Bismuth(III) Triflate-catalyzed Synthesis of Substituted 2-Alkenylfurans

Supporting information

Dominik Nitsch,[1] Thorsten Bach[1*]

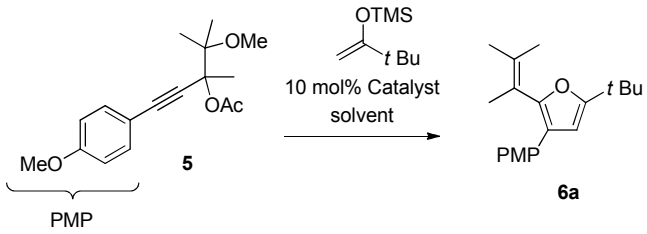
[1] Lehrstuhl für Organische Chemie I and Catalysis Research Center (CRC), Technische Universität München, Lichtenbergstr. 4, 85747 Garching, Germany

Corresponding Author

[*]Email: thorsten.bach@ch.tum.de

1. Optimization of Reaction Conditions for the Synthesis of Substituted 2-Alkenylfurans

Table S1 Optimization of reaction conditions for the reaction of propargylic acetate **5** with [(3,3-dimethylbut-1-en-2-yl)oxy]trimethylsilane to **6a**



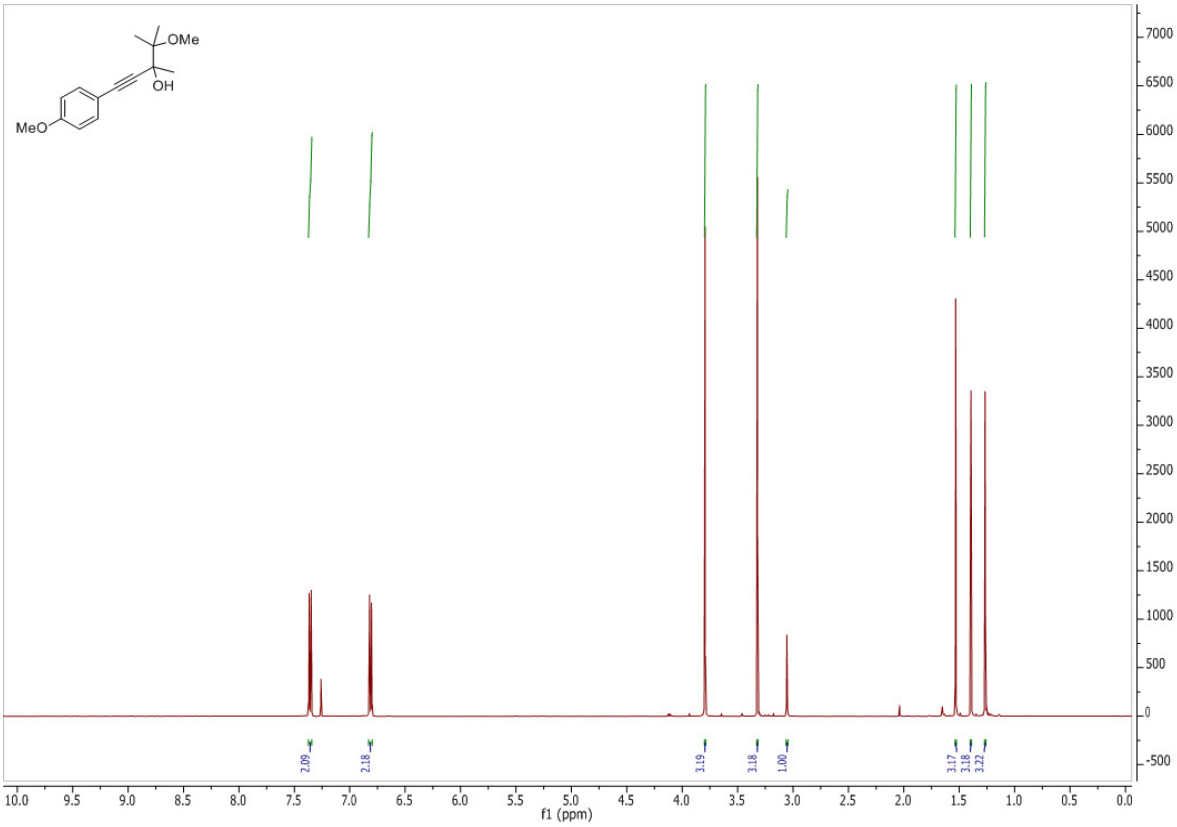
Entry[a]	Catalyst	Solvent	Temperature	Yield [%]
1	$Bi(OTf)_3$	$MeNO_2$	r.t.	67
2	$InCl_3$	$MeNO_2$	r.t.	63
3	AgOTf	$MeNO_2$	r.t.	62
4	$Sc(OTf)_3$	$MeNO_2$	r.t.	65
5	$AuCl_3$	$MeNO_2$	r.t.	37
6	HOTf	$MeNO_2$	r.t.	61
7	$Bi(OTf)_3$	CH_2Cl_2	r.t.	65
8	$Bi(OTf)_3$	MeCN	r.t.	47
9	$Bi(OTf)_3$	CH_2Cl_2	-78 °C	50
10	**$Bi(OTf)_3$**	**$MeNO_2$**	**0 °C**	**77**
11[b]	$Bi(OTf)_3$	$MeNO_2$	0 °C	70
12[c]	$Bi(OTf)_3$	$MeNO_2$	0 °C	74

[a] Reactions were performed with dry solvents (c = 125 mM), **5** (125 µmol), silyl enol ether (0.5 mmol) and catalyst (13 µmol). [b] Only 5 mol% catalyst loading was used. [c] **5** (2.5 mmol), silyl enol ether (10.0 mmol), $Bi(OTf)_3$ (250 µmol), $MeNO_2$ (20 mL).

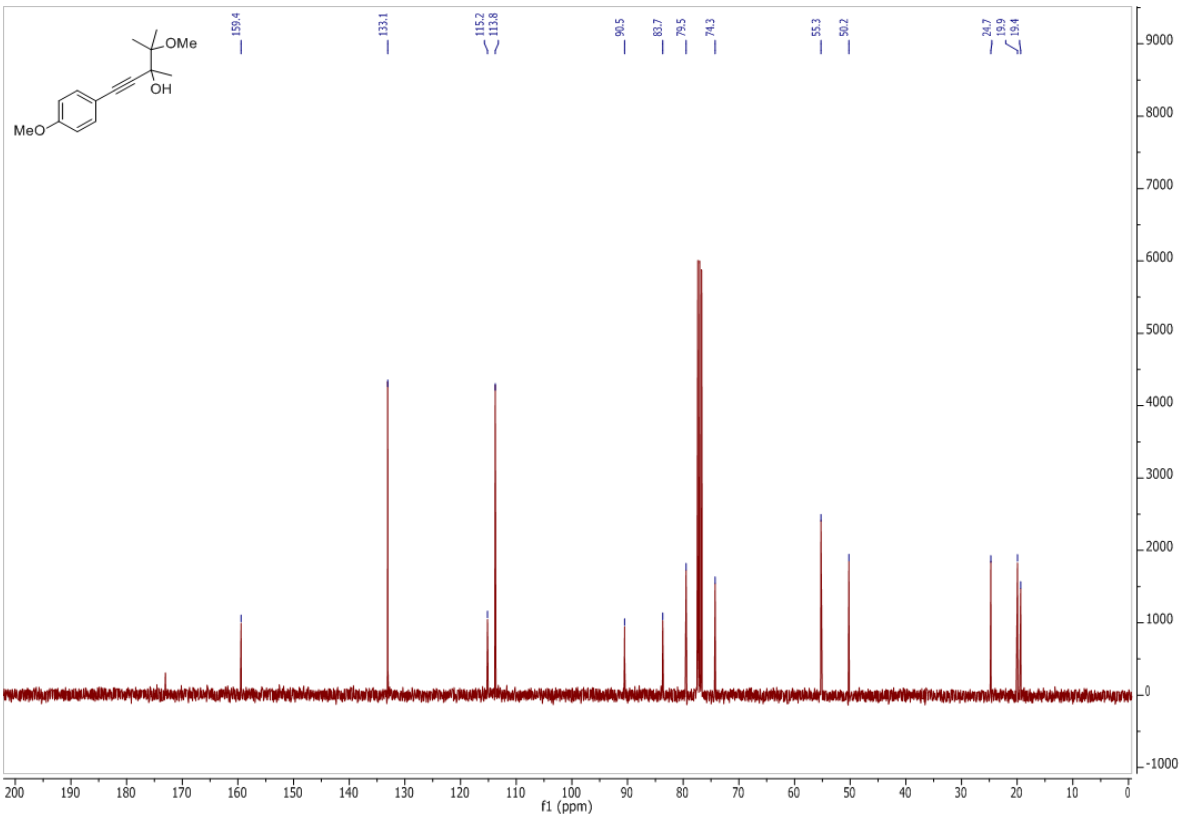
2. 1H and 13 C NMR spectra for compounds **5**, **6**, **7**, **8**, **9**, and **10**

4-Methoxy-1-(4-methoxyphenyl)-3,4-dimethylpent-1-yn-3-ol (7)

^{1}H NMR (300 K, $CDCl_3$)

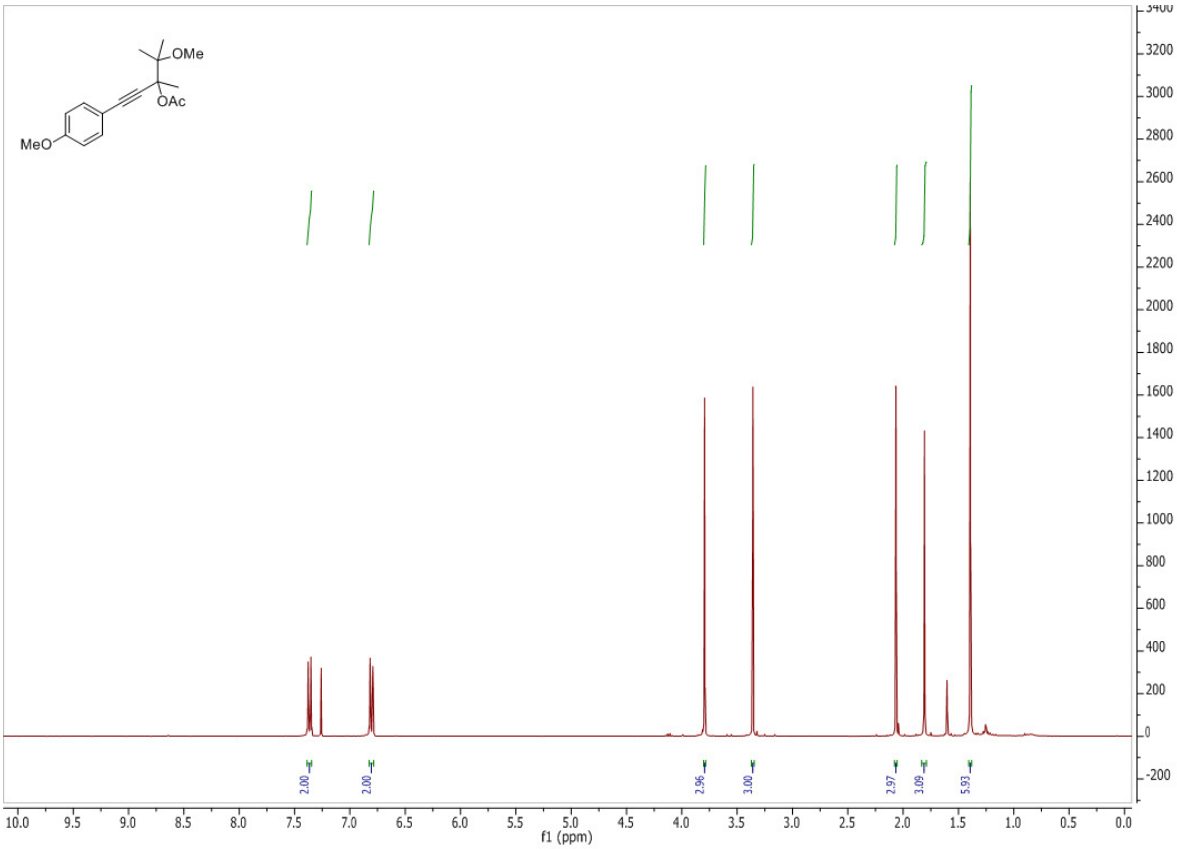


^{13}C NMR (300 K, $CDCl_3$)

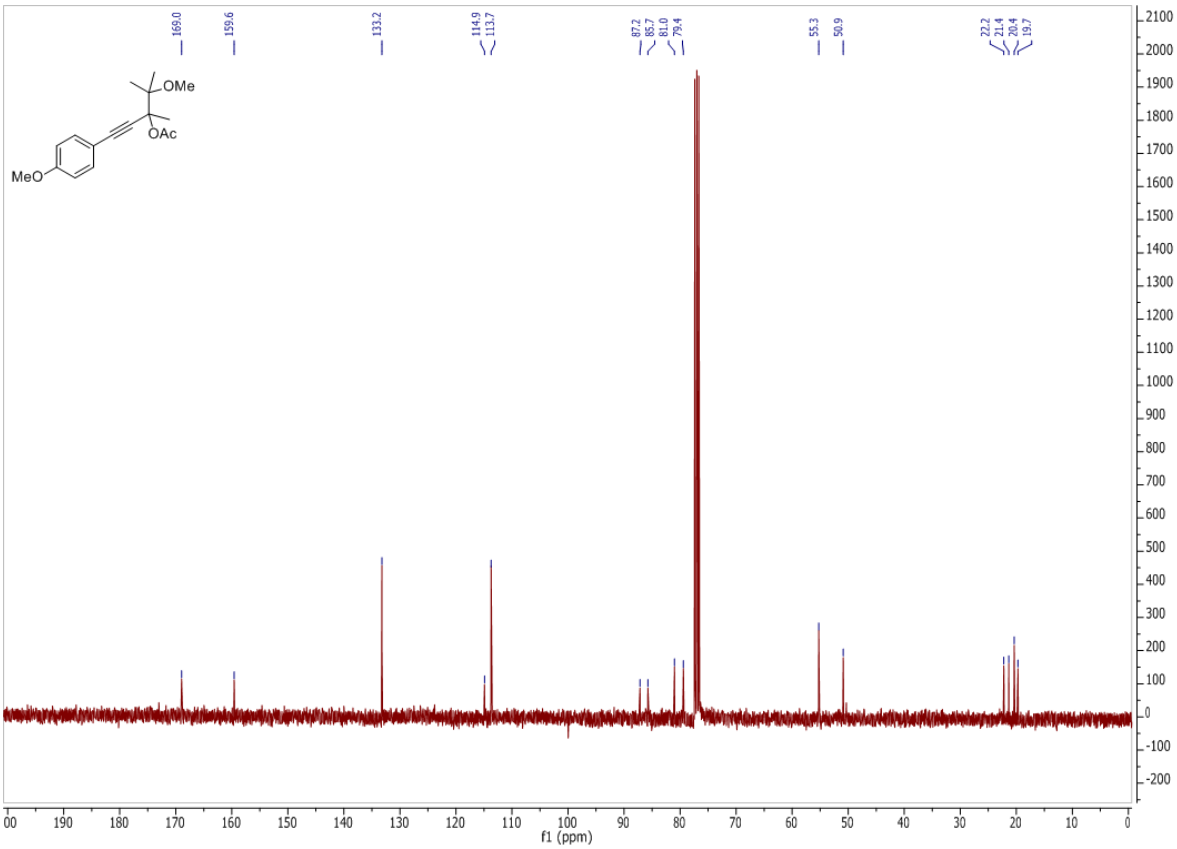


4-Methoxy-1-(4-methoxyphenyl)-3,4-dimethylpent-1-yn-3-yl acetate (5)

^{1}H NMR (300 K, $CDCl_3$)

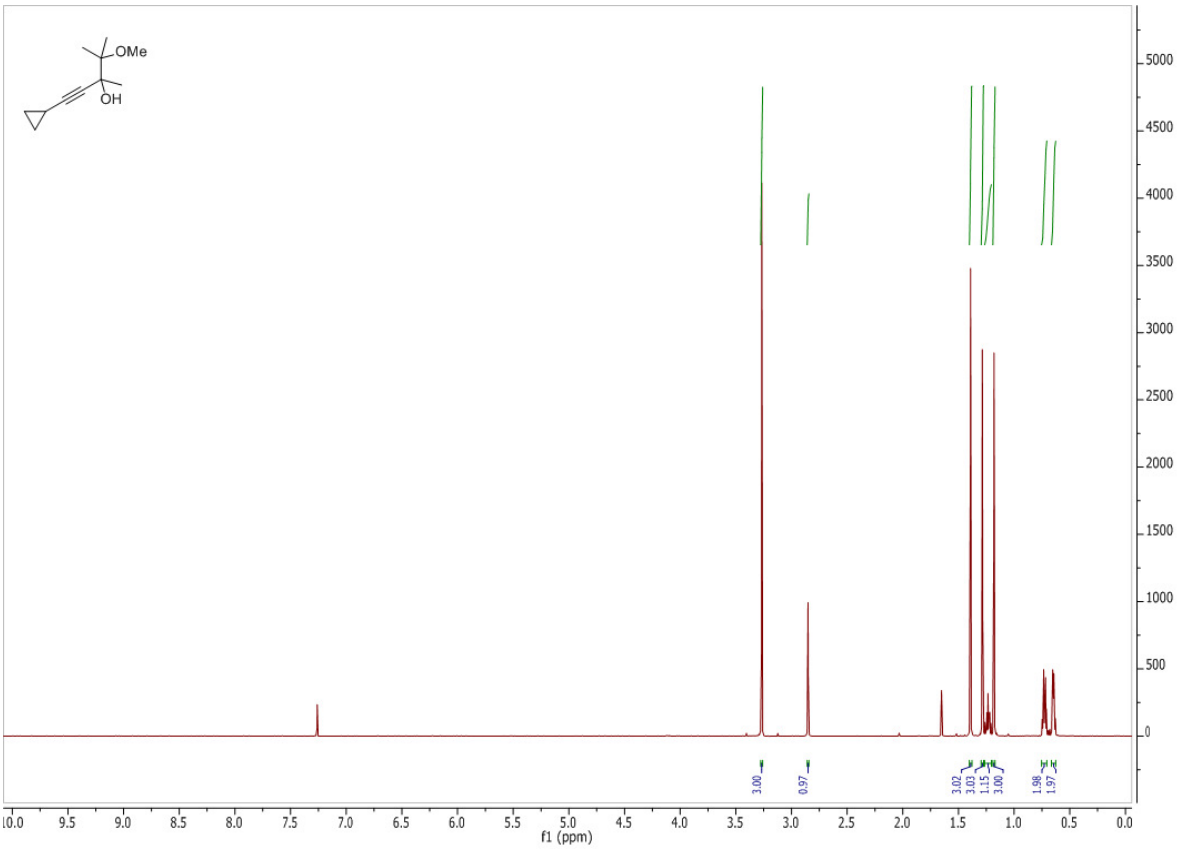


^{13}C NMR (300 K, $CDCl_3$)

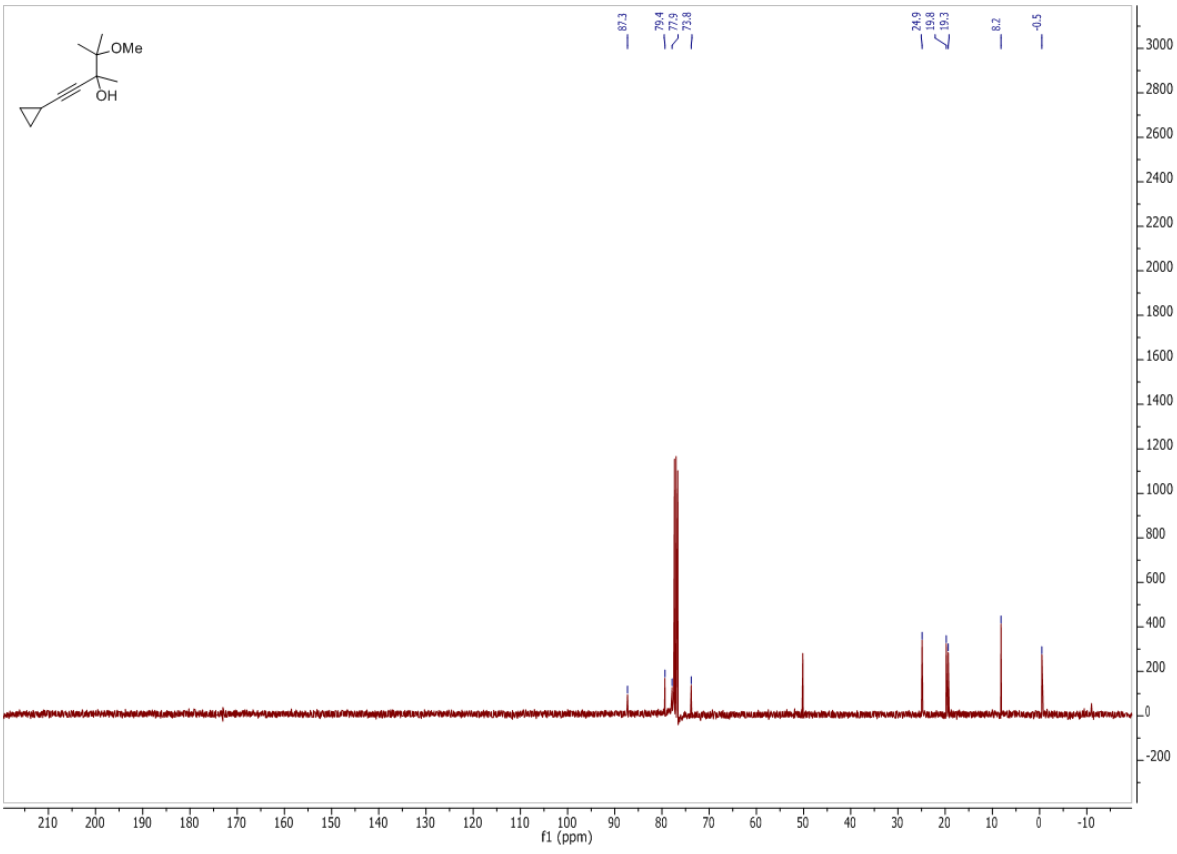


1-Cyclopropyl-4-methoxy-3,4-dimethylpent-1-yn-3-ol (8a)

^{1}H NMR (300 K, $CDCl_3$)

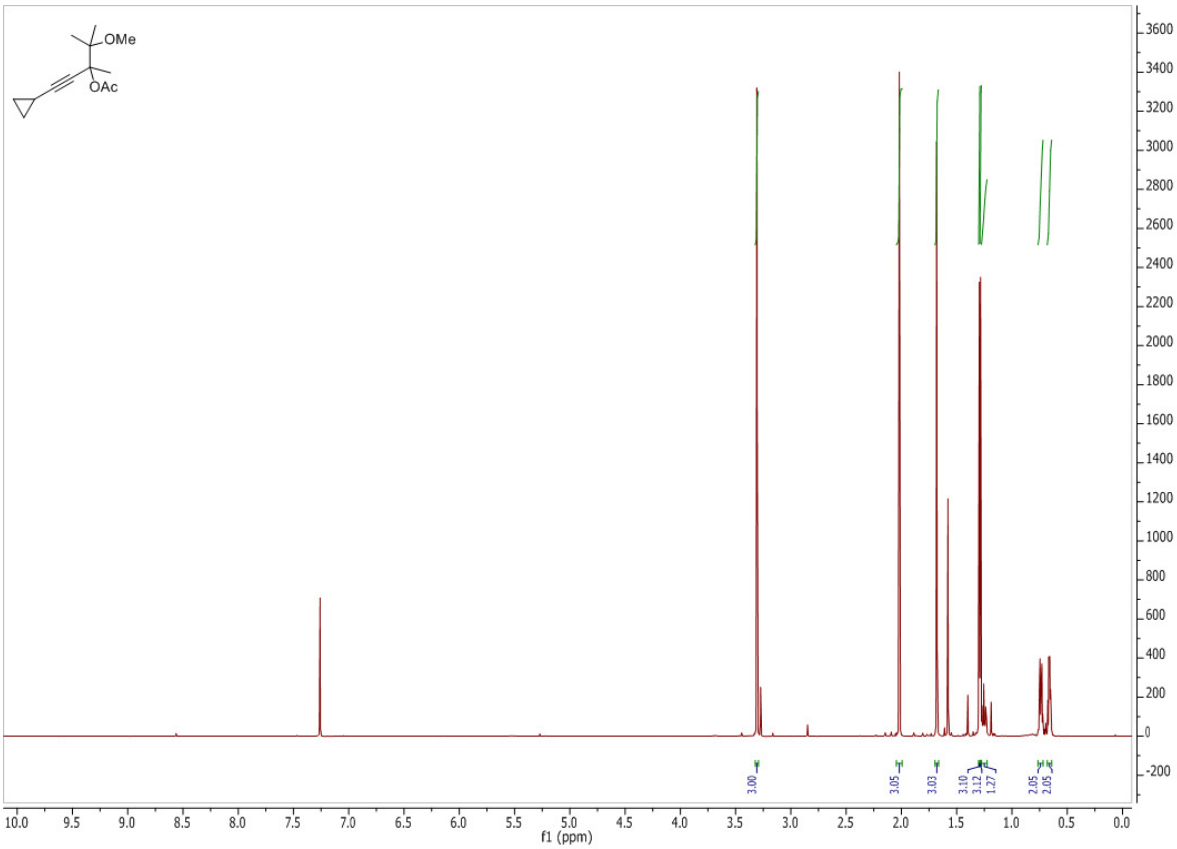


^{13}C NMR (300 K, $CDCl_3$)

