

Electronic Supplementary Information

# Palladium-catalyzed cross-coupling of arylcarboxylic acid 2-pyridyl esters with terminal alkynes

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## Contents

I. Optimization of reaction conditions	S2
II. NMR spectral copies of unreported reactants and the cross-coupling products	S3
1. NMR spectral copies of the unreported pyridyl esters	S4
2. NMR spectral copies of the cross-coupling products	S10

## I. Optimization of reaction conditions

Table S1. Optimization of reaction conditions<sup>a</sup>

Entry	[Pd]	L	Base	Solvent	Yield <sup>b</sup> (%)
1	Pd(OAc) <sub>2</sub>	iMes·HCl	Na <sub>2</sub> CO <sub>3</sub>	dioxane	0
2	Pd(OAc) <sub>2</sub>	PPh <sub>3</sub>	Na <sub>2</sub> CO <sub>3</sub>	dioxane	7
3	Pd(OAc) <sub>2</sub>	dppe	Na <sub>2</sub> CO <sub>3</sub>	dioxane	20
4	Pd(OAc) <sub>2</sub>	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	56
5	Pd(OAc) <sub>2</sub>	dppf	Na <sub>2</sub> CO <sub>3</sub>	dioxane	36
6	Pd(OAc) <sub>2</sub>	dcype	Na <sub>2</sub> CO <sub>3</sub>	dioxane	trace
7	Pd(OAc) <sub>2</sub>	xantphos	Na <sub>2</sub> CO <sub>3</sub>	dioxane	trace
8	PdCl <sub>2</sub>	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	37
9	PdCl <sub>2</sub> (CH <sub>3</sub> CN) <sub>2</sub>	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	39
10	[Pd(π-allyl)Cl] <sub>2</sub>	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	41
11	PEPPSI-IPr	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	34
12	PdCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub>	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	37
13	PdCl <sub>2</sub> (PCy <sub>3</sub> ) <sub>2</sub>	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	38
14	PdCl <sub>2</sub> (dppf)	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	71
15	Pd <sub>2</sub> dba <sub>3</sub>	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	63
16	Pd(PPh <sub>3</sub> ) <sub>4</sub>	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	47
17	PdCl <sub>2</sub> (dppf)	dppp	K <sub>2</sub> CO <sub>3</sub>	dioxane	<5
18	PdCl <sub>2</sub> (dppf)	dppp	Cs <sub>2</sub> CO <sub>3</sub>	dioxane	trace
19	PdCl <sub>2</sub> (dppf)	dppp	NaHCO <sub>3</sub>	dioxane	53
20	PdCl <sub>2</sub> (dppf)	dppp	NaOAc	dioxane	54
21	PdCl <sub>2</sub> (dppf)	dppp	tBuONa	dioxane	11
22	PdCl <sub>2</sub> (dppf)	dppp	Na <sub>2</sub> CO <sub>3</sub>	toluene	46
23	PdCl <sub>2</sub> (dppf)	dppp	Na <sub>2</sub> CO <sub>3</sub>	THF	47
24	PdCl <sub>2</sub> (dppf)	dppp	Na <sub>2</sub> CO <sub>3</sub>	DME	37
25 <sup>c</sup>	PdCl <sub>2</sub> (dppf)	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	88
26 <sup>d</sup>	PdCl <sub>2</sub> (dppf)	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	>99
27 <sup>e</sup>	PdCl <sub>2</sub> (dppf)	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	49
28 <sup>d,f</sup>	PdCl <sub>2</sub> (dppf)	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	90
29 <sup>d,g</sup>	PdCl <sub>2</sub> (dppf)	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	81
30 <sup>d,h</sup>	PdCl <sub>2</sub> (dppf)	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	>99

31 <sup>d</sup>	PdCl <sub>2</sub> (dppf)	dppp	-	dioxane	13
32 <sup>d,h,i</sup>	PdCl <sub>2</sub> (dppf)	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	86
33 <sup>d,h,j</sup>	PdCl <sub>2</sub> (dppf)	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	76
34 <sup>d,h</sup>	-	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	0
35 <sup>d,h,k</sup>	PdCl <sub>2</sub> (dppf)	dppp	Na <sub>2</sub> CO <sub>3</sub>	dioxane	67

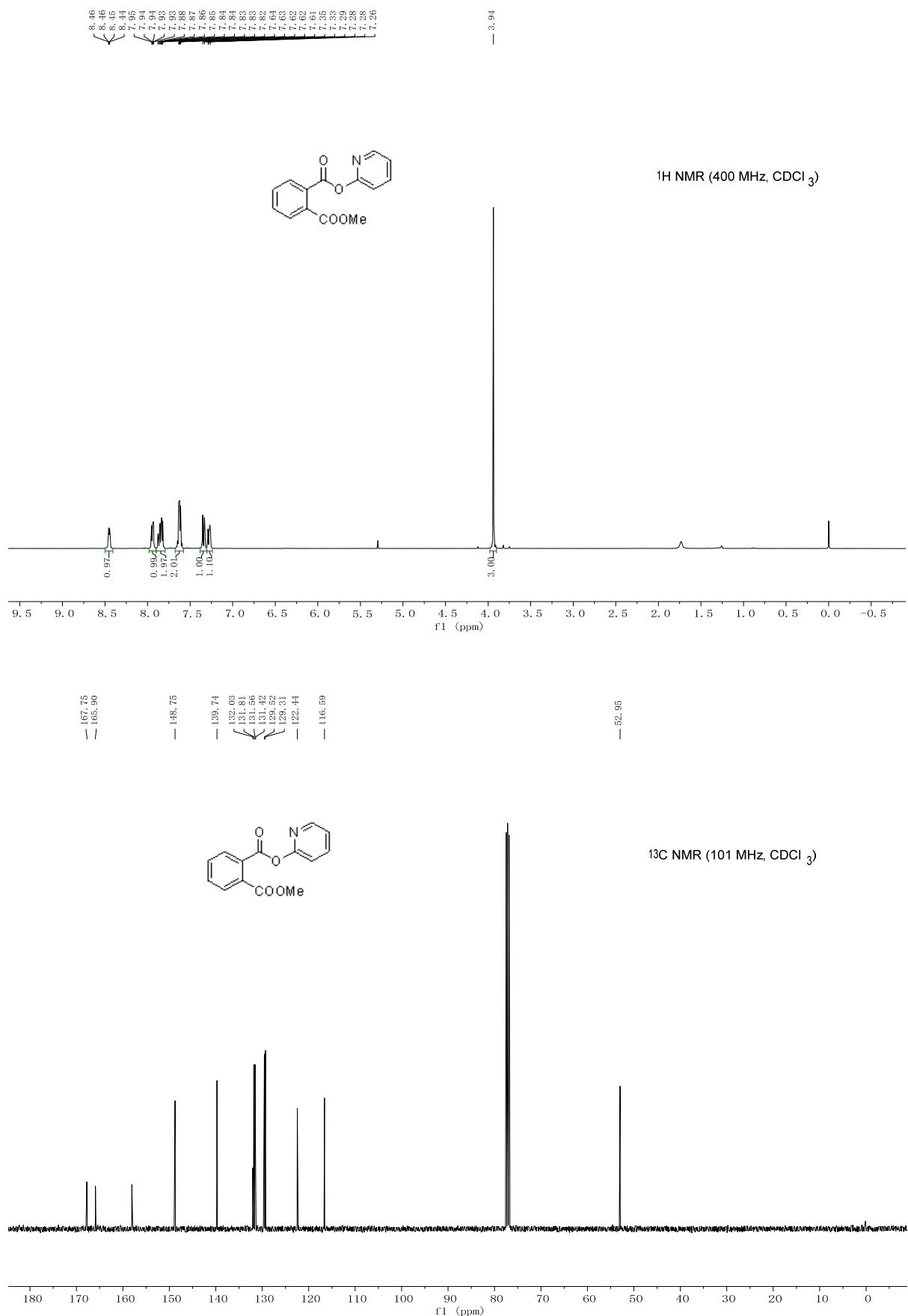
<sup>a</sup> Unless otherwise noted, the reactions were carried out according to the conditions indicated in the above equation. <sup>b</sup> Yields were determined by <sup>1</sup>H NMR spectral analysis with tetrachloroethane as an internal standard. <sup>c</sup> 2 equiv. of **2a** were used. <sup>d</sup> 3 equiv. of **2a** were used. <sup>e</sup> 0.3 mmol **1a** and 0.2 mmol **2a** were used. <sup>f</sup> 5 mol% CuI was used. <sup>g</sup> The reaction temperature was 130 °C. <sup>h</sup> 0.5 equiv. of Na<sub>2</sub>CO<sub>3</sub> was used. <sup>i</sup> 5 mol% of dppp were used. <sup>j</sup> 2.5 mol% PdCl<sub>2</sub>(dppf) and 5 mol% dppp were used. <sup>k</sup> The reaction was carried out in air.

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## II. NMR spectral copies of unreported reactants and the cross-coupling products

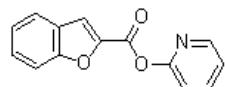
1. NMR spectral copies of the unreported pyridyl esters

(1) methyl pyridin-2-yl phthalate (**1j**)

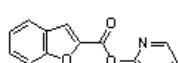
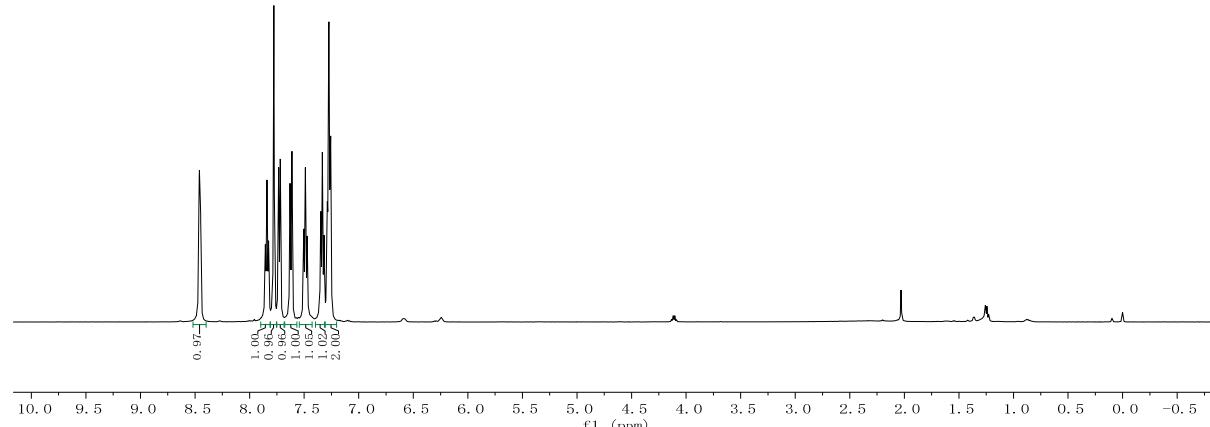


(2) pyridin-2-yl benzofuran-2-carboxylate (**1r**)

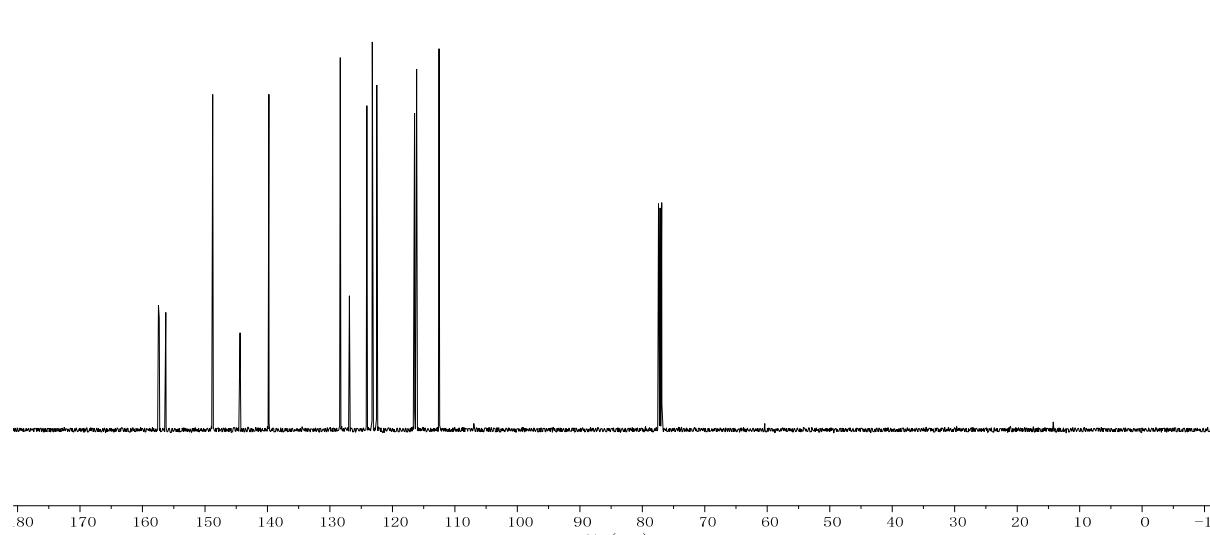
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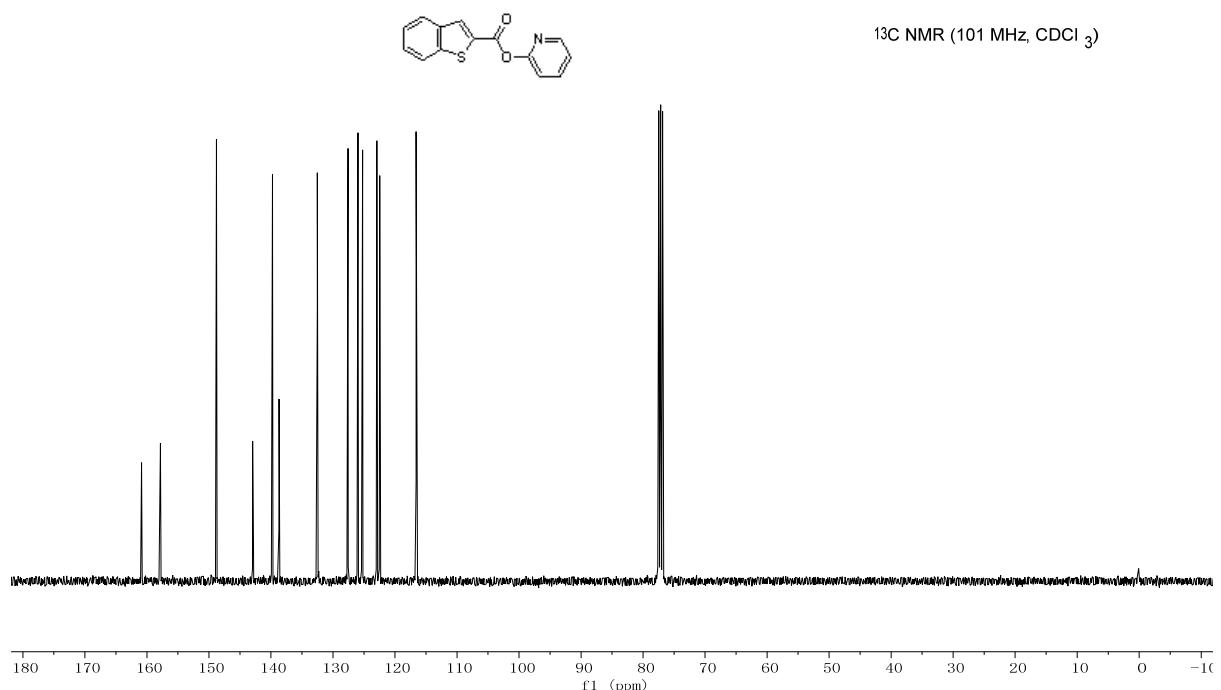
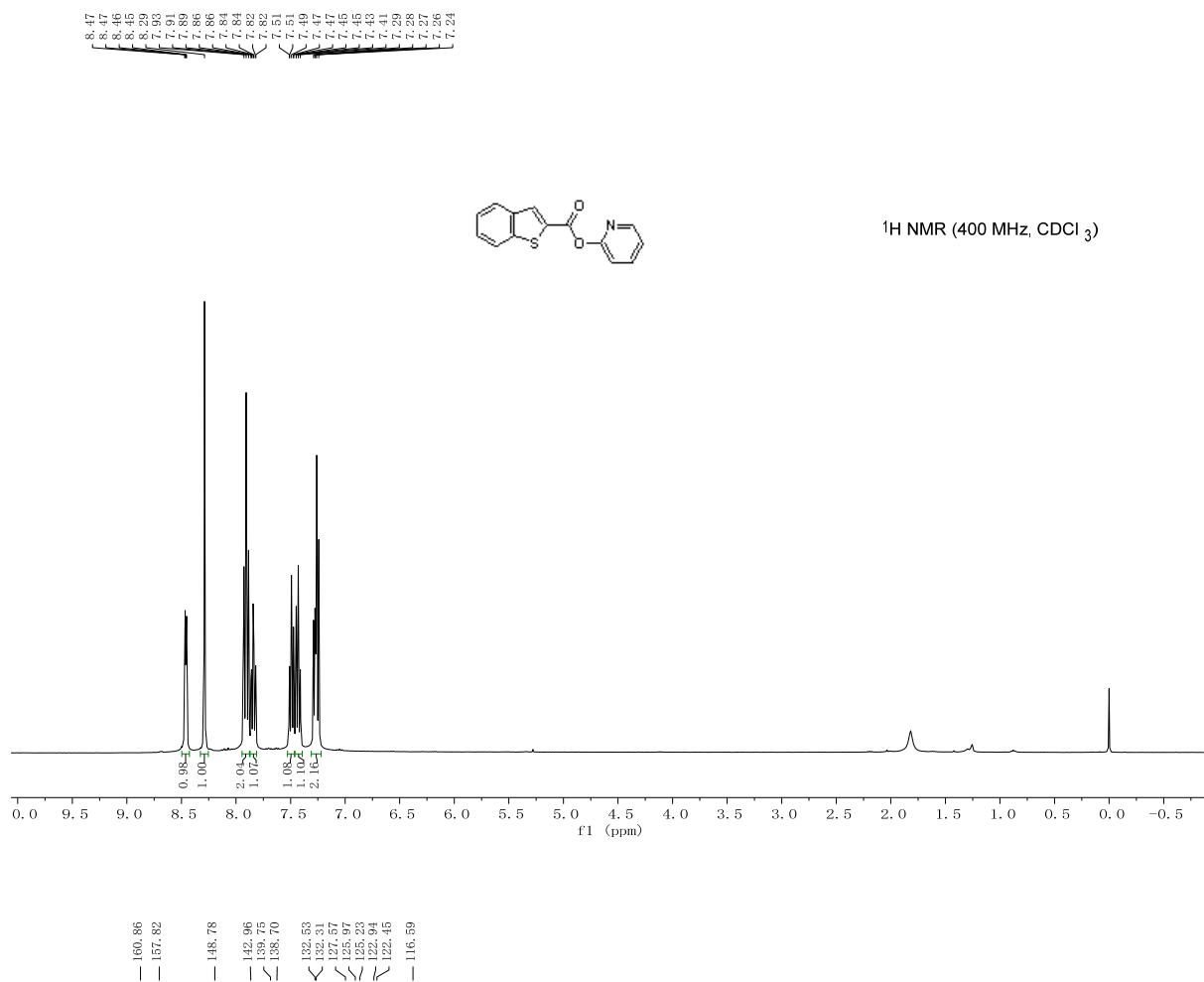
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)



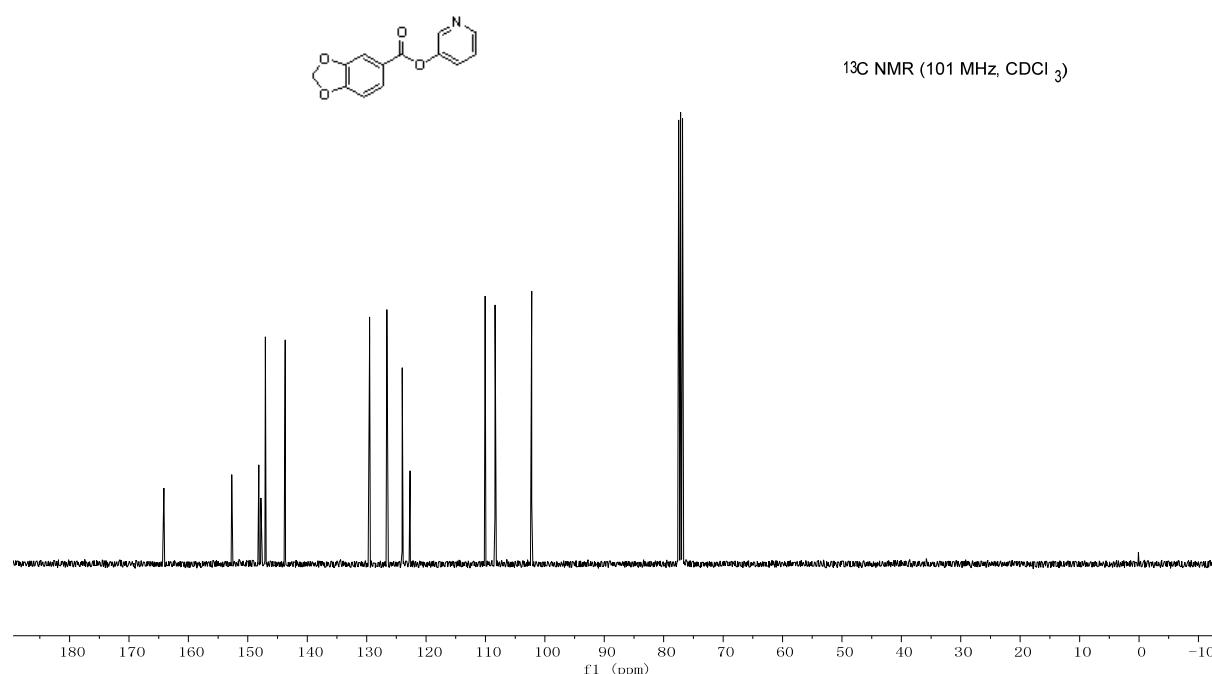
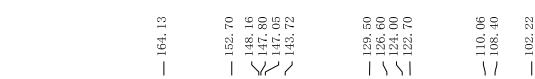
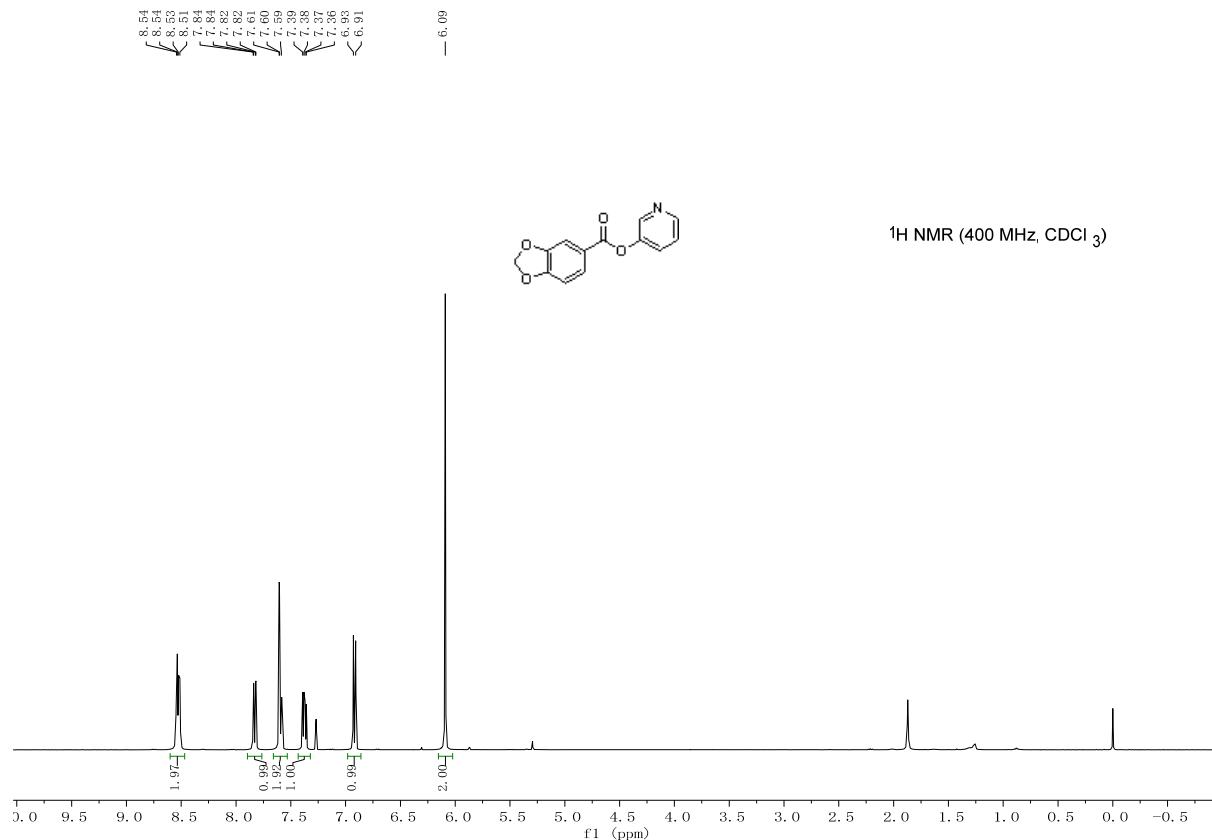
<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)



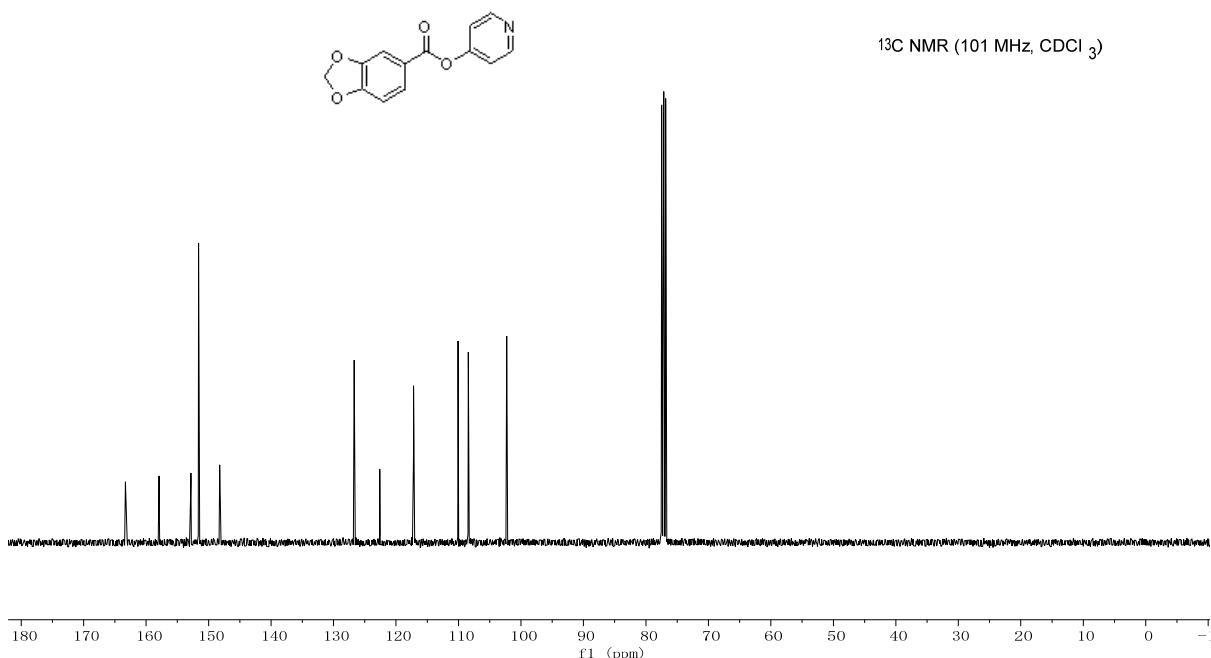
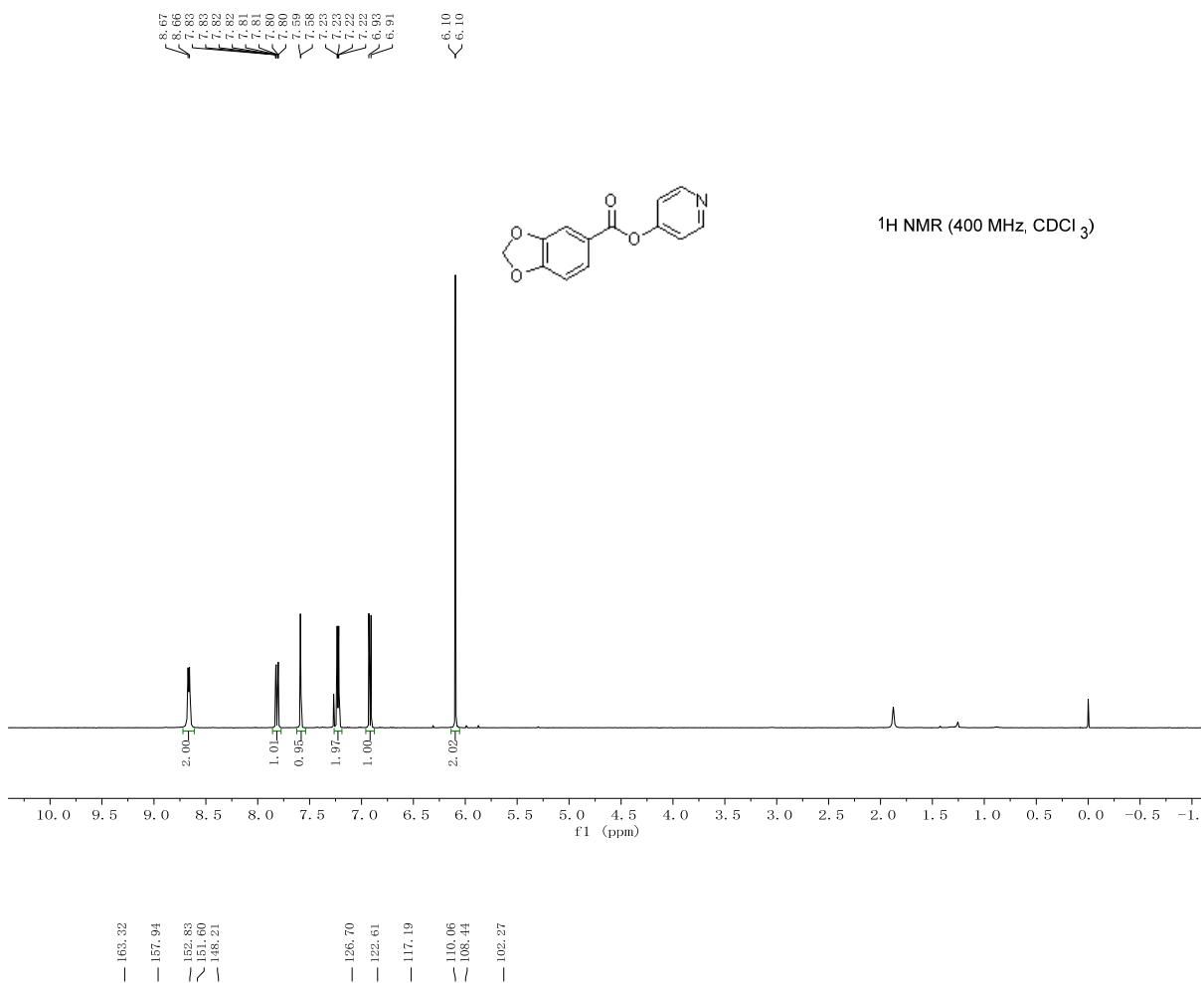
(3) pyridin-2-yl benzo[*b*]thiophene-2-carboxylate (**1s**)



