Supplementary Materials

Tetraalkynylstannanes in the Stille cross coupling reaction: a new effective approach to arylalkynes

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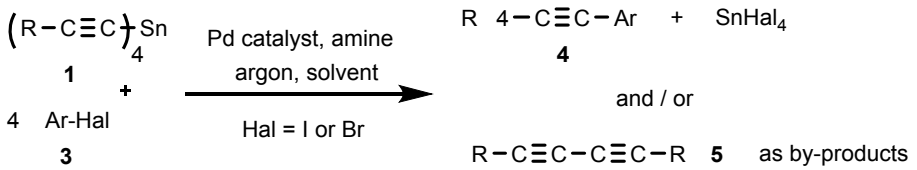
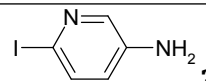
Supplementary Table 1. The results of the optimization experiments



Unless otherwise stated, the reaction conditions were as follows: tetraalkynylstannane **1** (0.038 mmol), aryl halide **3** (0.153 mmol), a catalyst (5 mol % with respect to aryl halide **3**), an amine (0.153 mmol) and the corresponding solvent (2 mL). The yields were determined by GCMS, except for the values marked with stars(*) which are isolated yields

No	Acetylene **1**	Aryl Halide **3**	Solvent	Catalyst (and amine, if any)	Temp. °C	Time, h	Yield of **4**, %	Yield of diacetylene by-product **5**, %
1	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	dioxane	$Pd(PPh_3)_2Cl_2$	80	2,5	0	4
		4-$NO_2C_6H_4$–I **3d**	dioxane	$Pd(PPh_3)_2Cl_2$	80	4,5	0	4
2	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	PhMe	$Pd(PPh_3)_2Cl_2$	80-100	3,5	0	8,7
3	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	PhMe	$Pd(PPh_3)_2Cl_2$ +PPh_3	80	2,5	0	10
4	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	PhMe	$Pd(PPh_3)_2Cl_2$, Et_3N	80	3	13	9,5
					80	12	14	9
5	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	Pure Et_3N	$Pd(PhCN)_2Cl_2$	80	3	68	2
					80	5	76	2,5
					80	12	83	2,6
6	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	Pure Et_3N	$Pd(PPh_3)_2Cl_2$	80		97	3
					80	2,5	95,5	4,5
7	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	Pure Et_3N	$Pd(PPh_3)_2Cl_2$ + PPh_3	80	3,5	2	2
8	$(PhC{\equiv}C)_4Sn$ **1a**	4-MeC_6H_4–I **3b**	Pure Et_3N	$Pd(PPh_3)_2Cl_2$	80	0,25	0	0
					80	0,5	4	0
					80	1	17	0
					80	2	31	0
					80	3,5	34	1,9
					80	5	41	1,7
					80	11	45	1,5

9	$(PhC{\equiv}C)_4Sn$ **1a**	 **3e**	Pure Et_3N	$Pd(PPh_3)_2Cl_2$	80	2	0	0
					80	6,5	8	3,5
10	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	PhMe-Et_3N (1:1)	$Pd(PPh_3)_2Cl_2$	80	1	62	9
					80	2	76	9
					80	4	78	9
					80	6	79	9
11	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	Pure Et_3N	$Pd(PPh_3)_2Cl_2$	75	2	70	3,5
					75	3	81	4,5
					75	5	87	4
					75	8	89	4
					75	16	95	4
12	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	Pure Et_2NH	$Pd(PPh_3)_2Cl_2$	75	3	0	3
					75	5	4	5
					75	9	42	6,5
					75	16	34	6,5
13	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	PhMe, DABCO (0,018г)	$Pd(PPh_3)_2Cl_2$	75	1	0	5
					75	2	17	8,5
					75	3	23	8,5
					75	9,3	20	7
14	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	PhMe, DABCO (0,15г)	$Pd(PPh_3)_2Cl_2$	75	1	50	9,5
					75	2	52	9,5
					75	3	55	9,5
					75	5	52	10
					75	9,5	53	9,5
					75	16	55	9,5
15	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	Pure Et_3N	$Pd(PPh_3)_2Cl_2$	80	0,5	49	3,4
					80	1	84	1,7
					80	2	91	5,2
					80	3	93	6,6
16	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	THF	$Pd(PPh_3)_2Cl_2$ DABCO	80	0,5	81	9
					80	1	87	8
					80	2	85	9

					80	4	83	10
17	$(PhC{\equiv}C)_4Sn$ **1a**	2-$NO_2C_6H_4$–I **3k**	Pure Et_3N	$Pd(PPh_3)_2Cl_2$	80	2	36	8,5
					80	3	42	9
					80	4	42	9
					80	6	39	8,6
					80	7	40,5	8
18	$(PhC{\equiv}C)_4Sn$ **1a**	2-IC_6H_4COOH **3l**	Pure Et_3N	$Pd(PPh_3)_2Cl_2$	80	4	63	40
19	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	THF	$Pd(PPh_3)_2Cl_2$ 2-methylimidazole	80	0,5	0	0
						2	0	0
						5	0	0
20	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	THF	$Pd(PPh_3)_2Cl_2$ Isophorone diamine	80	0,5	0	5,5
						2	4,5	9
						3	3	4
						5	6,5	8
						7	8,5	8,5
21	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	THF	$Pd(PPh_3)_2Cl_2$ Et_3N	80	0,5	0	8,2
						2	11	17
						3	8	17,5
						5	20	22
						7	16,5	19
22	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	dioxane	$Pd(PPh_3)_2Cl_2$ DABCO	100	0,5	67	6
						1	65	4
						2	75,5	6
						3	75	5
						5	73	5
23	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	dioxane	$Pd(PPh_3)_2Cl_2$ 2-methylimidazole	100	0,5	0	0
						1	0,02	5,5
						2	0	–
						5	0	–
24	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	dioxane	$Pd(PPh_3)_2Cl_2$ Isophorone diamine	100	1	8	3
						2	27	4
						3	38	4

						5	63	4,5
						7	75	5
						8	81	5
						9	86	5
25	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	dioxane	$Pd(PPh_3)_2Cl_2$ Et_3N	100	1	18	4
						2	44	6
						3	42	7
						5	42	7
26	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	PhMe	$Pd(PPh_3)_2Cl_2$ DABCO	100	0,5	87	8
						1	86	9
						2	87	8
27	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	PhMe	$Pd(PPh_3)_2Cl_2$ Isophorone diamine	100	1	10,5	7
						2	22	6,5
						3	36	7
						5	39	6
						7	54	7
						9	50	6
28	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	PhMe	$Pd(PPh_3)_2Cl_2$ Et_3N	100	1	23	14
						2	22	11
						3	18	12
						5	27	14
						7	28	15
						9	24	14
29	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	PhMe	$Pd(PPh_3)_2Cl_2$ Et_2NH	100	1	17	8
						2	26,5	8
						3	44	9
						5	50	10
						7	55	10
						9	53	9
30	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	dioxane	$Pd(PPh_3)_2Cl_2$ N-methylmorpholine	100	2	63	2
						3	73	1,8
						5	66	2

31	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	dioxane	$Pd(PPh_3)_2Cl_2$ pyridine	100	2	0	1
						3	0	1,3
32	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	dioxane	$Pd(PPh_3)_2Cl_2$ DABCO	100	0,5	78	5
						2	78	7
						3	69	8
						5	71	9,5
						7	73	9
33	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	dioxane	$Pd(PPh_3)_2Cl_2$ Et_2NH	100	0,5	47	2
						1	70	3
						2	79	2
						3	84	2
						5	82	2
						7	79	3
34	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	dioxane	$Pd(PPh_3)_2Cl_2$ morpholine	100	0,5	33,5	2
						1	54	4
						2	52	3
						3	56	4
						5	57	4
						7	69	5
35	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	EtOAc	$Pd(PPh_3)_2Cl_2$ DABCO	80	0,5	87	4
						1	88	4
						2	88,6	4
						3	85	4
36	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	EtOAc	$Pd(PPh_3)_2Cl_2$ Et_2NH	80	1	7	0
						2	52	1
						3	72	1
						5	81	1
						7	88	1
						9	91	1
37	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	EtOAc	$Pd(PPh_3)_2Cl_2$ N-methylmorpholine	80	1	27	3
						2	36	3
						3	25	2

						5	28	5
38	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	EtOAc	$Pd(PPh_3)_2Cl_2$ Et3N	80	0,5	40	4
						1	84	4
						2	87	4
						3	86,5	5
39	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	MeCN	$Pd(PPh_3)_2Cl_2$ DABCO	85	1	57	6
						2	51	6
						5	63	6,5
40	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	MeCN	$Pd(PPh_3)_2Cl_2$ N-methylmorpholine	85	1	44	4
						2	58	4,5
						5	57	5
41	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	MeCN	$Pd(PPh_3)_2Cl_2$ Et_3N	85	1	58	13
						2	59	14
						5	68	13
42	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	MeCN	$Pd(PPh_3)_2Cl_2$ Et_2NH	85	1	78	6
						2	79,5	7
						5	89	6
43	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PPh_3)_2Cl_2$ DABCO	125	1	83	6
						2	87	5
						5	89	10,5
44	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PPh_3)_2Cl_2$ N-methylmorpholine	125	1	12,5	0
						2	5	0
45	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PPh_3)_2Cl_2$ Et_3N	125	1	90	1,5
						2	98	2
						5	98	2
46	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PPh_3)_2Cl_2$ Et_2NH	125	0,5	83	6
						1	98,3	1,7
						2	98	2
47	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	Et_2O	$Pd(PPh_3)_2Cl_2$ DABCO	35	1	0	0
						2	10	3
						3	47,5	5
						5	70,6	5,7

						7	71,7	6
48	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	Et_2O	$Pd(PPh_3)_2Cl_2$ N-methylmorpholine	35	1	0	0
						3	0	0
						5	0	0
49	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	Et_2O	$Pd(PPh_3)_2Cl_2$ Et_3N	35	1	0	0
						3	0	0
						5	0	3
50	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	Et_2O	$Pd(PPh_3)_2Cl_2$ Et_2NH	35	1	0	0
						3	0	0
						5	0	0
51	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PPh_3)_2Cl_2$ DABCO	100	0,5	60	4
						1	55	5
						2	68	5
						5	63	6
52	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PPh_3)_2Cl_2$ Et_3N	100	0,5	71	2
						1	77	3
						2	76	2
						5	85	2
						7	89	11
53	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PPh_3)_2Cl_2$ Et_2NH	100	0,5	13	1
						1	81	2
						2	98	2
54	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PPh_3)_2Cl_2$ piperidine	100	2	62	1
						3	58	1,5
						5	52	3
55	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PPh_3)_2Cl_2$ $PhCH_2NH_2$	100	2	14	2,5
						3	25	2
						5	41	3
56	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PPh_3)_2Cl_2$ $H_2NCH_2CH_2NH_2$	100	2	0	0
						3	0	0
57	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PPh_3)_2Cl_2$ Bu_3N	100	1	53	4
						2	93	3

						3	97	3
58	$(PhC\equiv C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	dioxane	$Pd(PPh_3)_2Cl_2$ piperidine	100	0,5	41	2
						2	55	3
						3	46	3
						The product of piperidine alkylation was formed in up to 48,5% yield		
59	$(PhC\equiv C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	dioxane	$Pd(PPh_3)_2Cl_2$ $PhCH_2NH_2$	100	0,5	6	6
						2	32	8
						3	33	7
						5	56	7
						The unidentified by-product is formed		
60	$(PhC\equiv C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	dioxane	$Pd(PPh_3)_2Cl_2$ $(PhCH_2)_2NH$	100	0,5	0	0
						2	10	1,7
						3	23	2
						5	26	2
						Significant amounts of dibenzyl were detected		
61	$(PhC\equiv C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	dioxane	$Pd(PPh_3)_2Cl_2$ Bu_3N	100	1	72	2
						2	86	2,4
						3	87	2,4
						5	89	2
62	$(PhC\equiv C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	PhMe	$Pd(PPh_3)_2Cl_2$ piperidine	110	1	64	4
						3	46,5	5
						5	49	6
63	$(PhC\equiv C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	PhMe	$Pd(PPh_3)_2Cl_2$ $(PhCH_2)_2NH$	110	1	3,4	10
						3	11	10,6
						5	8	14
64	$(PhC\equiv C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	PhMe	$Pd(PPh_3)_2Cl_2$ Bu_3N	110	1	21	9
						3	28	9,5
						5	31	10
65	$(PhC\equiv C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	PhMe	$Pd(PPh_3)_2Cl_2$ $PhCH_2NH_2$	110	1	16,6	11
						2	24	10,5

						3	35	10
						5	43	11
66	$(PhC{\equiv}C)_4Sn$ **1a**	2-$NO_2C_6H_4$–I **3k**	BuOAc	$Pd(PPh_3)_2Cl_2$ Et_2NH	100	0,5	5	0,6
						1	43	1,6
						2	79	2
						3	74	2
						5	77	2
						7	67,5	2
						9	81	2
						10	83	2
67	$(PhC{\equiv}C)_4Sn$ **1a**	4-MeC_6H_4–I **3b**	BuOAc	$Pd(PPh_3)_2Cl_2$ Et_2NH	100	0,5	0	0
						1	3	1
						2	19	3,7
						3	36	5,6
						5	47	5
						7	45	5,4
						9	54	7
68	$(PhC{\equiv}C)_4Sn$ **1a**	2-IC_6H_4COOH **3l**	BuOAc	$Pd(PPh_3)_2Cl_2$ Et_2NH	100	0,5	0	11,5
						1	0	100
						2	0	100
						3	0	100
						5	0	100
69	$(PhC{\equiv}C)_4Sn$ **1a**	4-MeC_6H_4–Br **3h**	BuOAc	$Pd(PPh_3)_2Cl_2$ Et_2NH	100	0,5	0	0
						1	0	6
						2	0	18
						3	0	23
						5	10	30
						7	8	11
						Up to 57% of unidentified side product		
70	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$PdCl_2$, Et_2NH	100	1	0	0
						2	0	0
						3	0	0

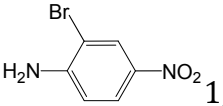
						5	10,5	0
						7	13	0
71	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$PdCl_2$, Et_3N	100	0,5	–	–
						1	61	0
						2	65	0
						3	72	0
						5	66	0
						7	77	0
						9	74	0
72	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$PdCl_2$ DABCO	100	1	53	4
						2	57	4
						3	56,5	4,5
						4	62	4
						7	53,5	5
						9	53,5	5
73	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$PdCl_2$ Bu_3N	100	1	38	0
						2	46	0
						3	49	0
						5	55	0
						7	60	0
						9	59,5	0
74	$(PhC{\equiv}C)_4Sn$ **1a**	4-$MeOC_6H_4$–Br **3i**	BuOAc	$Pd(PPh_3)_2Cl_2$ Et_2NH	100	1	0	1,5
						2	0,5	6
						3	4,5	7
						5	7	10,5
75	$(PhC{\equiv}C)_4Sn$ **1a**	4-$MeOC_6H_4$–Br **3i**	BuOAc	$Pd(PPh_3)_2Cl_2$ DABCO	100	1	11	9
						2	11	8
						3	15	9,5
						5	12	6,5
76	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PhCN)_2Cl_2$ Et_2NH	100	0,5	0	0
						1	0	0
						3	0	0

						5	7	0
77	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PhCN)_2Cl_2$ Et_3N	100	1	14	0
						2	71	0,3
						3	72	0
						5	71	1
78	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PhCN)_2Cl_2$ Bu_3N	100	1	22	1,7
						3	43,5	1,8
						5	50	1,5
						7	48,5	3
79	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PhCN)_2Cl_2$ DABCO	100	1	56	4
						3	50,5	3,4
						5	51	4
80	(tBuO$CH_2C{\equiv}C)_4Sn$ **1e**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PPh_3)_2Cl_2$ Et_2NH	100	1	9	0
						2	32,5	0
						3	26,5	0
81	(tBuO$CH_2C{\equiv}C)_4Sn$ **1e**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PPh_3)_2Cl_2$ DABCO	100	1	73,6	5
						2	72	5,5
						3	69	5
						5	65	5,5
82	$(C_8H_{17}C{\equiv}C)_4Sn$ **1g**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PPh_3)_2Cl_2$ Et_2NH	100	0,5	0	0
						1	0	0
						3	11,6	0
						5	30	0,8
						7	50	1
						9	47	6
83	$(C_8H_{17}C{\equiv}C)_4Sn$ **1g**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PPh_3)_2Cl_2$ DABCO	100	1	35,6	6,6
						3	45	5,5
						5	52	7
						Another by-product was also detected		
84	$(PhC{\equiv}C)_4Sn$ **1a** 0,25 mmol	4-$NO_2C_6H_4$–I **3d** 1 mmol	BuOAc 15 mL	$Pd(PPh_3)_2Cl_2$ Et_2NH 1eq, 103 μL	100	5	69	2
85	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PPh_3)_2Cl_2$	100	6	97	1,2

	0,25 mmol	1 mmol	15 mL	Et_2NH 145eq, 12 mL			86.4*	
86	$(PhC{\equiv}C)_4Sn$ **1a** 0,57 mmol	2-Br-pyridine **3g** 2,28 mmol	BuOAc 1 mL	$Pd(PPh_3)_2Cl_2$ Et_2NH 38eq, 9 mL	100	5	14,6*	–
87	$(PhC{\equiv}C)_4Sn$ **1a** 0,038 mmol	4-BrC_6H_4I **3c** 0,153 mmol	BuOAc 0,1 mL	$Pd(PPh_3)_2Cl_2$ Et_2NH 57eq, 0,9 mL	100	1	81,8	0
						2,5	84,4	2,4
						4.5	90.5	3,3
88	$(PhC{\equiv}C)_4Sn$ **1a** 0,076 mmol	4-BrC_6H_4I **3c** 0,153 mmol	BuOAc 0,1 mL	$Pd(PPh_3)_2Cl_2$ Et_2NH 57eq, 0,9 mL	100	1	80,0	16,4
						2,5	83,3	16,7
						4.5	84,4	15,6
89	$(PhC{\equiv}C)_4Sn$ **1a** 0,076 mmol	2,6-Br_2-4-$NO_2C_6H_4I$ **3j** 0,153 mmol	BuOAc 0,1 mL	$Pd(PPh_3)_2Cl_2$ Et_2NH 57eq, 0,9 mL	100	2,5	84,4	2,4
90	$(PhC{\equiv}C)_4Sn$ **1a** 0,382 mmol	4-$NO_2C_6H_4$–I **3d** 1,53 mmol	BuOAc 1 mL	$Pd(PPh_3)_2Cl_2$ Et_3N 42eq, 9 mL	100	4.5	76,3* (52,8% after recrystallization)	
91	$(PhC{\equiv}C)_4Sn$ **1a** 0,382 mmol	4-$NO_2C_6H_4$–I **3d** 1,53 mmol	BuOAc 1 mL	$Pd(PPh_3)_2Cl_2$ Et_3N 42eq, 9 mL	100	4.5	90.5	3,3
92	$(PhC{\equiv}C)_4Sn$ **1a** 0,038 mmol	4-$NO_2C_6H_4$–I **3d** 0,153 mmol	BuOAc 0,1 mL	$Pd(PPh_3)_2Cl_2$ Et_2NH 57eq, 0,9 mL	100	6	96.4	1,5
93	$(PhC{\equiv}C)_4Sn$ **1a** 0,038 mmol	2-Br-pyridine **3g** 0,153 mmol	BuOAc 0,1 mL	$Pd(PPh_3)_2Cl_2$ Et_2NH 57eq, 0,9 mL	100	6,5	72,5	12
94	$(PhC{\equiv}C)_4Sn$ **1a** 0,382 mmol	4-$NO_2C_6H_4$–I **3d** 1,53 mmol	BuOAc 0,5 mL	$Pd(PPh_3)_2Cl_2$ Et_2NH 23eq, 4 mL	100	6	70.8*	–
95	$(PhC{\equiv}C)_4Sn$ **1a** 0,382 mmol	4-$NO_2C_6H_4$–I **3d** 1,53 mmol	DMF 2 mL	$Pd(PPh_3)_2Cl_2$ Et_2NH 12eq, 2 mL	100	6	57,4*	–
96	$(PhC{\equiv}C)_4Sn$ **1a** 0,420 mmol	PhI **3a** 1,53 mmol	BuOAc 0,5 mL	$Pd(PPh_3)_2Cl_2$ Et_2NH 23eq 4 mL	100	6	84,1*	
97	$(PhC{\equiv}C)_4Sn$ **1a** 0,042 mmol	PhI **3a** 0,153 mmol	DMF, 0,5 mL	$Pd(PPh_3)_2Cl_2$ Et_2NH 32eq, 0,5 mL	100	6	88	12
98	$(PhC{\equiv}C)_4Sn$ **1a** 0,401 mmol	PhI **3a** 1,53 mmol	DMF, 2 mL	$Pd(PPh_3)_2Cl_2$ Et_2NH 12eq, 2 mL	100	3	69*	
99	$(PhC{\equiv}C)_4Sn$ **1a** 0,401 mmol	PhI **3a** 1,53 mmol	DMF, 2 mL	$Pd(PPh_3)_2Cl_2$ Et_2NH 12eq, 2 mL	100	6	68*	
100	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	DMF, 2 mL	$Pd(PPh_3)_2Cl_2$	100	3	68	32

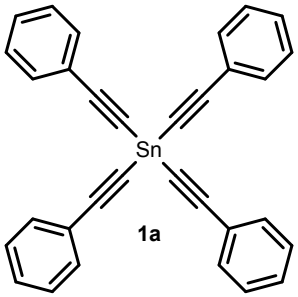
	0,401 mmol	1,53 mmol		Et_2NH 12eq, 2 mL			32*	
101	$(PhC{\equiv}C)_4Sn$ **1a** 0,401 mmol	4-MeC_6H_4–I **3b** 1,53 mmol	DMF, 2 mL	$Pd(PPh_3)_2Cl_2$ Et_2NH 12eq, 2 mL	100	6	84 32*	
102	$(PhC{\equiv}C)_4Sn$ **1a**	PhI **3a,** 0,153 mmol	DMF, 0,5 mL	$Pd(PPh_3)_2Cl_2$ Et_3N 23.4eq, 0,5 mL	100	2,5	86,6	1,8
						3,5	83,5	6,8
						5	84,1	9,1
103	$(PhC{\equiv}C)_4Sn$ **1a** 0,04 mmol	4-MeC_6H_4–I **3b** 0,153 mmol	DMF, 0,5 mL	$Pd(PPh_3)_2Cl_2$ Et_3N 23.4eq, 0,5 mL	100	2,5	94,5	2,4
						3,5	92,7	3,7
						5	95	4,4
104	$(PhC{\equiv}C)_4Sn$ **1a** 0,04 mmol	4-$NO_2C_6H_4$–I **3d** 0,153 mmol	DMF 0,5 mL	$Pd(PPh_3)_2Cl_2$ Et_3N 23.4eq, 0,5 mL	100	2,5	88,9	8,8
						3,5	84,8	13,2
						5	87	13
105	$(PhC{\equiv}C)_4Sn$ **1a** 0,42 mmol	4-MeC_6H_4–I **3b** 1,53 mmol	DMF 2 mL	$Pd(PPh_3)_2Cl_2$ Et_3N 9.4eq, 2 mL	100	2	40	30
106	$(PhC{\equiv}C)_4Sn$ **1a** 0,401 mmol	4-MeC_6H_4–I **3b** 1,53 mmol	DMF 5 mL	$Pd(PPh_3)_2Cl_2$ Et_3N 23.4eq, 5 mL	100	5	40	20
107	$(PhC{\equiv}C)_4Sn$ **1a** 0,04 mmol	4-$NO_2C_6H_4$–I **3d** 0,153 mmol	BuOAc 0,1 mL	$Pd(PPh_3)_2Cl_2$, Pr_2NH 42,9eq, 0,9 mL	100	0,5	90,5	1,2
						1	99	1
108	$(PhC{\equiv}C)_4Sn$ **1a** 0,04 mmol	4-$NO_2C_6H_4$–I **3d** 0,153 mmol	BuOAc 0,1 mL	$Pd(PPh_3)_2Cl_2$ Pr_2NH 42,9eq, 0,9 mL +2 mg $NaBH_4$	100	0,5	91,5	1,5
109	$(PhC{\equiv}C)_4Sn$ **1a** 0,04 mmol	4-$NO_2C_6H_4$–I **3d** 0,153 mmol	BuOAc 0,1 mL	$Pd(PPh_3)_2Cl_2$ i-Pr_2NH 41,9eq 0,9 mL	100	0,5	82,8	1,3
						1	94,7	0,9
						2	96,4	1
110	$(PhC{\equiv}C)_4Sn$ **1a** 0,04 mmol	4-BrC_6H_4I **3c** 0,153 mmol	BuOAc 0,1 mL	$Pd(PPh_3)_2Cl_2$ i-Pr_2NH 41,9eq 0,9 mL	100	0,5	2	0,9
						1	53,3	1,9
						2	64,1	2,6
111	$(PhC{\equiv}C)_4Sn$ **1a** 0,04 mmol	4-$NO_2C_6H_4$–I **3d** 0,153 mmol	BuOAc 0,98 mL	$Pd(PPh_3)_2Cl_2$ Pr_2NH 1:1 21 µL	100	1	99,5	0,5
112	$(PhC{\equiv}C)_4Sn$ **1a**	4-$NO_2C_6H_4$–I **3d**	BuOAc 0,9 mL	$Pd(PPh_3)_2Cl_2$	100	1	99,2(0,8)	0,8

	0,04 mmol	0,153 mmol		Pr_2NH 1:5 105 μL				
113	$(PhC{\equiv}C)_4Sn$ **1a** 0,04 mmol	4-$NO_2C_6H_4$–I **3d** 0,153 mmol	BuOAc 0,79 mL	$Pd(PPh_3)_2Cl_2$ Pr_2NH 1:10 210 μL	100	1	98,8(1,2)	1,2
114	$(PhC{\equiv}C)_4Sn$ **1a** 0,04 mmol	4-$NO_2C_6H_4$–I **3d** 0,153 mmol	BuOAc 0,16 mL	$Pd(PPh_3)_2Cl_2$ Pr_2NH 1:40 0,84 mL	100	1	98,9(1,1)	1,1
115	$(PhC{\equiv}C)_4Sn$ **1a** 0,04 mmol	4-$NO_2C_6H_4$–I **3d** 0,153 mmol	Pure Pr_2NH 1 mL	$Pd(PPh_3)_2Cl_2$	100	1	98,3(1,6)	1,6
						2	98,3(1,7)	1,7
116	$(PhC{\equiv}C)_4Sn$ **1a** 0,401 mmol	PhI **3a** 1,53 mmol	BuOAc 3 mL	$Pd(PPh_3)_2Cl_2$ Pr_2NH 1:10 2,1 mL	100	5	86,1*	–
117	$(PhC{\equiv}C)_4Sn$ **1a** 0,04 mmol	4-$NO_2C_6H_4$–I **3d** 0,153 mmol	BuOAc 0,96 mL	$Pd(PPh_3)_2Cl_2$ Bu_3N 1:1, 36 μL	100	0,5	27,8	0,3
						1	51,5	0,1
						2	63,6	1
						4	75,7	2
118	$(PhC{\equiv}C)_4Sn$ **1a** 0,04 mmol	4-$NO_2C_6H_4$–I **3d** 0,153 mmol	BuOAc 0,82 mL	$Pd(PPh_3)_2Cl_2$ Bu_3N 1:5 82 μL	100	0,5	93,9	0,5
						1	99,5	0,5
119	$(PhC{\equiv}C)_4Sn$ **1a** 0,04 mmol	4-$NO_2C_6H_4$–I **3d** 0,153 mmol	BuOAc 0,64 mL	$Pd(PPh_3)_2Cl_2$ Bu_3N 1:10 365 μL	100	0,5	98,8	0,5
						1	99,5	0,5
120	$(PhC{\equiv}C)_4Sn$ **1a** 0,04 mmol	4-$NO_2C_6H_4$–I **3d** 0,153 mmol	BuOAc 0,1 mL	$Pd(PPh_3)_2Cl_2$ Bu_3N 1:25 0,91 mL	100	0,5	58,5	5
						1	72,6	3,6
						2	78,3	6,7
						4	83,7	9,2
121	$(PhC{\equiv}C)_4Sn$ **1a** 0,42 mmol	PhI **3a** 1,53 mmol	BuOAc 3 mL	$Pd(PPh_3)_2Cl_2$ Pr_2NH 1:10 2,1 mL	100	4	78,1*	–
122	$(PhC{\equiv}C)_4Sn$ **1a** 0,42 mmol	4-MeC_6H_4–I **3b** 1,53 mmol	BuOAc 3 mL	$Pd(PPh_3)_2Cl_2$ Pr_2NH 1:10 2,1 mL	100	5	80,7*	–
123	$(PhC{\equiv}C)_4Sn$ **1a** 0,42 mmol	4-$NO_2C_6H_4$–I **3d** 1,53 mmol	BuOAc 8,85 mL	$Pd(PPh_3)_2Cl_2$ TMEDA 1:5 1,15 mL	100	1,3	87,2*	–
124	$(PhC{\equiv}C)_4Sn$ **1a** 0,42 mmol	4-$NO_2C_6H_4$–I **3d** 1,53 mmol	BuOAc 7,9 mL	$Pd(PPh_3)_2Cl_2$ Pr_2NH 1:10 2,1 mL	100	1,25	92.9*	–
125	$(PhC{\equiv}C)_4Sn$ **1a** 0,287 mmol	PhI **3a** 1,043 mmol	BuOAc 6 mL	$Pd(PPh_3)_2Cl_2$ TMEDA 1:5 782 μL	100	5	68.4*	–

126	$(PhC{\equiv}C)_4Sn$ **1a** 0,287 mmol	4-BrC_6H_4I **3c** 1,043 mmol	BuOAc 6 mL	$Pd(PPh_3)_2Cl_2$ TMEDA 1:5 782 µL	100	5	80*	–
127	$(PhC{\equiv}C)_4Sn$ **1a** 0,287 mmol	4-BrC_6H_4I **3c** 1,043 mmol	BuOAc, 6 mL	$Pd(PPh_3)_2Cl_2$ Pr_2NH 1:10 1,43 mL	100	5	80*	–
128	$(PhC{\equiv}C)_4Sn$ **1a** 0,287 mmol	4-$BrC_6H_4C(O)Me$ **3o** 1,043 mmol	BuOAc, 6 mL	$Pd(PPh_3)_2Cl_2$ TMEDA 1:5 782 µL	100	5	65*	–
129	$(PhC{\equiv}C)_4Sn$ **1a** 0,287 mmol	2-IC_6H_4COOEt **3m** 1,043 mmol	BuOAc	$Pd(PPh_3)_2Cl_2$ TMEDA 1:10	100	5	81*	–
130	$(PhC{\equiv}C)_4Sn$ **1a** 0,287 mmol	2-$NO_2C_6H_4$–I **3k** 1,043 mmol	BuOAc	$Pd(PPh_3)_2Cl_2$ Pr_2NH 1:10	100	3.5	87.9*	–
131	$(t\text{-}BuOCH_2C{\equiv}C)_4Sn$ **1e**	4-$NO_2C_6H_4$–I **3d**	BuOAc	$Pd(PPh_3)_2Cl_2$ Pr_2NH 1:10	100	5.5	48,6*	–
132	$(PhC{\equiv}C)_4Sn$ **1a** 0,287 mmol	 1,04 3 mmol	BuOAc	$Pd(PPh_3)_2Cl_2$ Pr_2NH 1:10	100	5	Traces by GC-MS	–

2. **Synthesis of starting tetraalkynylstannanes 1**

Synthesis of tetra(phenylethynyl)tin 1a



1a

A three-necked, round-bottomed flask equipped with a dropping funnel, a reflux condenser, with argon gas inlet tube and a magnetic stirrer was flushed with argon and charged with anhydrous $ZnCl_2$ (2.50 g, 0.018 mol), dry Et_2NH (2.7 g, 0.036 mol) and dry toluene (15 mL). The reaction mixture was heated up to the moment when all the $ZnCl_2$ will react to give a complex compound with Et_2NH (~ 50 °C, 0.25 h). The reaction was carried out under argon. The mixture was allowed to cool to the ambient temperature, and phenylacetylene (1.73 g, 0.017 mol) was added. Then, a solution of 1.67 g (0.0064 mol) of $SnCl_4$ in PhMe (10 mL) was added slowly, dropwise under vigorous stirring. The mixture was stirred for 20 min, then the upper layer was separated and the lower layer was extracted with PhMe. The combined toluene extracts were passed through a column of silanized silica and evaporated in vacuo. The residue was recrystallized from heptane to give a white solid of **1a**. Tetra(phenylethynyl)tin **1a** is a colorless crystalline solid, mp 174 °C, yield was 1,84 g (83%).