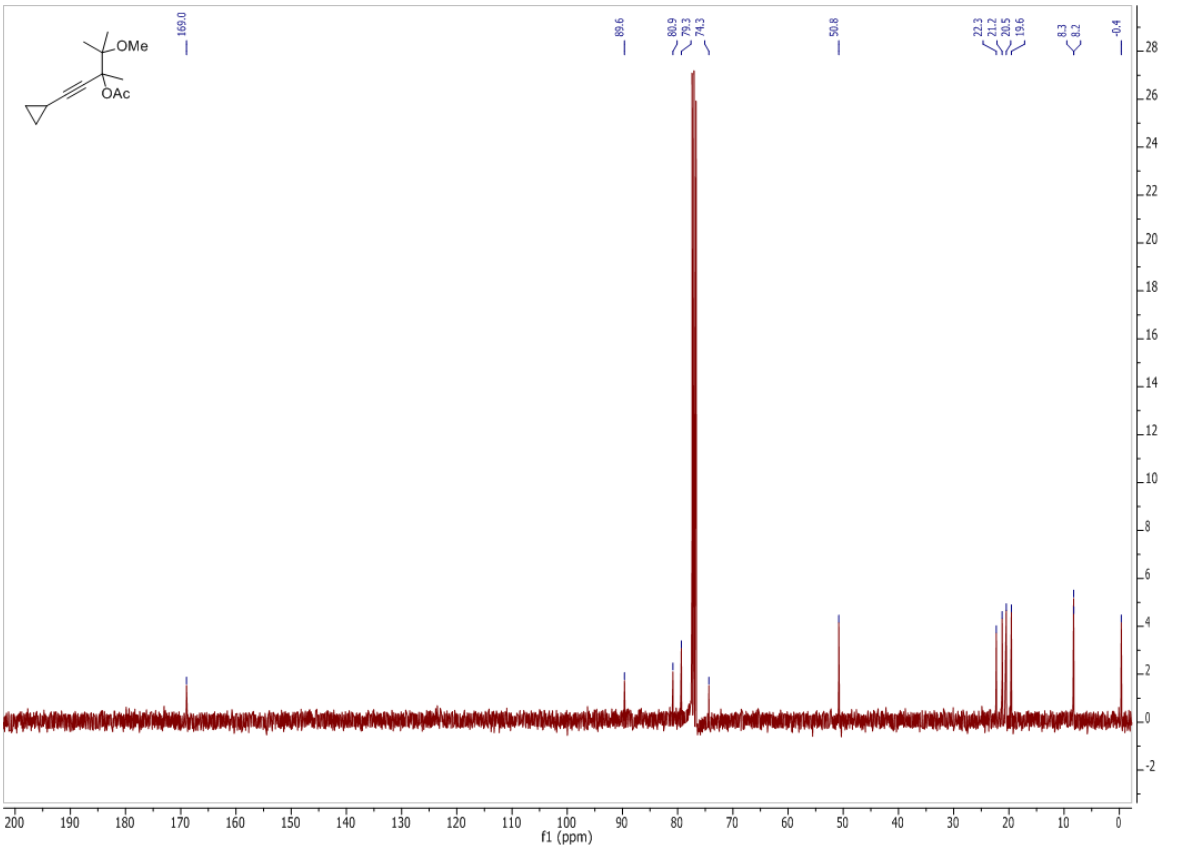


1-Cyclopropyl-4-methoxy-3,4-dimethylpent-1-yn-3-yl acetate (9a)

^{1}H NMR (300 K, $CDCl_3$)

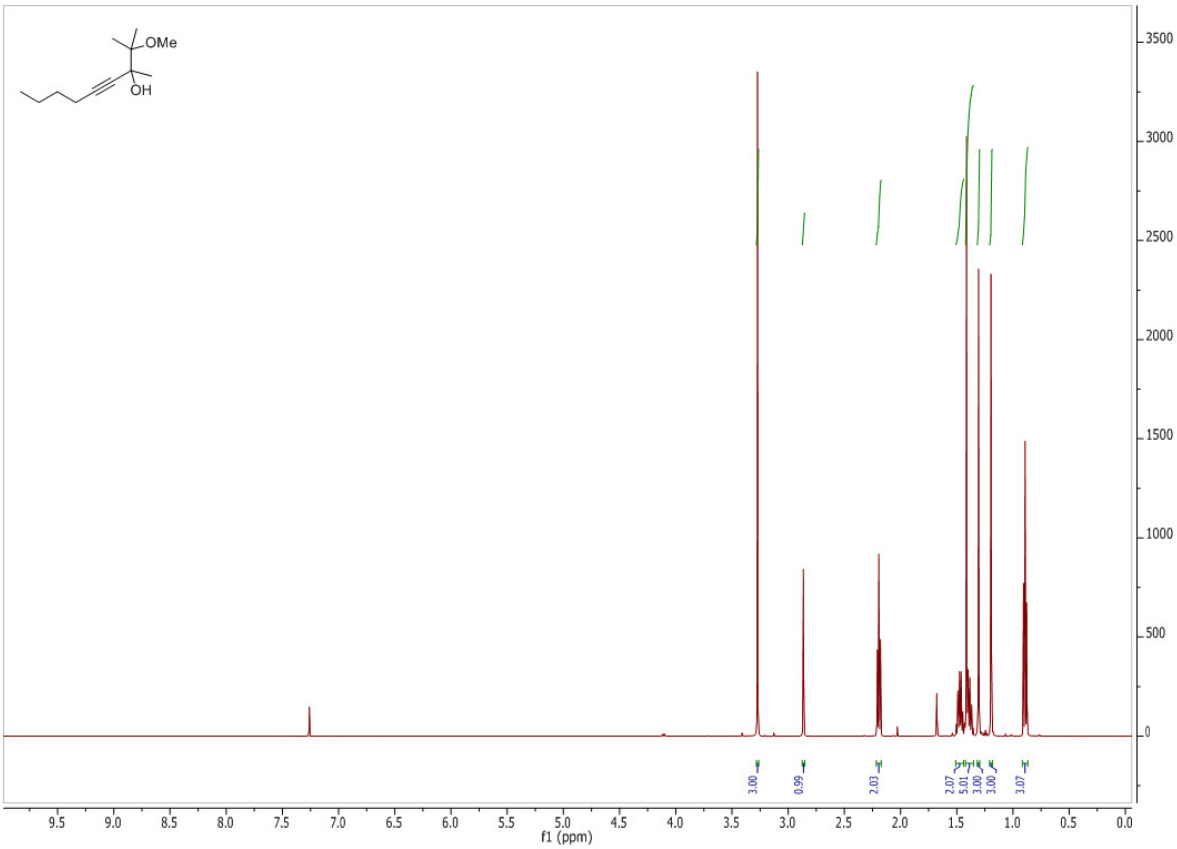


^{13}C NMR (300 K, $CDCl_3$)

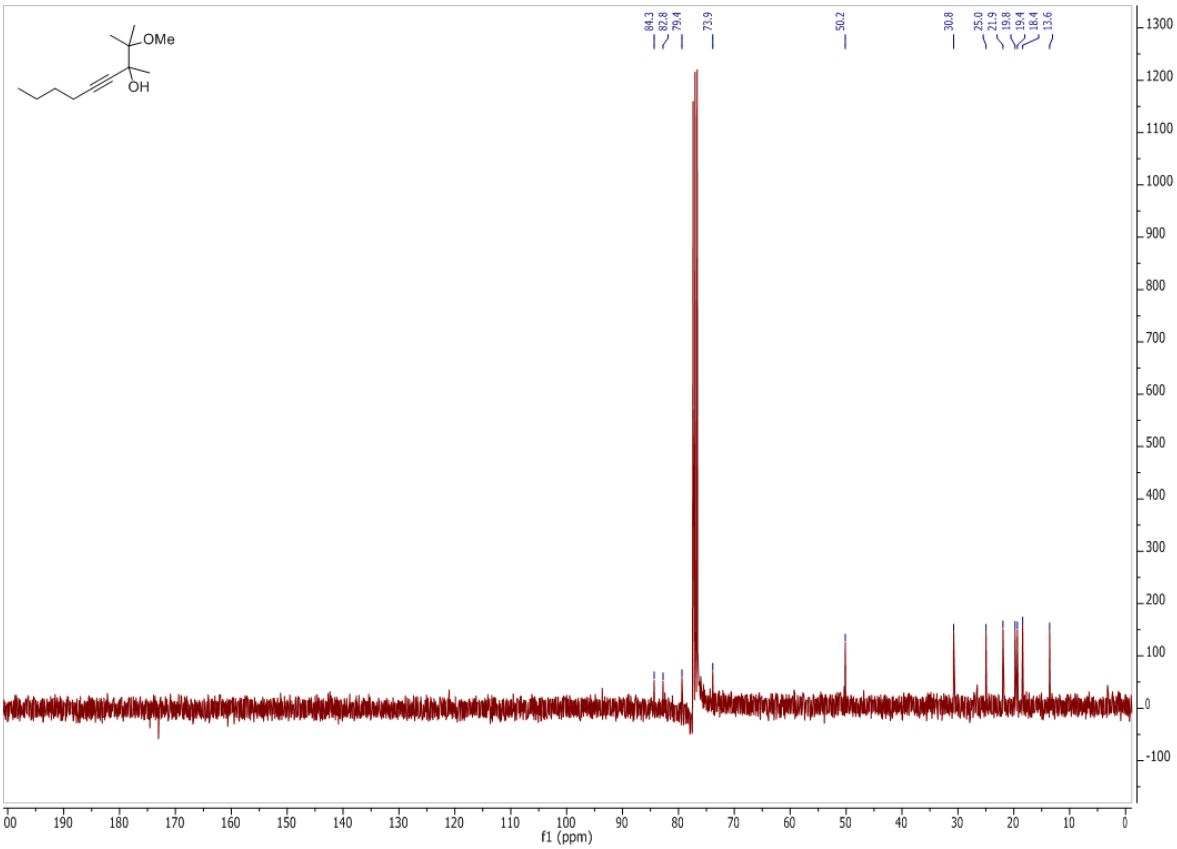


2-Methoxy-2,3-dimethylnon-4-yn-3-ol (8b)

^{1}H NMR (300 K, $CDCl_3$)

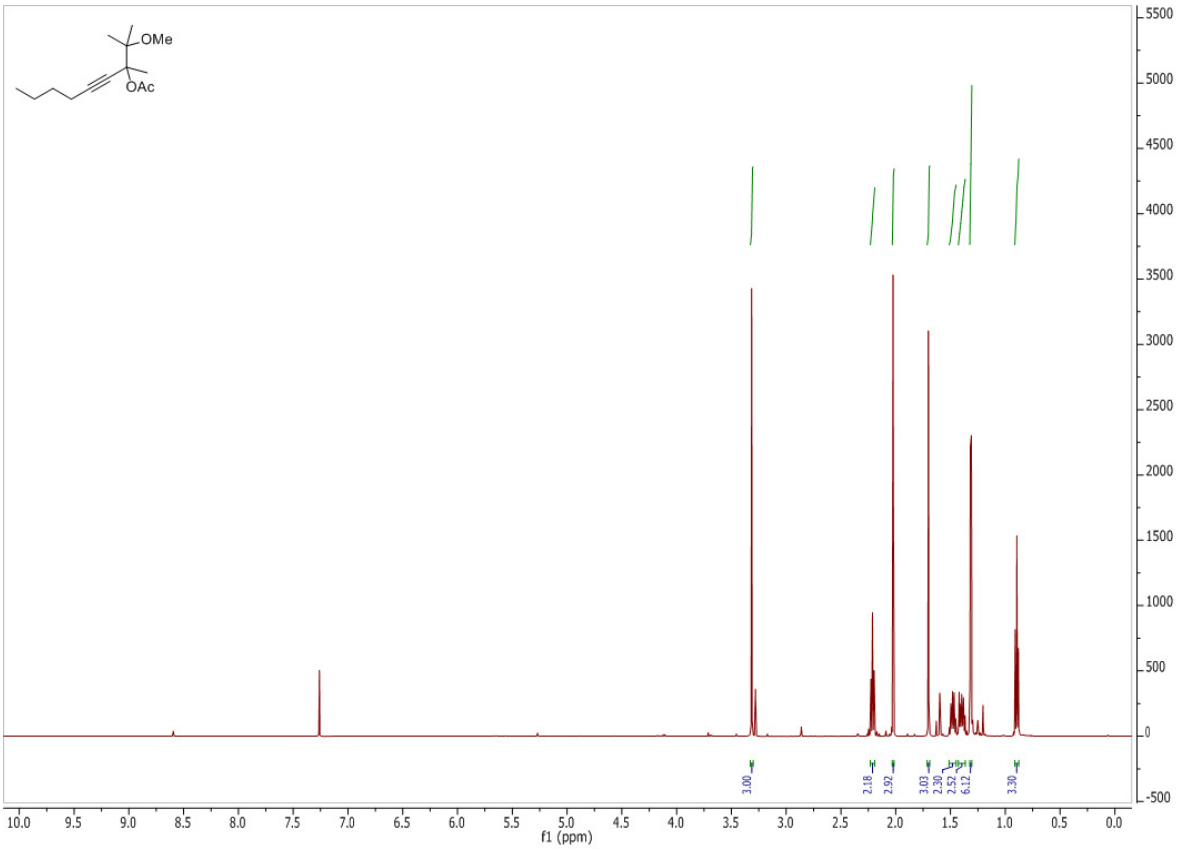


^{13}C NMR (300 K, $CDCl_3$)

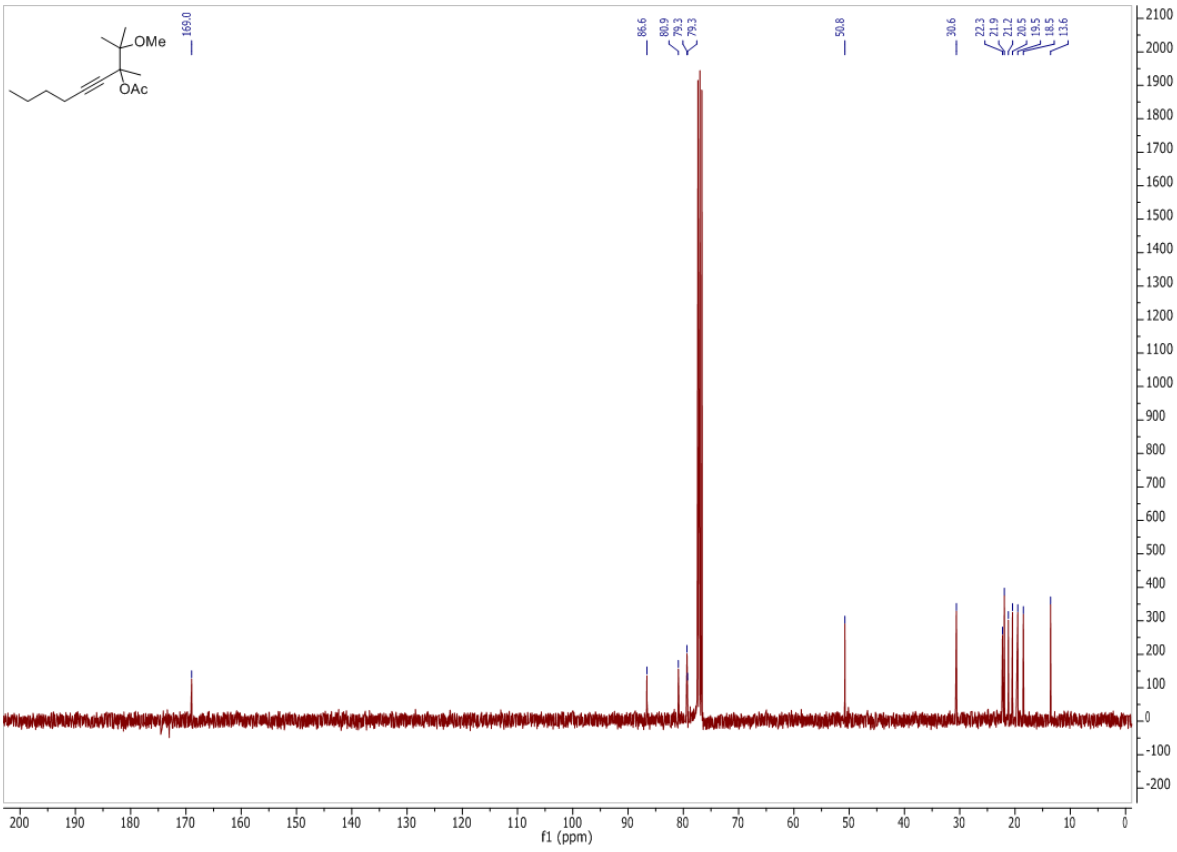


2-Methoxy-2,3-dimethylnon-4-yn-3-yl acetate (9b)

^{1}H NMR (300 K, $CDCl_3$)

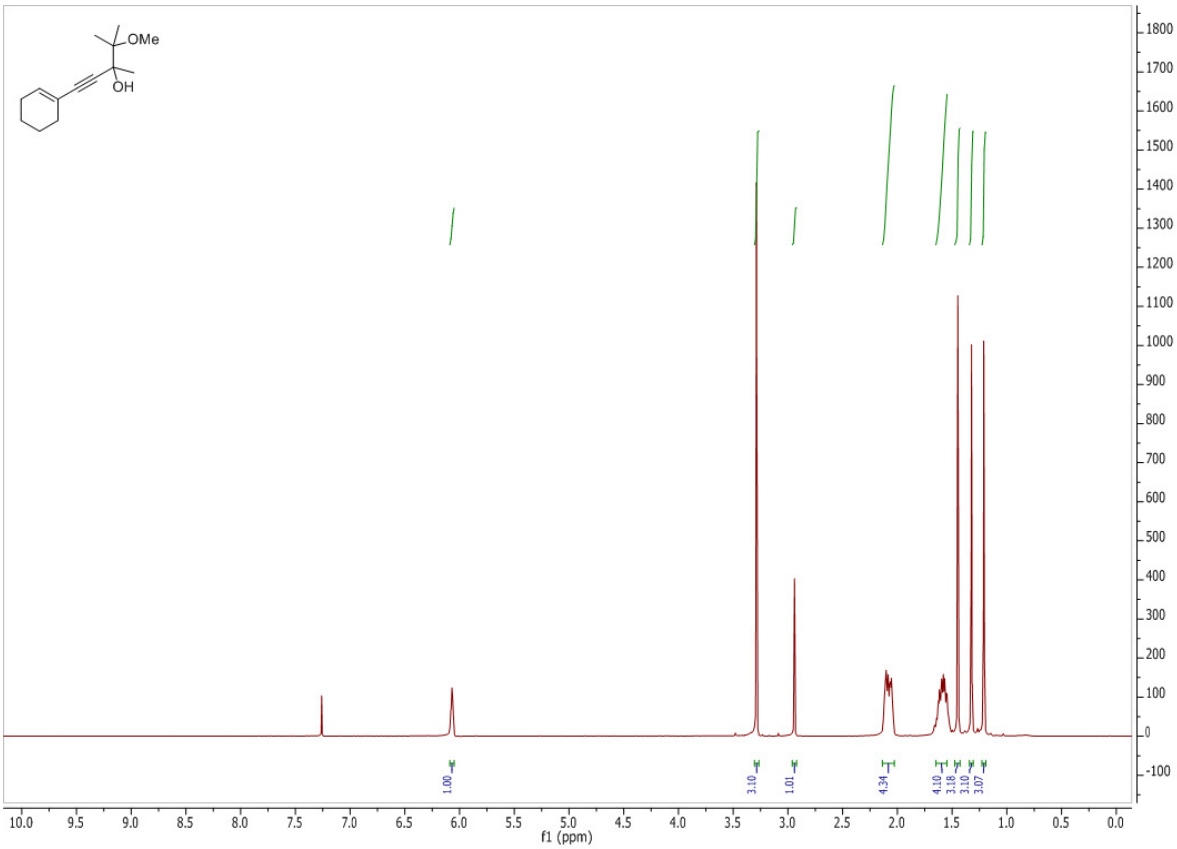


^{13}C NMR (300 K, $CDCl_3$)

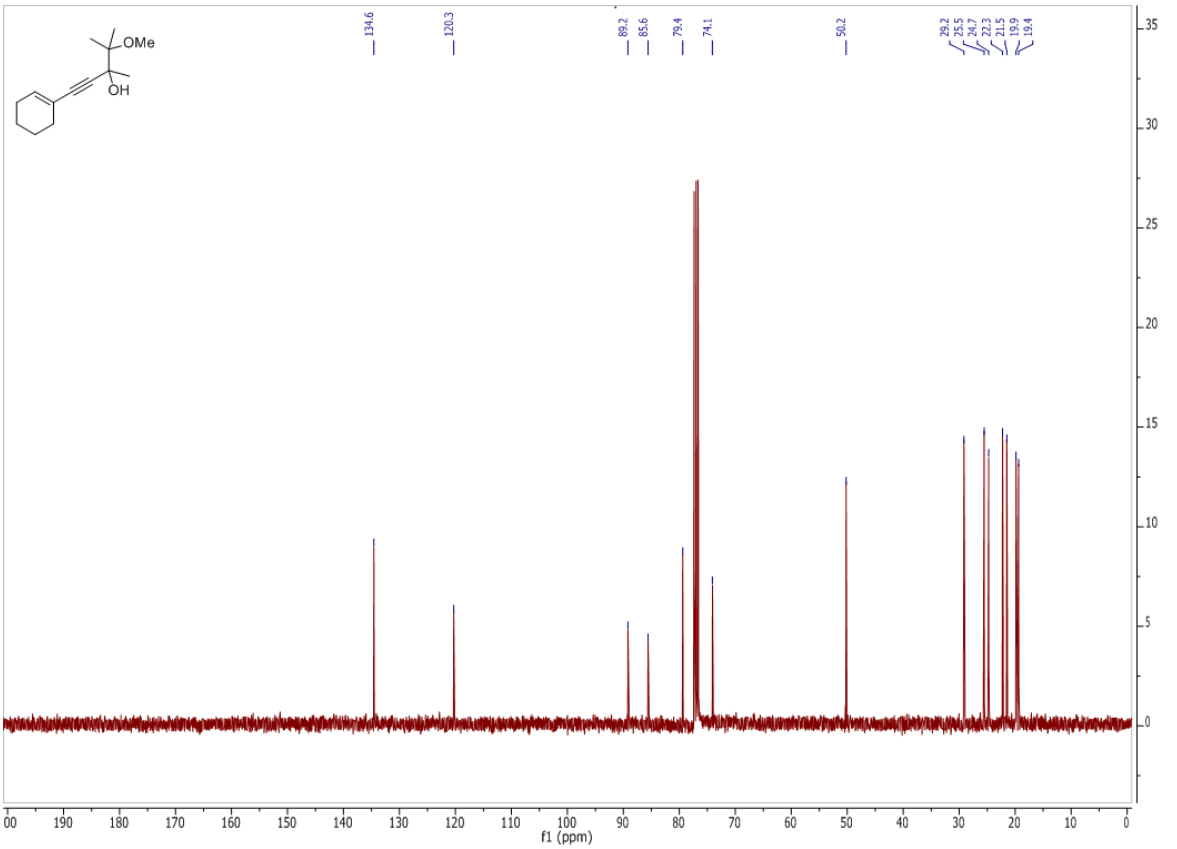


1-(Cyclohex-1-en-1-yl)-4-methoxy-3,4-dimethylpent-1-yn-3-ol (8c)