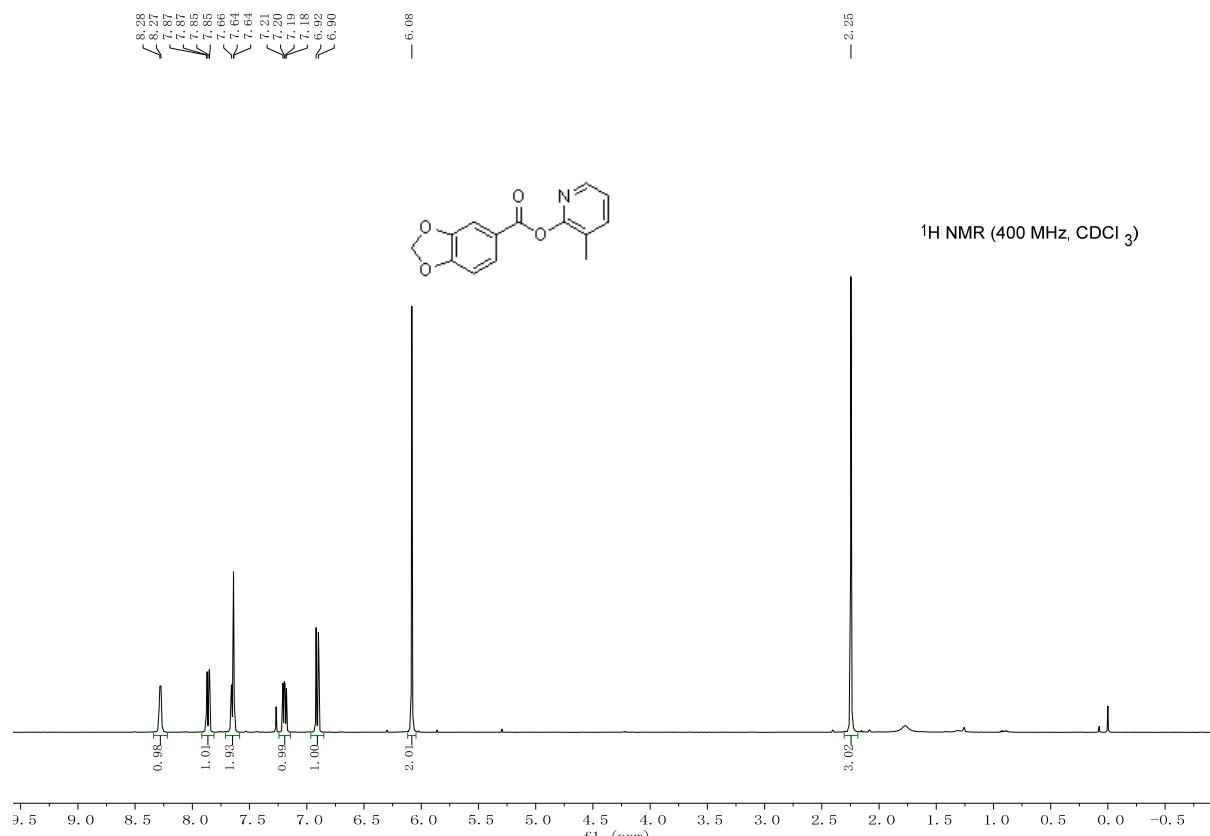
(4) pyridin-3-yl benzo[*d*][1,3]dioxole-5-carboxylate (**1w**)



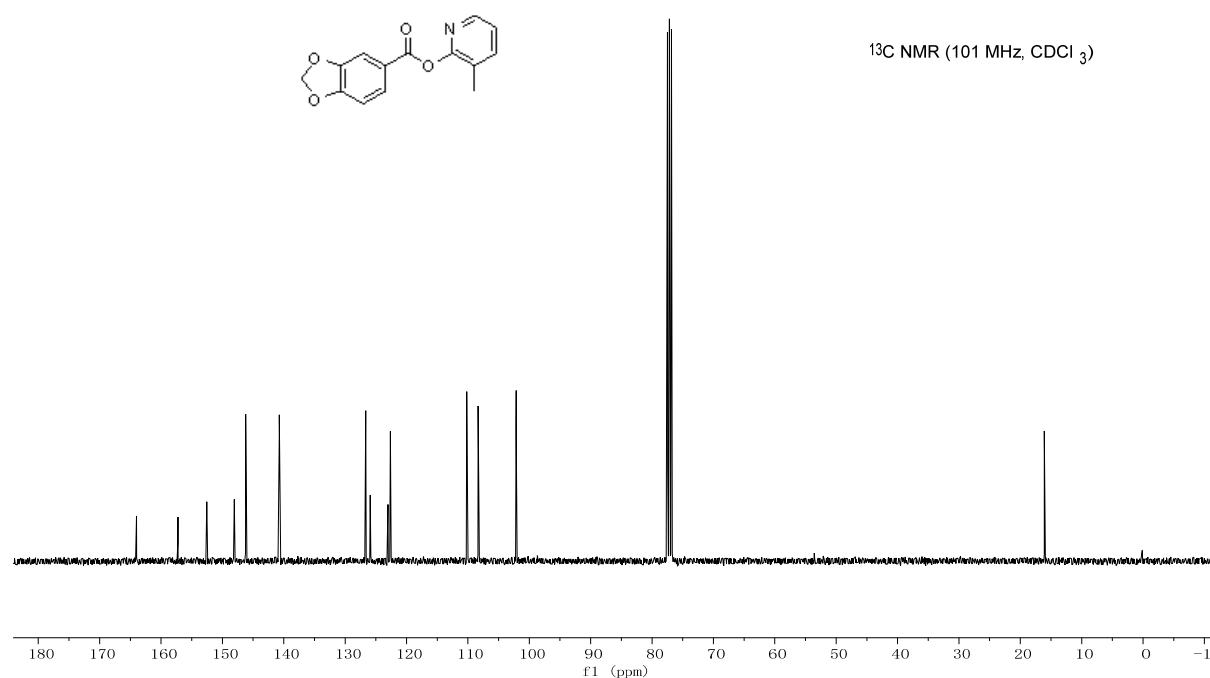
(5) pyridin-4-yl benzo[*d*][1,3]dioxole-5-carboxylate (**1x**)



(6) 3-methylpyridin-2-yl benzo[*d*][1,3]dioxole-5-carboxylate (**1y**)

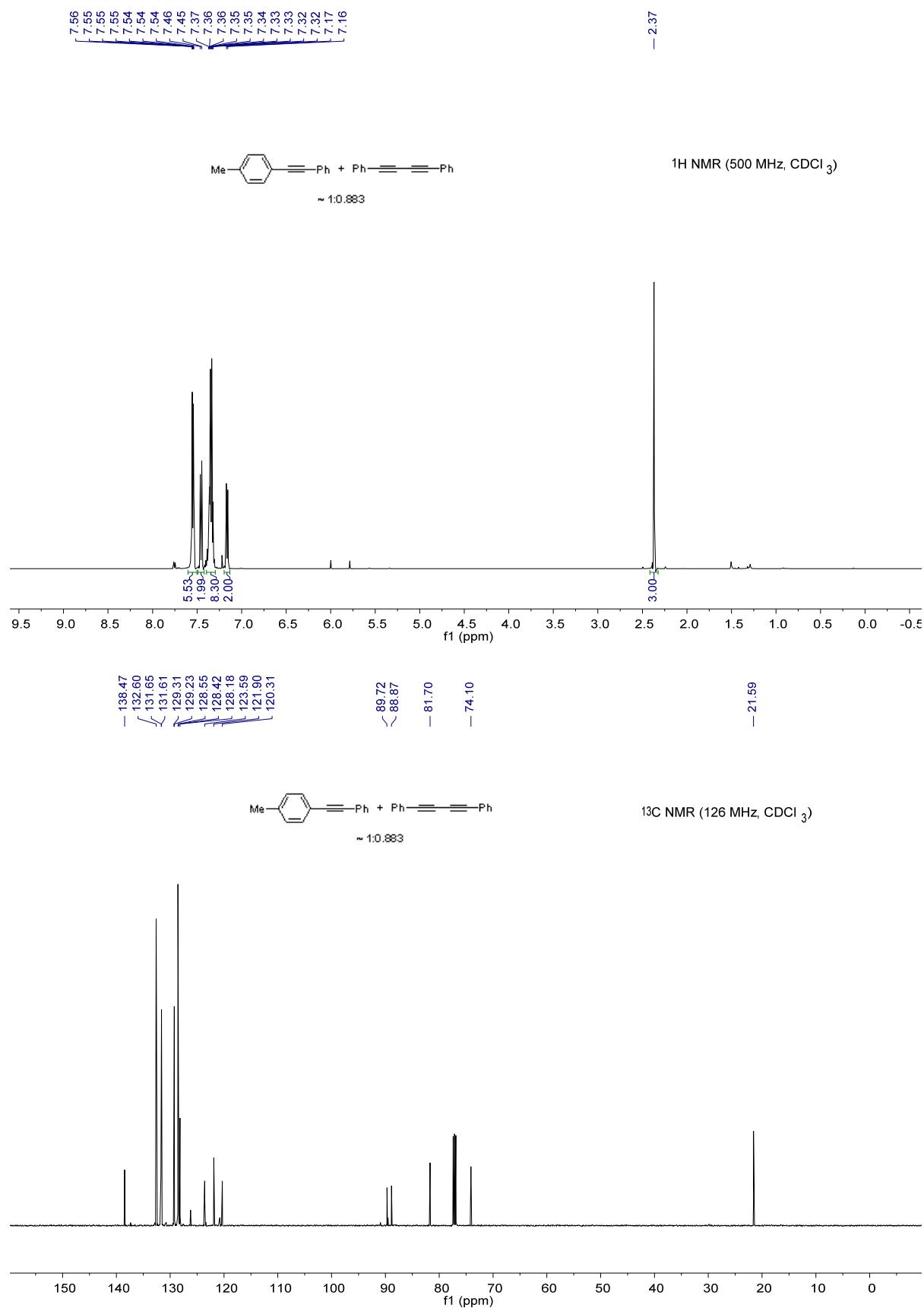


— 164.01  
— 157.25  
— 152.55  
— 148.06  
— 146.21  
— 140.72  
— 126.64  
— 125.92  
— 123.03  
— 122.63  
— 110.19  
— 108.33  
— 102.14  
— 16.08

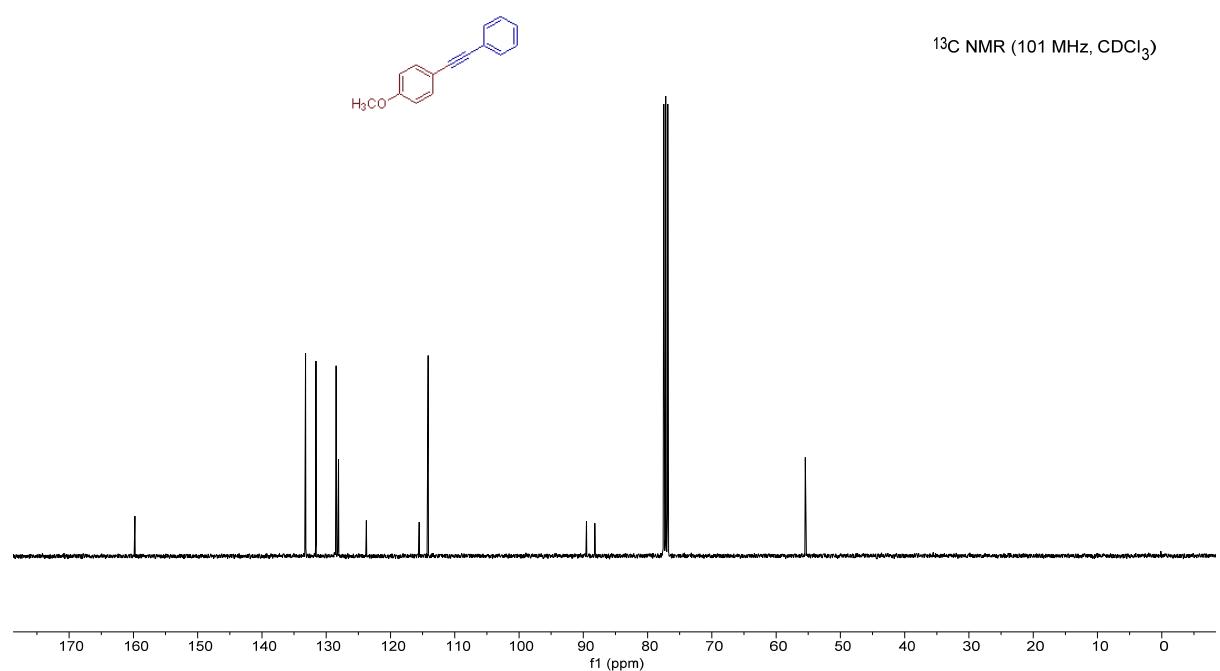
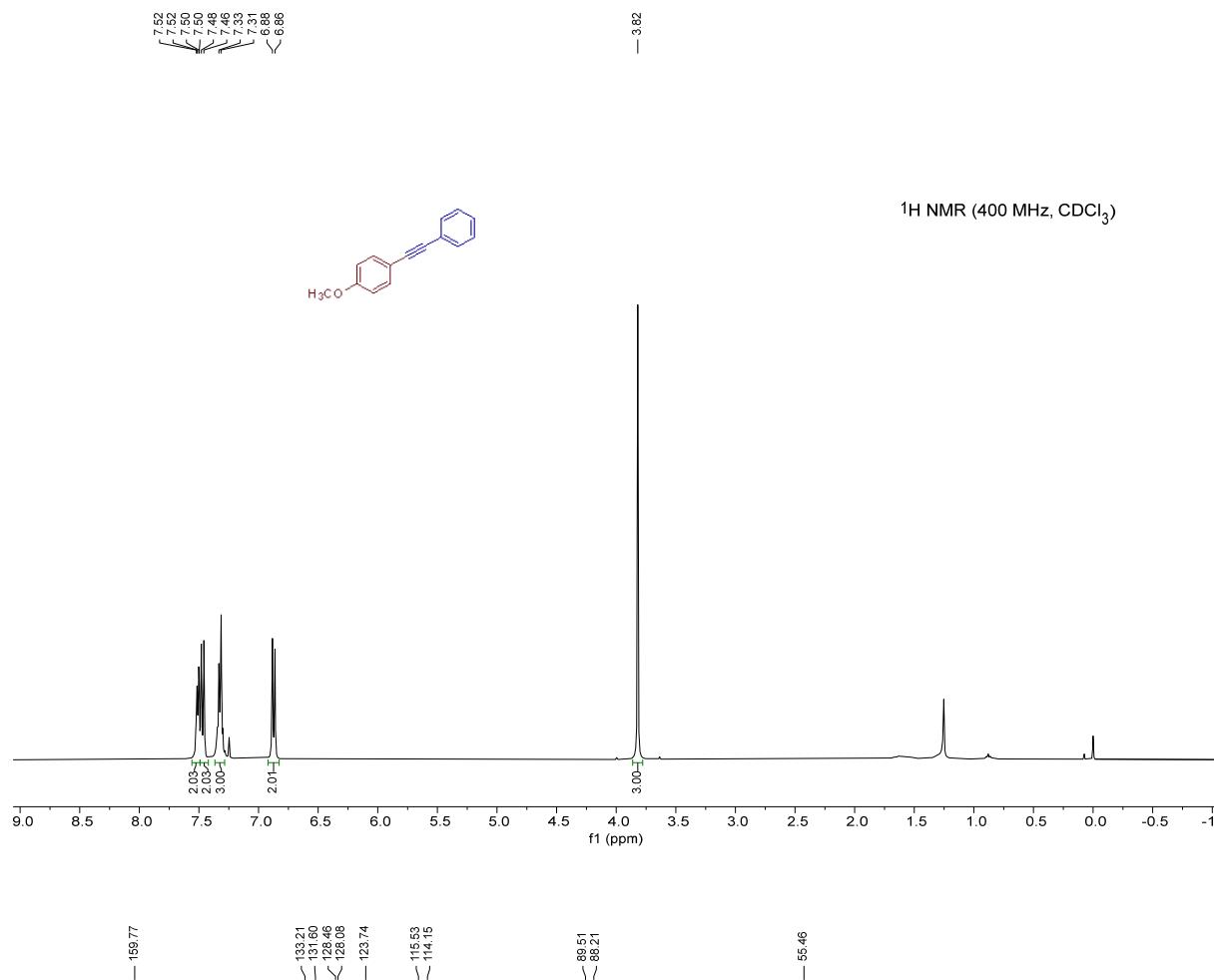


2. NMR spectral copies of the cross-coupling products

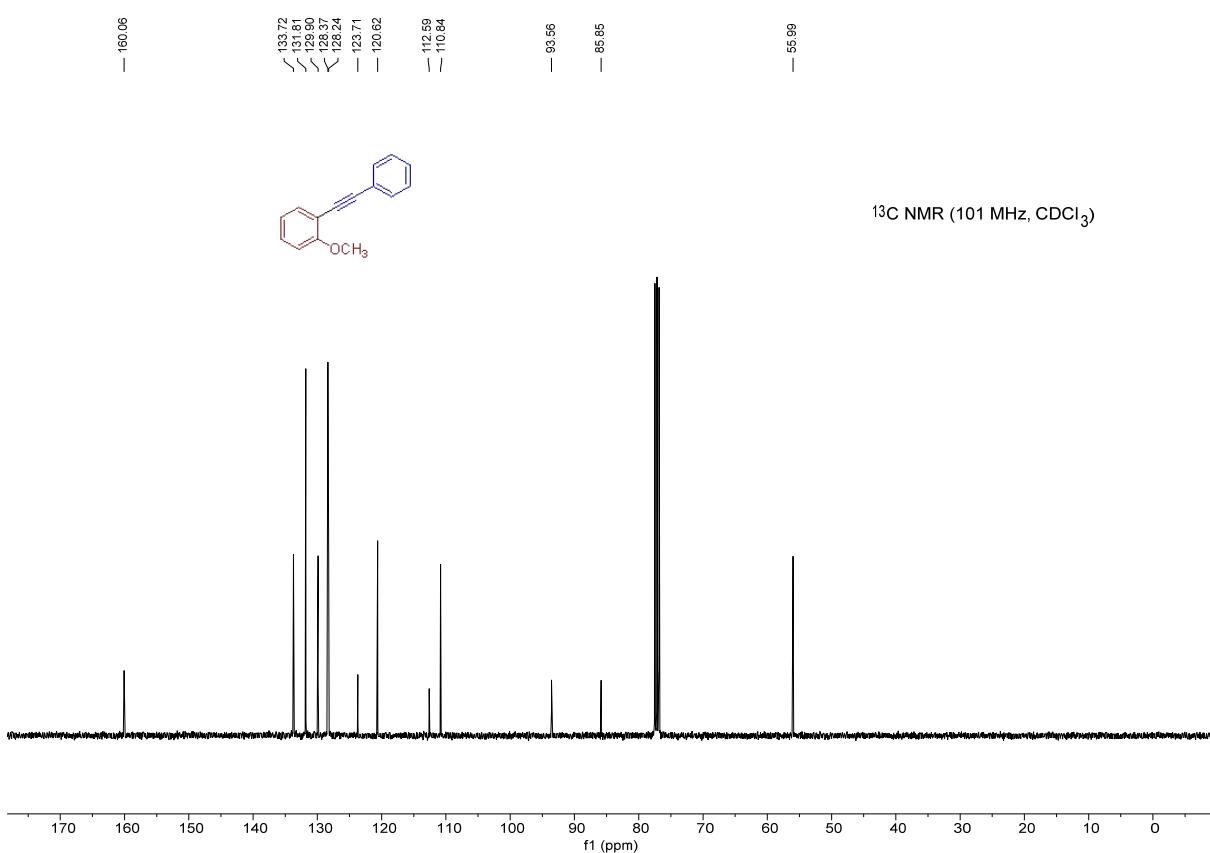
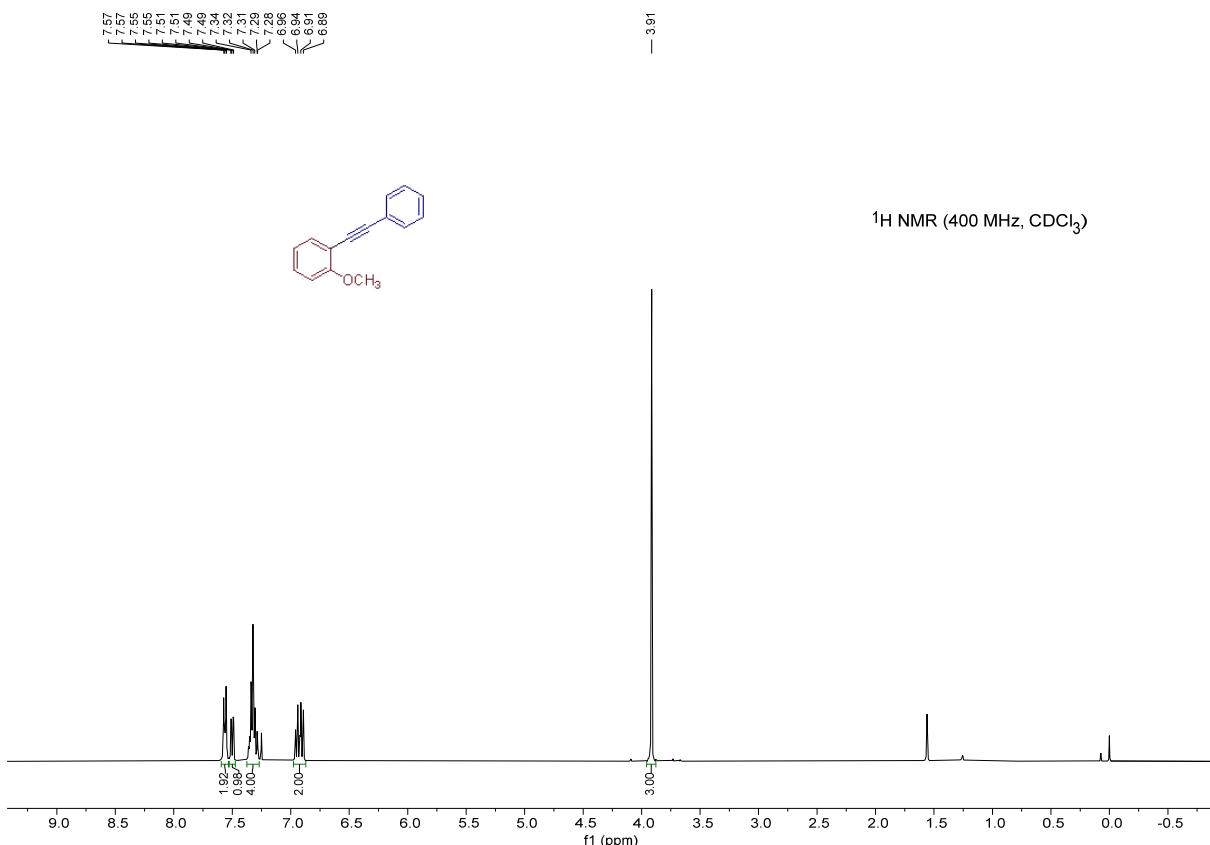
(1) 1-methyl-4-(phenylethyynyl)benzene (**3-1**) and 1,4-diphenylbuta-1,3-diyne



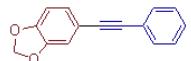
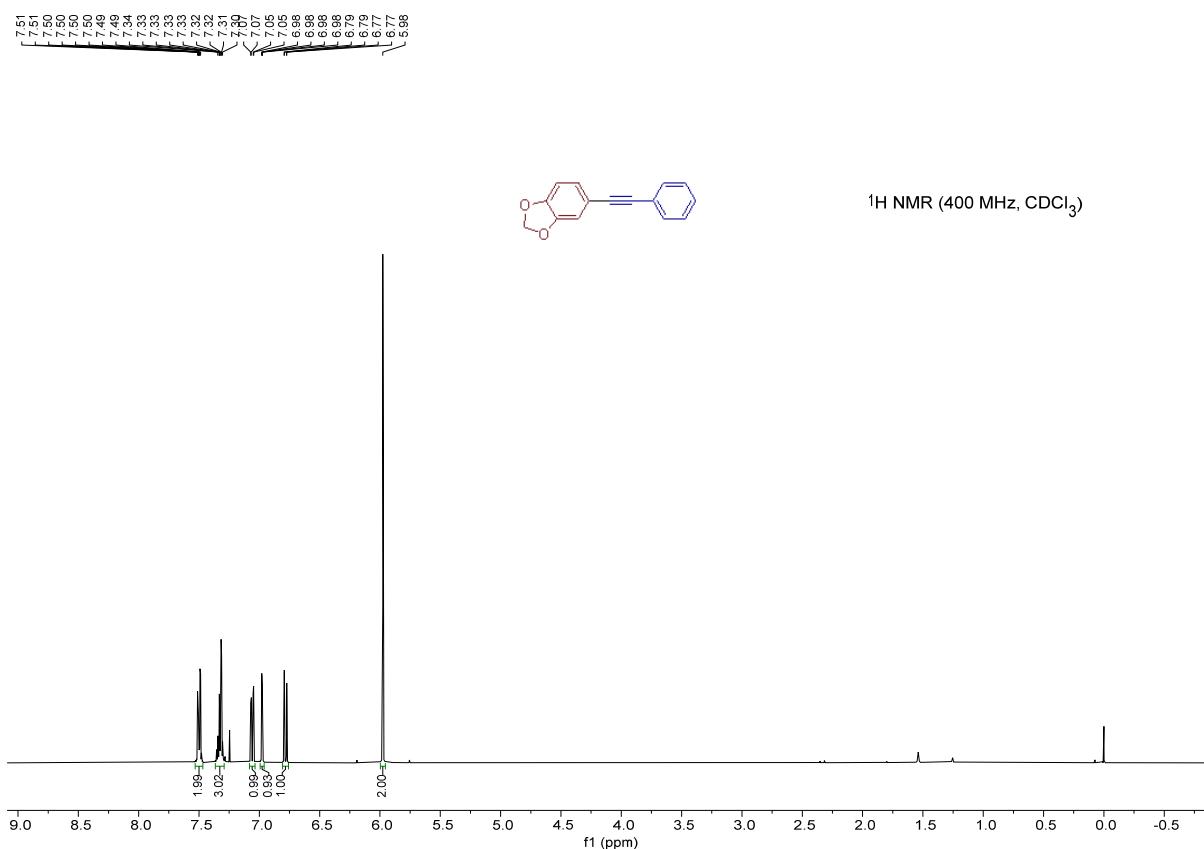
(2) 1-methoxy-4-(phenylethynyl)benzene (**3-2**)



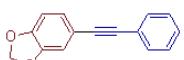
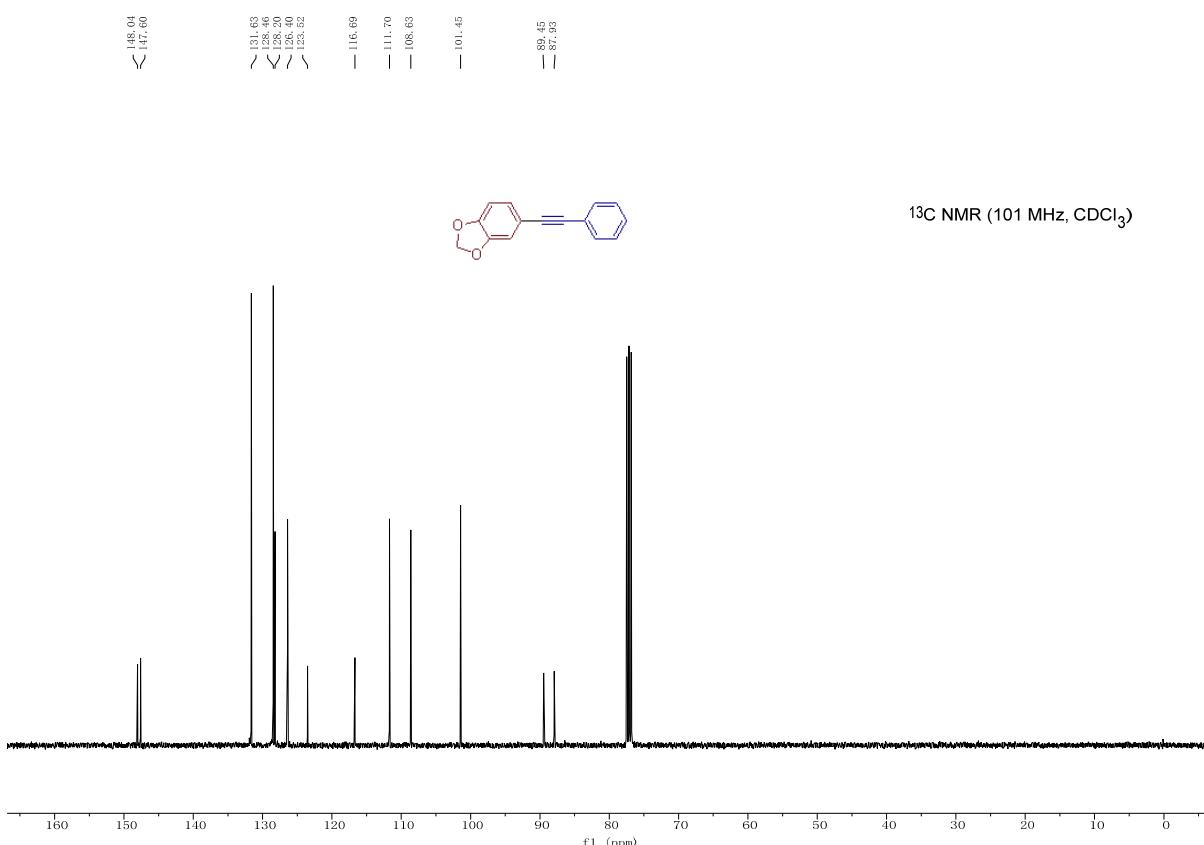
(3) 1-methoxy-2-(phenylethyynyl)benzene (**3-3**)



