

Metal- and Chloride Reagent-Free Synthesis of Mixed Thiophosphates

Jeffrey Ash,^a Hai Huang,^{a,b} and Jun Yong Kang^{*,a}

^a Department of Chemistry and Biochemistry, University of Nevada Las Vegas, 4505 S.

Maryland Parkway, Las Vegas, Nevada, 89154-4003, United States

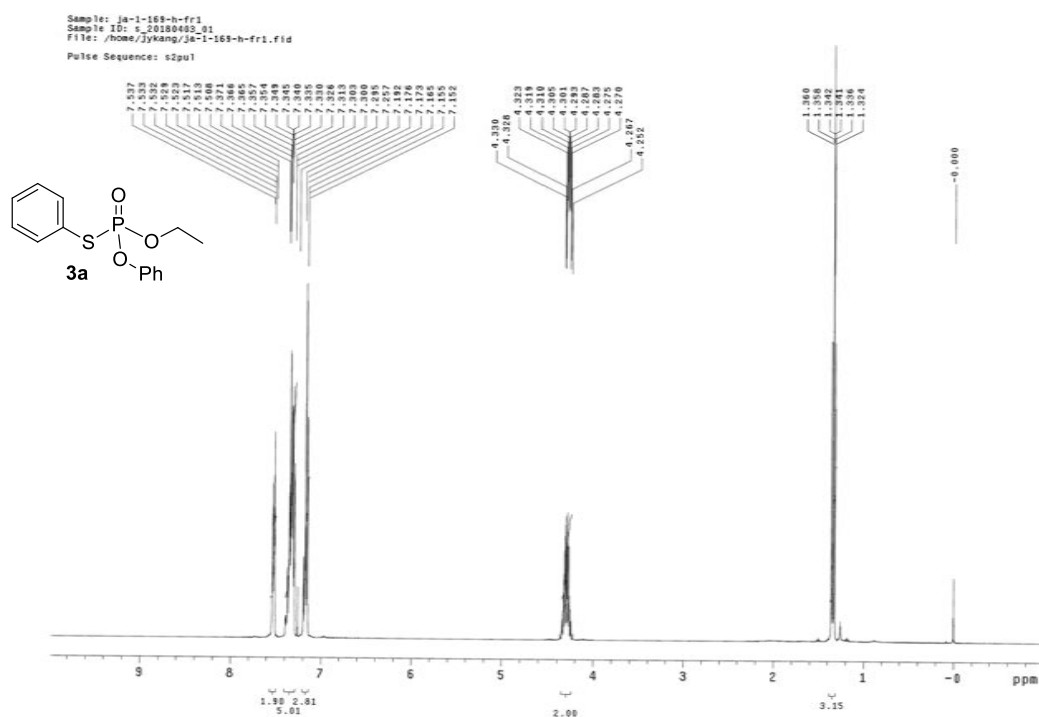
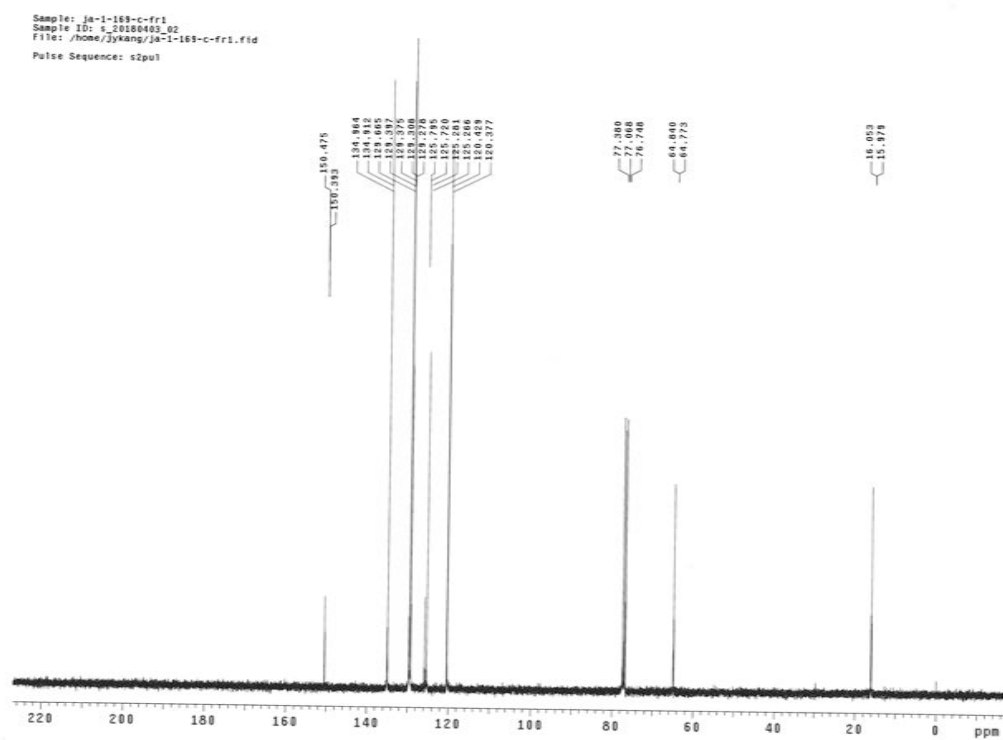
^b Department of Applied Chemistry, College of Chemistry and Molecular Engineering, Nanjing

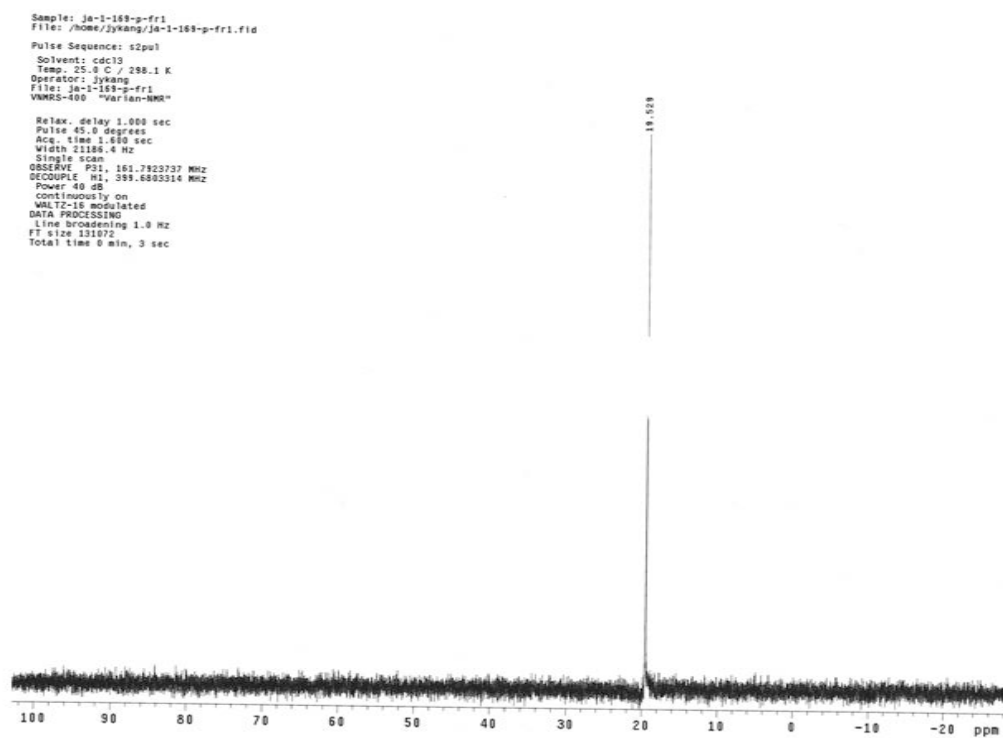
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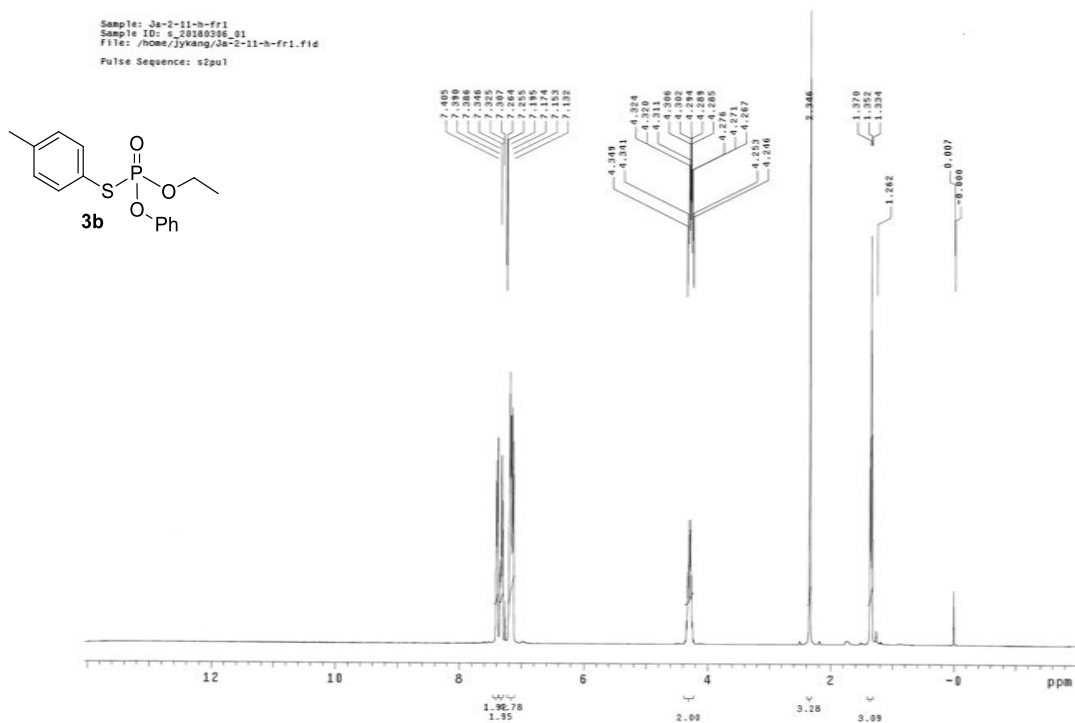
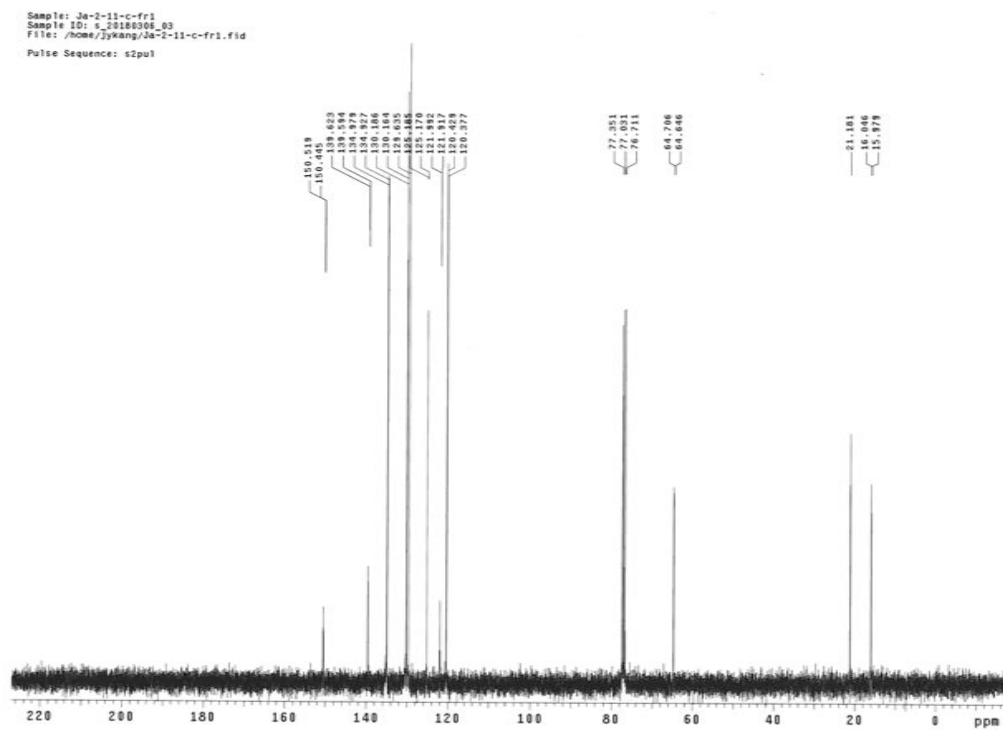
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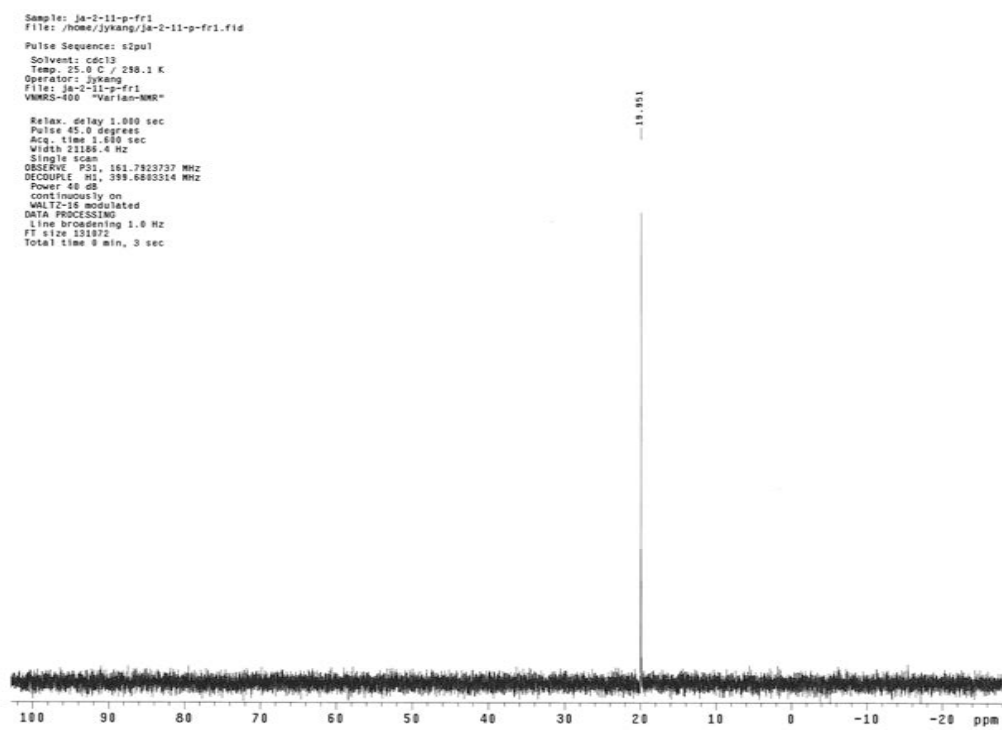
Supporting Information

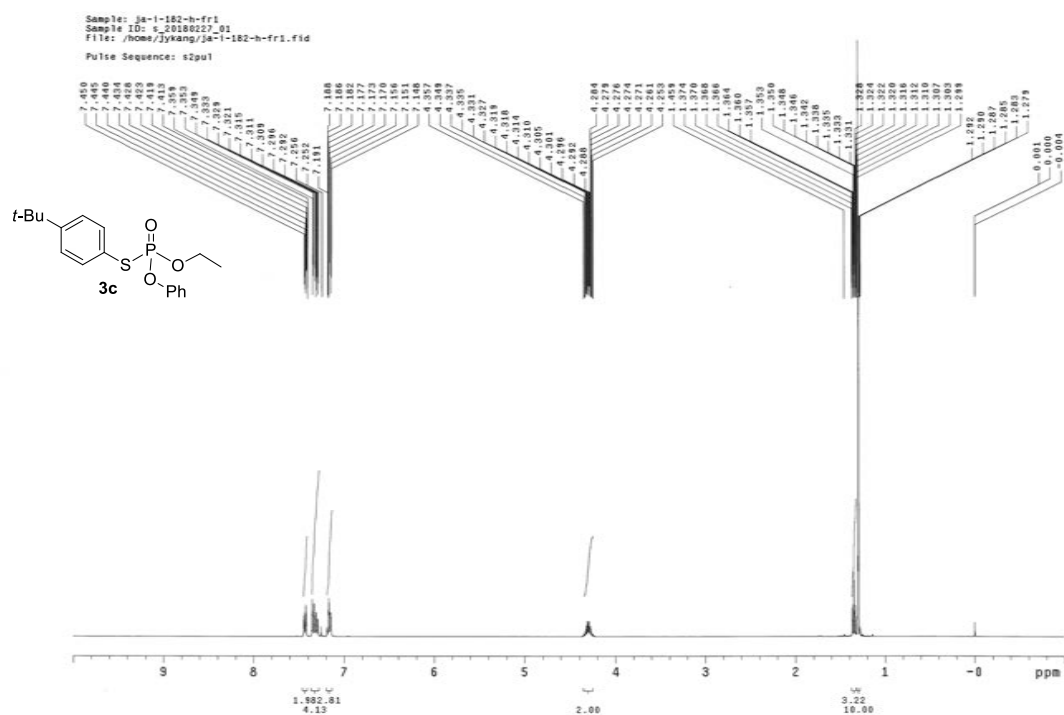
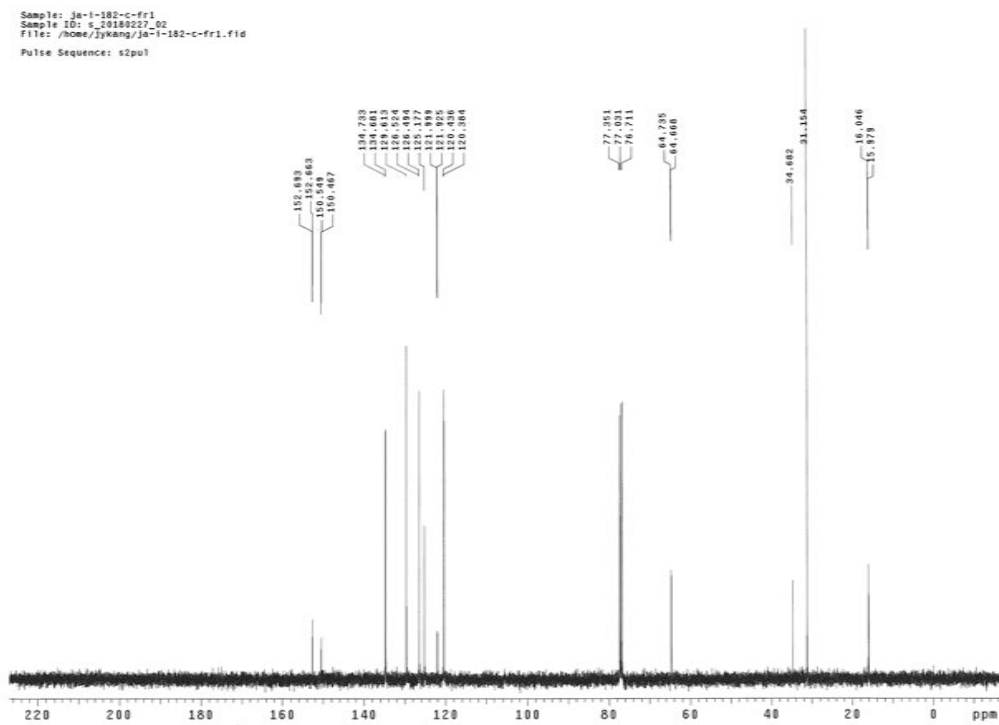
¹ H, ¹³ C, and ³¹ P NMR Spectra.....	S2-S45
Figure S1.....	S46

