

Base-Controlled Fe(Pc)-Catalyzed Aerobic Oxidation of Thiols for the Synthesis of S-S and S-P(O) Bond

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

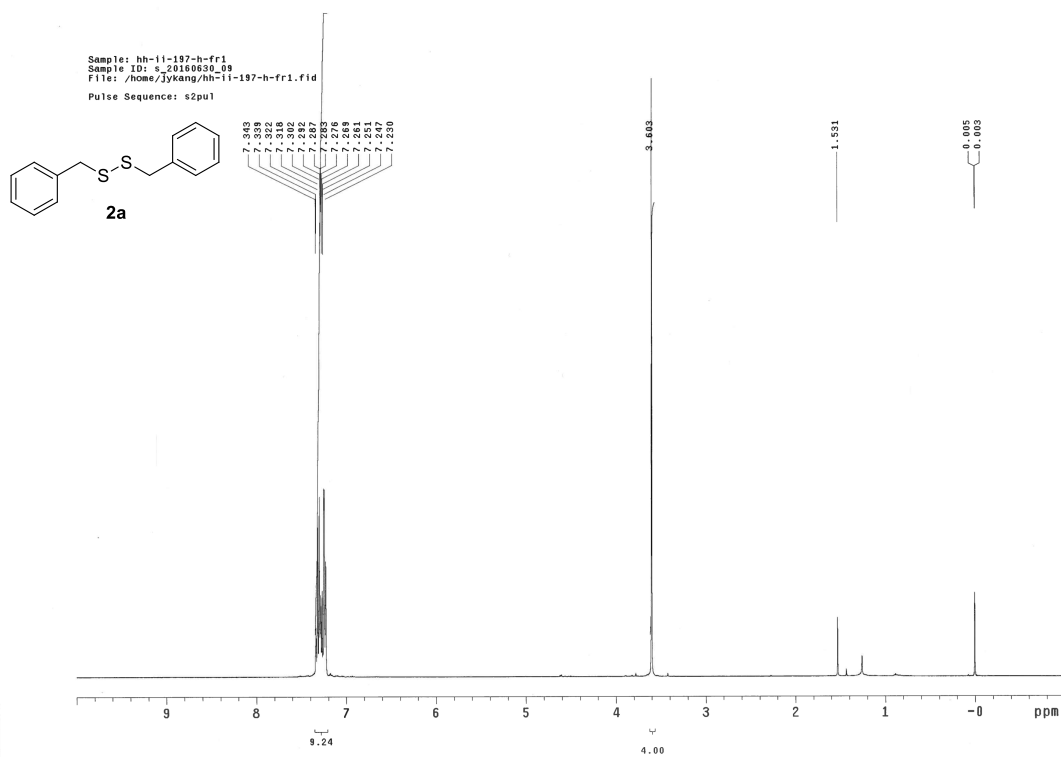
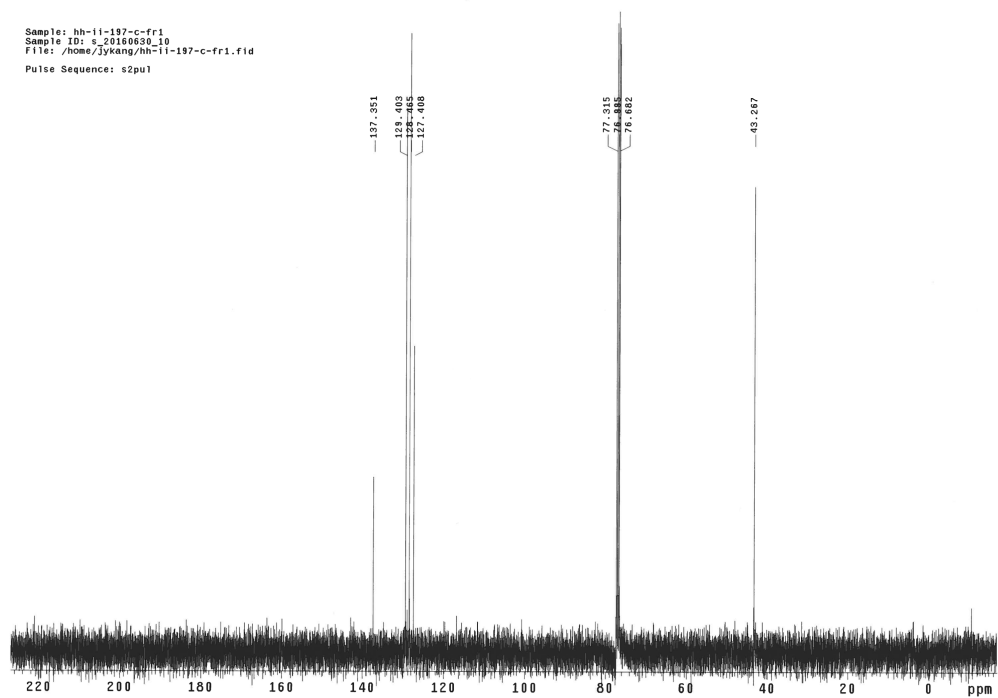
^a Department of Chemistry and Biochemistry, University of Nevada Las Vegas, 4505 S.
Maryland Parkway, Las Vegas, Nevada, 89154-4003, United States

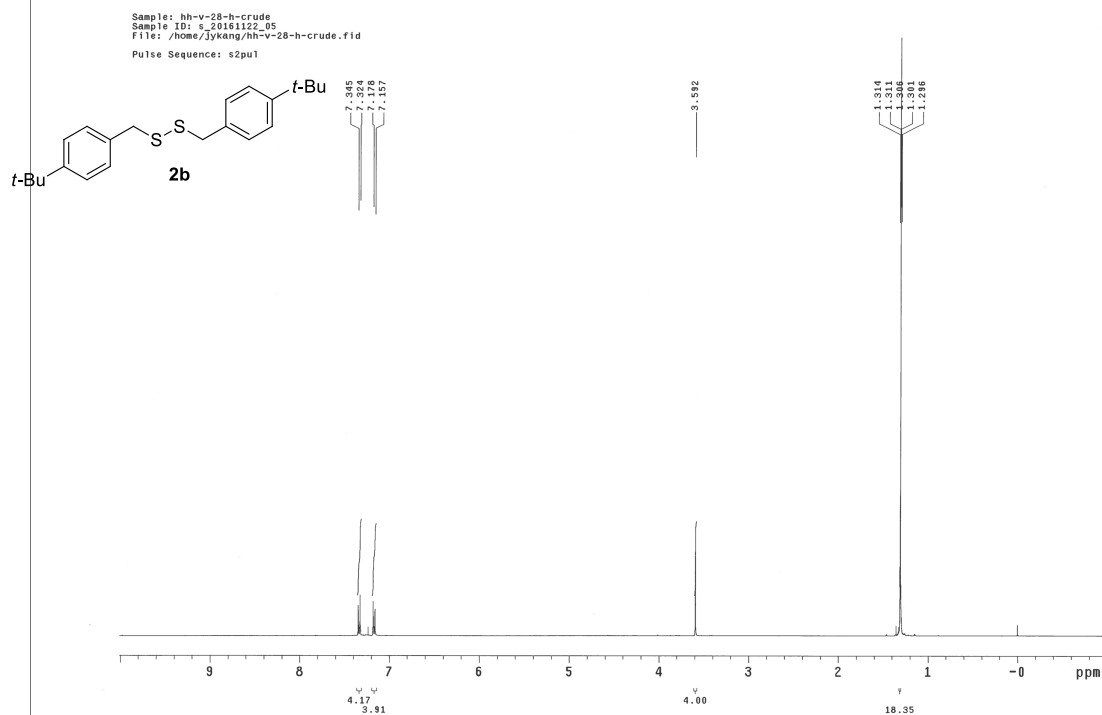
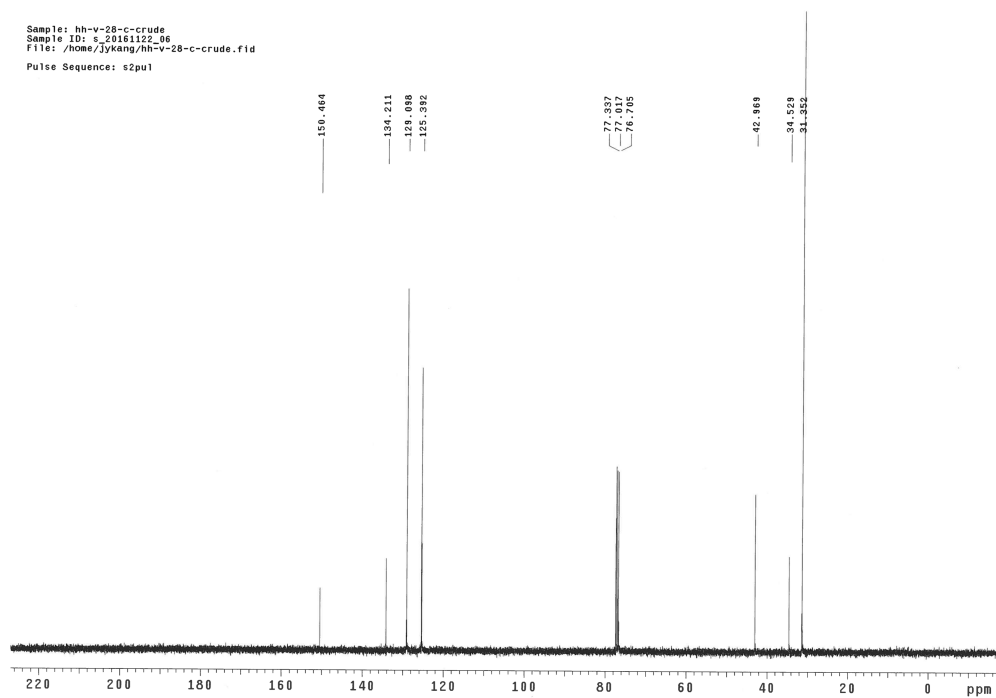
^b Department of Applied Chemistry, College of Chemistry and Molecular Engineering, Nanjing
Tech University, No. 30 Puzhu Road (S), Nanjing 211816, People's Republic of China

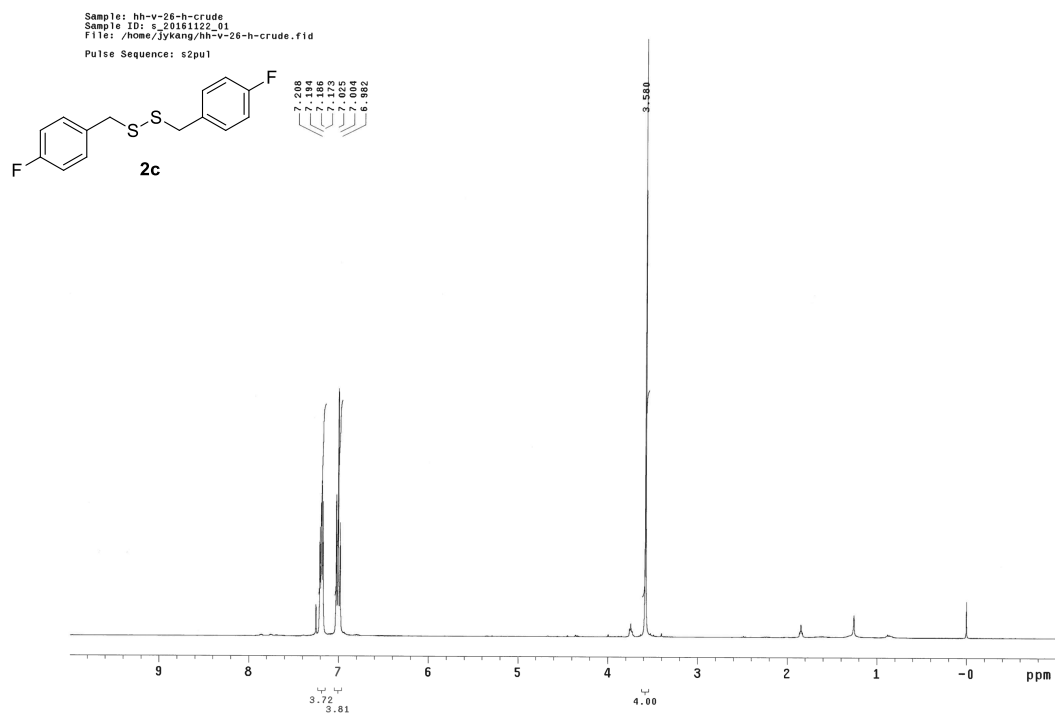
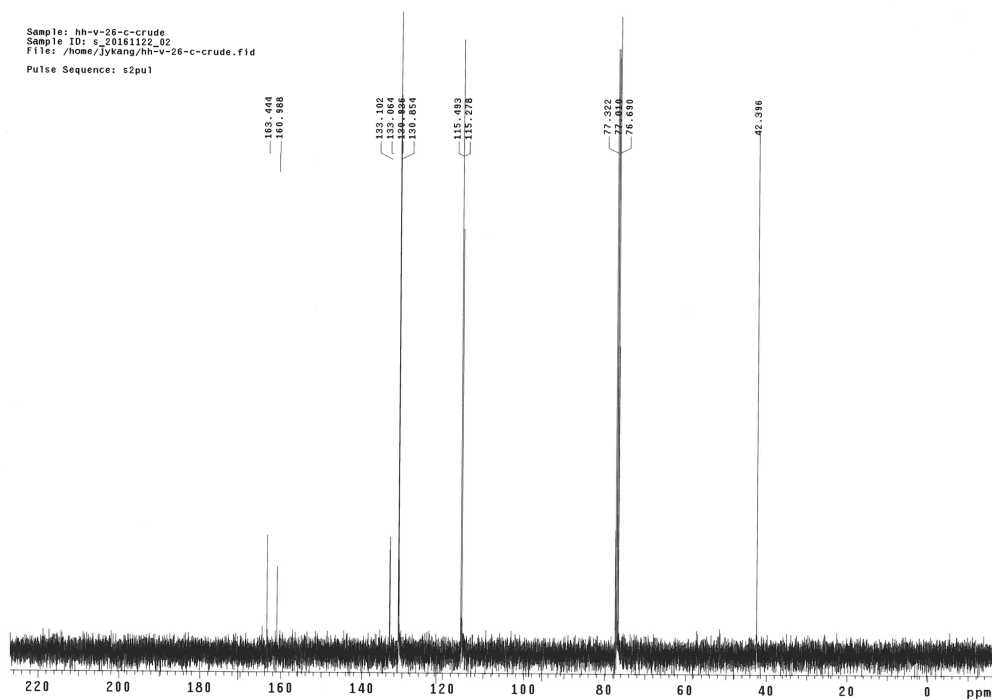
E-mail: junyong.kang@unlv.edu.

