Supporting Information — Spectra

Manuscript: Towards the Enantioselective Synthesis of (-)-Euonyminol – Preparation of a Fully Functionalised Lower-rim Model

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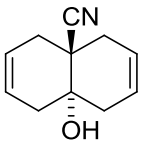
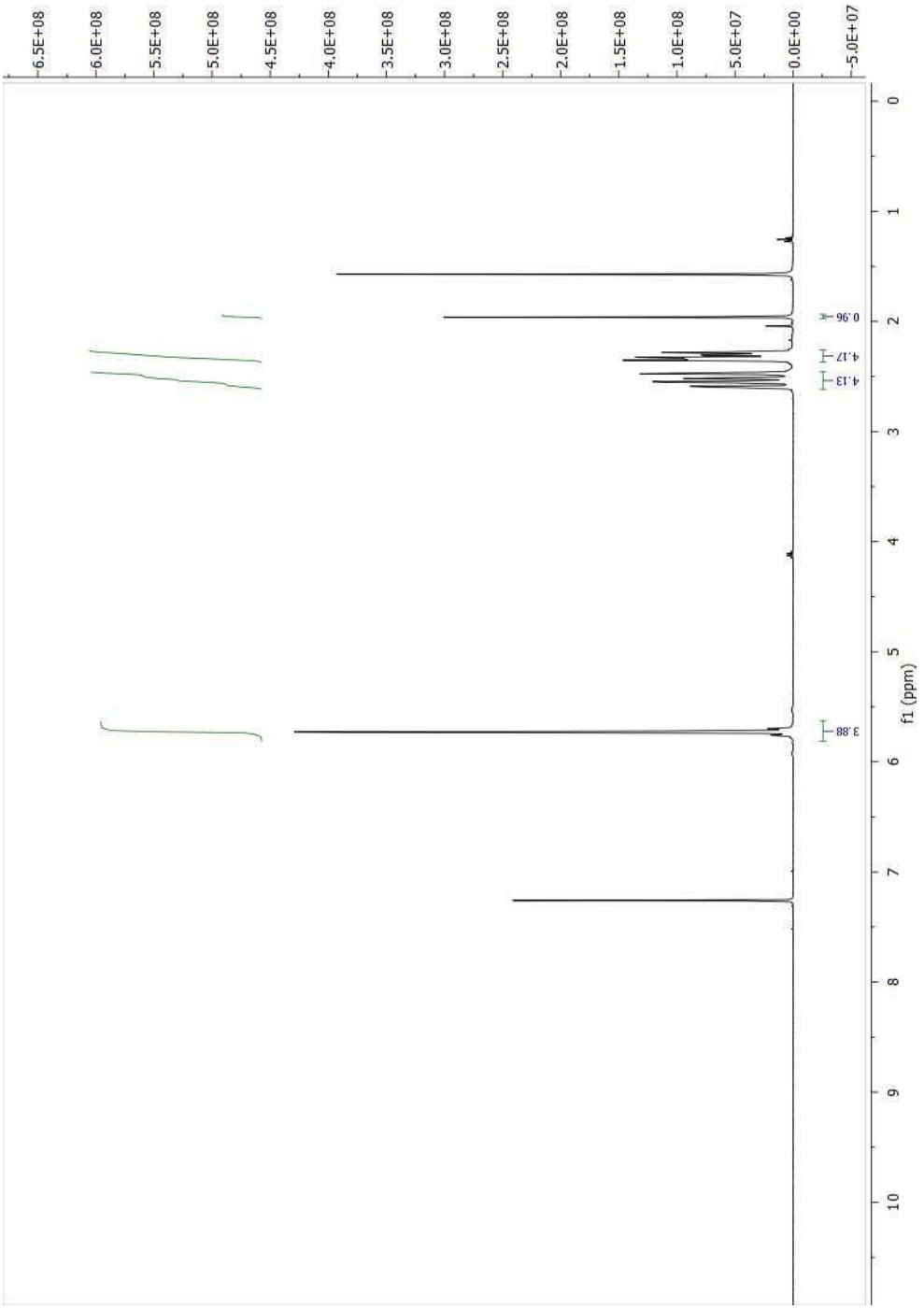
General Procedures:

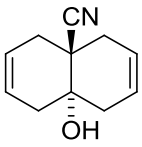
All reactions were performed under anhydrous conditions and an atmosphere of nitrogen in oven-dried glassware. Yields refer to chromatographically and spectroscopically (^{1}H NMR) homogenous materials. Reagents were used as obtained from commercial sources or purified according to known procedures. Flash chromatography was carried out using Merck Kiesegel 60 F_{254} (230-400 mesh) silica gel. Only distilled solvents were used as eluents. Thin layer chromatography (TLC) was performed on Merck DC-Alufolien or glass plates pre-coated with silica gel 60 F_{254} which were visualised either by quenching of ultraviolet fluorescence (λ_{max} = 254 nm) or by charring with 10% $KMnO_4$ in 0.1 M NaOH. All reaction solvents were distilled before use and stored over activated 4 Å molecular sieves, unless otherwise indicated. Anhydrous CH_2Cl_2 was obtained by refluxing over calcium hydride. Petrol refers to the fraction of light petroleum boiling between 40-60°C. High Resolution Mass Spectrometry (HRMS) measurements are valid to ±5 ppm. Optical rotation measurements are not reported for synthetic compounds derived from allylic alcohol (-)-**3** as the material used for this development work was of variable *ee* (see footnote to procedure for lactate ester **27**).

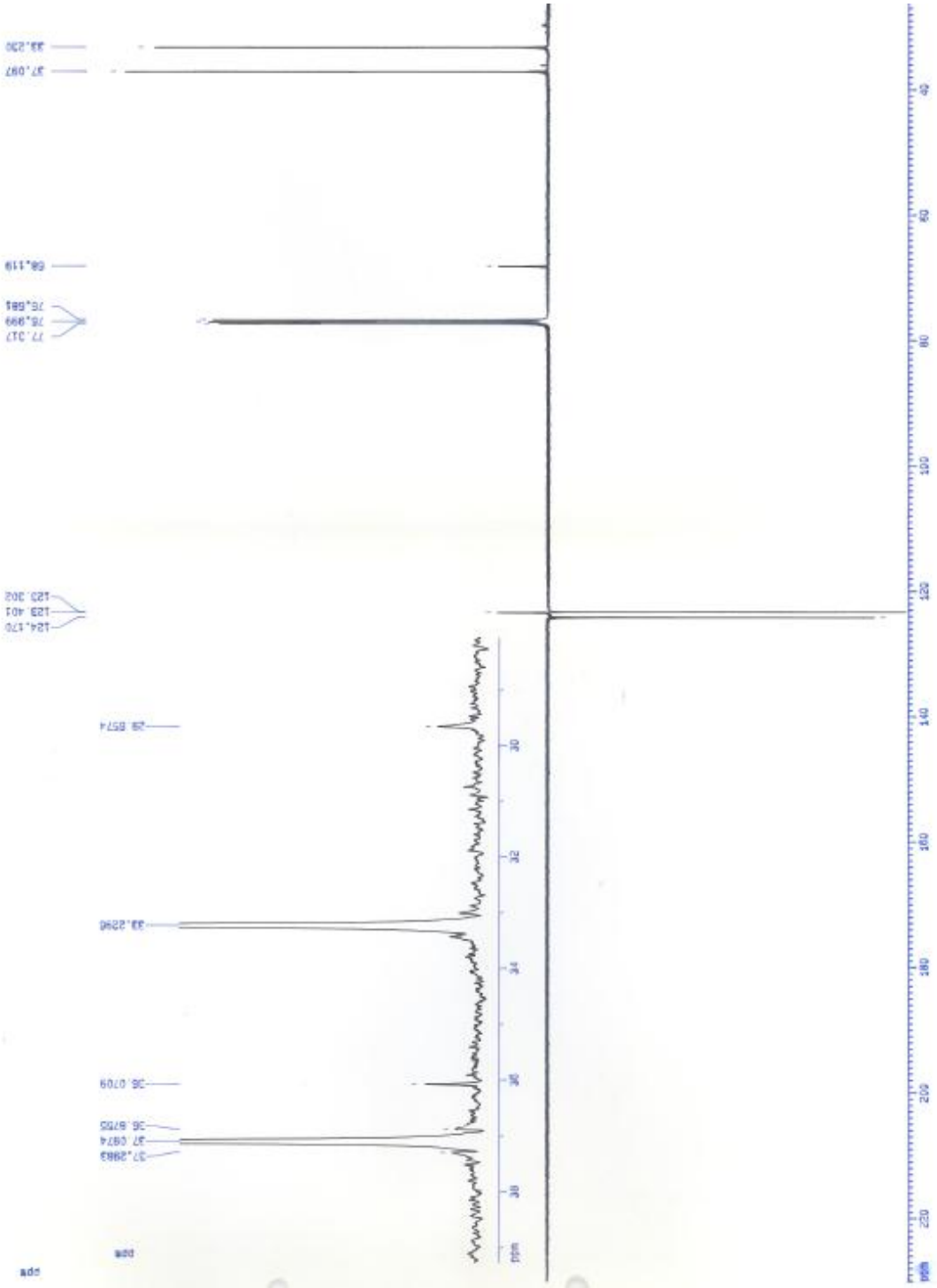
Cyanohydrin 6 – ^{1}H NMR (400 MHz)



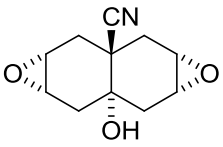


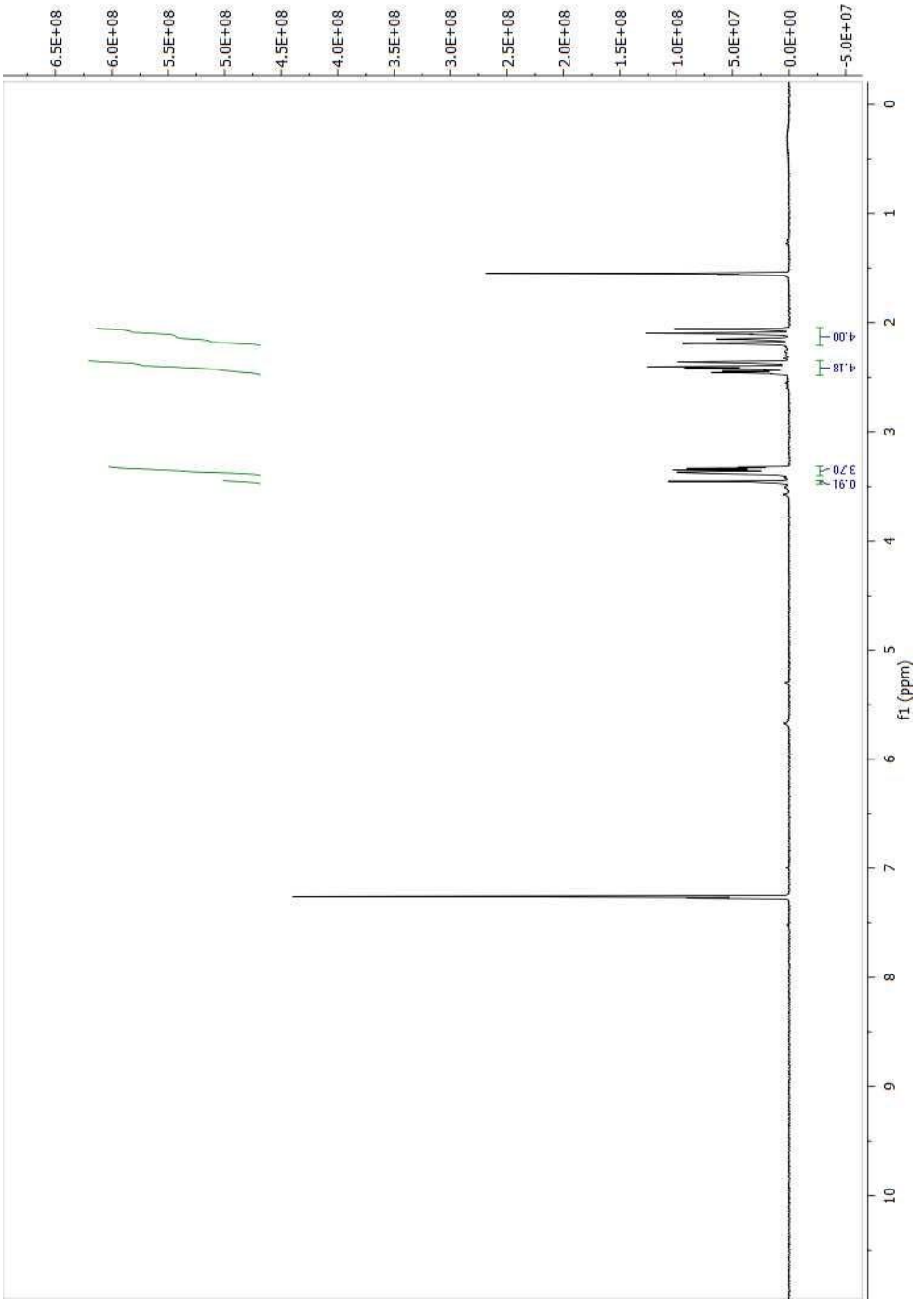
Cyanohydrin 6 – ^{13}C NMR (63 MHz)



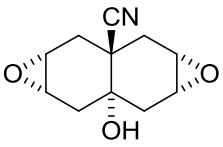


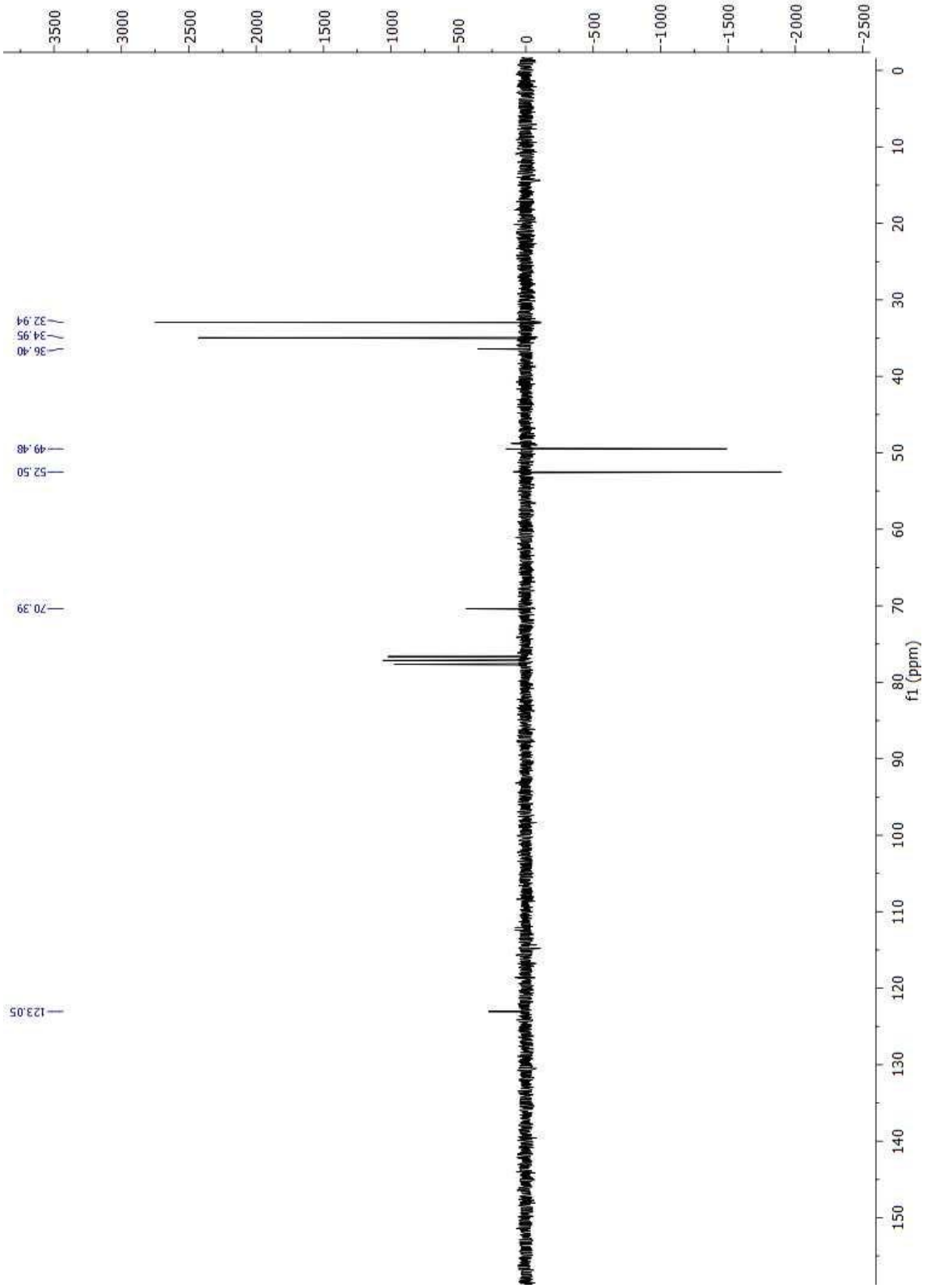
bis-Epoxide 7 – ^{1}H NMR (400 MHz)



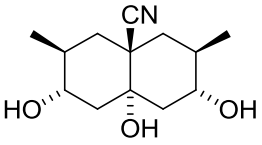


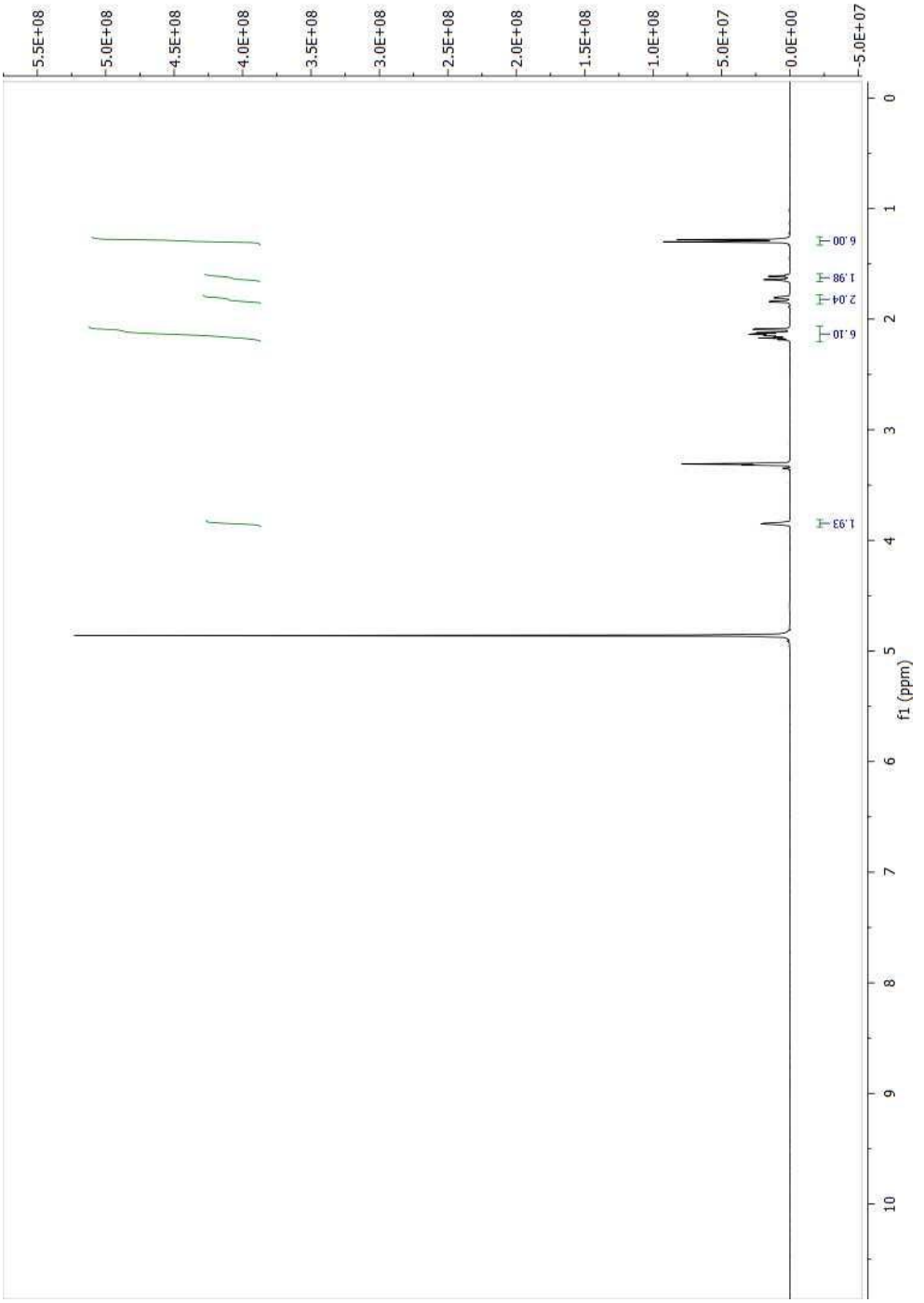
bis-Epoxide 7 – ^{13}C NMR (100 MHz)



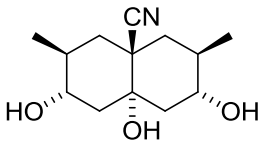


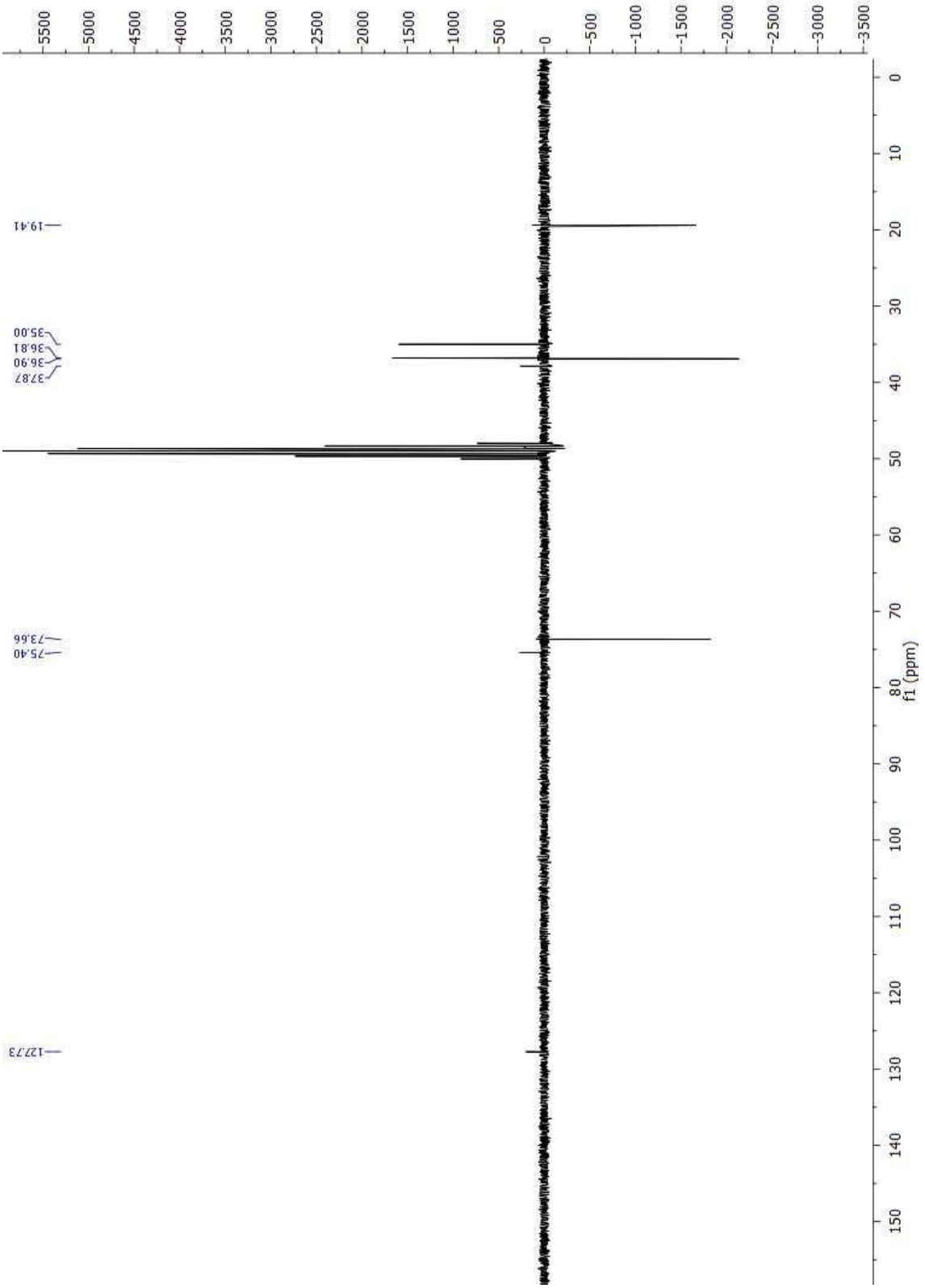
Triol 8 – ^{1}H NMR (400 MHz)



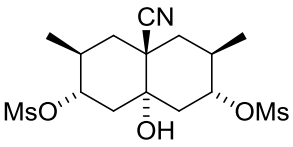


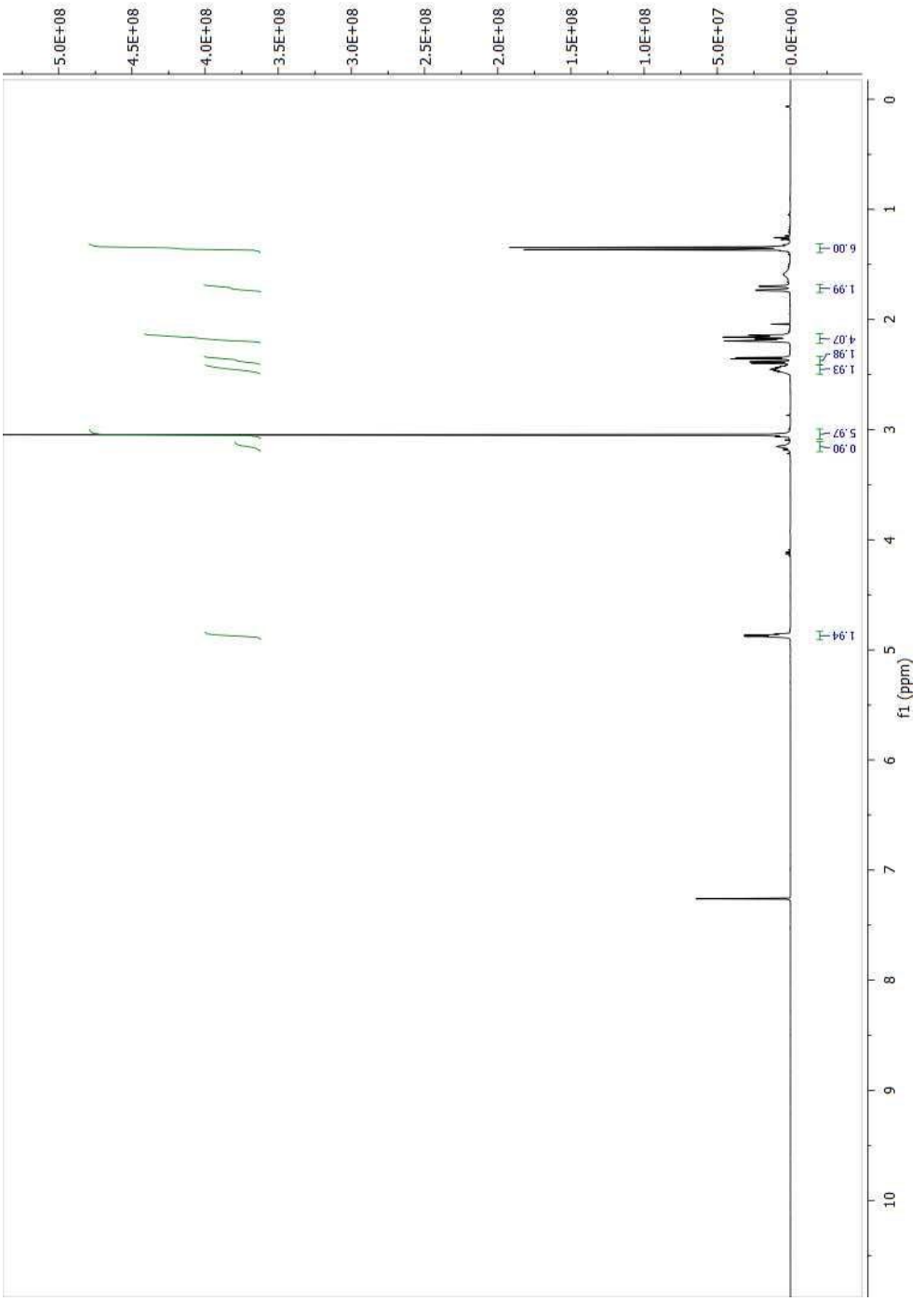
Triol 8 – ^{13}C NMR (100 MHz)



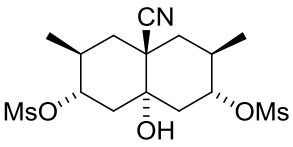


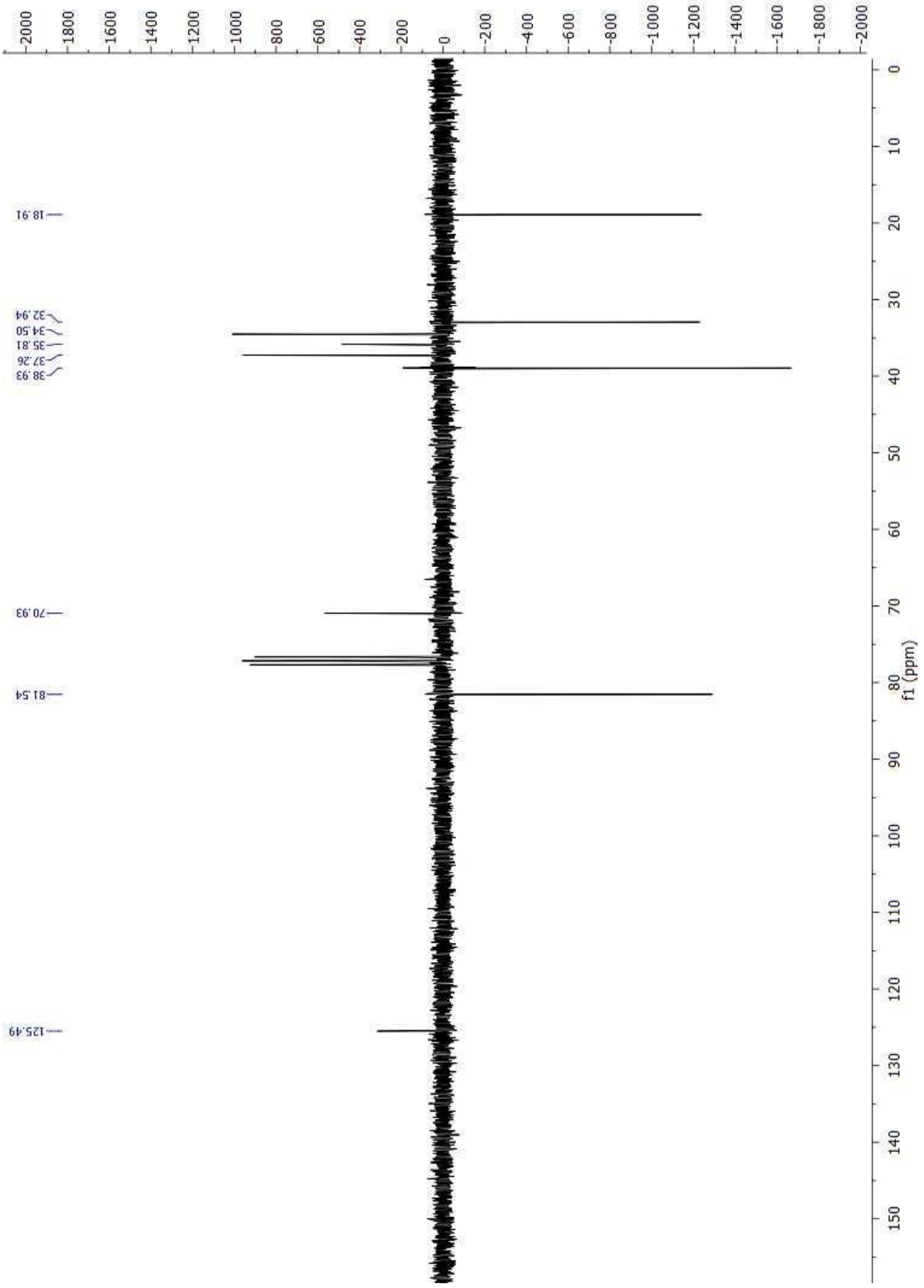
bis-Mesylate 9 – ^{1}H NMR (400 MHz)



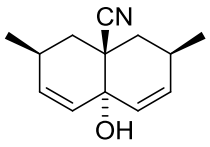


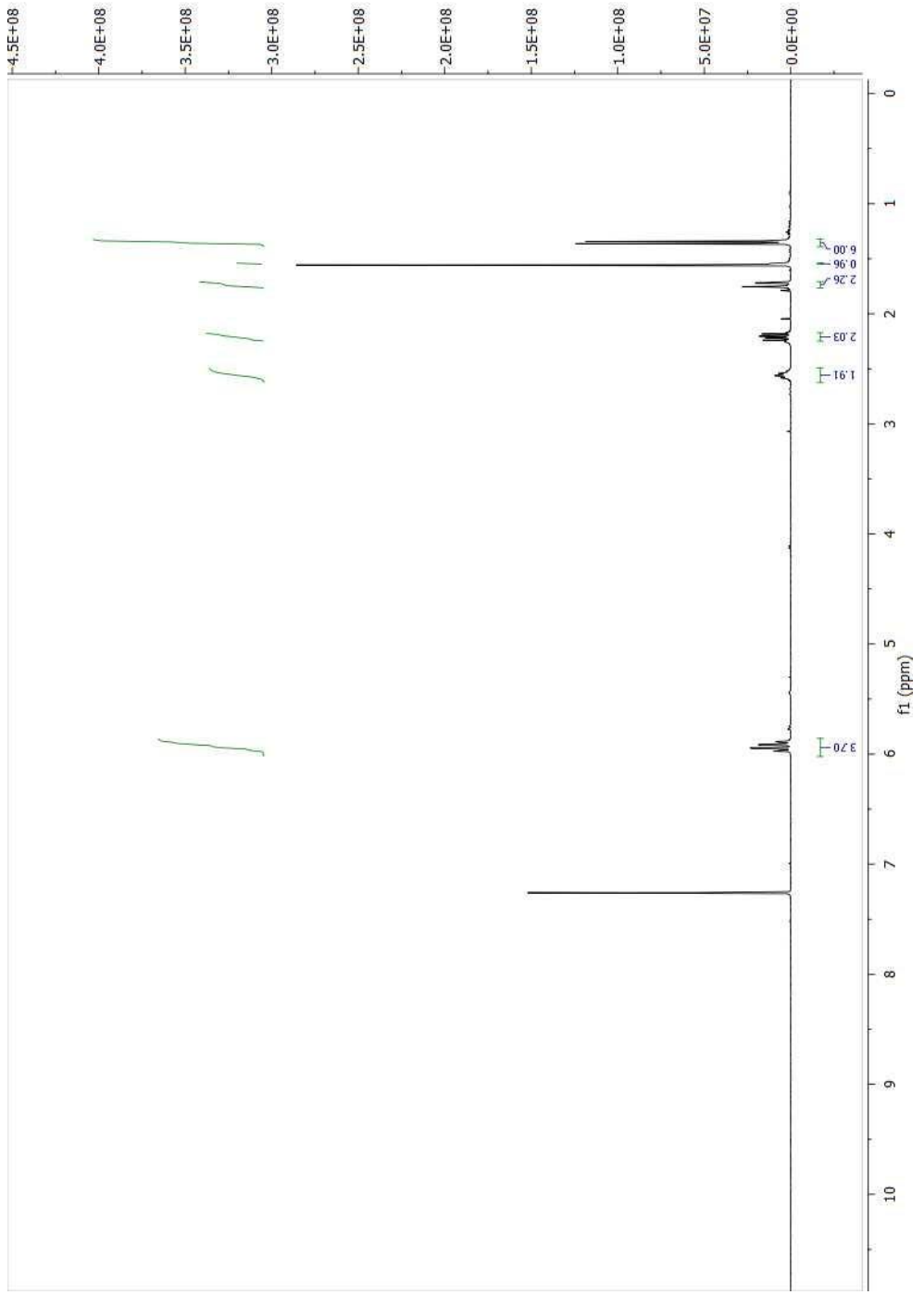
bis-Mesylate 9 – ^{13}C NMR (100 MHz)



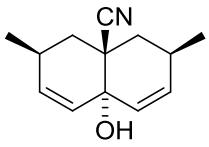


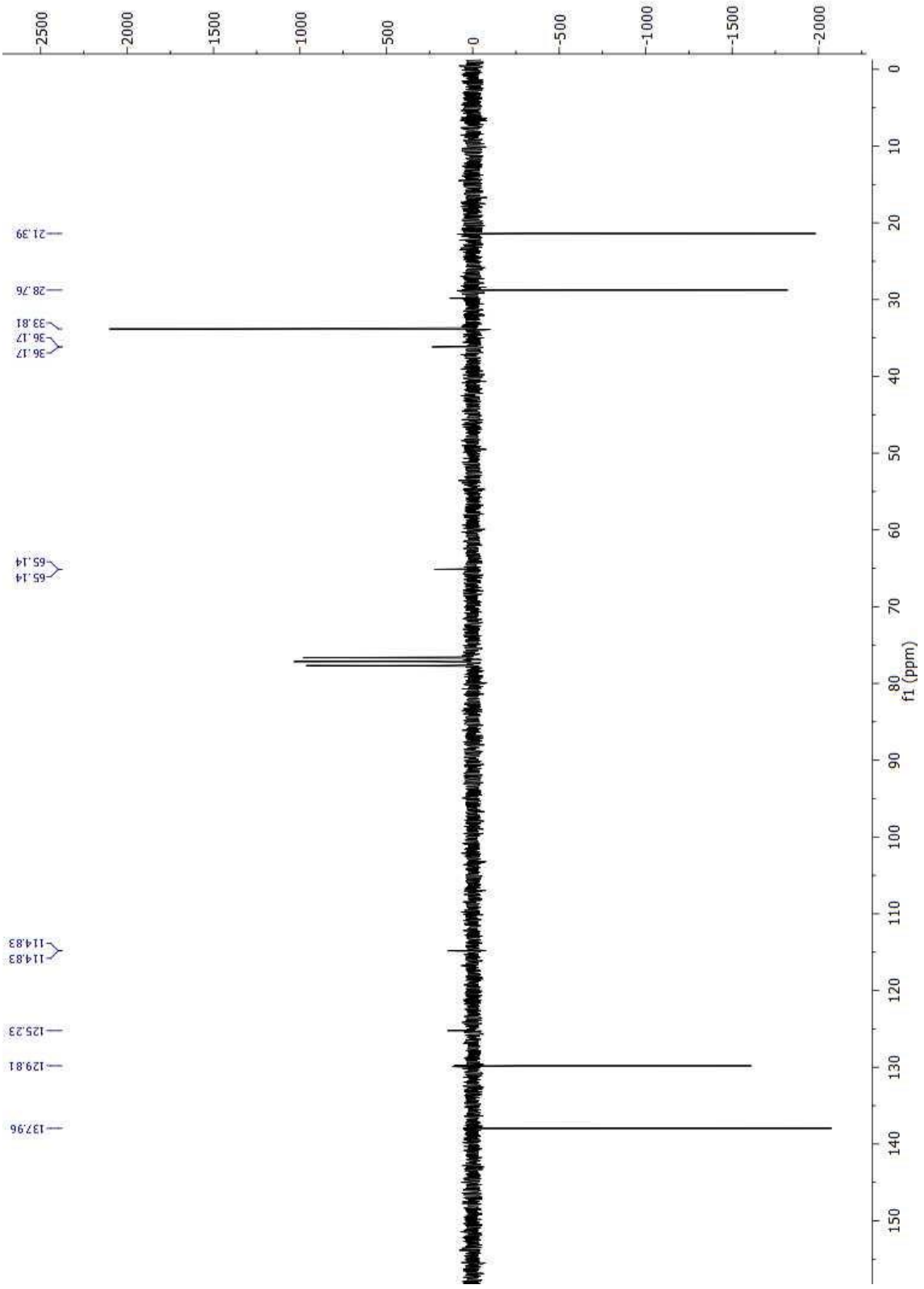
meso-Diallylic alcohol 10 – ^{1}H NMR (400 MHz)



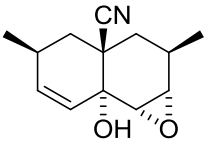


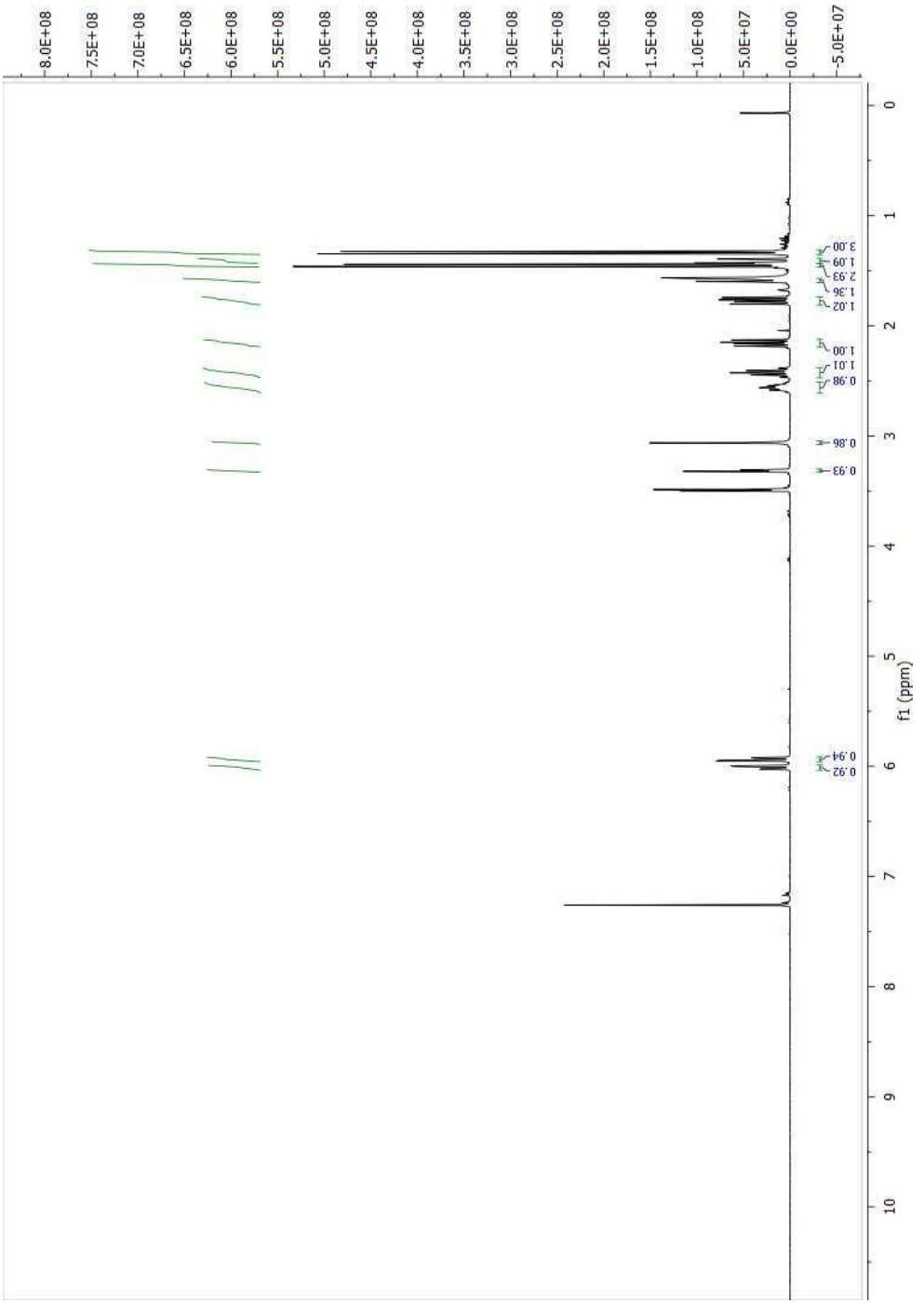
meso-Diallylic alcohol 10 – ^{13}C NMR (100 MHz)



