

Supporting Information – Electronic and NMR Spectra

Synthesis of 1,3-Diphenyl-6-alkyl/aryl Substituted Fulvene Chromophores: Observation of π - π interactions in a 6-Pyrene Substituted 1,3-Diphenylfulvene

Andrew J. Peloquin,[†] Rebecca L. Stone,[†] Sarah E. Avila,[†] Erlyn R. Rudico,[†] Christopher B. Horn,[†] Kim A. Gardner,[†] David W. Ball,[‡] Jane E.B. Johnson,[†] Scott T. Iacono,[†] and Gary J. Balaich^{*†}

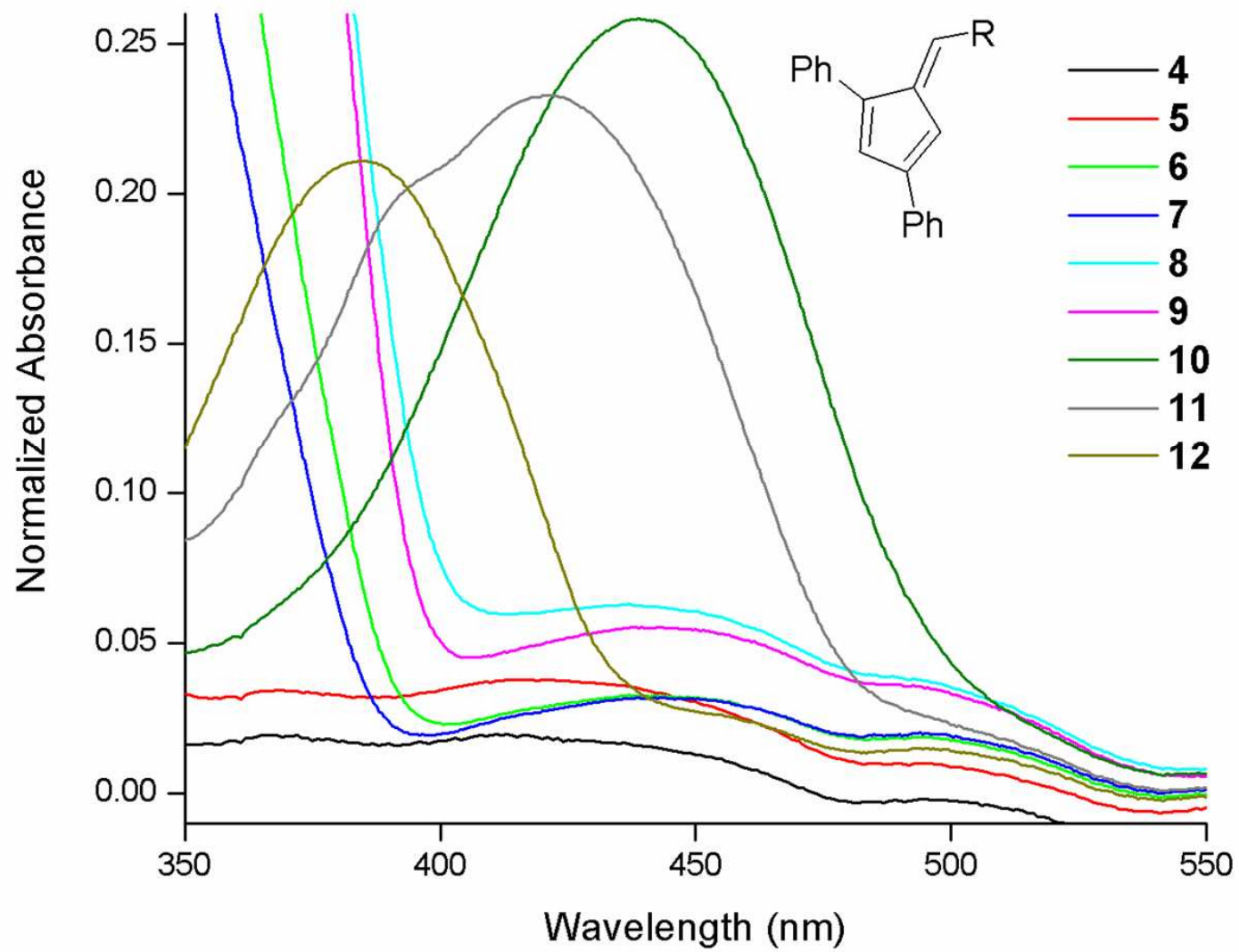
[†]Department of Chemistry, United States Air Force Academy, USAF Academy, CO 80840 (USA)

[‡]Department of Chemistry, Cleveland State University, Cleveland, OH 44115 (USA)

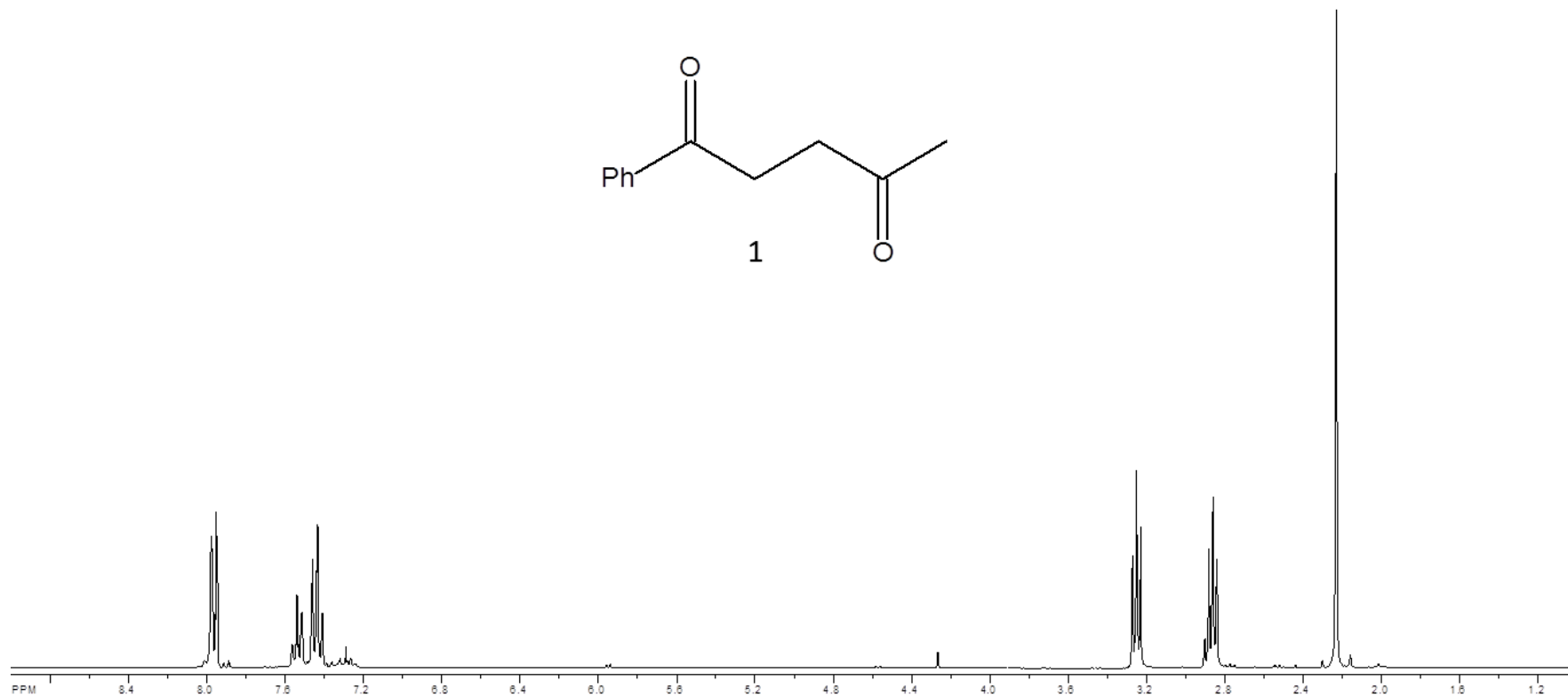
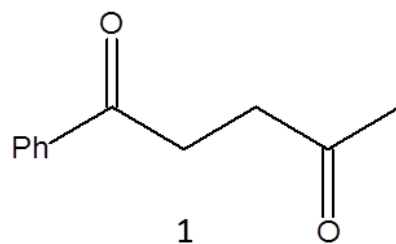
Corresponding author: ^{} gary.balaich@usafa.edu*

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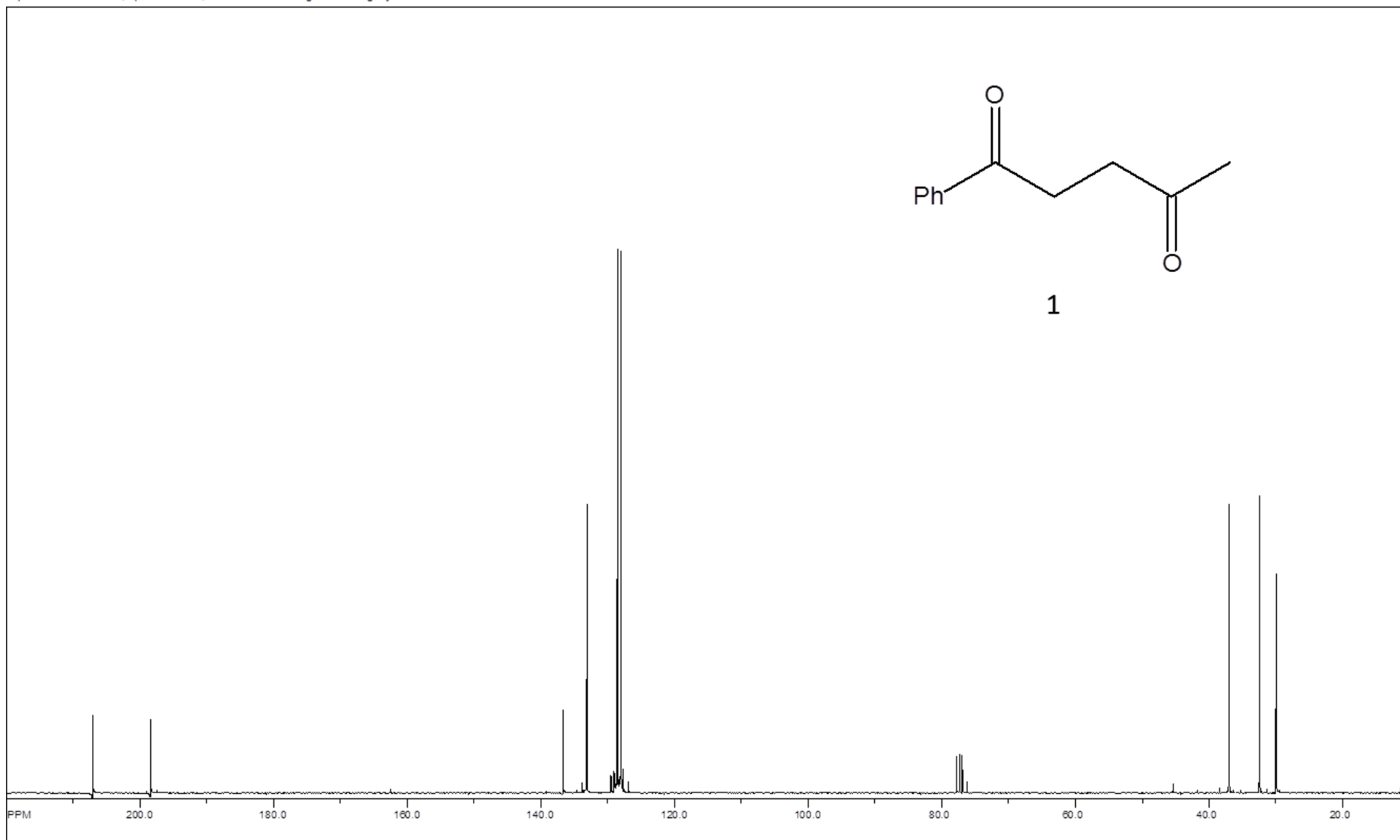
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300 MHz ^1H NMR spectrum of 1-phenyl-1,4-pentanedione **1** in CDCl_3



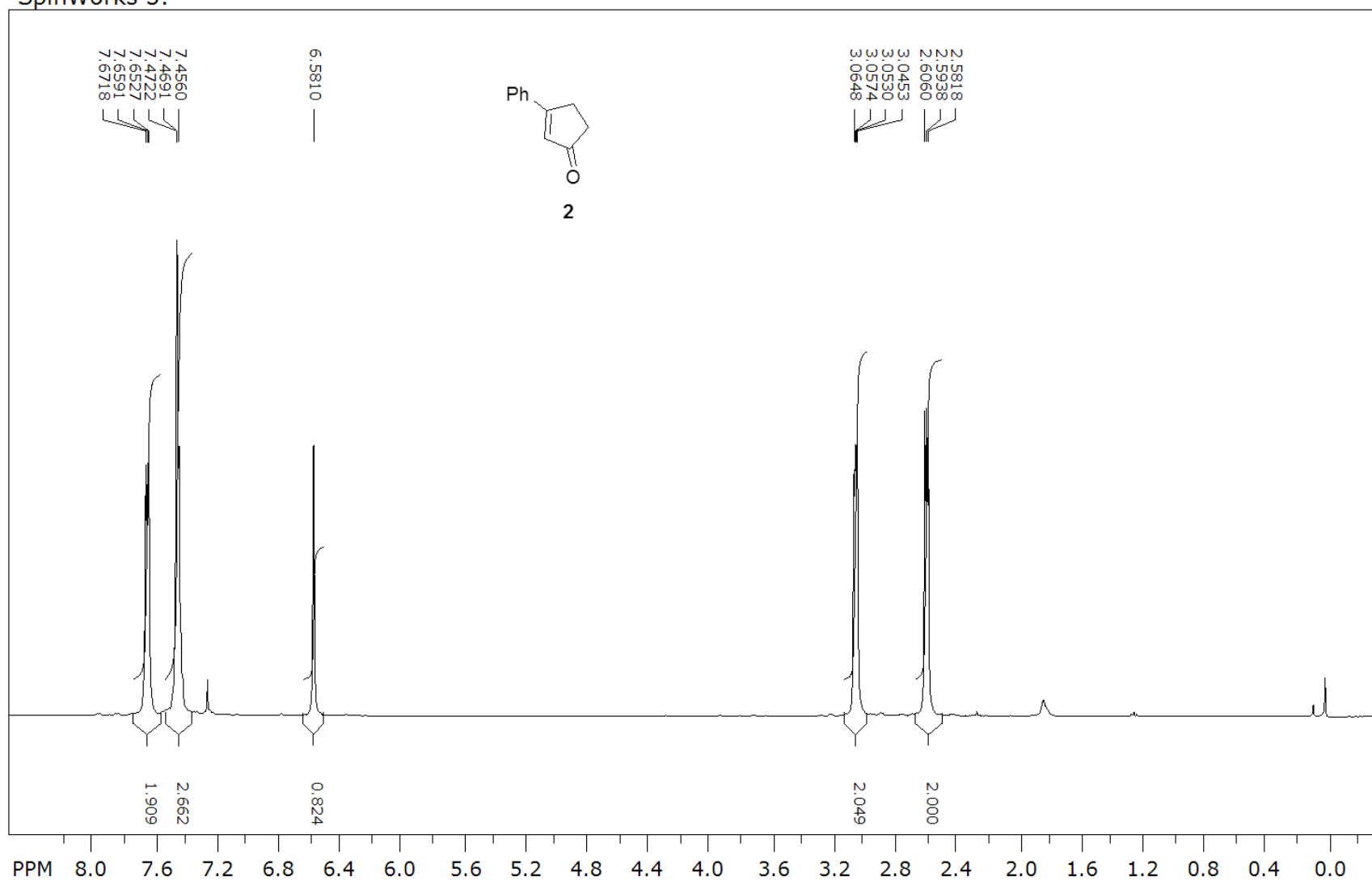
SpinWorks 2.5: 1Ph 1,4-pentanedione, vac. distilled 150 deg at 2 torr - light yellow oil - in



file: C:\Users\Gery\Bench\Documents\RESEARCH\PUBLICATIONS\FULLY\ENE_PAPER\J\INTEXT\NMR\data\1,4-Pentanedione_13C\1,4-Pentanedione_13C_1.fid (1) 10/10/10 10/10/10 10/10/10
transmitter freq.: 75.481967 MHz
time domain size: 47120 points
width: 18115.94 Hz = 240.074734 ppm = 0.384464 Hz/g
number of scans: 352

freq. of 0 ppm: 75.481967 MHz
processed size: 65536 complex points
LB: 1.000 GB: 0.0000

