

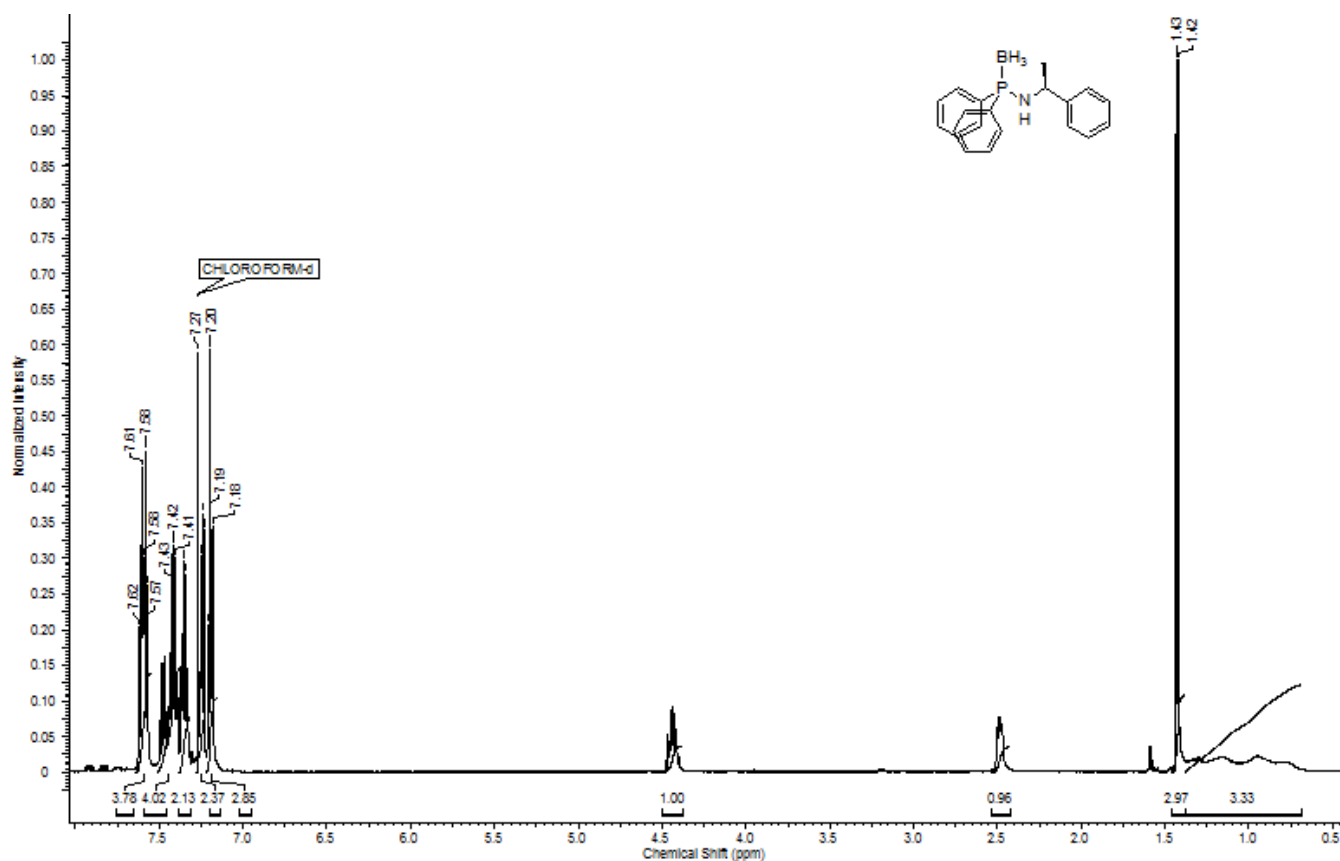
SUPPORTING INFORMATION

DOI:

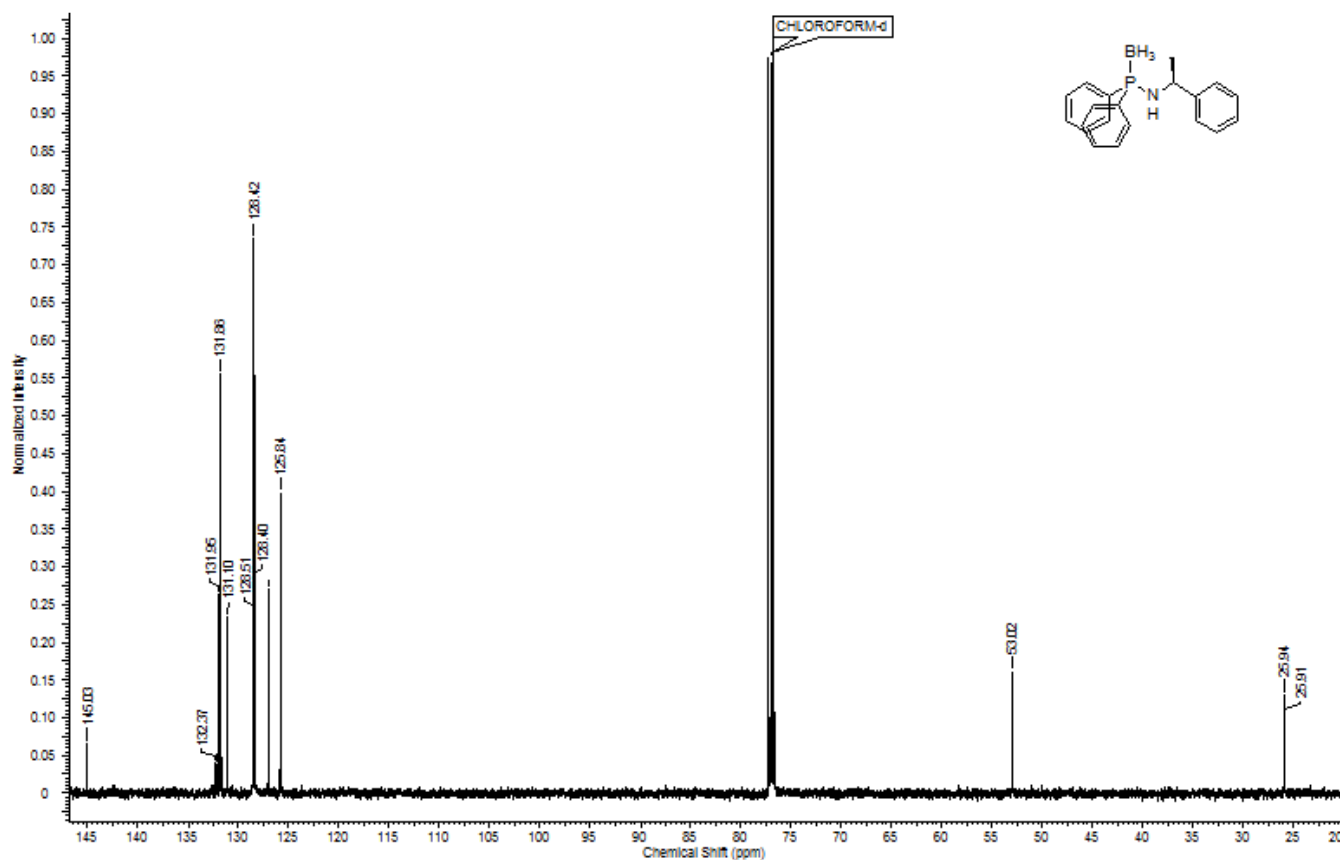
Title: Diastereoselective desymmetrization of diarylphosphinous acid-borane amides under Birch reduction

Authors: Marek Stankevič

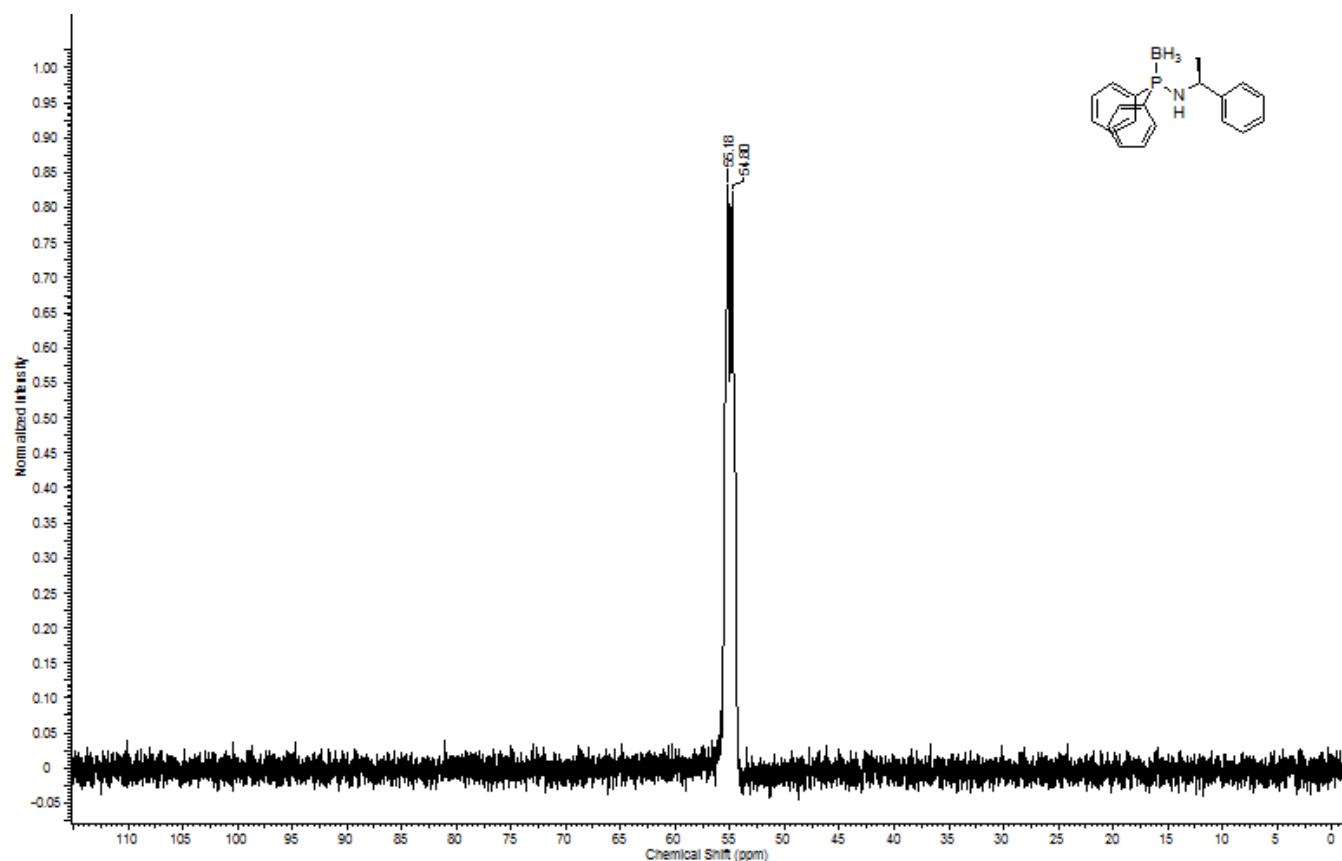
Copies of NMR spectras



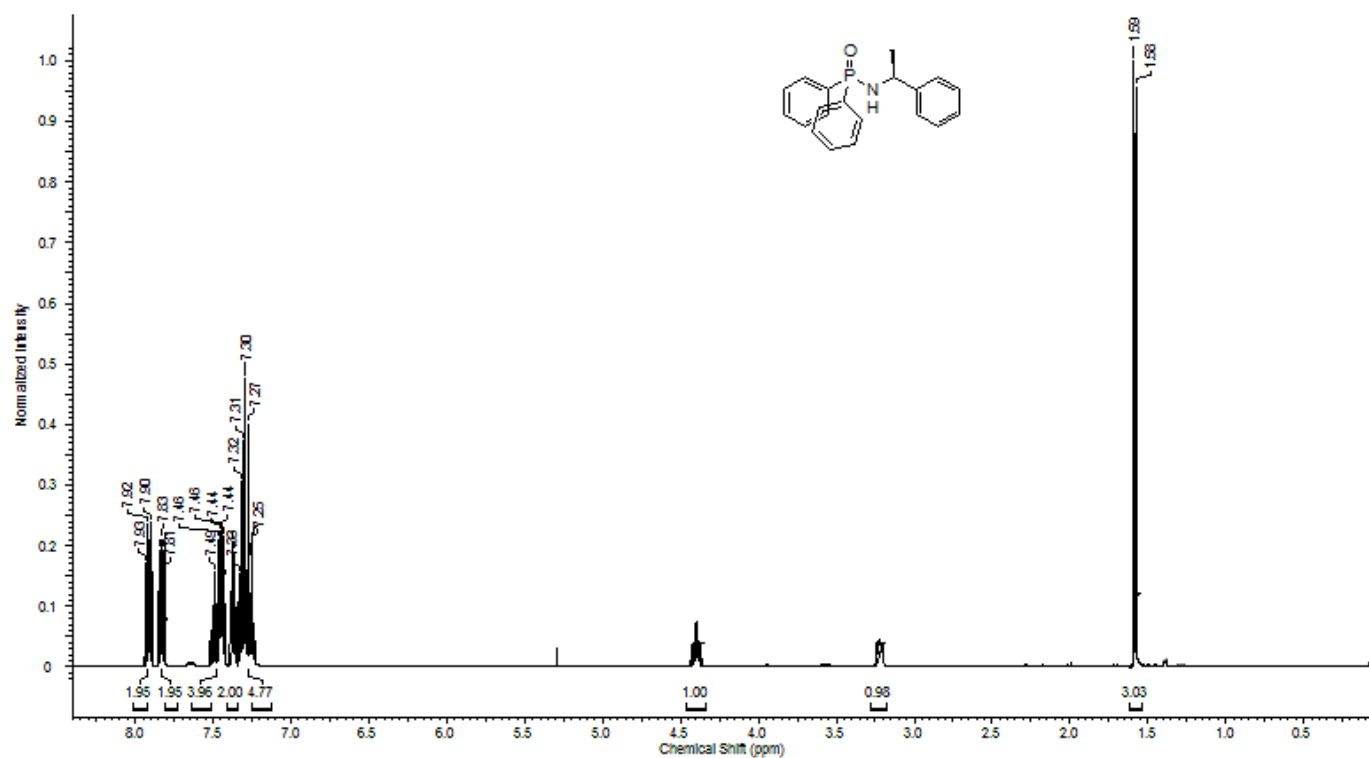
^1H NMR spectra of diphenylphosphinous acid-borane (*S*)- α -methylbenzylamide (**10a**) (500 MHz, CDCl_3).



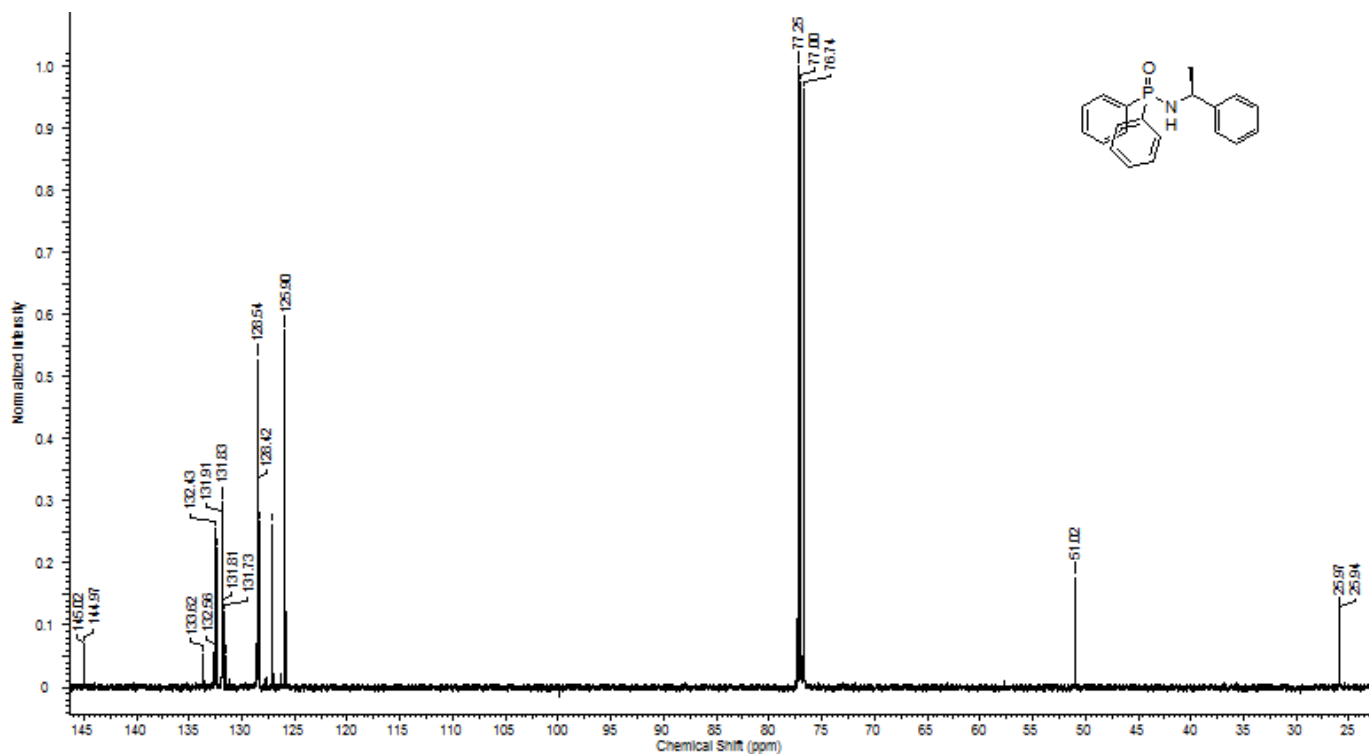
^{13}C NMR spectra of diphenylphosphinous acid-borane (*S*)- α -methylbenzylamide (**10a**) (126 MHz, CDCl_3).



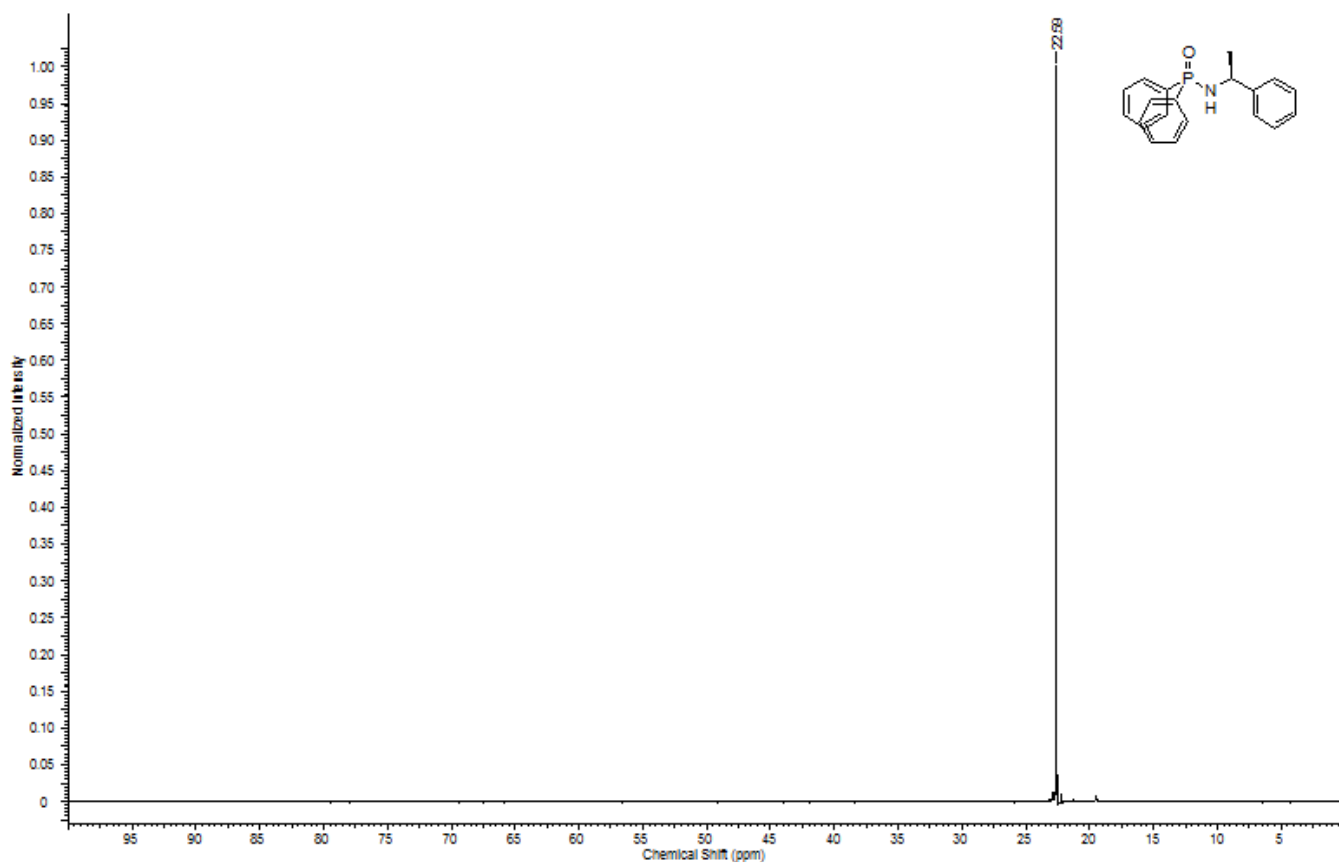
^{31}P NMR spectra of diphenylphosphinous acid-borane (*S*)- α -methylbenzylamide (**10a**) (126 MHz, CDCl_3).



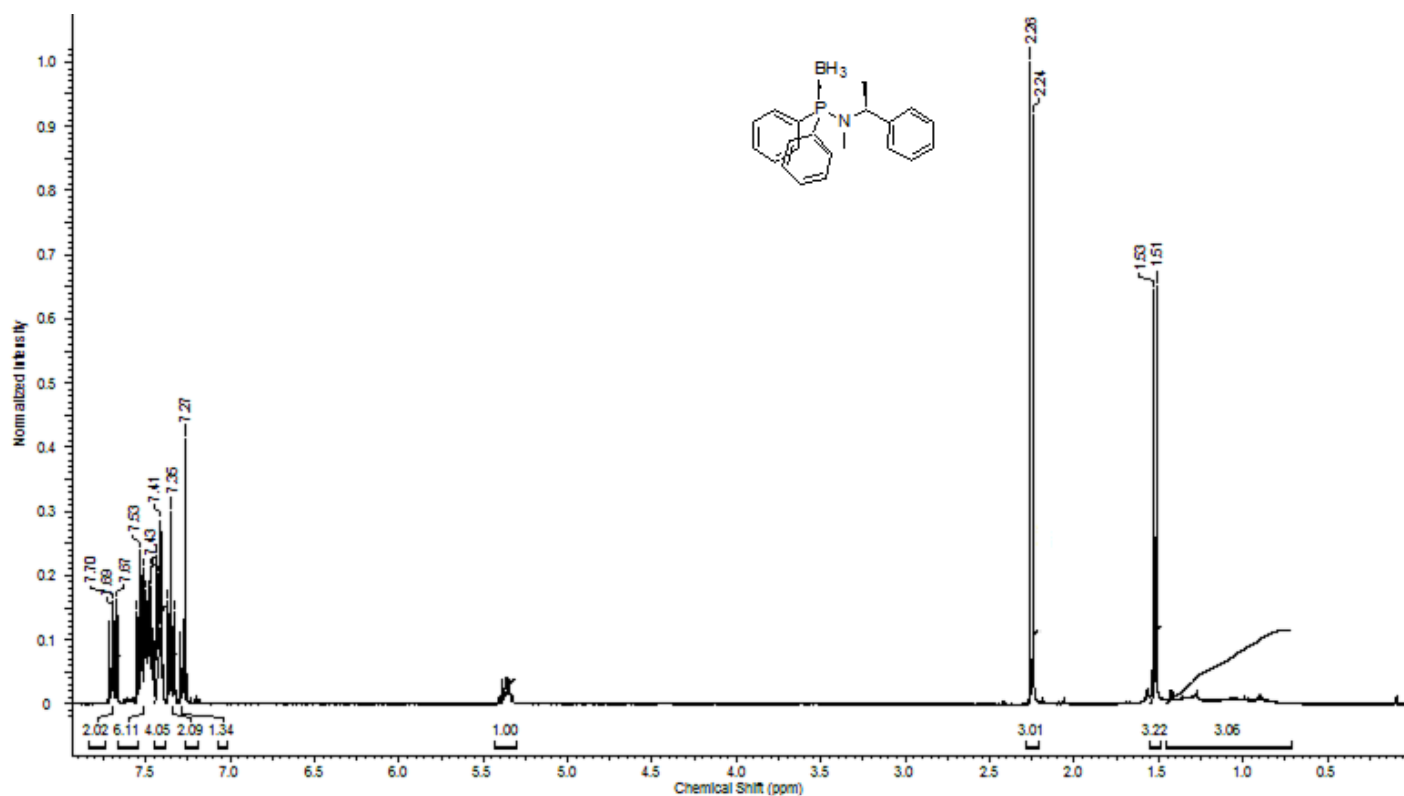
^1H NMR spectra of diphenylphosphinic acid (*S*)- α -methylbenzylamide (**11a**) (500 MHz, CDCl_3).



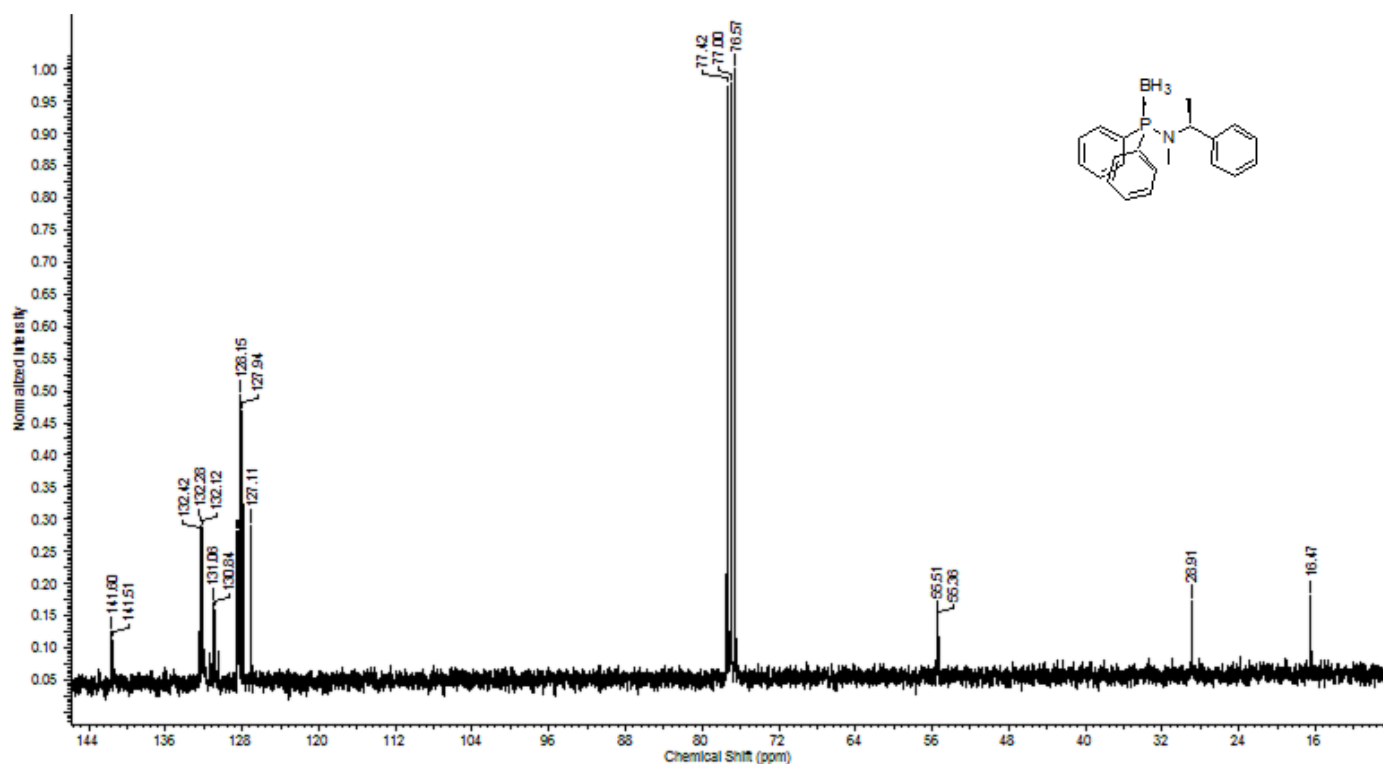
¹³C NMR spectra of diphenylphosphinic acid (*S*)- α -methylbenzylamide (**11a**) (126 MHz, CDCl₃).



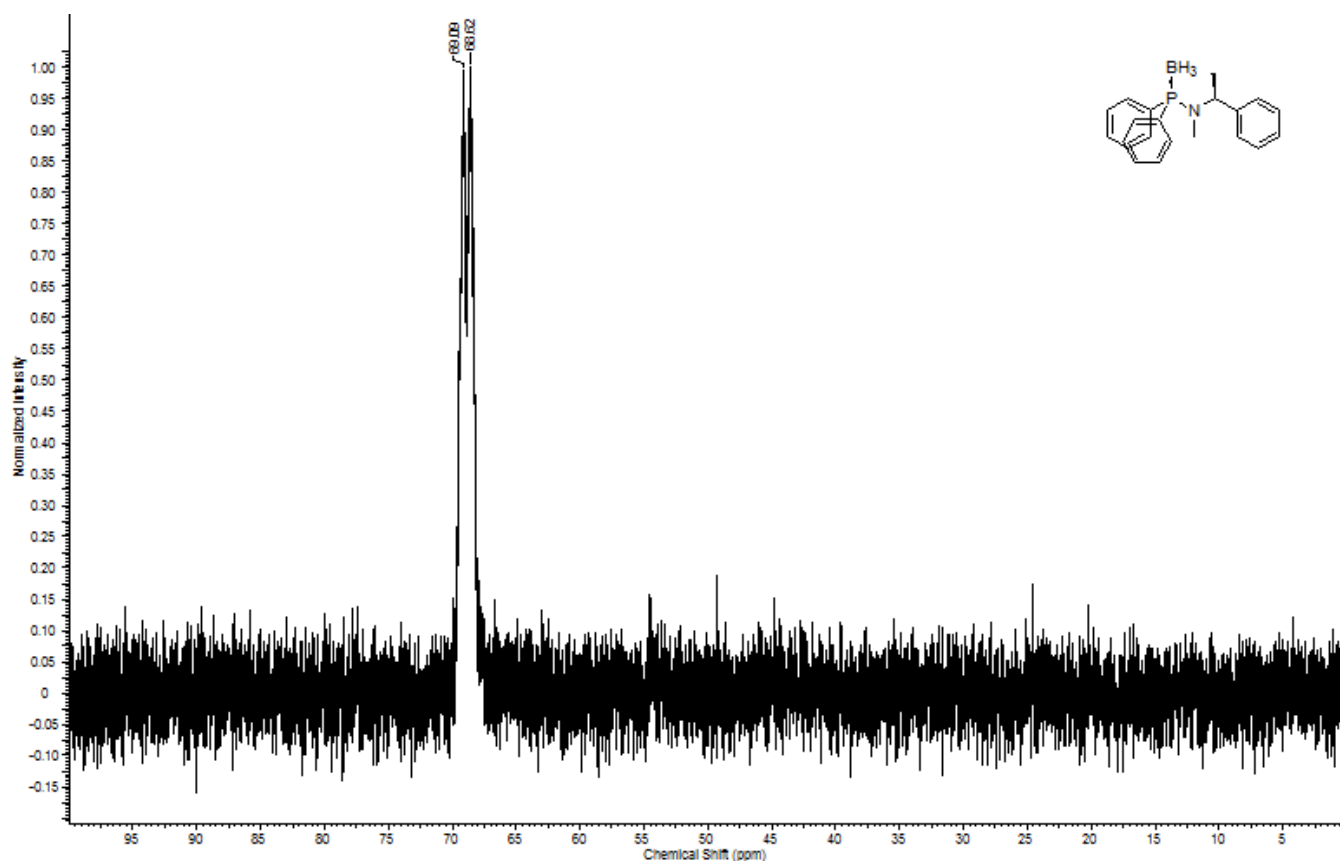
³¹P NMR spectra of diphenylphosphinic acid (*S*)- α -methylbenzylamide (**11a**) (202 MHz, CDCl₃).



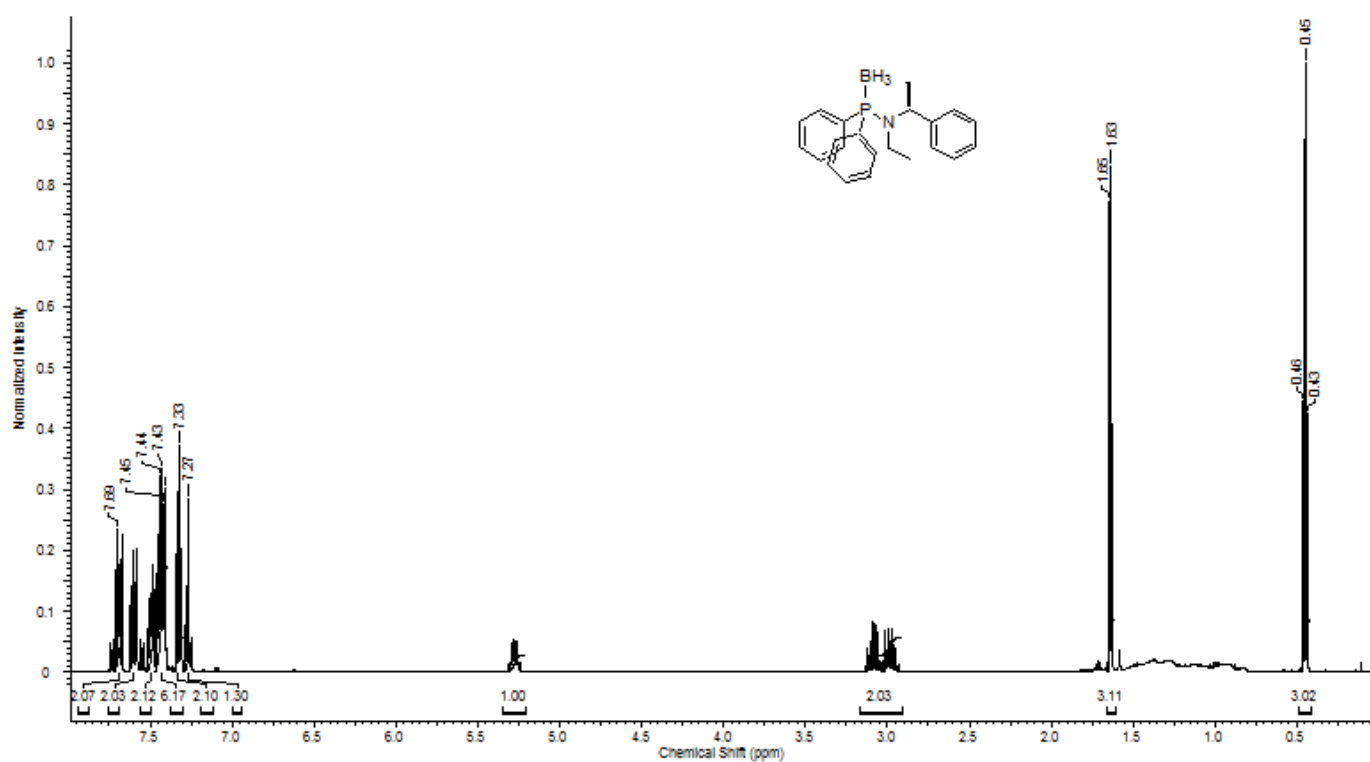
¹H NMR spectra of diphenylphosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**10b**) (400 MHz, CDCl₃).



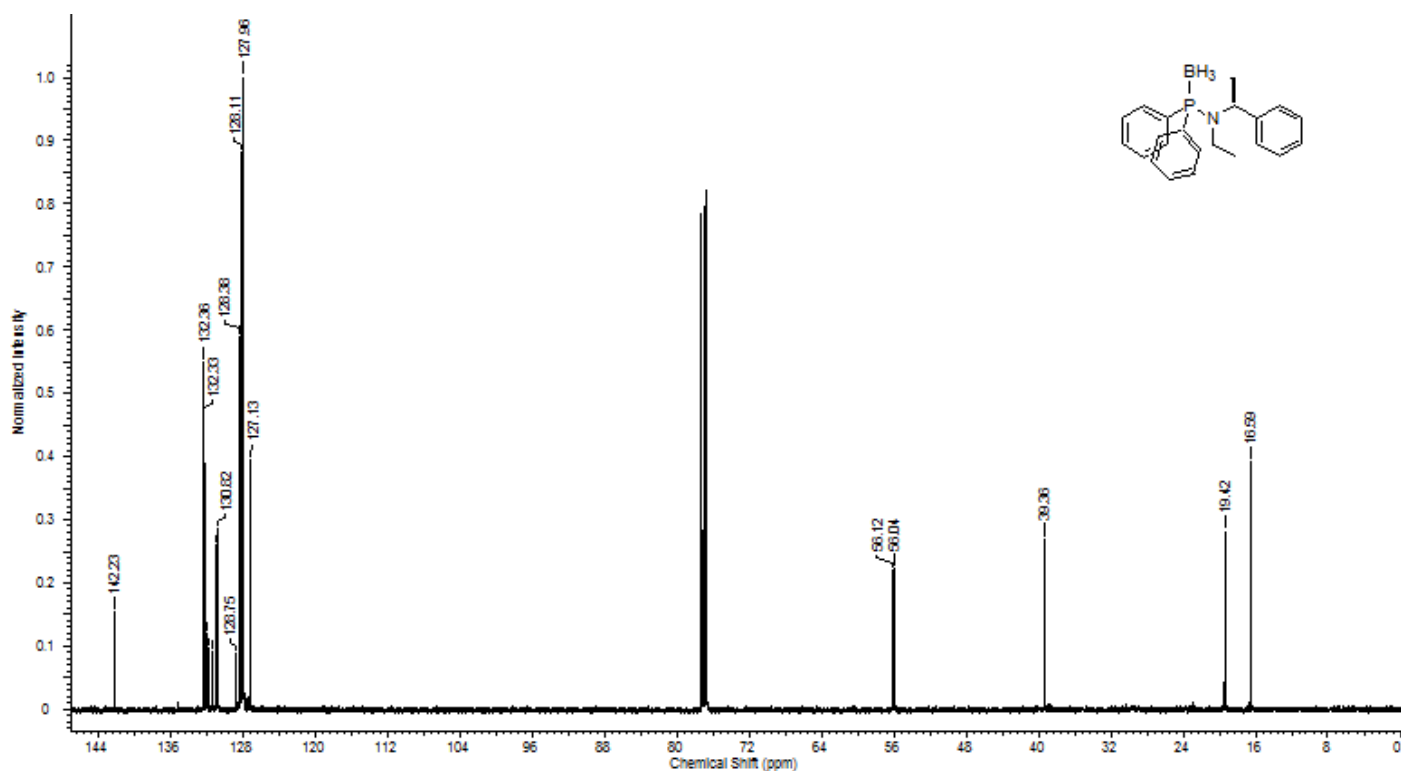
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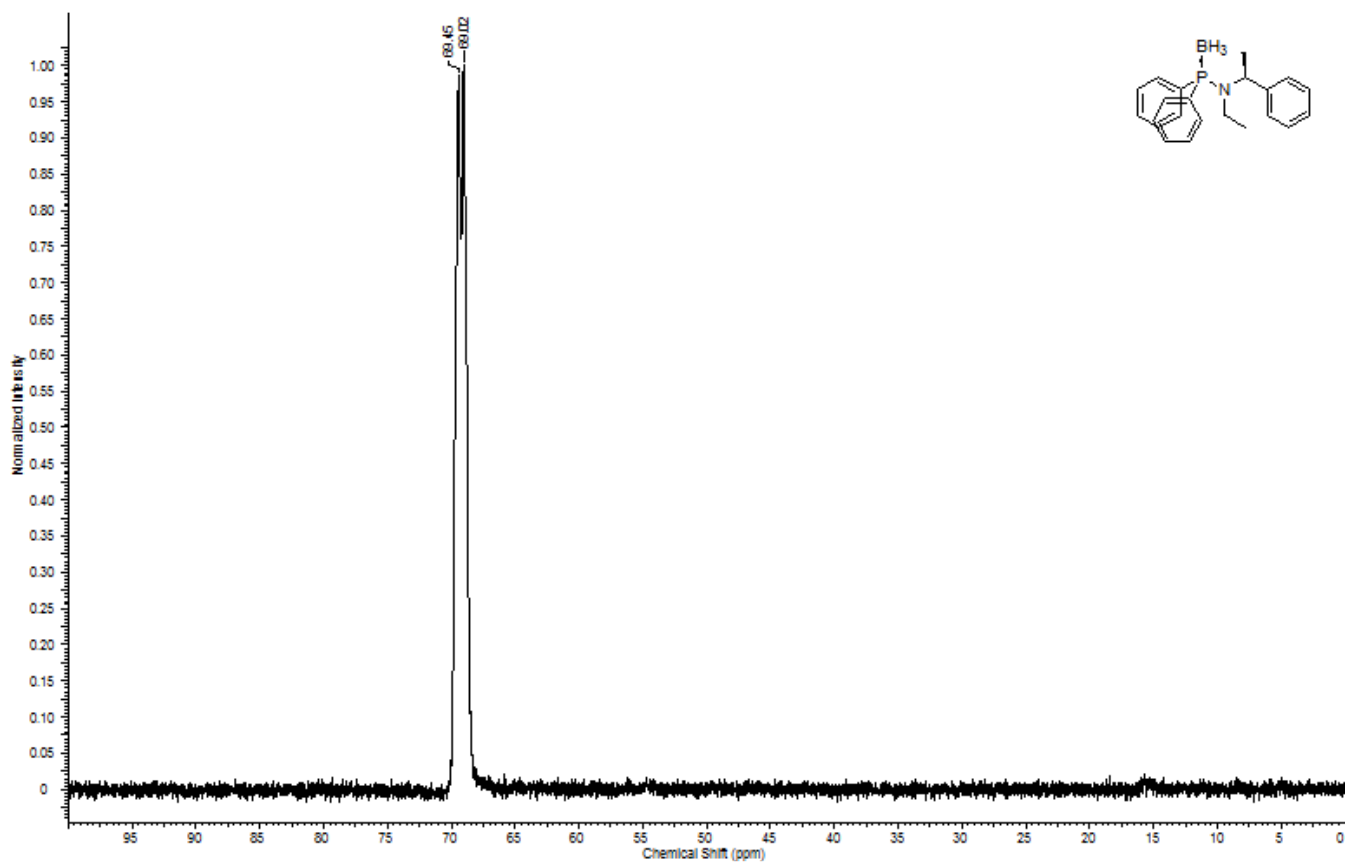
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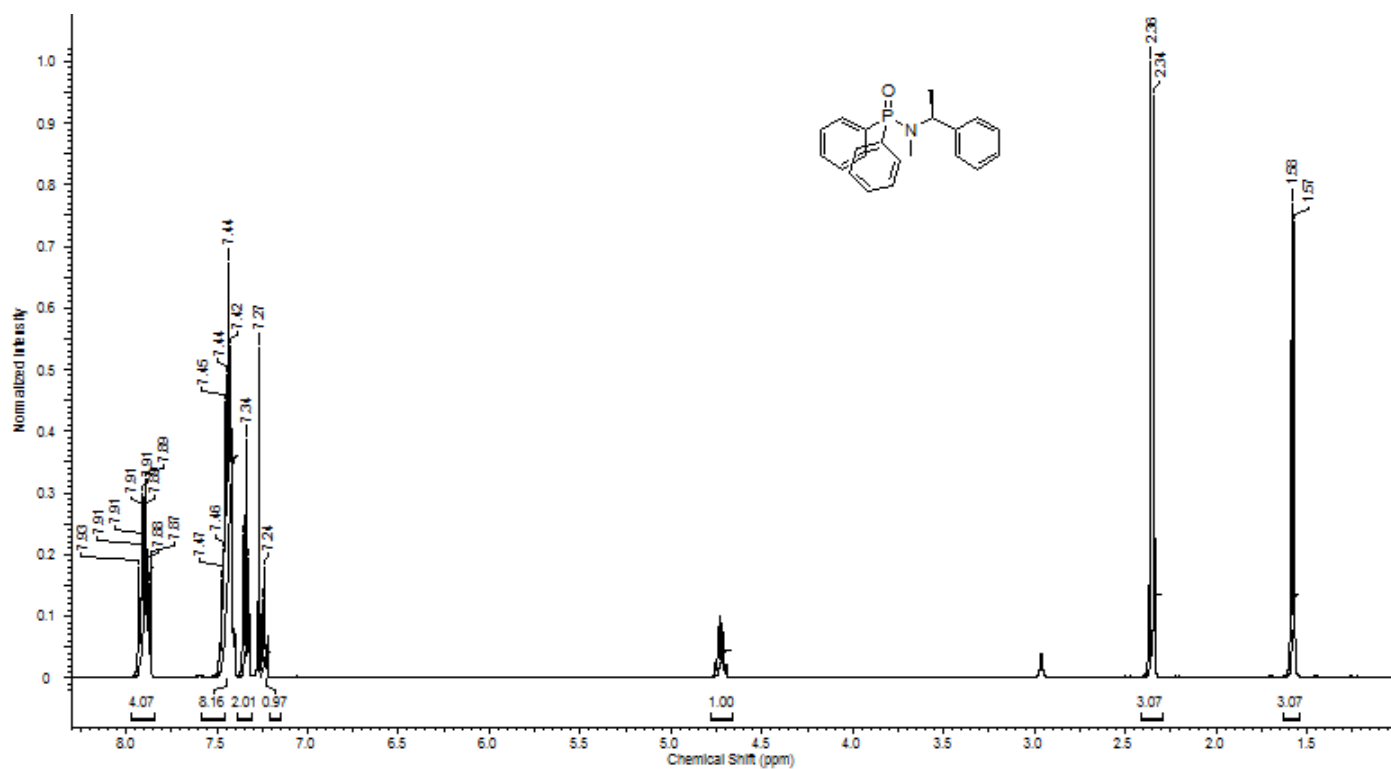
^1H NMR spectra of diphenylphosphinous acid-borane *N*-ethyl-(*S*)- α -methylbenzylamide (**10c**) (500 MHz, CDCl_3).