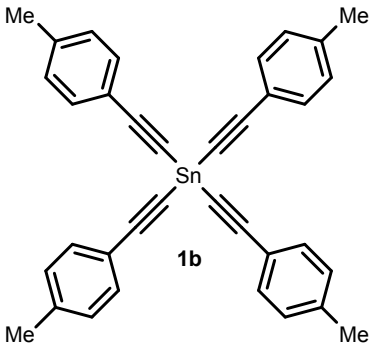
IR spectrum, ν, cm^{-1}: 2152 (C≡C).

^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 7.59–7.61 (8H, m), 7.32–7.40 (12H, m).

^{13}C NMR (100 MHz, $CDCl_3$): *δ* 132.47, 129.37, 128.38, 122.18, 109.78, 85.45.

^{119}Sn NMR (149 MHz, $CDCl_3$): *δ* -332.29.

Synthesis of tetra(4-methylphenylethynyl)tin 1b



1b

Tetra(4-methylphenylethynyl)tin **1b** was prepared according to a similar procedure as for **1a**, using 0,017 mol (1,97 g) of 4-(methylphenyl)acetylene, $SnCl_4$ (0,0064 mol, 1,67 g), anhydrous zinc chloride (2.5 g, 0,018 mol), dry diethylamine (2.7 g, 0,036 mol) and dry toluene (15 mL). The yield was 1,73 g (70%), mp 202-203 °C.

IR spectrum, ν, cm^{-1}: 2150 (C≡C).

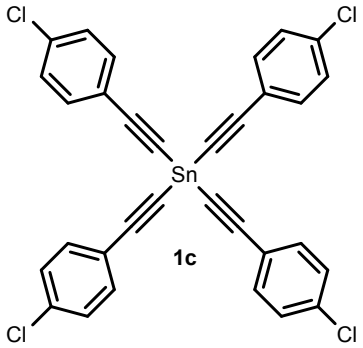
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 – 7.49 (2H, m), 7.12 – 7.15 (2H, m), 2.37 (3H, s).

^{13}C NMR (100 MHz, $CDCl_3$): δ 139.60, 132.41, 129.16, 119.22, 109.93, 84.85.

^{119}Sn NMR (149 MHz, $CDCl_3$), δ, ppm: -322.29.

MS (*m/z*, EI, 70 eV): 579 $[M]^+$ (9), 460 (27), 350 (89), 230 (100), 120 (40), 115 (51).

Synthesis of tetra(4-chlorophenylethynyl)tin 1c



1c

Tetra(4-chlorophenylethynyl)tin **1c** was prepared according to a similar procedure as for **1a**, using 0,017 mol of 4-(chlorophenyl)acetylene, $SnCl_4$ (0,0064 mol, 1,67 g), anhydrous zinc chloride (2.5 g, 0,018 mol), diethylamine (2.7 g, 0,036 mol) and toluene (15 mL). The yield was 2,0 g (72%), mp 161 °C.

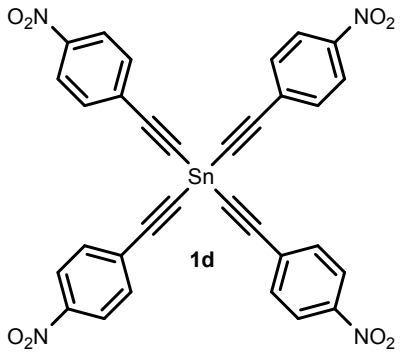
IR spectrum, ν, cm^{-1}: 2154 (C≡C).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50 – 7.53 (2H, m), 7.31 – 7.34 (2H, m).

^{13}C NMR (100 MHz, $CDCl_3$): δ 135.75, 133.80, 133.71, 128.86, 120.41, 108.80, 86.00.

MS (*m/z*, EI, 70 eV): 658 $[M]^+$ (7), 390 (71), 270 (100), 200 (49), 155 (60), 136 (41), 120(19).

Synthesis of tetra(4-nitrophenylethynyl)tin 1d



A three-necked, round-bottomed flask equipped with a dropping funnel, a reflux condenser, with argon gas inlet tube and a magnetic stirrer was flushed with argon and charged with anhydrous $ZnCl_2$ (0.66 g, 0.00484 mol), Et_2NH (1 mL, 0.00963 mol) and dry toluene (8 mL). The reaction was carried out under argon. The reaction mixture was heated up to the moment when all the $ZnCl_2$ will react to give a complex compound with Et_2NH (~ 50 °C, 0.25 h). The mixture was allowed to cool to the ambient temperature, and 4-nitrophenylacetylene (0.00448 mol) was added. Then, $SnCl_4$ (0.2 mL) was added dropwise under vigorous stirring. The mixture was stirred for 2 h, then the upper layer was separated and the lower layer was extracted with PhMe. The combined toluene extracts were passed through a column of silanized silica and evaporated *in vacuo*. The residue was recrystallized from heptane to give a white solid of **1d**. The product was purified by recrystallization from toluene. Light yellow crystal. mp 150 ºC (toluene).

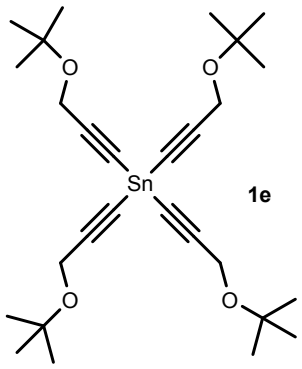
IR spectrum, ν, cm^{-1}: 2162.2 (C≡C).

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.22 – 8.25 (8H, m), 7.74 – 7.76 (8H, m).

^{13}C NMR (100 MHz, $CDCl_3$): δ 148.11, 133.36, 128.04, 123.80, 108.06, 89.30.

^{119}Sn NMR (149 MHz, $CDCl_3$): δ -336.76.

Synthesis of tetra(*tert*-buthoxypropynyl)tin 1e



A three-necked, round-bottomed flask equipped with a dropping funnel, a reflux condenser, with argon gas inlet tube and a magnetic stirrer was flushed with argon and charged with anhydrous $ZnCl_2$ (0.66 g, 0.00484 mol), Et_2NH (1 mL, 0.00963 mol) and dry toluene (5 mL). The reaction was carried out under argon. The reaction mixture was heated up to the moment when all the $ZnCl_2$ will react to give a complex compound with Et_2NH (~ 50 °C, 0.25 h). The mixture was allowed to cool to the ambient temperature, and *t*-buthoxypropyne (0,38 g, 0.003 mol) was added. Then, $SnCl_4$ (0.196 g, 0.00075 mol) was added dropwise under vigorous stirring. The mixture was stirred for 1 h, then the upper layer was separated and the lower layer was extracted with PhMe. The combined toluene extracts were passed through a column of silanized silica and evaporated *in vacuo*. The residue was recrystallized from heptane to give a white solid of **1e**. The yield was 55%. Colorless oil.

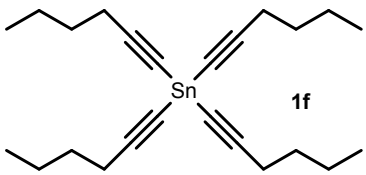
^{1}H NMR (400 MHz, $CDCl_3$): δ 4.06 (2H, s), 1.20 (9H, s).

^{13}C NMR (100 MHz, $CDCl_3$): δ 110.61, 75.62, 68.43, 38.93, 9.44.

^{119}Sn NMR (149 MHz, $CDCl_3$): δ -344.22 – (-343.64) (m).

IR (CCl_4): 2175 (C≡C)

Synthesis of tetra(hexyn-1-yl)tin 1f



Tetra(hexyn-1-yl)tin **1f** was prepared according to a similar procedure as for **1a**, using 0,017 mol (1.39 g) of 1-hexyne, $SnCl_4$ (0,0064 mol, 1,67 g), anhydrous zinc chloride (2.5 g, 0,018 mol), dry diethylamine (2.7 g, 0,036 mol) and dry hexane (20 mL). The yield was 1.26 g (67%). Colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$): δ 2.20 (2H, t), 1.32 – 1.52 (4H, m), 0.86 (3H, t).

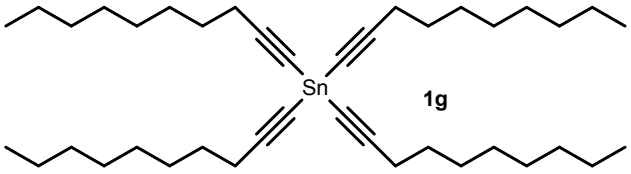
^{13}C NMR (100 MHz, $CDCl_3$): δ 111.40, 75.88, 30.23, 21.69, 19.55, 13.36.

^{119}Sn NMR (149 MHz, $CDCl_3$): δ -51.51 (m).

IR (CCl_4): 2165 (C≡C).

MS (*m/z*, EI, 70 eV): 444 [M^+] (10), 363 (100), 282 (22), 201 (20), 120 (10).

Synthesis of tetra(decyn-1-yl)tin 1g



A three-necked, round-bottomed flask equipped with a dropping funnel, a reflux condenser, with argon gas inlet tube and a magnetic stirrer was flushed with argon and charged with anhydrous $ZnCl_2$ (0.54 g, 0.004 mol), Et_2NH (0.8 mL, 0.008 mol) and dry toluene (5 mL). The reaction was carried out under argon. The reaction mixture was heated up to the moment when all the $ZnCl_2$ will react to give a complex compound with Et_2NH (~ 50 °C, 0.25 h). The mixture was allowed to cool to the ambient temperature, and decyne-1 (0,4 mL) was added. Then, $SnCl_4$ (0.1 mL) was added dropwise under vigorous stirring. The mixture was stirred for 1 h, then the upper layer was separated and the lower layer was extracted with PhMe. The combined toluene extracts were passed through a column of silanized silica and evaporated *in vacuo*. The residue was recrystallized from heptane to give **1g** (yield 51%) as colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$): δ 2.18 – 2.24 (2H, m), 1.47 – 1.54 (2H, m), 1.25 – 1.40 (10H, m), 0.84 – 0.87 (3H, m).

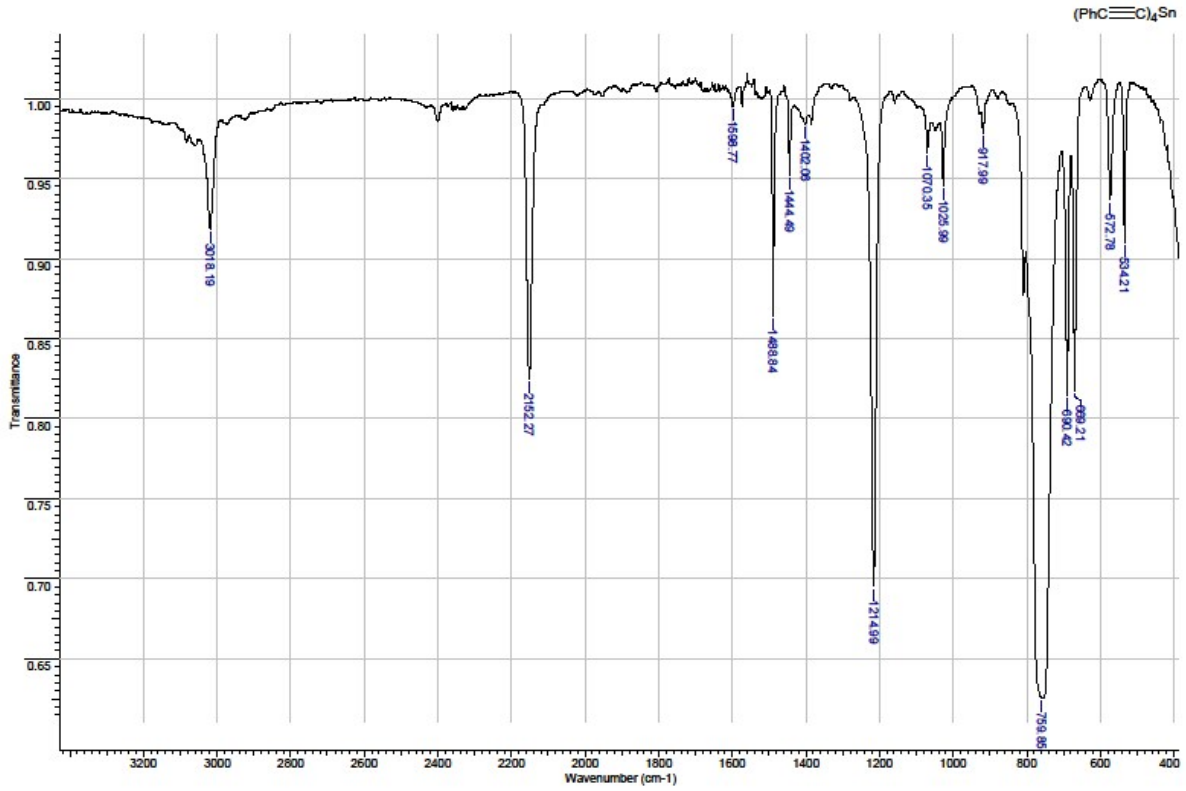
^{13}C NMR (100 MHz, $CDCl_3$): δ 111.71, 76.13, 31.90, 29.23, 29.14, 28.90, 28.41, 22.73, 20.14, 14.13.

^{119}Sn NMR (149 MHz, $CDCl_3$): δ -345.47 (m).

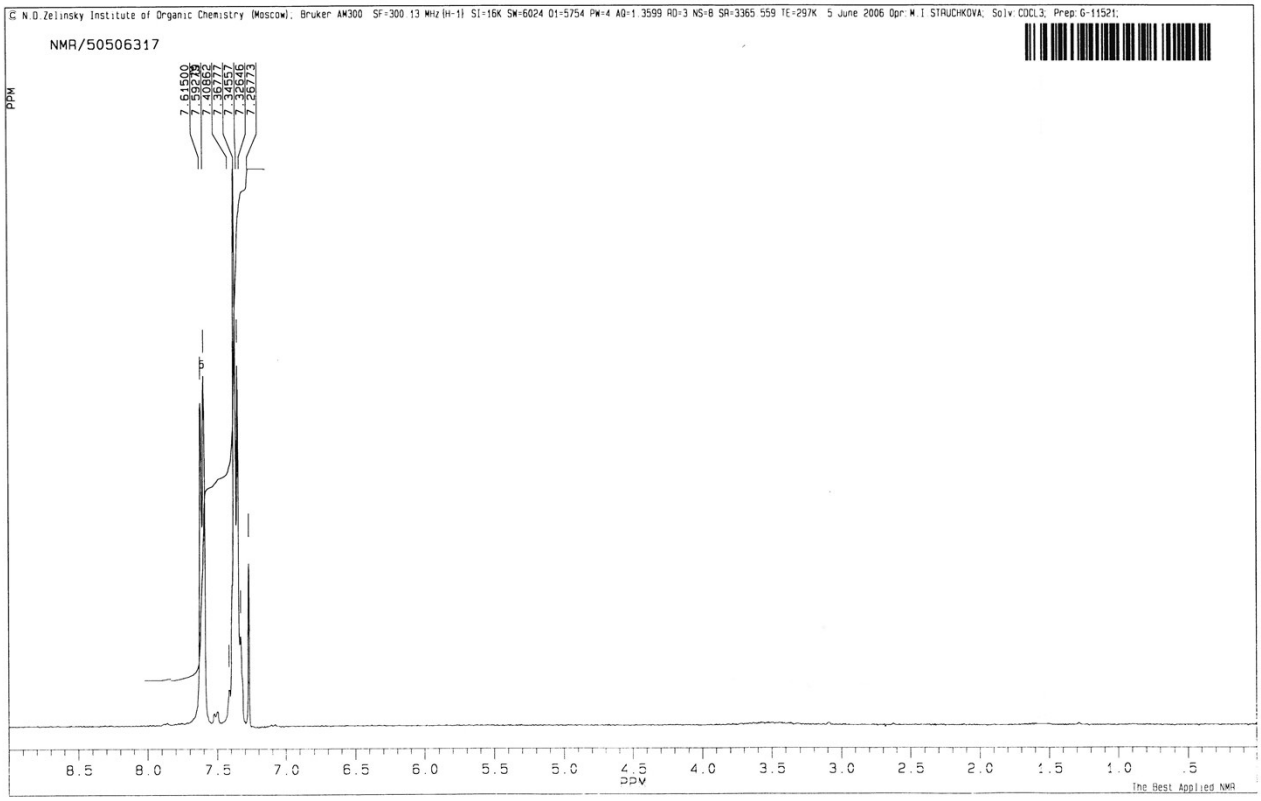
IR (CCl_4): 2164 (C≡C).

The selected spectra of tetraalkynylstannaes 1

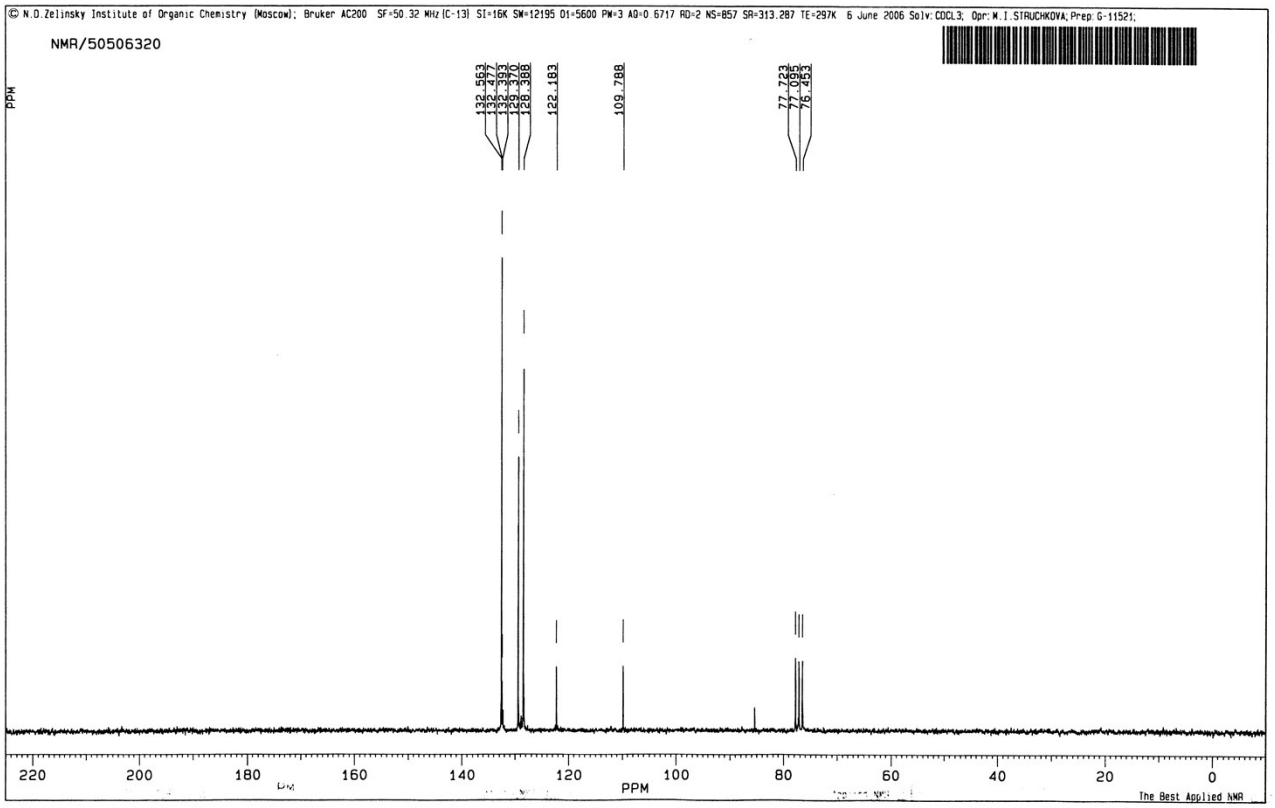
IR spectrum (KBr) of tetra(phenylethynyl)tin **1a**



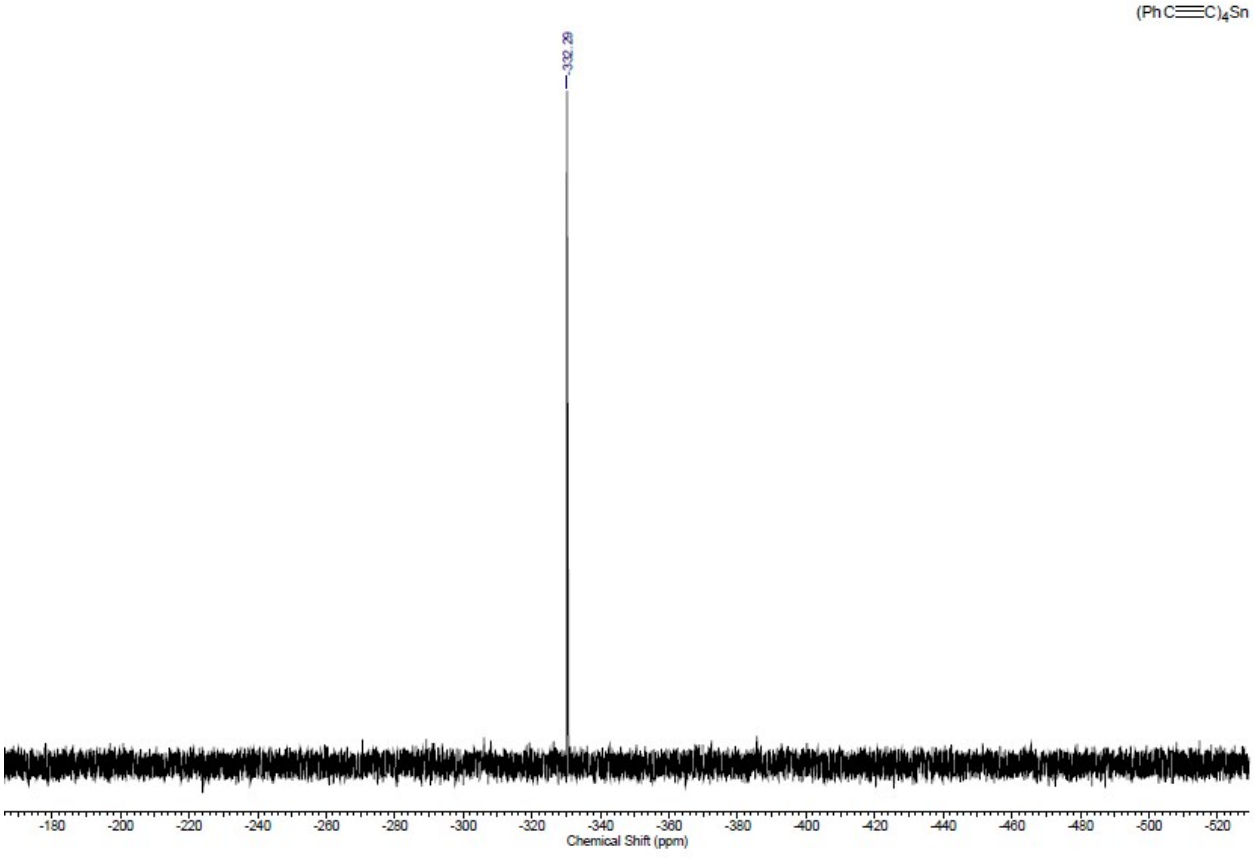
^{1}H NMR spectrum (300 MHz, $CDCl_3$) of tetra(phenylethynyl)tin **1a**



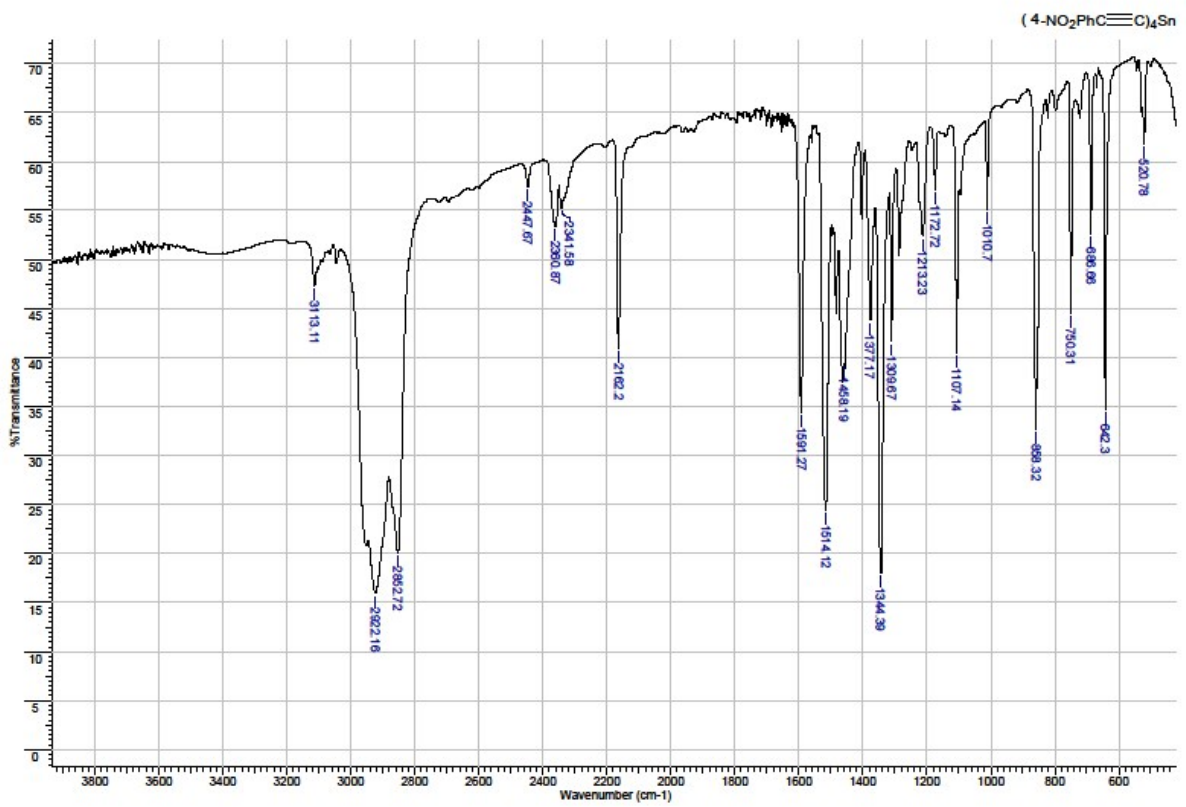
^{13}C NMR spectrum (50 MHz, $CDCl_3$) of tetra(phenylethynyl)tin **1a**



