Supporting Information

Mechanochemical Solid State Single Electron Transfer from Reduced Organic Hydrocarbon for Catalytic Aryl-halide Bond Activation

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Table of Contents **Page No.**

1. Instrumentation and Chemicals:

All commercially available reagents were purchased from Sigma Aldrich, TCI, Alfa Aesar, and Spectrochem. All the solvents and liquid arenes were purified by distillation following the standard literature report and KO^{t}Bu was purchased from Sigma Aldrich and stored in glove box. All the Phenalenyl-based ligands were synthesized following our previous literature report (*1*).

NMR Spectroscopy:

All ^{1}H, ^{13}C, ^{19}F, and ^{11}B NMR were recorded on a Bruker (500 MHz) and Jeol (400 MHz). All chemicals shift δ were reported in ppm using TMS as a reference and J values were reported in Hz. Chemical shifts δ (ppm) are reported relative to TMS δ (^{1} H) 0.0 ppm, δ (^{13}C, 0.0 ppm). The solvent's residual proton resonance and the respective carbon resonance $CHCl_3$, δ (^{1} H) 7.26 ppm, and δ (^{13}C) 77.16 ppm were used for calibration.

TLC:

Merck TLC silica gel 60 F_{254} plates, detected by UV light or by dipping into $KMnO_4$ solution.

Column Chromatography:

All column chromatography was performed on silica gel 100-200 mesh or aluminum oxide activated (neutral).

Cyclic Voltammetry: Cyclic voltammetry was performed using a CH instrument with glassy carbon and platinum wire as electrodes, Ag/AgCl as reference electrode, and tetrabutylammonium perchlorate as an electrolyte, under an inert atmosphere.

Ball Milling Machine and Jar:

All catalytic reactions were performed with a Retsch MM400 ball milling machine and 5 mL jar, and balls with 10 mm and 7 mm diameter. The ball and jar were made of stainless steel. The model of the Ball Milling machine was (Cat. No: 20.745.0001) MIXER MILL, MODEL: MM 400 HS Code: 84.74.20.00. The model of ball milling jar was CAT No. 01.462.0231 grinding jars MM 400, screw top design stainless steel, 5 ml.

GC-MS: GC-MS experiments were performed on a Perkin Elmer Clarus 590 gas chromatography using Clarus SQ 8 S mass spectrometer.

HRMS ESI (m/z): High-resolution mass spectrometry (HRMS) was obtained on a Bruker Maxi impact spectrometer.

UV-Visible Spectrometer: UV-Visible spectroscopic measurements were performed on Jasco (V-670) spectrophotometer.

EPR: EPR spectroscopic measurements were performed on a Bruker (X-band) spectrometer with 9.155 GHz microwave frequency, 100 kHz modulation frequency, 5 mW power, and 4 min of sweep time.

ICP-OES Measurement: ICP-OES measurements were performed on the Thermo Scientific Icap 7000 SERIES ICP spectrometer. The equipment no IC74DC193405.

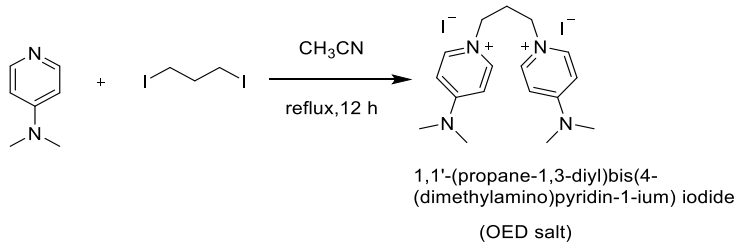
Pictorial Representation of Reaction Setup

(a) **(b)** **(c)**

Fig. 1: a) Retsch MM400 mixer mill used for electron transfer catalysis reactions. **b)** Ball Milling jar (5 mL) and balls (diameter 10 mm) for this reaction. **c)** Mortar pestle for the mechanistic study or solid-solid catalysis.

2. **Synthesis of OED salt (1,1'-(propane-1,3-diyl)bis(4-(dimethylamino)pyridin-1-ium) iodide):**

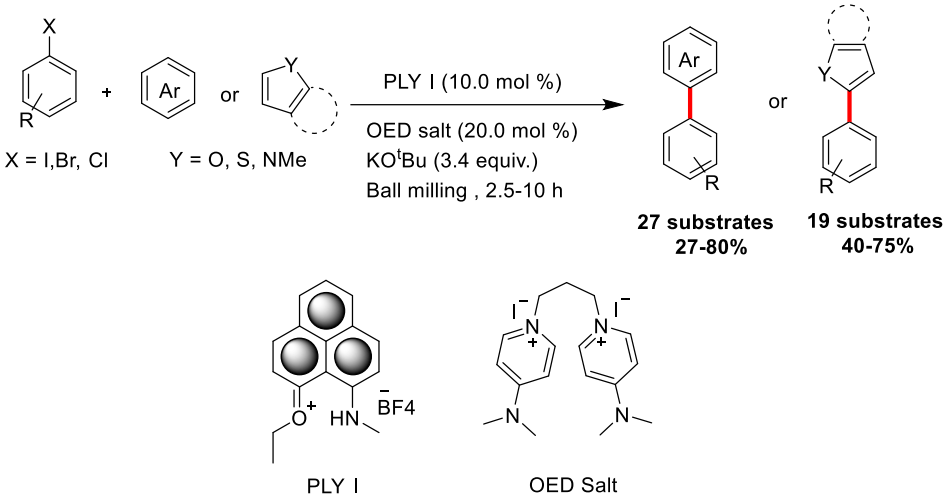
A flame-dried round bottom flask equipped with a magnetic stir bar was charged with 4-DMAP (2.0 g, 8.18 mmol, 2.5 eq.) and 1,3-diiodopropane (1.94 g or 0.753 mL, 6.57 mmol, 1.0 equiv.), and acetonitrile (40.0 mL). The reaction mixture was refluxed overnight under argon for 12 h. After completion of the reaction, the reaction mixture was cooled down and the solid was precipitated out which was further filtered. The solid was washed with acetonitrile (4 x 30 mL) and with diethyl ether (4 x 30 mL) and dried under vacuum to afford 1,3-bis(*N'*, *N'*-dimethyl-4-aminopyridinium)propane diiodide (1.33 g, 75% yield) as a white solid. The characterization data was matched with the previous literature report (*2*).



Scheme S1: Syntheis of OED Salt

3. General Procedure for Arylation, Borylation, Heck-type Coupling, Trifluoromethylationlaion

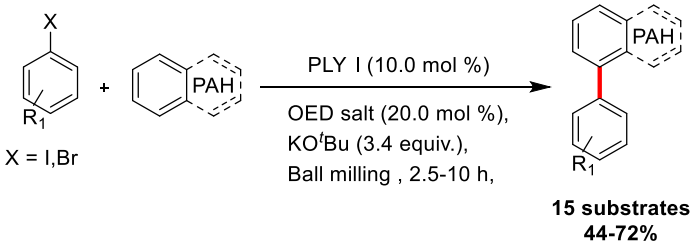
➢ General Arylation Procedures



Scheme S2: Arylation Reaction

A flame-dried ball mill jar equipped with two stainless steel balls was charged with OED salt (32.4 mg, 0.06 mmol) and KO^{t}Bu (14 mg, 0.12 mmol) inside a glove box. Subsequently, the mixture was grinded using a ball milling machine for 30 min. After that, the jar was again taken inside the glove box and PLY I (10 mol%, 9.75 mg, 0.03 mmol), aryl halide (0.3 mmol), heteroarene (10.0 equiv., 3.0 mmol) and KO^{t}Bu (3.0 equiv., 102 mg, 0.9 mmol) were added. Next, the reaction mixture was grinded for 2.5-10 h, outside the glove box. After completion of the reaction, the resultant mixture was extracted with 50-100 mL dichloromethane (DCM). The solvent was removed under reduced pressure, and the desired product was purified by column chromatography using 100-200 mesh silica gel and hexane/EtOAc mixture as eluent.

➢ General Reaction Procedure with Polyarenes

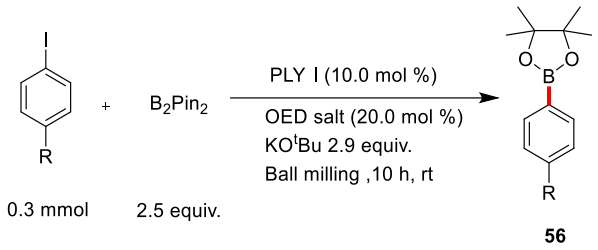


Scheme S3: Polyarylation Reaction

A flame-dried ball mill jar equipped with two stainless steel balls were charged with OED salt (20 mol %, 43.2 mg, 0.08 mmol) and KO^{t}Bu (40 mol %, 18.0 mg, 0.16 mmol) inside a glove box. Subsequently, the mixture was grinded using ball milling machine for 30 min. Thereafter, the jar was again taken inside the glove box and PLY I (10 mol %, 13.0 mg, 0.04 mmol), aryl

halide (0.4 mmol), polyarene (5.0-7.0 equiv., 2.0-2.8 mmol), DMSO as LAG (0.12 µL/mg) and KO^{t}Bu (3.0 equiv., 135 mg, 1.2 mmol) were added and subsequently, the reaction mixture was grinded for 2.5-10 h, outside the glove box (ball milling).

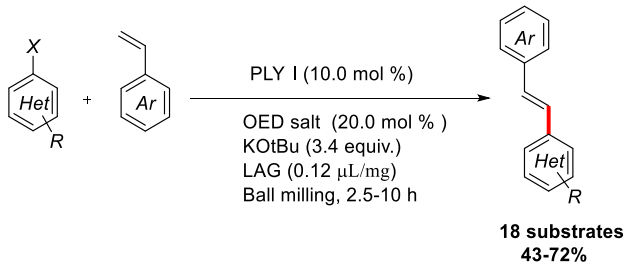
- **General Procedure for Borylation**



Scheme S4: Borylation Reaction

A flame-dried ball mill jar equipped with two stainless steel balls was charged with OED salt (20 mol %, 43.2 mg, 0.08 mmol) and KO^{t}Bu (40 mol %, 18.1 mg, 0.16 mmol) inside a glove box. Subsequently, the reaction mixture was grinded using a ball milling machine for 30 min. Thereafter, the jar was again taken inside the glove box and PLY I (10 mol %, 13.0 mg, 0.04 mmol), aryl halide (0.4 mmol), B_2Pin_2 (2.5 equivalent, 253.0 mg, 1.0 mmol) and KO^{t}Bu (2.5 equivalent, 113.0 mg, 1.0 mmol) were added and subsequently, the reaction mixture was grinded for respected hours at outside the glove box. After completion, the reaction mixture was passed through a short silica gel column eluted with hexane and ethyl acetate. The crude material was purified by column chromatography (silica, hexane/ethyl acetate) to give the borylated product.

- **General Heck-type Coupling Procedures**

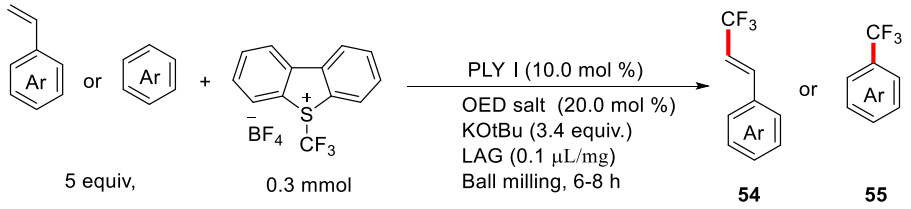


Scheme S5: Heck-type Coupling

A flame-dried ball mill jar equipped with two stainless steel balls was charged with OED salt (20 mol %, 32.4 mg, 0.06 mmol) and KO^{t}Bu (40 mol %, 14.0 mg, 0.16 mmol) inside a glove box. Subsequently, the mixture was grinded using ball milling machine for 30 min. After that, the jar was again taken inside the glove box and PLY I (10 mol %, 9.7 mg), Aryl halide (0.3 mmol), Aryl styrene (5.0 equiv., 2.0 mmol) and KO^{t}Bu (3.4 equivalent, 115.0 mg mg, 1.0 mmol), DMSO as LAG (0.12 µL/mg) were added and the reaction mixture was grinded for respected hours at outside the glove box. After completion, the reaction mixture was passed

through a short silica gel column eluting with hexane and ethyl acetate (EtOAc). The crude material was purified by column chromatography on silica gel (hexane/ethyl acetate) to give the desired product.

- **General Procedure for Trifluoromethylation of Arenes and Styrenes**



Scheme S6: Trifluoromethylation Reaction

A flame-dried ball mill jar equipped with two stainless steel balls charged with OED salt (20 mol%, 32.4 mg, 0.06 mmol) and KO^{t}Bu (40 mol %, 14 mg, 0.12 mmol) inside a glove box and grinded the mixture in a ball milling machine for 30 min. Thereafter, the jar was again taken inside the glove box and PLY I (10 mol %, 9.7 mg, 0.03 mmol), Umemoto's reagent (0.3 mmol, 1 equiv.), styrene (1.5 mmol, 5.0 equiv.) or arene (1.5 mmol, 5.0 equiv.), KO^{t}Bu (3.0 equiv., 101mg), DMSO as LAG (0.1 μL/mg) was added and subsequently, the reaction mixture was grinded for respected hours outside the glove box. After completion, the reaction mixture was passed through a short silica gel column eluting with hexane and ethyl acetate (EtOAc). The crude material was purified by column chromatography (silica, hexane/ethyl acetate) to give the desired product.

4. Pictorial Representation of the Reaction Setup

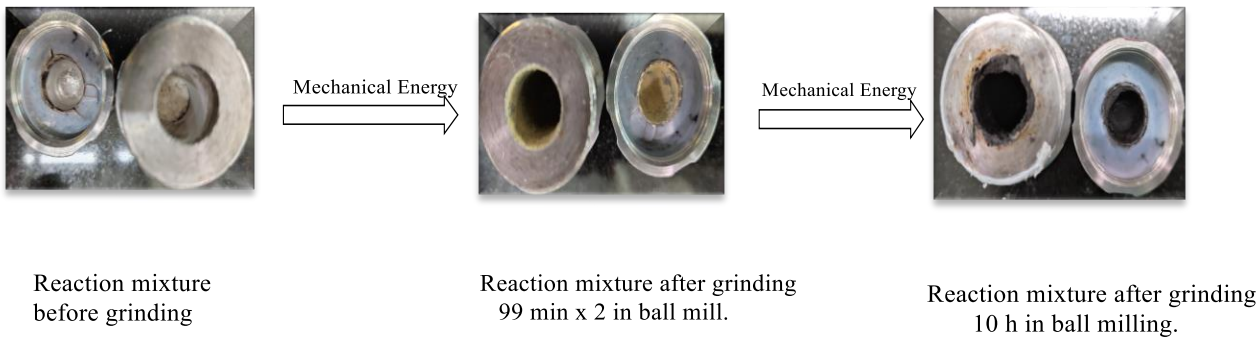


Fig. 2: Representative Procedure of Solid-State Reaction in Ball Milling Condition

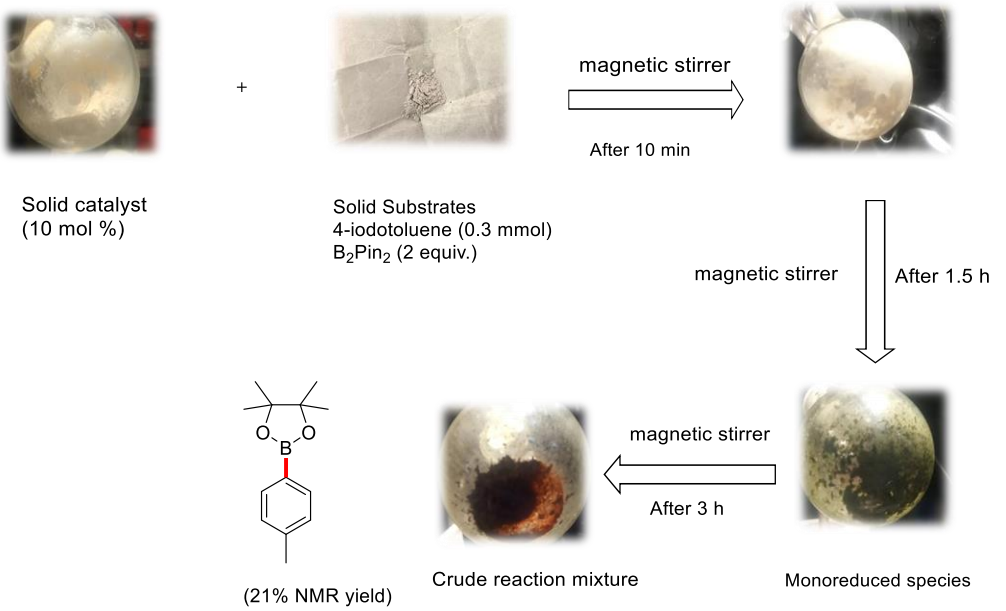


Fig. 3: Representative Procedure of Solid-State Coupling using Magnetic Stirrer

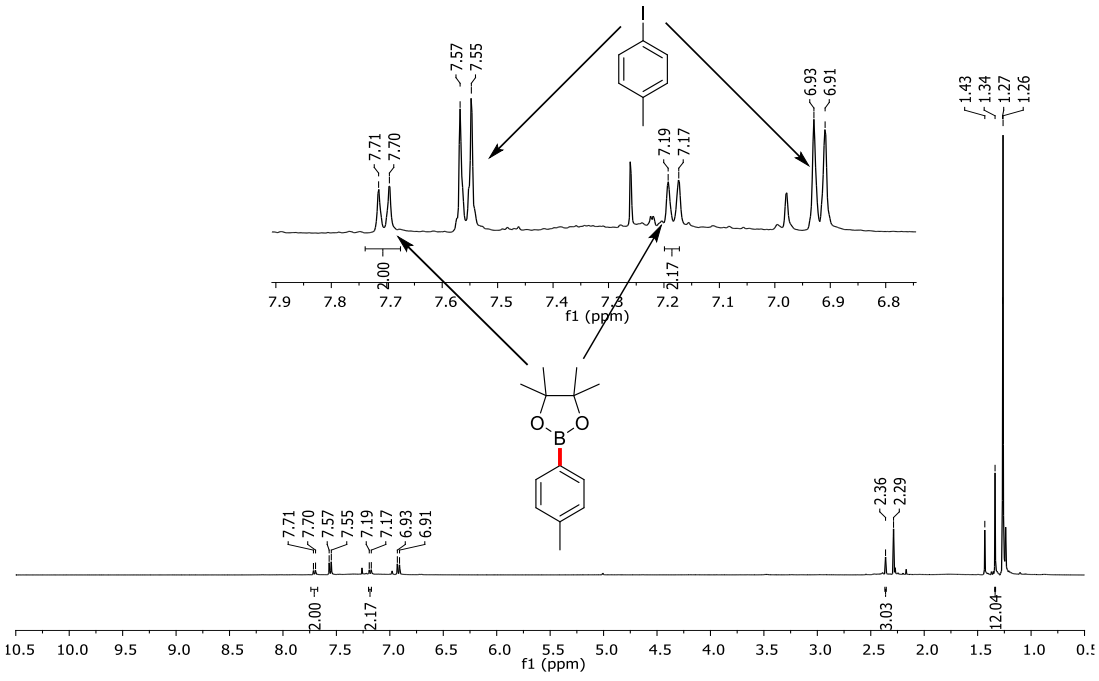


Fig. 4: ^{1}H NMR of the Crude Reaction Mixture

Representative Procedure of Solid-State Reaction in Mortar Pestle

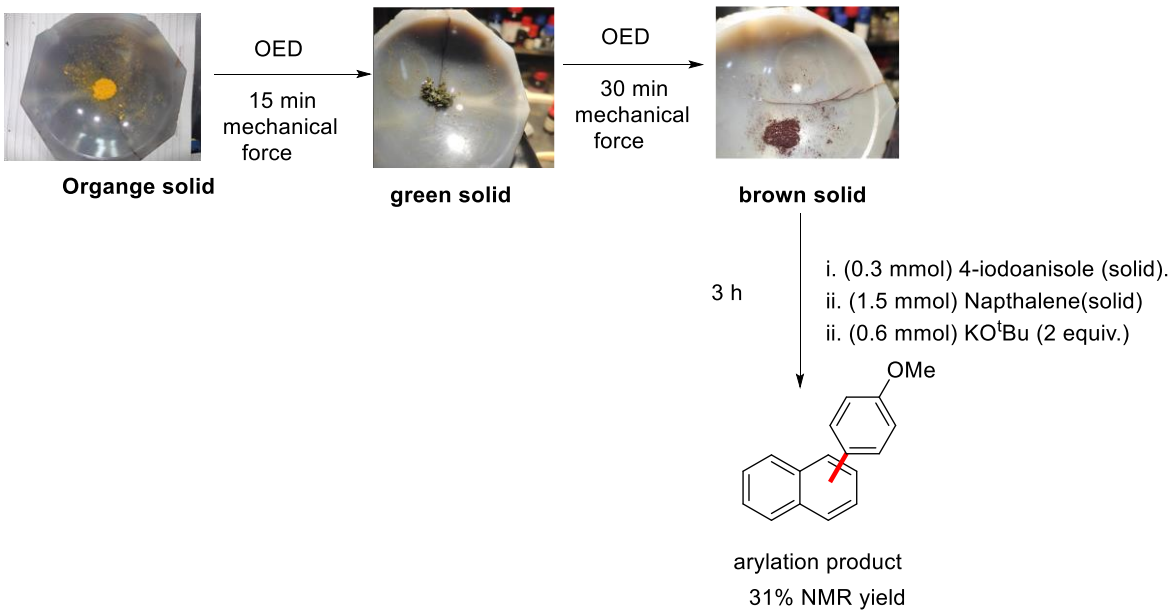


Fig. 5: Graphical Representation of Electron Transfer Catalysis in Solid State using Mortar Pestle

5. Large Scale Synthesis of Direct Coupling

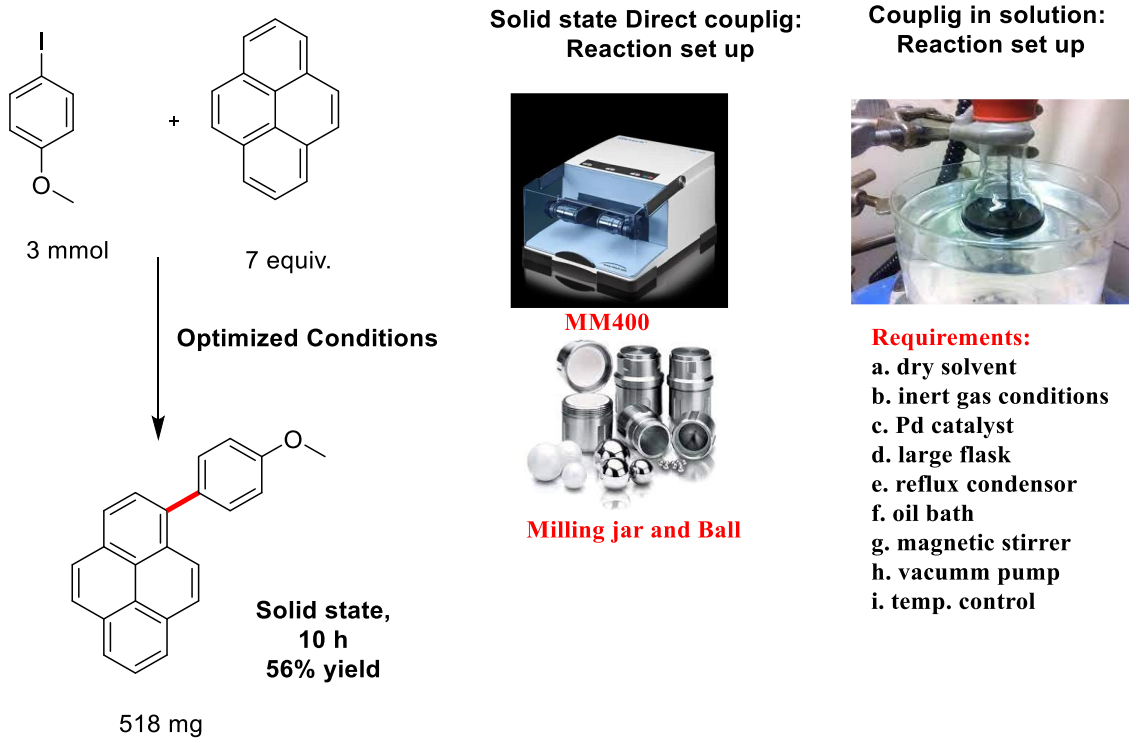
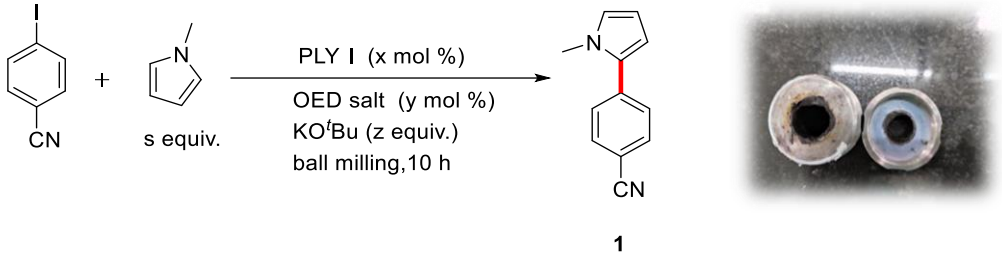


Fig. 6: Large-Scale Synthesis of Solid-Solid Poly-Arylation Product

A flame-dried ball mill jar equipped with two stainless steel balls were charged with OED salt (20 mol %, 324 mg, 0.6 mmol) and KO^{t}Bu (40 mol %, 135.0 mg, 1.2 mmol) inside a glove box and grinded the mixture using ball milling machine for 30 min. Thereafter, the jar was again taken inside the glove box and PLY I (10 mol %, 98.0 mg), 4-iodoanisole (3 mmol, 702.0 mg), pyrene (7 equiv., 4.24 g, 12.0 mmol) and KO^{t}Bu (3.0 equiv., 9.0 mmol) were added and subsequently, the reaction mixture was grinded for 10 h outside the glove box. After completion of the reaction, the mixture was extracted with 200-300 mL dichloromethane (DCM). The solvent was removed under reduced pressure, and the crude product was purified by column chromatography using 10-200 mesh silica gel and hexane/EtOAc mixture as eluent. After that we got desired product 56% yield (518 mg) in white solid.

6. Optimisation Studies



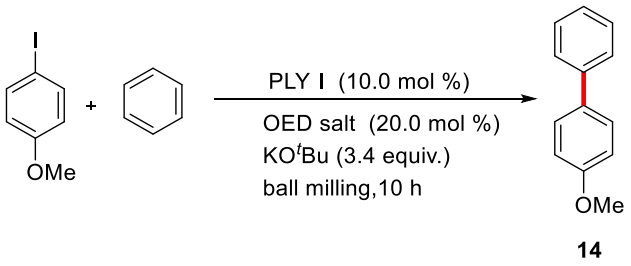
Scheme S7: General Optimization Studies with Heteroarenes

Table S1: Optimisation Studies with Heteroarene

Entry	4-iodobenzonitrile (mmol)	N-methyl-pyrole (s equiv.)	(N,O)PLY cation (x mol %)	OED salt (y mol %)	KO^{t}Bu (z equiv.)	Yield (%)
1.	0.3	1	5	10	1.4	19
2.	0.3	2	10	20	2.4	31
3.	0.3	5	10	20	3.4	41
4.	0.3	7	10	20	3.4	61
5.	**0.3**	**10**	**10**	**20**	**3.4**	**72**
6.	0.3	10	-	20	3.4	18
7.	0.3	10	10	-	3.4	<5
8.	0.3	10	10	20	-	<1

Conditions: 4-Iodobenzonitrile, 1-methyl-1*H*-pyrrole, PLY I, OED salt, KO^{t}Bu, were taken under inert conditions inside in a 5 ml jar with two 10 mm diameter balls and fixed with Retsch MM400 ball milling machine for 10 h. The average yield was reported by performing two same reactions.

Optimisation studies with benzene



Scheme S8: Optimisation Studies with Benzene

Table S2: Reaction time optimisation

Entry	Frequency (Hz)	Number of balls	Time (h)	Ball size (mm)	Isolated yield (%)
1	30	2	99 min	10	19
2	30	2	99 min x 2	10	26
3	30	2	99 min x 3	10	32
4	30	2	99 min x 4	10	43
5	30	2	99 min x 5	10	50
6	**30**	**2**	**99 min** x **6**	**10**	**61**

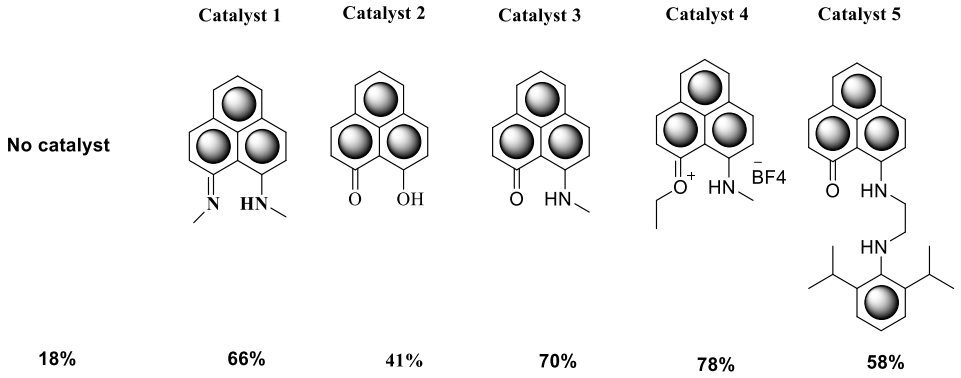
Reaction Conditions: 4-iodoanisole (0.3 mmol, 33.5 μL), benzene (3.0 mmol, 356.0 μL), PLY I (10 mol %, 9.7 mg), OED salt (20 mol %, 32.4 mg, 0.03 mmol), KO^{t}Bu (40 mol %,14.0 mg, 0.12 mmol), KO^{t}Bu (3.0 equiv., 101.0 mg, 0.9 mmol) were taken under inert conditions inside in a 5 ml jar with two 10 mm diameter balls and fixed with Retsch MM400 ball milling machine. Average yield was reported by performing two same reactions.

Table S3. Optimization of Base

Base (X)	Catalyst (mol%)	OED salt (mol%)	Yield%
No base	10.0	20.0	< 3
KO^{t}Bu (40%)	10.0	20.0	12
KO^{t}Bu (1 equiv.)	10.0	20.0	24
KO^{t}Bu (2 equiv.)	10.0	20.0	32
KO^{t}Bu (2.4 equiv.)	10.0	20.0	41
KO^{t}Bu (3 equiv.)	10.0	20.0	52
KO^{t}Bu (3.4 equiv.)	**10.0**	**20.0**	**61**
K_2CO_3 (3 equiv.)	10.0	20.0	10

Reaction conditions: The reactions were carried out using Retsch MM400 ball milling machine and 5 ml Jar and two 10 mm diameter balls. 4-iodoanisole (0.3 mmol, 33.5 μL), 2a (3.0 mmol, 356.0 μL), PLY I (10 mol %, 9.7 mg), OED salt (20 mol %, 32.4 mg), base (40 mol %), base (X equiv.). Yields were determined by the amount of the isolated product. In reaction, two balls are used. All the reactions were set up in the glove box at inert gas conditions.

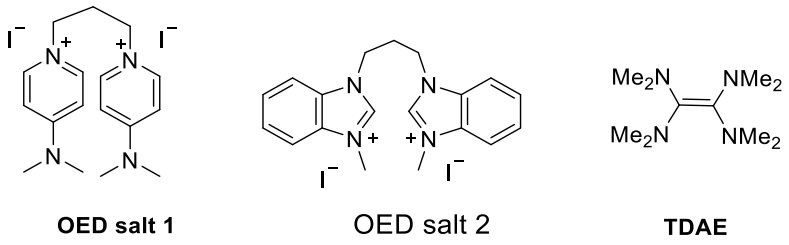
Table S4. Catalyst Optimization



Reaction conditions: All the reactions were carried out using Retsch MM400 ball milling machine and 5 ml Jar and two 10 mm diameter ball. iodobenzene (0.3 mmol, 33.5 μL), benzene (3.0 mmol, 356 μL), PLY I (10 mol %,9.7 mg), OED salt (0.06 mmol, 32.4 mg, base (40 mol %, 14.0 mg), base (0.9 mmol, 3.0 equiv., 101 mg). Biphenyl product yields were determined by the amount of the isolated product. The all reaction were set up in the glove box at inert gas conditions.

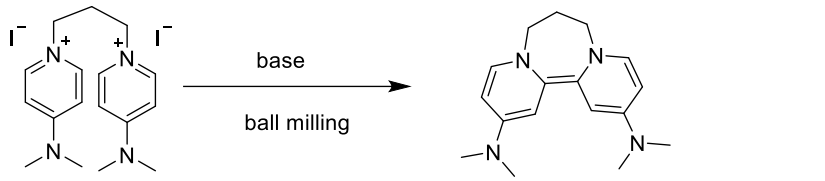
Table S5. Variation of the OED Salt

OED salt(Y) mol %	**Yield %**
TDAE (10 %)	<3
KO^{t}Bu (20 %)	<5
No OED salt	11
OED salt-1 (5 %)	32
OED salt-1 (10 %)	44
OED salt-1 (20 %)	**61**
OED salt-2 (20 %)	27
K (metal)	38



Reaction conditions: All the reactions were carried out using Retsch MM400 ball milling machine and 5 ml Jar and two 10 mm diameter balls. 4-iodoanisole (0.3 mmol, 33.5 μL), benzene (3.0 mmol, 356.0 μL), PLY I (10 mol %, 13.0 mg), OED salt (0.06 mmol, 32.4 mg), base (40 mol %, 14.0 mg), base (0.9 mmol, 3.0 equiv., 101.0 mg). Yields were determined by the amount of the isolated product. The all reaction were set up in the glove box at inert gas conditions.

Generation of OED from OED Salt and Base in Ball Milling Condition



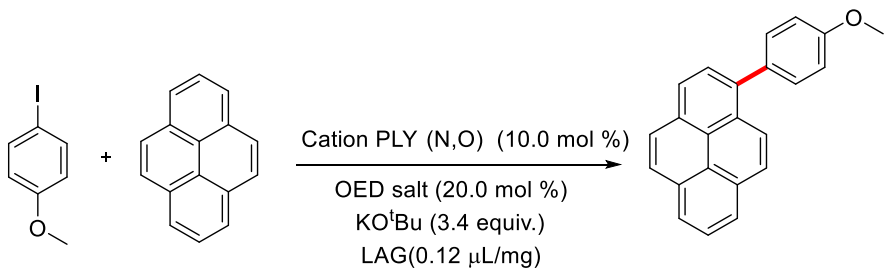
OED salt 1 **OED**

N^2,N^2,N^{12},N^{12}-tetramethyl-7,8-dihydro-6*H*-dipyrido[1,2-*a*:2',1'-*c*][1,4]diazepine-2,12-diamine

Scheme S9: Generation of OED

Frequency Optimisation of Mechano-SSSET with Polyarene

Solid-solid coupling:



46

Scheme S10: Solid-Solid Coupling

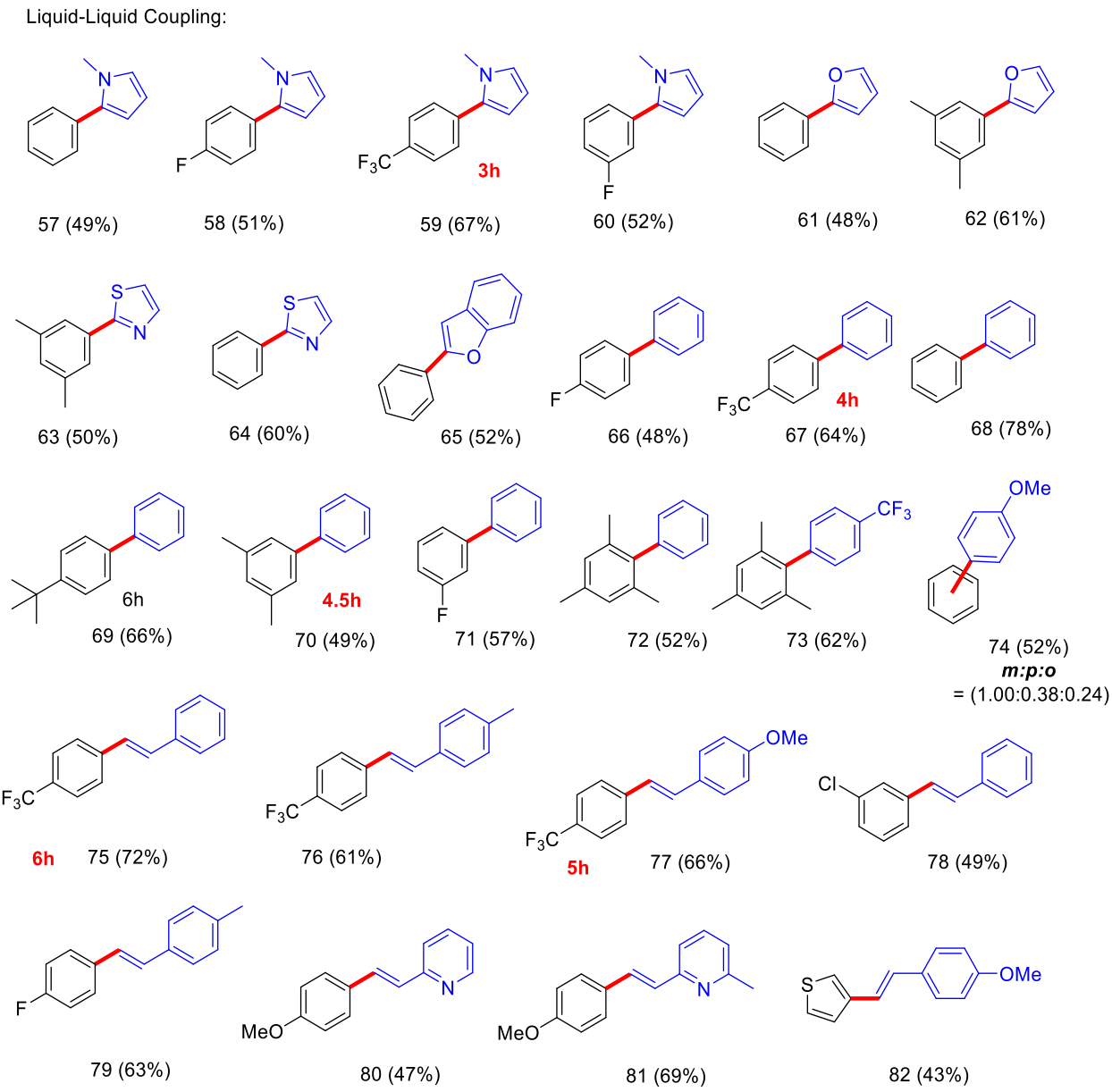
Table S6. Frequency Optimisation of Mechano-SSSET with Polyarene

Entry	Frequency (Hz)	Number of balls	Time (h)	Ball size (diameter in mm)	Yield (%)
1.	10	1	10 h	10 mm	22
2.	15	1	10 h	10 mm	31
3.	20	1	10 h	10 mm	40
4.	30	1	10 h	10 mm	51
5.	**30**	**2**	**10 h**	**10 mm**	**56**
6.	30	1	10 h	5 mm	37

Reaction conditions: All the reactions were carried out using Retsch MM400 ball milling machine and 5 ml Jar and two 10 mm diameter balls. 1-iodo-4-methoxybenzene (0.4 mmol, 93.6 mg), pyrene (2.0 mmol, 5.0 equiv., 404.0 mg), PLY I (10 mol %, 13.0 mg), OED salt (0.08 mmol, 43.2 mg), Base (40 mol %, 18.0 mg), Base (1.2 mmol, 3 equiv., 135.0 mg). Yields were

determined after the isolation of the product. All the reactions were set up in the glove box at inert gas conditions.

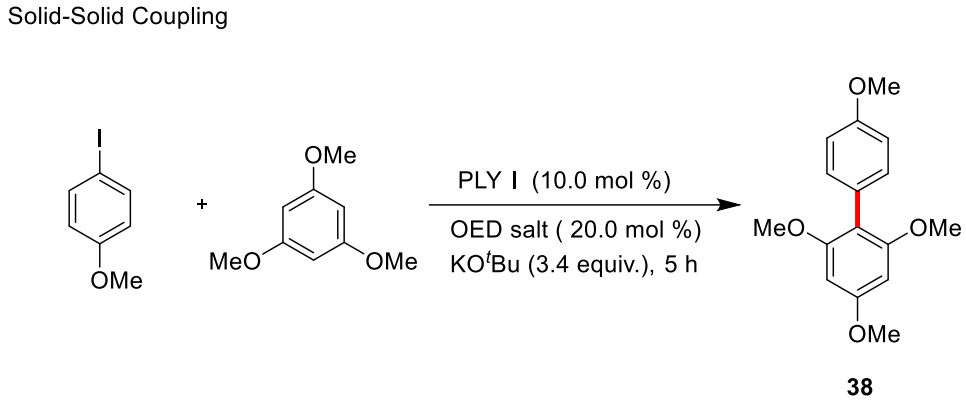
7. Liquid –Liquid Coupling Substrate Scope



Scheme S11: Substrate Scope of liquid-liquid coupling

8. Mechanistic Studies

Control Experiments: Importance of LAG on Ball Milling



Scheme S12: Control Experiments on Solid-Solid Coupling with LAG

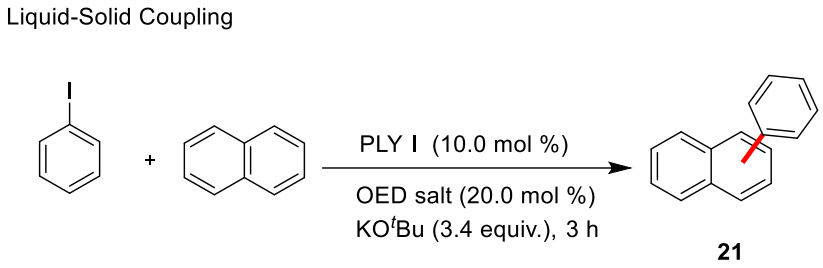
Table S7. Solid-Solid Coupling Reaction Conditions with Variation of LAG.

Entry	LAG (0.12µL/mg)	Reaction Conditions	Yield%
1.	No LAG	Neat Condition Without Ball Milling	No product
2.	With LAG	Neat Condition Without Ball Milling	10-15
3.	No LAG	Ball Milling	55
4.	**LAG**	**Ball Milling**	**80**

Reaction conditions: All the reactions were carried out using Retsch MM400 ball milling machine and 5 ml Jar and two 10 mm diameter balls. 1-iodo-4-methoxybenzene (0.3 mmol, 70.2 mg), 1,3,5-trimethoxybenzene (1.5 mmol, 5.0 equiv., 252.0 mg), PLY I (10 mol %, 9.7 mg), OED salt (20 mol %, 32.4 mg), base (40 mol %, 14.0 mg), base (3.0 equiv., 101.0 mg)

and LAG (DMSO) 0.12μL/mg . Yields were determined after isolation of the product. All the reactions were done in the glove box at inert gas (N_2, Ar gas conditions).

Liquid-Solid Coupling with Variation of LAG



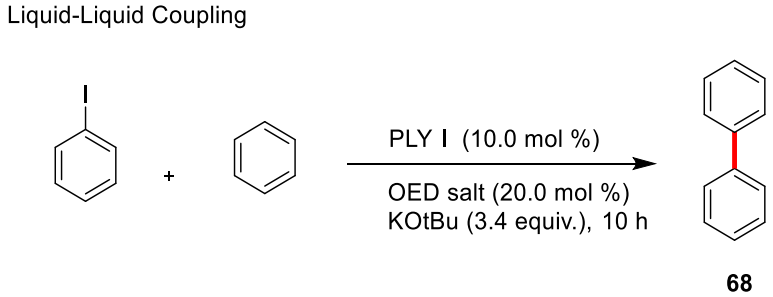
Scheme S13: Control experiments on Liquid-Solid coupling with LAG

Table S8. Liquid-Solid Coupling Reaction Conditions with Variation of LAG

Entry	LAG (0.12 μL/mg)	Reaction Conditions	Yield%
1.	No LAG	Neat Condition Without Ball Milling	No product
2.	With LAG	Neat Condition Without Ball Milling	15
3.	No LAG	Ball Milling	45
4.	**With LAG**	**Ball Milling**	**70**

Reaction condition: All the reactions were carried out using Retsch MM400 ball milling machine and 5 ml Jar and two 10 mm diameter balls. iodobenzene (0.4 mmol, 44 μL), naphthalene (2.8 mmol, 7.0 equiv., 358.0 mg), PLY I (10 mol %, 13.0 mg), OED salt (20 mol %, 43.2 mg), Base (40 mol %, 18.0 mg), Base (3 equiv., 135 mg). Yields were determined after the isolation of the product. All the reactions were set up in the glove box at inert gas (N_2, Ar gas conditions).

Liquid-Liquid Coupling Reaction Conditions with Variation of LAG



Scheme S14: Control Experiment on Liquid-Liquid Coupling with LAG

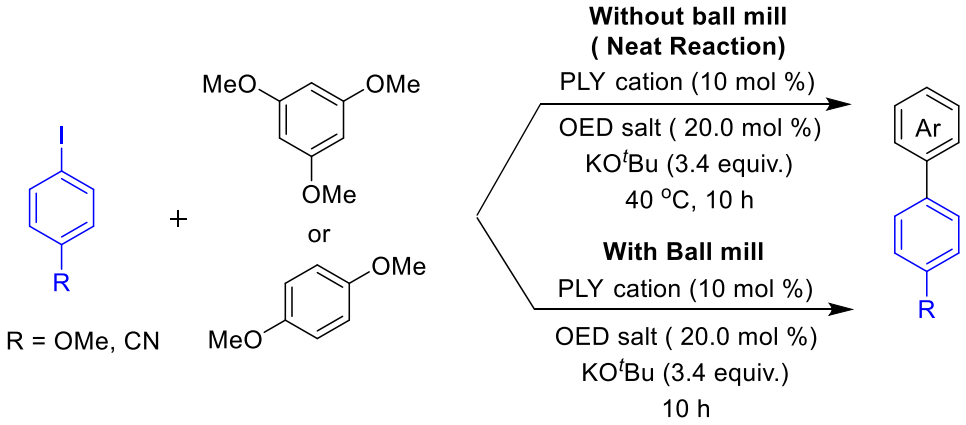
Table S9. Liquid-Liquid Coupling Reaction Conditions with Variation of LAG

Entry	LAG (0.12 μL/mg)	Reaction Conditions	Yield%
1.	No LAG	Neat Condition Without Ball Milling	60
2.	No LAG	Ball Milling	78

Reaction conditions: All the reactions were carried out using Retsch MM400 ball milling machine and 5 ml Jar and two 10 mm diameter balls. iodobenzene(0.3 mmol, 33.4 μL/mg), benzene (3.0 mmol, 10.0 equiv., 356.0 μL/mg), PLY I (10 mol %, 9.7 mg), OED salt (20 mol %, 32.4 mg), base (40 mol %, 13.5 mg), base (3.0 equiv., 101.0 mg). Yields were determined after the isolation of the product. All reactions were set up in the glove box at inert gas (N_2, Ar gas conditions).

The Above three set of experiments proves that the reaction in the solid-solid and solid-liquid phase needs LAG for efficient coupling reaction. However, for the liquid-liquid substrate system the reaction gave moderate yields in the absence of LAG also.

9. Impact of Ball Billing



Scheme S15: Competitive experiments performed without ball mill at 40 °C under neat conditions and the same reaction performed under ball milling conditions.

Table S10. Impact of ball milling: comparing the yield of neat reaction without ball-milling at 40 °C and that obtained after ball milling

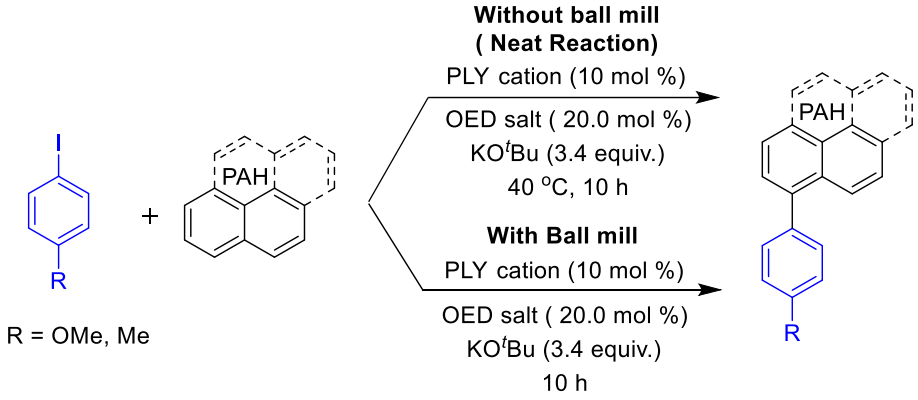
Aryl halide	***Arene***	***Neat reaction yield (%) (measured from a crude reaction mixture)***	***Ball milling process yield (after isolation)***
4-Iodoanisole	1,3,5-Trimethoxybenzene	**< 5**	**80** (product 38)
4-Iodoanisole	1,4-Dimethoxybenzene	**10**	**78** (product 44)
4-Iodobenzonitrile	1,3,5-Trimethoxybenzene	**< 5**	**60** (product 37)
4-Iodobenzonitrile	1,4-Dimethoxybenzene	**< 5**	**69** (product 42)

Ball Mill Reaction Conditions: All the ball milling reactions were carried out using Retsch MM400 ball milling machine and 5 ml Jar and two 10 mm diameter balls. Aryl halide (0.3 mmol), arenes (1.5 mmol, 5 equiv.), PLY I (10 mol %, 9.7 mg), OED salt (20 mol %, 32.4 mg), Base (3.4 equiv., 115.0 mg) Yields were determined after the isolation of the product. All the reactions were set up in the glove box at inert gas conditions. The effective temperature of the reaction mixture is 40 °C after ball milling.

Neat Reaction Conditions: PLY I (0.03 mmol, 9.7 mg) and OED salt (0.06 mmol, 32.4 mg) and KO^{t}Bu (0.12 mmol, 14.0 mg) were taken in a 25 mL oven dry pressure tube. This mixture

was stirred for 30 min. Substrates (0.3 mmol) and KO^{t}Bu (0.9 mmol, 101.0 mg) were added to the resulting solution of the catalyst inside a nitrogen filled glovebox. The final reaction mixture was stirred for an appropriate amount of time. After completion of the reaction, the products were extracted in 25 mL ethyl acetate and dried over anhydrous sodium sulphate. The solvent was removed under reduced pressure to obtain the crude products. The biarylated products were quantified with ^{1}H-NMR of the crude reaction mixture with an internal standard.

Next, we performed the comparison study without ball mill at 40 ^{o}C under neat conditions and the same reaction performed under ball milling conditions on polyarenes. The neat condition reactions failed to deliver the desired products.

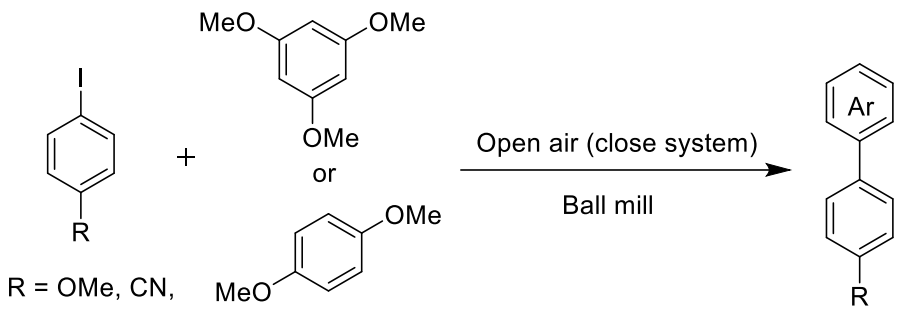


Scheme S16: Competitive Experiment on without Ball Mill and with Ball Mill using Polyarenes.

Table S11. Impact of ball milling: comparing the yield of neat reaction without ball-milling at 40 ^{o}C and that obtained after ball milling

Aryl halide	***Arene***	***Neat reaction yield (%) (Measured from a crude reaction mixture)***	***Ball milling process yield (After isolation)***
4-Iodoanisole	Pyrene	< 5%	56% (product **46**)
4-Iodoanisole	Anthracene	< 1%	51% (product **47**)
4-Iodoanisole	Phenanthrene	< 1%	47% (product **48**)
4-Iodotoluene	Pyrene	< 5%	68% (product **45**)

10. Open Air Reaction in Solid-Solid Coupling



***Scheme S17*:** Open air reactions in solid-state

Table S12: Open Air Reactions in Solid-State

Aryl Halide	Arene	Ball milling
4-Iodoanisole	1,3,5-Trimethoxybenzene	Product **38** 48%
4-Iodoanisole	1,4-Dimethoxybenzene	Product **44** 40%
4-Iodobenzonitrile	1,4-Dimethoxybenzene	Product **42** 47%
4-Iodobenzonitrile	1,3,5-Trimethoxybenzene	Product **37** 54%

All the ball milling reactions were carried out using Retsch MM400 ball milling machine and 5 ml Jar and two 10 mm diameter balls. All the reagents were added in the open air condition. Aryl halide (0.3 mmol), arenes (1.5 mmol, 5.0 equiv.), PLY I (10 mol %, 9.75 mg), OED salt (20 mol %, 32.4 mg), Base (40 mol %, 13.5 mg), Base (3.0 equiv., 101.0 mg)

11. Solid-Solid Coupling without LAG

A flame-dried ball mill jar equipped with two stainless steel balls were charged with OED salt (20 mol %, 32.4 mg, 0.06 mmol) and KO^{t}Bu (40 mol %, 14 mg, 0.12 mmol) inside a glove box and grinded the mixture in a ball milling machine for 30 min outside the glove box. Thereafter, the jar was again taken inside the glove box and PLY I (10 mol %, 9.7 mg), solid aryl halides (0.3 mmol, 1 equiv.), solid arene (1.5 mmol, 5.0 equiv.) and KO^{t}Bu (0.9 mmol, 3.0 equiv.) were added and subsequently, the reaction mixture was grinded for 10 h outside the glove box. The reaction was performed without any liquid-assisted grinding (DMSO). After completion

of the reaction, the mixture was extracted with 80-100 mL dichloromethane (DCM). The solvent was removed under reduced pressure and the crude product was purified by column chromatography using 100-200 mesh silica gel and hexane/EtOAc mixture as eluted with 80-100 mL dichloromethane (DCM). The solvent was removed under reduced pressure and the crude product was purified by column chromatography using 100-200 mesh silica gel and hexane/EtOAc mixture as eluent.

Table S13: Solid-solid Coupling without LAG

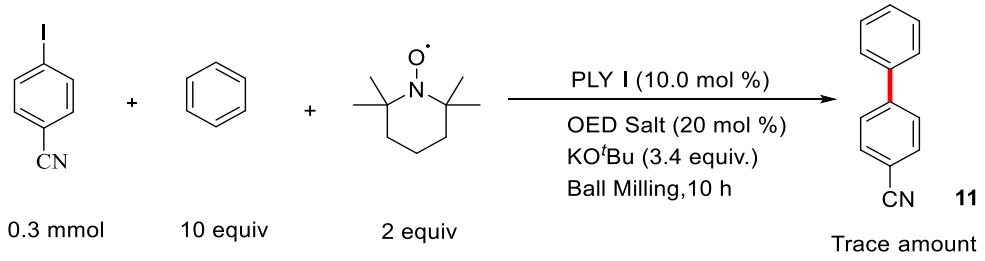
Entry	Aryl-halides (solid)	Arenes (solid)	LAG (0.12 μL/mg)	Products (isolated yield)
1.	4-Iodoanisole	1,3,5-Trimethoxybenzene	No LAG	Product **38** 50%
2.	4-Iodoanisole	1,4-Dimethoxybenzene	No LAG	Product **44** 51%
3.	4-Iodobenzonitrile	1,4-Dimethoxybenzene	No LAG	Product **42** 44%
4.	4-Iodobenzonitrile	1,3,5-Trimethoxybenzene	No LAG	Product **37** 52%

LAG = Liquid assisted grinding (DMSO)

12. Radical-Trapping Experiment in Solid State

Quenching Experiment with TEMPO: A flame-dried ball mill jar equipped with two stainless steel ball was charged with OED salt (20 mol %, 32.4 mg, 0.06 mmol) and KO^{t}Bu (40 mol %, 14 mg, 0.12 mmol) inside a glove box and grinded the mixture in ball milling machine for 30 min. Thereafter, the jar was again taken inside the glove box and PLY I (10 mol %, 9.7 mg), 4-iodobenzonitrile (68.7 mg, 0.3 mmol, 1 equiv.), TEMPO (93.6 mg, 0.6 mmol, 2.0 equiv.), benzene (268.0 μL, 3 mmol, 10.0 equiv.) and KO^{t}Bu (101 mg, 0.9 mmol, 3.0 equiv.) were added and subsequently the reaction mixture was grinded for 10 h outside the glove box. A very trace amount of arylated desired product was formed.

Conclusion: This study indicates that the reaction proceeds through the radical pathway.

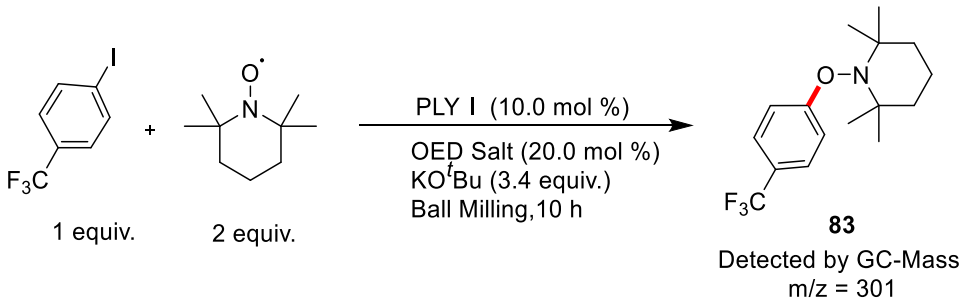


Scheme S18: Quenching Experiment with TEMPO.

Radical-Trapped experiment in Solid State: GC Study

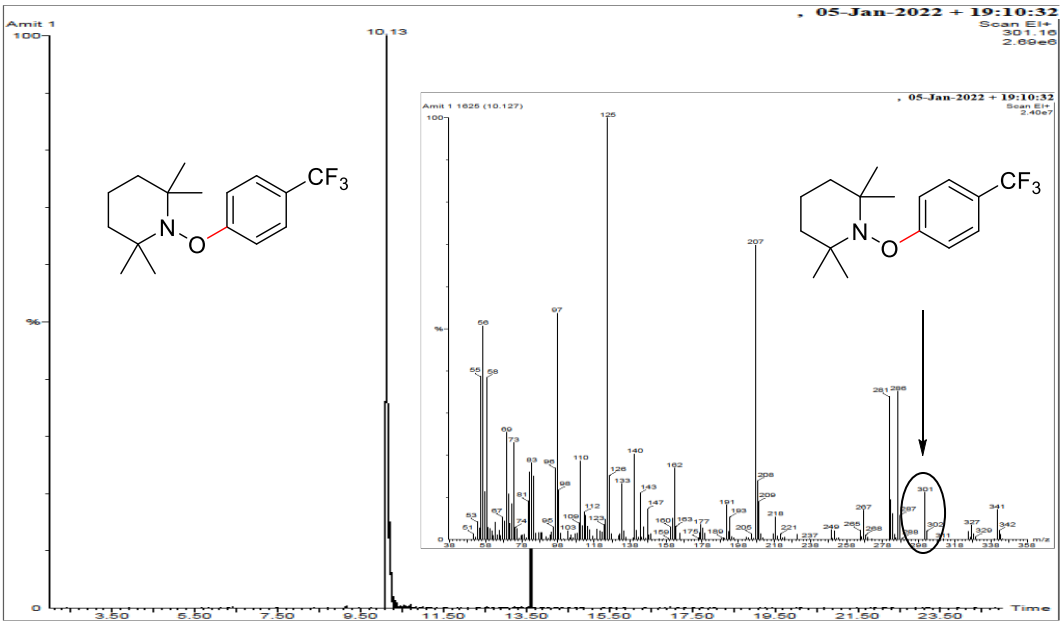
A flame-dried ball mill jar equipped with two stainless steel balls were charged with OED salt (20 mol %, 32.4 mg, 0.06 mmol) and KO^{t}Bu (40 mol %, 14 mg, 0.12 mmol) inside a glove box and grinded the mixture in a ball milling machine for 30 min outside the box. Thereafter, the jar was again taken inside the glove box and PLY I (10 mol %, 9.7 mg), 4-iodobenzonitrile (68.7 mg, 0.3 mmol, 1 equiv.), TEMPO (93.6 mg, 0.6 mmol, 2.0 equiv.) and KO^{t}Bu (101 mg, 0.9 mmol, 3.0 equiv.,) were added and subsequently, the reaction mixture was grinded for 10 h outside the glove box.

After completion of the reaction, we detected the tempo trapped product by GC-Mass with m/z value 301. This confirmed that the solid-state reaction was going through a radical pathway by the solid-state single electron transfer process.

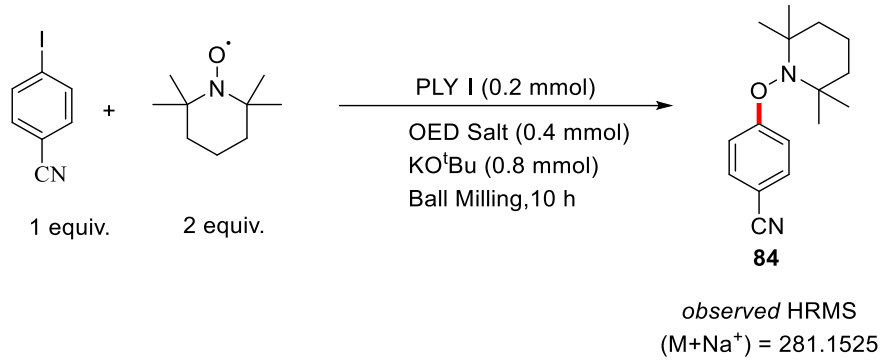


Scheme S19: Radical-Trapped Experiment in Solid State.

Fig. 7. GC-MS Chart.



TEMPO Trapping Experiment in Solid State

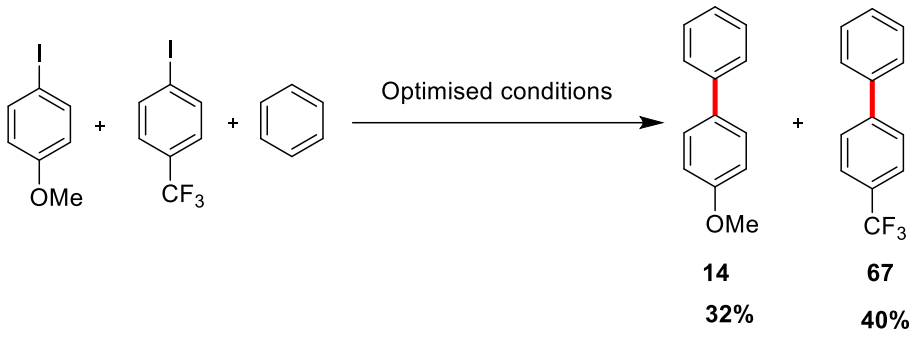


***Scheme S20* :** TEMPO Trapping Experiment in Solid State

The TEMPO aryl adduct mass was detected in HRMS. Calculated mass for [$C_{16}H_{22}N_2O$ + Na^+] is 281.1624 and the observed mass 281.1525.

13. Competition study

Reaction with Electron-Rich and Electron-Deficient Iodoarenes



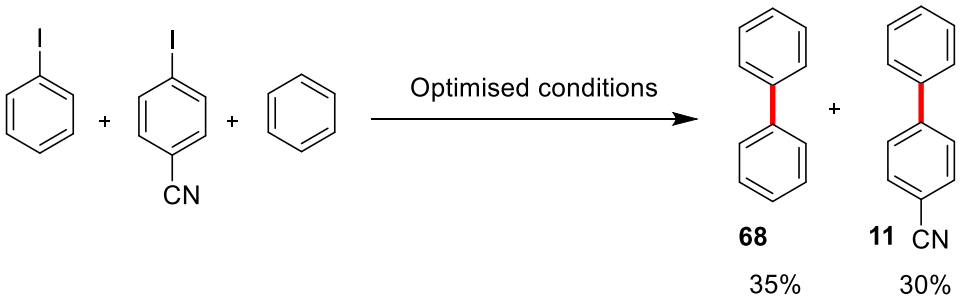
Scheme S21 : Reaction with Electron-rich and Electron-Deficient Iodoarenes

To a flame-dried milling jar equipped with two balls added OED salt (20 mol %, 32.4 mg, 0.06 mmol) and KO^{t}Bu (40 mol %, 14 mg, 0.12 mmol) inside the glove box and the resultant mixture was grinded outside in a ball milling machine for 30 min outside the box. Thereafter, the jar was again taken inside the glove box and PLY I (10 mol %, 9.7 mg), 4-iodoanisole (70.2 mg, 0.3 mmol, 1.0 equiv.), 4-iodo-1-trifluoromethylbenzene (44 µL, 0.3 mmol, 1.0 equiv.), benzene (268 µL, 3.0 mmol, 10 equiv.) and KO^{t}Bu (101 mg, 0.9 mmol, 3.0 equiv.,) were added and subsequently, the reaction mixture was grinded for 10 h outside the glove box.

After completion of the reaction, we have isolated products **14** and **67** with 32% and 40% yields respectively by column chromatography.

Conclusion: With both electron-donating and withdrawing substrates, we observed an almost similar amount of isolated product. It was concluded that the electronic factor does not affect the radical process. It gives an idea of radical-mediated process.

Reaction with Electron Neutral and Electron-Deficient Iodoarenes



Scheme S22: Reaction with Electron Neutral and Electron-Deficient Iodoarenes

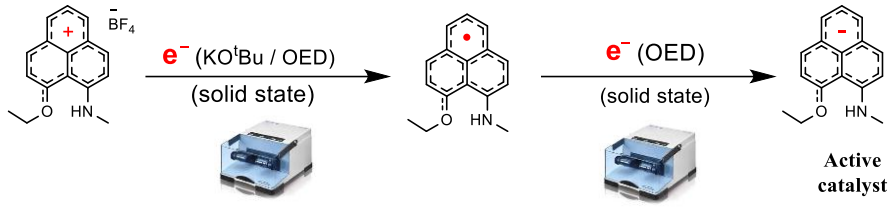
To a flame-dried milling jar equipped with two balls added OED salt (20 mol %, 32.4 mg, 0.06 mmol) and KO^{t}Bu (40 mol %, 14 mg, 0.12 mmol) inside the glove box and the resultant mixture

was grinded outside in a ball milling machine for 30 min outside the box. Thereafter, the jar was again taken inside the glove box and PLY I (10 mol %, 9.7 mg), iodobenzene (34 μL, 0.3 mmol, 1 equiv.), 4-iodo-1-benzonitrile (68.7 mg, 0.3 mmol, 1 equiv.), benzene (268 μL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (101 mg, 0.9 mmol, 3.0 equiv.,) were added and subsequently, the reaction mixture was grinded for 10 h outside the glove box.

After completion of the reaction, we have isolated products **68** and **11** with 35% and 30% yields.

Reaction conditions: All the reactions were carried out using a Retsch MM400 ball milling machine and 5 ml Jar and two 10 mm ball.

14. Synthesis and Characterization of PLY I, PLY II and PLY III



Scheme S23: Synthesis of PLY II and PLY III

PLY **I**

orange solid

PLY **II**

green solid

PLY **III**

brown solid

Fig 8: Synthesis of PLY II and PLY III

PLY I

Phenalenyl-based ligand (**PLY I**) were synthesized following our previous literature report[1].

PLY II

We generated PLY **II** using cationic PLY **I** with various electron donors by changing the stoichiometry of the reducing agents (TDAE or KO^{t}Bu or in situ generated organic electron donor (OED) or metallic K in the solid state under mechanical force within a ball mill for 15 min. The addition of one equivalent reducing agent generated a green compound identified as a mono-reduced radical PLY radical species (PLY **II**).

The radical PLY **II** was characterized by EPR and solid-state UV-Visible spectra.

We studied the catalytic activity of PLY **II** in the solid state. However, using a ball mill, we failed to activate aryl halide with this mono-reduced green solid compound (PLY **II**) generated under mechanical force. Mono-reduced PLY **II** could not activate aryl halides in ball milling, indicating that the reduction potential of PLY **II** is not sufficiently high to activate aryl halides.

PLY III

We treated **PLY II** with another equivalent of organic electron donor (OED) using mechanical force for 30 min, and we noticed the formation of brown solid PLY **III** which was characterized by EPR and UV experiments.

The brown solid showed EPR inactivity at rt.

Due to high reactivity, the double-reduced PLY **III** decomposes upon exposure to air. However, it can be stored in the glovebox for one week at -20 ^{o}C without losing its reactivity. When we exposed PLY **III** to the air, it changed its colour immediately from brown to black and decomposed.

Next, we used this brown solid (PLY **III**) for aryl halide bond activation. To our delight, the PLY **III** is an excellent reductant to undergo a solid-state single electron transfer, generating aryl radical by activating aryl halides.

Cyclic Voltammetry Experiment of (N,O) PLY Cation

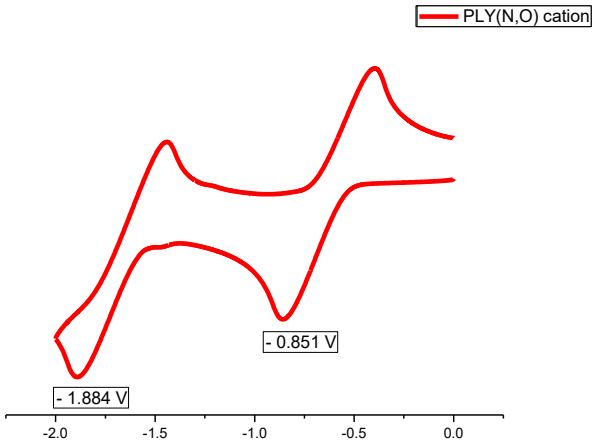


Fig. 9. Cyclic voltammetry of PLY I vs Ag/AgCl in DMF. This cyclic voltammetry was also reported in previous literature.

The cyclic voltammetry showed that the PLY I has a two consecutive double reduction potential ($E^1_{1/2}$ = -0.85 V and $E^2_{1/2}$ = -01.88 V).

Solid-State UV-Visible Spectroscopy

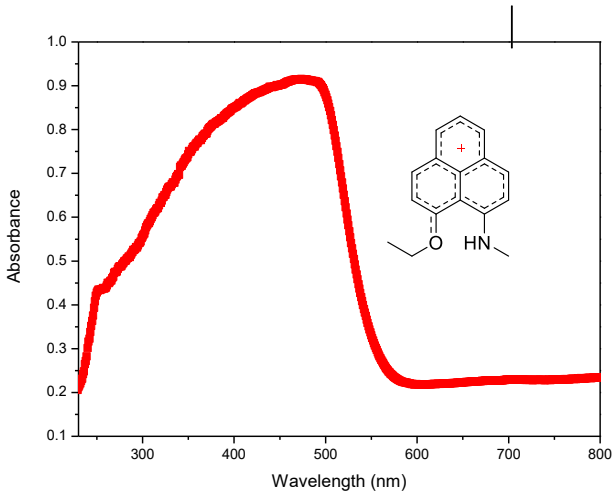


Fig.10. UV spectra of solid PLY I in solid-state at room temperature. The absorbance maximum of 474 nm of PLY I in Solid-State. 15 mg sample was prepared for the solid-state UV experiment. The UV-visible spectra showed absorbance maximum at room temperature at 474 nm.

UV-Visible spectra of Mono-Reduced PLY II in Solid-State

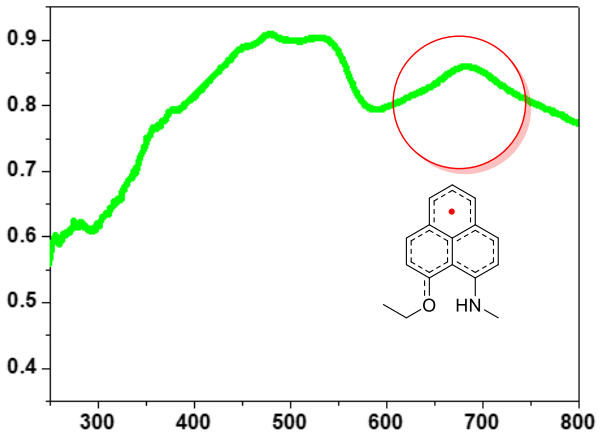


Fig. 11. UV spectrum of mono-reduced solid (N, O) PLY cation in solid-state at room temperature. The green solid compound showed maximum absorbance at 476 nm. The 682 nm region absorbance is identified as a radical anion peak **(Fig S13)**.

UV-Visible Spectra of Double-Reduced PLY III in Solid-State

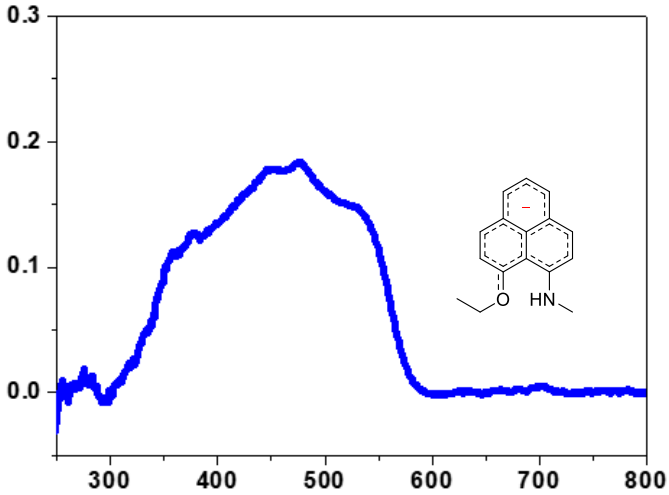


Fig. 12. UV spectrum of double-reduced solid PLY III in solid state at room temperature. The brown solid showed a maximum absorbance peak at 474 nm and a small hump at 682 nm.

Solid-State EPR Spectroscopy

The Green Solid (mono-reduced PLY II)

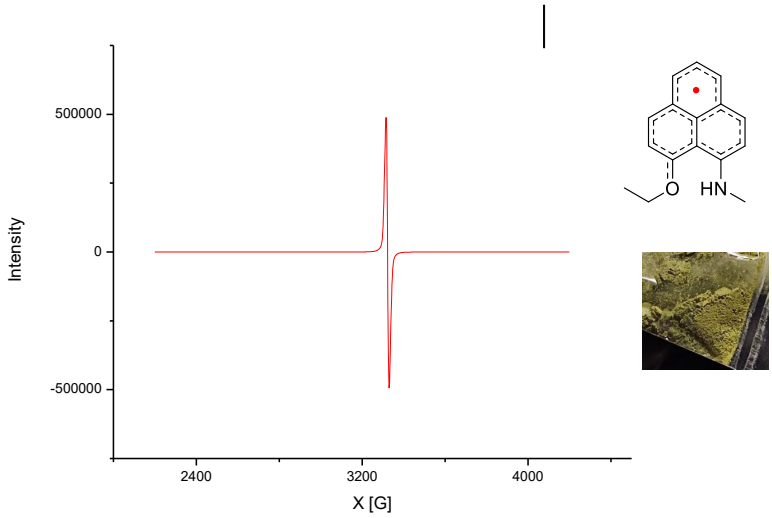


Fig. 13. EPR measurement of green mono-reduced PLY II in solid-state at room temperature. The green solid compound (**Fig S15**) showed EPR activity with a g value of 2.004.

The Brown-Solid (double reduced (N,O) PLY III)

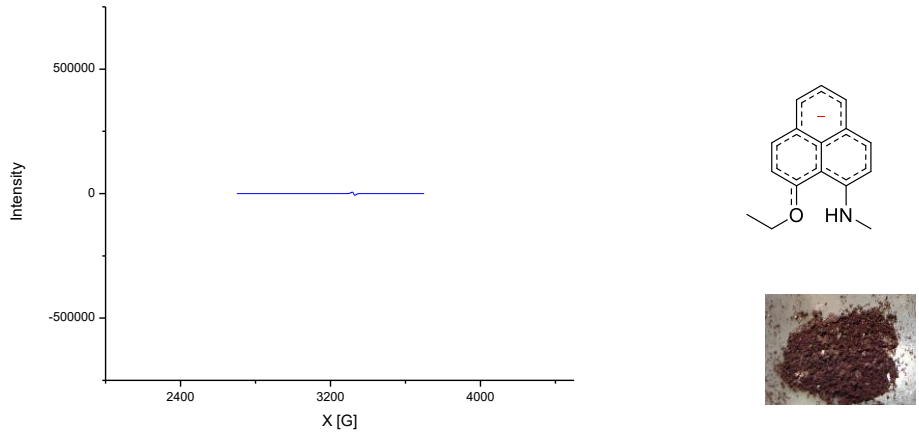
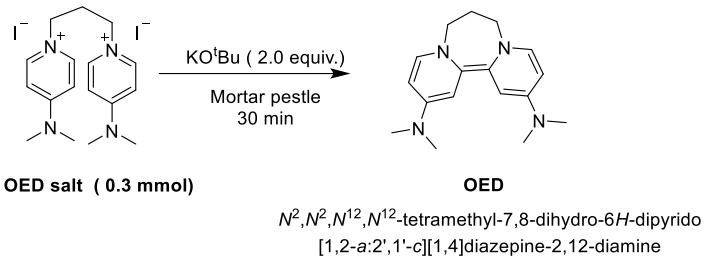


Fig. 14. EPR measurement of brown double-reduced PLY III in solid-state. The brown solid compound (**Fig S17**) showed EPR silent. The brown solid compound was very unstable and also air sensitive.

15. Pictorial Representation of the Solid State Catalyst Synthesis

Generation of OED from the OED salt



***Scheme S24*:** Generation of OED from OED Salt and KO^{t}Bu

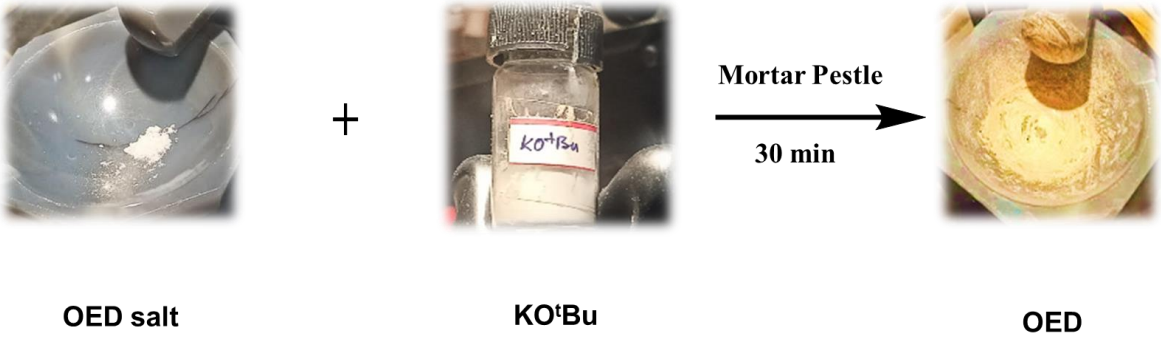
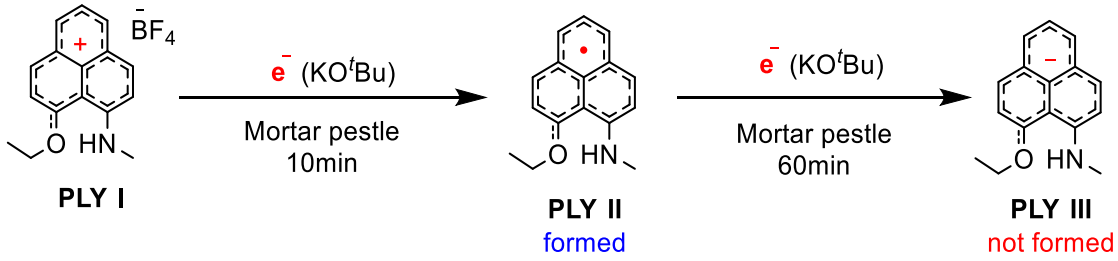


Fig. 15: Pictorial Representation of the Reaction Setup for OED Generation

We synthesized OED from OED salt (162 mg, 0.3 mmol) and KO^{t}Bu (68 mg, 0.6 mmol, 2.0 equiv.) in the mortar pestle under mechanical force for 30 min.