^{1}H NMR (300 K, $CDCl_3$)

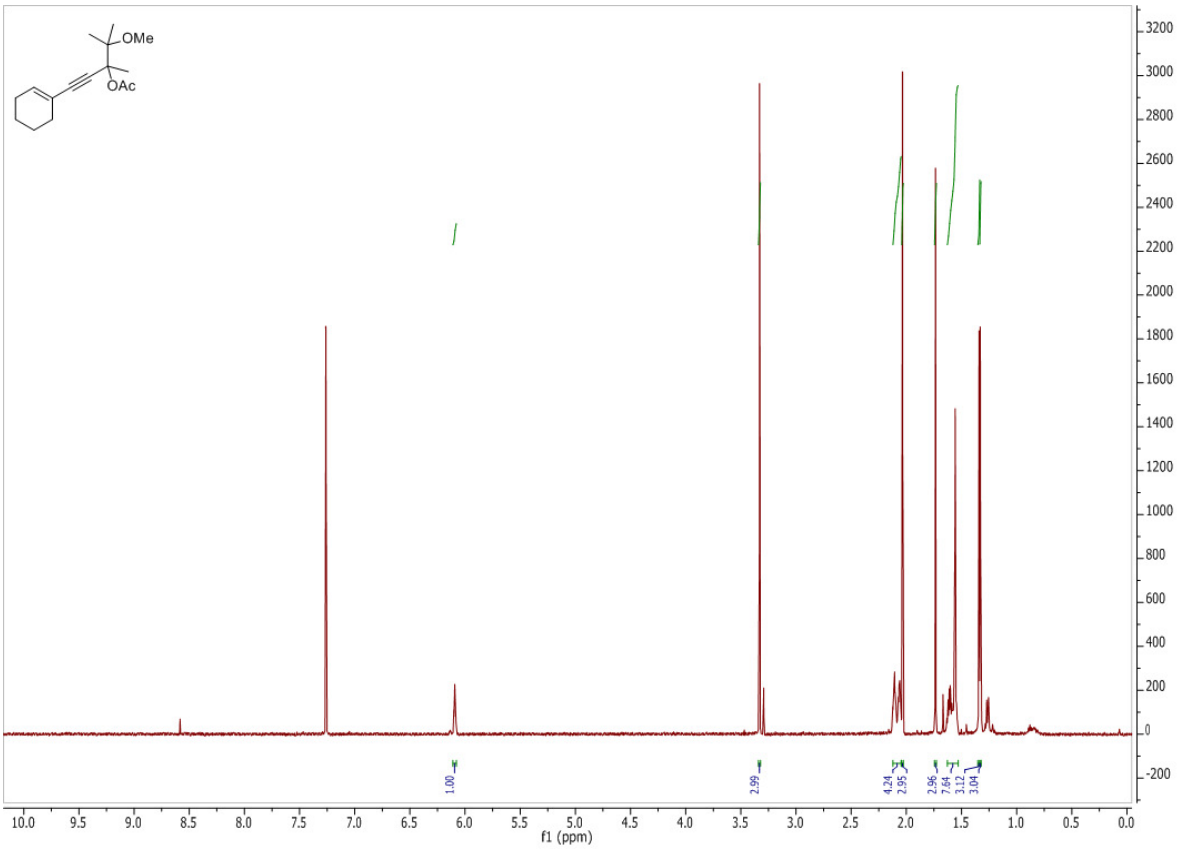


^{13}C NMR (300 K, $CDCl_3$)

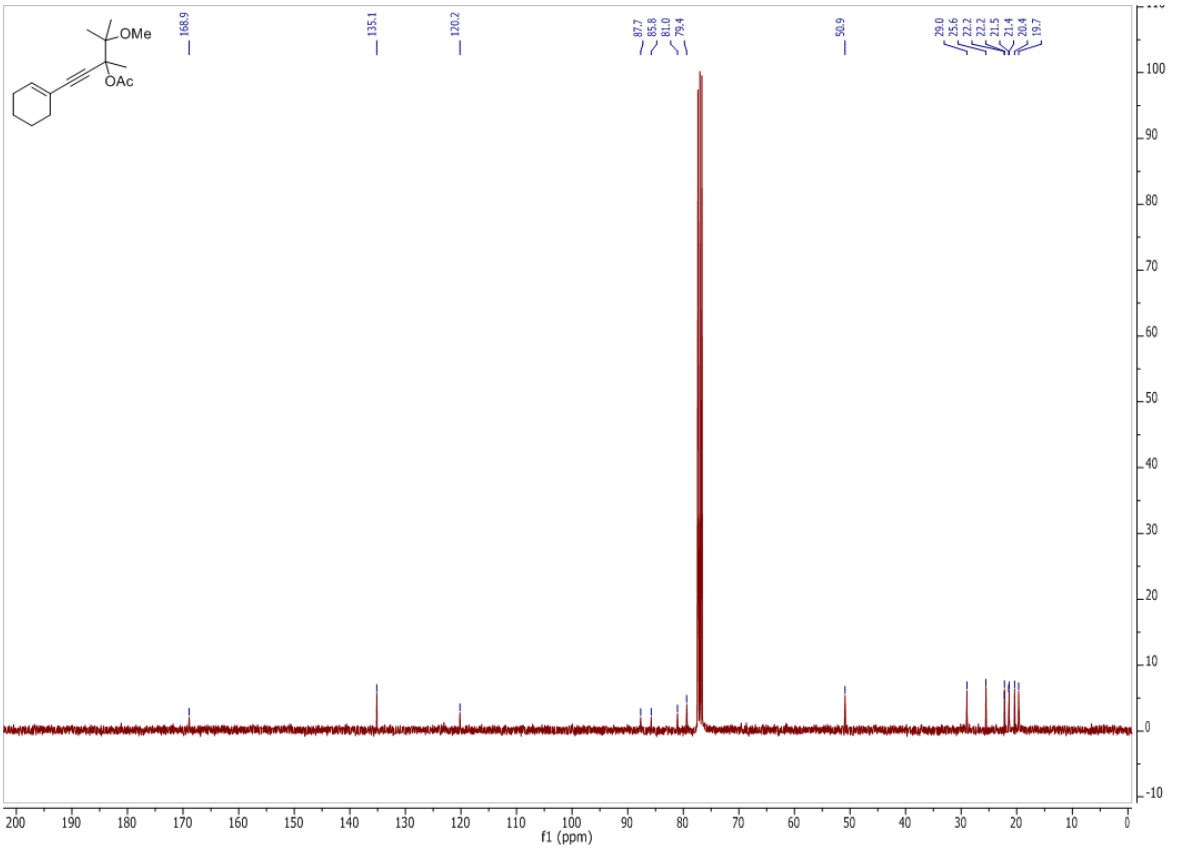


1-(Cyclohex-1-en-1-yl)-4-methoxy-3,4-dimethylpent-1-yn-3-yl acetate (9c)

^{1}H NMR (300 K, $CDCl_3$)

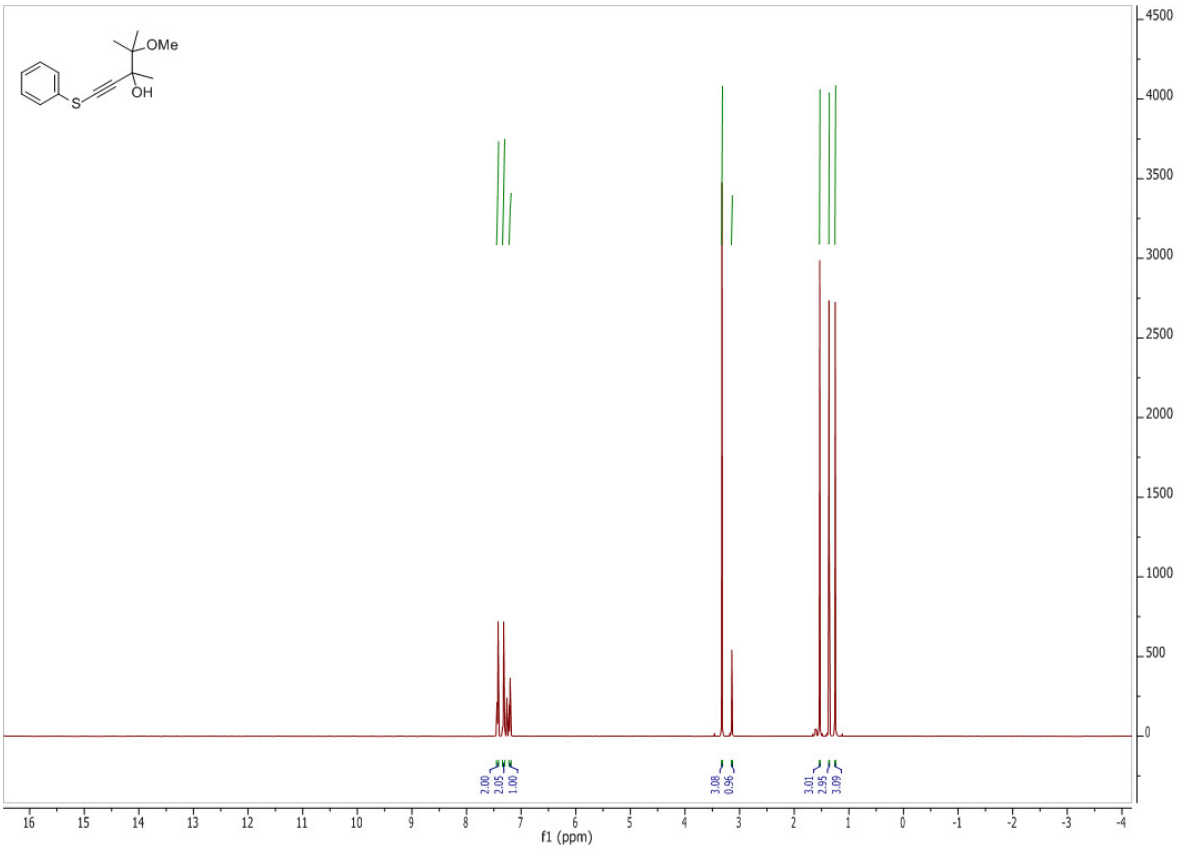


^{13}C NMR (300 K, $CDCl_3$)

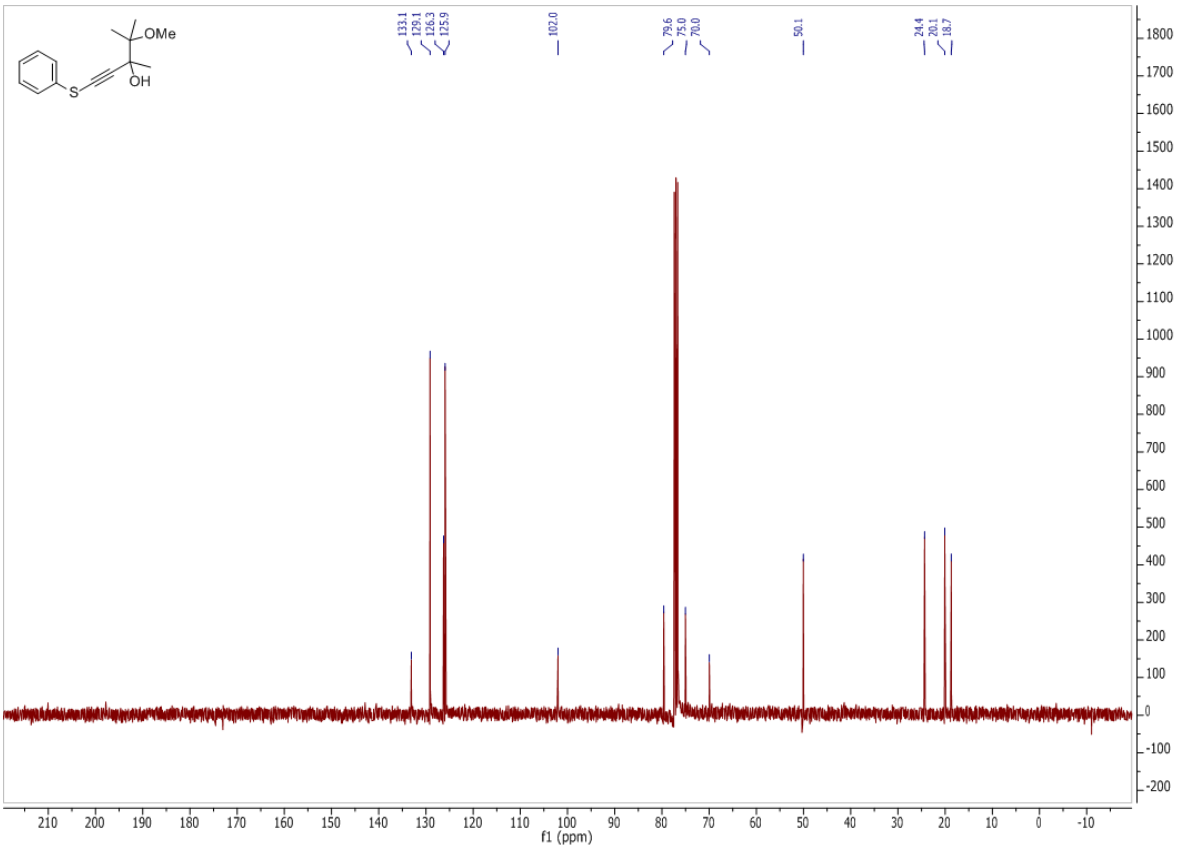


4-Methoxy-3,4-dimethyl-1-(phenylthio)pent-1-yn-3-ol (8d)

^{1}H NMR (300 K, $CDCl_3$)

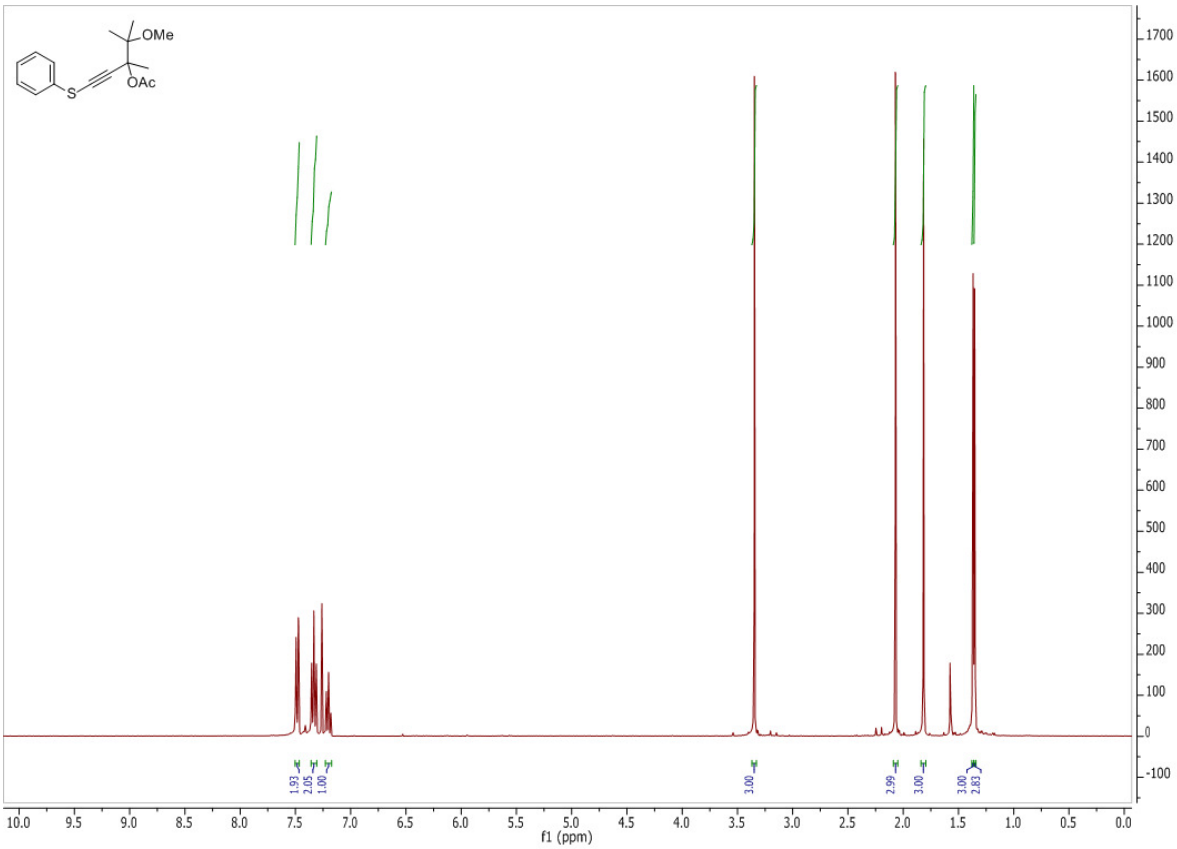


^{13}C NMR (300 K, $CDCl_3$)

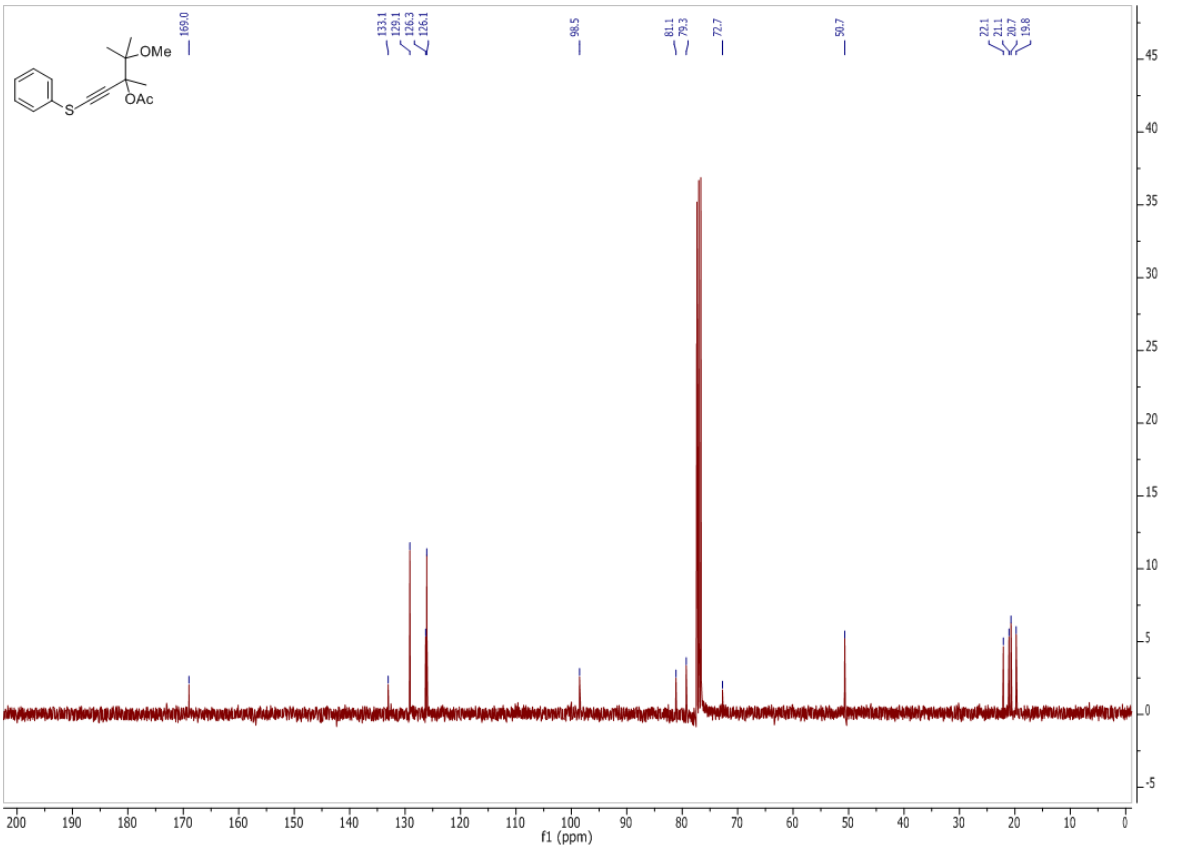


4-Methoxy-3,4-dimethyl-1-(phenylthio)pent-1-yn-3-yl acetate (9d)

^{1}H NMR (300 K, $CDCl_3$)

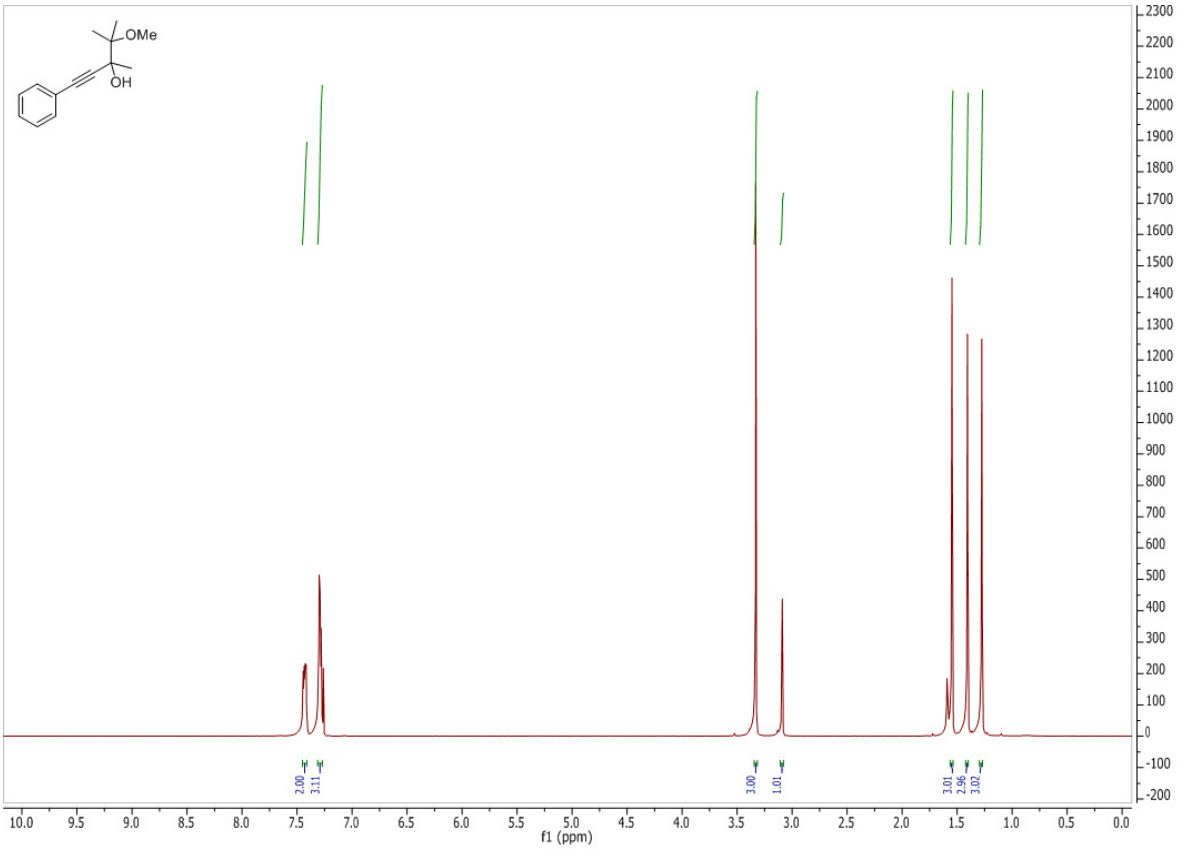


^{13}C NMR (300 K, $CDCl_3$)