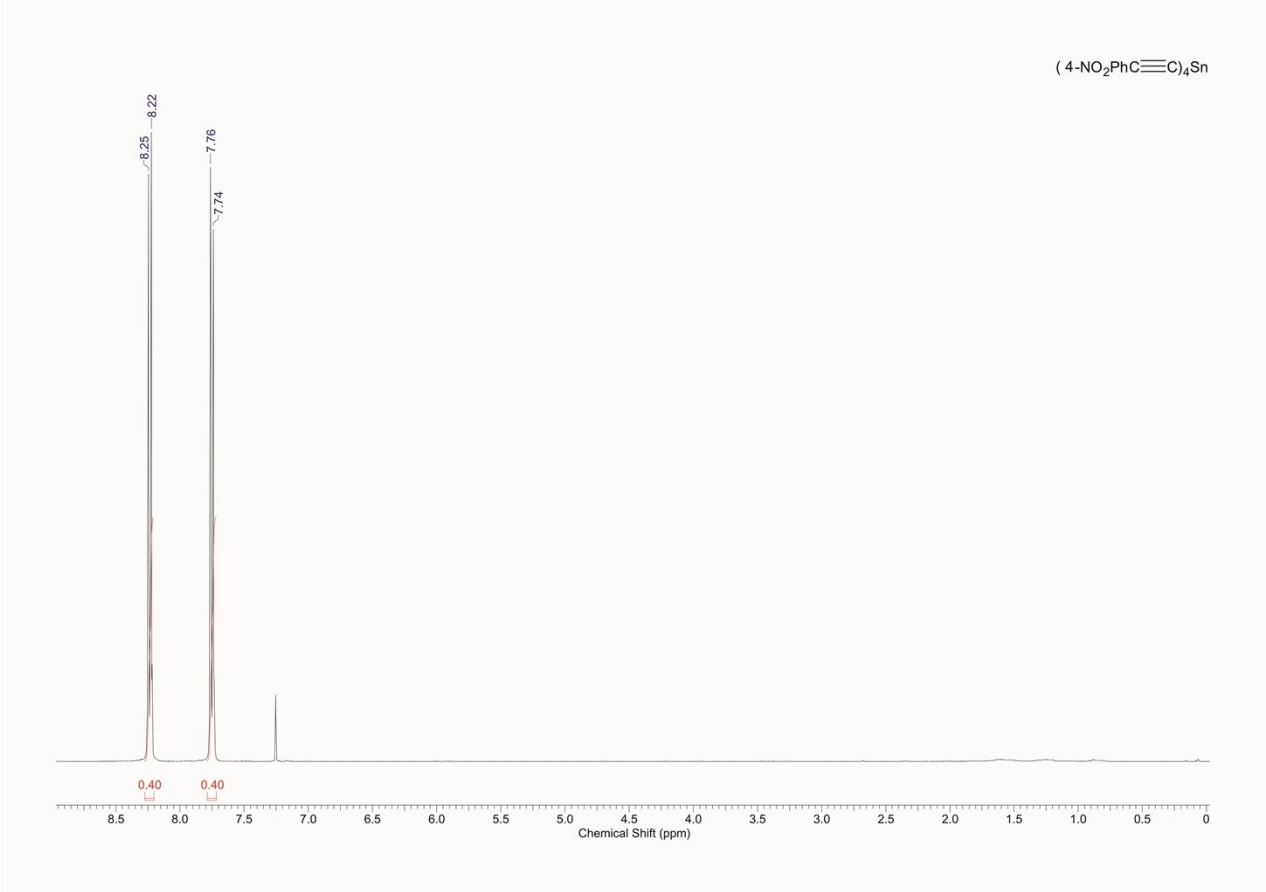
^{119}Sn NMR spectrum (149 MHz, $CDCl_3$) of tetra(phenylethynyl)tin **1a**



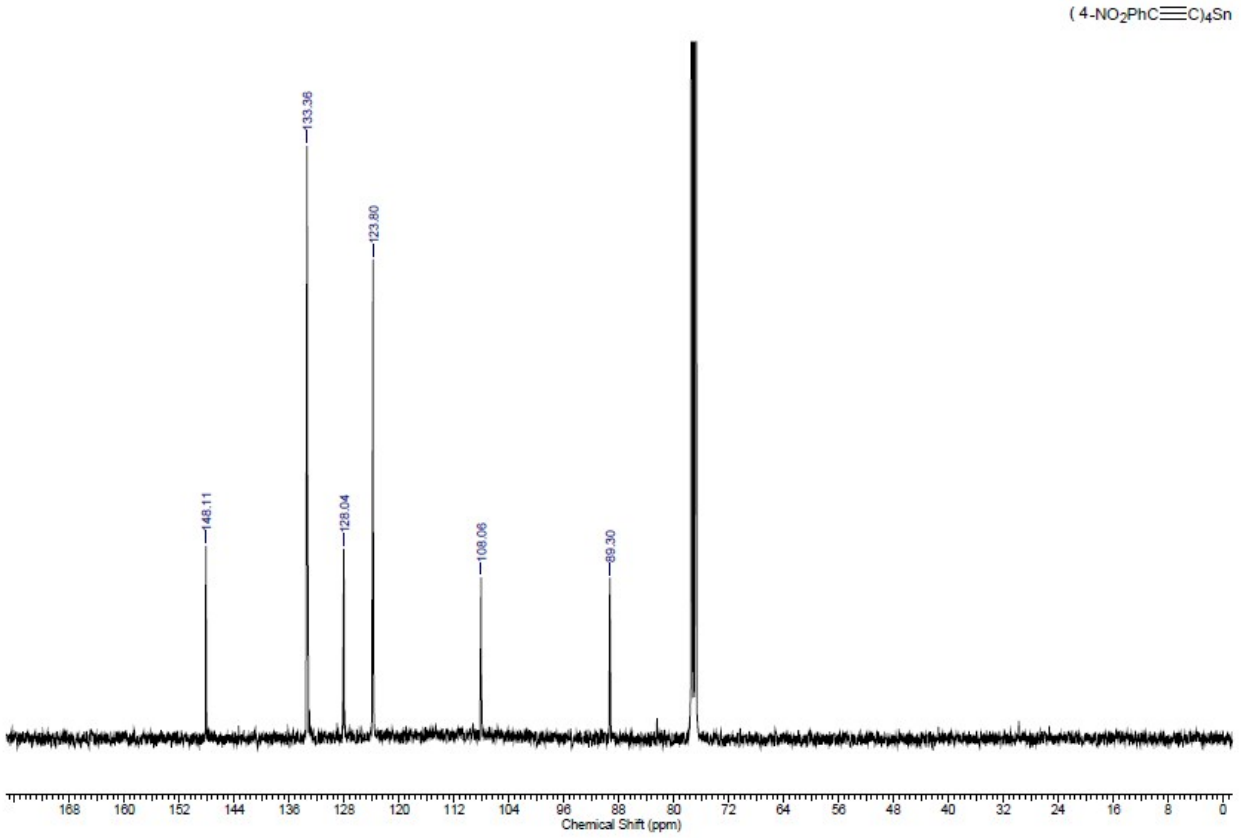
IR spectrum (KBr) of tetra(4-nitrophenylethynyl)tin **1d**



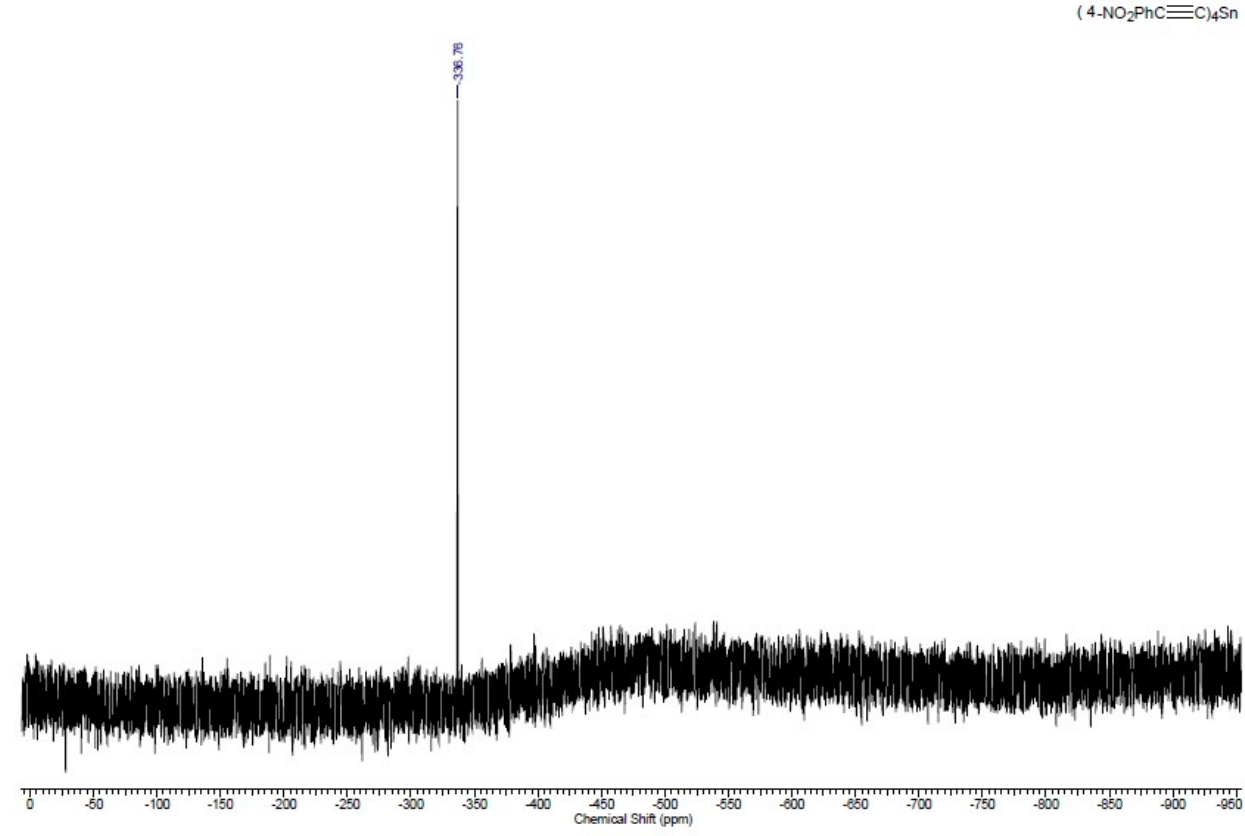
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of tetra(4-nitrophenylethynyl)tin **1d**



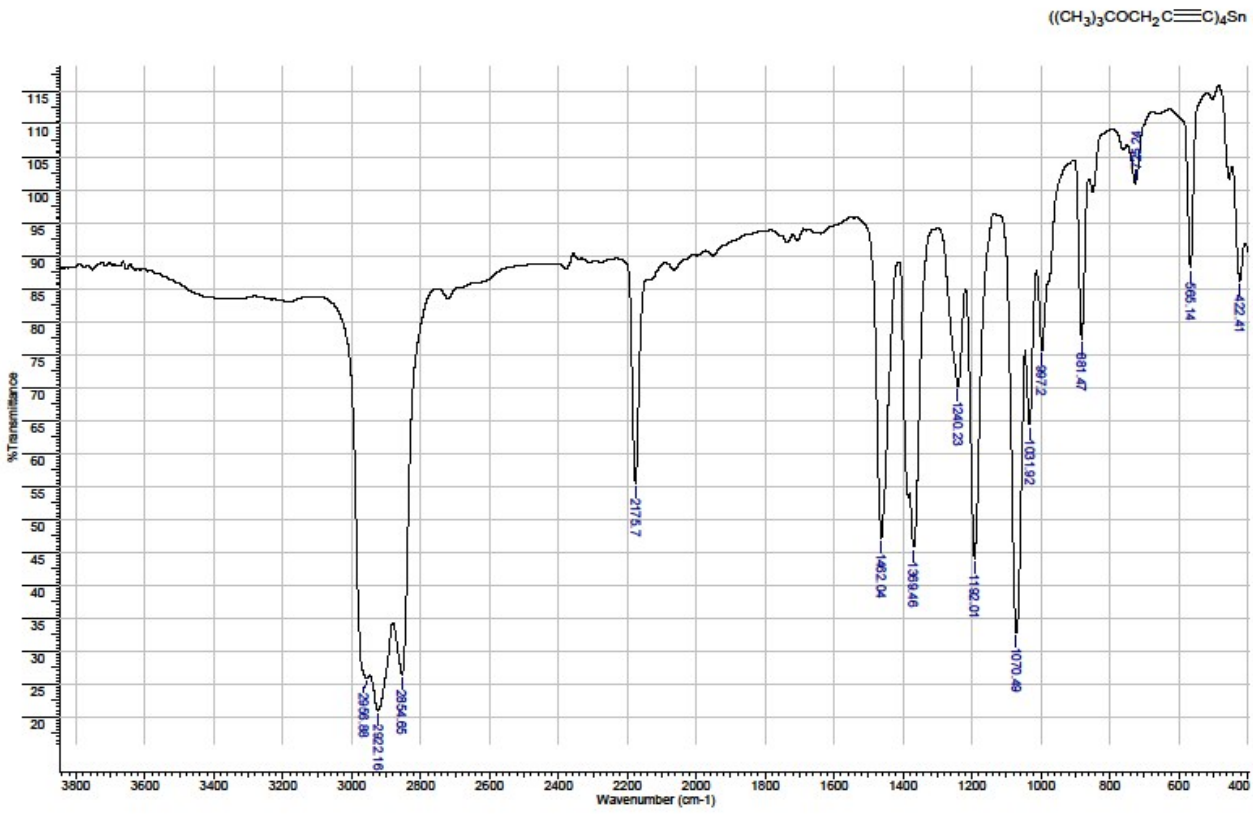
^{13}C NMR spectrum (100 MHz, $CDCl_3$) of tetra(4-nitrophenylethynyl)tin **1d**



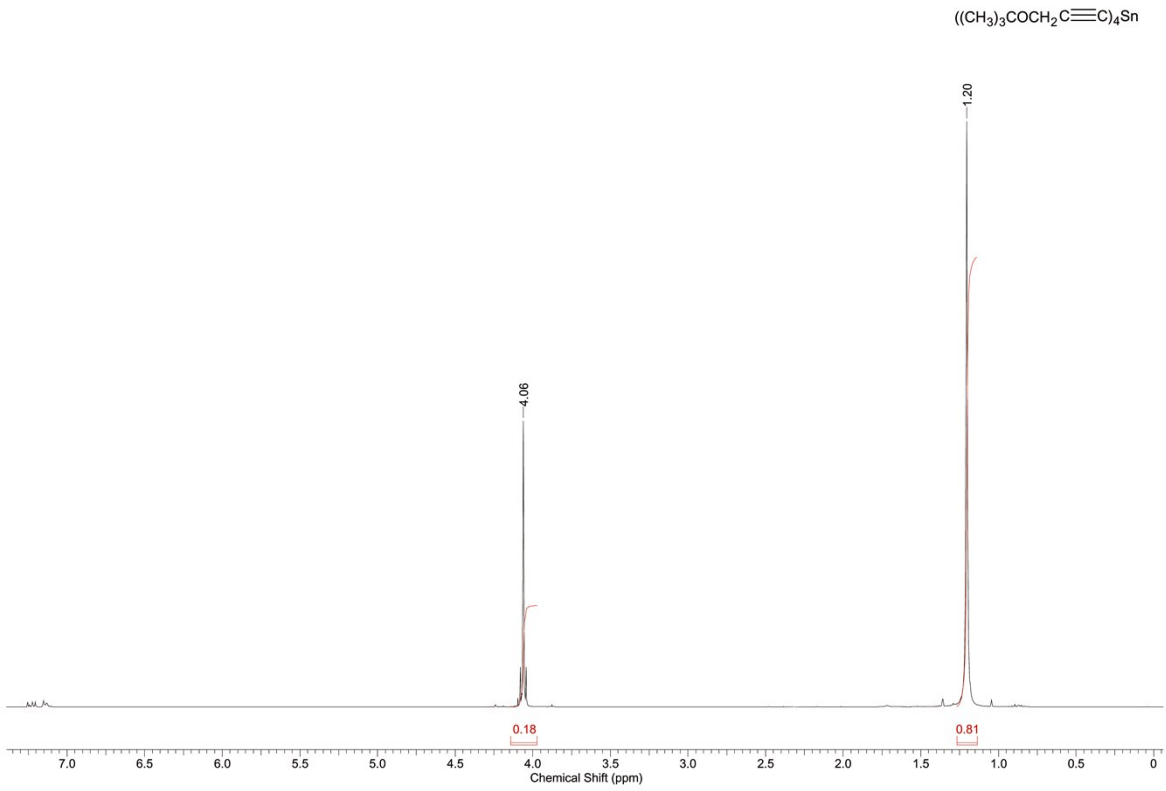
^{119}Sn NMR spectrum (149 MHz, $CDCl_3$) of tetra(4-nitrophenylethynyl)tin **1d**



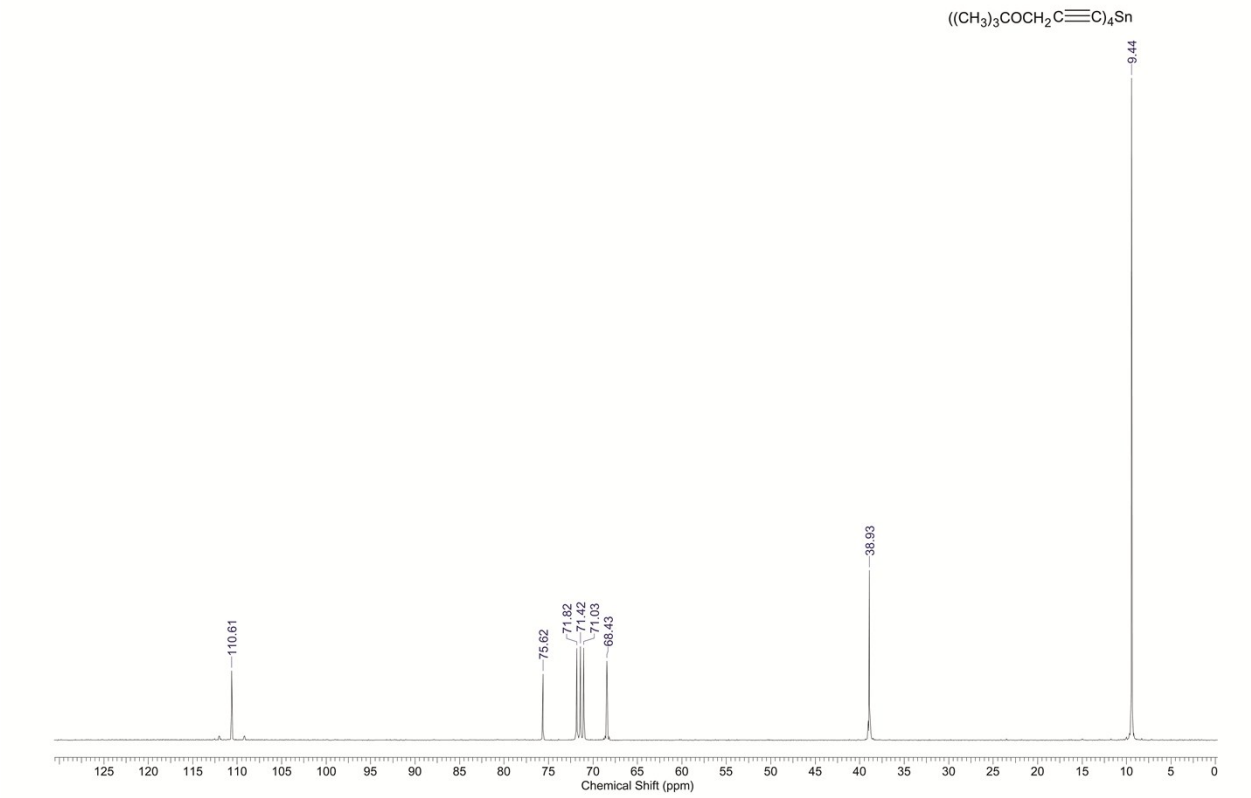
IR spectrum (KBr) of tetra(*tert*-buthoxypropynyl)tin **1e**



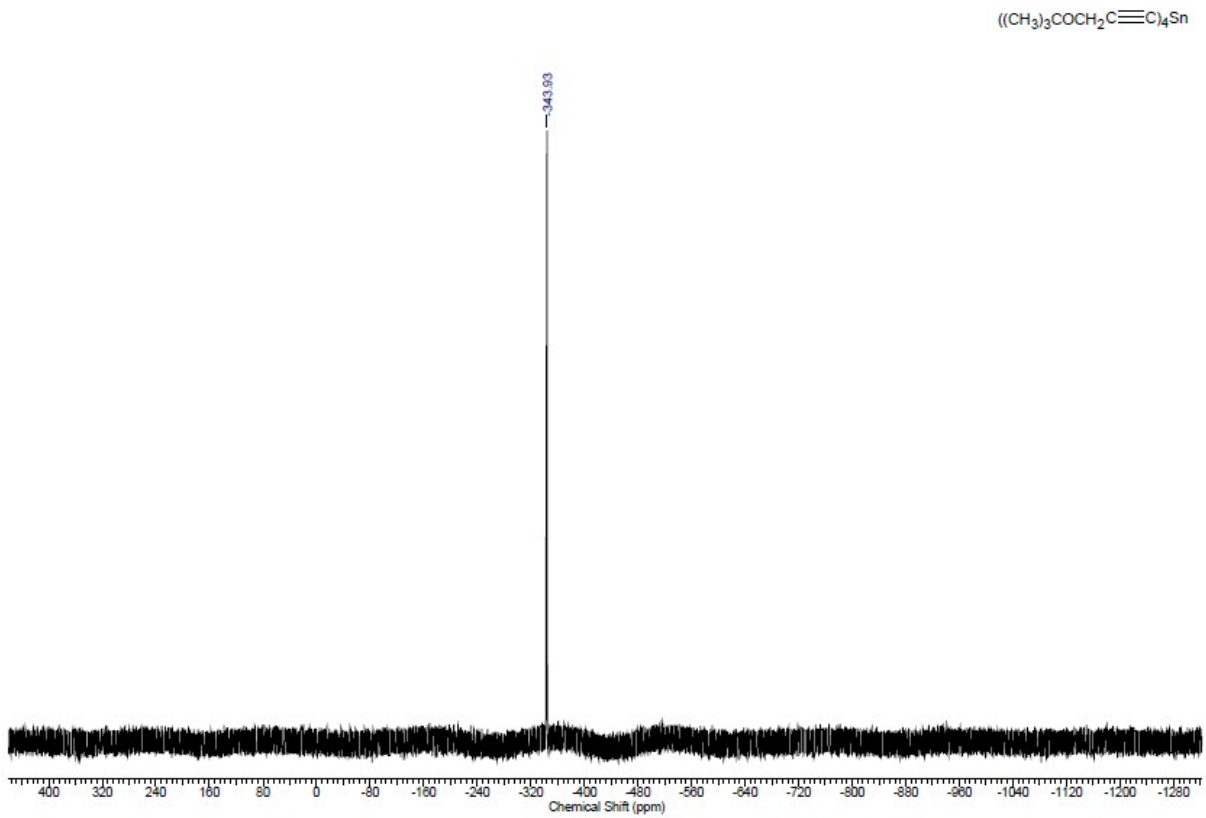
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of tetra(*tert*-buthoxypropynyl)tin **1e**



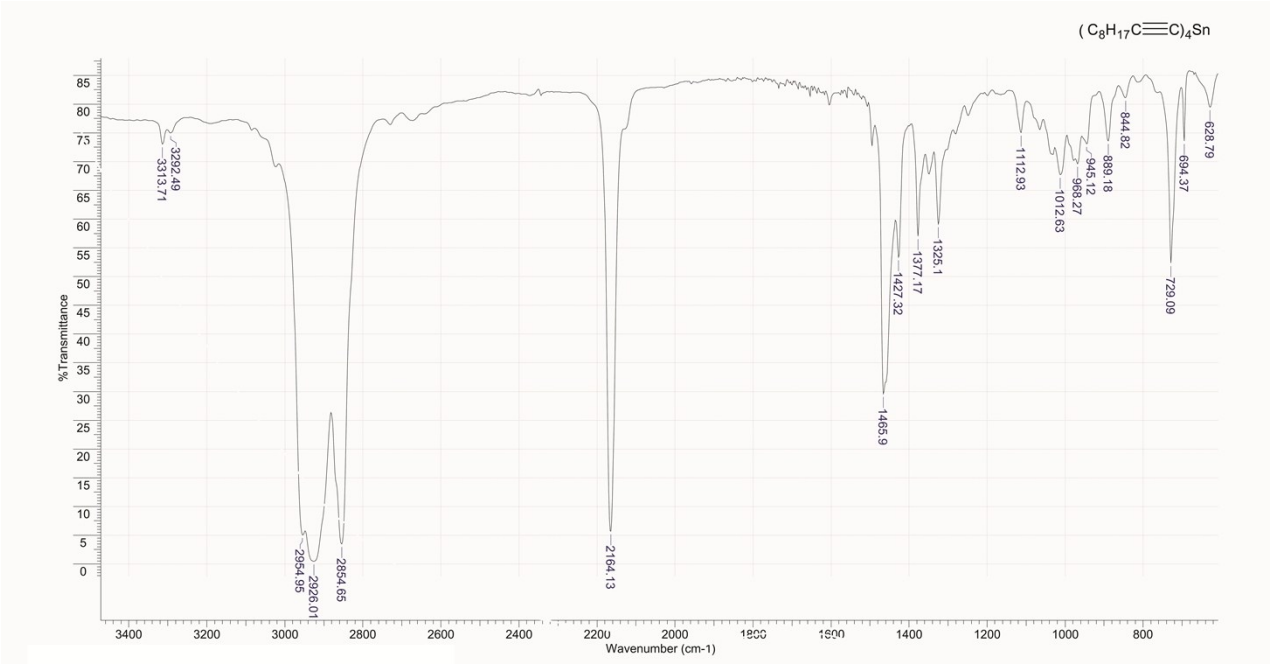
^{13}C NMR spectrum (100 MHz, $CDCl_3$) of tetra(*tert*-buthoxypropynyl)tin **1e**



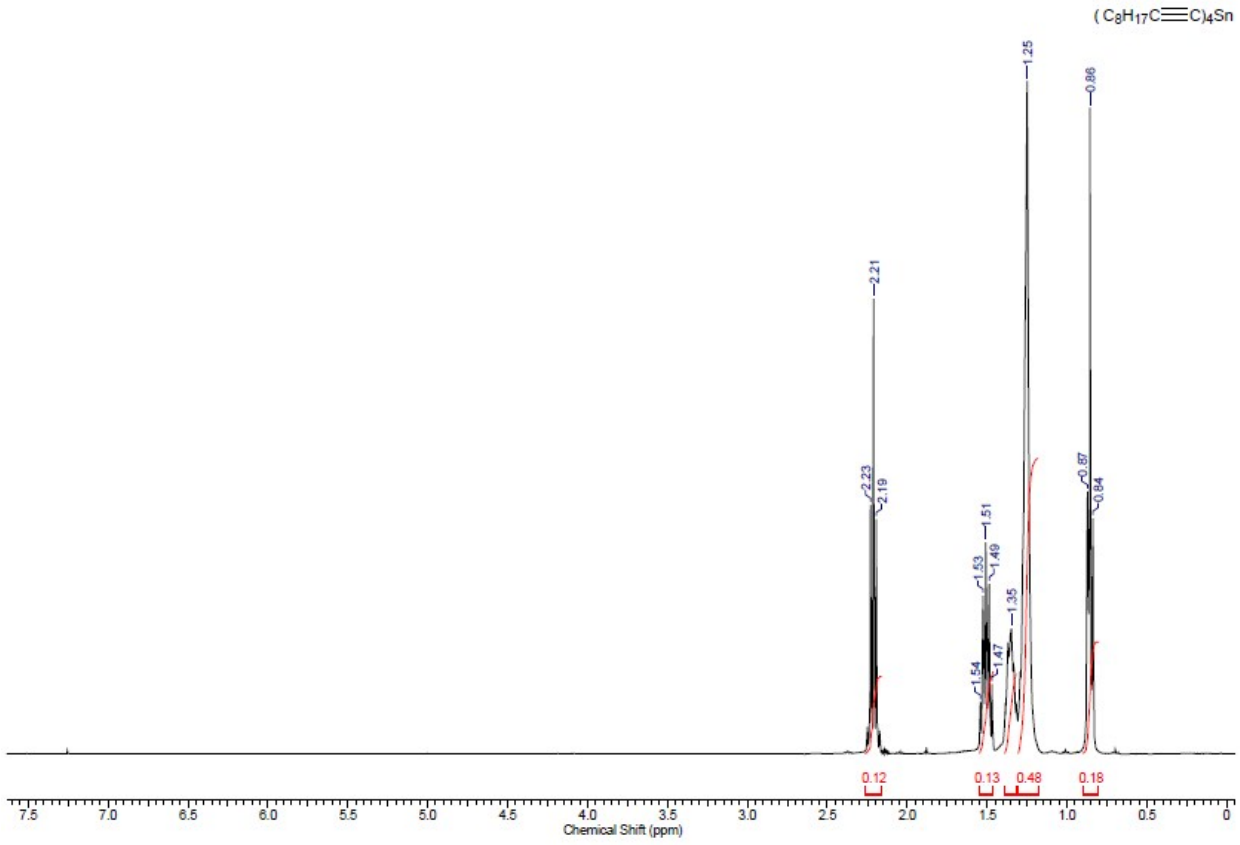
^{119}Sn NMR spectrum (149 MHz, $CDCl_3$) of tetra(*tert*-buthoxypropynyl)tin **1e**



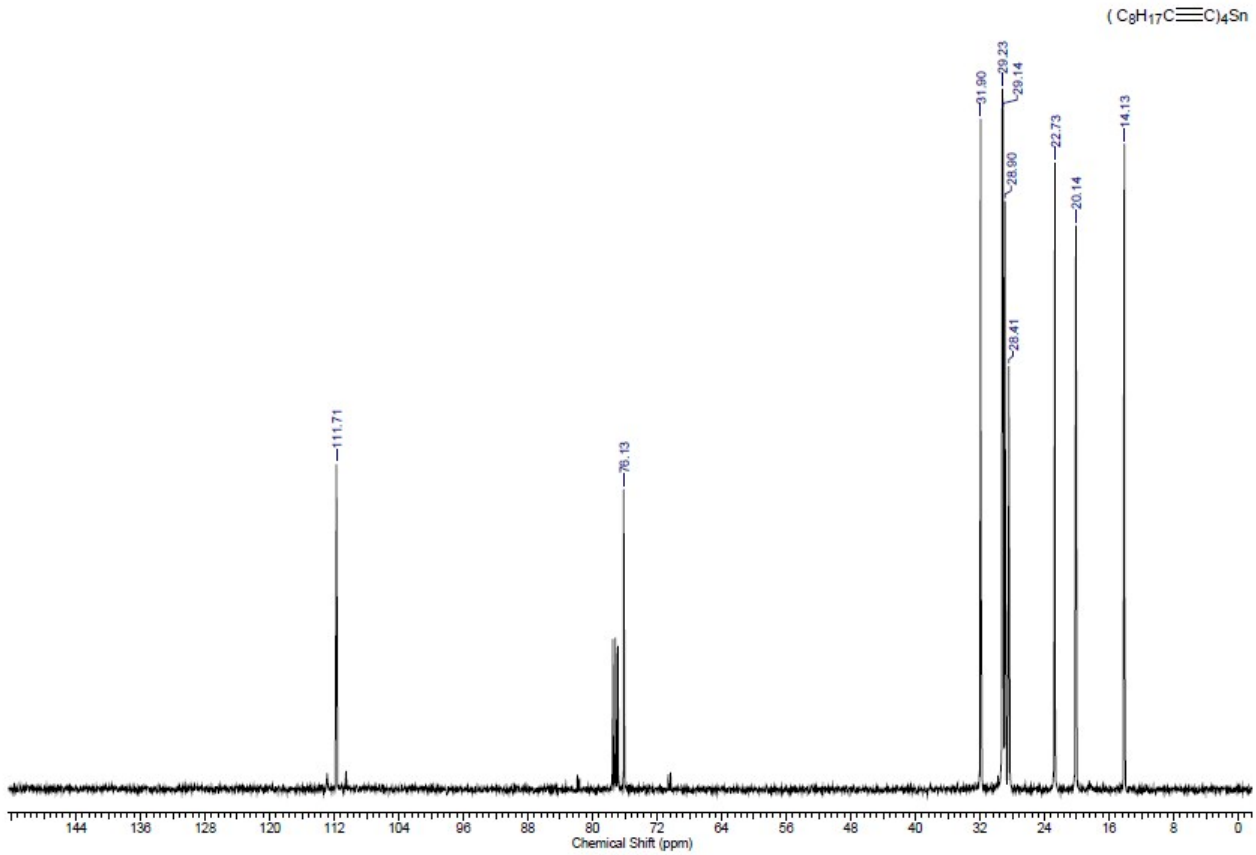
IR spectrum (KBr) of tetra(decyn-1-yl)tin **1g**