**(4) 5-(phenylethynyl)-1,3-benzodioxole (**3-4**)**

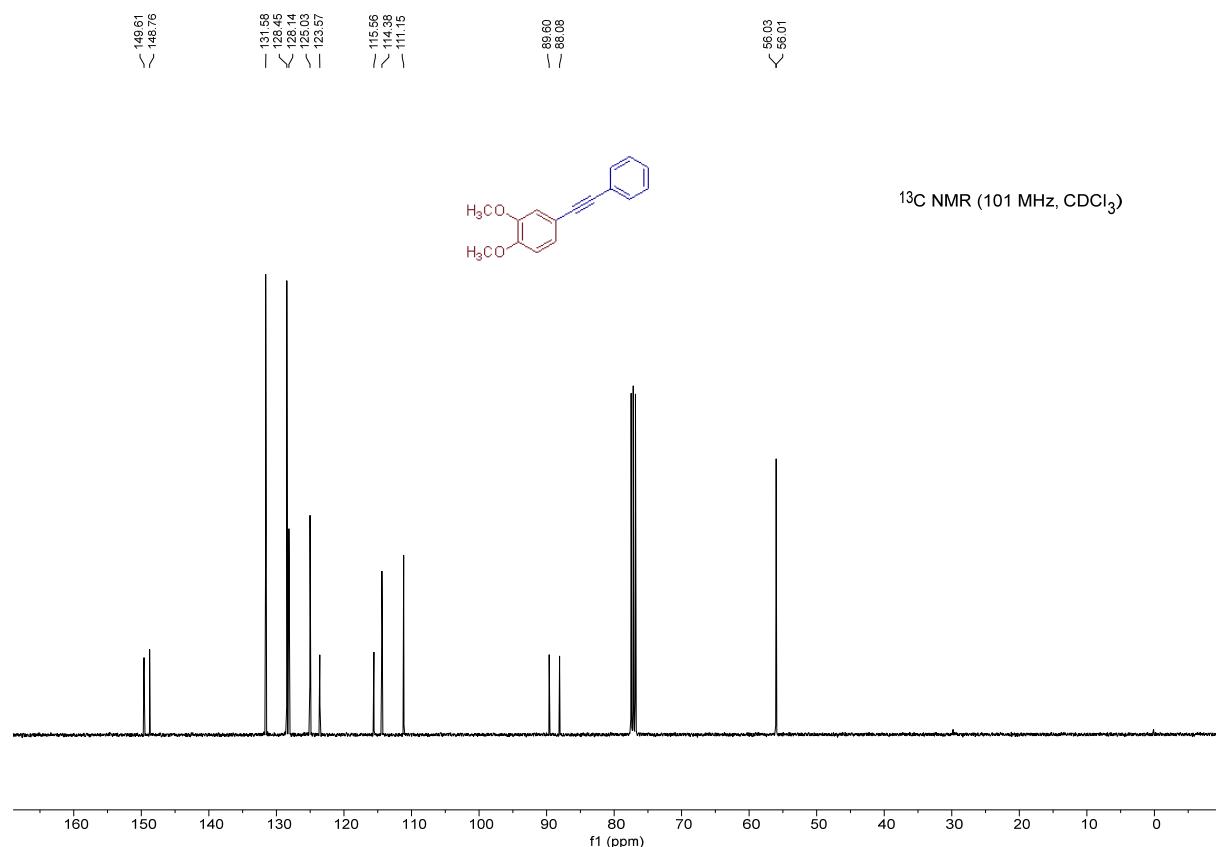
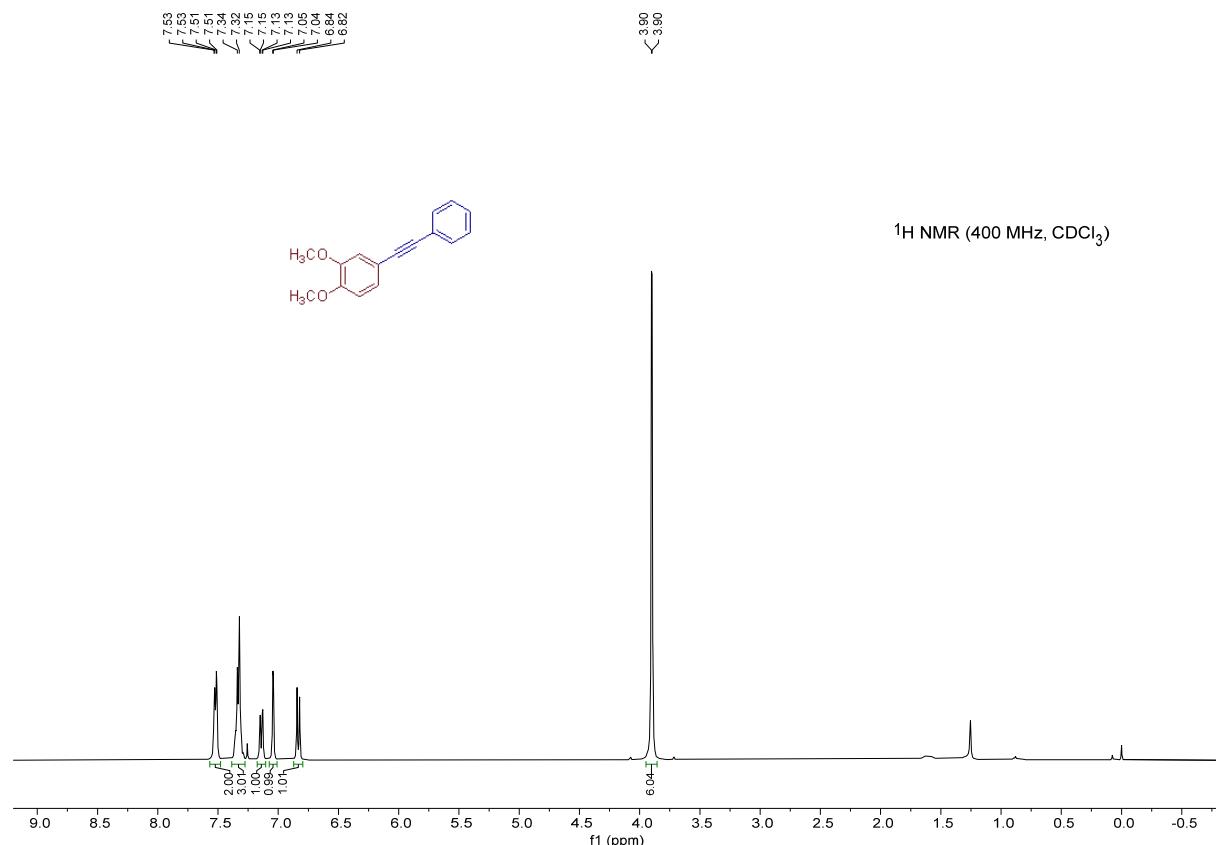


<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

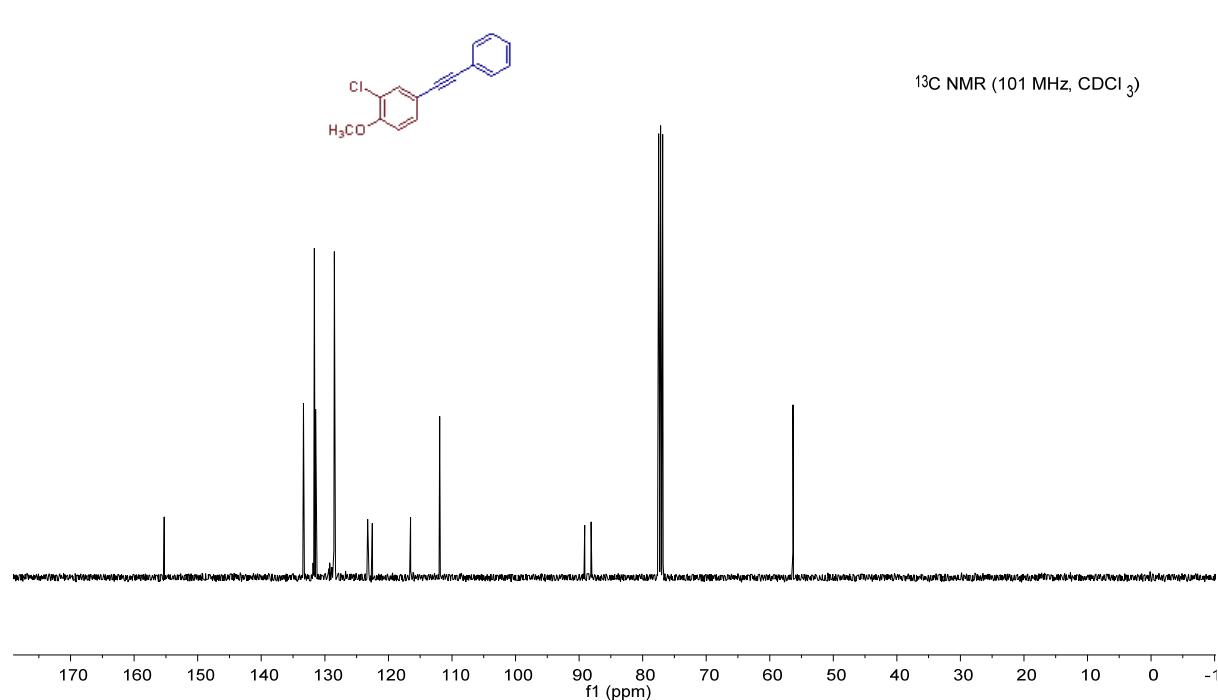
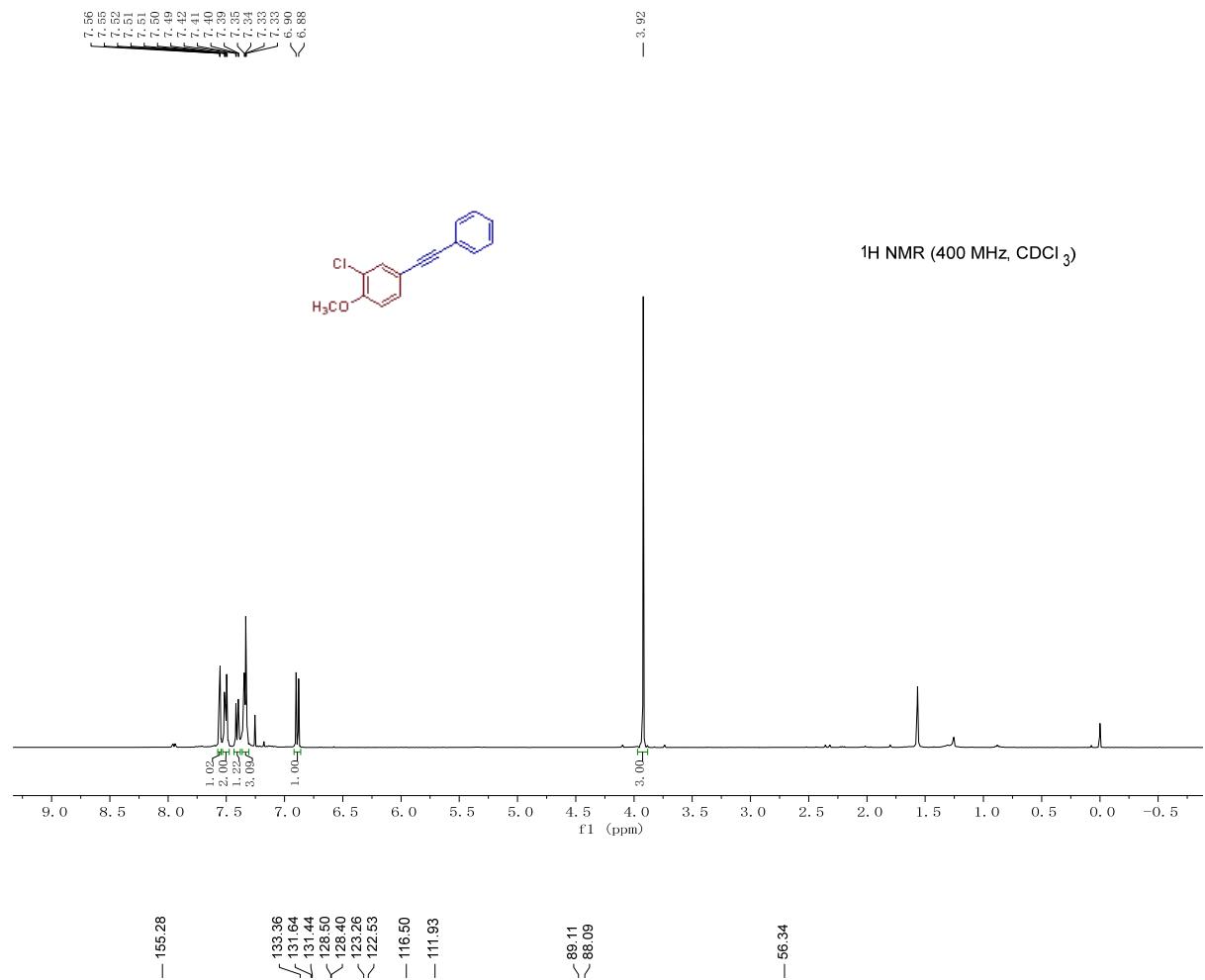


<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)

(5) 1,2-dimethoxy-4-(phenylethyynyl)benzene (**3-5**)



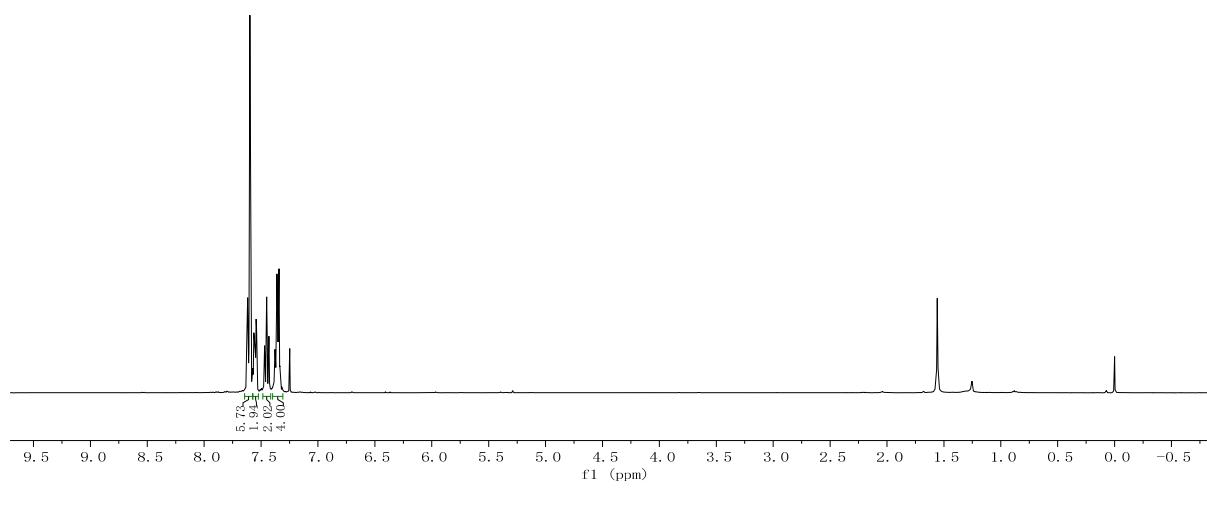
(6) 2-chloro-1-methoxy-4-(phenylethynyl)benzene (**3-6**)



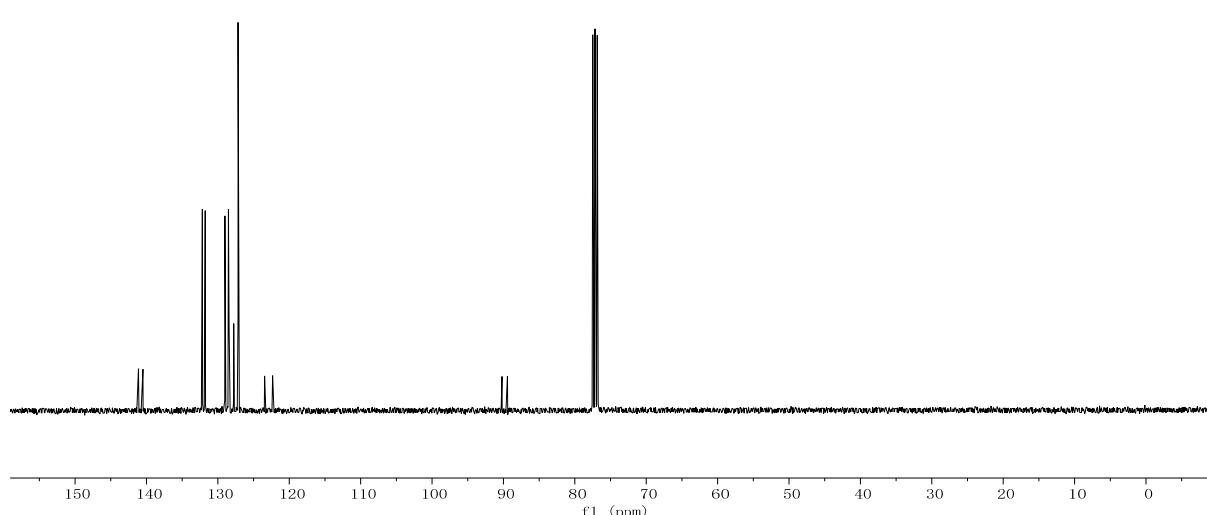
(7) 1-phenyl-4-(phenylethynyl)benzene (**3-7**)



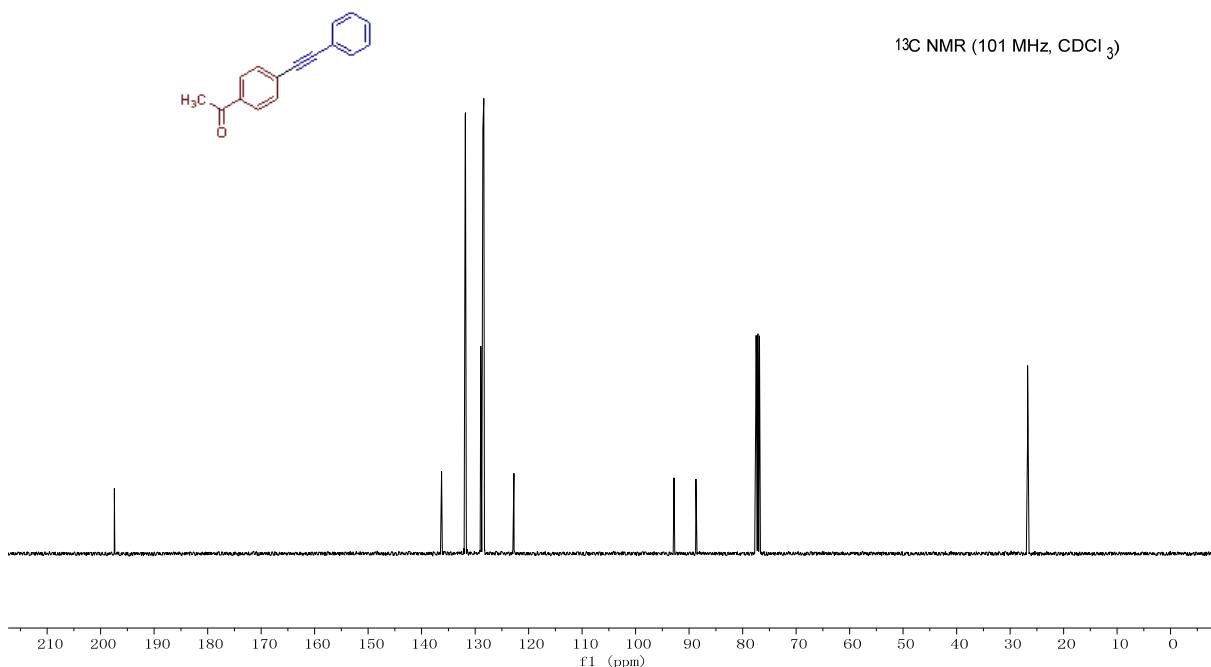
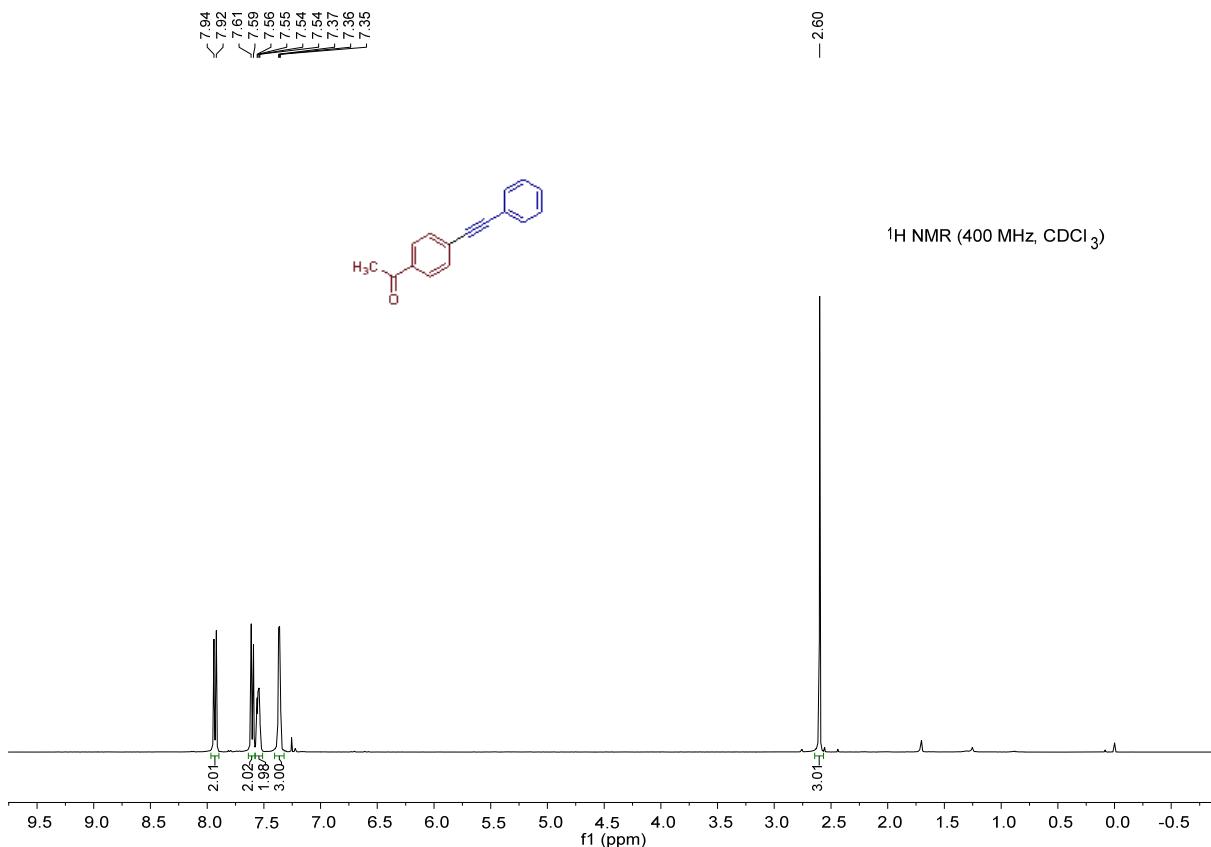
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



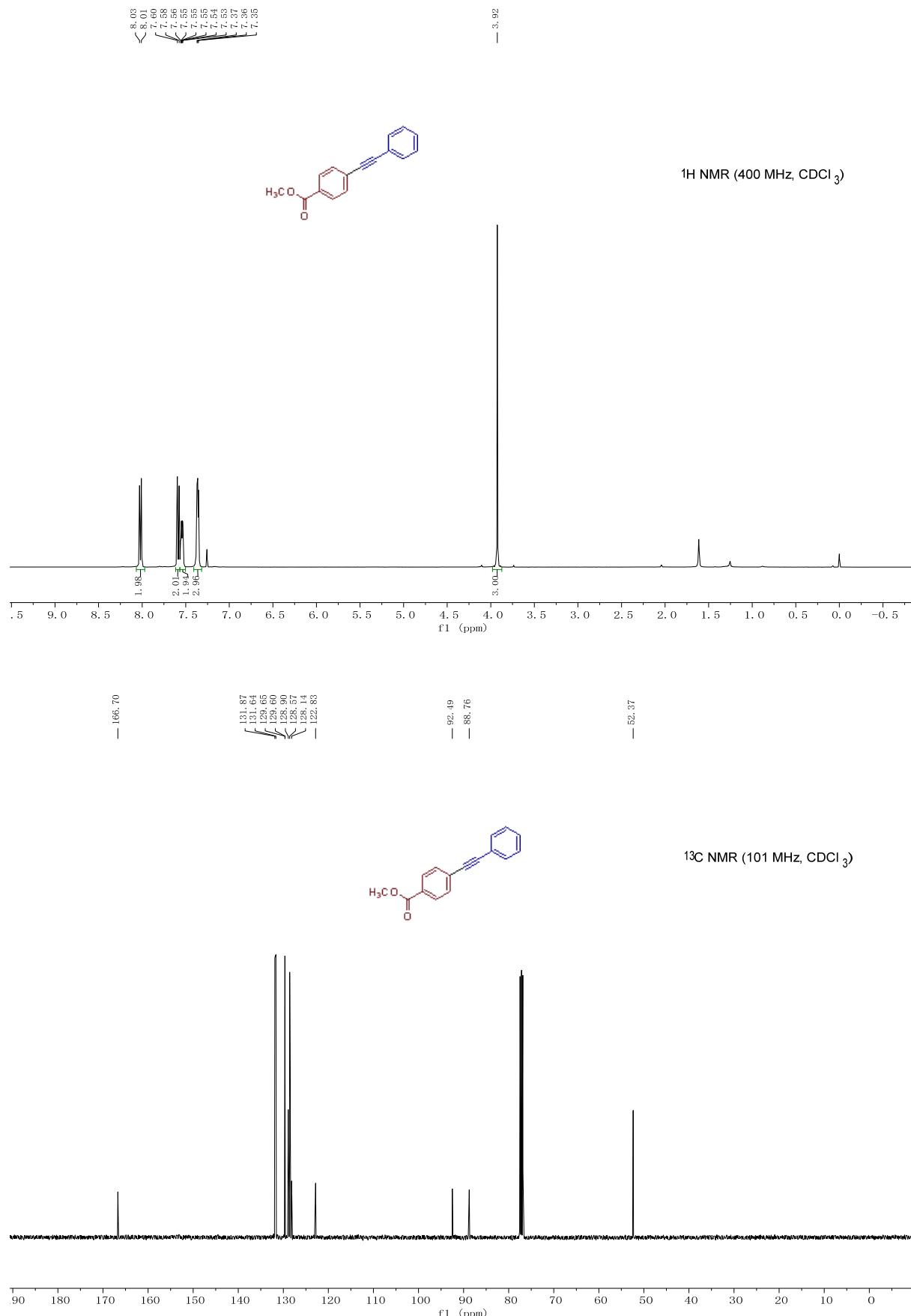
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)



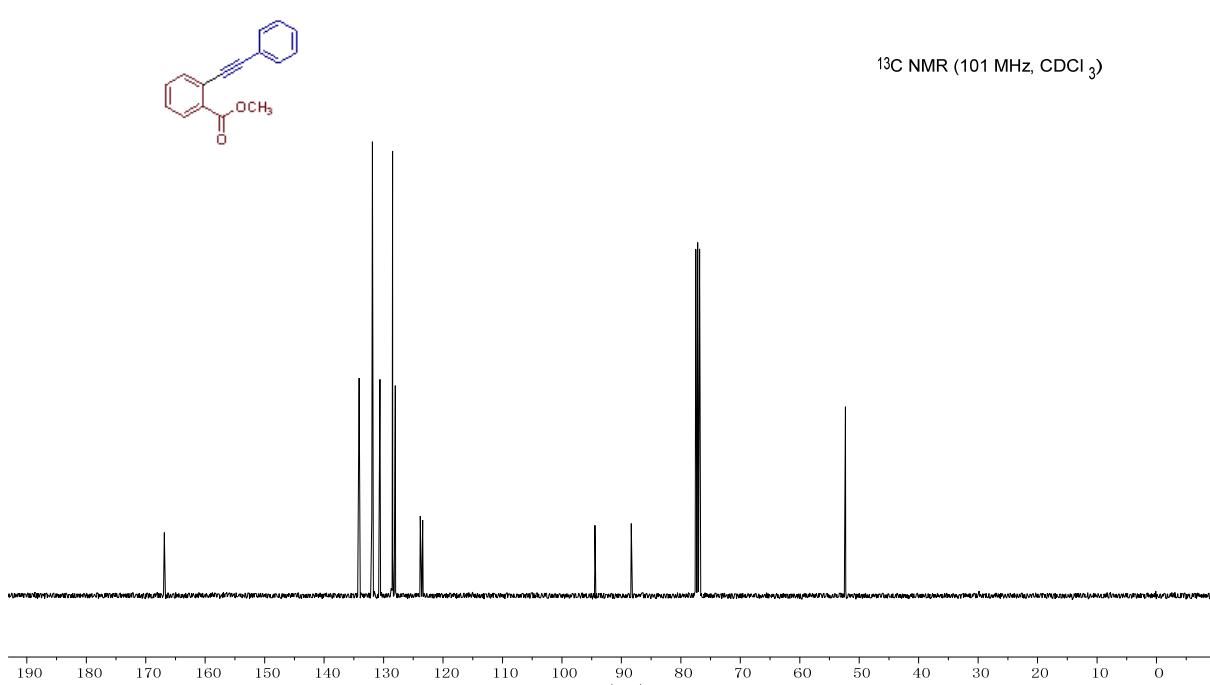
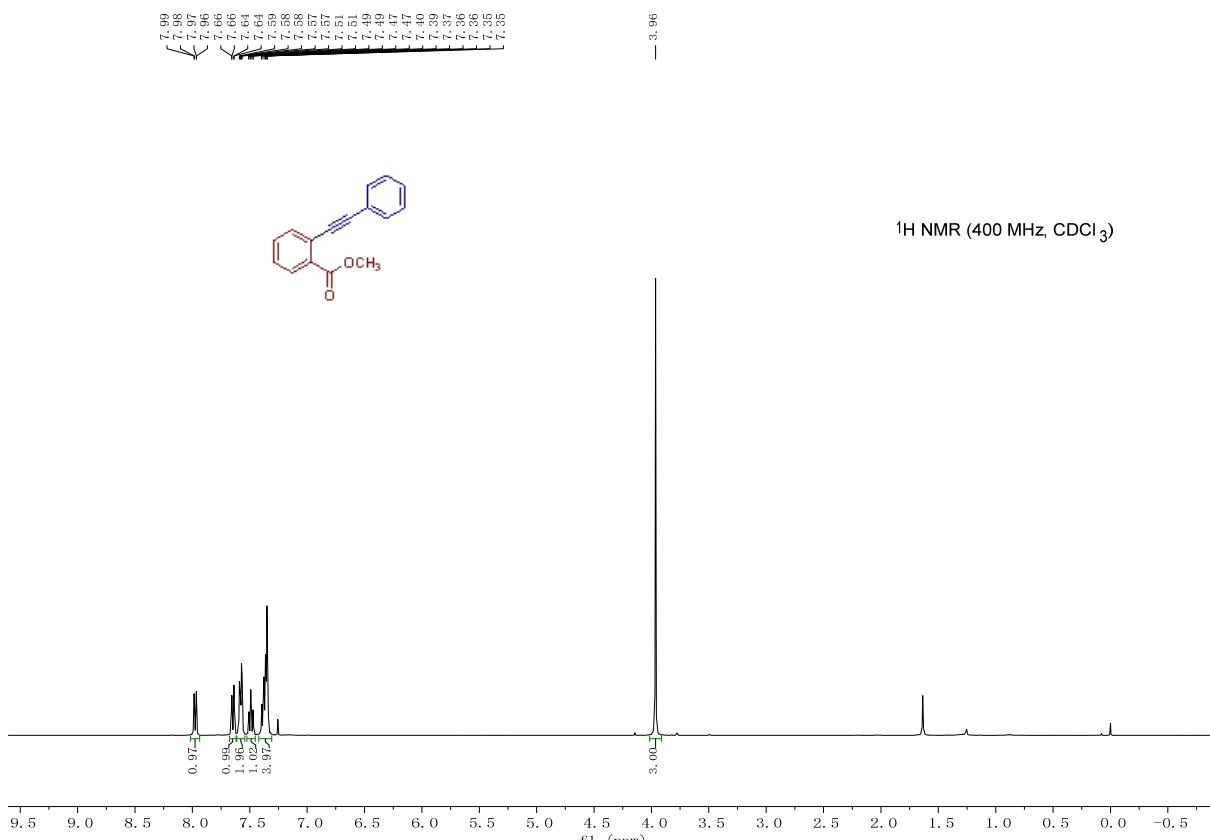
(8) 4-(phenylethynyl)acetophenone (**3-8**)



