

Supporting Information for article:

## **Myrtenal and Myrtanal as Auxiliaries in the Synthesis of Some C,P-Stereogenic Hydroxyphosphine Oxides and Hydroxyphosphine-Boranes Possessing up to Four Contiguous Centers of Chirality**

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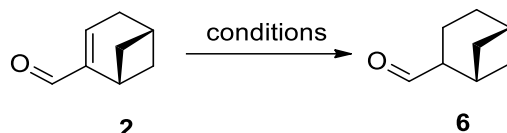
1D NMR spectra of ( <i>R<sub>P</sub></i> )-6,6-dimethylbicyclo[3.1.1]heptan-2-yl)(hydroxy)methyl)(t-butyl)(phenyl)phosphine-borane ( <i>R<sub>P</sub></i> )- <b>10a</b>	S69-S71
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## 1. Experimental

### 1.1 Hydrogenation of (1R)-myrtenal (**2**)

**Table S1.** Optimisation of the hydrogenation of (1R)-myrtenal (**2**).



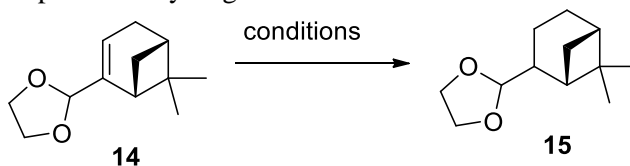
No	Conditions	Product	Yield (%)
1	<b>2</b> (1 equiv.), Pd/C (10% wt.), H <sub>2</sub> (1 atm.), MeOH, rt, 96 h	mixture	-
2	<b>2</b> (1 equiv.), Pd/C (10% wt.), H <sub>2</sub> (1 atm.), MeOH, 60 °C, 6 d	complicated mixture	-
3	<b>2</b> (1 equiv.), Pt/C (10% wt.), H <sub>2</sub> (1 atm.), MeOH, rt -24 h, then 60 °C- 8 d	<b>6</b>	69
4	<b>2</b> (1 equiv.), Pt/C (10% wt.), H <sub>2</sub> (20 atm.), AcOEt, rt, 3 d	no reaction	-
5	<b>2</b> (1 equiv.), Pt/C (10% wt.), H <sub>2</sub> (1 atm.), AcOEt, 70 °C, 3 d	<b>6</b>	63

### 1.2 Procedure of the synthesis of 2-(6,6-dimethylbicyclo[3.1.1]heptan-3-yl)-1,3-dioxolane (**14**) [1]

In the three-necked flask (100 mL) equipped with the Dean–Stark trap and balloon with argon, myrtenal (**2**) (1 mL, 0.987 g, 6.6 mmol), ethylene glycol (1.13 mL, 19.7 mmol) and tartaric acid (10% wt, 0.098 g) were placed in anhydrous benzene (7 mL). The mixture was refluxed for 2 h until has been observed in the trap. The crude product was distilled in K ugelrohr giving 67% of **14** (0.855 g) [2]. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 0.84 (s, 3H), 1.19 (d, J = 9 Hz, 1H), 1.13 (s, 3H), 2.10-2.12 (m, 1H), 2.24-2.46 (m, 4H), 3.87-4.00 (m, 2H), 5.14 (s, 1H), 5.75 (s, 1H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ 21.1, 26.0, 31.2, 31.6, 37.8, 40.6, 40.8, 65.0, 65.1, 104.6, 124.8, 144.7. HRMS (ESI-LTQ) m/z calcd for C<sub>12</sub>H<sub>19</sub>O<sub>2</sub>: 195.13851; found: 195.13863.

### 1.3 Hydrogenation of acetal **14** derived from (1R)-myrtenal (**2**)

**Table S2.** The attempts of the hydrogenation of acetal **14** derived from (1R)-myrtenal (**2**).

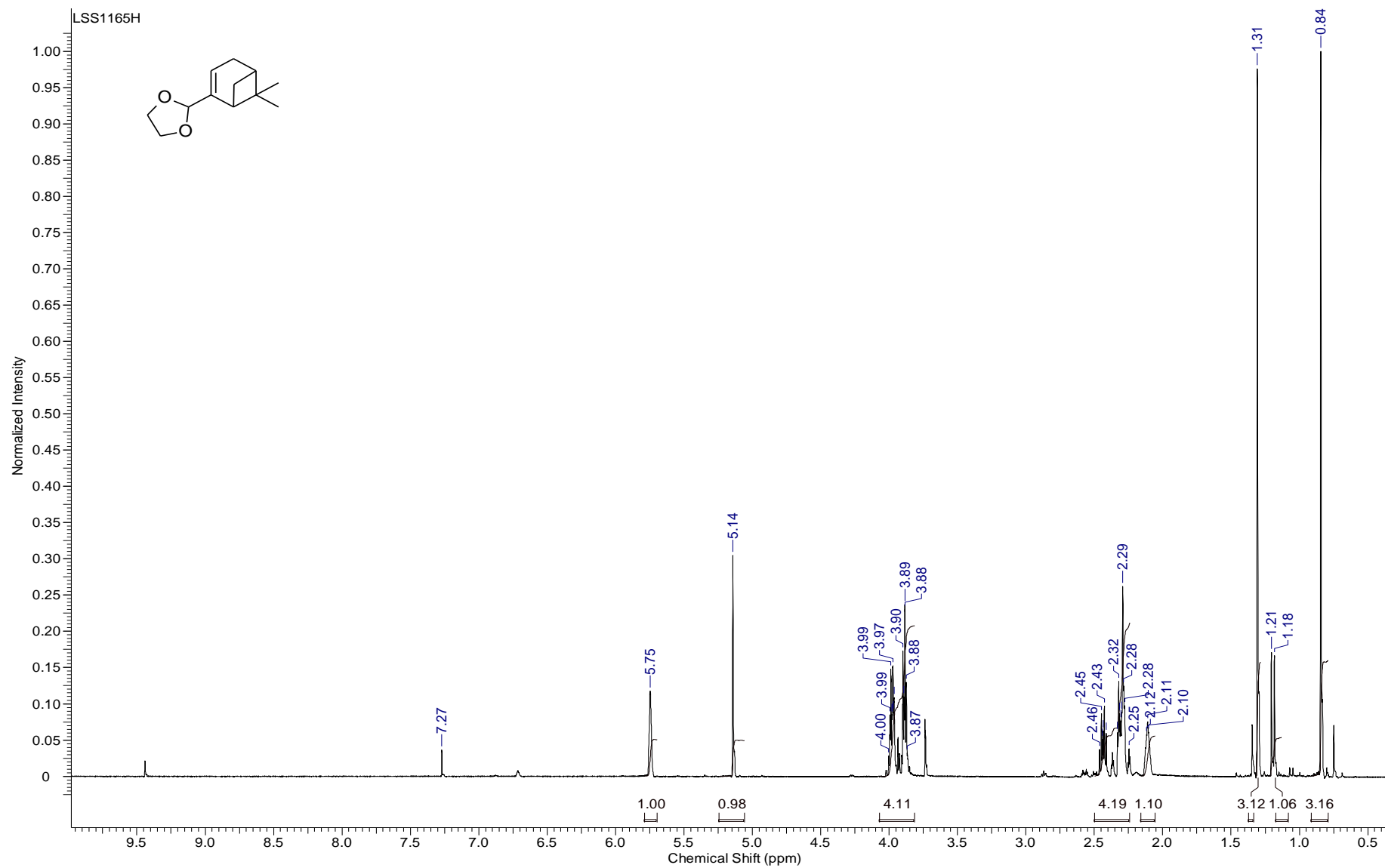


No	Conditions	Product <sup>a</sup>	Yield (%)
1	<b>14</b> (1 equiv.), Pd/C (10% wt.), H <sub>2</sub> (1 atm.), MeOH, rt, 4d		-
2	<b>14</b> (1 equiv.), Pd/C (10% wt.), H <sub>2</sub> (1 atm.), AcOEt, rt, 6 d	complicated mixture	-
3 [3]	<b>14</b> (1 equiv.), Pt <sub>2</sub> O (0.1 equiv.), H <sub>2</sub> (1 atm.), AcOEt, rt, then reflux, 3 d	no reaction	-

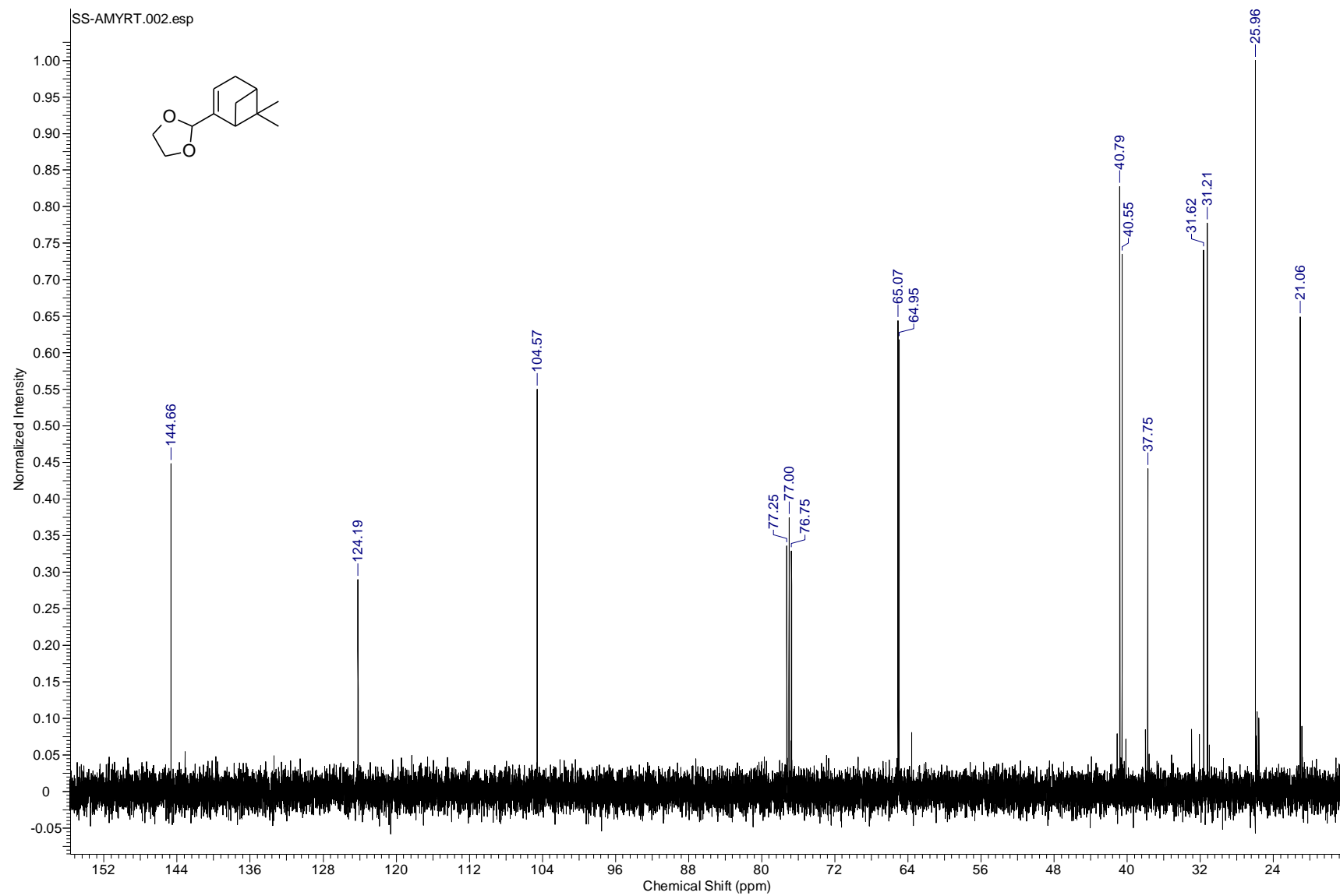
4	<b>14</b> (1 equiv.), Pd/C (10% wt.), H <sub>2</sub> (1 atm.), AcOEt, rt, 3 d, then 60 °C, 30 h	<b>14</b> <b>15</b>	30 12
5	<b>14</b> (1 equiv.), Grubbs I (5% mol), H <sub>2</sub> (1 atm.), NEt <sub>3</sub> (3 equiv.), DCM/MeOH, rt, 24 h	no reaction	-

<sup>a</sup> – according to GC-MS

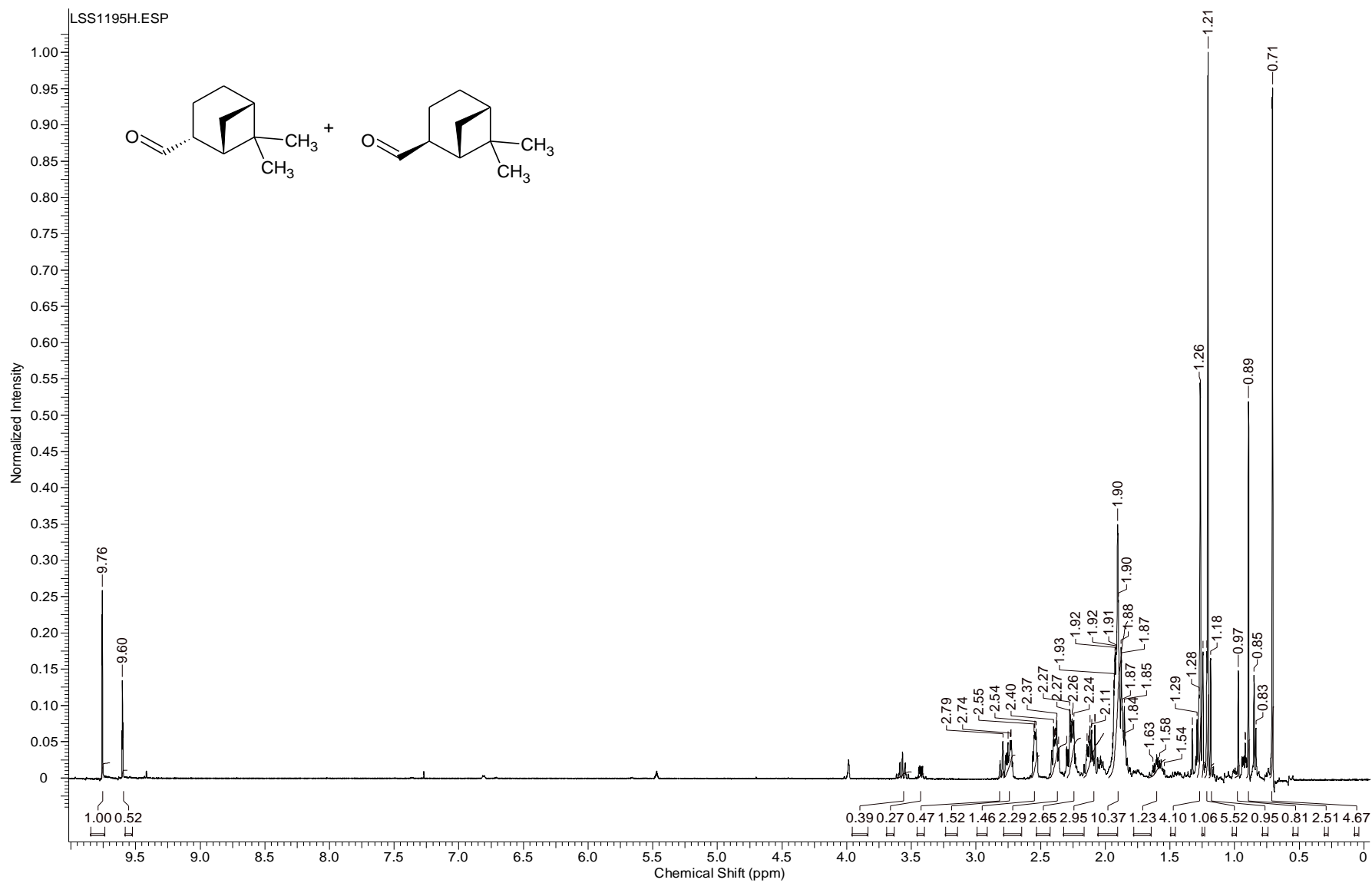
## 2. NMR spectra of compounds



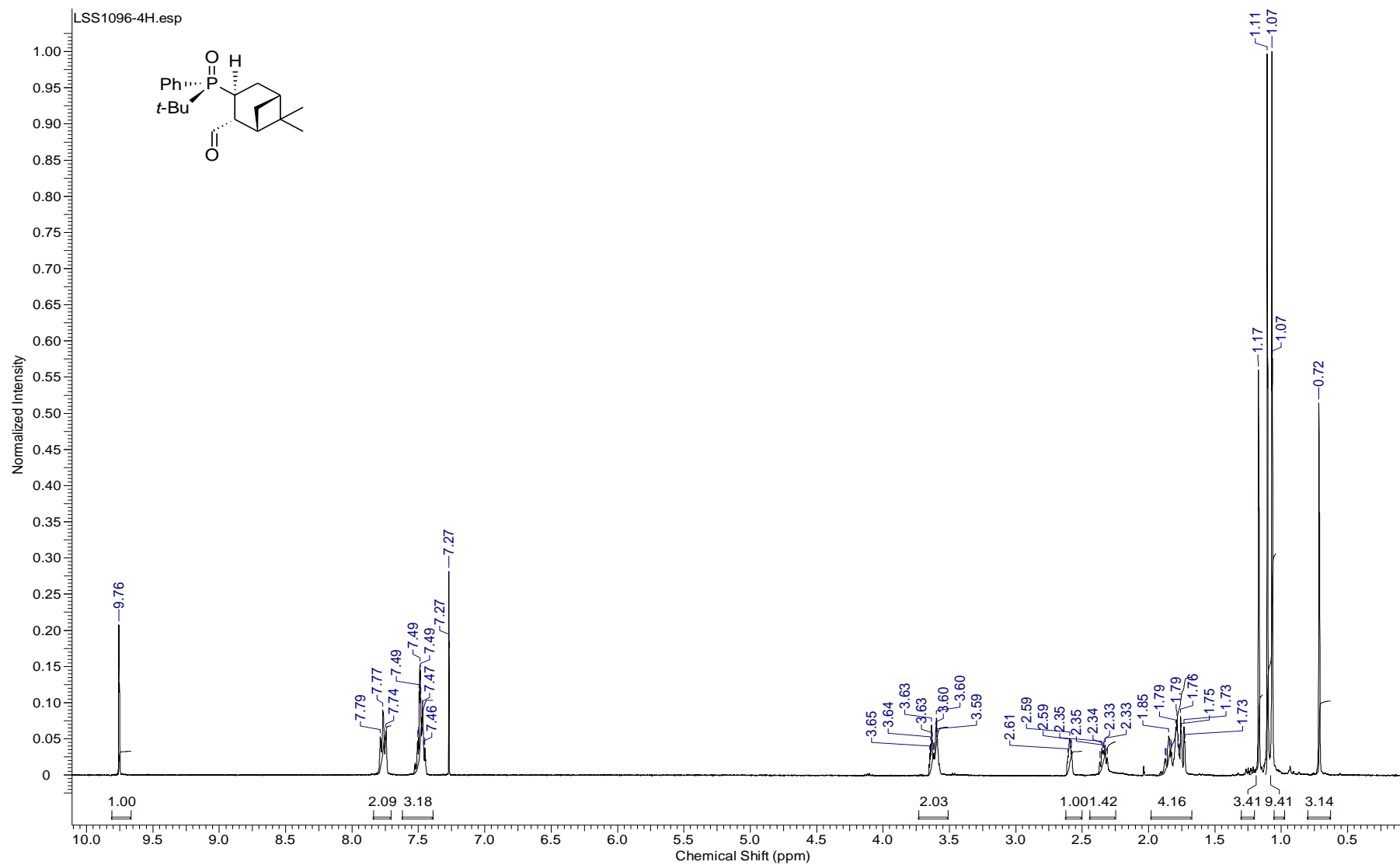
$^1\text{H}$  NMR spectrum of 2-(6,6-dimethylbicyclo[3.1.1]heptan-3-yl)-1,3-dioxolane (**14**) (400 MHz,  $\text{CDCl}_3$ )



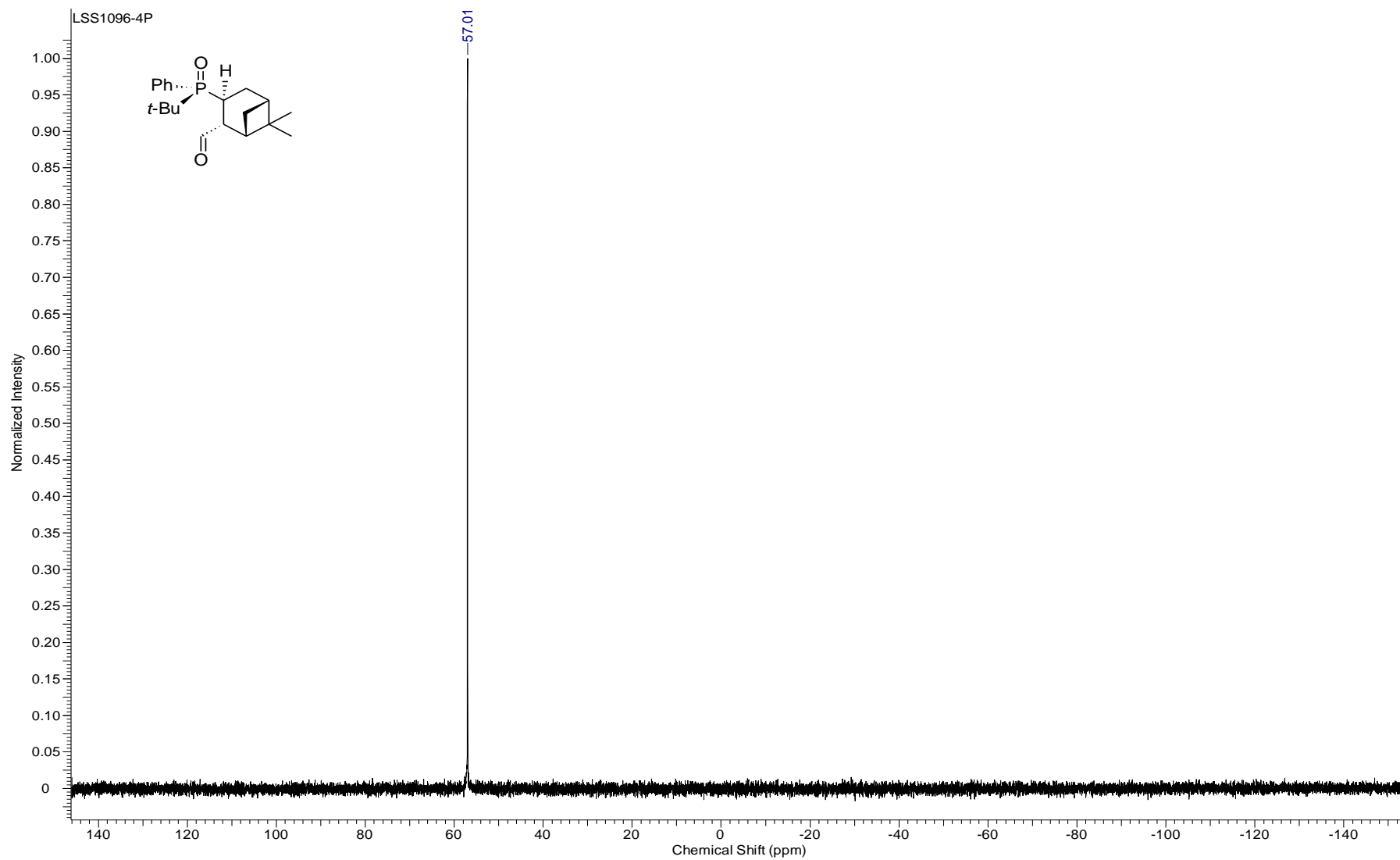
$^{13}\text{C}$  NMR spectrum of 2-(6,6-dimethylbicyclo[3.1.1]heptan-3-yl)-1,3-dioxolane (**14**) (125 MHz,  $\text{CDCl}_3$ )



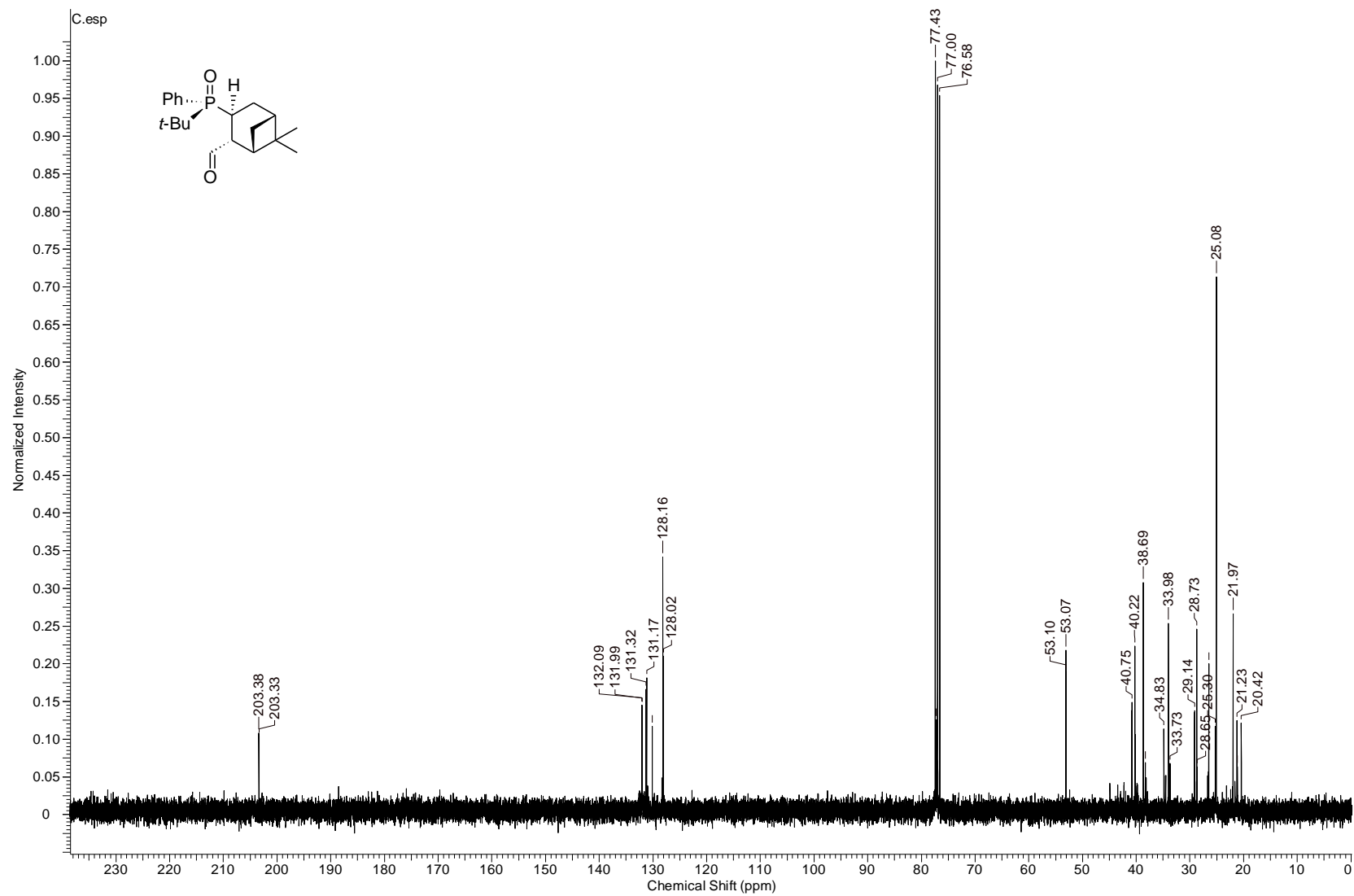
$^1\text{H}$  NMR spectrum of myrtanal (**6**) (400 MHz,  $\text{CDCl}_3$ )



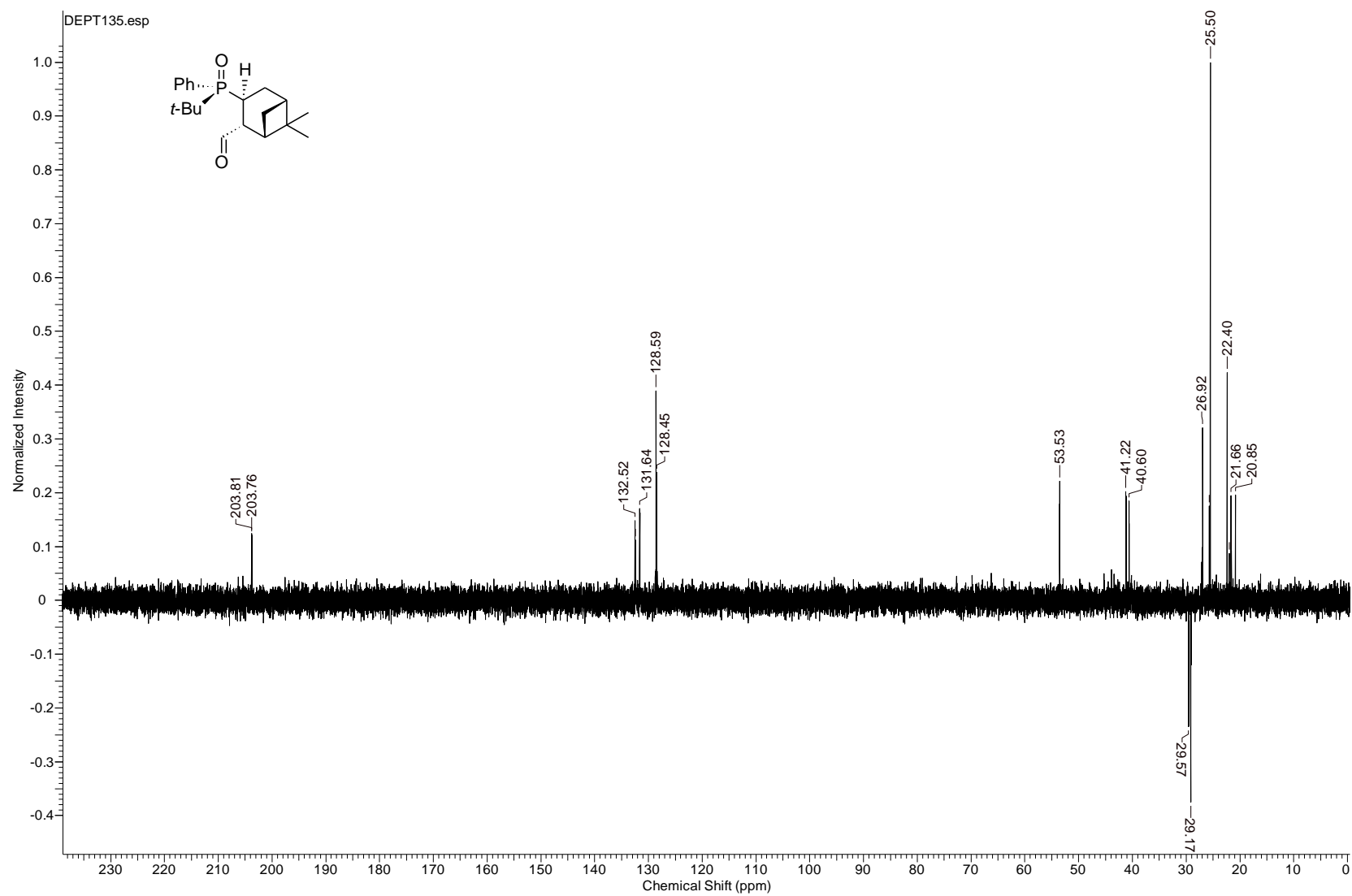
<sup>1</sup>H NMR spectrum of *trans*-(1*S*,2*R*,5*R*,*S<sub>P</sub>*)-3-(*t*-butylphenylphosphinoil)-6,6-dimethylbicyclo[3.1.1]heptane-2-carbaldehyde (*S<sub>P</sub>*)-(3a-I) (400 MHz, CDCl<sub>3</sub>) (see Figure 8)



$^{31}\text{P}$  NMR spectrum of *trans*-(1*S*,2*R*,5*R*,*S<sub>P</sub>*)-3-(*t*-butylphenylphosphinoil)-6,6-dimethylbicyclo[3.1.1]heptane-2-carboaldehyde (*S<sub>P</sub>*)-(3a-I) (400 MHz,  $\text{CDCl}_3$ )

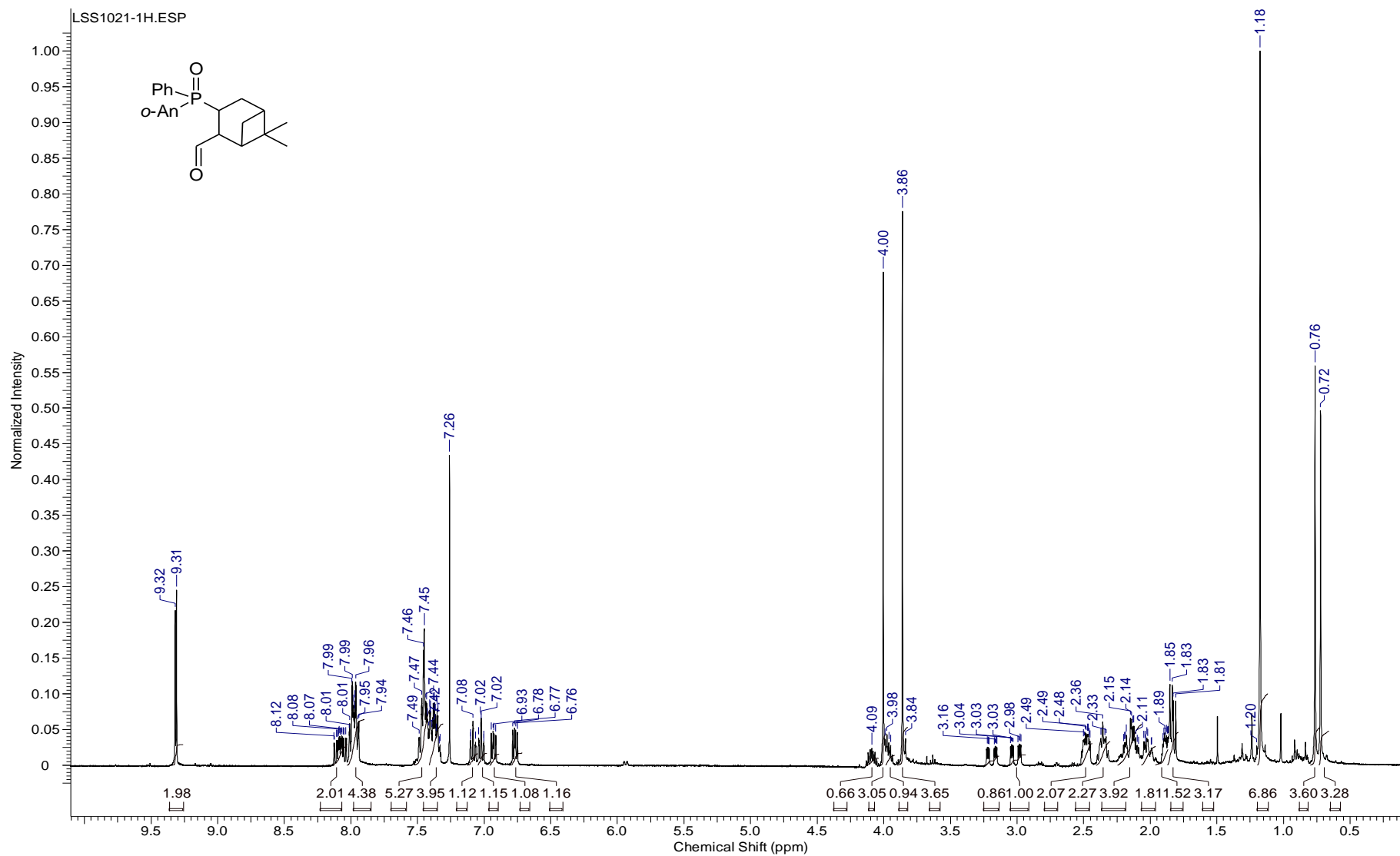


<sup>13</sup>C NMR spectrum of *trans*-(1*S*,2*R*,5*R*,*S<sub>P</sub>*)-3-(*t*-butylphenylphosphinoil)-6,6-dimethylbicyclo[3.1.1]heptane-2-carbaldehyde (*S<sub>P</sub>*)-(3a-I) (75 MHz, CDCl<sub>3</sub>)