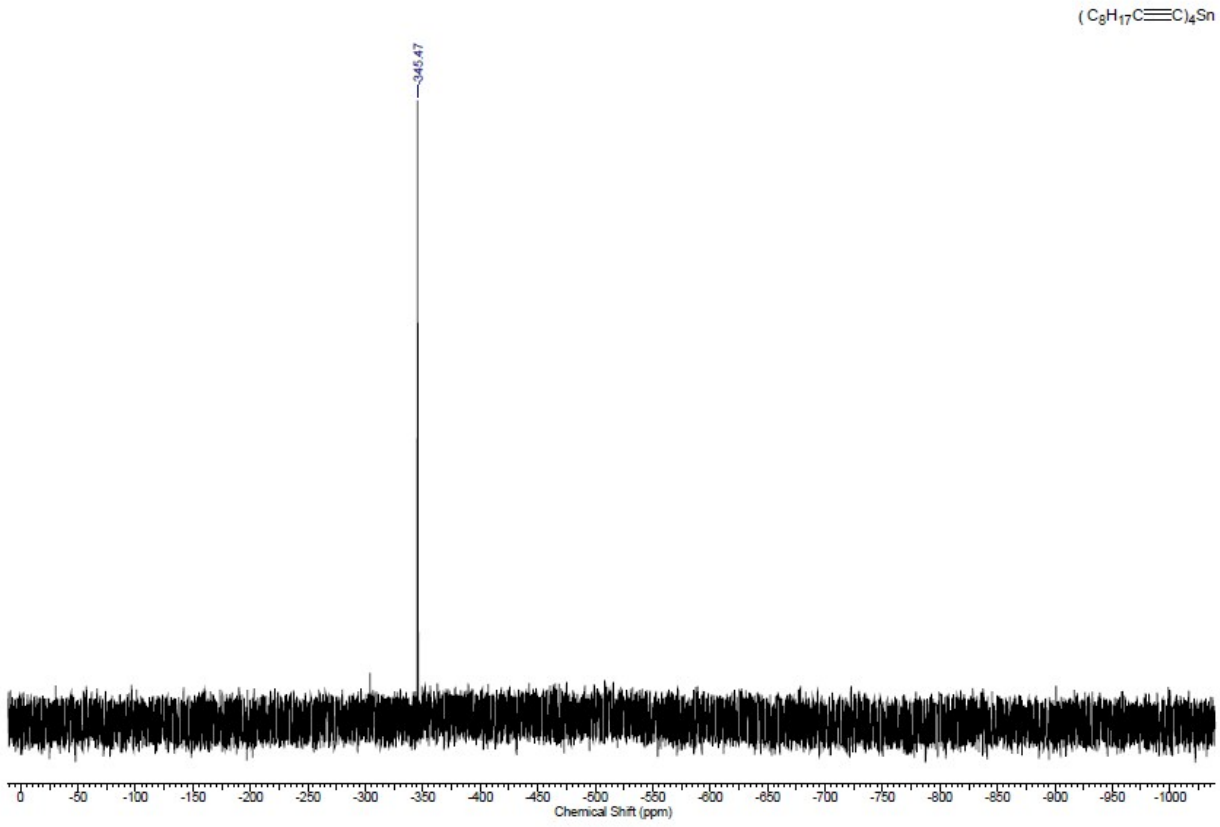
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of tetra(decyn-1-yl)tin **1g**



^{13}C NMR spectrum (100 MHz, $CDCl_3$) of tetra(decyn-1-yl)tin **1g**

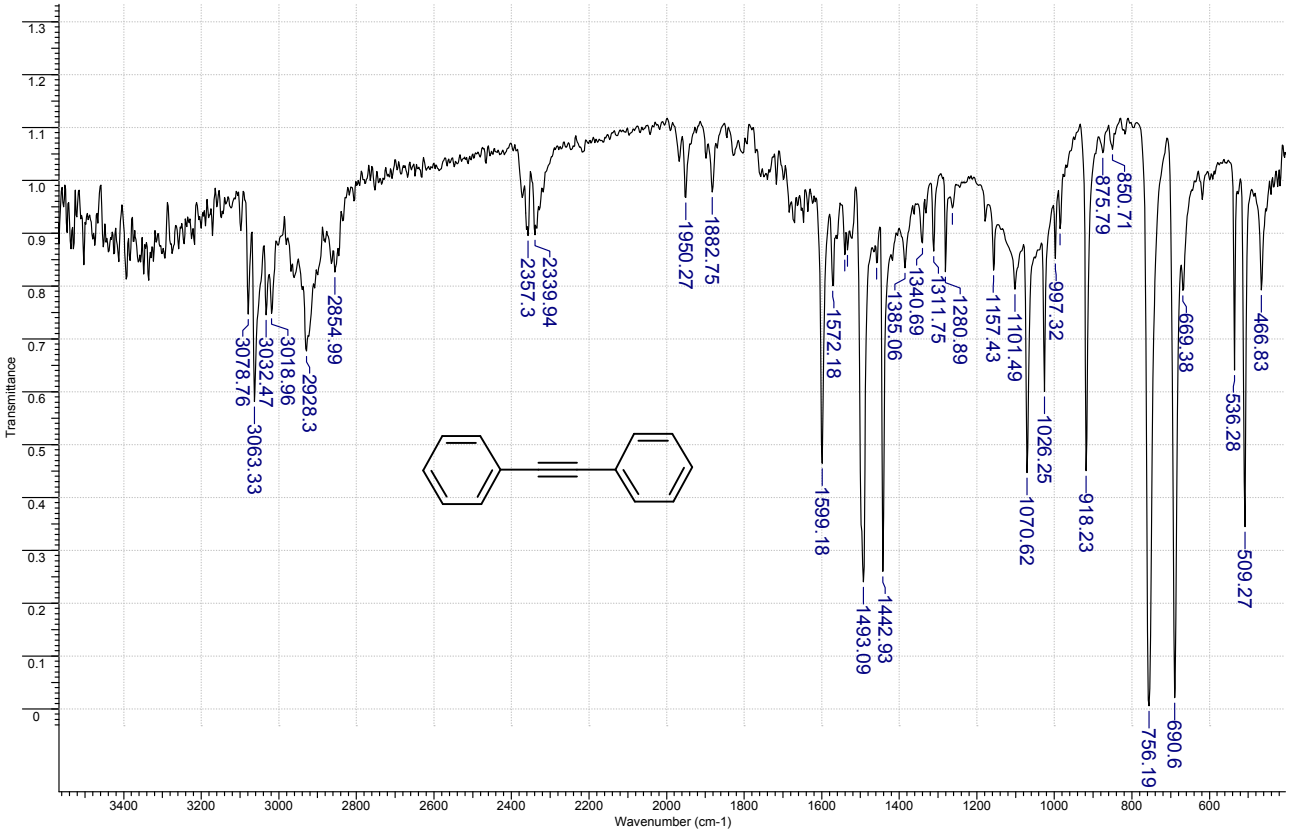


^{119}Sn NMR spectrum (149 MHz, $CDCl_3$) of tetra(decyn-1-yl)tin **1g**

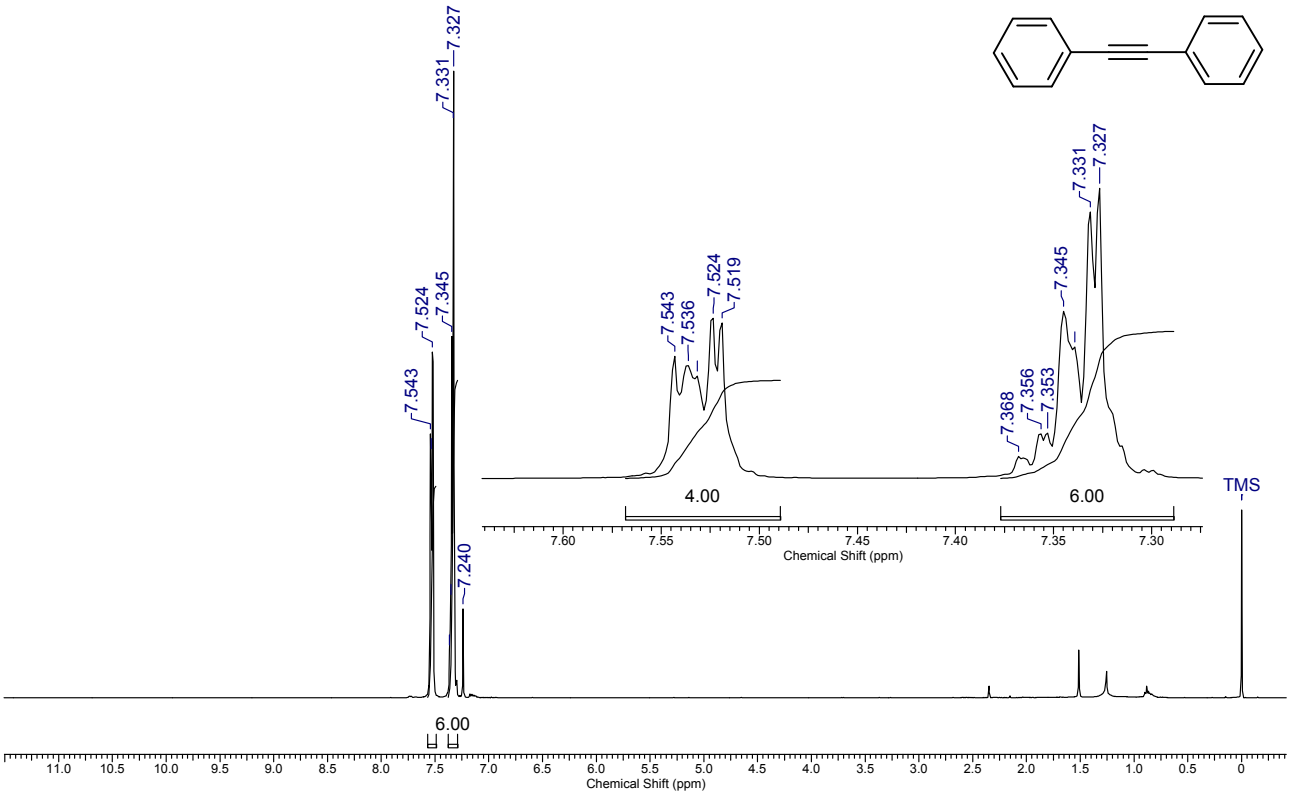


Spectra of acetylenes 4

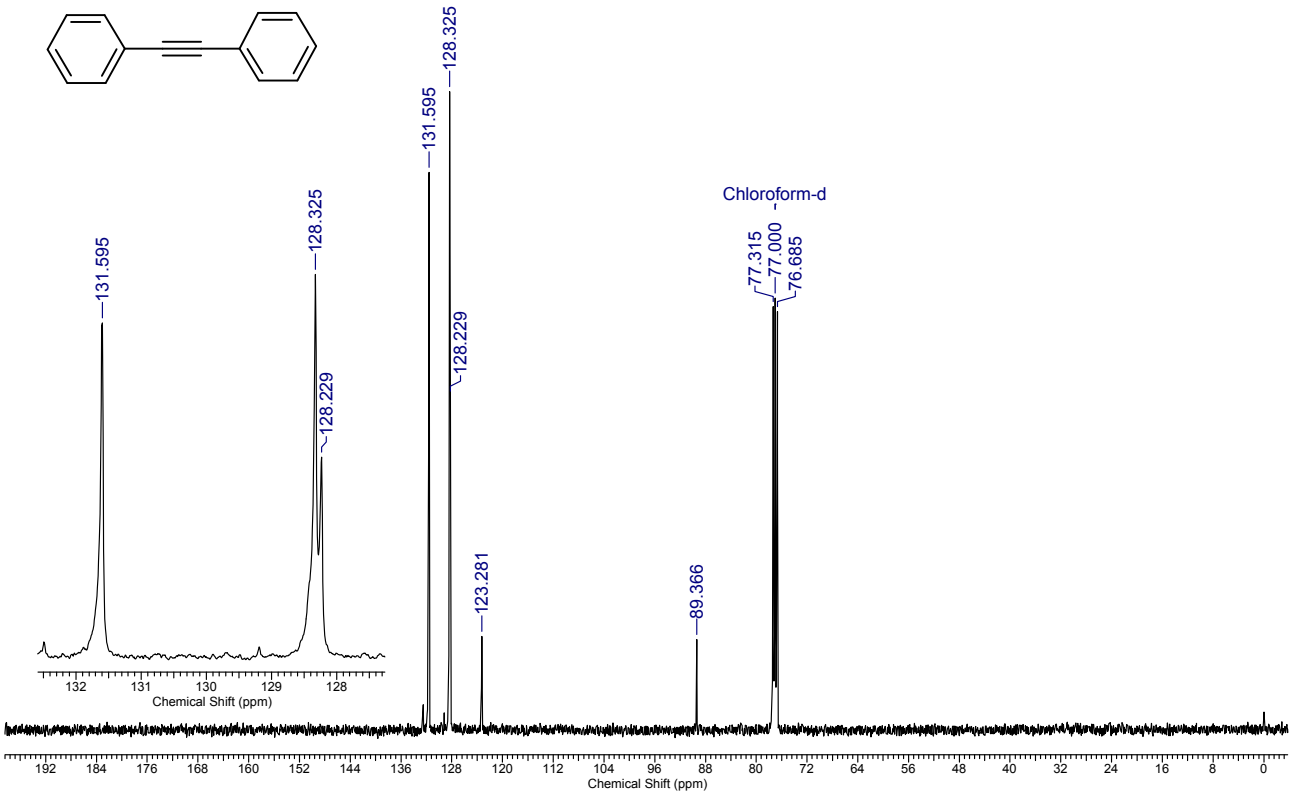
IR spectrum (KBr) of tolane (diphenyl acetylene) (**4aa**)



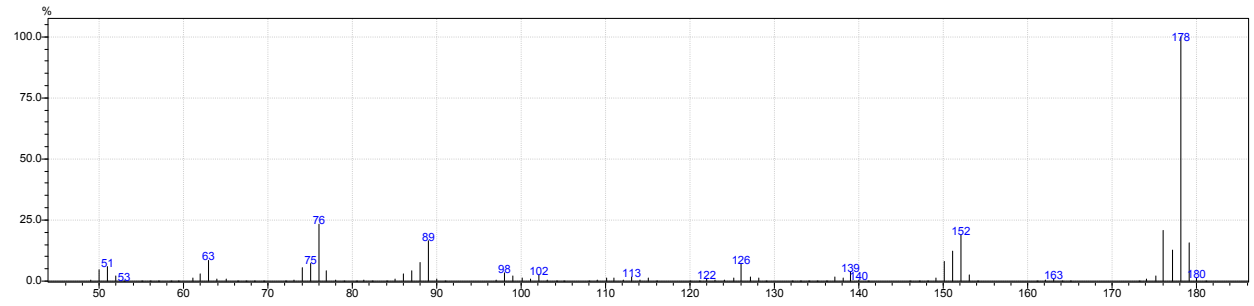
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of tolane (diphenyl acetylene) (**4aa**)



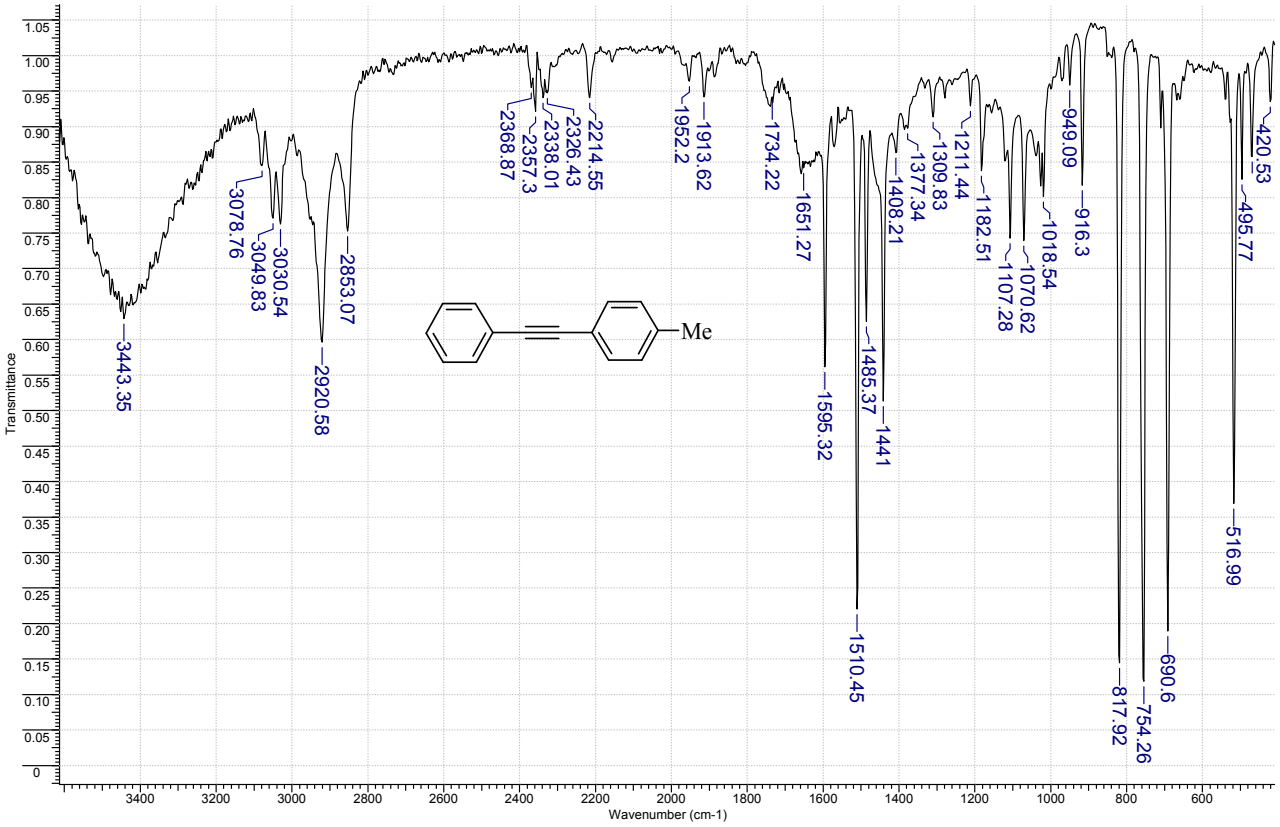
^{13}C NMR spectrum (100 MHz, $CDCl_3$) of tolane (diphenyl acetylene) (**4aa**)



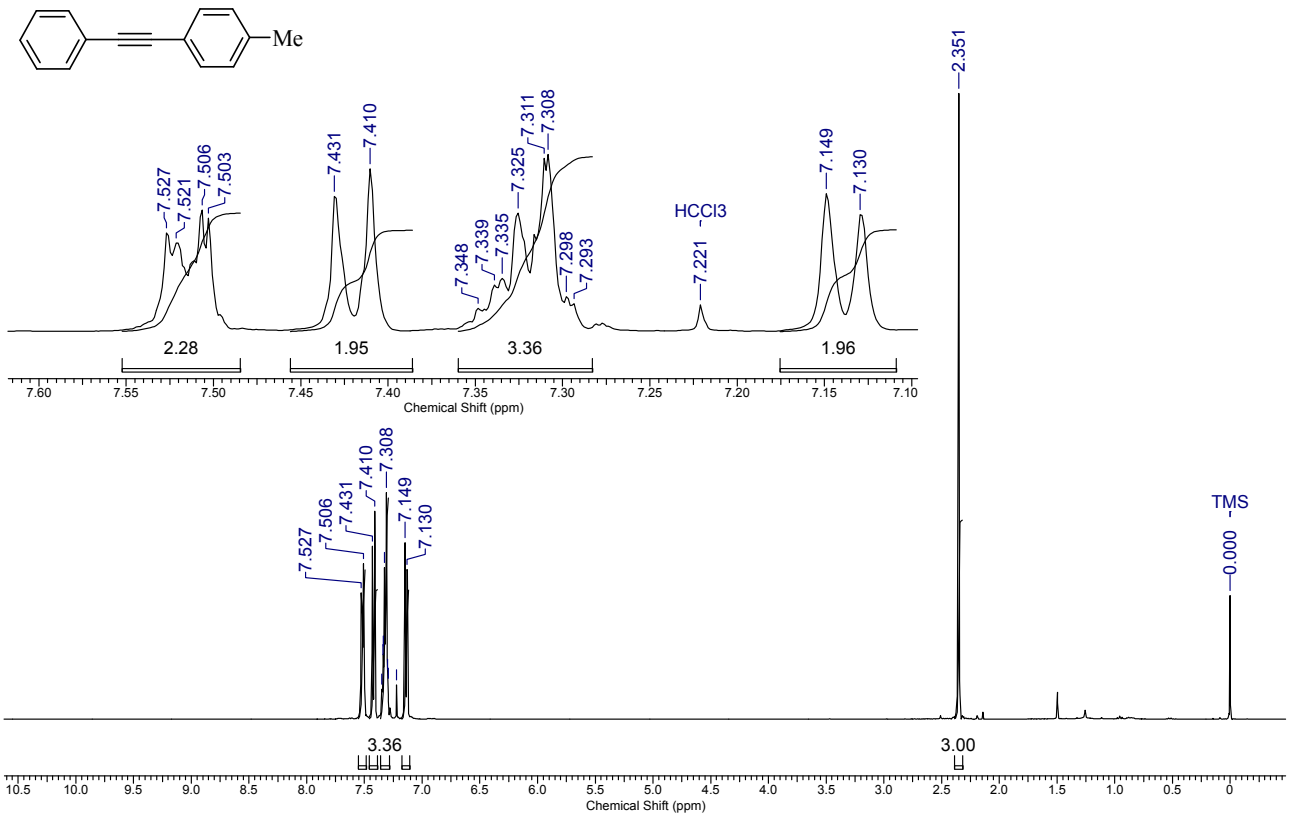
Mass-spectrum (EI, 70 eV) of tolane (diphenyl acetylene) (**4aa**)



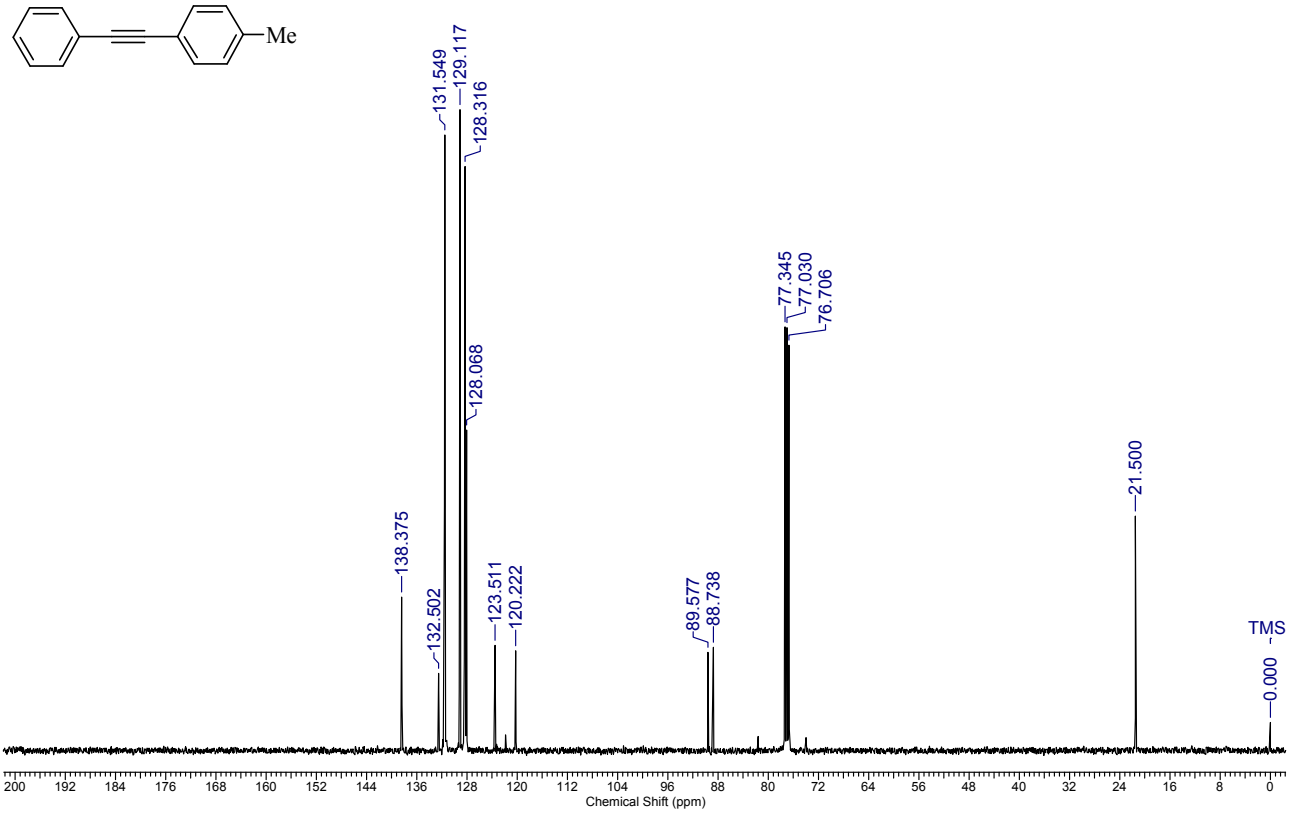
IR spectrum (KBr) of 4-methyltolane (1-methyl-4-(phenylethynyl)benzene) (**4ab**)



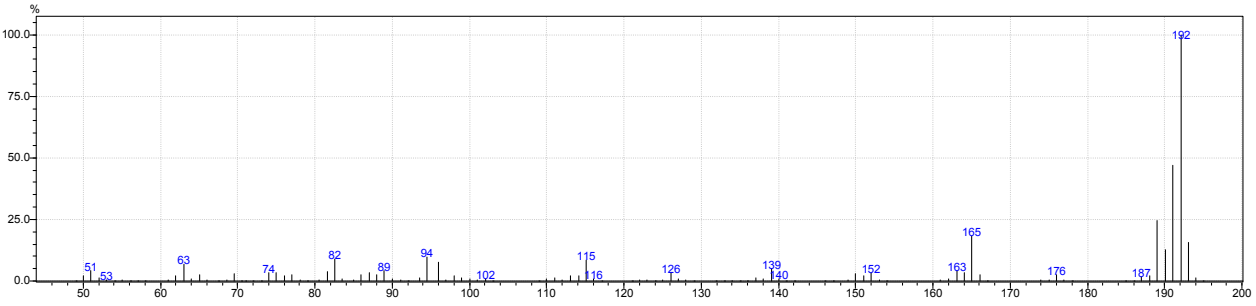
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 4-methyltolane (**4ab**)



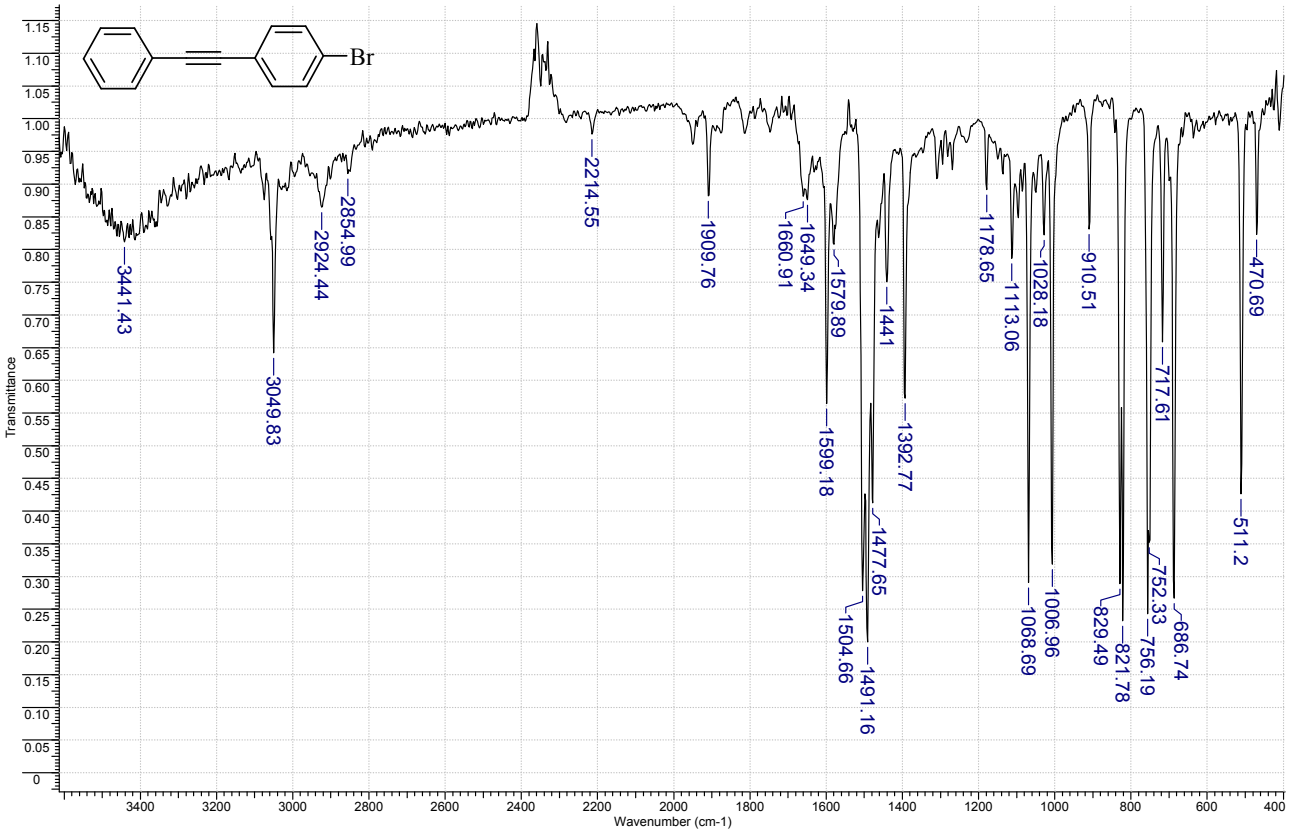
^{13}C NMR spectrum (100 MHz, $CDCl_3$) of 4-methyltolane (**4ab**)