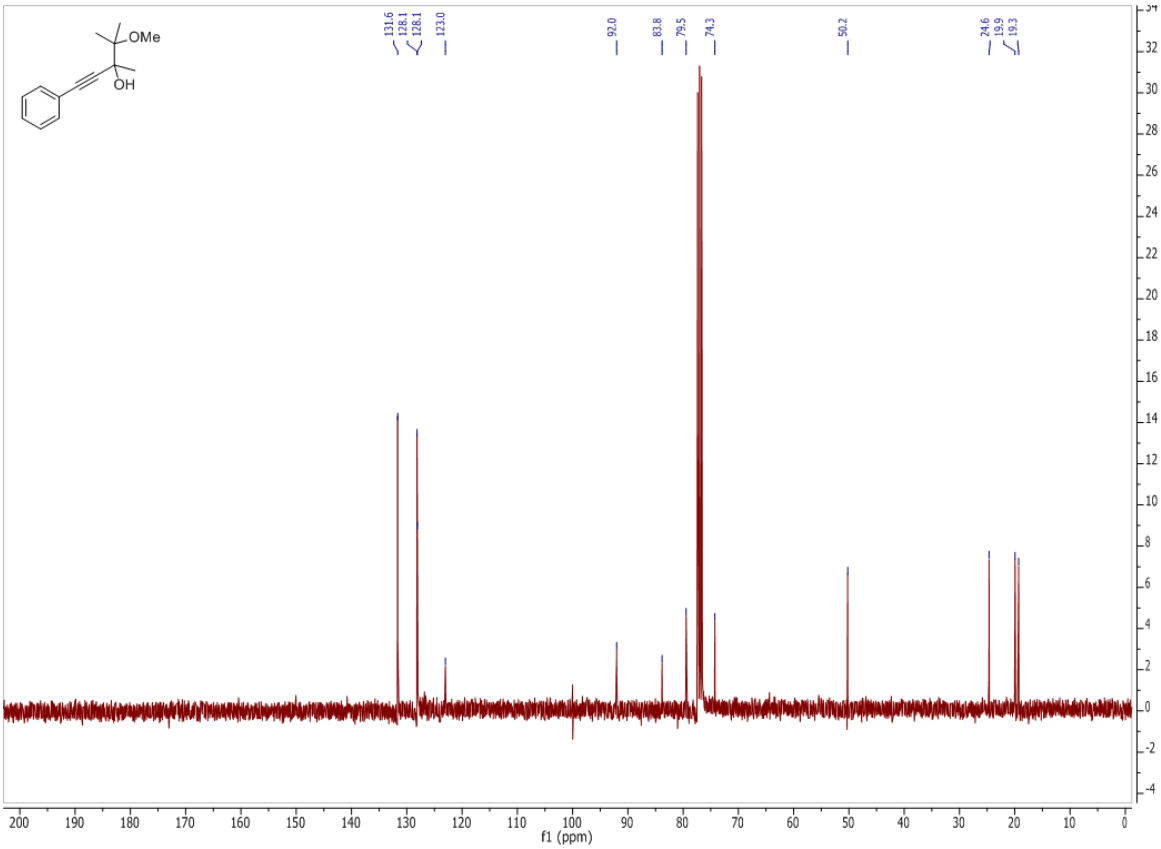


4-Methoxy-3,4-dimethyl-1-phenylpent-1-yn-3-ol (8e)

^{1}H NMR (300 K, $CDCl_3$)

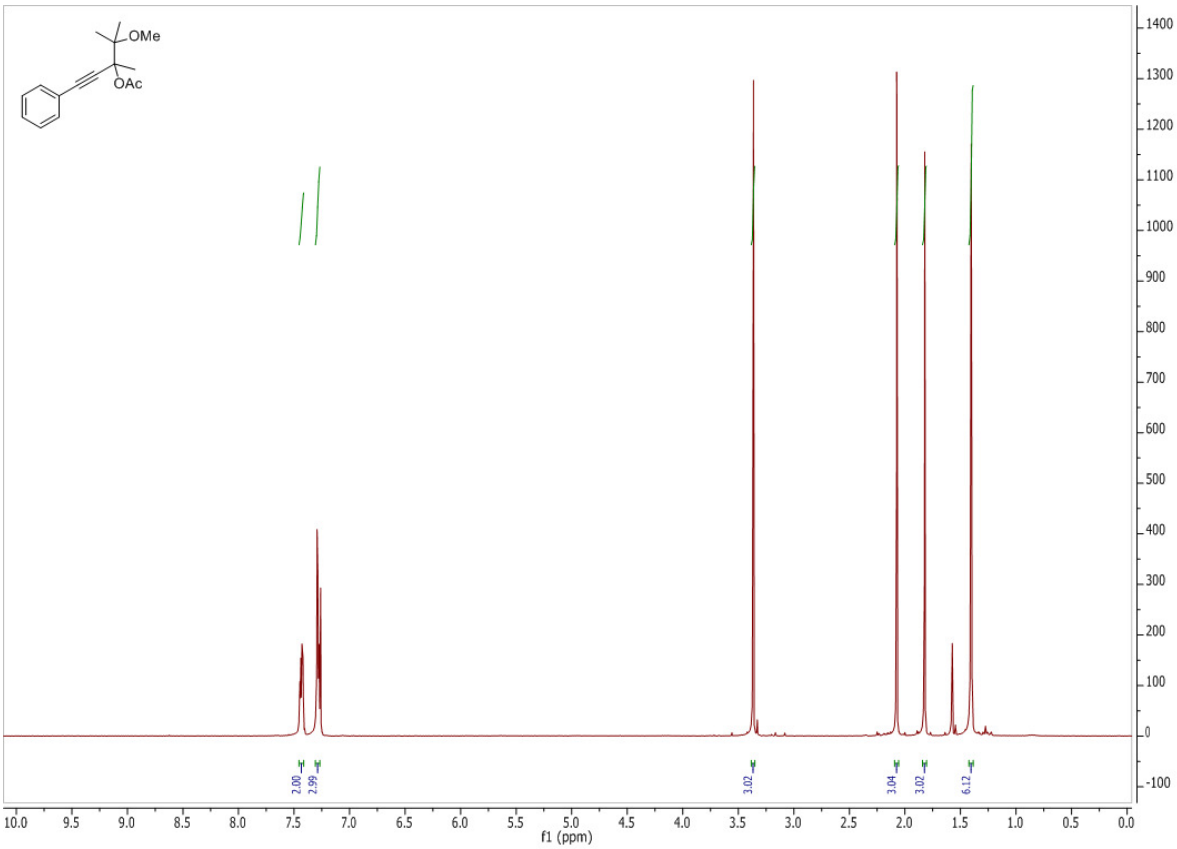


^{13}C NMR (300 K, $CDCl_3$)

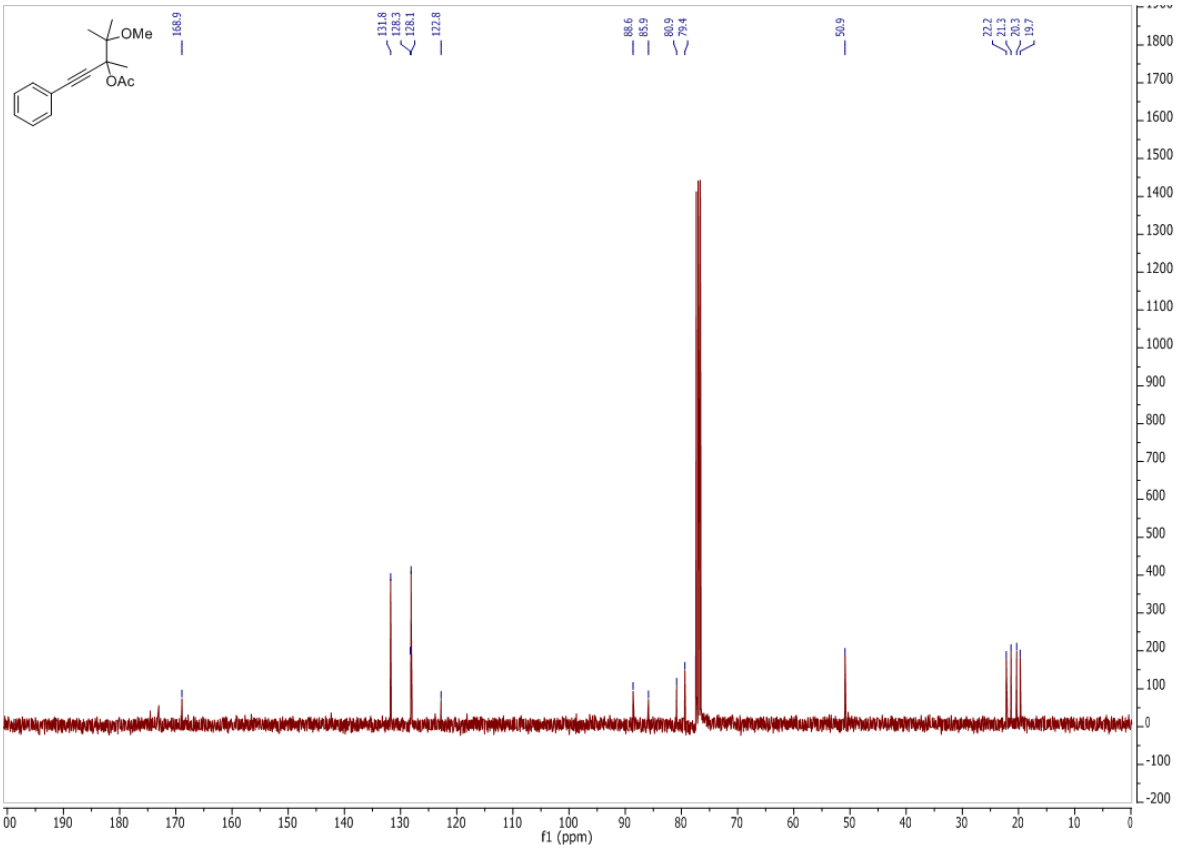


4-Methoxy-3,4-dimethyl-1-phenylpent-1-yn-3-yl acetate (9e)

^{1}H NMR (300 K, $CDCl_3$)

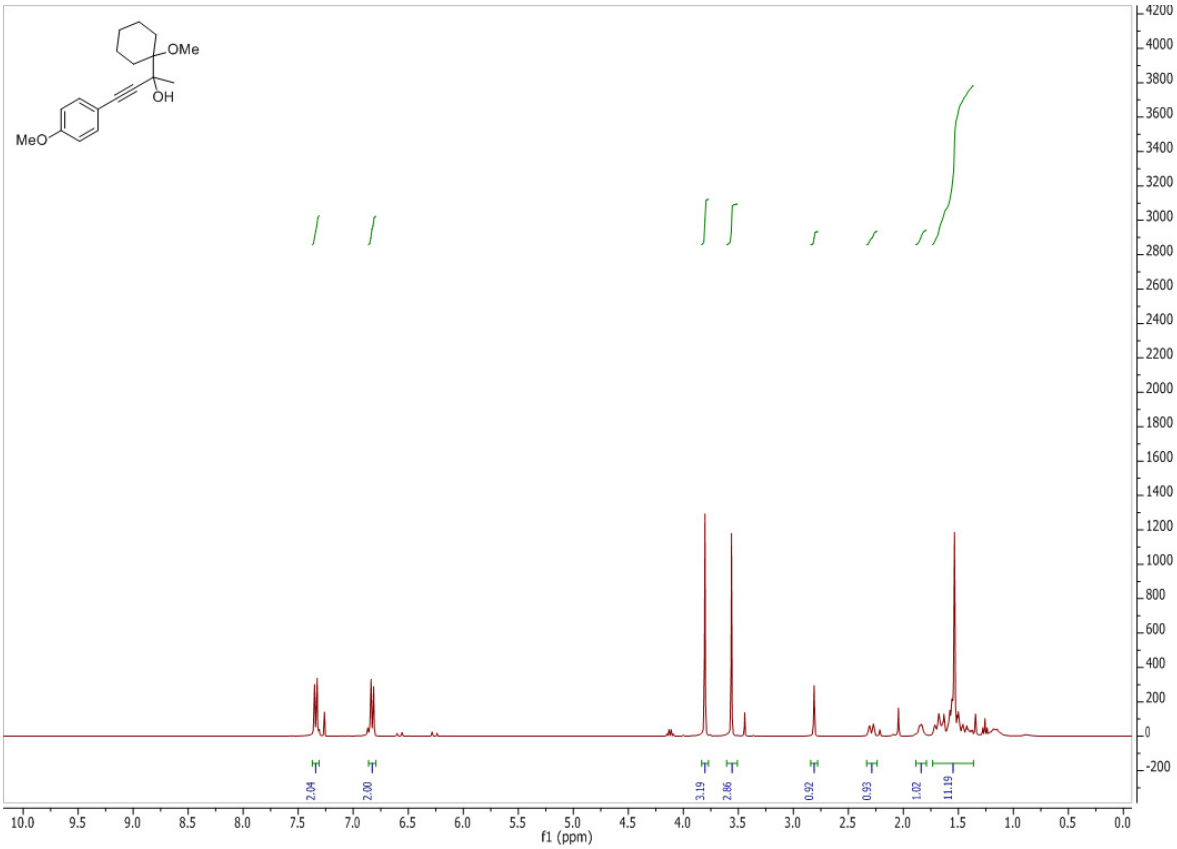


^{13}C NMR (300 K, $CDCl_3$)

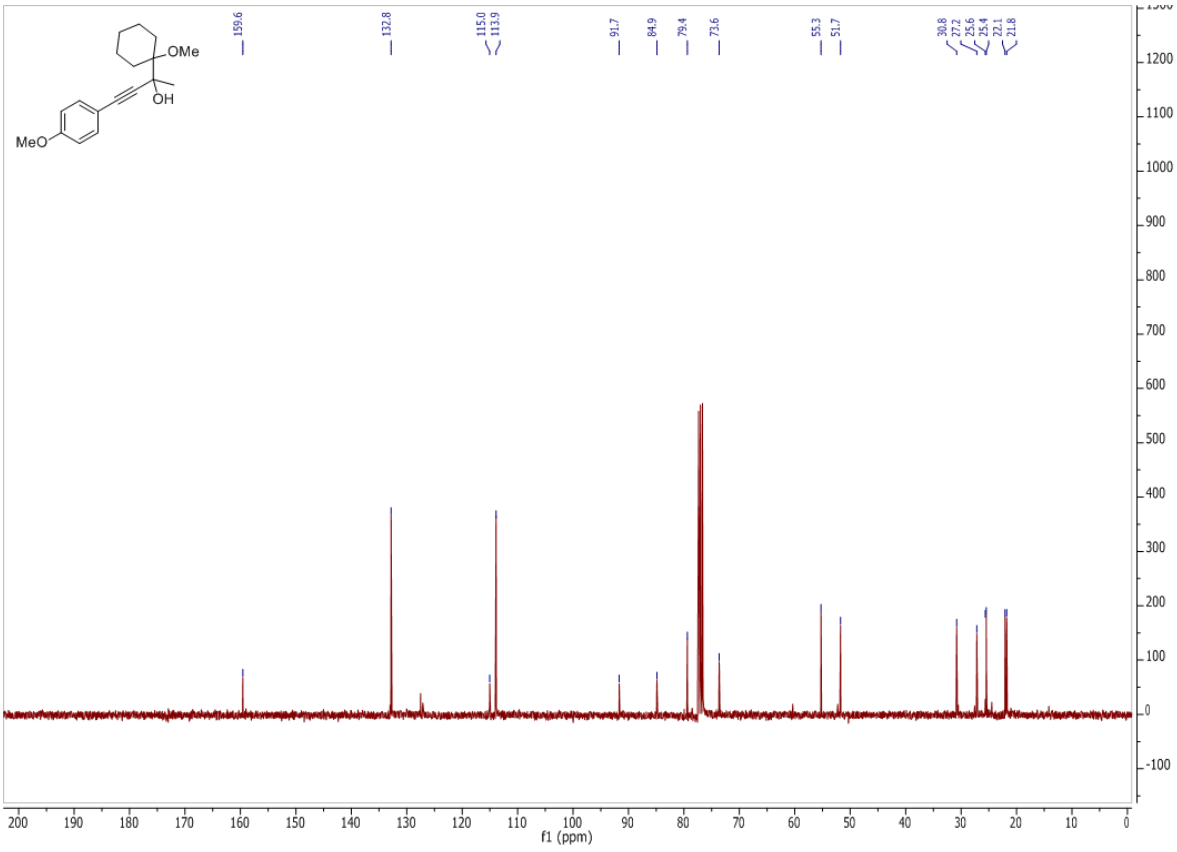


2-(1-Methoxycyclohexyl)-4-(4-methoxyphenyl)but-3-yn-2-ol (8f)

^{1}H NMR (300 K, $CDCl_3$)

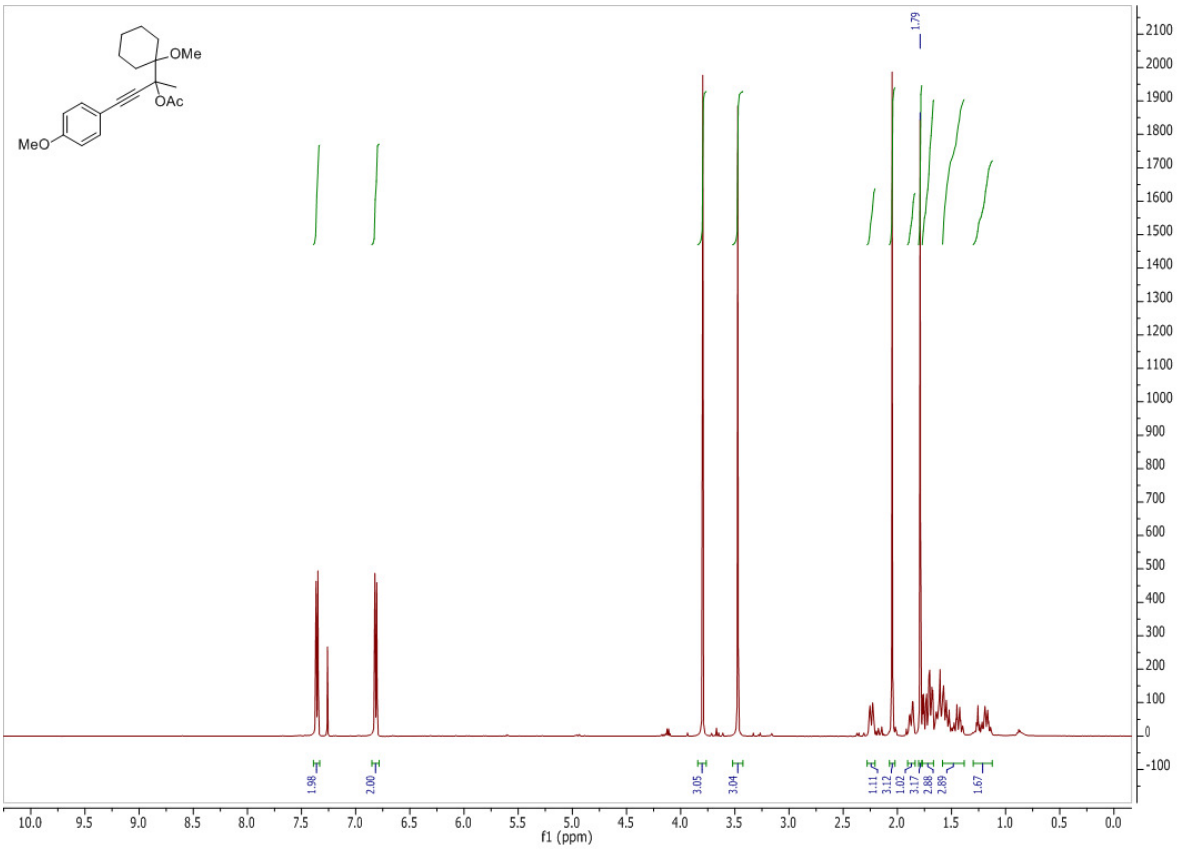


^{13}C NMR (300 K, $CDCl_3$)

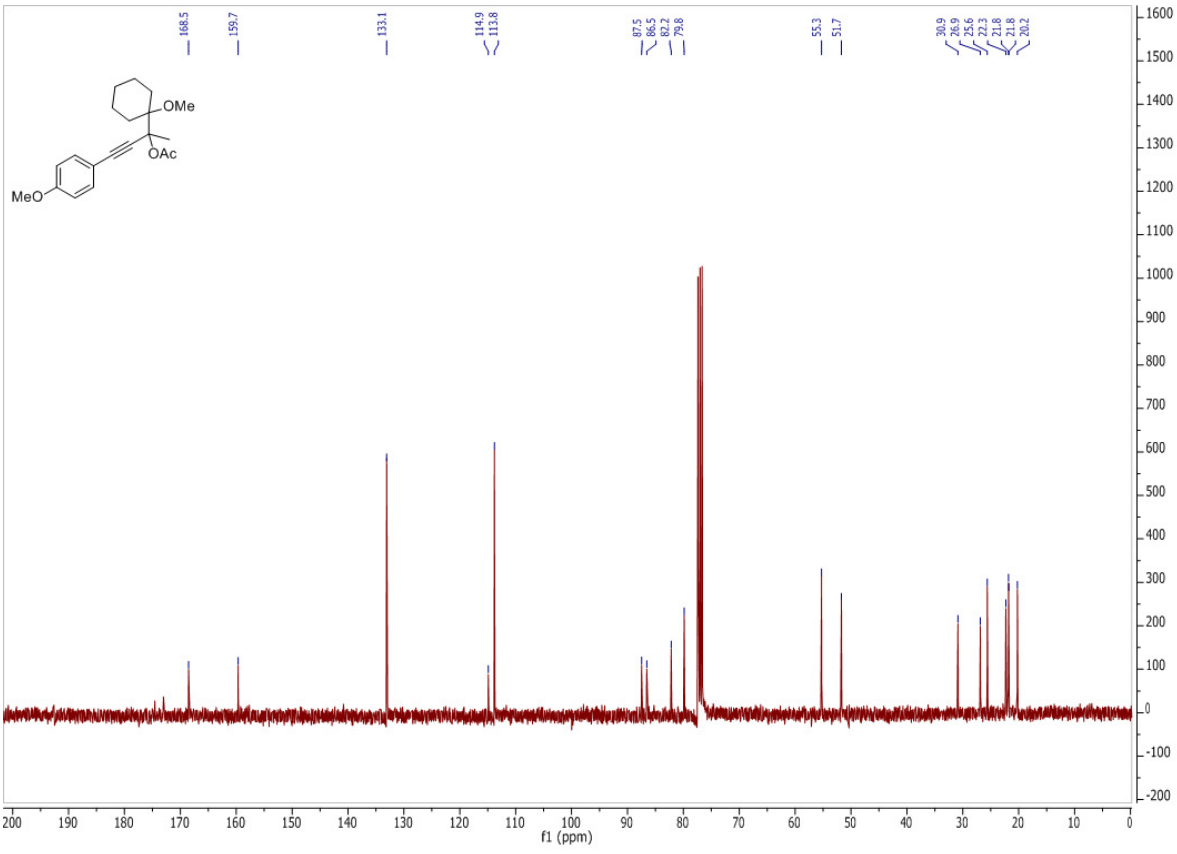


2-(1-Methoxycyclohexyl)-4-(4-methoxyphenyl)but-3-yn-2-yl acetate (9f)

^{1}H NMR (300 K, $CDCl_3$)