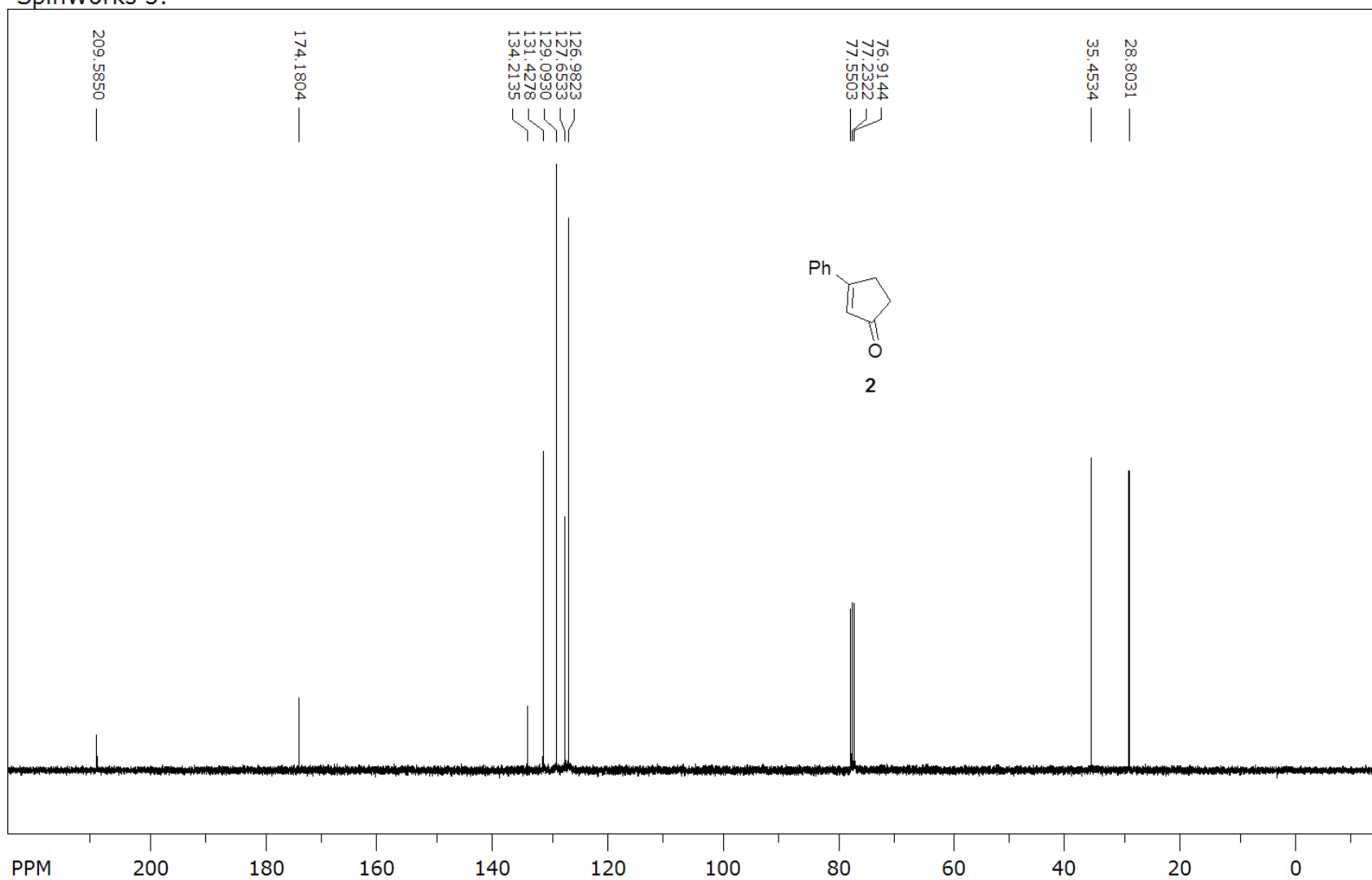
SpinWorks 3:



file: ..._20110916_1.fid\Compound 2 HNMRfid block# 1 expt: "s2pul"
 transmitter freq.: 400.108727 MHz
 time domain size: 25582 points
 width: 6402.05 Hz = 16.0008 ppm = 0.250256 Hz/pt
 number of scans: 16

freq. of 0 ppm: 400.106321 MHz
 processed size: 32768 complex points
 LB: 1.500 GF: 0.0000
 Hz/cm: 142.752 ppm/cm: 0.35678

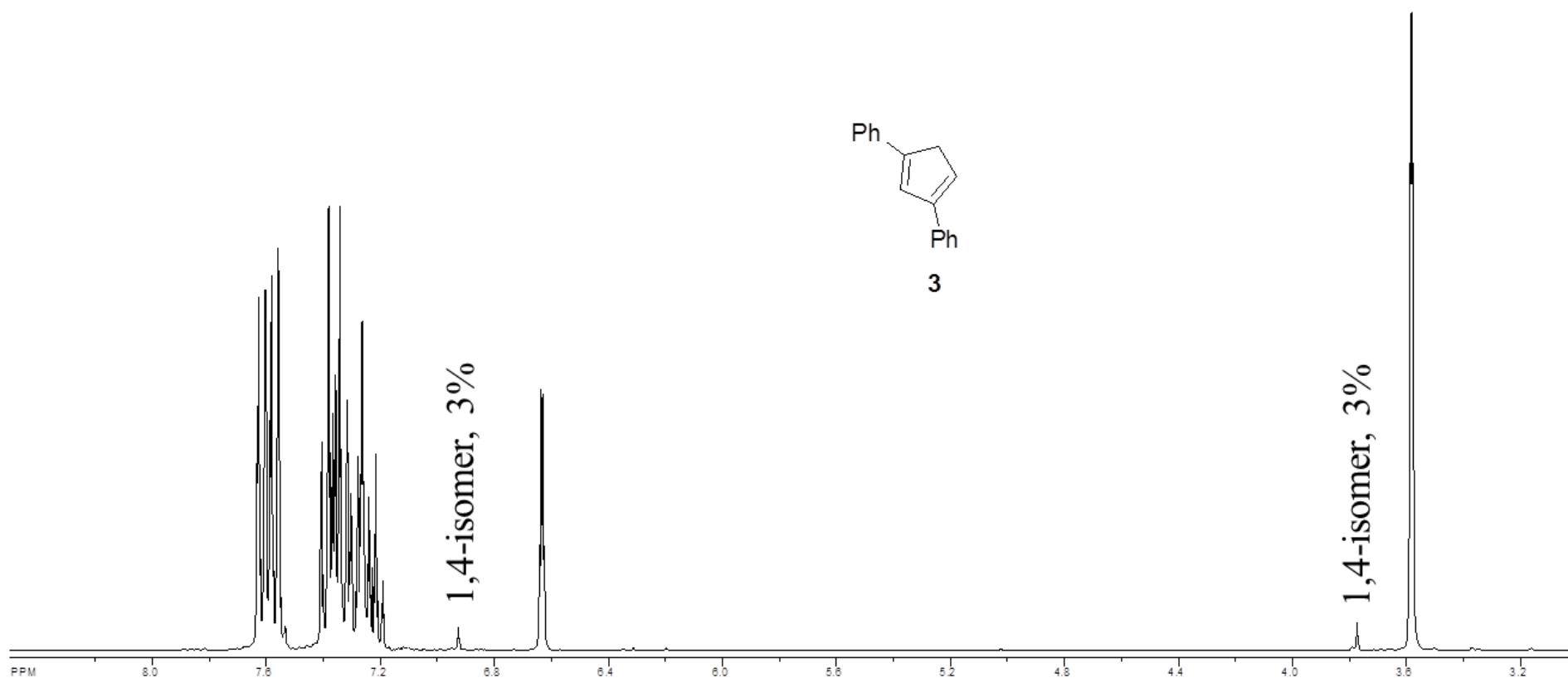
SpinWorks 3:



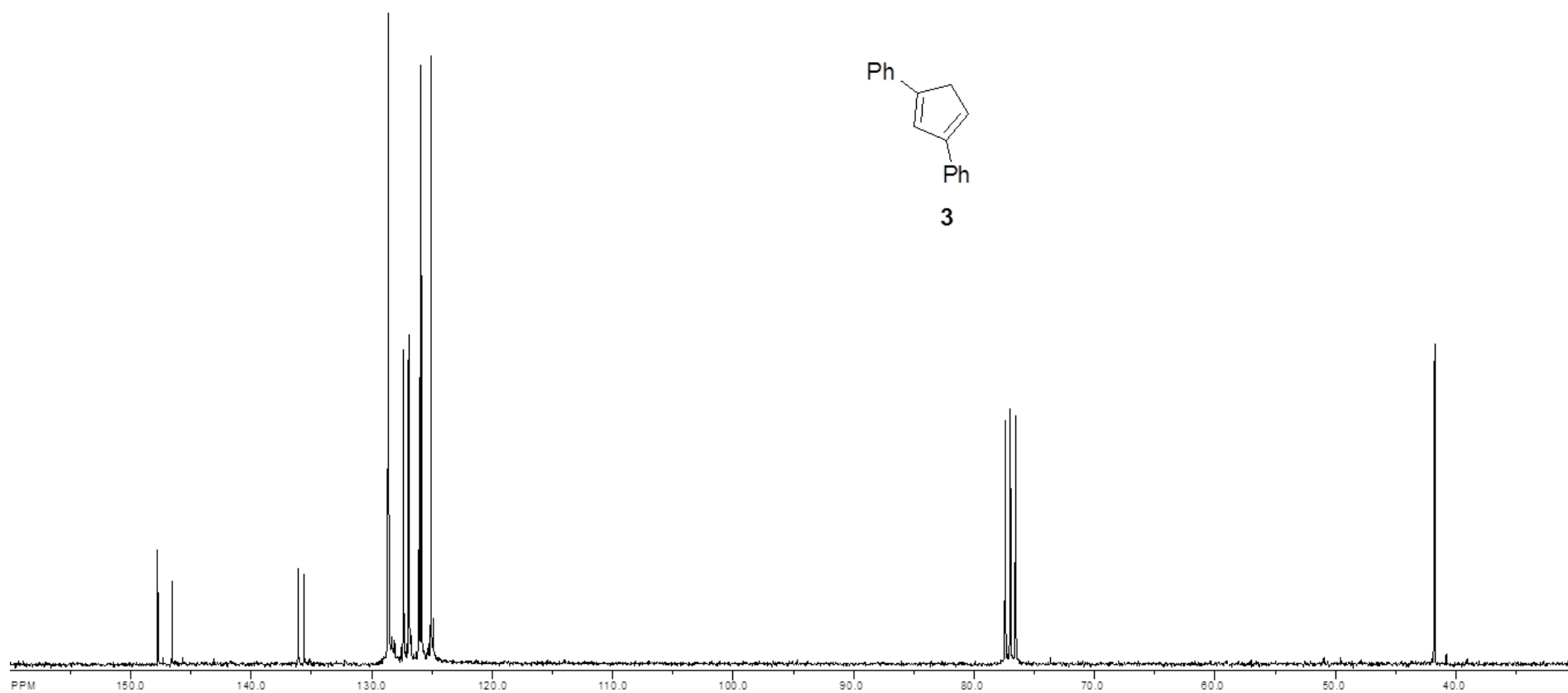
file: ...10916_1.fid\Compound 2 13C NMR fid block# 1 expt: "s2pul"
 transmitter freq.: 100.617379 MHz
 time domain size: 62826 points
 width: 24154.59 Hz = 240.0638 ppm = 0.384468 Hz/pt
 number of scans: 256

freq. of 0 ppm: 100.606797 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 966.184 ppm/cm: 9.60255

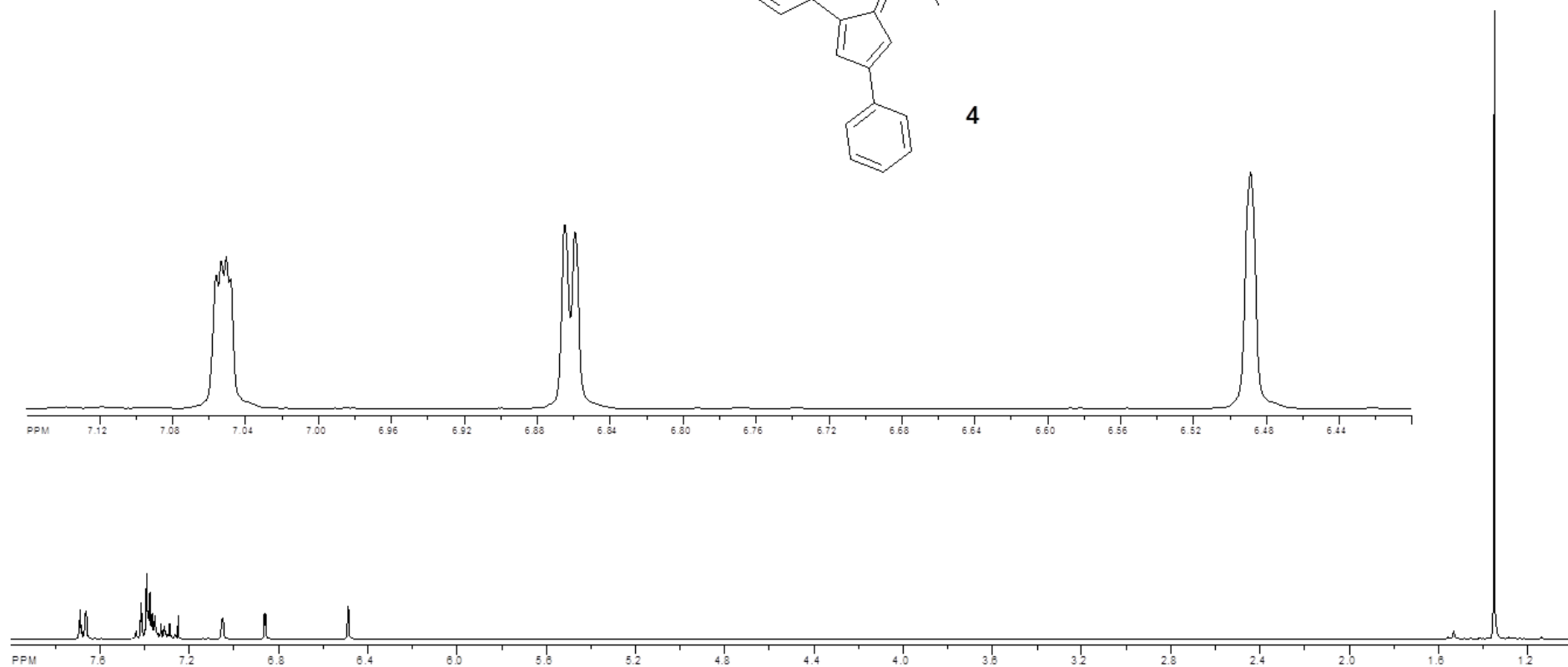
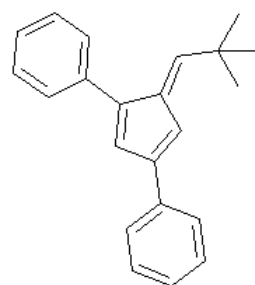
300 MHz ^1H NMR spectrum of 1,3-diphenylcyclopentadiene (recrystallized from EtOH) in CDCl_3 (presence of 1,4-isomer due to acid catalyzed isomerization during reaction workup)

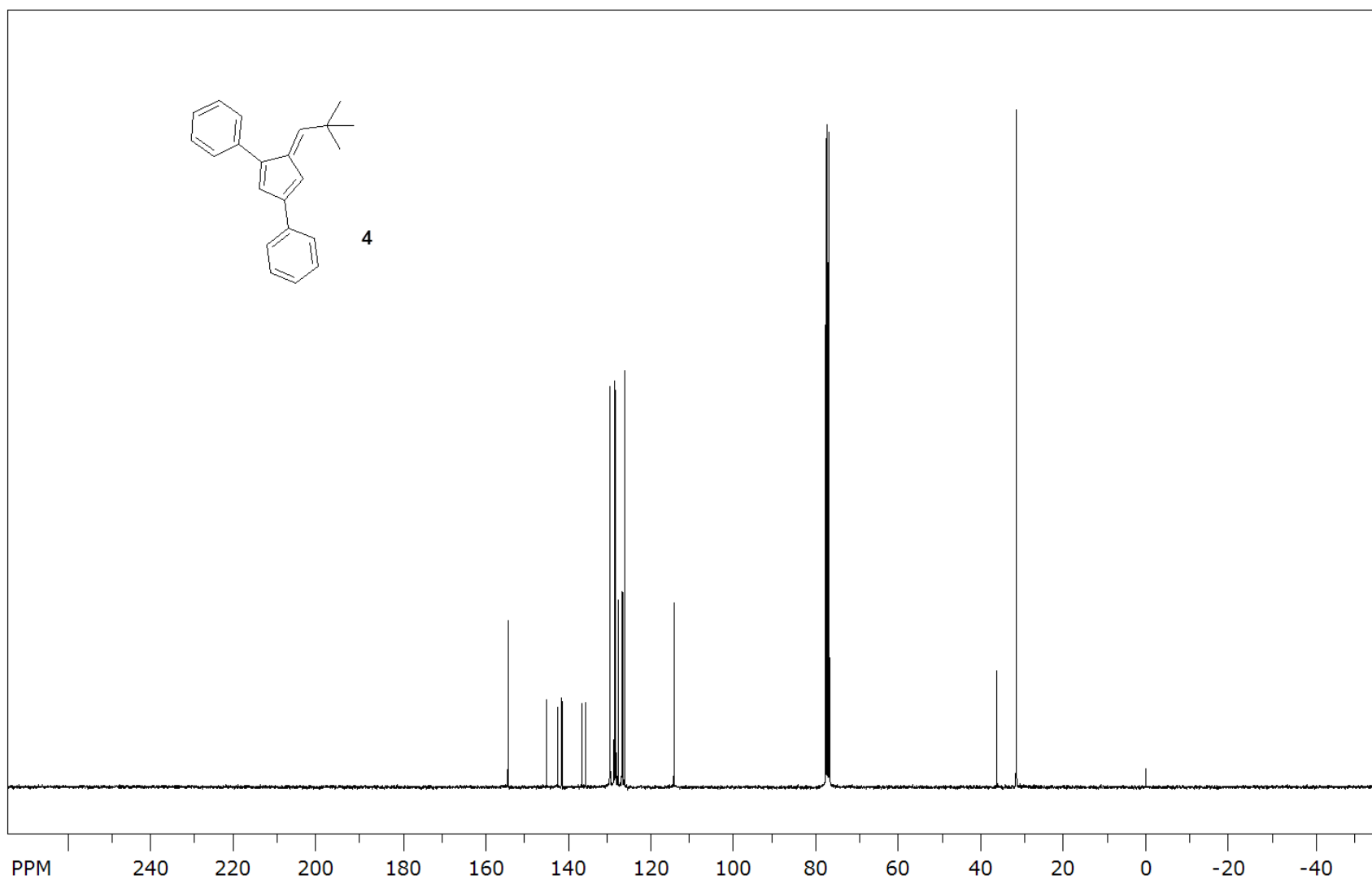


75 MHz ^{13}C NMR spectrum of 1,3-diphenylcyclopentadiene (recrystallized from EtOH) in CDCl_3



300 MHz ^1H NMR spectrum of 1,3-diphenyl-6-(tert-butyl)fulvene (recrystallized from EtOH) - dilute solution in CDCl_3 showing expansion of fulvene ring proton region.

