ELETTRONIC SUPPLEMENTARY INFORMATION

Valorisation of urban waste to access low-cost heterogeneous palladium catalyst for cross-coupling reactions in biomass-derived γ-valerolactone.

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1. General Remarks

Unless otherwise stated, all chemicals were purchased and used without any further purification. GLC analyses were performed by using Hewlett-Packard HP 5890 SERIES II equipped with a capillary column DB-5MS (30 m, 0.32 mm), a FID detector, and helium as gas carrier. GC-EIMS analyses were carried out by using a Hewlett-Packard HP 6890N Network GC system/5975 mass selective detector equipped with an electron impact ionizer at 70 eV. Melting points were measured on a Büchi 510 apparatus. NMR spectra were recorded on a Bruker DRX-ADVANCE 400 MHz (^{1}H at 400 MHz, ^{13}C at 100.6 MHz and ^{19}F at 376.4 MHz) in $CDCl_3$. Chemical shifts are reported in ppm (δ), coupling constant (J) in hertz and multiplicity are reported as follows: *s* = singlet, *bs* = broad singlet, *d* = doublet, *dd* = double doublet, *td* = double triplet, *t* = triplet, *m* = multiplet. Products purification was performed using 230-400 mesh silica gel. Elemental Analysis (EA) were conducted on Elementar UNICUBE® elemental analyzer. Metal loading was measured using MP-AES 4210 instrument. TEM images were obtained using a PHILIPS CM 12 transmission electron microscopy operating in the range 20 to 120 kV with an image resolution of 0.34nm.

Characterization data, ^{1}H and ^{13}C NMR spectra are reported below.

2. General procedures

Pine needle pre-treatment

Pine needles from urban waste were chopped into pieces of 1-2 cm and dried at 110°C for 1 day.[1] Then, the dry biomass was grinded and sieved. 7.05 g of the obtained material was extracted in Soxhlet apparatus with toluene methanol azeotrope (98% recovered) for 8h in order to remove oil, waxes and proteins and dried at 80°C under vacuum to afford 6.57 g of dry lignocellulosic biomass.[2]

Preparation of PiNe support

1 g of the pre-treated pine needles material was placed in Erlenmeyer flask and sonicated for 30 min with 20 mL of sulfuric acid. After sonication, the material was kept in sulfuric acid for 3 days[3] and then filtered and washed several times with water until neutral pH. The recovered material (736 mg) was then dried under vacuum at 110°C. The formation of active carbon has been confirmed by methylene blue test.

Preparation of Pd/PiNe

In a 50 mL two neck round bottom flask 400 mg of PiNe support were suspended in 20 mL of diethylene glycol. Aqueous solution of H_2PdCl_4 (prepared dissolving 87 mg of $PdCl_2$ in 1 mL HCl) was added dropwise under sonication. The pH was adjusted to 12-13 by adding NaOH 5M solution. The reduction step has been carried out at 130°C for 3h under Ar atmosphere. The suspension was filtered and washed several times with hot water.[4] The loading of Pd (9.9 wt%) was measured by MP-AES analysis.

Typical procedure for Heck-Mizoroki reaction (extraction work up).

In a 4 mL screw-capped vial equipped with a magnetic stirrer, the aryl iodide **1** (1mmol), the alkene **2** or **4** (1.2 mmol), GVL (0.8 M, 1.25 mL), diethylaminomethyl-polystyrene (1.5 equiv, 0.47 g) and **Pd/PiNe** (0.1 mol%, 1.1 mg) were consecutively added, and the resulting mixture was left under stirring at 130 °C. After 4 h, the heterogeneous base and catalyst were filtered off and washed with 1 mL of GVL. The solvent was distilled and recovered (94%) as pure solvent and the product extracted with 3 mL of CPME. The organic phase was washed with water (3x2 mL), dried over Na_2SO_4 and the solvent removed under vacuum to afford pure products **3** or **5**.

Typical procedure for Heck-Mizoroki reaction (precipitation work up).

In a 4 mL screw-capped vial equipped with a magnetic stirrer, the aryl iodide **1** (1mmol), the alkene **2** or **4** (1.2 mmol), GVL (0.8 M, 1.25 mL), diethylaminomethyl-polystyrene (1.5 equiv, 0.47 g) and **Pd/PiNe** (0.1 mol%, 1.1 mg) were consecutively added, and the resulting mixture was left under stirring at 130 °C. After 4 h, the heterogeneous base and catalyst were filtered off and washed with 1 mL of GVL. The solvent was distilled and recovered (94%) as pure solvent and the product precipitated with 1 mL of water at 0°C. The precipitate was filtered off and washed with 1 mL of cold water, then dried under vacuum to afford compounds **3** or **5**.

Regeneration procedure for PS-TEA.

The catalyst-base system (470.3 mg) filtered from the reaction mixture was charged into a screw-capped vial equipped with a magnetic stirrer, and TEA (1 eq, 1.5 mmol, 209 µL) in 1 mL GVL was added. The mixture was kept under stirring for 30 min, at 28°C. Next, the catalyst-base system was filtered off, washed with 1 mL of GVL, and dried under vacuum. GVL was distilled and recovered (94%) as pure solvent.

Typical procedure for Hiyama reaction.

In a 4 mL screw-capped vial equipped with a magnetic stirrer, the aryl halide **1** (1mmol), the silane **6** (1.5 mmol), GVL:H_2O (4:1, 1 mL), TBAF (1.5 equiv, 488 mg) and **Pd/PiNe** (0.5 mol%, 5.3 mg) were consecutively added, and the resulting mixture was left under stirring at 130 °C. After 20 h, the catalyst was filtered off and washed with 1 mL of GVL. The product was precipitated with 1 mL of water at 0°C. The precipitate was filtered off, washed with 1 mL of cold water, and purified by column chromatography (petroleum ether:EtOAc, 95:5) to afford product **7**.

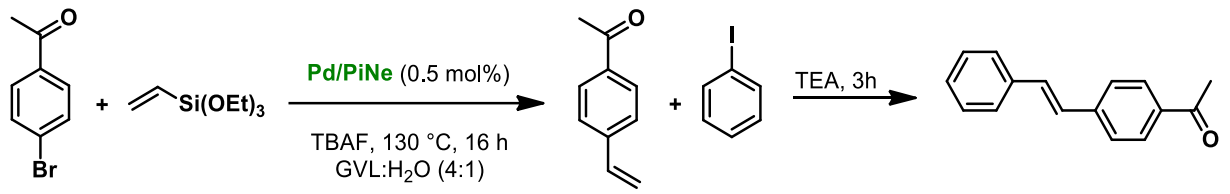
Typical procedure for consecutive Hiyama-Heck reaction.

In a 4 mL screw-capped vial equipped with a magnetic stirrer, the aryl halide **1** (1mmol), the silane **6c** (1.5 mmol), GVL (0.8 mL), aq. t-Bu-phenol (7µM, 0.2 mL) TBAF (1.5 equiv, 488 mg) and **Pd/PiNe** (0.5 mol%, 5.3 mg) were consecutively added, and the resulting mixture was left under stirring at 130 °C. After 16 h, aryl iodide **1a** (1.1 eq) was added and the mixture was kept under stirring at 130°C. After 3 h, the catalyst was filtered off and washed with 1 mL of GVL. The crude was precipitated with 1 mL of water at 0°C and pure product purified by column chromatography (petroleum ether:EtOAc, 95:5).

General procedure for leaching determination

The crude reaction mixture, after separation from the catalyst/ base system was dried under vacuum, dissolved in 2 mL of aqua regia, and stirred for 1h at room temperature. The mixture was transferred in a 10 mL graduated flask and Milli-Q water was added to reach the final volume. If present, residual solid was filtered off and the sample was analyzed by MP-AES 4210 instrument.

Table ESI-1. Optimization of consecutive Hiyama-Heck reaction with **Pd/PiNe** catalyst.



Entry	6c (eq)	TBAF (eq)	1a (eq)	Conv (%)	Sel (%)
1	1.5	1.5	1.1	87	49
2	1	1.1	1.2	74	59
3[a]	1	1.25	1.2	79	68
4[a]	1	1.5	1.5	>99	58
5[a,b]	1	1.5	1.5	84	77

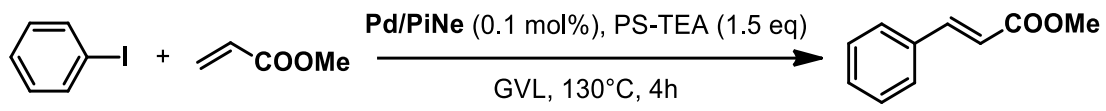
Reaction conditions: **1f** (1 mmol), **6c**, **Pd/PiNe** (0.5 mol%), TBAF, GVL:H_2O (4:1), 130°C, 16 h. **1a**, TEA (1.5 eq), 130°C, 3h. [a]20% of 7µM aq solution of t-Bu phenol was used in GVL as reaction medium. [b]reaction performed without TEA.

References:

1. Varma, A. K.; Mondal. P.*;* J. Therm. Anal. Calorim, 2016, 124, 487-497
2. Xiao, S.; Gao, R.; Lu, Y.; Li, J.; Sun, Q.; *Carbohydrates Polymers*, **2015**, *119*, 202-209
3. Kiruba, V. S. A.; Dakshinamurthy, A.; Subramanian, P. S.; Selvakumar, P. M.; *Journal of Experimental Nanoscience*, **2013**, DOI: 10.1080/17458080.2013.848295
4. Li, H.; Sun, G.; Li, N.; Su, D.; Xin, Q.*; J. Phys. Chem*. C **2007**, *111*, 5605-5617

3. Characterization data

Chem.Name	Methyl cinnamate (**3aa**)
Lit.Ref	F. Valentini, H. Mahmoudi, L. A. Bivona, O. Piermatti, M. Bagherzadeh, L. Fusaro, C. Aprile, A. Marrocchi, L. Vaccaro, *ACS Sustainable Chem. Eng.* 2019, **7**, 6939–6946



1 a **2 a** **3 aa**

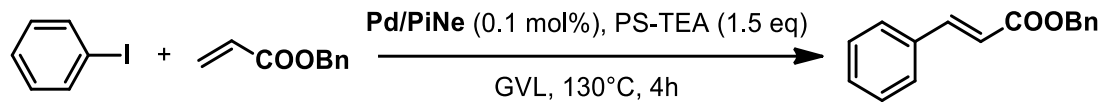
MW: 162,19 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer catalyst **Pd/PiNe** (9.9 wt%, 1.1 mg, 0.1 mmol%), PS-TEA (469 mg, 1.5 mmol), iodobenzene **1a** (114 µL, 1 mmol, 97% purity), methyl acrylate **2a** (110 µL, 1.2 mmol, 98% purity) and 1.25 mL of GVL were consecutively added and the resulting mixture was left under stirring at 130 °C for 4 h. After reaction completion the catalyst was removed through filtration and washed with 1 mL of GVL (94% recovered). The product **3aa** was isolated by precipitation work-up as white solid (149 mg, 92% yield).

Mol Formula	$C_{10}H_{10}O_2$	**m.p.**	36-38°C	
Elemental Analysis: Calc.: C: 74.06; H: 6.22; found: C: 74.08; H: 6.20.				
^{1}H NMR (400 MHz, $CDCl_3$)	δ value:	No. H	Mult	J value/Hz
	7.70	1	*d*	16.0
	7.54-7.51	2	*m*	
	7.40-7.37	3	*m*	
	6.45	1	*d*	16.0
	3.81	3	*s*	
^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 167.6, 145.0, 134.5, 130.4, 129.0, 128.2, 117.9, 51.8				
GC-EIMS (m/z, %): 162 (M+, 50), 161 (25), 131 (100), 103 (71), 102 (25), 77(39), 51 (22).				

Chem.Name	Benzyl cinnamate (**3ab**)
Lit.Ref	J. Tummatorn, P. A. Albiniak, G. B. Dudley *J. Org. Chem*. 2007, **72**, 8962–8964



1a **2b** **3ab**

MW: 238,28 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer catalyst **Pd/PiNe** (9.9 wt%, 1.1 mg, 0.1 mmol%), PS-TEA (469 mg, 1.5 mmol), iodobenzene **1a** (114 µL, 1 mmol, 97% purity), benzyl acrylate **2b** (189 µL, 1.2 mmol, 98% purity) and 1.25 mL of GVL were consecutively added and the resulting mixture was left under stirring at 130 °C for 4 h. After reaction completion the catalyst was removed through filtration and washed with 1 mL of GVL (94% recovered). The product **3ab** was isolated by extraction work-up as colourless oil (235 mg, 99% yield).

Mol Formula	$C_{16}H_{14}O_2$	**m.p.**	oil

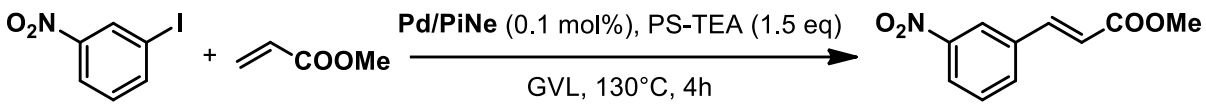
Elemental Analysis: Calc.: C: 80.65; H: 5.92; found: C: 80.63; H: 5.93.

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	7.74	1	*d*	16.0
	7.54-7.45	2	*m*	
	7.45-7.35	8	*m*	
	6.40	1	*d*	16.0
	5.27	2	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 166.9, 145.3, 136.2, 134.5, 129.0, 128.7, 128.4, 128.4, 128.2, 118.0, 66.5.

GC-EIMS (m/z, %): 238 (m+, 30), 193 (66), 192 (100), 178 (11), 132 (11), 131 (84), 115 (18), 104 (15), 103 (47), 91 (92), 77 (51), 65 (20), 51 (22)

Chem.Name	(E)-methyl 3-(3-nitrophenyl)acrylate (**3ba**)
Lit.Ref	J. Ruan, X. Li, O. Saidi, J. Xiao. *J. Am. Chem. Soc*. 2008, **130**, 2424–2425



1b **2a** **3ba**

MW: 207,05 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer catalyst **Pd/PiNe** (9.9 wt%, 1.1 mg, 0.1 mmol%), PS-TEA (469 mg, 1.5 mmol), 1-iodo-3-nitrobenzene **1b** (251 mg, 1 mmol, 99% purity), methyl acrylate **2a** (110 µL, 1.2 mmol, 98% purity) and 1.25 mL of GVL were consecutively added and the resulting mixture was left under stirring at 130 °C for 4 h. After reaction completion the catalyst was removed through filtration and washed with 1 mL of GVL (94% recovered). The product **3ba** was isolated by precipitation work-up as pale-yellow solid (186 mg, 90% yield).

Mol Formula	$C_{10}H_9NO_4$	**m.p.**	122-124 °C

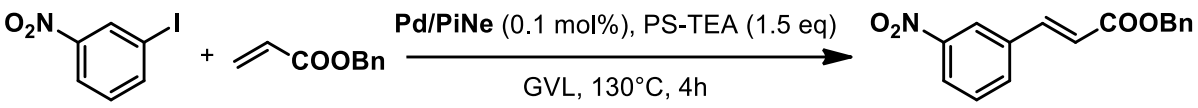
Elemental Analysis: Calc.: C: 57.97; H: 4.38; N: 6.76; found: C: 57.99; H: 4.37; N: 6.78;

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	8.38	1	*s*	
	8.24	1	*dd*	8.4 / 1.2
	7.82	1	*d*	8.0
	7.73	1	*d*	16.0
	7.59	1	*t*	8.0
	7.56	1	*d*	16.0
	3.84	3	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 166.7, 148.9, 142.1, 136.3, 133.8, 130.1, 124.7, 122.6, 121.1, 52.2.

GC-EIMS (m/z, %): 207 (M+, 47), 190 (34), 177 (11), 176 (100), 130 (15), 129 (21), 118 (25), 102 (49), 75 (15), 51 (12), 50 (13).

Chem.Name	(E)-benzyl 3-(3-nitrophenyl)acrylate (**3bb**)
Lit.Ref	A. R. Katritzky, S. Zhang, A. Soares, M. Wang, A facile synthesis of benzyl-α, β-unsaturated carboxylic esters. ARKIVOC, 2001, **10**, 54-57.



1b **2b** **3bb**

MW: 283, 28 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer catalyst **Pd/PiNe** (9.9 wt%, 1.1 mg, 0.1 mmol%), PS-TEA (469 mg, 1.5 mmol), 1-iodo-3-nitrobenzene **1b** (251 mg, 1 mmol, 99% purity), benzyl acrylate **2b** (189 µL, 1.2 mmol, 98% purity) and 1.25 mL of GVL were consecutively added and the resulting mixture was left under stirring at 130 °C for 4 h. After reaction completion the catalyst was removed through filtration and washed with 1 mL of GVL (94% recovered). The product **3bb** was isolated by precipitation work-up as plae yellow solid (266 mg, 94% yield).

Mol Formula	$C_{16}H_{13}NO_4$	**m.p.**	68-69 °C

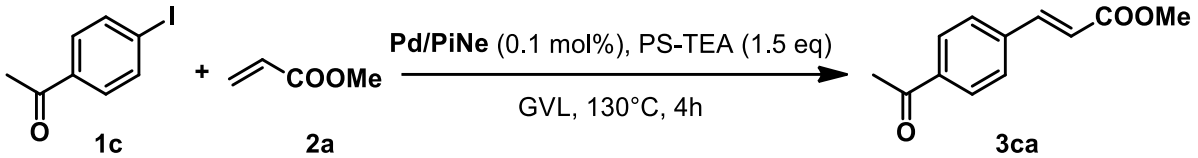
Elemental Analysis: Calc.: C: 67.84; H: 4.63; N: 4.94; found: C: 67.85; H: 4.62; N: 4.95

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	8.38	1	*s*	
	8.23	1	*d*	8.0
	7.82	1	*d*	8.0
	7.76	1	*d*	16.0
	7.58	1	*t*	8.0
	7.42-7.36	5	*m*	
	6.61	1	*d*	16.0
	5.28	2	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 166.1, 148.9, 142.4, 136.2, 135.8, 133.8, 130.1, 128.8, 128.6, 128.5, 124.8, 122.6, 121.3, 66.9.

GC-EIMS (m/z, %): 283 (M+, 6), 267 (14), 268 (80), 238 (46), 237 (18), 222 (11), 221 (13), 220 (13), 191 (16), 176 (61), 130 (14), 129 (11), 102 (38), 91 (100), 79 (11), 77 (16), 76 (17), 65 (16)

Chem.Name	(E)-methyl 3-(4-acetylphenyl)acrylate (**3ca**)
Lit.Ref	F. Valentini, H. Mahmoudi, L. A. Bivona, O. Piermatti, M. Bagherzadeh, L. Fusaro, C. Aprile, A. Marrocchi, L. Vaccaro, *ACS Sustainable Chem. Eng.* 2019, **7**, 6939–6946



MW: 204,08 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer catalyst **Pd/PiNe** (9.9 wt%, 1.1 mg, 0.1 mmol%), PS-TEA (469 mg, 1.5 mmol), 4-iodoacetophenone **1c** (249 mg, 1 mmol, 99% purity), methyl acrylate **2a** (110 µL, 1.2 mmol, 98% purity) and 1.25 mL of GVL were consecutively added and the resulting mixture was left under stirring at 130 °C for 4 h. After reaction completion the catalyst was removed through filtration and washed with 1 mL of GVL (94% recovered). The product **3ca** was isolated by precipitation work-up as pale yellow solid (191 mg, 94% yield).

Mol Formula	$C_{12}H_{12}O_3$	**m.p.**	104-106°C

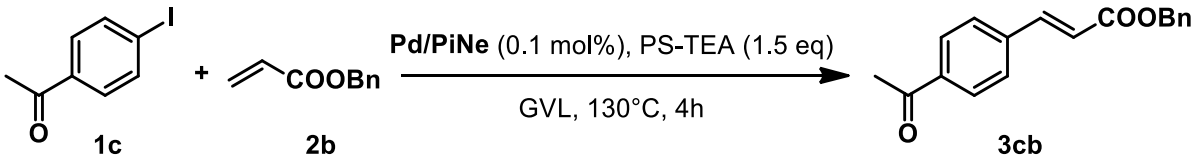
Elemental Analysis: Calc.: C: 70.58; H: 5.92; found: C: 70.61; H: 5.93.

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	7.97	2	*d*	8.0
	7.71	1	*d*	16.0
	7.61	2	*d*	8.0
	6.53	1	*d*	16.0
	3.83	3	*s*	
	2.62	3	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 197.5, 167.1, 143.5, 138.9, 138.2, 129.0, 128.3, 120.5, 52.1, 26.8

GC-EIMS (m/z, %): 205 (22), 204 (M^+, 100), 190 (34), 189 (81), 188 (24), 173 (24), 161 (62),131 (37), 130 (16), 129 (27), 118 (26), 103 (20), 102 (72), 101 (18), 76 (31), 75 (24), 74 (16), 51 (26), 50 (19).

Chem.Name	(E)-benzyl 3-(4-acetylphenyl)acrylate (**3cb**)
Lit.Ref	T. Ichikawa, M. Mizuno, S. Ueda, N. Ohneda, H. Odajima, Y. Sawama, Y. Monguchia, H. Sajiki *Tetrahedron*, 2018, **74**, 1810-1816



MW: 280,32 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer catalyst **Pd/PiNe** (9.9 wt%, 1.1 mg, 0.1 mmol%), PS-TEA (469 mg, 1.5 mmol), 4-iodoacetophenone **1c** (249 mg, 1 mmol, 99% purity), benzyl acrylate **2b** (189 µL, 1.2 mmol, 98% purity) and 1.25 mL of GVL were consecutively added and the resulting mixture was left under stirring at 130 °C for 4 h. After reaction completion the catalyst was removed through filtration and washed with 1 mL of GVL (94% recovered). The product **3cb** was isolated by extraction work-up as white solid (277 mg, 98% yield).

Mol Formula	$C_{18}H_{16}O_3$	**m.p.**	

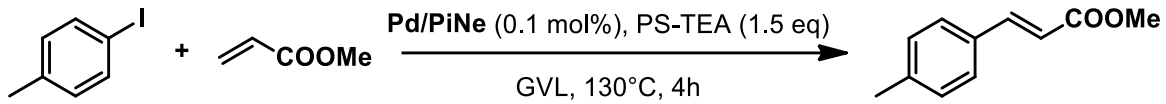
Elemental Analysis: Calc.: C: 77.12; H: 5.75; found: C: 77.13; H: 5.75

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	7.97	2	*d*	8.0
	7.73	1	*d*	16.0
	7.60	2	*d*	8.0
	7.44-7.35	5	*m*	
	6.57	1	*d*	16.0
	5.27	2	*s*	
	2.61	3	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 197.4, 166.4, 143.7, 138.8, 138.2, 136.0, 129.0, 128.8, 128.5, 128.5, 128.3, 120.6, 66.8, 26.8.

GC-EIMS (m/z, %): 281 (20), 280 (M+, 100), 265 (37), 237 (27), 236 (12), 235 (57), 234 (59), 219 (15), 193 (17), 197 (14), 191 (22), 173 (50), 115 (13), 91 (94), 77 (13), 65 (13)

Chem.Name	(E)-methyl 3-(p-tolyl)acrylate (**3da**)
Lit.Ref	F. Valentini, H. Mahmoudi, L. A. Bivona, O. Piermatti, M. Bagherzadeh, L. Fusaro, C. Aprile, A. Marrocchi, L. Vaccaro, *ACS Sustainable Chem. Eng.* 2019, **7**, 6939–6946



1d **2a** **3da**

MW: 176,21 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer catalyst **Pd/PiNe** (9.9 wt%, 1.1 mg, 0.1 mmol%), PS-TEA (469 mg, 1.5 mmol), 4-iodotoluene **1d** (220 mg, 1 mmol, 99% purity), methyl acrylate **2a** (110 µL, 1.2 mmol, 98% purity) and 1.25 mL of GVL were consecutively added and the resulting mixture was left under stirring at 130 °C for 4 h. After reaction completion the catalyst was removed through filtration and washed with 1 mL of GVL (94% recovered). The product **3da** was isolated by precipitation work-up as white solid (162 mg, 92% yield).

Mol Formula	$C_{11}H_{12}O_2$	**m.p.**	54-56 °C

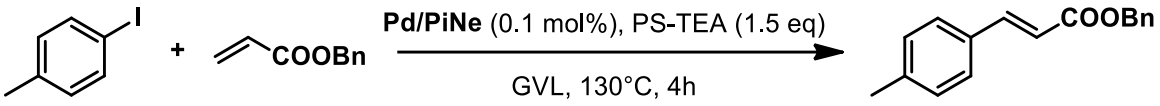
Elemental Analysis: Calc.: C: 74.98; H: 6.86; found: C: 75.01; H: 6.89.

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	7.67	1	*d*	16.0
	7.42	2	*d*	8.0
	7.19	2	*d*	8.0
	6.39	1	*d*	16.0
	3.80	3	*s*	
	2.37	3	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 167.8, 145.0, 140.9, 131.8, 129.8, 128.2, 116.8, 51.8, 21.6.

GC-EIMS (m/z, %): 176 (M^+, 100), 175 (56), 161 (22), 146 (40), 145 (100), 131 (16), 118 (25), 117 (100), 116 (56), 115 (100), 102 (17), 91 (67), 89 (25), 65 (32), 63 (26), 57 (16), 51 (16).

Chem.Name	(E)-benzyl 3-(p-tolyl)acrylate (**3db**)
Lit.Ref	J. Liu, Y. Wang, Y. Yue, N. Liu, J. Zhang, S. Zhao, Q. Tang, K. Zhuo, *Eur. J. Org. Chem*., 2017, 2641-2647.



1d **2b** **3db**

MW: 252,31 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer catalyst **Pd/PiNe** (9.9 wt%, 1.1 mg, 0.1 mmol%), PS-TEA (469 mg, 1.5 mmol), 4-iodotoluene **1d** (220 mg, 1 mmol, 99% purity), benzyl acrylate **2b** (189 µL, 1.2 mmol, 98% purity) and 1.25 mL of GVL were consecutively added and the resulting mixture was left under stirring at 130 °C for 4 h. After reaction completion the catalyst was removed through filtration and washed with 1 mL of GVL (94% recovered). The product **3db** was isolated by precipitation work-up as white solid (237 mg, 94% yield).

Mol Formula	$C_{17}H_{16}O_2$	**m.p.**	

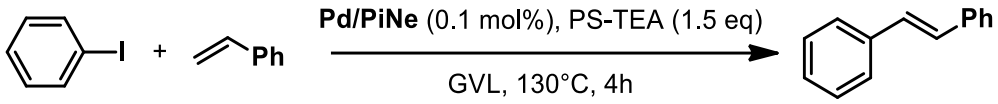
Elemental Analysis: Calc.: C: 80.93; H: 6.39; found: C: 80.95; H: 6.38

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	7.71	1	*d*	16
	7.43-7.36	4	*m*	
	7.35-7.34	2	*m*	
	7.19	2	*d*	8
	6.44	1	*d*	16
	5.25	2	*s*	
	2.37	3	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 167.2, 145.4, 140.1, 136.3, 131.8, 129.8, 128.7, 128.4, 128.4, 128.3, 116.9, 66.4, 21.6.

GC-EIMS (m/z, %): 253 (11), 252 (M+, 55), 234 (11), 207 (54), 206 (62), 192 (11), 145 (65), 118 (20), 117 (27), 116 (15), 115 (52), 91 (100), 89 (13), 65 (27).

Chem.Name	(E)-1,2-diphenylethene (**5a**)
Lit.Ref	F. Valentini, H. Mahmoudi, L. A. Bivona, O. Piermatti, M. Bagherzadeh, L. Fusaro, C. Aprile, A. Marrocchi, L. Vaccaro, *ACS Sustainable Chem. Eng.* 2019, **7**, 6939–6946



1a **4** **5a**

MW: 180.25 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer catalyst **Pd/PiNe** (9.9 wt%, 1.1 mg, 0.1 mmol%), PS-TEA (469 mg, 1.5 mmol), iodobenzene **1a** (114 µL, 1 mmol, 97% purity), styrene **4** (128 µL, 1.2 mmol, 98% purity) and 1.25 mL of GVL were consecutively added and the resulting mixture was left under stirring at 130 °C for 4 h. After reaction completion the catalyst was removed through filtration and washed with 1 mL of GVL (94% recovered). The product **3ab** was isolated by precipitation work-up as white solid (159 mg, 88% yield).

Mol Formula	$C_{14}H_{12}$	**m.p.**	126-128 °C

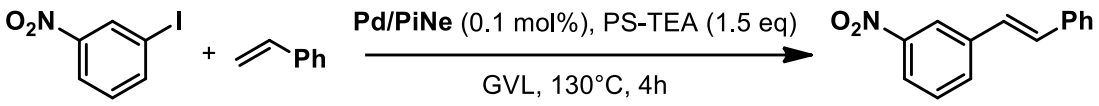
Elemental Analysis: Calc.: C: 93.29; H: 6.71; found: C: 93.32; H: 6.72.

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	7.54	4	*d*	8.0
	7.38	4	*t*	8.0
	7.30-7.26	2	*m*	
	7.14	2	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 137.5, 128.8, 127.8, 126.7.

GC-EIMS (m/z, %): 181 (40), 180 (M+, 100), 179 (100), 178 (91), 177 (24), 176 (27), 166 (19), 165 (86), 152 (35), 151 (17), 102 (23), 89 (46), 77 (19), 76 (33), 63 (18), 51 (23).

Chem.Name	(E)-1-nitro-3-styrylbenzene (**5b**)
Lit.Ref	A. Maji, O. Singh, S. Singh, A. Mohanty, P.K. Maji, K. Ghosh, *Eur. J. Inorg. Chem*., 2020, 1596-1611



1b **4** **5b**

MW: 225,24 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer catalyst **Pd/PiNe** (9.9 wt%, 1.1 mg, 0.1 mmol%), PS-TEA (469 mg, 1.5 mmol), 1-iodo-3-nitrobenzene **1b** (251 mg, 1 mmol, 99% purity), styrene **4** (128 µL, 1.2 mmol, 98% purity) and 1.25 mL of GVL were consecutively added and the resulting mixture was left under stirring at 130 °C for 4 h. After reaction completion the catalyst was removed through filtration and washed with 1 mL of GVL (94% recovered). The product **5b** was isolated by precipitation work-up as pale yellow solid (171 mg, 76% yield).

Mol Formula	$C_{14}H_{11}NO_2$	**m.p.**	106-107 °C

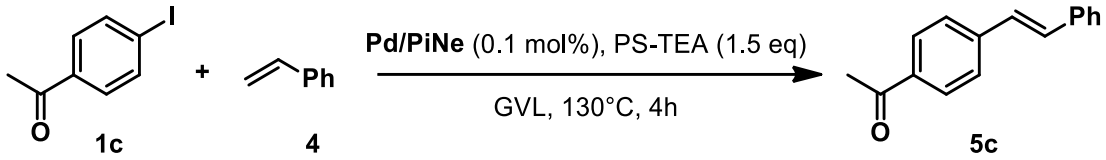
Elemental Analysis: Calc.: C, 74.65; H, 4.92; N, 6.22; found: C: 74.67; H: 4.91; N: 6.21

^{1}H NMR (400 MHz, $CDCl_3$)	δ value:	No. H	Mult	J value/Hz
	8.38	1	*s*	
	8.11	1	*d*	7.6
	7.80	1	*d*	7.6
	7.56-7.51	3	*m*	
	7.41	2	*t*	7.6
	7.35-7.31	1	*m*	
	7.25	1	*d*	16.0
	7.14	1	*d*	16.0

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 148.9, 139.3, 136.4, 132.4, 131.9, 129.7, 129.0, 128.7, 127.0, 126.2, 122.2, 121.0.