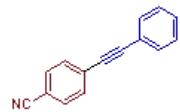
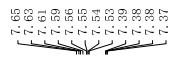
(9) methyl 4-(phenylethynyl)benzoate (**3-9**)



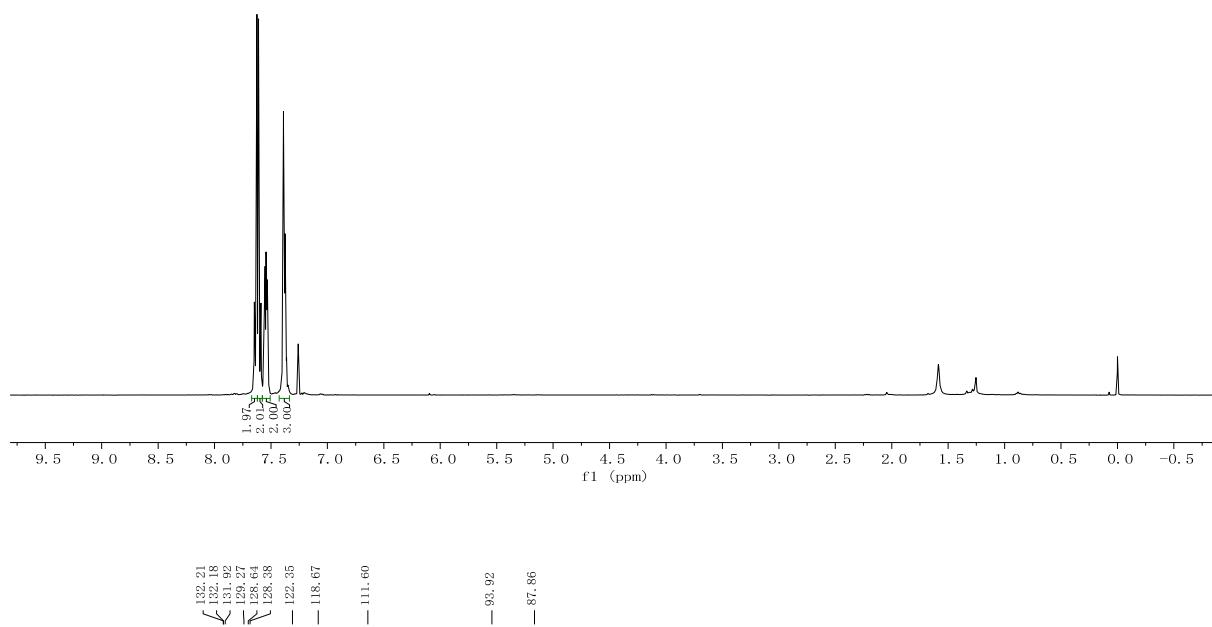
(10) methyl 2-(phenylethyynyl)benzoate (**3-10**)



(11) 4-(2-phenylethynyl)benzonitrile (**3-11**)

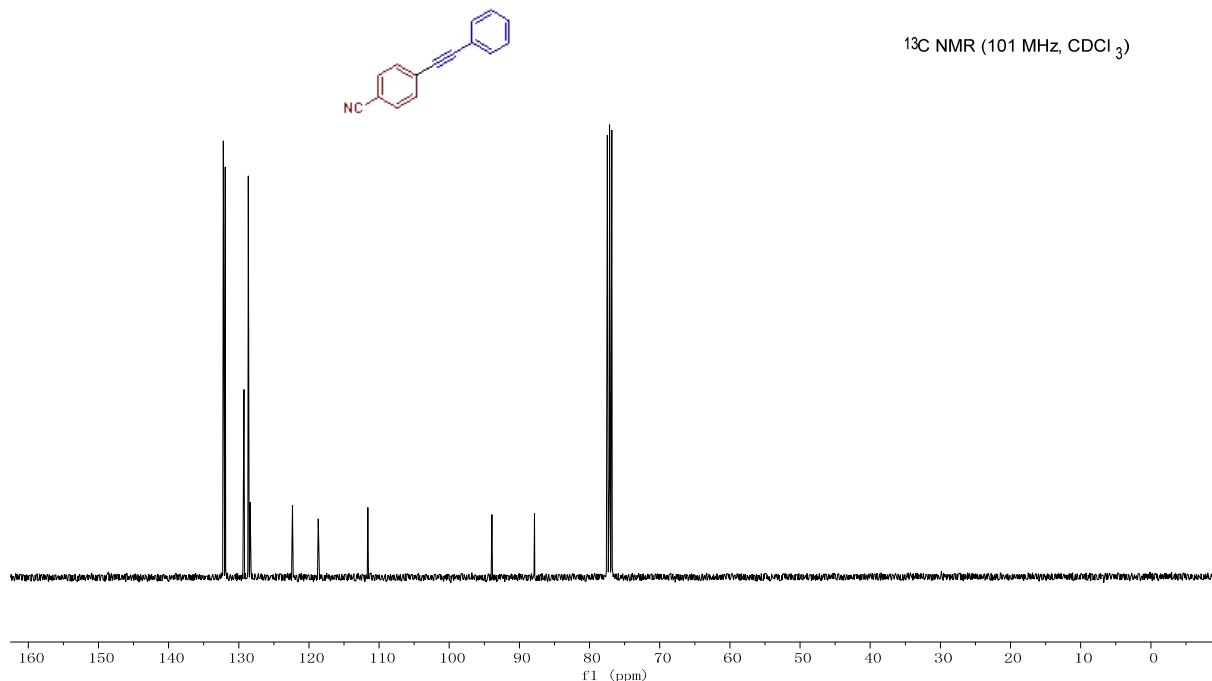


<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

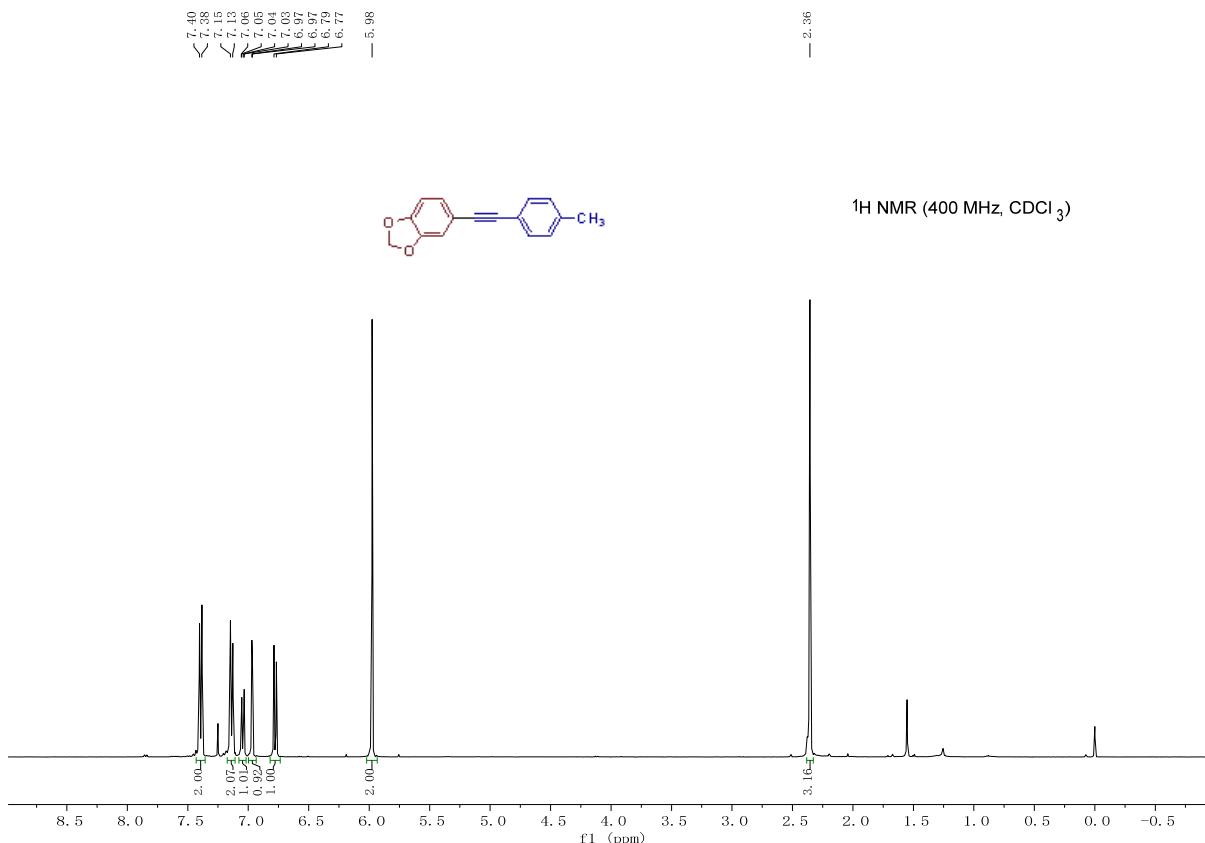


132.21  
132.18  
132.18  
131.92  
131.92  
139.27  
139.27  
128.64  
128.64  
128.38  
128.38  
122.35  
122.35  
118.67  
118.67  
— 111.60  
— 93.92  
— 87.86

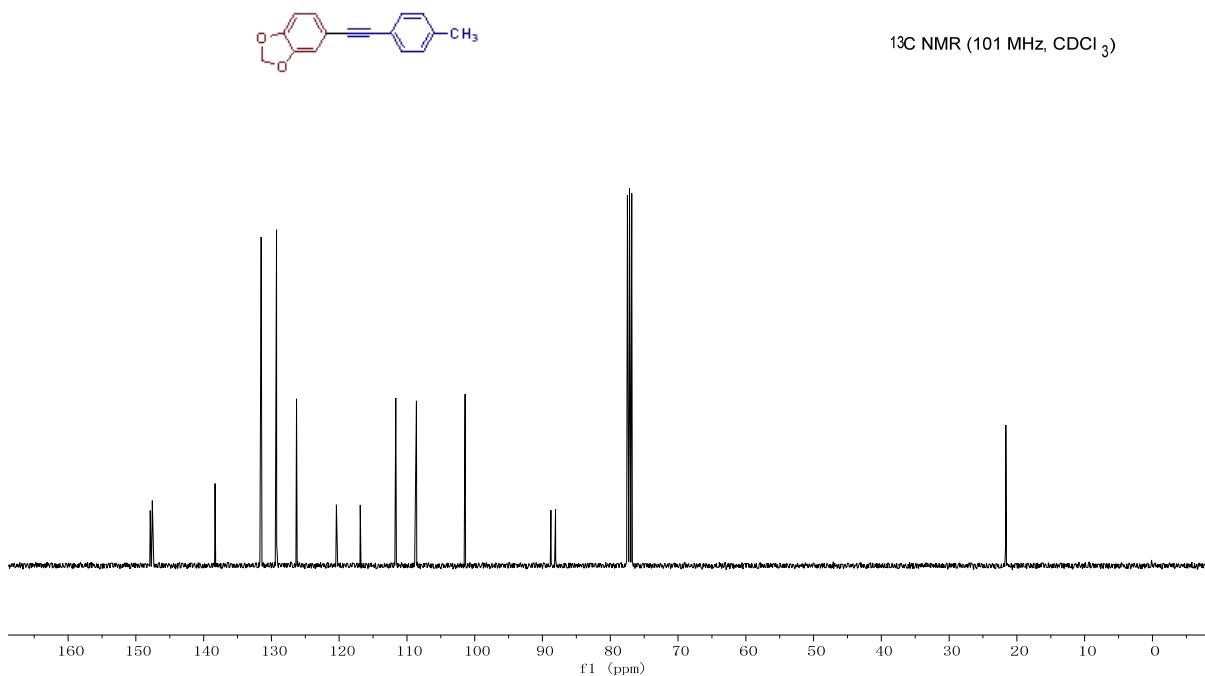
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)



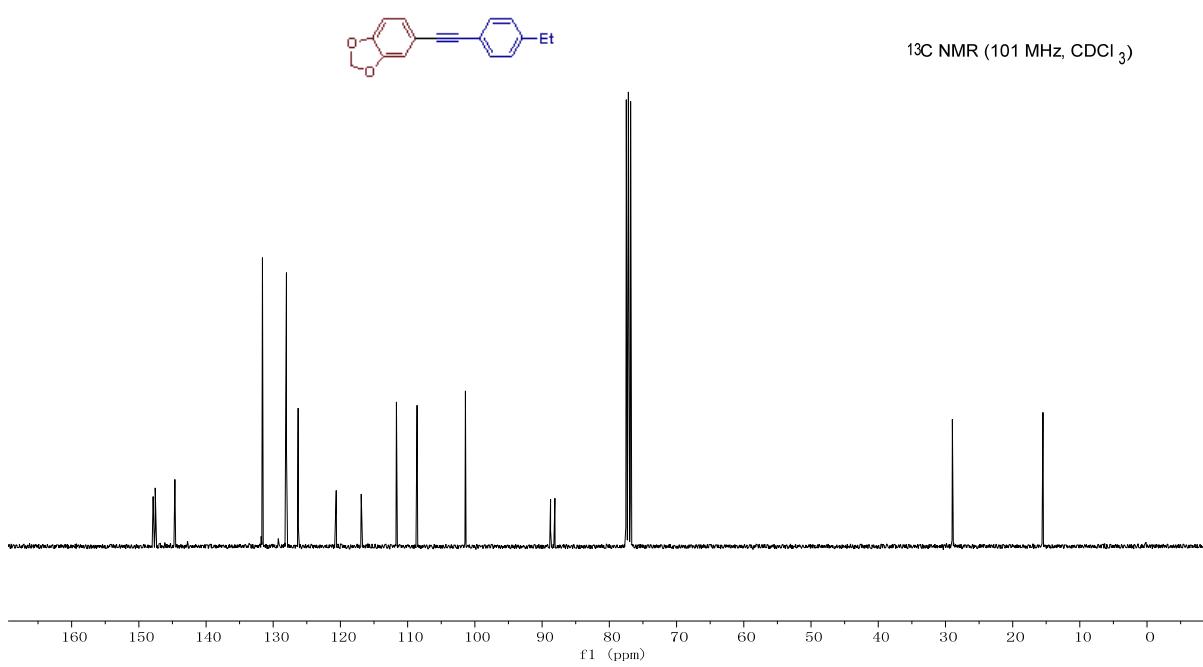
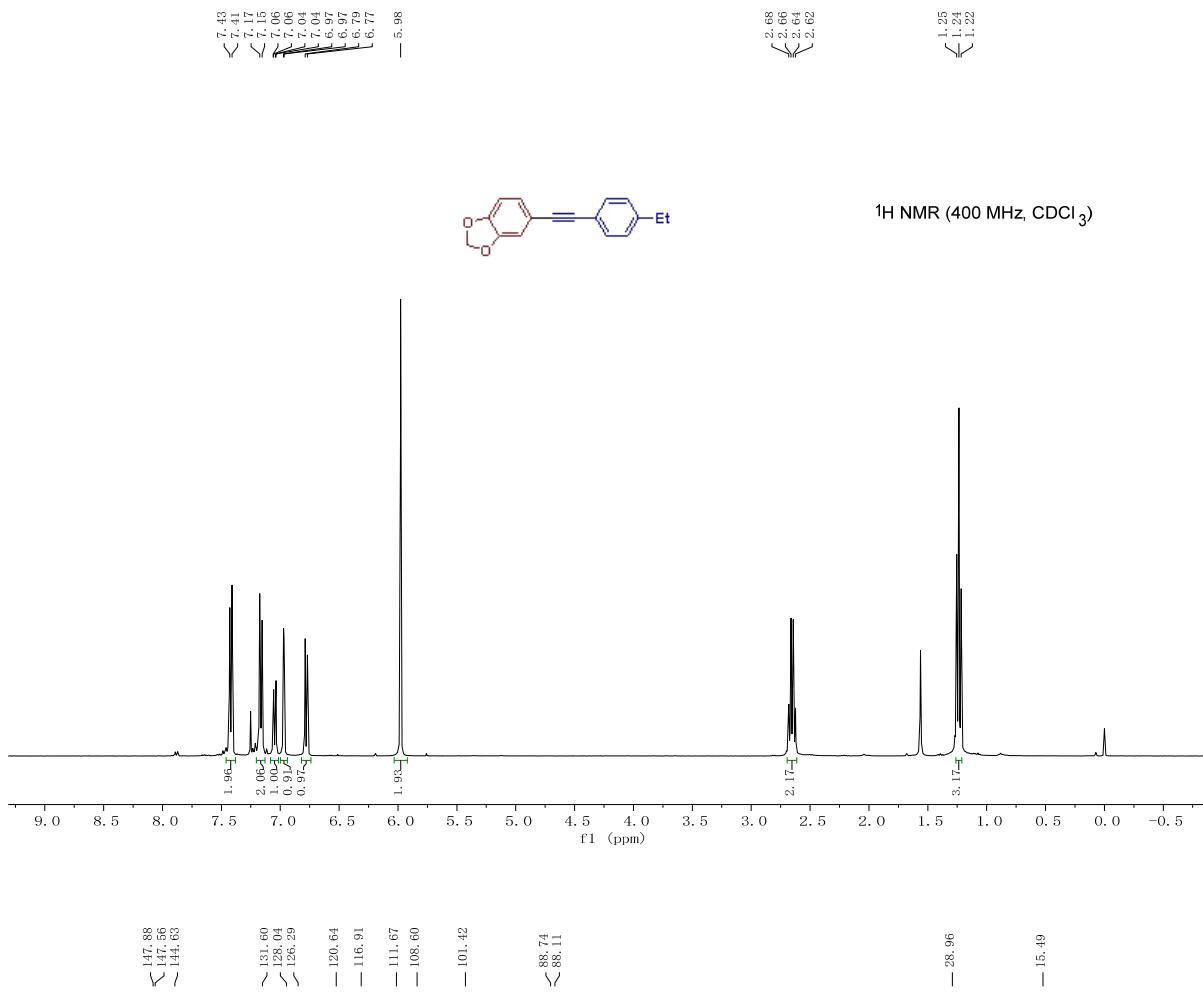
(12) 5-((4-methylphenyl)ethynyl)-1,3-benzodioxole (**3-12**)



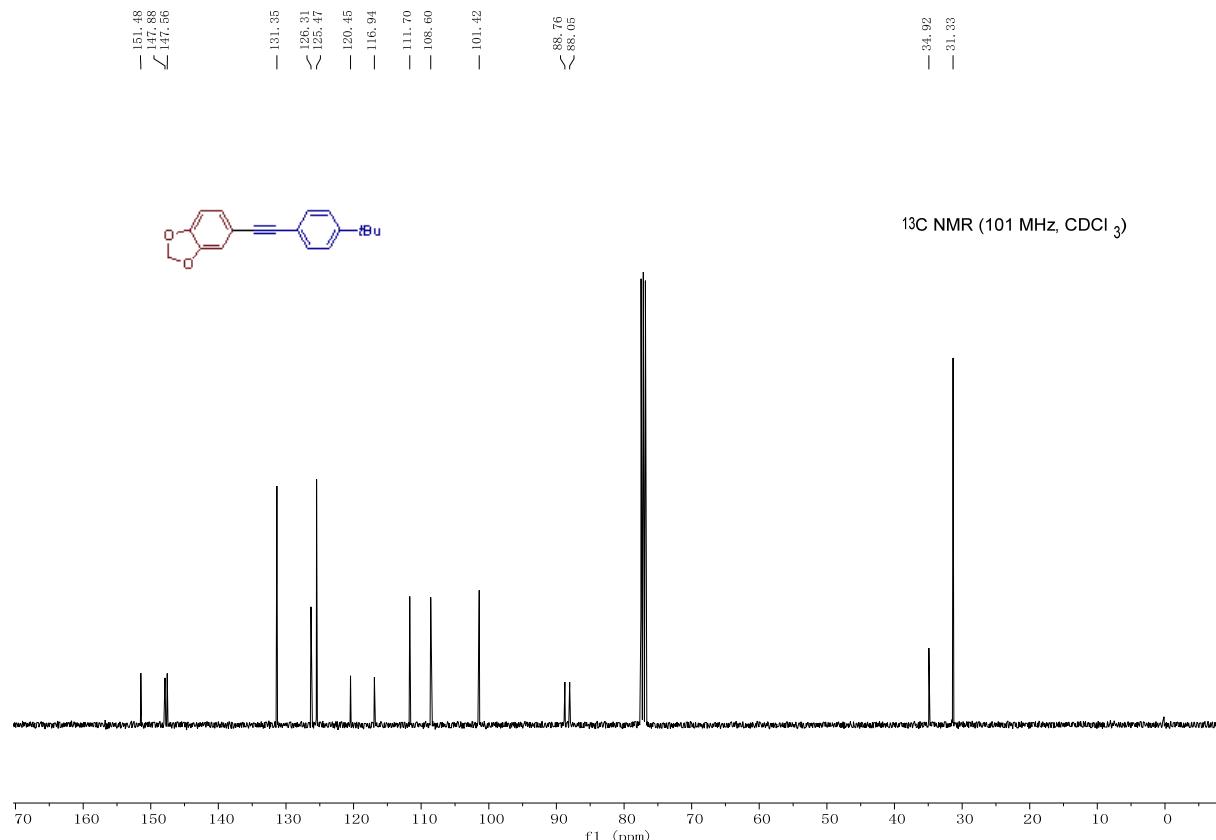
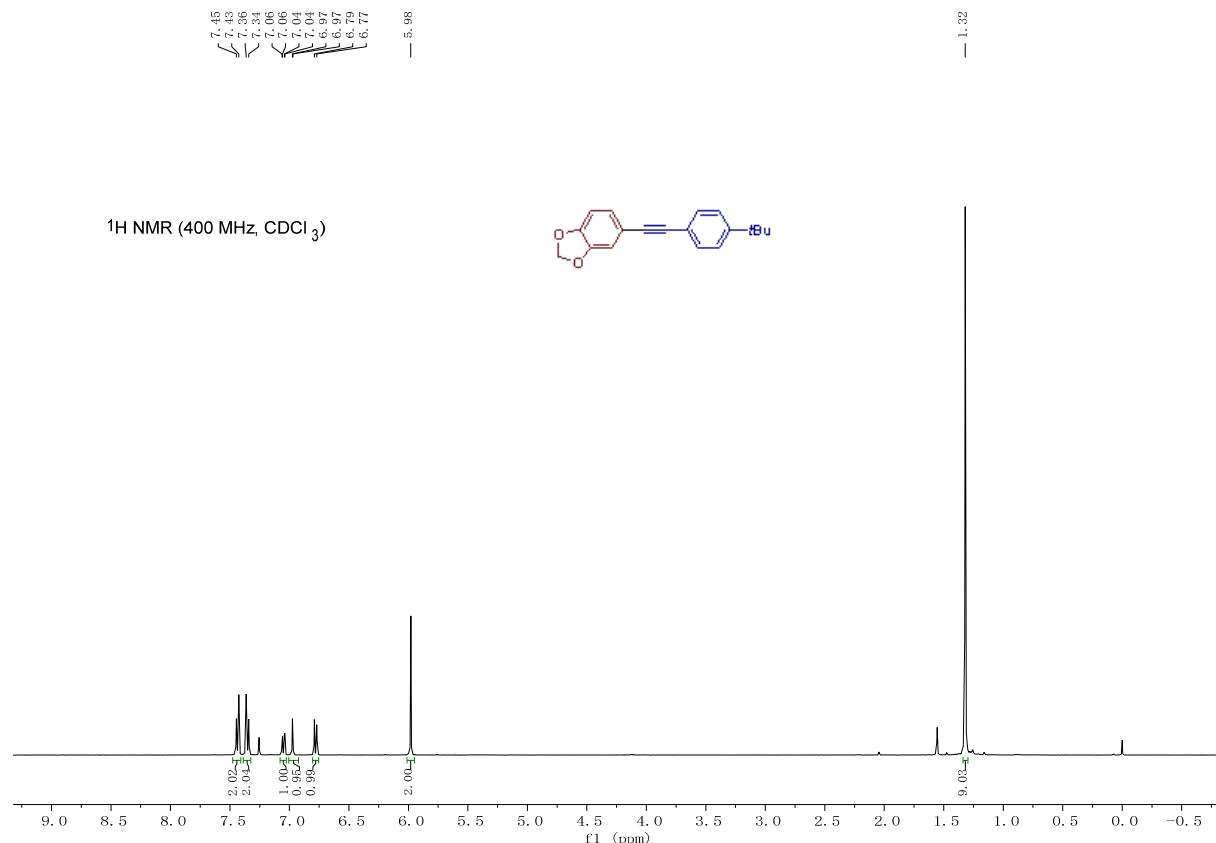
147.89  
147.56  
138.32  
131.51  
129.23  
126.28  
120.40  
116.89  
111.65  
108.60  
101.42  
88.76  
88.08  
21.63



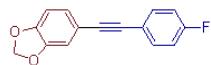
(13) 5-((4-ethylphenyl)ethynyl)-1,3-benzodioxole (3-13)



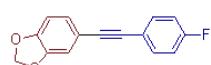
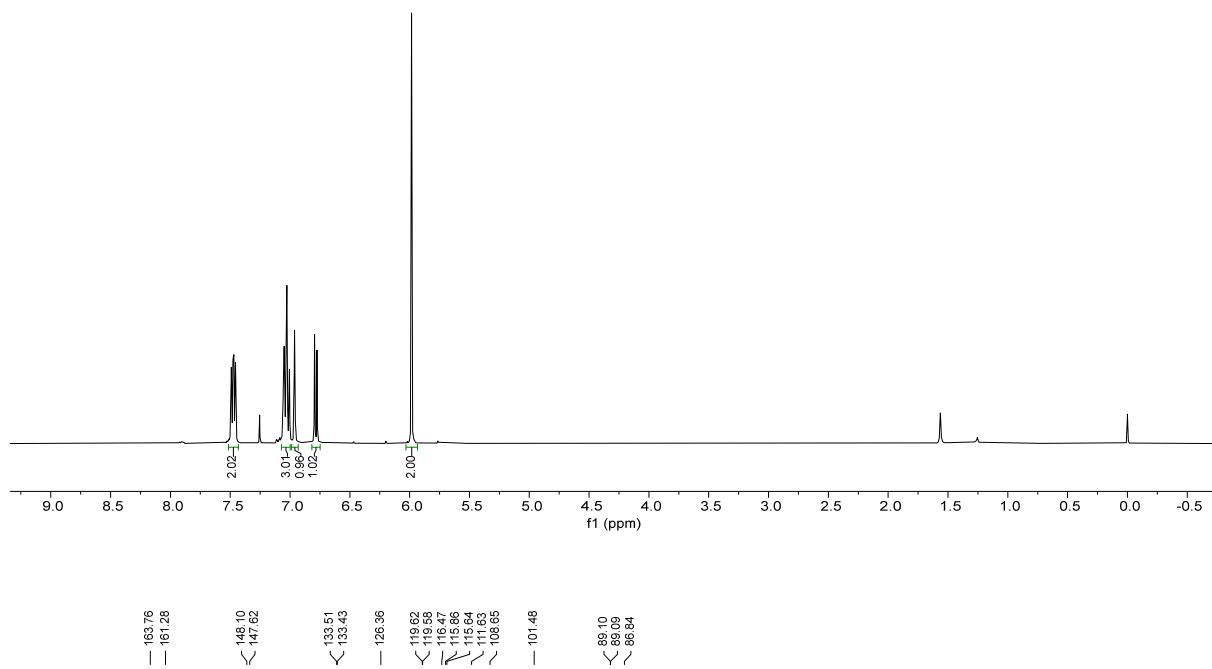
(14) 5-((4-*tert*-butylphenyl)ethynyl)-1,3-benzodioxole (**3-14**)



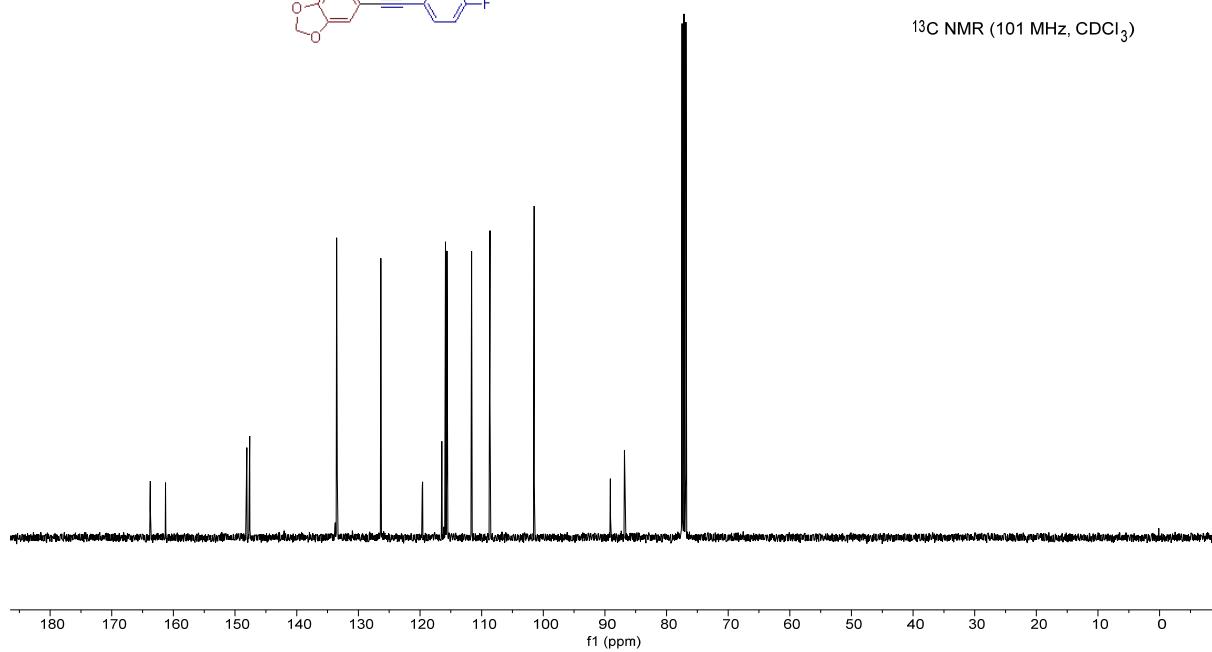
(15) 5-((4-fluorophenyl)ethynyl)-1,3-benzodioxole (**3-15**)

