

Highly Efficient Triphenyl (3-sulfopropyl) phosphonium functionalized Phosphotungstic acid on Silica as a Solid Acid Catalyst for Selective mono-Allylation of Acetals

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Experimental procedure for the synthesis of acetals using PVP-PWA catalyst

To the solution of aldehydes (1 mmol) in 3 mL of methanol/ethanol, the catalyst PVP-PWA (0.01 mmol) was added and the mixture was stirred for 2 h at room temperature under inert condition. After completion of the reaction monitored by TLC, reaction crude was filtered through sintered glass plug using celite to separate the catalyst and washed two times with 3 mL methanol. Finally methanol was evaporated at reduced pressure using rota vapour to obtain the product with excellent yields. If required column chromatography were done over neutral alumina.

Ammonia-TPD study

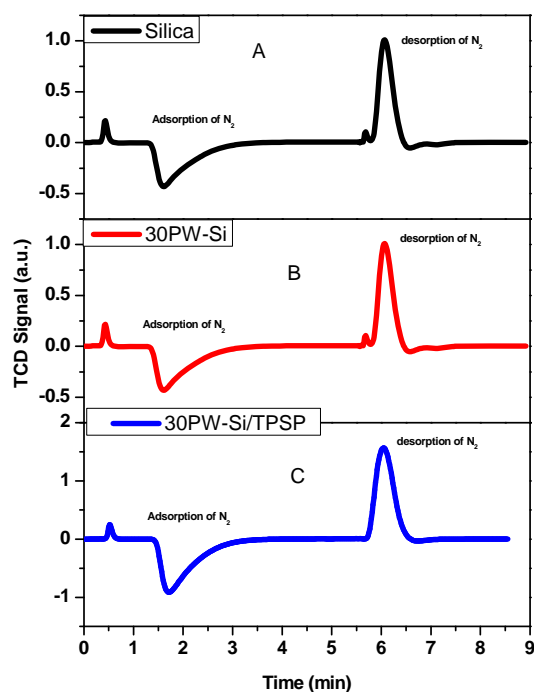
Table S1. Total acidity measurement of catalysts using ammonia-TPD

Sr. No.	Sample Name	Temperature, °C	Total acidity (NH ₃ mmol/gm)
1	Silica	100-200,	0.01191
2	PW	100-200, 550-650	0.3983
3	PW-TPSP	250-550	0.2381
4	30PW-Si/TPSP	250-550	0.176

Order of total acidity:

PW (0.3983 NH₃ mmoles / gm) > PW-TPSP (0.2381 NH₃ mmoles / gm) > 30PW-Si/TPSP (0.176 NH₃ mmoles / gm) > Silica (0.01191 NH₃ mmoles / gm)

Figure S1. BET Surface area of Silica (A), 30PW-Si (B) and 30PW-Si/TPSP (C)



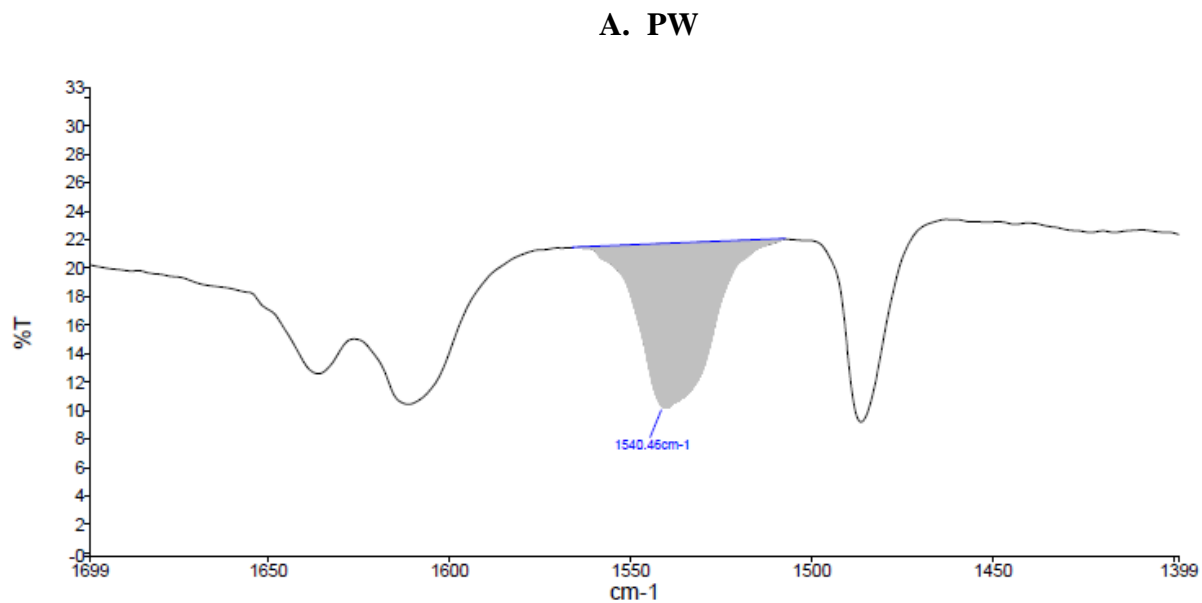
Surface Area: Silica; 232.57 m²/g, 30PW-Si; 116.70 m²/g and 30PW-Si/TPSP; 110.69 m²/g.

Table S2. Comparison of Brønsted acidity under Pyridine-FTIR study

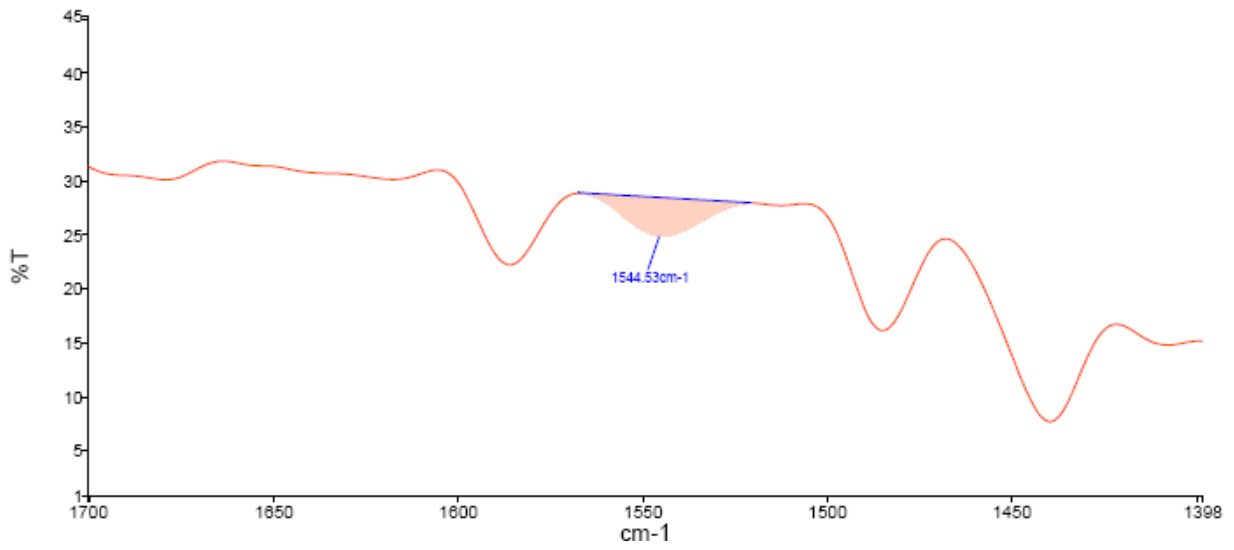
Sr. No.	Sample Name	Area, (%T)
1	PW	-257.1
2	PW-TPSP	-79.04
3	30PW-Si/TPSP	-71.58

Order of Brønsted acidity: PW >PW-TPSP>30PW-Si/TPSP

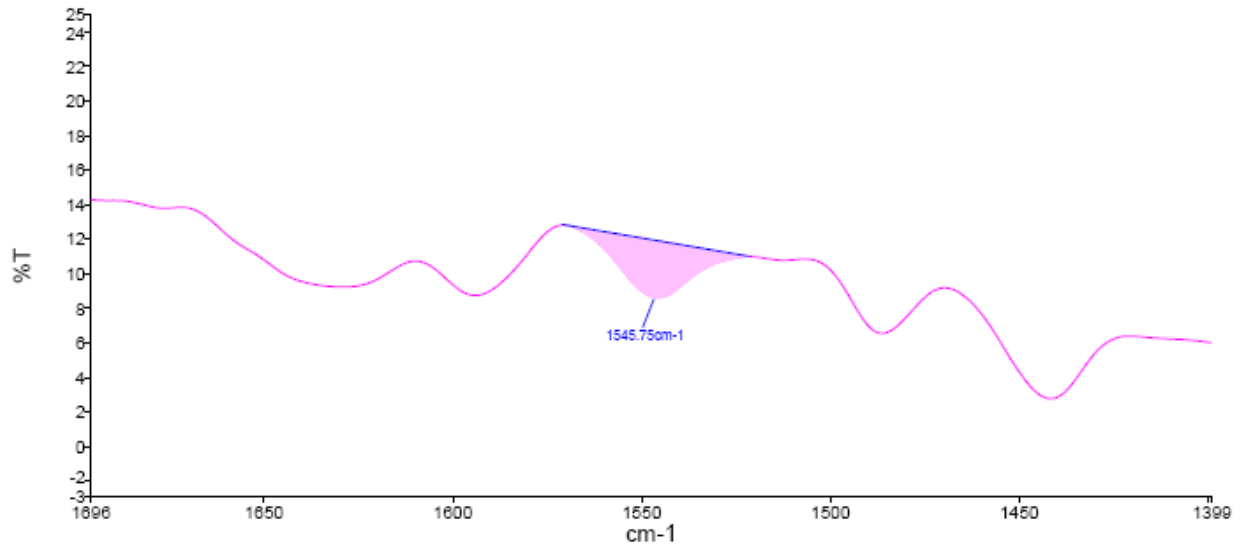
Figure S2. Area under curve by Pyridine-FTIR



B. PW-TPSP



C. 30PW-Si/TPSP



Energy-dispersive X-ray spectroscopy (EDAX) compositional study

Fig. S3. 30PW-Si/TPSP fresh

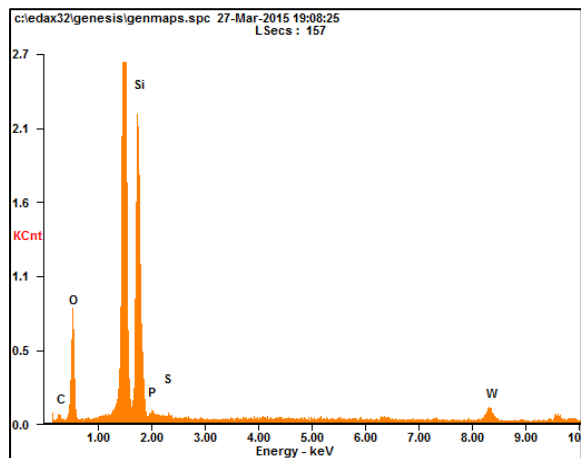


Table S3. 30PW-Si/TPSP fresh

<i>Element</i>	<i>Wt%</i>	<i>At%</i>
<i>CK</i>	16.07	25.21
<i>OK</i>	46.83	55.15
<i>SiK</i>	26.46	17.75
<i>PK</i>	00.97	00.59
<i>SK</i>	00.61	00.36
<i>WL</i>	09.05	00.93
<i>Matrix</i>	Correction	ZAF

Fig. S4. 30PW-Si/TPSP recovered

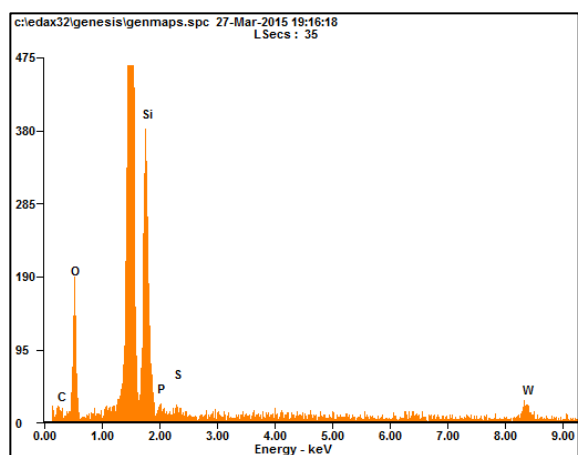
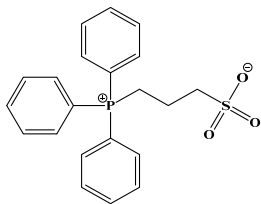


Table S4. 30PW-Si/TPSP recovered

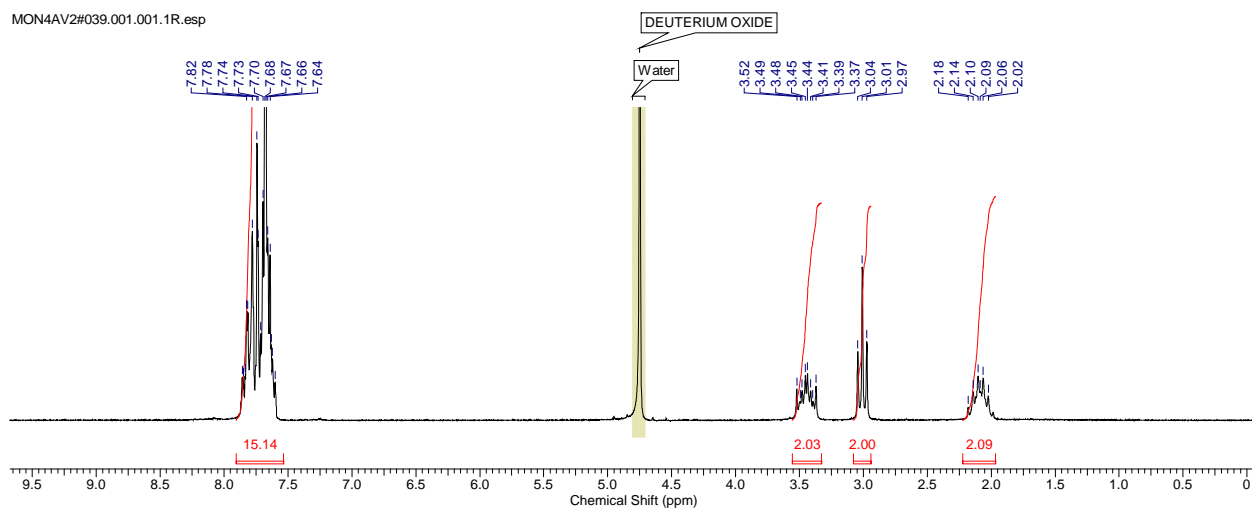
<i>Element</i>	<i>Wt%</i>	<i>At%</i>
<i>CK</i>	19.99	30.18
<i>OK</i>	47.60	53.94
<i>SiK</i>	21.69	14.00
<i>PK</i>	00.91	00.53
<i>SK</i>	00.80	00.45
<i>WL</i>	09.00	00.89
<i>Matrix</i>	Correction	ZAF

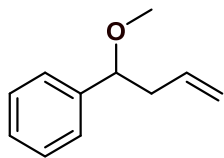
$^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ of products



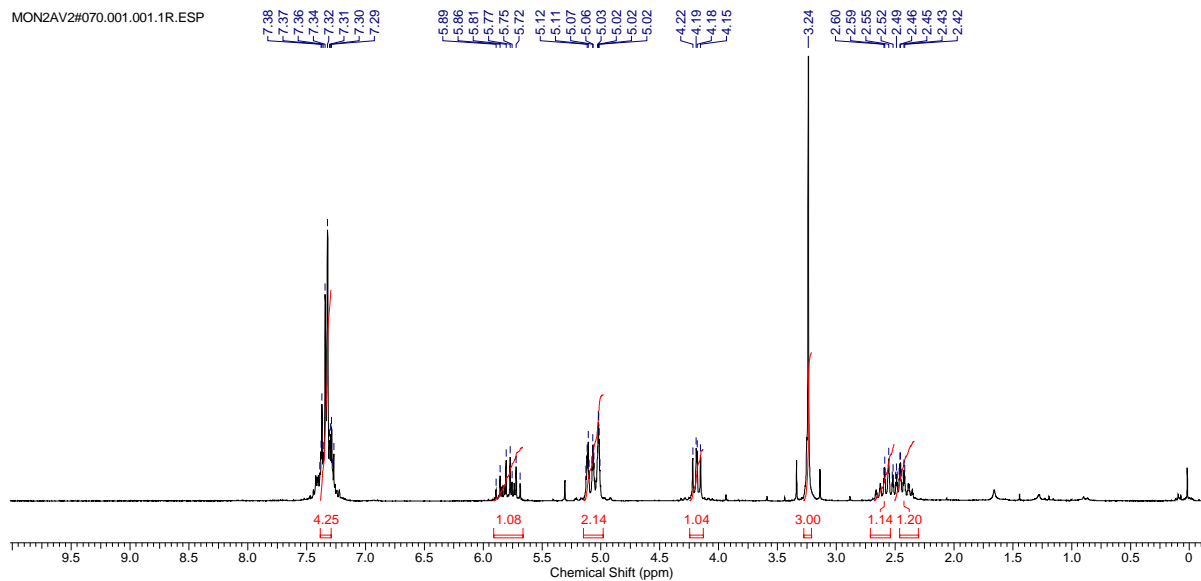
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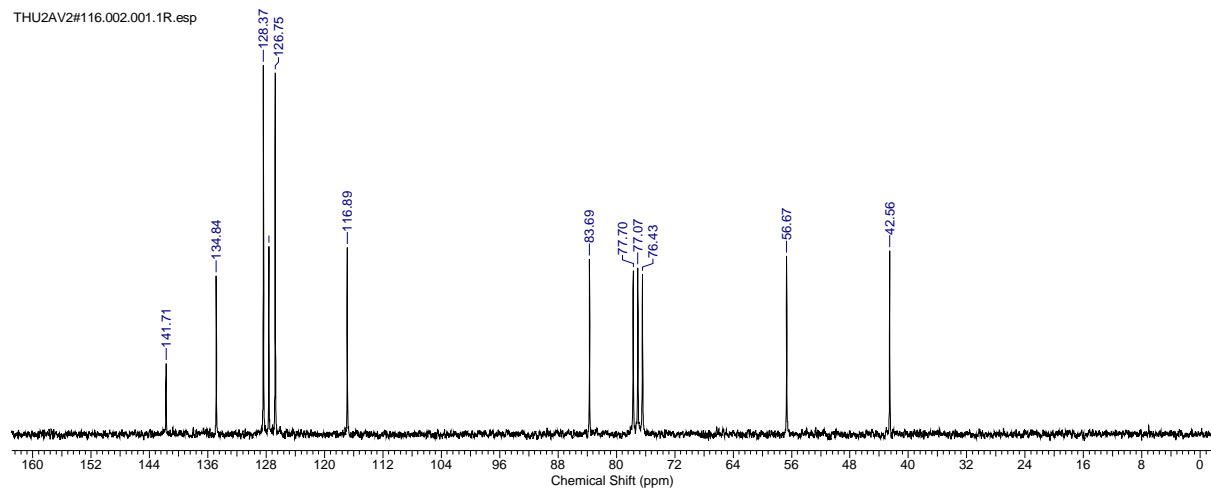