^1H NMR (400 MHz) in CDCl_3  ^{13}C NMR (100.5 MHz) in CDCl_3 

^{31}P NMR (162 MHz) in CDCl_3 

^1H NMR (400 MHz) in CDCl_3  ^{13}C NMR (100.5 MHz) in CDCl_3 

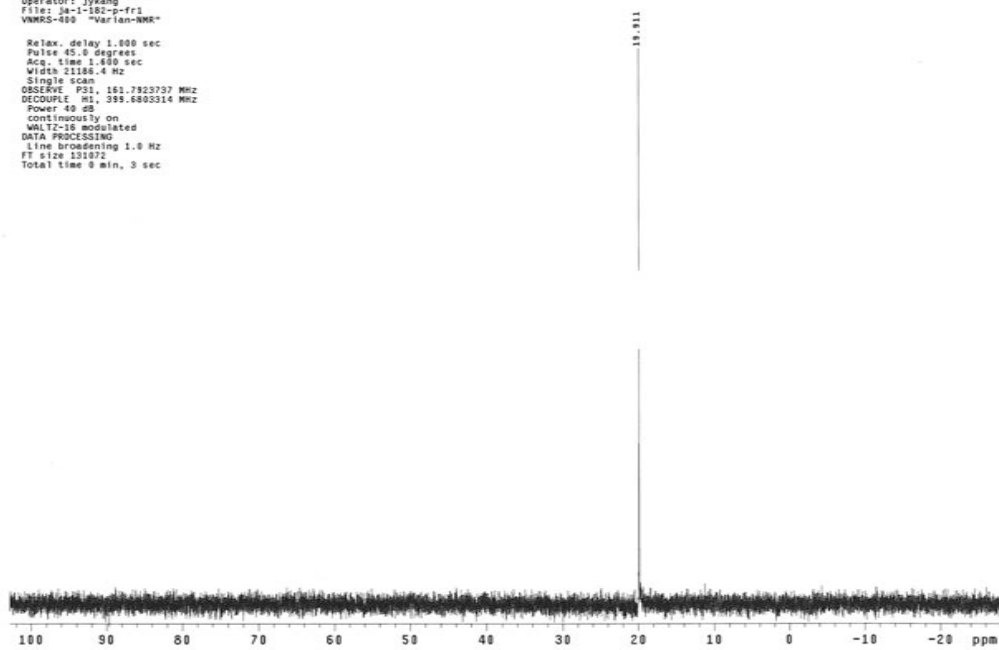
^{31}P NMR (162 MHz) in CDCl_3 

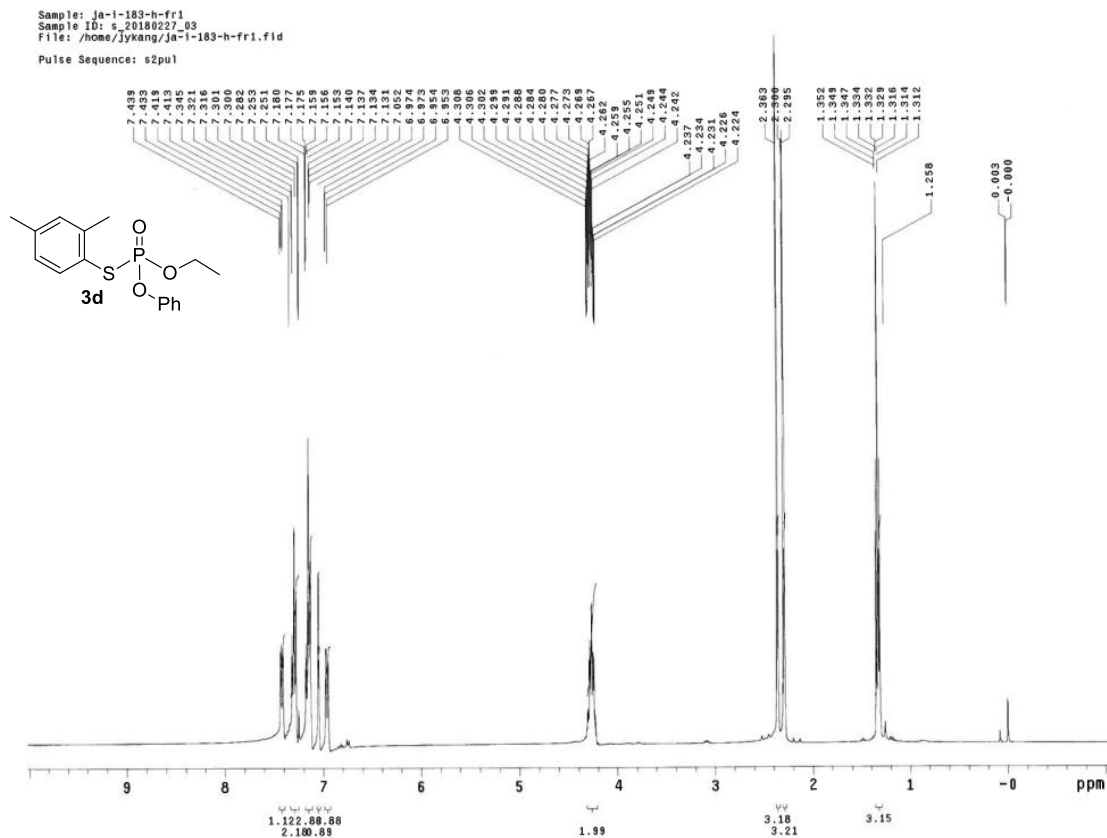
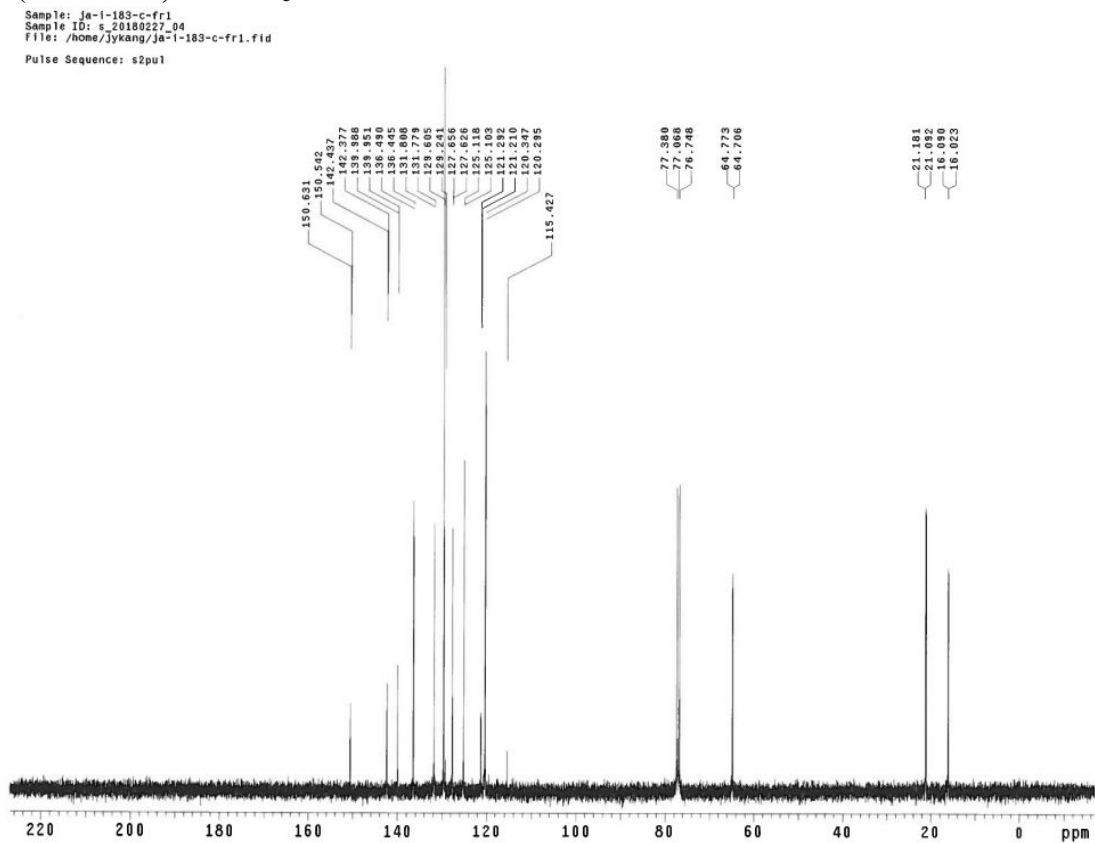
^1H NMR (400 MHz) in CDCl_3  ^{13}C NMR (100.5 MHz) in CDCl_3 

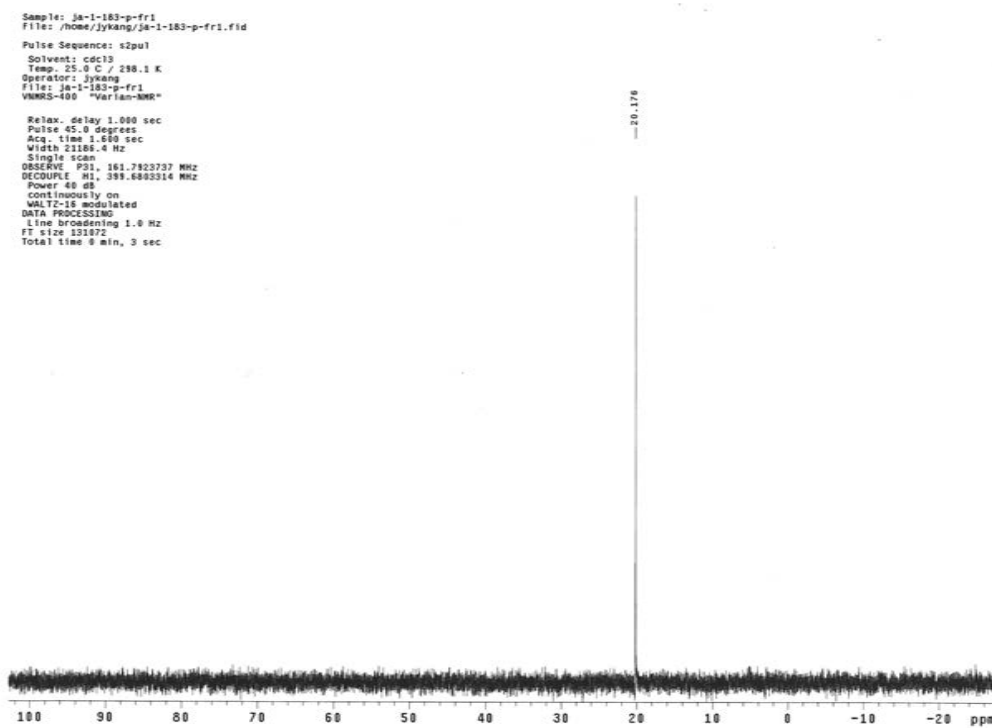
^{31}P NMR (162 MHz) in CDCl_3

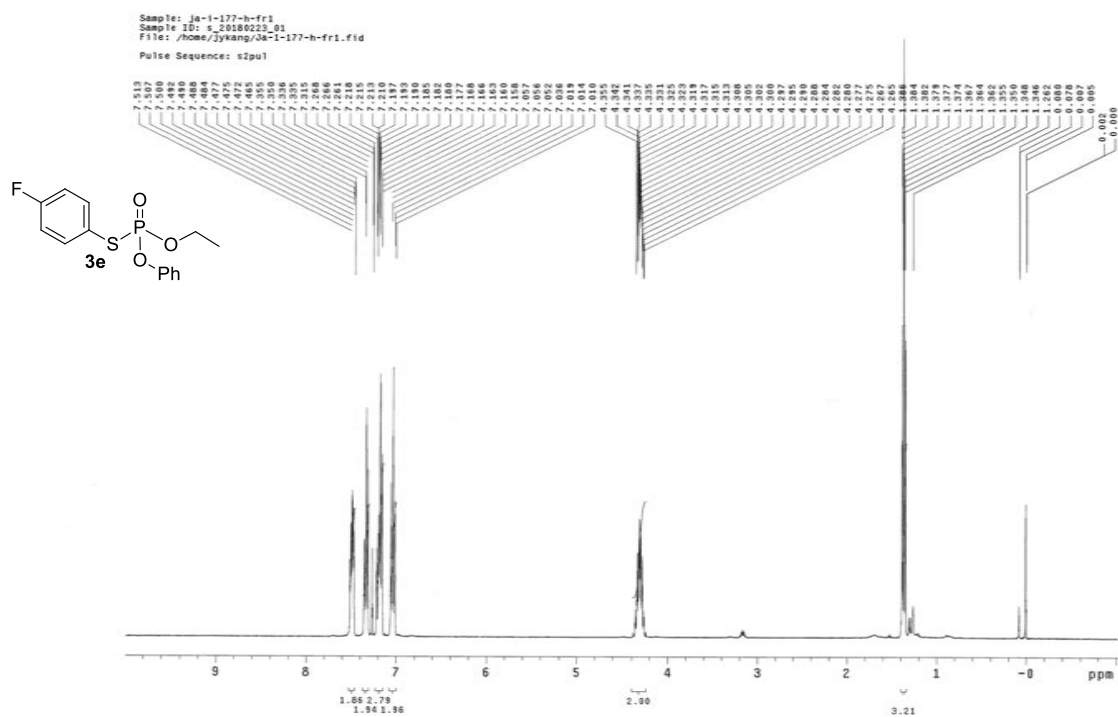
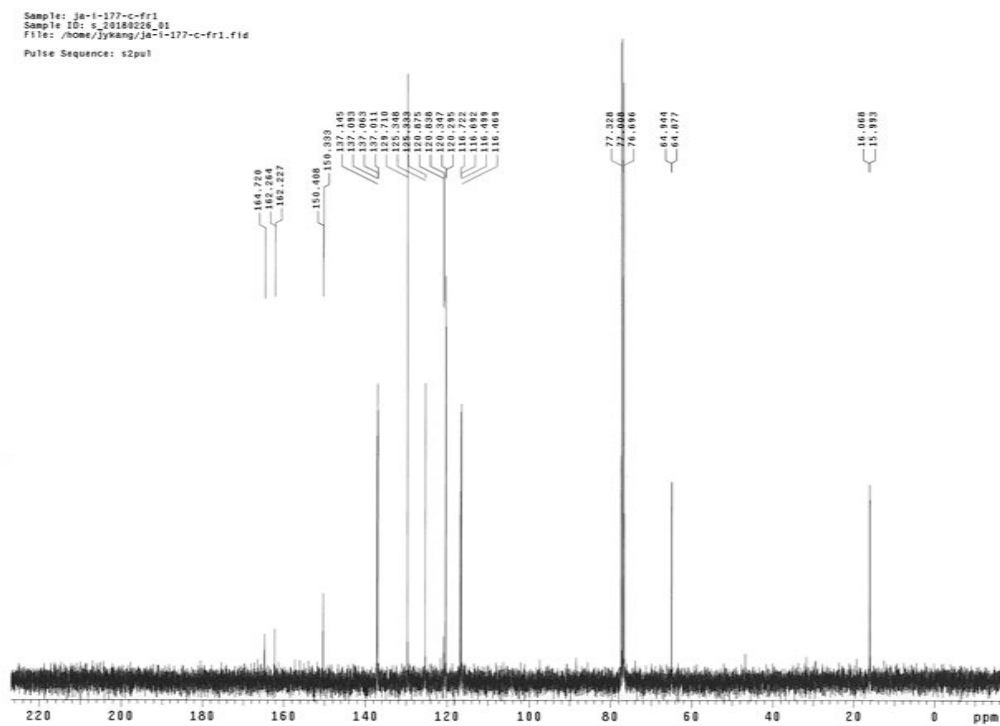
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Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: jykwang
File: ja-1-182-p-fr1
VWEGS-450 "Varian-MMR"

Relax. delay 1.000 sec
Pulse: 45.0 degree
Acq. time 1.600 sec
Width 21186.4 Hz
Single scan
OBSERV: F31: 161.7823737 MHz
DECUPLE: M1: 399.6403314 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 9 min, 3 sec
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^1H NMR (400 MHz) in CDCl_3  ^{13}C NMR (100.5 MHz) in CDCl_3 

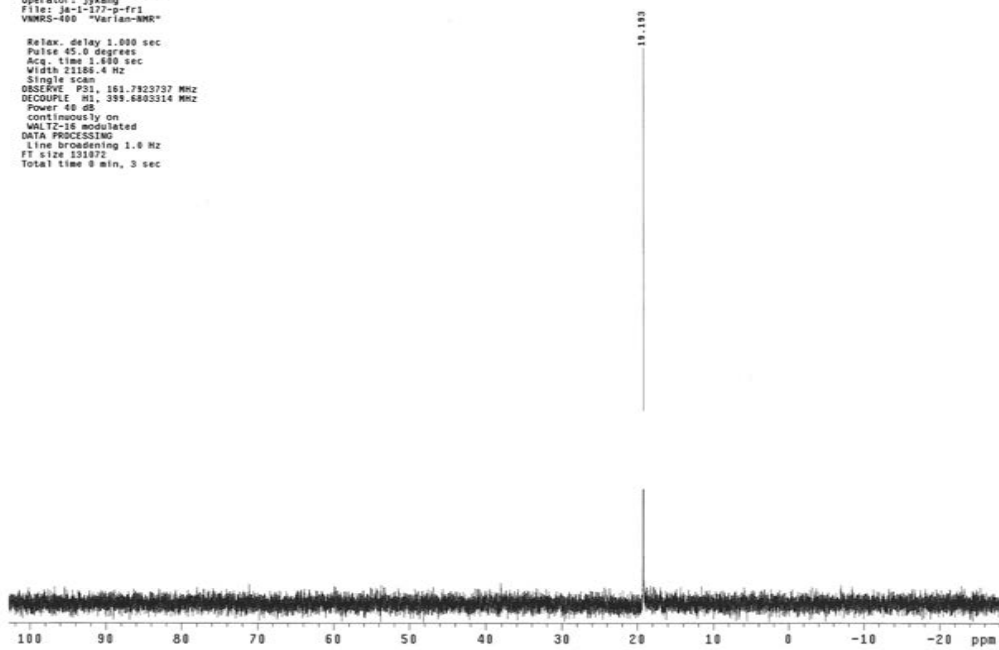
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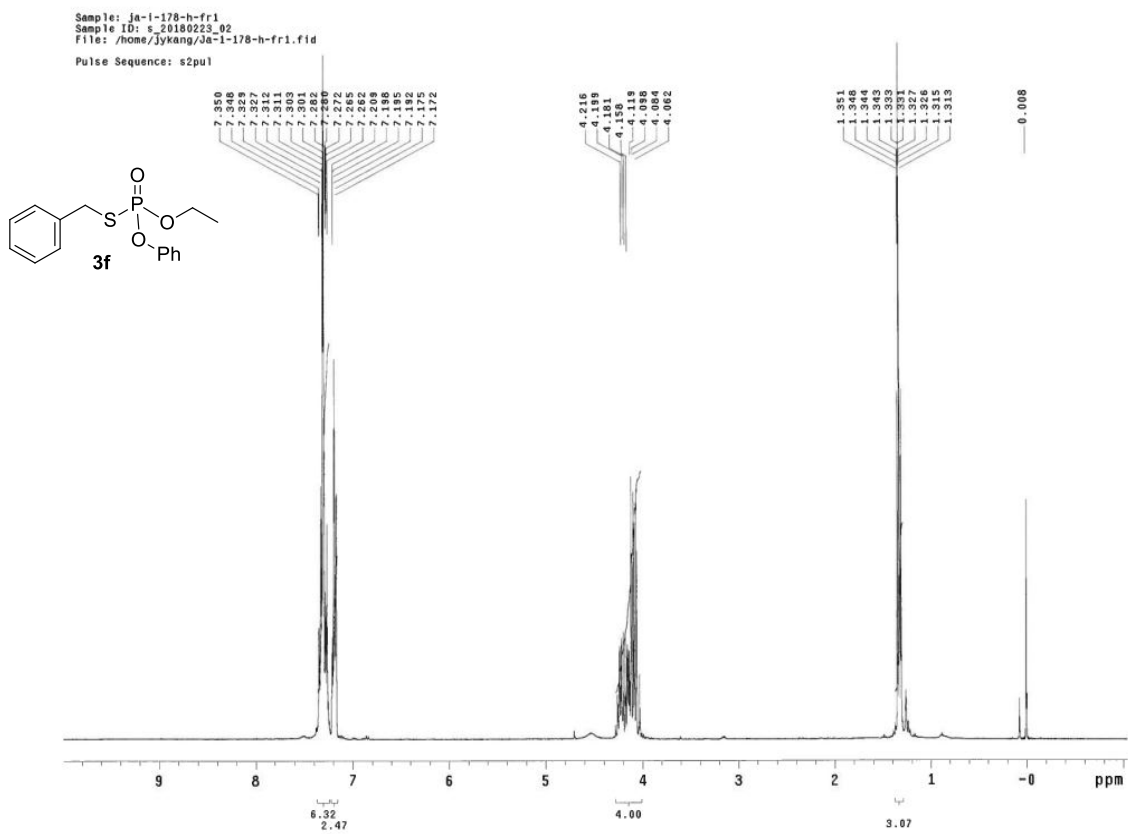
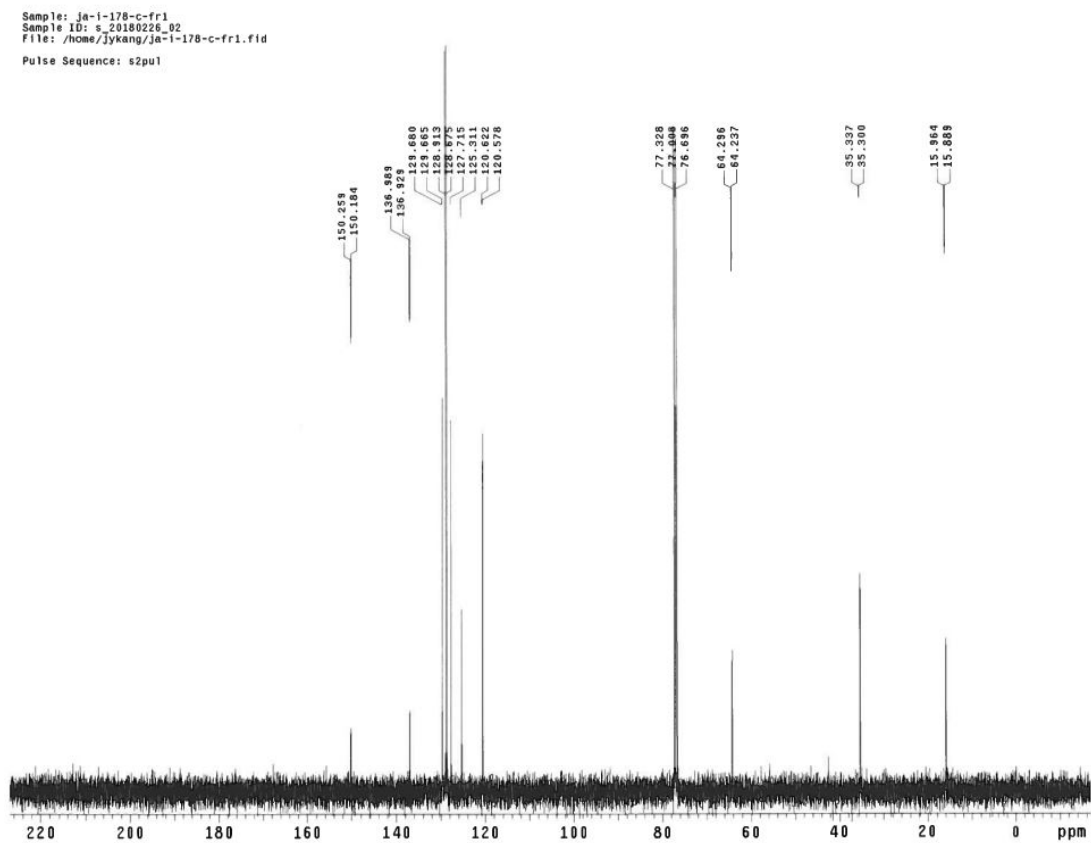
^1H NMR (400 MHz) in CDCl_3  ^{13}C NMR (100.5 MHz) in CDCl_3 