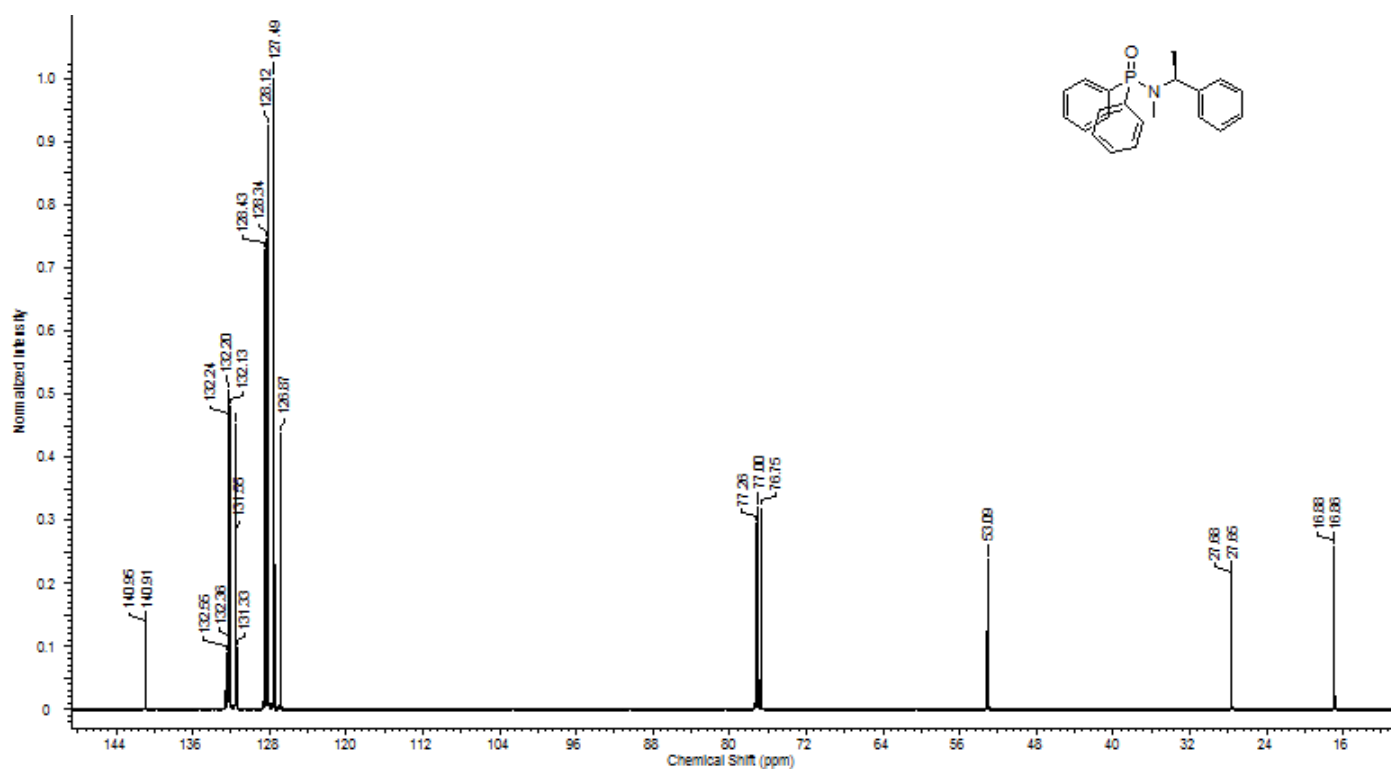
^{13}C NMR spectra of diphenylphosphinous acid-borane *N*-ethyl-(*S*)- α -methylbenzylamide (**10c**) (126 MHz, CDCl_3).



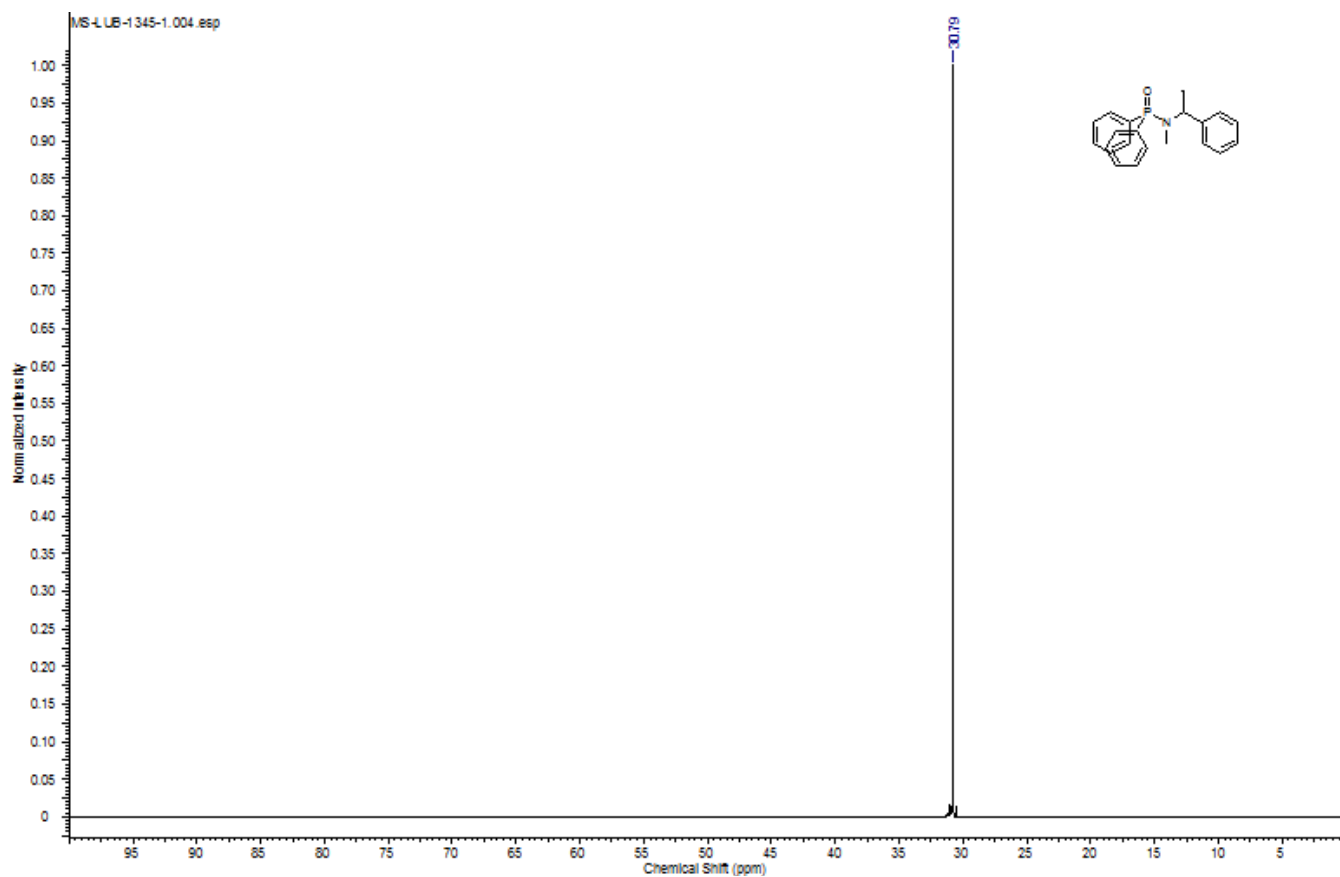
^{31}P NMR spectra of diphenylphosphinous acid-borane *N*-ethyl-(*S*)- α -methylbenzylamide (**10c**) (202 MHz, CDCl_3).



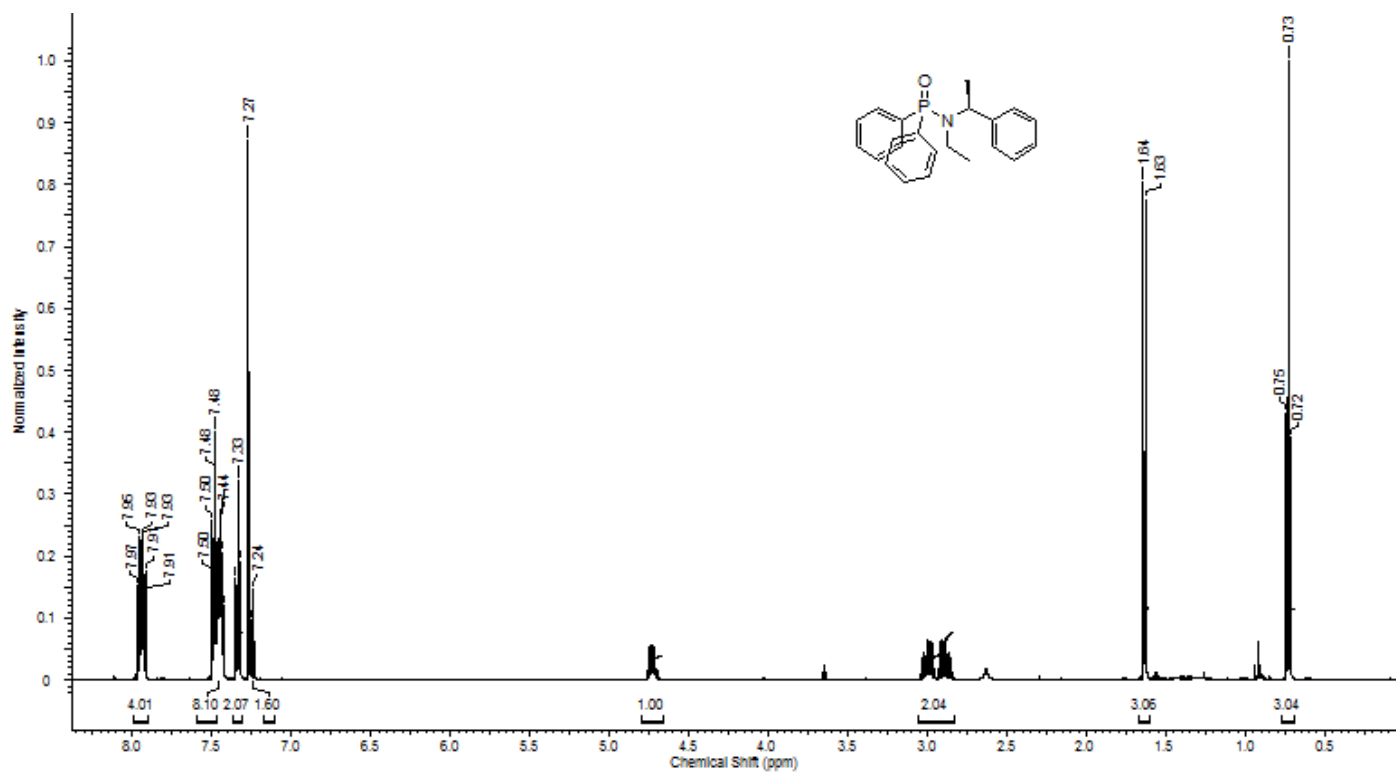
^1H NMR spectra of diphenylphosphinic acid *N*-methyl-(*S*)- α -methylbenzylamide (**11b**) (500 MHz, CDCl_3).



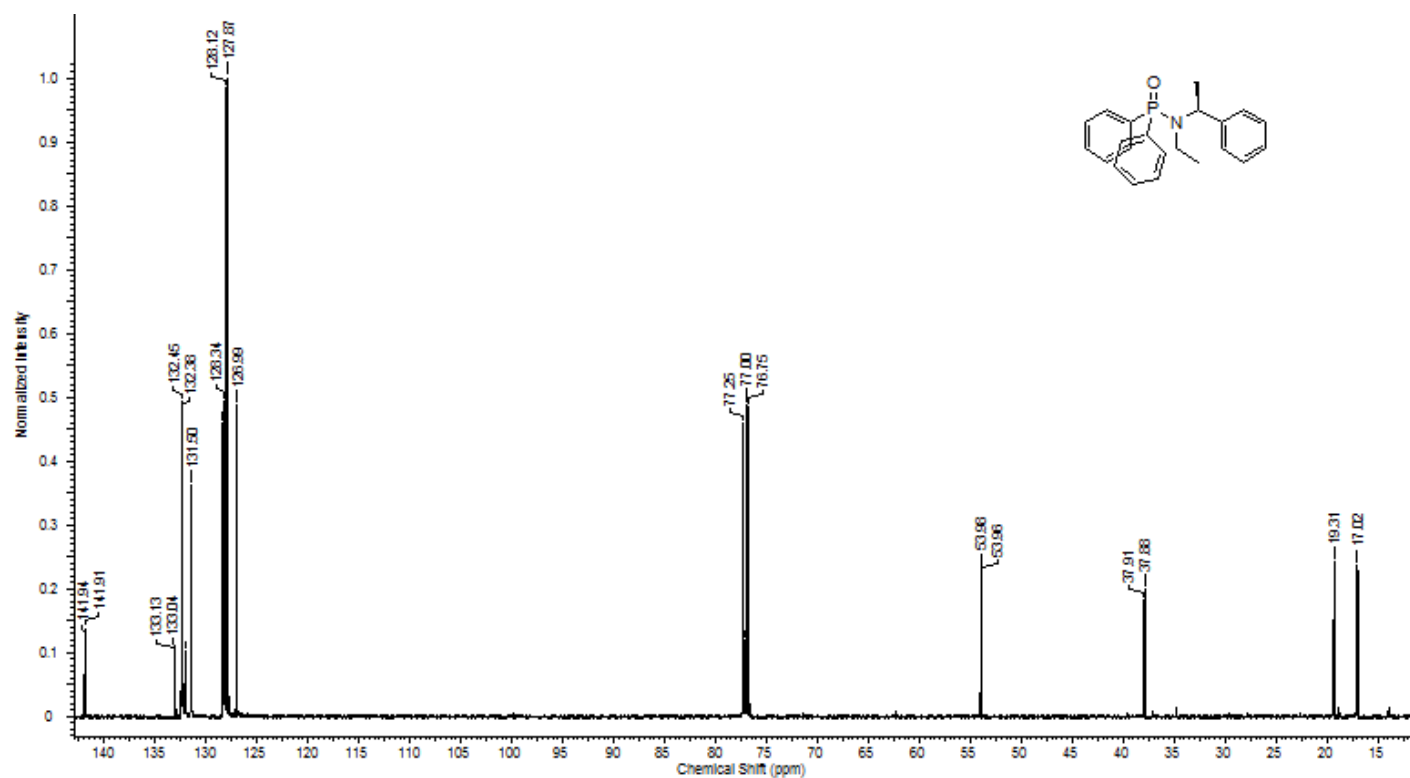
^{13}C NMR spectra of diphenylphosphinic acid *N*-methyl-(*S*)- α -methylbenzylamide (**11b**) (126 MHz, CDCl_3).



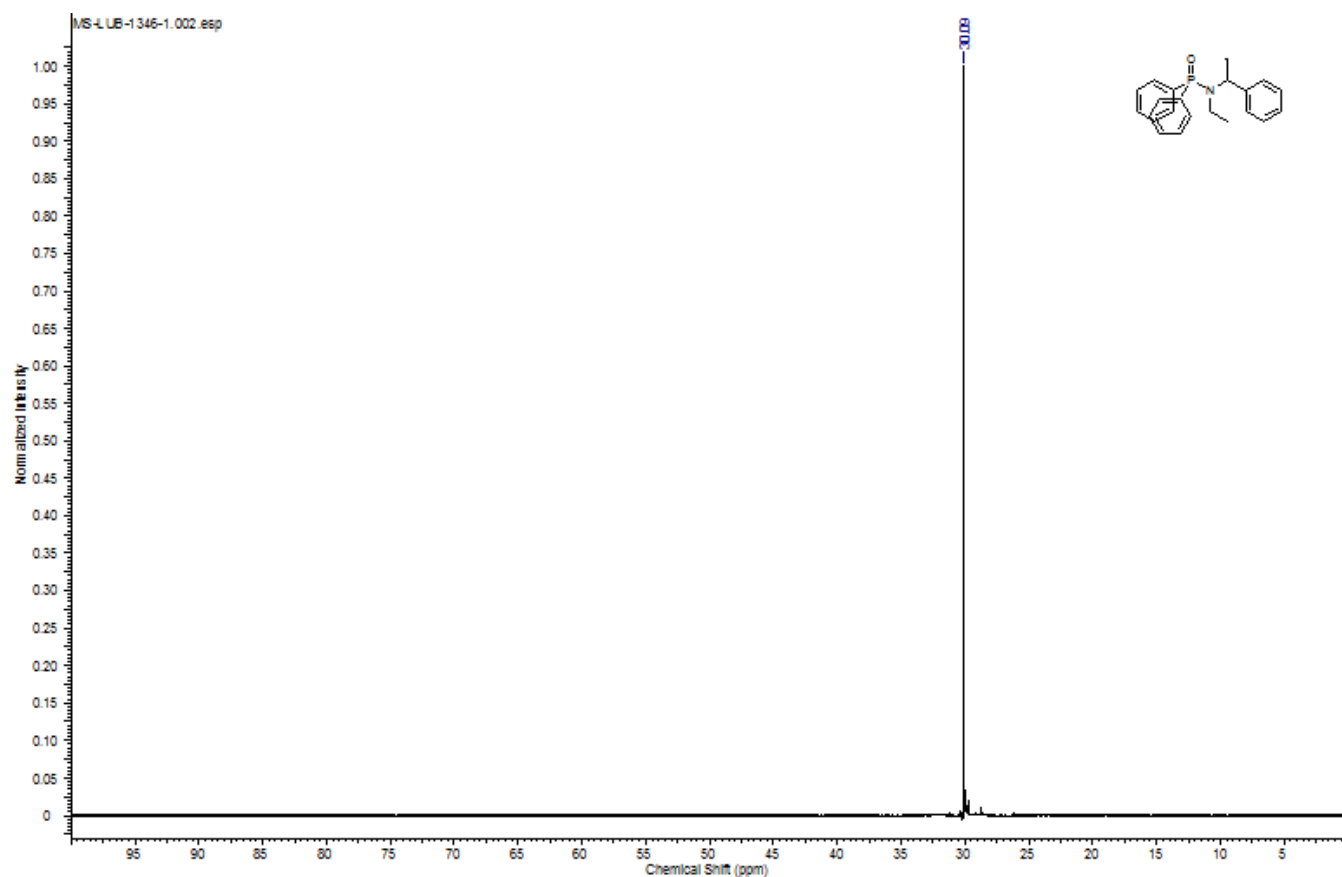
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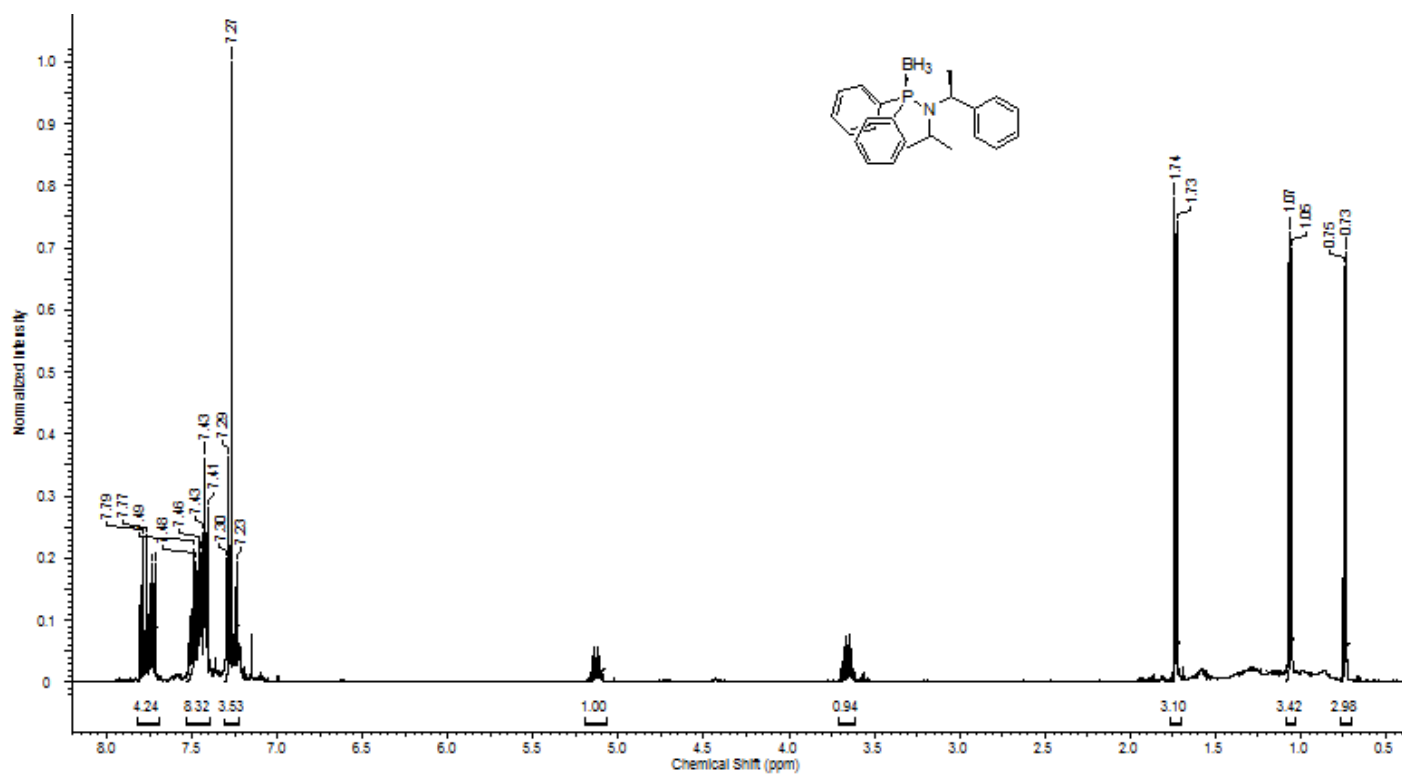
^1H NMR spectra of diphenylphosphinic acid *N*-ethyl-(*S*)- α -methylbenzylamide (**11c**) (500 MHz, CDCl_3).



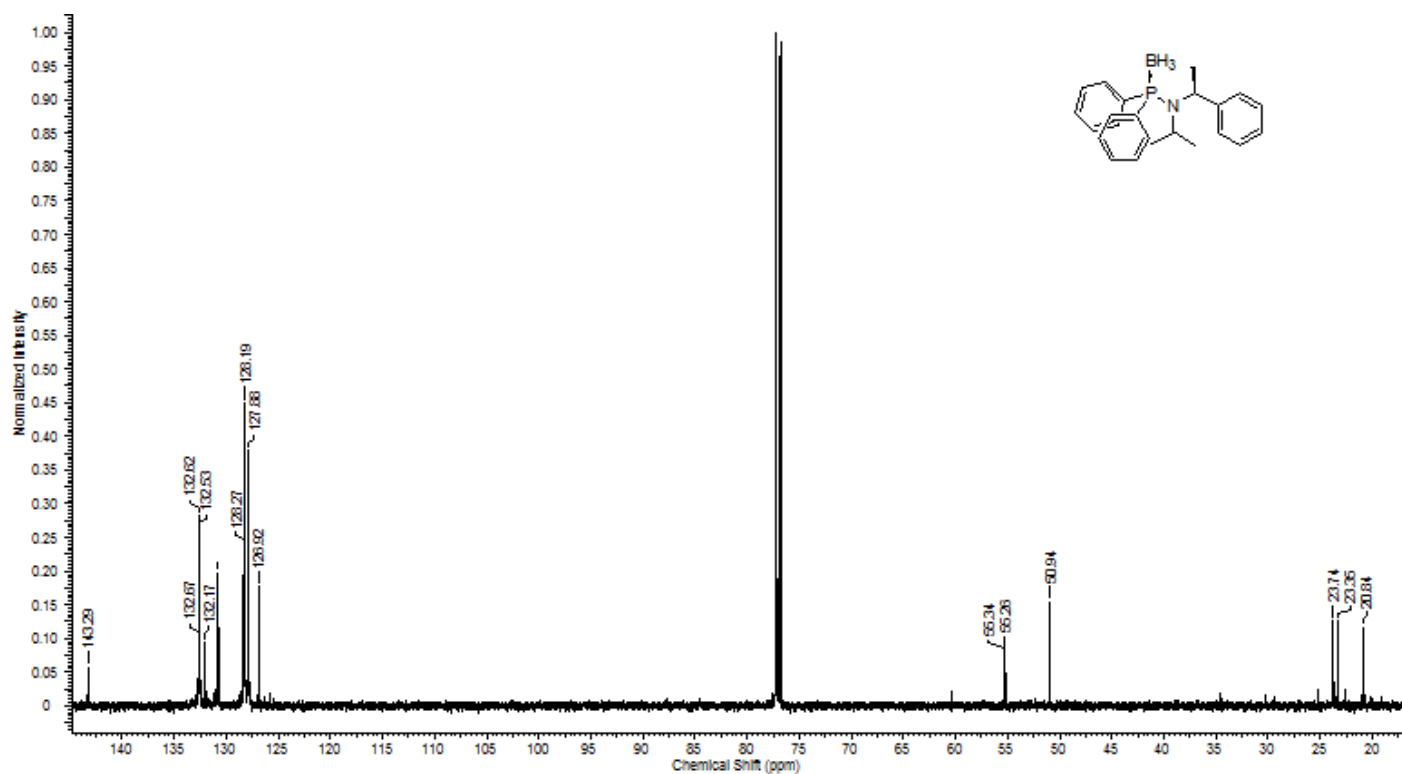
^{13}C NMR spectra of diphenylphosphinic acid *N*-ethyl-(*S*)- α -methylbenzylamide (**11b**) (126 MHz, CDCl_3).



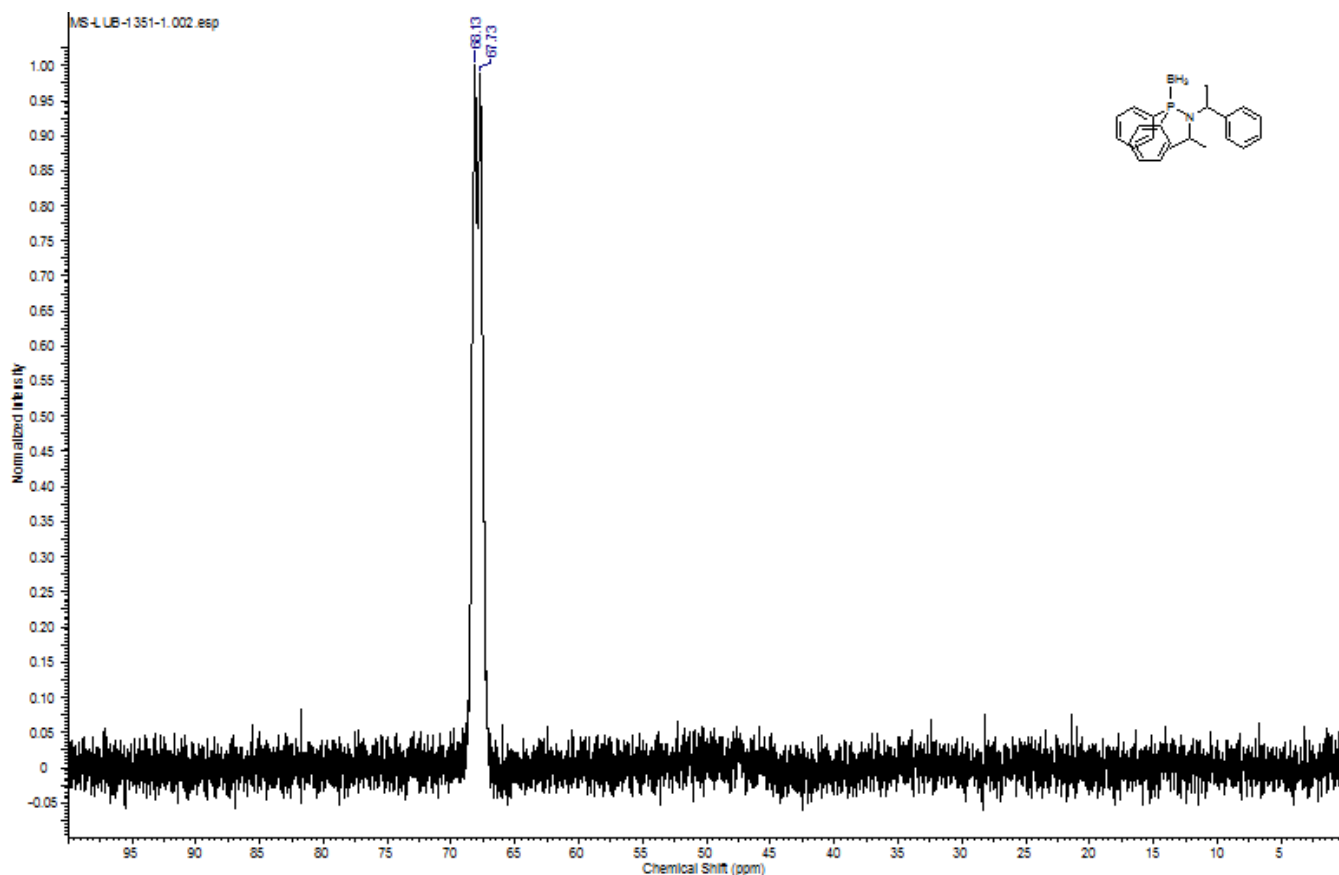
^{31}P NMR spectra of diphenylphosphinic acid *N*-ethyl-(*S*)- α -methylbenzylamide (**11b**) (202 MHz, CDCl_3).



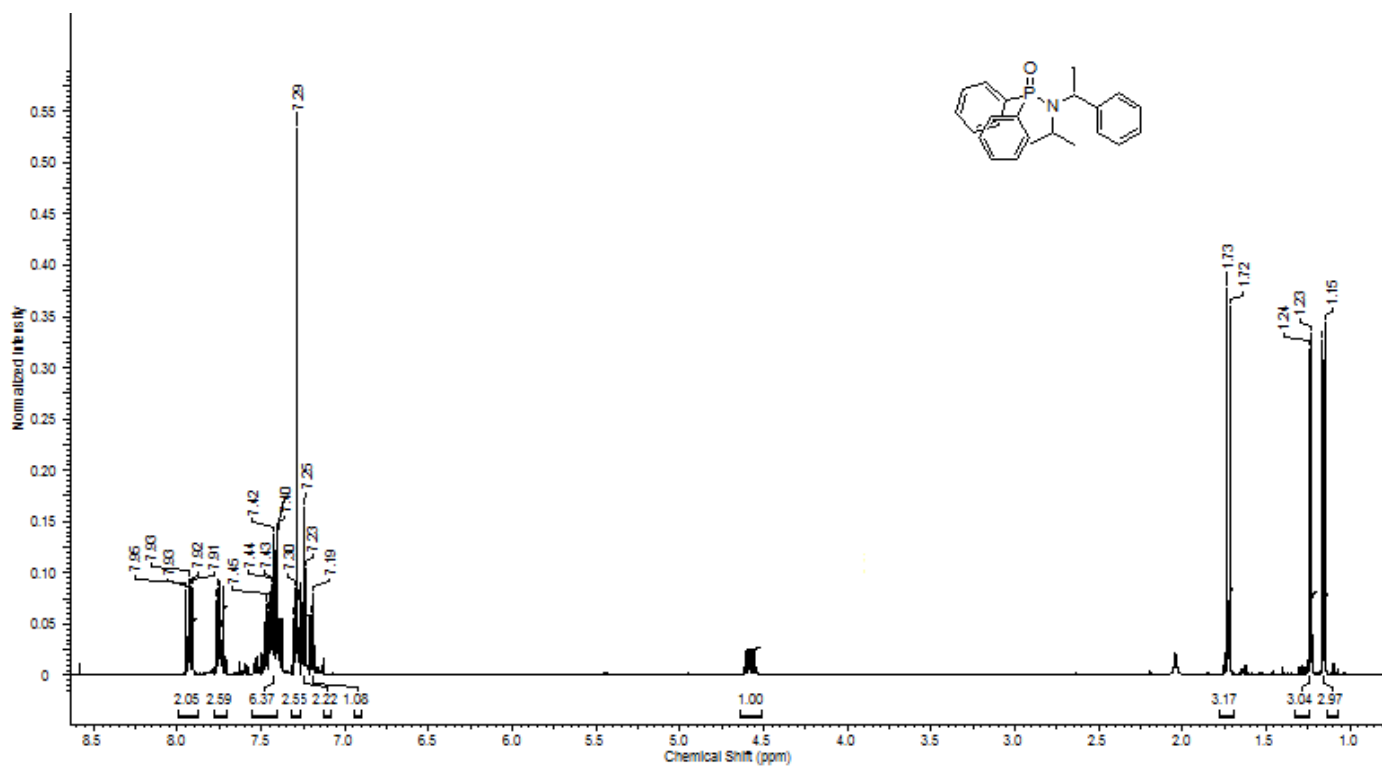
¹H NMR spectra of diphenylphosphinous acid-borane *N*-isopropyl-(*S*)- α -methylbenzylamide (**10d**) (500 MHz, CDCl₃).



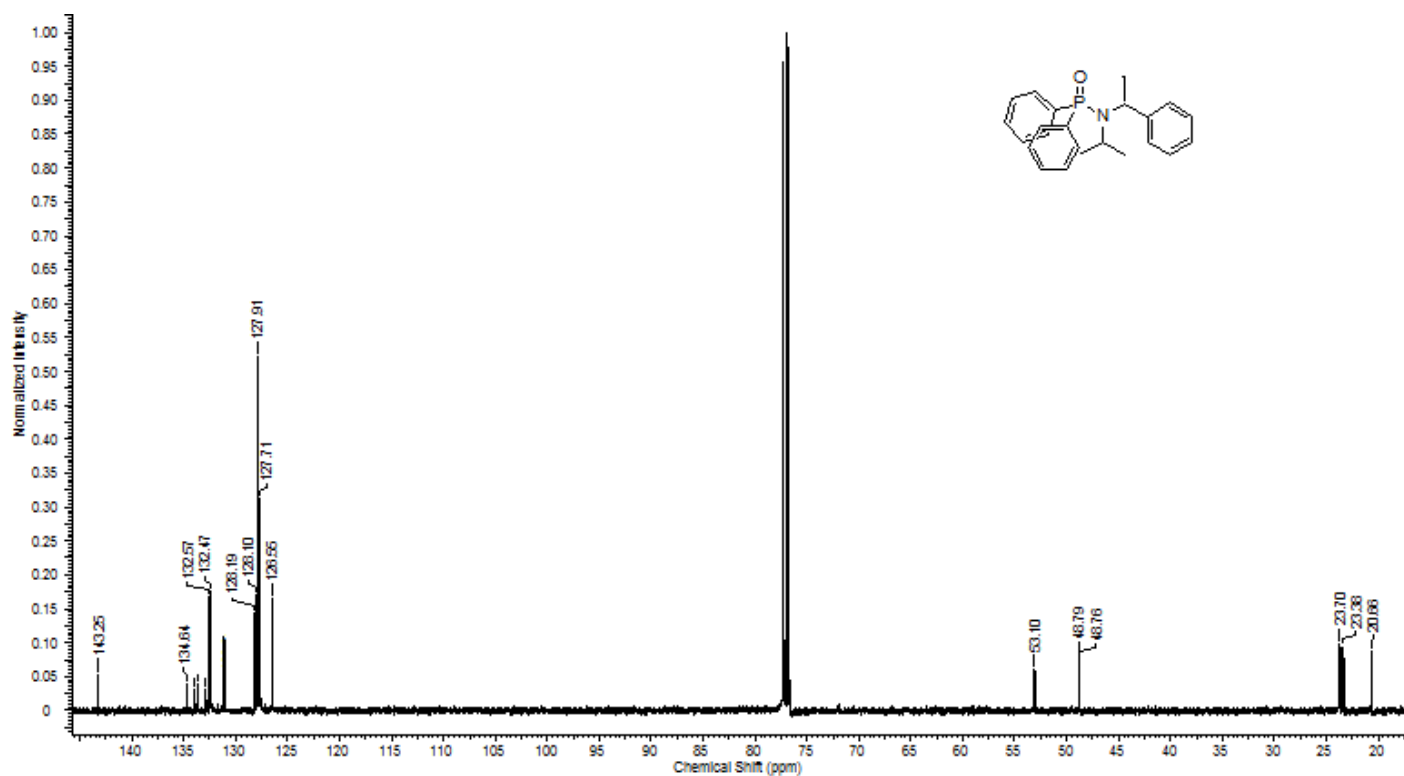
¹³C NMR spectra of diphenylphosphinous acid-borane *N*-isopropyl-(*S*)- α -methylbenzylamide (**10d**) (126 MHz, CDCl₃).