Generation of PLY II from PLY I with KO^{t}Bu as the Reductant



***Scheme S25*:** Generation of PLY II from PLY I using KO^{t}Bu as Reductant

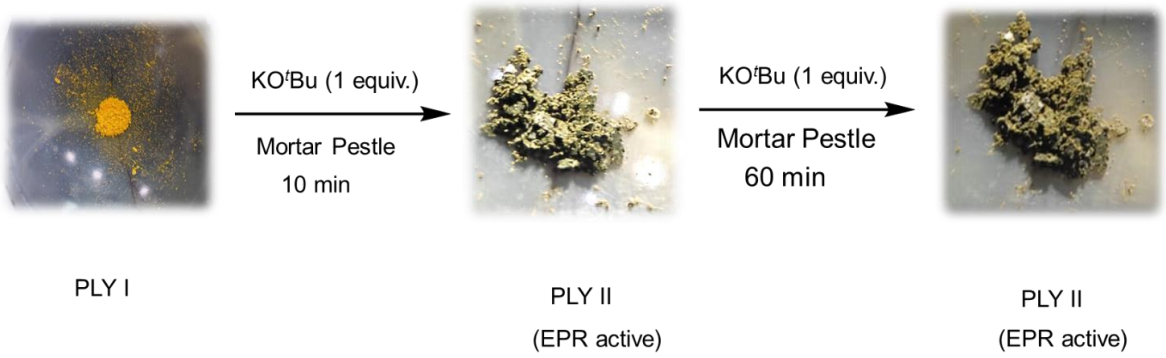
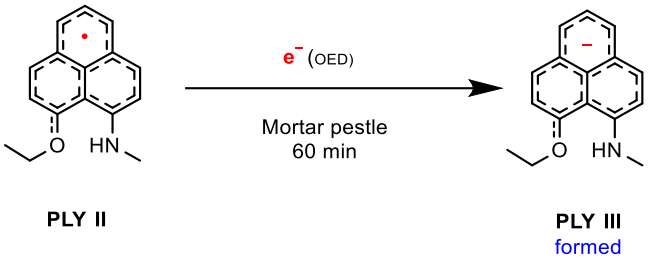


Fig. 16: Pictorial Representation of the Reaction Setup with KO^{t}Bu as Reductant

PLY II (green compound) is prepared from cationic PLY I using KO^{t}Bu in the solid state upon treatment of mechanical force within a mortar pestle for 10 min. In ball-mill, we have similar observation after 15 minutes of grinding.

Subsequently, we added another 1.0 equivalent of KO^{t}Bu and continued the mortar pestle grinding for additional 60 minutes. However, the further reduction of PLY II to PLY III was not observed in this process, which indicates that the KO^{t}Bu is not sufficient enough to generate the active redox catalyst PLY III.

Generation of PLY III from PLY II and OED



***Scheme S26*:** Generation of PLY III from PLY II using OED

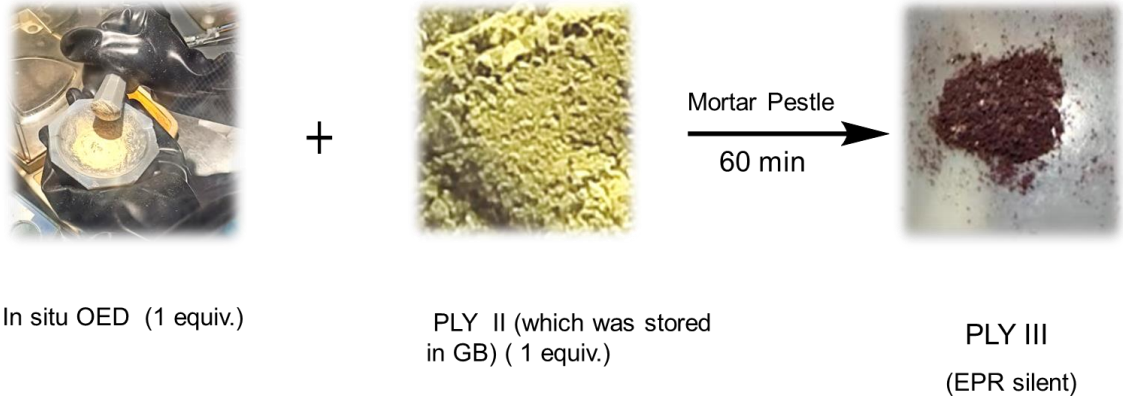
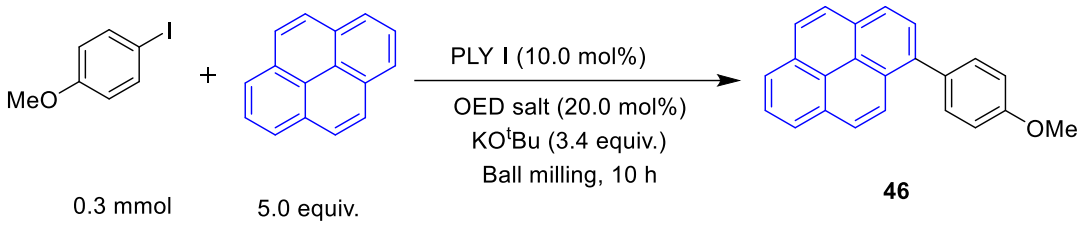


Fig. 17: Pictorial Representation of the Reaction Setup using OED as Reductant

PLY III (brown compound) is prepared by the reaction of PLY II (green compound) with OED in the solid state upon treatment of mechanical force by a mortar pestle for 60 minutes. In ball-mill, we have similar observation after 30 minutes of grinding.

16. Measurements of the Ball Milling Jar Temperature at Six Reactions (6 x 99 min)



***Scheme S27*:** Measurements of the Ball Milling Jar Temperature at Six Reactions (6 x 99 min)

The temperature was measured with an Infrared Thermometer Z000K, Infra Temp, Related Current 100 mA, and Power Supply DC 3V (2 AAA batteries). The temperature was measured immediately after opening the milling jar. We measured the temperature of a total of six reactions with a time interval of 99 minutes for which we set up total of six reactions using 4-Iodoanisole (0.3 mmol, 1.0 equiv.) and pyrene (1.5 mmol, 5.0 equiv.) at the optimized conditions. The temperature of the empty jar = 32 ^{o}C (89.7 ^{o}F). The temperature of the jar after the generation of OED (after 30 minutes of grinding with OED salt and KO^{t}Bu = 33.3 ^{o}C (92 ^{o}F).

The temperature of the reaction after 1x 99 minutes = 37.05 ^{o}C (98.7 ^{o}F).

The temperature of the reaction after 2x99 minutes = 37.27 ^{o}C (99.1 ^{o}F).

The temperature of the reaction after 3x 99 minutes = 38.38 ^{o}C (101.1 ^{o}F).

The temperature of the reaction after 4x99 minutes = 39.55 ^{o}C (103.2 ^{o}F).

The temperature of the reaction after 5x99 minutes = 39.66 ^{o}C (103.4 ^{o}F).

The temperature of the reaction after 6x99 minutes = 40.27 ^{o}C (104.5 ^{o}F).

The maximum temperature recorded was 40.27 ^{o}C after 6 x 99 minutes, which suggests that the temperature did not significantly increase under the optimized mechanical conditions.

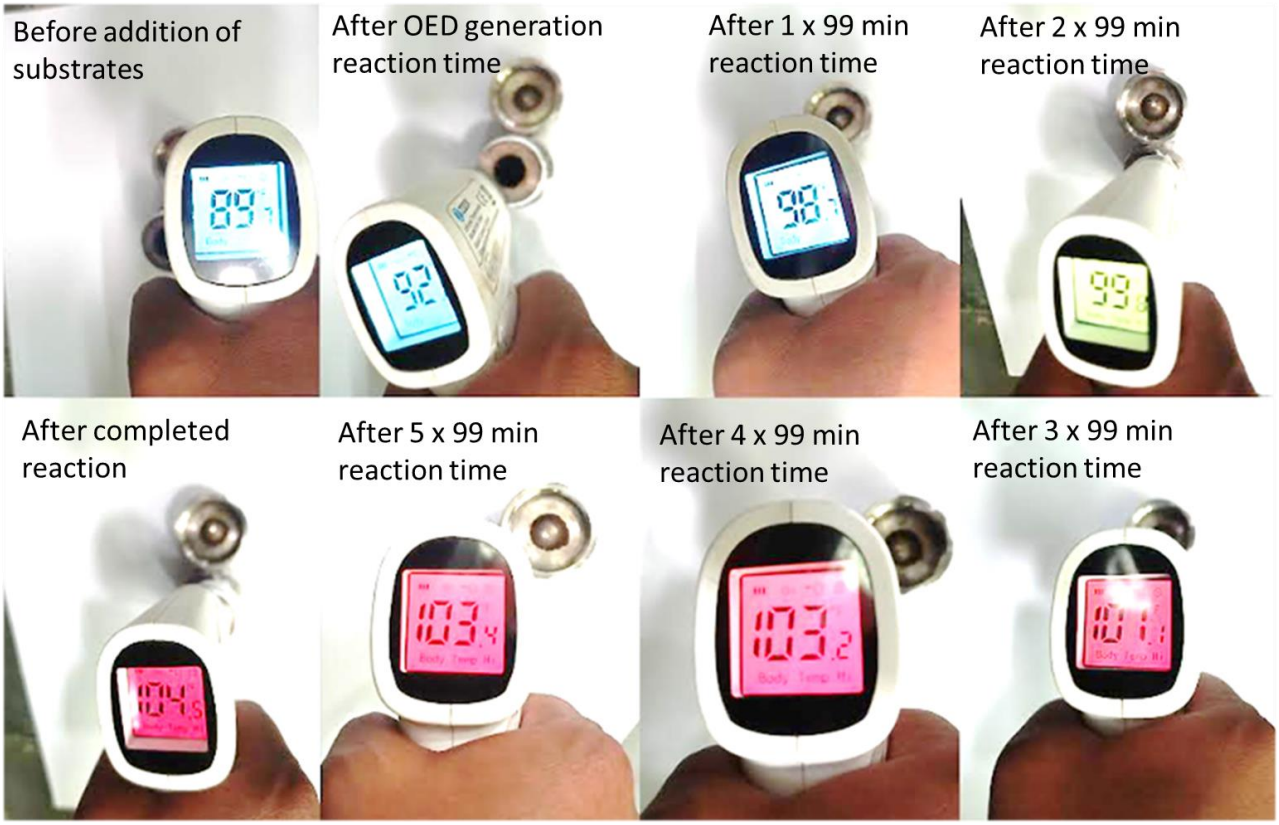
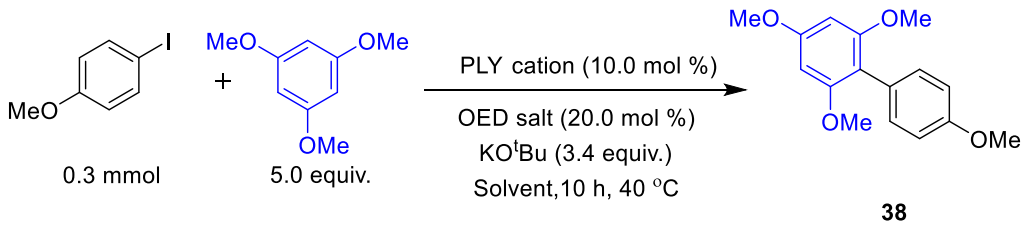


Fig. 18: Temperature Measurement on Various Point of the Reaction in the Ball Milling

We observed the maximum temperature <u>104.5 º F (40.27 º C)</u> at the end of the ball milling reaction.

17. Solid-Solid Coupling in Solvent-Phase Reaction at 40 ºC

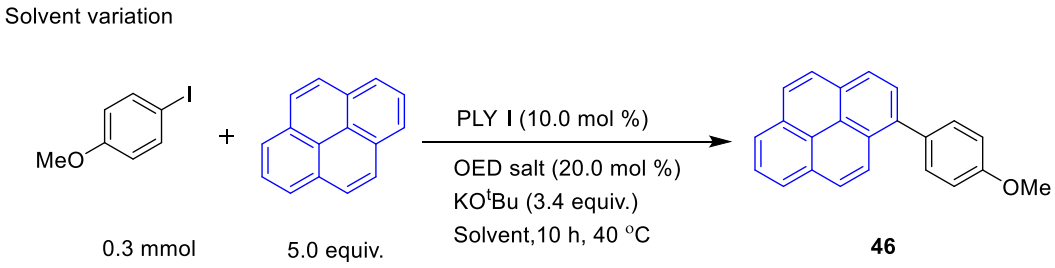
Comparison of reaction yield performed using various organic solvents at 40 °C and under ball milling conditions between 4-iodoanisole and 1,3,5-trimethoxybenzene



***Scheme S28*:** Coupling of 4-iodoanisole and 1,3,5-trimethoxybenzene using various solvents at 40 °C and its comparison with that under ball-milling conditions.

Table S14: Yields on Various Solvent System

Entry	Solvent	NMR Yield %
1	**THF**	**<1**
2	**DCM**	**<1**
3	**DMF**	**<5**
4	**Reaction under ball milling conditions**	**80%** (isolated yield)



***Scheme S29*:** Solid-Solid Coupling in Solvent-phase Reaction

All the reaction was performed at 40 °C.

Reaction Conditions: A flame-dried pressure tube was charged with OED salt (20 mol %, 32.4 mg, 0.06 mmol) and KO^{t}Bu (115.0 mg, 3.4 equiv.) in various solvent inside a glove box for 30 min. Thereafter, the PLY I (10 mol %, 9.7 mg, 0.03 mmol), 4-Iodoanisole (0.3 mmol, 1 equiv.), and pyrene (1.5 mmol, 5.0 equiv.) was added in various solvent and subsequently, the reaction mixture was stirred at 40 °C for 10 h outside the glove box. After completion, the reaction mixture was passed through celite with ethyl acetate (EtOAc) and measured ^{1}H-NMR with 1,4-dimethoxybenzene as an internal standard.

Table S15: Yields on Various Solvent System

Entry	Solvent	NMR Yield %
1	THF	5
2	DCM	0
3	DMF	9
4	Hexane	0
5	CH_3CN	0
6	Toluene	8
7	Reaction without solvent in ball mill	56 (isolated yield)

Fig. 19: ^1H NMR in $CDCl_3$ of crude reaction mixture (reaction was performed in dry THF solvent)

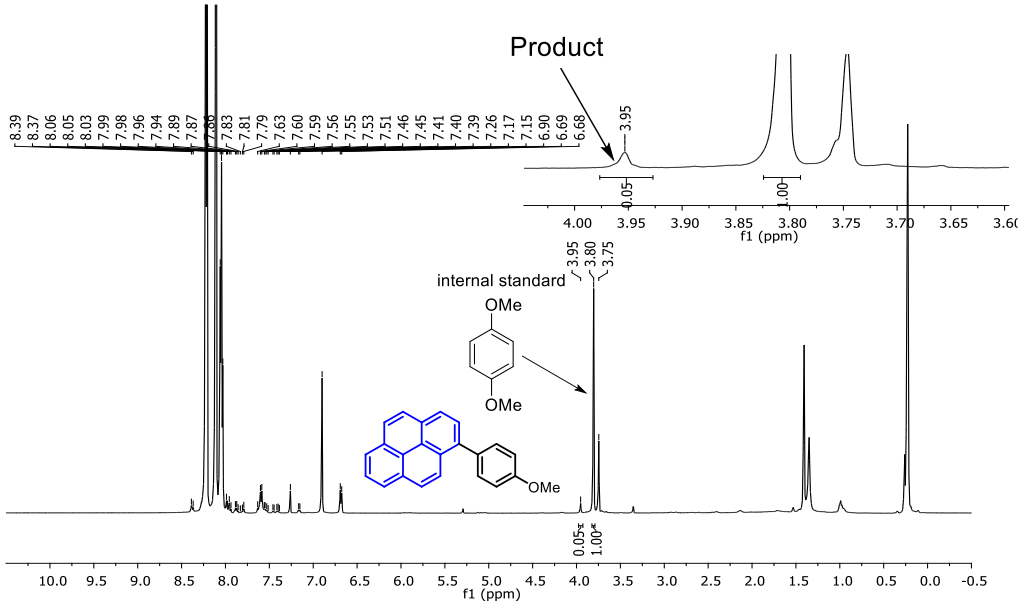


Fig. 20: ^{1}H NMR in $CDCl_3$ of crude reaction mixture (reaction was in dry DCM solvent)

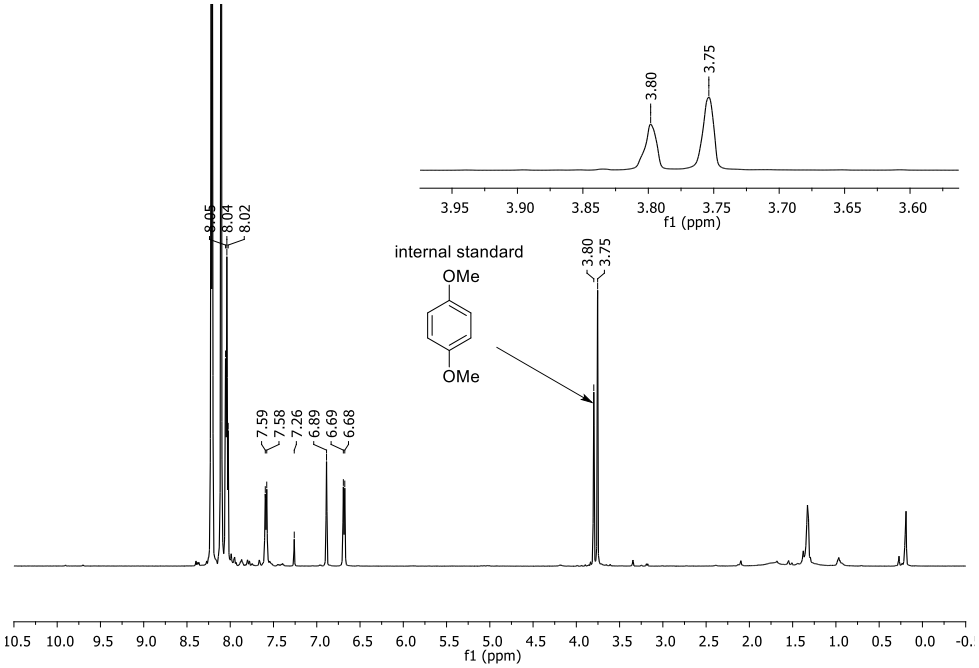


Fig. 21: ^{1}H NMR in $CDCl_3$ of crude reaction mixture (reaction has been performed in dry DMF solvent)

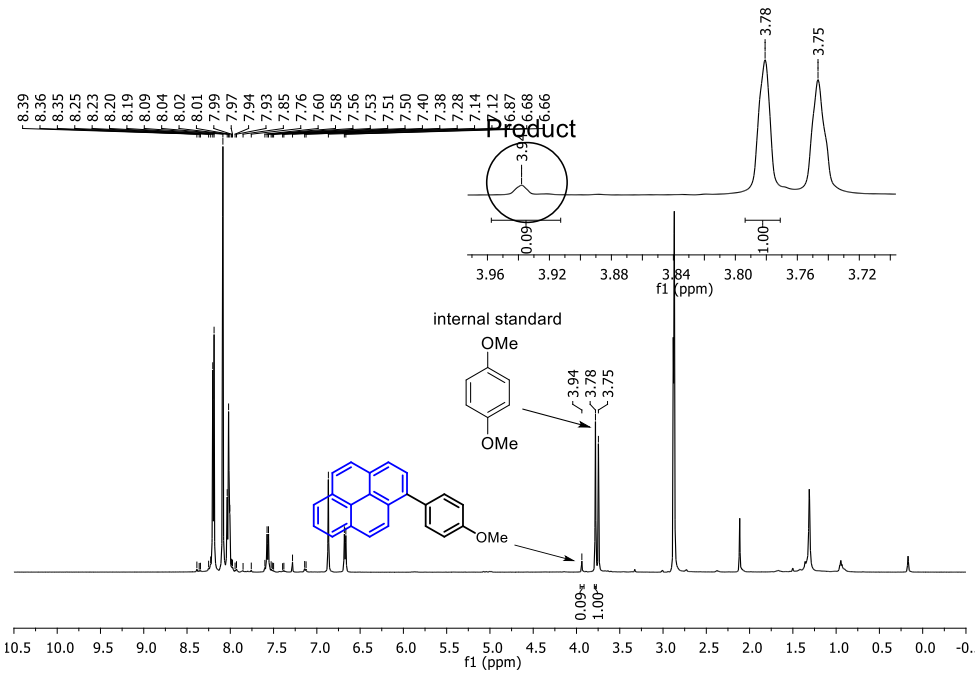


Fig. 22: ^{1}H NMR in $CDCl_3$ of crude reaction mixture (reaction was performed in dry hexane solvent)

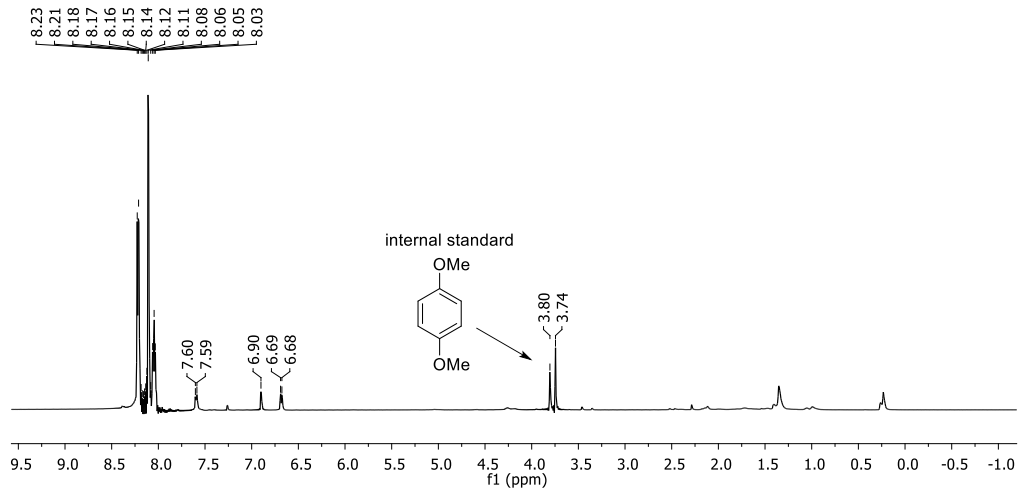


Fig. 23: ^{1}H NMR in $CDCl_3$ of crude reaction mixture (reaction was in dry CH_3CN solvent)

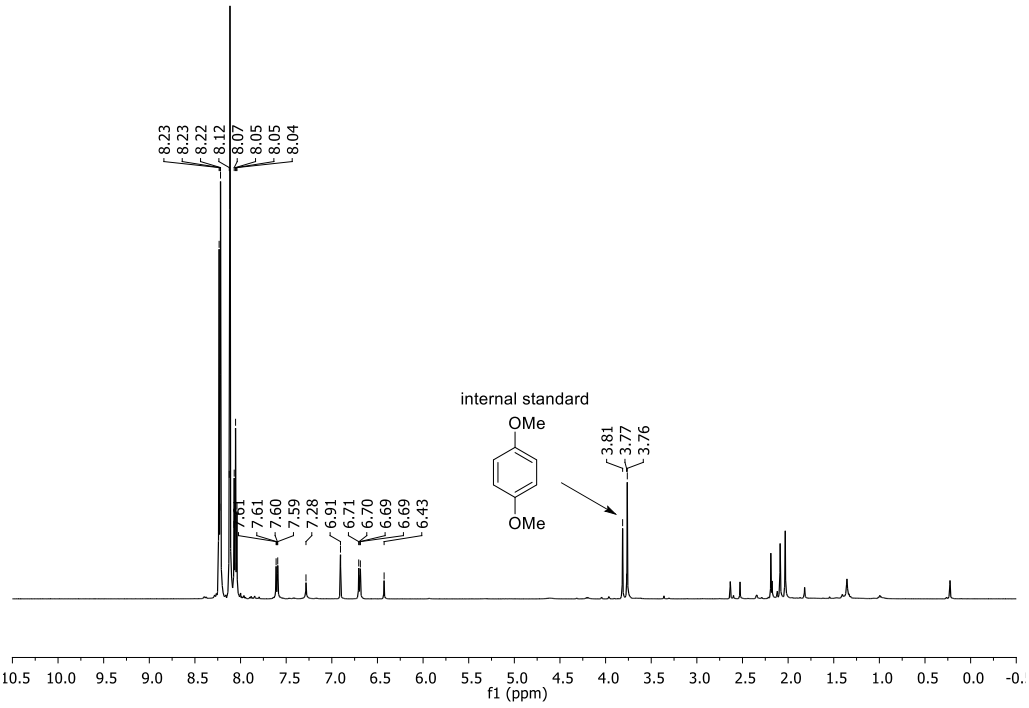
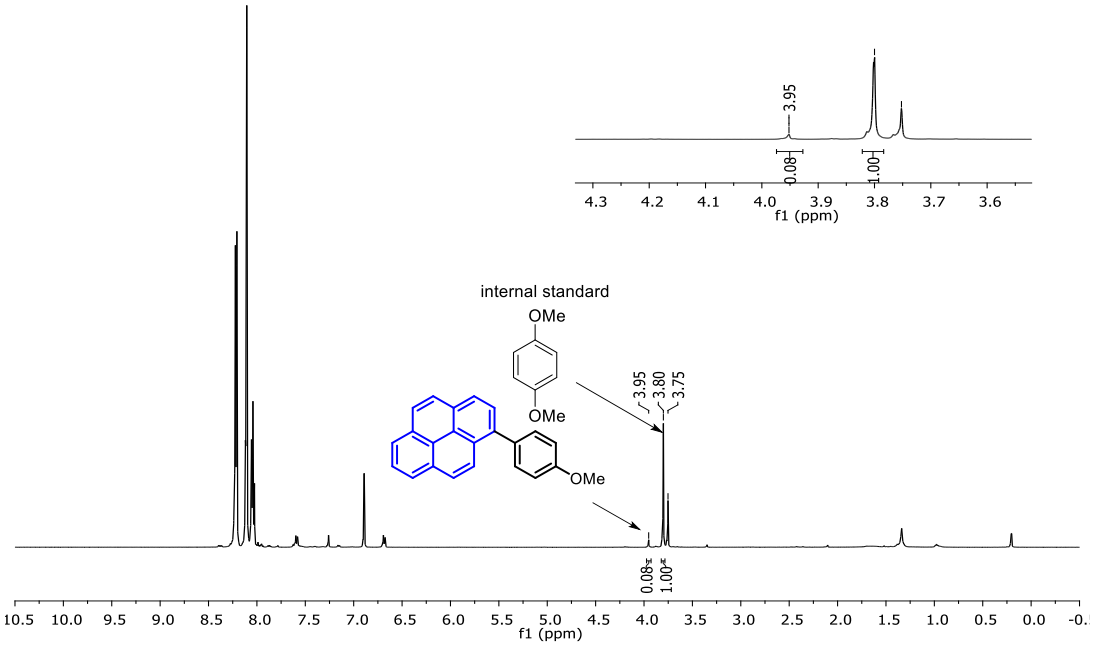
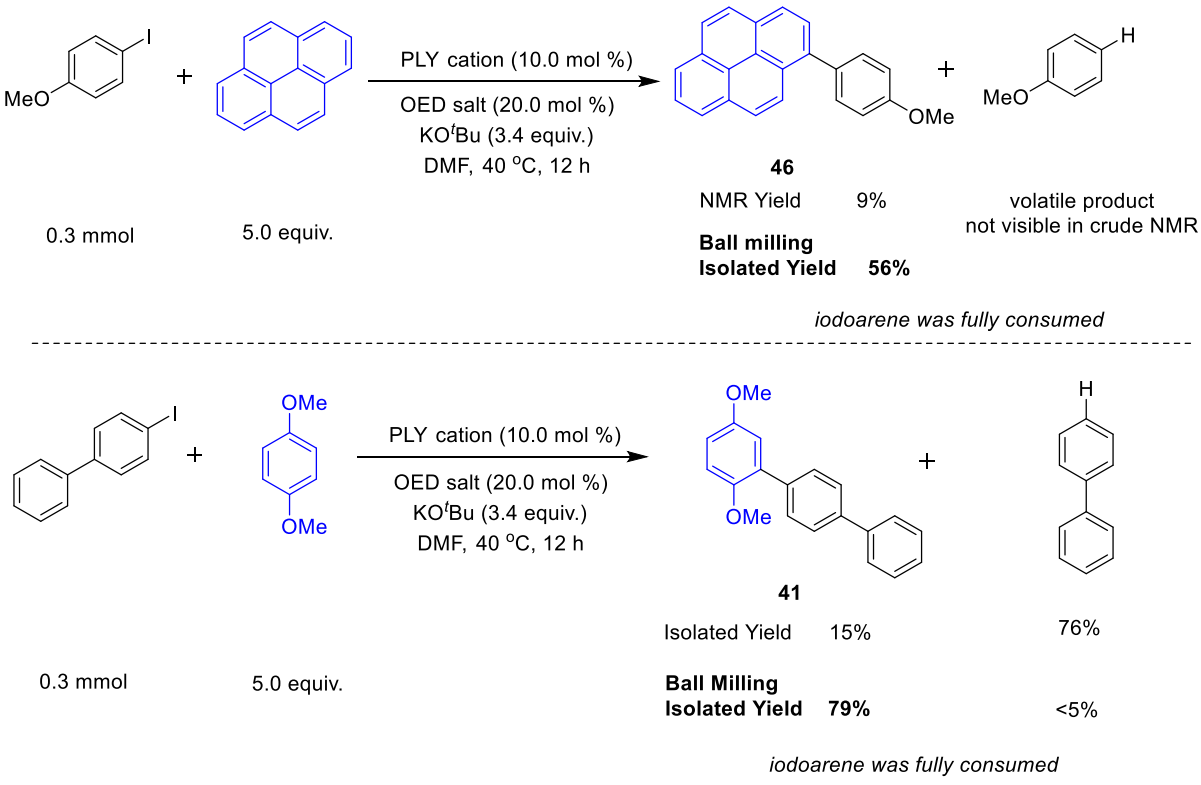


Fig. 24: ^{1}H NMR in $CDCl_3$ of crude reaction mixture (reaction was performed in dry toluene solvent)



In addition, we have conducted a set of reaction to understand the difference of the solvent phase reaction and ball milling reaction.

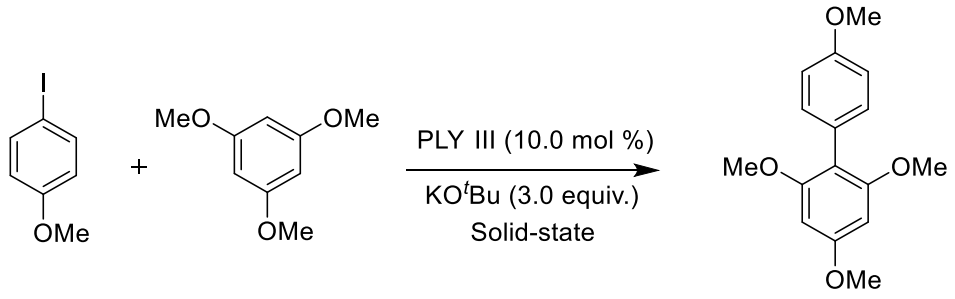


***Scheme S30*:** Control experiments for the Measurement of Dehalohydrogenation Products in Solution Phase.

Reaction Condition: PLY I (0.03 mmol, 9.7 mg) and OED salt (0.06 mmol, 32.4 mg) and KO^{t}Bu (0.12 mmol, 14.0 mg) were taken in a 25 mL oven dry pressure tube. This mixture was stirred for 30 min. 4-Iodobiphenyl (84 mg, 0.3 mmol), 1,4-dimethoxybenzene (207 mg, 1.5 equiv.) and KO^{t}Bu (0.9 mmol, 101.0 mg) were added to the DMF (1 ml) solution of the catalyst inside a nitrogen filled glovebox. The final reaction mixture was stirred for an appropriate amount of time. After completion of the reaction, the products were extracted in 25 mL ethyl acetate and dried over anhydrous sodium sulphate. The solvent was removed under reduced pressure to obtain the crude products. We isolated the expected product (34 mg, 40%) as well as the dehydrohalogenated product (24 mg, 52%) by column chromatography in silica gel. We observed major amount of dehalohydrogenated product.

These results indicate that the reaction in the solution phase is more prone to deliver the dehalohydrogenated by-product. However, in the absence of the solvent the amount of the dehalohydrogenated by-product was minimized.

18. Reactions with isolated PLY III (active) catalyst: comparison of yields in ball mill and without ball mill conditions



38

i. Without ball mill yield 5%
ii. With ball mill yield 80%

Scheme S31: Reactions with Isolated PLY III Active Catalyst

At first, we generated PLY III using KO^{t}Bu / OED under ball milling condition. Then we stored the active PLY III catalyst in glove box at -20 ^{o}C.

Next, we tested the reactivity of isolated PLY **III** catalyst with various conditions.

i. Without Ball Mill: PLY III (0.03 mmol) was taken in a 25 mL pressure tube. After that 4-iodoanisole (0.3 mmol), trimethoxybenzene (1.5 mmol) and KO^{t}Bu (0.9 mmol) were added to the reaction mixture of the catalyst inside a nitrogen filled glovebox. The reaction kept for 12 h without solvent. After completion of the reaction, the products were extracted in 25 mL ethyl acetate and dried over anhydrous sodium sulphate. The solvent was removed under reduced pressure to obtain the crude products. The biarylated products were isolated by column chromatography over silica gel using a hexane and ethyl acetate mixture solvent as the eluent. we observed very less product formation (5% yield).

ii. With Ball Mill: PLY III (0.03 mmol) was taken in a ball milling jar (5 ml). After that 4-iodoanisole (0.3 mmol), trimethoxybenzene (1.5 mmol) and KO^{t}Bu (0.9 mmol) were added to the reaction mixture of the catalyst inside a nitrogen filled glovebox. The reaction set up in ball milling for 5 h without solvent. After completion of the reaction, the products were extracted in 25 mL ethyl acetate and dried over anhydrous sodium sulphate. The solvent was removed under reduced pressure to obtain the crude products. The biarylated products were isolated by column chromatography over silica gel using a hexane and ethyl acetate mixture solvent as the eluent. We observed (80% yield) the desired product was formed.

19. Proposed Catalytic Cycle for Solid-State Catalysis

This is a plausible mechanism for direct arylation of heteroarenes /arenes /polyarenes. The solid-state reaction was going through single electron transfer (SET) catalysis in the presence of mechanical energy. Here the mechanical force has an excellent role in the electron transfer catalysis in solid-state. The intermediates of this catalyst were characterized by UV and EPR measurements. Additionally, we also trapped the TEMPO-trapped product by GC-MS and HRMS measurements. An extra base was needed due to the aromatization of desired C-C coupling products in the solid-state. This Mechano-SET also applied liquid-liquid, liquid-solid, and solid-solid (without LAG also) conditions and we observed moderate to good yield.

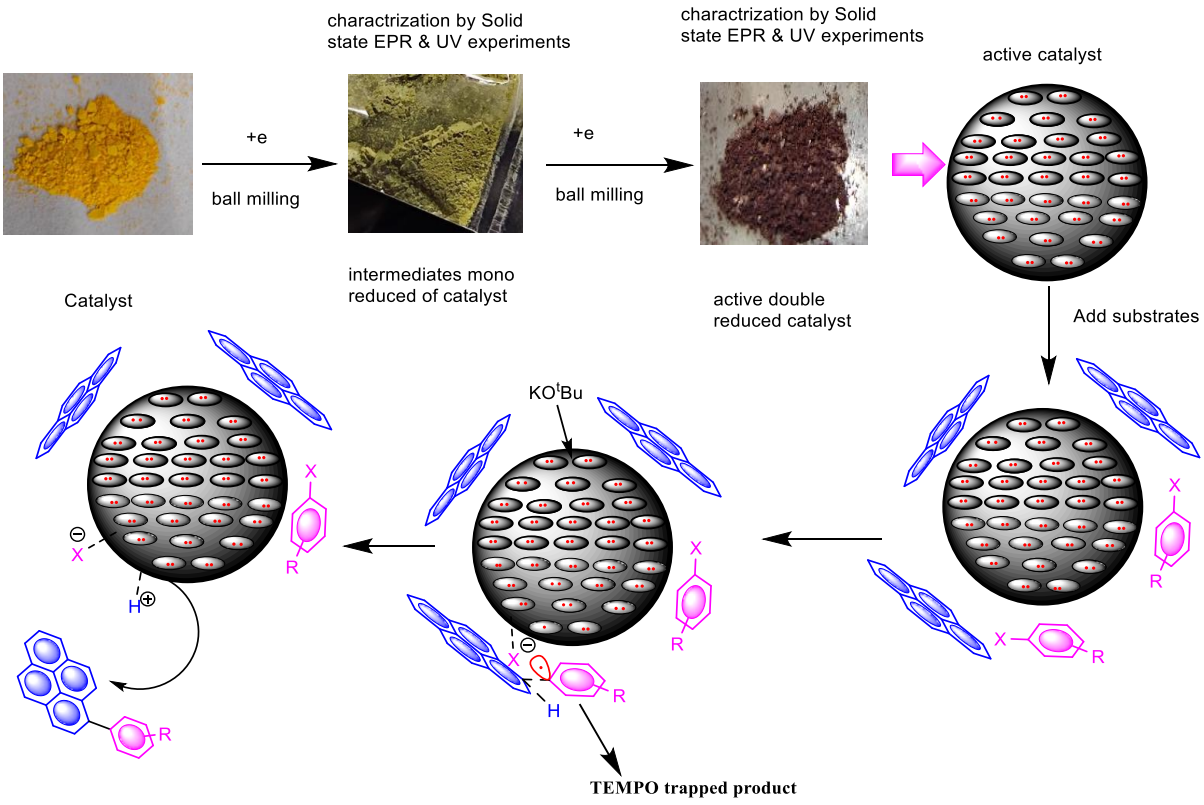


Fig. 25. Proposed Mechanism for Direct Arylation of Heteroarenes /Arenes /Polyarenes.

Proposed Catalytic Cycle for Solid-State Catalysis for Heck-type Coupling

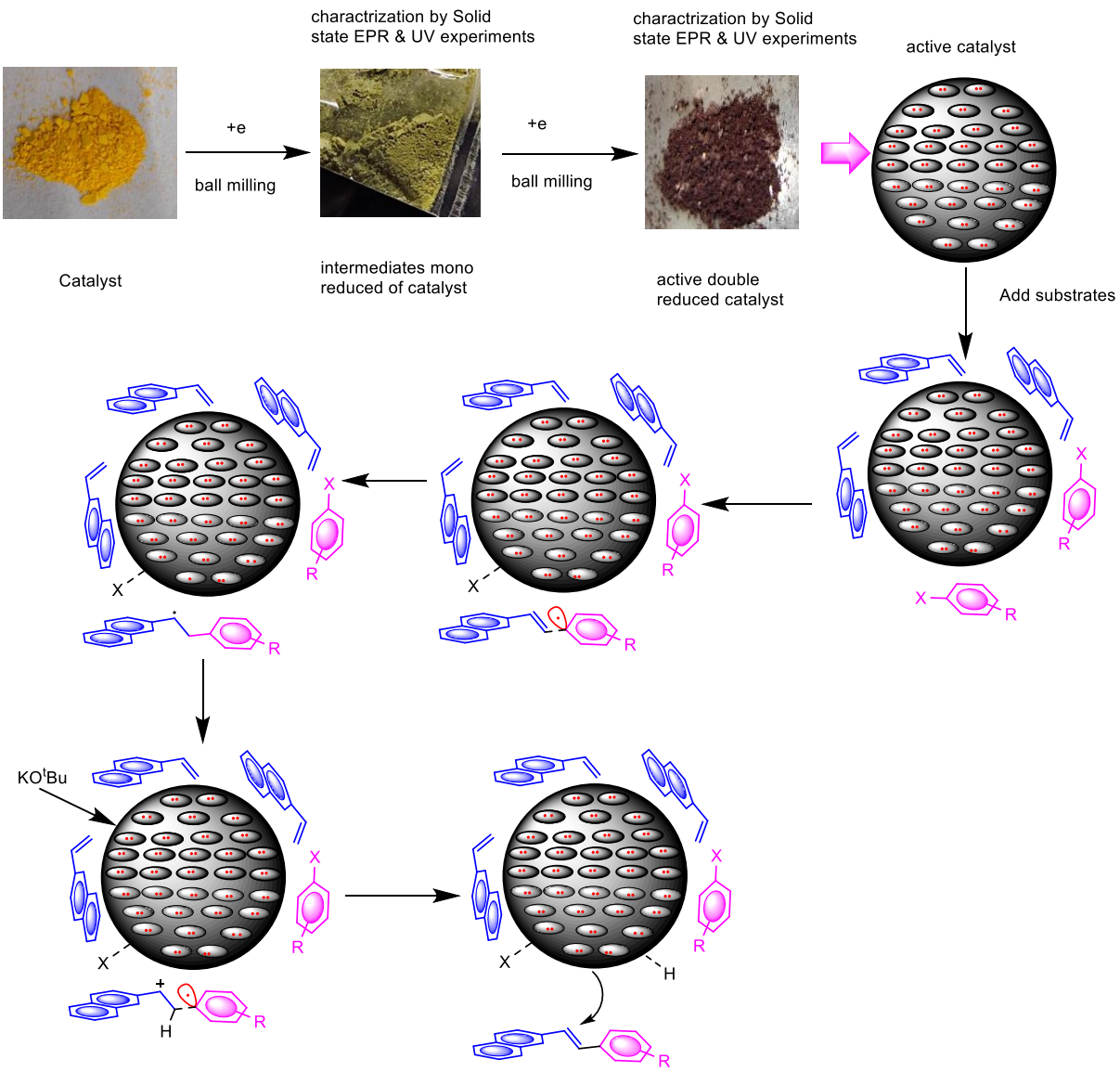


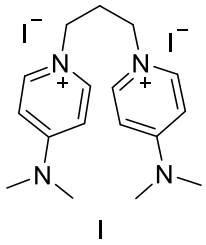
Fig. 26. Proposed Mechanism for Heck-type Coupling in Solid-State Catalysis.

20. ICP-OES Measurement:

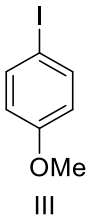
We performed ICP-OES measurements for detection of ^{105}Pd Trace metal into the catalysts, OED salt and KO^{t}Bu and 4-iodoanisolene in ppb level.

Table S16: ICP-OES Measurement

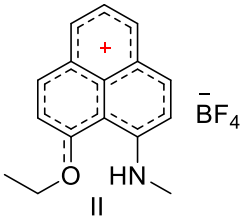
Entry	Compund	^{105}Pd (ppb)
I	OED salt	0.4
II	PLY cation	0.00
III	4-Iodoanisole	0.00
IV	KO^{t}Bu	0.1



I



III



II

KO^{t}Bu

IV

ICP-OES data:

1 = OED Salt

6 = KO^{t}Bu

7 = (N,O) PLY Cation

8 = 4-Iodoanisole

Table S17: ICP-OES Data

No	Date / Time	Label	Pd 340.458 {99} (Axial)
1	5/29/2022 9:53:51 AM	BLK	-20

No	Date / Time	Label	Pd 340.458 {99} (Axial)
2	5/29/2022 9:55:27 AM	STD 1	92

No	Date / Time	Label	Pd 340.458 {99} (Axial)
3	5/29/2022 9:57:02 AM	STD 2	472

No	Date / Time	Label	Pd 340.458 {99} (Axial)

4	5/29/2022 9:58:38 AM	STD 3	3,165

No	Date / Time	Label	Pd 340.458 {99} (Axial)
5	5/29/2022 10:00:12 AM	wash	-16

No	Date / Time	Label	Pd 340.458 {99} (Axial)
11	5/29/2022 10:09:43 AM	6	-19

No	Date / Time	Label	Pd 340.458 {99} (Axial)
12	5/29/2022 10:11:19 AM	7	-20

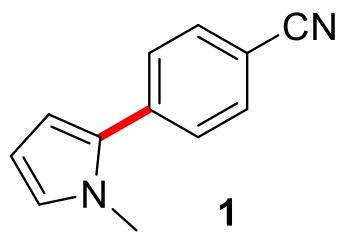
No	Date / Time	Label	Pd 340.458 {99} (Axial)
13	5/29/2022 10:12:55 AM	8	-20

No	Date / Time	Label	Pd 340.458 {99} (Axial)
14	5/29/2022 10:14:32 AM	1	-16

No	Date / Time	Label	Pd 340.458 {99} (Axial)
15	5/29/2022 10:16:09 AM	WASH	-13

21. Characterization and Analysis of Isolated Products.

4-(1-Methyl-1*H*-pyrrol-2-yl)benzonitrile (1)



1

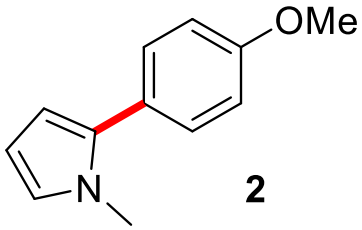
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol, 10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 4-iodobenzonitrile (68 mg, 0.3 mmol,1.0 equiv.) or 4-bromobenzonitrile (54 mg, 0.3 mmol, 1 equiv.) or 4-chlorobenzonitrile (41 mg, 0.3 mmol, 1 equiv.), *N*-methylpyrole (267 µL, 3 mmol, 10 equiv.). and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 4-(1-methyl-1*H*-pyrrol-2-yl)benzonitrile **1** was isolated by column chromatography (hexane : EA = 100: 5). Isolated yield X = I, 72%, (39 mg, white solid), X = Br, 61% (33 mg, white solid), X = Cl, 46% (25 mg, white solid),

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.67 (d, J = 8.4 Hz, 2H), 7.50 (d, J = 8.0 Hz, 2H), 6.78 (t, J = 2.2 Hz, 1H), 6.35 (dd, J_1 = 1.8 Hz, J_2 = 3.7 Hz, 1H), 6.24 – 6.20 (m, 1H), 3.71 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 137.8, 132.8, 132.4, 128.4, 126.0, 119.2, 110.9, 109.8, 108.7, 35.6.

The spectral data are matched with those reported in the literature (3).

2-(4-Methoxyphenyl)-1-methyl-1*H*-pyrrole (2)



2

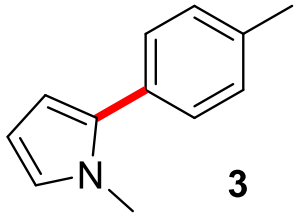
The reaction was performed according to general procedure with (*N,O*)-PLY cation I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 4-iodoanisole (70.2 mg, 0.3 mmol, 1.0 equiv.), N-methylpyrole (266 µL, 3 mmol, 10 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 2-(4-methoxyphenyl)-1-methyl-1*H*-pyrrole **2** was isolated by column chromatography (hexane: EA = 100: 2). Isolated yield, X = I, 65%, (36 mg, white solid)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.32 (m, 2H), 6.97 – 6.90 (m, 2H), 6.70-6.69 (m, 1H), 6.20-6.19 (m, 1H), 6.17-6.16 (m, 1H), 3.85 (s, 3H), 3.63 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.8, 138.3, 134.5, 126.2, 123.1, 113.9, 108.1, 107.7, 55.4, 35.0.

The spectral data are matched with those reported in the literature (4).

1-Methyl-2-(*p*-tolyl)-1*H*-pyrrole (3)



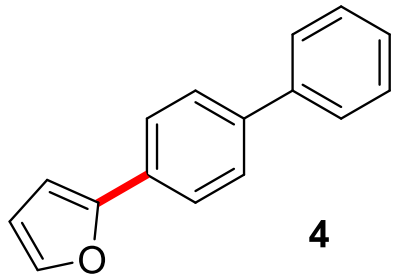
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 4-iodotoluene (65.4 mg, 0.3 mmol,1.0 equiv.), N-methylpyrole (266 µL, 3 mmol, 10 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 1-methyl-2-(*p*-tolyl)-1*H*-pyrrole **3** was isolated by column chromatography (hexane: EA = 100: 2). Isolated yield, X = I, 61%, (31 mg, liquid)

^{1}H NMR (400 MHz, CDCl3) δ: 7.29 (d, *J* = 8.0 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 6.69 (t, *J* = 2.2 Hz, 1H), 6.19 (d, *J* = 2.2 Hz, 2H), 3.64 (s, 3H), 2.38 (s, 3H).

^{13}C NMR (100 MHz, CDCl3) δ: 136.6, 134.8, 130.6, 129.2, 128.8, 123.4, 108.4, 107.8, 35.1, 31.7.

The spectral data are matched with those reported in the literature (4).

2-([1,1'-Biphenyl]-4-yl)furan (4)



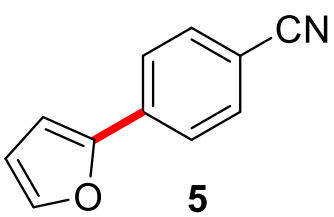
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), iodobenzonitrile (68 mg, 0.3 mmol,1.0 equiv.), furan (218 µL, 3 mmol, 10 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction with 4.5h, the desired product 2-([1,1'-biphenyl]-4-yl)furan **4** was isolated by column chromatography (hexane: EA = 100 : 5). Isolated yield, X = I, 59%, (38 mg, white solid)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.77–7.74 (m, 2H), 7.66 – 7.62 (m, 4H), 7.50-7.47 (m, 1H), 7.45-7.43 (m, 2H), 7.37-7.35 (m, 1H), 6.70 (d, *J* = 3.4 Hz, 1H), 6.51-6.49 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 153.9, 142.3, 140.8, 140.1, 130.0, 129.0, 127.5 (2C overlapped), 127.1, 124.3, 111.9, 105.3.

The spectral data are matched with those reported in the literature (5).

4-(Furan-2-yl)benzonitrile (5)



The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), iodobenzonitrile (68 mg, 0.3 mmol,1.0 equiv.), furan (218 µL, 3 mmol, 10 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 4-(furan-2-yl) benzonitrile **5** was isolated by

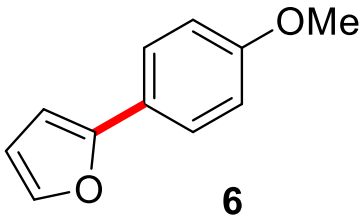
column chromatography (hexane: EA = 100 : 5). Isolated yield X = Cl, 40% (20 mg, white solid), X = Br, 57% (28 mg, white solid), X = I, 70%, (35 mg, white solid)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.75-7.73 (m, 2H), 7.72 – 7.62 (m, 2H), 7.54-7.53 (m, 1H), 6.81 (m, 1H), 6.53-6.52 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 152.1, 143.8, 138.6, 134.8, 132.7, 124.1, 119.1, 112.4, 108.3.

The spectral data are matched with those reported in the literature (6).

2-(4-Methoxyphenyl)furan (6)



6

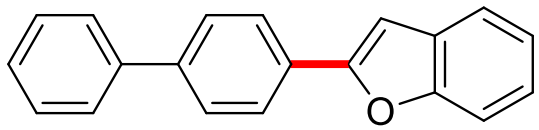
The reaction was performed according to general procedure with (*N*,*O*)-PLY cation I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), iodobenzonitrile (68 mg, 0.3 mmol,1.0 equiv.), thiophene (240 μL, 3 mmol, 10 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction with 3h, the desired product 2-(4-methoxyphenyl)furan **6** was isolated by column chromatography (hexane: EA = 100 : 5). Isolated yield, X = I, 60% (31 mg, white solid)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.62 (d, *J* = 8.8 Hz, 2H), 7.44 (s, 1H), 7.934-7.93 (m, 2H), 6.53-6.52 (m, 1H), 6.46 – 6.45 (m, 1H), 3.83 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.1, 154.1, 141.4, 125.3, 124.1, 114.2, 111.5, 103.4, 55.3.

The spectral data are matched with those reported in the literature (7).

2-([1,1'-Biphenyl]-4-yl)benzofuran (7)



7

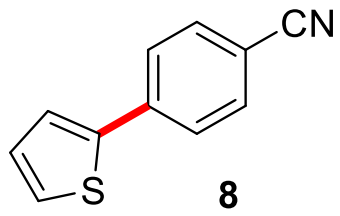
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %),), 4-iodo-1,1'-biphenyl (84 mg, 0.3 mmol, 1 equiv.), benzofuran (330 μL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction with 5h, the desired product 2-([1,1'-biphenyl]-4-yl)benzofuran **7** was isolated by column chromatography (hexane: EA = 100 : 10). Isolated yield, X = I, 70%, (56 mg, white solid)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.95 (d, *J* = 8.3 Hz, 2H), 7.70 (d, *J* = 8.3 Hz, 2H), 7.65 (d, *J* = 7.6 Hz, 2H), 7.60 (d, *J* = 7.6 Hz, 1H), 7.55 (d, *J* = 8.1 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.38 (t, *J* = 7.3 Hz, 1H), 7.30 (m, 1H), 7.24 (m, 1H), 7.07 (s, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.8, 155.1, 141.4, 140.6, 129.5, 129.4, 129.0, 127.7, 127.6, 127.1, 125.5, 124.5, 123.1, 121.0, 111.3, 101.6.

The spectral data are matched with those reported in the literature (8).

4-(Thiophen-2-yl)benzonitrile (8)



8

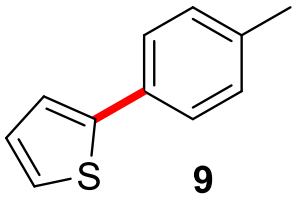
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 4-iodobenzonitrile (68 mg, 0.3 mmol, 1.0 equiv.) or 4-bromobenzonitrile (54 mg, 0.3 mmol, 1 equiv.) or 4-chlorobenzonitrile (41 mg, 0.3 mmol, 1 equiv.), thiophene (240 µL, 3 mmol, 10 equiv.). and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 4-(thiophen-2-yl) benzonitrile **8** was isolated by column chromatography (hexane: EA = 100 : 5). Isolated yield X = Cl, 45% (24 mg, white solid), X = Br, 60% (33 mg, white solid), X = I, 75%, (41 mg, white solid)

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.73 – 7.68 (m, 2H), 7.68 – 7.63 (m, 2H), 7.42 (dd, J_1 = 1.1 Hz, J_2 = 3.7 Hz, 1H), 7.40 (dd, J_1 = 1.1Hz, J_2= 5.1Hz, 1H), 7.13 (dd, J_1= 3.7 Hz, dd, J_2= 5.1Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 142.2, 138.8, 132.9, 128.7, 127.2, 126.2, 125.3, 119.0, 110.7.

The spectral data are matched with those reported in the literature (9).

2-(*p*-Tolyl)thiophene (9)



9

The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), iodotoluene (65.4 mg, 0.3 mmol, 1.0 equiv.), thiophene (240 µL, 3 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 2-(*p*-tolyl)thiophene **9** was isolated by column chromatography (hexane: EA = 100: 2). Isolated yield, X = I, 51%, (26 mg, white solid).

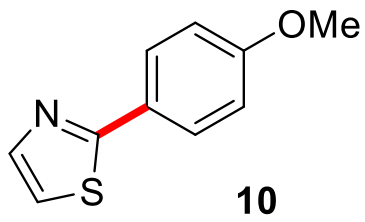
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.53 – 7.47 (m, 2H), 7.28 – 7.22 (m, 2H), 7.18 (m, 2H), 7.06 (dd, J_1 =3.6 Hz, J_2 = 4.9 Hz, 1H), 2.36 (s, 3H). Due to Grease and impurity the yield of this compound has 5-7% error.

^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.7, 137.5, 131.8, 129.7, 128.1, 126.0, 124.4, 122.7, 21.3.

The spectral data are matched with those reported in the literature (10).

2-(4-Methoxyphenyl)thiazole (10)

The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 4-iodoanisole (70.2 mg, 0.3 mmol, 1.0 equiv.), thiazole (213 µL, 3 mmol, 10 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.).



10

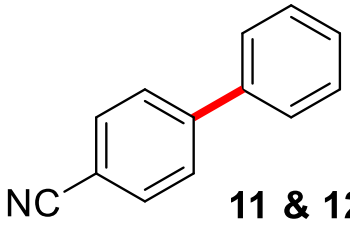
After completion of the reaction, the desired product 2-(4-methoxyphenyl)thiazole **10** was isolated by column chromatography (hexane : EA = 100: 2). Isolated yield, X = I, 67% (38 mg, white solid)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.90-7.88 (m, 2H), 7.79-7.78 (m, 1H), 7.24-7.23 (m, 1H), 6.96-6.93 (m, 2H), 3.84 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 168.5, 161.3, 143.5, 128.2, 126.7, 118.0, 114.4, 55.5.

The spectral data are matched with those reported in the literature (11).

[1,1'-Biphenyl]-4-carbonitrile (11 & 12)



11 & 12

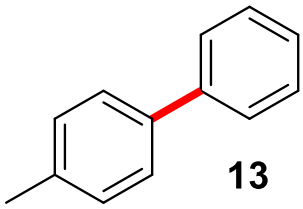
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 4-iodobenzonitrile (68 mg, 0.3 mmol, 1.0 equiv.), benzene (268 μL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product [1,1'-biphenyl]-4-carbonitrile **11** was isolated by column chromatography (hexane: EA = 100 : 2). Isolated yield, X = I, 80%, (42 mg, white solid), X = Br, 52%, (27 mg, white solid)

^{1}H NMR (400 MHz, $CHCl_3$) δ: 7.76 – 7.64 (m, 4H), 7.61 – 7.55 (m, 2H), 7.51 – 7.44 (m, 2H), 7.43-7.39 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 145.8, 139.3, 132.7, 129.3, 128.8, 127.9, 127.4, 119.1, 111.1.

The spectral data are matched with those reported in the literature (12).

4-Methyl-1,1'-biphenyl (13)



13

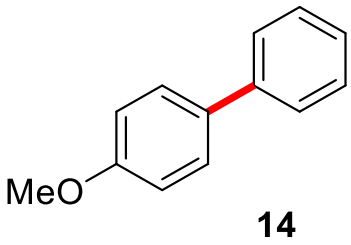
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 4-iodotoluene (65.4 mg, 0.3 mmol,1.0 equiv.), benzene (267 μL, 3.0 mmol, 10 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 4-methyl-1,1'-biphenyl **13** was isolated by column chromatography (hexane). Isolated yield 65% (31 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.58 (d, *J* = 7.8 Hz, 2H), 7.49 (t, *J* = 8.9 Hz, 2H), 7.43 (t, *J* = 7.7 Hz, 2H), 7.32 (t, *J* = 7.3 Hz, 1H), 7.24-7.23 (m, 2H), 2.40 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 141.3, 138.5, 137.2, 129.6, 128.8, 127.1, 127.1, 127.1, 21.2.

The spectral data are matched with those reported in the literature (13).

4-Methoxy-1,1'-biphenyl (14)



14

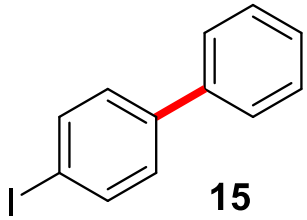
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 1-iodoanisole (70.2 mg, 0.3 mmol, 1.0 equiv.), benzene (267 µL, 3.0 mmol, 10 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 4-methoxy-1,1'-biphenyl **14** was isolated by column chromatography (hexane: EA = 100 : 5). Isolated yield 61% (33 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.57-7.52 (m, 4H), 7.42 (t, *J* = 7.3 Hz, 2H), 7.31 (m, 1H), 6.98 (d, *J* = 8.4 Hz, 2H), 3.86 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 159.3, 141.0, 133.9, 128.9, 128.3, 126.9, 126.8, 114.4, 55.5.

The spectral data are matched with those reported in the literature (13).

4-Iodo-1,1'-biphenyl (15)



15

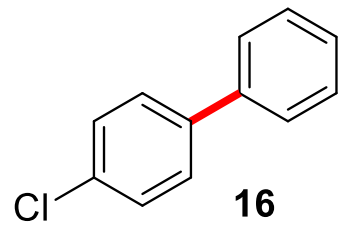
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 1,4-diiodobenzene (98.7 mg, 0.3 mmol, 1.0 equiv.), benzene (267 µL, 3.0 mmol, 10 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 4-iodo-1,1'-biphenyl **15** was isolated by column chromatography (hexane). Isolated yield 70% (58 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.81 – 7.73 (m, 2H), 7.56 – 7.54 (m, 2H), 7.46-7.42 (m, 2H), 7.38 – 7.32 (m, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 141.9, 140.2, 138.0, 129.2, 129.0, 127.8, 127.0, 93.2.

The spectral data are matched with those reported in the literature (14).

4-Chloro-1,1'-biphenyl (16)



16

The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 1-chloro-4-iodobenzene (71.4 mg, 0.3 mmol, 1.0 equiv.), benzene (267 µL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 4-chloro-1,1'-biphenyl **16** was isolated by column chromatography (hexane). Isolated yield 45% (25 mg, white solid).

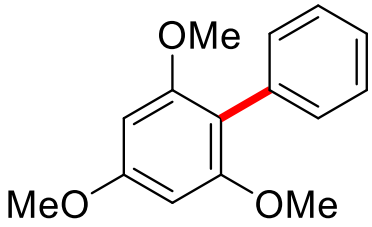
^{1}H NMR (400 MHz, CDCl3) δ: 7.64 – 7.49 (m, 4H), 7.49 – 7.32 (m, 5H).

^{13}C NMR (100 MHz, CDCl3) δ 140.2, 139.8, 133.5, 129.0 (2 C peak overlapped), 128.5, 127.7, 127.1.

The spectral data are matched with those reported in the literature (15).

2,4, 6-Trimethoxy-1,1'-biphenyl (17)

The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), iodobenzene (34 μL, 0.3 mmol,1.0 equiv.), Trimethoxybenzene (352 mg, 2.1 mmol, 7 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction with 4h, the desired product 2, 4, 6-trimethoxy-1,1'-biphenyl **17** was isolated by column chromatography (hexane : EA = 100 : 5). Isolated yield 57% (41 mg, white solid).



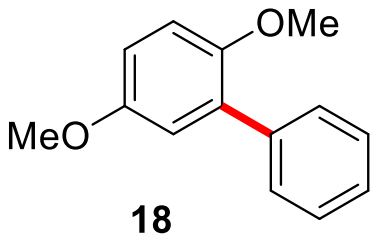
17

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.58 – 7.12 (m, 5H), 6.23 (s, 2H), 3.87 (s, 3H), 3.72 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 160.7, 158.5, 134.3, 131.3, 127.8, 126.6, 112.7, 91.1, 56.0, 55.5.

The spectral data are matched with those reported in the literature (16).

2, 5-Dimethoxy-1, 1'-biphenyl (18)



18

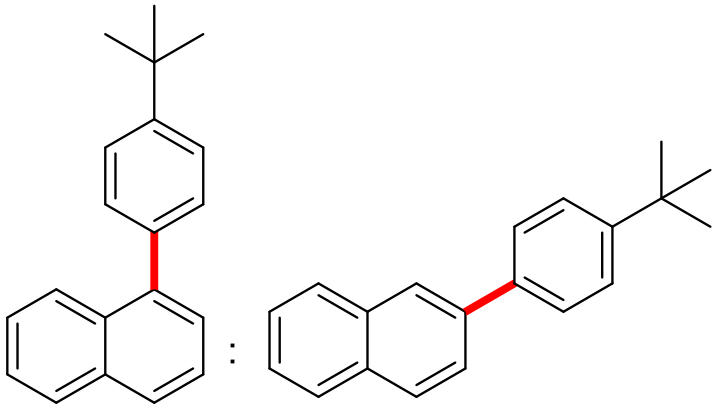
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol%), OED salt (32.4 mg, 0.06 mmol, 20 mol %), iodobenzene (34 μL, 0.3 mmol, 1.0 equiv.), dimethoxybenzene (289 mg, 2.1 mmol, 7 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction with 2.5h, the desired product 2, 5-dimethoxy-1, 1'-biphenyl **18** was isolated by column chromatography (hexane : EA = 100 : 5). Isolated yield 61% (39 mg, solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.53 (d, *J* = 7.3 Hz, 2H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.33 (t, *J* = 7.4 Hz, 1H), 6.93-6.90 (m, 2H), 6.87-6.84 (m, 1H), 3.81 (s, 3H), 3.75 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 153.9, 151.0, 138.6, 131.9, 129.6, 128.2, 127.2, 116.9, 113.3, 112.9, 56.5, 56.0.

The spectral data are matched with those reported in the literature (17).

1-(4-(*tert*-Butyl) phenyl) naphthalene and 2-(4-(*tert*-Butyl) phenyl) naphthalene (19 & 19')



19 & 19'

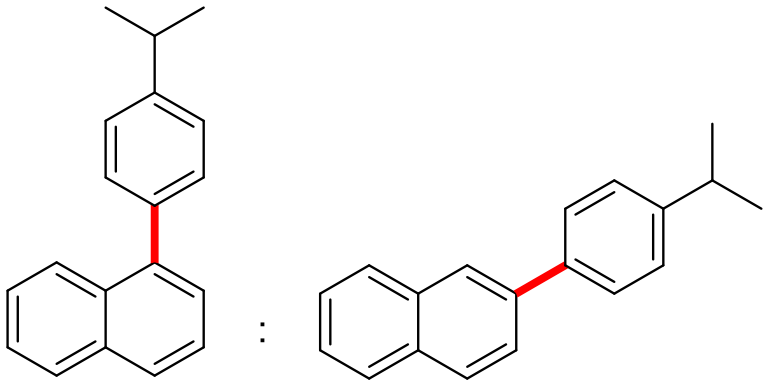
The reaction was performed according to general procedure with PLY I (13.0 mg mg, 0.04 mmol,10 mol %), OED salt (44 mg, 0.08 mmol, 20 mol), 1-butyl-4-iodobenzene (69 µL, 0.04 mmol, 1 equiv.), naphthalene (358 mg, 2.8 mmol, 7 equiv.) and KO^{t}Bu (153 mg, 1.36 mmol, 3.4 equiv.), After completion of the reaction, the desired product was isolated as regioisomeric mixture of 1-(4-(*tert*-butyl) phenyl)naphthalene and 2-(4-(*tert*-butyl) phenyl)naphthalene **19 & 19'** by column chromatography (hexane). Isolated yield 44% (45 mg, solid, α:β = 1:0.51).

^{1}H NMR (500 MHz, CDCl3) δ: Major isomer 7.96-7.98 (m, 1H), 7.90-7.92 (m, 2H), 7.84-7.86 (m, 1H), 7.67-7.69 (m, 1H), 7.43-7.53(m, 6H), 1.42 (s, 9H). Characteristic peaks of minor isomer 8.04 ppm, 7.75-7.77 ppm, 1.39 ppm.

^{13}C NMR (125 MHz, $CDCl_3$) δ: Major isomer 150.2, 140.4, 137.9, 134.0, 131.9, 129.9, 128.4, 127.5, 127.0, 126.3, 126.0, 125.8, 125.5, 125.3, 34.8, 31.6, Minor isomer 150.6, 138.6, 138.3, 133.9, 132.7, 128.5, 128.3, 127.8, 127.2, 126.0, 125.9, 125.8,125.7, 125.7, 34.7, 31.5.

The spectral data are matched with those reported in the literature (18).

1-(4-Isopropylphenyl)naphthalene and 2-(4-Isopropylphenyl)naphthalene (20 & 20')



20 & 20'

The reaction was performed according to general procedure with PLY I (13 mg, 0.04 mmol,10 mol%), OED salt (44 mg, 0.08 mmol), 1-iodo-4-isopropylbenzene (65 µL, 0.4 mmol, 1 equiv.), naphthalene (358 mg, 2.8 mmol, 7 equiv.) and KO^{t}Bu (153 mg, 1.36 mmol, 3.4 equiv.), After completion of the reaction, the desired mixture of (isopropyl phenyl)naphthalene and 2-(4-isopropyl phenyl)naphthalene **20**

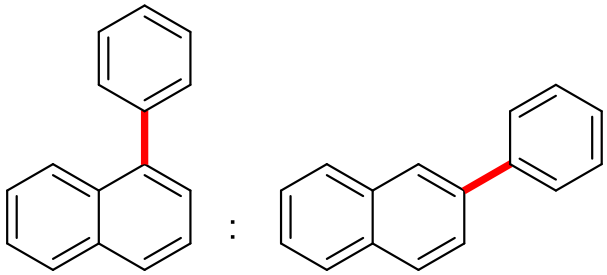
& 20' was isolated by column chromatography (hexane). Isolated yield 65% (63 mg, solid, α:β = 1:0.81).

^{1}H NMR (400 MHz, CDCl3) δ: Major isomer 7.96 (m, 1H), 7.93 – 7.82 (m, 2H), 7.67 (d, *J* = 8.2 Hz, 1H), 7.56 – 7.39 (m, 5H), 7.36 (d, *J* = 7.8 Hz, 2H), 3.05-2.95 (m, 1H), 1.35 (d, *J* = 7.0 Hz, 6H). Characteristic peaks of minor isomer 8.03 (s), 7.96 (d, *J* = 8.3 Hz, 1H), 7.75 (dd, *J* = 8.5, 1.7 Hz, 1H), 1.32 (d, *J* = 6.9 Hz).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 148.3, 148.0, 140.5, 138.8, 138.3, 134.0, 133.9, 132.7, 131.9, 130.1(2C), 128.5, 128.4, 128.3, 127.8, 127.5, 127.5, 127.1, 127.0, 126.5, 126.3, 126.3, 126.0, 125.9, 125.8, 125.8, 125.6, 125.5, 34.0, 34.0, 24.2, 24.2. (Indistinguishable peaks for both the isomers).

The spectral data are matched with those reported in the literature (19).

1-Phenylnaphthalene and 2-Phenylnaphthalene (21 & 21')



21 &21'

The reaction was performed according to general procedure with PLY I (13.0 mg, 0.04 mmol,10 mol %), OED salt (32.4 mg, 0.08 mmol, 20 mol %), iodobenzene (44 μL, 0.4 mmol, 1.0 equiv.), naphthalene (358 mg, 2.8 mmol, 7 equiv.) and KO^{t}Bu (153 mg, 1.36 mmol, 3.4 equiv.). After completion of the reaction with 3h, the desired product 1-phenylnaphthalene and 2-phenylnaphthalene **21 & 21'** was isolated by column chromatography (hexane). Isolated yield 70% (57 mg, white solid, α:β = 1:0.78).

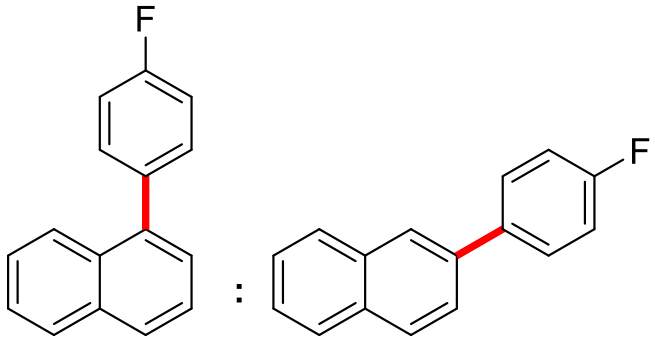
^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.05 (s, 1H), 7.89 (m, 6H), 7.78 -7.70 (m, 2H), 7.56-7.35 (m, 15H). Non distinguishable mixture of minor and major product

^{13}C NMR (125 MHz, $CDCl_3$) δ: 141.3, 140.9, 140.4, 138.7, 134.0, 133.9, 132.8, 131.8, 130.2, 129.0, 128.6, 128.4, 128.3, 127.8, 127.8, 127.6, 127.5, 127.4, 127.1, 126.4, 126.2, 126.1, 126.1, 126.0, 126.0, 125.9, 125.7, 125.5. Both major and minor product peaks are indistinguishable.

The spectral data are matched with those reported in the literature (20).

1-(4-Fluorophenyl)naphthalene and 2-(4-Fluorophenyl)naphthalene (22 & 22')

The reaction was performed according to general procedure with PLY I (13.0 mg, 0.04 mmol,10 mol %), OED salt 43.2 mg, 0.08 mmol, 20 mol), 1-iodo-4-Fluorobenzene (46 μL, 0.4 mmol, 1 equiv.), naphthalene (358 mg, 2.8 mmol, 7 equiv.) and KO^{t}Bu (153.0 mg, 1.36 mmol,



22 & 22'

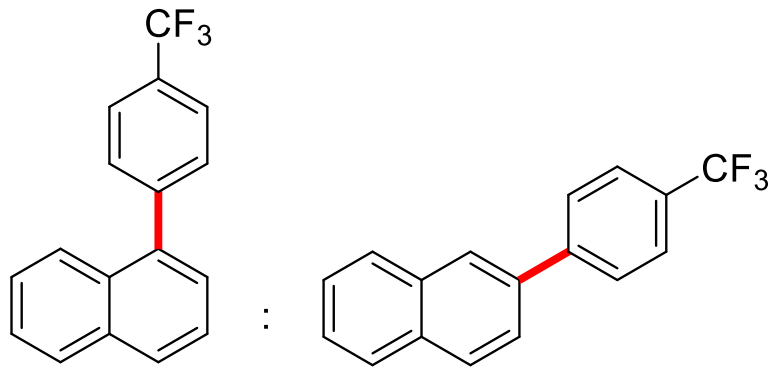
3.4 equiv.). After completion of the reaction, the desired 1-(4-fluorophenyl)naphthalene and 2-(4-fluorophenyl)naphthalene **22 & 22'** was isolated by column chromatography (hexane). Isolated yield 55% (48 mg, white solid, α:β = 1:0.80).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.95 – 7.81 (m, 6H), 7.77 – 7.62 (m, 2H), 7.47 (m, 9H), 7.24 – 7.11 (m, 4H). Mixture of major and minor products were isolated**.** For minor product characterisation peak 7.99 (s, 1H) was observed.

^{13}C NMR: (125 MHz, $CDCl_3$) δ: 162.6 (d, *J* = 246 Hz), 162.4 (d, *J* = 246 Hz), 139.3, 137.7, 137.4 (d, *J* = 3.9 Hz), 136.8 (d, *J* = 3.2 Hz), 133.9, 133.9, 132.7 (d, *J* = 3.8 Hz), 131.8, 131.7 (d, *J* = 8.0 Hz), 129.1 (d, *J* = 8.1 Hz), 128.7, 128.5, 128.3, 127.9, 127.8, 127.7, 127.1, 126.5, 126.5, 126.3, 126.1, 127.0, 125.9, 125.5, 115.9 (d, *J* = 21.3 Hz), 115.3 (d, *J* = 21.3 Hz). Indistinguishable peaks for the regioisomers.

The spectral data are matched with those reported in the literature (21).

1-(4-(Trifluoromethyl)phenyl)naphthalene and 2 (4(Trifluoromethyl)phenyl)naphthalene (23 & 23')



23 & 23'

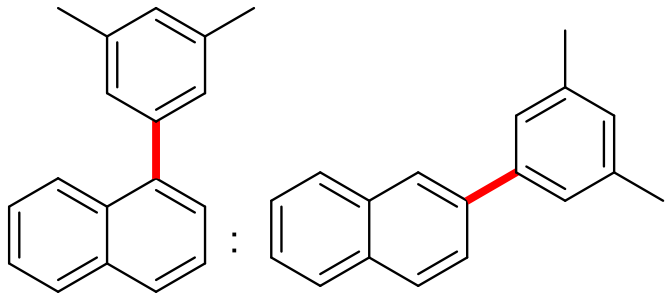
The reaction was performed according to general procedure with PLY I (13.0 mg, 0.04 mmol,10 mol %), OED salt (44 mg, 0.06 mmol, 20 mol),1-iodo-4-trifluoromethylbenzene (58 μL, 0.4 mmol, 1 equiv.), naphthalene (358 mg, 2.8 mmol, 7 equiv.) and KO^{t}Bu (115 mg, 1.36 mmol, 3.4 equiv.). After completion of the reaction, the desired product 1-(4-trifluoromethyl)phenyl)naphthalene and 2-(4-trifluoromethyl)phenyl)naphthalene **23 & 23'** was isolated by column chromatography (hexane). Isolated yield 60% (65 mg, white solid, α:β = 1:70).

^{1}H NMR (500 MHz, $CDCl_3$) δ: both major and minor isomer mixture was isolated. For major isomer 7.99 – 7.87 (m, 2H), 7.84-7.73 (m, 3H), 7.69 – 7.38 (m, 6H).the distinguishable peak of minor isomer 8.07 (s, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: Both major and minor peaks are non-distinguishable.144.83, 144.64, 138.87, 137.20, 133.93, 133.70, 133.13, 131.41, 130.57, 130.54, 128.92, 128.58, 128.53, 128.46, 127.85, 127.82, 127.15, 126.75, 126.63, 126.58, 126.51, 126.18, 126.08, 125.94(q, *J* = 3.7 Hz), 125.6 (q, *J* = 3.7 Hz), 125.56, 125.49, 125.41, 125.37, 125.35.

The spectral data are matched with those reported in the literature (22).

1-(3,5-Dimethylphenyl)naphthalene and 2-(3,5-Dimethylphenyl)naphthalene (24 & 24')



24 & 24'

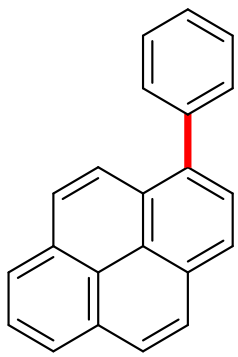
The reaction was performed according to general procedure with PLY I (13 mg, 0.04 mmol,10 mol %), OED salt (43.2 mg, 0.08 mmol, 20 mol), 1-iodo-3,5-dimethylbenzene (58 μL, 0.4 mmol, 1 equiv.), naphthalene (358 mg, 2.8 mmol, 7 equiv.) and KO^{t}Bu (153 mg, 1.36 mmol, 3.4 equiv.). After completion of the reaction, the desired product was isolated as the regioisomeric mixture of 1-(3,5-dimethylphenyl)naphthalene and 2-(3,5-dimethylphenyl)naphthalene **24 & 24'** by column chromatography (hexane). Isolated yield 58% (53 mg, solid, white colour solid, α:β = 1:0.65).

^{1}H NMR (400 MHz, $CDCl_3$) δ: Minor isomer's characterization peak 8.03 (s, 1H), 7.75 – 7.73 (m, 1H), 7.58 – 7.38 (m, 1H). Major isomer's characterization peak 7.94 – 7.83 (m, 3 H), 7.53-7.40 (m, 4H), 7.20 – 6.87 (m, 3H).

^{13}C NMR: (125 MHz, $CDCl_3$) δ: 141.1, 140.7, 140.5, 138.8, 138.4, 137.7, 133.8, 133.7, 131.7, 129.0, 128.8, 128.2, 128.2, 128.2, 127.9, 127.6, 127.6, 127.6, 127.4, 126.7, 126.2, 125.9, 125.8, 125.8, 125.7, 125.7, 125.7, 125.3, 21.5, 21.4. Indistinguishable peaks for both the isomers.

The spectral data are matched with those reported in the literature (23).

1-Phenylpyrene (25)



25

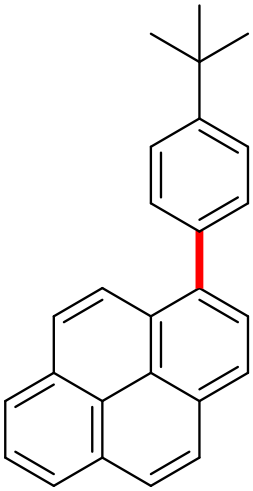
The reaction was performed according to general procedure with PLY I (13 mg, 0.04 mmol,10 mol %), OED salt (43.2 mg, 0.08 mmol, 20 mol), iodobenzene (44 μL, 0.4 mmol, 1.0 equiv.), pyrene (404.0 mg, 2.0 mmol, 5 equiv.) and KO^{t}Bu (153 mg, 1.36 mmol, 3.4 equiv.), After completion of the reaction with 6 h, the desired product 1-phenylpyrene **25** was isolated by column chromatography (hexane). Isolated yield 72% (80 mg, yellow solid) & 55% (61 mg) .

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.27 – 8.14 (m, 4H), 8.10 (s, 2H), 8.06-7.93 (m, 3H), 7.71-7.61 (m, 2H), 7.61-7.54 (m, 2H), 7.54-7.45 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 141.4, 137.9, 131.6, 131.1, 130.7, 130.3, 128.6, 128.5, 127.9, 127.7, 127.6, 127.4, 126.1, 125.9, 125.4, 125.2, 125.0, 124.8, 124.1.

The spectral data are matched with those reported in the literature (24).

1-(4-(*tert*-Butyl)phenyl)pyrene (26)



26

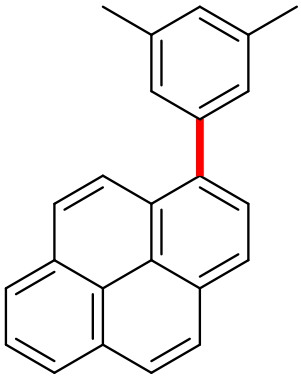
The reaction was performed according to general procedure with PLY I (13 mg, 0.03 mmol,10 mol %), OED salt (43.2 mg, 0.06 mmol, 20 mol),), 1-butyl-4-iodobenzene (69 μL, 0.03 mmol, 1 equiv.), pyrene (303 mg, 1.5 mmol, 5 equiv.) and KO^{t}Bu (153 mg, 1.36 mmol, 3.4 equiv.), After completion of the reaction, the desired 1-(4-(*tert*-butyl)phenyl)pyrene **26** was isolated by column chromatography (hexane). Isolated yield 60% (80 mg, yellow solid).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.29 – 8.15 (m, 4 H), 8.10 (d, *J* = 0.8 Hz, 2 H), 8.05 – 7.93 (m, 3 H), 7.59 (s, 4H), 1.46 (s, 9H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 150.3, 138.3, 137.9, 131.7, 131.2, 130.6, 130.4, 129.9, 128.7, 127.9, 127.8, 127.6, 127.4, 126.1, 125.7, 125.5, 125.5, 125.1, 124.9, 124.8, 34.8, 31.6.

HRMS Calculated mass for [$C_{16}H_{22}N_2O$ + H^+] is 335.1794 and the observed mass 335.1806.