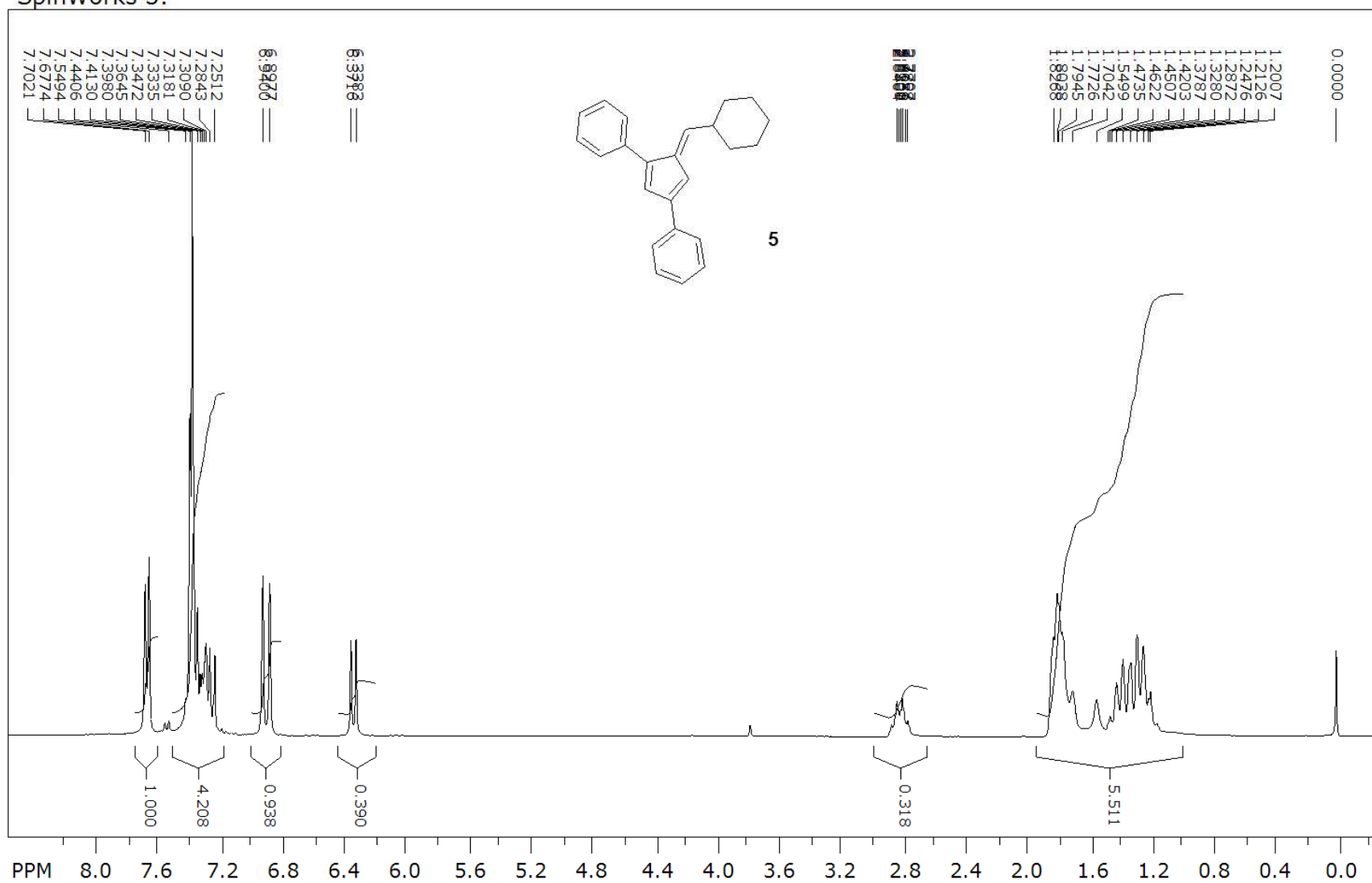




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time domain size: 90752 points
width: 25000.00 Hz = 331.3017 ppm = 0.275476 Hz/pt
number of scans: 30000

freq. of 0 ppm: 75.451647 MHz
processed size: 131072 complex points
LB: 0.000 GF: 0.0000
Hz/cm: 1000.000 ppm/cm: 13.25207

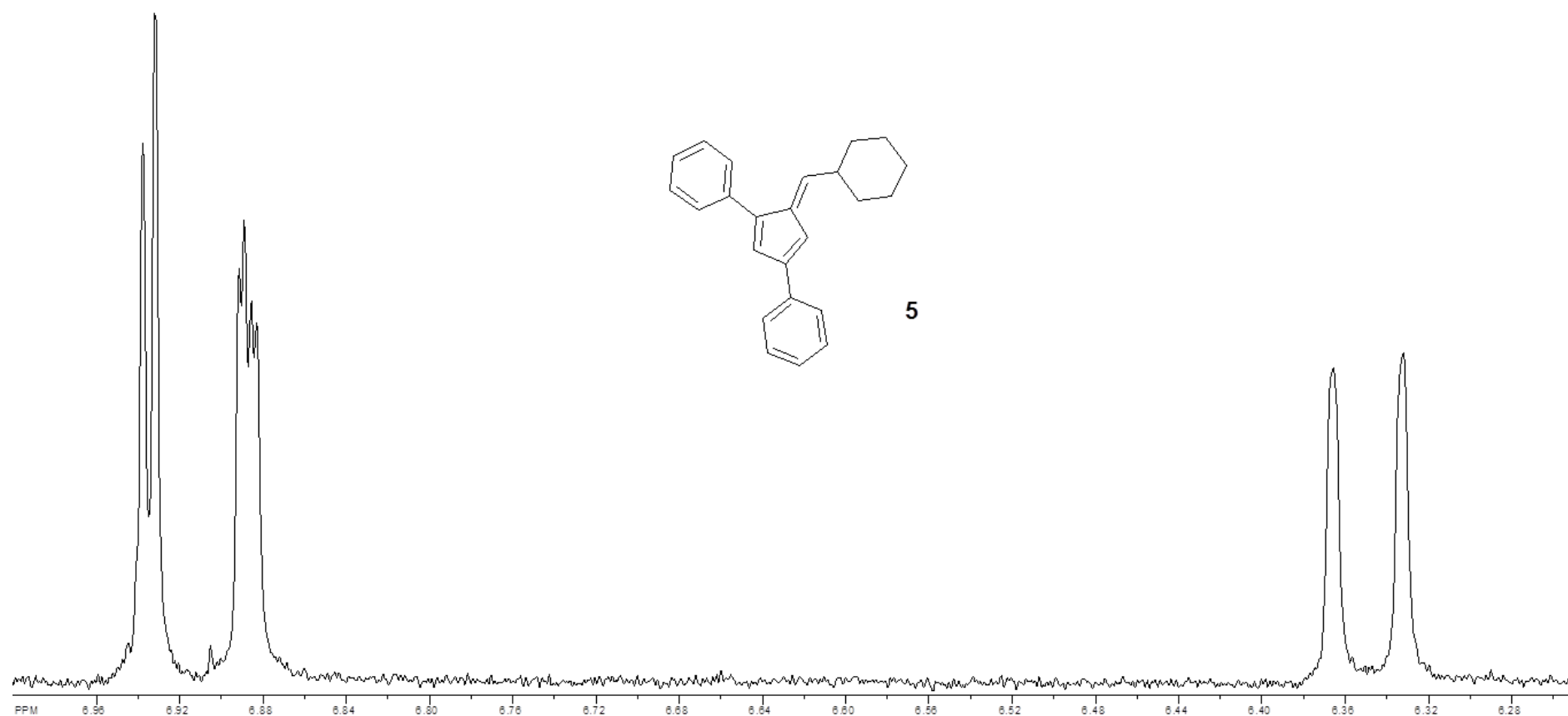
SpinWorks 3:



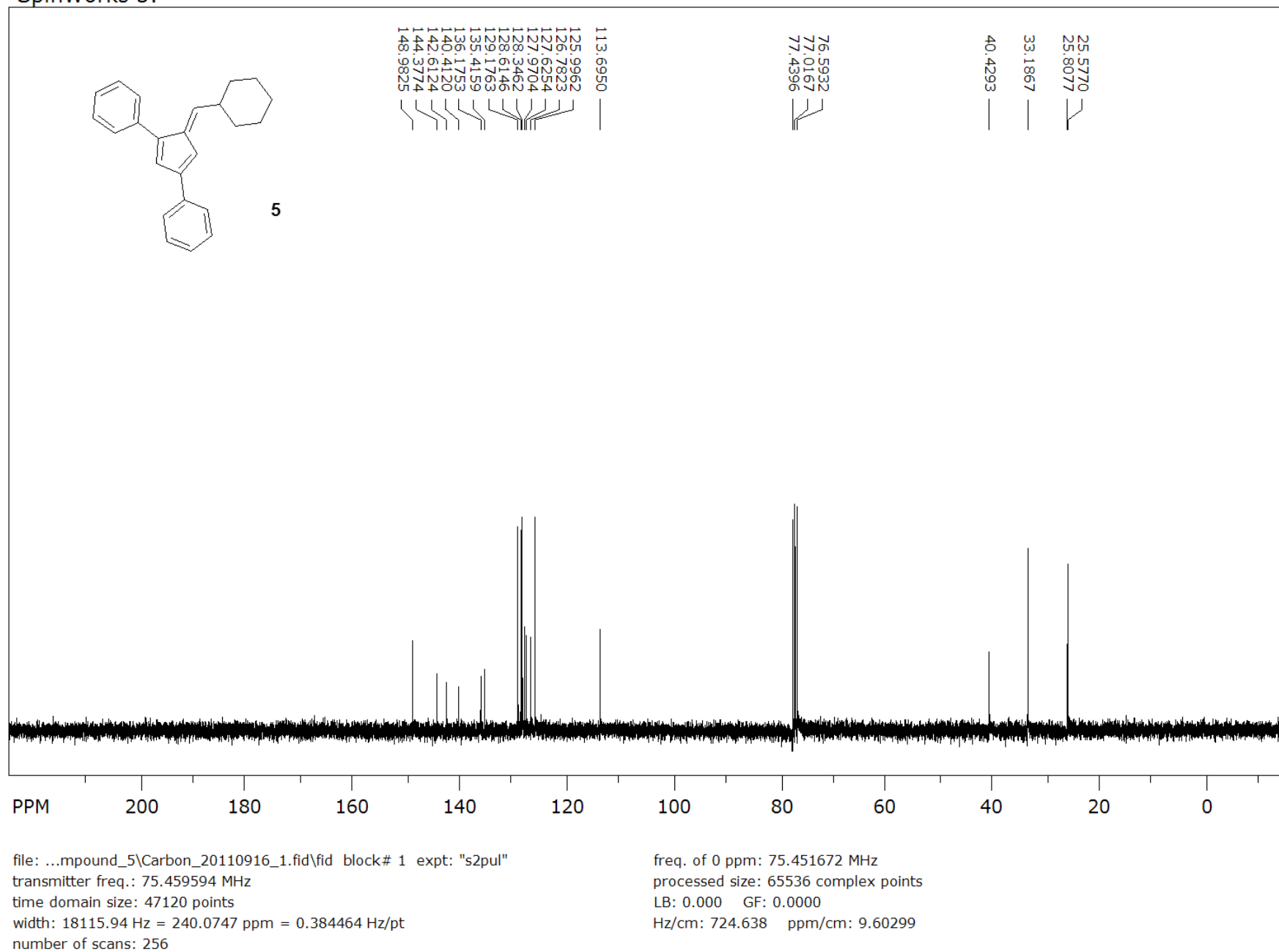
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 time domain size: 19184 points
 width: 4800.77 Hz = 15.9989 ppm = 0.250249 Hz/pt
 number of scans: 16

freq. of 0 ppm: 300.066067 MHz
 processed size: 32768 complex points
 LB: 0.000 GF: 0.0000
 Hz/cm: 106.464 ppm/cm: 0.35480

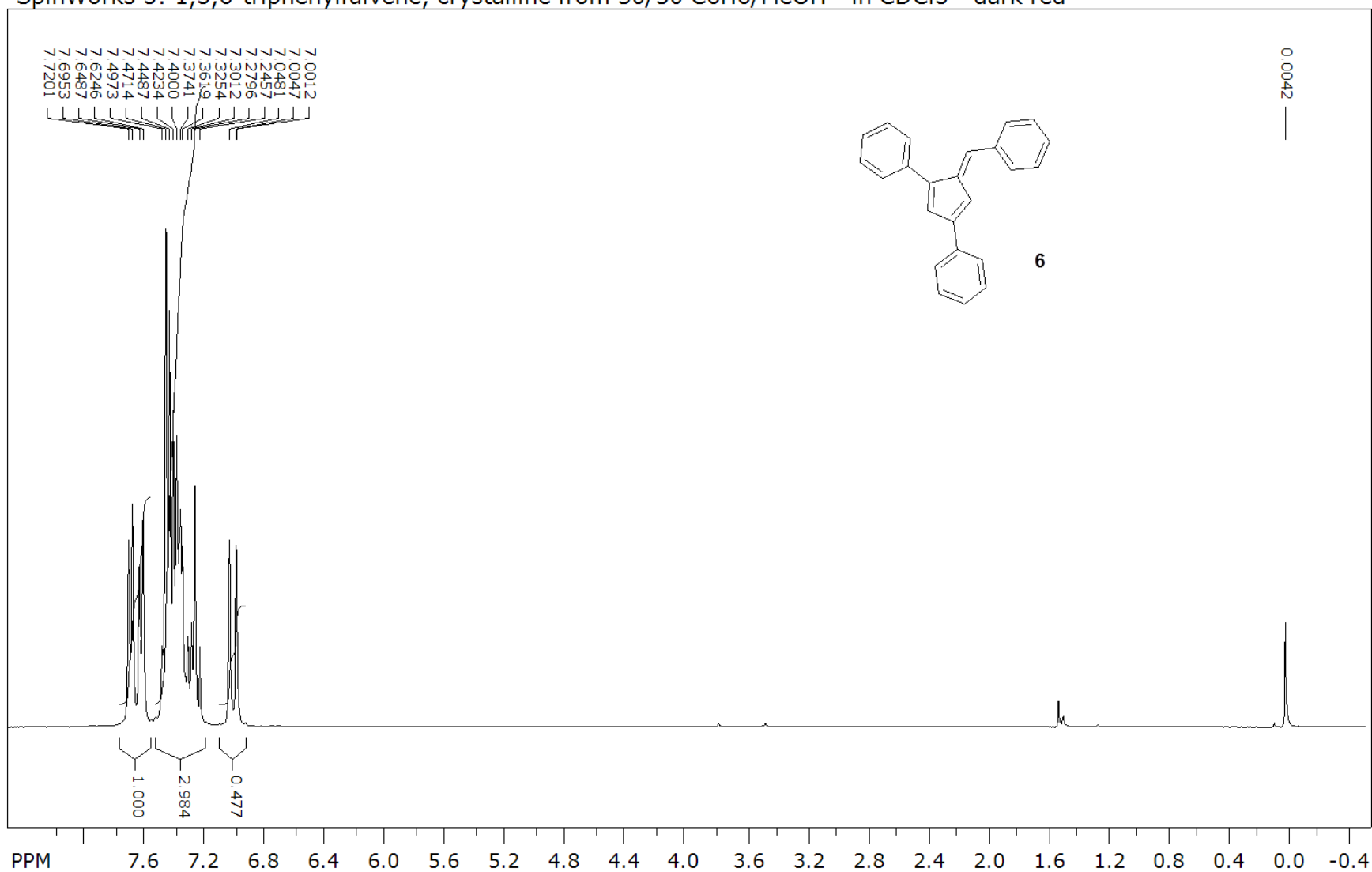
300 MHz ^1H NMR spectrum of 1,3-diphenyl-6-(cyclohexyl)fulvene - dilute solution in CDCl_3 showing expansion of fulvene ring proton region.