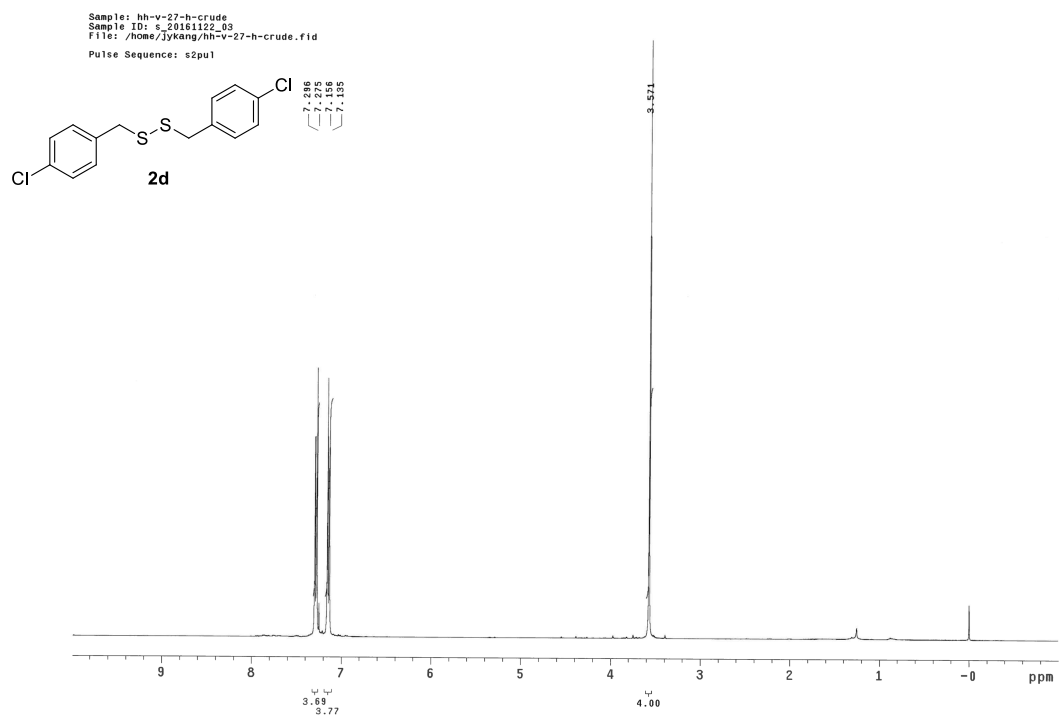
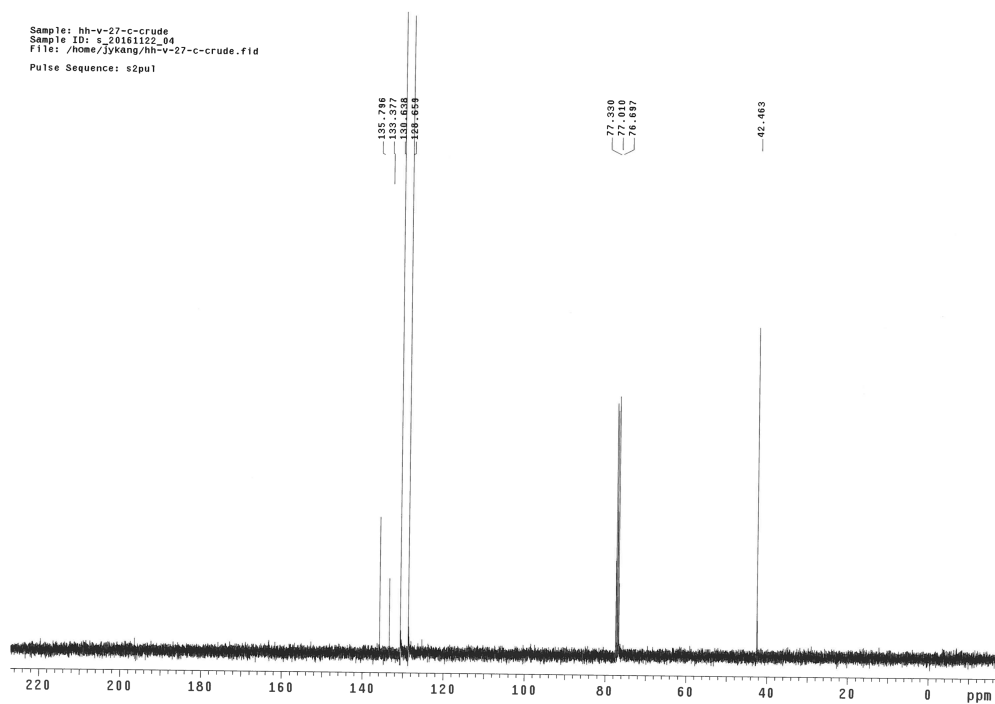
Supporting Information

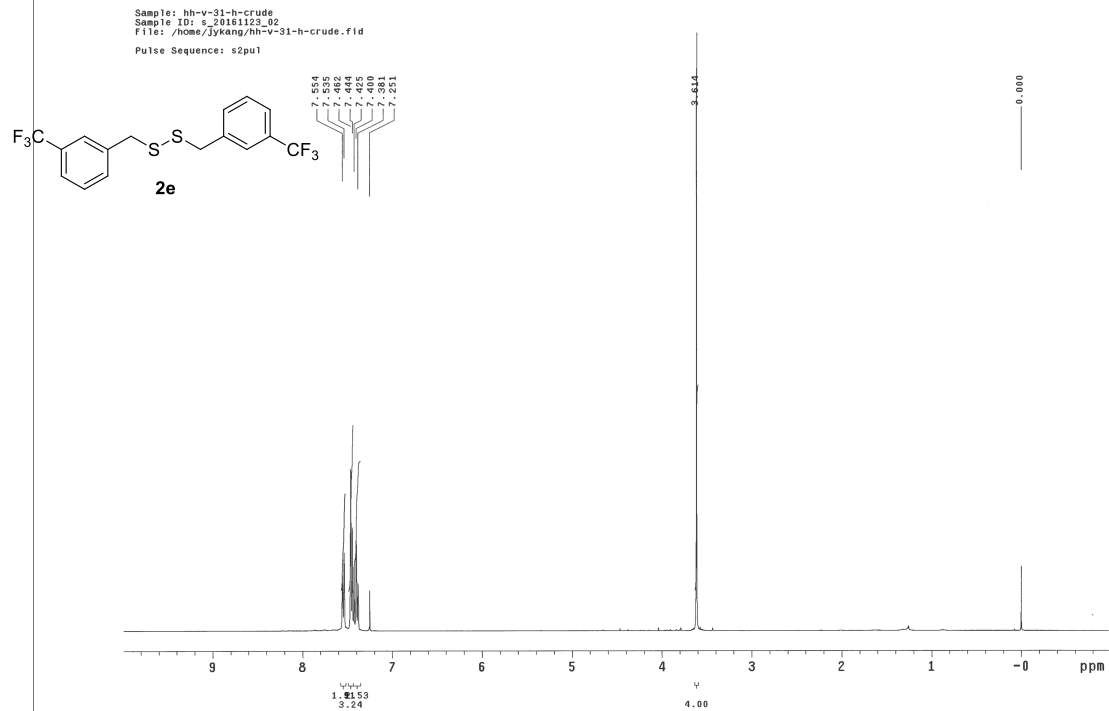
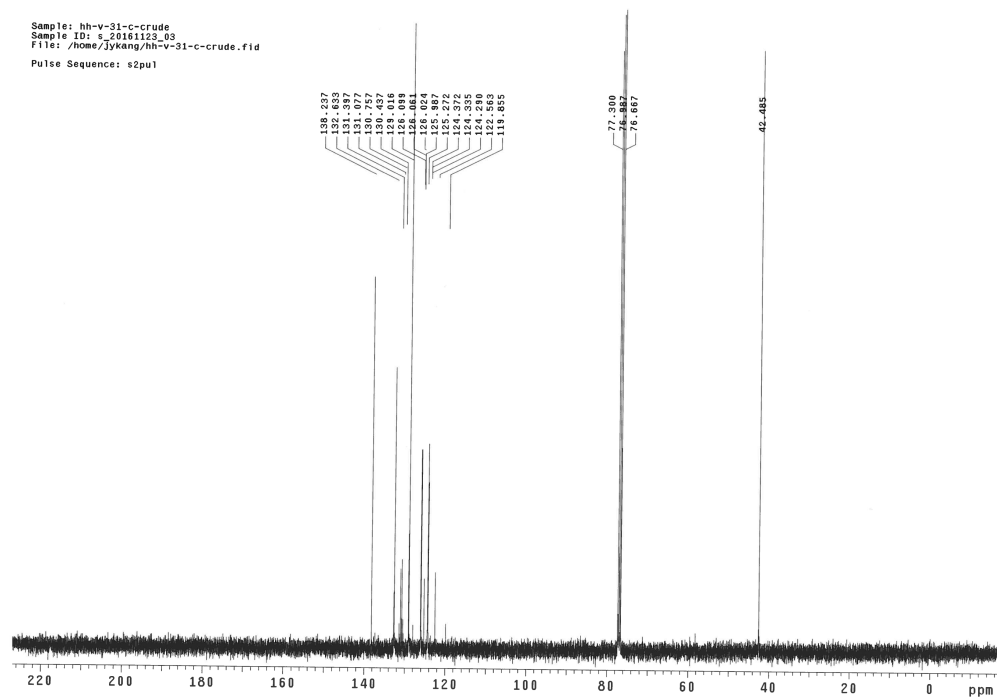
¹H and ¹³C NMR Spectra.....S2-S28

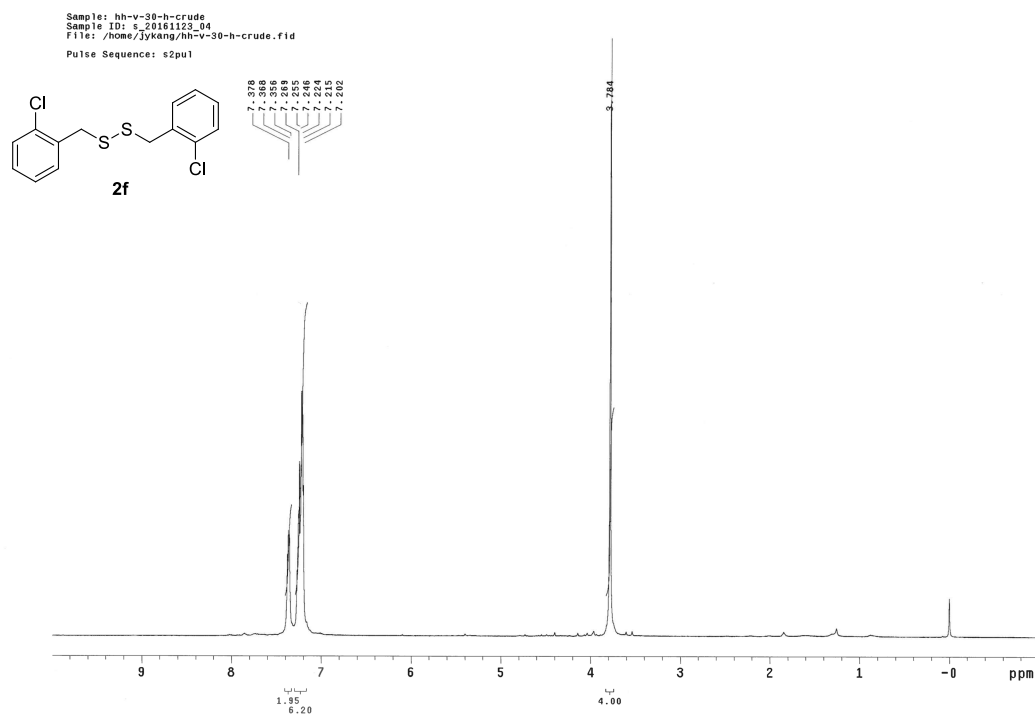
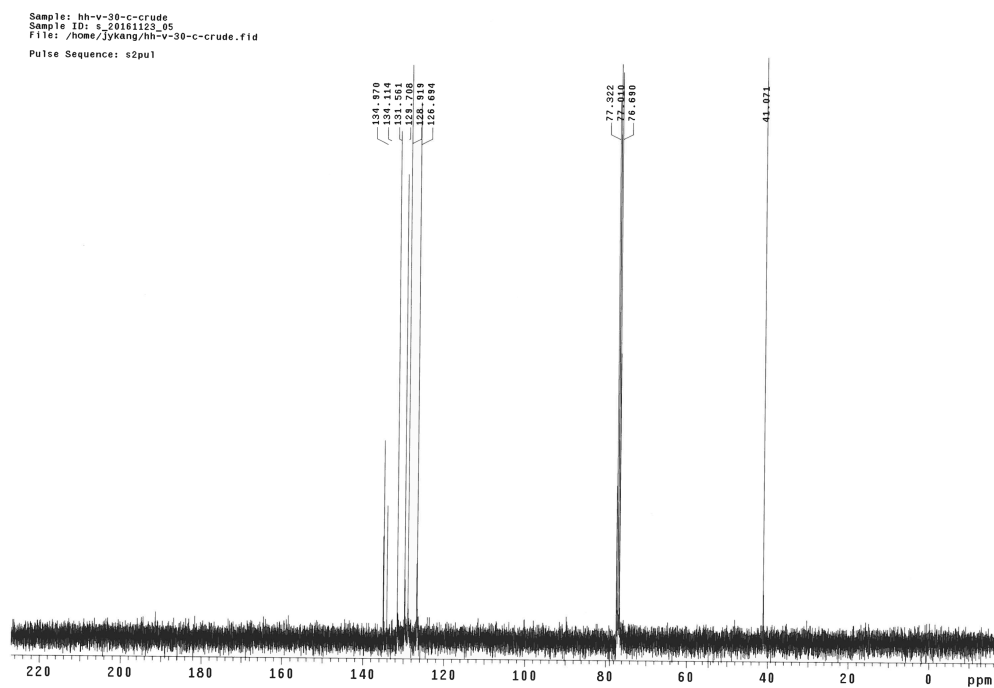
^1H NMR (400 MHz) in CDCl_3  **^{13}C NMR (100.5 MHz) in CDCl_3** 