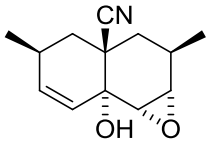


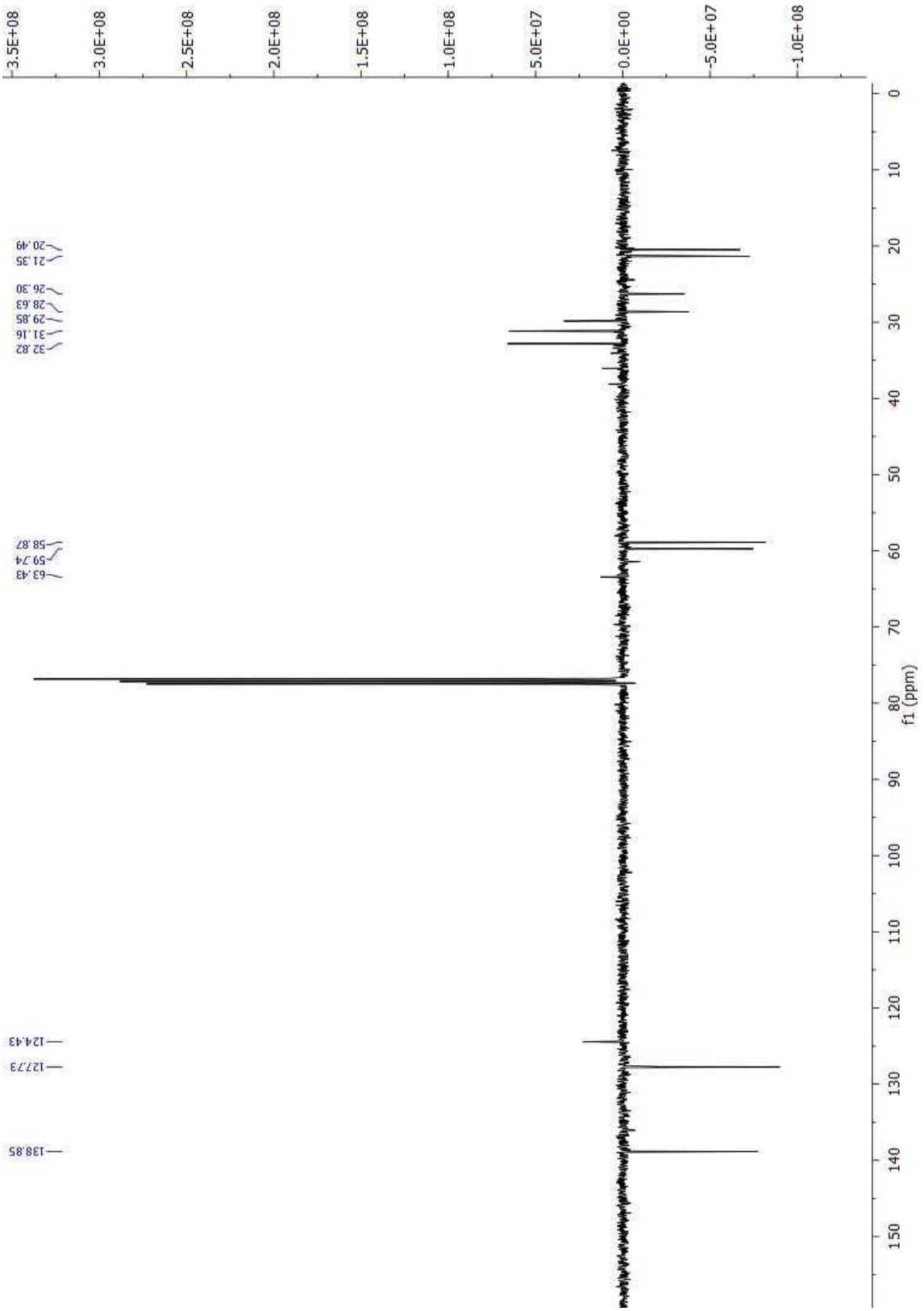
(±)-Monoepoxide 3 – ^{1}H NMR (400 MHz)



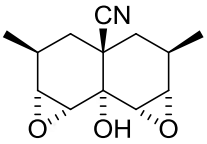


(±)-Monoepoxide 3 – ^{13}C NMR (100 MHz)

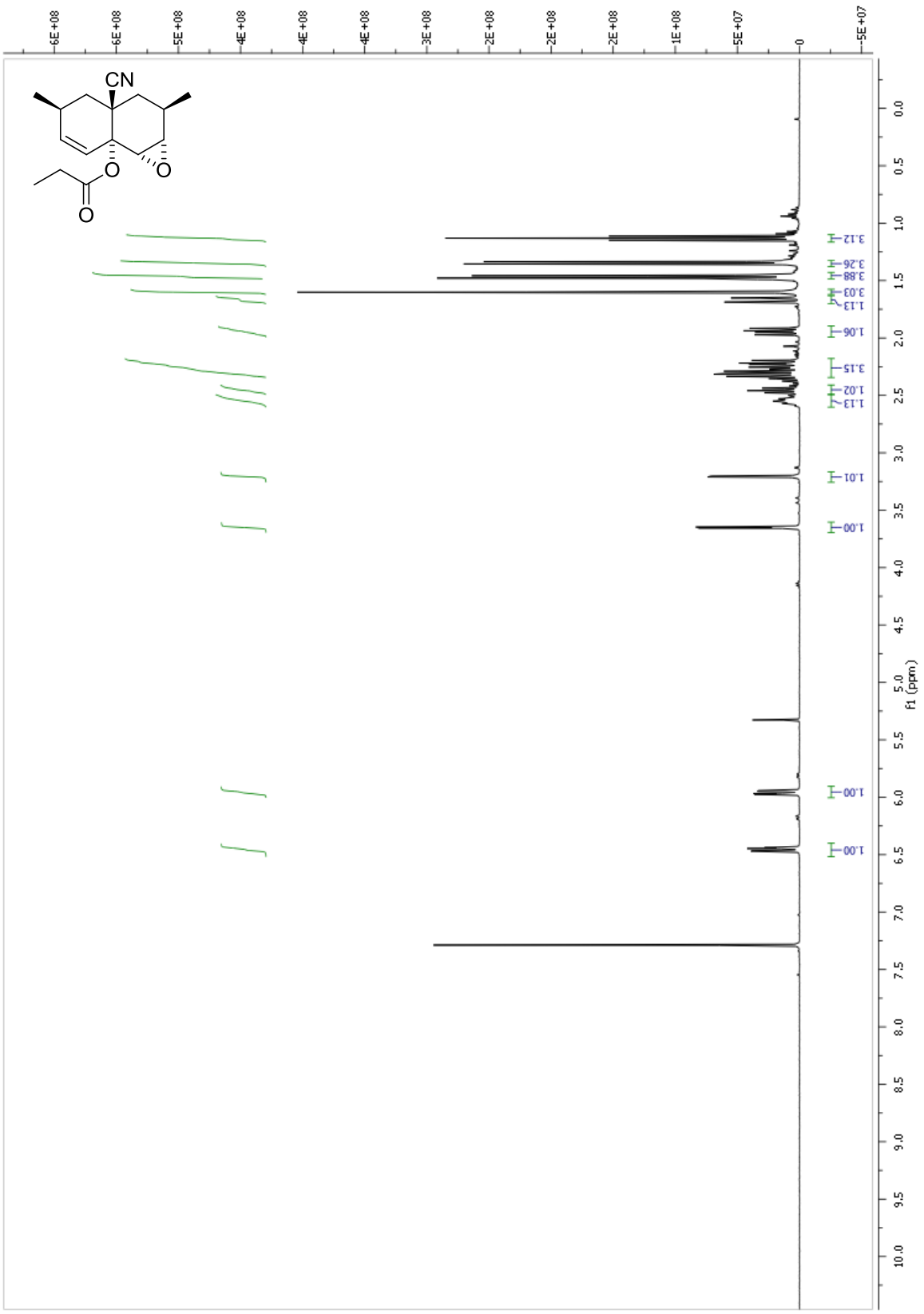




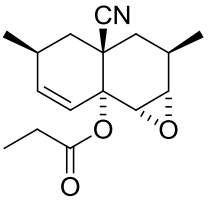
Meso-bis-epoxide 11 – ^{1}H NMR (250 MHz)

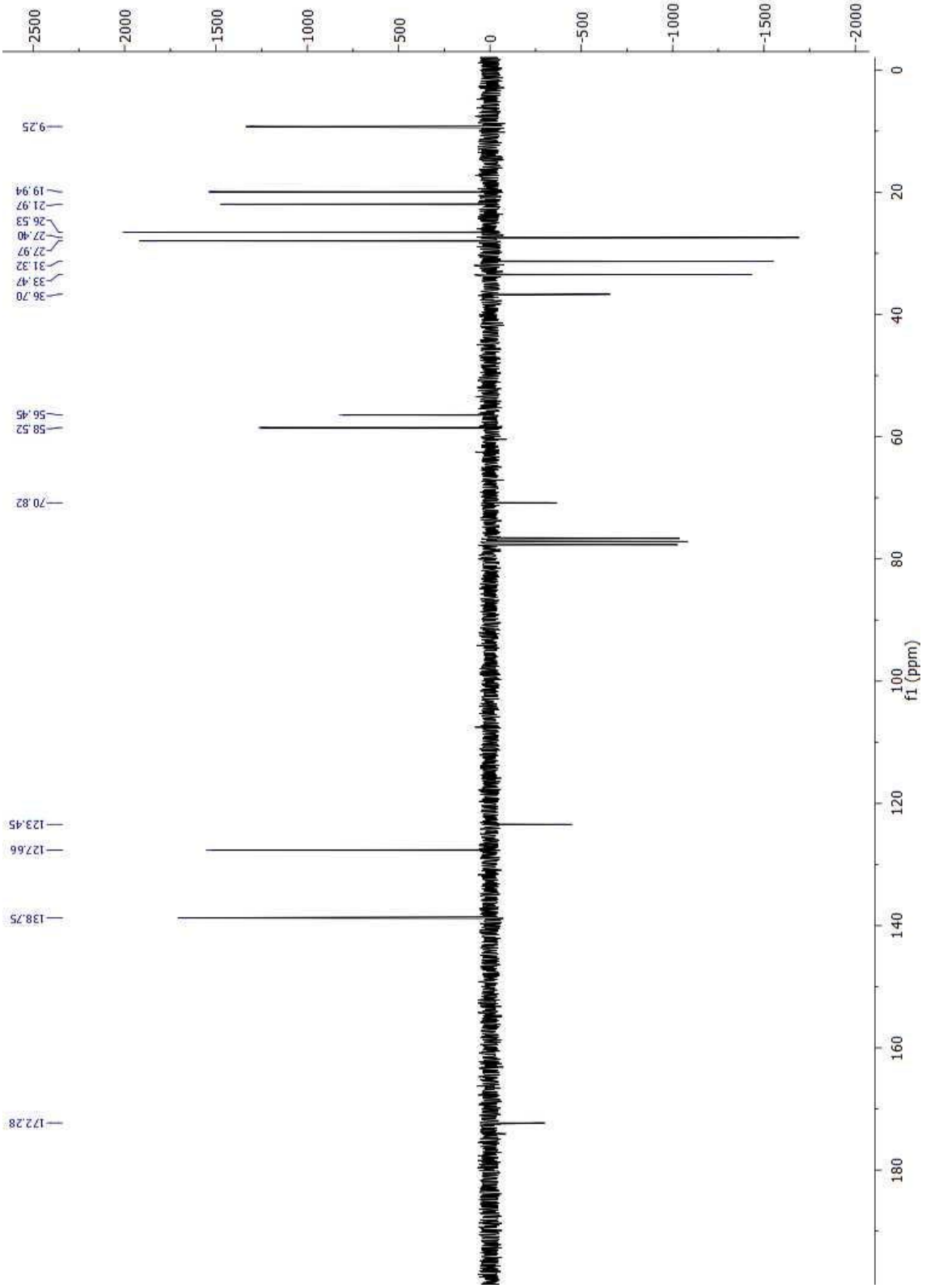


Propionate 12 – ^{1}H NMR (270 MHz)

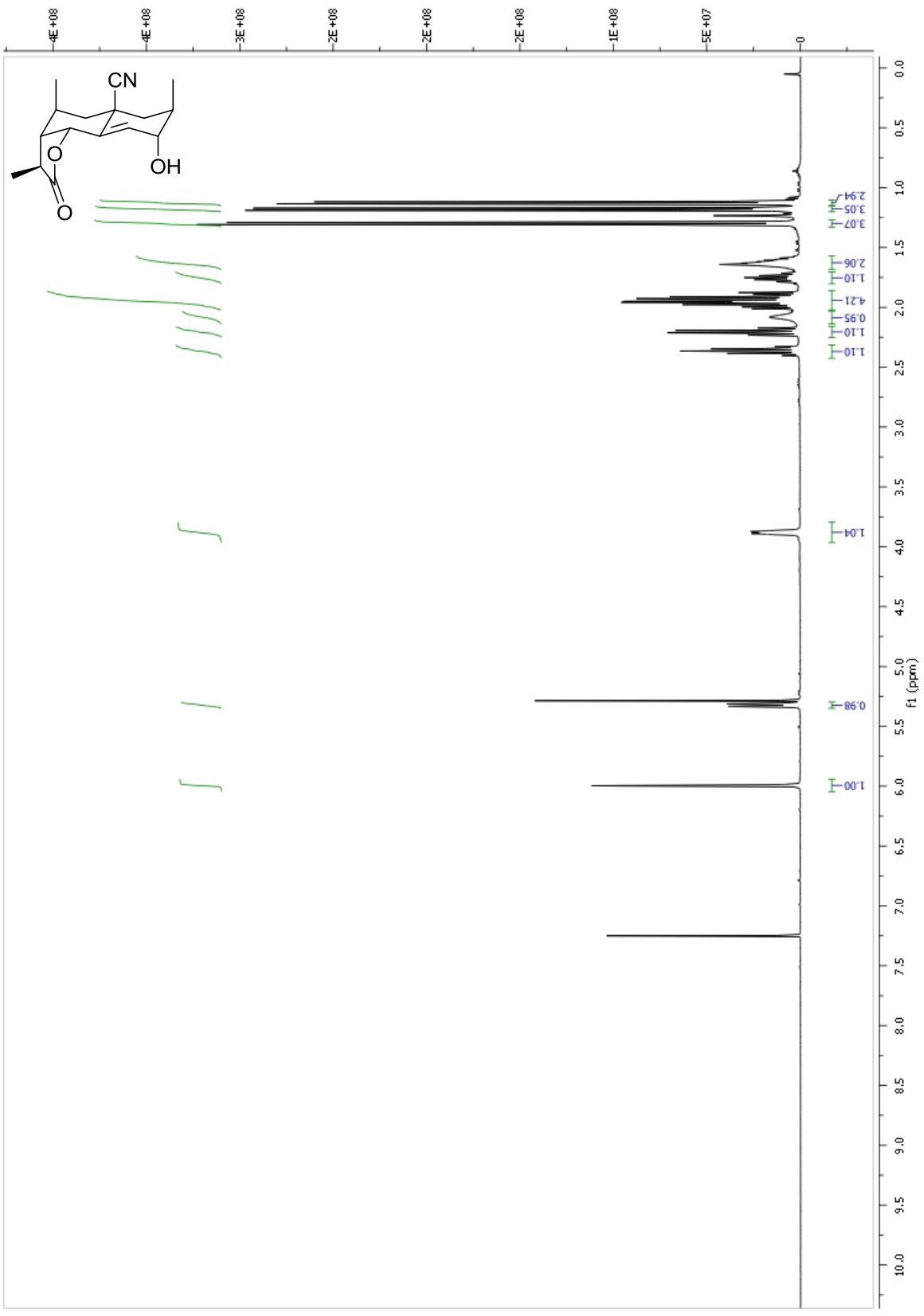


Propionate 12 – ^{13}C DEPT NMR (63 MHz)

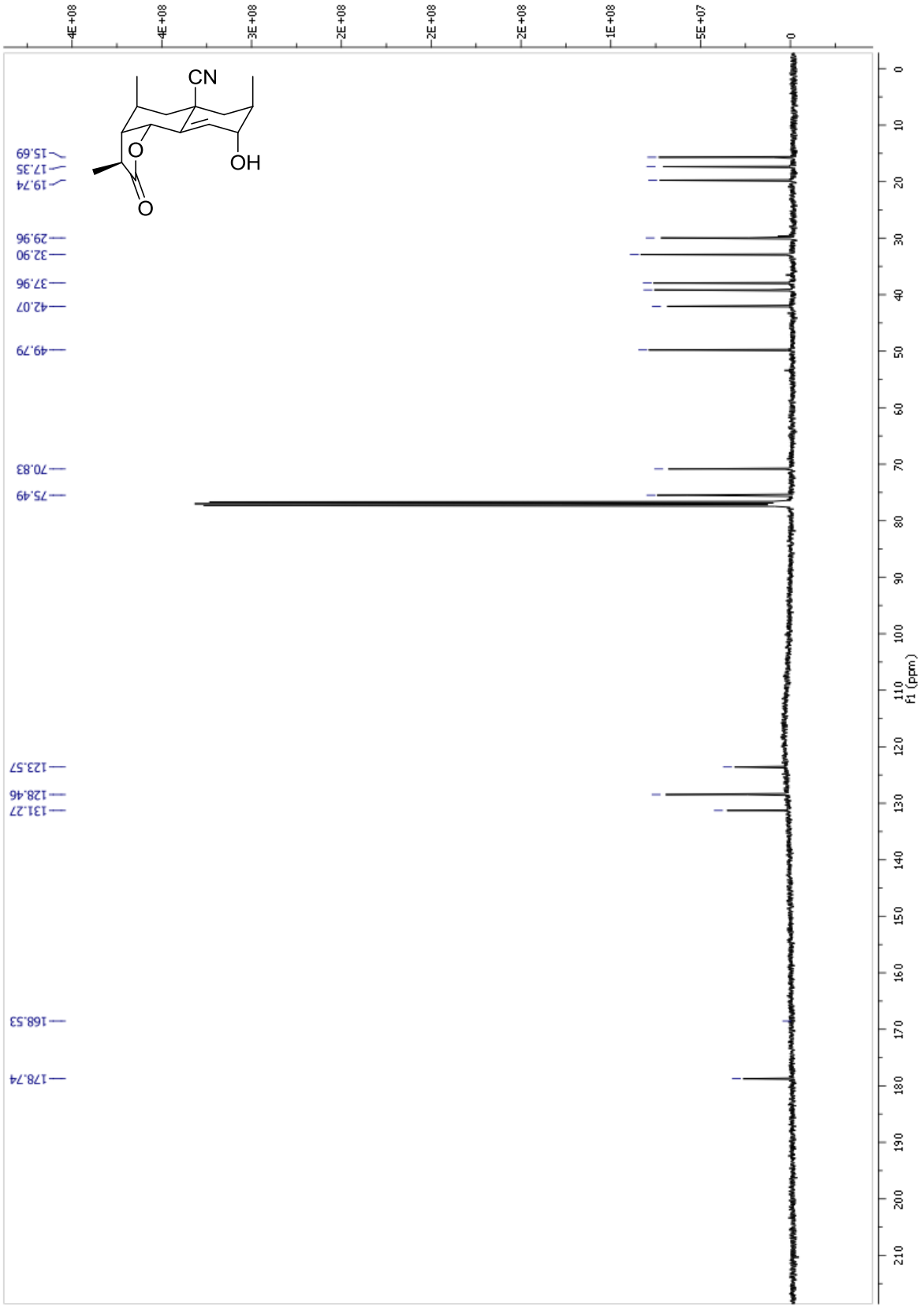




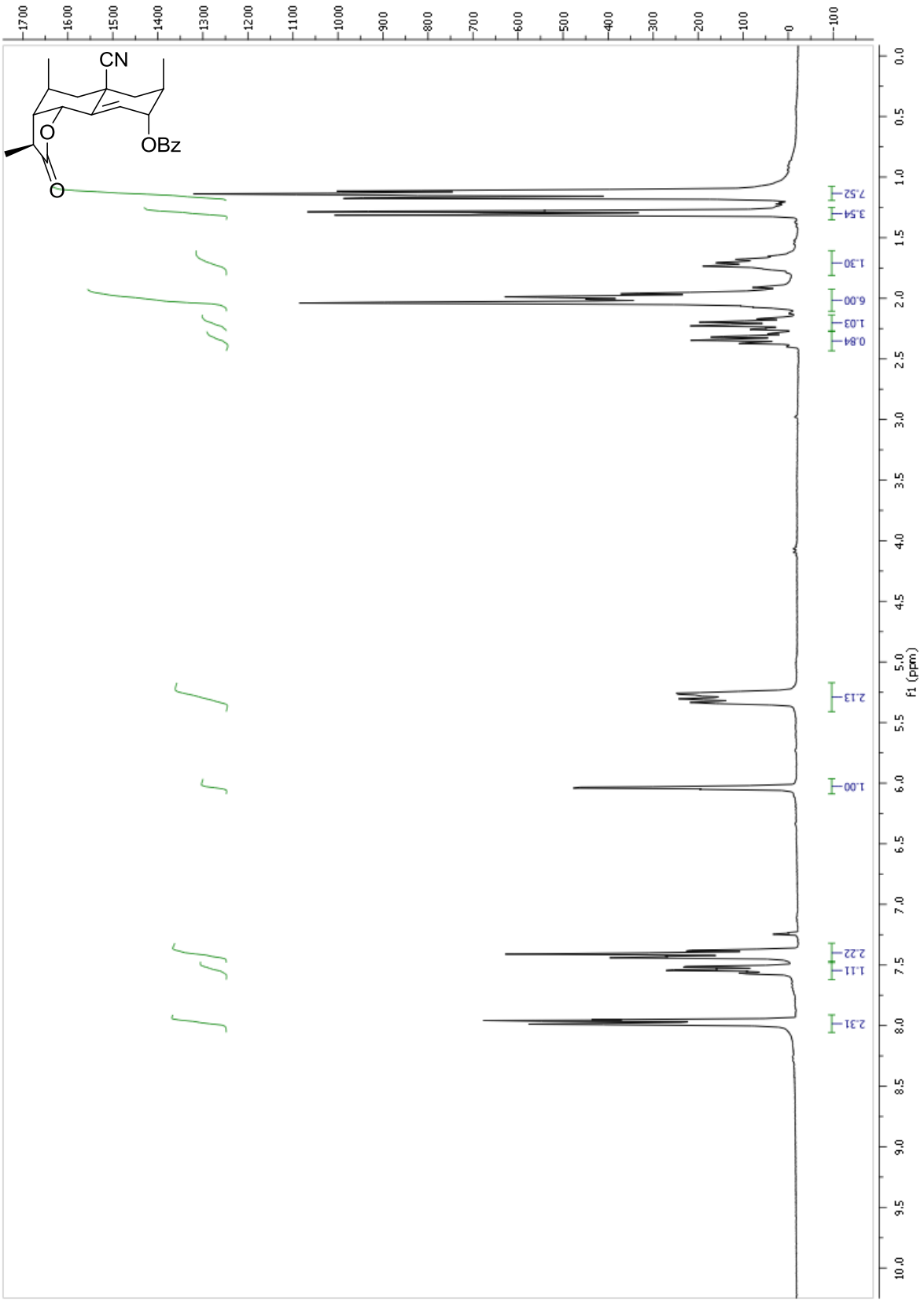
Lactone 13a – ^{1}H NMR (300 MHz)



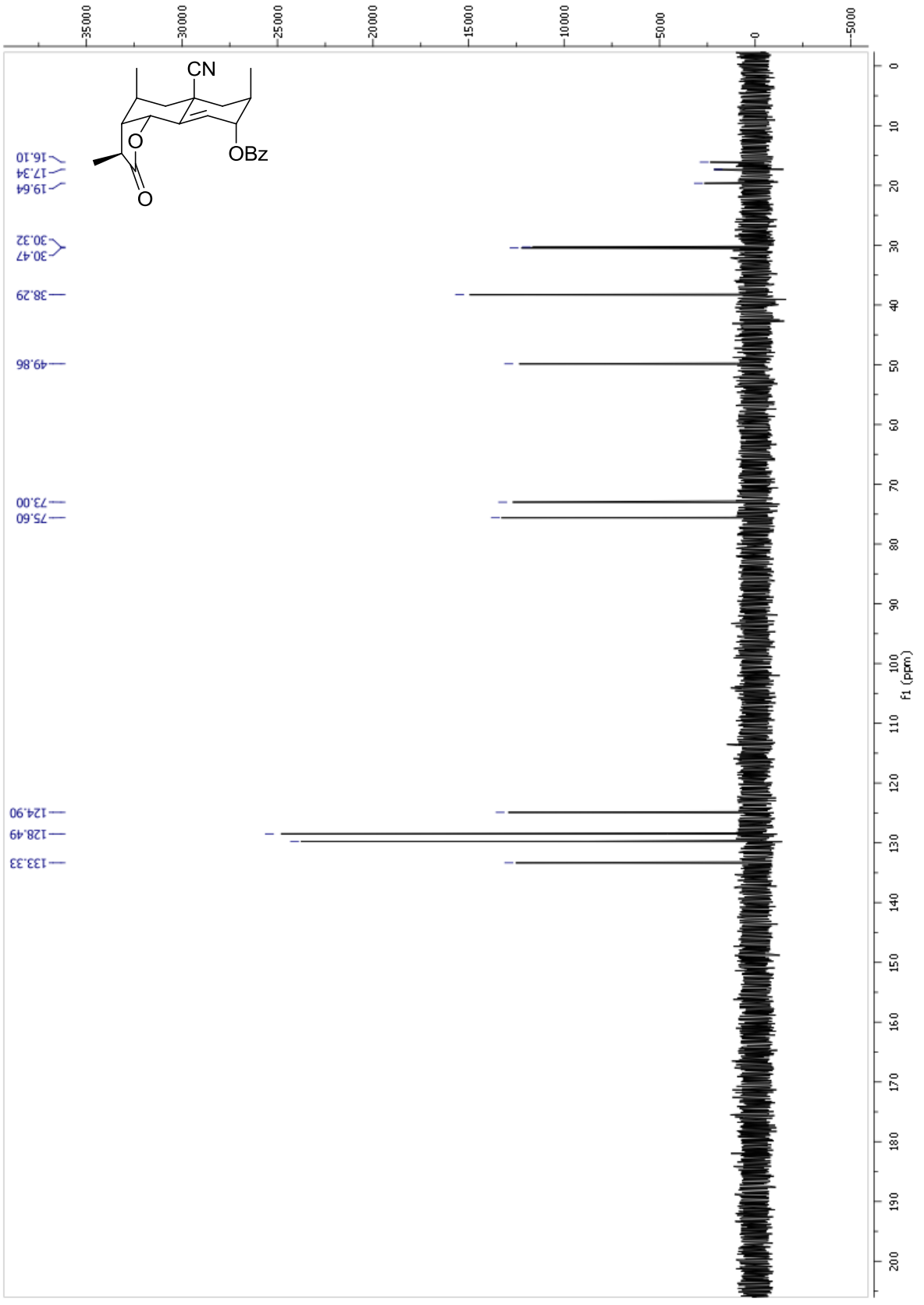
Lactone 13a - ^{13}C NMR (63 MHz)



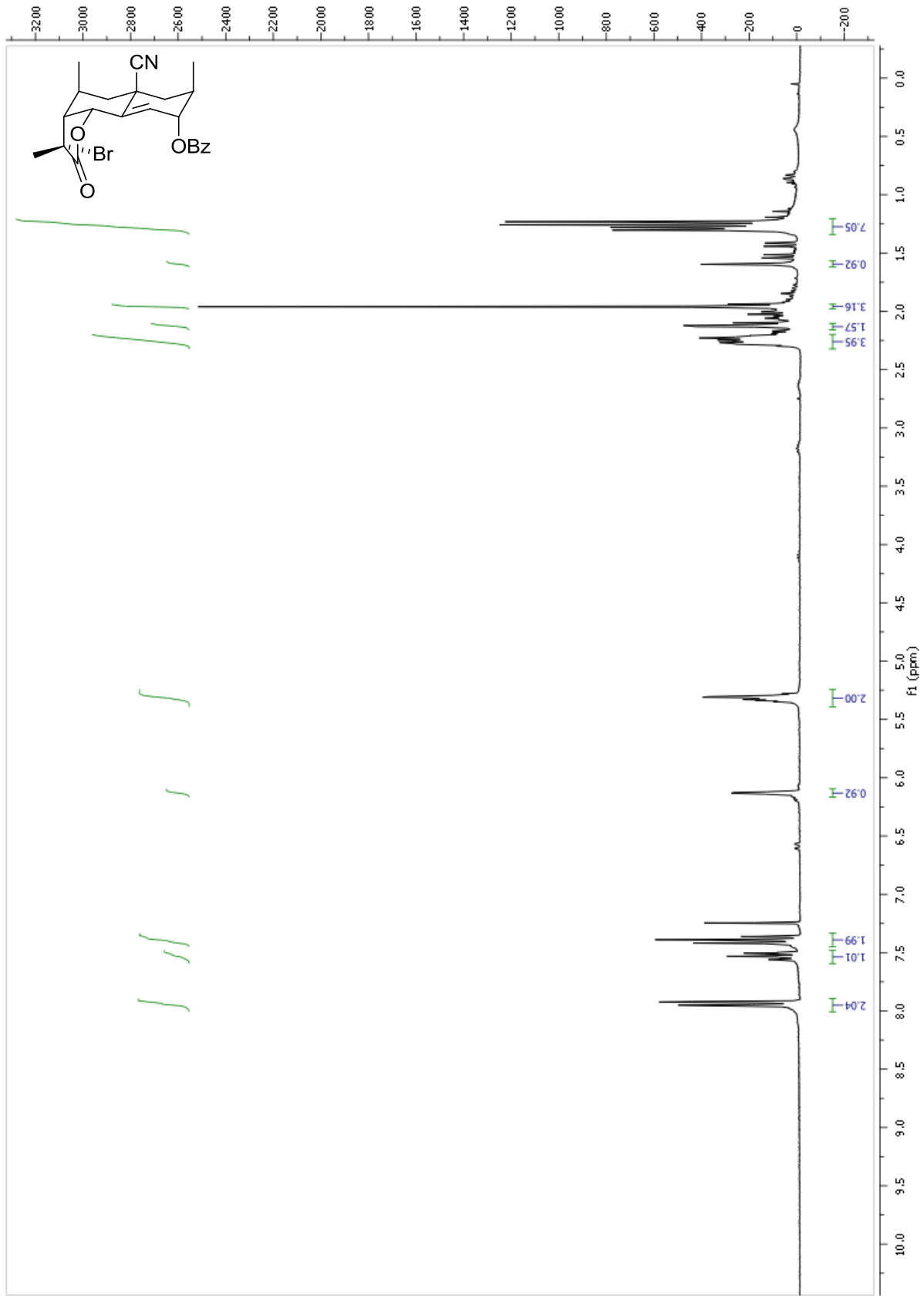
Benzoate ester 14 – ^{1}H NMR (270 MHz)



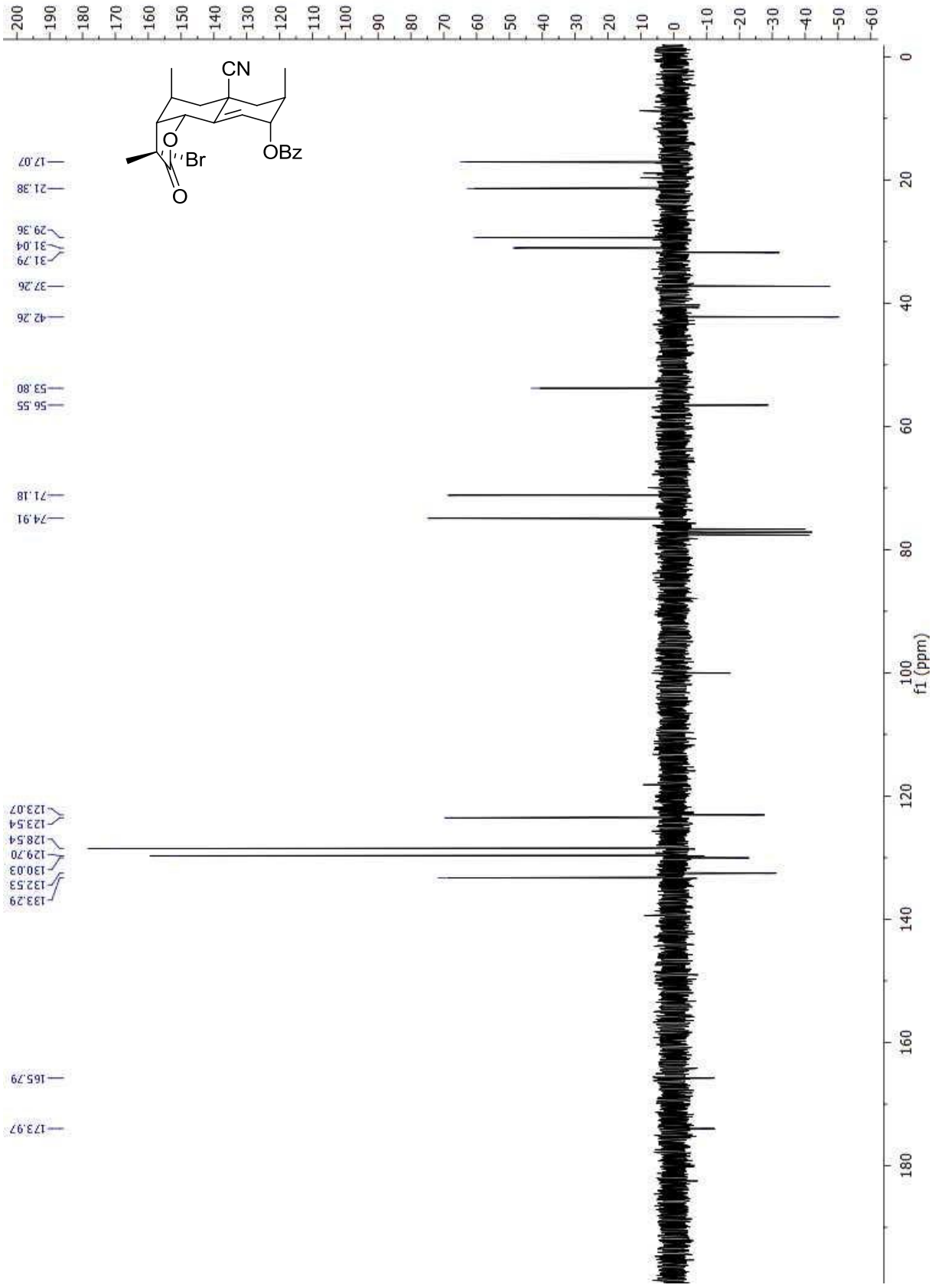
Benzoate ester 14 - ^{13}C NMR (68 MHz)



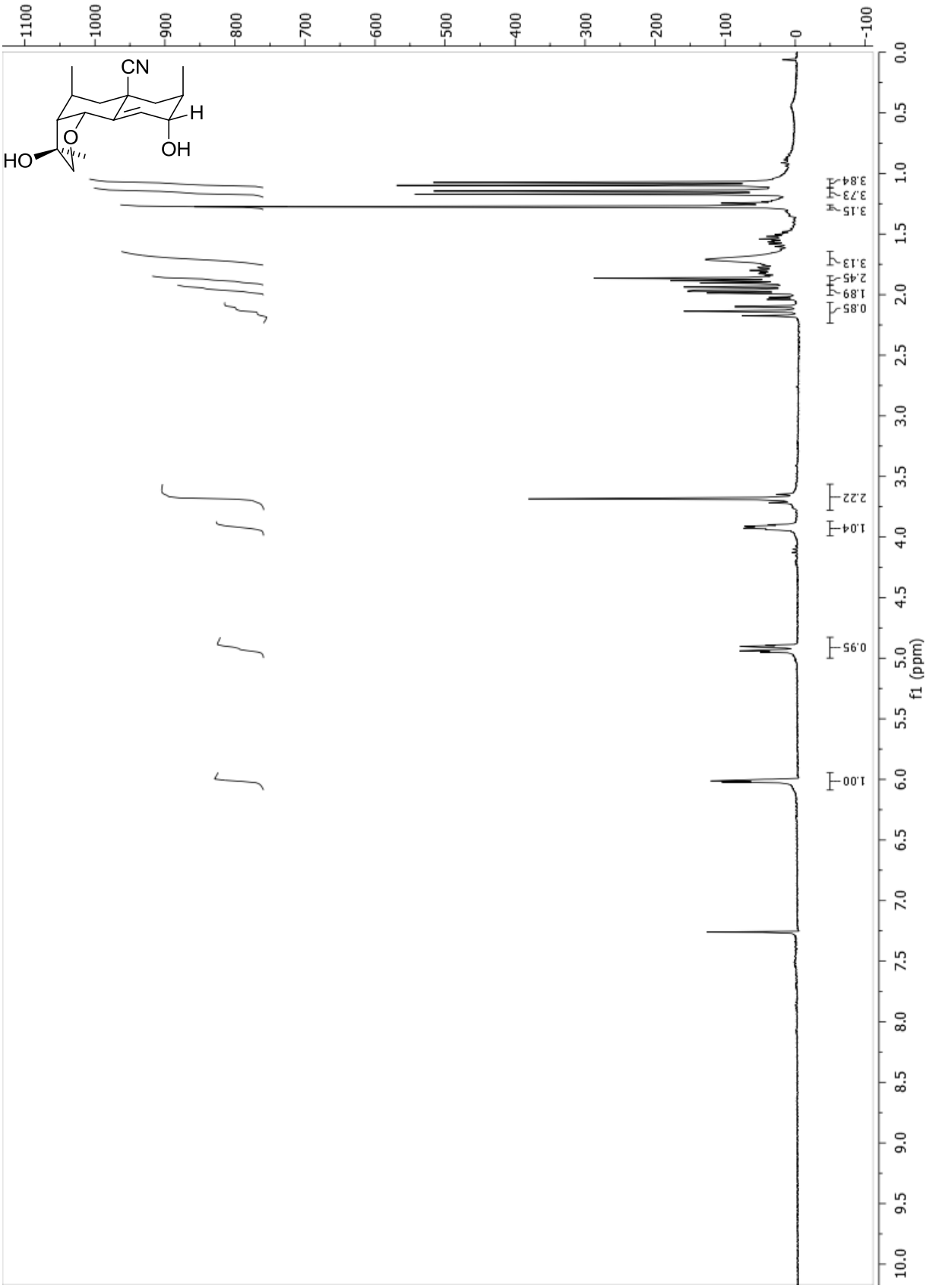
Bromide 15 - ^{1}H NMR (270 MHz)



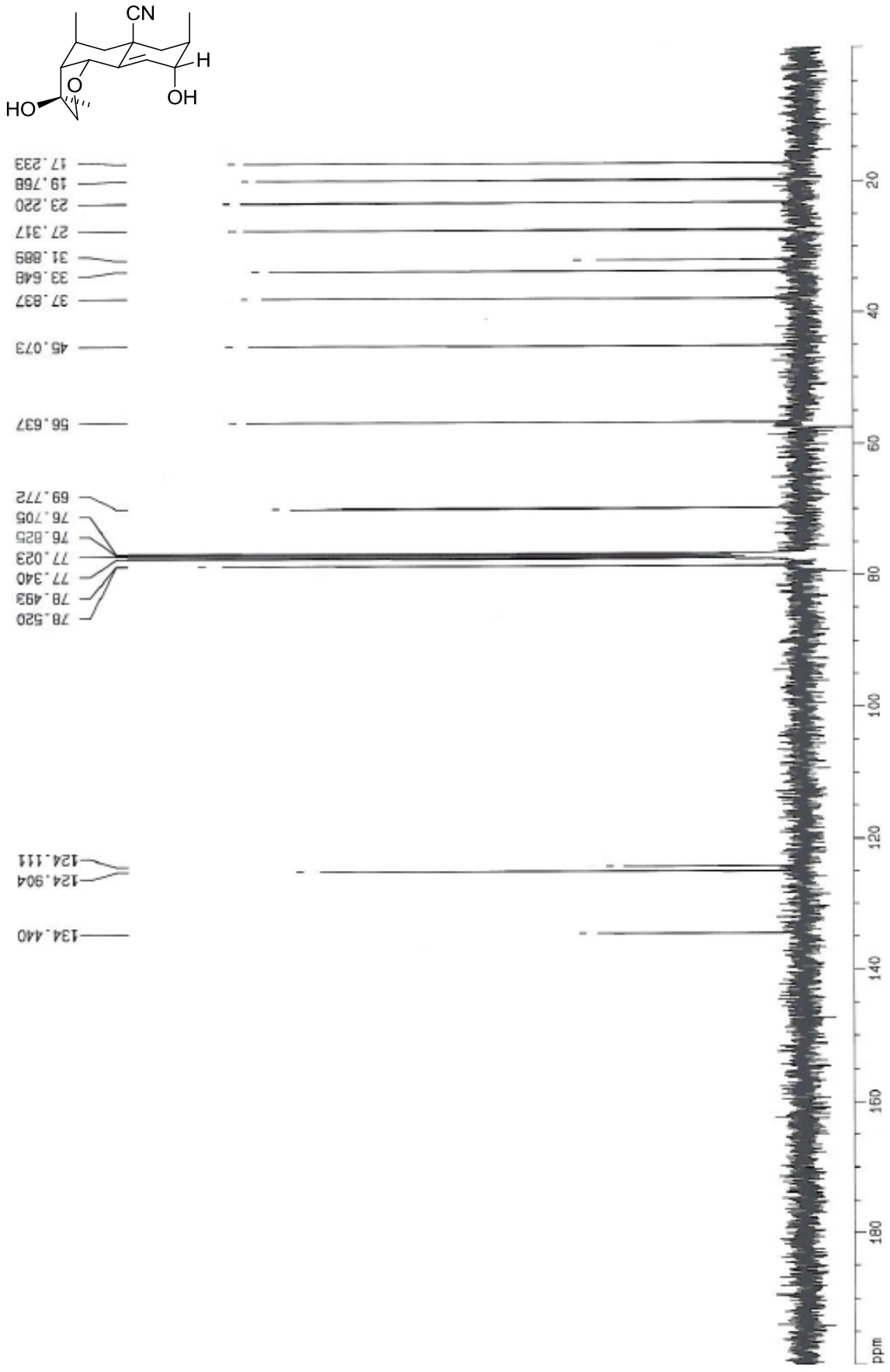
Bromide 15 - ^{13}C DEPT NMR (68 MHz)



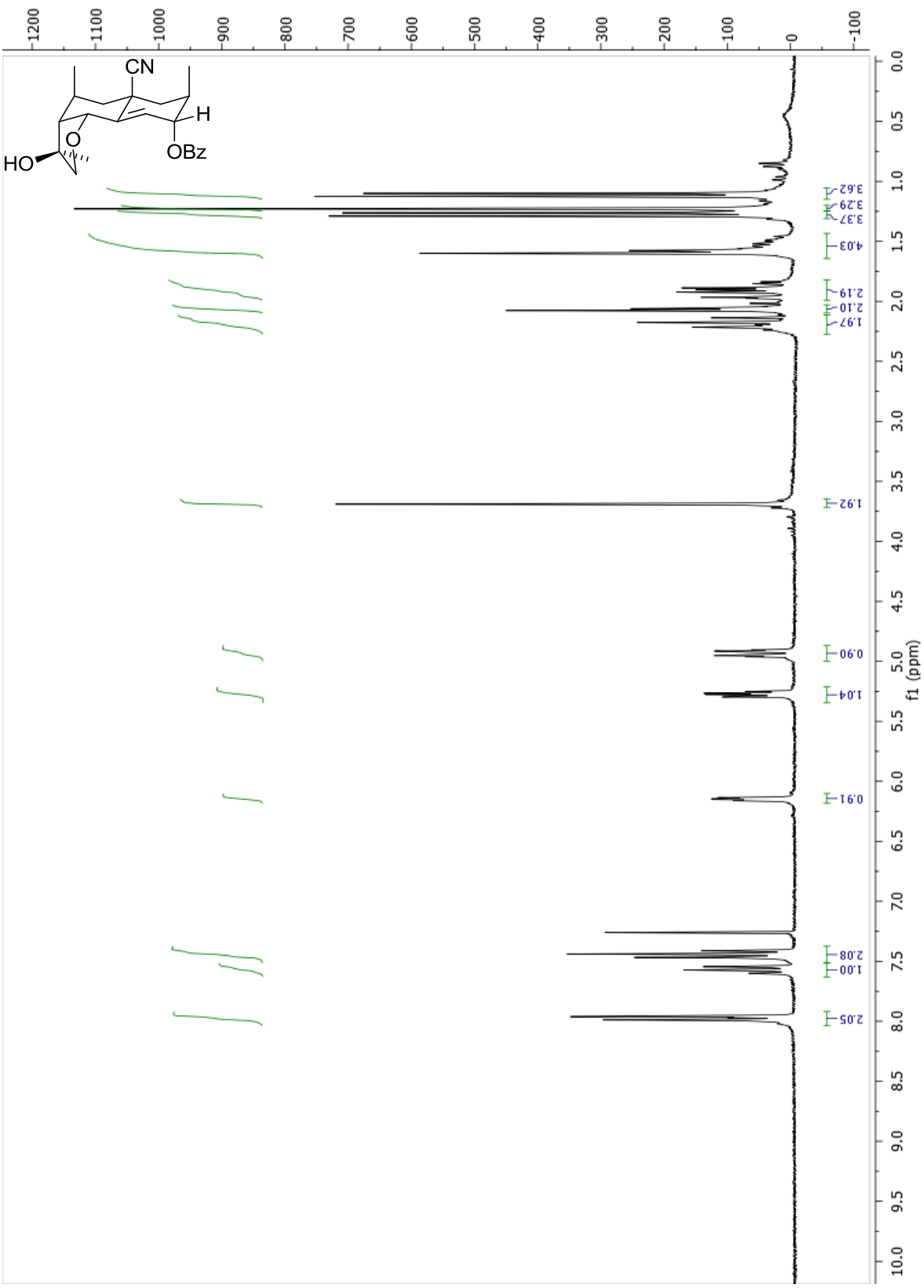
Hydroxytetrahydrofuran 16 - ^{1}H NMR (400 MHz)