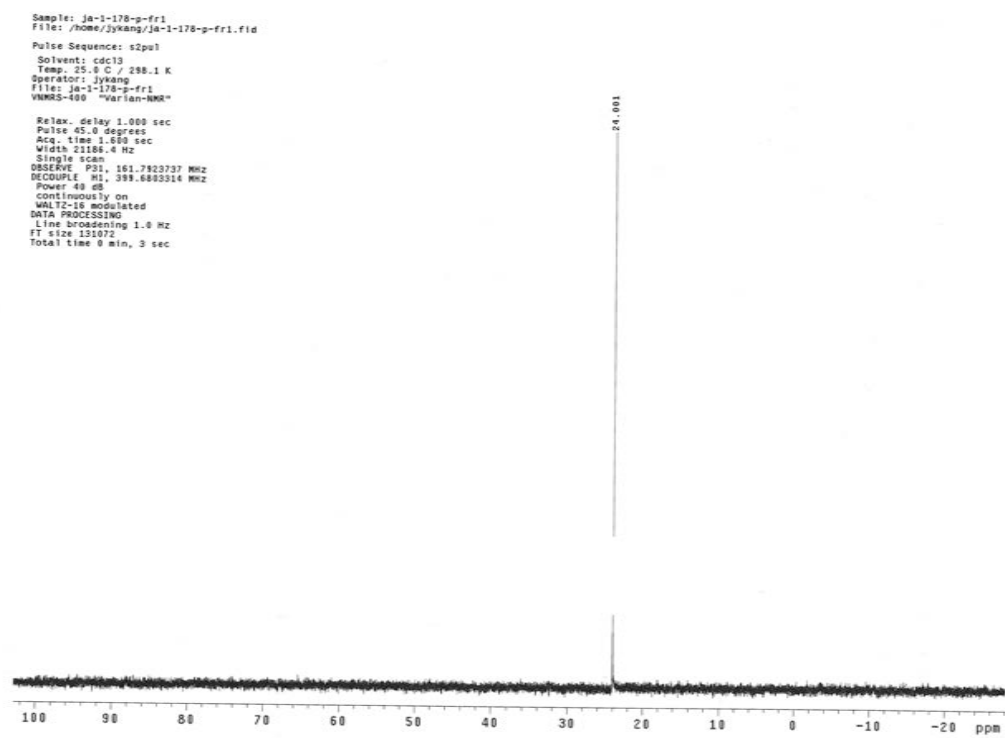
^{31}P NMR (162 MHz) in CDCl_3

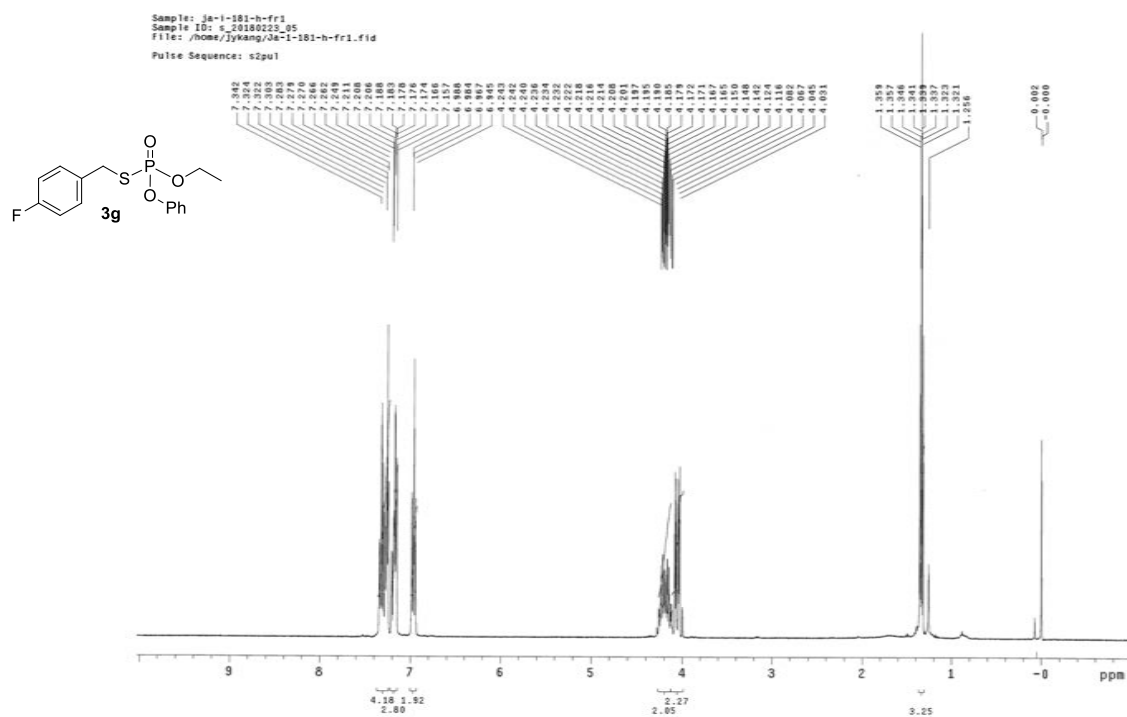
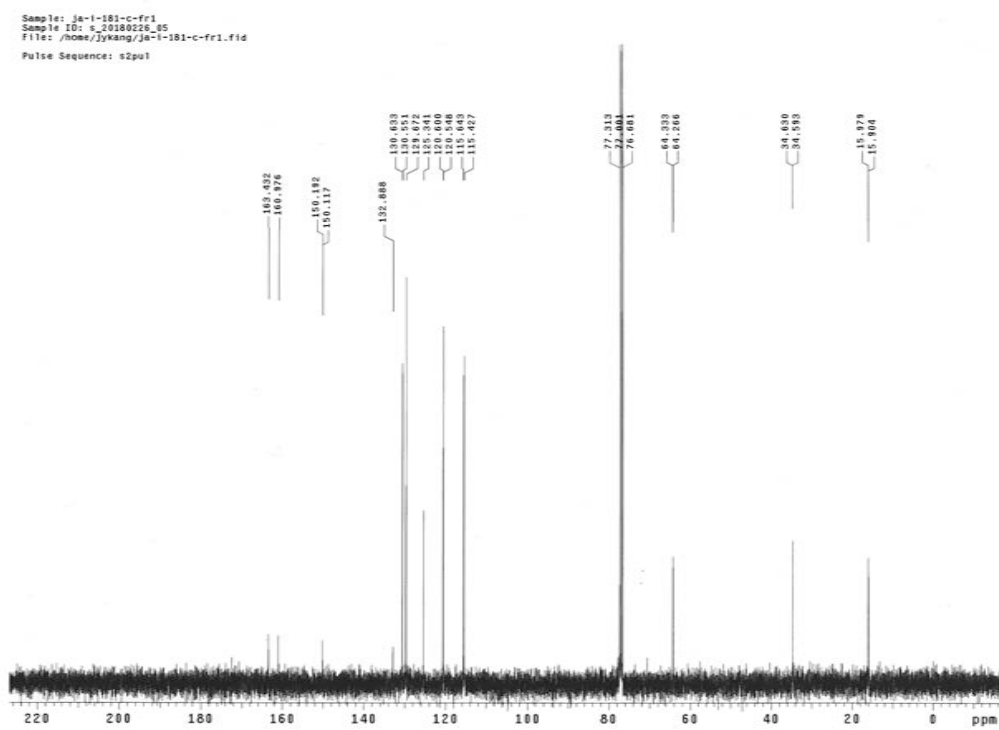
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Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 25.0 C / 230.1 K
Operator: jykwang
File: ja-1-177-p-fr1
VMRS-3D0 "Varian-NMR"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 21186.4 Hz
Single scan
OBSERVE F31: 161.7823737 MHz
DECOUPLE H1: 399.6403314 MHz
Power 46 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 9 min, 3 sec
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^1H NMR (400 MHz) in CDCl_3  **^{13}C NMR (100.5 MHz) in CDCl_3** 

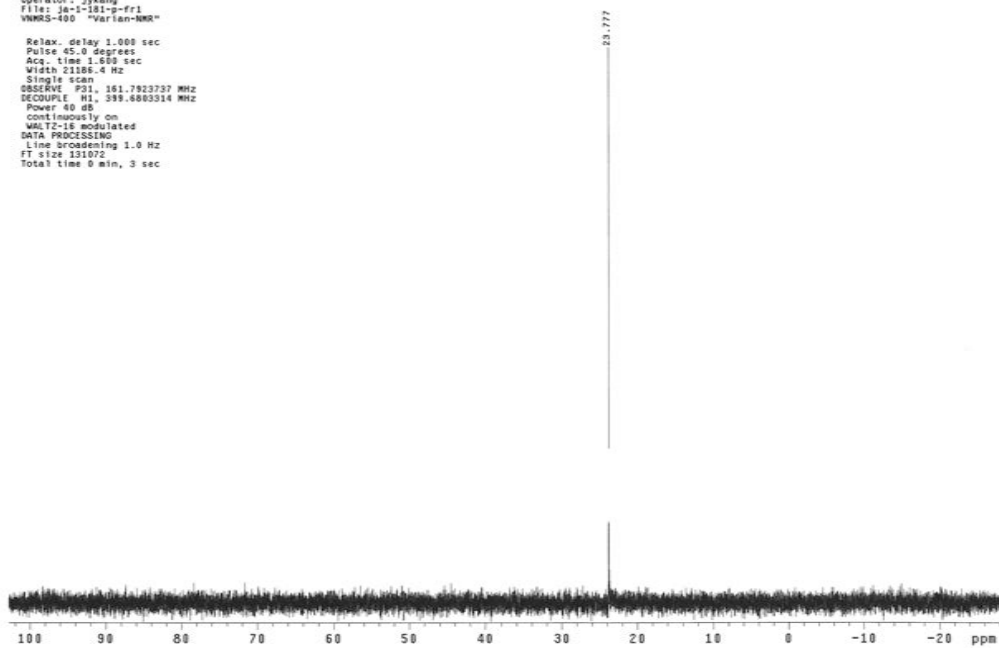
^{31}P NMR (162 MHz) in CDCl_3 

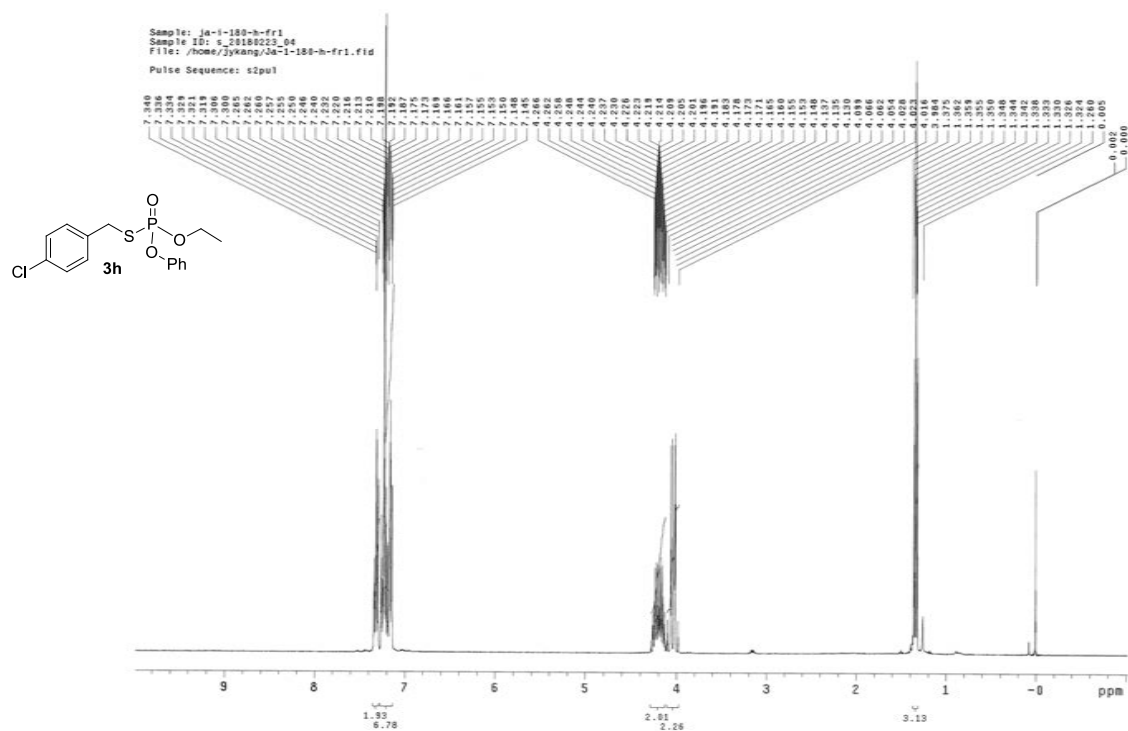
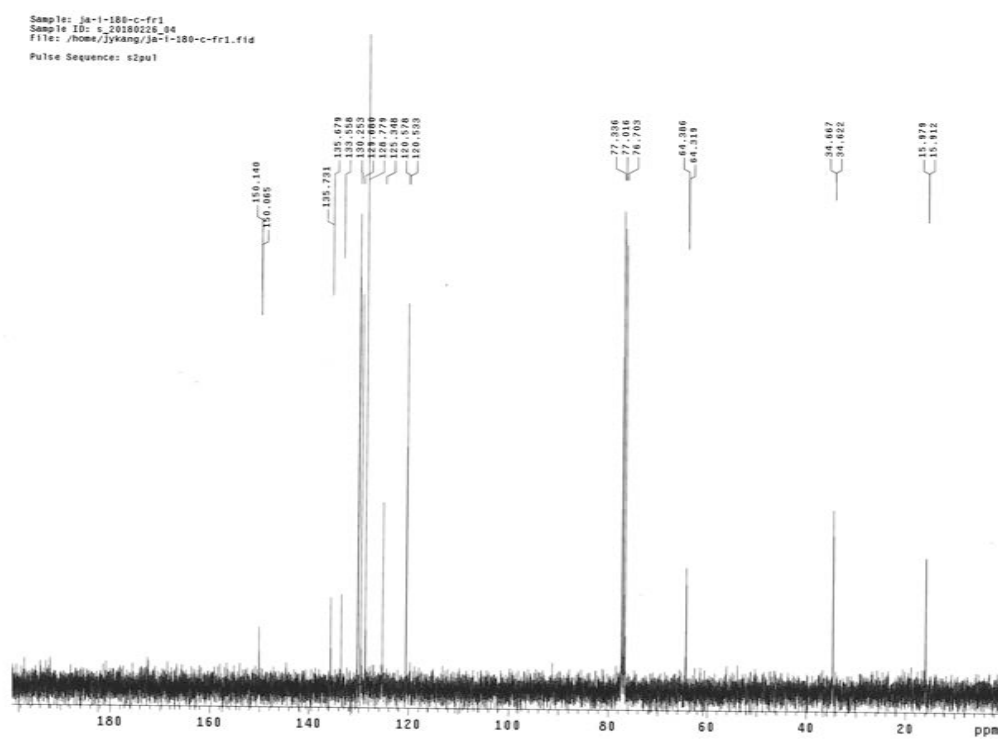
^1H NMR (400 MHz) in CDCl_3  ^{13}C NMR (100.5 MHz) in CDCl_3 