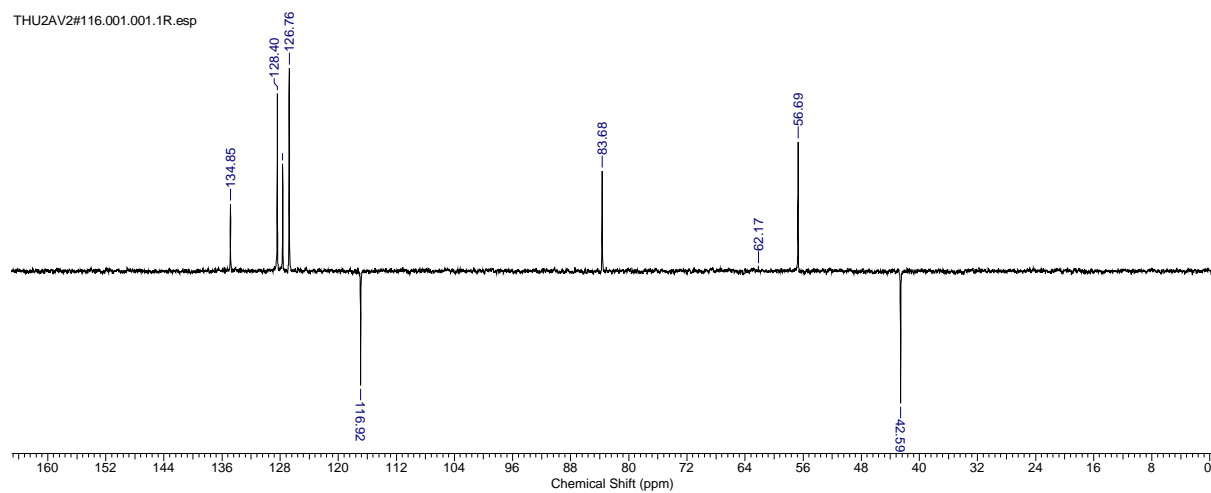
(1-methoxybut-3-en-1-yl) benzene (Table 2, entry 1)

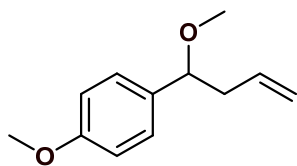


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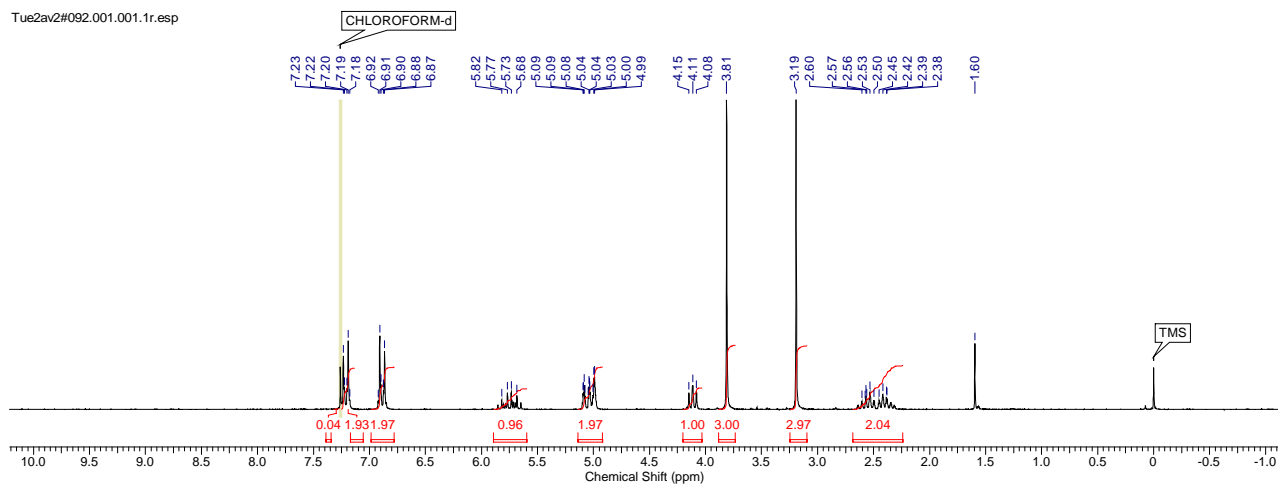


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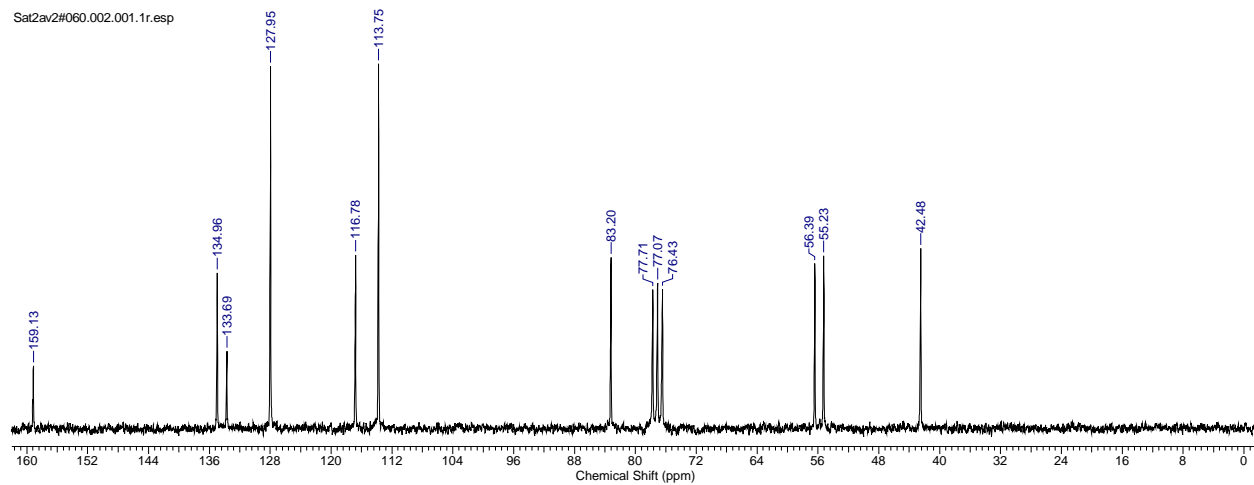




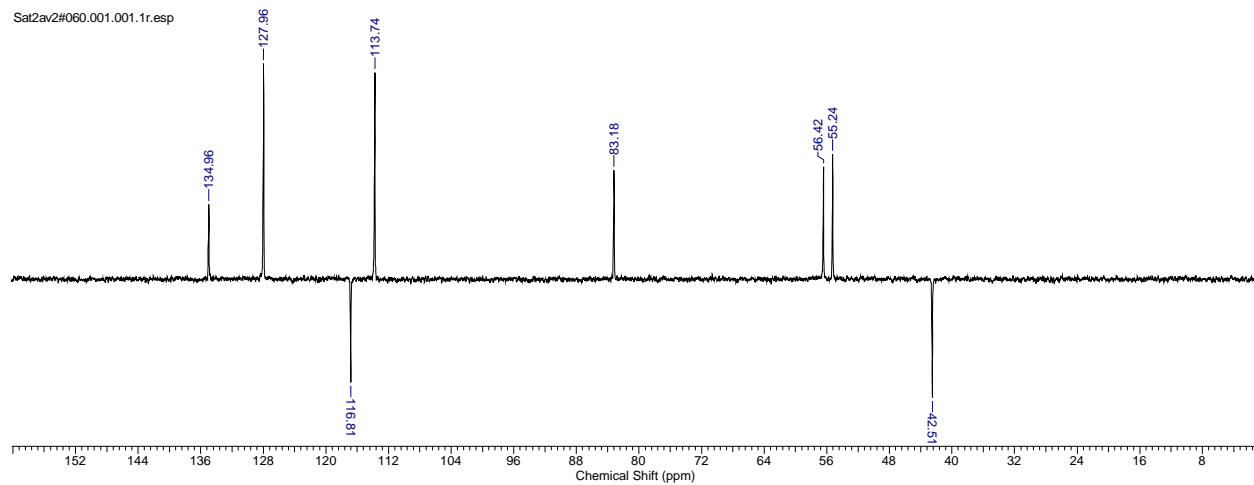
1-methoxy-4-(1-methoxybut-3-en-1-yl) benzene (Table 2, entry 2)

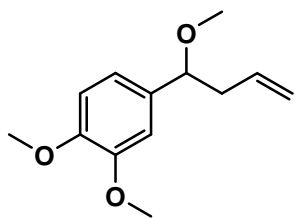


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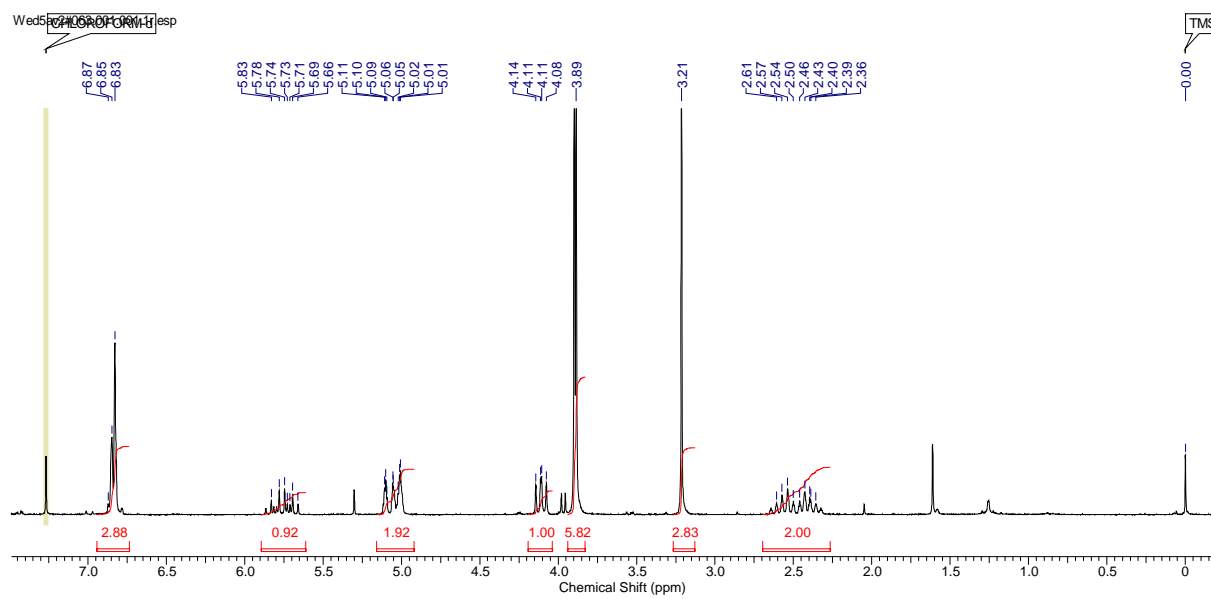


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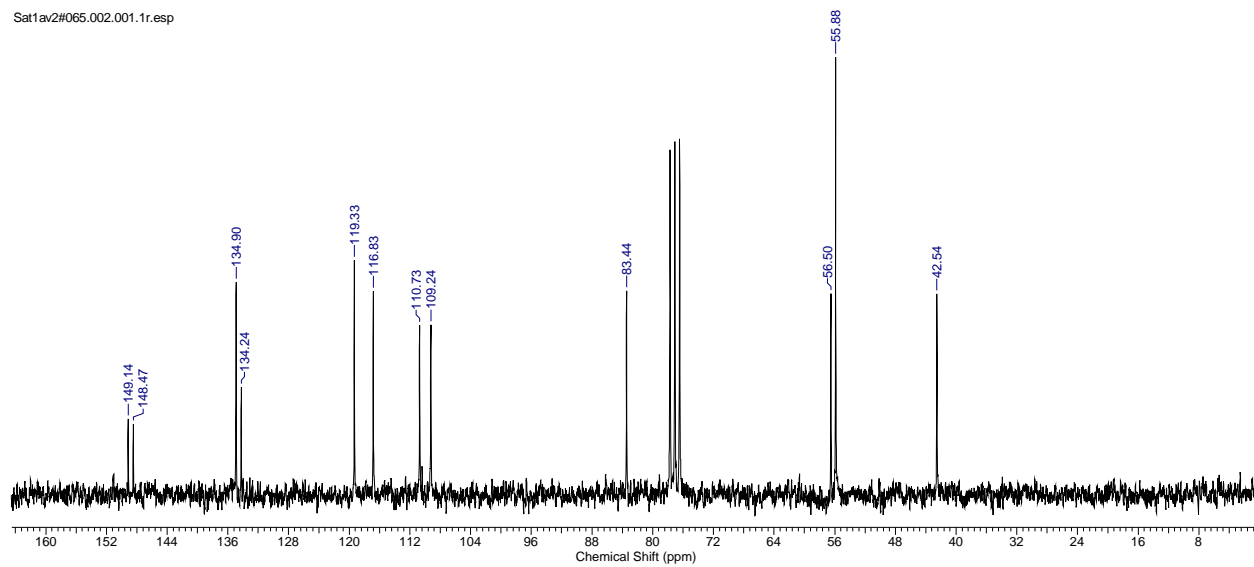




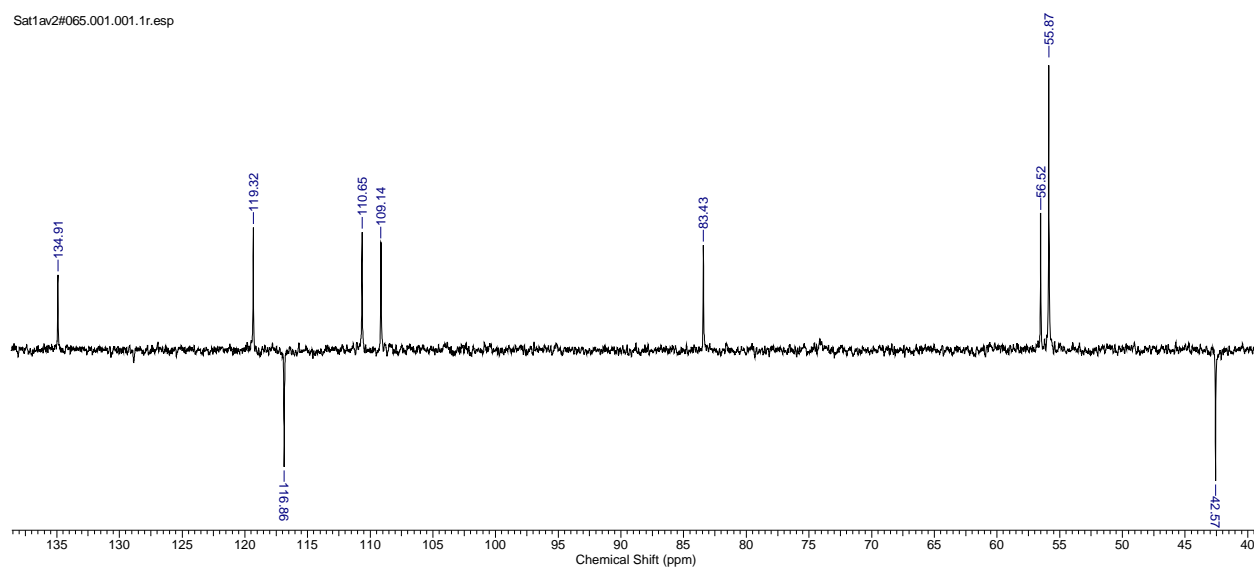
1, 2-dimethoxy-4-(1-methoxybut-3-en-1-yl) benzene (Table 2, entry 3)

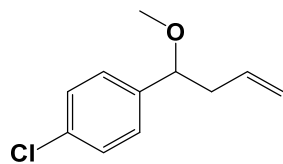


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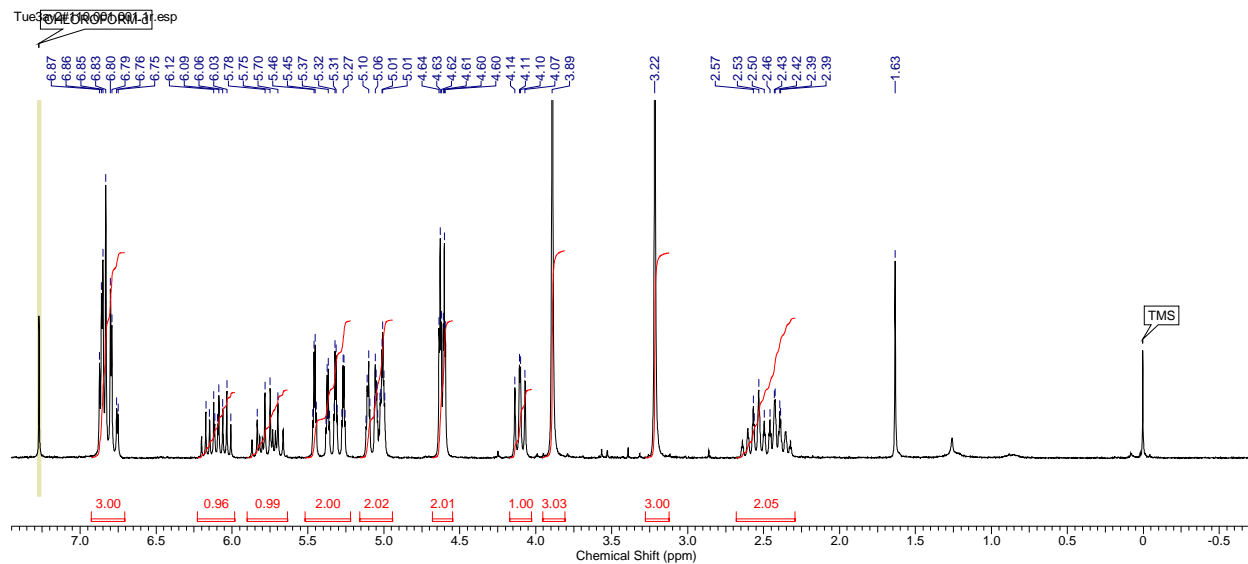


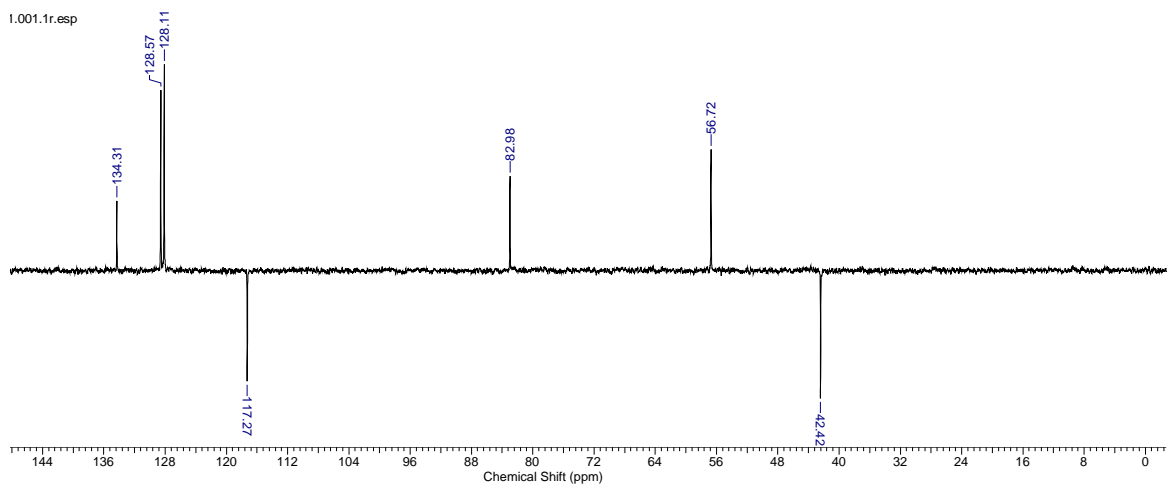
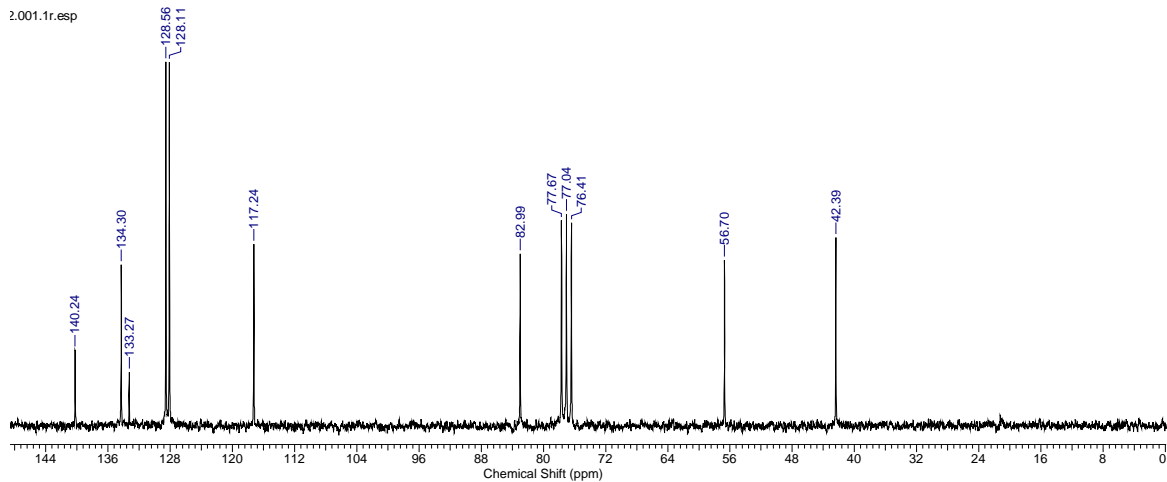
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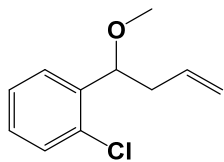