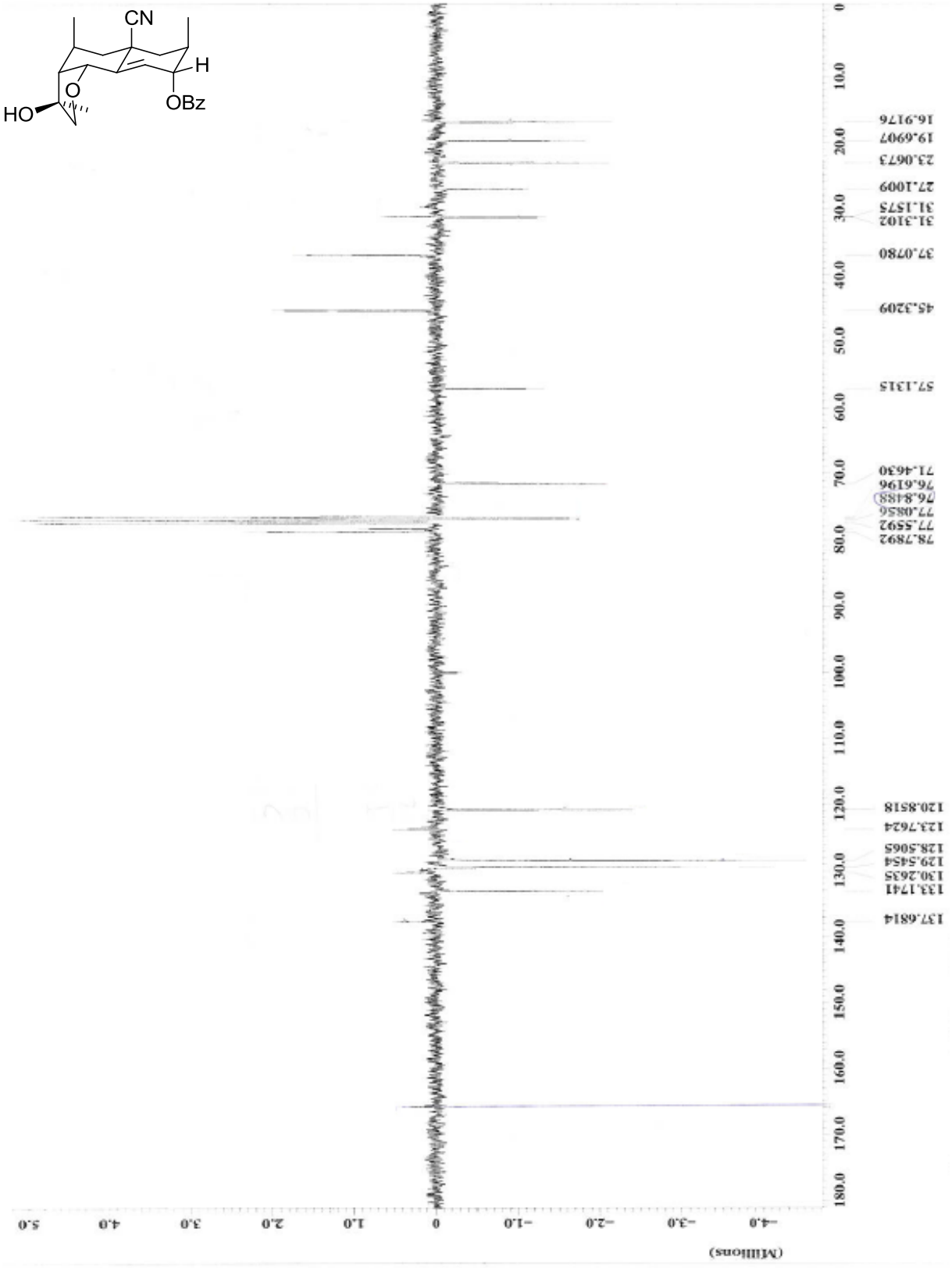
Hydroxytetrahydrofuran 16 - ^{13}C NMR (100 MHz)



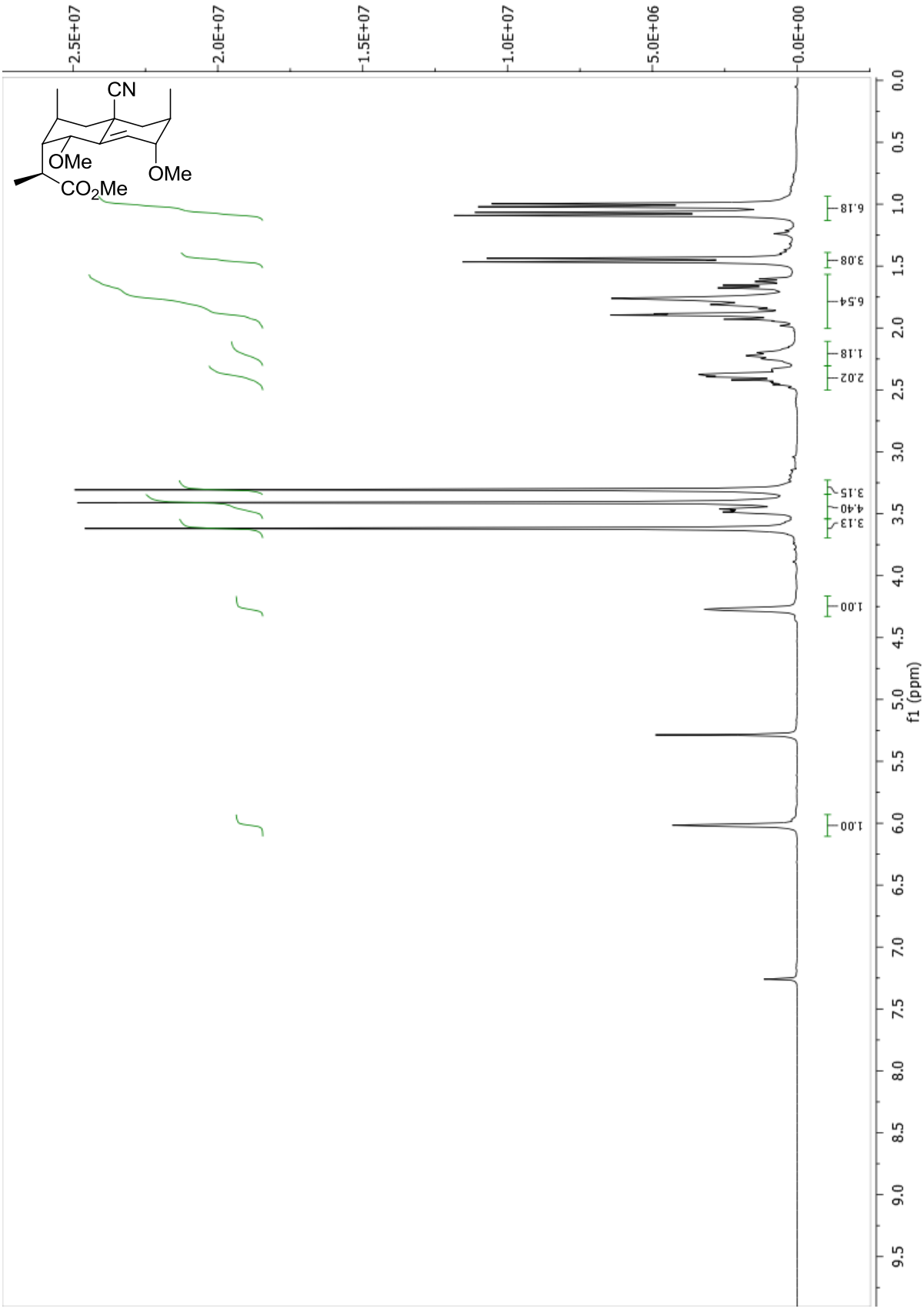
Benzoyloxytetrahydrofuran 17 - ^{1}H NMR (270 MHz)



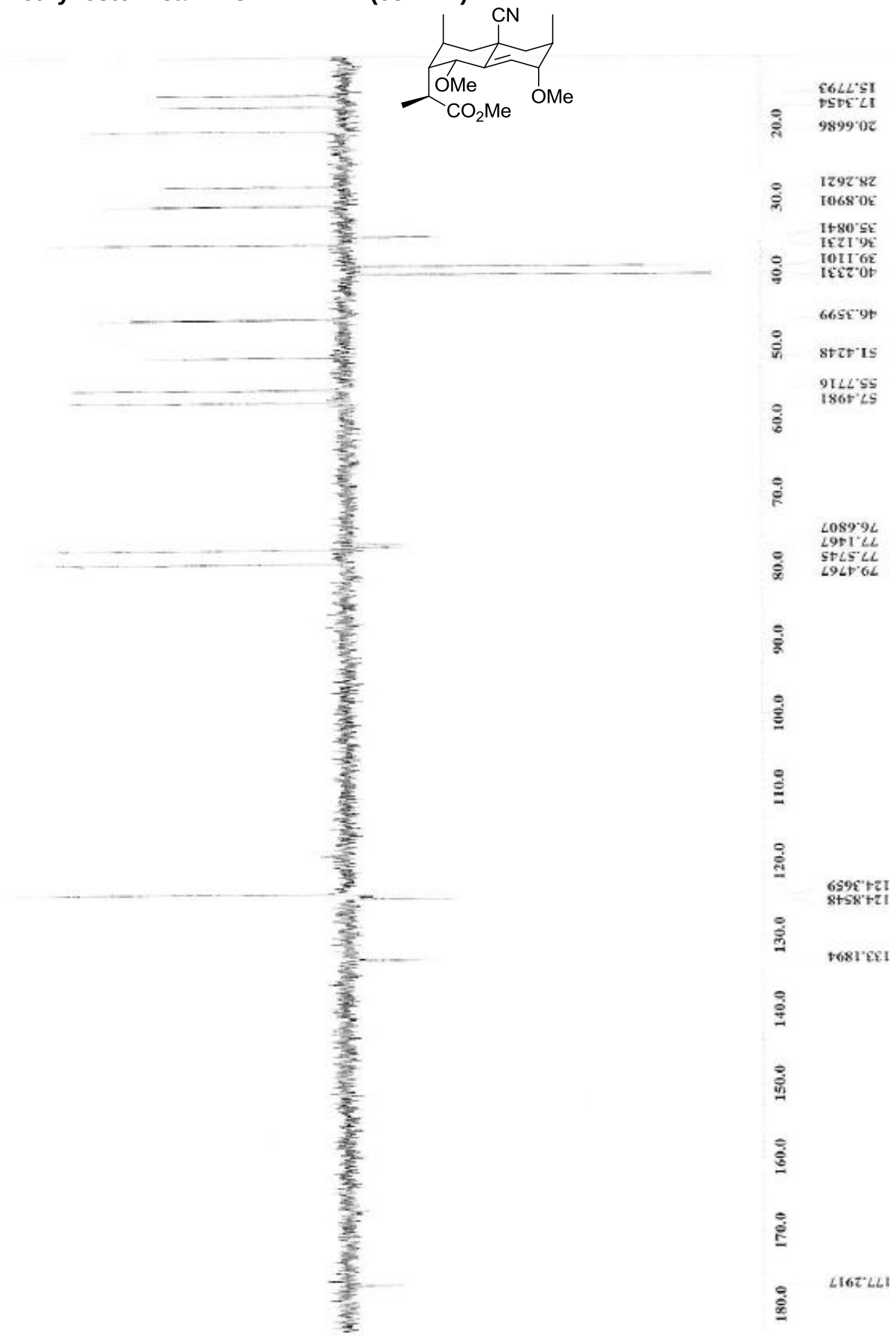
Benzoyloxytetrahydrofuran 17 – ^{13}C DEPT NMR (68 MHz)



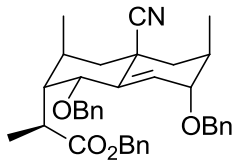
Methyl ester 18a – ^{1}H NMR (270 MHz)

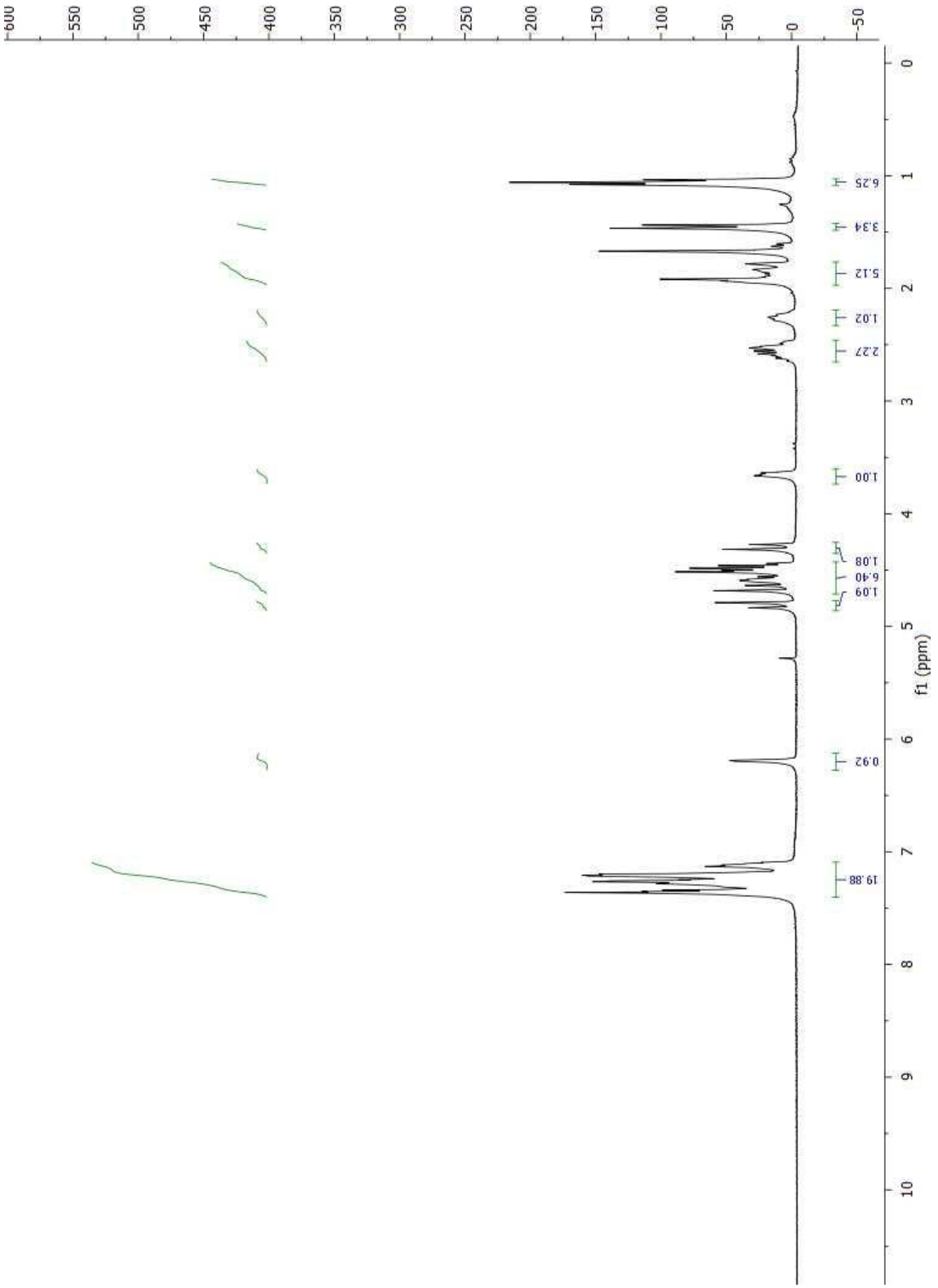


Methyl ester 18a – ^{13}C APT NMR (68 MHz)

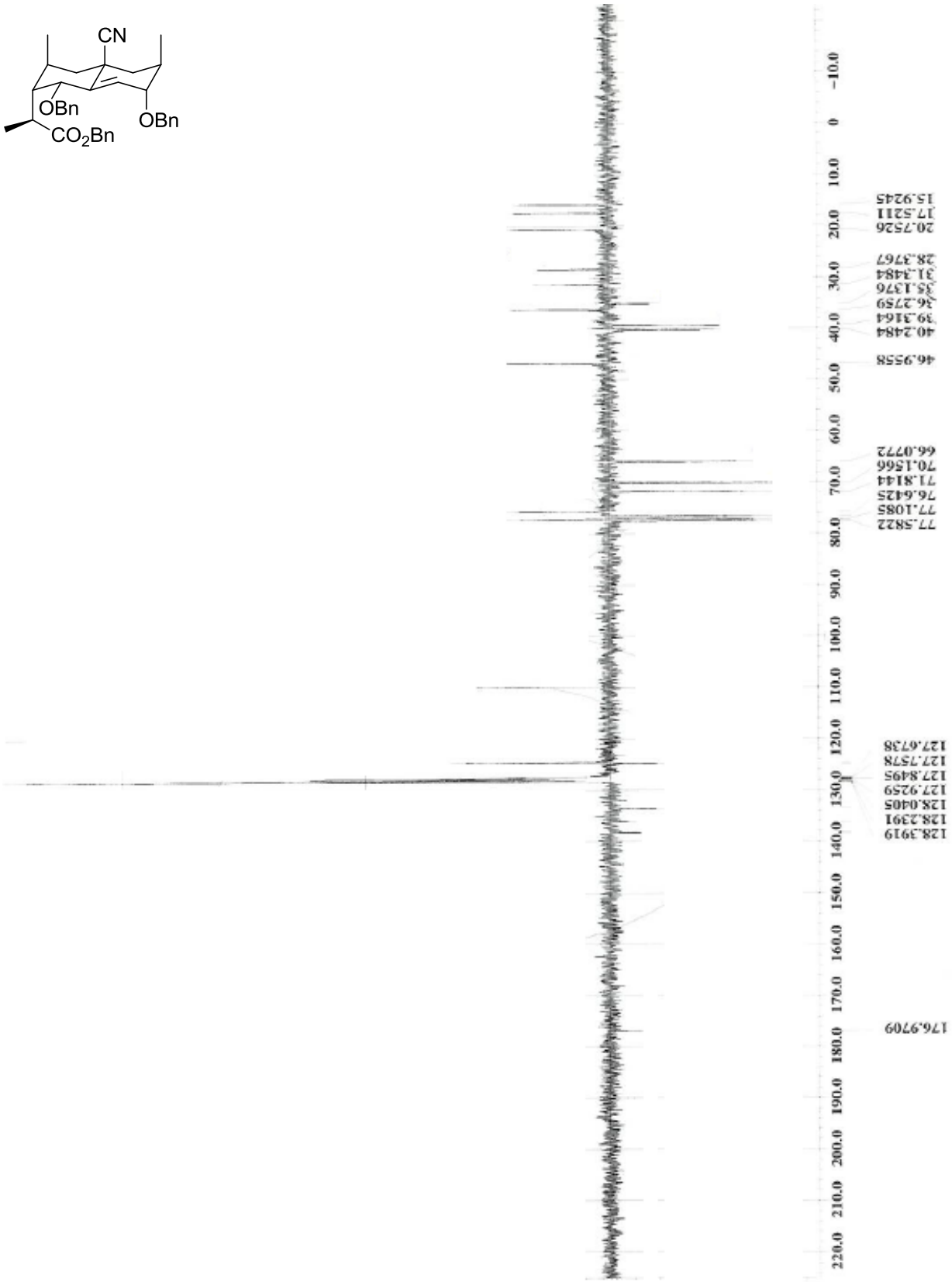


Benzyl ester 18b – ^{1}H NMR (270 MHz)

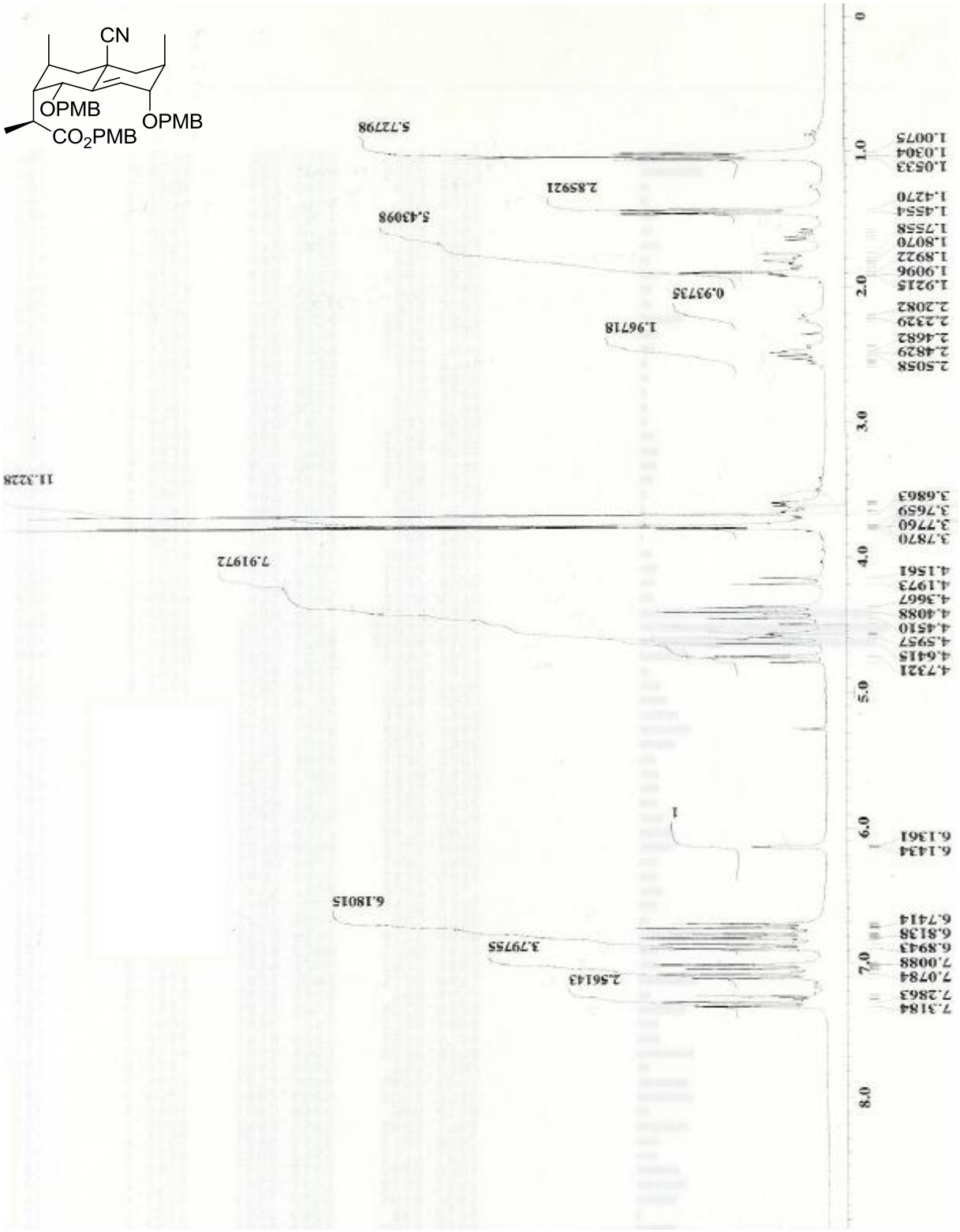




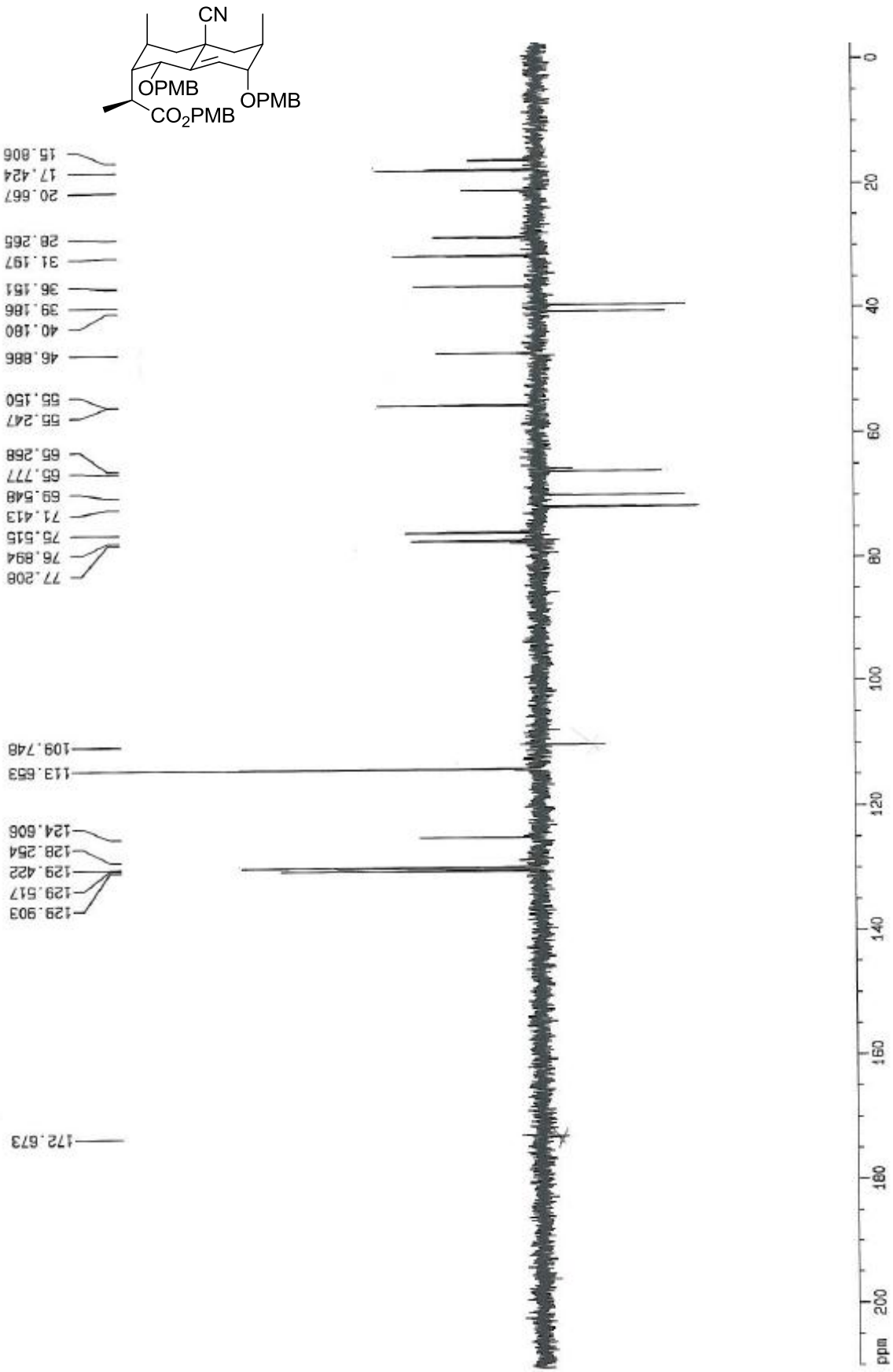
Benzyl ester 18b – ^{13}C APT NMR (75 MHz)



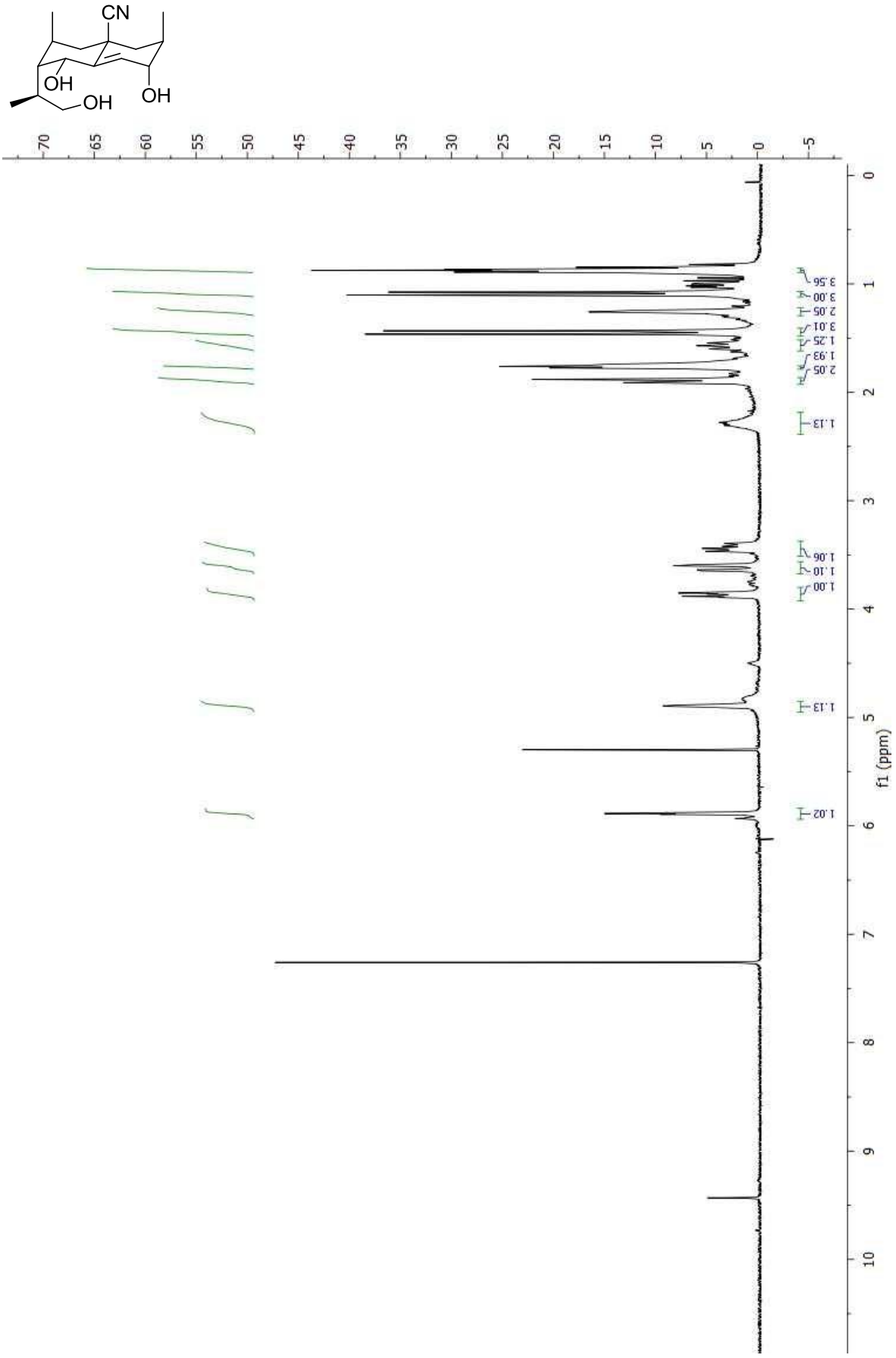
PMB ester 18c – ^{1}H NMR (270 MHz)



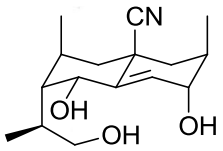
PMB ester 18c – ^{13}C APT NMR (100 MHz)

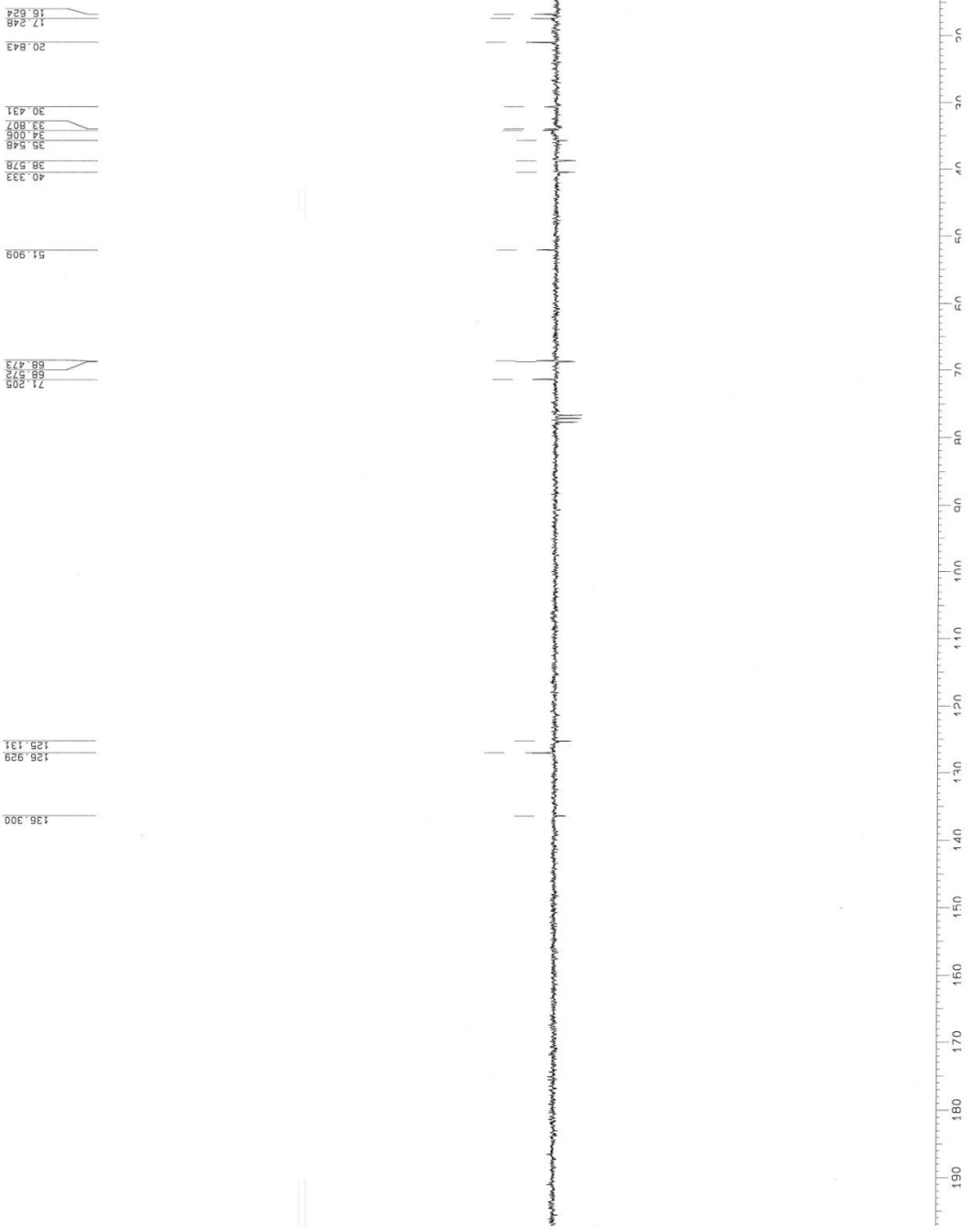


Triol 19 – ^{1}H NMR (250 MHz)

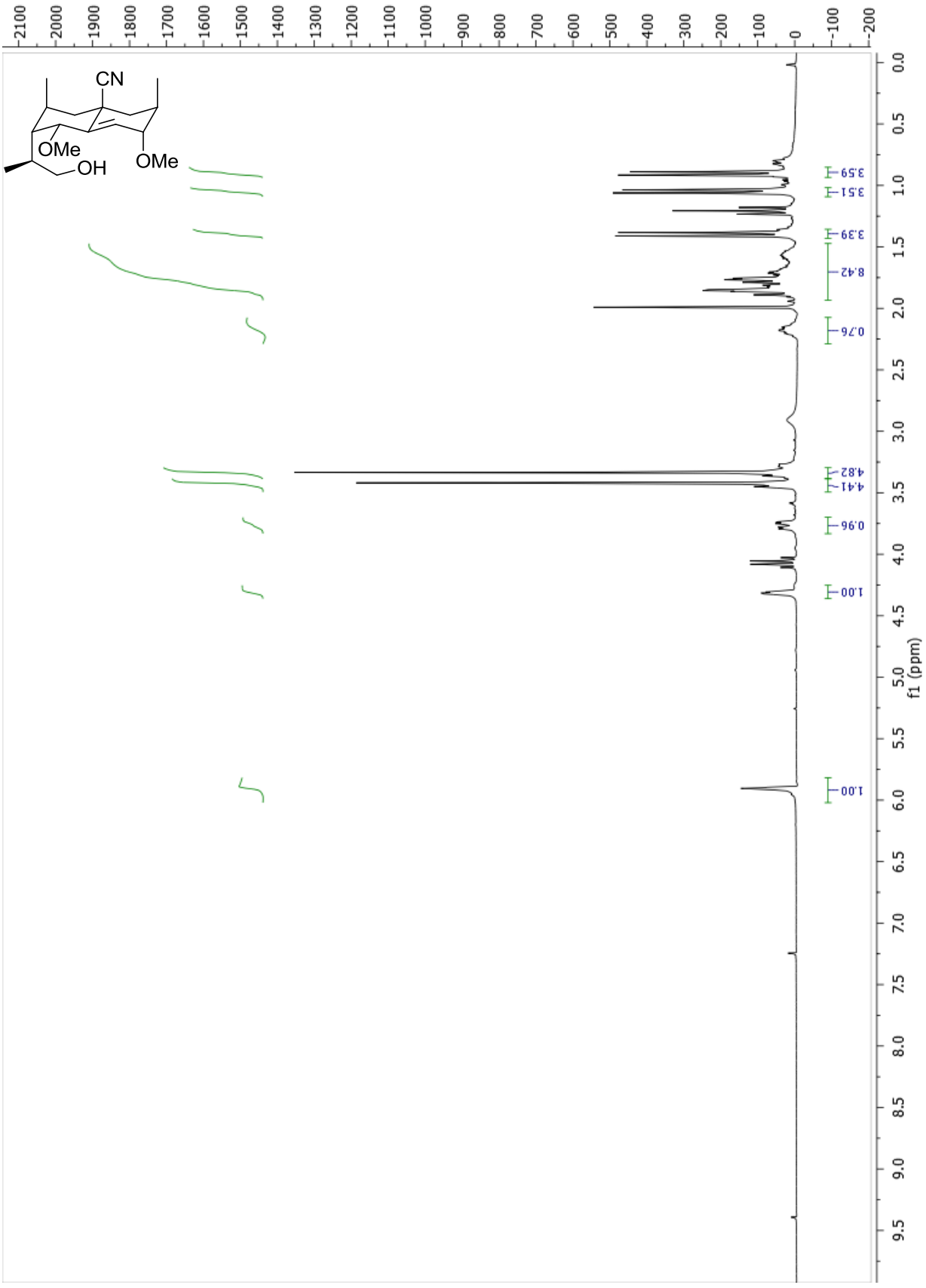


Triol 19 – ^{13}C APT NMR (63 MHz)

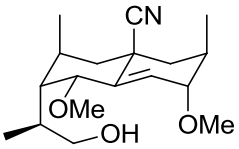




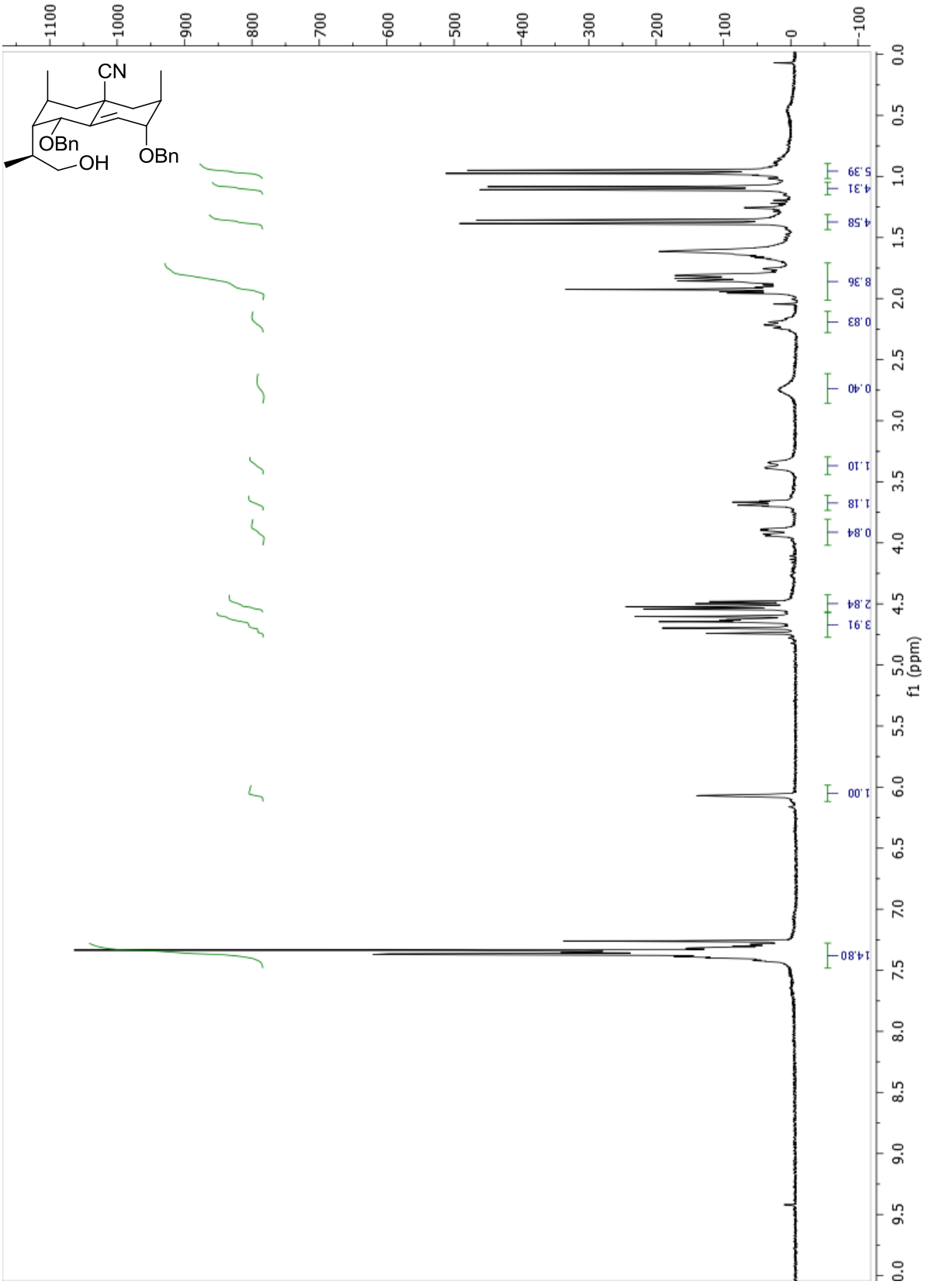
Dimethyl ether alcohol 19a – ^{1}H NMR (270 MHz)



