

Synthesis and Reactivity of Alkyl-1,1,1- Trisphosphonate Esters

Jacqueline P. Smits and David F. Wiemer*

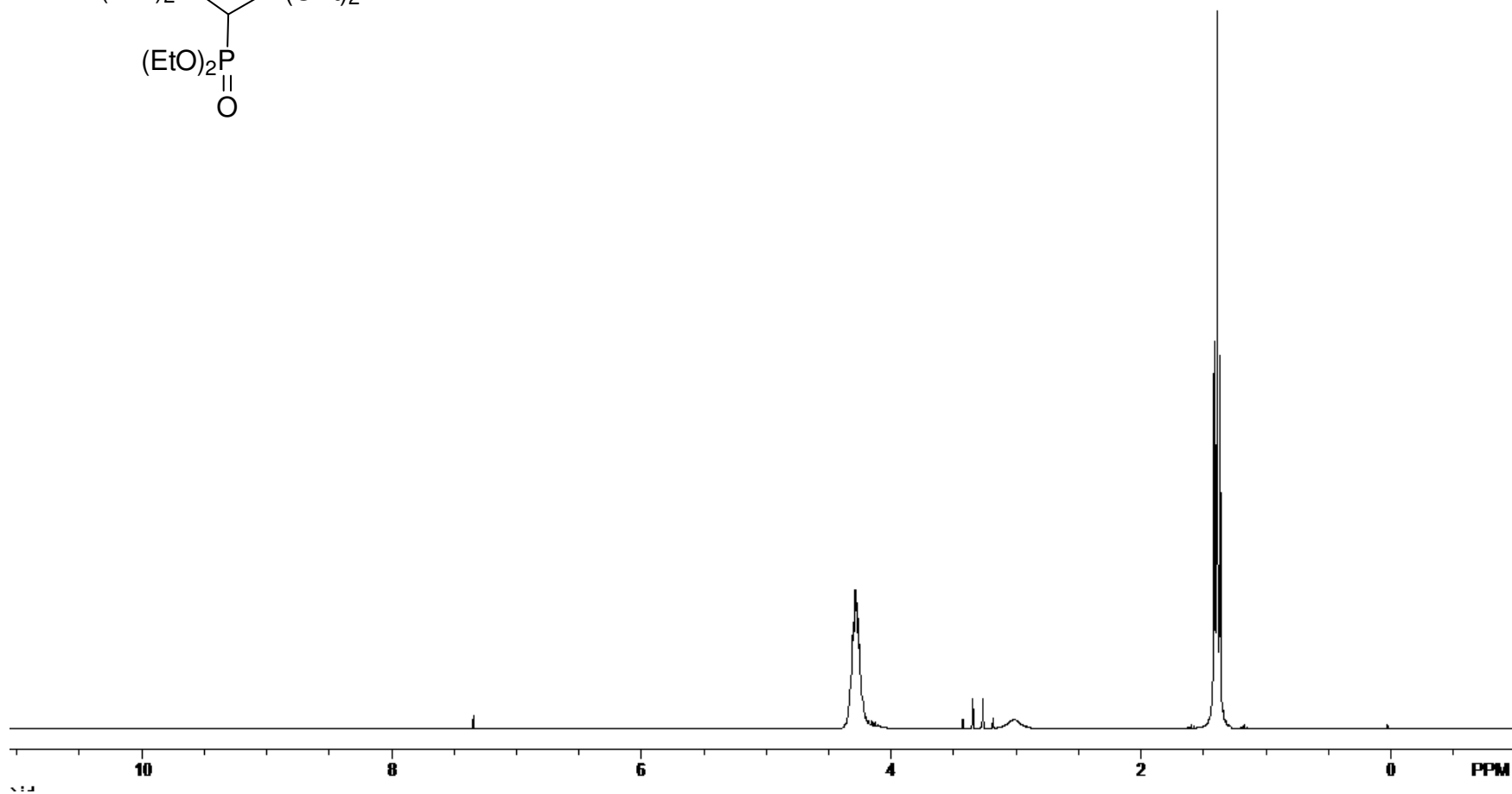
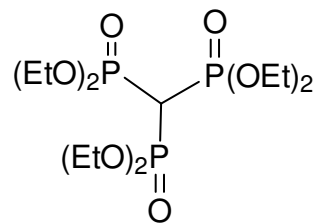
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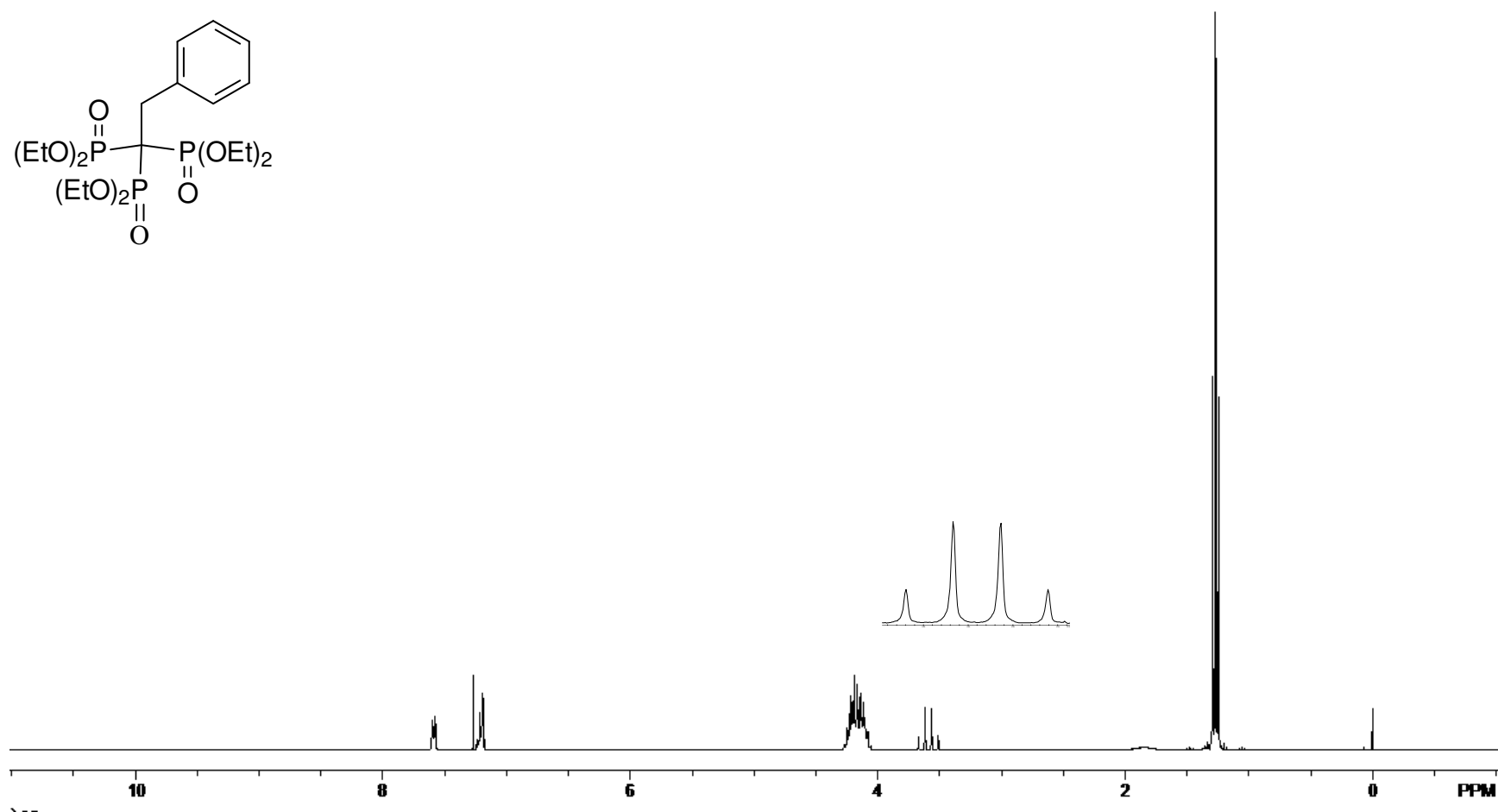
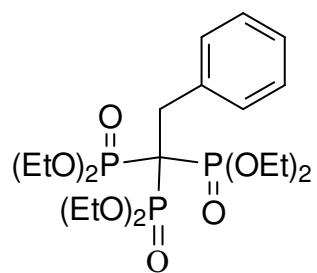
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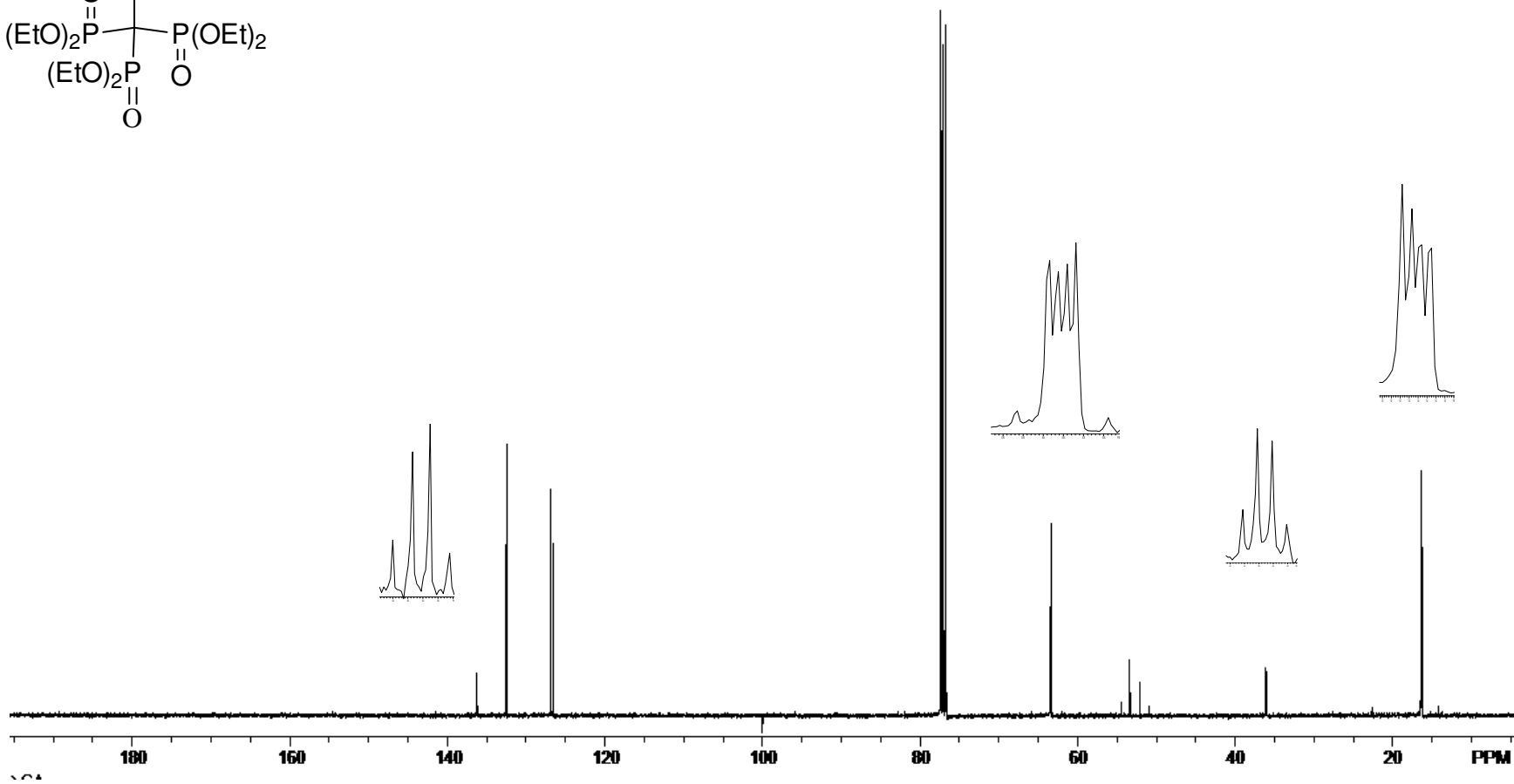
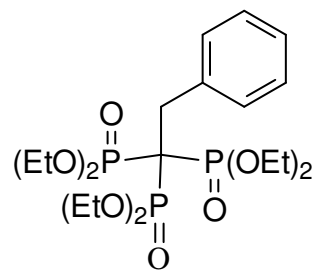
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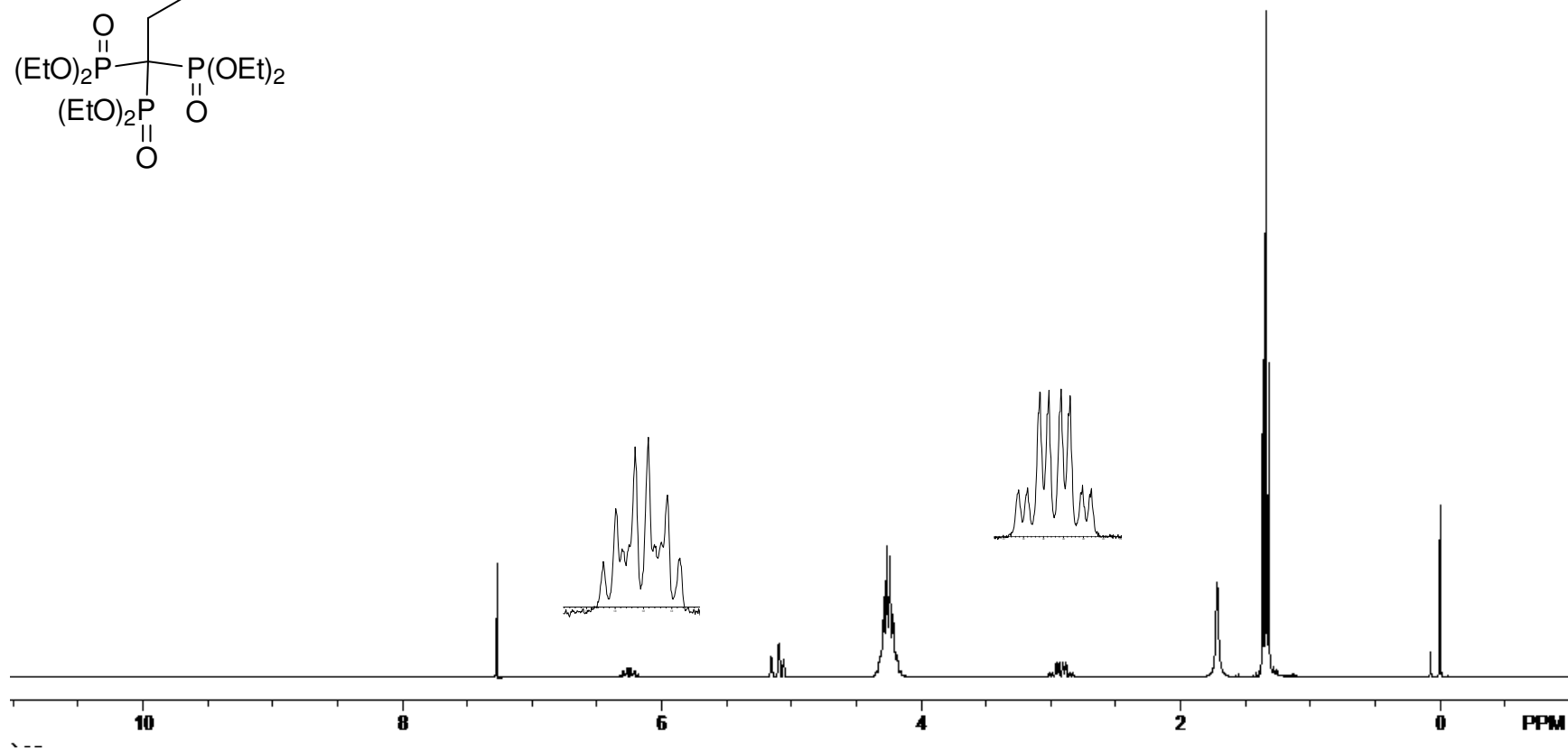
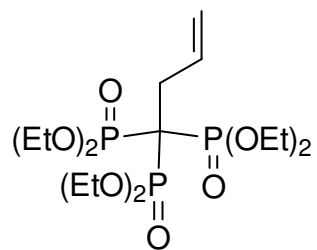
300 MHz ^1H NMR spectrum of methylidynetrisphosphonic acid, hexaethyl ester (**7**). The resonances at 3.31 ppm and at 3.01 ppm are exchanged upon addition of D_2O .