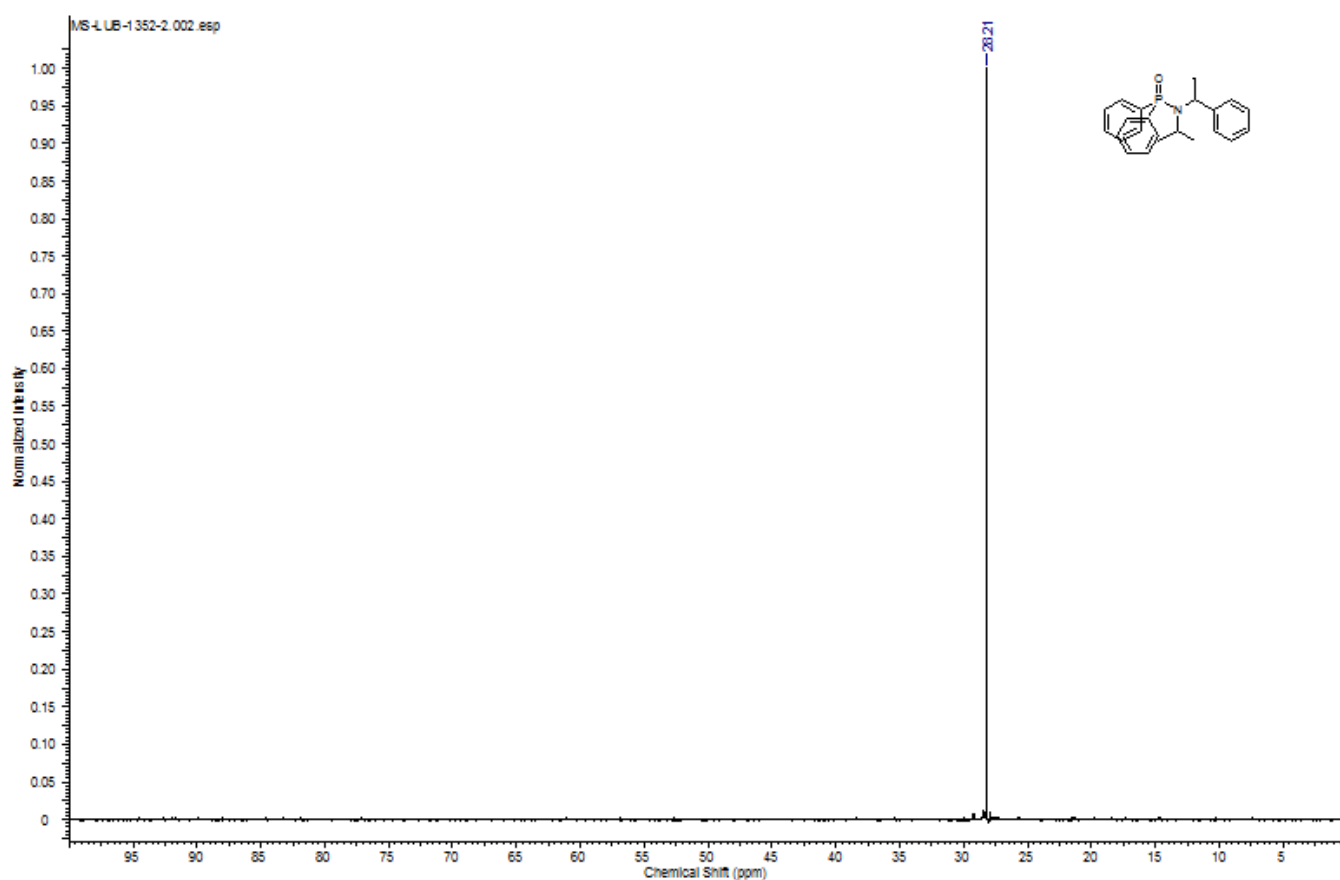
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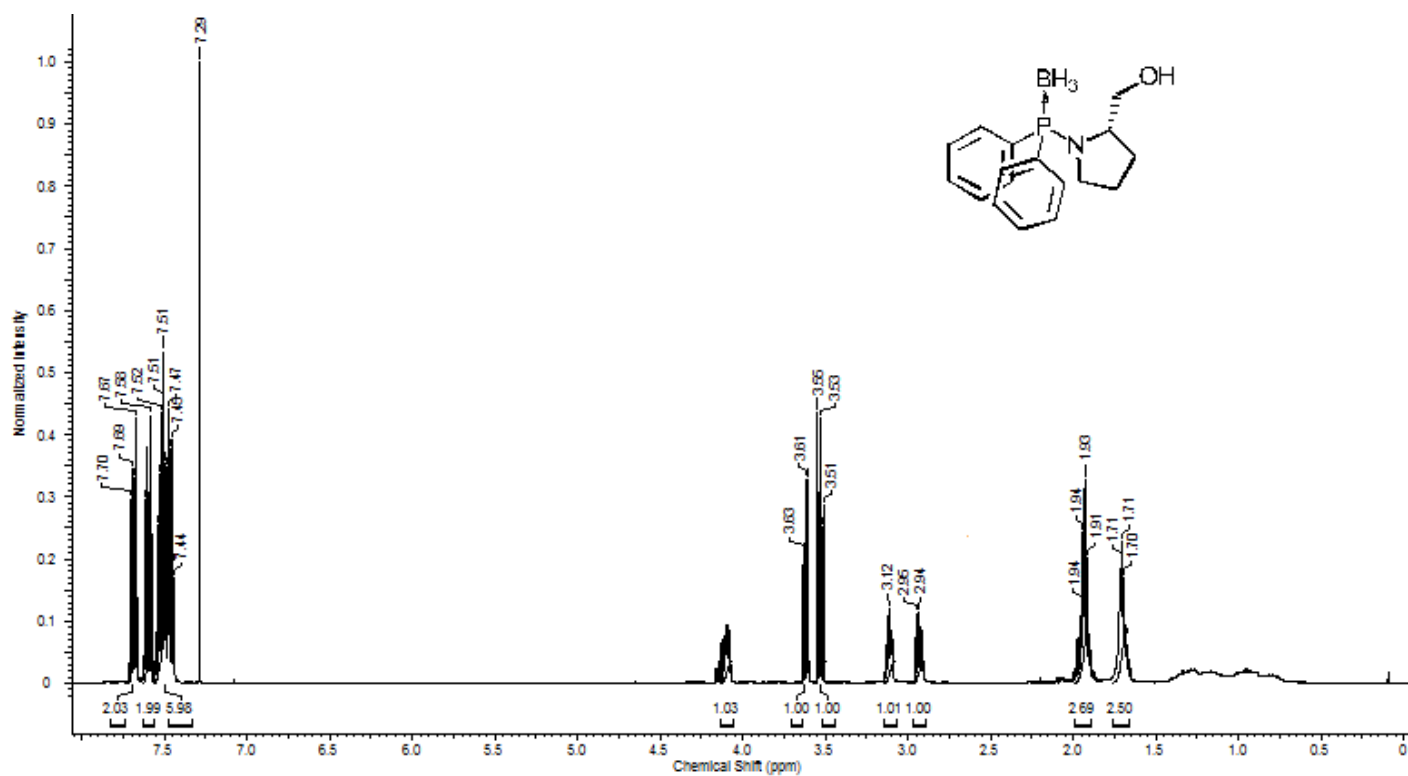
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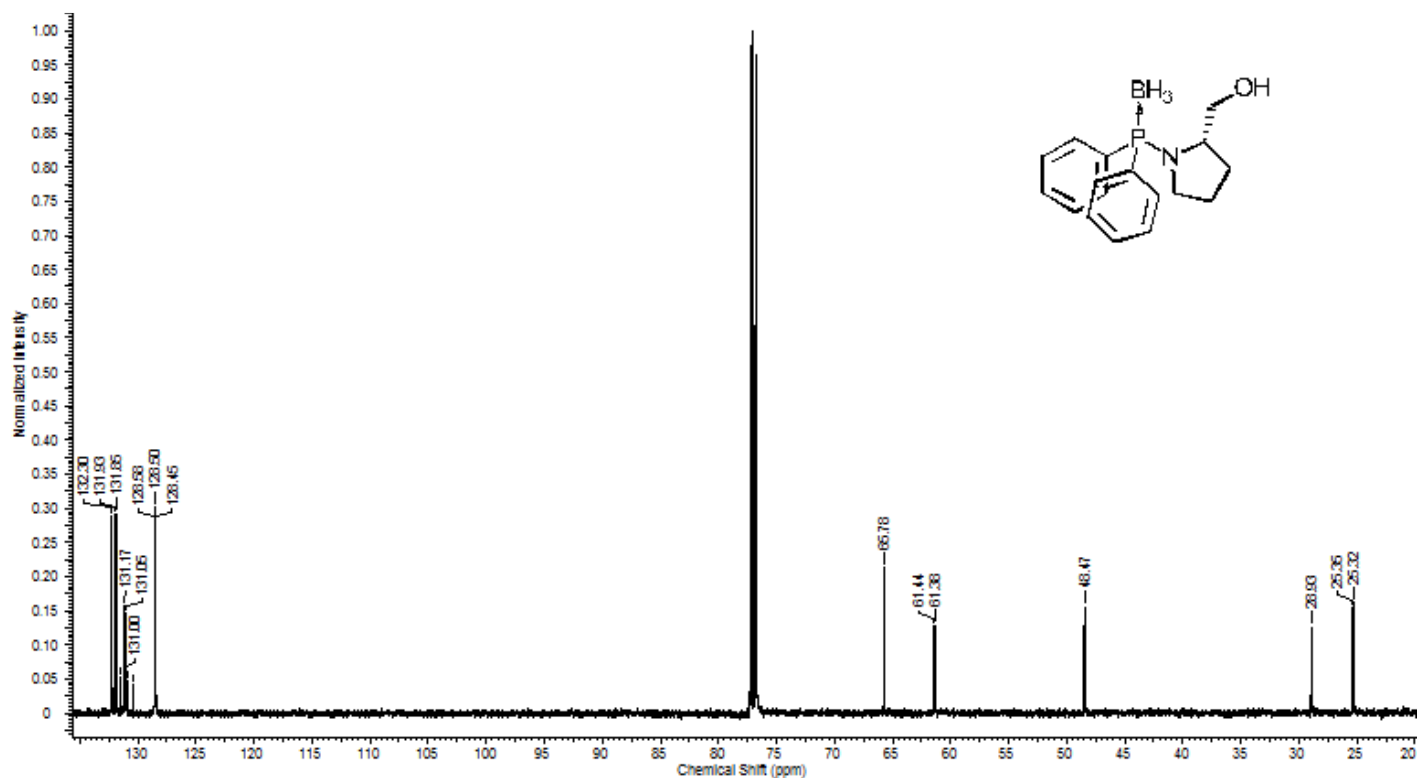
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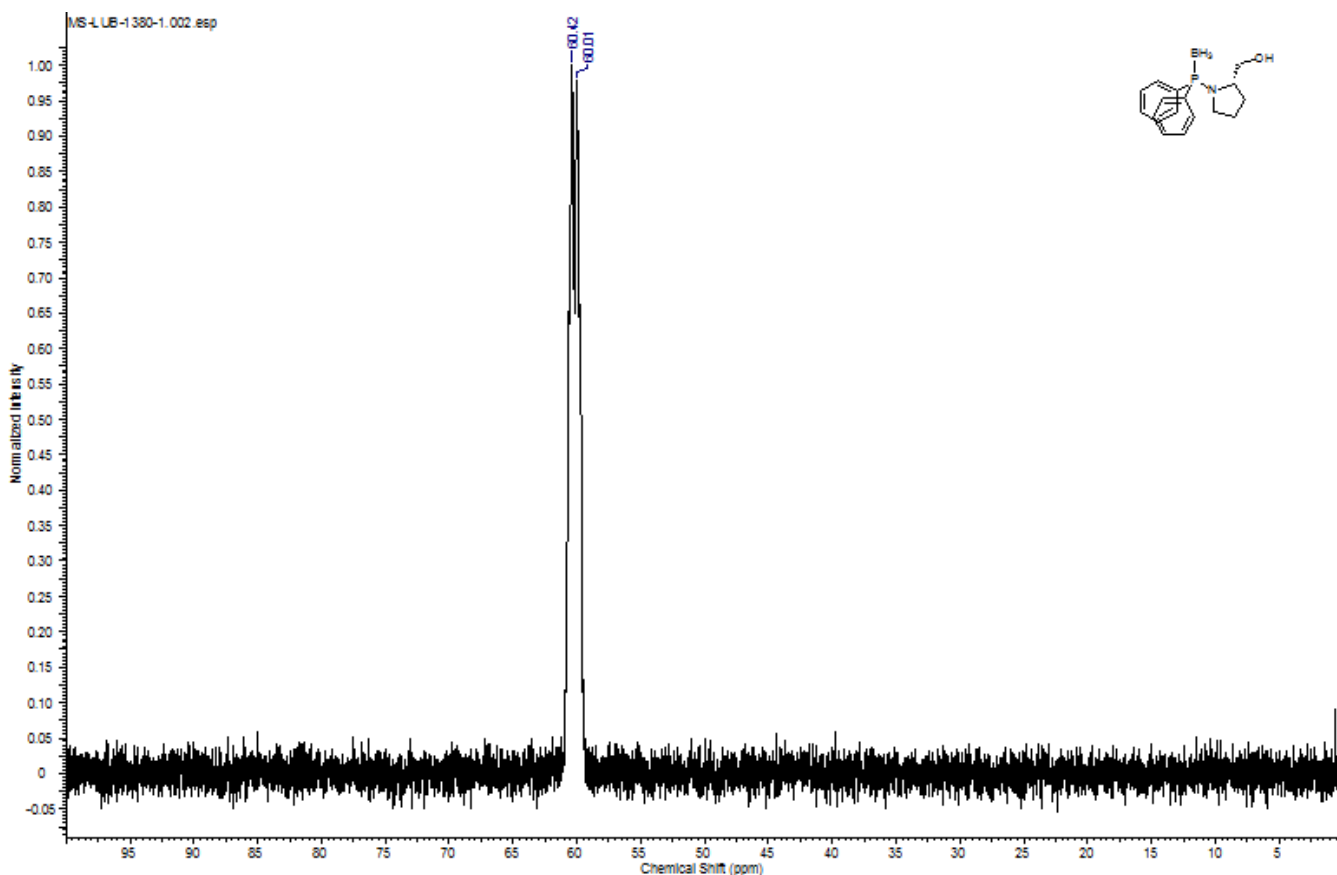
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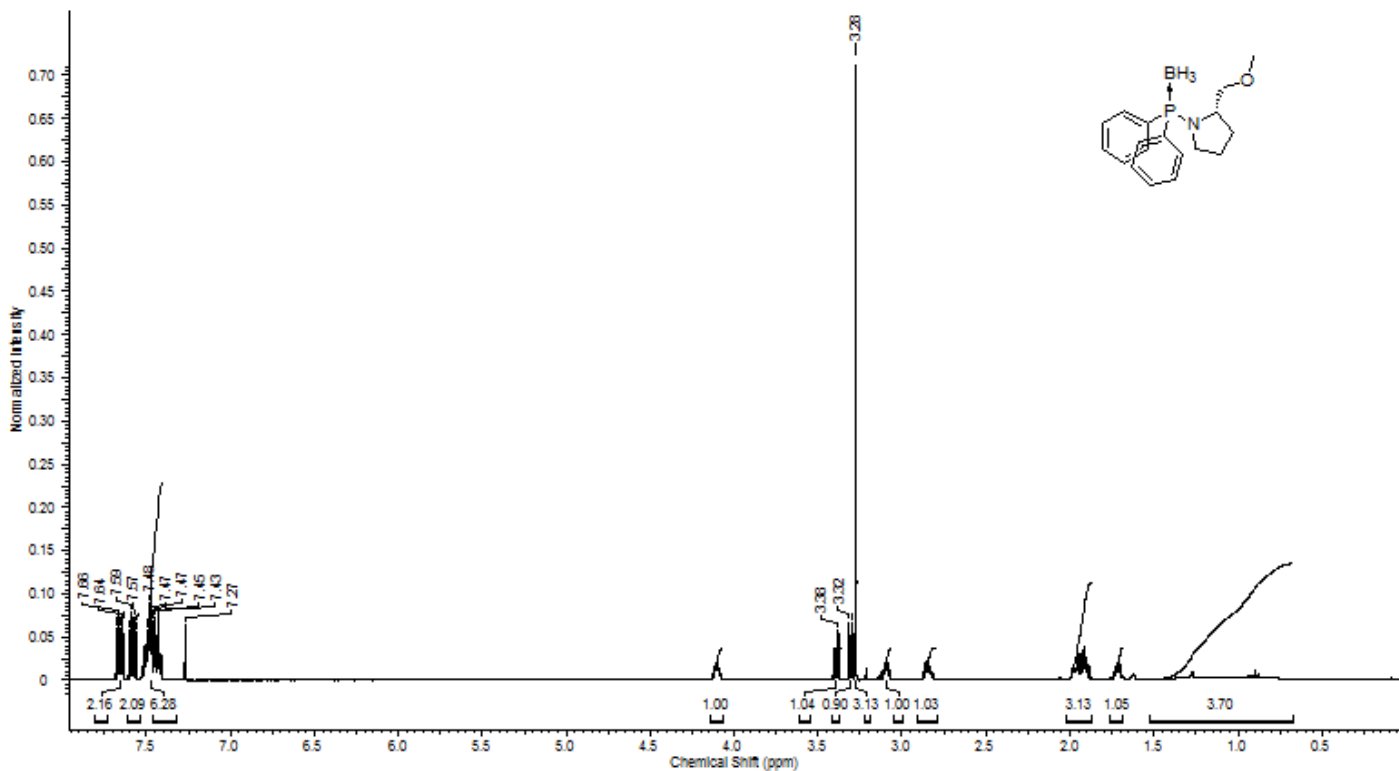
¹H NMR spectra of Diphenylphosphinous acid-borane (2*S*)-2-(hydroxymethyl)pyrrolidinamide (**16a**) (500 MHz, CDCl₃)



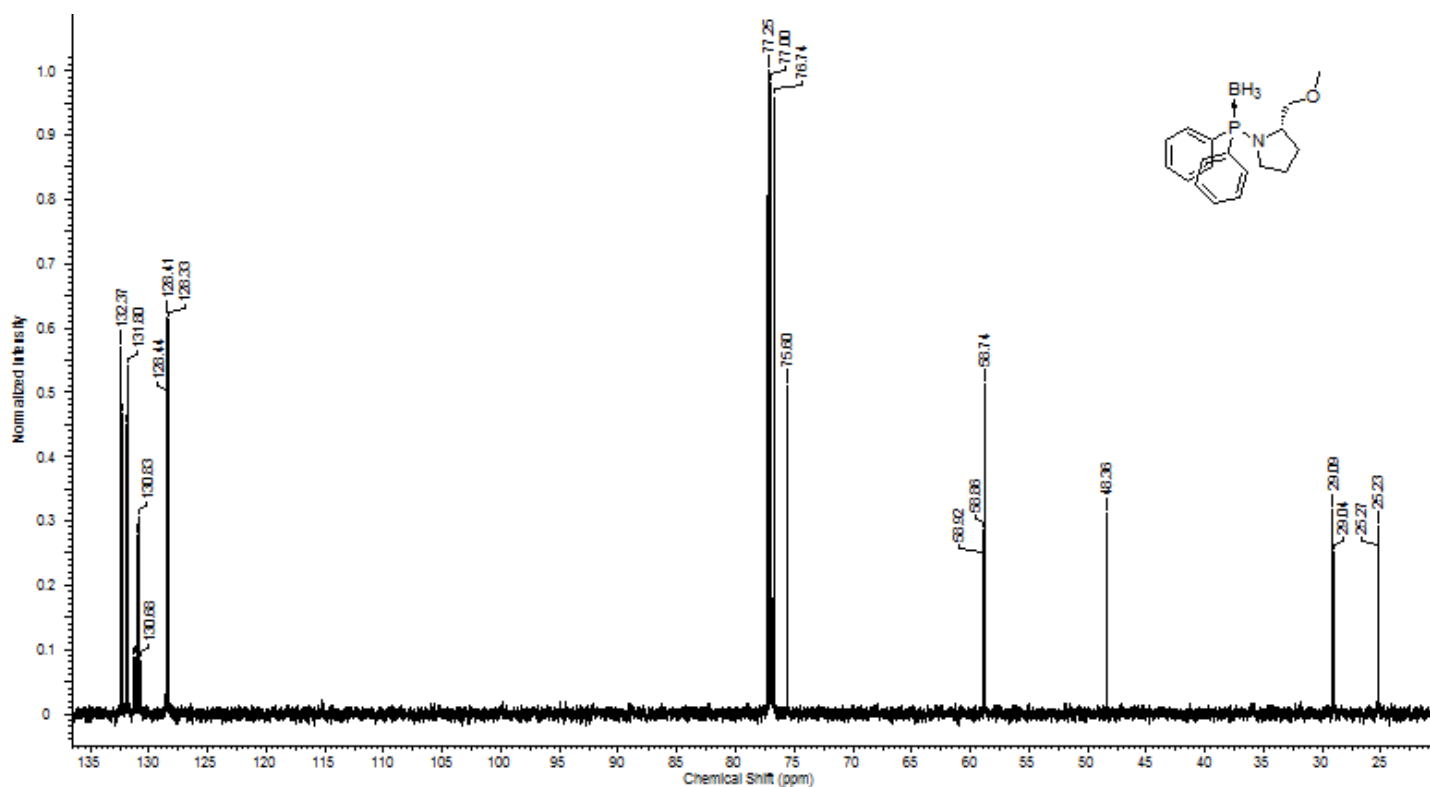
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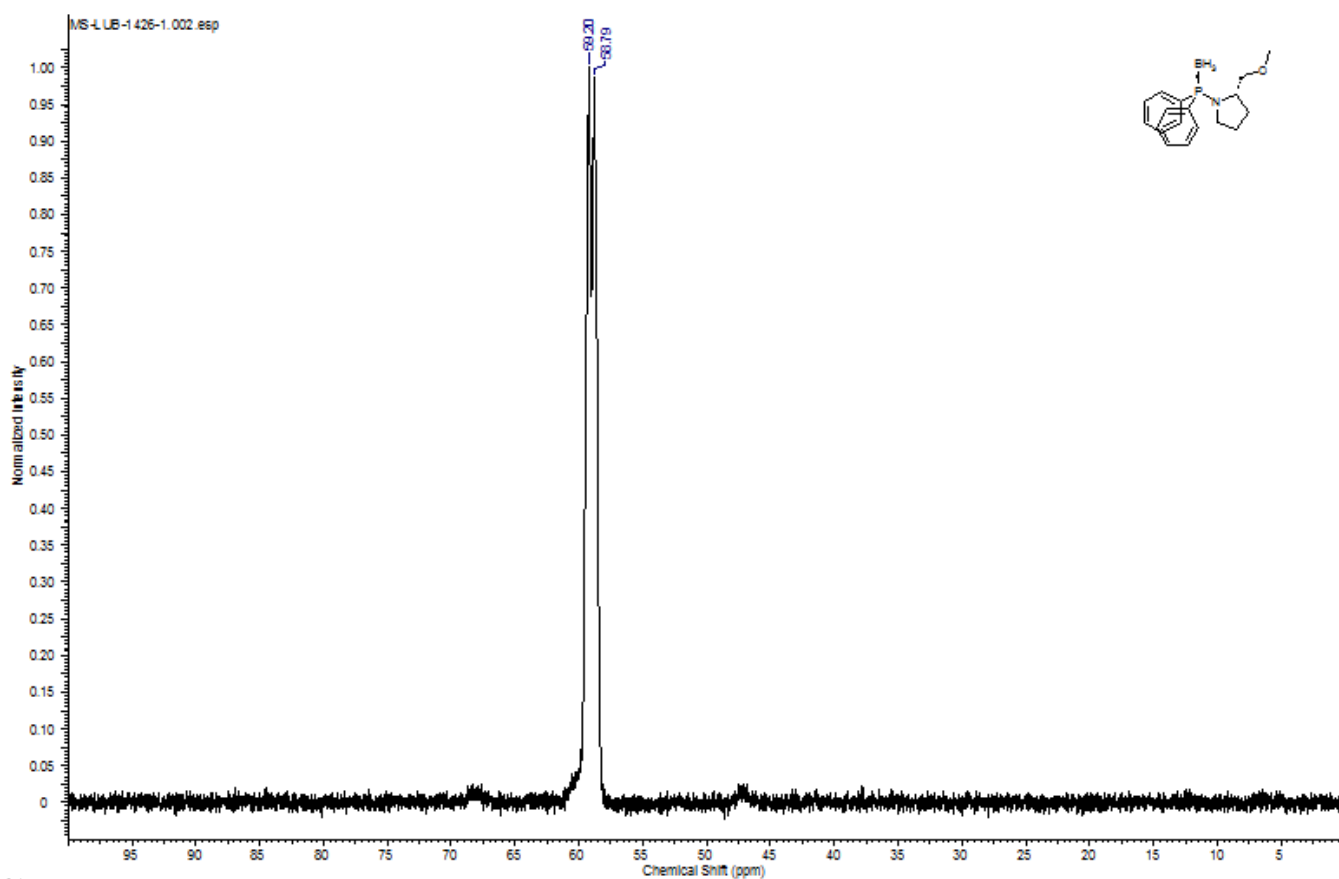
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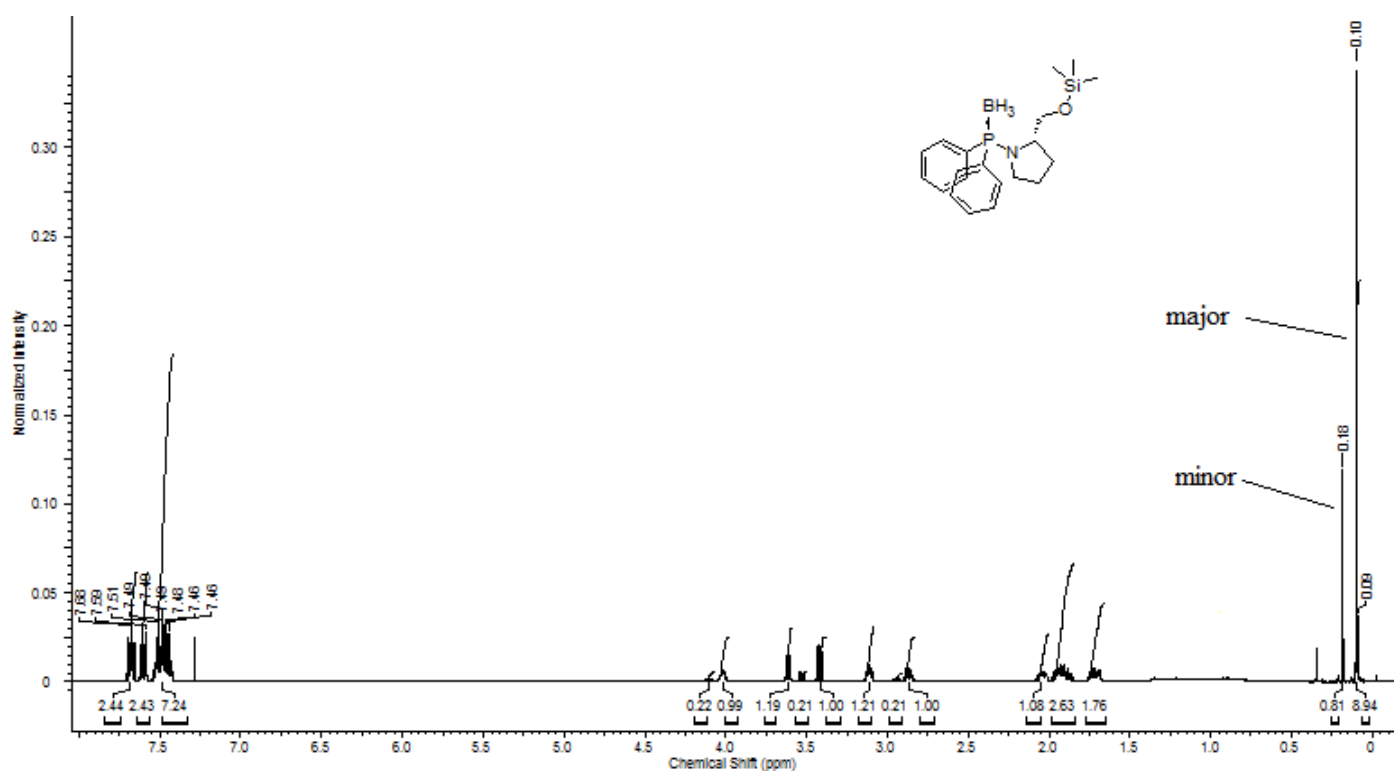
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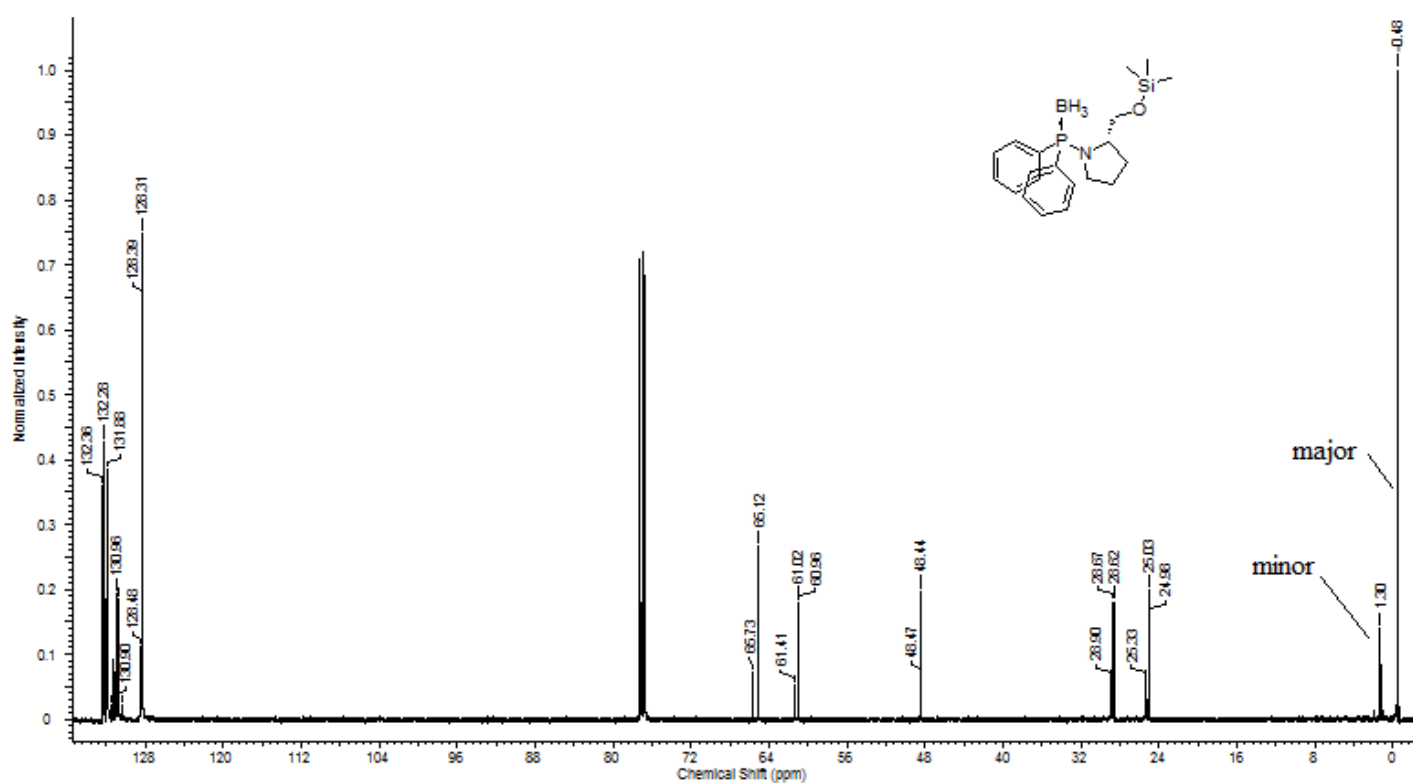
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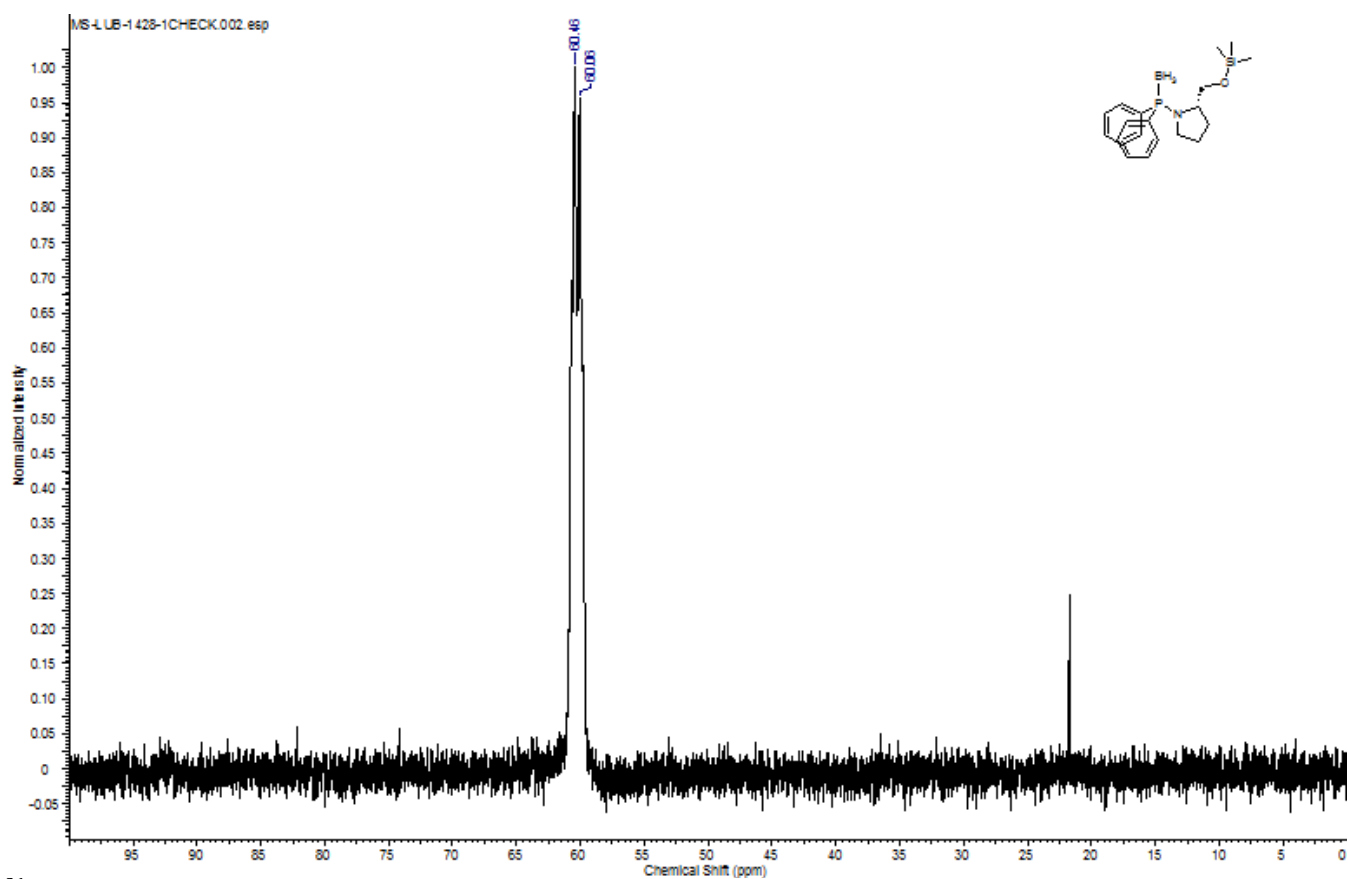
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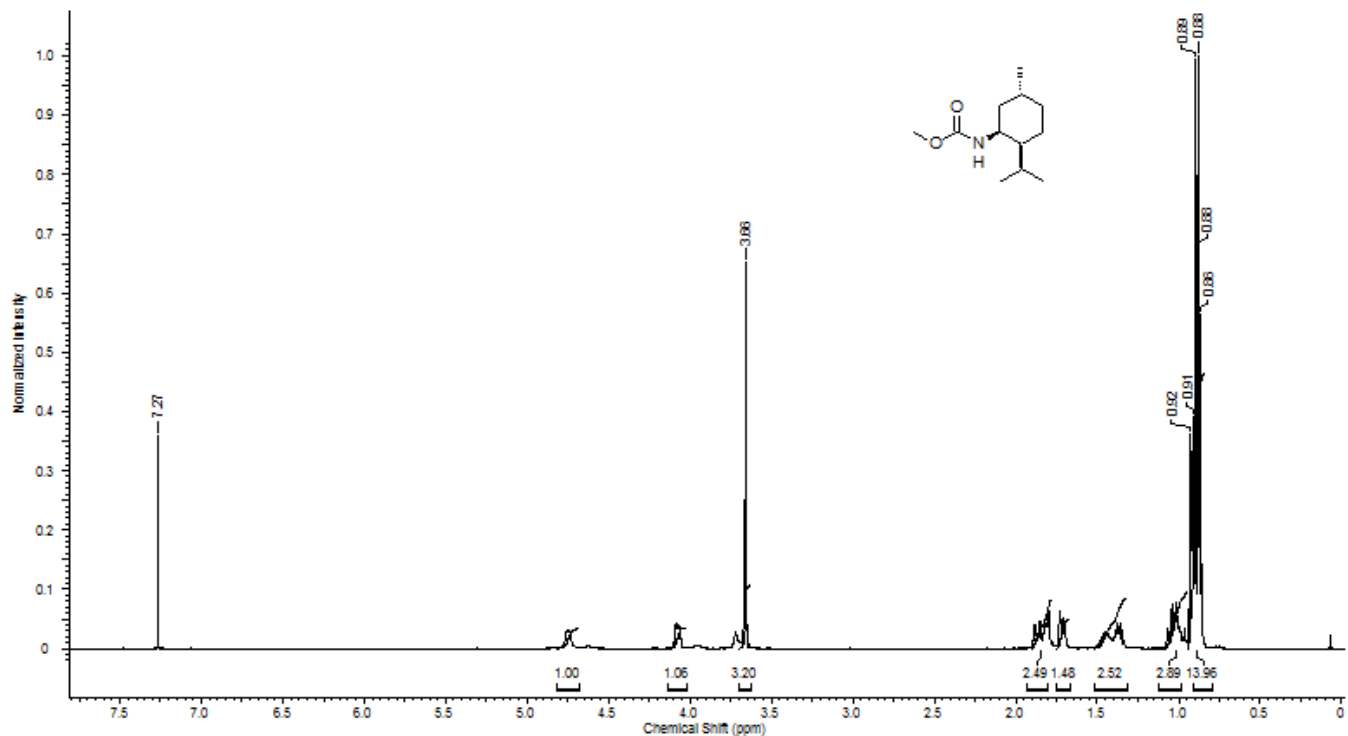
¹H NMR spectra of diphenylphosphinous acid-borane (2*S*)-2-(trimethylsilyloxymethyl)pyrolidinamide (**16c**) (500 MHz, CDCl₃)



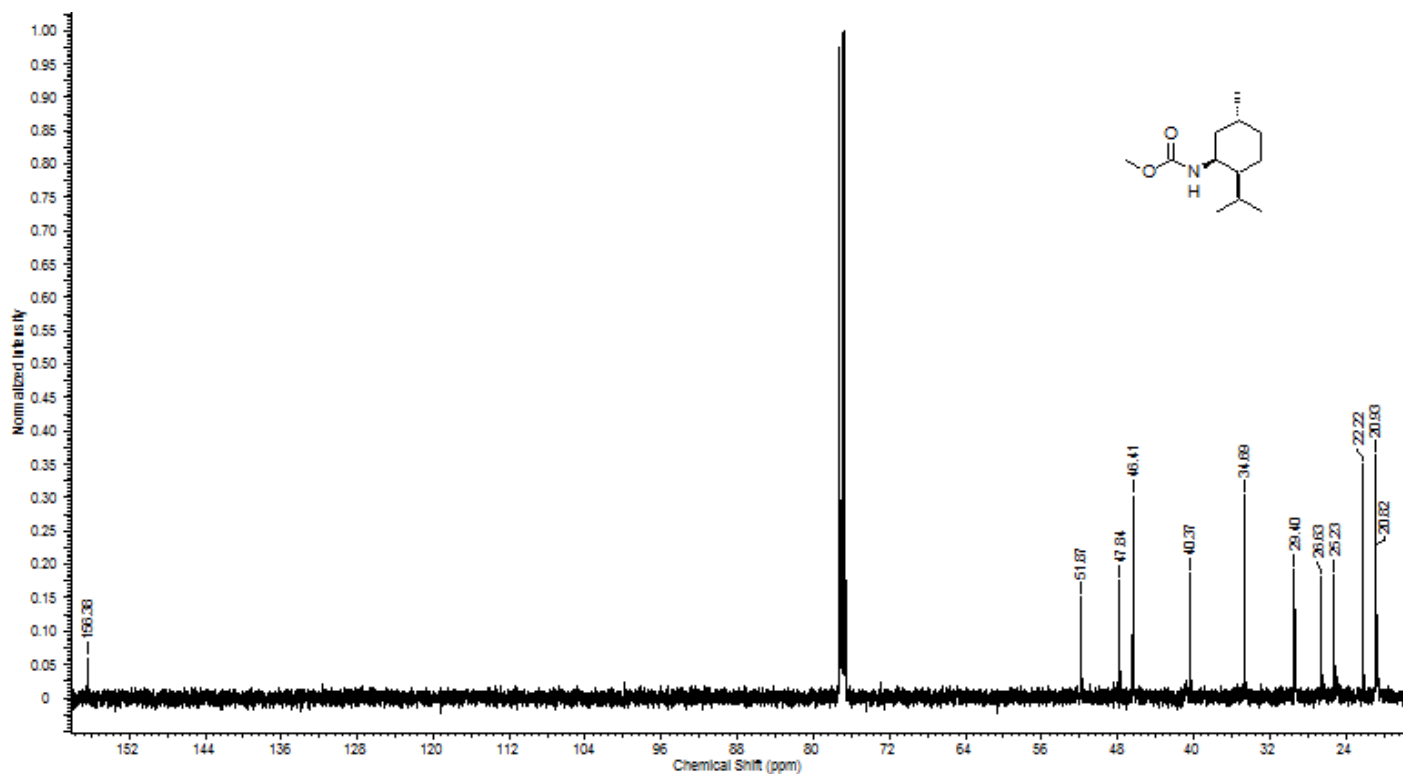
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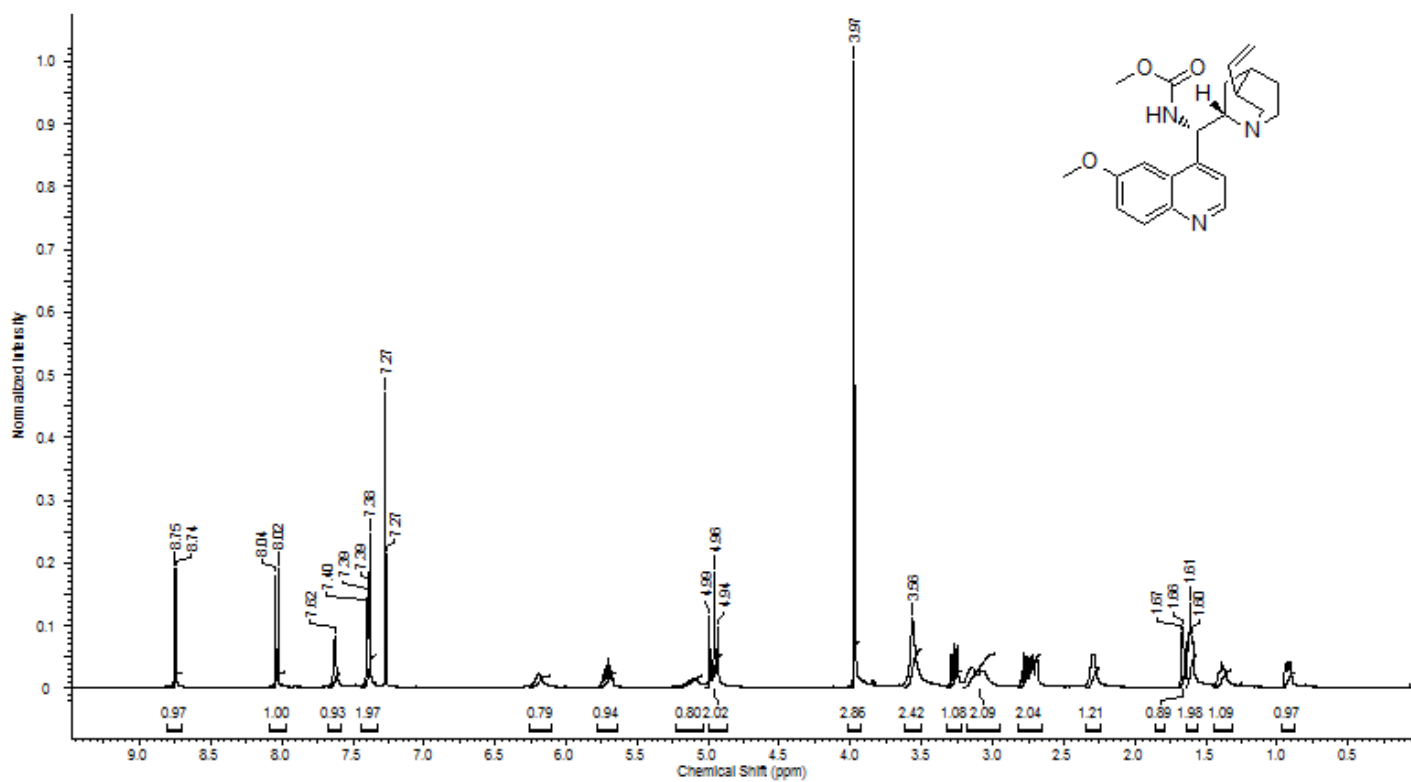
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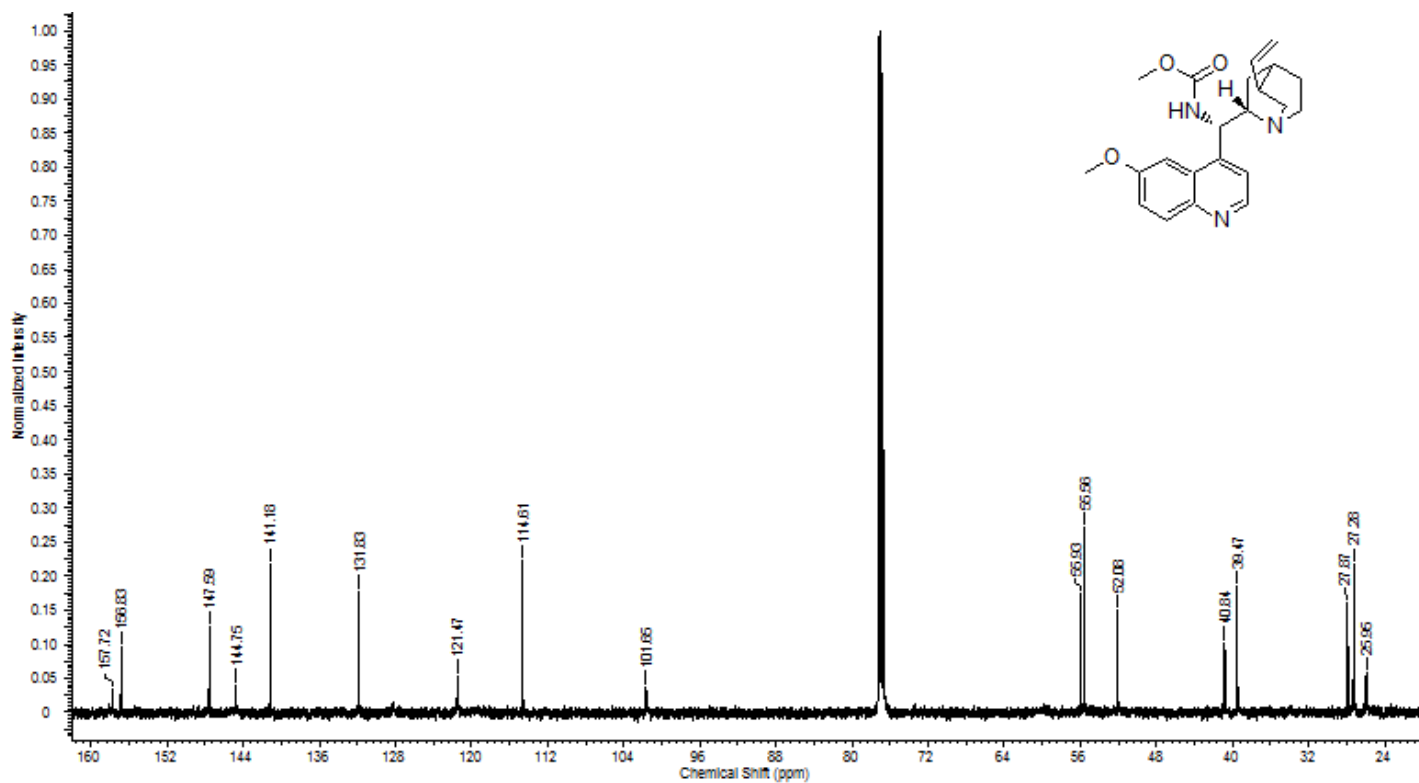
^1H NMR spectra of *N*-neomenthyl methyl carbamate (**23a**) (500 MHz, CDCl_3)