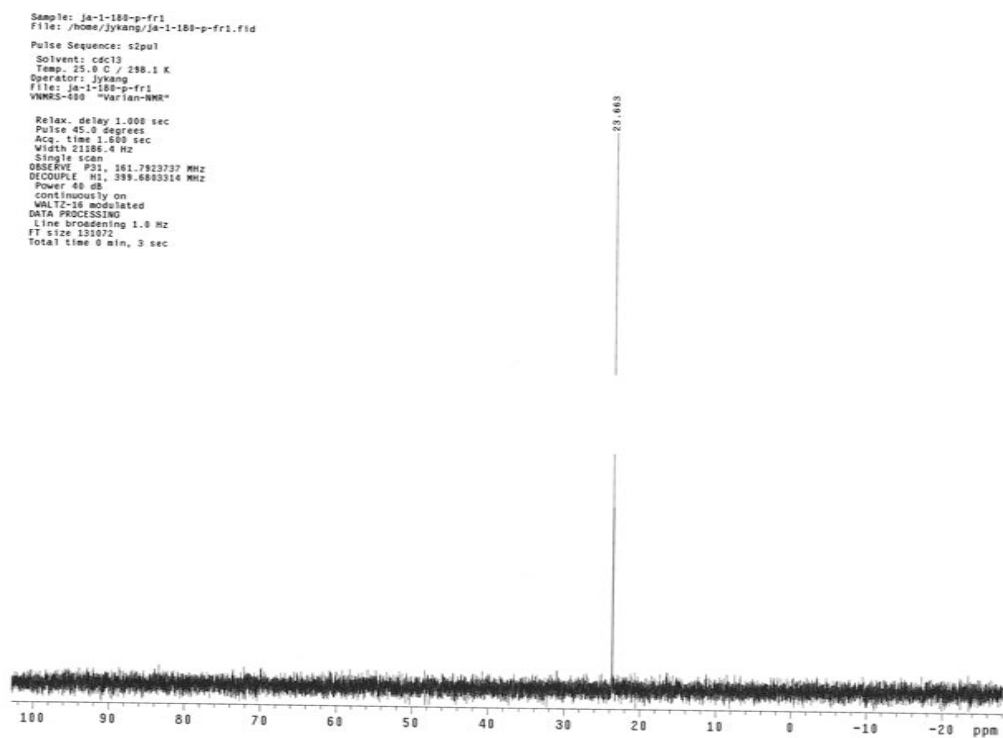
^{31}P NMR (162 MHz) in CDCl_3

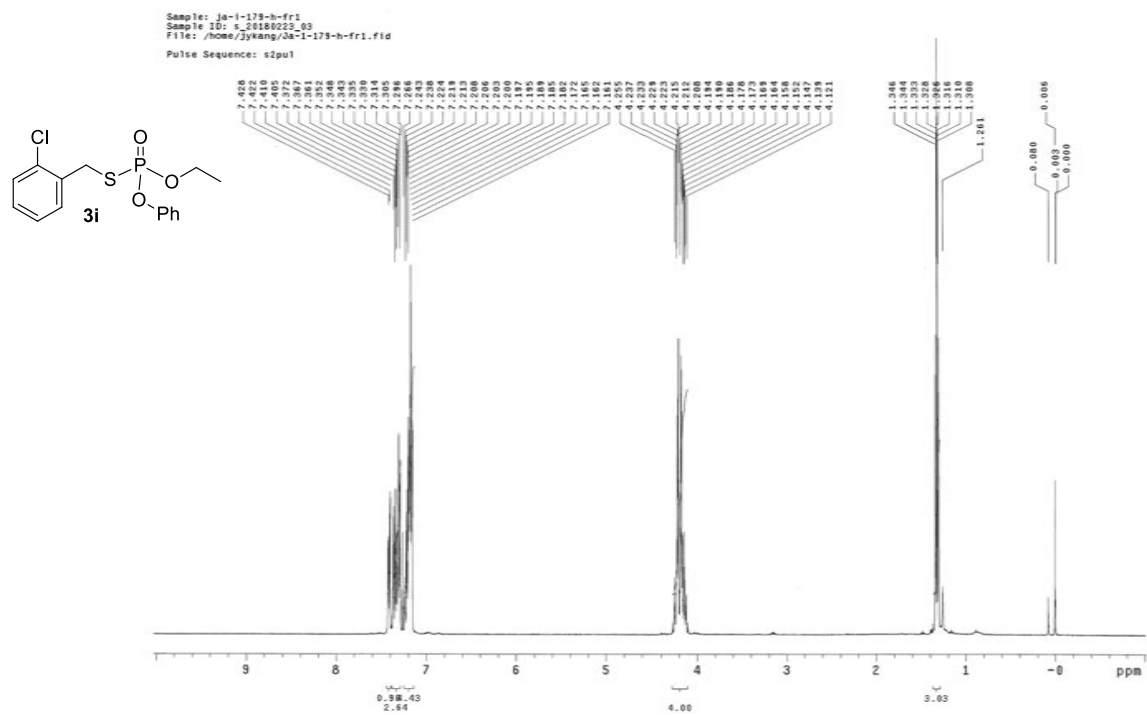
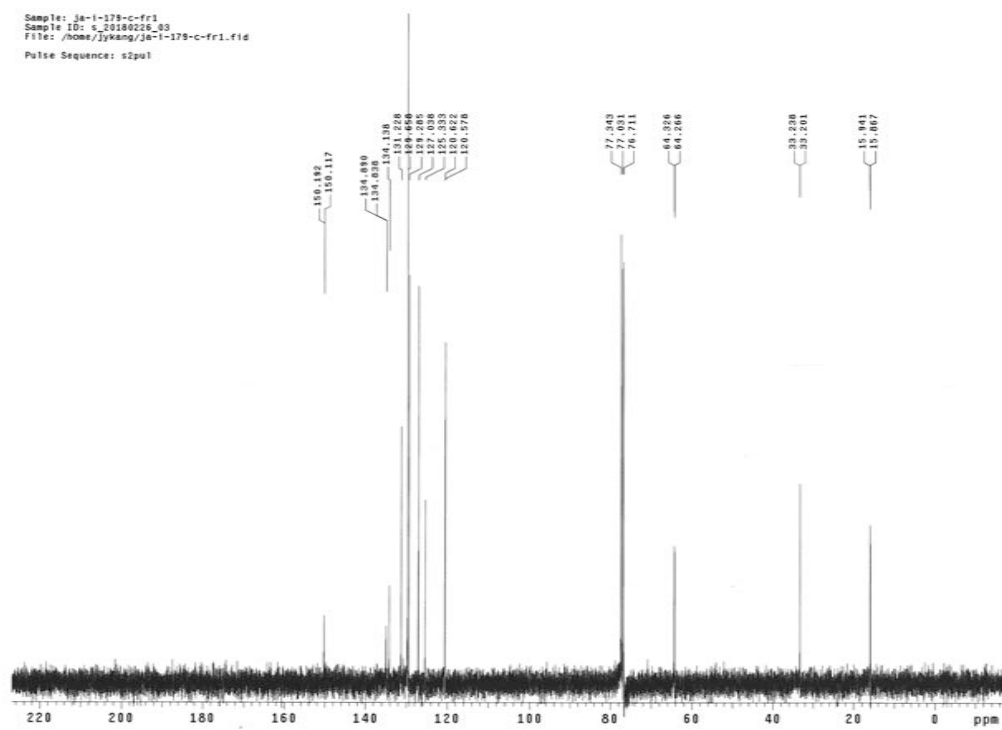
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File: /home/jykwang/ja-1-181-p-fr1.fid
Pulse Sequence: s2pu1
Solvent: cdcl3
Temp: 25.0 C / 288.1 K
Operator: jykwang
File: ja-1-181-p-fr1
VNMR3-400 "Varian-NMR"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 23386.4 Hz
Single scan
OBSERVE P21 161.7823707 MHz
DECOUPLE H1 399.6803314 MHz
Power 60 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 0 min, 3 sec



^1H NMR (400 MHz) in CDCl_3  ^{13}C NMR (100.5 MHz) in CDCl_3 

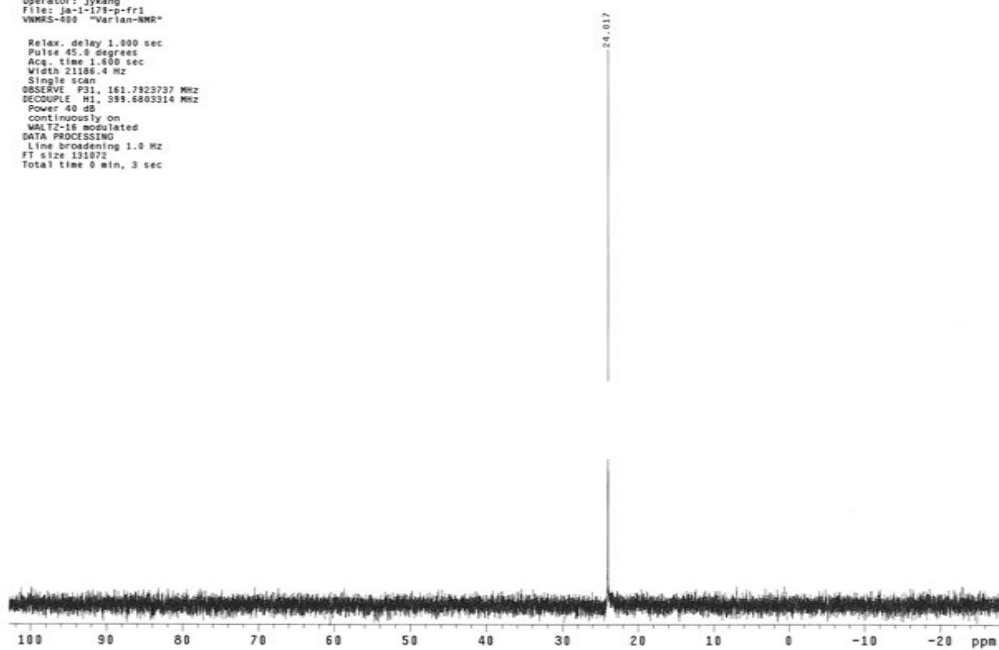
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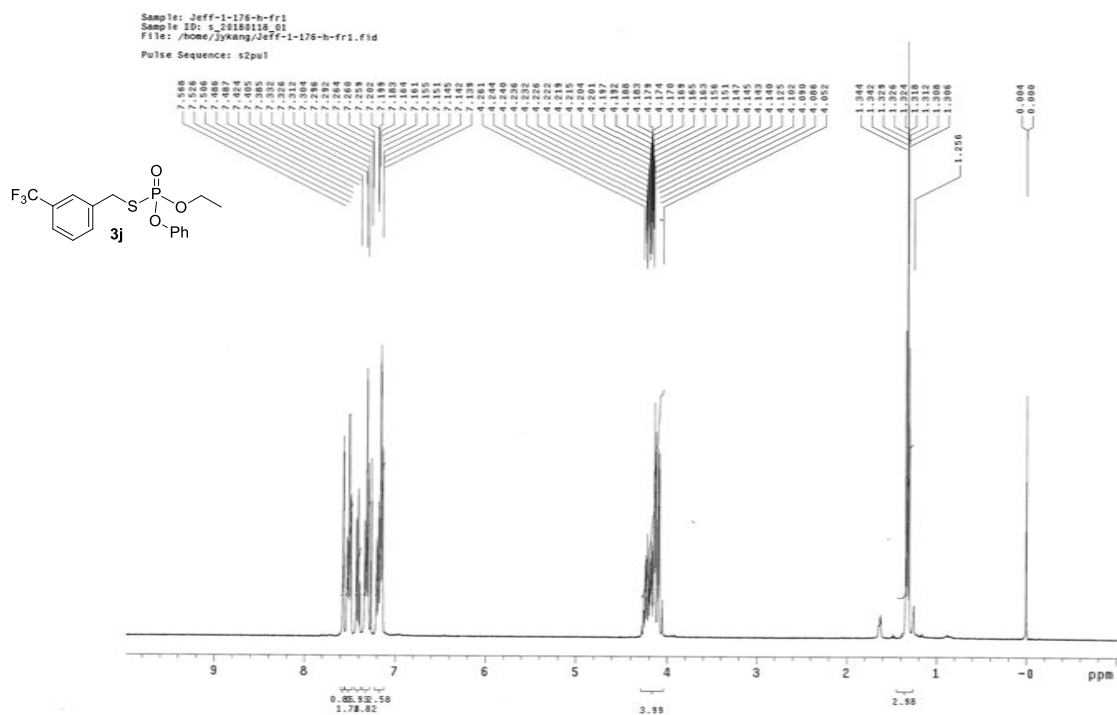
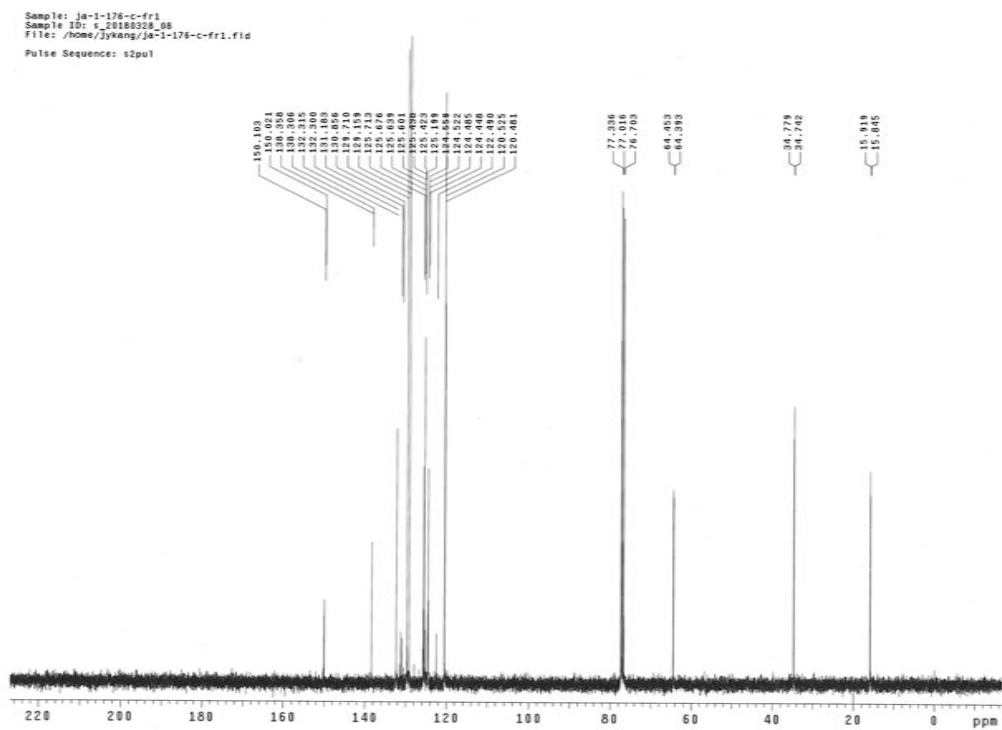
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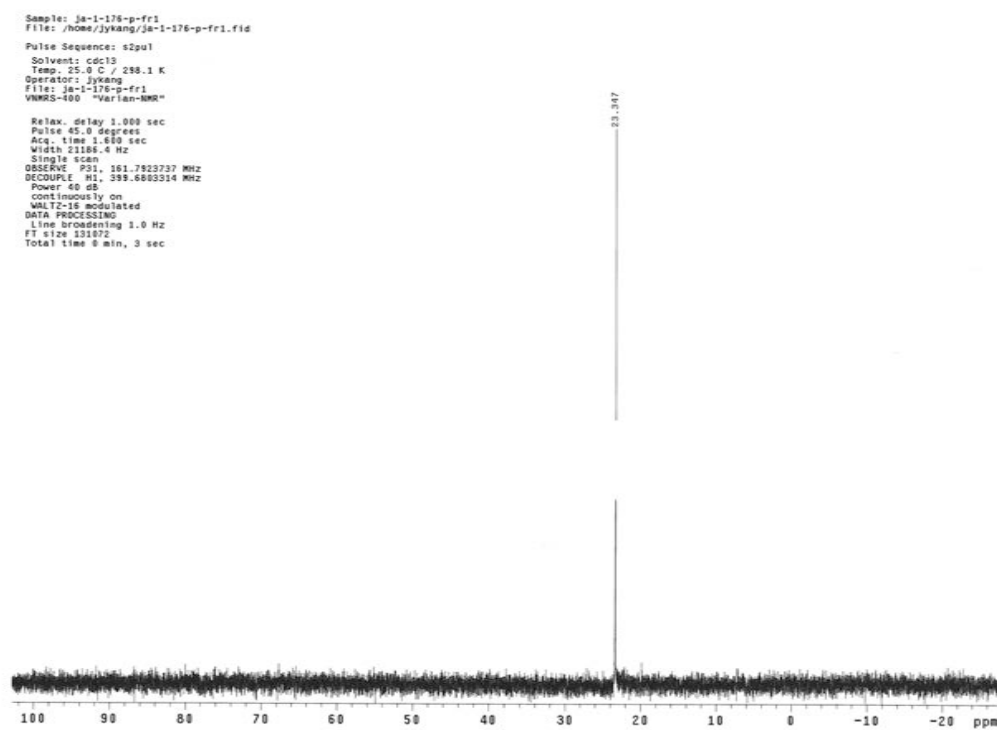
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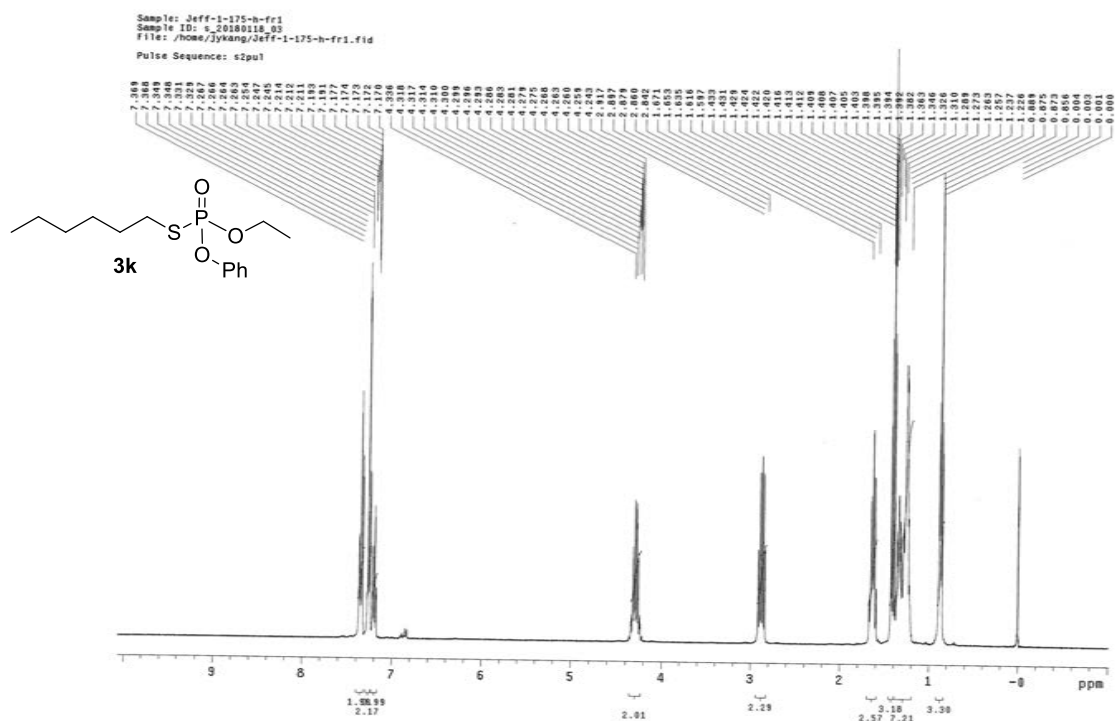
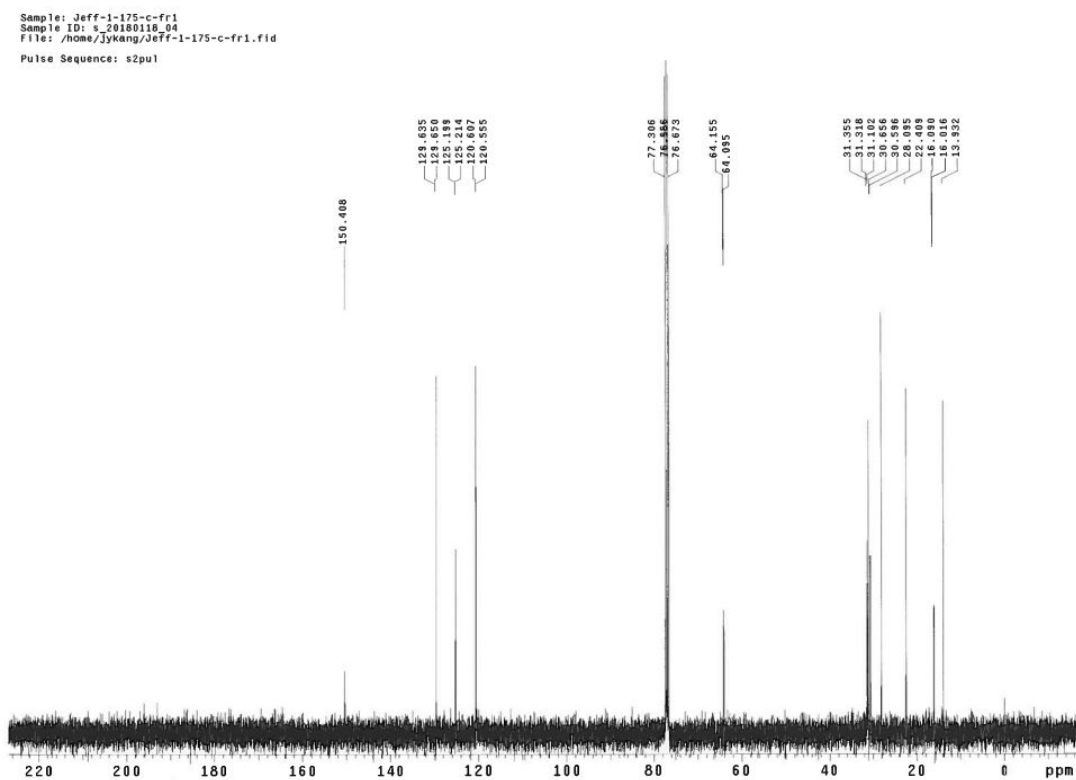
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File: /home/jykwang/ja-1-178-p-fr1.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: jykwang
File: ja-1-178-p-fr1
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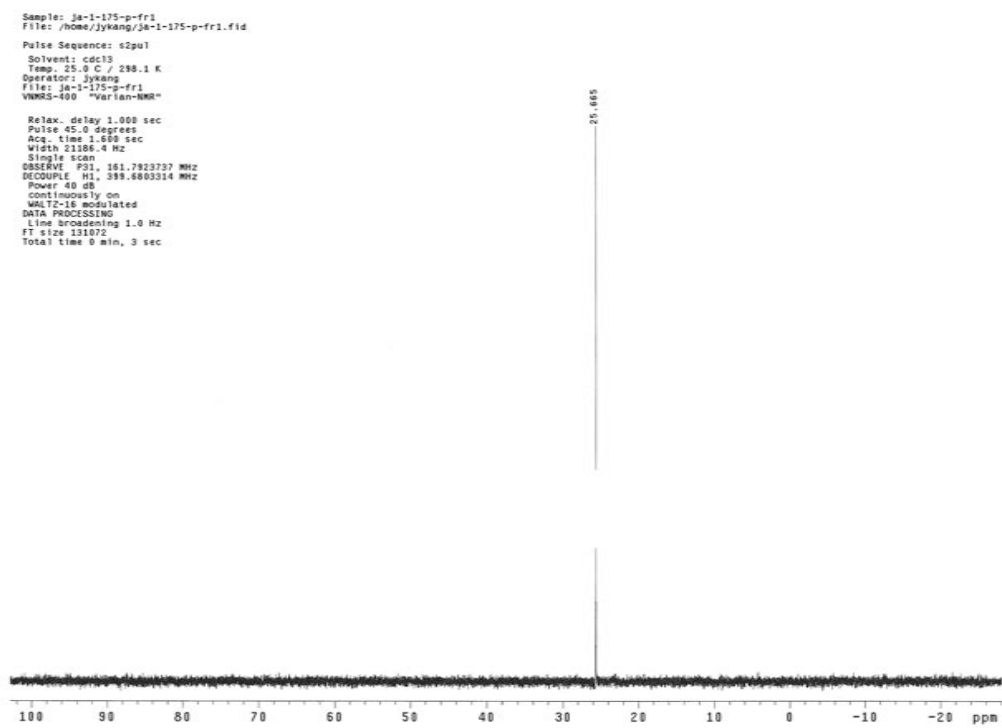
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 21186.4 Hz
Single scan
OBSERVE F31, 161.7923737 MHz
DECOUPLE H1, 399.6405314 MHz
Power 40 db
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 0 min, 3 sec
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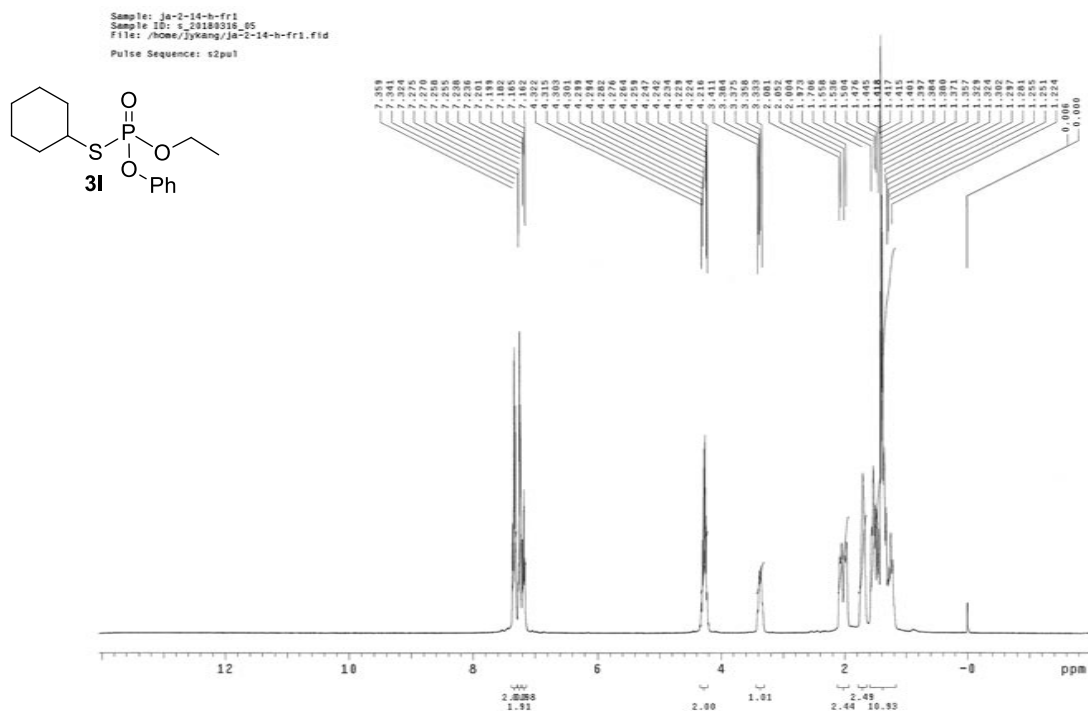
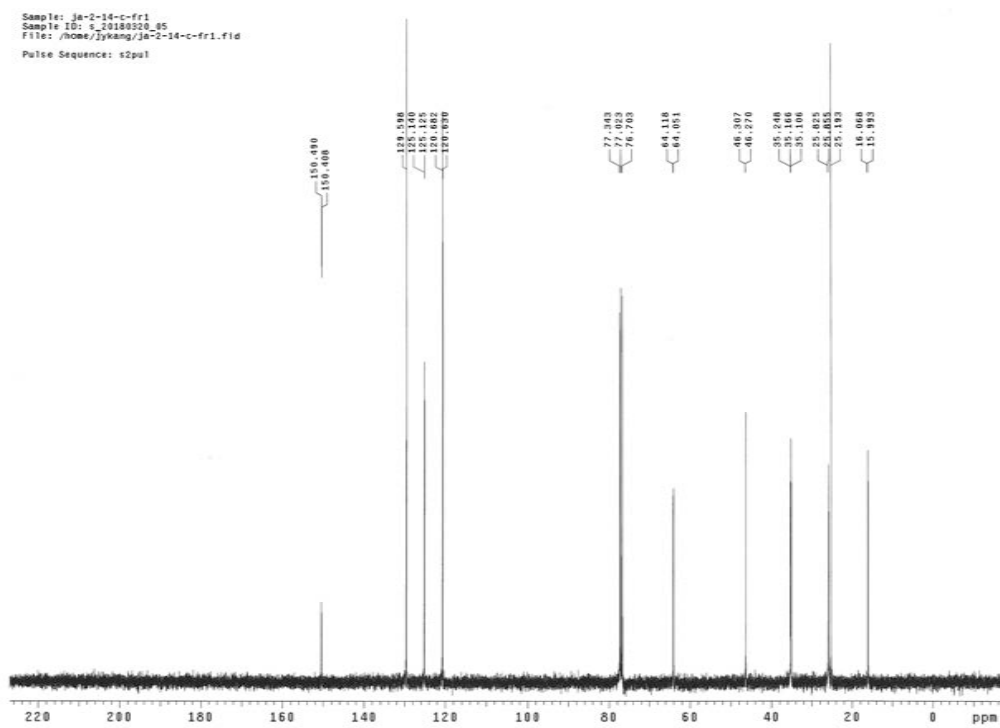


