

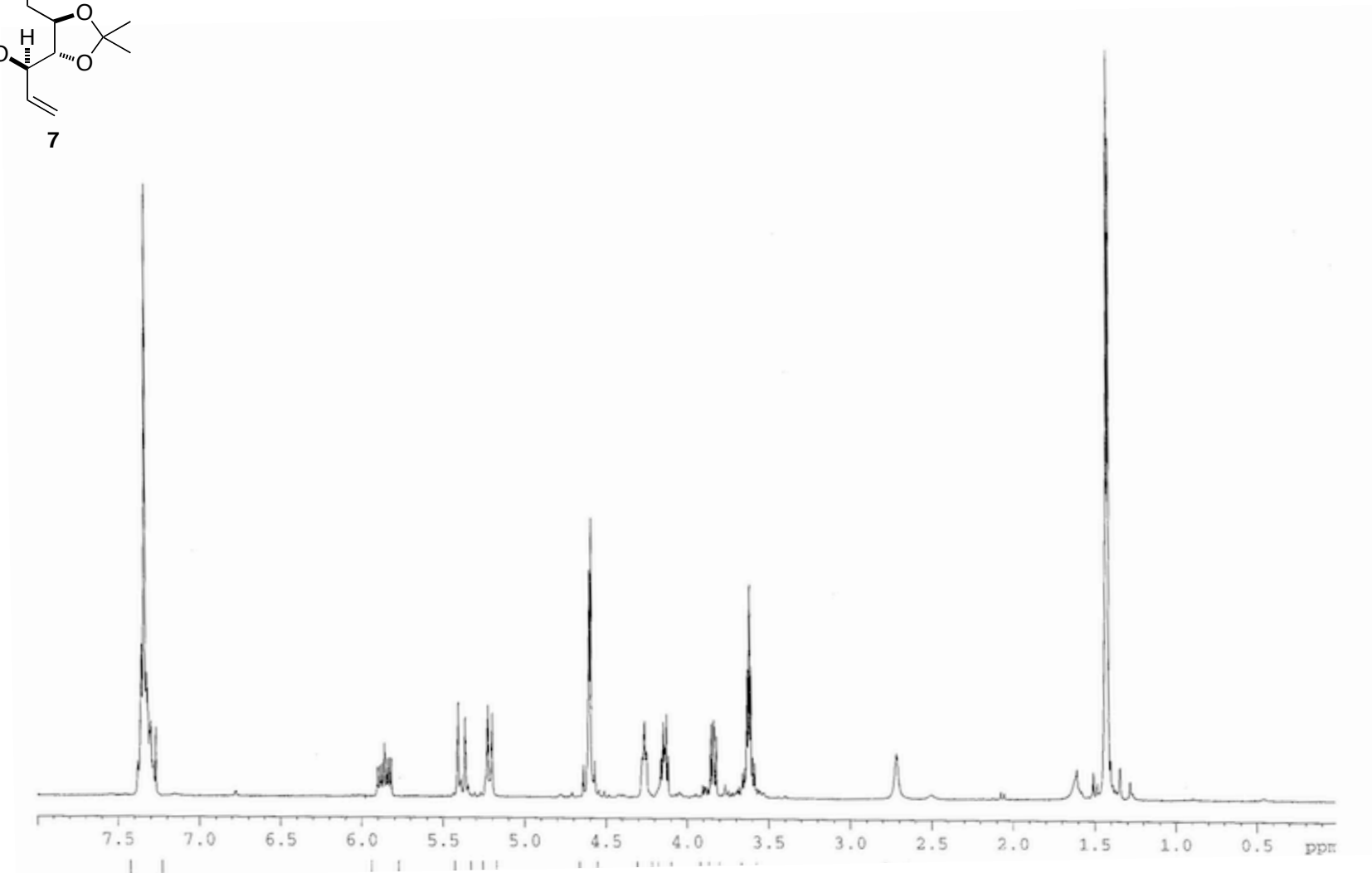
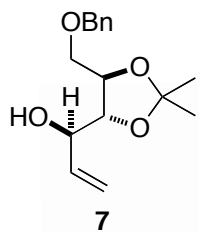
Synthesis of the bis-tetrahydropyran Core of Amphidinol 3

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*Kenan and Caudill Laboratories of Chemistry, University of North Carolina at
Chapel Hill, North Carolina 27599*

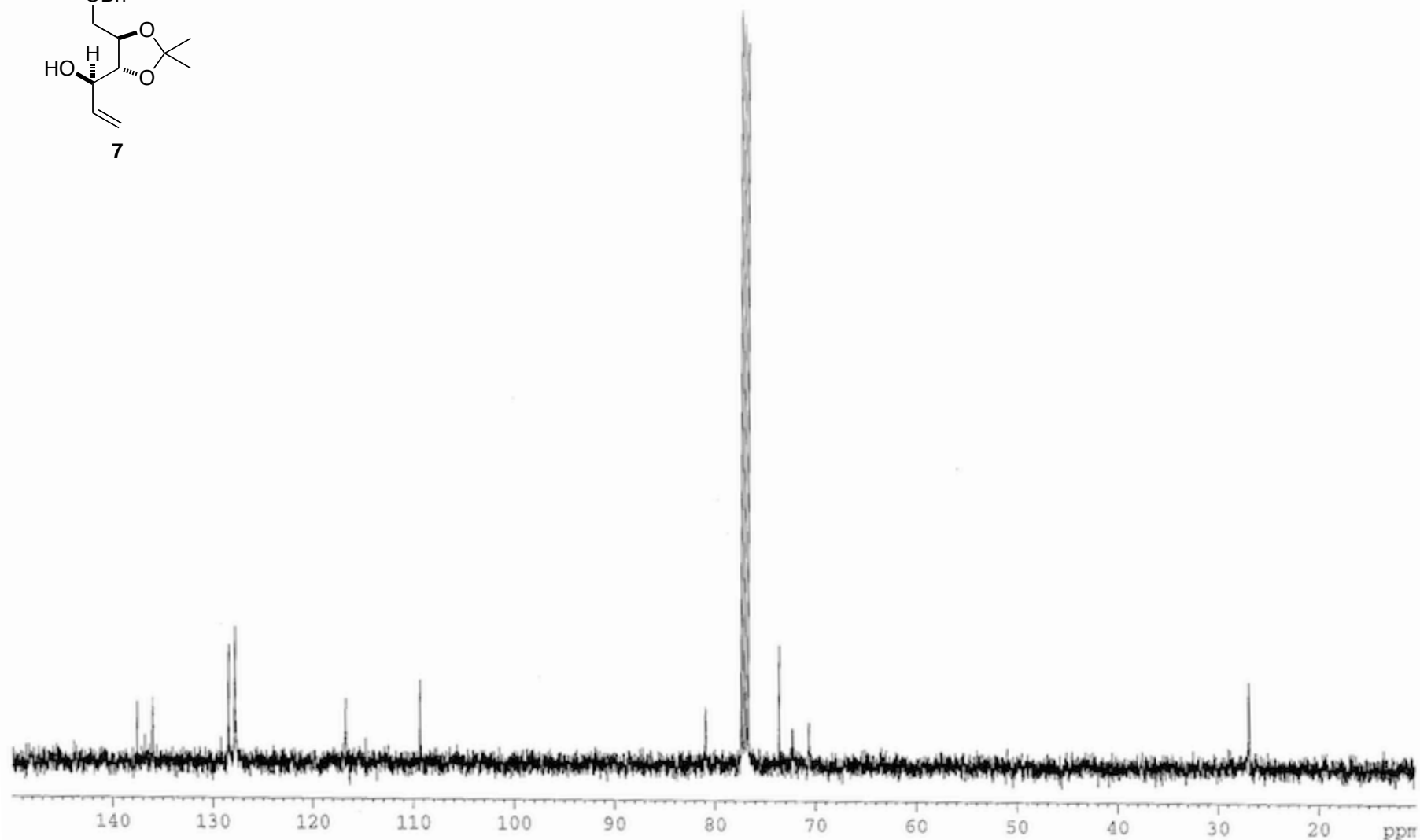
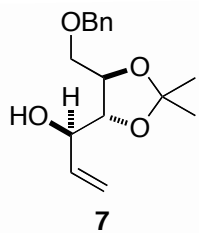
crimmins@email.unc.edu

Supporting Information: ^1H and ^{13}C spectra

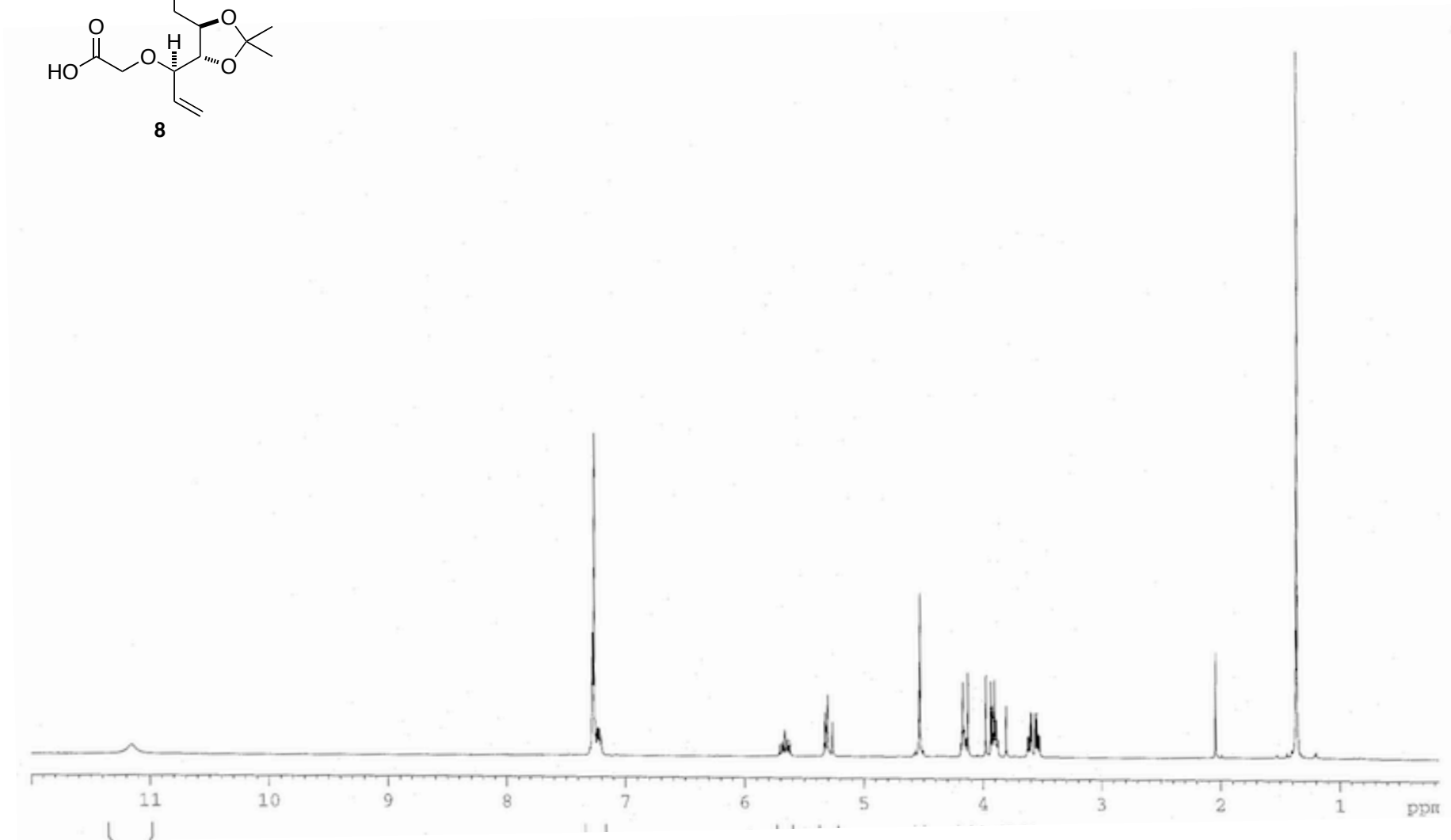
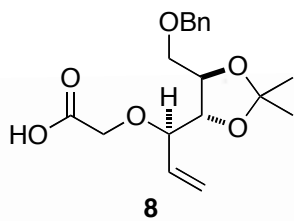
^1H NMR (400 MHz) of allylic alcohol **7** (in CDCl_3)



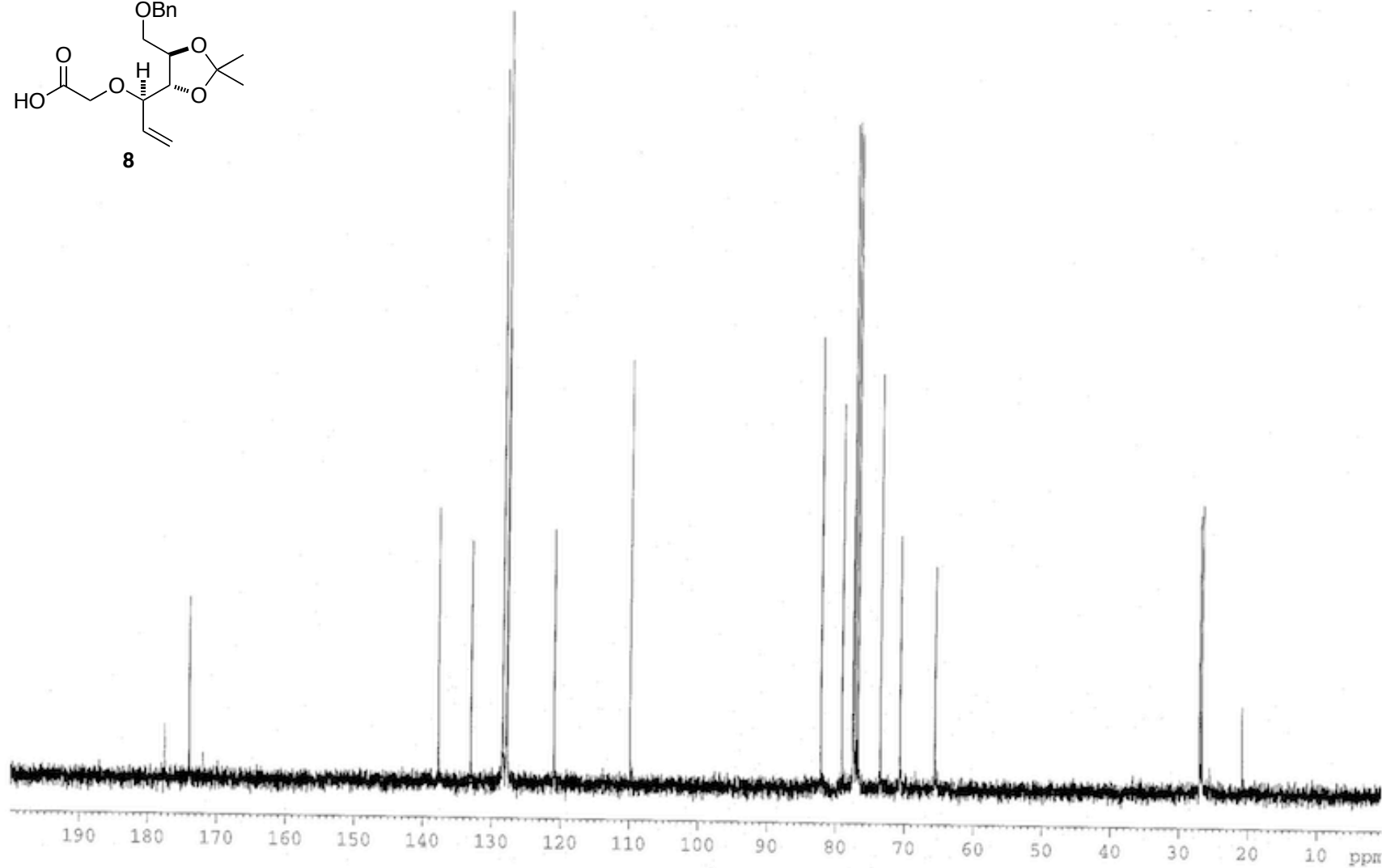
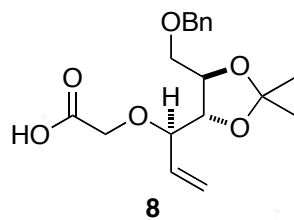
^{13}C NMR (100 MHz) of alcohol **7** (in CDCl_3)



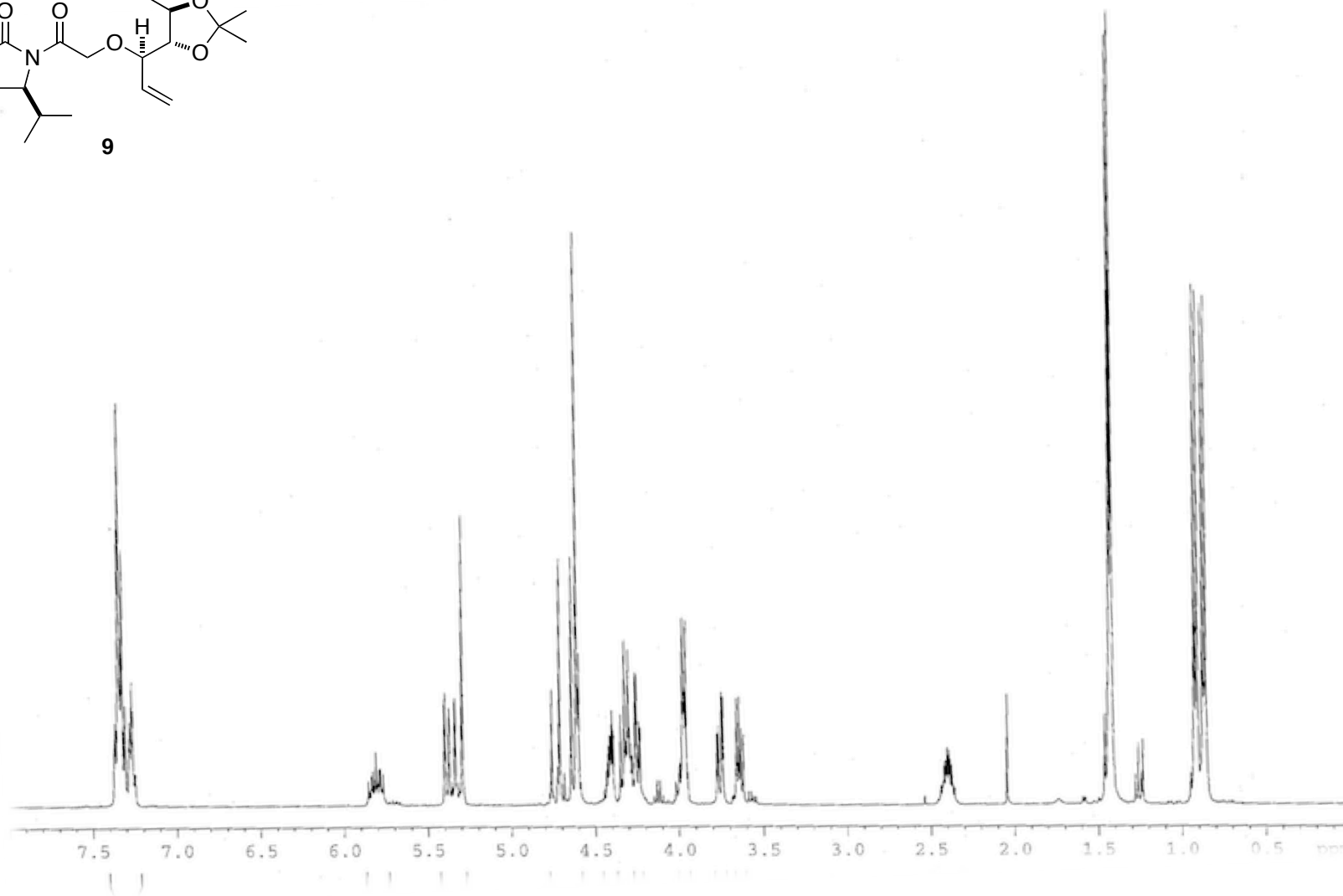
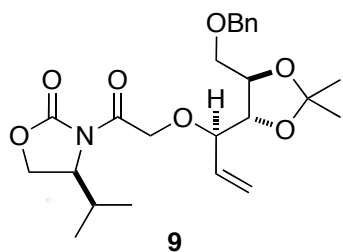
^1H NMR (400 MHz) of acid **8** (in CDCl_3)