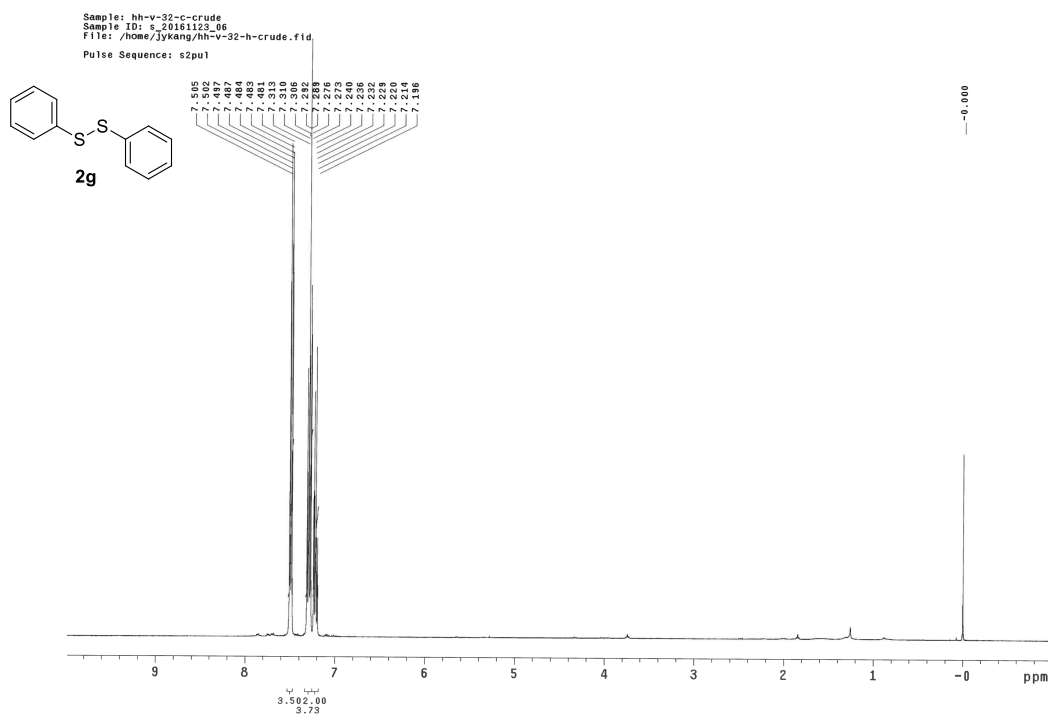
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^1H NMR (400 MHz) in CDCl_3 

Sample: hh-v-40-h-crude
Sample ID: s_20161126_13
File: /home/jykang/hh-v-40-h-crude.fid
Pulse Sequence: s2pu1

Cc1ccc(SSc2ccc(C)cc2)cc1
2h

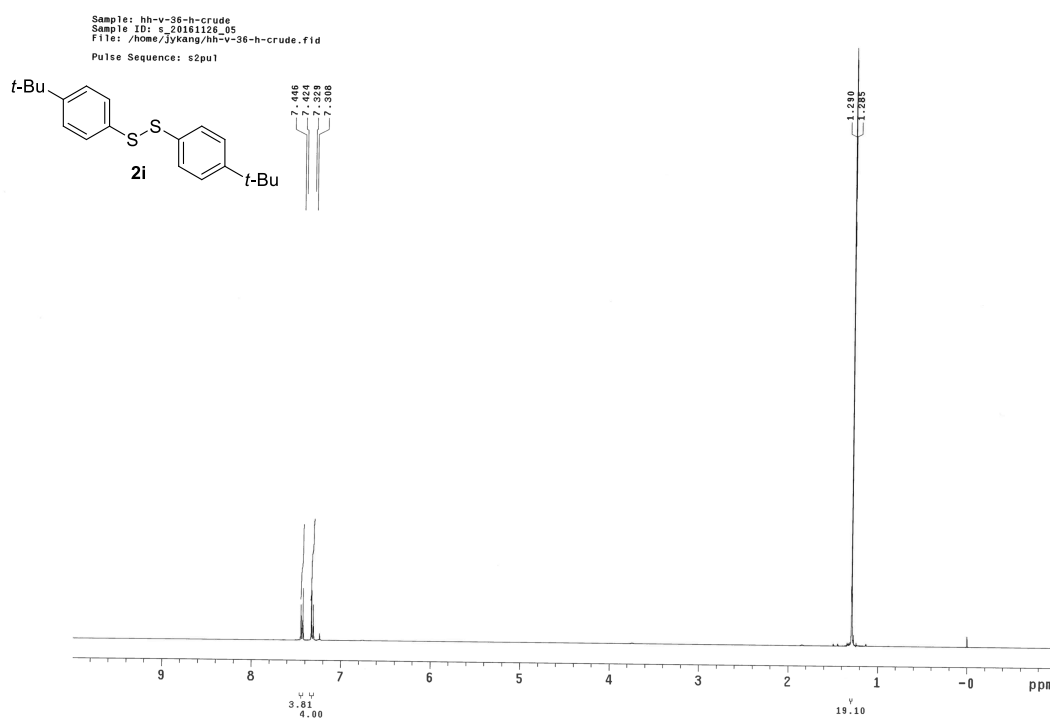
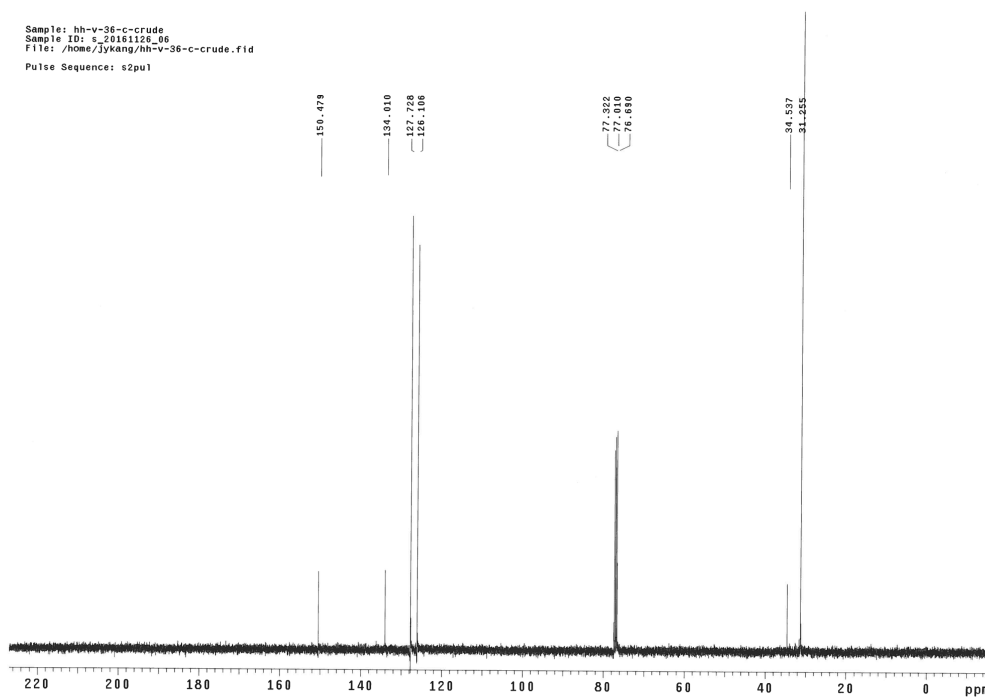
7.384
7.383
7.382
7.381
7.261
7.260
7.259
7.258
2.967
-0.000

3.64
3.75
6.00

ppm

Sample: hh-v-40-c-crude
Sample ID: s_20181126_14
File: /home/jykang/hh-v-40-c-crude.fid
Pulse Sequence: s2pu1

137.486
135.091
134.727
126.525
77.537
77.017
76.687
21.044

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

Sample: hh-v-38-h-crude
Sample ID: s_20161126_09
File: /home/jykang/hh-v-38-h-crude.fid
Pulse Sequence: s2pul

Fc1ccc(SSc2ccc(F)cc2)cc1
2j

7.463
7.459
7.450
7.443
7.438
7.435
7.426
7.421
7.411
7.391
7.318
7.310
7.302
6.897
6.885
6.880
6.872

0.000

9 8 7 6 5 4 3 2 1 0 ppm

3.90 4.00

Sample: hh-v-38-c-crude
Sample ID: s_20181126_10
File: /home/jykang/hh-v-38-c-crude.fid
Pulse Sequence: szpul

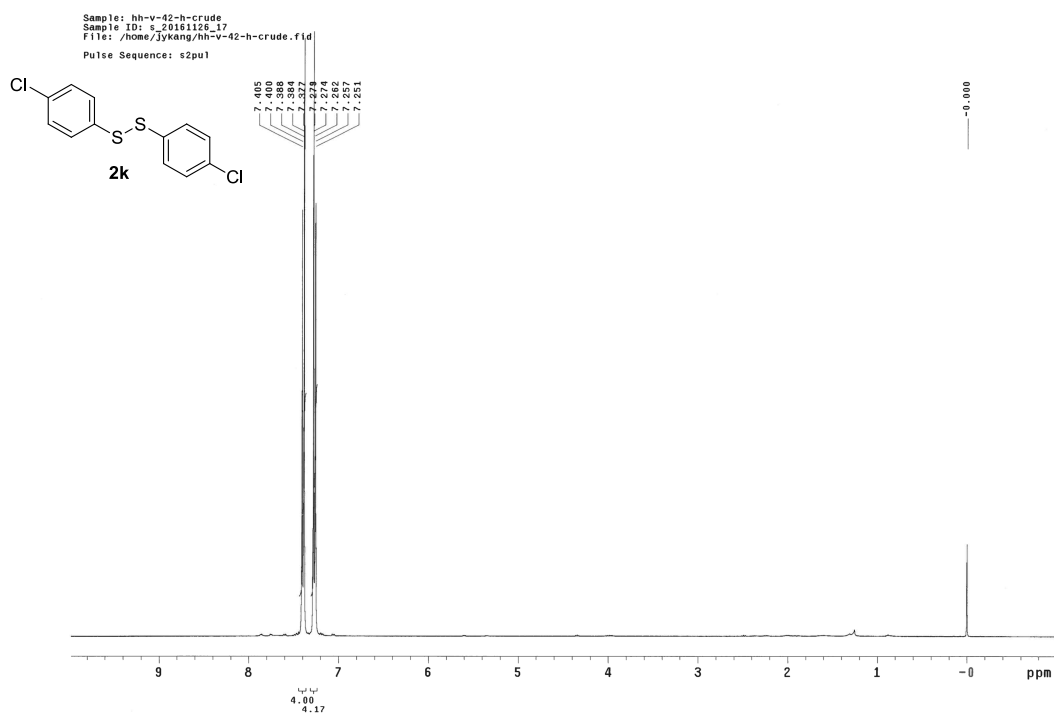
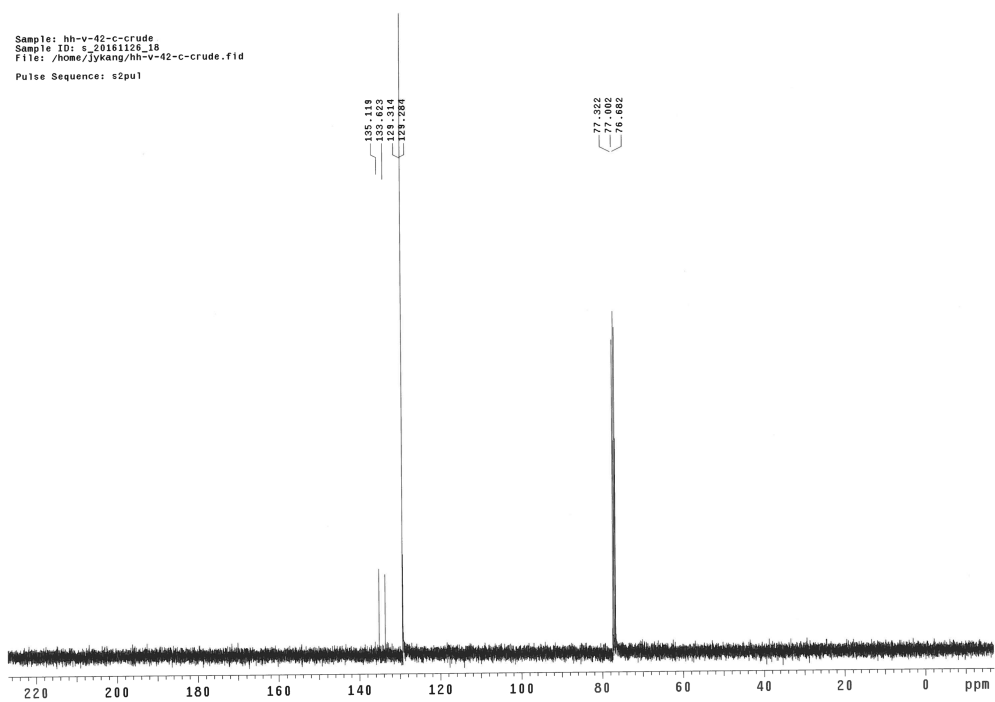
163.823
161.360
161.360