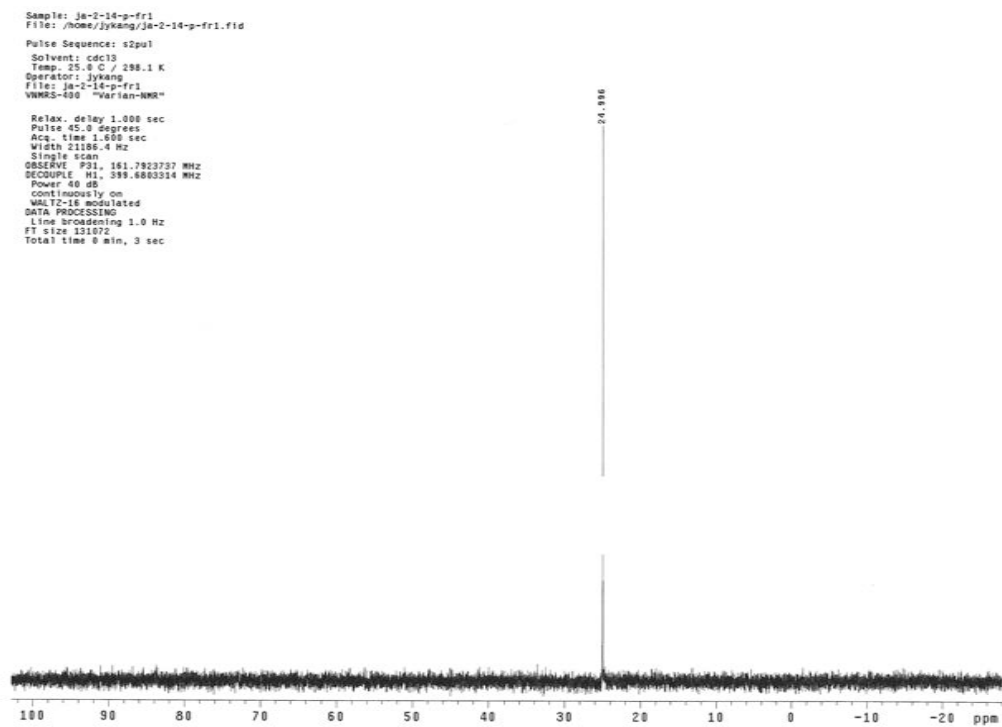
^1H NMR (400 MHz) in CDCl_3  ^{13}C NMR (100.5 MHz) in CDCl_3 

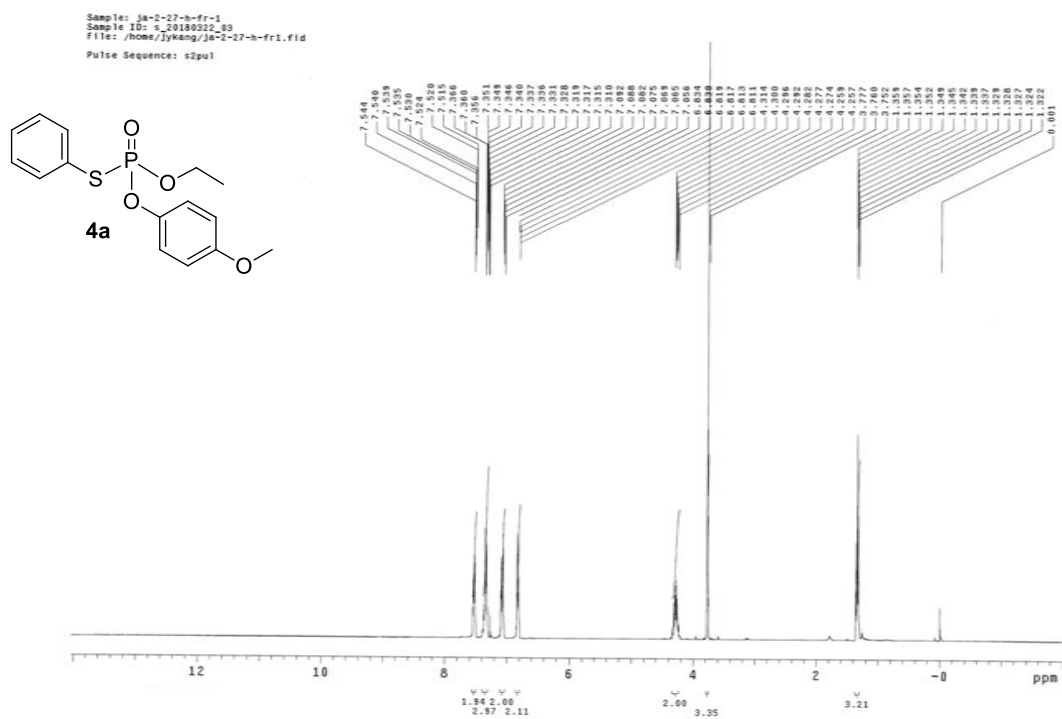
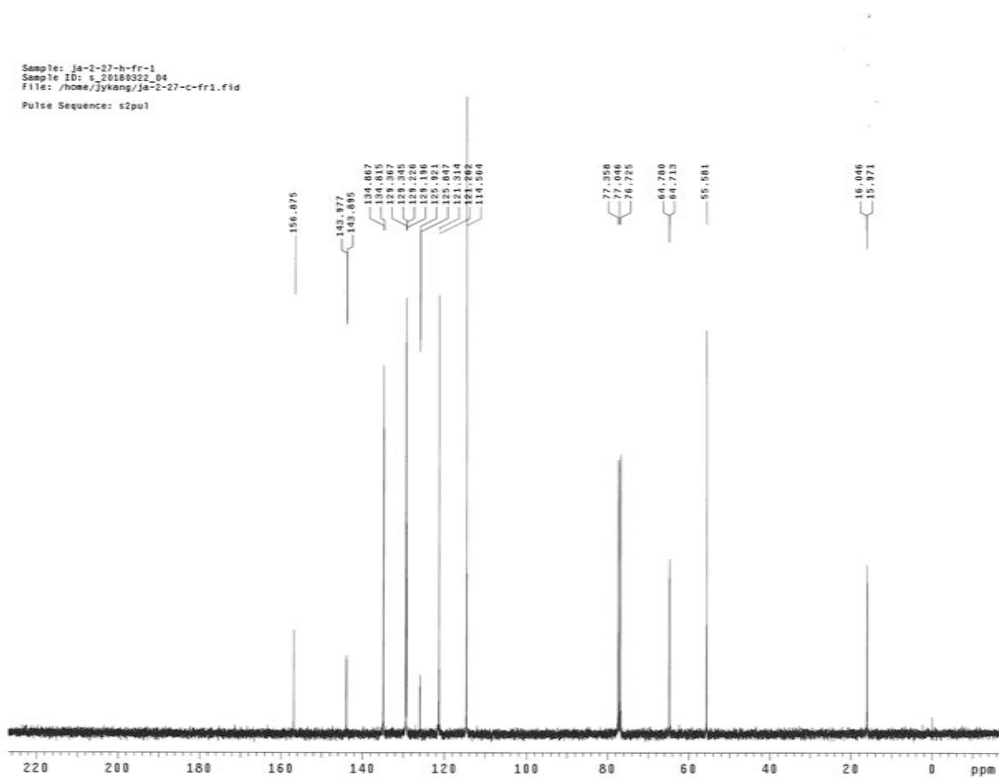
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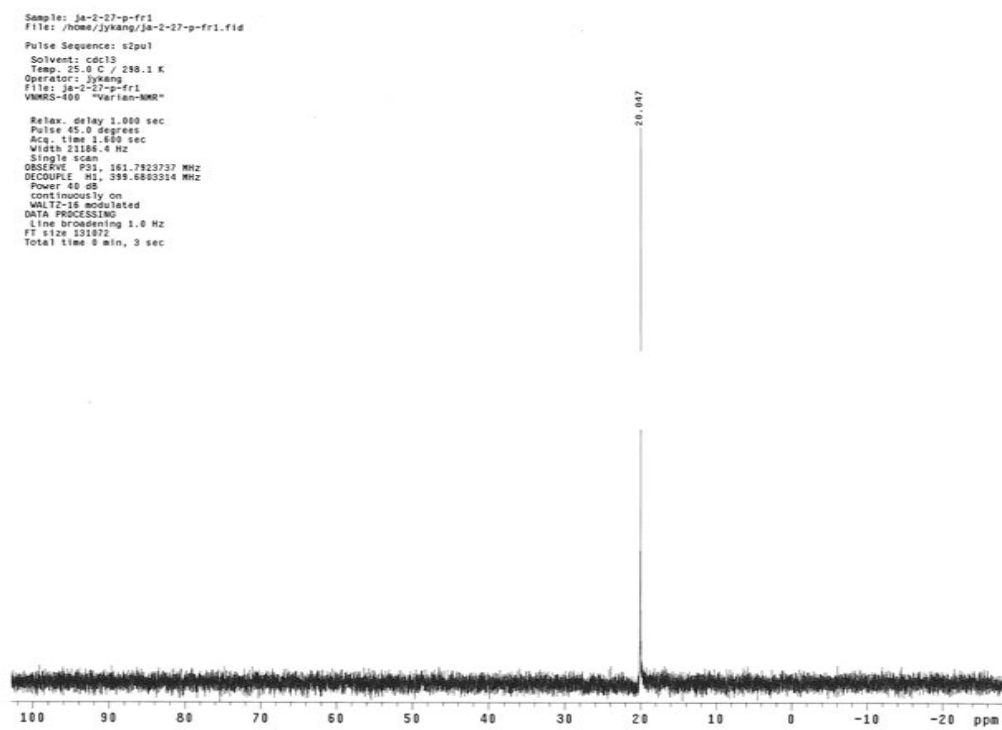
^1H NMR (400 MHz) in CDCl_3  ^{13}C NMR (100.5 MHz) in CDCl_3 