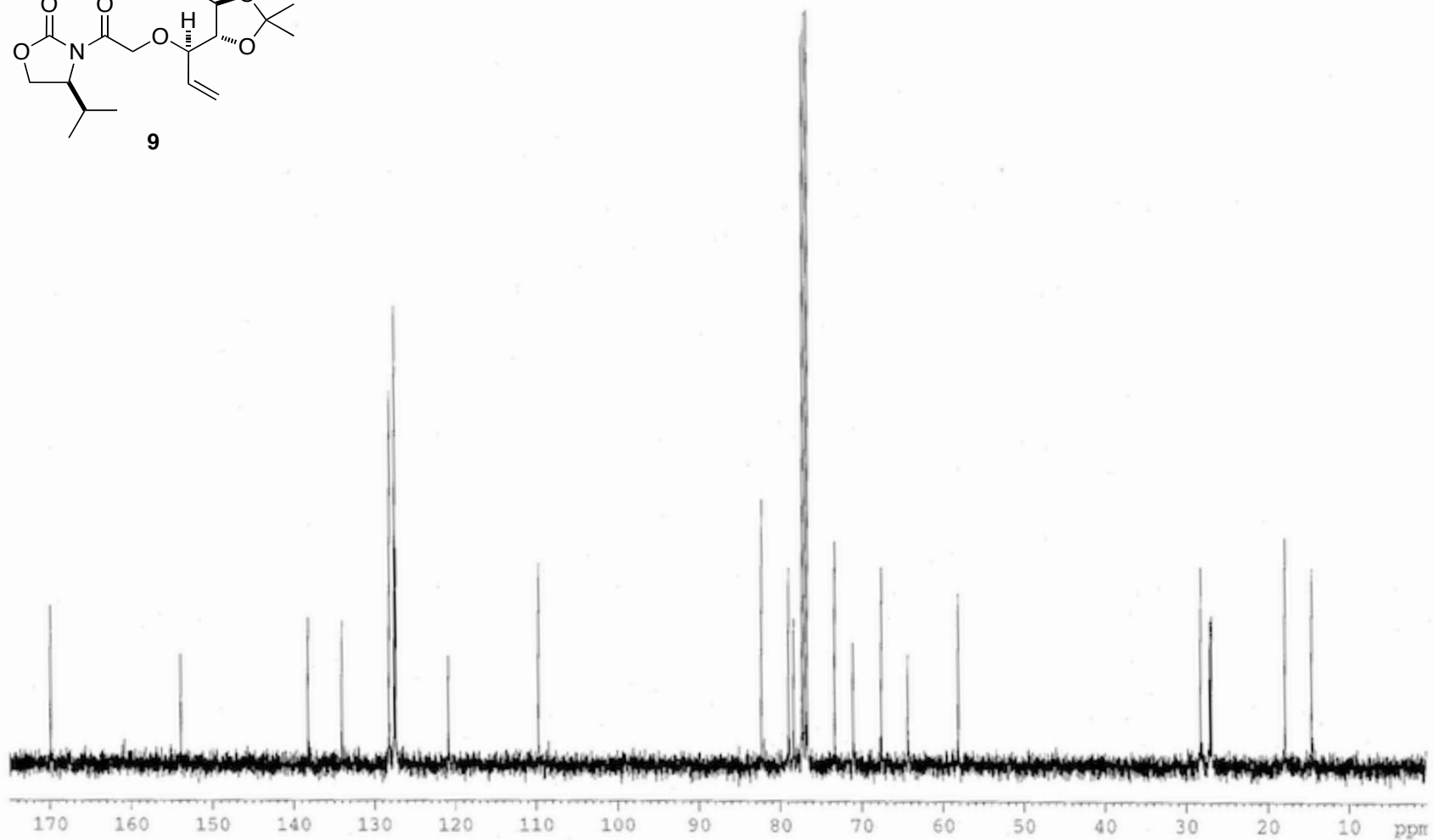
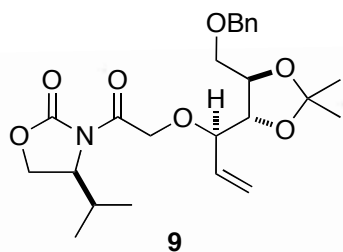
^{13}C NMR (100 MHz) of acid **8** (in CDCl_3)



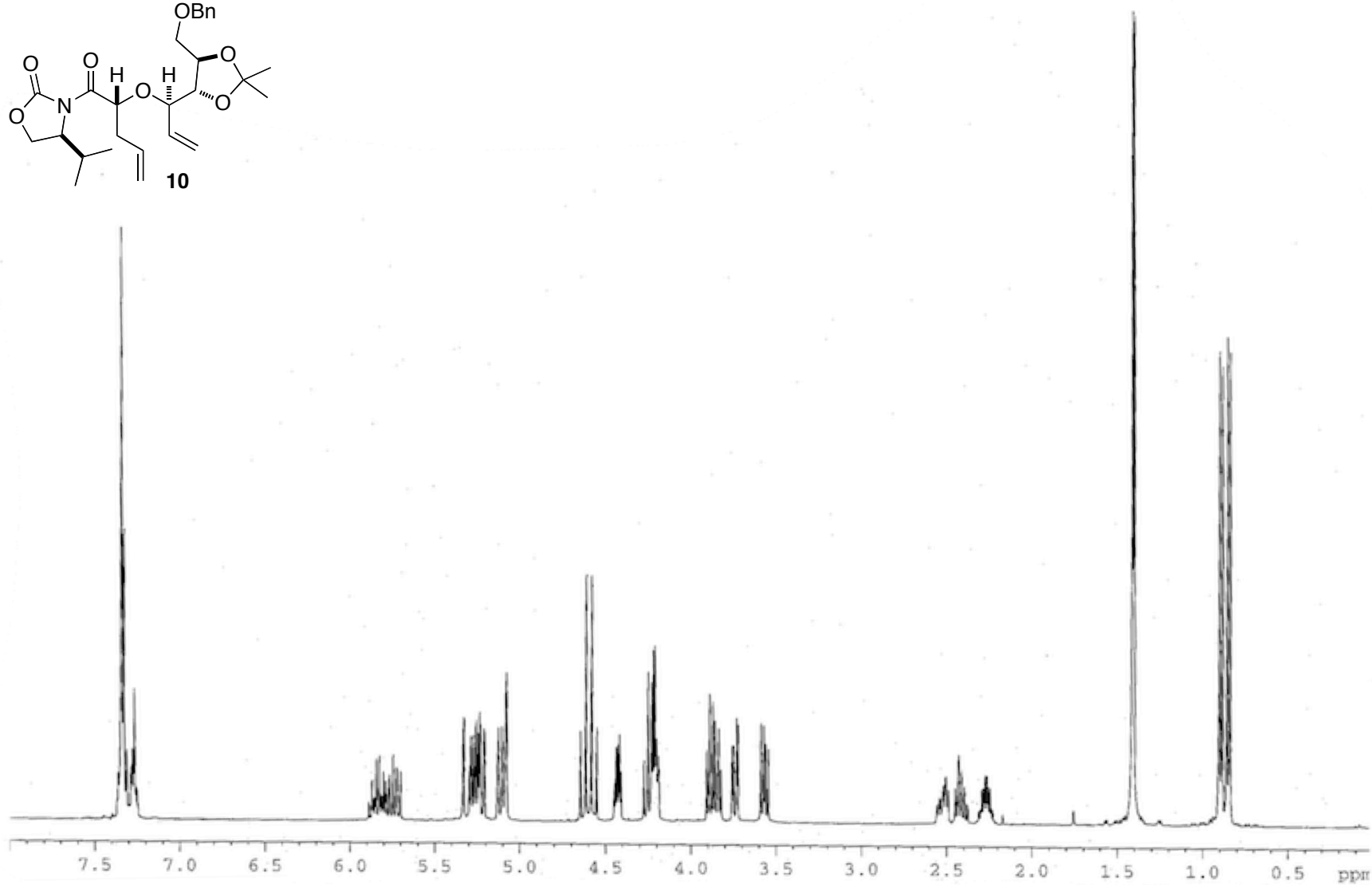
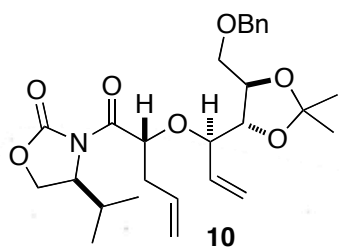
^1H NMR (400 MHz) of glycolate **9** (in CDCl_3)



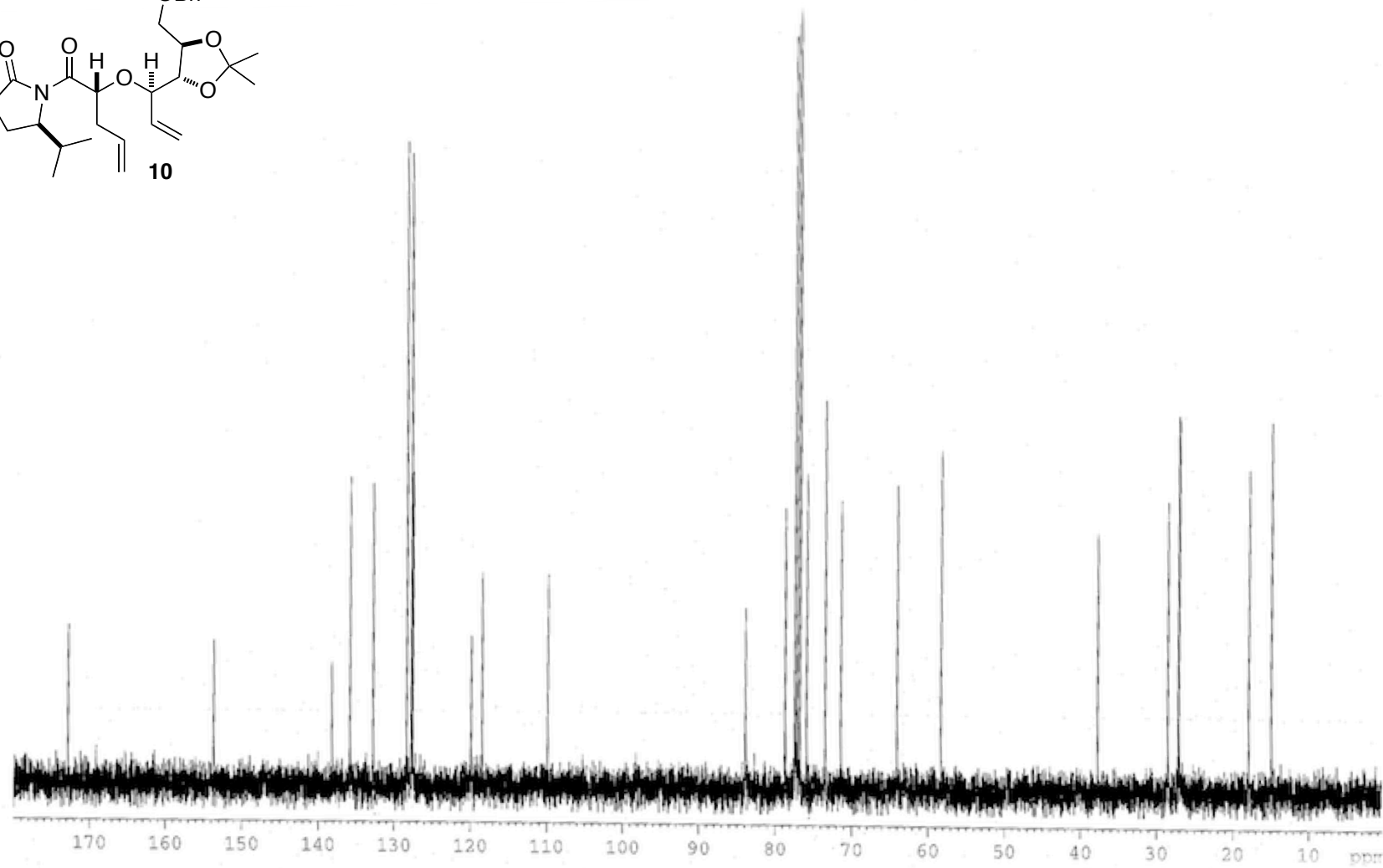
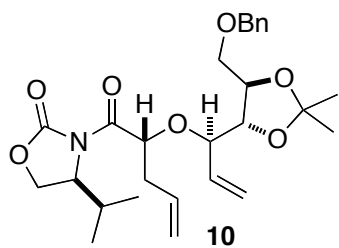
^{13}C NMR (100 MHz) of glycolate **9** (in CDCl_3)



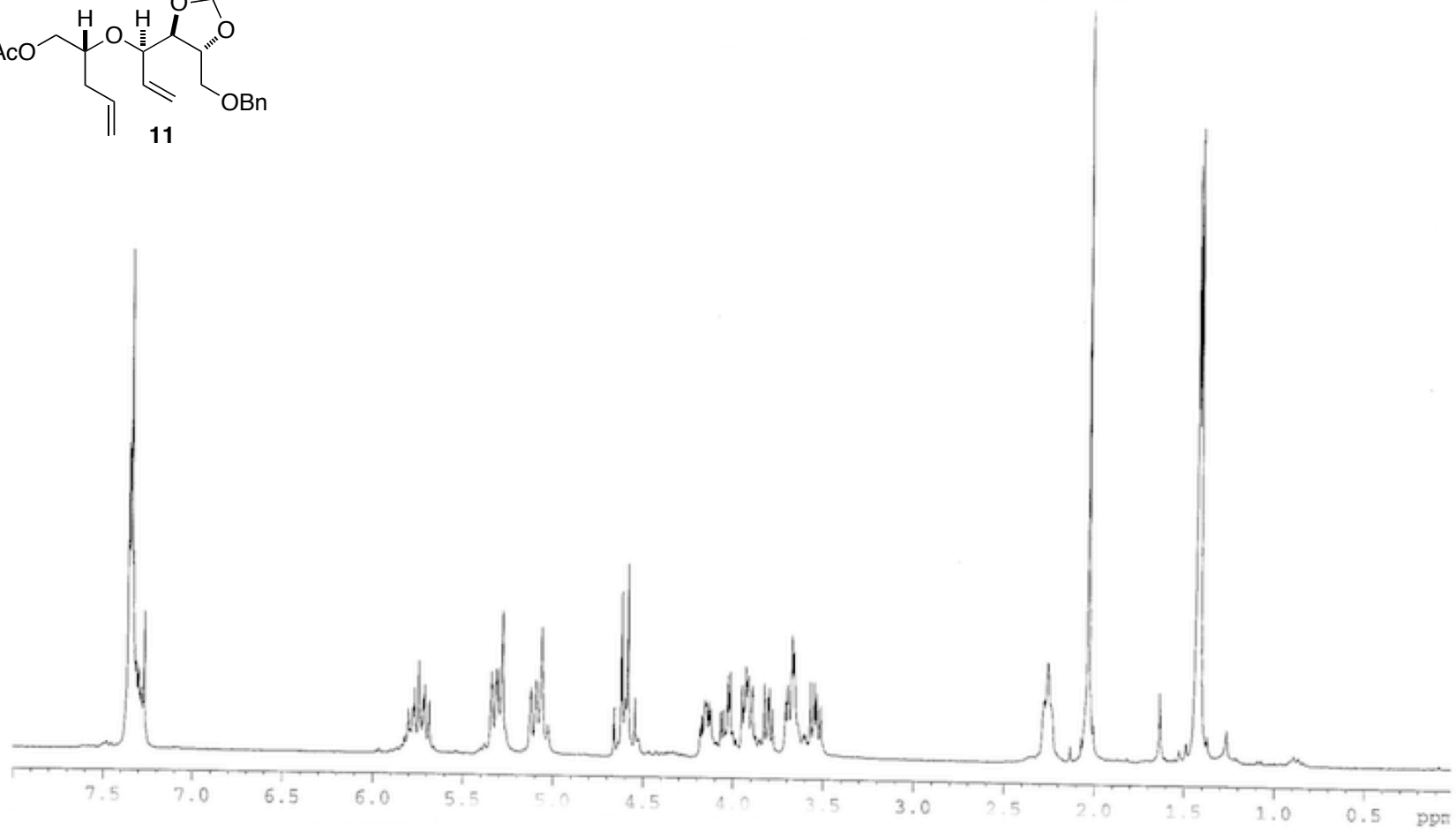
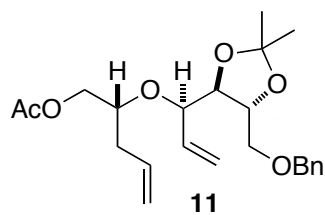
^1H NMR (400 MHz) of alkylation product **10** (in CDCl_3)



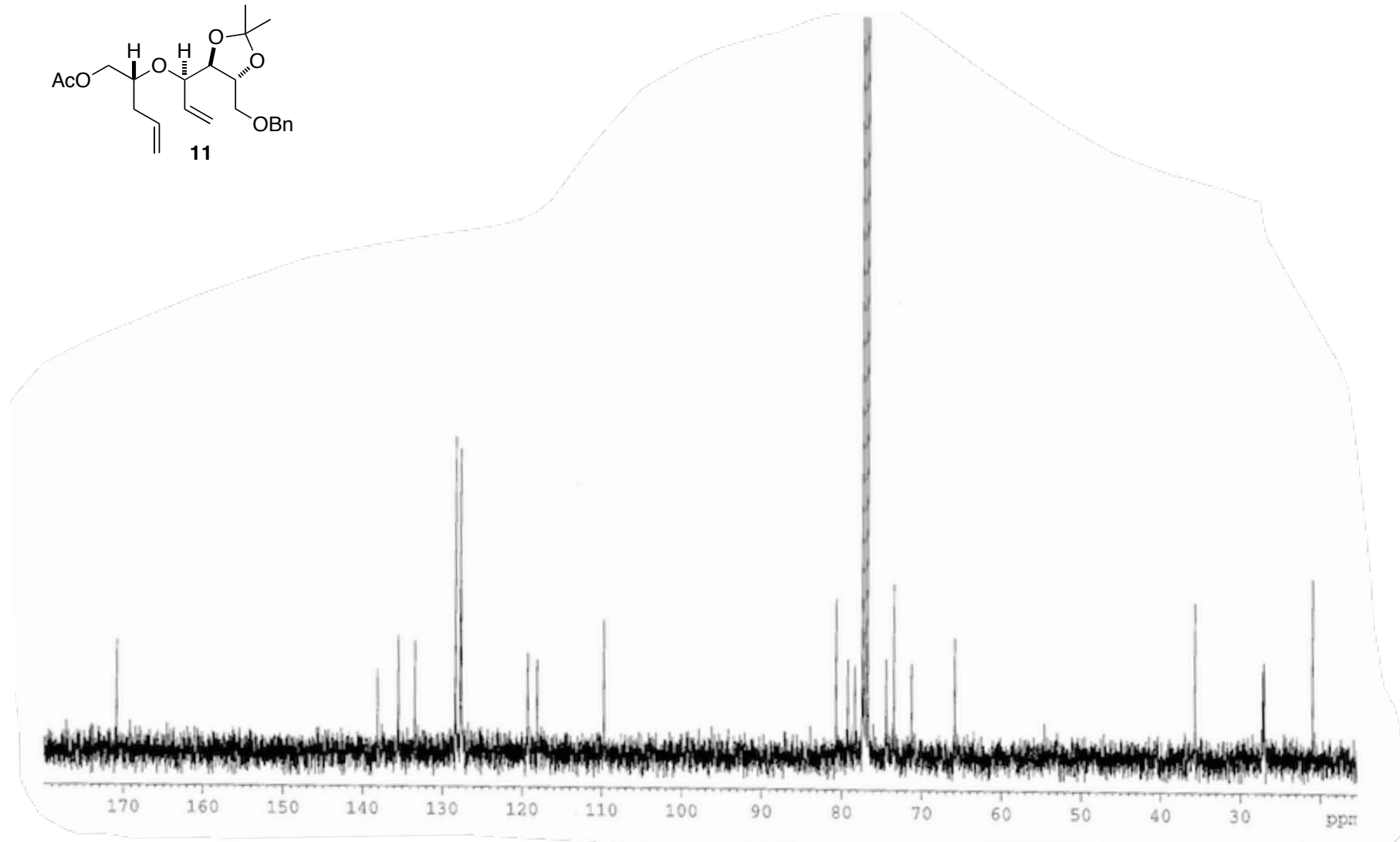
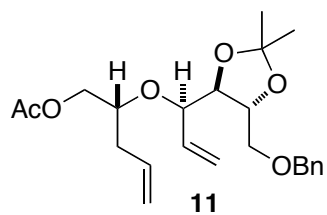
^{13}C NMR (100 MHz) of alkylation product **10** (in CDCl_3)