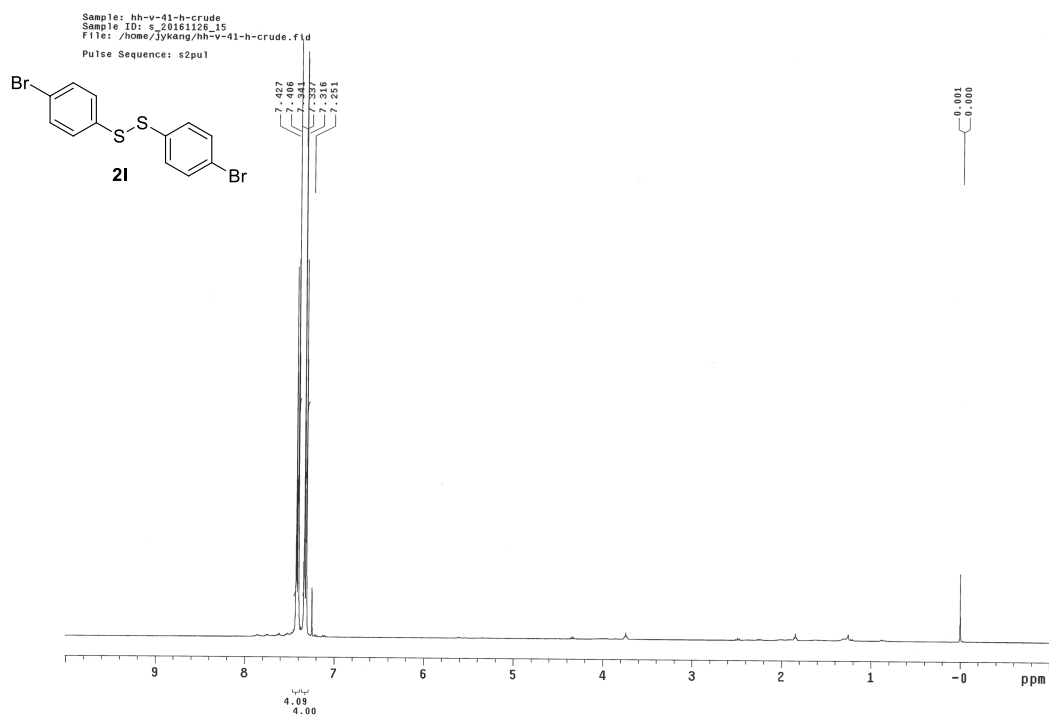
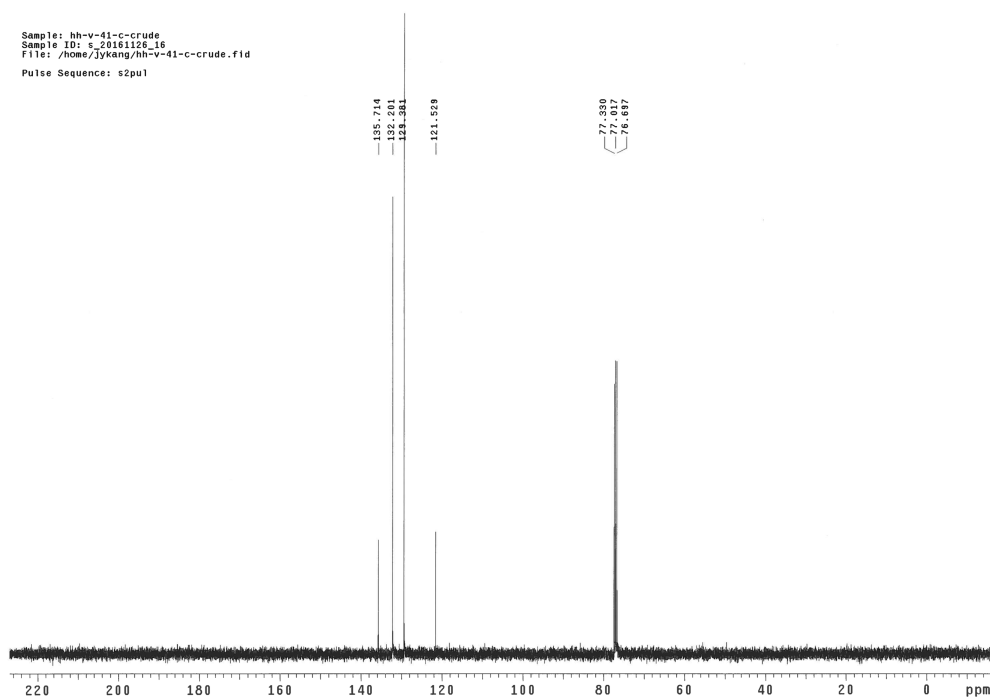
132.171
132.142
131.301
131.219

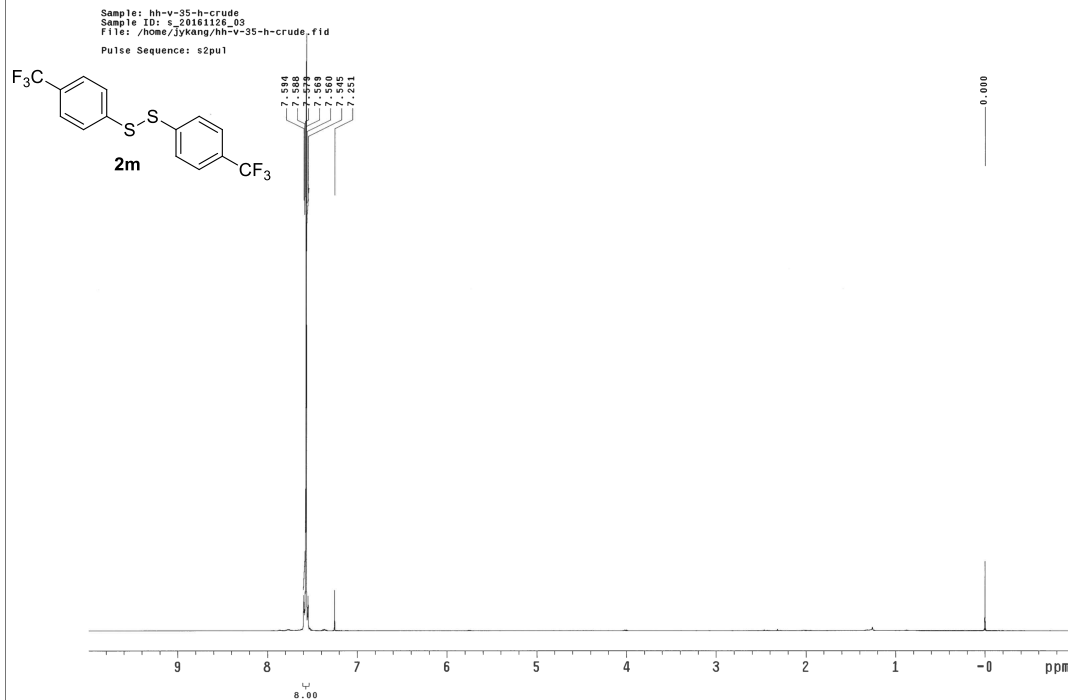
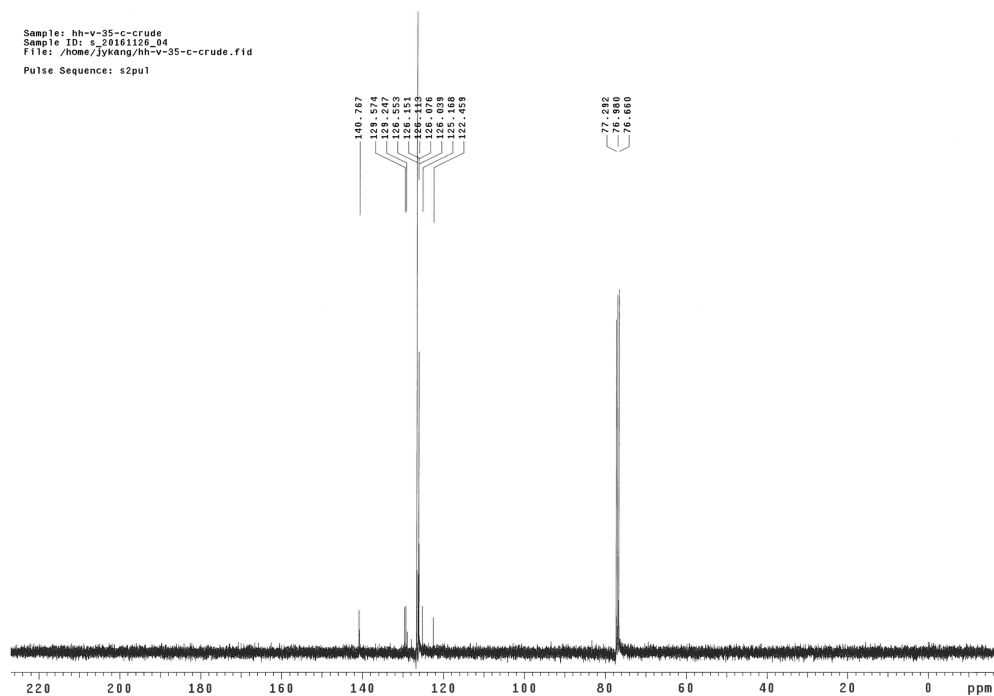
116.372
116.146

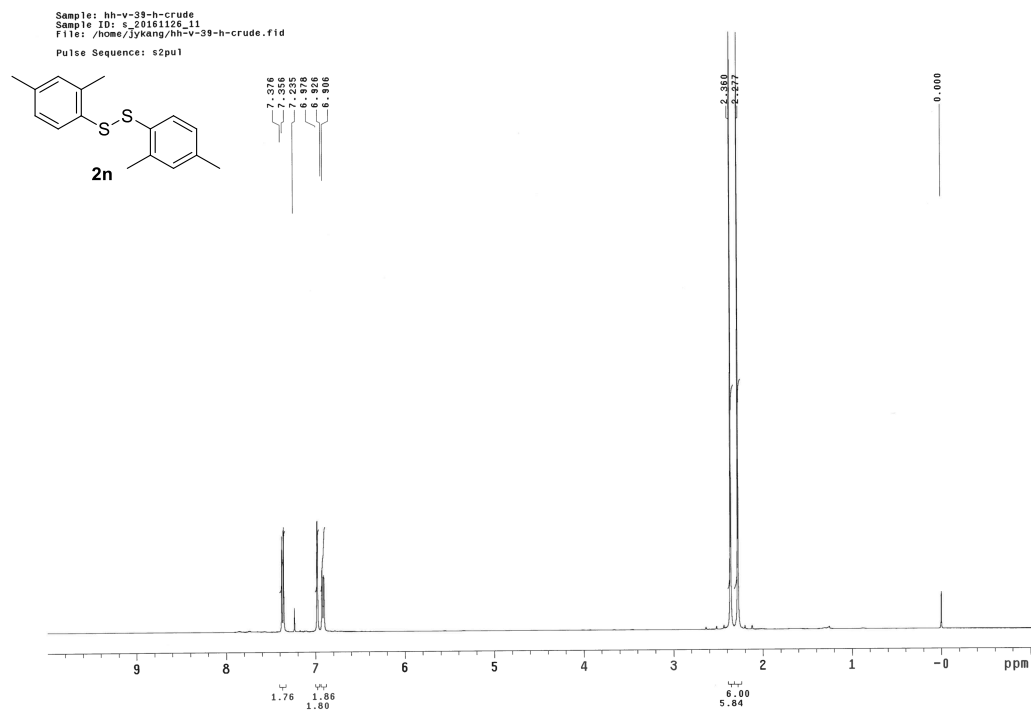
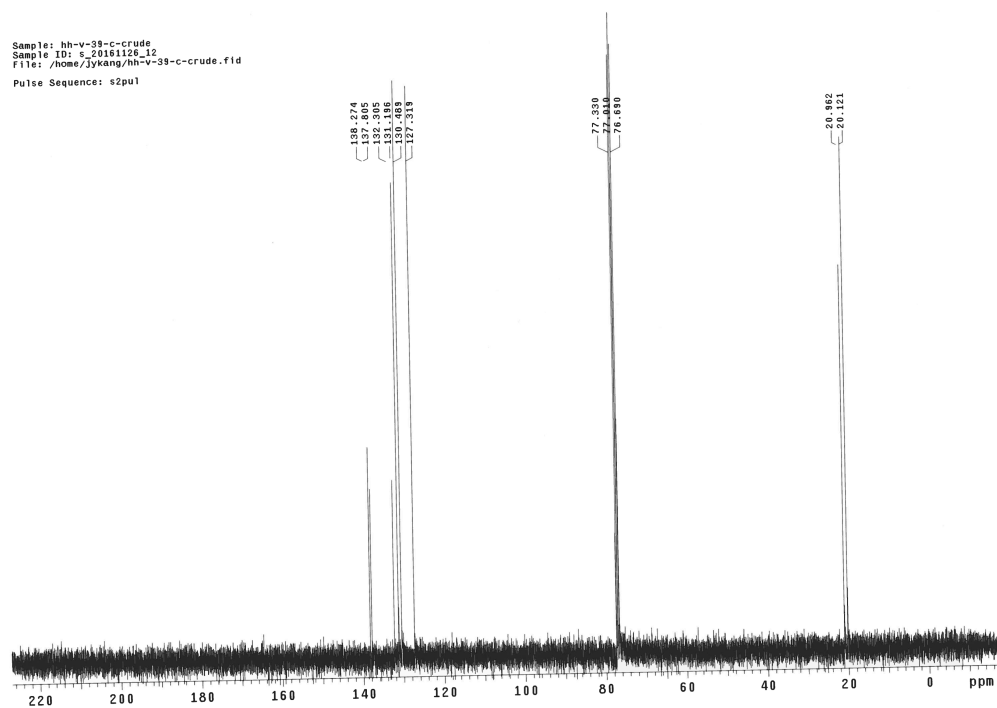
77.215
76.685
76.675