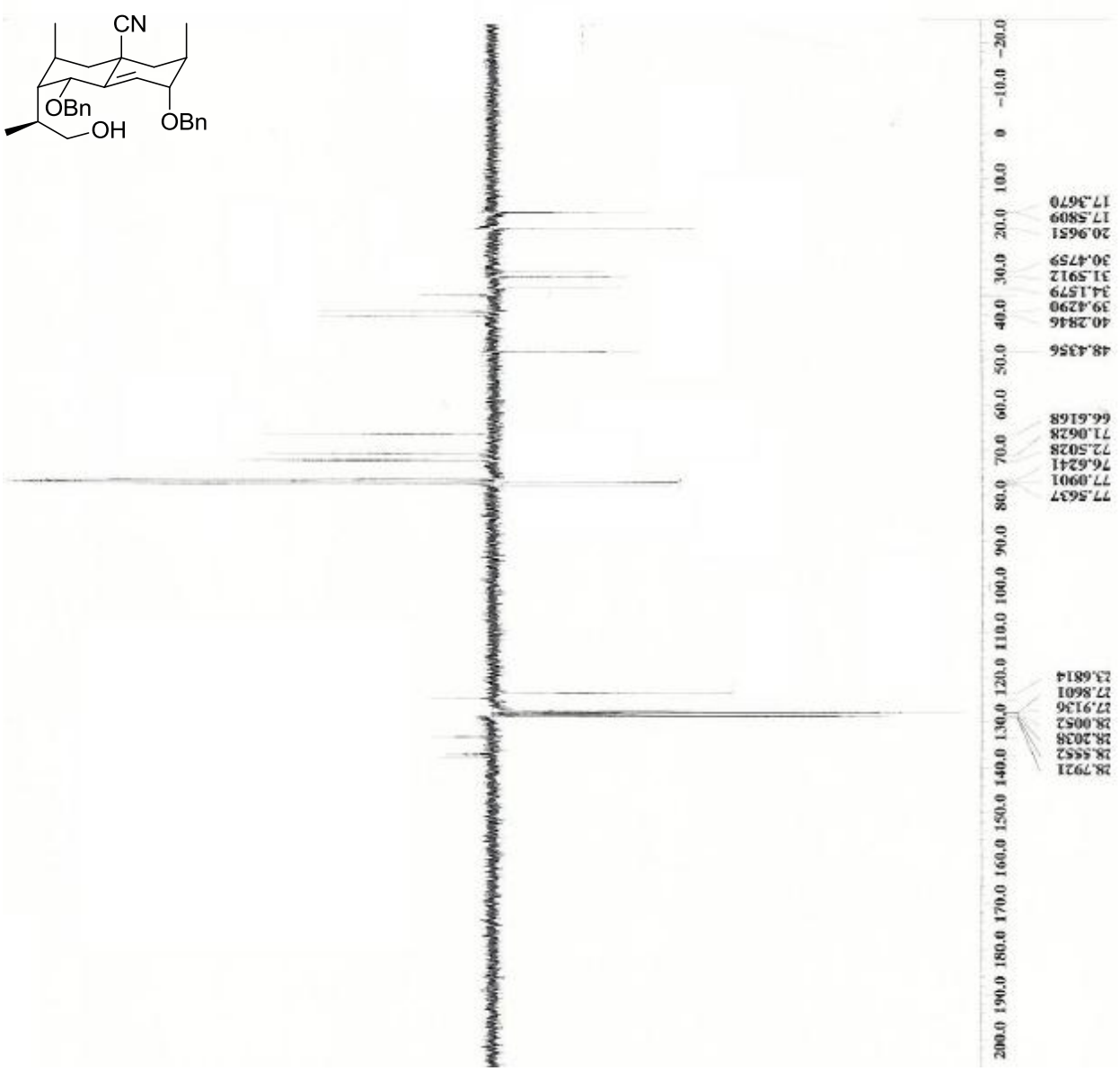
Dimethyl ether alcohol 19a – ^{13}C APT NMR (68 MHz)



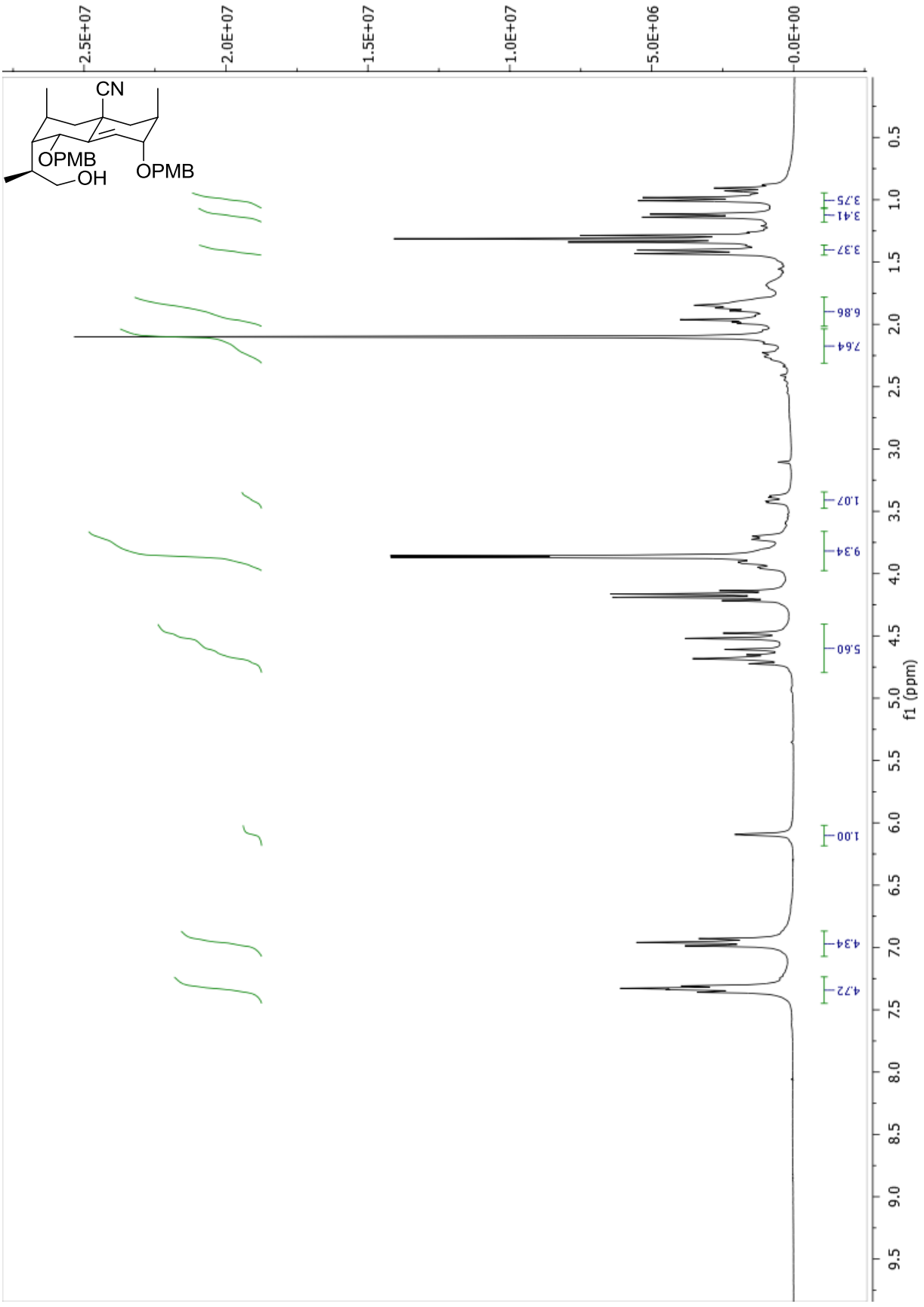
Dibenzyl ether alcohol 19b – ^{1}H NMR (300 MHz)



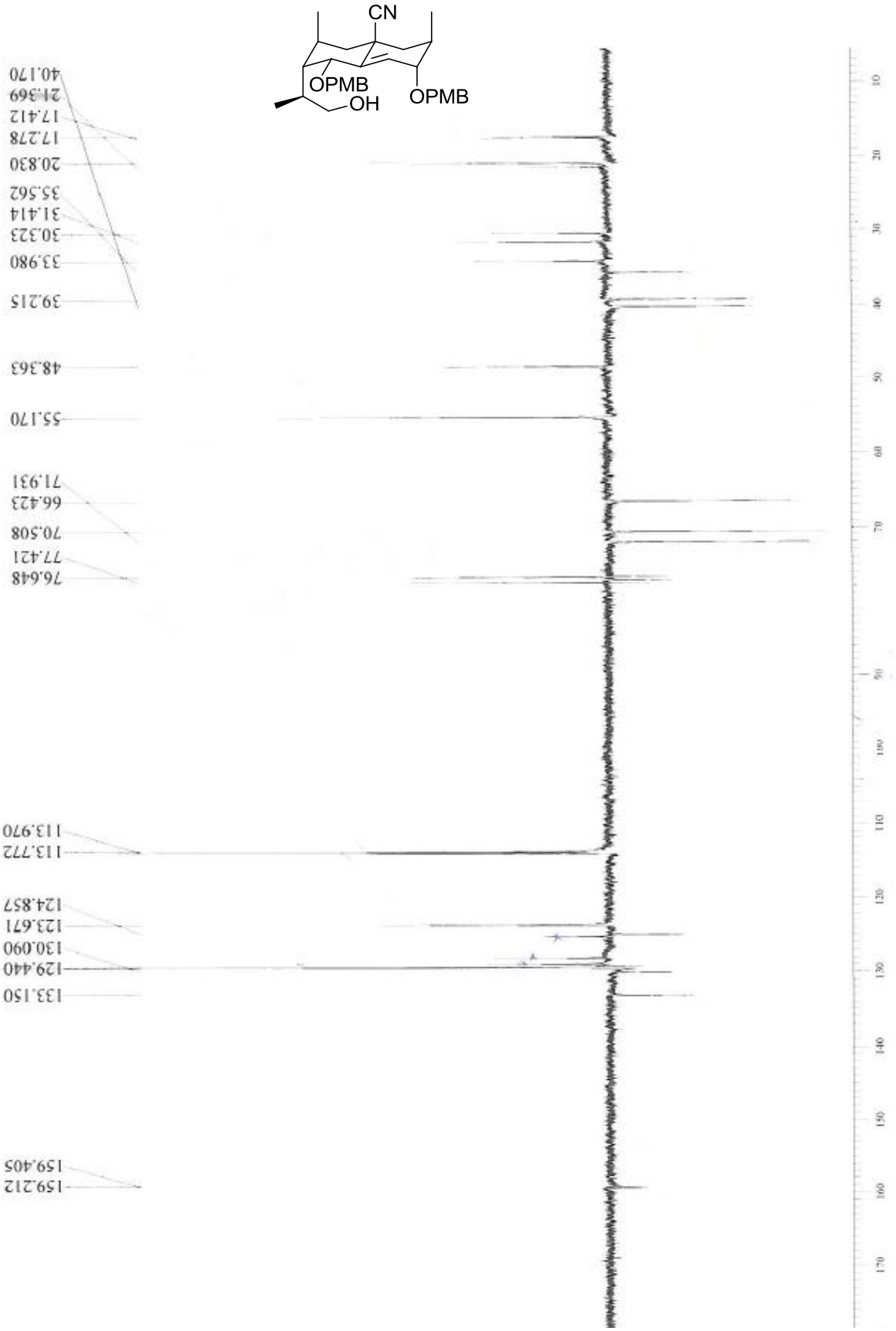
Dibenzyl ether alcohol 19b – ^{13}C APT NMR (75 MHz)



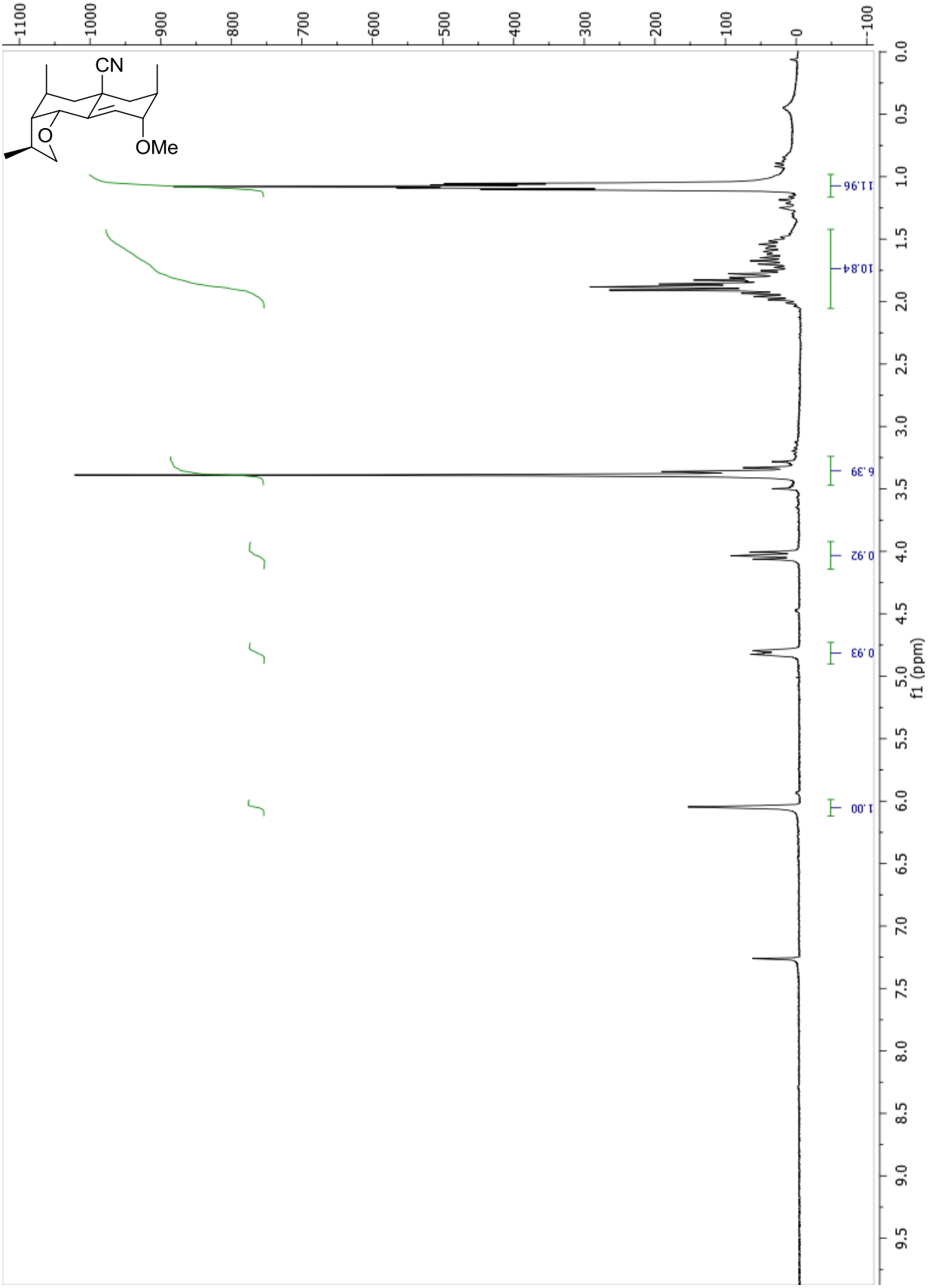
Di-PMB ether alcohol 19c – ^{1}H NMR (270 MHz)



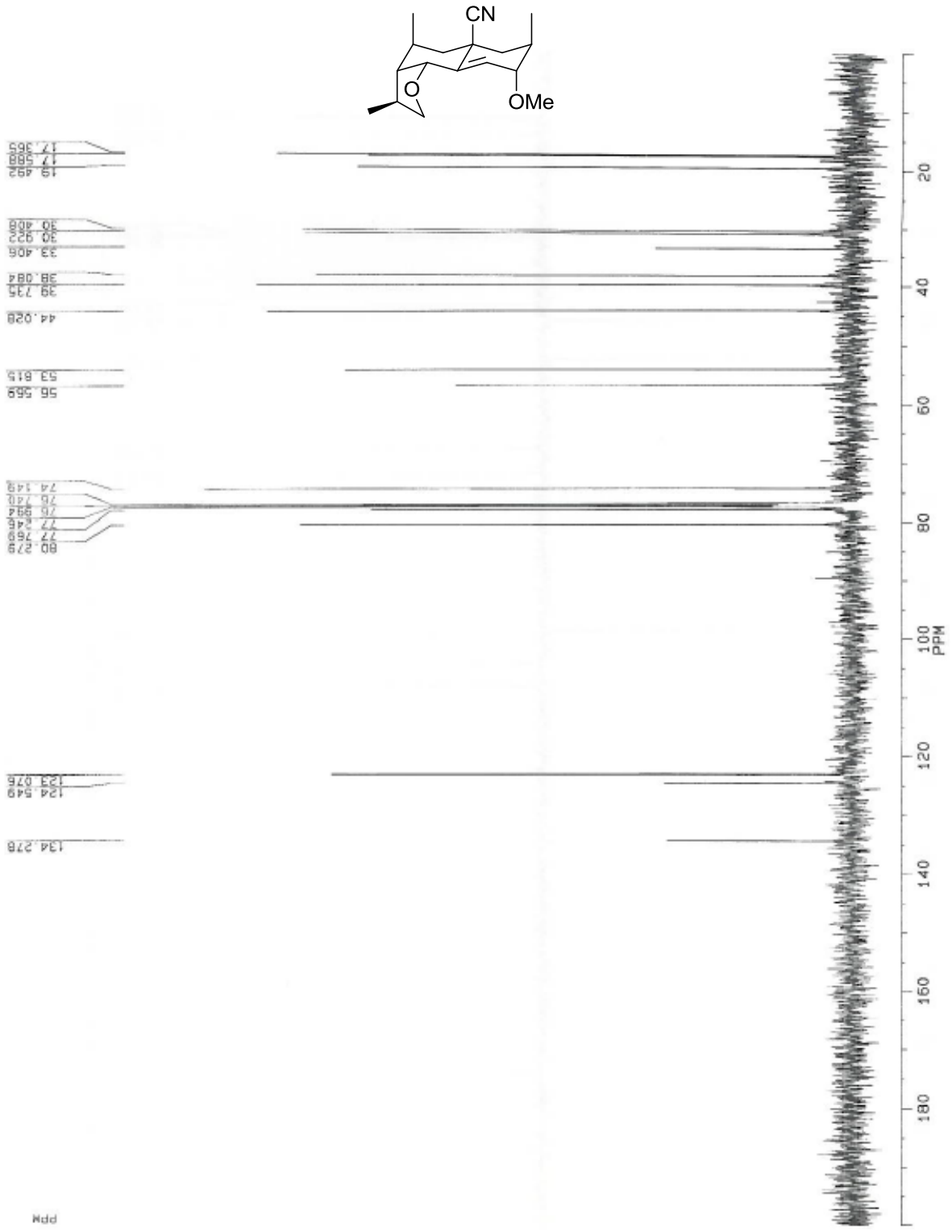
Di-PMB ether alcohol 19c – ^{13}C APT NMR (68 MHz)



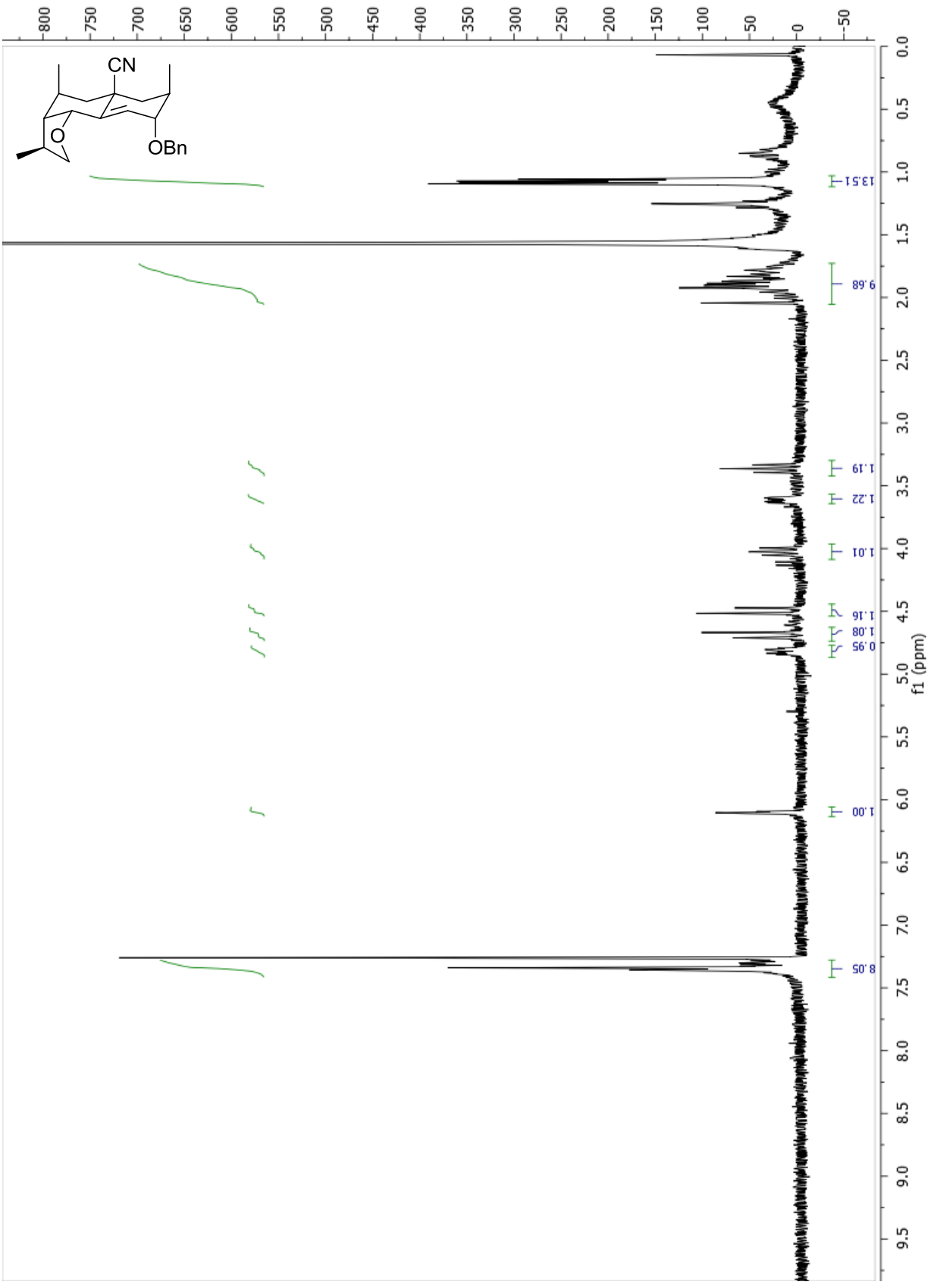
Tetrahydrofuran methyl ether 20a – ^{1}H NMR (270 MHz)



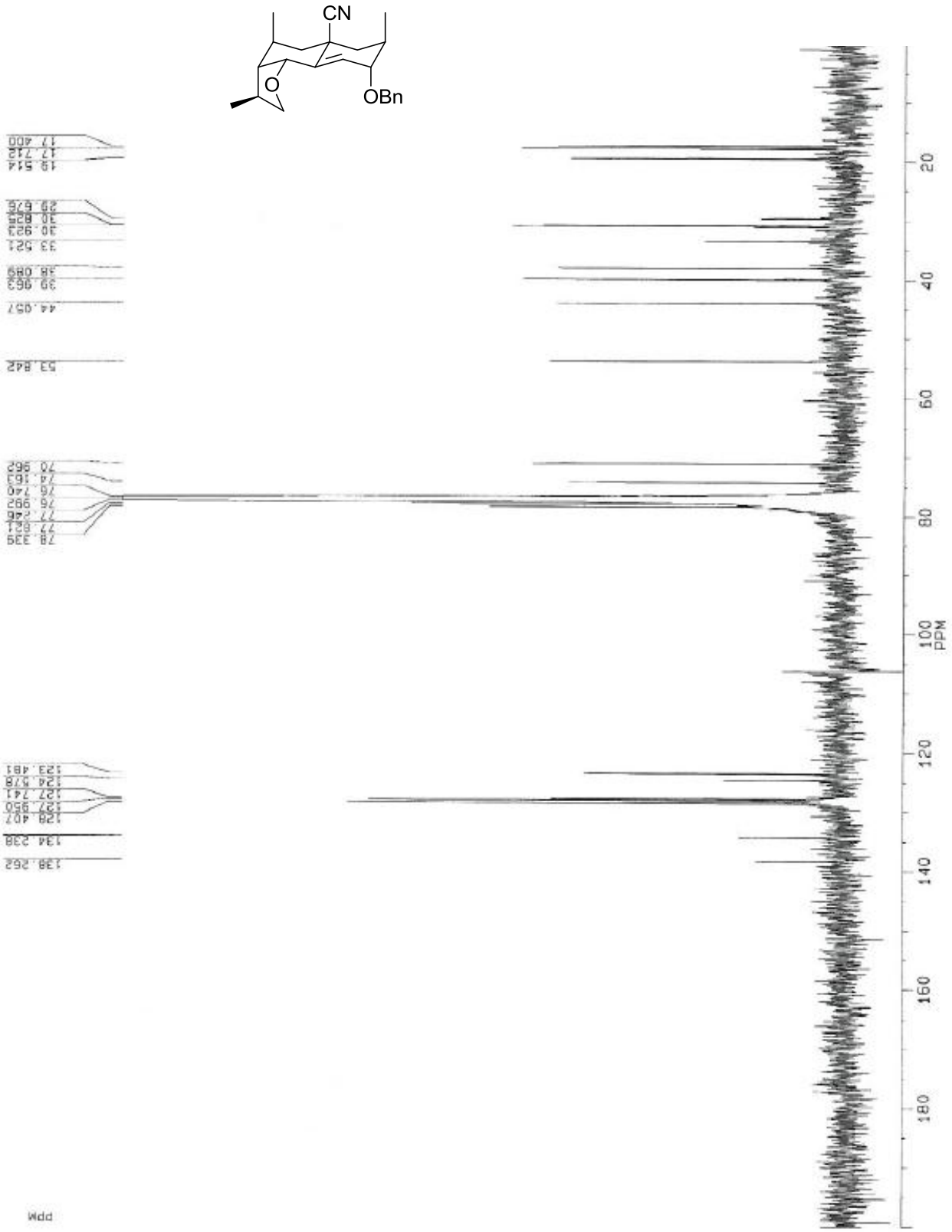
Tetrahydrofuran methyl ether 20a – ^{13}C NMR (68 MHz)



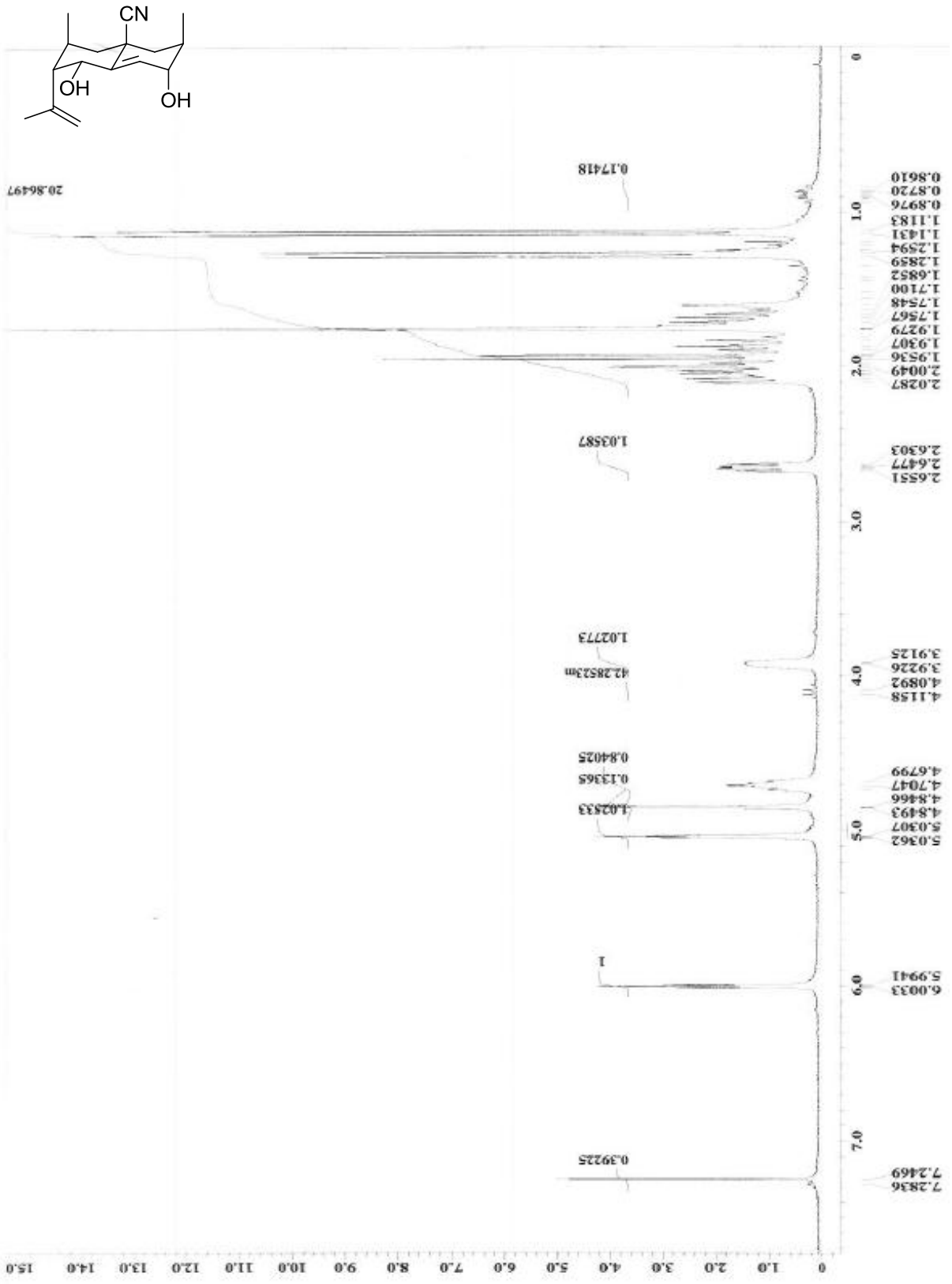
Tetrahydrofuran benzyl ether 20b – ^{1}H NMR (270 MHz)



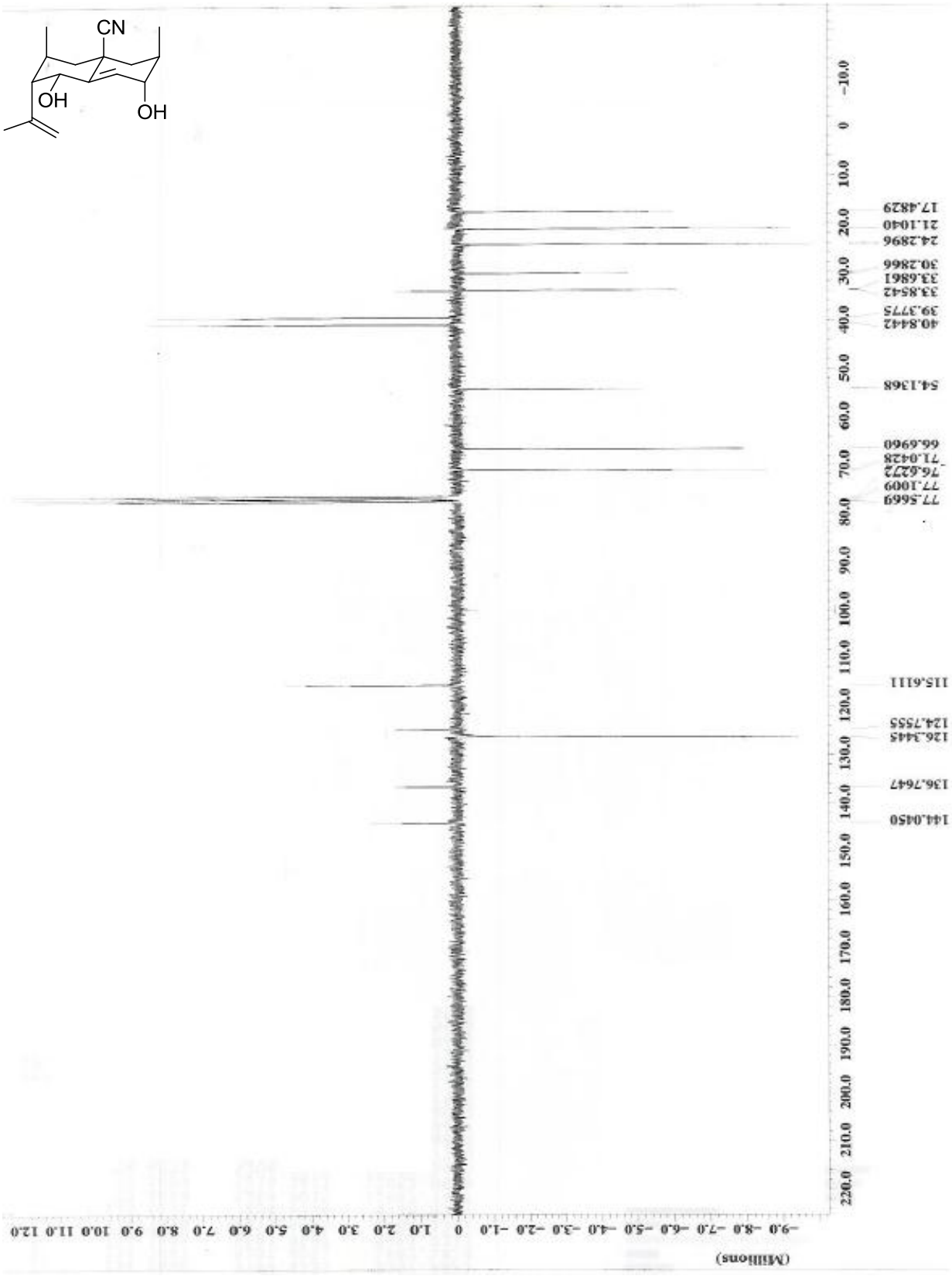
Tetrahydrofuran benzyl ether 20b – ^{13}C NMR (125 MHz)



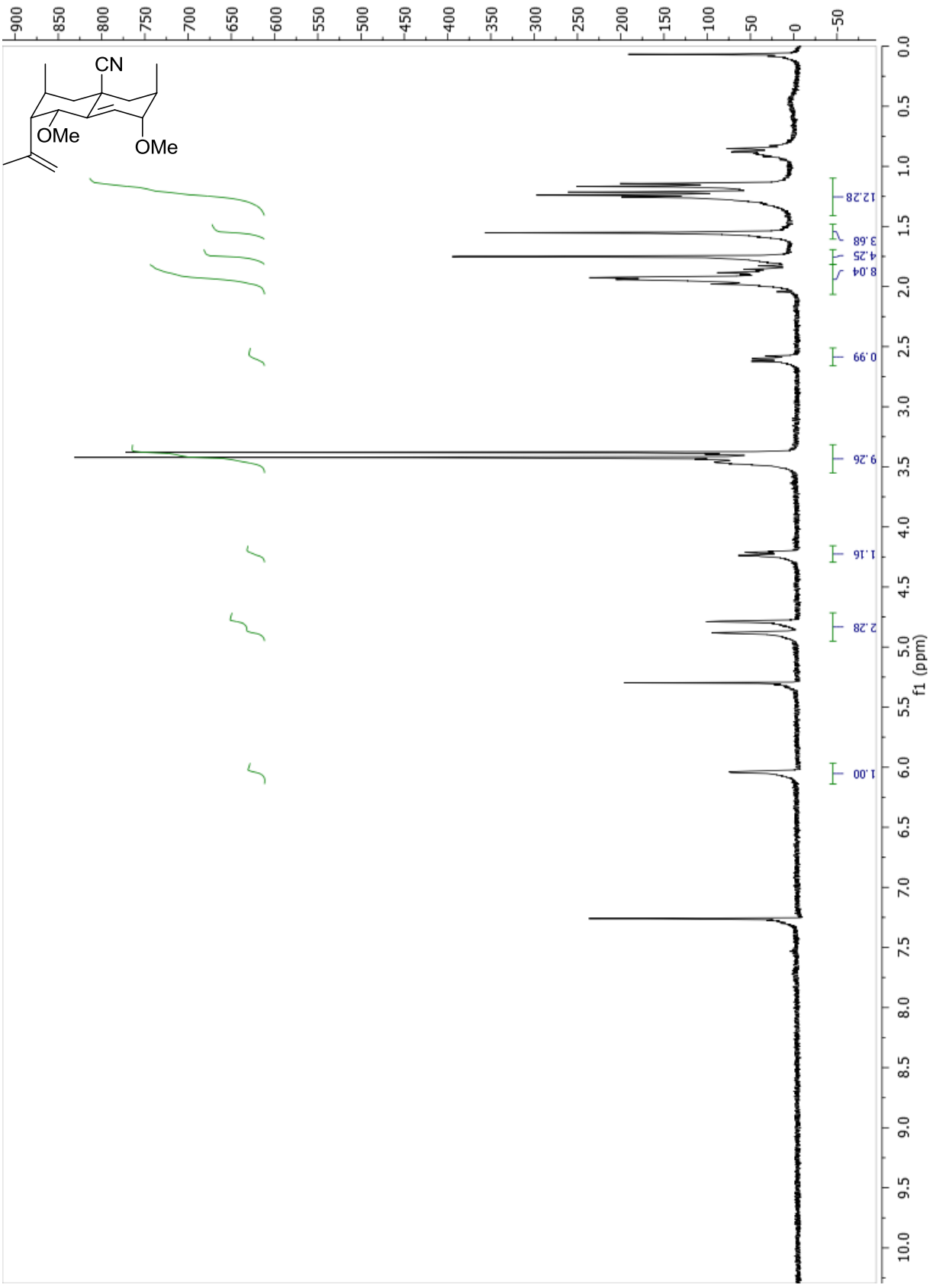
Diene diol 21 – ^{1}H NMR (270 MHz)



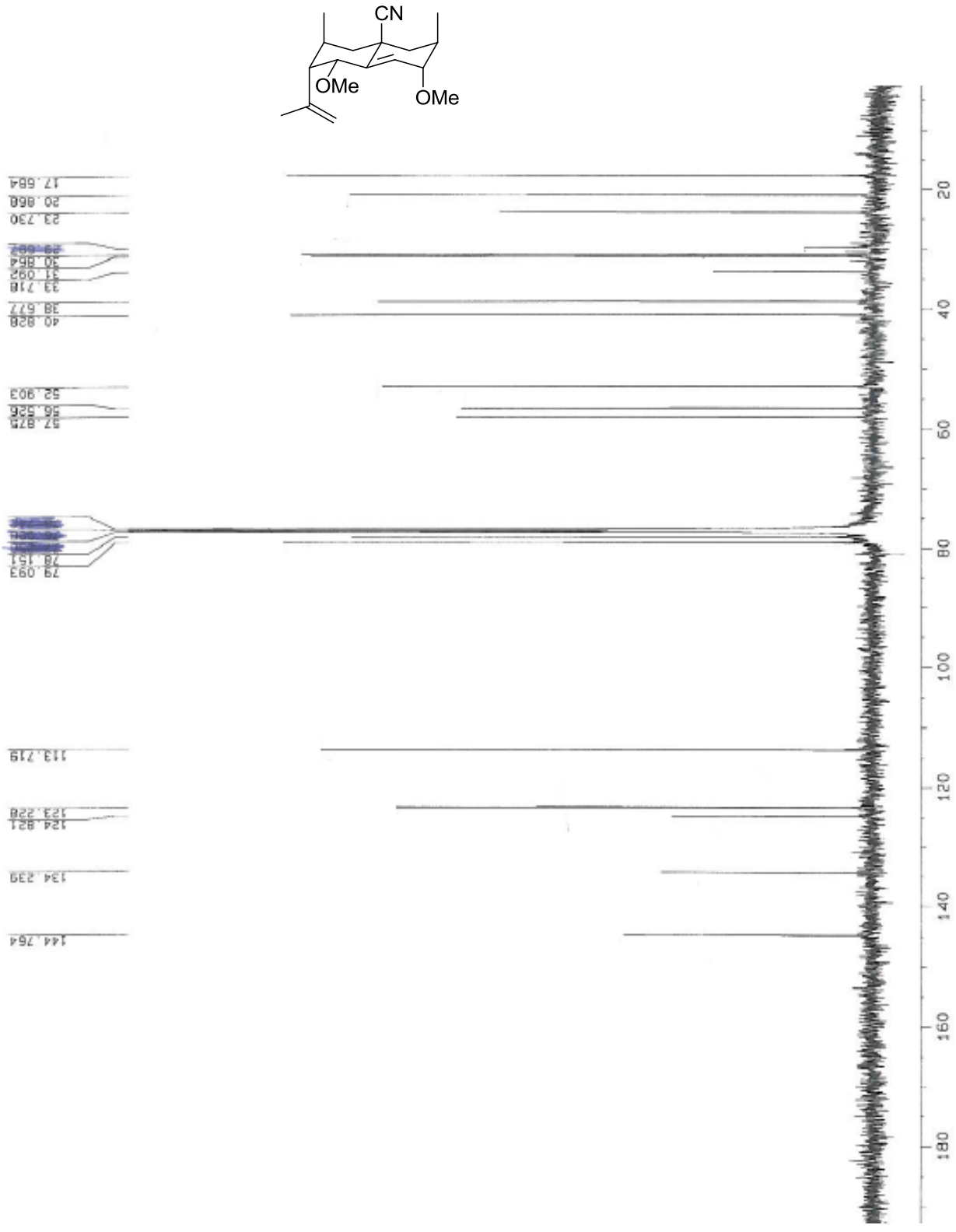
Diene diol 21 – ^{13}C APT NMR (125 MHz)



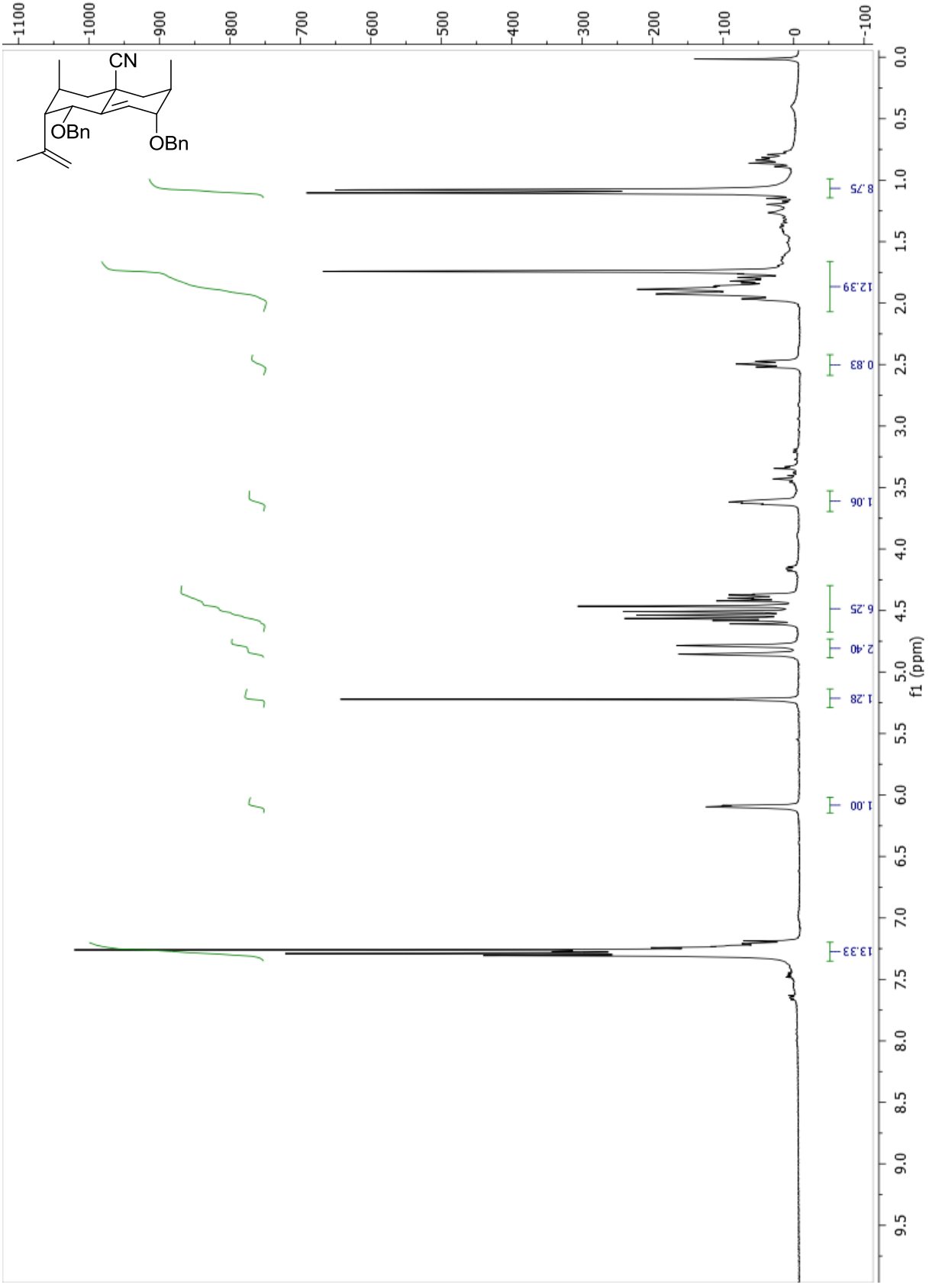
Diene dimethyl ether 21a – ^{1}H NMR (270 MHz)