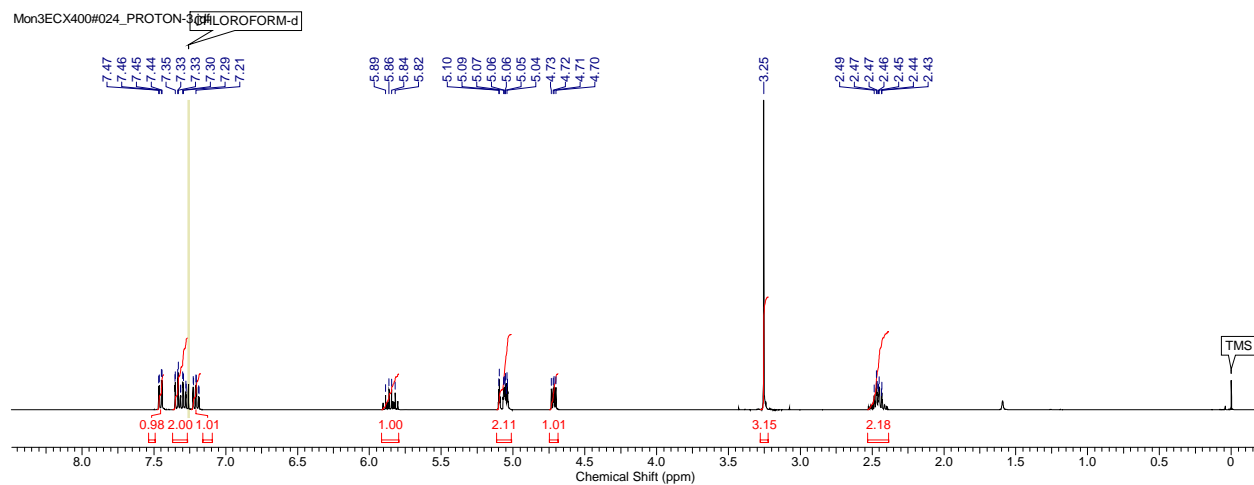
1-chloro-4-(1-methoxybut-3-en-1-yl) benzene (Table 2, entry 4)



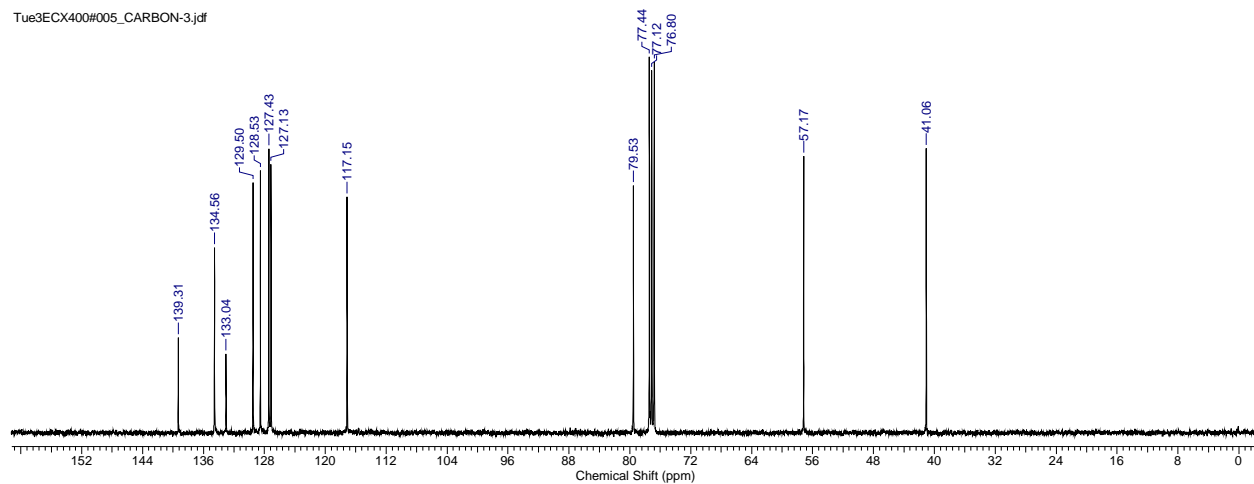




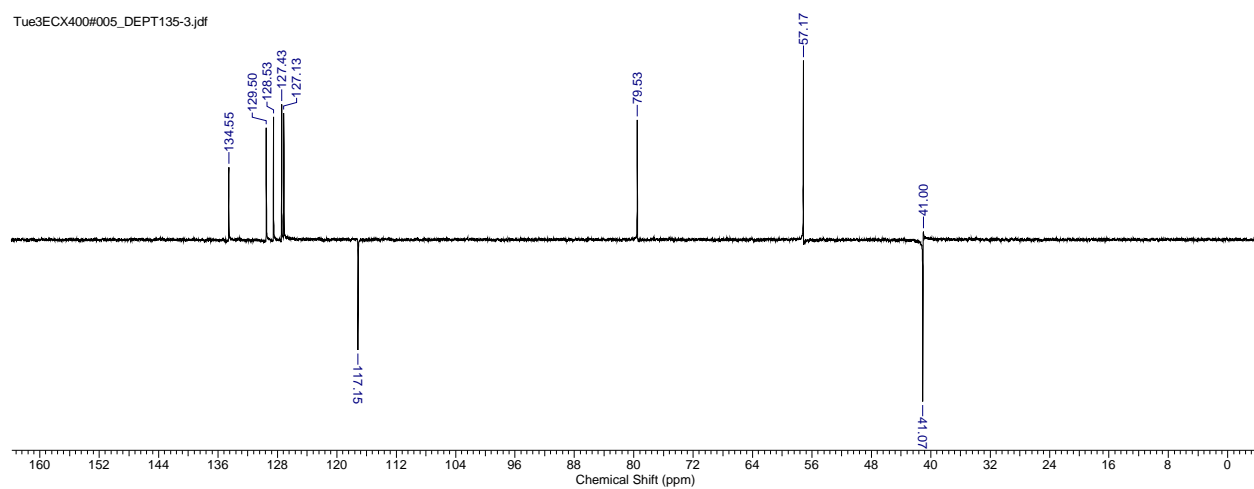
1-chloro-2-(1-methoxybut-3-en-1-yl) benzene (Table 2, entry 5)

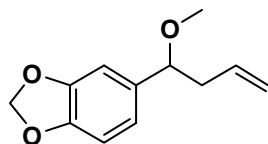


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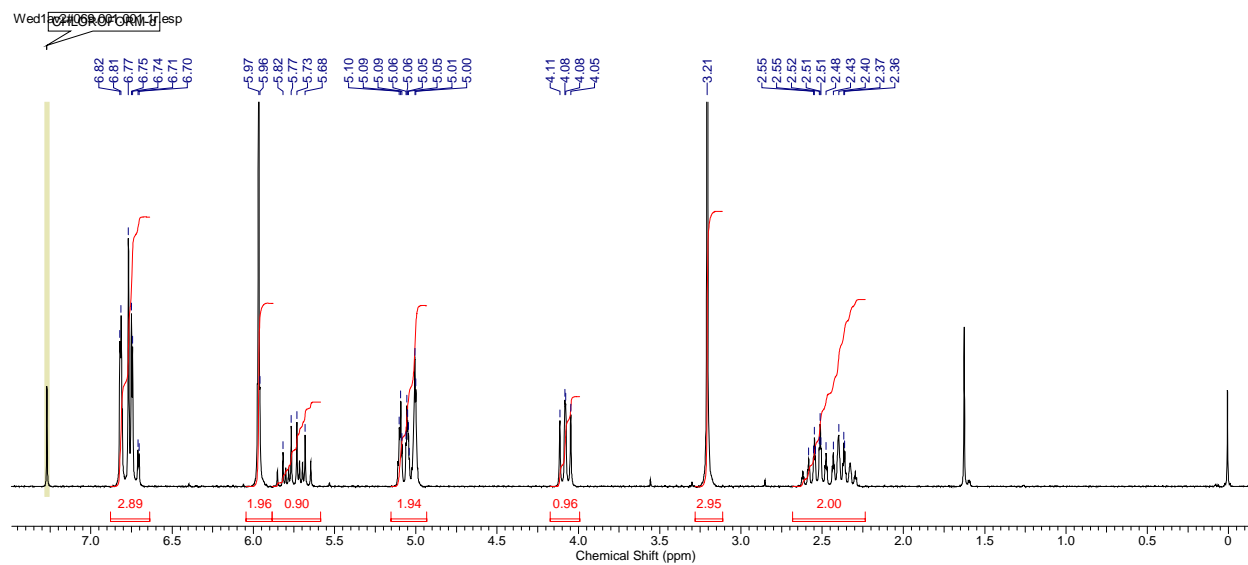


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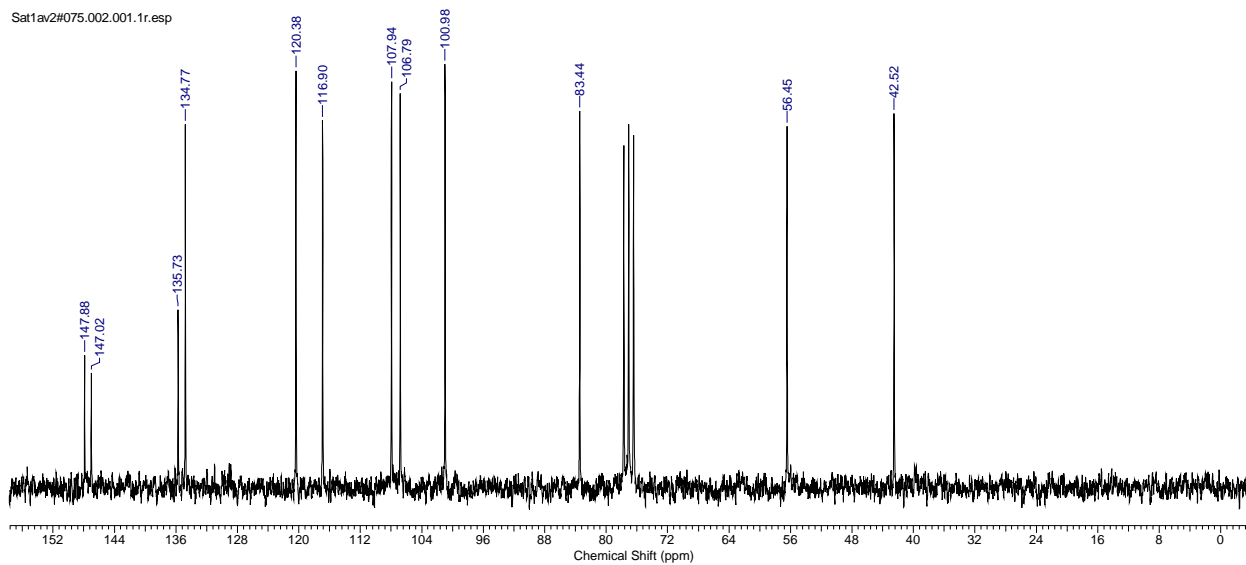




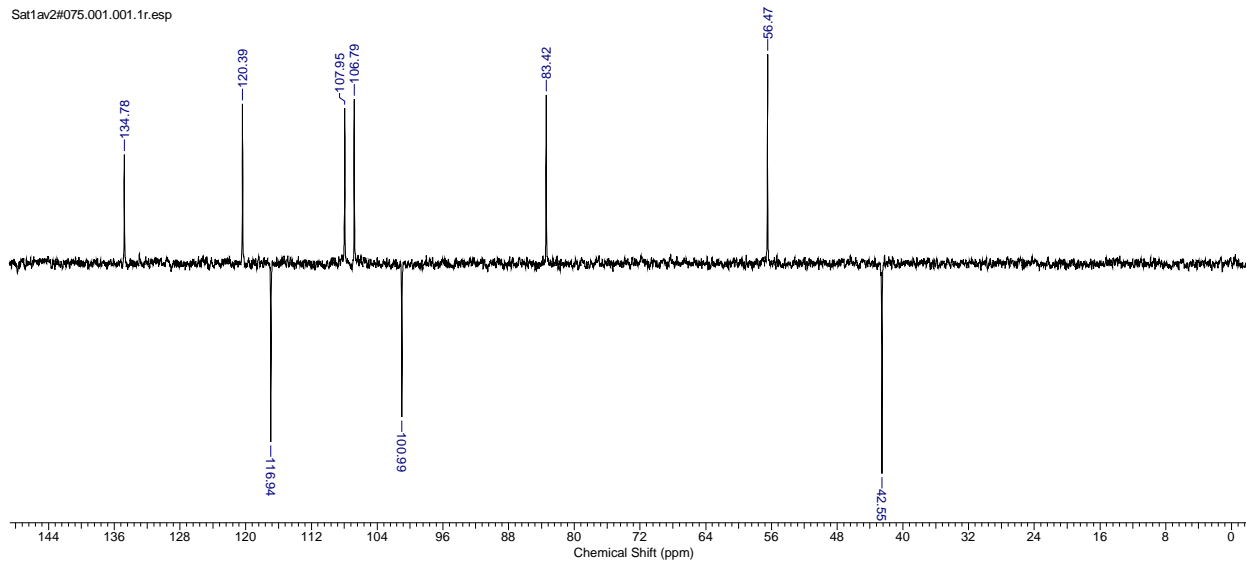
5-(1-methoxybut-3-en-1-yl) benzo [d] [1, 3] dioxole (Table 2, entry 6)

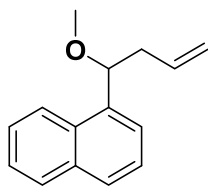


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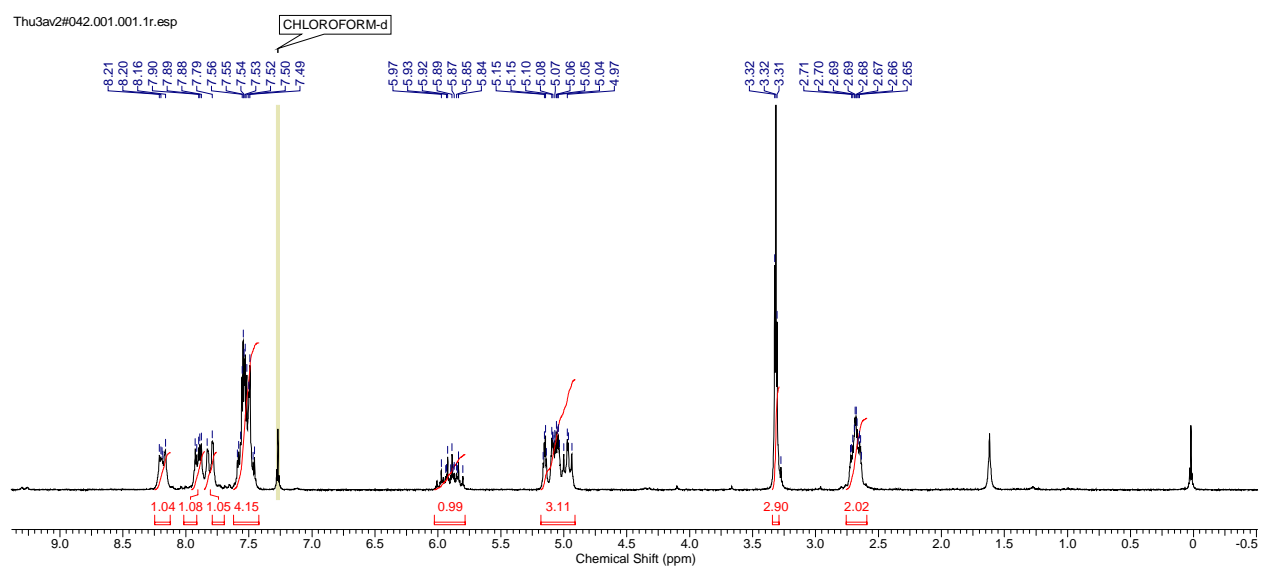


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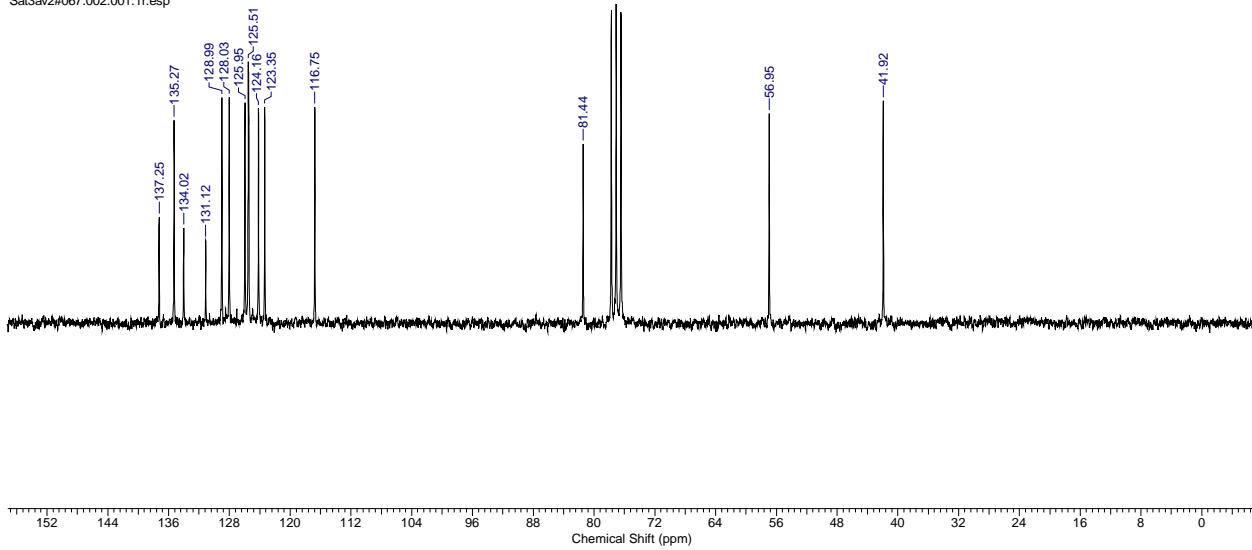