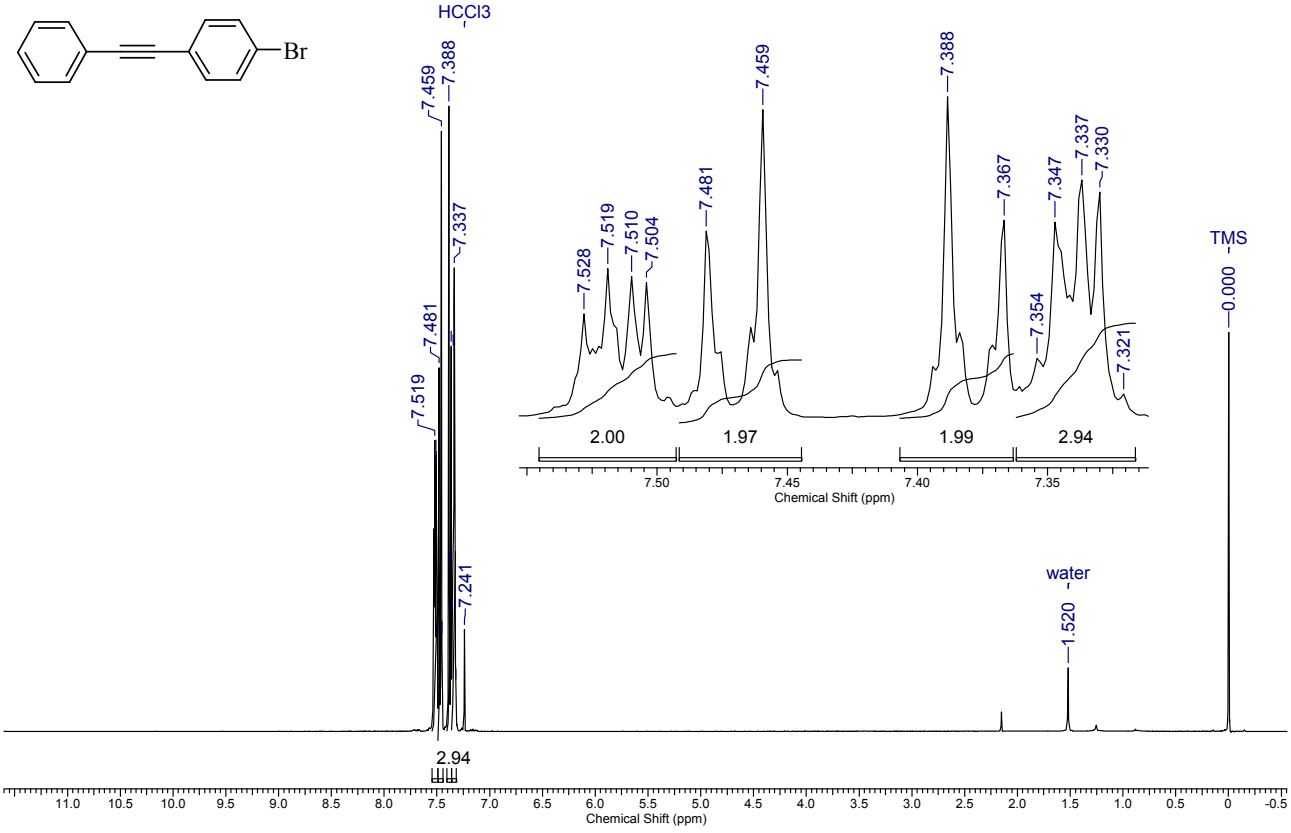
Mass-spectrum (EI, 70 eV) of 4-methyltolane (**4ab**)



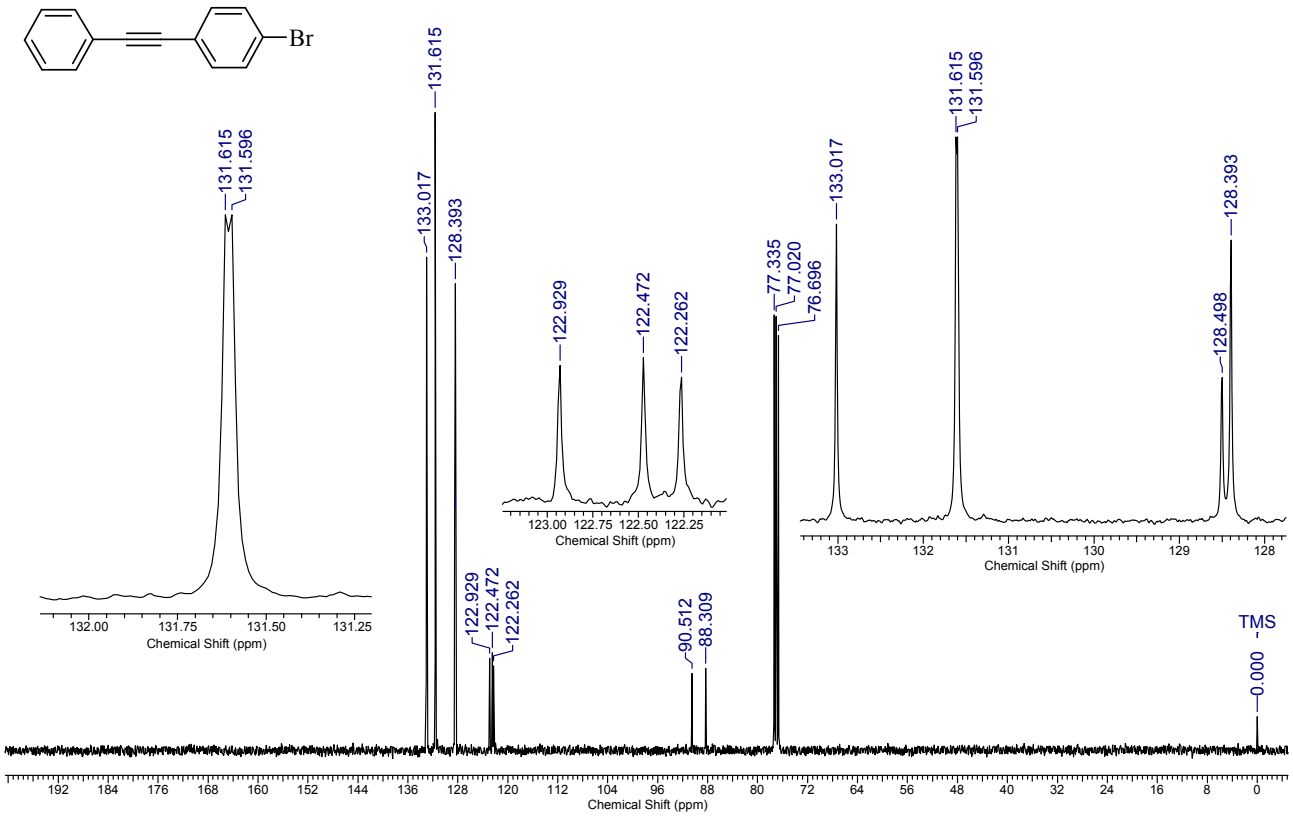
IR spectrum (KBr) of 4-bromotolane (**4ac**)



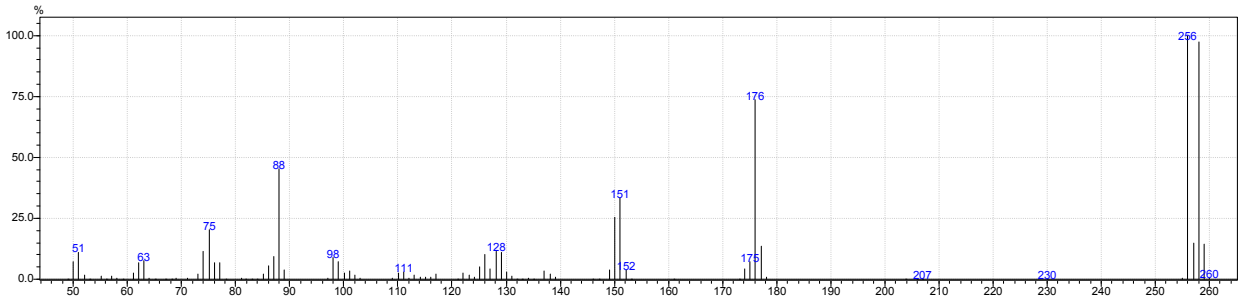
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 4-bromotolane (**4ac**)



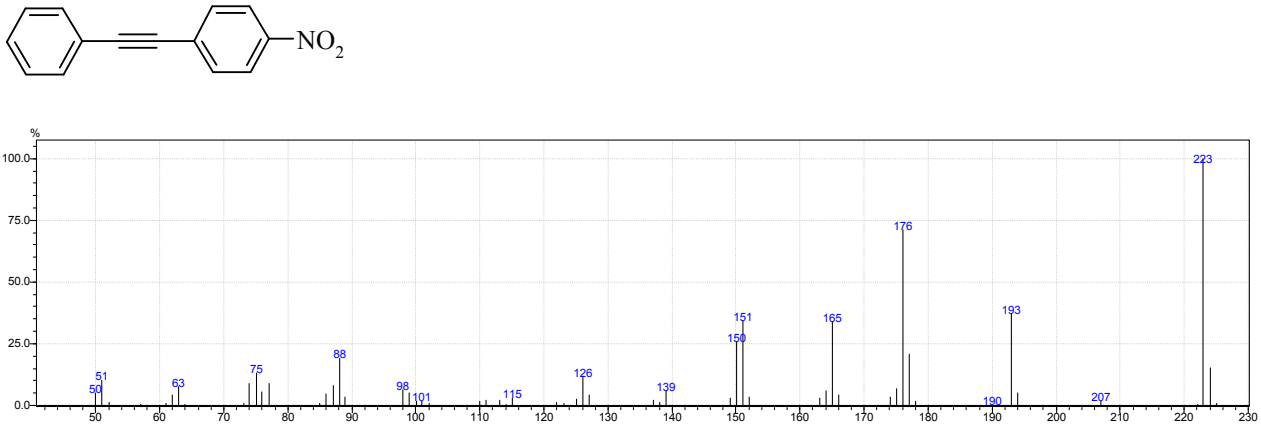
^{13}C NMR spectrum (100 MHz, $CDCl_3$) of 4-bromotolane (**4ac**)



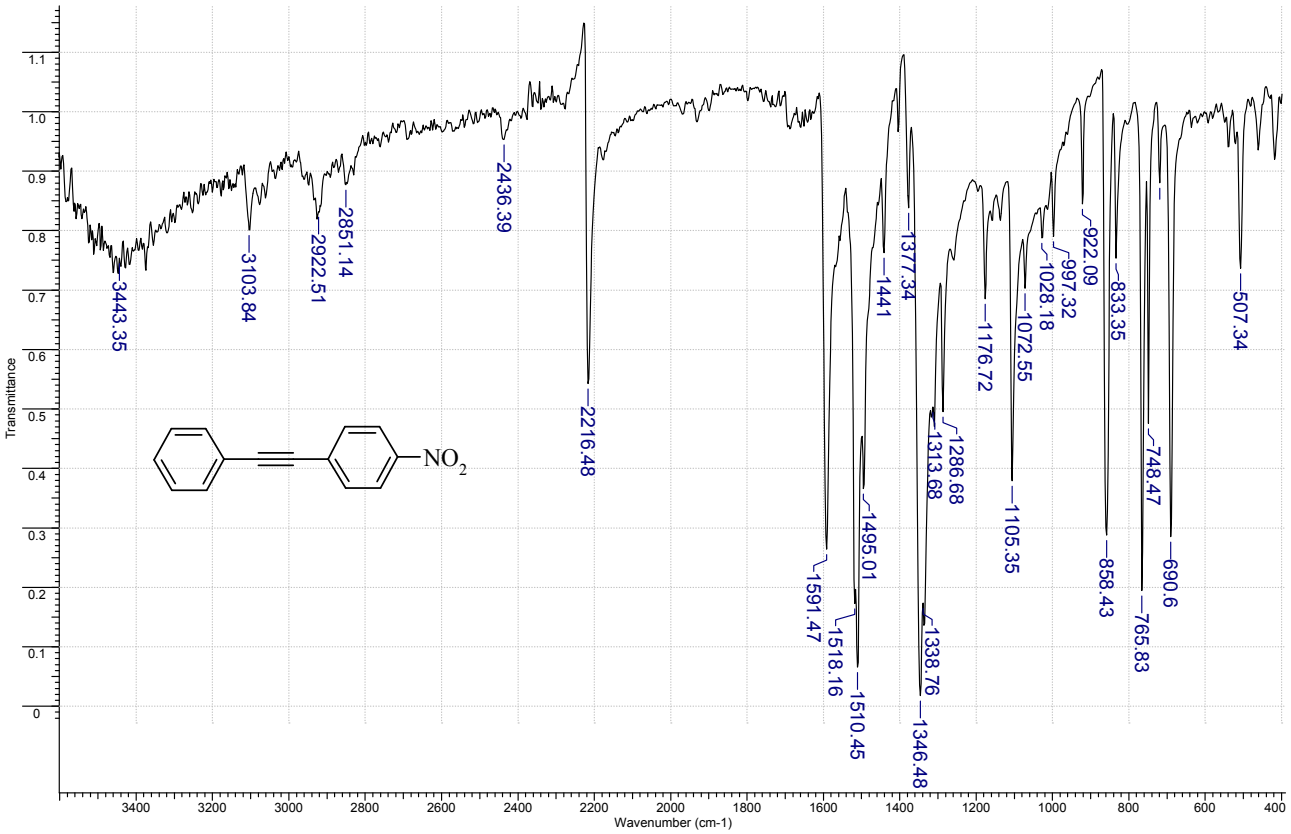
Mass-spectrum (EI, 70 eV) of 4-bromotolane (**4ac**)



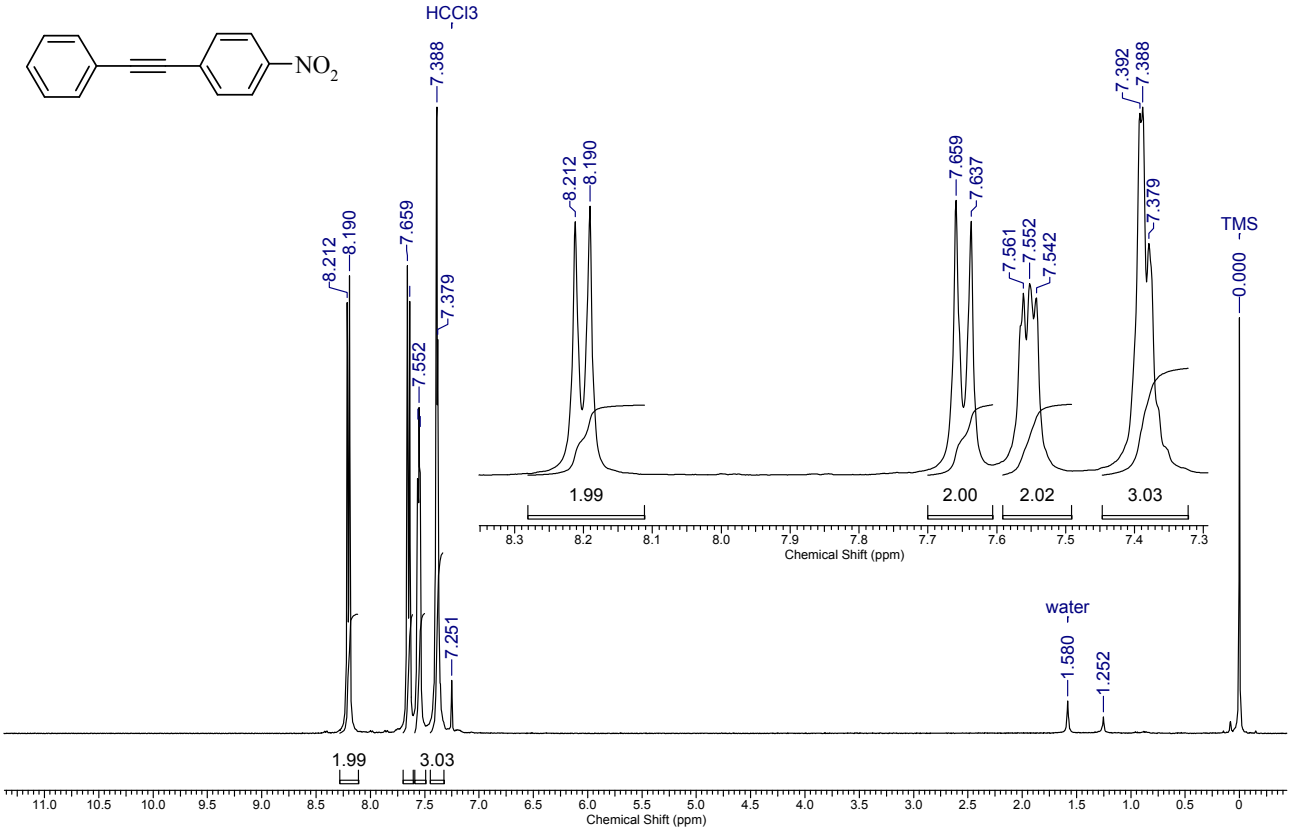
Mass-spectrum (EI, 70 eV) of 4-nitrotolane (**4ad**)



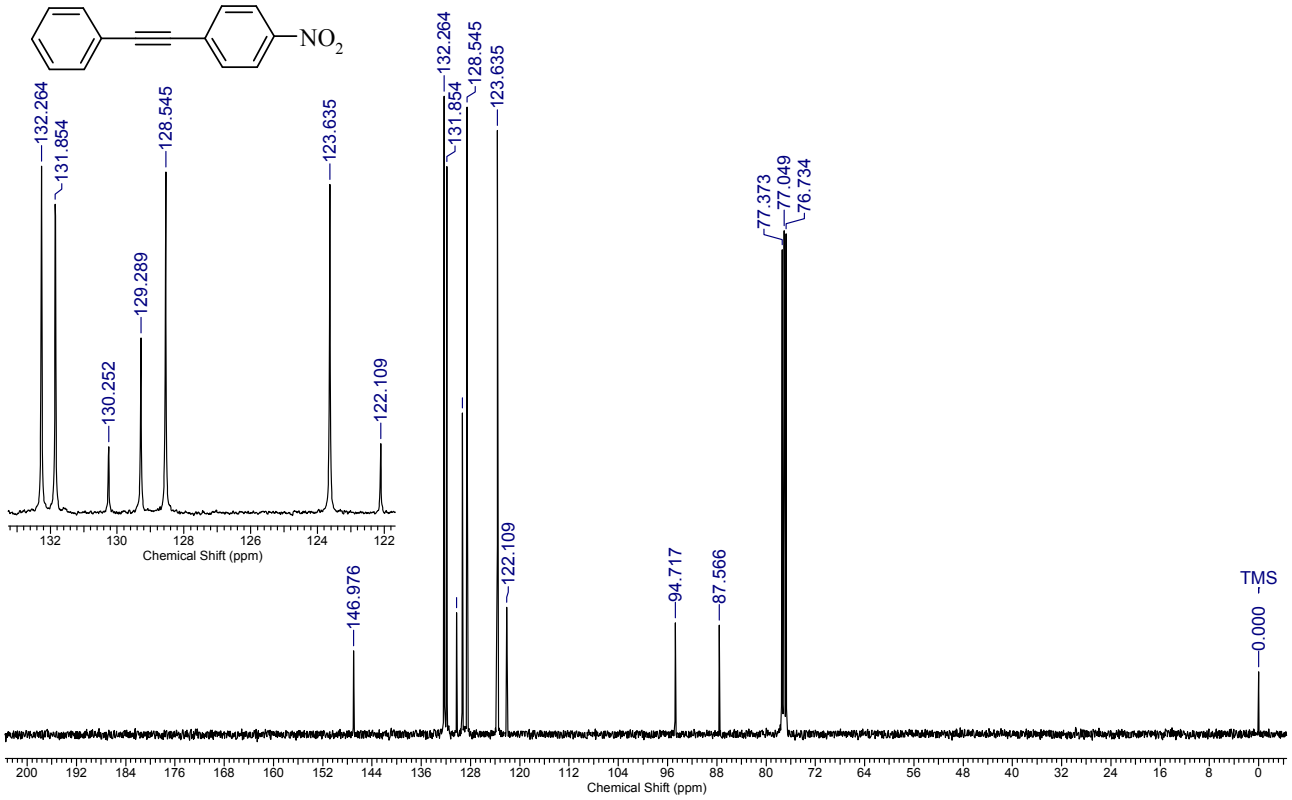
IR spectrum (KBr) of 4-nitrotolane (**4ad**)



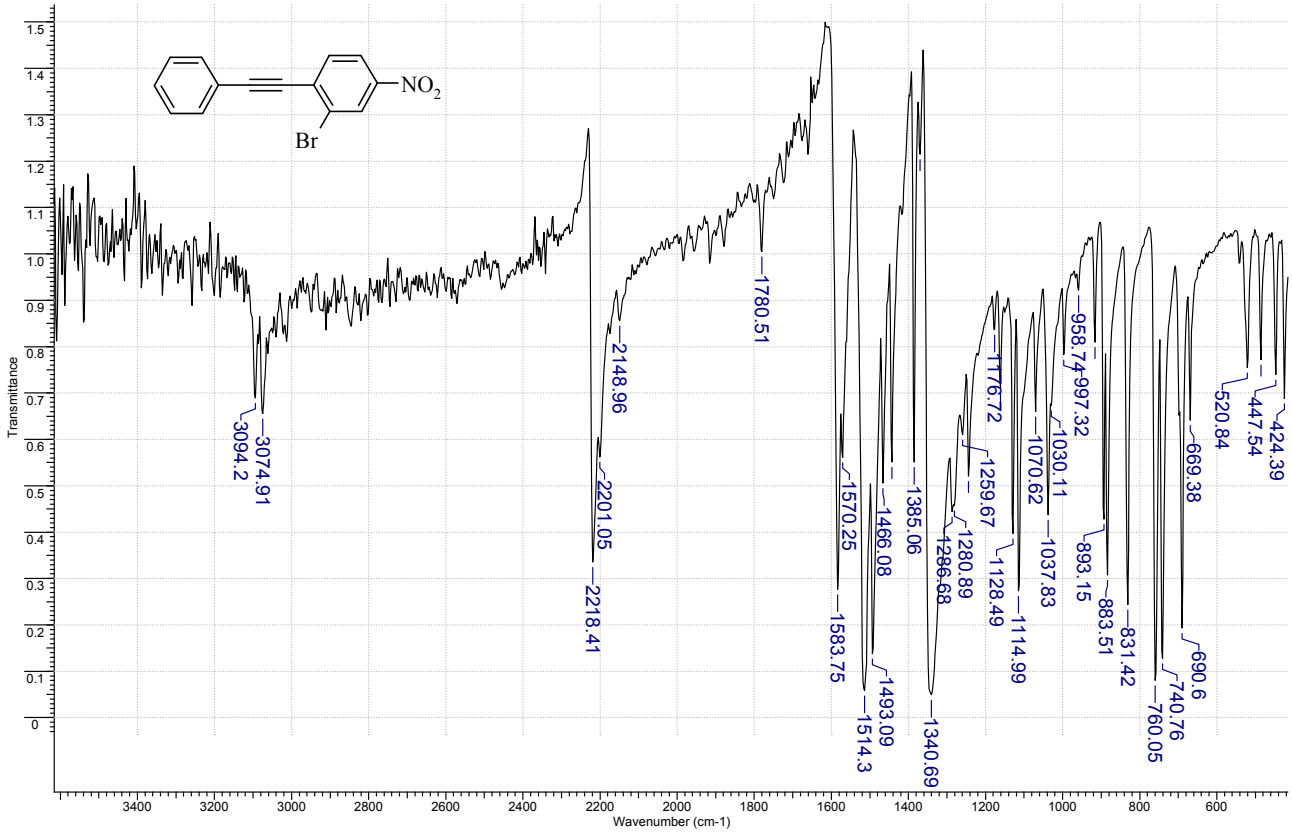
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 4-nitrotolane (**4ad**)



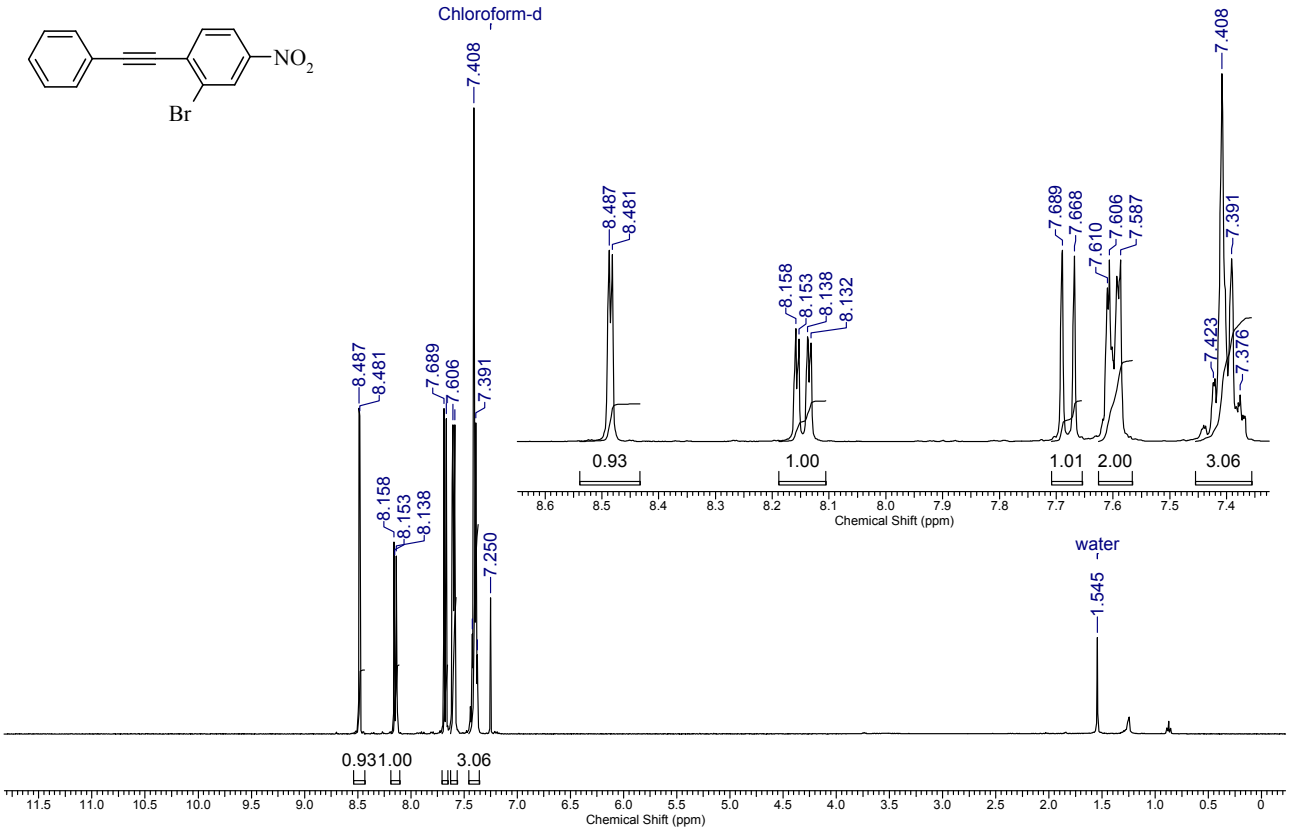
^{13}C NMR spectrum (100 MHz, $CDCl_3$) of 4-nitrotolane (**4ad**)



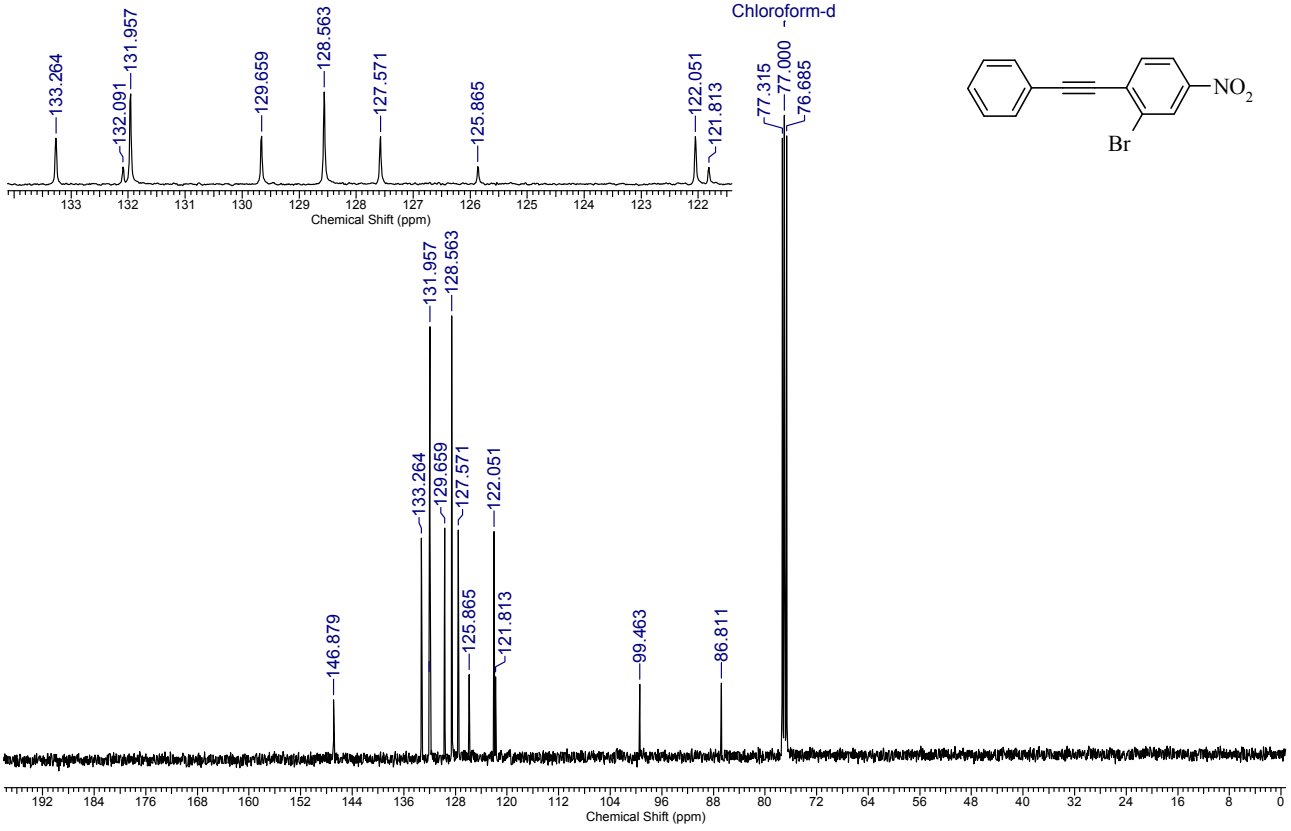
IR spectrum (KBr) of 2-bromo-4-nitrotolane (2-bromo-4-nitro-1-(phenylethynyl)benzene) (**4af**)