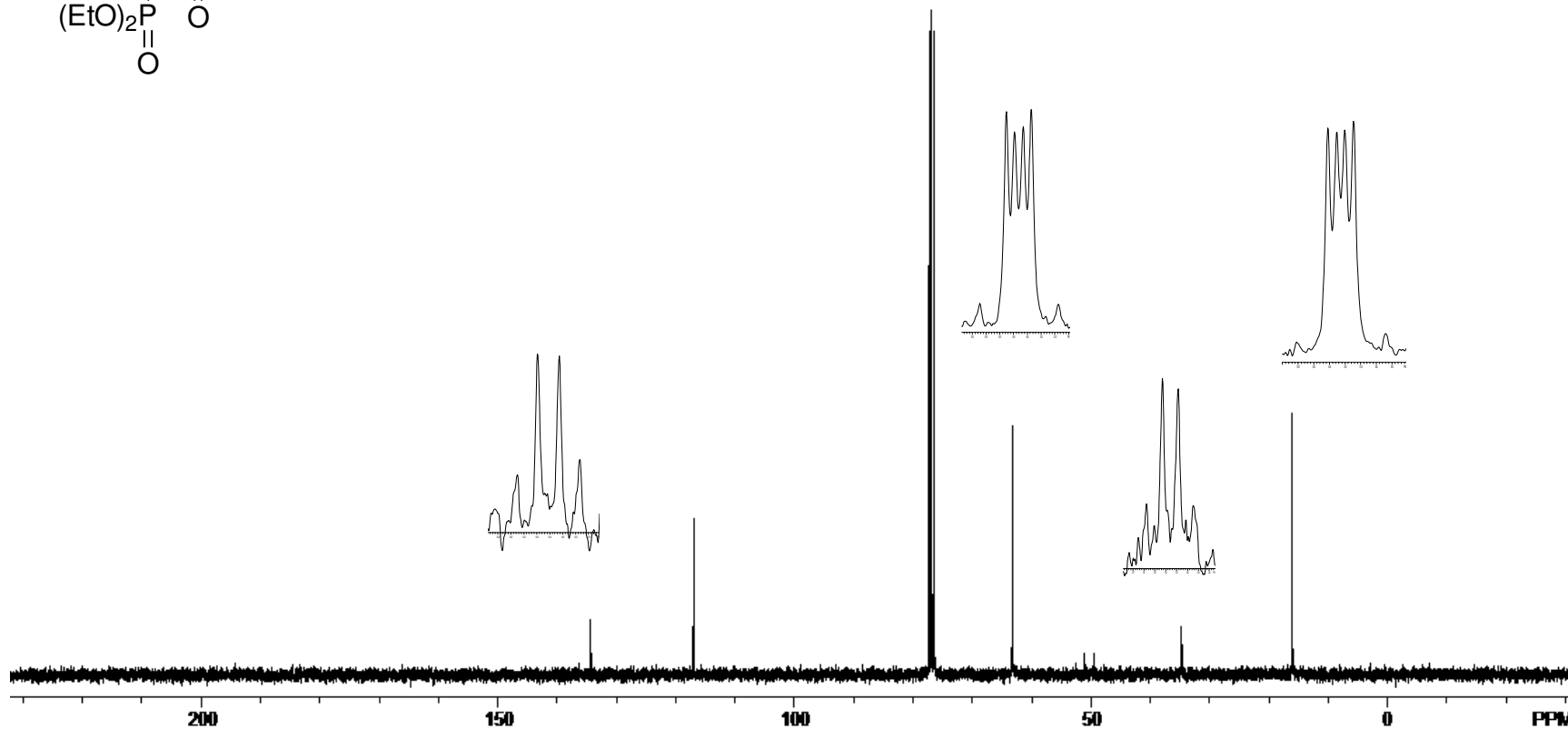
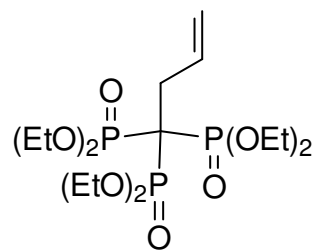
300 MHz ^1H NMR spectrum of 2-phenylethylidynetrisphosphonic acid, hexaethyl ester (**10**).



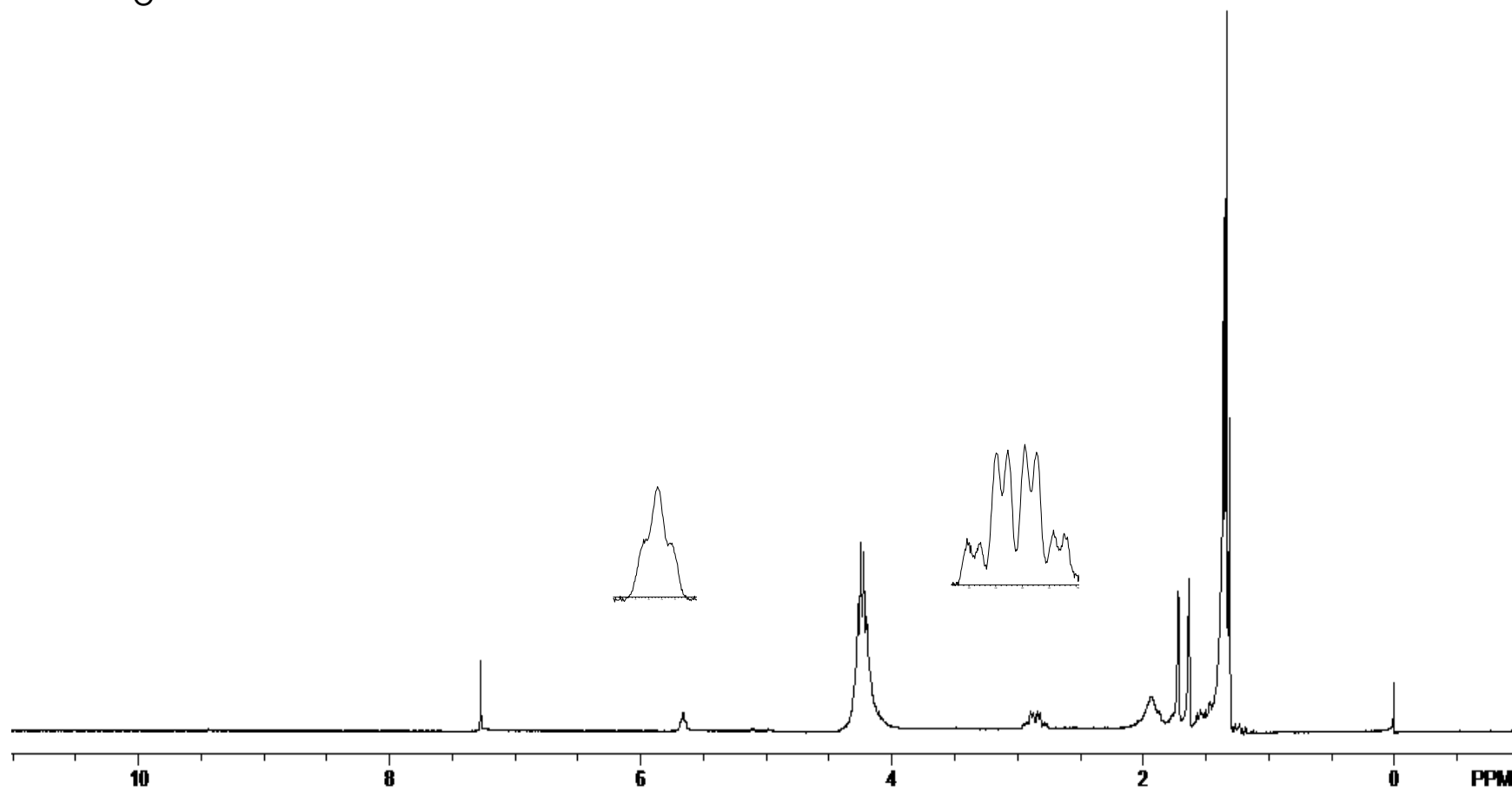
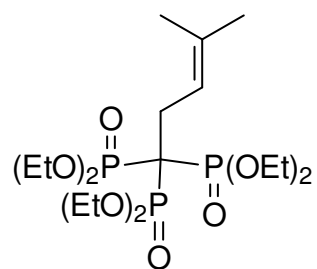
100 MHz ^{13}C NMR spectrum of 2-phenylethylidynetrisphosphonic acid, hexaethyl ester (**10**).



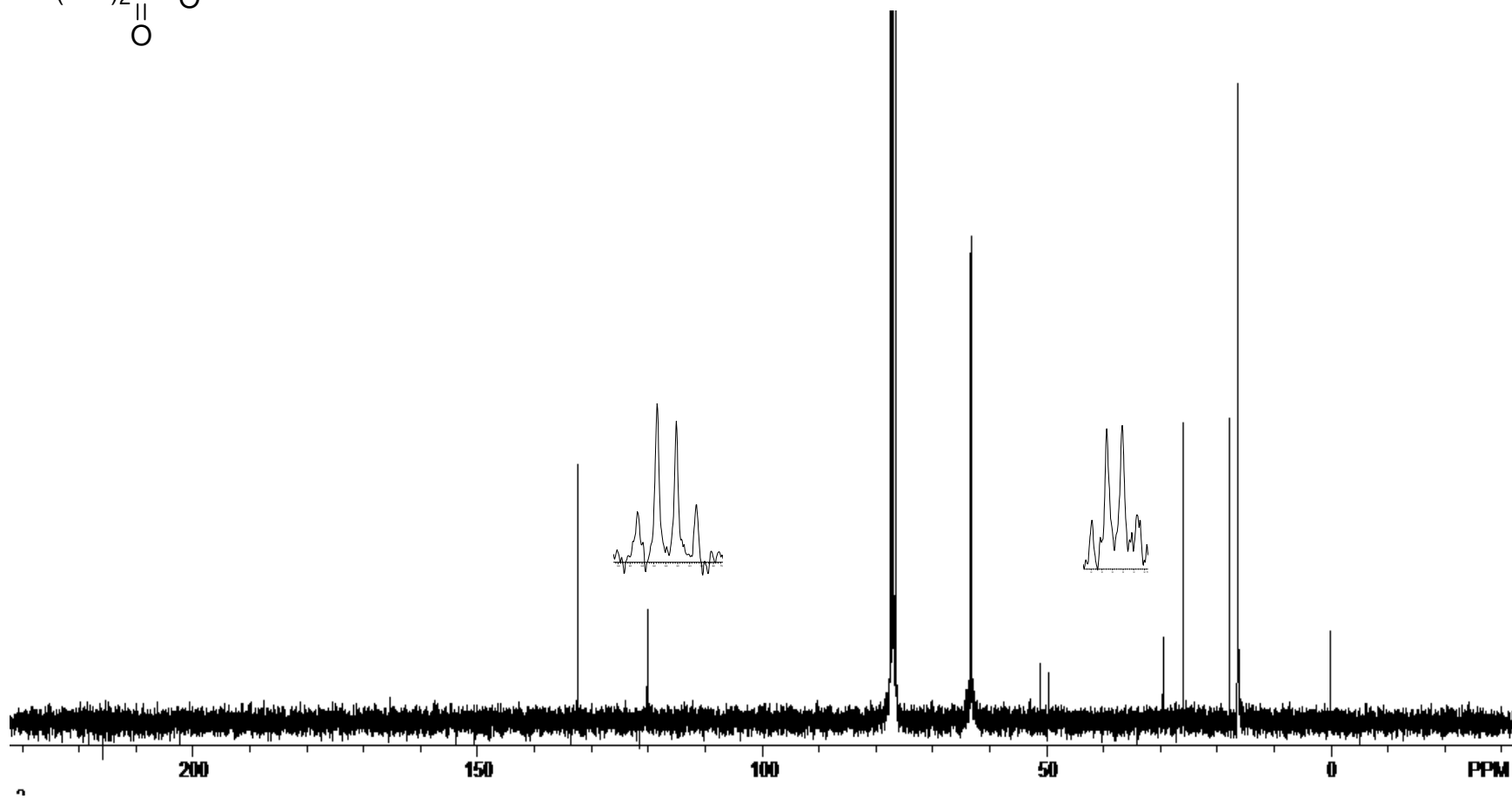
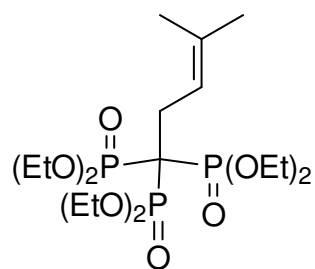
300 MHz ¹H NMR spectrum of 3-butenylidynetrisphosphonic acid, hexaethyl ester (**11**).



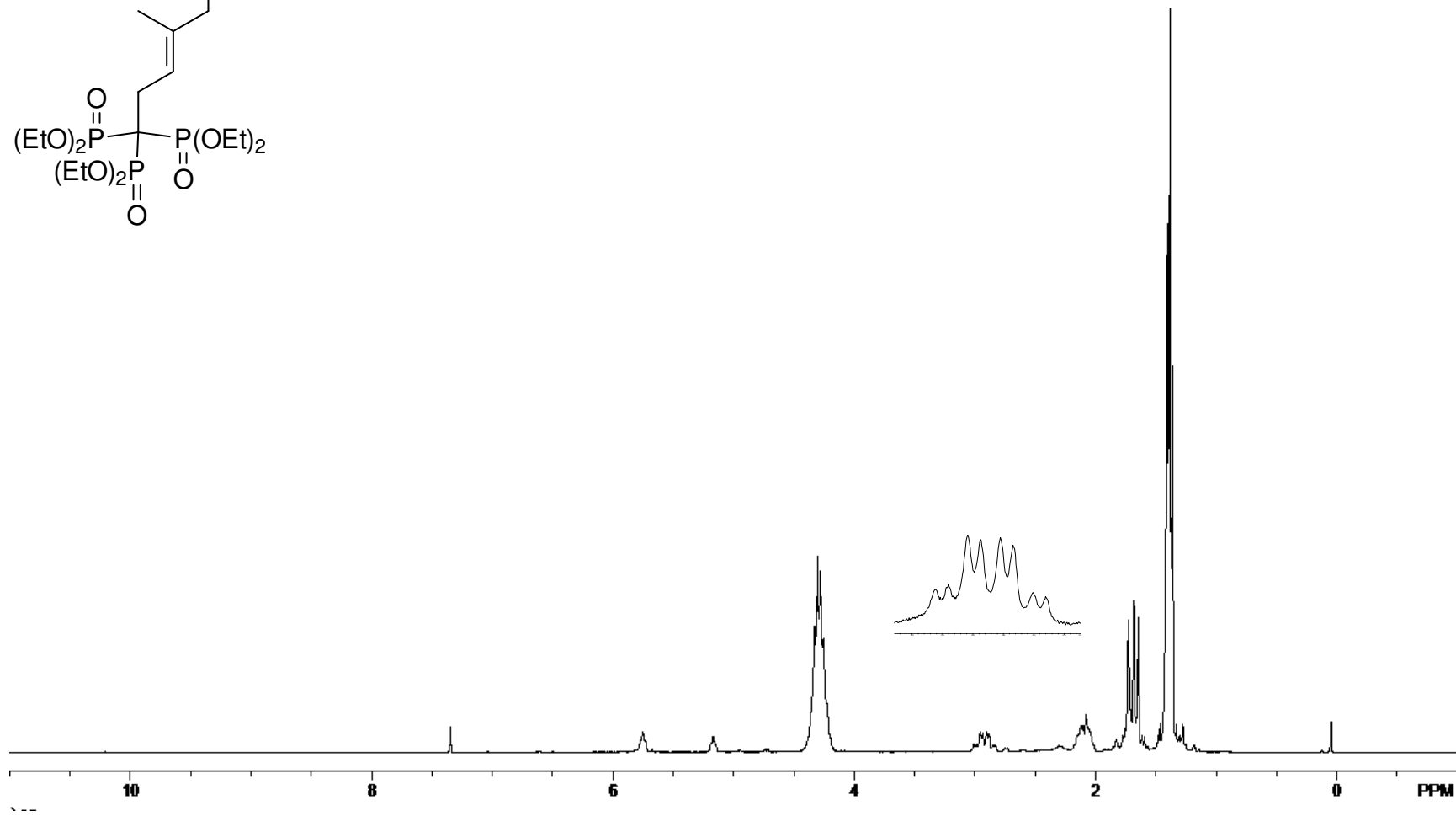
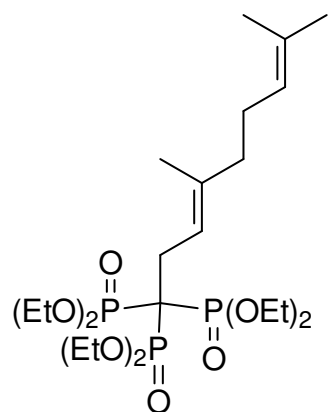
75 MHz ^{13}C NMR spectrum of 3-butenylidynetrisphosphonic acid, hexaethyl ester (**11**).