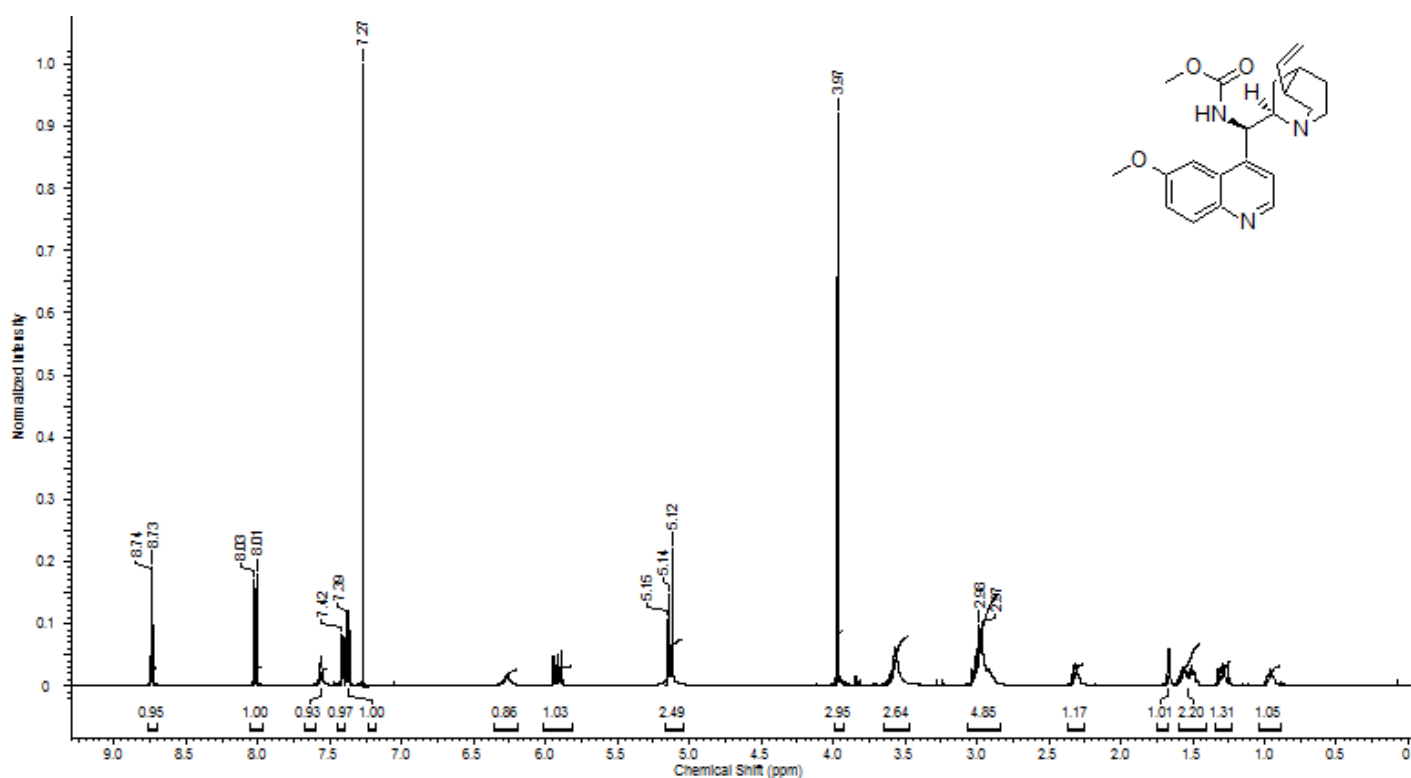
¹³C NMR spectra of *N*-neomenthyl methyl carbamate (**23a**) (500 MHz, CDCl₃)



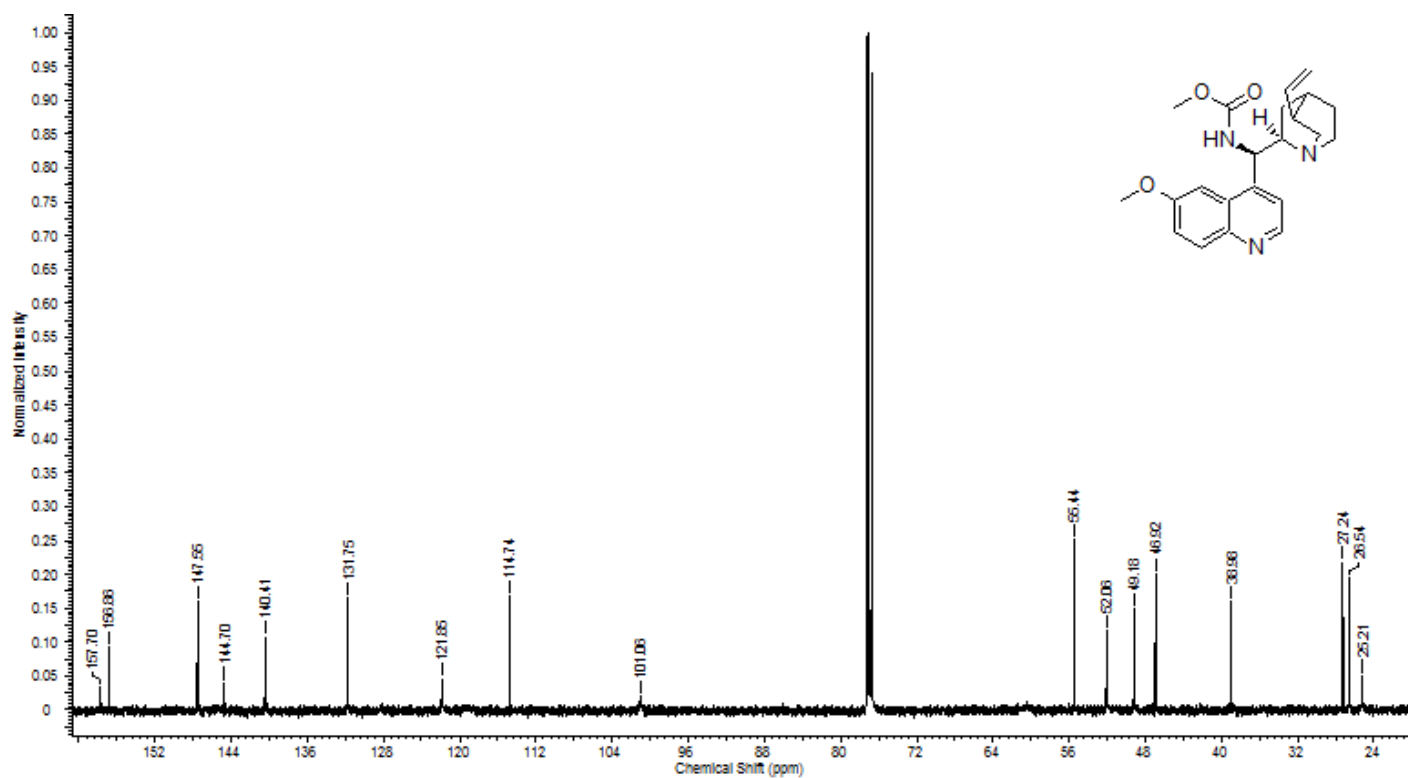
¹H NMR spectra of 9-((methoxycarbonyl)amino)-9-*epi*-quinine (**23b**) (500 MHz, CDCl₃)



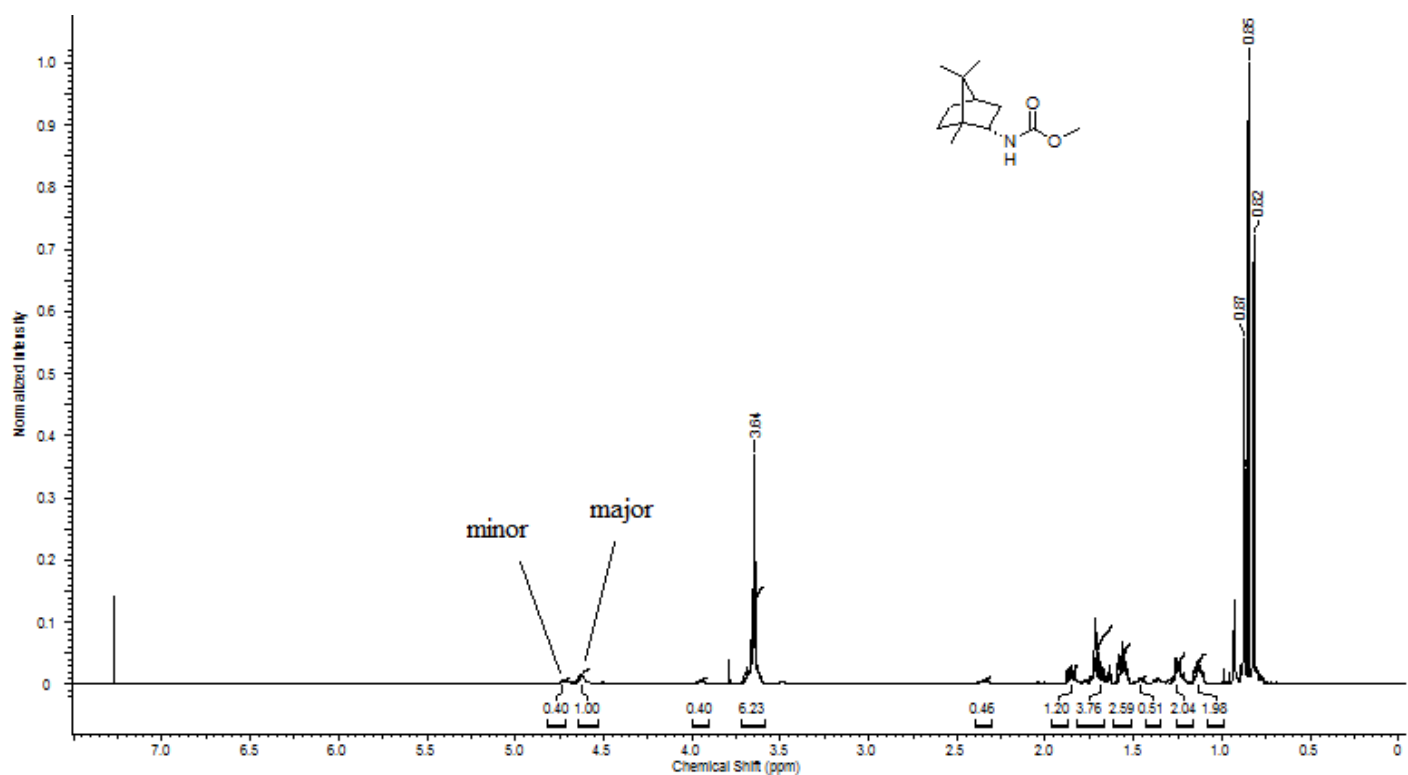
^{13}C NMR spectra of 9-((methoxycarbonyl)amino)-9-*epi*-quinine (**23b**) (126 MHz, CDCl_3)



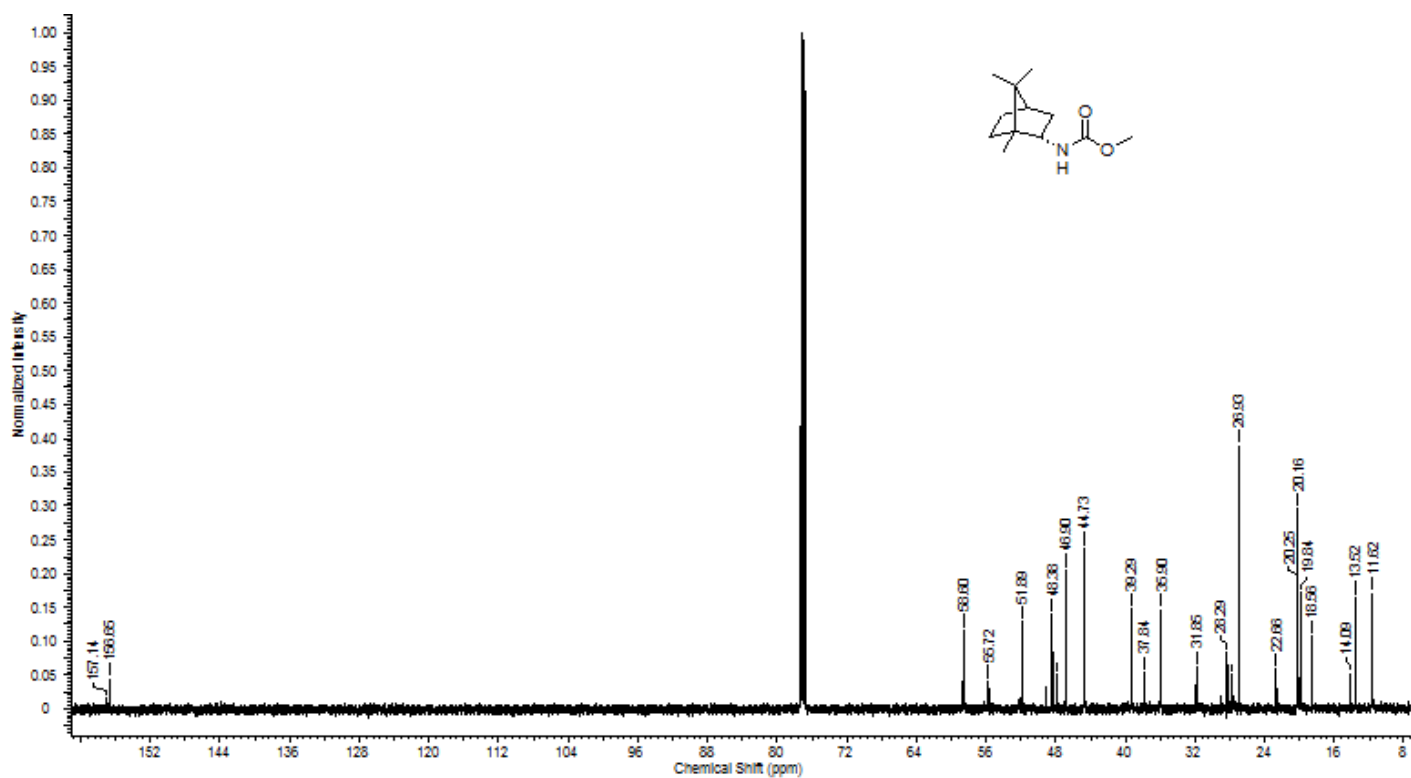
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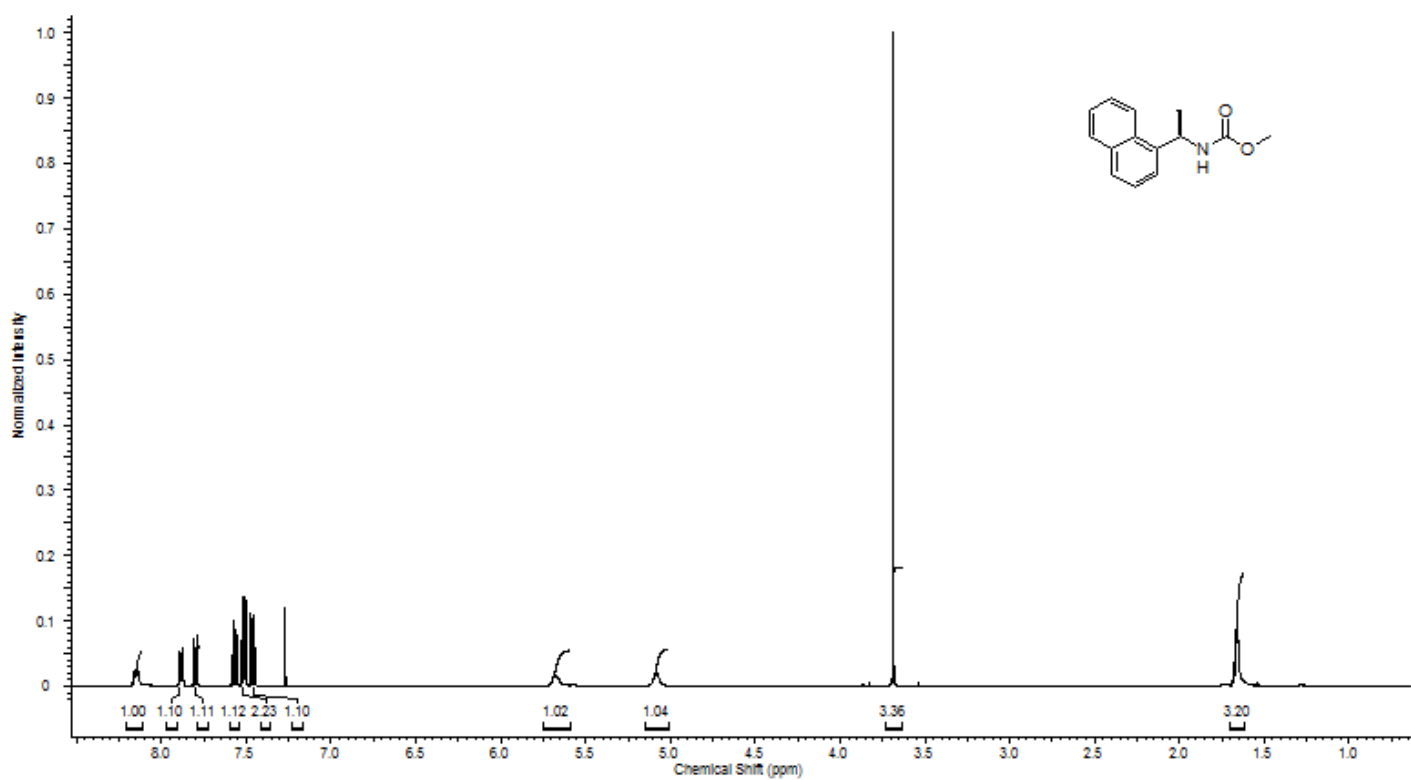
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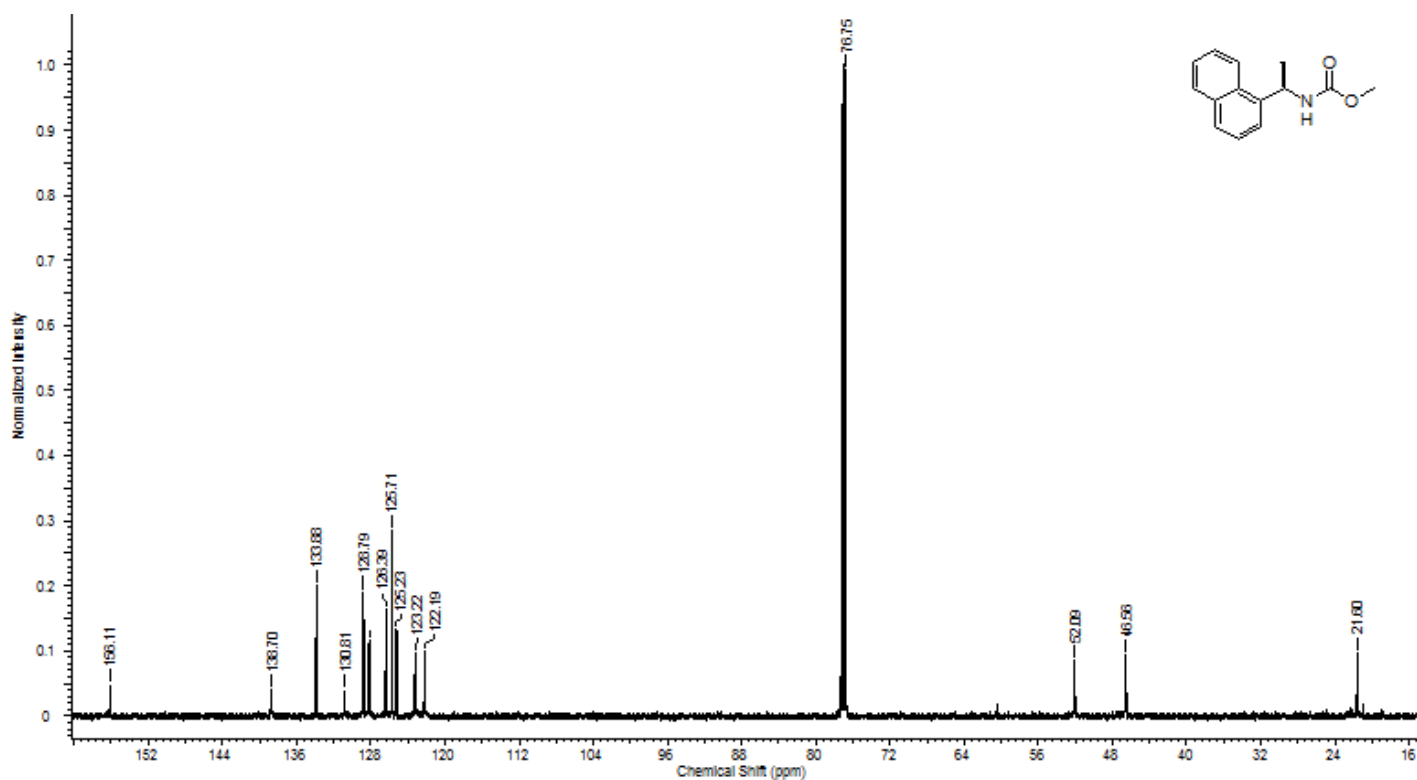
¹H NMR spectra of N-bornyl methyl carbamate (**23d**) (500 MHz, CDCl₃)



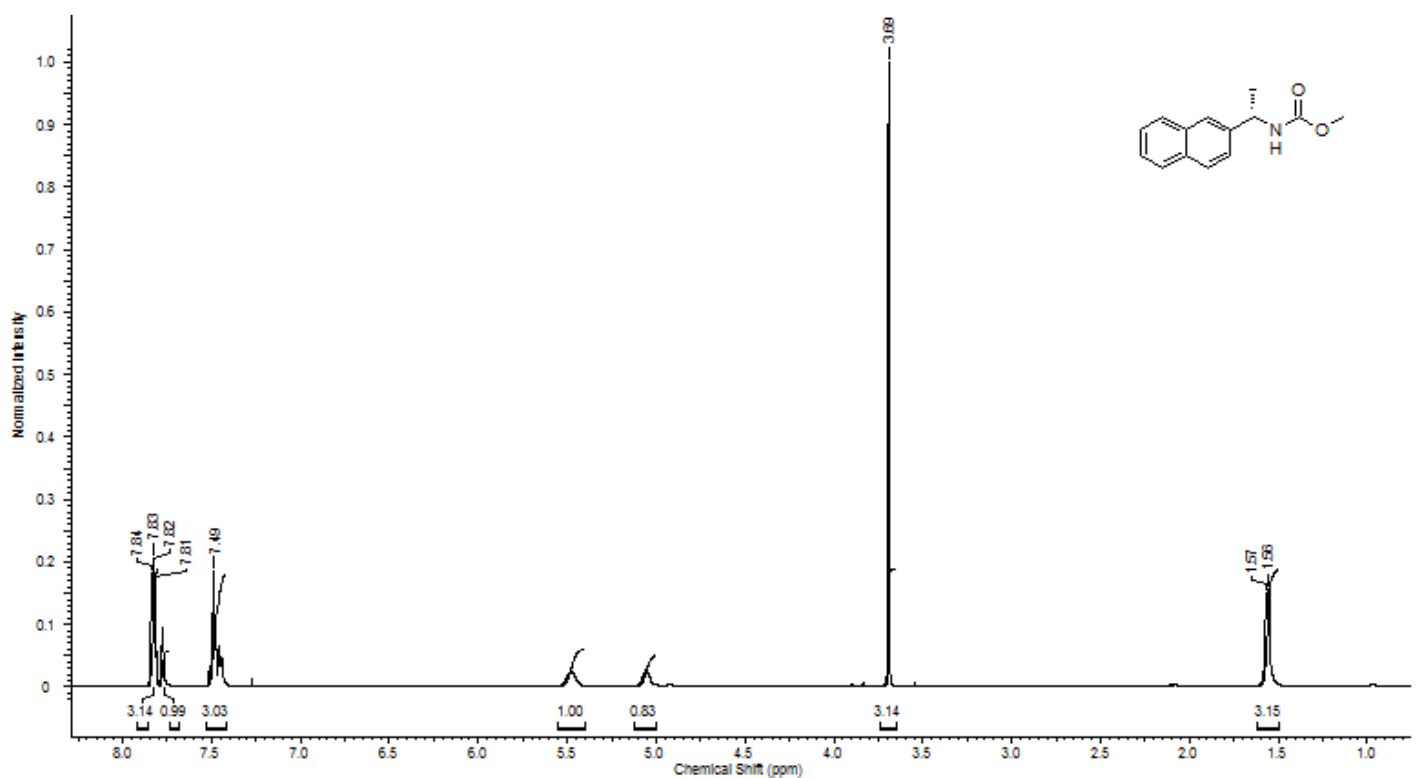
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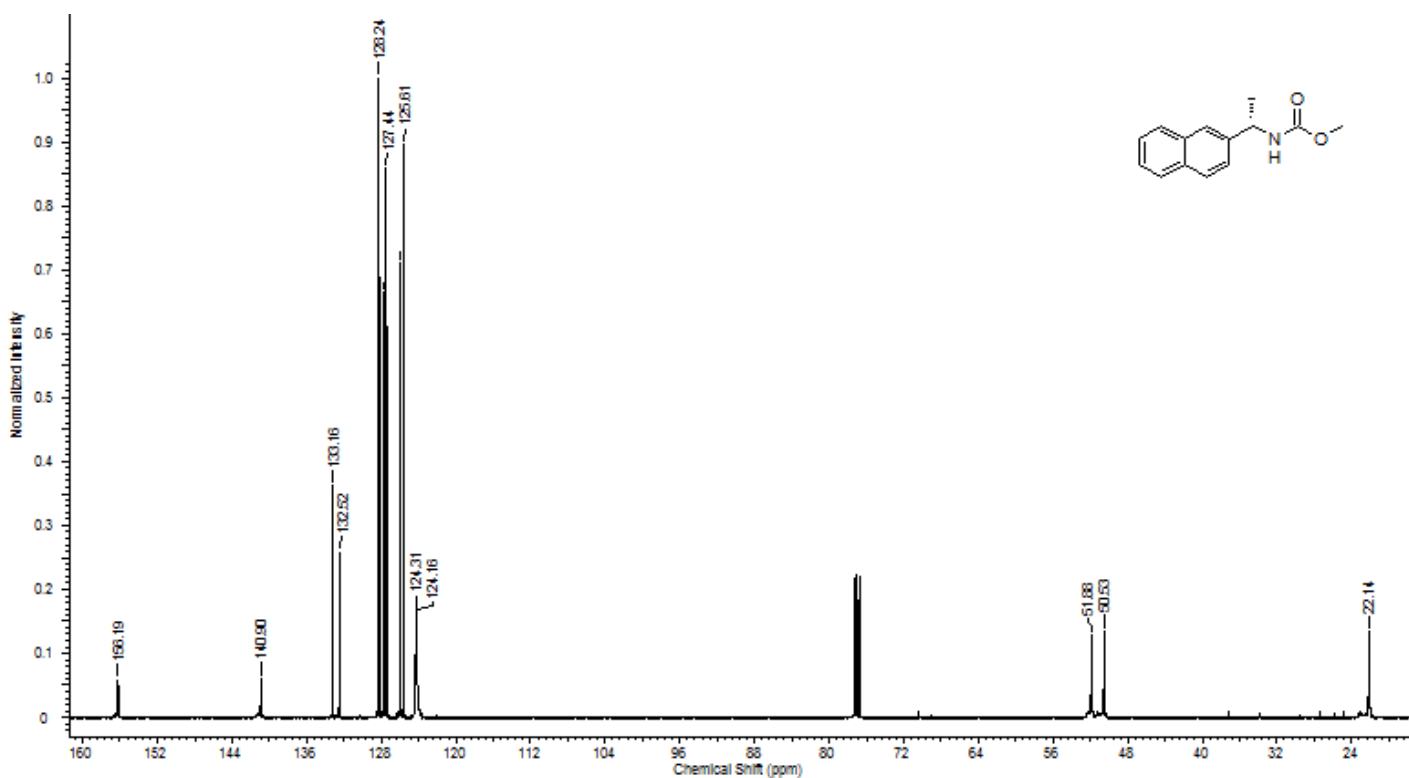
¹H NMR spectra of *N*-(*R*)-1-(1-naphthyl)ethyl methyl carbamate (**23e**) (500 MHz, CDCl₃)



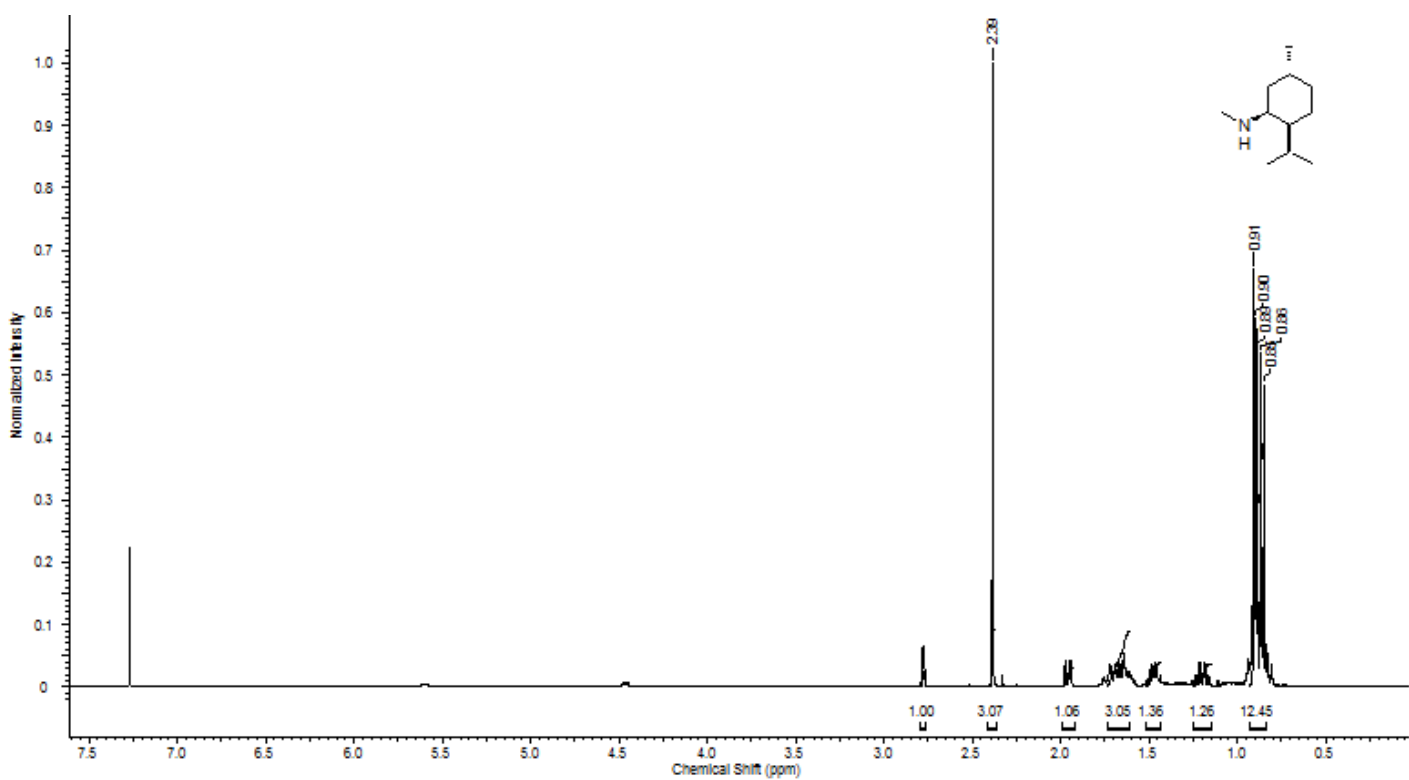
^{13}C NMR spectra of *N*-(*R*)-1-(1-naphthyl)ethyl methyl carbamate (**23e**) (126 MHz, CDCl_3)



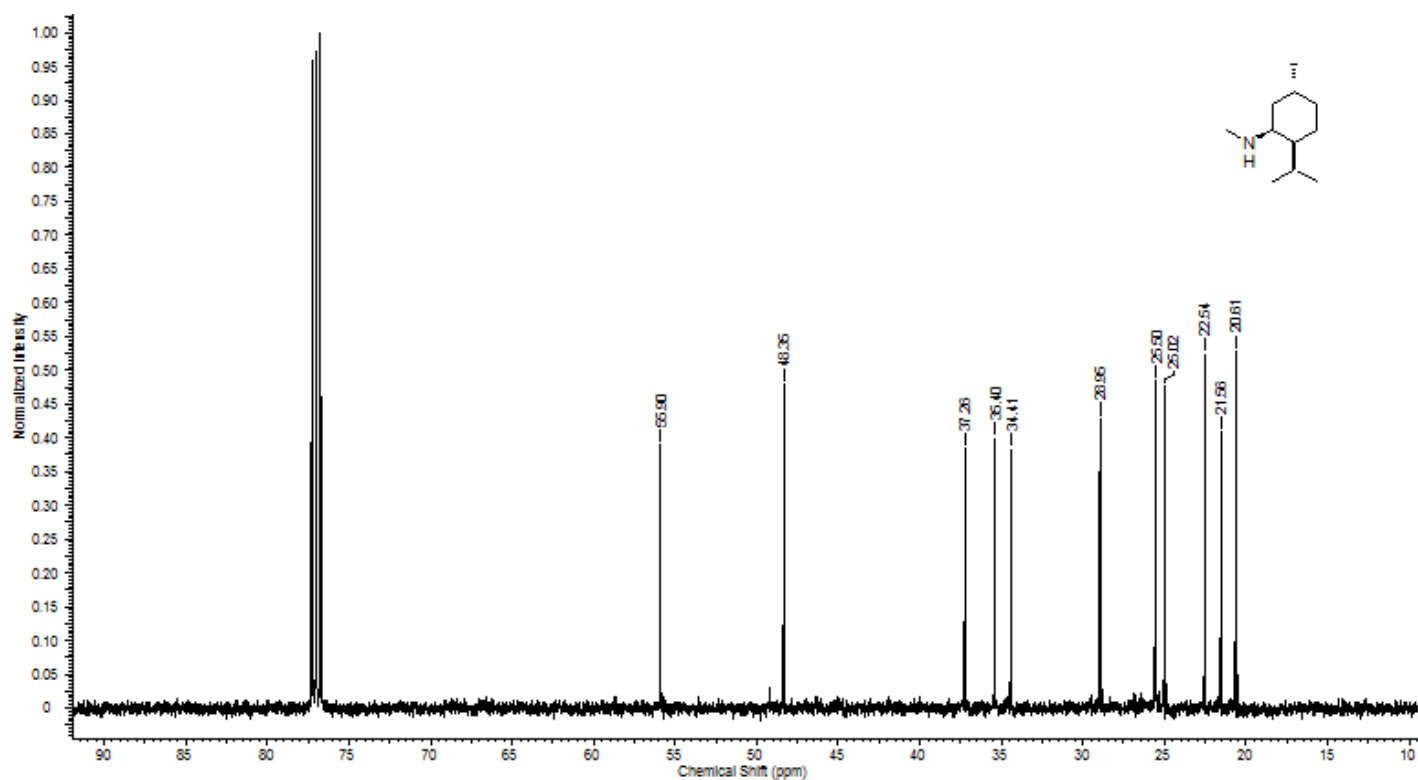
^1H NMR spectra of *N*-(*S*)-1-(2-naphthyl)ethyl methyl carbamate (**23f**) (500 MHz, CDCl_3)



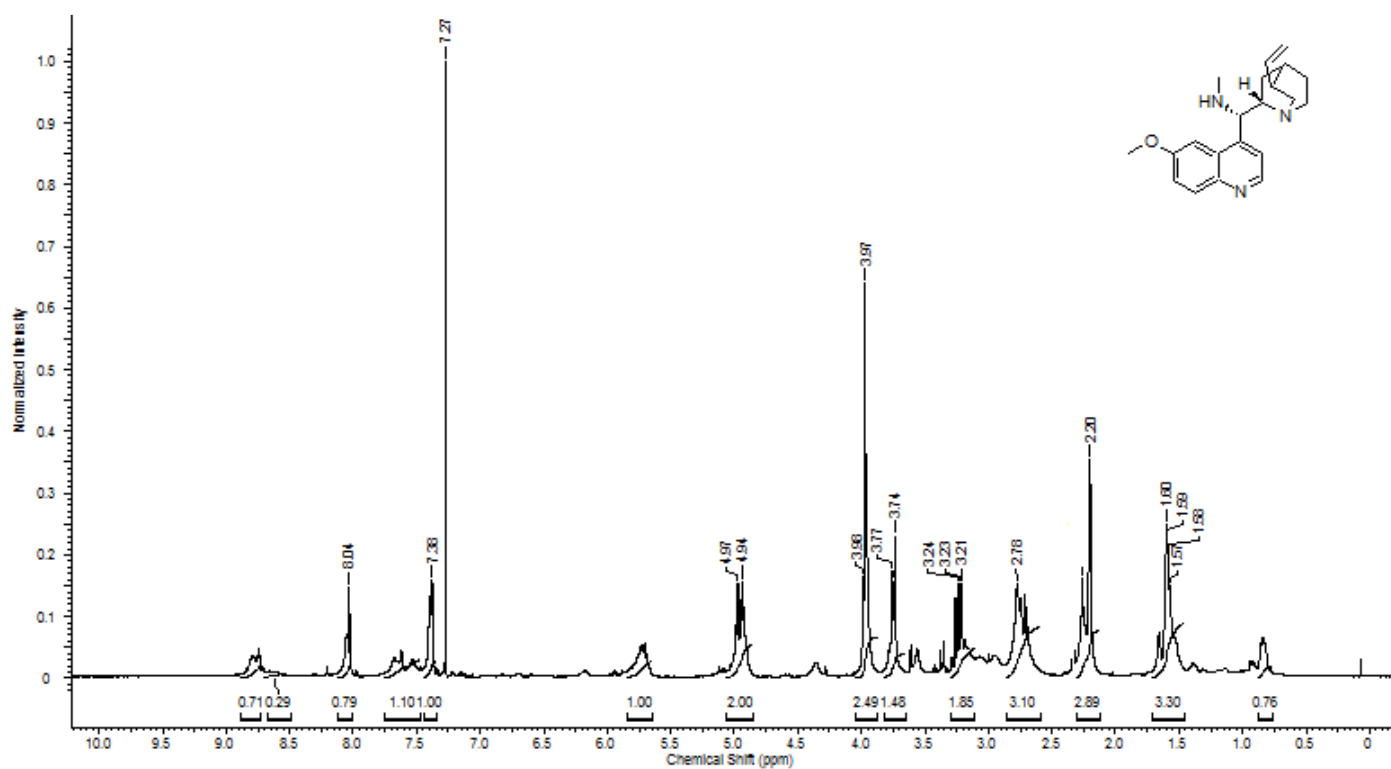
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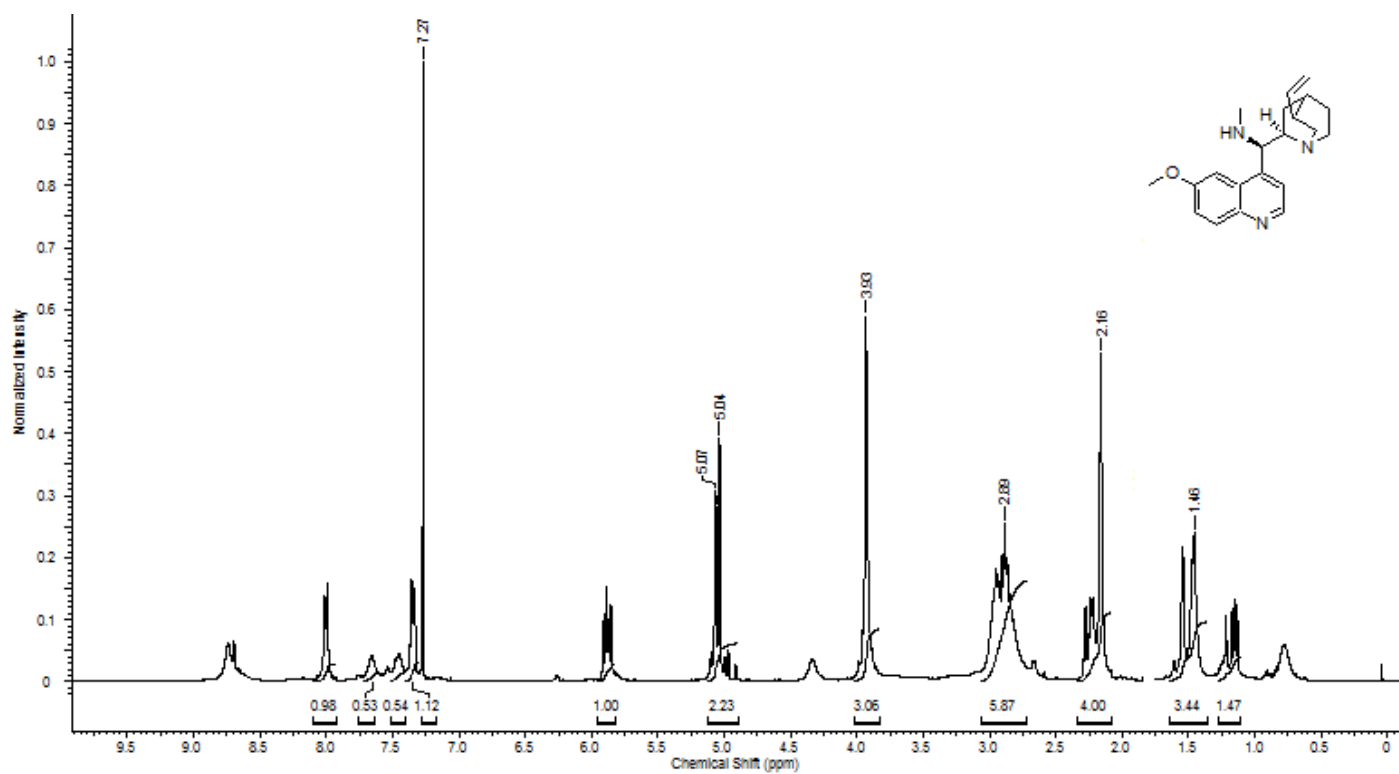
^1H NMR spectra of *N*-methylneomenthylamine (**24a**) (500 MHz, CDCl_3)