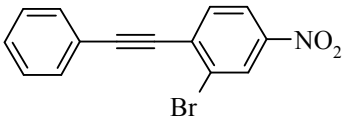
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 2-bromo-4-nitrotolane (**4af**)

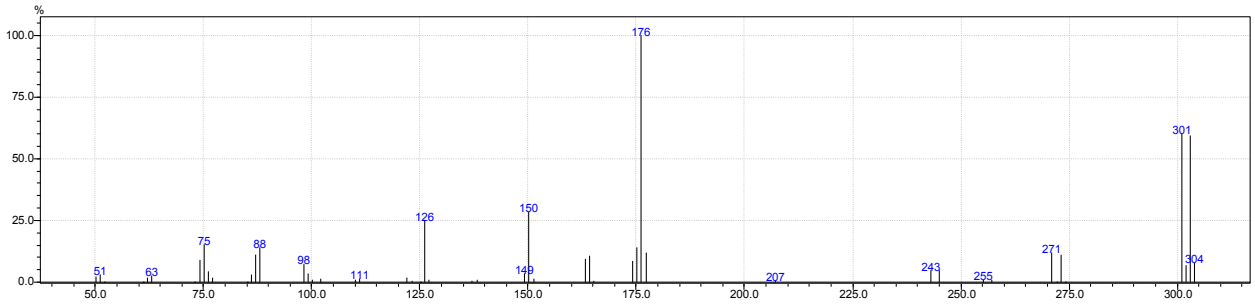


^{13}C NMR spectrum (100 MHz, $CDCl_3$) of 2-bromo-4-nitrotolane (**4af**)

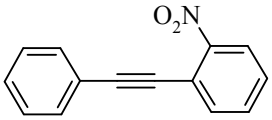


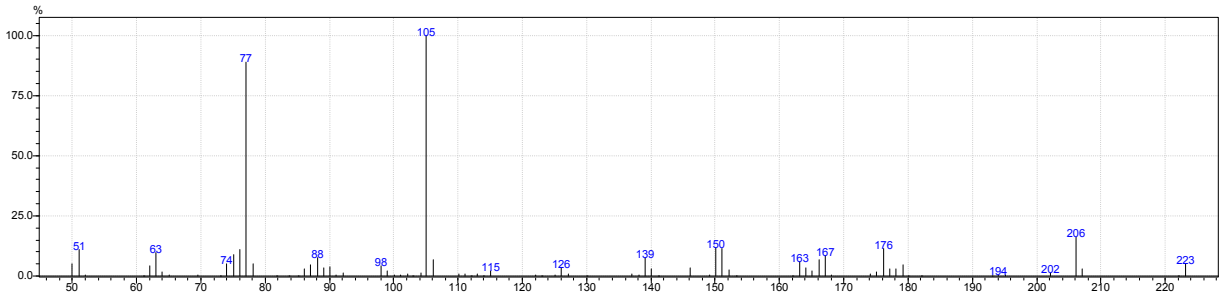
Mass-spectrum (EI, 70 eV) of 2-bromo-4-nitrotolane (**4af**)



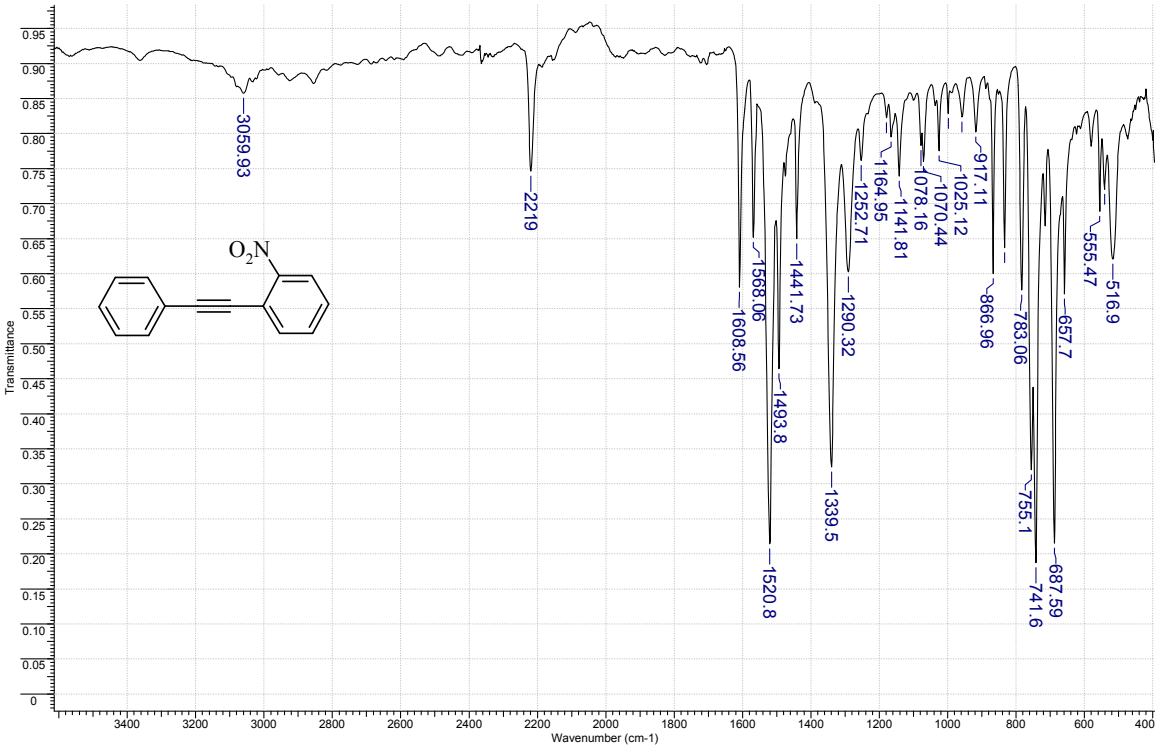


Mass-spectrum (EI, 70 eV) of 2-nitrotolane (**4ak**)

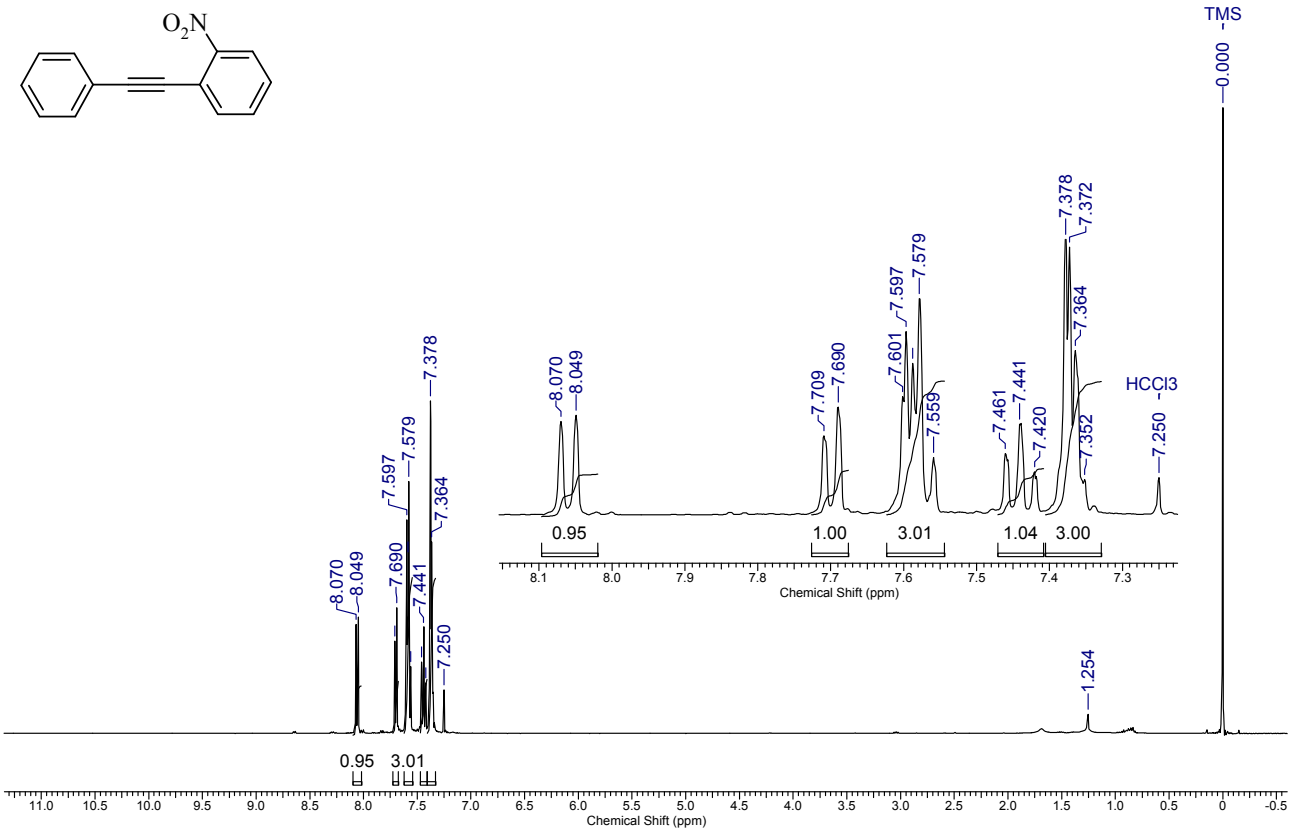




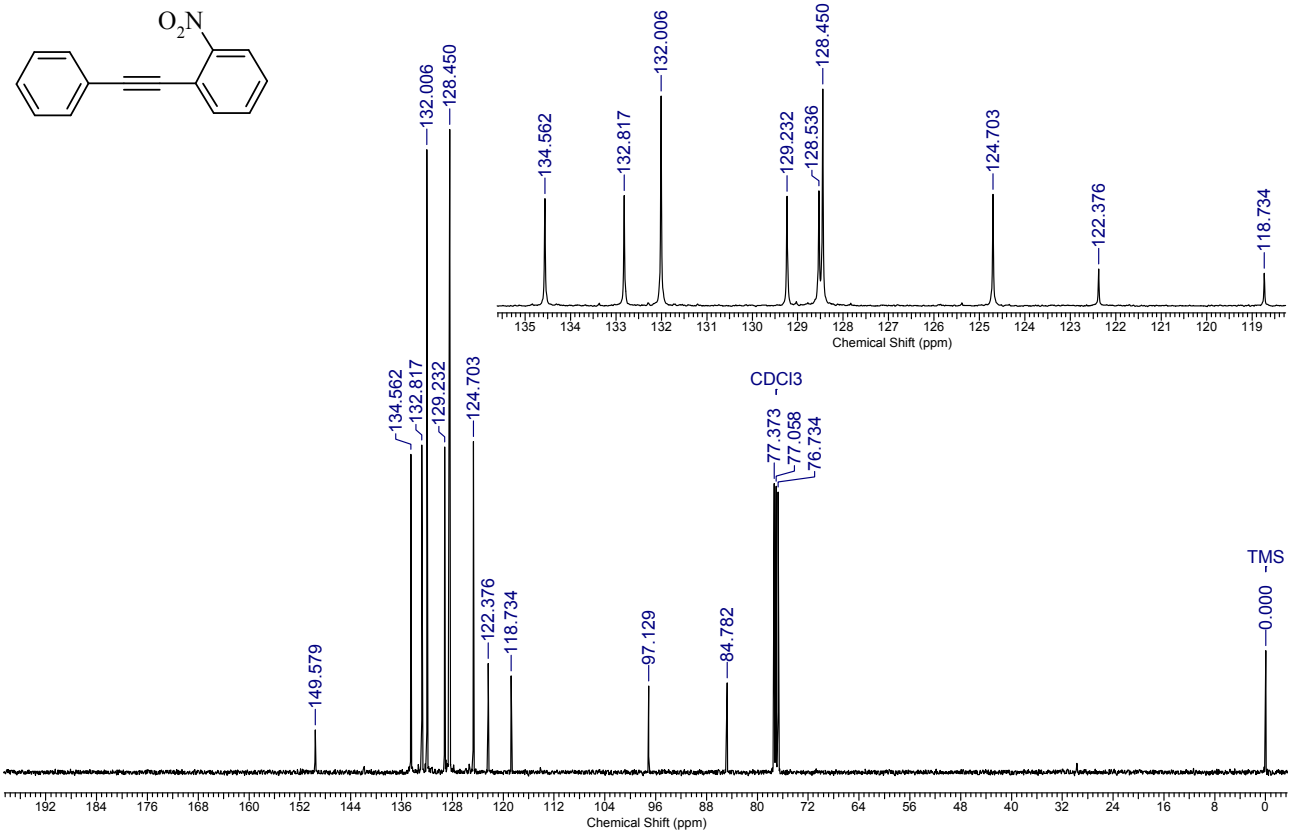
IR spectrum (KBr) of 2-nitrotolane (**4ak**)



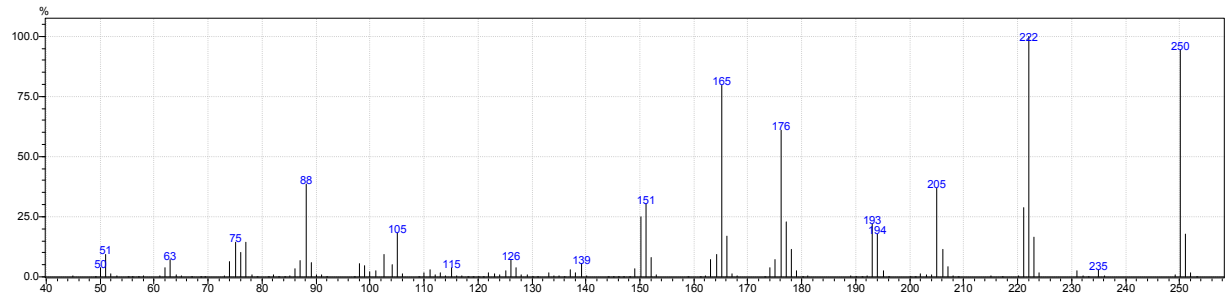
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 2-nitrotolane (**4ak**)



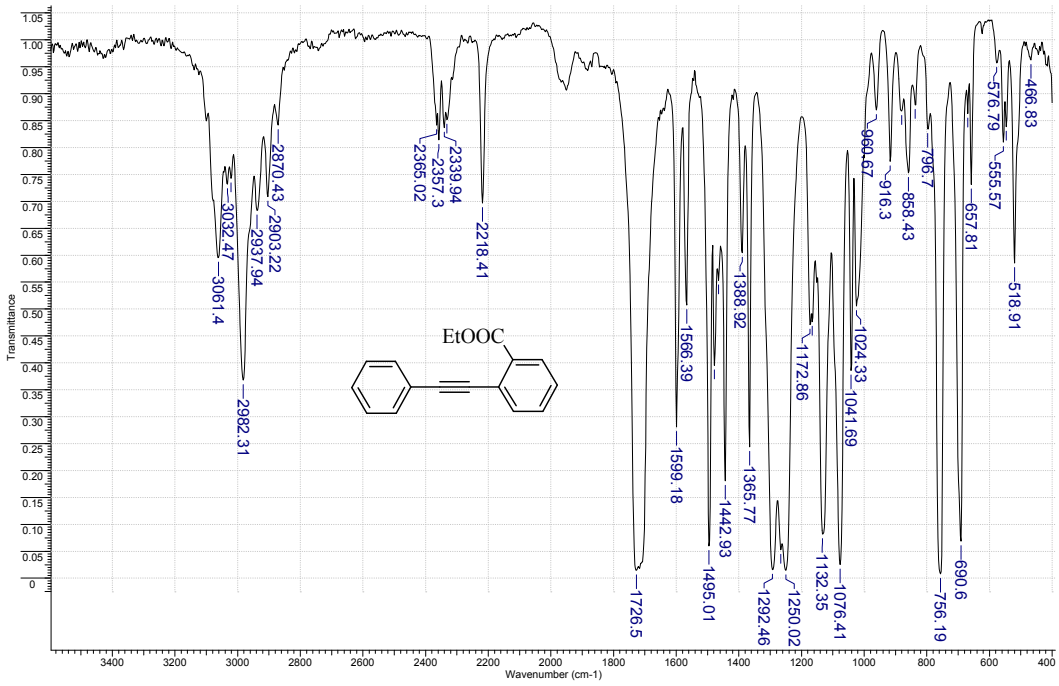
^{13}C NMR spectrum (100 MHz, $CDCl_3$) of 2-nitrotolane (**4ak**)



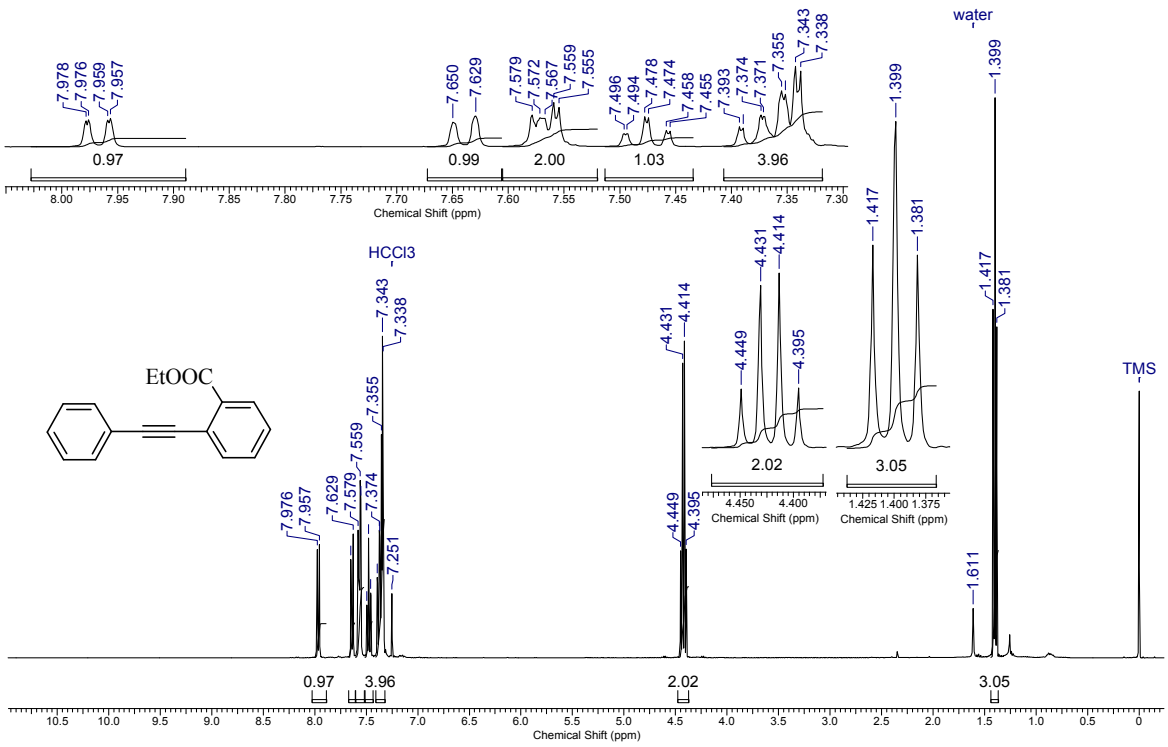
Mass-spectrum (EI, 70 eV) of ethyl 2-(phenylethynyl)benzoate (**4am**)



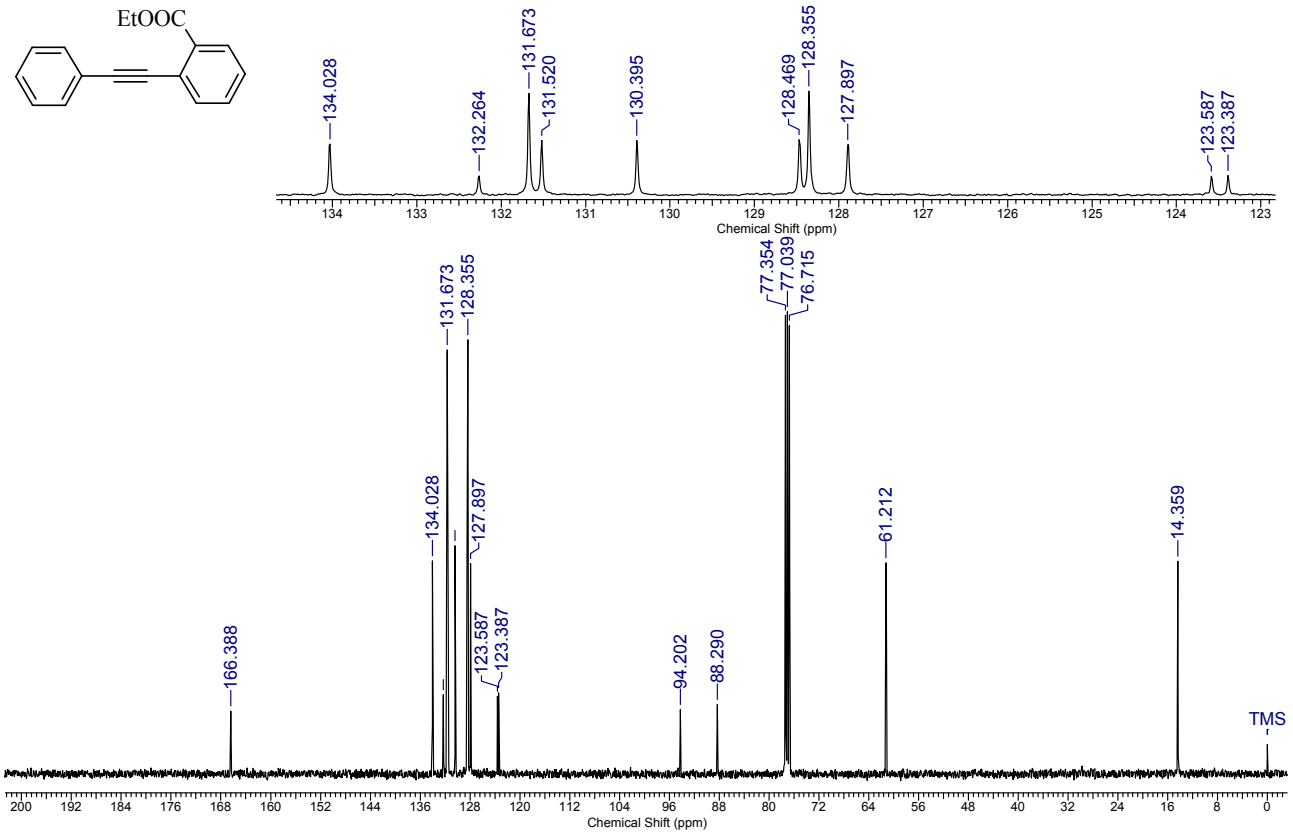
IR spectrum (KBr) of ethyl 2-(phenylethynyl)benzoate (**4am**)



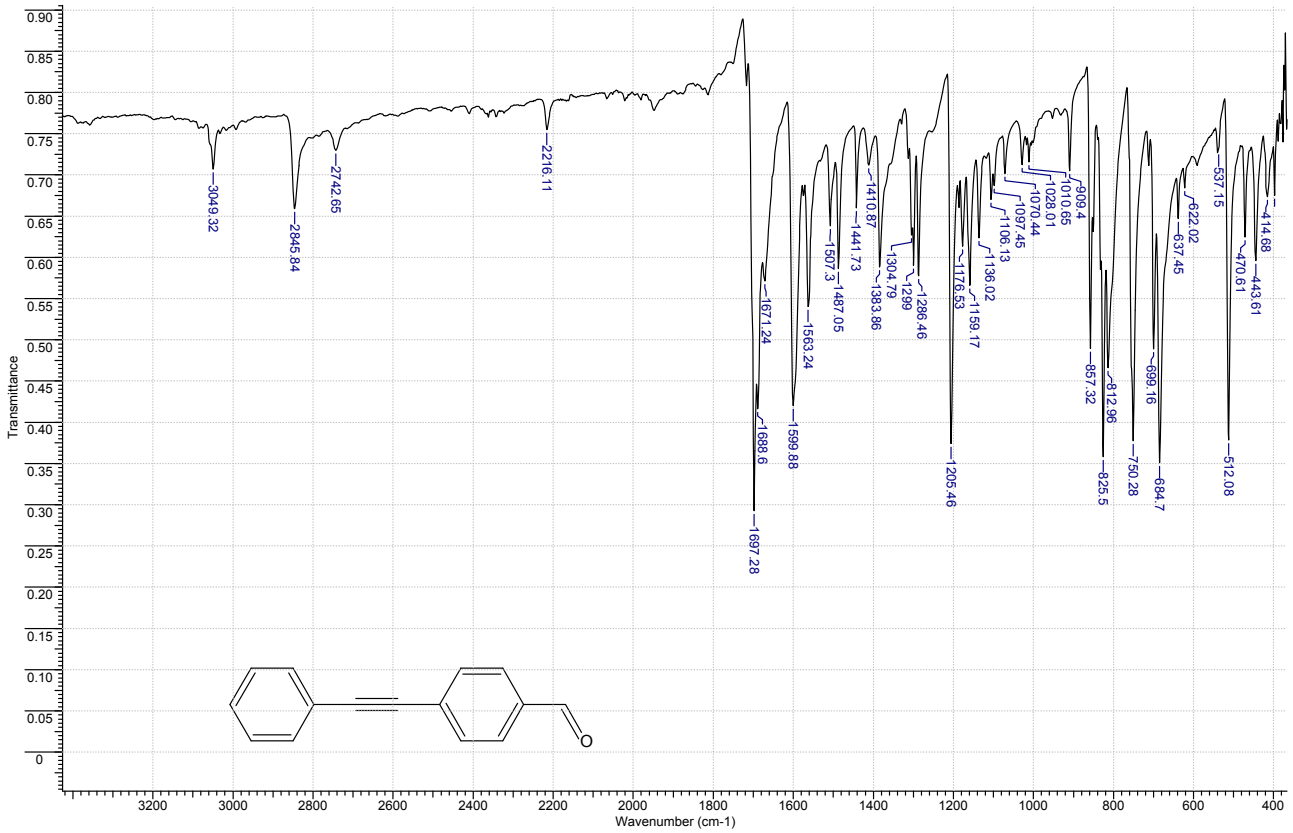
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of ethyl 2-(phenylethynyl)benzoate (**4am**)



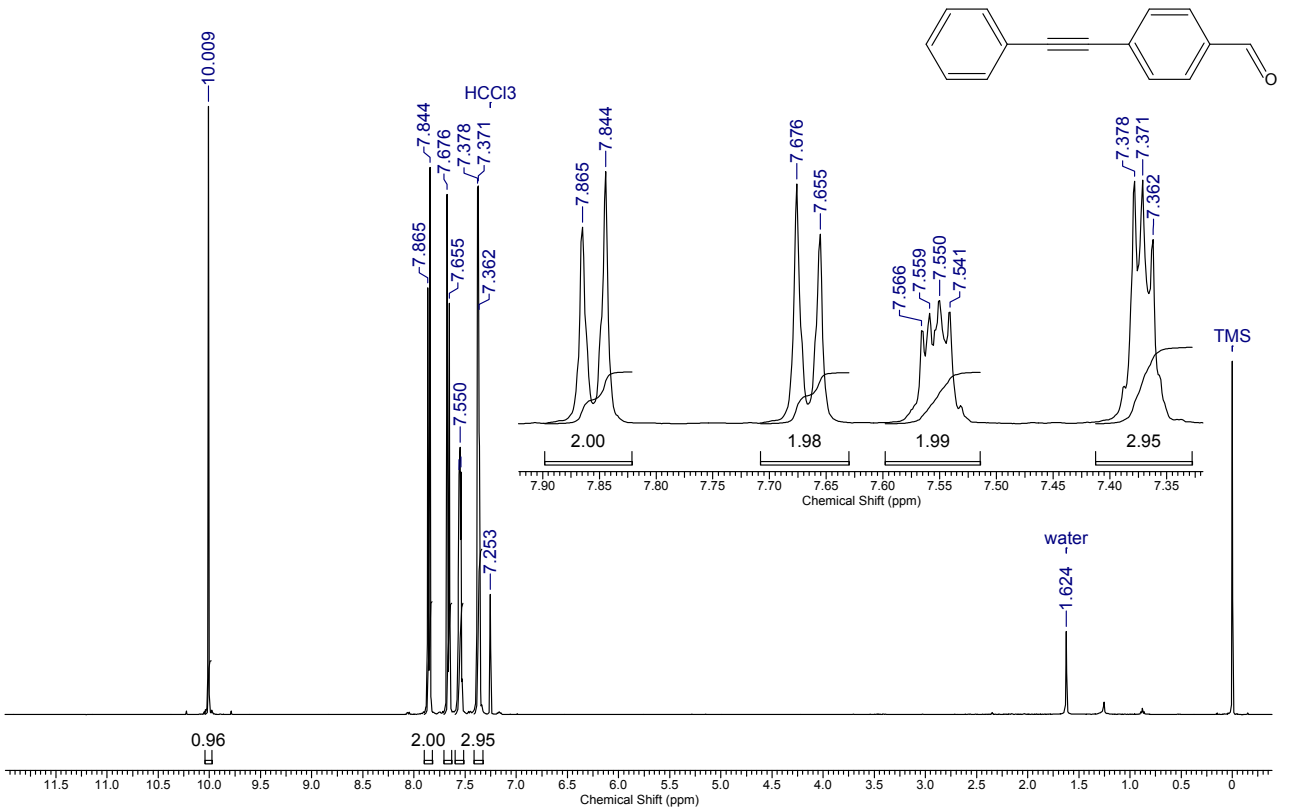
^{13}C NMR spectrum (100 MHz, $CDCl_3$) of ethyl 2-(phenylethynyl)benzoate (**4am**)