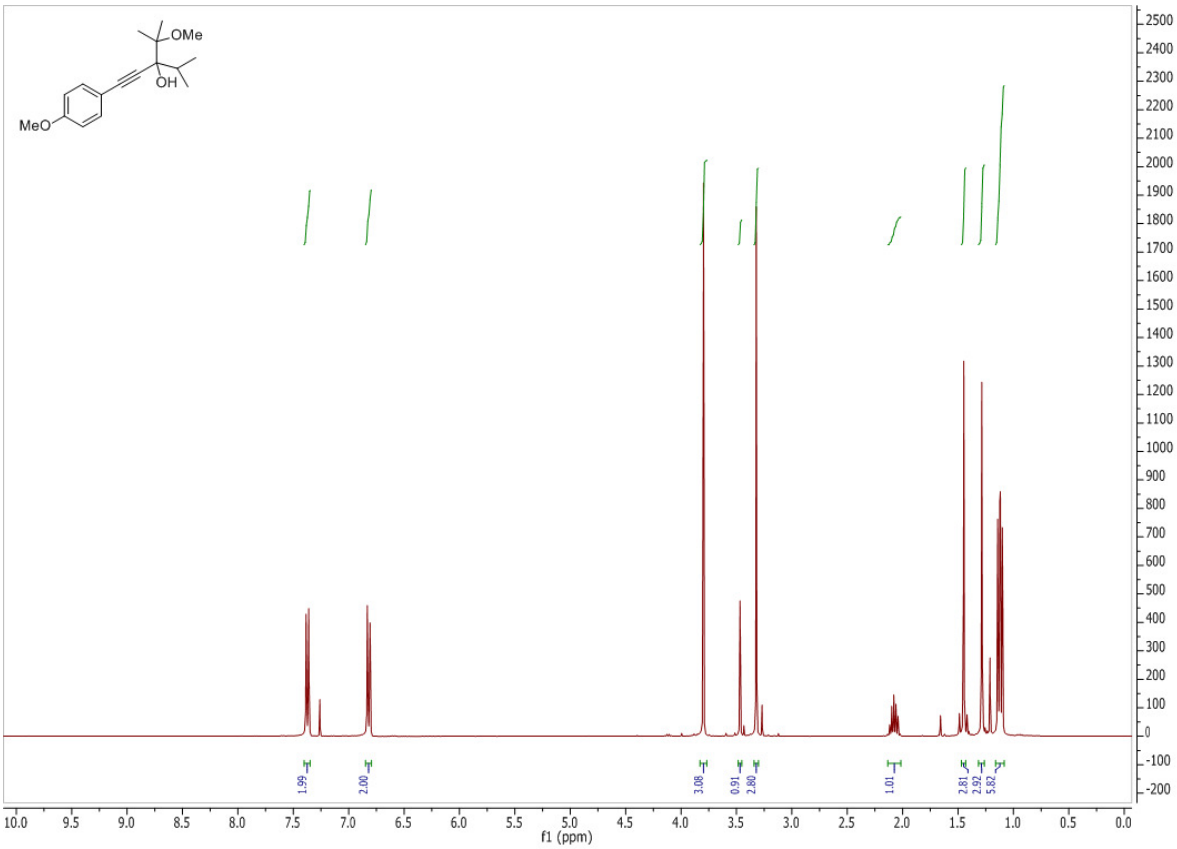


^{13}C NMR (300 K, $CDCl_3$)

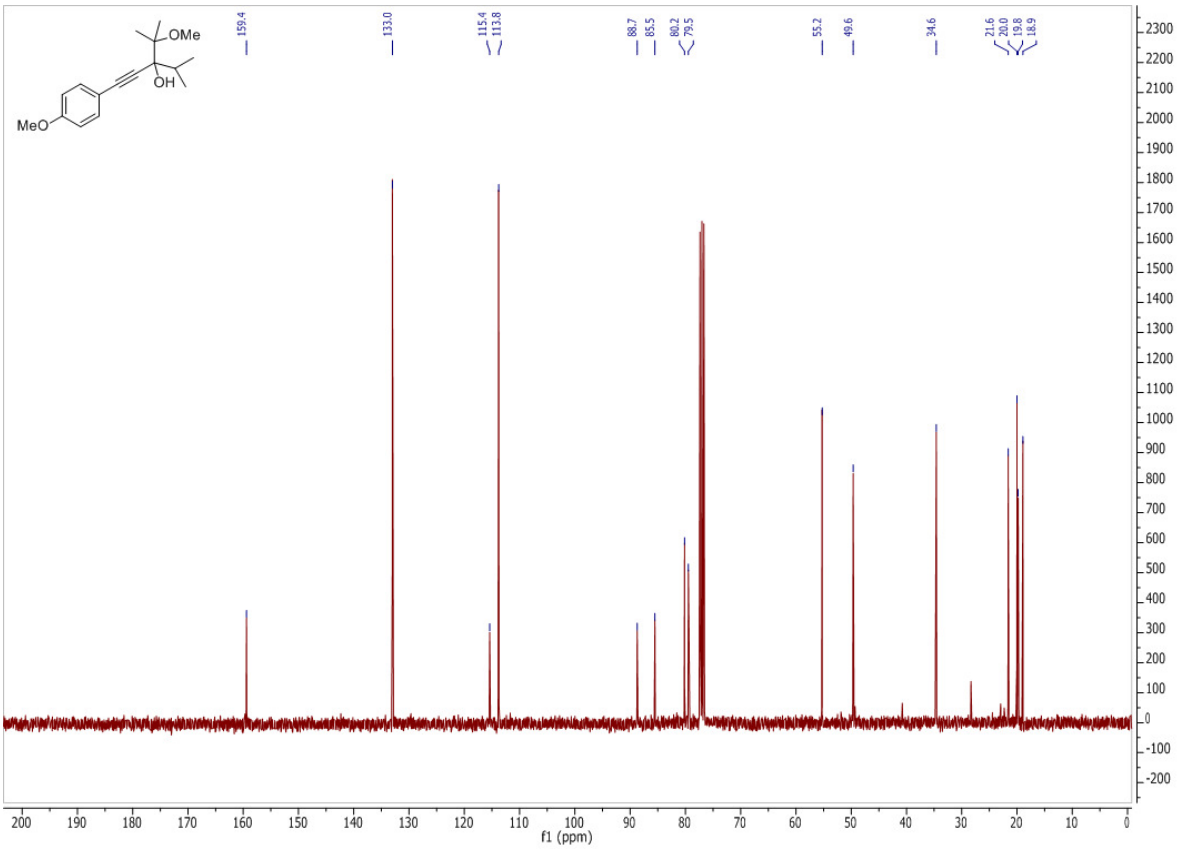


3-Isopropyl-4-methoxy-1-(4-methoxyphenyl)-4-methylpent-1-yn-3-ol (8g)

^{1}H NMR (300 K, $CDCl_3$)

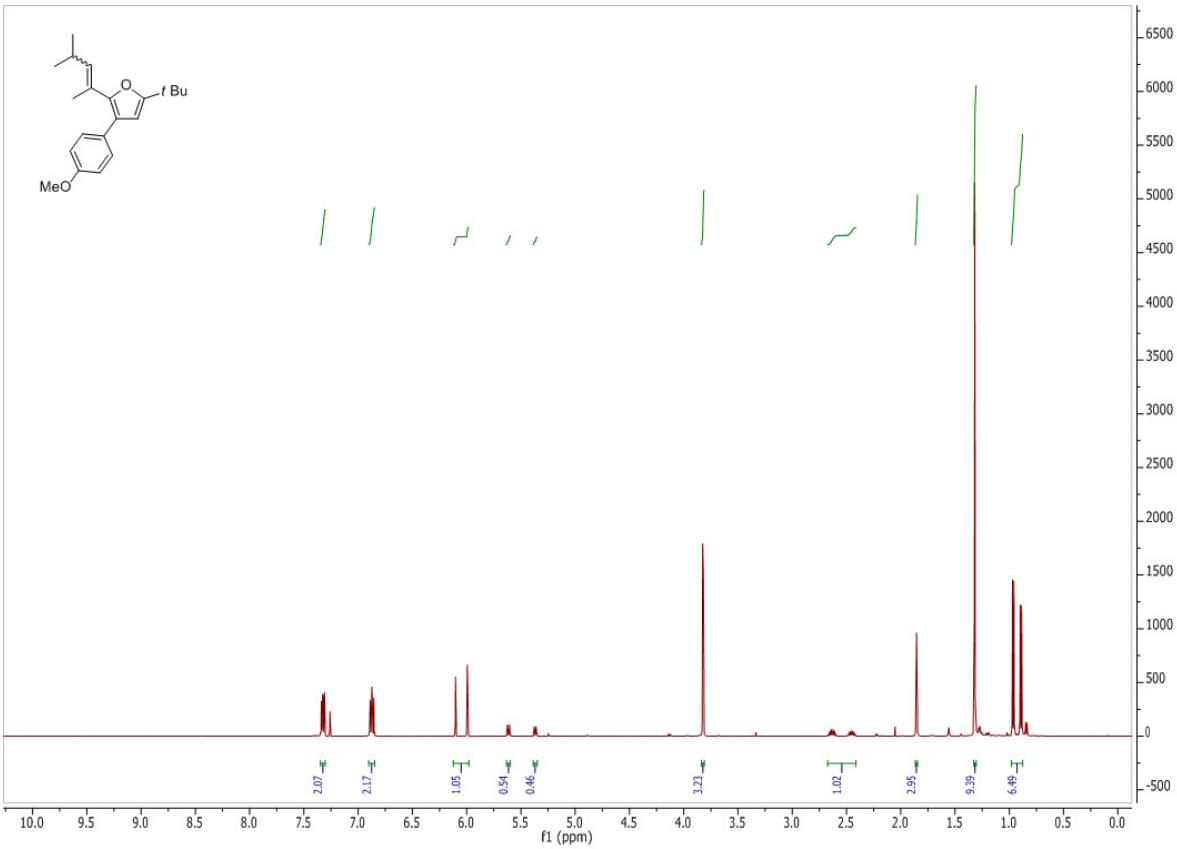


^{13}C NMR (300 K, $CDCl_3$)

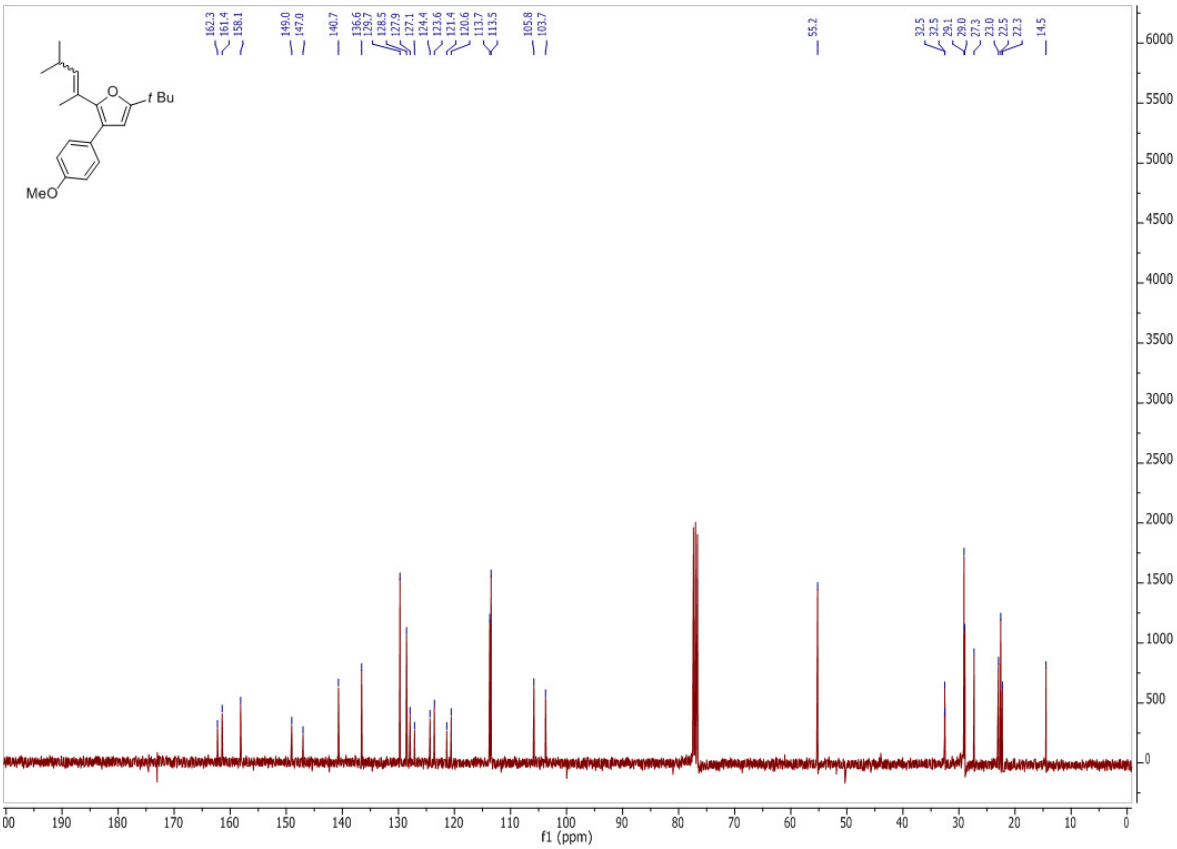


5-(*tert*-Butyl)-3-(4-methoxyphenyl)-2-(4-methylpent-2-en-2-yl)furan (2)

^{1}H NMR (300 K, $CDCl_3$)

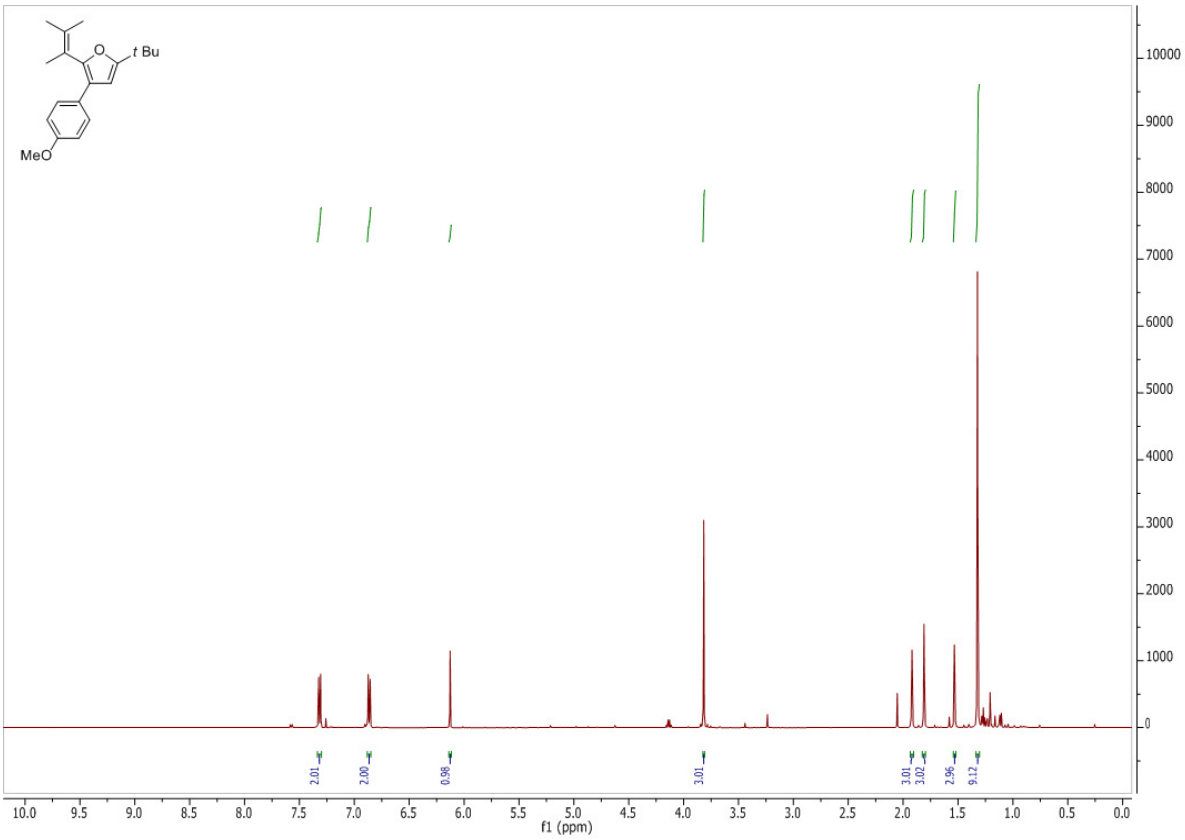


^{13}C NMR (300 K, $CDCl_3$)

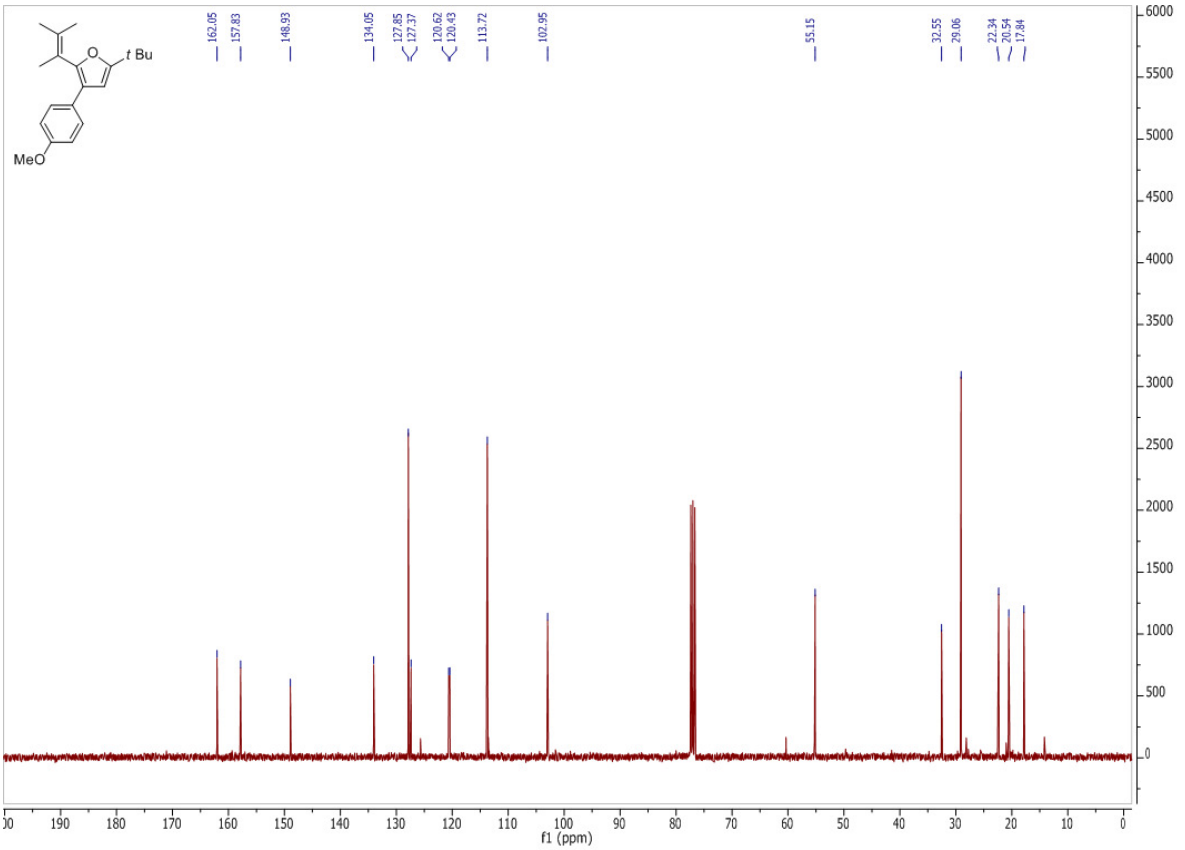


5-(*tert*-Butyl)-3-(4-methoxyphenyl)-2-(3-methylbut-2-en-2-yl)furan (6a)

^{1}H NMR (300 K, $CDCl_3$)

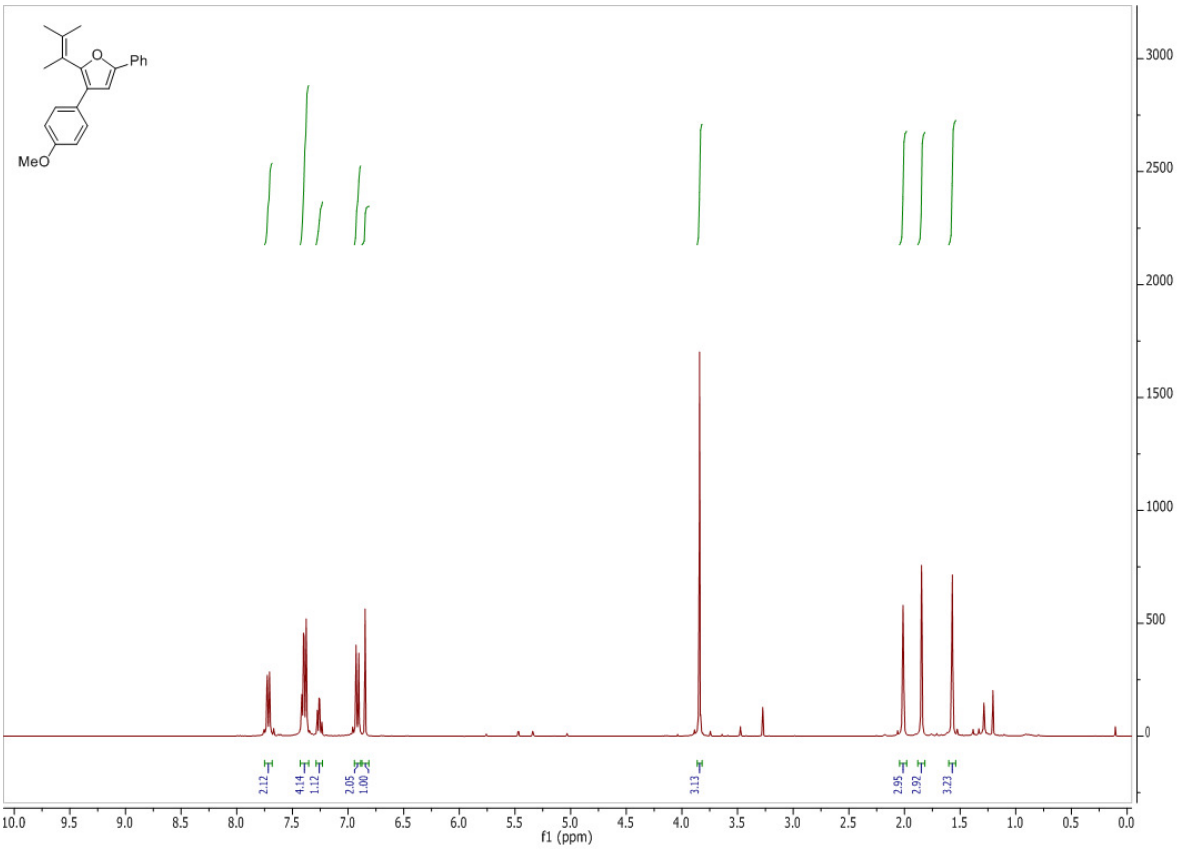


^{13}C NMR (300 K, $CDCl_3$)