1-(3, 5-Dimethylphenyl)pyrene (27)



27

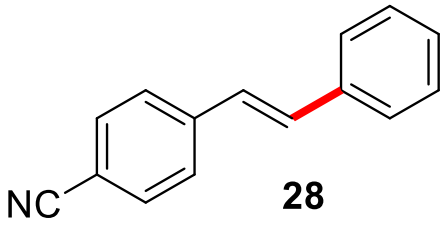
The reaction was performed according to general procedure with PLY I (13g, 0.04 mmol, 10 mol %), OED salt (32.4 mg, 0.08 mmol),), 1-iodo-3,5-dimethylbenzene (58 μL, 0.4 mmol, 1 equiv.), pyrene (404 mg, 2.0 mmol, 5 equiv.) and KO^{t}Bu (153.0 mg, 1.36 mmol, 3.4 equiv.), After completion of the reaction, the desired 1-(3, 5-dimethylphenyl)pyrene **27** was isolated by column chromatography (hexane). Isolated yield 72% (88 mg, light yellow solid).

^{1}H NMR (400 MHz, CDCl3) δ: 8.23-8.13 (m, 4H), 8.08 (s, 2H), 8.04 – 7.99 (m, 2H), 7.97 (m, 1H), 7.25 (m, 2H), 7.13 (s, 1H), 2.46 (s, 6H).

^{13}C NMR (100 MHz, CDCl3) δ: 141.3, 138.3, 138.0, 131.7, 131.2, 130.6, 129.0, 128.7, 128.6, 128.6, 128.0, 127.7, 127.6, 127.4, 126.1, 125.7, 125.2, 125.1, 124.9, 124.7, 21.6.

The spectral data are matched with those reported in the literature (25).

(*E*)-4-Styrylbenzonitrile (28)



28

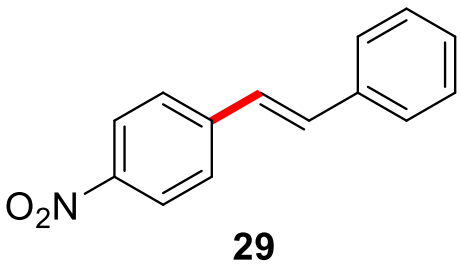
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol, 10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 4-iodobenzonitrile (68 mg, 0.3 mmol, 1 equiv.), styrene (229 µL, 2.0 mmol, 5 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction with 2.5h (*E*)-4-styrylbenzonitrile **28** was isolated by column chromatography (hexane: EA = 100: 5). Isolated yield 61% (37 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.64 (d, *J* = 8.4 Hz, 2H), 7.58 (d, *J* = 8.4 Hz, 2H), 7.54 (d, *J* = 7.3 Hz, 2H), 7.41-7.39 (m, 2H), 7.34-7.31 (m, 1H), 7.22 (d, *J* = 16.3 Hz, 1H), 7.09 (d, *J* = 16.3 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 142.0, 136.4, 132.6, 132.6, 129.0, 128.8, 127.1, 127.0, 126.9, 119.2, 110.7.

The spectral data are matched with those reported in the literature (26).

(*E*)-1-Nitro-4-styrylbenzene (29)



29

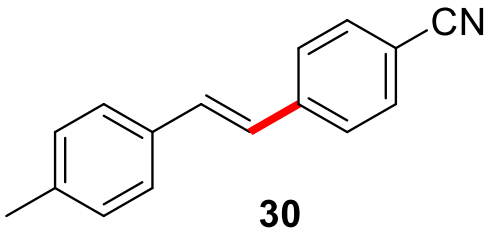
The reaction was performed according to general procedure with PLY I (9.75 mg, 0.03 mmol, 10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 4-iodobenzonitrile (69 mg, 0.3 mmol, 1.0 equiv.), styrene (229 µL, 2.0 mmol, 5 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction (*E*)-1-nitro-4-styrylbenzene **(29)** was isolated by column chromatography (hexane: EA = 100: 5). Isolated yield 62% (41 mg, yellow solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.24 (d, *J* = 8.7 Hz, 2H), 7.65 (d, *J* = 8.7 Hz, 2H), 7.57 (d, *J* = 7.7 Hz, 2H), 7.44-7.41 (m, 2H), 7.37-7.34 (m, 1H), 7.31-7.27 (m, 1H), 7.16 (d, *J* = 16.3 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 146.9, 143.9, 136.3, 133.4, 129.0, 128.9, 127.1, 126.9, 126.4, 124.2.

The spectral data are matched with those reported in the literature (27).

(*E*)-4-(4-Methylstyryl)benzonitrile (30)



30

The reaction was performed according to general procedure with PLY I (9.75 mg, 0.03 mmol, 10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 4-iodobenzonitrile (68 mg, 0.3 mmol, 1 equiv.), 4-methyl styrene (171 µL, 1.5 mmol, 5 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After

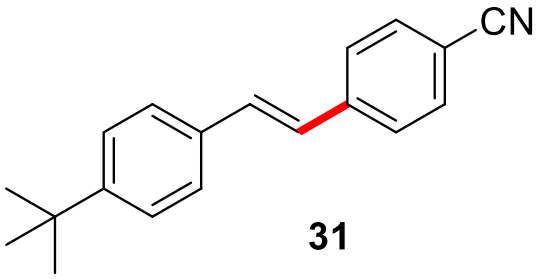
completion of the reaction (*E*)-4-(4-methylstyryl)benzonitrile **30** was isolated by column chromatography (hexane: EA = 100 : 5). Isolated yield 59% (38 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.62 (d, *J* = 8.2 Hz, 2H), 7.56 (d, *J* = 8.1 Hz, 2H), 7.43 (d, *J* = 7.8 Hz, 2H), 7.24 – 7.15 (m, 3H), 7.04 (d, *J* = 16.3 Hz, 1H), 2.38 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 142.2, 138.9, 133.7, 132.6, 132.5, 129.7, 127.0, 126.9, 125.9, 119.2, 110.5, 21.5.

The spectral data are matched with those reported in the literature (28).

(*E*)-4-(4-(*tert*-Butyl)styryl)benzonitrile (31)



31

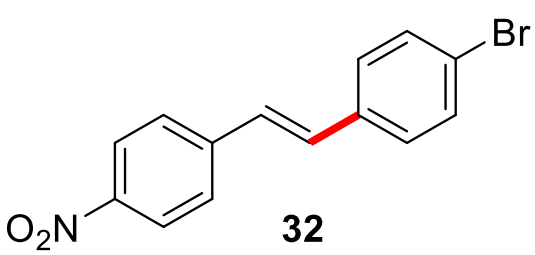
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol, 10 mol %), OED salt (44 mg, 0.06 mmol, 20 mol %), 4-iodobenzonitrile (68 mg, 0.3 mmol, 1 equiv.), 4-*tert*-butyl styrene (274 µL, 1.5 mmol, 5 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction with 5h (*E*)-4-(4-(*tert*-butyl)styryl)benzonitrile **31** was isolated by column chromatography (hexane: EA = 100: 5). Isolated yield 72% (56 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.62 (d, *J* = 8.0 Hz, 2H), 7.57 (d, *J* = 8.2 Hz, 2H), 7.47 (d, *J* = 8.3 Hz, 2H), 7.41 (d, *J* = 8.2 Hz, 2H), 7.20 (d, *J* = 16.3 Hz, 1H), 7.05 (d, *J* = 16.3 Hz, 1H), 1.34 (s, 9H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 152.0, 142.1, 133.5, 132.5, 132.3, 126.8, 126.7, 125.9, 125.8, 119.1, 110.3, 34.8, 31.2.

The spectral data are matched with those reported in the literature (29).

(*E*)-1-Bromo-4-(4-nitrostyryl)benzene (32)



32

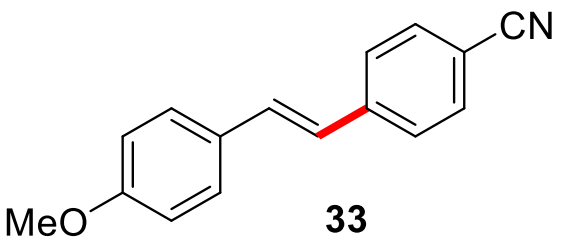
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.04 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol), 1-iodo-4-trifluoromethylbenzene (44 µL, 0.4 mmol, 1 equiv.), 4-bromostyrene (196 µL, 2.0 mmol, 5 equiv.) and KO^{t}Bu (115 mg, 1.36 mmol, 3.4 equiv.). After completion of the reaction (*E*)-1-bromo-4-(4-nitrostyryl)benzene **(32)** was isolated by column chromatography (hexane : EA = 100 : 5). Isolated yield 55% (50 mg, yellow solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.23 (d, *J* = 8.6 Hz, 2H), 7.63 (d, *J* = 8.7 Hz, 2H), 7.51 (t, *J* = 9.8 Hz, 2H), 7.41 (d, *J* = 8.4 Hz, 2H), 7.20 (d, *J* = 16.3 Hz, 1H), 7.13 (d, *J* = 16.3 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 147.1, 143.6, 134.8, 134.7, 132.1, 129.3, 128.3, 127.1, 127.0, 124.3.

The spectral data are matched with those reported in the literature (30).

(*E*)-4-(4-Methoxystyryl)benzonitrile (33)



33

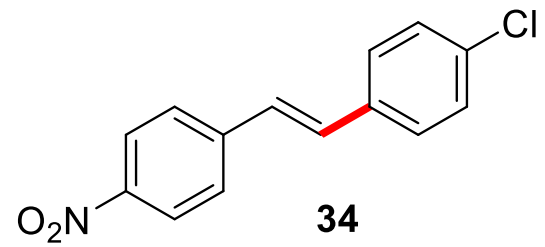
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol, 10 mol %), OED salt (44 mg, 0.06 mmol, 20 mol %), 4-iodobenzonitrile (68 mg, 0.3 mmol, 1 equiv.), 4-methoxystyrene (201 µL, 2.0 mmol, 5 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction (*E*)-4-(4-methoxystyryl)benzonitrile **33** was isolated by column chromatography (hexane: EA = 100 : 10). Isolated yield 69% (53 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.61 (d, *J* = 8.1 Hz, 2H), 7.54 (d, *J* = 8.1 Hz, 2H), 7.47 (d, *J* = 8.5 Hz, 2H), 7.16 (d, *J* = 16.3 Hz, 1H), 6.96 - 6.91 (m, 3H), 3.84 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 160.2, 142.4, 132.6, 132.1, 129.2, 128.4, 126.7, 124.7, 119.3, 114.4, 110.2, 55.5.

The spectral data are matched with those reported in the literature (31).

(*E*)-1-Chloro-4-(4-nitrostyryl)benzene (34)



34

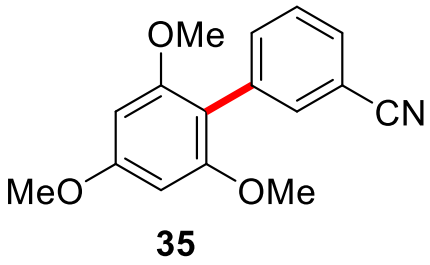
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol, 10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 1-chloro-4-iodobenzene (71.0 mg, 0.3 mmol, 1.0 equiv.), 4-methoxystyrene (201 µL, 1.5 mmol, 5 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction (*E*)-1-chloro-4-(4-methoxystyryl) benzene **34** was isolated by column chromatography (hexane : EA = 100 : 5). Isolated yield 62% (48 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.23 (d, *J* = 8.7 Hz, 2H), 7.63 (d, *J* = 8.7 Hz, 2H), 7.48 (d, *J* = 8.4 Hz, 2H), 7.37 (d, *J* = 8.4 Hz, 2H), 7.21 (d, *J* = 16.4 Hz, 1H), 7.11 (d, *J* = 16.3 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 147.1, 143.6, 134.8, 134.7, 132.1, 129.3, 128.3, 127.1, 127.0, 124.3.

The spectral data are matched with those reported in the literature (30)

2',4',6'-Trimethoxy-[1,1'-biphenyl]-3-carbonitrile (35)



35

The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol),), 3-iodobenzonitrile (68.7 mg, 0.3 mmol, 1 equiv.), trimethoxybenzene (352 mg, 2.1 mmol, 7 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.), After completion of the reaction, the 2, 4, 6-trimethoxy-1,1':4',1''-terphenyl **35** was isolated by column chromatography (hexane : EA = 100 : 10). Isolated yield X= I, 72% (58 mg, white solid) & X = Cl, 58% (46 mg) .

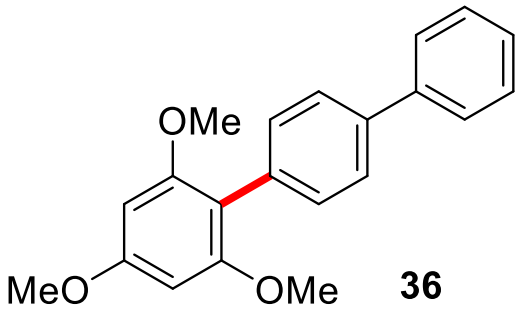
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.64 (s, 1H), 7.60 – 7.52 (m, 2H), 7.46 (d, *J* = 7.7 Hz, 1H), 6.22 (s, 2H), 3.87 (s, 3H), 3.73 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 161.4, 158.3, 136.2, 135.6, 135.3, 130.0, 128.4, 119.6, 111.9, 110.1, 91.0, 55.9, 55.6.

HRMS Calculated mass for [$C_{16}H_{22}N_2O + Na^+$] is 292.0944 and the observed mass 292.0924.

2, 4, 6-Trimethoxy-1,1':4',1''-terphenyl (36)

The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol),), 4-iodo-1,1'-biphenyl (84 mg, 0.3 mmol, 1 equiv.), trimethoxybenzene (352 mg, 2.1 mmol, 7 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.), After completion of the reaction with 3h, the 2, 4, 6-trimethoxy-1,1':4',1''-terphenyl **36** was isolated by column chromatography (hexane : EA = 100 : 10). Isolated yield X= I, 75% (72 mg, yellow solid) & X= Cl, 0%.



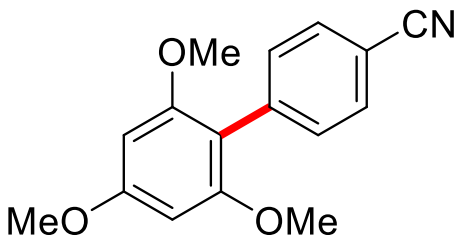
36

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.66-6.62 (m, 4H), 7.53 – 7.40 (m, 4H), 7.33 (t, *J* = 7.4 Hz, 1H), 6.26 (s, 2H), 3.88 (s, 3H), 3.76 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 160.7, 158.6, 141.5, 139.3, 133.3, 131.7, 128.8, 127.3, 127.1, 126.6, 112.2, 91.1, 56.1, 55.5.

The spectral data are matched with those reported in the literature (32).

2',4',6'-Trimethoxy-[1,1'-biphenyl]-4-carbonitrile (37)



37

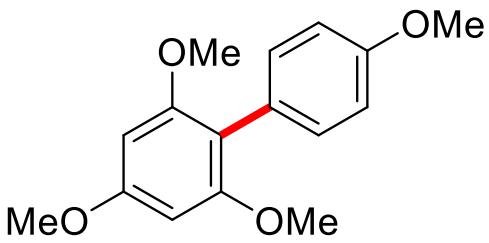
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol),), 4-iodobenzonitrile (68 mg, 0.3 mmol, 1 equiv.), trimethoxybenzene (352 mg, 2.1 mmol, 7 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.), After completion of the reaction, the 2',4',6'-trimethoxy-[1,1'-biphenyl]-4-carbonitrile **37** was isolated by column chromatography (hexane : EA = 100 : 10). Isolated yield X= I, 69% (55 mg, white solid) & X= Cl, 48% (38 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66 (d, *J* = 8.2 Hz, 2H), 7.47 (d, *J* = 8.1 Hz, 2H), 6.25 (s, 2H), 3.89 (s, 3H), 3.75 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 161.5, 158.2, 139.7, 132.3, 131.4, 119.6, 110.6, 109.9, 91.0, 55.9, 55.6.

The spectral data are matched with those reported in the literature (32).

2,4, 4',6-Tetramethoxy-1,1'-biphenyl (38)



38

The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol),), 4-iodoanisole (70.2 mg, 0.3 mmol, 1.0 equiv.), trimethoxybenzene (352 mg, 2.1 mmol, 7 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.), After completion of the reaction with 4.5 h, the 2,4, 4',6-tetramethoxy-1,1'-biphenyl **38** was isolated by column chromatography (hexane : EA = 100 : 10). Isolated yield 80% (65 mg, yellow solid).

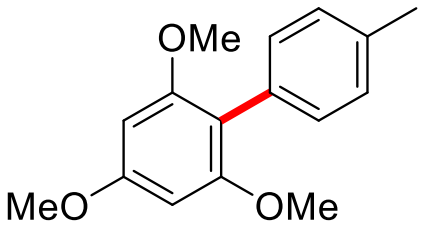
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.26 (d, *J* = 58.7 Hz, 2H), 6.94 (d, *J* = 8.7 Hz, 2H), 6.23 (s, 2H), 3.86 (s, 3H), 3.83 (s, 3H), 3.73 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 160.4, 158.6, 158.2, 132.3, 126.3, 113.4, 112.3, 91.1, 56.0, 55.5, 55.3.

The spectral data are matched with those reported in the literature (32).

2, 4, 6-Trimethoxy-4'-methyl-1,1'-biphenyl (39)

The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol, 10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol),), 4-iodotoluene (65.4 mg, 0.3 mmol, 1.0 equiv.), trimethoxybenzene (352 mg, 2.1 mmol, 7 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4



39

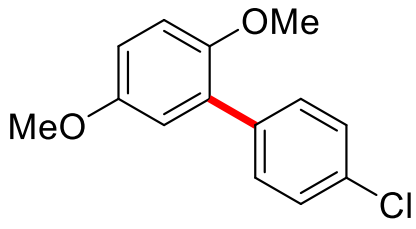
equiv.), After completion of the reaction, the 2, 4, 6-trimethoxy-4'-methyl-1,1'-biphenyl **39** was isolated by column chromatography (hexane : EA = 100 : 10). Isolated yield 65% (50 mg, whhite solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.23 (d, *J* = 8.2 Hz, 2H), 7.20 (d, *J* = 8.1 Hz, 2H), 6.23 (s, 2H), 3.87 (s, 3H), 3.72 (s, 6H), 2.38 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 160.5, 158.5, 136.1, 131.2, 131.1, 128.6, 112.6, 91.0, 56.0, 55.5, 21.5.

The spectral data are matched with those reported in the literature (33).

4'-Chloro-2, 5-dimethoxy-1,1'-biphenyl (40)



40

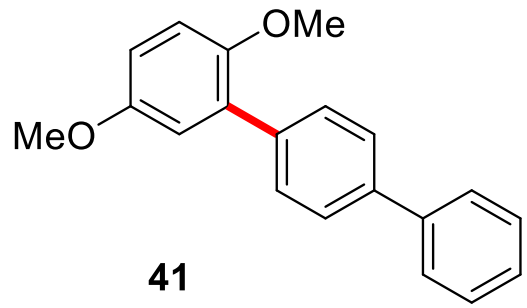
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol, 10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol),), 1-iodo-4-chlorobenzene(98.7 mg, 0.3 mmol,1.0 equiv.), dimethoxybenzene (289 mg, 2.1 mmol, 7 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.), After completion of the reaction, the 4'-Chloro-2,5-dimethoxy-1,1'-biphenyl **40** was isolated by column chromatography (hexane : EA = 100 : 5). Isolated yield X=I, 55% (40 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.47 (m, 2H), 7.37 (m, 2H), 6.89 (m, 3H), 3.81 (s,3H), 3.75 (s,3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 154.0, 150.8, 136.9, 133.2, 130.9, 128.3, 116.7, 114.8, 113.5, 112.9, 56.5, 56.0.

The spectral data are matched with those reported in the literature (34).

2, 5-Dimethoxy-1,1': 4',1''-terphenyl (41)



41

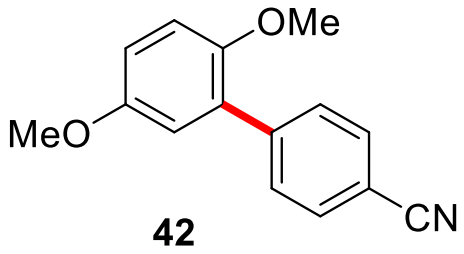
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol),), 4-iodo-1,1'-biphenyl (84 mg, 0.3 mmol, 1 equiv.), dimethoxybenzene (289 mg, 2.1 mmol, 7 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.), After completion of the reaction with 2.5h, the 2, 5-dimethoxy-1,1': 4',1''-terphenyl **41** was isolated by column chromatography (hexane : EA = 100 : 5). Isolated yield 79% (68 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.70 – 7.59 (m, 6 H), 7.46 (t, *J* = 7.7 Hz, 2H), 7.36 (t, *J* = 7.4 Hz, 1H), 6.96 (m, 2H), 6.90 – 6.84 (m, 1H), 3.83 (s, 3H), 3.80 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 154.0, 151.0, 141.1, 140.1, 137.5, 131.4, 130.0, 128.9, 127.4, 127.3, 127.0, 116.8, 113.4, 112.8, 56.5, 56.0.

HRMS: Calculated mass for [$C_{16}H_{22}N_2O$ + Na^+] is 313.1199 and the observed mass 313.1182.

2', 5'-Dimethoxy-[1, 1'-biphenyl]-4-carbonitrile (42)



42

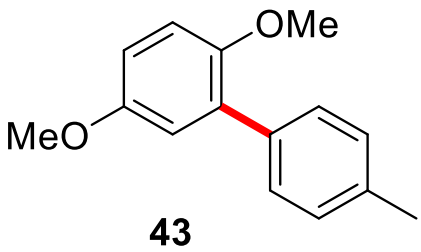
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol) 4-iodobenzonitrile (68 mg, 0.3 mmol, 1 equiv.), dimethoxybenzene (289 mg, 2.1 mmol, 7 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.), After completion of the reaction, the 2', 5'-dimethoxy-[1,1'-biphenyl]-4-carbonitrile **42** was isolated by column chromatography (hexane : EA = 100 : 10). Isolated yield X= I, 60% (43 mg, light yellow solid) & X= Cl, 47% (33 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.68 (d, *J* = 8.2 Hz, 2H), 7.63 (d, *J* = 8.2 Hz, 2H), 6.93 (m, 2H), 6.87 (d, *J* = 2.6 Hz, 1H), 3.81 (s, 3H), 3.76 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 150.7, 148.0, 147.1, 131.9, 130.4, 130.3, 119.2, 116.7, 114.5, 112.9, 110.8, 56.4, 56.0.

The spectral data are matched with those reported in the literature (35).

2, 5-Dimethoxy-4'-methyl-1,1'-biphenyl (43)



43

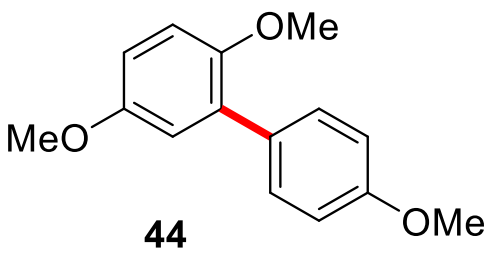
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol),), 1-iodo-3,5-dimethylbenzene (43 µL, 0.03 mmol, 1 equiv.), dimethoxybenzene (289 mg, 2.1 mmol, 7 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.), After completion of the reaction, the 2, 5-dimethoxy-4'-methyl-1,1'-biphenyl **43** was isolated by column chromatography (hexane). Isolated yield 69% (47 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.43 (d, *J* = 7.9 Hz, 2H), 7.23 (d, *J* = 7.8 Hz, 2H), 6.90 (m, 2H), 6.84 (dd, J_1= 3.1 Hz, J_2 = 8.8 Hz, 1H), 3.80 (s, 3H), 3.75 (s, 3H), 2.39 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 153.9, 151.0, 136.9, 135.6, 131.9, 129.4, 128.9, 116.8, 113.0, 112.8, 56.5, 55.9, 21.3.

The spectral data are matched with those reported in the literature (36).

2, 4',5-Trimethoxy-1,1'-biphenyl (44)



44

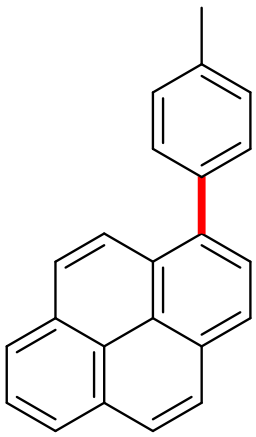
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol),), 1-iodo-3,5-dimethylbenzene (43 μL, 0.03 mmol, 1 equiv.), dimethoxybenzene (289 mg, 2.1 mmol, 7 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.), After completion of the reaction with 4h, the desired 2, 4',5-trimethoxy-1,1'-biphenyl **44** was isolated by column chromatography (hexane). Isolated yield 78% (57 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.49 (d, *J* = 8.6 Hz, 2H), 6.96 (d, *J* = 8.6 Hz, 2H), 6.92-6.90 (m, 2H), 6.83 (dd, J_1 = 3.0 Hz J_2 = 8.8 Hz, 1H), 3.85 (s, 3H), 3.81 (s, 3H), 3.76 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 158.9, 153.9, 150.9, 131.5, 130.9, 130.6, 116.7, 113.7, 112.8, 112.7, 56.5, 55.9, 55.4.

The spectral data are matched with those reported in the literature (37).

1-(*p*-Tolyl) pyrene (45)



45

The reaction was performed according to general procedure with PLY I (13 mg, 0.04 mmol,10 mol %), OED salt (44 mg, 0.08 mmol, 20 mol %),), 4-iodotoluene (87 mg, 0.4 mmol, 1.0 equiv.), Pyrene (404 mg, 2.0 mmol, 5.0 equiv.) and KO^{t}Bu (153 mg, 1.36 mmol, 3.4 equiv.), After completion of the reaction with 6.5h, 1-(*p*-tolyl) pyrene **45** was isolated by column chromatography (hexane). Isolated yield 68% (79 mg, yellow solid).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.31 – 8.14 (m, 4H), 8.10 (s, 2H), 8.07 – 7.93 (m, 3H), 7.61-7.55 (m, 2H), 7.40 (d, *J* = 8.0 Hz, 2H), 2.53 (s, 3H).

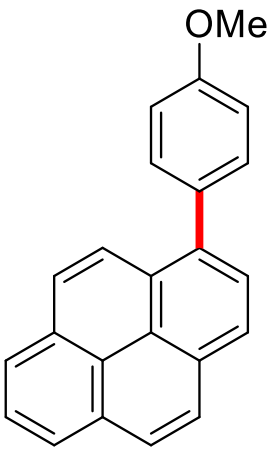
^{13}C NMR (100 MHz, $CDCl_3$) δ: 138.4, 137.9, 137.1, 131.7, 131.2, 130.6, 130.1, 129.2, 128.7, 127.8, 127.6, 127.5, 126.1, 125.8, 125.5, 125.2, 124.9, 124.8, 124.1, 21.4.

The spectral data are matched with those reported in the literature (38).

1-(4-Methoxyphenyl)pyrene (46)

The reaction was performed according to general procedure with PLY I (13 mg, 0.04 mmol, 10 mol %), OED salt (44 mg, 0.06 mmol, 20 mol %), 4-iodoanisole (93 mg, 0.3 mmol, 1.0 equiv.), Pyrene (404 mg, 2.0 mmol, 5.0 equiv.) and KO^{t}Bu (153 mg, 1.36 mmol, 3.4 equiv.), After completion of the reaction, 1-(4-methoxyphenyl)pyrene **46** was isolated by column chromatography (hexane : EA = 100 : 2). Isolated yield 56% (68 mg, yellow solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.27 – 8.13 (m, 4H), 8.09 (s, 2H), 8.00 (m, 3H), 7.57 (d, *J* = 7.2 Hz, 2H), 7.11 (d, *J* = 7.6 Hz, 2H), 3.94 (s, 3H).

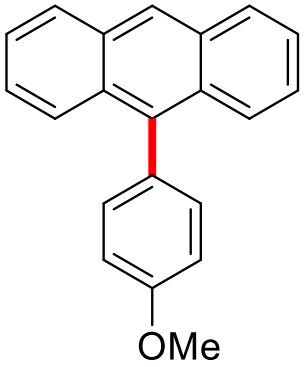


46

^{13}C NMR (125 MHz, $CDCl_3$) δ: 159.2, 137.6, 133.7, 131.8, 131.7, 131.3, 131.2, 130.5, 128.7, 127.8, 127.6, 127.5, 127.4, 126.2, 126.1, 125.5, 125.1, 124.9, 124.8, 114.0, 55.6.

The spectral data are matched with those reported in the literature (39).

9-(4-Methoxyphenyl)anthracene (47)



47

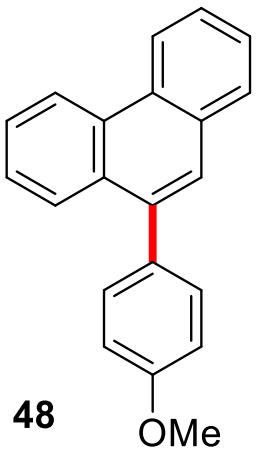
The reaction was performed according to general procedure with PLY I (13 mg, 0.04 mmol, 10 mol %), OED salt (44 mg, 0.08 mmol, 20 mol %),), 4-iodoanisole (93 mg, 0.3 mmol, 1.0 equiv.), anthracene (356 mg, 1.5 mmol, 5.0 equiv.) and KO^{t}Bu (153 mg, 1.36 mmol, 3.4 equiv.), After completion of the reaction, the 9-(4-methoxyphenyl)anthracene **47** was isolated by column chromatography (hexane). Isolated yield 51% (57 mg, white solid).

^{1}H NMR (400 MHz, $CHCl_3$) δ: 8.48 (s, 1H), 8.04 (d, *J* = 8.5 Hz, 2H), 7.76 – 7.66 (m, 2H), 7.49 – 7.41 (m, 2H), 7.36-7.34 (m, 4H), 7.13-7.11 (m, 2H), 3.95 (s, 3H).

^{13}C NMR: (100 MHz, $CHCl_3$) δ: 159.2, 137.4, 132.4, 131.5, 130.7, 128.5, 127.9, 127.1, 126.5, 125.4, 125.2, 114.0, 55.5.

The spectral data are matched with those reported in the literature (40).

9-(4-Methoxyphenyl)phenanthrene (48)



48

The reaction was performed according to general procedure with PLY I (13 mg, 0.04 mmol,10 mol %), OED salt (44 mg, 0.06 mmol, 20 mol %),), 4-iodoanisole (93 mg, 0.3 mmol, 1.0 equiv.), phenanthrene (356 mg, 2.0 mmol, 5.0 equiv.) and KO^{t}Bu (153 mg, 1.02 mmol, 3.4 equiv.), After completion of the reaction with 3.5 h, the 9-(4-methoxyphenyl)phenanthrene **48** was isolated by column chromatography (hexane : EA = 100 : 2). Isolated yield 47% (53 mg, solid).

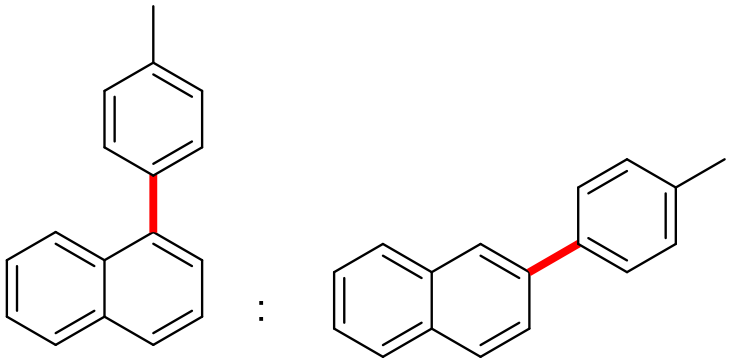
^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.78 (d, *J* = 8.3 Hz, 1H), 8.72 (d, *J* = 8.1 Hz, 1H), 7.95 (d, *J* = 8.4 Hz, 1H), 7.89 (d, *J* = 7.5 Hz, 1H), 7.67-7.63 (m, 3H),

7.61 (t, *J* = 7.3 Hz, 1H), 7.54 (t, *J* = 7.5 Hz, 1H), 7.48 (d, *J* = 8.0 Hz, 2H), 7.06 (d, *J* = 7.9 Hz, 2H), 3.92 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 159.2, 138.6, 133.3, 131.8, 131.6, 131.3, 130.8, 130.0, 128.7, 127.6, 127.4, 127.1, 126.9, 126.6, 126.5, 123.0, 122.7, 113.9, 55.5.

The spectral data are matched with those reported in the literature (41).

1-(*p*-Tolyl) naphthalene and 2-(*p*-Tolyl)naphthalene (49 & 49')



49 & 49'

The reaction was performed according to general procedure with PLY I (44 mg, 0.04 mmol,10 mol %), OED salt (44 mg, 0.08 mmol, 20 mol),), 4-iodotoluene (87.2 mg, 0.4 mmol, 1.0 equiv.), naphthalene (358 mg, 2.1 mmol, 7 equiv.) and KO^{t}Bu (153 mg, 1.36 mmol, 3.4 equiv.), After completion of the reaction, the 1-(*p*-tolyl) naphthalene and 2-(*p*-tolyl)naphthalene **49 & 49'** was isolated together by column chromatography (hexane). Isolated yield 61% (53 mg, colourless liquid, α:β = 1:0.58).The yield was isolated with 5-10% error due to some little amount of grease.

^{1}H NMR (500 MHz, $CDCl_3$) δ: Major isomer 7.93-7.84 (m, 3H), 7.53-7.47 (m, 2H), 7.44-7.39 (m, 4H), 7.32-7.30 (m, 2H)

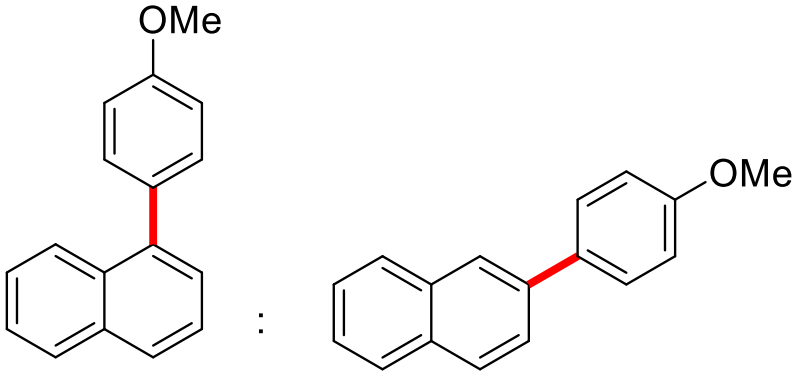
Minor isomers characterization peak 8.03 (s), 7.75-7.74 (m), 7.64-7.63 (m).

^{13}C NMR (126 MHz, $CDCl_3$) δ 140.4, 138.0, 137.1, 135.5, 134.0, 131.9, 130.1, 129.7, 129.1, 128.5, 128.4, 128.3, 128.3, 127.8, 127.6, 127.4, 127.0, 126.5, 126.4, 126.2, 126.2, 126.1, 126.0, 125.9, 125.8, 125.7, 125.6, 125.5, 21.4, 21.3. Indistinguishable peaks for both the isomers.

The spectral data are matched with those reported in the literature (42).

1-(4-Methoxyphenyl) naphthalene and 2-(4-Methoxyphenyl) naphthalene (50 & 50')

The reaction was performed according to general procedure with PLY I (13 mg, 0.04 mmol,10 mol %), OED salt (44 mg, 0.08 mmol, 20 mol),), 4-iodoanisole (93 mg, 0.4 mmol, 1.0 equiv.), naphthalene (358 mg, 2.1 mmol, 7 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.), After completion of the reaction, the 1-(4-methoxyphenyl) naphthalene and 2-(4-methoxyphenyl) naphthalene **50 & 50'** was isolated together by column chromatography (hexane : EA = 100 : 2). Isolated yield 51% (47 mg, white solid, α:β = 1:0.62).



50 & 50'

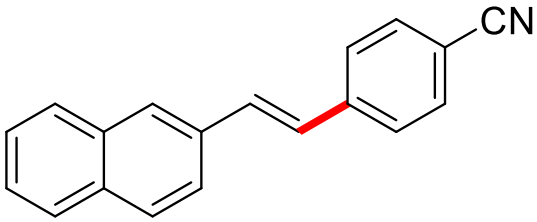
^{1}H NMR (400 MHz, $CDCl_3$) δ: **Major isomer** 7.96 – 7.82 (m, 3H), 7.55 – 7.38 (m, 6H), 7.08 – 7.00 (m, 2H), 3.90 (s, 3H). **Minor isomer** detected peak 8.00 (s), 7.74-7.72 (m), 7.67 (d, *J* = 8.6 Hz).

^{13}C NMR (100 MHz, $CDCl_3$) δ: Major Isomer 159.1, 137.5, 133.6, 131.7, 131.6, 131.3, 127.8, 127.5, 127.4, 127.3, 126.2, 126.0, 125.2, 113.9, 55.5.

Distinguishable peaks of minor isomer 159.1, 130.4, 125.7, 125.5, 124.8, 124.7, 123.9, 113.9

The spectral data are matched with those reported in the literature (43).

(*E*)-4-(2-(Naphthalen-2-yl)vinyl)benzonitrile (51)



51

The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 4-iodobenzonitrile (68 mg, 0.3 mmol, 1 equiv.), 2-vinylnaphthalene (231 mg, 2.0 mmol, 5 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction (*E*)-4-(2-(naphthalen-2-yl) vinyl)benzonitrile **51** was isolated by column chromatography (hexane : EA = 100 :2). Isolated yield 58% (44 mg, white solid).

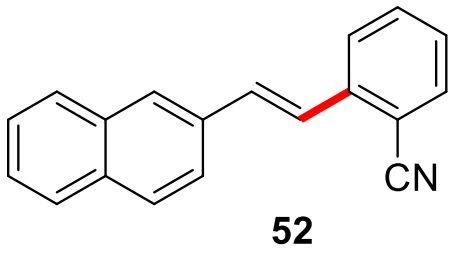
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.90-7.83 (m, 1H), 7.74 (d, *J* = 8.4 Hz, 1H), 7.67-7.62 (m, 1H), 7.52-7.47 (s, 1H), 7.40-7.37 (d, *J* = 16.3 Hz, 1H), 7.23-7.20 (d, *J* = 16.3 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 142.0, 133.9, 133.7, 133.6, 132.7, 132.7, 128.7, 128.3, 127.9, 127.8, 127.2, 127.0, 126.8, 126.7, 123.4, 119.2, 110.8.

The spectral data are matched with those reported in the literature (44).

(*E*)-2-(2-(Naphthalen-2-yl)vinyl)benzonitrile (52)

The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 2-iodobenzonitrile (68 mg, 0.4 mmol, 1 equiv.), 2-vinylnaphthalene (231 mg, 2.0 mmol, 5 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4



52

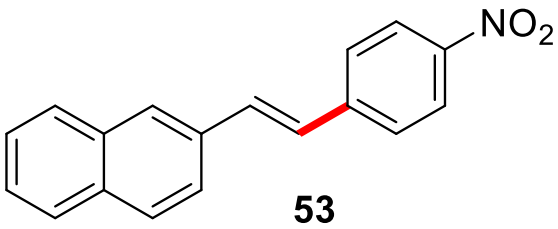
equiv.). After completion of the reaction with 6h (*E*)-2-(2-(naphthalen-2-yl)vinyl)benzonitrile **52** was isolated by column chromatography (hexane : EA = 100 : 1). Isolated yield 70% (53 mg, yellow solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.93 (s, 1H), 7.84 (m, 5H), 7.68 (d, *J* = 7.7 Hz, 1H), 7.62 - 7.57 (m, 2H), 7.50 (m, 2H), 7.47 – 7.41 (m, 1H), 7.35 (t, *J* = 7.5 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 140.7, 133.8, 133.7, 133.7, 133.6, 133.3, 132.9, 128.7, 128.4, 128.0, 127.9, 127.7, 126.7, 126.7, 125.4, 124.4, 123.7, 118.2, 111.4.

The spectral data are matched with those reported in the literature (45).

(*E*)-2-(4-Nitrostyryl)naphthalene (53)



53

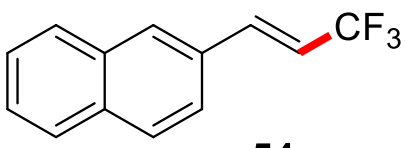
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 1-iodo-4-nitrobenzene (74 mg, 0.3 mmol, 1 equiv.), 2-vinylnaphthalene (231 mg, 1.5 mmol, 5 equiv.) and KO^{t}Bu (115 mg, 1.36 mmol, 3.4 equiv.). After completion of the reaction (*E*)-2-(4-nitrostyryl) naphthalene **53** was isolated by column chromatography (hexane : EA = 100 :1). Isolated yield 64 % (52 mg, yellow solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.24 (d, *J* = 8.5 Hz, 2H), 7.92 (s, 1H), 7.85 (t, *J* = 10.0 Hz, 3H), 7.75 (d, *J* = 8.6 Hz, 1H), 7.68 (d, *J* = 8.6 Hz, 2H), 7.56 – 7.46 (m, 2H), 7.43 (d, *J* = 16.3 Hz, 1H), 7.25 (d, *J* = 16.3 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 146.9, 144.0, 133.8, 133.7, 133.5, 128.8, 128.4, 128.1, 1279, 127.0, 126.8, 126.8, 126.7(2C overlapped), 124.3, 123.4.

The spectral data are matched with those reported in the literature (46).

(*E*)-2-(3, 3,3-Trifluoroprop-1-en-1-yl)naphthalene (54)



54

The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 5-(trifluoromethyl)-5H-dibenzo[b,d]thiophen-5-ium tetrafluoroborate (Umemoto's reagent) (102 mg, 0.3 mmol, 1 equiv.), 2-vinylnaphthalene (231 mg, 1.5 mmol, 5 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion

of the reaction (*E*)-2-(3,3,3-trifluoroprop-1-en-1-yl)naphthalene **54** was isolated by column chromatography (hexane). Isolated yield 61% (40 mg, white solid).

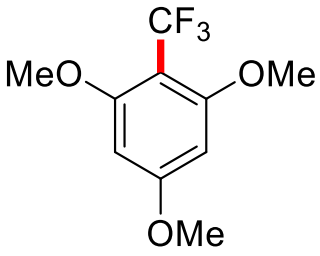
^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.90 – 7.77 (m, 4H), 7.67 – 7.56 (m, 1H), 7.50 (dd, *J* = 6.2, 3.2 Hz, 2H), 7.07-7.02 (m, 1H), 6.49 – 6.32 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 137.3 (m) 134.0, 133.5, 132.1, 128.8, 128.4, 1279, 127.0, 126.8, 123.5, 121.4 (m), 117.4(m), 115.6.

^{19}F NMR (475 MHz, CDCl3) δ: –63.14 .

The spectral data are matched with those reported in the literature (47).

1, 3,5-Trimethoxy-2-(trifluoromethyl)benzene (55)



55

The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 5-(trifluoromethyl)-5*H*-dibenzo[b,d]thiophen-5-ium tetrafluoroborate (Umemoto's reagent) (102 mg, 0.3 mmol, 1 equiv.), trimethoxybenzene (252 mg, 1.5 mmol, 5 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction 1, 3,5-trimethoxy-2-(trifluoromethyl)benzene **55** was isolated by column chromatography (hexane). Isolated yield 52% (36 mg, liquid).

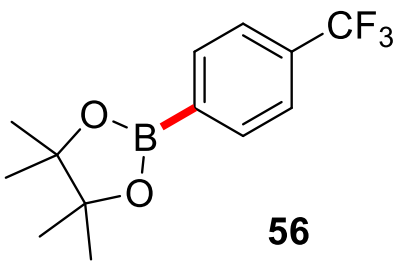
^{1}H NMR (500 MHz, $CDCl_3$) δ: 6.13 (s, 2H), 3.84 (s, 9H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 163.7, 160.6, 124.5 (d, *J* = 273.4 Hz), 100.8 (m), 91.5, 56.4, 55.5.

^{19}F NMR (471 MHz, $CDCl_3$) δ: -54.17.

The spectral data are matched with those reported in the literature (48).

4,4,5,5-Tetramethyl-2-(4-(trifluoromethyl)phenyl)-1,3,2-dioxaborolane (56)



56

The reaction was performed according to general procedure with PLY I (13 mg, 0.04 mmol,10 mol %), OED salt (44 mg, 0.08 mmol, 20 mol %), 1-iodo-4-trifluoromethylbenzene (44 µL, 0.4 mmol, 1 equiv.), B_2Pin_2 (253 mg, 1.0 mmol, 2.5 equiv.) and KO^{t}Bu (153 mg, 1.36 mmol, 3.4 equiv.). After completion of the reaction 4,4,5,5-tetramethyl-2-(4-(trifluoromethyl)phenyl)-1,3,2-dioxaborolane **56** was

isolated by column chromatography (hexane : EA = 100 : 1). Isolated yield 65 % (70 mg, yellow solid).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.91 (d, *J* = 7.9 Hz, 2H), 7.61 (d, *J* = 8.1 Hz, 2H), 1.36 (s, 12H).

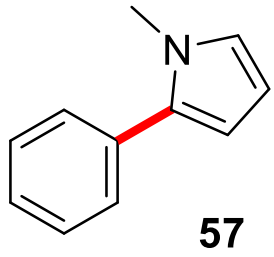
^{13}C NMR (100 MHz, $CDCl_3$) δ: 135.1, 133.3, 132.9, 132.7, 132.4, 125.5, 124.5, 122.8, 84.4, 25.0.

^{19}F NMR (376 MHz, CDCl3) δ: -62.93.

^{11}B NMR (128 MHz, CDCl3) δ: 29.99.

The spectral data are matched with those reported in the literature (49).

1-Methyl-2-phenyl-1*H*-pyrrole (57)



57

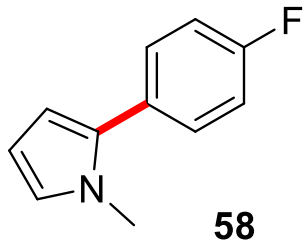
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), iodobenzene (34 μL, 0.3 mmol,1.0 equiv.), *N*-methylpyrole (267 μL, 3 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 1-methyl-2-phenyl-1*H*-pyrrole **57** was isolated by column chromatography (hexane). Isolated yield 49% (23 mg, solid).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.44 – 7.37 (m, 4H), 7.35 – 7.26 (m, 1H), 6.78 – 6.68 (m, 1H), 6.24-7.22 (m, 1H), 6.22 – 6.19 (m, 1H), 3.67 (s, 3H).

^{13}C NMR (100 MHz, $CHCl_3$) δ: 134.7, 133.4, 128.7, 128.4, 126.8, 123.7, 108.7, 107.8, 35.1.

The spectral data are matched with those reported in the literature (50).

2-(4-Fluorophenyl)-1-methyl-1*H*-pyrrole (58)



58

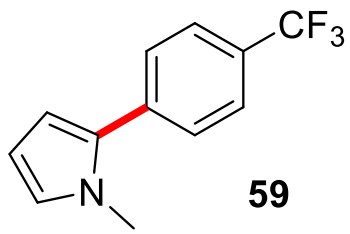
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 4-iodo-1-fluorobenzene (34.6 μL, 0.3 mmol,1.0 equiv.),), *N*-methylpyrole (267 μL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 2-(4-fluorophenyl)-1-methyl-1H-pyrrole **58** was isolated by column chromatography (hexane:EA = 100 : 1). Isolated yield 51% (26 mg, light yellow solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.35 (dd, J_1 = 8.8 Hz, J_2 = 5.4 Hz, 2H), 7.09 (t, *J* = 8.7 Hz, 2H), 6.71 (t, *J* = 2.2 Hz, 1H), 6.19 (t, *J* = 2.0 Hz, 2H), 3.63 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 161.9 (d, *J* = 246.3 Hz), 132.6, 130.3 (d, *J* = 8.1 Hz), 129.5 (d, *J* = 3.4 Hz), 123.7, 115.3 (d, *J* = 21.4 Hz), 108.8, 107.9, 31.7.

The spectral data are matched with those reported in the literature (51).

1-Methyl-2-(4-(trifluoromethyl)phenyl)-1*H*-pyrrole (59)



59

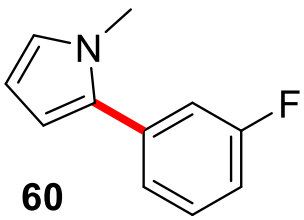
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 4-iodo-1-trifluoromethylbenzene (44 µL, 0.3 mmol, 1.0 equiv.),), *N*-methylpyrole (267 µL, 3 mmol, 10 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction with 3h, the desired product 1-methyl-2-(4-(trifluoromethyl) phenyl)-1*H*-pyrrole **59** was isolated by column chromatography (hexane : EA = 100 : 2). Isolated yield 67% (45 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.65 (d, *J* = 8.2 Hz, 2H), 7.51 (d, *J* = 8.1 Hz, 2H), 6.82 – 6.73 (m, 1H), 6.31 (dd, *J* = 3.6, Hz, *J* = 1.8 Hz, 1H), 6.23 (dd, *J* = 3.5 Hz, *J* = 2.8 Hz, 1H), 3.70 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 137.0, 133.3, 128.8 (*J* = 65.2 Hz), 128.5, 125.5 (q, *J* = 273.1 Hz), 125.1, 123.4 (q, *J*= 273.1 Hz), 110.1, 108.4, 35.4.

The spectral data are matched with those reported in the literature (52).

2-(3-Fluorophenyl)-1-methyl-1*H*-pyrrole (60)



60

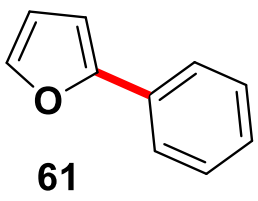
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 3-iodo-1-fluorobenzene (35 µL, 0.3 mmol, 1.0 equiv.), *N*-methylpyrole (267.0 µL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 2-(3-fluorophenyl)-1-methyl-1*H*-pyrrole **60** was isolated by column chromatography (hexane: EA = 100: 2). Isolated yield 52% (27 mg, yellow liquid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.42 – 7.30 (m, 1H), 7.23 – 7.15 (m, 1H), 7.12-7.09 (m, 1H), 7.01-6.99 (m, 1H), 6.76 – 6.69 (m, 1H), 6.26-6.25 (m, 1H), 6.23 – 6.18 (m, 1H), 3.68 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 162.7 (d, *J* = 245.4 Hz), 135.4 (d, *J* = 8.4 Hz), 133.5, 129.8 (d, *J* = 8.7 Hz), 124.4, 124.3, 115.2 (d, *J* = 21.9 Hz), 113.5 (d, *J* = 21.2 Hz), 109.4, 108.1, 35.3.

The spectral data are matched with those reported in the literature (53).

2-Phenylfuran (61)



61

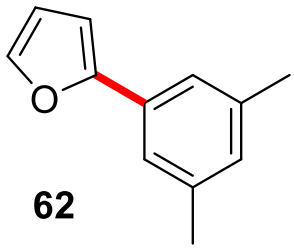
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), iodobenzene (34 µL, 0.3 mmol, 1.0 equiv.),), furan (218 µL, 3.0 mmol, 10.0 equiv.). and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 2-phenylfuran **61** was isolated by column chromatography (hexane: EA = 100: 2). Isolated yield 48% (20 mg, colourless liquid).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.70 (d, *J* = 7.9 Hz, 2H), 7.49 (s, 1H), 7.40 (t, *J* = 7.7 Hz, 2H), 7.27 (m, 1H), 6.67 (d, *J* = 3.3 Hz, 1H), 6.49 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.1, 142.2, 131.0, 128.8, 127.5, 123.9, 111.8, 105.1.

The spectral data are matched with those reported in the literature (54).

2-(3,5-Dimethylphenyl)furan (62)



62

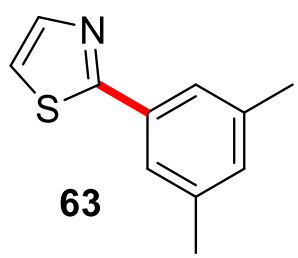
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 1-iodo-3,5-dimethylbenzene (43.5 µL, 0.3 mmol, 1.0 equiv.),), furan (218 µL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 2-(3,5-dimethylphenyl)furan **62** was isolated by column chromatography (hexane : EA = 100 : 2). Isolated yield 61% (31 mg, colourless liquid).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.43 (d, *J* = 1.4 Hz, 1H), 7.29 (s, 2H), 6.89 (s, 1H), 6.60 (d, *J* = 3.3 Hz, 1H), 6.51 – 6.39 (m, 1H), 2.33 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.4, 141.9, 138.3, 130.9, 129.2, 121.8, 111.7, 104.8, 21.5.

The spectral data are matched with those reported in the literature (55).

2-(3,5-Dimethylphenyl)thiazole (63)



63

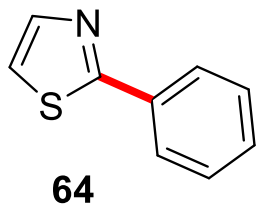
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 1-iodo-3,5-dimethylbenzene (43.5 mL, 0.3 mmol,1.0 equiv.),), thiazole (213 µL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 2-(3,5-dimethylphenyl)thiazole **63** was isolated by column chromatography (hexane : EA = 100 : 5). Isolated yield 50% (28 mg, colourless oil).

^{1}H NMR (400 MHz, CDCl3) δ: 7.84 (d, *J* = 3.2 Hz, 1H), 7.59 (s, 2H), 7.30 (d, *J* = 3.3 Hz, 1H), 7.07 (s, 1H), 2.38 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 169.0, 143.7, 138.8, 133.5, 131.9, 124.6, 118.7, 21.4.

The spectral data are matched with those reported in the literature (56).

2-Phenylthiazole (64)



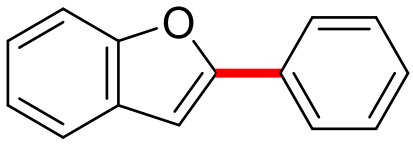
64

The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol, 10 mol%), OED salt (32.4 mg, 0.06 mmol, 20 mol %), iodobenzene (34 µL, 0.3 mmol,1.0 equiv.),), thiazole (213 µL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 2-phenylthiazole **64** was isolated by column chromatography (hexane: EA = 100: 5). Isolated yield 60% (28 mg, colourless liquid).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.98-7.96 (m, 2H), 7.87 (d, *J* = 3.4 Hz, 1H), 7.45-7.44 (m, 3H), 7.35 – 7.32 (m, 1H).

^{13}C NMR (100 MHz, $CHCl_3$) δ: 168.5, 143.8, 133.7, 130.1, 129.0, 126.6, 118.9.
The spectral data are matched with those reported in the literature (56).

2-Phenylbenzofuran (65)



65

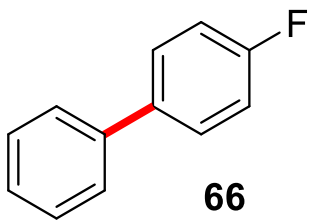
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol, 10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol%), iodobenzene (34 µL, 0.3 mmol,1.0 equiv.), benzofuran (330 µL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 2-Phenylbenzofuran **65** was isolated by column chromatography (hexane: EA = 100: 10). Isolated yield 52% (30 mg, solid).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.88 (d, *J* = 7.9 Hz, 2H), 7.59 (d, *J* = 7.5 Hz, 1H), 7.53 (d, *J* = 8.2 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.38-7.34 (m, 1H), 7.33 – 7.27 (m, 1H), 7.27 – 7.21 (m, 1H), 7.04 (s, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ: 156.1, 155.0, 130.6, 129.4, 128.9, 128.7, 125.1, 124.4, 123.1, 121.0, 111.3, 101.4.

The spectral data are matched with those reported in the literature (57).

4-Fluoro-1,1'-biphenyl (66)



66

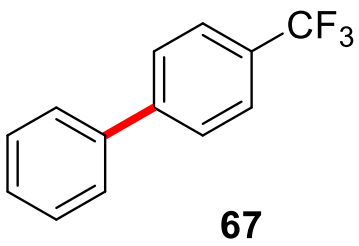
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 1-iodo-4-fluorobenzene (35 μL, 0.3 mmol, 1.0 equiv.),), benzene (268 μL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 4-Fluoro-1,1'-biphenyl **66** was isolated by column chromatography (hexane). Isolated yield 48% (24 mg, colourless liquid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.61 – 7.51 (m, 4H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.35 (t, *J* = 7.3 Hz, 1H), 7.13 (t, *J* = 8.5 Hz, 2H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 162.6 (d, *J* = 246.4 Hz), 140.4, 137.5 (d, *J* = 2.6 Hz), 129.0, 128.8 (d, *J* = 8.0 Hz), 127.4, 127.2, 115.8 (d, *J* = 21.4 Hz).

The spectral data are matched with those reported in the literature (58).

4-(Trifluoromethyl)-1,1'-biphenyl (67)



67

The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 1-iodo-4-trifluoromethylbenzene (44 μL, 0.3 mmol, 1.0 equiv.), benzene (268 μL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction with 4h, the desired product 4-(trifluoromethyl)-1,1'-biphenyl **67** was isolated by column chromatography (hexane). Isolated yield 64% (42 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.70 (s, 4H), 7.63 – 7.57 (m, 2H), 7.52 – 7.45 (m, 2H), 7.44 – 7.38 (m, 1H).

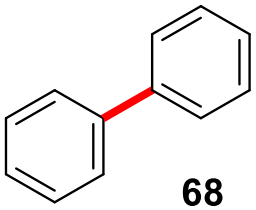
^{13}C NMR (125 MHz, $CDCl_3$) δ: 144.8, 140.0, 129.5, 129.1, 128.3, 127.6, 127.4, 125.9 (t, *J* = 3.7 Hz), 123.0.

^{19}F NMR (470 MHz, $CDCl_3$) δ: -60.40.

The spectral data are matched with those reported in the literature (59).

1,1'-Biphenyl (68)

The reaction was performed according to general procedure with (*N*,*O*)-PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), iodobenzene (34 μL, 0.3 mmol, 1.0 equiv.),), benzene (268 μL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 1,1'-Biphenyl **68** was isolated by column chromatography (hexane). Isolated yield 78% (36 mg, white solid).



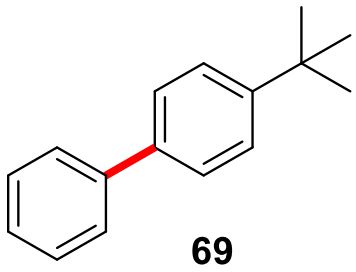
68

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.67 – 7.59 (m, 4H), 7.49-7.45 (m, 4H), 7.39-7.36 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 141.4, 128.9, 127.4, 127.3.

The spectral data are matched with those reported in the literature (60).

4-(*tert*-Butyl)-1,1'-biphenyl (69)



69

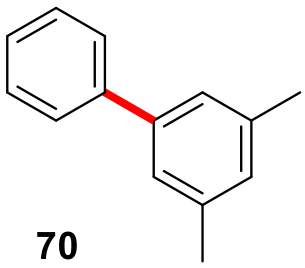
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol%), OED salt (32.4 mg, 0.06 mmol, 20 mol%), 4-tertbutyl-1-iodobenzene (53 µL, 0.3 mmol,1.0 equiv.),), benzene (268 µL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 4-(*tert*-butyl)-1,1'-biphenyl **69** was isolated by column chromatography (hexane). Isolated yield 66% (41 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.61-7.53 (m, 4H), 7.50 – 7.40 (m, 4H), 7.35 – 7.31 (m, 1H), 1.37 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 150.4, 141.2, 138.5, 137.2, 128.8, 127.2, 126.9, 125.9, 34.7, 31.5.

The spectral data are matched with those reported in the literature (61).

3,5-Dimethyl-1,1'-biphenyl (70)



70

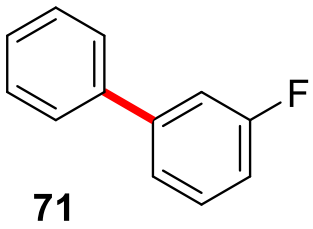
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 3,5-dimthyl-1-iodobenzene (43.5 µL, 0.3 mmol,1.0 equiv.), benzene (268 µL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction with 4.5h, the desired product 3,5-Dimethyl-1,1'-biphenyl **70** was isolated by column chromatography (hexane). Isolated yield 49% (26 mg, colourless solid).

^{1}H NMR (400 MHz, $CHCl_3$) δ: 7.64 – 7.55 (m, 2H), 7.45-7.43 (m, 2H), 7.40 – 7.30 (m, 1H), 7.23 (s, 2H), 7.01 (s, 1H), 2.40 (s, 6H).

^{13}C NMR (100 MHz, $CHCl_3$) δ: 141.6, 141.4, 138.3, 129.0, 128.8, 127.3, 127.2, 125.2, 21.5.

The spectral data are matched with those reported in the literature (62).

3-Fluoro-1,1'-biphenyl (71)



71

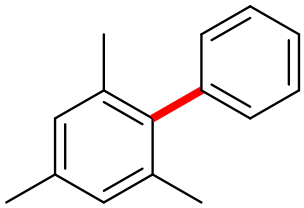
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 3-iodo-1-fluorobenzene (35 µL, 0.3 mmol,1.0 equiv.), benzene (268 µL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 3-fluoro-1,1'-biphenyl **71** was isolated by column chromatography (hexane). Isolated yield 57% (29 mg, colourless solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.58 (d, *J* = 7.5 Hz, 2H), 7.50-7.44 (m, 2H), 7.43 – 7.34 (m, 3H), 7.29 (d, *J* = 10.2 Hz, 1H), 7.14 – 6.99 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 163.3 (d, *J* = 245.5 Hz), 143.7 (d, *J* = 8.1 Hz), 140.1 (d, *J* = 1.9 Hz), 130.3 (d, *J* = 8.4 Hz), 129.0, 128.0, 127.2, 122.9 (d, *J* = 2.5 Hz), 114.3 (d, *J* = 21.2 Hz), 114.1 (d, *J* = 21.2 Hz).

The spectral data are matched with those reported in the literature (63).

2,4,6-trimethyl-1,1'-biphenyl (72)



72

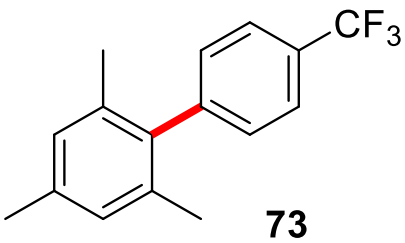
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), iodobenzene (34 µL, 0.3 mmol, 1.0 equiv.), mesitylene (416 µL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 2, 4, 6-trimethyl-1, 1'-biphenyl **72** was isolated by column chromatography (hexane). Isolated yield 52% (30 mg, solid).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.45-7.39 (m, 2H), 7.36 – 7.29 (m, 1H), 7.14 (d, *J* = 8.0 Hz, 2H), 6.94 (s, 2H), 2.33 (s, 3H), 2.00 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 141.2, 139.2, 136.7, 136.1, 129.4, 128.5, 128.2, 126.6, 21.1, 20.9.

The spectral data are matched with those reported in the literature (64).

2,4,6-Trimethyl-4'-(trifluoromethyl)-1,1'-biphenyl (73)



73

The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol%), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 1-iodo-4-trifluoromethylbenzene (44 µL, 0.3 mmol, 1.0 equiv.), mesitylene (416 µL, 3.0 mmol, 10.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product 2,4,6-trimethyl-4'-

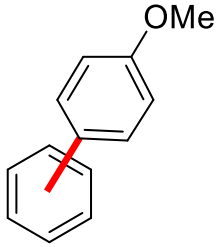
(trifluoromethyl)-1,1'-biphenyl **73** was isolated by column chromatography (hexane). Isolated yield 62% (49 mg, solid).

^{1}H NMR (400 MHz, CDCl3) δ: 7.68 (d, *J* = 8.1 Hz, 2H), 7.27 (d, *J* = 8.2 Hz, 2H), 6.96 (s, 2H), 2.34 (s, 3H), 1.98 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 145.2, 137.7, 137.4, 135.8, 129.9, 129.2, 128.4, 125.5 (d, *J* = 3.7 Hz), 124.9, 21.2, 20.8.

The spectral data are matched with those reported in the literature (65).

2-Methoxy-1,1'-biphenyl, 3-Methoxy-1,1'-biphenyl and 4-Methoxy-1,1'-biphenyl (74)



74

m:p:o = (1.00:0.38:0.24)

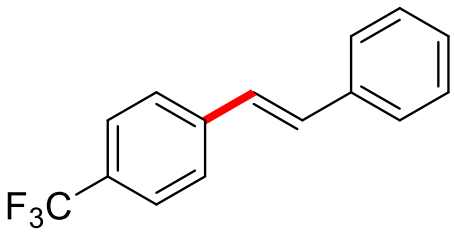
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), iodobenzene (34 μL, 0.3 mmol, 1.0 equiv.),), anisole (326 μL, 3.0 mmol, 10.0 equiv.). and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction, the desired product a mixture of (ortho, meta, para) of 4-methoxy-1,1'-biphenyl **74** was isolated by column chromatography (hexane : EA = 100 : 2). Isolated yield 52% (28 mg, colourless liquid). The percentage of different regioisomers was calculated by relative integration of the protons arising from the respective isomers. meta(m): para(p): ortho(o) = (1.00:.38:0.24)

^{1}H NMR: 7.61-6.89 (9H)

^{13}C NMR (125 MHz, $CDCl_3$) δ: 158.5, 157.9, 156.6, 141.0, 138.8, 138.7, 133.9, 131.0, 129.9, 129.7, 128.9, 128.7, 128.3, 128.1, 127.6, 127.3, 127.0, 126.9, 126.8, 121.0, 115.1, 114.3, 113.2, 113.1, 112.9, 112.8, 111.5, 111.4, , 55.7, 55.5, 55.5. Non distinguishable peaks.

The spectral data are matched with those reported in the literature (66)

(*E*)-1-Styryl-4-(trifluoromethyl)benzene (75)



75

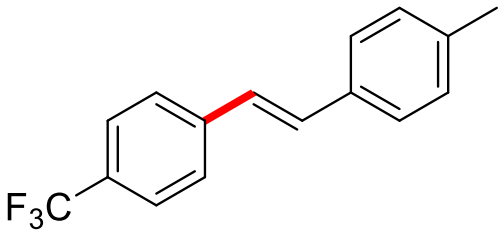
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 1-iodo-4-trifluoromethylbenzene (44 μL, 0.4 mmol, 1 equiv.), styrene (173 μL, 1.5 mmol, 5.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.), After completion of the reaction with 6h, (*E*)-1styryl-4-(trifluoromethyl)benzene **75** was isolated by column chromatography (hexane). Isolated yield 72% (53 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.61 (m, 4H), 7.54 (d, *J* = 7.6 Hz, 2H), 7.39 (t, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 7.3 Hz, 1H), 7.20 (d, *J* = 16.3 Hz, 1H), 7.12 (d, *J* = 16.3 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 140.9, 136.8, 131.3, 129.4 (d, *J* = 32.4 Hz), 128.9, 128.4, 127.3, 126.9, 126.7, 125.8 (q, *J* = 3.8 Hz), 124.4 (d, *J* = 271.8 Hz).

The spectral data are matched with those reported in the literature (67).

(*E*)-1-Methyl-4-(4-(trifluoromethyl)styryl)benzene (76)



76

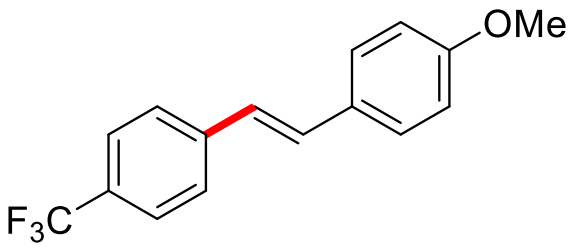
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 1-iodo-4-trifluoromethylbenzene (44 μL, 0.4 mmol, 1.0 equiv.), 4-methylstyrene (173 μL, 2.0 mmol, 5.0 equiv.) and KO^{t}Bu (115 mg, 1.36 mmol, 3.4 equiv.). After completion of the reaction, (*E*)-1-methyl-4-(4-(trifluoromethyl)styryl)benzene **76** was isolated by column chromatography (hexane : EA = 100 : 1). Isolated yield 47% (63 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.59 (s, 4H), 7.43 (d, *J* = 7.9 Hz, 2H), 7.22 – 7.13 (m, 3H), 7.07 (d, *J* = 16.3 Hz, 1H), 2.37 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 141.2, 138.5, 134.0, 131.3, 129.7, 129.2 (q, *J* = 32.2 Hz), 126.8, 126.6, 126.3, 125.7 (q, *J* =3.6 Hz), 121.0 (q, *J* = 594.0 Hz), 21.5.

The spectral data are matched with those reported in the literature (68).

(*E*)-1-Methoxy-4-(4-(trifluoromethyl)styryl)benzene (77)



77

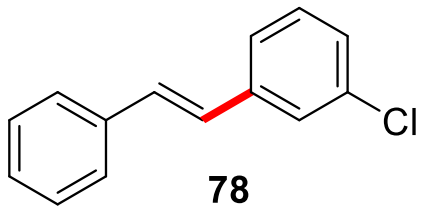
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol%), 1-iodo-4-trifluoromethylbenzene (44 μL, 0.3 mmol, 1.0 equiv.), 4-methoxystyrene (201 μL, 1.5 mmol, 5.0 equiv.) and KO^{t}Bu (115 mg, 1.36 mmol, 3.4 equiv.). After completion of the reaction with 5h (*E*)-1-methoxy-4-(4-(trifluoromethyl)styryl)benzene **77** was isolated by column chromatography (hexane : EA = 100 : 5). Isolated yield 66% (55 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.58 (m, 4H), 7.48 (d, *J* = 8.5 Hz, 2H), 7.15 (d, *J* = 16.3 Hz, 1H), 6.98 (d, *J* = 16.3 Hz, 1H), 6.92 (d, *J* = 8.5 Hz, 2H), 3.84 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 160.0, 141.3, 130.9, 129.6, 129.1, 128.8, 128.2, 126.4, 125.6 (q, *J* =3.8 Hz), 125.1, 114.4, 55.5.

The spectral data are matched with those reported in the literature (69).

(*E*)-1-Chloro-3-styrylbenzene (78)



78

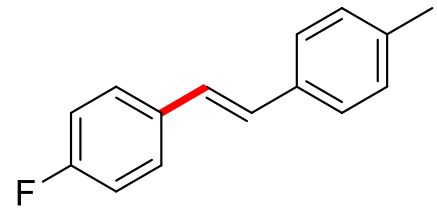
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol,10 mol%), OED salt (32.4 mg, 0.06 mmol, 20 mol%), 1-chloro-3-iodobenzene (37 μL, 0.3 mmol, 1.0 equiv.), styrene (173 μL, 1.5 mmol, 5 equiv.) and KO^{t}Bu (153 mg, 1.36 mmol, 3.4 equiv.). After completion of the reaction (*E*)-1-chloro-3-styrylbenzene **78** was isolated by column chromatography (hexane : EA = 100 : 1). Isolated yield 49% (31 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.59 – 7.48 (m, 3H), 7.39-7.36 (m, 3H), 7.30-7.27 (m, 2H), 7.25 – 7.18 (m, 1H), 7.12 (d, *J* = 16.3 Hz, 1H), 7.04 (d, *J* = 16.3 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 139.4, 137.0, 134.8, 130.3, 130.0, 128.9, 128.2, 127.6, 127.4, 126.8, 126.4, 124.9.

The spectral data are matched with those reported in the literature (70).

(*E*)-1-Fluoro-4-(4-methylstyryl) benzene (79)



79

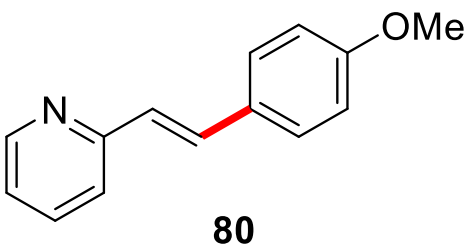
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol, 10 mol %), OED salt (32.4mg, 0.06 mmol, 20 mol %), 1-iodo-4-Fluorobenzene (35 μL, 0.3 mmol, 1 equiv.), 4-methyl styrene (198 μL, 2.0 mmol, 5 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction (*E*)-1-fluoro-4-(4-methylstyryl) benzene **79** was isolated by column chromatography (hexane). Isolated yield 63% (40 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 (dd, J_1 = 5.6 Hz, J_2 = 8.3 Hz, 2H), 7.42 (d, *J* = 7.9 Hz, 2H), 7.19 (d, *J* = 7.8 Hz, 2H), 7.06 (m, 2H), 7.03 (m, 2H), 2.39 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 162.4 (d, *J* = 246.8 Hz), 137.6, 134.4, 133.7 (d, *J* = 3.5 Hz), 129.4, 128.6 (d, *J* = 2.3 Hz), 127.8 (d, *J* = 8.0 Hz), 126.5, 126.4, 115.6 (d, *J* = 21.6 Hz), 21.2.

The spectral data are matched with those reported in the literature (71).

(*E*)-2-(4-Methoxystyryl) pyridine (80)



80

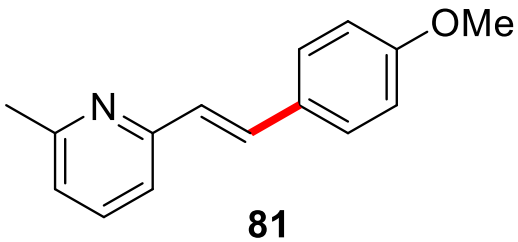
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol, 10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 2-Iodopyridine (32 µL, 0.3 mmol, 1 equiv.), 4-methoxystyrene (201 µL, 1.5 mmol, 5.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction (*E*)-2-(4-methoxystyryl)pyridine **80** was isolated by column chromatography (hexane : EA = 10:1). Isolated yield 47% (29 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.58 (d, *J* = 3.7 Hz, 1H), 7.65-7.64 (m, 1H), 7.58 (d, *J* = 16.1 Hz, 1H), 7.52 (d, *J* = 8.7 Hz, 2H), 7.35 (d, *J* = 7.8 Hz, 1H), 7.11 (dd, J_1= 6.8 Hz, J_2 = 4.9 Hz, 1H), 7.04 (d, *J* = 16.1 Hz, 1H), 6.91 (d, *J* = 8.7 Hz, 2H), 3.83 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 160.0, 156.1, 149.7, 136.6, 132.4, 129.6, 128.6, 126.0, 121.9, 121.8, 114.3, 55.5.

The spectral data are matched with those reported in the literature (72).

(*E*)-2-(4-Methoxystyryl)-6-methylpyridine (81)



81

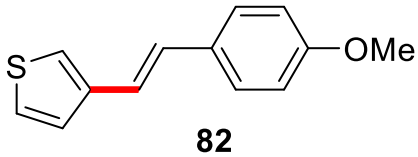
The reaction was performed according to general procedure with PLY I (9.7 mg, 0.04 mmol, 10 mol %), OED salt (32.4, 0.08 mmol, 20 mol %), 2-iodo-6-methylpyridine (36 µL, 0.3 mmol, 1 equiv.), 4-methoxystyrene (201 µL, 2.0 mmol, 5.0 equiv.) and KO^{t}Bu (115 mg, 1.36 mmol, 3.4 equiv.). After completion of the reaction (*E*)-2-(4-methoxystyryl)pyridine **81** was isolated by column chromatography (hexane : EA= 10 : 1). Isolated yield 69% (46 mg, white solid).*

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.58 – 7.46 (m, 4H), 7.20 (d, *J* = 7.7 Hz, 1H), 7.04 (d, *J* = 16.1 Hz, 1H), 6.98 (d, *J* = 7.6 Hz, 1H), 6.90 (d, *J* = 8.3 Hz, 2H), 3.83 (s, 3H), 2.58 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ: 159.9, 158.3, 155.6, 136.7, 132.1, 129.7, 128.5, 126.5, 121.4, 118.6, 114.3, 55.4, 24.8.

The spectral data are matched with those reported in the literature (73).

(*E*)-3-(4-Methoxystyryl)thiophene (82)



82

The reaction was performed according to general procedure with PLY I (9.7 mg, 0.03 mmol, 10 mol %), OED salt (32.4 mg, 0.06 mmol, 20 mol %), 3-iodothiophene (31 μL, 0.4 mmol, 1.0 equiv.), 4-methoxystyrene (201 μL, 2.0 mmol, 5.0 equiv.) and KO^{t}Bu (115 mg, 1.02 mmol, 3.4 equiv.). After completion of the reaction (*E*)-3-(4-methoxystyryl)thiophene **82** was isolated by column chromatography (hexane). Isolated yield 43% (27 mg, white solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.41 (d, *J* = 8.7 Hz, 2H), 7.32 (m, 2H), 7.21 (d, *J* = 1.8 Hz, 1H), 7.00 (d, *J* = 16.3 Hz, 1H), 6.90-6.88 (m, 3H), 3.83 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 159.3, 140.5, 130.3, 128.4, 127.6, 126.2, 125.0, 121.6, 121.1, 114.3, 55.5.

The spectral data are matched with those reported in the literature (74).