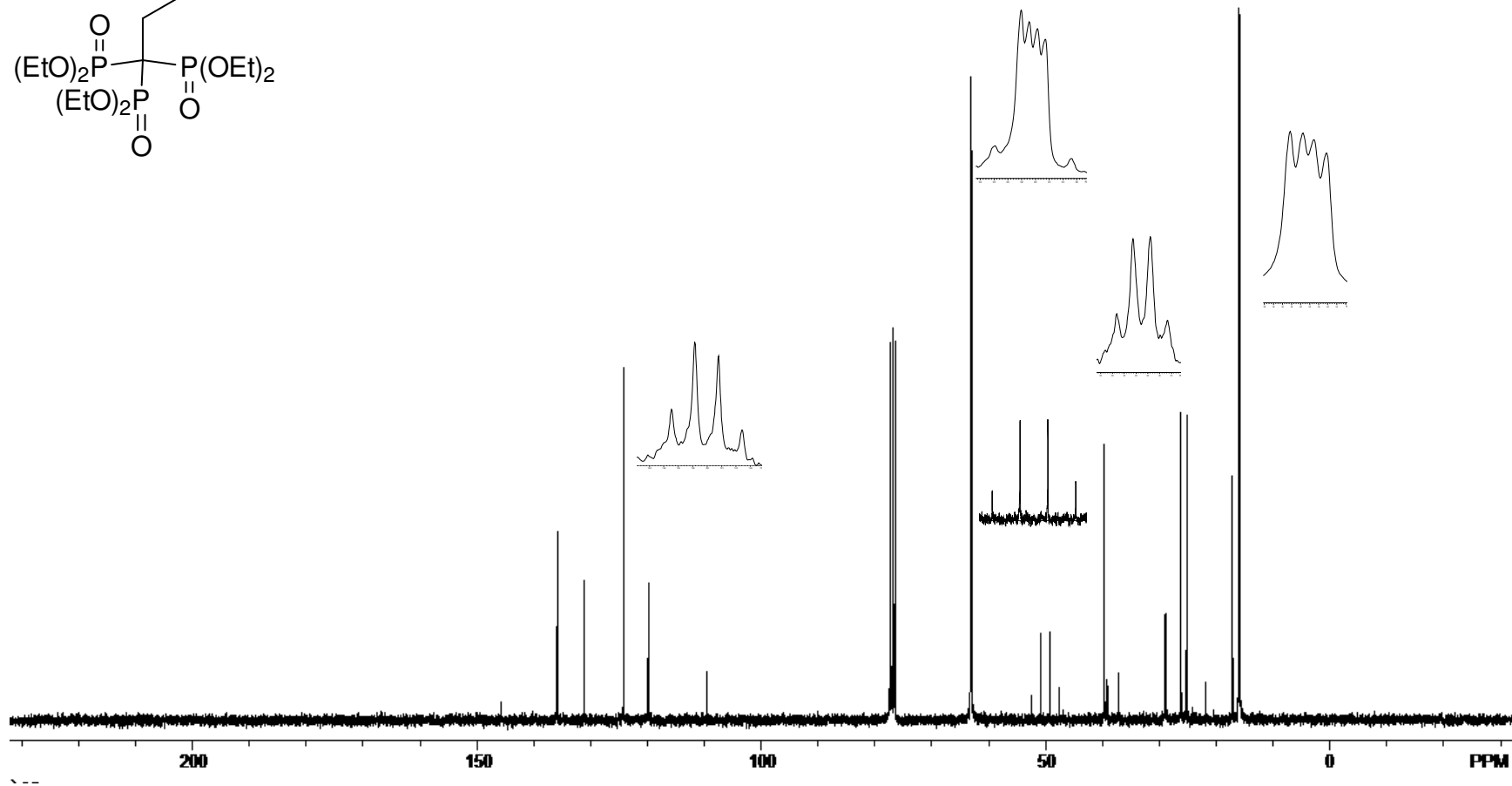
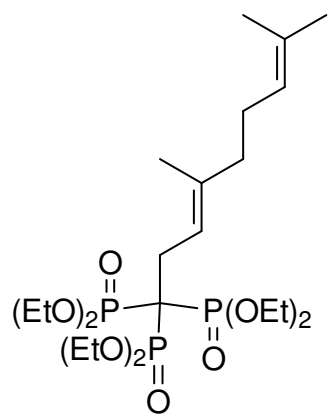
^1H NMR spectrum of 4-methyl-3-pentenylidynetrisphosphonic acid, hexaethyl ester (**12**).



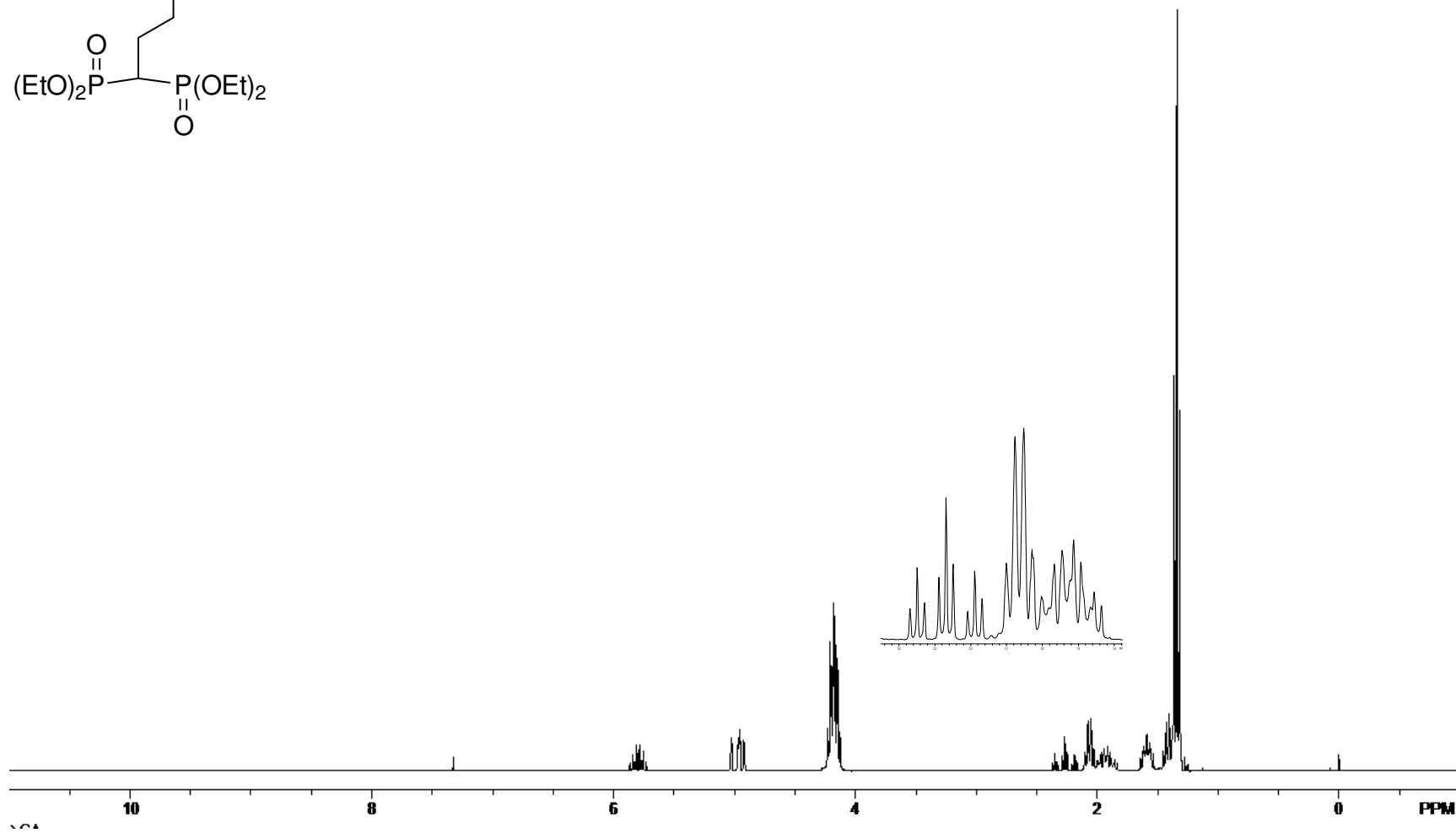
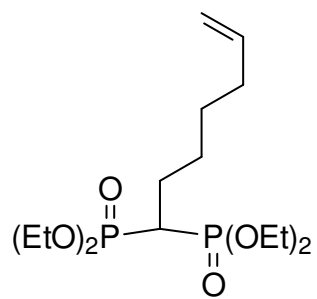
75 MHz ^{13}C NMR spectrum of 4-methyl-3-pentenylidynetrisphosphonic acid, hexaethyl ester (**12**).



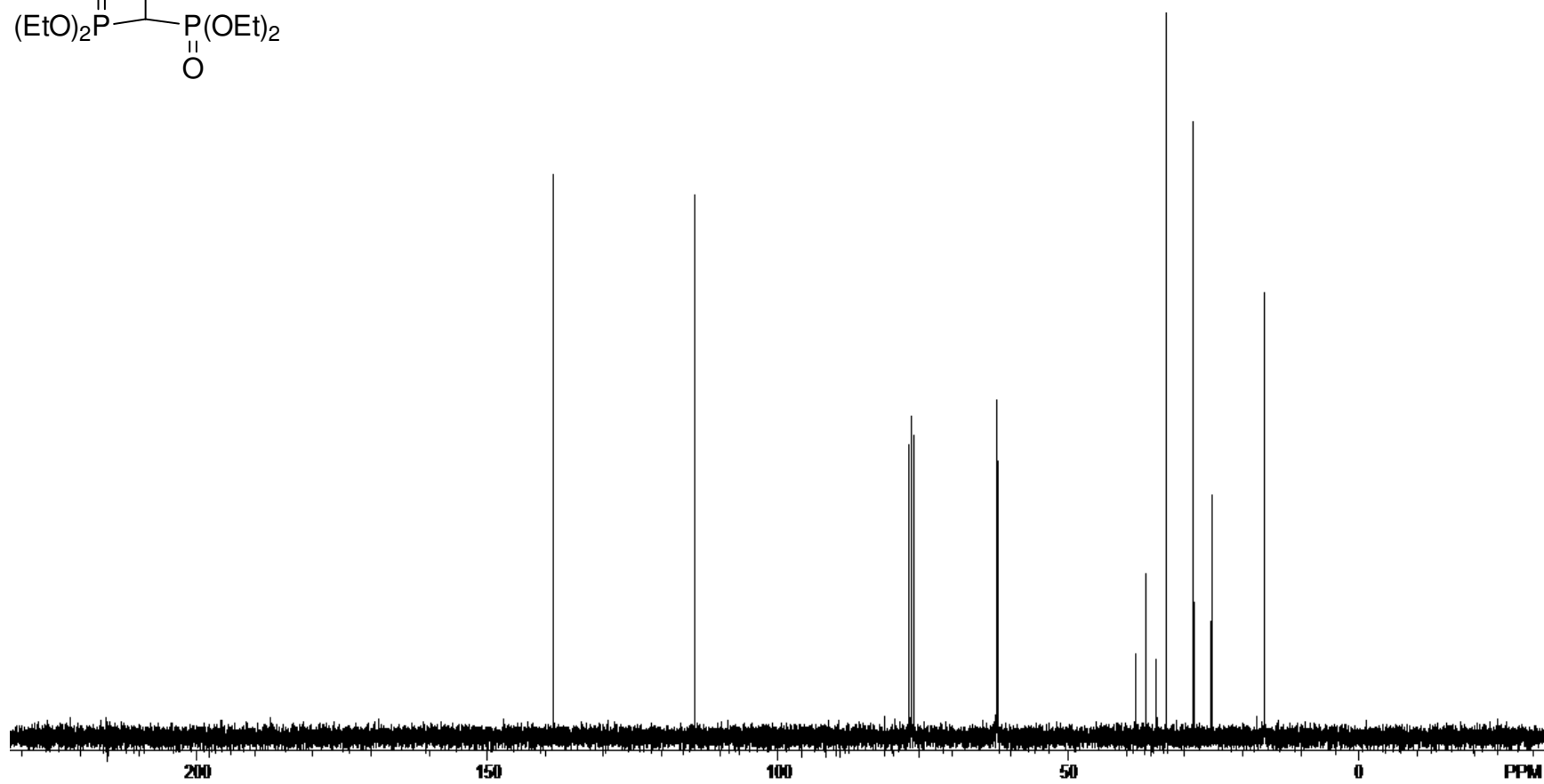
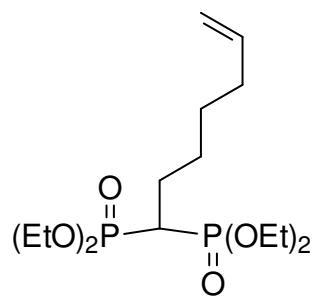
300 MHz ^1H NMR spectrum of (*3E*)-4,9-dimethyl-3-nonadienyldynetrisphosphonic acid, hexaethyl ester (**13**).



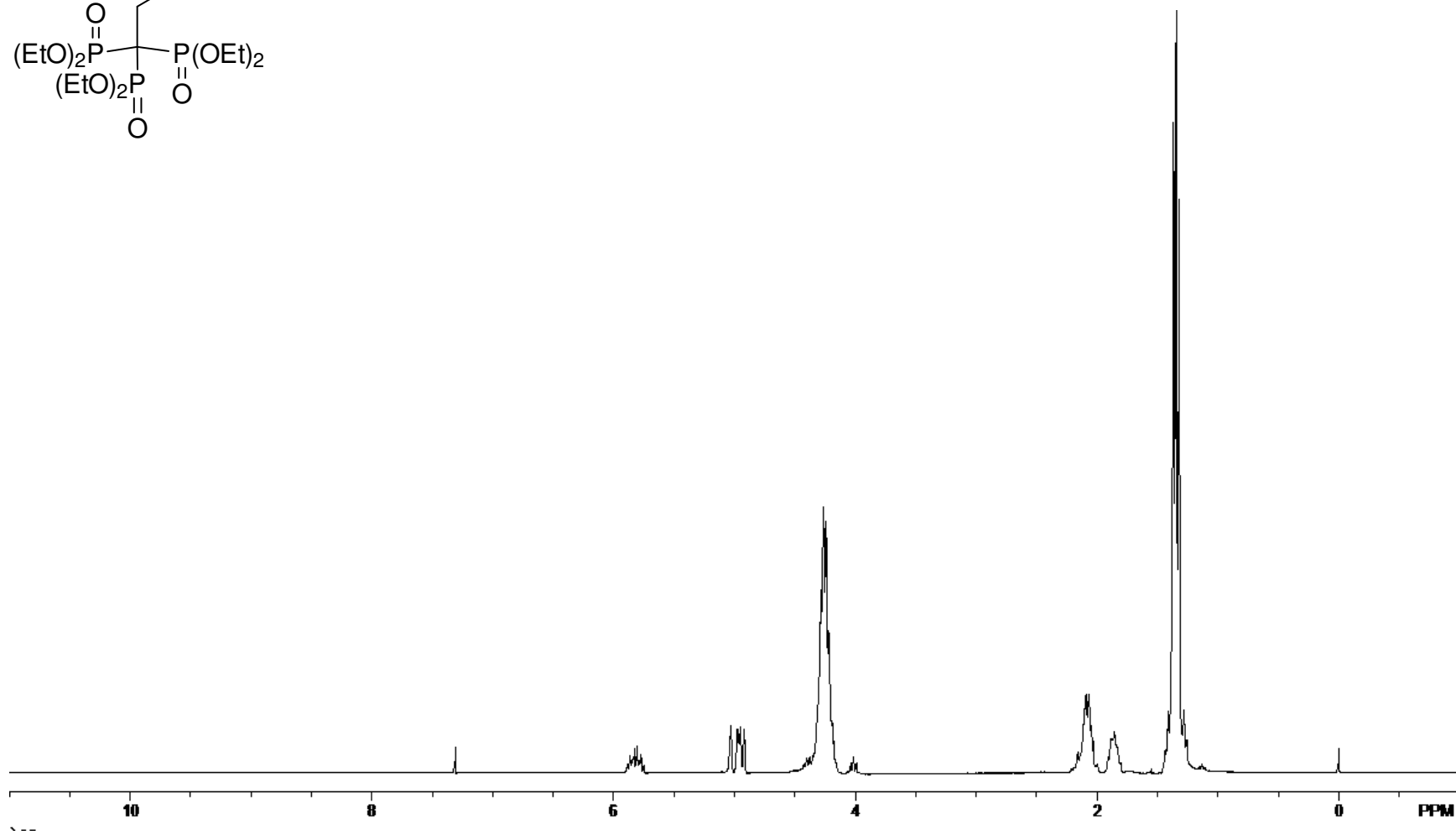
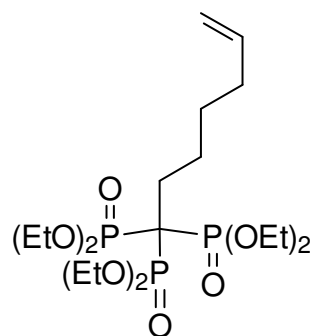
75 MHz ^{13}C NMR spectrum of (3*E*)-4,9-dimethyl-3-nonadienyldinetrisphosphonic acid, hexaethyl ester (**13**).