GC-EIMS (m/z, %): 226 (13), 225 (M^+, 79), 180 (15), 179 (27), 178 (100), 176 (18), 165 (12), 152 (21)

Chem.Name	(E)-1-(4-styrylphenyl)ethanone (**5c**)
Lit.Ref	F. Valentini, H. Mahmoudi, L. A. Bivona, O. Piermatti, M. Bagherzadeh, L. Fusaro, C. Aprile, A. Marrocchi, L. Vaccaro, *ACS Sustainable Chem. Eng.* 2019, **7**, 6939–6946



MW: 222,28 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer catalyst **Pd/PiNe** (9.9 wt%, 1.1 mg, 0.1 mmol%), PS-TEA (469 mg, 1.5 mmol), 4-iodoacetophenone **1c** (249 mg, 1 mmol, 99% purity), styrene **4** (128 µL, 1.2 mmol, 98% purity) and 1.25 mL of GVL were consecutively added and the resulting mixture was left under stirring at 130 °C for 4 h. After reaction completion the catalyst was removed through filtration and washed with 1 mL of GVL (94% recovered). The product **5c** was isolated by precipitation work-up as pale yellow solid (211 mg, 95% yield).

Mol Formula	$C_{16}H_{14}O$	**m.p.**	140-142 °C

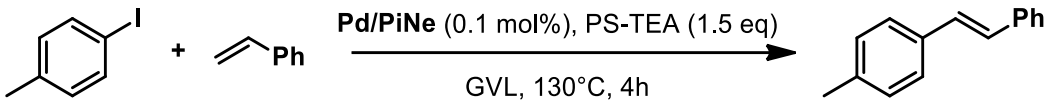
Elemental Analysis: Calc.: C: 86.45; H: 6.35; found: C: 86.42; H: 6.34.

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	7.96	2	*d*	8.4
	7.59	2	*d*	8.0
	7.54	2	*d*	7.6
	7.40-7.37	2	*m*	
	7.30	1	*t*	8
	7.24	1	*d*	16
	7.14	1	*d*	16
	2.61	3	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 197.6, 142.2, 136.8, 136.1, 131.6, 129.0, 128.9, 128.5, 127.6, 127.0, 126.7, 26.8.

GC-EIMS (m/z, %): 223 (27), 222 (M^+, 89), 208 (38), 207 (100), 179 (52), 178 (89), 177 (25), 176 (26), 152 (23), 89 (21).

Chem.Name	(E)-1-methyl-4-styrylbenzene (**5d**)
Lit.Ref	F. Valentini, H. Mahmoudi, L. A. Bivona, O. Piermatti, M. Bagherzadeh, L. Fusaro, C. Aprile, A. Marrocchi, L. Vaccaro, *ACS Sustainable Chem. Eng.* 2019, **7**, 6939–6946



1d **4** **5d**

MW: 194,27 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer catalyst **Pd/PiNe** (9.9 wt%, 1.1 mg, 0.1 mmol%), PS-TEA (469 mg, 1.5 mmol), 4-iodotoluene **1d** (220 mg, 1 mmol, 99% purity), styrene **4** (128 µL, 1.2 mmol, 98% purity) and 1.25 mL of GVL were consecutively added and the resulting mixture was left under stirring at 130 °C for 4 h. After reaction completion the catalyst was removed through filtration and washed with 1 mL of GVL (94% recovered). The product **5d** was isolated by precipitation work-up as white solid (171 mg, 88% yield).

Mol Formula	$C_{15}H_{14}$	**m.p.**	120-122 °C

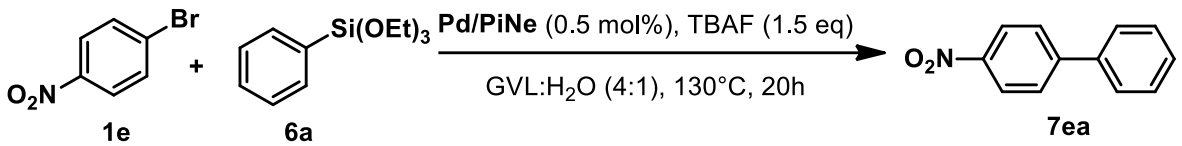
Elemental Analysis: Calc.: C: 92.74; H: 7.26; found: C: 92.71; H: 7.24.

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	7.51	2	*d*	7.6
	7.42	2	*d*	8.0
	7.35	2	*t*	7.6
	7.26-7.20	1	*m*	
	7.17	2	*d*	8.0
	7.10-7.06	2	*m*	
	2.36	3	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 137.7, 134.7, 129.5, 128.8, 127.9, 127.6, 126.6, 126.5, 21.4.

GC-EIMS (m/z, %): 195 (31), 194 (M^+, 97), 193 (46), 180 (29), 179 (100), 178 (94), 165 (27), 152 (19), 115 (26), 89 (17).

Chem.Name	4-nitro-1,1'-biphenyl (**7ea**)
Lit.Ref	E. Ismalaj, G. Strappaveccia, E. Ballerini, F. Elisei, O. Piermatti, D. Gelman, L. Vaccaro *ACS Sustainable Chem. Eng.***, 2014,** 2, 2461-2464



1e **6a** **7ea**

MW: 199,21 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer, the aryl halide **1e** (204 mg, 1mmol), triethoxy(phenyl) silane **6a** (368 mg, 0.370 mL 1.5 mmol), GVL: H_2O (4:1, 1 mL), TBAF (1.5 equiv, 488 mg) and **Pd/PiNe** (0.5 mol%, 5.3 mg) were consecutively added, and the resulting mixture was left under stirring at 130 °C. After 20 h, the catalyst was filtered off and washed with 1 mL of GVL. The product was precipitated with 1 mL of water at 0°C. The precipitate was filtered off, washed with 1 mL of cold water, and purified by column chromatography (petroleum ether:EtOAc, 95:5) to afford product **7ea** as white solid (162 mg, 81% yield).

Mol Formula	$C_{12}H_9NO_2$	**m.p.**	116-117°C

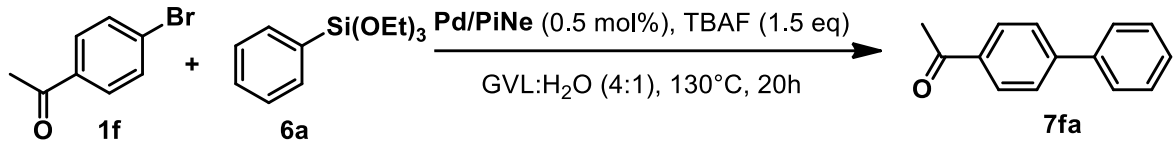
Elemental Analysis: Calc.: C: 72.35; H: 4.55; N: 7.03. found: C, 72.40; H: 4.59; N: 7.01

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	8.30	2	*d*	8.0
	7.74	2	*d*	8.0
	7.63	2	*d*	8.0
	7.52-7.43	3	*m*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 147.8, 147.2, 138.9, 129.2, 129.0, 127.9, 127.5, 124.2.

GC-EIMS (m/z, %): 199 (M^+, 83), 169 (44), 153 (31), 152 (100), 151 (33), 150 (11), 141 (40), 127 (11), 126 (11), 115 (19), 77 (12), 76 (14), 63 (13), 51 (12)

Chem.Name	1-([1,1'-biphenyl]-4-yl)ethanone (**7fa**)
Lit.Ref	E. Ismalaj, G. Strappaveccia, E. Ballerini, F. Elisei, O. Piermatti, D. Gelman, L. Vaccaro *ACS Sustainable Chem. Eng.***, 2014,** 2, 2461-2464



MW: 196,24 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer, the aryl halide **1f** (203 mg, 1mmol), triethoxy(phenyl) silane **6a** (368 mg, 0.370 mL 1.5 mmol), GVL: H_2O (4:1, 1 mL), TBAF (1.5 equiv, 488 mg) and **Pd/PiNe** (0.5 mol%, 5.3 mg) were consecutively added, and the resulting mixture was left under stirring at 130 °C. After 20 h, the catalyst was filtered off and washed with 1 mL of GVL. The product was precipitated with 1 mL of water at 0°C. The precipitate was filtered off, washed with 1 mL of cold water, and purified by column chromatography (petroleum ether:EtOAc, 95:5) to afford product **7fa** as white solid (164 mg, 84% yield).

Mol Formula	$C_{14}H_{12}O$	**m.p.**	115-117 °C

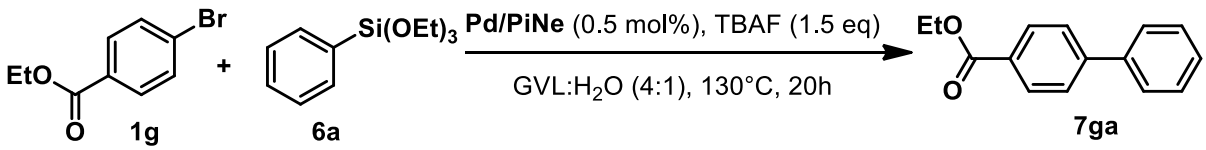
Elemental Analysis: Calc.: C: 85.68; H: 6.16. found: C: 85.78; H: 6.13

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	8.04	2	d	8.0
	7.69	2	d	8.0
	7.65-7.62	2	m	
	7.48	2	t	8.0
	7.42-7.38	1	m	
	2.64	3	s	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 197.9, 145.9, 140.0, 136.0, 129.1, 129.0, 128.4, 127.4, 127.3, 26.8

GC-EIMS (m/z, %): 196 (M^+, 48), 182 (14), 181 (100), 153 (42), 152 (69), 151 (21), 76 (11), 43 (12)

Chem.Name	ethyl [1,1'-biphenyl]-4-carboxylate (**7ga**)
Lit.Ref	Q. Cao, J. L. Howard, E. Wheatley, D. L. Browne, *Angew. Chem. Int. Ed.,* 2018, **57**, 11339 -11343



MW: 226,27 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer, the aryl halide **1g** (234 mg, 0.167 mL, 1mmol), triethoxy(phenyl) silane **6a** (368 mg, 0.370 mL 1.5 mmol), GVL: H_2O (4:1, 1 mL), TBAF (1.5 equiv, 488 mg) and **Pd/PiNe** (0.5 mol%, 5.3 mg) were consecutively added, and the resulting mixture was left under stirring at 130 °C. After 20 h, the catalyst was filtered off and washed with 1 mL of GVL. The product was precipitated with 1 mL of water at 0°C. The precipitate was filtered off, washed with 1 mL of cold water, and purified by column chromatography (petroleum ether:EtOAc, 95:5) to afford product **7ga** as pale yellow solid (188 mg, 83% yield).

Mol Formula	$C_{15}H_{14}O_2$	**m.p.**	93-94°C

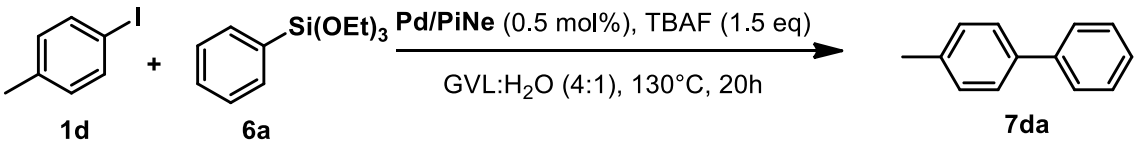
Elemental Analysis: C: 79.62; H: 6.24; found: C: 79.66; H: 6.20

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	8.12	2	*d*	8.0
	7.66	2	*d*	8.0
	7.63	2	*d*	8.0
	7.47	2	*t*	8.0
	7.41-7.38	1	*m*	
	4.41	2	*q*	8.0
	1.42	3	*t*	8.0

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 166.7, 145.7, 140.2, 130.2, 129.4, 129.1, 128.2 ,127.4, 127.1, 61.1, 14.5

GC-EIMS (m/z, %): 226 (M+, 65), 198 (34), 182 (18), 181 (100), 153 (30), 152 (51), 76 (12)

Chem.Name	4-methyl-1,1'-biphenyl (**7da**)
Lit.Ref	E. Ismalaj, G. Strappaveccia, E. Ballerini, F. Elisei, O. Piermatti, D. Gelman, L. Vaccaro *ACS Sustainable Chem. Eng.***, 2014,** 2, 2461-2464



1d **6a** **7da**

MW: 168,23 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer, the aryl halide **1d** (220 mg, 1mmol), triethoxy(phenyl) silane **6a** (368 mg, 0.370 mL 1.5 mmol), GVL: H_2O (4:1, 1 mL), TBAF (1.5 equiv, 488 mg) and **Pd/PiNe** (0.5 mol%, 5.3 mg) were consecutively added, and the resulting mixture was left under stirring at 130 °C. After 20 h, the catalyst was filtered off and washed with 1 mL of GVL. The product was precipitated with 1 mL of water at 0°C. The precipitate was filtered off, washed with 1 mL of cold water, and purified by column chromatography (petroleum ether:EtOAc, 95:5) to afford product **7da** as white solid (130 mg, 77% yield).

Mol Formula	$C_{13}H_{12}$	**m.p.**	45-47 °C

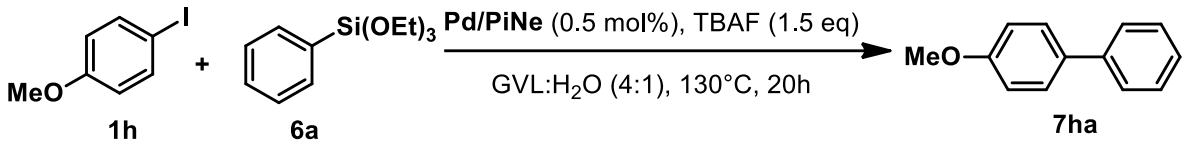
Elemental Analysis: Calc.: C: 92.81; H: 7.19. found: C, 92.76; H: 7.23

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	7.61	2	*d*	8.0
	7.52	2	*d*	8.0
	7.45	2	*t*	8.0
	7.35	1	*t*	8.0
	7.29-7.27	2	*m*	
	2.43	3	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 141.3, 138.5, 137.1, 129.6, 128.9, 127.1, 127.1, 127.1, 21.2

GC-EIMS (m/z, %): 169 (14), 168 (M^+, 100), 167 (66), 165 (28), 153 (15), 152 (21), 115 (11)

Chem.Name	4-methoxy-1,1'-biphenyl (**7ha**)
Lit.Ref	E. Ismalaj, G. Strappaveccia, E. Ballerini, F. Elisei, O. Piermatti, D. Gelman, L. Vaccaro *ACS Sustainable Chem. Eng.***, 2014,** 2, 2461-2464



1h **6a** **7ha**

MW: 184,23 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer, the aryl halide **1h** (234 mg, 1mmol), triethoxy(phenyl) silane **6a** (368 mg, 0.370 mL 1.5 mmol), GVL: H_2O (4:1, 1 mL), TBAF (1.5 equiv, 488 mg) and **Pd/PiNe** (0.5 mol%, 5.3 mg) were consecutively added, and the resulting mixture was left under stirring at 130 °C. After 20 h, the catalyst was filtered off and washed with 1 mL of GVL. The product was precipitated with 1 mL of water at 0°C. The precipitate was filtered off, washed with 1 mL of cold water, and purified by column chromatography (petroleum ether) to afford product **7ha** as yellow solid (118 mg, 64% yield).

Mol Formula	$C_{13}H_{12}O$	**m.p.**	87-89 °C

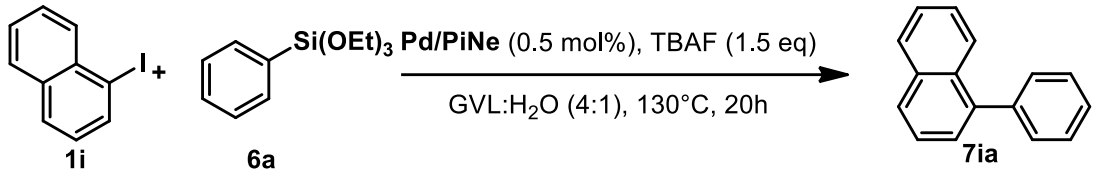
Elemental Analysis: Calc.: C: 84.75; H: 6.57. found: C: 84.77; H: 6.55

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	7.55	4	*t*	8.0
	7.42	2	*t*	8.0
	7.33-7.26	1	*m*	
	6.99	2	*d*	8.0
	3.86	3	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 159.3, 141.0, 133.9, 128.9, 128.3, 126.9, 126.8, 114.3, 55.5

GC-EIMS (m/z, %): 185 (14), 184 (M^+, 100), 169 (51), 141 (61), 139 (17), 115 (53)

Chem.Name	1-phenylnaphthalene (**7ia**)
Lit.Ref	E. Ismalaj, G. Strappaveccia, E. Ballerini, F. Elisei, O. Piermatti, D. Gelman, L. Vaccaro *ACS Sustainable Chem. Eng.***, 2014,** 2, 2461-2464



1i **6a** **7ia**

MW: 204,27 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer, the aryl halide **1i** (254 mg, 1mmol), triethoxy(phenyl) silane **6a** (368 mg, 0.370 mL 1.5 mmol), GVL: H_2O (4:1, 1 mL), TBAF (1.5 equiv, 488 mg) and **Pd/PiNe** (0.5 mol%, 5.3 mg) were consecutively added, and the resulting mixture was left under stirring at 130 °C. After 20 h, the catalyst was filtered off and washed with 1 mL of GVL. The product was precipitated with 1 mL of water at 0°C. The precipitate was filtered off, washed with 1 mL of cold water, and purified by column chromatography (petroleum ether) to afford product **7ia** as colourless oil (153 mg, 75% yield).

Mol Formula	$C_{16}H_{12}$	**m.p.**	44-46 °C

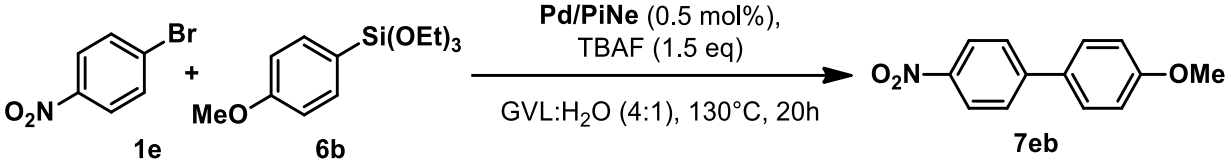
Elemental Analysis: Calc.: C: 94.08; H: 5.92. found: C: 94.16; H: 5.89

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	7.91	2	*d*	8
	7.87	1	*d*	8
	7.55-7.48	6	*m*	
	7.46-7.41	3	*m*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 140.9, 140.4, 133.9, 131.8, 130.2, 128.4, 127.8, 127.4, 127.1, 126.2, 126.1, 125.9, 125.5.

GC-EIMS (m/z, %): 204 (M^+, 100), 203 (98), 202 (61), 201 (13), 200 (13), 101 (16)

Chem.Name	4-methoxy-4'-nitro-1,1'-biphenyl (**7eb**)
Lit.Ref	E. Ismalaj, G. Strappaveccia, E. Ballerini, F. Elisei, O. Piermatti, D. Gelman, L. Vaccaro *ACS Sustainable Chem. Eng.***, 2014,** 2, 2461-2464



1e **6b** **7eb**

MW: 229,23 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer, the aryl halide **1e** (204 mg, 1mmol), triethoxy(4-methoxyphenyl) silane **6b** (418 mg, 0.406 mL, 1.5 mmol), GVL: H_2O (4:1, 1 mL), TBAF (1.5 equiv, 488 mg) and **Pd/PiNe** (0.5 mol%, 5.3 mg) were consecutively added, and the resulting mixture was left under stirring at 130 °C. After 20 h, the catalyst was filtered off and washed with 1 mL of GVL. The product was precipitated with 1 mL of water at 0°C. The precipitate was filtered off, washed with 1 mL of cold water, and purified by column chromatography (petroleum ether:EtOAc, 95:5) to afford product **7eb** as pale yellow solid (209 mg, 91% yield).

Mol Formula	$C_{13}H_{11}NO_3$	**m.p.**	110-112 °C

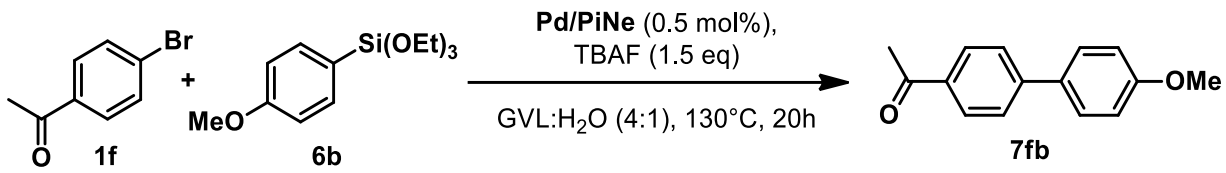
Elemental Analysis: Calc.: C: 68.11; H: 4.84; N: 6.11. found: C: 68.21; H: 4.90; N: 6.05

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	8.27	2	*d*	8.0
	7.69	2	*d*	8.0
	7.58	2	*d*	8.0
	7.02	2	*d*	8.0
	3.88	3	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 160.6, 147.4, 146.7, 131.2, 128.7, 127.2, 124.3, 114.8, 55.6

GC-EIMS (m/z, %): 230 (14), 229 (M^+, 100), 199 (25), 183 (11), 171 (18), 168 (24), 152 (18), 140 (31), 139 (57), 128 (13)

Chem.Name	1-(4'-methoxy-[1,1'-biphenyl]-4-yl)ethanone (**7fb**)
Lit.Ref	E. Ismalaj, G. Strappaveccia, E. Ballerini, F. Elisei, O. Piermatti, D. Gelman, L. Vaccaro *ACS Sustainable Chem. Eng.***, 2014,** 2, 2461-2464



MW: 226,27 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer, the aryl halide **1f** (203 mg, 1mmol), triethoxy(4-methoxyphenyl) silane **6b** (418 mg, 0.406 mL, 1.5 mmol), GVL: H_2O (4:1, 1 mL), TBAF (1.5 equiv, 488 mg) and **Pd/PiNe** (0.5 mol%, 5.3 mg) were consecutively added, and the resulting mixture was left under stirring at 130 °C. After 20 h, the catalyst was filtered off and washed with 1 mL of GVL. The product was precipitated with 1 mL of water at 0°C. The precipitate was filtered off, washed with 1 mL of cold water, and purified by filtration on silica pad with toluene to afford product **7fb** as white solid (168 mg, 74% yield).

Mol Formula	$C_{15}H_{14}O_2$	**m.p.**	153-156 °C

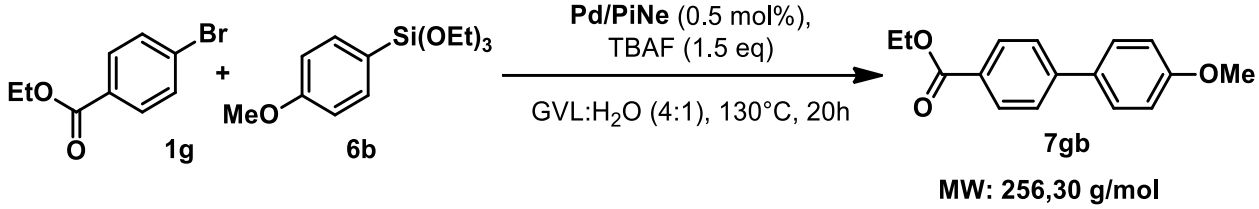
Elemental Analysis: Calc.: C: 79.62; H: 6.24. Found: C. 79.69; H: 6.21

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	8.01	2	*d*	8.0
	7.65	2	*d*	8.0
	7.58	2	*d*	8.0
	7.00	2	*d*	8.0
	3.87	3	*s*	
	2.63	3	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 197.9, 160.1, 145.5, 135.4, 132.4, 129.1, 128.5, 126.8, 114.6, 55.5, 26.8

GC-EIMS (m/z, %): 227 (10), 226 (M^+, 55), 212 (12), 211 (100), 183 (13), 168 (20), 152 (12), 140 (29), 139 (49), 63 (11), 43 (18)

Chem.Name	ethyl 4'-methoxy-[1,1'-biphenyl]-4-carboxylate (**7gb**)
Lit.Ref	Q. Cao, J. L. Howard, E. Wheatley, D. L. Browne, *Angew. Chem. Int. Ed.,* 2018, **57**, 11339 -11343



MW: 256,30 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer, the aryl halide **1g** (234 mg, 1mmol), triethoxy(4-methoxyphenyl) silane **6b** (418 mg, 0.406 mL, 1.5 mmol), GVL: H_2O (4:1, 1 mL), TBAF (1.5 equiv, 488 mg) and **Pd/PiNe** (0.5 mol%, 5.3 mg) were consecutively added, and the resulting mixture was left under stirring at 130 °C. After 20 h, the catalyst was filtered off and washed with 1 mL of GVL. The product was precipitated with 1 mL of water at 0°C. The precipitate was filtered off, washed with 1 mL of cold water, and purified by filtration on silica pad with toluene to afford product **7gb** as white solid (226 mg, 88% yield).

Mol Formula	$C_{16}H_{16}O_3$	**m.p.**	100-103 °C

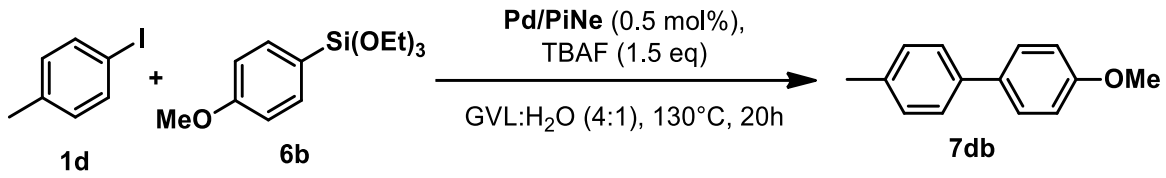
Elemental Analysis: Calc.: C: 74.98; H: 6.29; found: C: 75.01; H: 6.30

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	8.08	2	*d*	8.4
	7.62	2	*d*	8.4
	7.57	2	*d*	8.8
	7.00	2	*d*	8.8
	4.40	2	*q*	7.2
	3.86	3	*s*	
	1.41	3	*t*	7.2

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 166.8, 160.0, 145.4, 132.6, 130.2, 128.8, 128.5, 126.6, 114.5, 61.1, 55.5, 14.5

GC-EIMS (m/z, %): 257 (18), 256 (M+, 100), 212 (13), 211 (64), 168 (13), 140 (14), 139 (22)

Chem.Name	4-methoxy-4'-methyl-1,1'-biphenyl (**7db**)
Lit.Ref	E. Ismalaj, G. Strappaveccia, E. Ballerini, F. Elisei, O. Piermatti, D. Gelman, L. Vaccaro *ACS Sustainable Chem. Eng.***, 2014,** 2, 2461-2464



1d **6b** **7db**

MW: 198,26 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer, the aryl halide **1d** (220 mg, 1mmol), triethoxy(4-methoxyphenyl) silane **6b** (418 mg, 0.406 mL, 1.5 mmol), GVL: H_2O (4:1, 1 mL), TBAF (1.5 equiv, 488 mg) and **Pd/PiNe** (0.5 mol%, 5.3 mg) were consecutively added, and the resulting mixture was left under stirring at 130 °C. After 20 h, the catalyst was filtered off and washed with 1 mL of GVL. The product was precipitated with 1 mL of water at 0°C. The precipitate was filtered off, washed with 1 mL of cold water, and purified by column chromatography (petroleum ether:EtOAc, 95:5) to afford product **7db** as white solid (149 mg, 75% yield).

Mol Formula	$C_{14}H_{14}O$	**m.p.**	110-112 °C

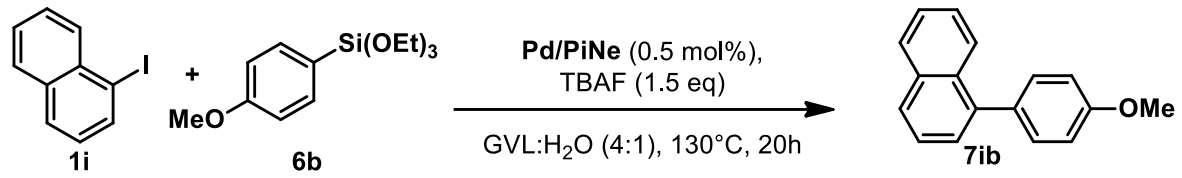
Elemental Analysis: Calc.: C: 84.81; H: 7.12. found: C: 84.79; H: 7.10

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	7.51	2	*d*	8
	7.45	2	*d*	8
	7.23	2	*d*	8
	6.97	2	*d*	8
	3.85	3	*s*	
	2.39	3	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 159.1, 138.1, 136.5, 133.9, 129.6, 128.1, 126.7, 114.3, 55.5, 21.2

GC-EIMS (m/z, %): 199 (14), 198 (M^+, 100), 183 (59), 155 (44), 154 (11), 153 (17), 152 (17), 128 (16), 127 (10), 115 (12)

Chem.Name	1-(4-methoxyphenyl)naphthalene (**7ib**)
Lit.Ref	E. Ismalaj, G. Strappaveccia, E. Ballerini, F. Elisei, O. Piermatti, D. Gelman, L. Vaccaro *ACS Sustainable Chem. Eng.***, 2014,** 2, 2461-2464



1i **6b** **7ib**

MW: 234,29 g/mol

Method:

Prepared according to general procedure: in a 4 mL screw-capped vial equipped with a magnetic stirrer, the aryl halide **1i** (254 mg, 1mmol), triethoxy(4-methoxyphenyl) silane **6b** (418 mg, 0.406 mL, 1.5 mmol), TBAF (1.5 equiv, 488 mg) and **Pd/PiNe** (0.5 mol%, 5.3 mg) were consecutively added, and the resulting mixture was left under stirring at 130 °C. After 20 h, the catalyst was filtered off and washed with 1 mL of GVL. The product was precipitated with 1 mL of water at 0°C. The precipitate was filtered off, washed with 1 mL of cold water, and purified by column chromatography (petroleum ether) to afford product **7ib** as white solid (211 mg, 90% yield).

Mol Formula	$C_{17}H_{14}O$	**m.p.**	114-116°C

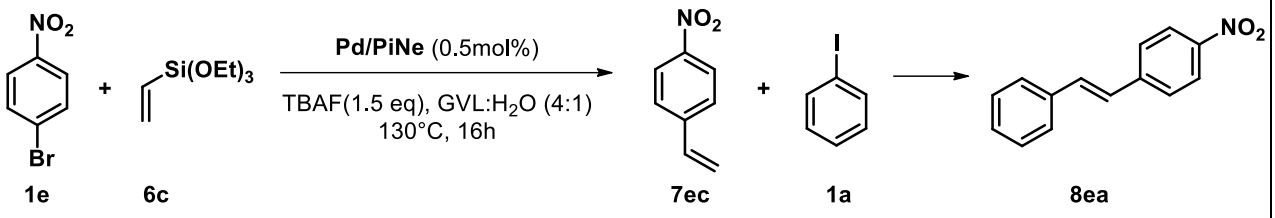
Elemental Analysis: Calc.: C: 87.15; H: 6.02. found: C: 87.25; H: 6.04

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	7.91	2	*t*	8
	7.84	1	*d*	8
	7.50	2	*q*	8
	7.45-7.40	4	*m*	
	7.04	2	*d*	8
	3.90	3	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 159.1, 140.1, 134.0, 133.3, 132.0, 131.3, 128.4, 127.5, 127.1, 126.2, 126.1, 125.8, 125.6, 113.9, 55.5.

GC-EIMS (m/z, %): 235 (31), 234 (M^+, 100), 219 (50), 203 (15), 202 (11), 191 (33), 190 (39), 189 (56), 101 (10), 94 (11)

Chem.Name	(E)-1-nitro-4-styrylbenzene (**8ea**)
Lit.Ref	*ChemistrySelect*, **2019**, 4, 6913-6916



MW: 225.24 g/mol

Method:
Prepared according to general procedure: In a 4 mL screw-capped vial equipped with a magnetic stirrer were consecutively added 1-bromo-4-nitrobenzene **1e** (1 mmol, 202 mg), **Pd/PiNe** (0.5 mol%, 5.3 mg), TBAF (1.5 equiv, 488 mg), triethoxyvinylsilane **6c** (1.5 mmol, 326 µL), GVL (0.8 mL), and aq. t-Bu-phenol solution (7µM, 0.2 mL) and the resulting mixture was left under stirring at 130 °C. After 16 h, iodobenzene **1a** (1.1 eq) was added and the mixture was kept under stirring at 130°C. After 3 h, the catalyst was filtered off and washed with 1 mL of GVL. The crude material was precipitated with 1 mL of water at 0°C and purified by column chromatography (petroleum ether:EtOAc, 95:5) to afford product **8ea** as a pale yellow solid (136 mg, 60% yield).