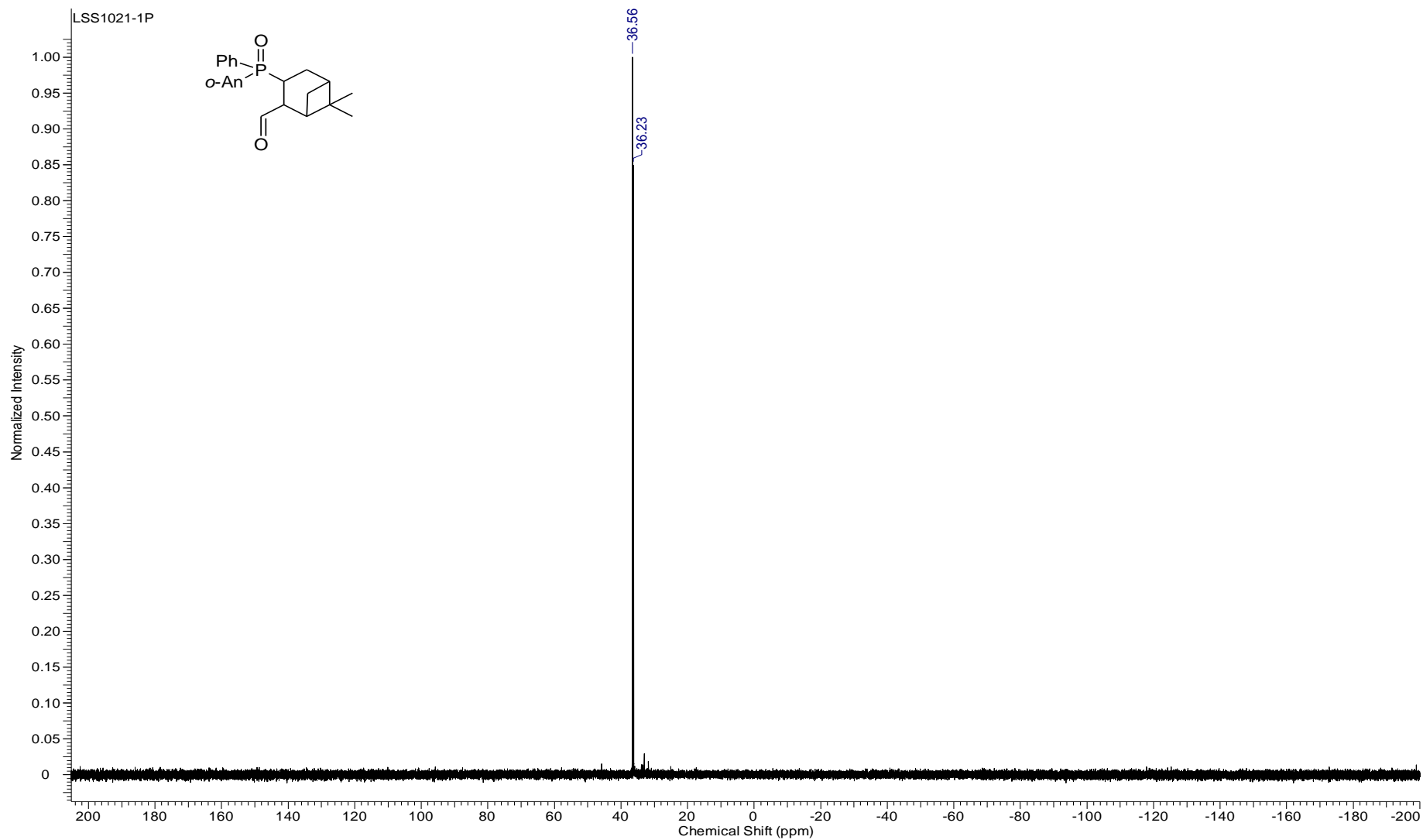


DEPT 135 NMR spectrum of *trans*-(1*S*,2*R*,5*R*,*S<sub>P</sub>*)-3-(*t*-butylphenylphosphinoil)-6,6-dimethylbicyclo[3.1.1]heptane-2-carbaldehyde (*S<sub>P</sub>*)-(3a-I) (75 MHz, CDCl<sub>3</sub>)



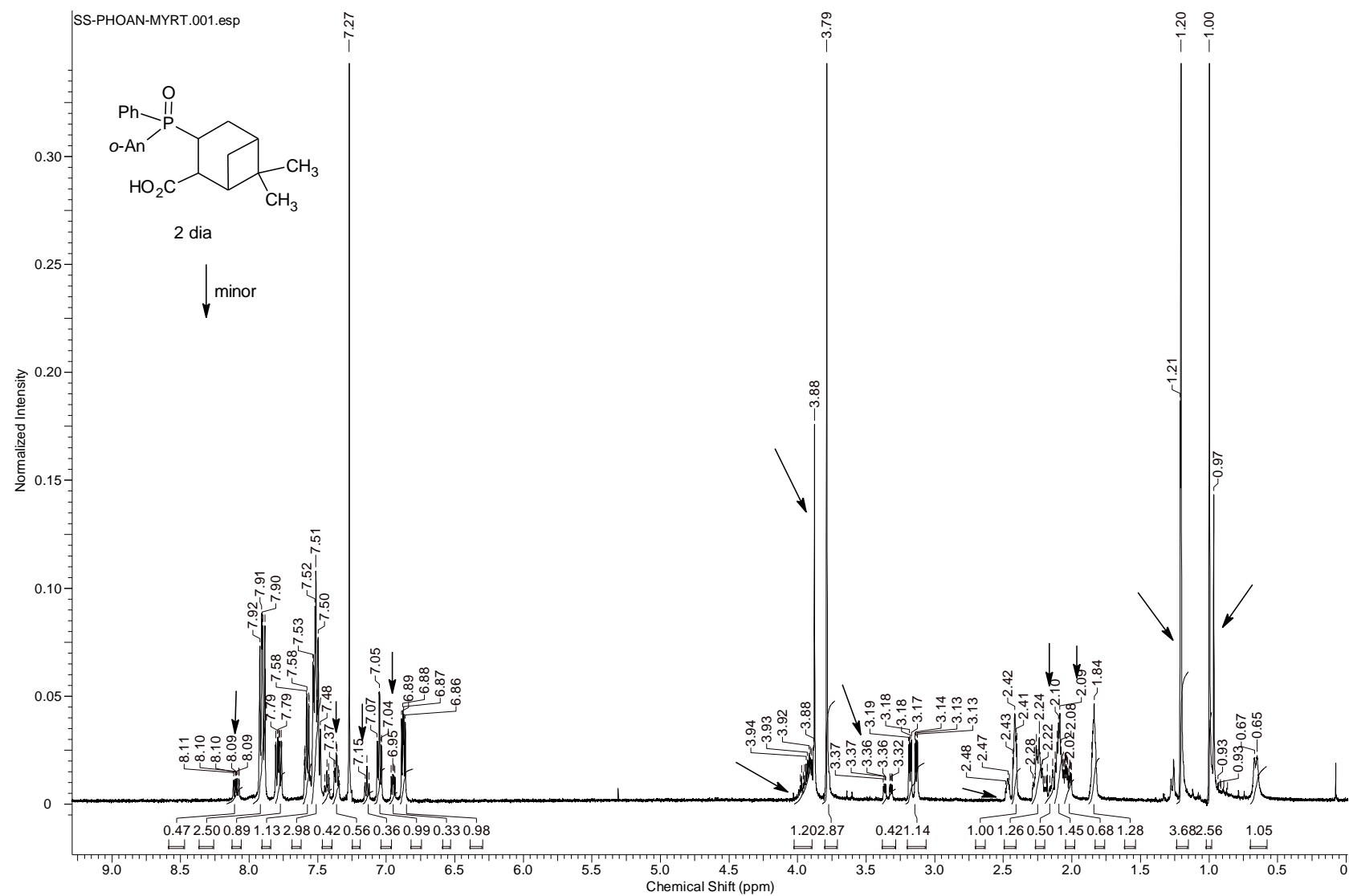


$^1\text{H}$  NMR spectrum of *(1S,5R)*-3-((2-anisyl)(phenyl)phosphoryl)-6,6-dimethylbicyclo[3.1.1]heptane-2-carbaldehyde (**3b**) (400 MHz,  $\text{CDCl}_3$ )

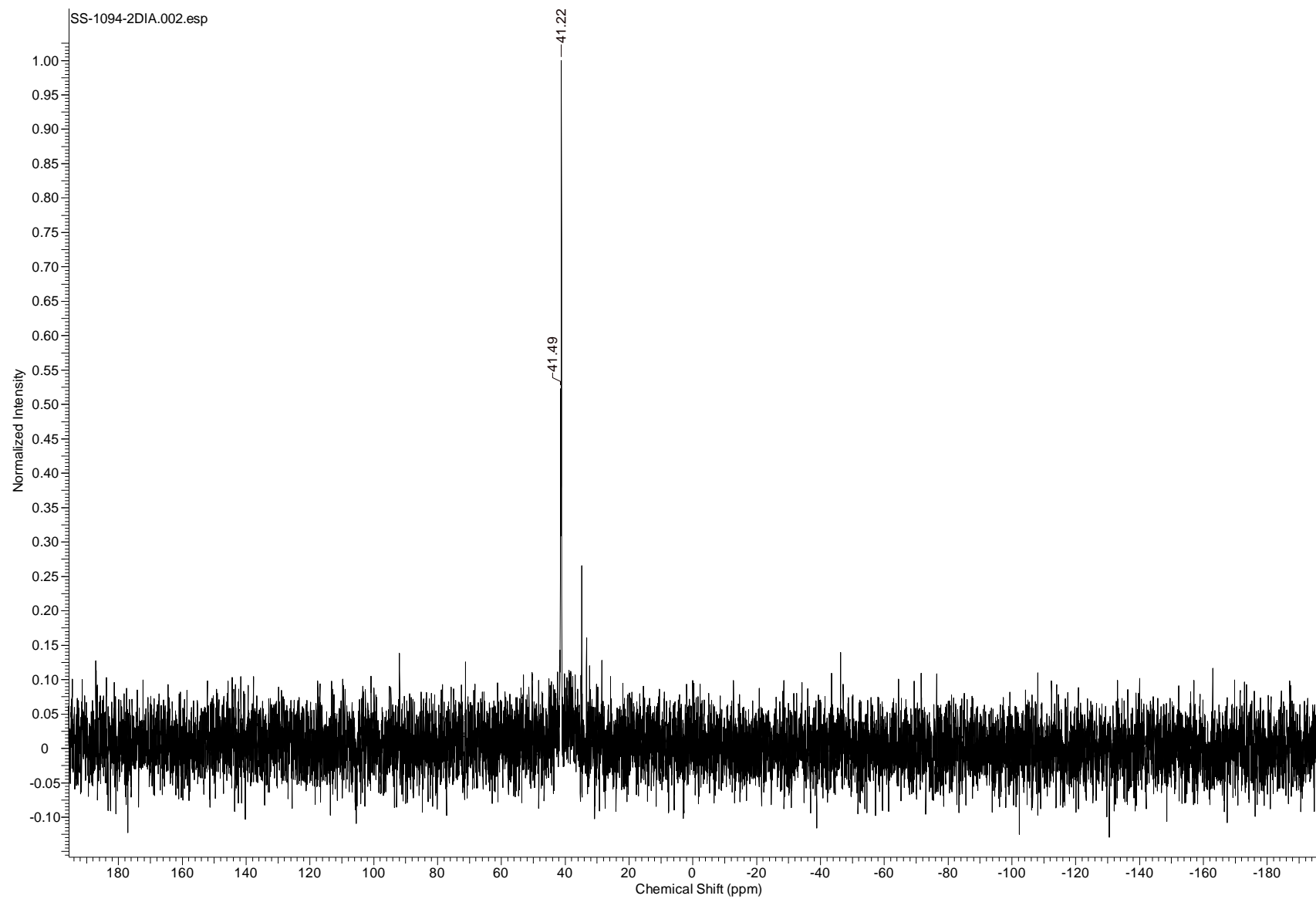


$^{31}\text{P}$  NMR spectrum of (*1S,5R*)-3-((2-anisyl)(phenyl)phosphoryl)-6,6-dimethylbicyclo[3.1.1]heptane-2-carbaldehyde (**3b**) (400 MHz,  $\text{CDCl}_3$ )

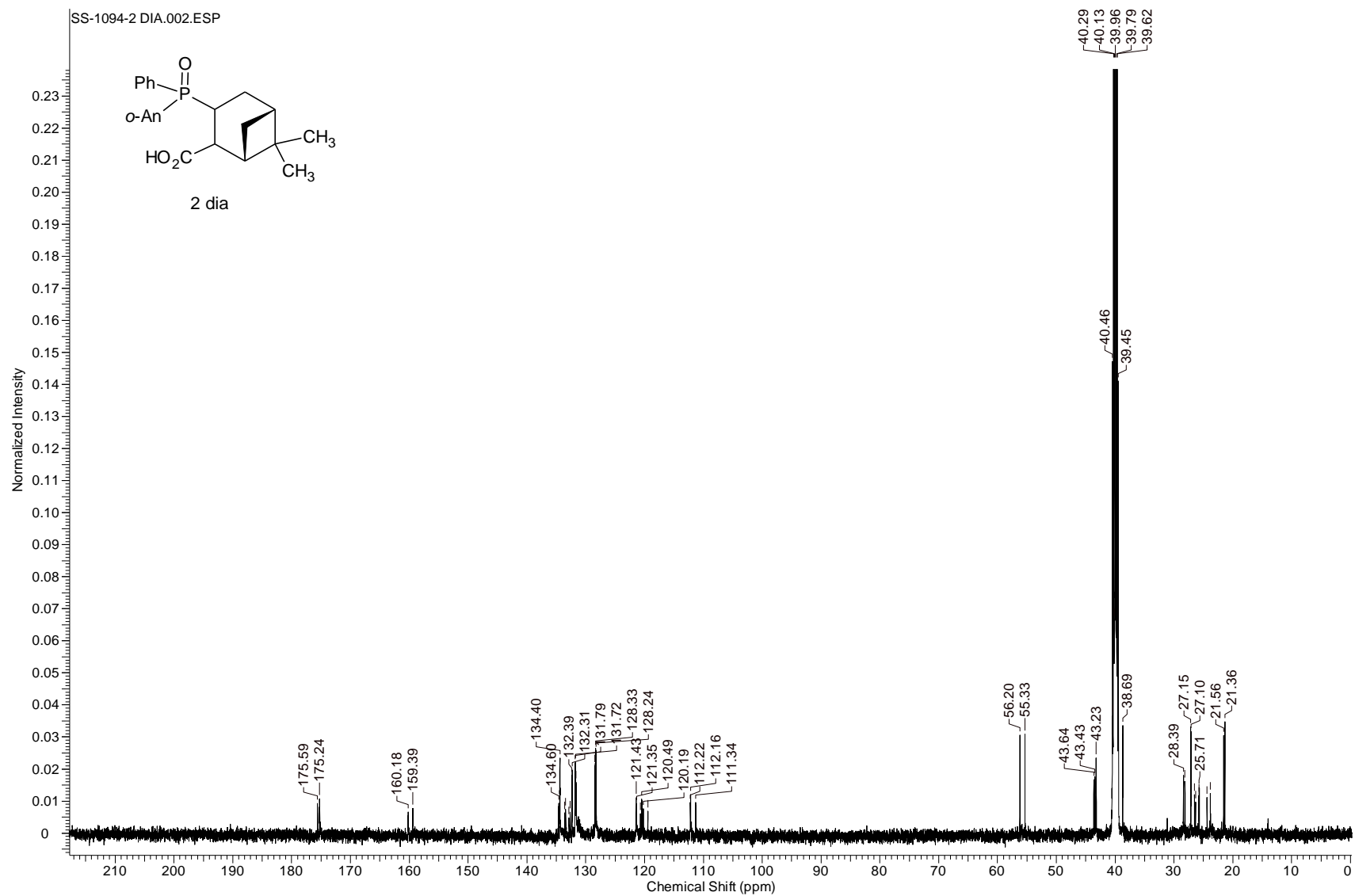




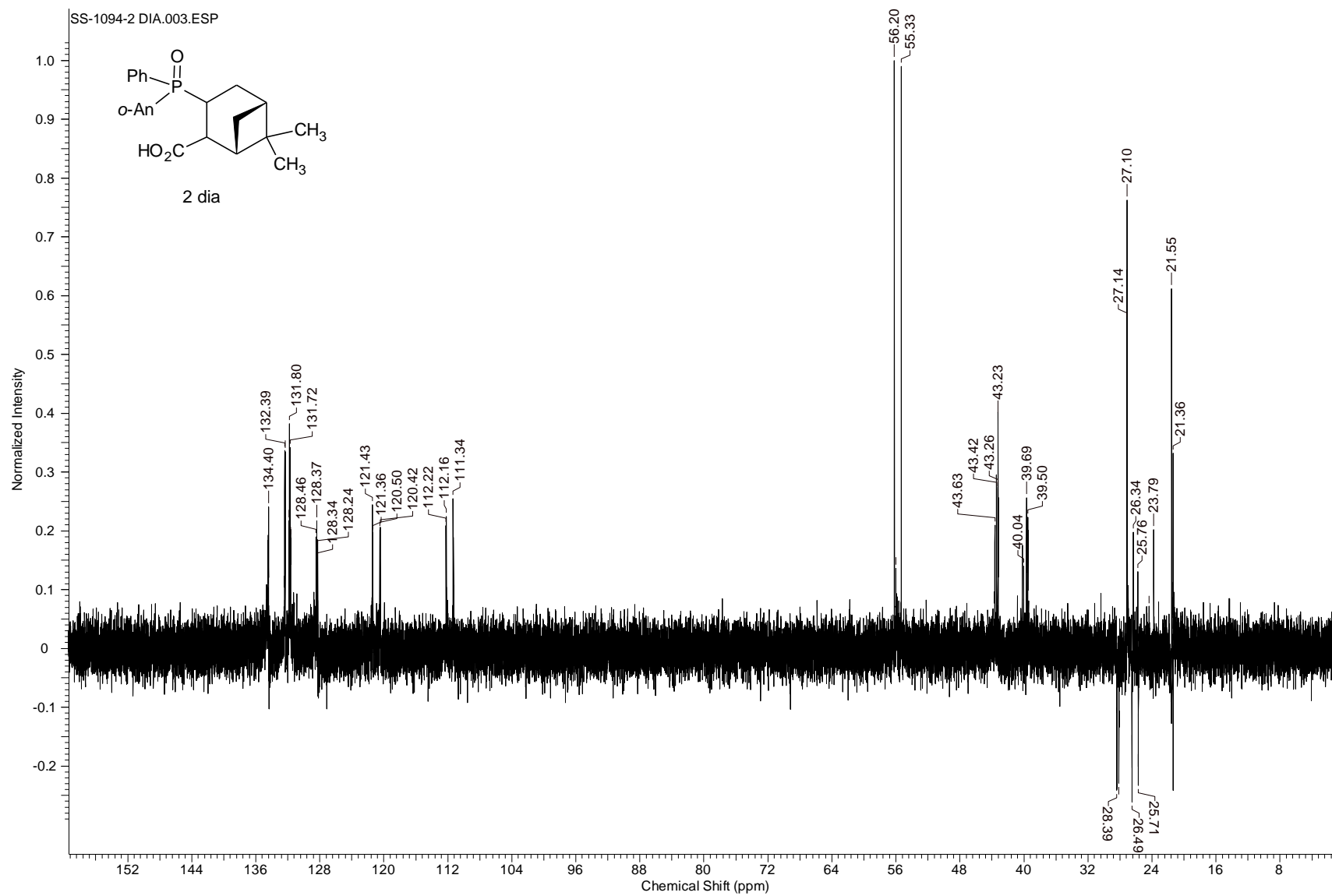
$^1\text{H}$  NMR spectrum of (1*S*,5*R*)-3-((2-methoxyphenyl)(phenyl)phosphoryl)-6,6-dimethylbicyclo[3.1.1]heptane-2-carboxylic acid (**5b**) (500 MHz,  $\text{CDCl}_3$ )



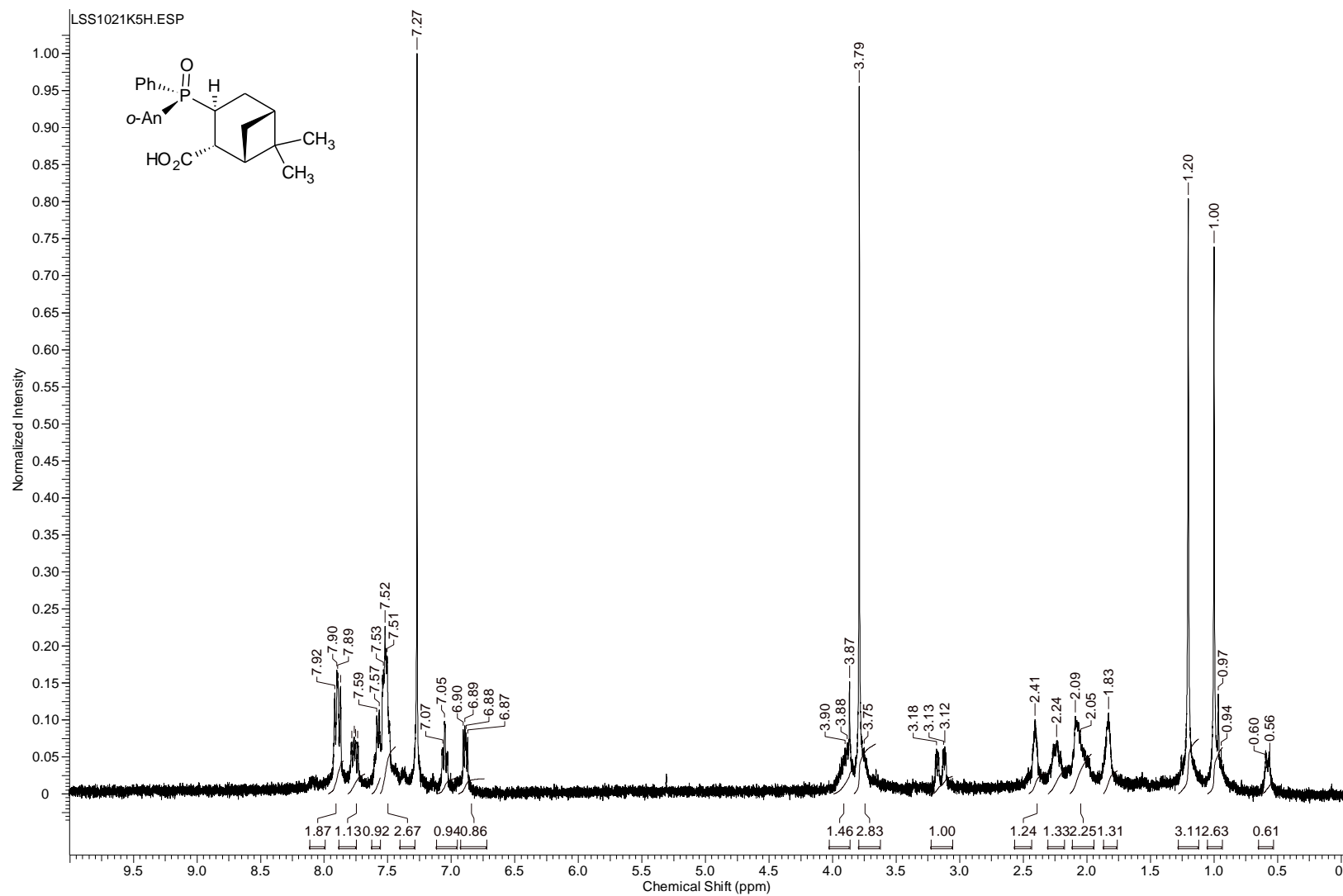
$^{31}\text{P}$  NMR spectrum of *(1S,5R)*-3-((2-anisyl)(phenyl)phosphoryl)-6,6-dimethylbicyclo[3.1.1]heptane-2-carboxylic acid (**5b**) (202 MHz,  $\text{CDCl}_3$ )



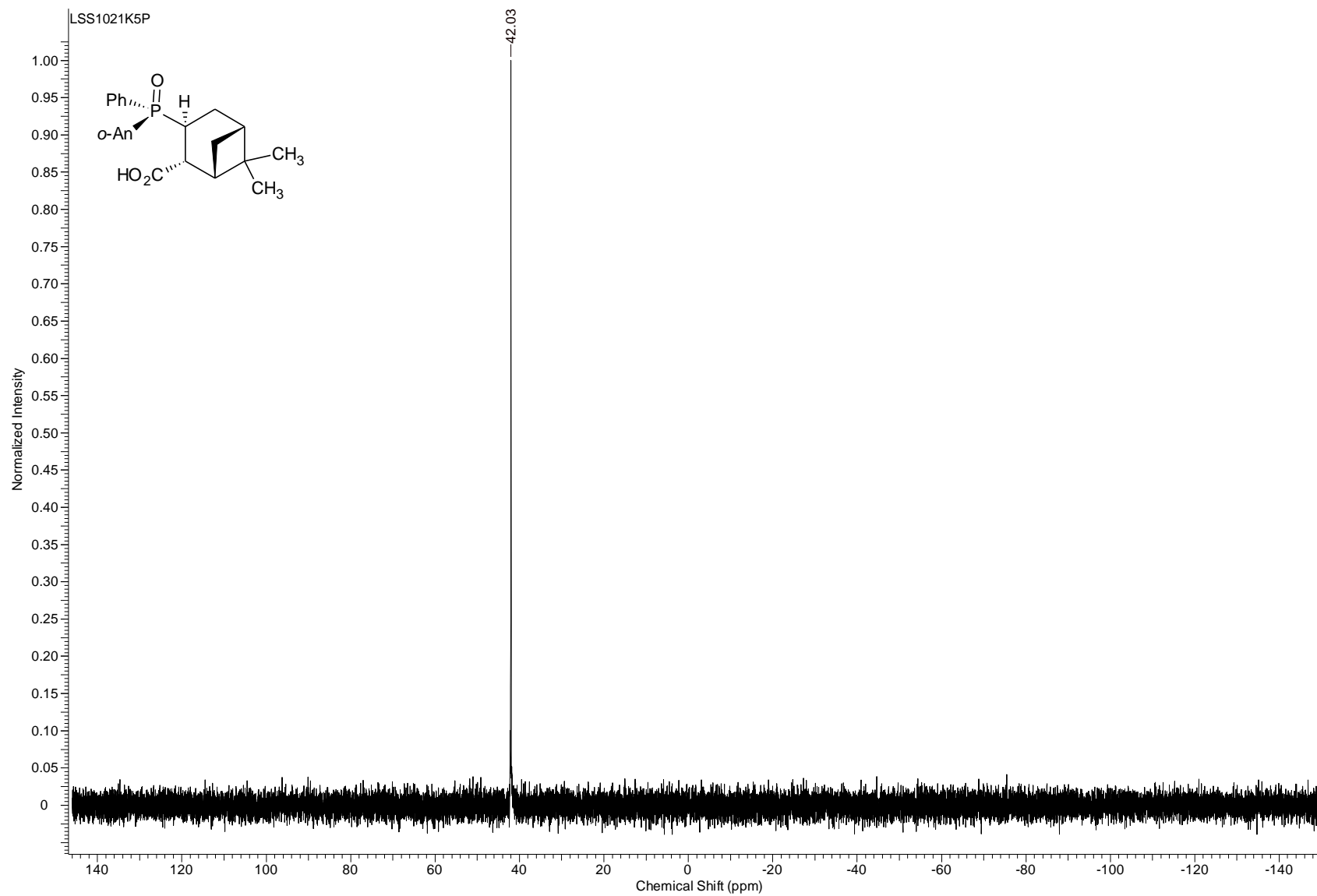
$^{13}\text{C}$  NMR spectrum of *(1S,5R)*-3-((2-anisyl)(phenyl)phosphoryl)-6,6-dimethylbicyclo[3.1.1]heptane-2-carboxylic acid (**5b**) (125 MHz, DMSO- $d_6$ )



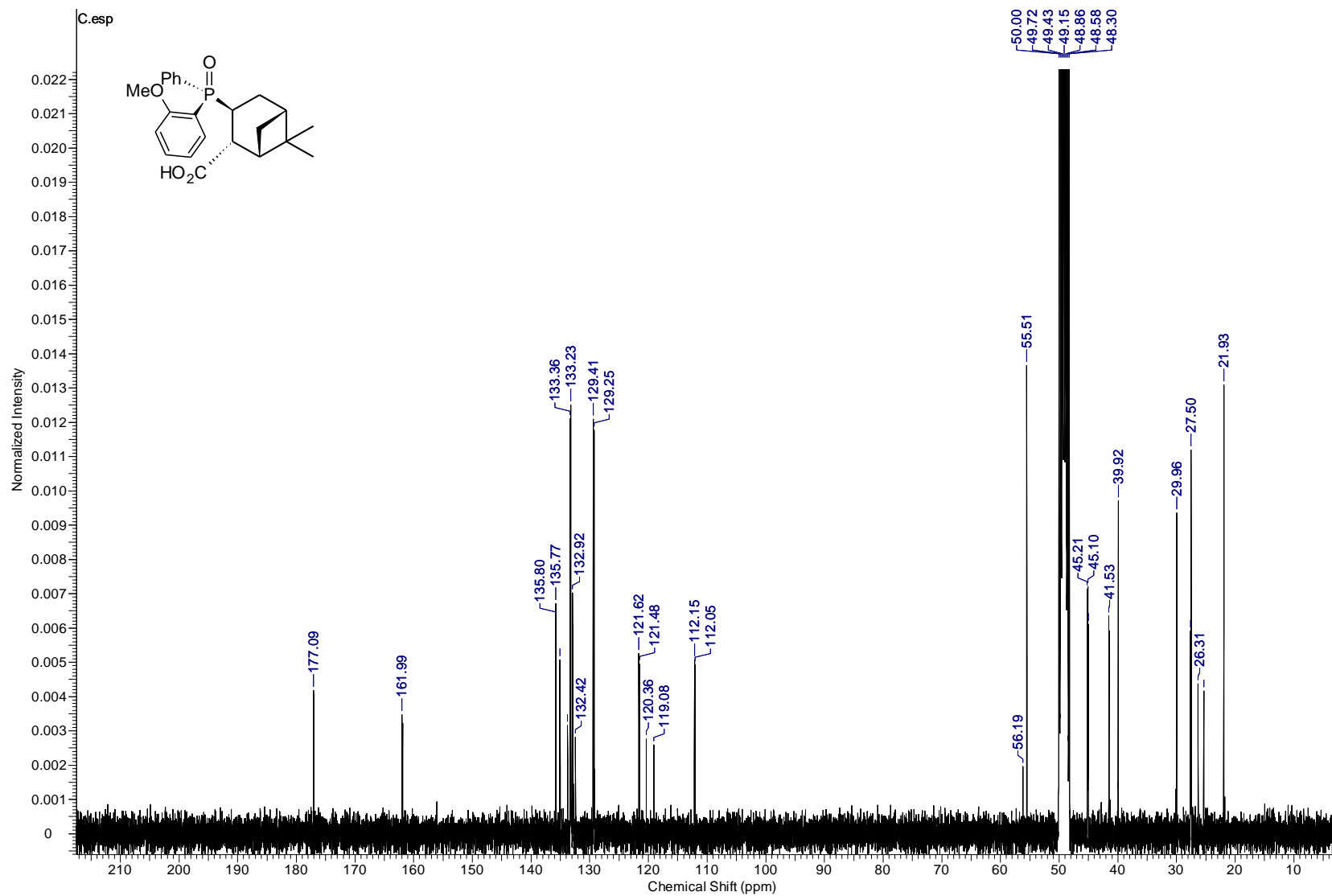
DEPT 135 NMR spectrum of (1S,5R)-3-((2-anisyl)(phenyl)phosphoryl)-6,6-dimethylbicyclo[3.1.1]heptane-2-carboxylic acid (**5b**) (125 MHz, DMSO-d<sub>6</sub>)



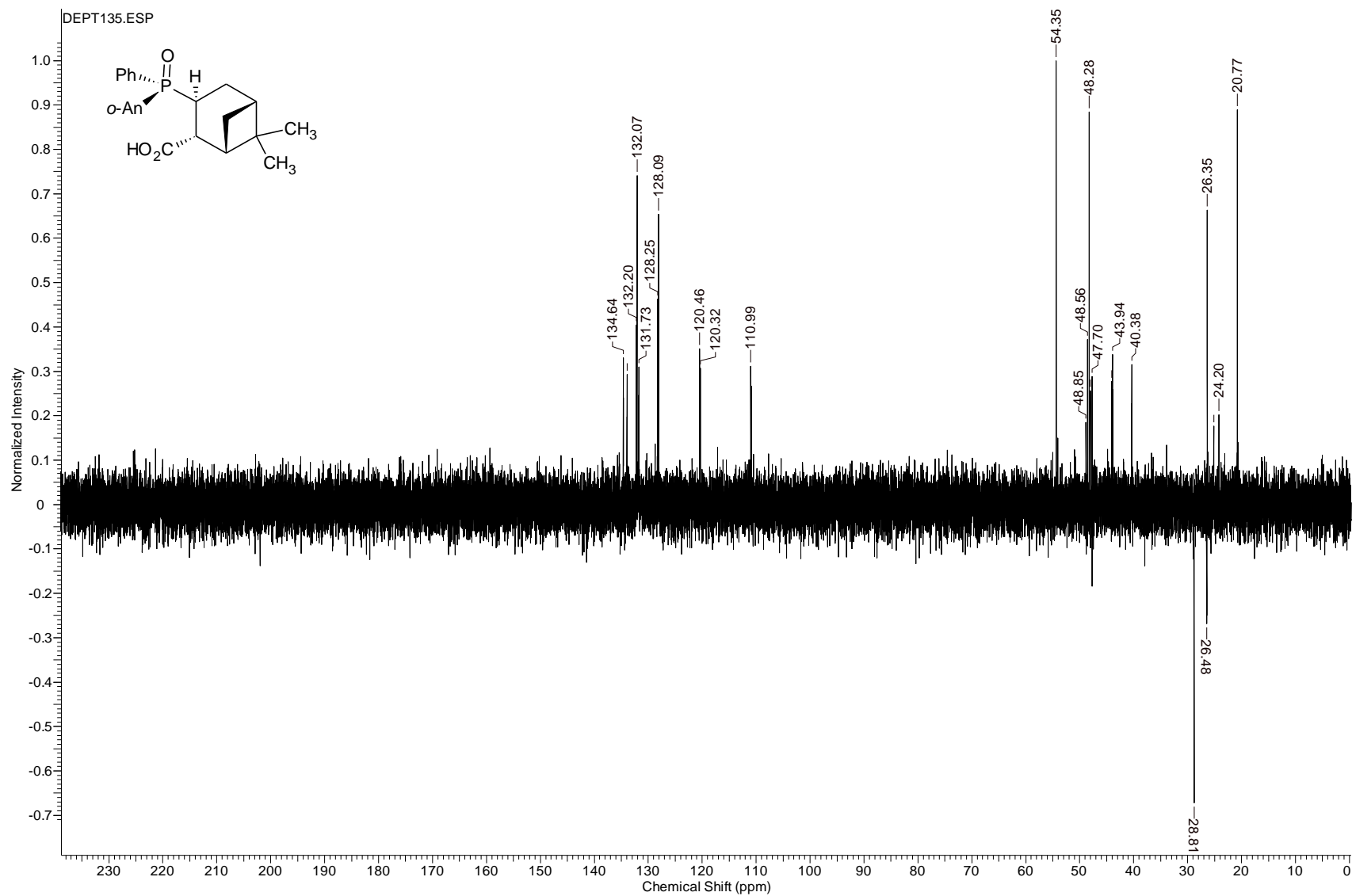
<sup>1</sup>H NMR spectrum of *(1S,2R,3S,5R, RP)*-3-((2-anisyl)(phenyl)phosphoryl)-6,6-dimethylbicyclo[3.1.1]heptane-2-carboxylic acid *(RP)*-**5b-I** (400 MHz, CDCl<sub>3</sub>) (low solubility)