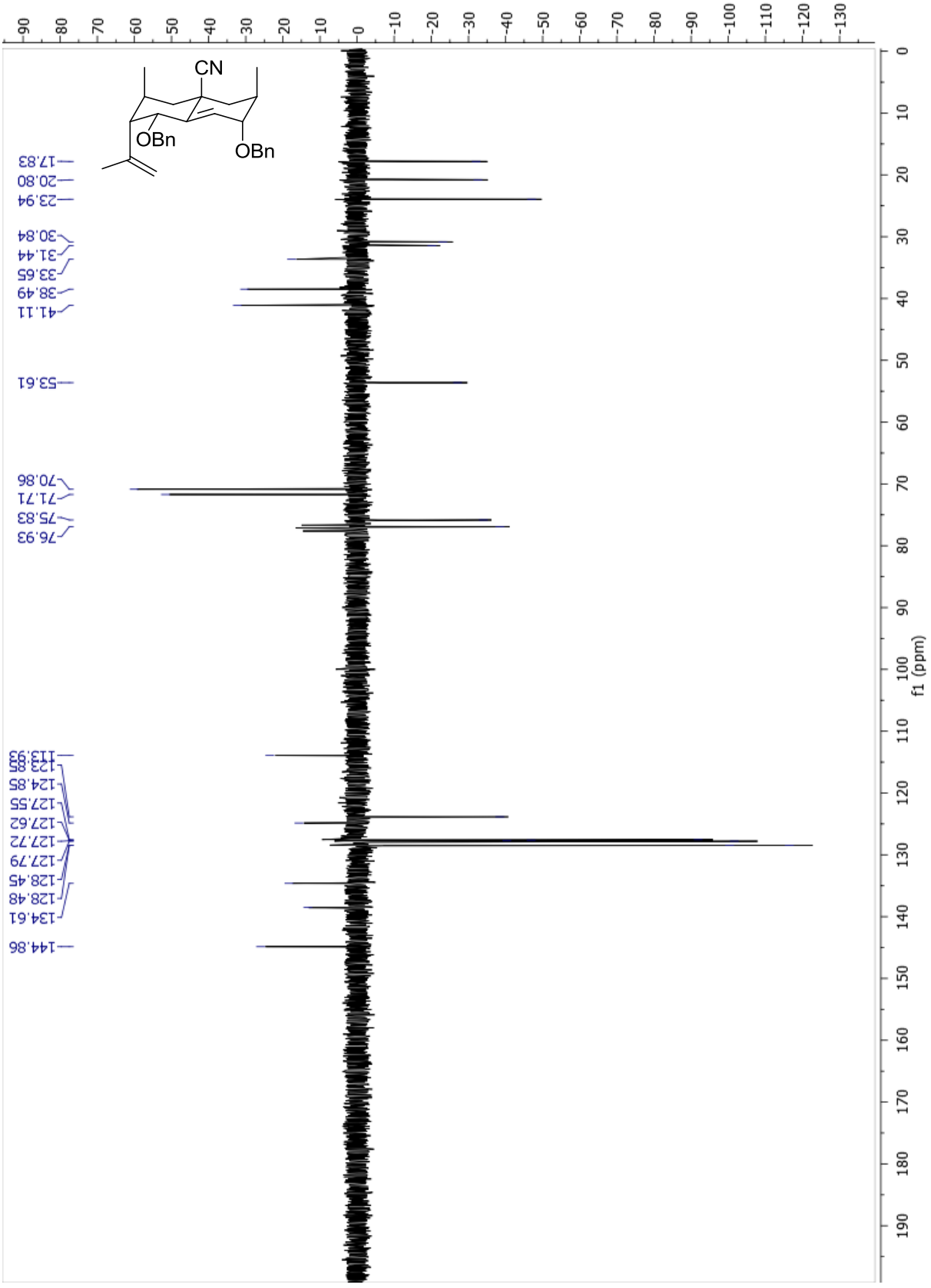
Diene dimethyl ether 21a – ^{13}C NMR (125 MHz)



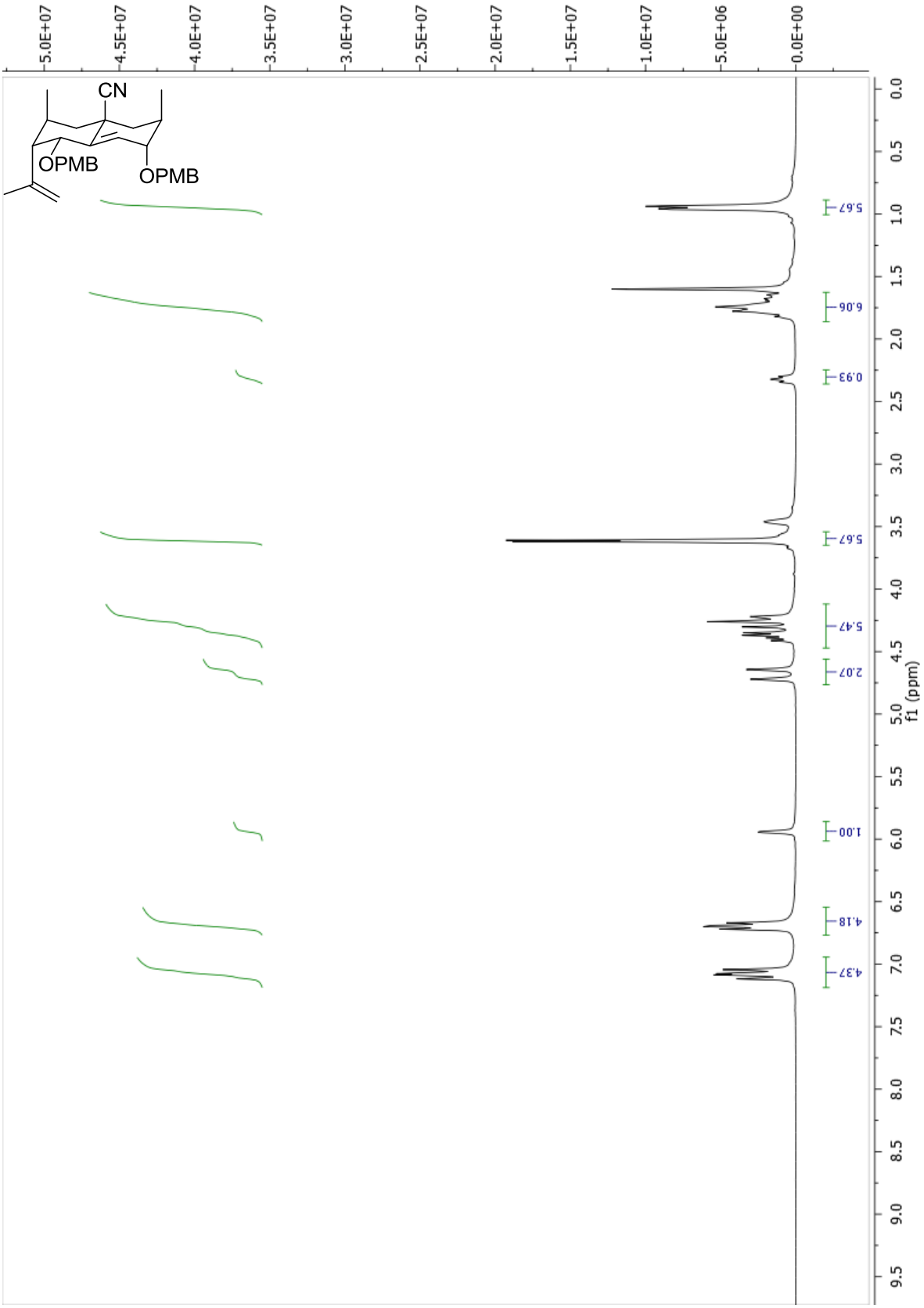
Diene dibenzyl ether 21b – ^{1}H NMR (270 MHz)



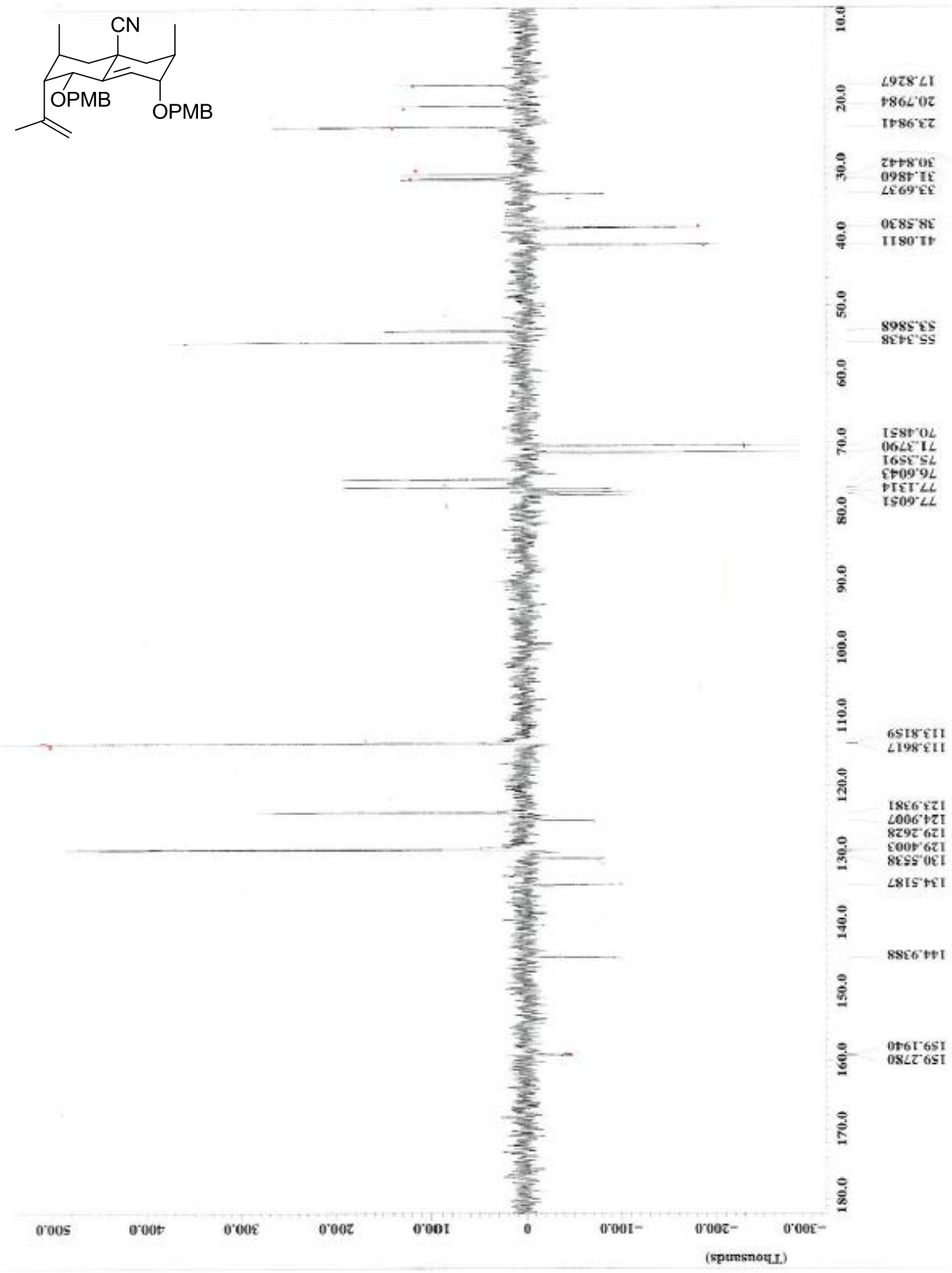
Diene dibenzyl ether 21b – ^{13}C APT NMR (100 MHz)



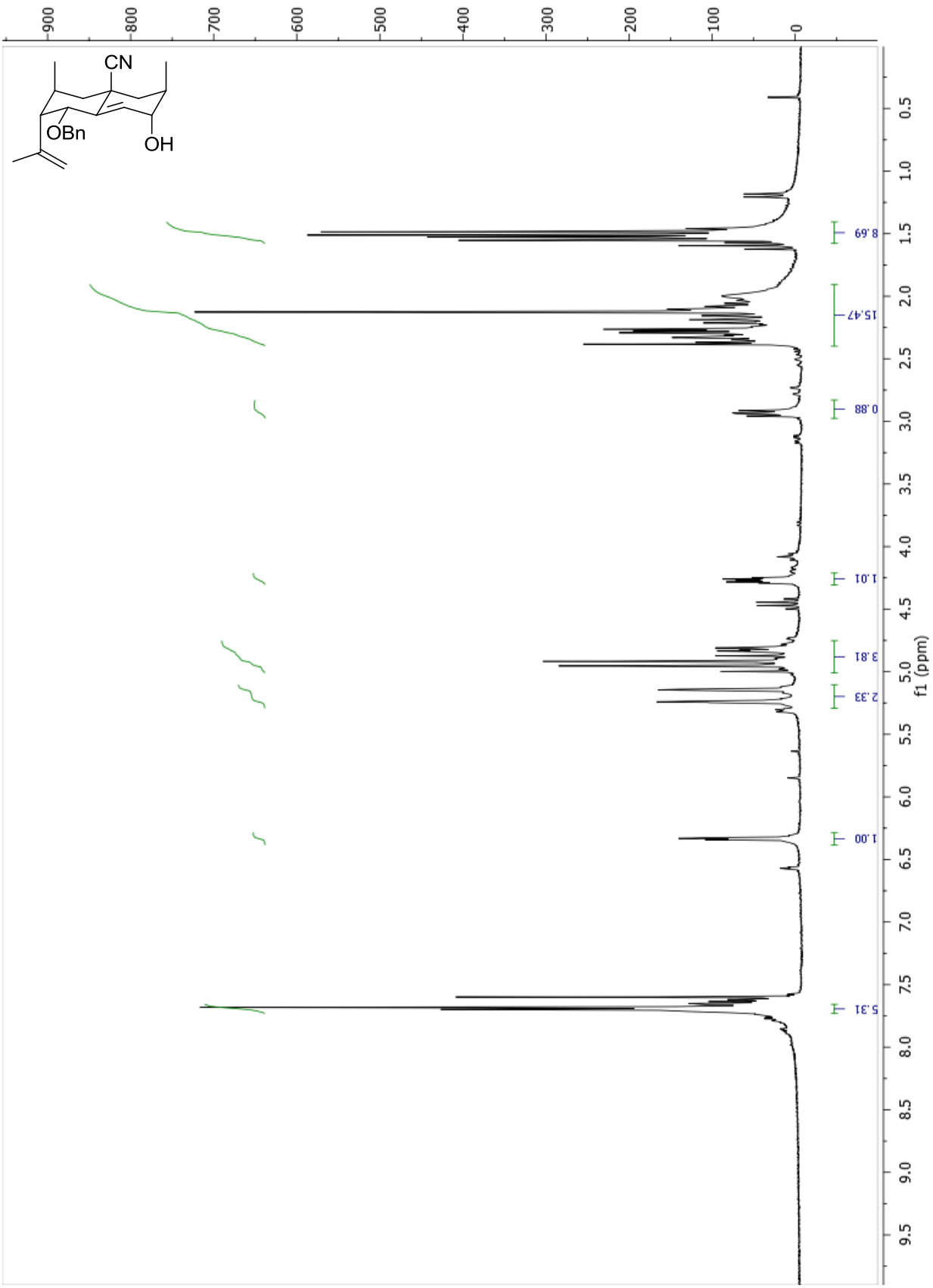
Diene di-PMB ether 21c – ^{1}H NMR (270 MHz)



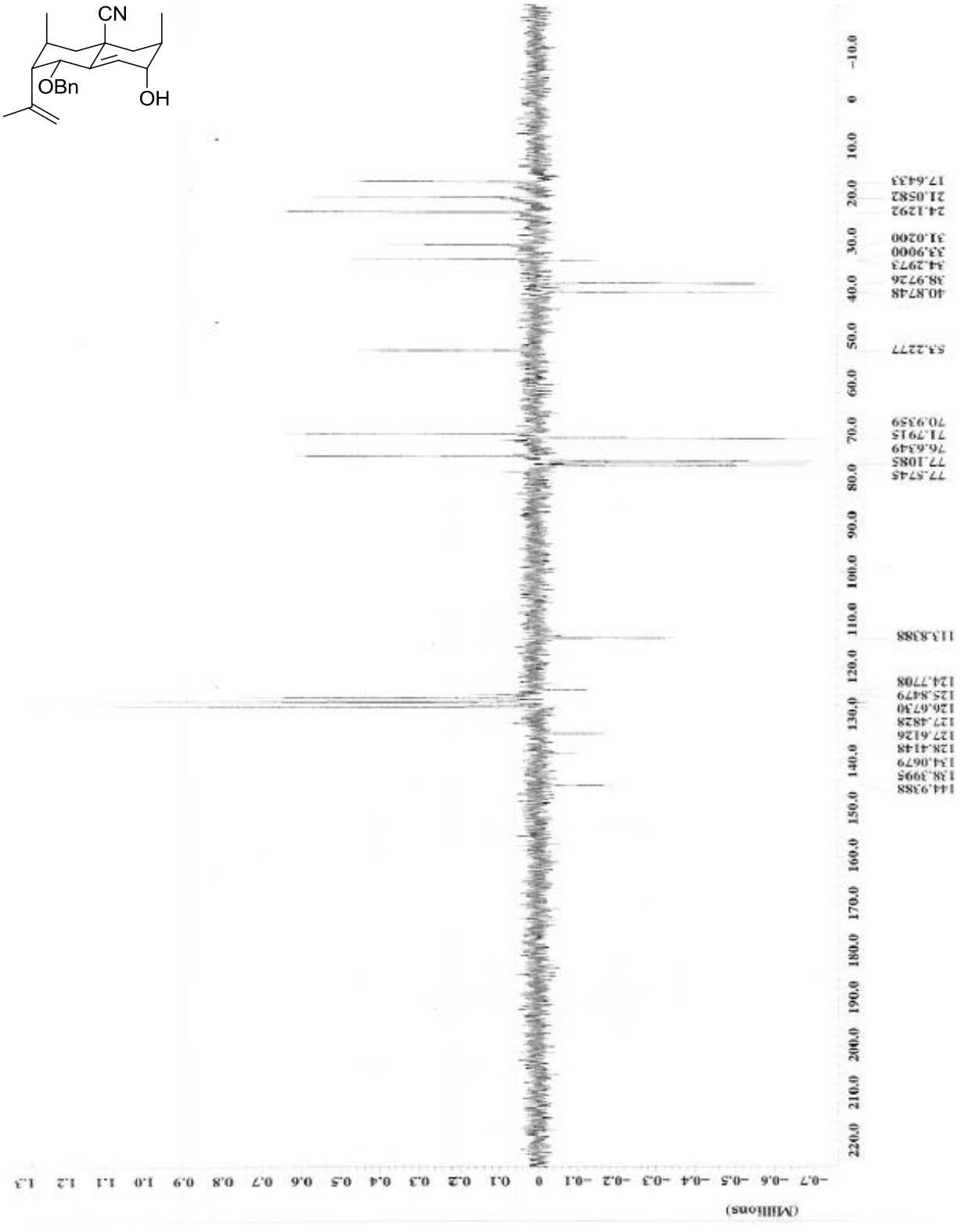
Diene di-PMB ether 21c – ^{13}C APT NMR (68 MHz)



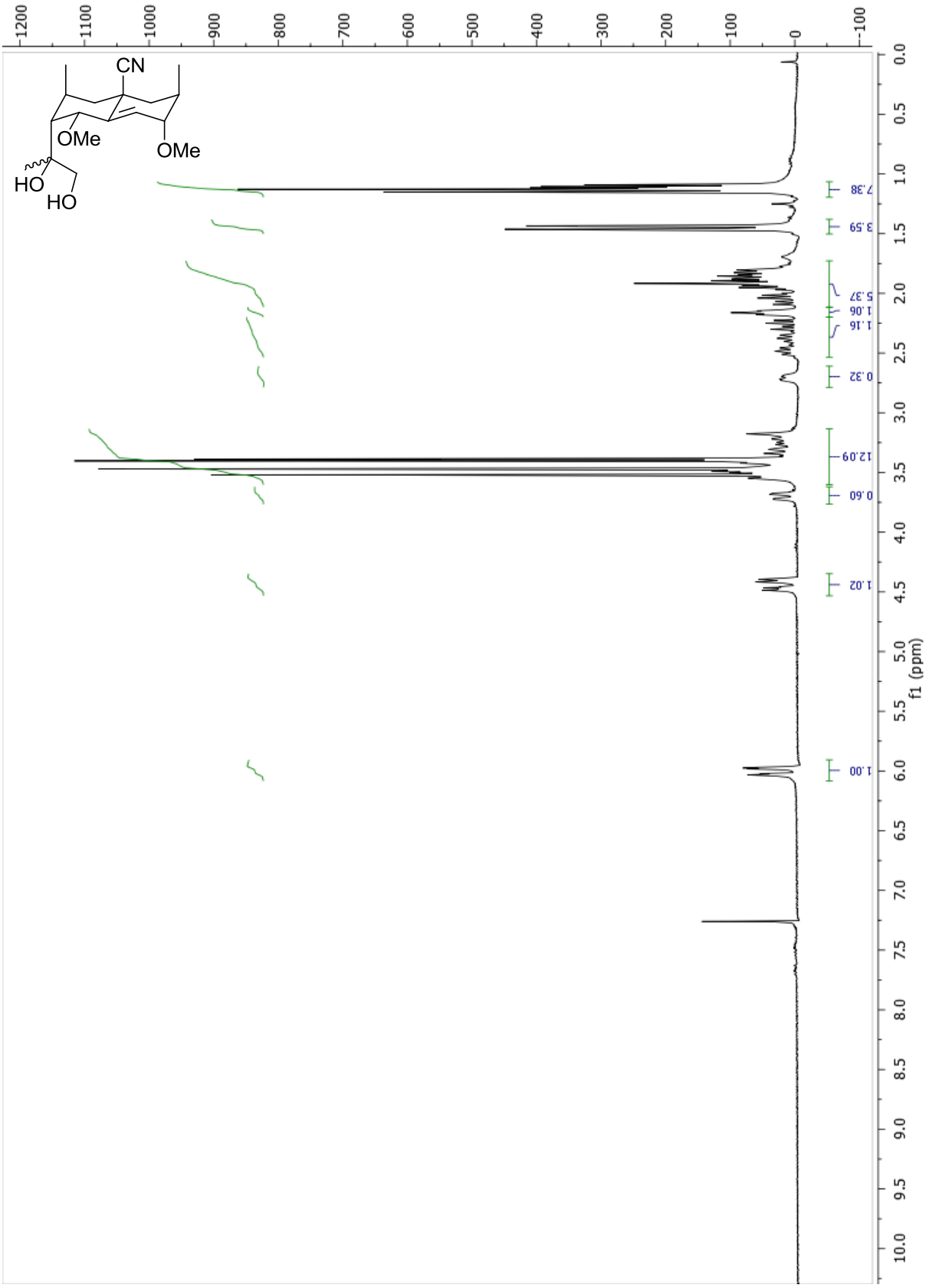
Diene momobenzyl ether 21d – ^{1}H NMR (270 MHz)



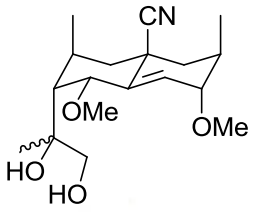
Diene momobenzyl ether 21d – ^{13}C APT NMR (68 MHz)



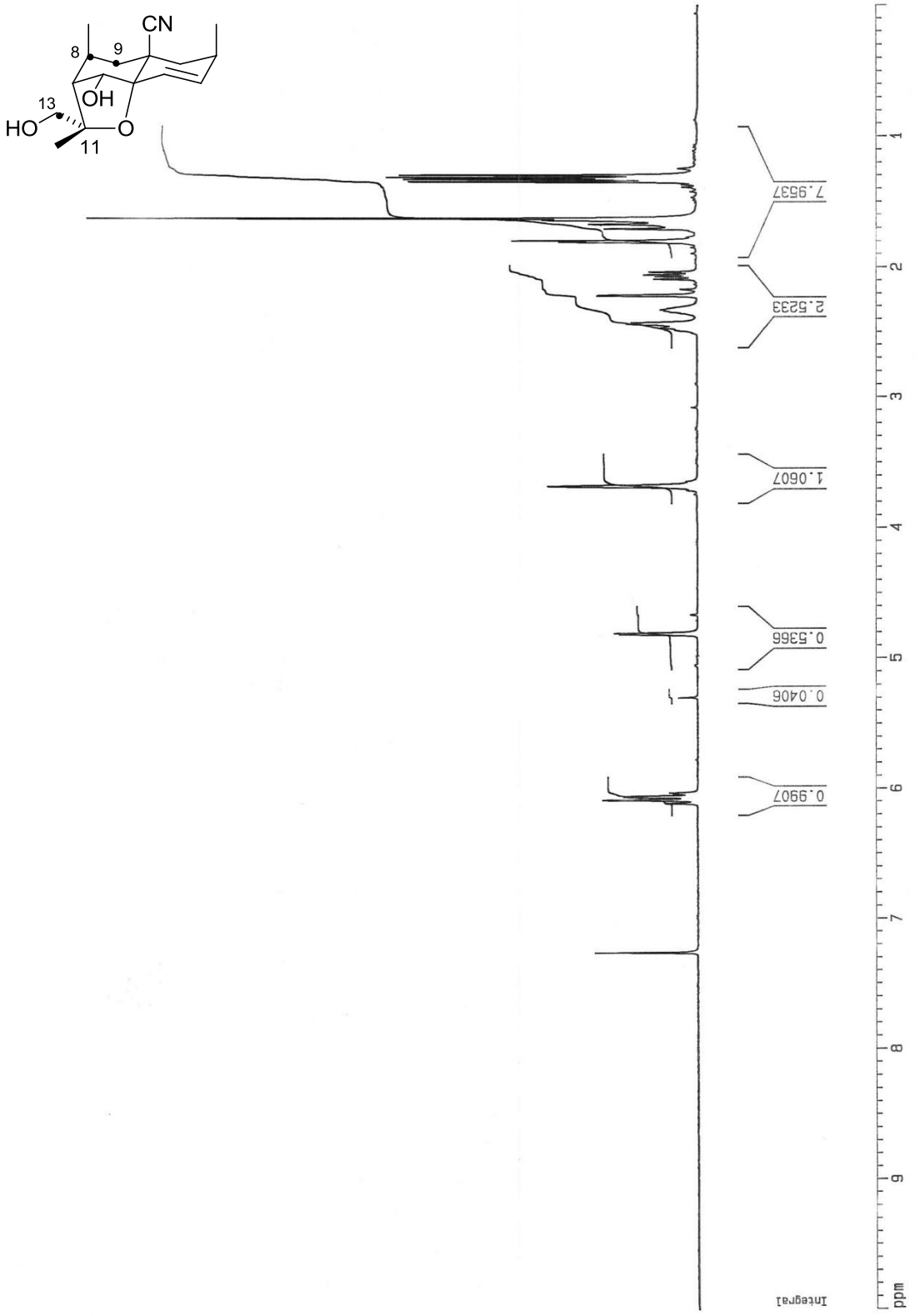
Diol 22 – ^{1}H NMR (270 MHz)



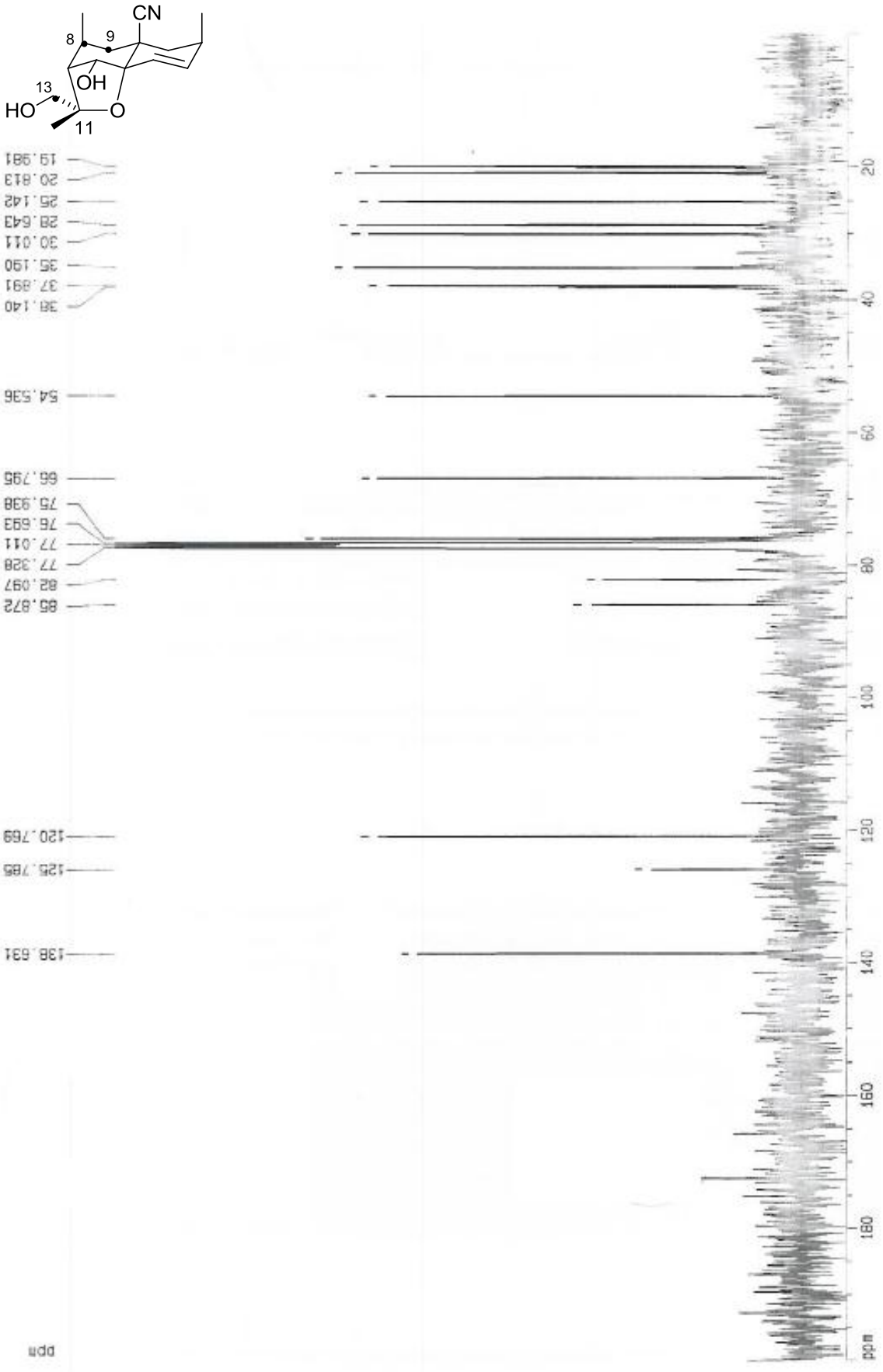
Diol 22 – ^{13}C NMR (100 MHz)



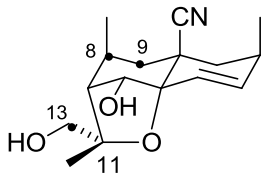
β-Dihydroagarofuran 23 – ^{1}H NMR (400 MHz)



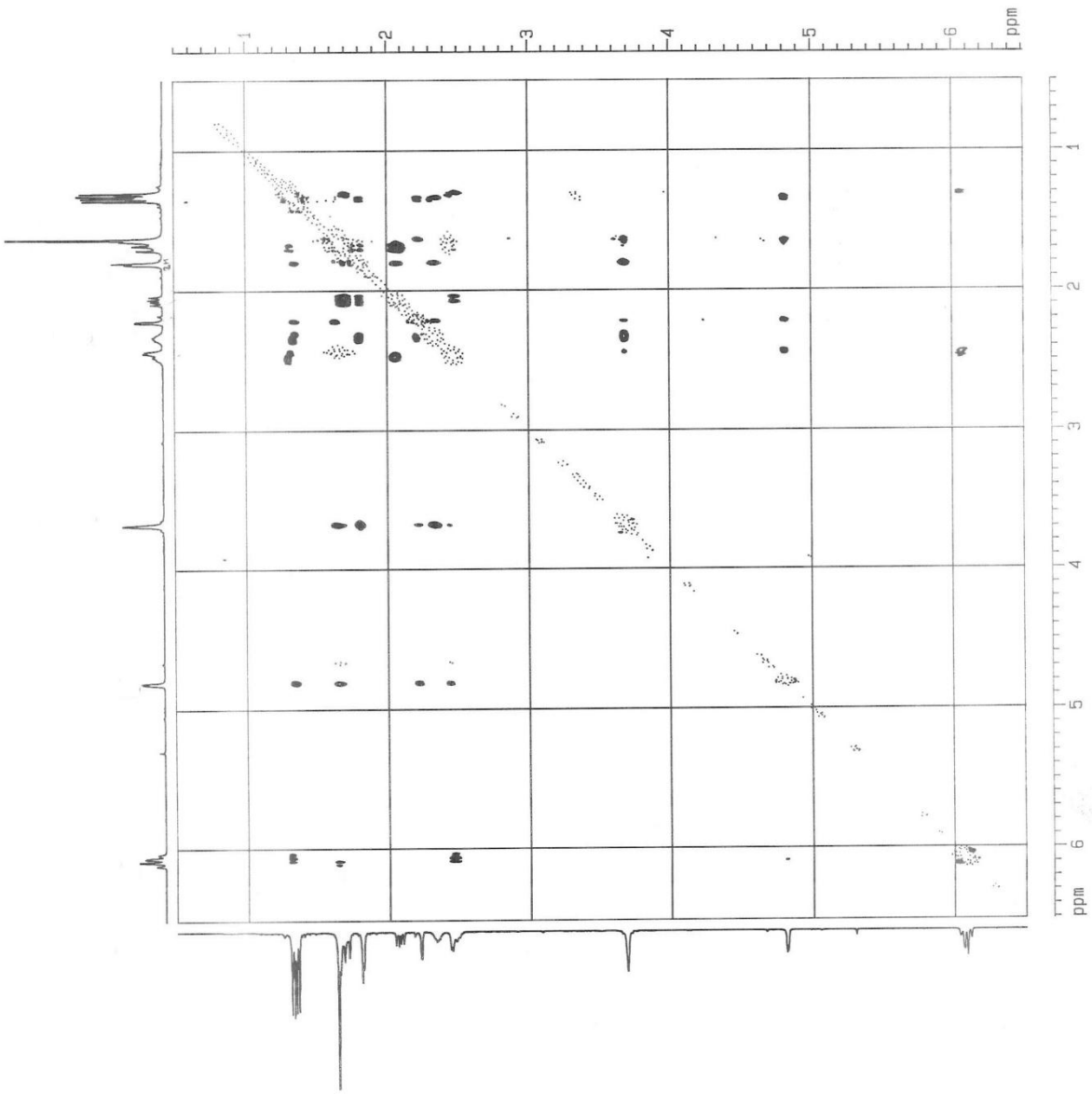
β-Dihydroagarofuran 23 – ^{13}C NMR (100 MHz)



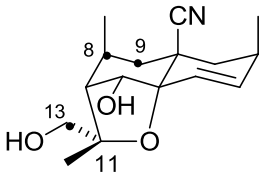
β-Dihydroagarofuran 23 – NOESY (400 MHz)

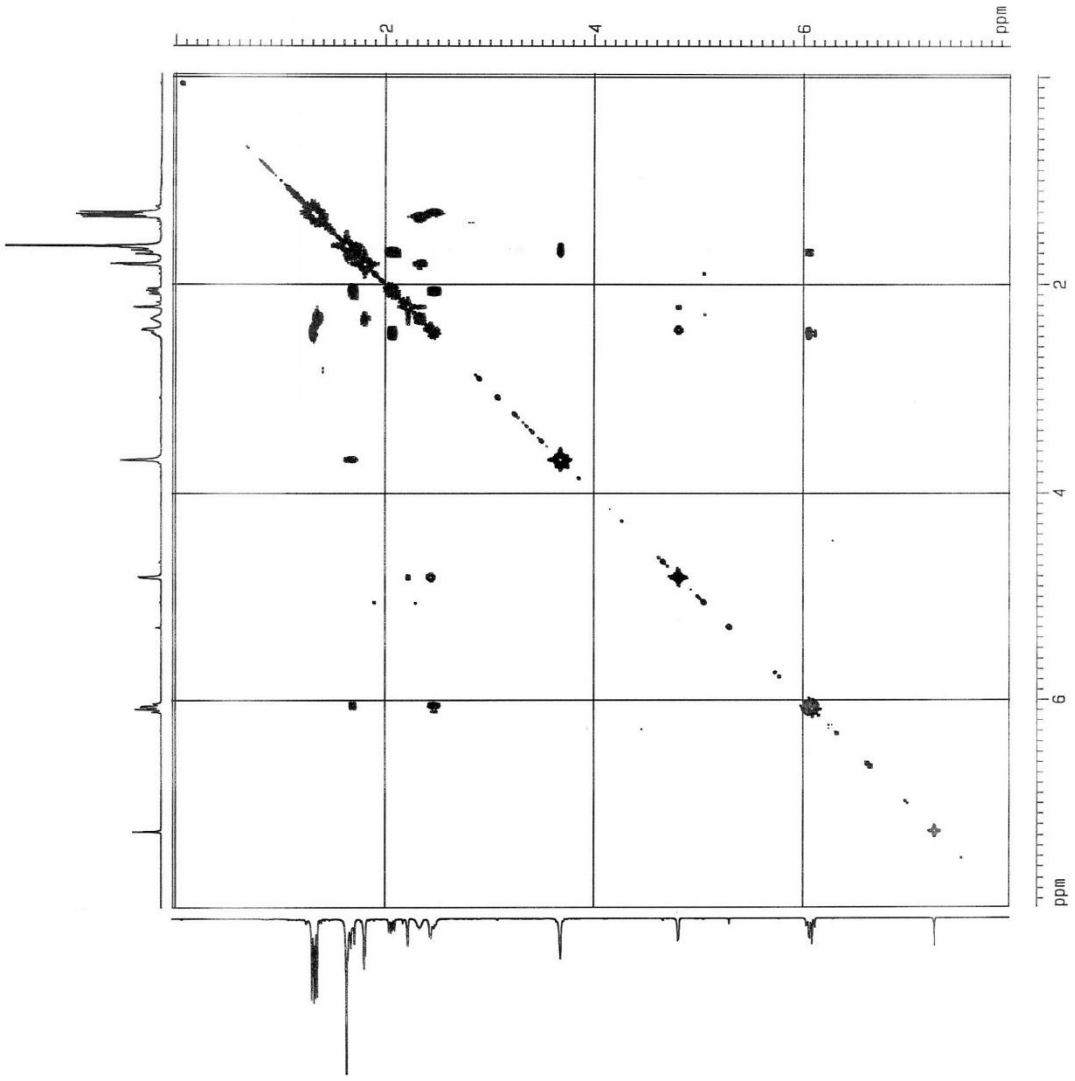


NOESY	13	8	9
13	-	✔	✔
8	✔	-	✔
9	✔	✔	-

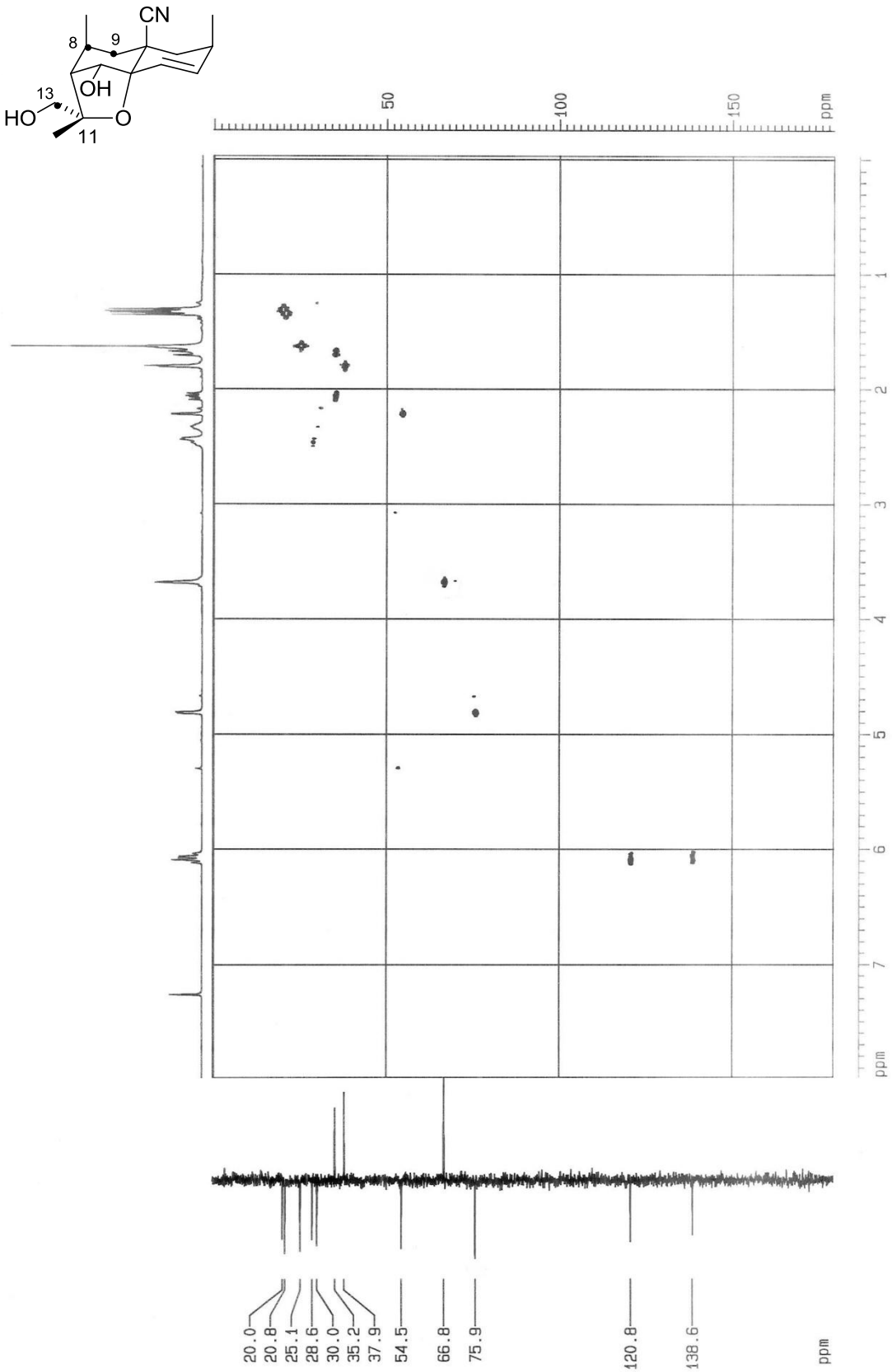


β-Dihydroagarofuran 23 – COSY (400 MHz)