SpinWorks 3:



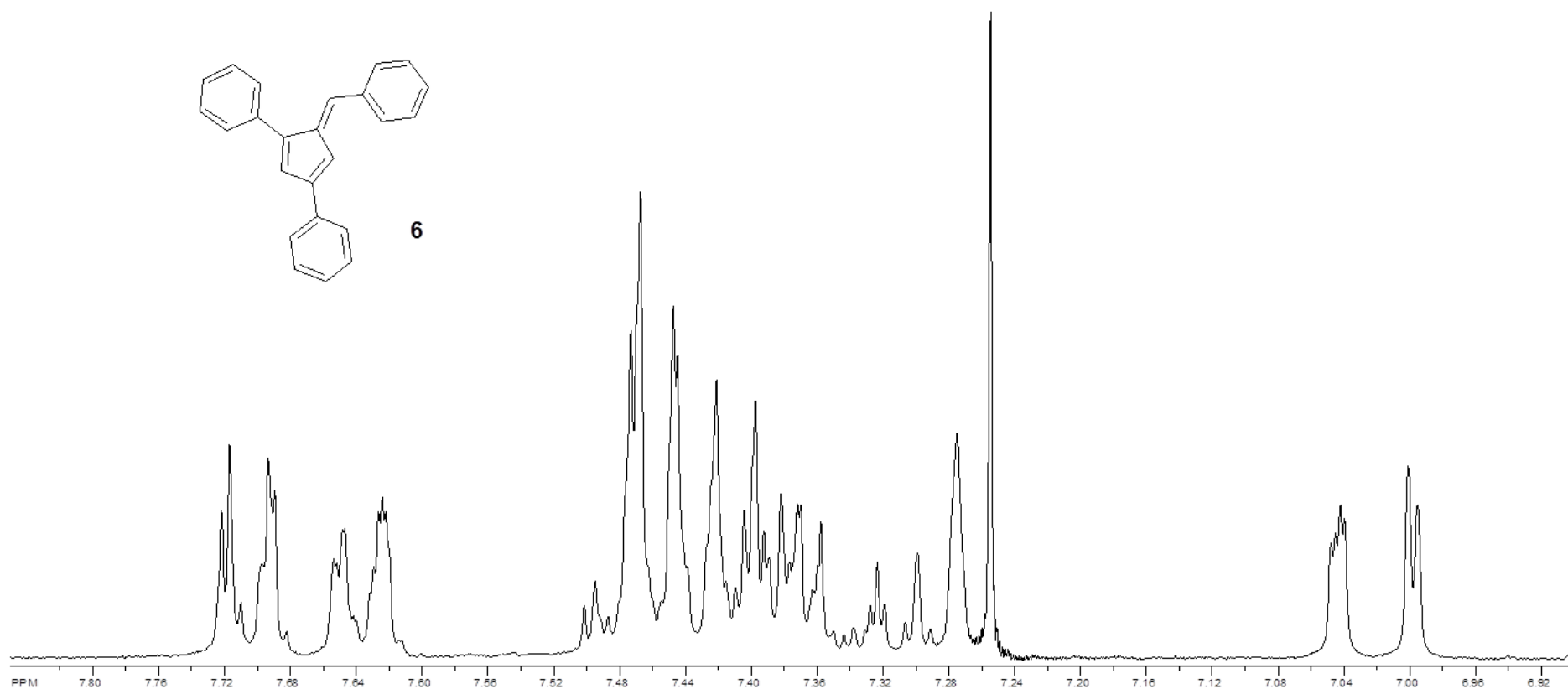
SpinWorks 3: 1,3,6-triphenylfulvene, crystalline from 50/50 C6H6/MeOH - in CDCl3 - dark red



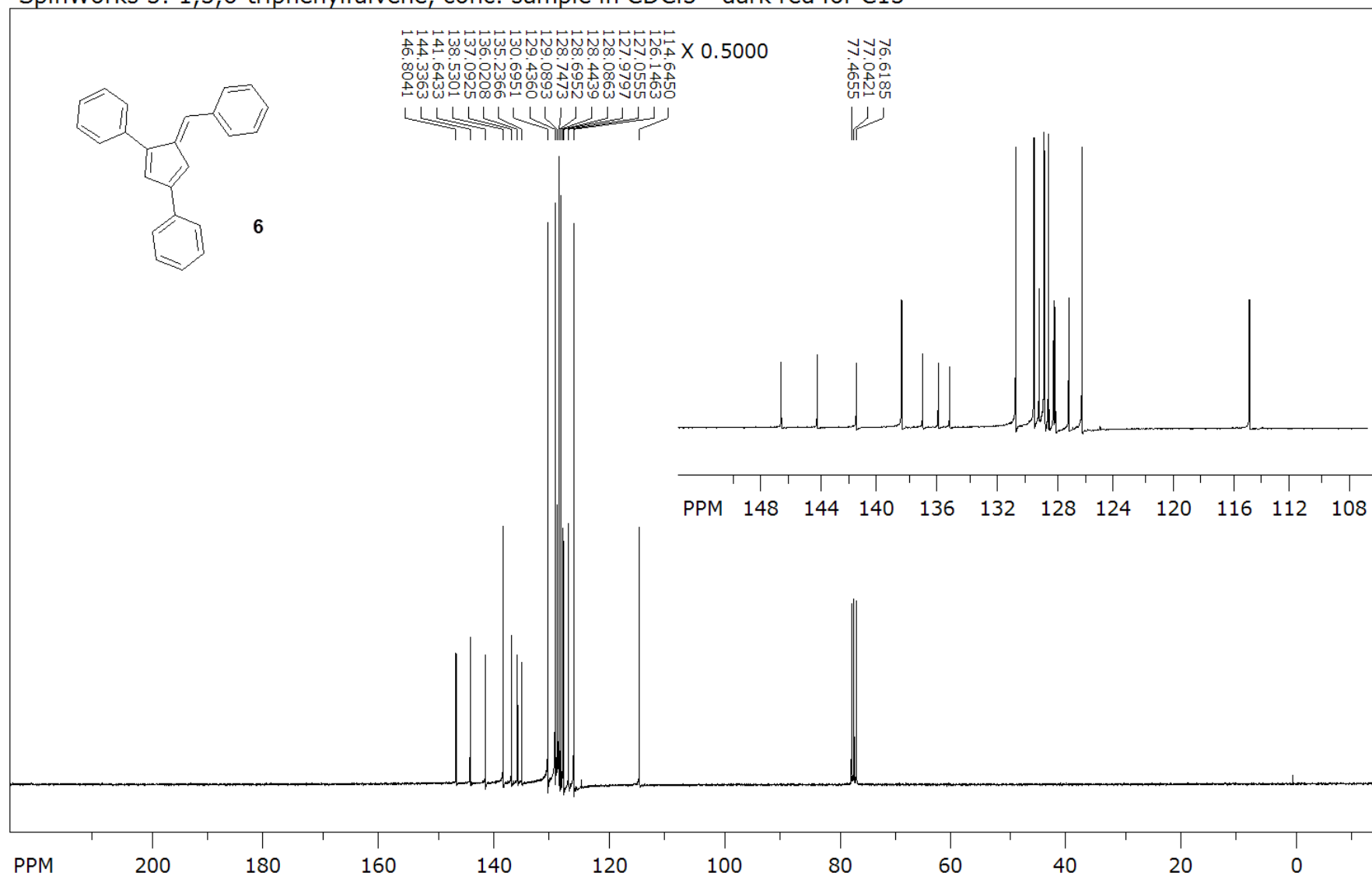
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 time domain size: 19184 points
 width: 4800.77 Hz = 15.9989 ppm = 0.250249 Hz/pt
 number of scans: 16

freq. of 0 ppm: 300.066068 MHz
 processed size: 32768 complex points
 LB: 0.000 GF: 0.0000
 Hz/cm: 108.768 ppm/cm: 0.36248

300 MHz ^1H NMR spectrum of 1,3,6-triphenylfulvene - dilute solution in CDCl_3 showing expansion of fulvene ring and Ph ring proton region.

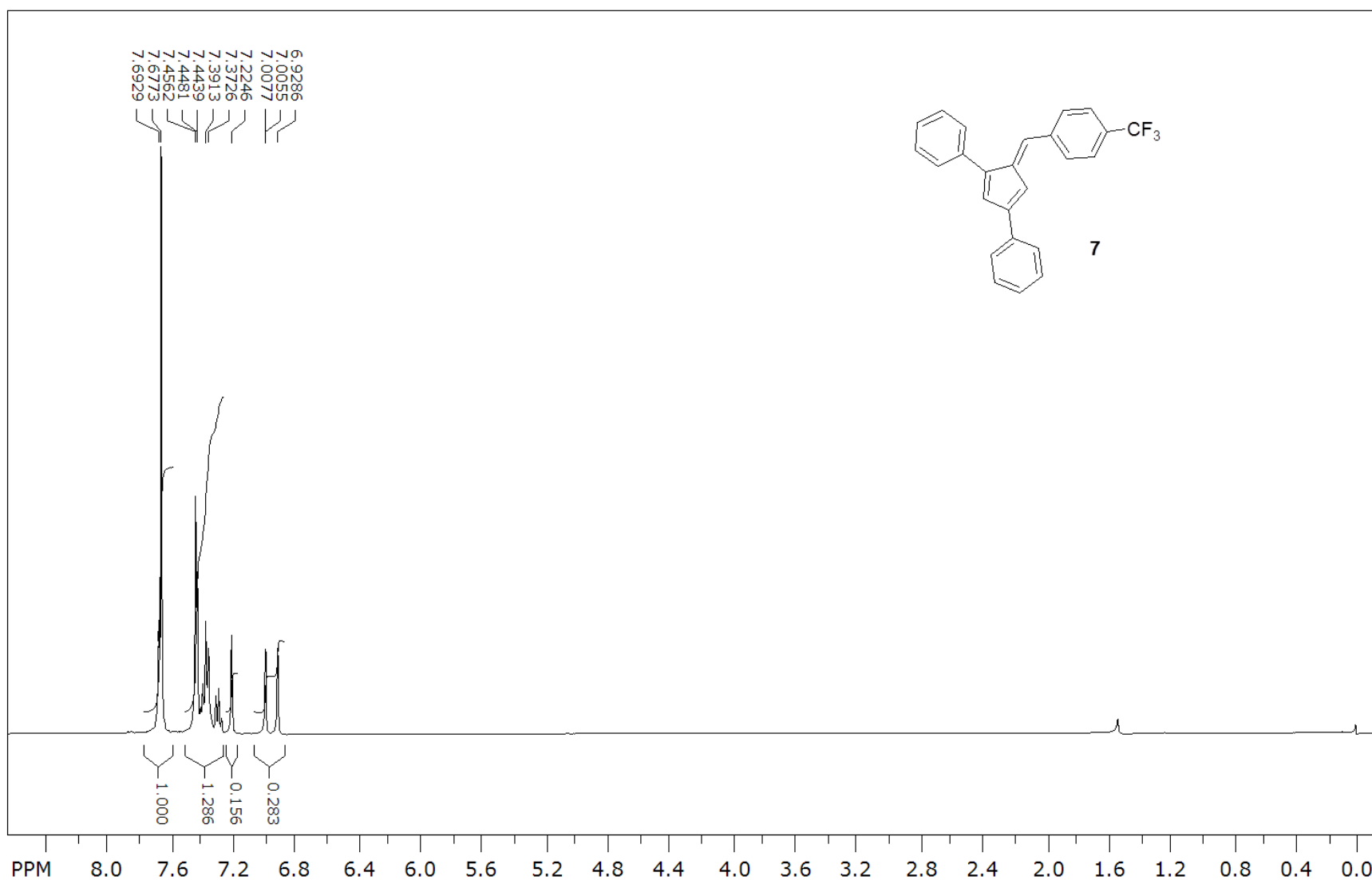


SpinWorks 3: 1,3,6-triphenylfulvene, conc. sample in CDCl₃ - dark red for C13



file: ...6H6_MeOH\Carbon_20090803_2.fid\fid block# 1 expt: "s2pul"
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 time domain size: 47120 points
 width: 18115.94 Hz = 240.0747 ppm = 0.384464 Hz/pt
 number of scans: 16176

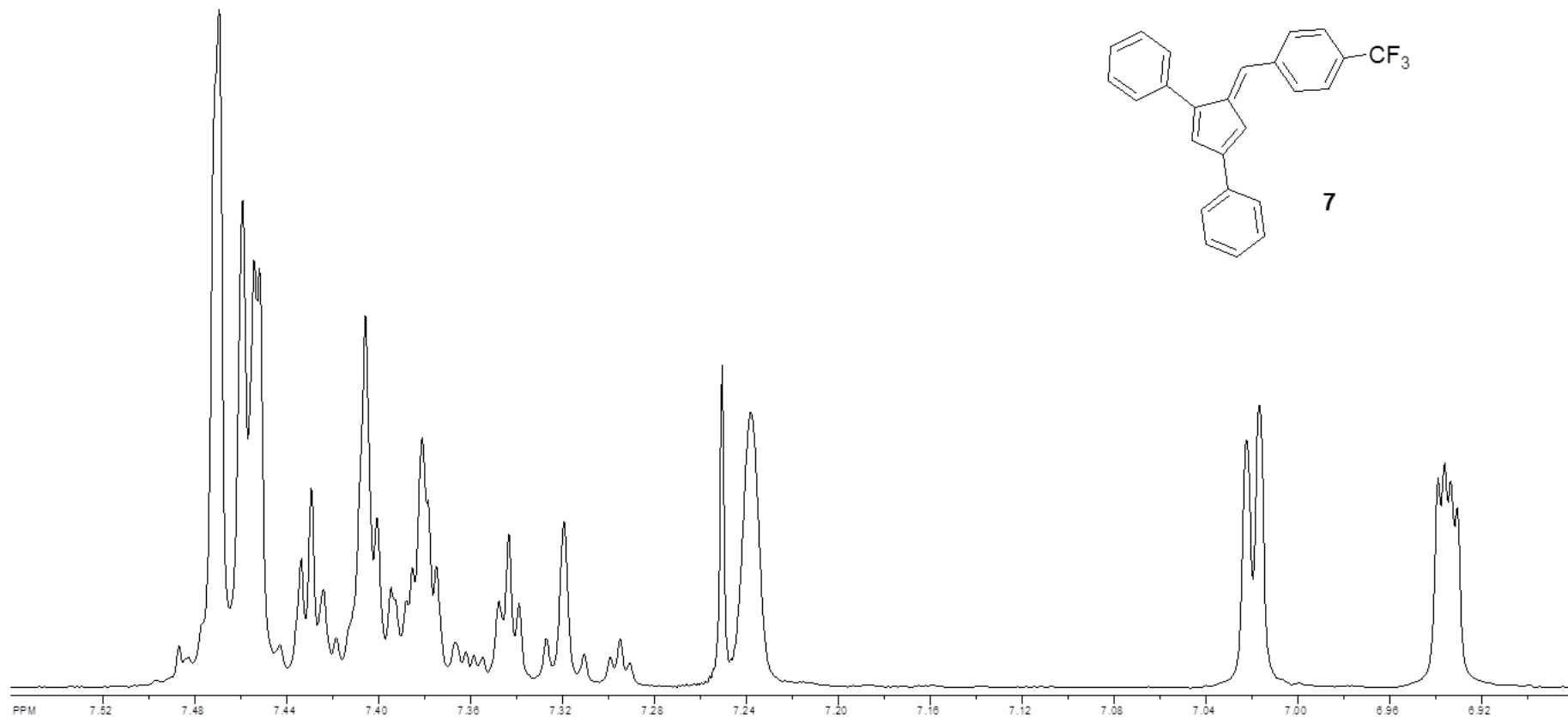
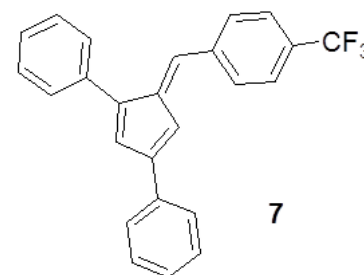
freq. of 0 ppm: 75.451672 MHz
 processed size: 65536 complex points
 LB: 0.000 GF: 0.0000
 Hz/cm: 724.638 ppm/cm: 9.60299