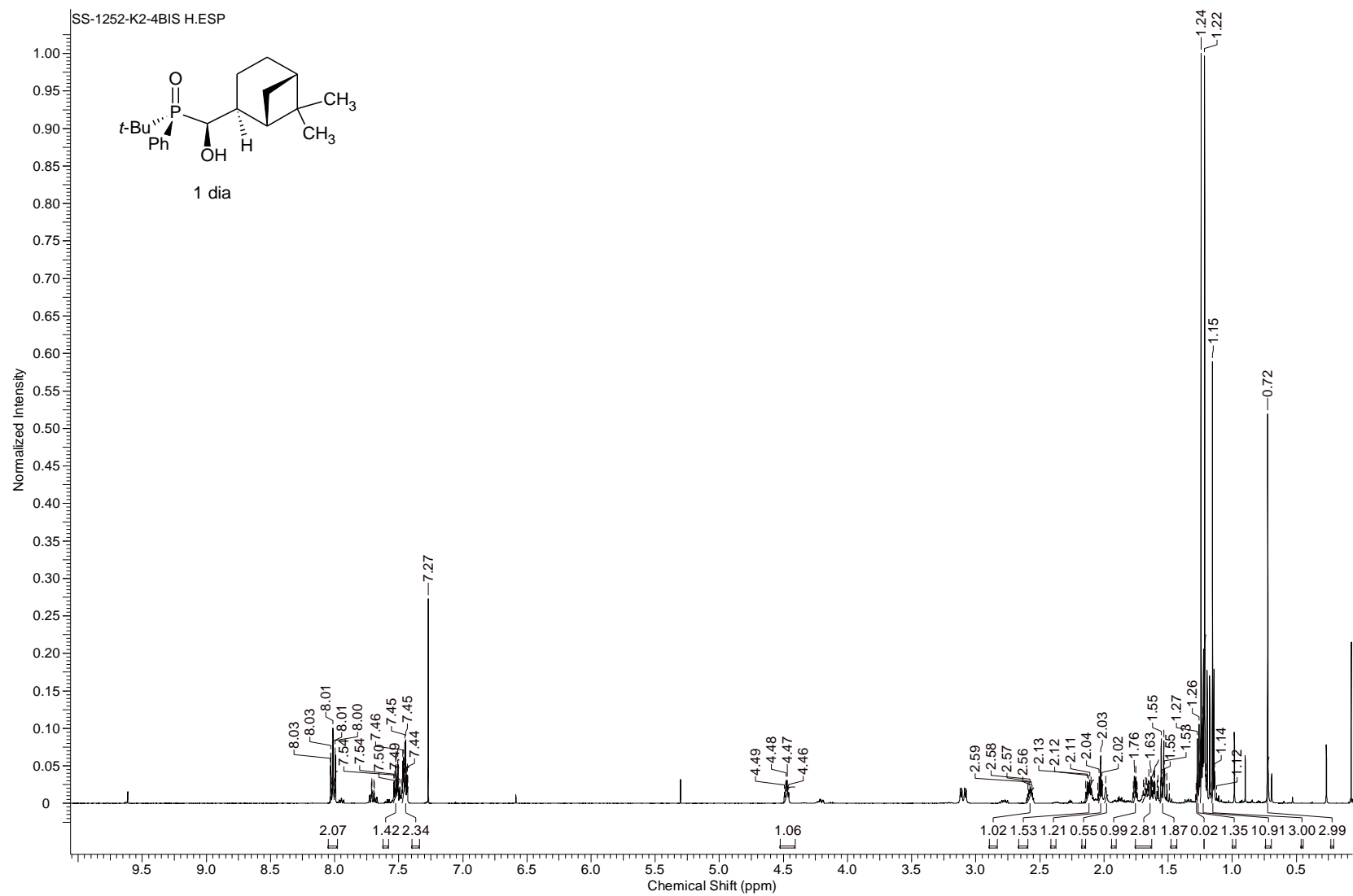
$^{31}\text{P}$  NMR spectrum of *(1S,2R,3S,5R, RP)*-3-((2-anisyl)(phenyl)phosphoryl)-6,6-dimethylbicyclo[3.1.1]heptane-2-carboxylic acid (*RP*)-**5b-I** (162 MHz,  $\text{CDCl}_3$ )



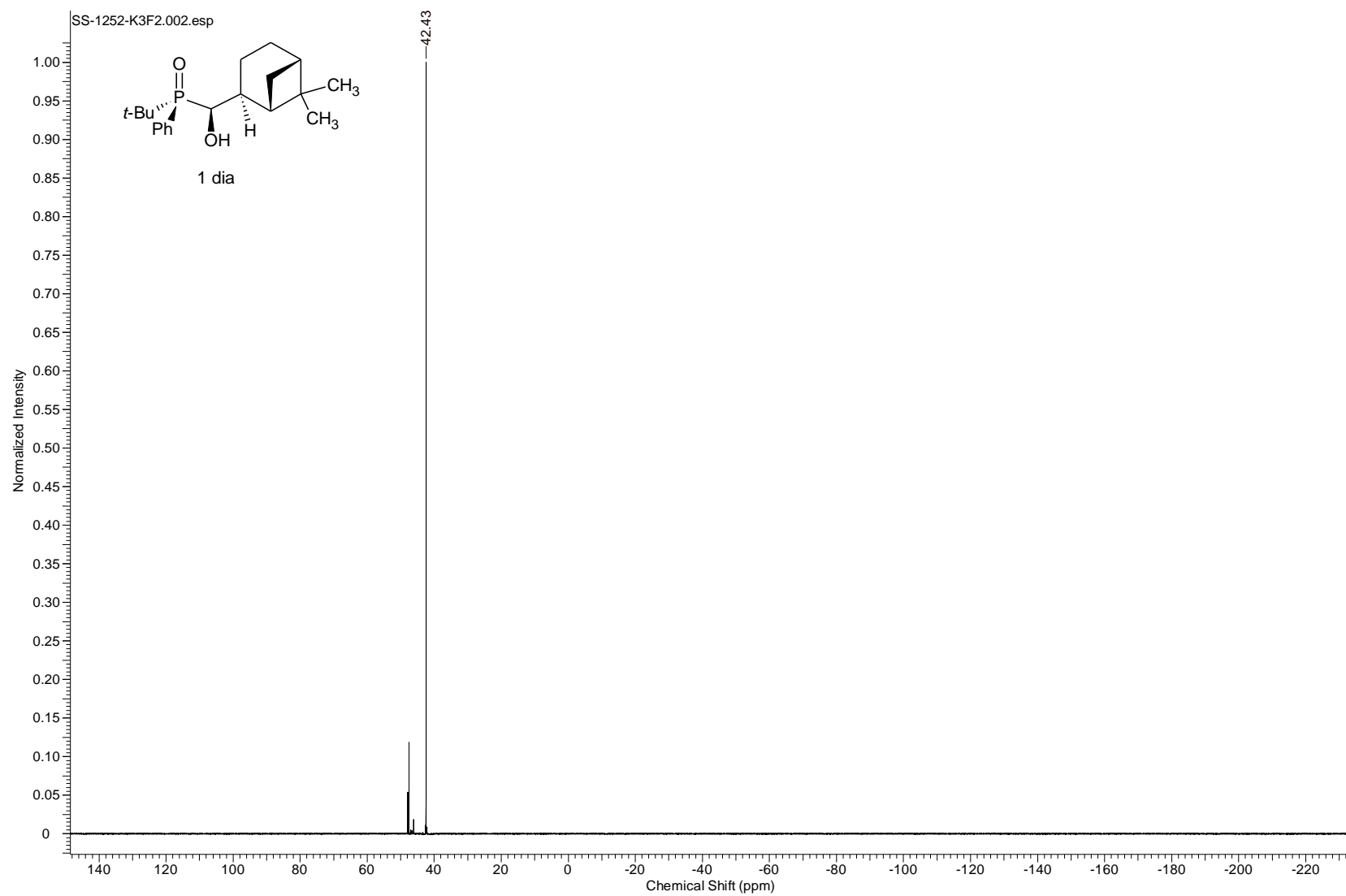
<sup>13</sup>C NMR spectrum of (1*S*,2*R*,3*S*,5*R*, *R<sub>P</sub>*)-3-((2-anisyl)(phenyl)phosphoryl)-6,6-dimethylbicyclo[3.1.1]heptane-2-carboxylic acid (*R<sub>P</sub>*)-**5b-I** (75 MHz, CD<sub>3</sub>OD) (low solubility)



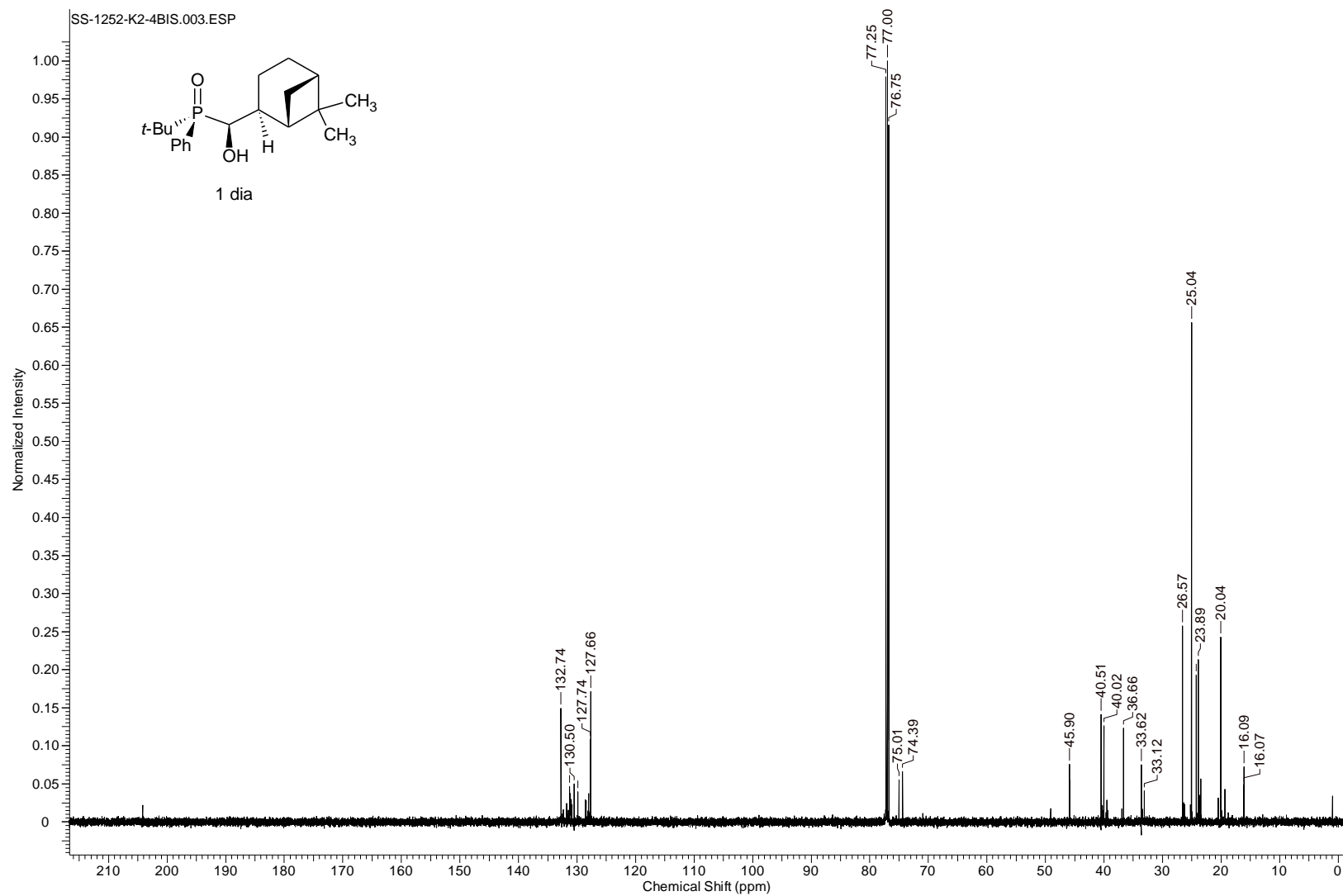
DEPT 135 NMR spectrum of (*1S,2R,3S,5R, RP*)-3-((2-anisyl)(phenyl)phosphoryl)-6,6-dimethylbicyclo[3.1.1]heptane-2-carboxylic acid (*RP*)-**5b-I** (75 MHz, CD<sub>3</sub>OD)



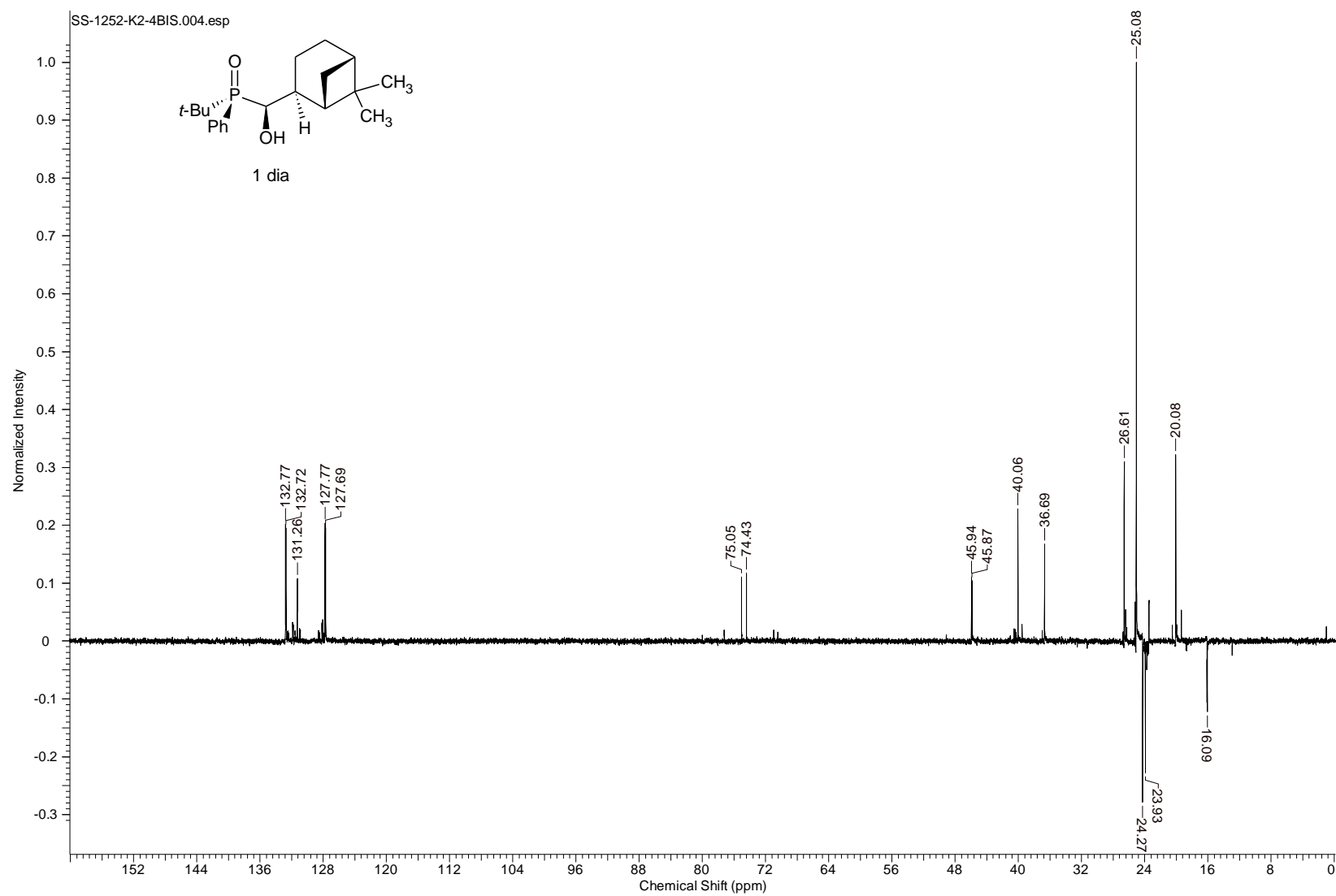
$^1\text{H}$  NMR spectrum of (*R<sub>P</sub>*)-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(*t*-butyl)(phenyl)phosphine oxide (*R<sub>P</sub>*)-(**7a-I**) (500 MHz,  $\text{CDCl}_3$ )



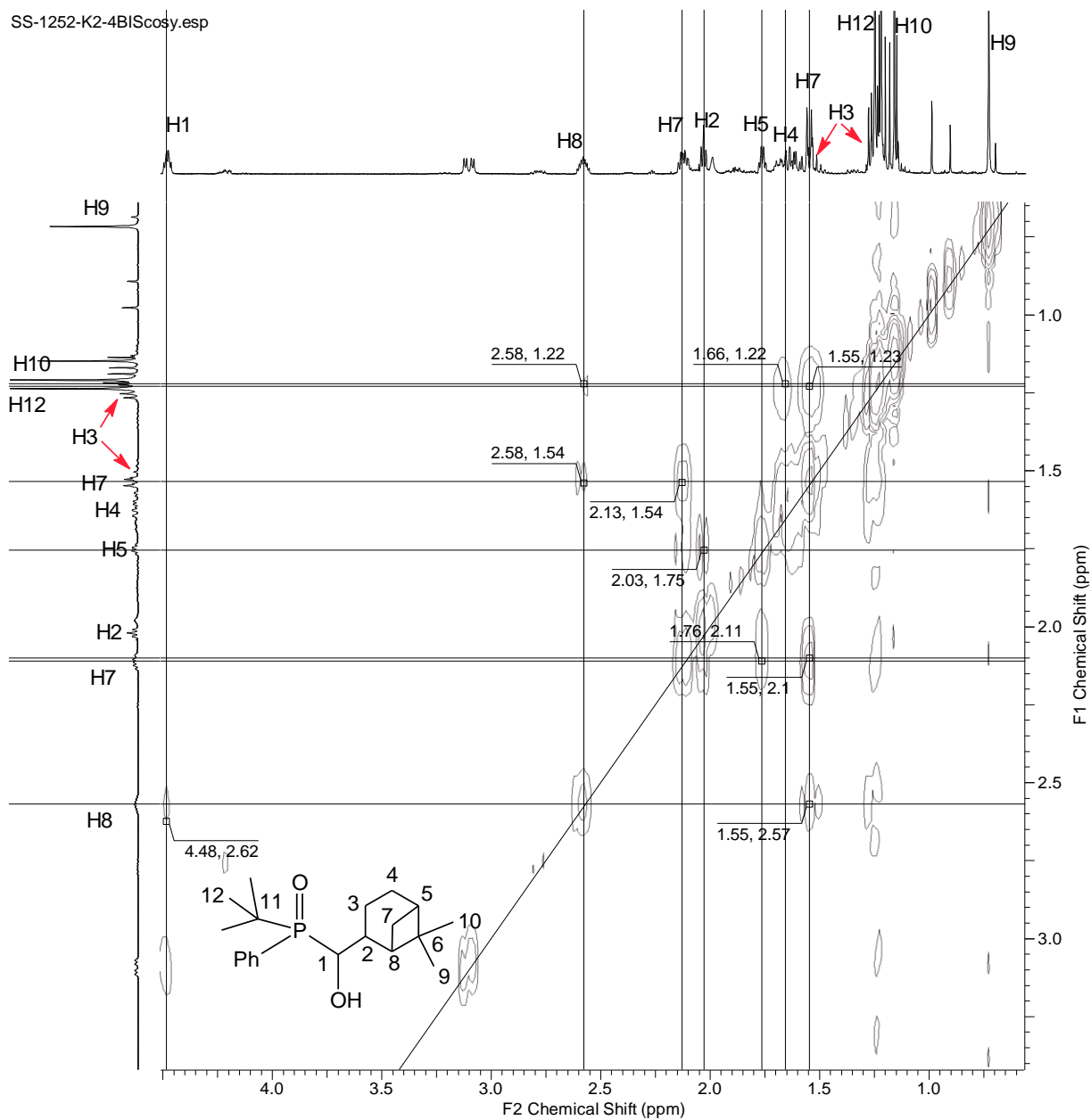
$^{31}\text{P}$  NMR spectrum of (*R<sub>P</sub>*)-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(*t*-butyl)(phenyl)phosphine oxide (*R<sub>P</sub>*)-(**7a-I**) (202 MHz,  $\text{CDCl}_3$ )



$^{13}\text{C}$  NMR spectrum of (*R<sub>P</sub>*)-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(*t*-butyl)(phenyl)phosphine oxide (*R<sub>P</sub>*)-(7a-I) (125 MHz,  $\text{CDCl}_3$ )

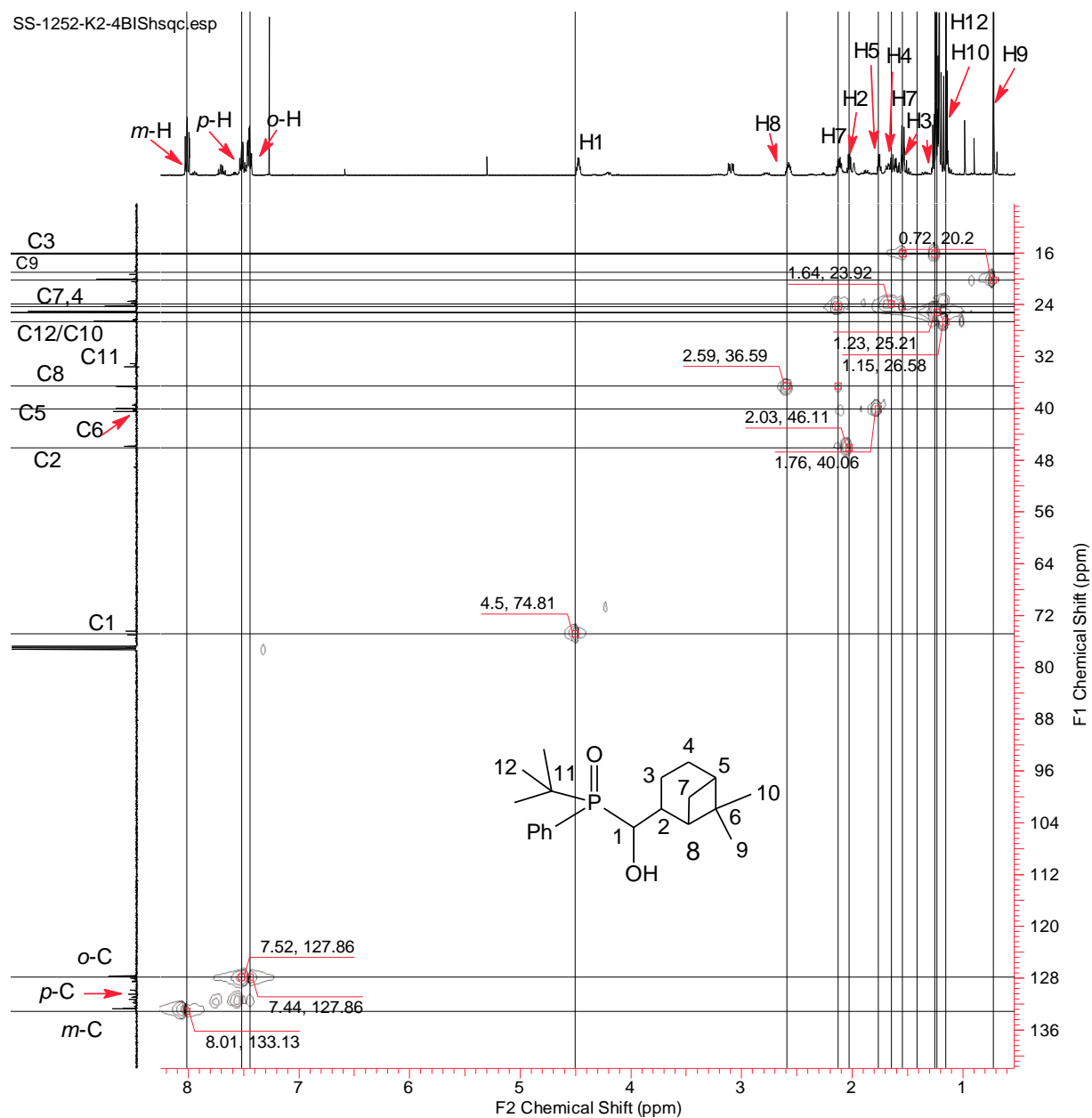


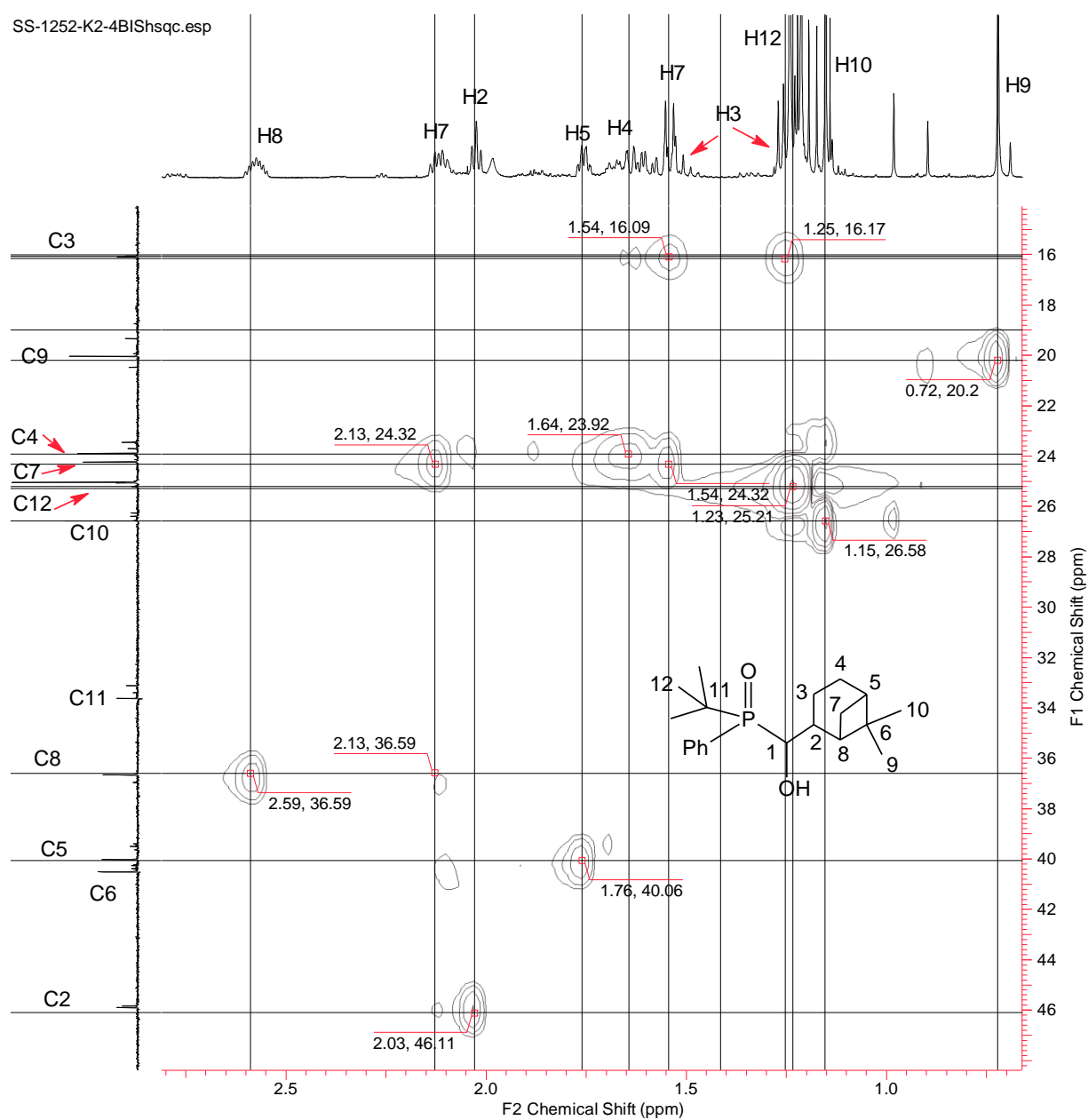
DEPT 135 spectrum of (*R<sub>P</sub>*)-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(*t*-butyl)(phenyl)phosphine oxide (*R<sub>P</sub>*)-(**7a-I**) (125 MHz, CDCl<sub>3</sub>)



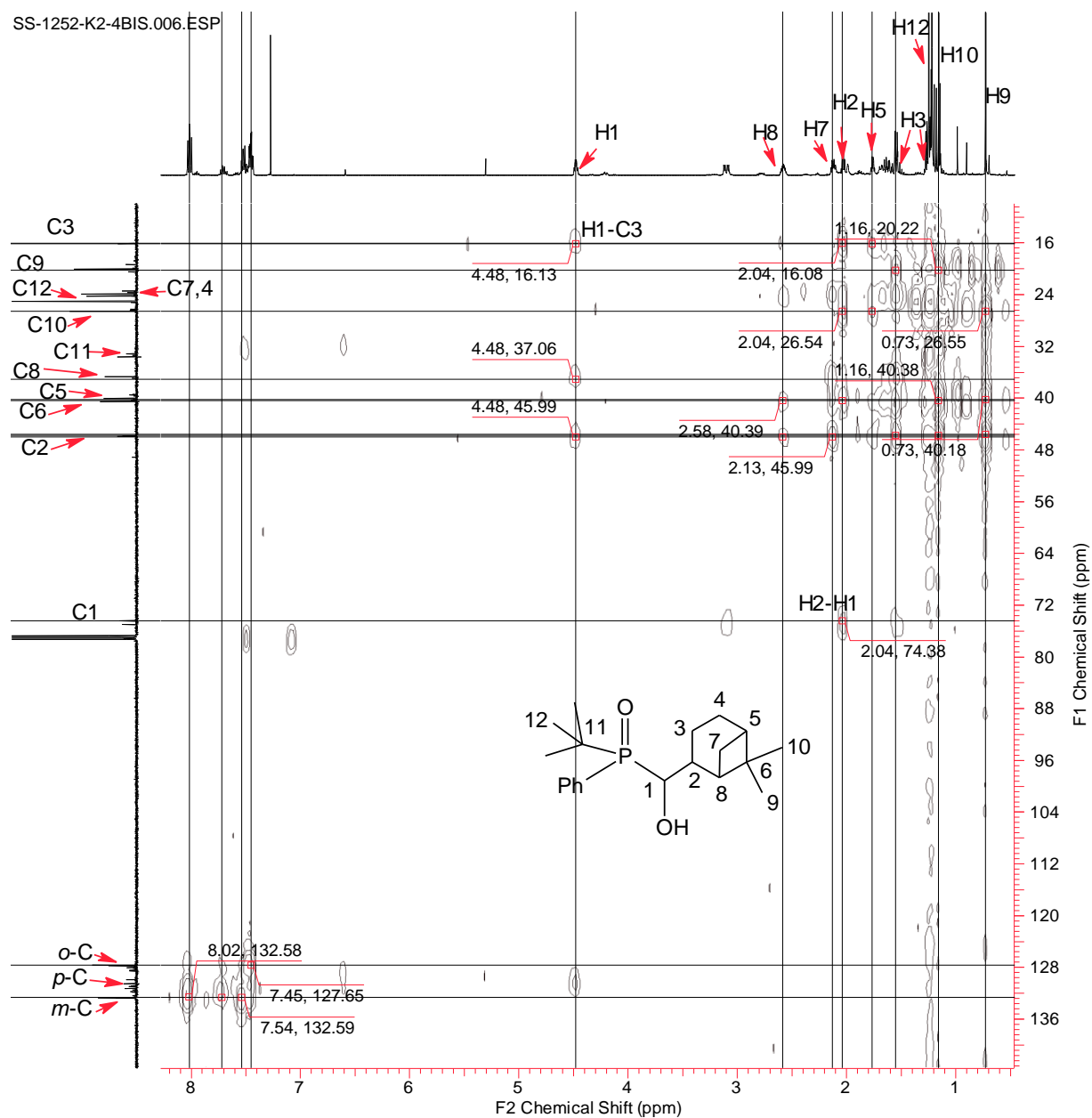
COSY NMR spectrum of  $(R_P)$ -6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(*t*-butyl)(phenyl)phosphine oxide ( $R_P$ )-(**7a-I**) (500, 125 MHz,  $\text{CDCl}_3$ )

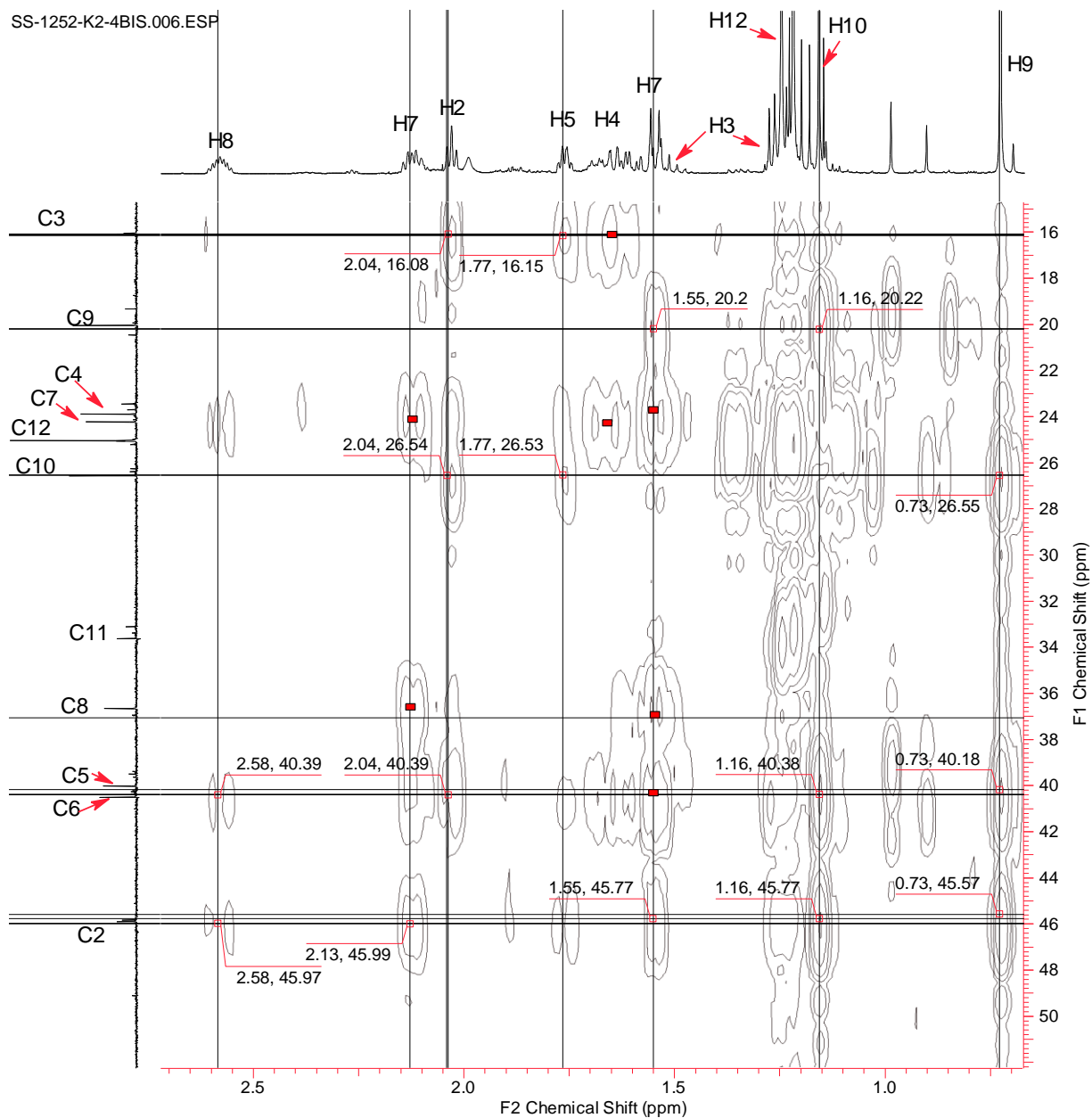
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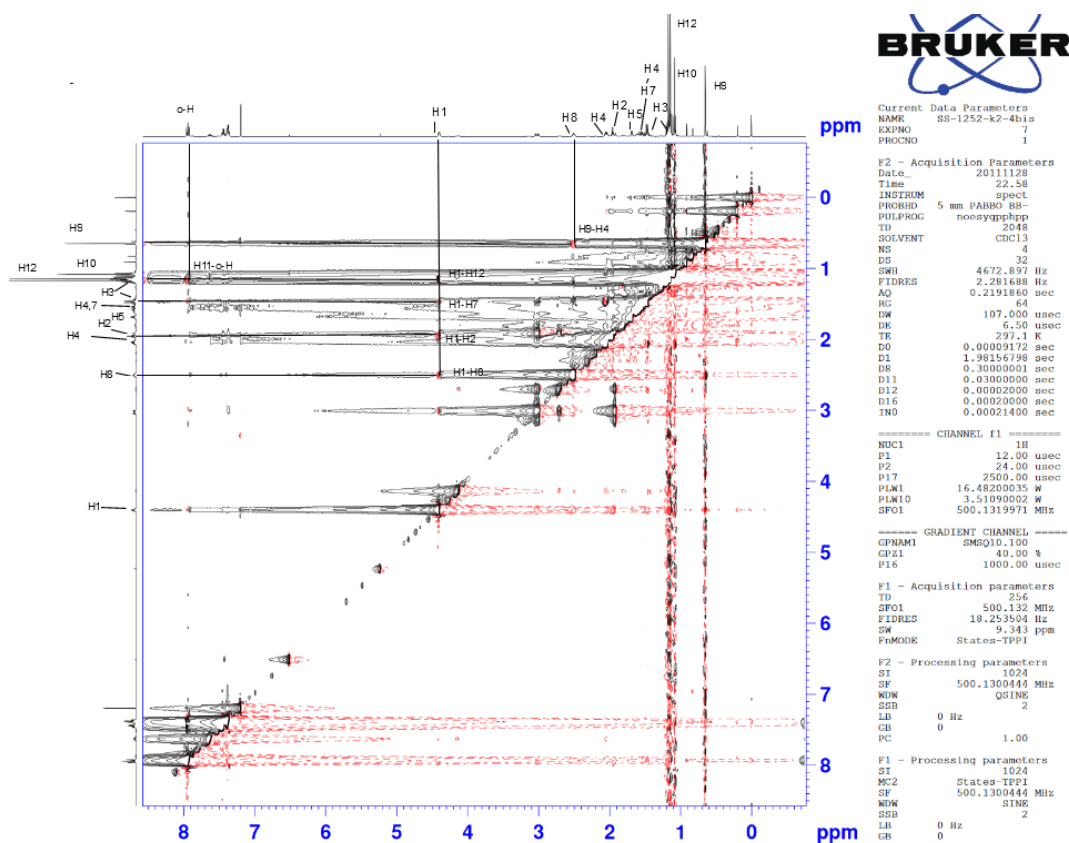


HSQC NMR spectrum of  $(R_P)$ -6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(t-butyl)(phenyl)phosphine oxide  $(R_P)$ -(7a-I) (500, 125 MHz,  $\text{CDCl}_3$ )