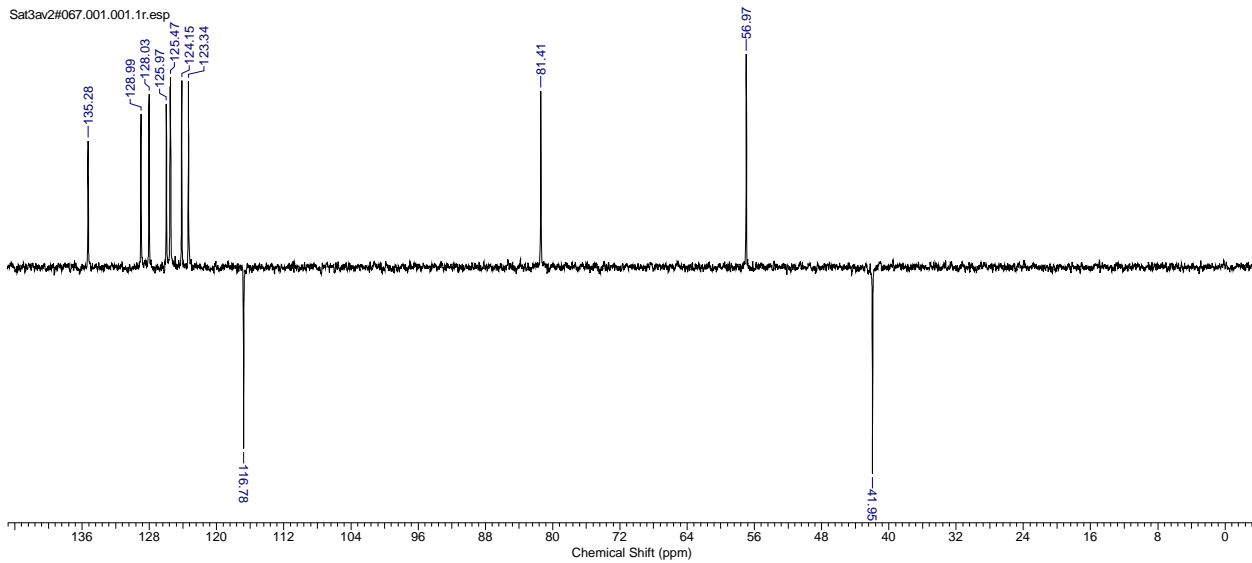
1-(1-methoxybut-3-en-1-yl) naphthalene (Table 2, entry 7)

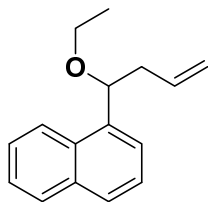


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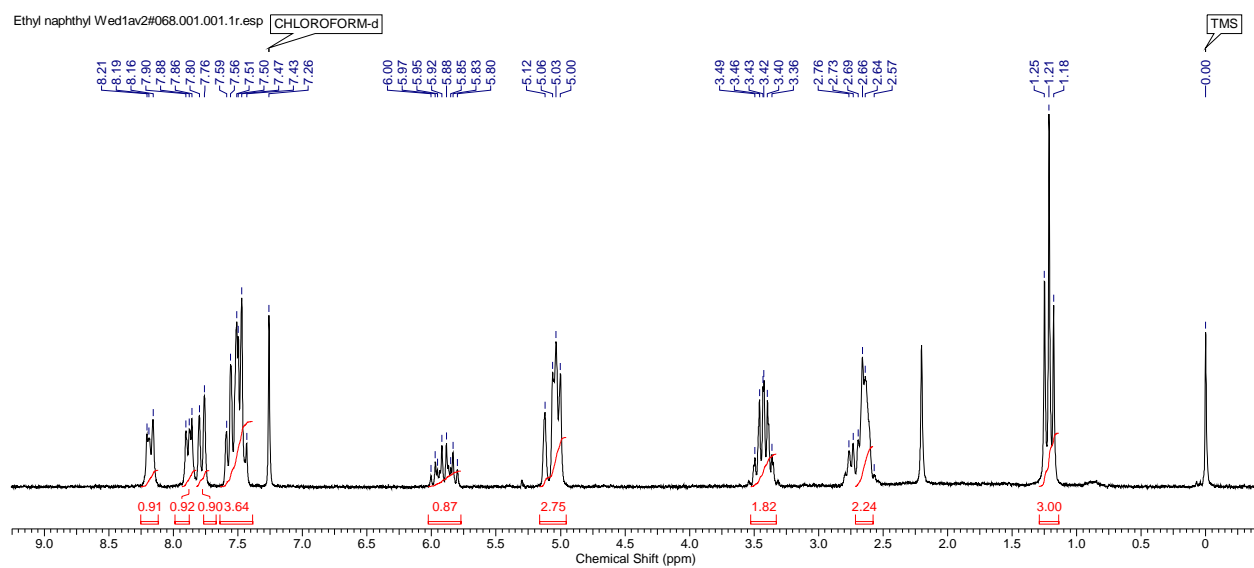


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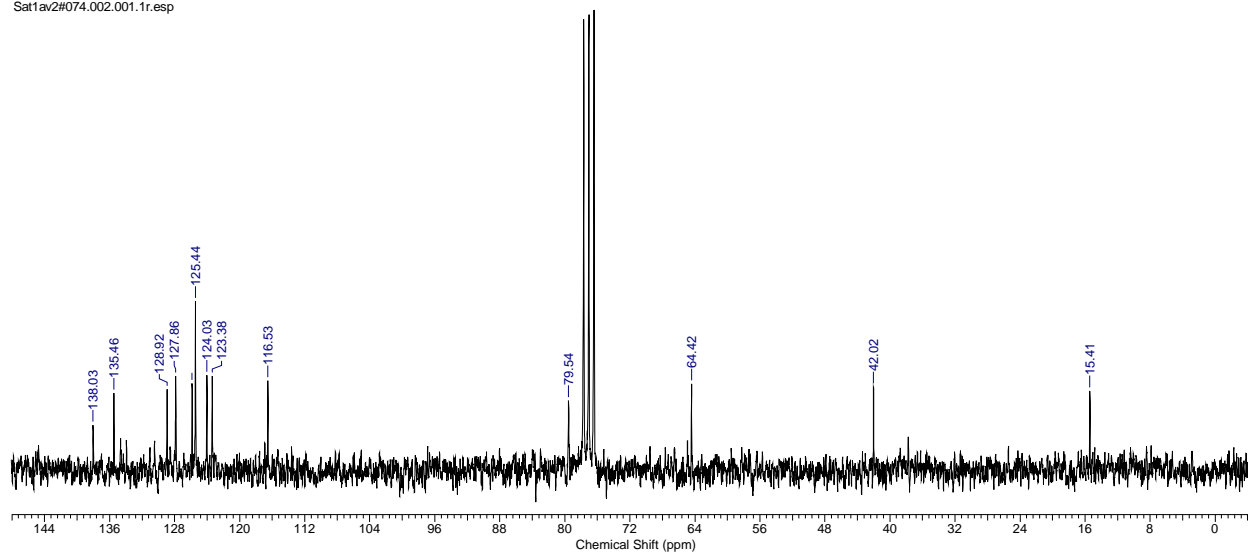




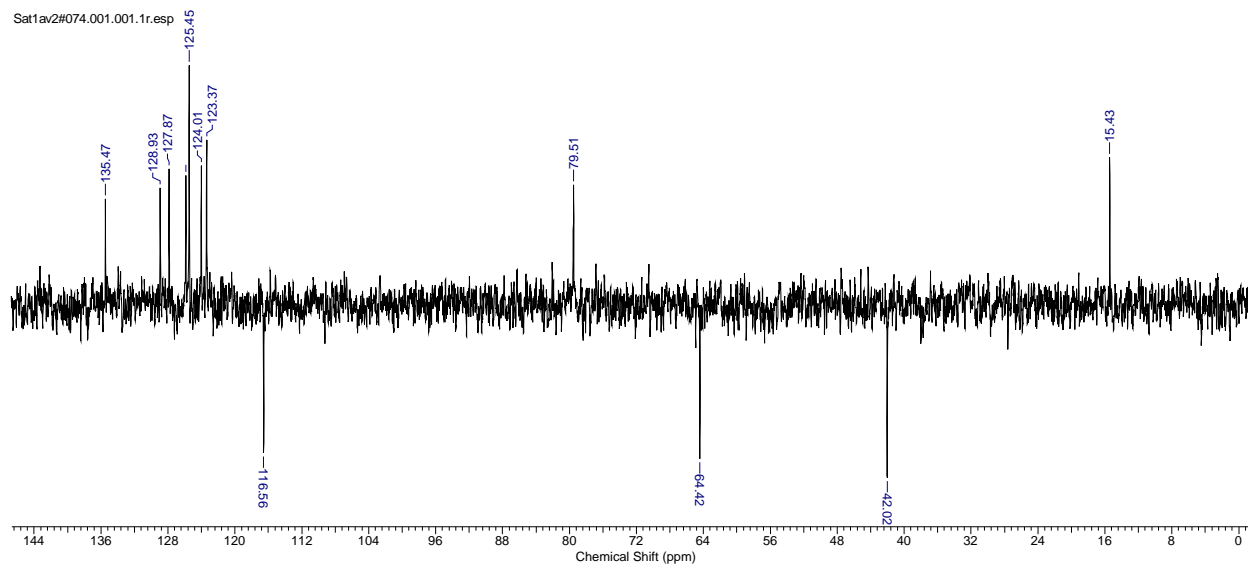
1-(1-ethoxybut-3-en-1-yl) naphthalene (Table 2, entry 8)

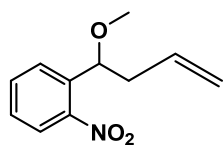


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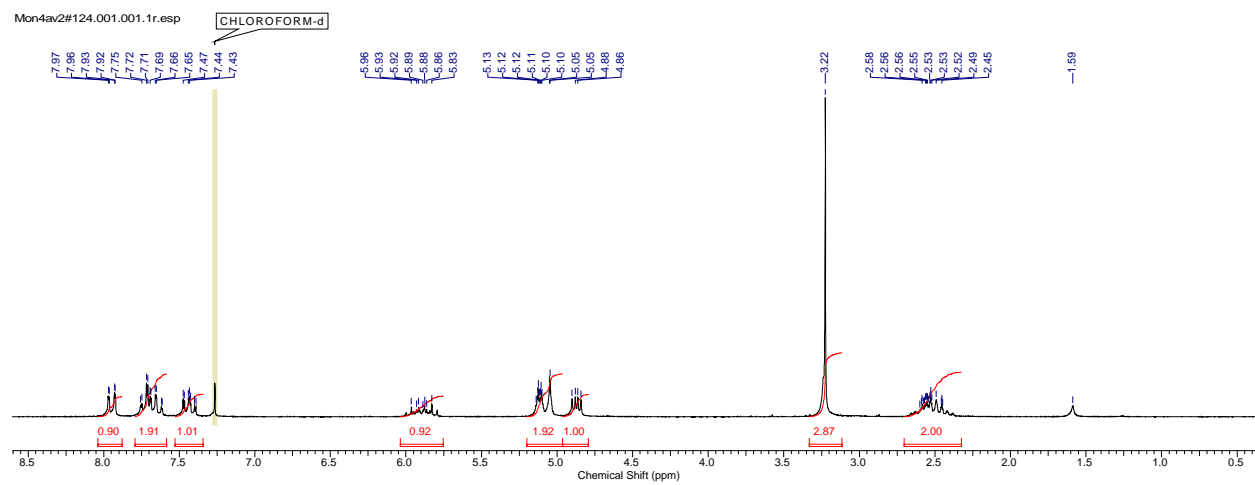


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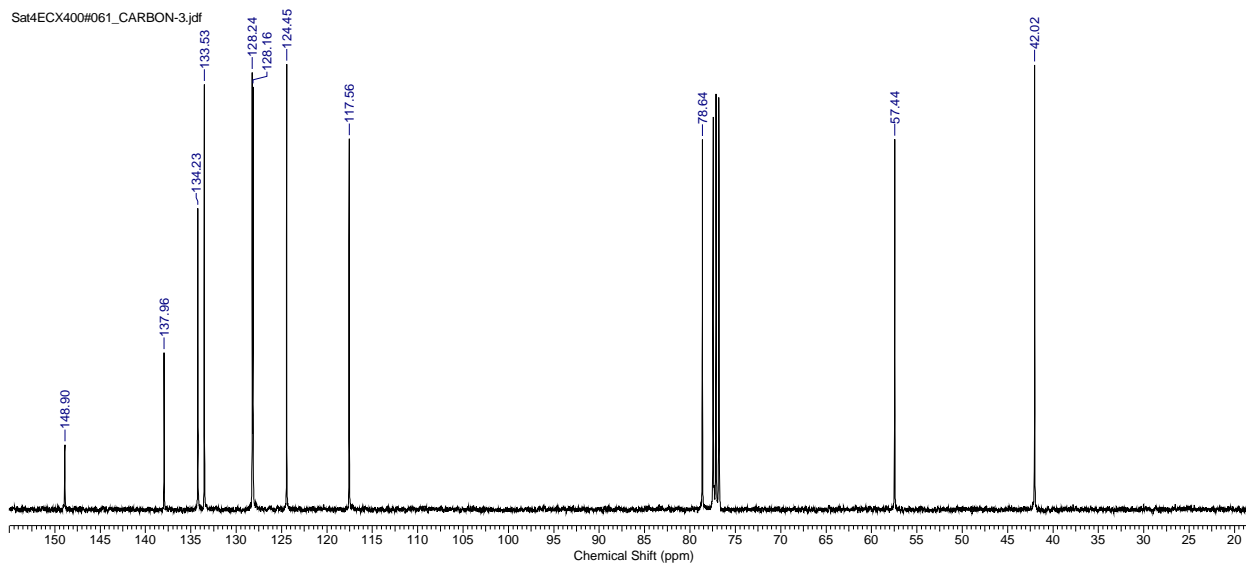




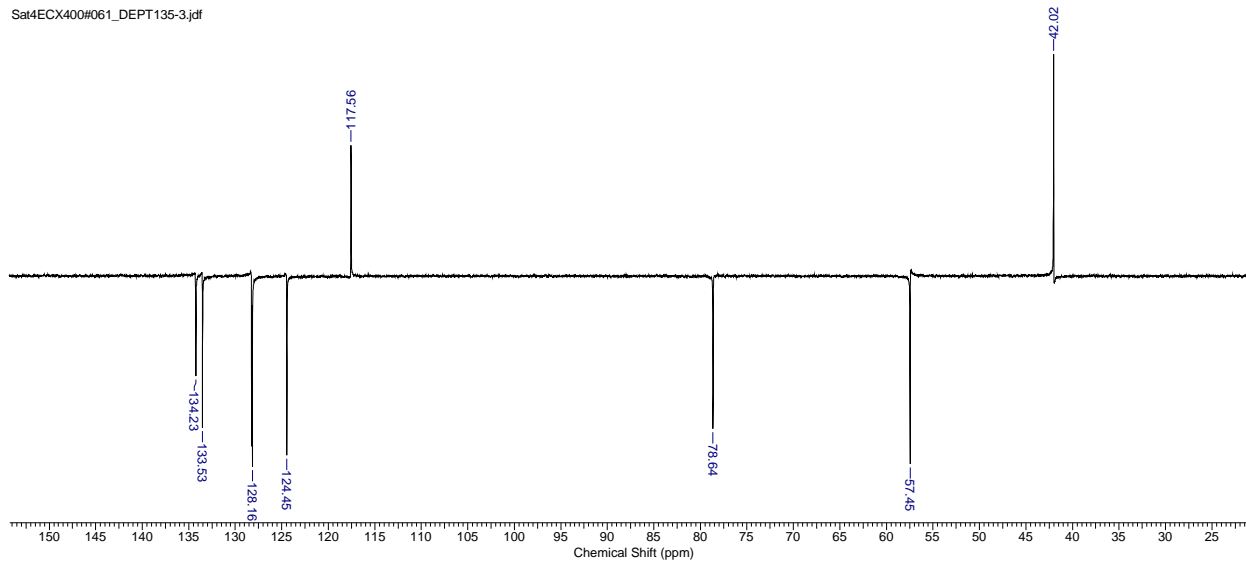
1-(1-methoxybut-3-en-1-yl)-2-nitrobenzene (Table 2, entry 9)

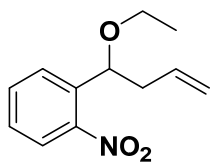


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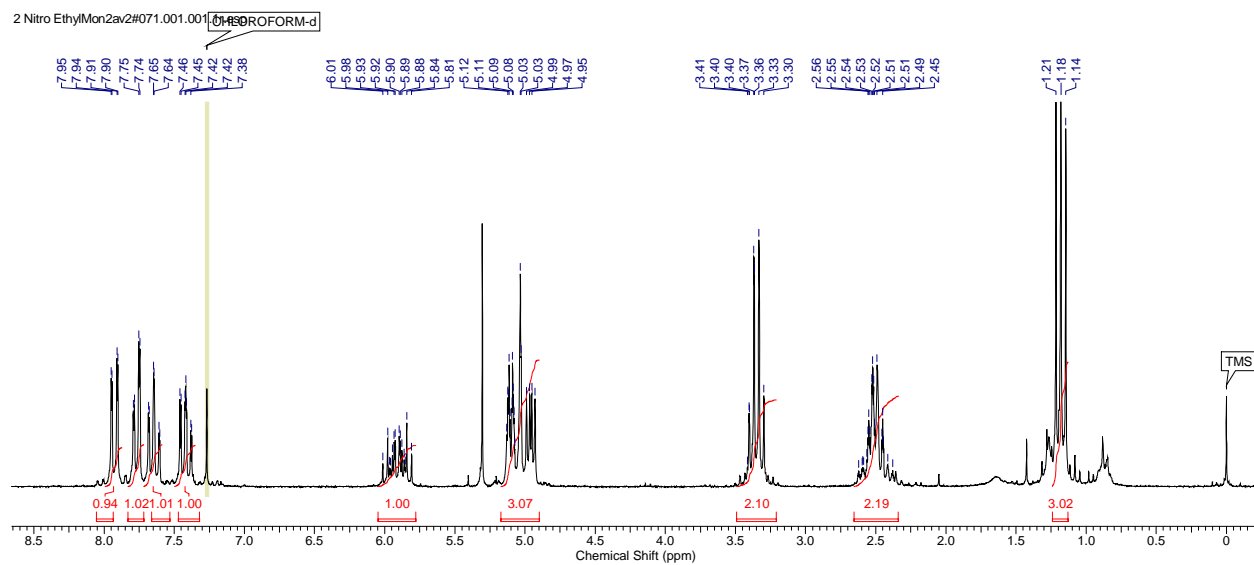


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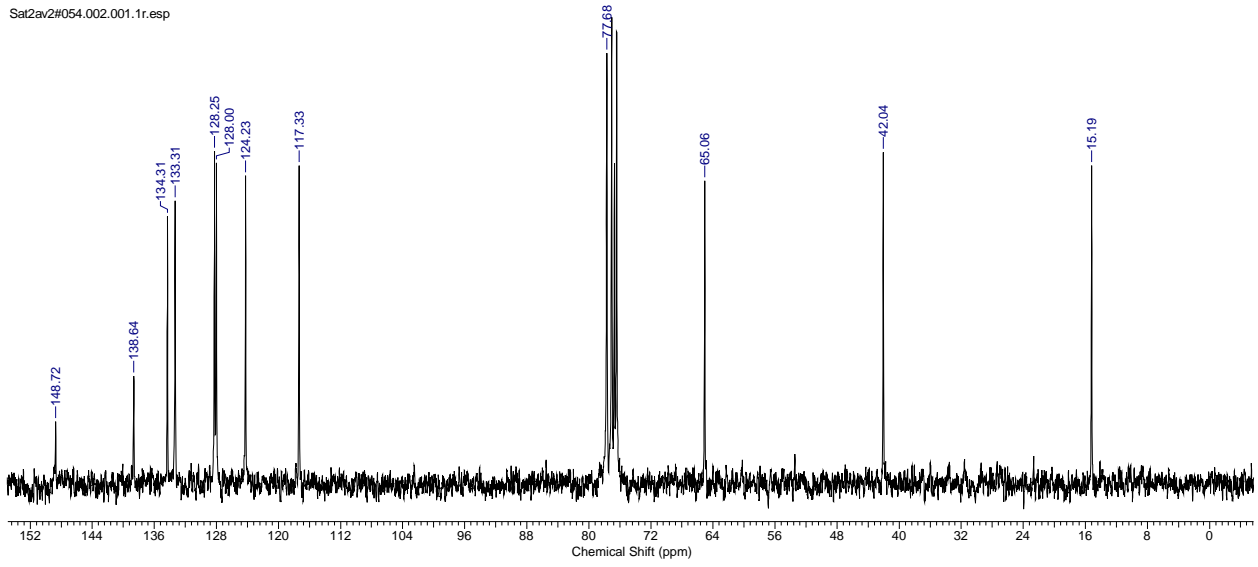