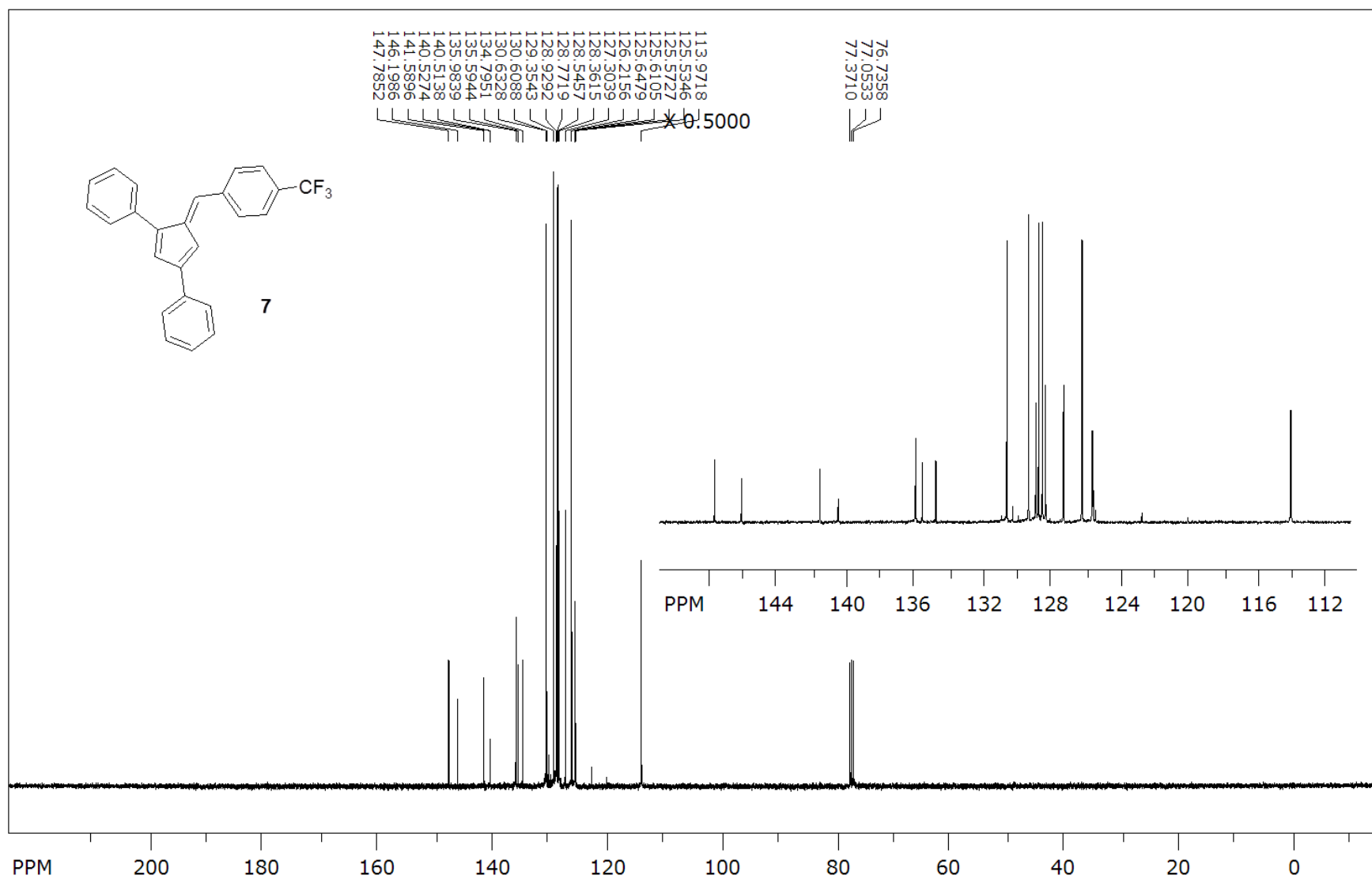


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 transmitter freq.: 400.108727 MHz
 time domain size: 25582 points
 width: 6402.05 Hz = 16.0008 ppm = 0.250256 Hz/pt
 number of scans: 16

freq. of 0 ppm: 400.106343 MHz
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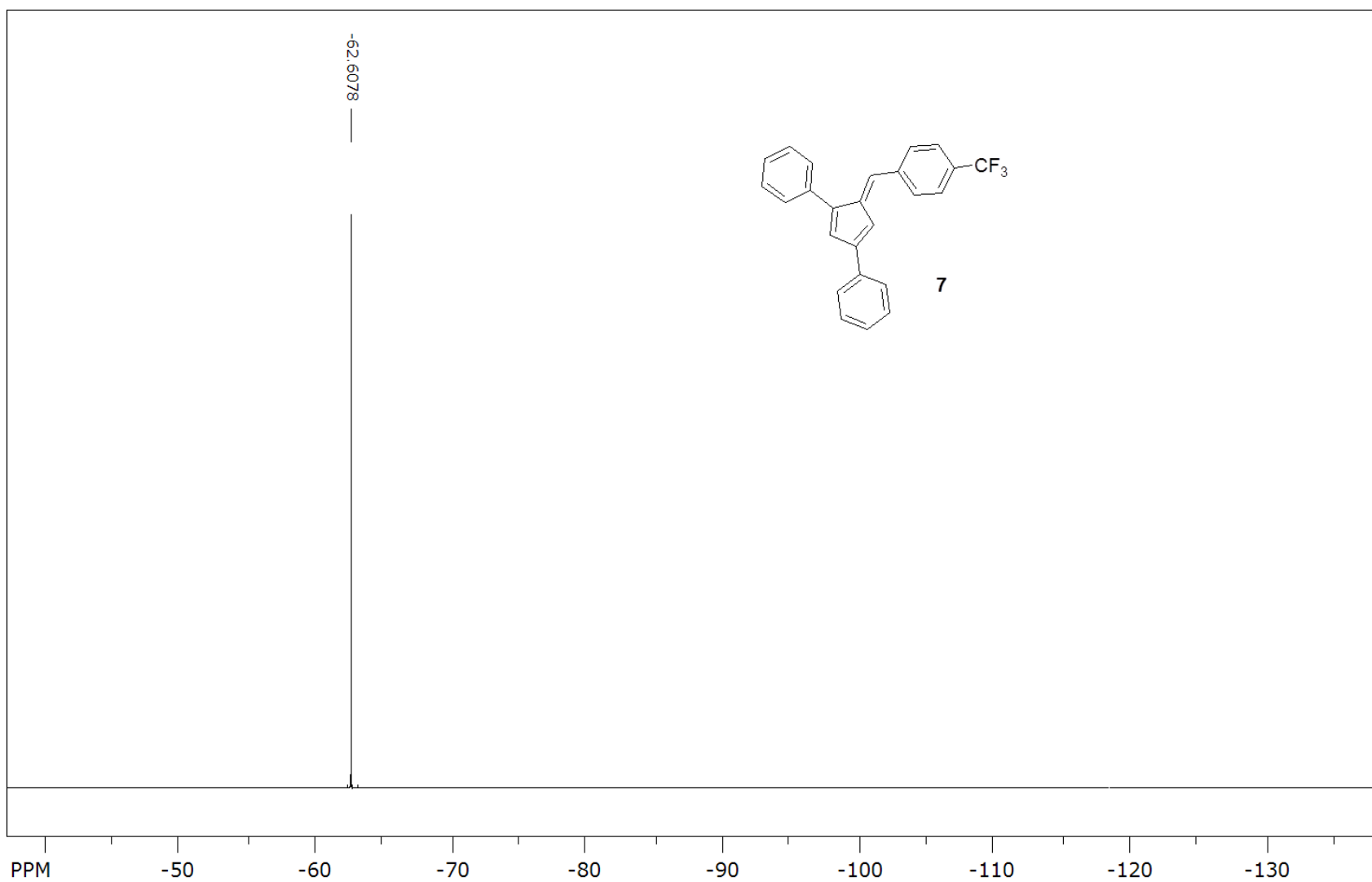
300 MHz ^1H NMR spectrum of 1,3-diphenyl-6-(4-trifluoromethylphenyl)fulvene - dilute solution in CDCl_3 showing expansion of fulvene ring, Ph ring and 4-trifluoromethylphenyl ring proton region.





file: ...nstr_13C\Carbon_20110706_1.fid\fid_block# 1 expt: "s2pul"
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 time domain size: 62826 points
 width: 24154.59 Hz = 240.0638 ppm = 0.384468 Hz/pt
 number of scans: 512

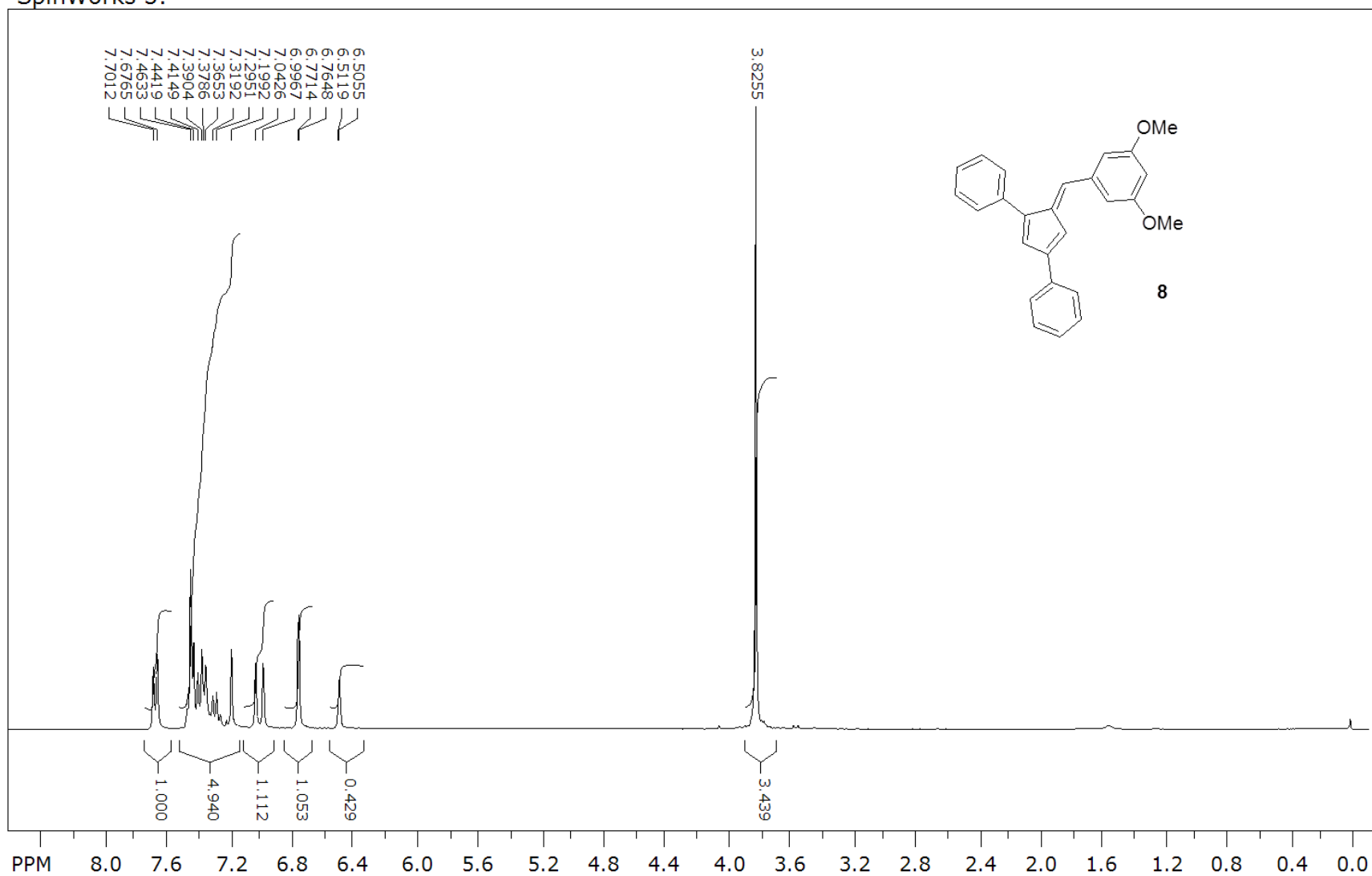
freq. of 0 ppm: 100.606816 MHz
 processed size: 65536 complex points
 LB: 0.000 GF: 0.0000
 Hz/cm: 966.184 ppm/cm: 9.60255



file: ...tr_19F\Fluorine_20110706_1.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 376.427148 MHz
time domain size: 128000 points
width: 75187.97 Hz = 199.7411 ppm = 0.587406 Hz/pt
number of scans: 16

freq. of 0 ppm: 376.476090 MHz
processed size: 131072 complex points
LB: 0.000 GF: 0.0000
Hz/cm: 1525.665 ppm/cm: 4.05302

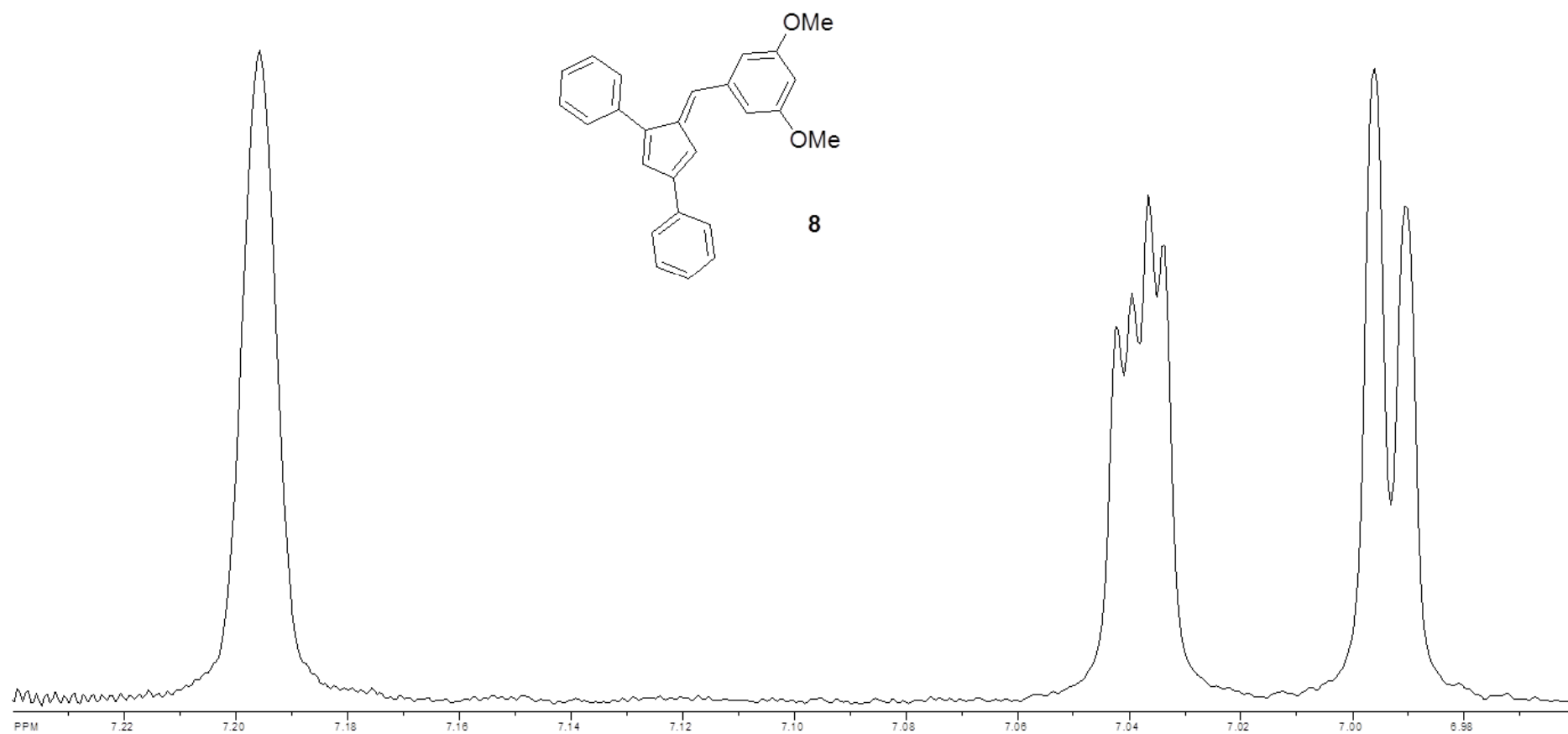
SpinWorks 3:



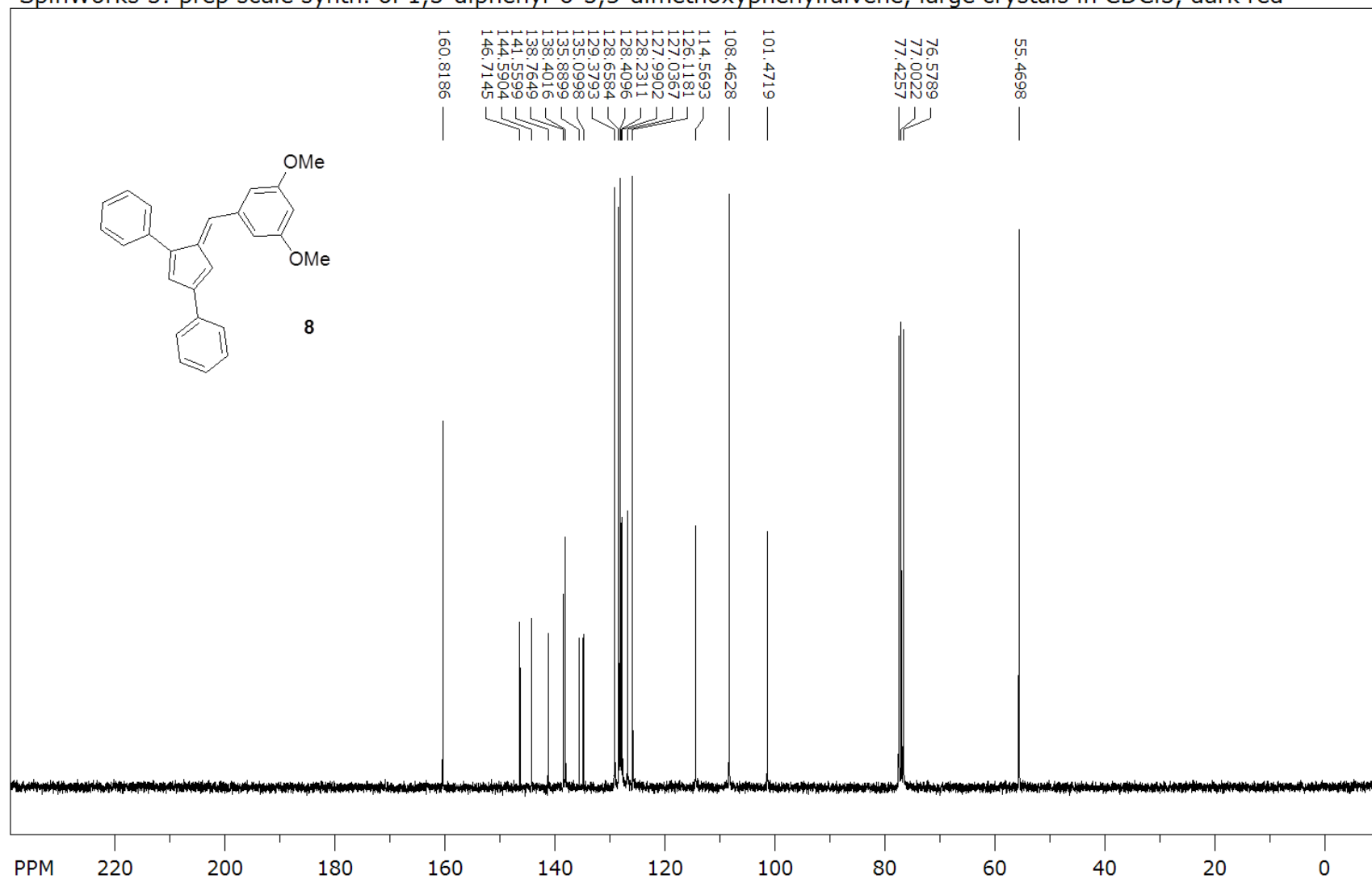
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 width: 4800.77 Hz = 15.9989 ppm = 0.250249 Hz/pt
 number of scans: 16

freq. of 0 ppm: 300.066073 MHz
 processed size: 32768 complex points
 LB: 0.000 GF: 0.0000
 Hz/cm: 105.148 ppm/cm: 0.35041

300 MHz ^1H NMR spectrum of 1,3-diphenyl-6-(3,5-dimethoxyphenyl)fulvene - dilute solution in CDCl_3 showing expansion of fulvene ring proton region.