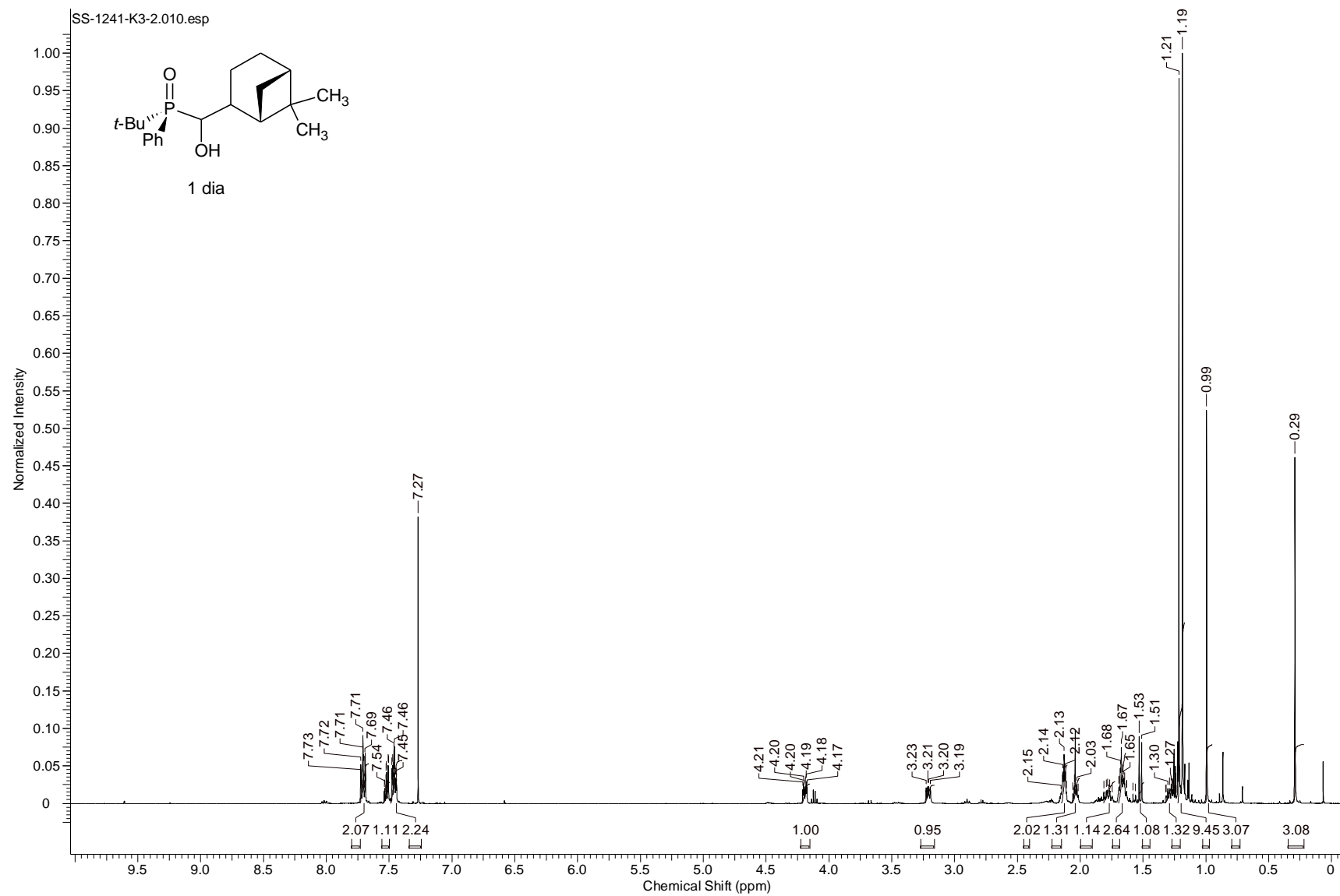




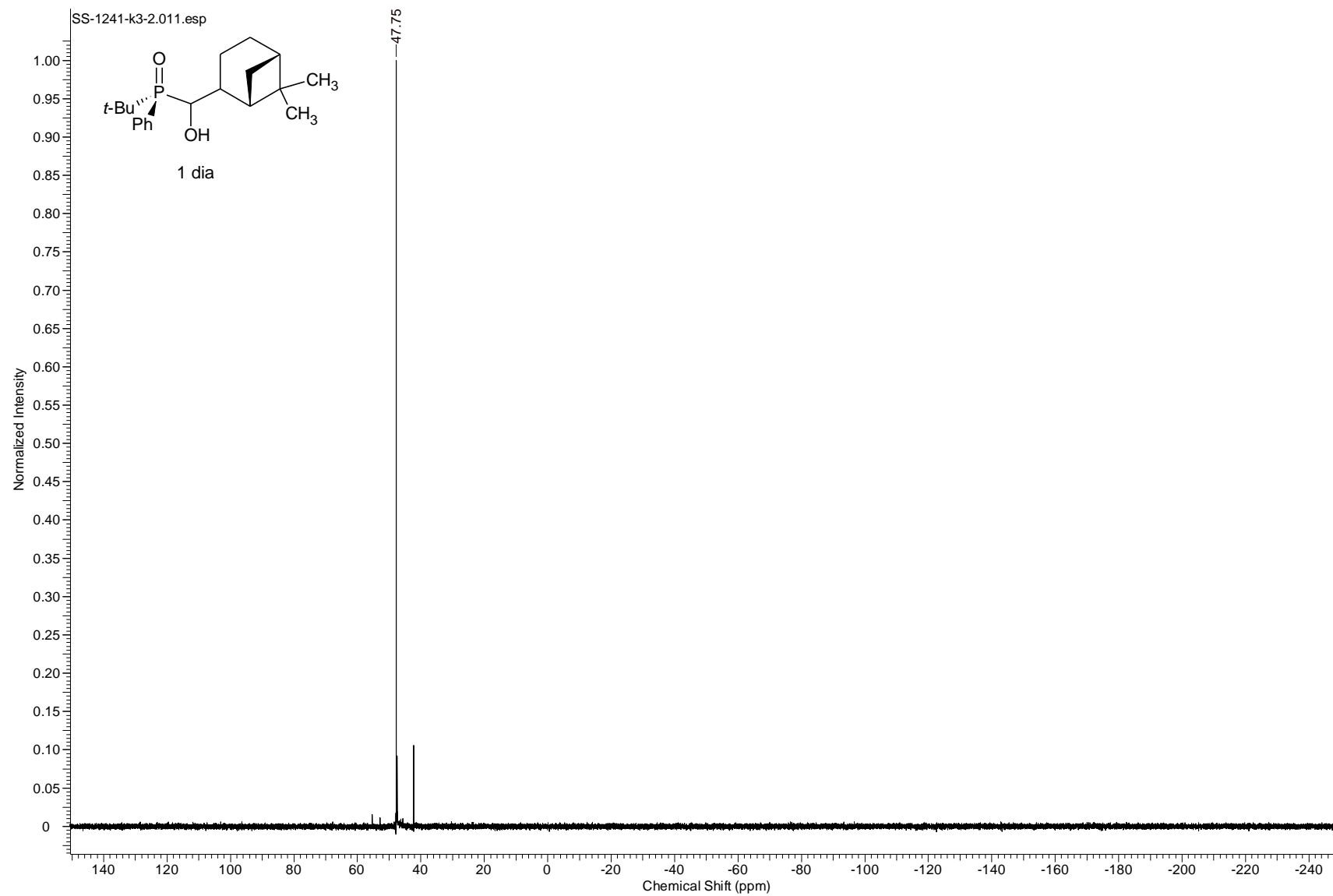
HMBC NMR spectrum of (*R*)-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(*t*-butyl)(phenyl)phosphine oxide (*R*)-**(7a-I)** (500, 125 MHz, CDCl<sub>3</sub>)



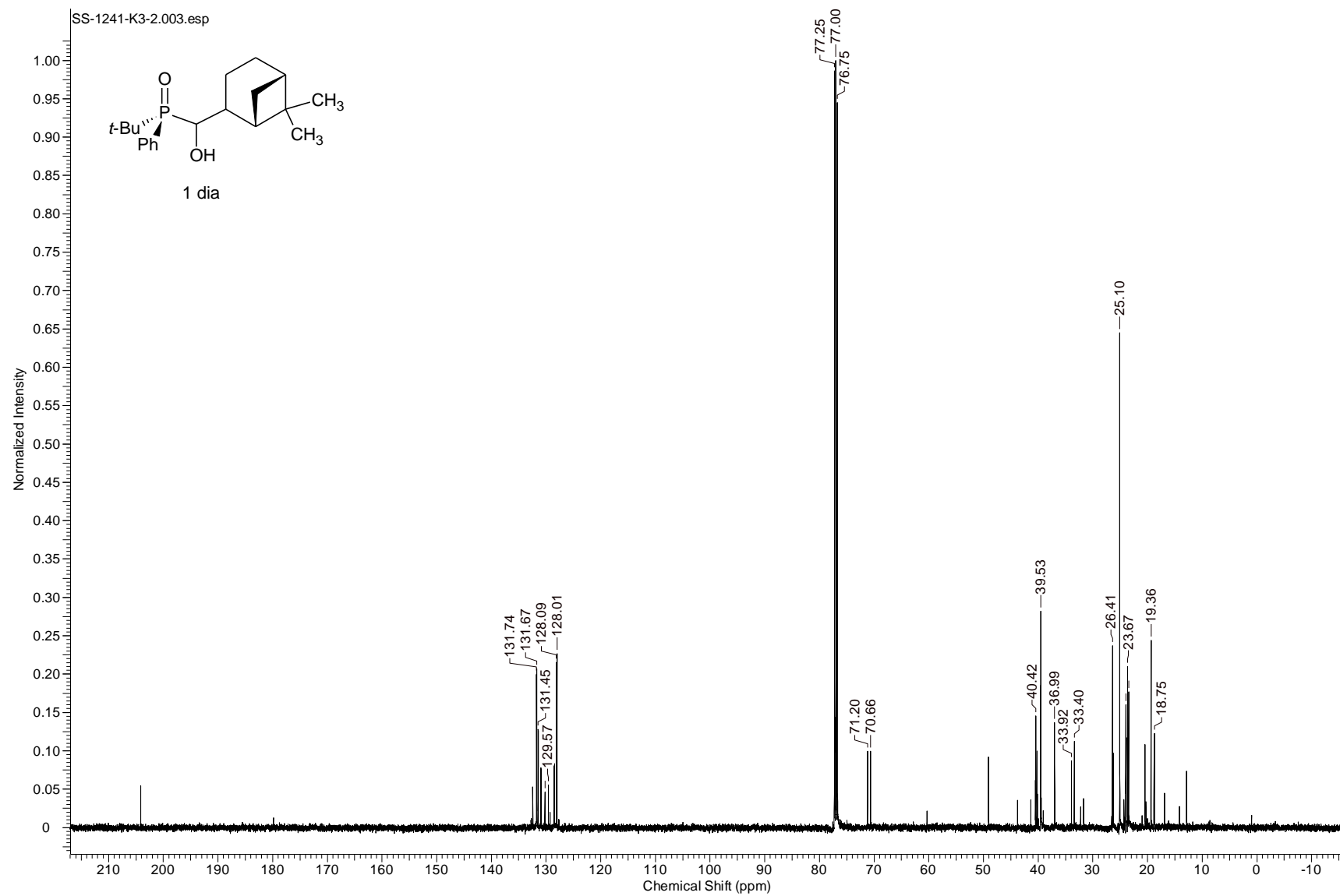
NOESY NMR spectrum of (*R<sub>P</sub>*)-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(*t*-butyl)(phenyl)phosphine oxide (*R<sub>P</sub>*)-(7a-I) (500 MHz, CDCl<sub>3</sub>)



<sup>1</sup>H NMR spectrum of (*R<sub>P</sub>*)-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(*t*-butyl)(phenyl)phosphine oxide (**7a-II**) (500 MHz, CDCl<sub>3</sub>)

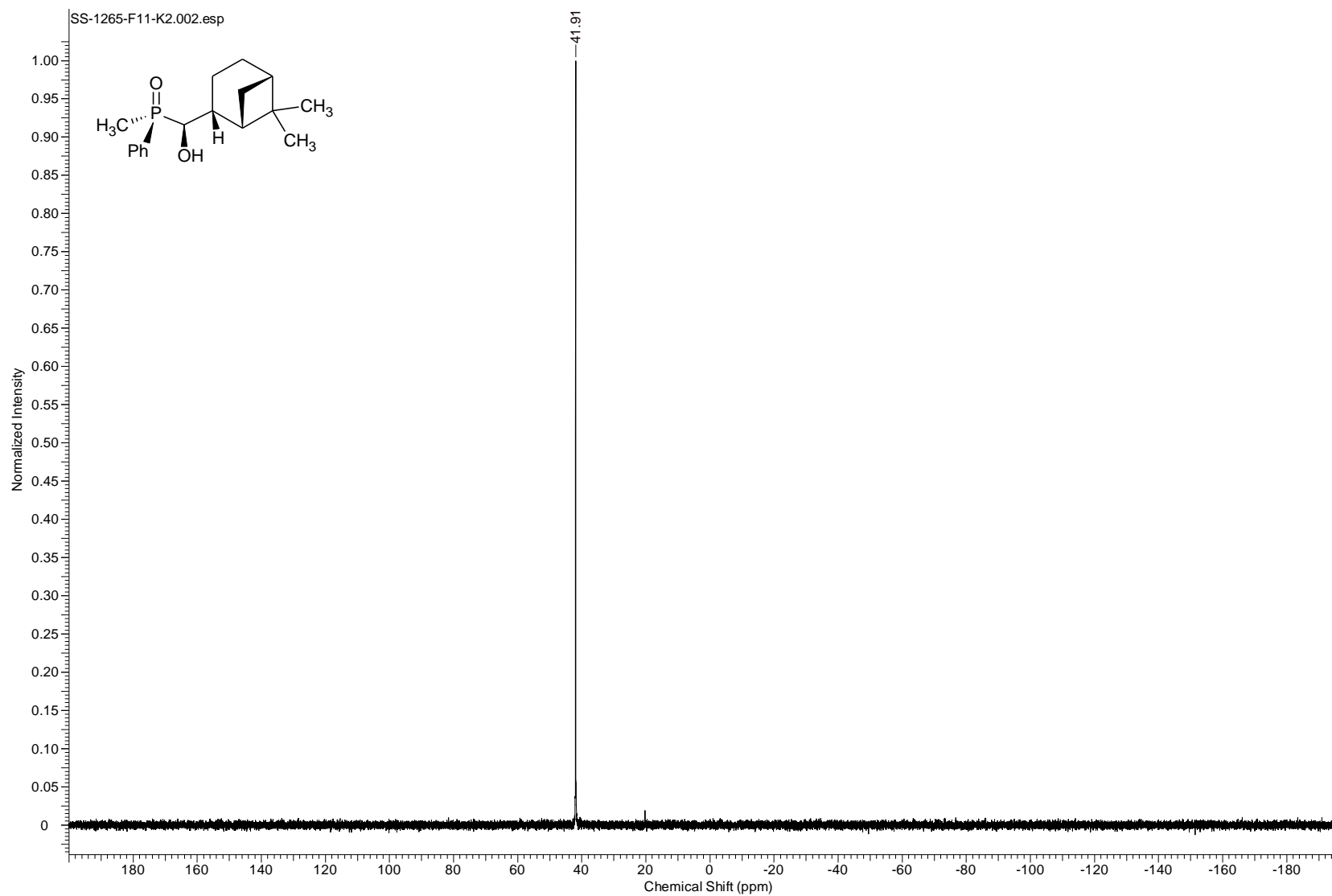


$^{31}\text{P}$  NMR spectrum of (*R<sub>P</sub>*)-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(*t*-butyl)(phenyl)phosphine oxide (**7a-II**) (202 MHz,  $\text{CDCl}_3$ )



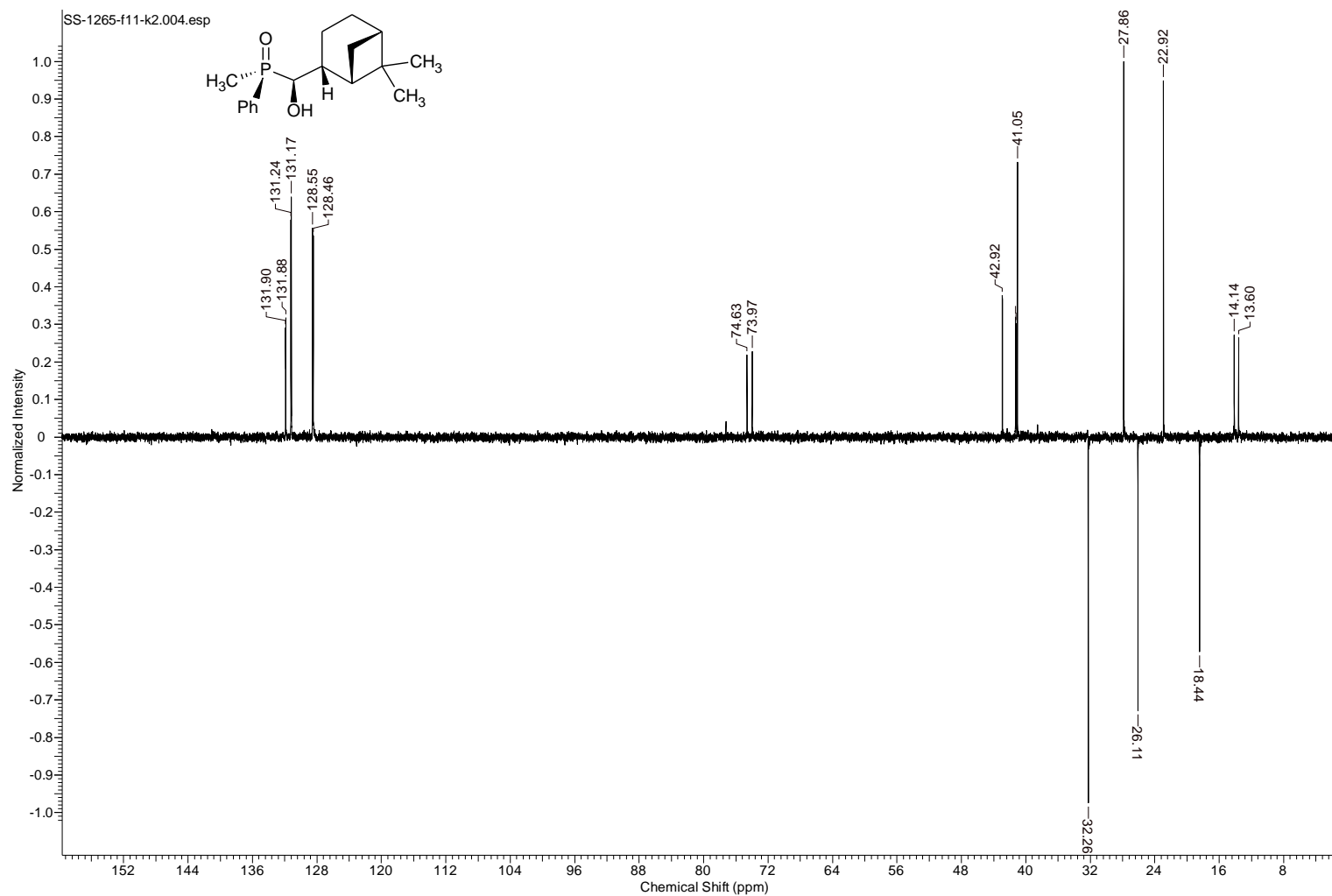
$^{13}\text{C}$  NMR spectrum of  $(R_P)$ -6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(*t*-butyl)(phenyl)phosphine oxide (**7a-II**) (125 MHz,  $\text{CDCl}_3$ )



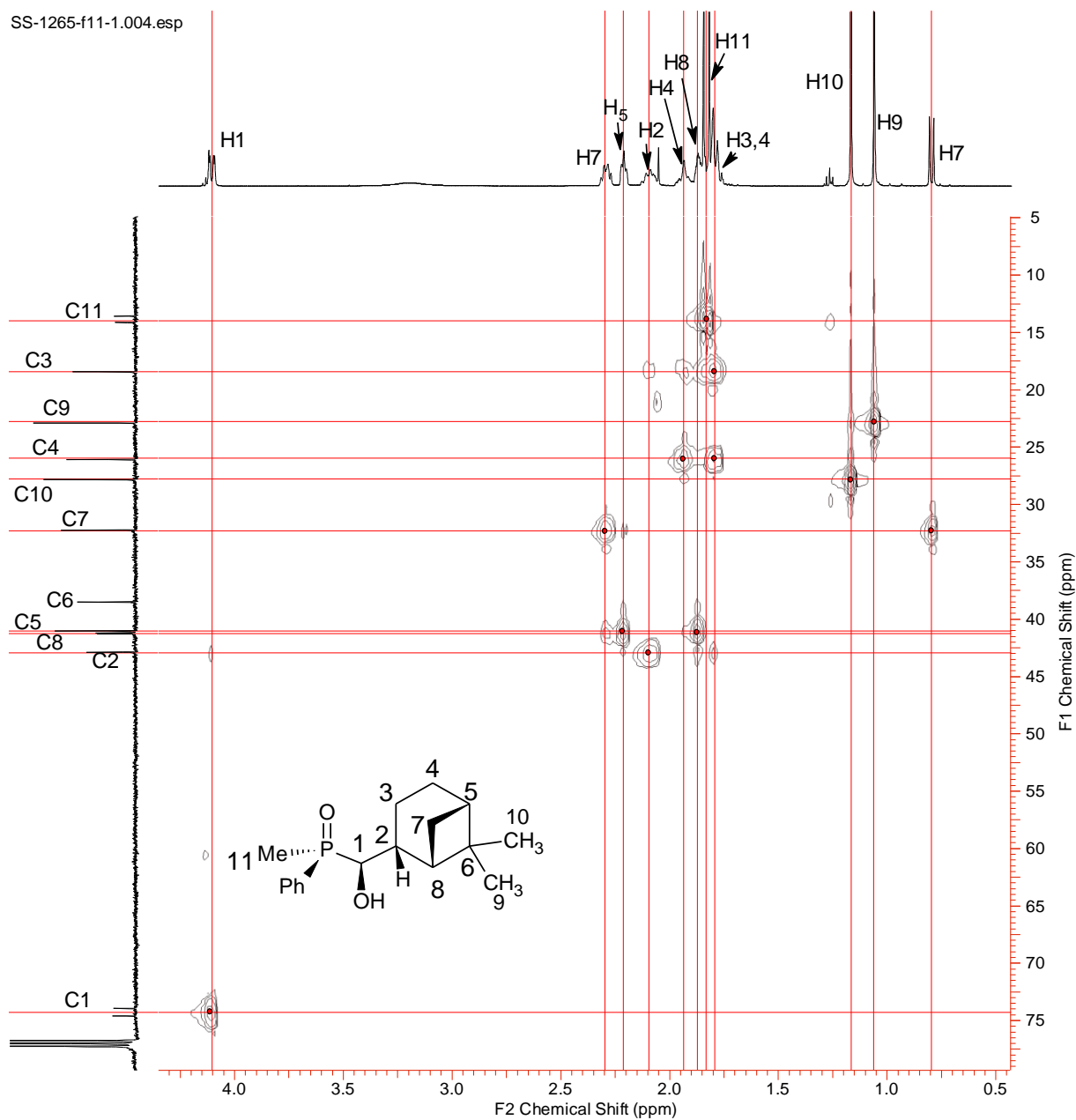


$^{31}\text{P}$  NMR spectrum of  $(R_P)$ -6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(methyl)(phenyl)phosphine oxide ( $R_P$ -**9c-I**) (202 MHz,  $\text{CDCl}_3$ )



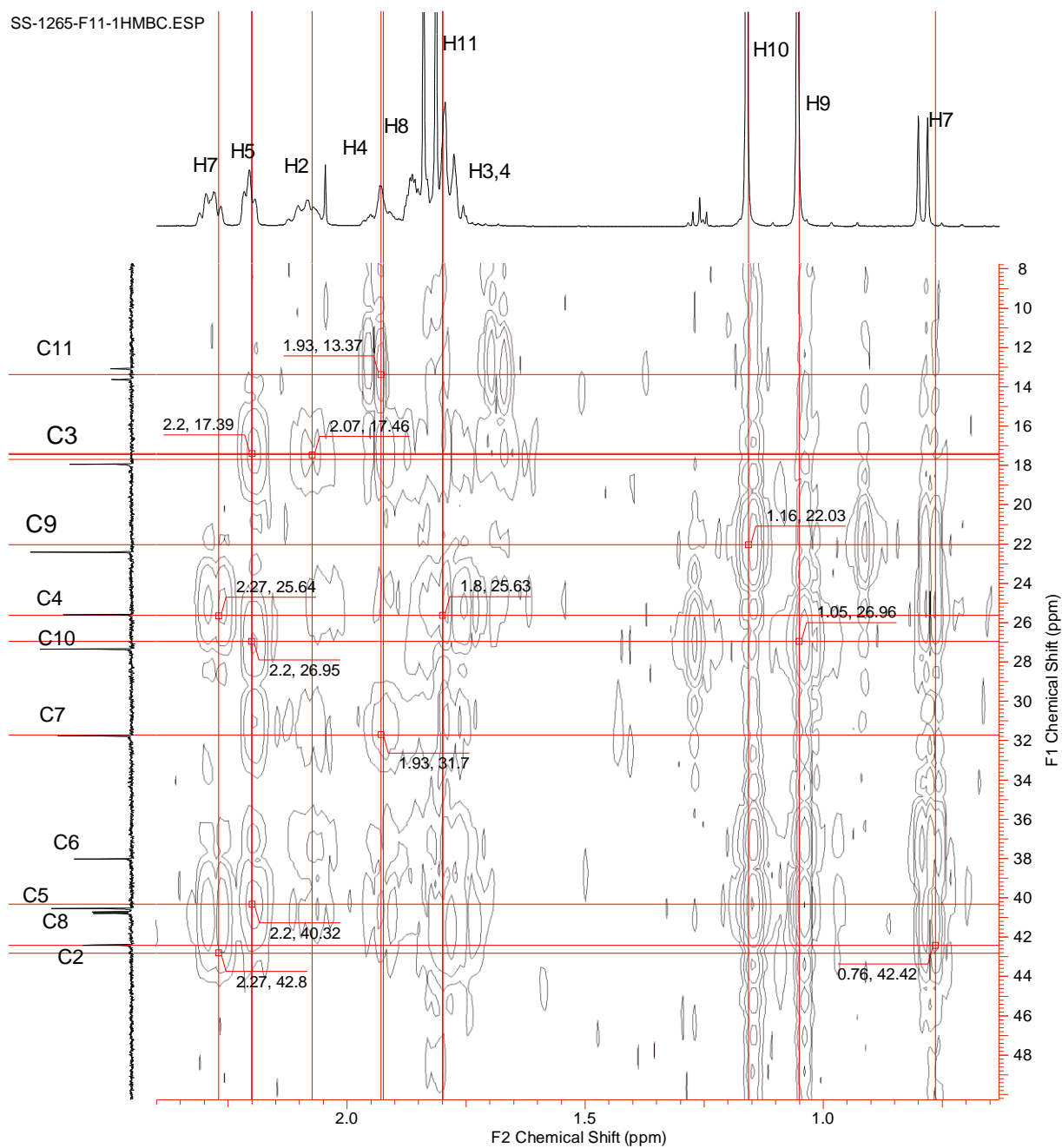


DEPT 135 NMR spectrum of  $(R_P)$ -6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(methyl)(phenyl)phosphine oxide ( $R_P$ )-**9c-I** (125 MHz,  $\text{CDCl}_3$ )

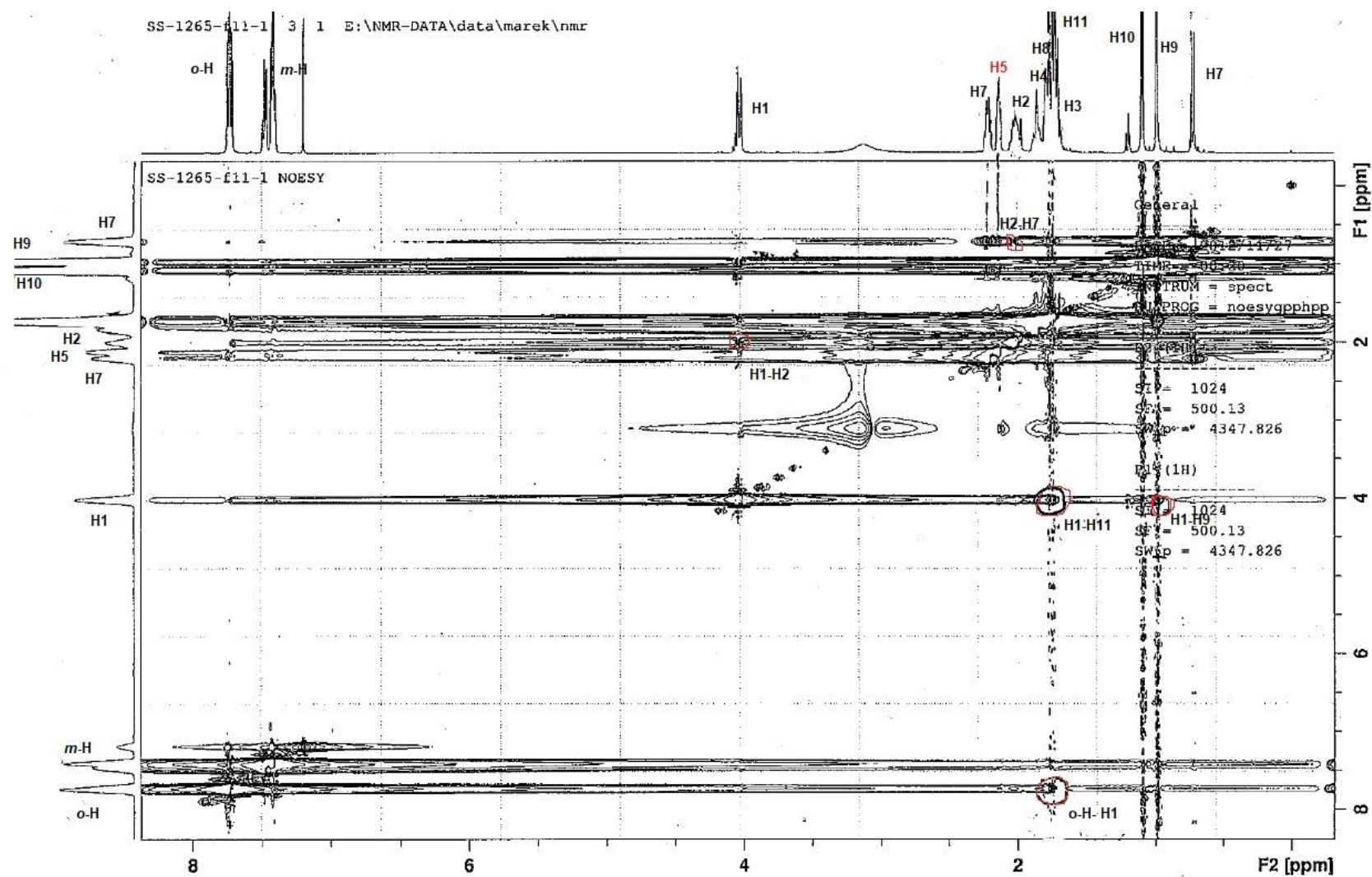


HSQC spectrum of *(R)*-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(methyl)(phenyl)phosphine oxide (*(R)*-**9c-I**) (500,125 MHz,  $\text{CDCl}_3$ )

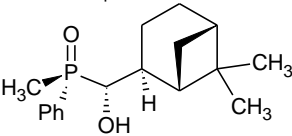




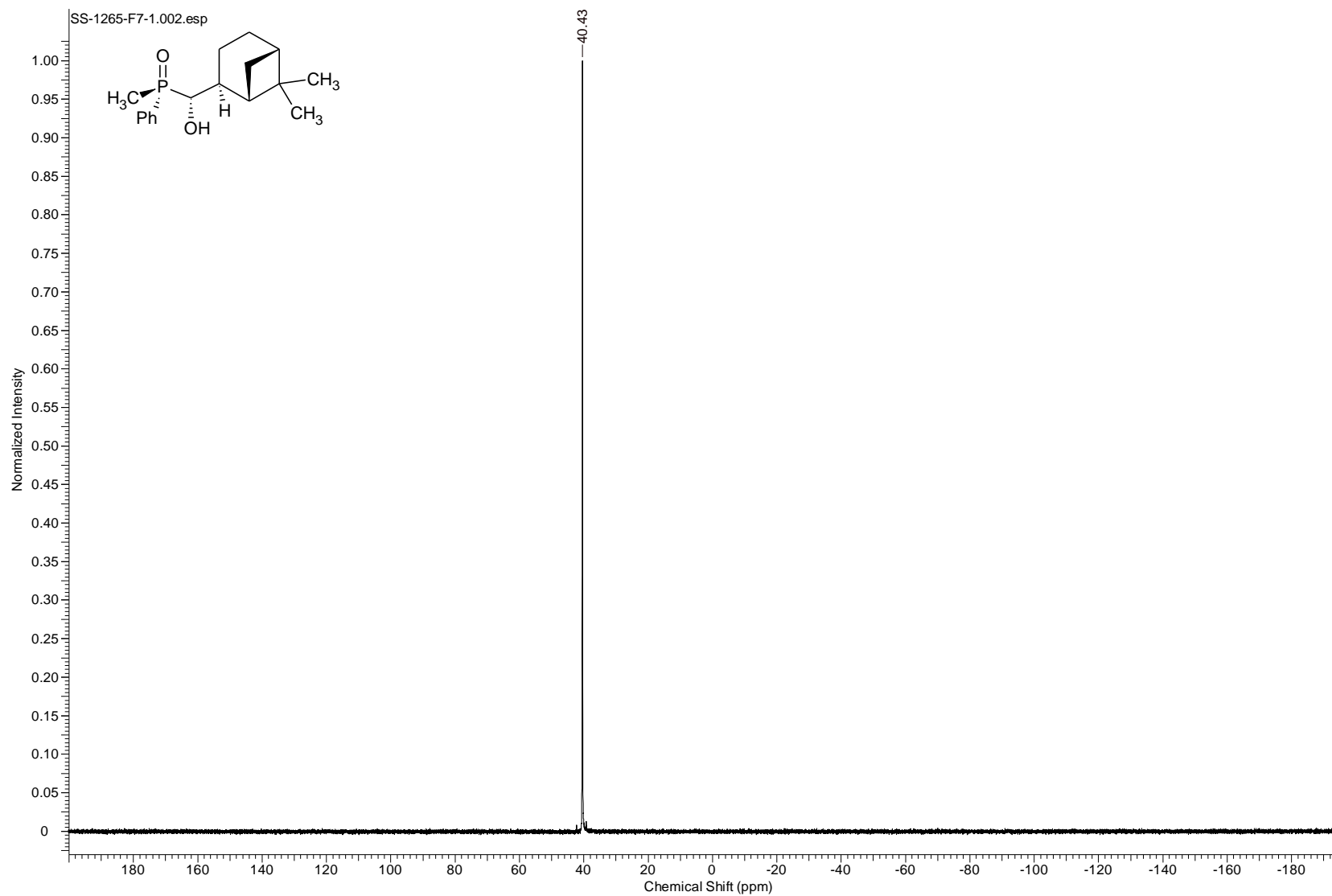
HMBC spectrum of (*R<sub>P</sub>*)-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(methyl)(phenyl)phosphine oxide (*R<sub>P</sub>*)-**9c-I** (500,125 MHz, CDCl<sub>3</sub>)



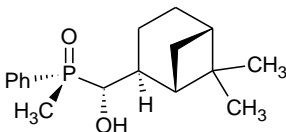
NOESY spectrum of (*R<sub>P</sub>*)-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(methyl)(phenyl)phosphine oxide (*R<sub>P</sub>*)-**9c-I** (500MHz, CDCl<sub>3</sub>)