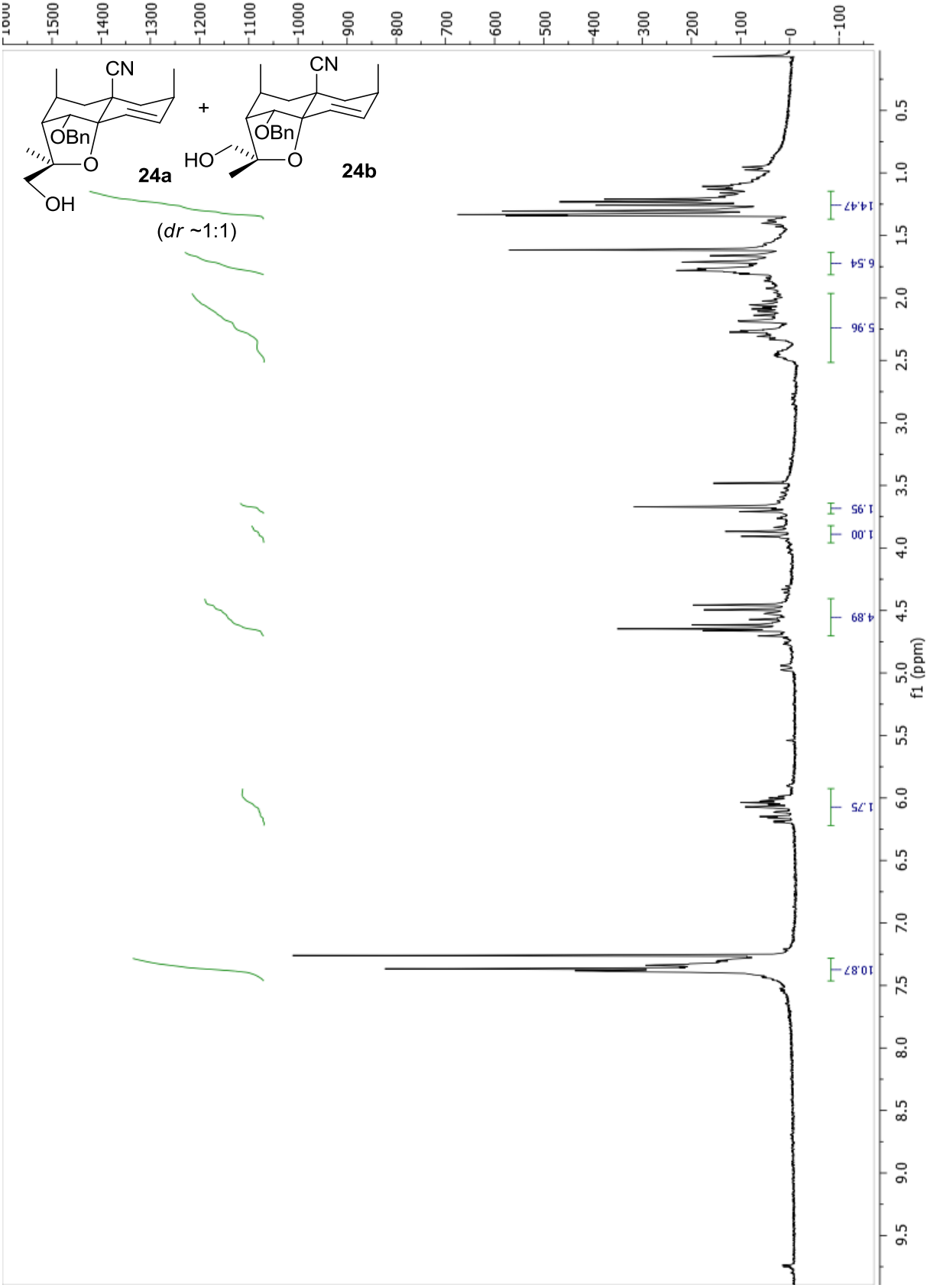




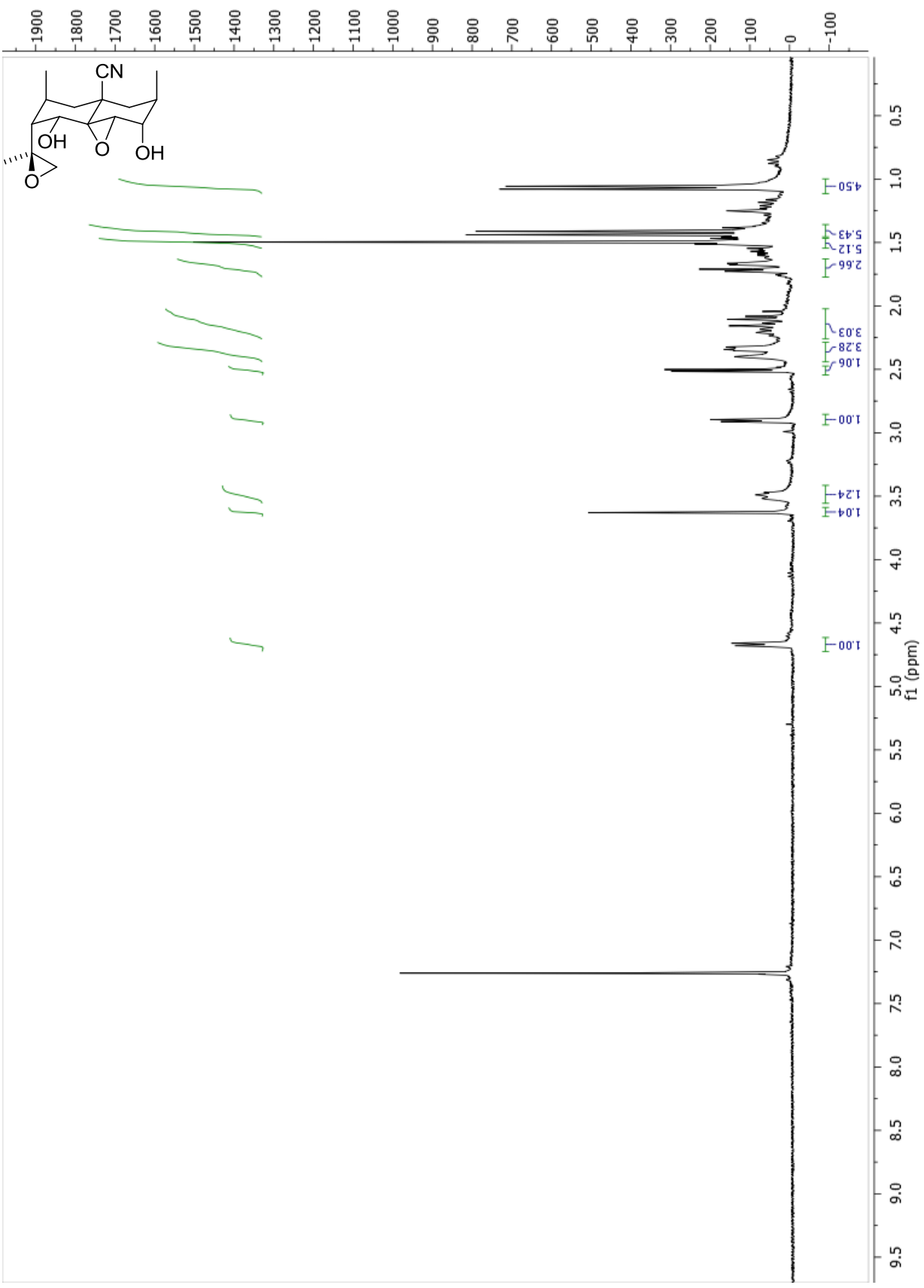
β-Dihydroagarofuran 23 – HMQC (400 MHz)



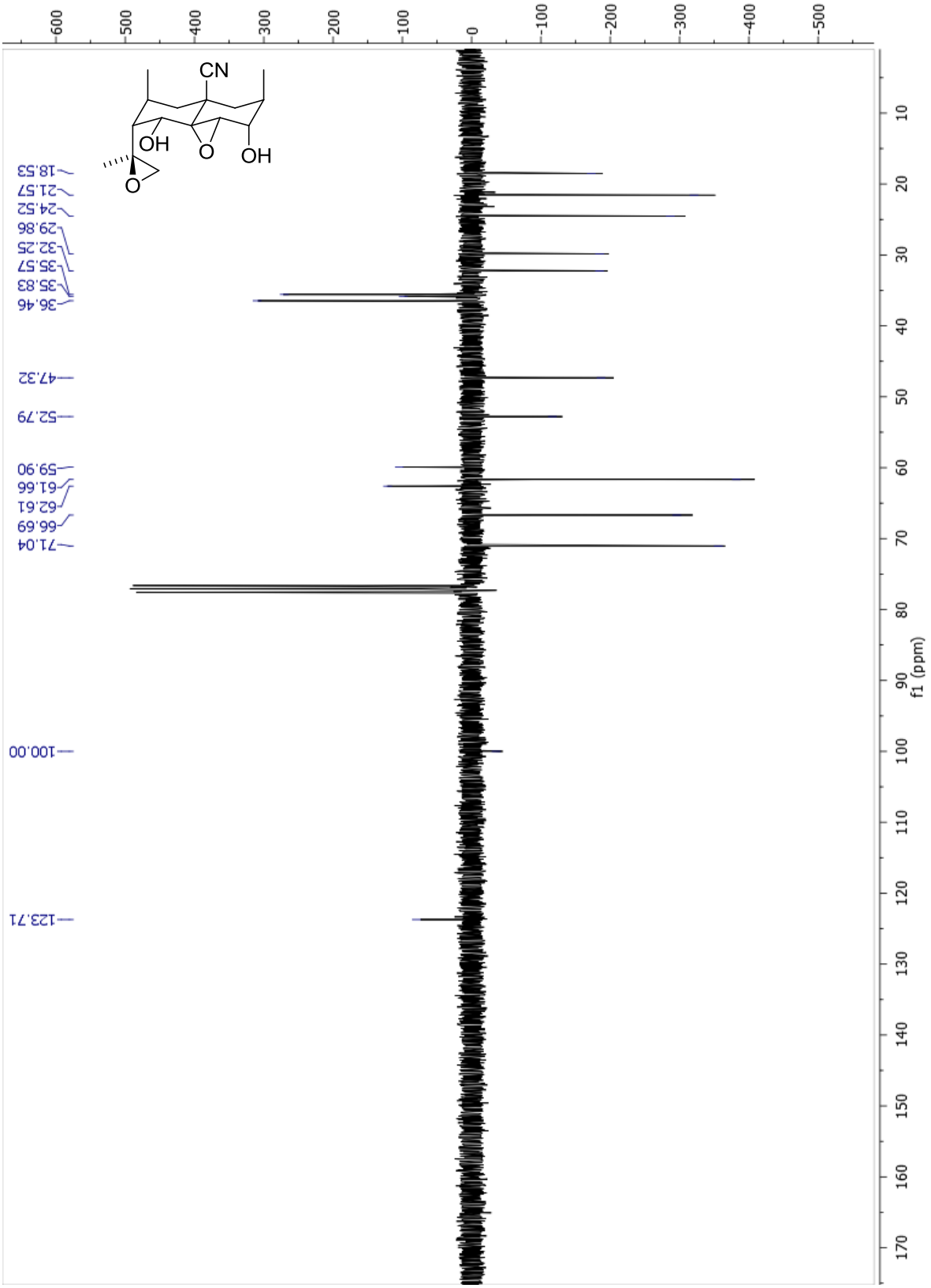
β-Dihydroagarofurans 24a/24b – ^{1}H NMR (270 MHz)



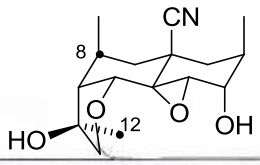
bis-Epoxide 25 – ^{1}H NMR (270 MHz)

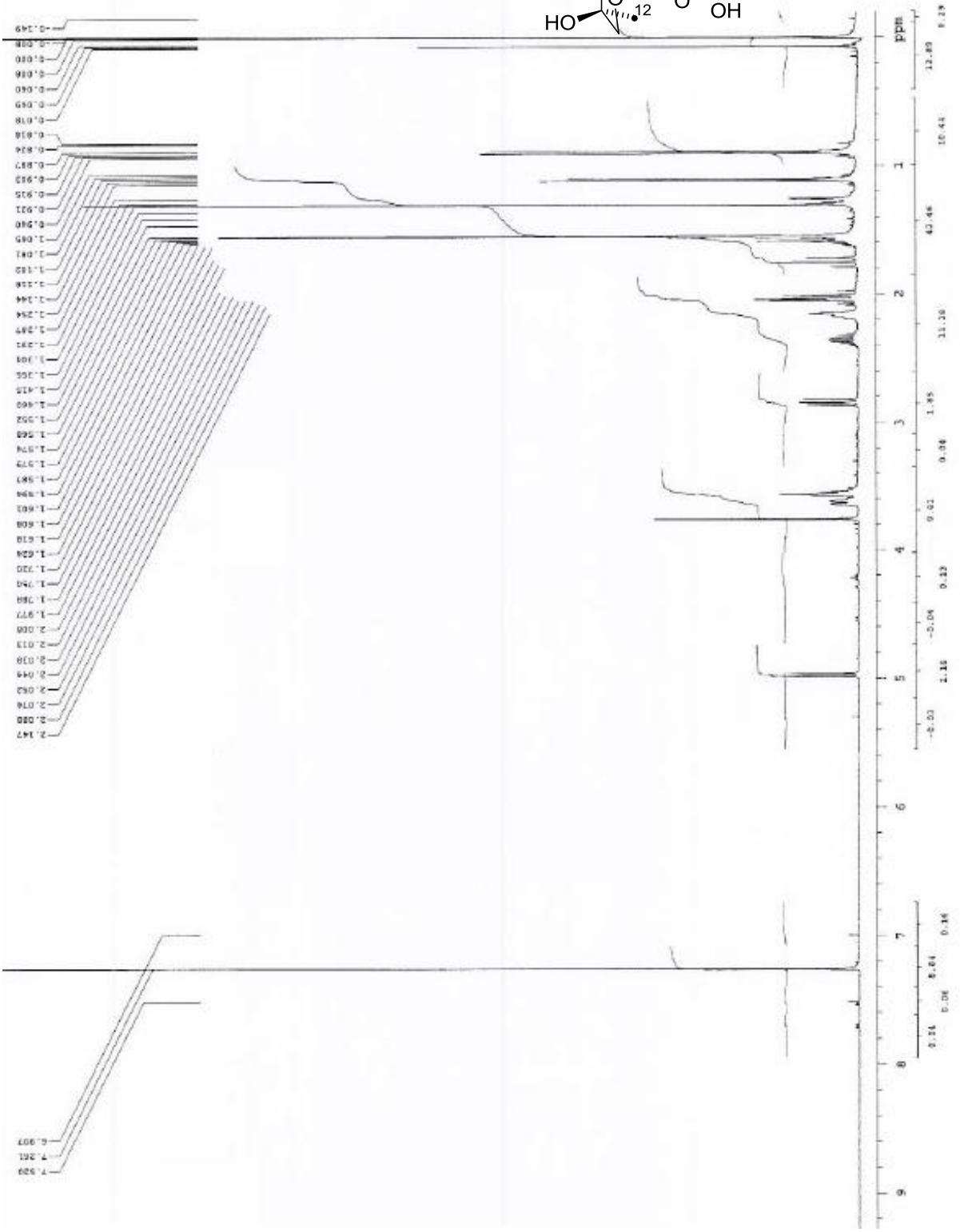


bis-Epoxide 25 – ^{13}C DEPT NMR (68 MHz)

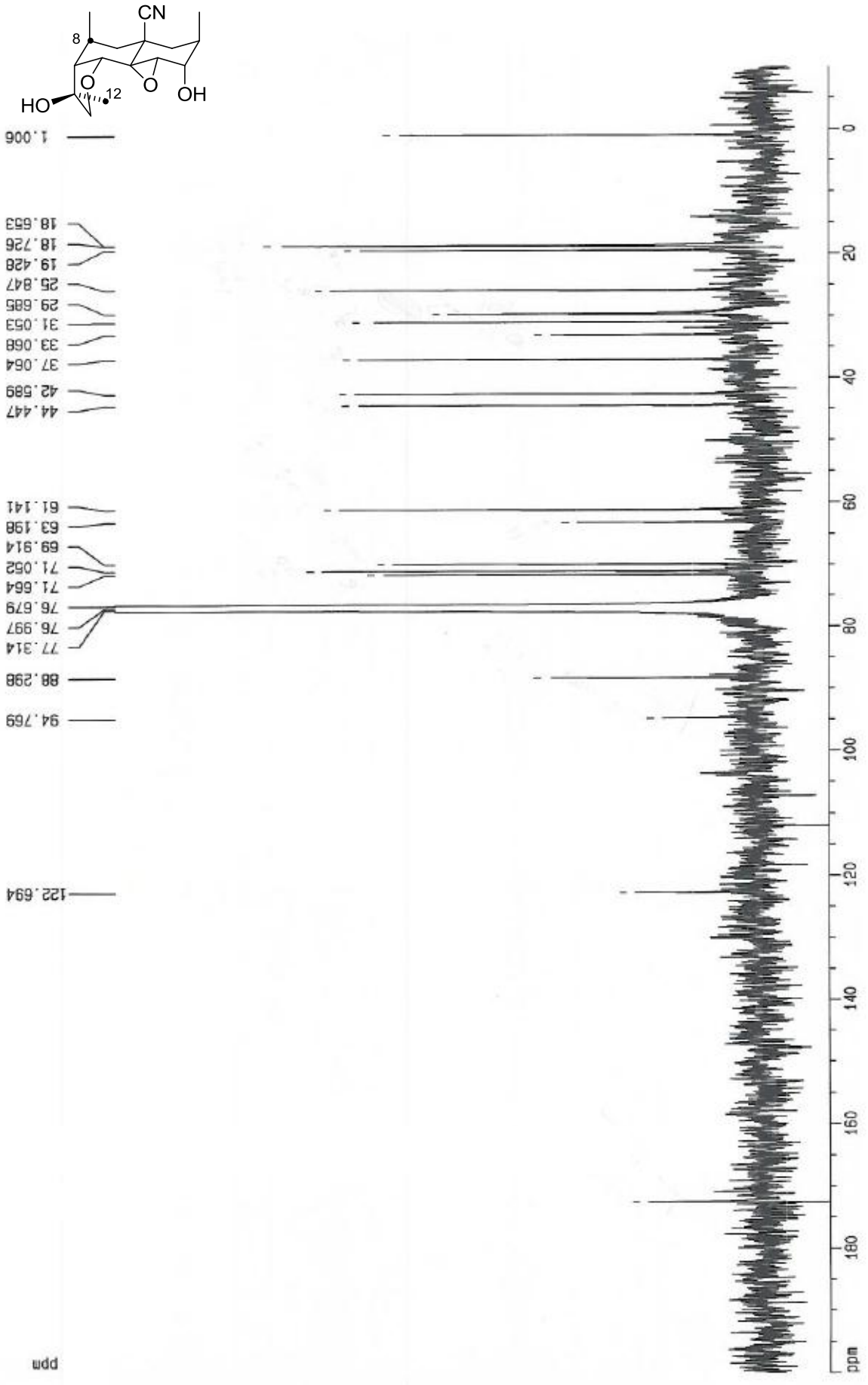


β-Dihydroagarofuran 26 – ^{1}H NMR (400 MHz)

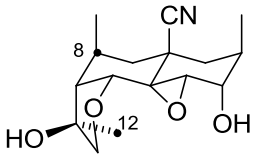




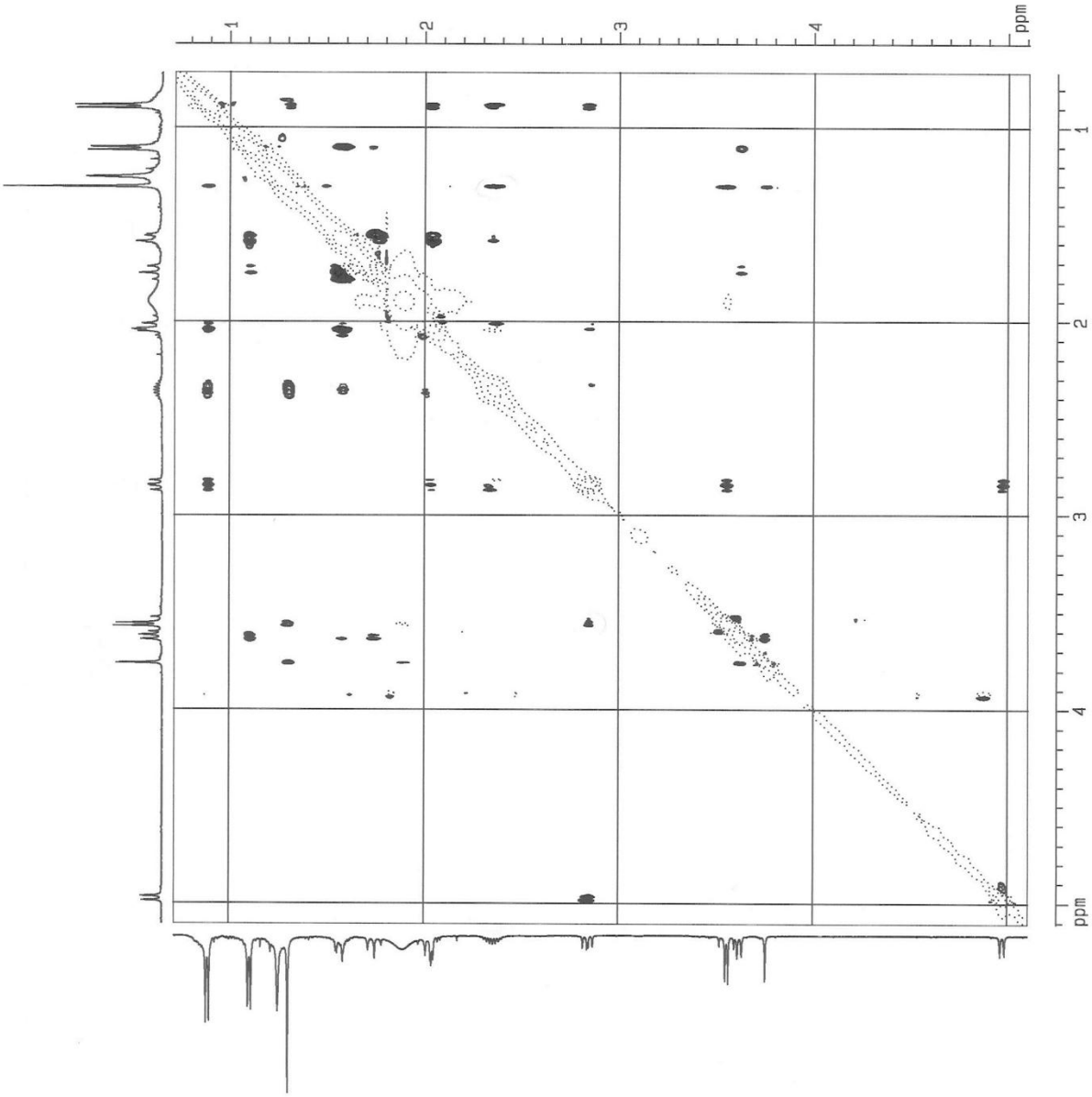
β-Dihydroagarofuran 26 – ^{13}C NMR (100 MHz)



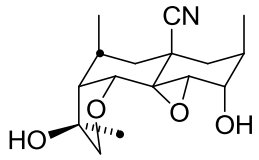
β-Dihydroagarofuran 26 – NOESY (400 MHz)

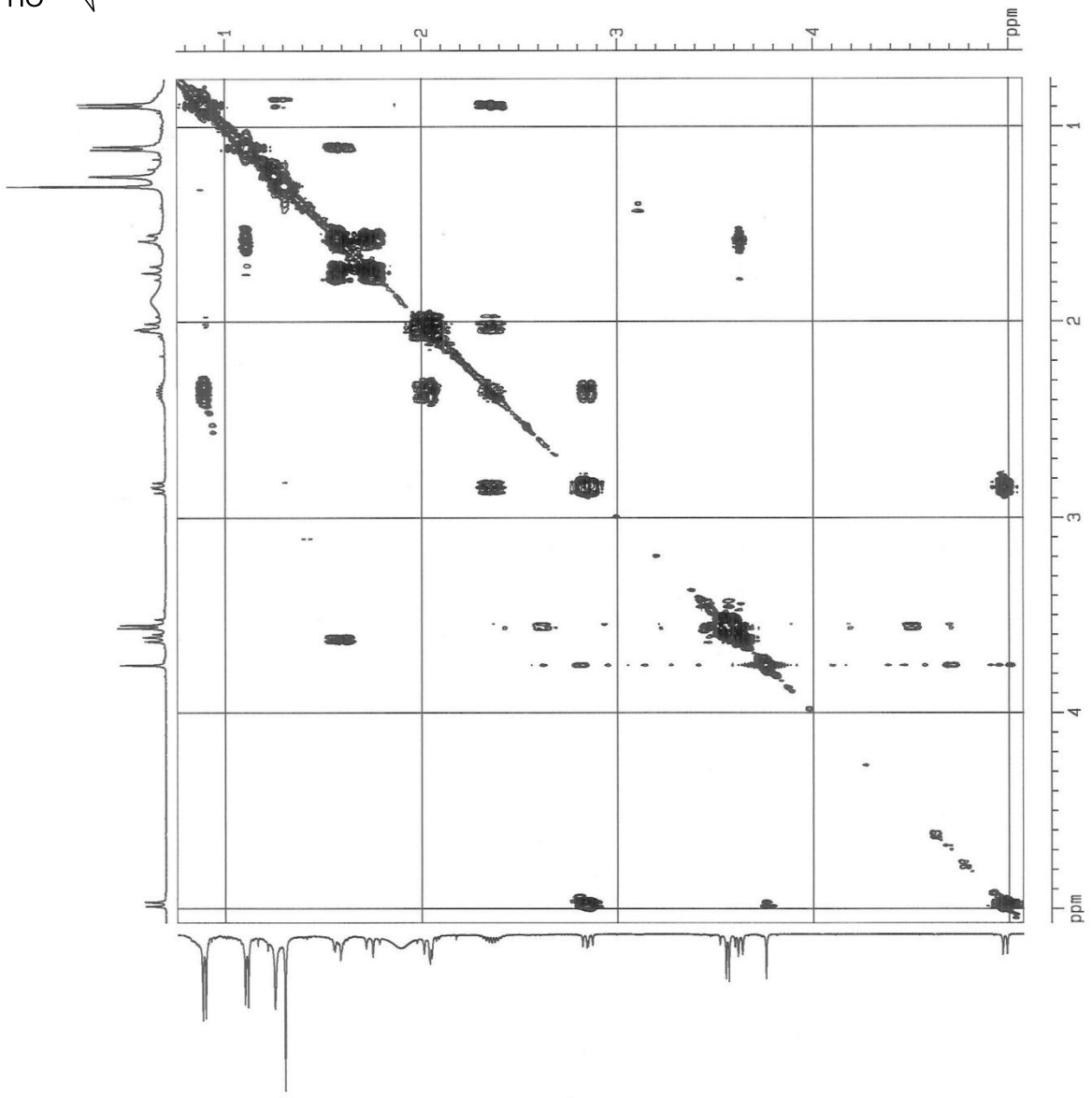


NOESY	8	12
8	-	✔
12	✔	-

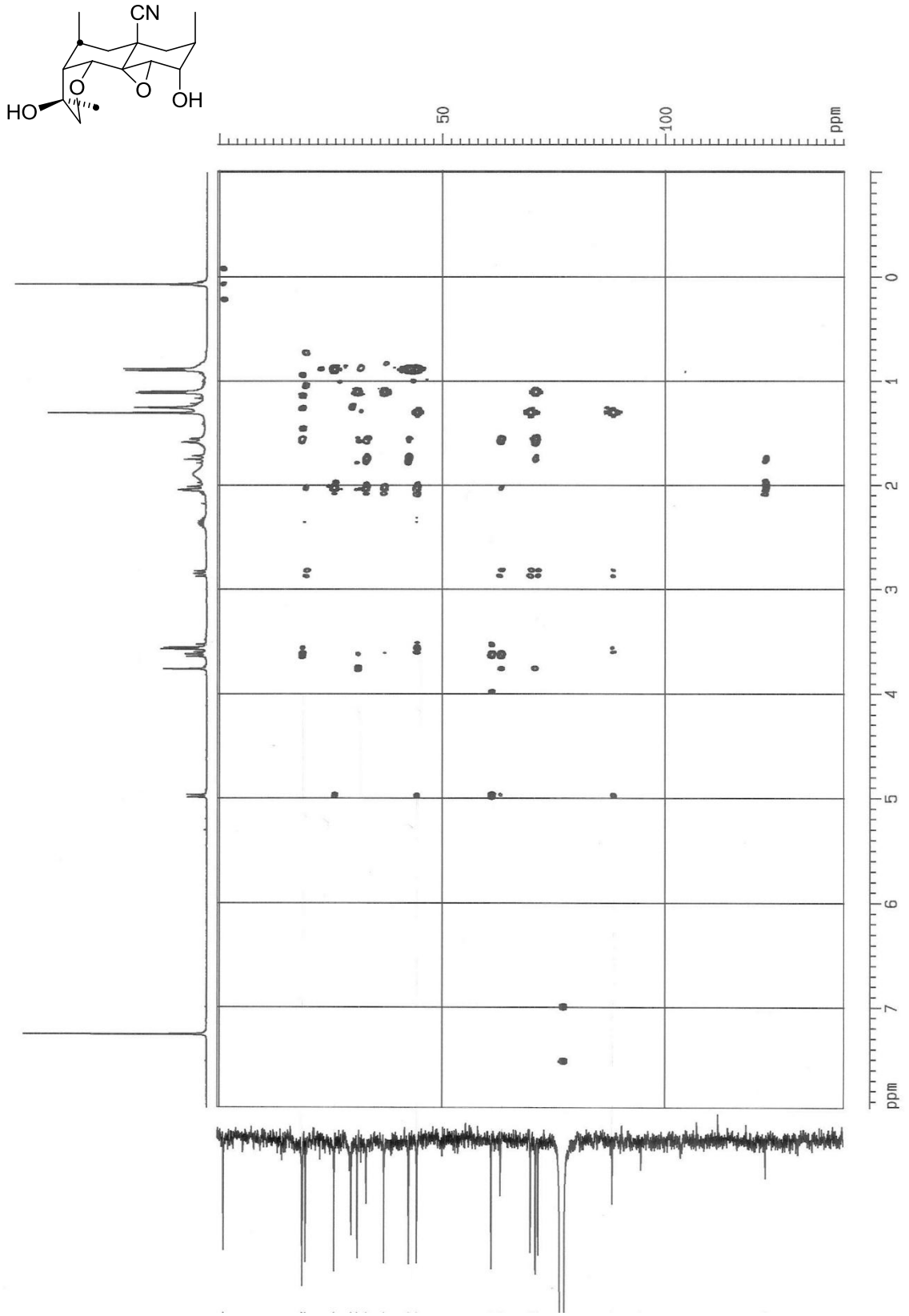


β-Dihydroagarofuran 26 – COSY (400 MHz)

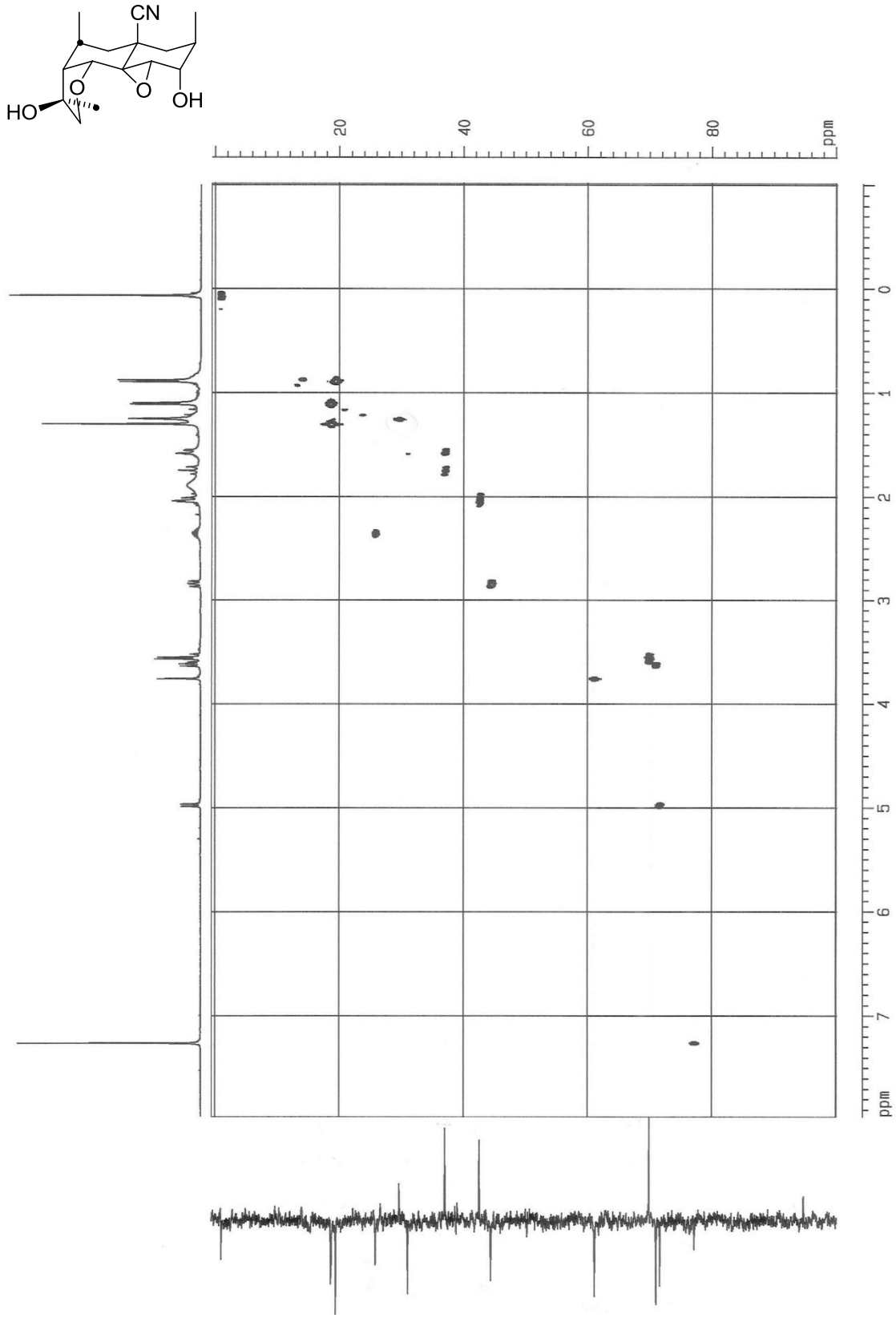




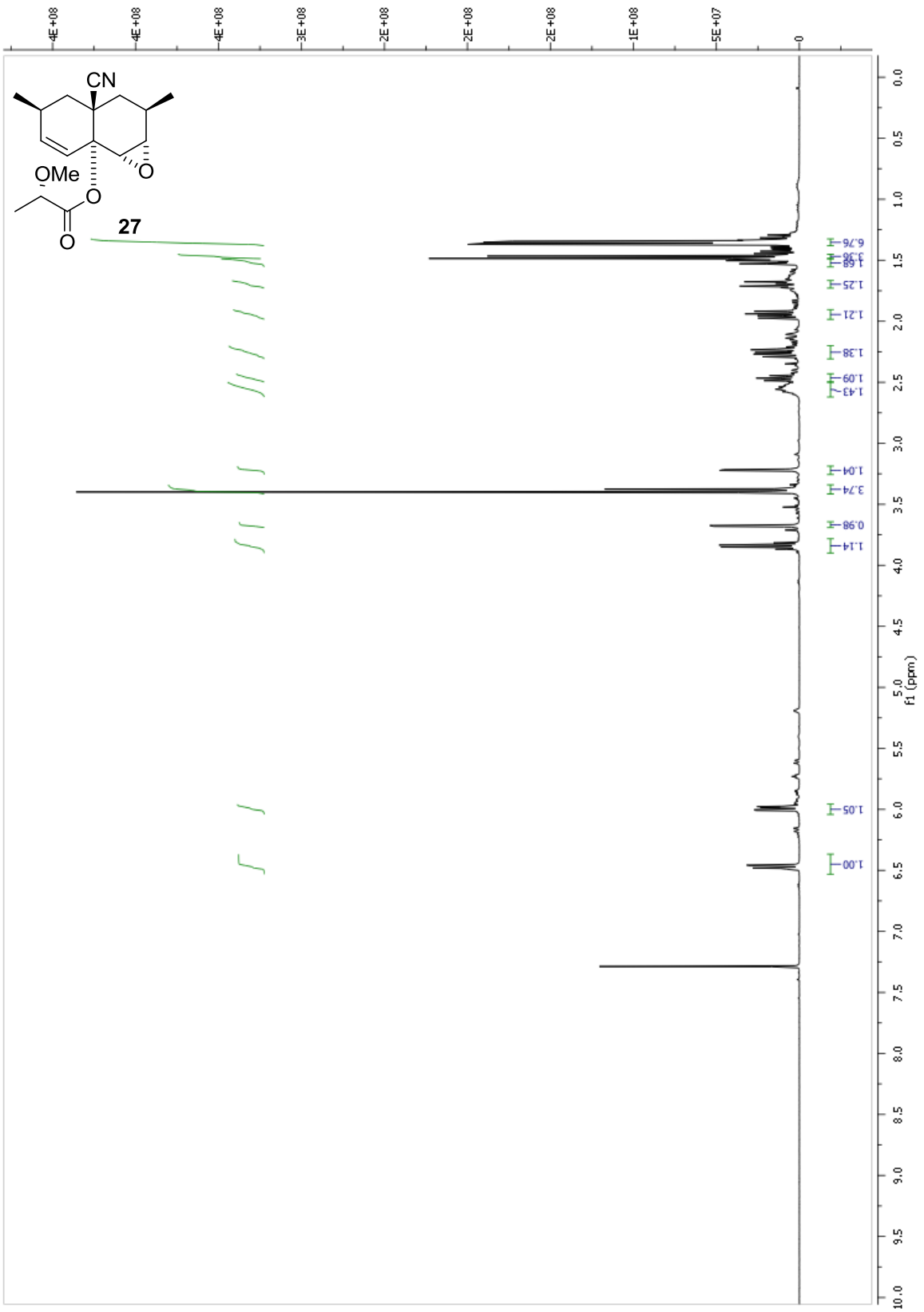
β-Dihydroagarofuran 26 – HMBC (400 MHz)



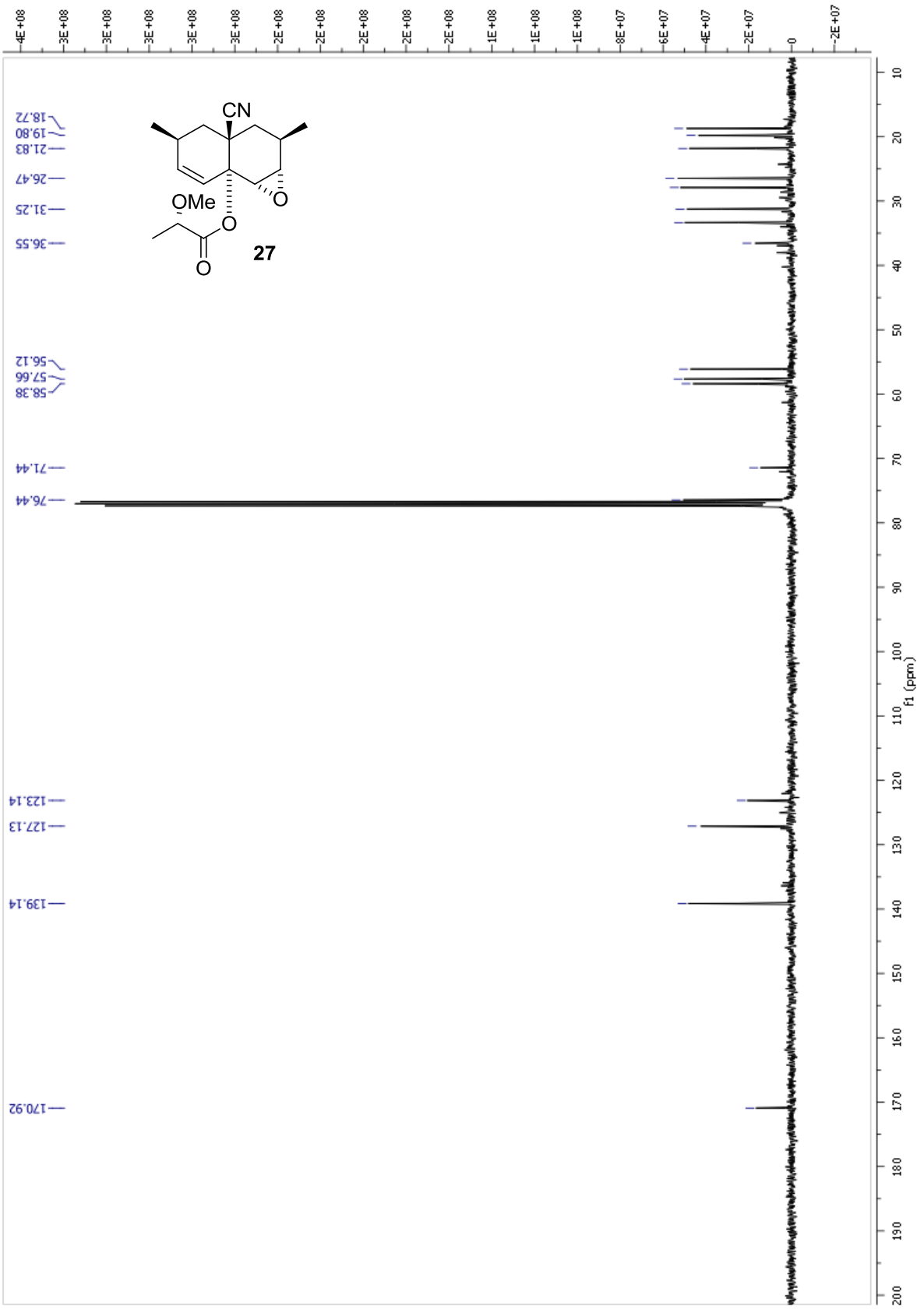
β-Dihydroagarofuran 26 – HMQC (400 MHz)



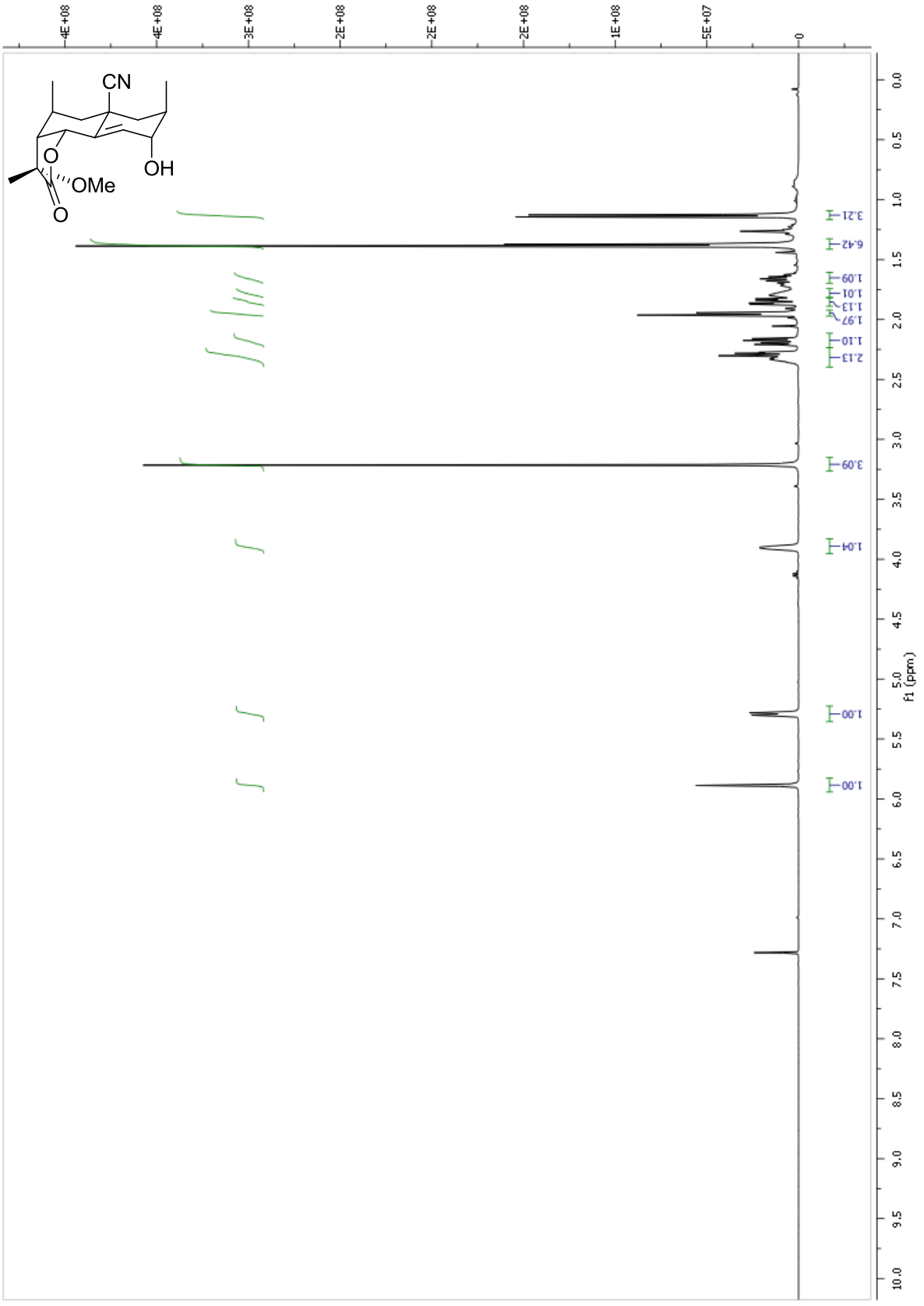
Lactate ester 27 – ^{1}H NMR (400 MHz)