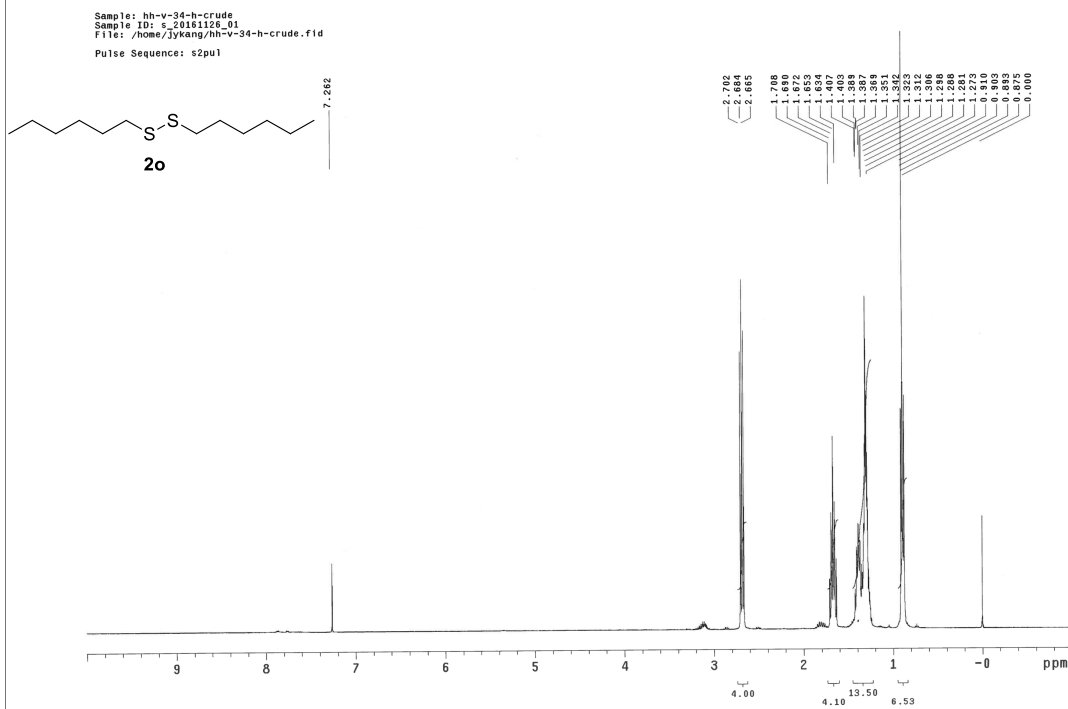
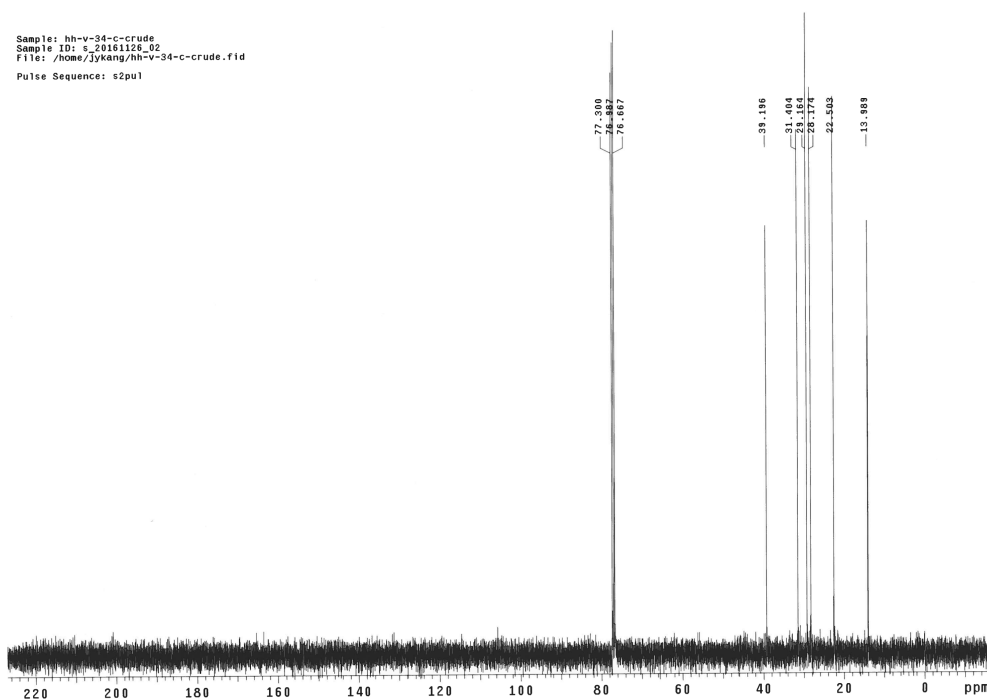
220 200 180 160 140 120 100 80 60 40 20 0 ppm

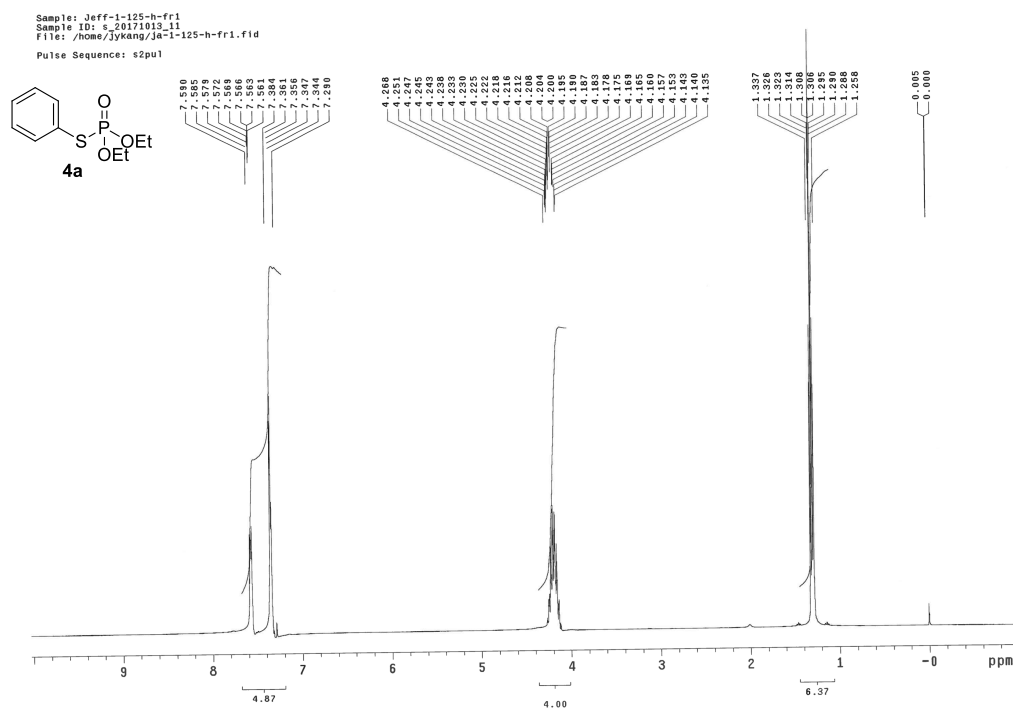
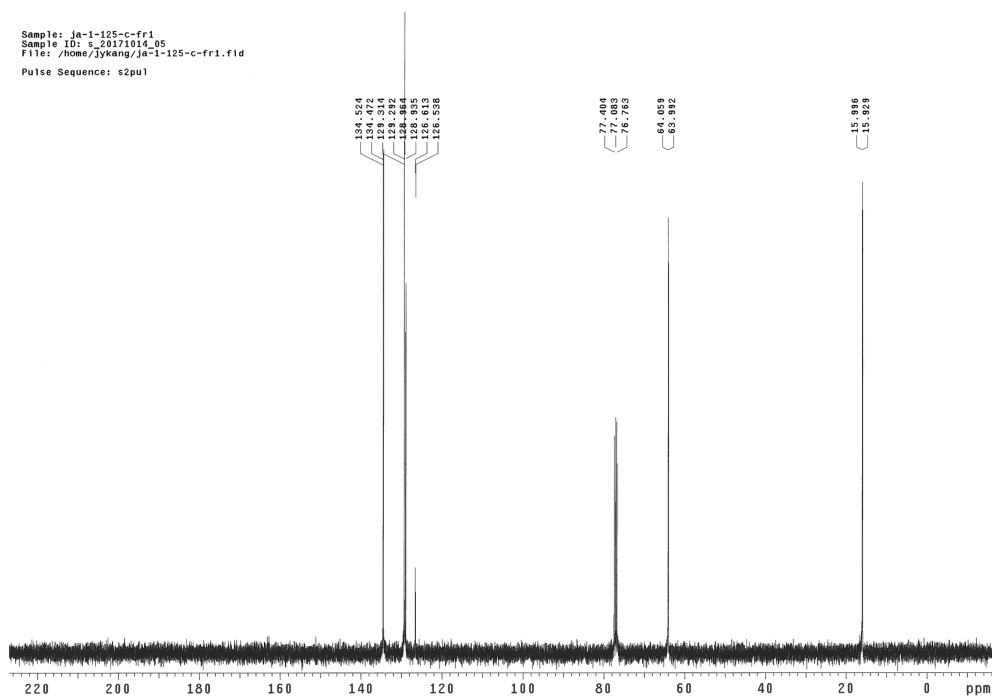
^1H NMR (400 MHz) in CDCl_3  **^{13}C NMR (100.5 MHz) in CDCl_3** 