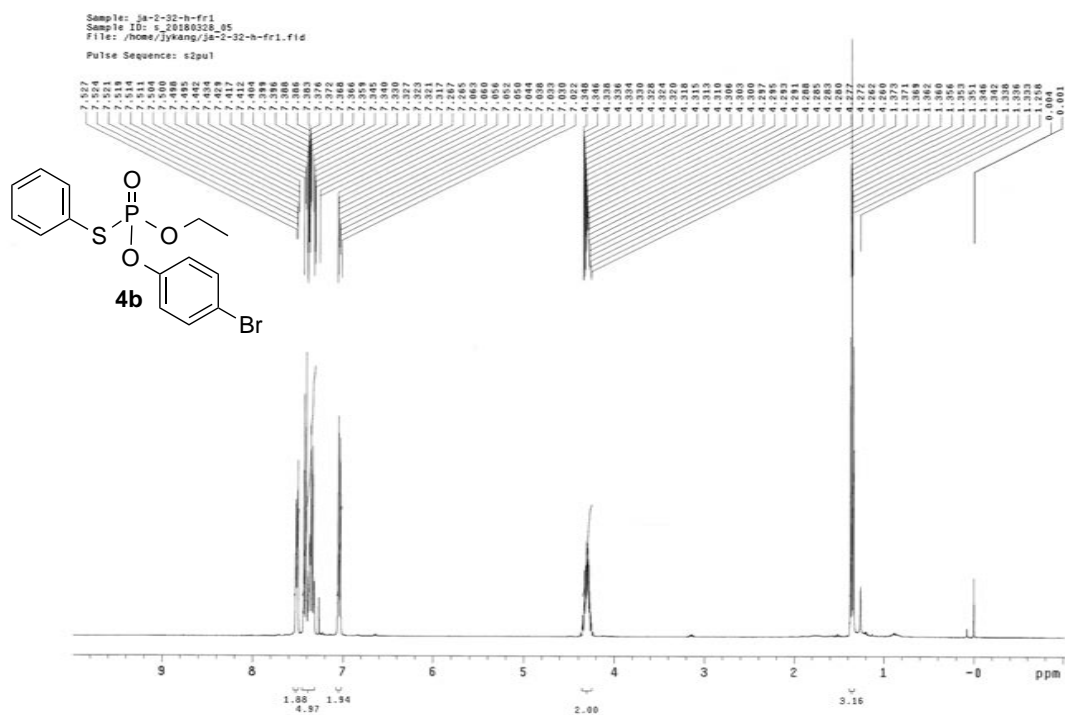
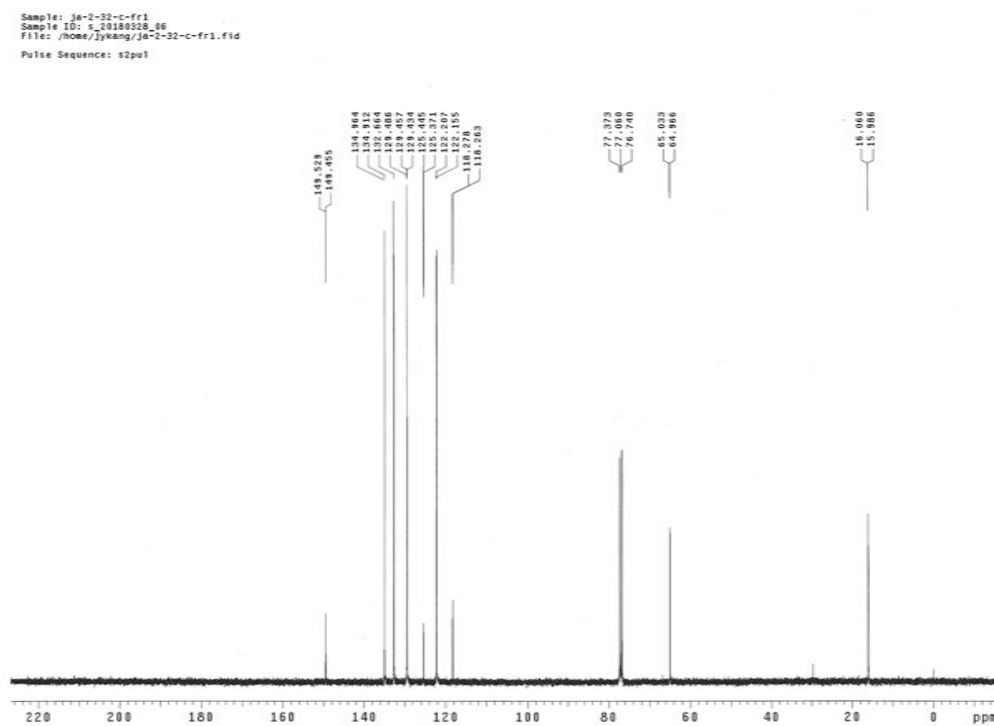
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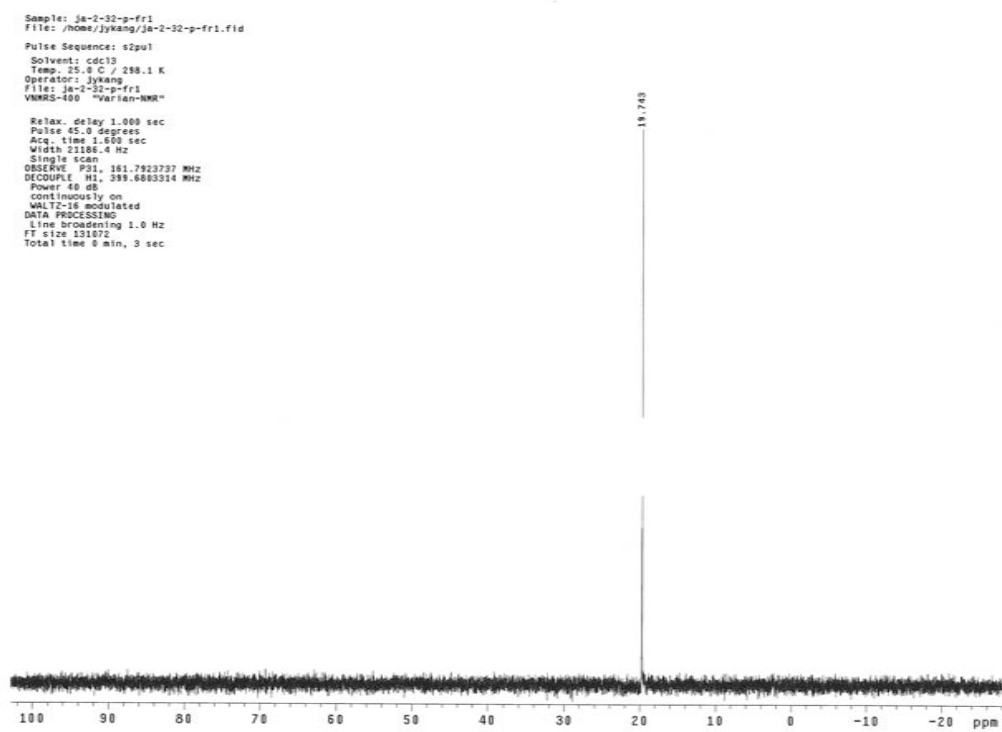
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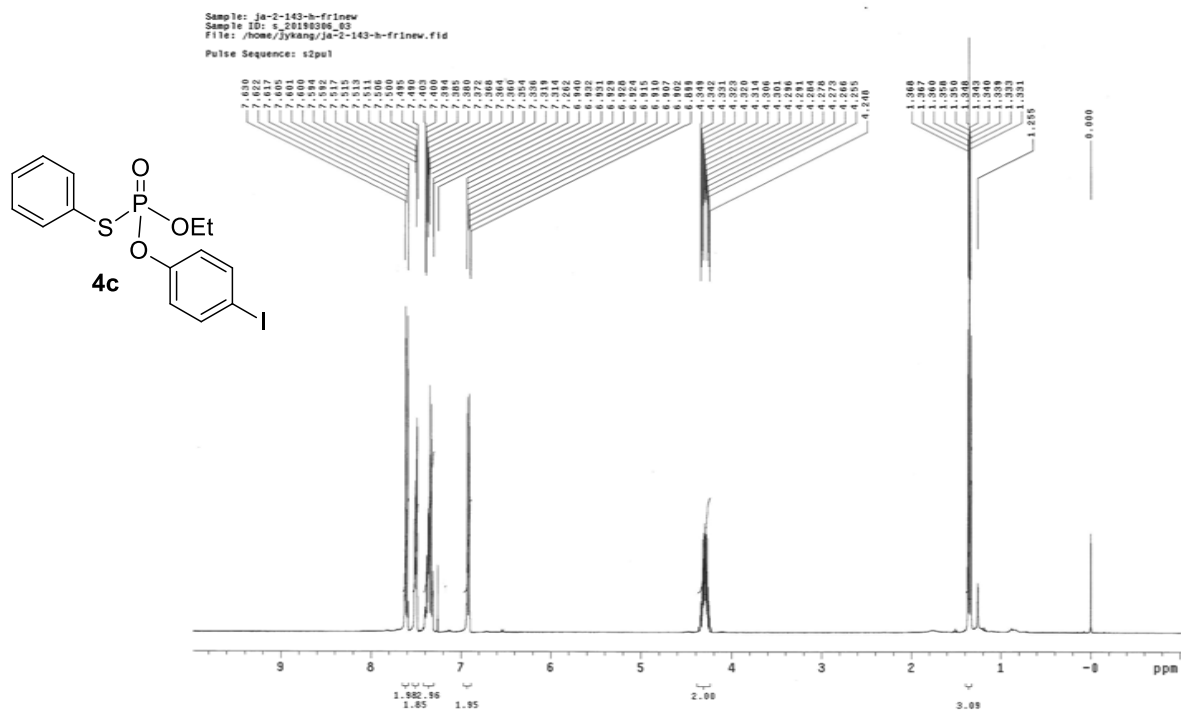
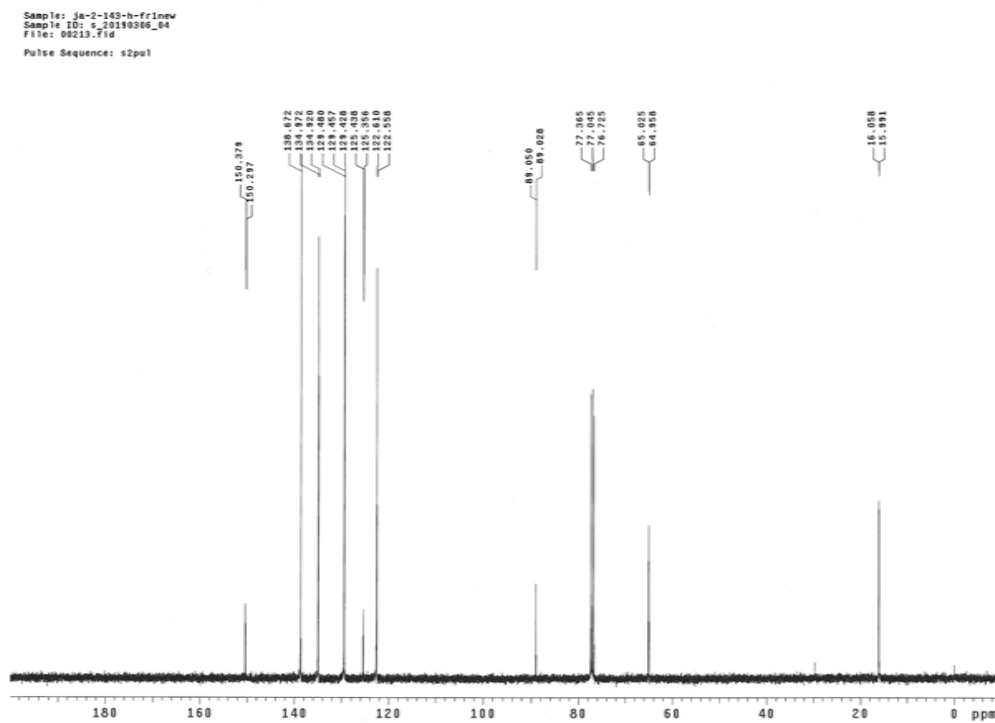
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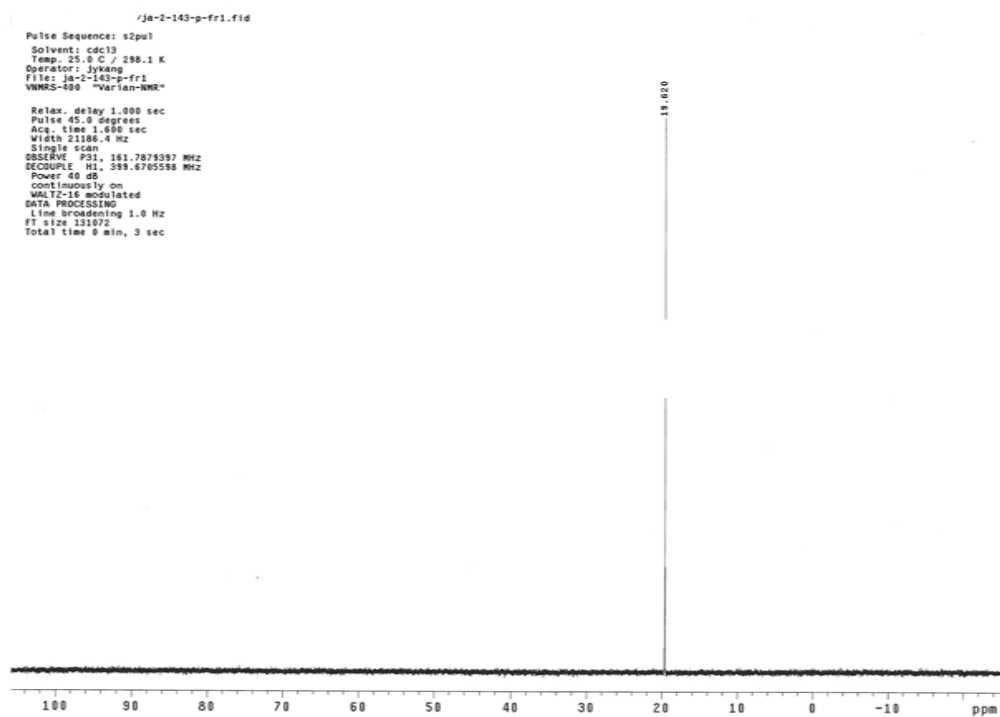
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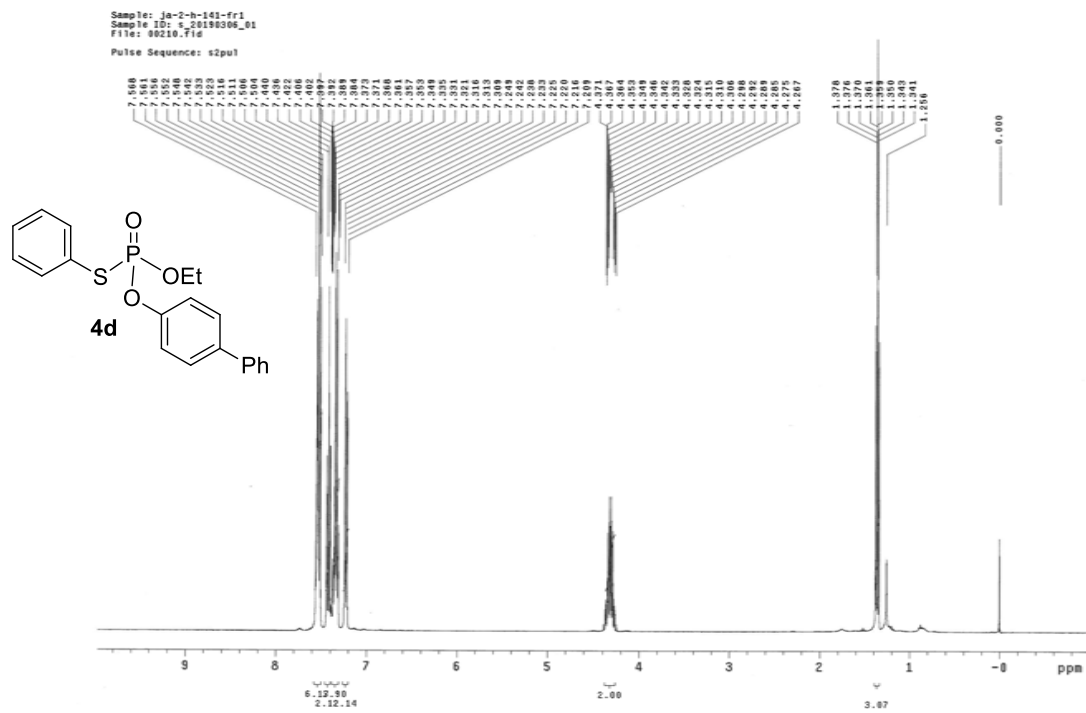
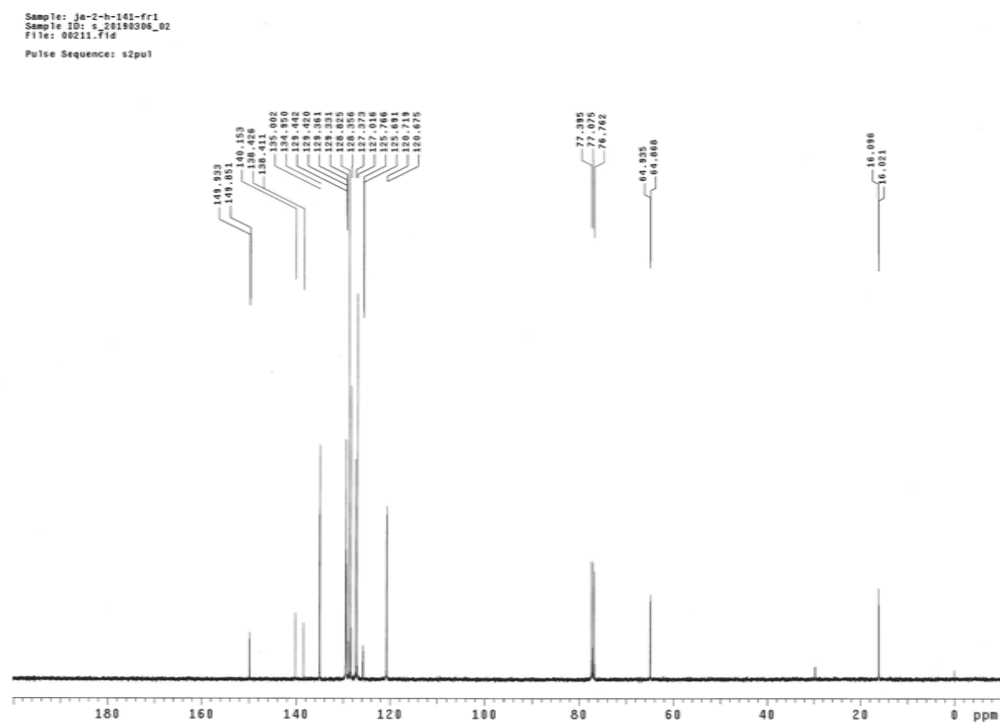
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^{31}P NMR (162 MHz) in CDCl_3 