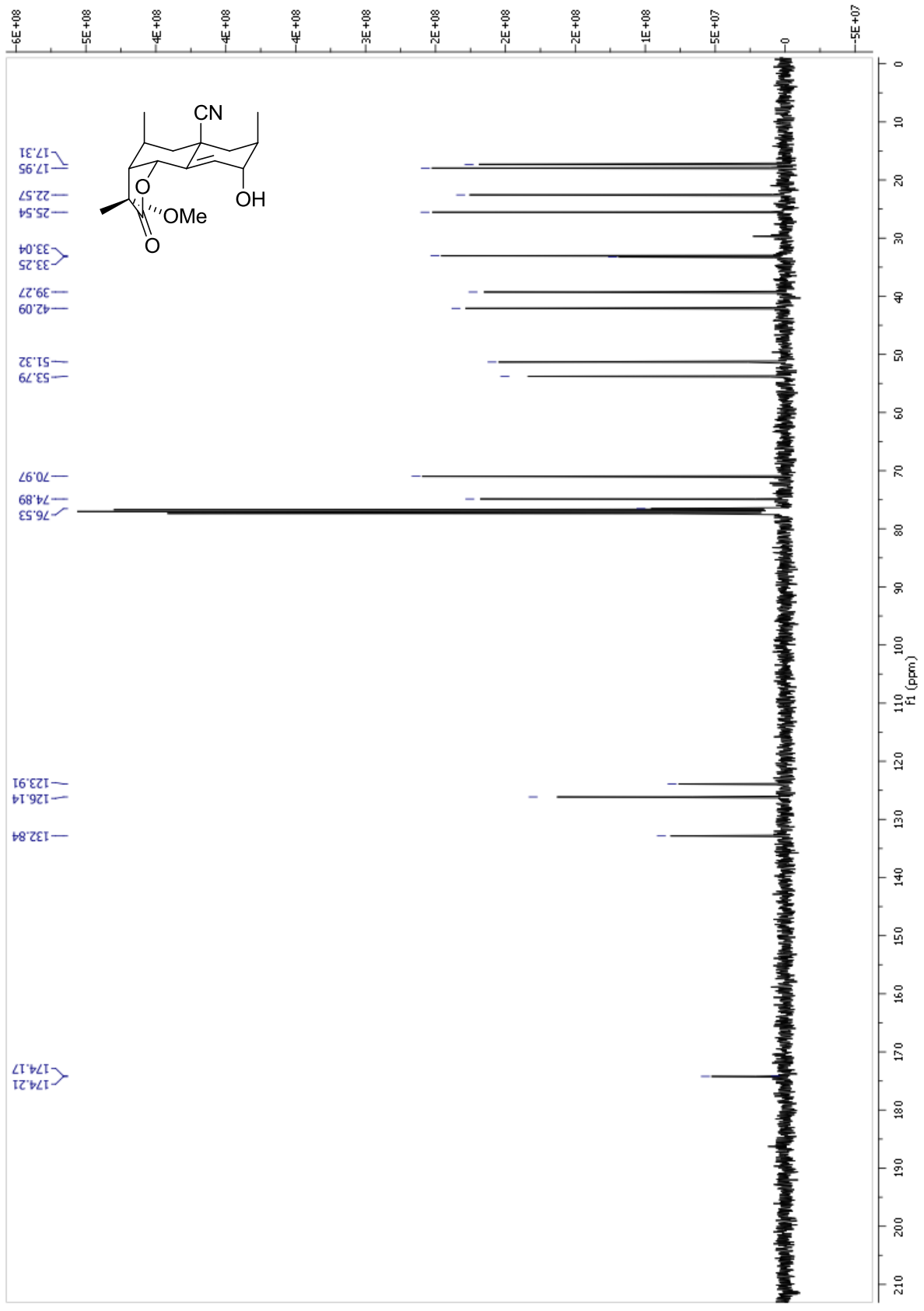
Lactate ester 27 – ^{13}C NMR (125 MHz)



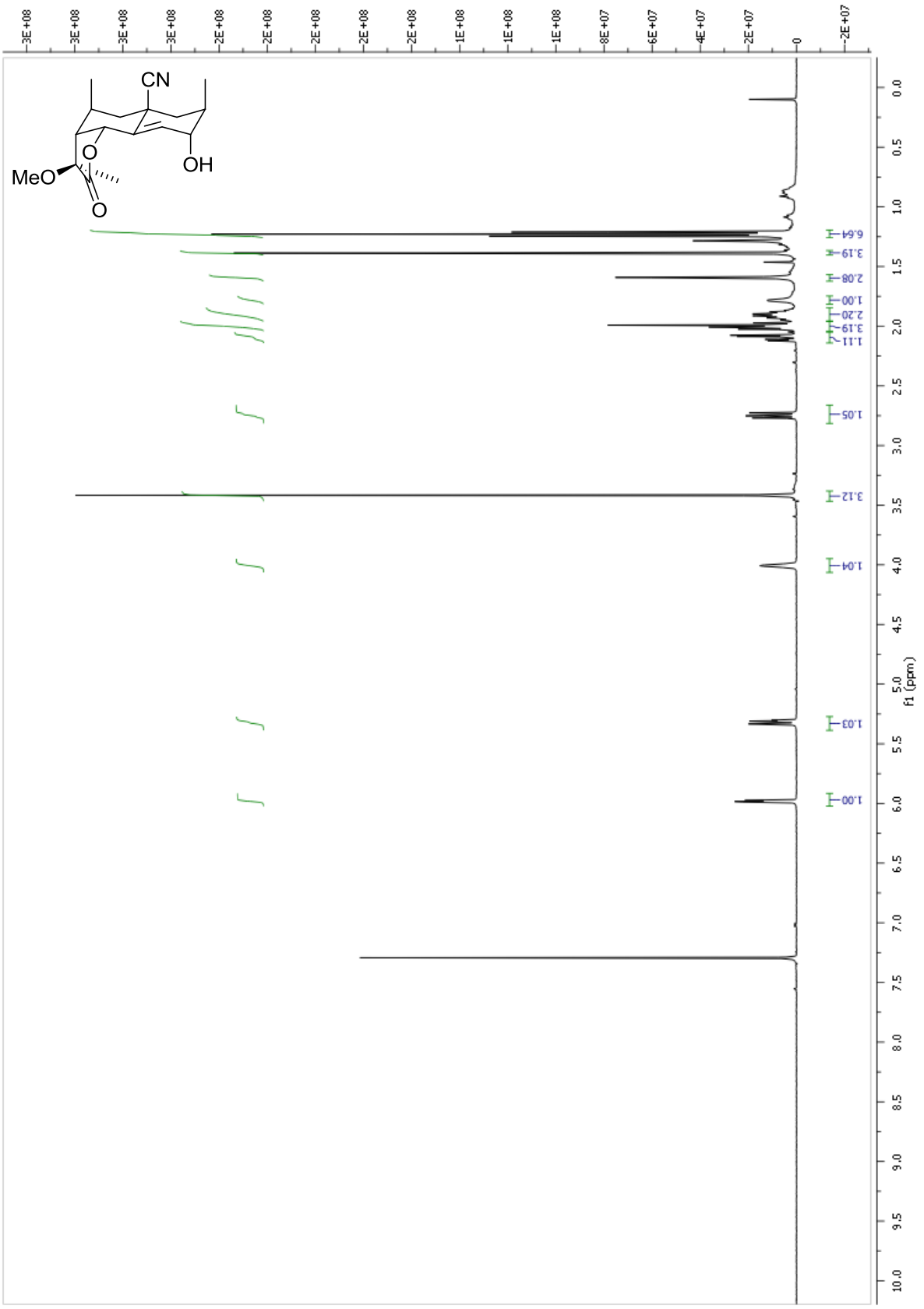
Lactone 28a – ^{1}H NMR (400 MHz)



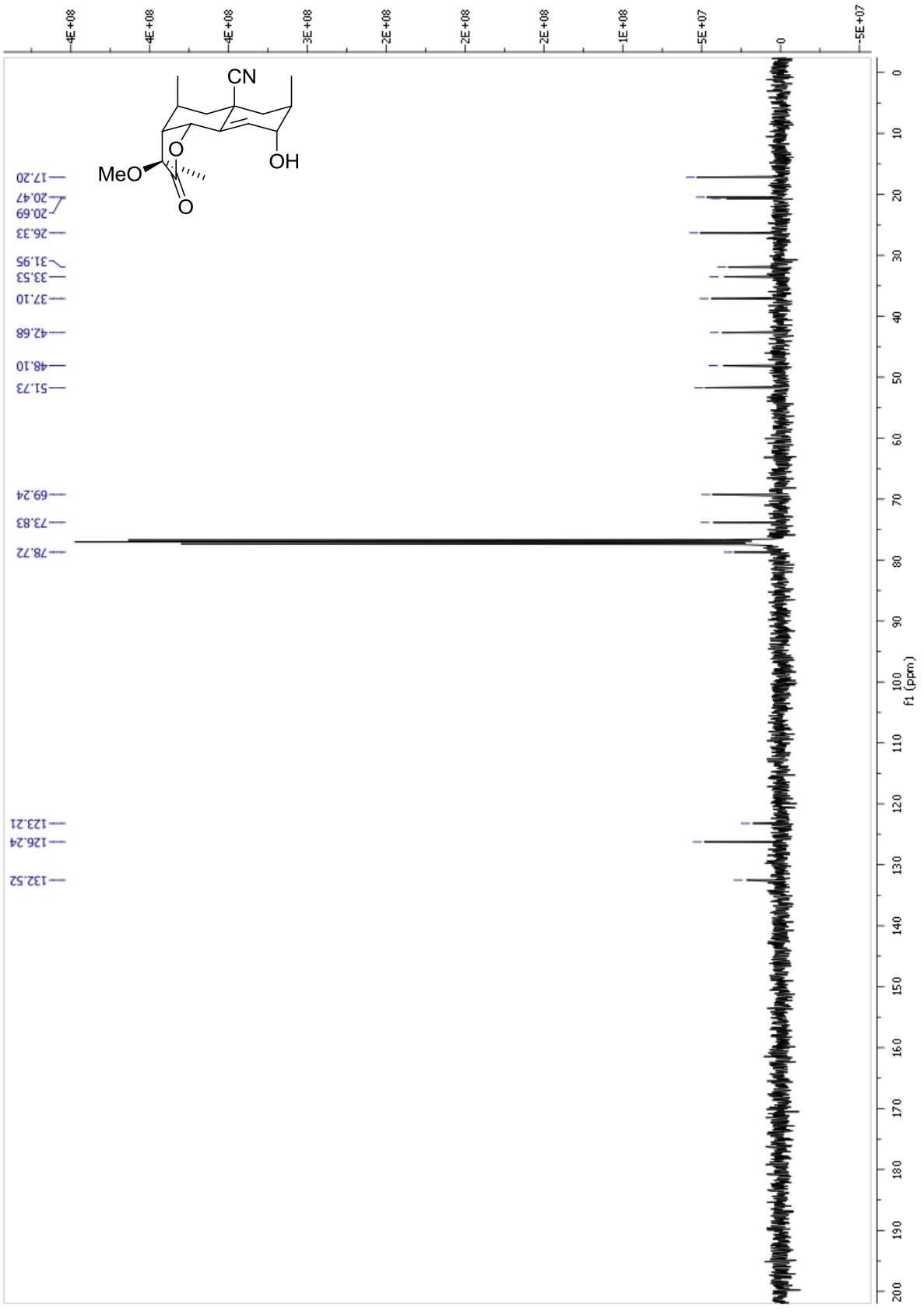
Lactone 28a – ^{13}C NMR (100 MHz)



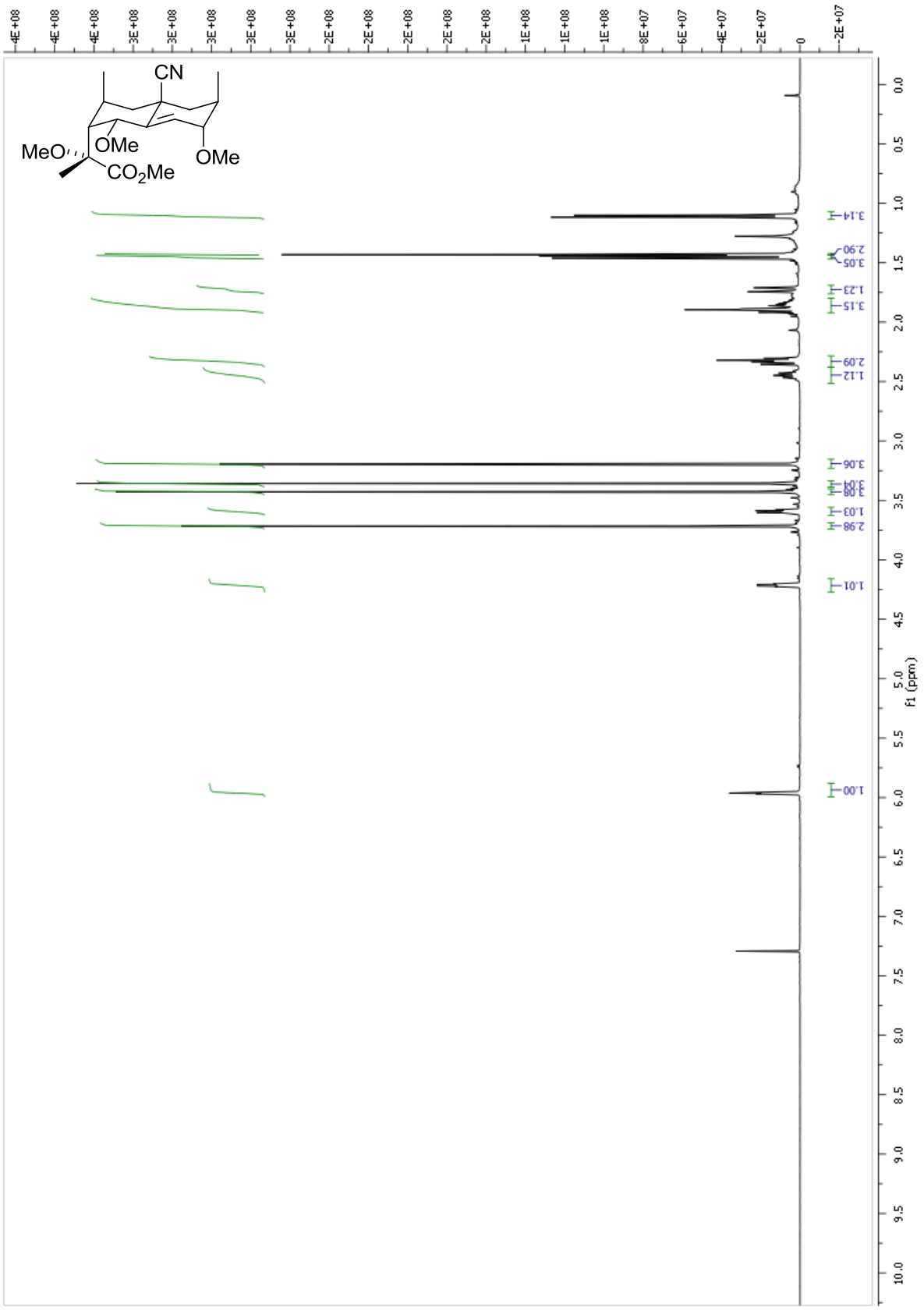
Lactone 28b – ^{1}H NMR (400 MHz)



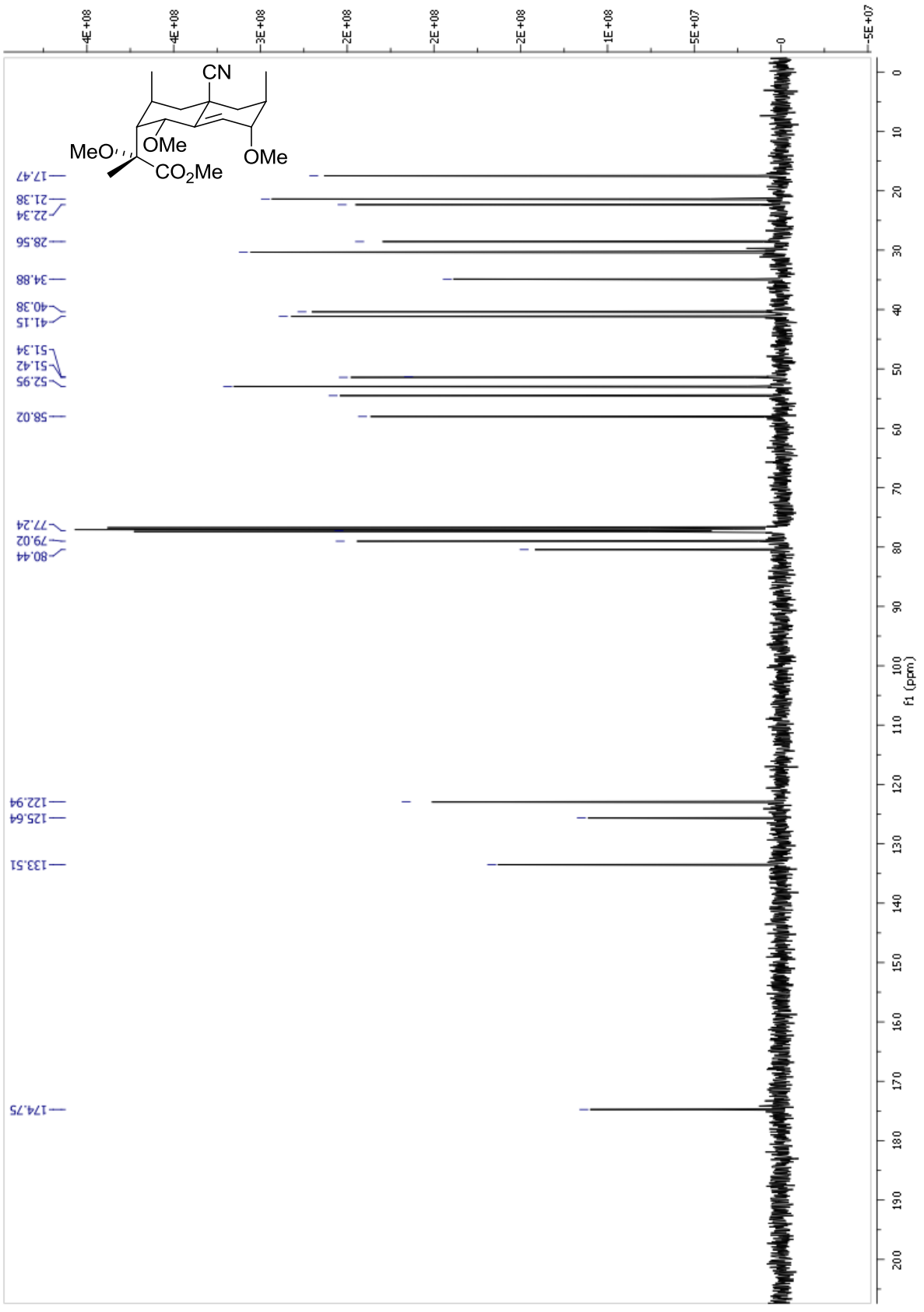
Lactone 28b – ^{13}C NMR (100 MHz)



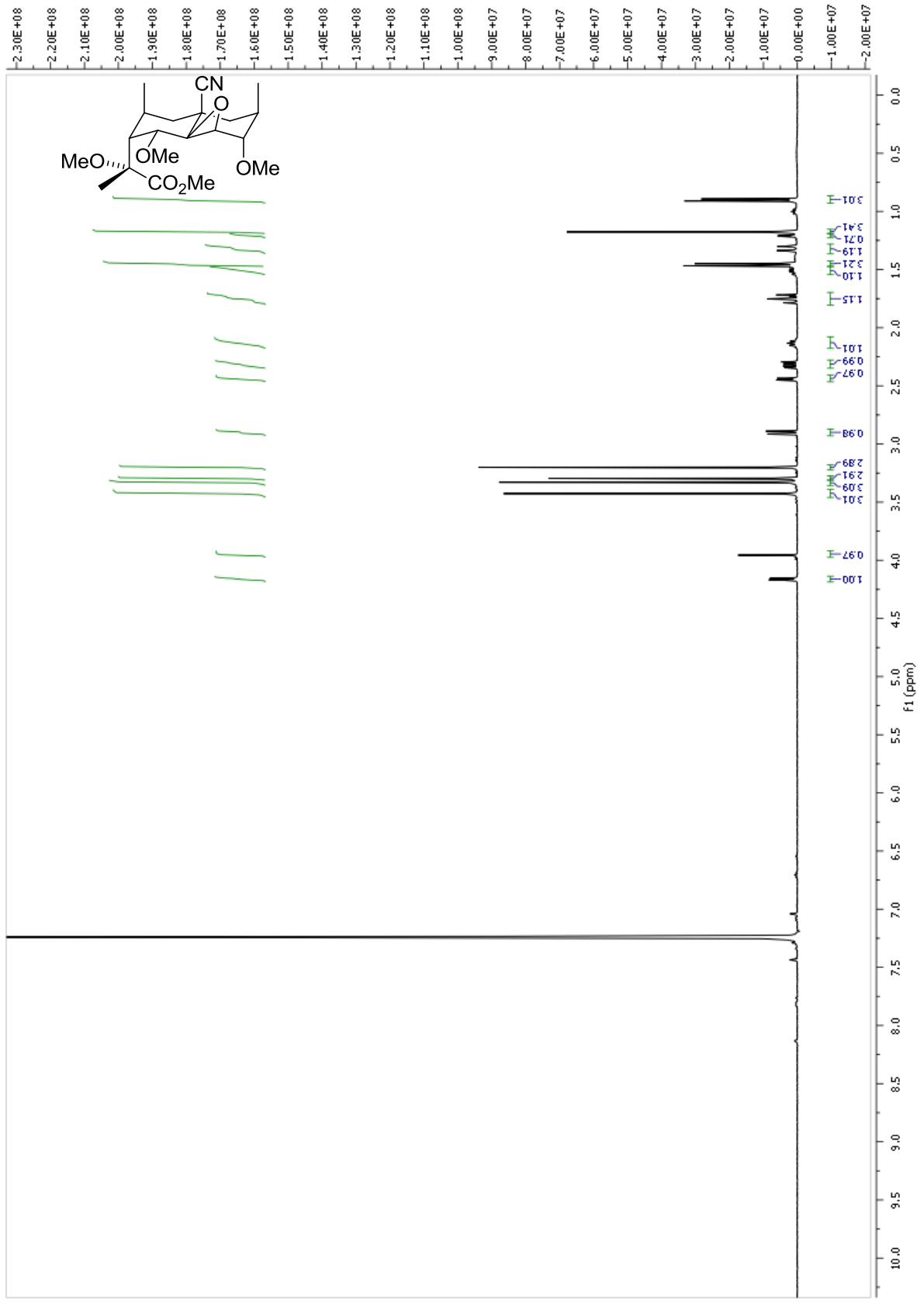
Methyl ester 29 – ^{1}H NMR (400 MHz)



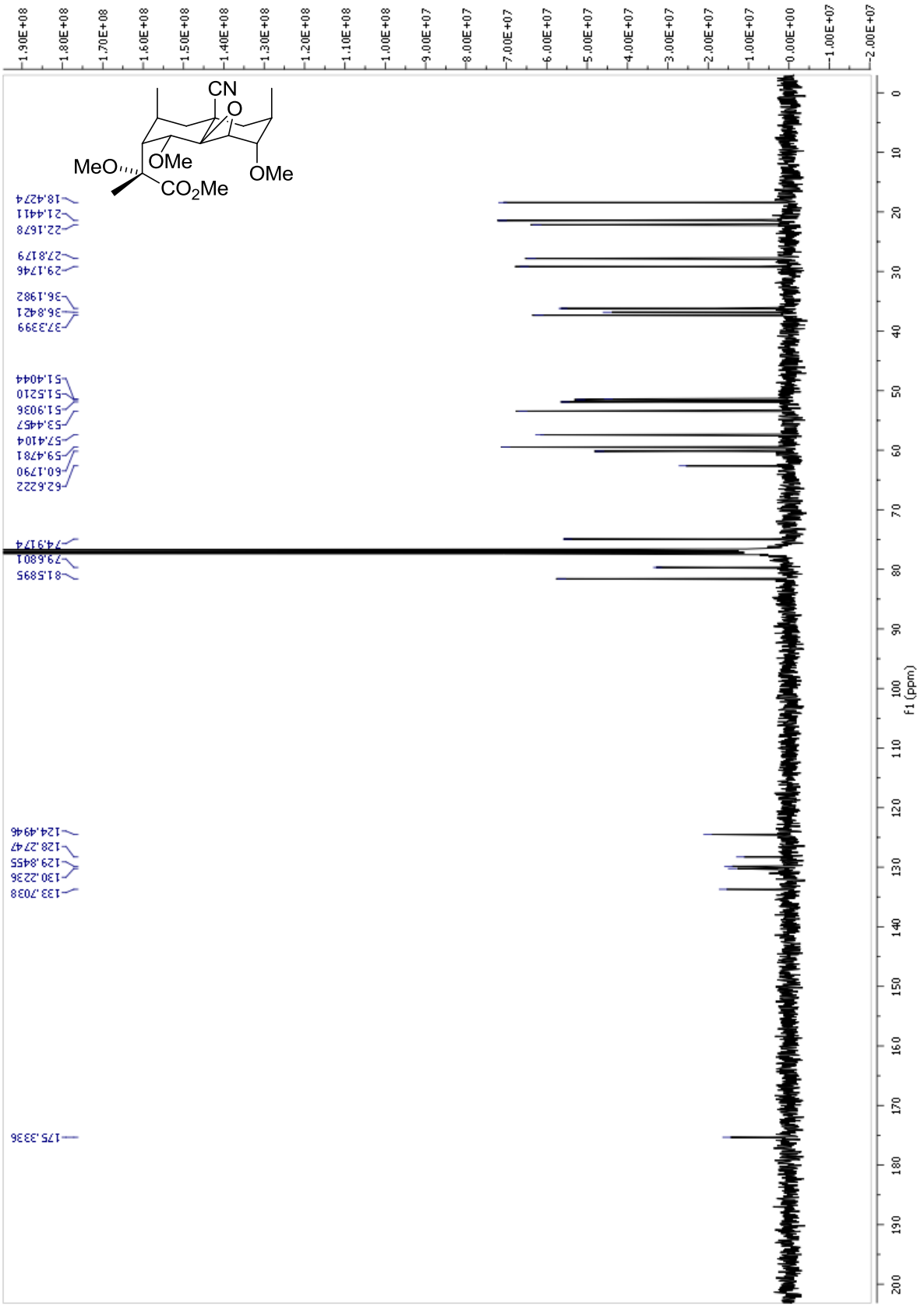
Methyl ester 29 – ^{13}C NMR (100 MHz)



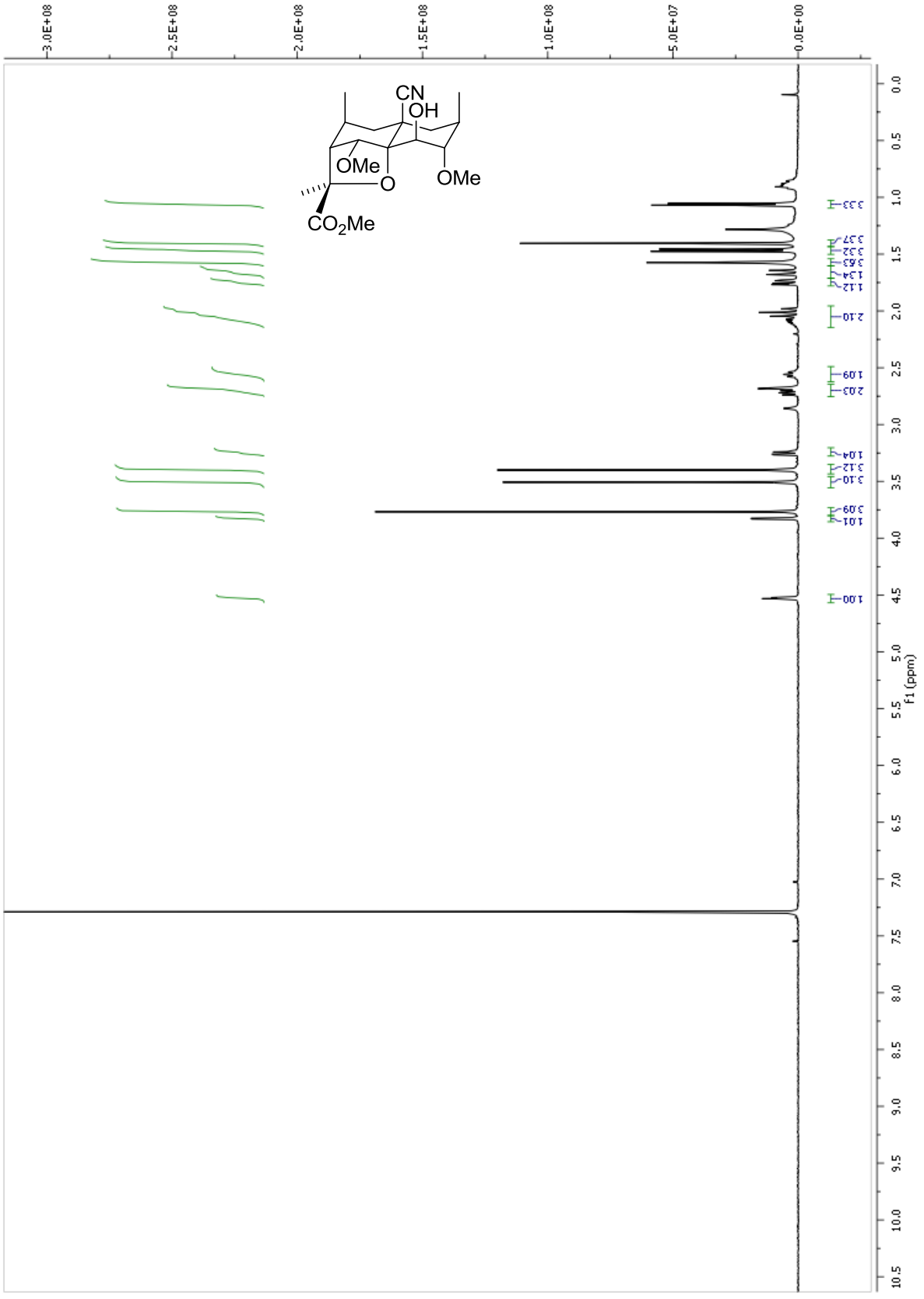
Epoxide 30 – ^{1}H NMR (400 MHz)



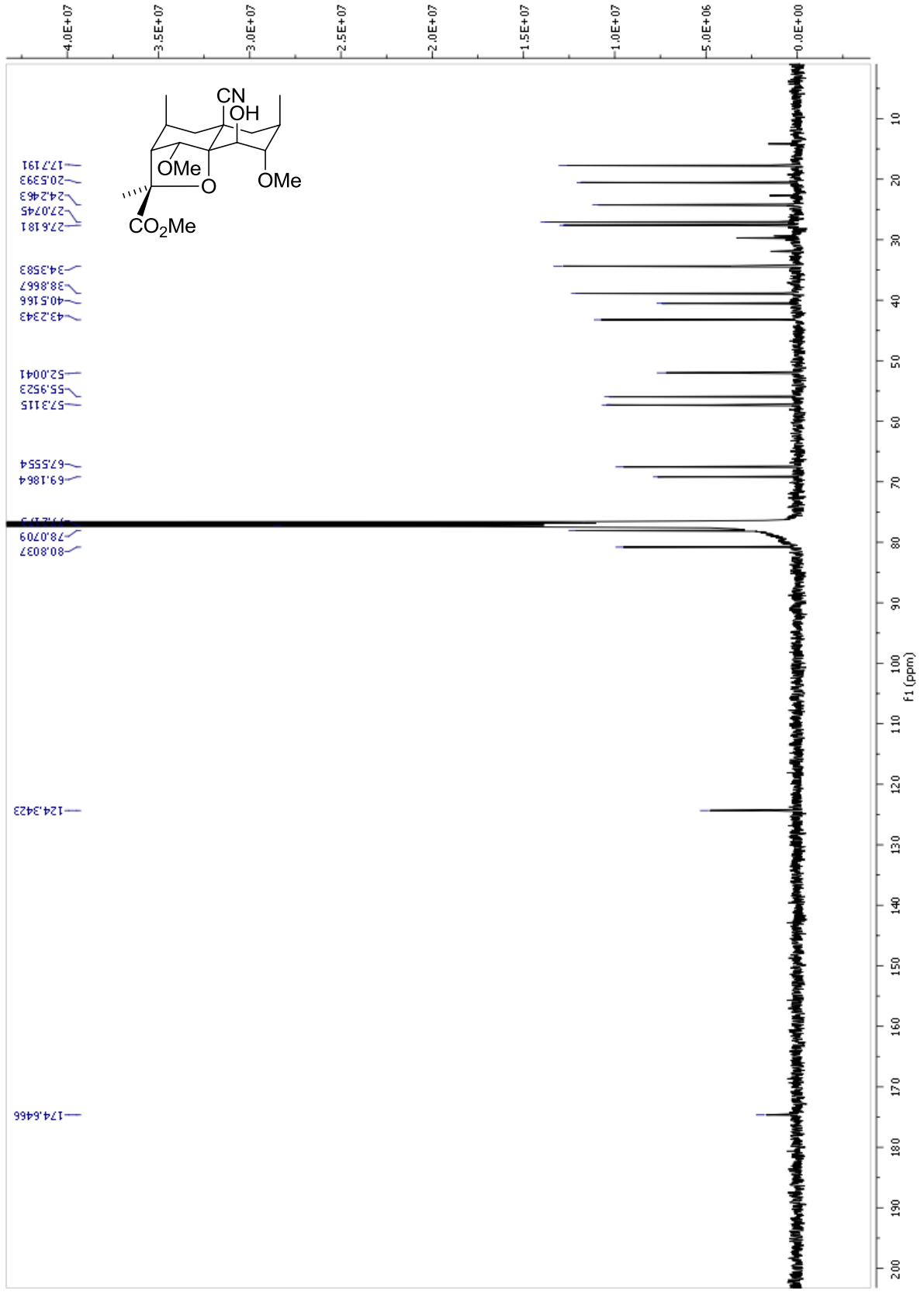
Epoxide 30 – ^{13}C NMR (100 MHz)



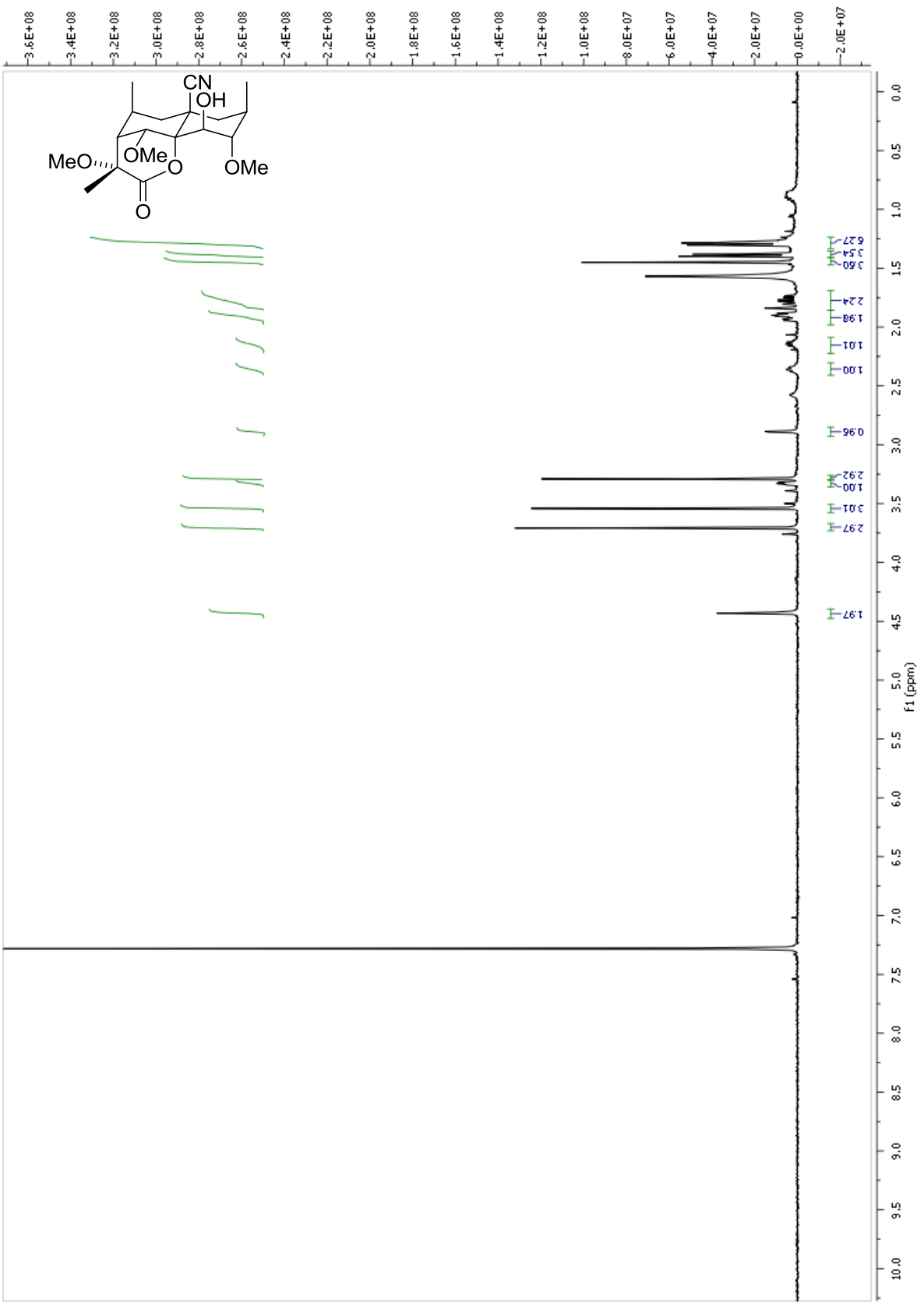
β-Dihydroagarofuran 31 – ^{1}H NMR (400 MHz)



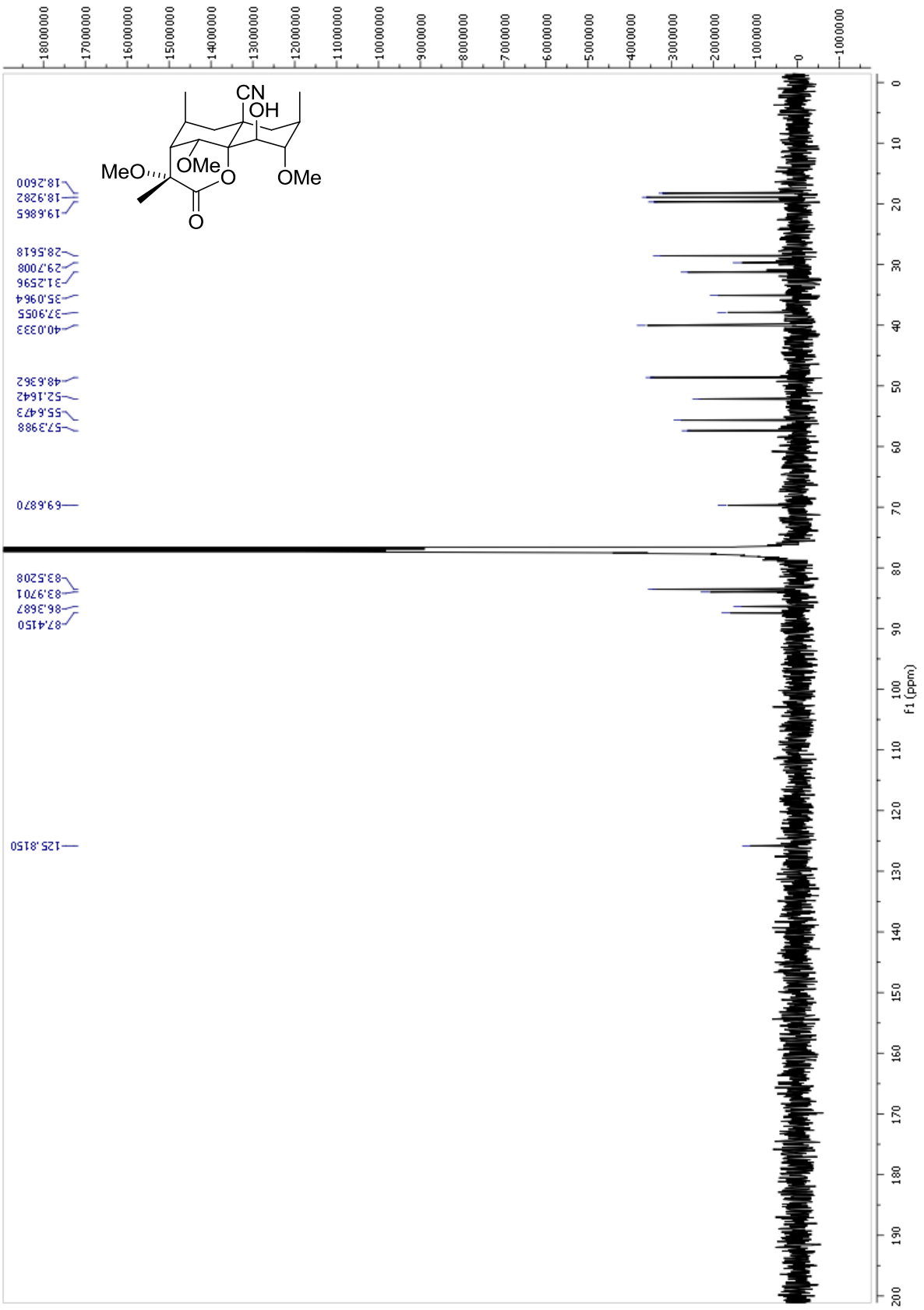
β-Dihydroagarofuran 31 – ^{13}C NMR (100 MHz)



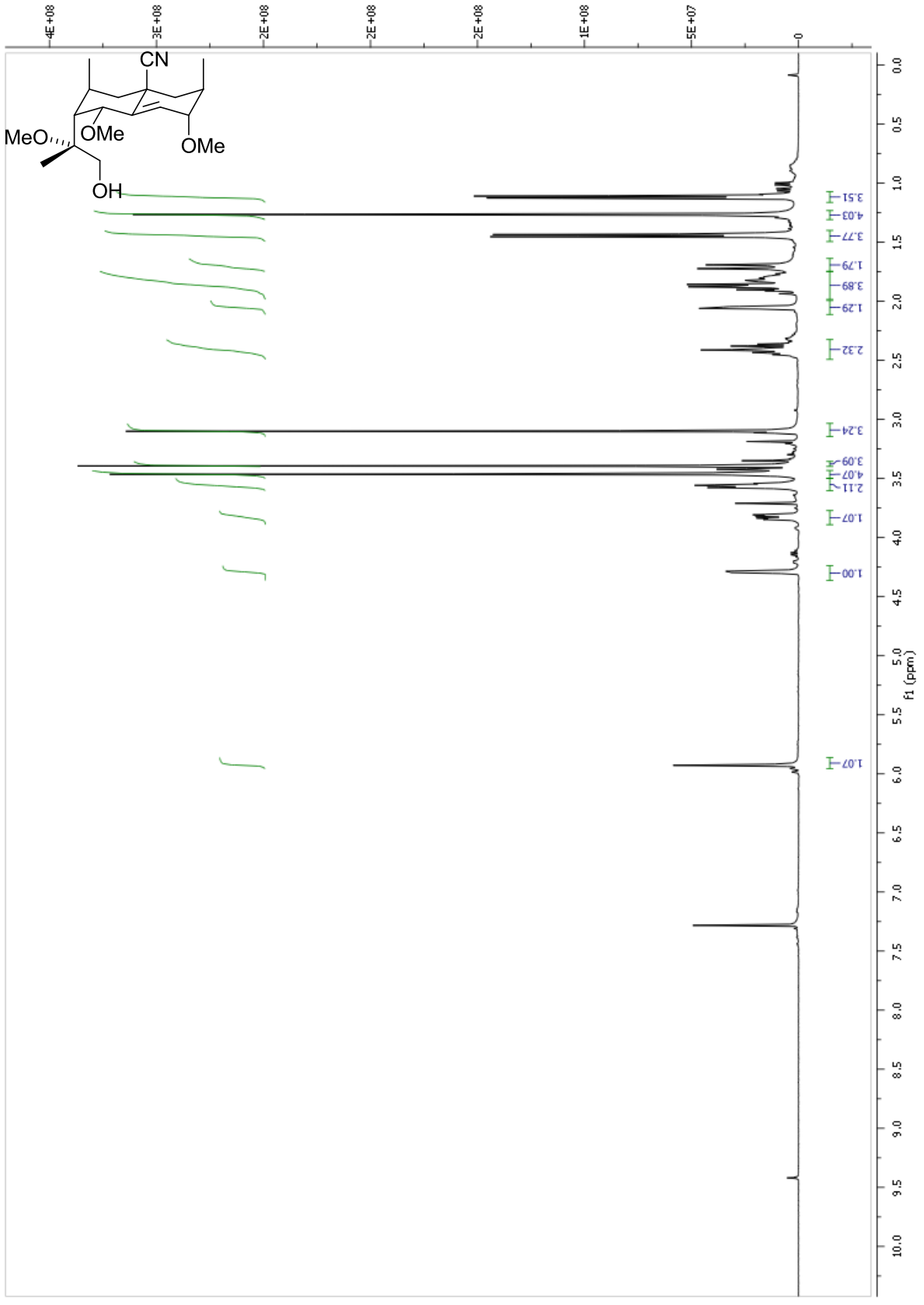
δ-Lactone 32 – ^{1}H NMR (400 MHz)