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Fig. 27. ^{1}H NMR of 4-(1-Methylpyrrole) benzonitrile in $CDCl_3$ **(1)**[3]**.**

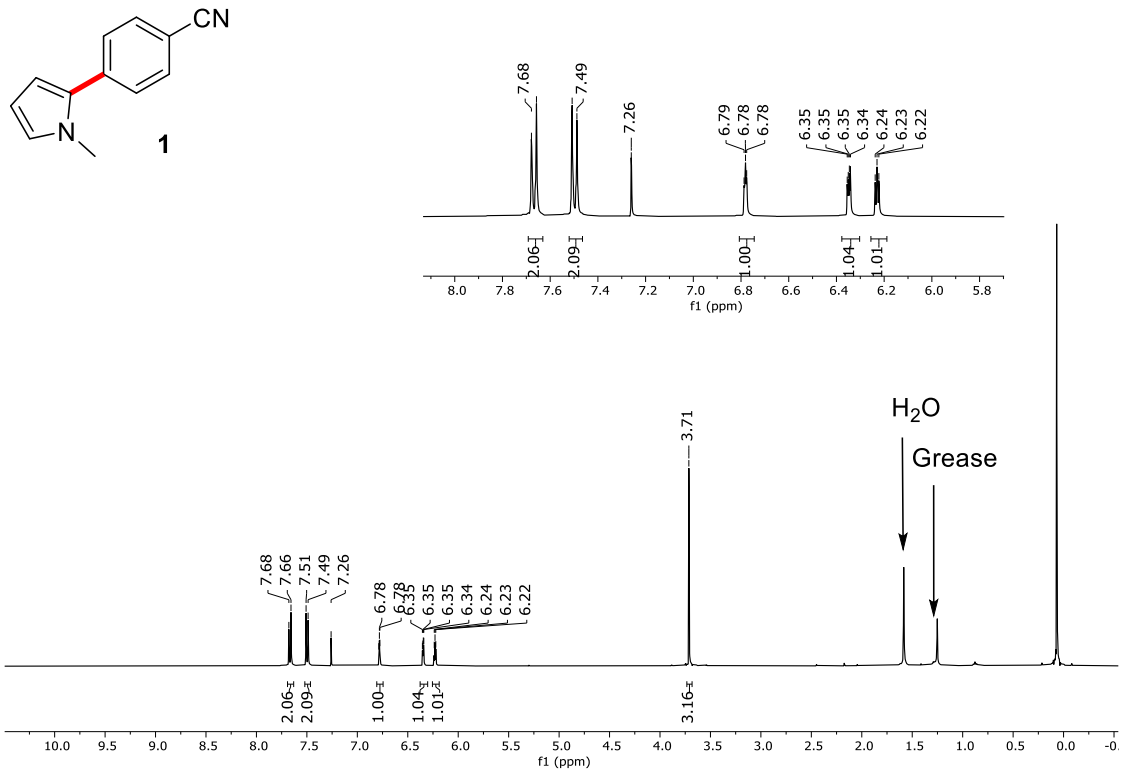

Fig. 28. ^{13}C NMR of 4-(1-Methylpyrrole) benzonitrile in $CDCl_3$ **(1)**[3]**.**

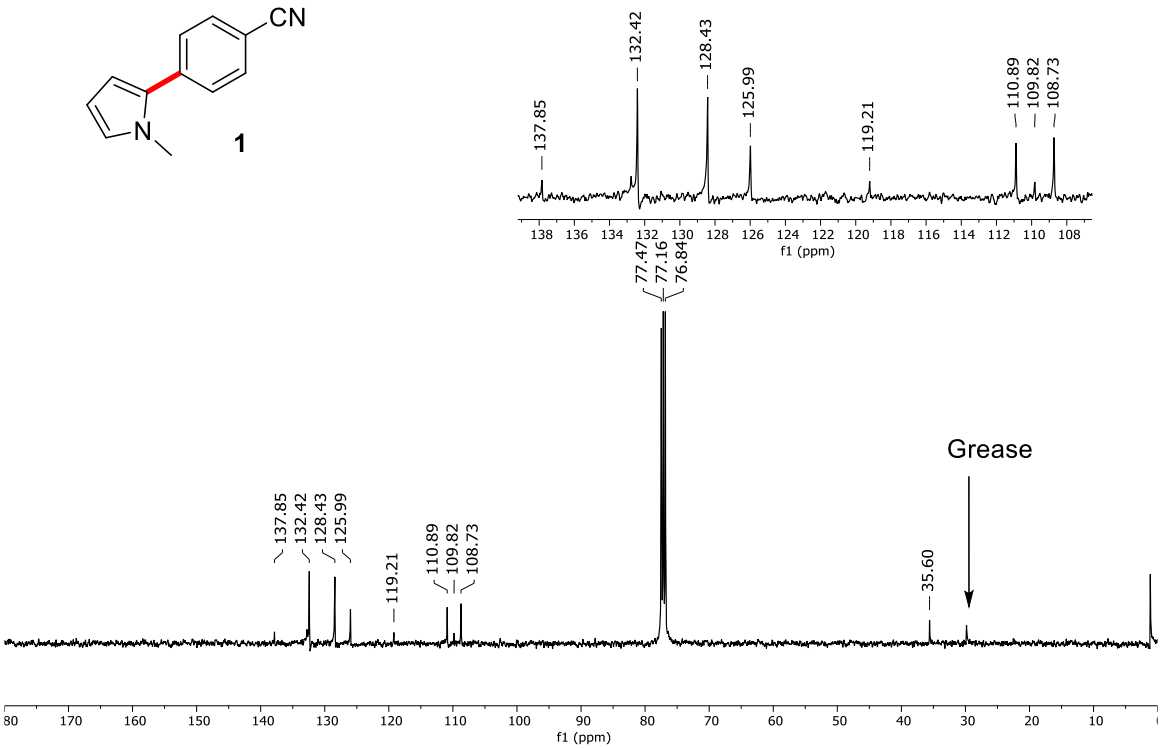

Fig. 29. [1] H NMR of 2-(4-Methoxyphenyl)-1-methyl-1*H*-pyrrole in $CDCl_3$ **(2)**[4]**.**

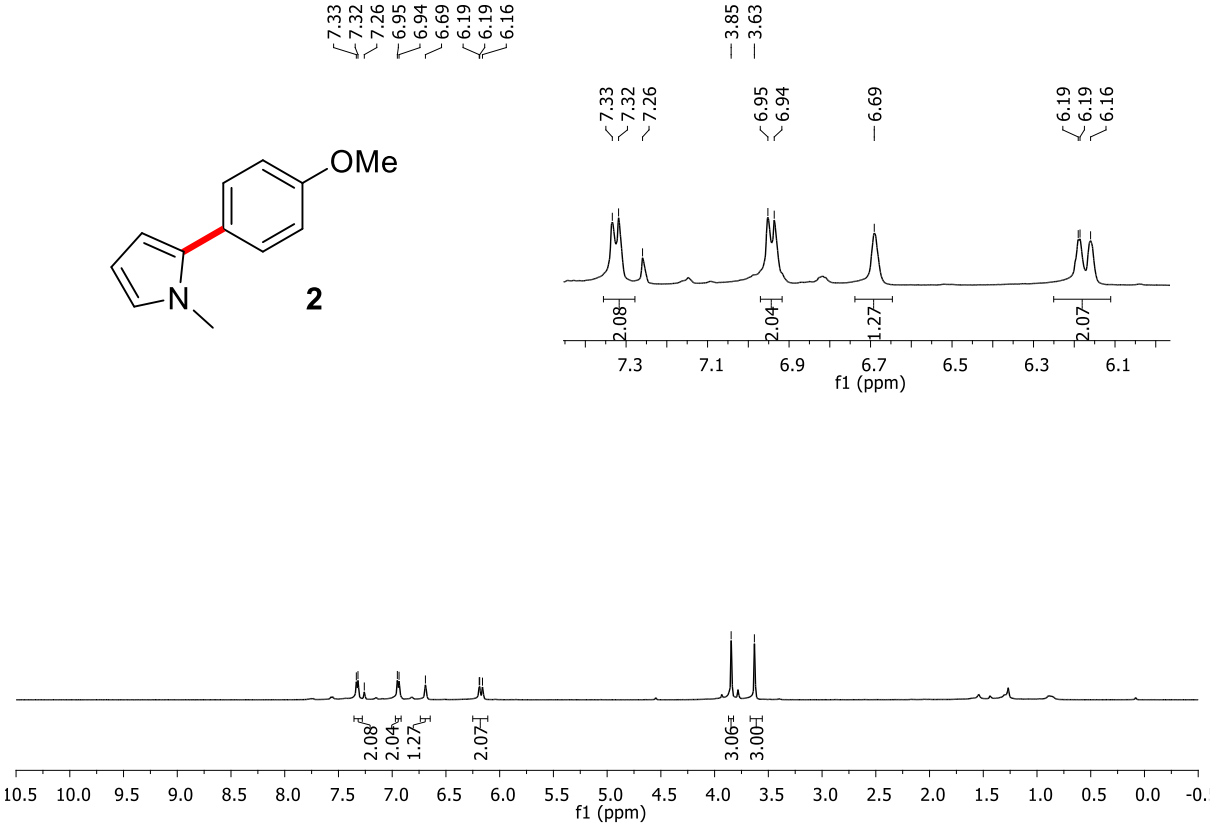

Fig. 30. [13] C NMR of 2-(4-Methoxyphenyl)-1-methyl-1*H*-pyrrole in $CDCl_3$ **(2)**[4]**.**

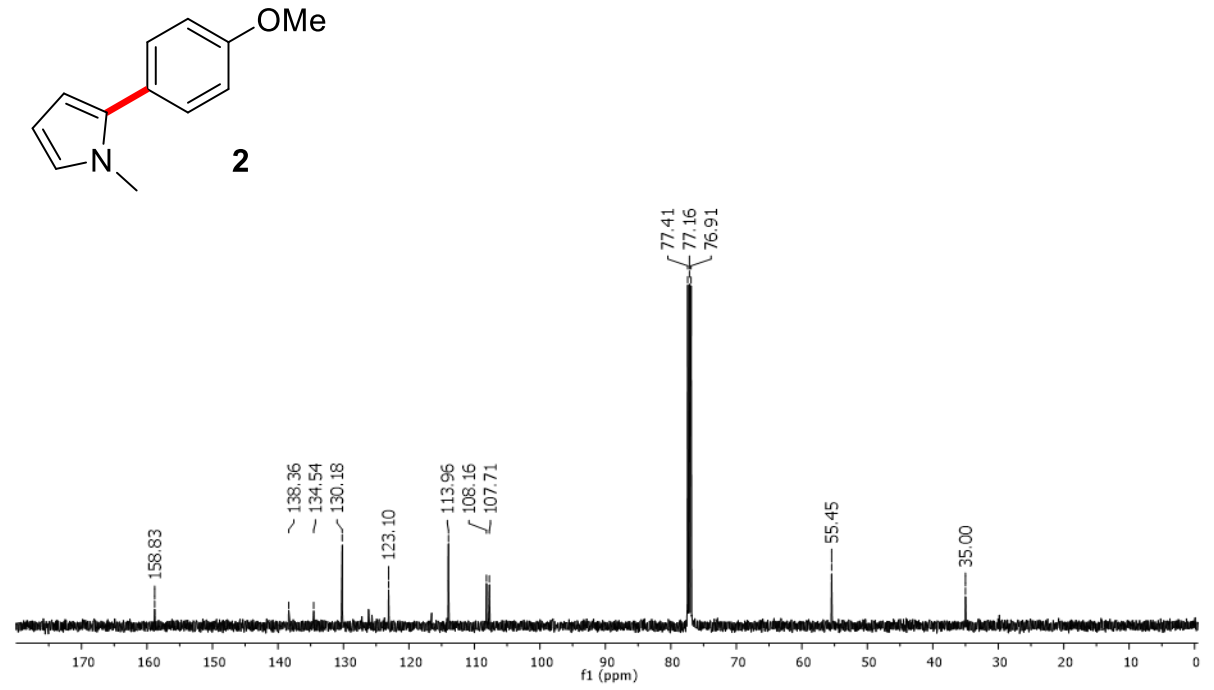

Fig. 31. [1] H NMR of 1-Methyl-2-(*p*-tolyl)-1*H*-pyrrole in $CDCl_3$ **(3)**[4]**.**

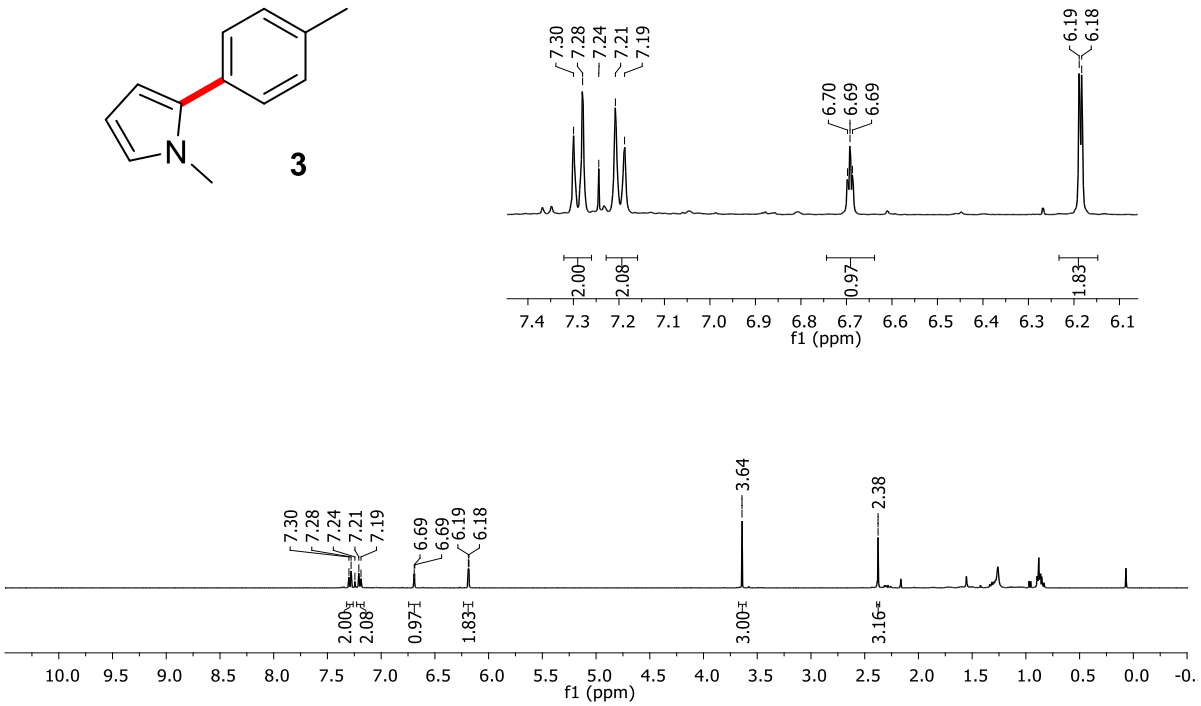

Fig. 32. [13] C NMR of 1-Methyl-2-(*p*-tolyl)-1*H*-pyrrole in $CDCl_3$ **(3)**[4]**.**

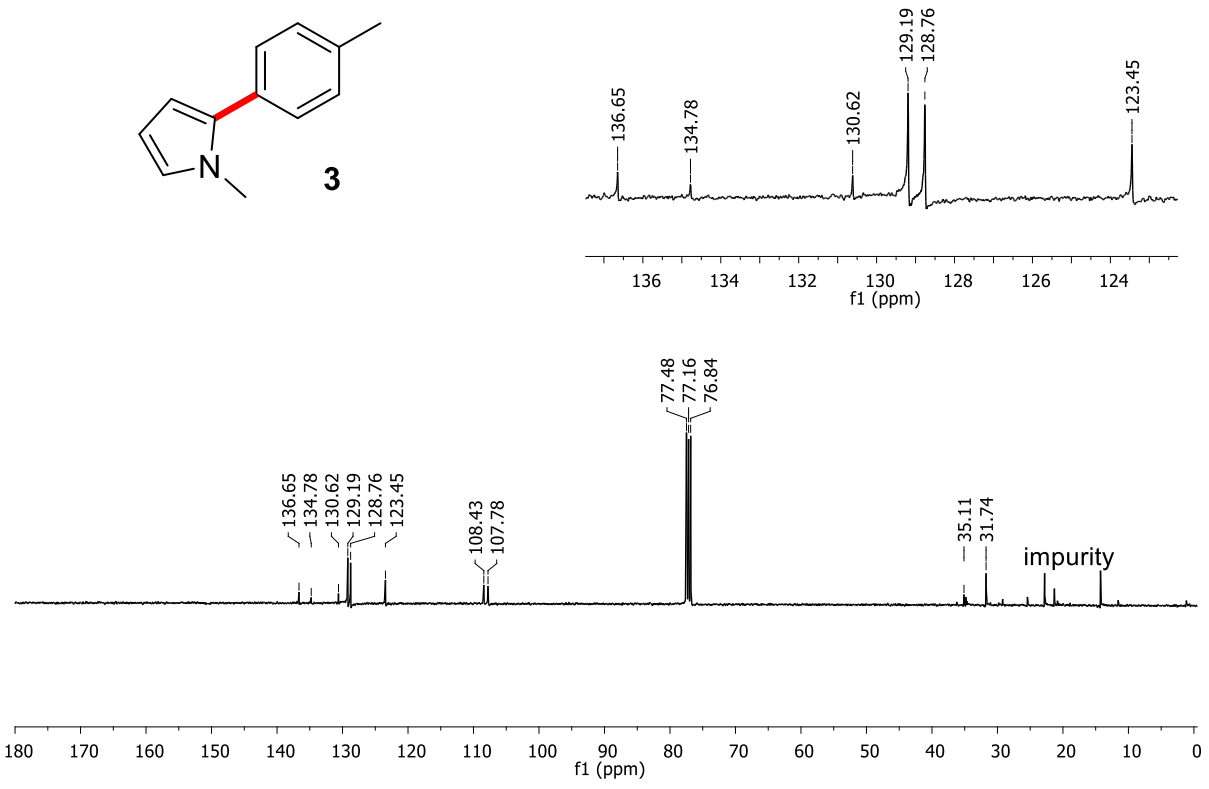

Fig. 33. ^{1}H NMR of 2-([1,1'-Biphenyl]-4-yl)furan in $CDCl_3$ **(4)**[5].

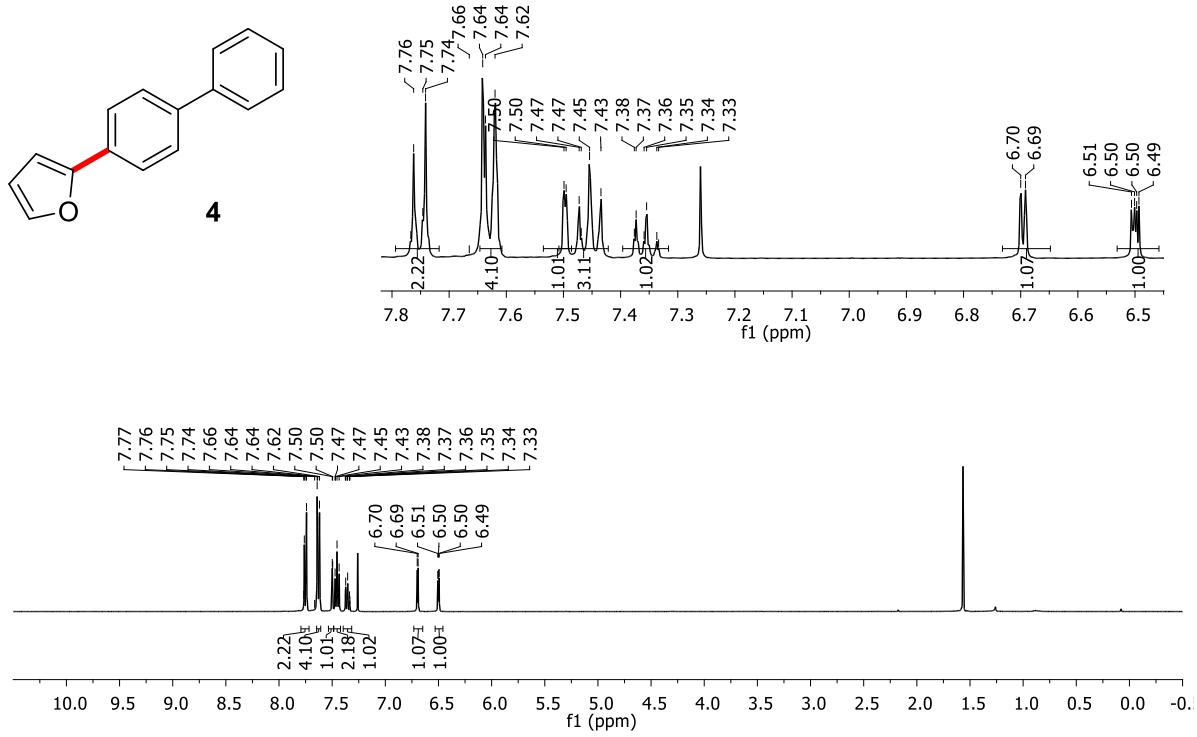

Fig. 34. ^{13}C NMR of 2-([1,1'-Biphenyl]-4-yl)furan in $CDCl_3$ **(4)**[5].

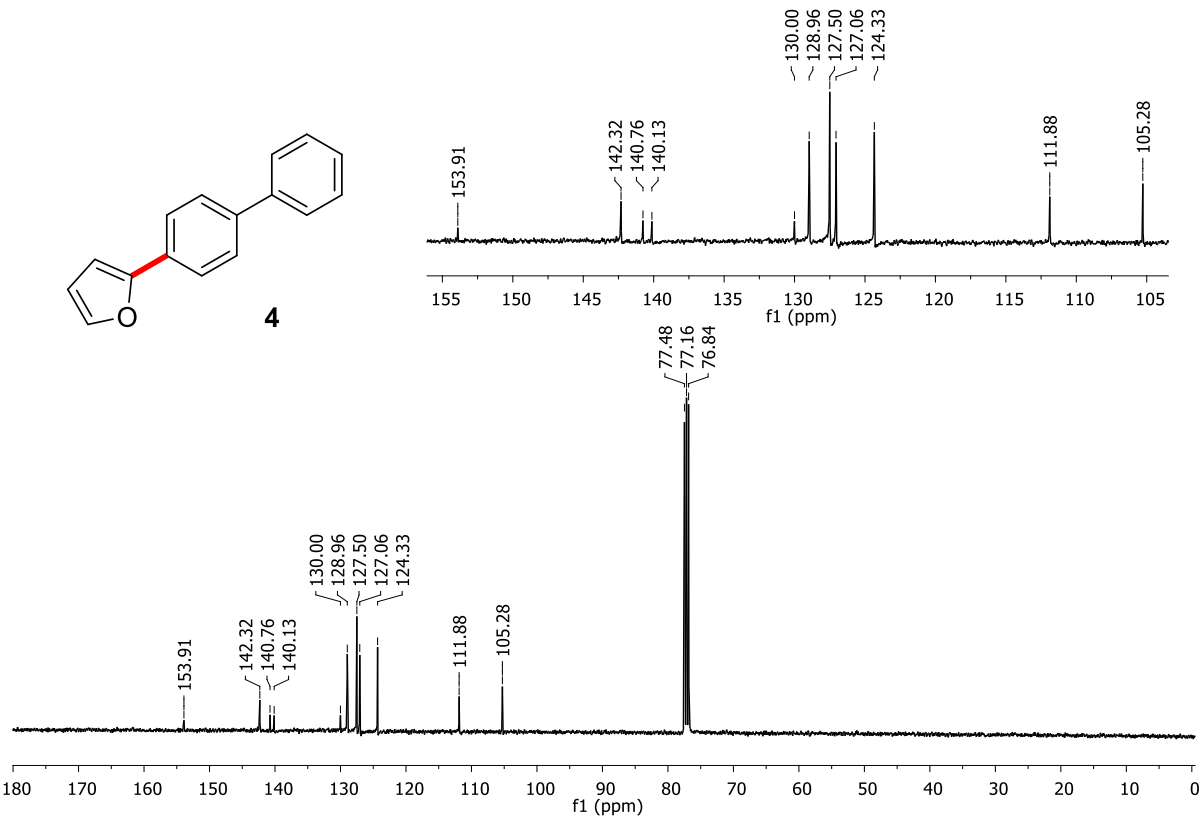

Fig. 35. ^{1}H NMR of 4-(Furan-2-yl)benzonitrile in $CDCl_3$ **(5)**[6]**.**

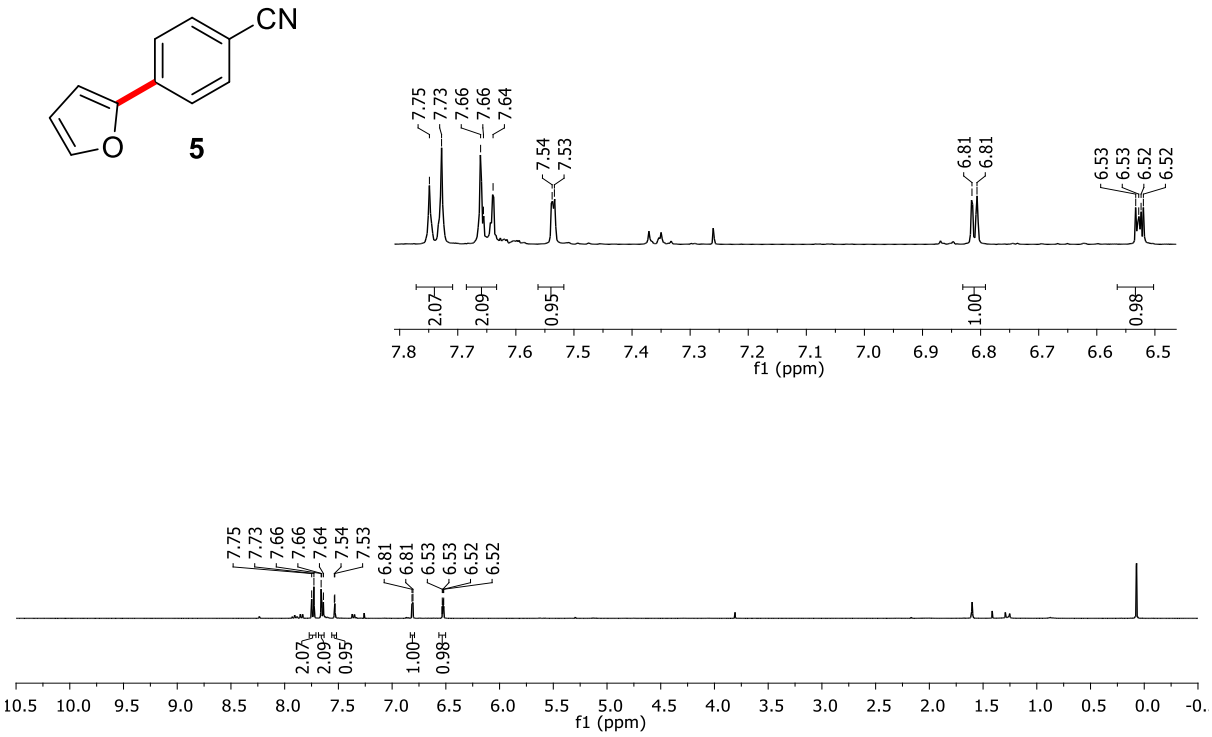

Fig. 36. ^{13}C NMR of 4-(Furan-2-yl)benzonitrile in $CDCl_3$ **(5)**[6]

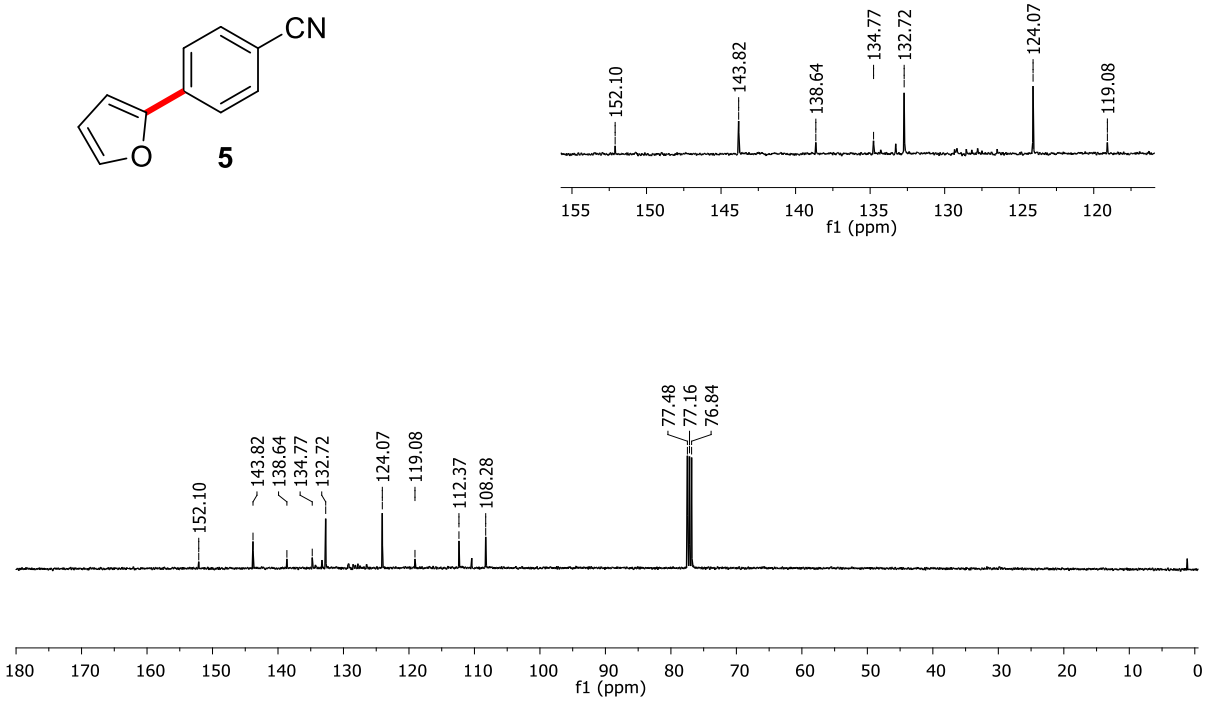

Fig. 37. ^{1}H NMR of 2-(4-Methoxyphenyl)furan in $CDCl_3$ **(6)**[7].

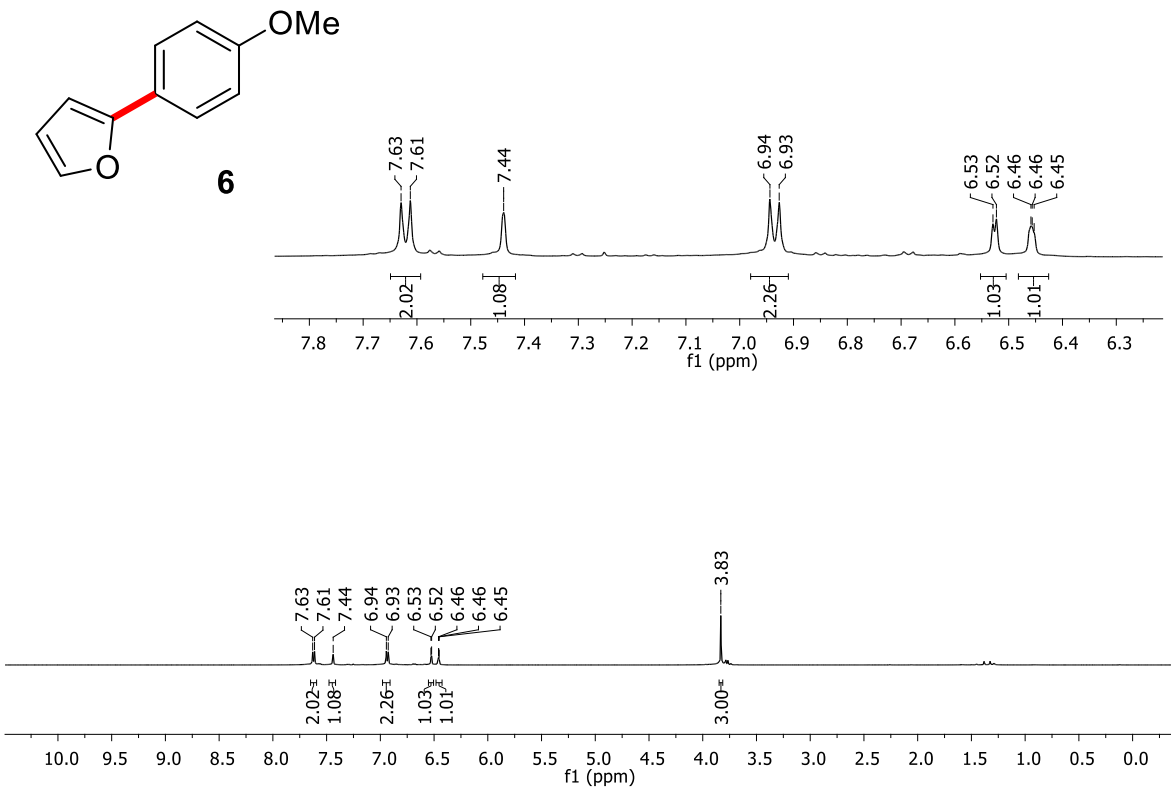

Fig. 38. ^{13}C NMR of 2-(4-Methoxyphenyl)furan in $CDCl_3$ **(6)**[7].

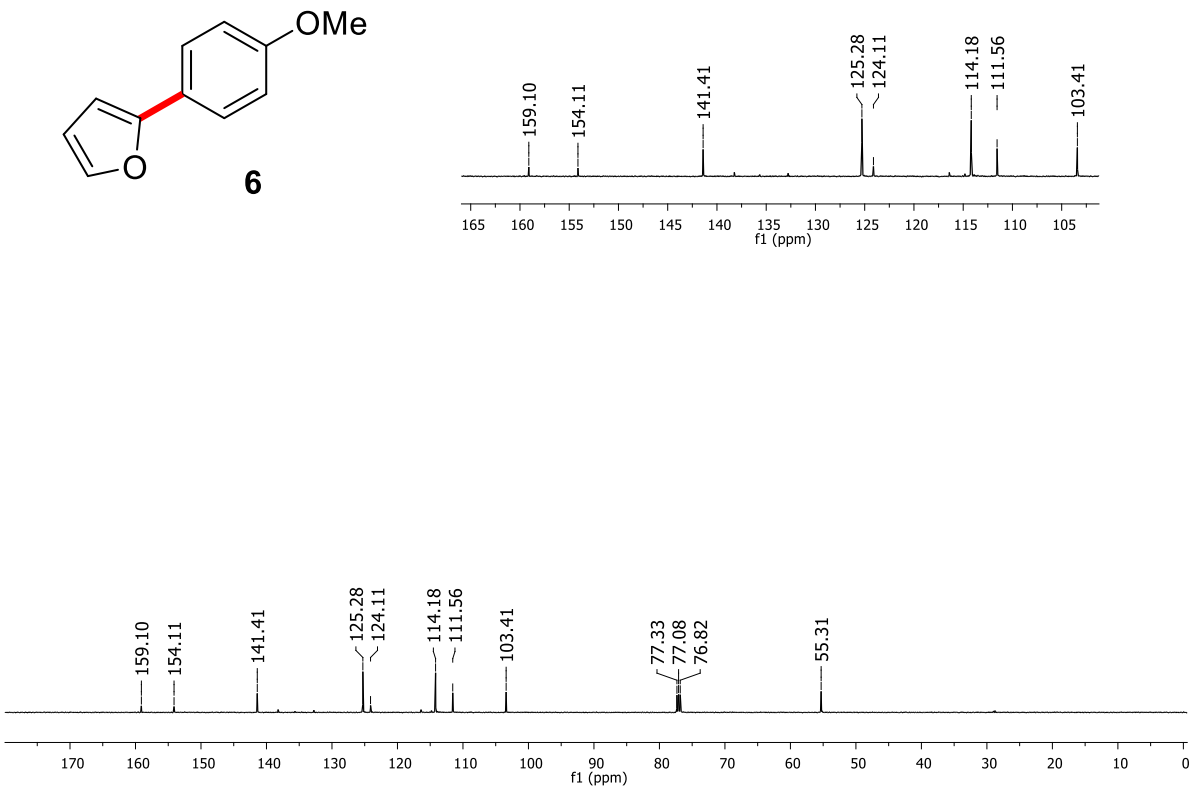

Fig. 39. ^{1}H NMR of 2-([1,1'-Biphenyl]-4-yl)benzofuran in $CDCl_3$ **(7)**[8]**.**

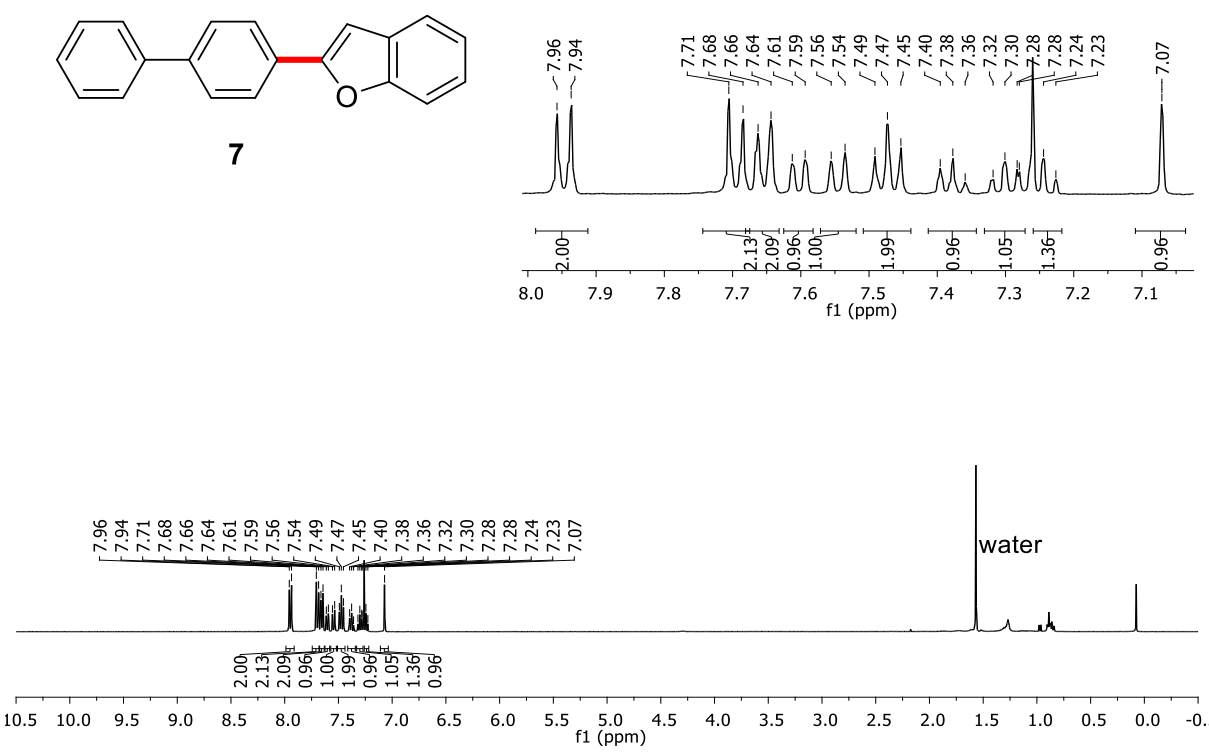

Fig. 40. ^{13}C NMR of 2-([1,1'-Biphenyl]-4-yl)benzofuran in $CDCl_3$ **(7)**[8].

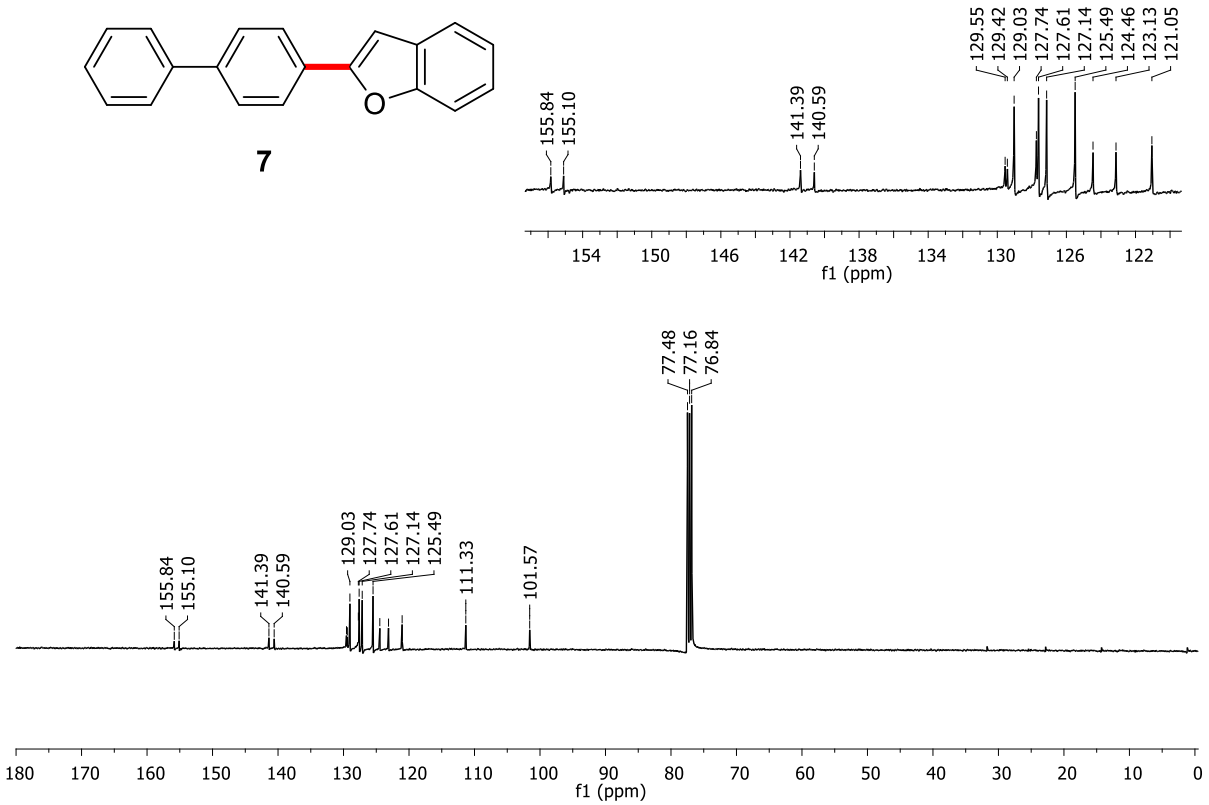

Fig. 41. ^{1}H NMR of 4-(Thiophen-2-yl)benzonitrile in $CDCl_3$ **(8)**[9]**.**

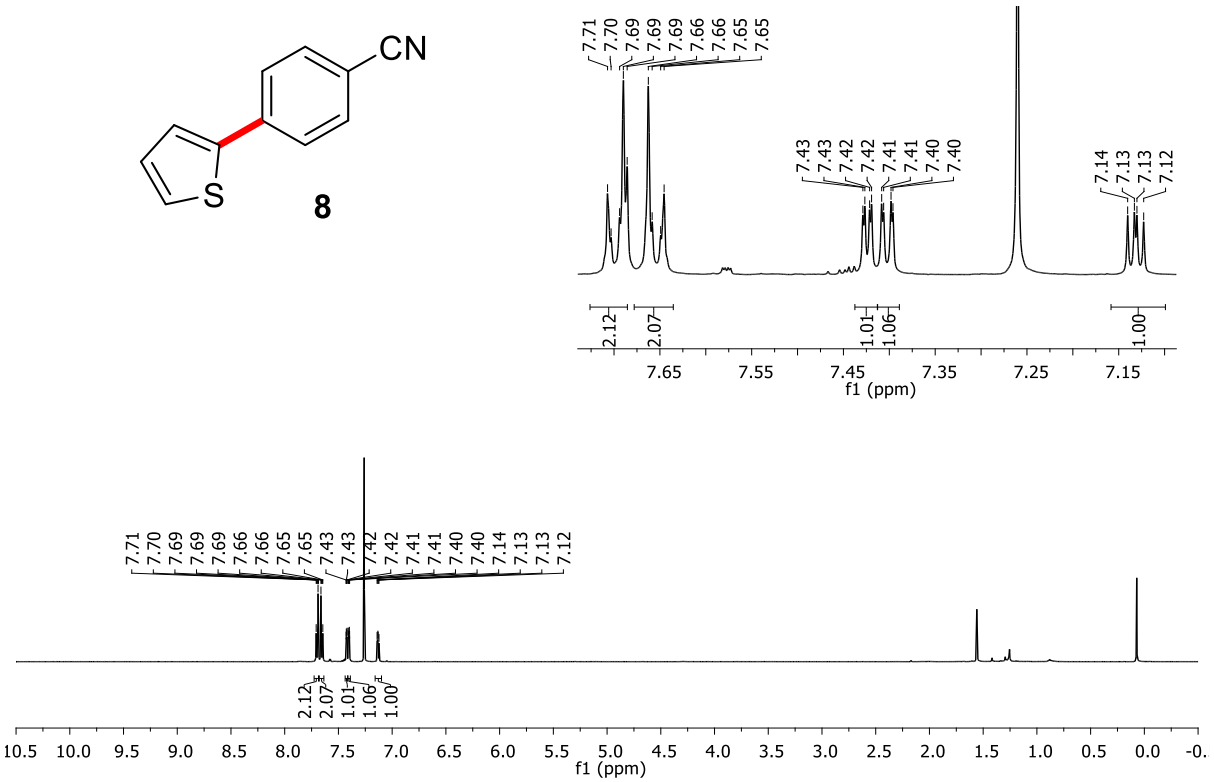

Fig. 42. ^{13}C NMR of 4-(Thiophen-2-yl)benzonitrile in $CDCl_3$ **(8)**[9]**.**

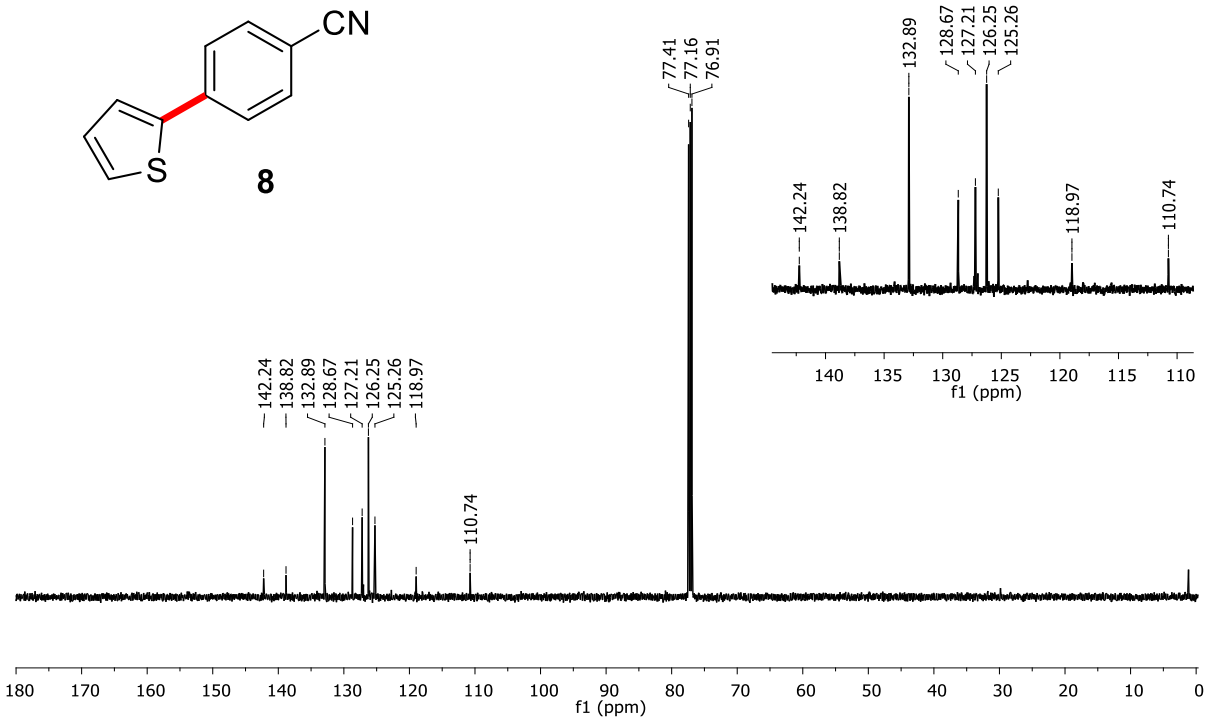

Fig. 43. ^{1}H NMR of 2-(p-Tolyl)thiophene in $CDCl_3$ **(9)**[10]**.**

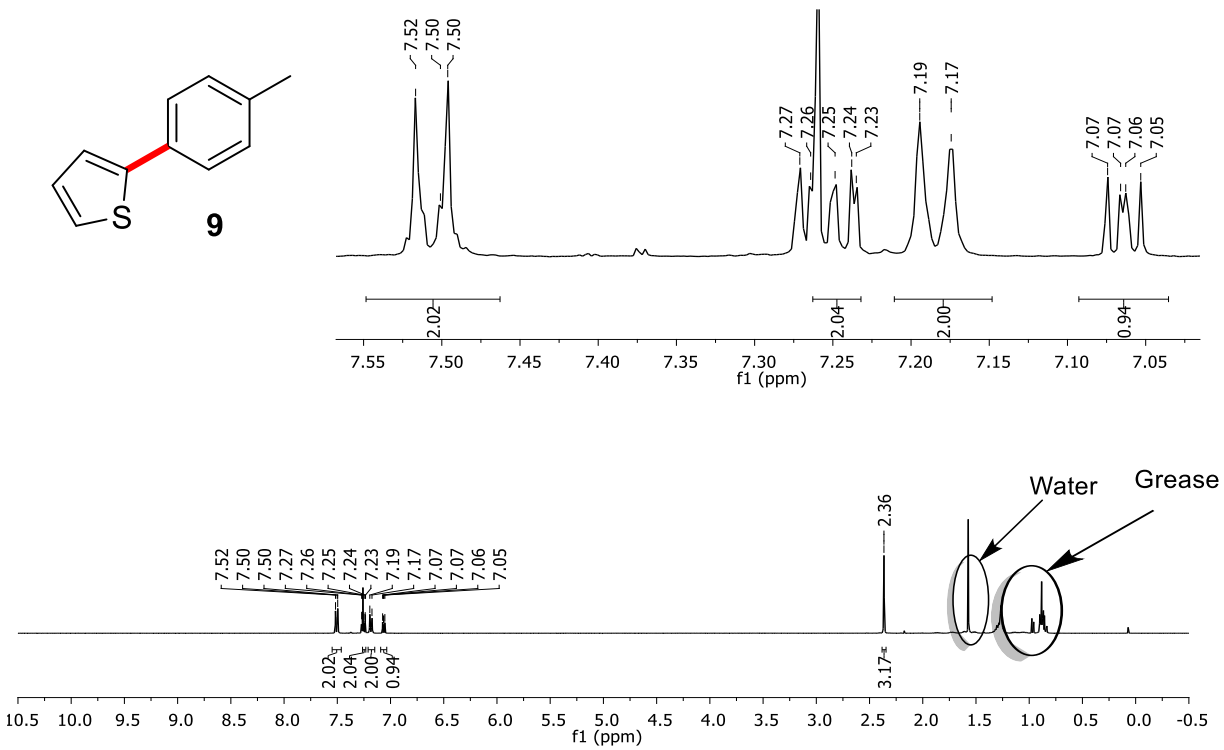

Fig. 44. ^{13}C NMR of 2-(p-Tolyl)thiophene in $CDCl_3$ **(9)**[10]**.**

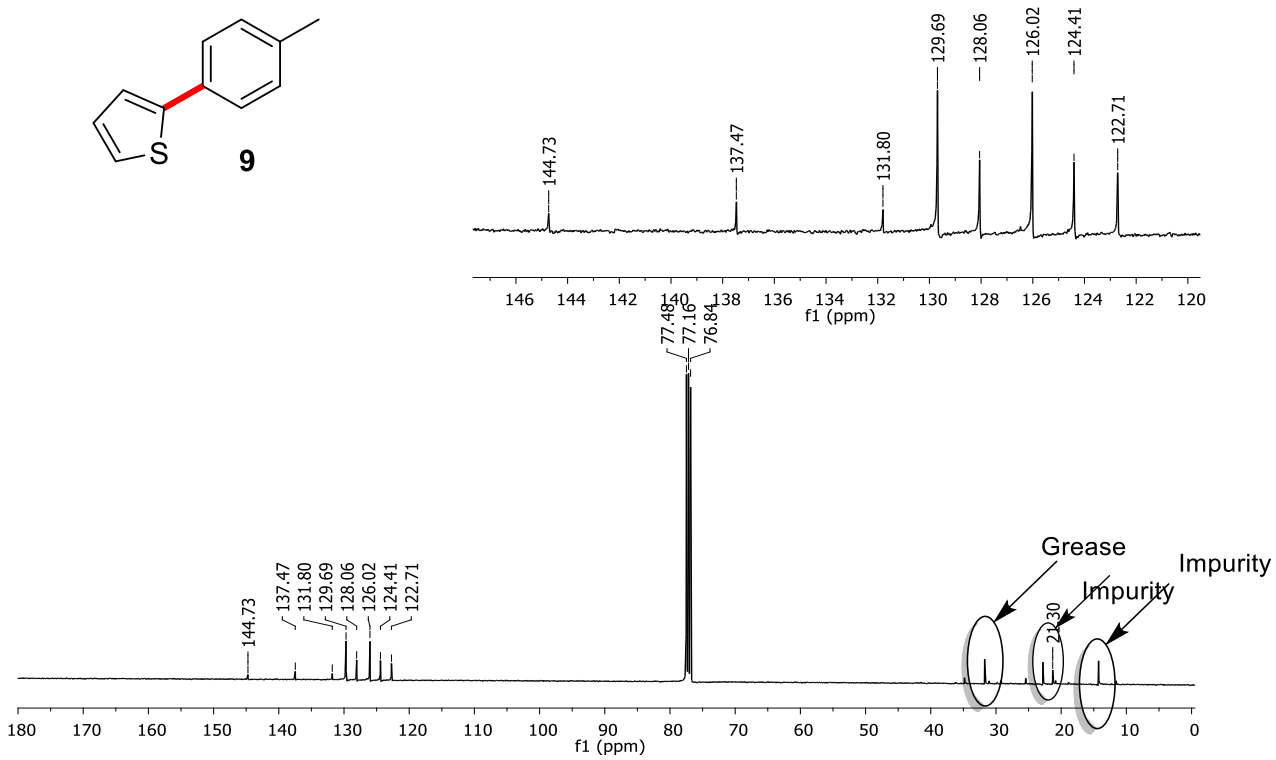

Fig. 45. ^{1}H NMR of 2-(4-Methoxyphenyl)thiazole in $CDCl_3$ **(10)**[11]**.**

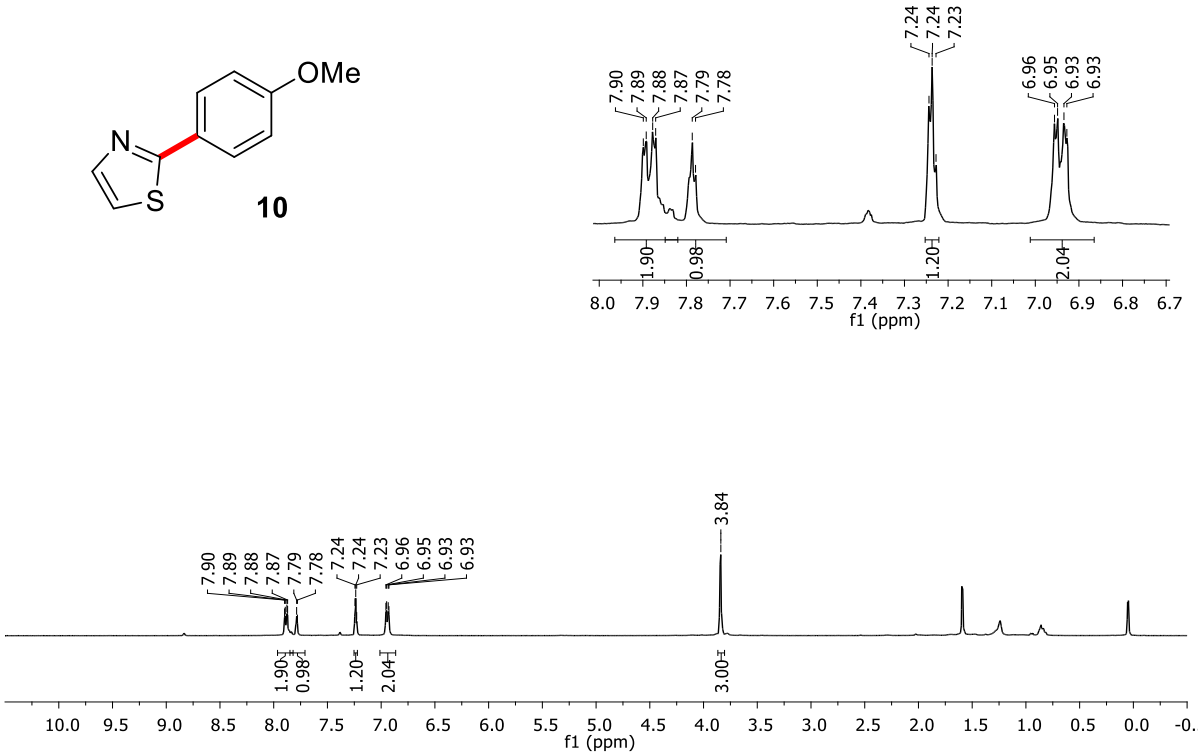

Fig. 46. ^{13}C NMR of 2-(4-Methoxyphenyl)thiazole in $CDCl_3$ **(10)**[11]**.**

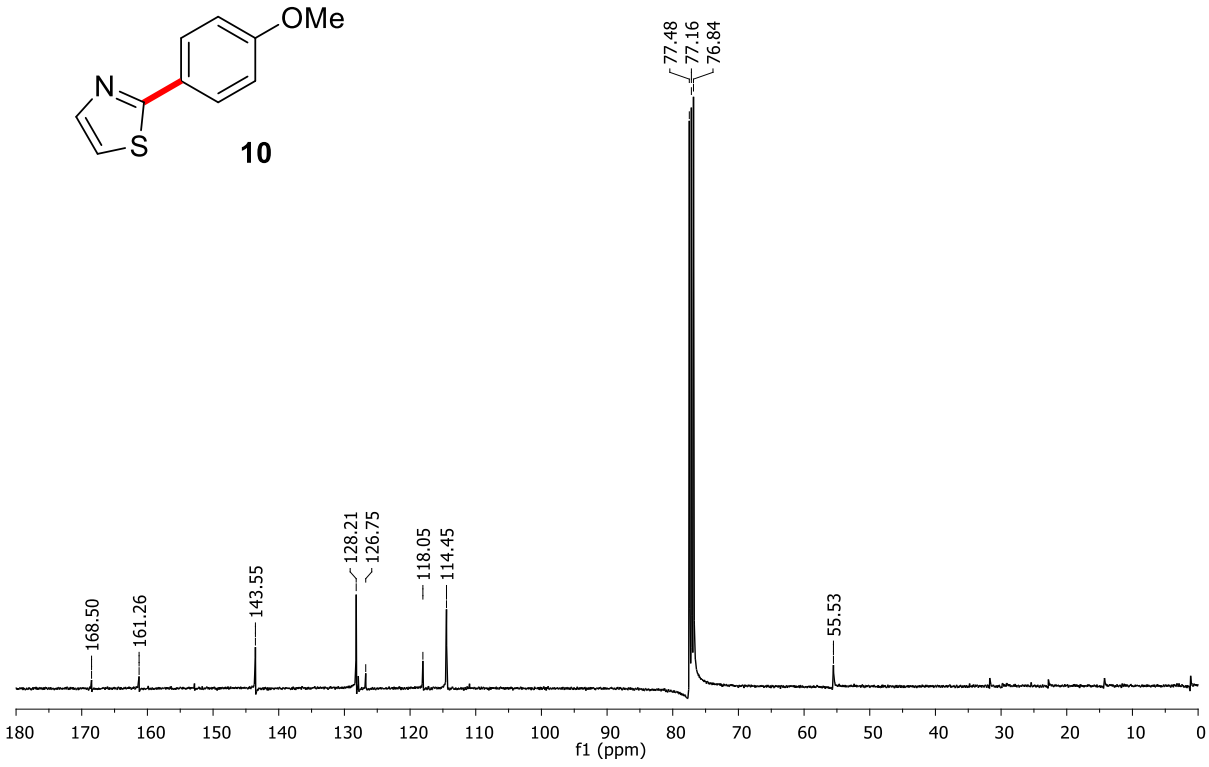

Fig. 47. [1] H NMR of [1, 1'-Biphenyl]-4-carbonitrile in $CDCl_3$ **(11 & 12)**[12]**.**

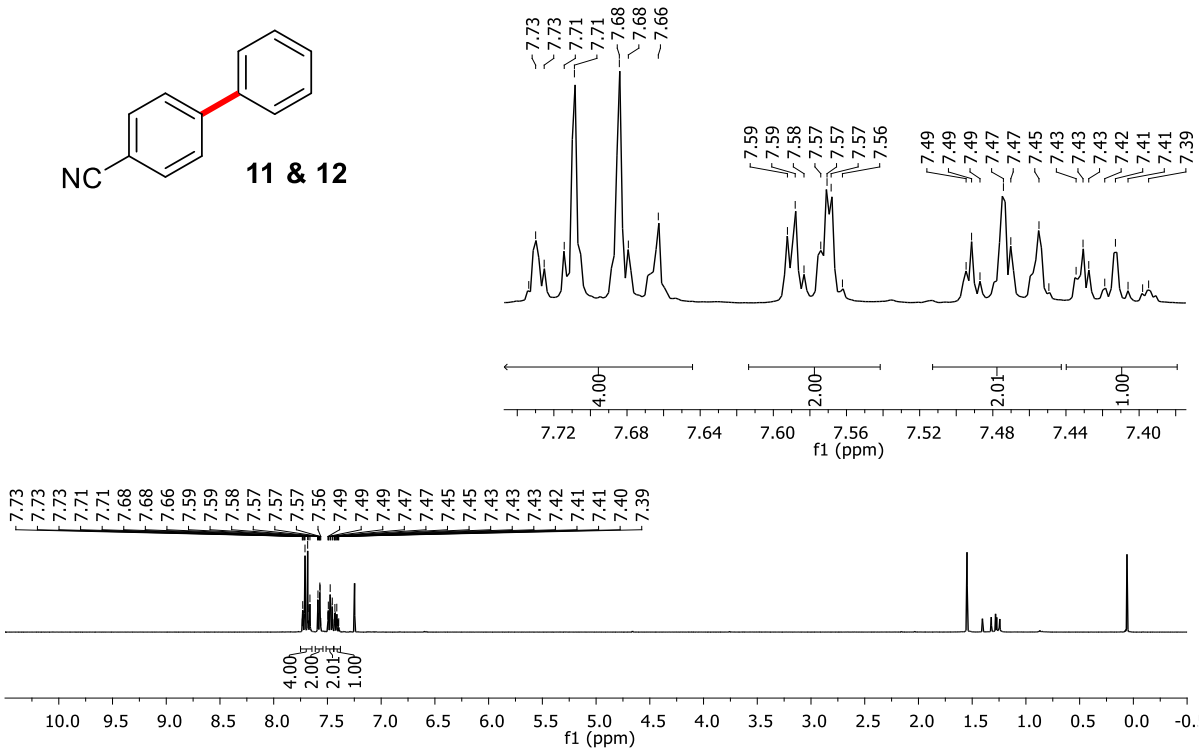

Fig. 48. [13] C NMR of [1, 1'-Biphenyl]-4-carbonitrile in $CDCl_3$ **(11 & 12)**[12]**.**

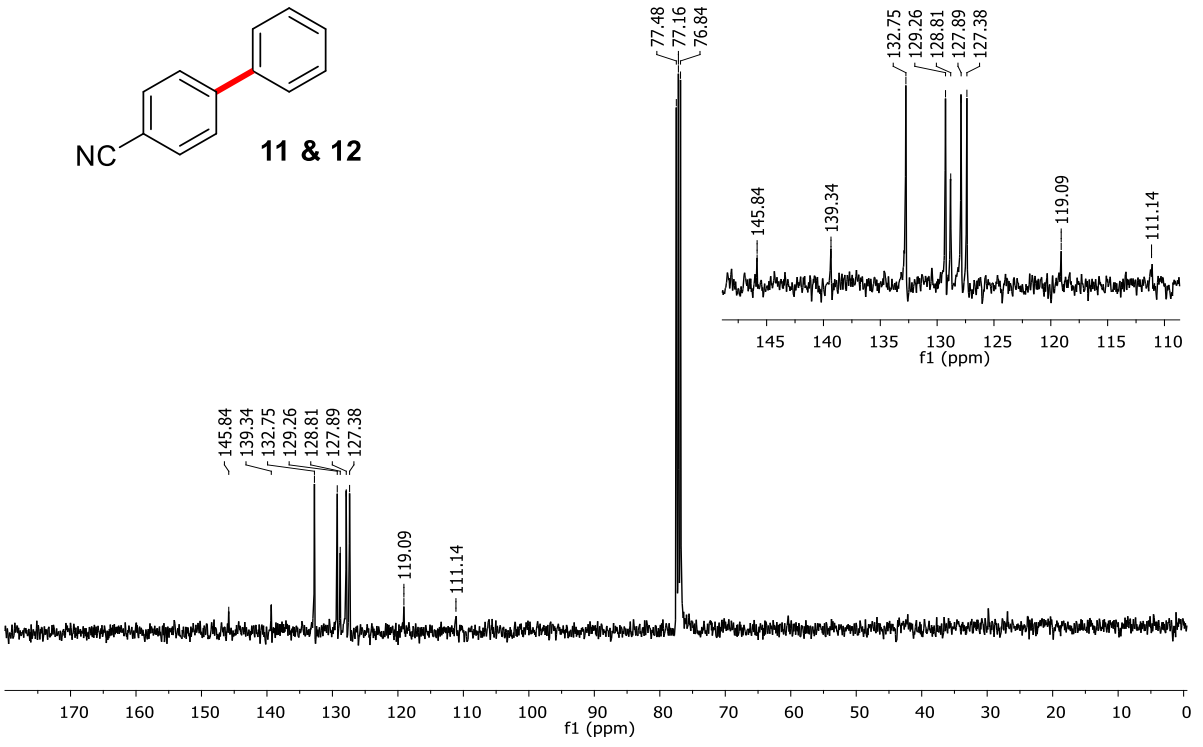

Fig. 49. ^{1}H NMR of 4-Methyl-1,1'-biphenyl in $CDCl_3$ **(13)**[13].

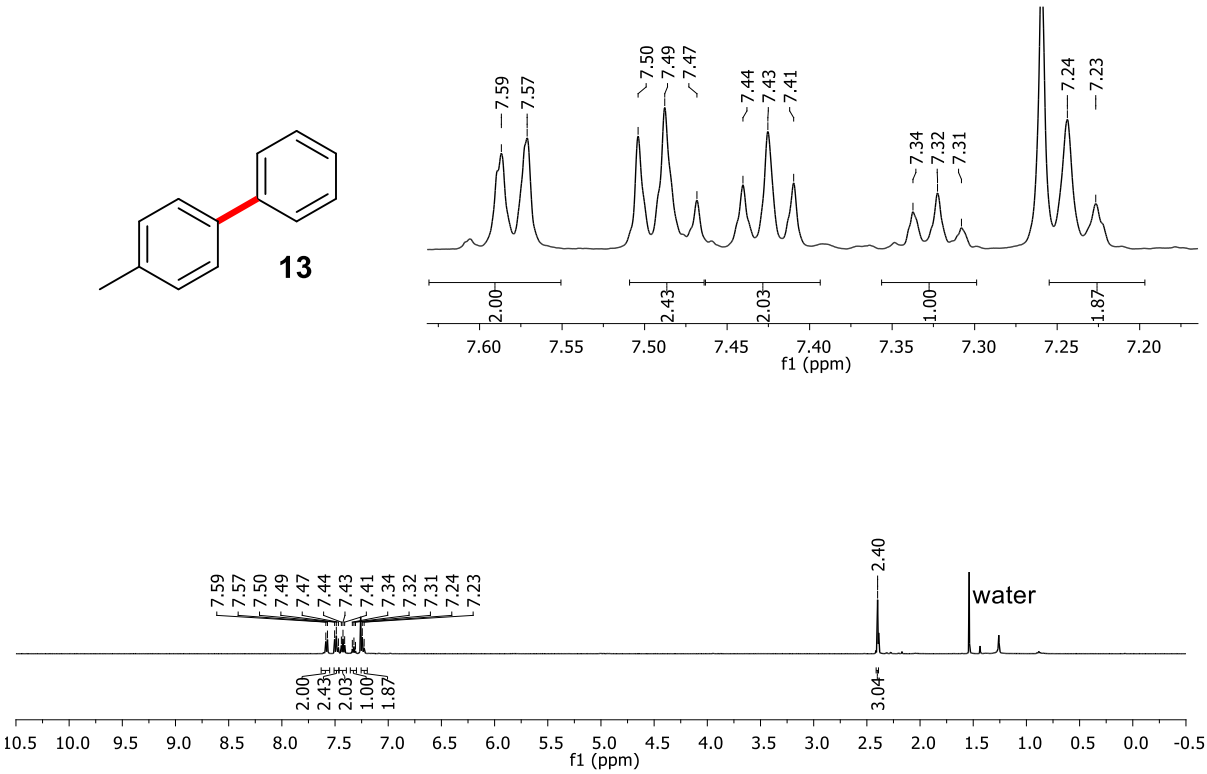

Fig. 50. ^{13}C NMR of 4-Methyl-1,1'-biphenyl in $CDCl_3$ **(13)**[13].

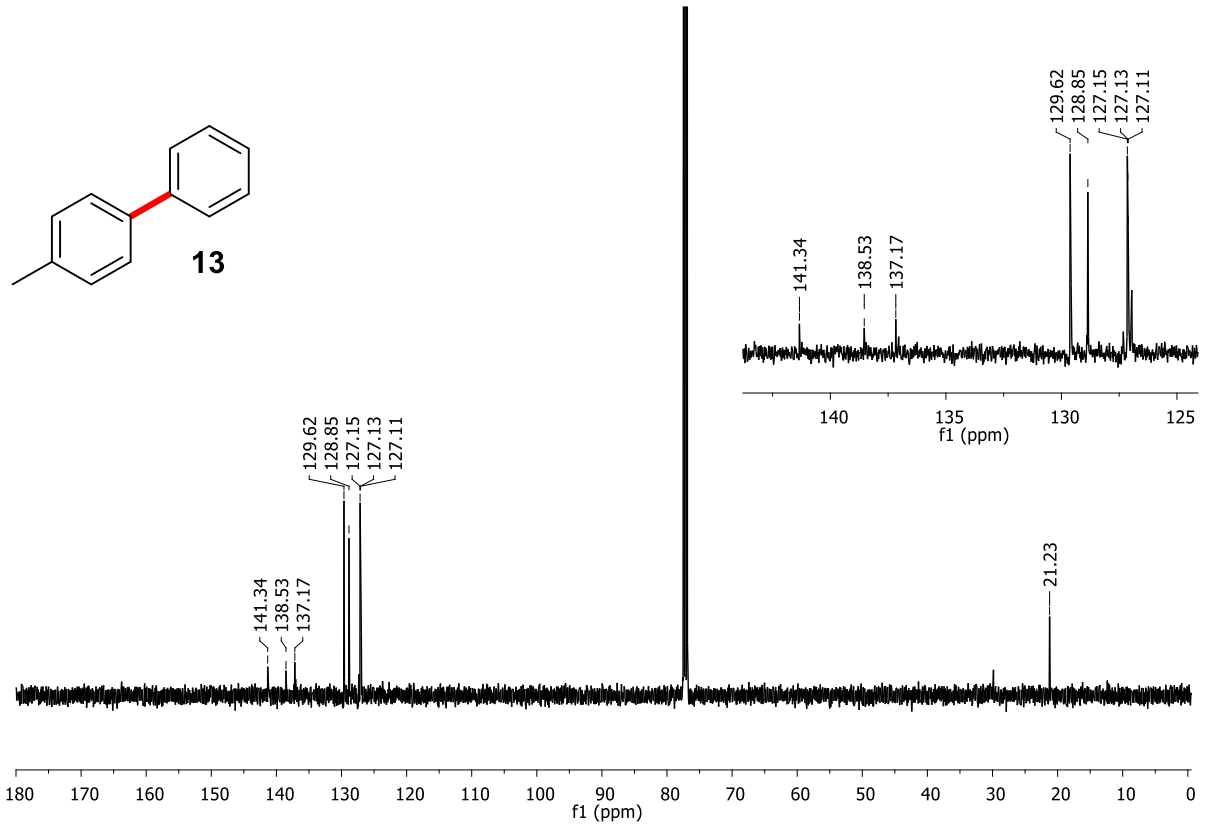

Fig. 51. [1] H NMR of 4-Methoxy-1,1'-biphenyl in $CDCl_3$ **(14)**[13].

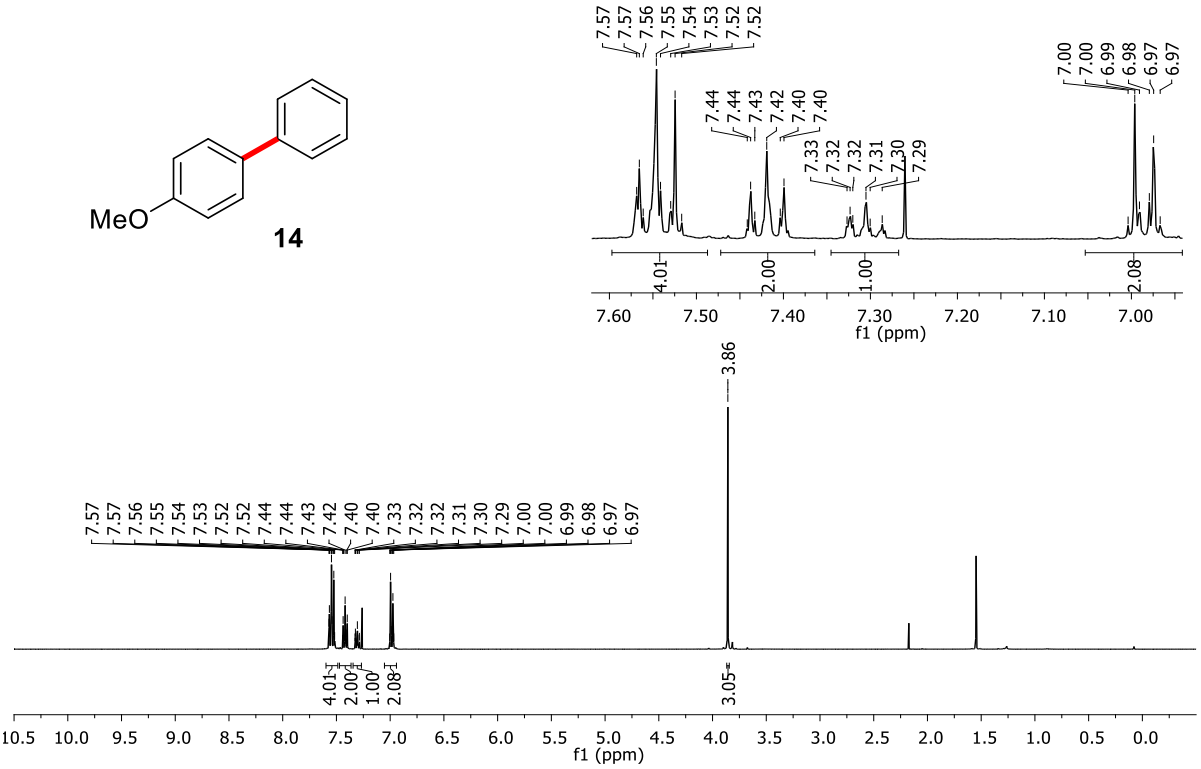

Fig. 52. [13]C NMR of 4-Methoxy-1,1'-biphenyl in $CDCl_3$ **(14)**[13].

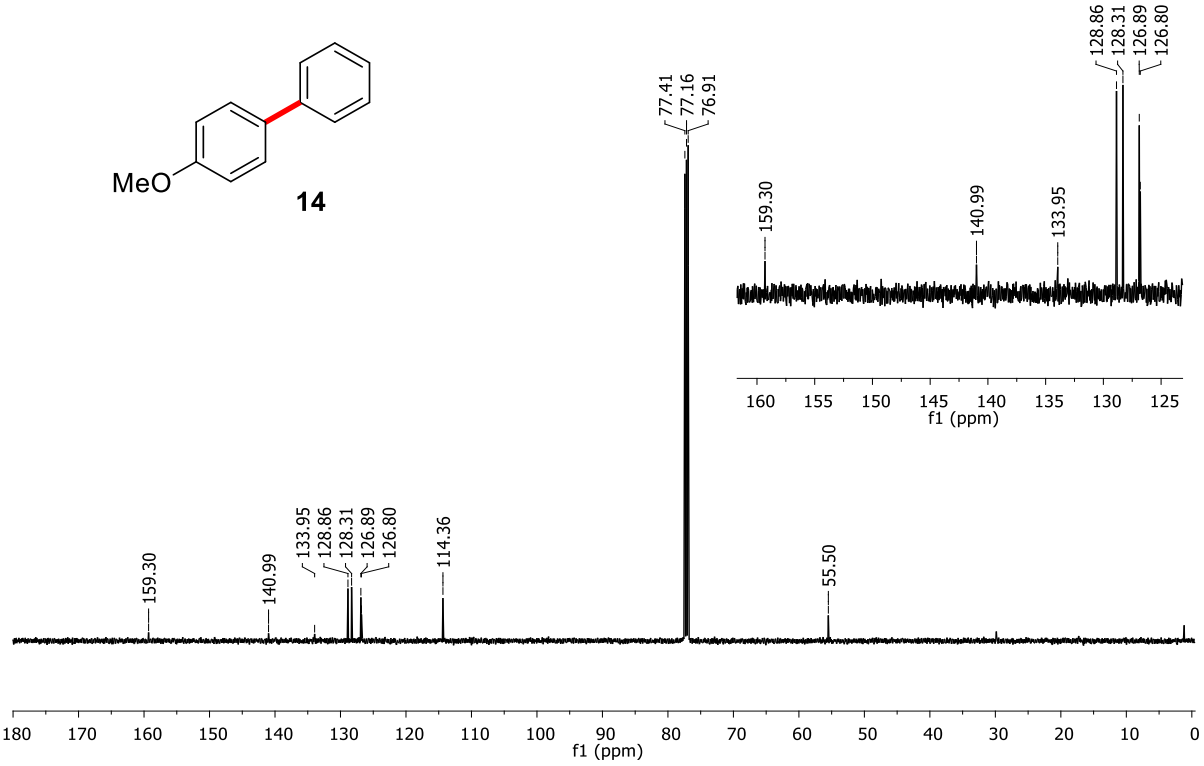

Fig. 53. ^{1}H NMR of 4-Iodo-1,1'-biphenyl in $CDCl_3$ **(15)**[14]**.**

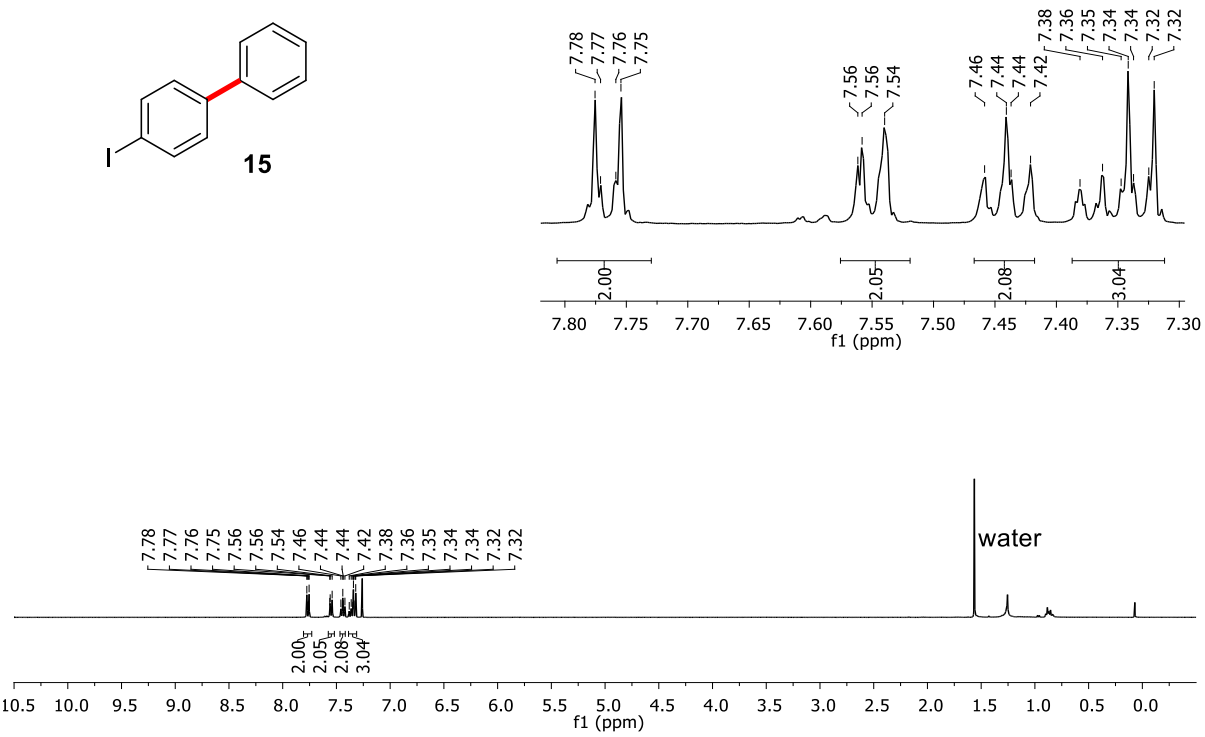

Fig. 54. ^{13}C NMR of 4-Iodo-1,1'-biphenyl in $CDCl_3$ **(15)**[14]**.**

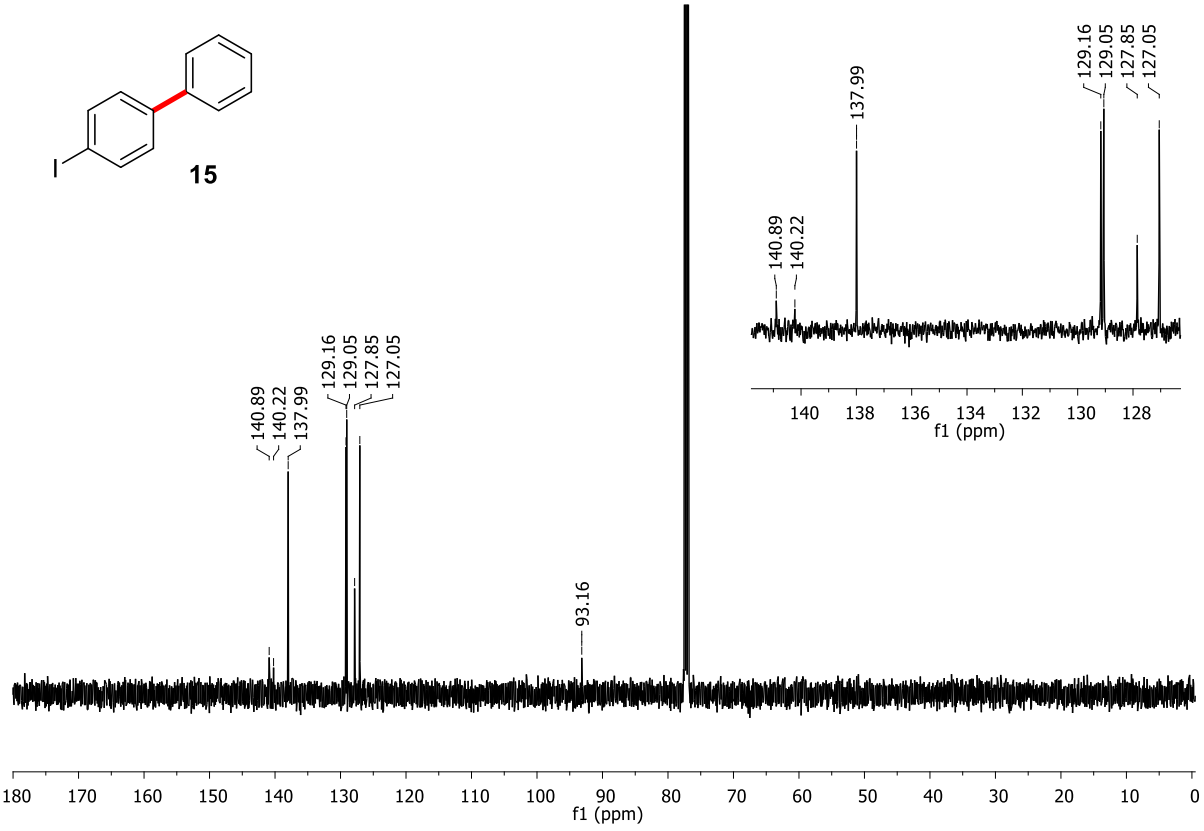

Fig. 55. ^{1}H NMR of 4-Chloro-1,1'-biphenyl in $CDCl_3$ **(16)**[15].