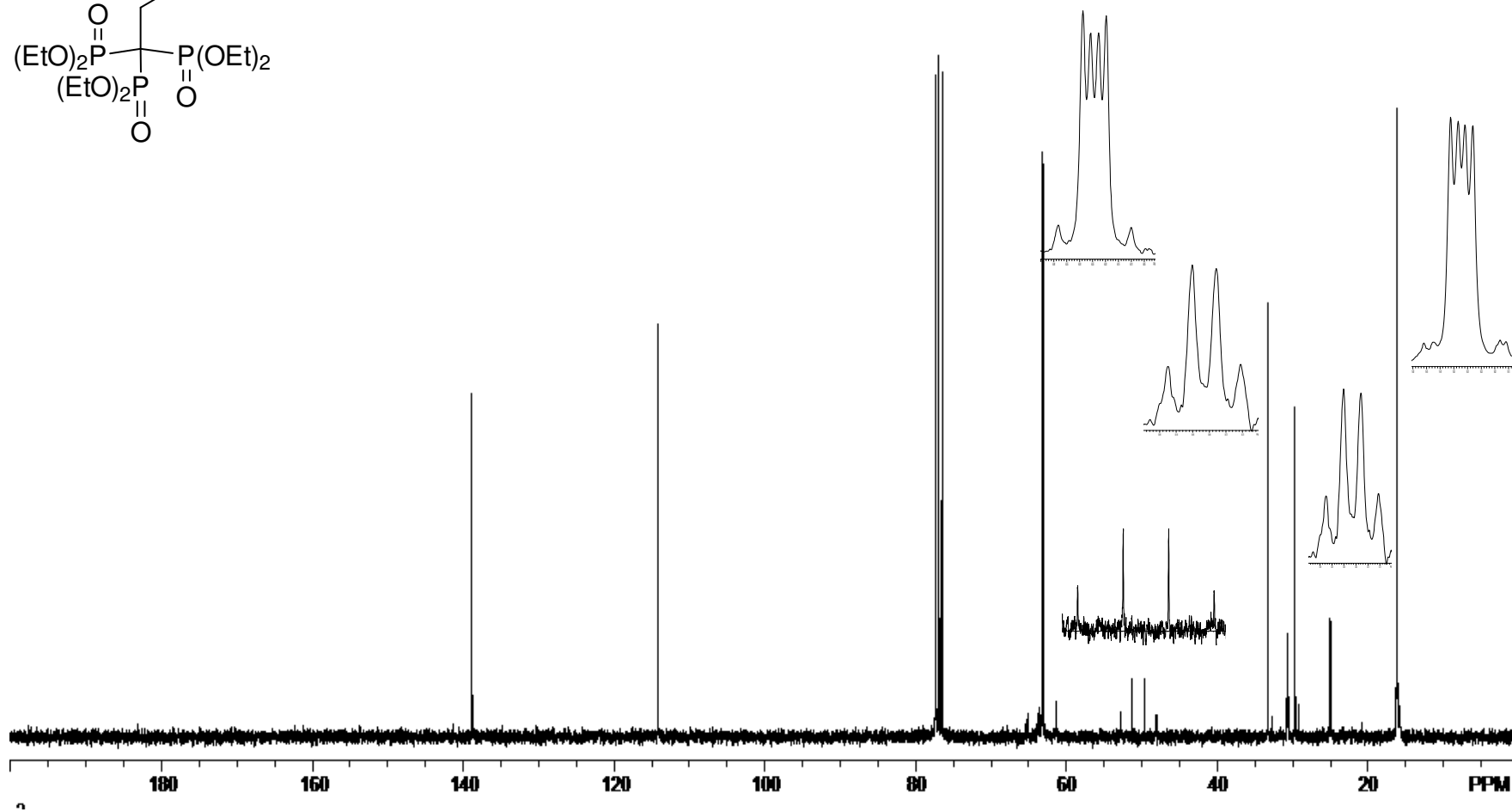
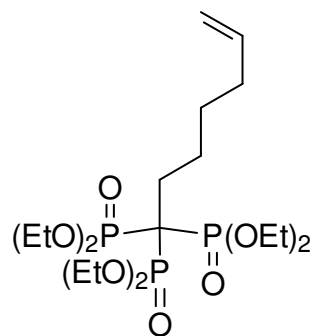
300 MHz ¹H NMR spectrum of tetraethyl 6-hepten-1,1-bisphosphonate.



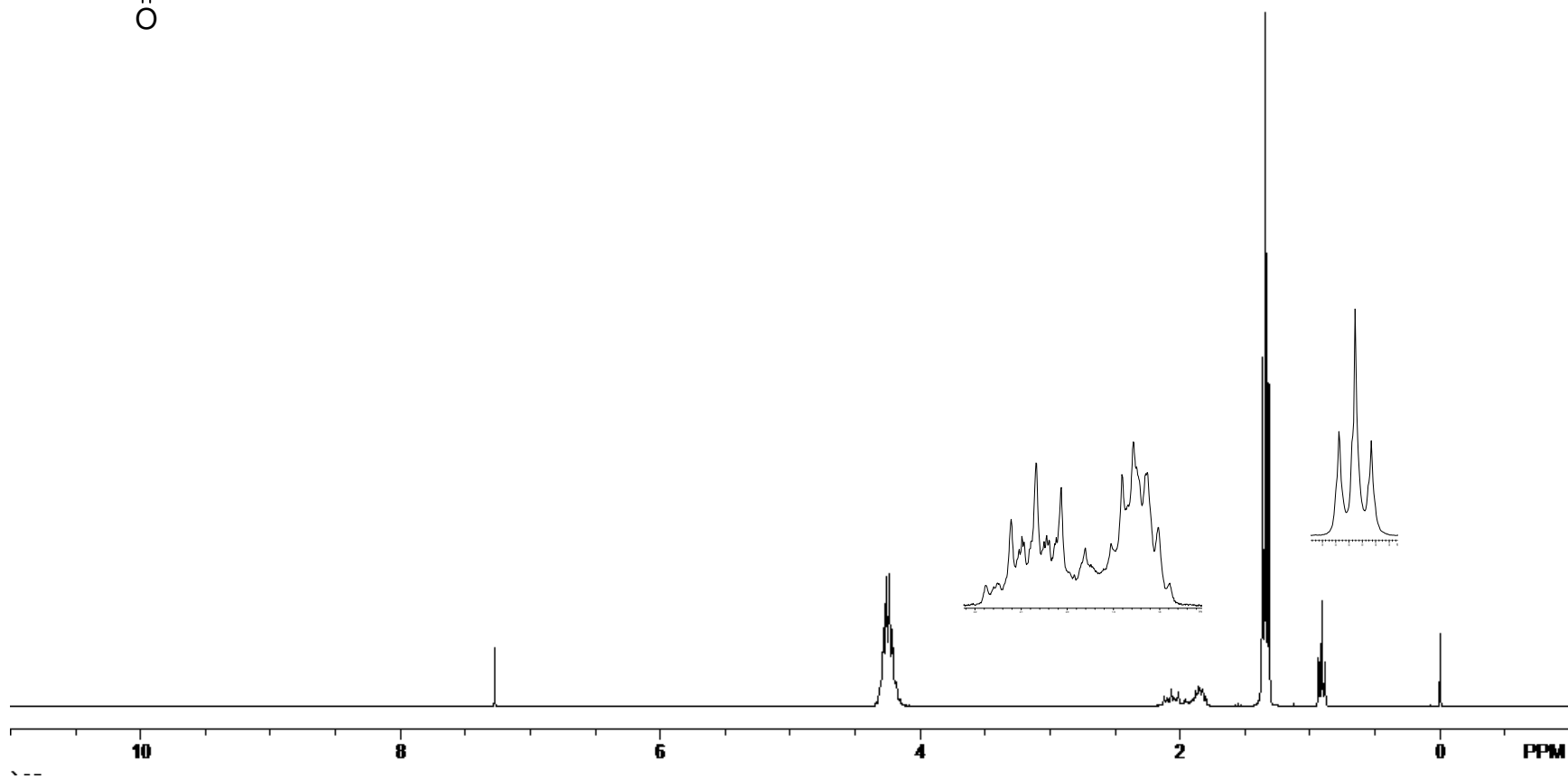
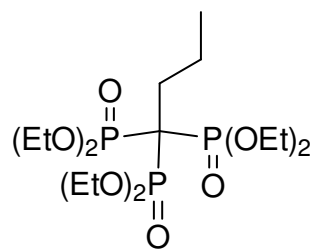
75 MHz ^{13}C NMR spectrum of tetraethyl 6-hepten-1,1-bisphosphonate.



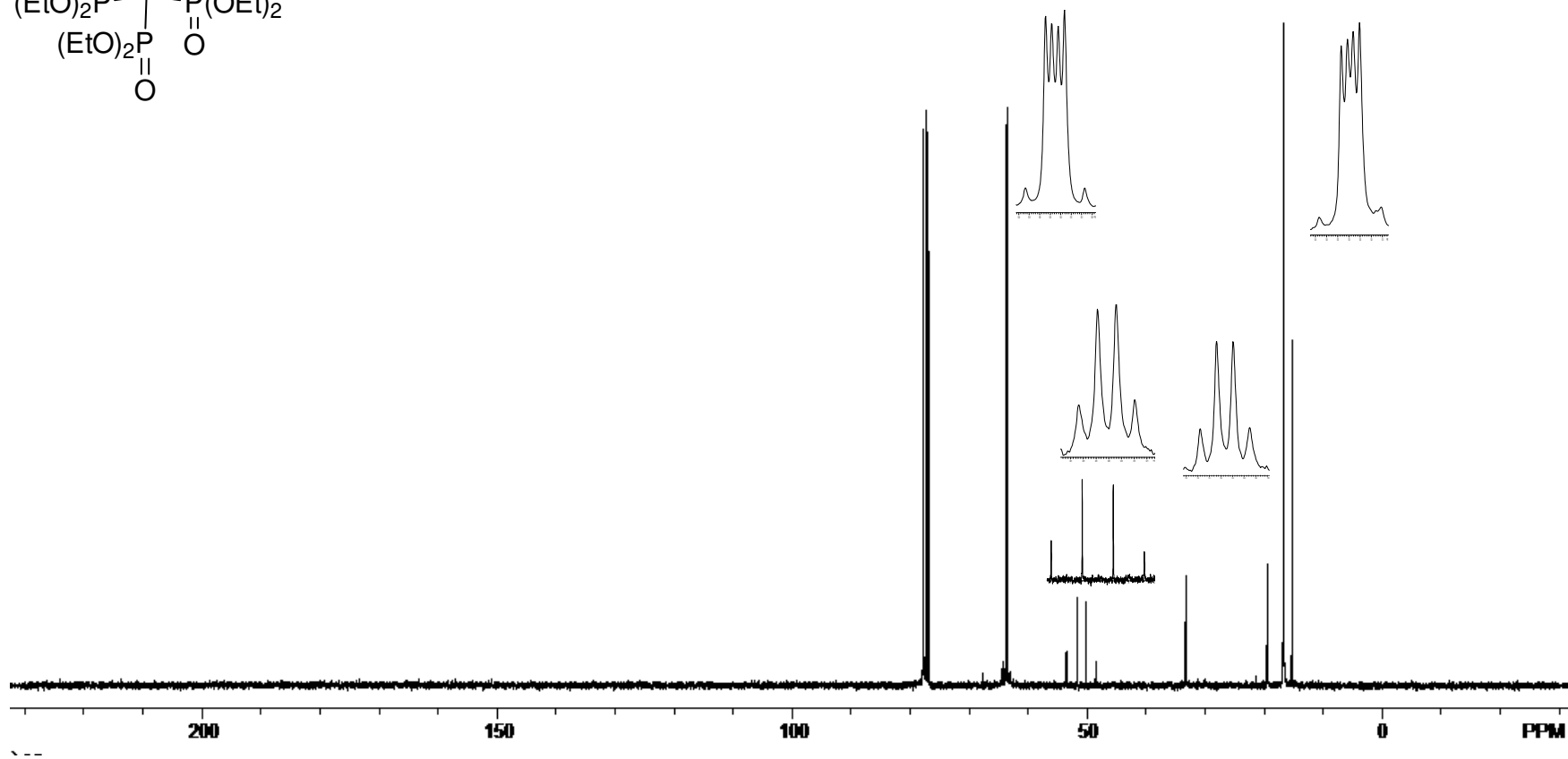
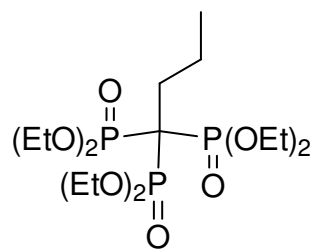
300 MHz ^1H NMR spectrum of 6-heptenyldynetrakisphosphonic acid, hexaethyl ester (**14**).