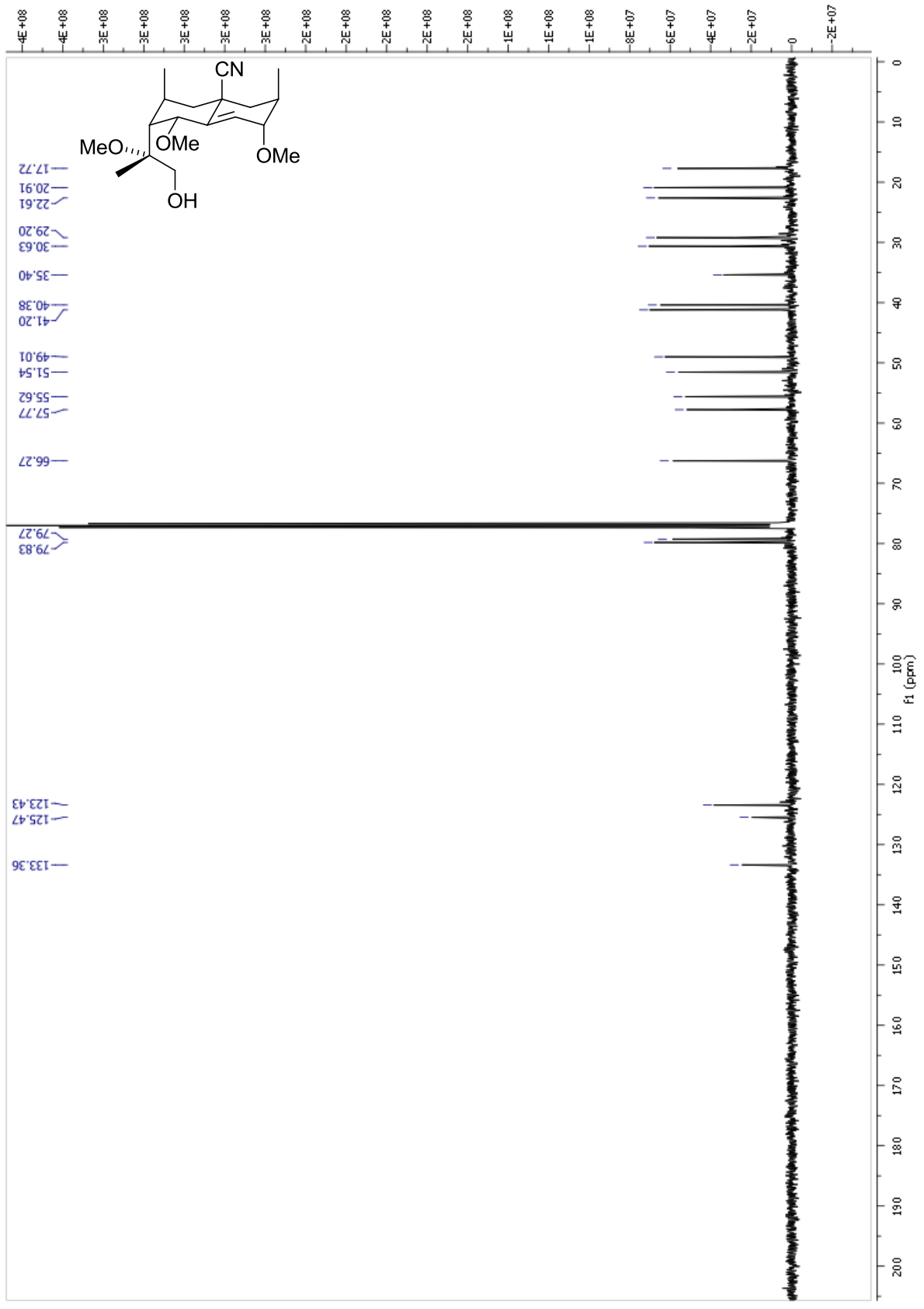
δ-Lactone 32 – ^{13}C NMR (100 MHz)



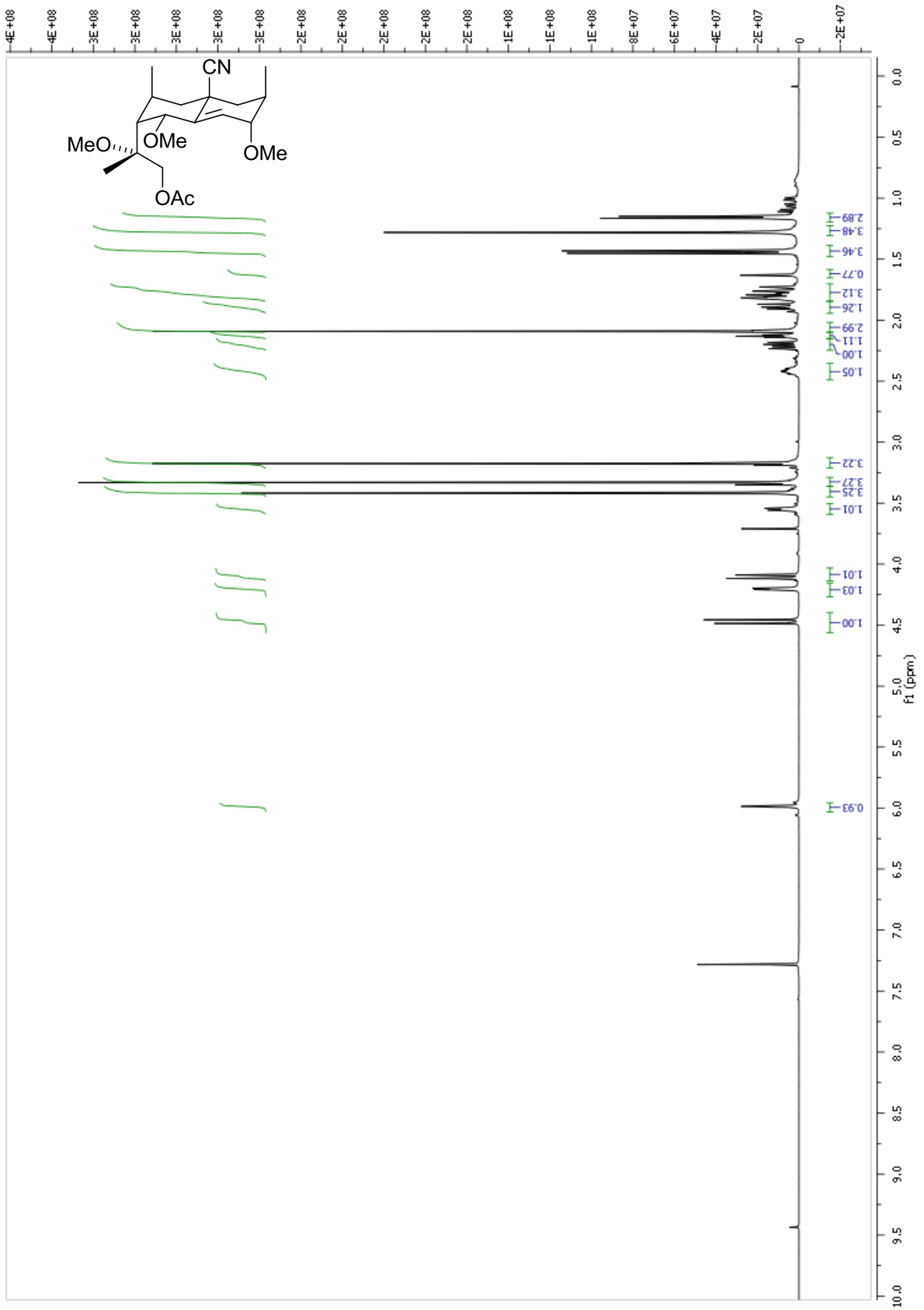
Alcohol 33 – ^{1}H NMR (400 MHz)



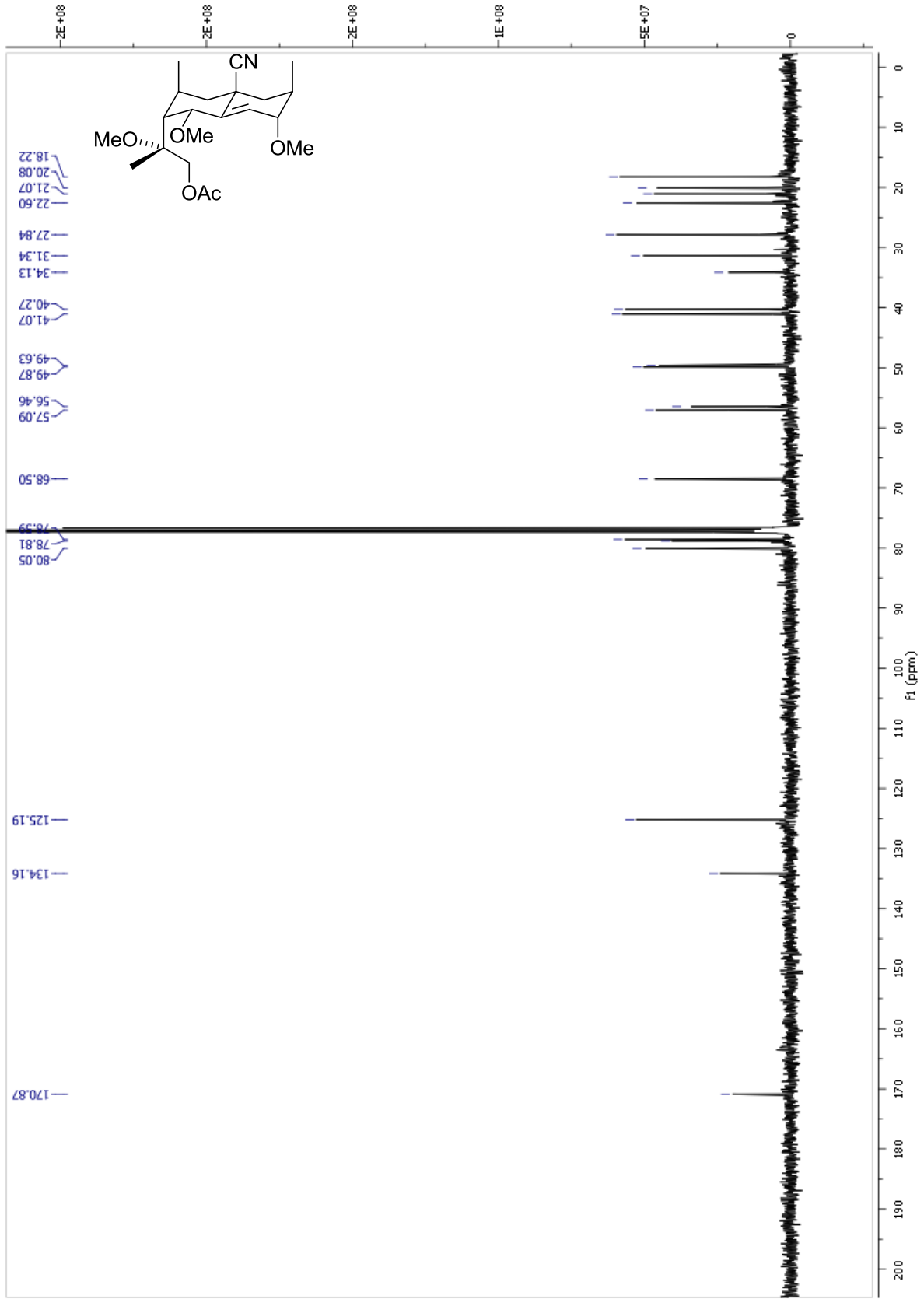
Alcohol 33 – ^{13}C NMR (100 MHz)



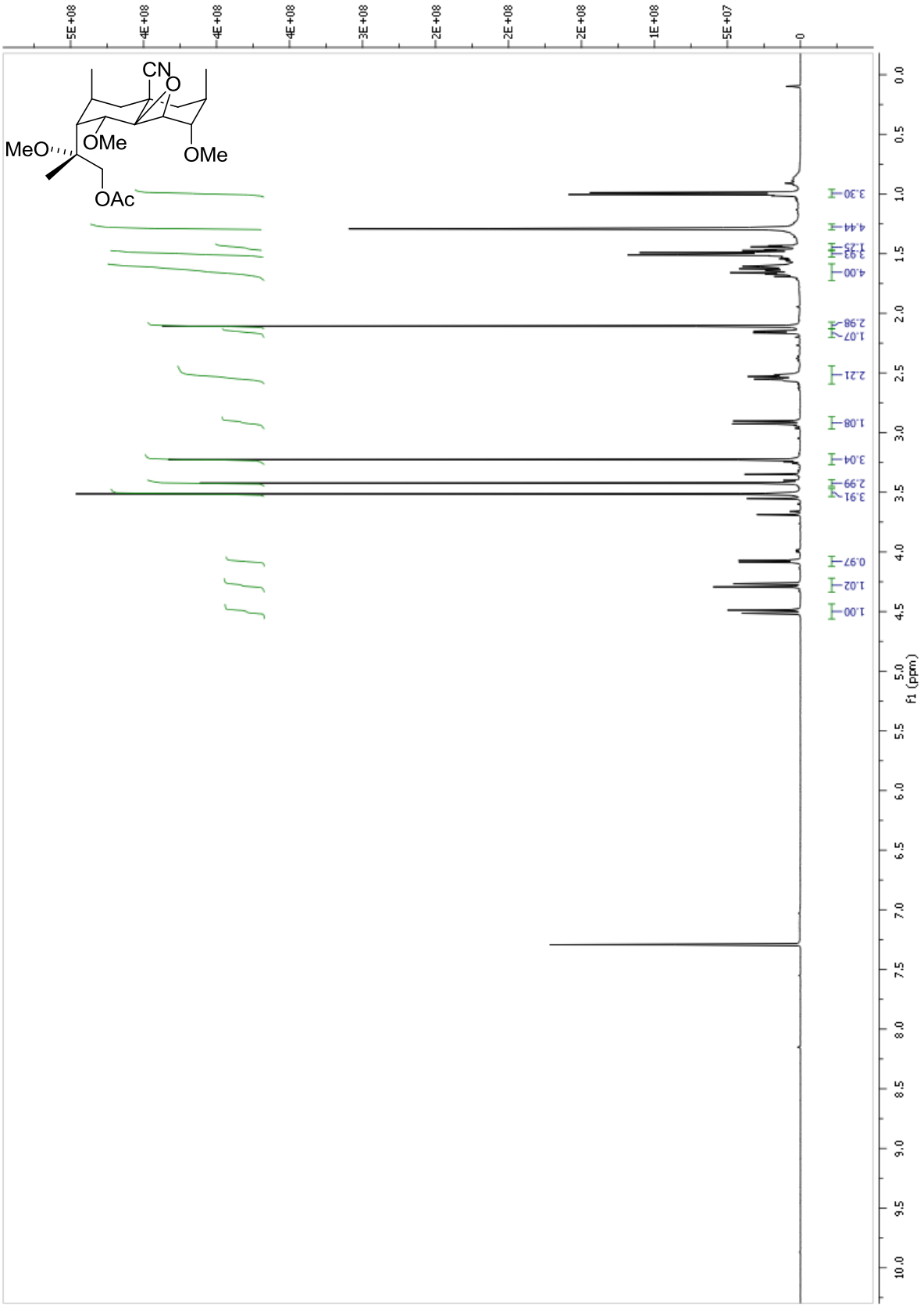
Acetate 34 – ^{1}H NMR (400 MHz)



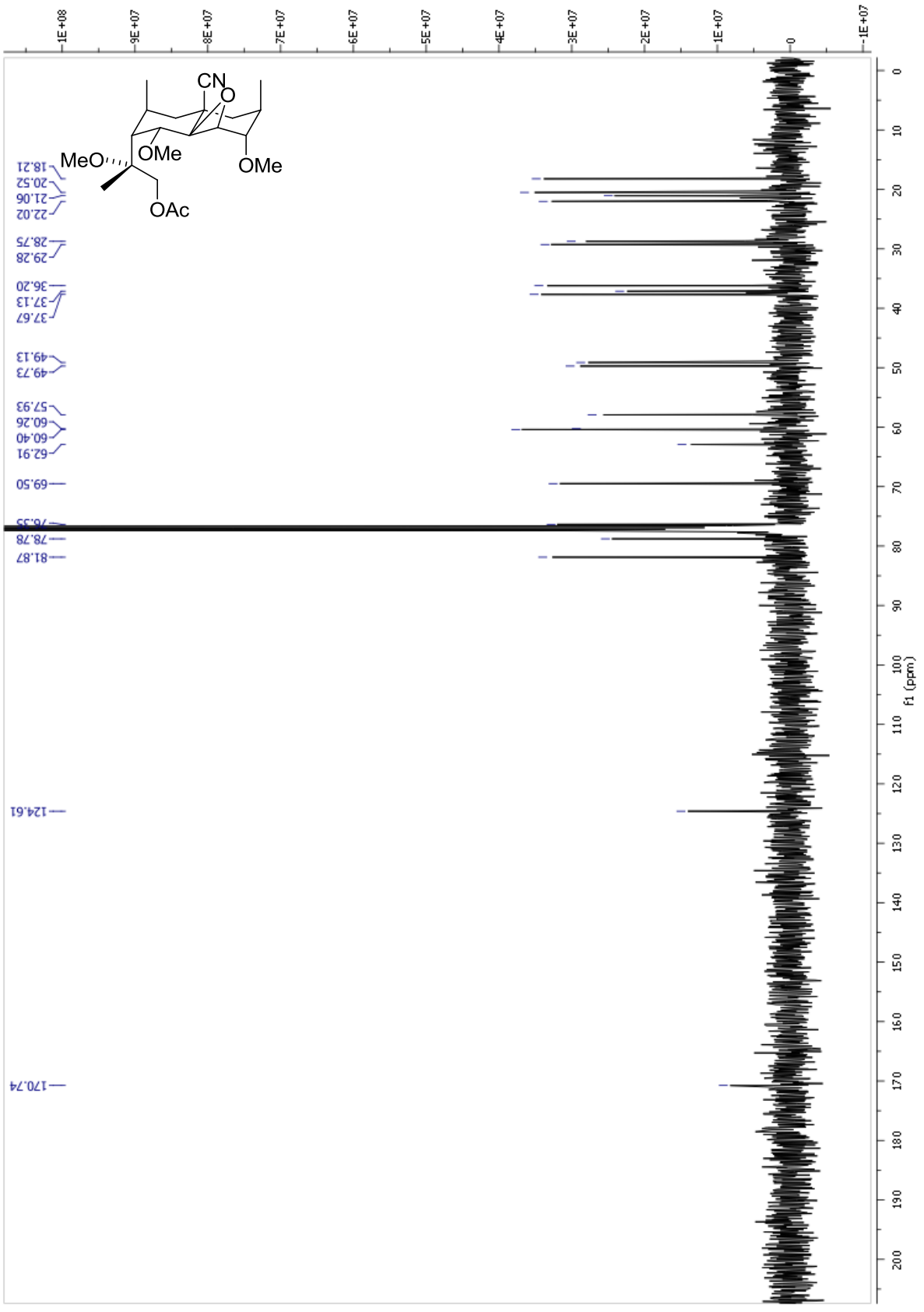
Acetate 34 – ^{13}C NMR (100 MHz)



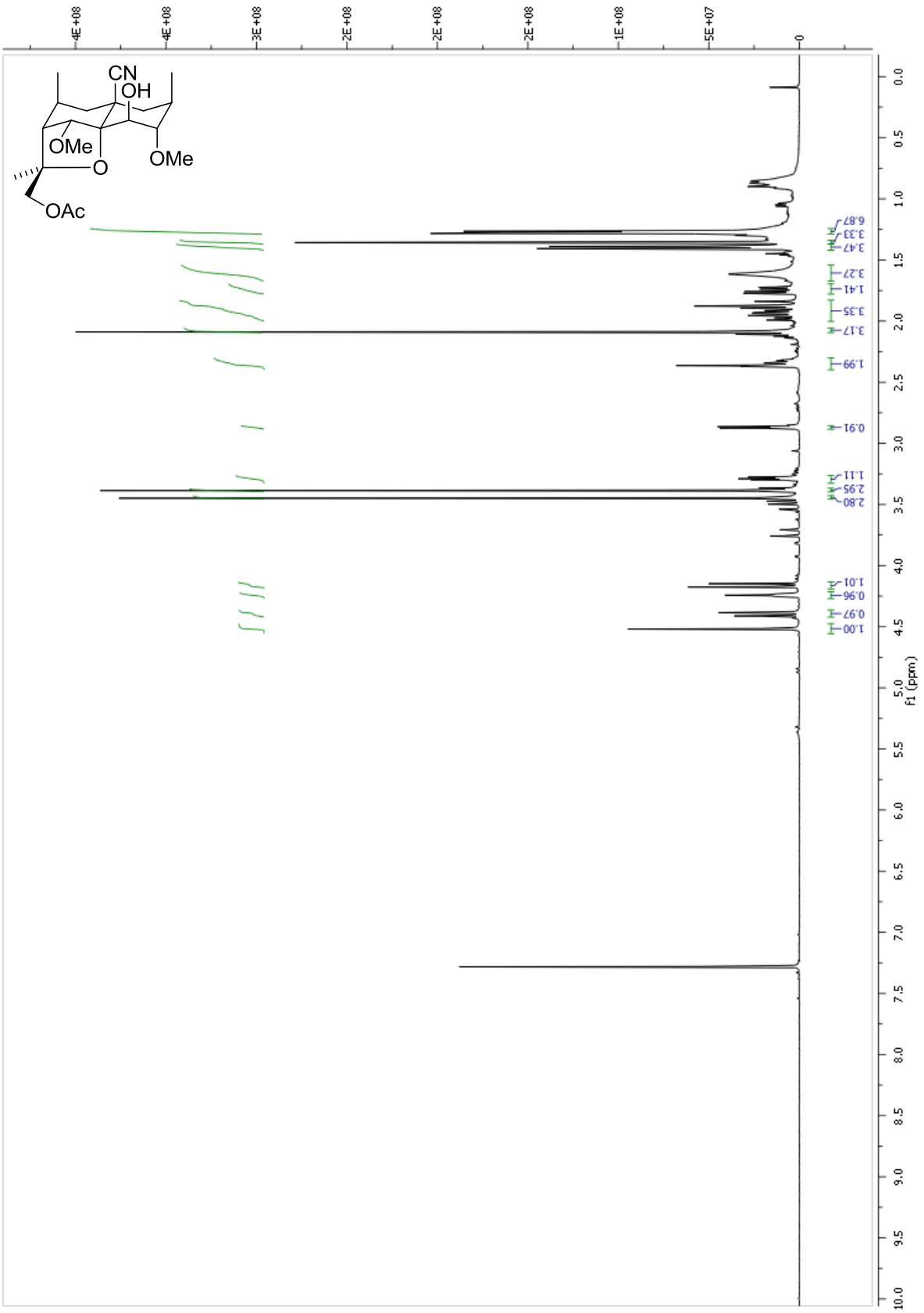
Epoxide 35 – ^{1}H NMR (400 MHz)



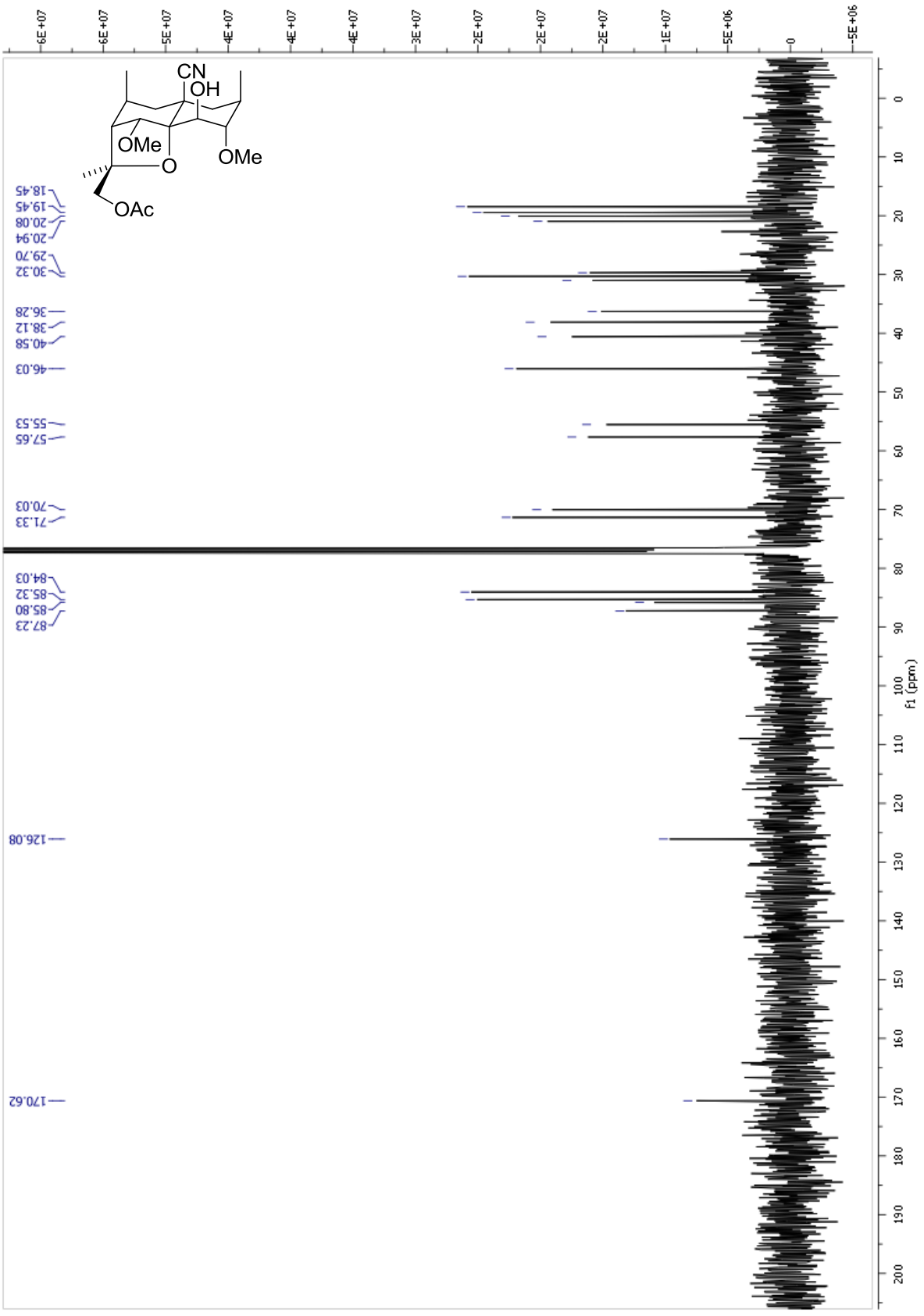
Epoxide 35 – ^{13}C NMR (100 MHz)



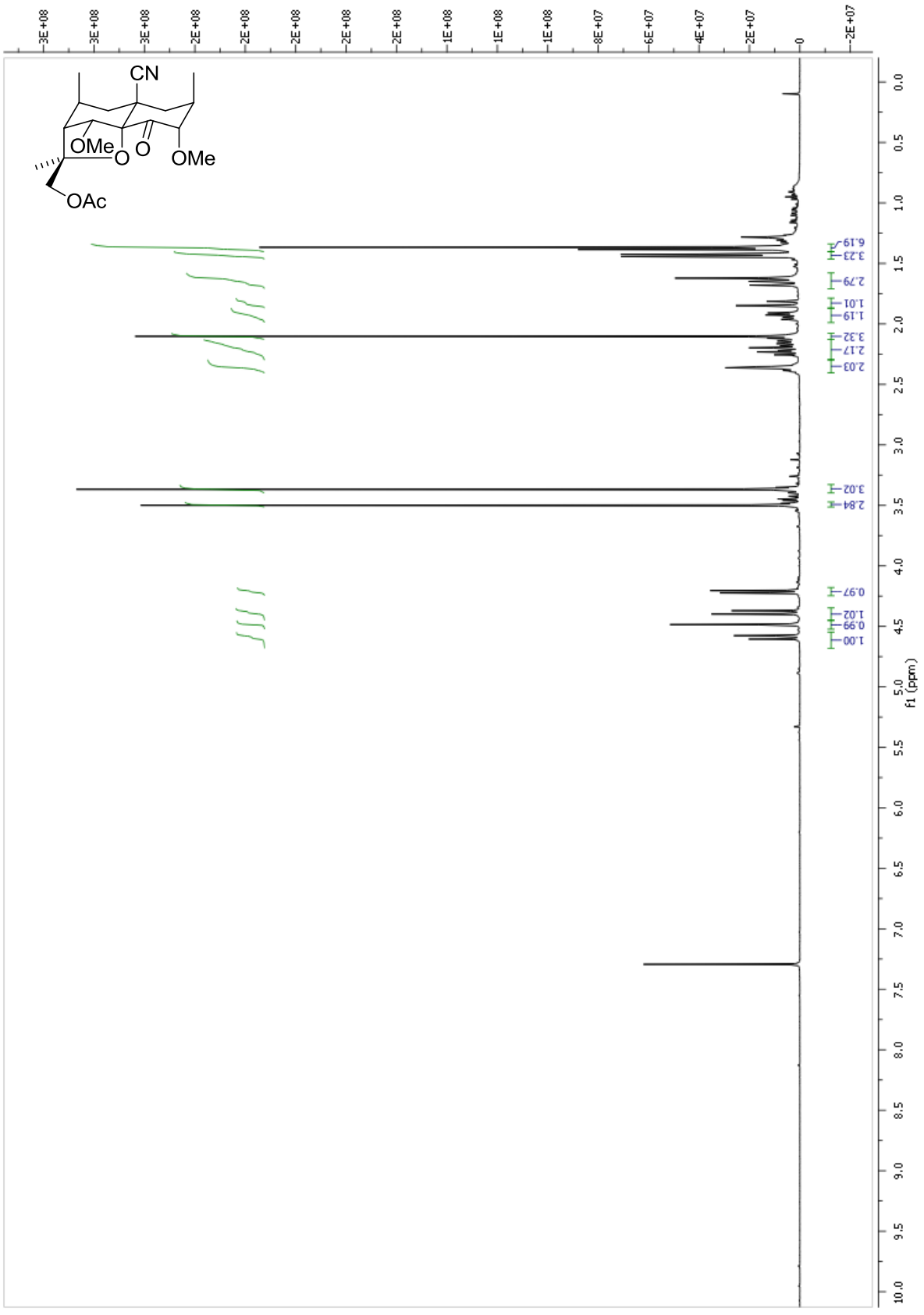
β-Dihydroagarofuran 36 – ^{1}H NMR (400 MHz)



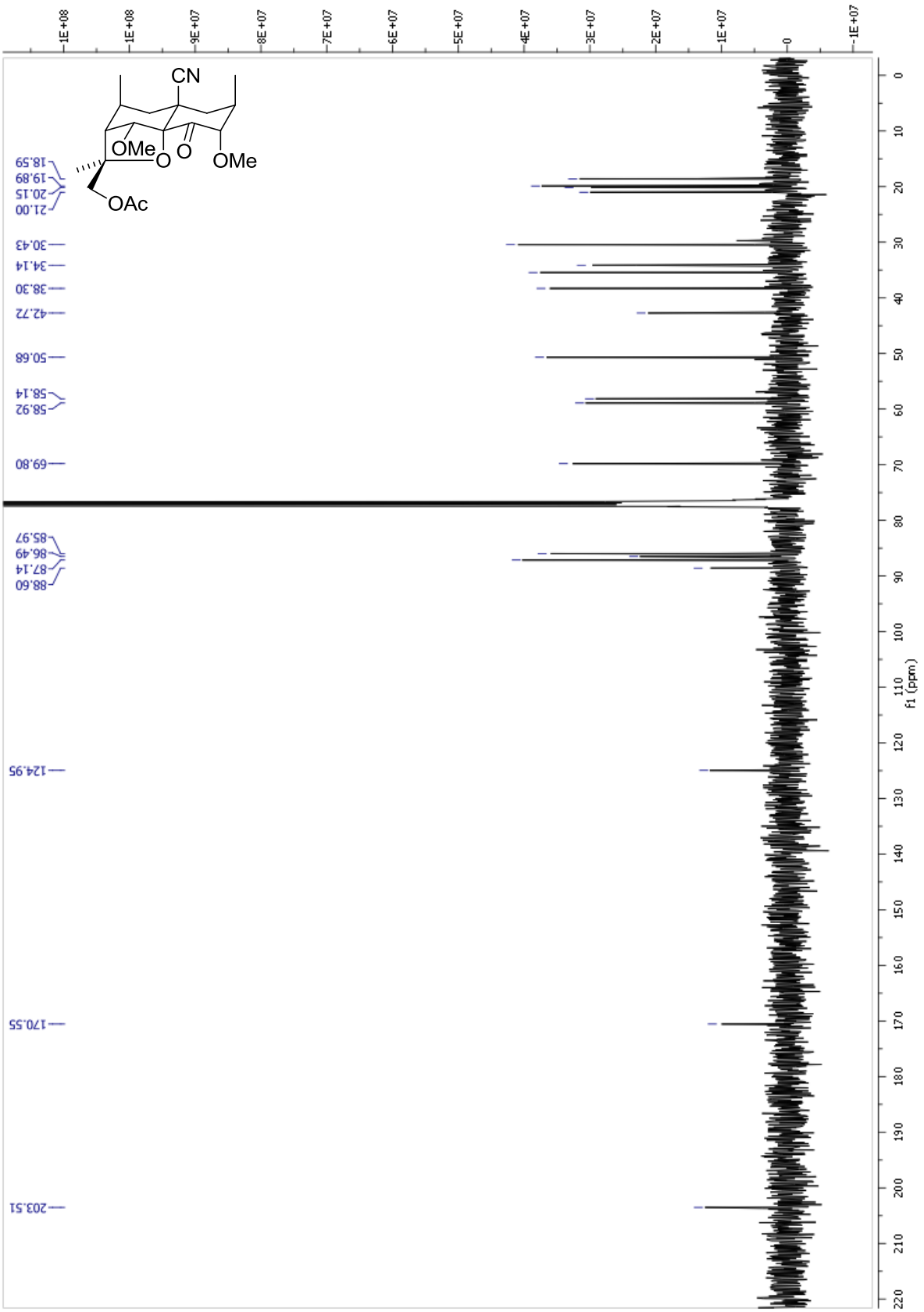
β-Dihydroagarofuran 36 – ^{13}C NMR (100 MHz)



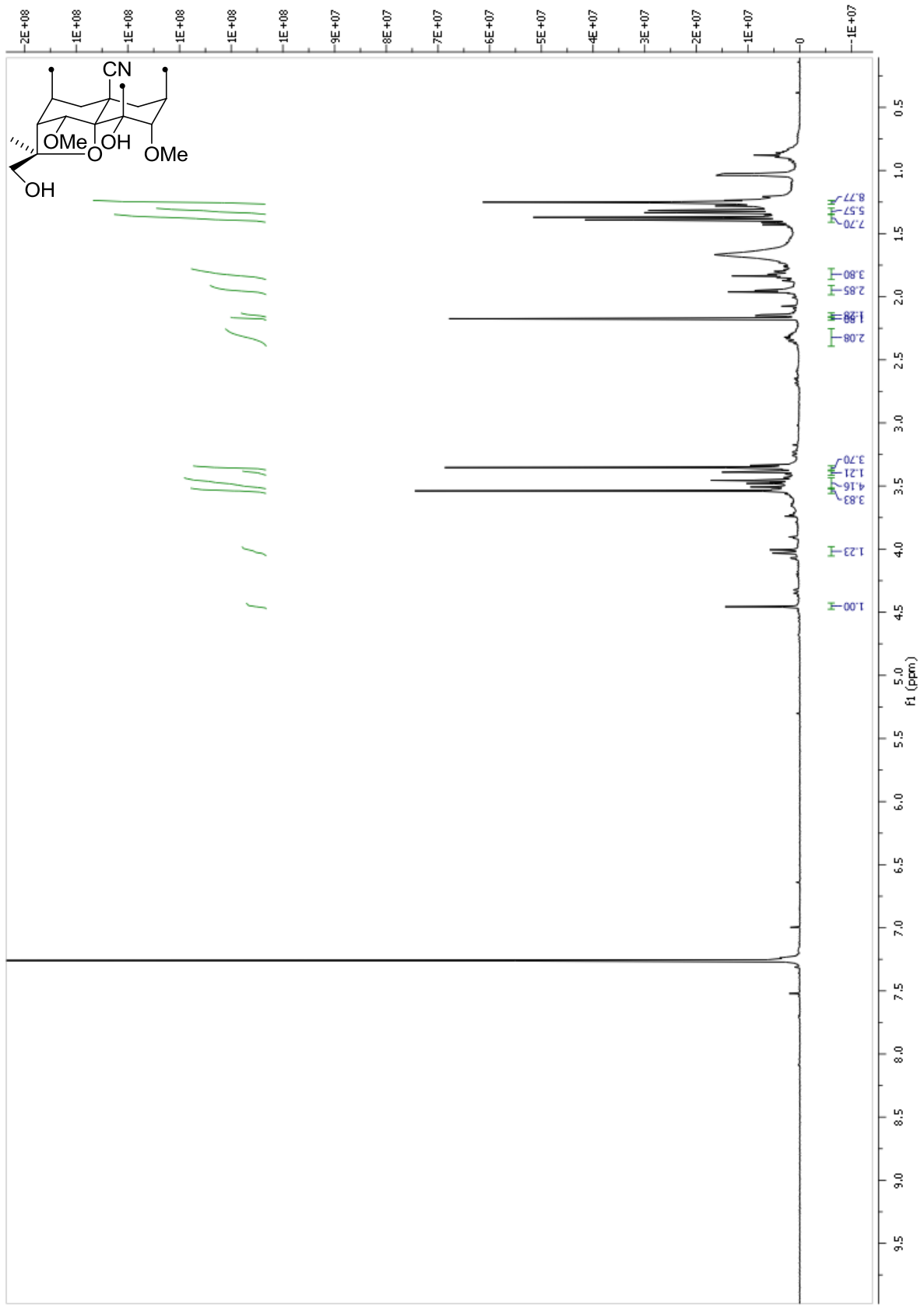
Ketone 37 – ^{1}H NMR (400 MHz)



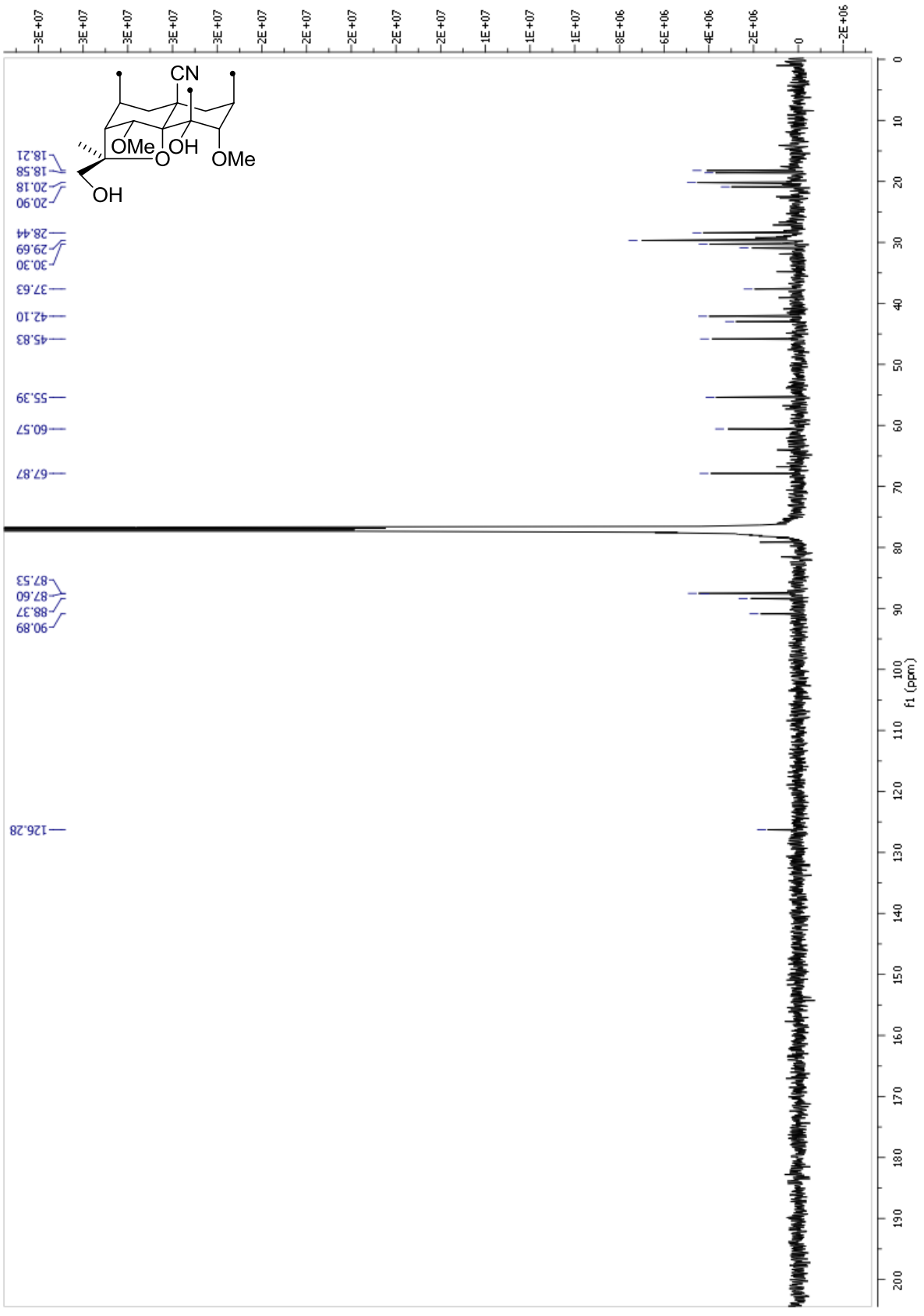
Ketone 37 – ^{13}C NMR (100 MHz)



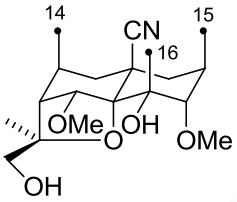
Diol 4 – ^{1}H NMR (400 MHz)

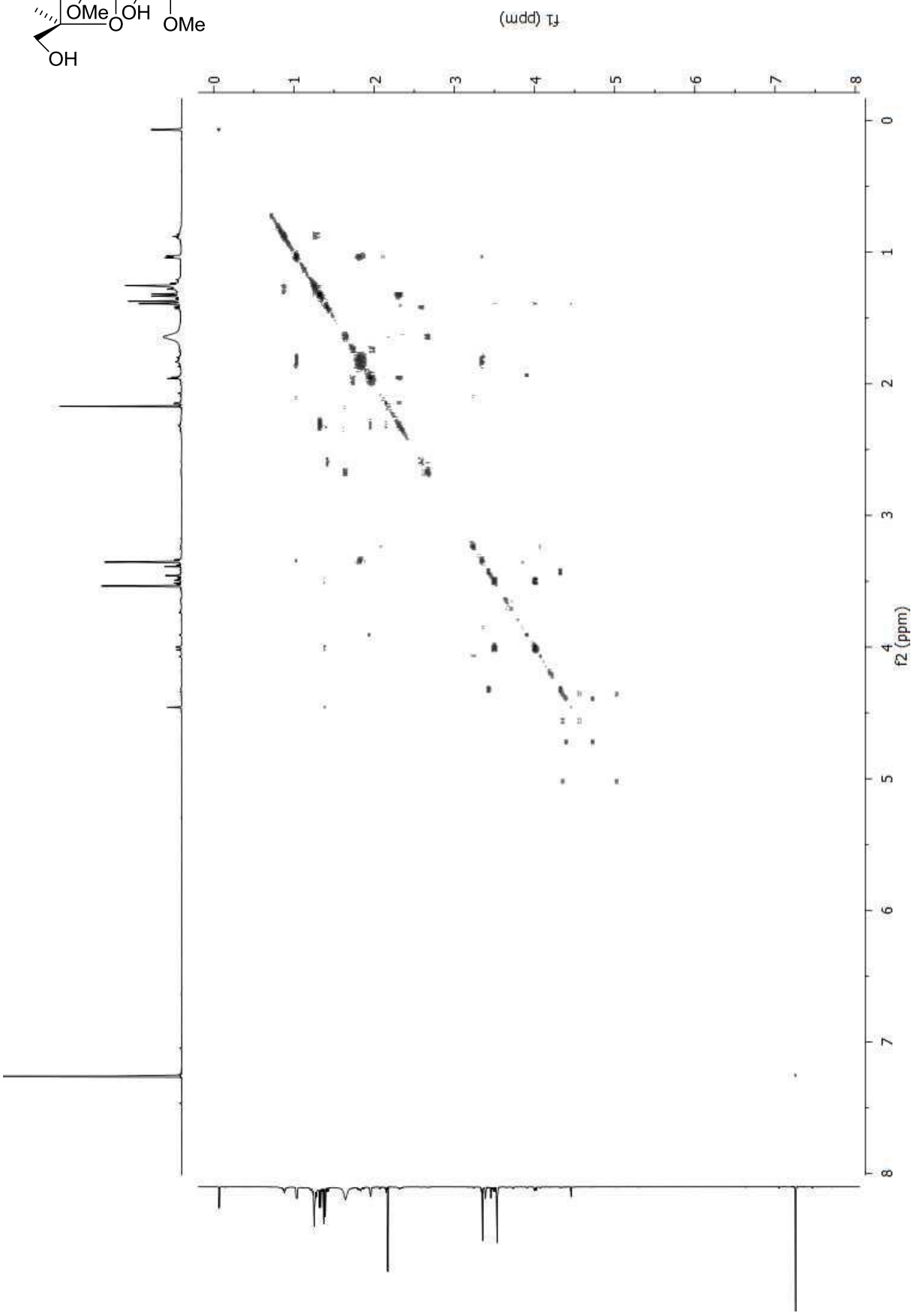


Diol 4 – ^{13}C NMR (100 MHz)



Diol 4 - – COSY (500 MHz)





Diol 4 - HSQC (500 MHz)