Mol Formula	$C_{14}H_{11}NO_2$	**m.p.**	155-159 °C

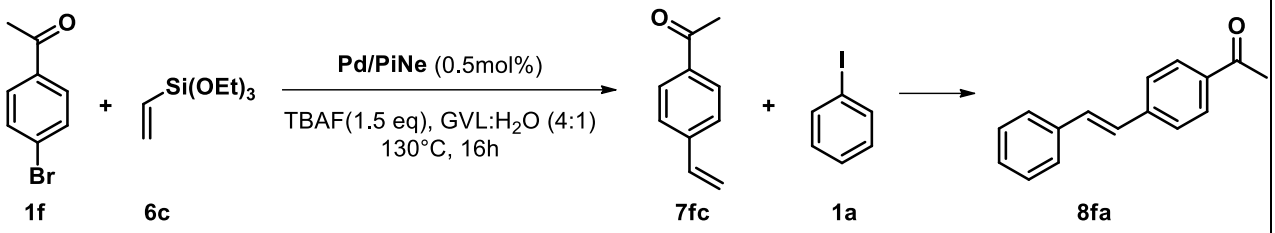
Elemental Analysis: Calc: C: 74.65; H: 4.92; N: 6.22; found: C: 74.72; H: 4.94; N: 6.20

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	8.19	2	*d*	8.4
	7.60	2	*d*	8.8
	7.53	2	*d*	7.6
	7.37	2	*t*	7.6
	7.32-7.29	1	*m*	
	7.22	1	*d*	16.4
	7.14-7.10	1	*d*	16.4

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 146.9, 144.0, 136.3, 133.5, 129.0, 129.0, 127.2, 127.0, 126.4, 124.3

GC-EIMS (m/z, %): 226 (15), 225 (M+, 100), 179 (40), 176 (17), 165 (15), 152 (27).

Chem.Name	(E)-1-(4-styrylphenyl)ethenone (**8fa**)
Lit.Ref	*ChemistrySelect*, **2019**, 4, 6913-6916



1f **6c** **7fc** **1a** **8fa**

MW: 222.28 g/mol

Method:

Prepared according to general procedure: In a 4 mL screw-capped vial equipped with a magnetic stirrer were consecutively added 4-bromo-acetophenone **1f** (1 mmol, 199 mg), **Pd/PiNe** (0.5 mol%, 5.3 mg), TBAF (1.5 equiv, 488 mg), triethoxyvinylsilane **6c** (1.5 mmol, 326 µL), GVL (0.8 mL), and aq. t-Bu-phenol solution (7µM, 0.2 mL) and the resulting mixture was left under stirring at 130 °C. After 16 h, iodobenzene **1a** (1.1 eq) was added and the mixture was kept under stirring at 130°C. After 3 h, the catalyst was filtered off and washed with 1 mL of GVL. The crude material was precipitated with 1 mL of water at 0°C and purified by column chromatography (petroleum ether:EtOAc, 95:5) to afford product **8fa** as a white solid (89 mg, 40% yield).

Mol Formula	$C_{16}H_{14}O$	**m.p.**	141-142 °C

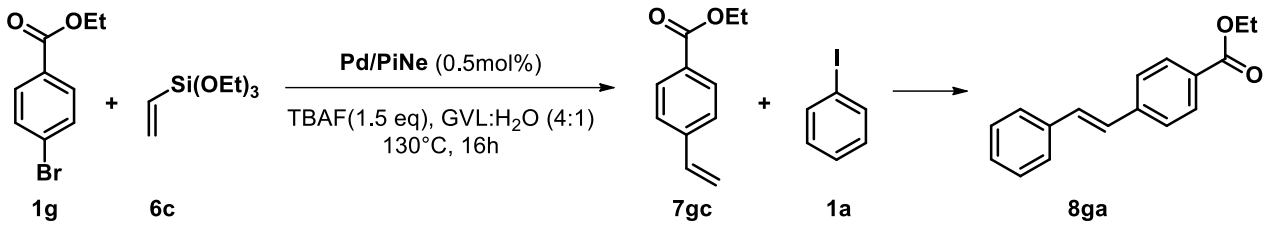
Elemental Analysis: Calc.: C: 86.45; H: 6.35; found: C: 86.52; H: 6.36

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	7.96	2	*d*	8.4
	7.59	2	*d*	8.4
	7.55	2	*d*	7.6
	7.39	2	*t*	7.6
	7.33-7.28	1	*m*	
	7.24	1	*d*	16.4
	7.13	1	*d*	16.4
	2.61	3	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 197.6, 142.2, 136.8, 136.1, 131.6, 129.0, 128.9, 128.5, 127.6, 127.0, 126.6, 26.7

GC-EIMS (m/z, %): 223 (27), 222 (M+, 89), 208 (38), 207 (100), 179 (52), 178 (89), 177 (25), 176 (26), 152 (23), 89 (21).

Chem.Name	(E)-ethyl 4-styrylbenzoate (**8ga**)
Lit.Ref	*Chemistry, A European Journal*, **2015**, 21, 7061-7065



MW: 252.31 g/mol

Method:

Prepared according to general procedure: In a 4 mL screw-capped vial equipped with a magnetic stirrer were consecutively added ethyl 4-bromobenzoate **1g** (1 mmol, 229 mg), **Pd/PiNe** (0.5 mol%, 5.3 mg), TBAF (1.5 equiv, 488 mg), triethoxyvinylsilane **6c** (1.5 mmol, 326 µL), GVL (0.8 mL), and aq. t-Bu-phenol solution (7µM, 0.2 mL) and the resulting mixture was left under stirring at 130 °C. After 16 h, iodobenzene **1a** (1.1 eq) was added and the mixture was kept under stirring at 130°C. After 3 h, the catalyst was filtered off and washed with 1 mL of GVL. The crude material was precipitated with 1 mL of water at 0°C and purified by column chromatography (petroleum ether:EtOAc, 95:5) to afford product **8ga** as a white solid (94 mg, 37% yield).

Mol Formula	$C_{17}H_{16}O_2$	**m.p.**	108-109 °C

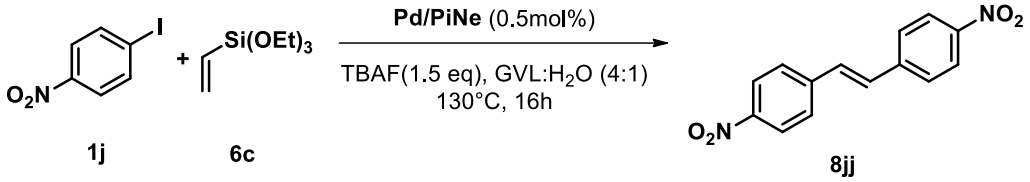
Elemental Analysis: Calc.: C: 80.93; H: 6.39; found: C: 80.98; H: 6.36

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	8.03	2	*d*	8.4
	7.58-7.53	4	*m*	
	7.38	2	*t*	7.6
	7.31-7.24	1	*m*	
	7.22	1	*d*	16.4
	7.13	1	*d*	16.4
	4.39	2	*q*	7.2
	1.41	3	*t*	7.2

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 166.6, 141.9, 136.9, 131.3, 130.1, 129.4, 128.9, 128.4, 127.8, 126.9, 126.4, 61.1, 14.5

GC-EIMS (m/z, %): 253 (19), 252 (M+, 100), 207 (54), 180 (12), 179 (77), 178 (63).

Chem.Name	(E)-1,2-bis(4-nitrophenyl)ethene (**8jj**)
Lit.Ref	*EurJOC*, **2006**, 5, 1323-1334



1j **6c** **8jj**

MW: 270.24 g/mol

Method:

Prepared according to general procedure: In a 4 mL screw-capped vial equipped with a magnetic stirrer were consecutively added 1-iodo-4-nitrobenzene **1e** (1 mmol, 254 mg), **Pd/PiNe** (0.5 mol%, 2.6 mg), TBAF (1.1 eq, 179 mg), triethoxyvinylsilane **6c** (0.5 mmol, 109 µL), GVL (0.8 mL), and aq. t-Bu-phenol solution (7µM, 0.2 mL) and the resulting mixture was left under stirring at 130 °C. After 16 h, the catalyst was filtered off and washed with 1 mL of GVL. The crude material was precipitated with 1 mL of water at 0°C and washed with petroleum ether to afford product **8jj** as a yellow solid (70 mg, 52% yield).

Mol Formula	$C_{14}H_{10}N_2O_4$	**m.p.**	234-235 °C

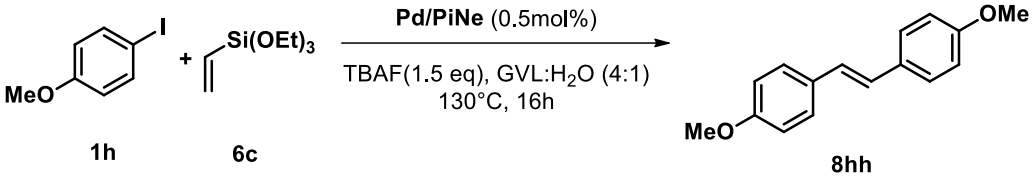
Elemental Analysis: Calc.: C: 62.22; H: 3.73; N: 10.37; found C: 62.32; H: 3.75; N: 10.40

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	8.27	4	*d*	8.4
	7.69	4	*d*	8.4
	7.30	2	*s*	

^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 147.6, 142.6, 130.8, 127.6, 124.4

GC-EIMS (m/z, %): 271 (16) 270 (M+,100), 178 (24), 177 (20), 176 (30), 166 (26), 165 (44).

Chem.Name	(E)-1,2-bis(4-methoxyphenyl)ethene (**8hh**)
Lit.Ref	*Appl Organometal Chem.*, **2019**; 33, e4618



1h **6c** **8hh**

MW: 240.30 g/mol

Method:

Prepared according to general procedure: In a 4 mL screw-capped vial equipped with a magnetic stirrer were consecutively added 4-iodoanisole **1h** (1 mmol, 238 mg), **Pd/PiNe** (0.5 mol%, 2.6 mg), TBAF (1.1 eq, 179 mg), triethoxyvinylsilane **6c** (0.5 mmol, 109 µL), GVL (0.8 mL), and aq. t-Bu-phenol solution (7µM, 0.2 mL) and the resulting mixture was left under stirring at 130 °C. After 16 h, the catalyst was filtered off and washed with 1 mL of GVL. The crude material was precipitated with 1 mL of water at 0°C and washed with petroleum ether to afford product **8hh** as a withe solid (49 mg, 41% yield).

Mol Formula	$C_{16}H_{16}O_2$	**m.p.**	100-101 °C

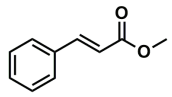
Elemental Analysis: Calc: C: 79.97; H: 6.71; found: C: 80.00; H: 6.72

	δ value:	No. H	Mult	J value/Hz
^{1}H NMR (400 MHz, $CDCl_3$)	7.43	4	*d*	8.8
	6.93	2	*s*	
	6.89	4	*d*	8.8
	3.83	6	*s*	

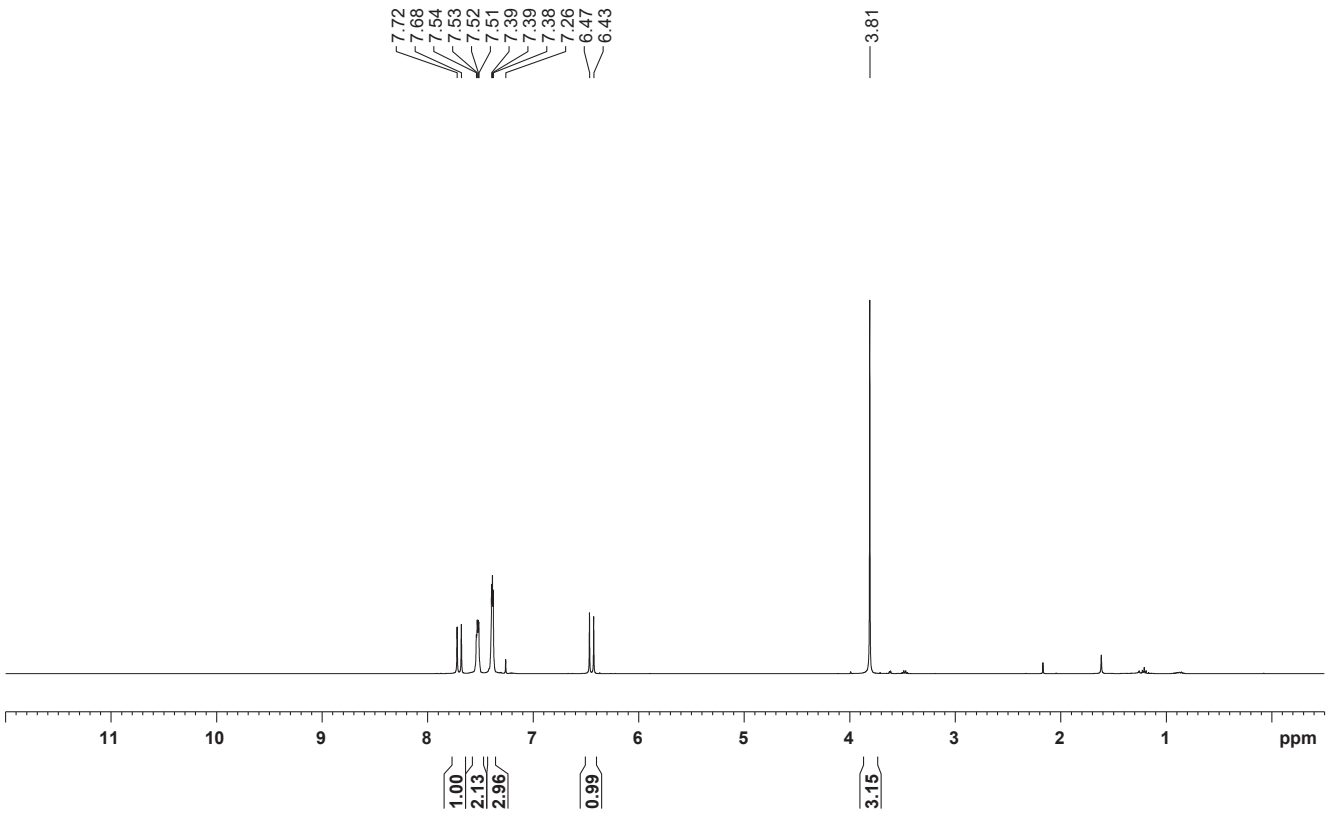
^{13}C NMR (100.6 Hz, $CDCl_3$) δ: 159.2, 130.6, 127.6, 126.3, 114.3, 55.5

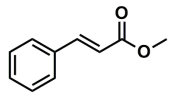
GC-EIMS (m/z, %): 241 (18), 240 (M+, 100), 225 (51).

4. ^{1}H and ^{13}C NMR spectra

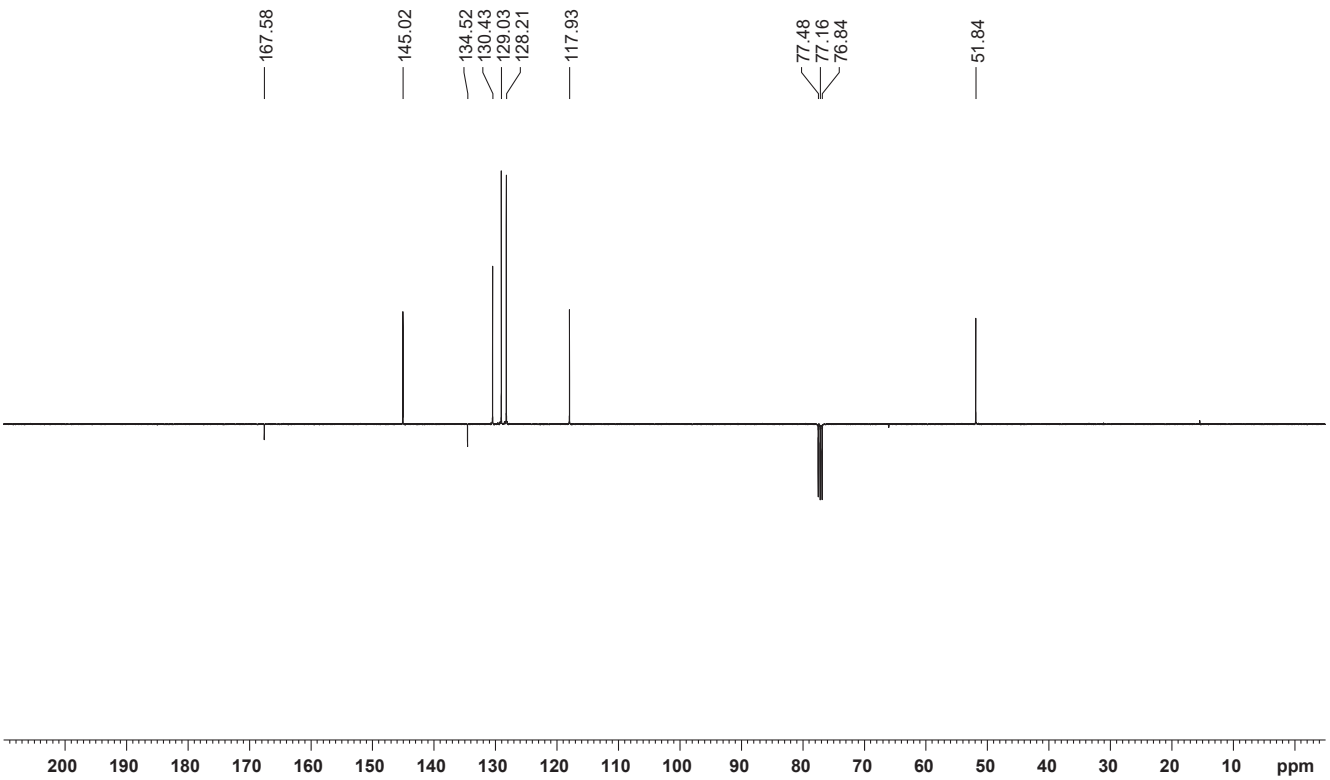


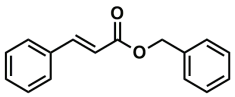
methyl cinnamate (**3aa**)



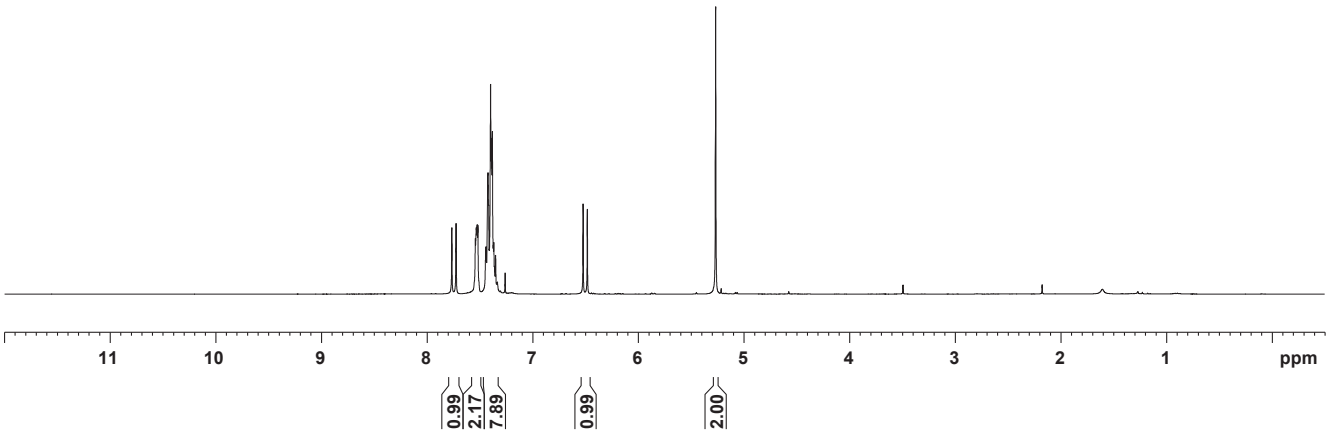


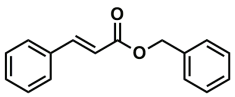
methyl cinnamate (**3aa**)



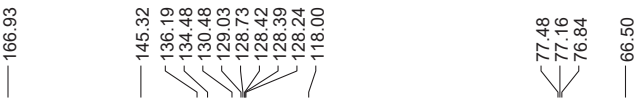


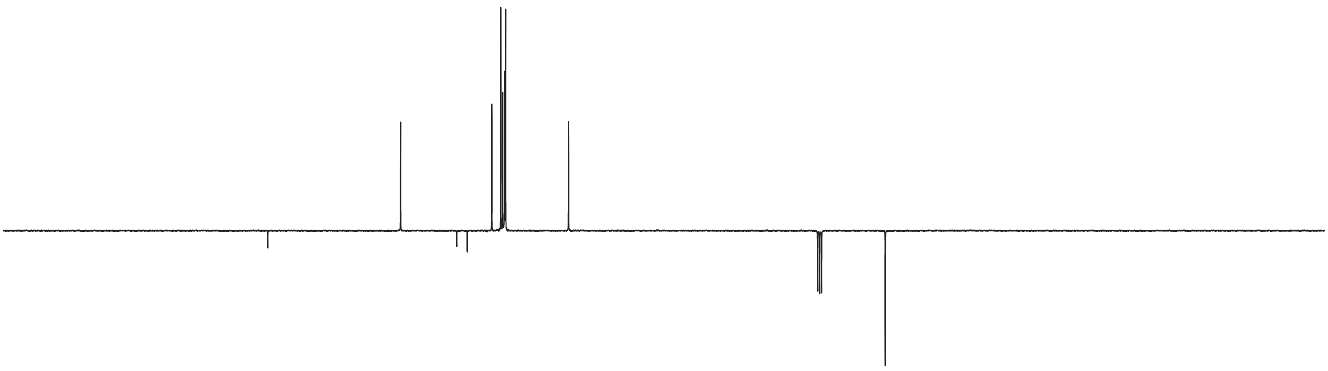
benzyl cinnamate (**3ab**)

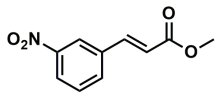




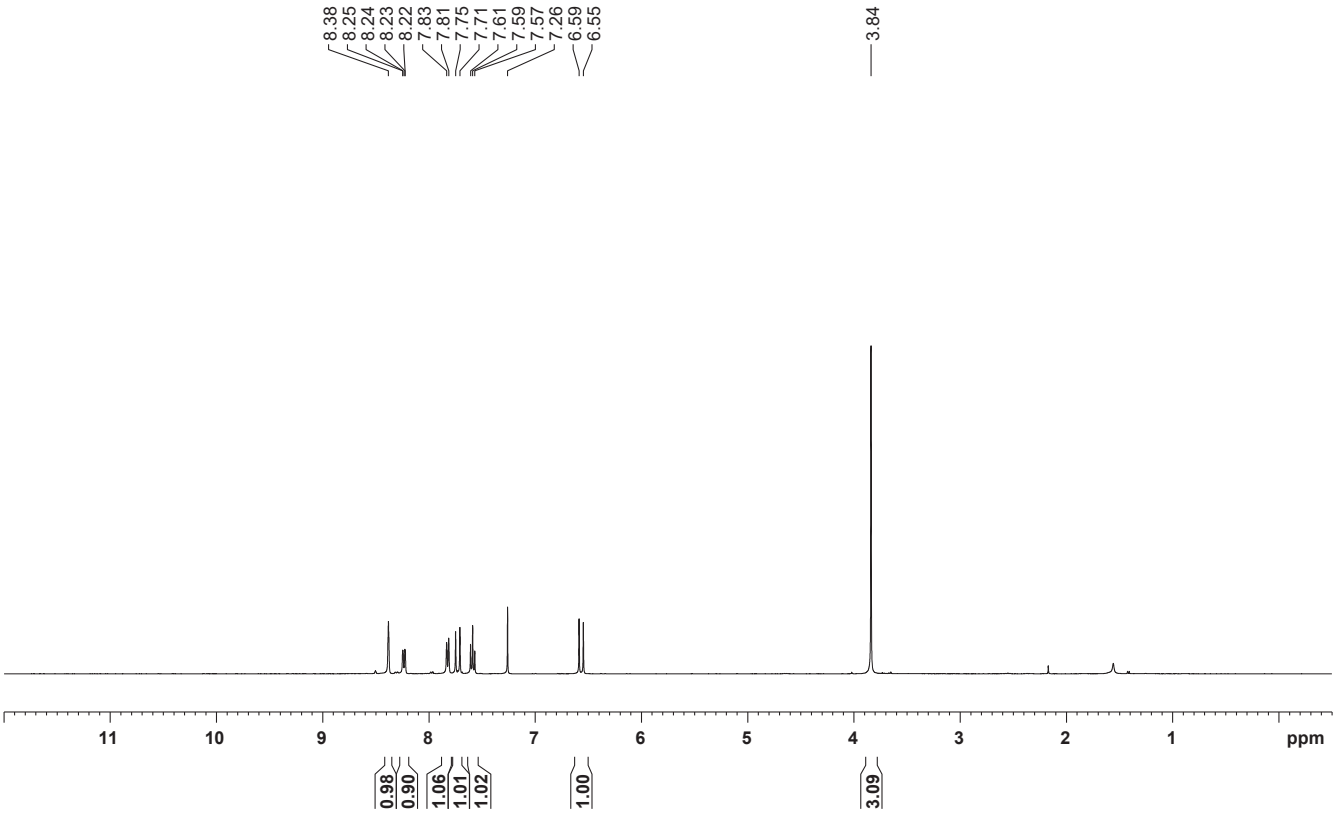
benzyl cinnamate (**3ab**)

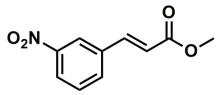




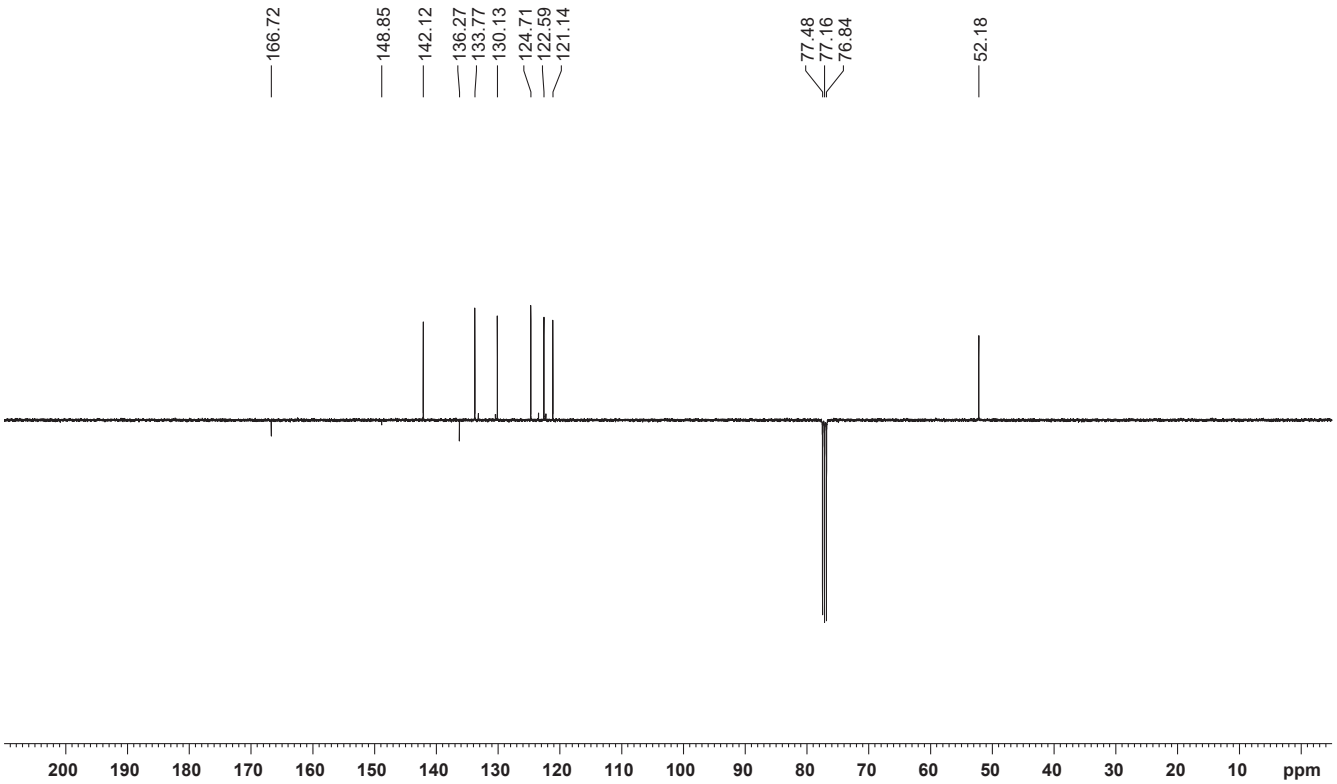


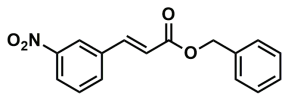
(E)-methyl 3-(3-nitrophenyl)acrylate (**3ba**)



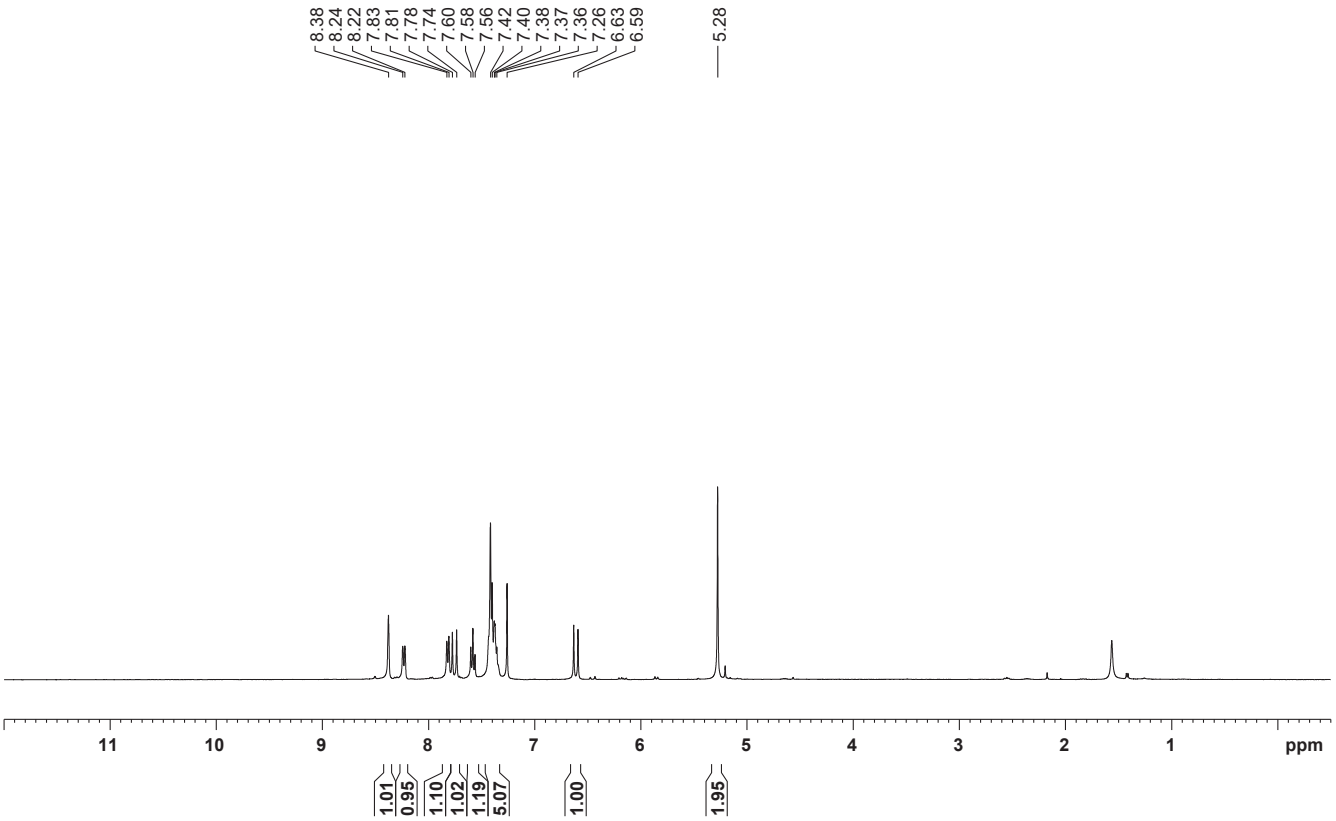


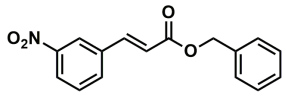
(E)-methyl 3-(3-nitrophenyl)acrylate (**3ba**)