S46

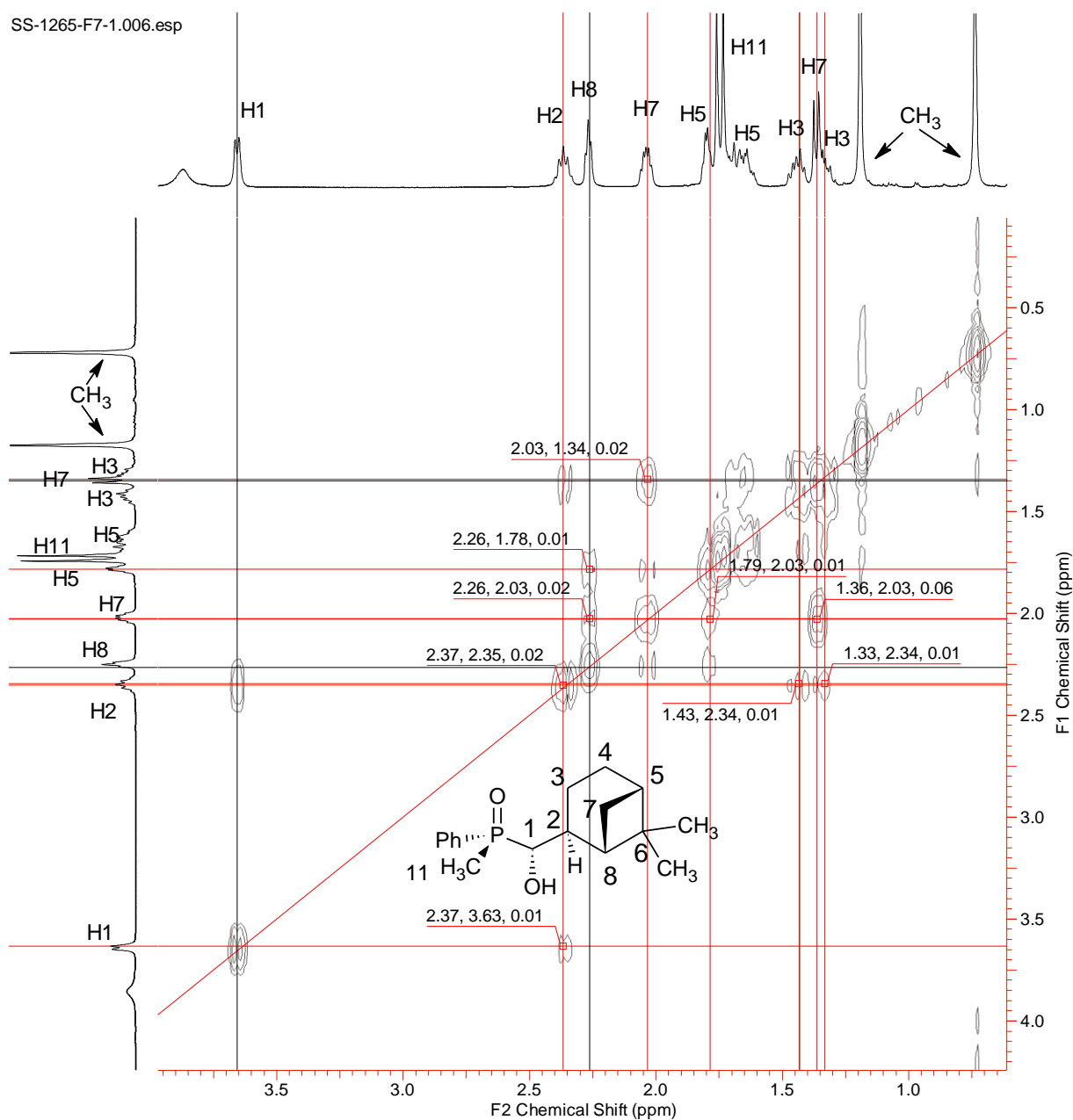


$^{31}\text{P}$  NMR spectrum of *(S\_P)*-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(methyl)(phenyl)phosphine oxide (*(S\_P)*-**9c-II**) (202 MHz,  $\text{CDCl}_3$ )

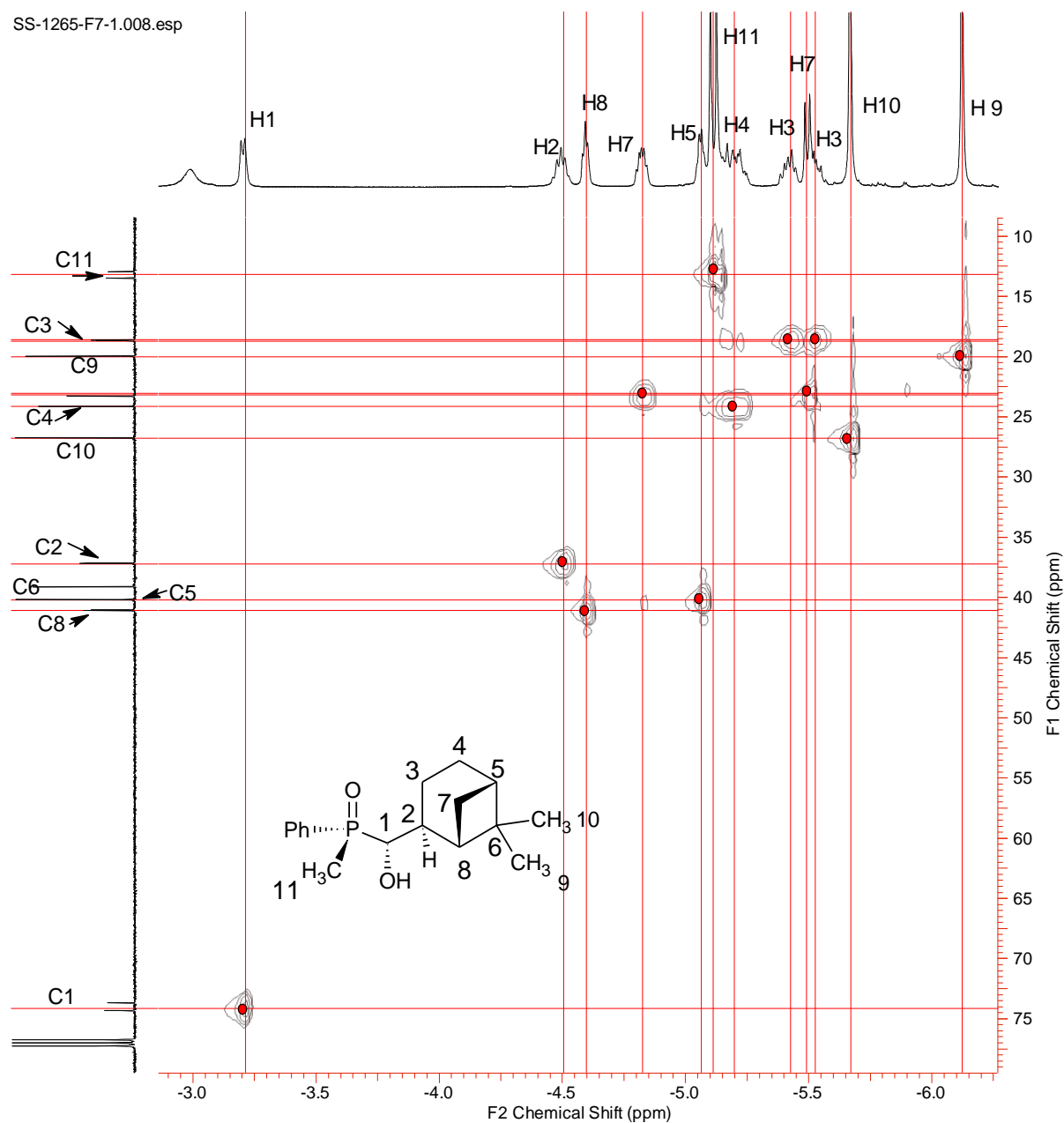


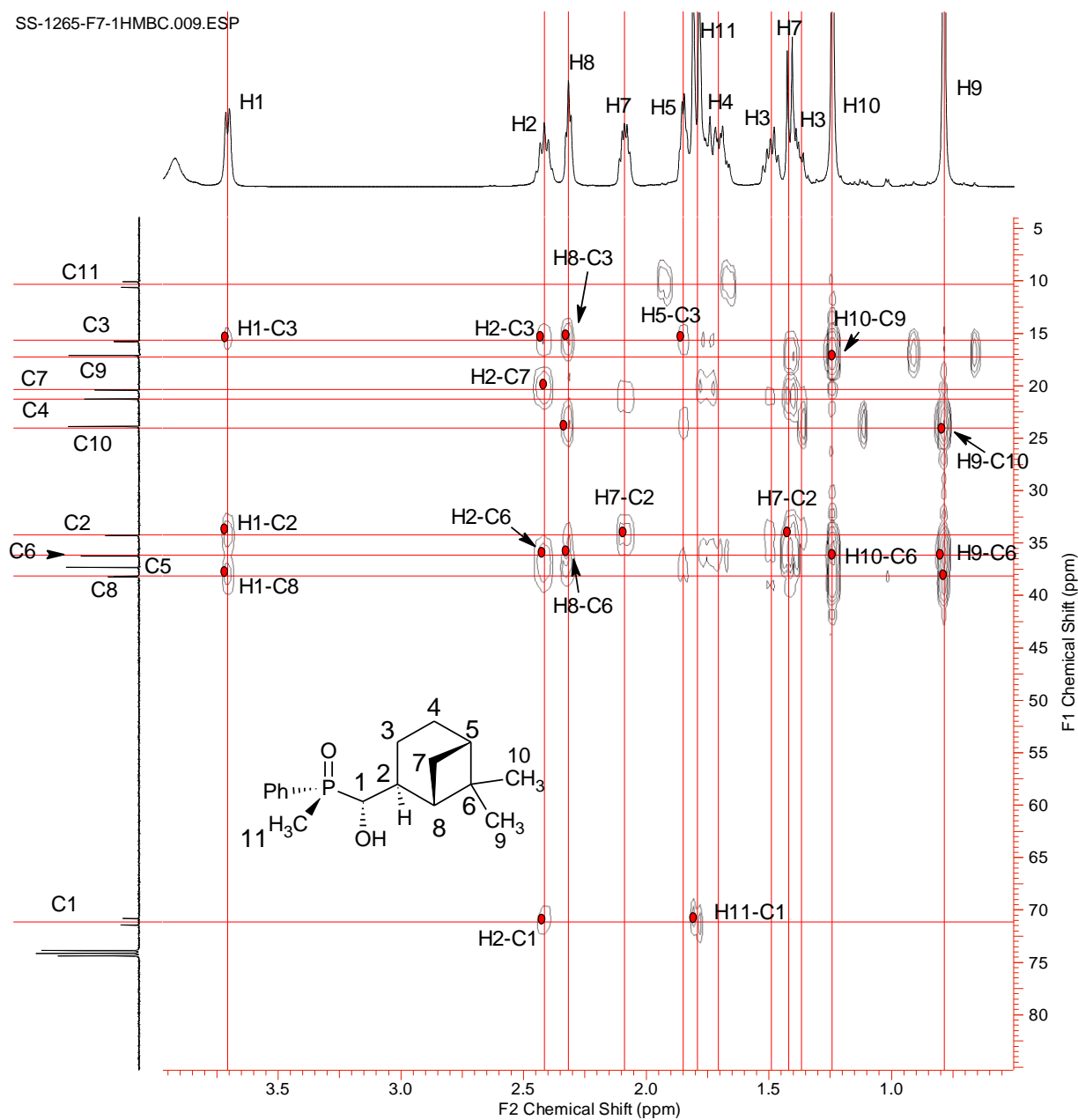
S48

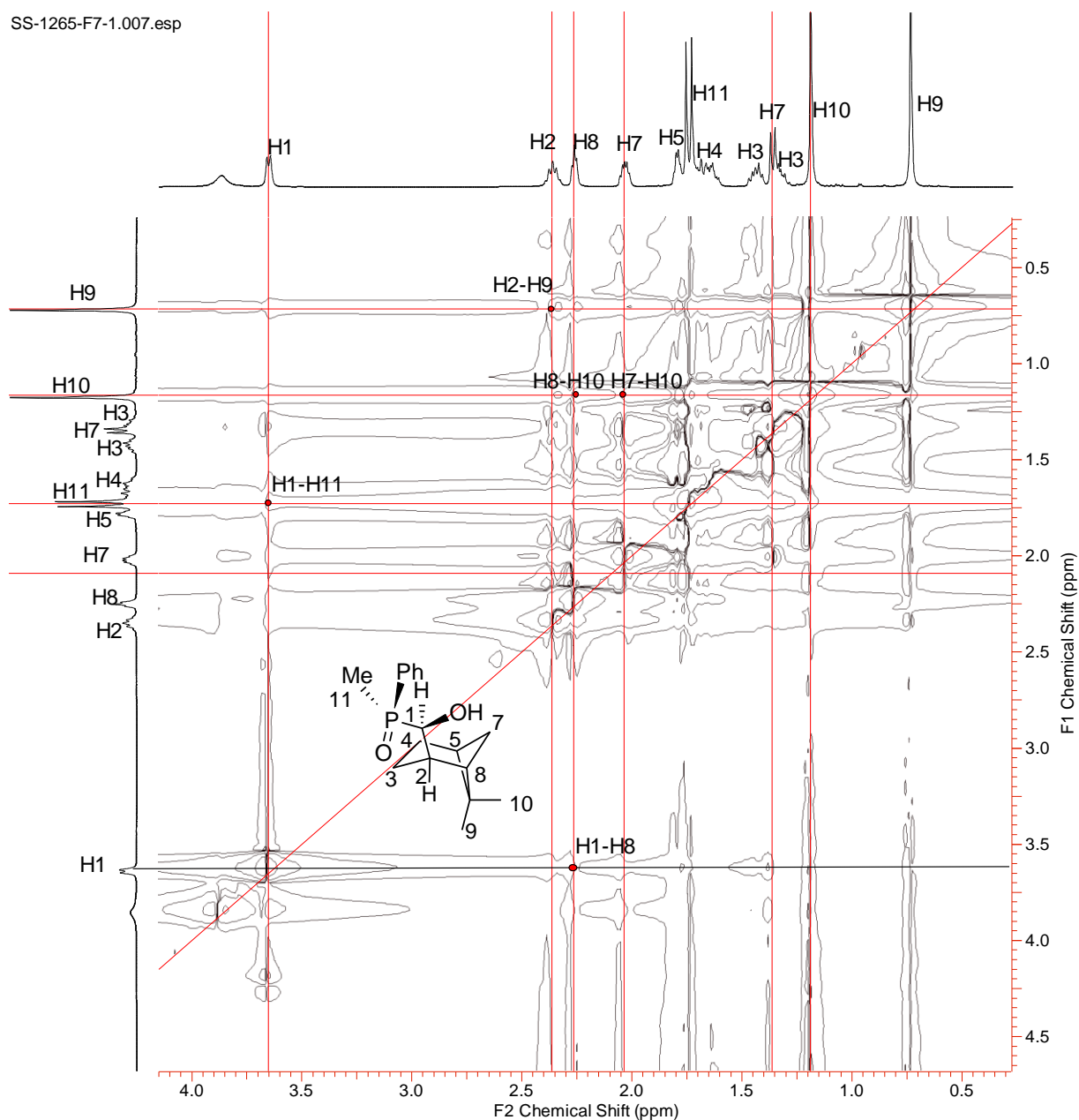


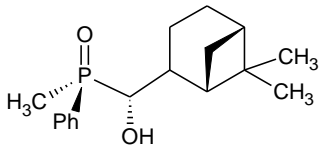


COSY spectrum of (*S<sub>P</sub>*)-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(methyl)(phenyl)phosphine oxide (*S<sub>P</sub>*)-**9c-II** (500 MHz, CDCl<sub>3</sub>)

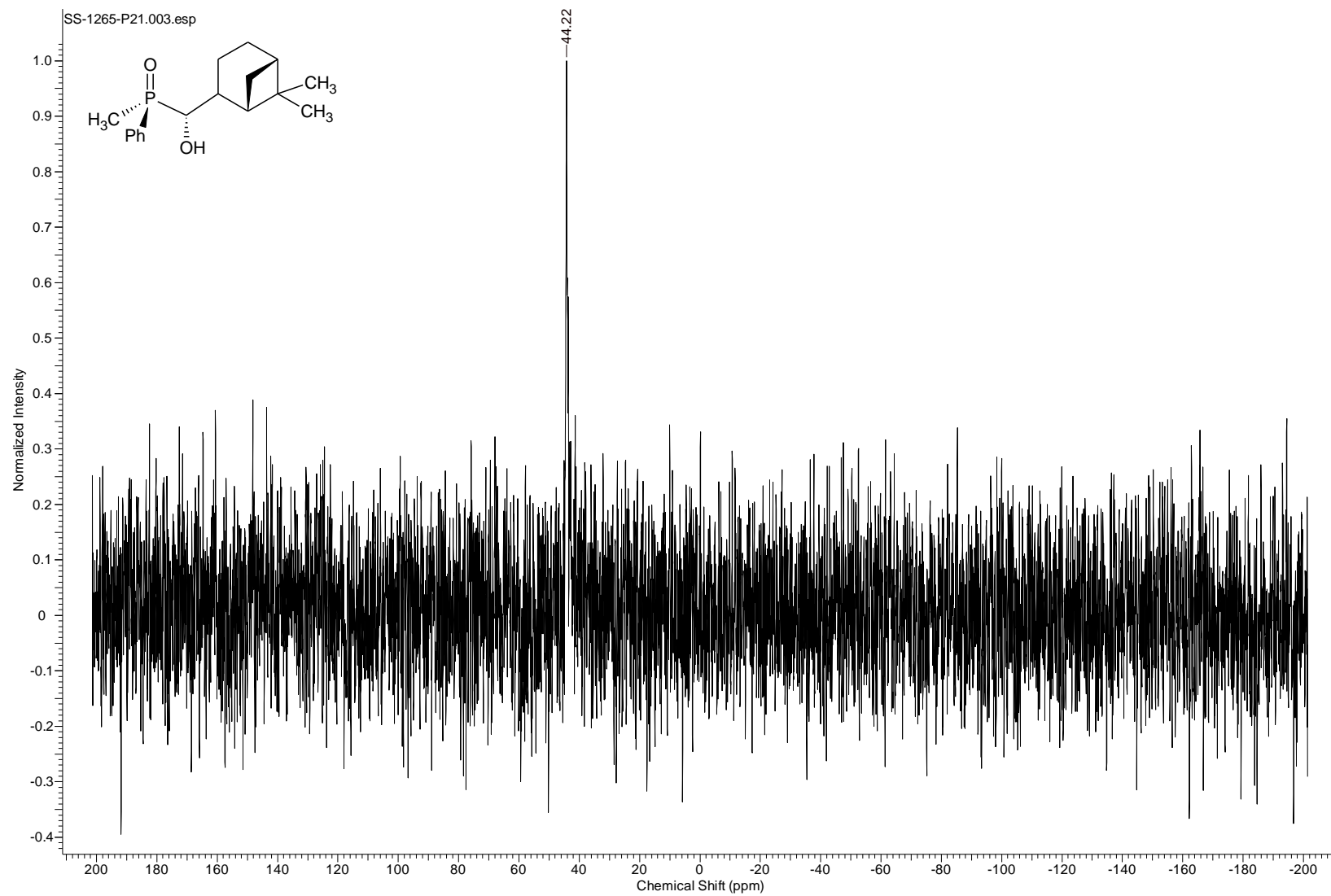




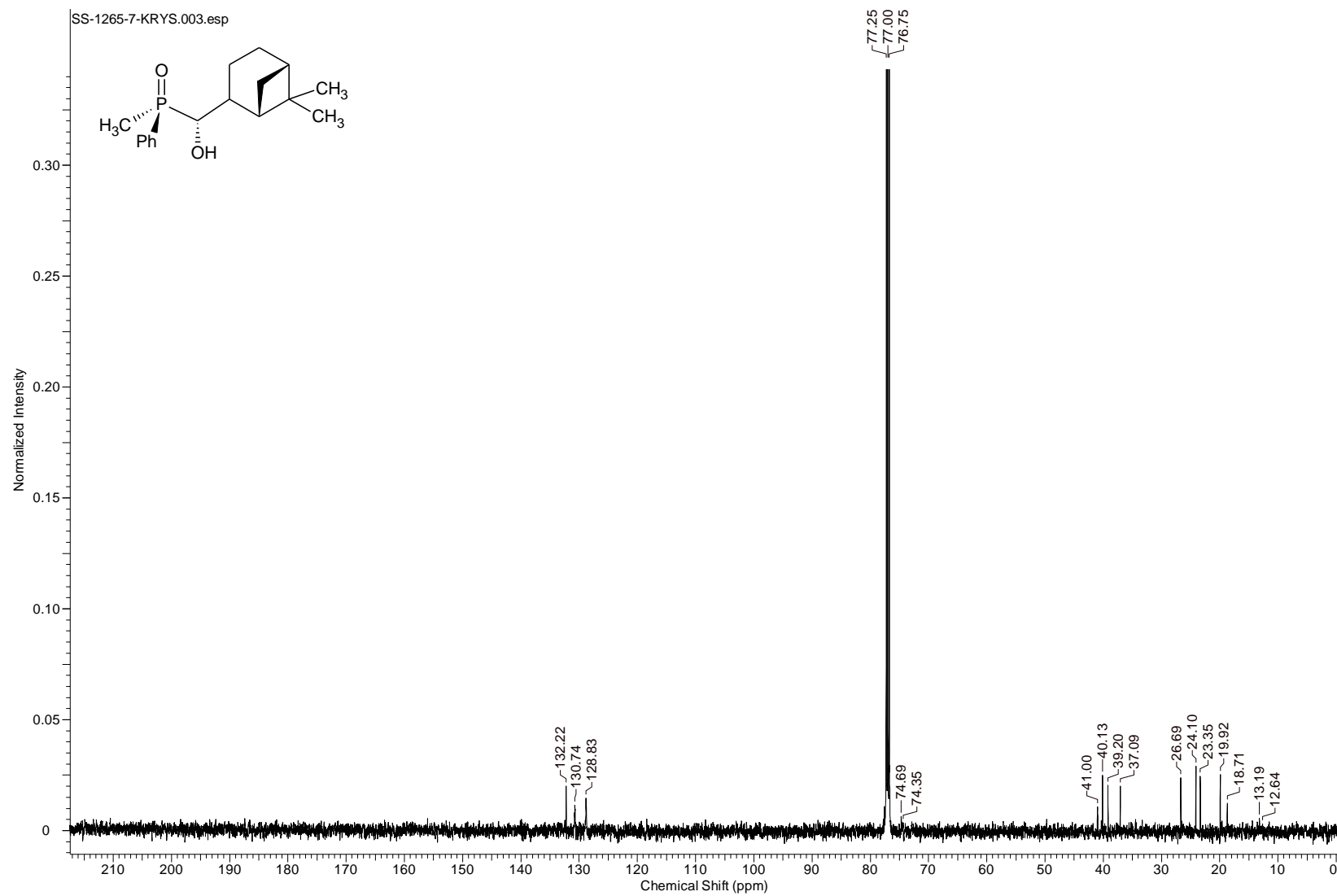




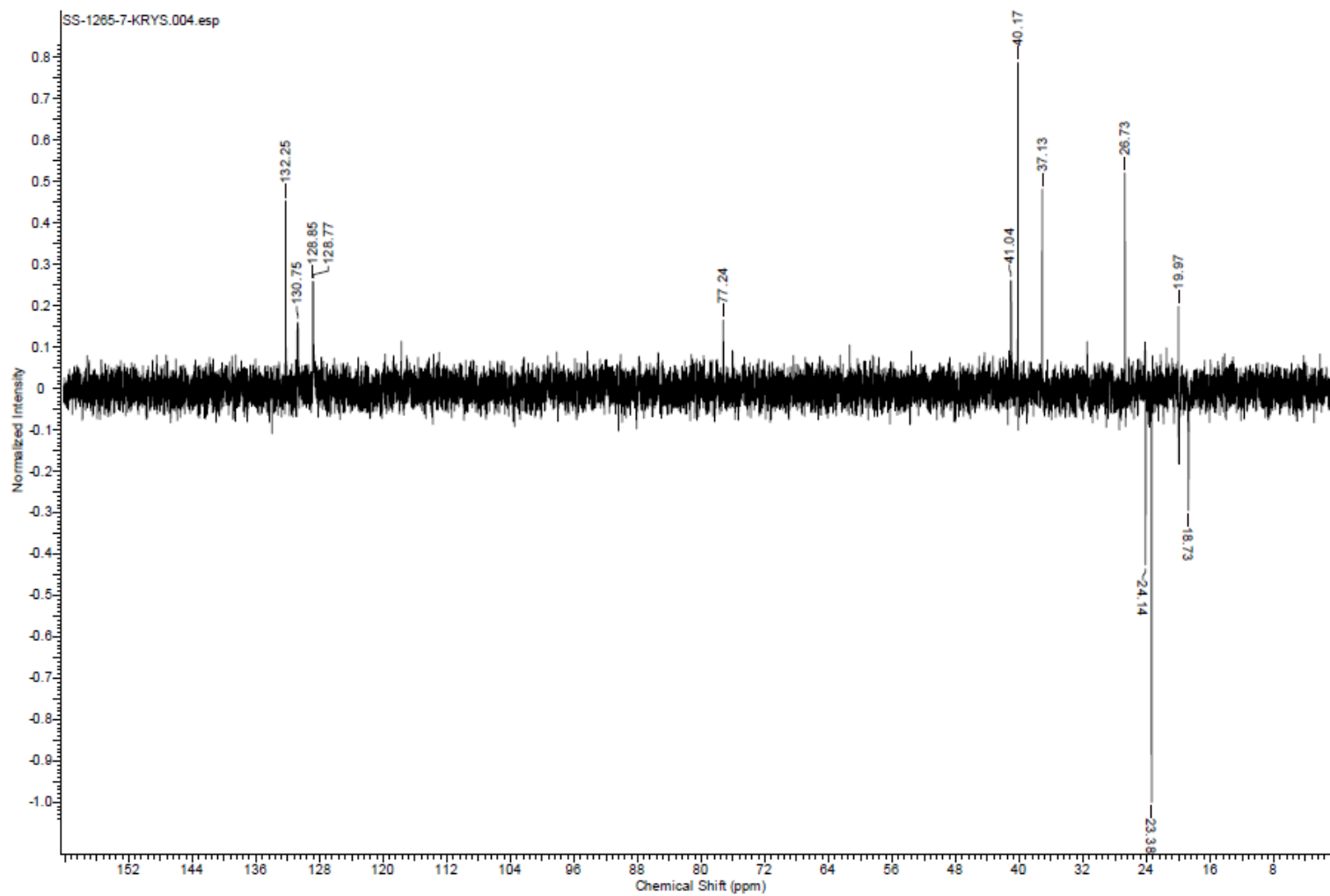
S54



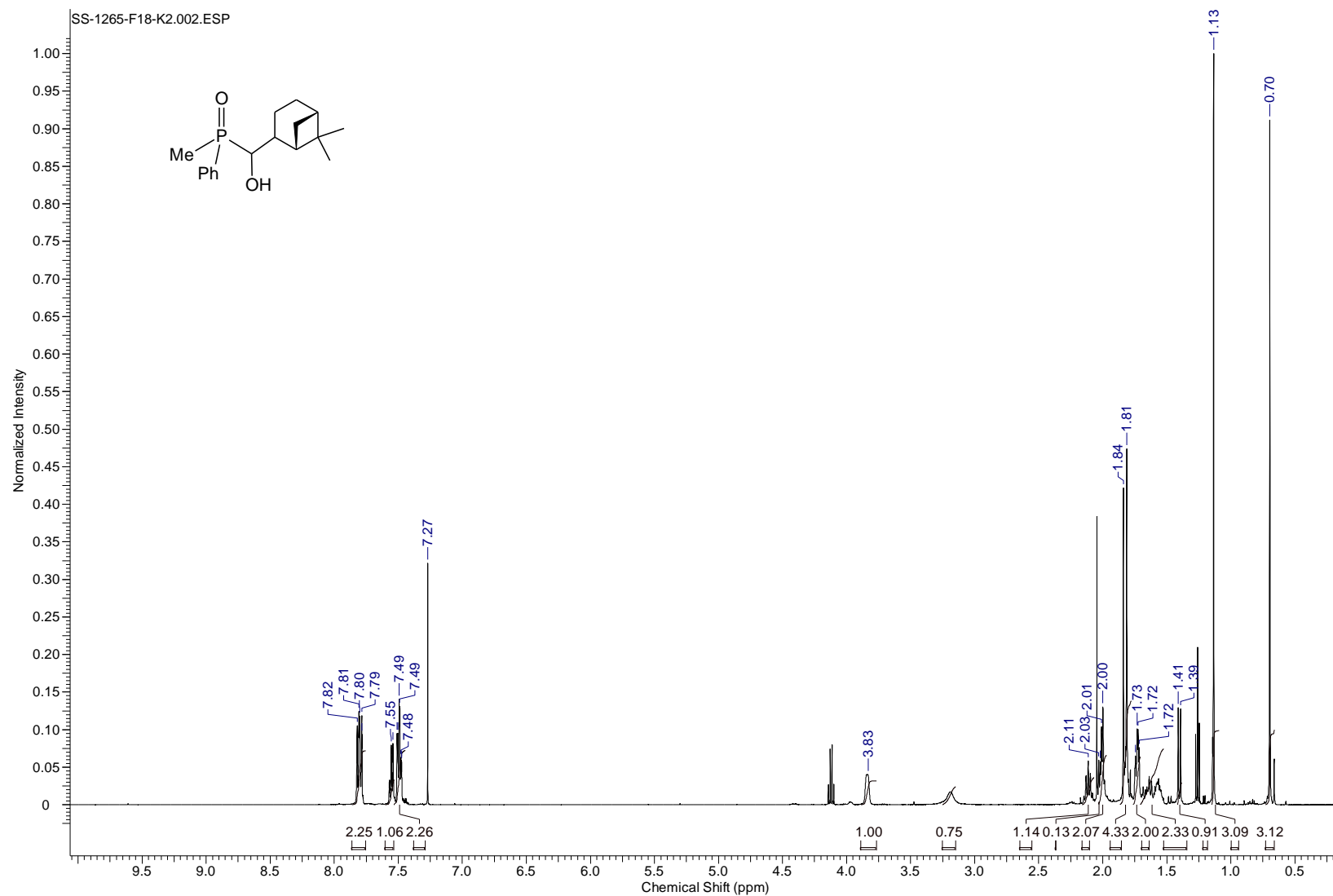
$^{31}\text{P}$  NMR spectrum of  $(R_P)$ -6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(methyl)(phenyl)phosphine oxide ( $R_P$ )-**9c-III** (202 MHz,  $\text{CDCl}_3$ )



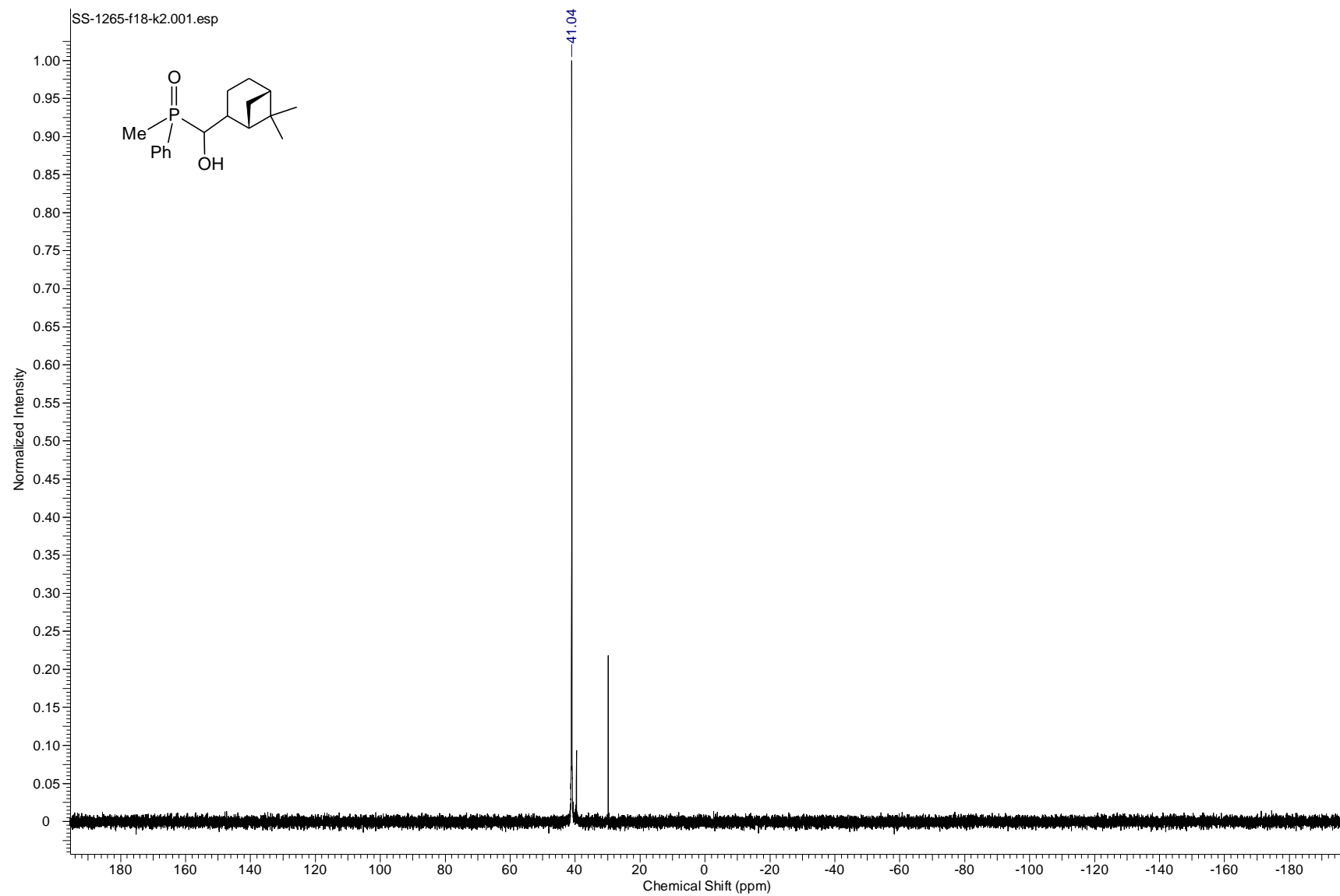
$^{13}\text{C}$  NMR spectrum of *(R\_P)*-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(methyl)(phenyl)phosphine oxide (*(R\_P)*-**9c-III**) (125 MHz,  $\text{CDCl}_3$ )



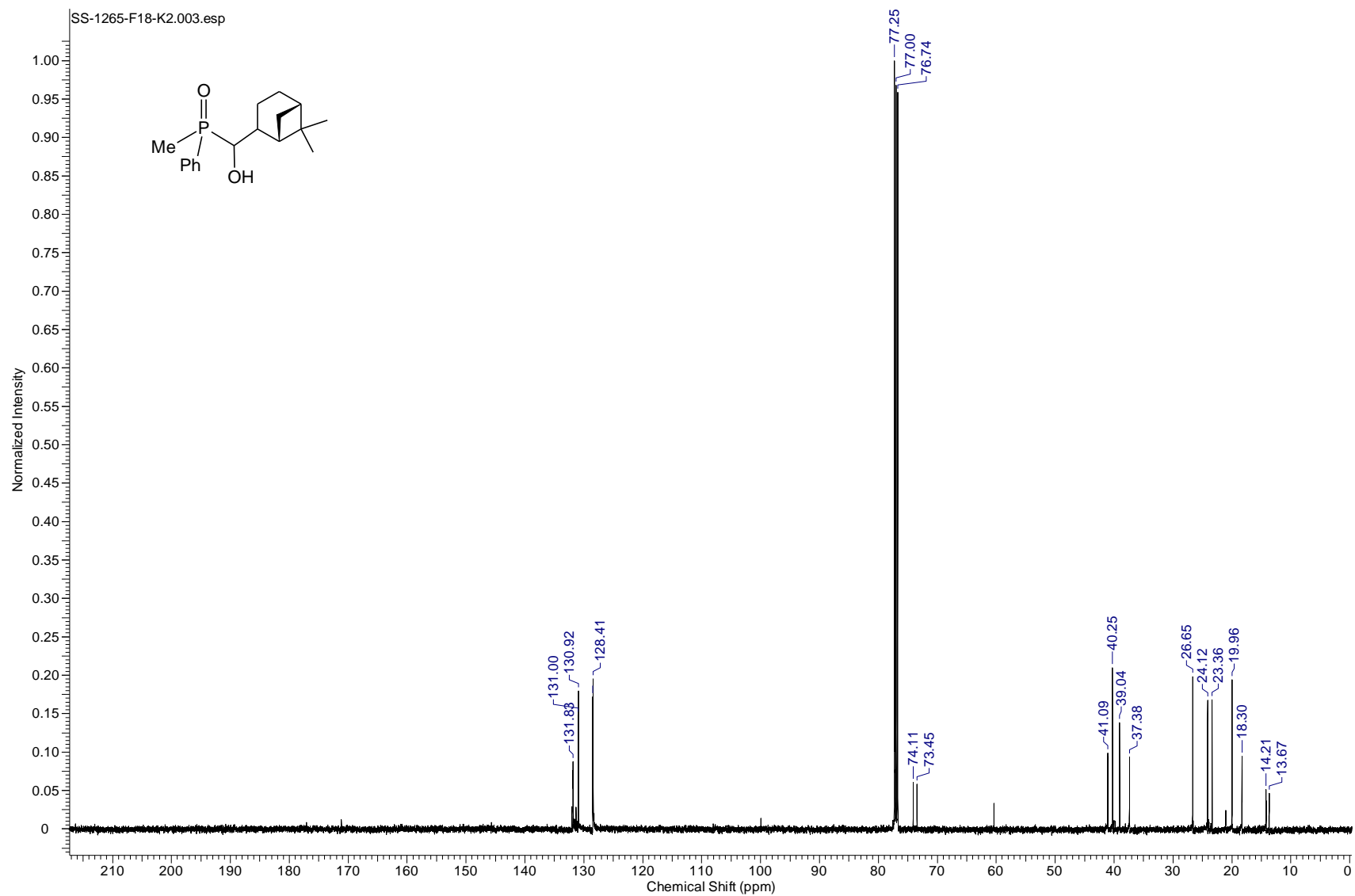
DEPT135 NMR spectrum of (*R<sub>P</sub>*)-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(methyl)(phenyl)phosphine oxide (*R<sub>P</sub>*)-9c-III (125 MHz, CDCl<sub>3</sub>)