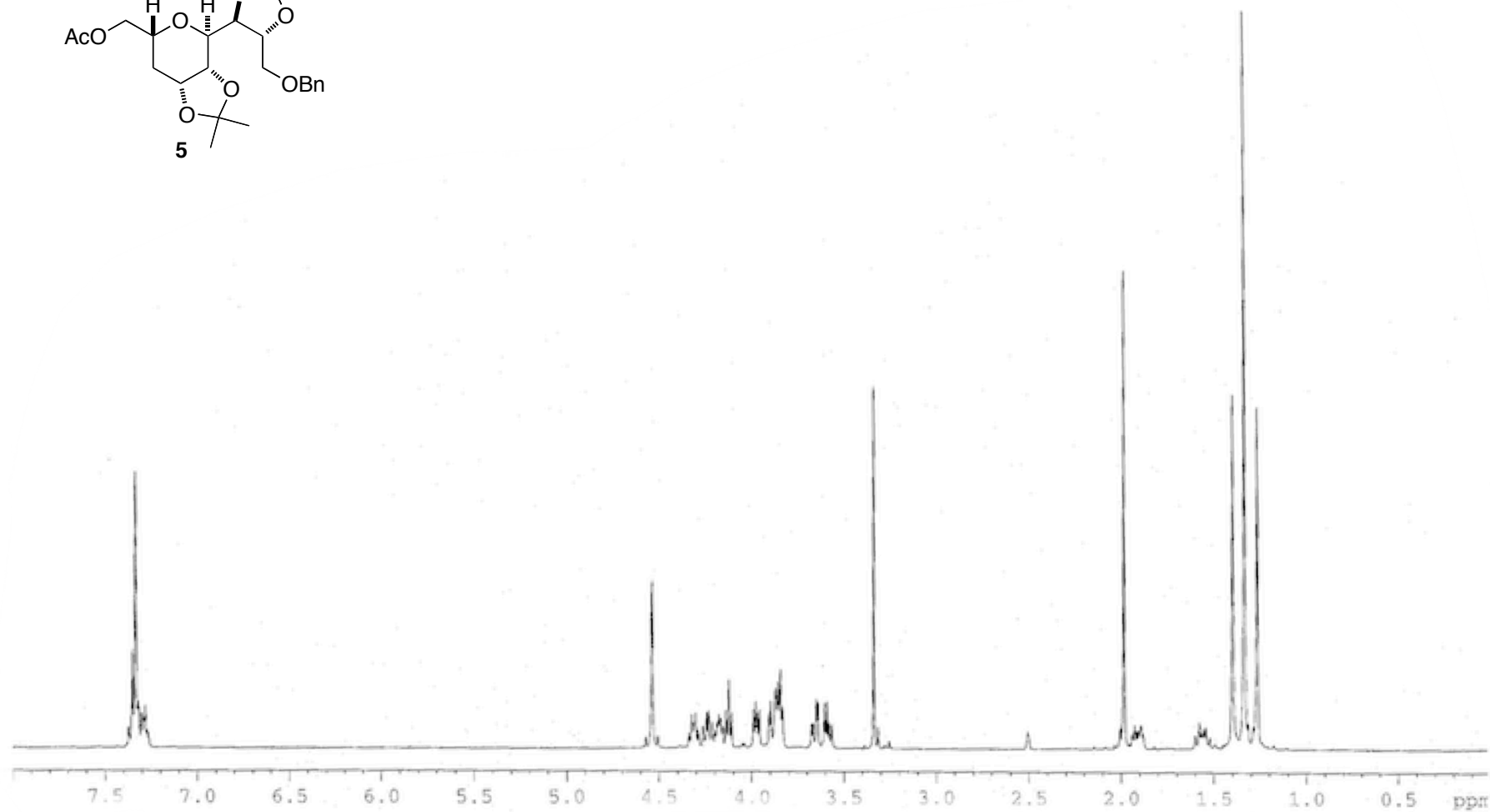
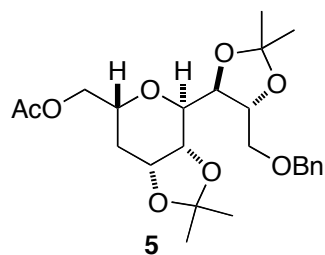
^1H NMR (300 MHz) of diene **11** (in CDCl_3)



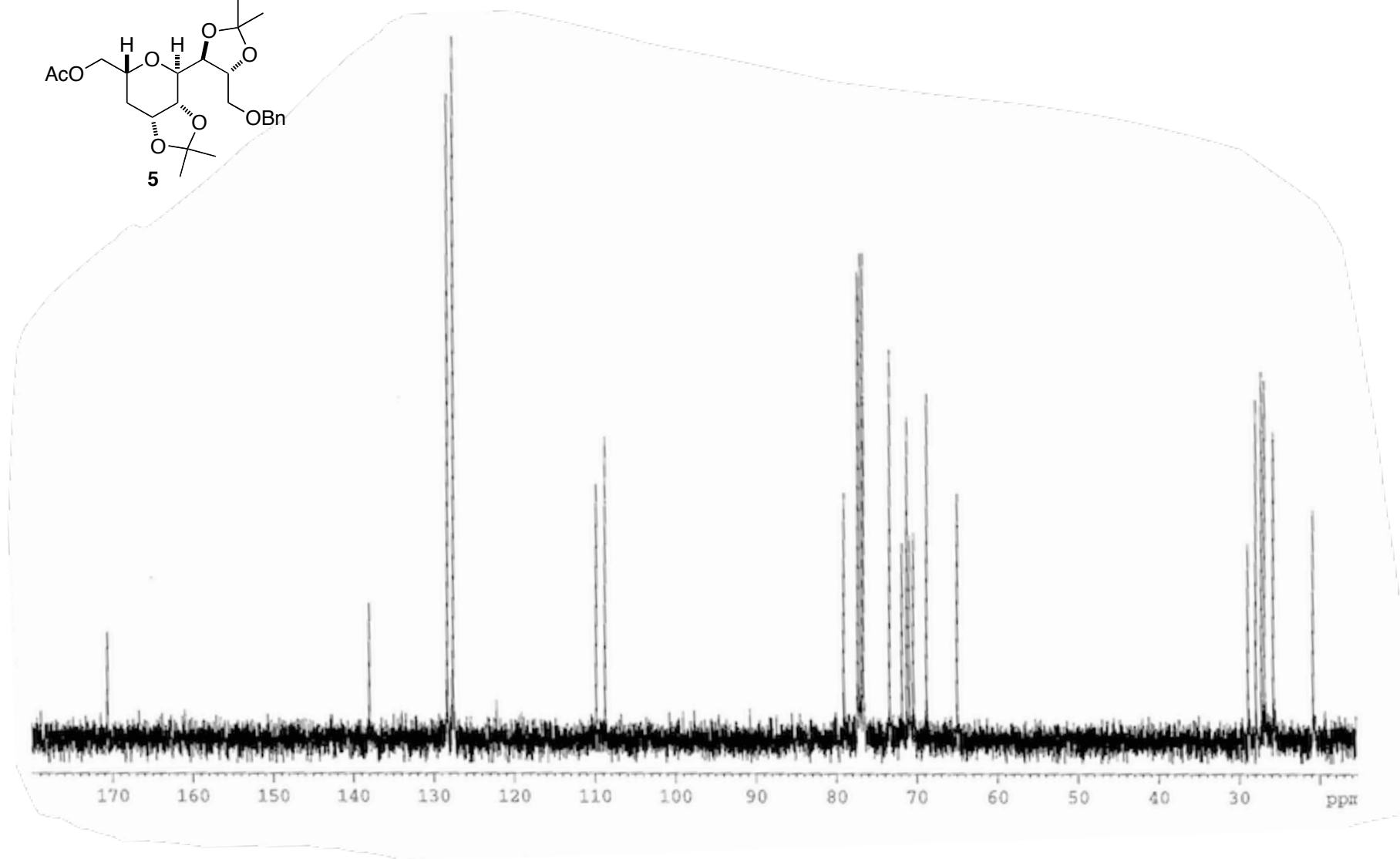
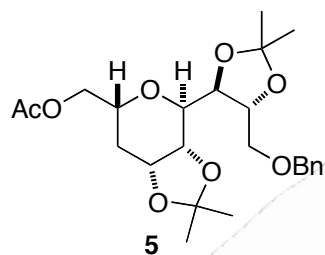
^{13}C NMR (100 MHz) of diene **11** (in CDCl_3)



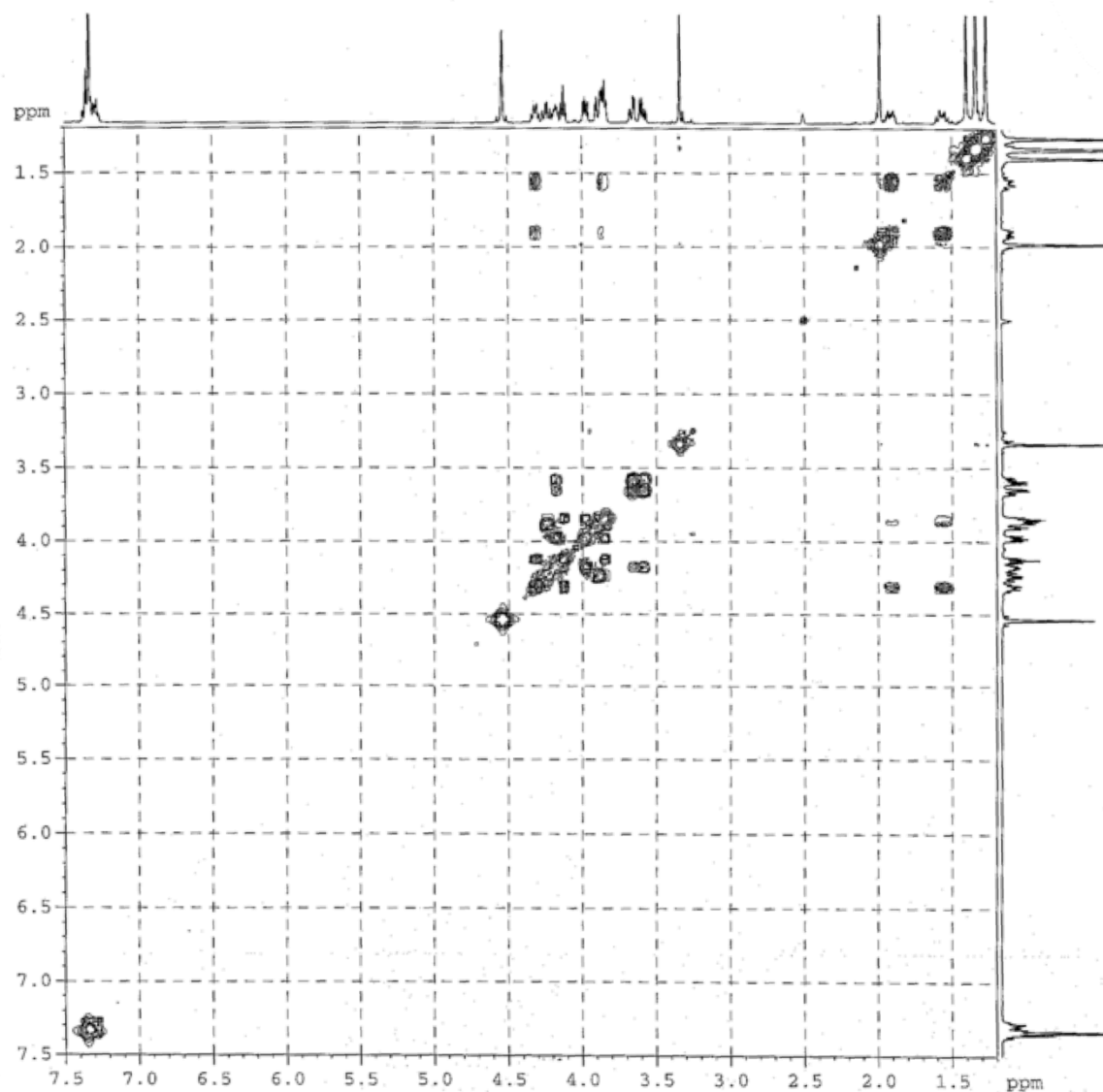
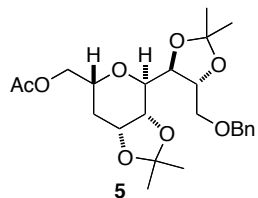
^1H NMR (400 MHz) of THP **5** (in DMSO)



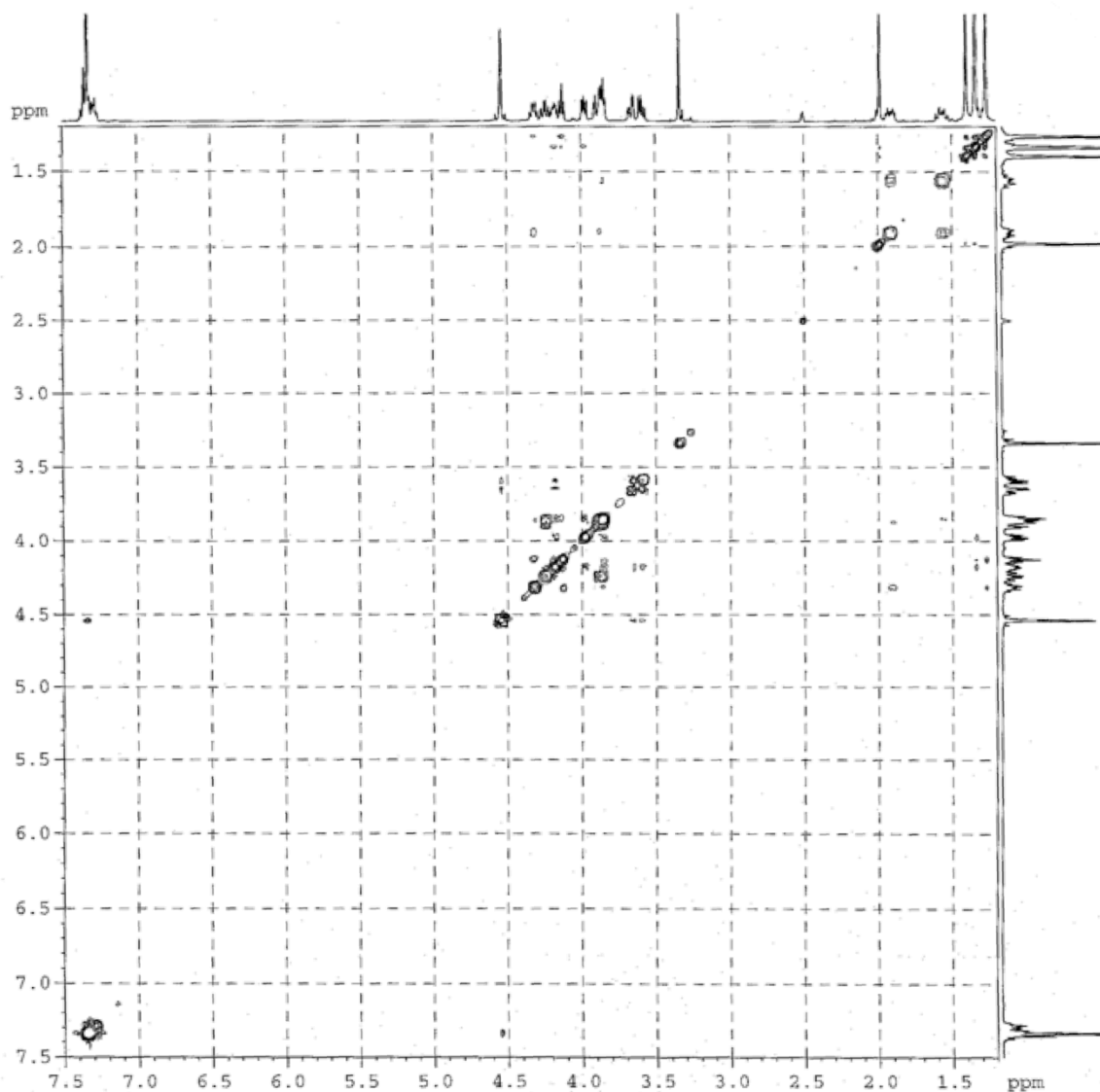
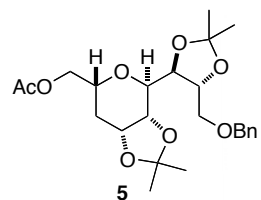
^{13}C NMR (100 MHz) of THP **5** (in CDCl_3)



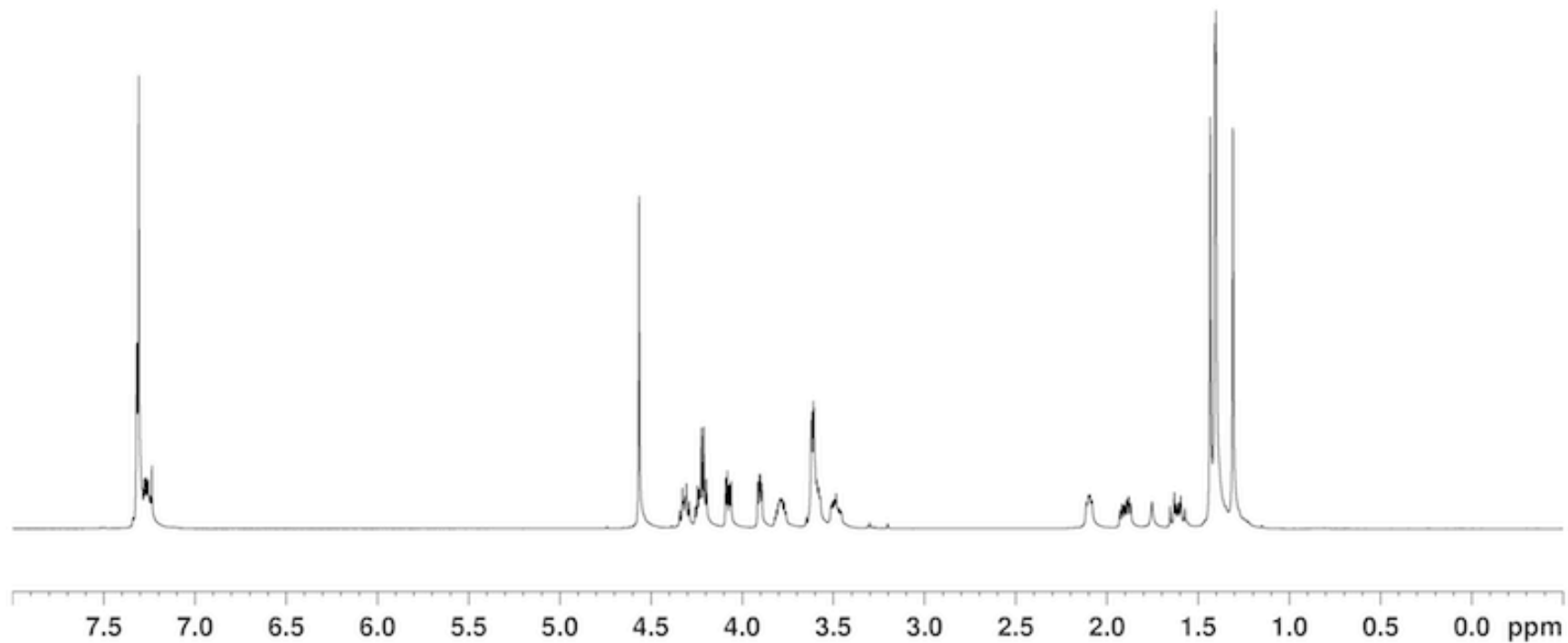
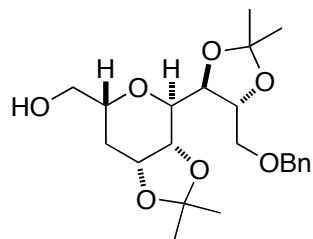
COSY (400 MHz) of THP 5 (in DMSO)