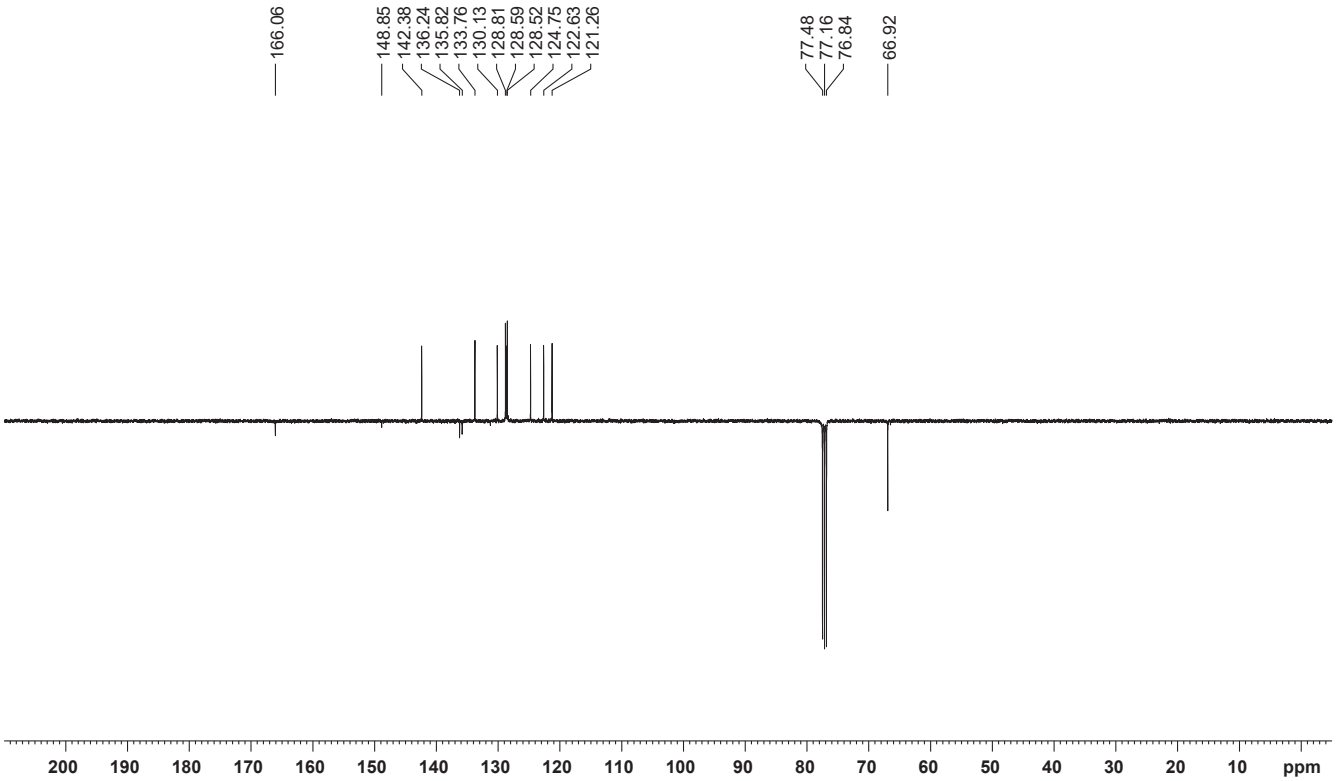


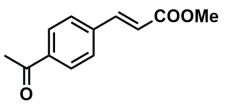
(E)-benzyl 3-(3-nitrophenyl)acrylate (**3bb**)



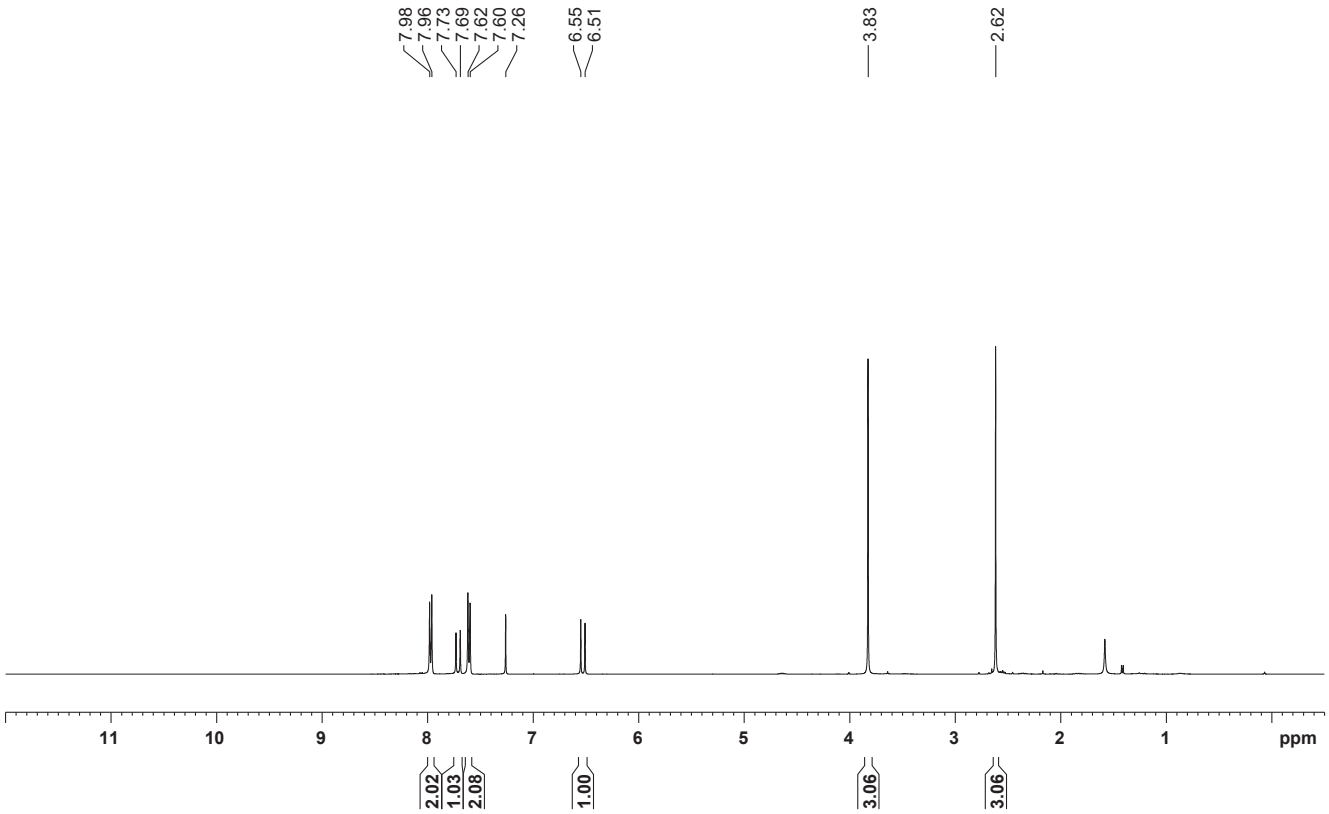


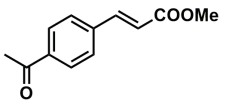
(E)-benzyl 3-(3-nitrophenyl)acrylate (**3bb**)



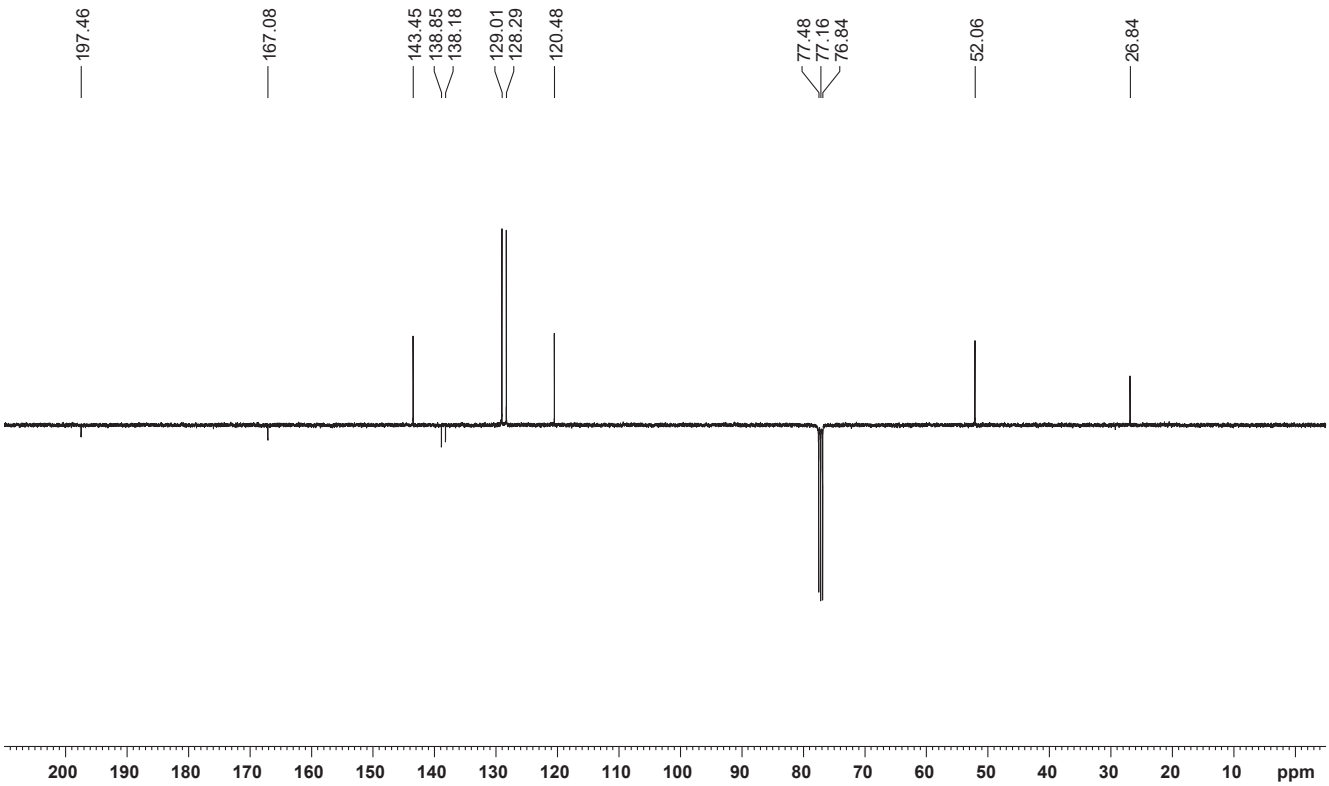


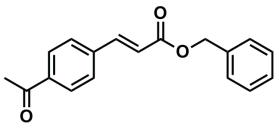
(E)-methyl 3-(4-acetylphenyl)acrylate (**3ca**)



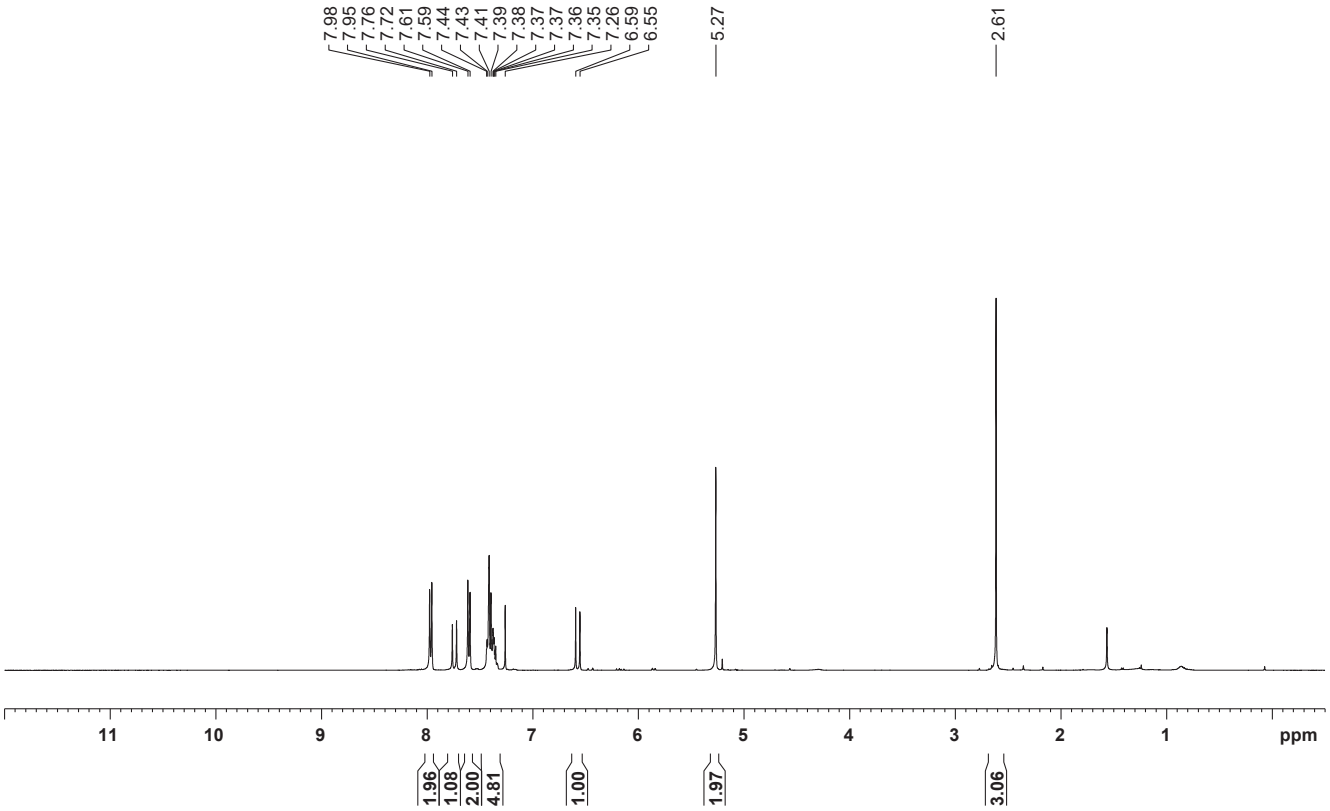


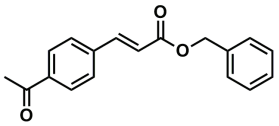
(E)-methyl 3-(4-acetylphenyl)acrylate (**3ca**)



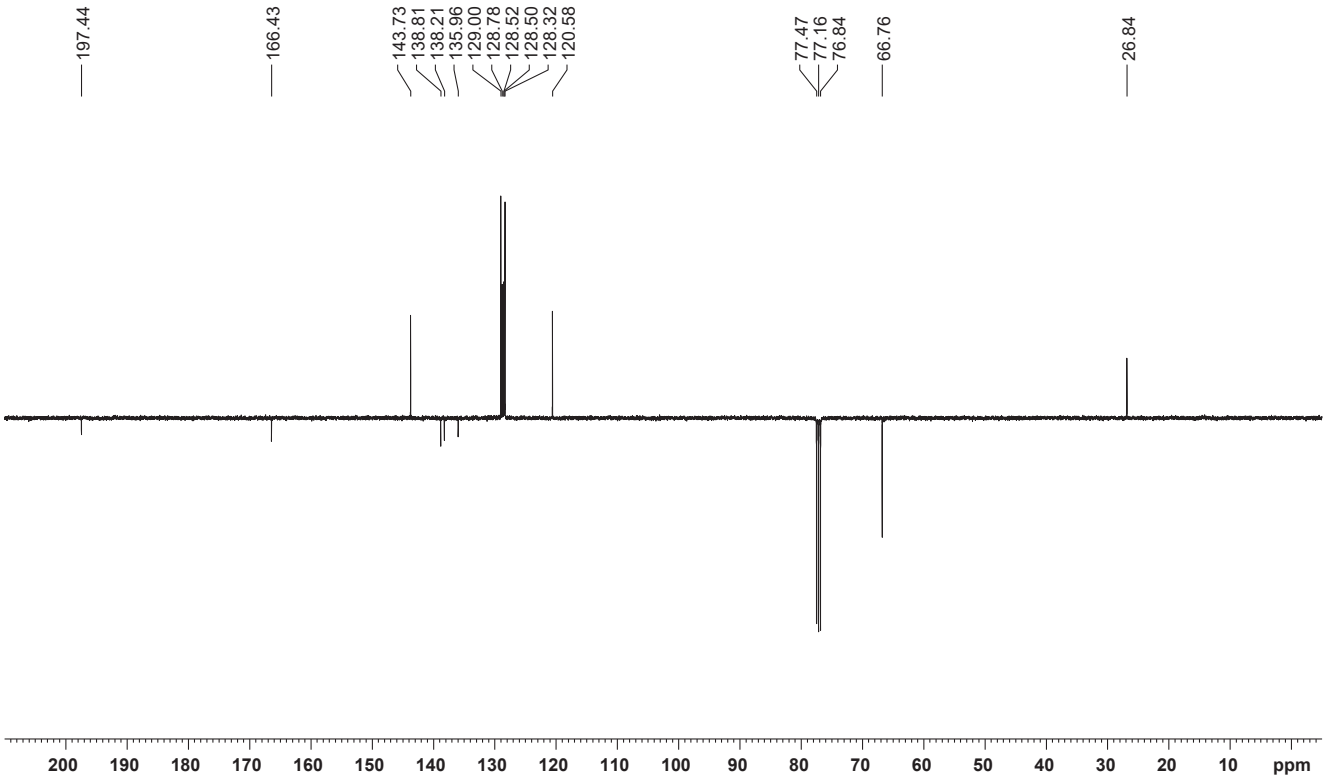


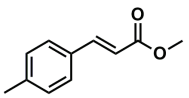
(E)-benzyl 3-(4-acetylphenyl)acrylate (**3cb**)



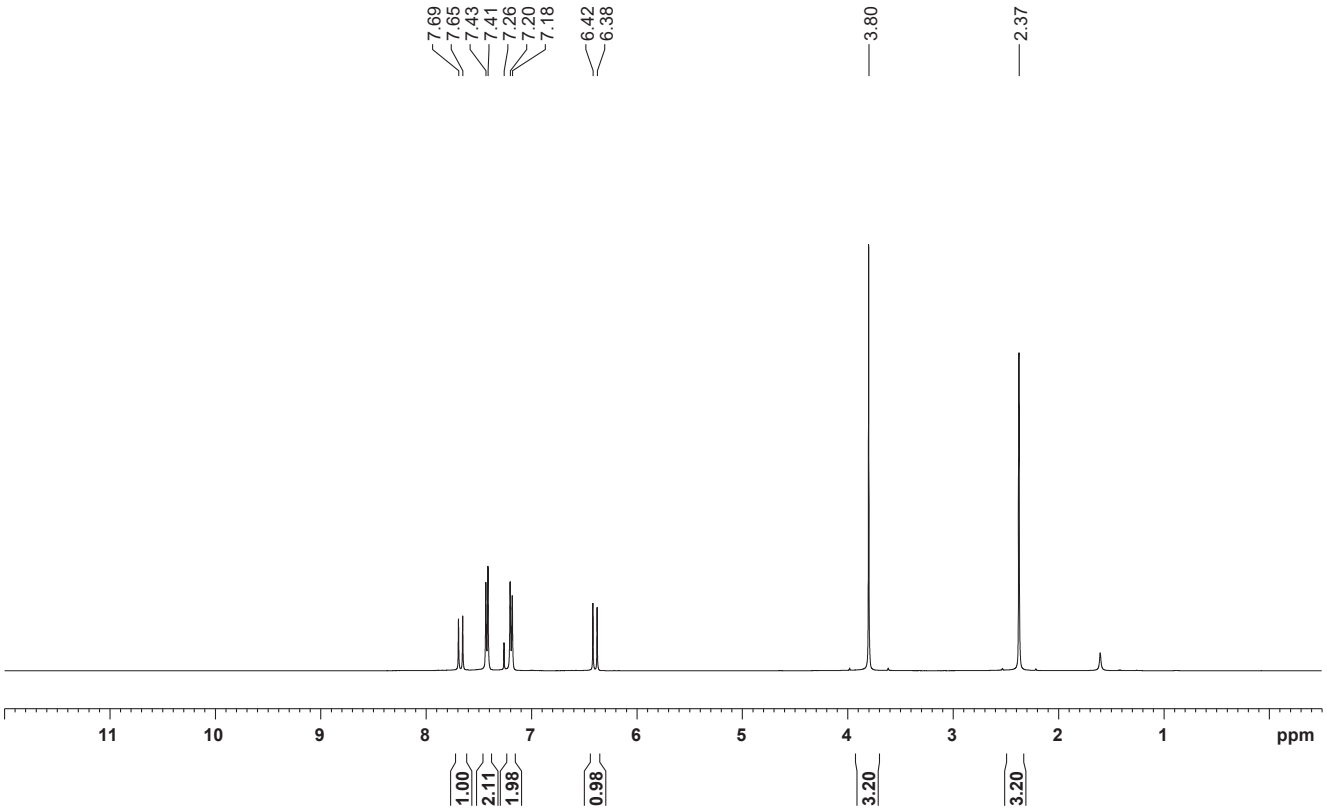


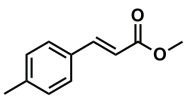
(E)-benzyl 3-(4-acetylphenyl)acrylate (**3cb**)



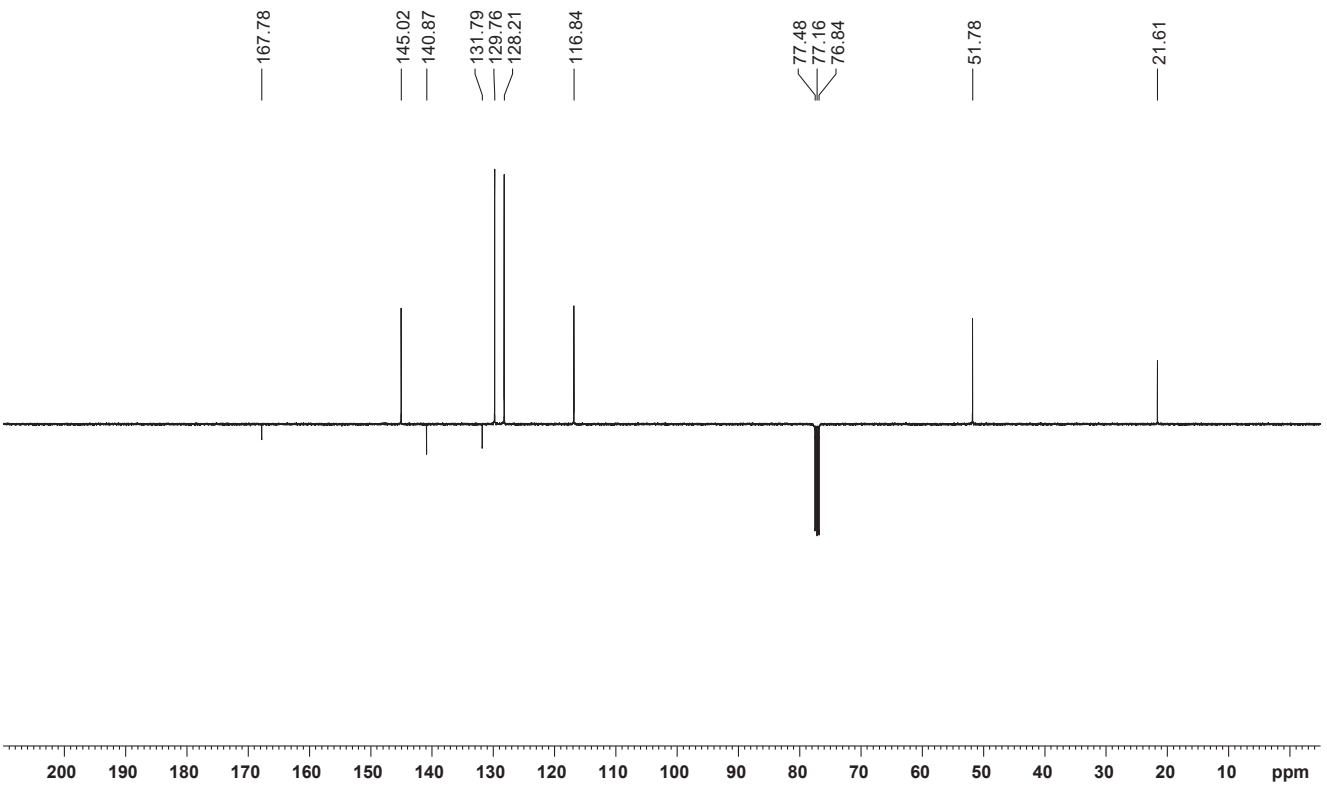


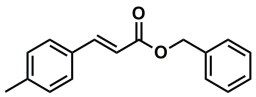
(E)-methyl 3-(p-tolyl)acrylate (**3da**)



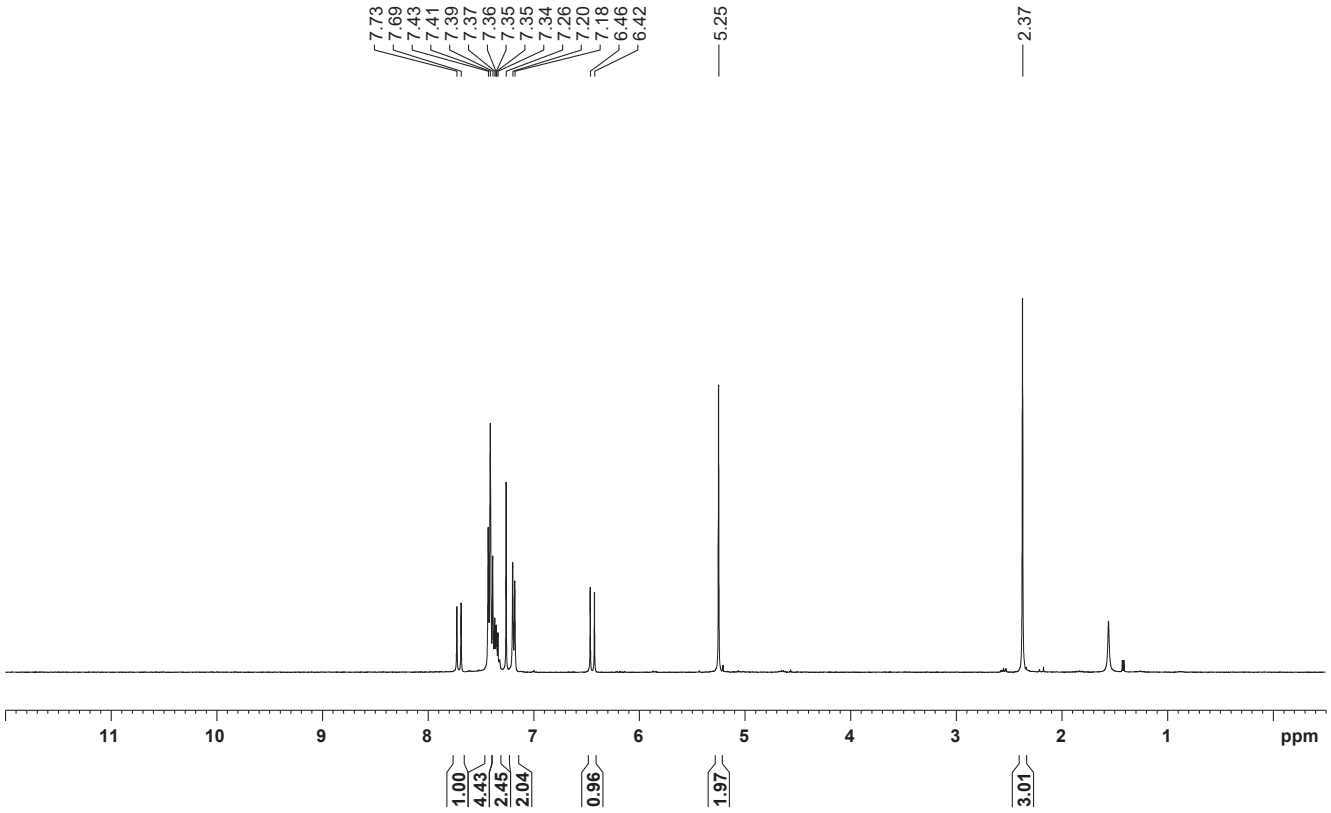


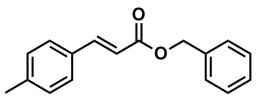
(E)-methyl 3-(p-tolyl)acrylate (**3da**)



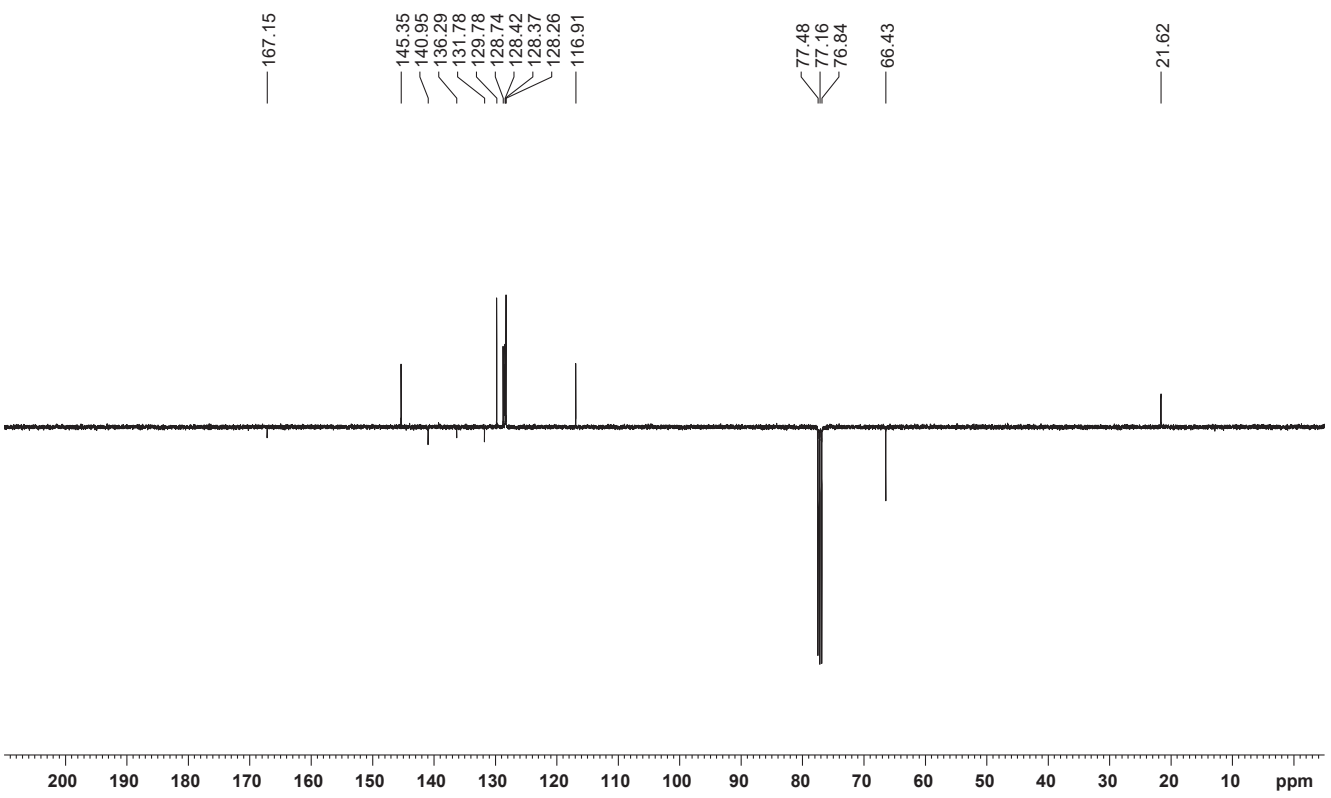


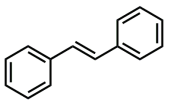
(E)-benzyl 3-(p-tolyl)acrylate (**3db**)