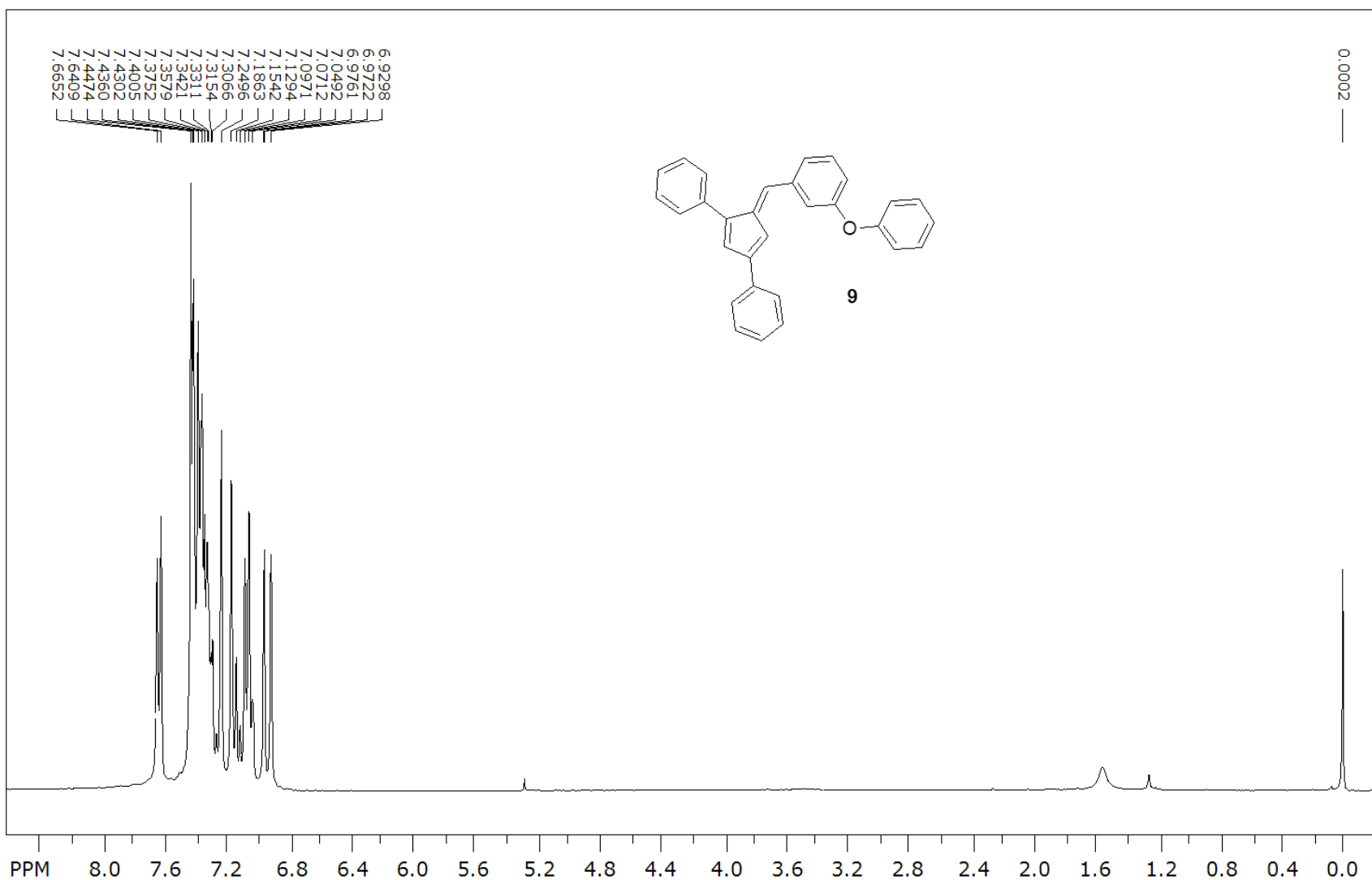


SpinWorks 3: prep scale synth. of 1,3-diphenyl-6-3,5-dimethoxyphenylfulvene, large crystals in CDCl₃, dark red



file: ..._CH2Cl2_ETOH_cold_small_refrig\fid_block# 1_expt: "s2pul"
transmitter freq.: 75.459929 MHz
time domain size: 90752 points
width: 25000.00 Hz = 331.3017 ppm = 0.275476 Hz/pt
number of scans: 1522

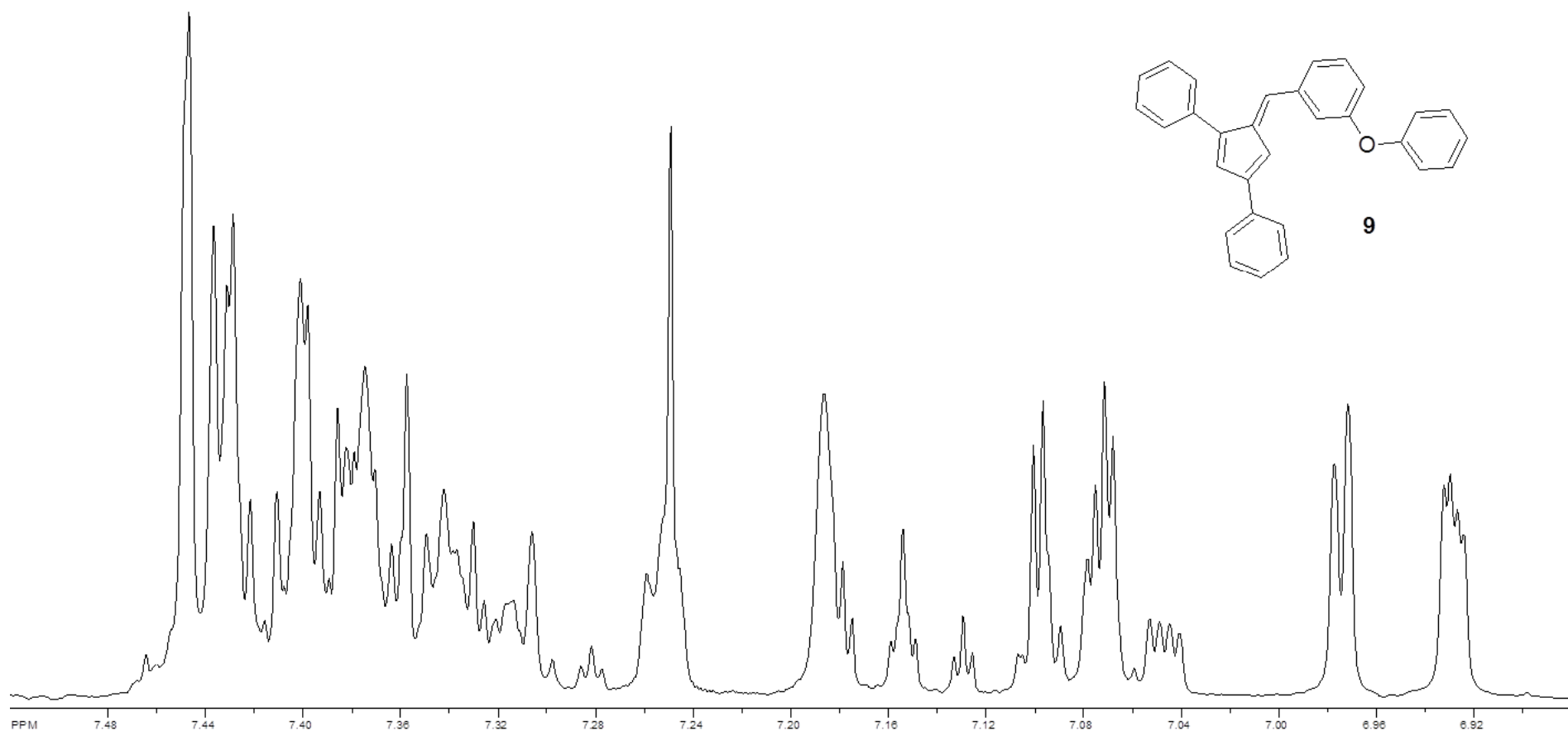
freq. of 0 ppm: 75.451650 MHz
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LB: 0.000 GF: 0.0000
Hz/cm: 755.784 ppm/cm: 10.01570

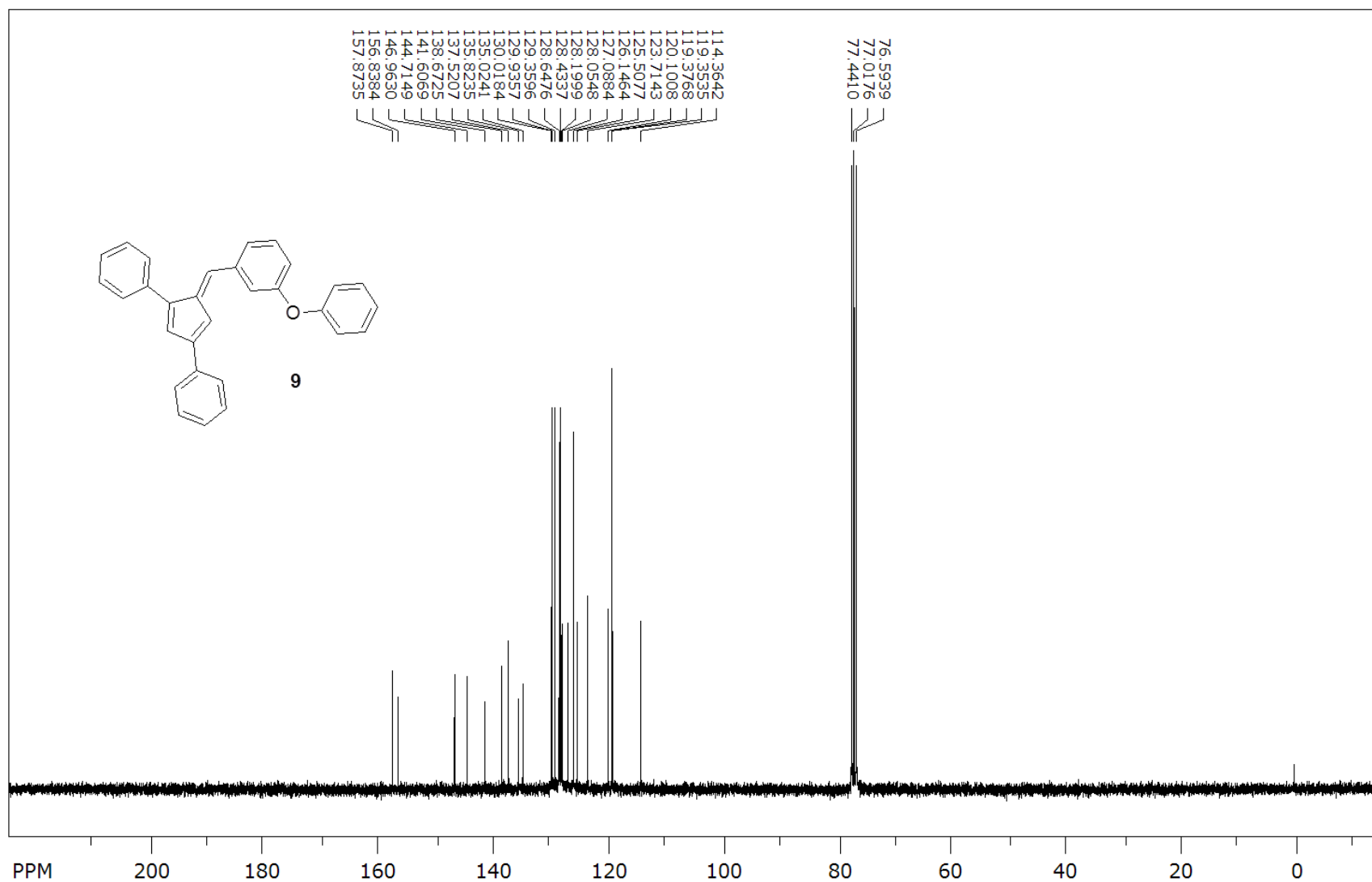


file: ...Compound\Proton_20100505_1.fid\fid block# 1 expt: "s2pul"
 transmitter freq.: 300.067865 MHz
 time domain size: 19184 points
 width: 4800.77 Hz = 15.9989 ppm = 0.250249 Hz/pt
 number of scans: 16

freq. of 0 ppm: 300.066067 MHz
 processed size: 32768 complex points
 LB: 0.000 GF: 0.0000
 Hz/cm: 106.300 ppm/cm: 0.35425

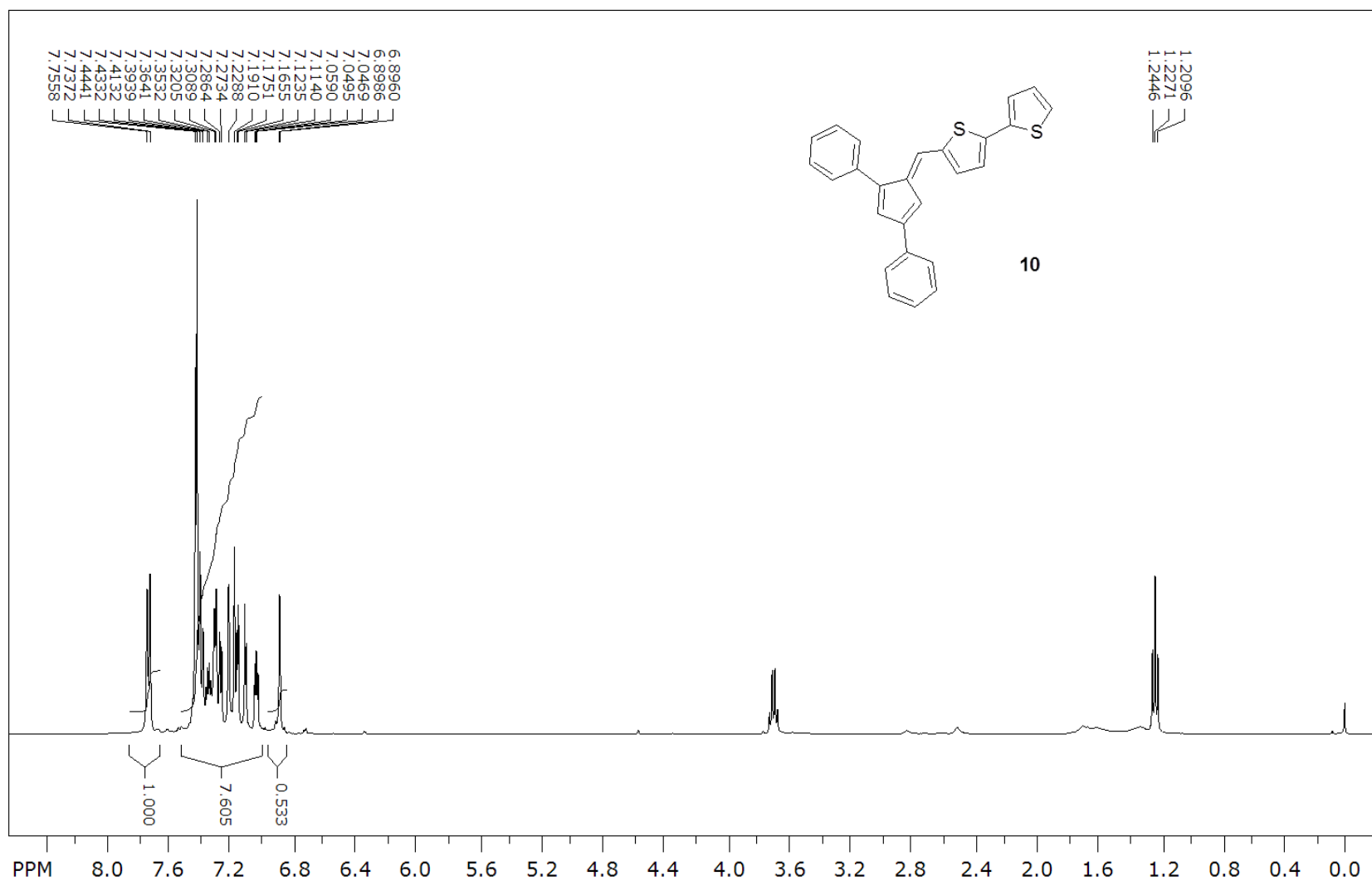
300 MHz ^1H NMR spectrum of 1,3-diphenyl-6-(3-phenoxyphenyl)fulvene - dilute solution in CDCl_3 showing expansion of fulvene ring, Ph ring, and phenoxyphenyl ring proton region.