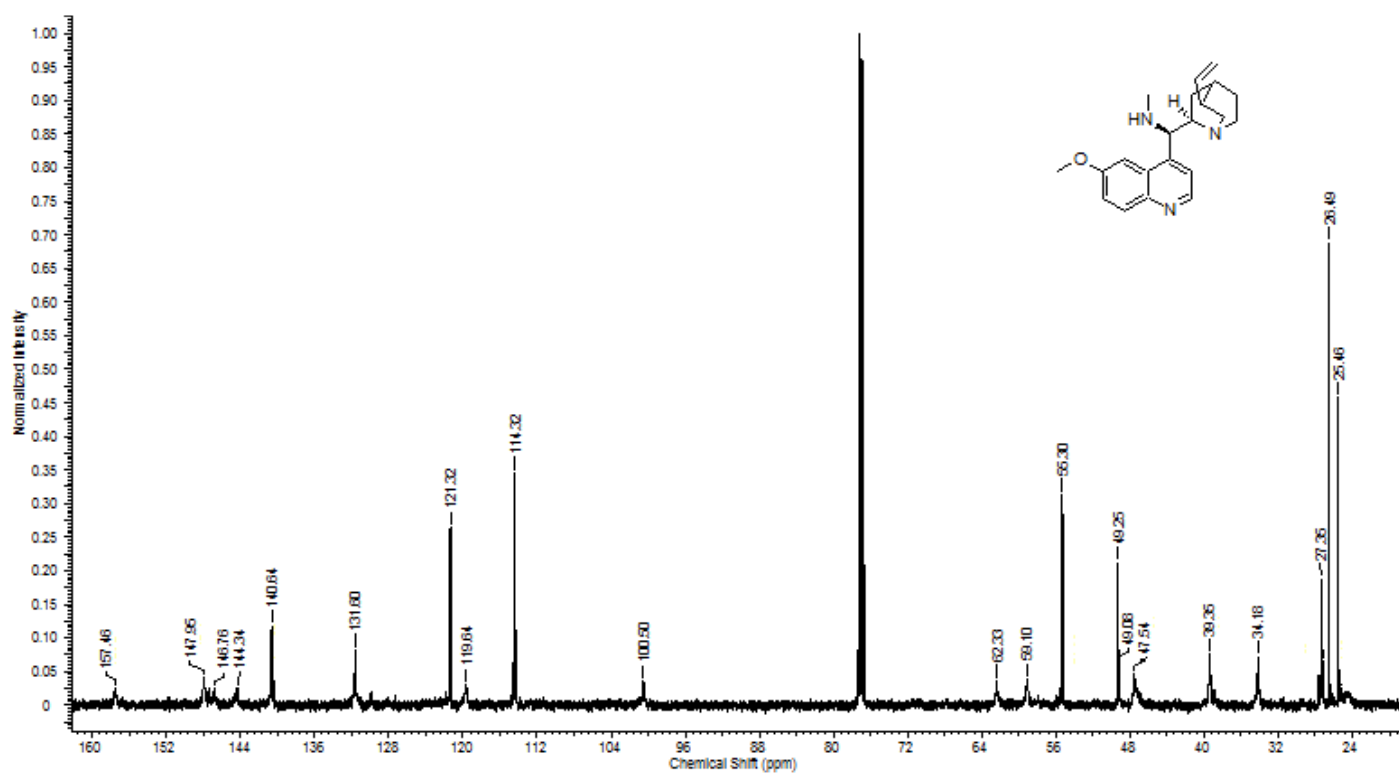
¹³C NMR spectra of *N*-methylnemomenthylamine (**24a**) (126 MHz, CDCl₃)



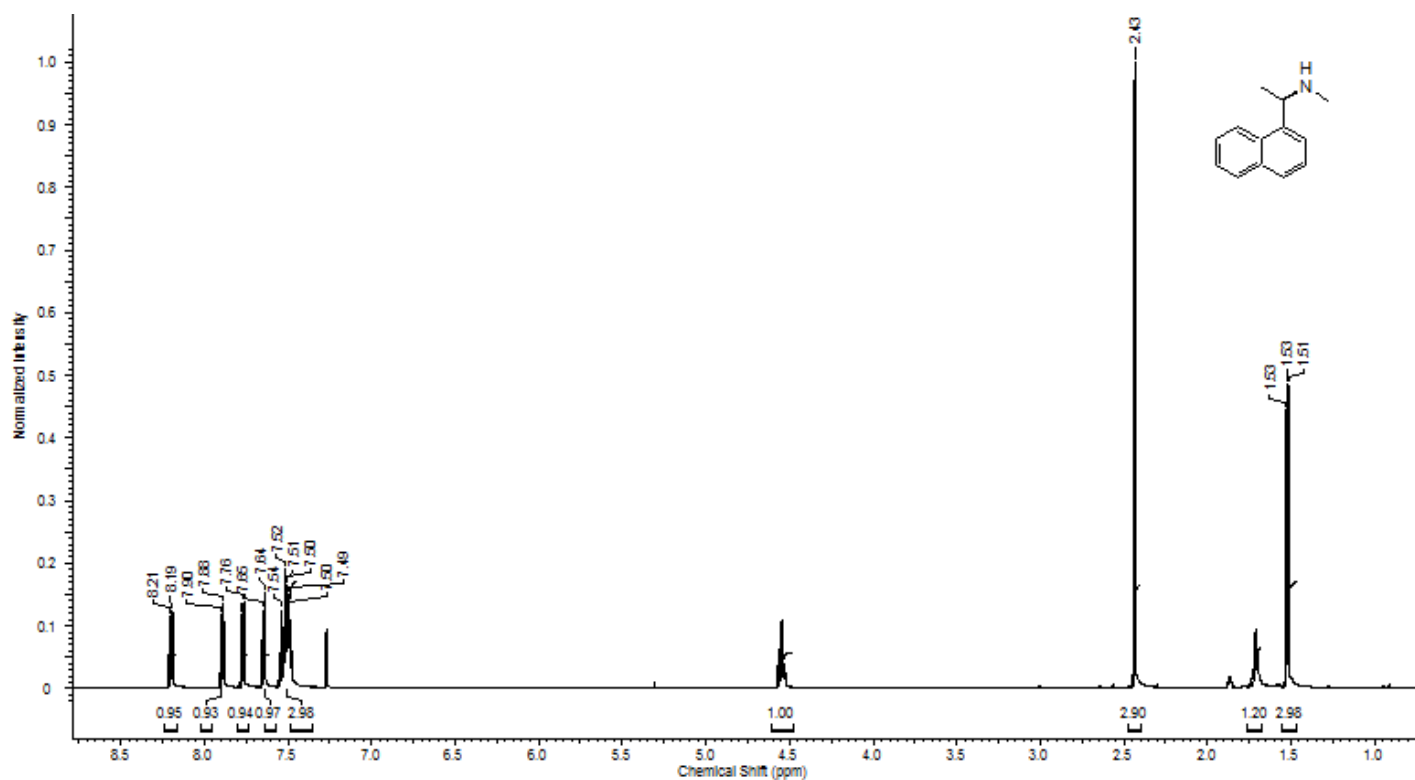
¹H NMR spectra of 9-(methylamino)-9-*epi*-quinine (**24b**) (500 MHz, CDCl₃)



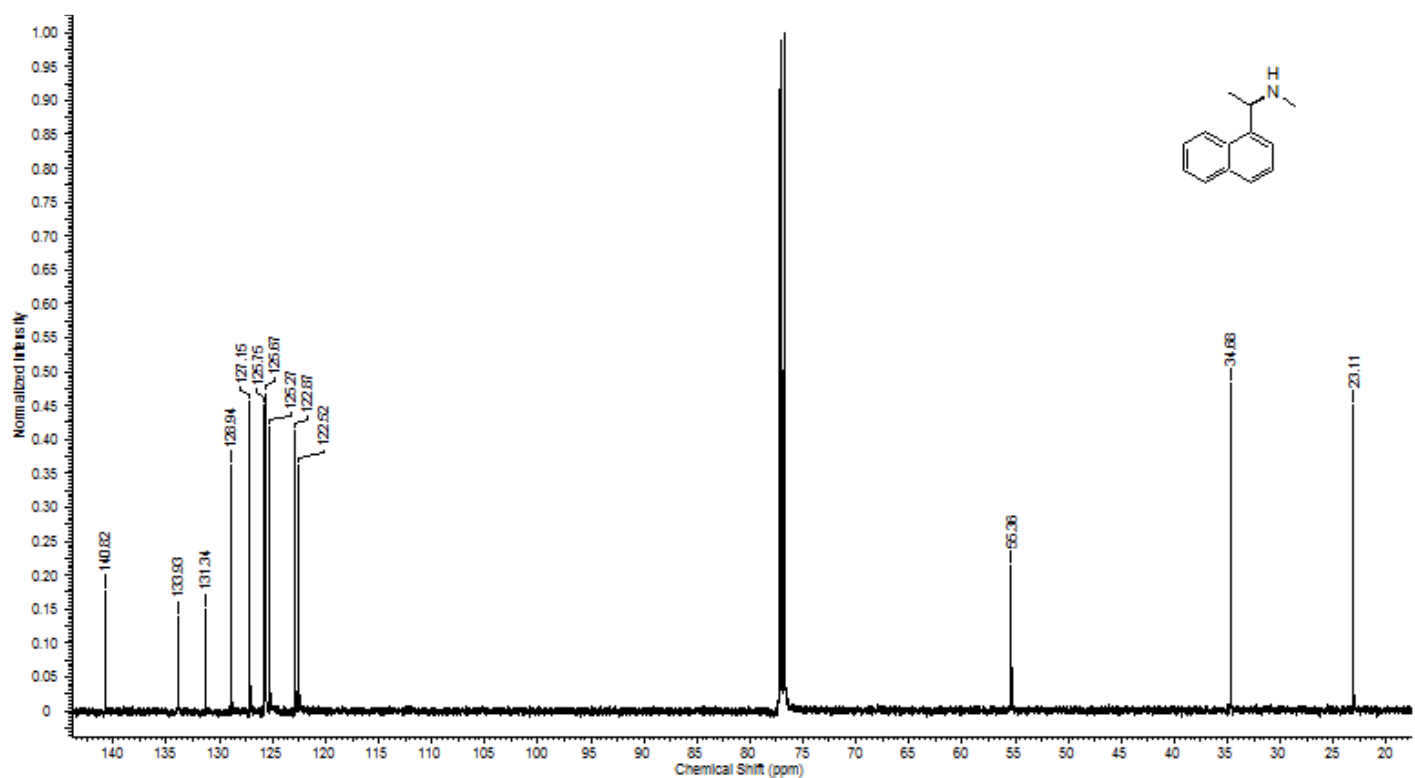
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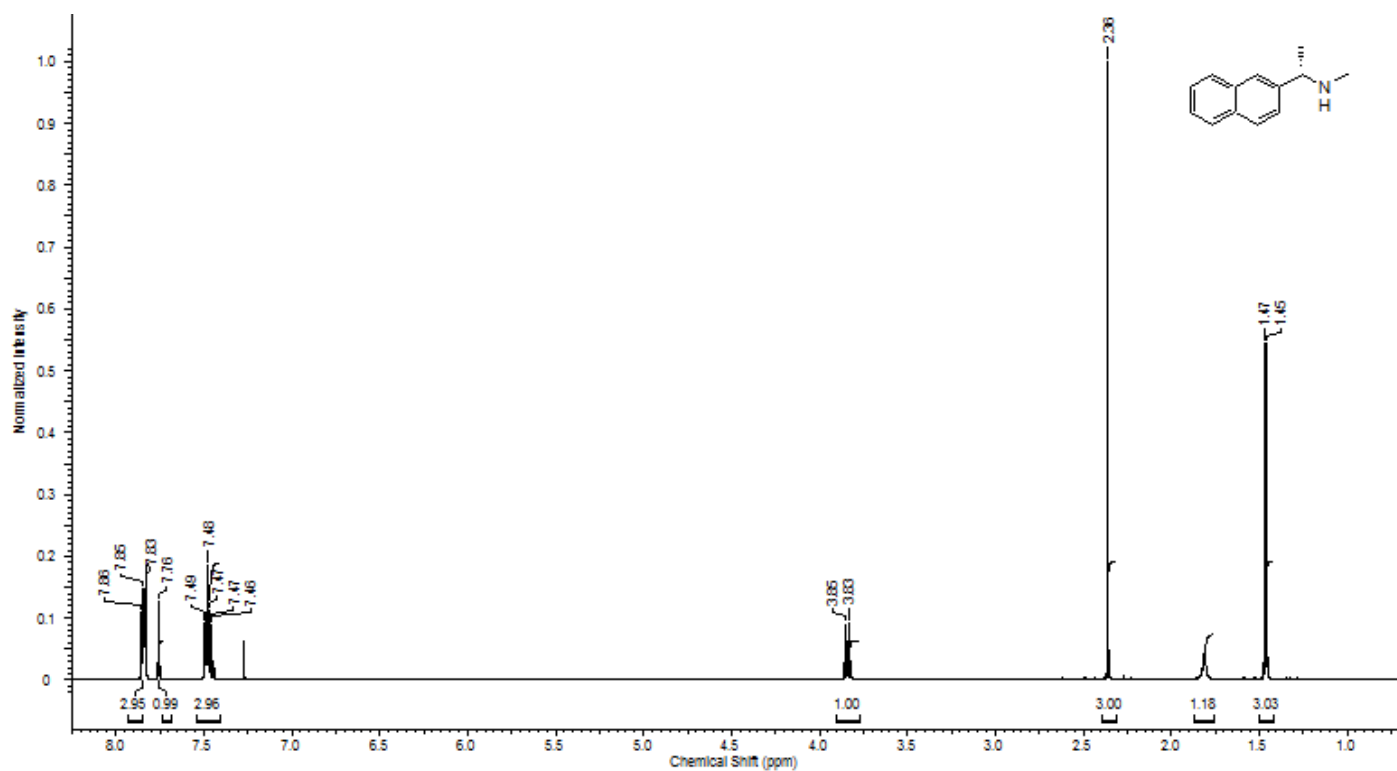
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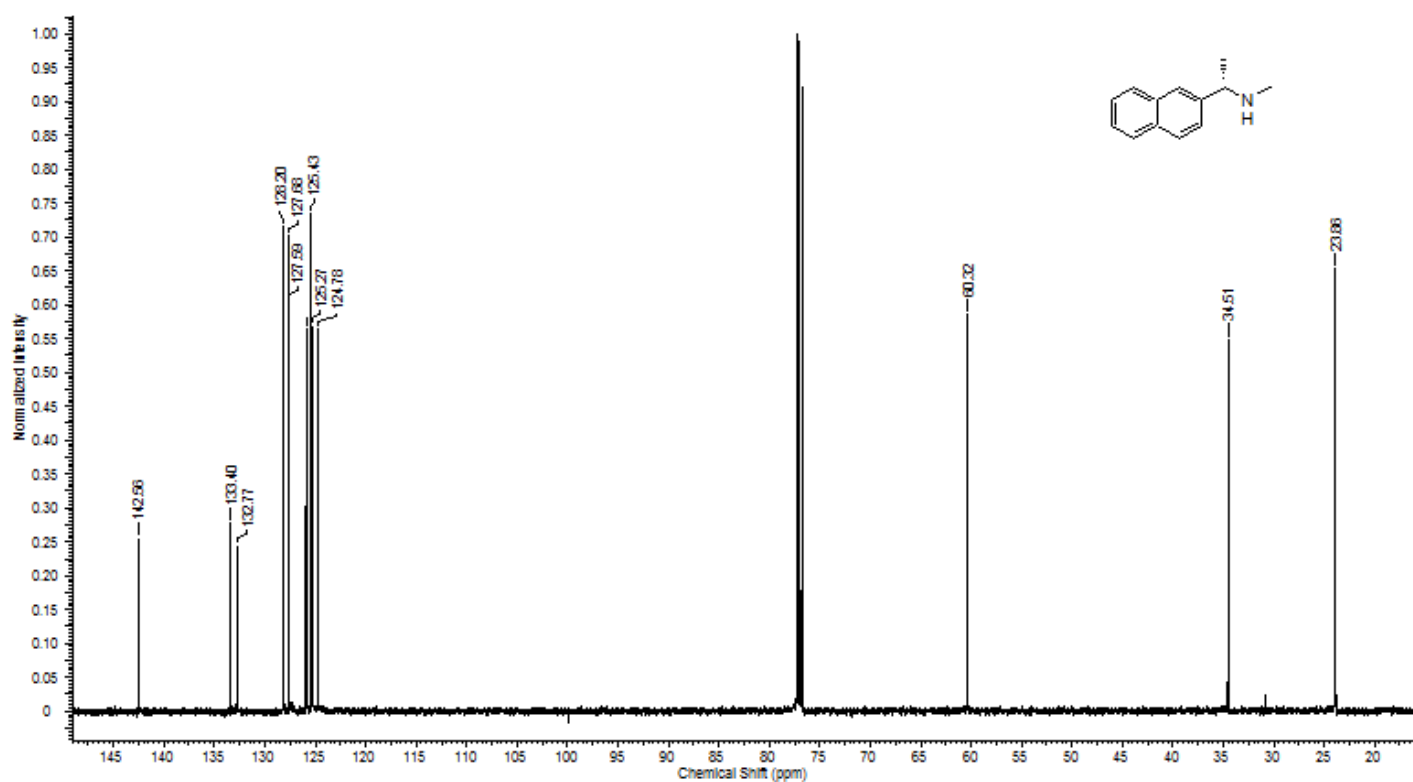
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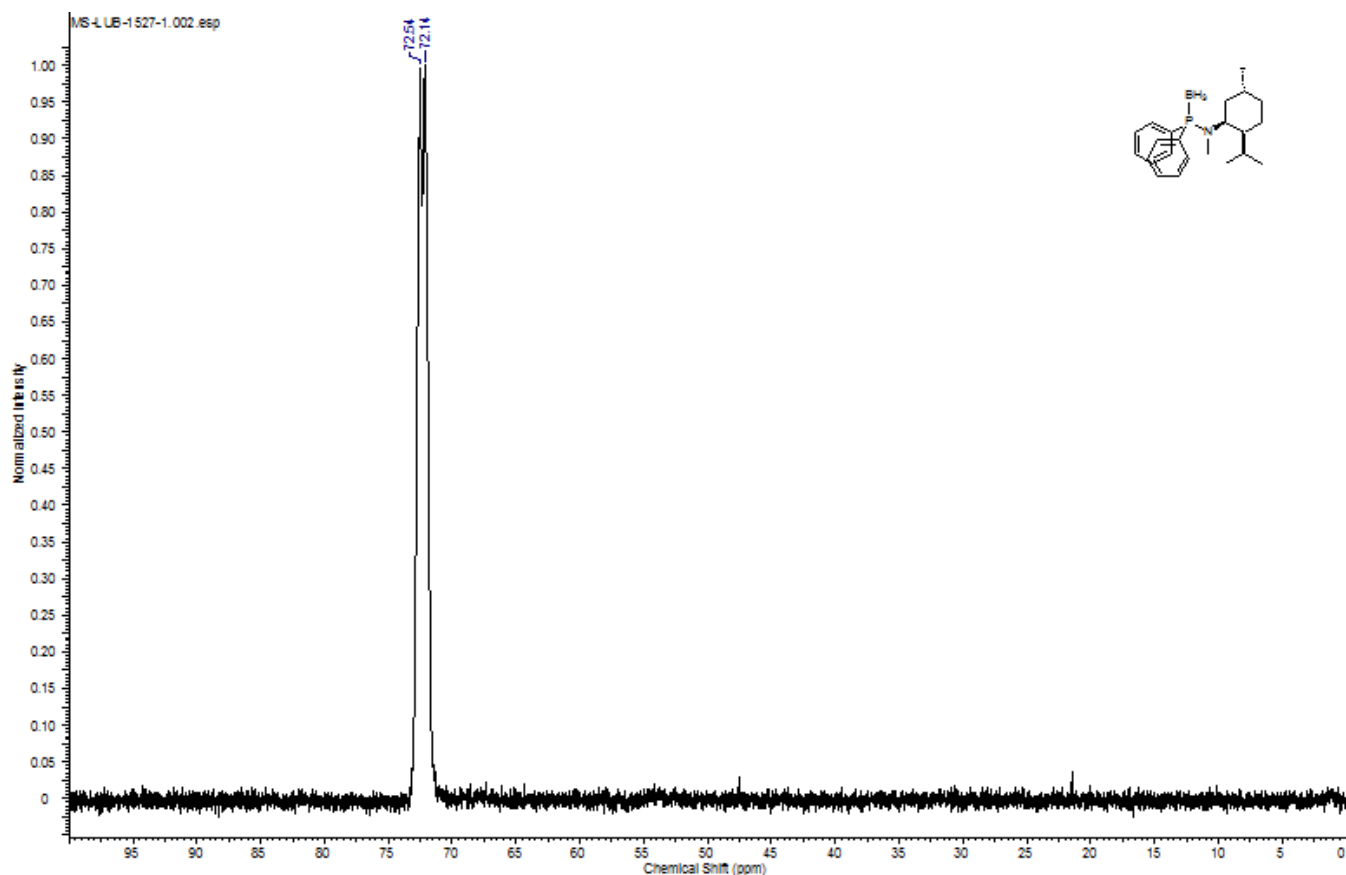
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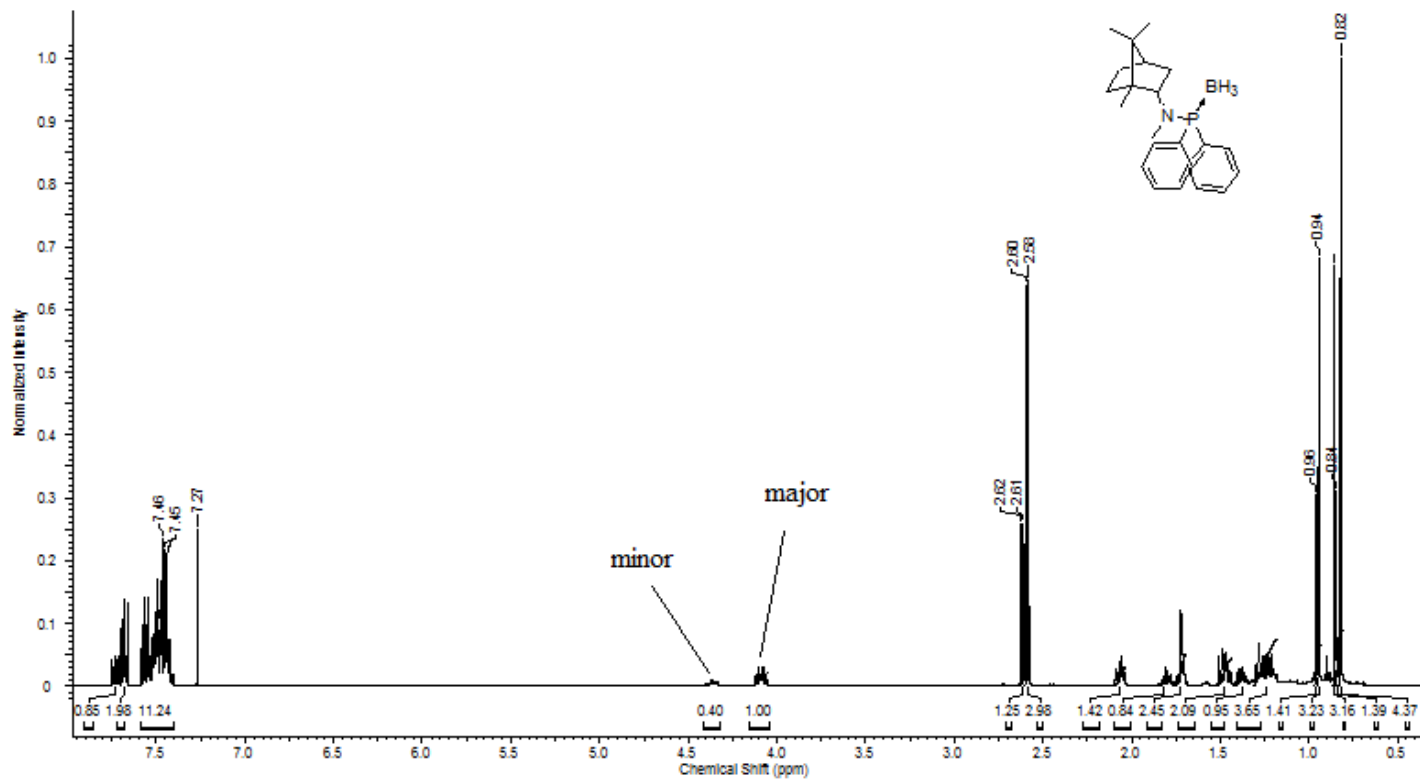
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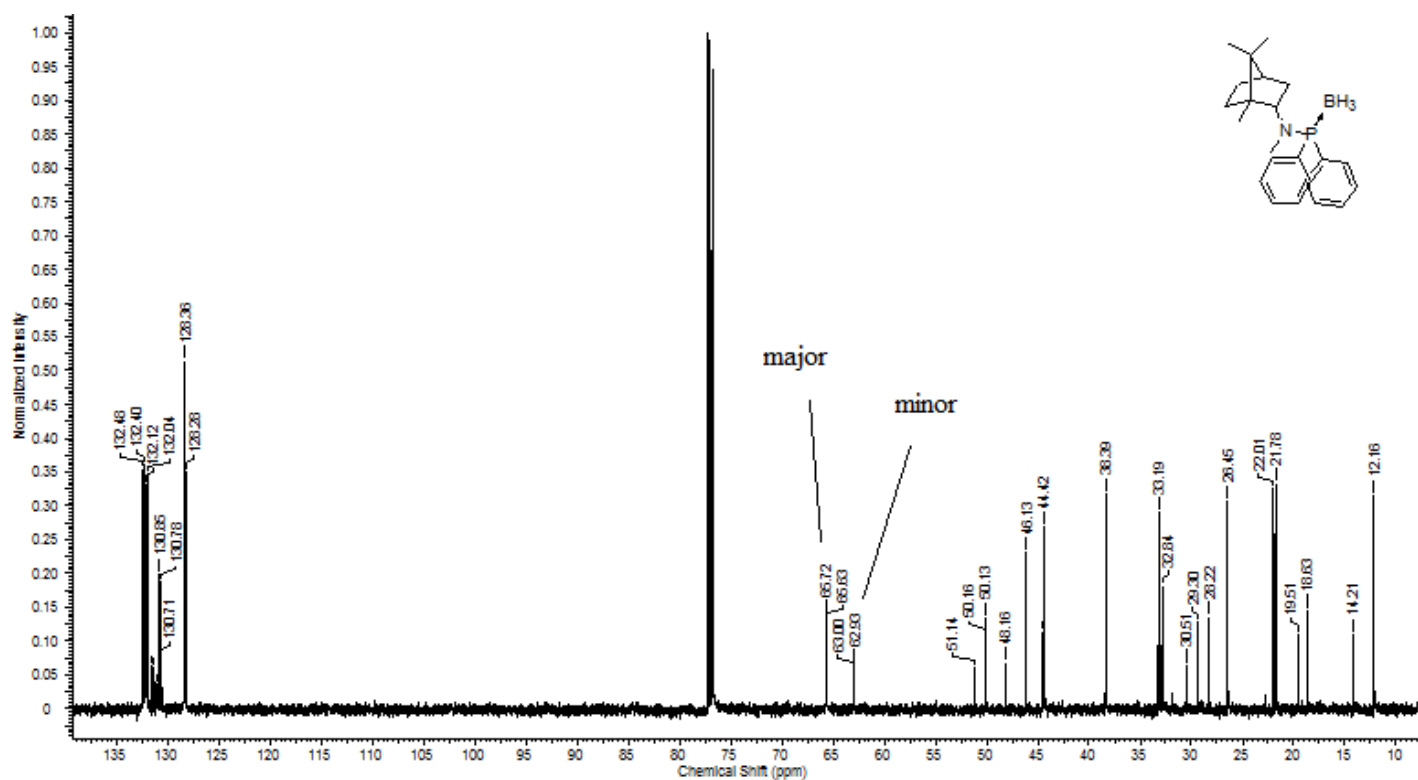
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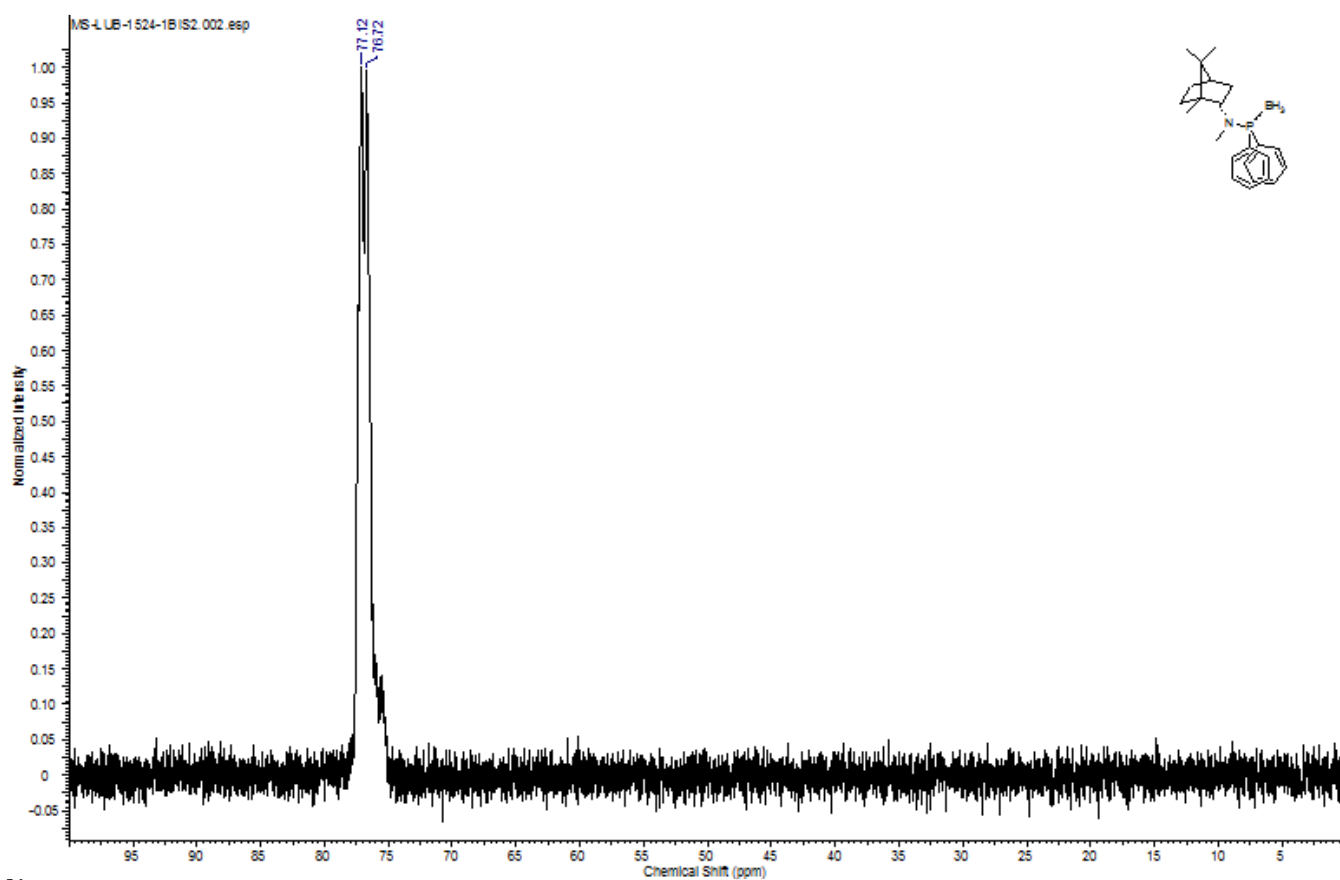
^{31}P NMR spectra of diphenylphosphinous acid-borane *N*-methylneomenthylamide (**25a**) (202 MHz, CDCl_3)



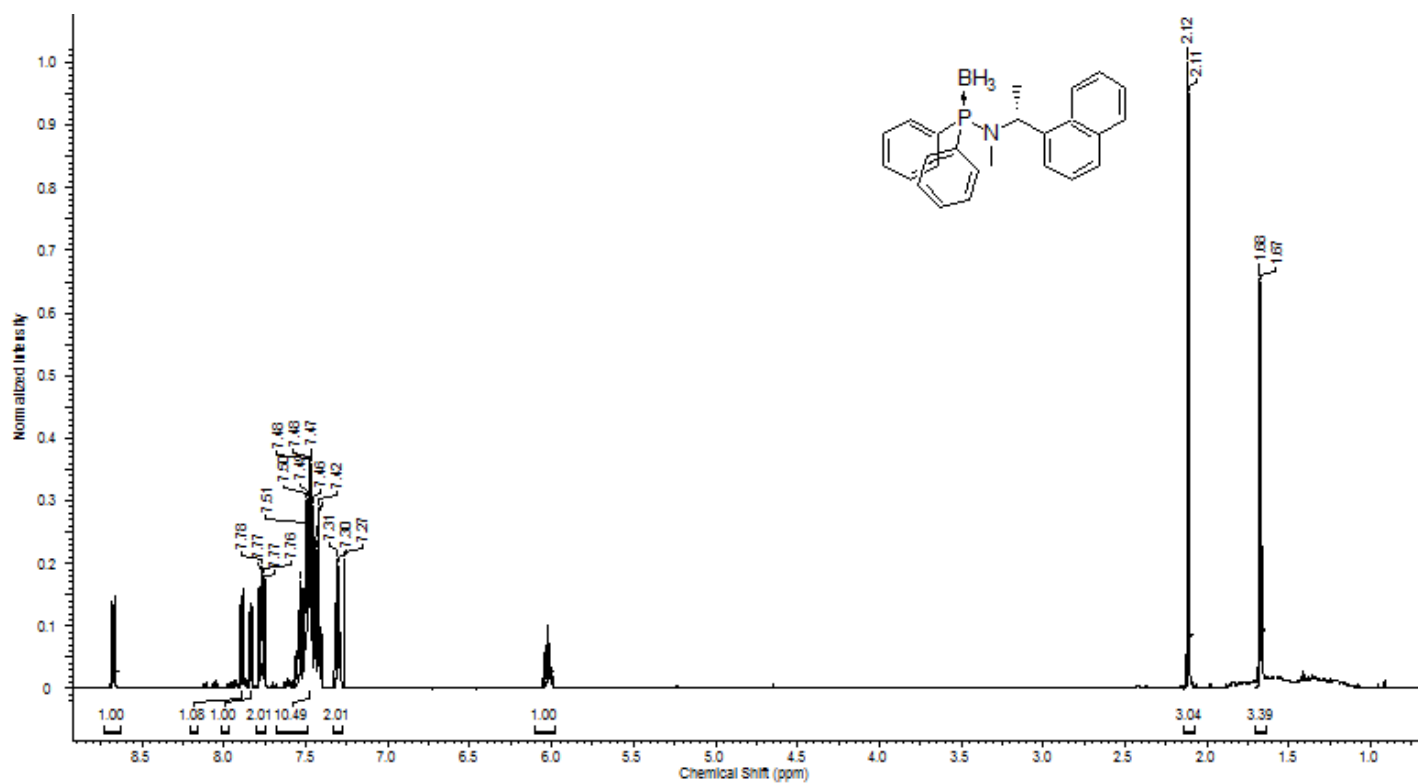
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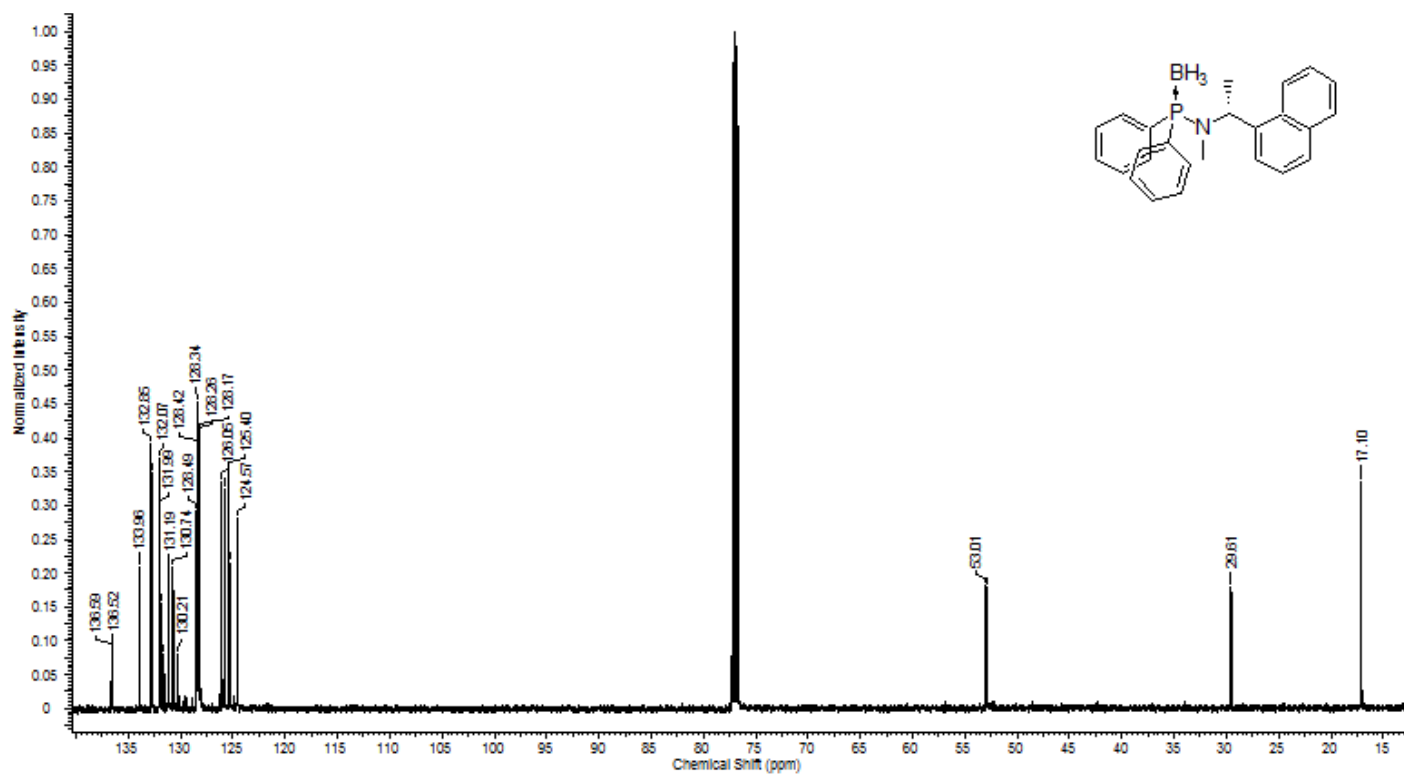
^{13}C NMR spectra of diphenylphosphinous acid-borane *N*-methyl-bornylamide (**25d**) (126 MHz, CDCl_3)



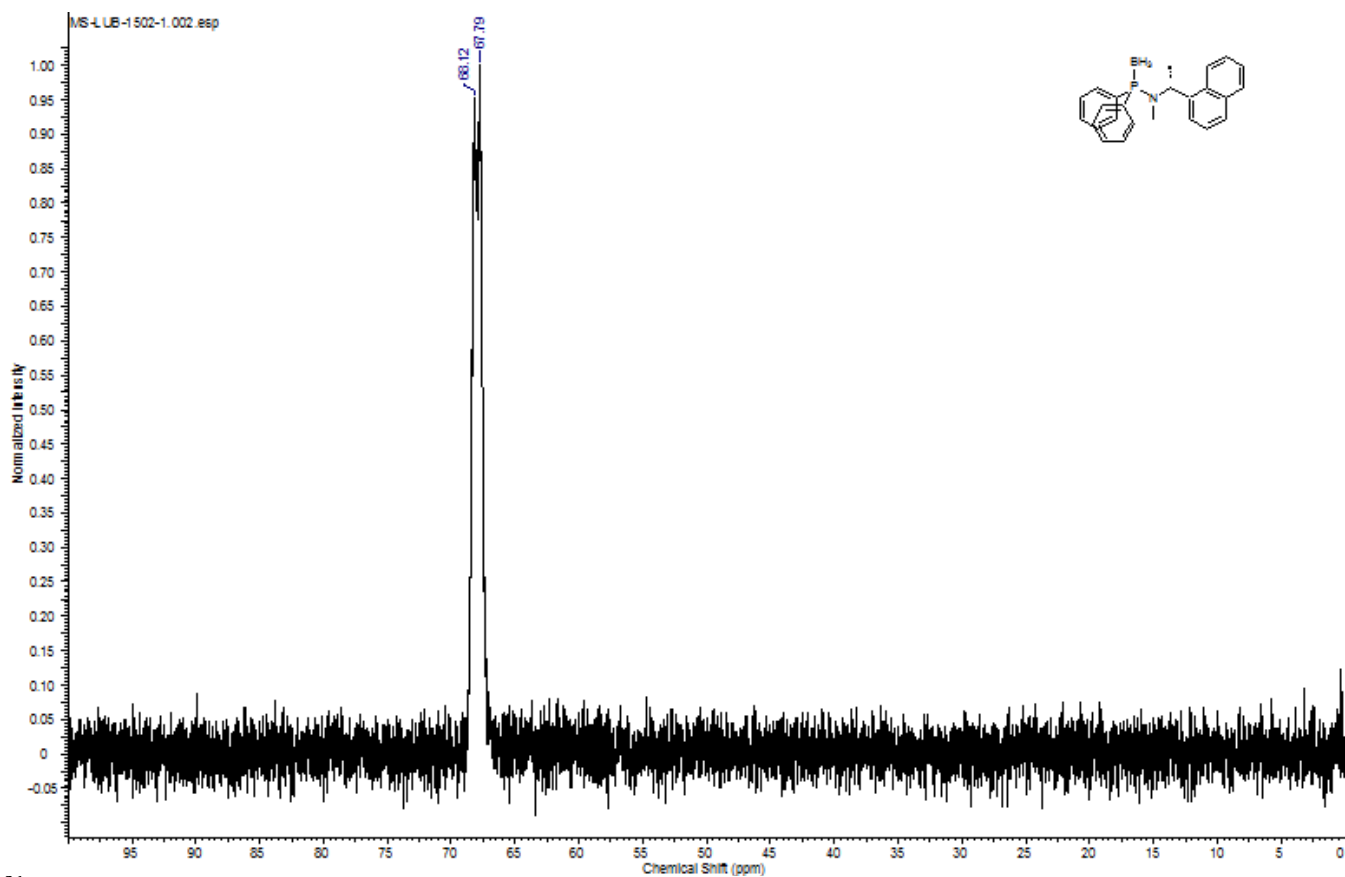
^{31}P NMR spectra of diphenylphosphinous acid-borane *N*-methyl-bornylamide (**25d**) (202 MHz, CDCl_3)