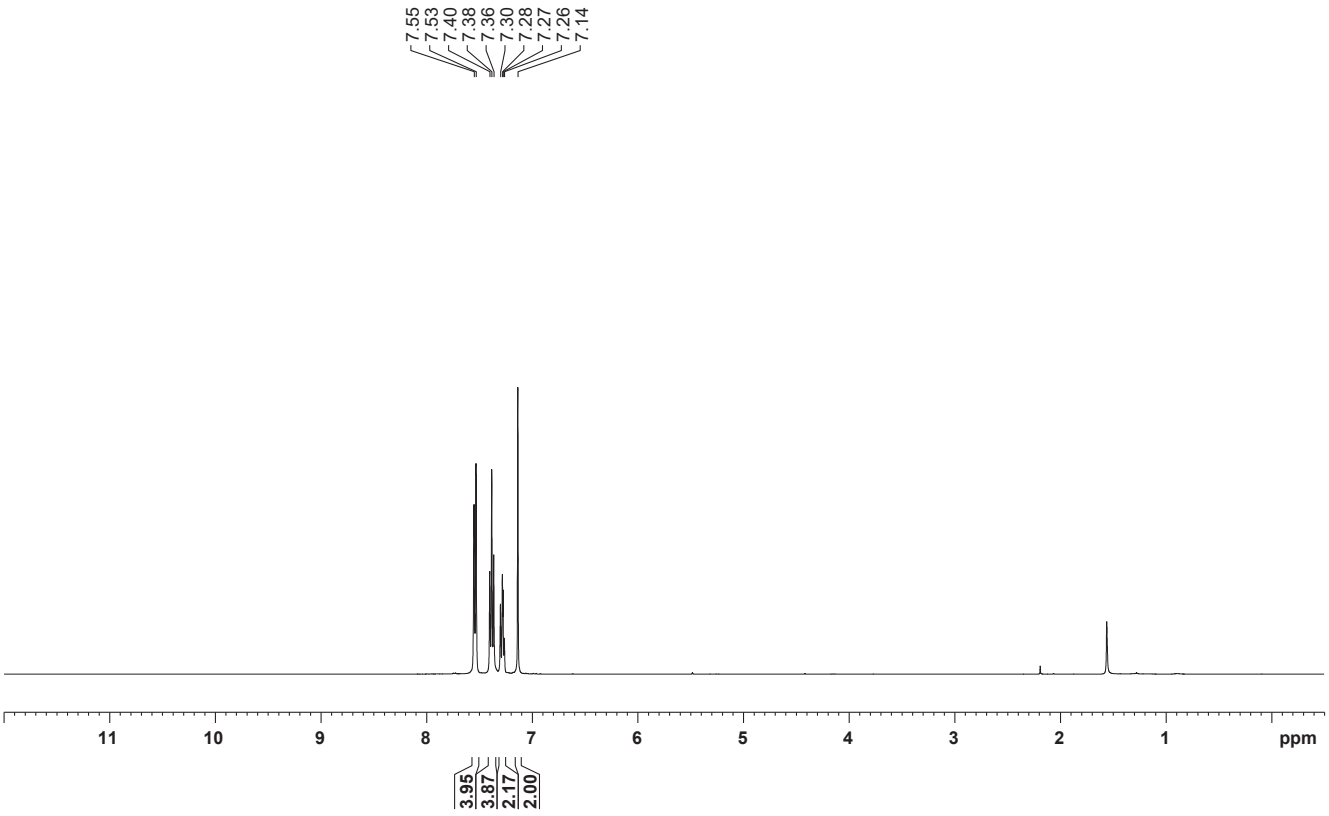


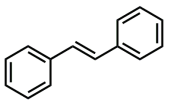
(E)-benzyl 3-(p-tolyl)acrylate (**3db**)



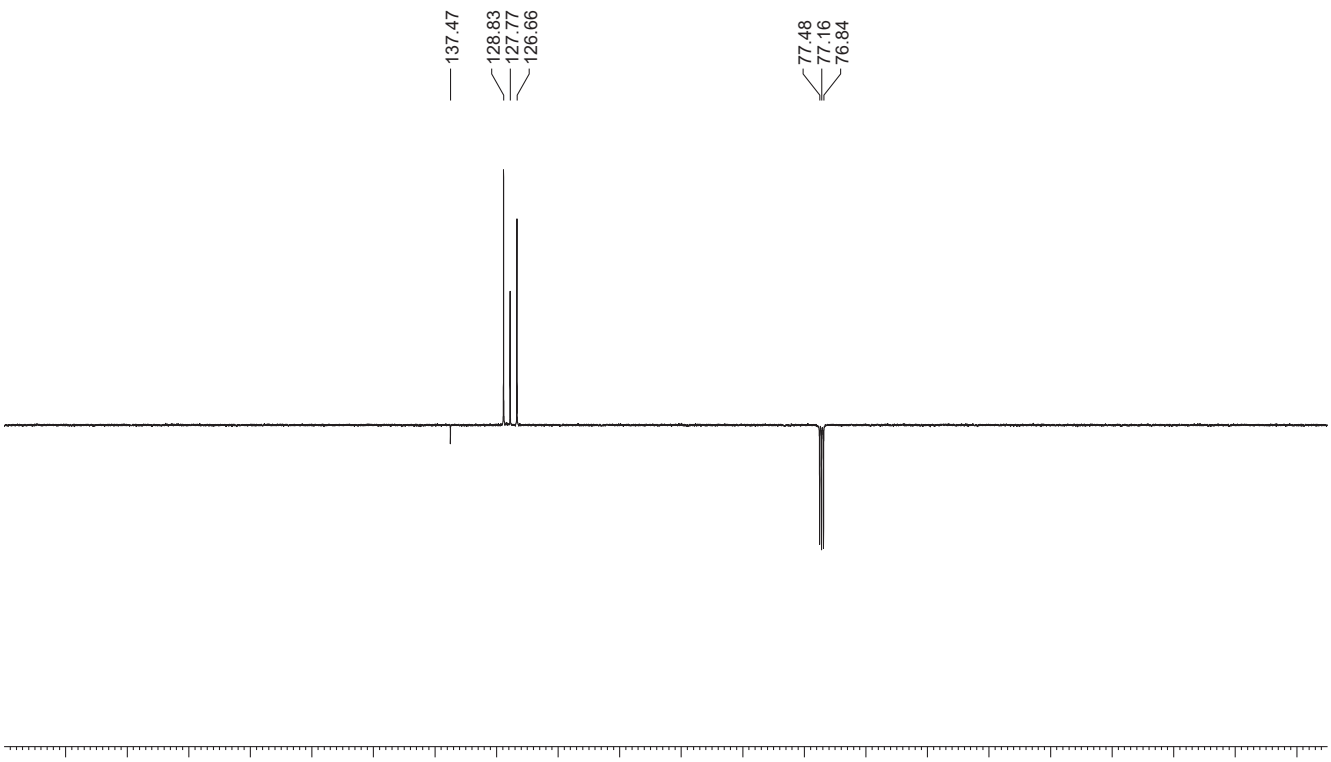


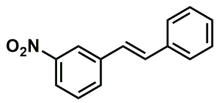
(E)-1,2-diphenylethene (**5a**)



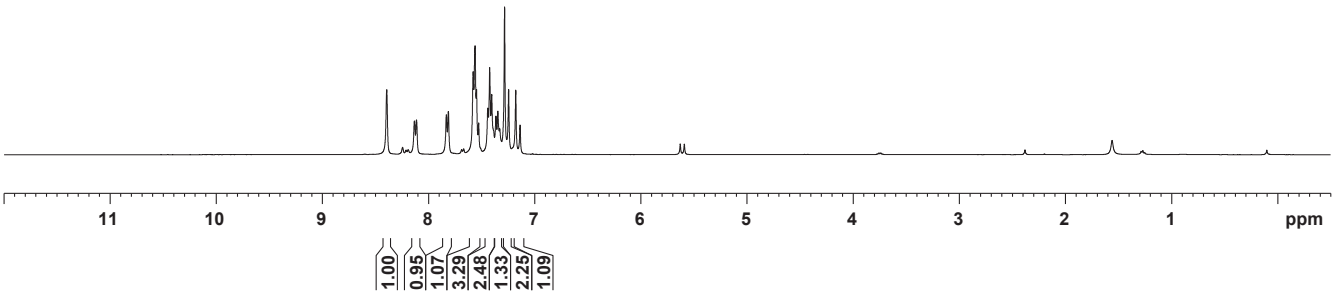


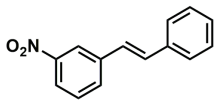
(E)-1,2-diphenylethene (**5a**)



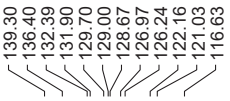


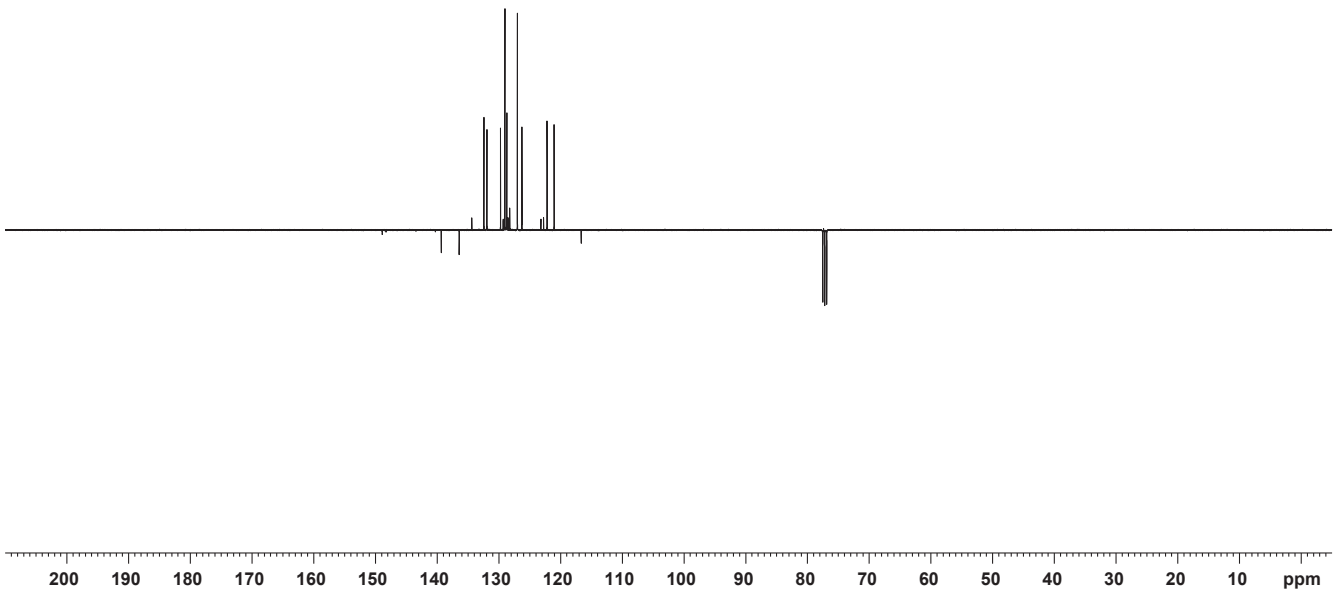
(E)-1-nitro-3-styrylbenzene (**5b**)

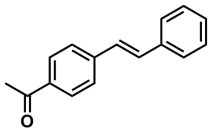




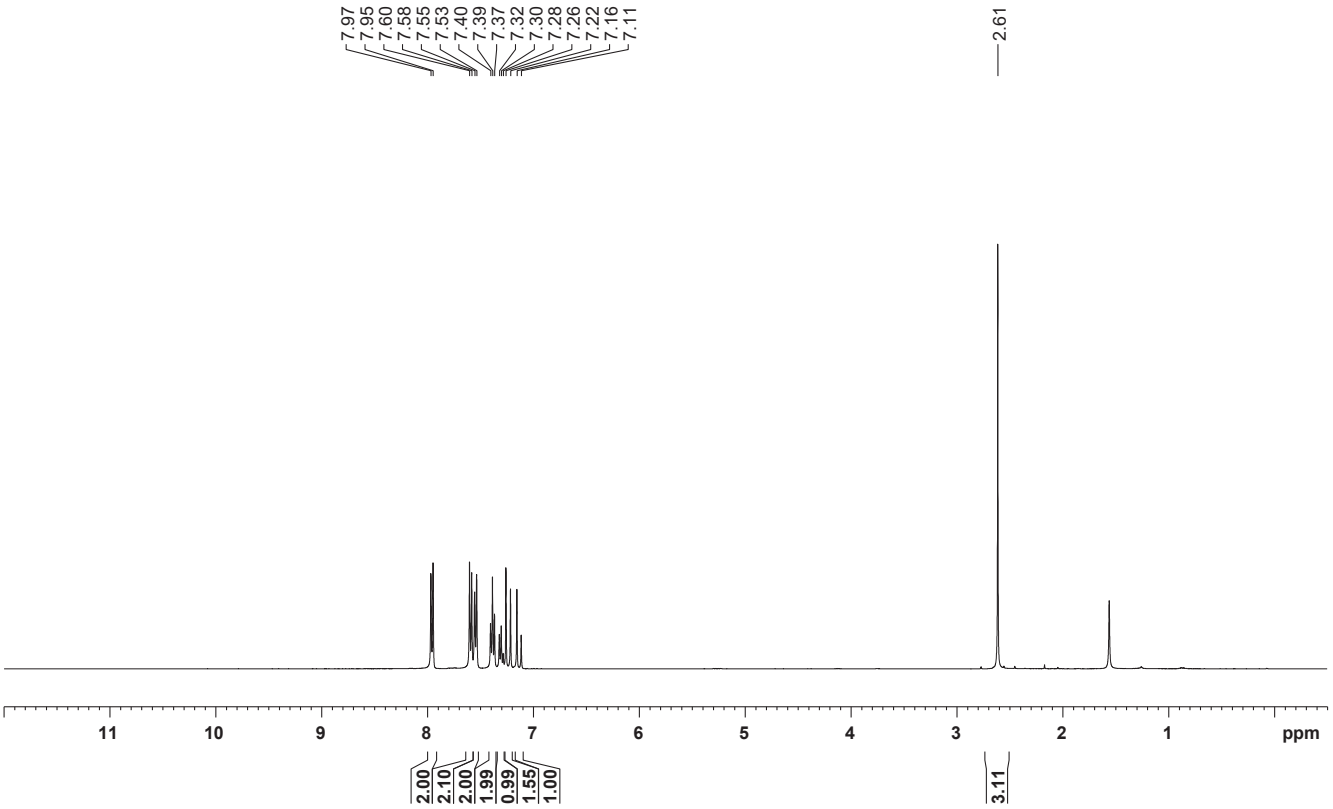
(E)-1-nitro-3-styrylbenzene (**5b**)

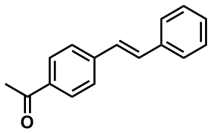




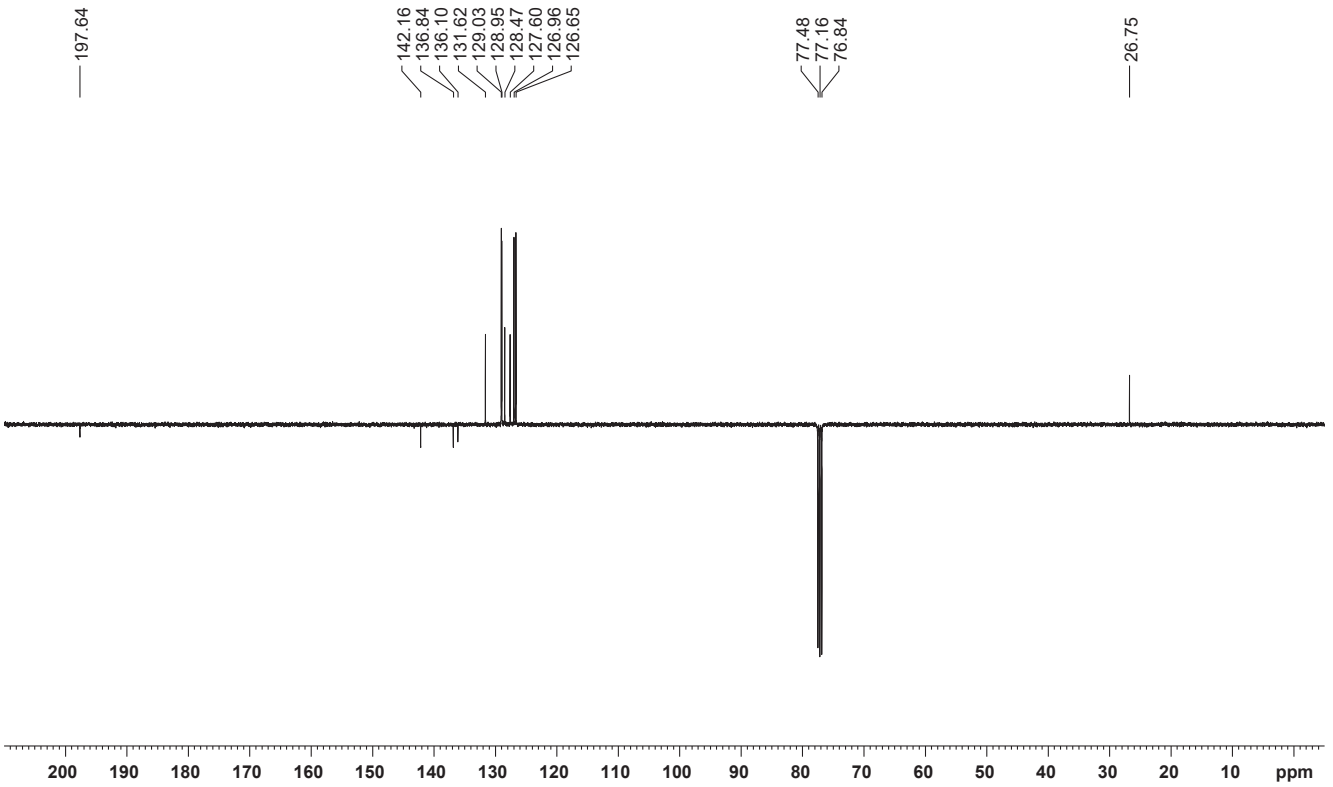


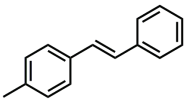
(E)-1-(4-styrylphenyl)ethanone (**5c**)



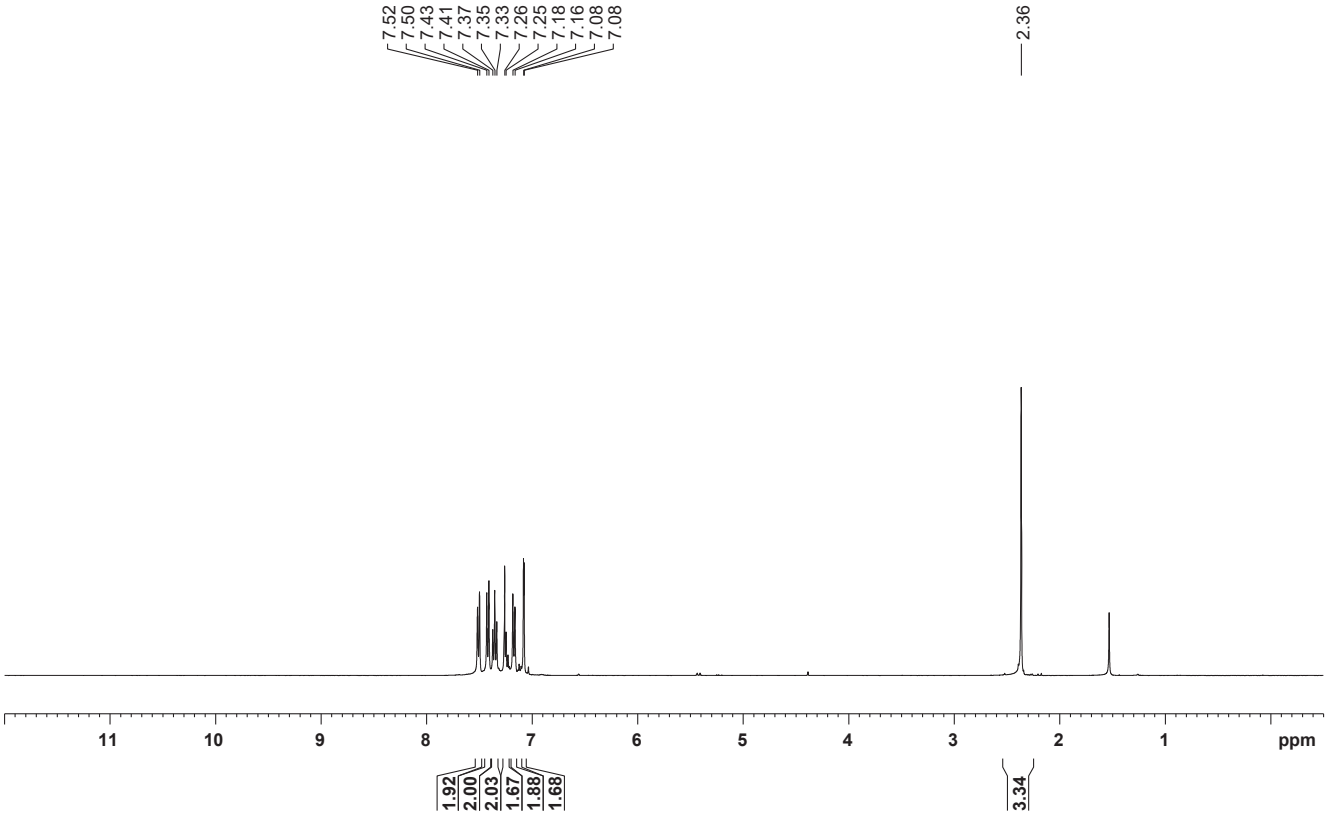


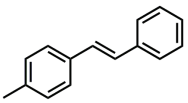
(E)-1-(4-styrylphenyl)ethanone (**5c**)



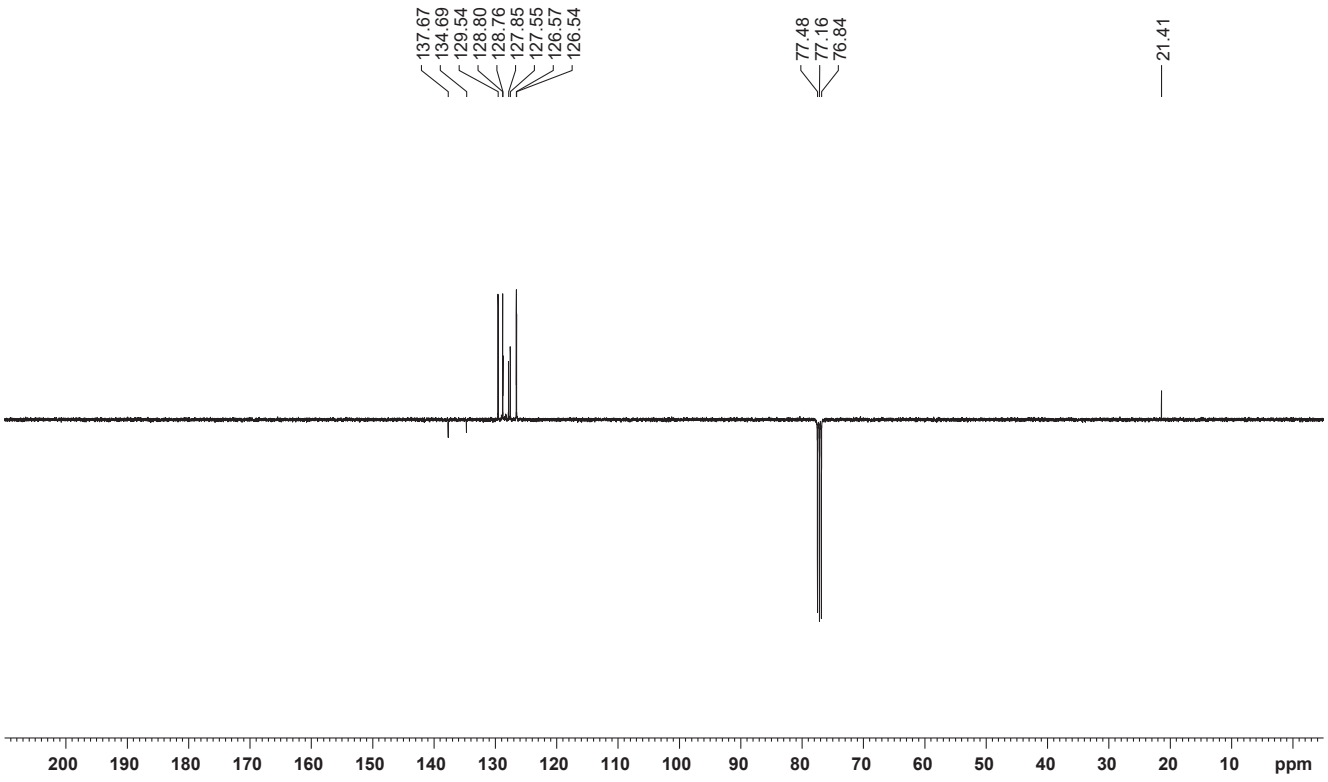


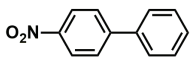
(E)-1-methyl-4-styrylbenzene (**5d**)



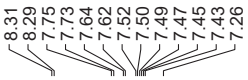


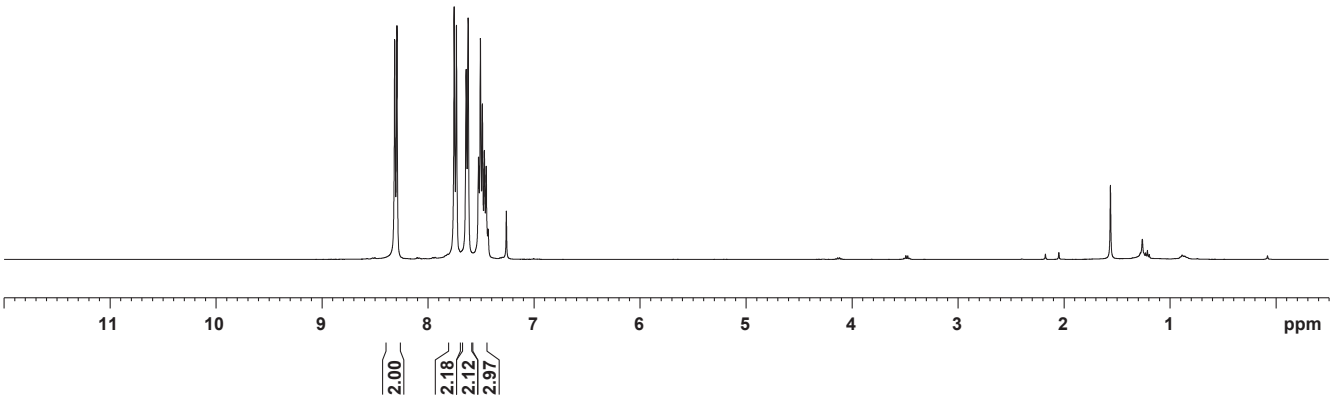
(E)-1-methyl-4-styrylbenzene (**5d**)

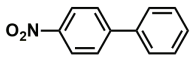




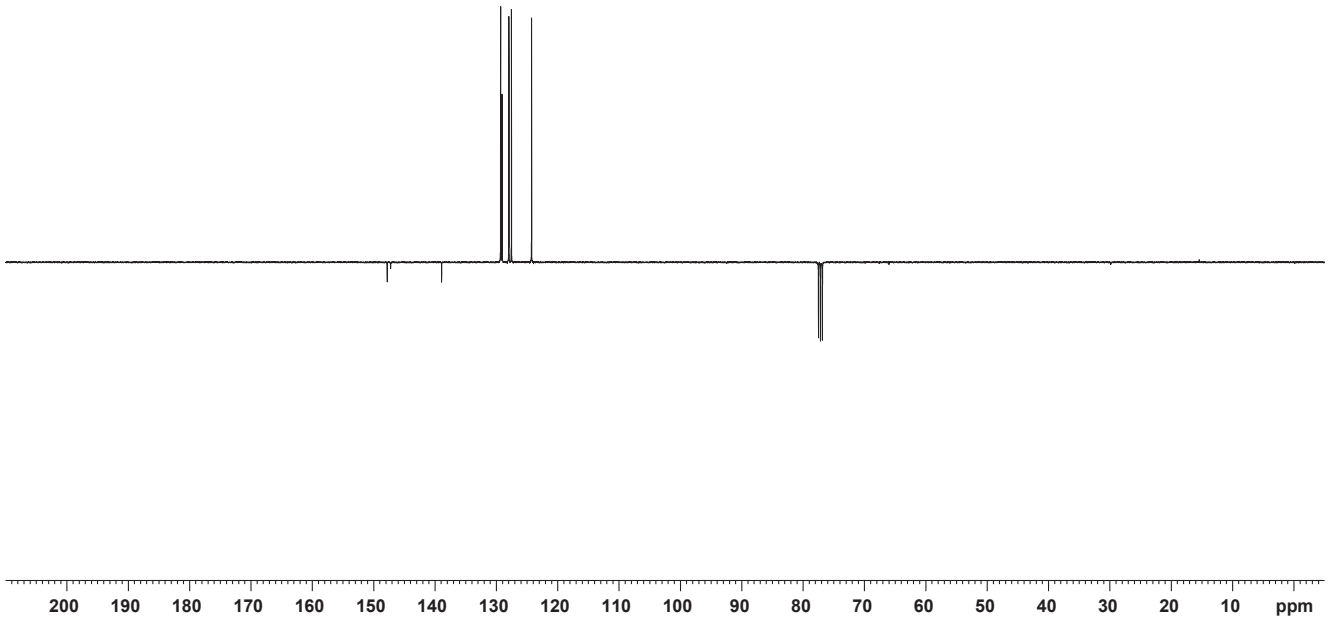
4-nitro-1,1'-biphenyl (**7ea**)