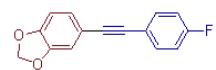


<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

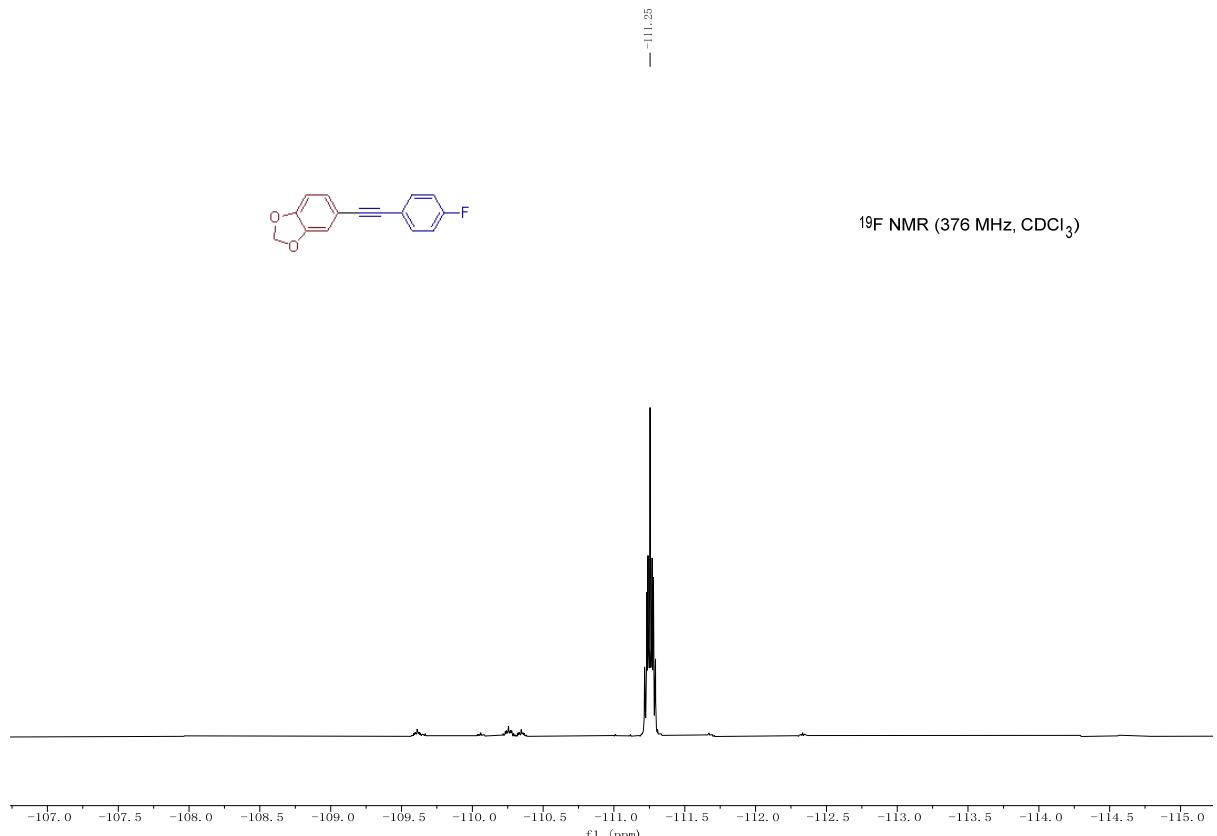


<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)



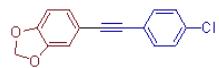


<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)

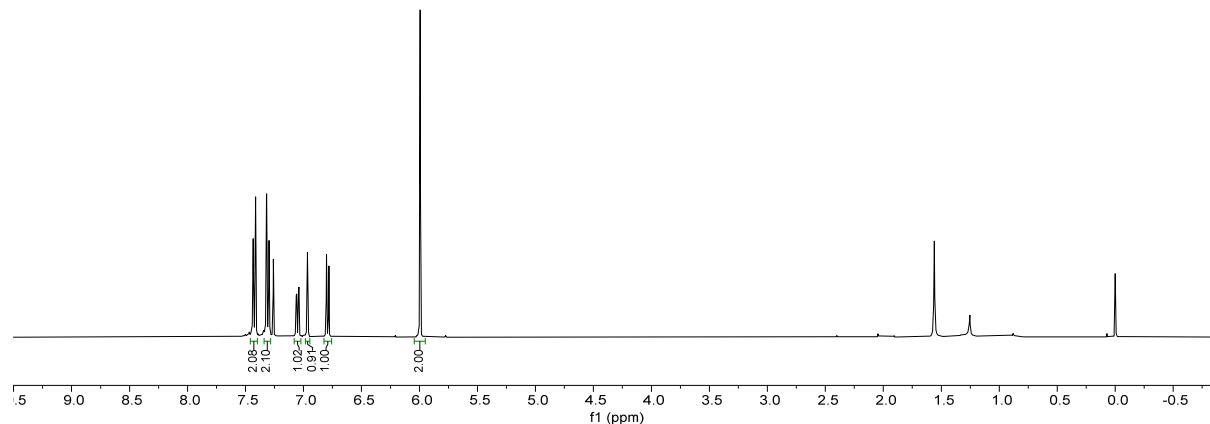


(16) 5-((4-chlorophenyl)ethynyl)-1,3-benzodioxole (**3-16**)

7.43  
7.41  
7.32  
7.30  
7.06  
7.06  
7.04  
7.04  
6.97  
6.96  
6.90  
6.88  
— 5.99

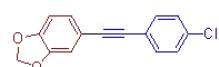


<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

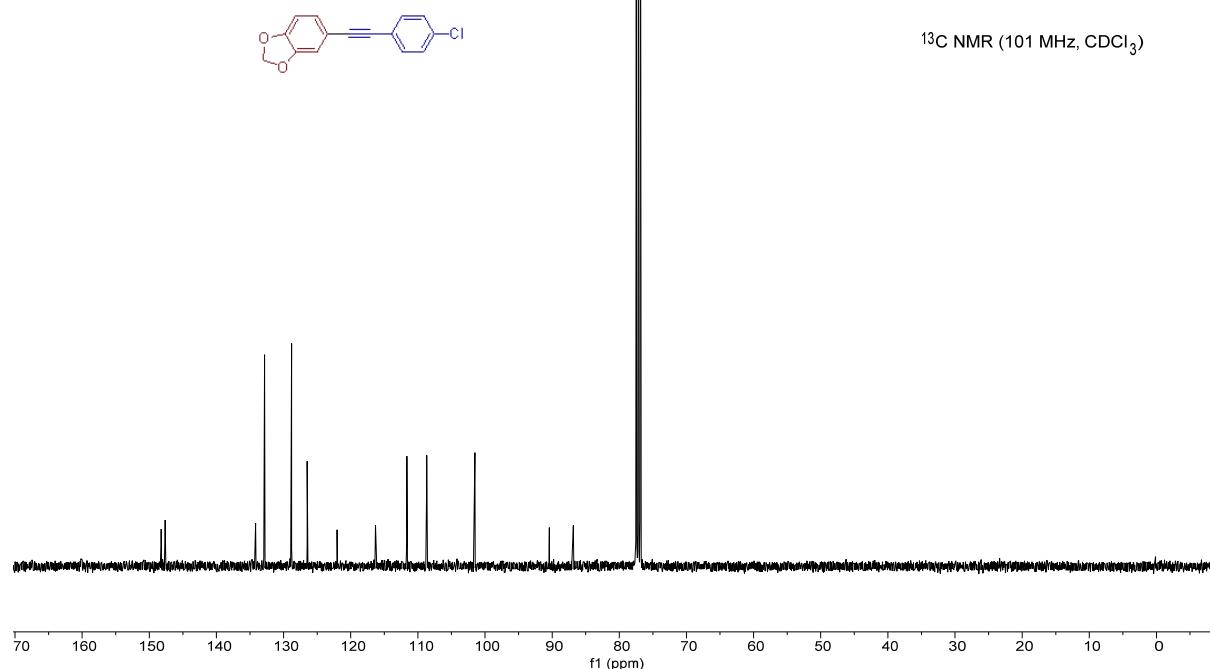


148.24  
147.65

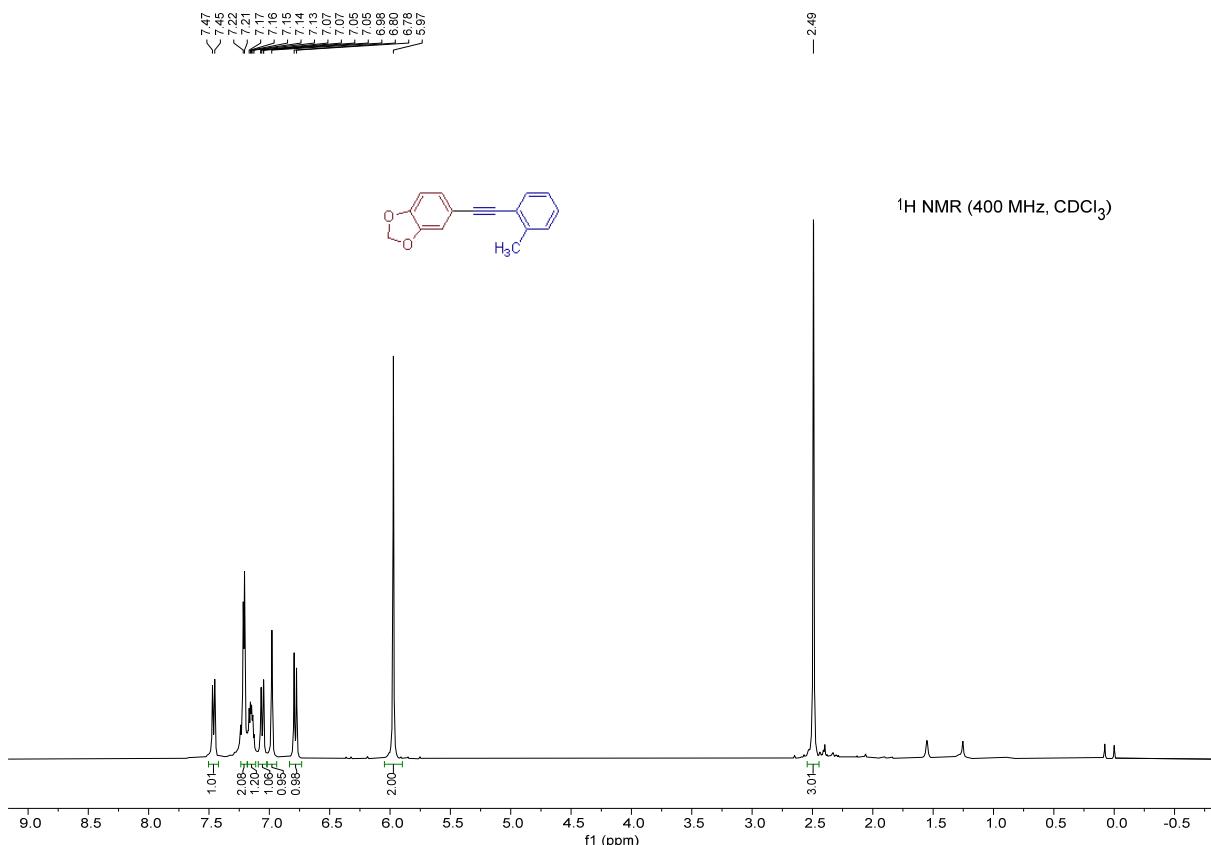
134.18  
132.82  
128.81  
128.48  
122.03  
116.31  
111.65  
108.69  
— 101.52



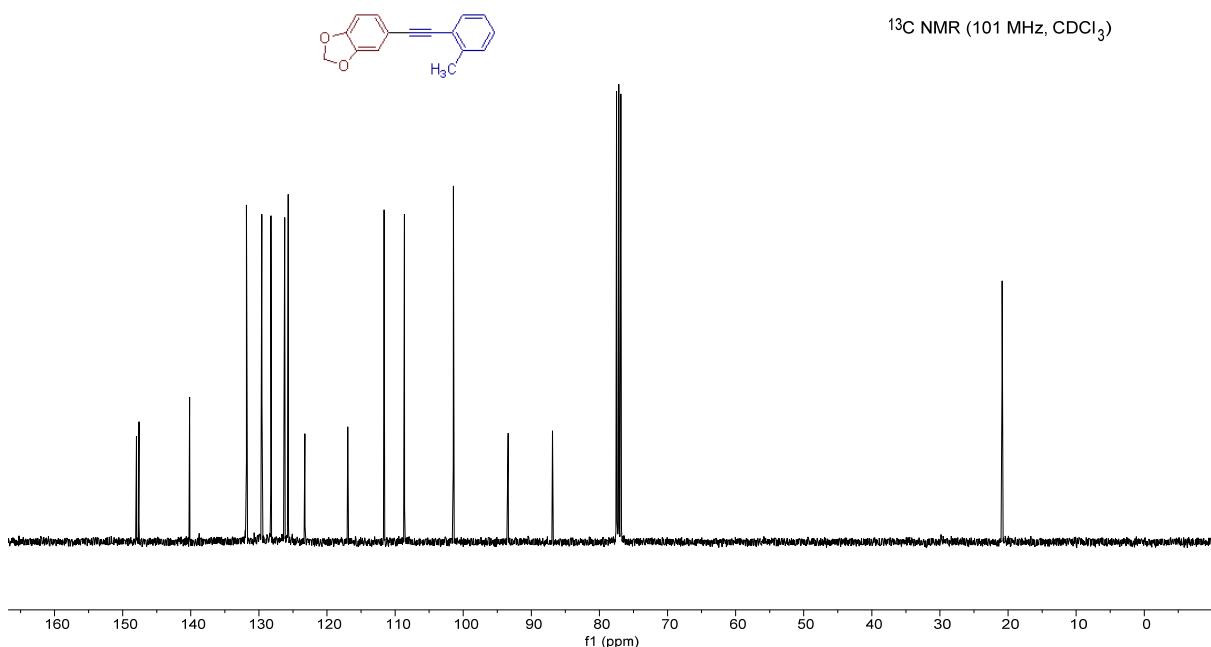
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)



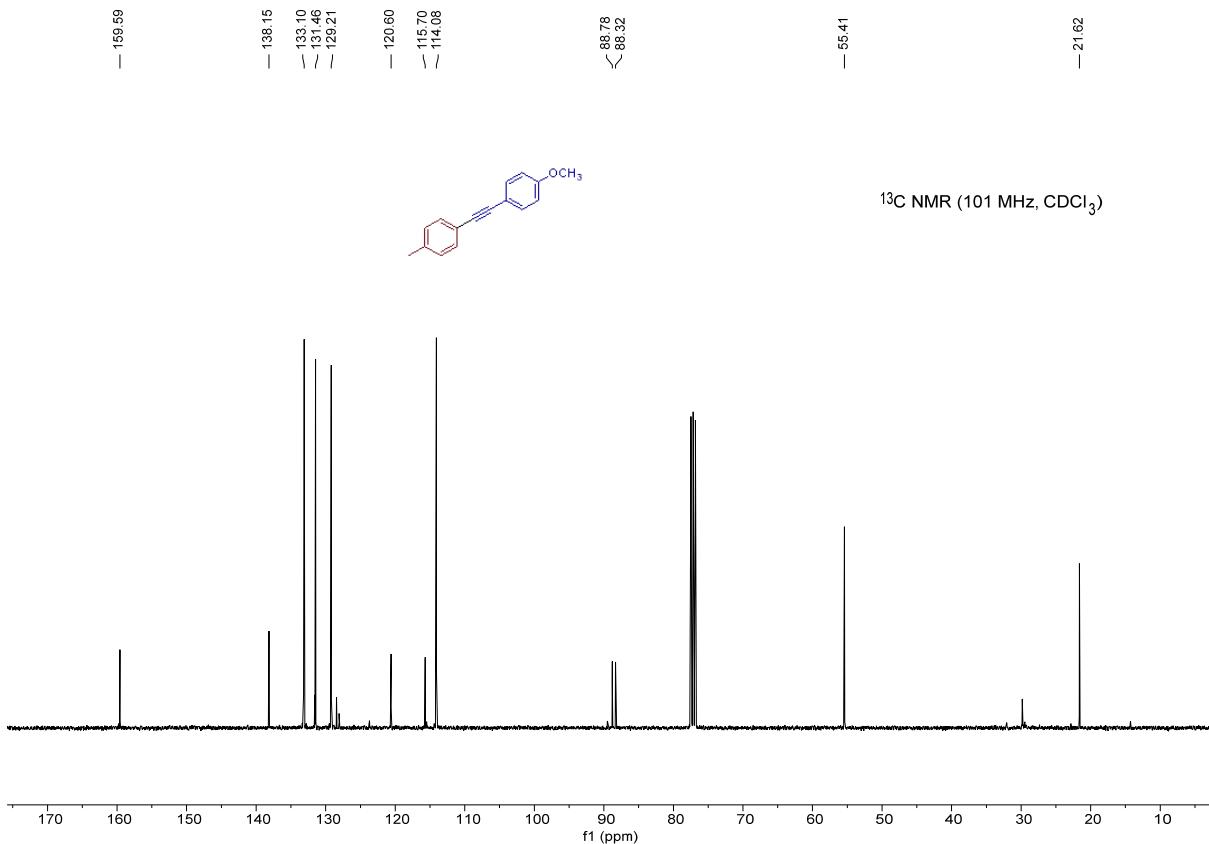
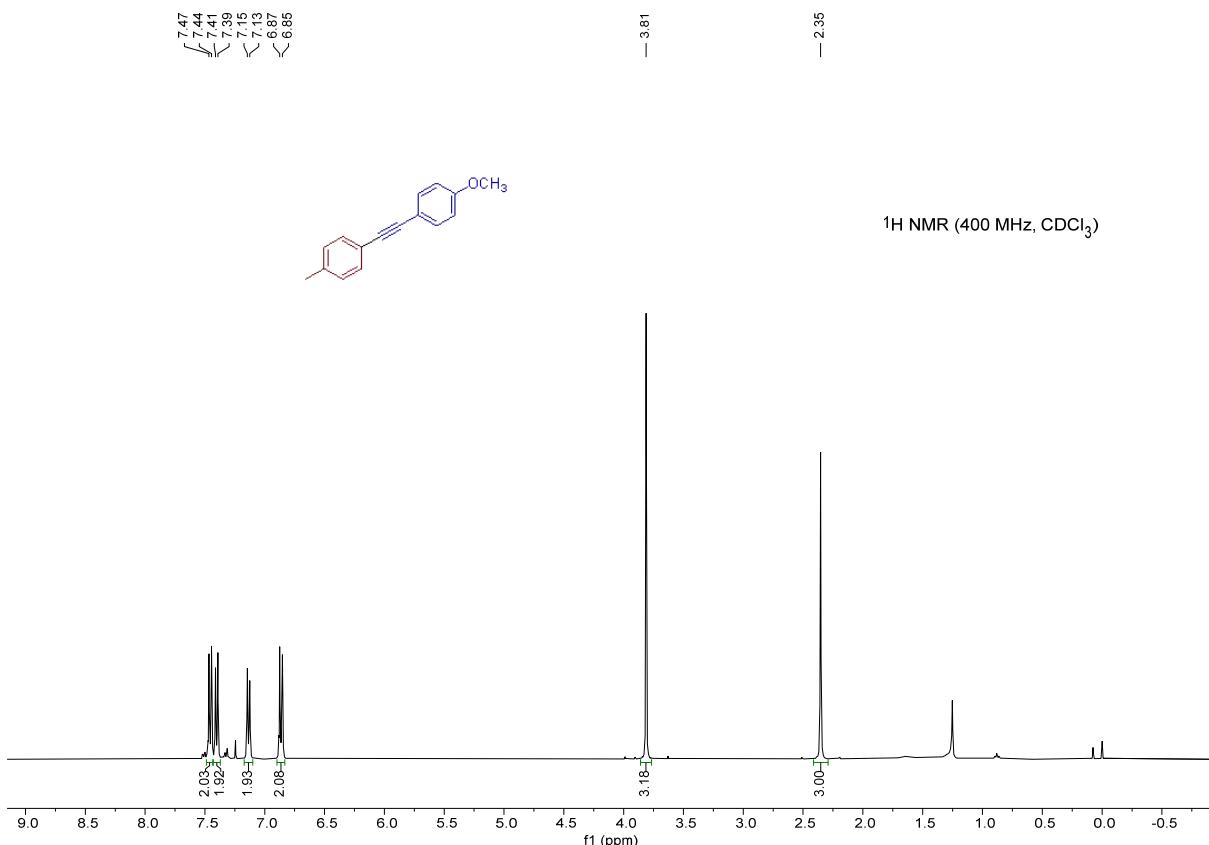
(17) 5-((2-methylphenyl)ethynyl)-1,3-benzodioxole (**3-18**)



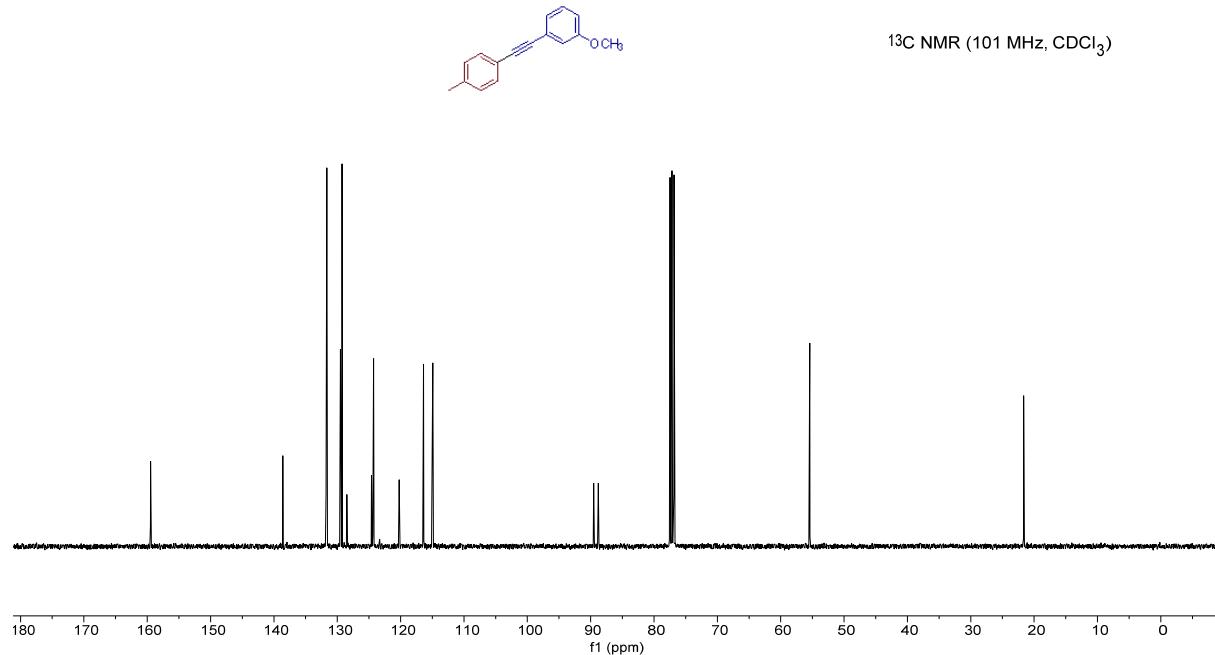
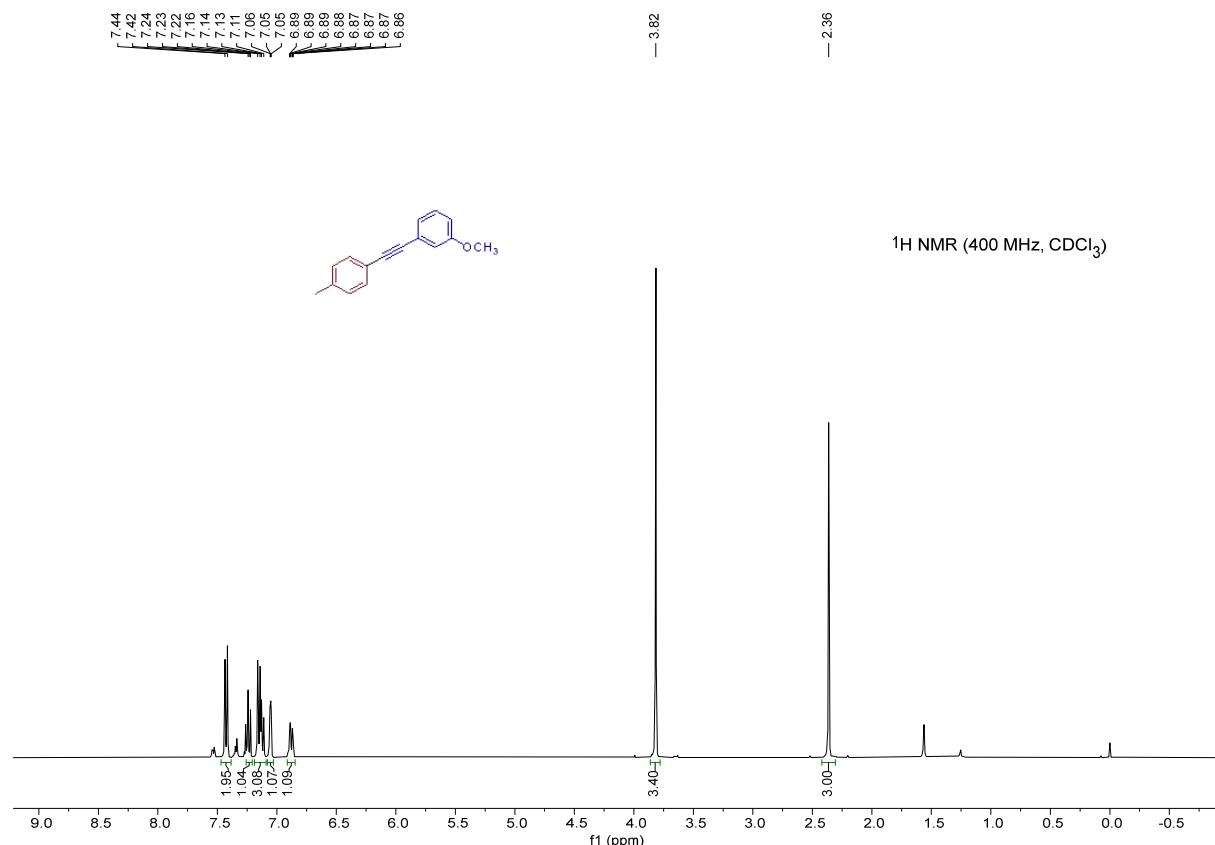
<147.97	-
<147.60	-
-140.16	-
<131.82	-
<129.57	-
>128.24	-
>126.22	-
>125.70	-
>123.24	-
-116.95	-
-111.61	-
-108.63	-
-101.44	-
-101.44	-
-93.41	-
-86.89	-
-	-20.87



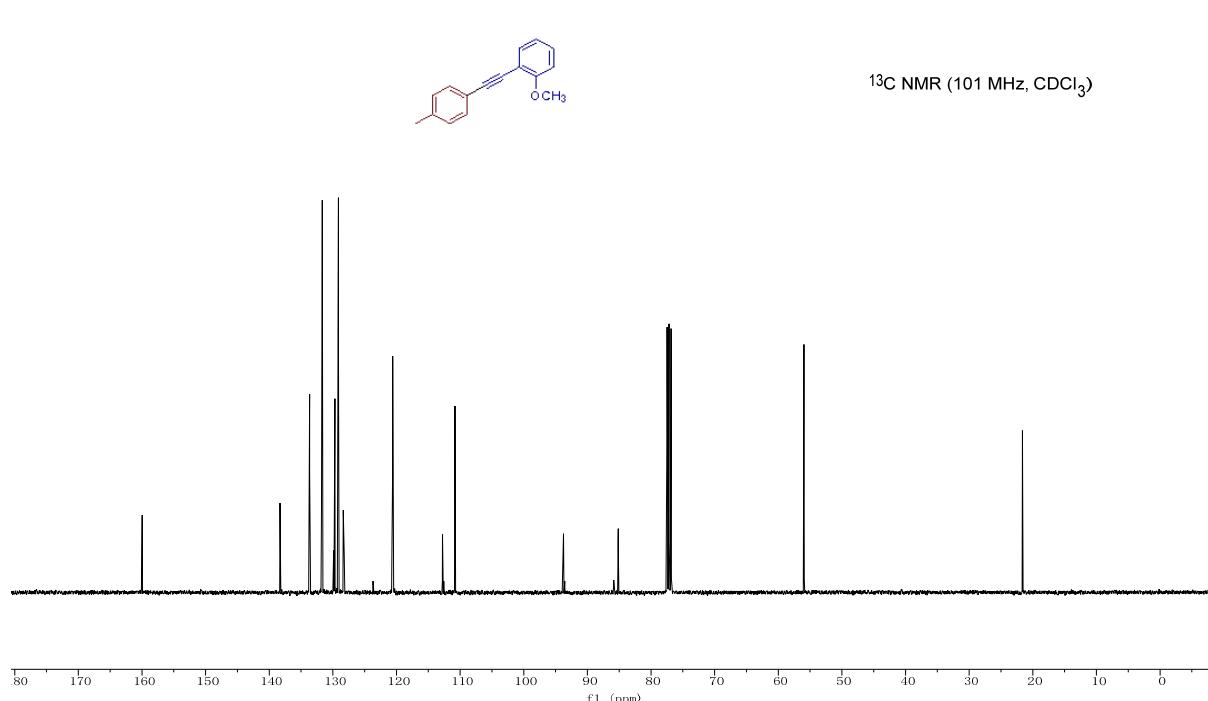
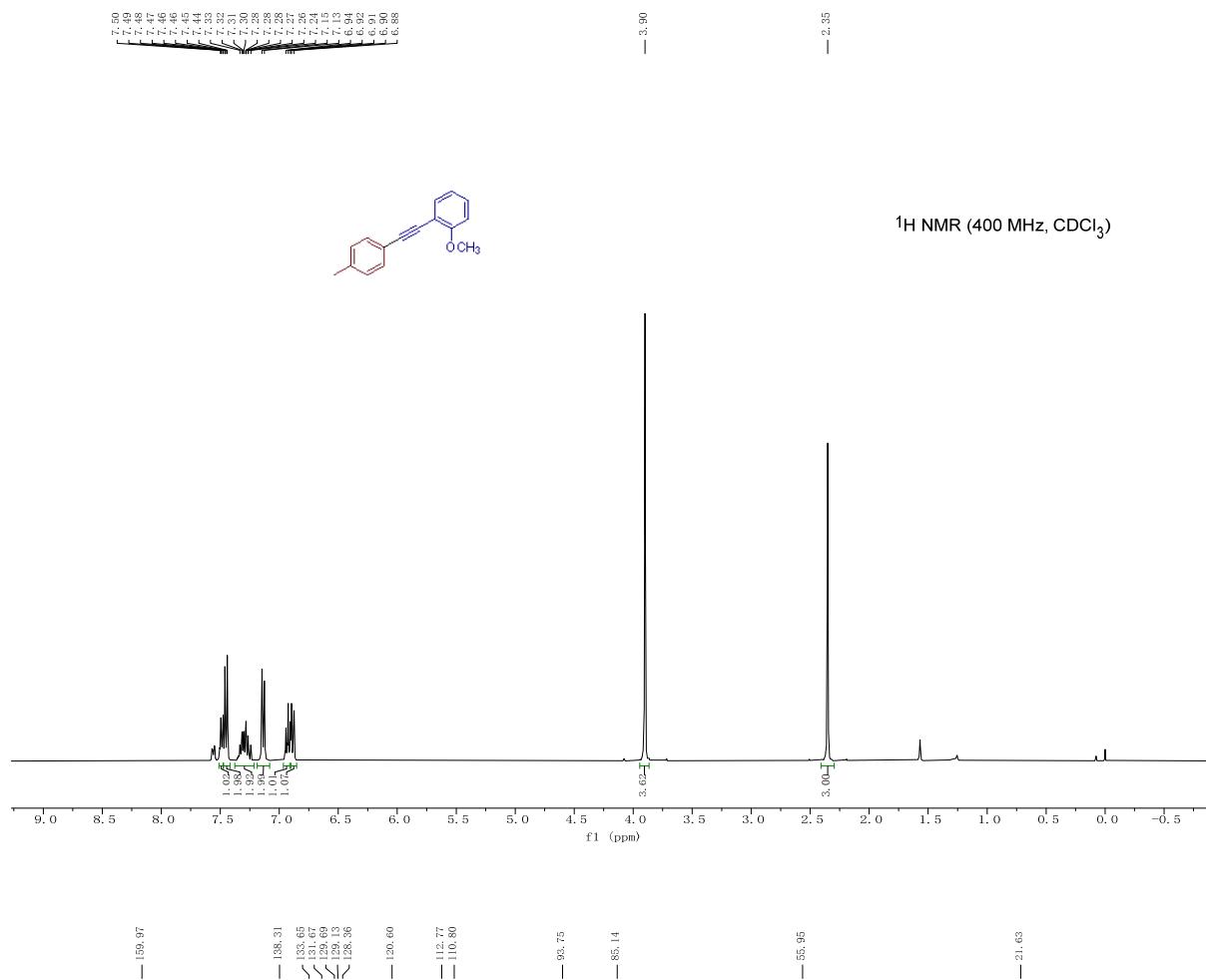
(18) 1-methoxy-4-((4-methylphenyl)ethynyl)benzene (**3-19**)