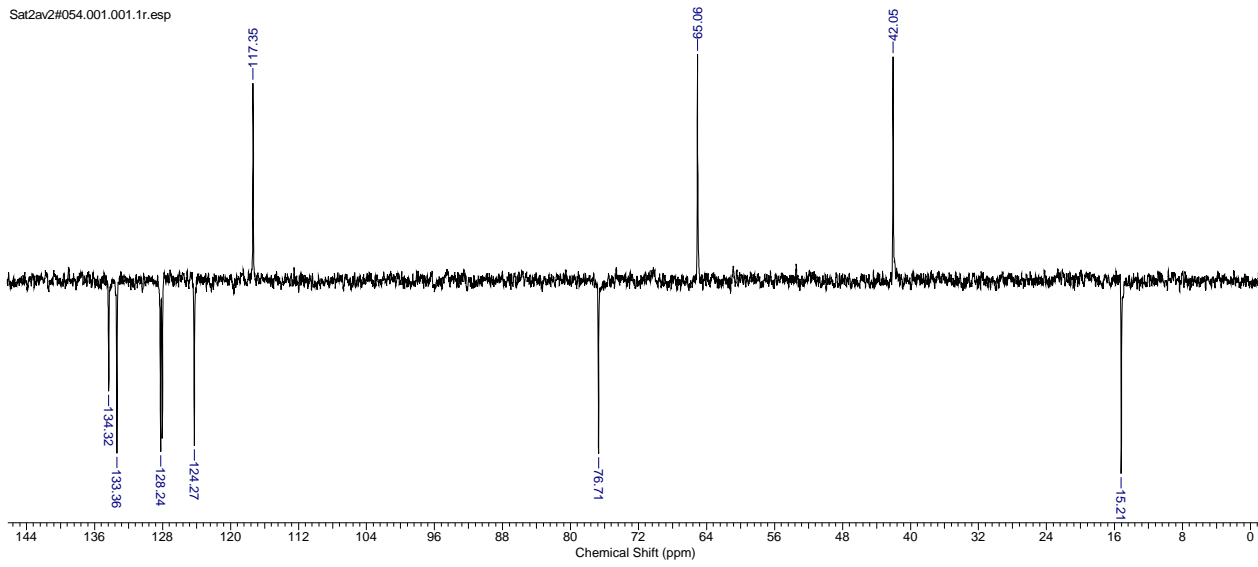
1-(1-ethoxybut-3-en-1-yl)-2-nitrobenzene (Table 2, entry 10)

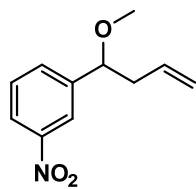


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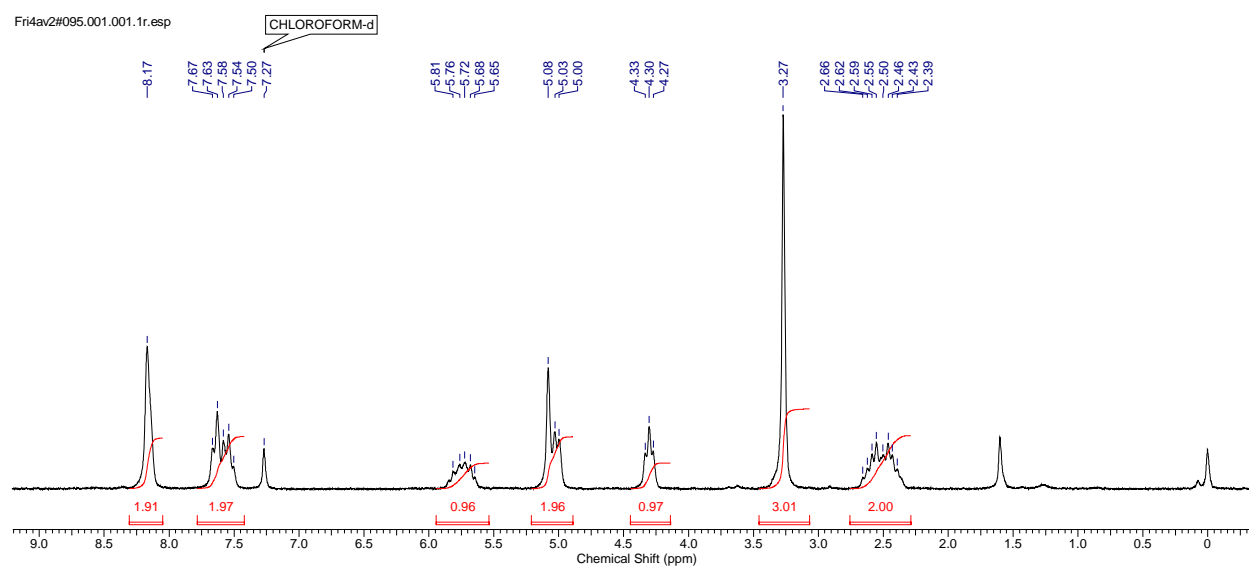


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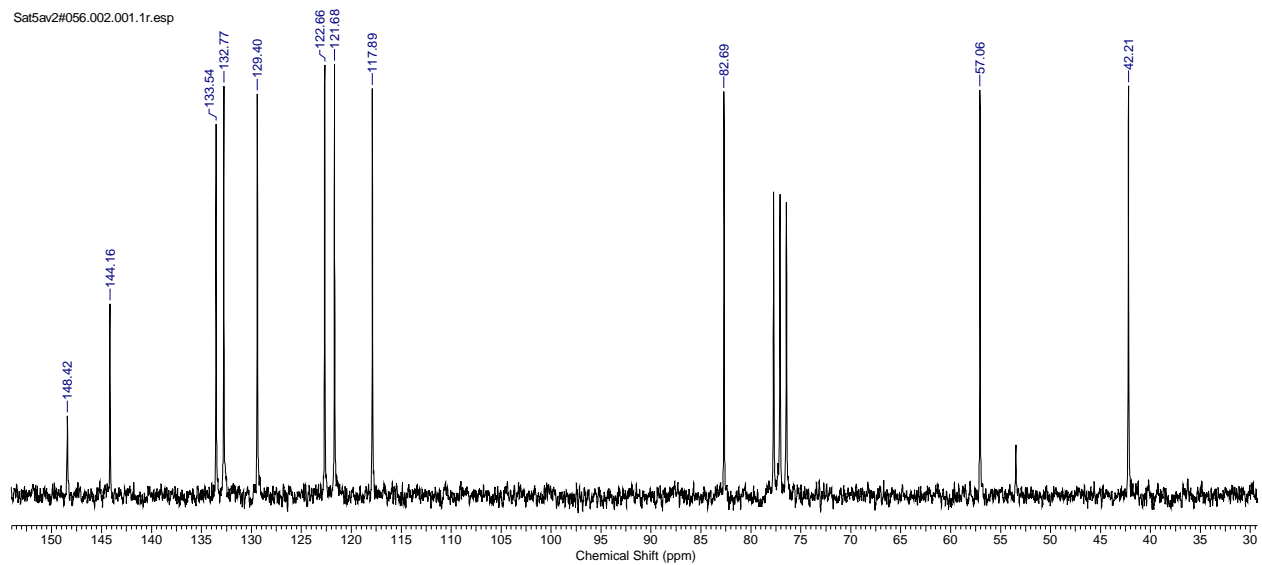




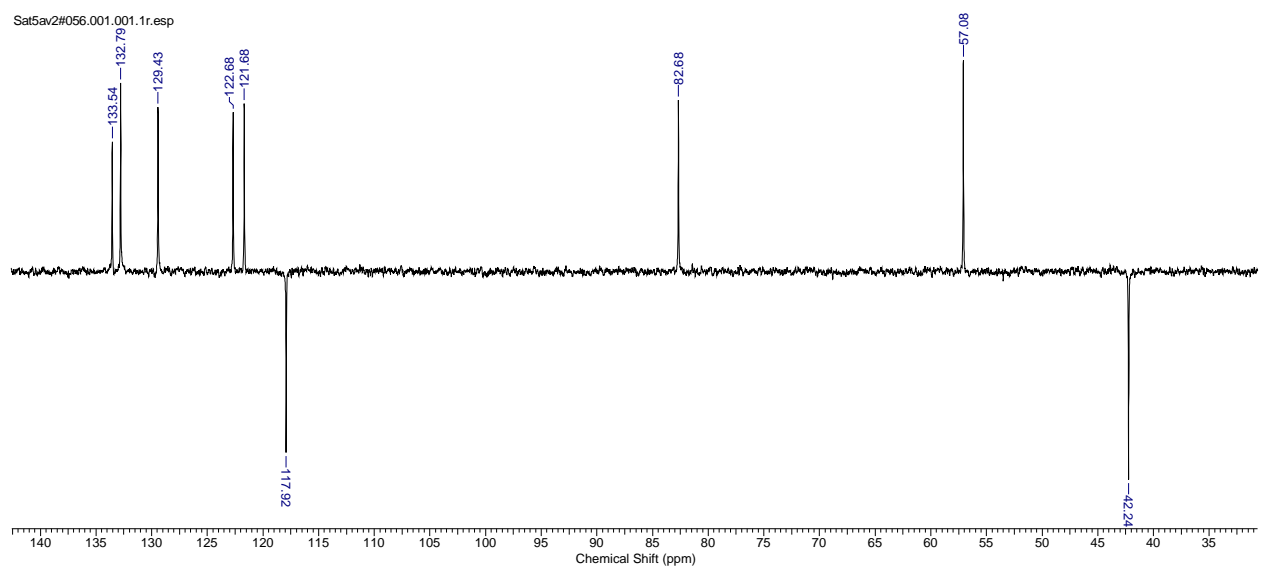
1-(1-methoxybut-3-en-1-yl)-3-nitrobenzene (Table 2, entry 11)

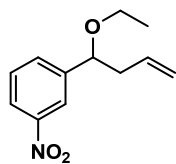


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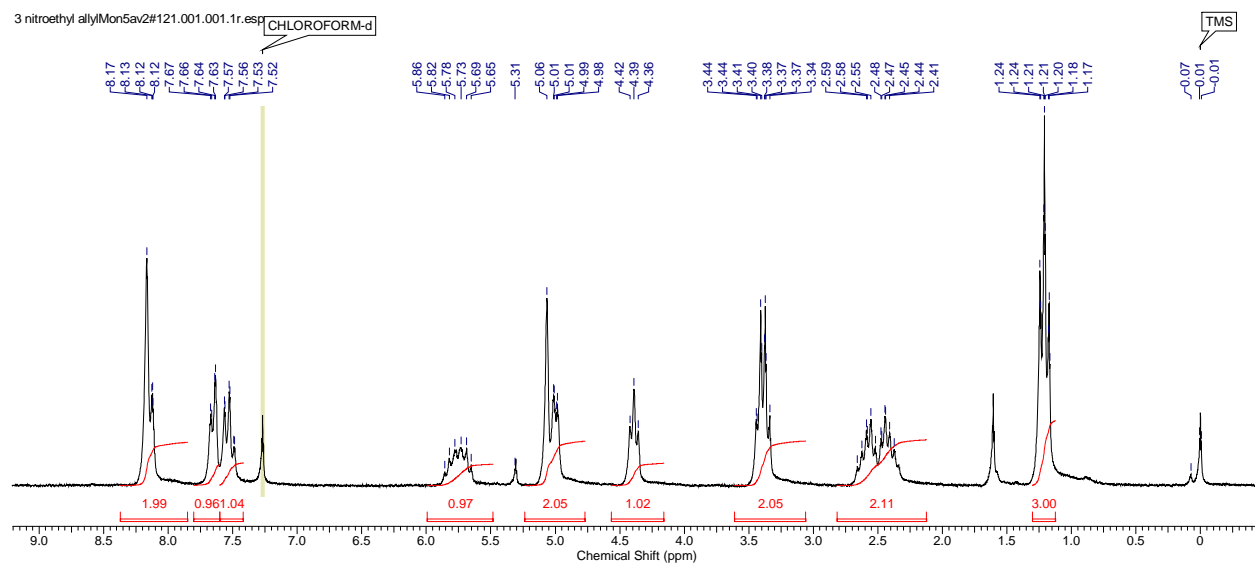


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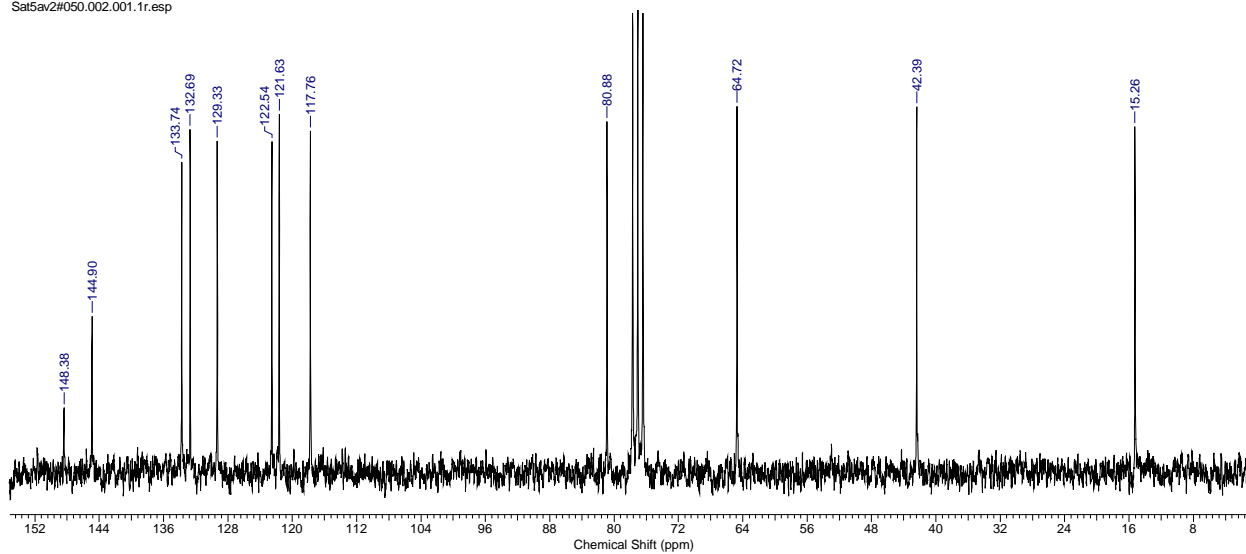




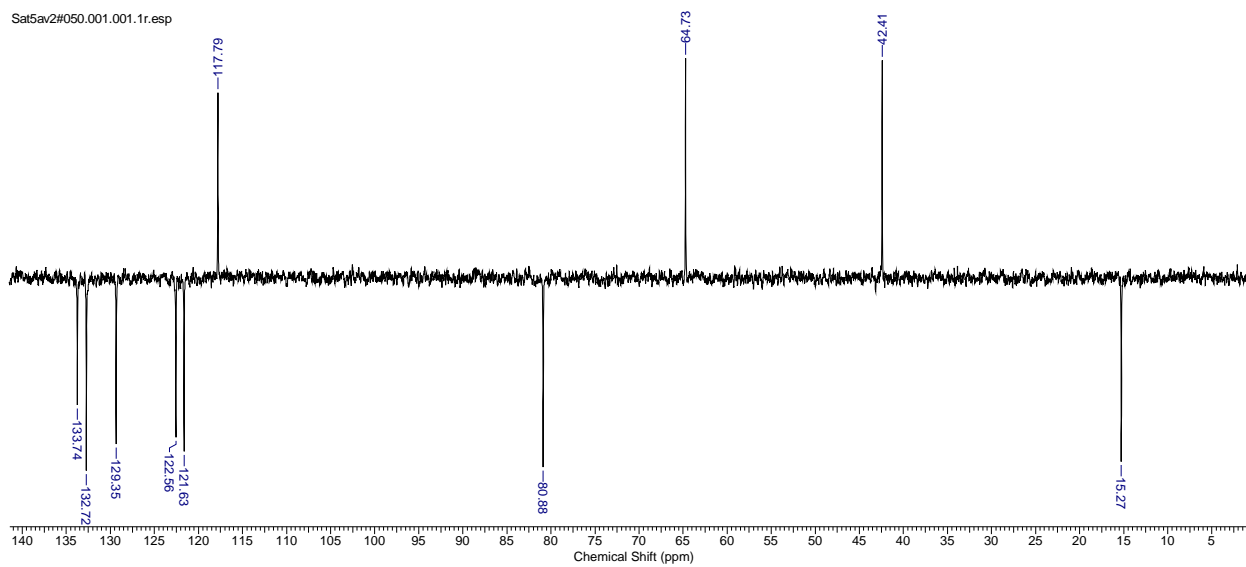
1-(1-ethoxybut-3-en-1-yl)-3-nitrobenzene (Table 2, entry 12)

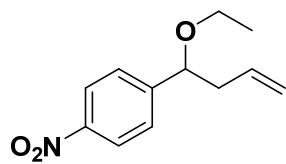


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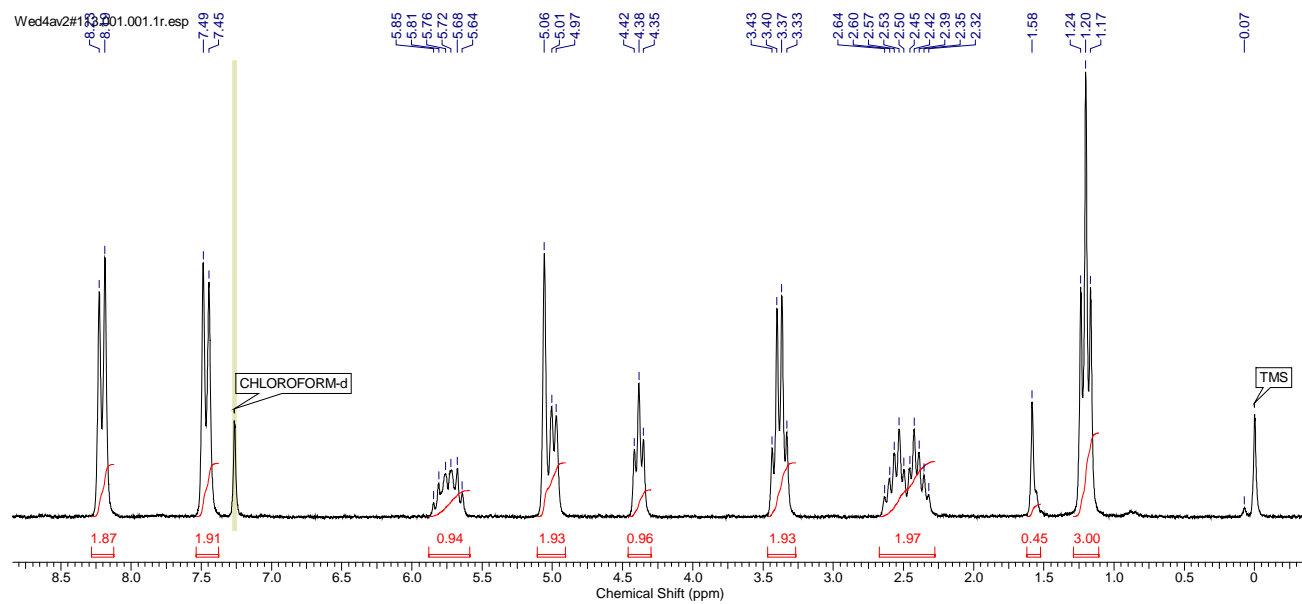