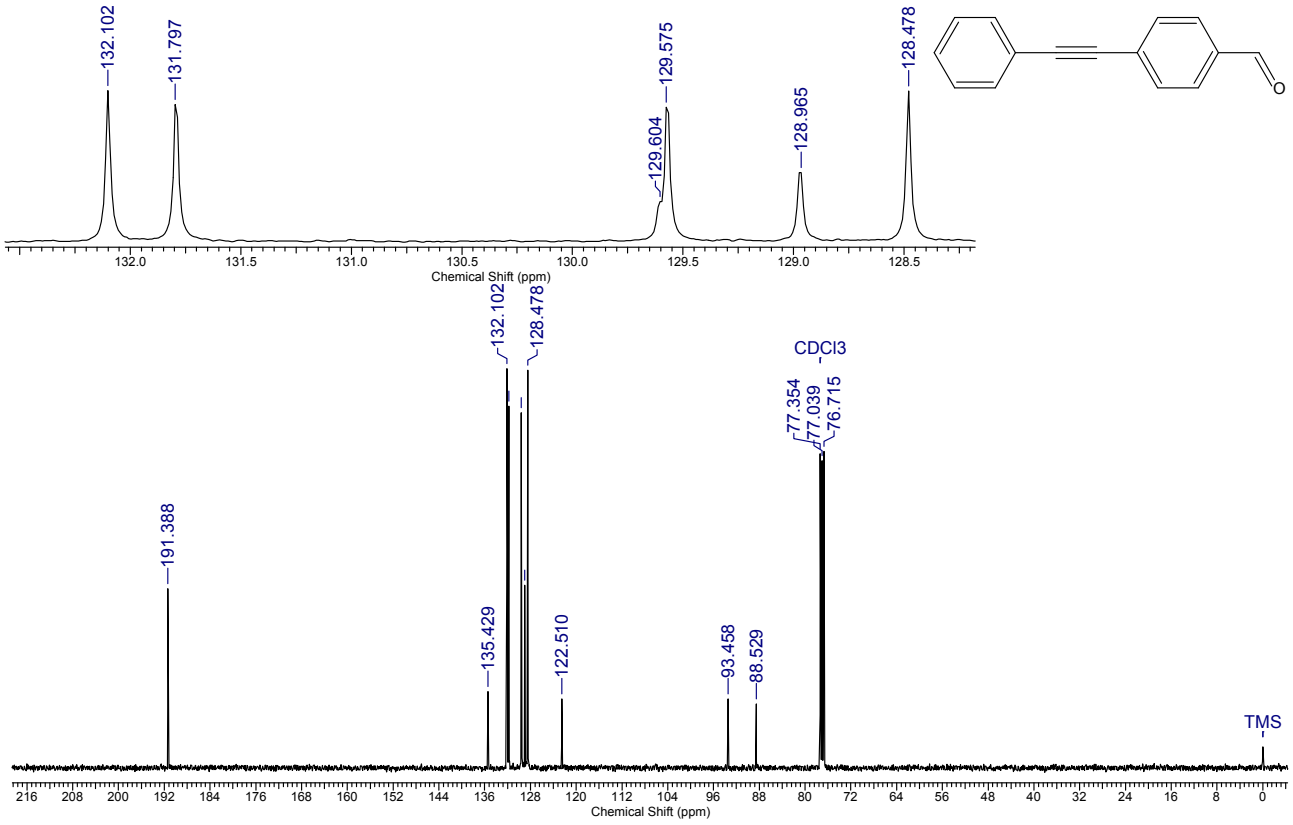
IR spectrum (KBr) of 4-(phenylethynyl)benzaldehyde (**4an**)



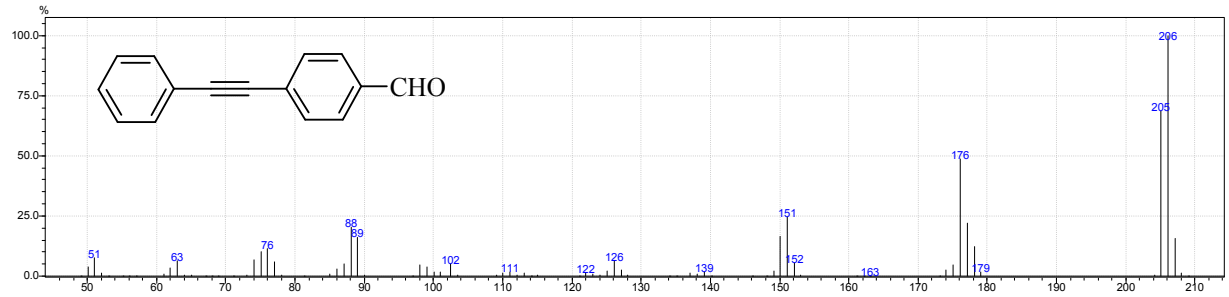
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 4-(phenylethynyl)benzaldehyde (**4an**)



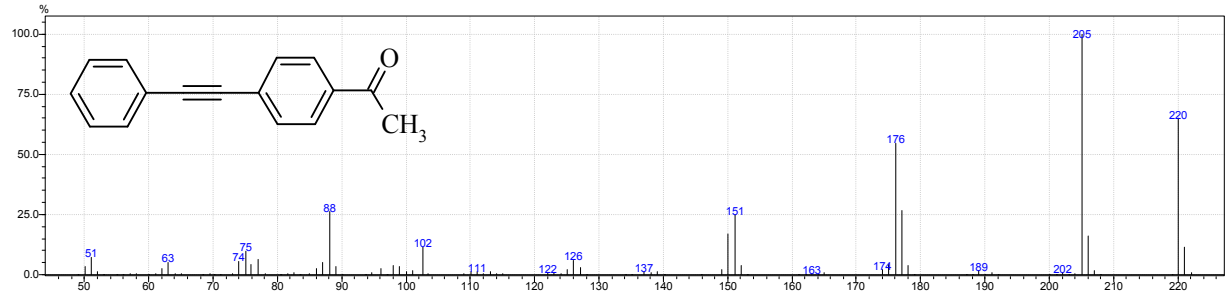
^{13}C NMR spectrum (100 MHz, $CDCl_3$) of 4-(phenylethynyl)benzaldehyde (**4an**)



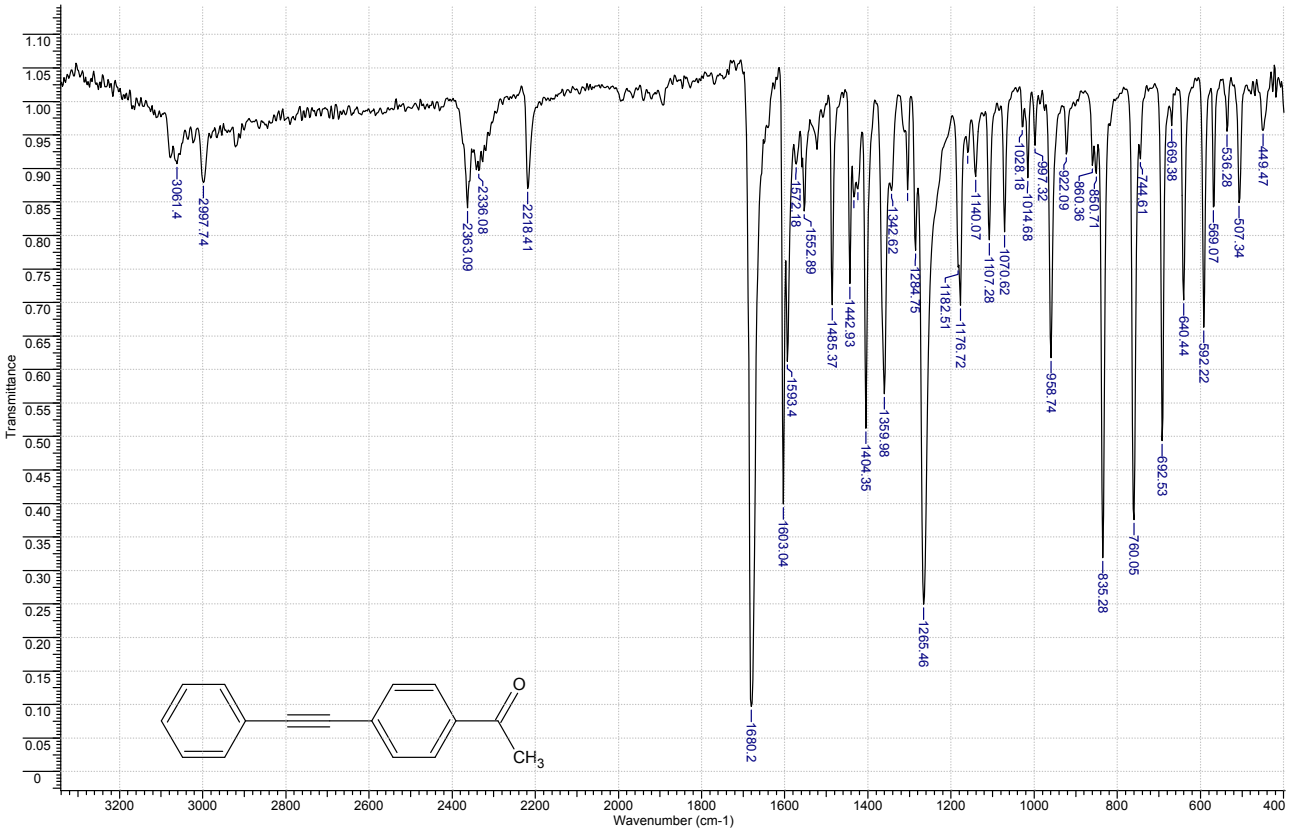
Mass-spectrum (EI, 70 eV) of 4-(phenylethynyl)benzaldehyde (**4an**)



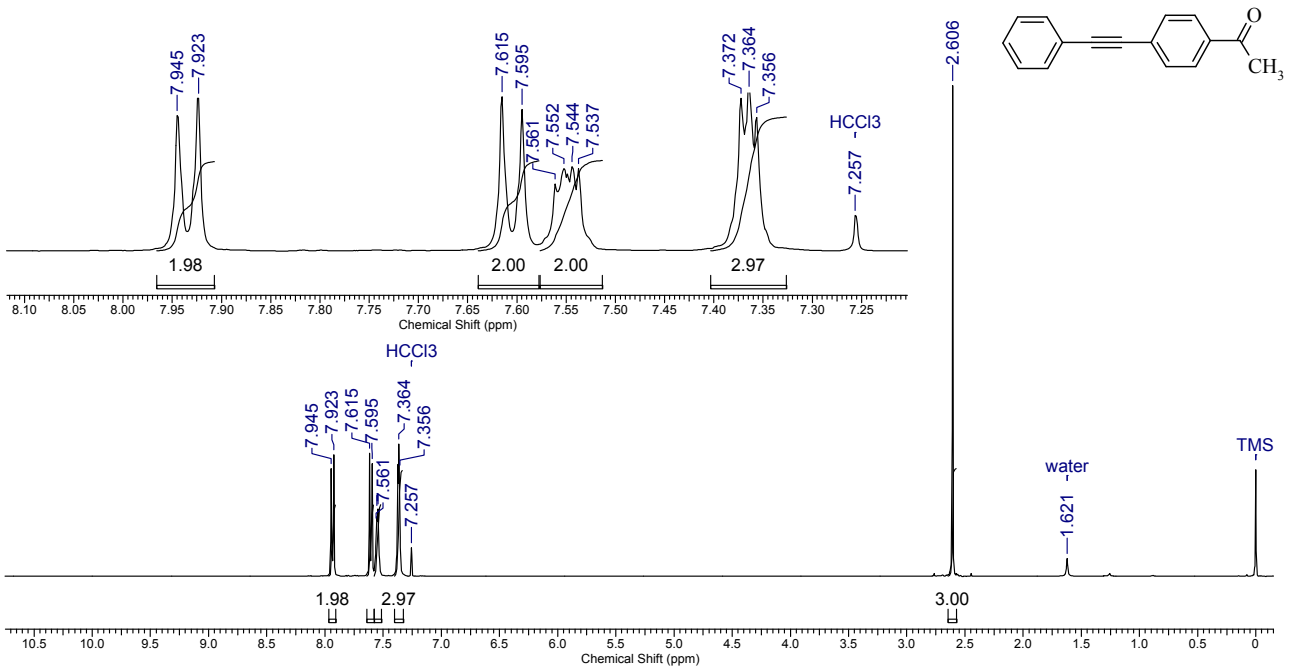
Mass-spectrum (EI, 70 eV) of 1-[4-(phenylethynyl)phenyl]ethanone (**4ao**)



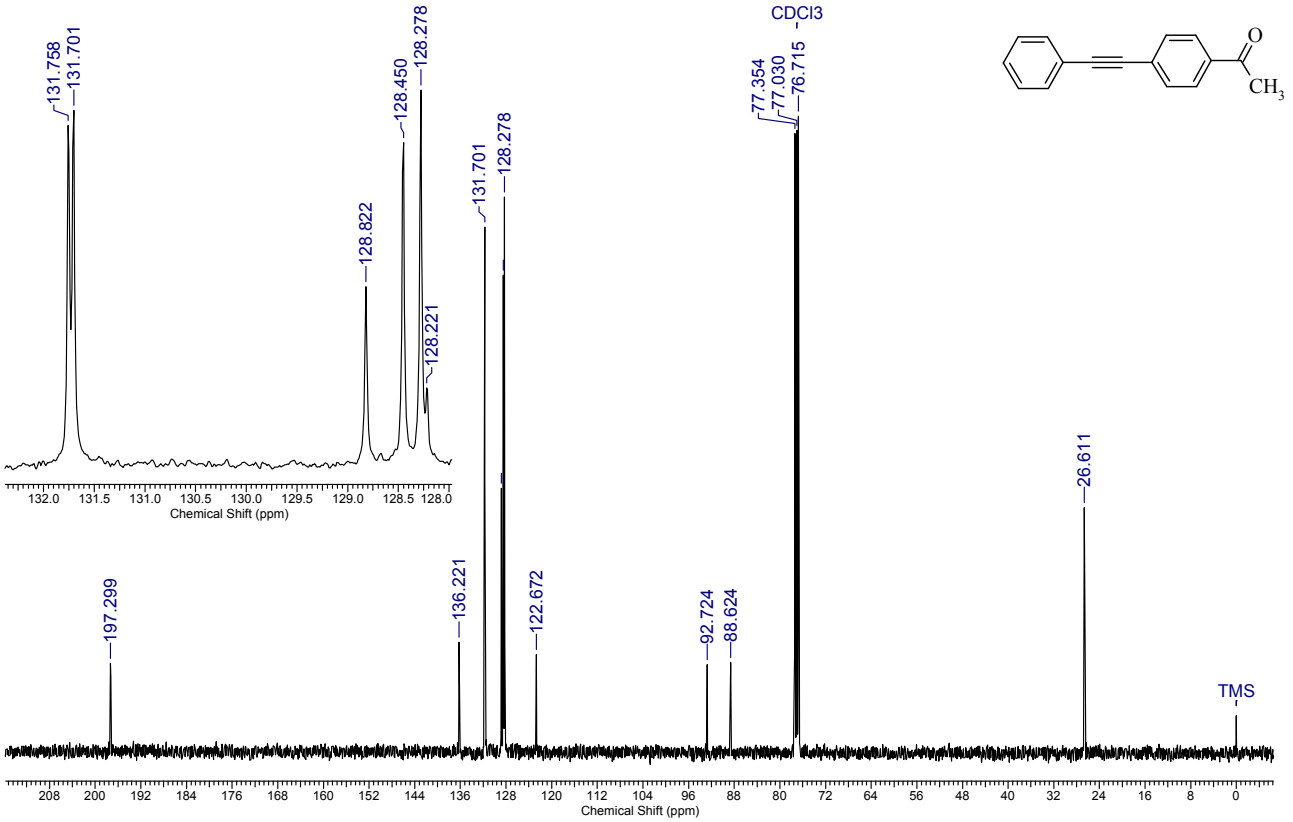
IR spectrum (KBr) of 1-[4-(phenylethynyl)phenyl]ethanone (**4ao**)



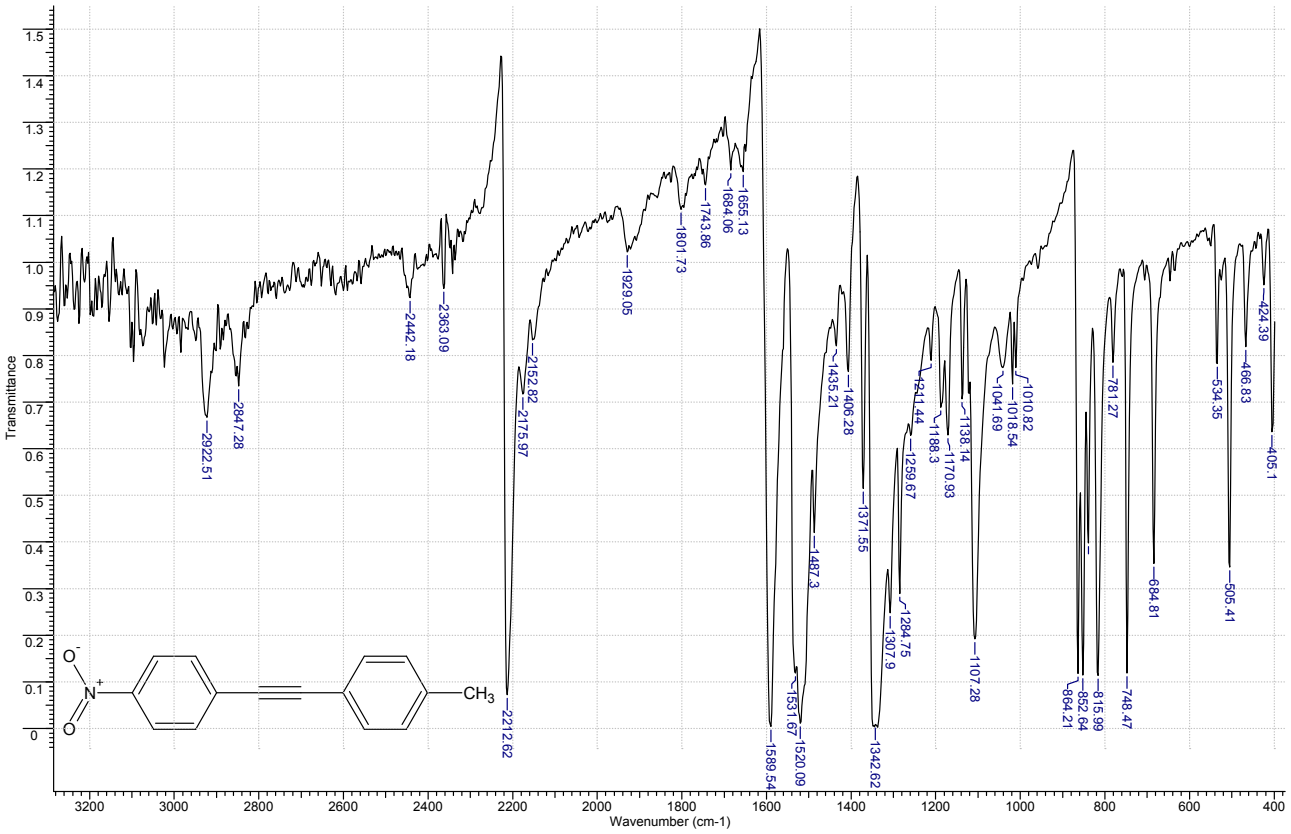
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 1-[4-(phenylethynyl)phenyl]ethanone (**4ao**)



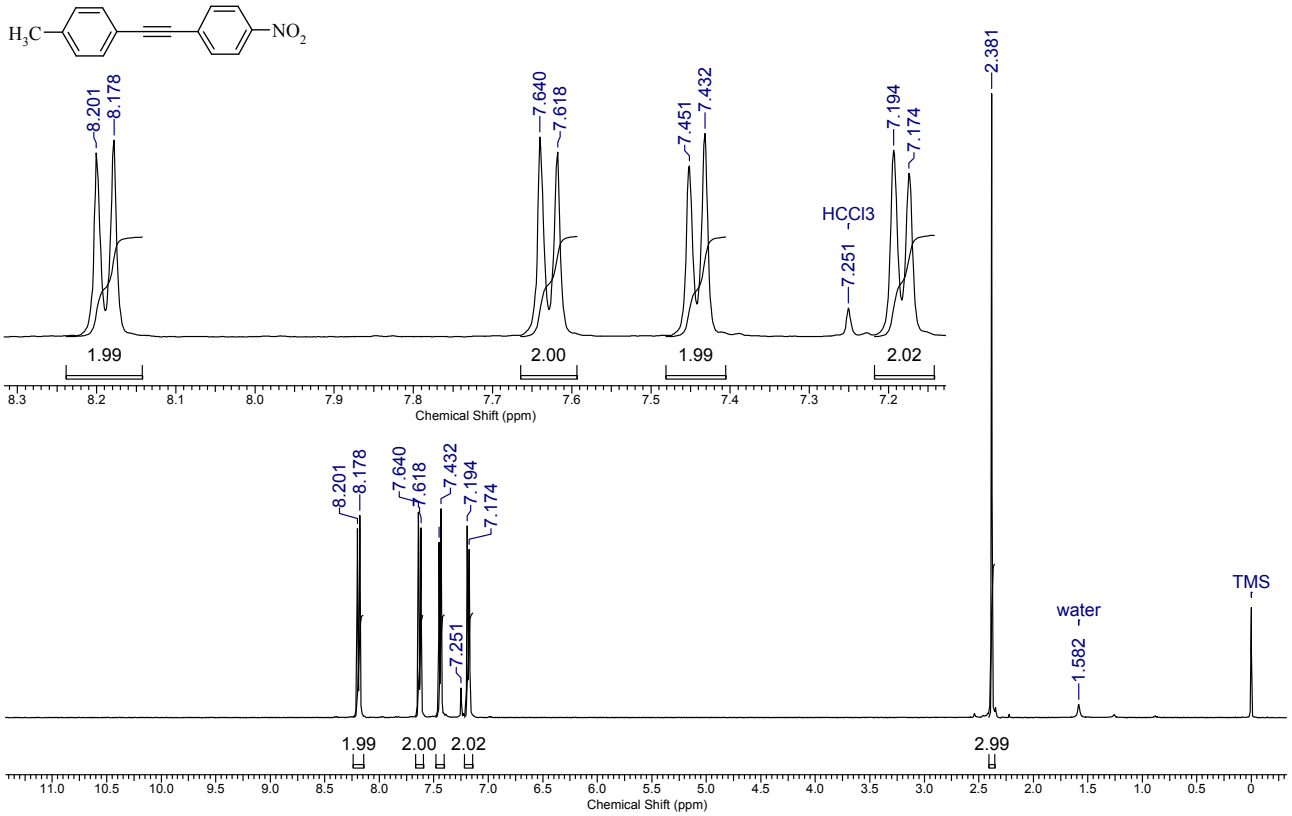
^{13}C NMR spectrum (100 MHz, $CDCl_3$) of 1-[4-(phenylethynyl)phenyl]ethanone (**4ao**)



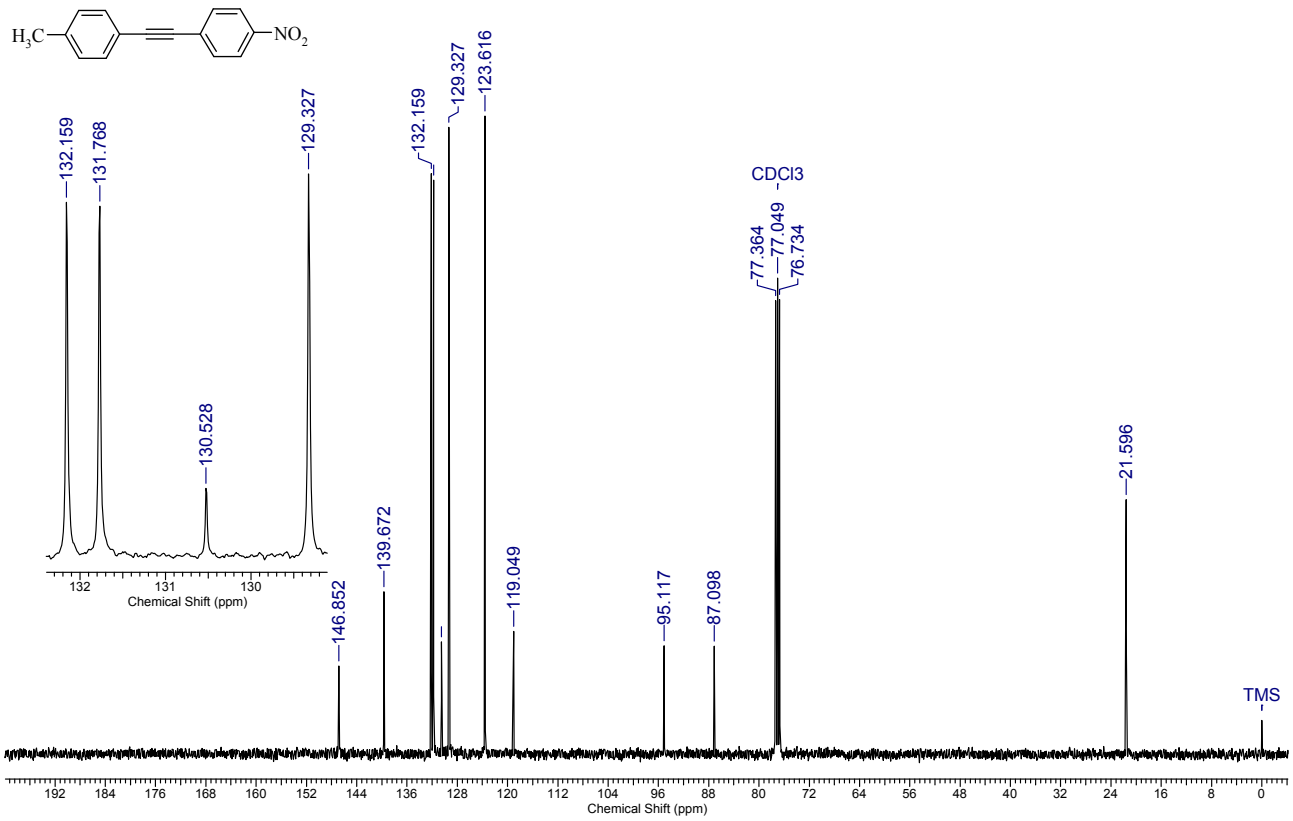
IR spectrum (KBr) of 1-methyl-4-[(4-nitrophenyl)ethynyl]benzene (**4bd**)



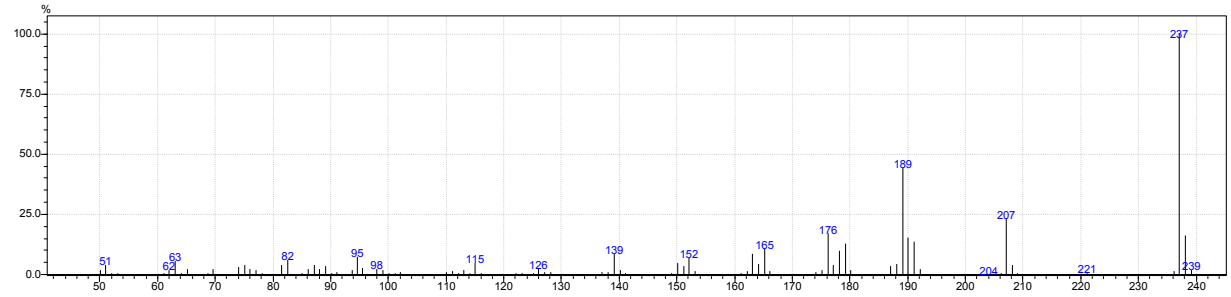
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 1-methyl-4-[(4-nitrophenyl)ethynyl]benzene (**4bd**)



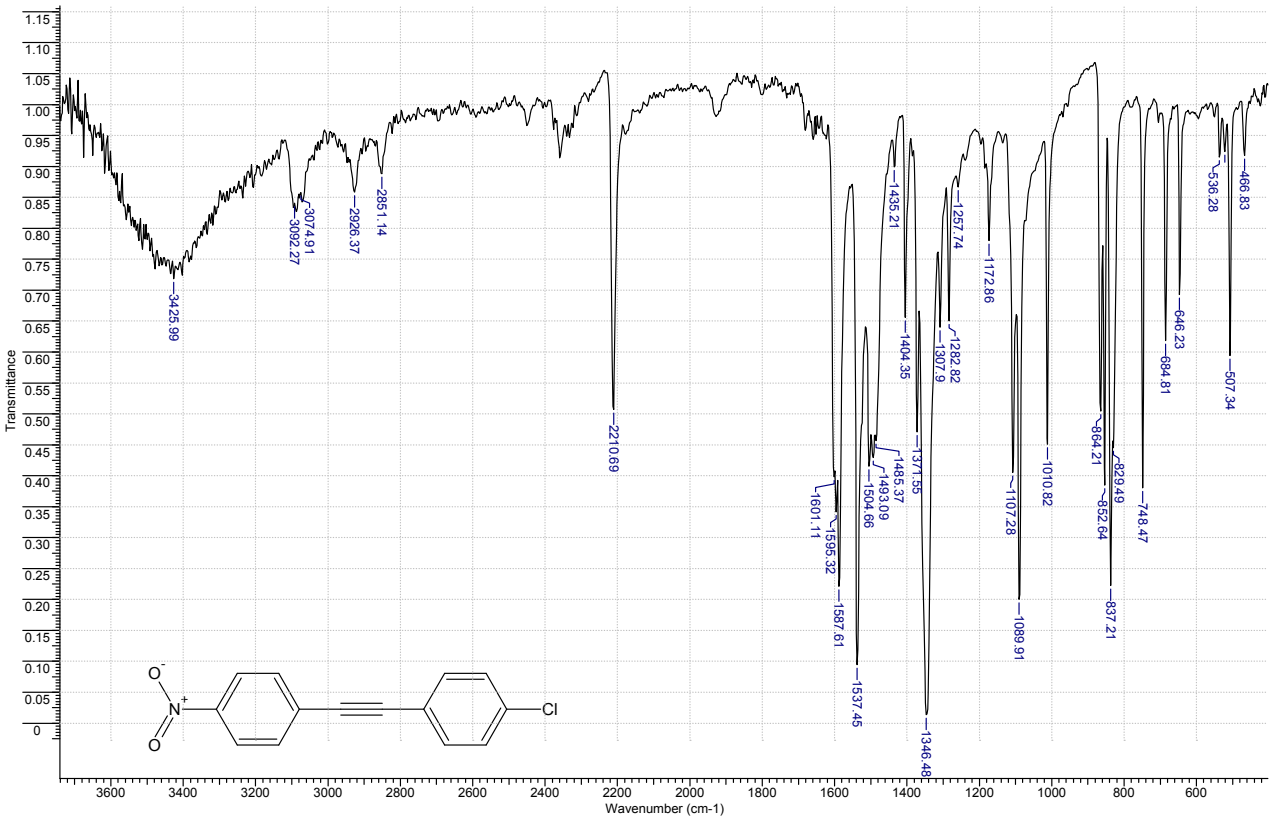
^{13}C NMR spectrum (100 MHz, $CDCl_3$) of 1-methyl-4-[(4-nitrophenyl)ethynyl]benzene (**4bd**)