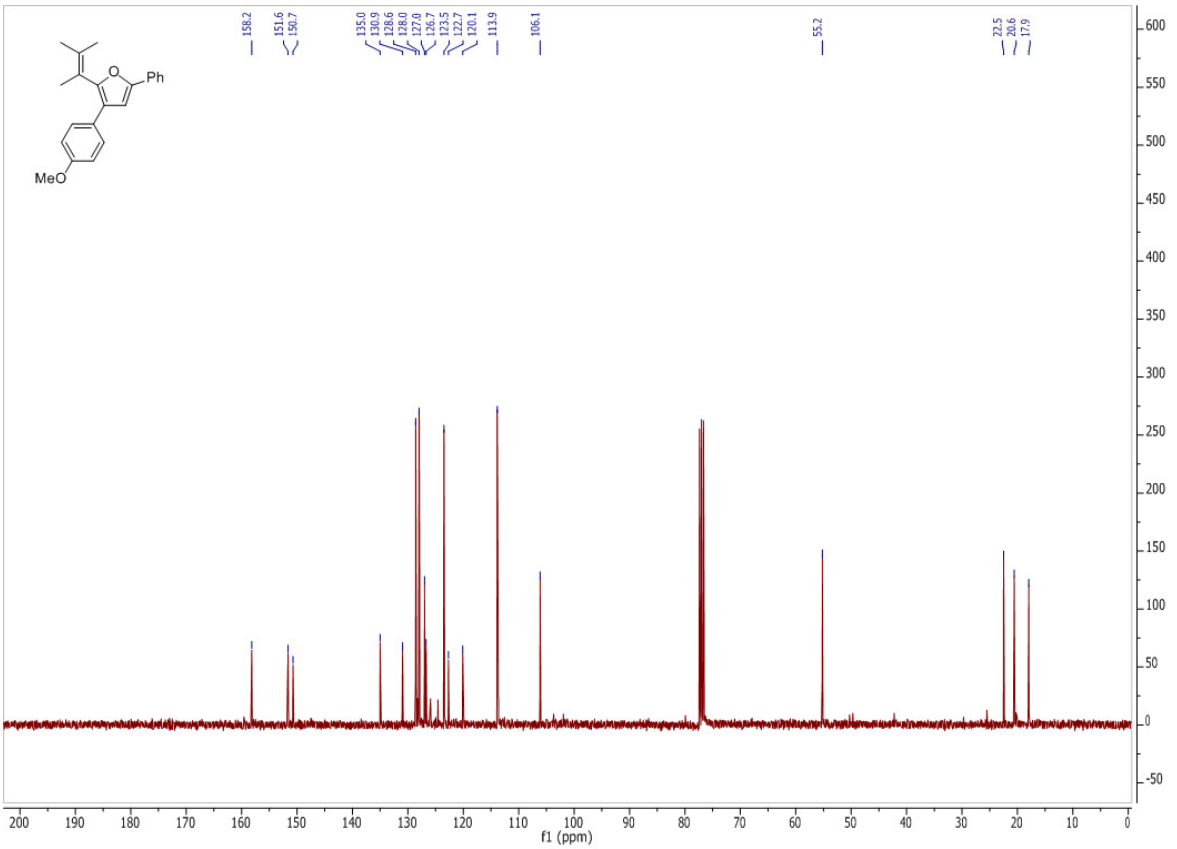


3-(4-Methoxyphenyl)-2-(3-methylbut-2-en-2-yl)-5-phenylfuran (6b)

^{1}H NMR (300 K, $CDCl_3$)

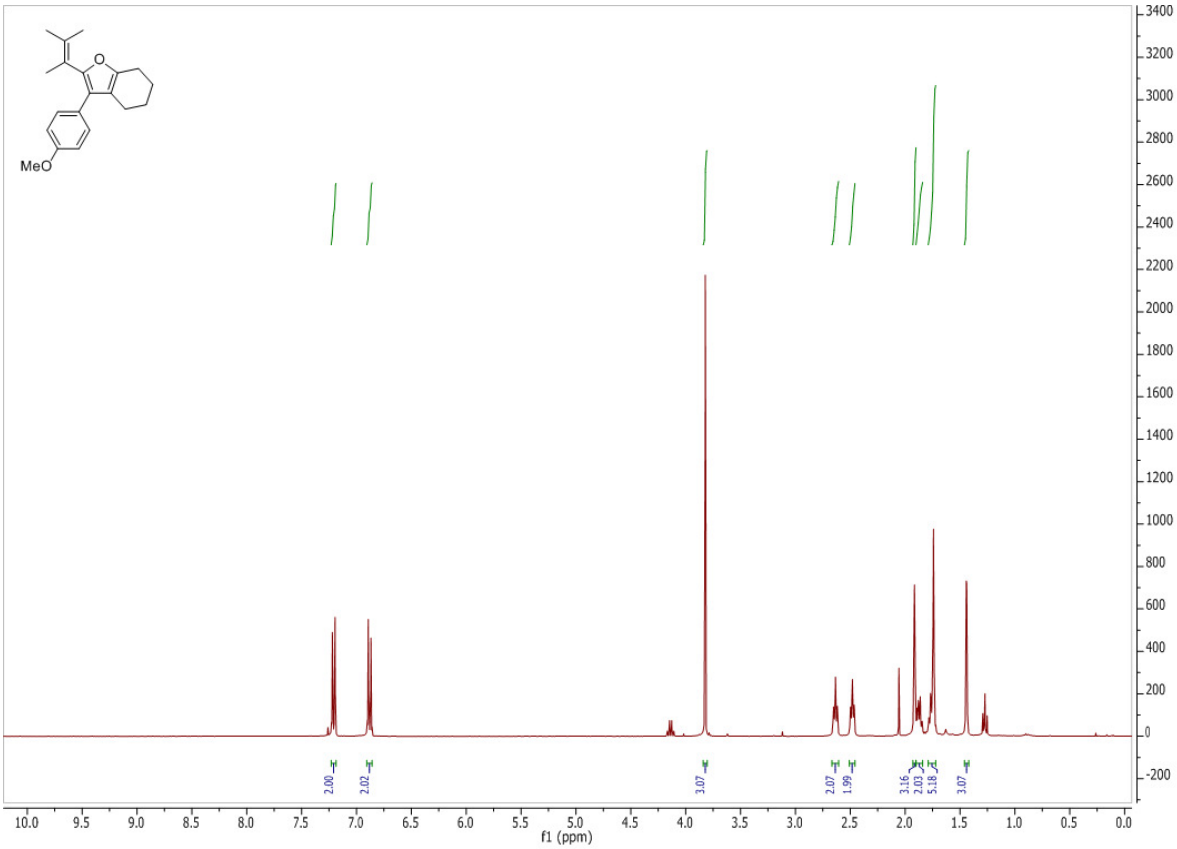


^{13}C NMR (300 K, $CDCl_3$)

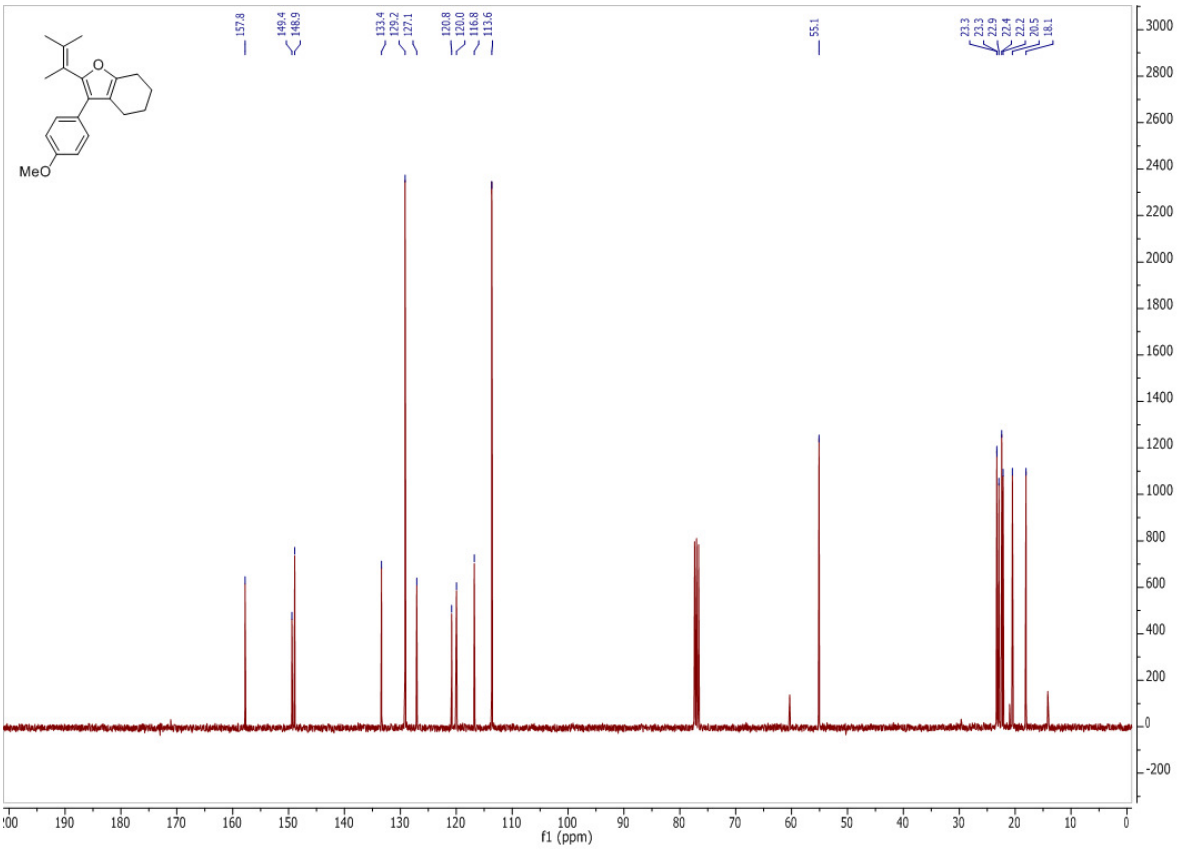


3-(4-Methoxyphenyl)-2-(3-methylbut-2-en-2-yl)-4,5,6,7-tetrahydrobenzofuran (6c)

^{1}H NMR (300 K, $CDCl_3$)

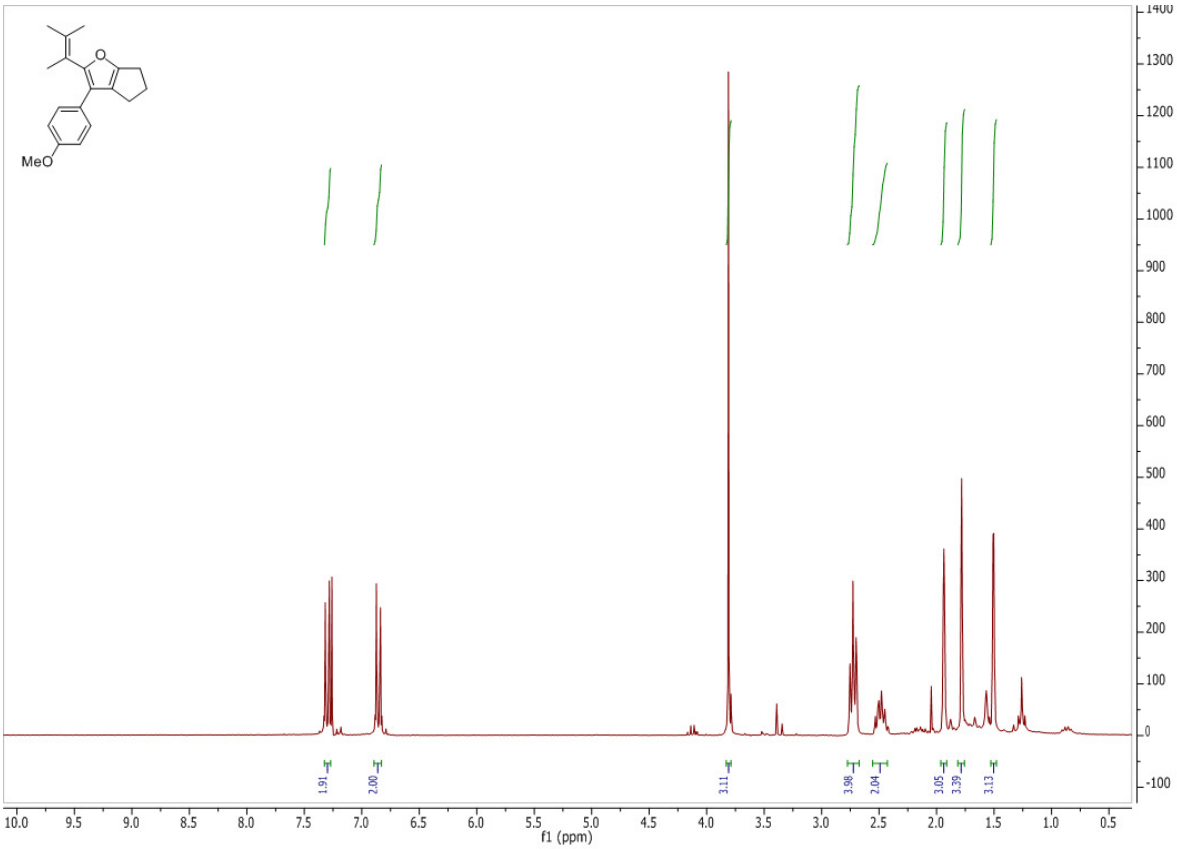


^{13}C NMR (300 K, $CDCl_3$)

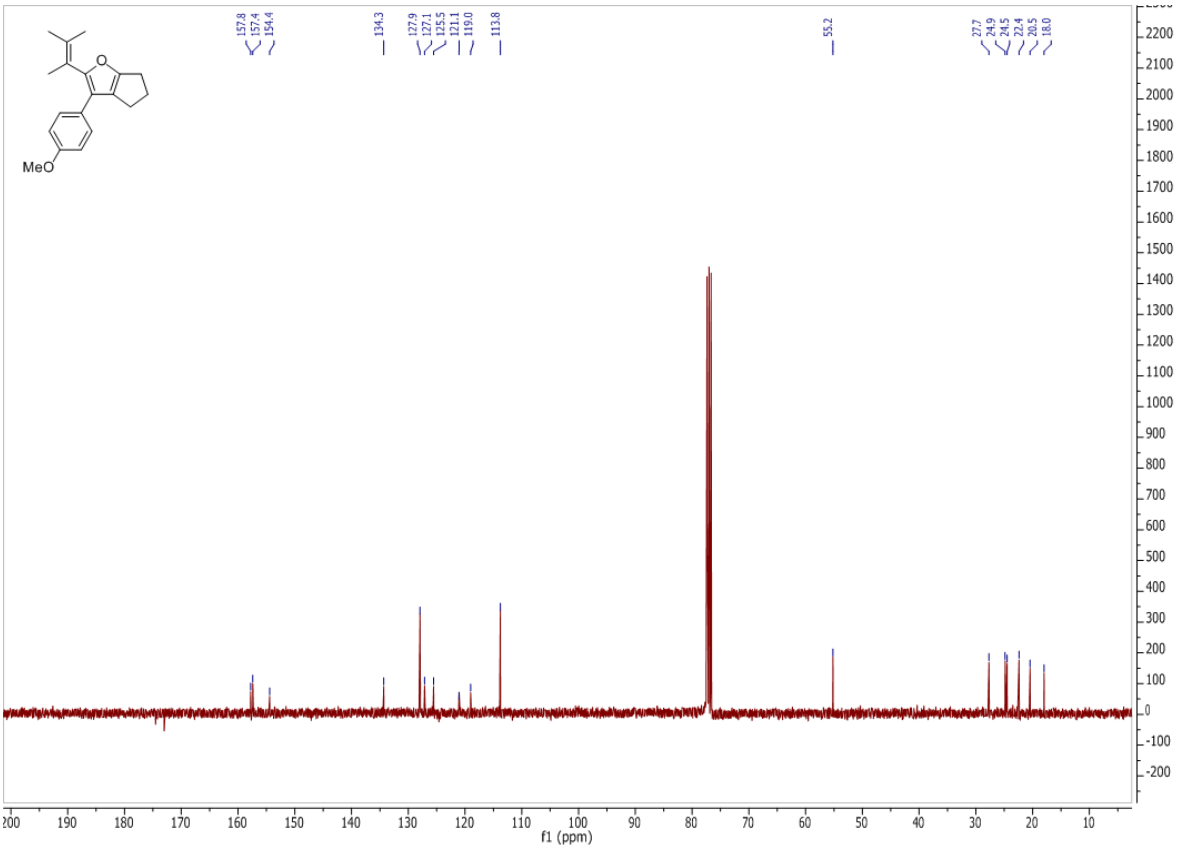


3-(4-Methoxyphenyl)-2-(3-methylbut-2-en-2-yl)-5,6-dihydro-4H-cyclopenta[b]furan (6d)

^{1}H NMR (300 K, $CDCl_3$)

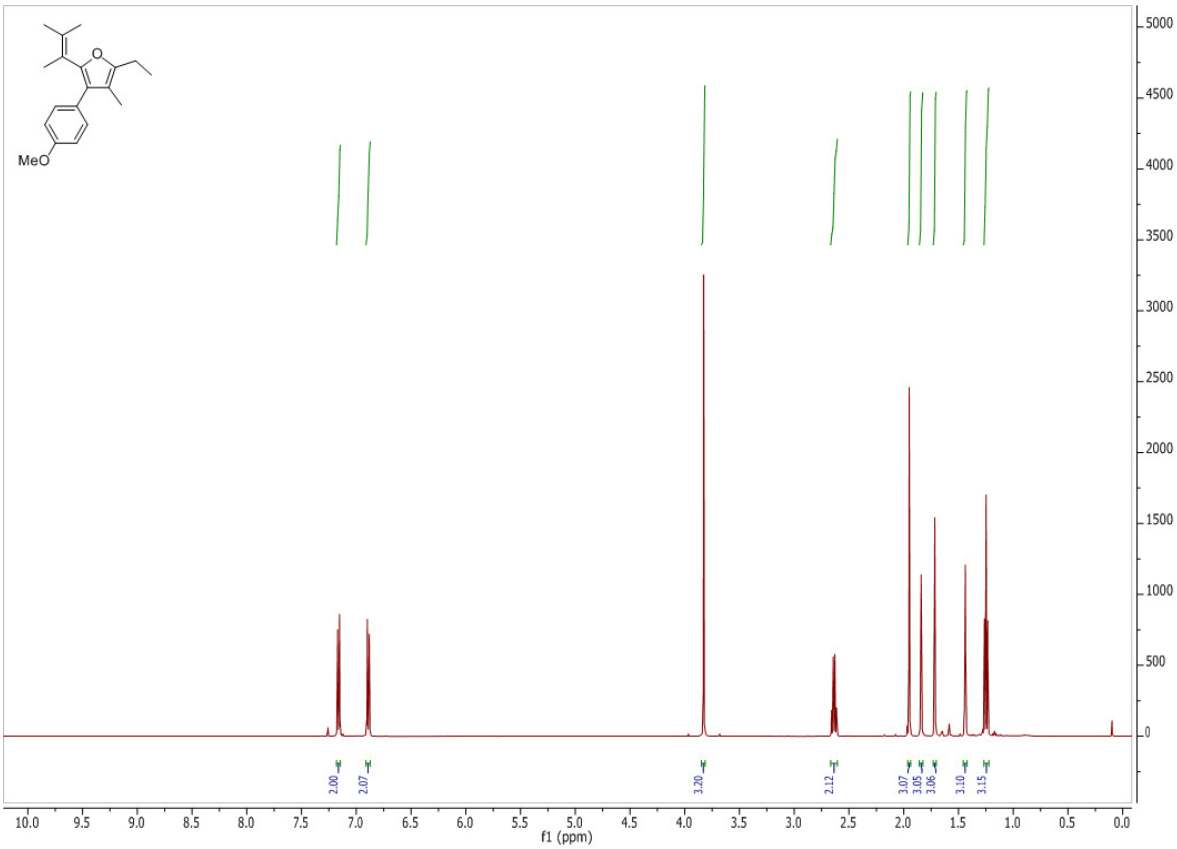


^{13}C NMR (300 K, $CDCl_3$)

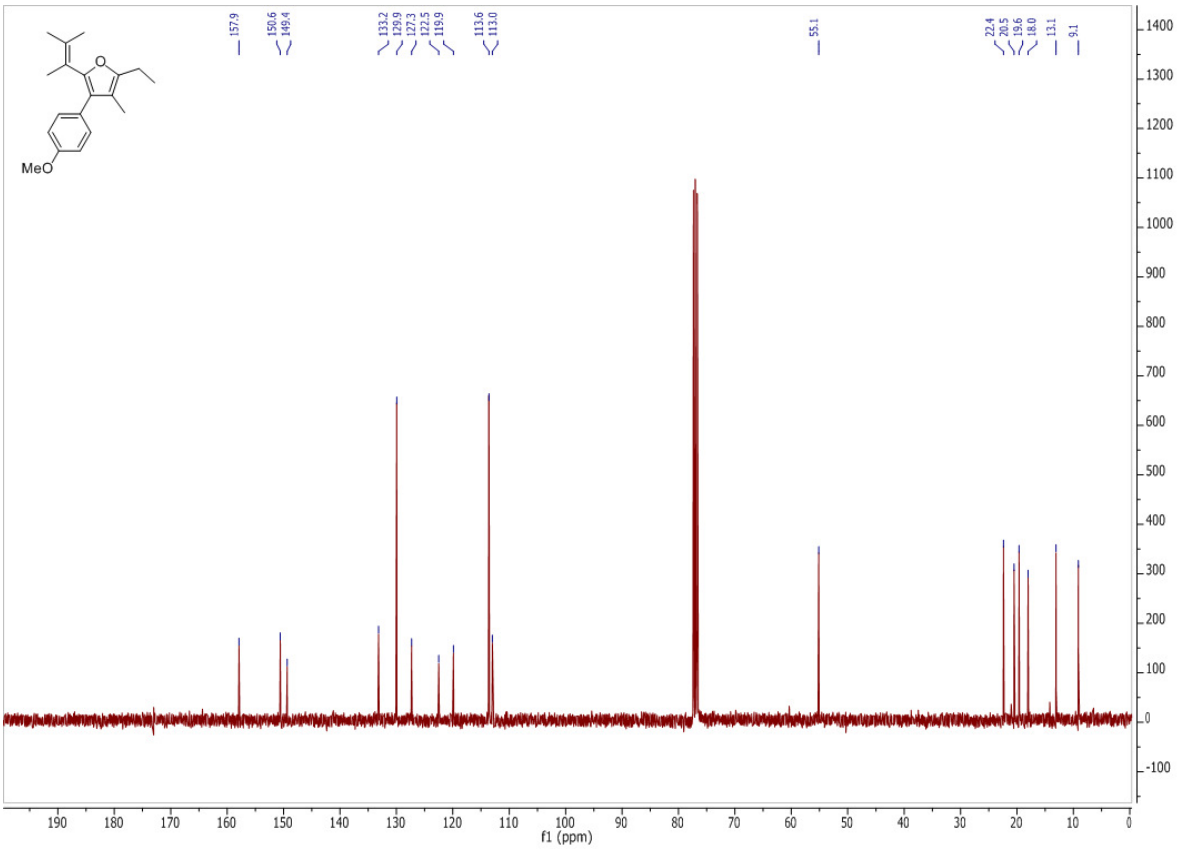


2-Ethyl-4-(4-methoxyphenyl)-3-methyl-5-(3-methylbut-2-en-2-yl)furan (6e)

^{1}H NMR (300 K, $CDCl_3$)

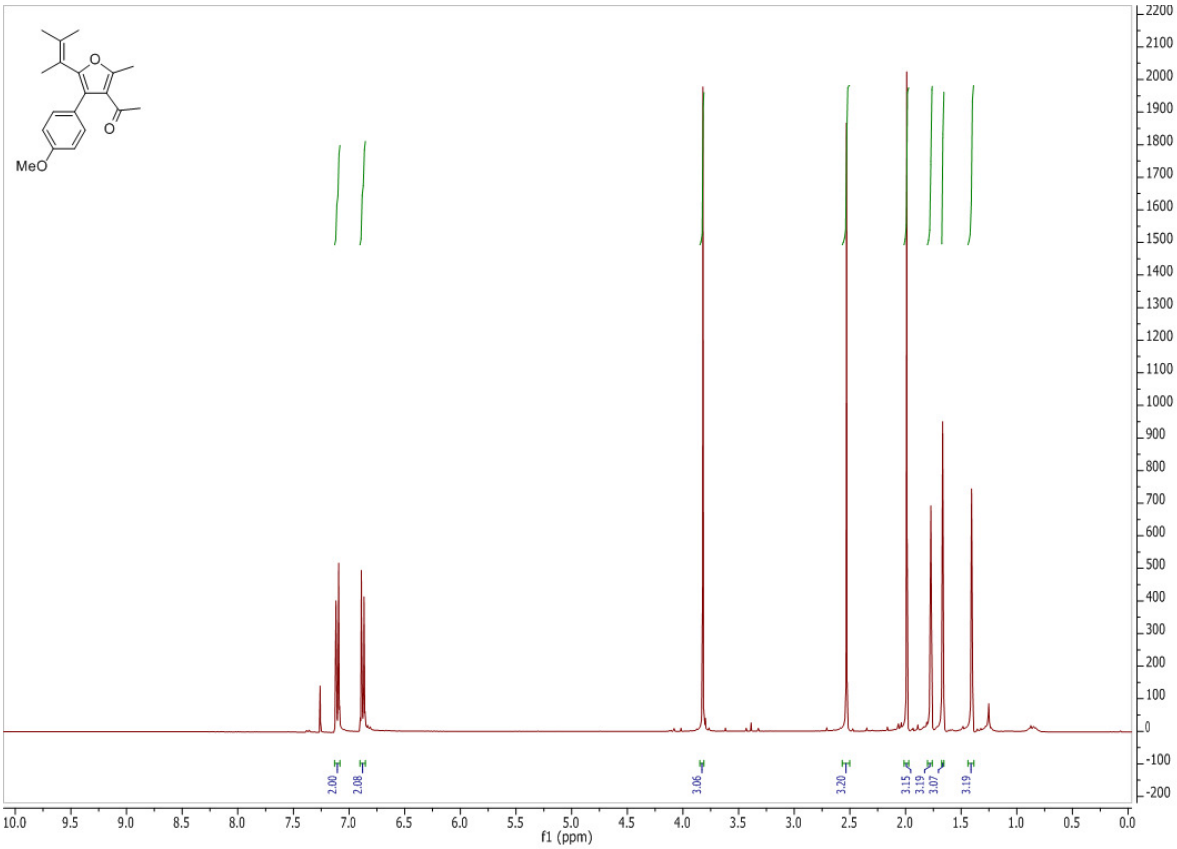


^{13}C NMR (300 K, $CDCl_3$)