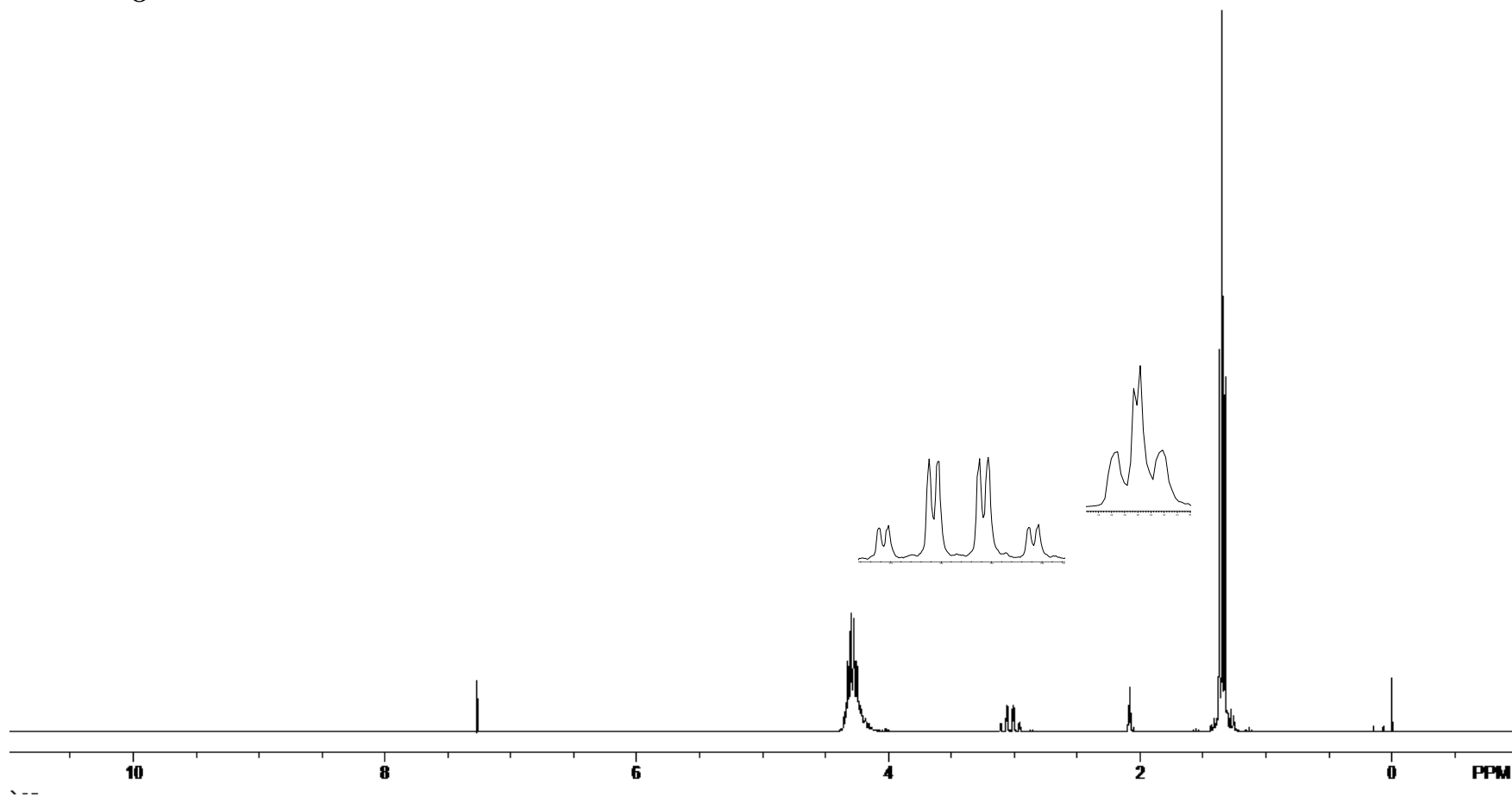
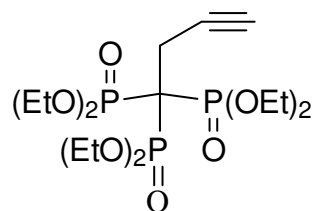
75 MHz ^{13}C NMR spectrum of 6-heptenyldynetrakisphosphonic acid, hexaethyl ester (14).



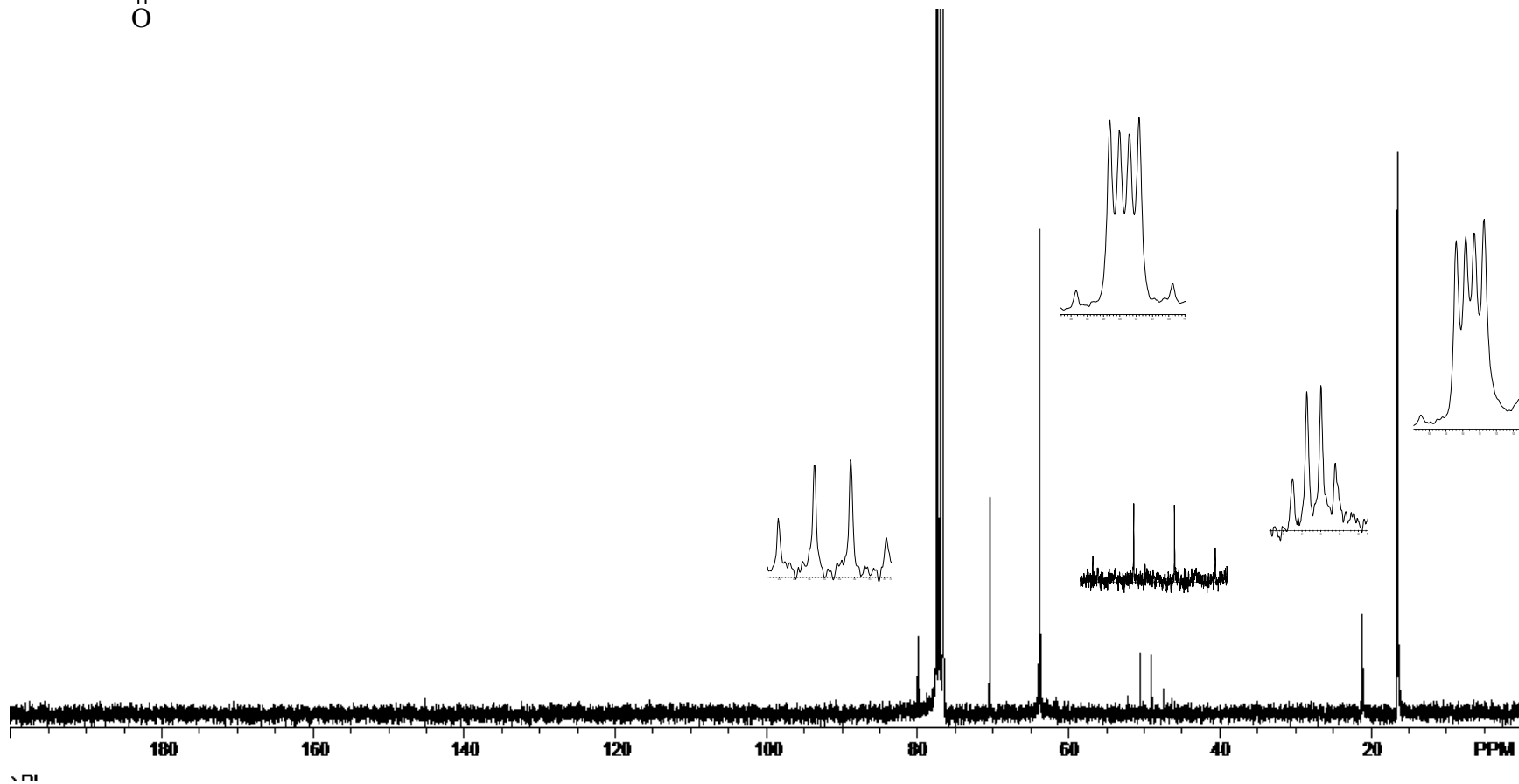
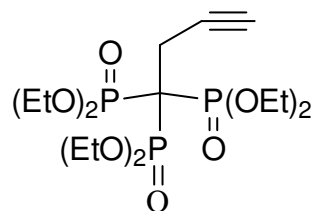
300 MHz ¹H NMR spectrum of butylidynetrisphosphonic acid, hexaethyl ester (**15**).



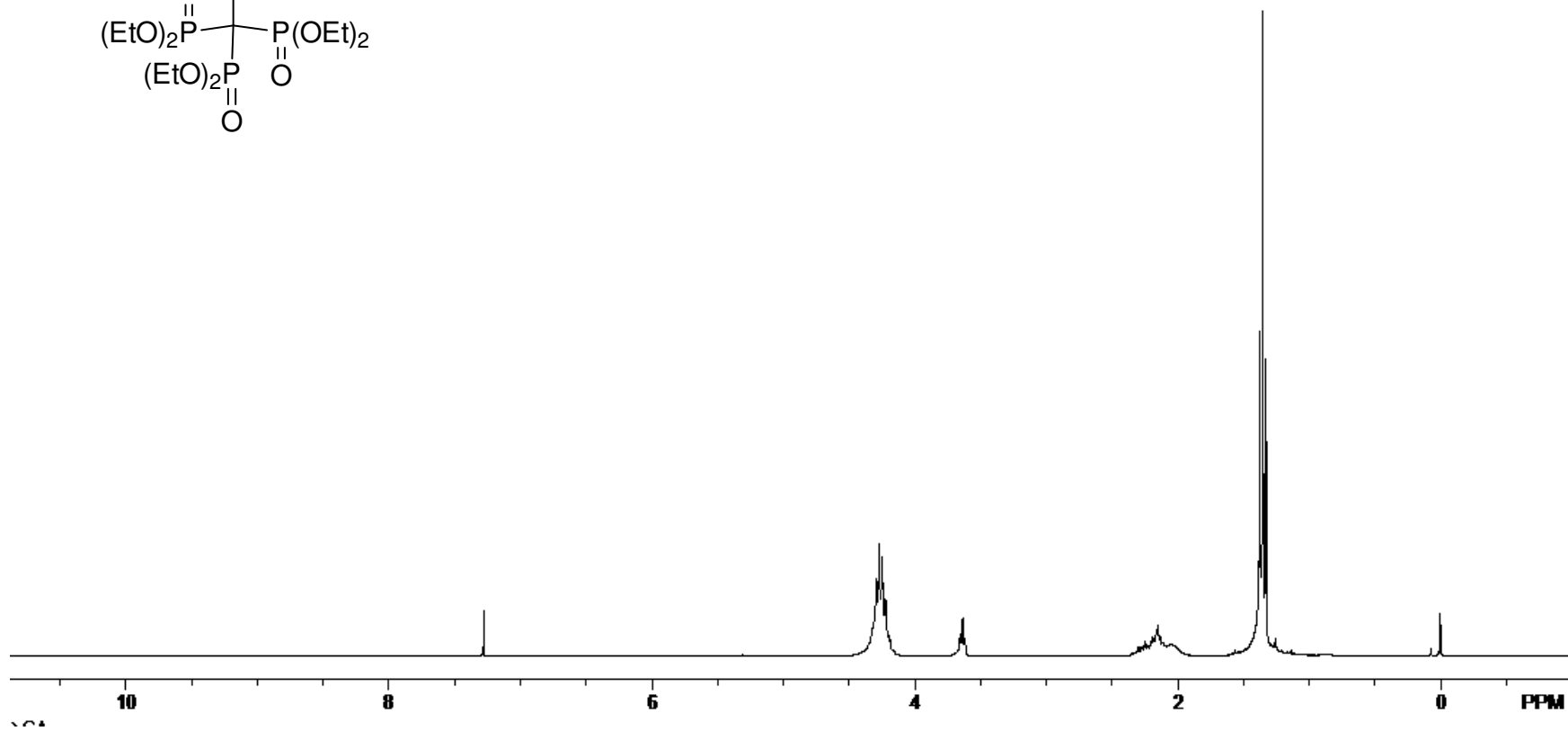
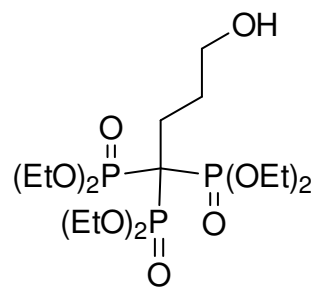
75 MHz ¹³C NMR spectrum of butylidynetrisphosphonic acid, hexaethyl ester (**15**).



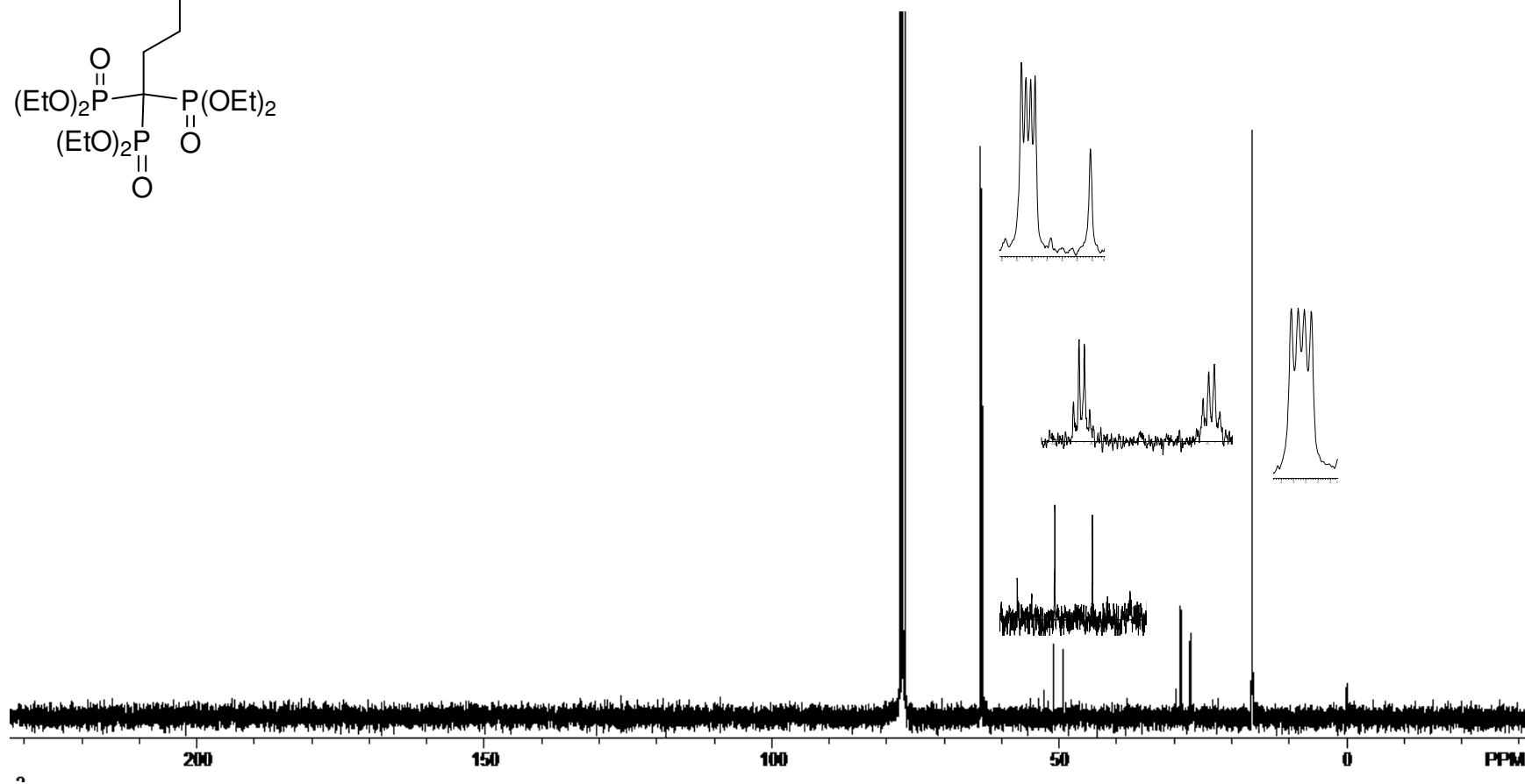
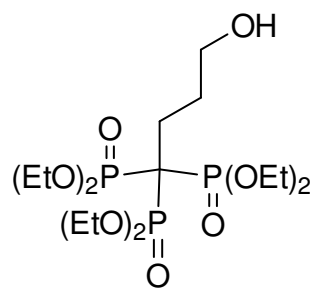
300 MHz ^1H NMR spectrum of 3-butyn-1-ylidynetrisphosphonic acid, hexaethyl ester (**16**).