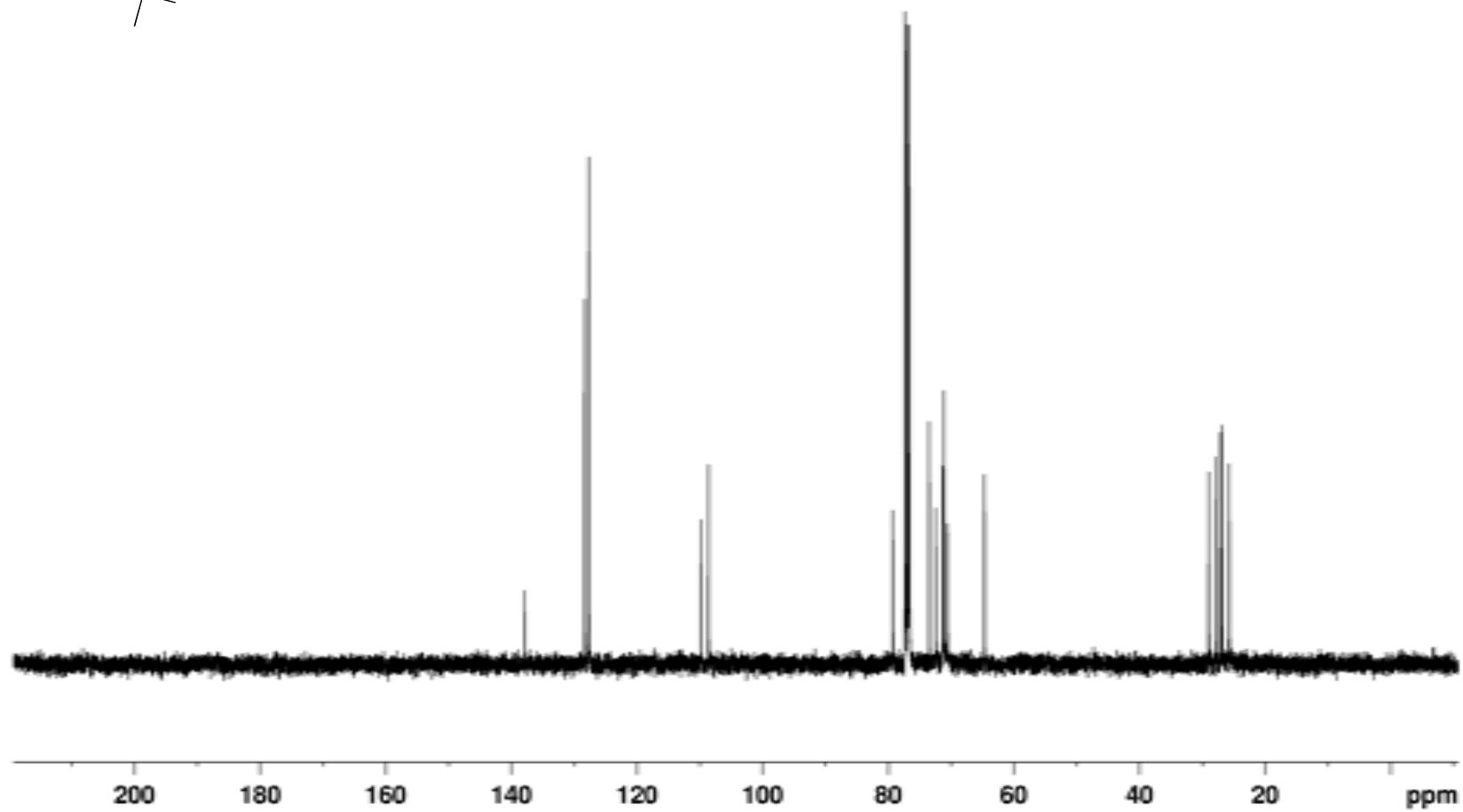
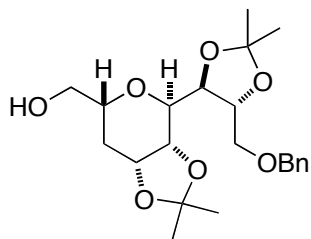
NOESY (400 MHz) of THP 5 (in DMSO)



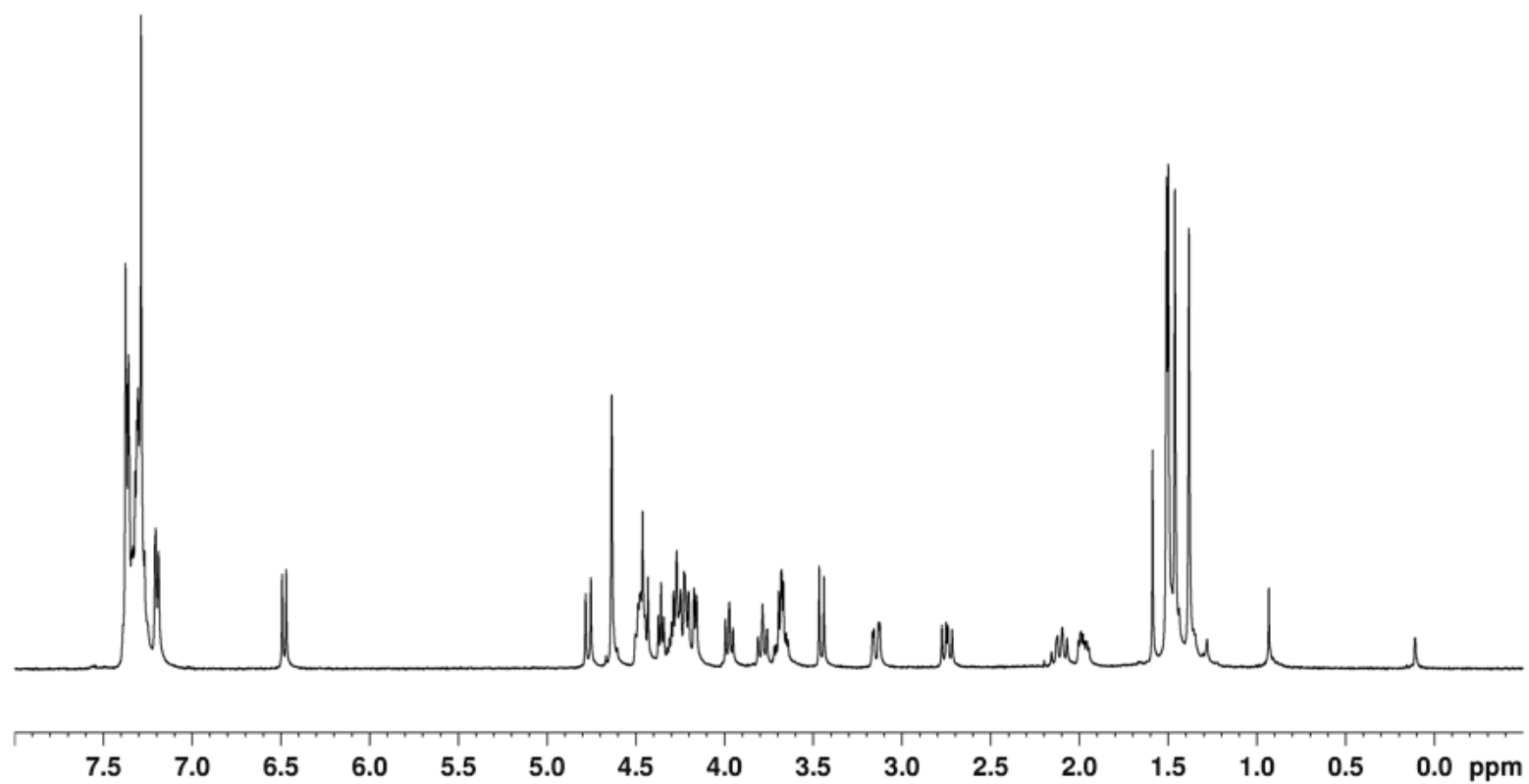
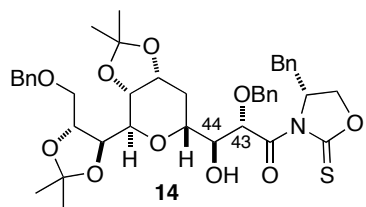
^1H NMR (400 MHz) of primary alcohol (in CDCl_3)



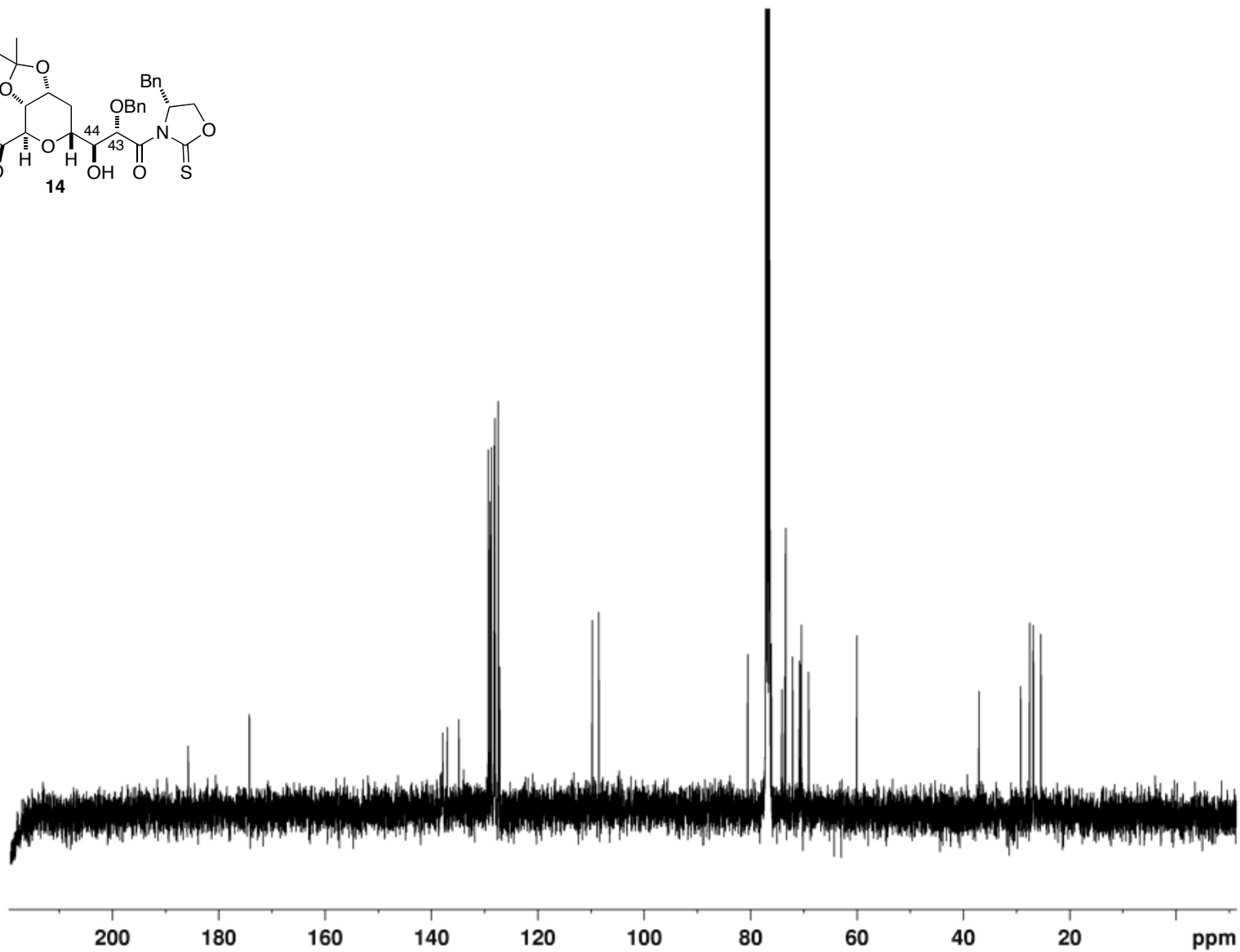
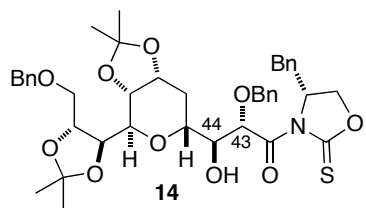
^{13}C NMR (100 MHz) of primary alcohol (in CDCl_3)



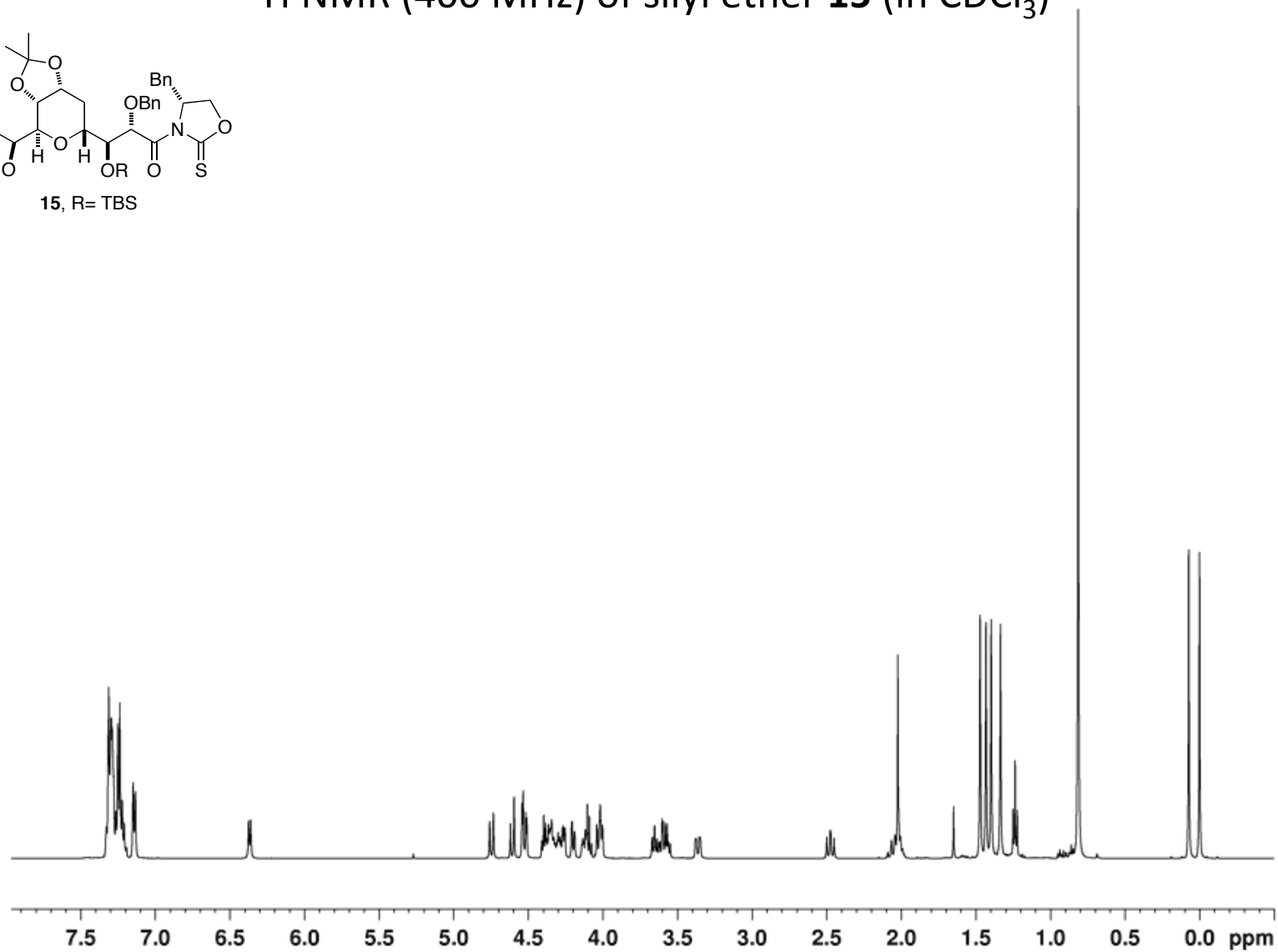
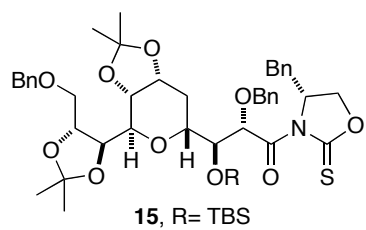
^1H NMR (400 MHz) of *anti* aldol adduct **14** (in CDCl_3)



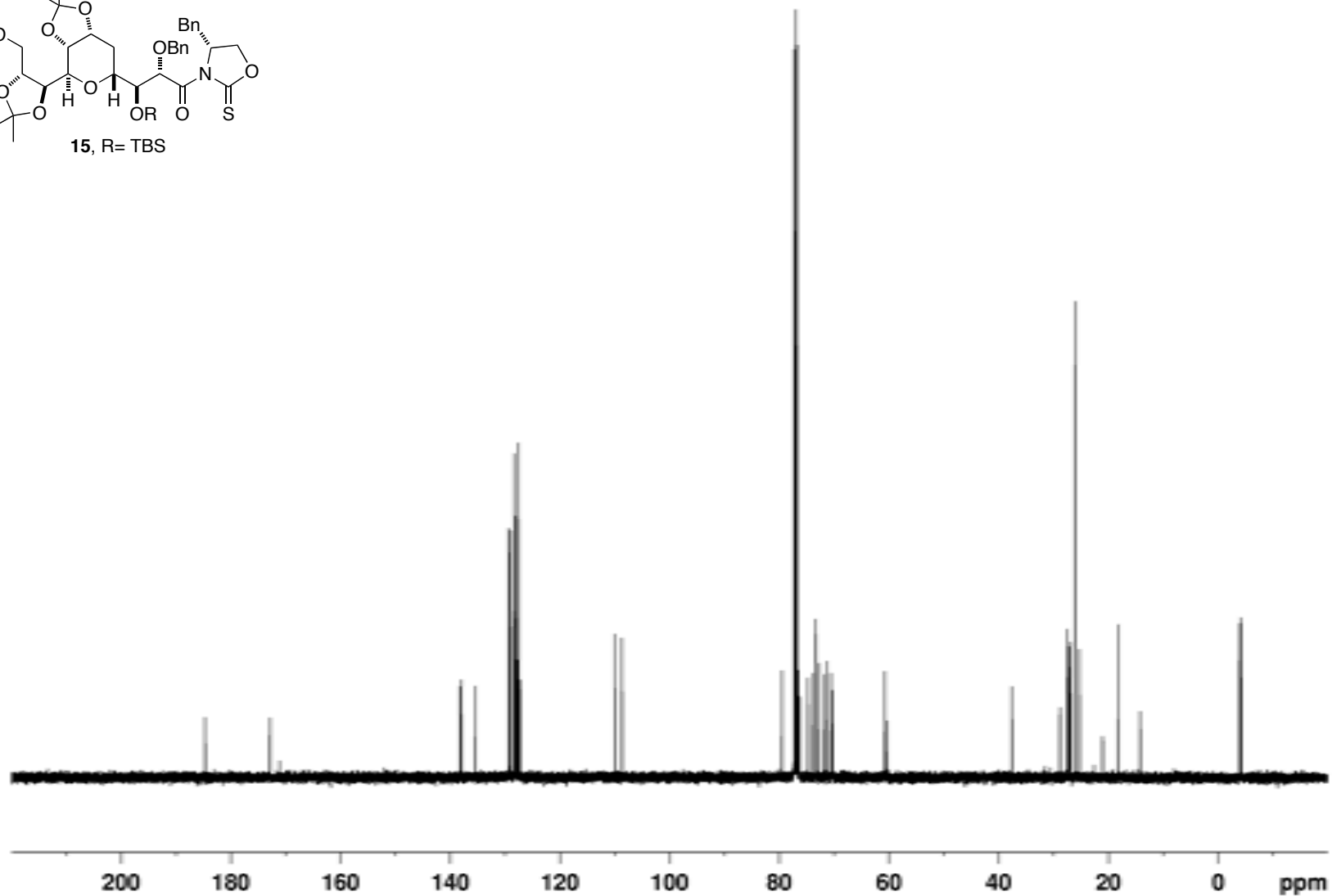
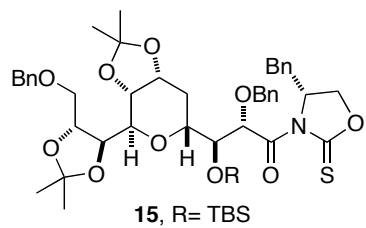
^{13}C NMR (100 MHz) of *anti* aldol adduct **14** (in CDCl_3)



