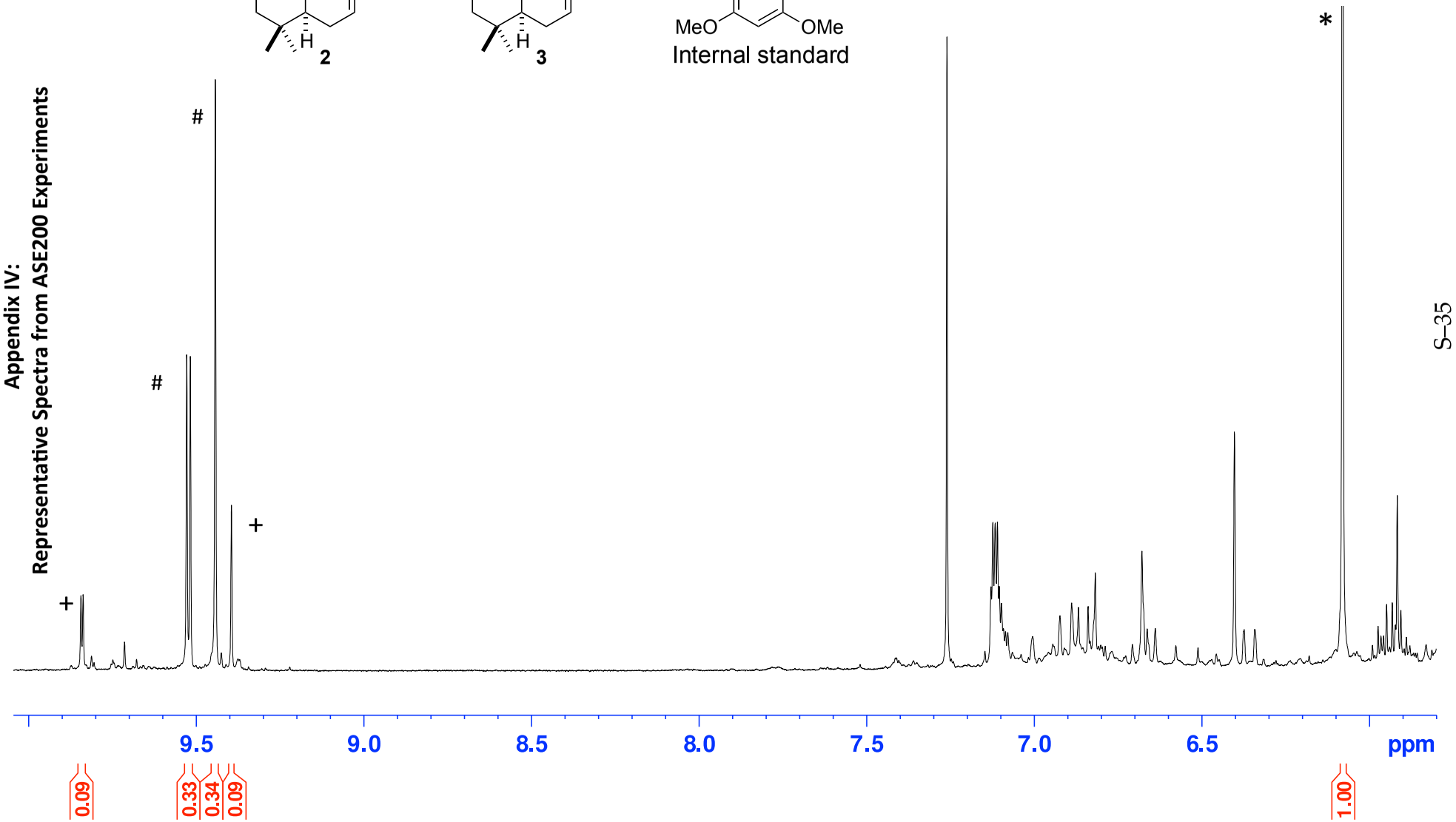
Representative Spectra from ASE200 Experiments



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