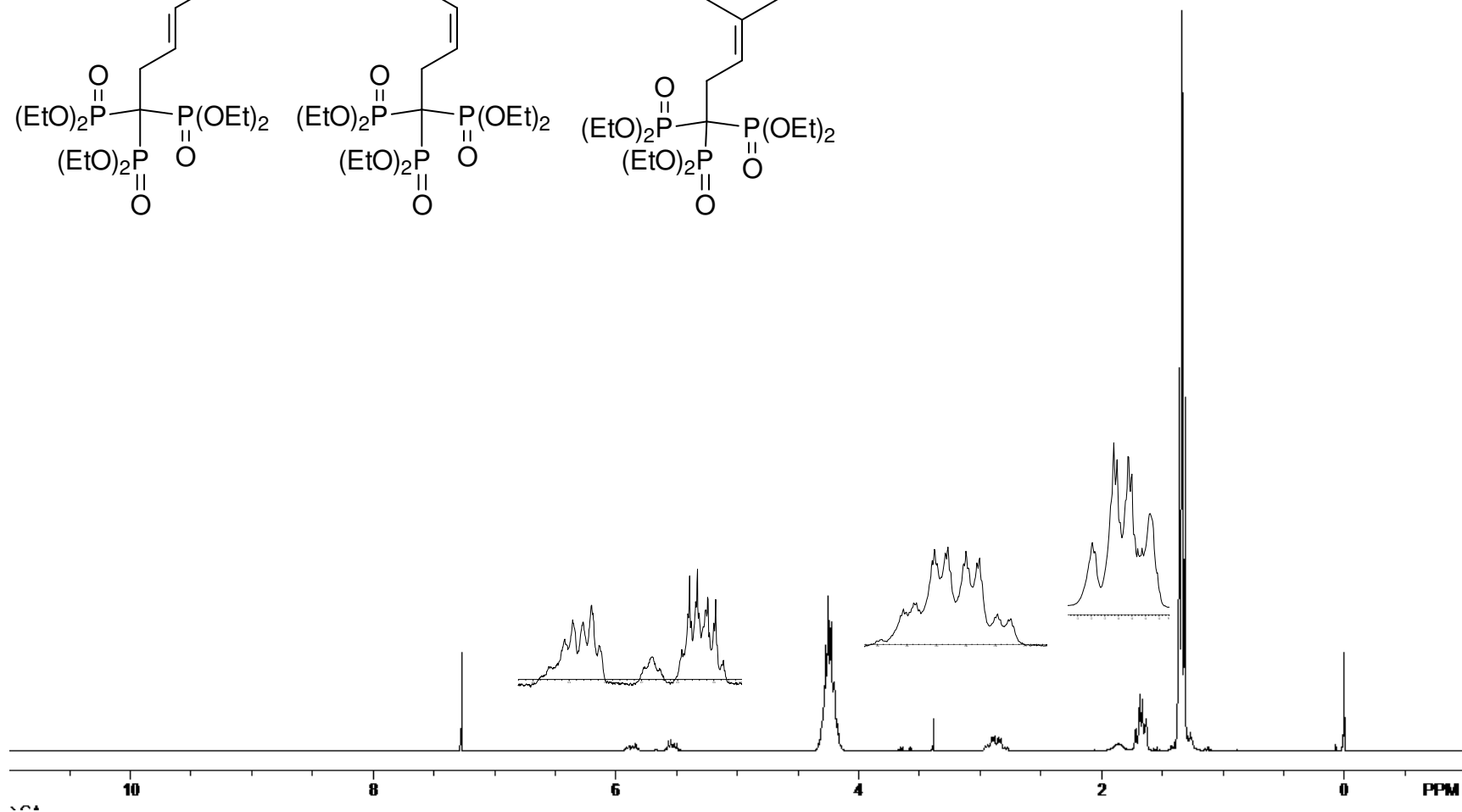
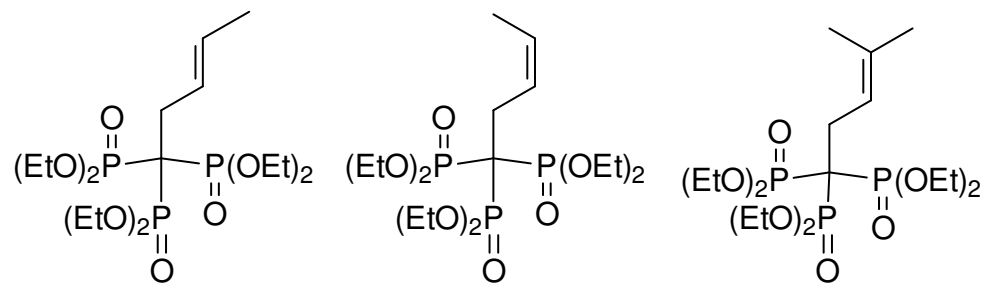
75 MHz ^{13}C NMR spectrum of 3-butyn-1-ylidynetrisphosphonic acid, hexaethyl ester (**16**).



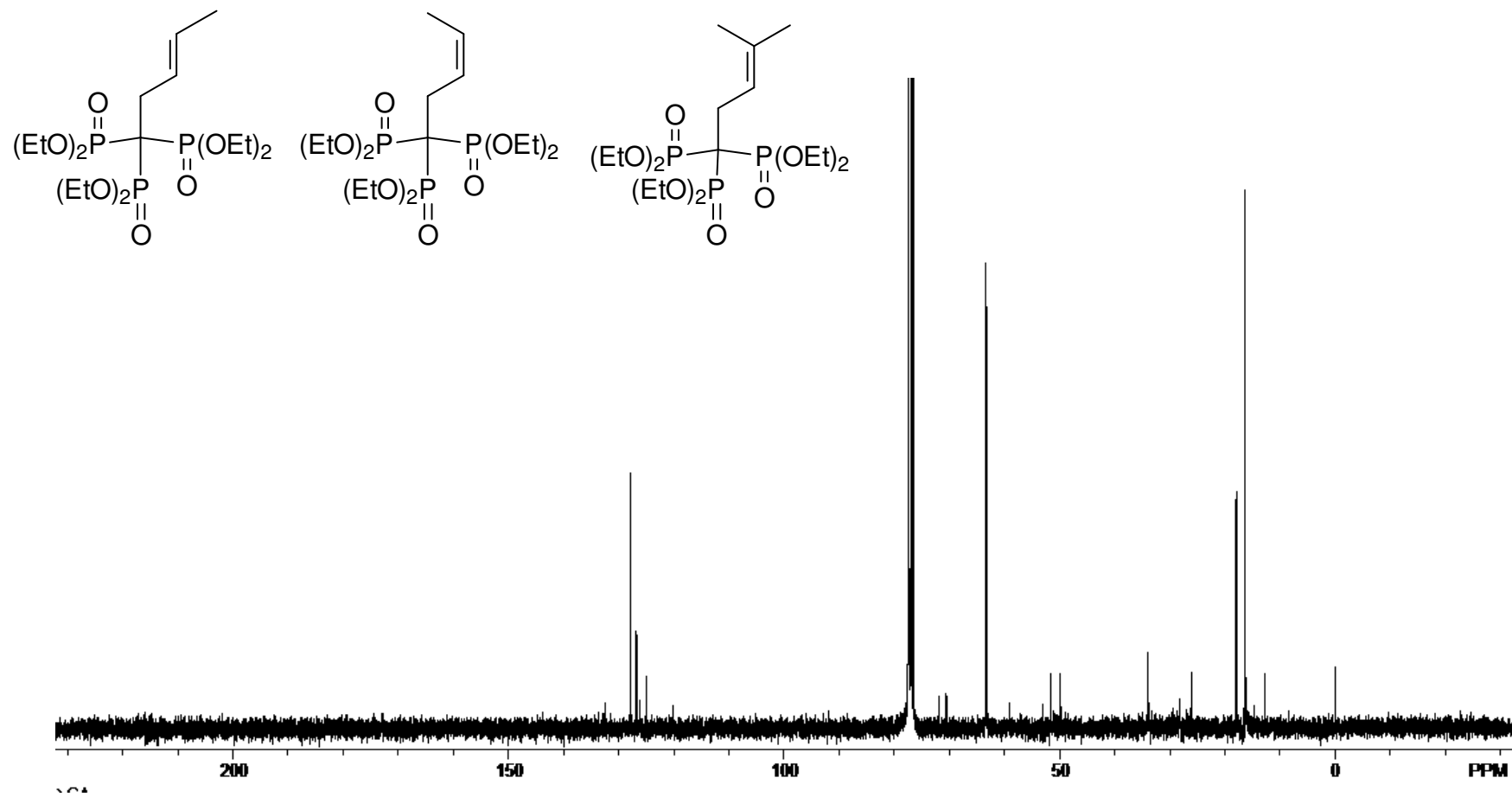
300 MHz ^1H NMR spectrum of 4-hydroxybutylidynetrisphosphonic acid, hexaethyl ester (**19**).



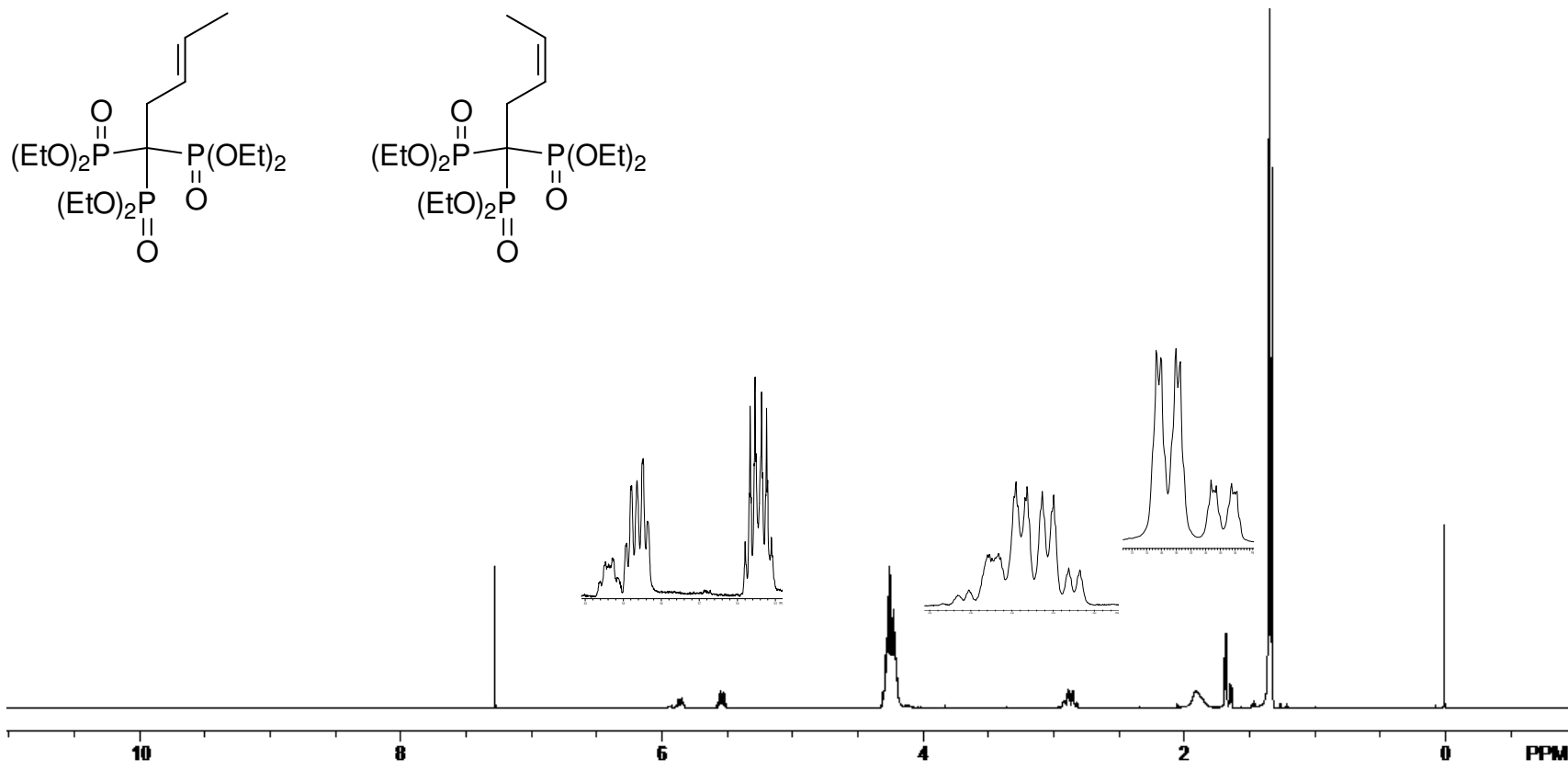
75 MHz ^{13}C NMR spectrum of 4-hydroxybutylidynetrisphosphonic acid, hexaethyl ester (**19**).



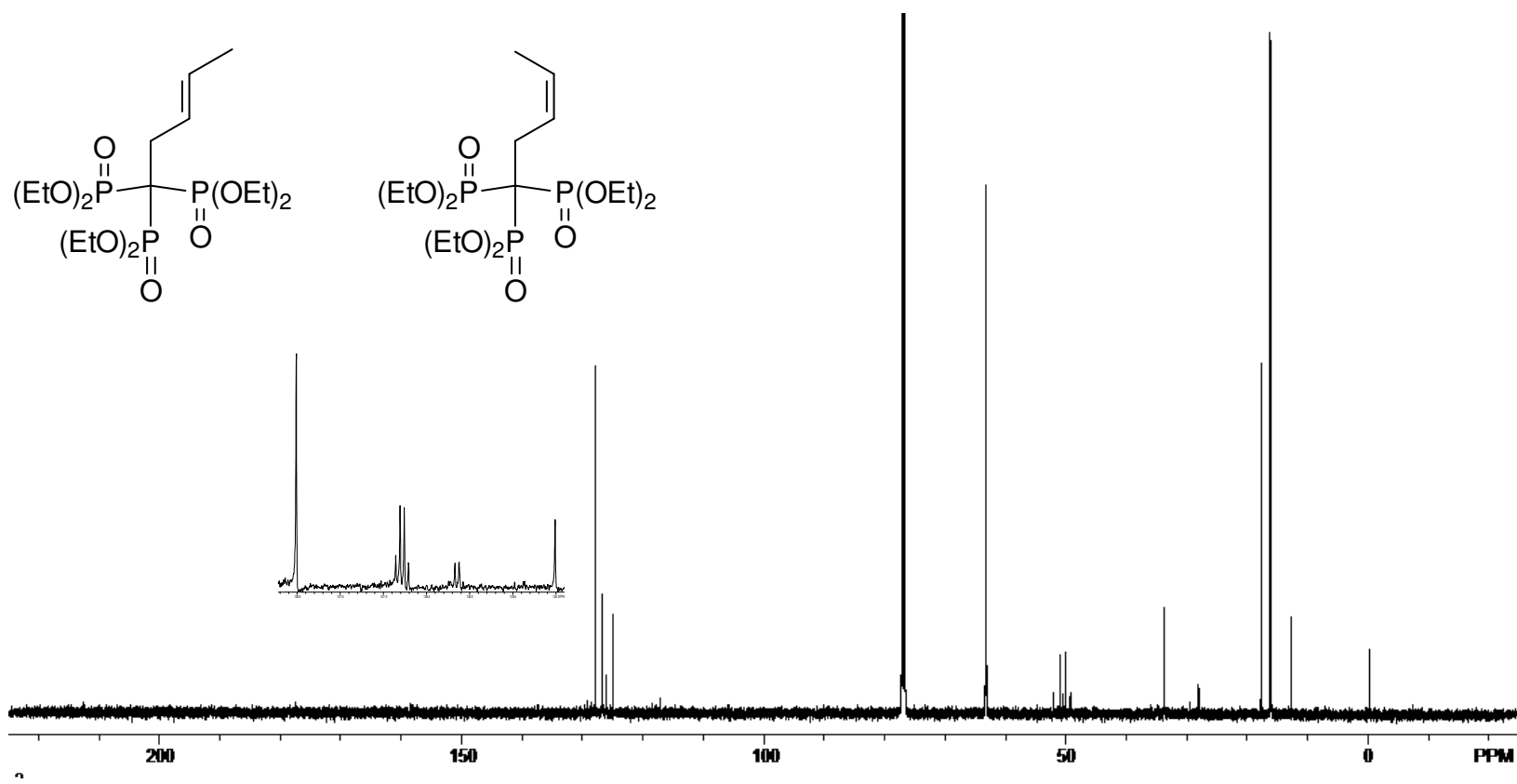
300 MHz ^1H NMR spectrum of 3-pentenylidynetrisphosphonic acid, hexaethyl ester (**20**) and compound **12**