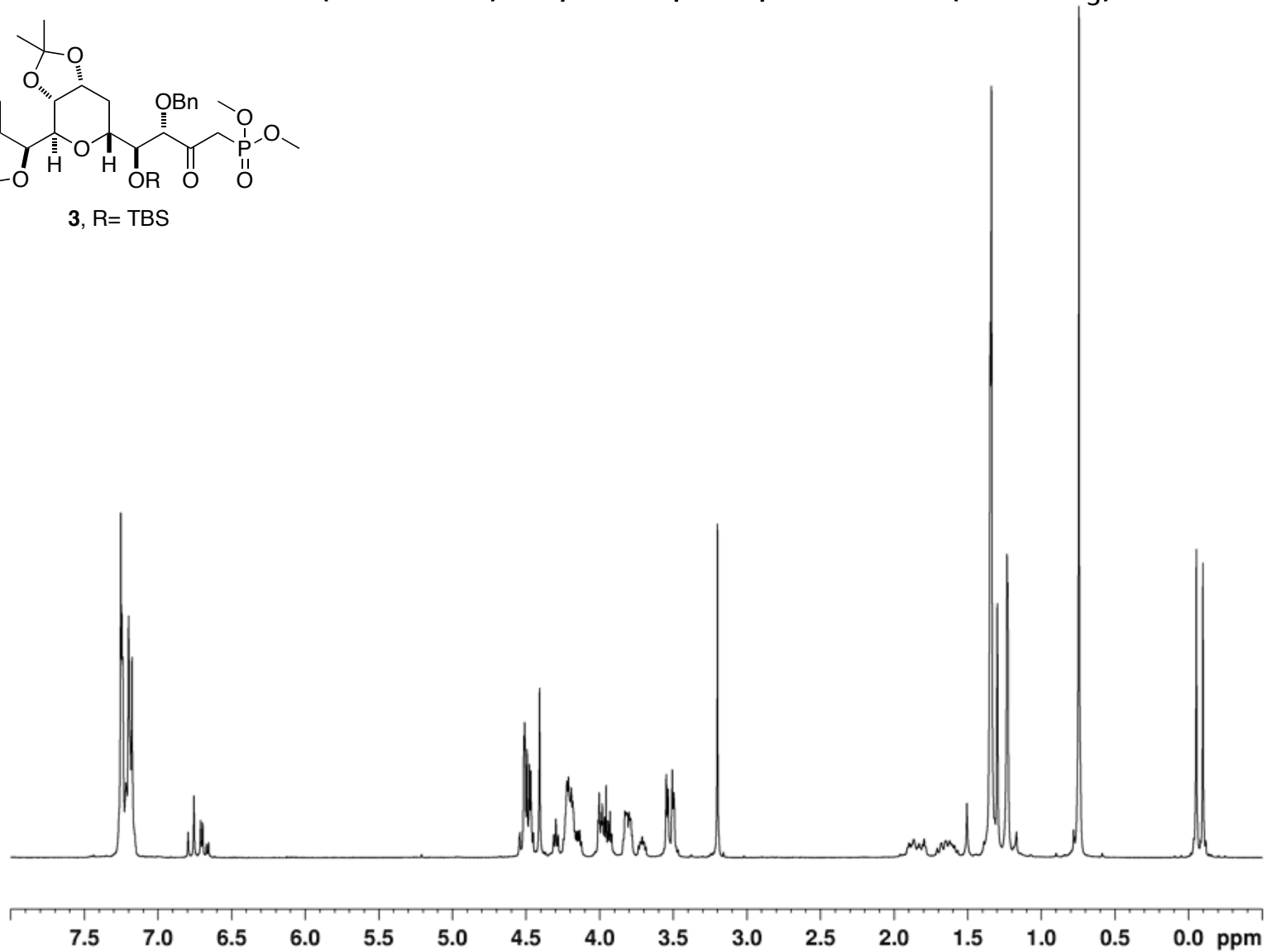
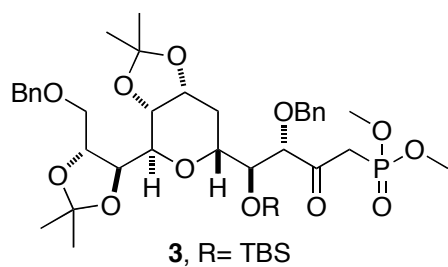
^1H NMR (400 MHz) of silyl ether **15** (in CDCl_3)



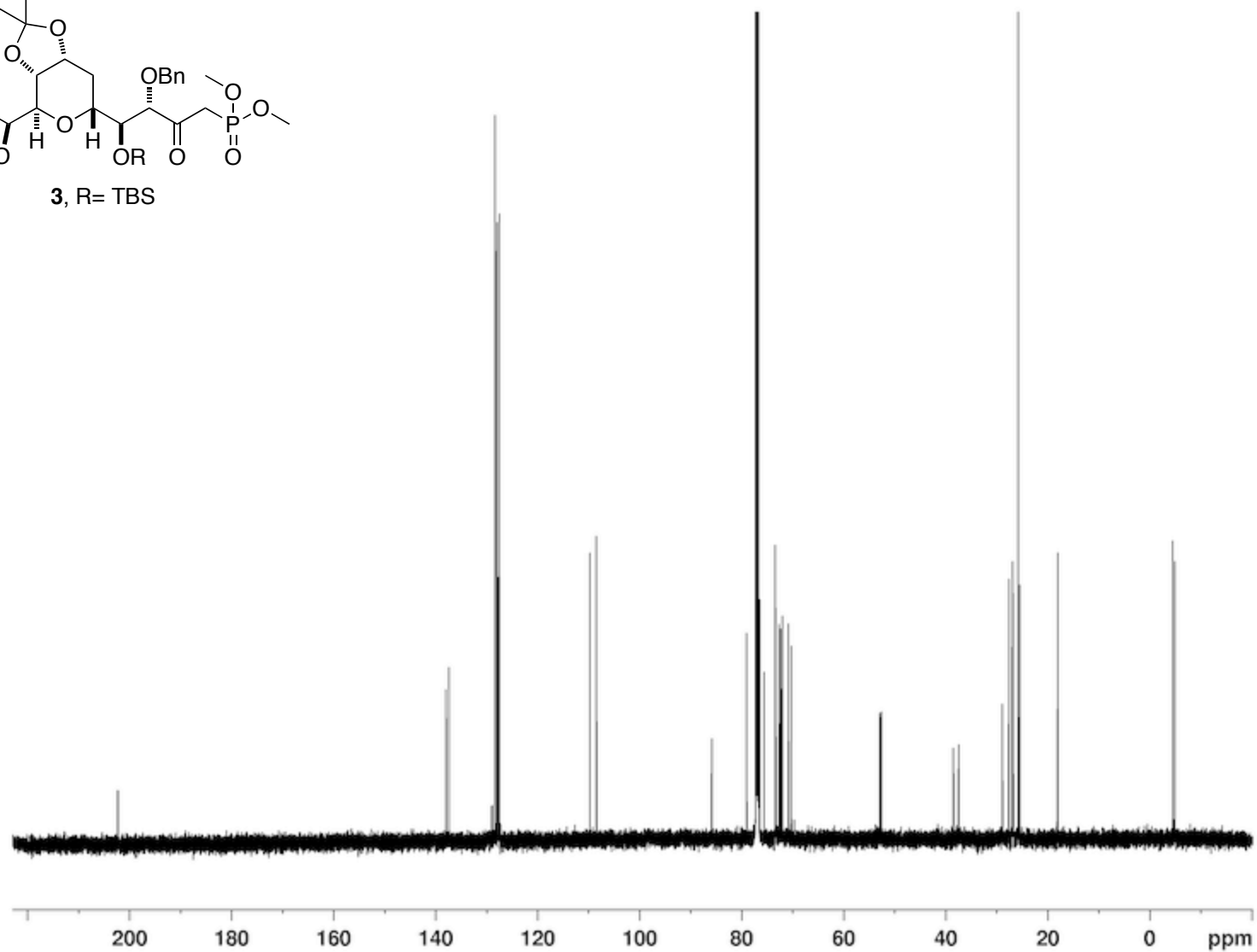
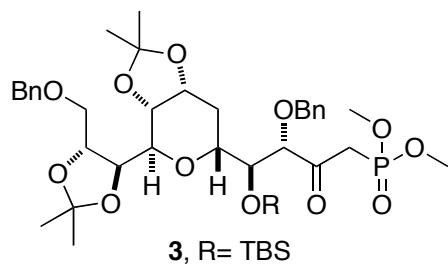
^{13}C NMR (100 MHz) of silyl ether **15** (in CDCl_3)



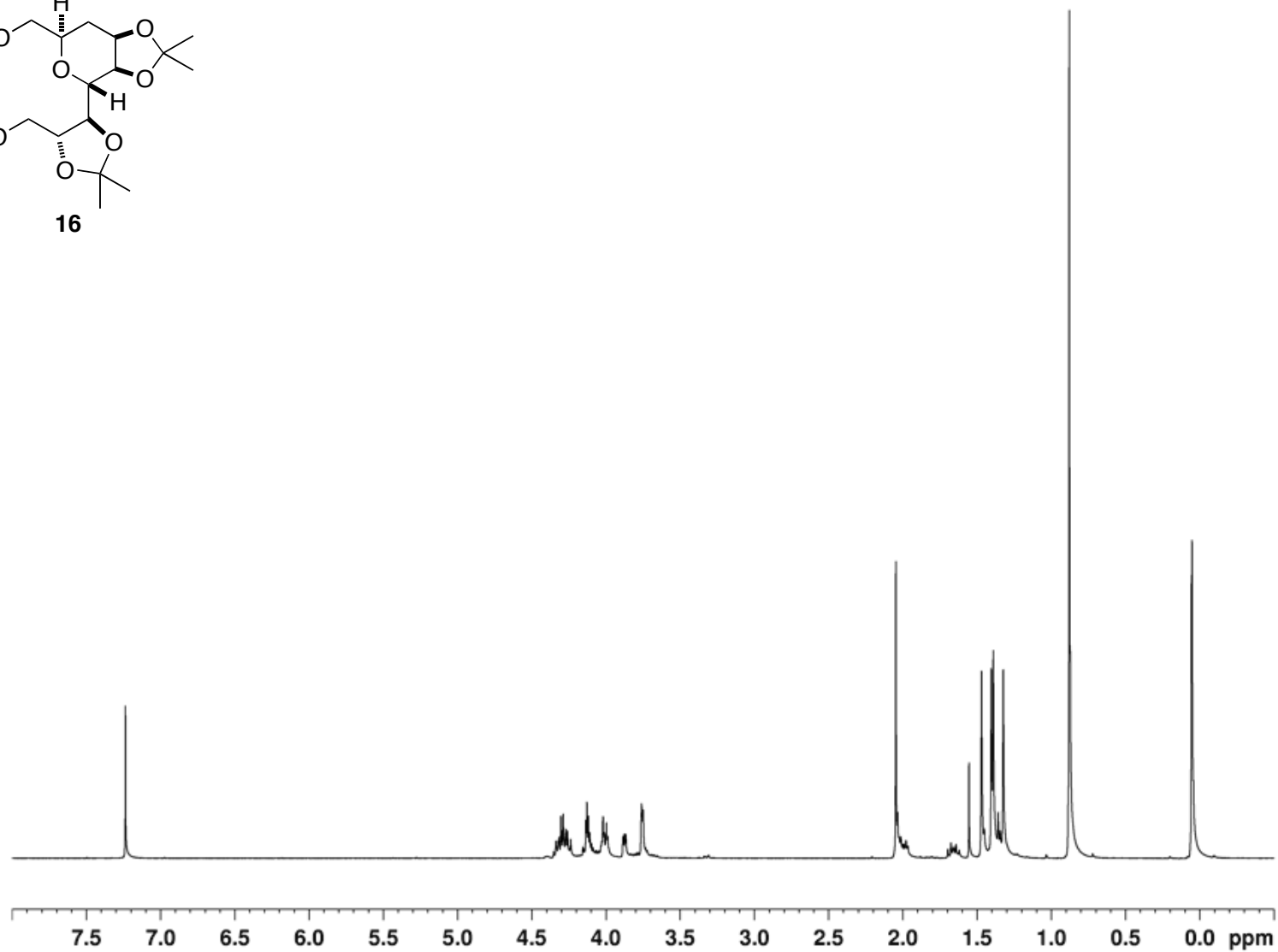
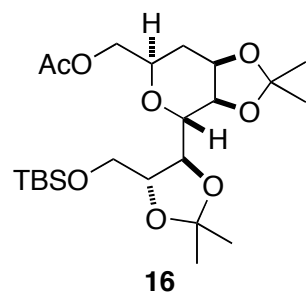
^1H NMR (400 MHz) of β -ketophosphonate **3** (in CDCl_3)



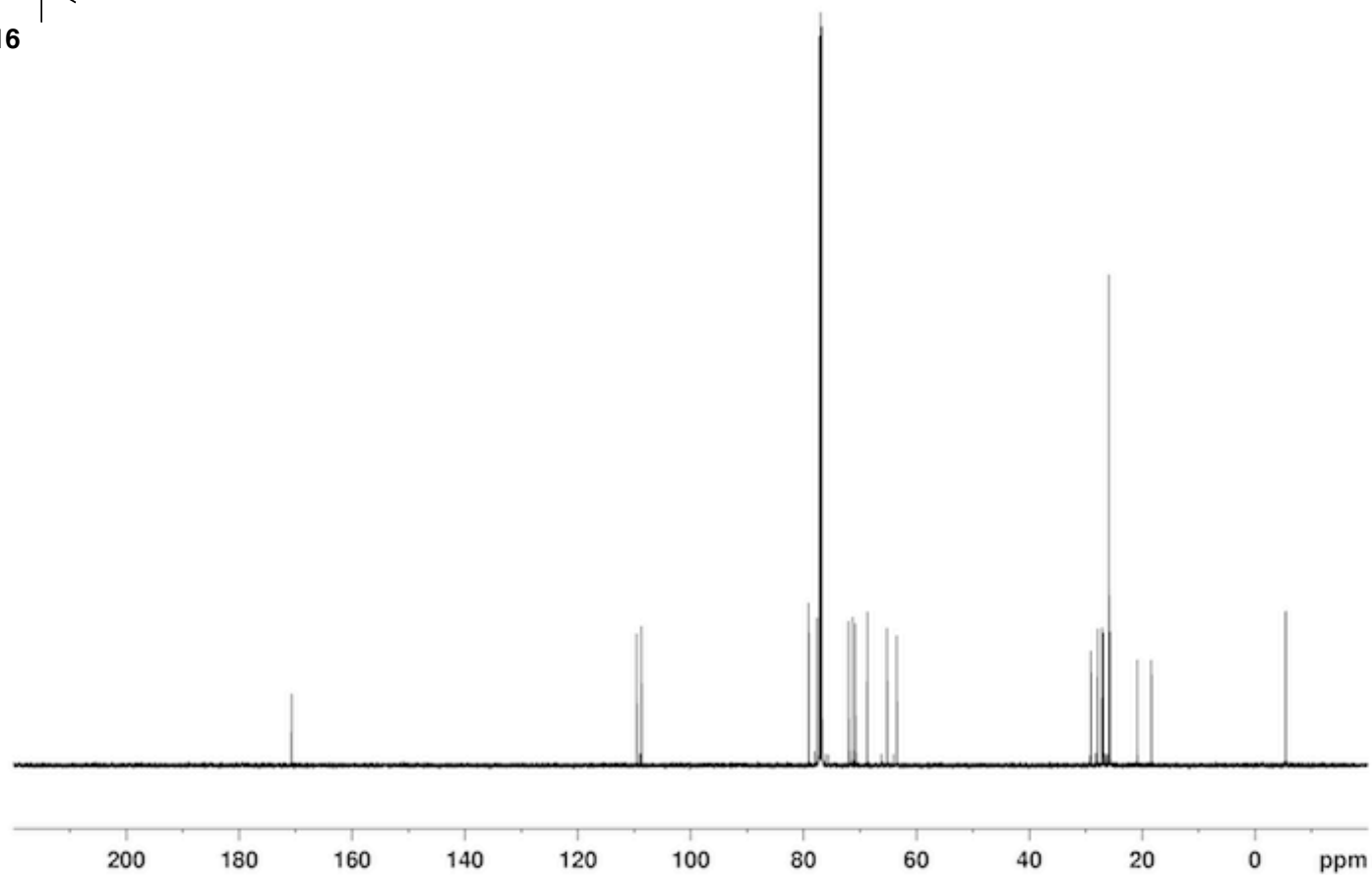
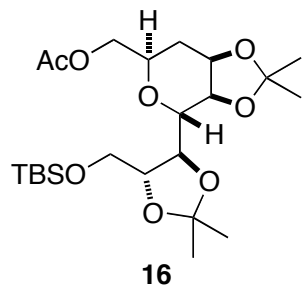
^{13}C NMR (125 MHz) of β -ketophosphonate **3** (in CDCl_3)



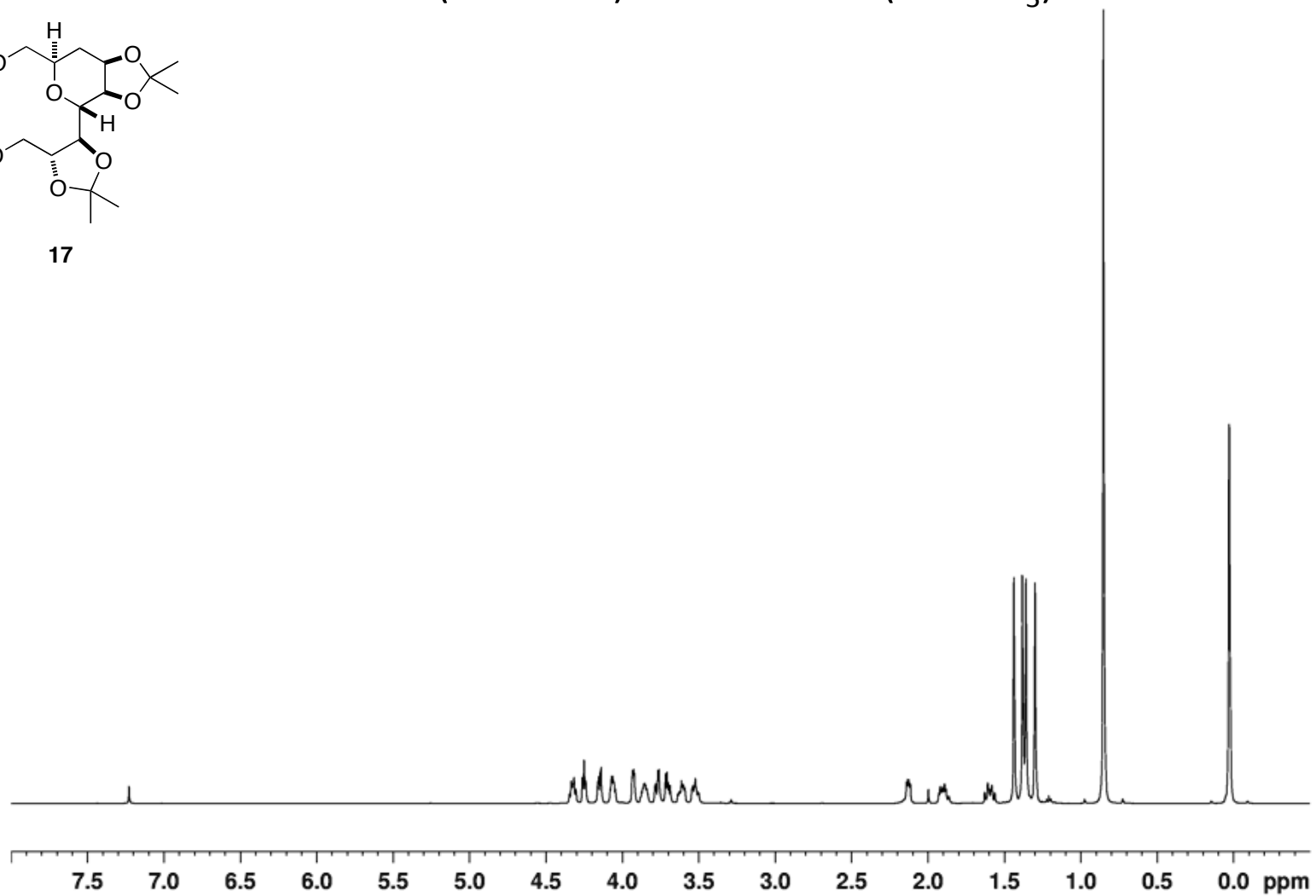
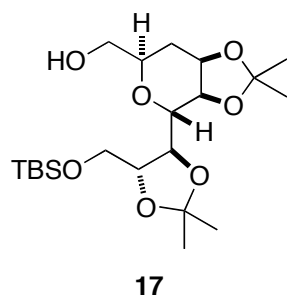
^1H NMR (400 MHz) of acetate **16** (in CDCl_3)