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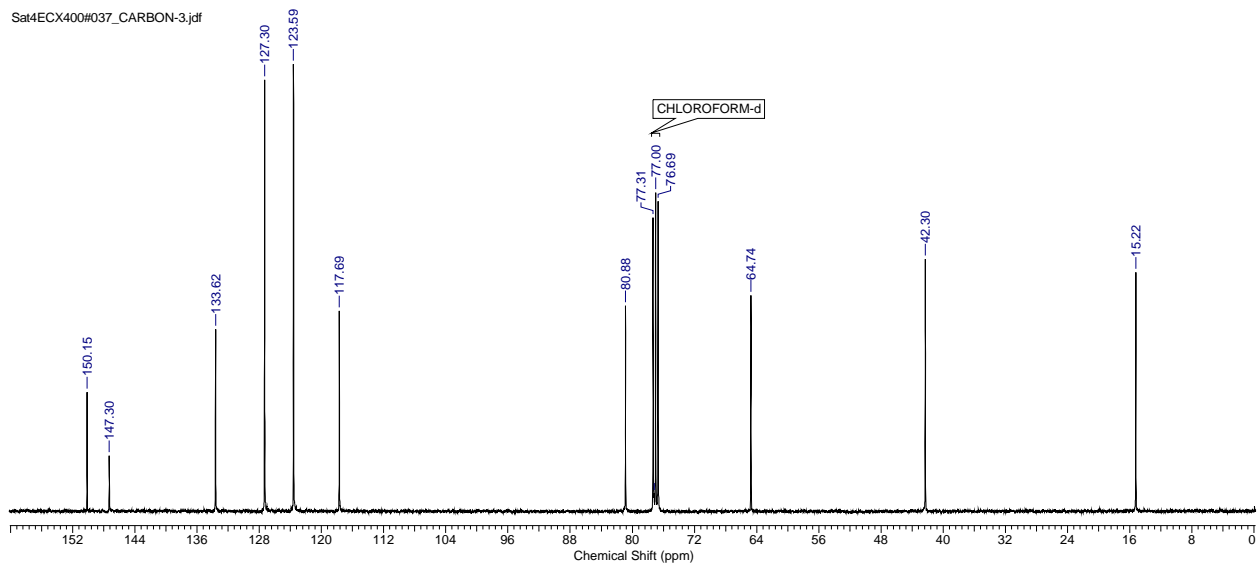




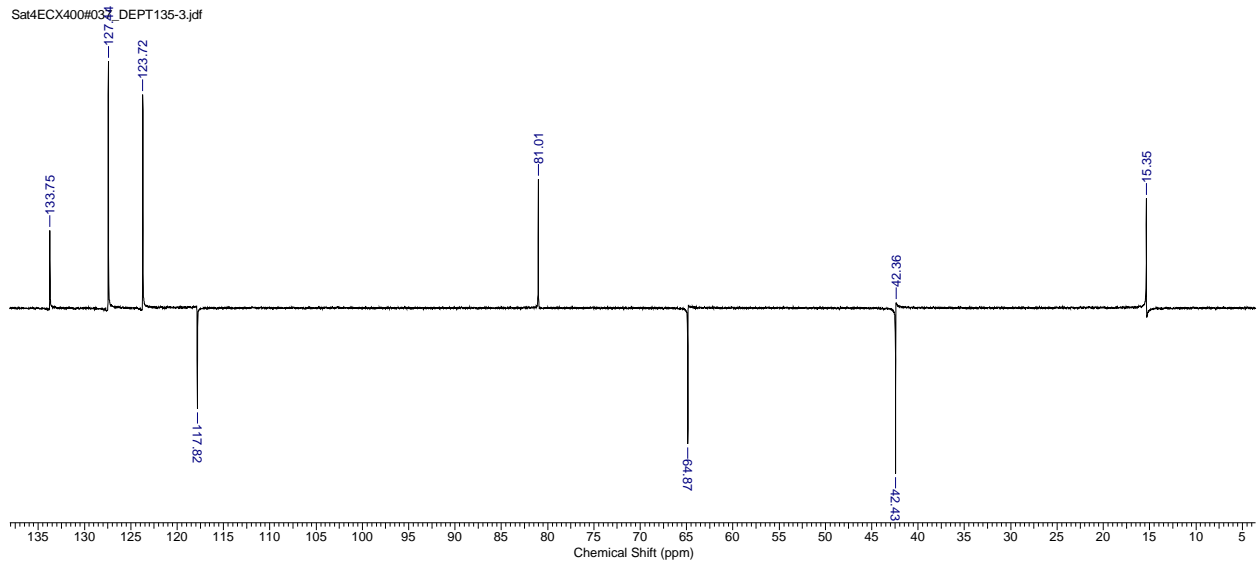
1-(1-ethoxybut-3-en-1-yl)-4-nitrobenzene (Table 2, entry 13)

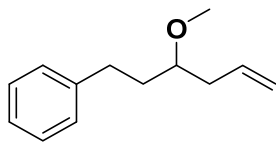


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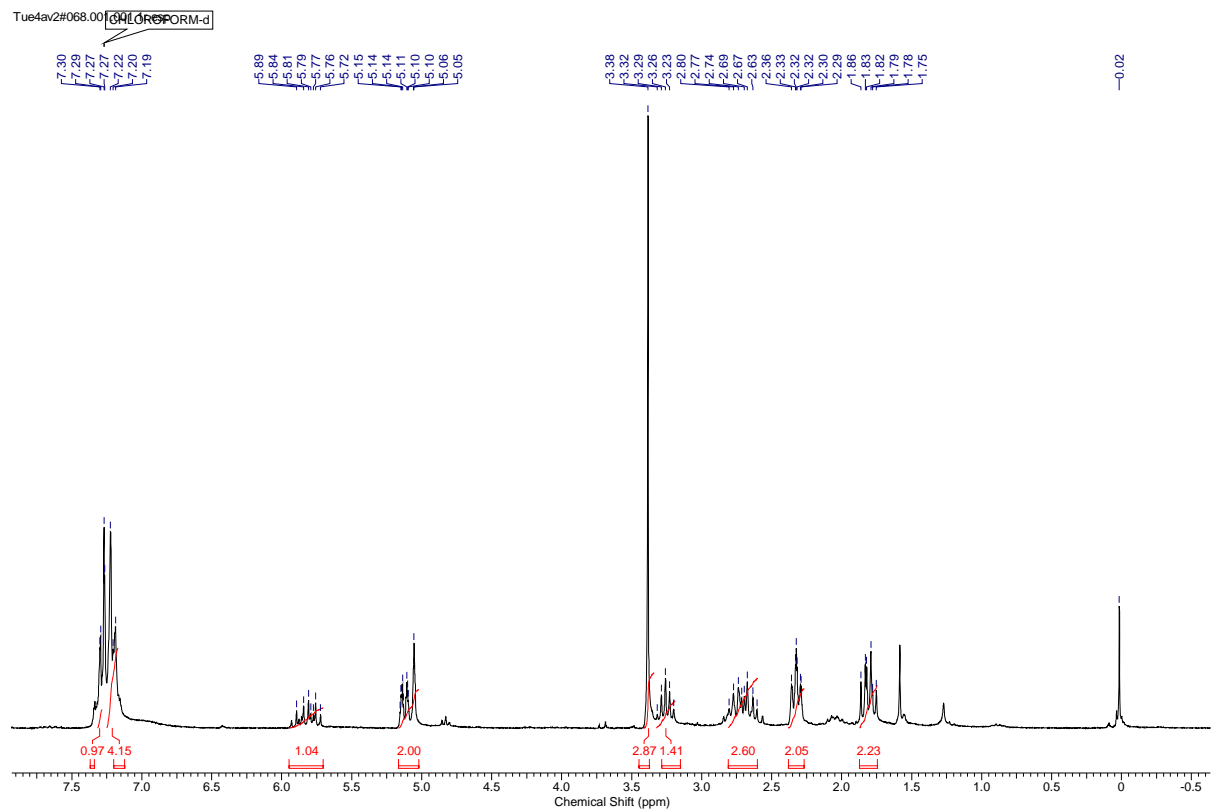


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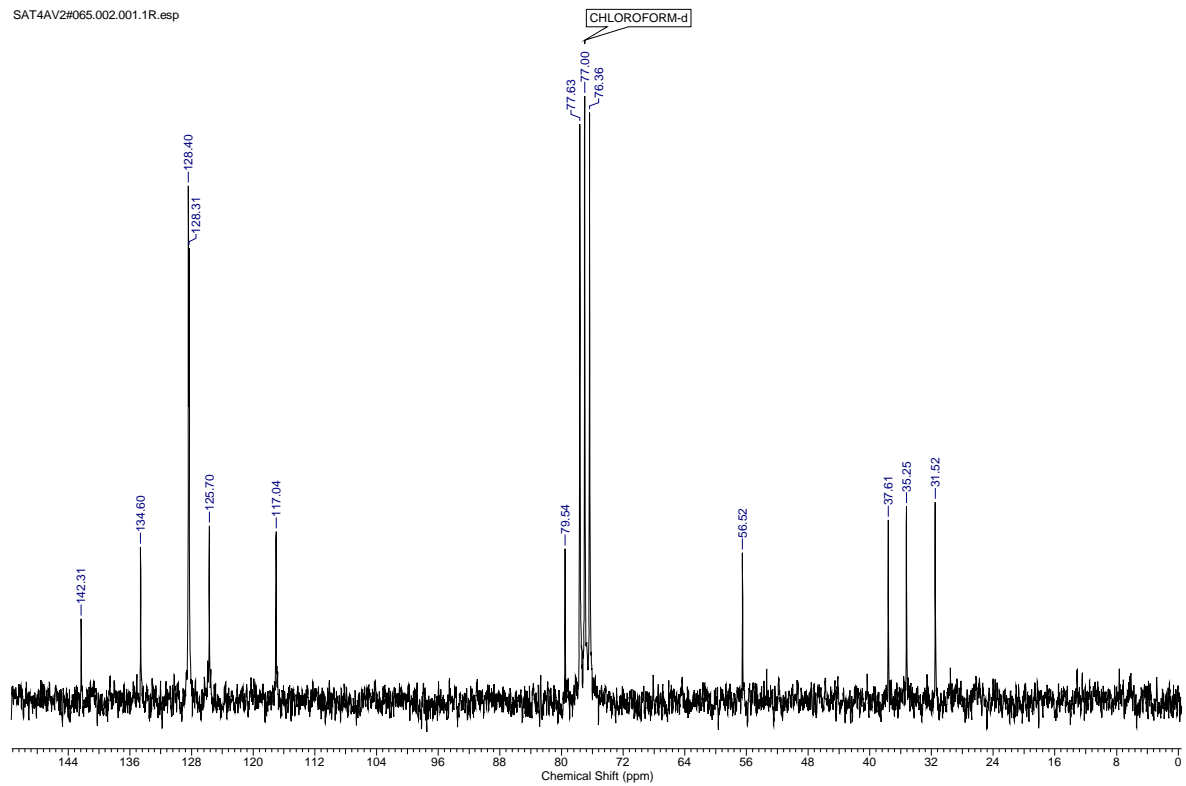




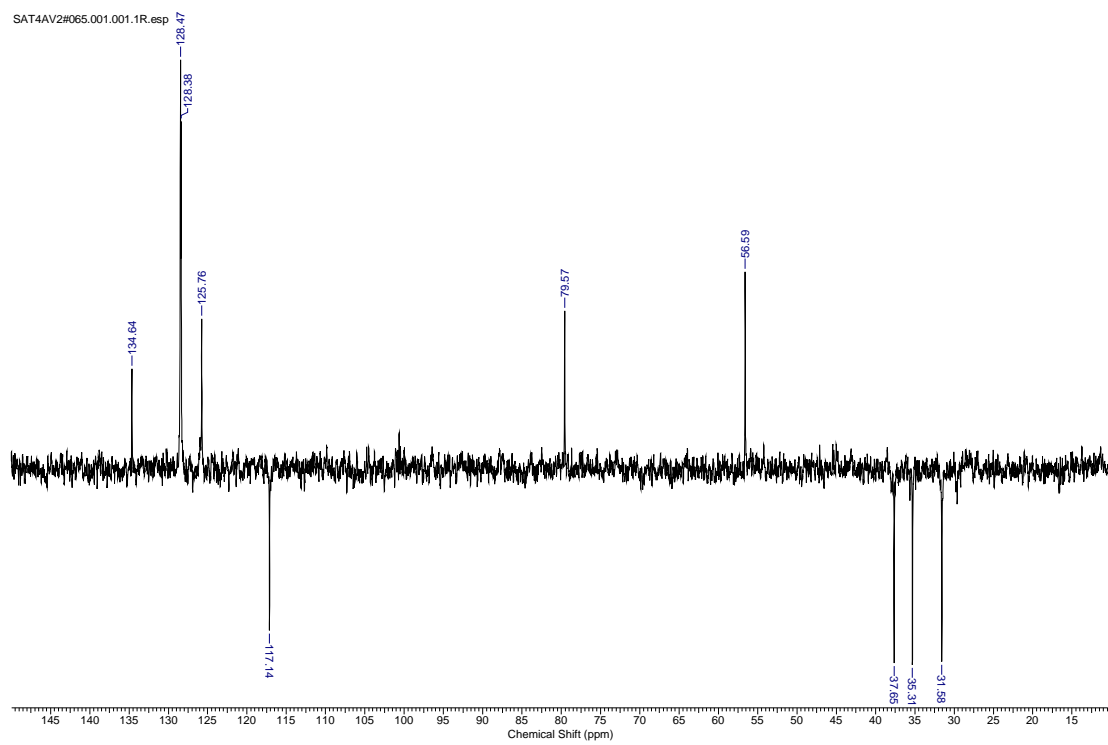
(3-methoxyhex-5-en-1-yl)benzene (Table 2, entry 14)

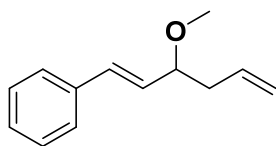


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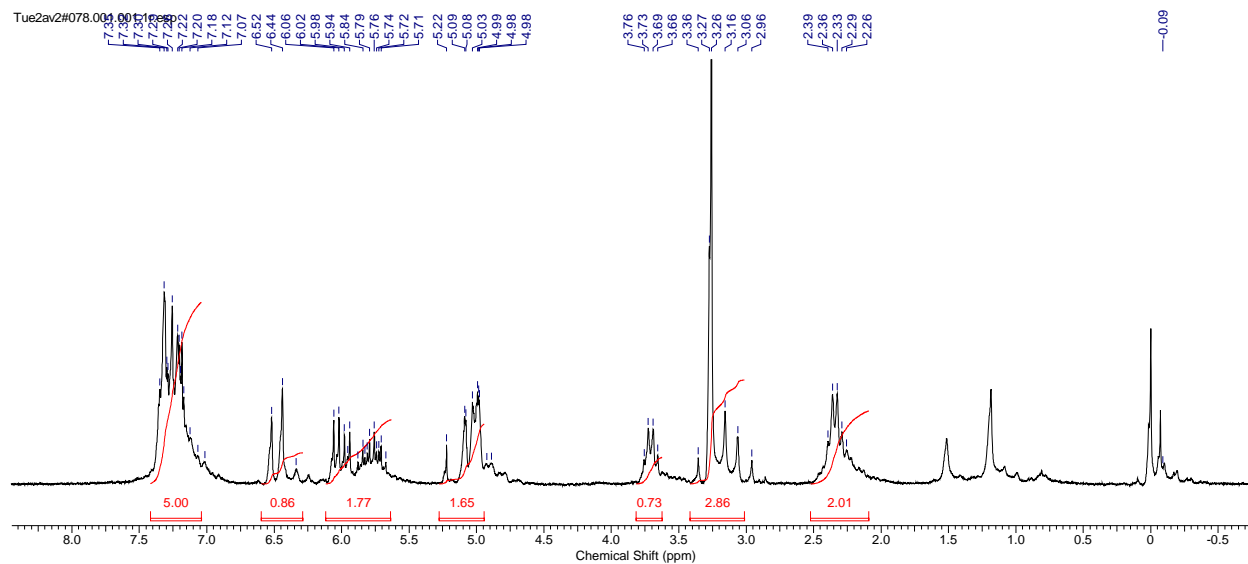


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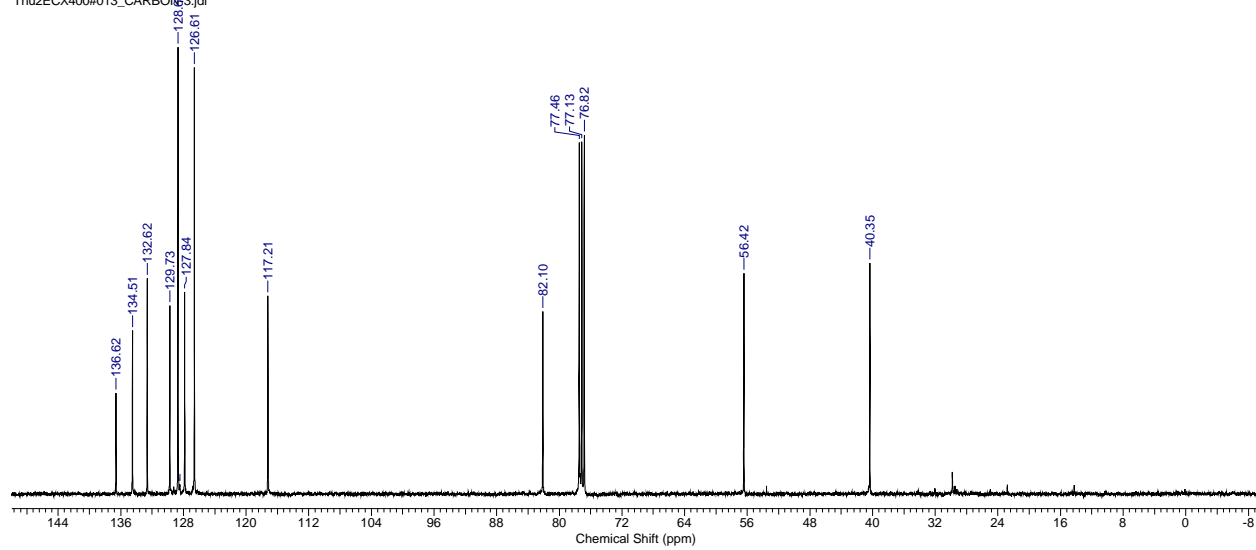