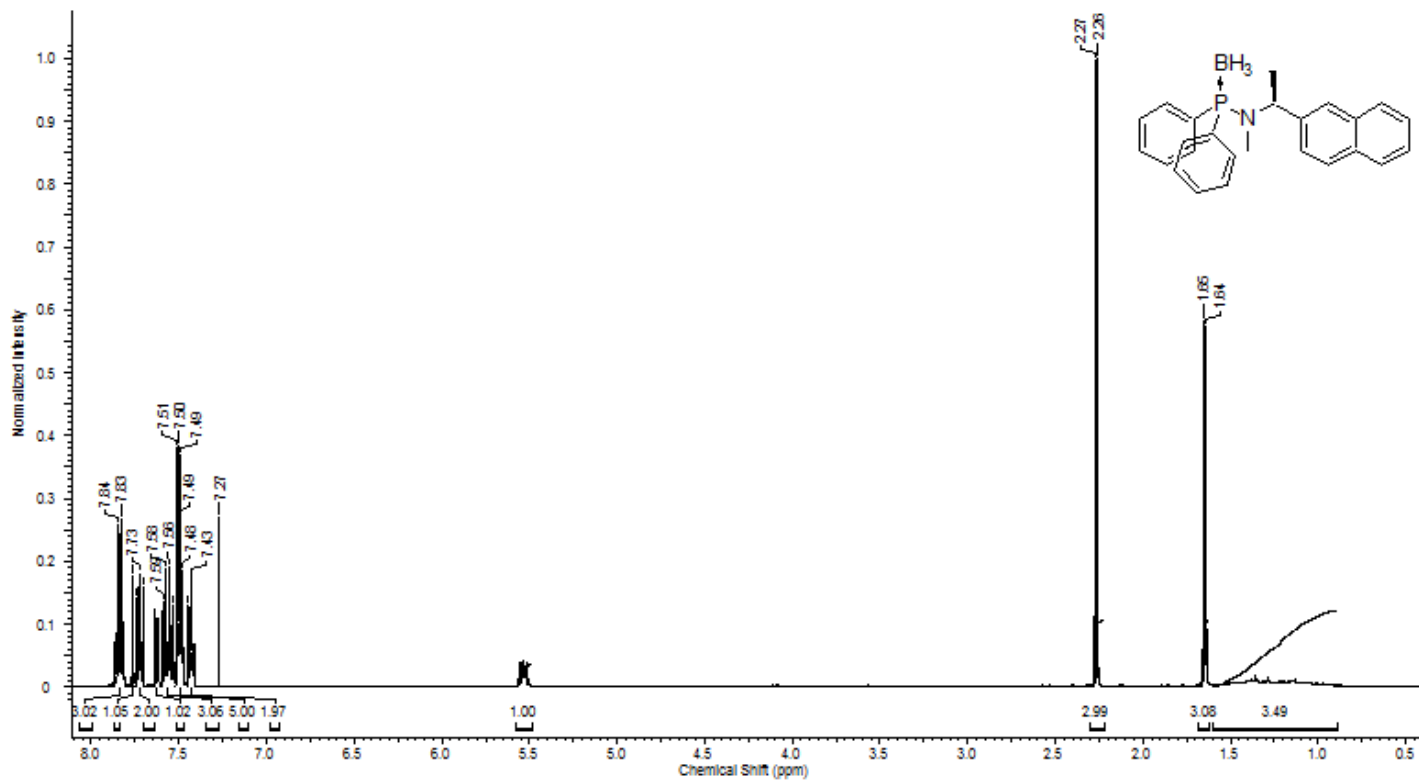
¹H NMR spectra of diphenylphosphinous acid-borane *N*-methyl-(*R*)-1-(1-naphthyl)ethylamide (**25e**) (500 MHz, CDCl₃)



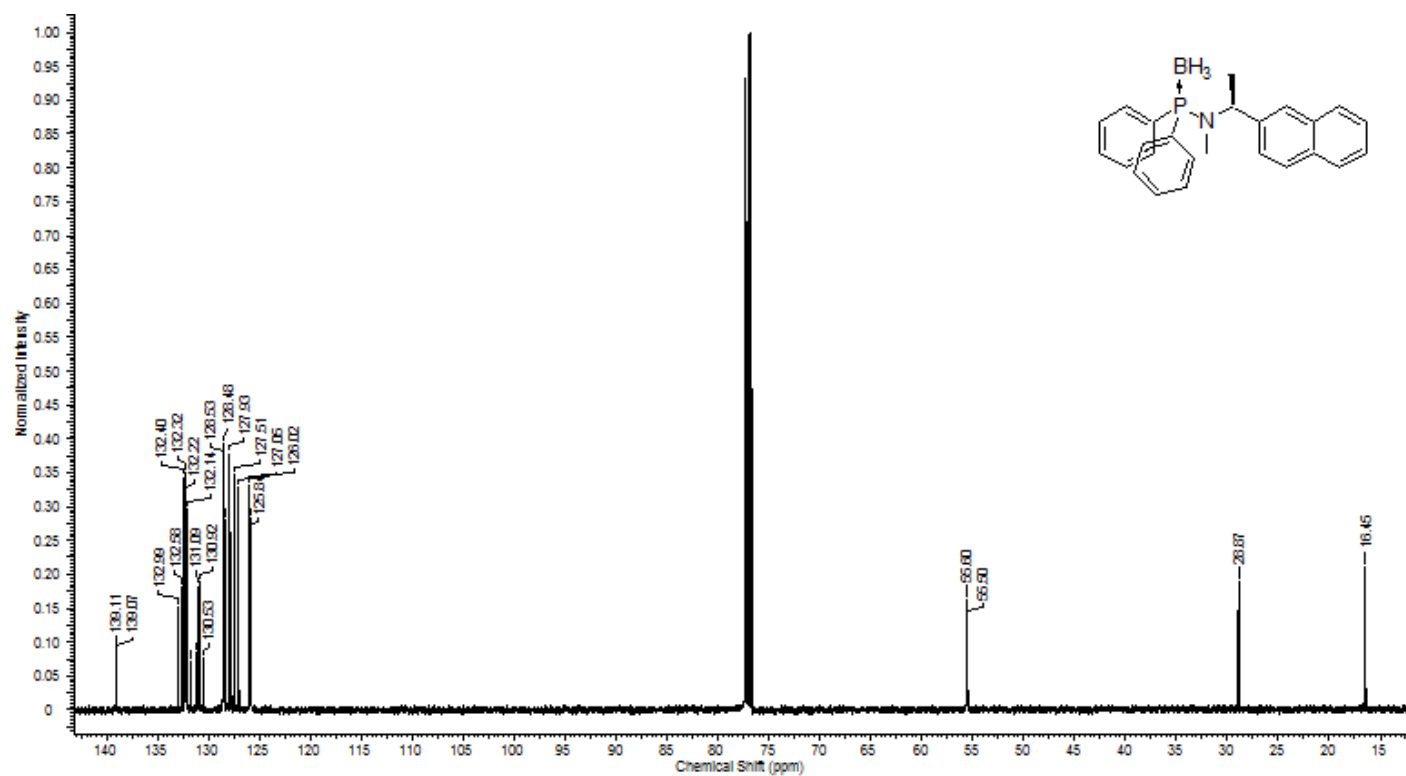
¹³C NMR spectra of diphenylphosphinous acid-borane *N*-methyl-(*R*)-1-(1-naphthyl)ethylamide (**25e**) (126 MHz, CDCl₃)



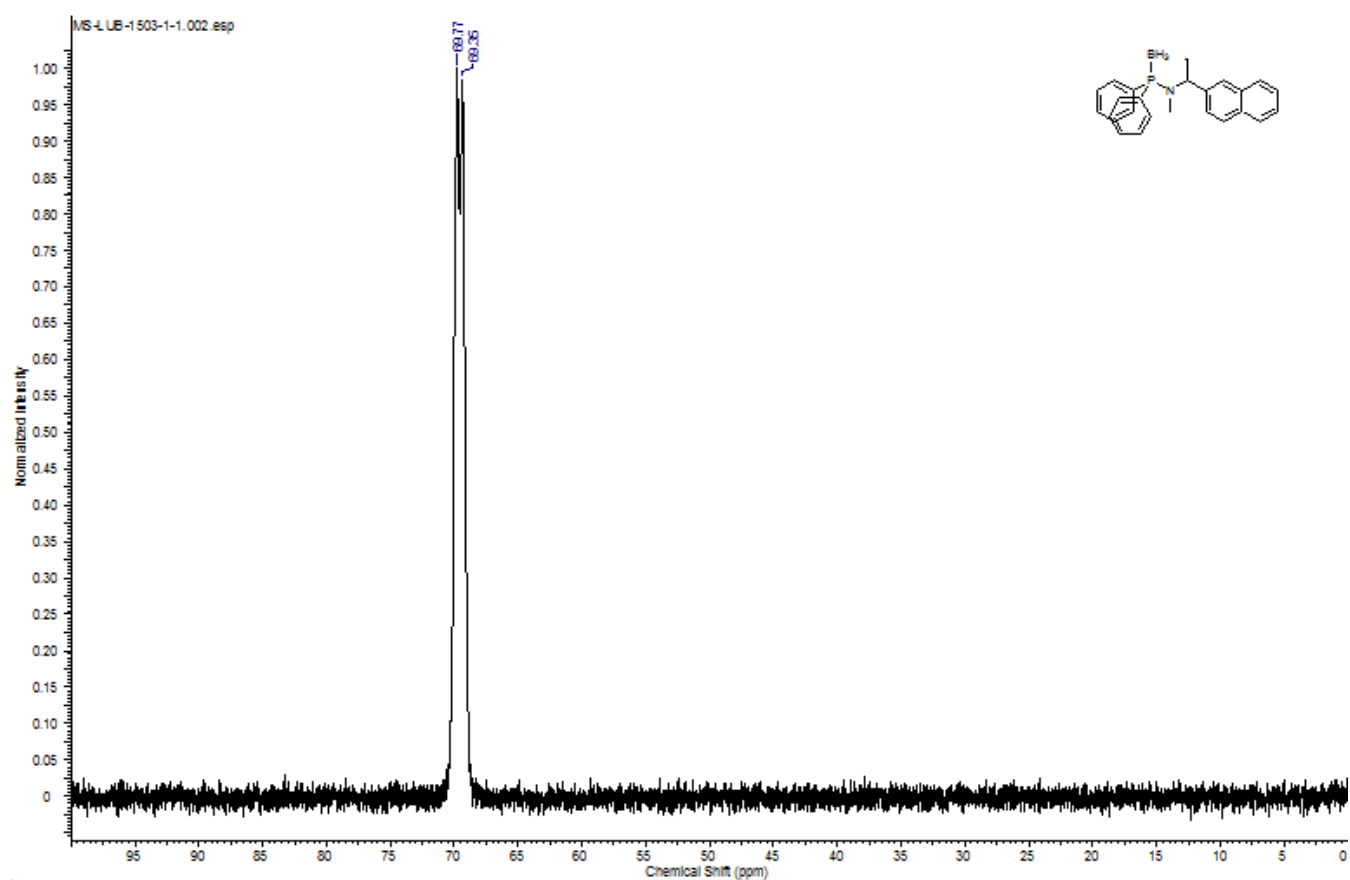
^{31}P NMR spectra of diphenylphosphinous acid-borane *N*-methyl-(*R*)-1-(1-naphthyl)ethylamide (**25e**) (202 MHz, CDCl_3)



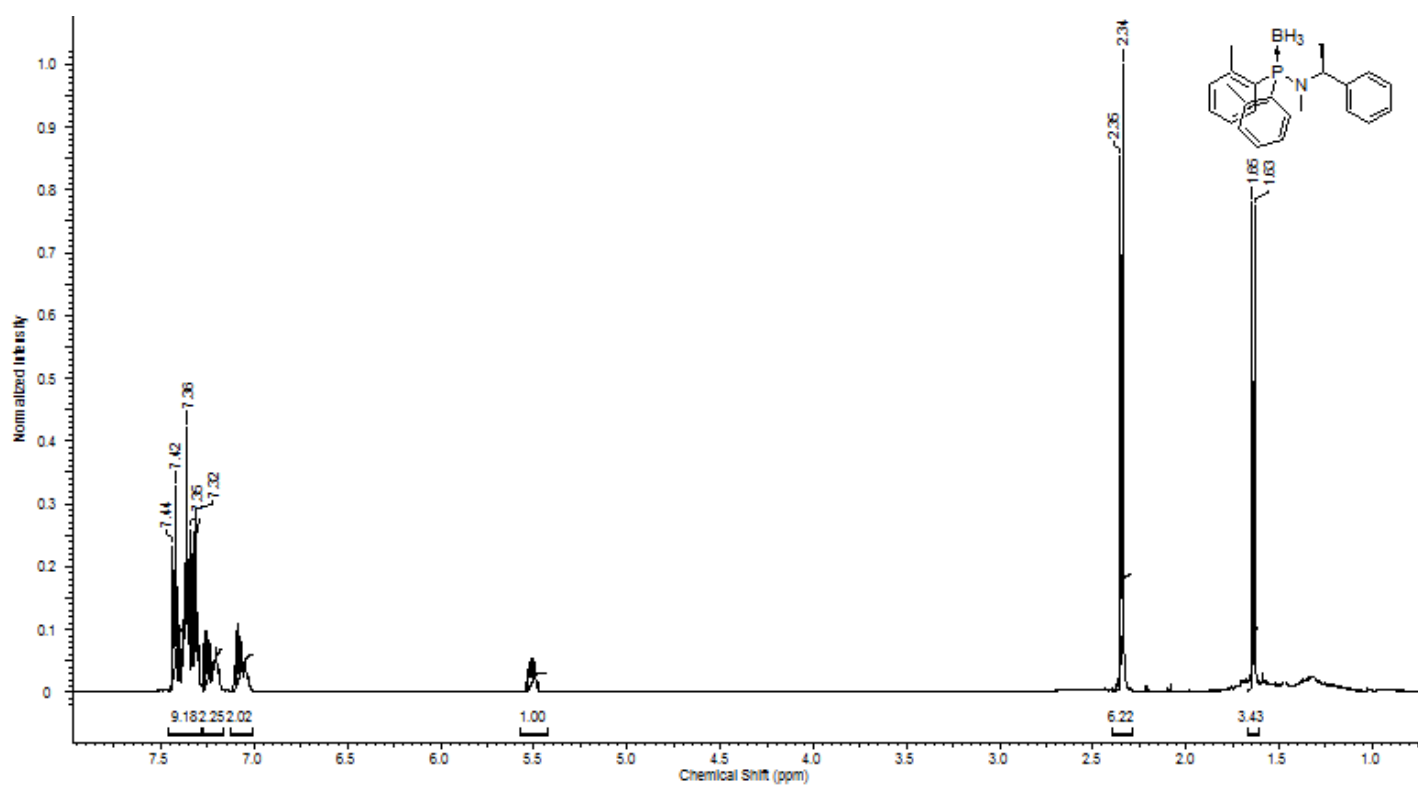
^1H NMR spectra of diphenylphosphinous acid-borane *N*-methyl-(*S*)-1-(2-naphthyl)ethylamide (**25f**) (500 MHz, CDCl_3)



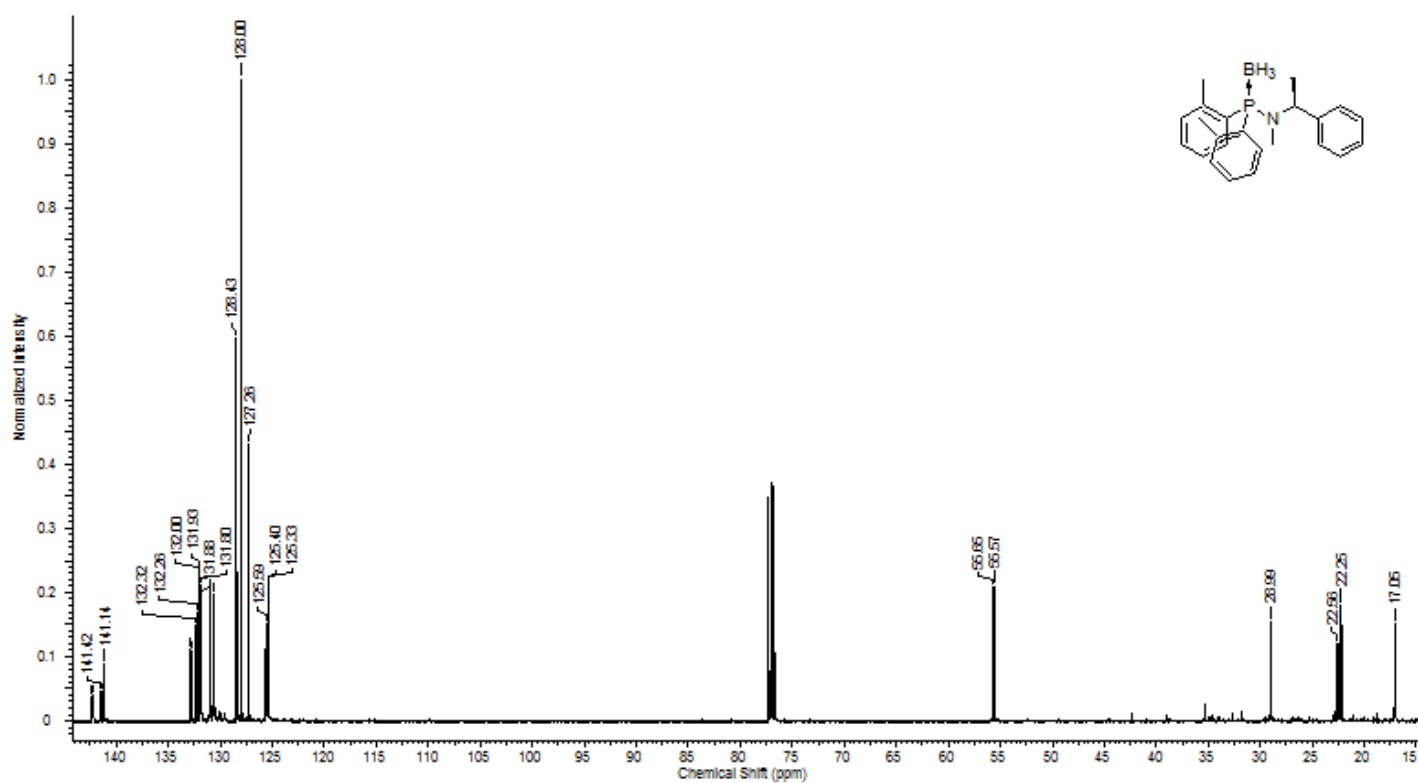
¹³C NMR spectra of diphenylphosphinous acid-borane *N*-methyl-(*S*)-1-(2-naphthyl)ethylamide (**25f**) (126 MHz, CDCl₃)



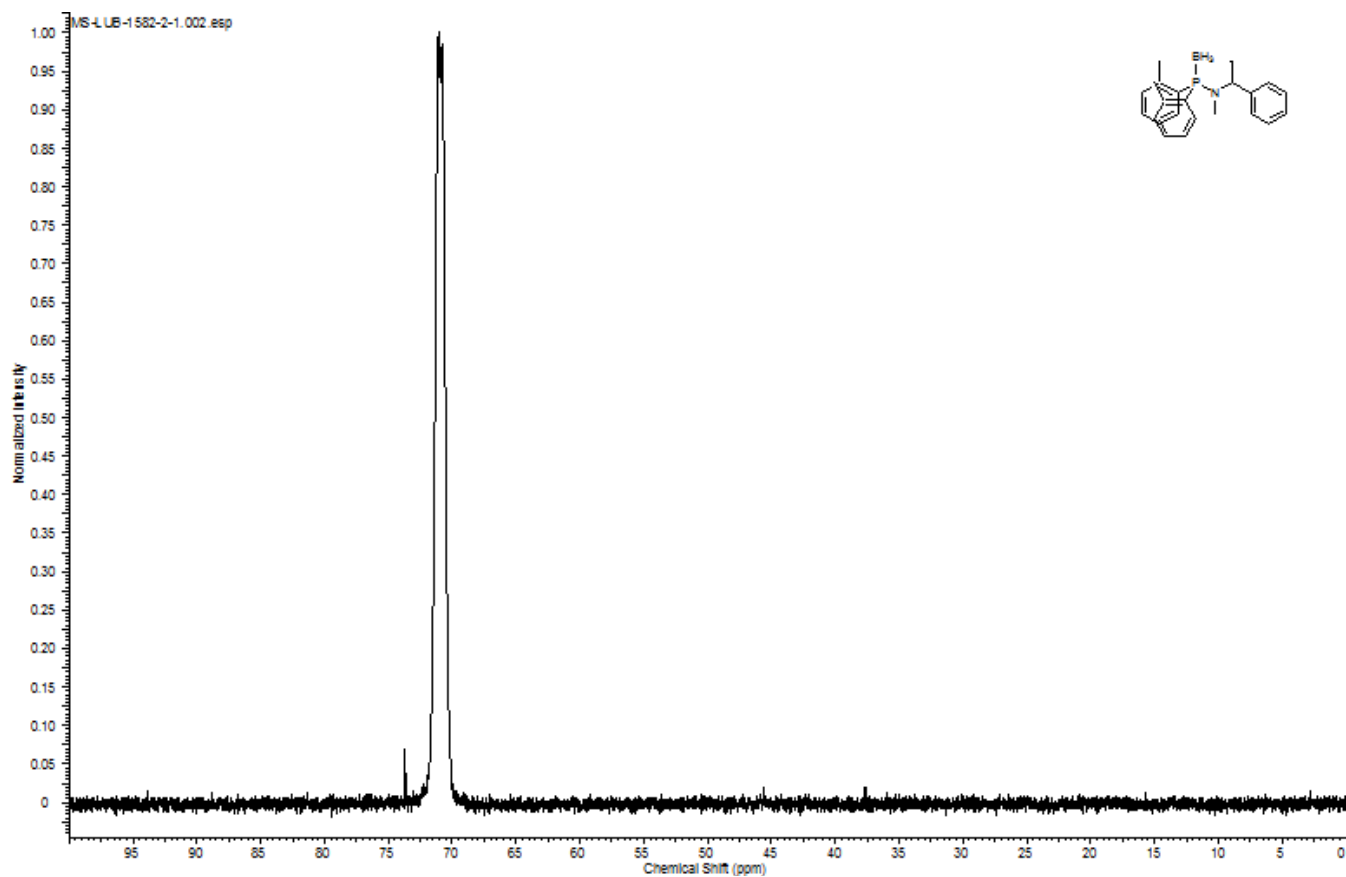
³¹P NMR spectra of diphenylphosphinous acid-borane *N*-methyl-(*S*)-1-(2-naphthyl)ethylamide (**25f**) (202 MHz, CDCl₃)



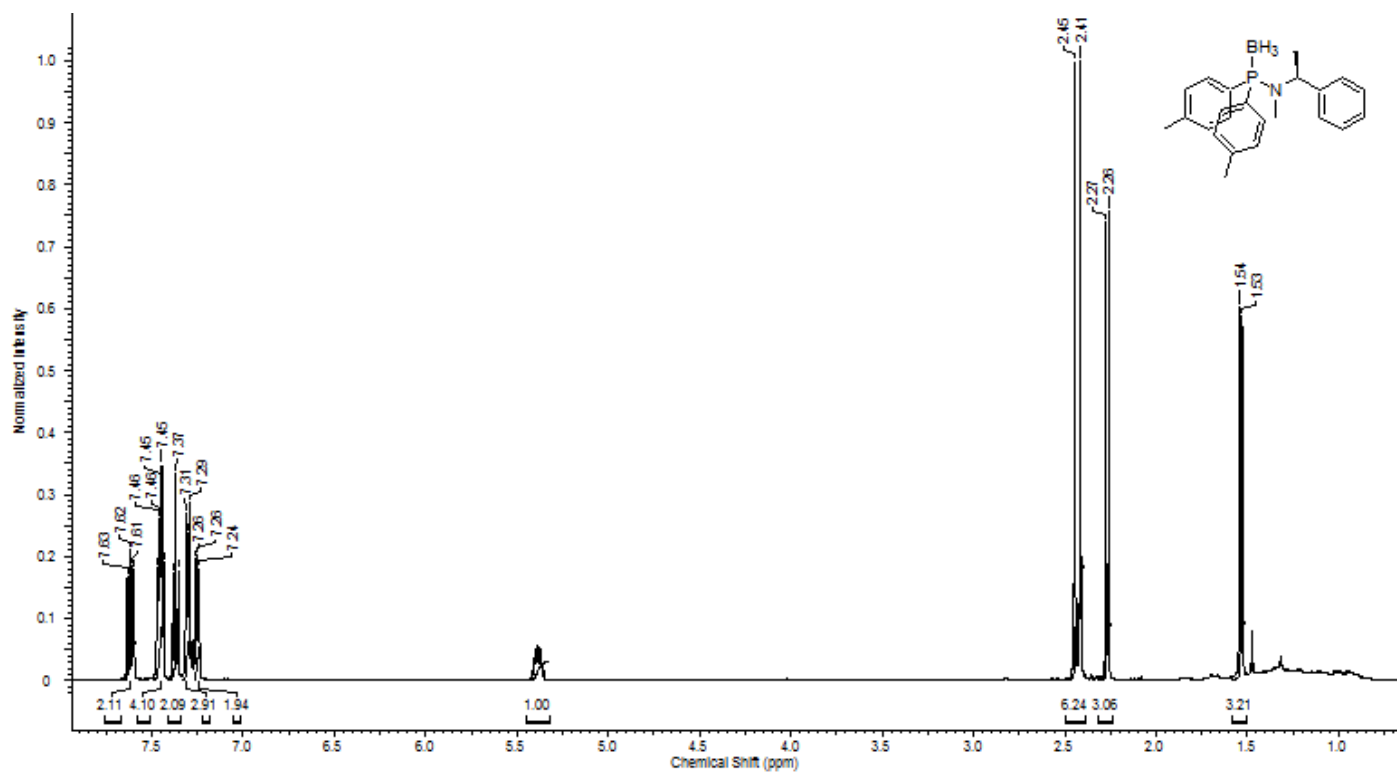
¹H NMR spectra of di(*o*-tolyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**33a**) (500 MHz, CDCl₃)



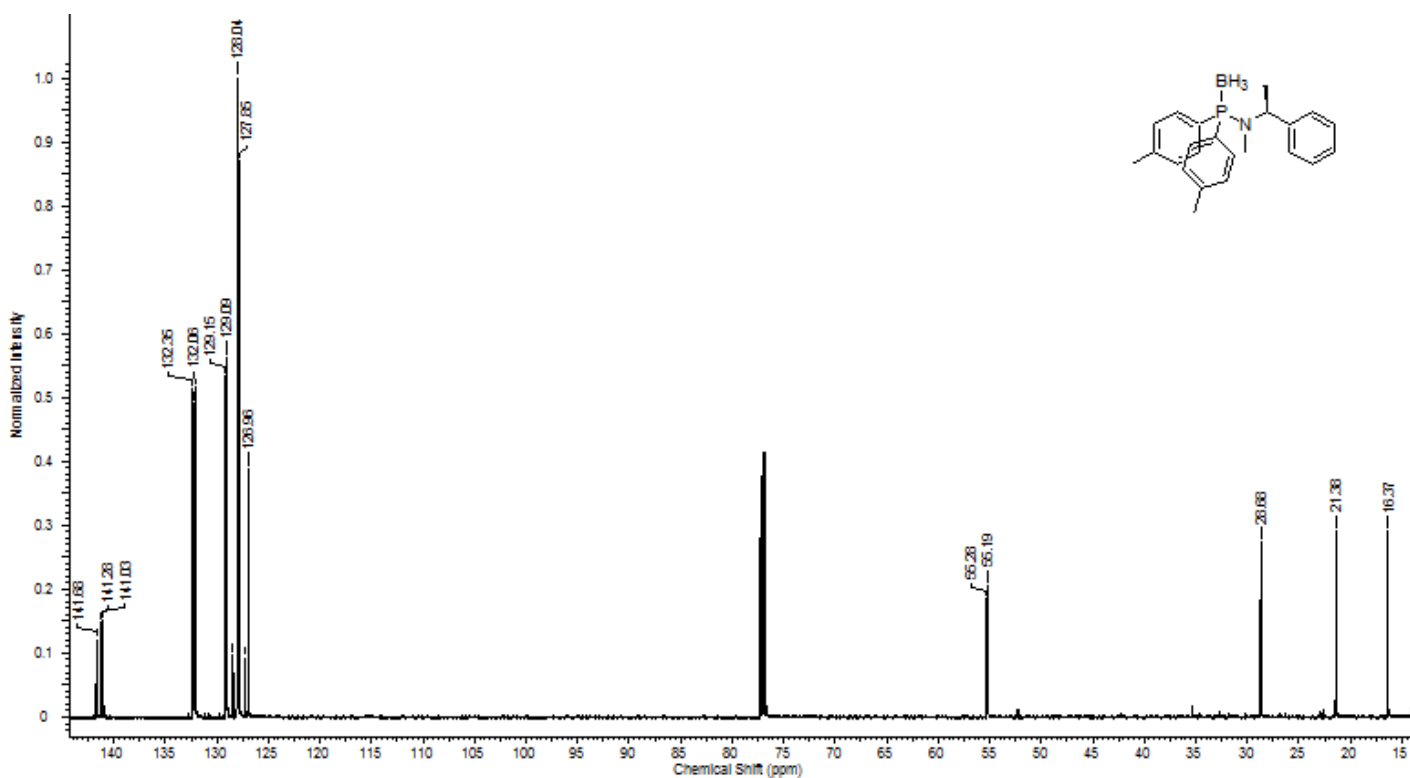
¹³C NMR spectra of di(*o*-tolyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**33a**) (126 MHz, CDCl₃)



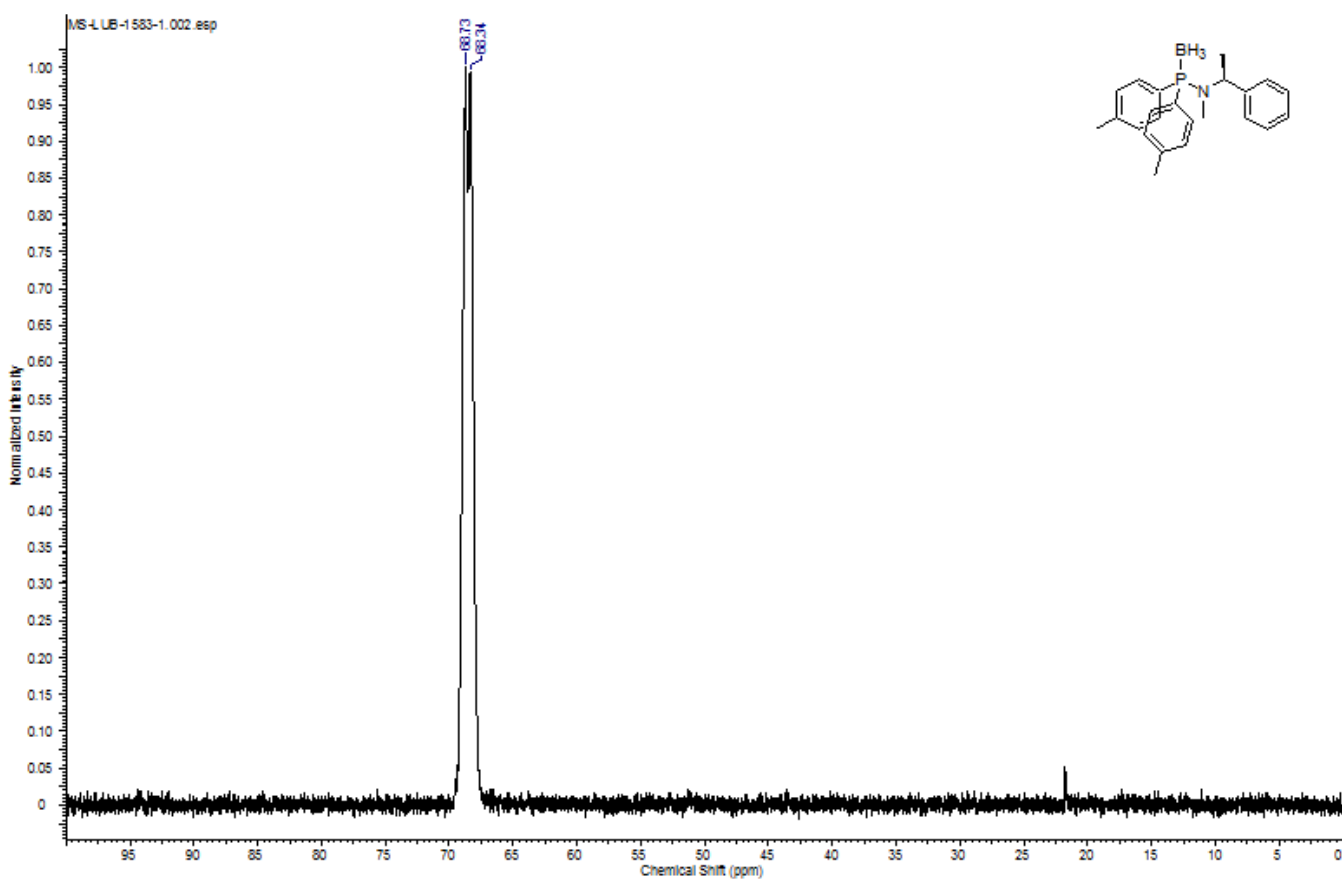
^{31}P NMR spectra of di(*o*-tolyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**33a**) (202 MHz, CDCl_3)



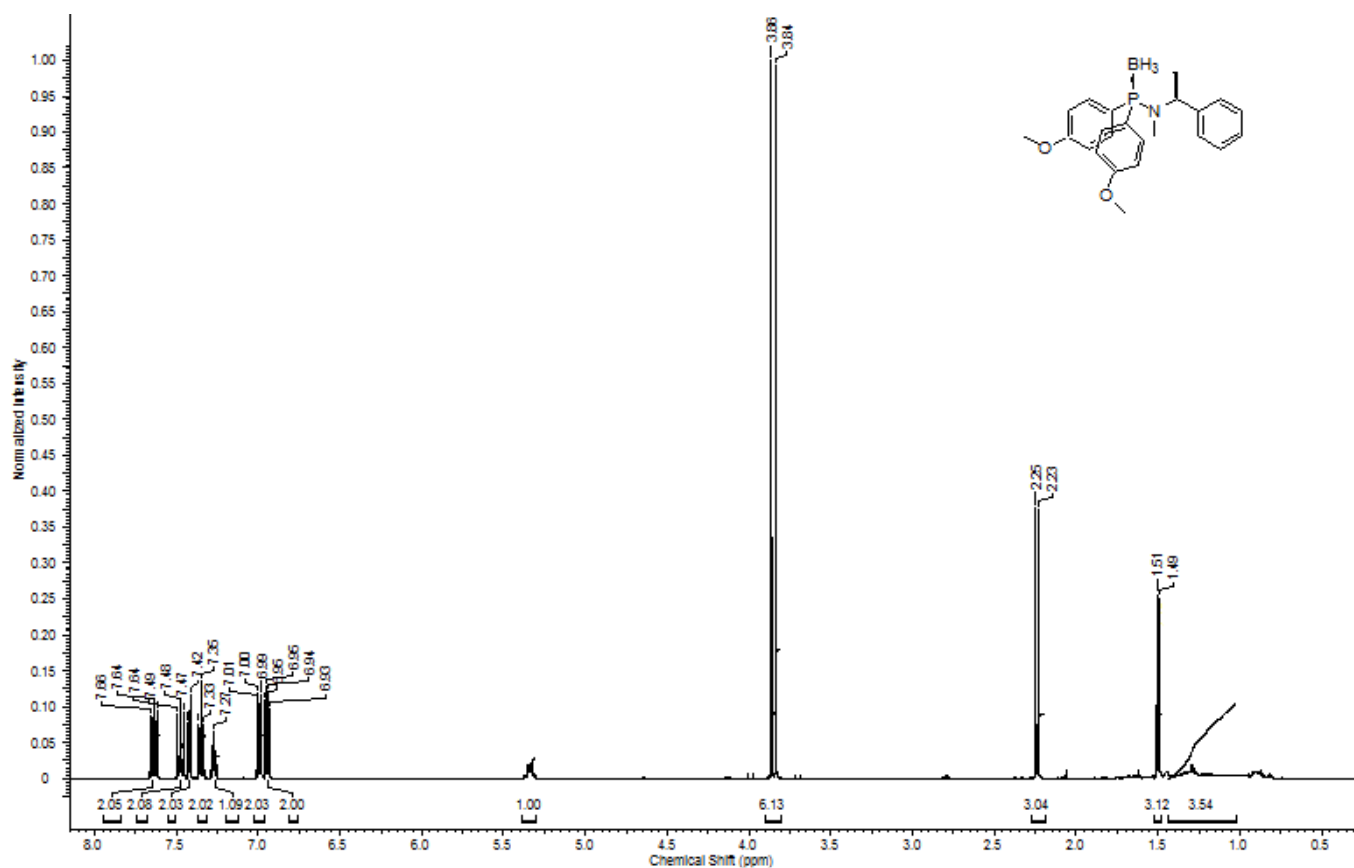
^1H NMR spectra of di(*p*-tolyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**33b**) (500 MHz, CDCl_3)



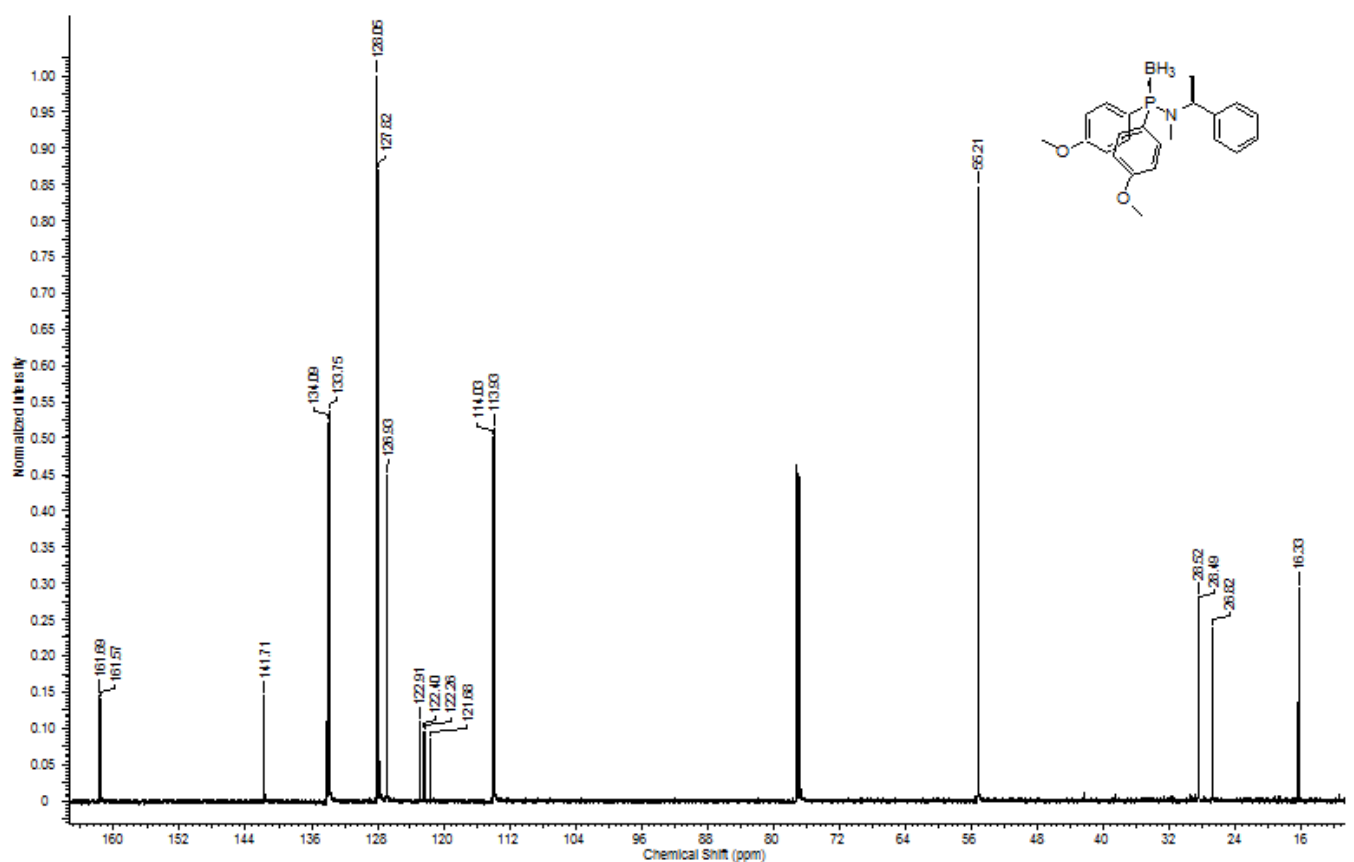
^{13}C NMR spectra of di(*p*-tolyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**33b**) (126 MHz, CDCl_3)