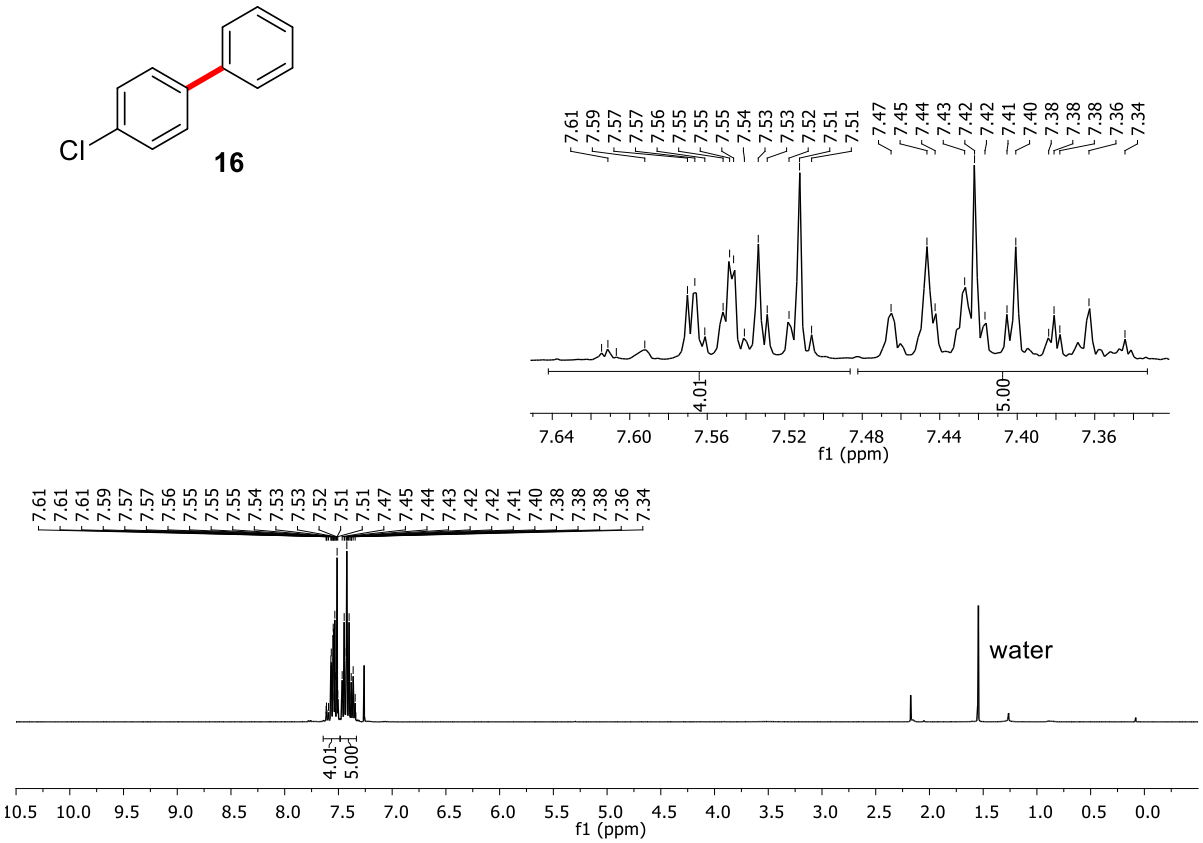

Fig. 56. ^{13}C NMR of 4-Chloro-1,1'-biphenyl in $CDCl_3$ **(16)**[15].

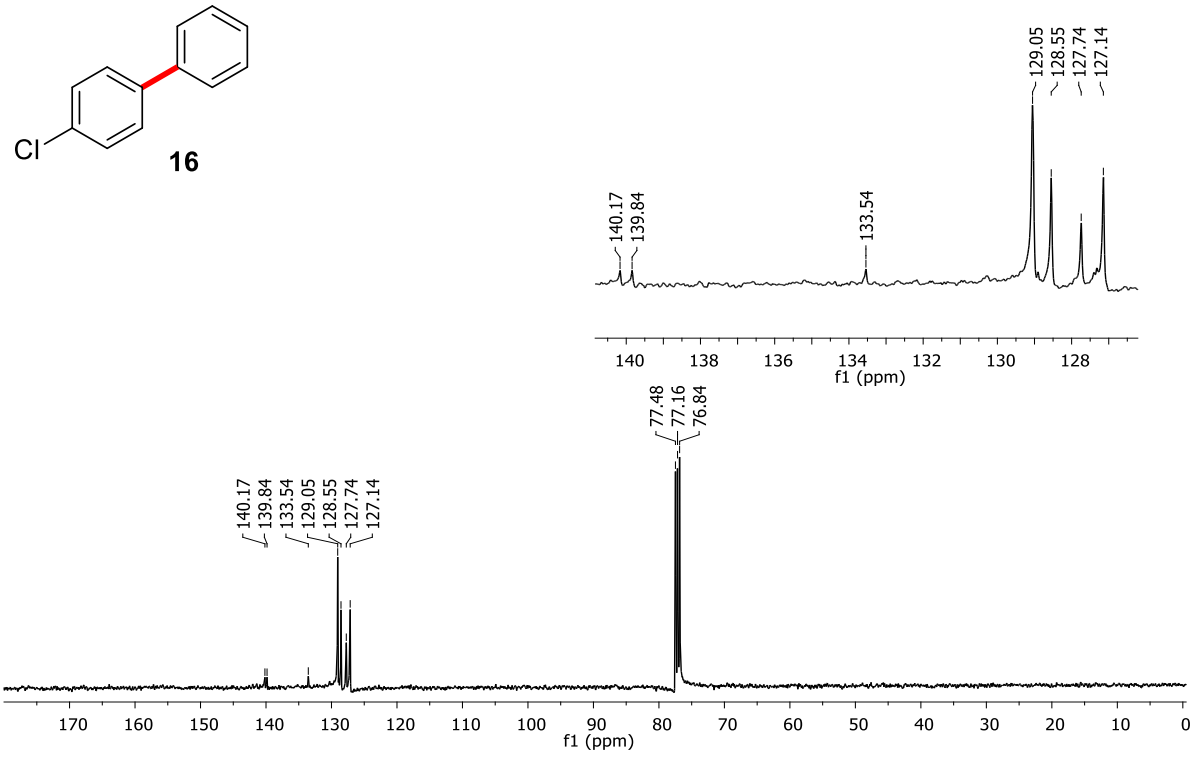

Fig. 57. [1] H NMR of 2,4,6-Trimethoxy-1,1'-biphenyl in $CDCl_3$ **(17)**[16].

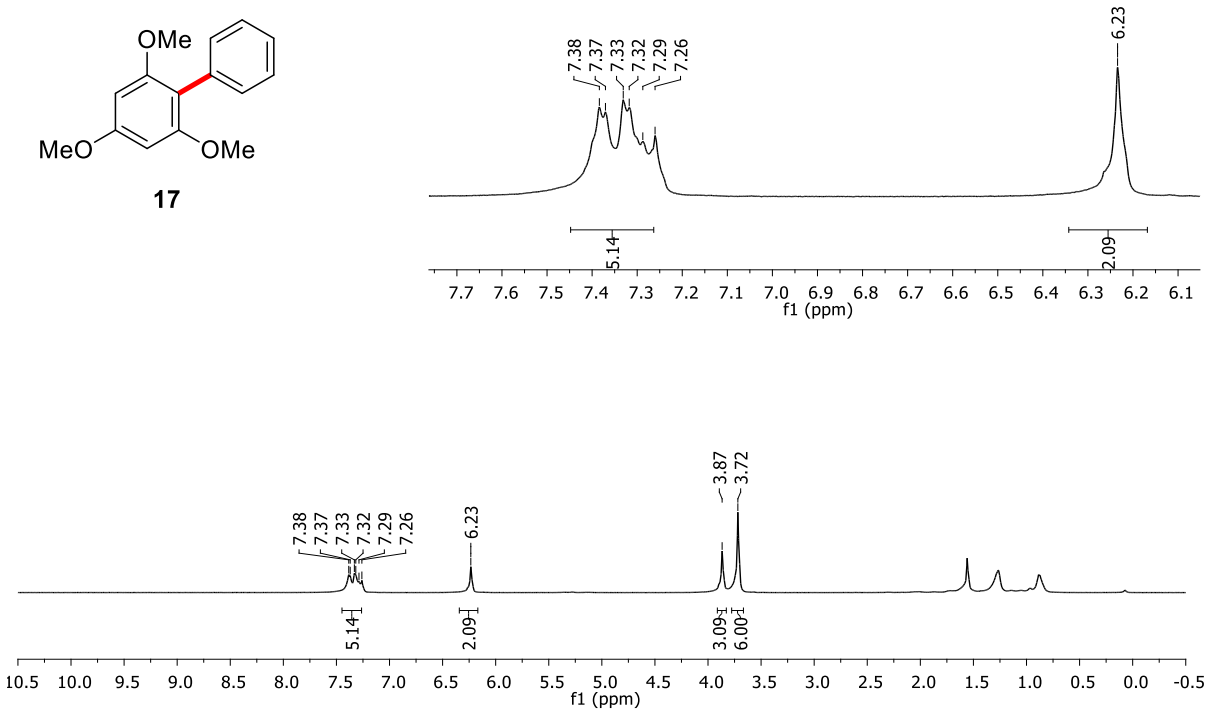

Fig. 58. [13]C NMR of 2,4,6-Trimethoxy-1,1'-biphenyl in $CDCl_3$ **(17)**[16].

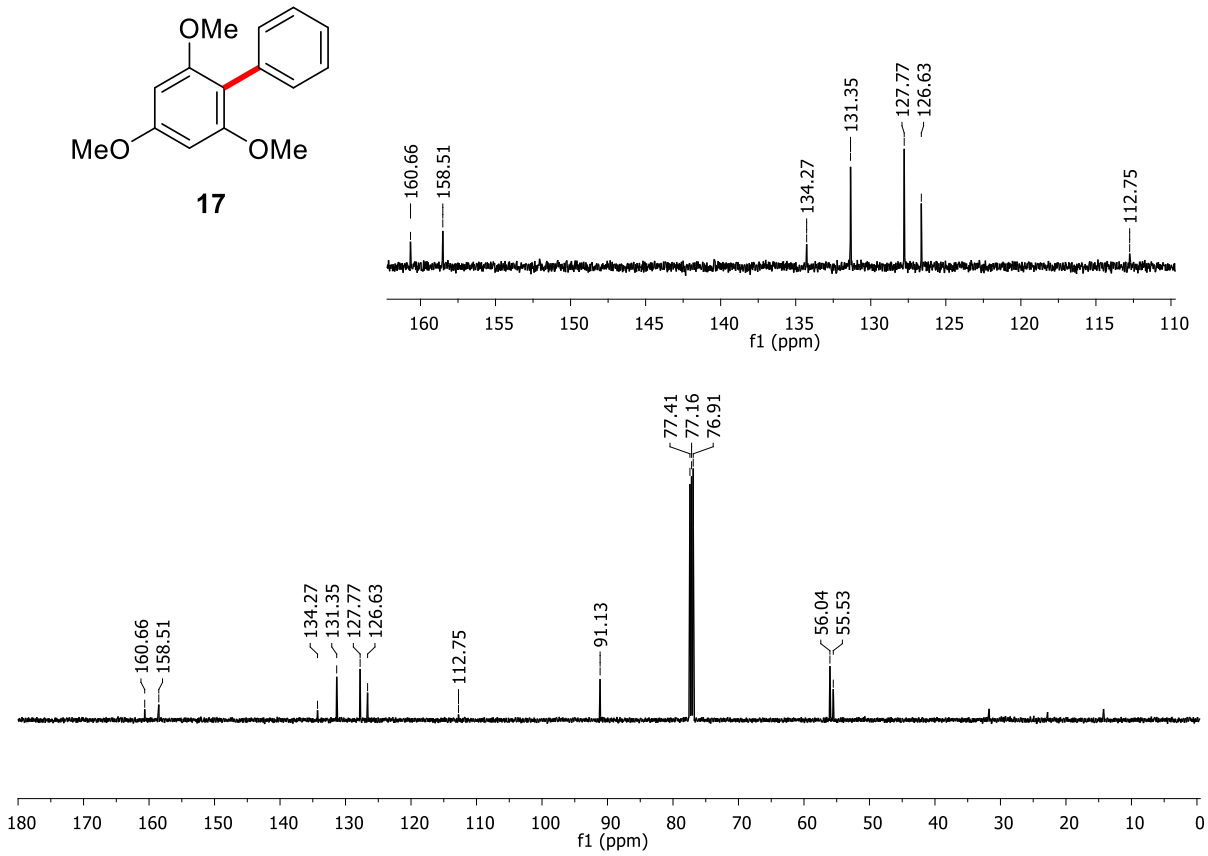

Fig. 59. [1] H NMR of 2,5-Dimethoxy-1,1'-biphenyl in $CDCl_3$ **(18)**[17].

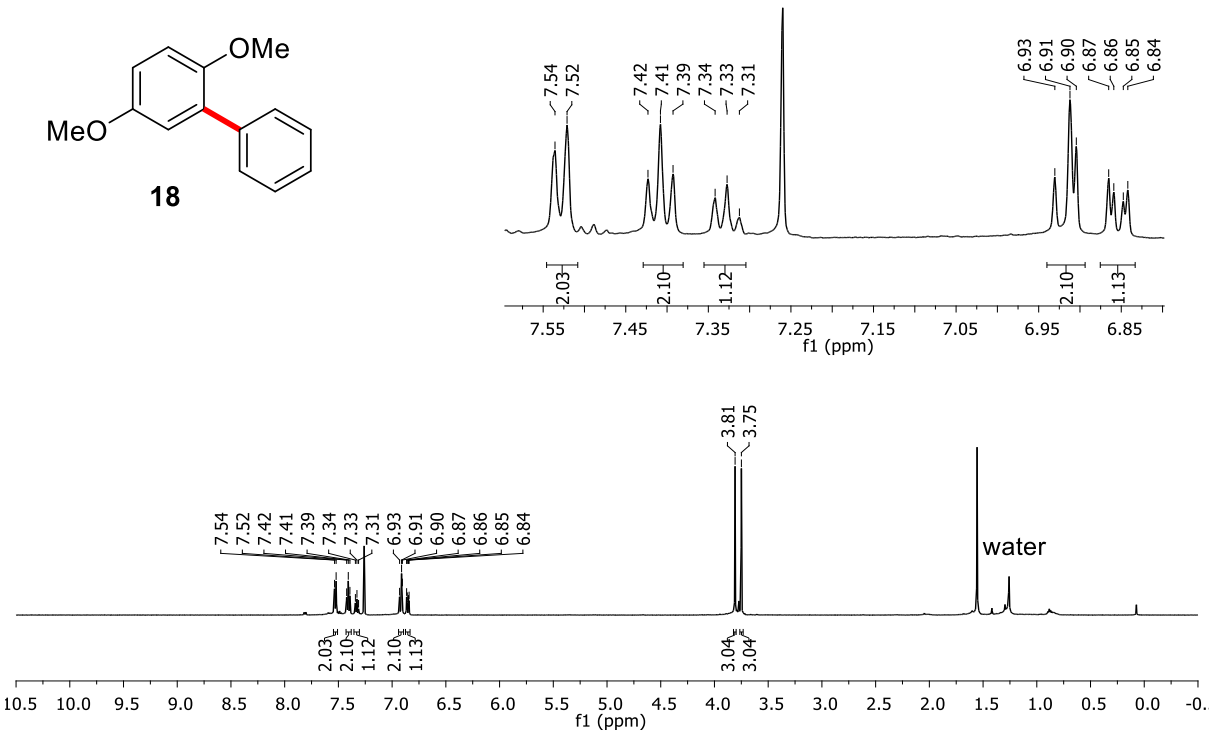

Fig. 60. [13]C NMR of 2,5-Dimethoxy-1,1'-biphenyl in $CDCl_3$ **(18)**[17].

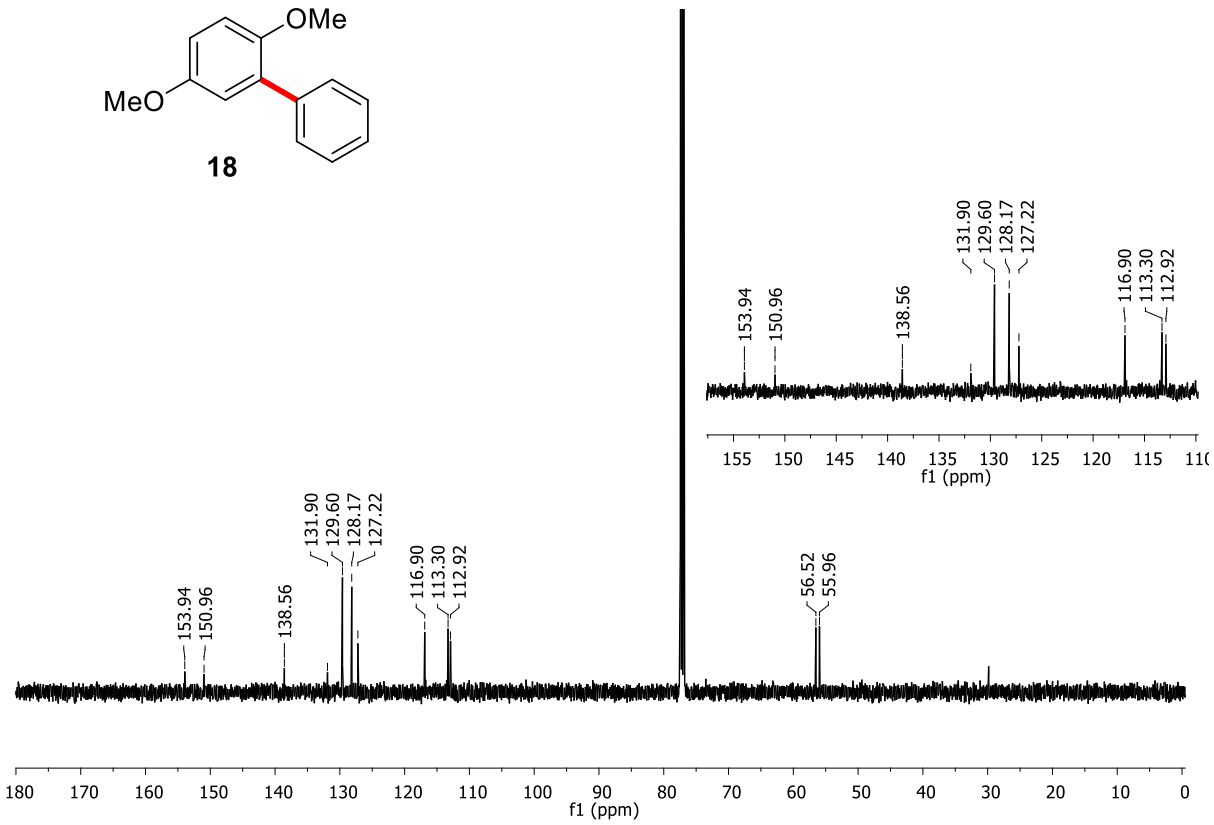

Fig. 61. [1] H NMR of 1-(4-(*tert*-Butyl)phenyl)naphthalene and 2-(4-(*tert*-Butyl)phenyl)naphthalene in $CDCl_3$ **(19)**[18].

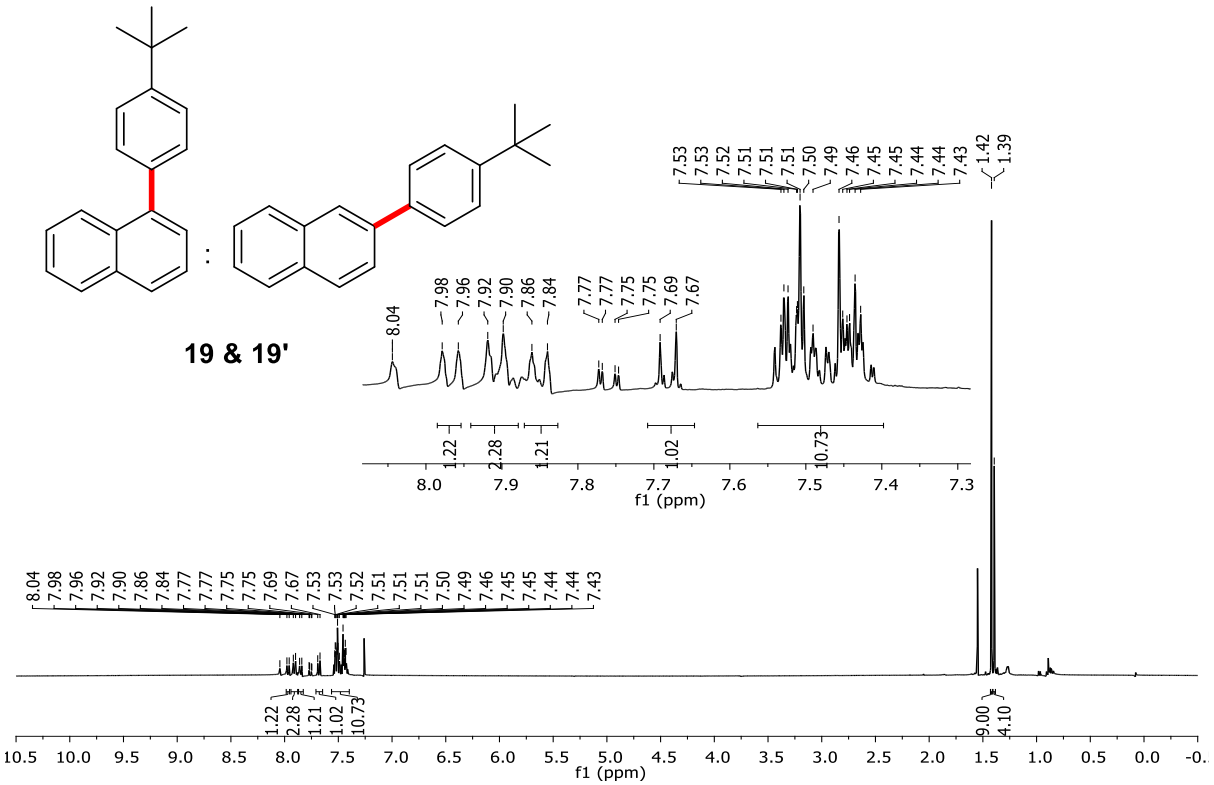

Fig. 62. [13]C NMR of 1-(4-(*tert*-Butyl)phenyl)naphthalene and 2-(4-(*tert*-Butyl)phenyl)naphthalene in $CDCl_3$ **(19)**[18].

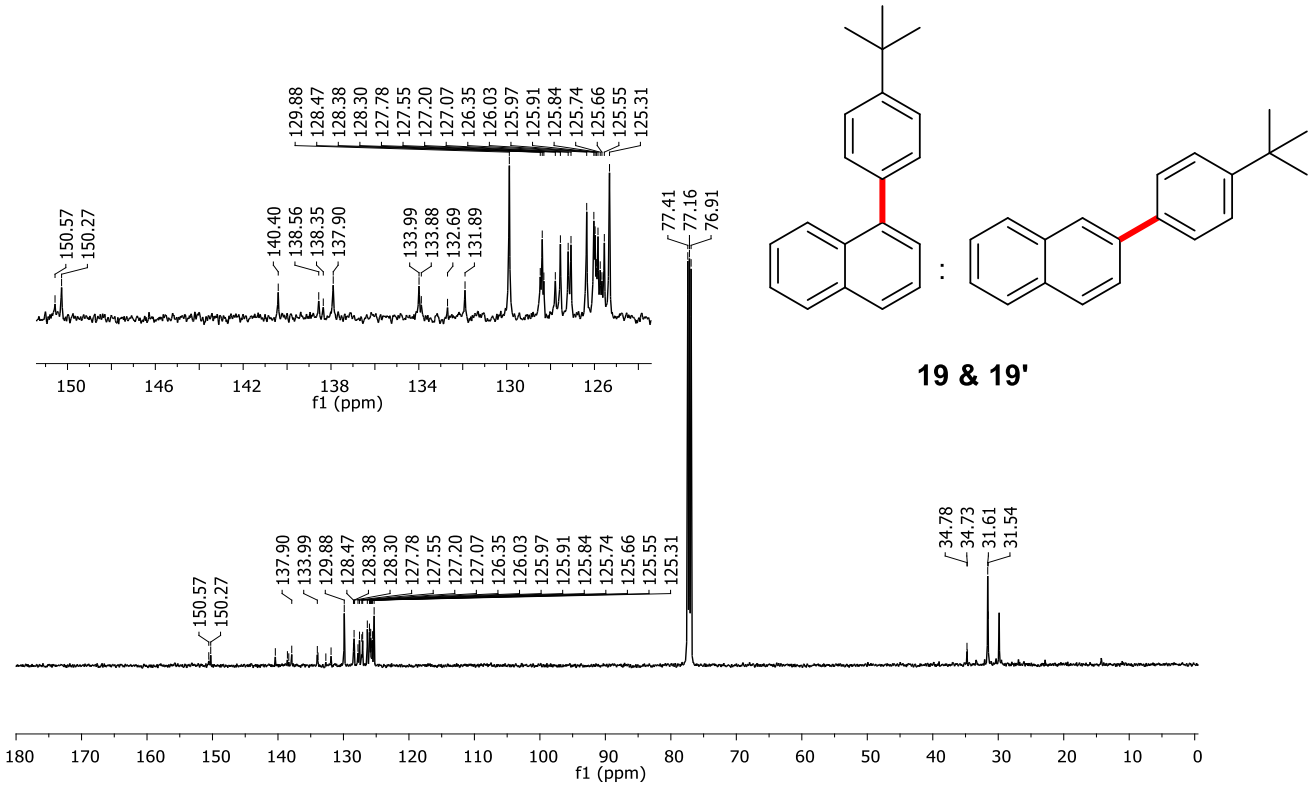

Fig. 63. [1] H NMR of 1-(4-Isopropylphenyl)naphthalene & 2-(4-Isopropylphenyl)naphthalene in $CDCl_3$ **(20)**[19].

(40)

^{1}H NMR:

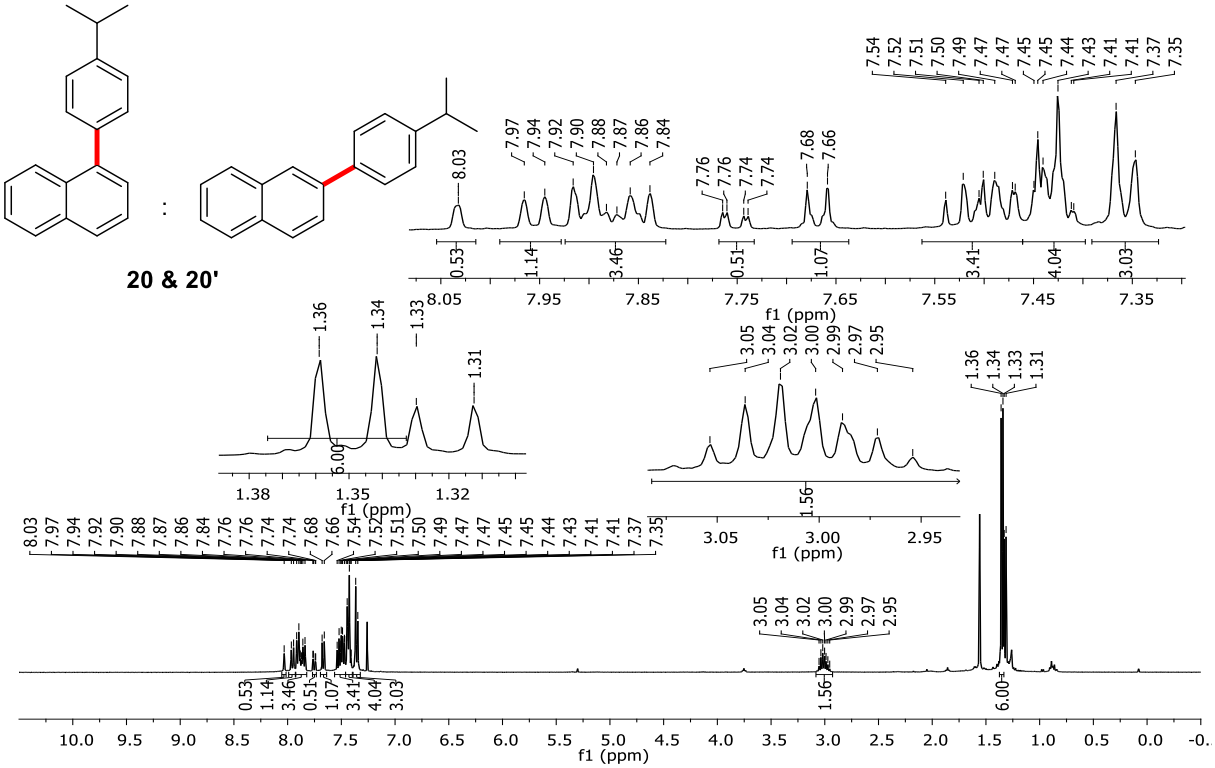

Fig. 64. ^{13}C NMR of 1-(4-Isopropylphenyl)naphthalene & 2-(4-Isopropylphenyl)naphthalene in $CDCl_3$ **(20)**[19].

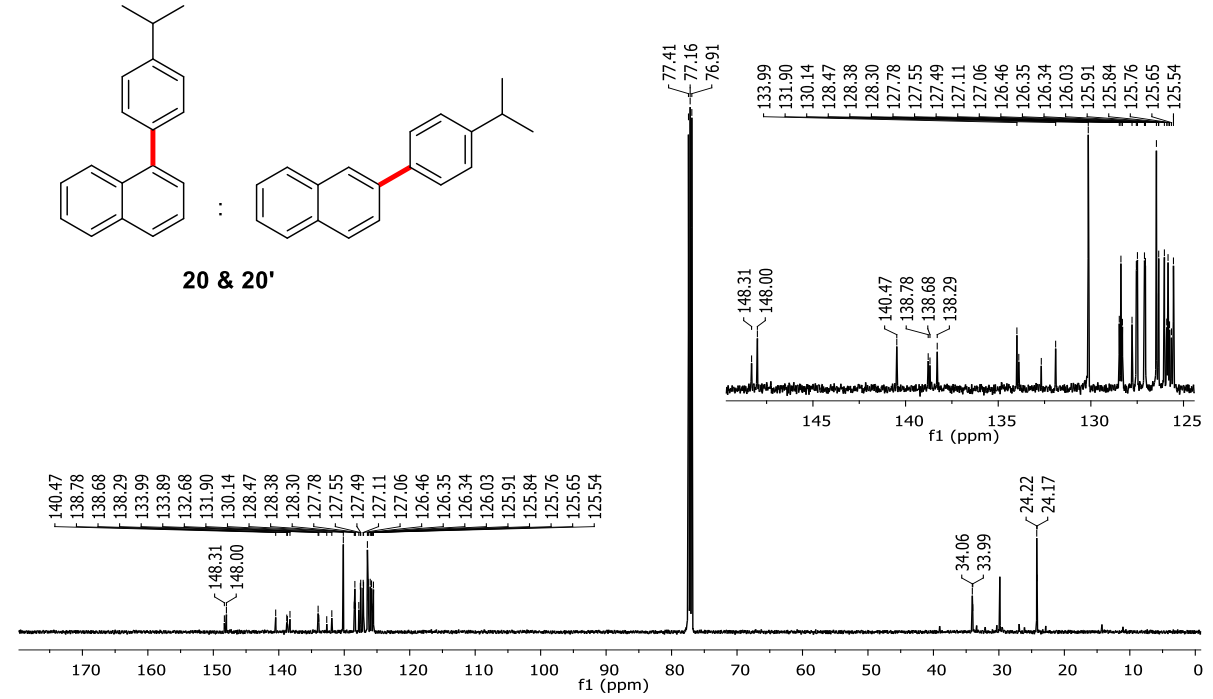

Fig. 65. [1] H NMR of 1-Phenylnaphthalene and 2-Phenylnaphthalene in $CDCl_3$ **(21)**[20].

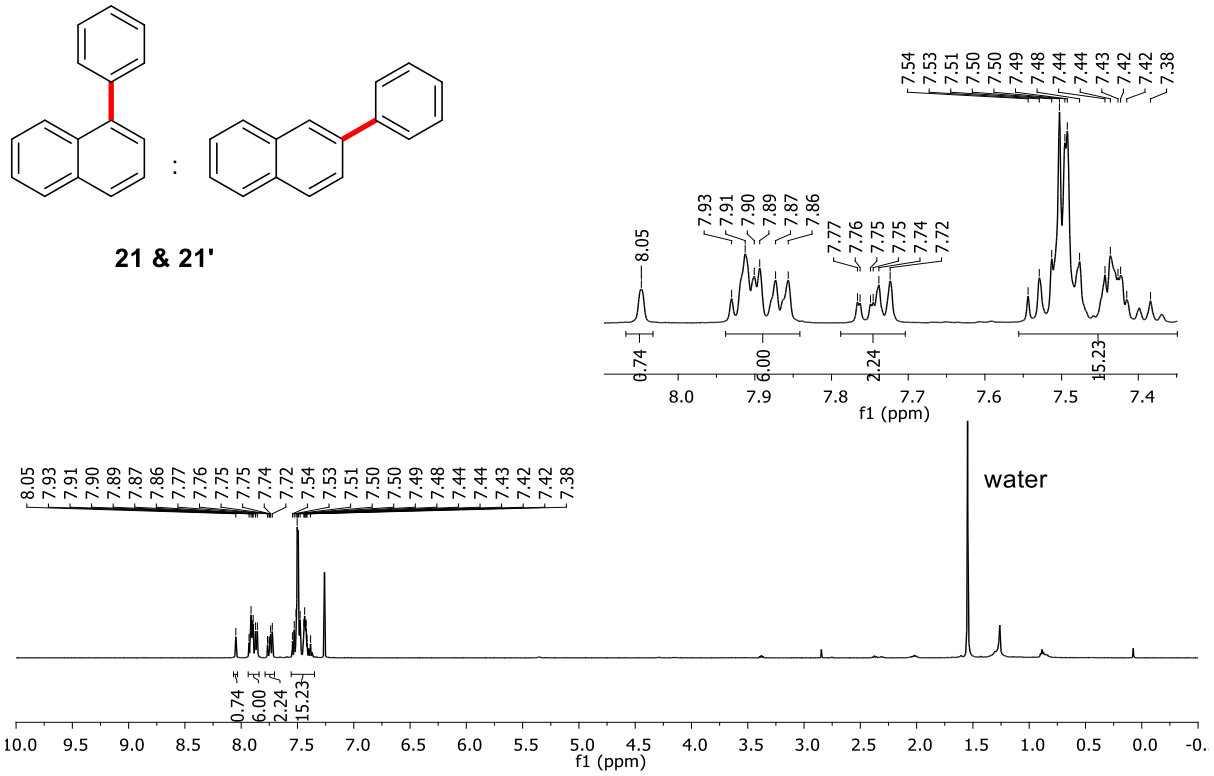

Fig. 66. [13]C NMR of 1-Phenylnaphthalene and 2-Phenylnaphthalene in $CDCl_3$ **(21)**[20].

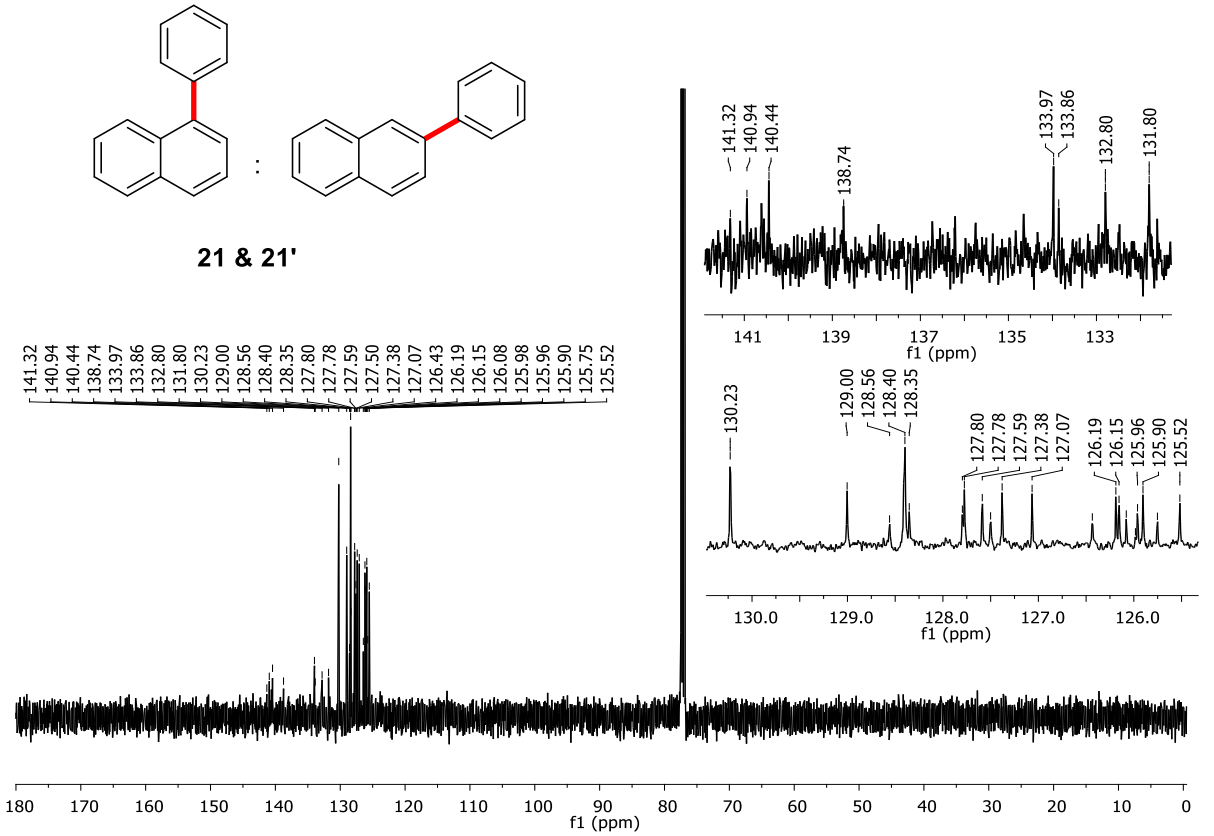

Fig. 67. [1] H NMR of 1-(4-Fluorophenyl)naphthalene and 2-(4-Fluorophenyl)naphthalene in $CDCl_3$ **(22)**[18].

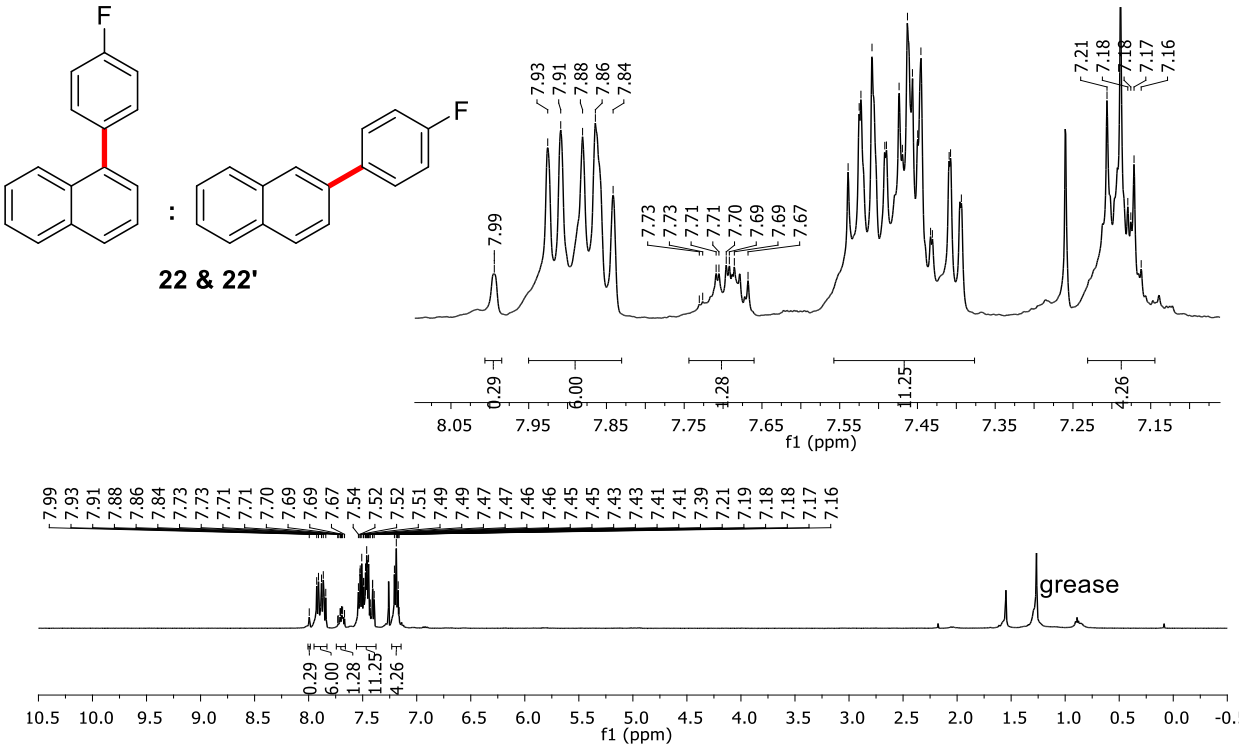

Fig. 68. [13]C NMR of 1-(4-Fluorophenyl)naphthalene and 2-(4-Fluorophenyl)naphthalene in $CDCl_3$ **(22)**[18].

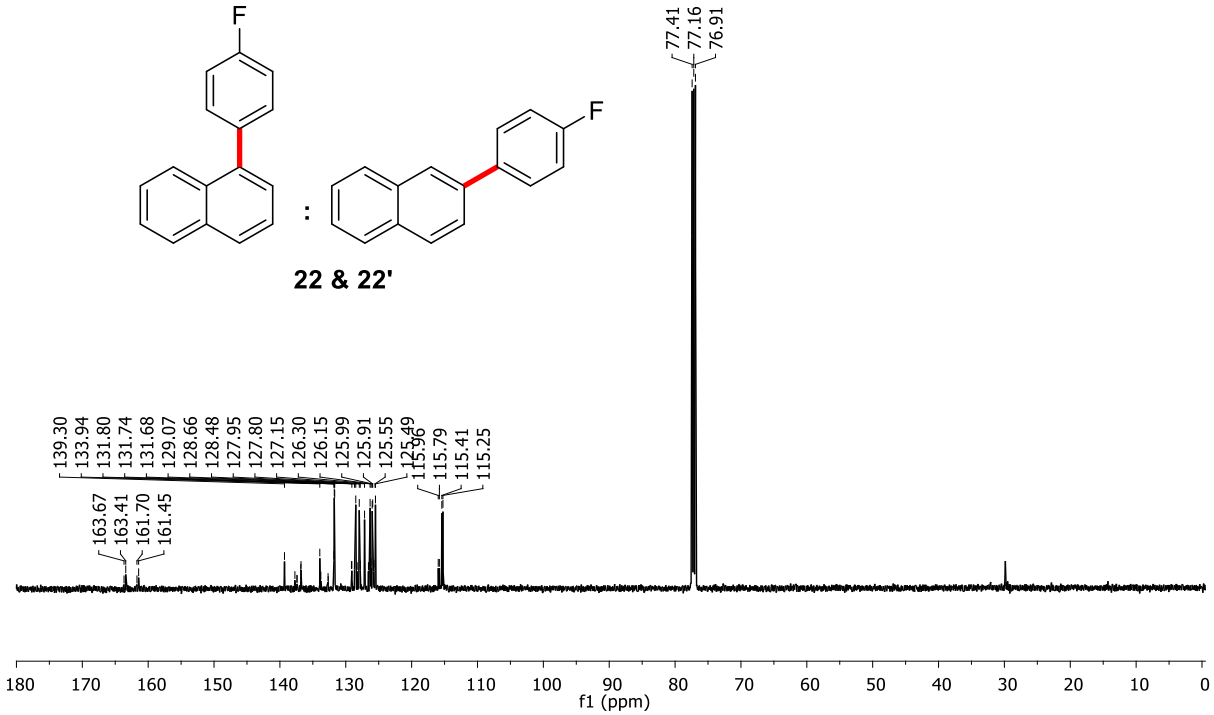

Fig. 69. [1] H NMR of 1-(4-(Trifluoromethyl)phenyl)naphthalene and 2-(4-(Trifluoromethyl)phenyl)naphthalene in $CDCl_3$ **(23)**[22].

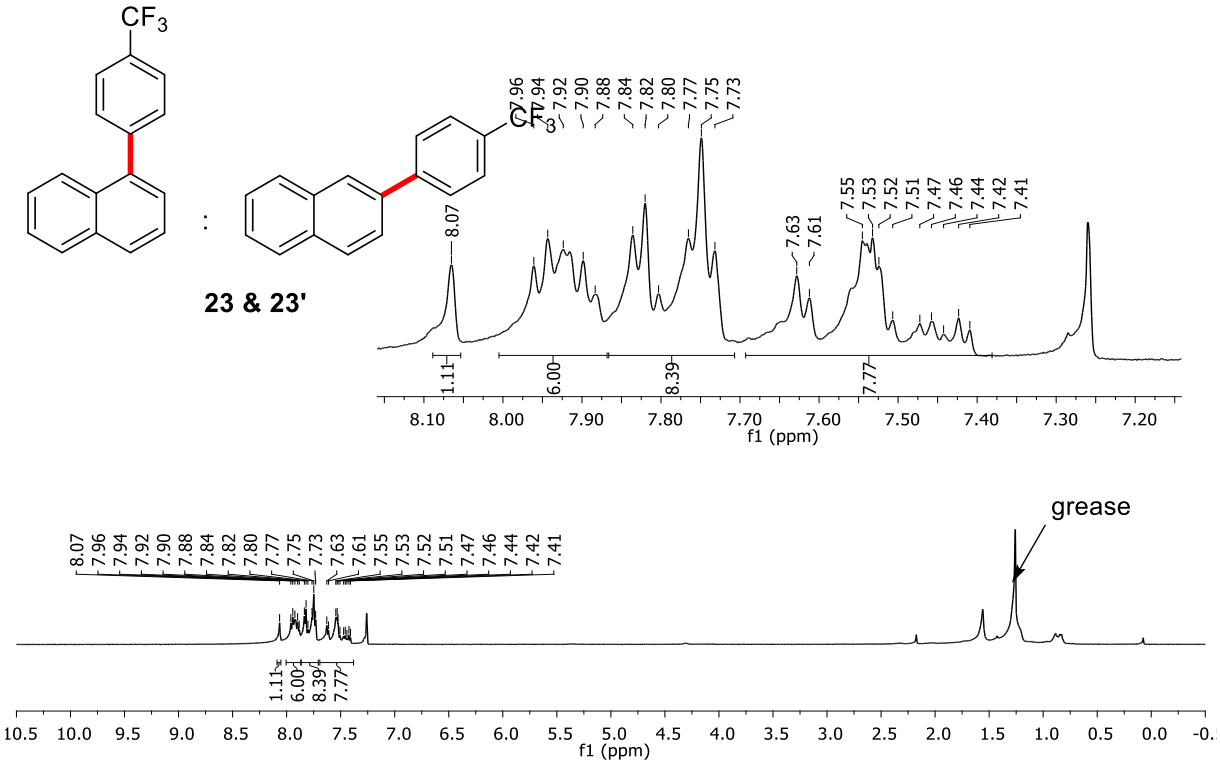

Fig. 70. [13]C NMR of 1-(4-(Trifluoromethyl)phenyl)naphthalene and 2-(4-(Trifluoromethyl)phenyl)naphthalene in $CDCl_3$ **(23)**[22].

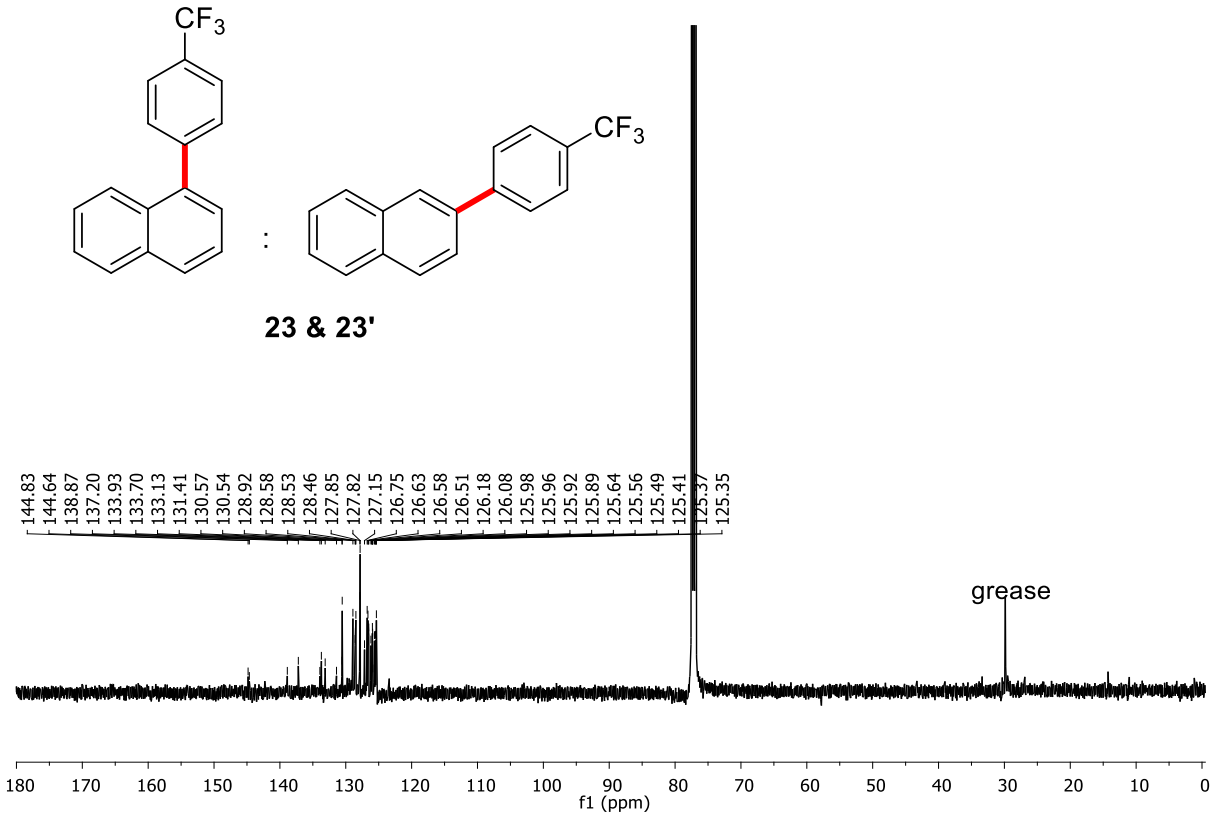

Fig. 71. [1] H NMR of 1-(3, 5-dimethylphenyl)naphthalene and 2-(3,5-dimethylphenyl)naphthalene in $CDCl_3$ **(24)**[23].

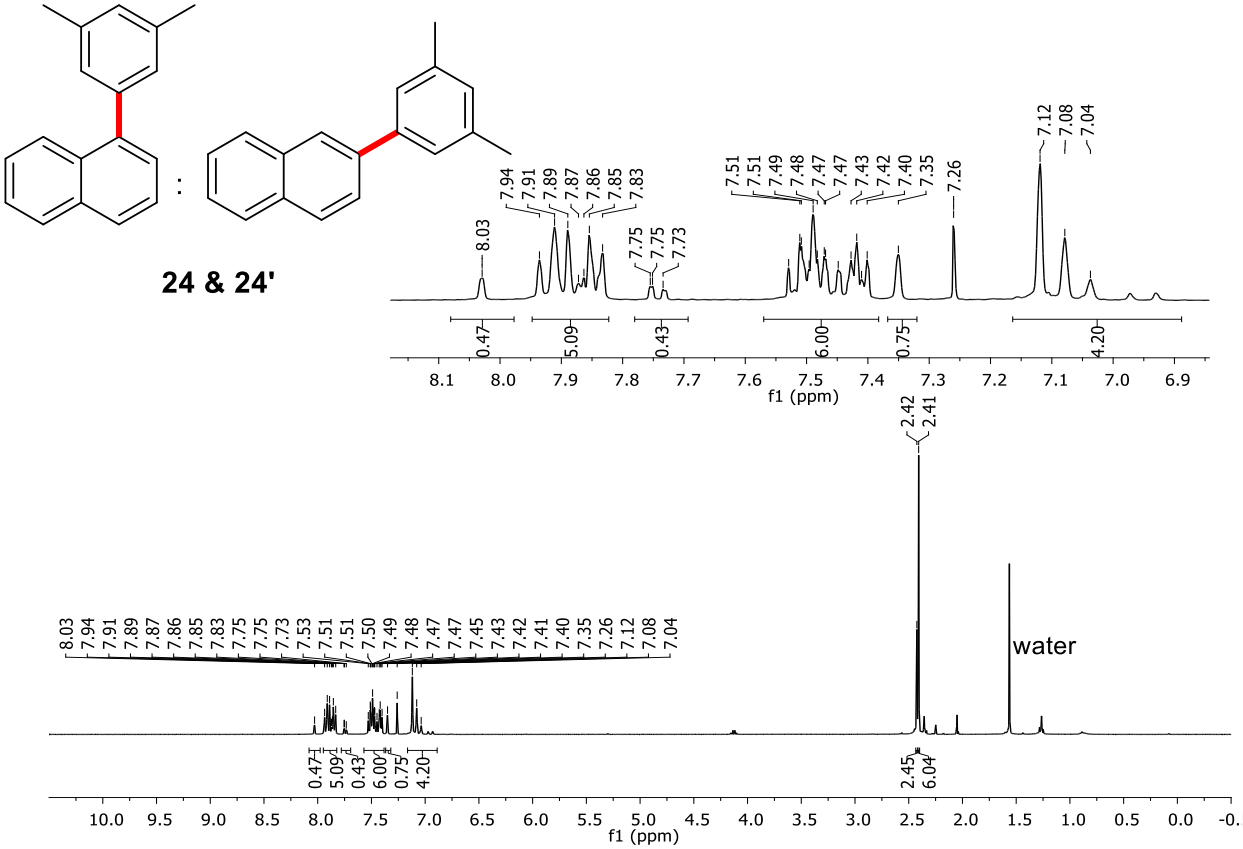

Fig. 72. [13]C NMR of 1-(3, 5-dimethylphenyl)naphthalene and 2-(3,5-dimethylphenyl)naphthalene in $CDCl_3$ **(24)**[23].

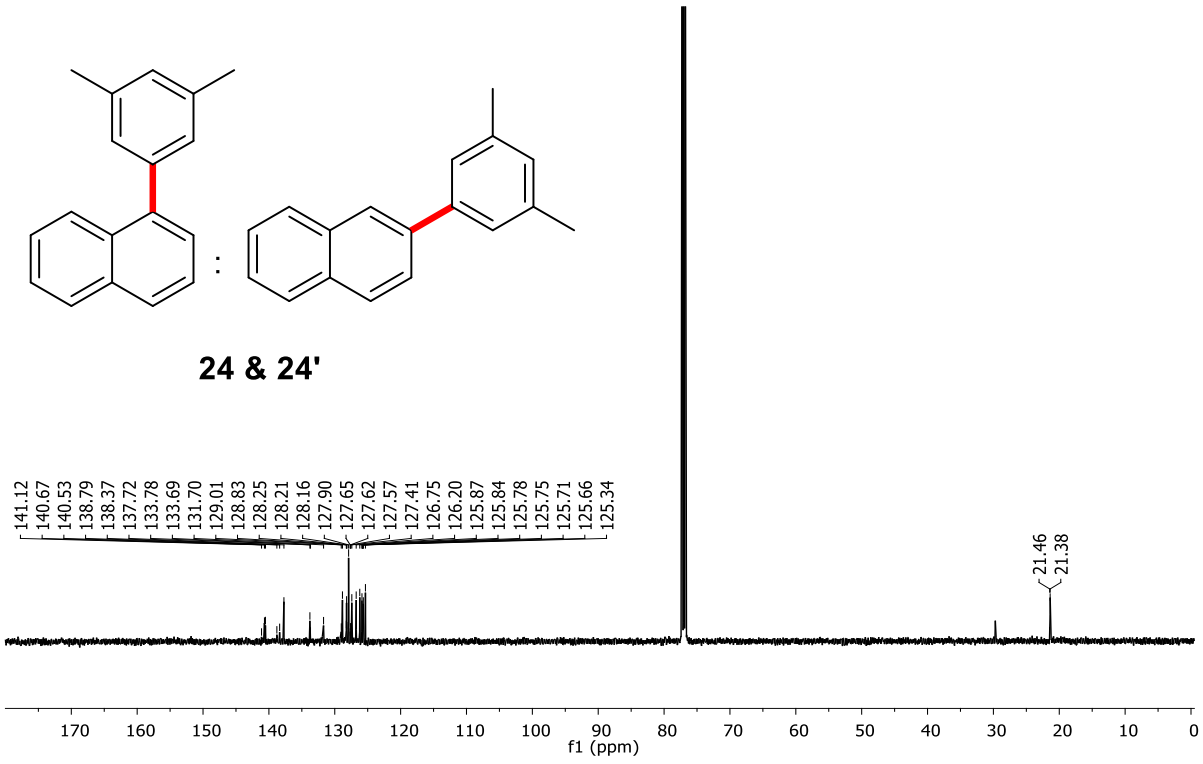

Fig. 73. [1] H NMR of 1-Phenylpyrene in $CDCl_3$ **(25)**[24].

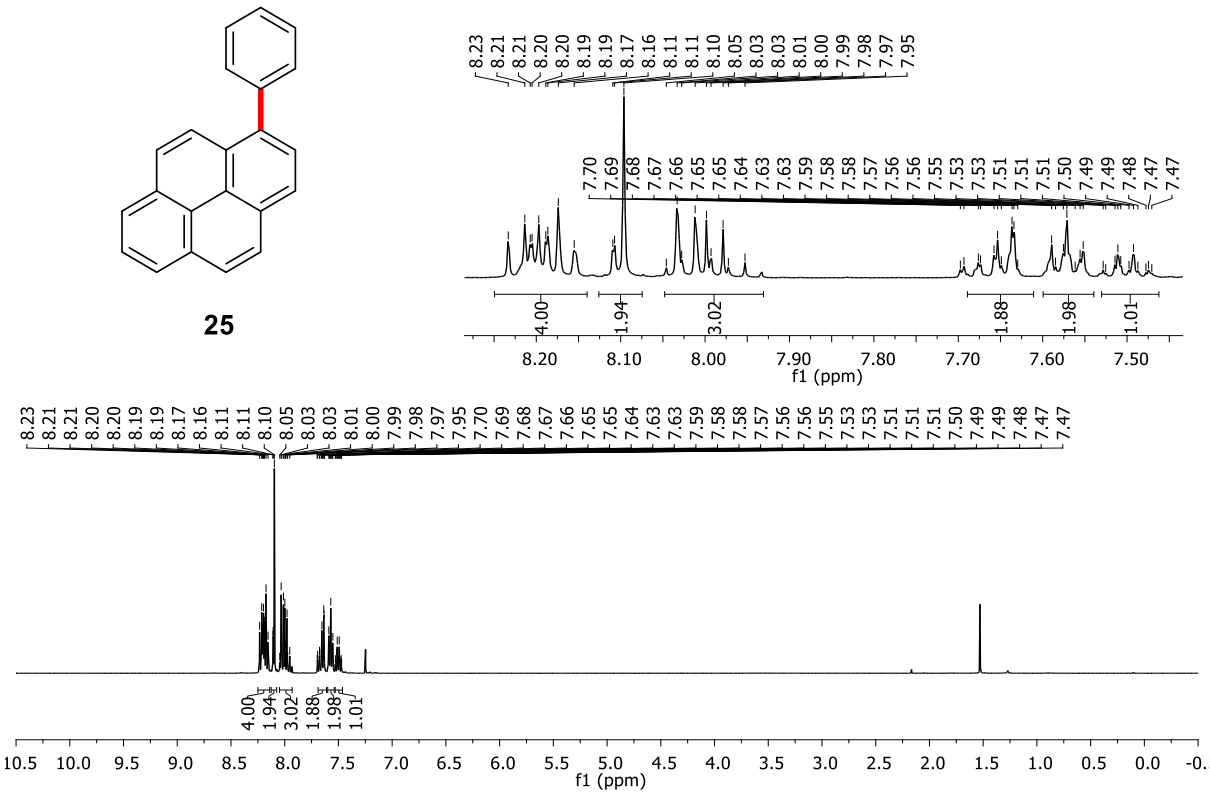

Fig. 74. [13]C NMR of 1-Phenylpyrene in $CDCl_3$ **(25)**[24].

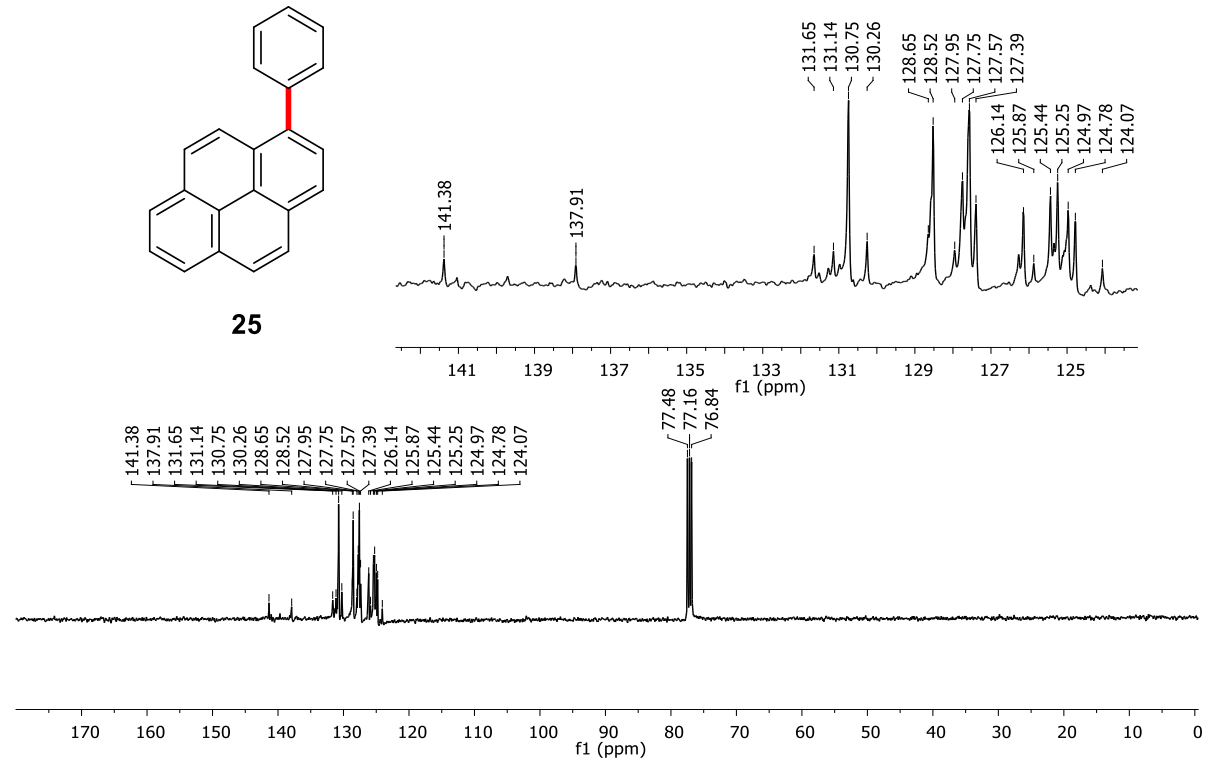

Fig. 75. [1] H NMR of 1-(4-(*tert*-Butyl)phenyl)pyrene in $CDCl_3$ **(26)**.

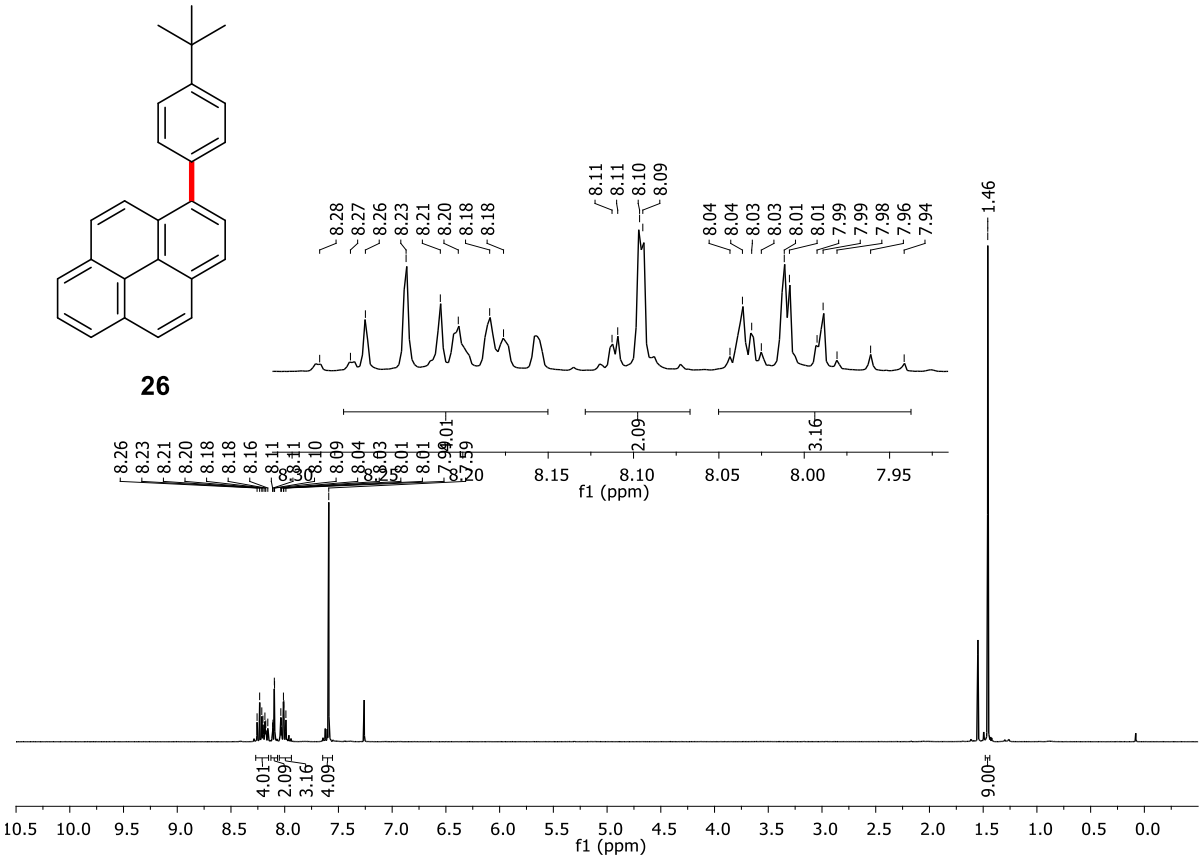

Fig. 76. [13]C NMR of 1-(4-(*tert*-Butyl)phenyl)pyrene in $CDCl_3$ **(26)**

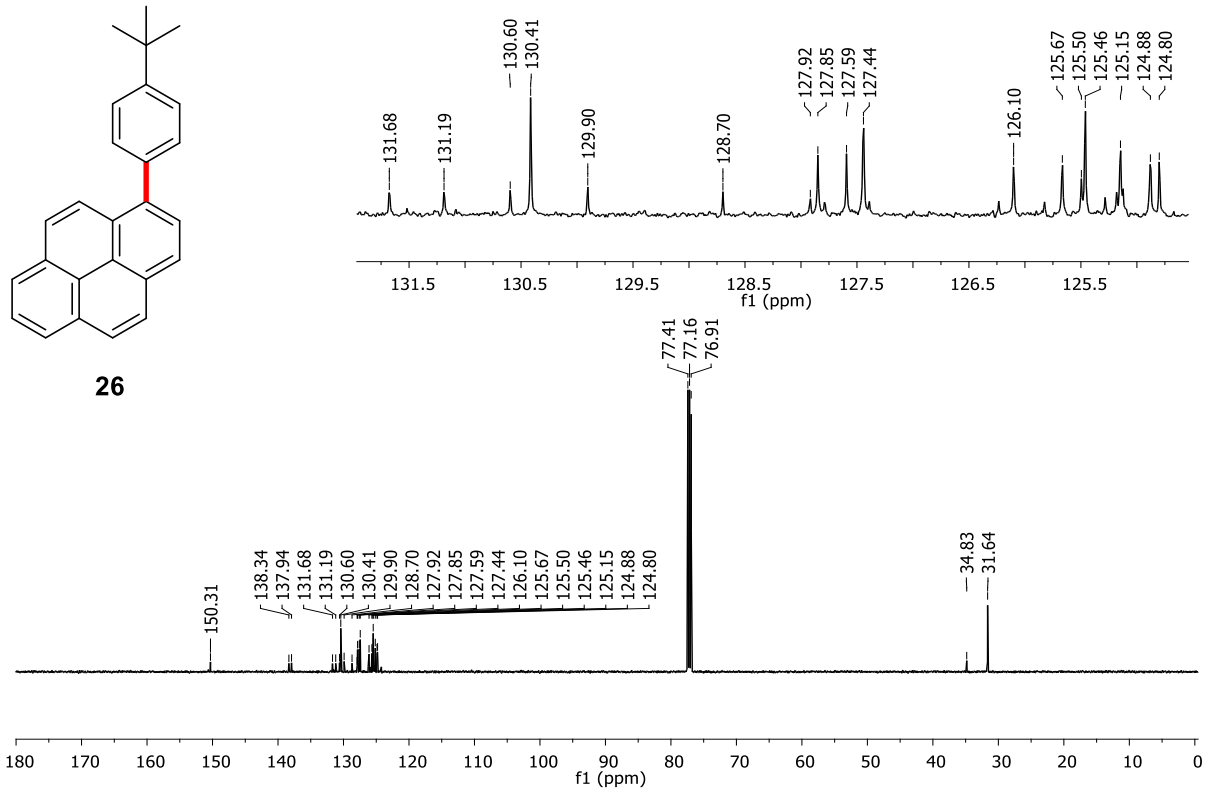

Fig. 77. [1] H NMR of 1-(3,5-Dimethylphenyl)pyrene in $CDCl_3$ **(27)**[25].

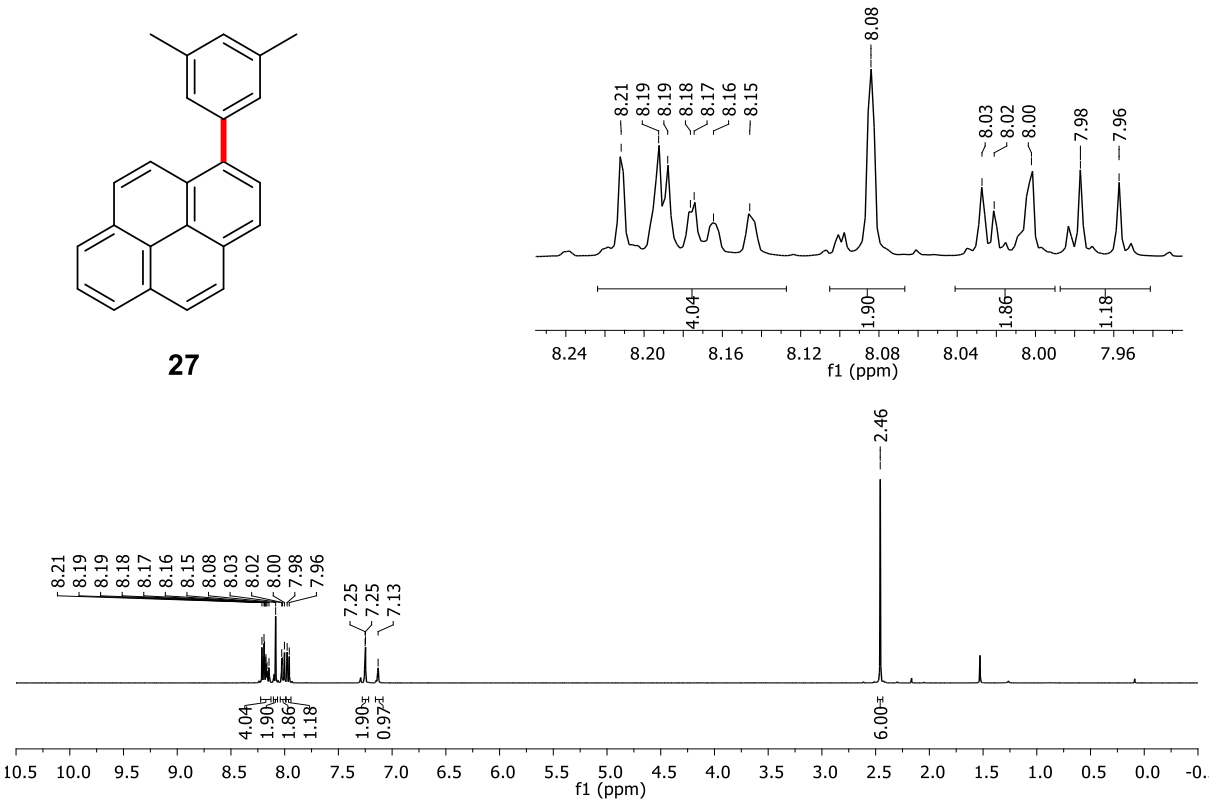

Fig. 78. [13]C NMR of 1-(3,5-Dimethylphenyl)pyrene in $CDCl_3$ **(27)**[25].

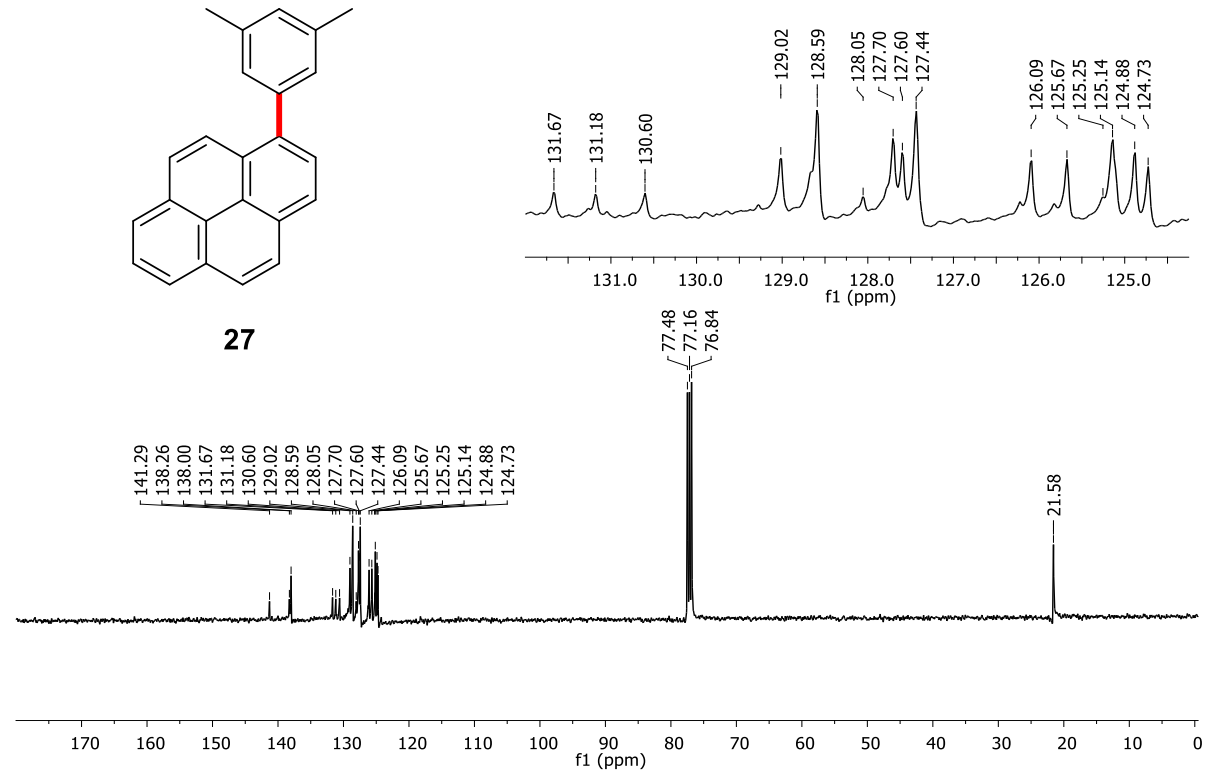

Fig. 79. ^{1}H NMR of Styrylbenzonitrile in $CDCl_3$ **(28)**[26].

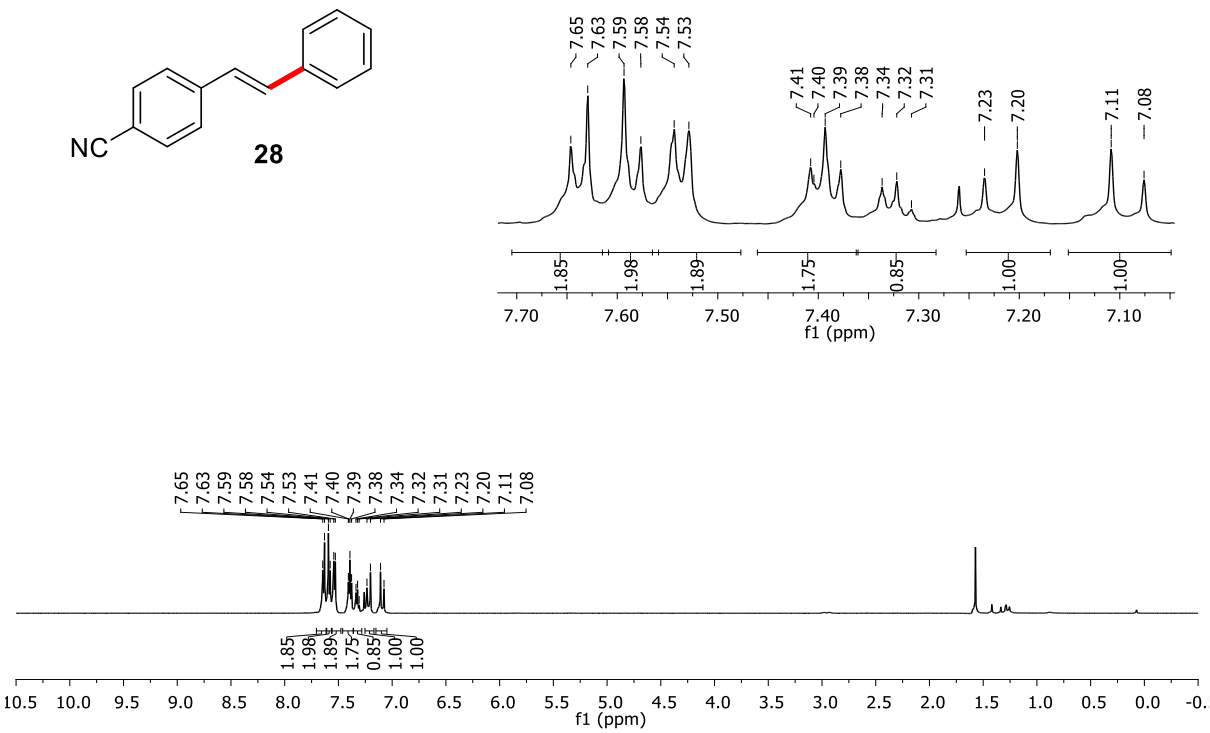

Fig. 80. ^{13}C NMR of Styrylbenzonitrile in $CDCl_3$ **(28)**[26].

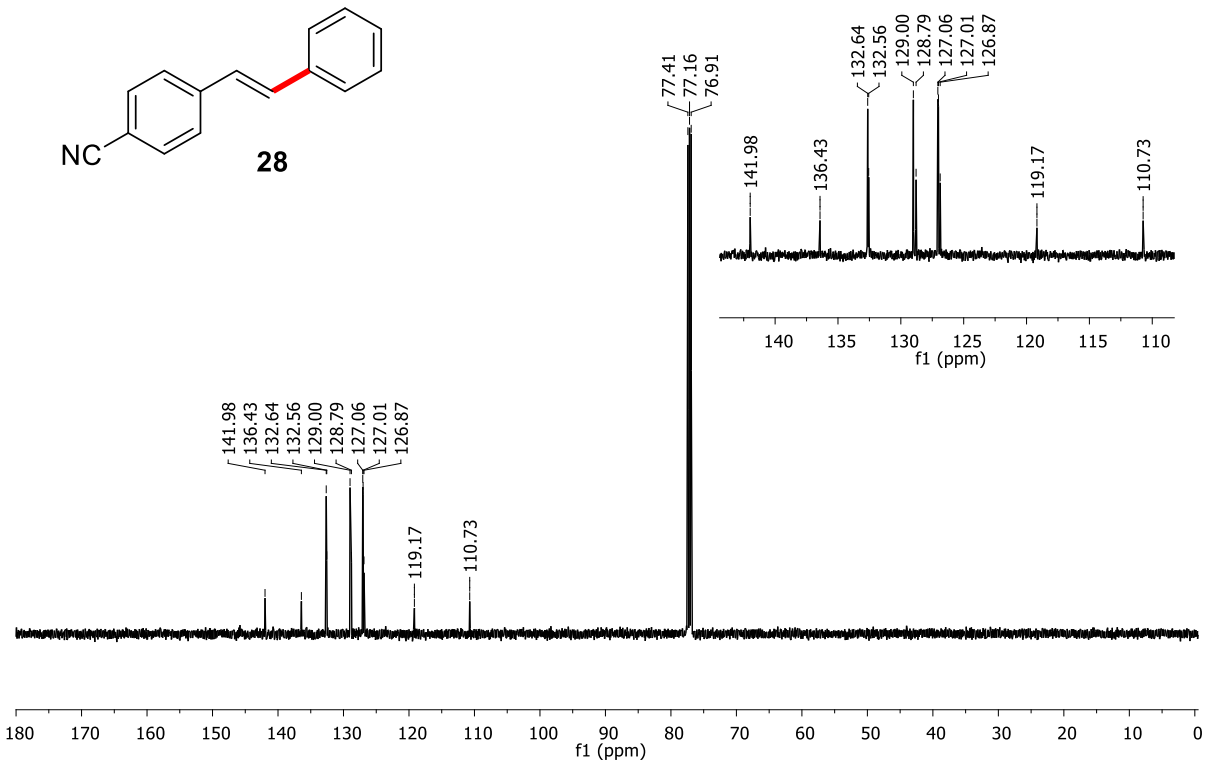

Fig. 81. [1] H NMR of (*E*)-1-Nitro-4-styrylbenzene in $CDCl_3$ **(29)**[27].