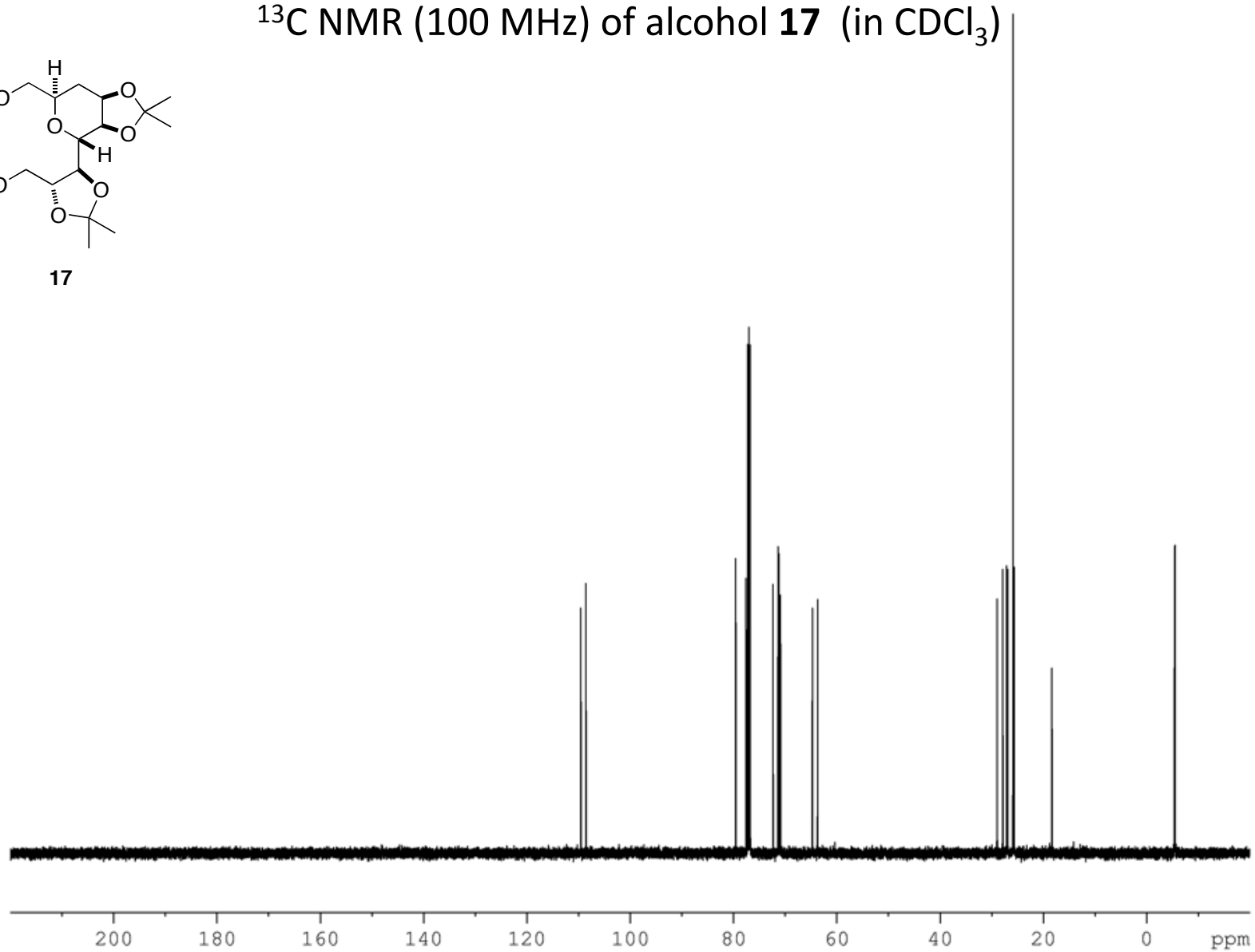
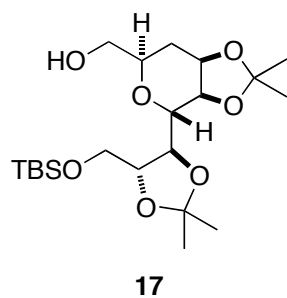
^{13}C NMR (500 MHz) of acetate **16** (in CDCl_3)



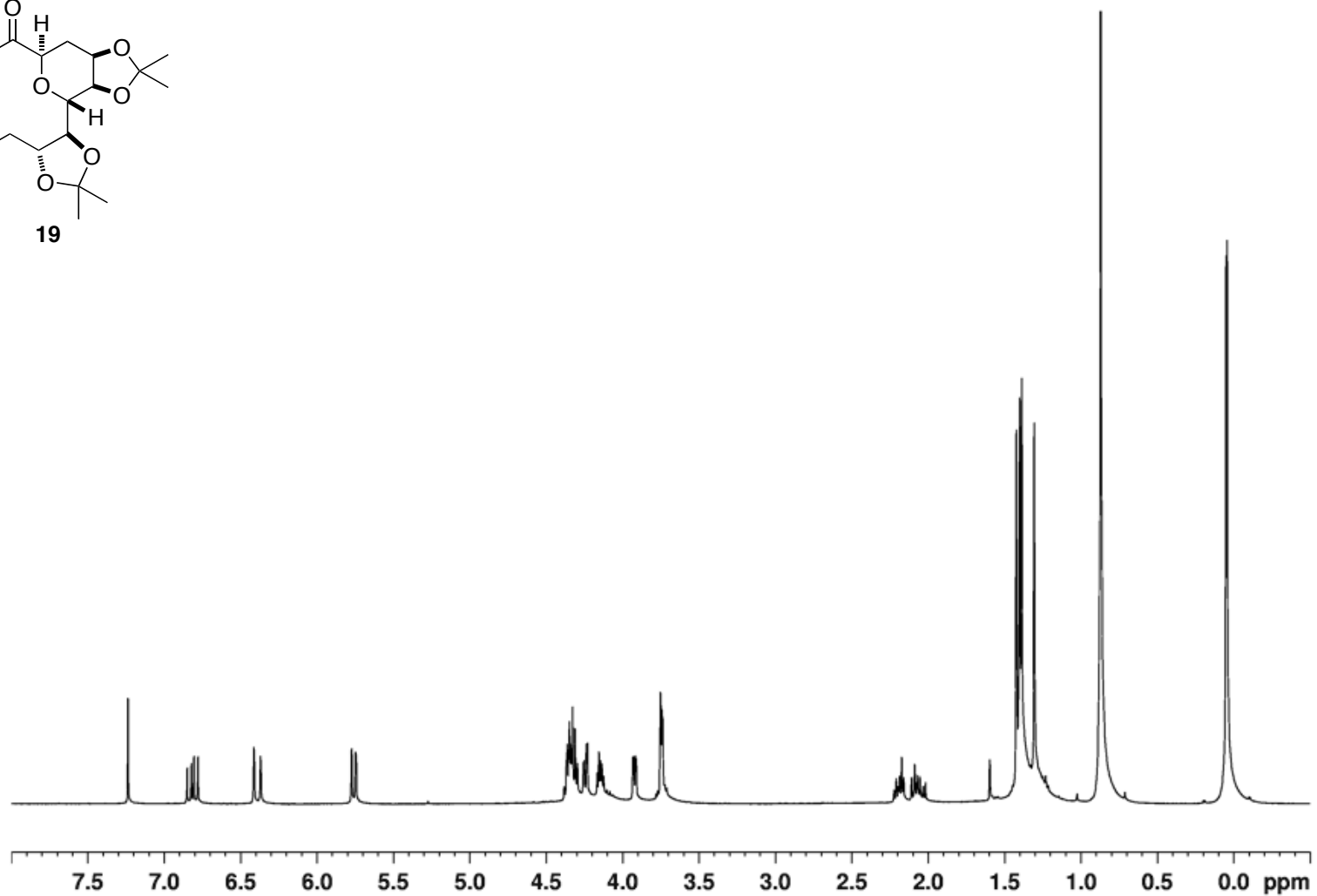
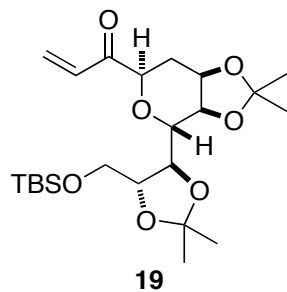
^1H NMR (400 MHz) of alcohol **17** (in CDCl_3)



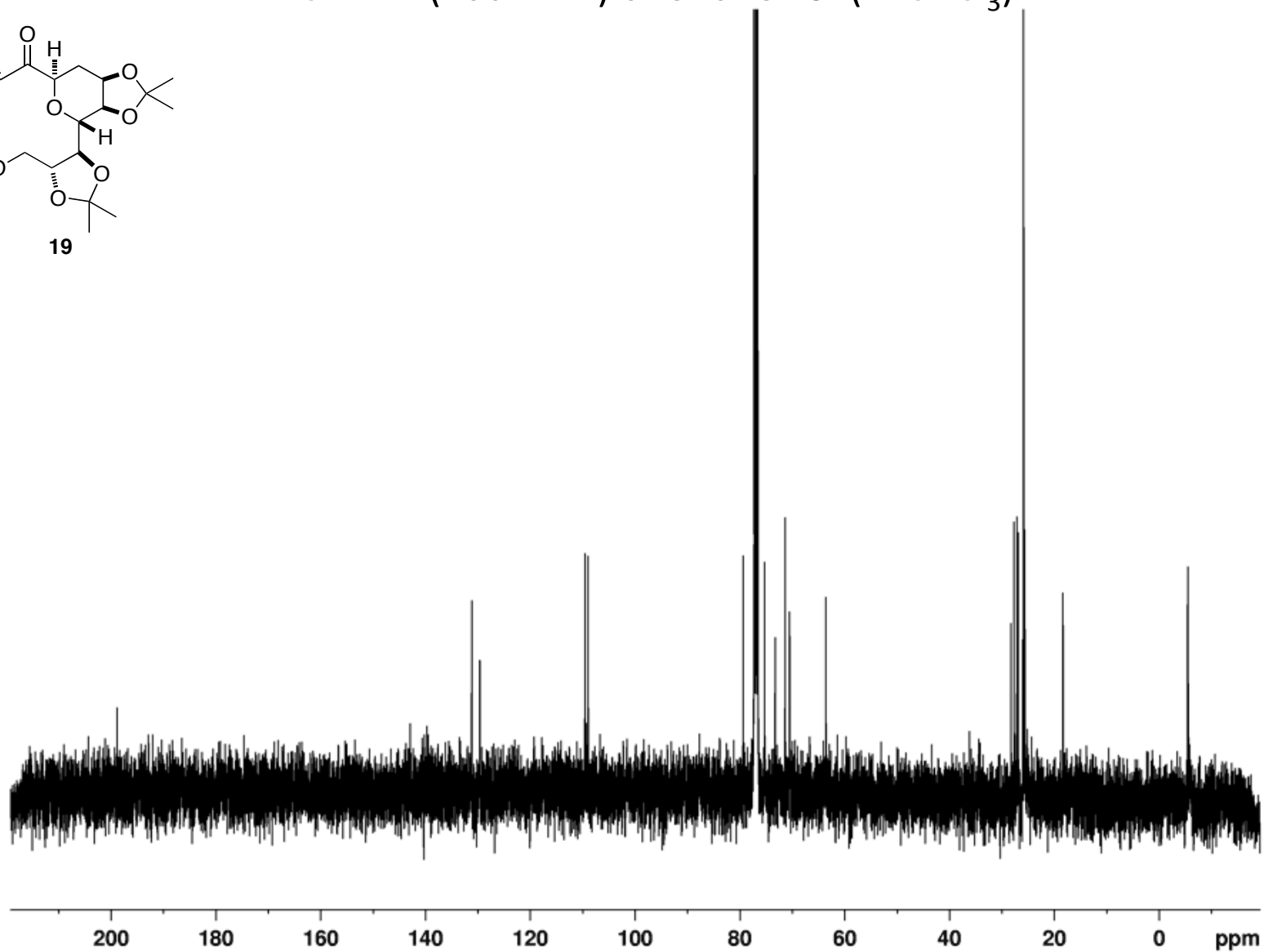
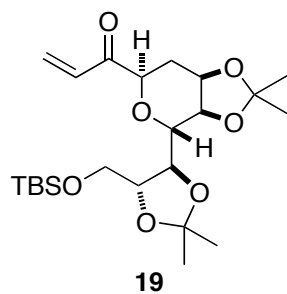
^{13}C NMR (100 MHz) of alcohol **17** (in CDCl_3)



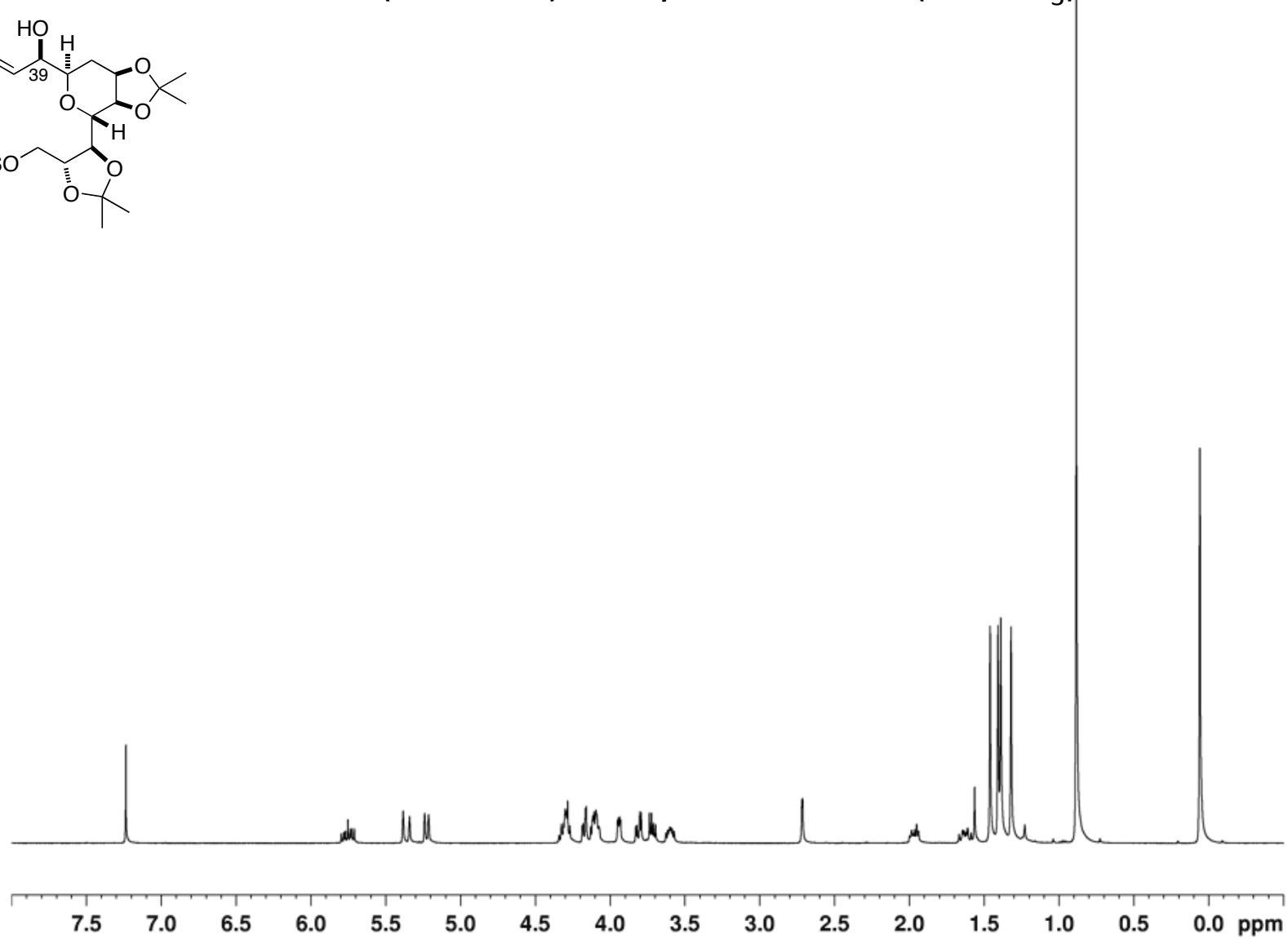
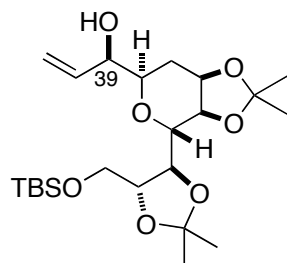
^1H NMR (400 MHz) of enone **19** (in CDCl_3)