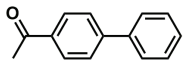




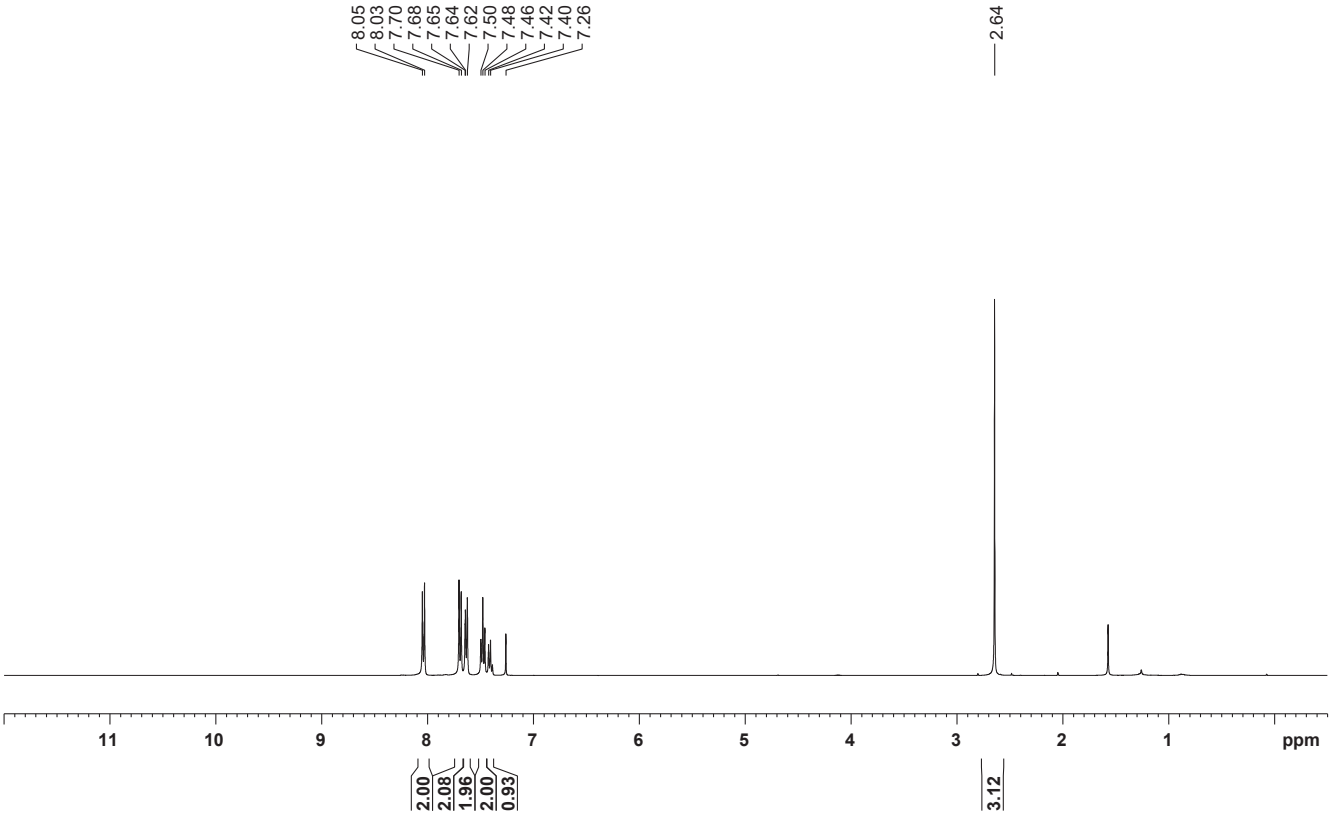


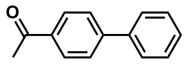
4-nitro-1,1'-biphenyl (**7ea**)



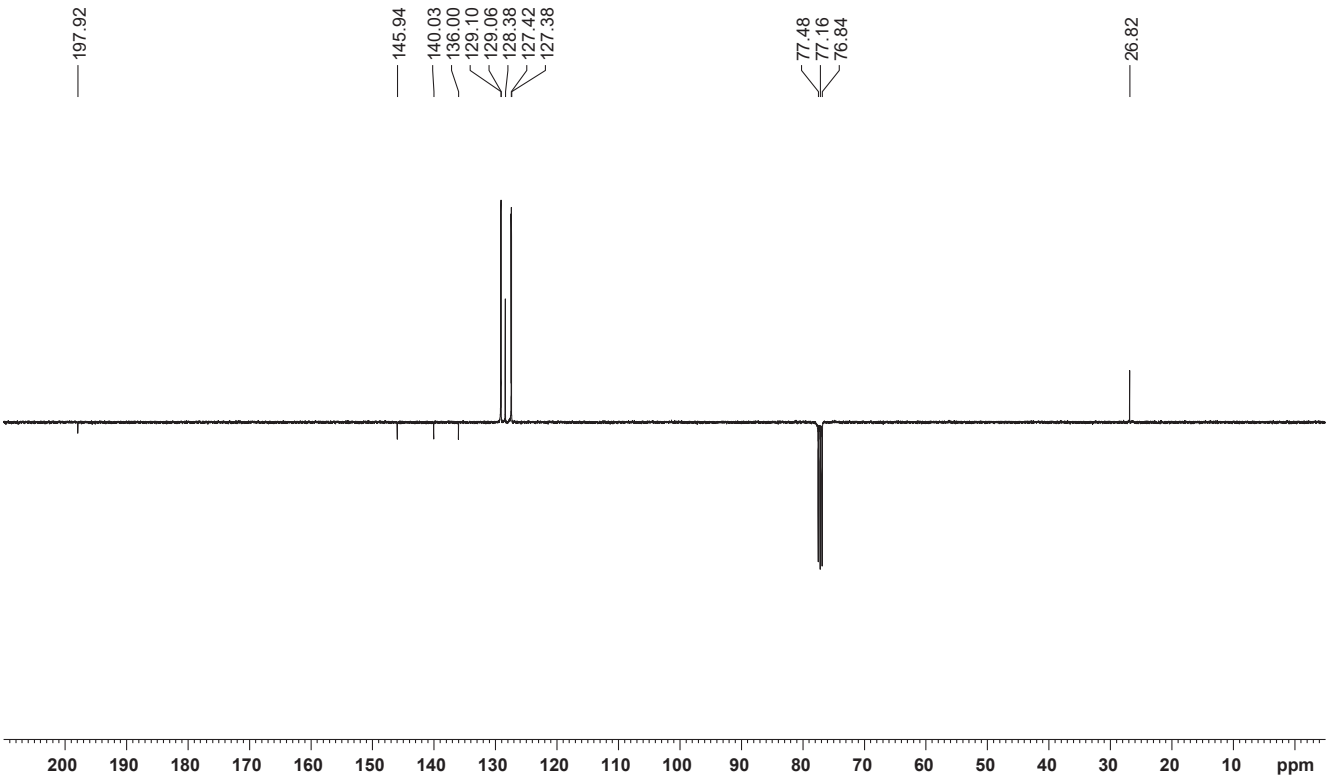


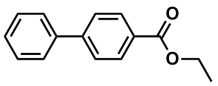
1-([1,1'-biphenyl]-4-yl)ethanone (**7fa**)



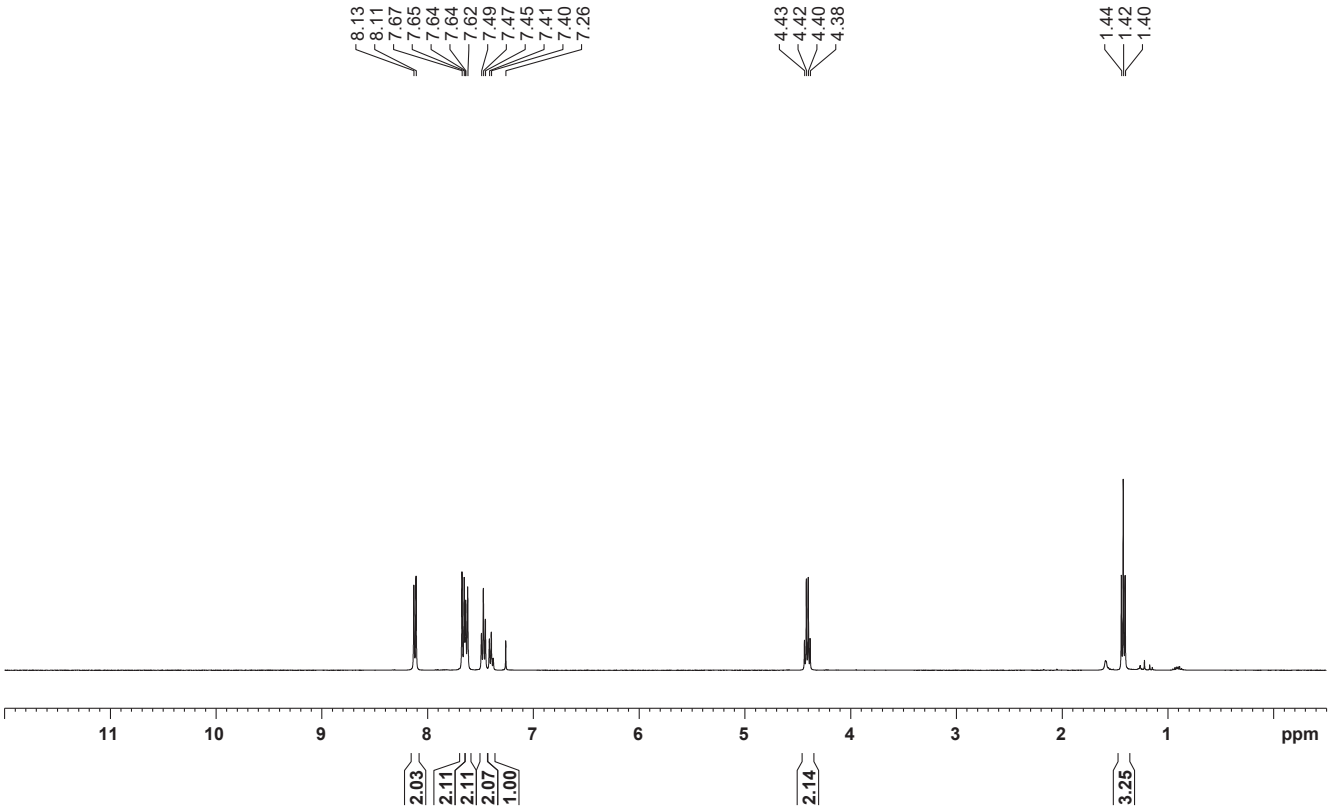


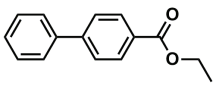
1-([1,1'-biphenyl]-4-yl)ethanone (**7fa**)



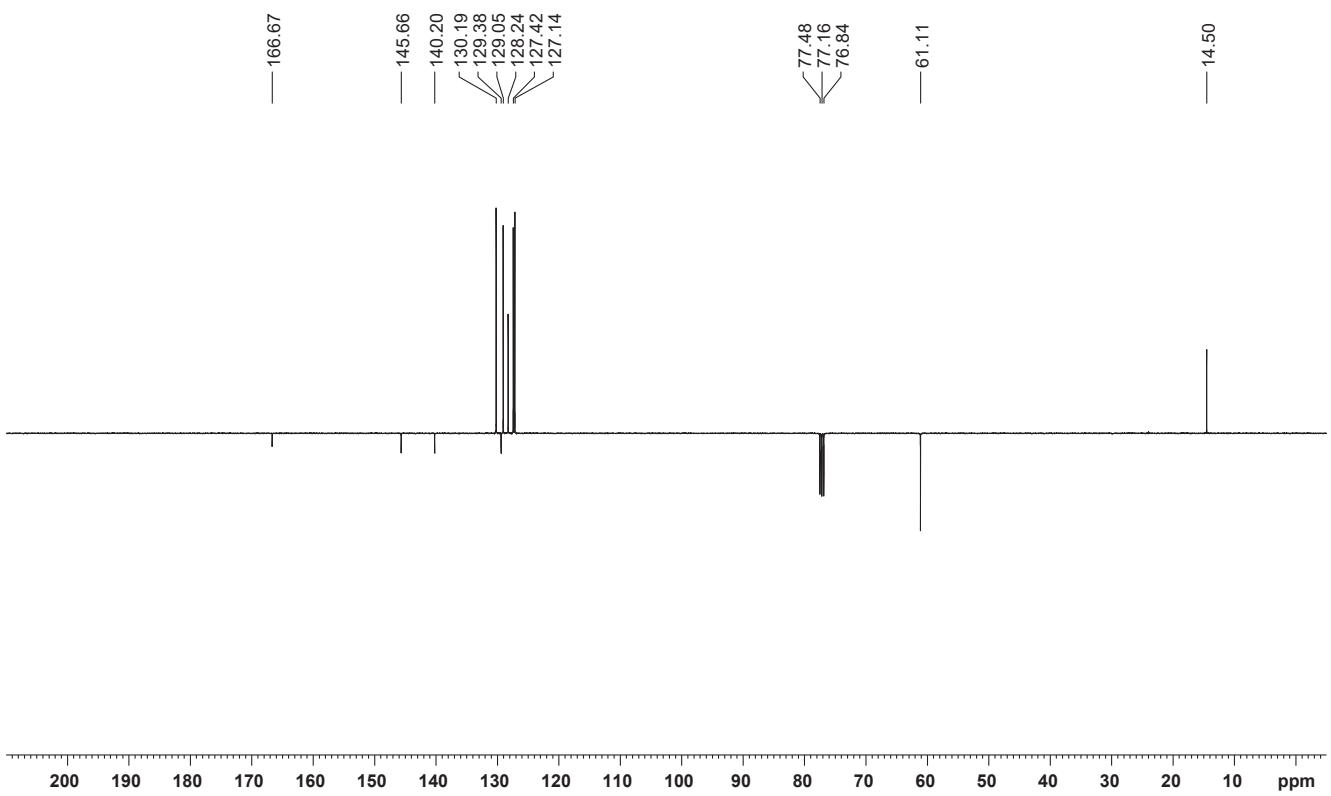


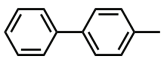
ethyl [1,1'-biphenyl]-4-carboxylate (**7ga**)



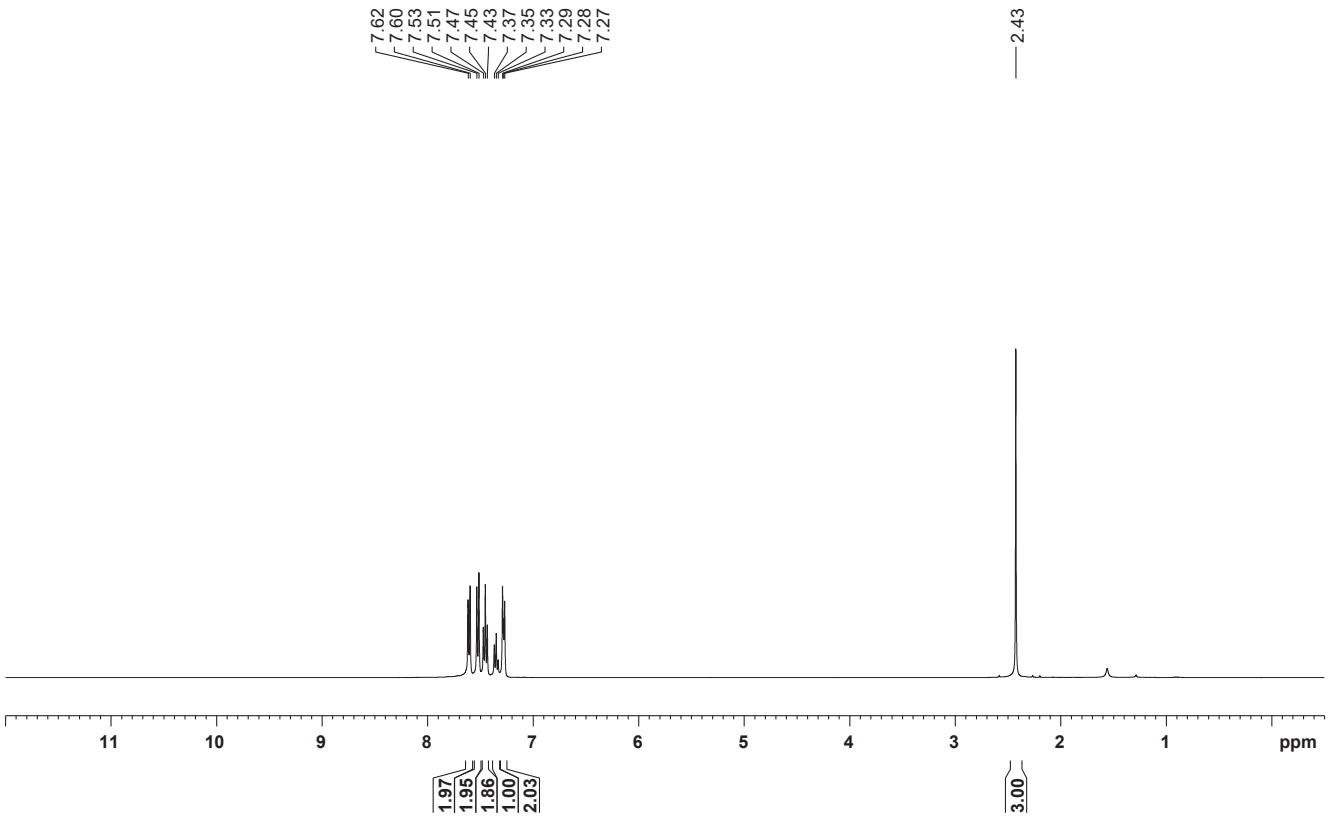


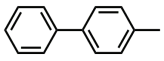
ethyl [1,1'-biphenyl]-4-carboxylate (**7ga**)



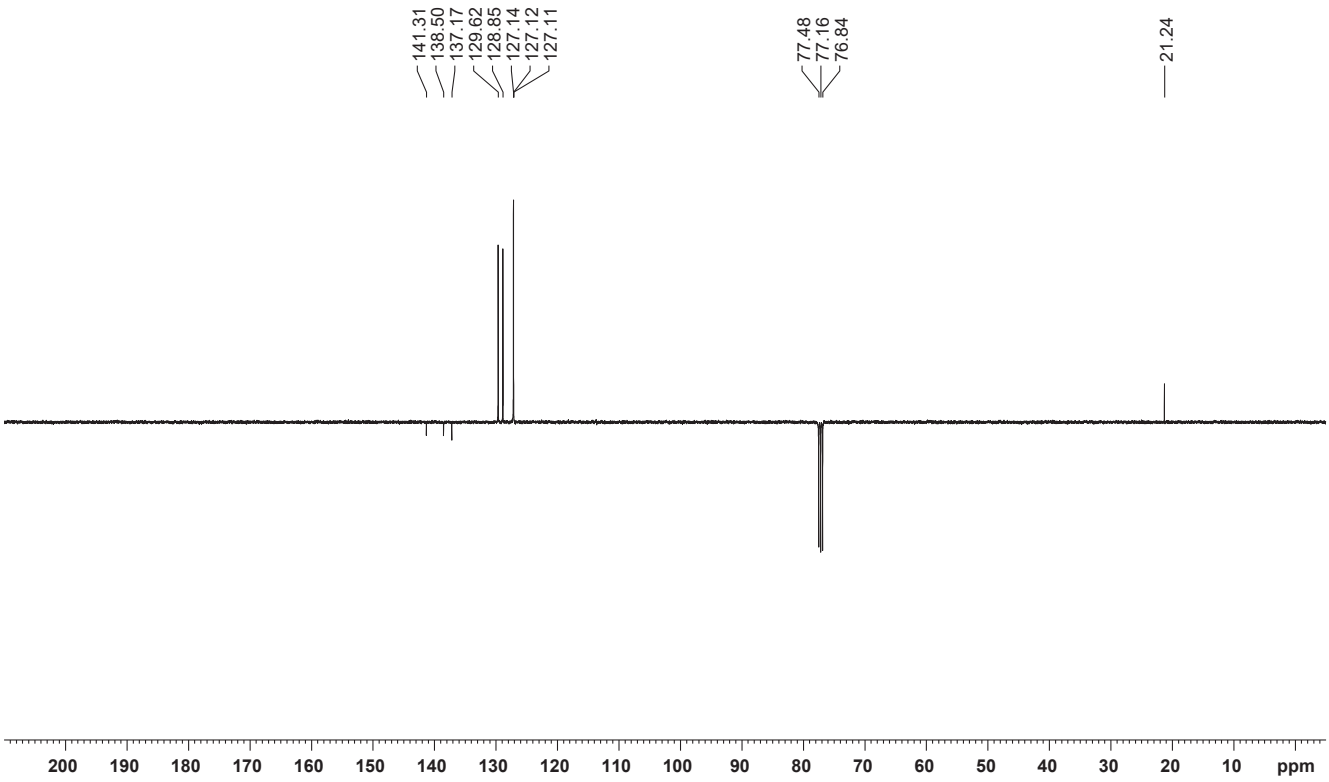


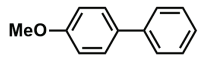
4-methyl-1,1'-biphenyl (**7da**)



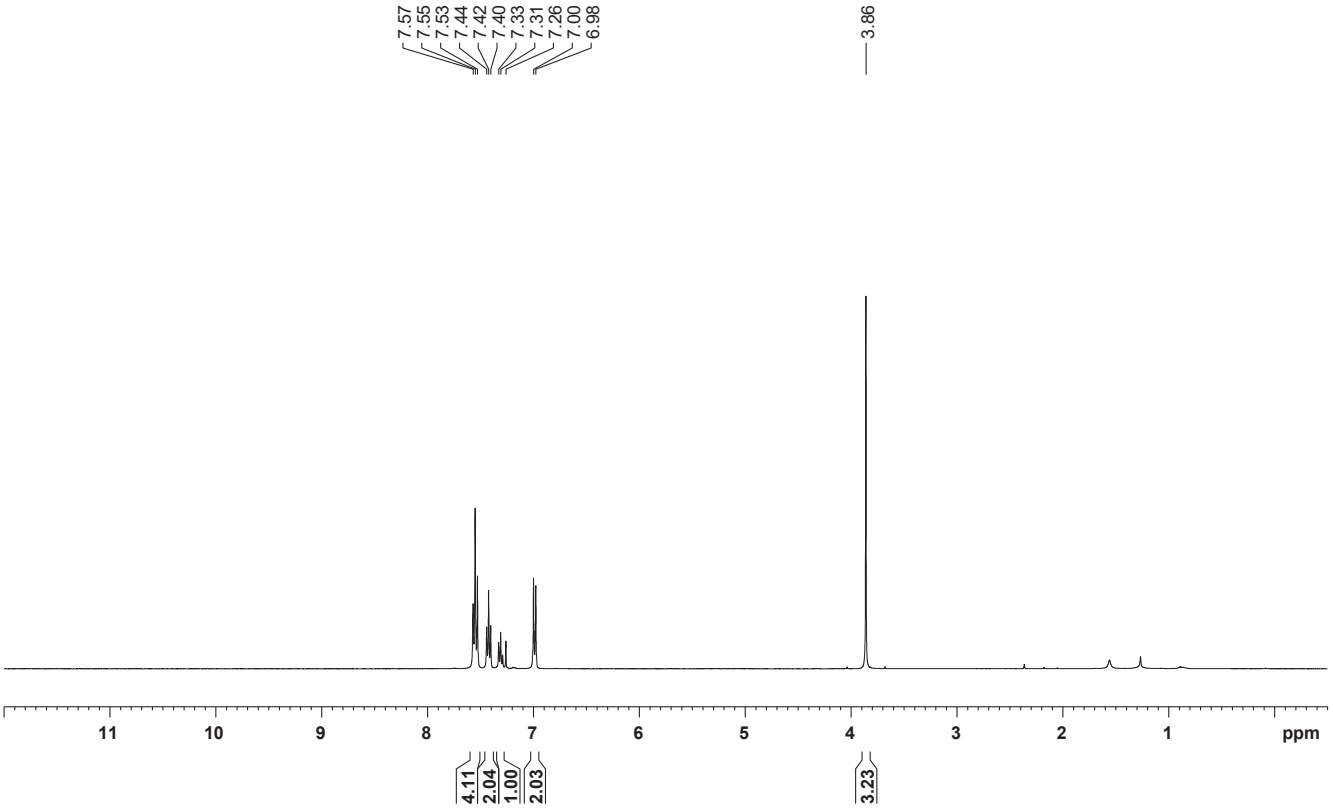


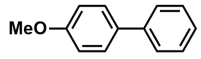
4-methyl-1,1'-biphenyl (**7da**)



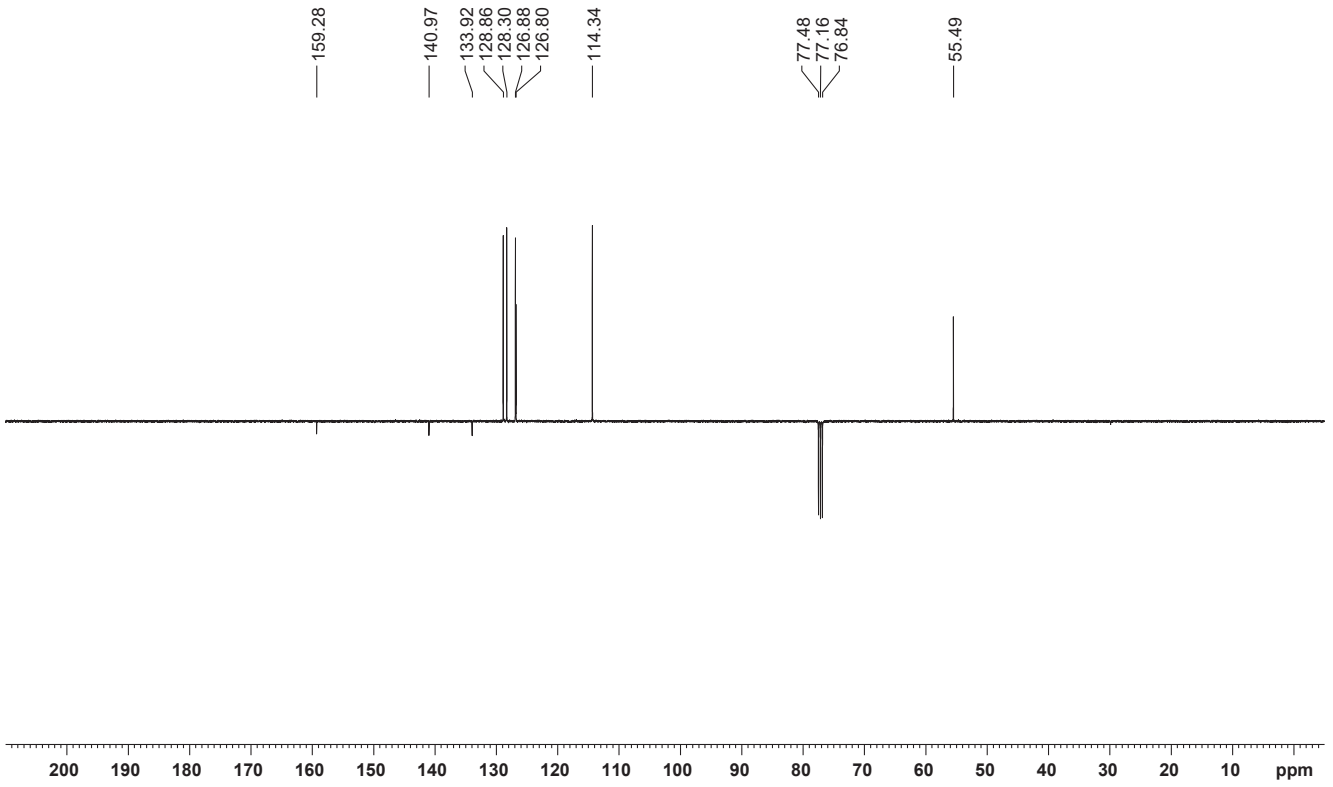


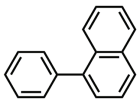
4-methoxy-1,1'-biphenyl (**7ha**)



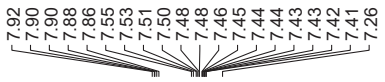


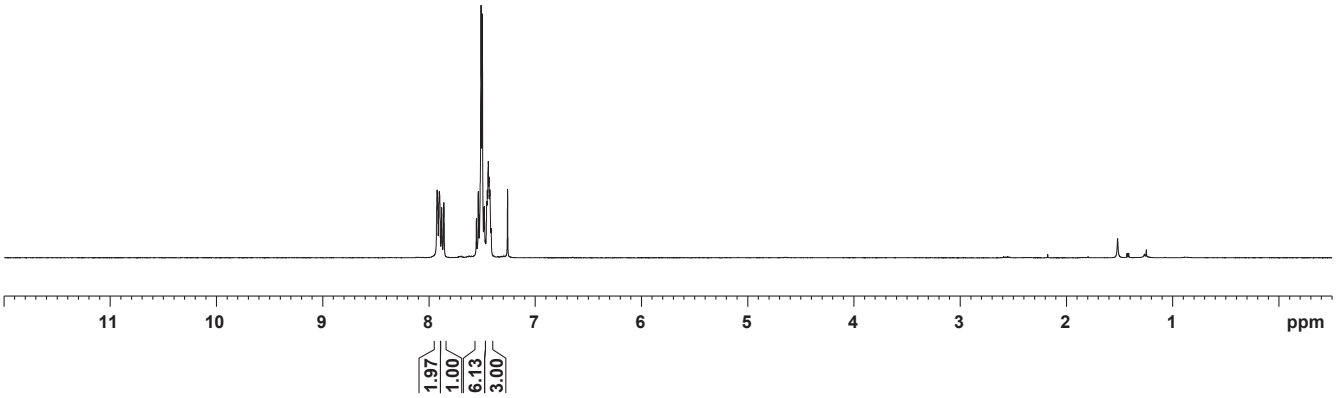
4-methoxy-1,1'-biphenyl (**7ha**)

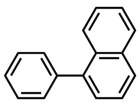




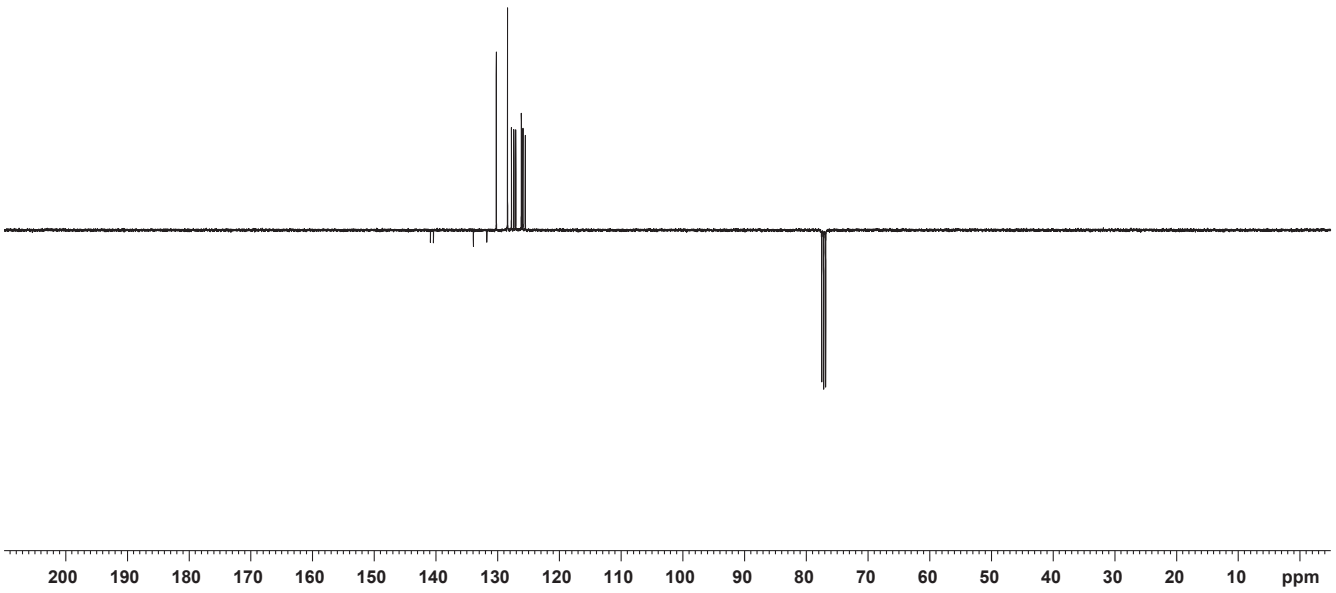
1-phenylnaphthalene (**7ia**)