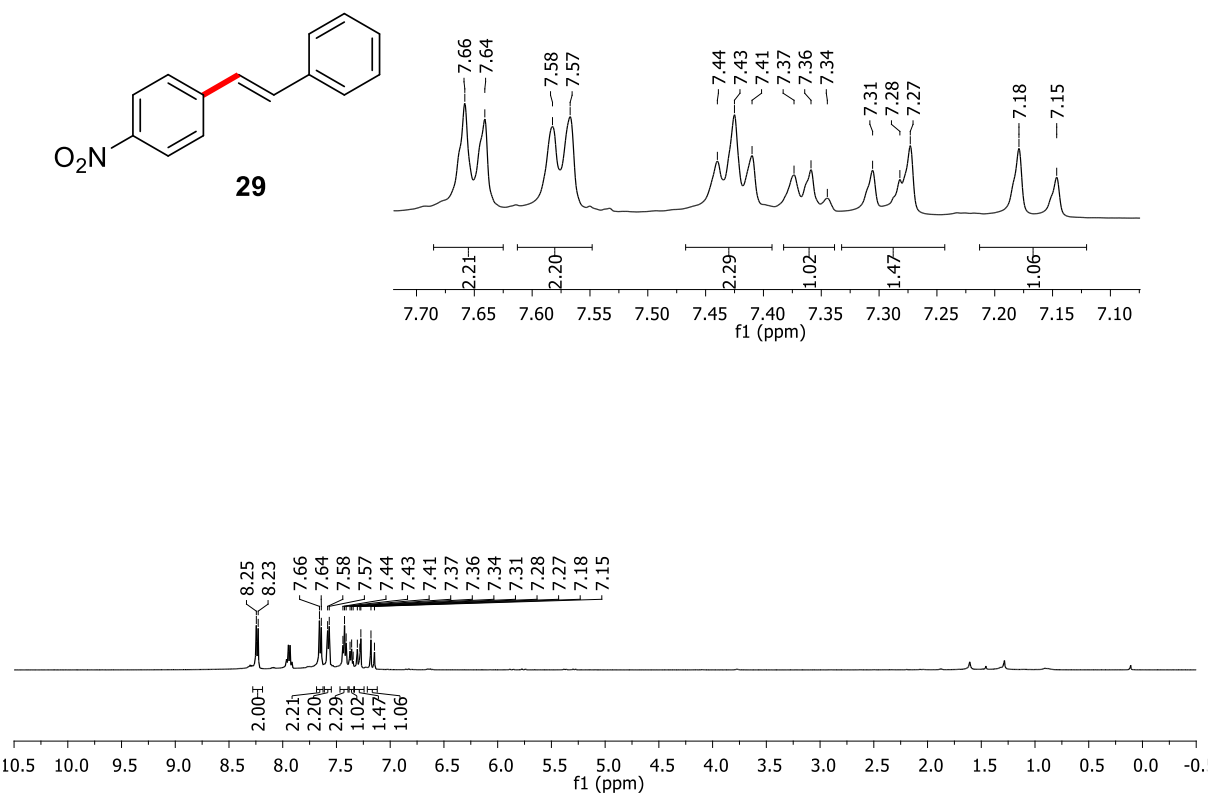

Fig. 82. [13]C NMR of (*E*)-1-Nitro-4-styrylbenzene in $CDCl_3$ **(29)**[27].

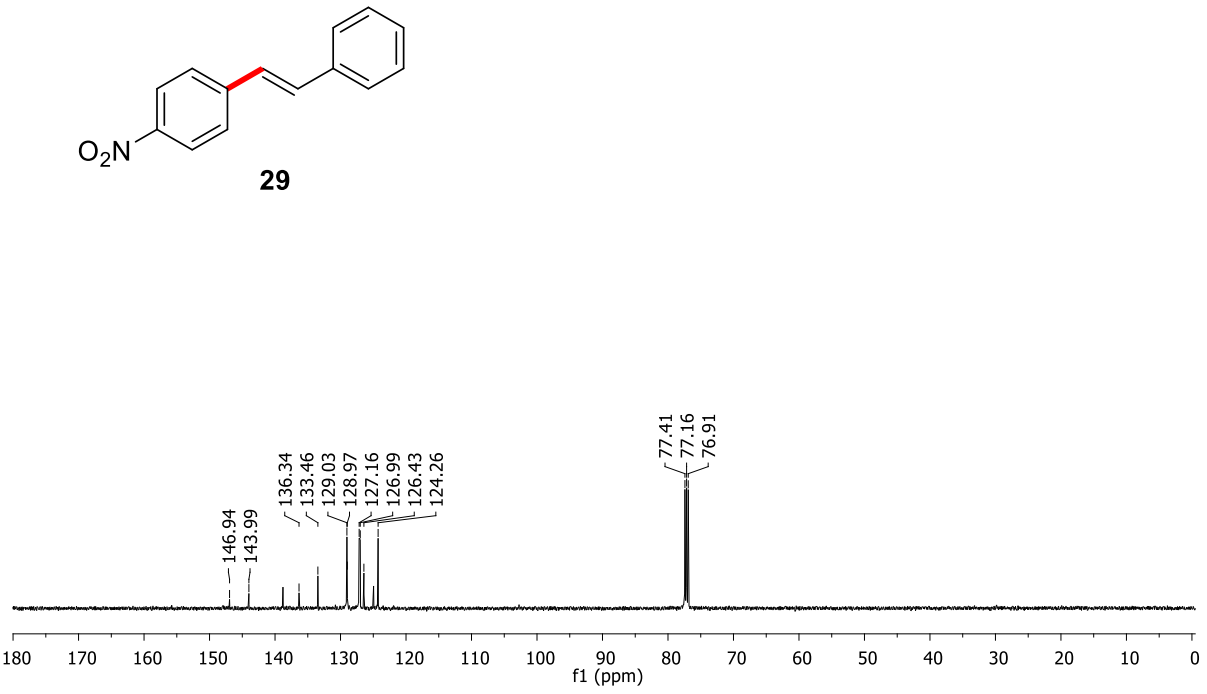

Fig. 83. [1] H NMR of (*E*)-4-(4-Methylstyryl)benzonitrile in $CDCl_3$ **(30)**[28].

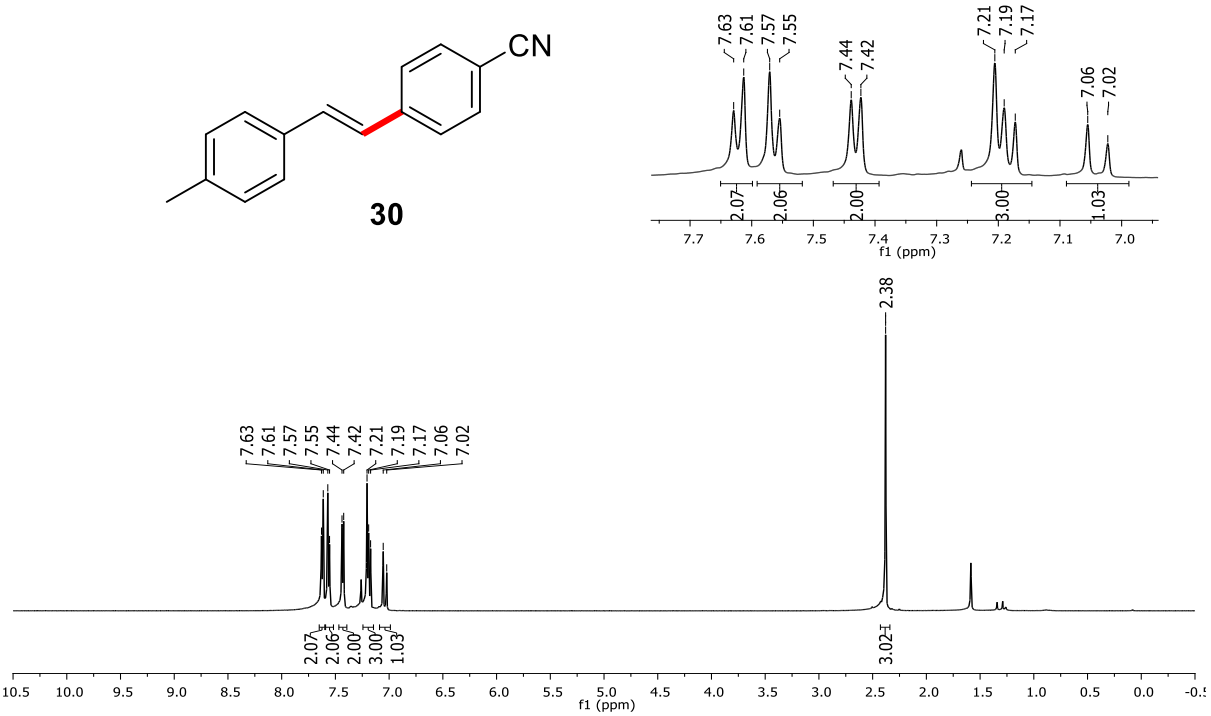

Fig. 84. [13]C NMR of (*E*)-4-(4-Methylstyryl)benzonitrile in $CDCl_3$ **(30)**[28].

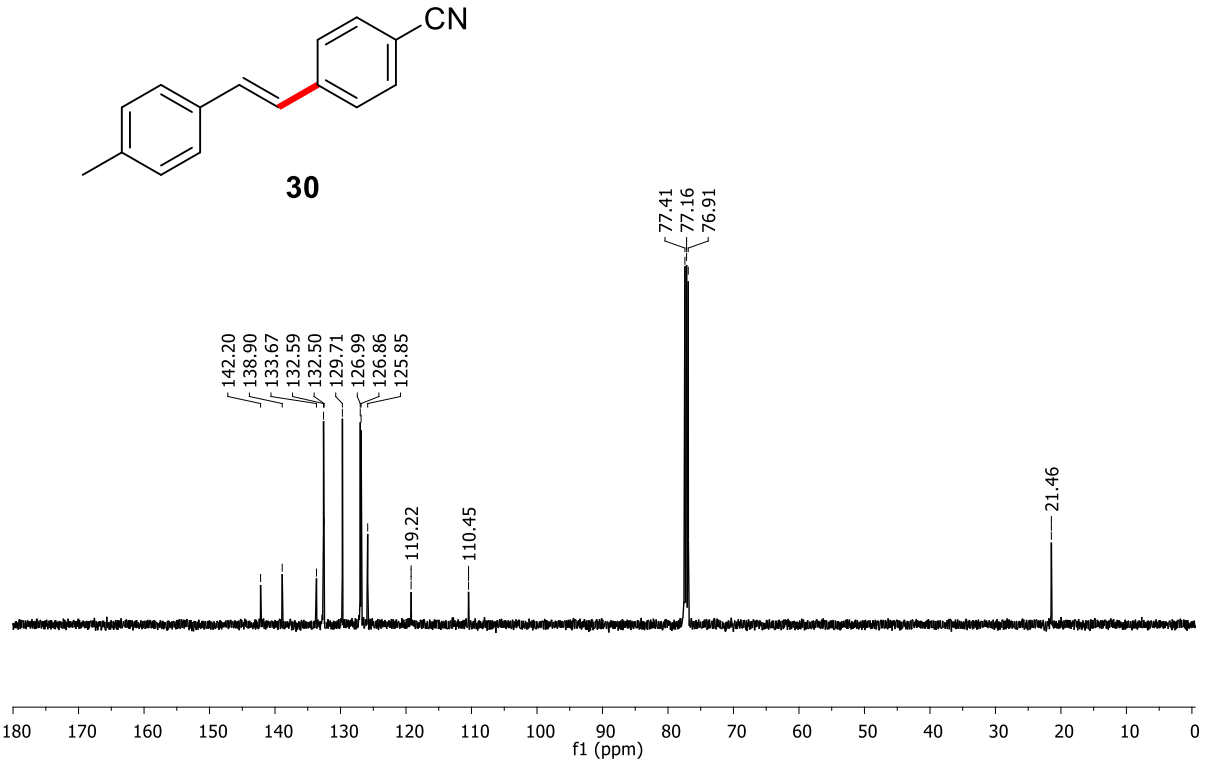

Fig. 85. [1] H NMR of (*E*)-4-(4-(*tert*-Butyl)styryl)benzonitrile in $CDCl_3$ **(31)**[29].

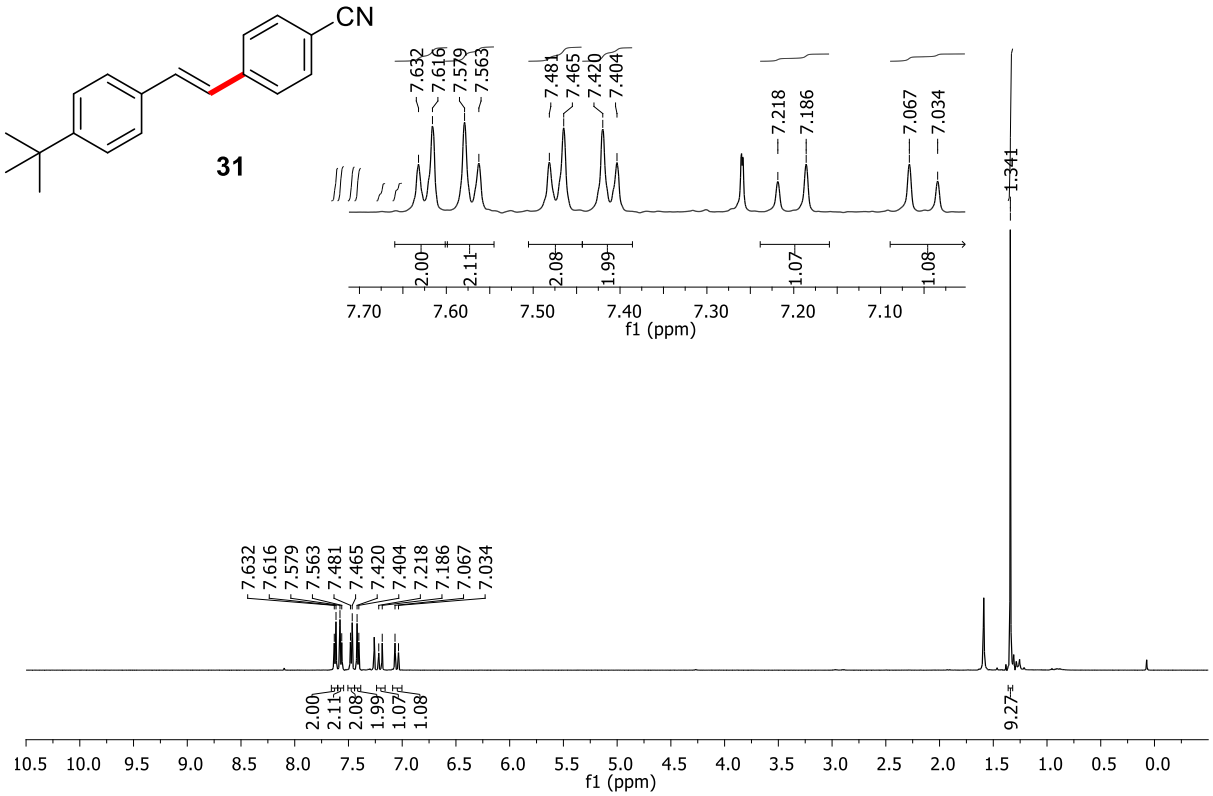

Fig. 86. [13]C NMR of (*E*)-4-(4-(*tert*-Butyl)styryl)benzonitrile in $CDCl_3$ **(31)**[29].

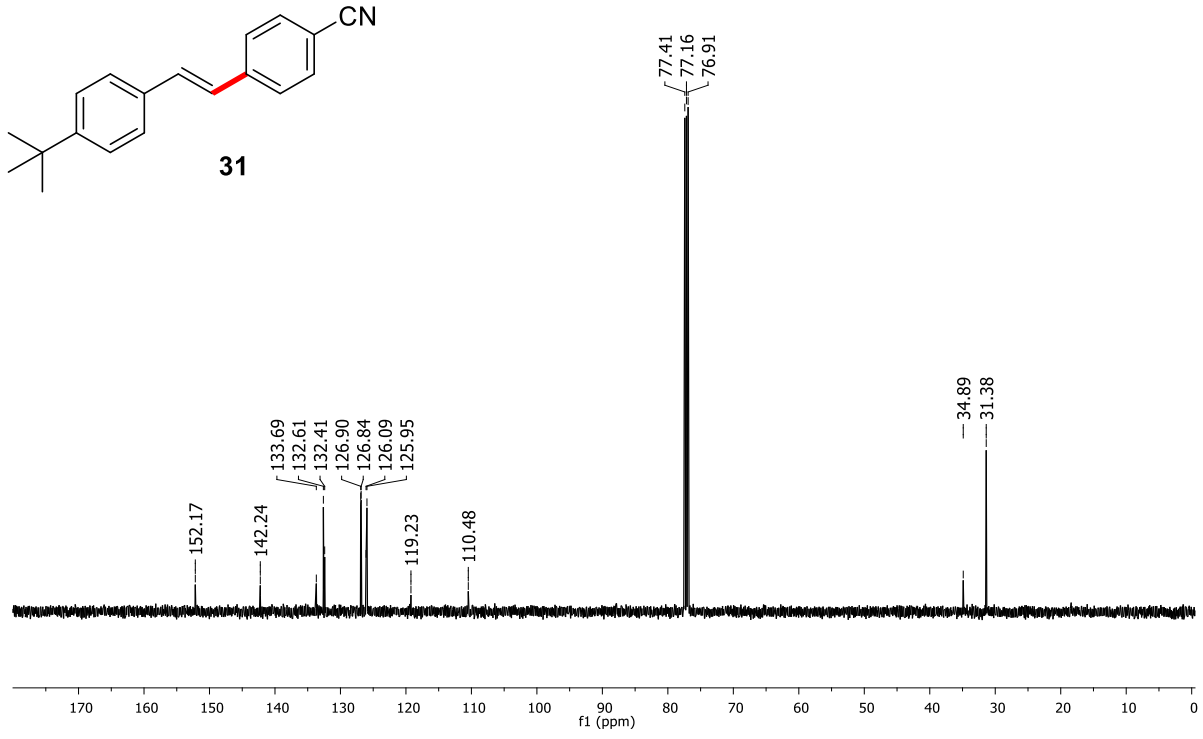

Fig. 87. [1] H NMR of (*E*)-1-Bromo-4-(4-nitrostyryl)benzene in $CDCl_3$ **(32)**[30].

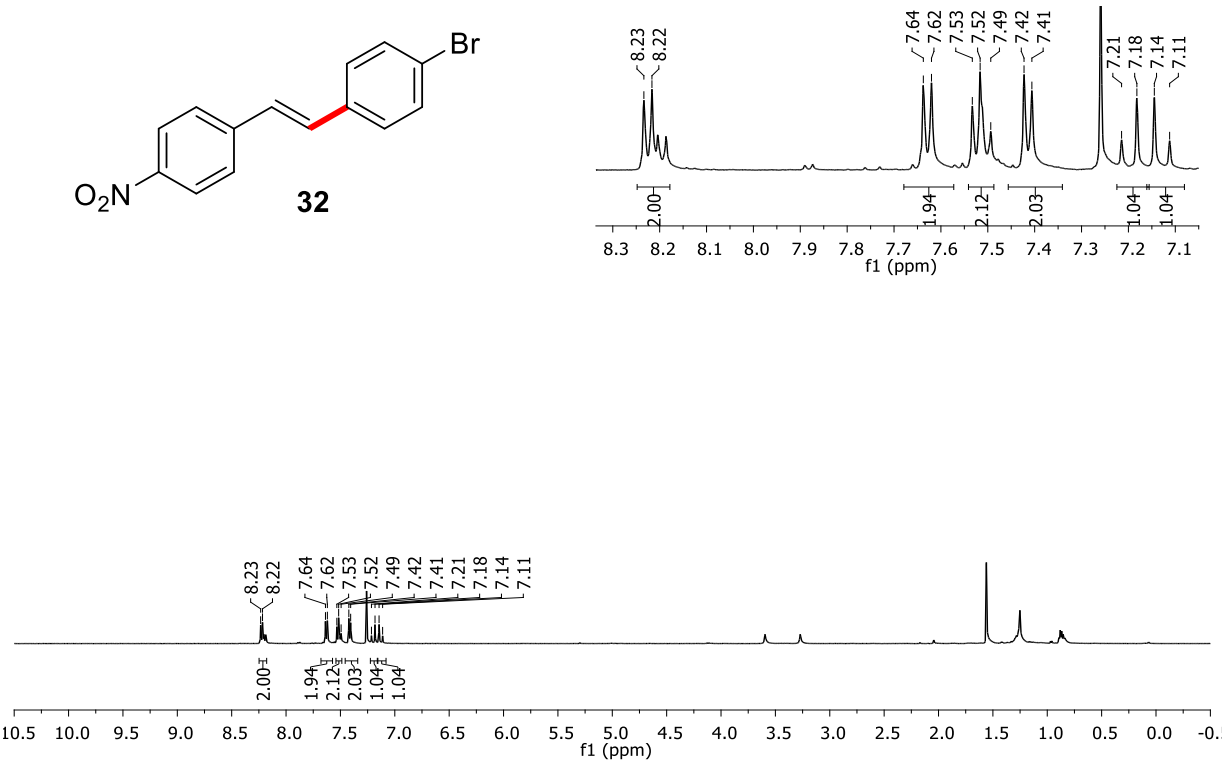

Fig. 88. [13]C NMR of (*E*)-1-Bromo-4-(4-nitrostyryl)benzene in $CDCl_3$ **(32)**[30].

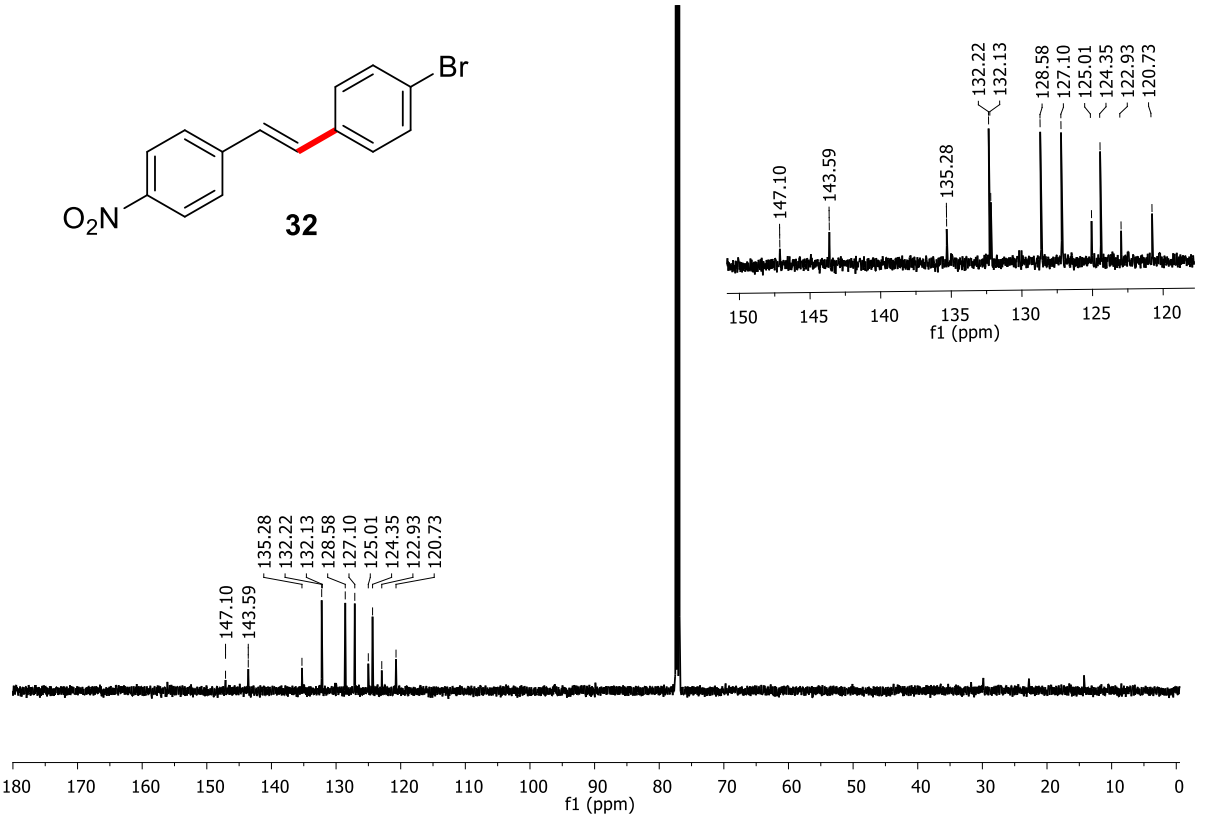

Fig. 89. [1] H NMR of (*E*)-4-(4-Methoxystyryl)benzonitrile in $CDCl_3$ **(33)**[31].

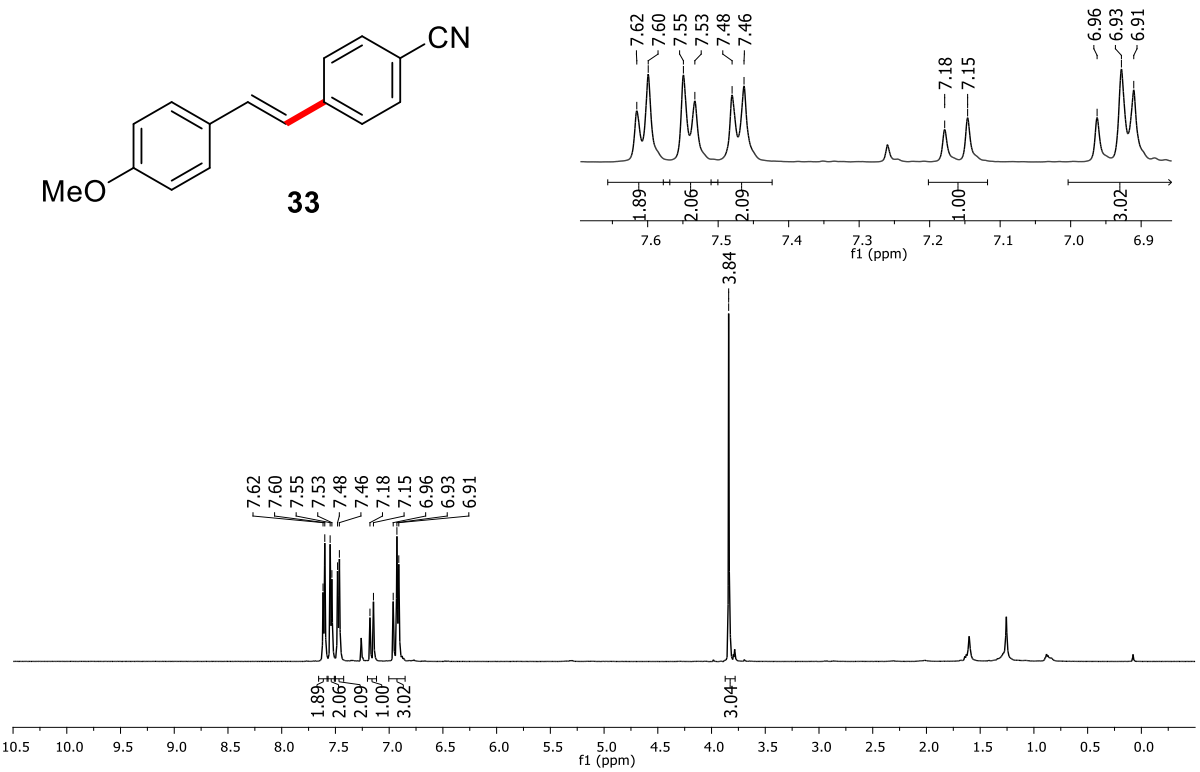

Fig. 90. [13]C NMR of (*E*)-4-(4-Methoxystyryl)benzonitrile in $CDCl_3$ **(33)**[31].

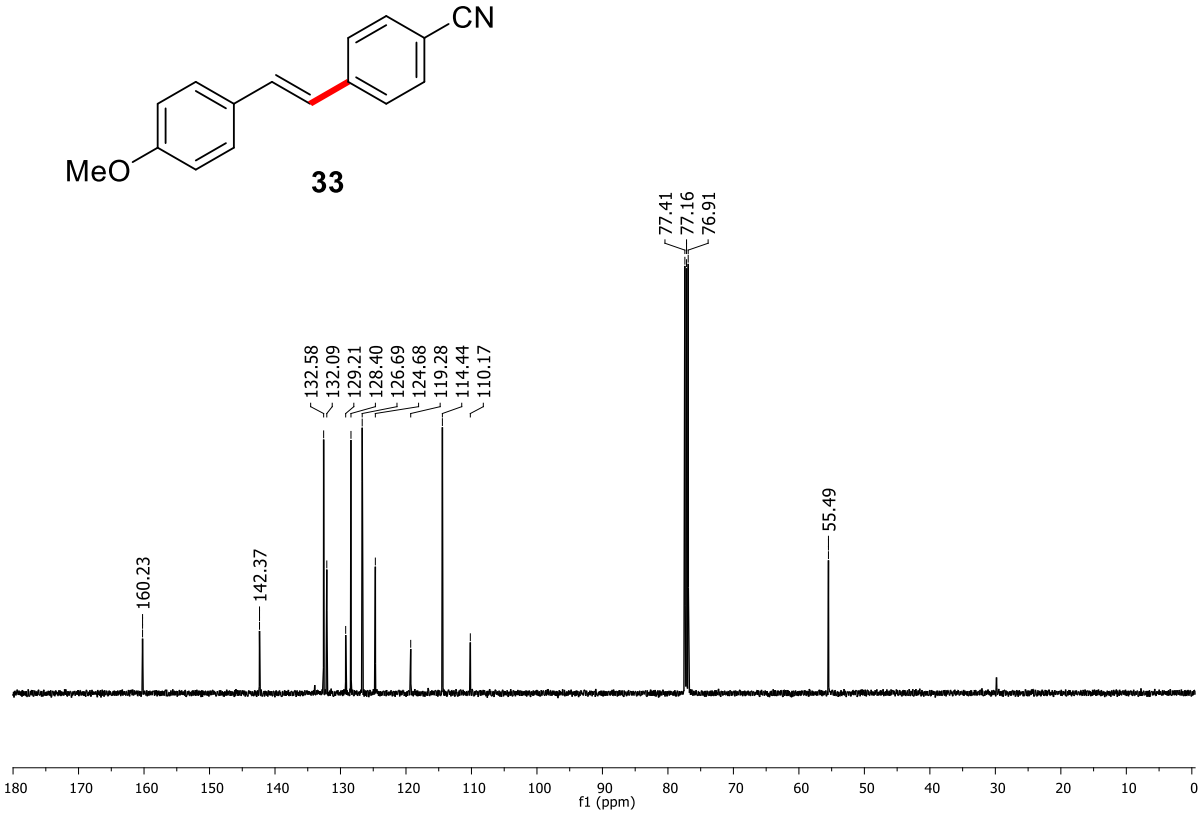

Fig. 91. [1] H NMR of (*E*)-1-Chloro-4-(4-nitrostyryl)benzene in $CDCl_3$ **(34)**[30].

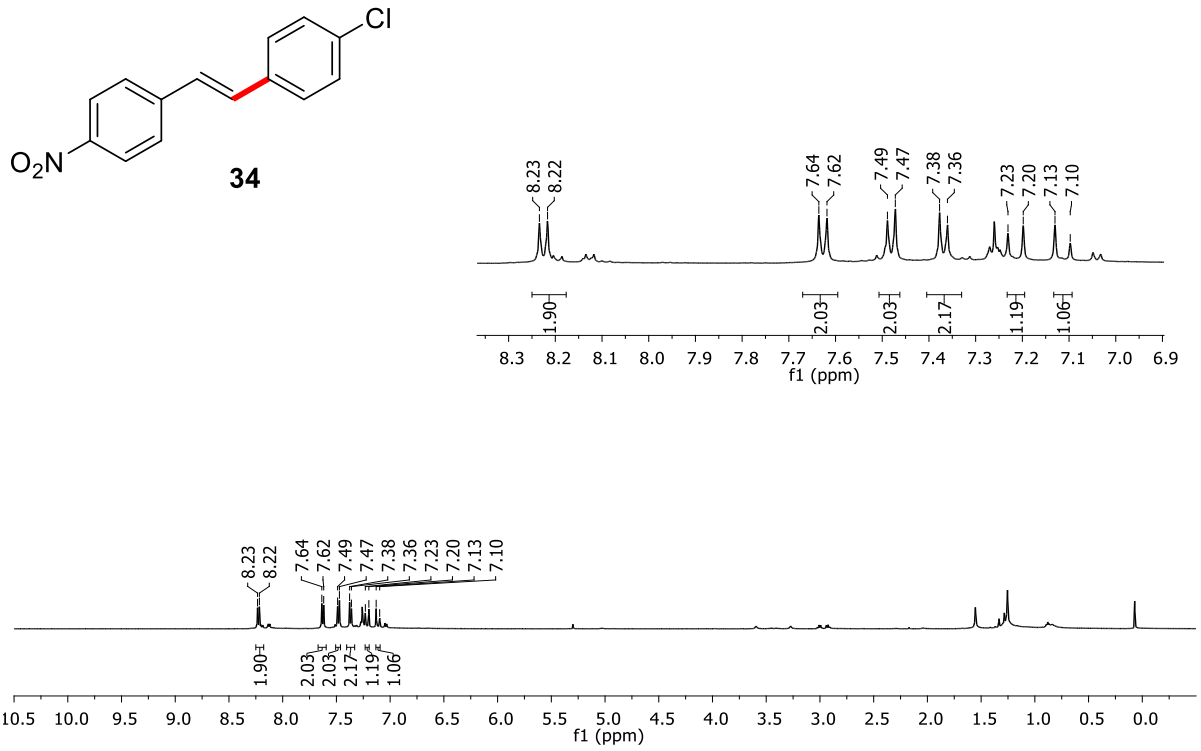

Fig. 92. [13]C NMR of (*E*)-1-Chloro-4-(4-nitrostyryl)benzene in $CDCl_3$ **(34)**[30].

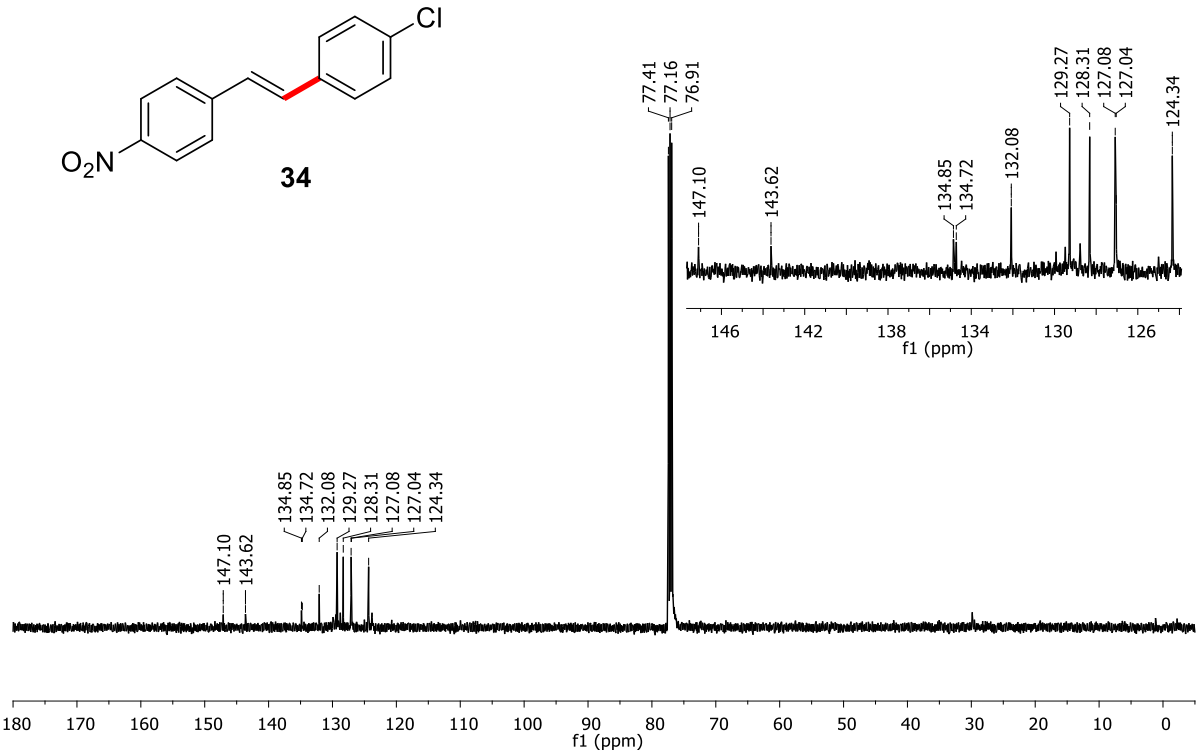

Fig. 93. ^{1}H NMR of 2',4',6'-Trimethoxy-[1,1'-biphenyl]-3-carbonitrile in $CDCl_3$ **(35)**.

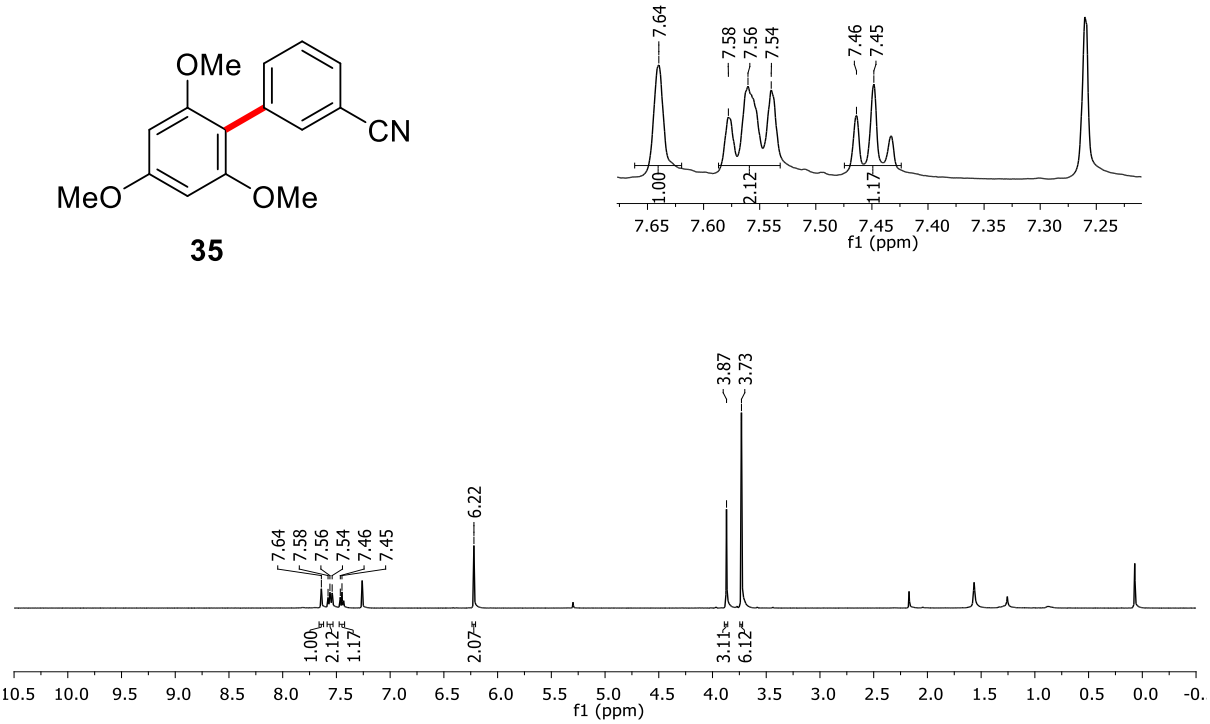

Fig. 94. ^{13}C NMR of 2',4',6'-Trimethoxy-[1,1'-biphenyl]-3-carbonitrile in $CDCl_3$ **(35)**.

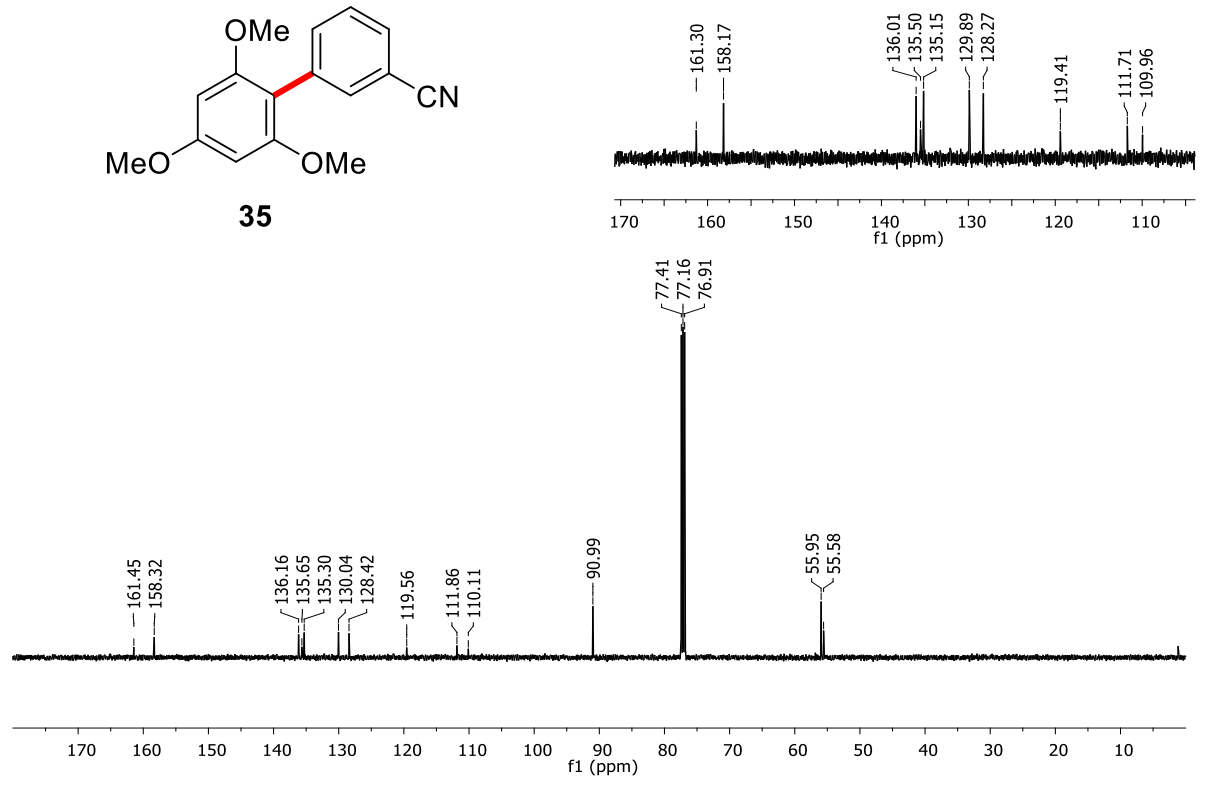

Fig. 95. [1] H NMR of 2,4,6-Trimethoxy-1,1',4',1''-terphenyl in $CDCl_3$ **(36)**[32].

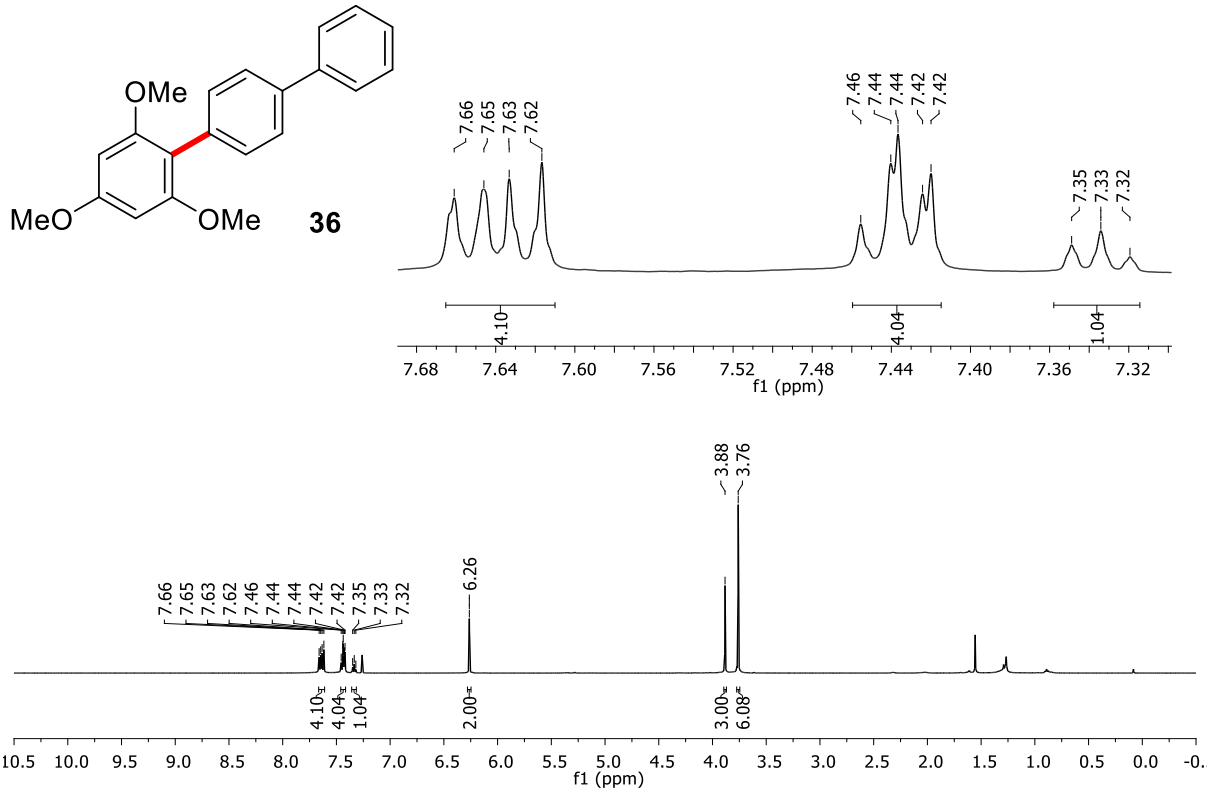

Fig. 96. [13]C NMR of 2,4,6-Trimethoxy-1,1',4',1''-terphenyl in $CDCl_3$ **(36)**[32].

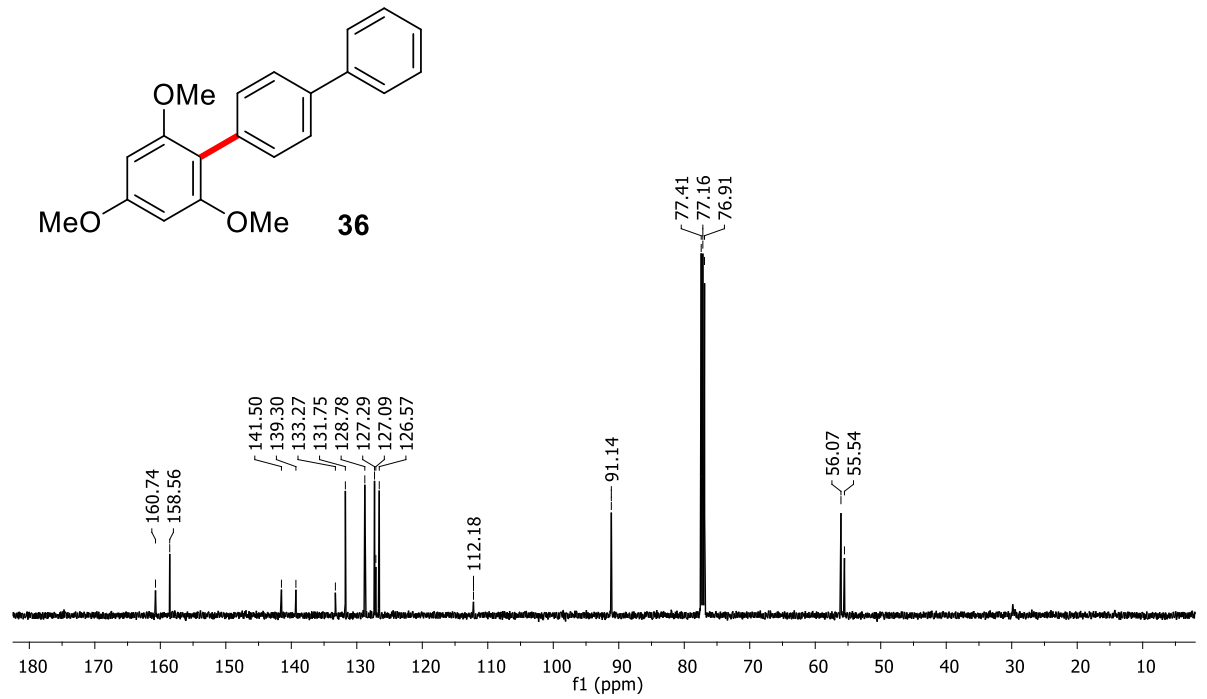

Fig. 97. [1] H NMR of 2',4',6'-Trimethoxy-[1,1'-biphenyl]-4-carbonitrile in $CDCl_3$ **(37)**[32].

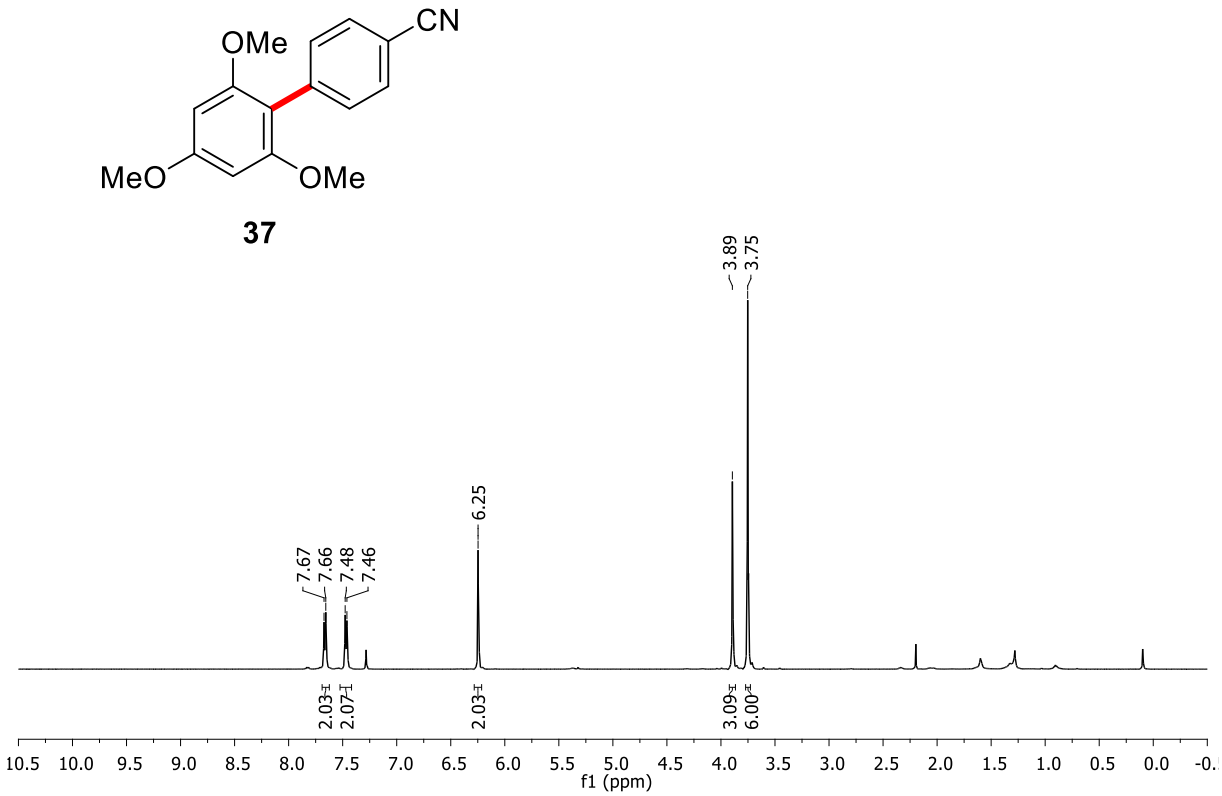

Fig. 98. [13]C NMR of 2',4',6'-Trimethoxy-[1,1'-biphenyl]-4-carbonitrile in $CDCl_3$ **(37)**[32].

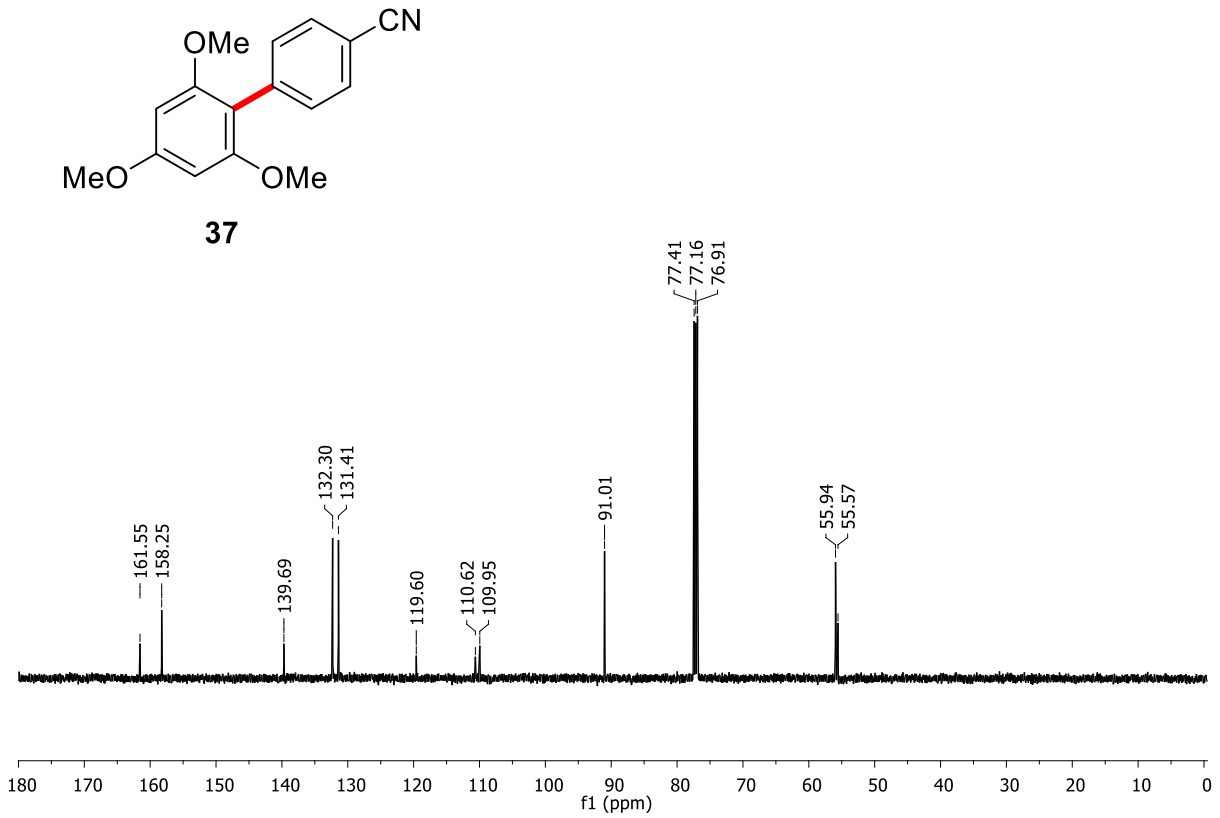

Fig. 99. ^{1}H NMR of 2,4, 4',6-Tetramethoxy-1,1'-biphenyl in $CDCl_3$ **(38)**[32].

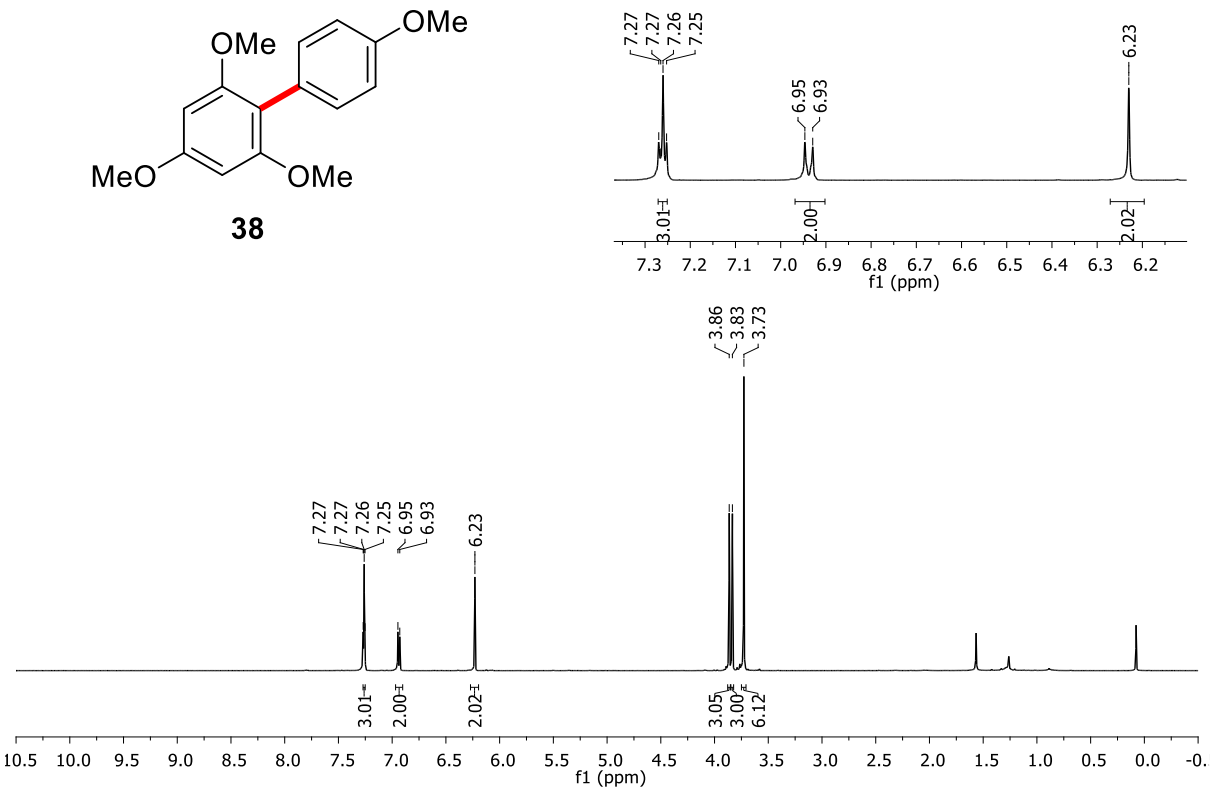

Fig. 100. ^{13}C NMR of 2,4, 4',6-Tetramethoxy-1,1'-biphenyl in $CDCl_3$ **(38)**[32].

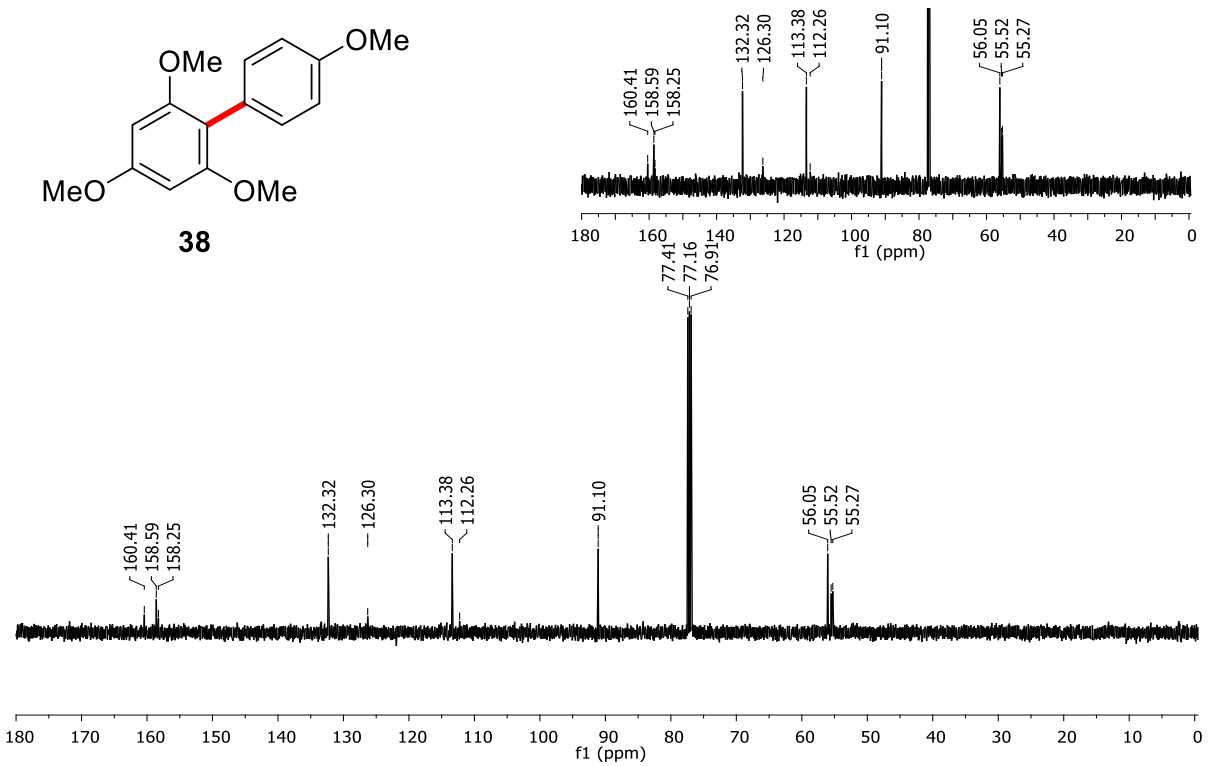

Fig. 101. [1] H NMR of 2, 4, 6-Trimethoxy-4'-methyl-1,1'-biphenyl in $CDCl_3$ **(39)**[33].

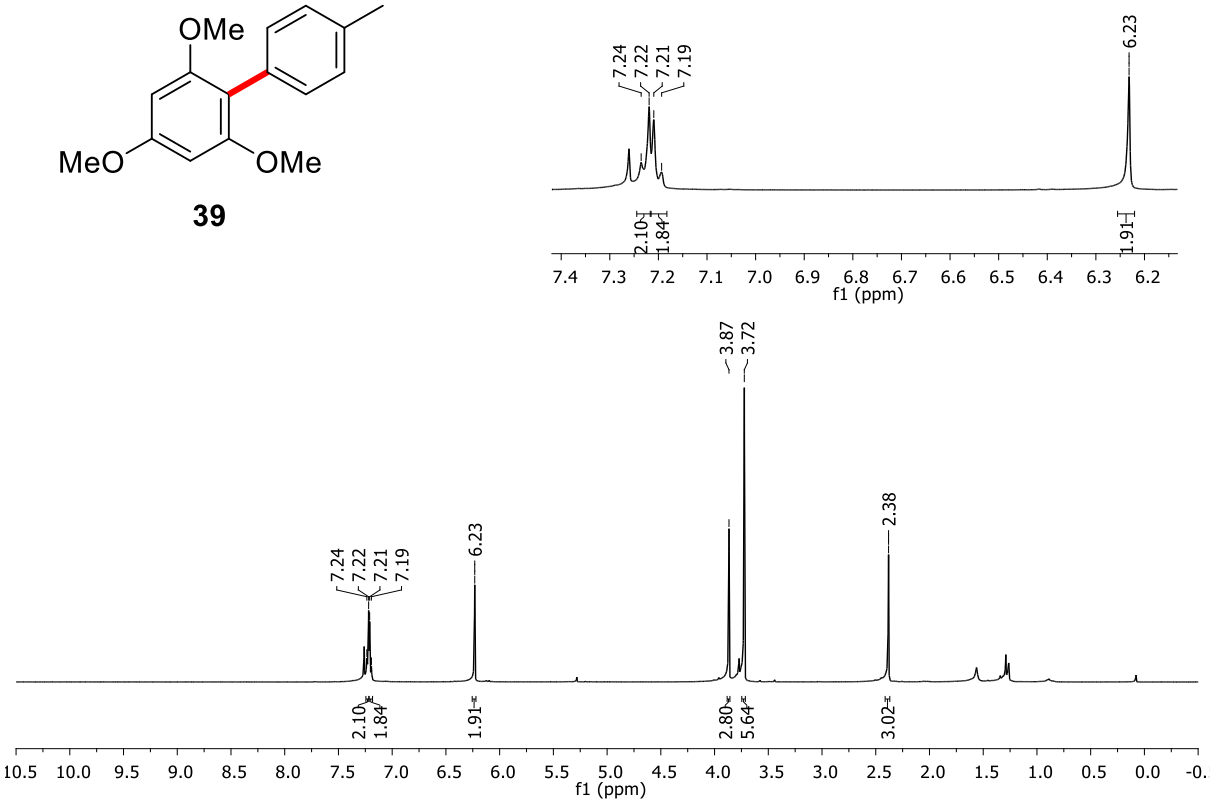

Fig. 102. [13]C NMR of 2, 4, 6-Trimethoxy-4'-methyl-1,1'-biphenyl in $CDCl_3$ **(39)**[33].

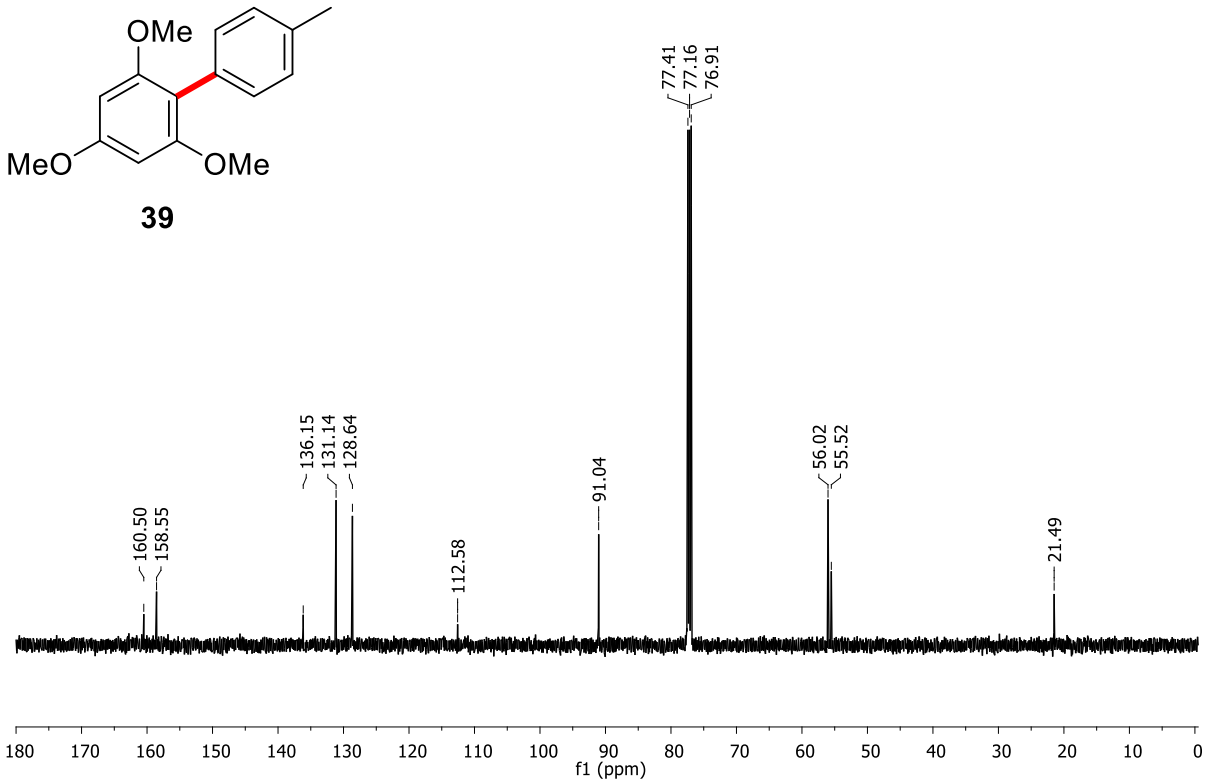

Fig. 103. ^{1}H NMR of 4'-Chloro-2,5-dimethoxy-1,1'-biphenyl in $CDCl_3$ **(40)**[34].

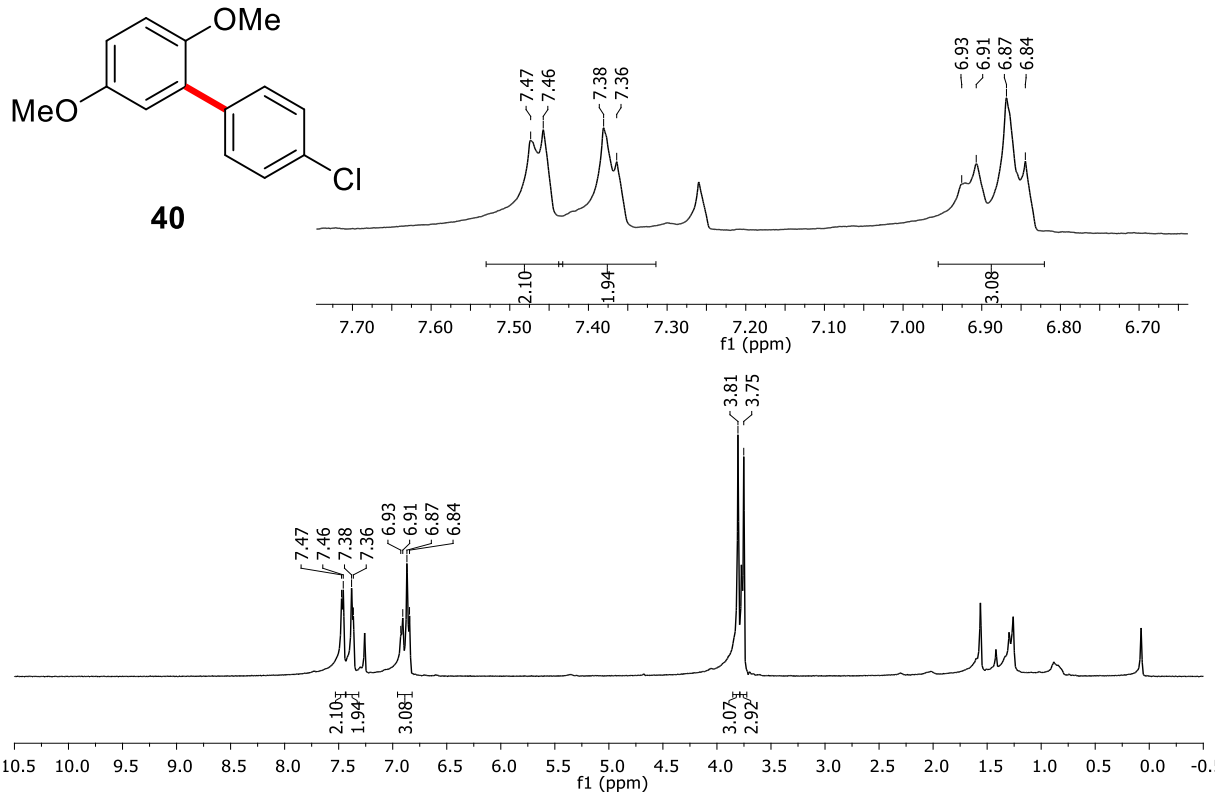

Fig. 104. ^{13}C NMR of 4'-Chloro-2,5-dimethoxy-1,1'-biphenyl in $CDCl_3$ **(40)**[34].

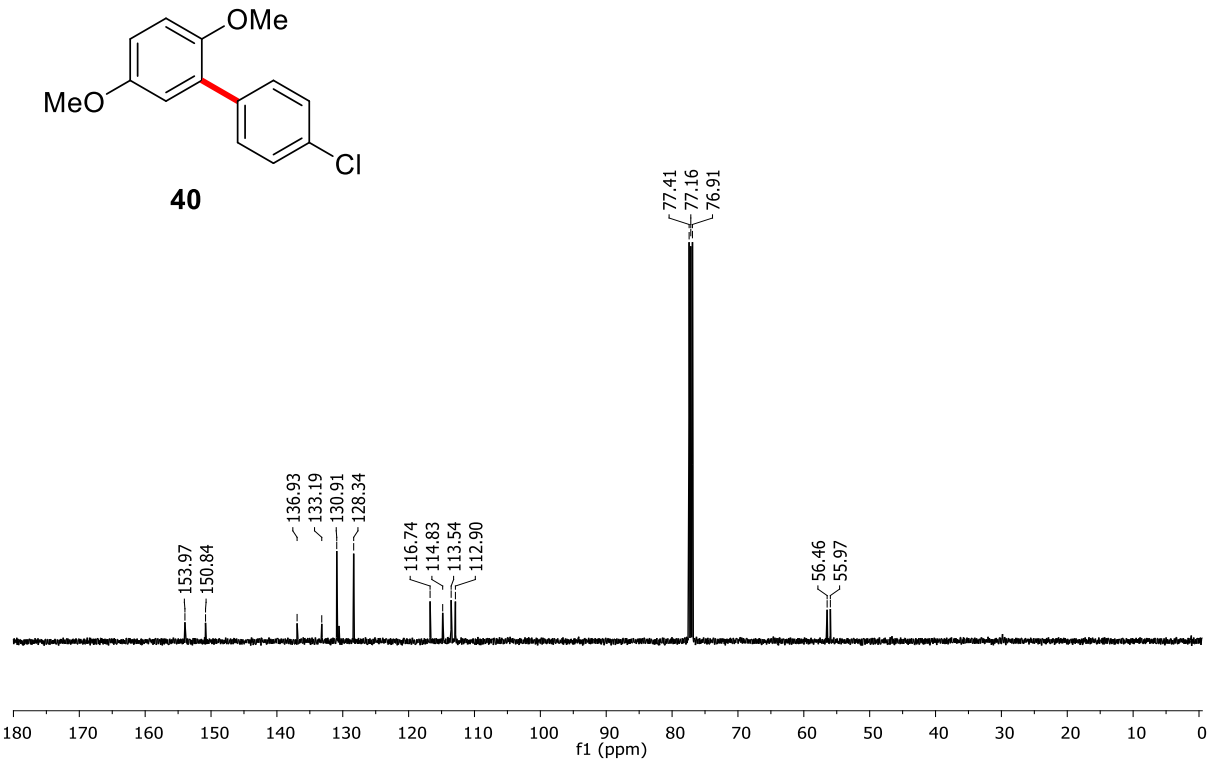

Fig. 105. ^{1}H NMR of 2,5-Dimethoxy-1,1':4',1''-terphenyl in $CDCl_3$ **(41)**.

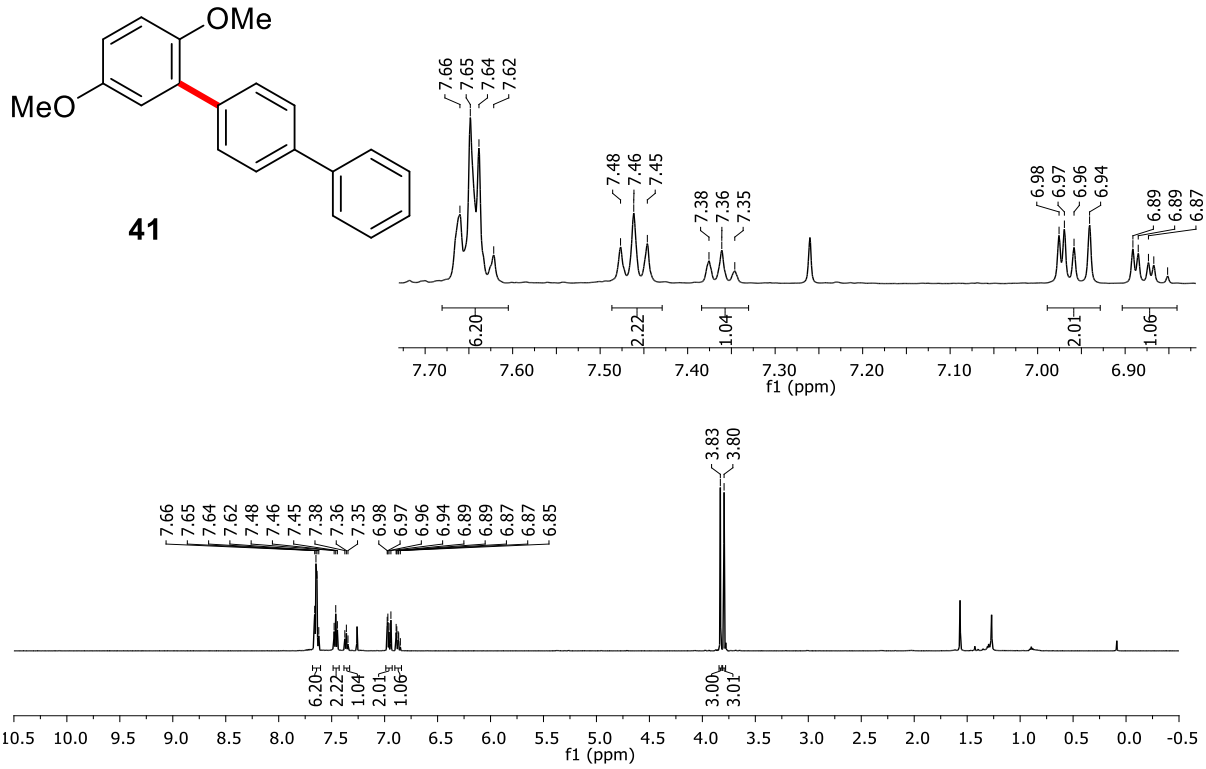

Fig. 106. ^{13}C NMR of 2,5-Dimethoxy-1,1':4',1''-terphenyl in $CDCl_3$ **(41)**.

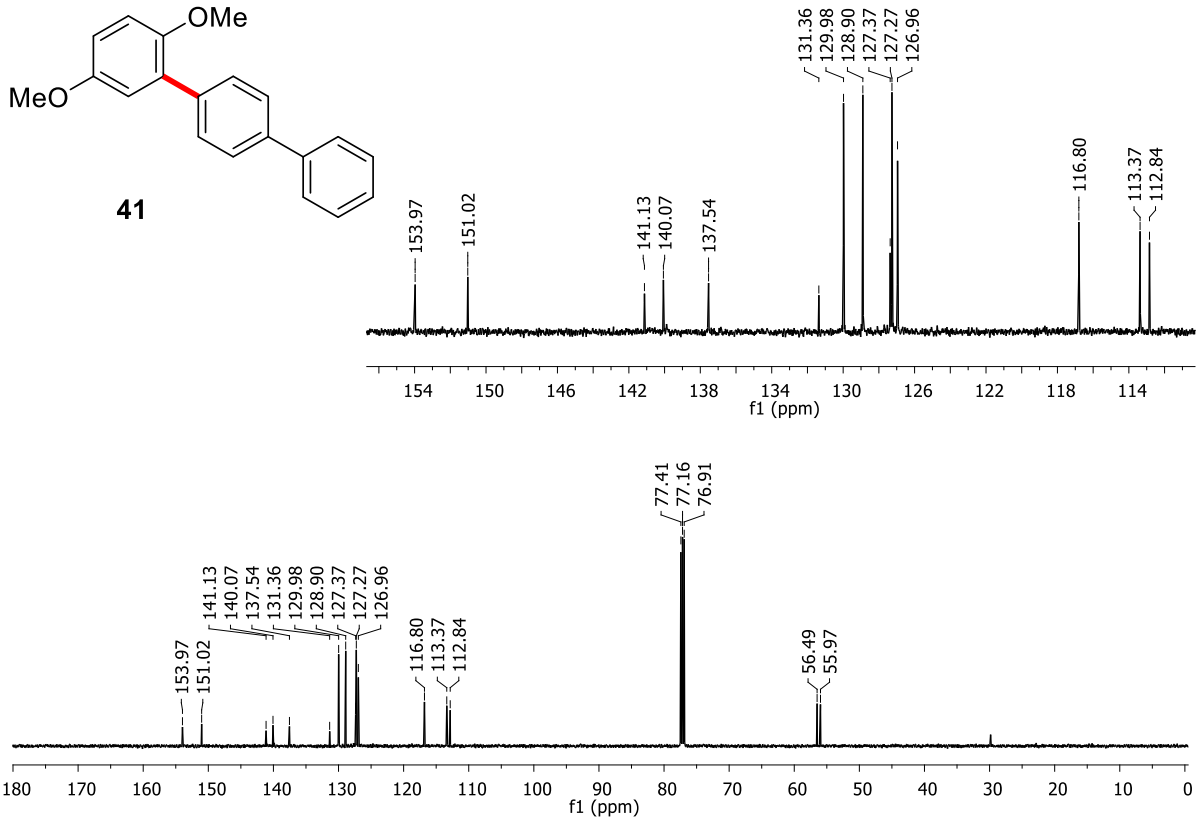

Fig. 107. [1] H NMR of 2', 5'-Dimethoxy-[1,1'-biphenyl]-4-carbonitrile in $CDCl_3$ **(42)**[35].