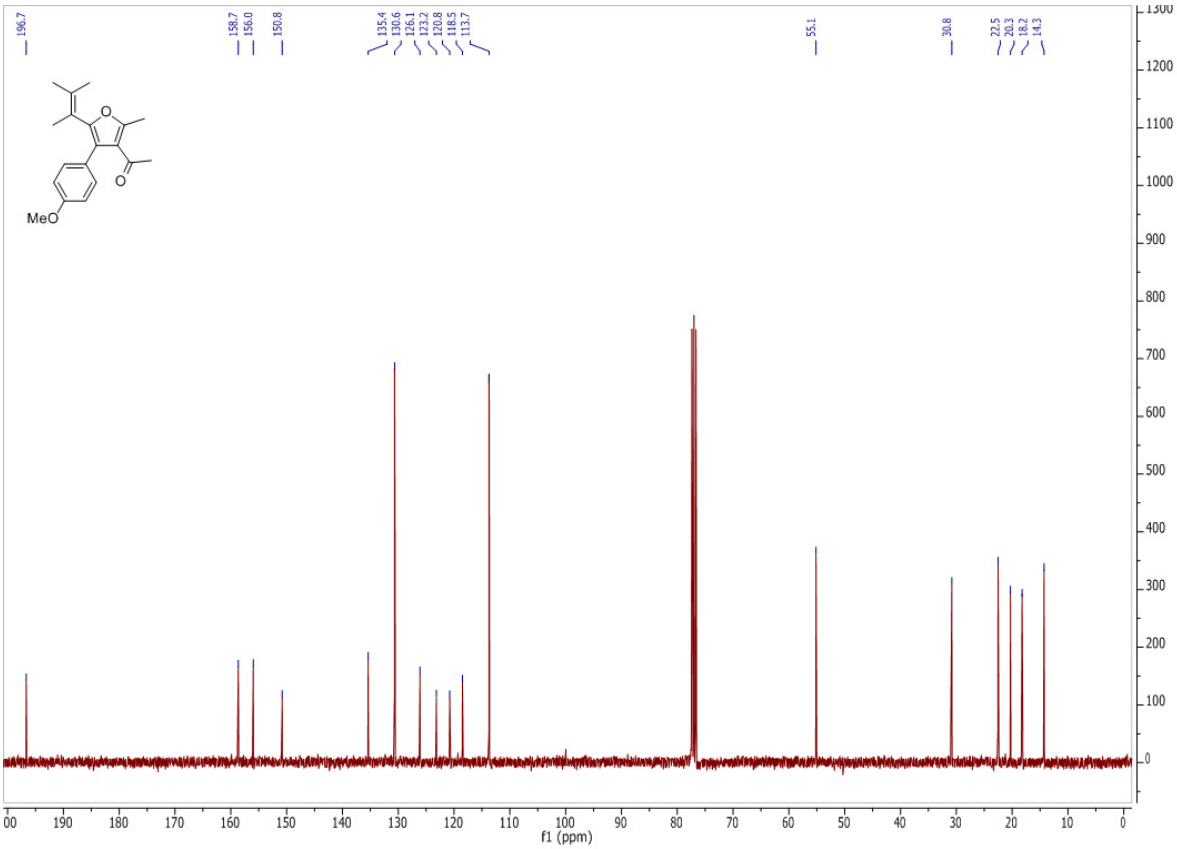


1-[4-(4-Methoxyphenyl)-2-methyl-5-(3-methylbut-2-en-2-yl)furan-3-yl]ethanone (6f)

^{1}H NMR (300 K, $CDCl_3$)

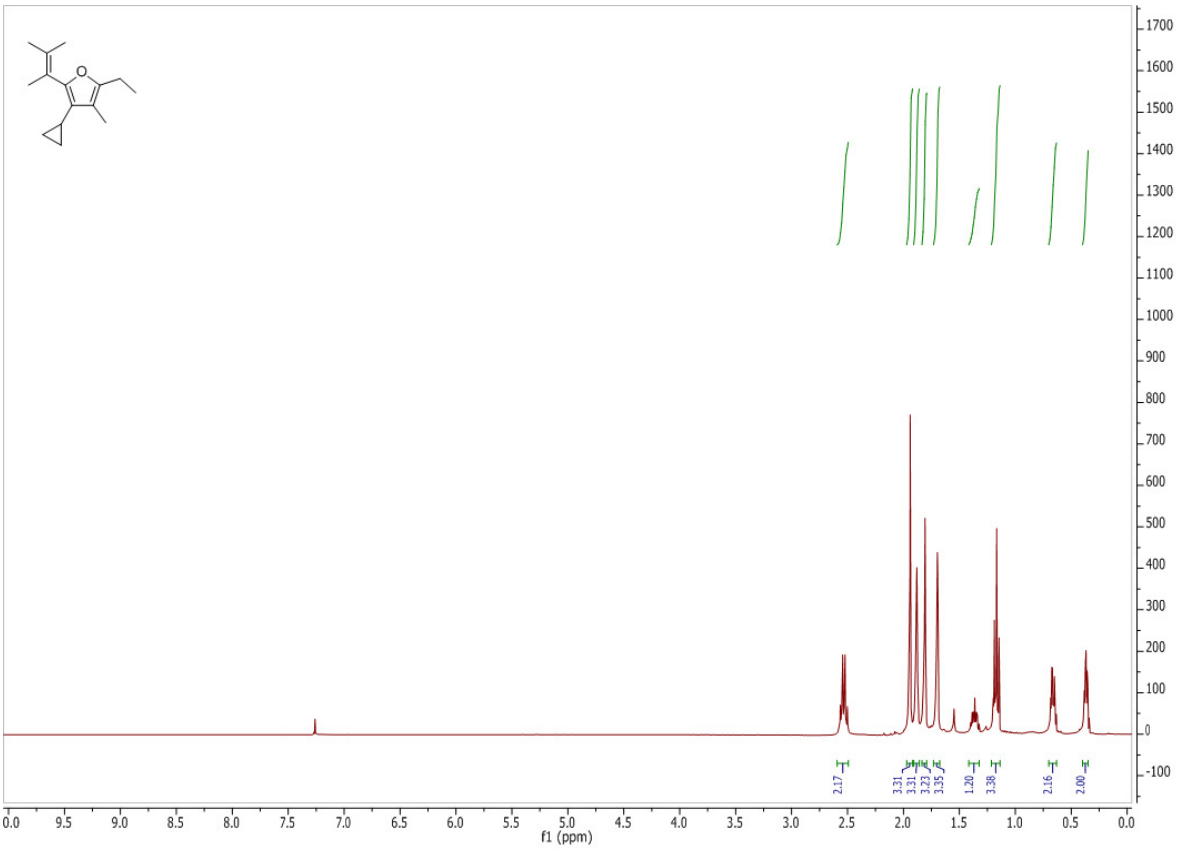


^{13}C NMR (300 K, $CDCl_3$)

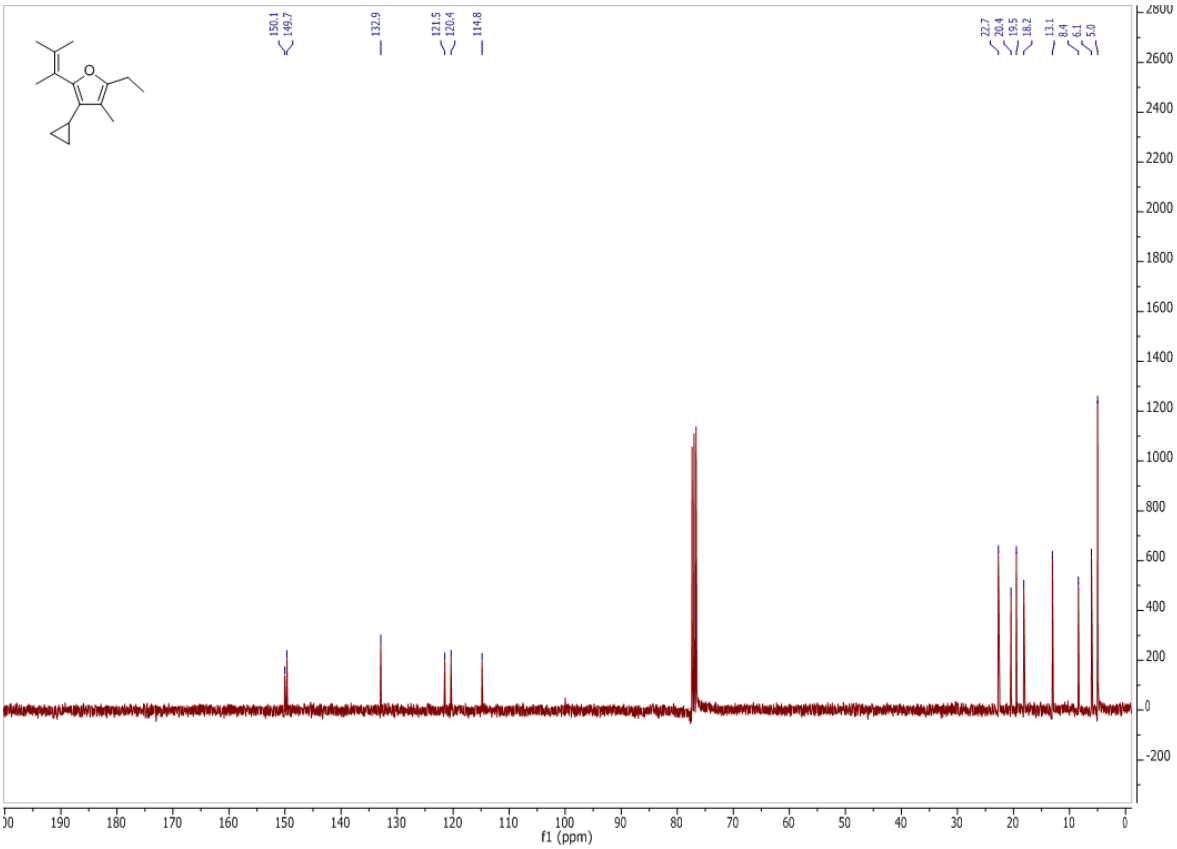


3-Cyclopropyl-5-ethyl-4-methyl-2-(3-methylbut-2-en-2-yl)furan (10a)

^{1}H NMR (300 K, $CDCl_3$)

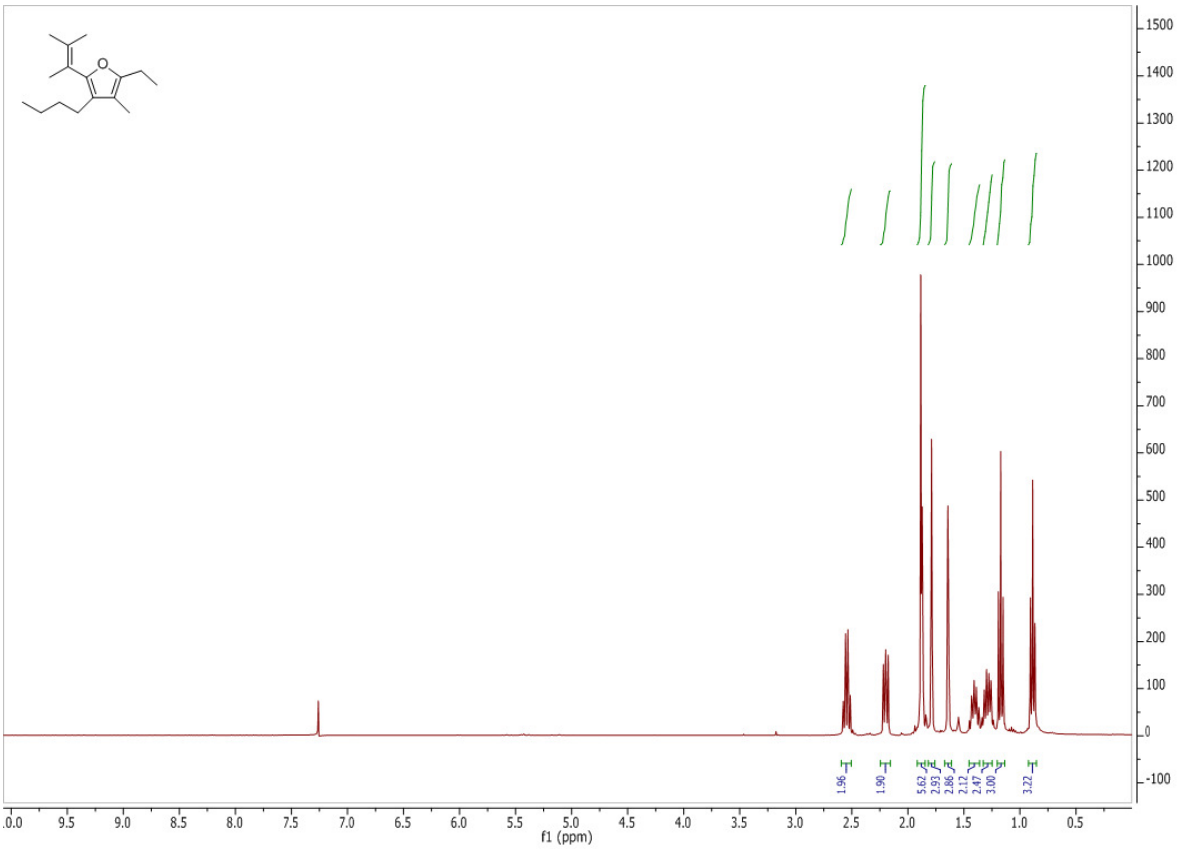


^{13}C NMR (300 K, $CDCl_3$)

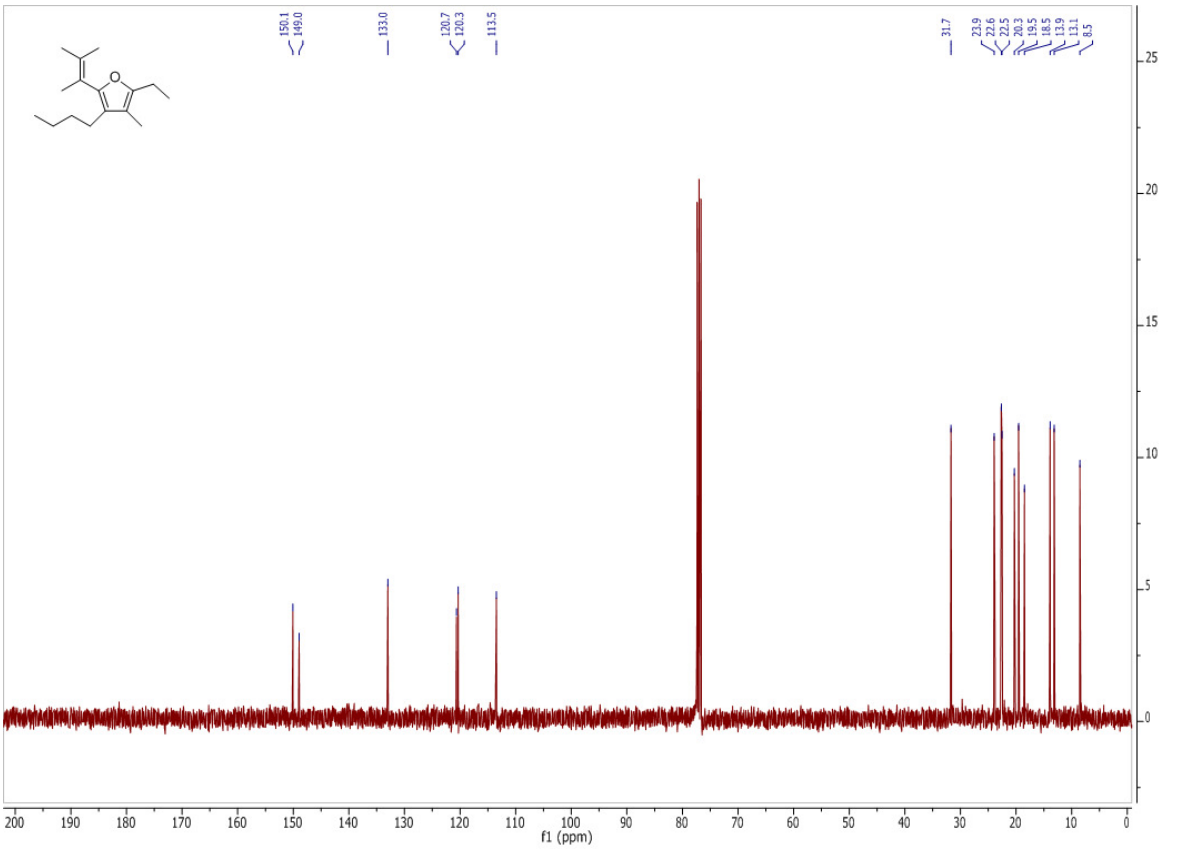


3-*n*-Butyl-5-ethyl-4-methyl-2-(3-methylbut-2-en-2-yl)furan (10b)

^{1}H NMR (300 K, $CDCl_3$)

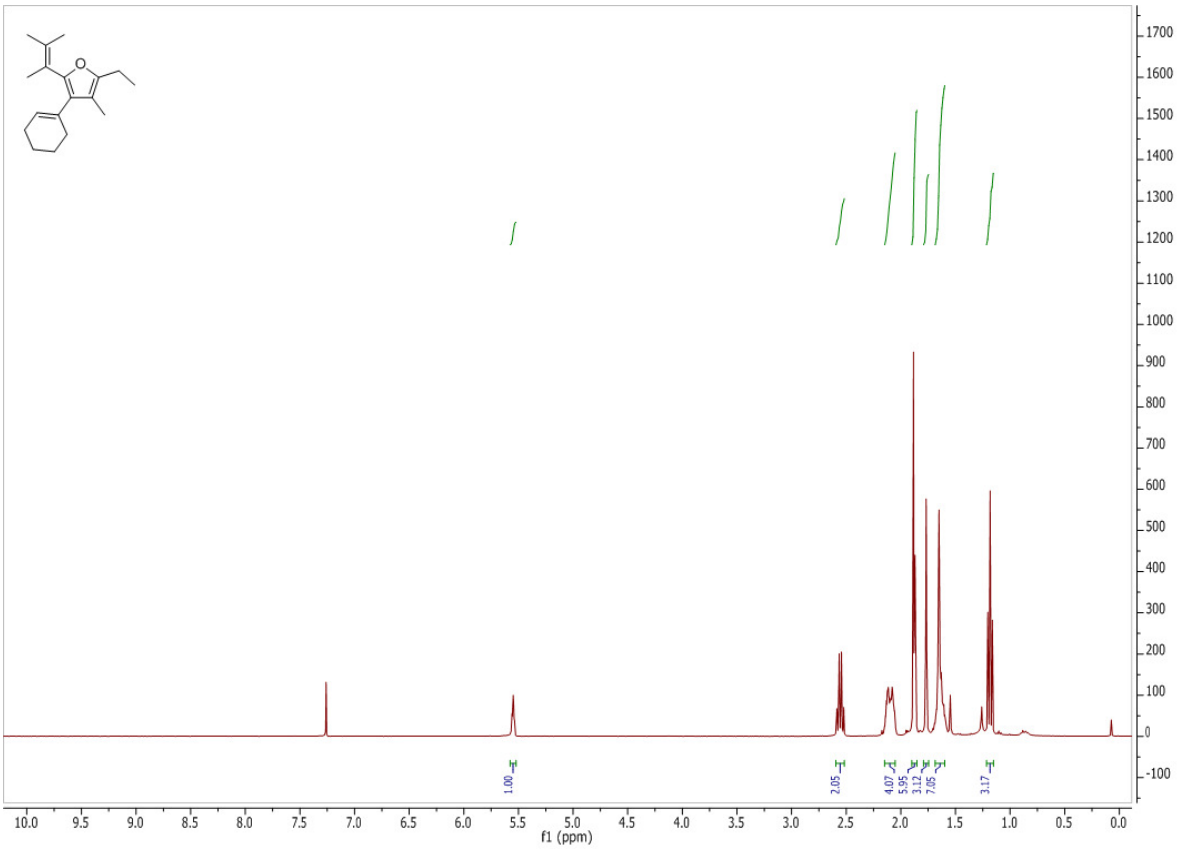


^{13}C NMR (300 K, $CDCl_3$)

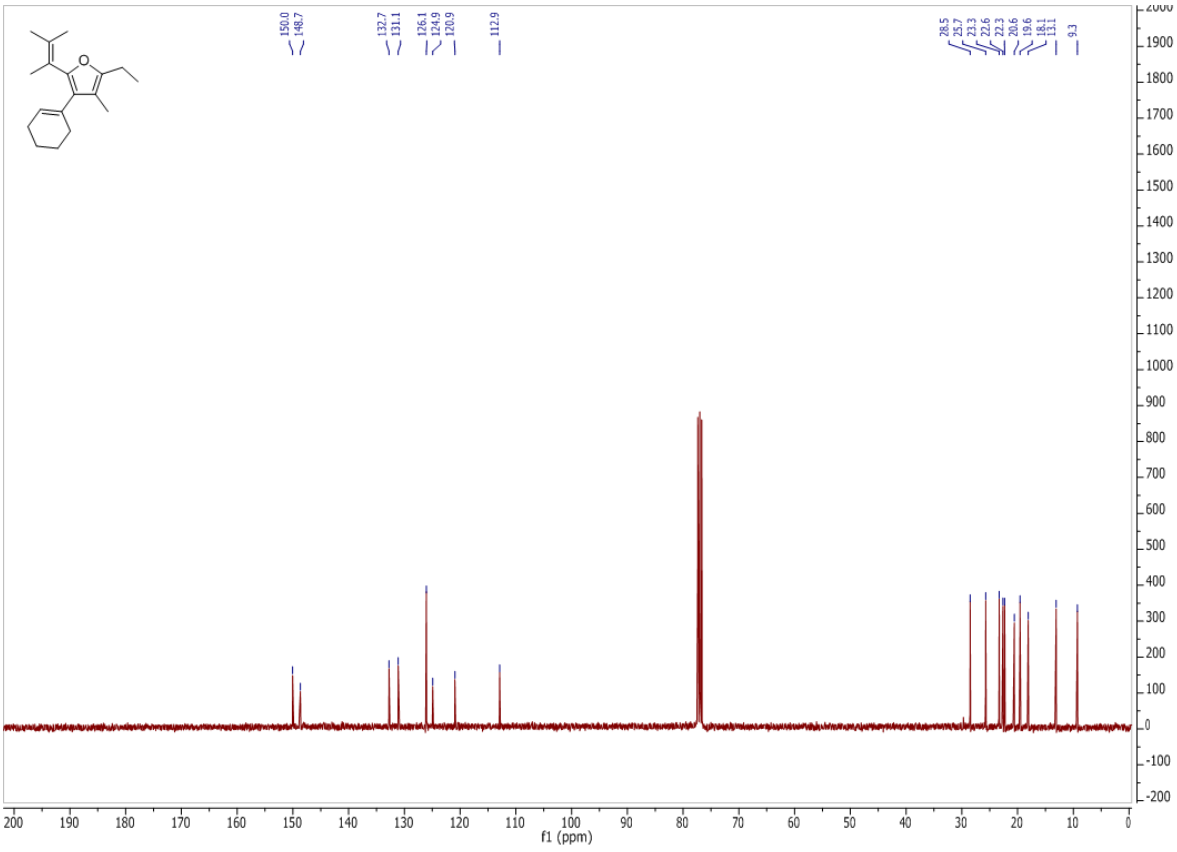


3-(Cyclohex-1-en-1-yl)-5-ethyl-4-methyl-2-(3-methylbut-2-en-2-yl)furan (10c)