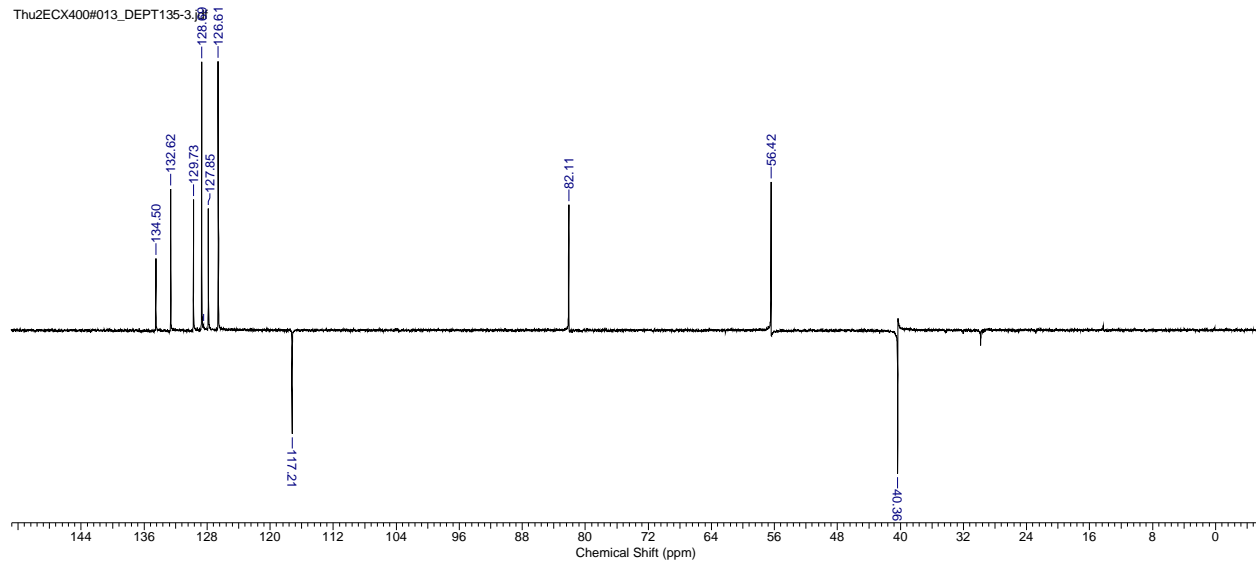
(E)-(3-methoxyhexa-1,5-dien-1-yl)benzene (Table 2, entry 15)

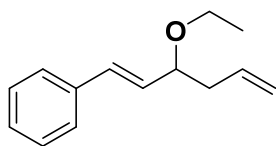


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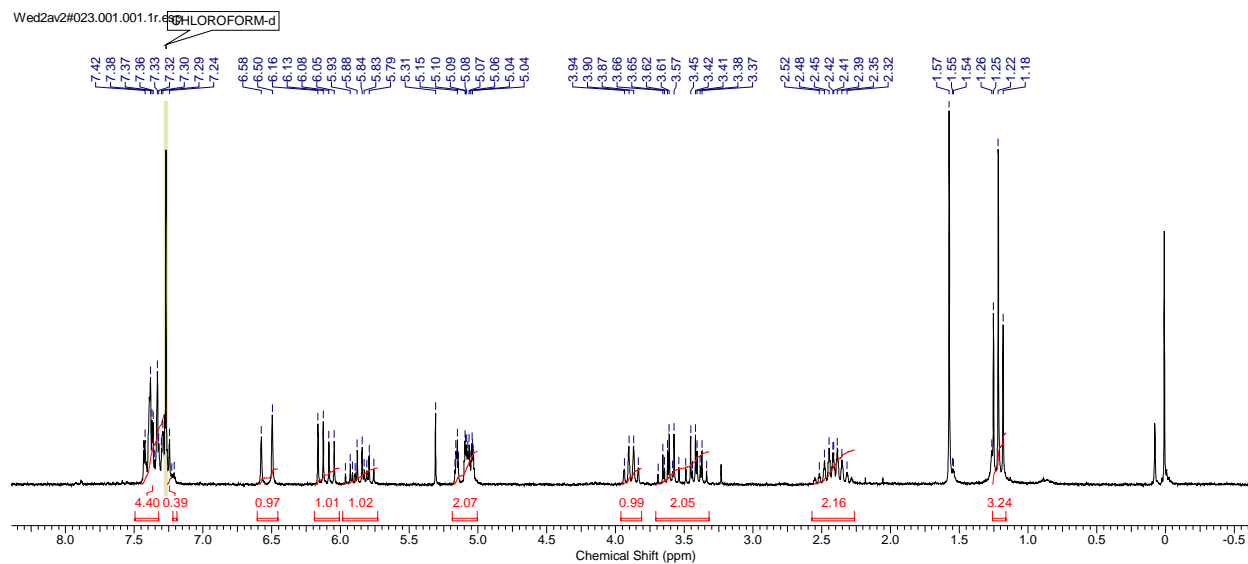


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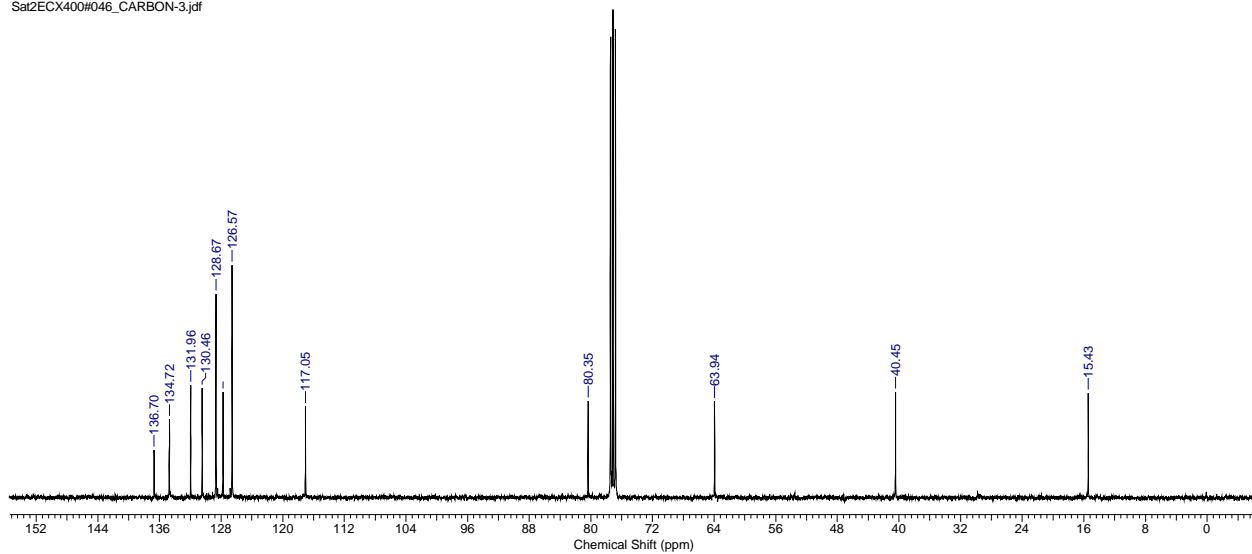




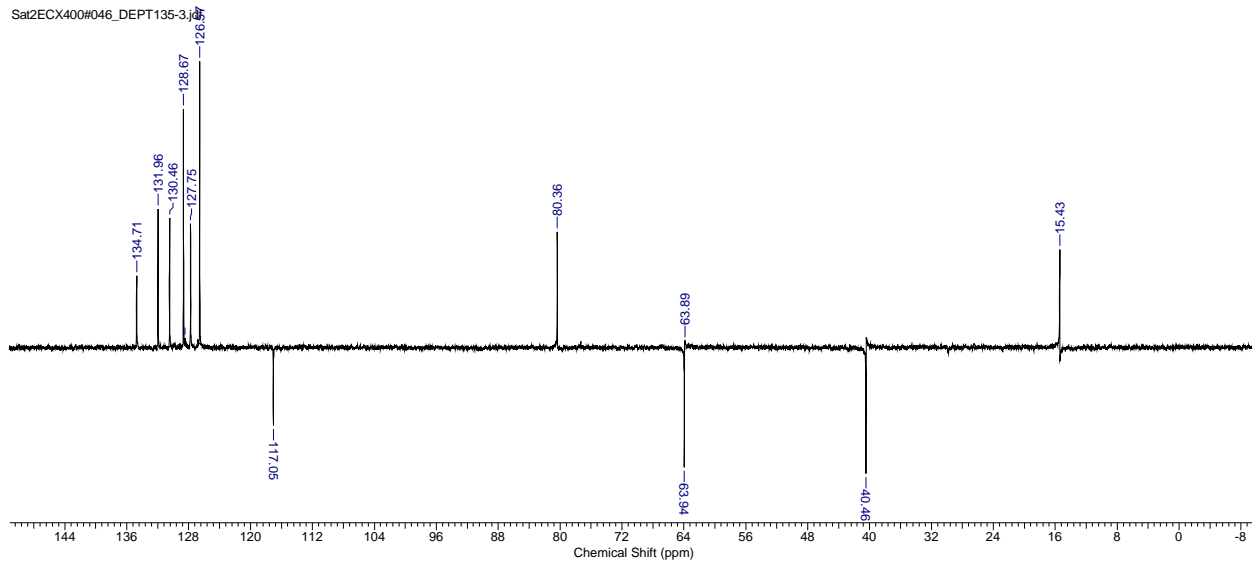
(E)-(3-ethoxyhexa-1,5-dien-1-yl)benzene (Table 2, entry 16)

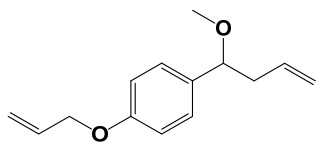


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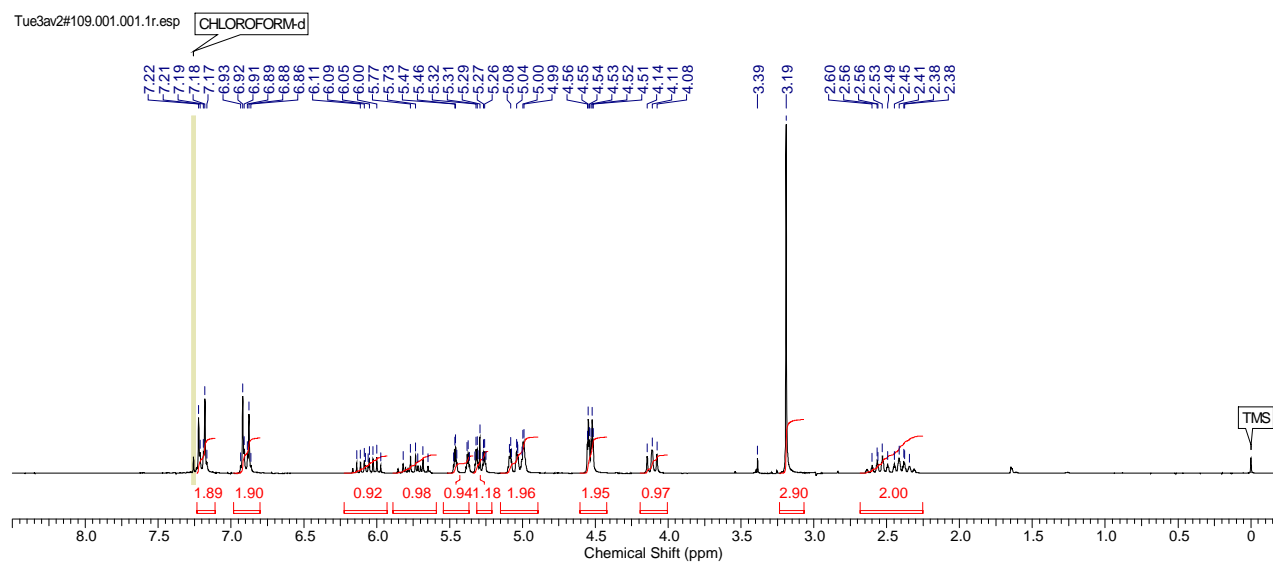


Sat2ECX400#046_DEPT135-3.jdf

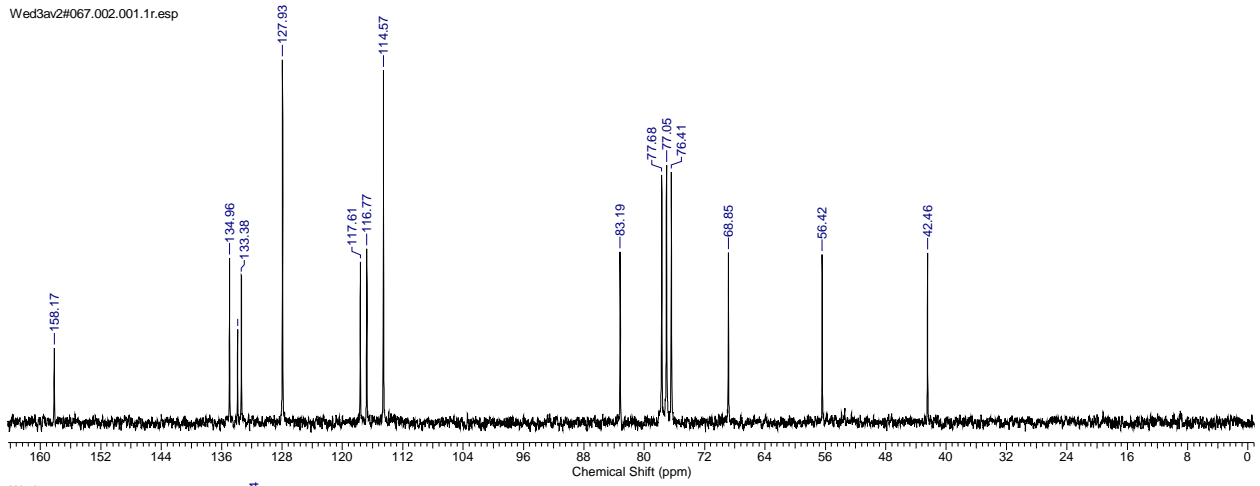




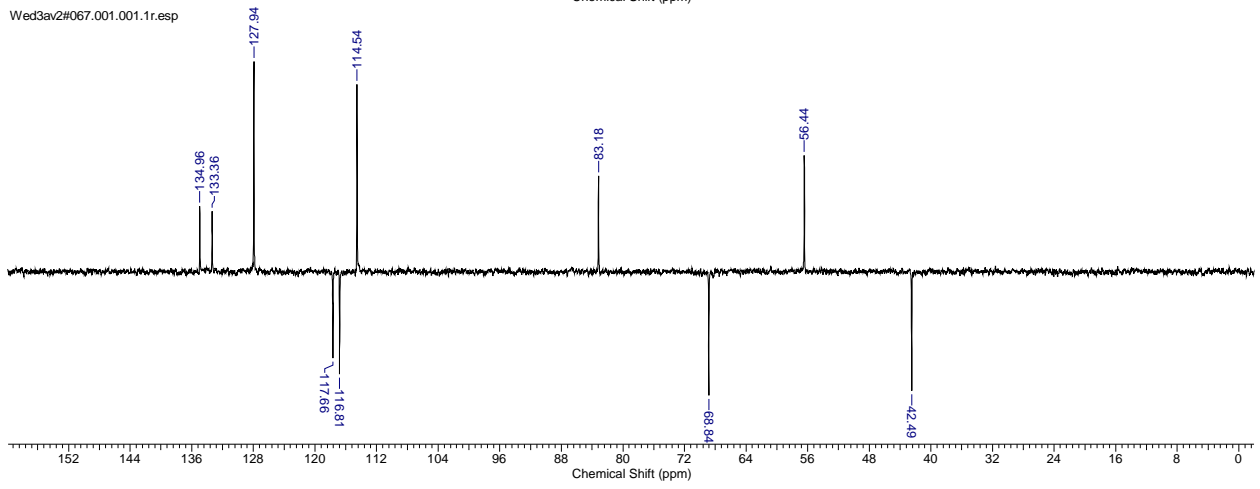
1-(allyloxy)-4-(1-methoxybut-3-en-1-yl) benzene (Table 2, entry 17)

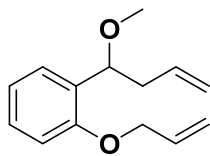


Wed3av2#067.002.001.1r.esp

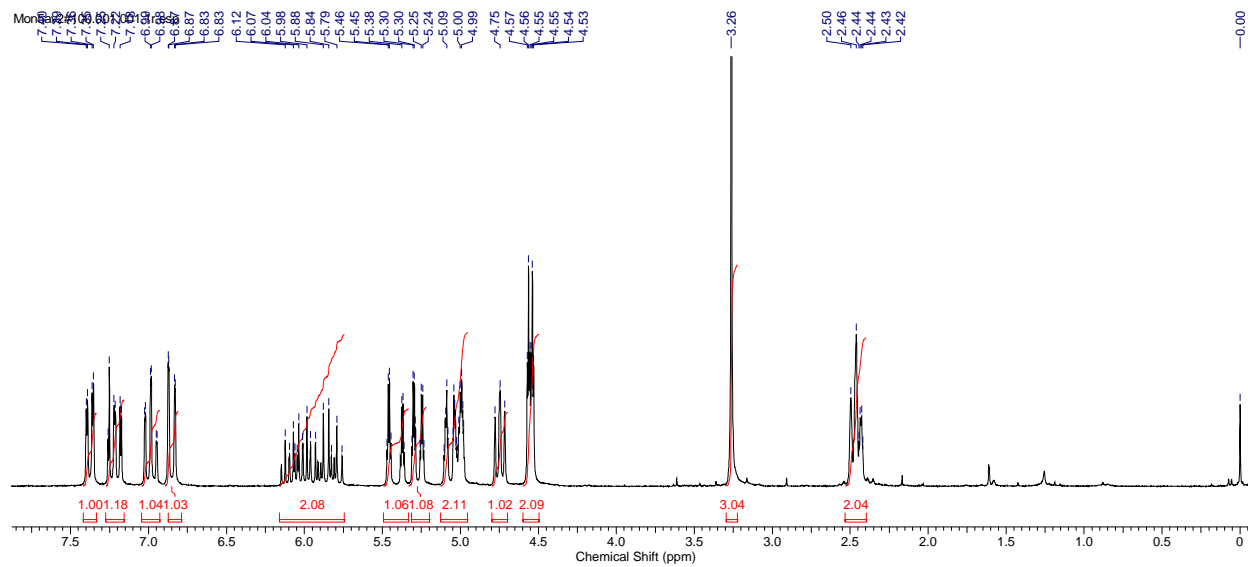


Wed3av2#067.001.001.1r.esp

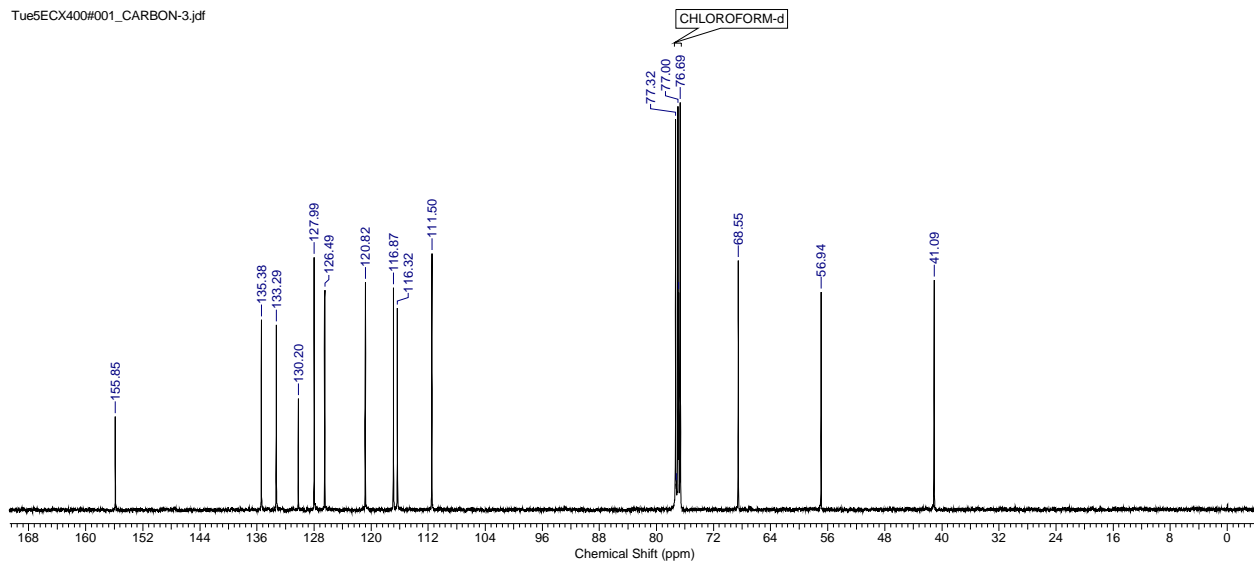




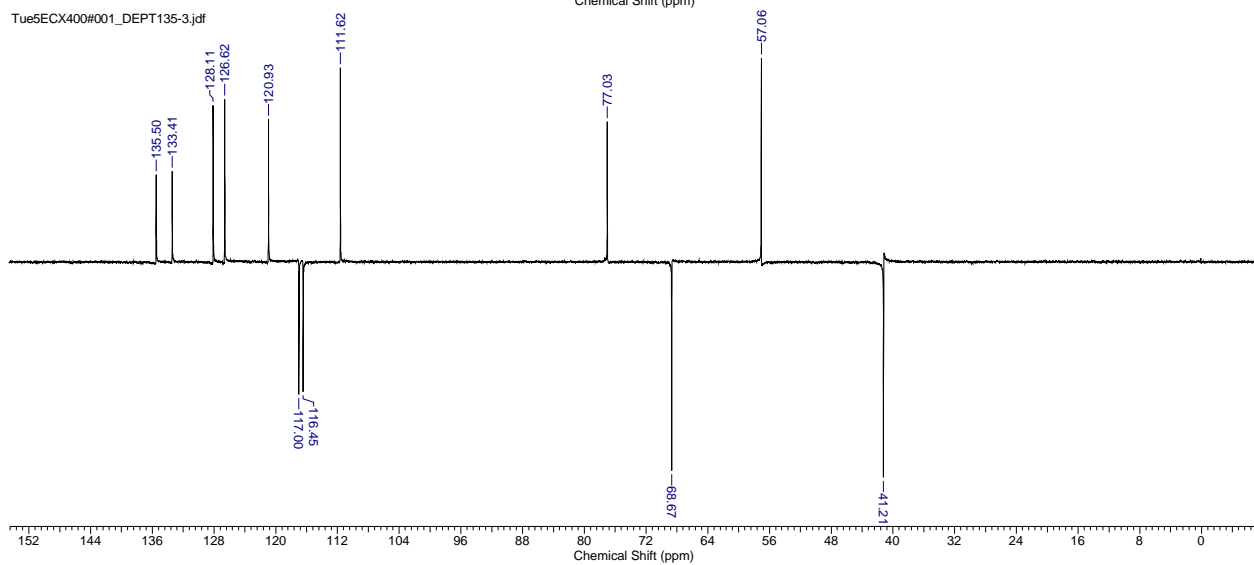
1-(allyloxy)-2-(1-methoxybut-3-en-1-yl) benzene (Table 2, entry 18)