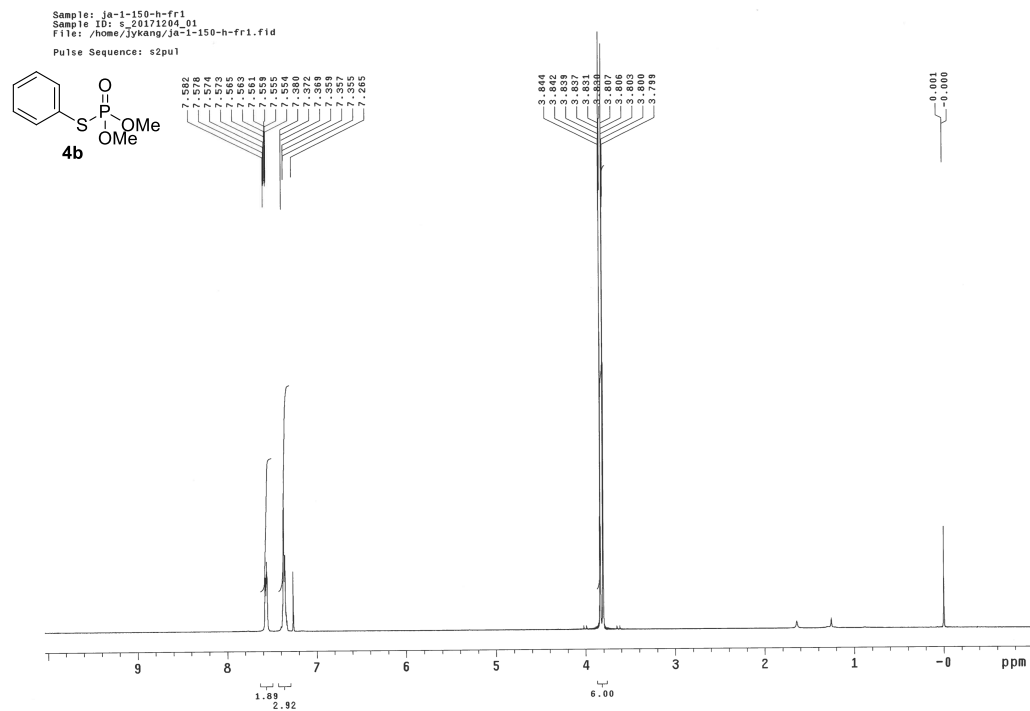
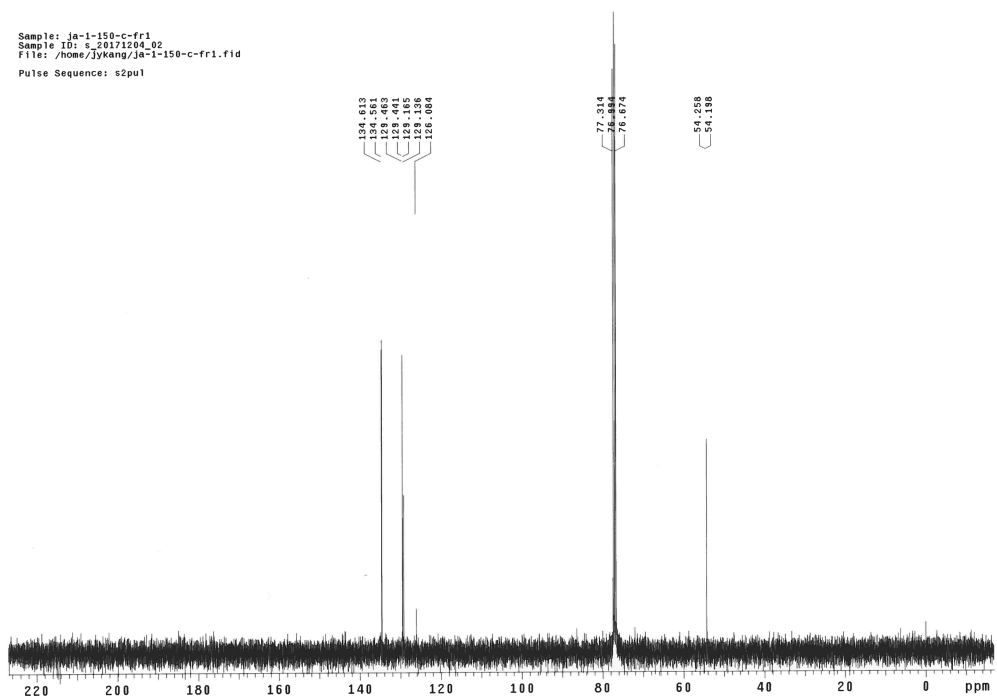
^1H NMR (400 MHz) in CDCl_3  **^{13}C NMR (100.5 MHz) in CDCl_3** 

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

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

Sample: ja-1-149-h-fr1
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File: /home/jykang/ja-1-149-h-fr1.fid
Pulse Sequence: s2pu1

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7.857
7.857
7.846
7.846
7.842
7.842
7.838
7.838
7.821
7.821
7.825
7.825
7.807
7.807
7.802
7.802
7.788
7.788
7.759
7.759
7.757
7.757
7.747
7.747
7.746
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7.729
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7.616
7.615
7.615
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7.613
7.613
7.612
7.612

0.145
0.009

4c

3.88 8.00 2.00 2.97

ppm

Sample: ja-1-149-c-fr1
Sample ID: s_20171204_04
File: /home/jy kang/ja-1-149-c-fr1.fid
Pulse Sequence: s2pu1

135.365
135.328
135.328
132.276
132.247
132.247
131.651
131.547
131.547
128.076
128.076
128.890
128.890
128.583
128.583
128.429
128.429
128.188
128.188

77.337
77.017
76.896

220 200 180 160 140 120 100 80 60 40 20 0 ppm

Sample: ja-i-108-h-fr1
Sample ID: s_20170915_01
File: /home/jykang/ja-i-108-h-fr1.fid
Pulse Sequence: s2pu1

4d

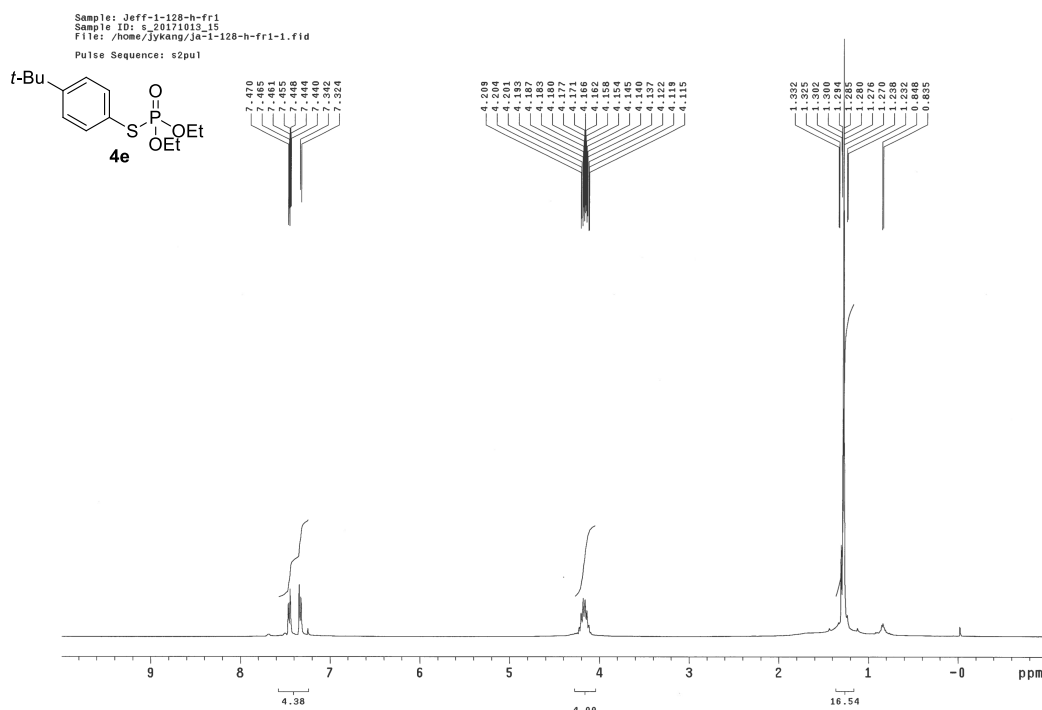
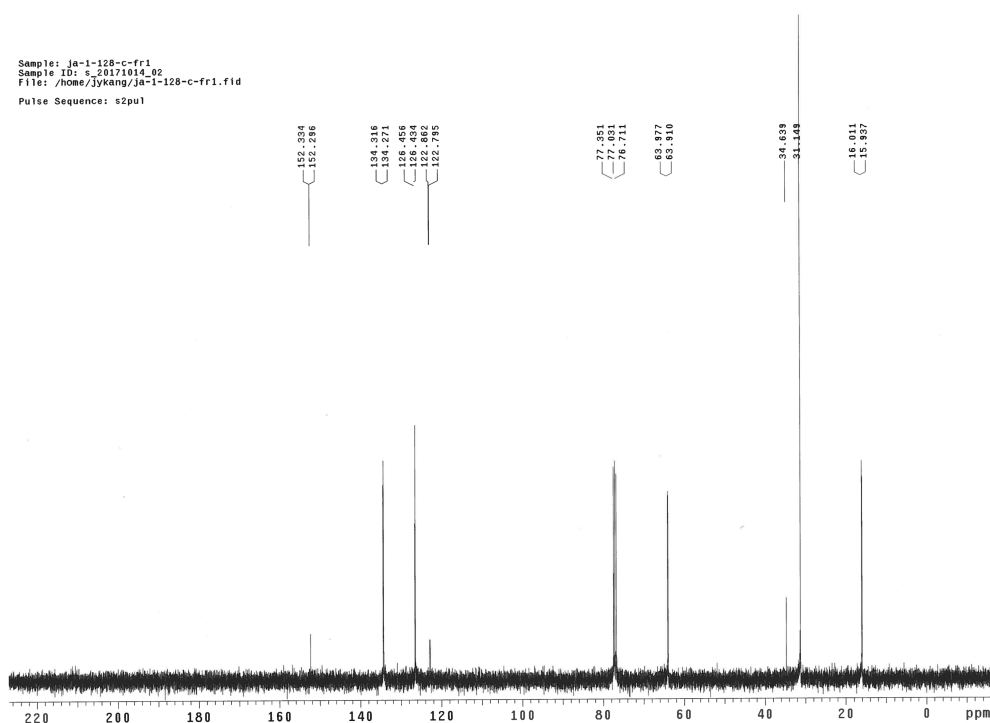
Chemical structure of **4d**: CCOP(=O)(c1ccc(C)cc1)S(=O)(=O)c2ccc(C)cc2

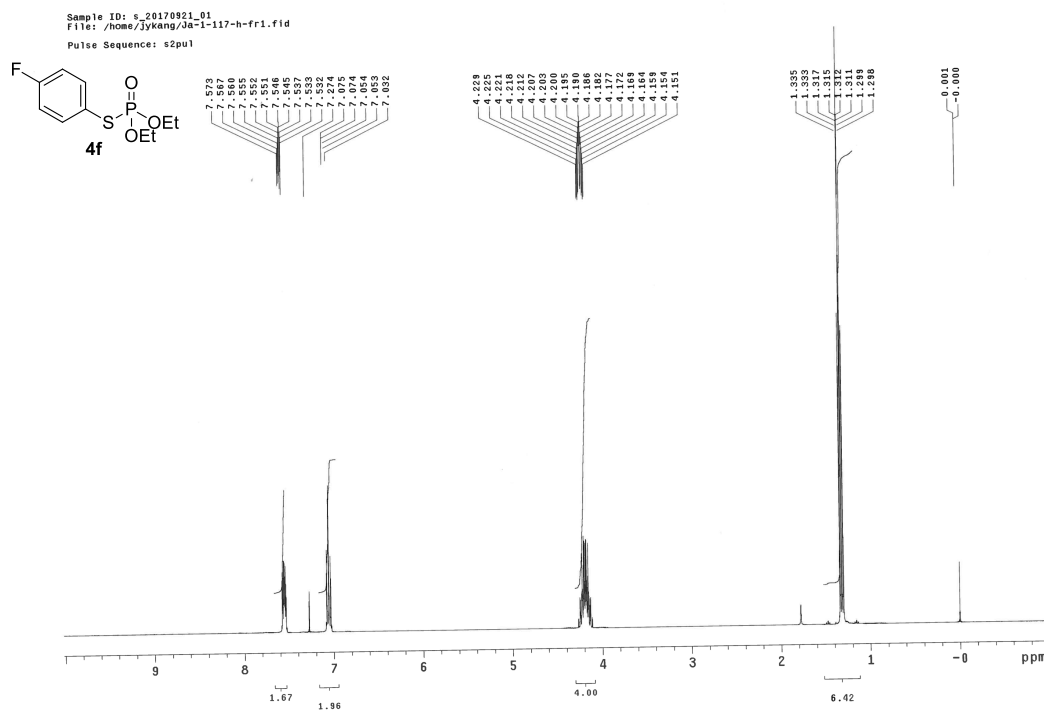
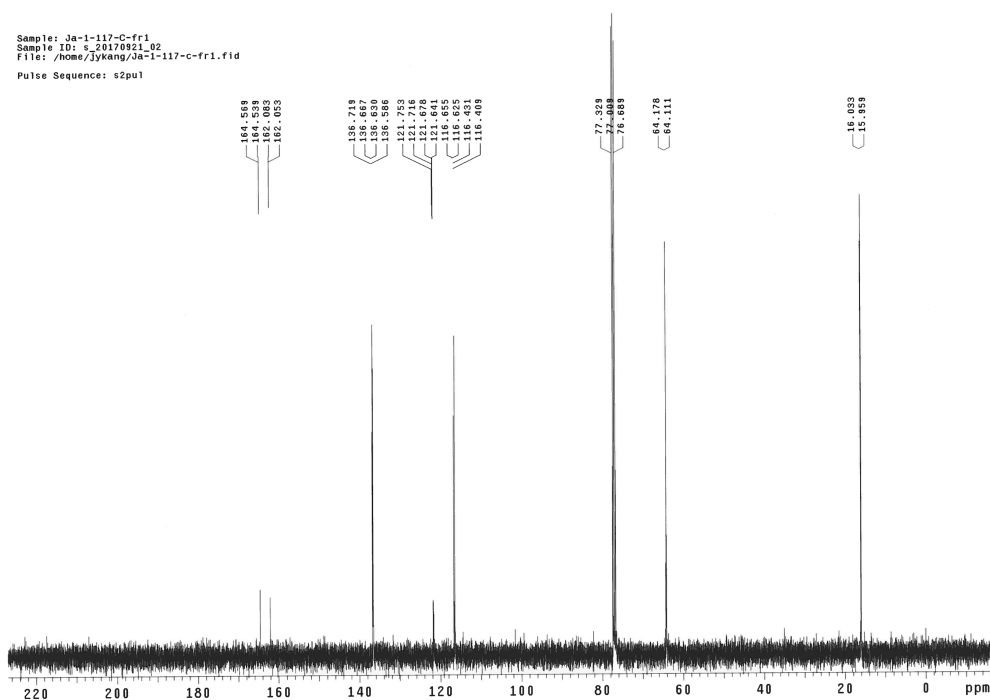
¹H NMR spectrum (CDCl₃) of compound **4d**. The spectrum shows peaks at 7.453, 7.449, 7.441, 7.437, 7.162, 7.147, 7.142 ppm (aromatic, integration 1.85, 2.01), 4.244, 4.221, 4.217, 4.207, 4.202, 4.189, 4.186, 4.182, 4.178, 4.173, 4.170, 4.160, 4.156, 4.144, 4.135 ppm (multiplet, integration 4.00), 2.346 ppm (singlet, integration 3.33), and 1.332, 1.326, 1.316, 1.310, 1.300, 1.288, 1.291 ppm (triplet, integration 6.37). A solvent peak for CDCl₃ is visible at 7.26 ppm.