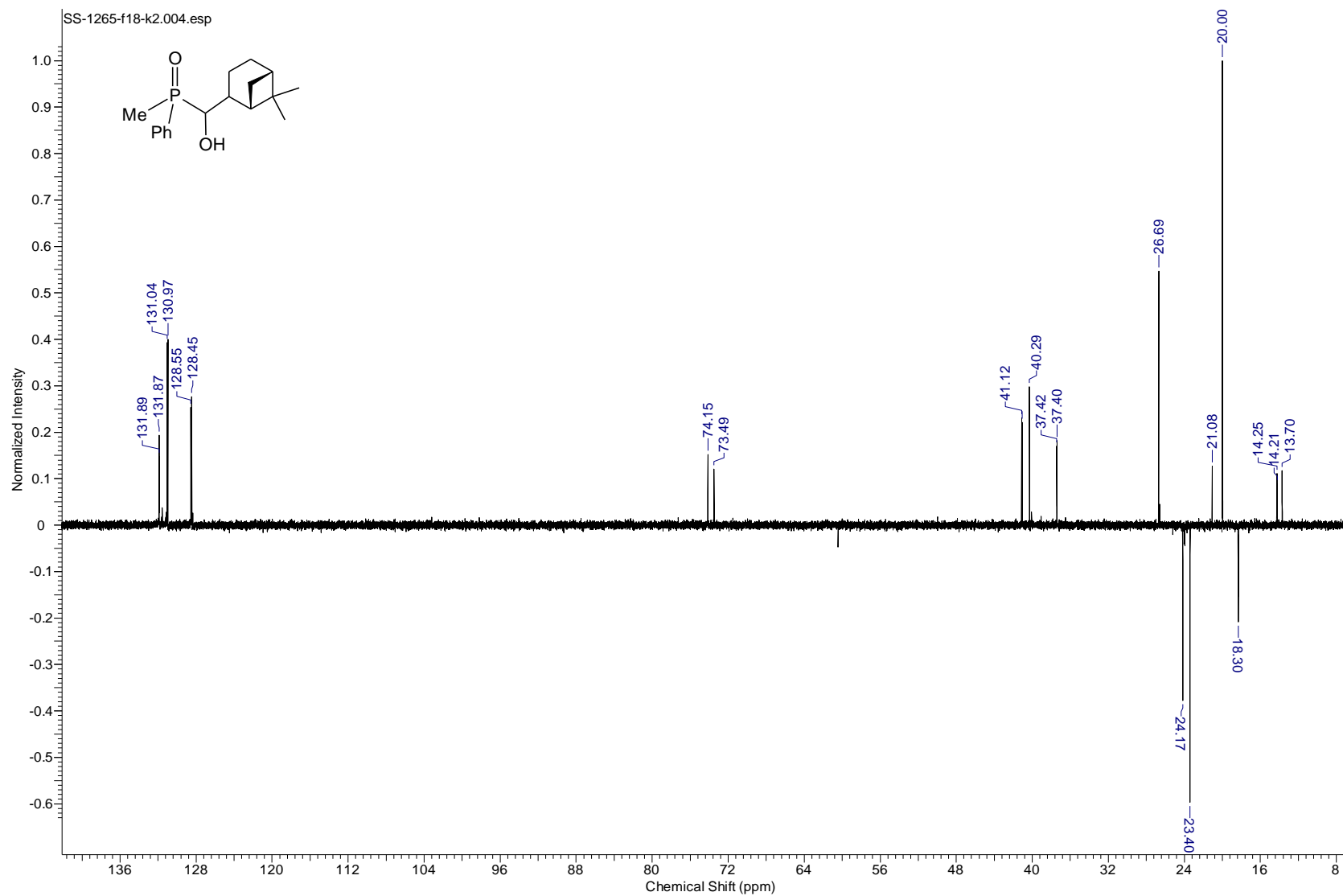
$^1\text{H}$  NMR spectrum of 6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(methyl)(phenyl)phosphine oxide **9c-IV** (500 MHz,  $\text{CDCl}_3$ )



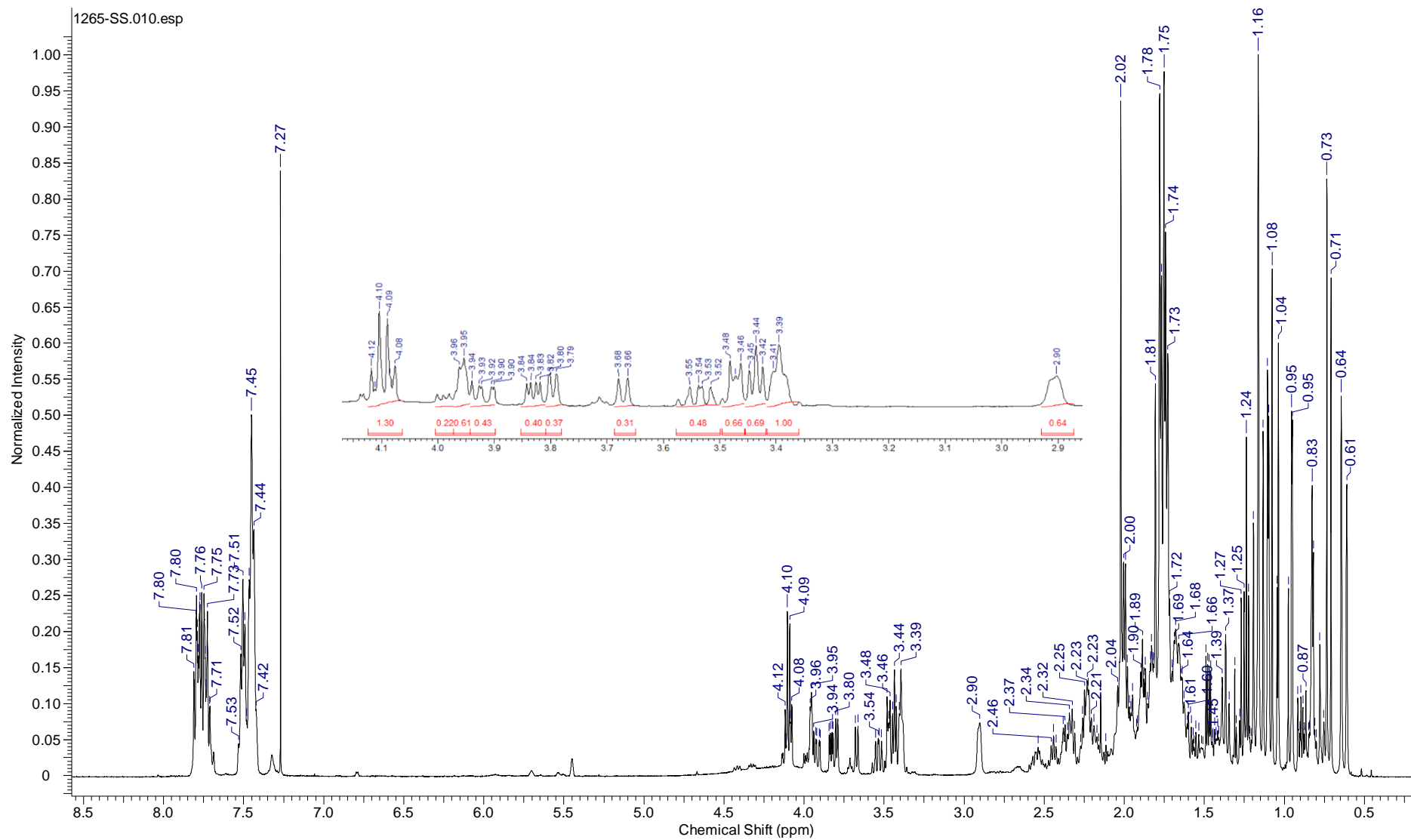
$^{31}\text{P}$  NMR spectrum of 6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(methyl)(phenyl)phosphine oxide **9c-IV** (202 MHz,  $\text{CDCl}_3$ )



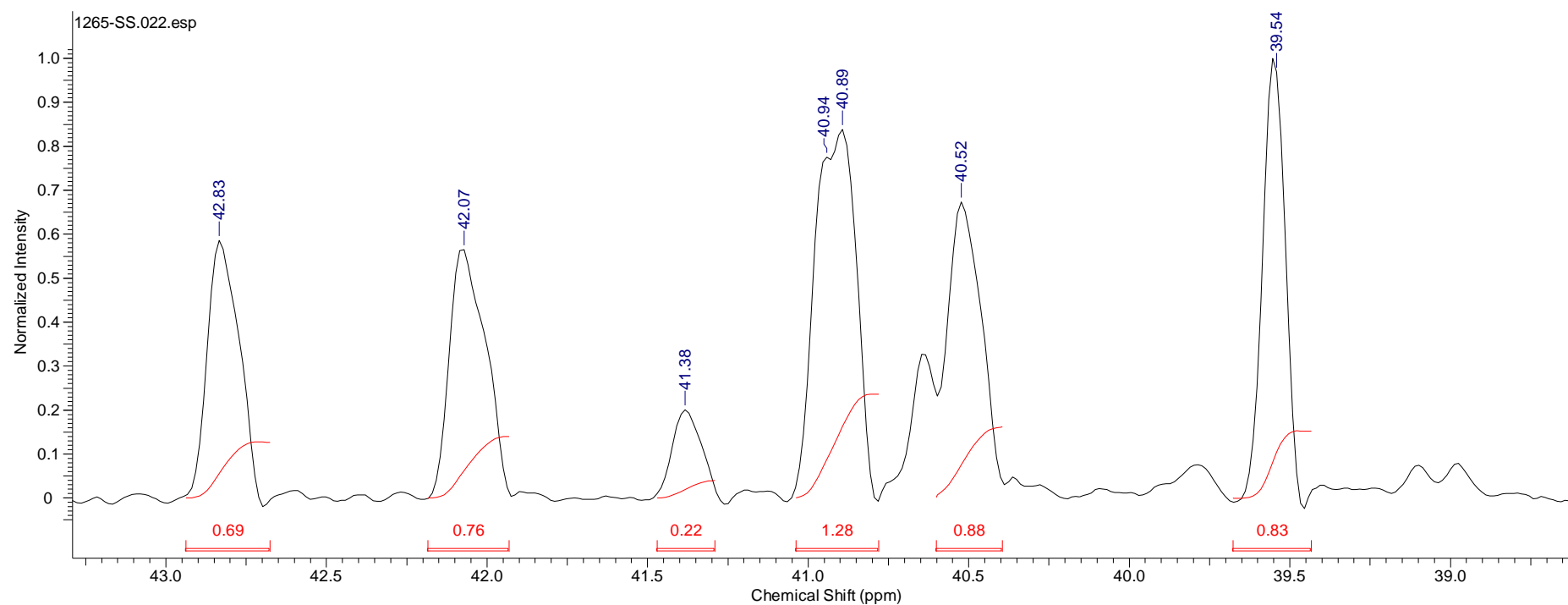
$^{13}\text{C}$  NMR spectrum of 6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(methyl)(phenyl)phosphine oxide **9c-IV** (125 MHz,  $\text{CDCl}_3$ )



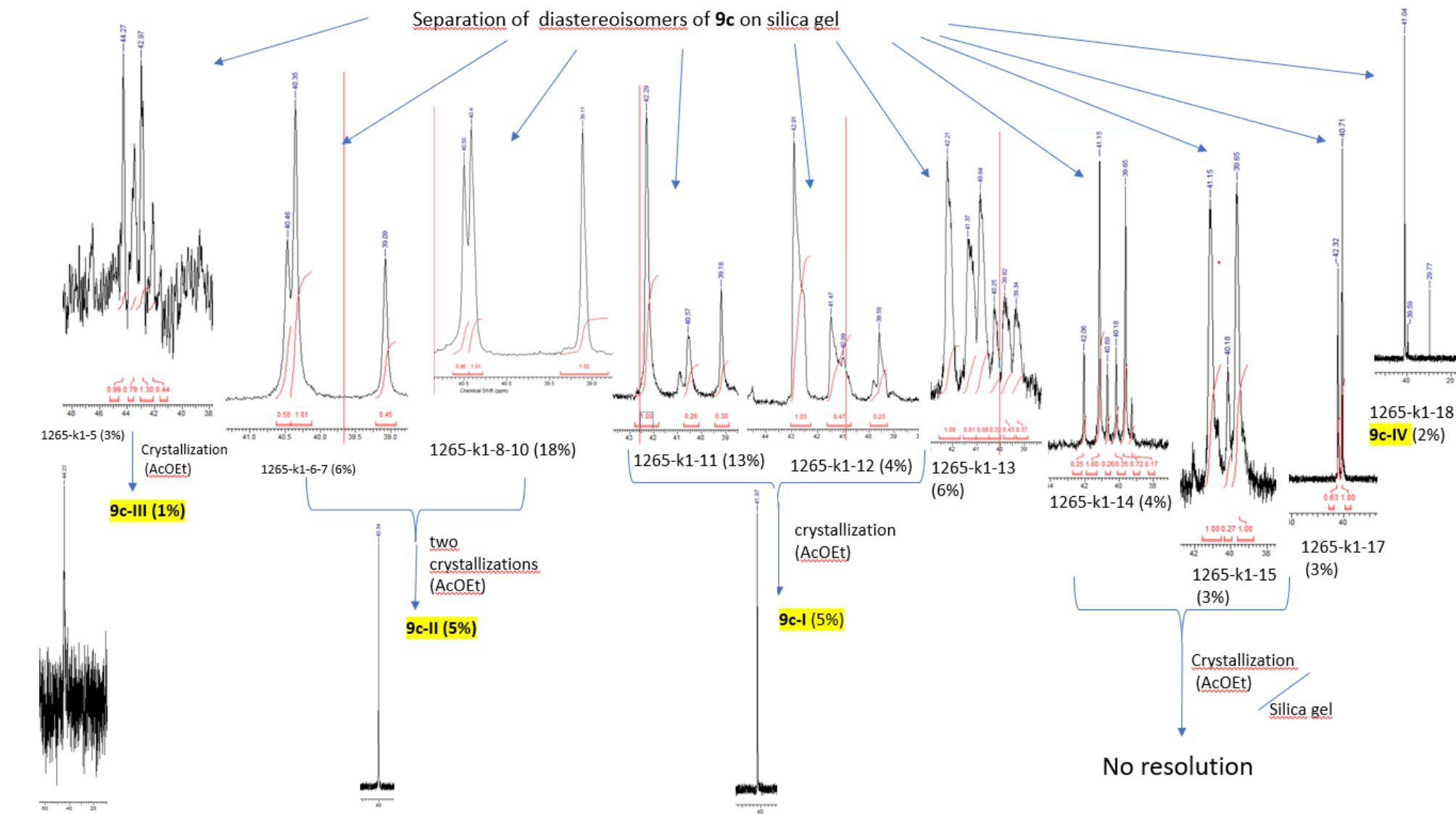
DEPT 135 NMR spectrum of 6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(methyl)(phenyl)phosphine oxide **9c-IV** (125 MHz, CDCl<sub>3</sub>)



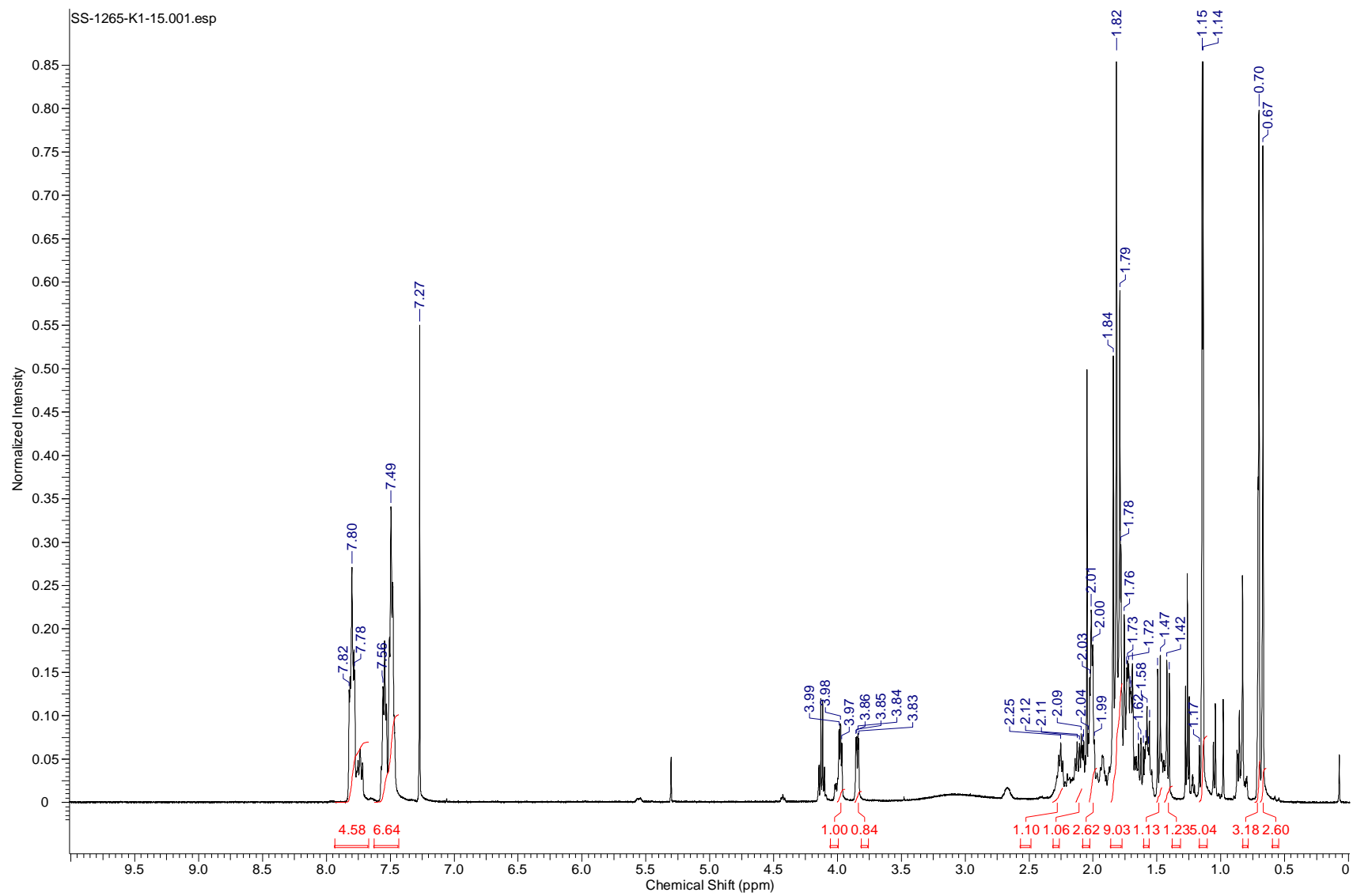
$^1\text{H}$  NMR spectrum of the crude mixture of **9c** (mixture of diastereoisomers) (500 MHz,  $\text{CDCl}_3$ )



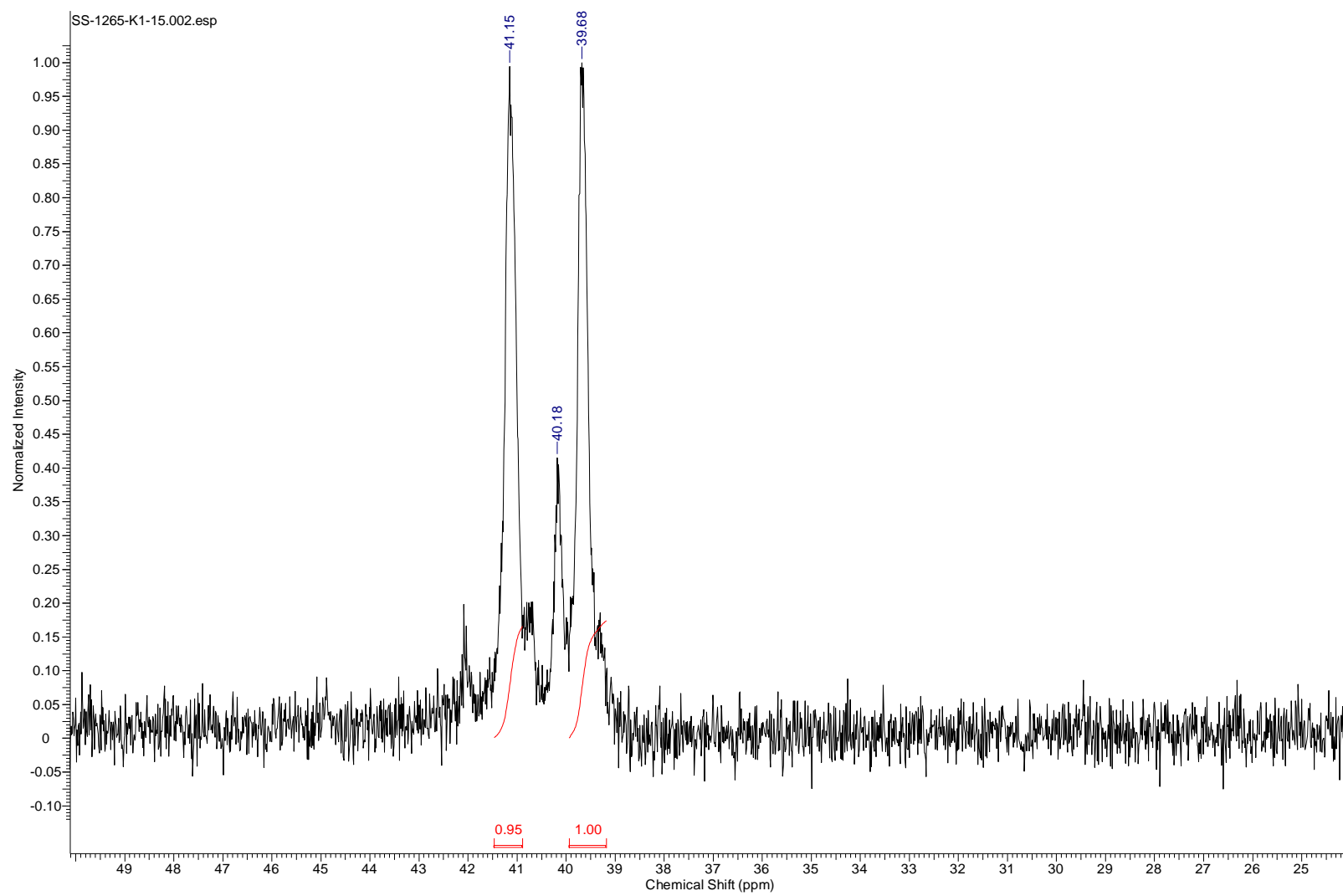
$^{31}\text{P}$  NMR spectrum of the crude mixture of **9c** (mixture of diastereoisomers) (202 MHz,  $\text{CDCl}_3$ )



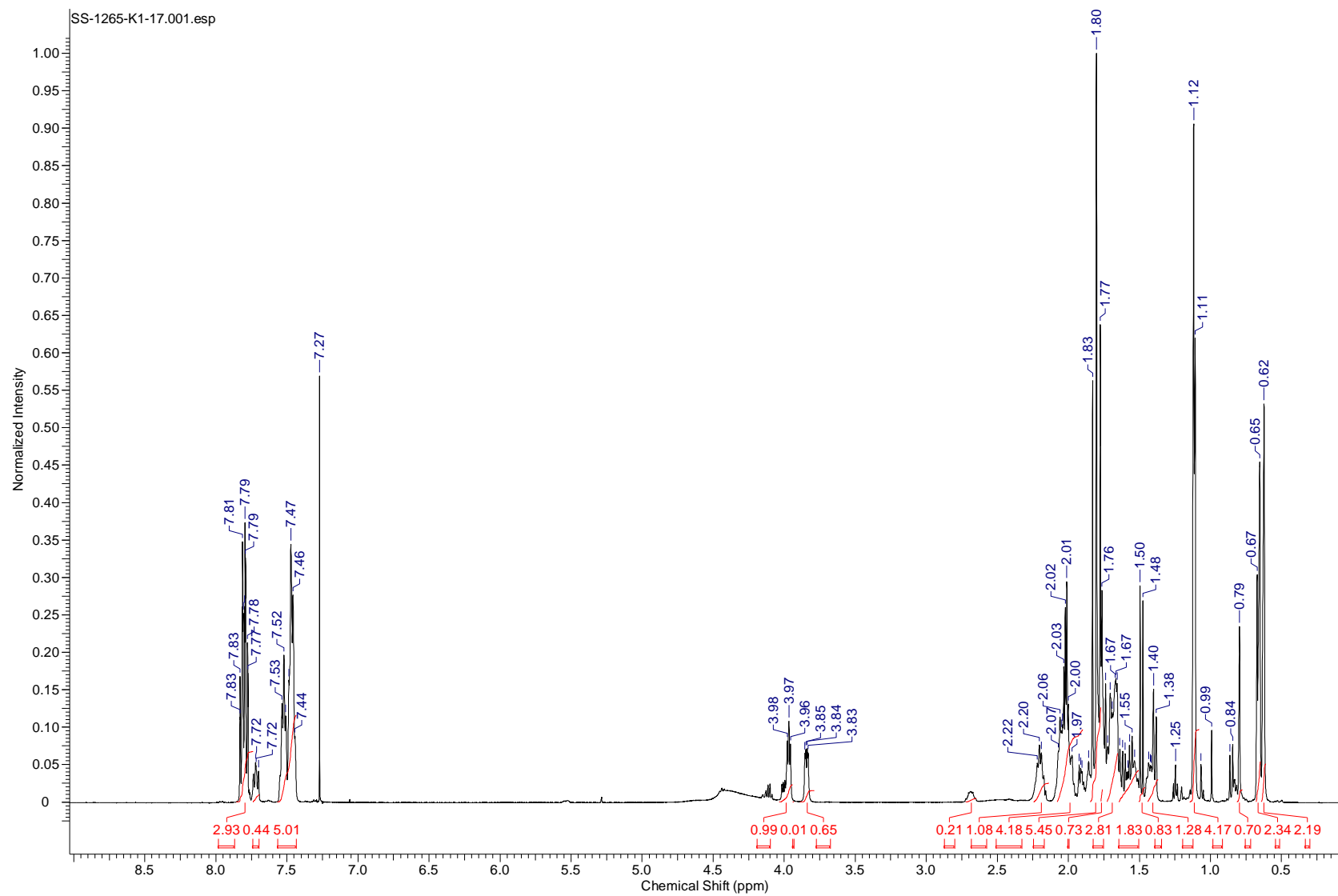
**Scheme S1.** Separation of diastereoisomers of **9c** on silica gel.



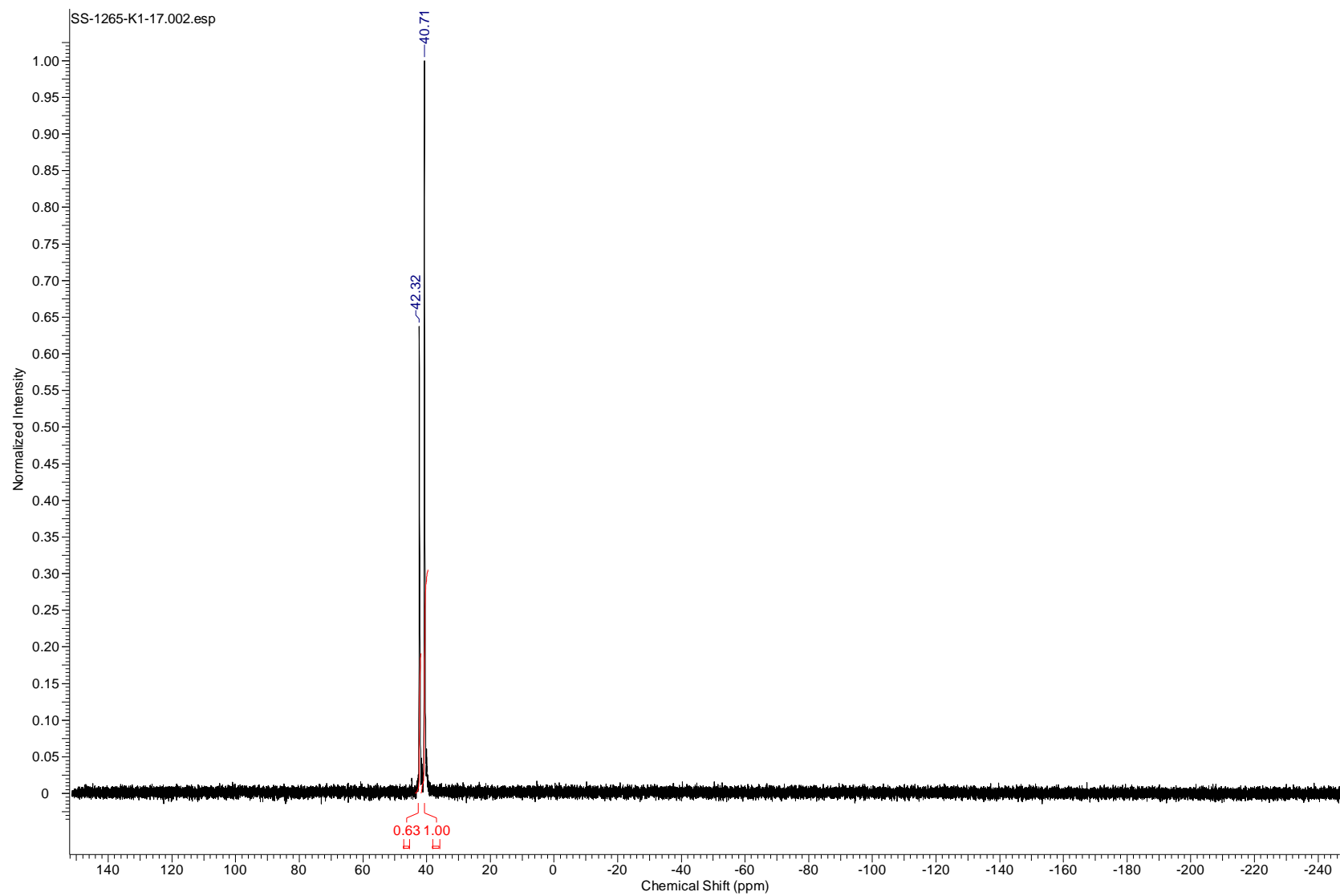
$^1\text{H}$  NMR spectrum of the fraction (1265-k1-15) of **9c** (two diastereoisomers) (500 MHz,  $\text{CDCl}_3$ )



$^{31}\text{P}$  NMR spectrum of the fraction (1265-k1-15) of **9c** (two diastereoisomers) (202 MHz,  $\text{CDCl}_3$ )

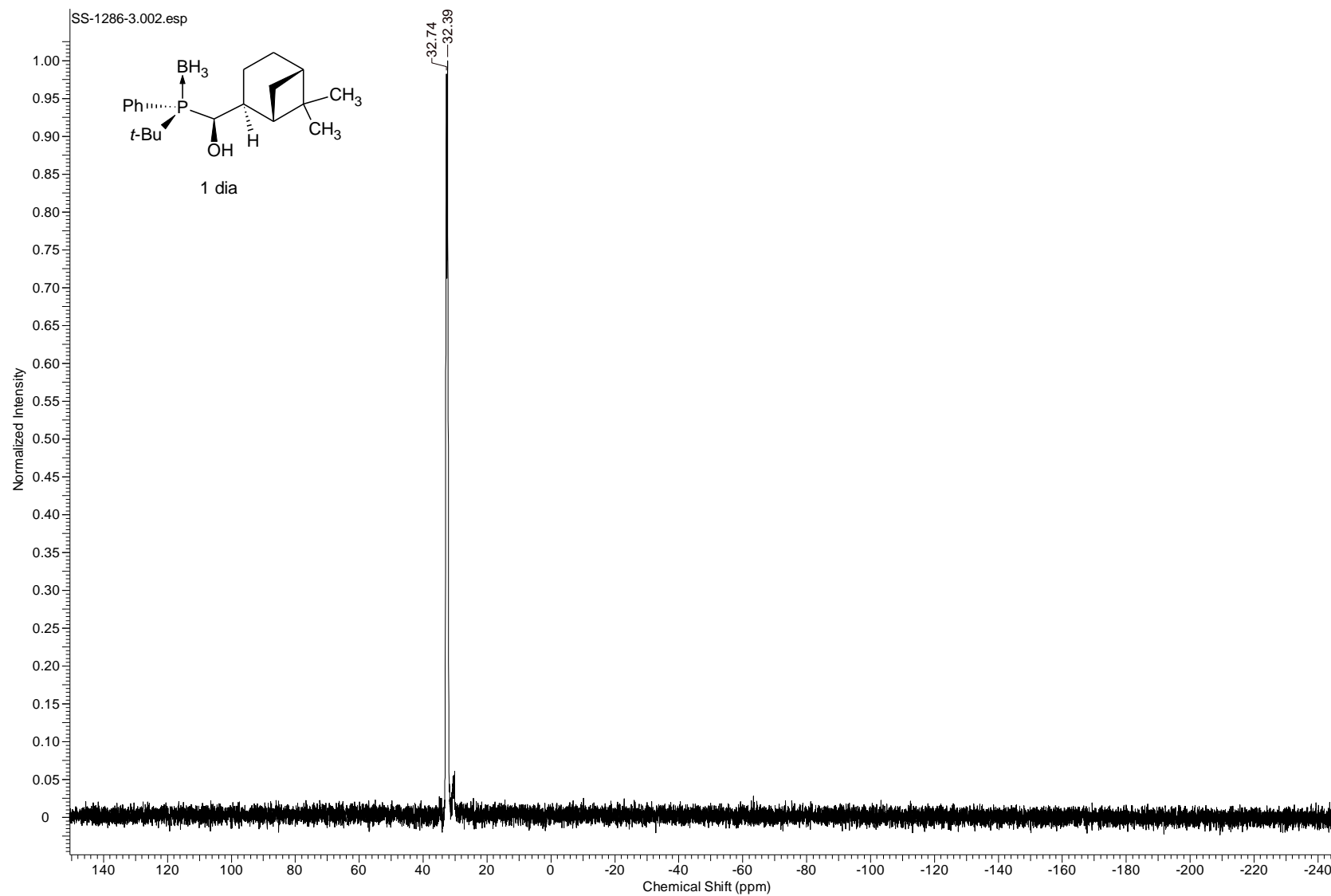


$^1\text{H}$  NMR spectrum of the fraction (1265-k1-17) of **9c** (two diastereoisomers) (500 MHz,  $\text{CDCl}_3$ )

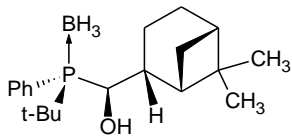


$^{31}\text{P}$  NMR spectrum of the fraction (1265-k1-17) of **9c** (two diastereoisomers) (202 MHz,  $\text{CDCl}_3$ )

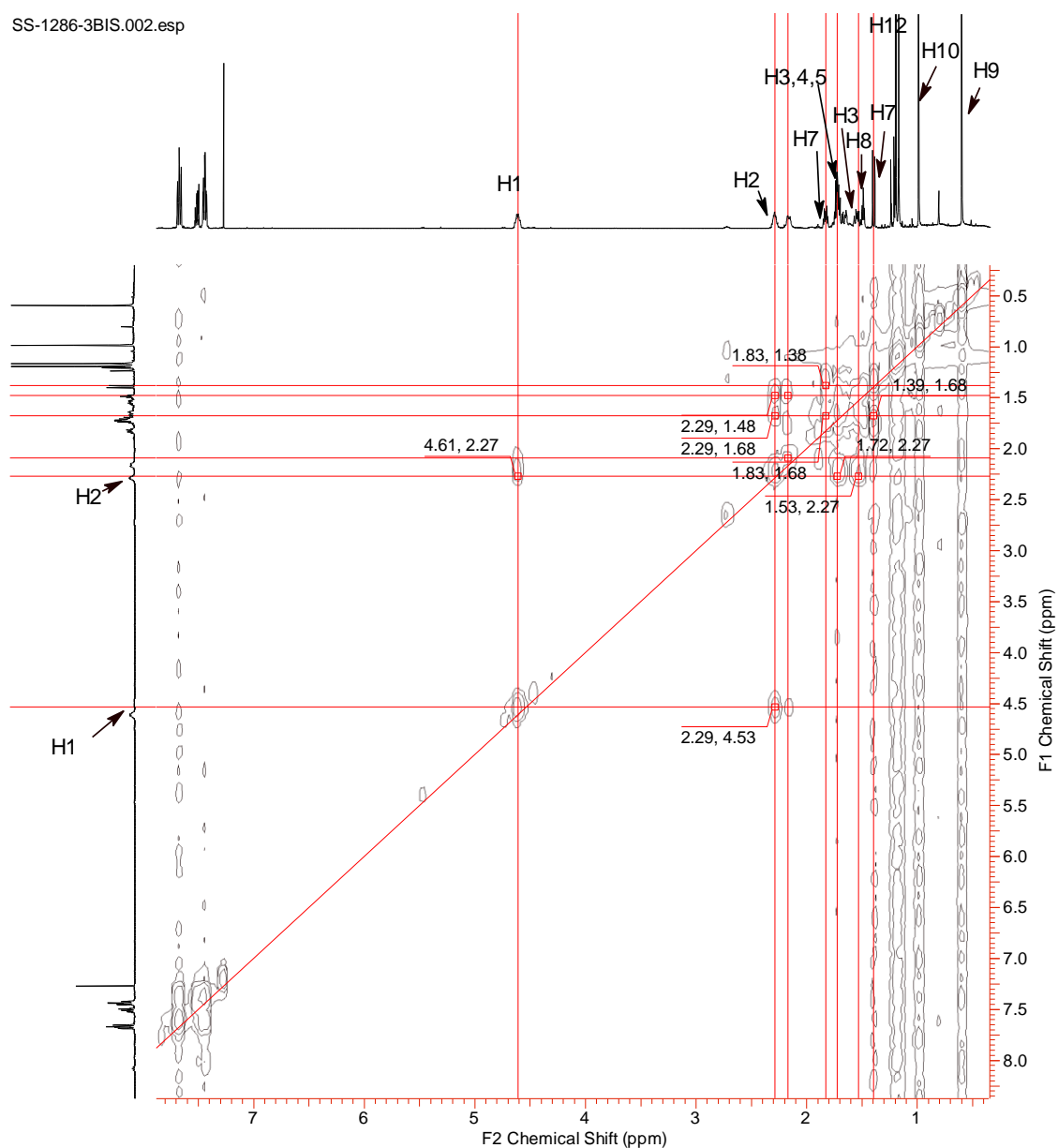




$^{31}\text{P}$  NMR spectrum of ( $R_P$ )-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl( $t$ -butyl)(phenyl)phosphine-borane ( $R_P$ )-**10a** (202 MHz,  $\text{CDCl}_3$ )