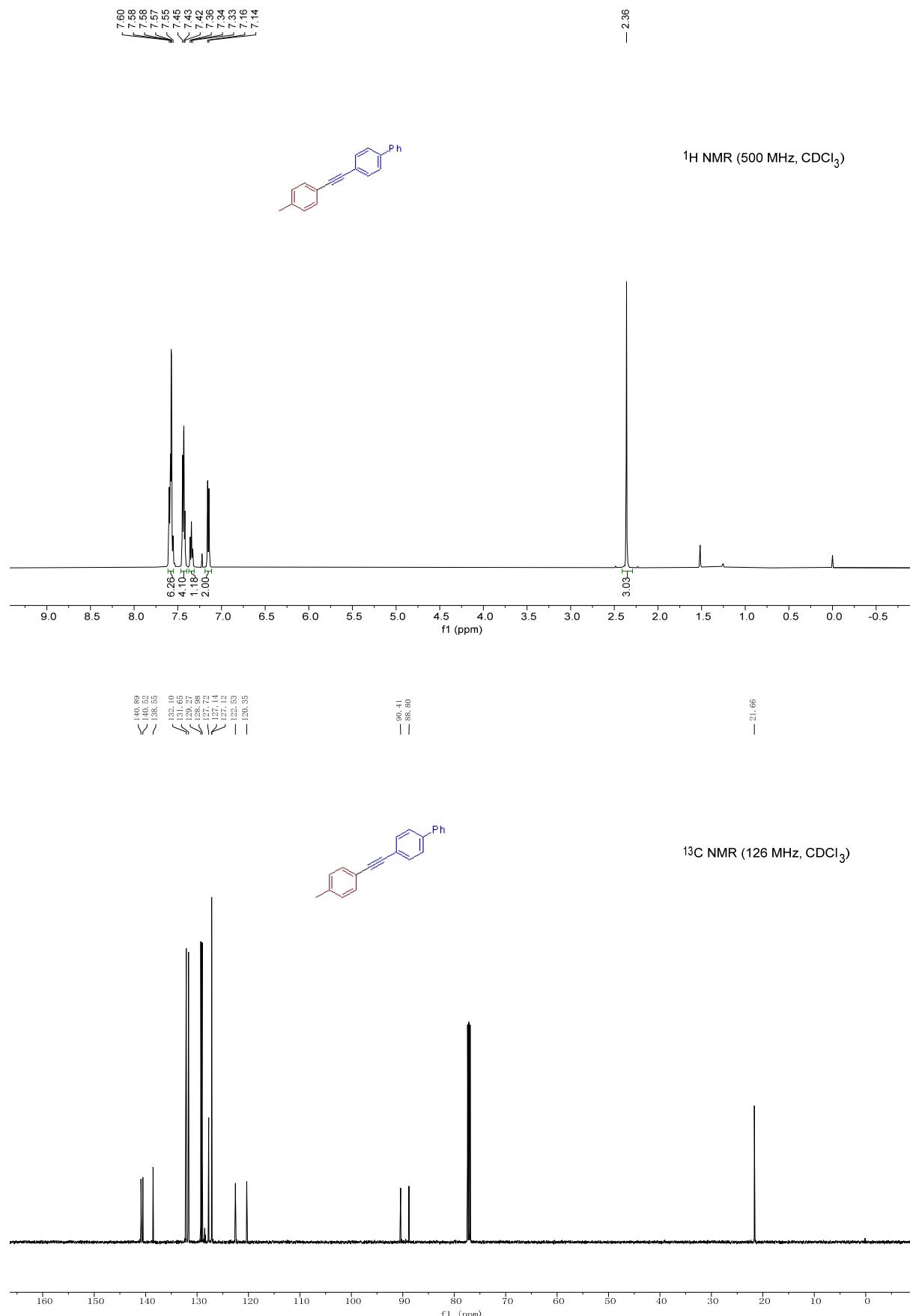
(19) 1-methoxy-3-((4-methylphenyl)ethynyl)benzene (**3-20**)



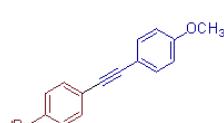
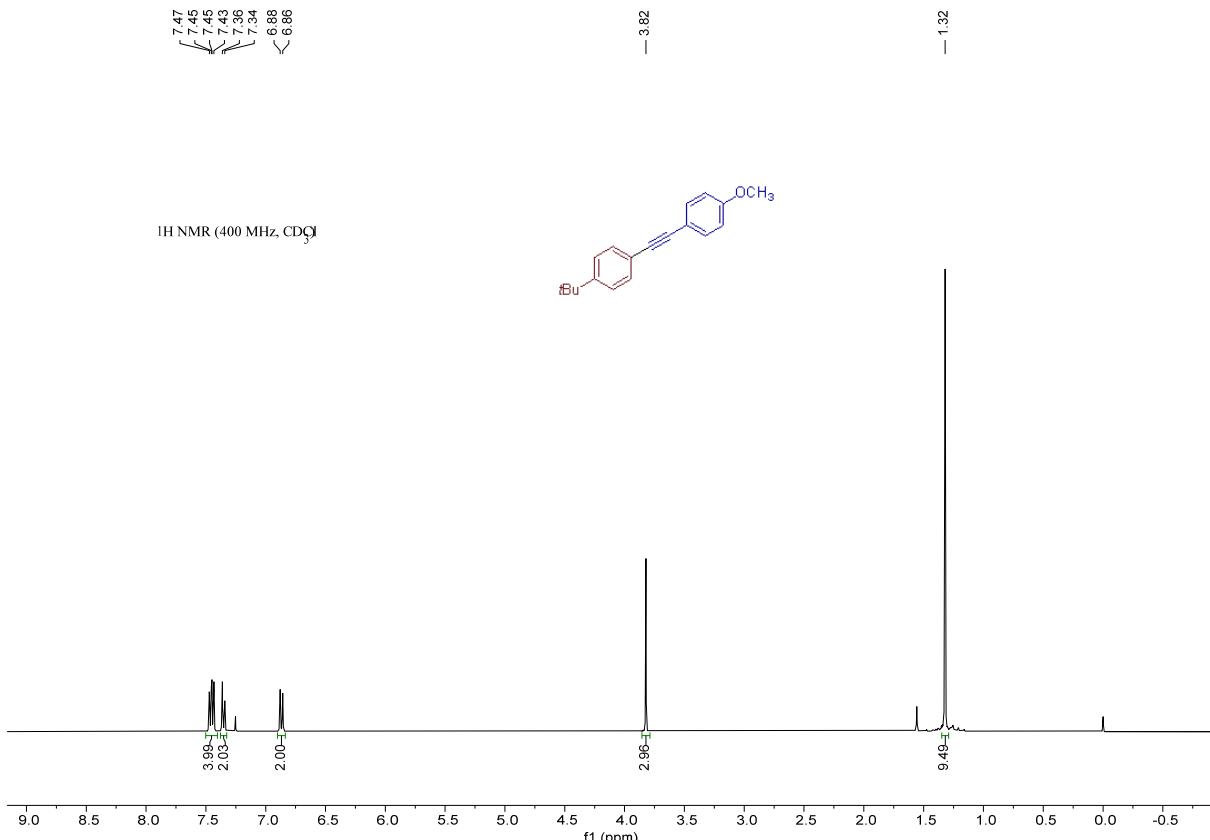
(20) 1-methoxy-2-((4-methylphenyl)ethynyl)benzene (**3-21**)



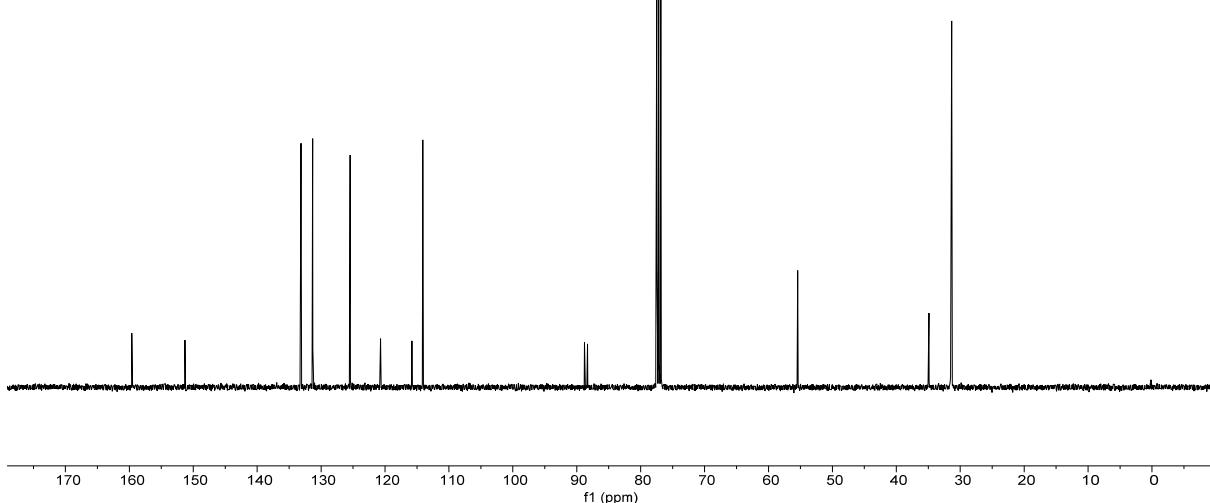
(21) 1-methyl-4-((4-phenylphenyl)ethynyl)benzene (**3-22**)



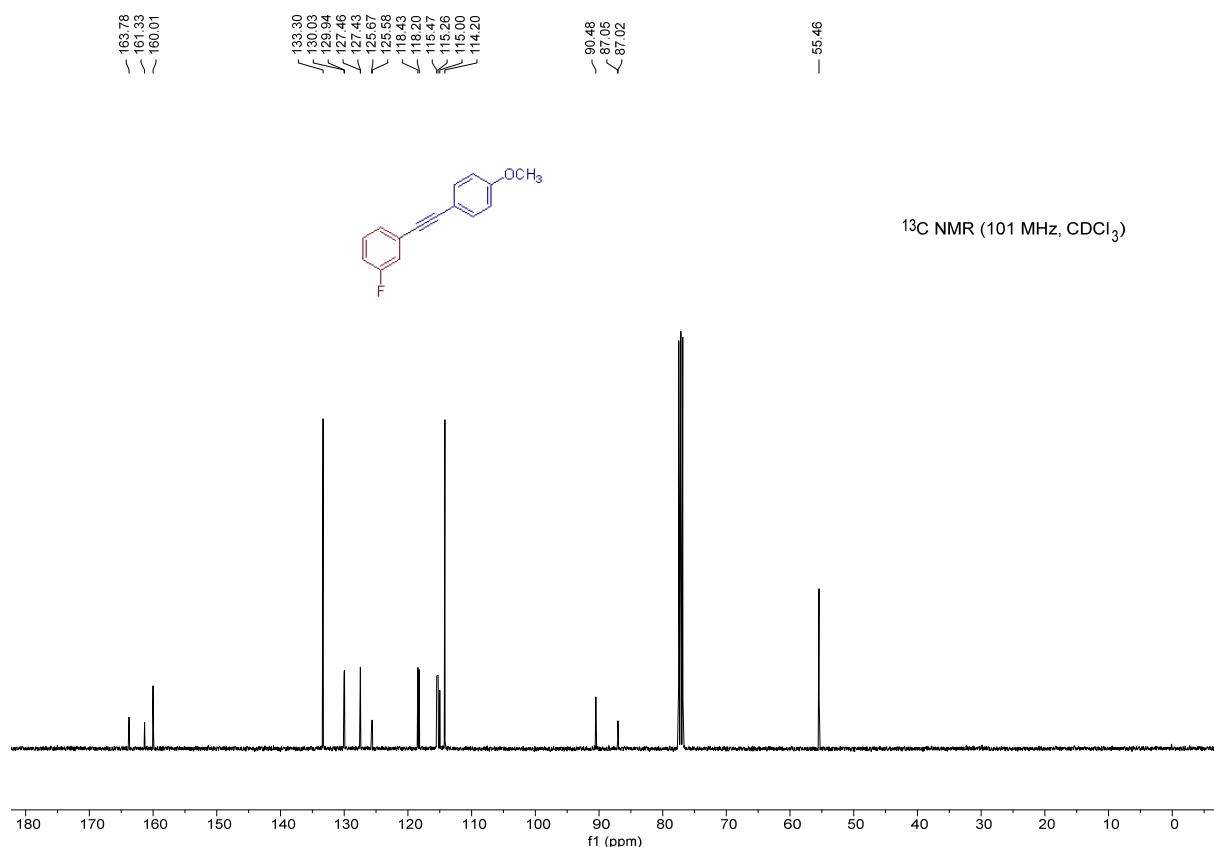
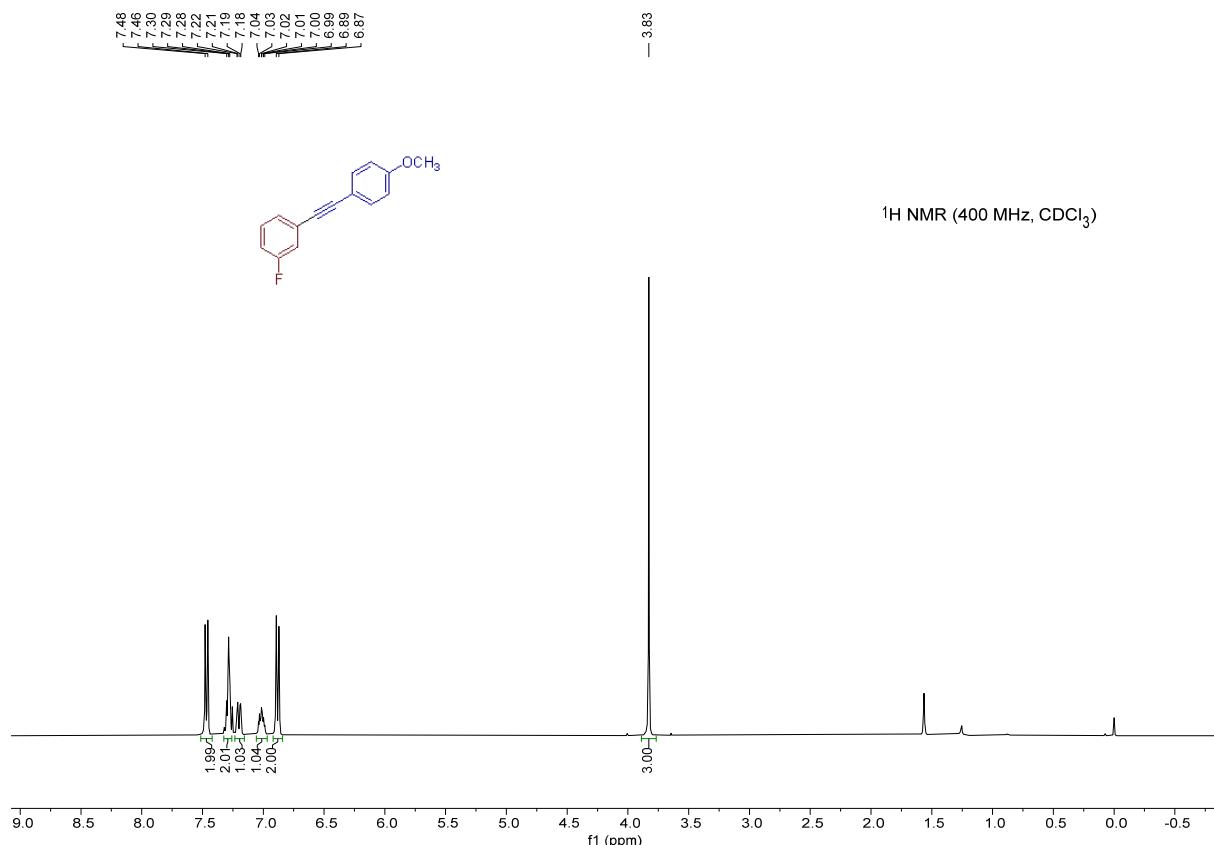
(22) 1-(*tert*-butyl)-4-((4-methoxyphenyl)ethynyl)benzene (3-23)

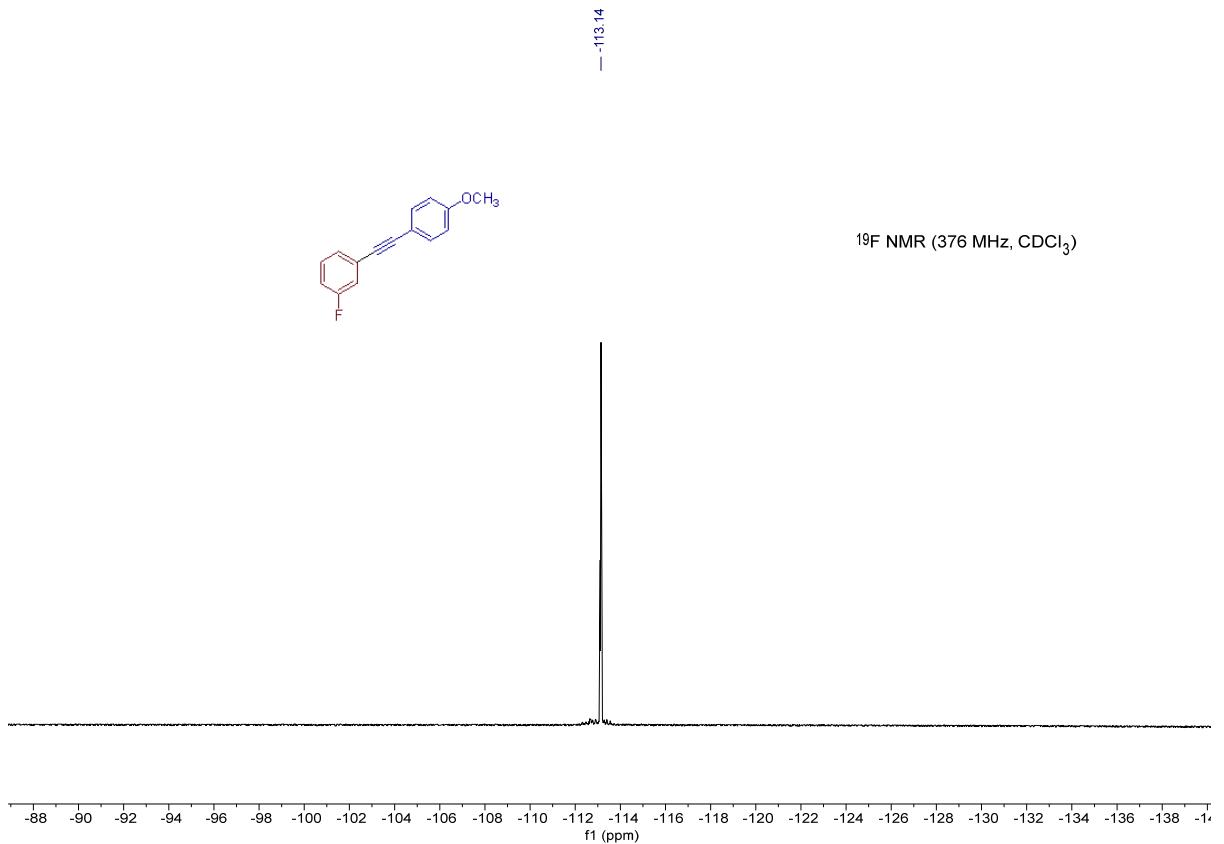


$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )

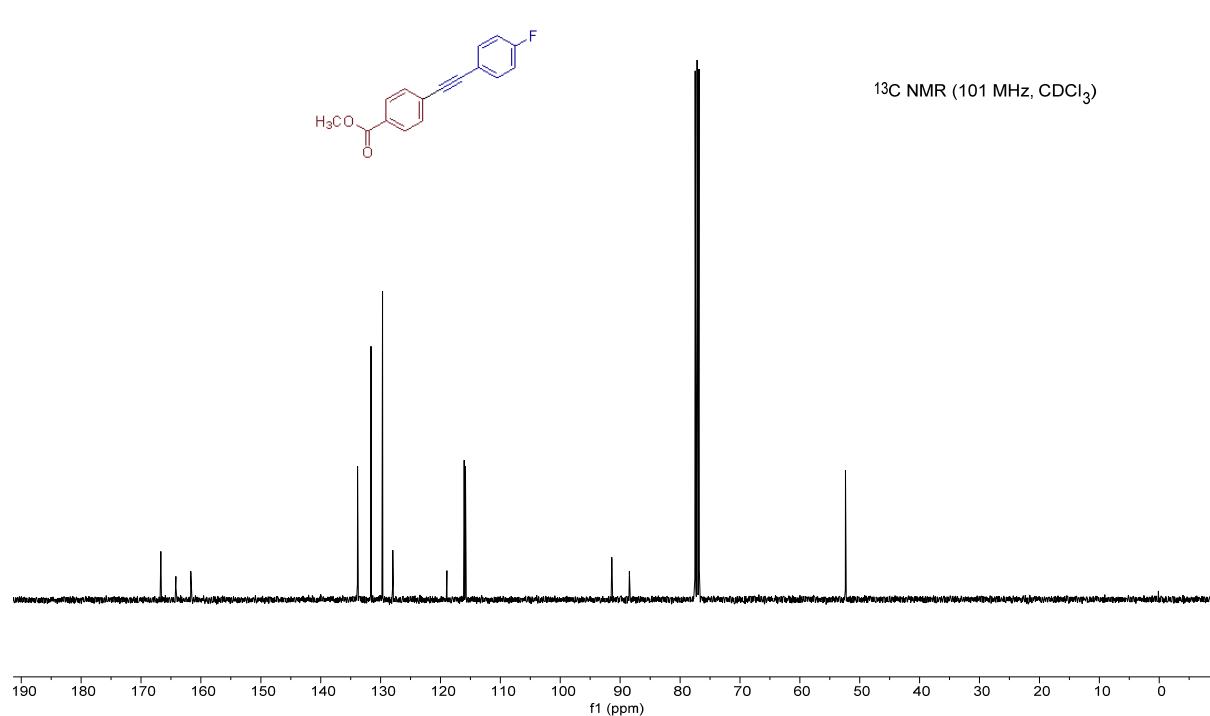
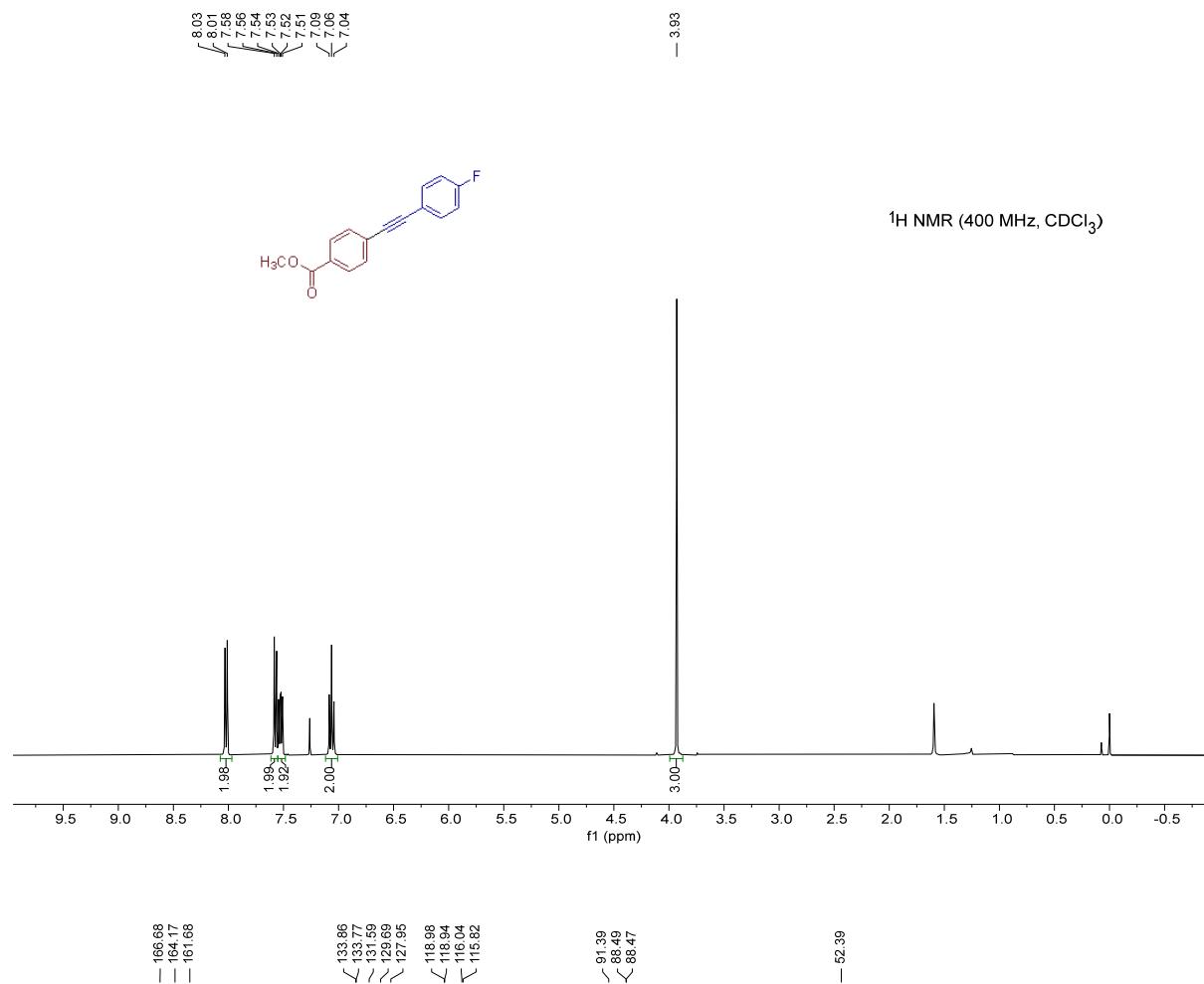


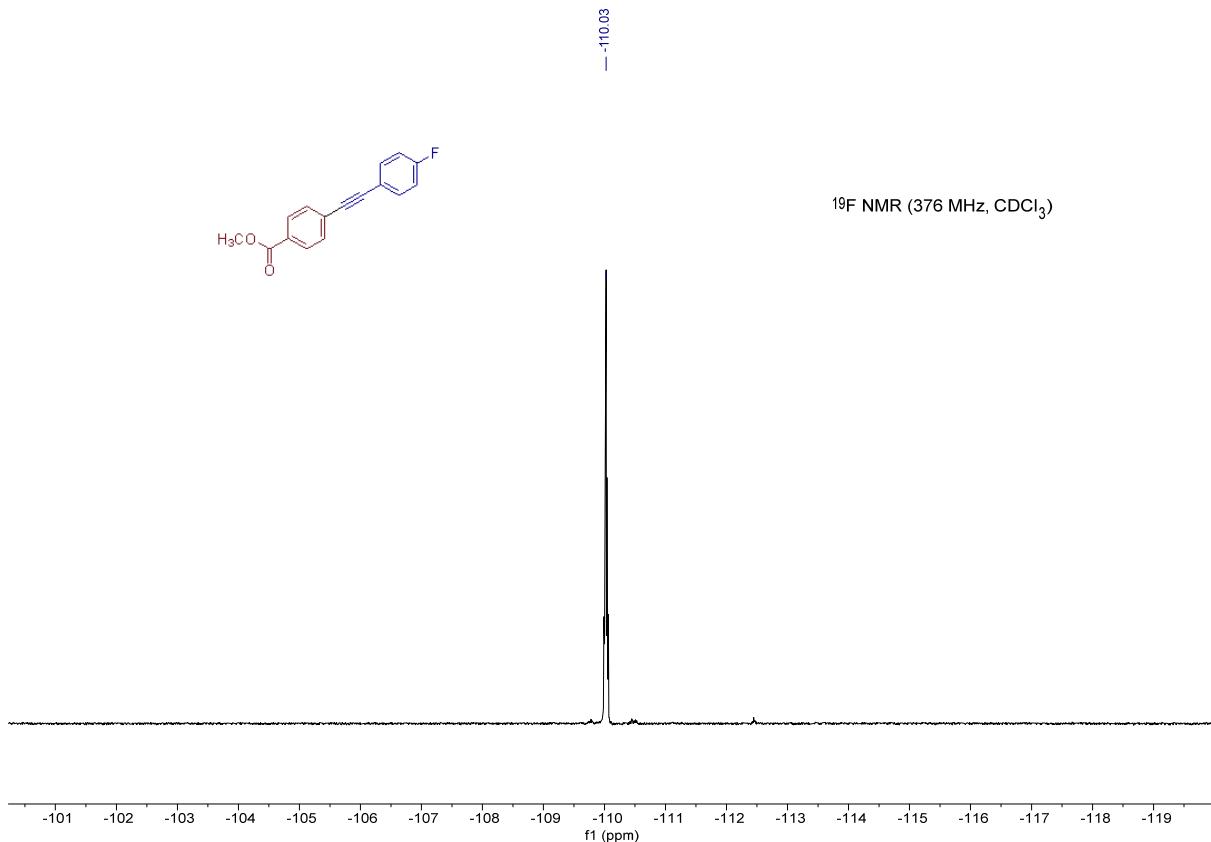
(23) 1-fluoro-3-((4-methoxyphenyl)ethynyl)benzene (**3-24**)