^{1}H NMR (300 K, $CDCl_3$)

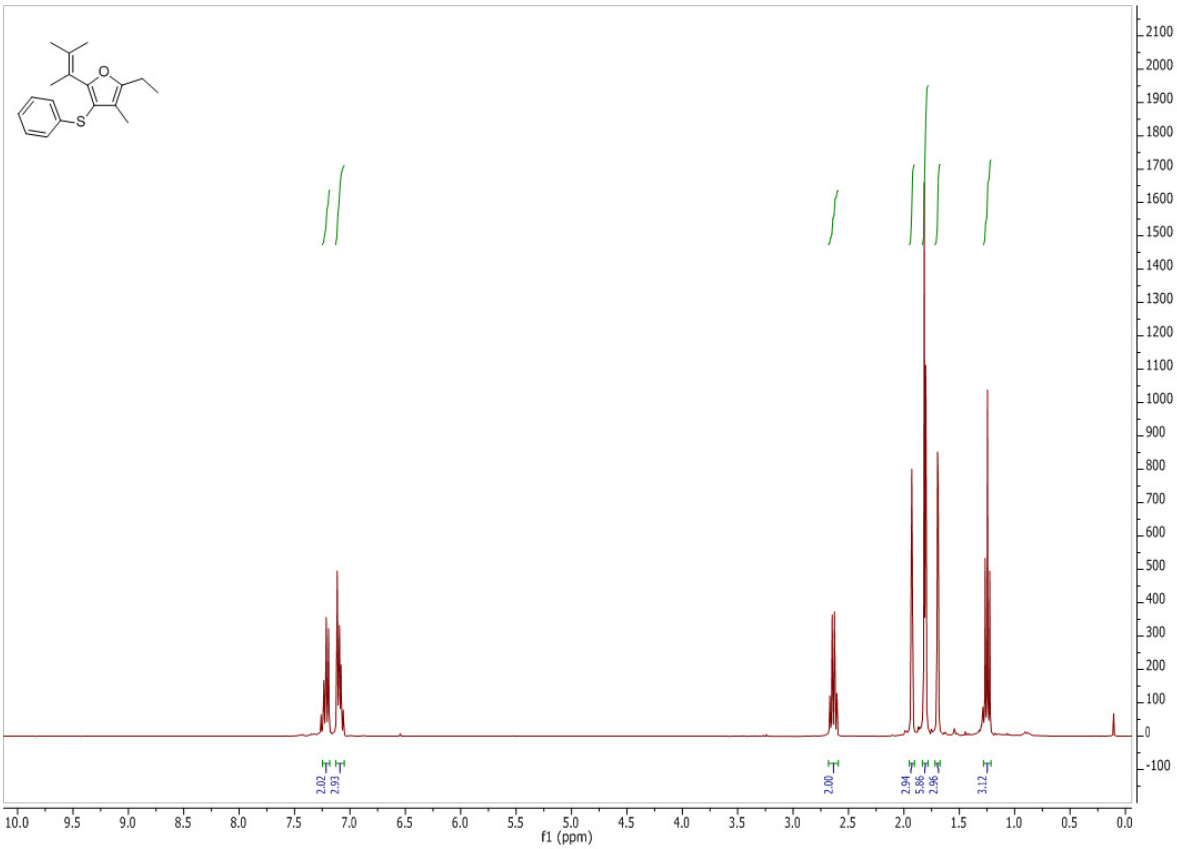


^{13}C NMR (300 K, $CDCl_3$)

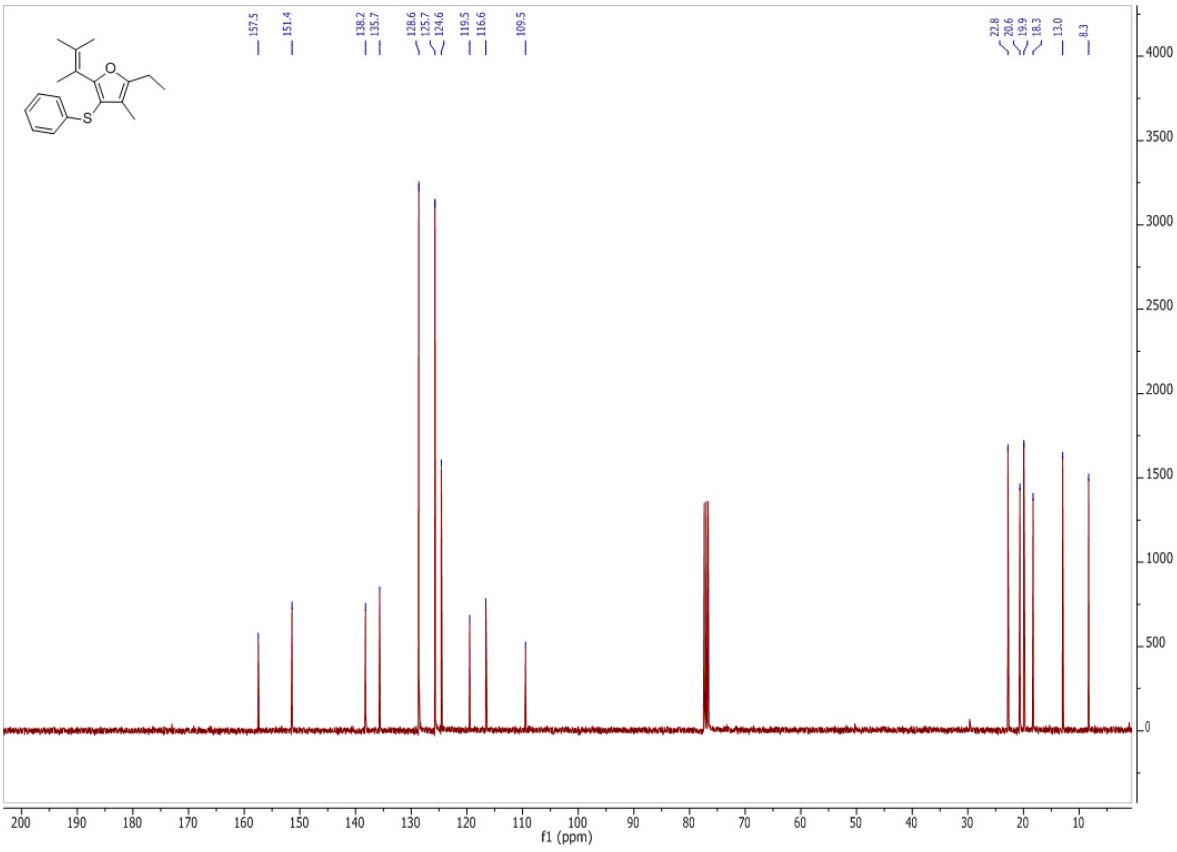


2-Ethyl-3-methyl-5-(3-methylbut-2-en-2-yl)-4-(phenylthio)furan (10d)

^{1}H NMR (300 K, $CDCl_3$)

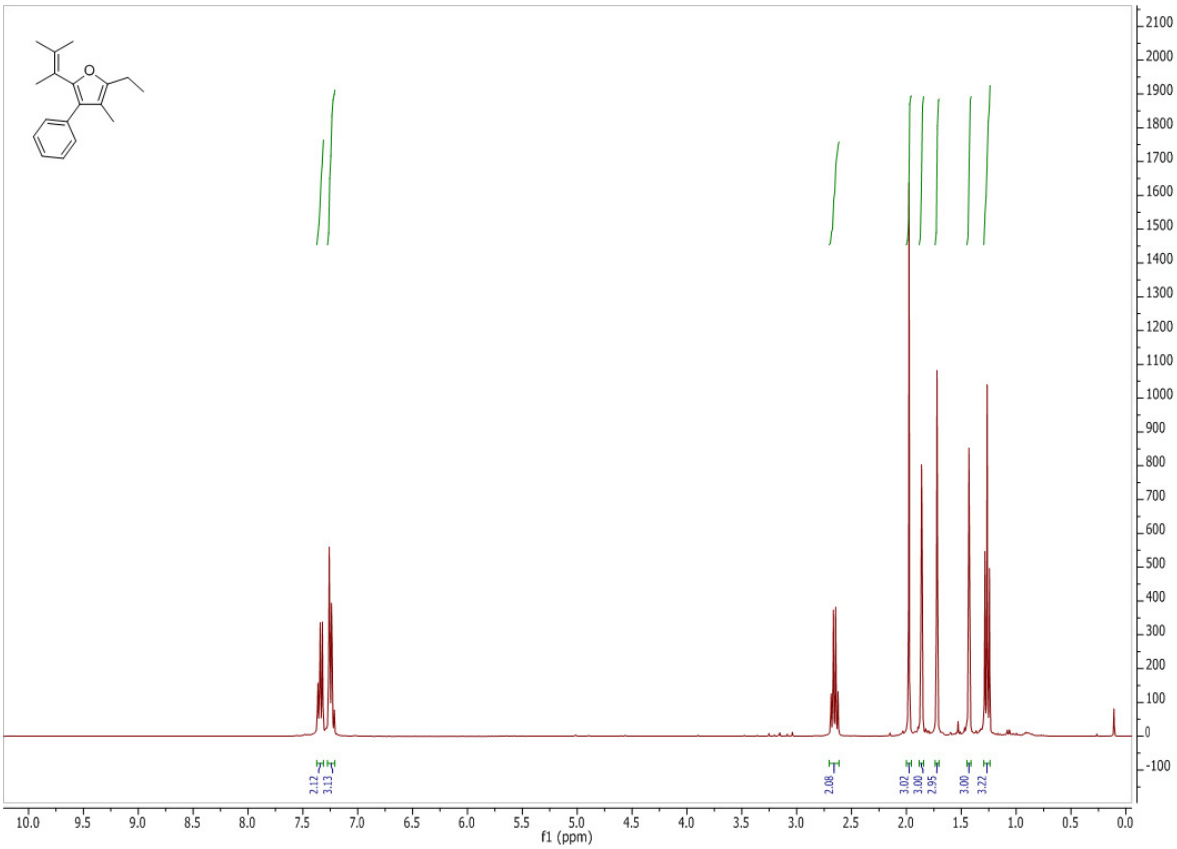


^{13}C NMR (300 K, $CDCl_3$)

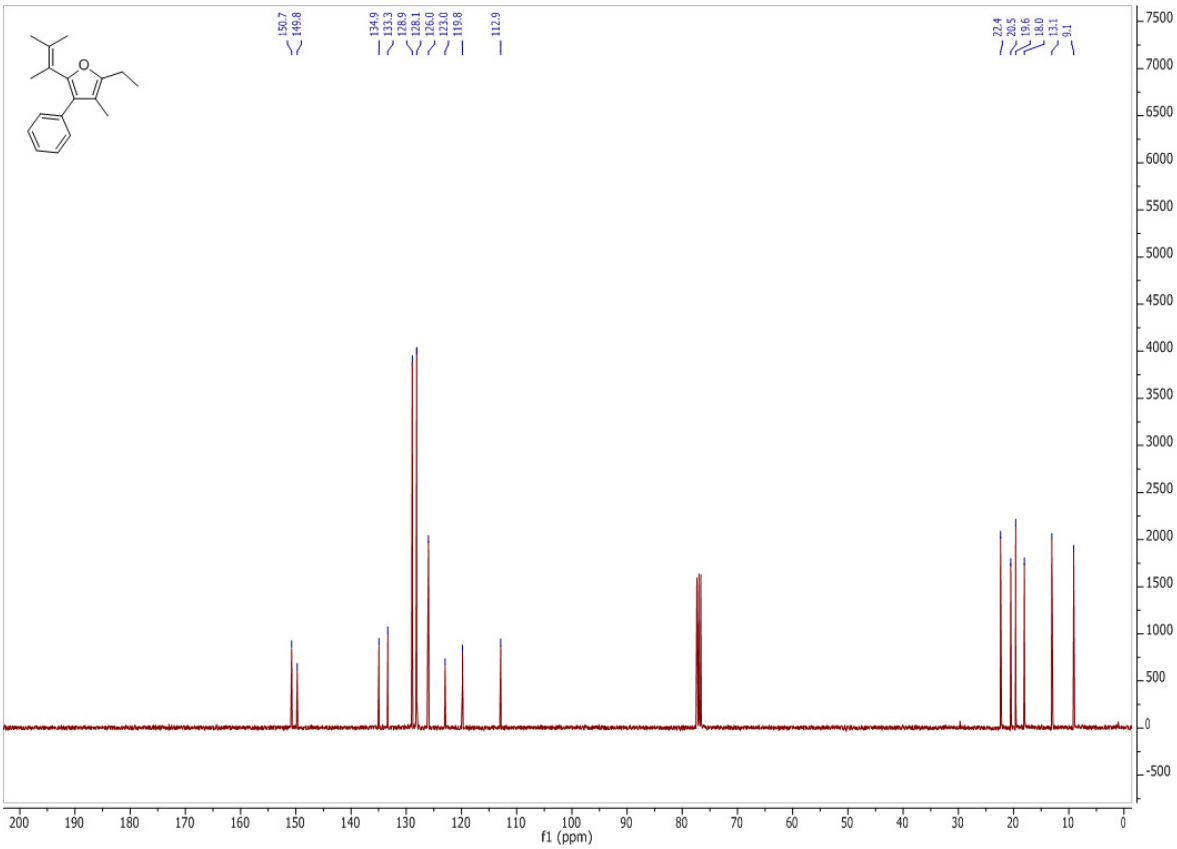


2-Ethyl-3-methyl-5-(3-methylbut-2-en-2-yl)-4-phenylfuran (10e)

^{1}H NMR (300 K, $CDCl_3$)

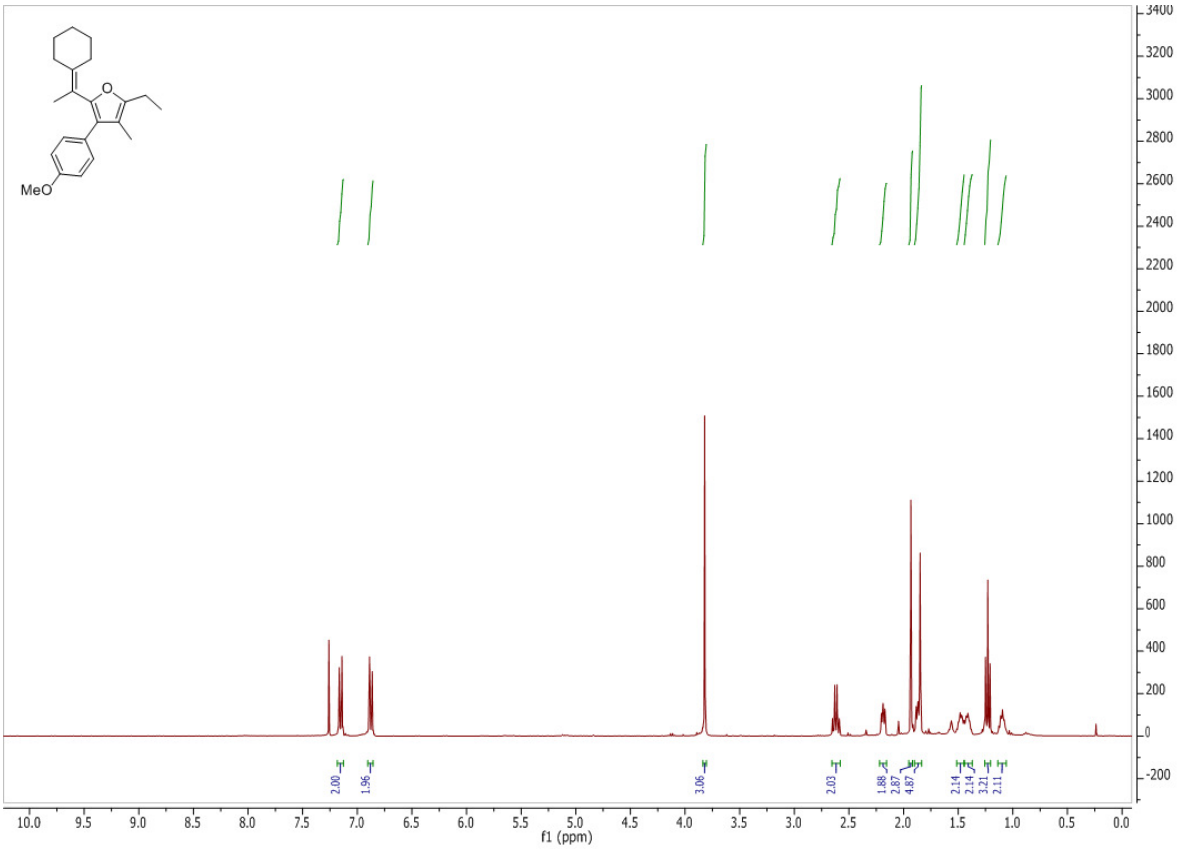


^{13}C NMR (300 K, $CDCl_3$)

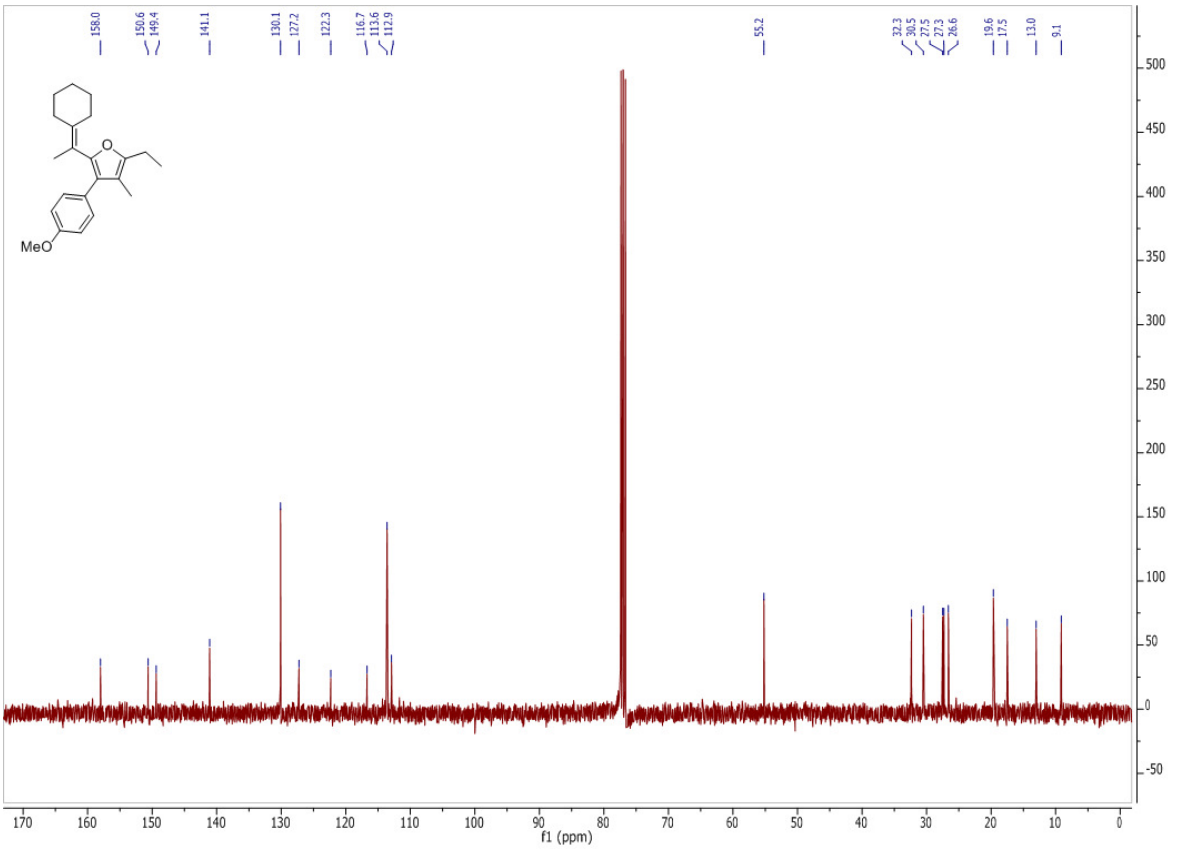


2-(1-Cyclohexylideneethyl)-5-ethyl-3-(4-methoxyphenyl)-4-methylfuran (10f)

^{1}H NMR (300 K, $CDCl_3$)

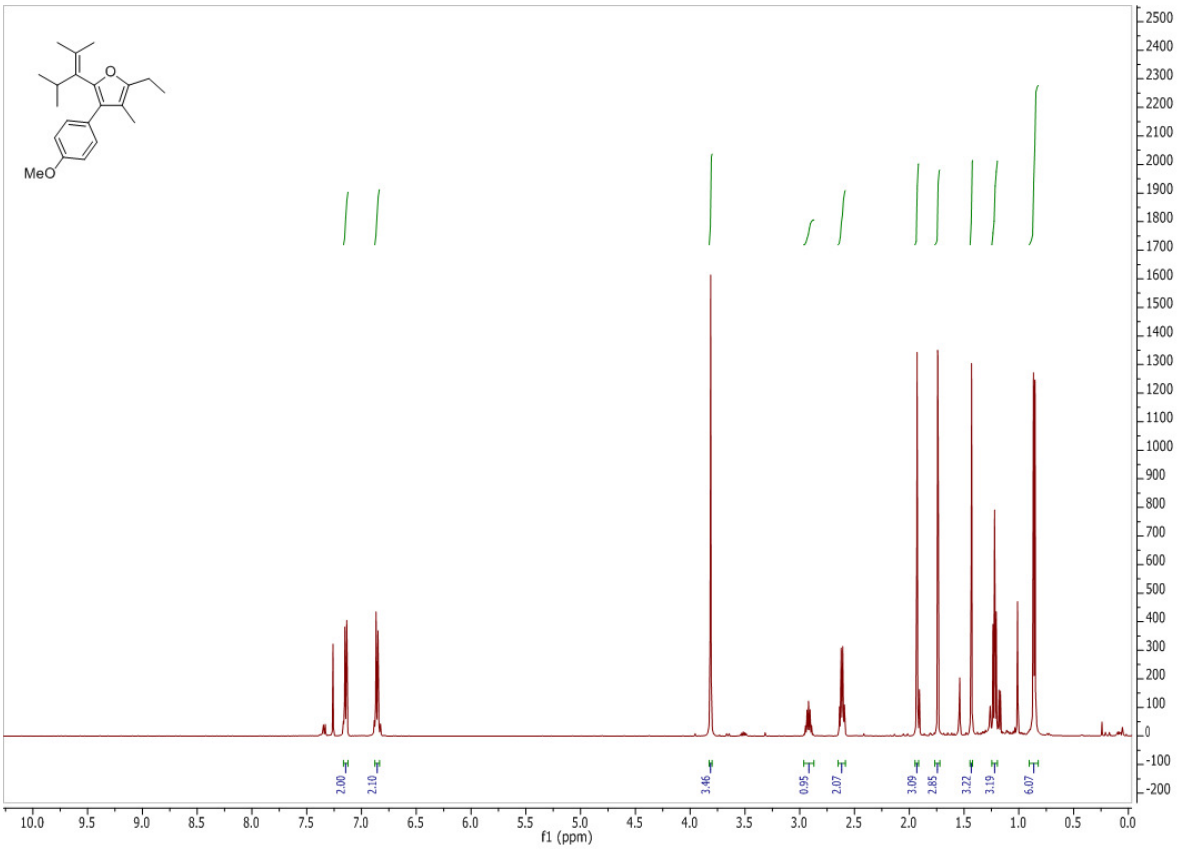


^{13}C NMR (300 K, $CDCl_3$)



2-(2,4-Dimethylpent-2-en-3-yl)-5-ethyl-3-(4-methoxyphenyl)-4-methylfuran (10g)

^{1}H NMR (300 K, $CDCl_3$)



^{13}C NMR (300 K, $CDCl_3$)