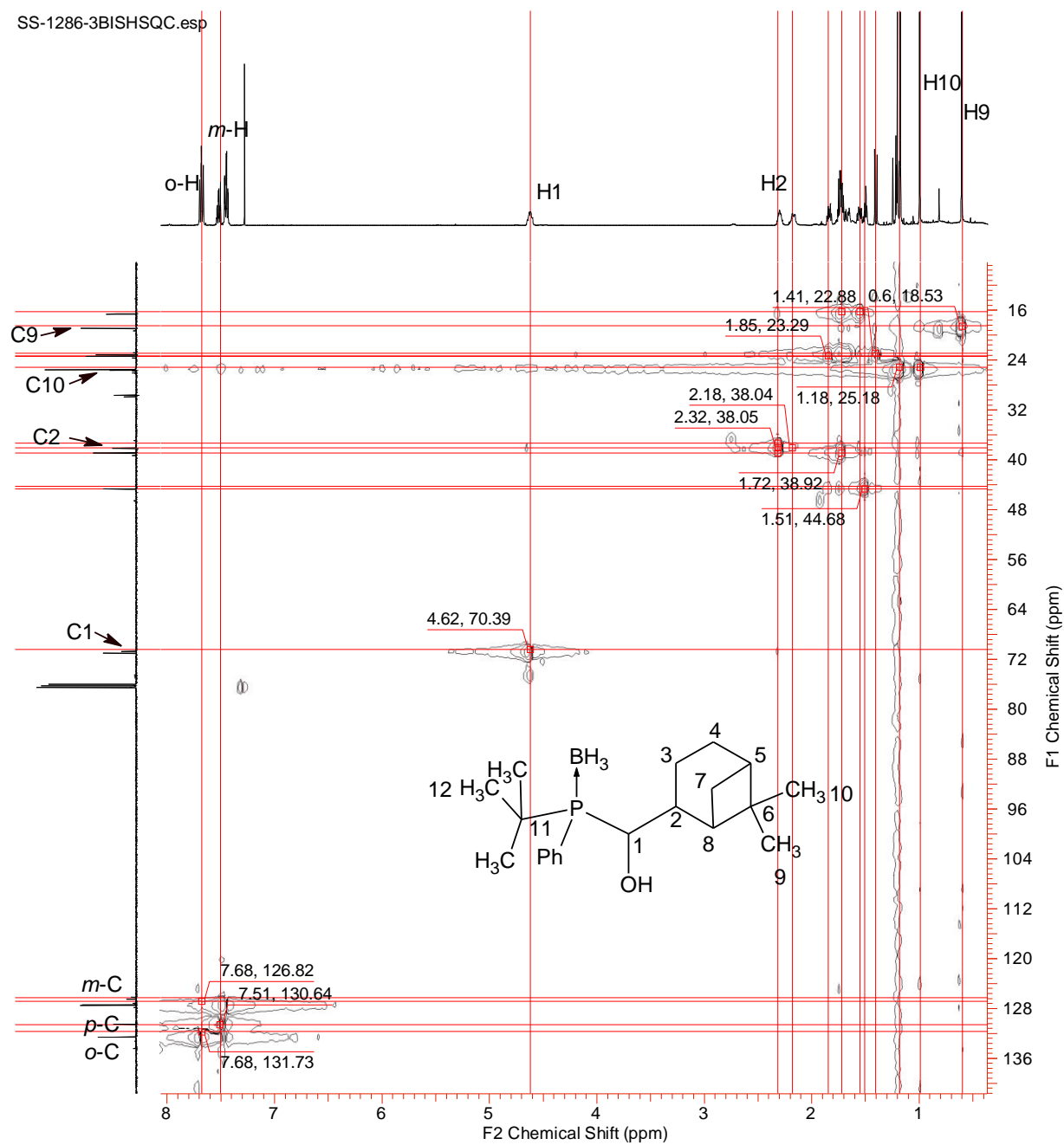


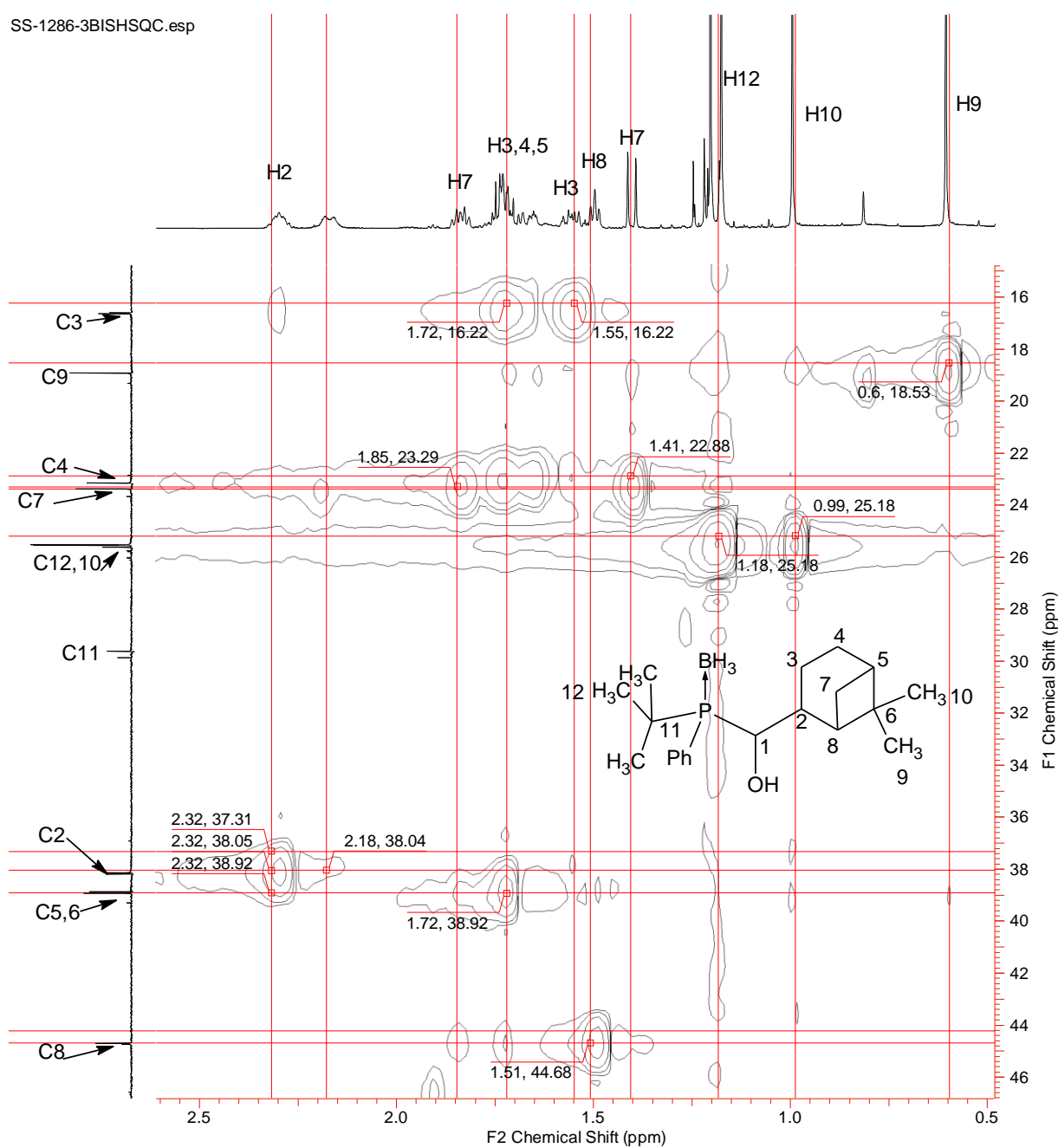
S71



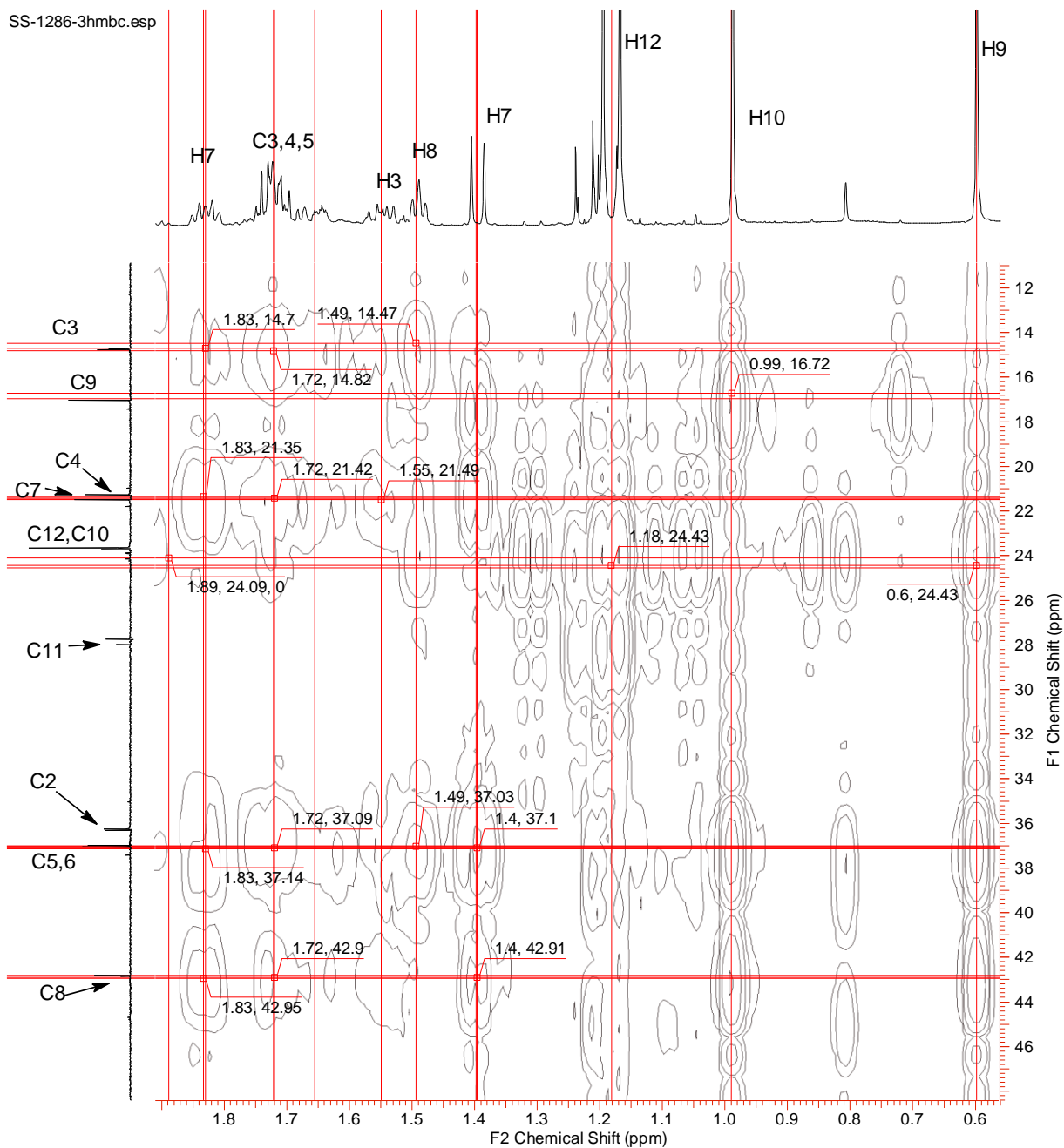
COSY spectrum of (*R<sub>P</sub>*)-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(*t*-butyl)(phenyl)phosphine-borane (*R<sub>P</sub>*)-**10a** (500 MHz,  $\text{CDCl}_3$ ), see Figure 11 in the ms

SS-1286-3BISHSQC.esp

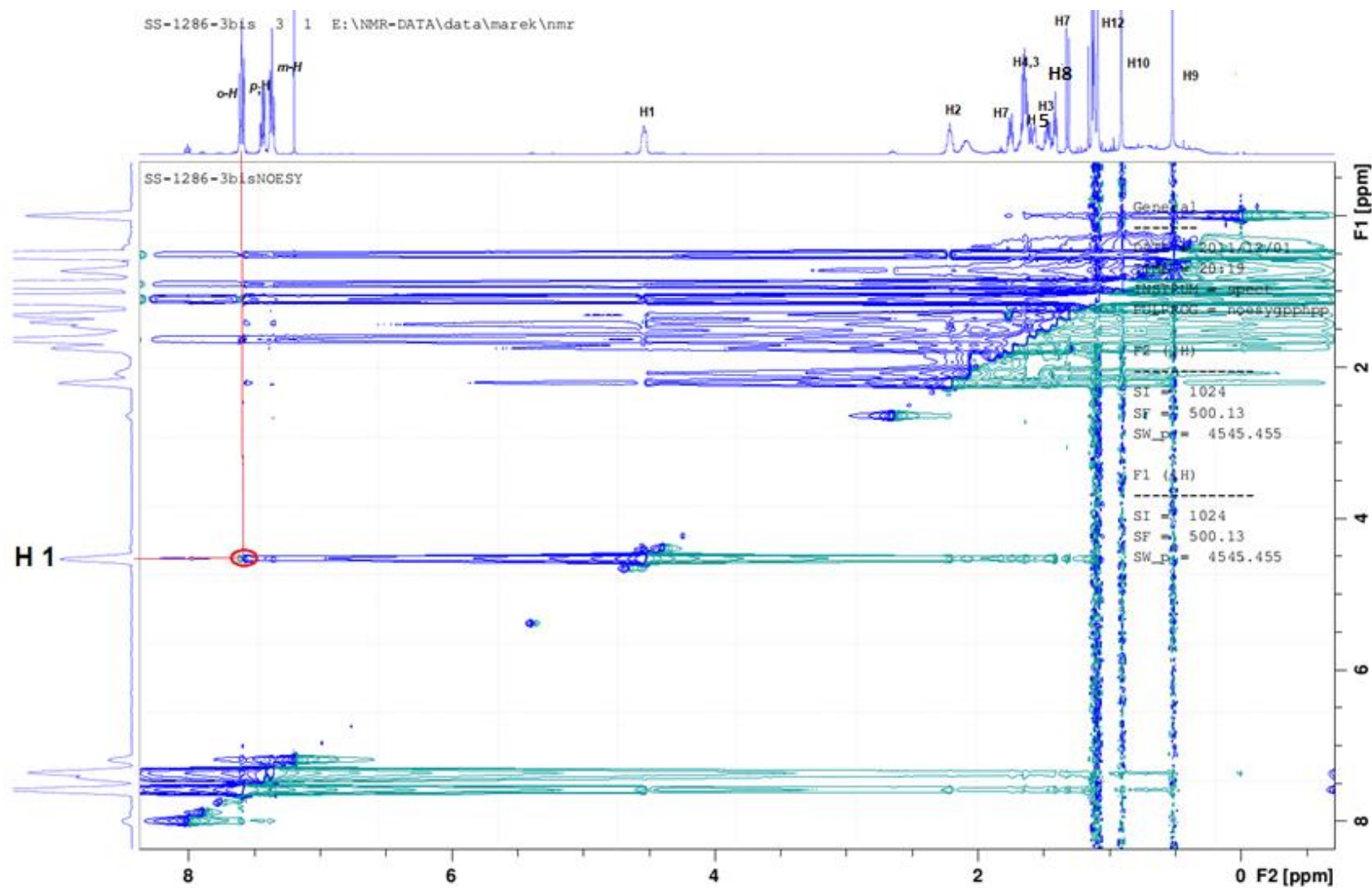




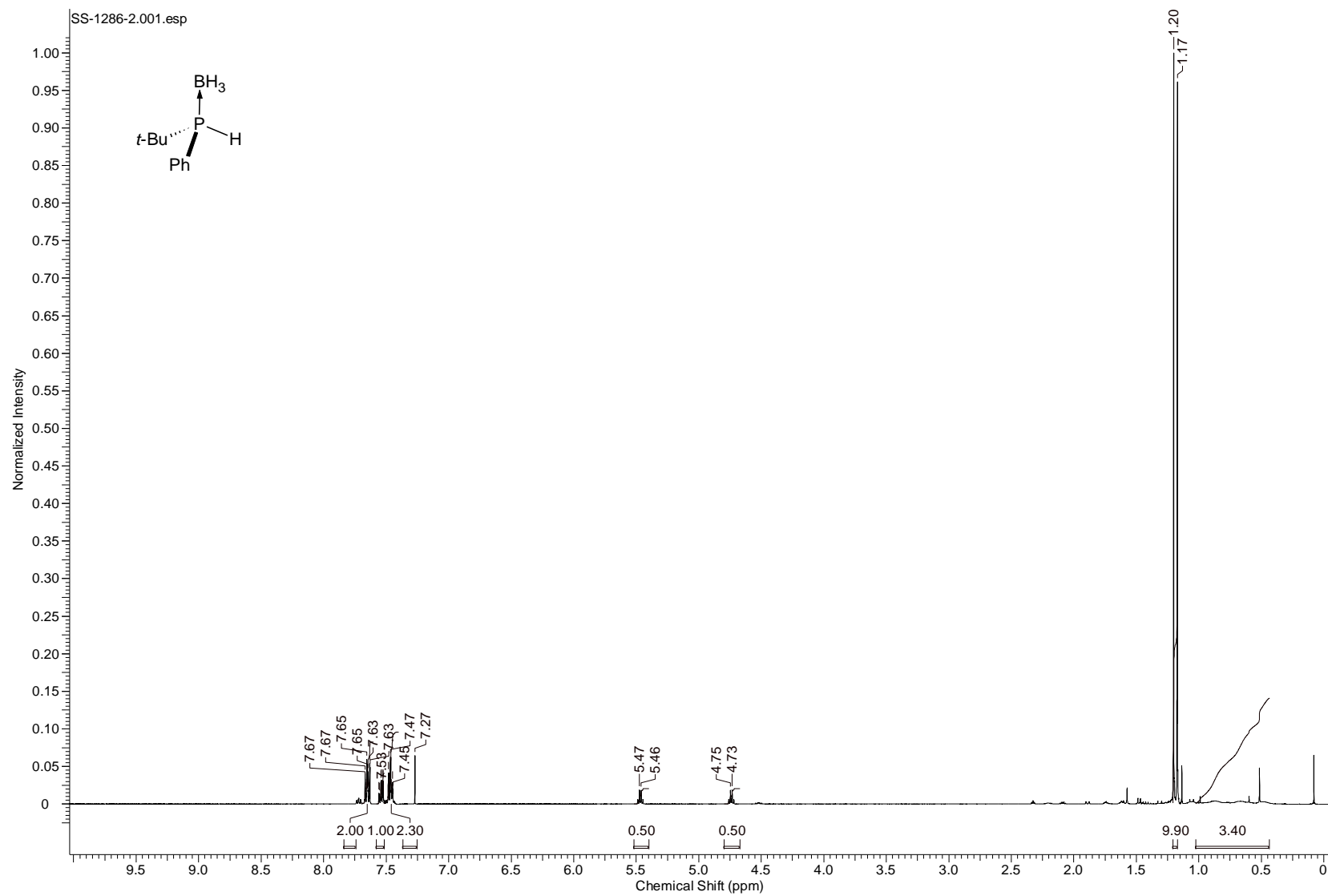




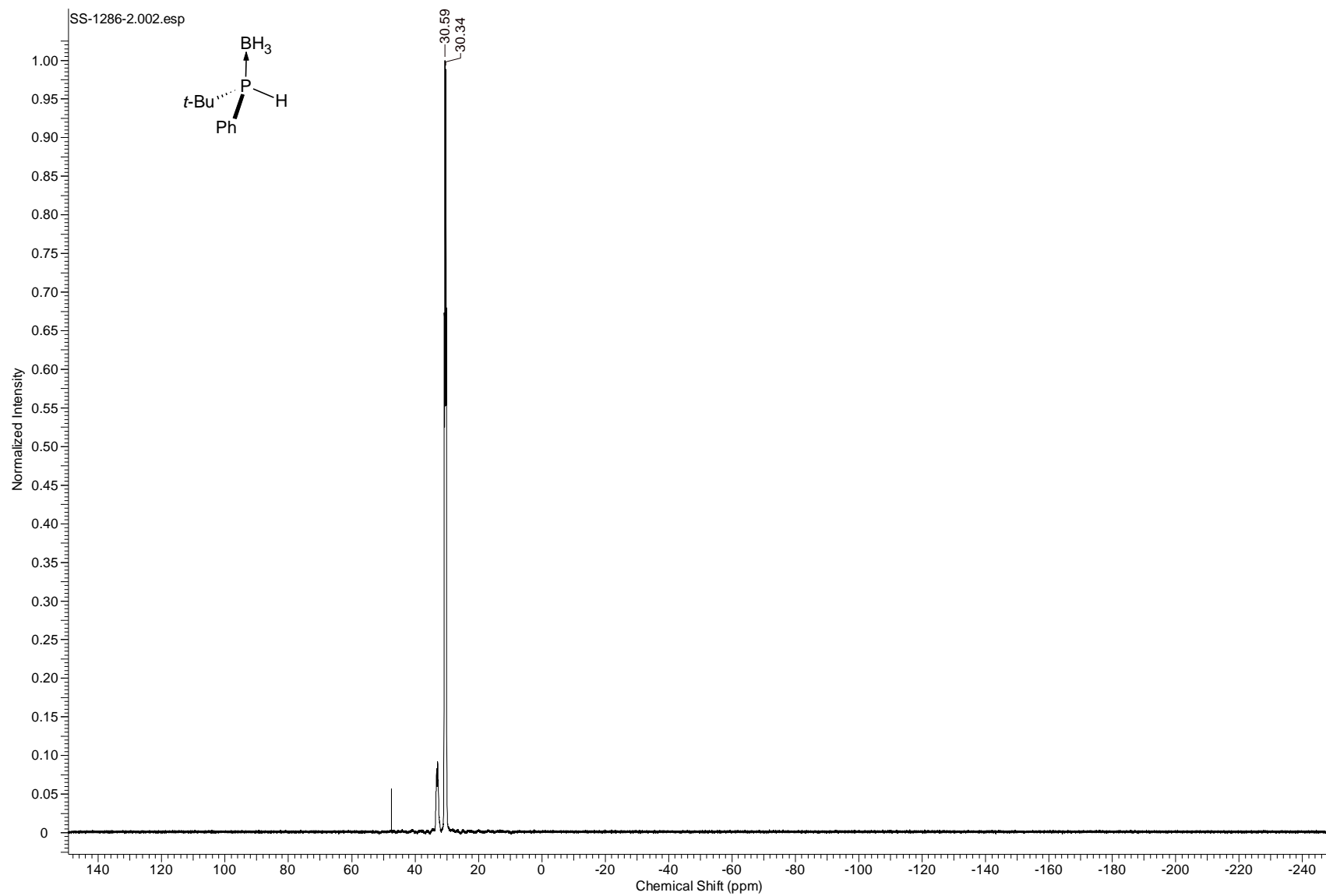
HMBC spectrum of (*R<sub>P</sub>*)-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(*t*-butyl)(phenyl)phosphine-borane (*R<sub>P</sub>*)-**10a** (500, 125 MHz, CDCl<sub>3</sub>)



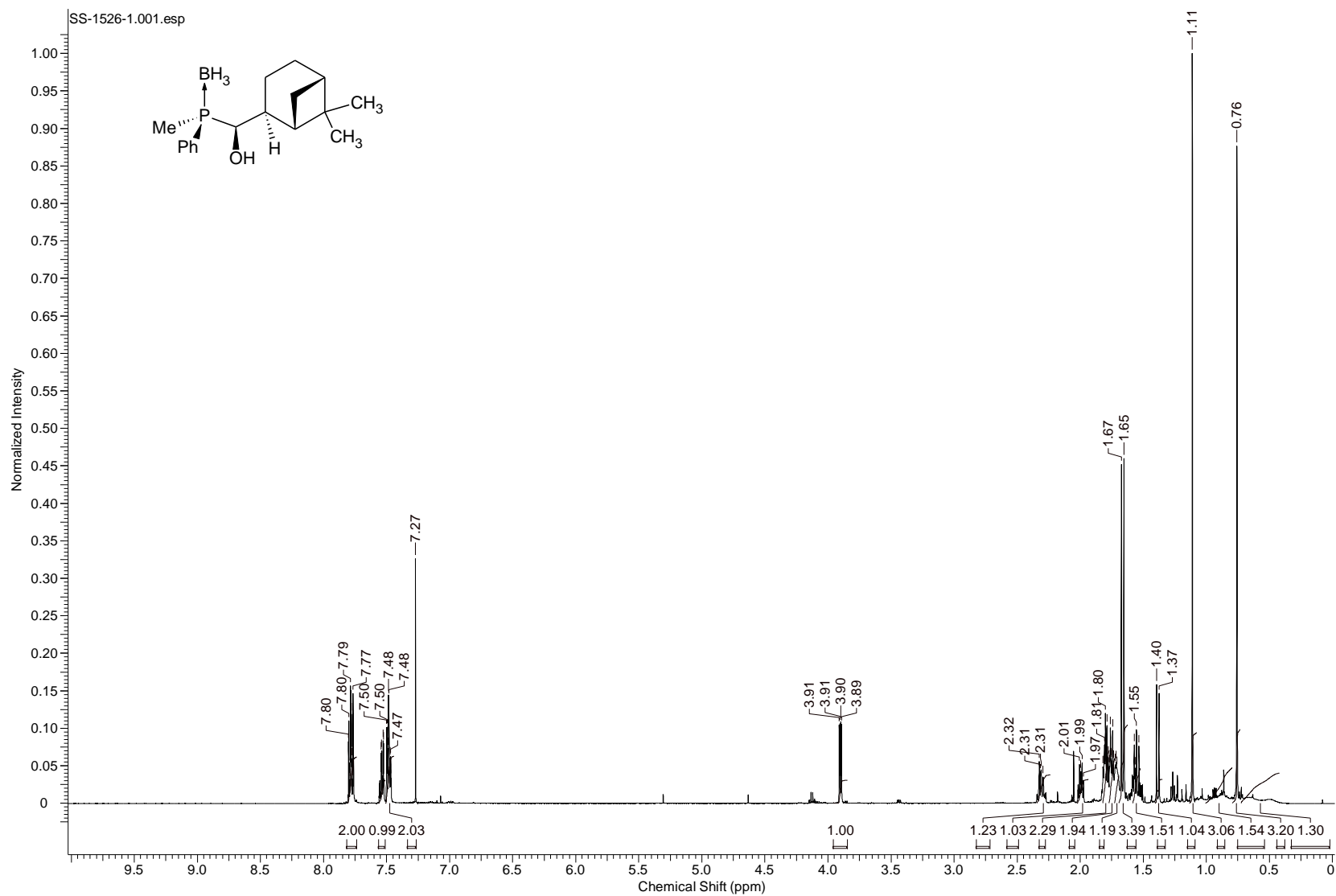
NOESY spectrum of (*R<sub>P</sub>*)-6,6-dimethylbicyclo[3.1.1]heptan-2-yl(hydroxy)methyl(*t*-butyl)(phenyl)phosphine-borane (*R<sub>P</sub>*)-**10a** (500 MHz, CDCl<sub>3</sub>)



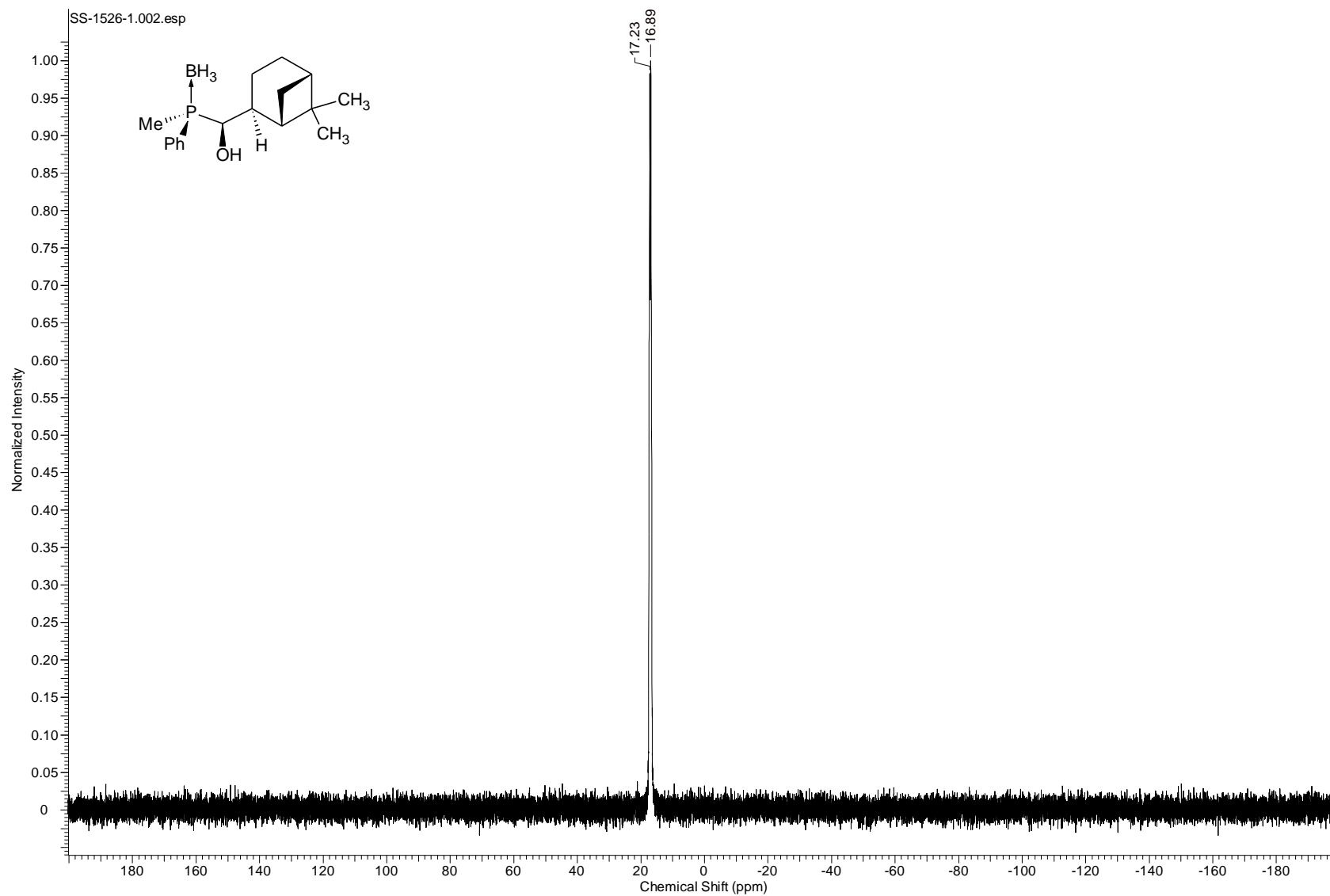
$^1H$  NMR spectrum of  $(R_P)$ -(-)-*t*-butylphenylphosphine-borane ( $R_P$ )-(-)-**11a** (500 MHz,  $CDCl_3$ ) [4]



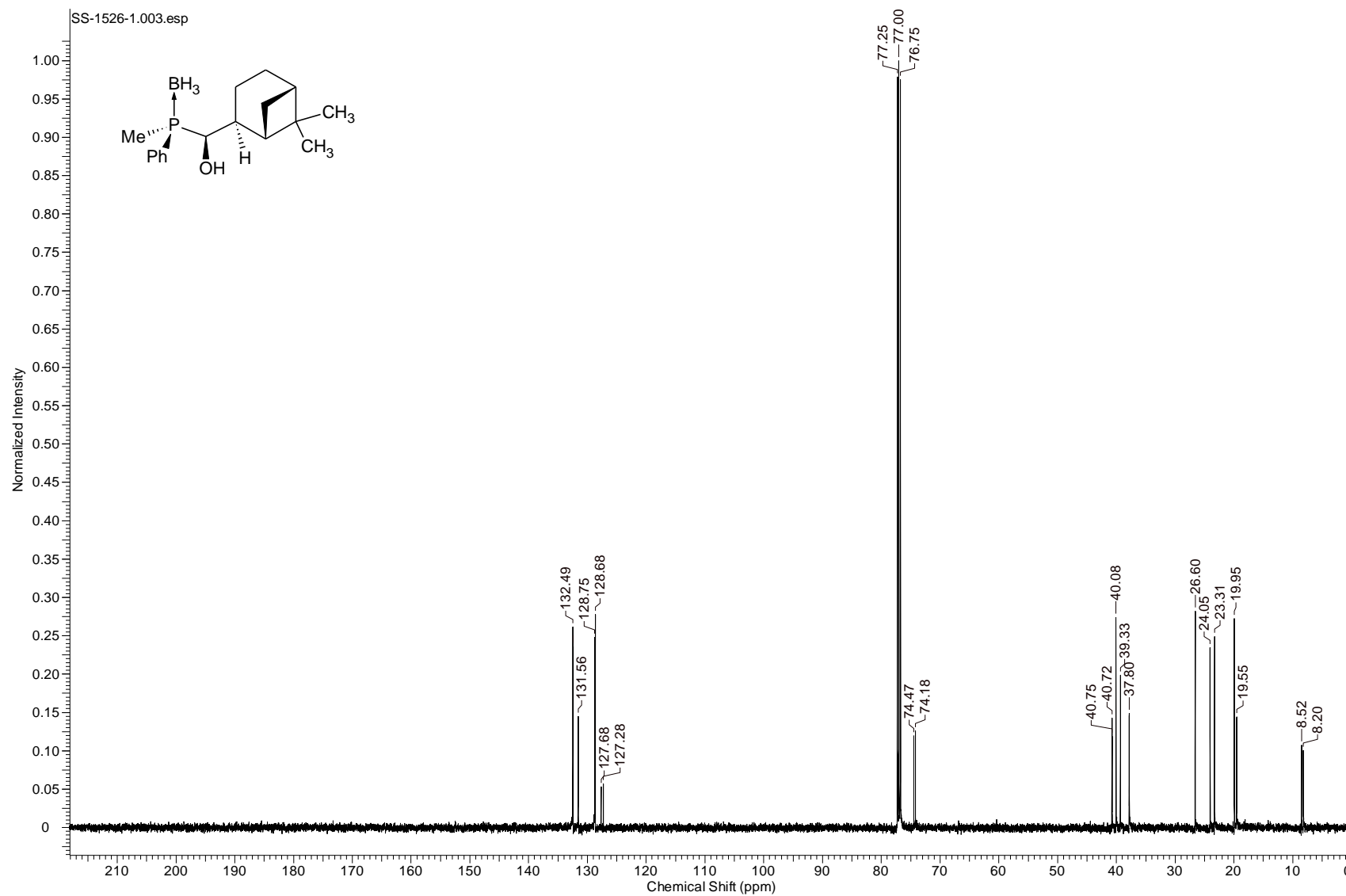
$^{31}\text{P}$  NMR spectrum of  $(R_P)$ -(-)-*t*-butylphenylphosphine-borane ( $R_P$ )-(-)-**11a** (202 MHz,  $\text{CDCl}_3$ ) [4]