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 number of scans: 2528

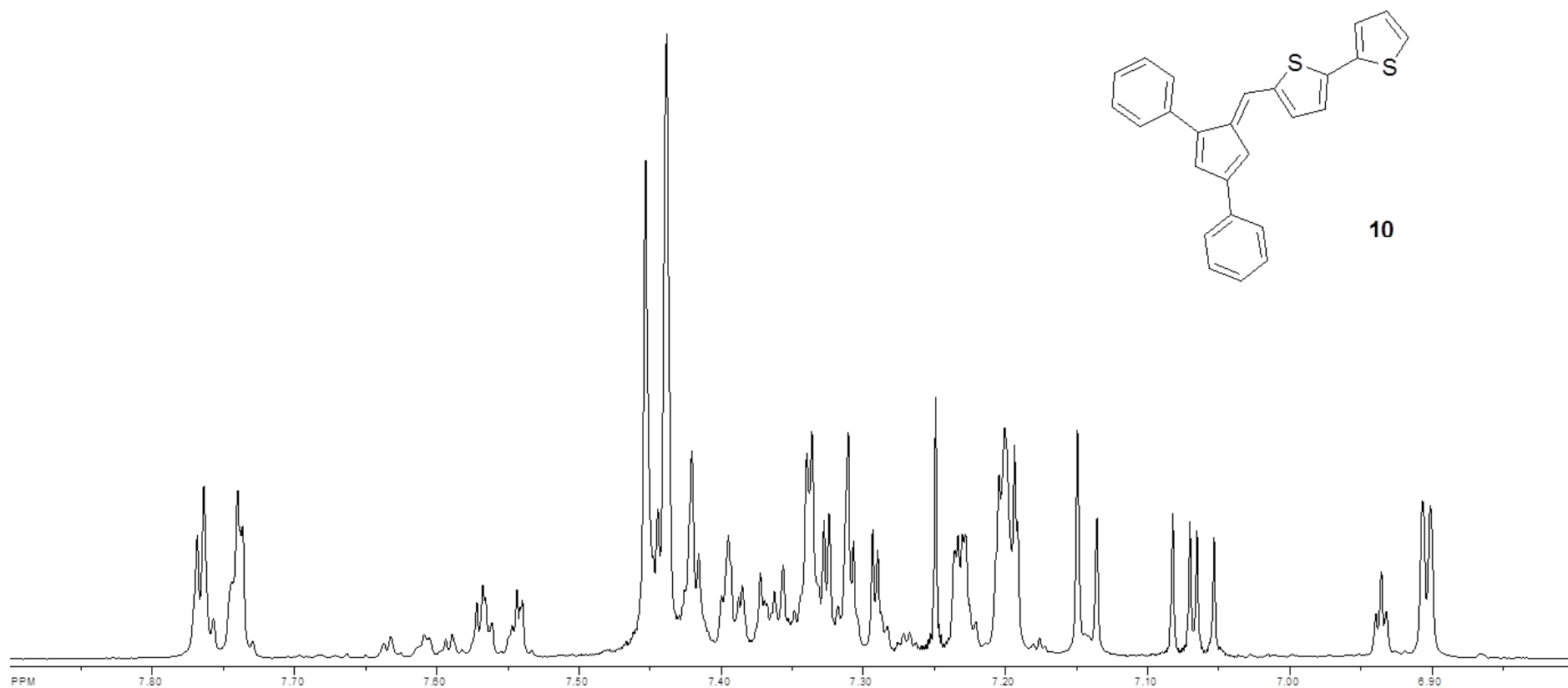
freq. of 0 ppm: 75.451672 MHz
 processed size: 65536 complex points
 LB: 0.000 GF: 0.0000
 Hz/cm: 724.638 ppm/cm: 9.60299

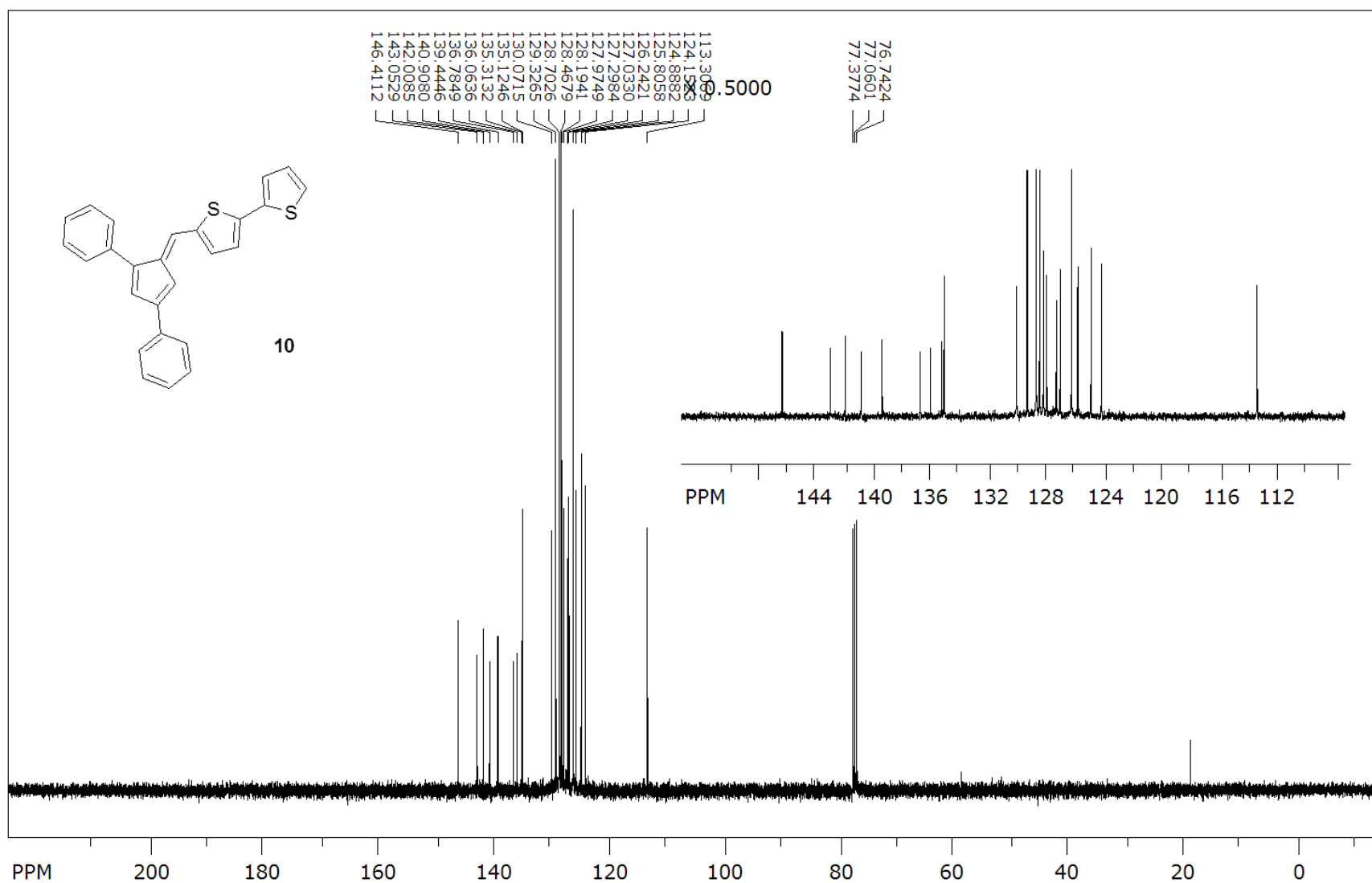


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number of scans: 8

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Hz/cm: 141.975 ppm/cm: 0.35484

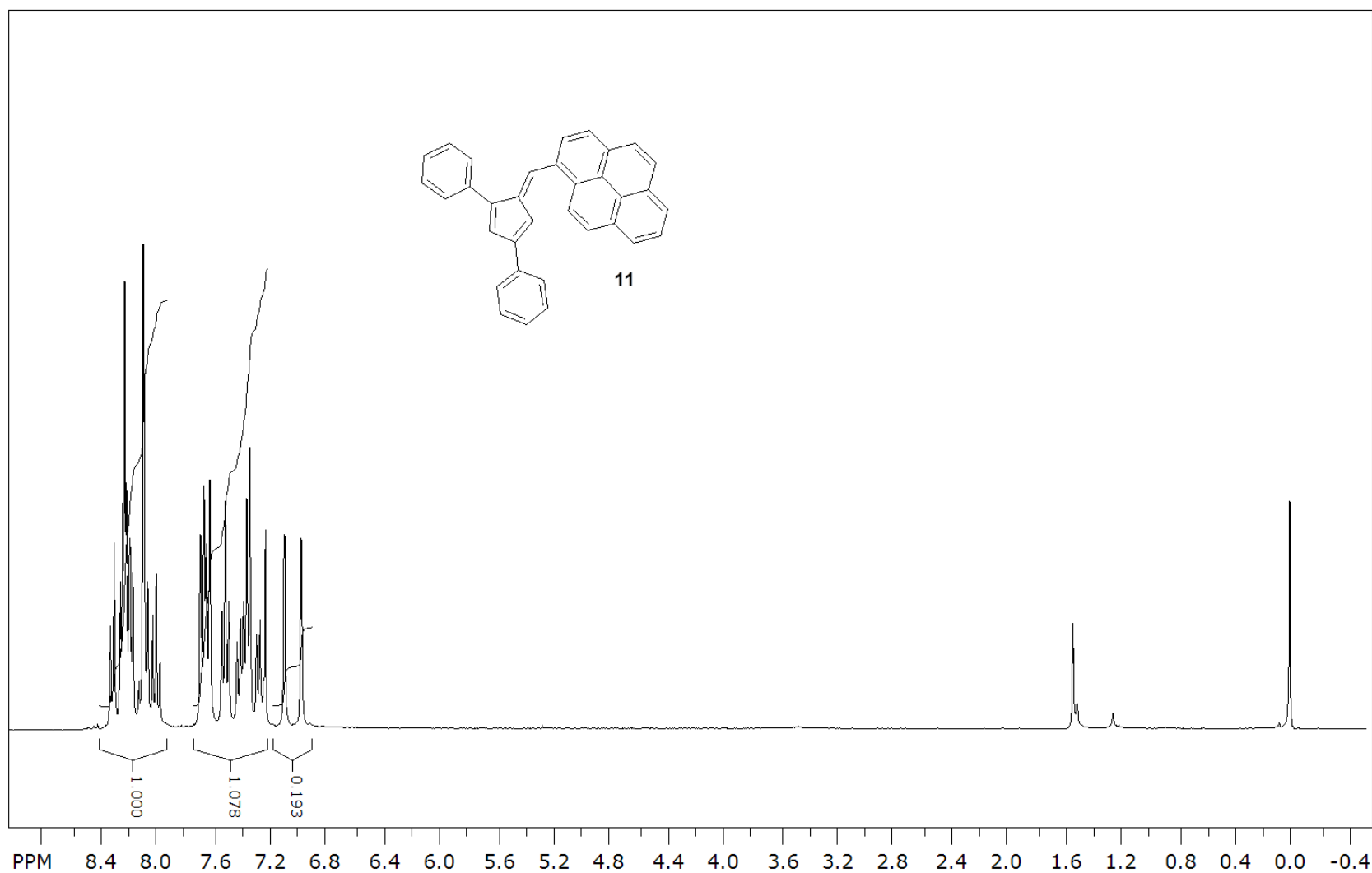
300 MHz ^1H NMR spectrum of 1,3-diphenyl-6-(5-[2,2'-bithiophene])fulvene - dilute solution in CDCl_3 showing expansion of fulvene ring, Ph ring, and bithiophene ring proton region.





file: ...13C_data\Carbon_20110706_1.fid\fid block# 1 expt: "s2pul"
 transmitter freq.: 100.617379 MHz
 time domain size: 62826 points
 width: 24154.59 Hz = 240.0638 ppm = 0.384468 Hz/pt
 number of scans: 256

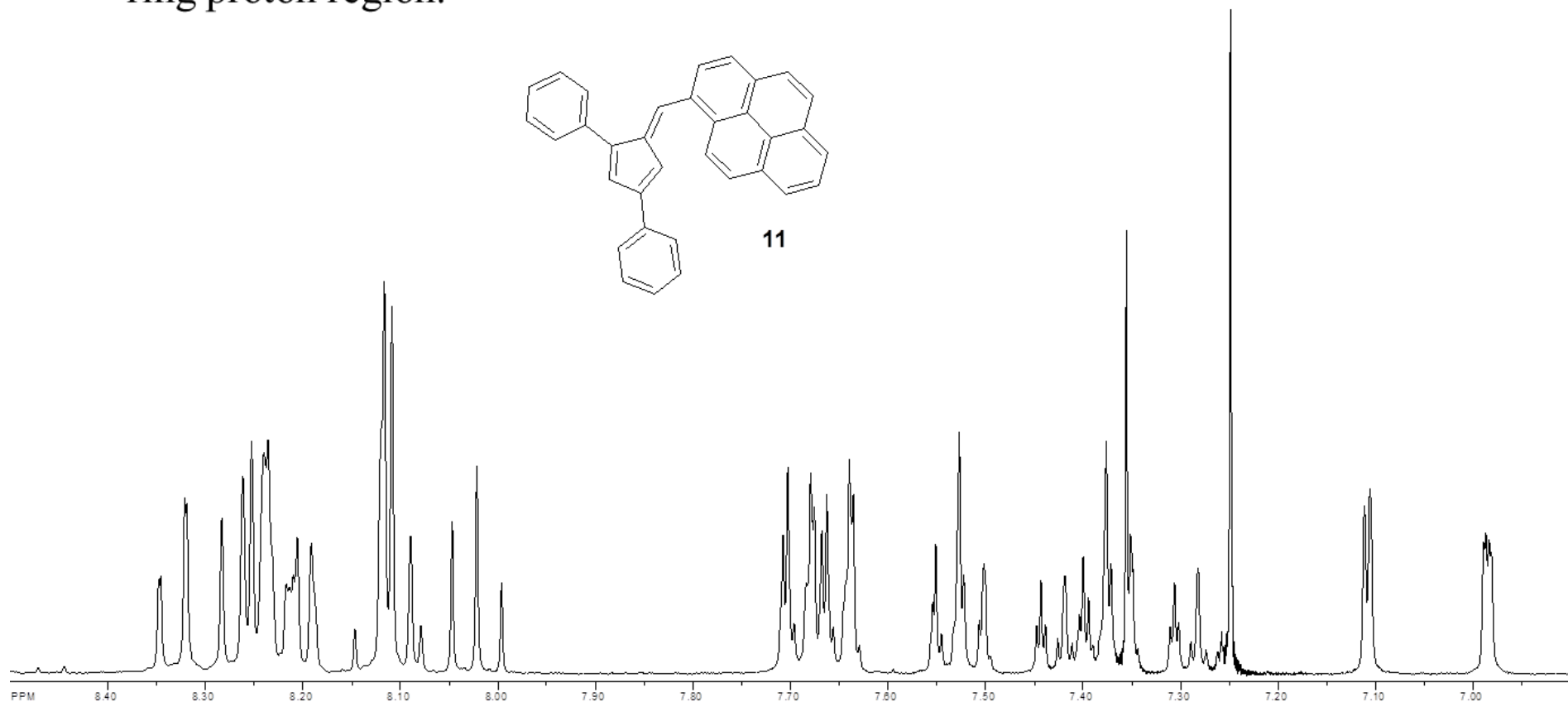
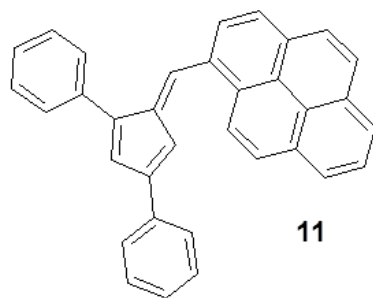
freq. of 0 ppm: 100.606816 MHz
 processed size: 65536 complex points
 LB: 0.000 GF: 0.0000
 Hz/cm: 966.184 ppm/cm: 9.60255