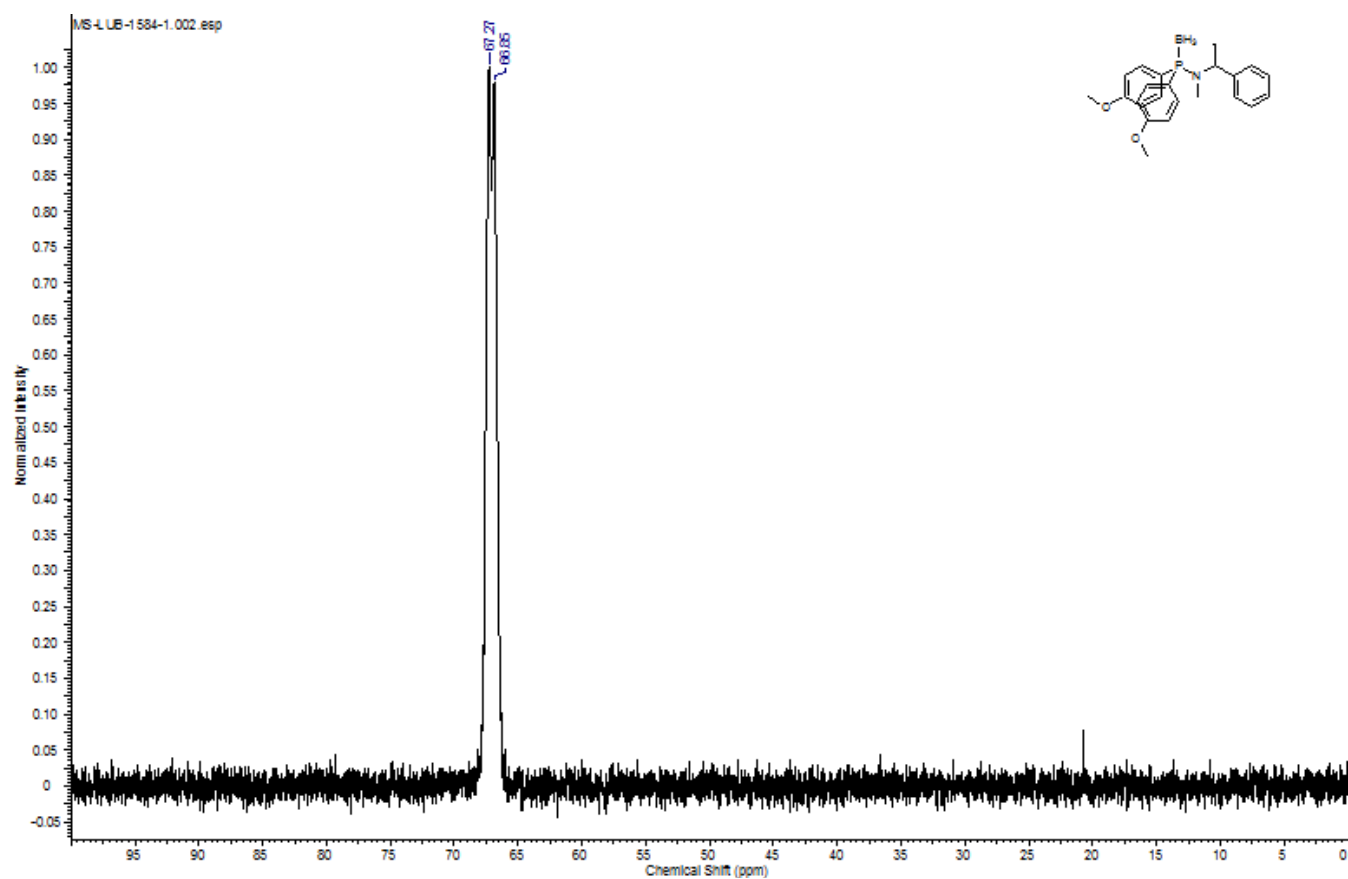
^{31}P NMR spectra of di(*p*-tolyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**33b**) (202 MHz, CDCl_3)



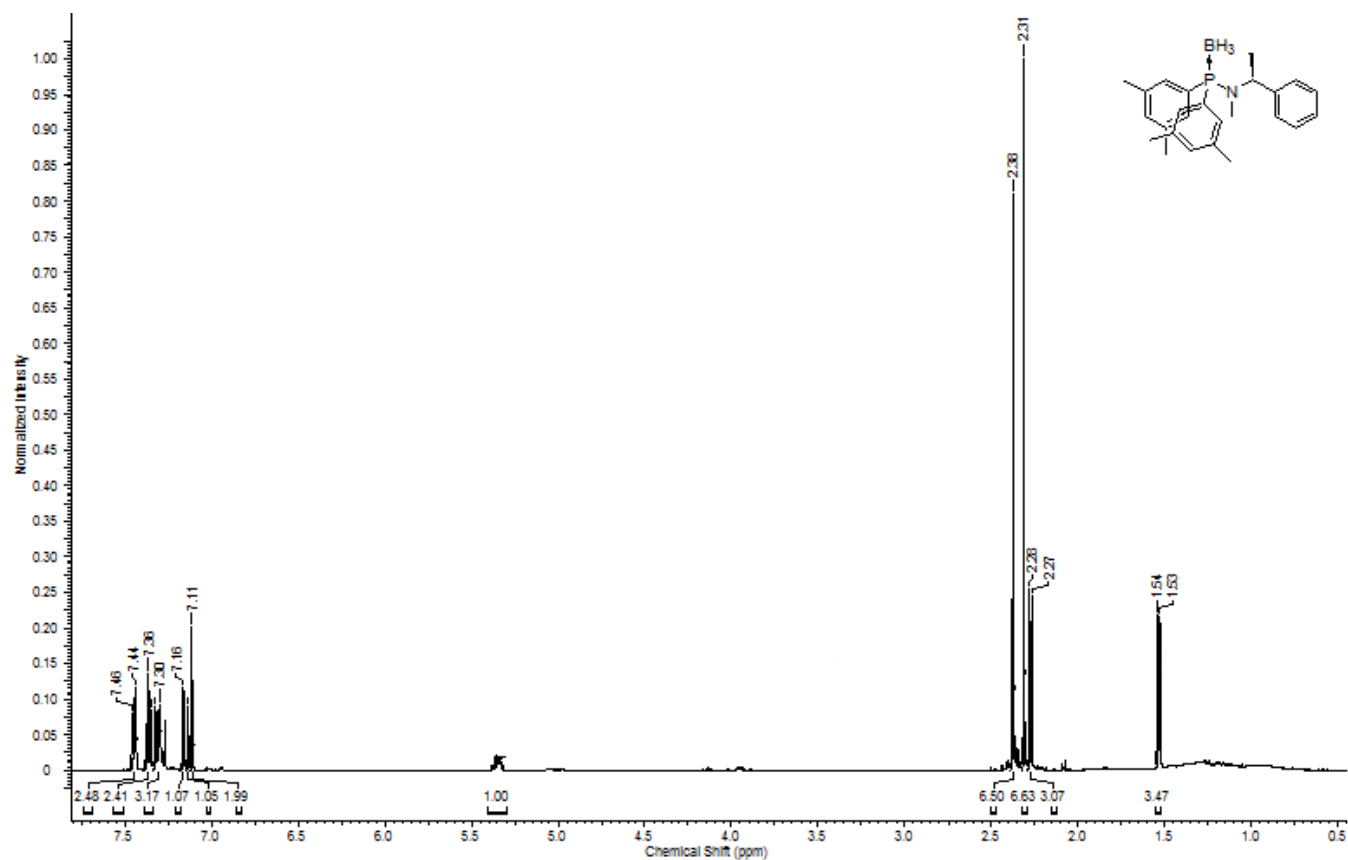
¹H NMR spectra of di(*p*-anisyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**33c**) (500 MHz, CDCl₃)



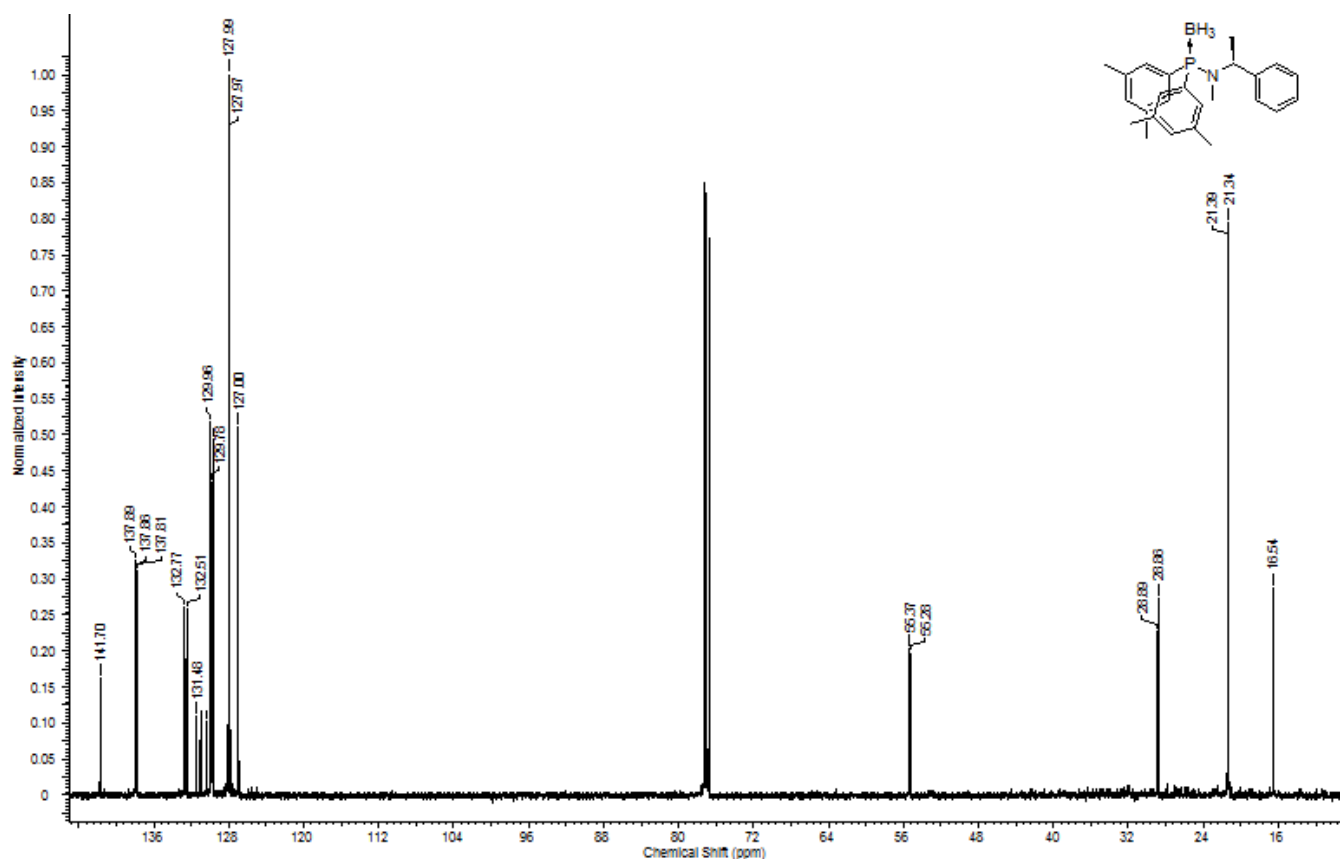
¹³C NMR spectra of di(*p*-anisyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**33c**) (126 MHz, CDCl₃)



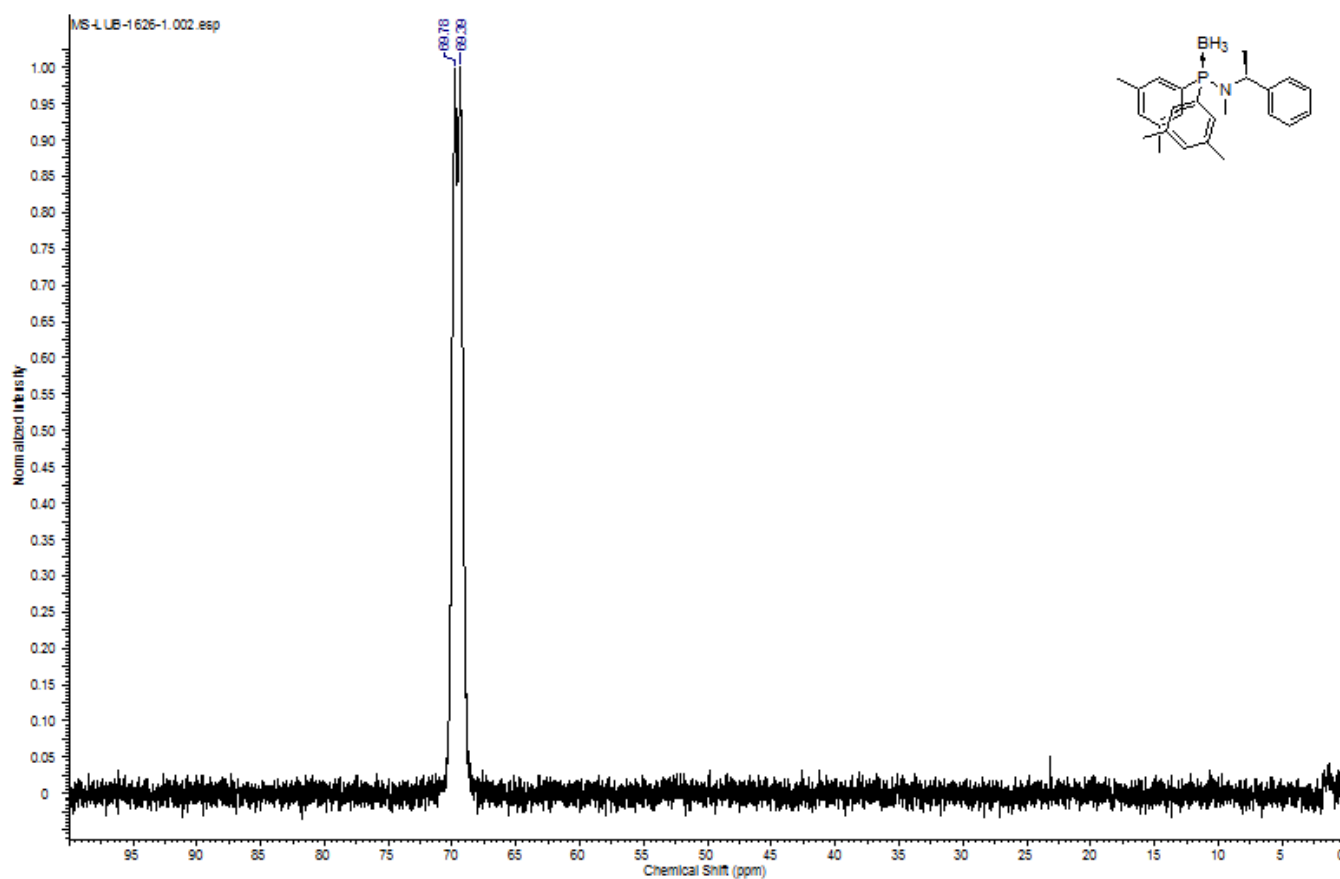
^{31}P NMR spectra of di(*p*-anisyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**33c**) (202 MHz, CDCl_3)



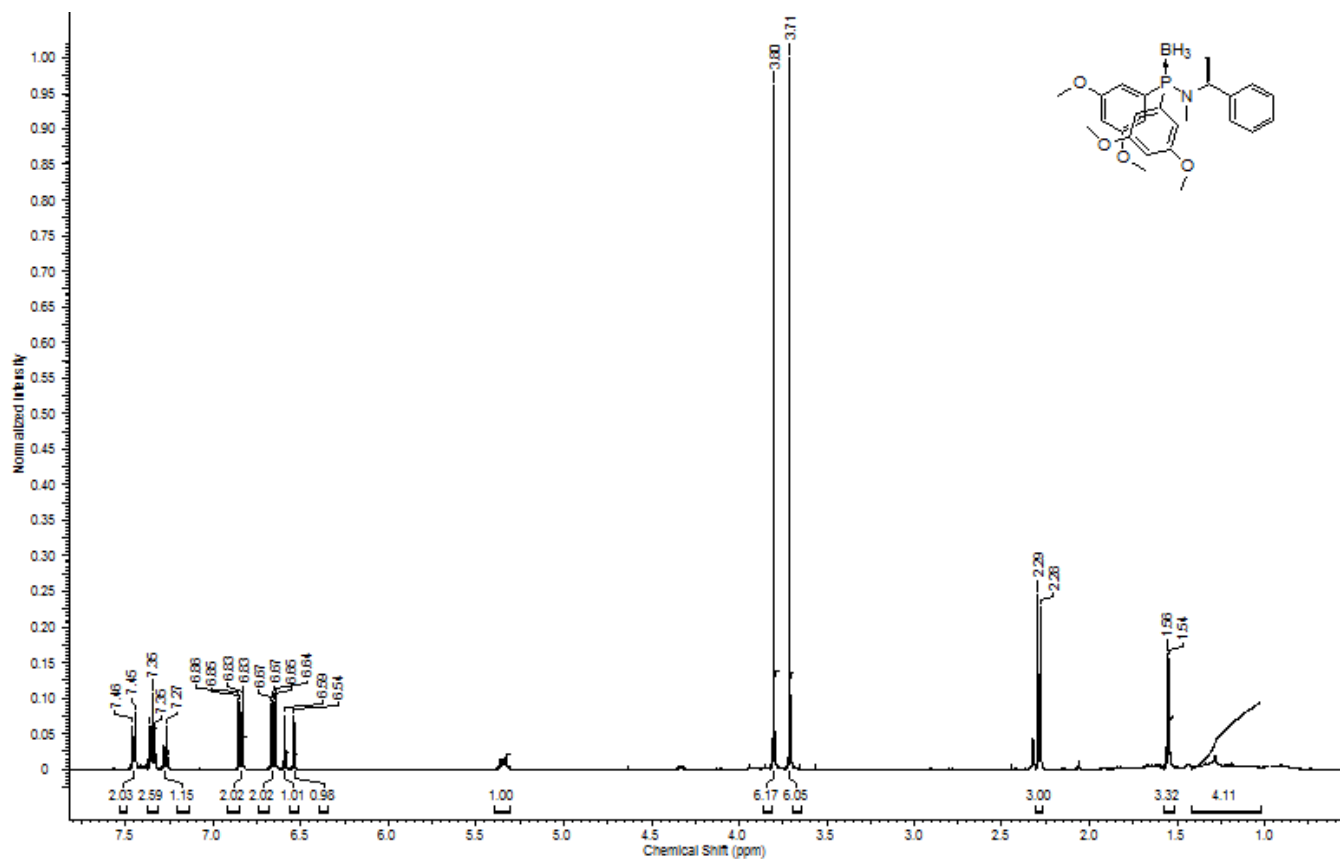
^1H NMR spectra of di(3,5-dimethylphenyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**33d**) (500 MHz, CDCl_3)



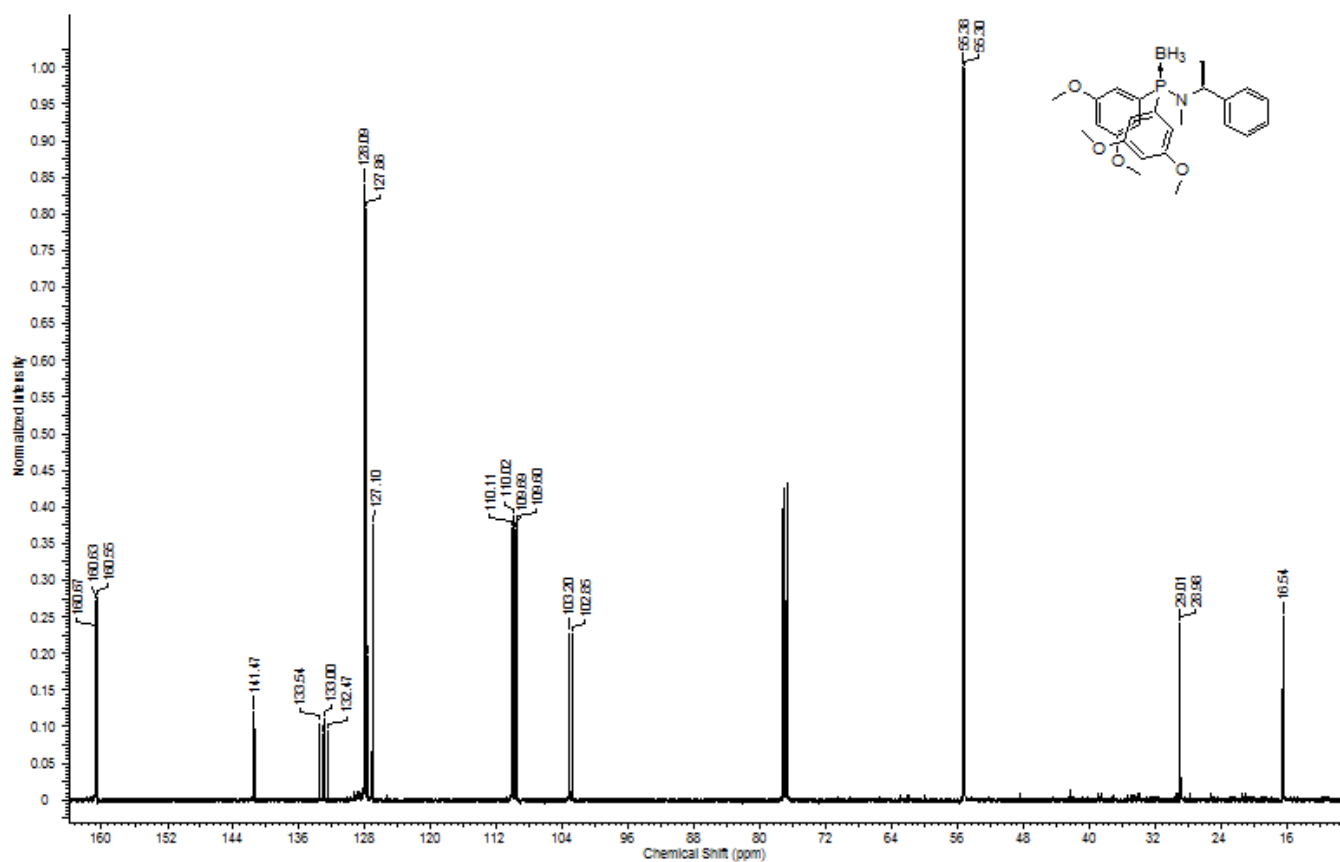
^{13}C NMR spectra of di(3,5-dimethylphenyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**33d**) (126 MHz, CDCl_3)



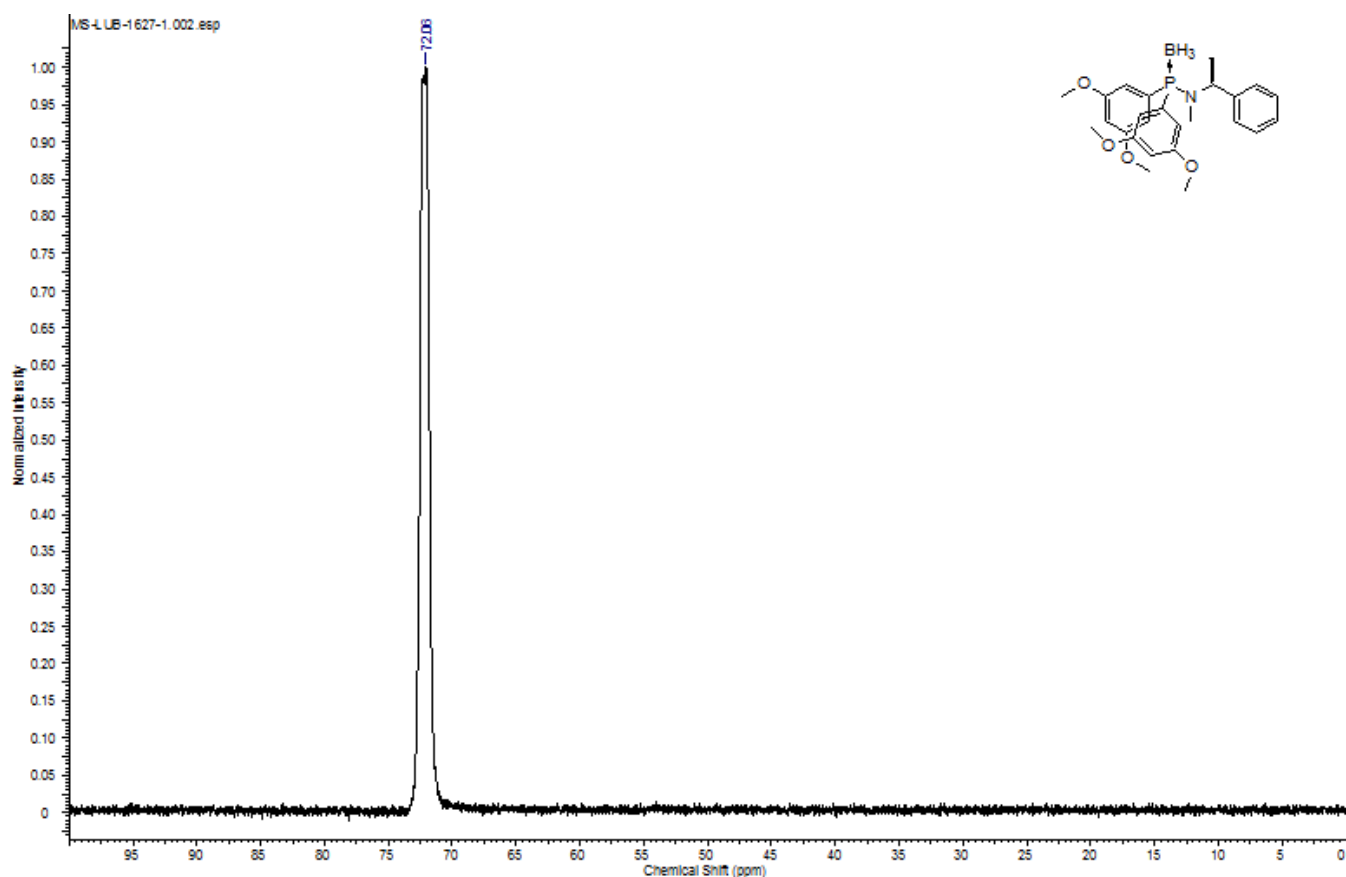
^{31}P NMR spectra of di(3,5-dimethylphenyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**33d**) (202 MHz, CDCl_3)



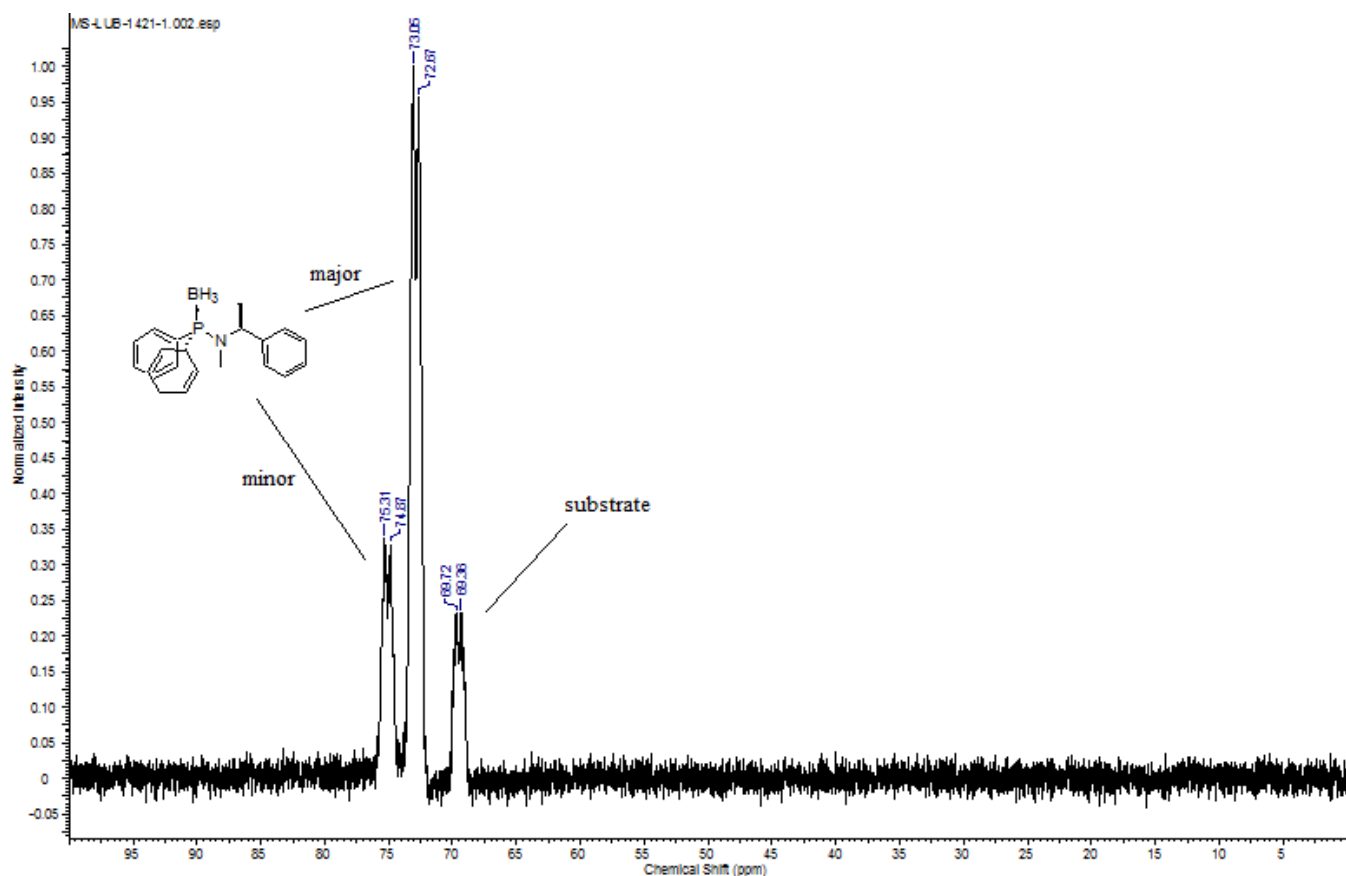
¹H NMR spectra of di(3,5-dimethoxyphenyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**33e**) (500 MHz, CDCl₃)



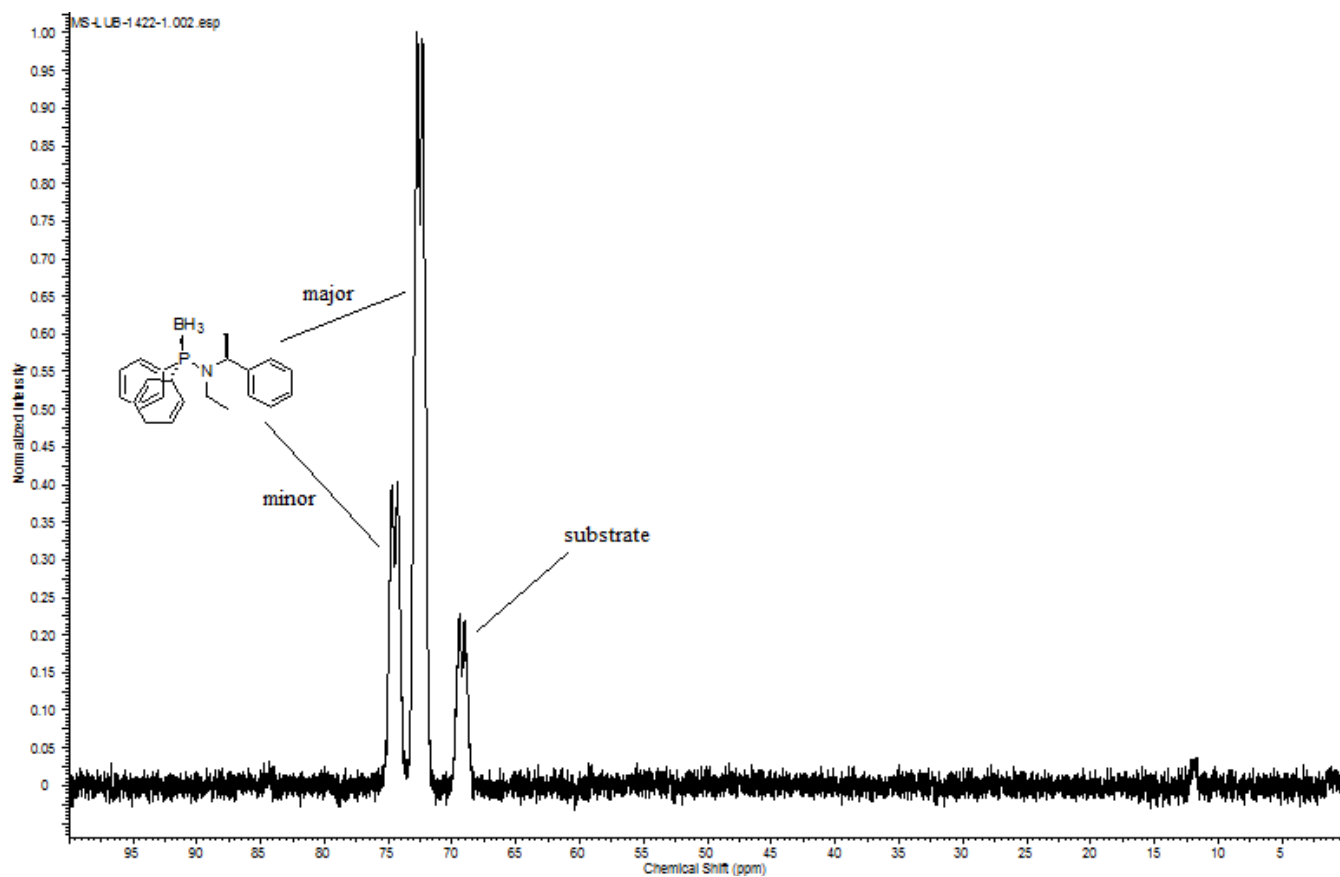
¹³C NMR spectra of di(3,5-dimethoxyphenyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**33e**) (126 MHz, CDCl₃)



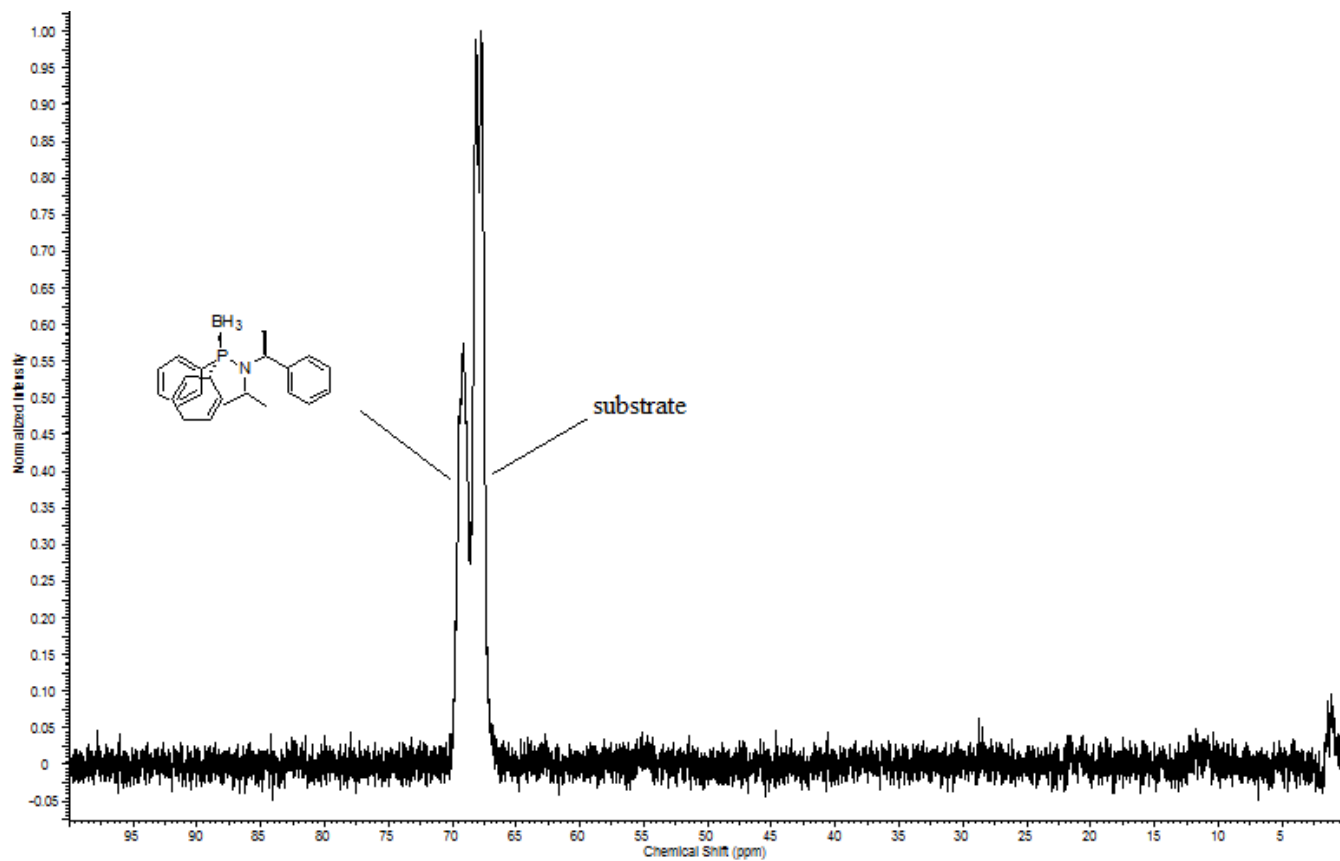
^{31}P NMR spectra of di(3,5-dimethoxyphenyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**33e**) (202 MHz, CDCl_3)



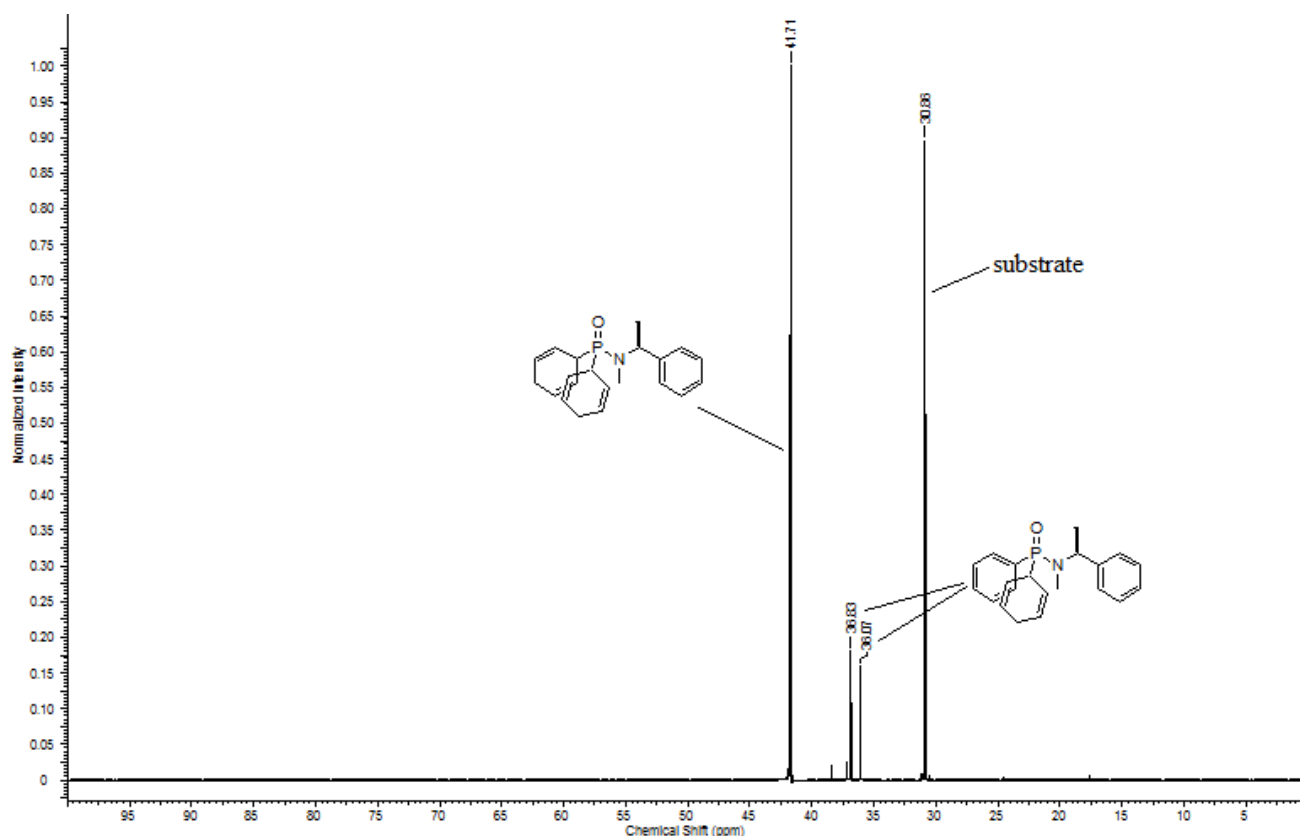
^{31}P NMR spectra of (1,4-cyclohexadien-3-yl)phenylphosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**13b**) (2 diastereomers) along with starting material **10b** (202 MHz, CDCl_3)



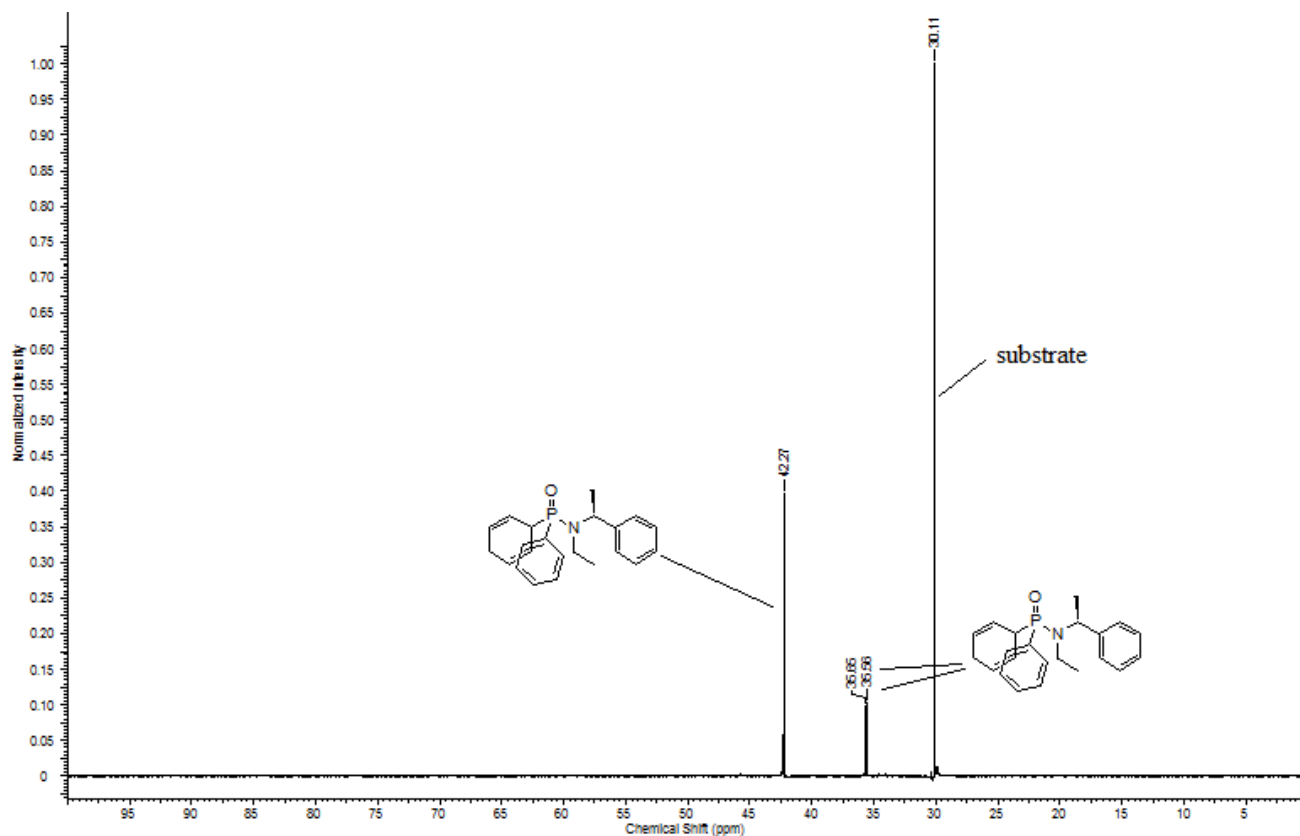
³¹P NMR spectra of (1,4-cyclohexadien-3-yl)phenylphosphinous acid-borane *N*-ethyl-(*S*)-α-methylbenzylamide (**13c**) (2 diastereomers) along with starting material **10c** (202 MHz, CDCl₃)