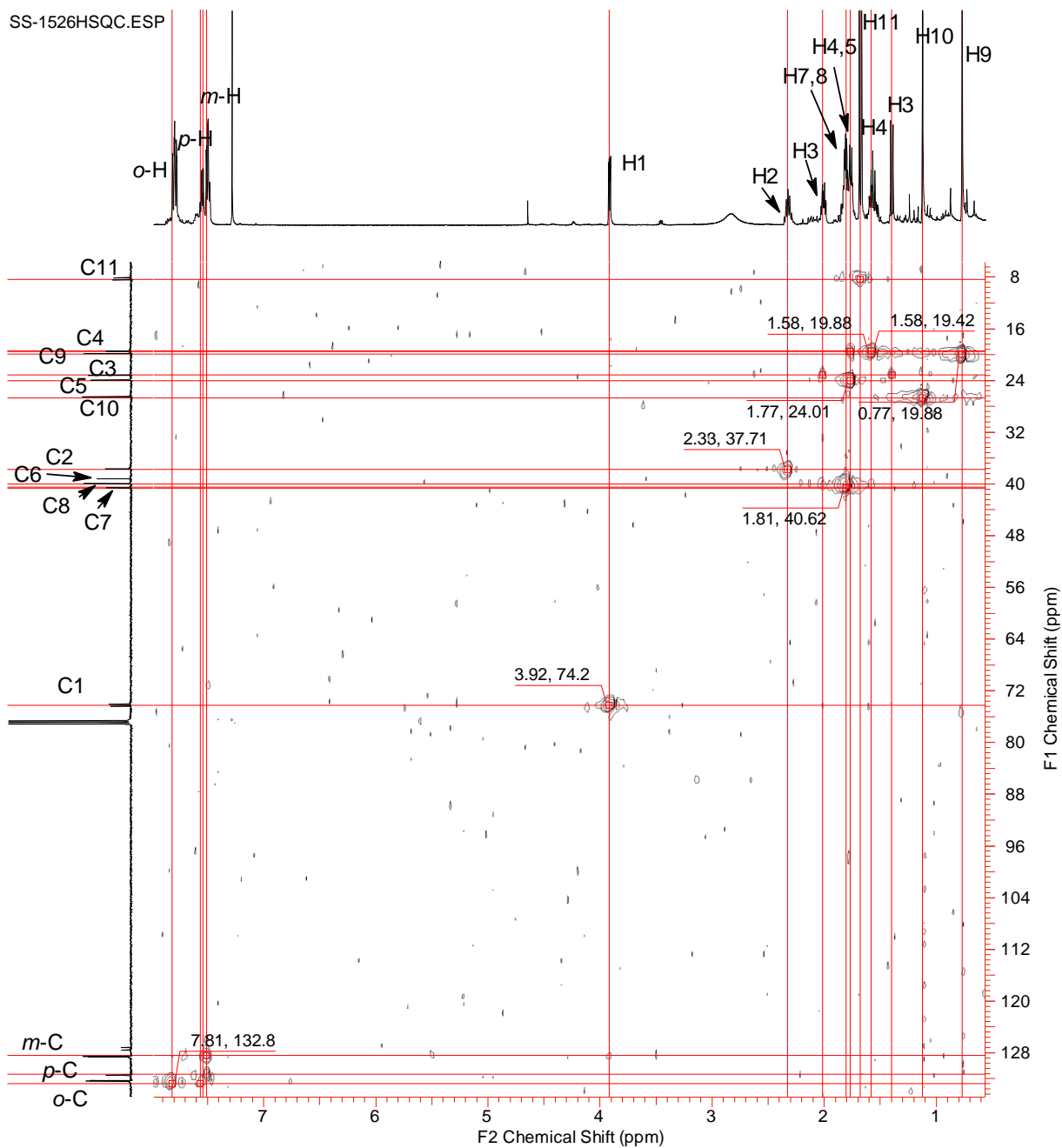
<sup>1</sup>H NMR spectrum of (*S\_P*)-(6,6-dimethylbicyclo[3.1.1]heptan-2-yl)(hydroxy)methyl(methyl)(phenyl) phosphine-borane (*(S\_P)*)-**12c** (500 MHz, CDCl<sub>3</sub>)

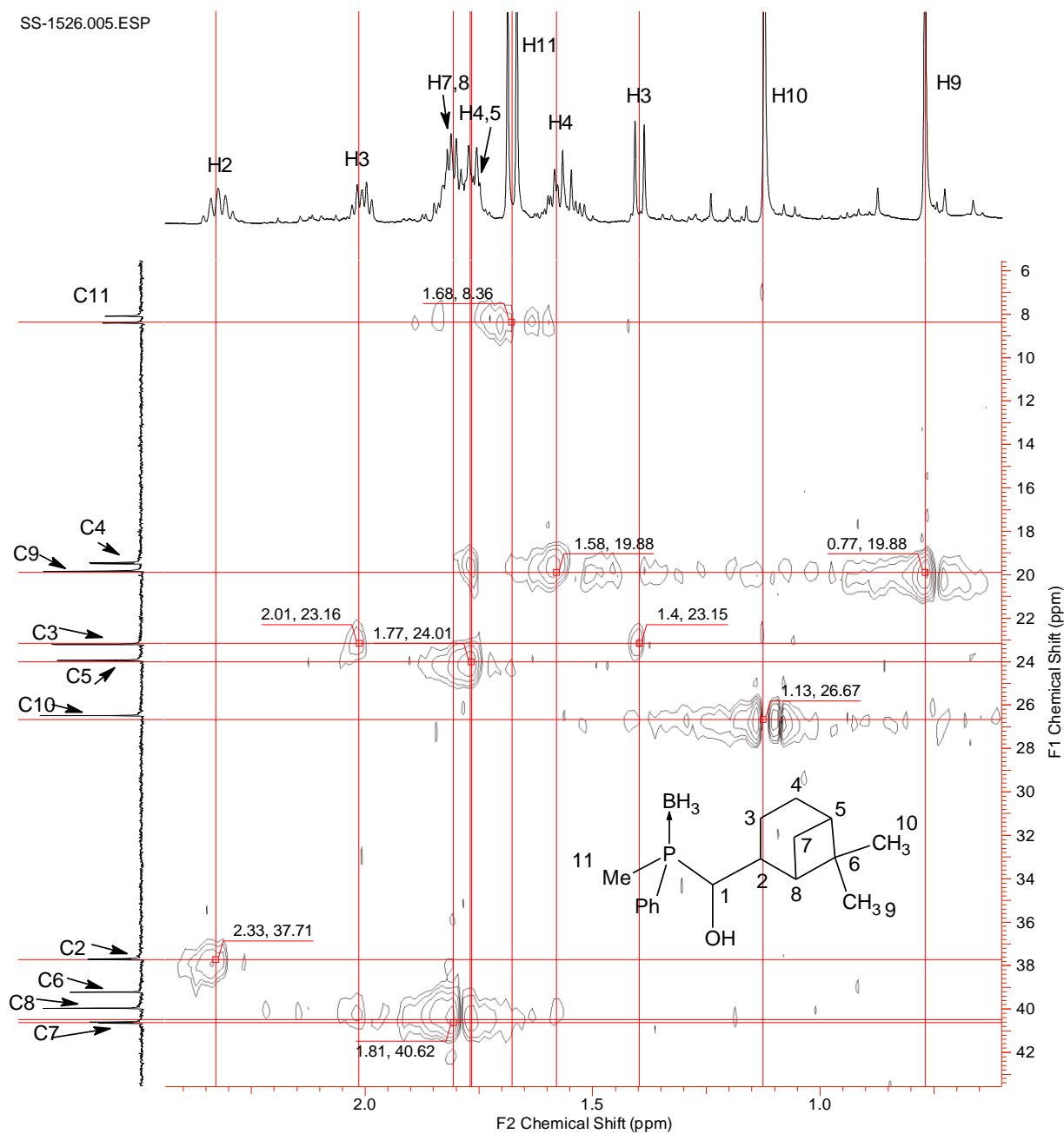


$^{31}\text{P}$  NMR spectrum of (*S<sub>P</sub>*)-(6,6-dimethylbicyclo[3.1.1]heptan-2-yl)(hydroxy)methyl(methyl)(phenyl) phosphine-borane (*S<sub>P</sub>*)-**12c** (202 MHz,  $\text{CDCl}_3$ )

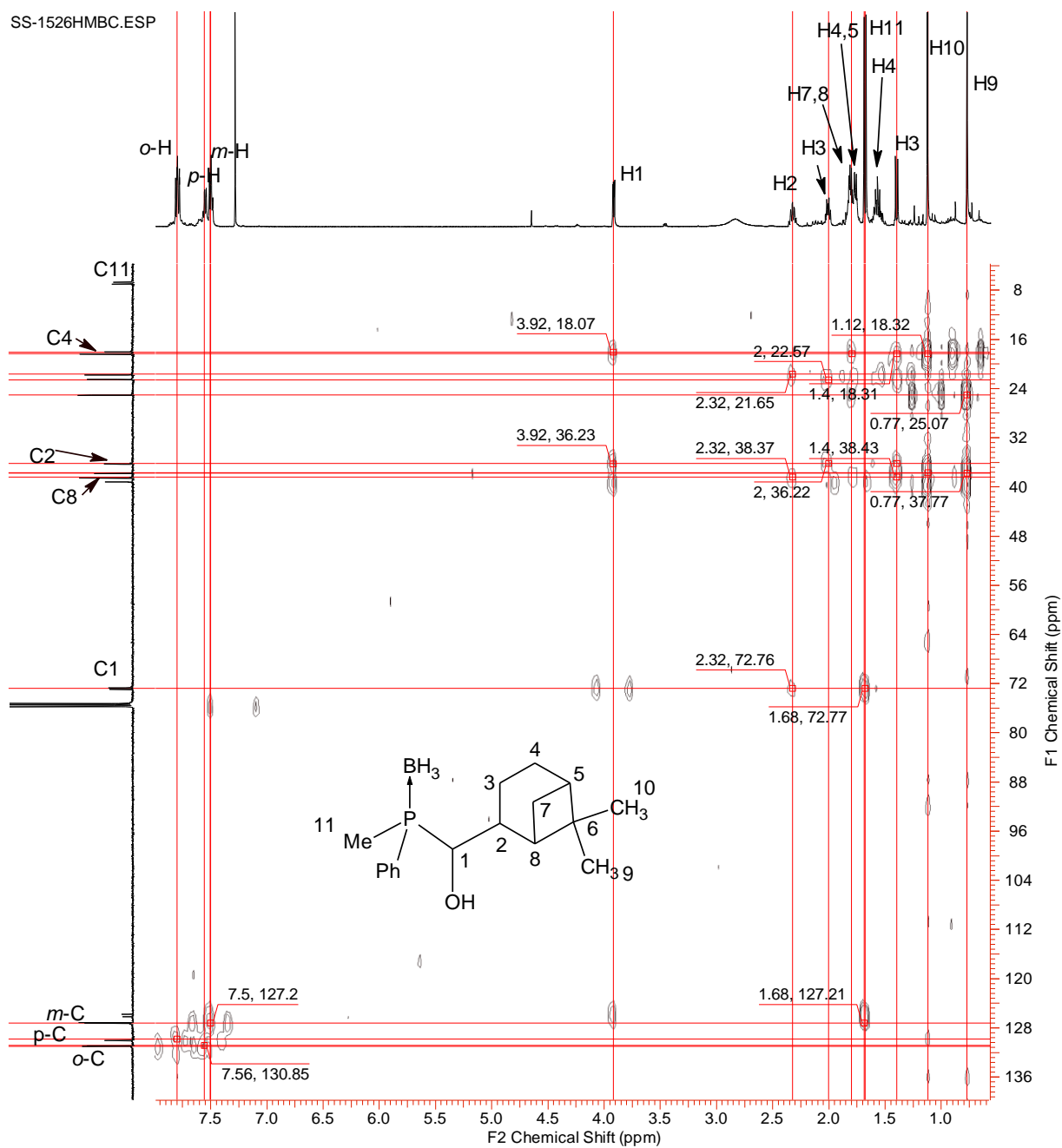


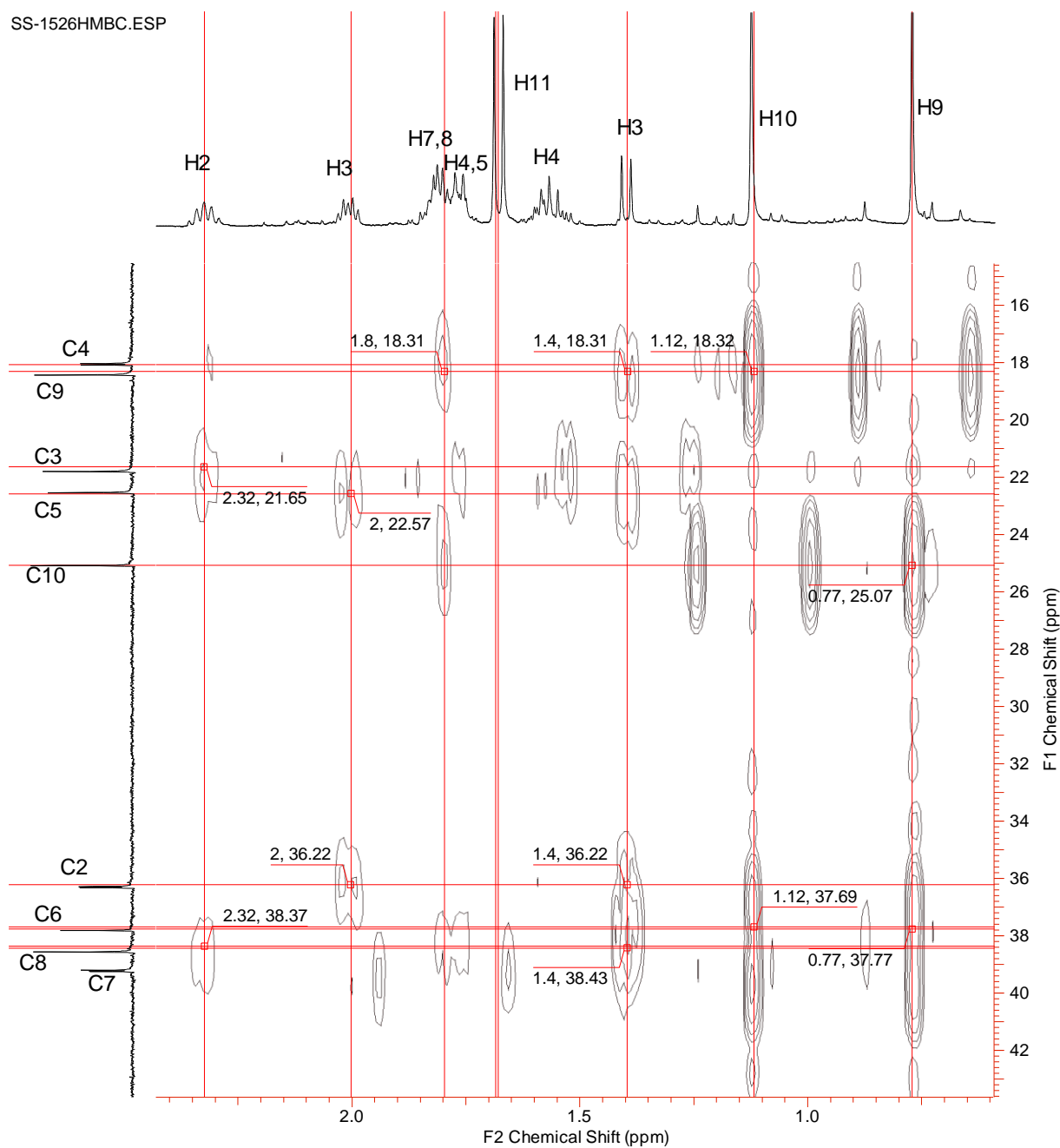
<sup>13</sup>C NMR spectrum of (*S\_P*)-(6,6-dimethylbicyclo[3.1.1]heptan-2-yl)(hydroxy)methyl(methyl)(phenyl) phosphine-borane (*S\_P*)-**12c** (125 MHz, CDCl<sub>3</sub>)



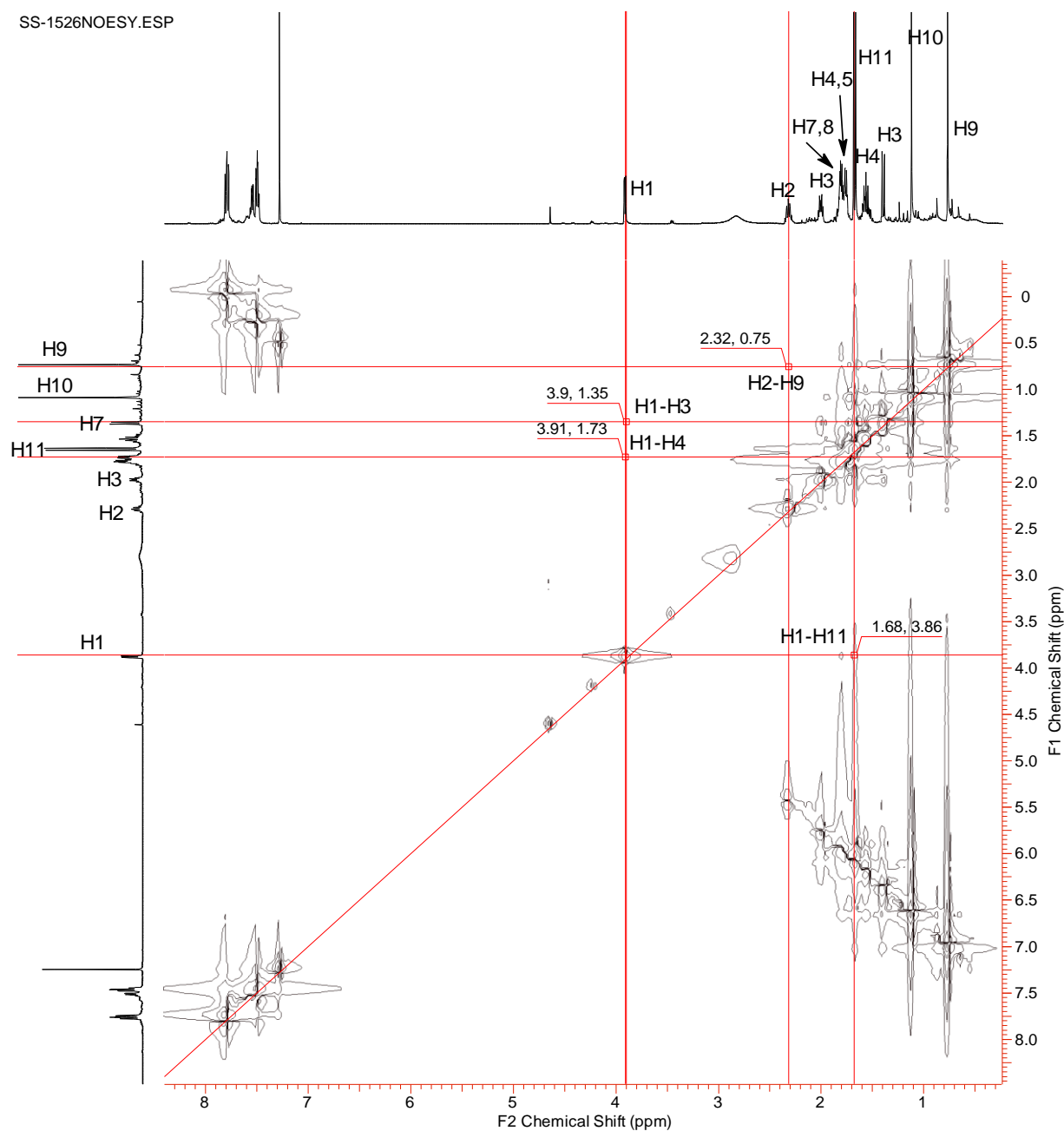


HSQC spectrum of *(S\_P)*-(6,6-dimethylbicyclo[3.1.1]heptan-2-yl)(hydroxymethyl)(methyl)(phenyl) phosphine-borane (*S\_P*)-**12c** (500, 125 MHz, CDCl<sub>3</sub>), see Figure 12 in the ms

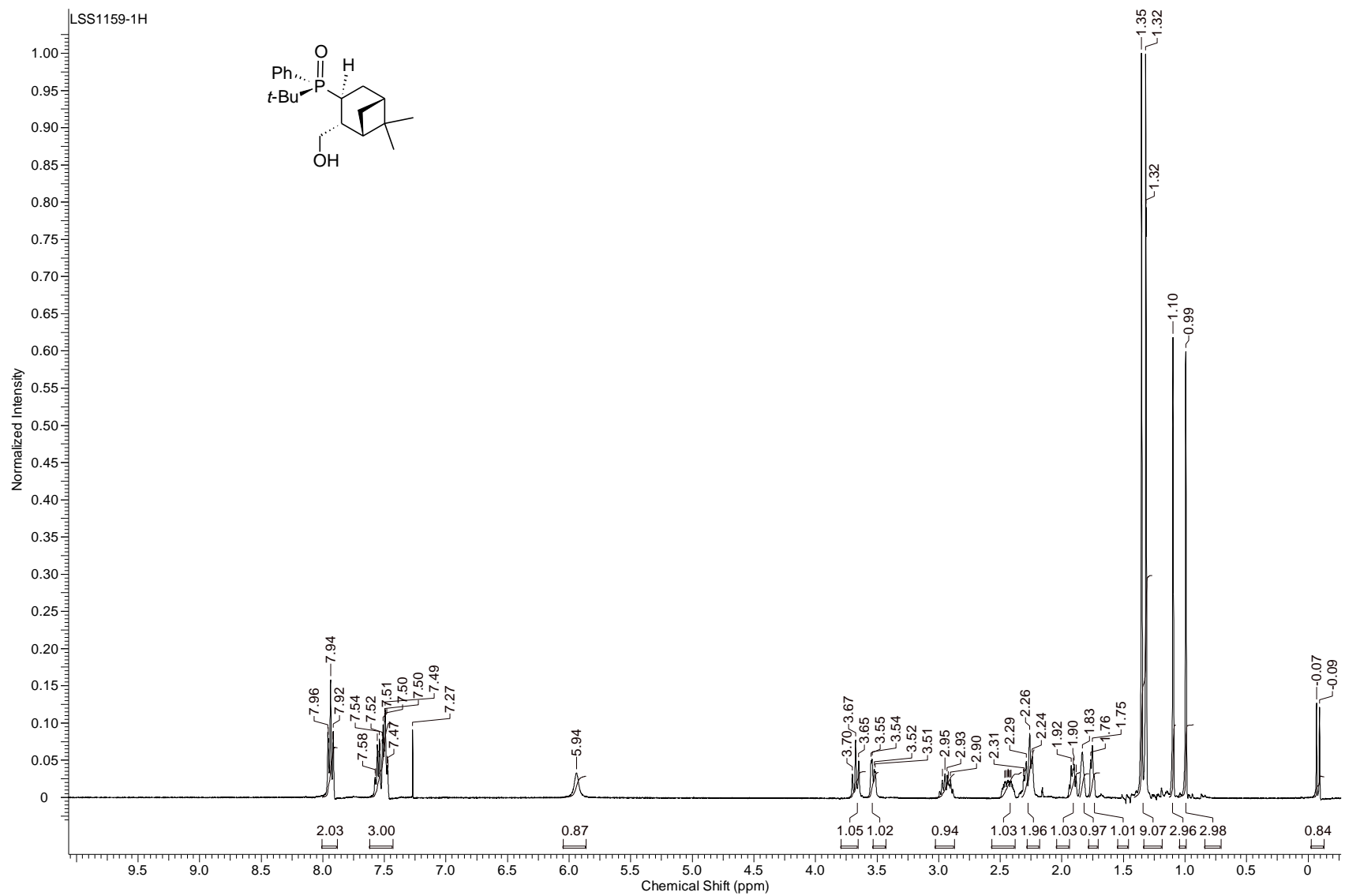




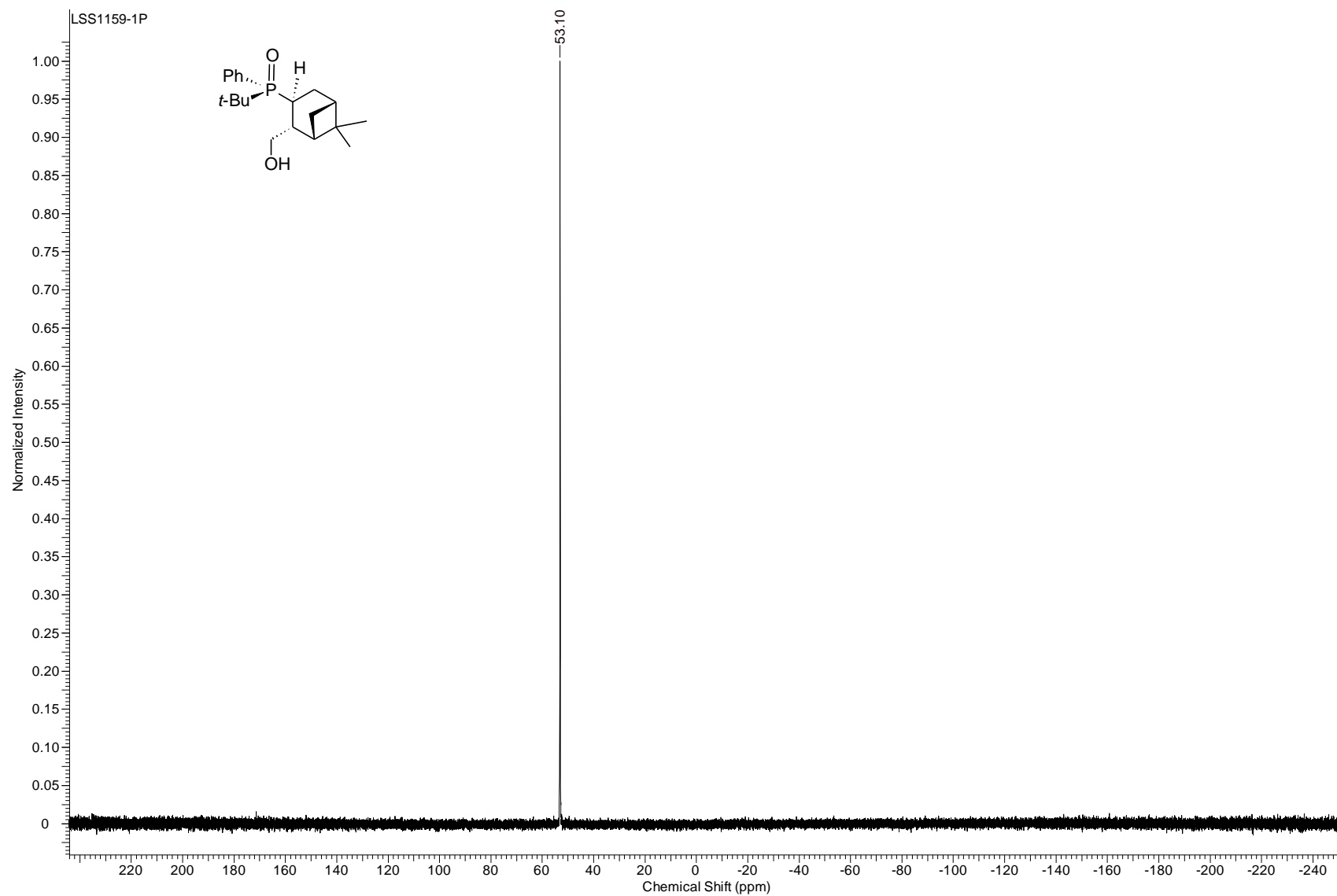
HMBC spectrum of (*S<sub>P</sub>*)-(6,6-dimethylbicyclo[3.1.1]heptan-2-yl)(hydroxy)methyl(methyl)(phenyl) phosphine-borane (*S<sub>P</sub>*)-**12c** (500, 125 MHz,  $\text{CDCl}_3$ )



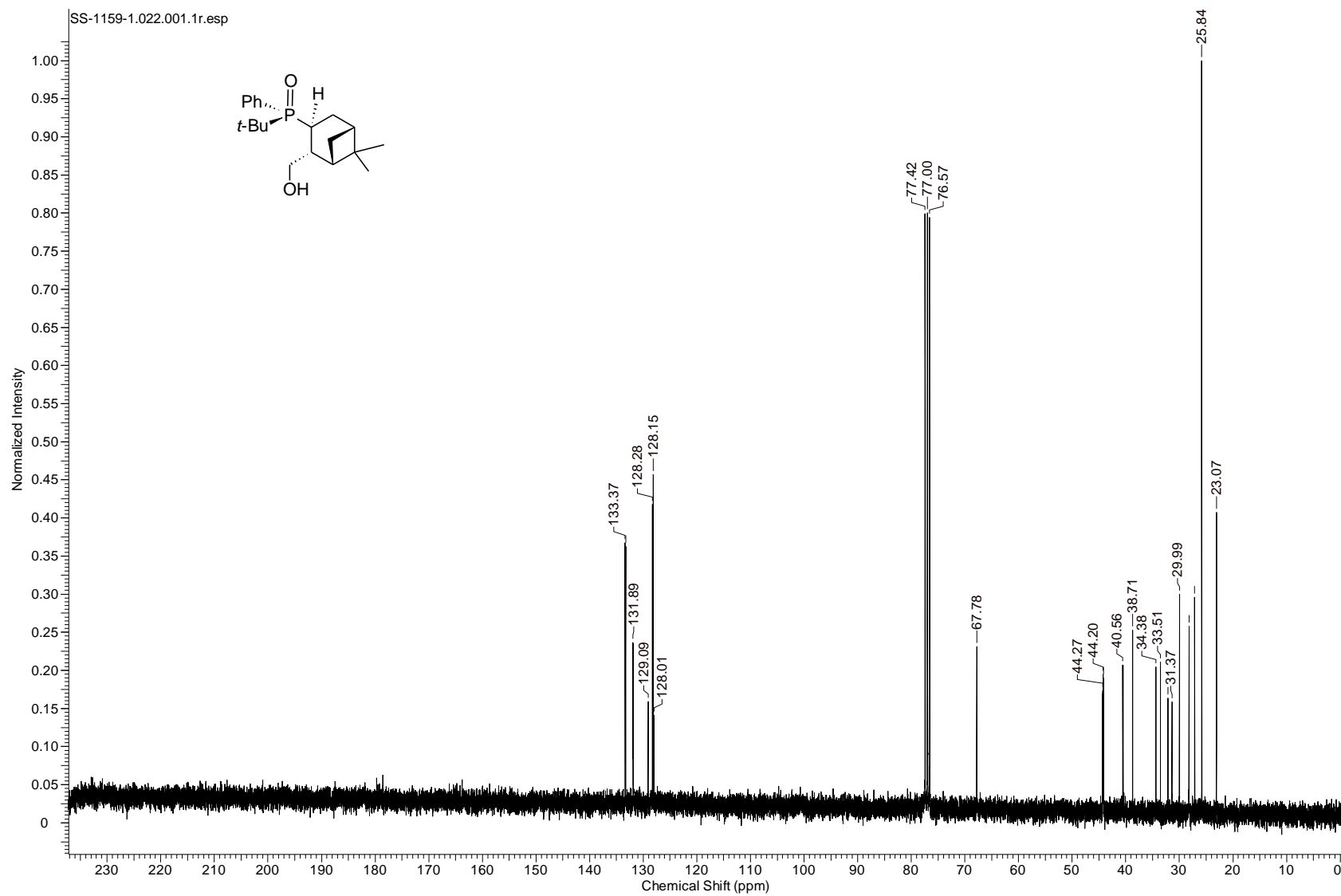
NOESY spectrum of (*S<sub>P</sub>*)-(6,6-dimethylbicyclo[3.1.1]heptan-2-yl)(hydroxymethyl)(methyl)(phenyl) phosphine-borane (*S<sub>P</sub>*)-**12c** (500MHz,  $\text{CDCl}_3$ )



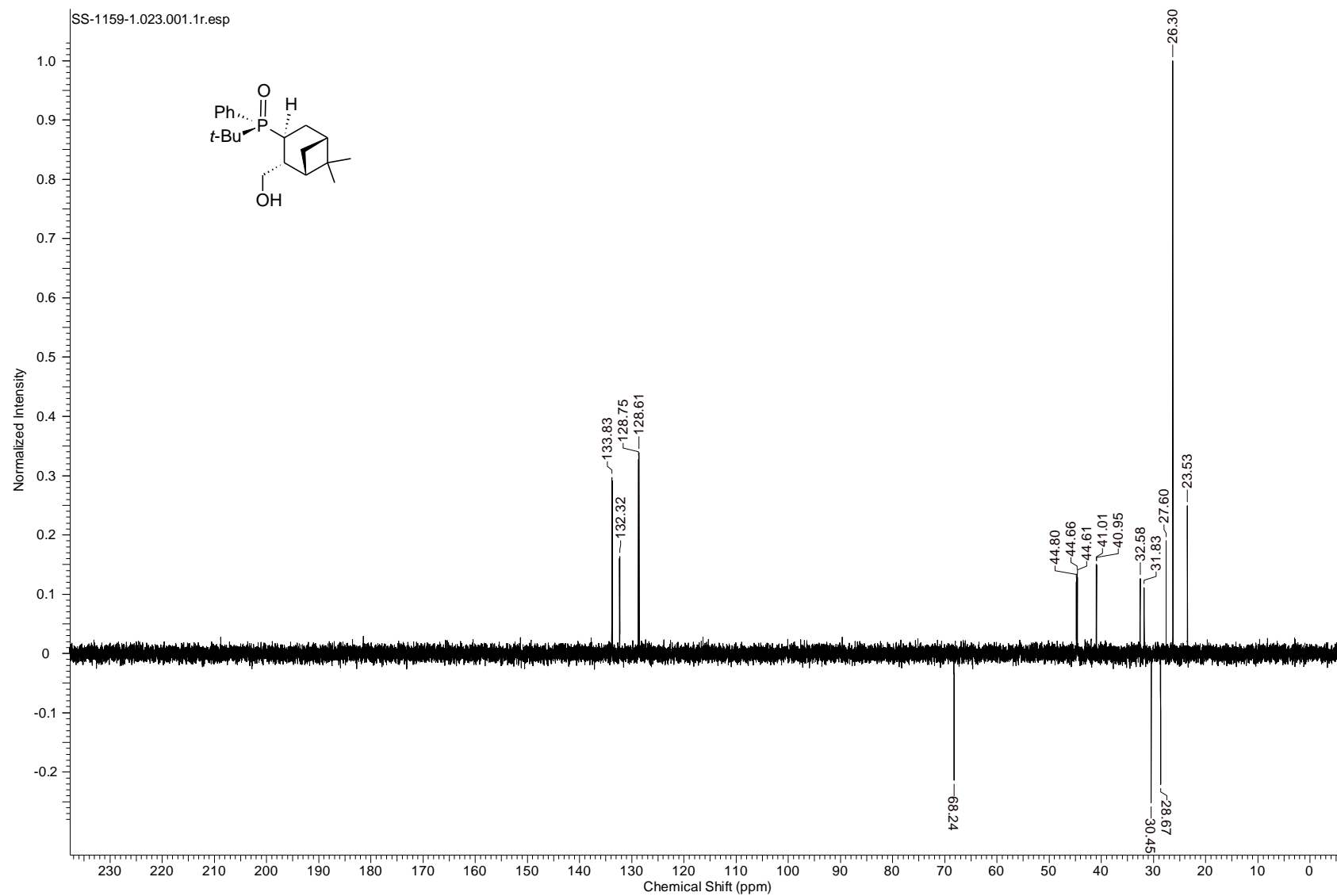
$^1\text{H}$  NMR spectrum of  $(S_P)$ -*t*-butyl-2-(hydroxymethyl)-6,6-dimethylbicyclo[3.1.1]heptan-3-yl(phenyl)phosphine oxide ( $S_P$ )-**13** (400 MHz,  $\text{CDCl}_3$ )



$^{31}\text{P}$  NMR spectrum of  $(S_P)$ -*t*-butyl-2-(hydroxymethyl)-6,6-dimethylbicyclo[3.1.1]heptan-3-yl(phenyl)phosphine oxide ( $S_P$ )-**13** (162 MHz,  $\text{CDCl}_3$ )



<sup>13</sup>C NMR spectrum of (*S\_P*)-*t*-butyl-2-(hydroxymethyl)-6,6-dimethylbicyclo[3.1.1]heptan-3-yl(phenyl)phosphine oxide (*S\_P*)-**13** (75 MHz, CDCl<sub>3</sub>)



DEPT 135 NMR spectrum of  $(S_P)$ -*t*-butyl-2-(hydroxymethyl)-6,6-dimethylbicyclo[3.1.1]heptan-3-yl(phenyl)phosphine oxide ( $S_P$ )-**13** (75 MHz,  $\text{CDCl}_3$ )

### 3. References

- [1] Sakai, K.; Kobori, T.; Fujisawa, T. Prostaglandin synthesis from a fulvene with the  $\omega$ -side chain equivalent. *Tetrahedron Lett.* **1981**, 22, 115 – 118.
- [2] Lee, A. S.-Y., Cheng, C.-L. A Novel and selective method for hydrolysis of acetals and ketals. *Tetrahedron* **1997**, 53, 42, 14255-14262.
- [3] (a) Bessiere-Chretien, Y.; El Gaied, M.M. *Bull. Soc. Chim. Fr.* **1971**, 2189 – 2194 (b) El Gaied, M. M.; Bessiere-Chretien, Y. Réduction par le lithium dessous dans l'ammoniac liquide de cétones cyclopropaniques dérivées de terpènes. *Bull. Soc. Chim. Fr.* **1973**, 1351–1356.
- [4] Stankevič M., Pietrusiewicz K. M. An expedient reduction of *sec*-phosphine oxides to *sec*-phosphine-boranes by  $\text{BH}_3\text{-SMe}_2$ . *Synlett*, **2003**, 7, 1012-1016.