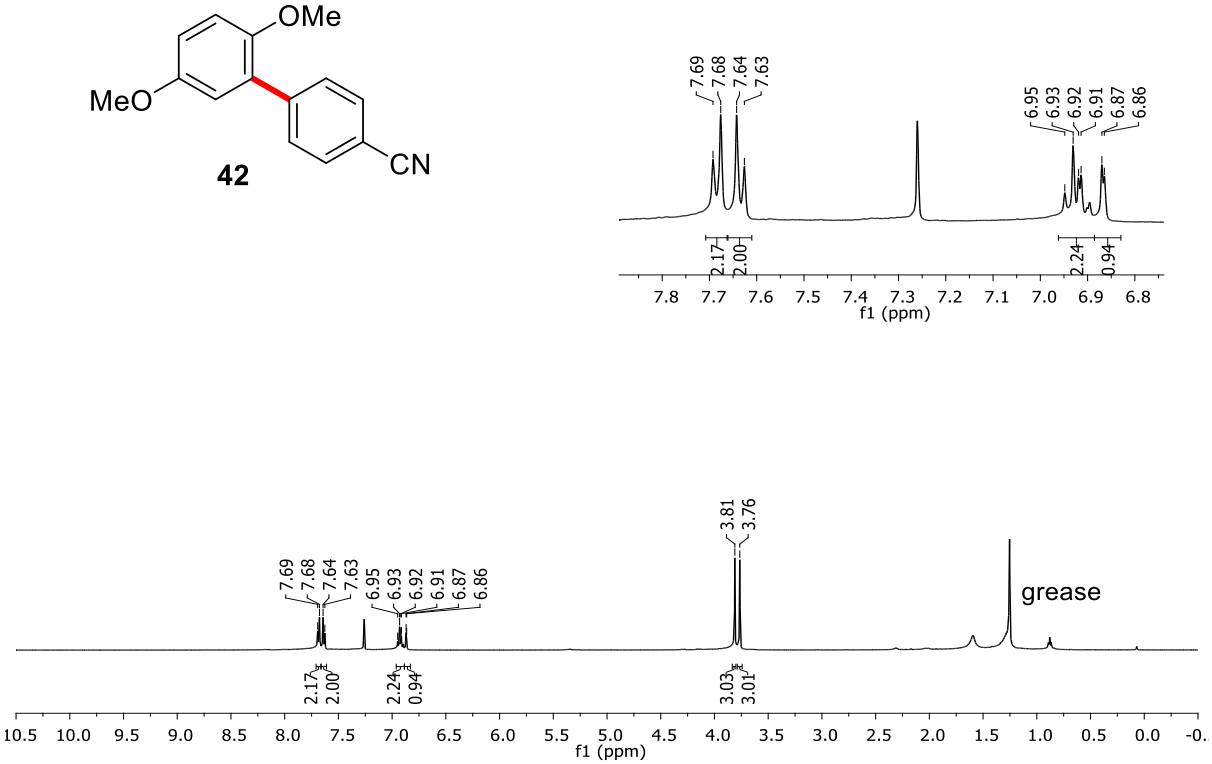

Fig. 108. [13]C NMR of 2', 5'-Dimethoxy-[1,1'-biphenyl]-4-carbonitrile in $CDCl_3$ **(42)**[35].

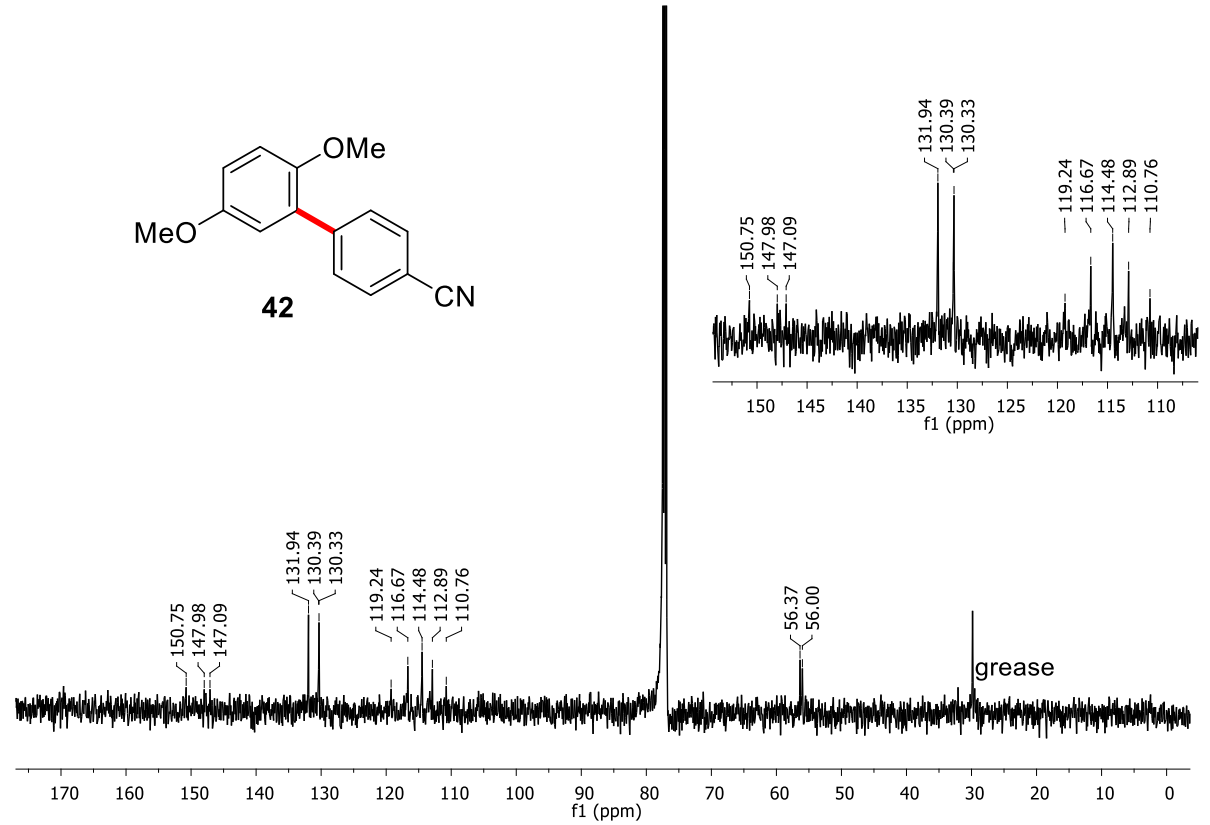

Fig. 109. ^{1}H NMR of 2,5-Dimethoxy-4'-methyl-1,1'-biphenyl in $CDCl_3$ **(43)**[36].

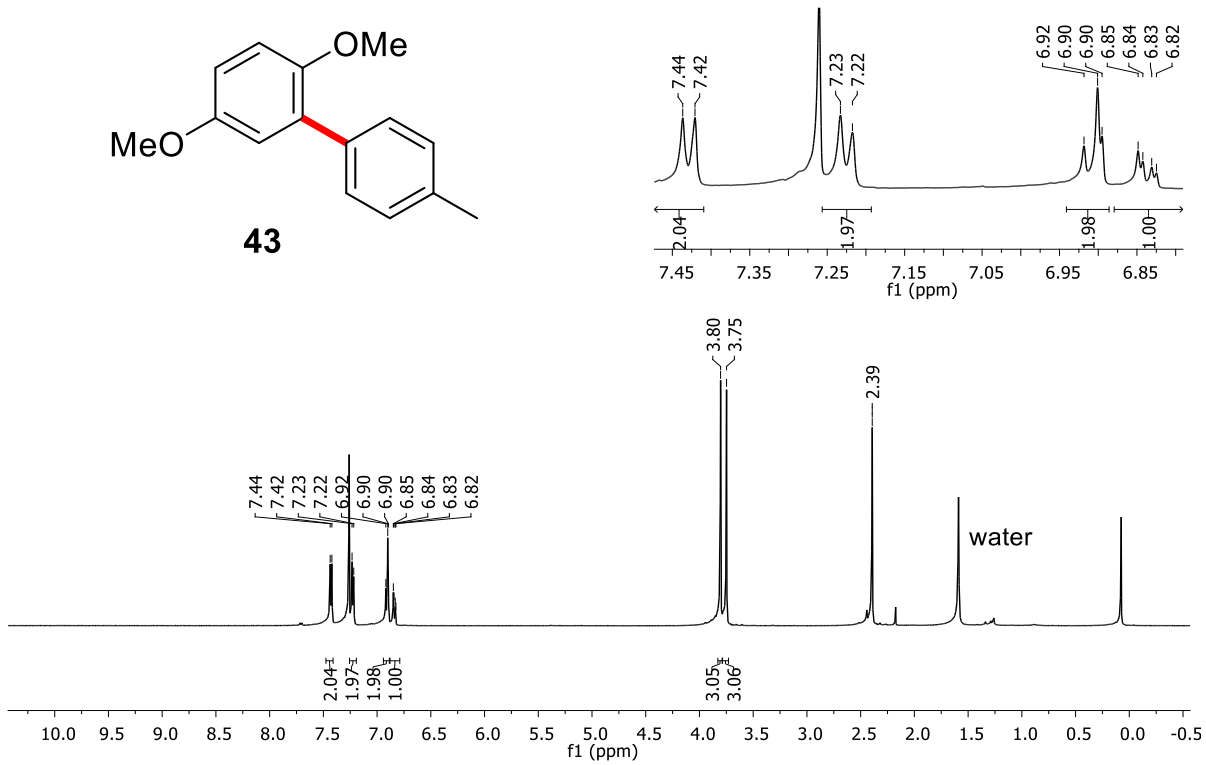

Fig. 110. ^{13}C NMR of 2,5-Dimethoxy-4'-methyl-1,1'-biphenyl in $CDCl_3$ **(43)**[36].

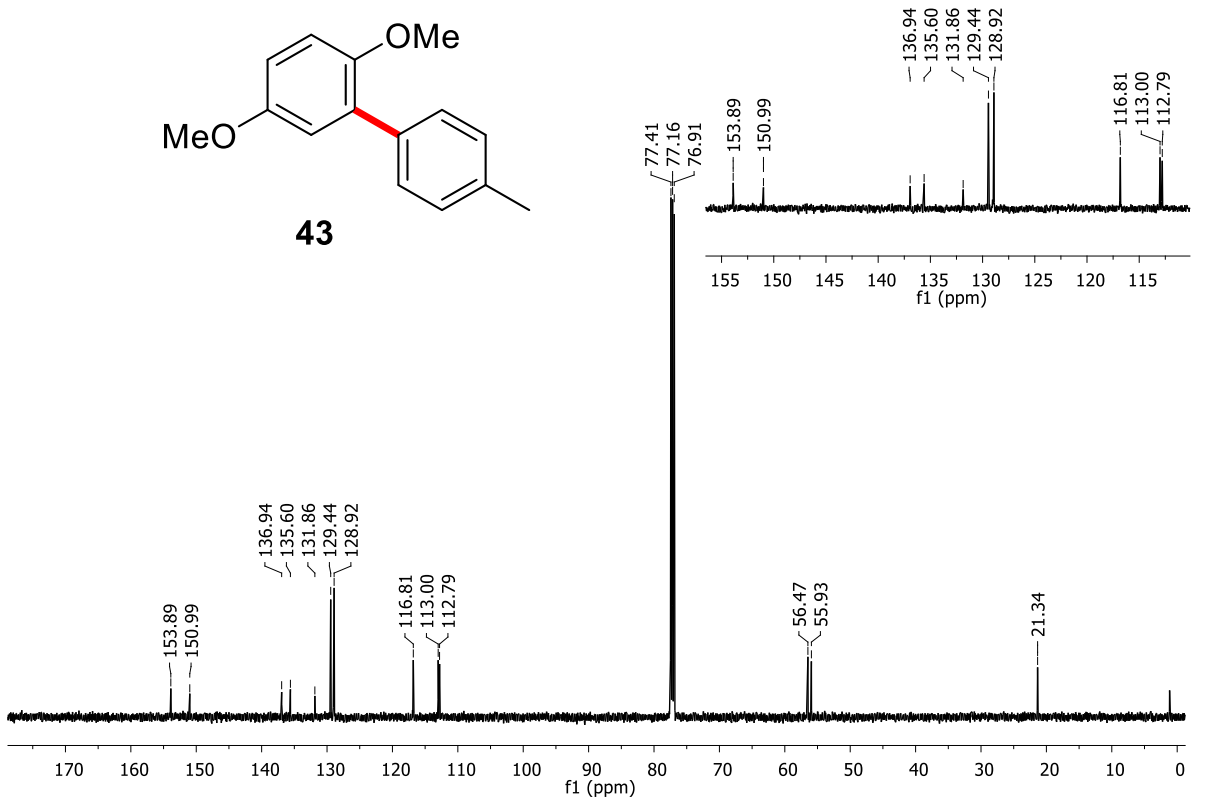

Fig. 111. [1] H NMR of 2,4',5-Trimethoxy-1,1'-biphenyl in $CDCl_3$ **(44)**[37].

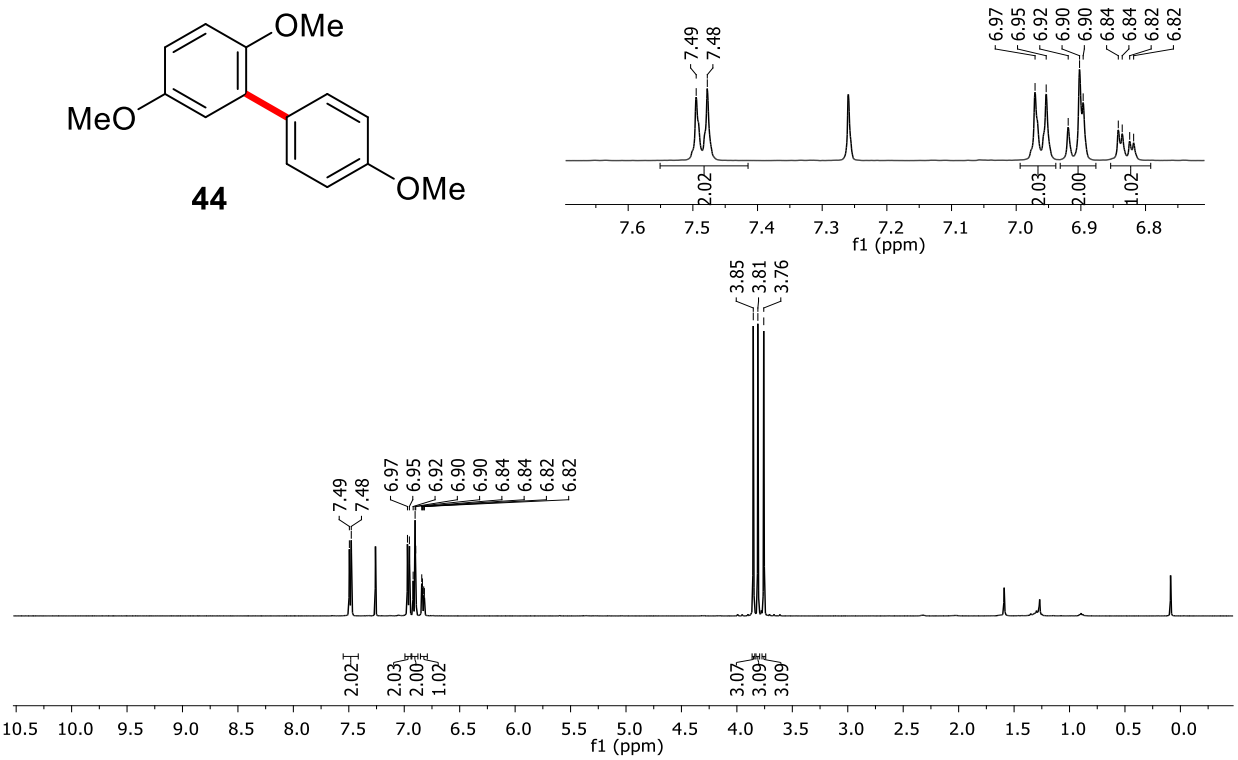

Fig. 112. [13]C NMR of 2,4',5-Trimethoxy-1,1'-biphenyl in $CDCl_3$ **(44)**[37].

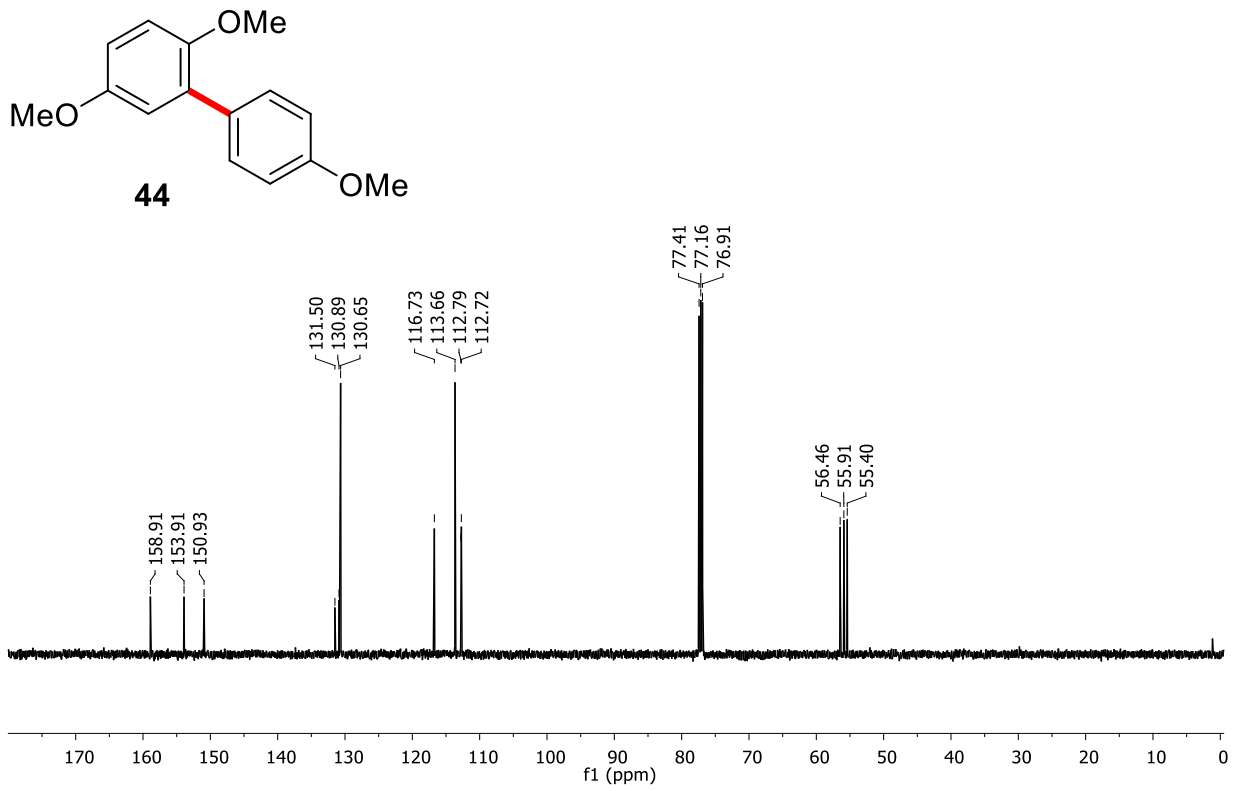

Fig. 113. ^{1}H NMR of 1-(p-Tolyl)pyrene in $CDCl_3$ **(45)**[38].

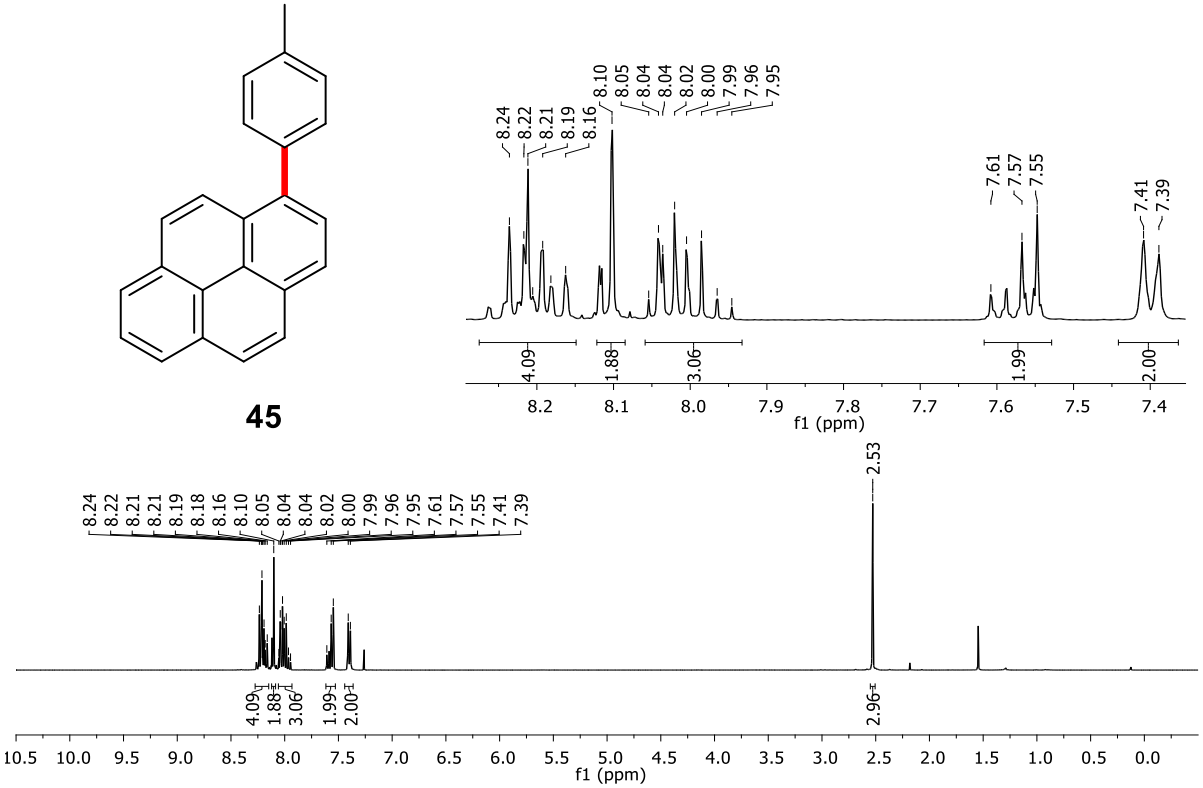

Fig. 114. ^{13}C NMR of 1-(p-Tolyl)pyrene in $CDCl_3$ **(45)**[38].

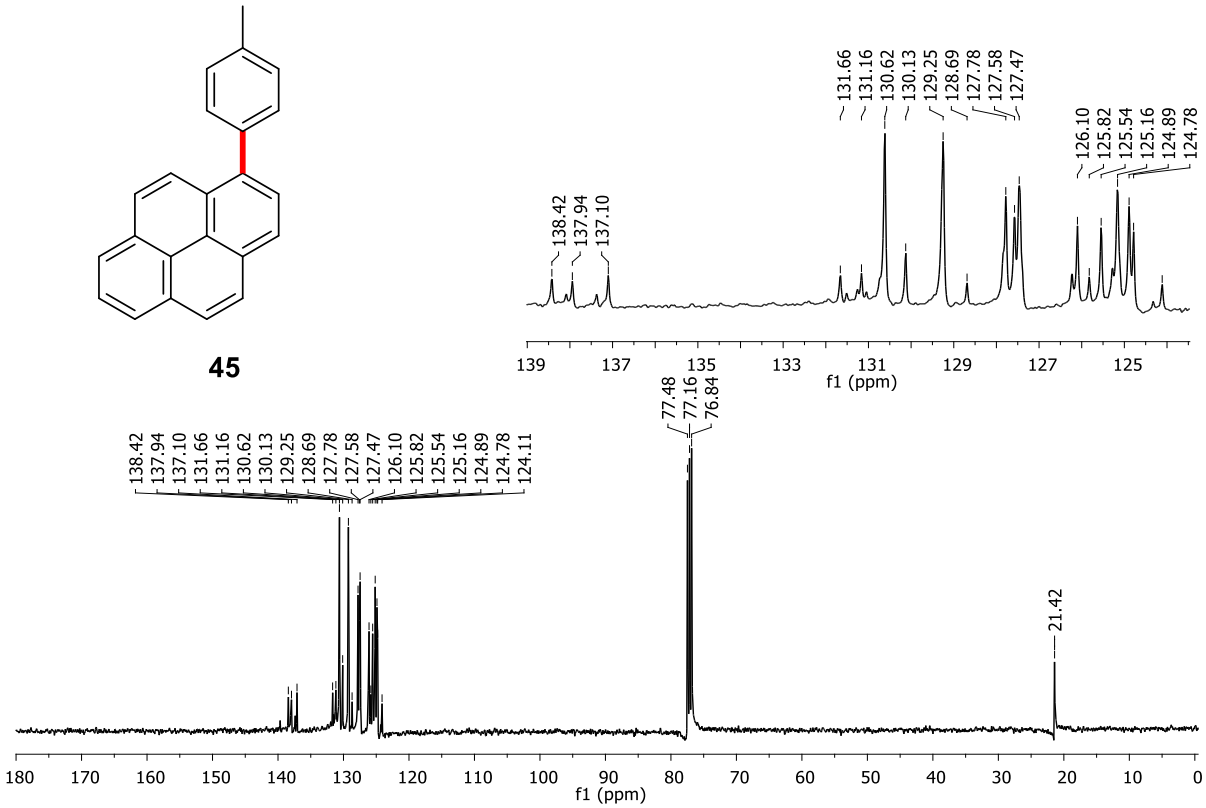

Fig. 115. ^{1}H NMR of 1-(4-Methoxyphenyl)pyrene in $CDCl_3$ **(46)**[39].

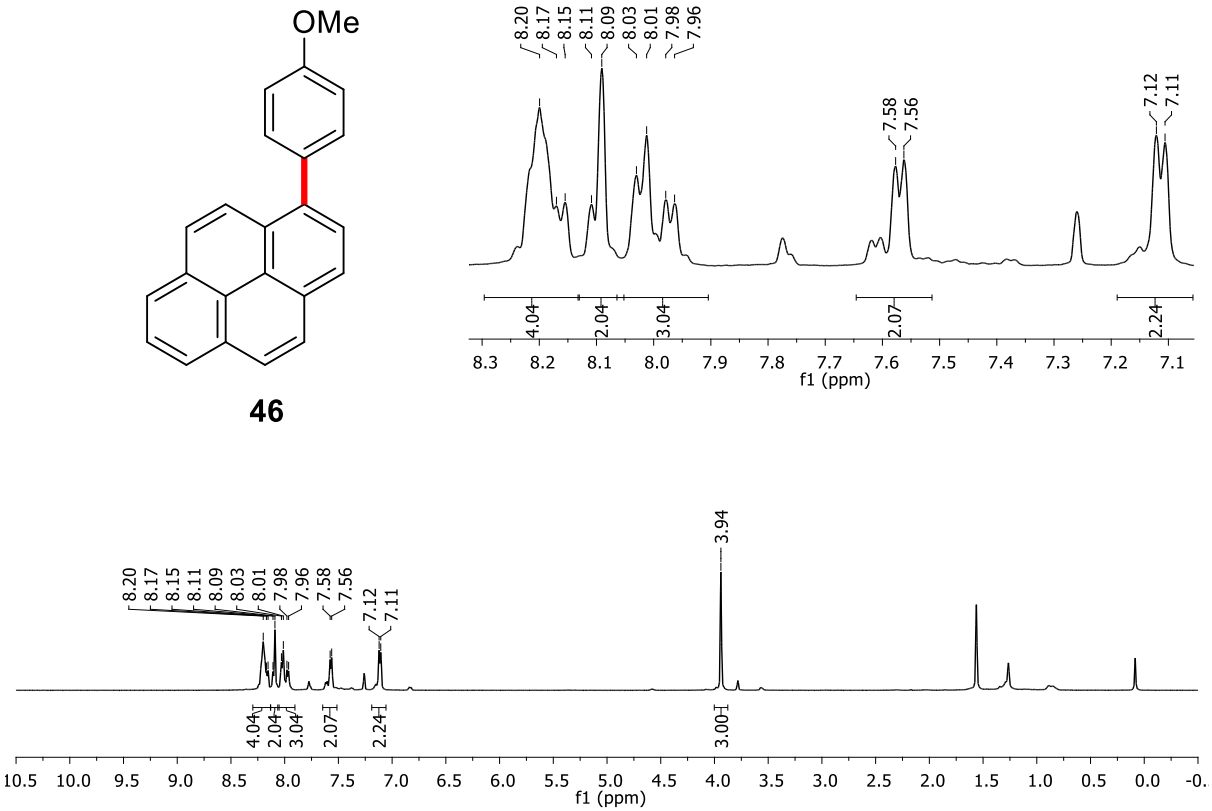

Fig. 116. ^{13}C NMR of 1-(4-Methoxyphenyl)pyrene in $CDCl_3$ **(46)**[39].

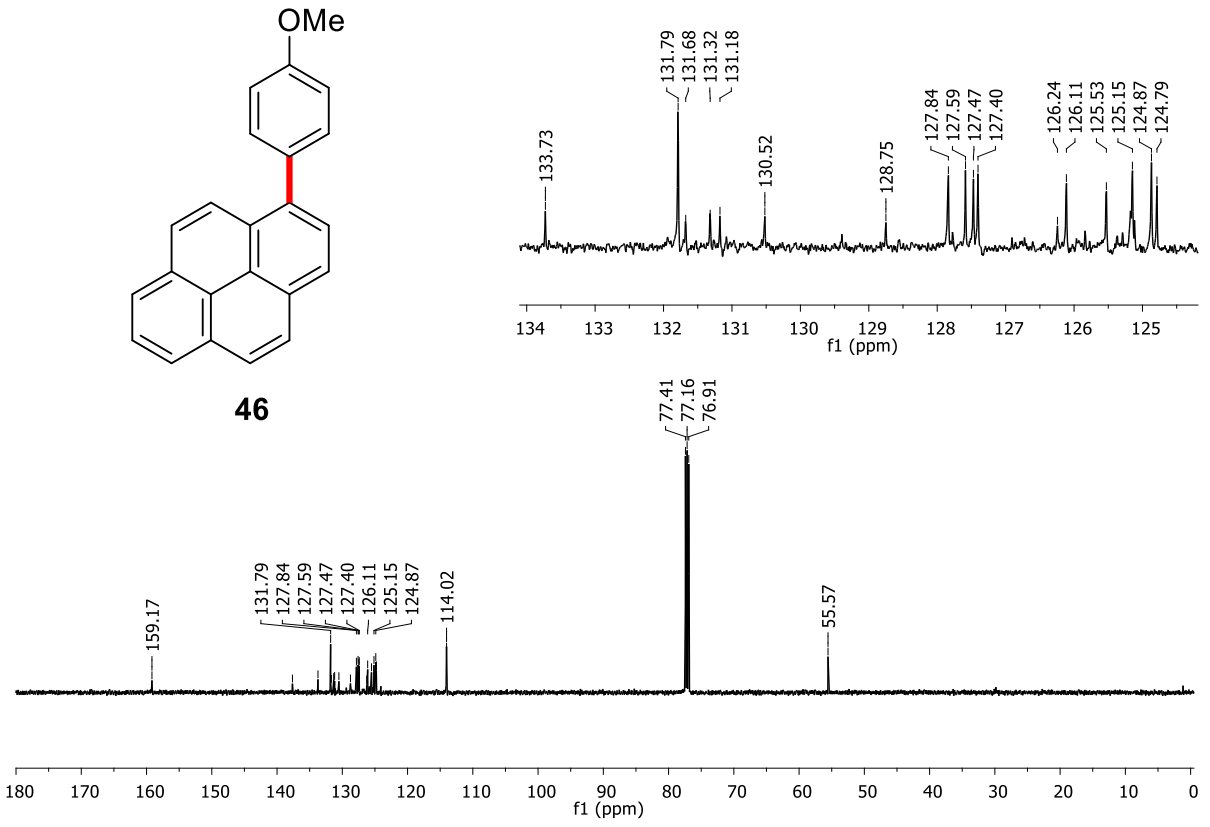

Fig. 117. [1] H NMR of 9-(4-Methoxyphenyl)anthracene in $CDCl_3$ **(47)**[40].

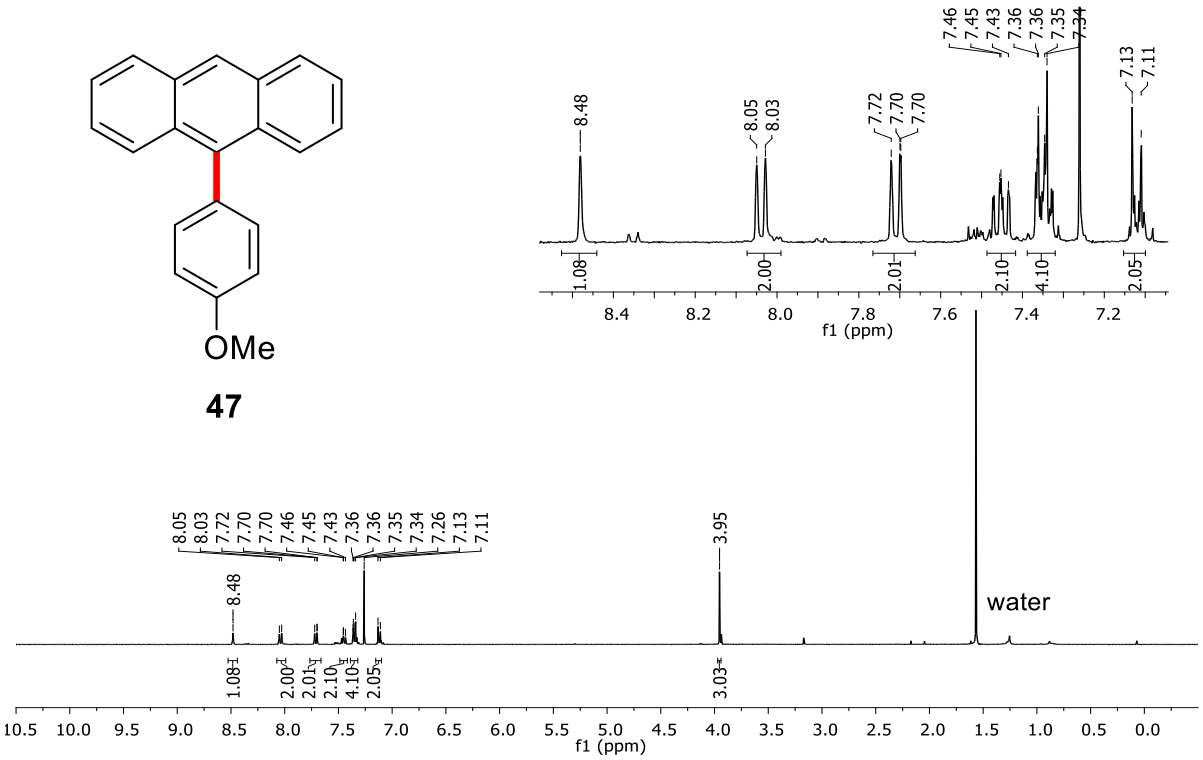

Fig. 118. [13]C NMR of 9-(4-Methoxyphenyl)anthracene in $CDCl_3$ **(47)**[40].

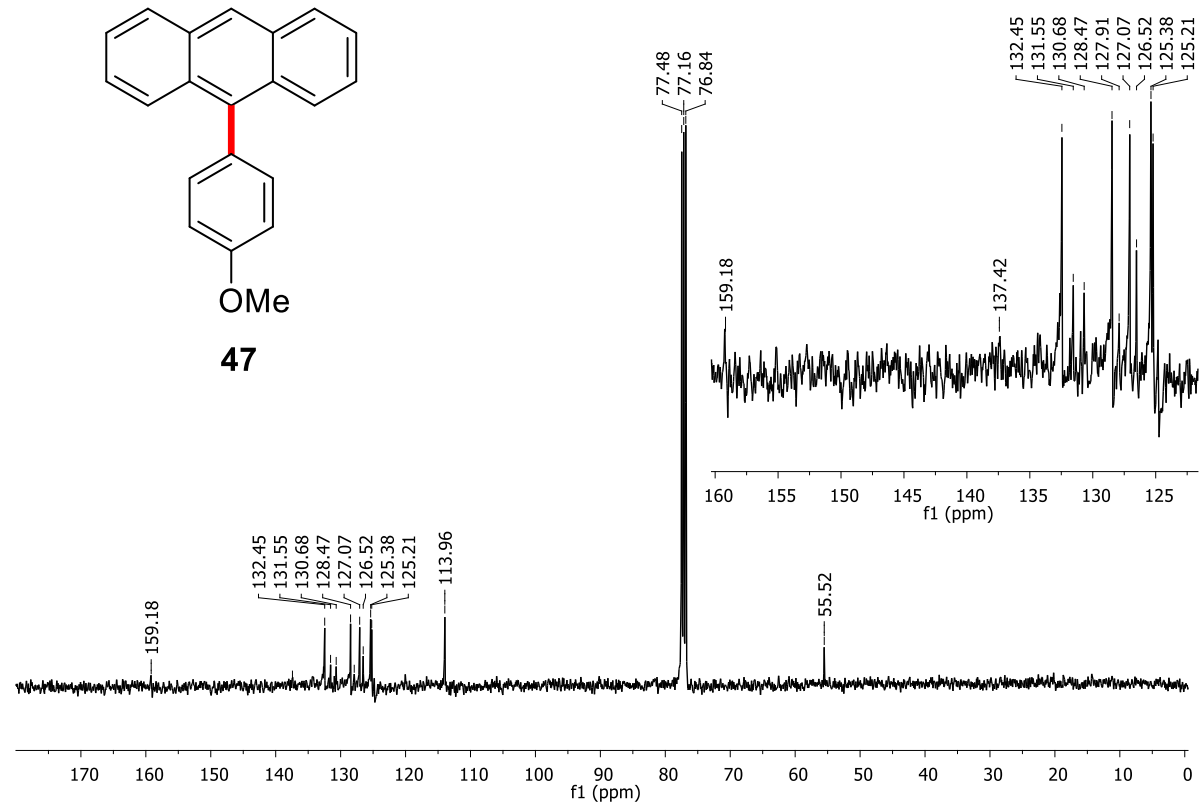

Fig. 119. [1] H NMR of 9-(4-Methoxyphenyl)phenanthrene in $CDCl_3$ **(48)**[41].

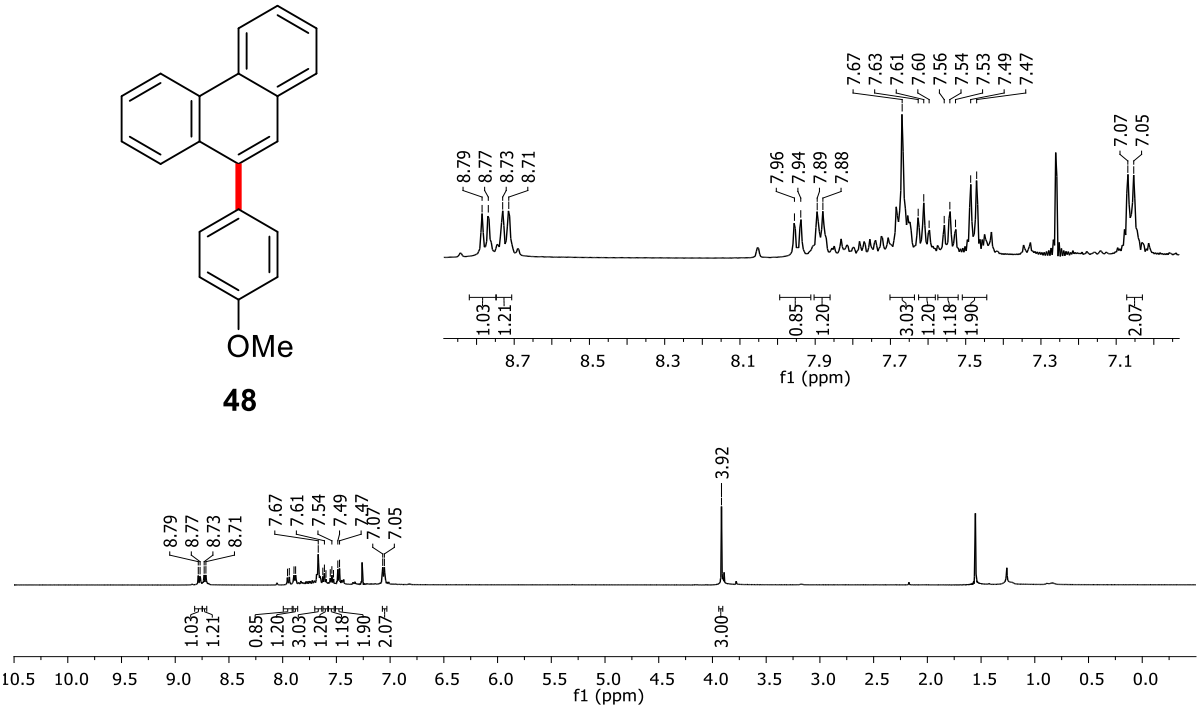

Fig. 120. [13]C NMR of 9-(4-Methoxyphenyl)phenanthrene in $CDCl_3$ **(48)**[41].

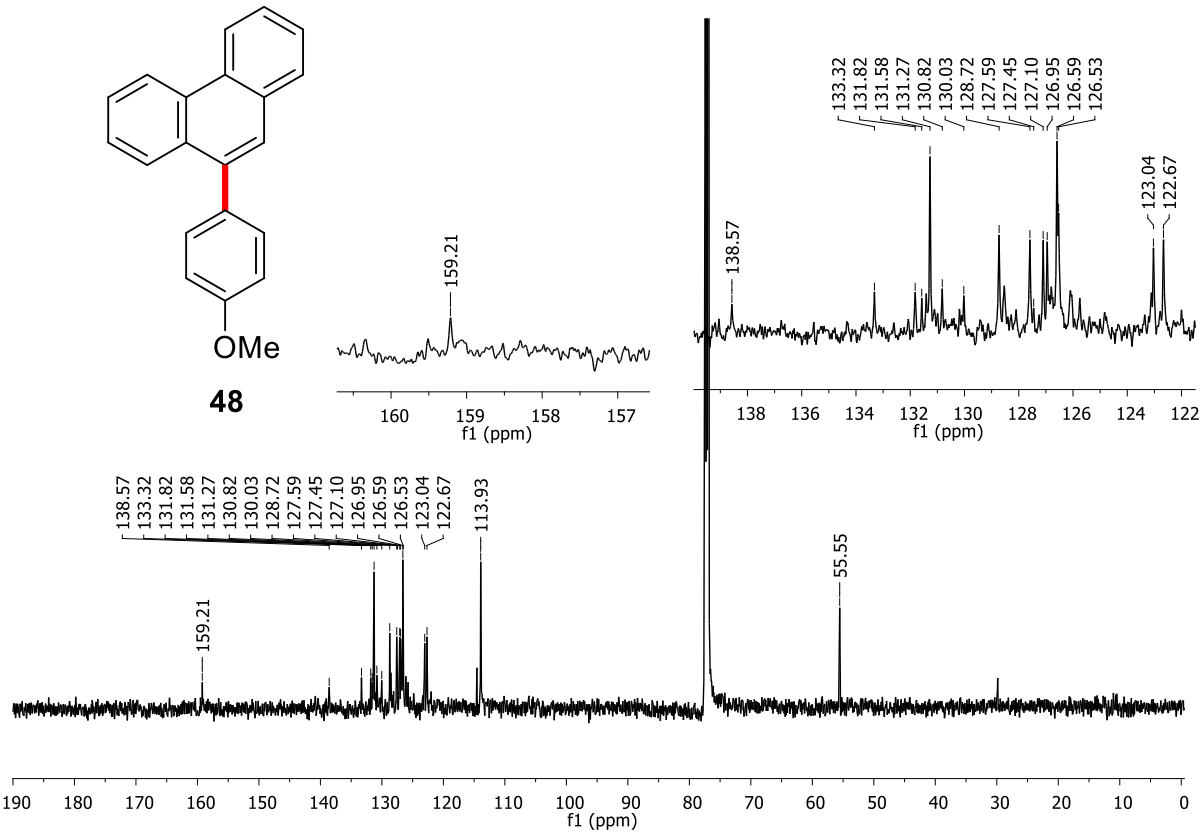

Fig. 121. [1] H NMR of 1-(*p*-Tolyl)naphthalene & 2-(*p*-Tolyl)naphthalene in $CDCl_3$ **(49)**[42].

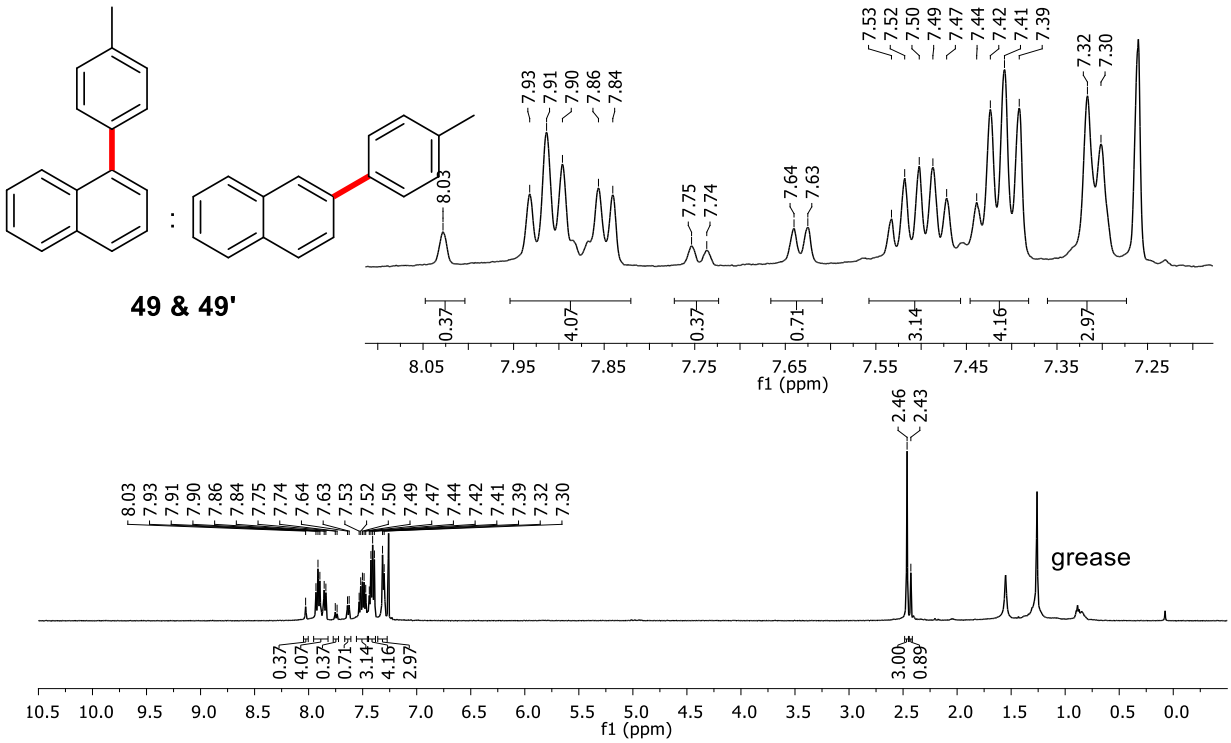

Fig. 122. [13]C NMR of 1-(*p*-Tolyl)naphthalene & 2-(*p*-Tolyl)naphthalene in $CDCl_3$ **(49)**[42].

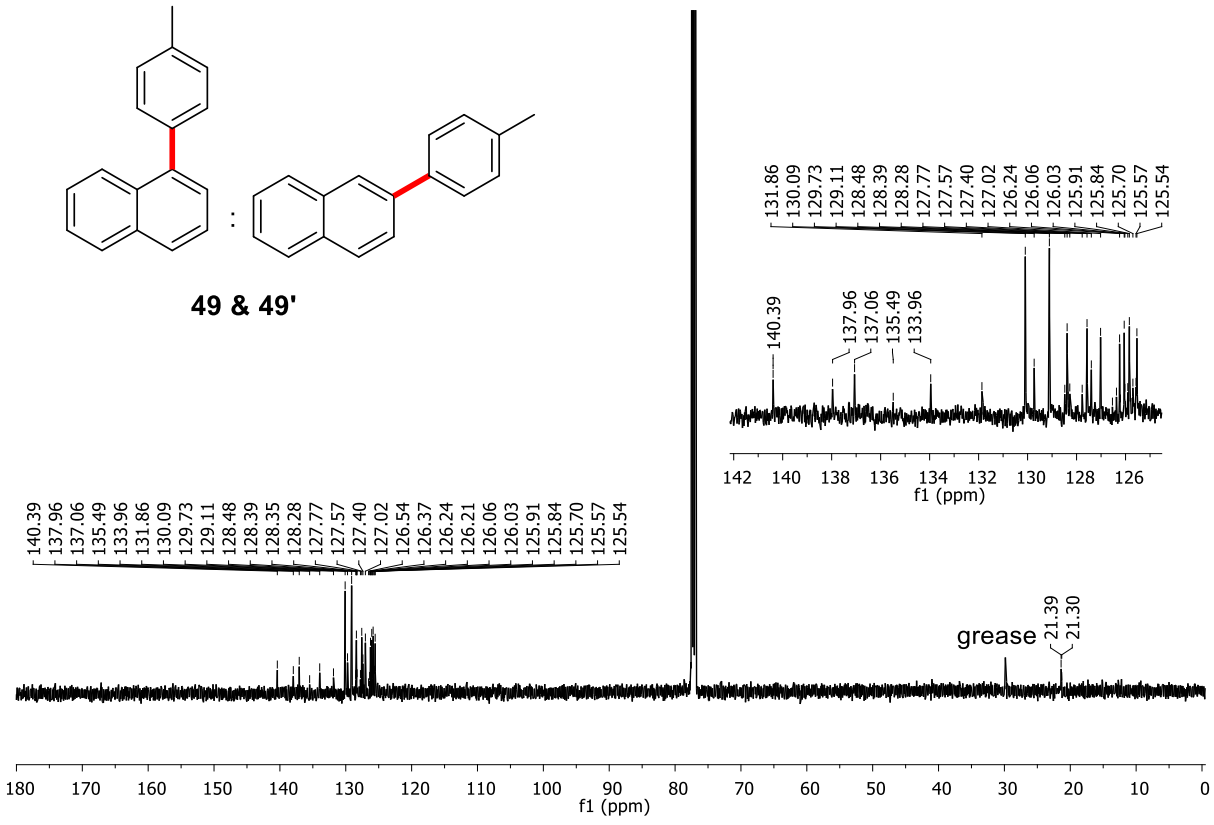

Fig. 123. [1] H NMR of 1-(4-Methoxyphenyl)naphthalene and 2-(4-Methoxyphenyl)naphthalene in $CDCl_3$ **(50)**[43].

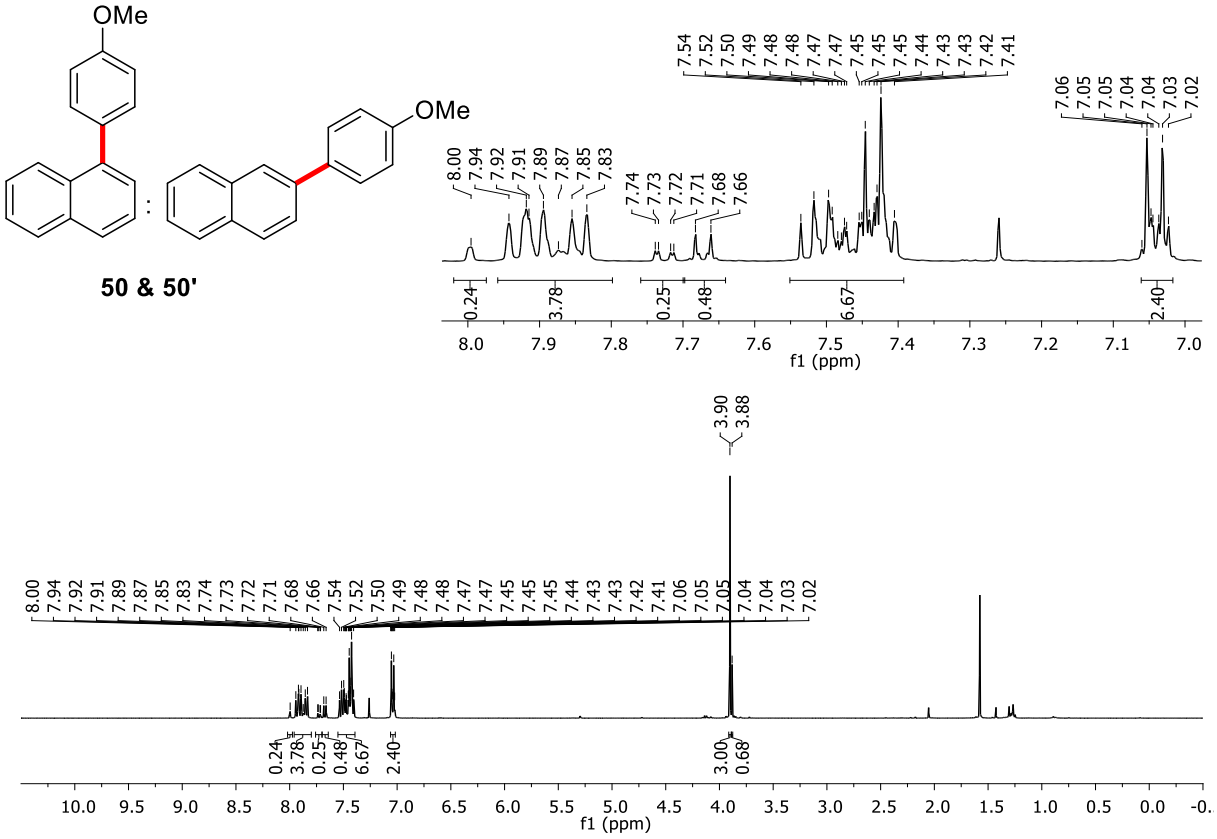

Fig. 124. [13]C NMR of 1-(4-Methoxyphenyl)naphthalene and 2-(4-Methoxyphenyl)naphthalene in $CDCl_3$ **(50)**[43].

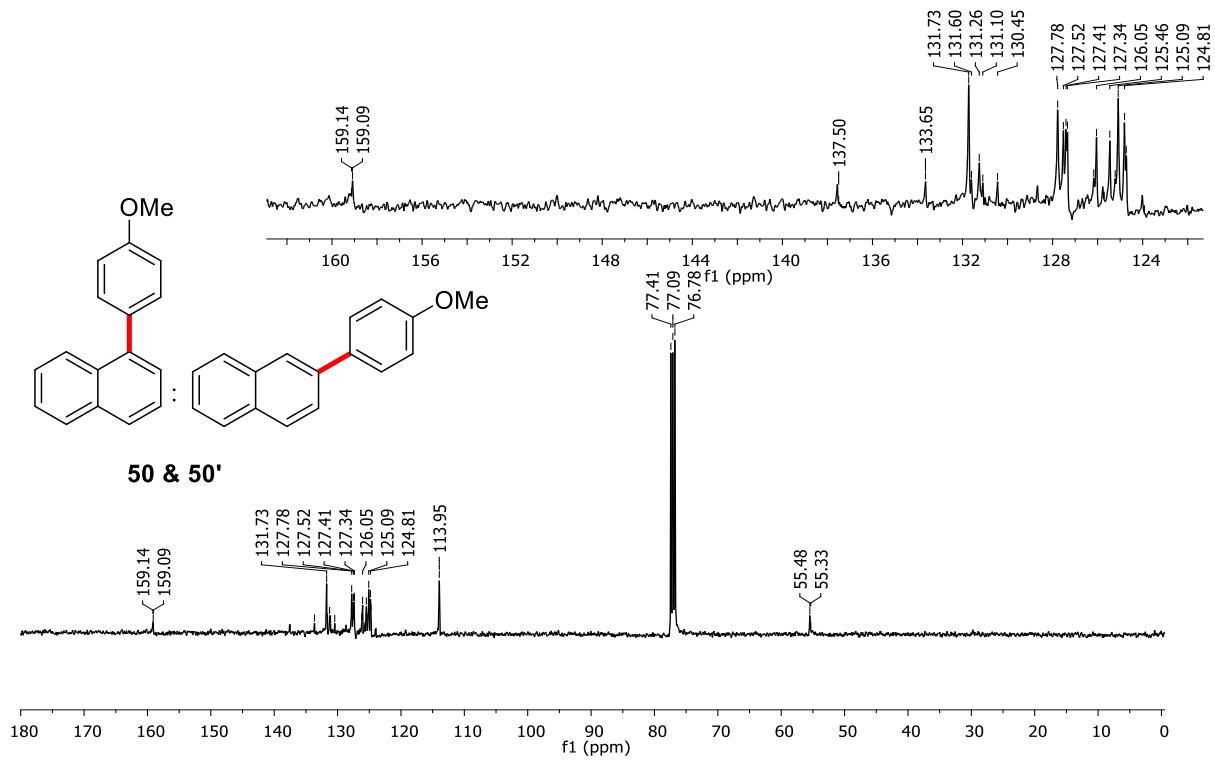

Fig. 125. ^{1}H NMR of (E)-4-(2-(naphthalen-2-yl)vinyl)benzonitrile in $CDCl_3$ **(51)**[44].

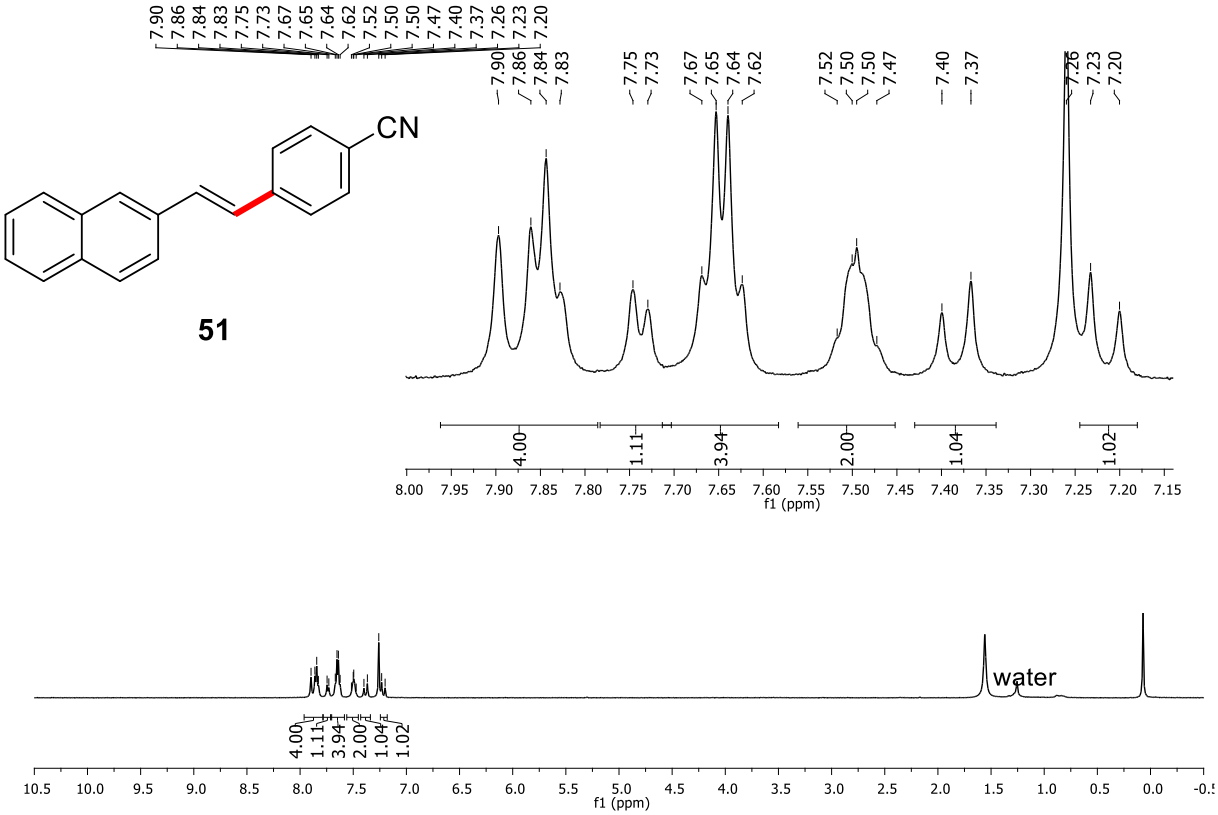

Fig. 126. ^{13}C NMR of (E)-4-(2-(naphthalen-2-yl)vinyl)benzonitrile in $CDCl_3$ **(51)**[44].

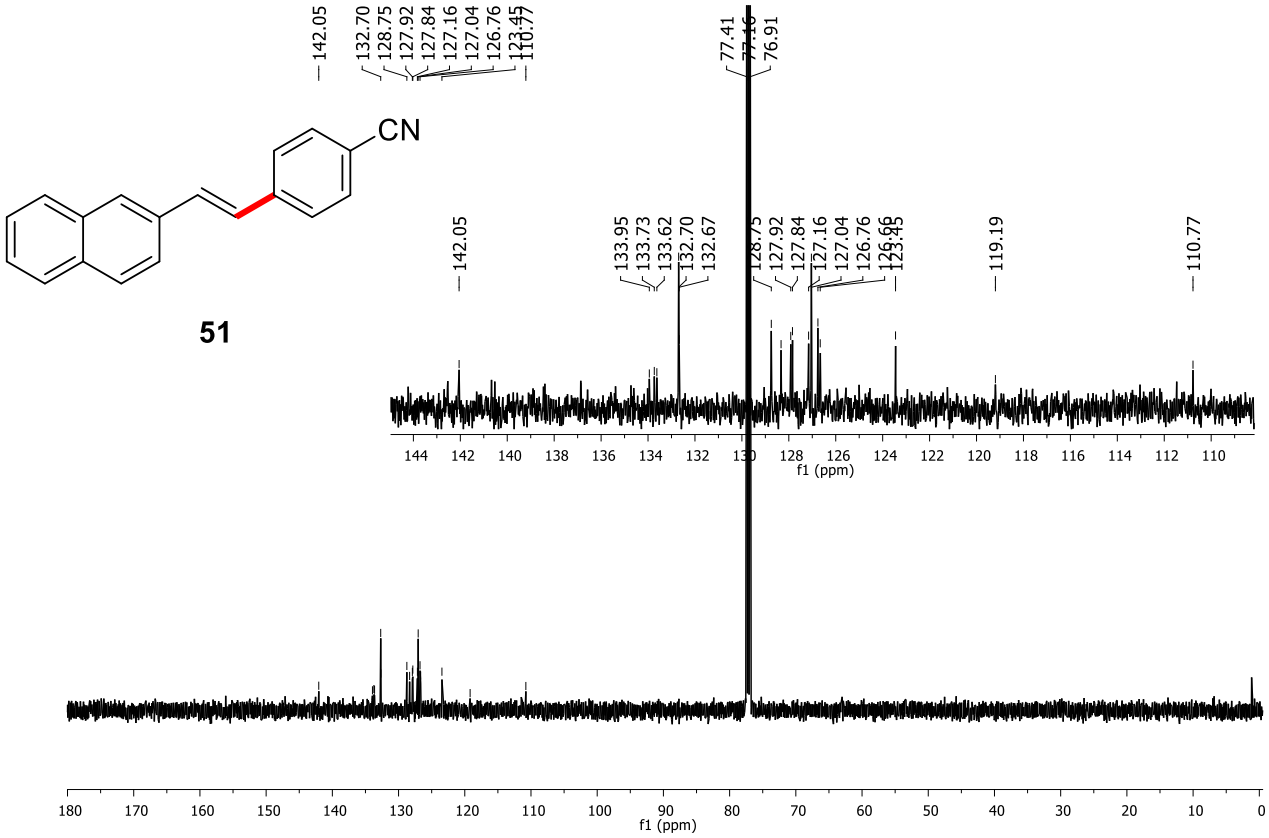

Fig. 127. [1] H NMR of (*E*)-2-(2-(Naphthalen-2-yl)vinyl) benzonitrile in $CDCl_3$ **(52)**[45].

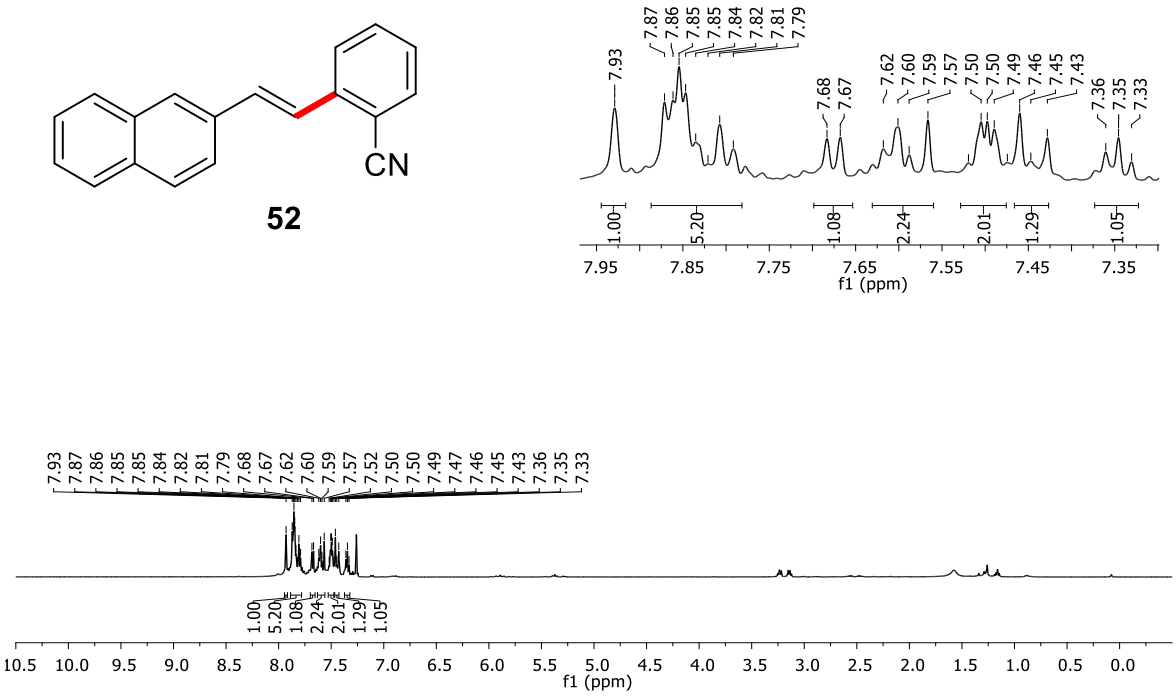

Fig. 128. [13]C NMR of (*E*)-2-(2-(Naphthalen-2-yl)vinyl) benzonitrile in $CDCl_3$ **(52)**[45].

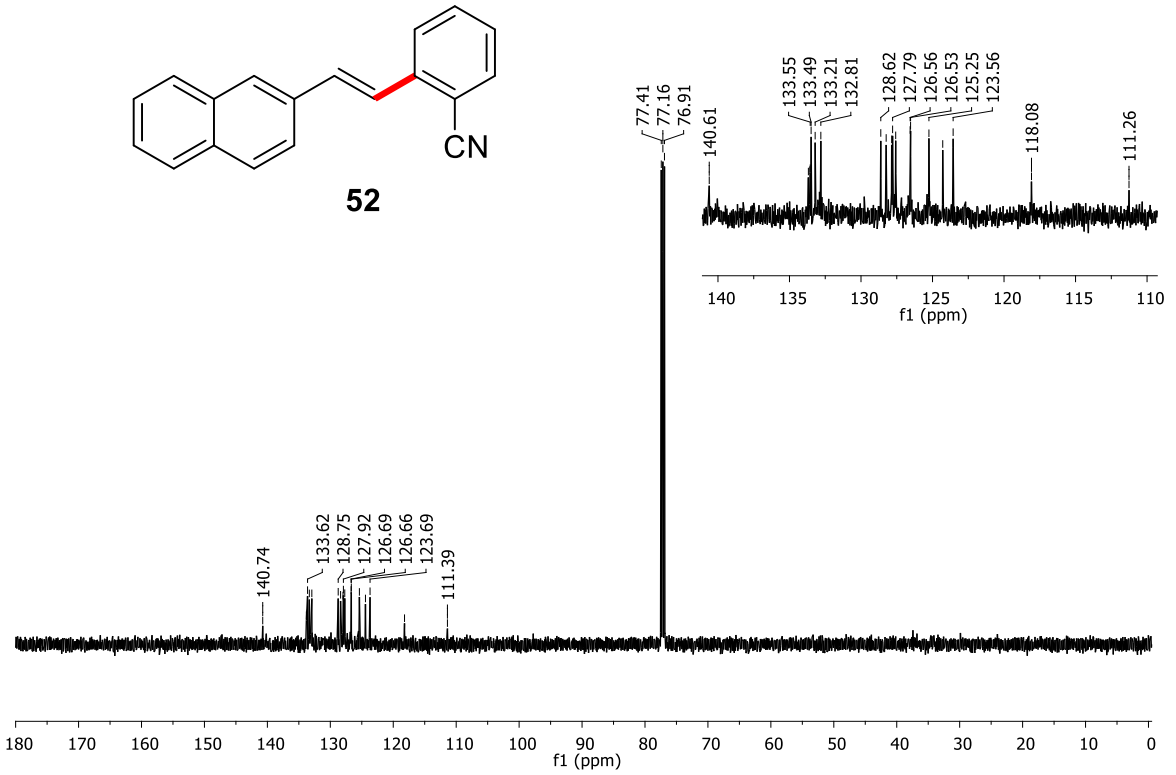

Fig. 129. ^{1}H NMR of (*E*)-2-(4-Nitrostyryl)naphthalene in $CDCl_3$ **(53)**[46].

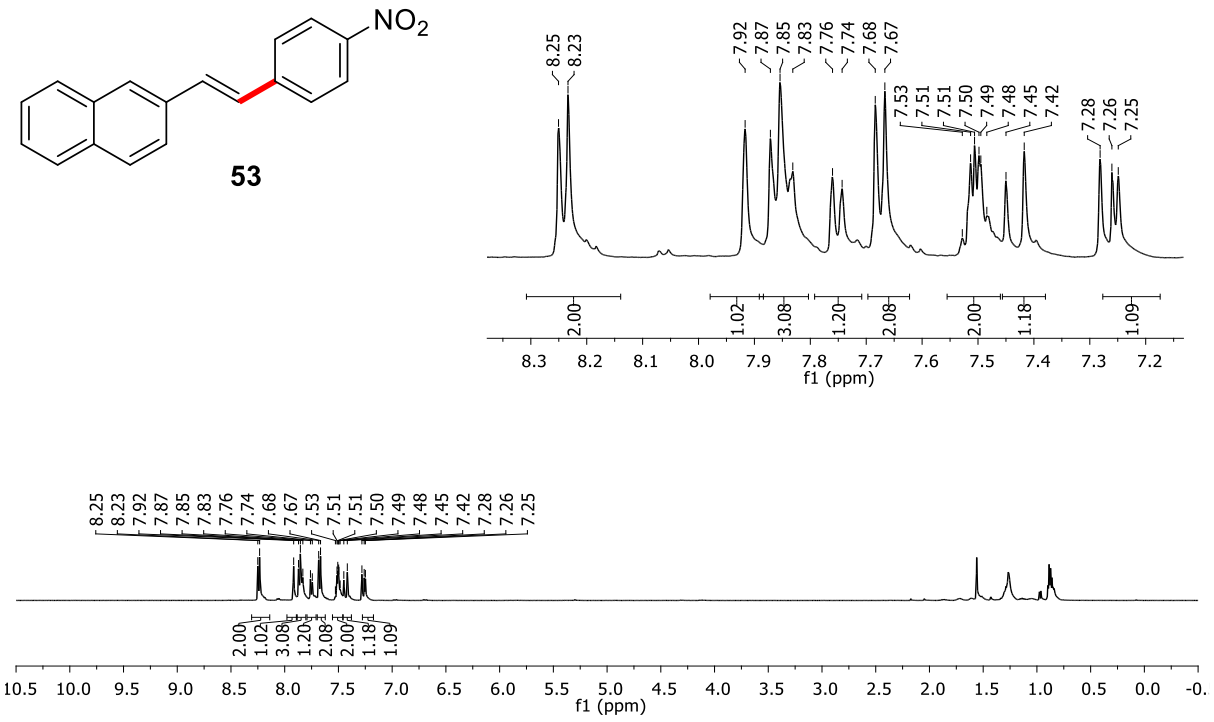

Fig. 130. ^{13}C NMR of (*E*)-2-(4-Nitrostyryl)naphthalene in $CDCl_3$ **(53)**[46].

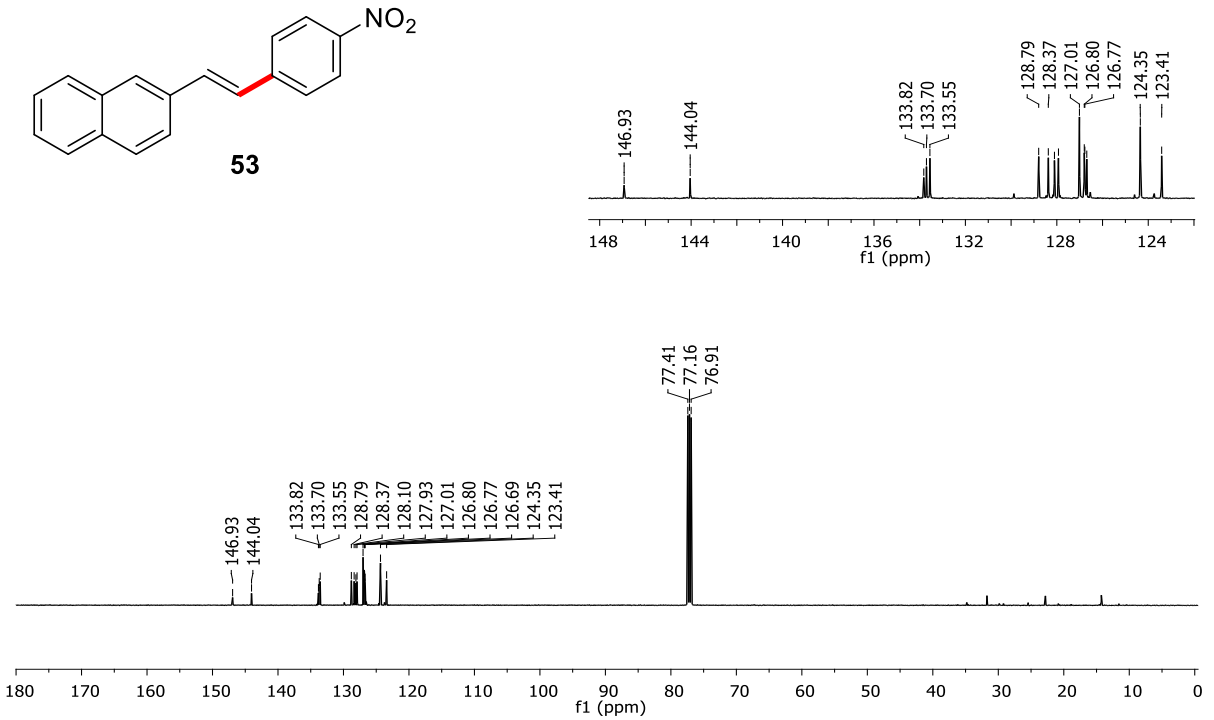

Fig. 131. [1] H NMR of (*E*)-2-(3,3,3-Trifluoroprop-1-en-1-yl)naphthalene in $CDCl_3$ **(54)**[47].

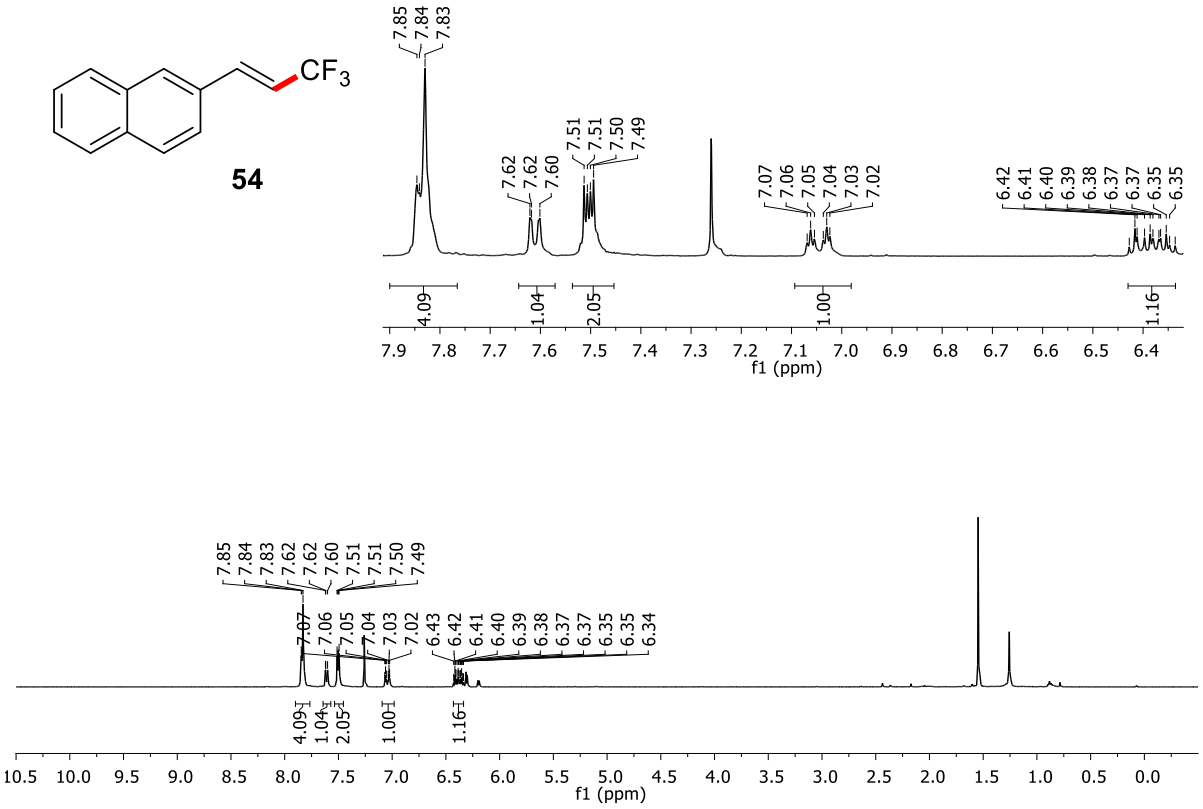

Fig. 132. [13]C NMR of (*E*)-2-(3,3,3-Trifluoroprop-1-en-1-yl)naphthalene in $CDCl_3$ **(54)**[47].

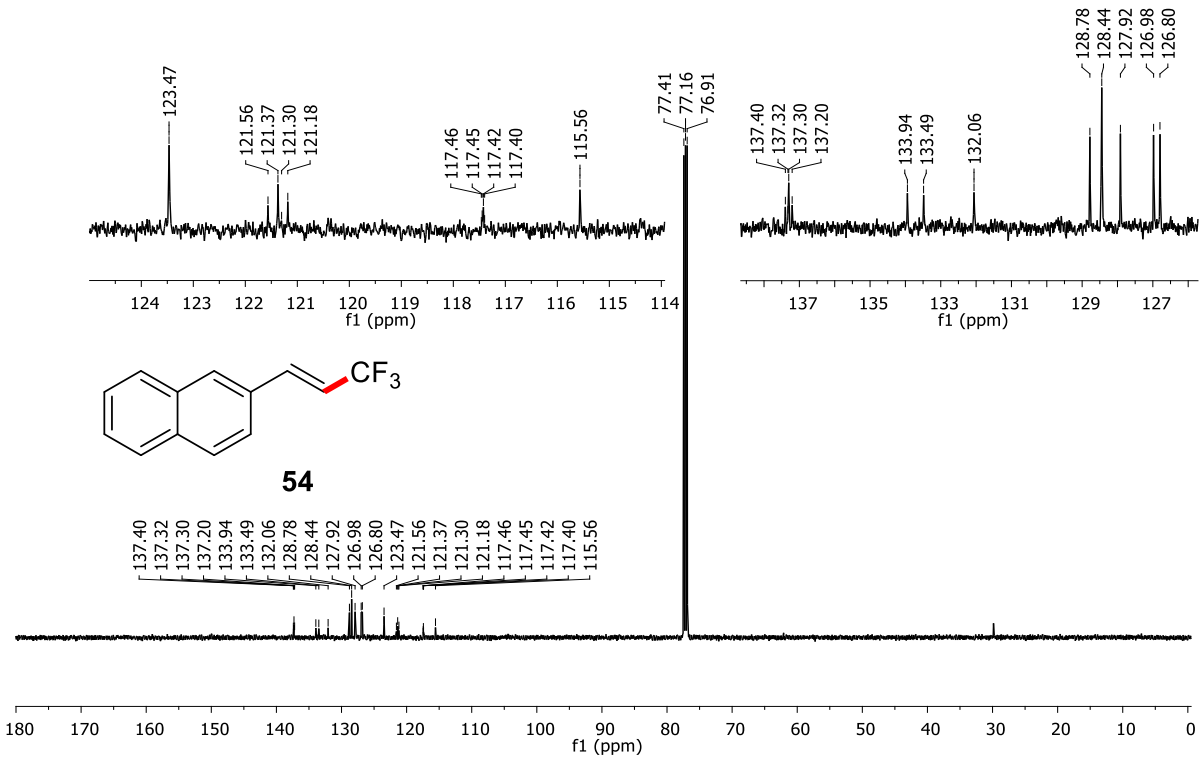

Fig. 133. ^{19}F NMR of (*E*)-2-(3,3,3-Trifluoroprop-1-en-1-yl)naphthalene in $CDCl_3$ **(54)**[47].