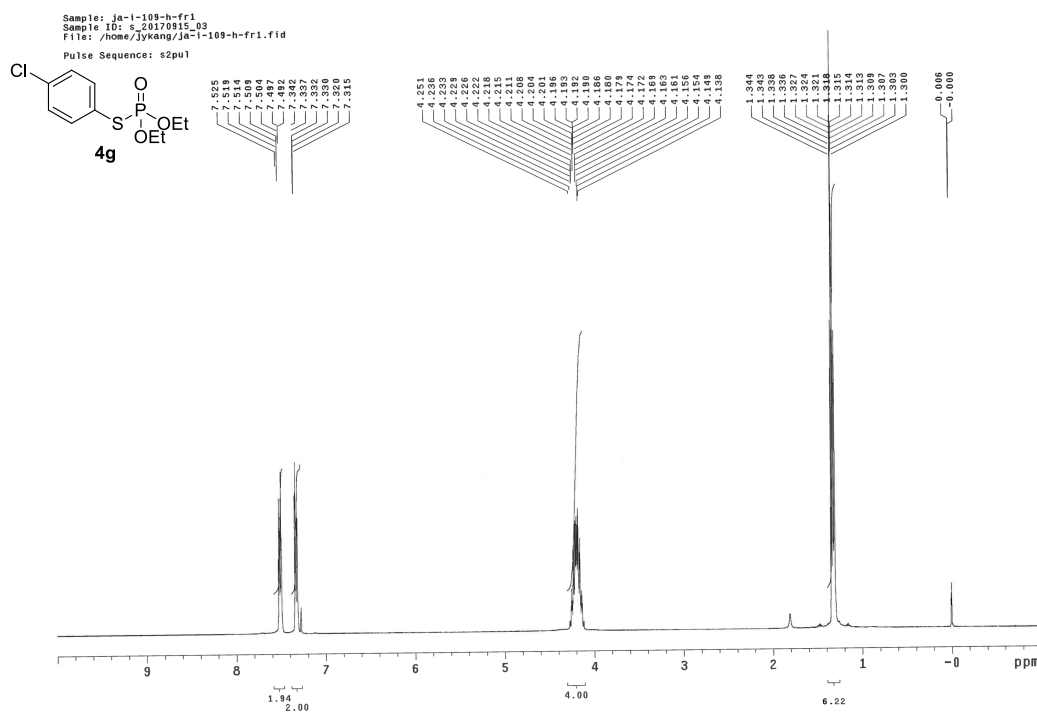
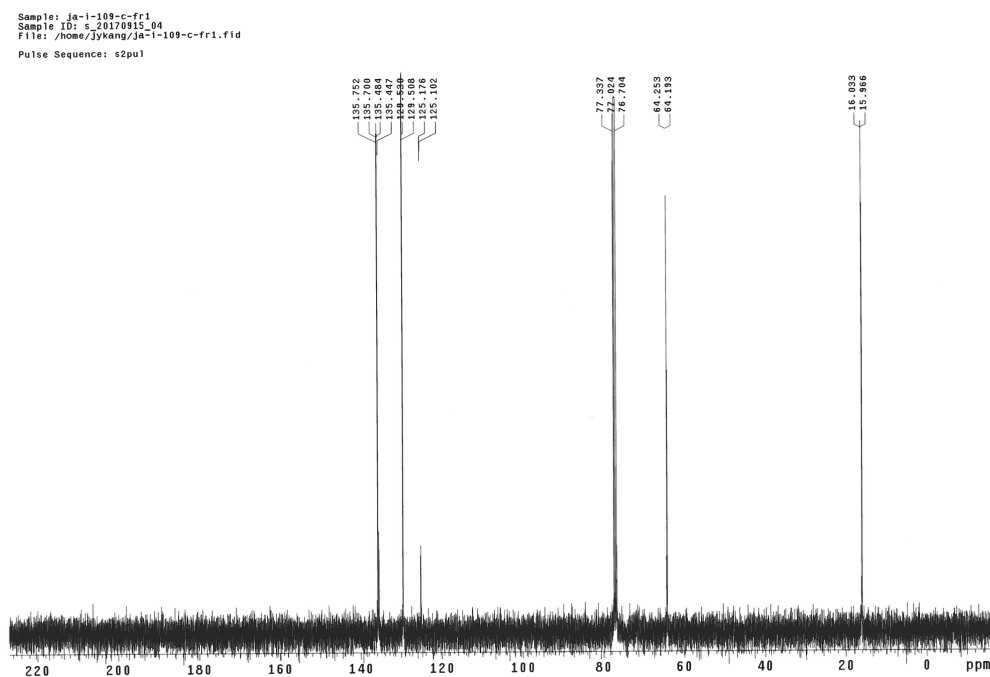
Sample: ja-1-108-c-fr1
Sample ID: 6_20170915_02
File: /home/jykang/ja-1-108-c-fr1.fid
Pulse Sequence: s2pul

Chemical shift (ppm):

- 138.265
- 137.582
- 136.586
- 136.111
- 135.111
- 122.839
- 122.765
- 77.344
- 77.024
- 76.711
- 63.963
- 63.983
- 21.154
- 16.026
- 15.952

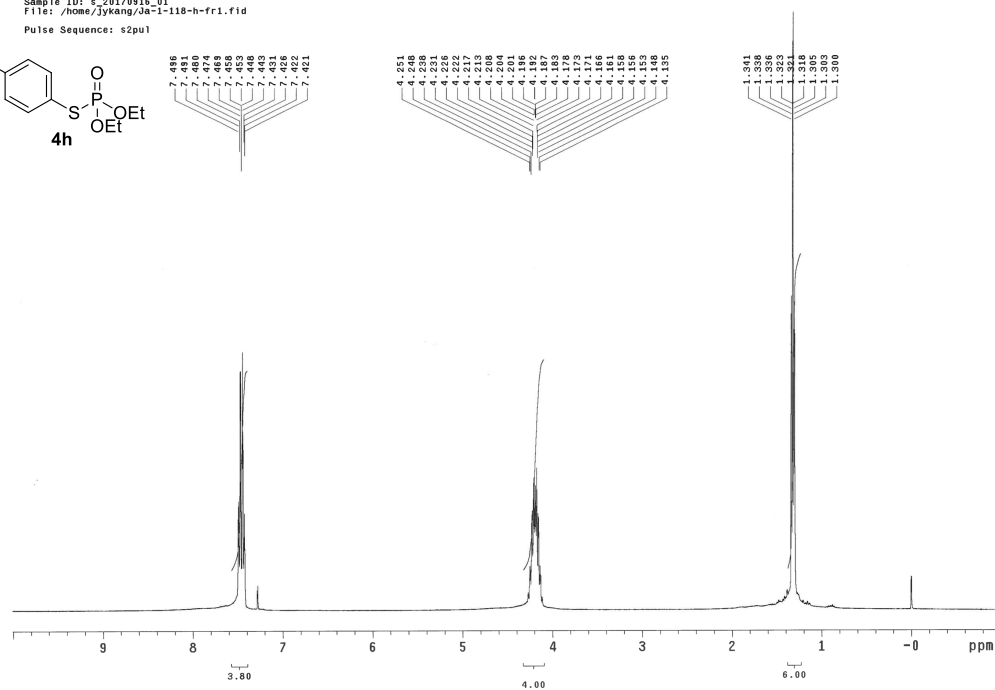
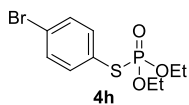
^1H NMR (400 MHz) in CDCl_3  ^{13}C NMR (100.5 MHz) in CDCl_3 

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

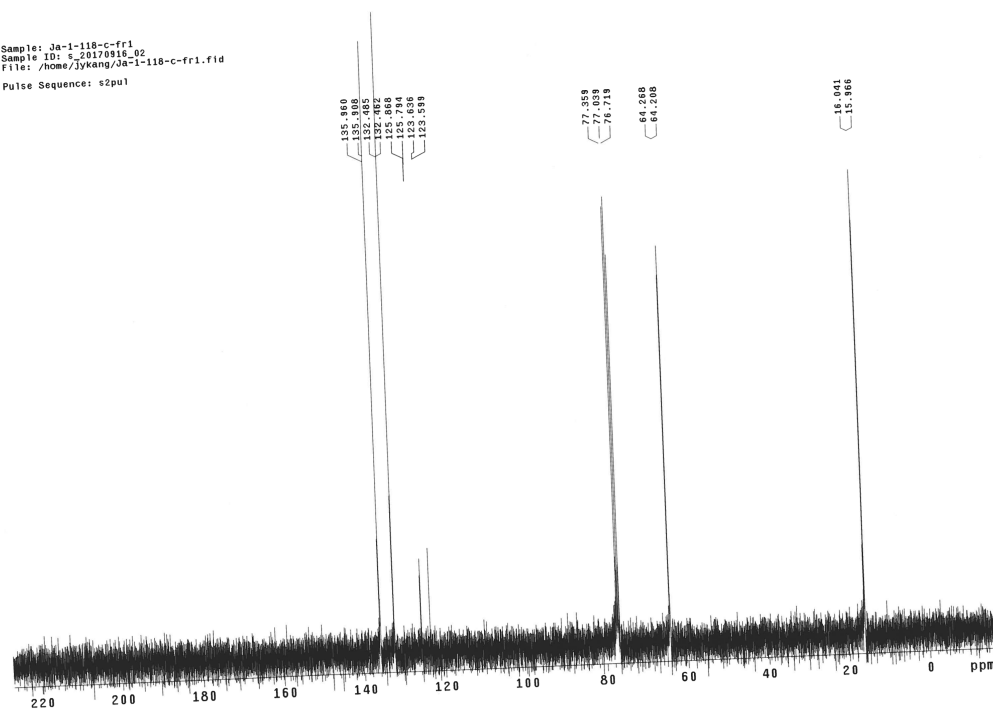
^1H NMR (400 MHz) in CDCl_3  ^{13}C NMR (100.5 MHz) in CDCl_3 

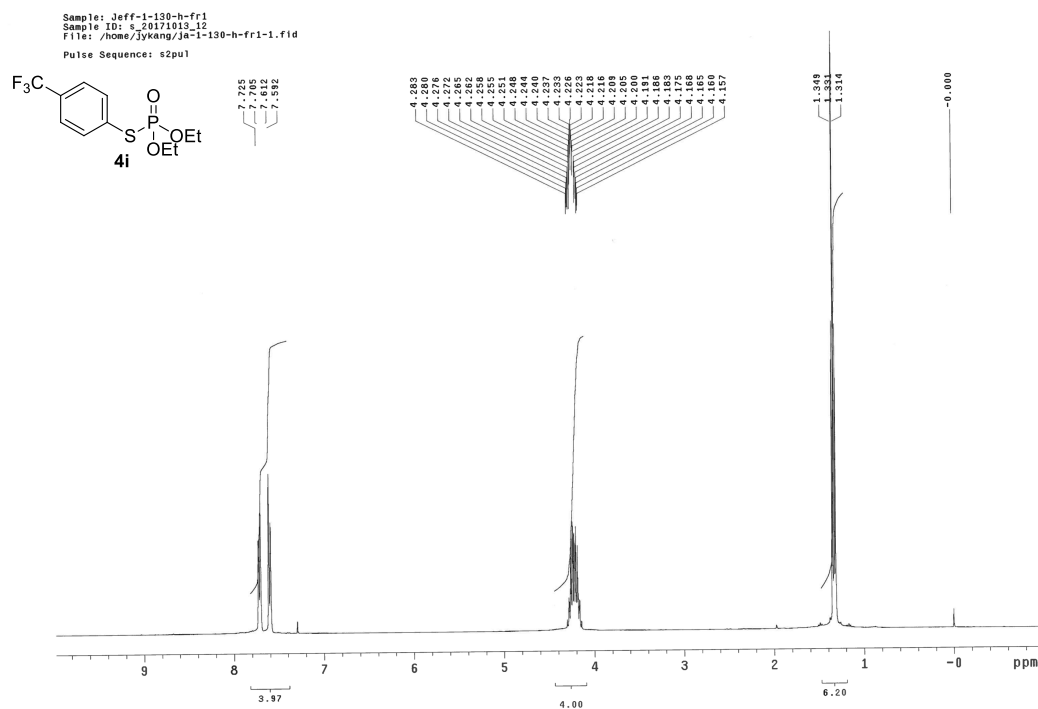
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Sample: Ja-1-118-h-fr1
Sample ID: s20170916_01
File: /home/jykang/Ja-1-118-h-fr1.fid