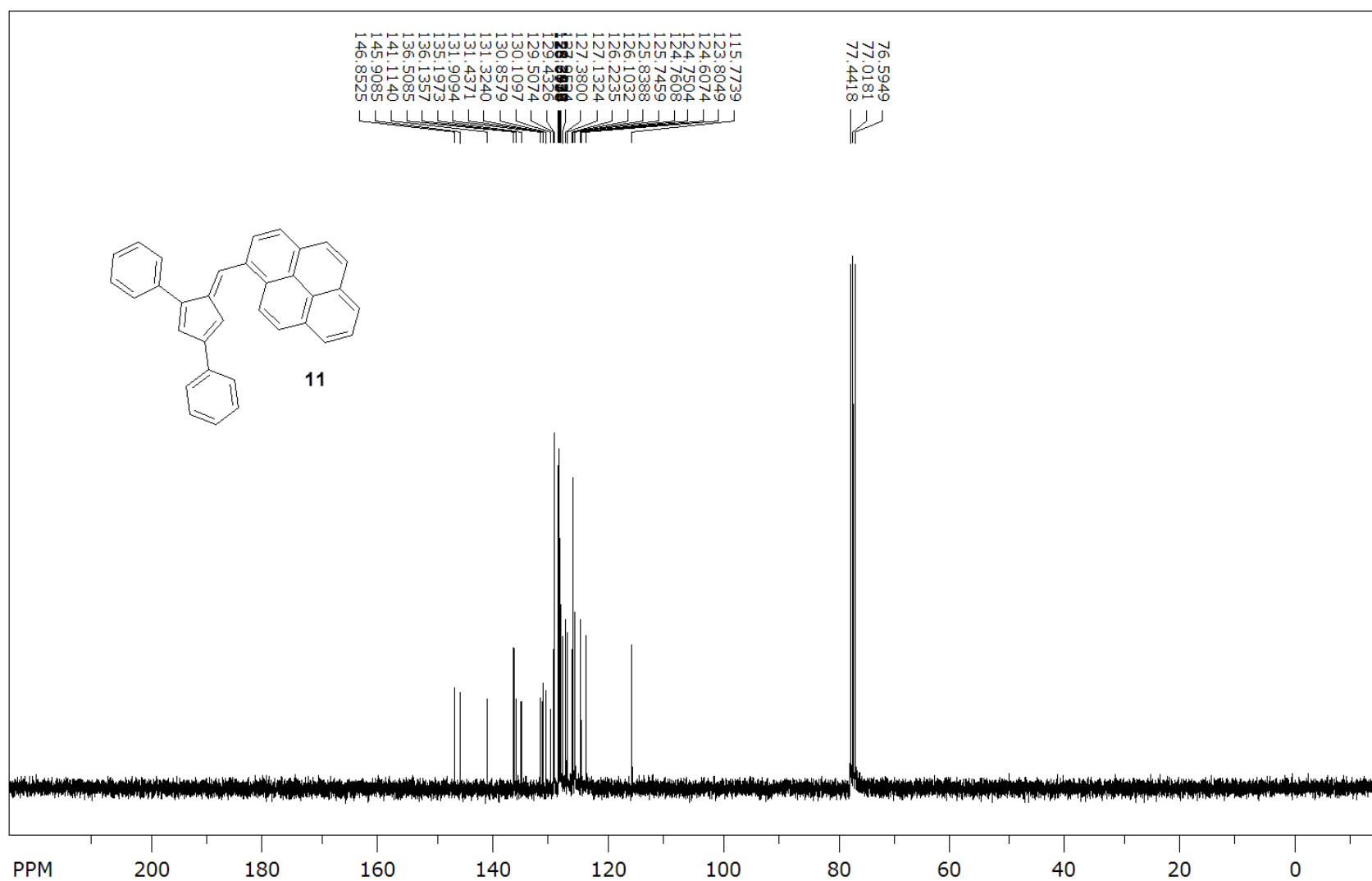


file: ...6H6_MeOH\Proton_20100505_1.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 300.067865 MHz
time domain size: 19184 points
width: 4800.77 Hz = 15.9989 ppm = 0.250249 Hz/pt
number of scans: 16

freq. of 0 ppm: 300.066068 MHz
processed size: 32768 complex points
LB: 0.000 GF: 0.0000
Hz/cm: 115.350 ppm/cm: 0.38441

300 MHz ^1H NMR spectrum of 1,3-diphenyl-6-(1-pyrene)fulvene - dilute solution in CDCl_3 showing expansion of fulvene ring, Ph ring and pyrene ring proton region.

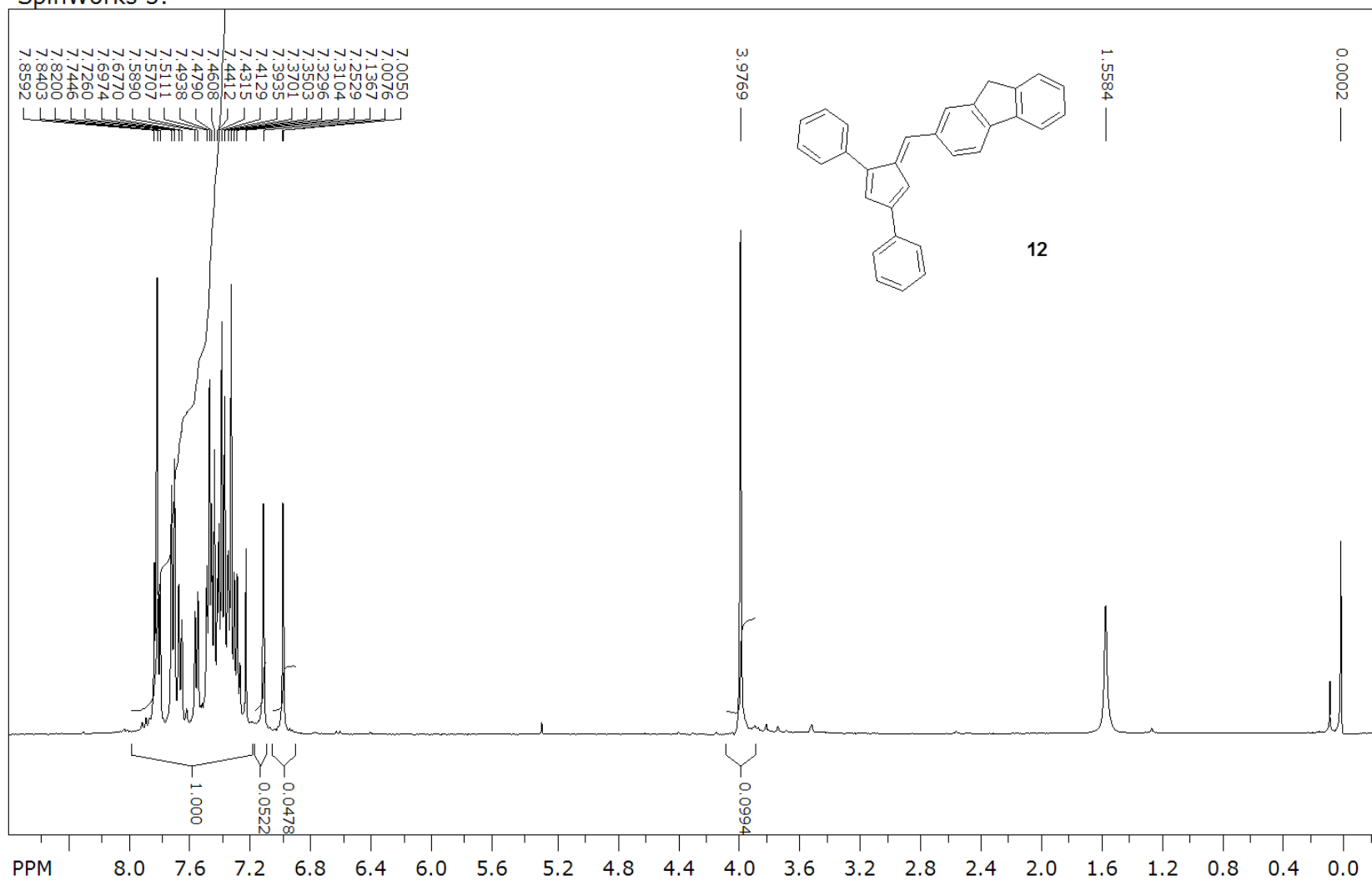




file: ...6H6_MeOH\Carbon_20100505_1.fid\fid block# 1 expt: "s2pul"
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 number of scans: 1648

freq. of 0 ppm: 75.451672 MHz
 processed size: 65536 complex points
 LB: 0.000 GF: 0.0000
 Hz/cm: 724.638 ppm/cm: 9.60299

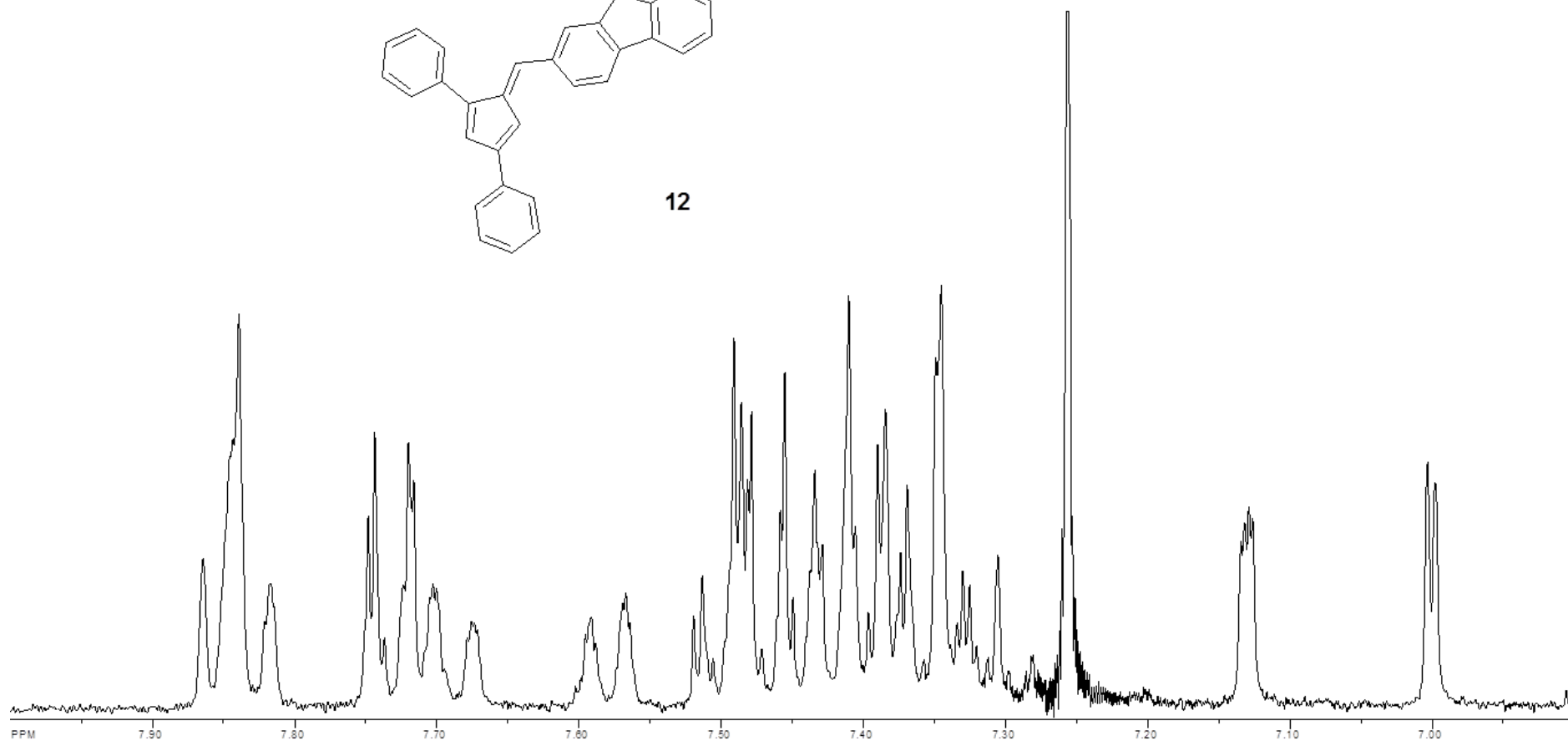
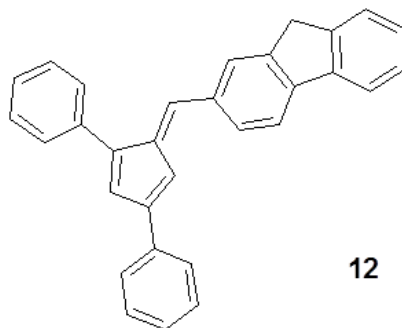
SpinWorks 3:



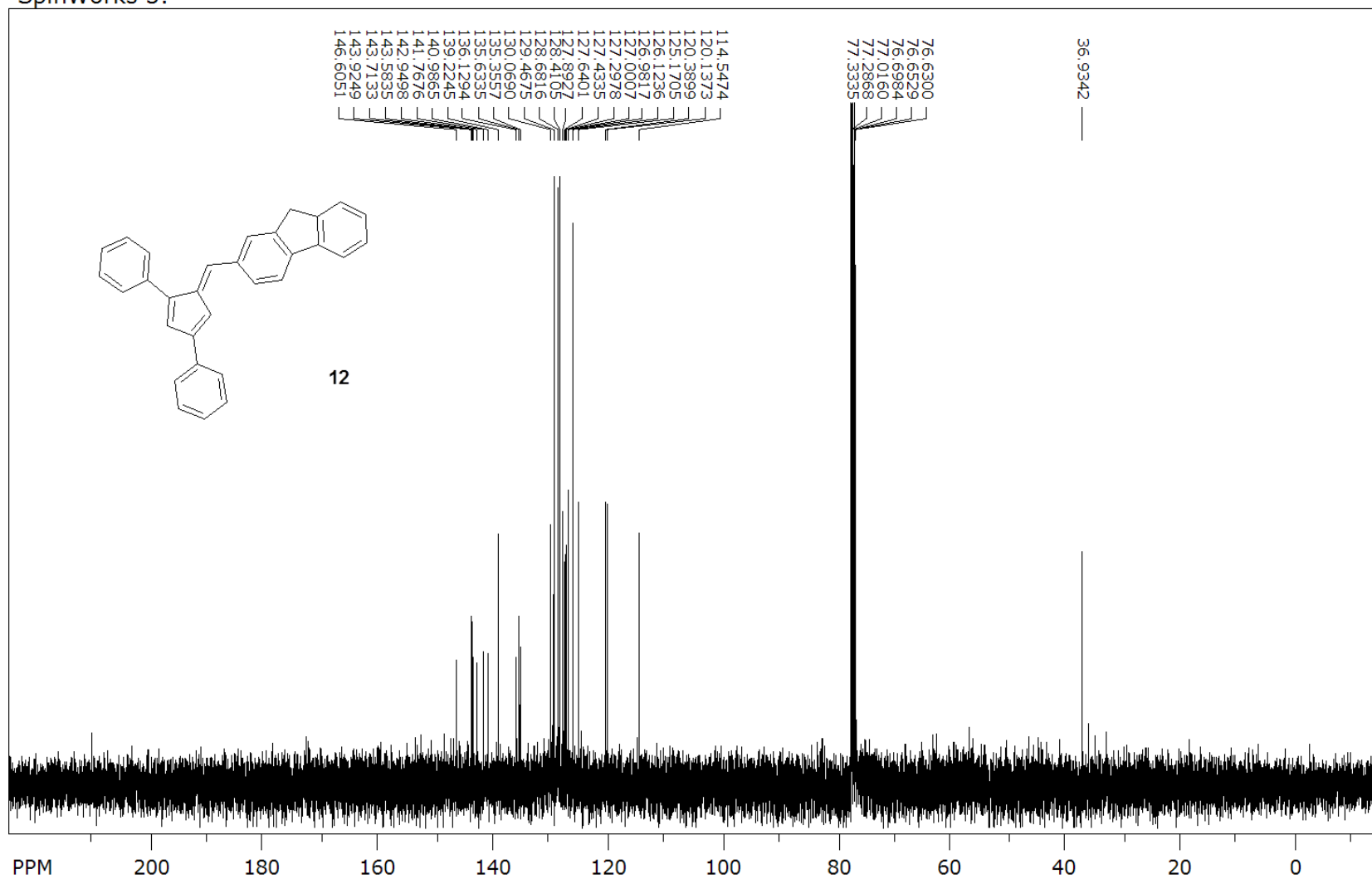
file: ...pound_12\Proton_20110916_1.fid\fid block# 1 expt: "s2pul"
 transmitter freq.: 400.108727 MHz
 time domain size: 25582 points
 width: 6402.05 Hz = 16.0008 ppm = 0.250256 Hz/pt
 number of scans: 16

freq. of 0 ppm: 400.106328 MHz
 processed size: 32768 complex points
 LB: 0.000 GF: 0.0000
 Hz/cm: 145.047 ppm/cm: 0.36252

300 MHz ^1H NMR spectrum of 1,3-diphenyl-6-(2-fluorenyl)fulvene - dilute solution in CDCl_3 showing expansion of fulvene ring, Ph ring and fluorenyl ring proton region.



SpinWorks 3:



file: ...pound_12\Carbon_20110916_1.fid\fid block# 1 expt: "s2pul"
 transmitter freq.: 100.617379 MHz
 time domain size: 62826 points
 width: 24154.59 Hz = 240.0638 ppm = 0.384468 Hz/pt
 number of scans: 256

freq. of 0 ppm: 100.606816 MHz
 processed size: 65536 complex points
 LB: 0.000 GF: 0.0000
 Hz/cm: 966.184 ppm/cm: 9.60255