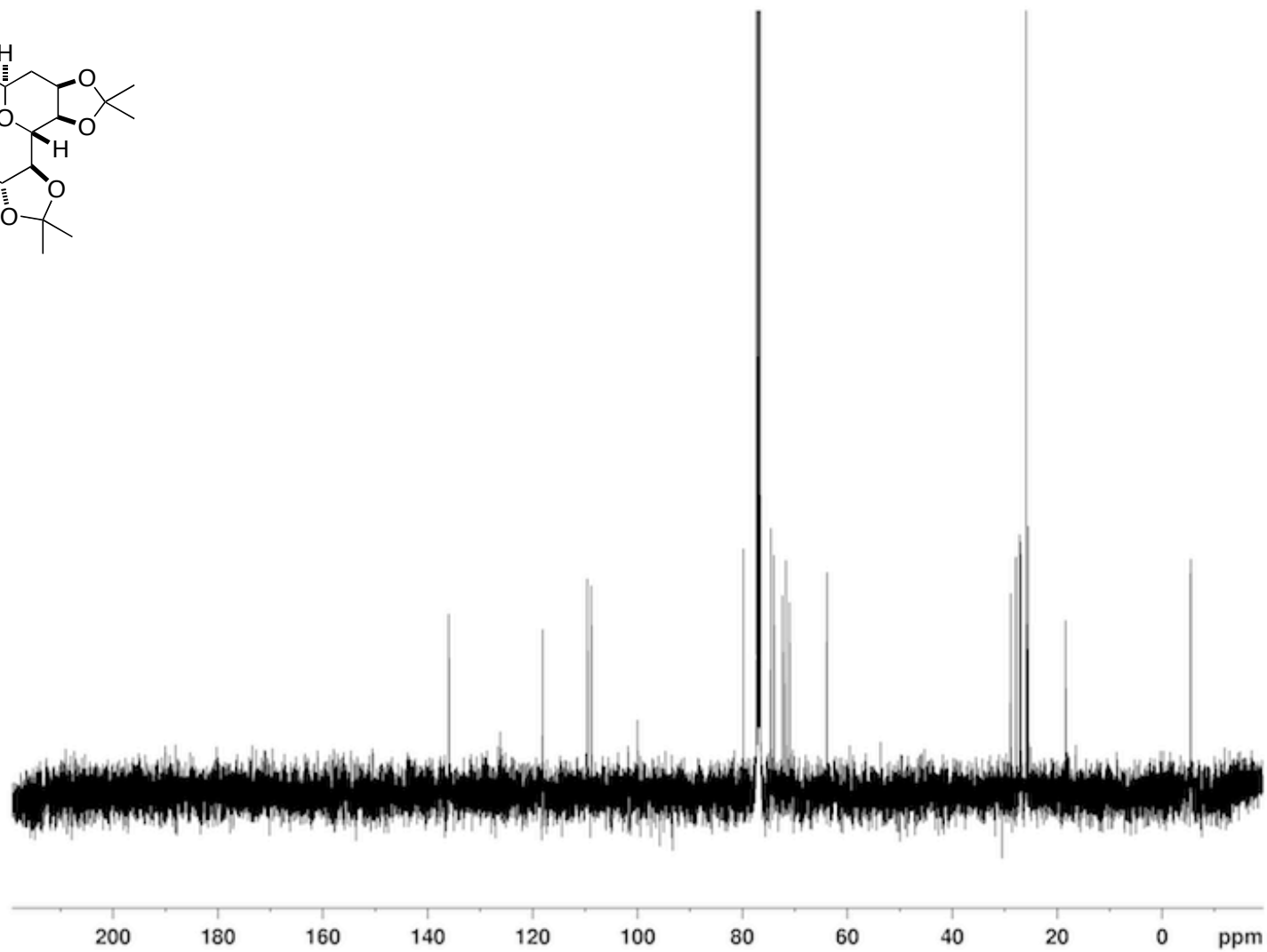
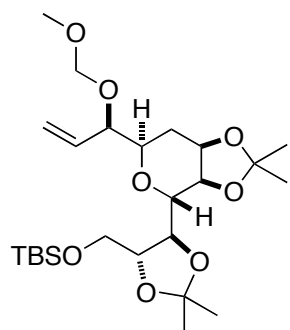
^{13}C NMR (100 MHz) of enone **19** (in CDCl_3)



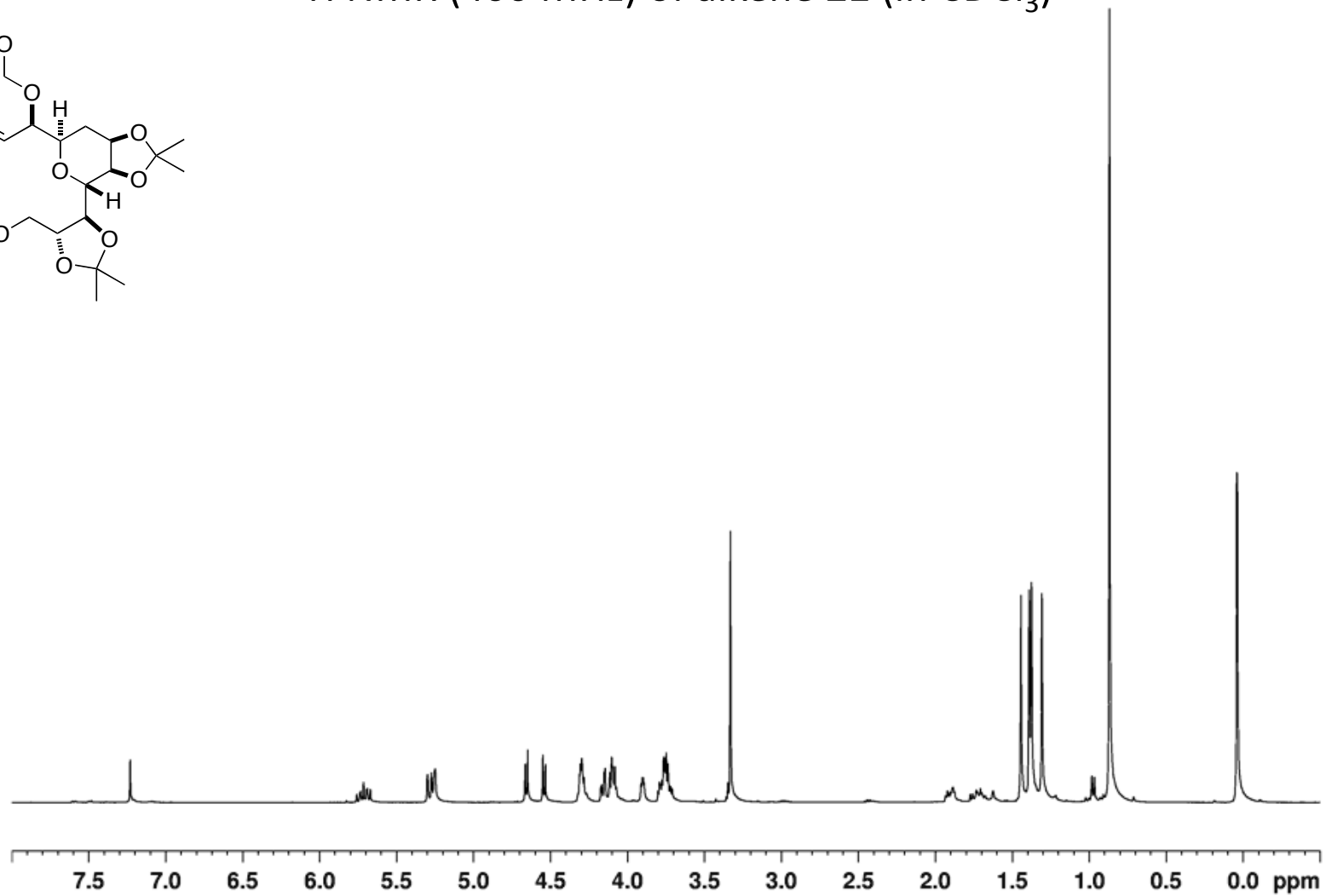
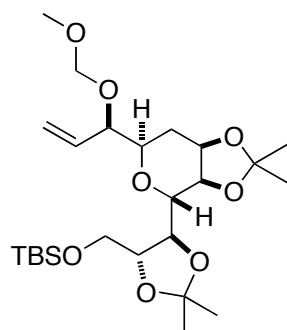
^1H NMR (400 MHz) of allylic alcohol **20** (in CDCl_3)



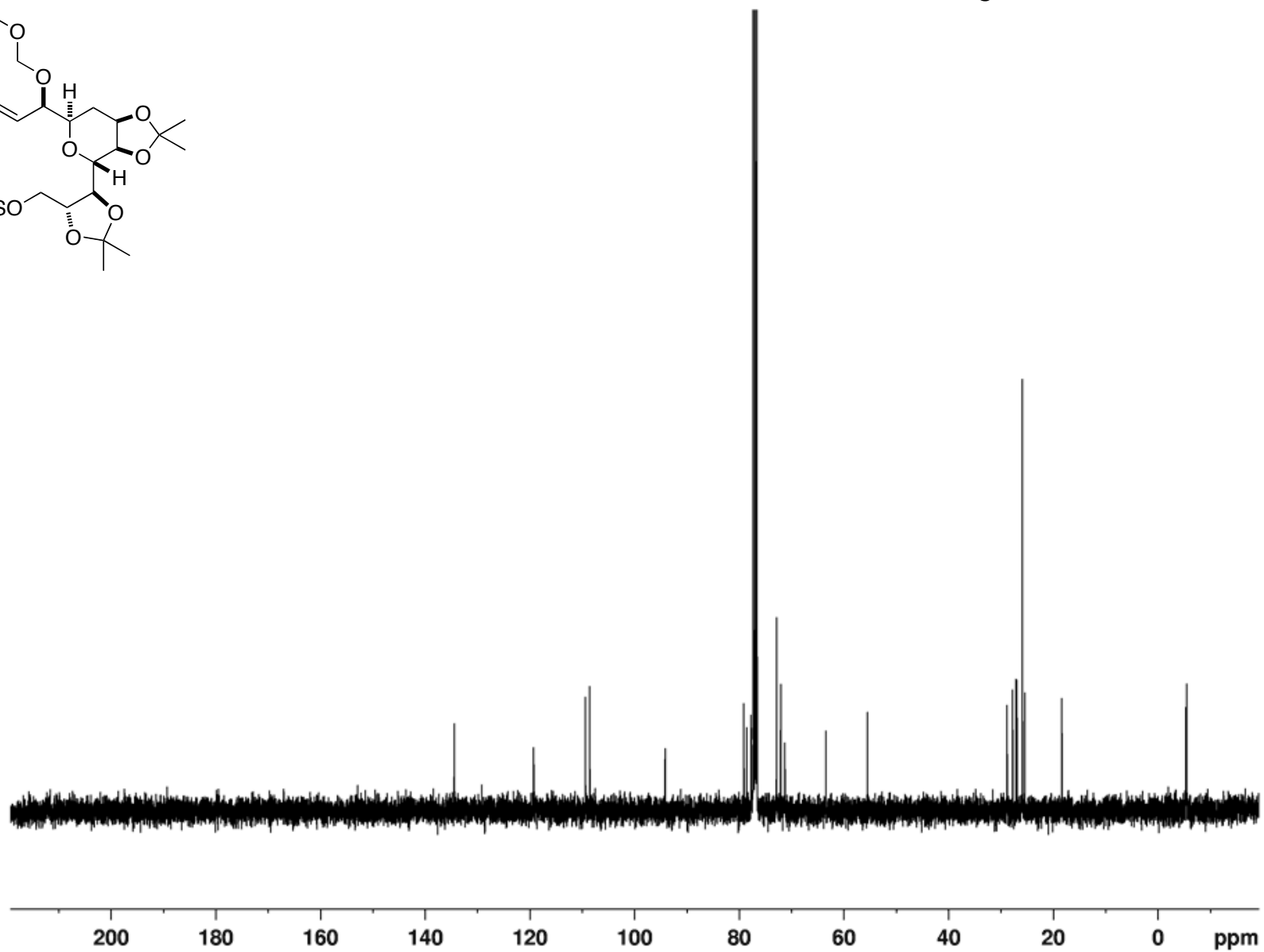
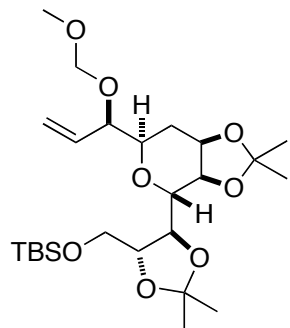
^{13}C NMR (100 MHz) of allylic alcohol **20** (in CDCl_3)



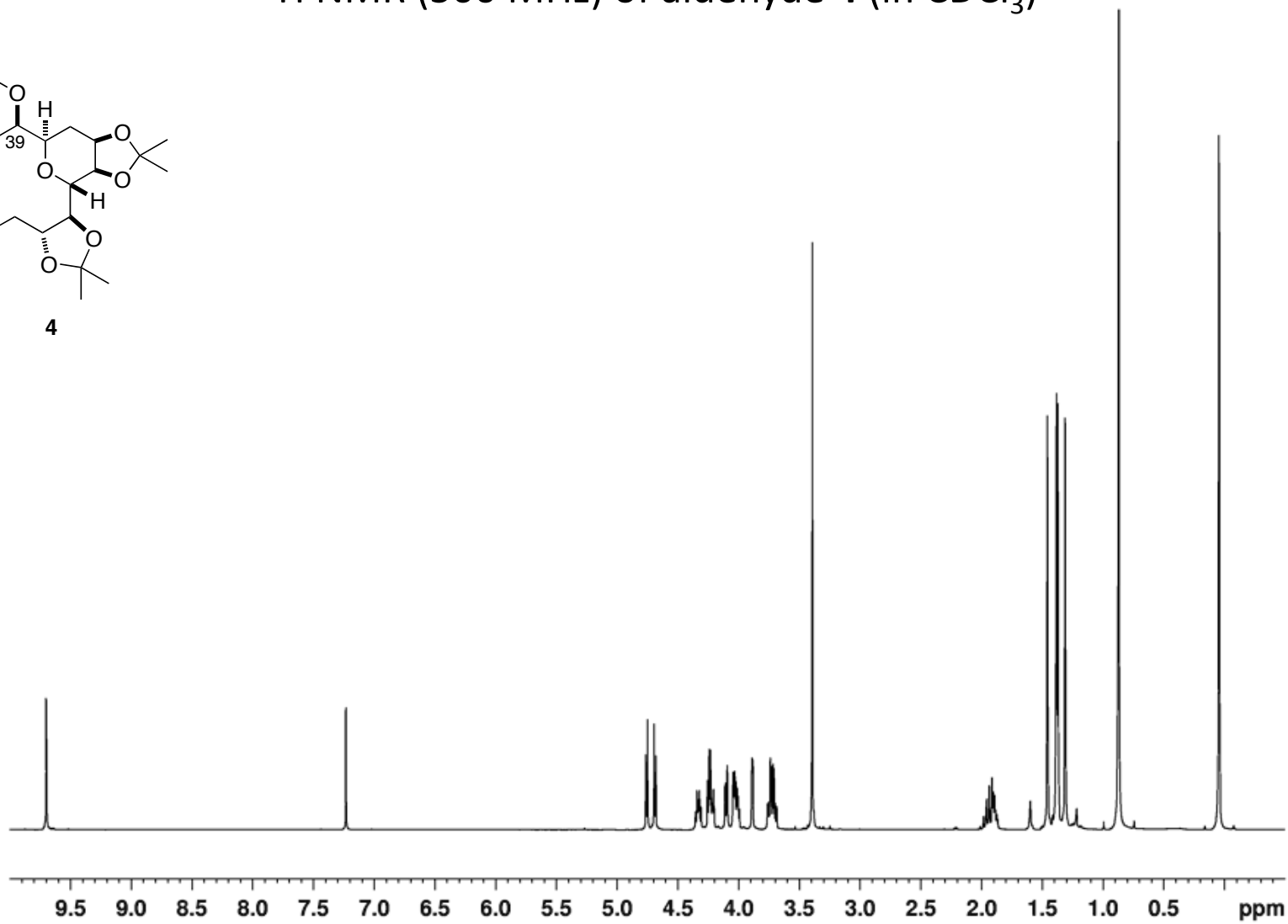
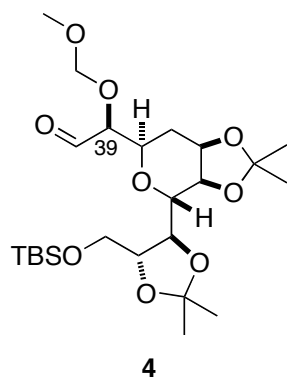
^1H NMR (400 MHz) of alkene **21** (in CDCl_3)



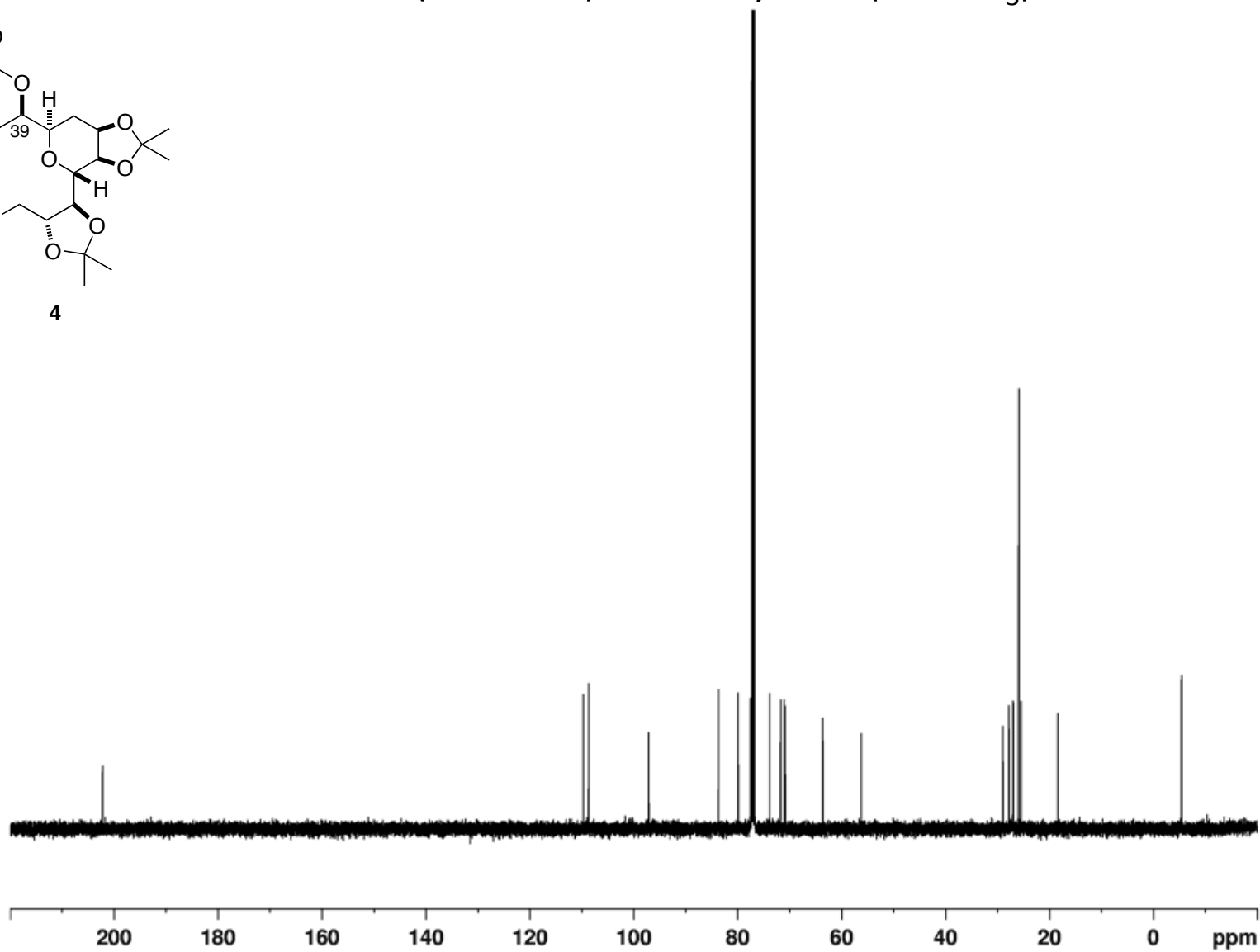
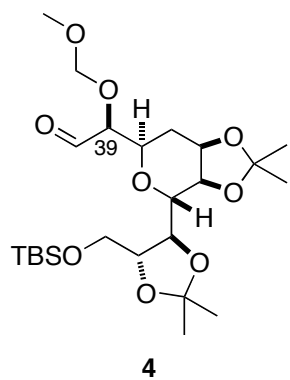
^{13}C NMR (100 MHz) of alkene **21** (in CDCl_3)