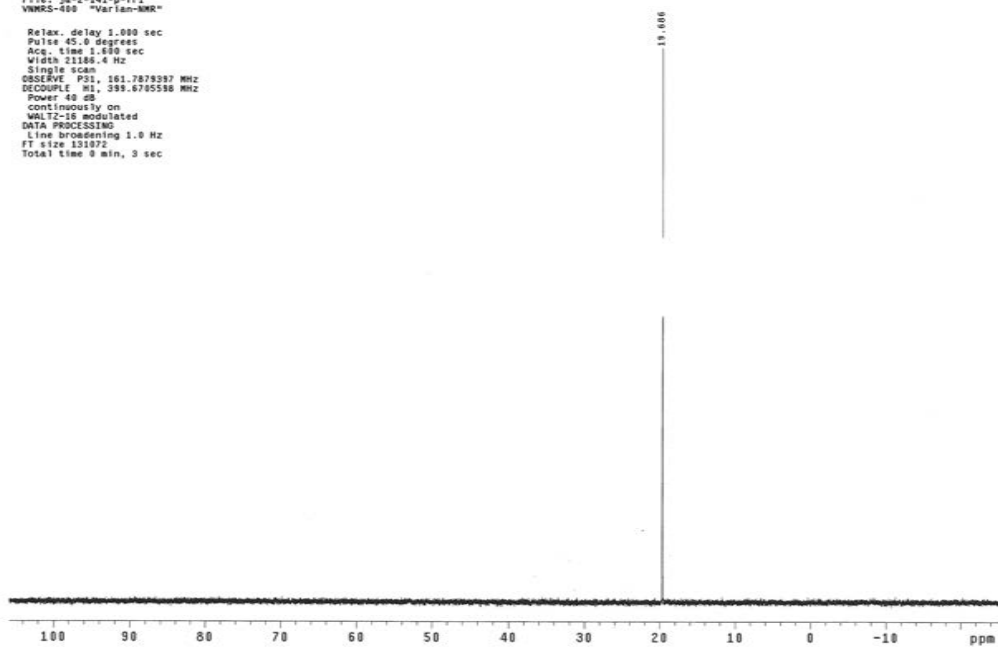
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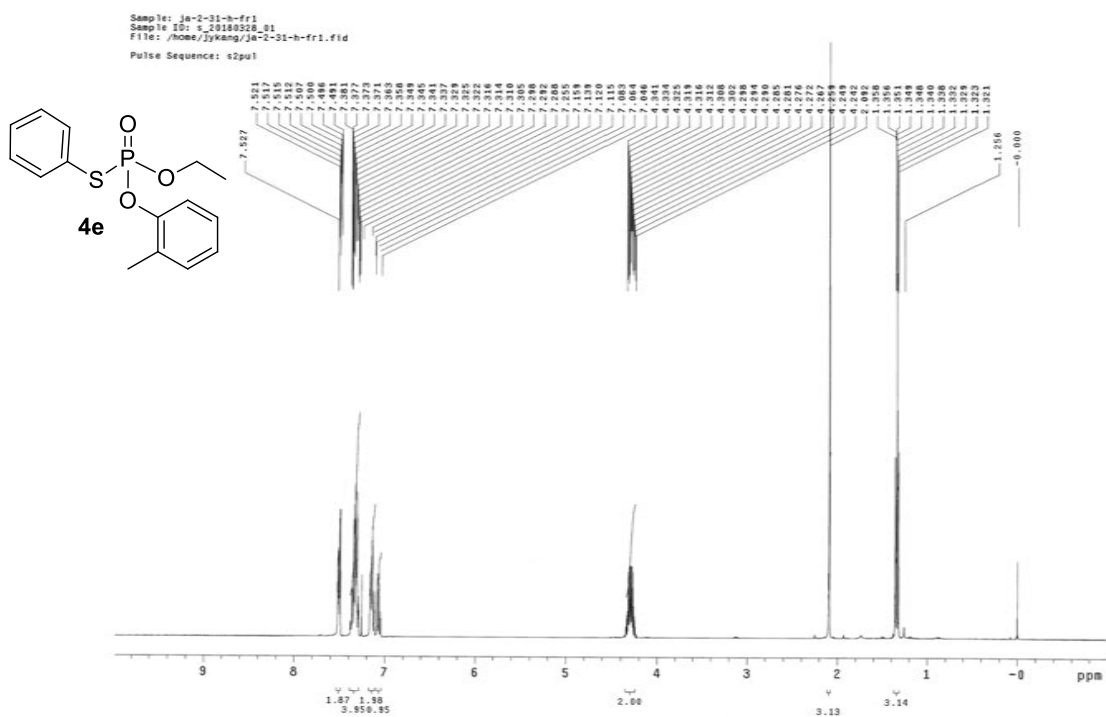
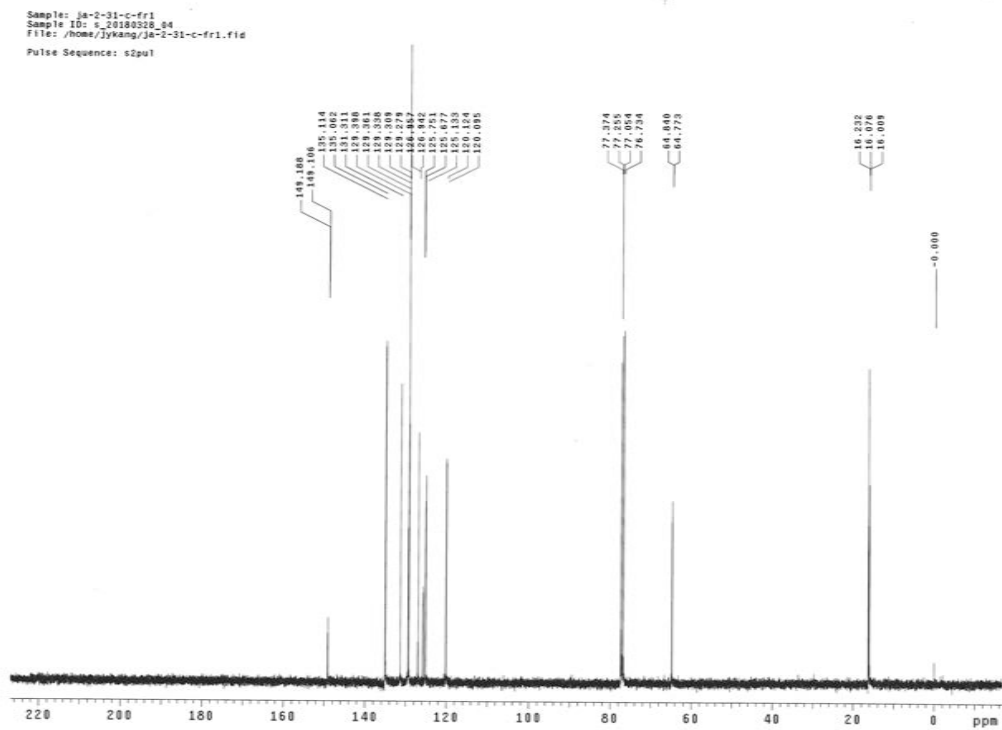
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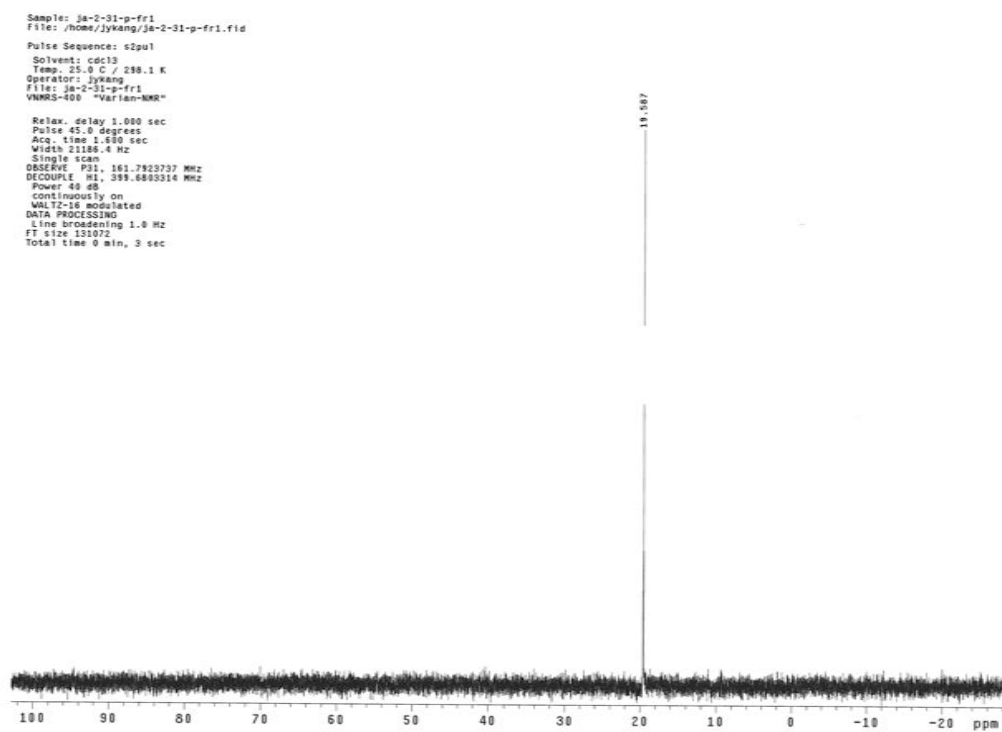
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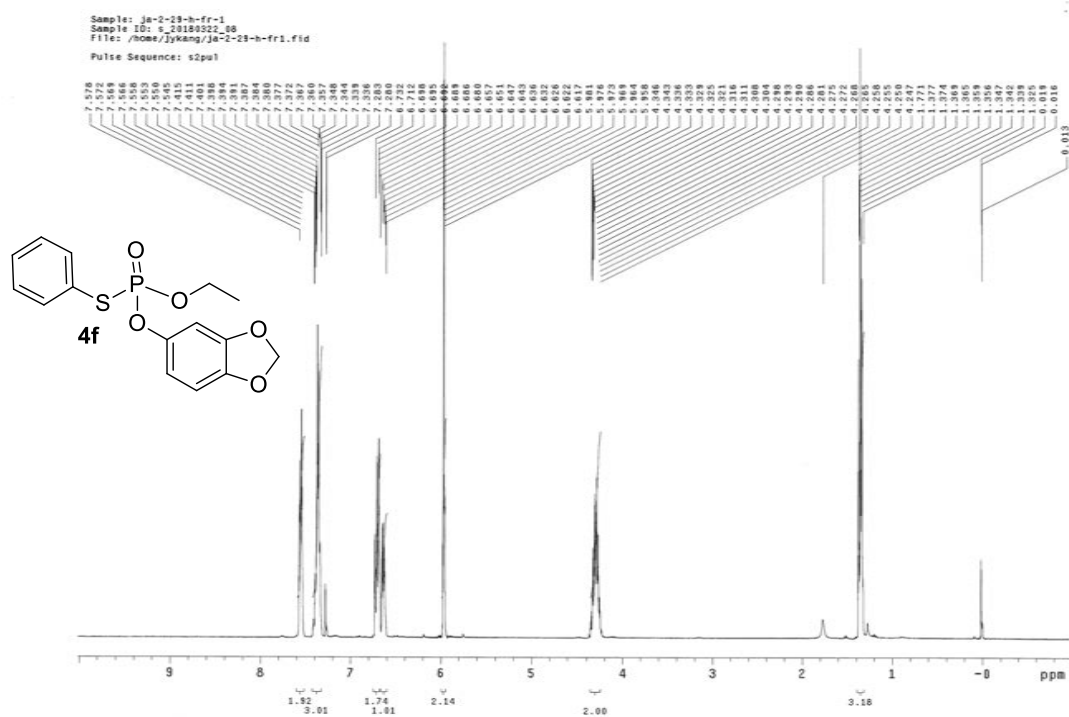
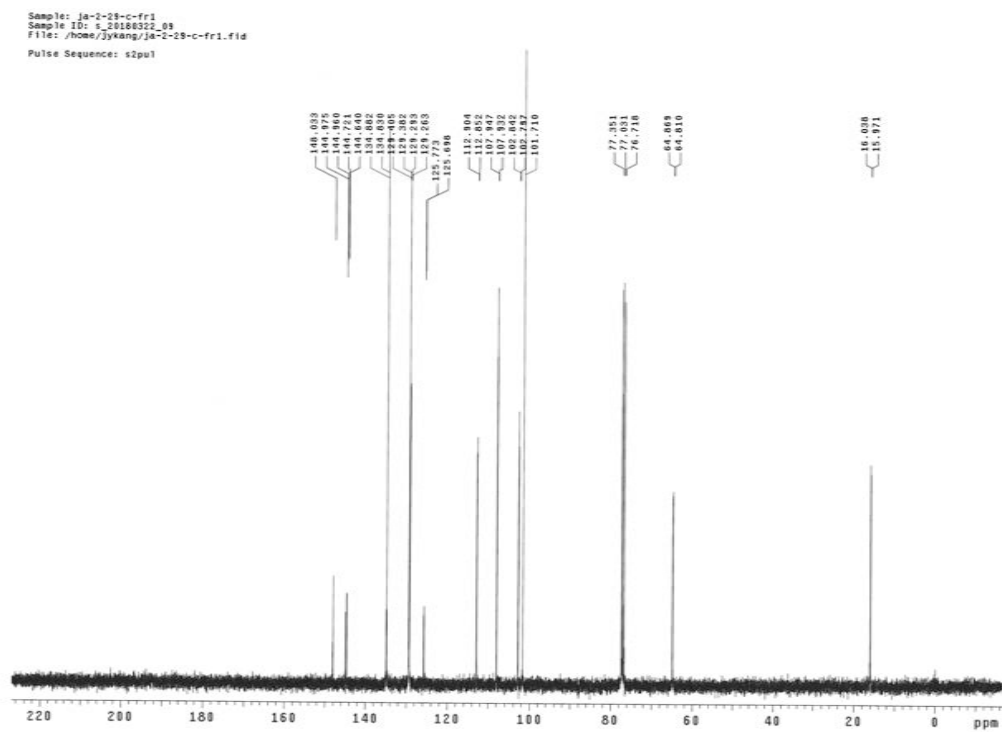
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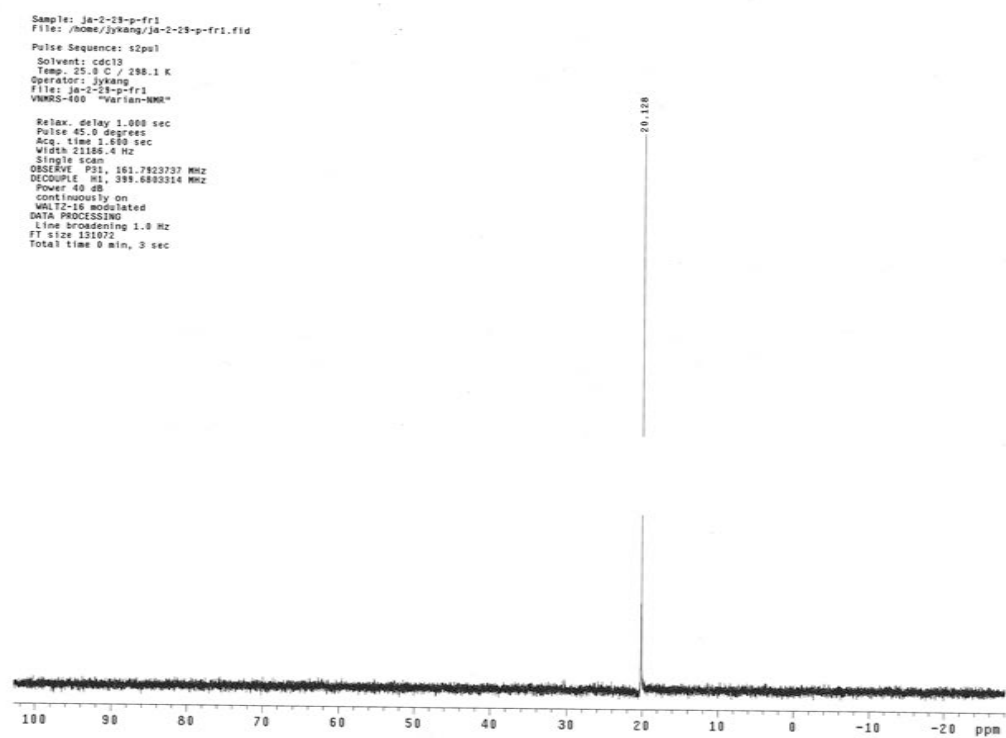
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Pulse Sequence: s2pu1
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: jywang
File: ja-2-141-p-fr1
VMS-100 "Varian-Max"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 2186.4 Hz
Single scan
OBSERVE P31, 161.7879997 MHz
DECOUPLE H1, 398.6795588 MHz
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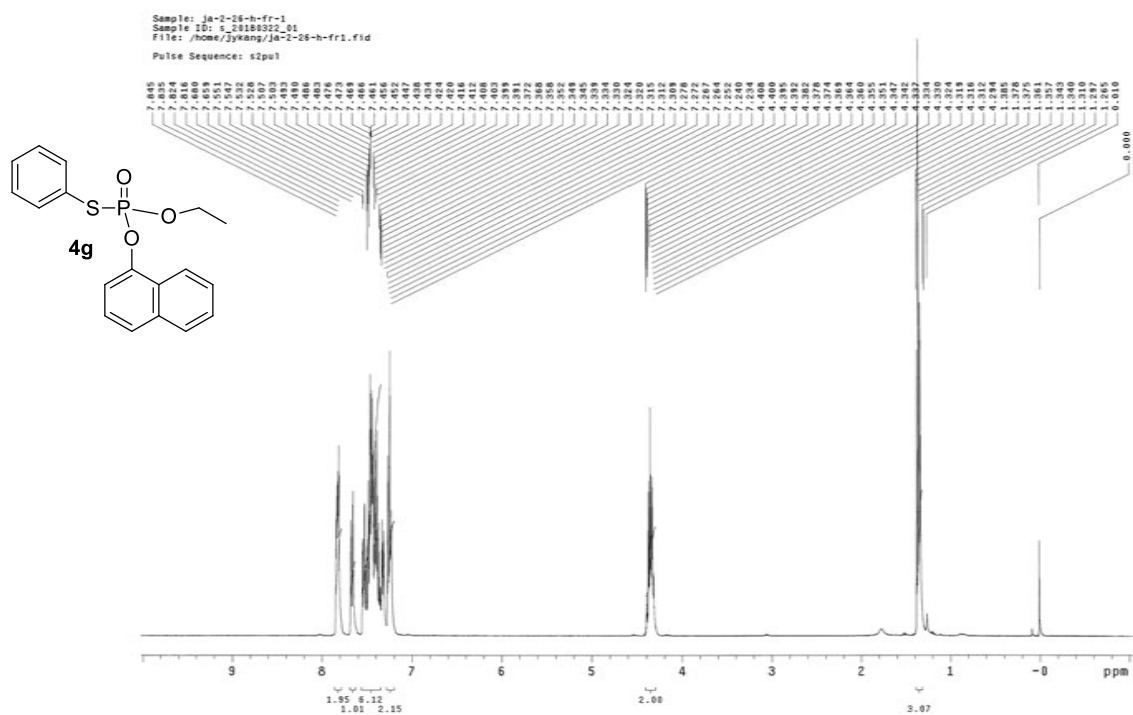
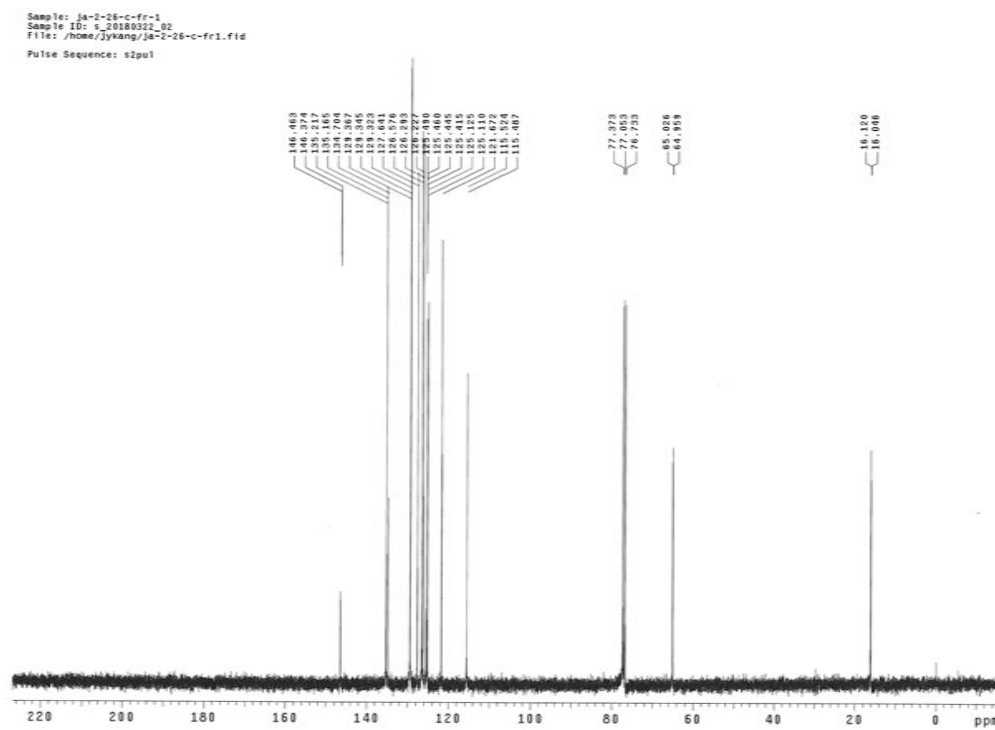