Pulse Sequence: s2pul
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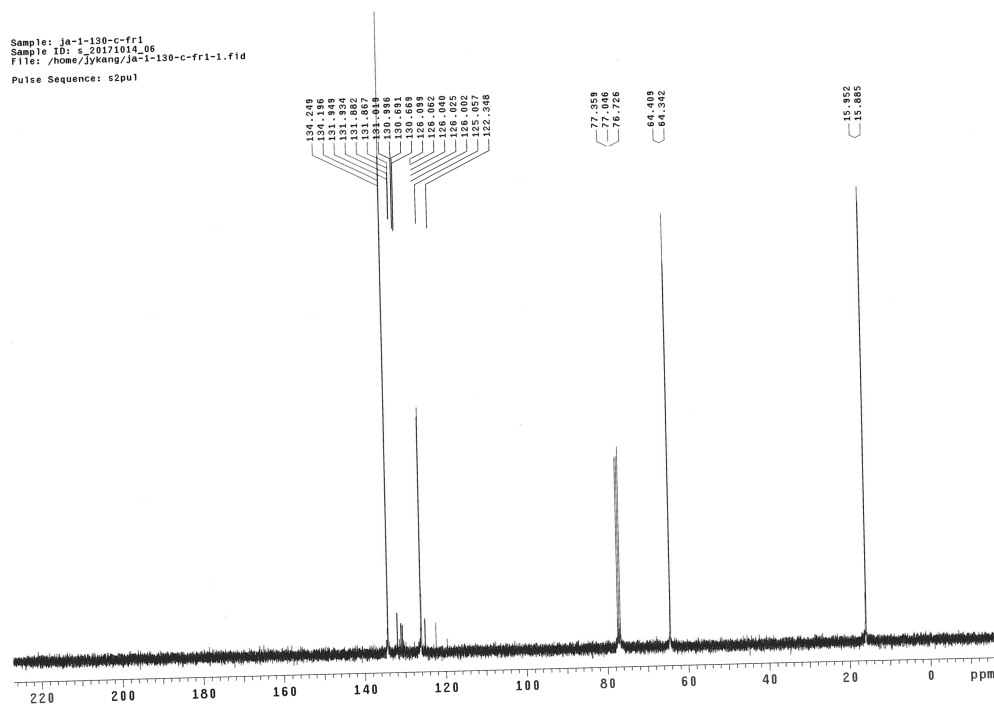


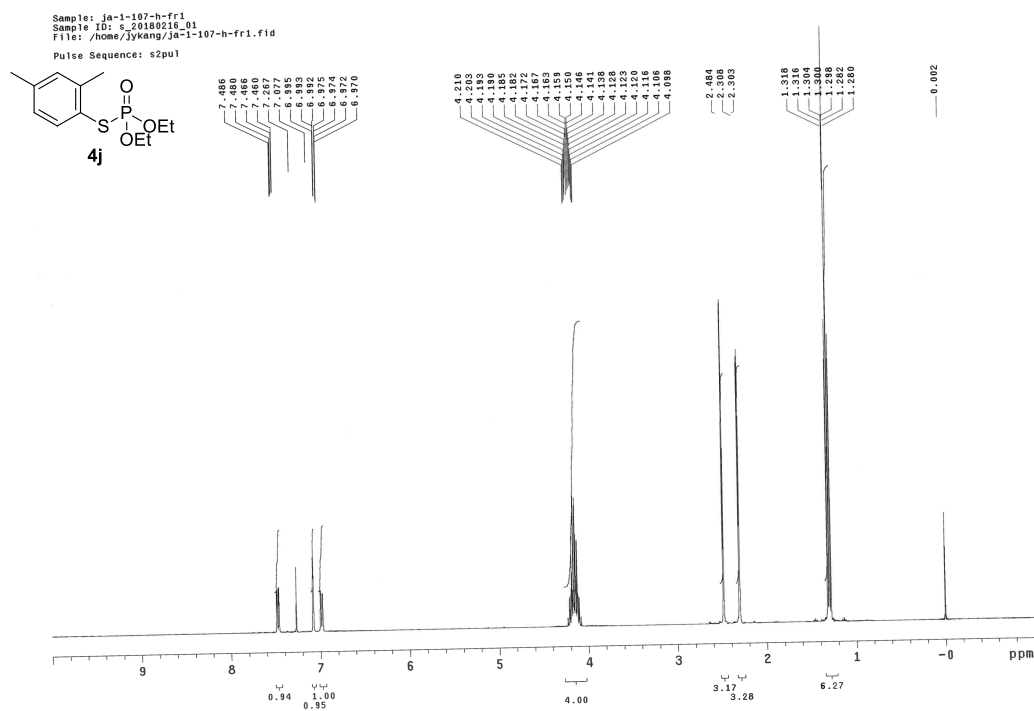
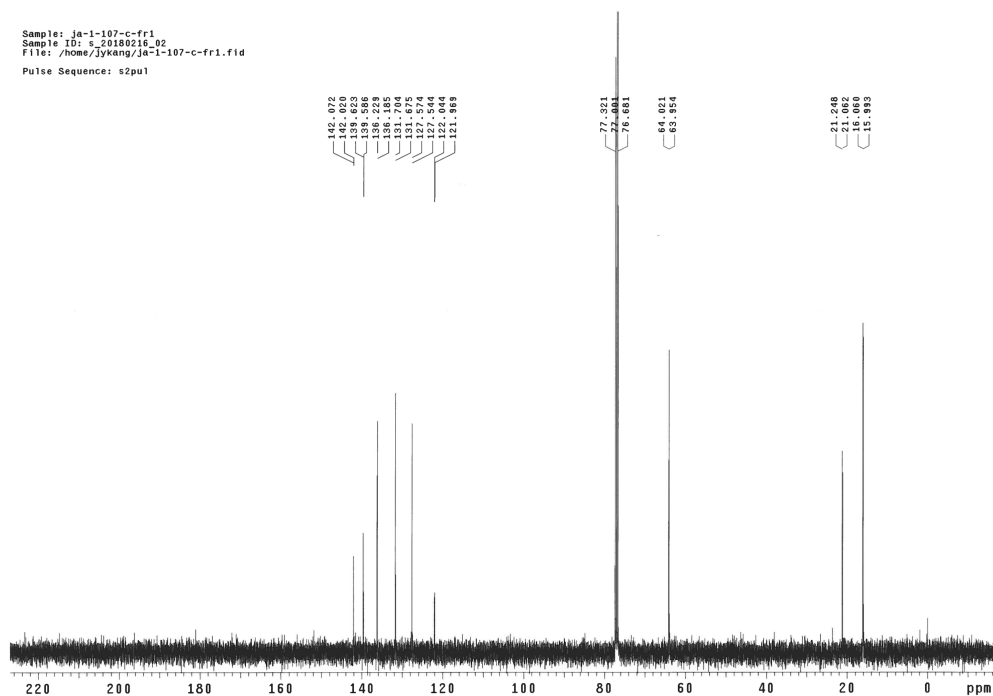
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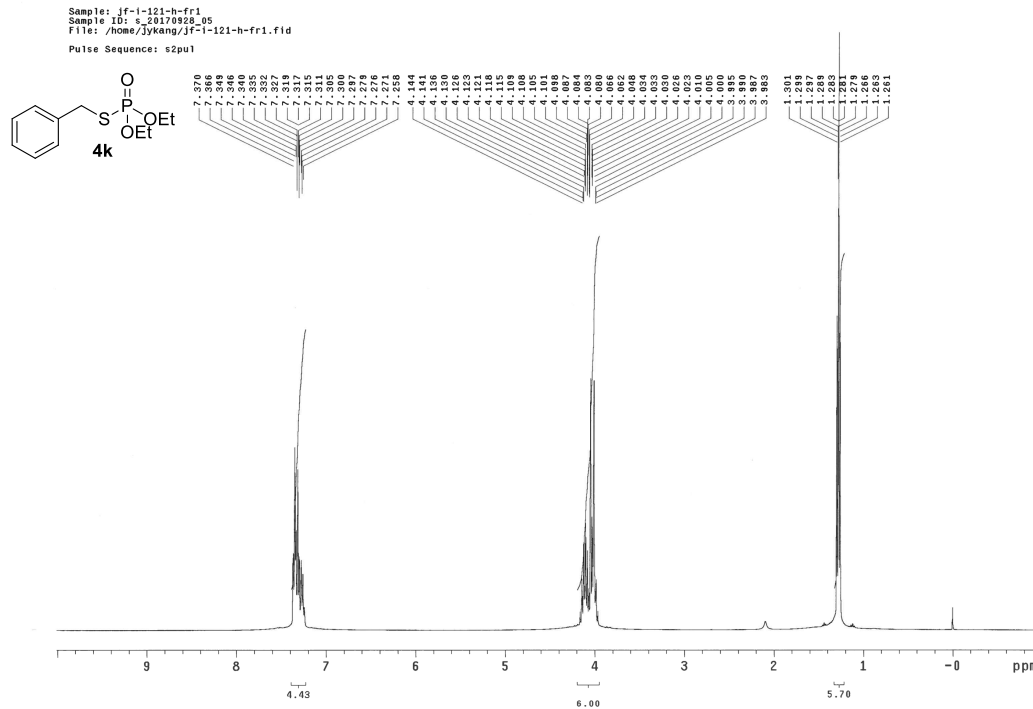
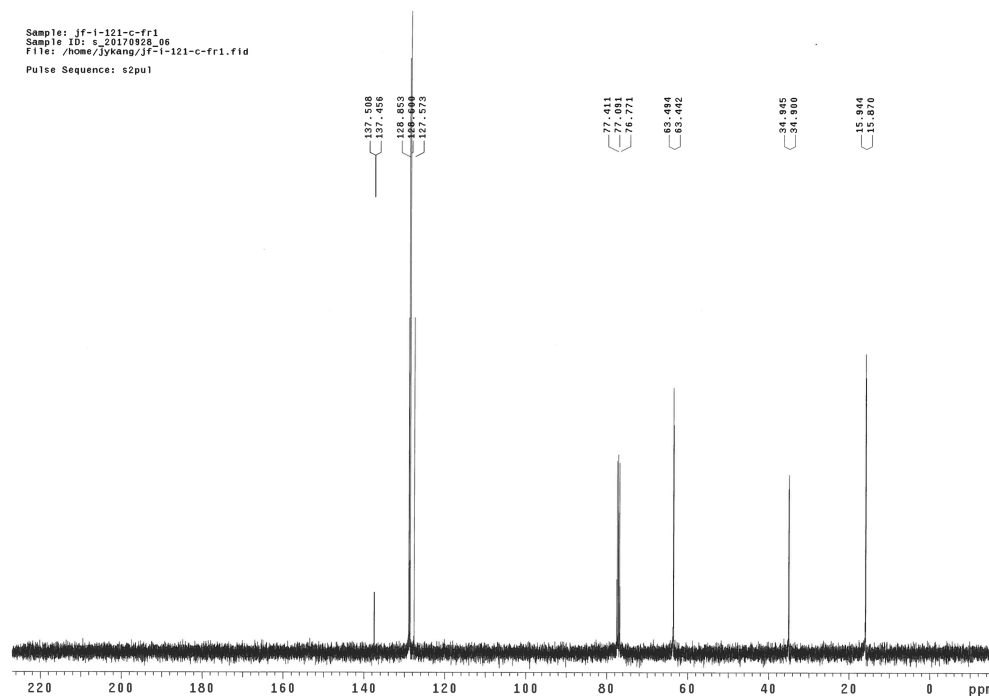


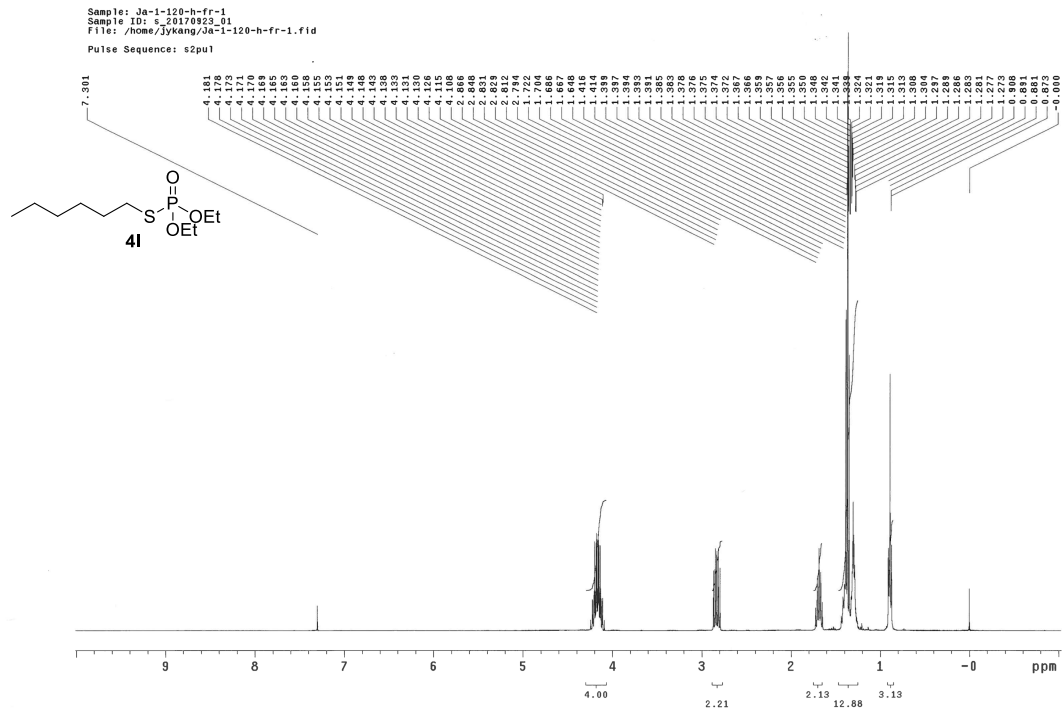
^1H NMR (400 MHz) in CDCl_3  ^{13}C NMR (100.5 MHz) in CDCl_3

Sample: ja-1-130-c-fr1
Sample ID: s_20171014_06
File: /home/jykang/ja-1-130-c-fr1-1.fid
Pulse Sequence: s2pul



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

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

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