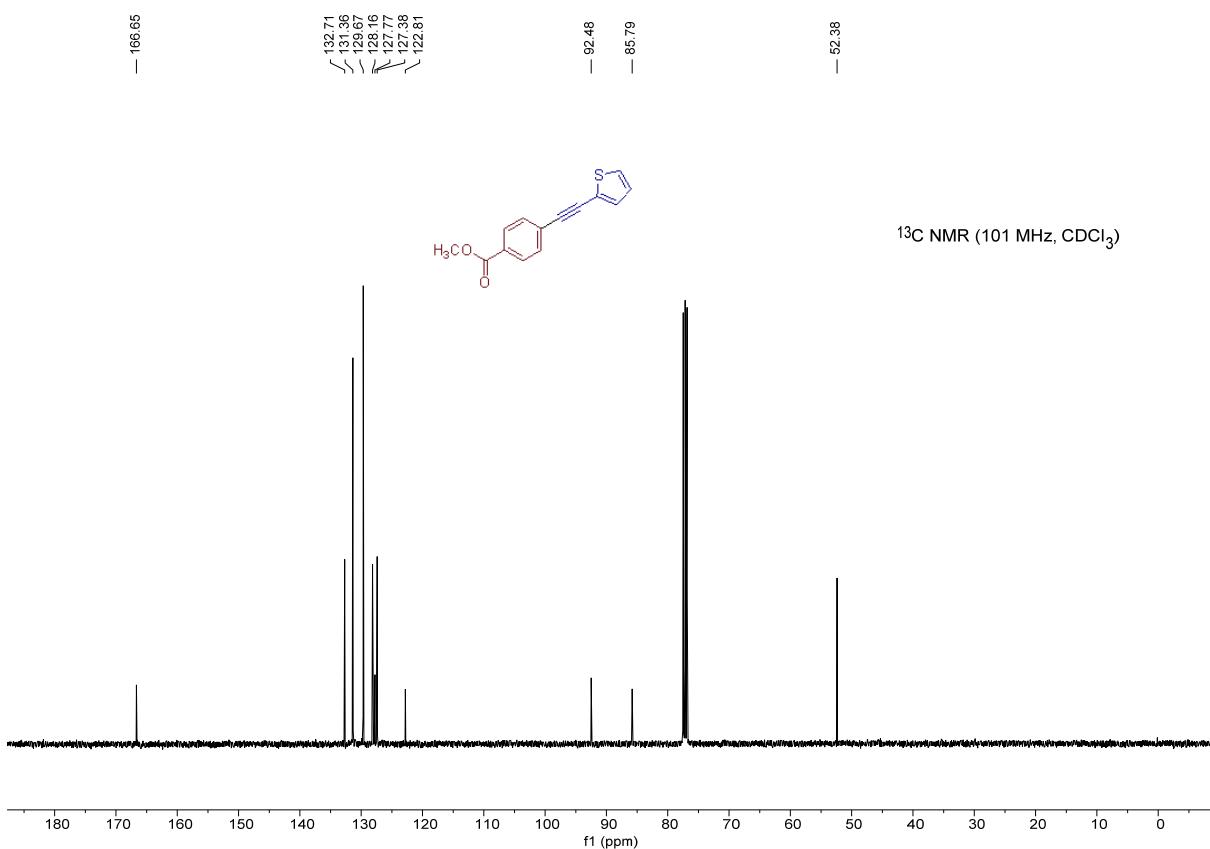
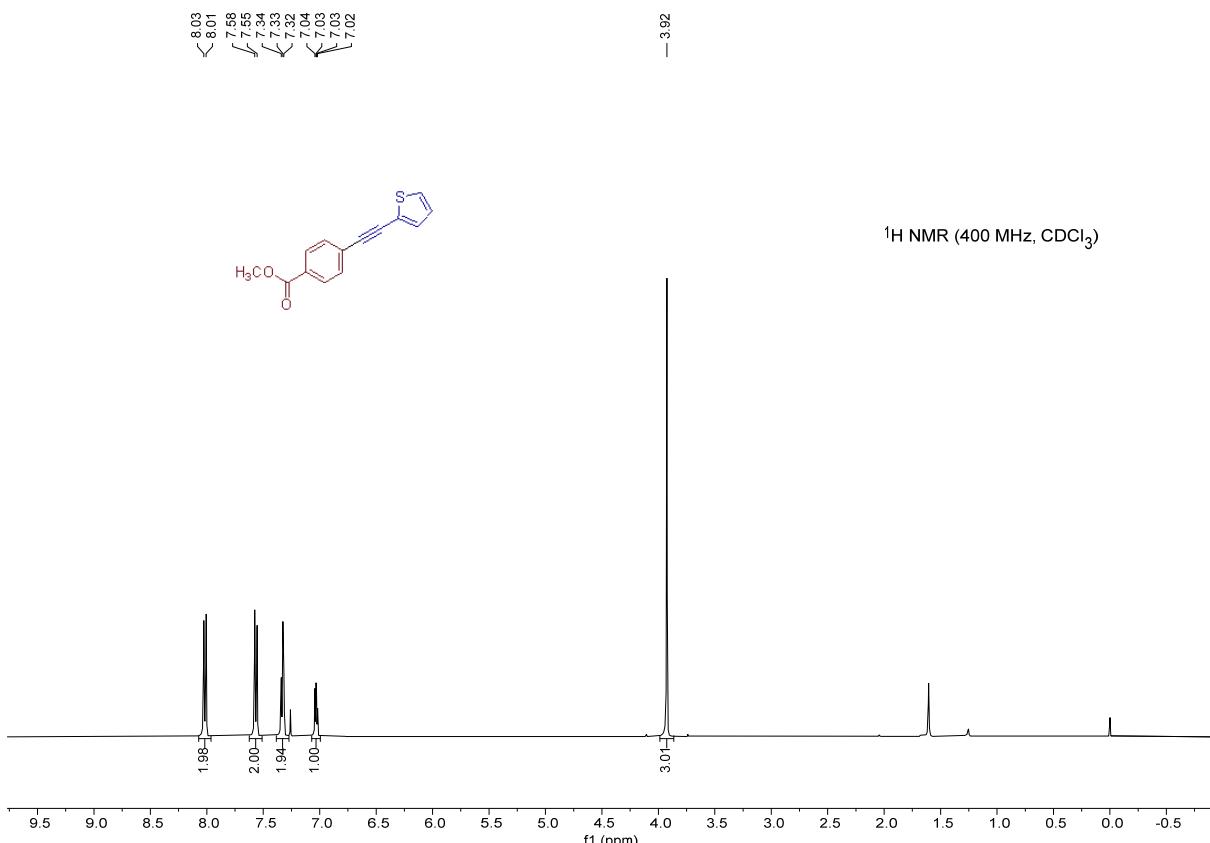


(24) methyl 4-((4-fluorophenyl)ethynyl)benzoate (**3-25**)

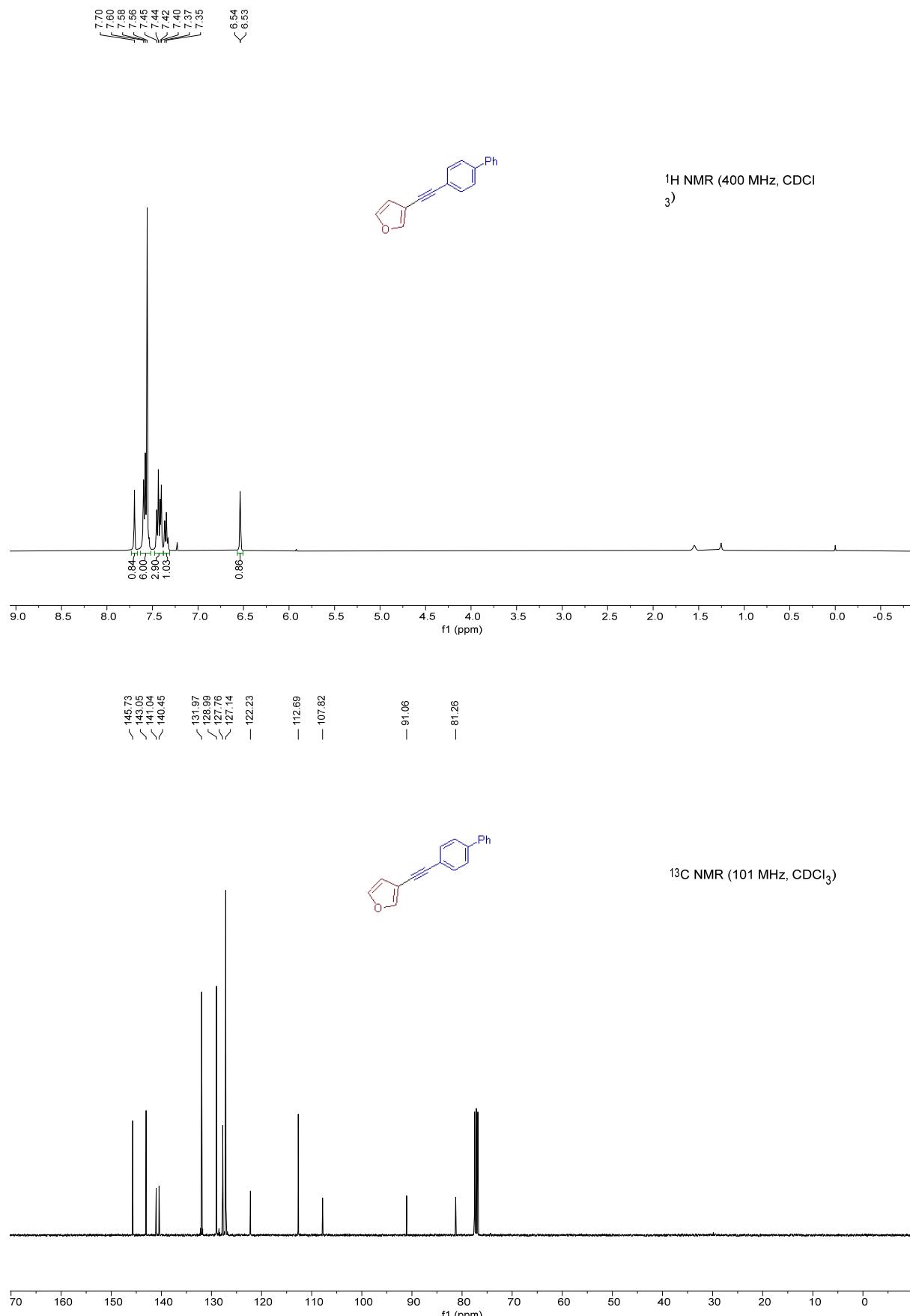




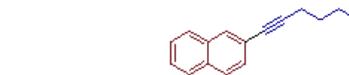
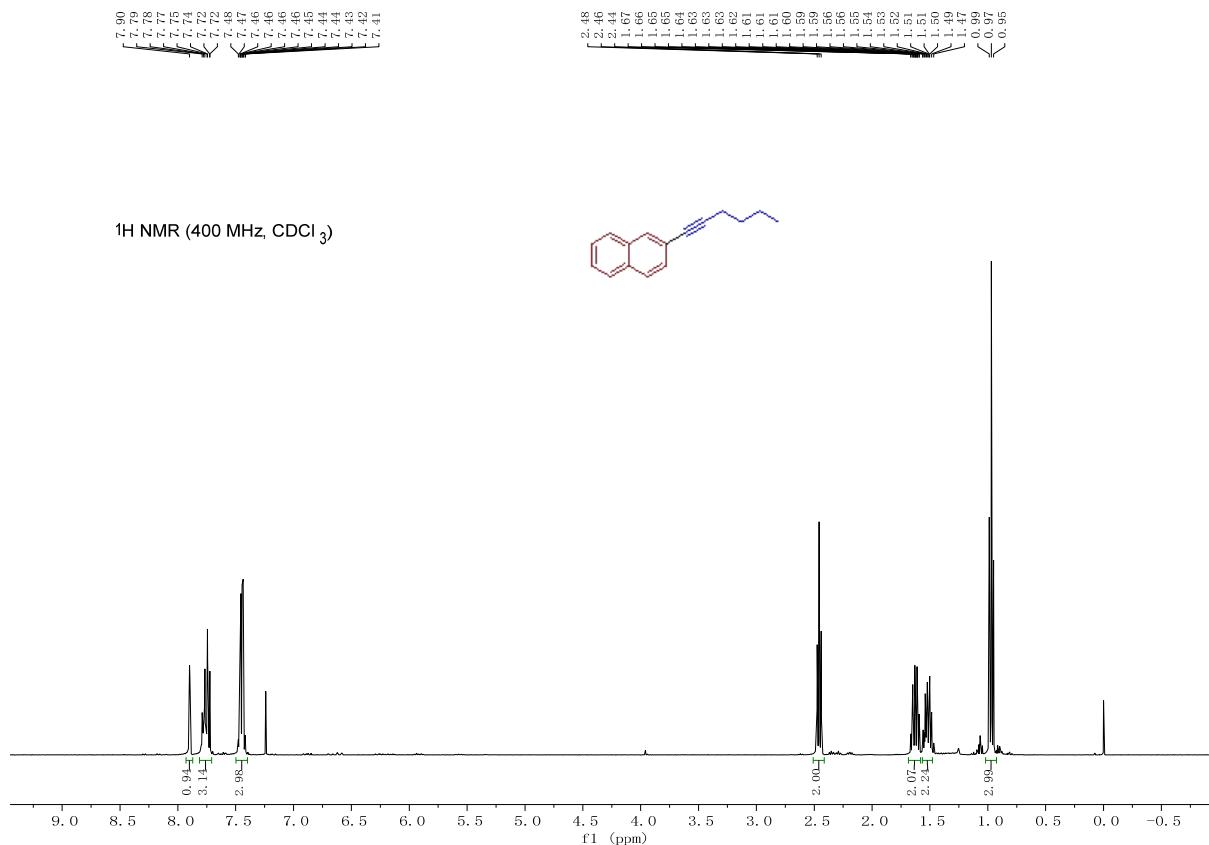
(25) methyl 4-(thiophen-2-ylethynyl)benzoate (**3-26**)



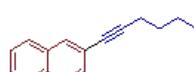
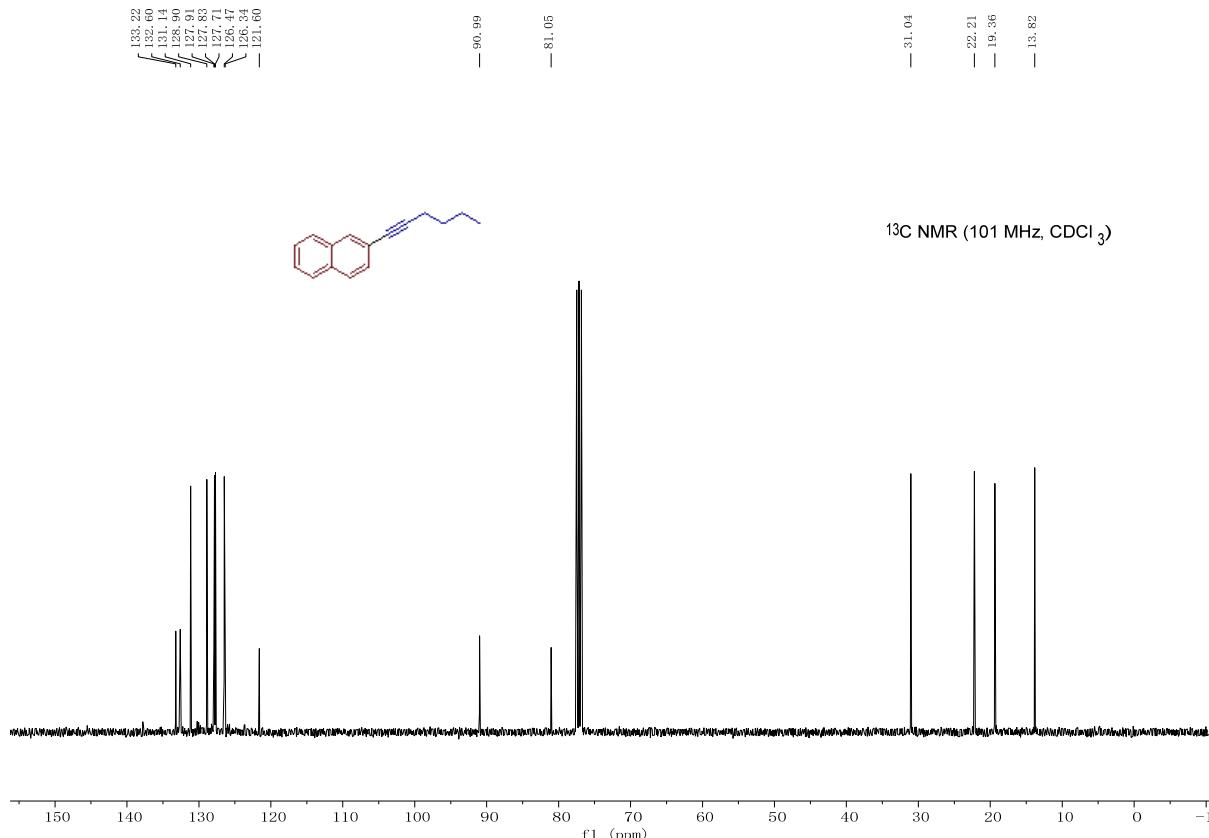
(26) 3-([1,1'-biphenyl]-4-ylethynyl)furan (**3-27**)



(27) 2-(hex-1-ynyl)naphthalene (**3-28**)

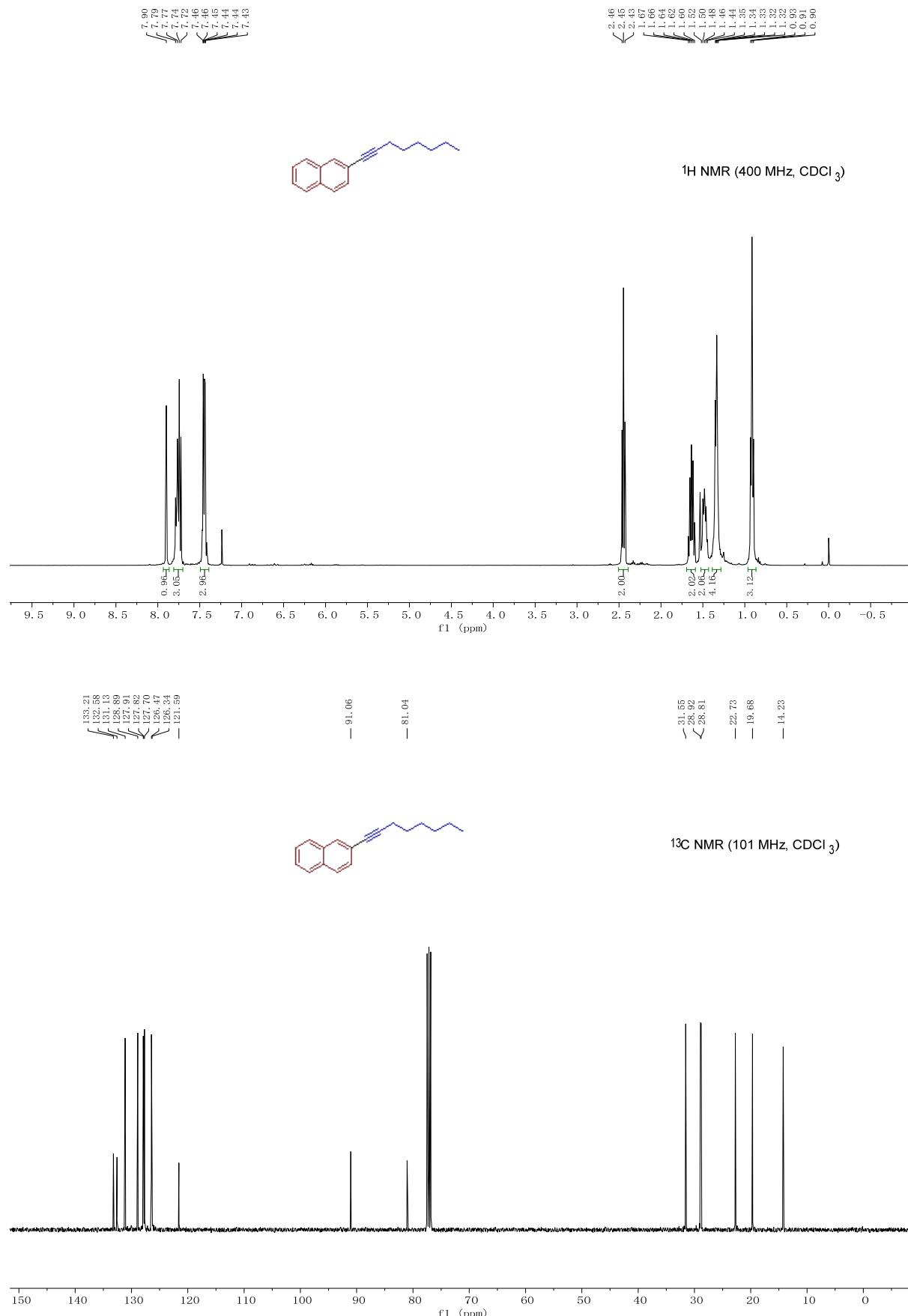


<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

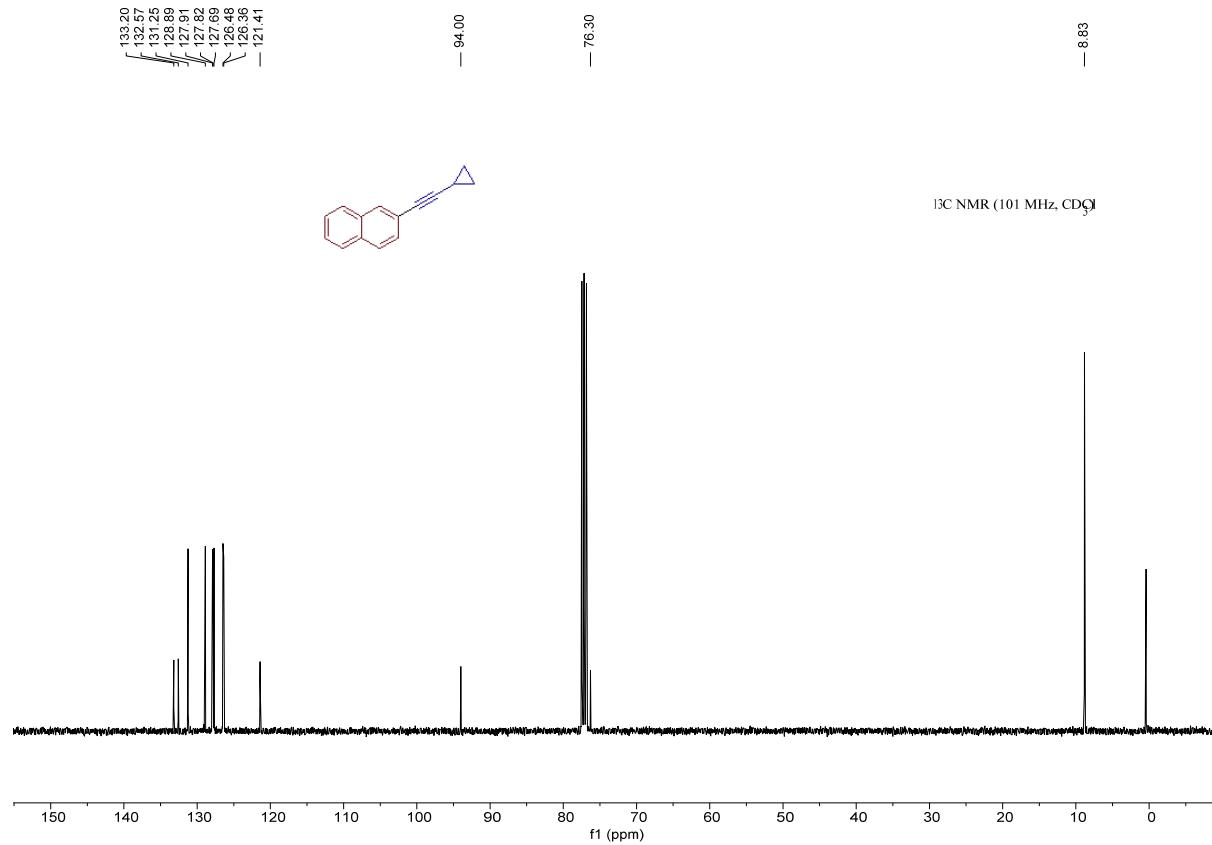
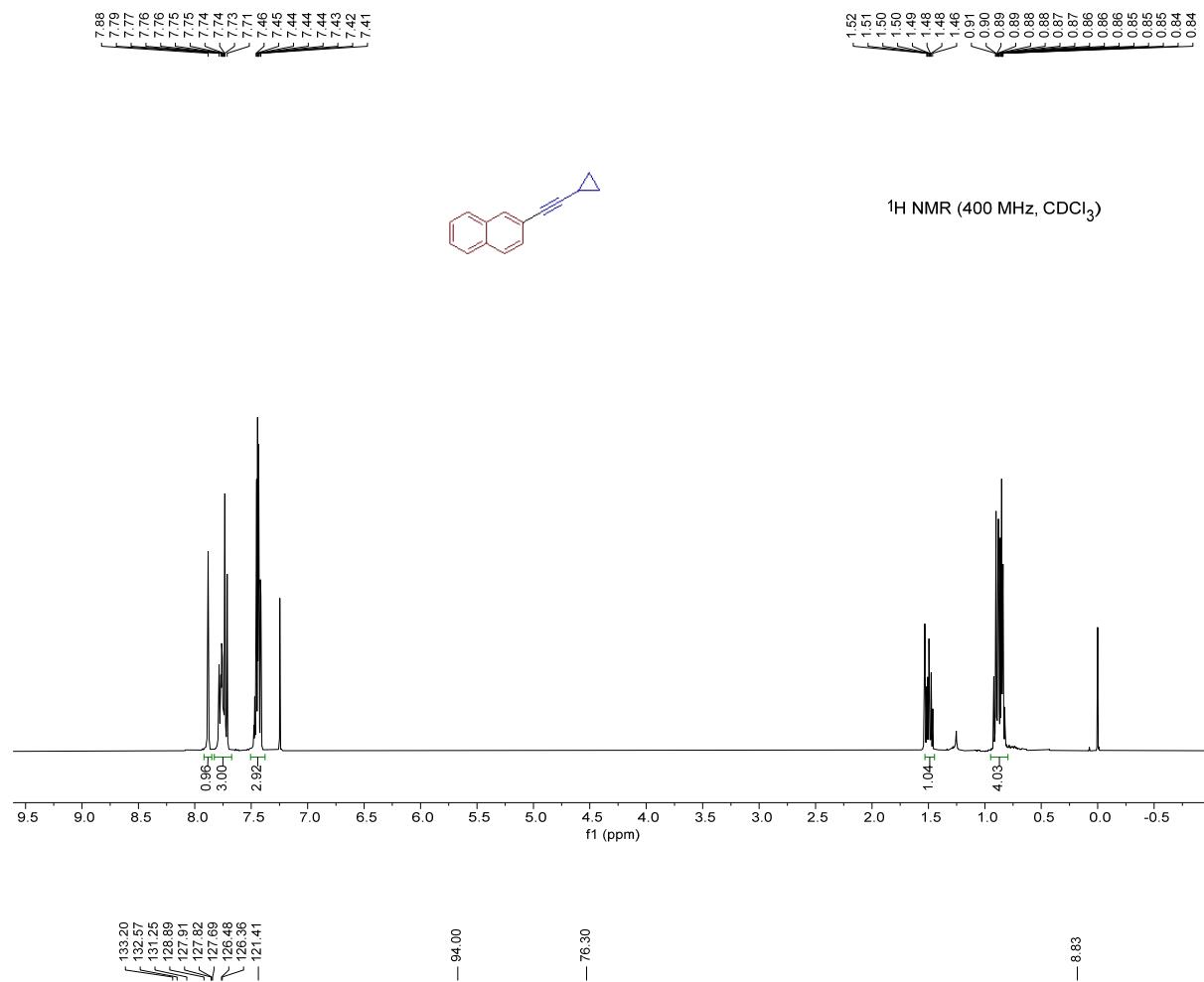


<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)

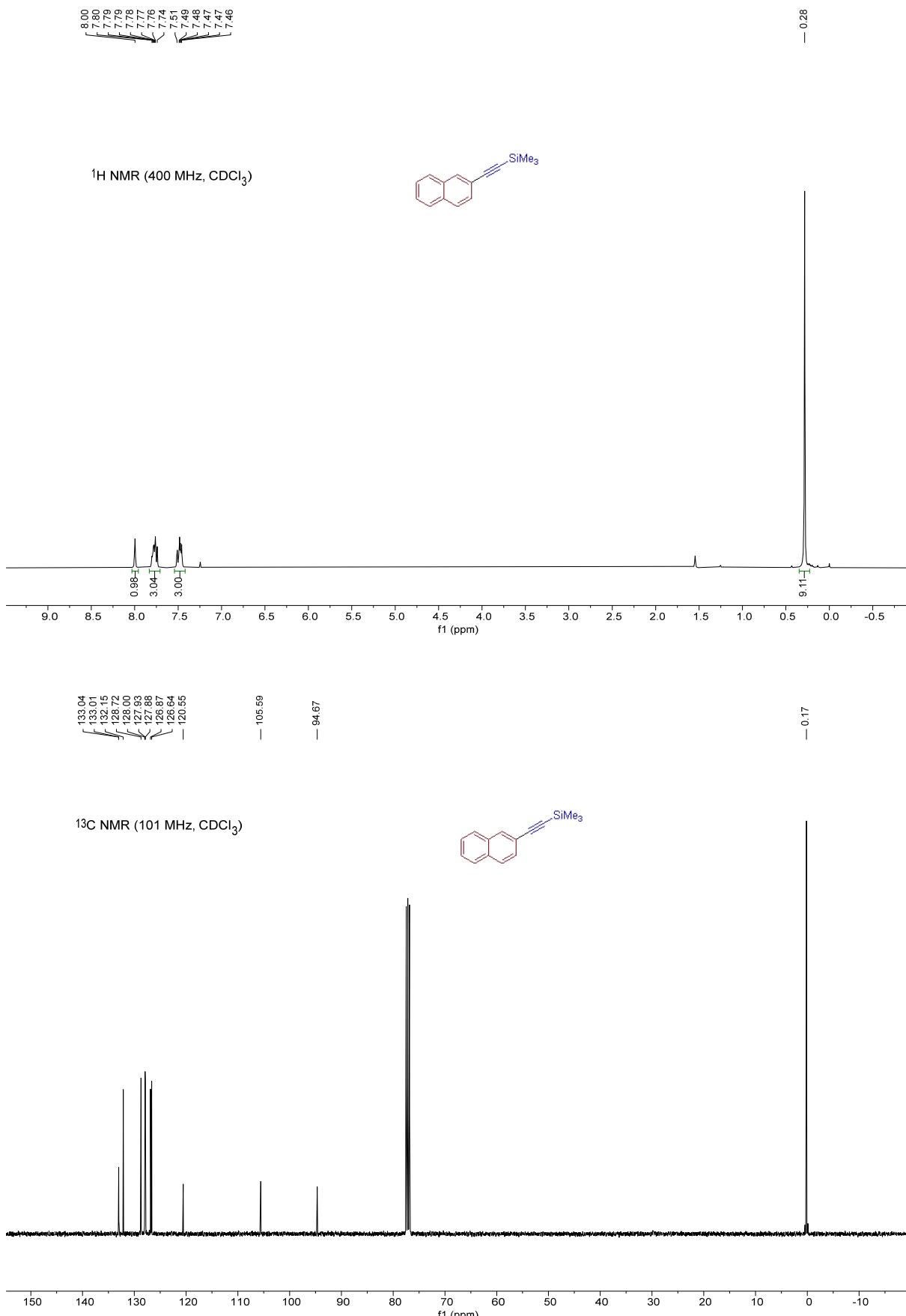
(28) 2-(oct-1-yn-1-yl)naphthalene (**3-29**)