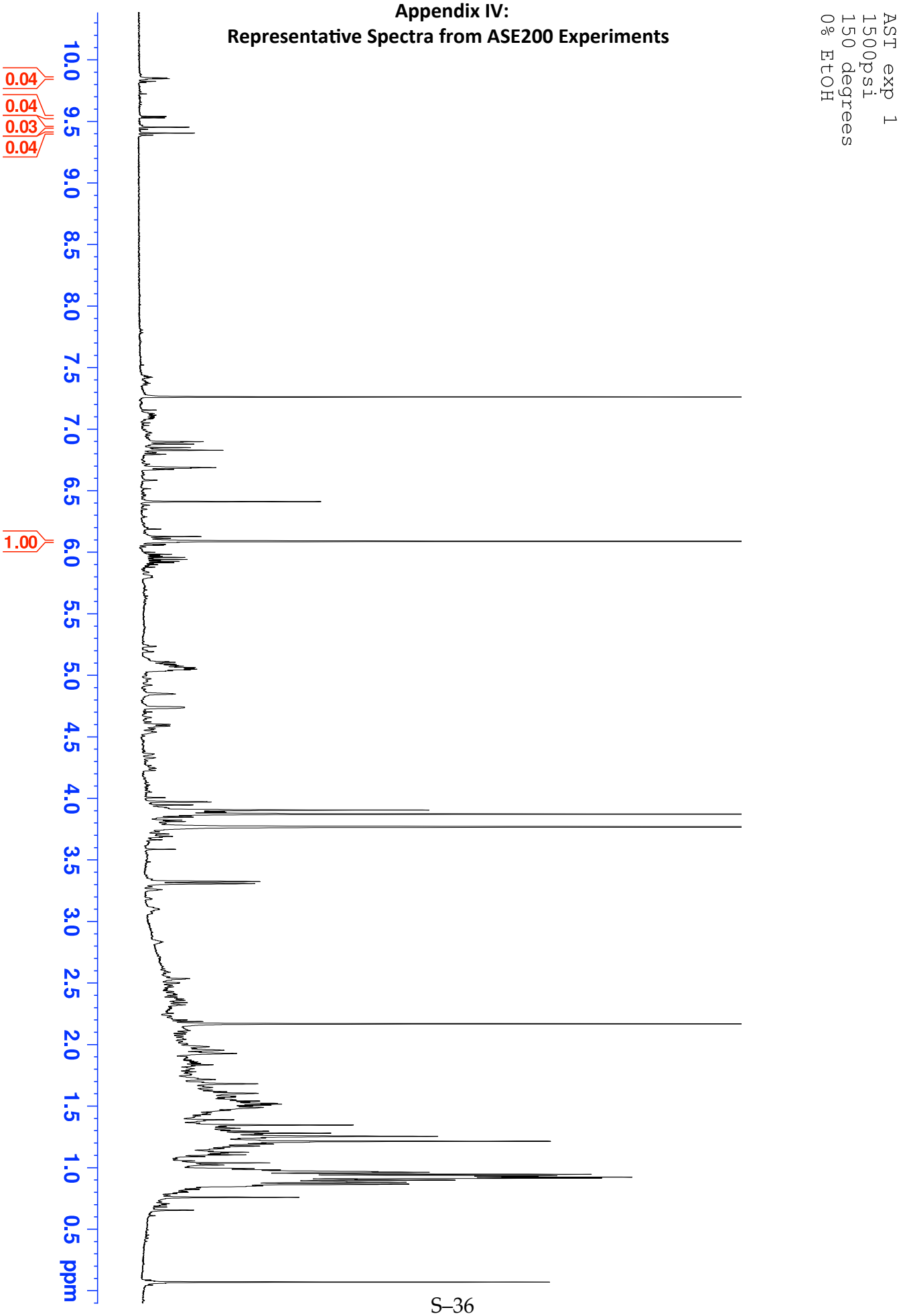

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