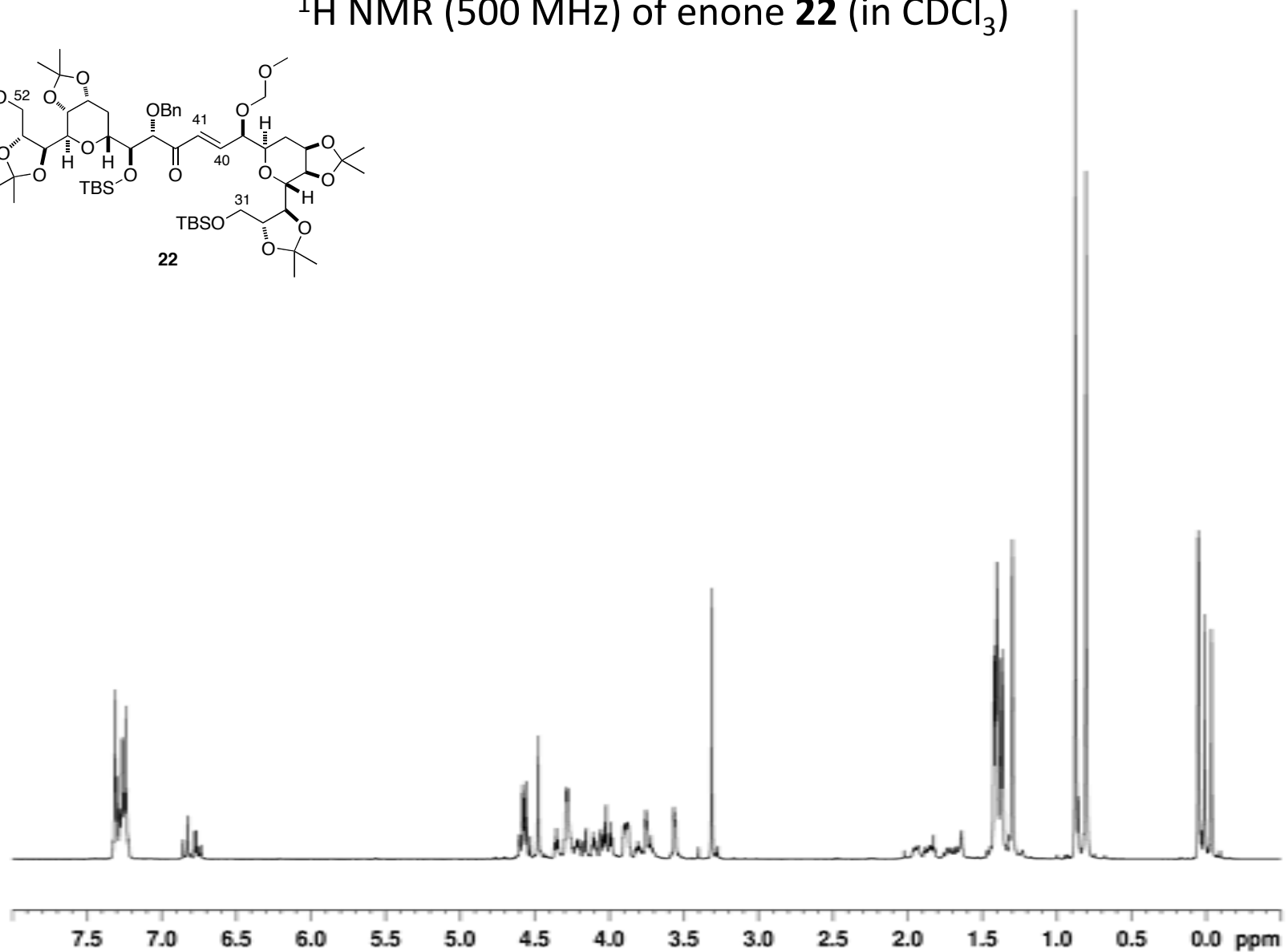
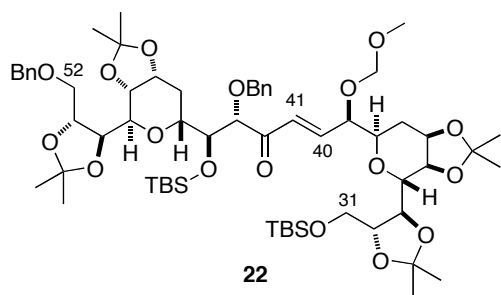
^1H NMR (500 MHz) of aldehyde **4** (in CDCl_3)



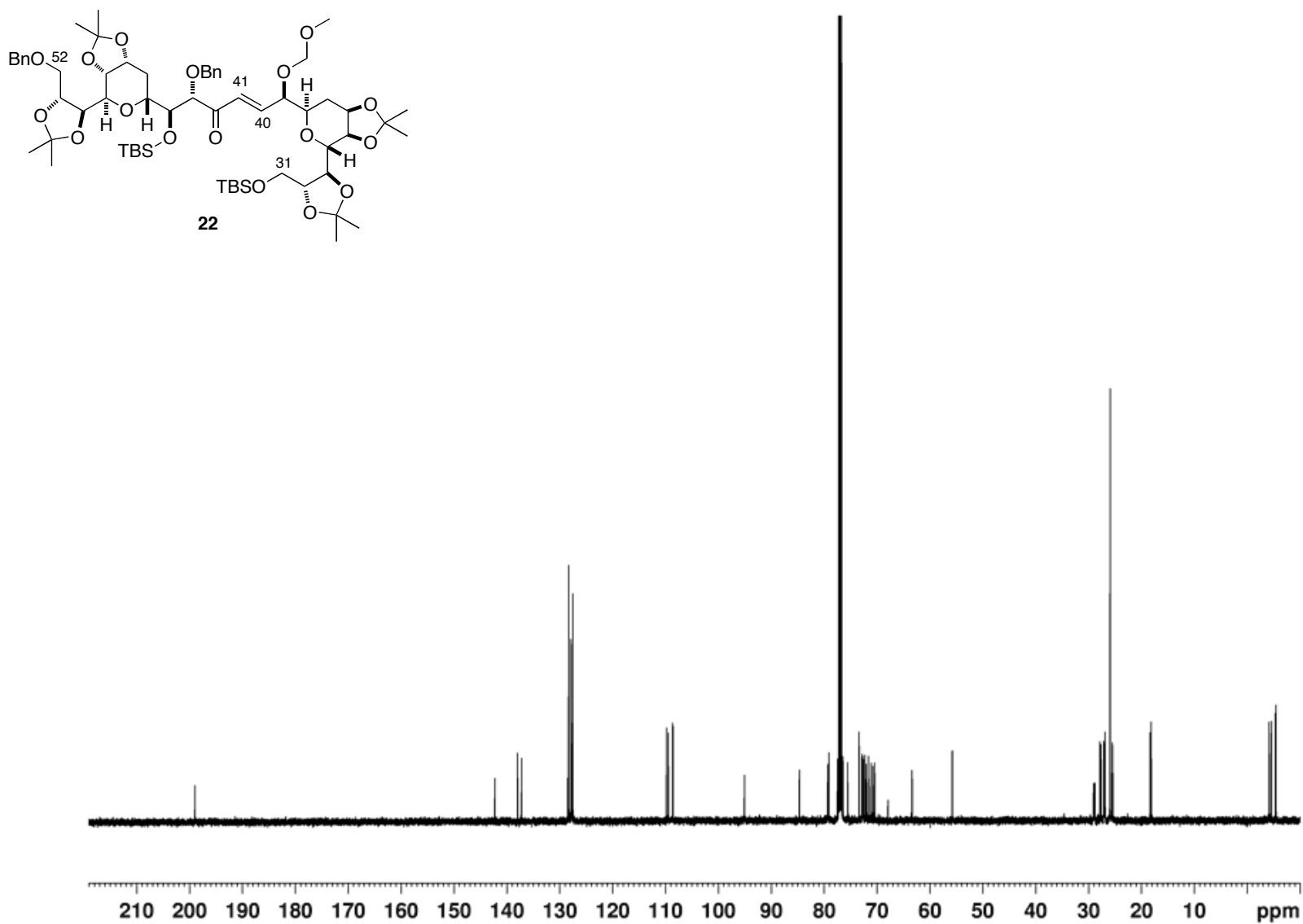
^{13}C NMR (125 MHz) of aldehyde **4** (in CDCl_3)



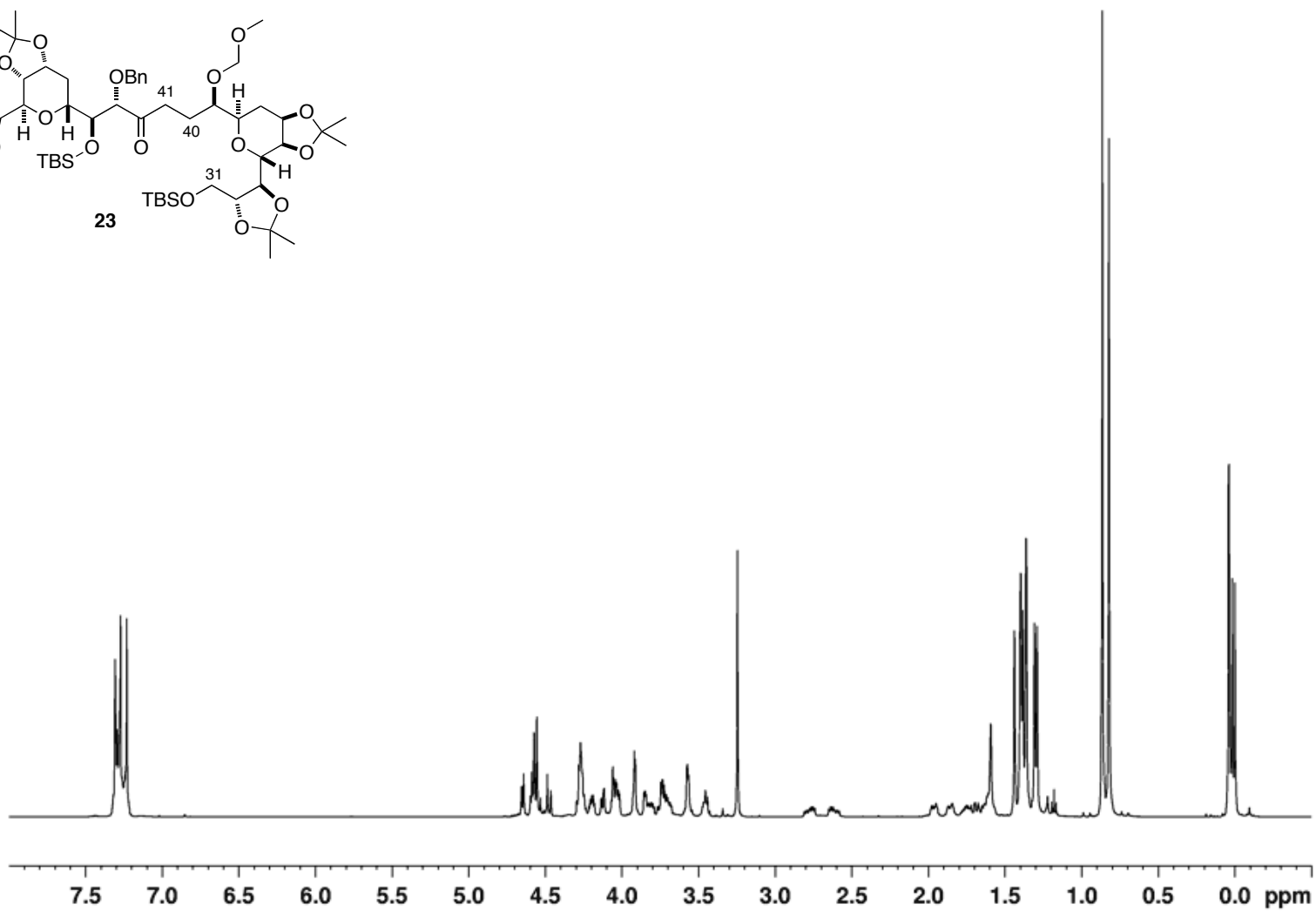
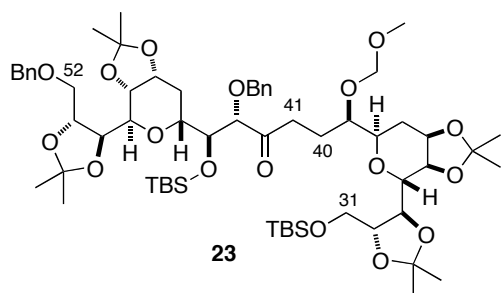
^1H NMR (500 MHz) of enone **22** (in CDCl_3)



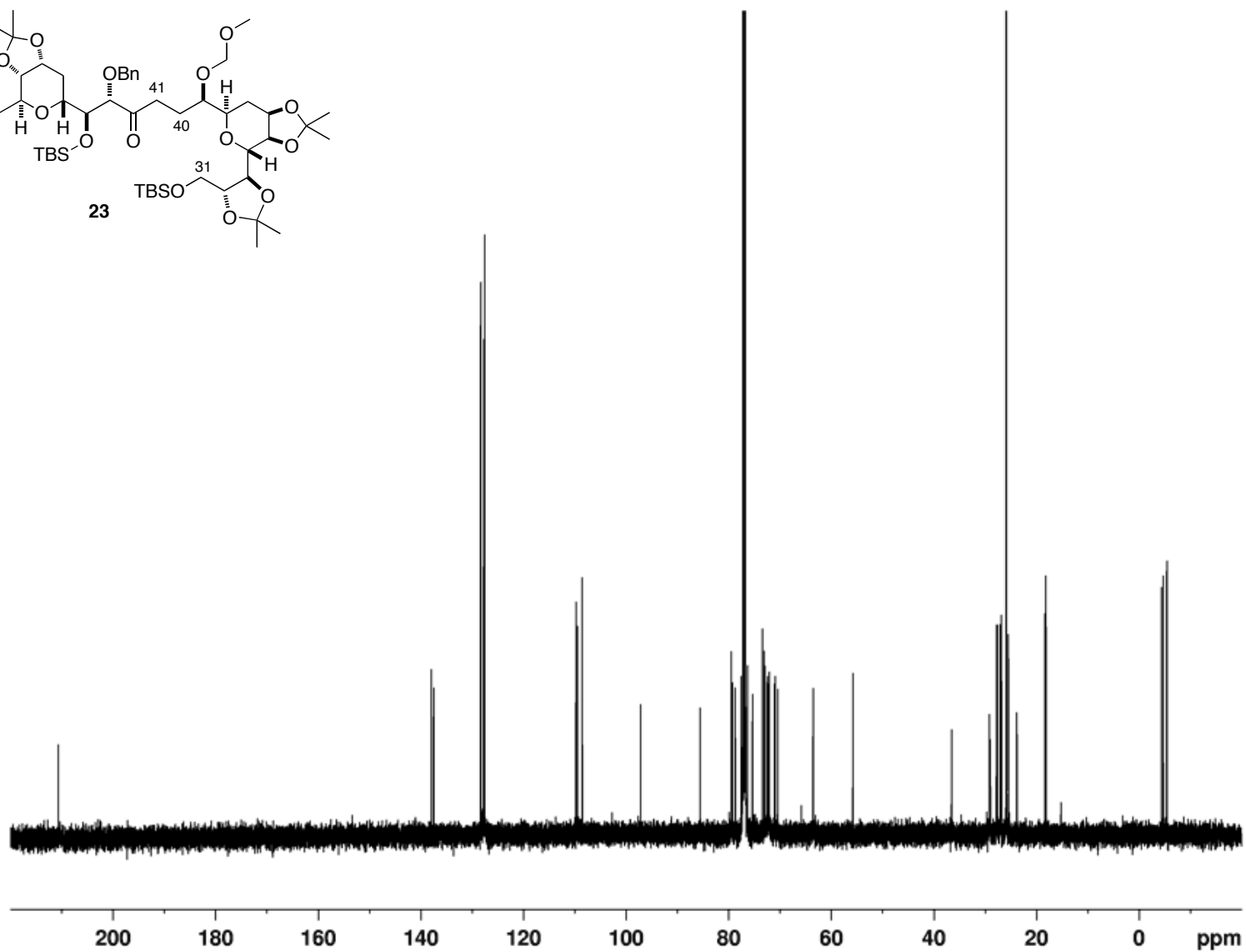
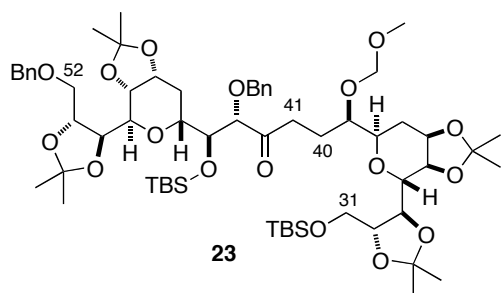
^{13}C NMR (125 MHz) of enone **22** (in CDCl_3)



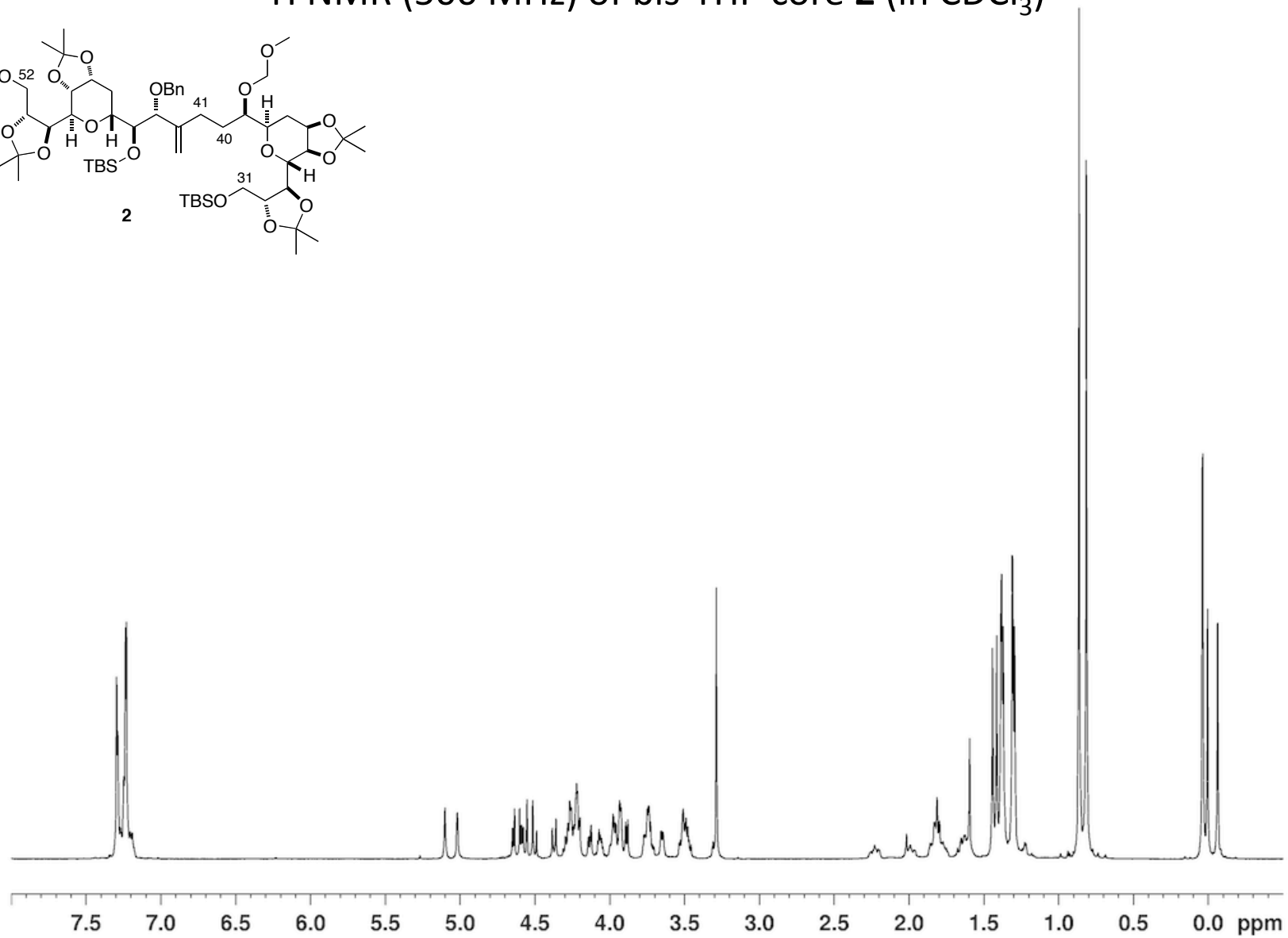
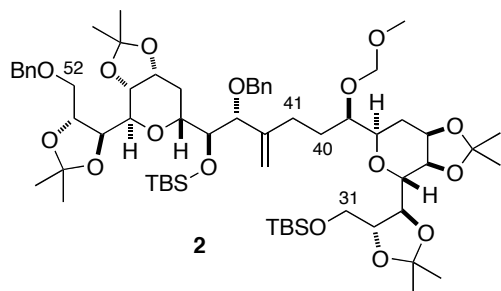
^1H NMR (500 MHz) of ketone **23** (in CDCl_3)



^{13}C NMR (125 MHz) of ketone **23** (in CDCl_3)



^1H NMR (500 MHz) of bis-THP core **2** (in CDCl_3)



^{13}C NMR (125 MHz) of bis-THP core **2** (in CDCl_3)