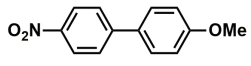




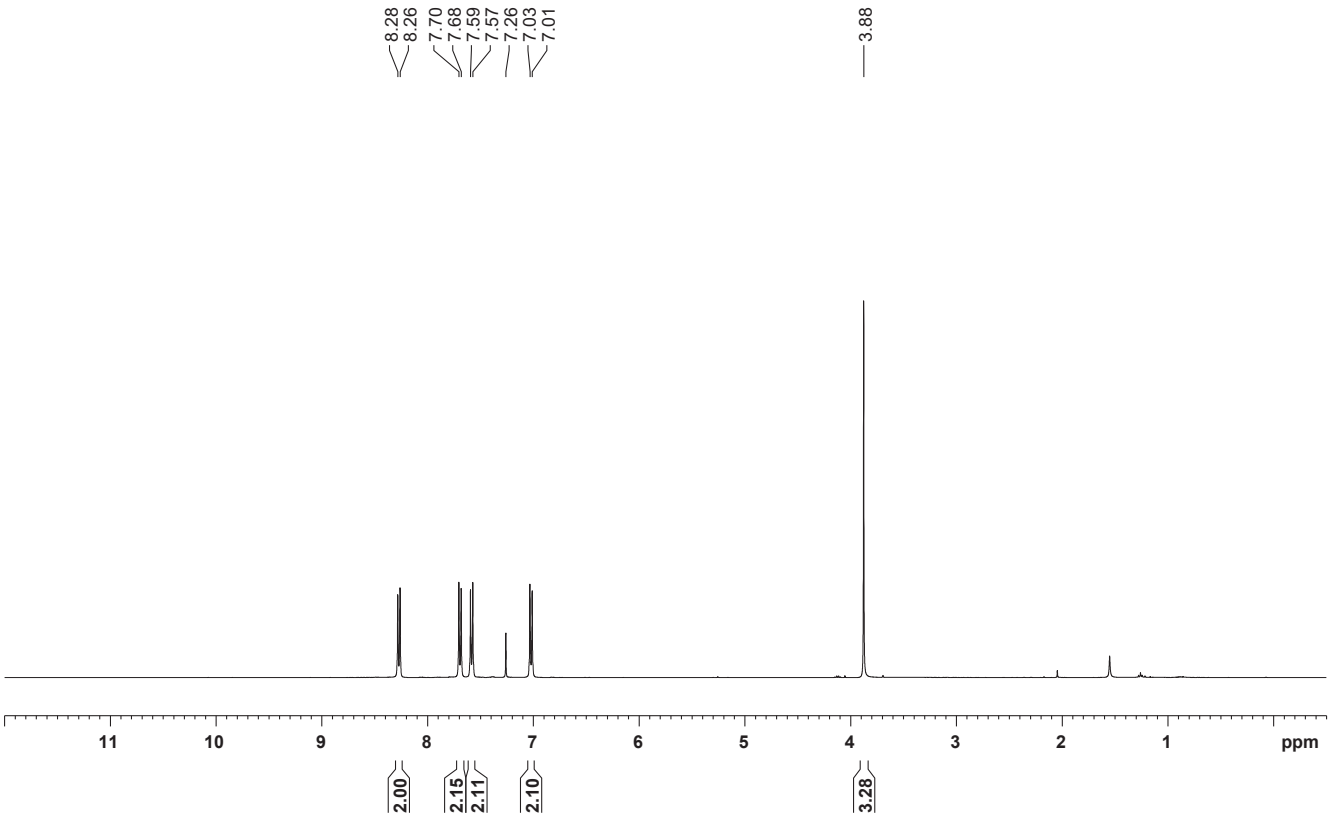


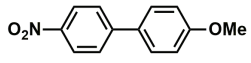
1-phenylnaphthalene (**7ia**)



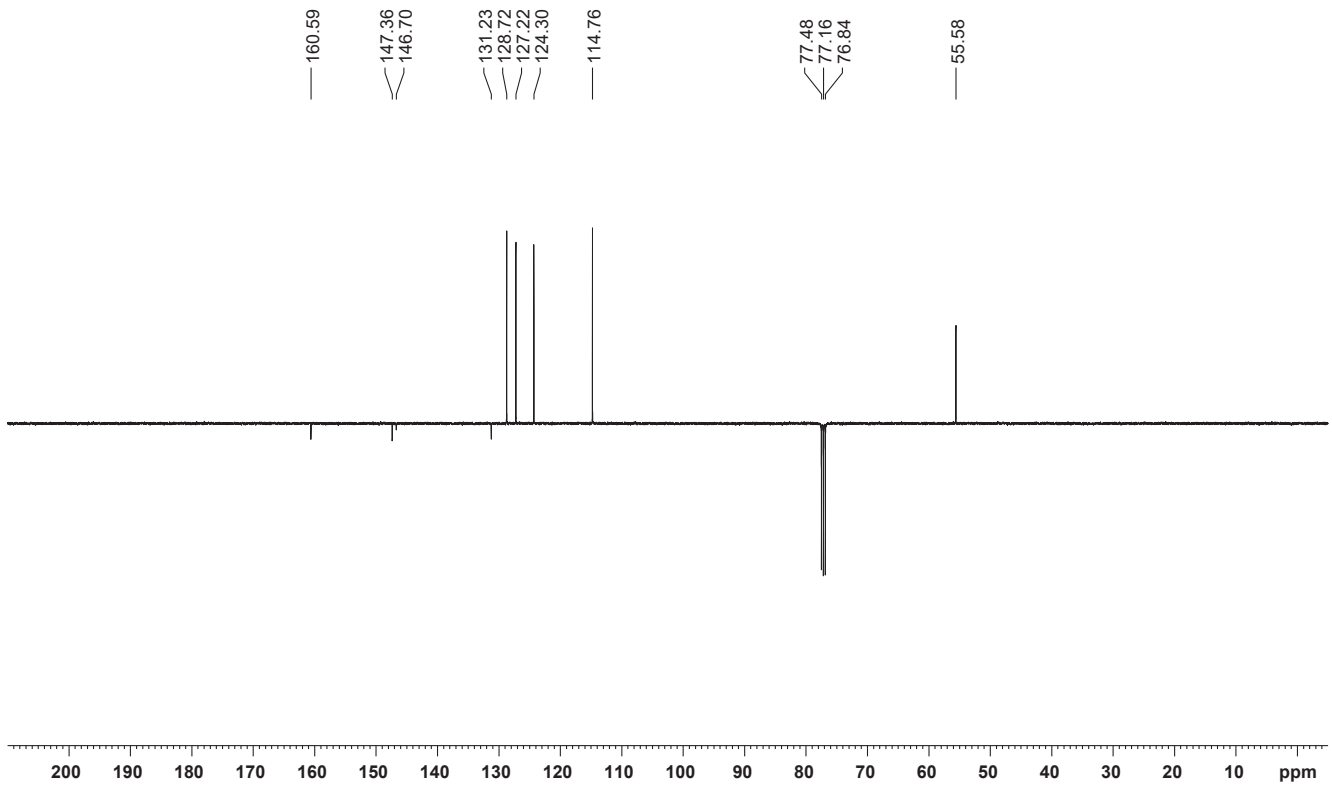


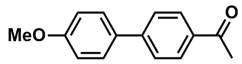
4-methoxy-4'-nitro-1,1'-biphenyl (**7eb**)



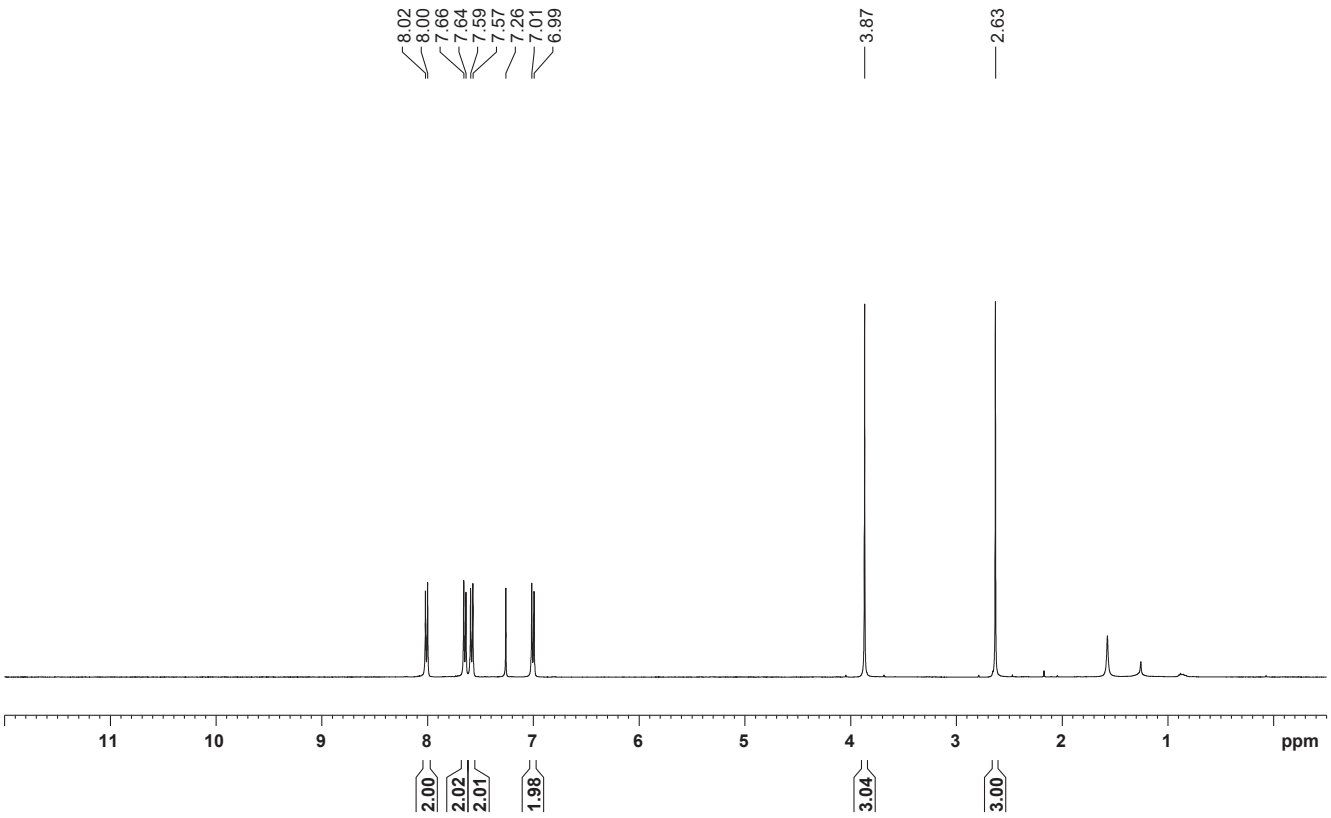


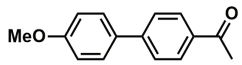
4-methoxy-4'-nitro-1,1'-biphenyl (**7eb**)



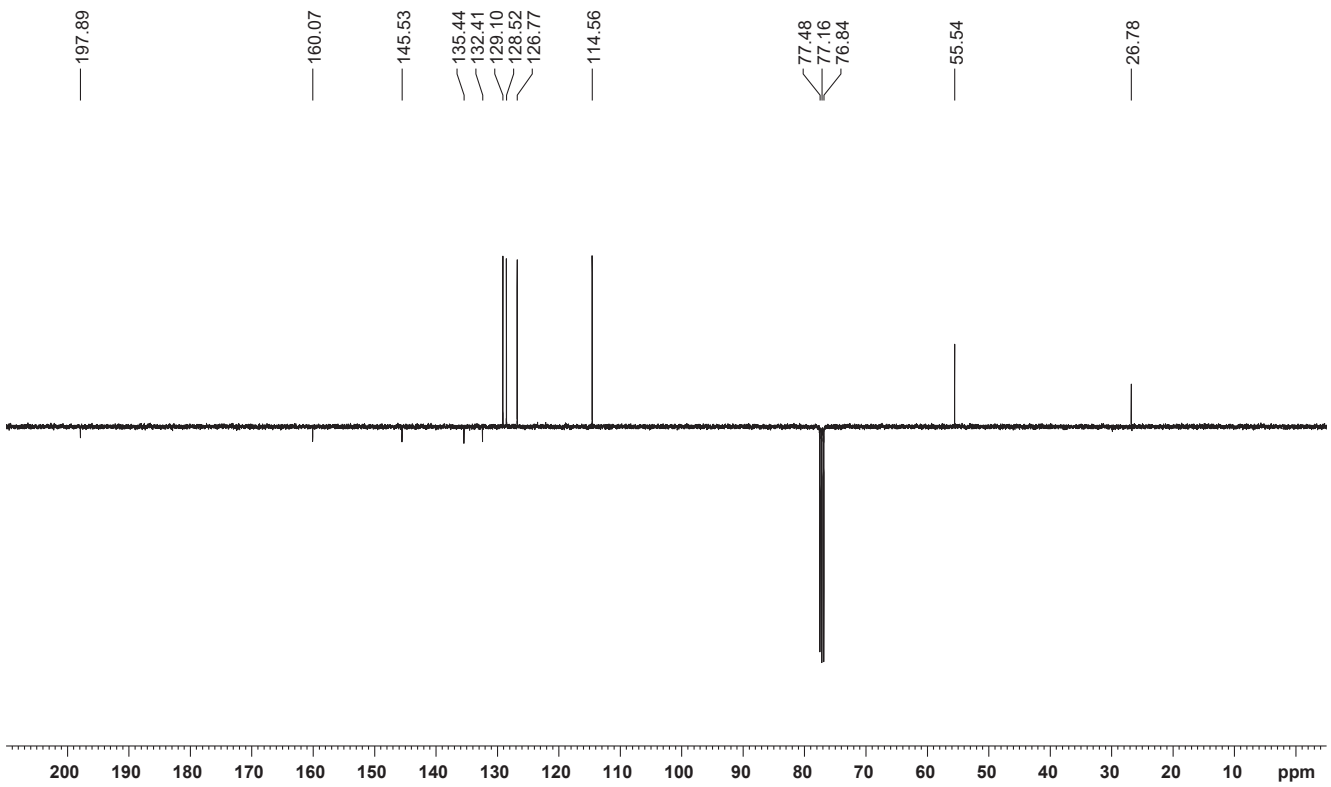


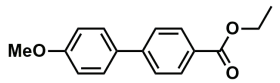
1-(4'-methoxy-[1,1'-biphenyl]-4-yl)ethanone (**7fb**)



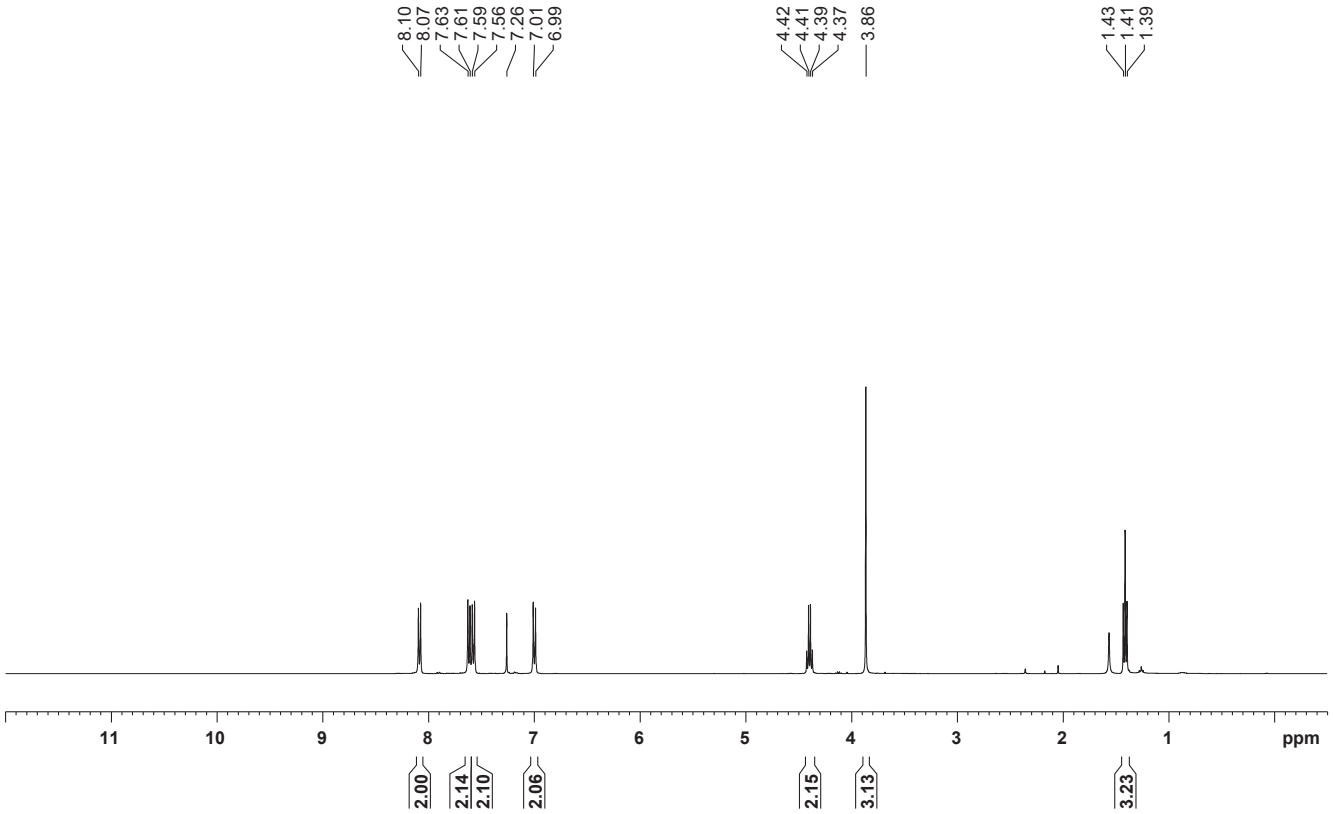


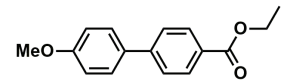
1-(4'-methoxy-[1,1'-biphenyl]-4-yl)ethanone (**7fb**)



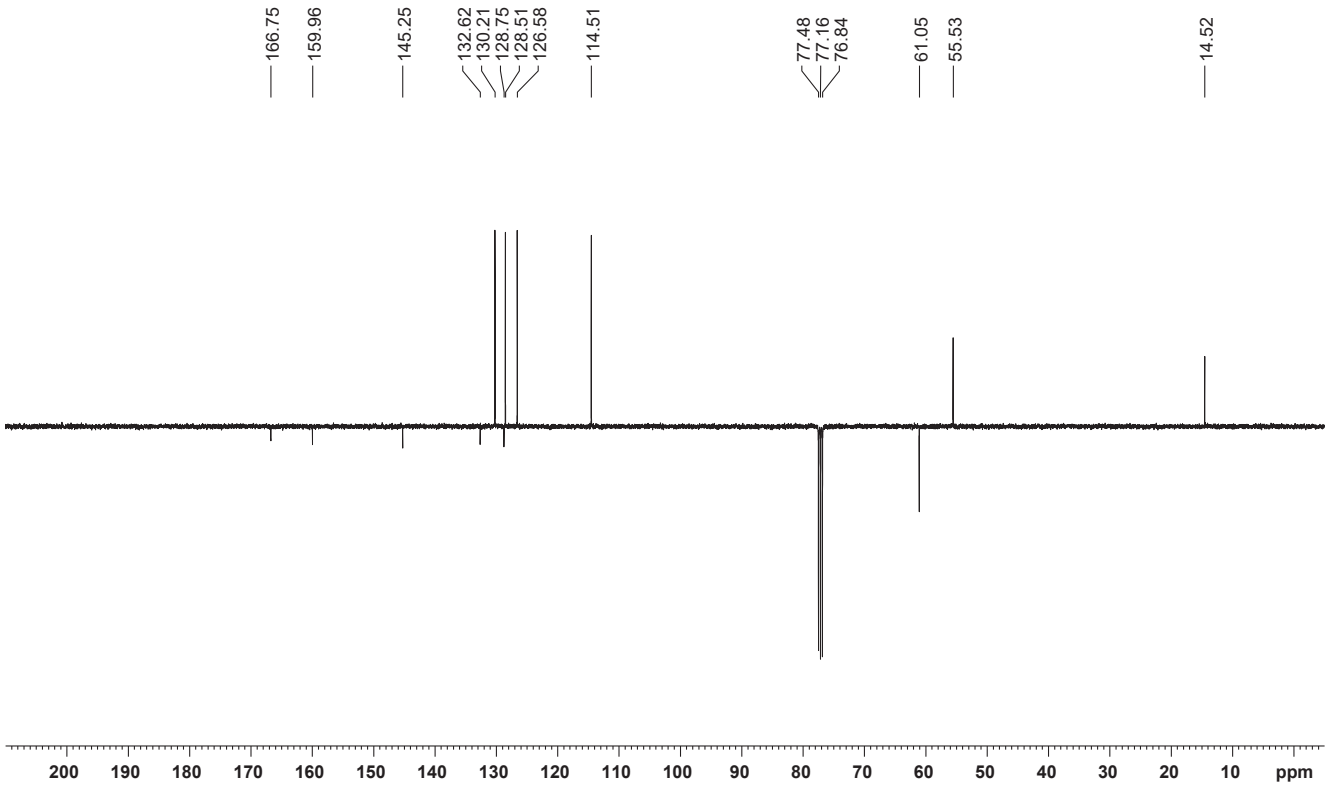


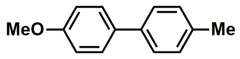
ethyl 4'-methoxy-[1,1'-biphenyl]-4-carboxylate (**7gb**)



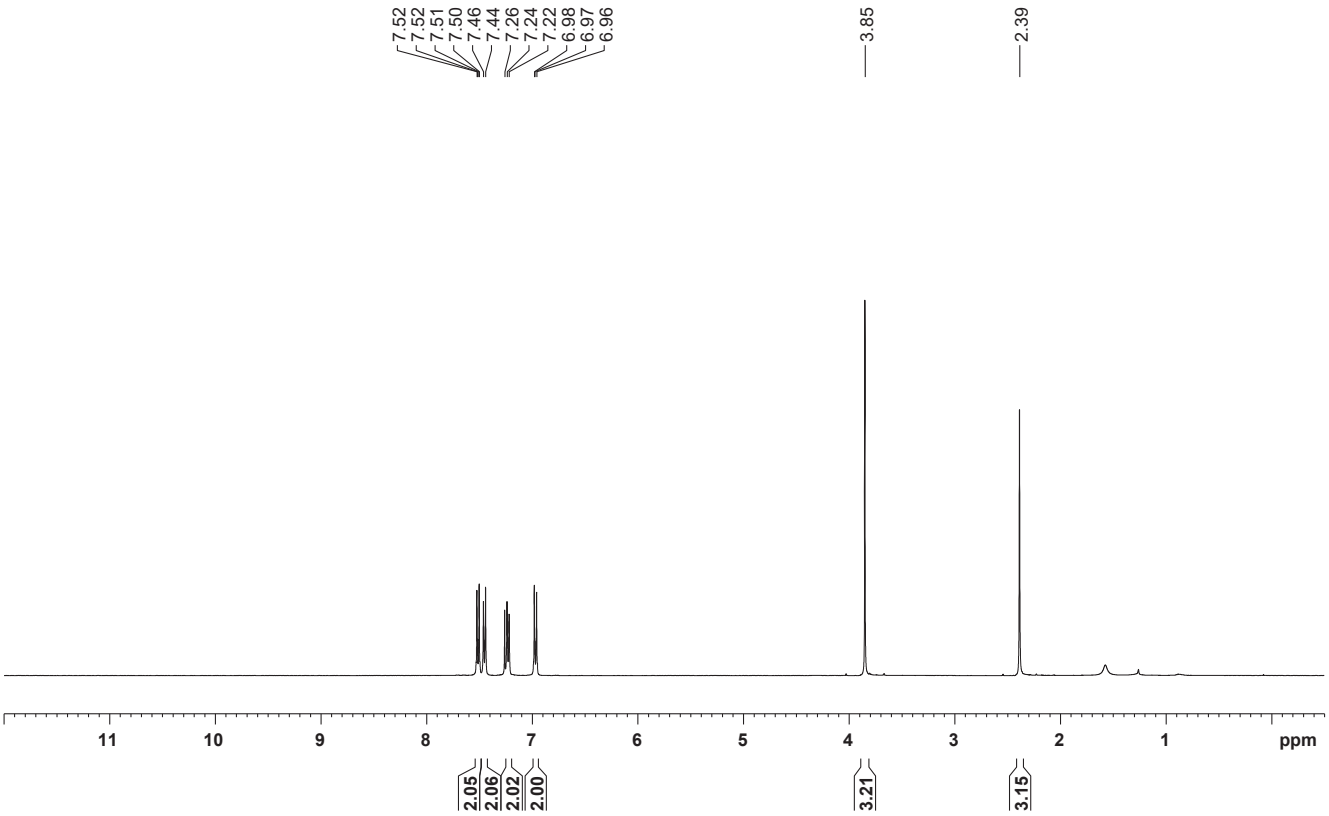


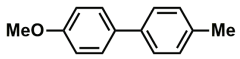
ethyl 4'-methoxy-[1,1'-biphenyl]-4-carboxylate (**7gb**)



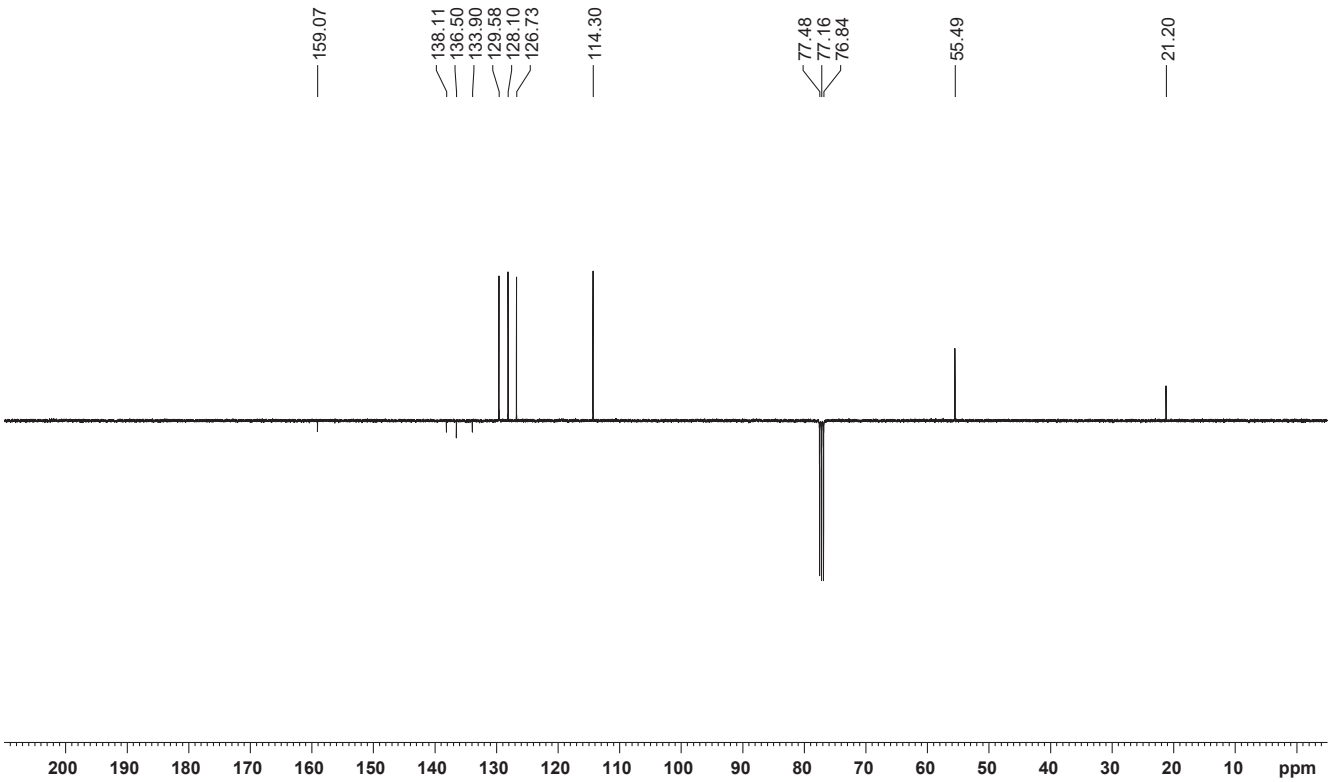


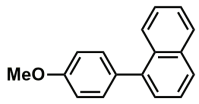
4-methoxy-4'-methyl-1,1'-biphenyl (**7db**)



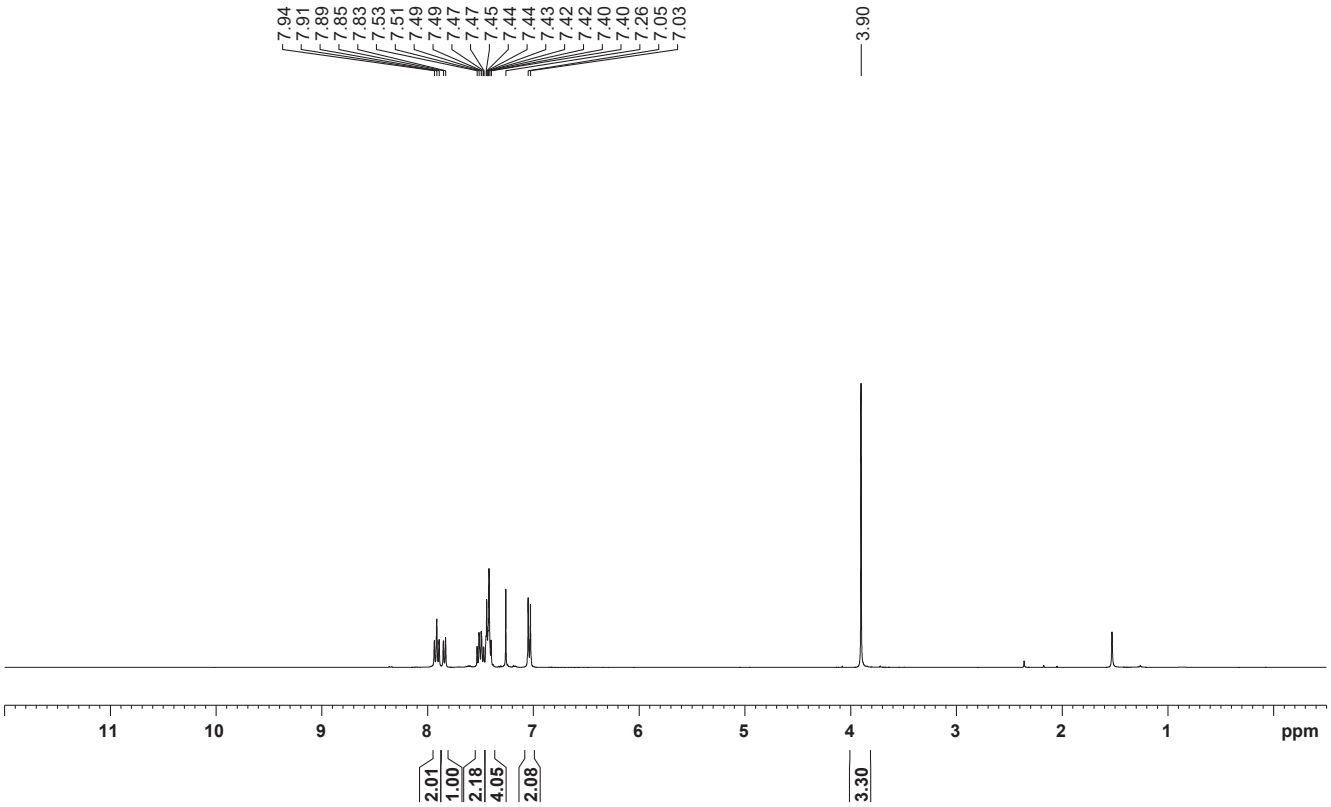


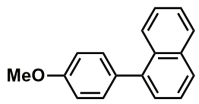
4-methoxy-4'-methyl-1,1'-biphenyl (**7db**)