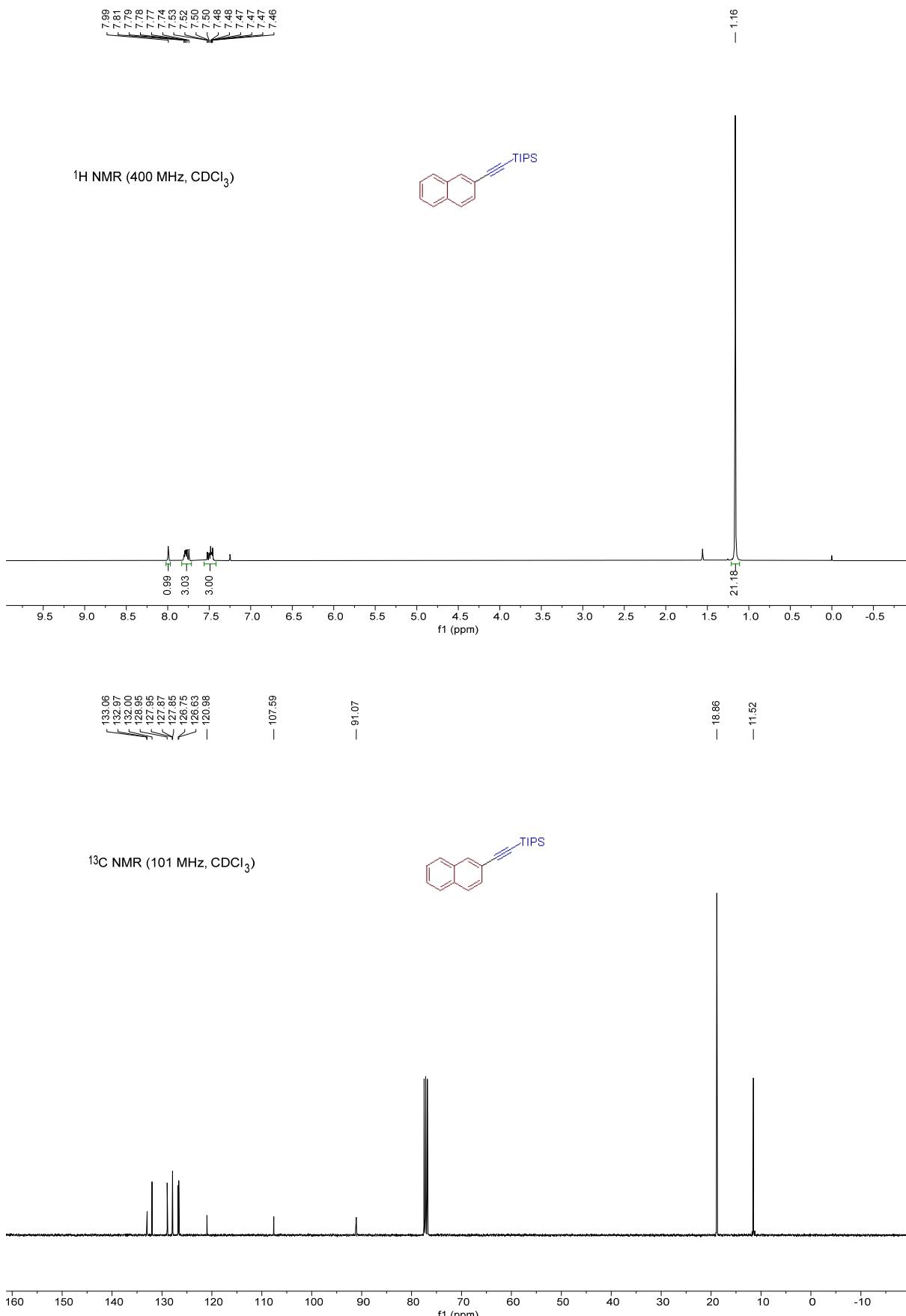
(29) 2-(cyclopropylethynyl)naphthalene (**3-30**)



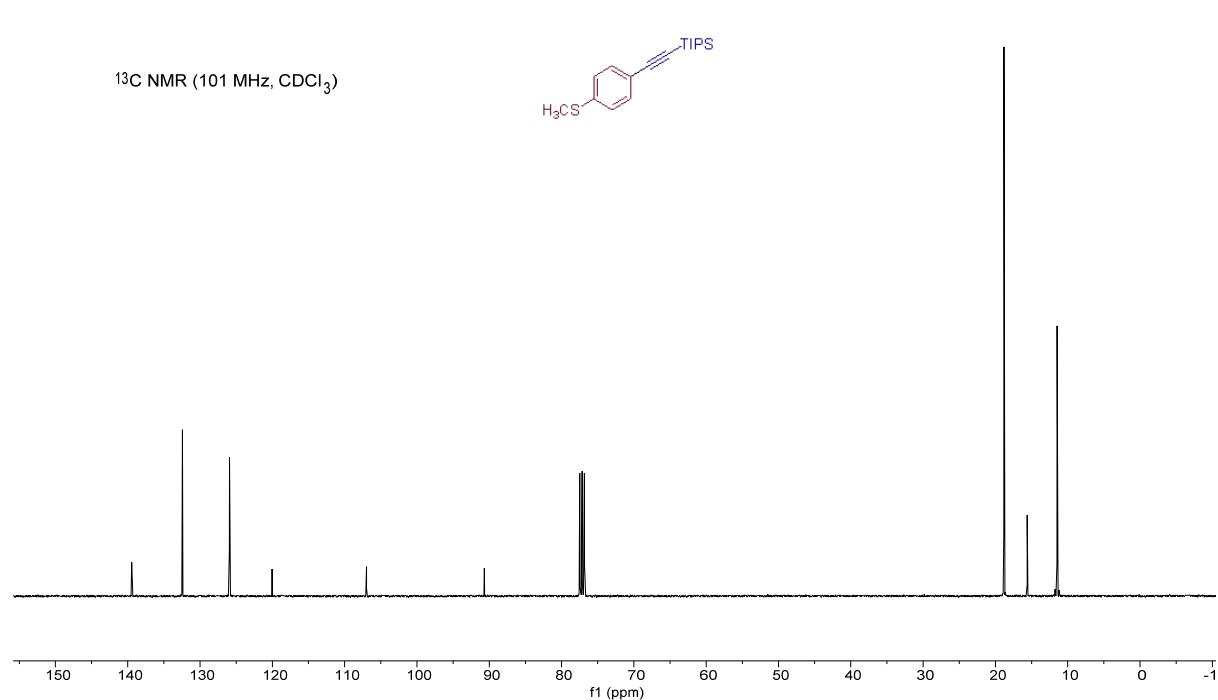
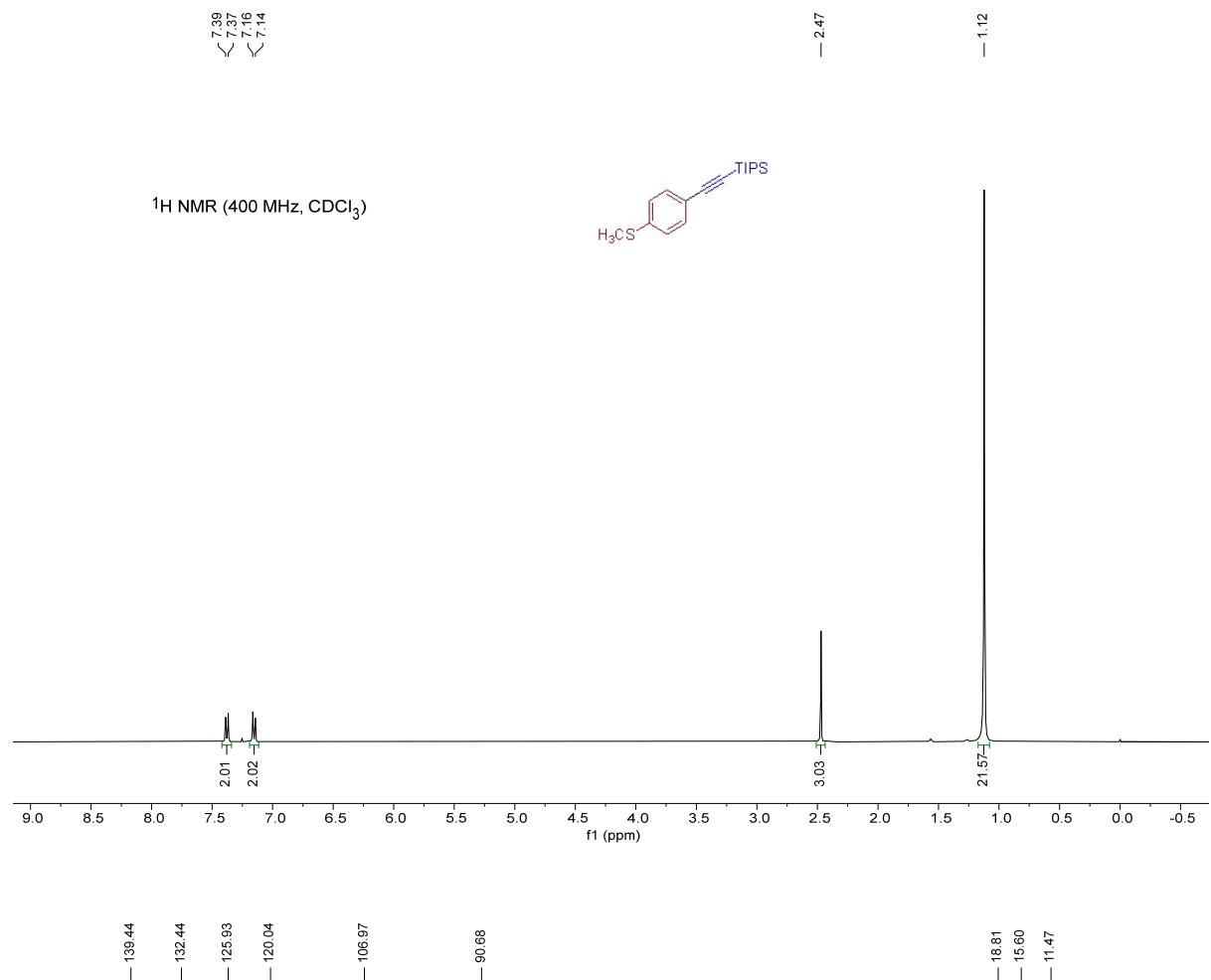
(30) trimethyl(naphthalen-2-ylethynyl)silane (**3-31**)



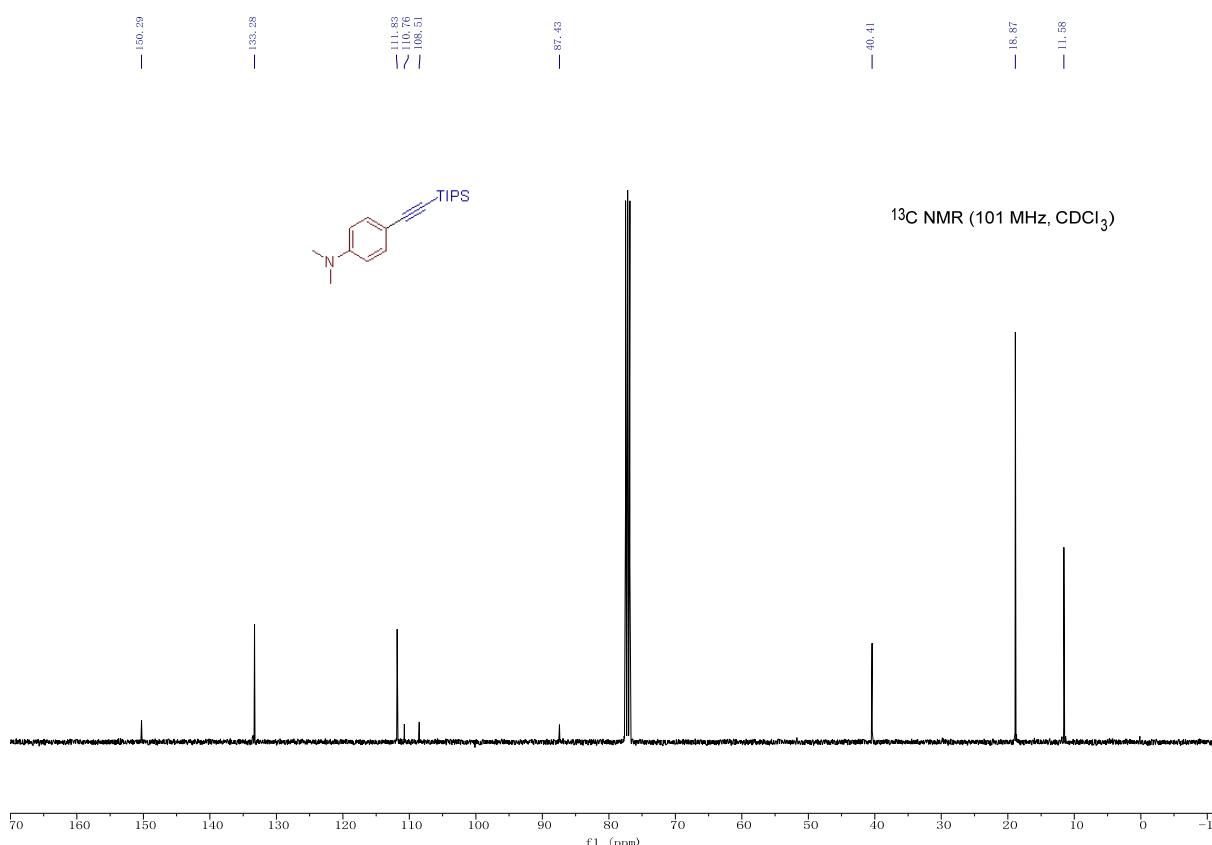
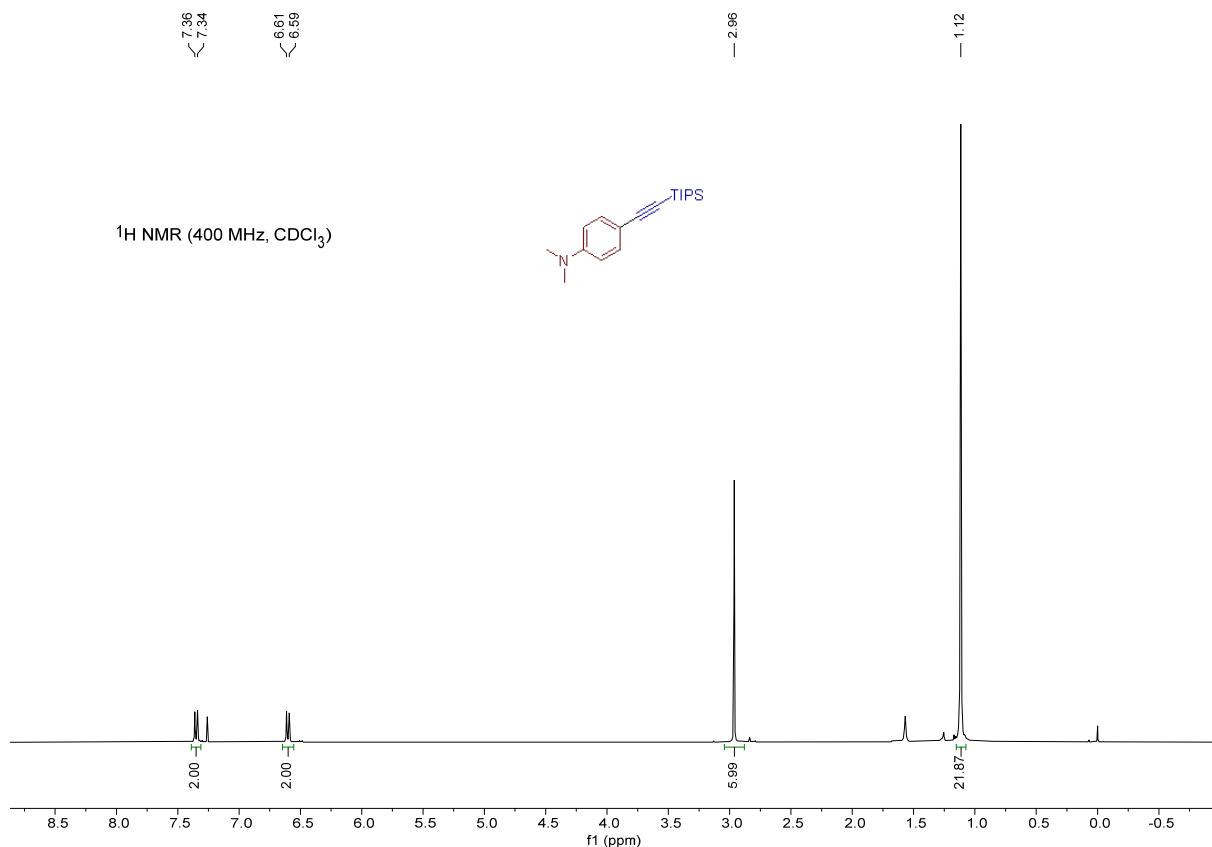
(31) (naphthalen-2-ylethyynyl)(tripropan-2-yl)silane (**3-32**)



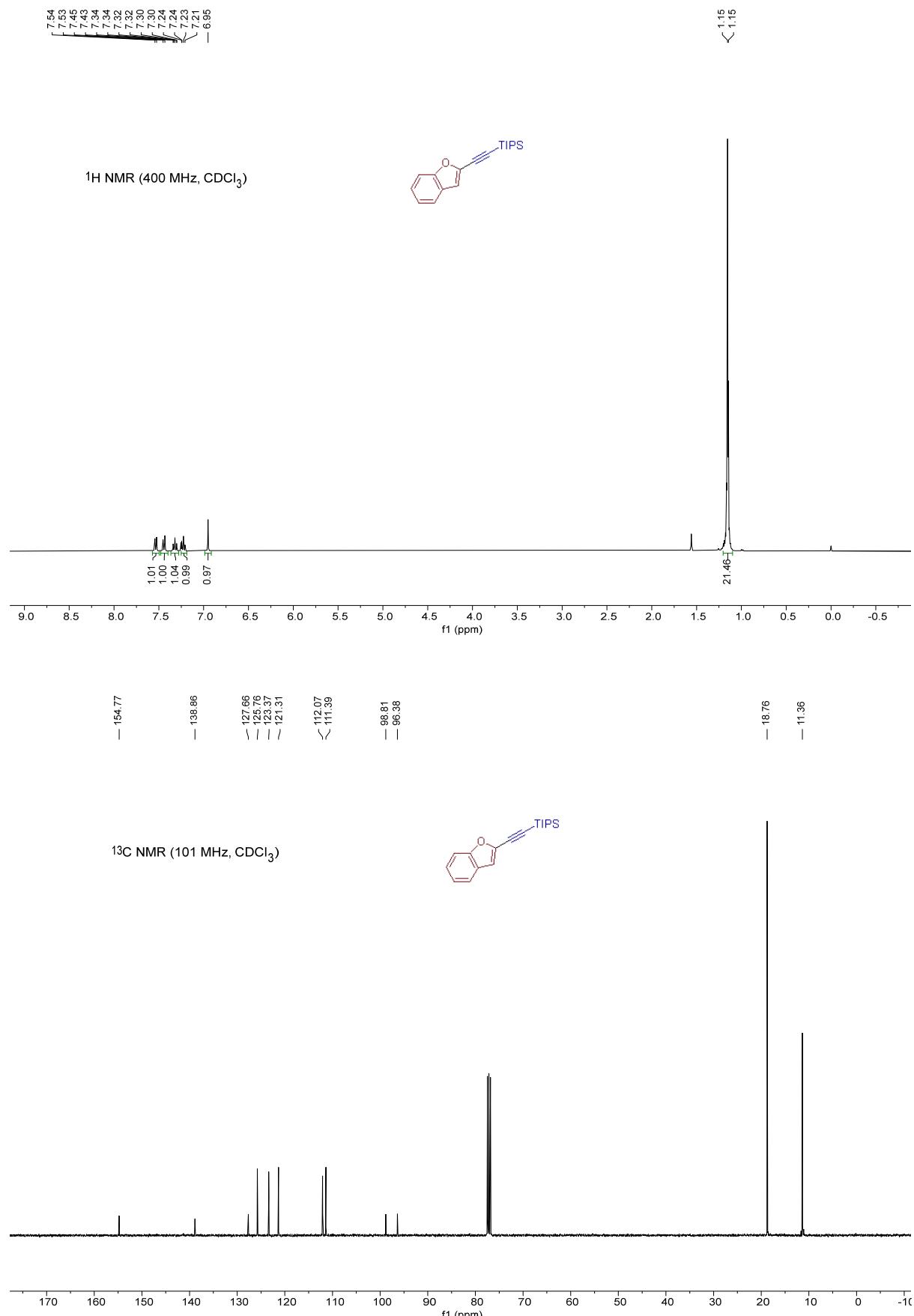
(32) ((4-(methylsulfanyl)phenyl)ethynyl)(tripropan-2-yl)silane (**3-33**)



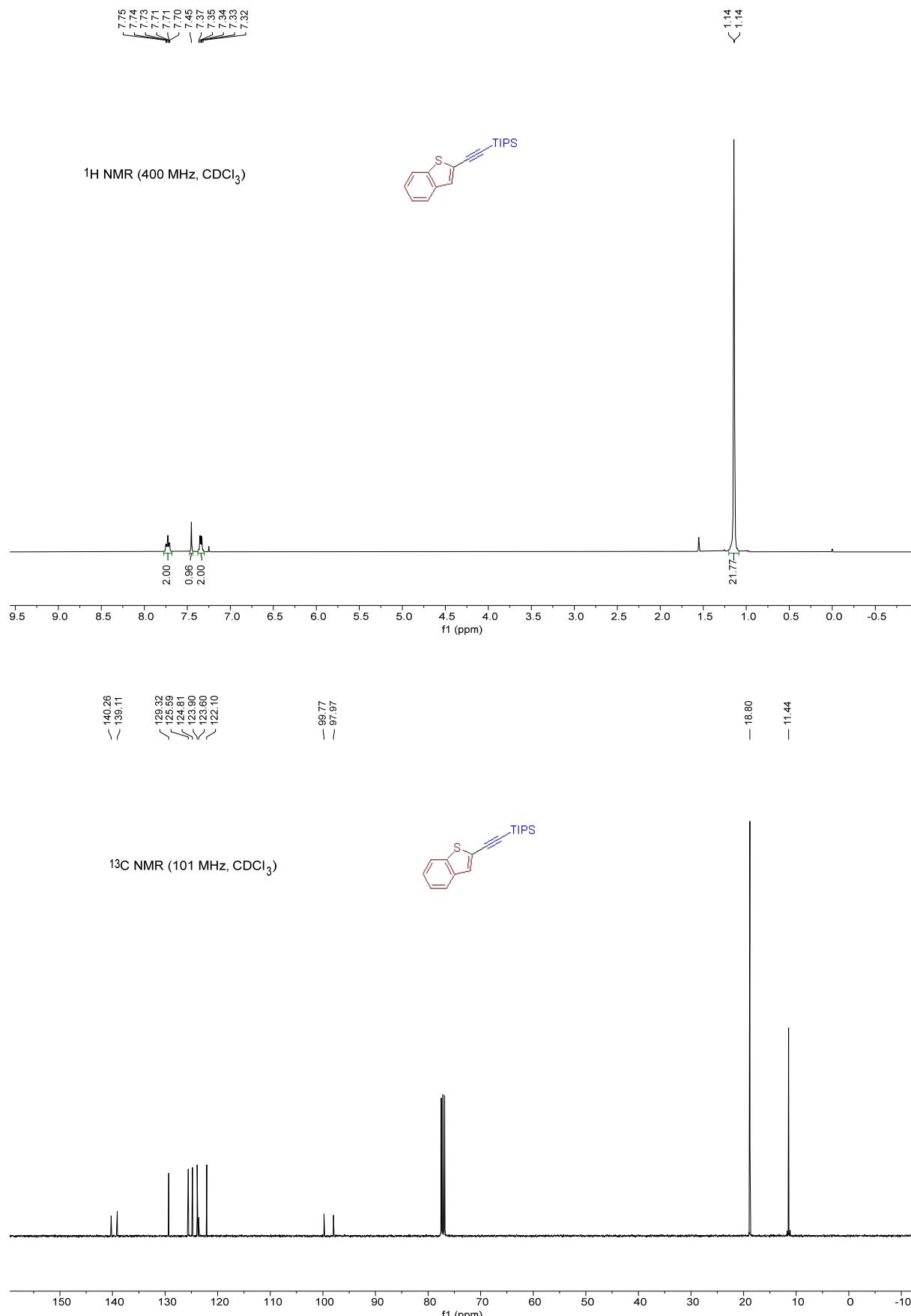
(33) N,N-dimethyl-4-((triisopropylsilyl)ethynyl)aniline (3-34)



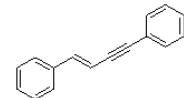
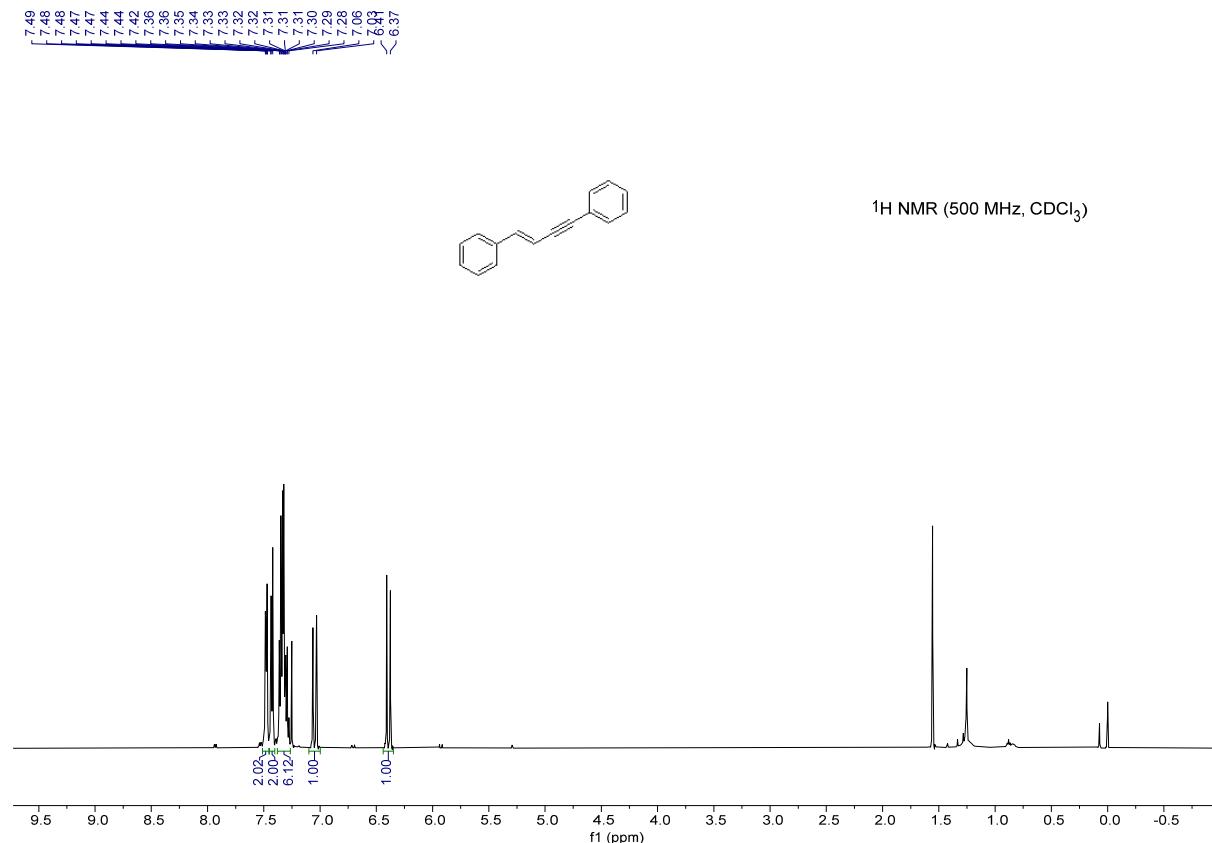
(34) (benzofuran-2-ylethyynyl)triisopropylsilane (**3-35**)



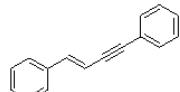
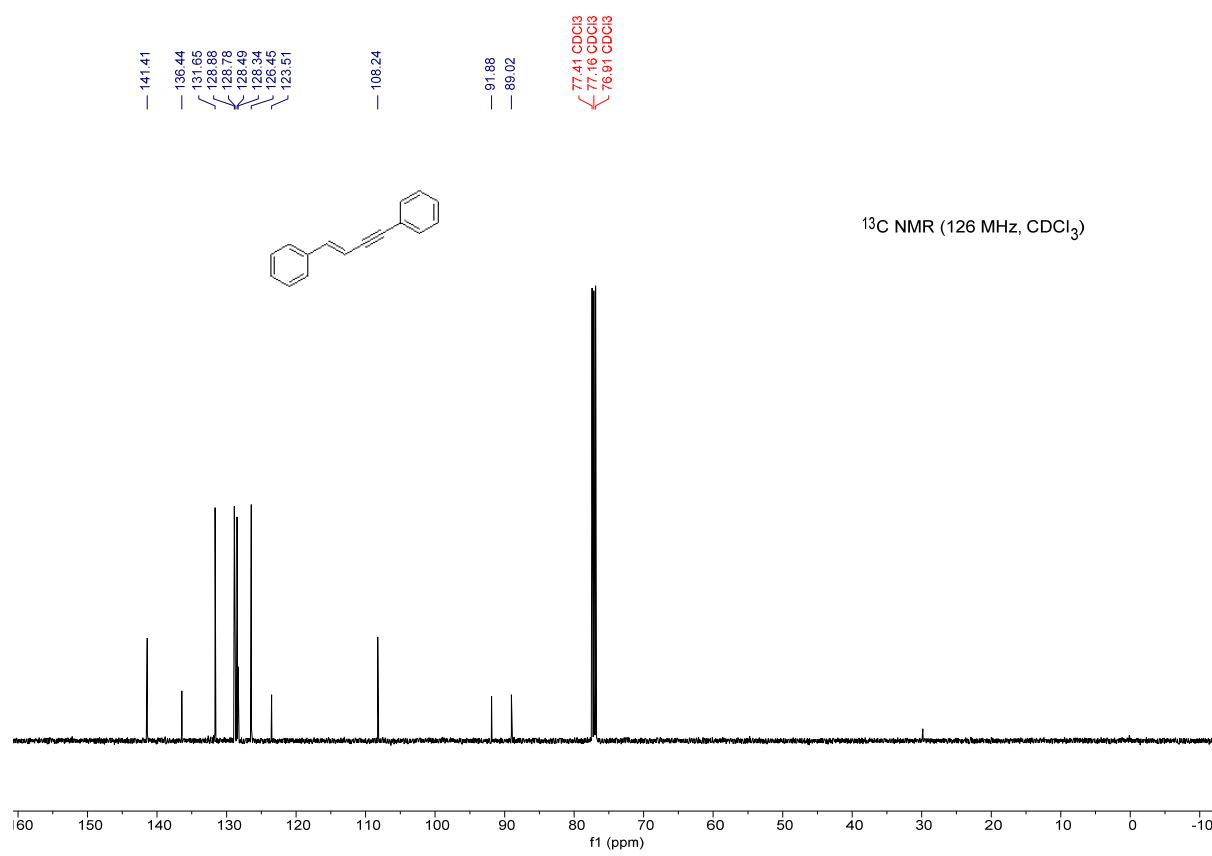
(35) (benzo[b]thiophen-2-ylethylynyl)triisopropylsilane (**3-36**)



(36) (E)-but-1-en-3-yne-1,4-diyldibenzene (3-37)



<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)



<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)

(37) A mixture of (*E*)-pent-3-en-1-yn-1-ylbenzene (**3-38**) and 1,4-diphenylbuta-1,3-diyne