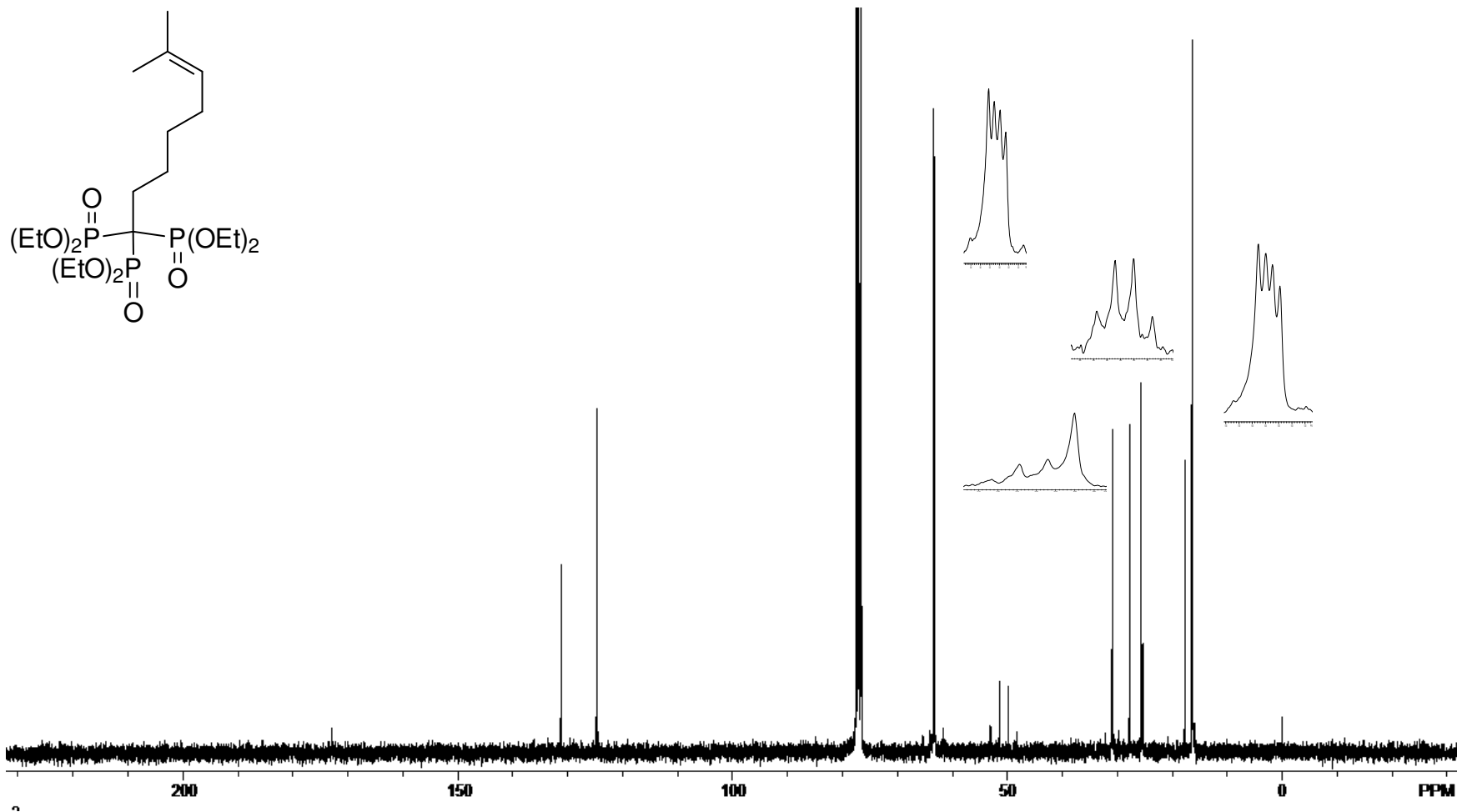
75 MHz ^{13}C NMR spectrum of 3-pentenylidynetrisphosphonic acid, hexaethyl ester (**20**) and compound **12**.



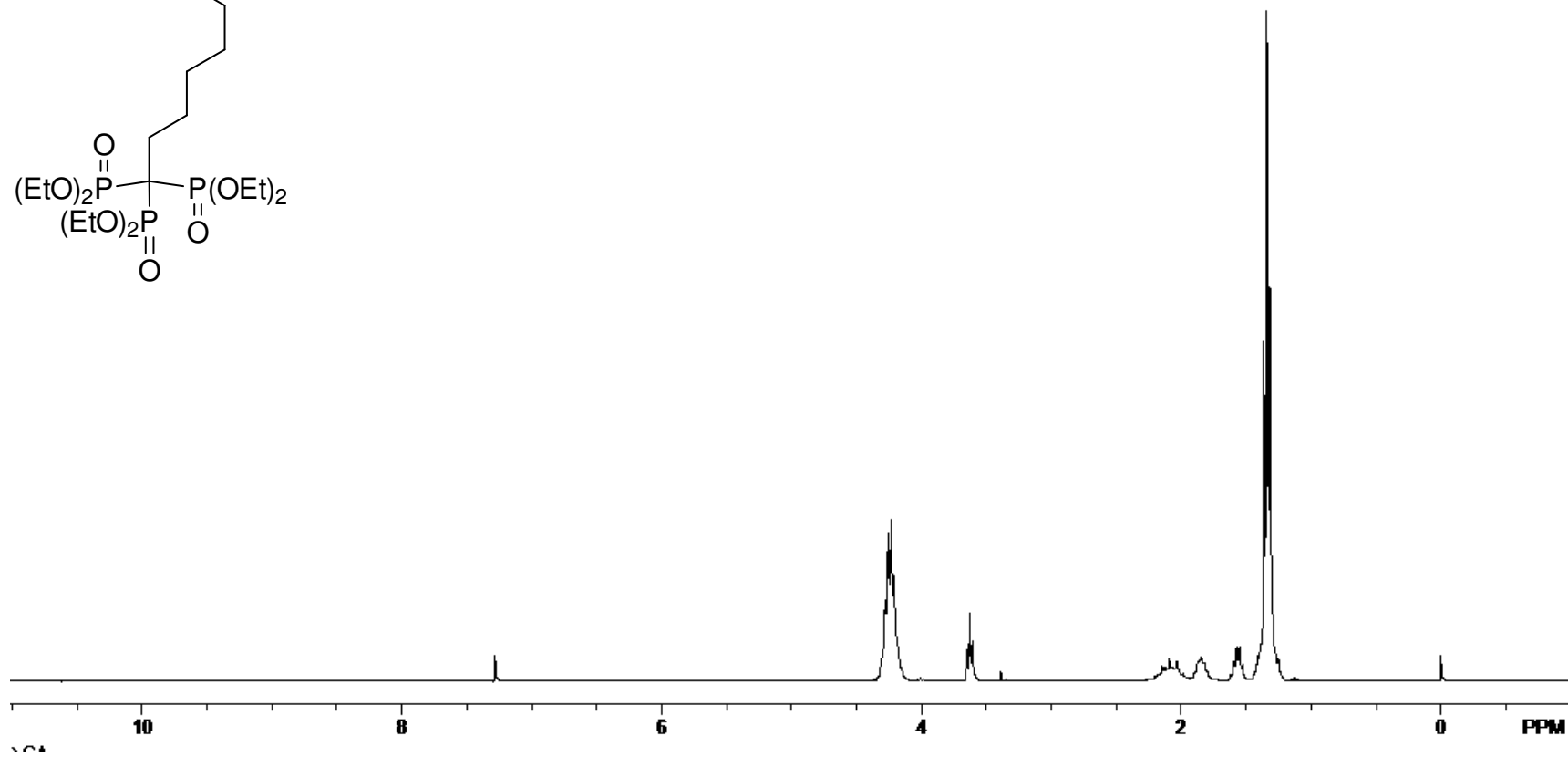
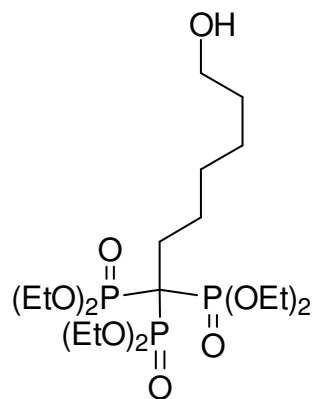
500 MHz ¹H NMR spectrum of 3-pentenylidynetrisphosphonic acid, hexaethyl ester (**20**).



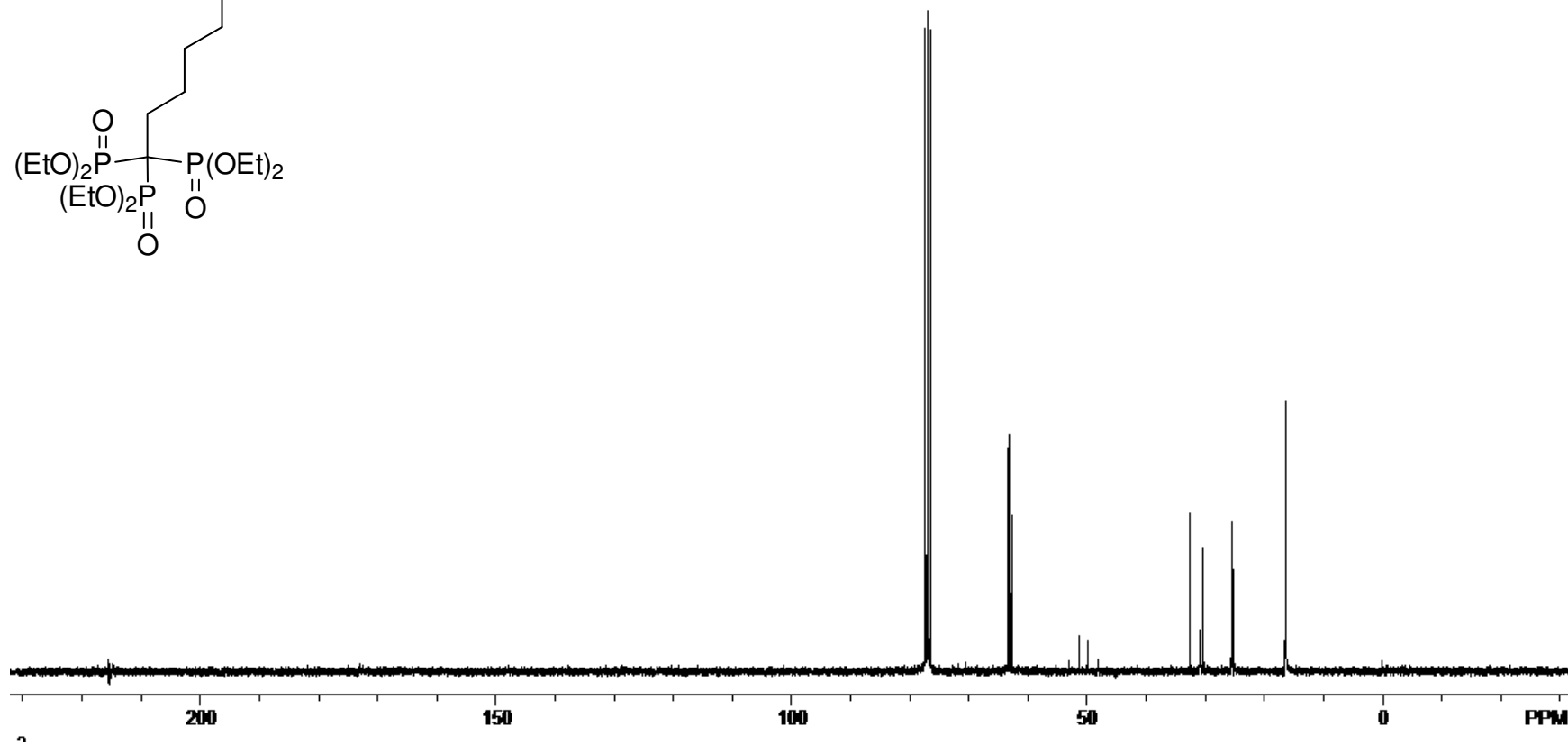
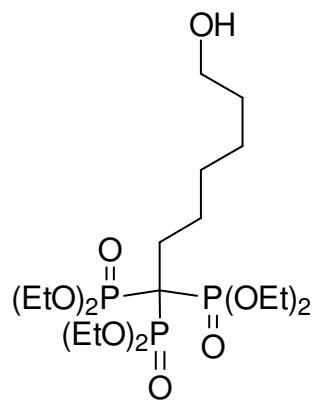
125 MHz ^{13}C NMR spectrum of 3-pentenylidynetrisphosphonic acid, hexaethyl ester (**20**).



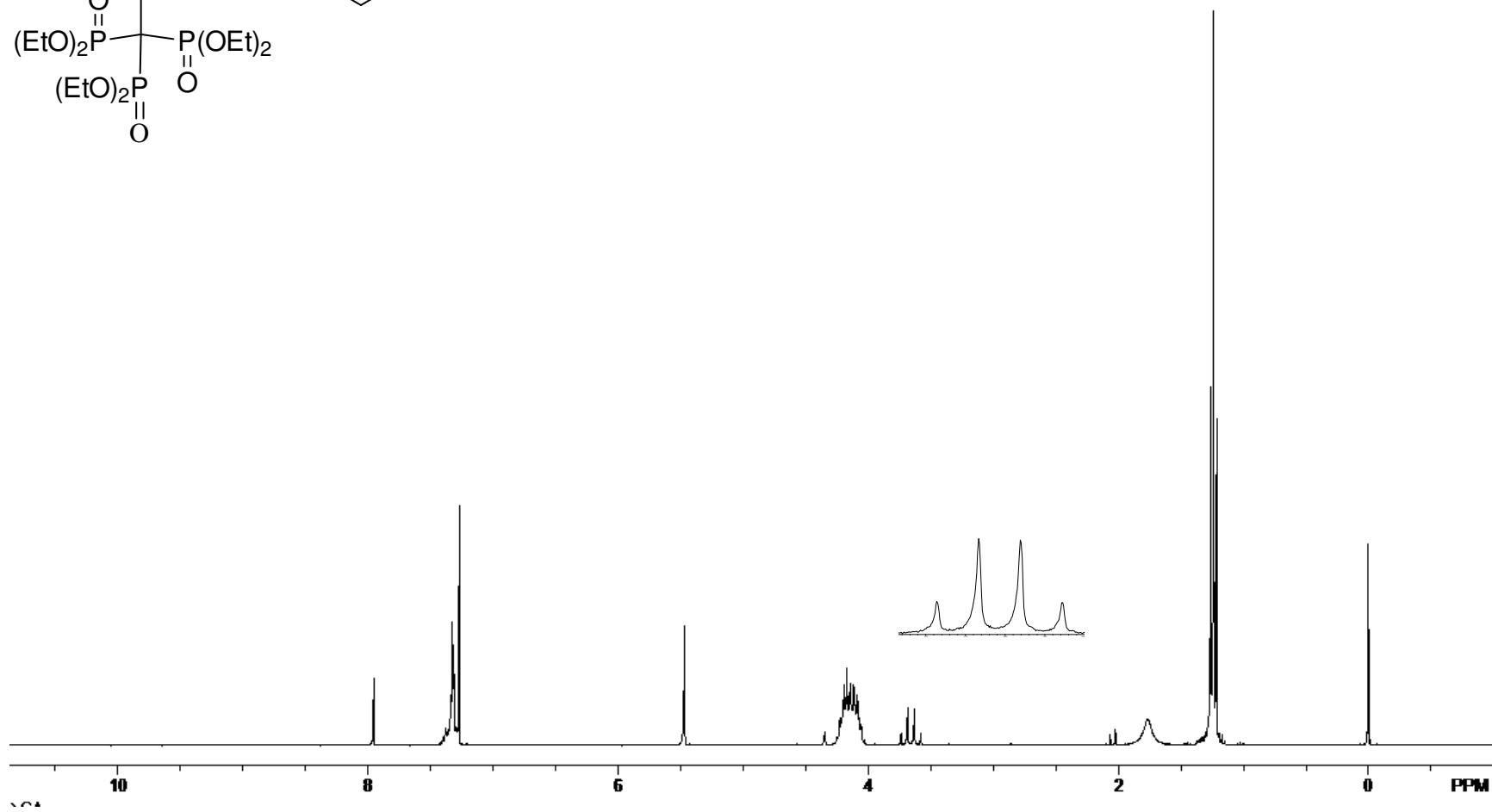
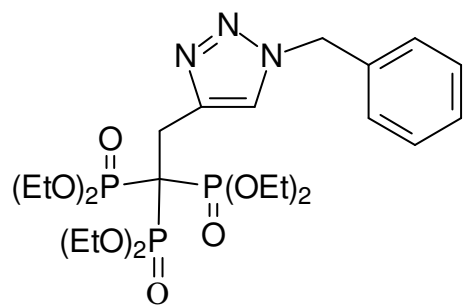
75 MHz ^{13}C NMR spectrum of 7-methyl-6-octenyldynetrphosphonic acid, hexaethyl ester (**21**).