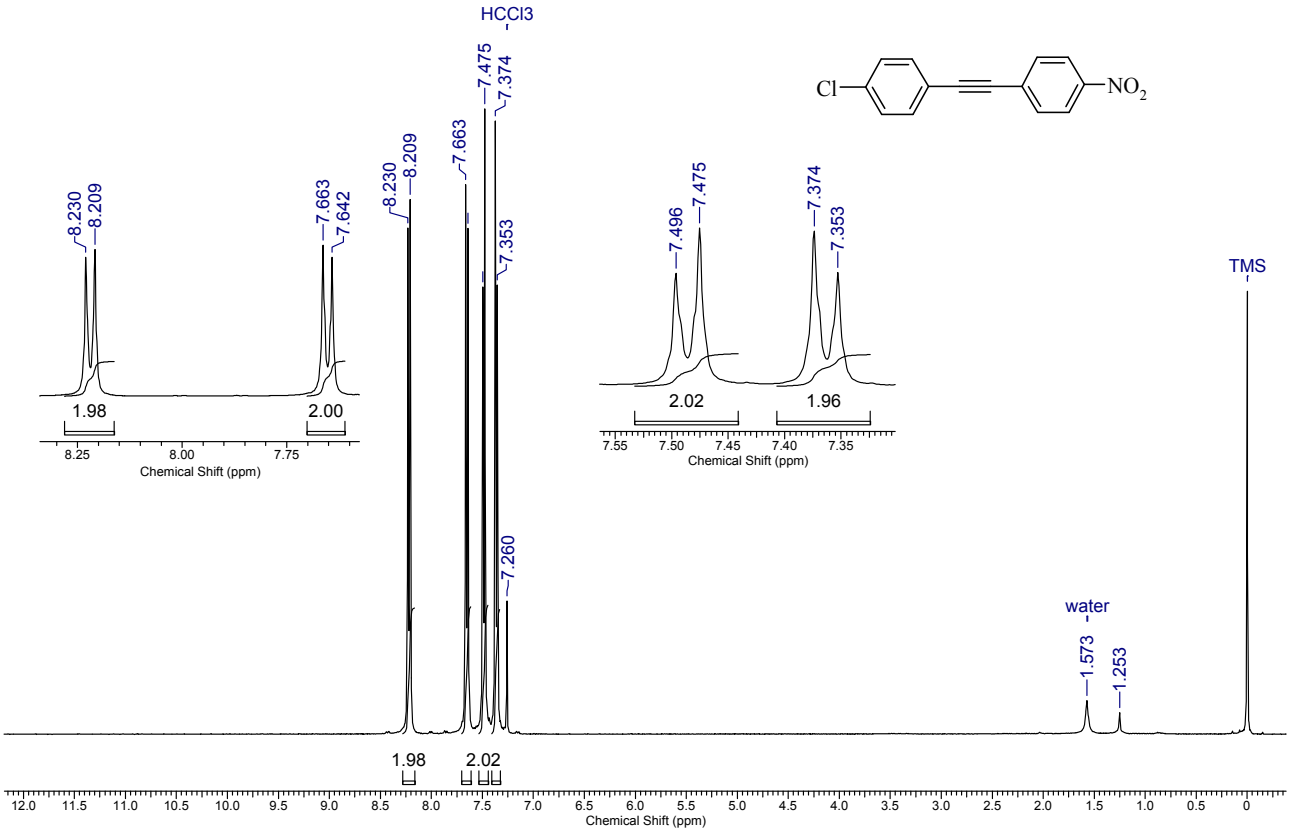
Mass-spectrum (EI, 70 eV) of 1-methyl-4-[(4-nitrophenyl)ethynyl]benzene (**4bd**)



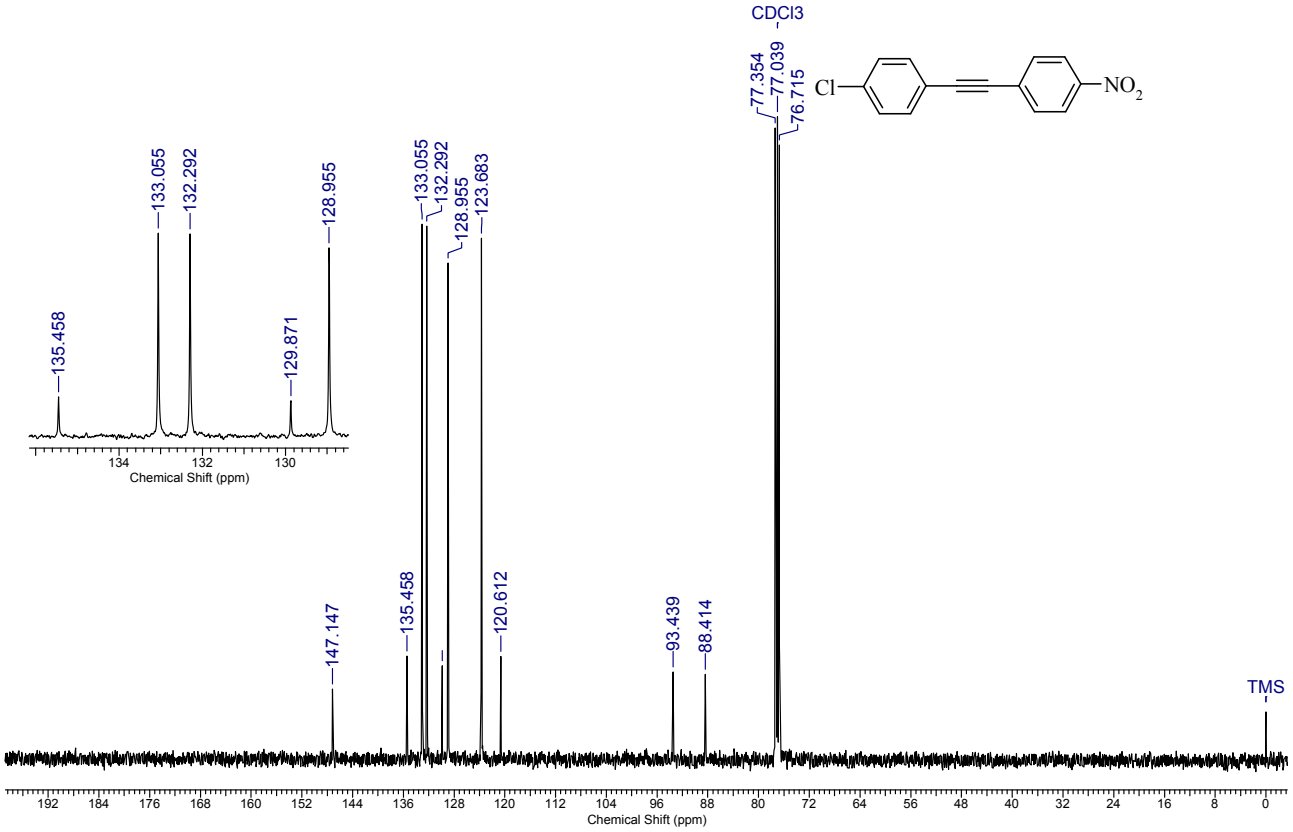
IR spectrum (KBr) of 1-chloro-4-[(4-nitrophenyl)ethynyl]benzene (**4cd**)



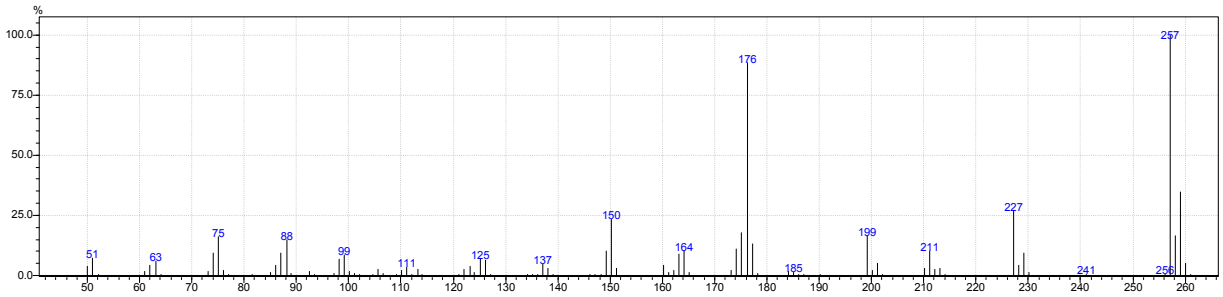
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 1-chloro-4-[(4-nitrophenyl)ethynyl]benzene (**4cd**)



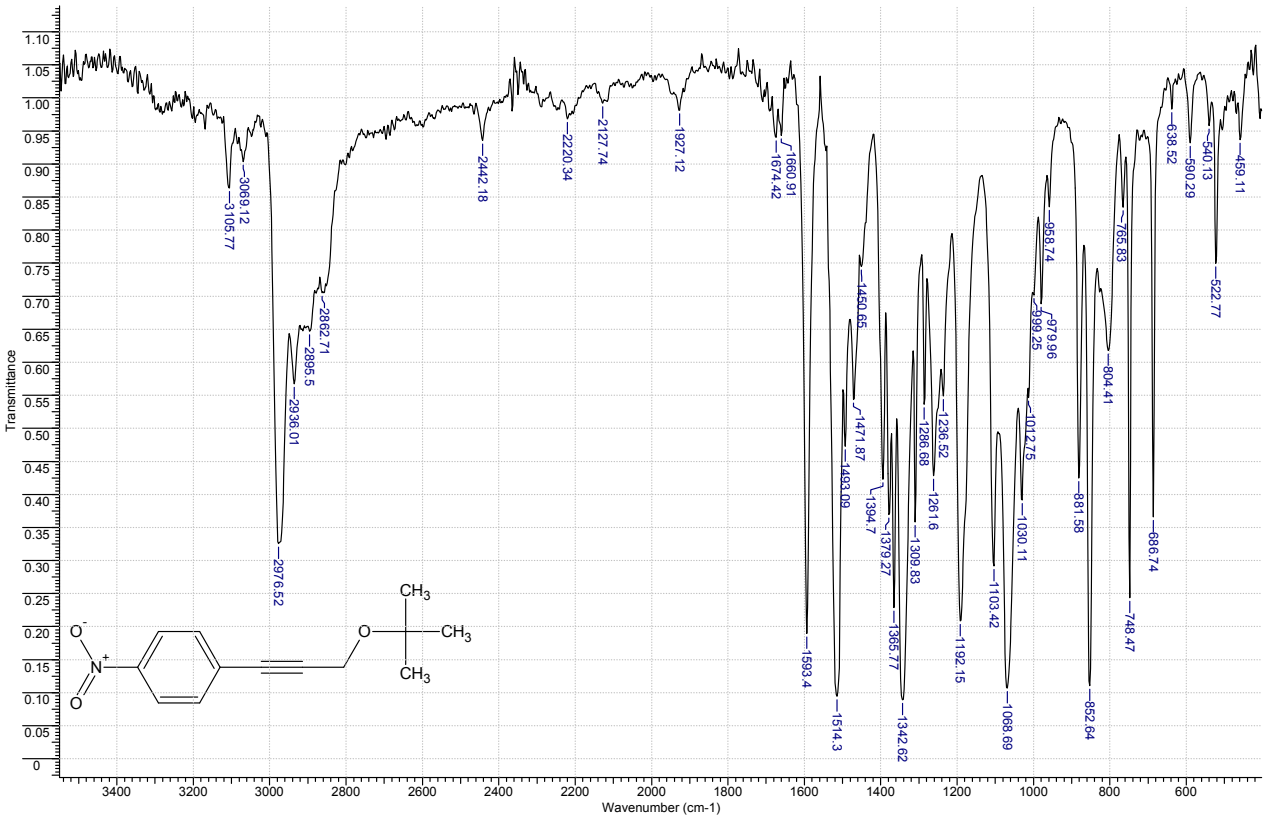
^{13}C NMR spectrum (100 MHz, $CDCl_3$) of 1-chloro-4-[(4-nitrophenyl)ethynyl]benzene (**4cd**)



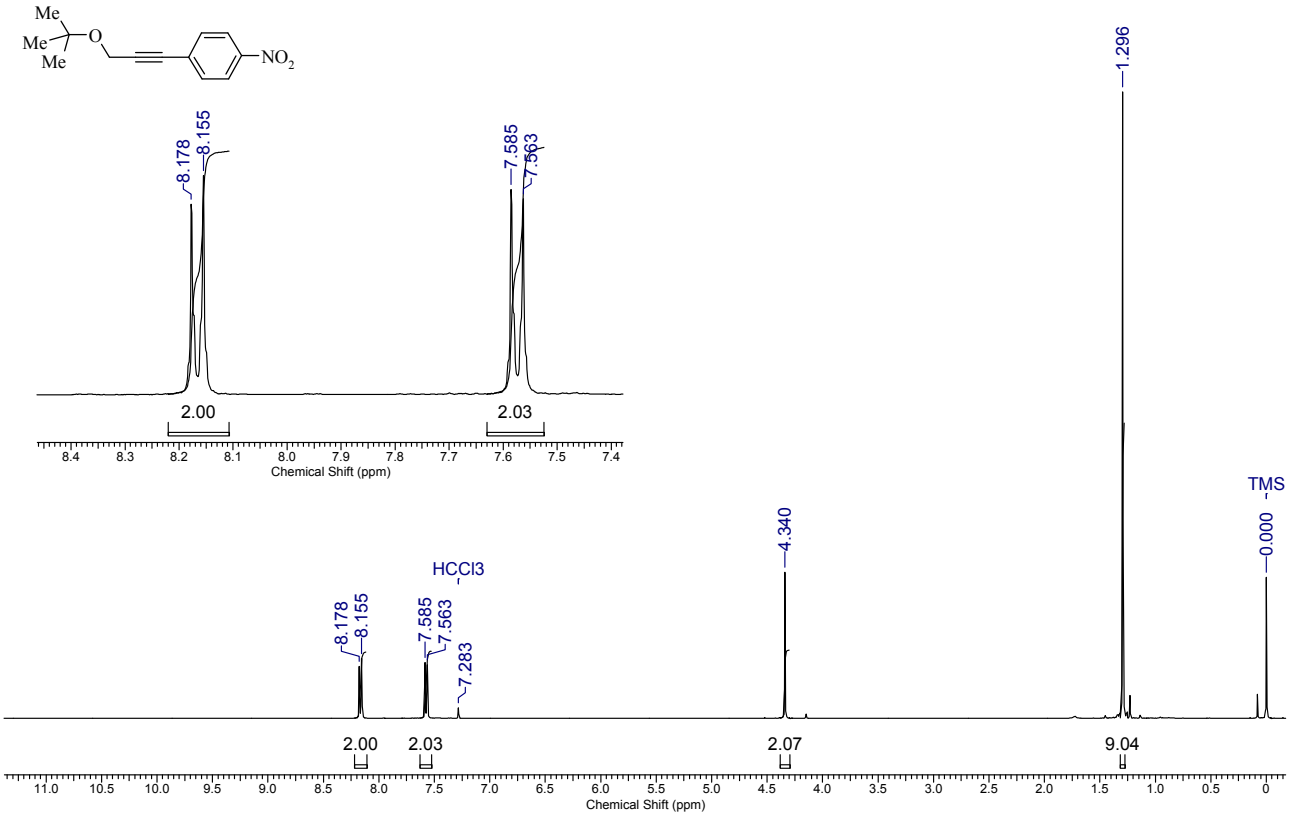
Mass-spectrum (EI, 70 eV) of 1-chloro-4-[(4-nitrophenyl)ethynyl]benzene (**4cd**)



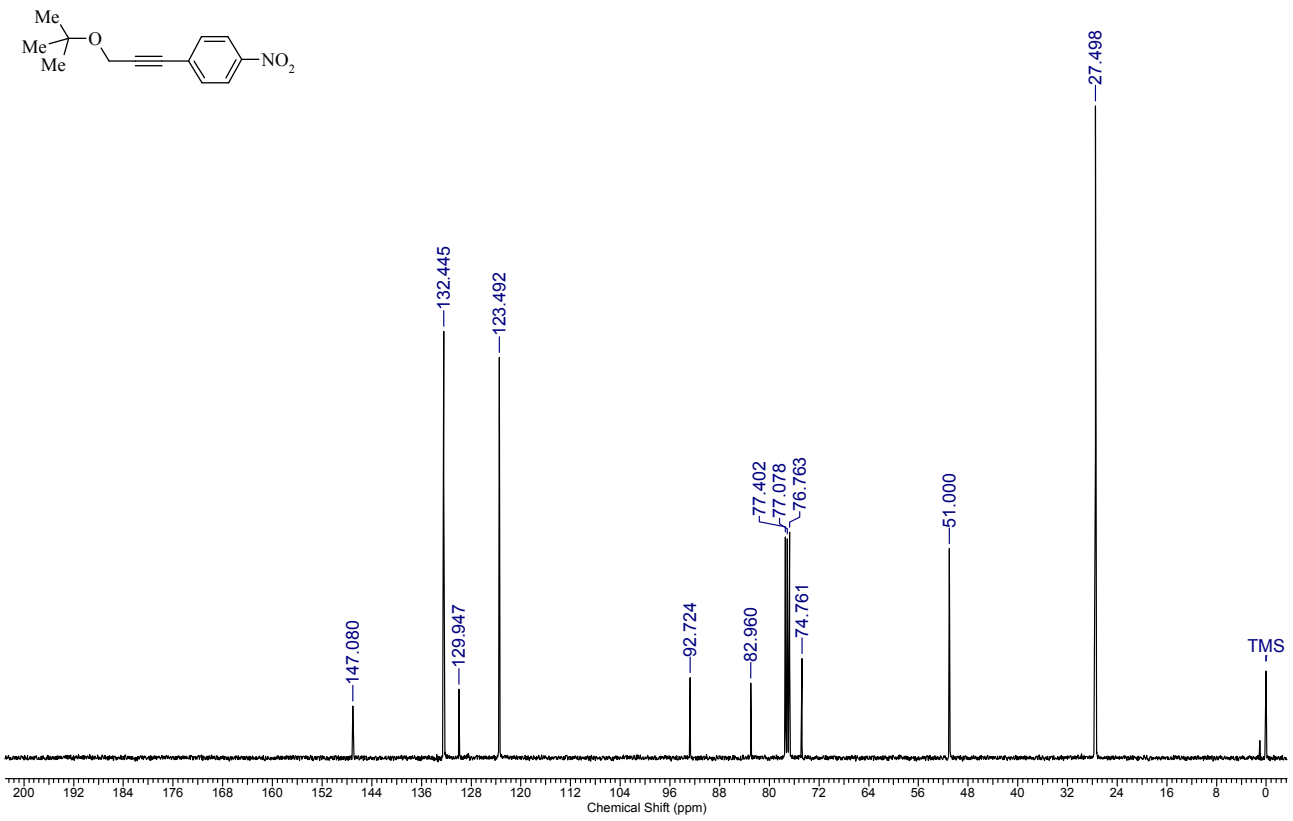
IR spectrum (KBr) of *tert*-Butyl 3-(4-nitrophenyl)prop-2-ynyl ether (**4ed**)



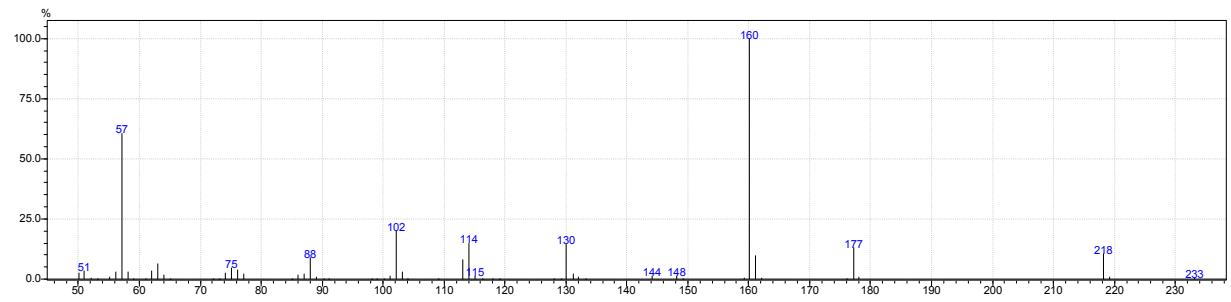
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of *tert*-Butyl 3-(4-nitrophenyl)prop-2-ynyl ether (**4ed**)



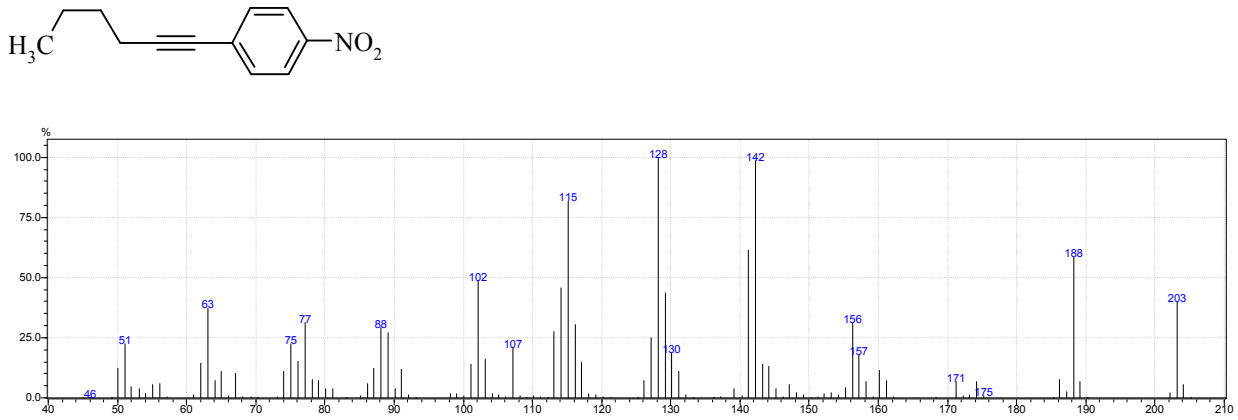
^{13}C NMR spectrum (100 MHz, $CDCl_3$) of *tert*-butyl 3-(4-nitrophenyl)prop-2-ynyl ether (**4ed**)



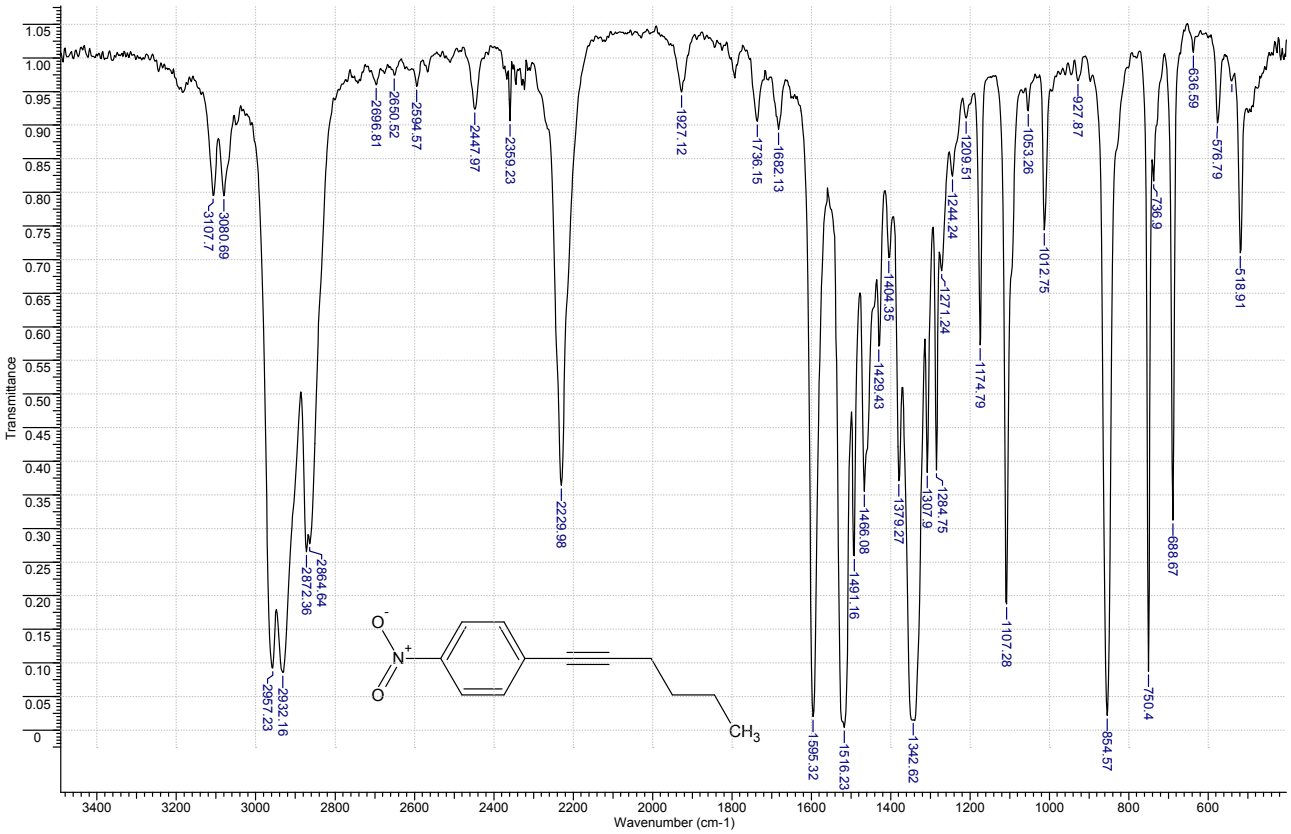
Mass-spectrum (EI, 70 eV) of *tert*-butyl 3-(4-nitrophenyl)prop-2-ynyl ether (**4ed**)



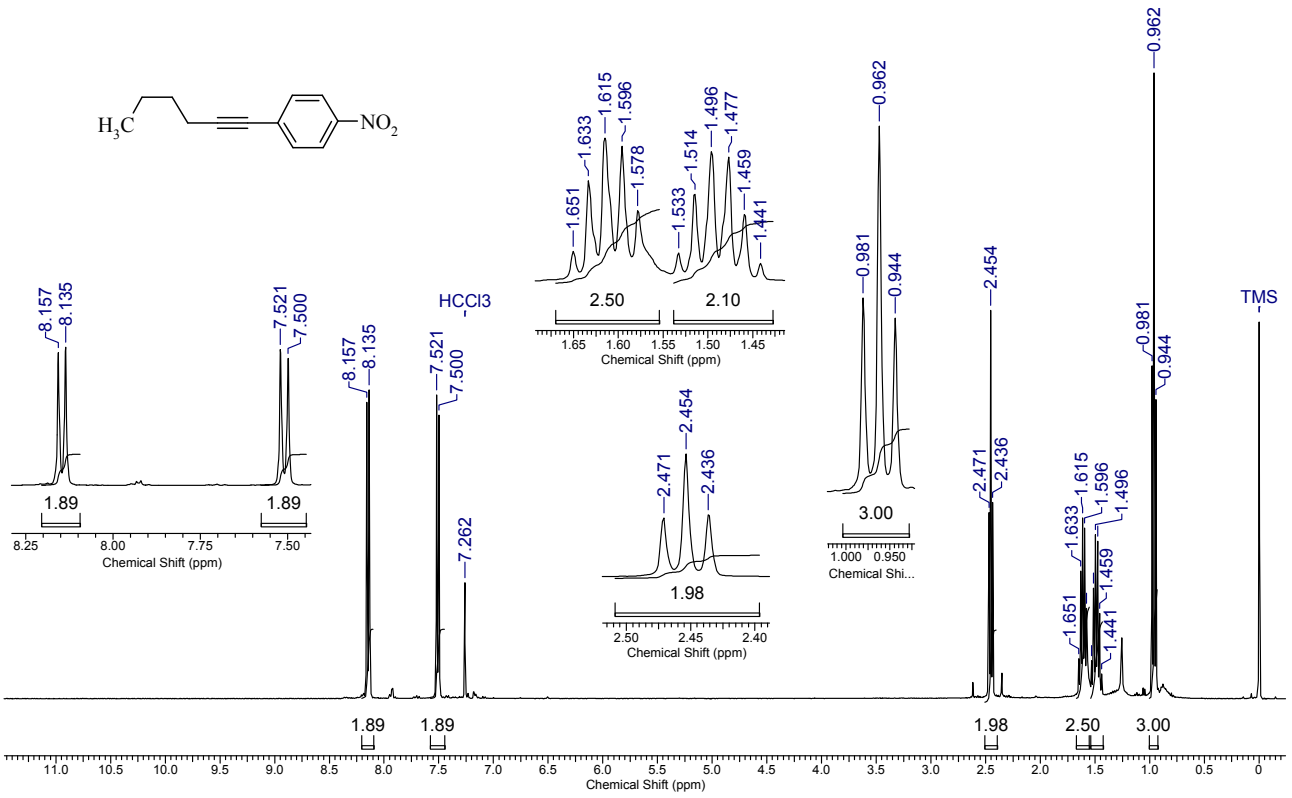
Mass-spectrum (EI, 70 eV) of 1-hex-1-ynyl-4-nitrobenzene (**4fd**)



IR spectrum (KBr) of 1-hex-1-ynyl-4-nitrobenzene (**4fd**)



^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 1-hex-1-ynyl-4-nitrobenzene (**4fd**)



^{13}C NMR spectrum (100 MHz, $CDCl_3$) of 1-hex-1-ynyl-4-nitrobenzene (**4fd**)