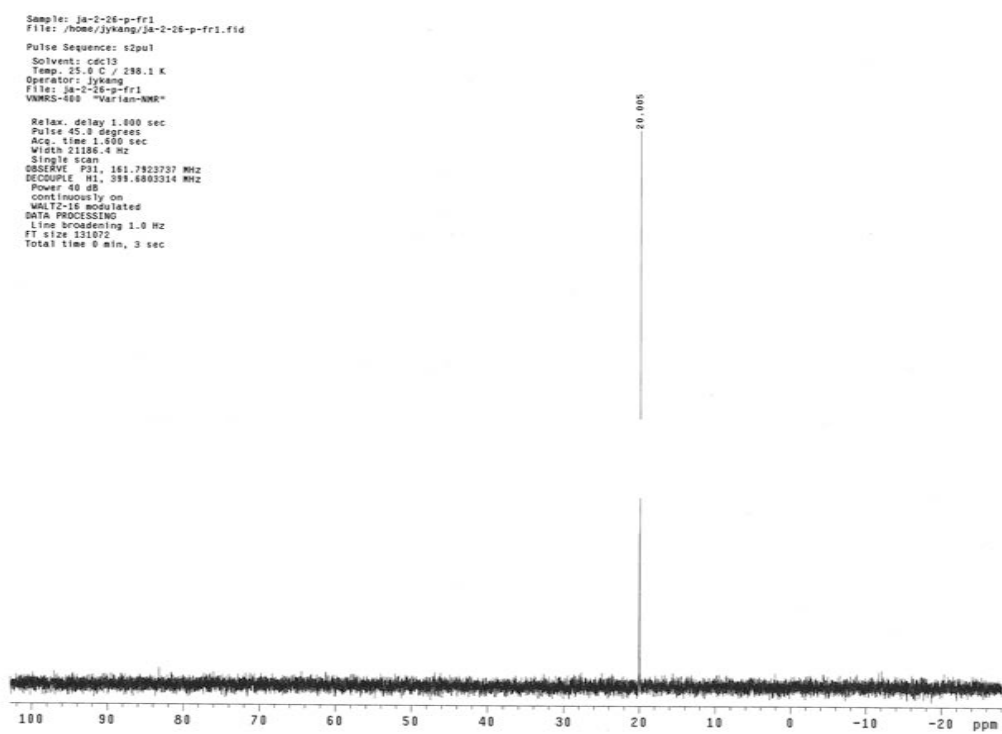
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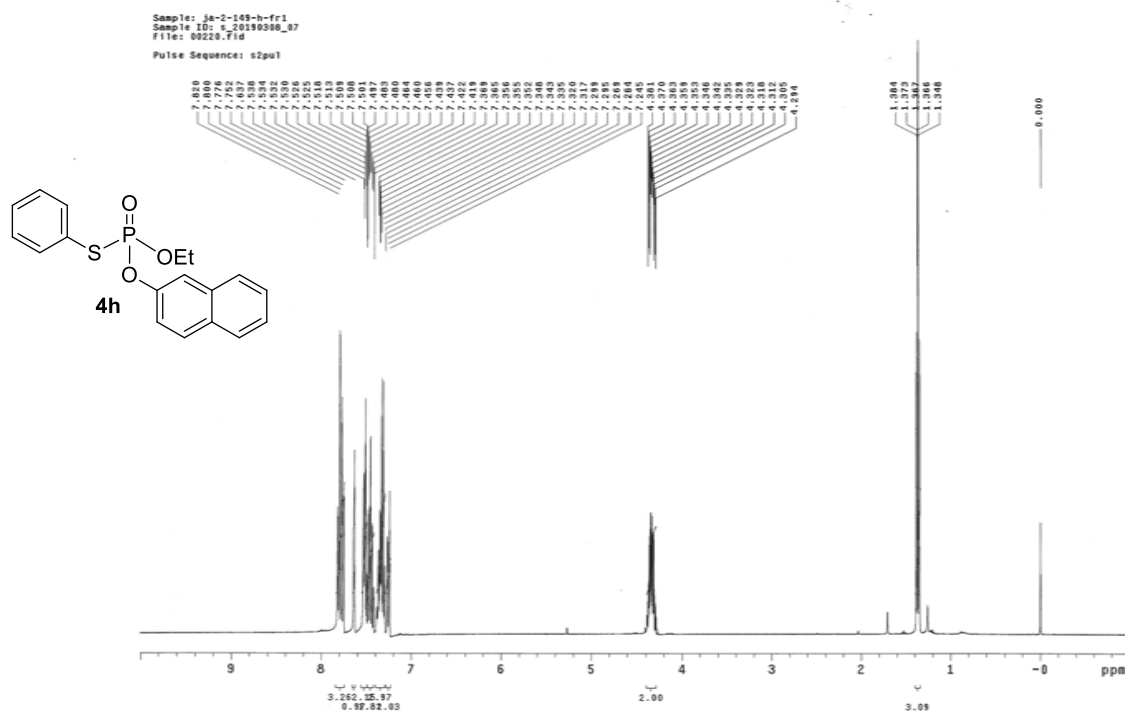
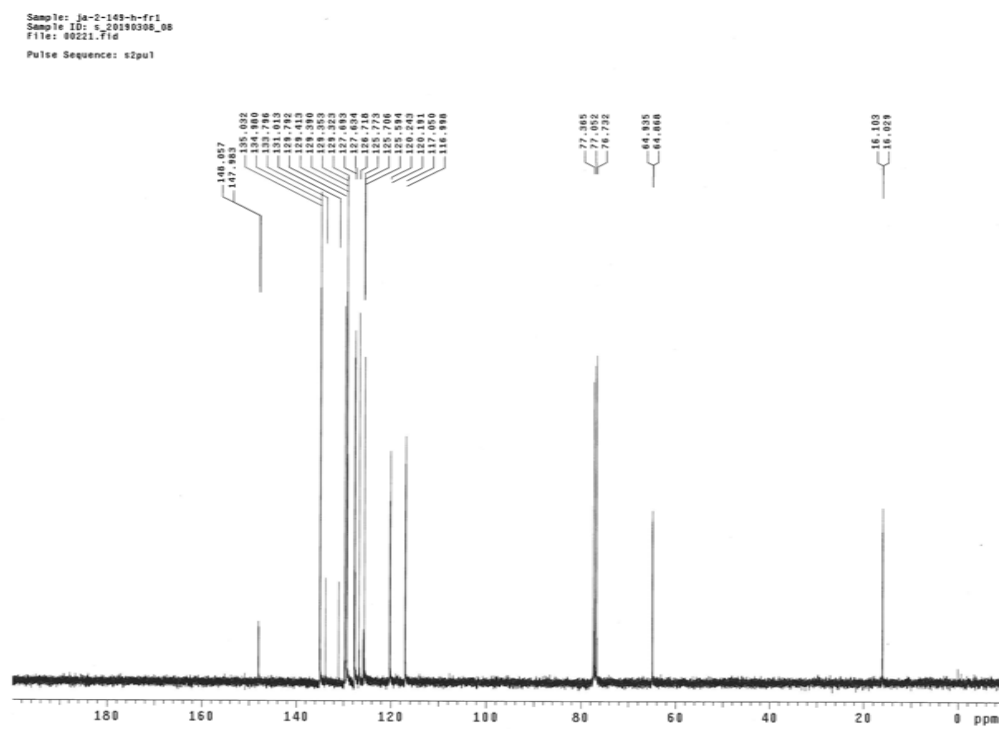
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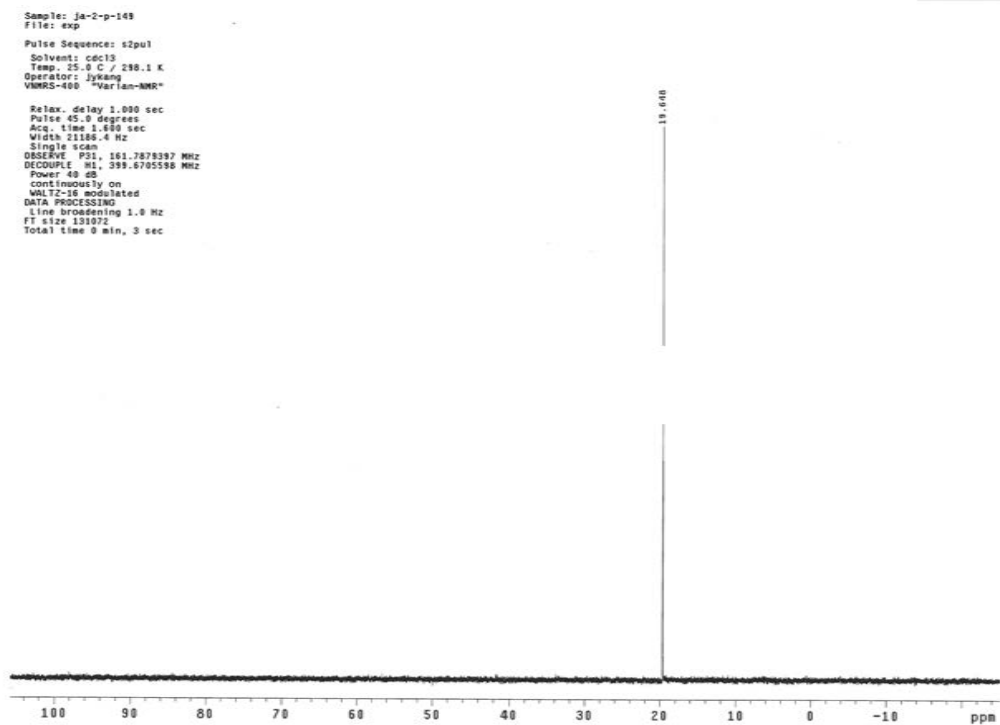
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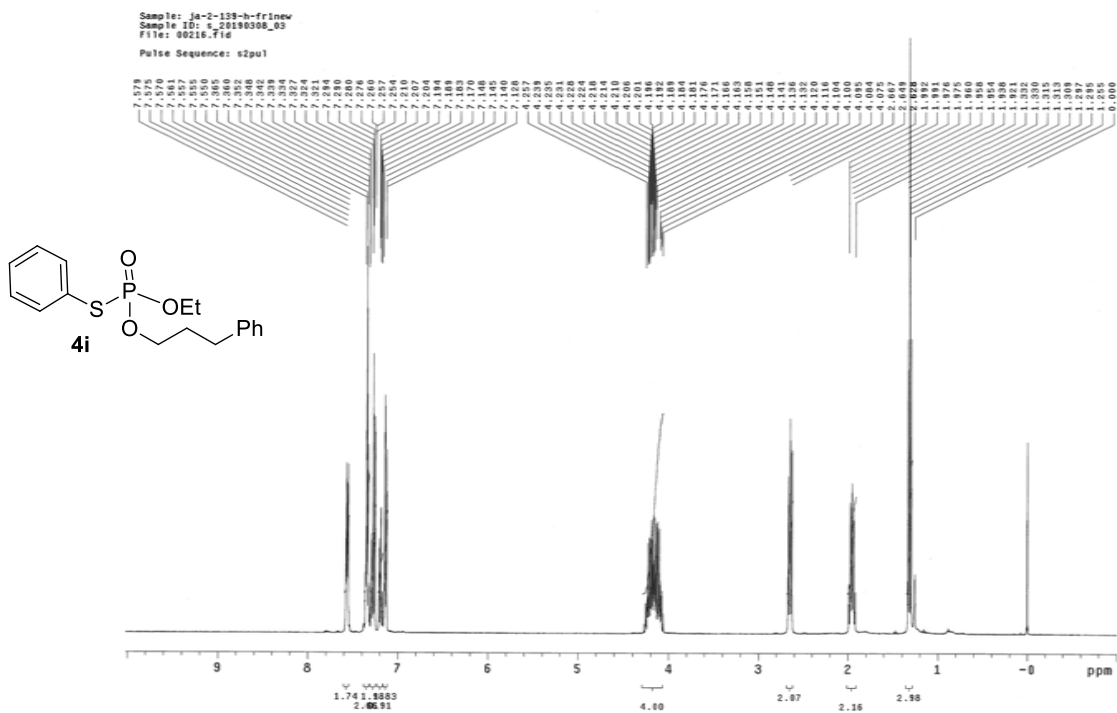
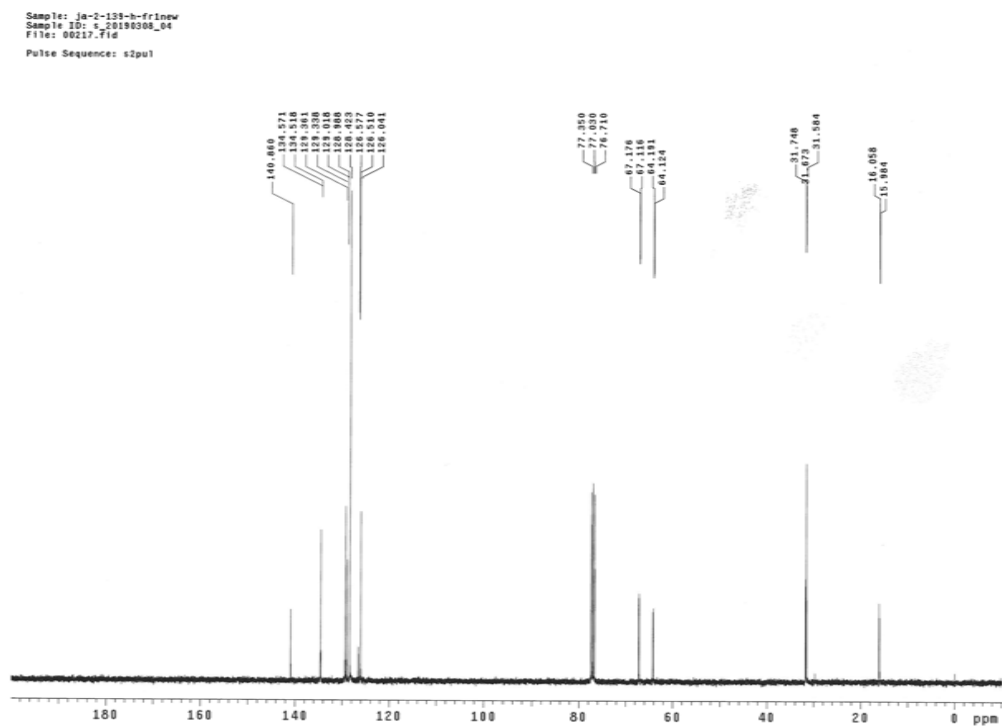
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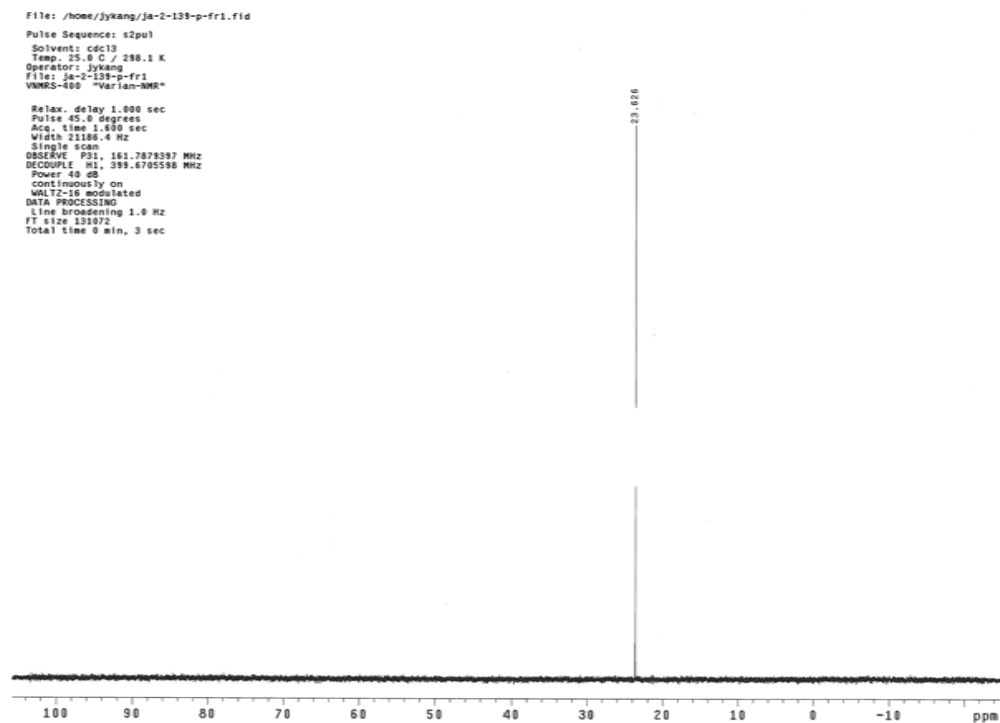
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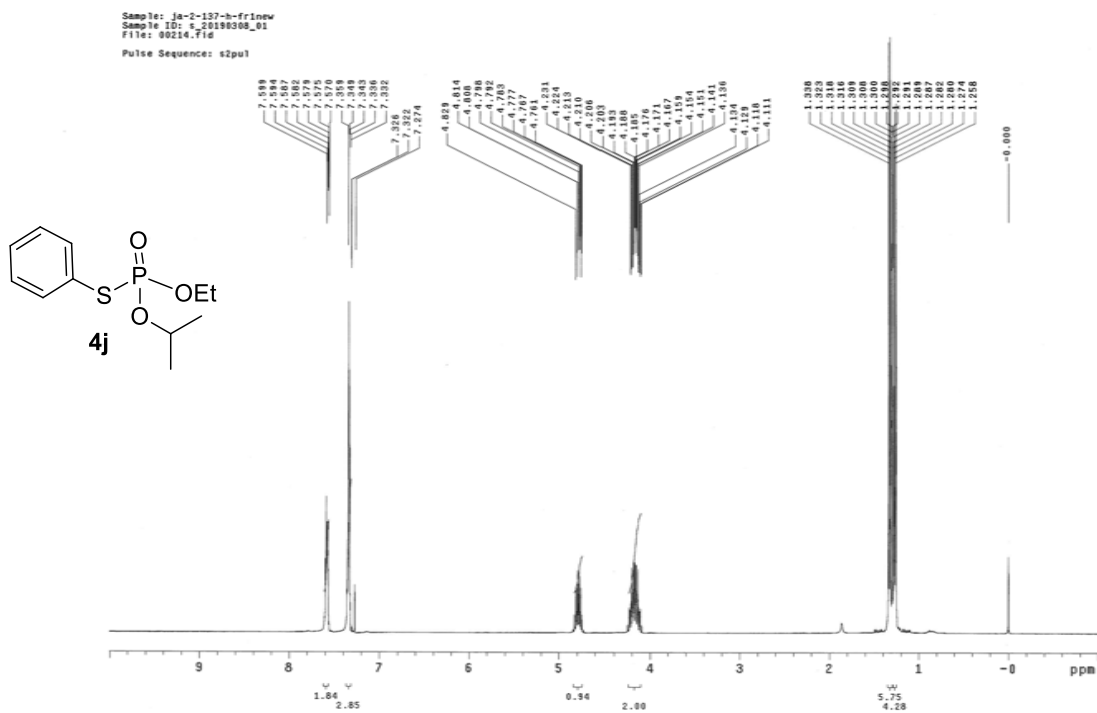
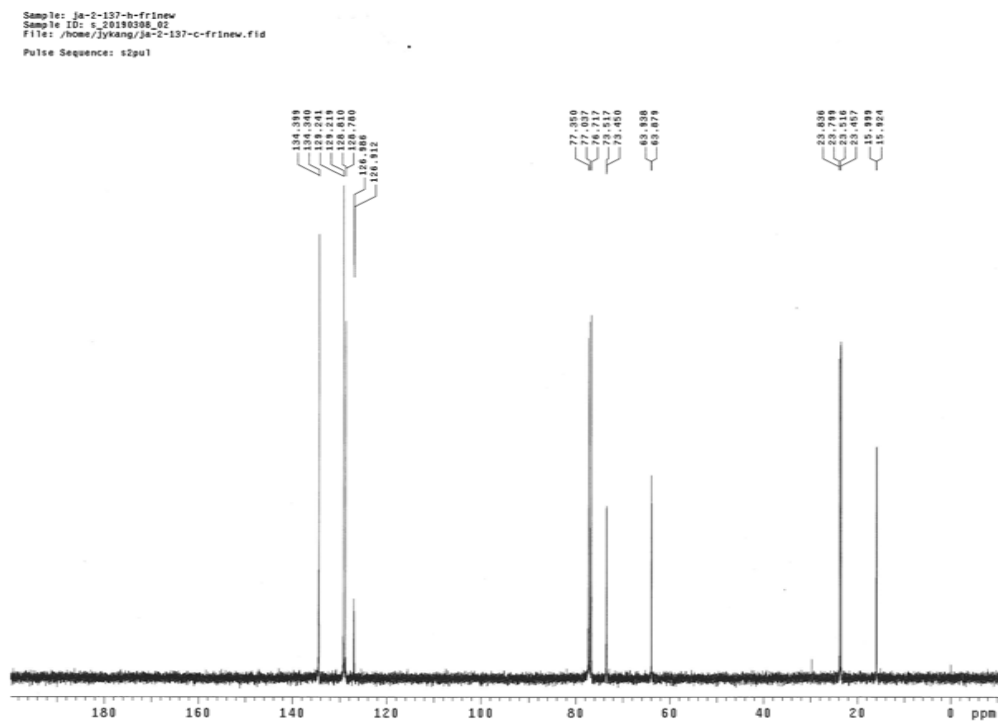
^{31}P NMR (162 MHz) in CDCl_3 

^1H NMR (400 MHz) in CDCl_3  ^{13}C NMR (100.5 MHz) in CDCl_3 

^{31}P NMR (162 MHz) in CDCl_3 

^1H NMR (400 MHz) in CDCl_3  ^{13}C NMR (100.5 MHz) in CDCl_3 

^{31}P NMR (162 MHz) in CDCl_3 

^1H NMR (400 MHz) in CDCl_3  ^{13}C NMR (100.5 MHz) in CDCl_3 

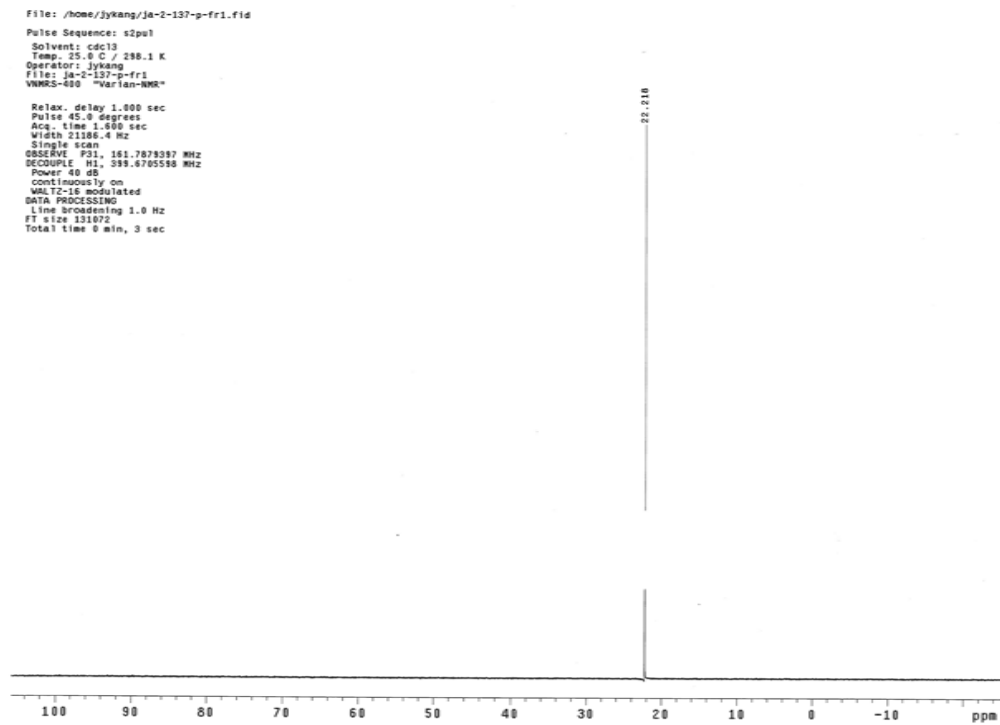
^{31}P NMR (162 MHz) in CDCl_3 



Figure S1. Flow setup for the synthesis of **3**, **4**