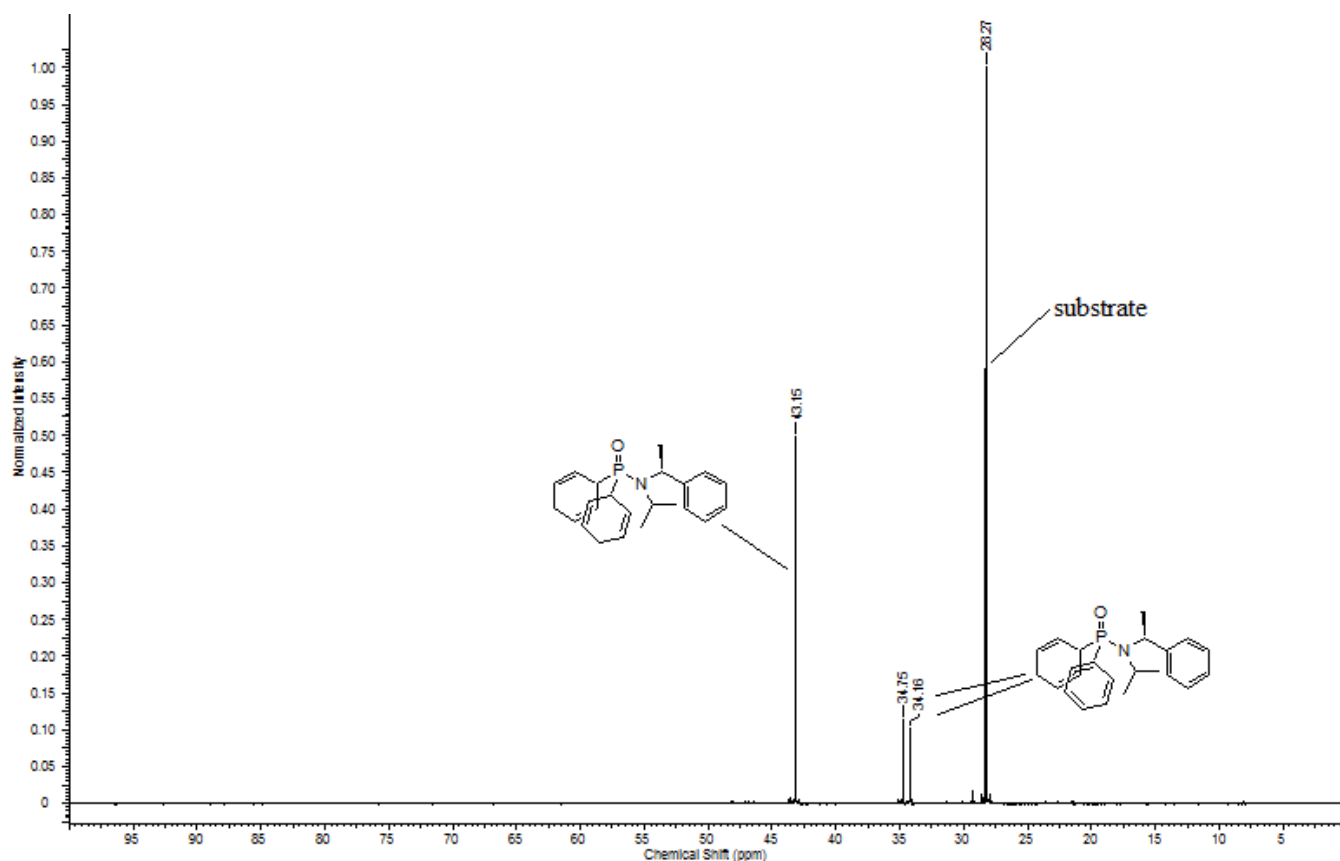
³¹P NMR spectra of (1,4-cyclohexadien-3-yl)phenylphosphinous acid-borane *N*-isopropyl-(*S*)-α-methylbenzylamide (**13d**) along with starting material **10d** (202 MHz, CDCl₃)



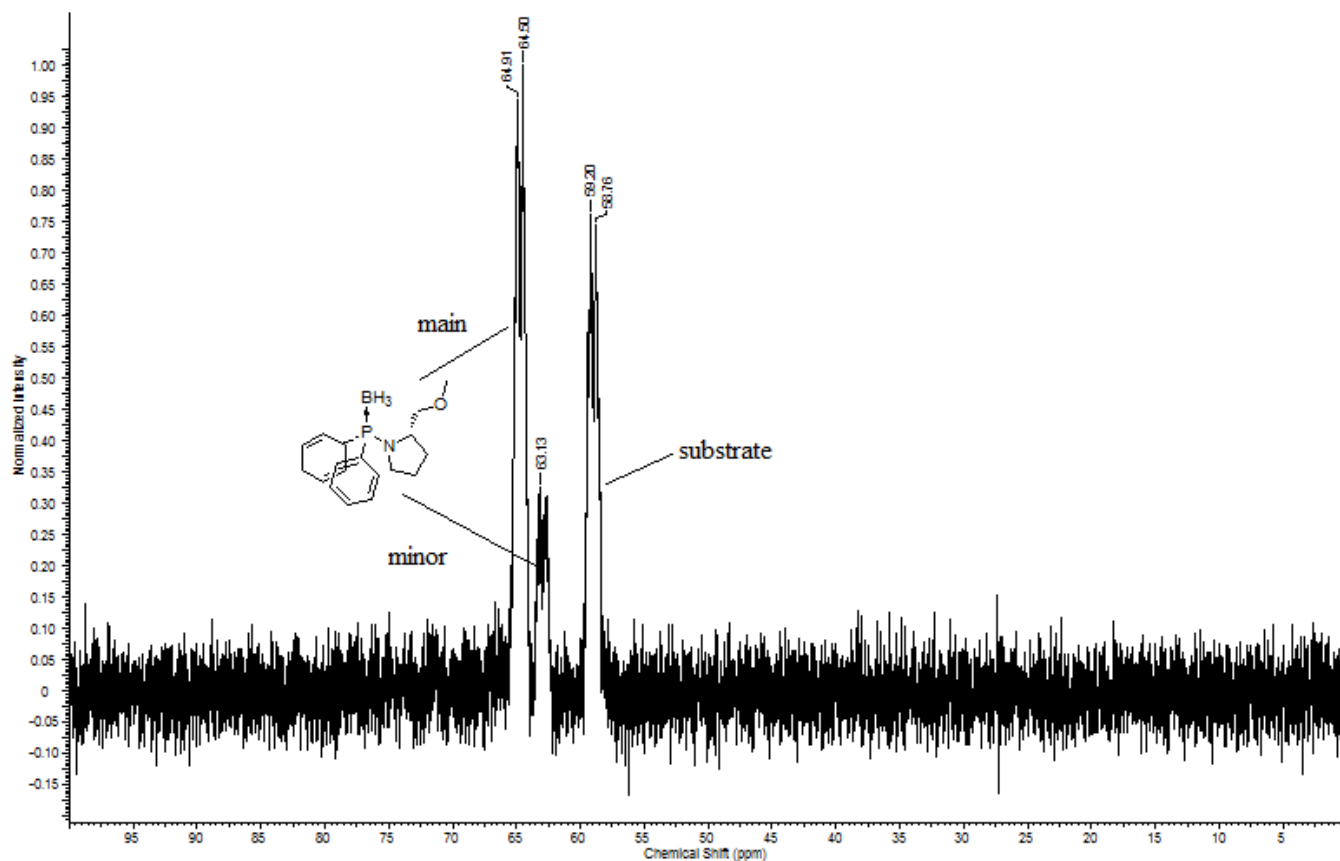
³¹P NMR spectra of (1,4-cyclohexadien-3-yl)phenylphosphinic acid *N*-methyl-(*S*)- α -methylbenzylamide (**14b**) along with bis(1,4-cyclohexadien-3-yl)phosphinic acid *N*-methyl-(*S*)- α -methylbenzylamide (**15b**) and starting material **11b** (202 MHz, CDCl₃)



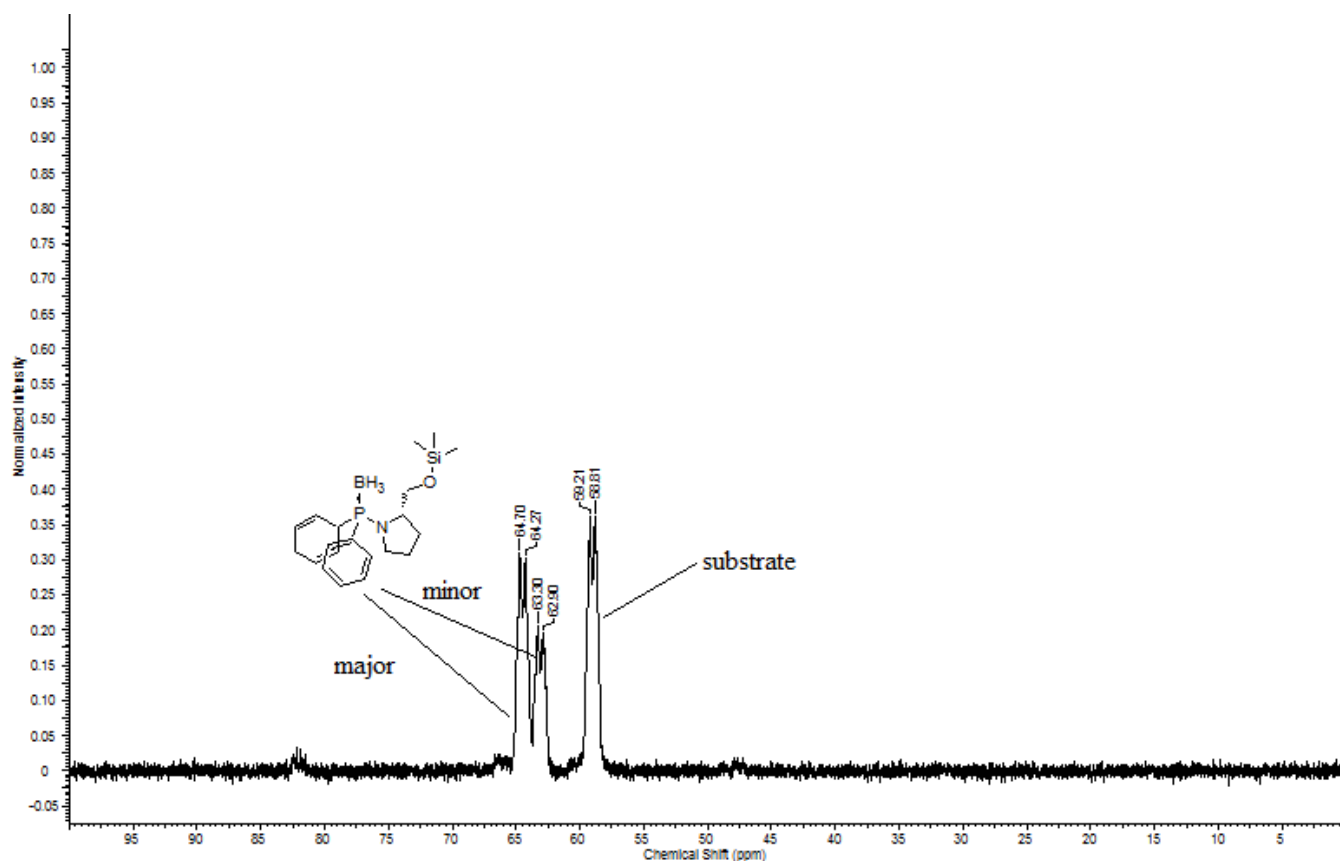
³¹P NMR spectra of (1,4-cyclohexadien-3-yl)phenylphosphinic acid *N*-ethyl-(*S*)- α -methylbenzylamide (**14c**) along with bis(1,4-cyclohexadien-3-yl)phosphinic acid *N*-ethyl-(*S*)- α -methylbenzylamide (**15c**) and starting material **11c** (202 MHz, CDCl₃)



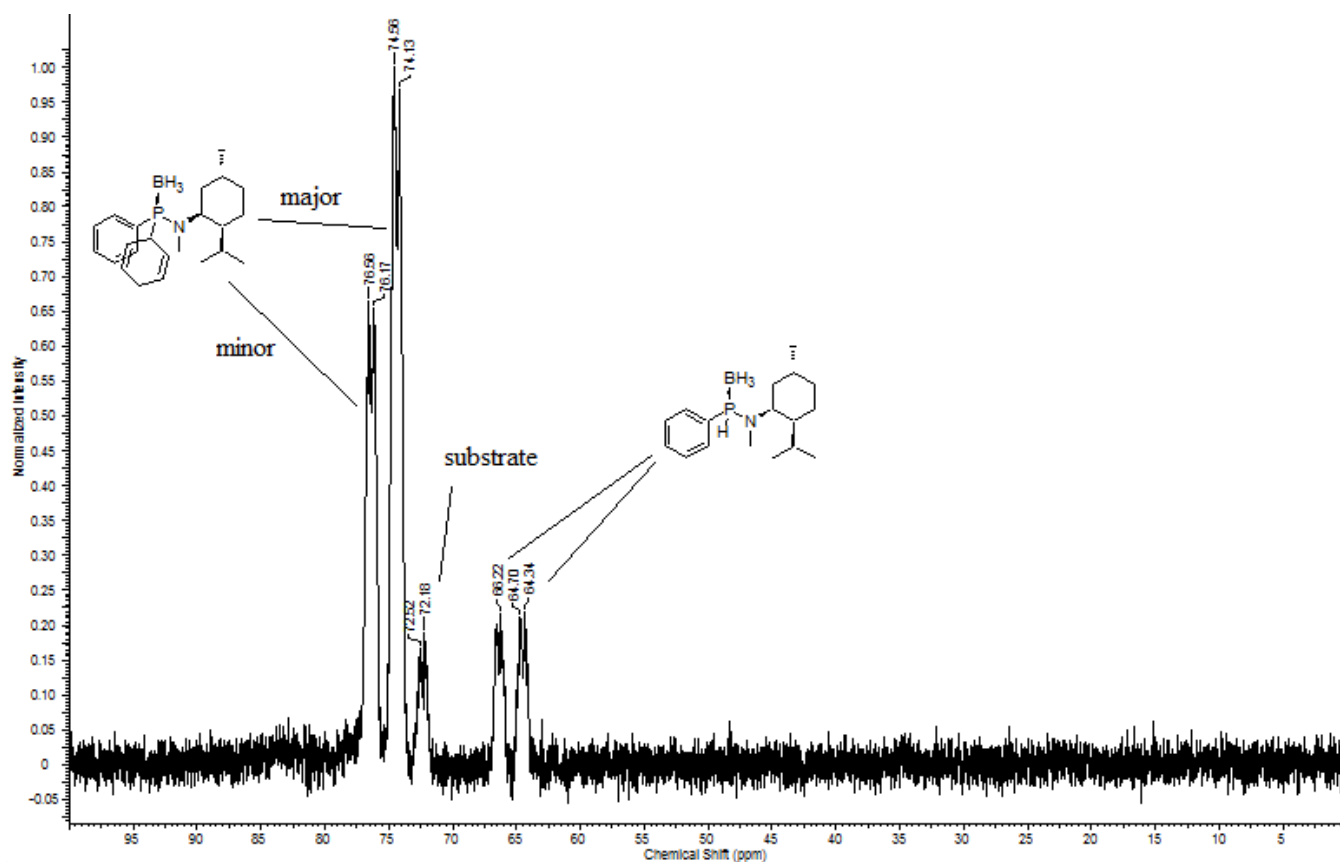
³¹P NMR spectra of (1,4-cyclohexadien-3-yl)phenylphosphinic acid *N*-isopropyl-(*S*)- α -methylbenzylamide (**14d**) along with bis(1,4-cyclohexadien-3-yl)phosphinic acid *N*-isopropyl-(*S*)- α -methylbenzylamide (**15d**) and starting material **11d** (202 MHz, CDCl₃)



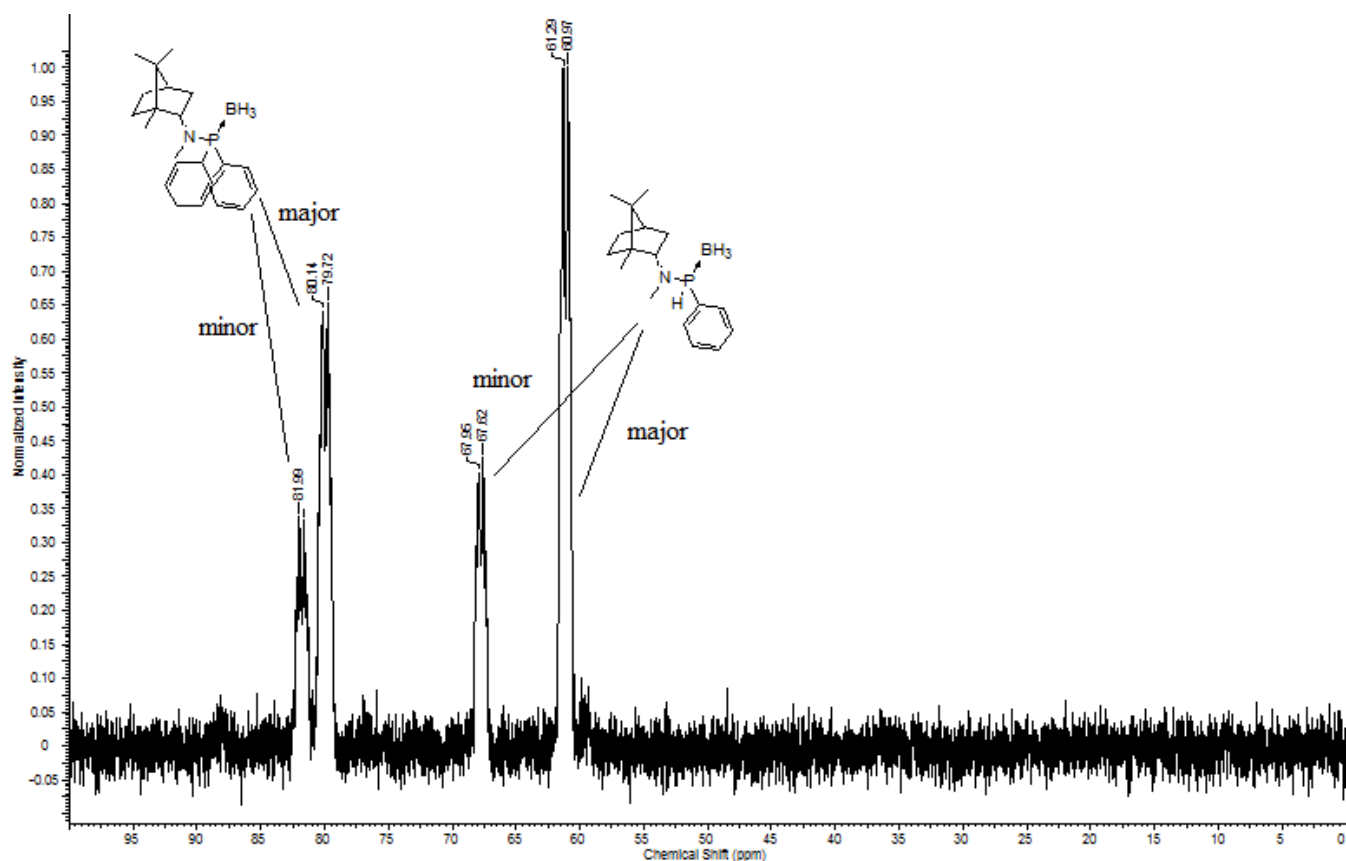
³¹P NMR spectra of (1,4-cyclohexadien-3-yl)phenylphosphinous acid-borane (2*S*)-2-(methoxymethyl)pyrrolidinamide (**26a**) along with starting material **16b** (202 MHz, CDCl₃)



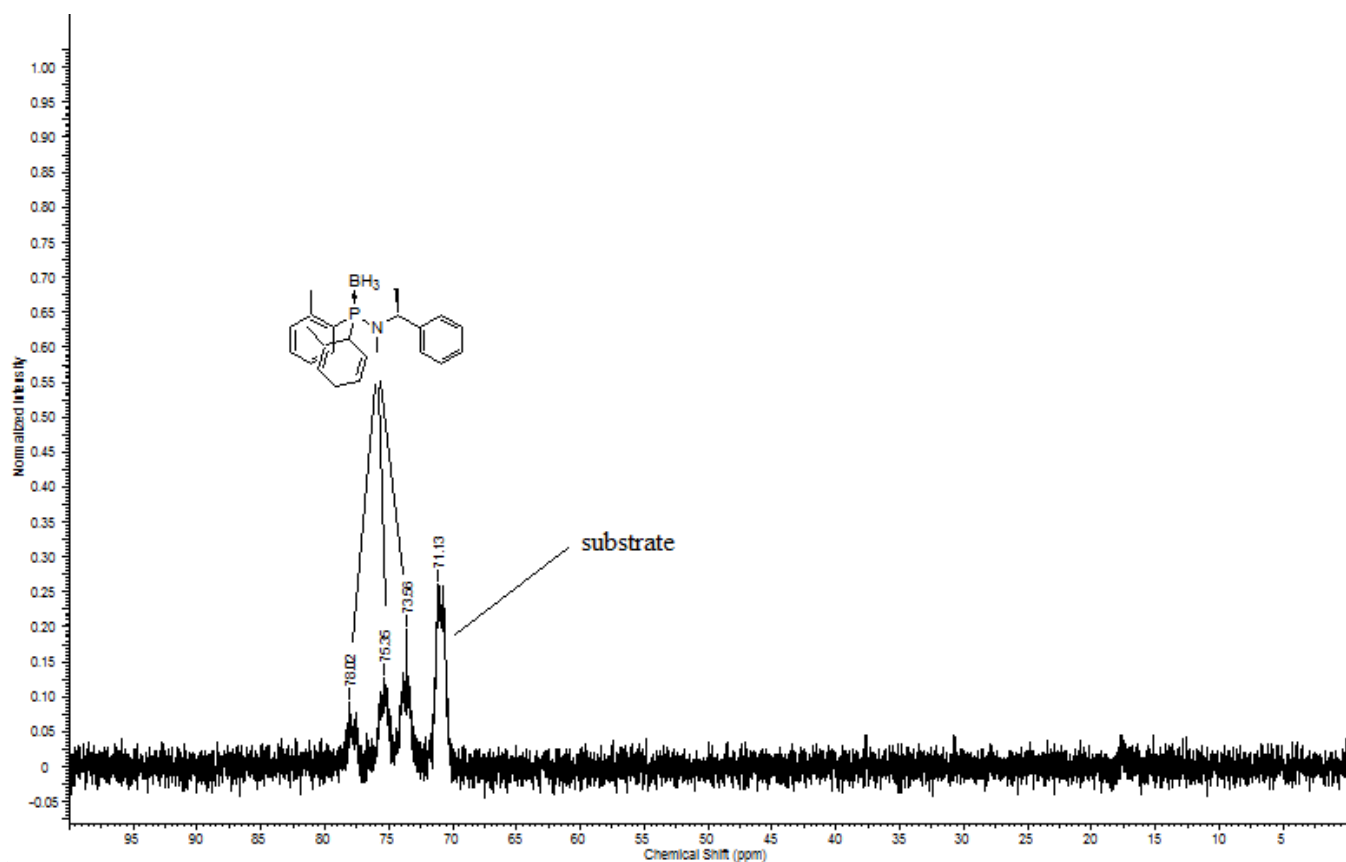
³¹P NMR spectra of (1,4-cyclohexadien-3-yl)phenylphosphinous acid-borane (2*S*)-2-(trimethylsilyloxymethyl)pyrrolidinamide (**26b**) along with starting material **16b** (202 MHz, CDCl₃)



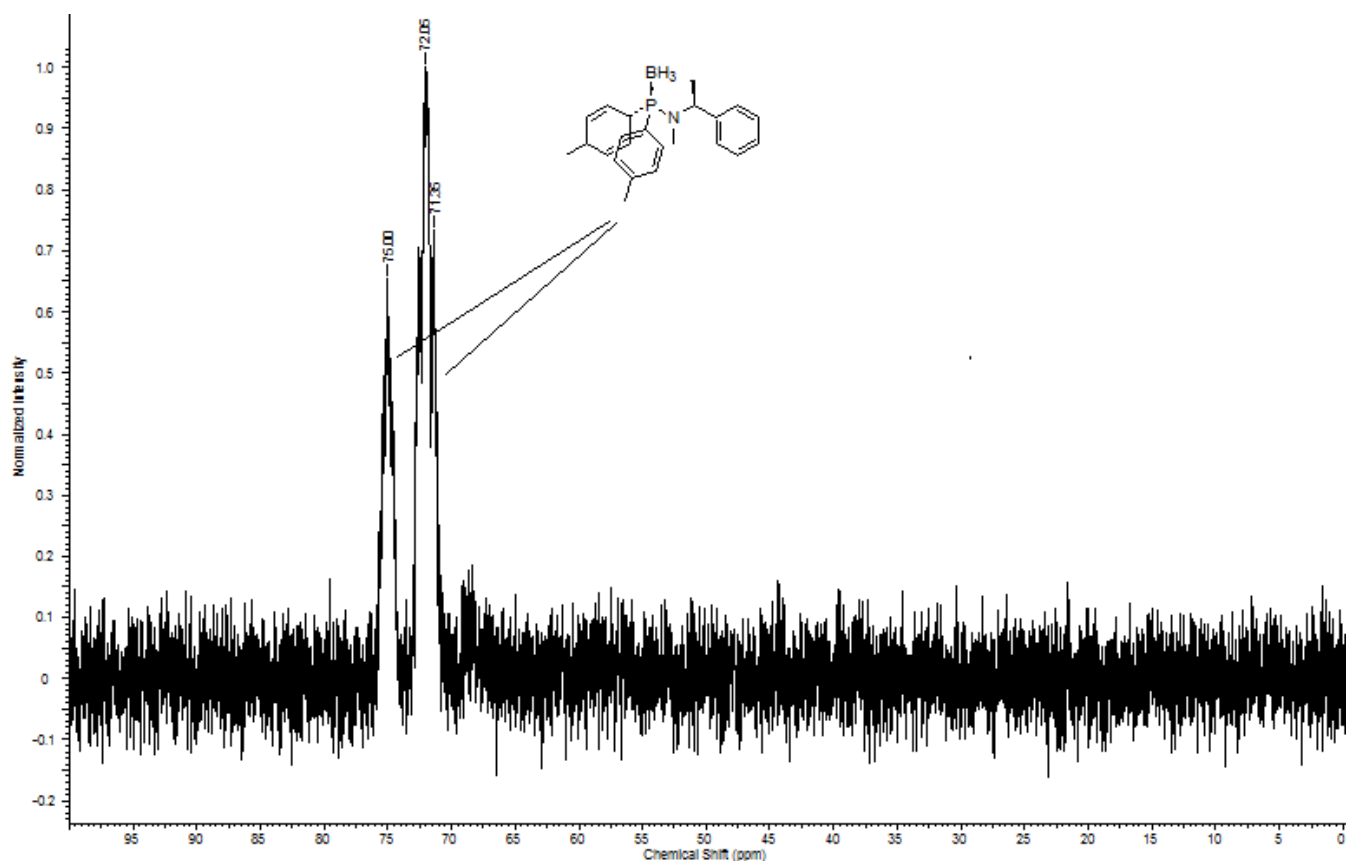
³¹P NMR spectra of (1,4-cyclohexadien-3-yl)phenylphosphinous acid-borane *N*-methylneomenthylamide (**26c**) along with phenylphosphinous acid-borane *N*-methylneomenthylamide (**28**) and starting material **16b** (202 MHz, CDCl₃)



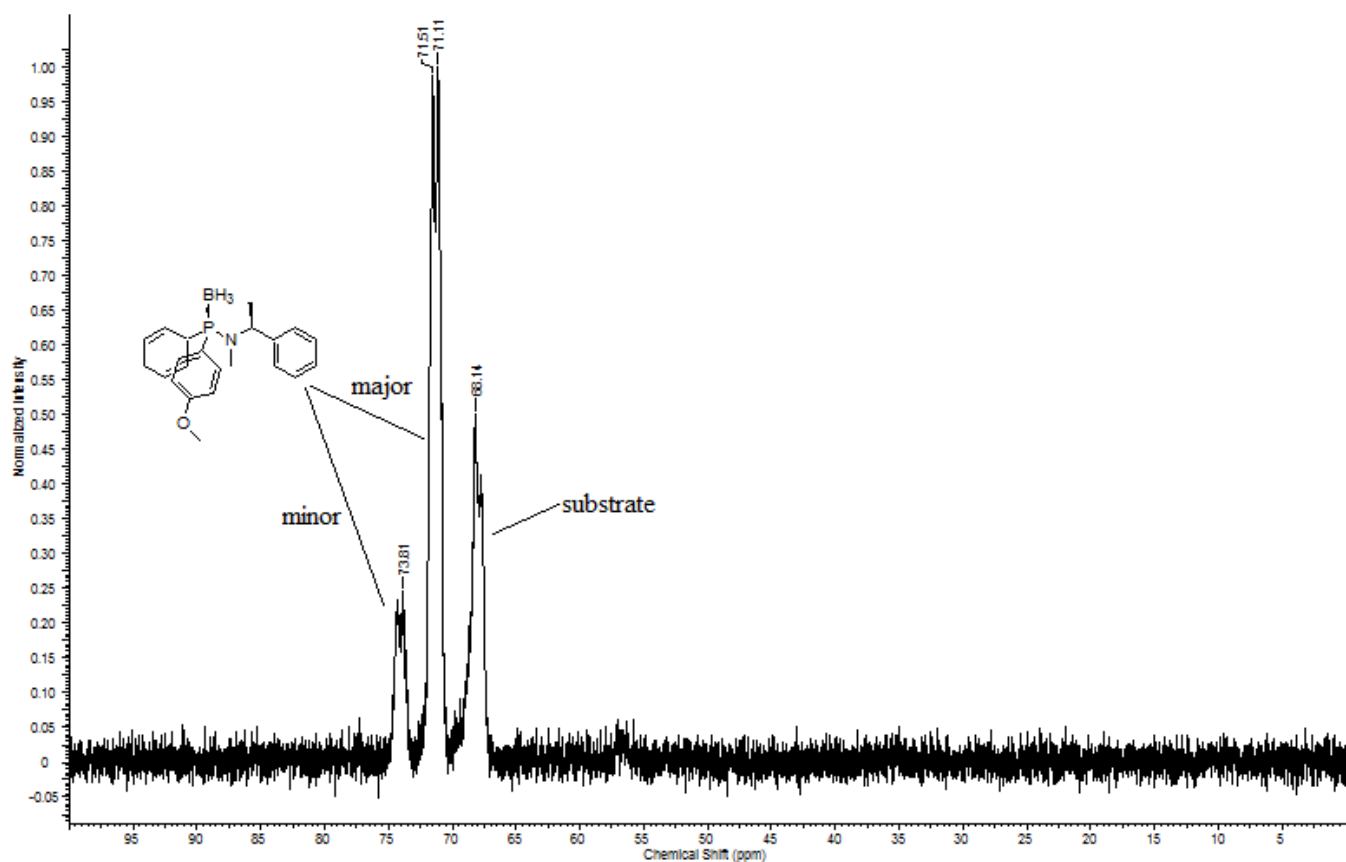
^{31}P NMR spectra of (1,4-cyclohexadien-3-yl)phenylphosphinous acid-borane *N*-methylbornylamide (**26d**) along with phenylphosphinous acid-borane *N*-methylbornylamide (**29**) (202 MHz, CDCl_3)



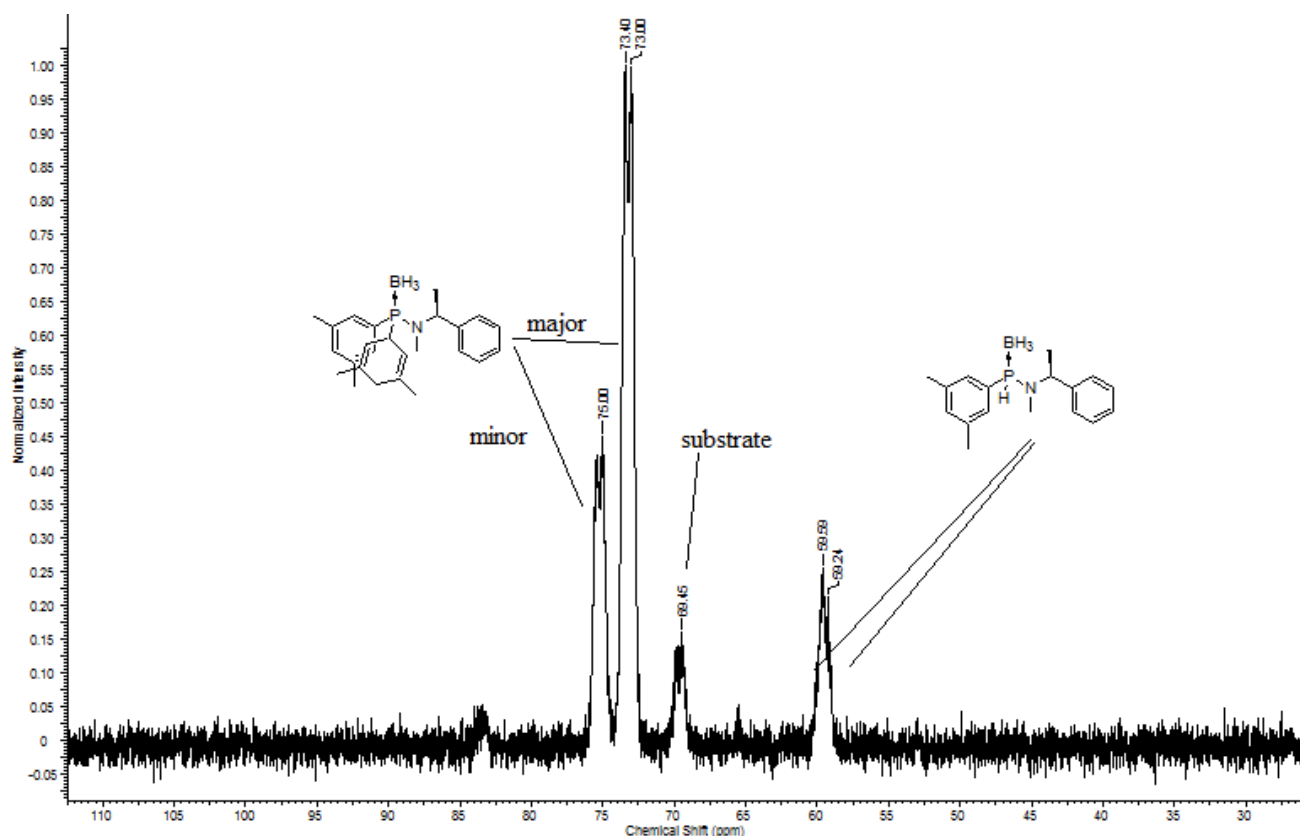
^{31}P NMR spectra of (4-methyl-1,4-cyclohexadien-3-yl)(*o*-tolyl)phosphinous acid-borane *N*-methyl(*S*)- α -methylbenzylamide (**34a**) along with substrate (**32a**) (202 MHz, CDCl_3)



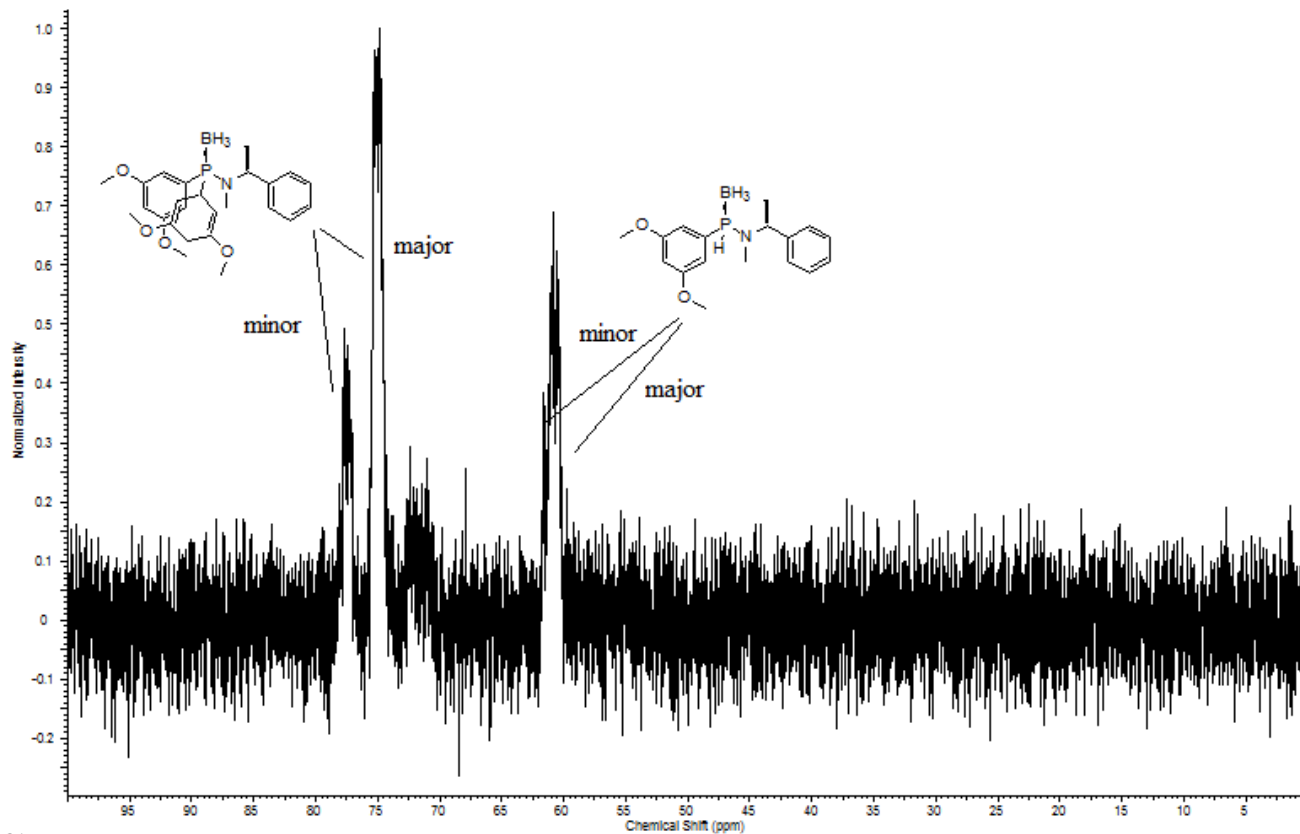
³¹P NMR spectra of (6-methyl-1,4-cyclohexadien-3-yl)(*p*-tolyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**34b**) (202 MHz, CDCl₃)



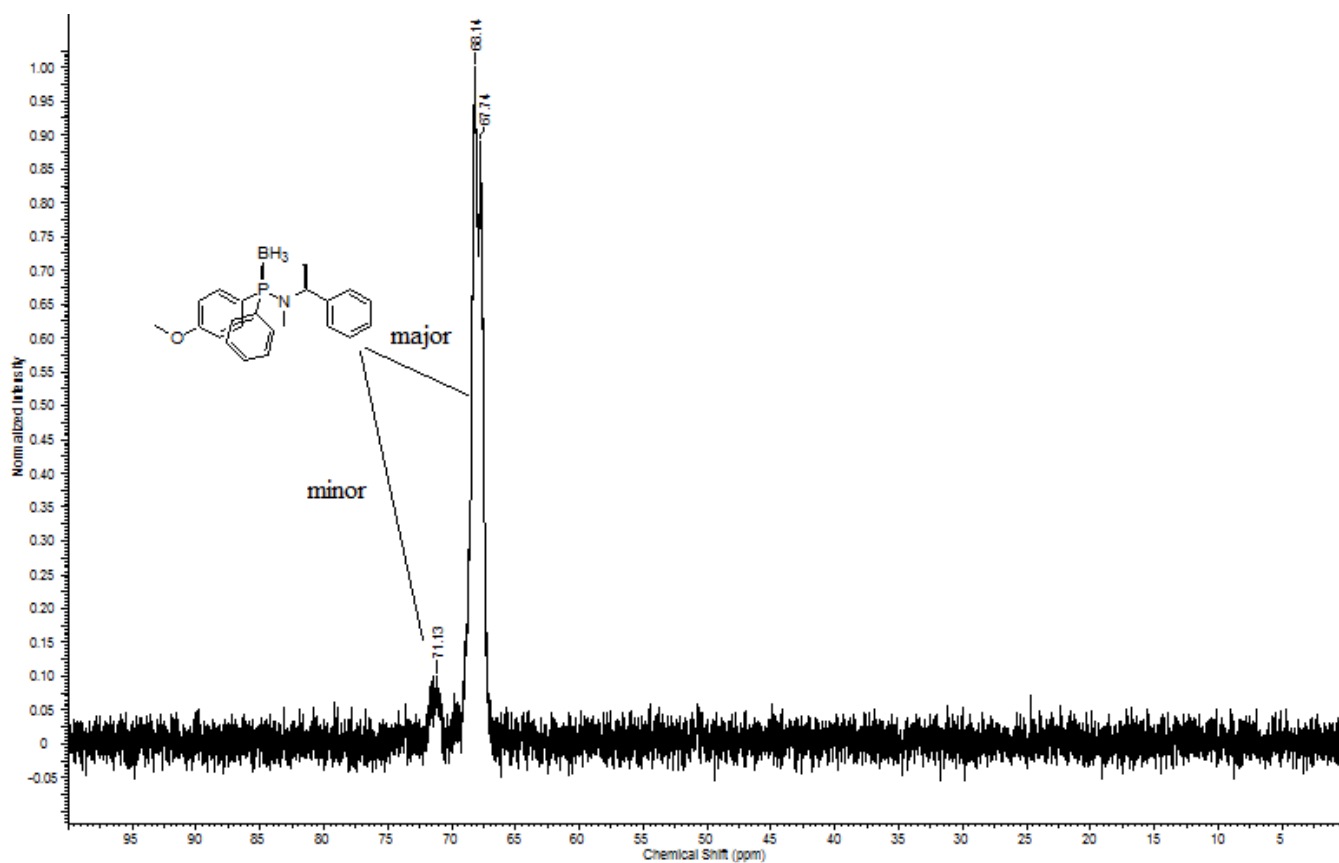
³¹P NMR spectra of (1,4-cyclohexadien-3-yl)(*p*-anisyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**34c**) along with substrate **32c** (202 MHz, CDCl₃)



^{31}P NMR spectra of (1,5-dimethyl-1,4-cyclohexadien-3-yl)(*m*-xylyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**34d**) along with *m*-xylylphosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**35a**) and substrate **32c** (202 MHz, CDCl_3)



^{31}P NMR spectra of (1,5-dimethoxy-1,4-cyclohexadien-3-yl)(3,5-dimethoxyphenyl)phosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**34e**) along with 3,5-dimethoxyphenylphosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**35b**) (202 MHz, CDCl_3)



^{31}P NMR spectra of (*p*-anisyl)phenylphosphinous acid-borane *N*-methyl-(*S*)- α -methylbenzylamide (**36**) (202 MHz, CDCl_3)