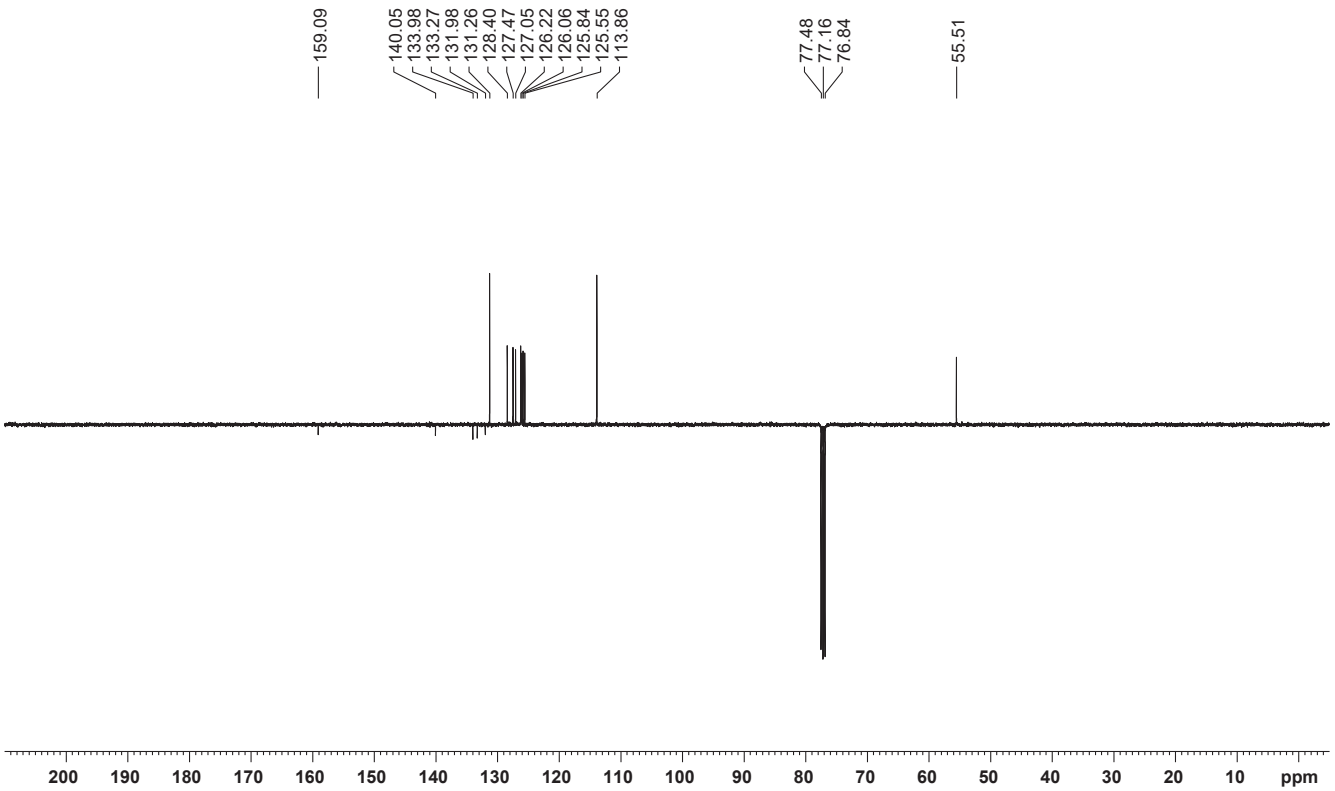


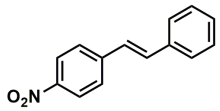
1-(4-methoxyphenyl)naphthalene (**7ib**)



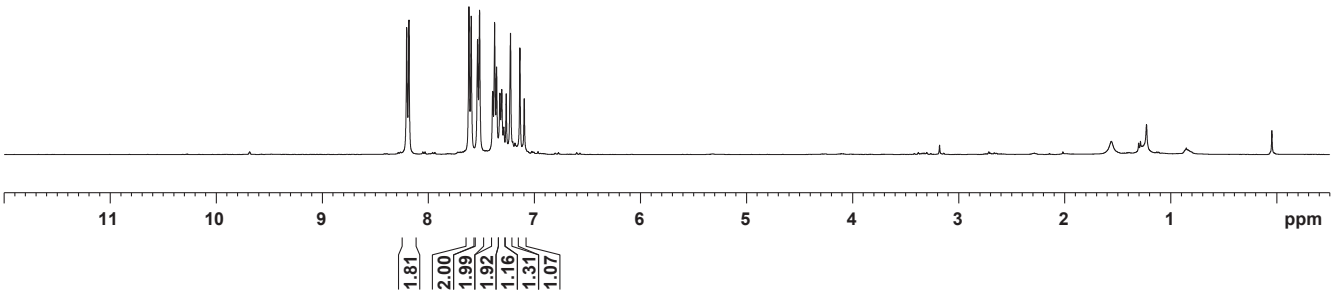


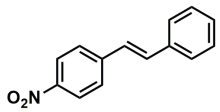
1-(4-methoxyphenyl)naphthalene (**7ib**)



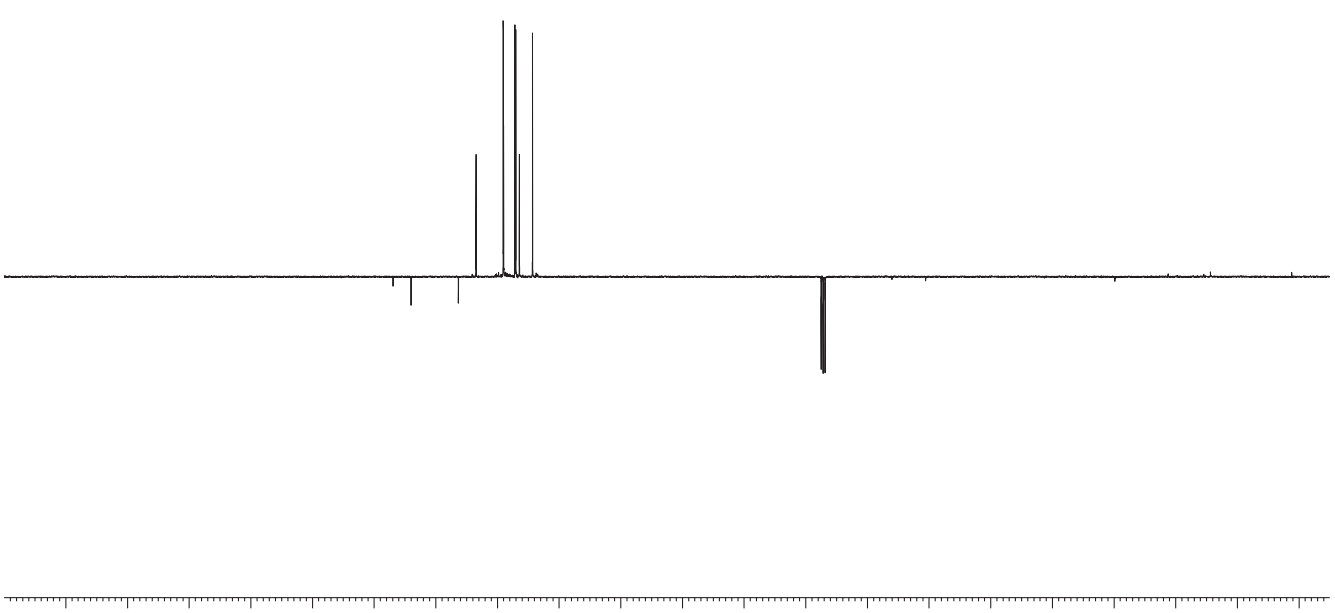


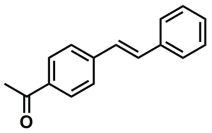
(E)-1-nitro-4-styrylbenzene (**8ea**)



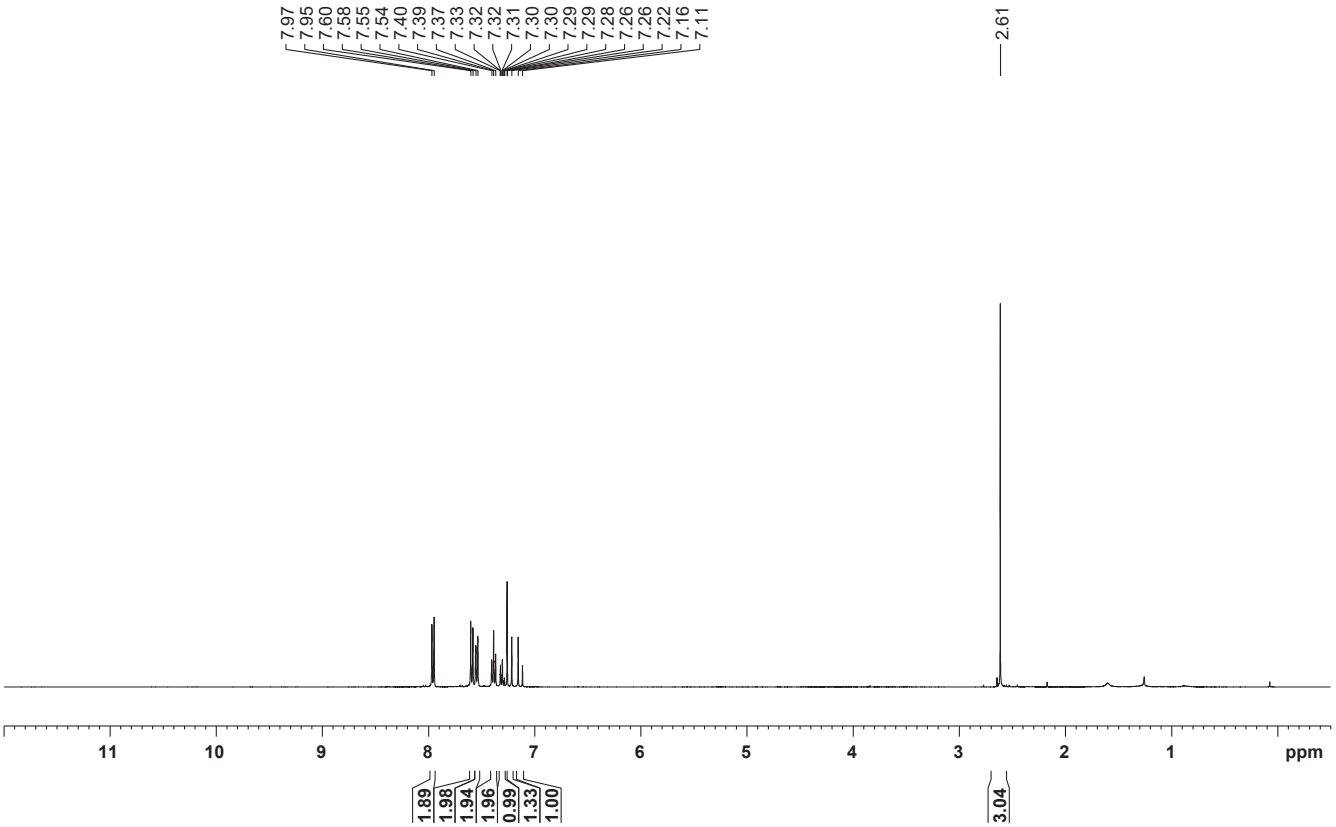


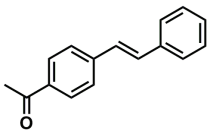
(E)-1-nitro-4-styrylbenzene (**8ea**)



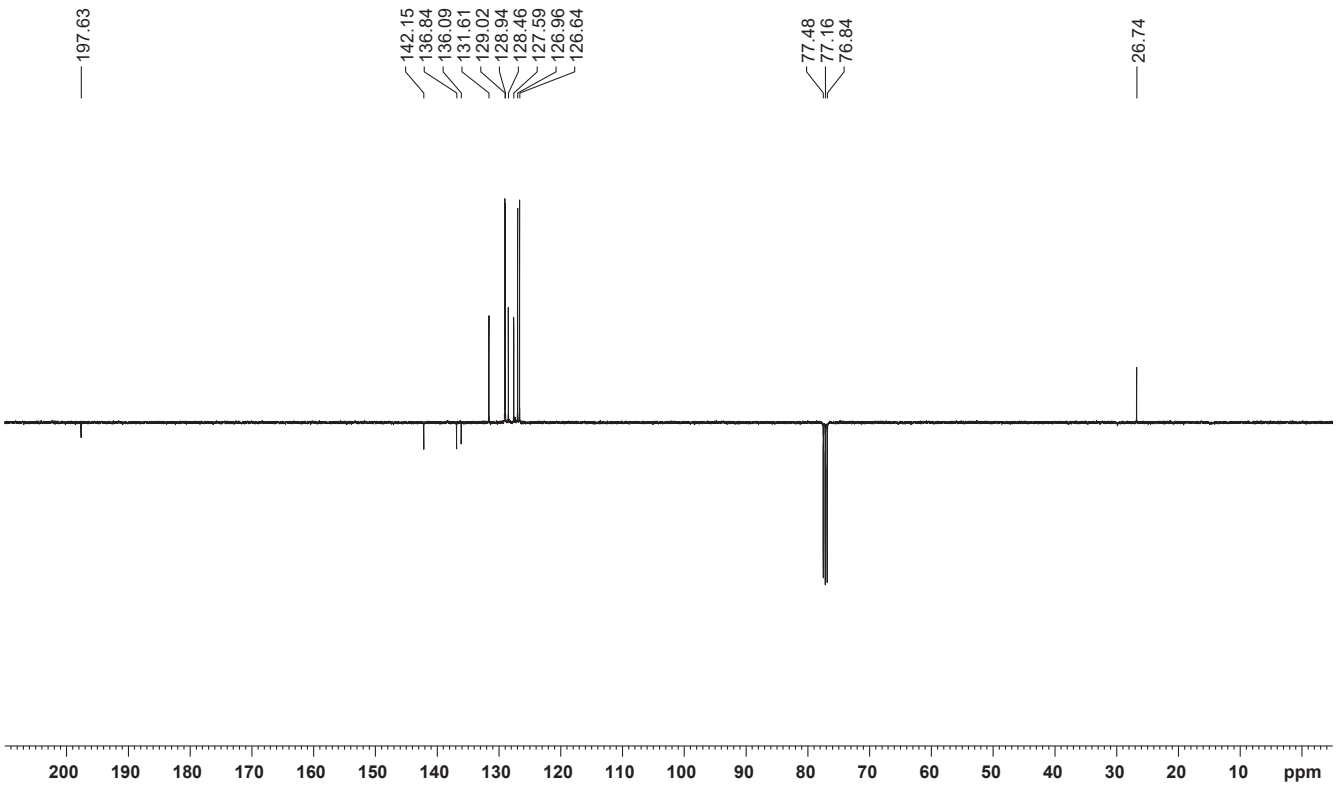


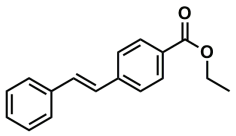
(E)-1-(4-styrylphenyl)ethanone (**8fa**)



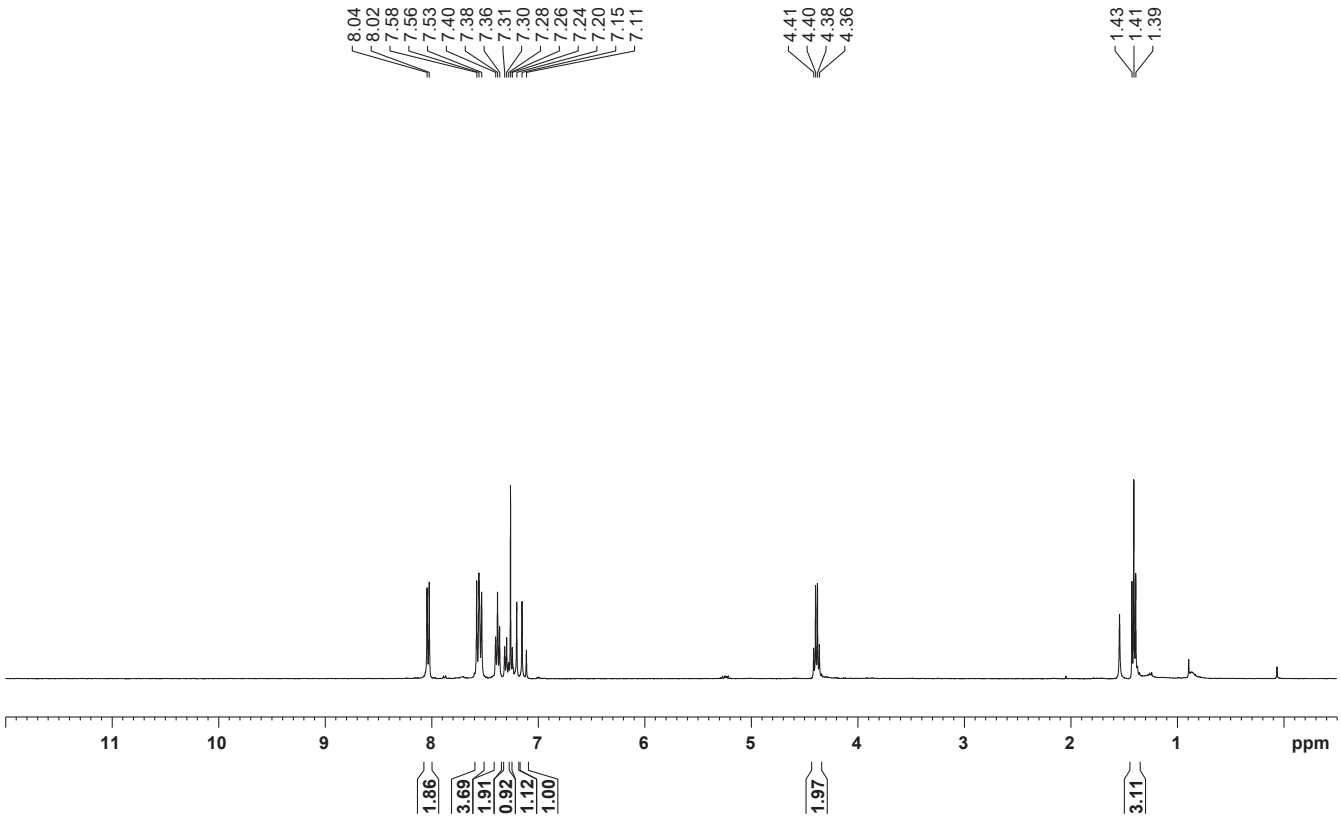


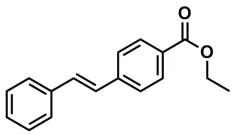
(E)-1-(4-styrylphenyl)ethanone (**8fa**)



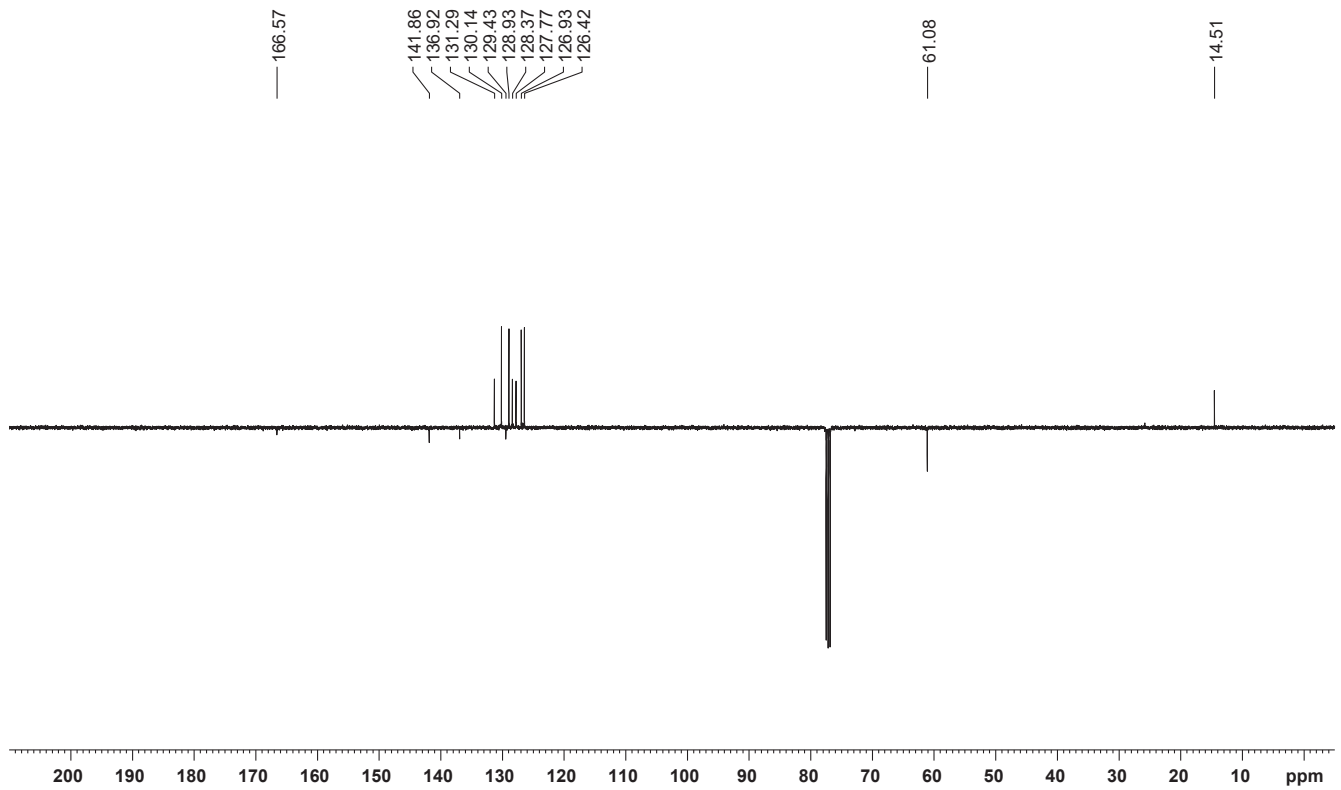


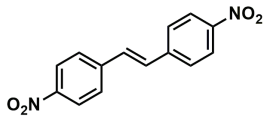
(E)-ethyl 4-styrylbenzoate (**8ga**)



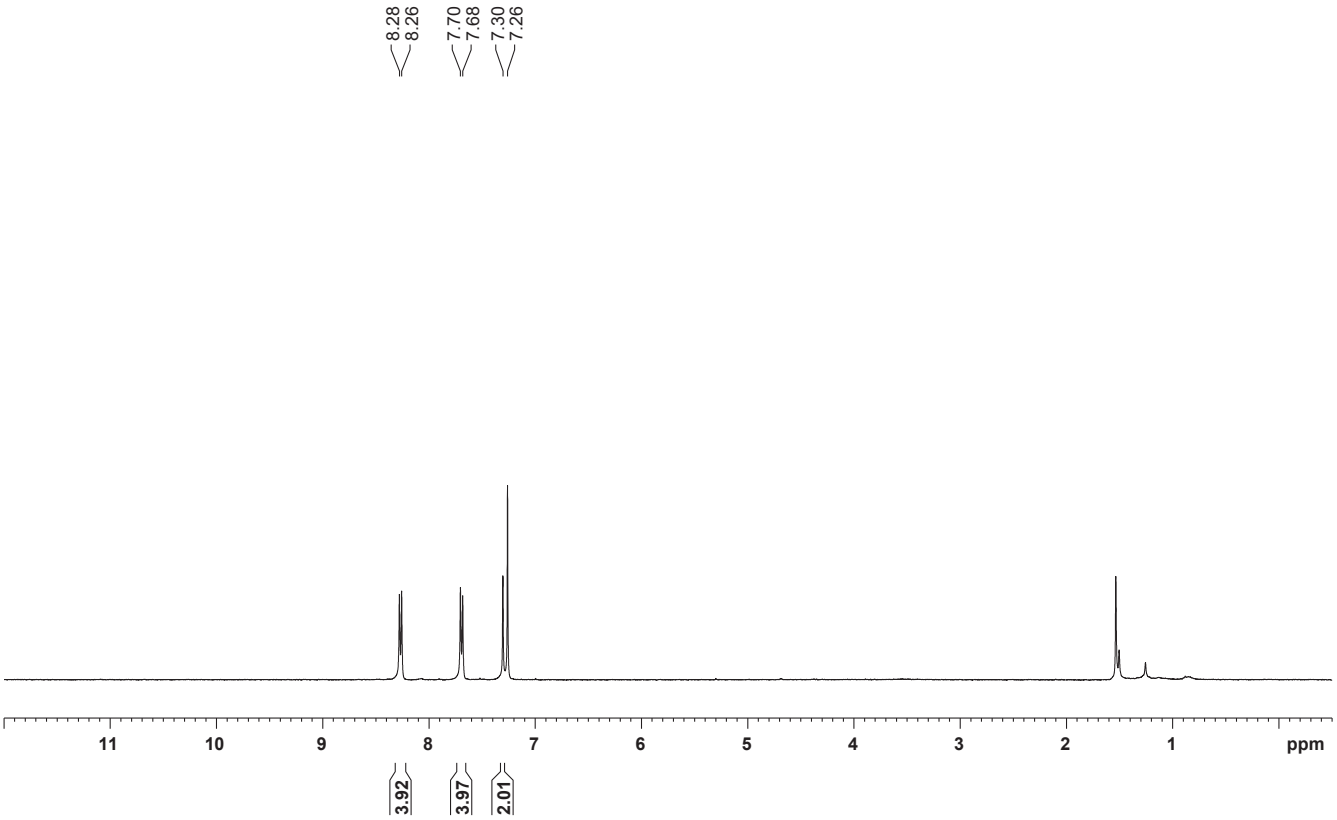


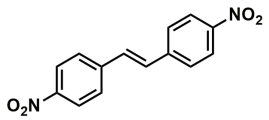
(E)-ethyl 4-styrylbenzoate (**8ga**)



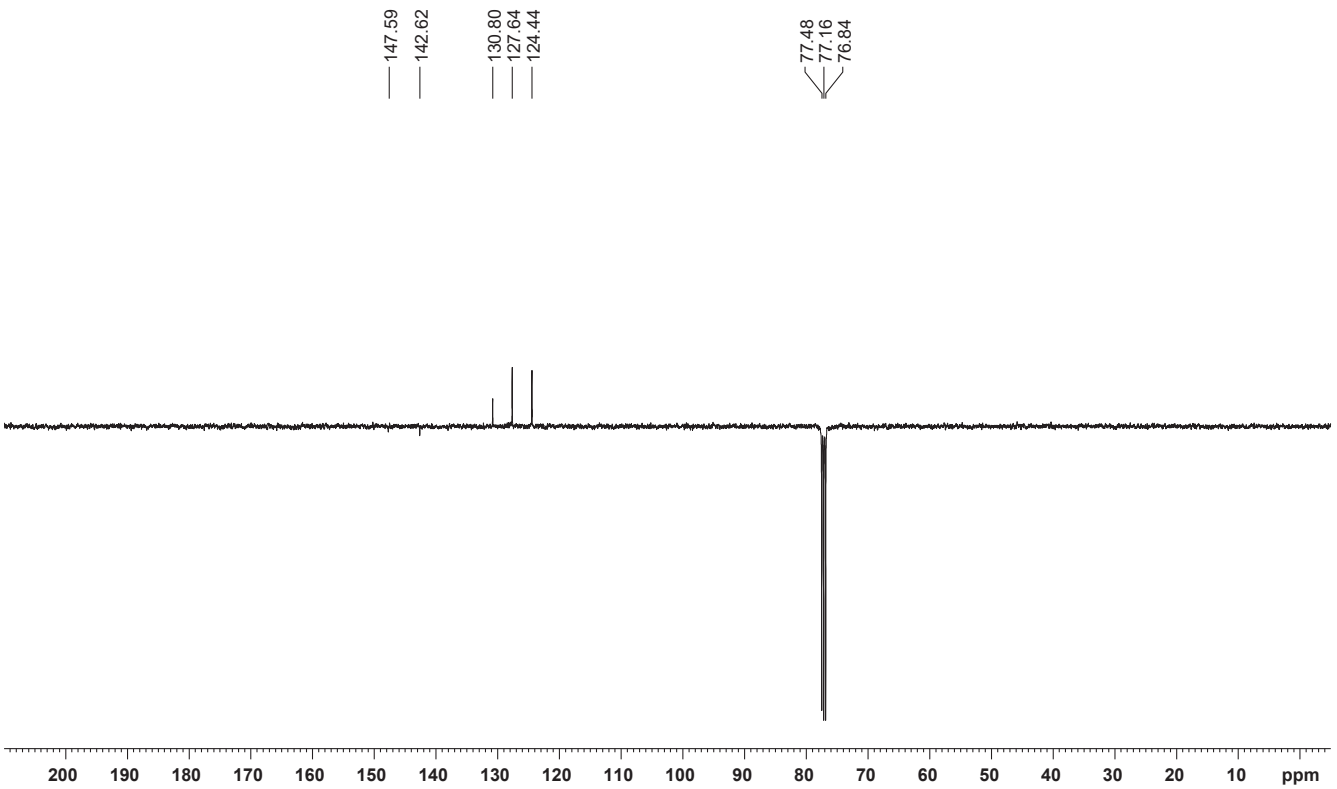


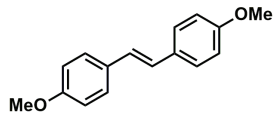
(E)-1,2-bis(4-nitrophenyl)ethene (**8jj**)



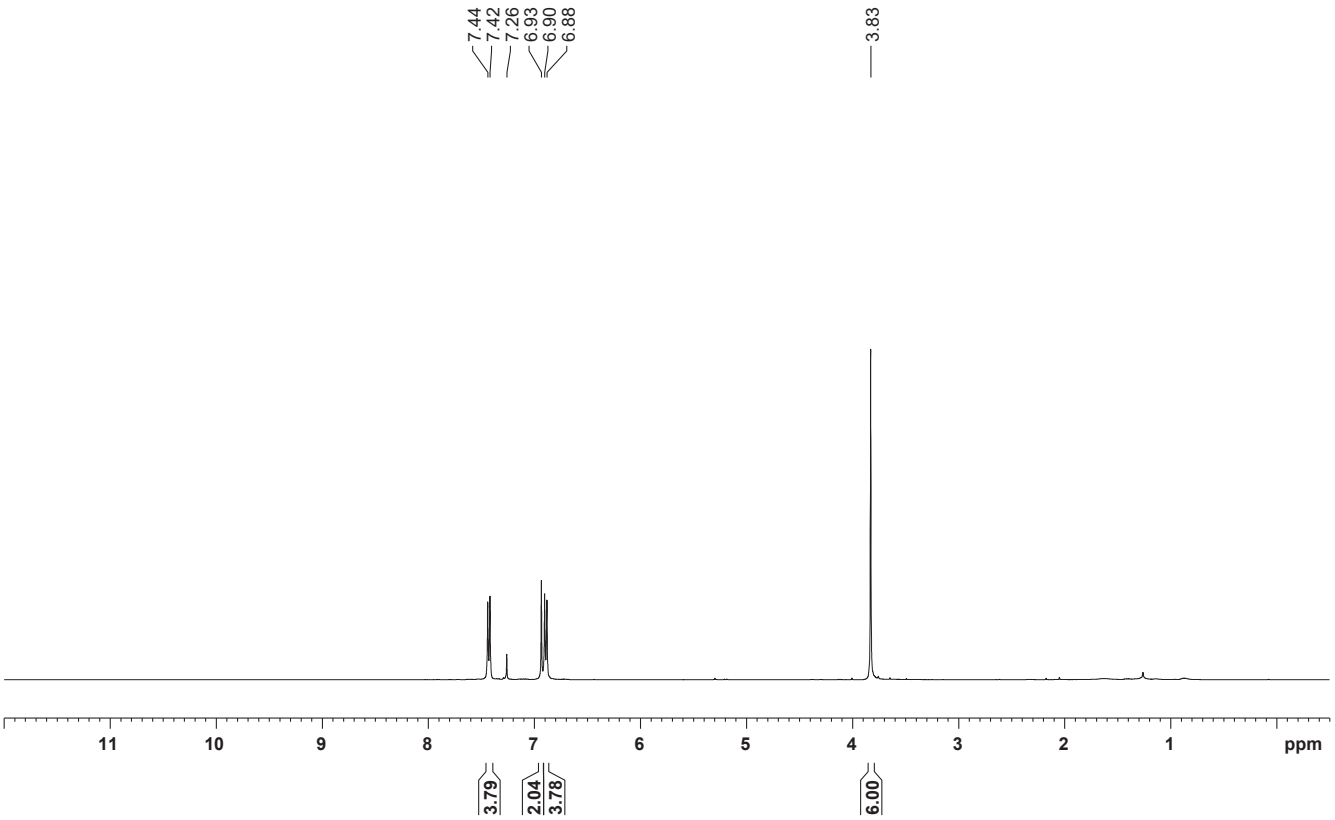


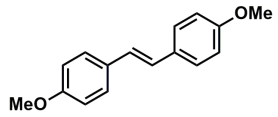
(E)-1,2-bis(4-nitrophenyl)ethene (**8jj**)





(E)-1,2-bis(4-methoxyphenyl)ethene (**8hh**)





(E)-1,2-bis(4-methoxyphenyl)ethene (**8hh**)