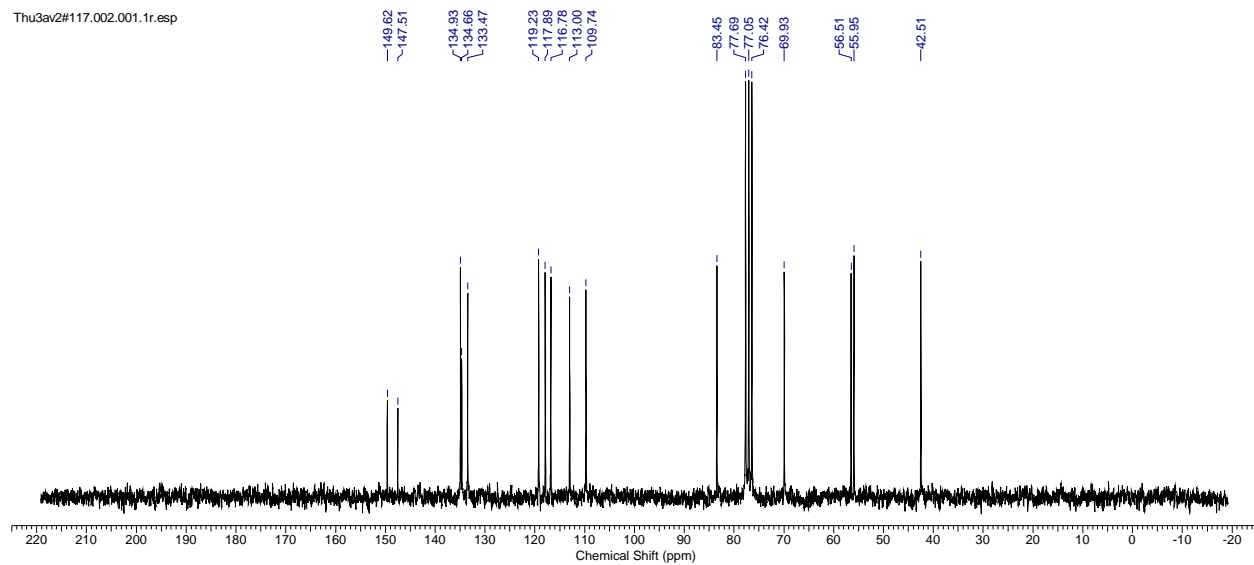
Tue5ECX400#001_CARBON-3.jdf



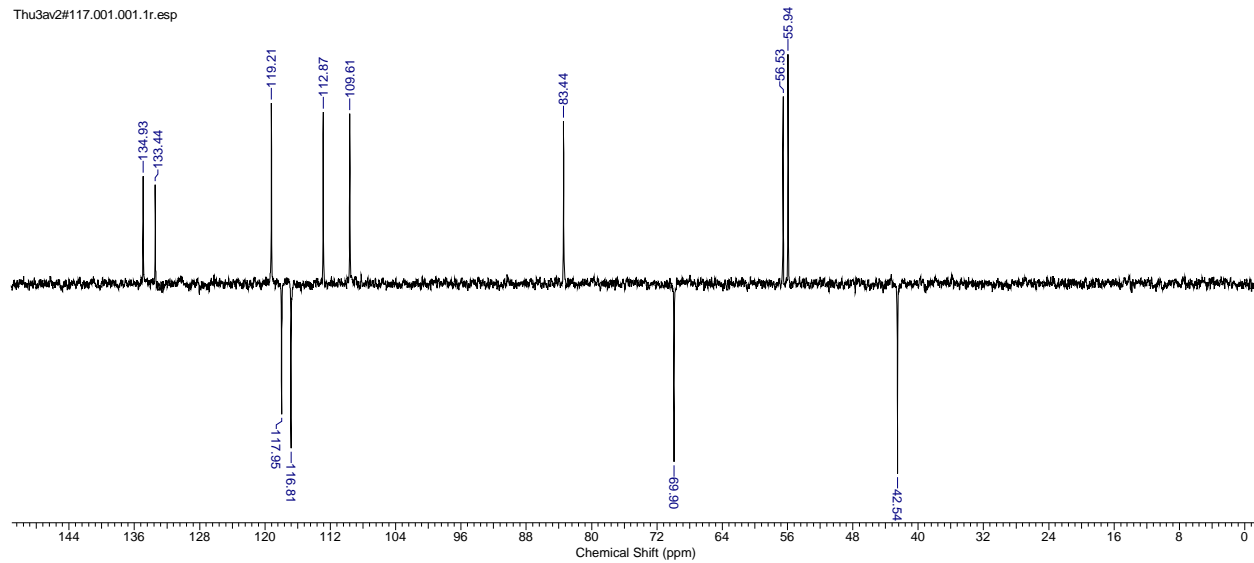
Tue5ECX400#001_DEPT135-3.jdf

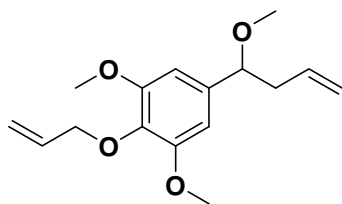


Thu3av2#117.002.001.1r.esp



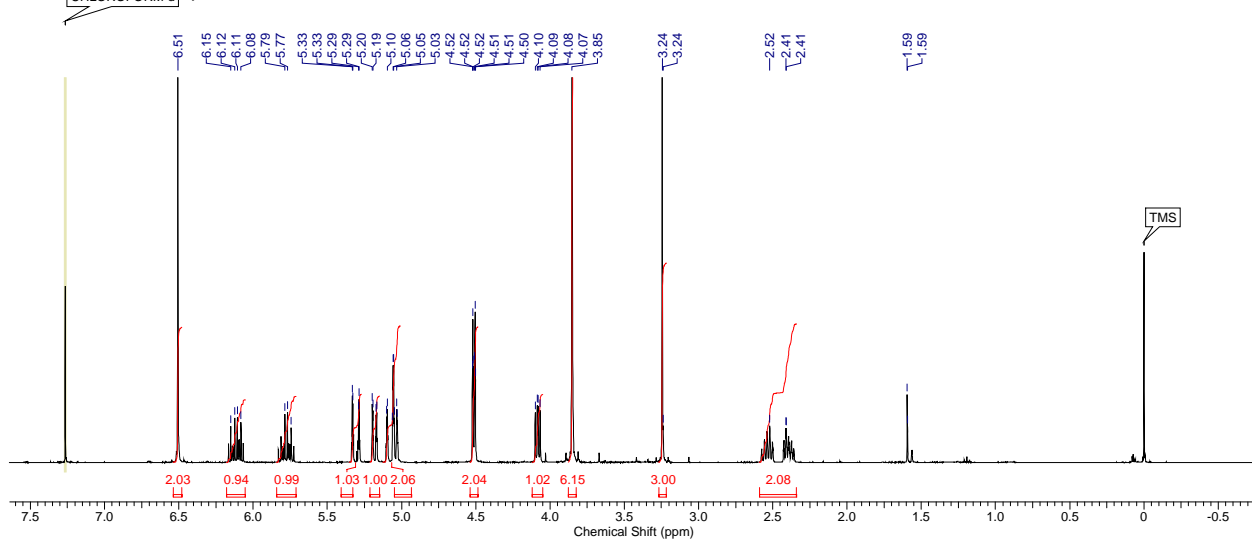
Thu3av2#117.001.001.1r.esp



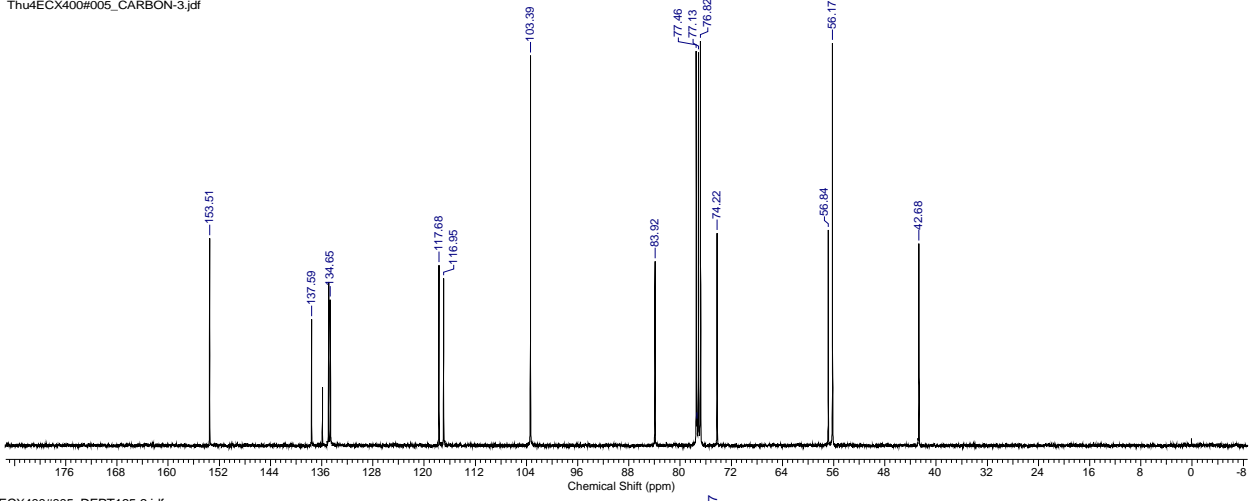


2-(allyloxy)-1,3-dimethoxy-5-(1-methoxybut-3-en-1-yl)benzene (Table 2, entry 20)

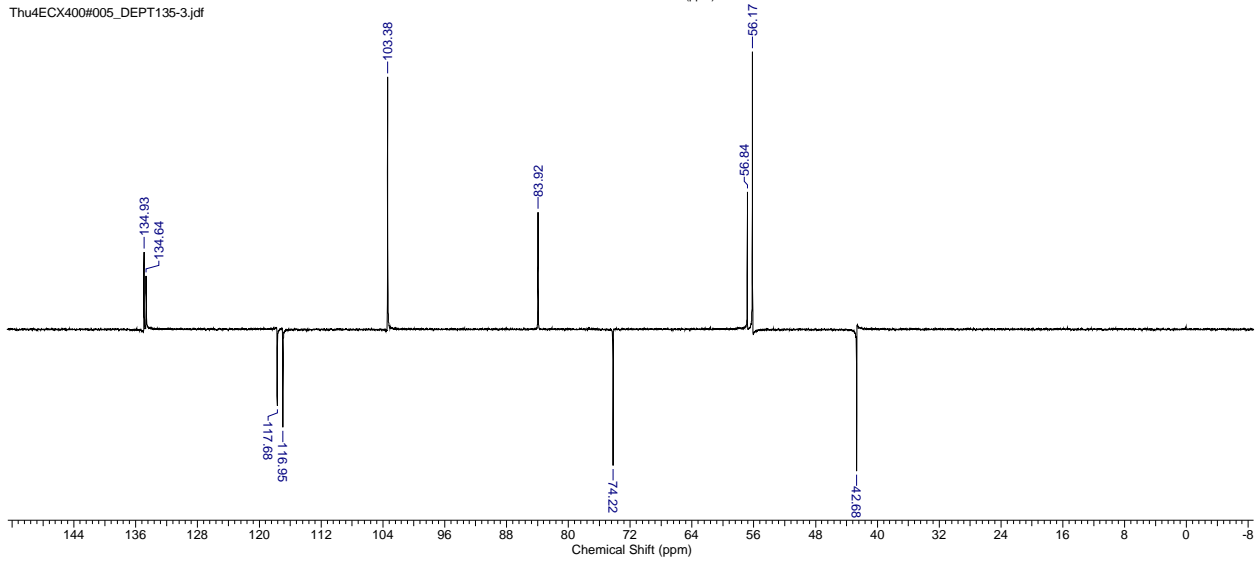
Tue4ECX1601286CP030101-3.jdf

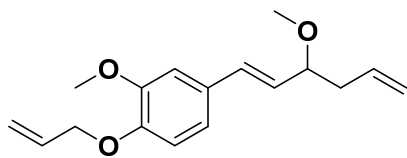


Thu4ECX400#005_CARBON-3.jdf

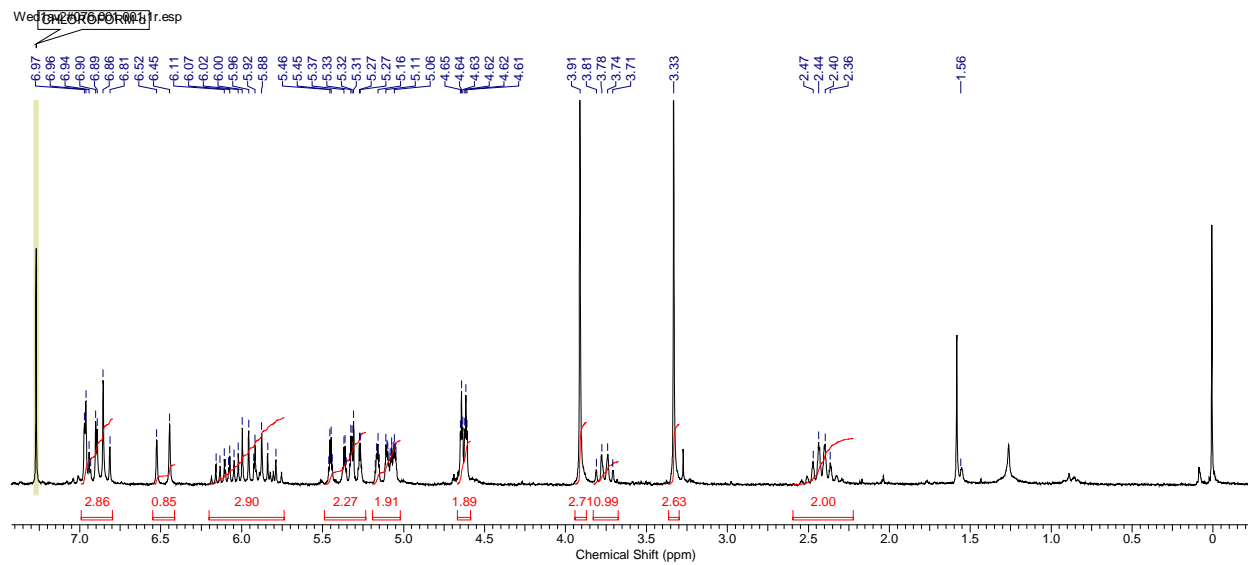


Thu4ECX400#005_DEPT135-3.jdf

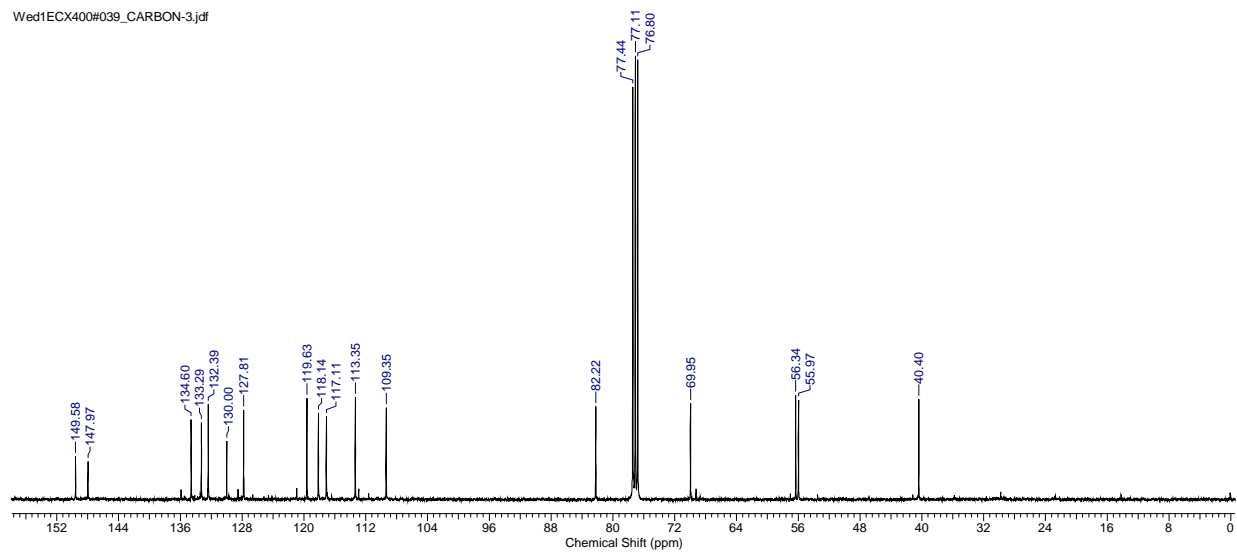




1-(allyloxy)-2-methoxy-4-(3-methoxyhexa-1,5-dien-1-yl)benzene (Table 2, entry 21)



Wed1ECX400#039_CARBON-3.jdf



Wed1ECX400#039_DEPT135-3.jdf