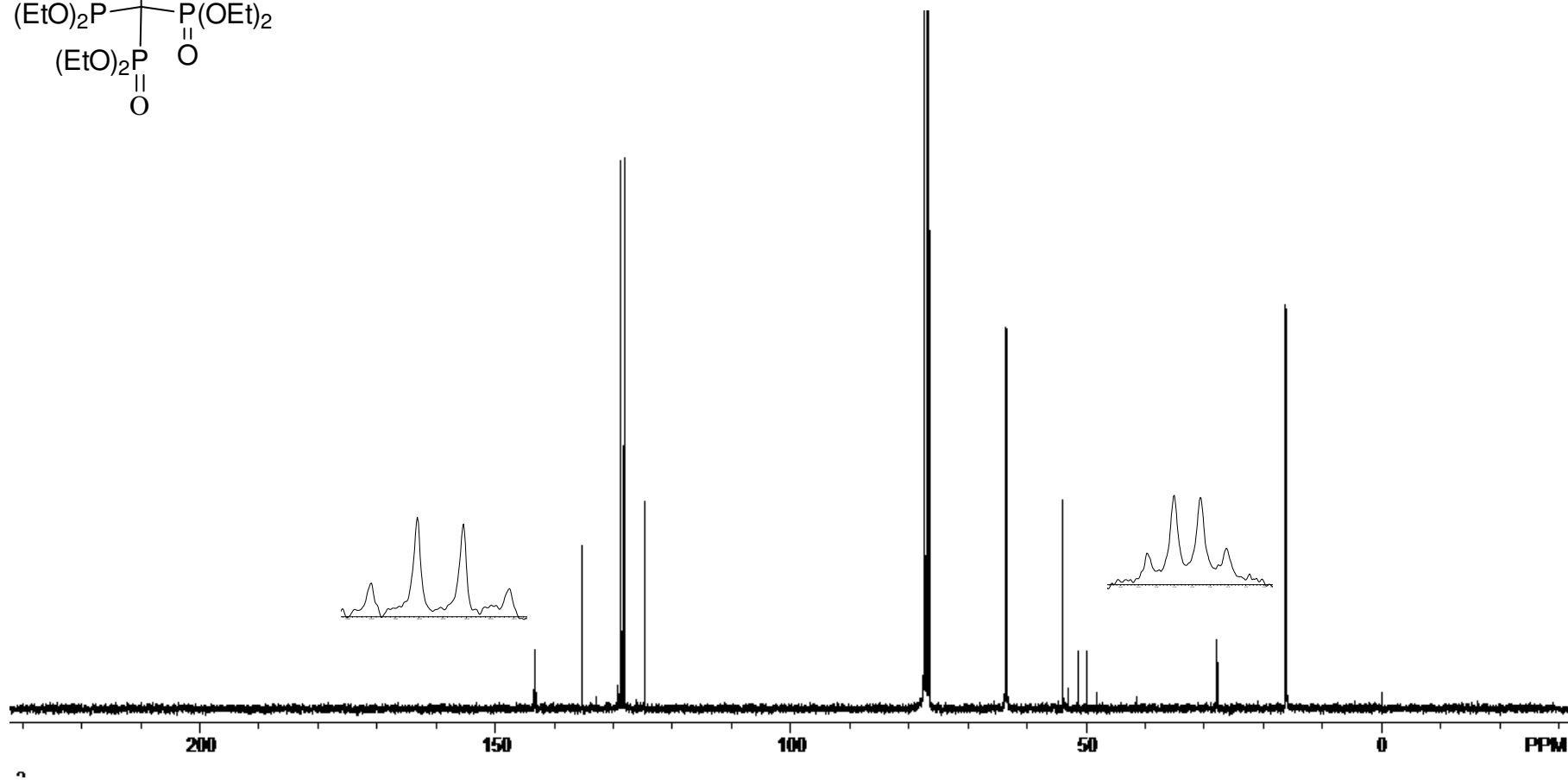
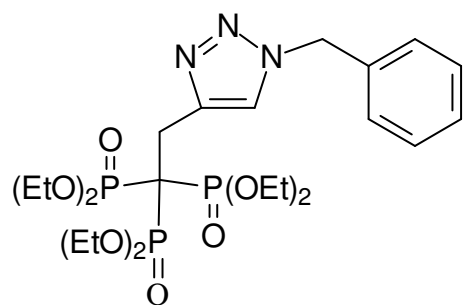
300 MHz ¹H NMR spectrum of 7-hydroxyheptylidynetrisphosphonic acid, hexaethyl ester (**22**).



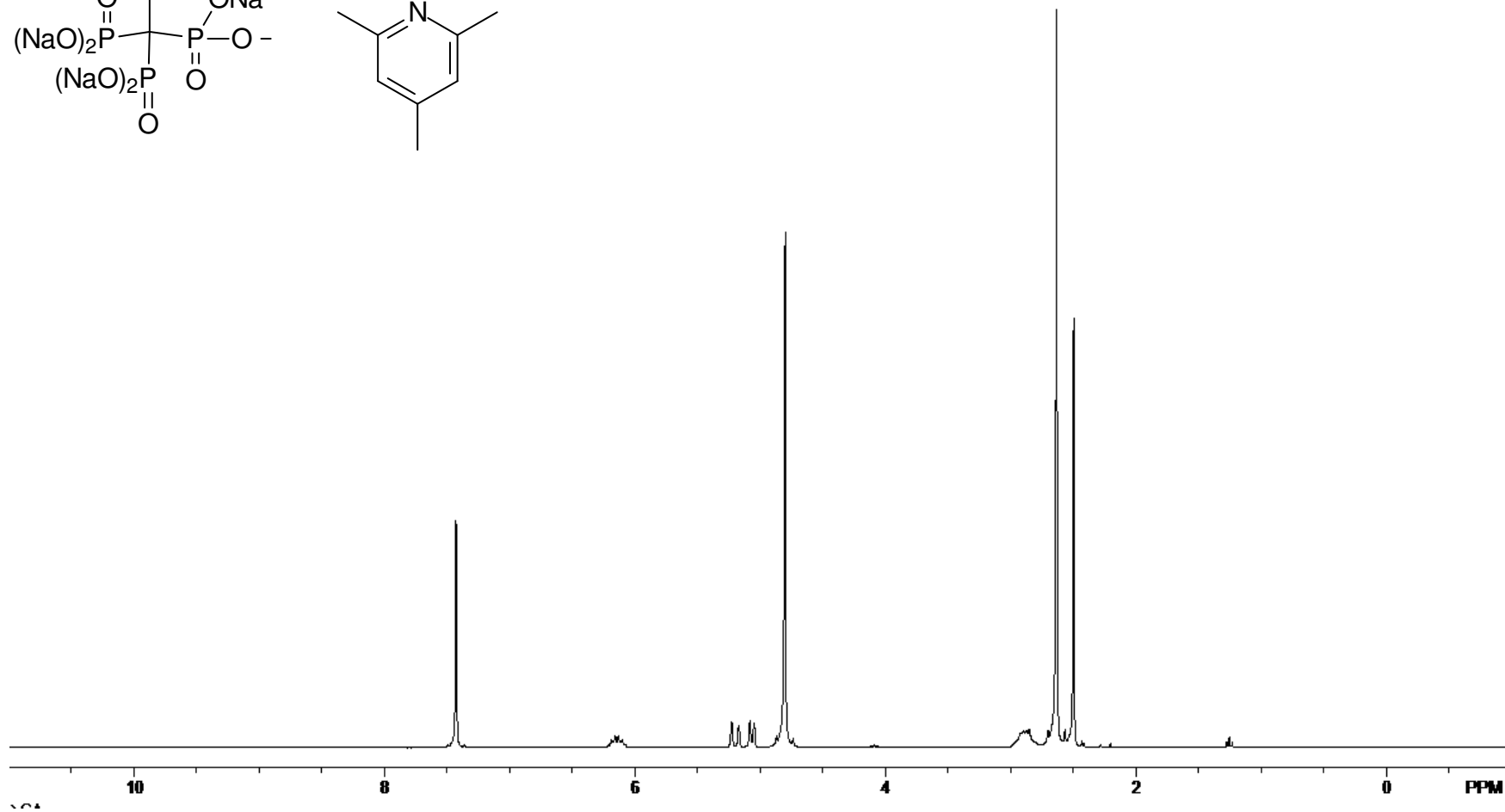
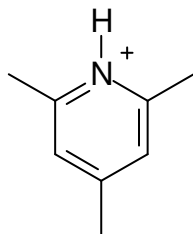
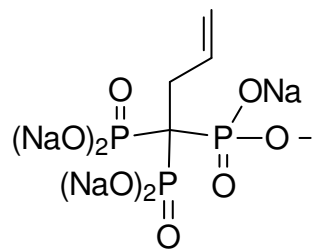
75 MHz ¹³C NMR spectrum of 7-hydroxyheptylidynetrisphosphonic acid, hexaethyl ester (**22**).



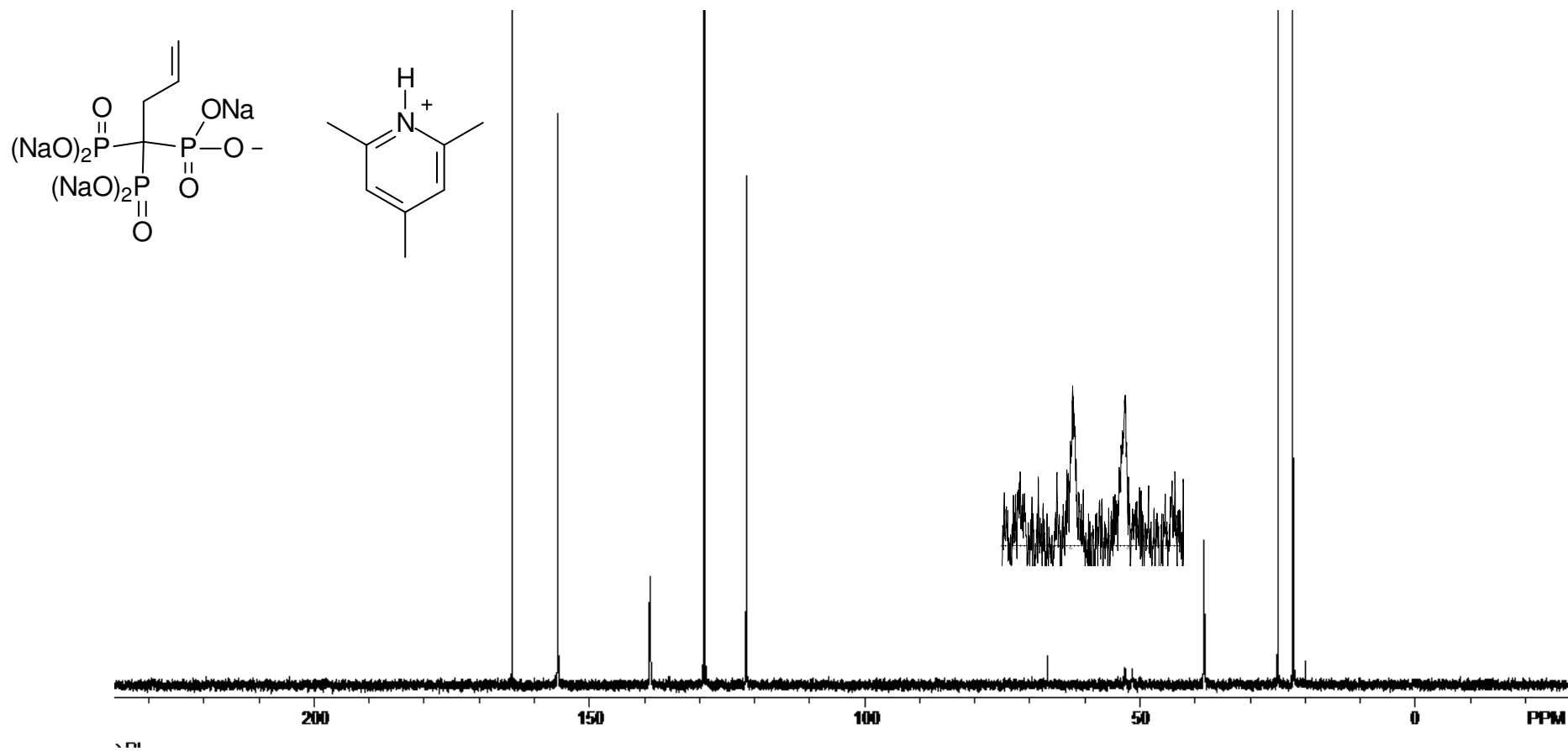
300 MHz ¹H NMR spectrum of 1-benzyl-4-[2,2,2-tris(diethoxyphosphinyl)ethyl]-1H-1,2,3-triazole (**23**).



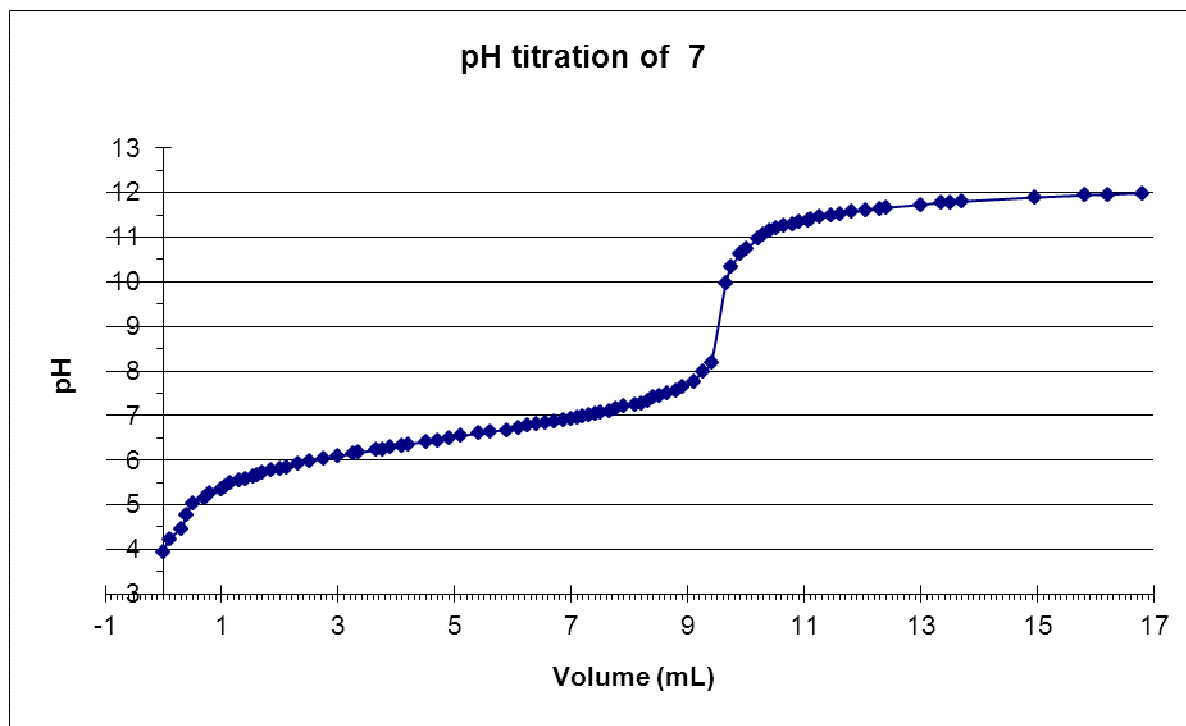
75 MHz ^{13}C NMR spectrum of 1-benzyl-4-[2,2,2-tris(diethoxyphosphiny)ethyl]-1H-1,2,3-triazole (**23**).



300 MHz ¹H NMR spectrum of 3-butenyldynetrtrisphosphonic acid, pentasodium, 2,4,6-trimethylpyridinium salt (**24**) (in D₂O).



75 MHz ¹³C NMR spectrum of 3-butenylidynetrisphosphonic acid, pentasodium, 2,4,6-trimethylpyridinium salt (**24**) (in D₂O with 1,4-dioxane as an internal standard at 67.0 ppm).



pH Titration of Compound 7 with NaOH.