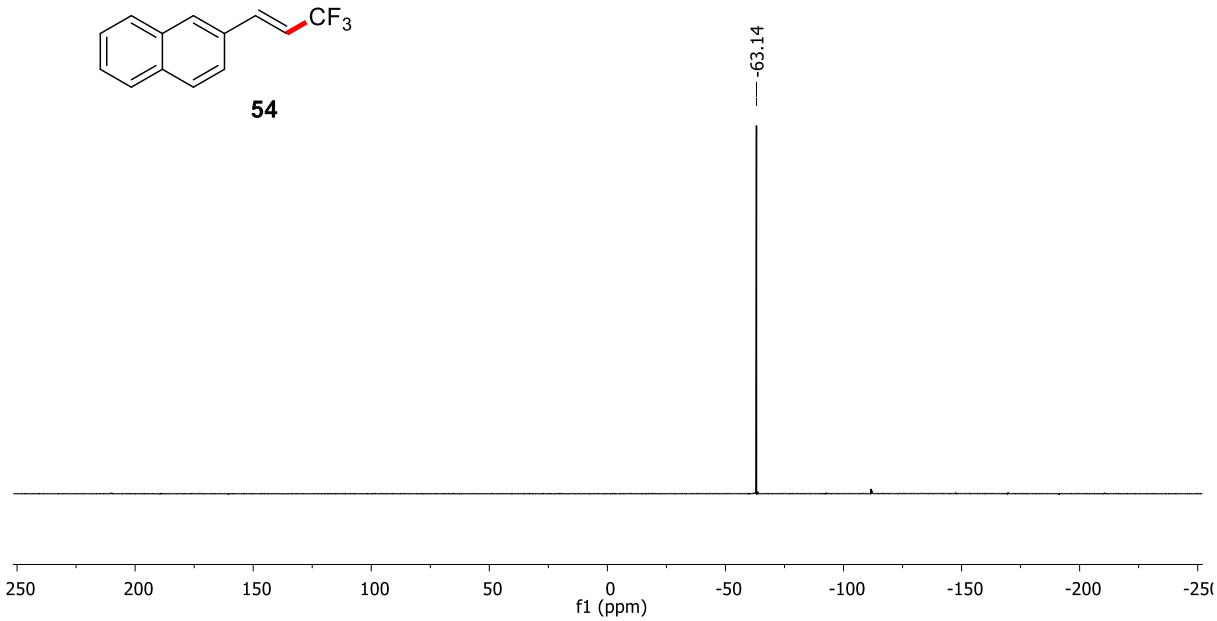

Fig. 134. ^{1}H NMR of 1,3,5-Trimethoxy-2-(trifluoromethyl)benzene in $CDCl_3$ **(55)**[47].

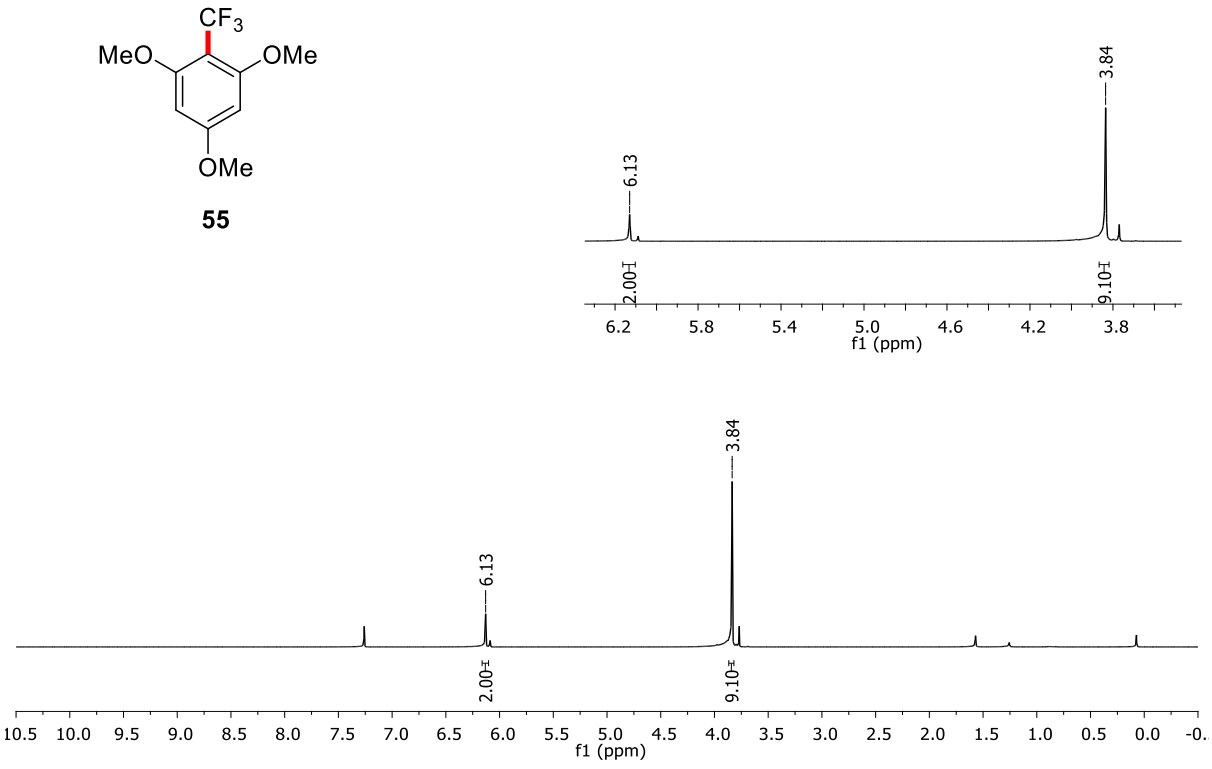

Fig. 135. ^{13}C NMR of 1,3,5-Trimethoxy-2-(trifluoromethyl)benzene in $CDCl_3$ **(55)**[48].

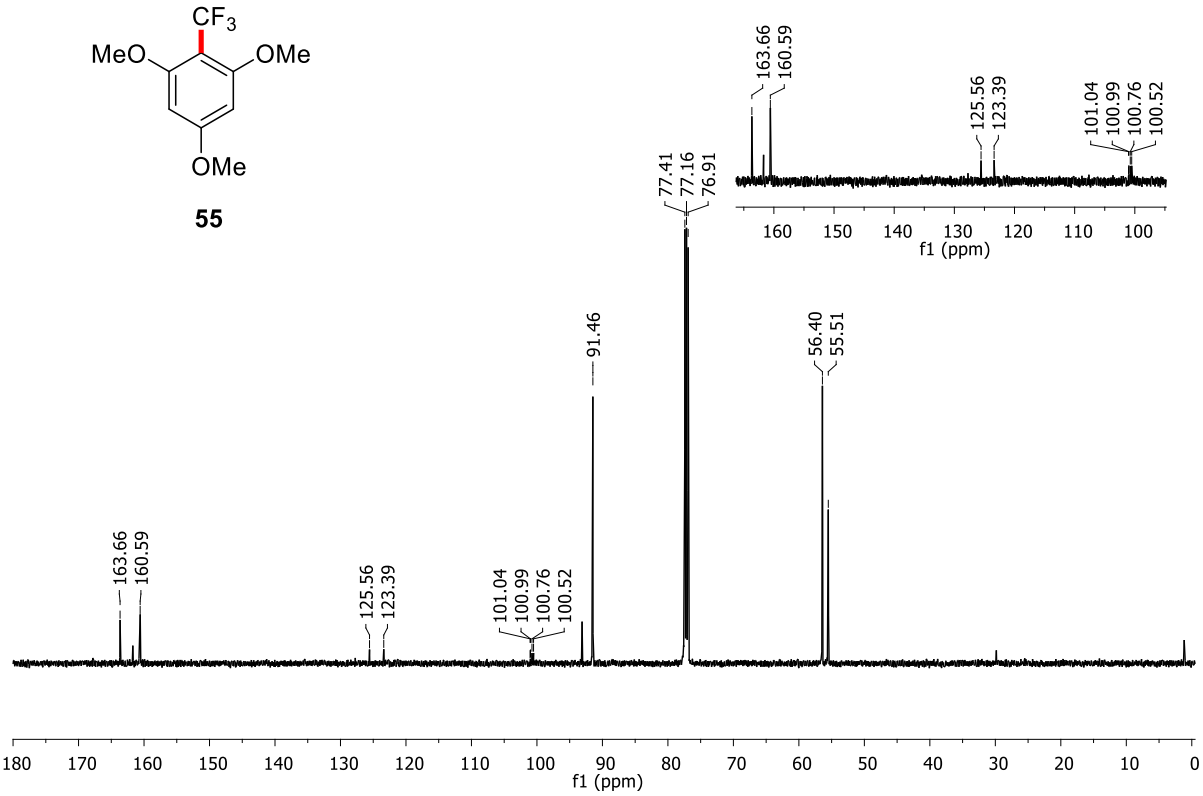

Fig. 136. ^{19}F NMR of 1,3,5-Trimethoxy-2-(trifluoromethyl)benzene in $CDCl_3$ **(55)**[48].

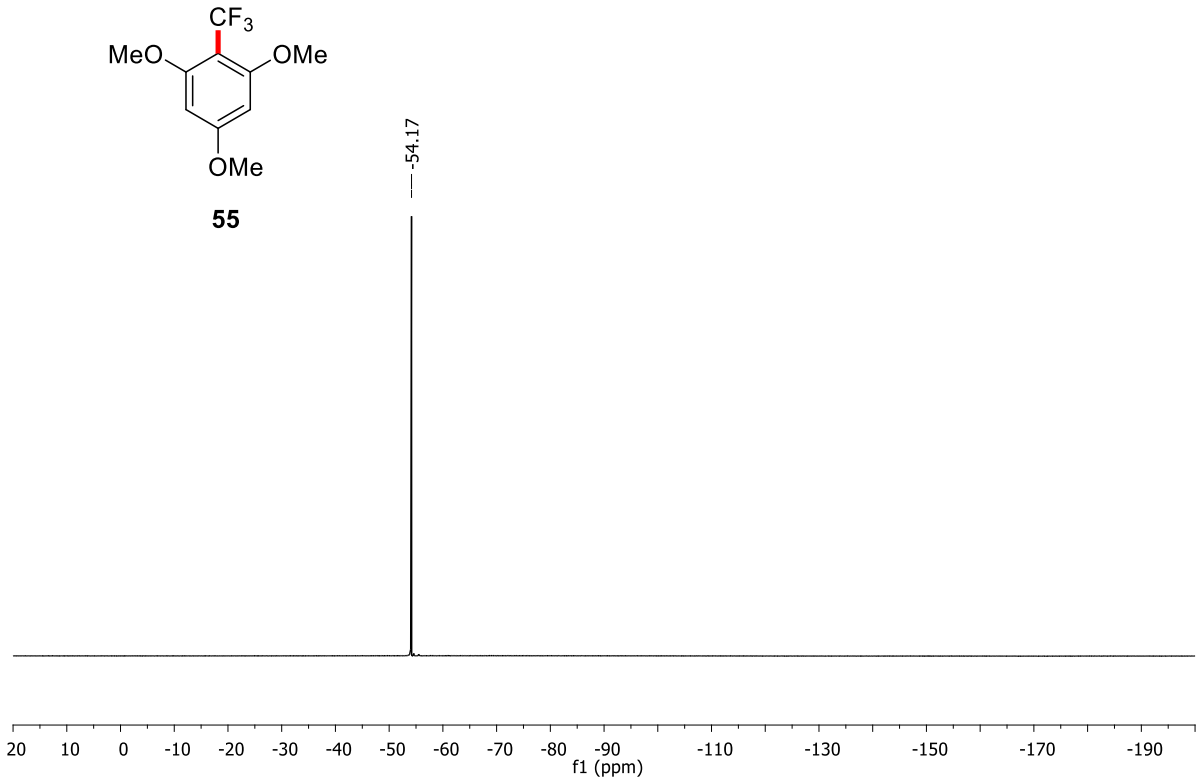

Fig. 137. [1] H NMR of 4, 4, 5, 5-Tetramethyl-2-(4-(trifluoromethyl)phenyl)-1,3,2-dioxaborolane in $CDCl_3$ **(56)**[49].

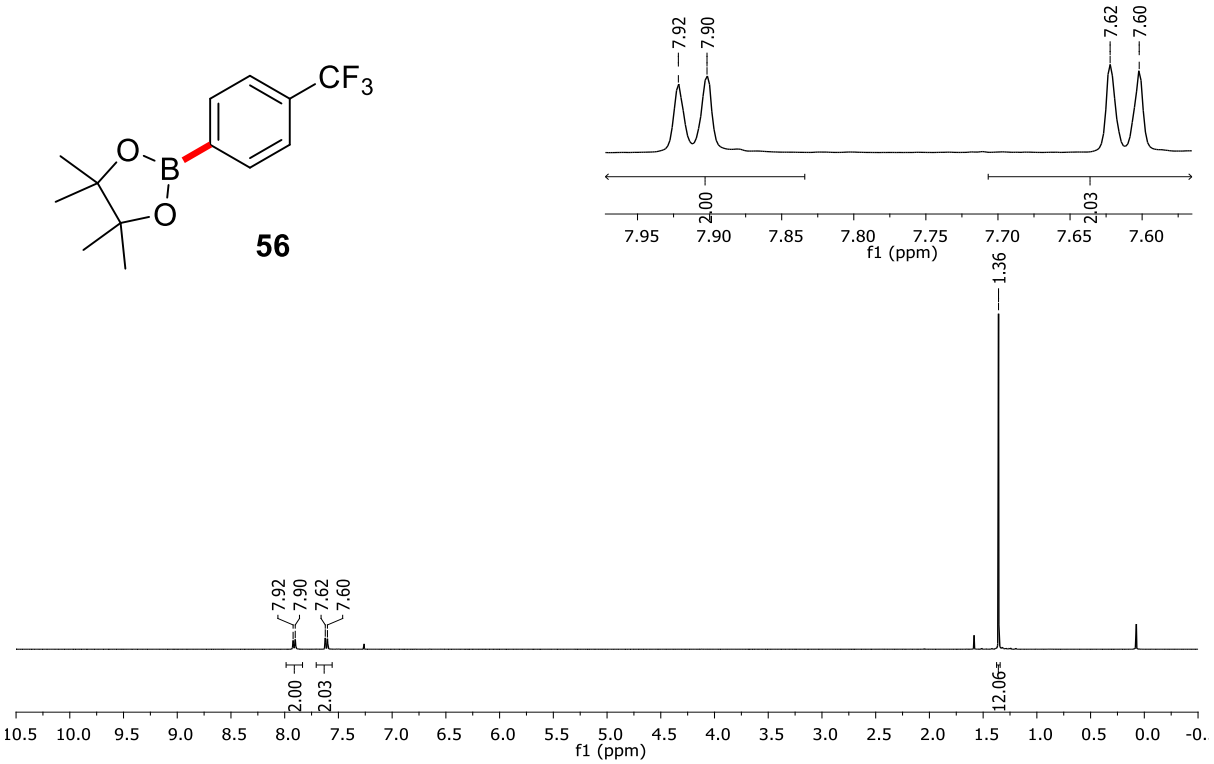

Fig. 138. [13]C NMR of 4, 4, 5, 5-Tetramethyl-2-(4-(trifluoromethyl)phenyl)-1,3,2-dioxaborolane in $CDCl_3$ **(56)**[49].

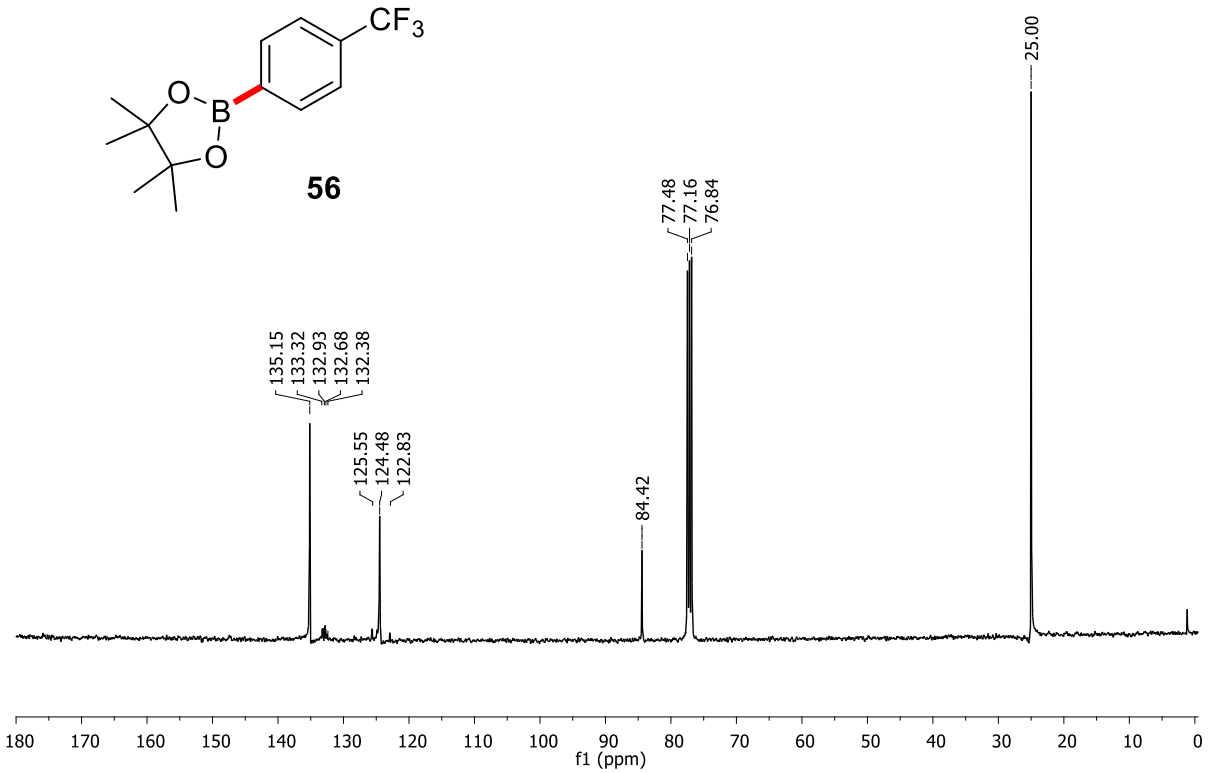

Fig. 139. ^{11}B NMR of 4, 4, 5, 5-Tetramethyl-2-(4-(trifluoromethyl)phenyl)-1,3,2-dioxaborolane in $CDCl_3$ **(56)**[49].

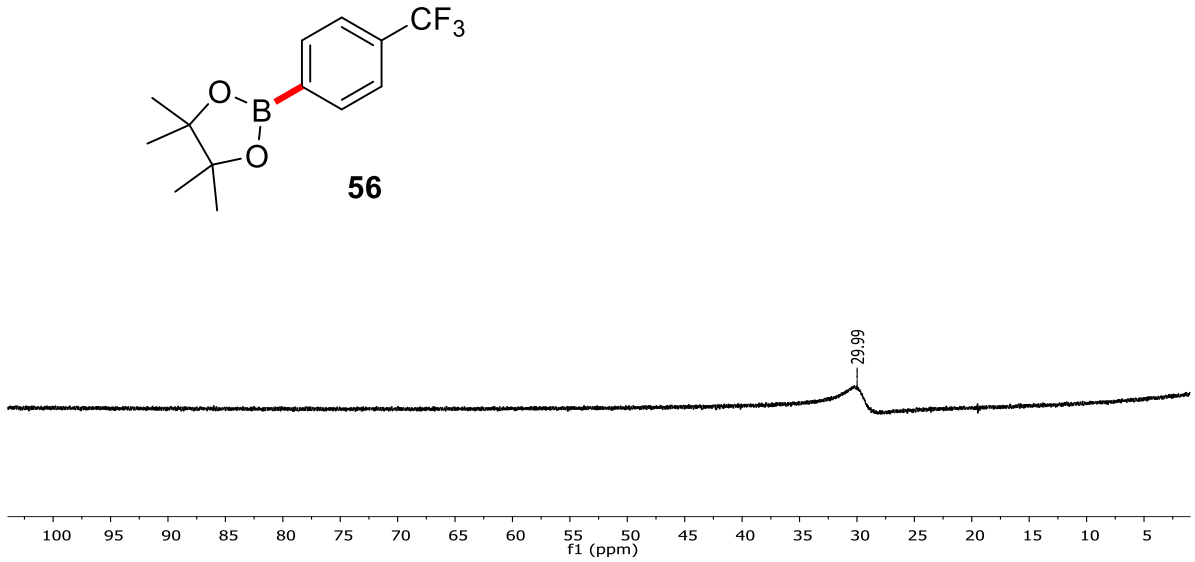

Fig. 140. ^{19}F NMR of 4, 4, 5, 5-Tetramethyl-2-(4-(trifluoromethyl)phenyl)-1,3,2-dioxaborolane in $CDCl_3$ **(56)**[49].

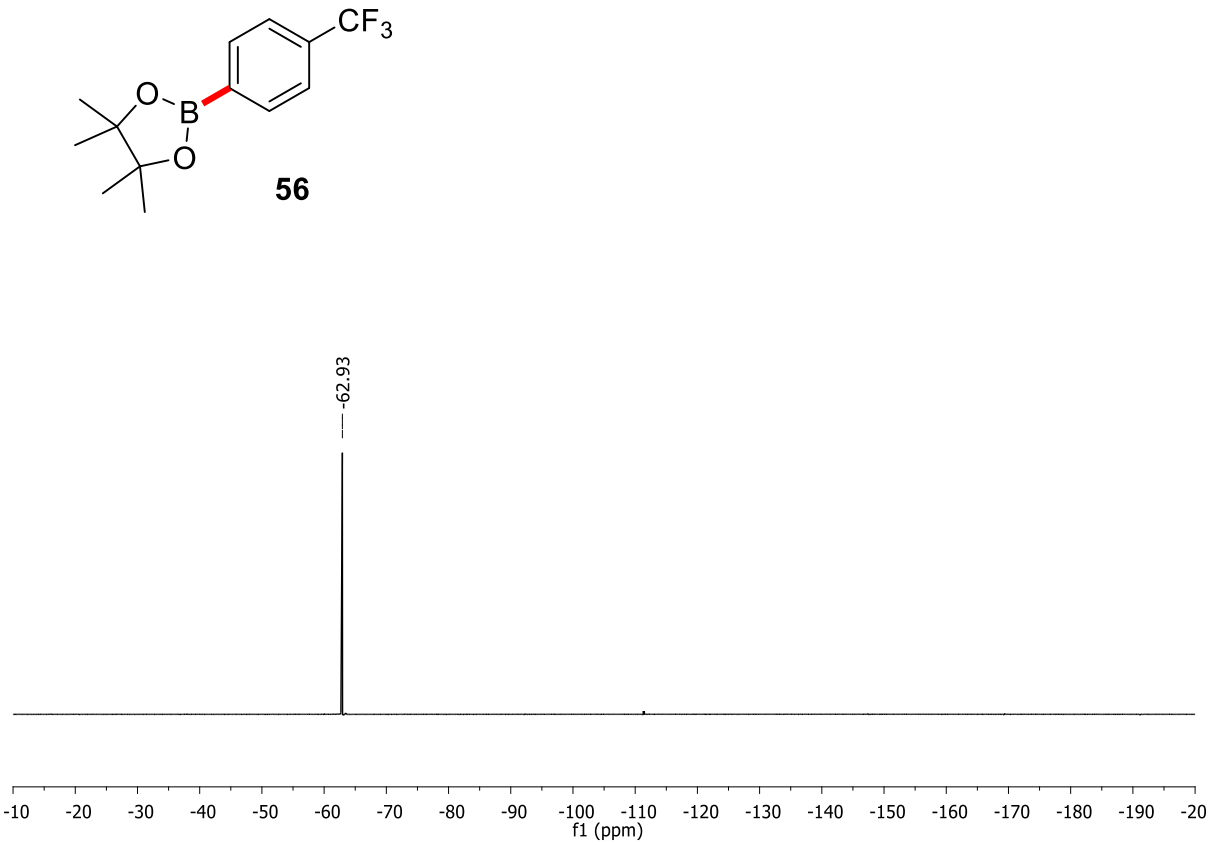

Fig. 141. ^{1}H NMR of 1-Methyl-2-phenyl-1*H*-pyrrole in $CDCl_3$ (**57**)[50]

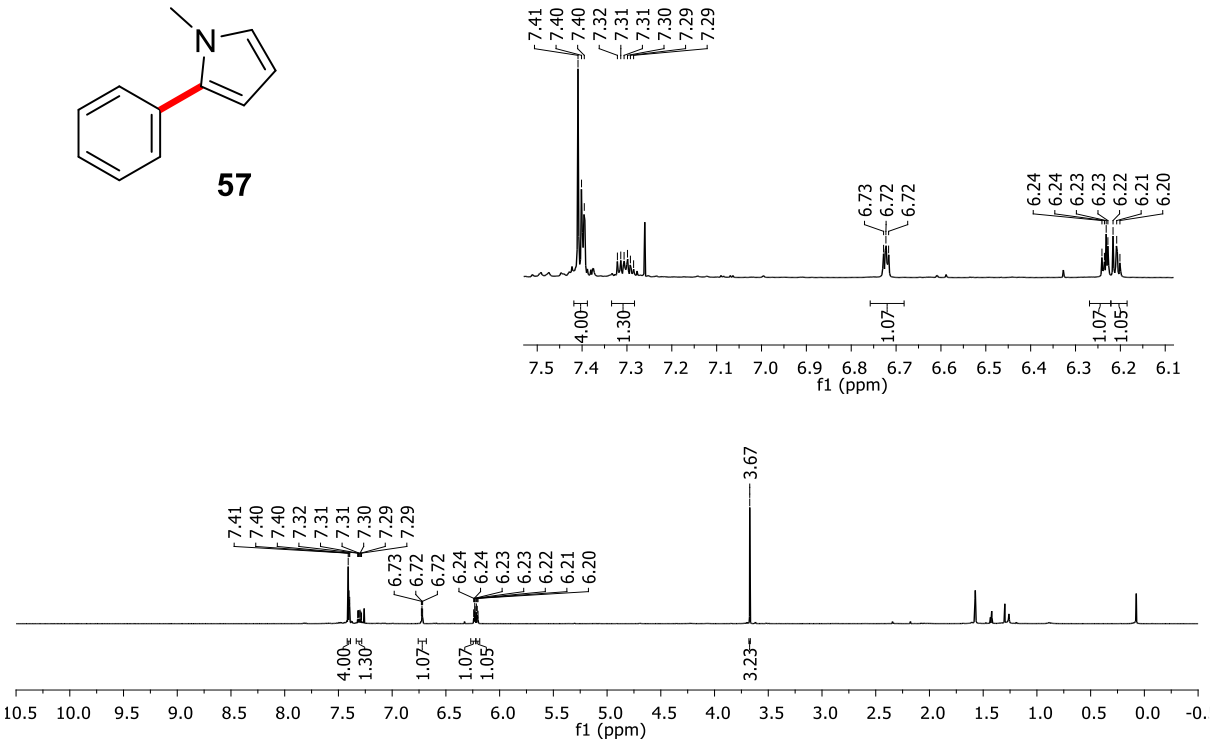

Fig. 142. ^{13}C NMR of 1-Methyl-2-phenyl-1*H*-pyrrole in $CDCl_3$ (**57**)[50]

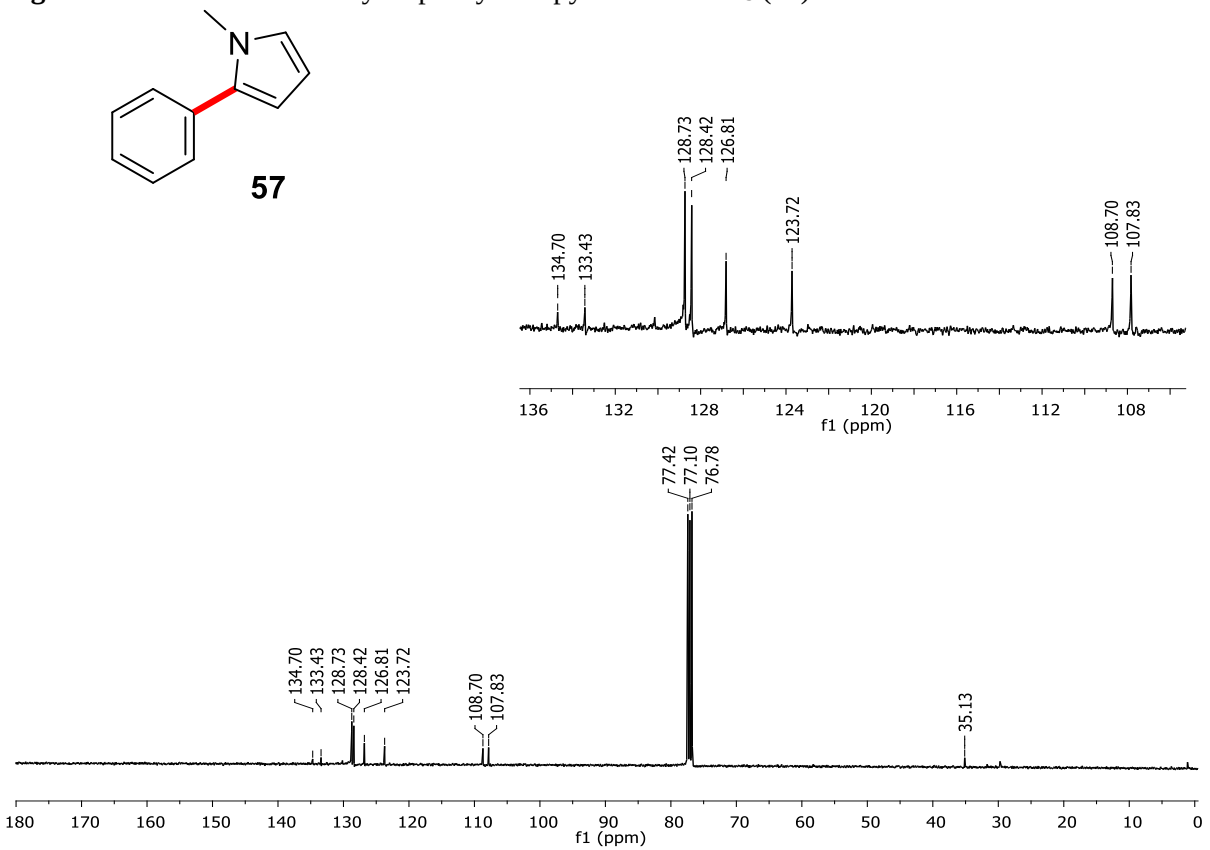

Fig. 143. ^{1}H NMR of 2-(4-Fluorophenyl)-1-methyl-1H-pyrrole in $CDCl_3$ **(58)**[51]

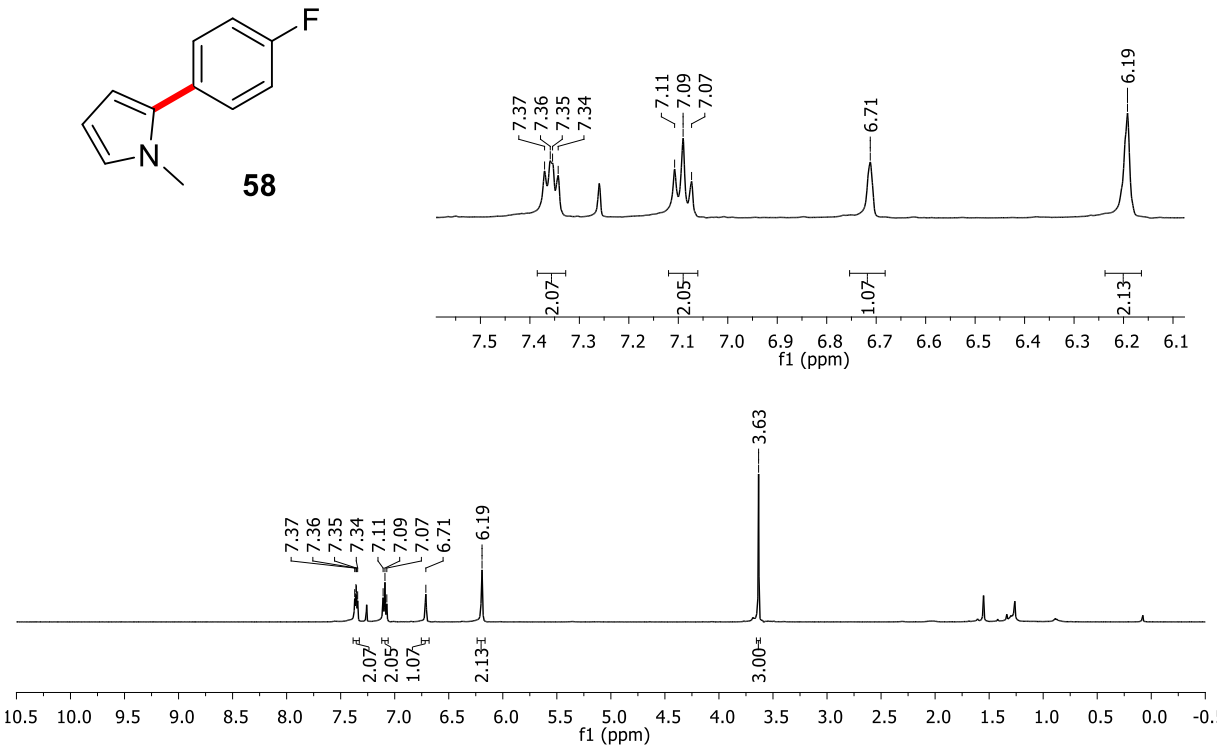

Fig. 144. ^{13}C NMR of 2-(4-Fluorophenyl)-1-methyl-1H-pyrrole in $CDCl_3$ **(58)**[51]

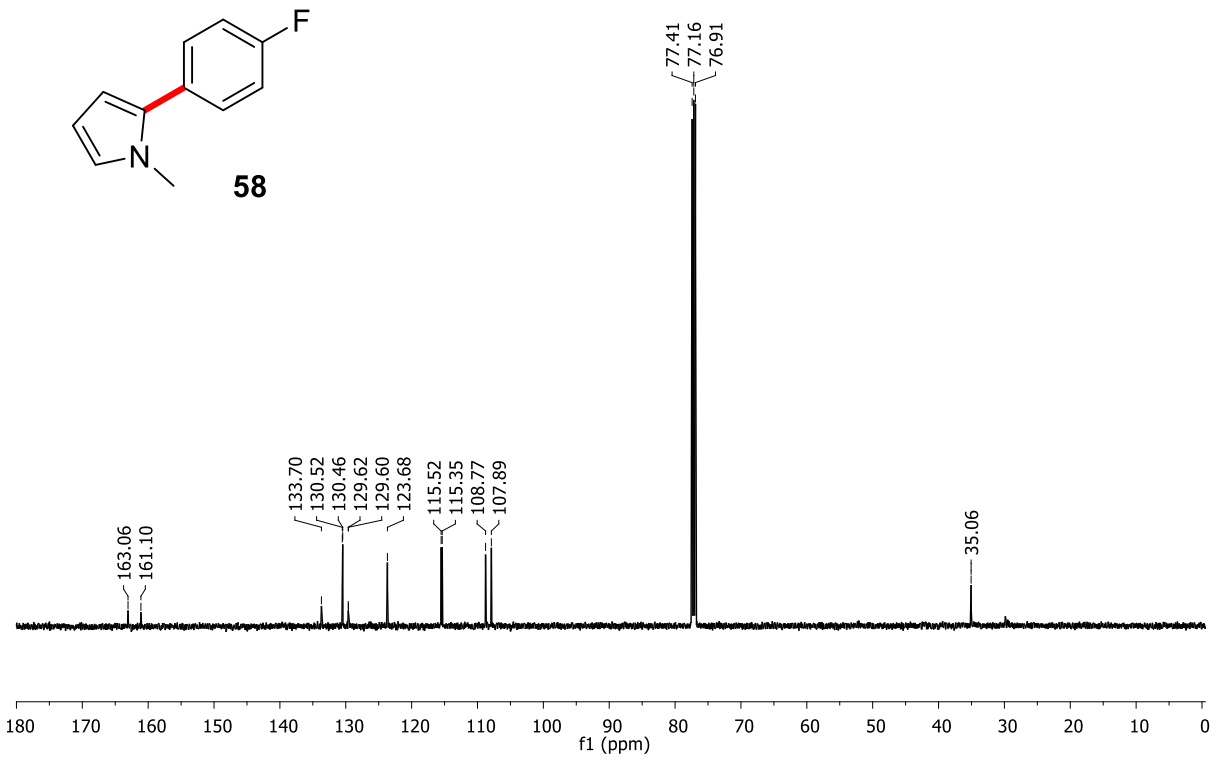

Fig. 145. [1]H NMR of 1-Methyl-2-(4-(trifluoromethyl)phenyl)-1*H*-pyrrole **(59)**[52]

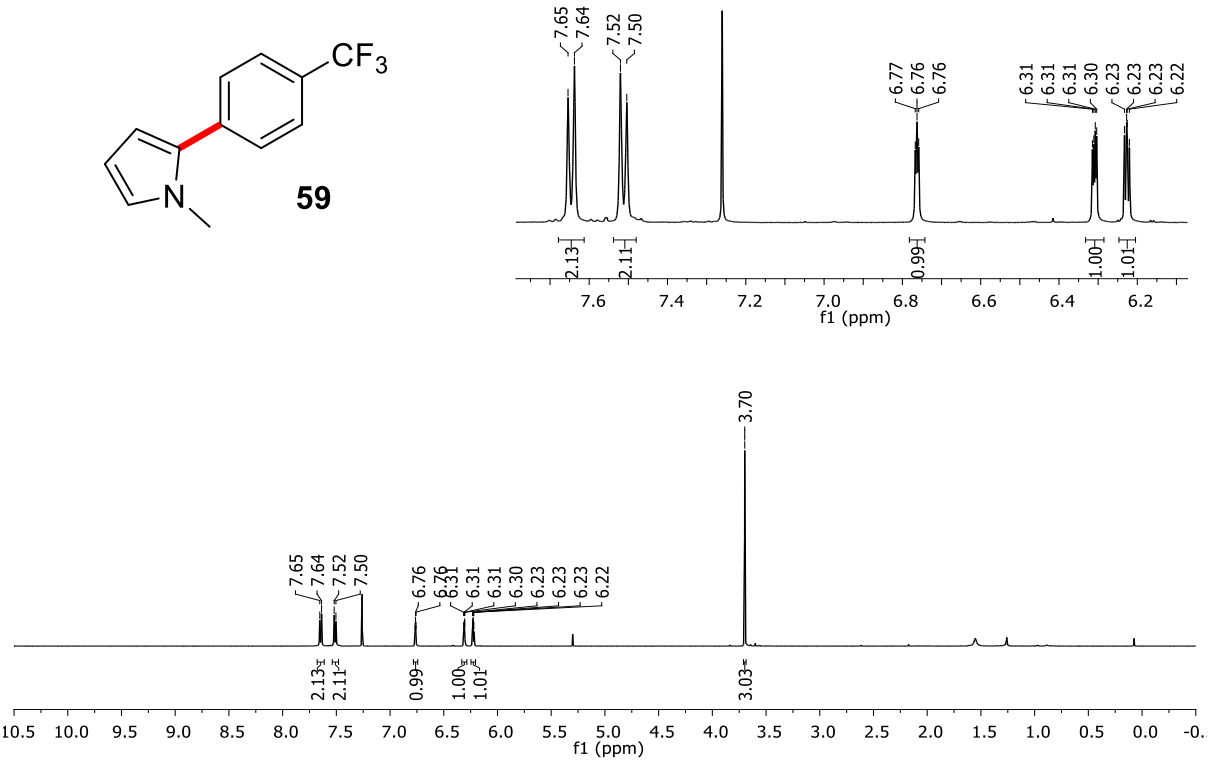

Fig. 146. [13]C NMR of 1-Methyl-2-(4-(trifluoromethyl) phenyl)-1*H*-pyrrole **(4)**[52]

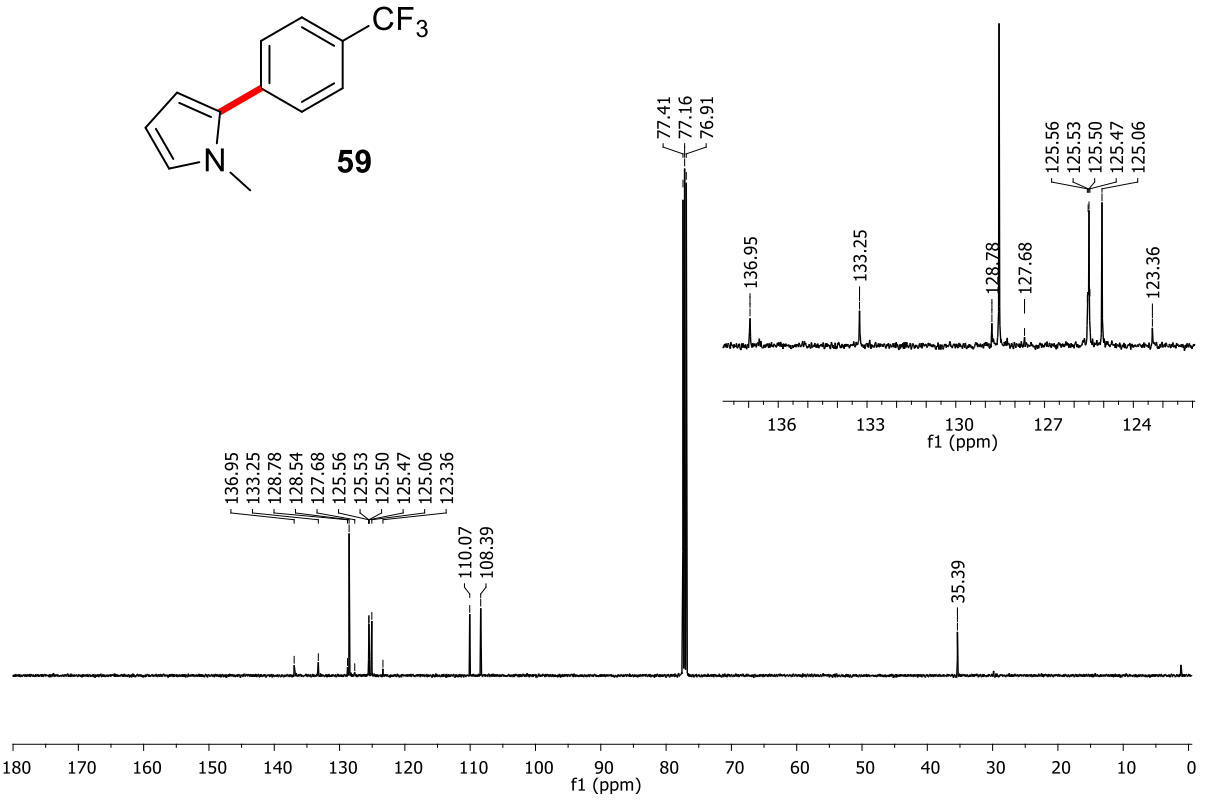

Fig. 147. [1]H NMR of 2-(3-Fluorophenyl)-1-methyl-1*H*-pyrrole **(60)**[53]

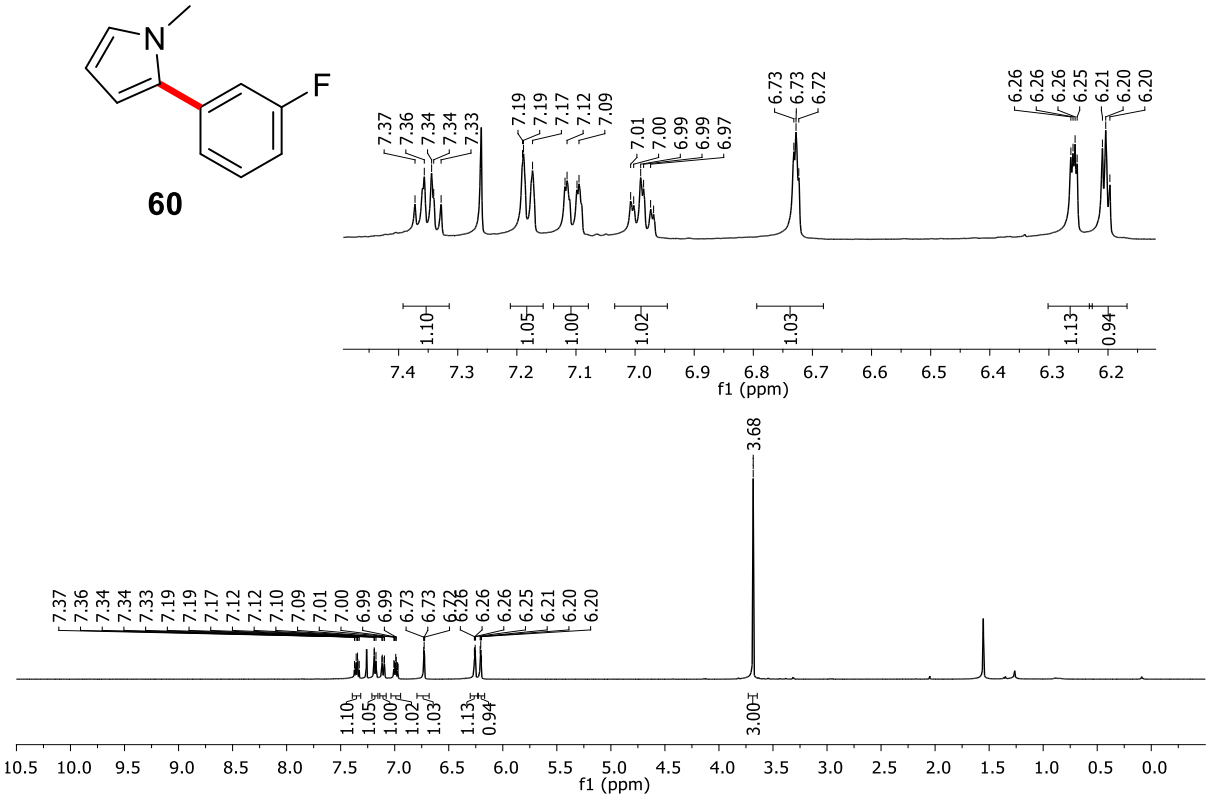

Fig. 148. [13]C NMR of 2-(3-Fluorophenyl)-1-methyl-1*H*-pyrrole in $CDCl_3$ **(60)**[53].

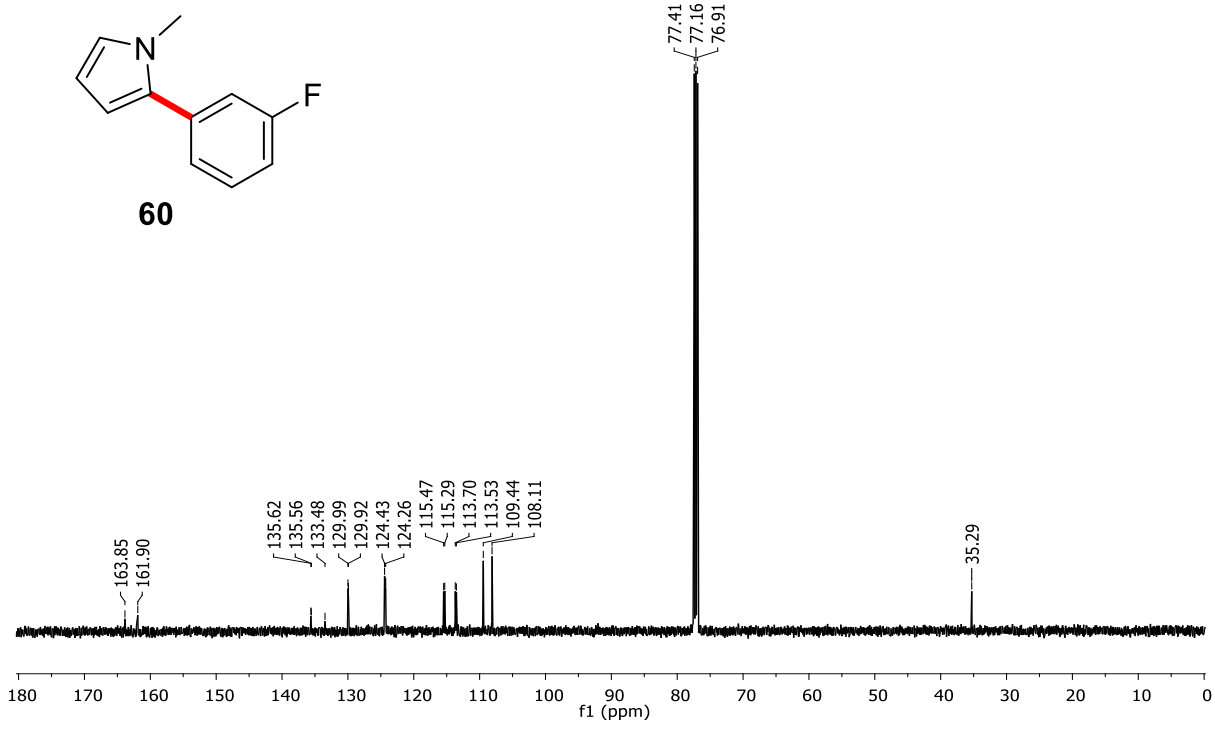

Fig. 149. ^{1}H NMR of 2-Phenylfuran in $CDCl_3$ **(61)**[54].

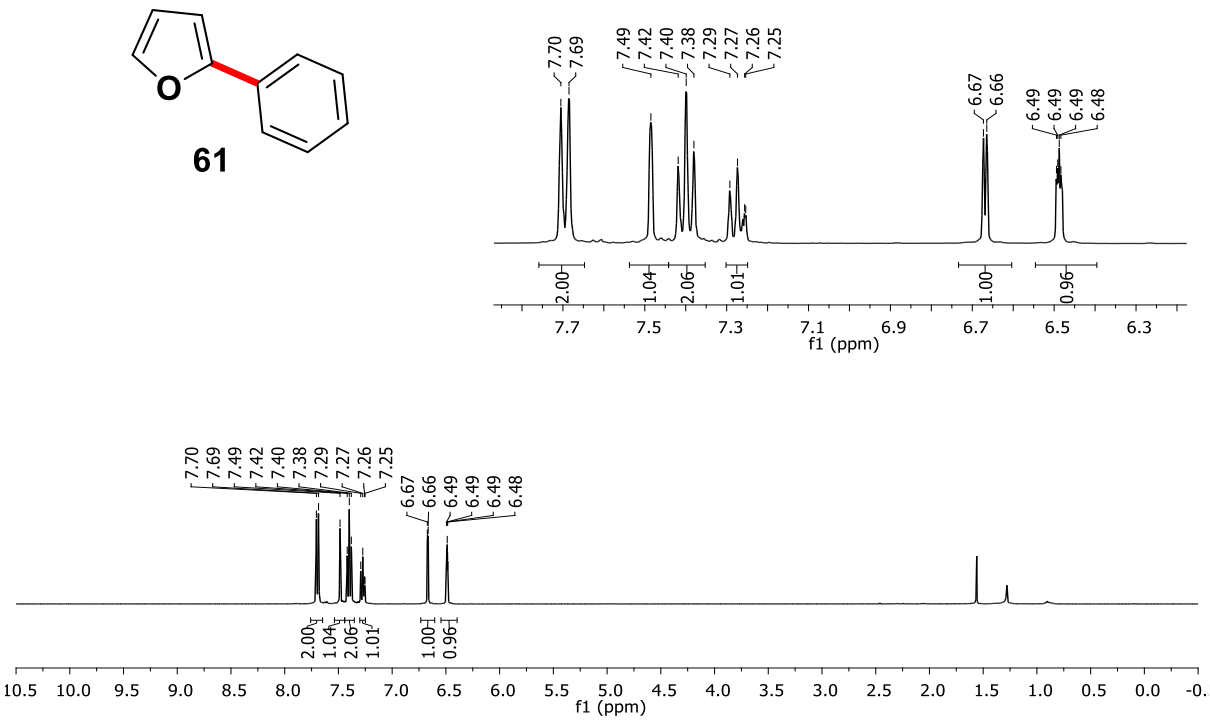

Fig. 150. ^{13}C NMR of 2-Phenylfuran in $CDCl_3$ **(61)**[54].

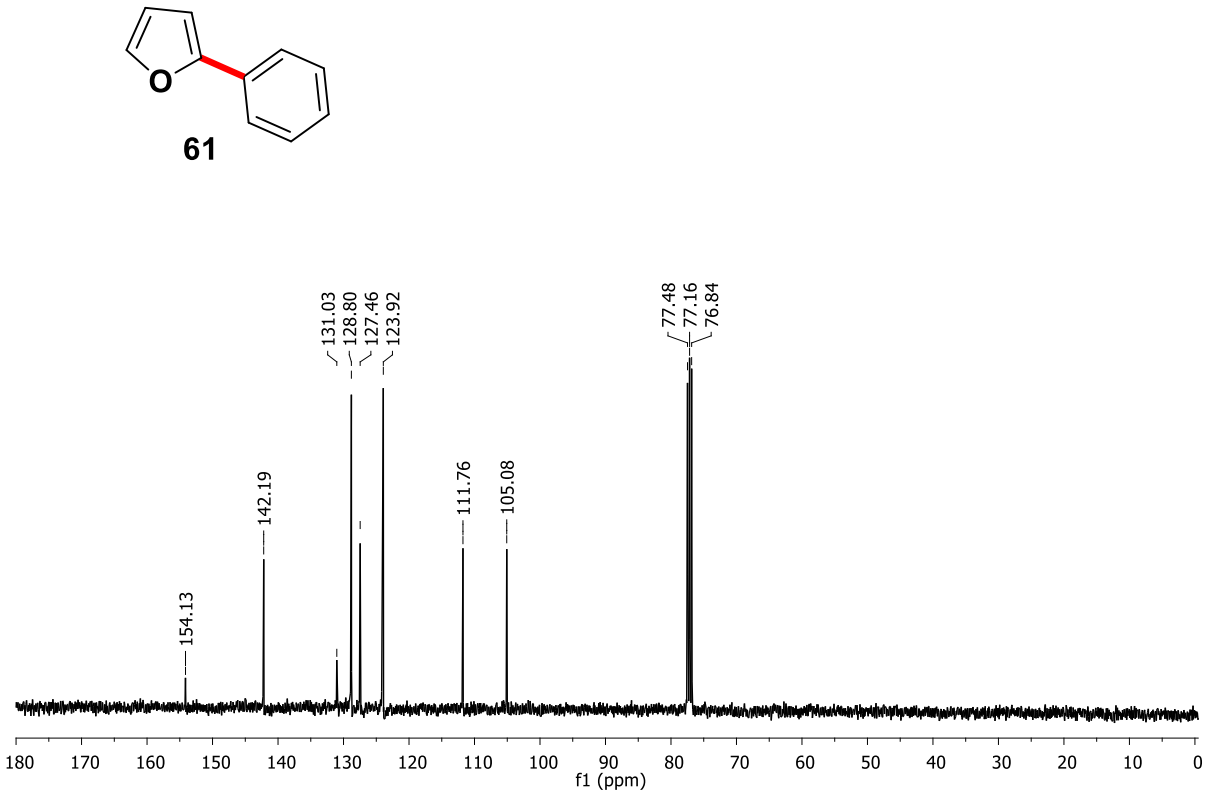

Fig. 151. [1]H NMR of 2-(3, 5-Dimethylphenyl) furan in $CDCl_3$**(62)**[55].

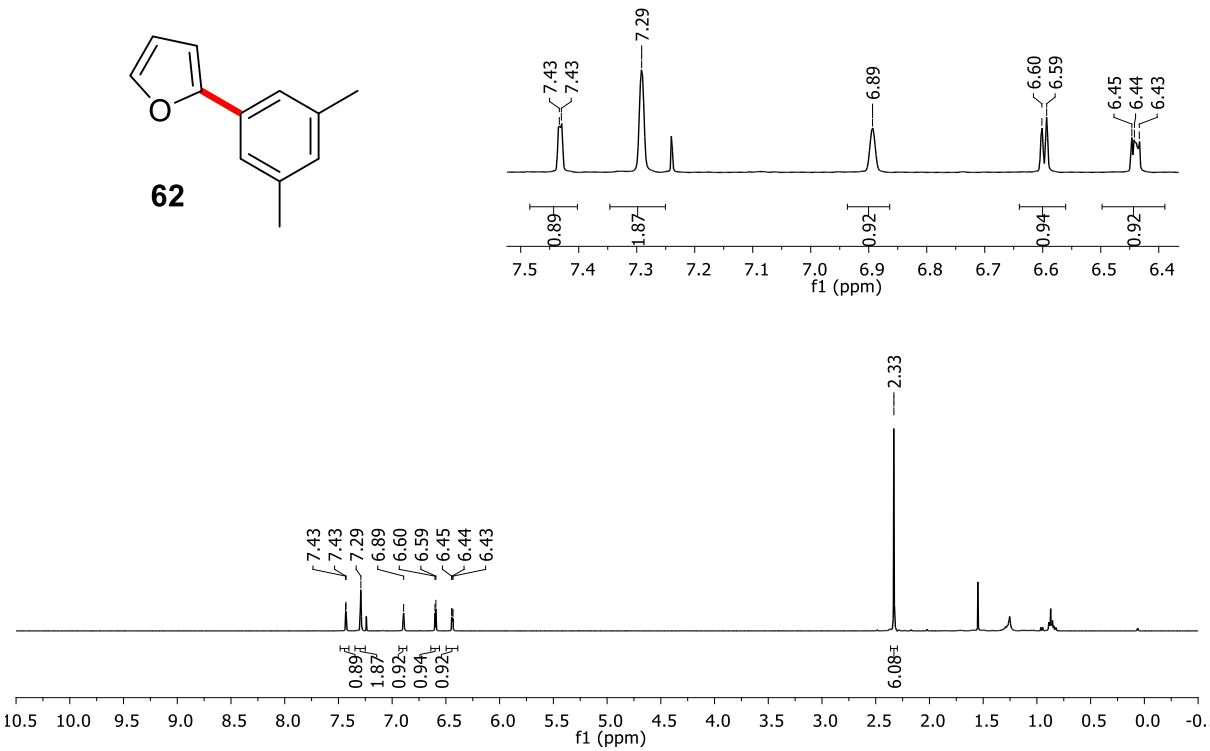

Fig. 152. [13]C NMR of 2-(3, 5-Dimethylphenyl) furan in $CDCl_3$ **(62)**[55].

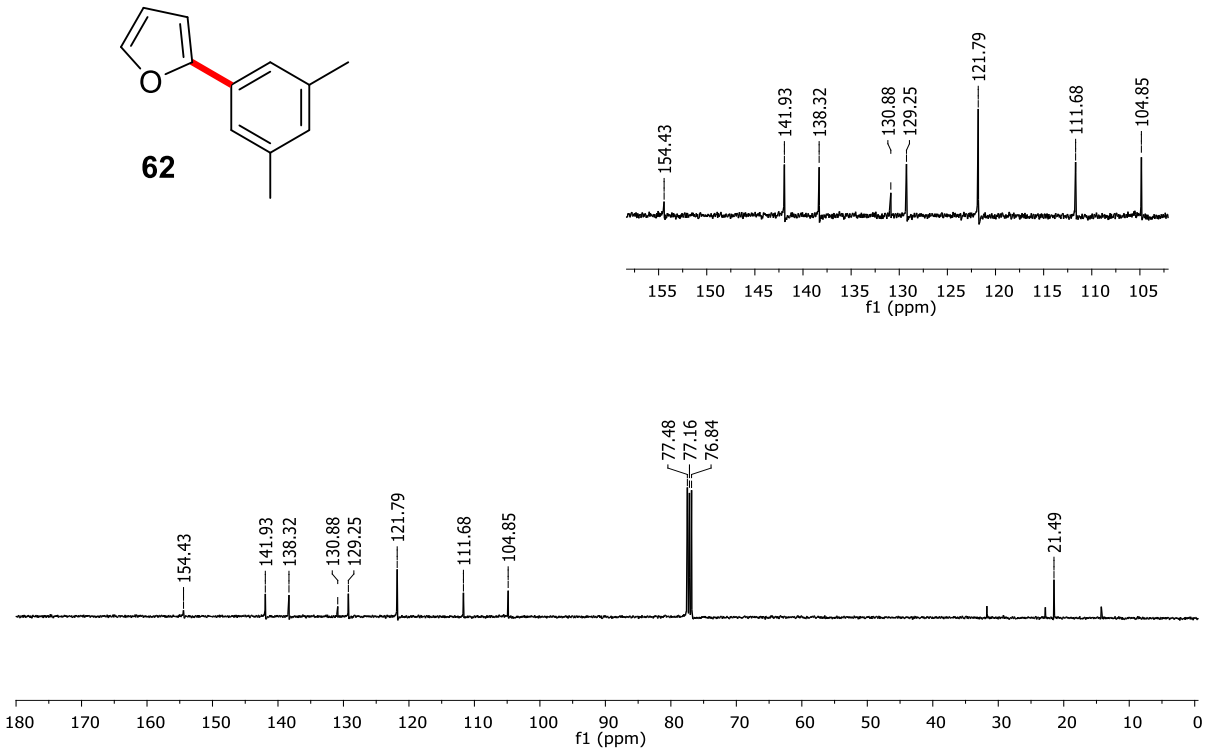

Fig. 153. ^{1}H NMR of 2-(3,5-Dimethylphenyl)thiazole in $CDCl_3$**(63)**[56].

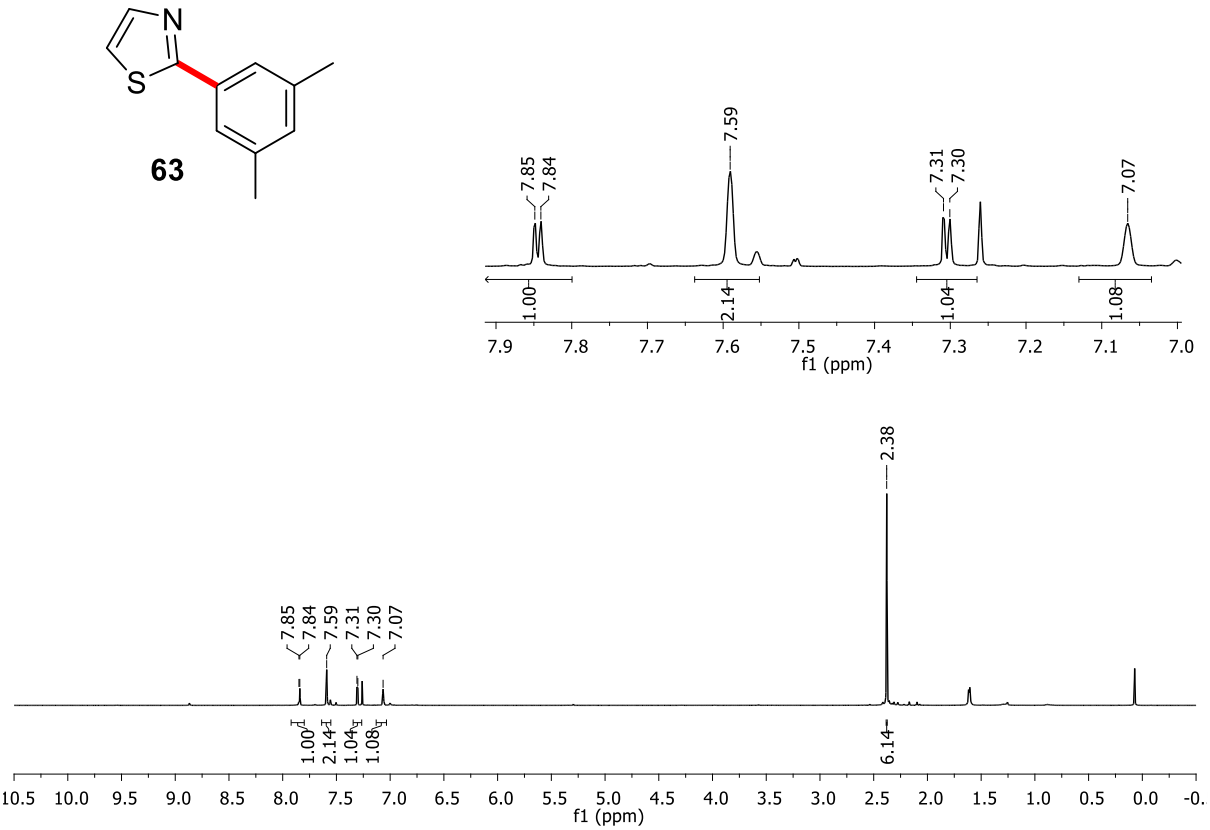

Fig. 154. ^{13}C NMR of 2-(3,5-Dimethylphenyl)thiazole in $CDCl_3$**(63)**[56].

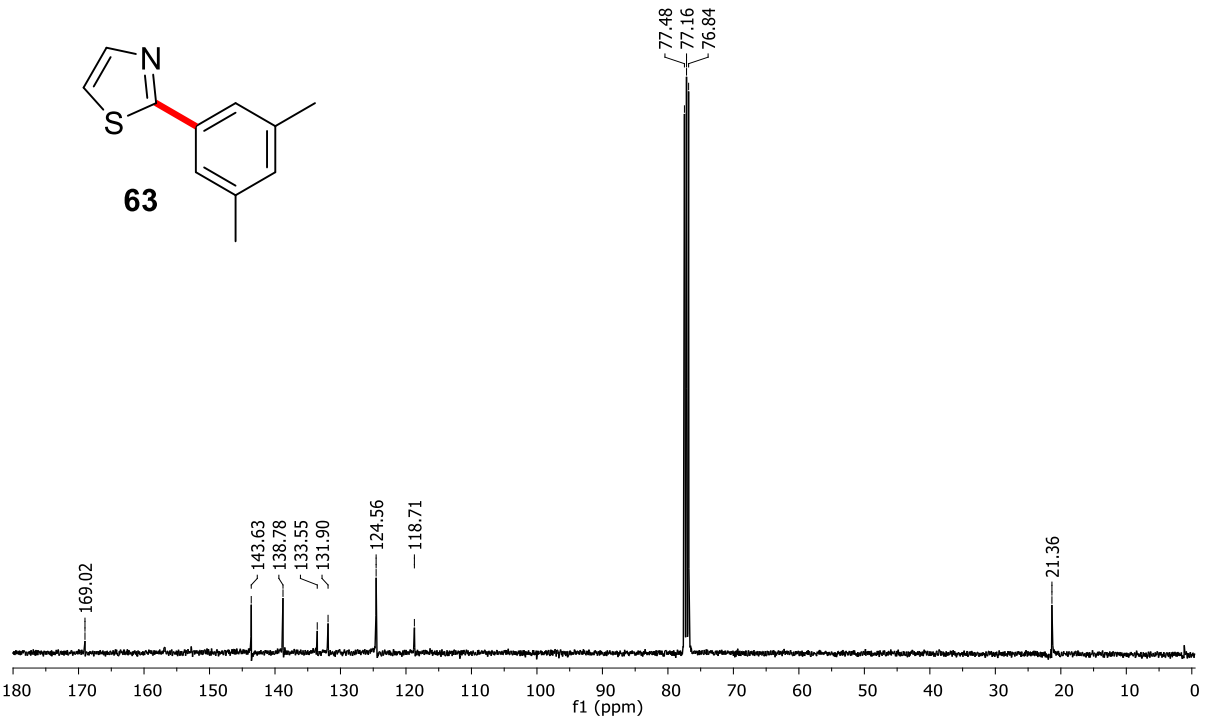

Fig. 155. ^{1}H NMR of 2-Phenylthiazole in $CDCl_3$ **(64)**[56].

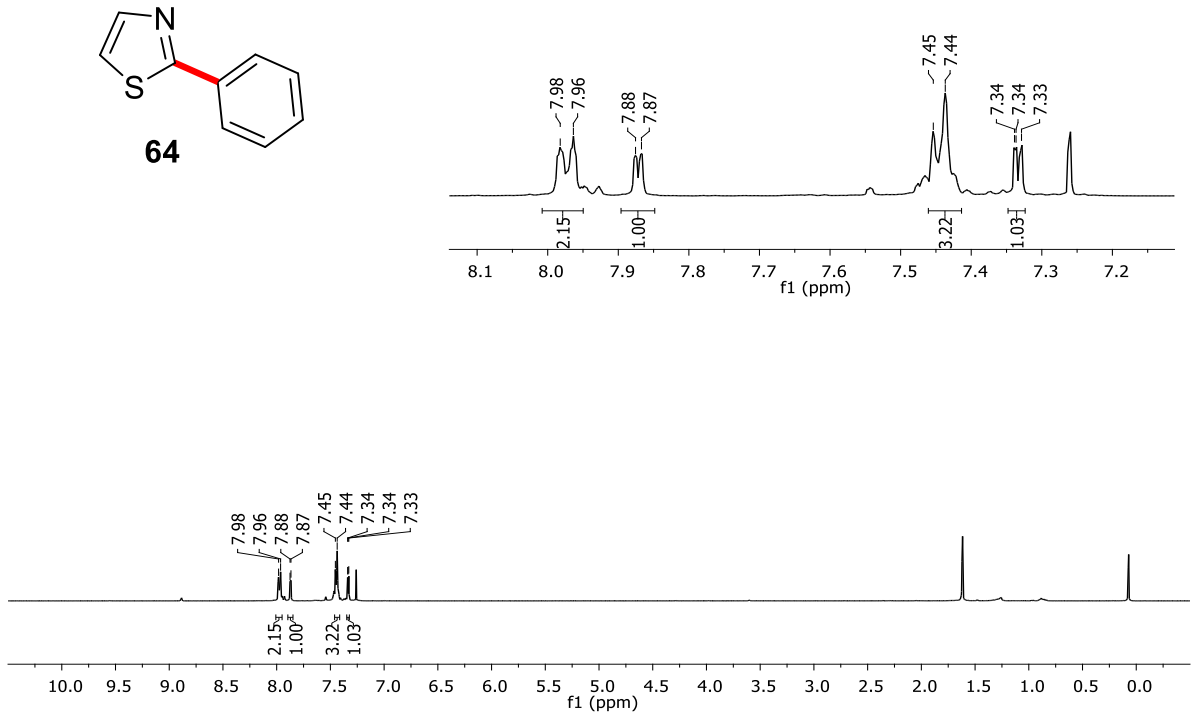

Fig. 156. ^{13}C NMR of 2-Phenylthiazole in $CDCl_3$ **(64)**[56].

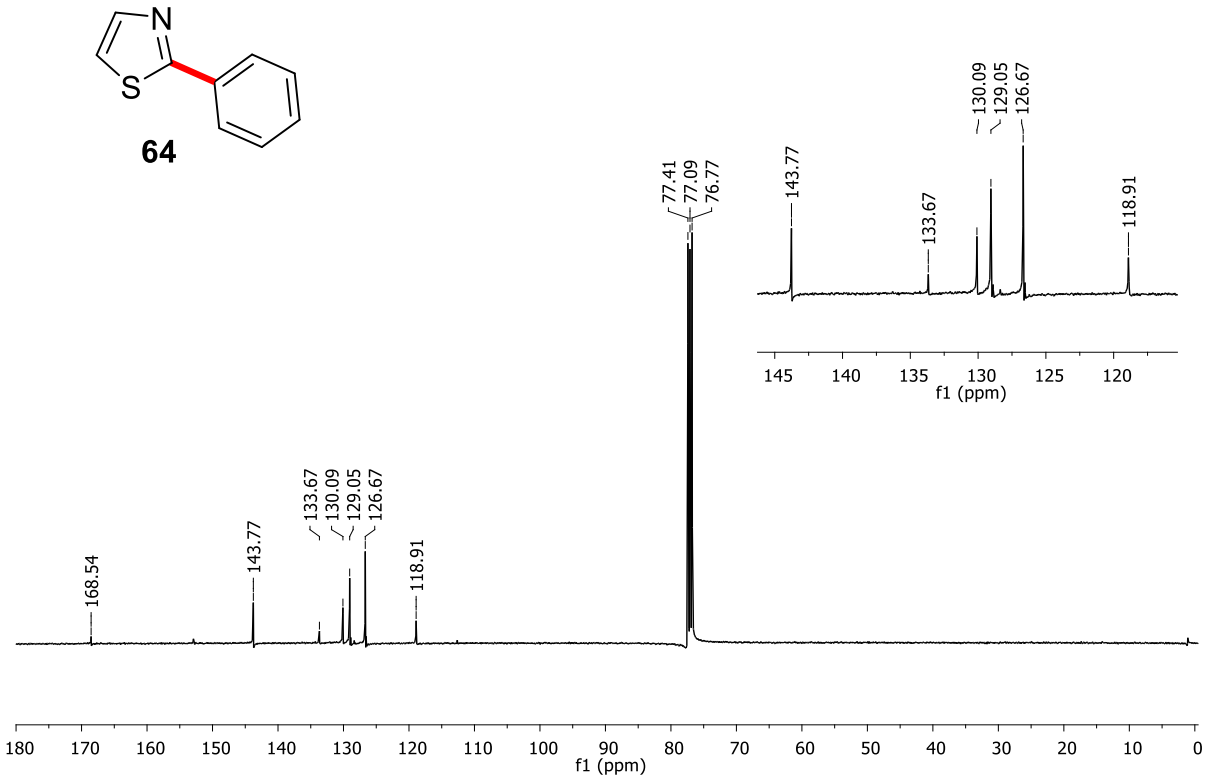

Fig. 157. ^{1}H NMR of 2-Phenylbenzofuran in $CDCl_3$ **(65)**[57].

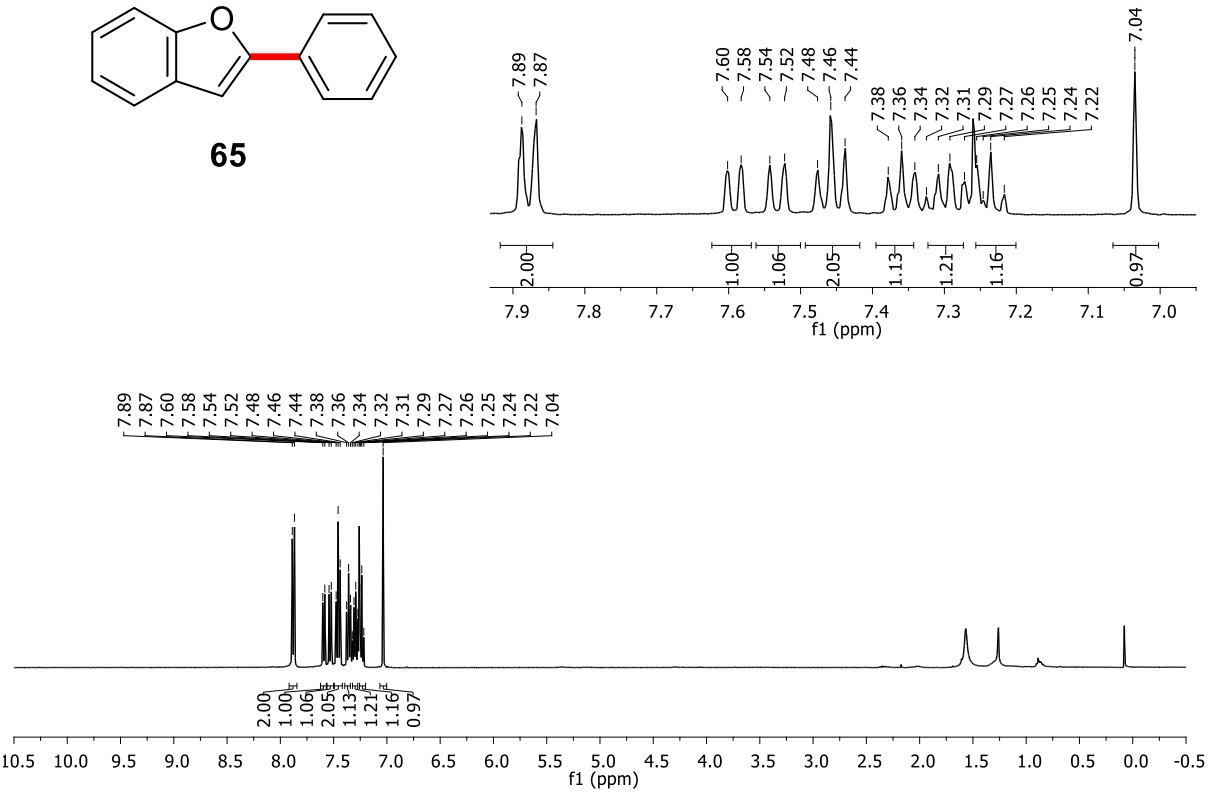

Fig. 158. ^{13}C NMR of 2-Phenylbenzofuran in $CDCl_3$ **(65)**[57].

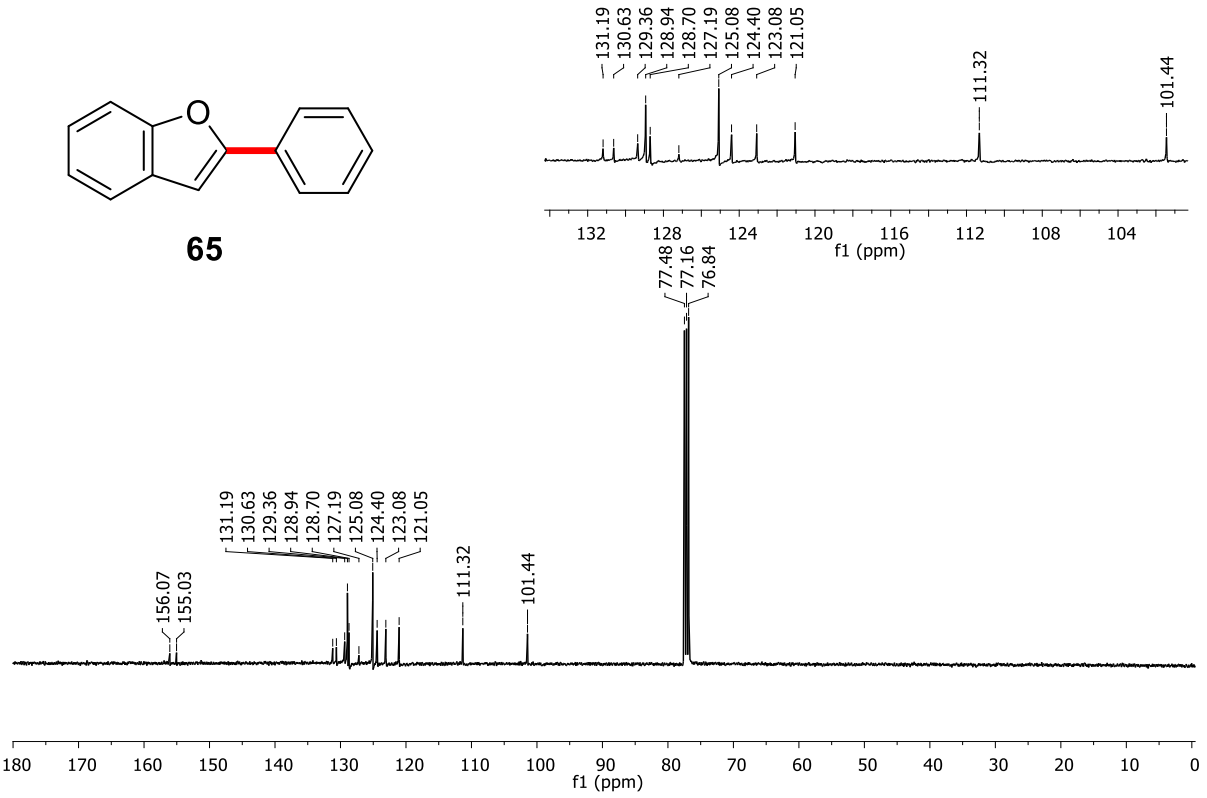

Fig. 159. ^{1}H NMR of 4-Fluoro-1,1'-biphenyl in $CDCl_3$ **(66)**[58].

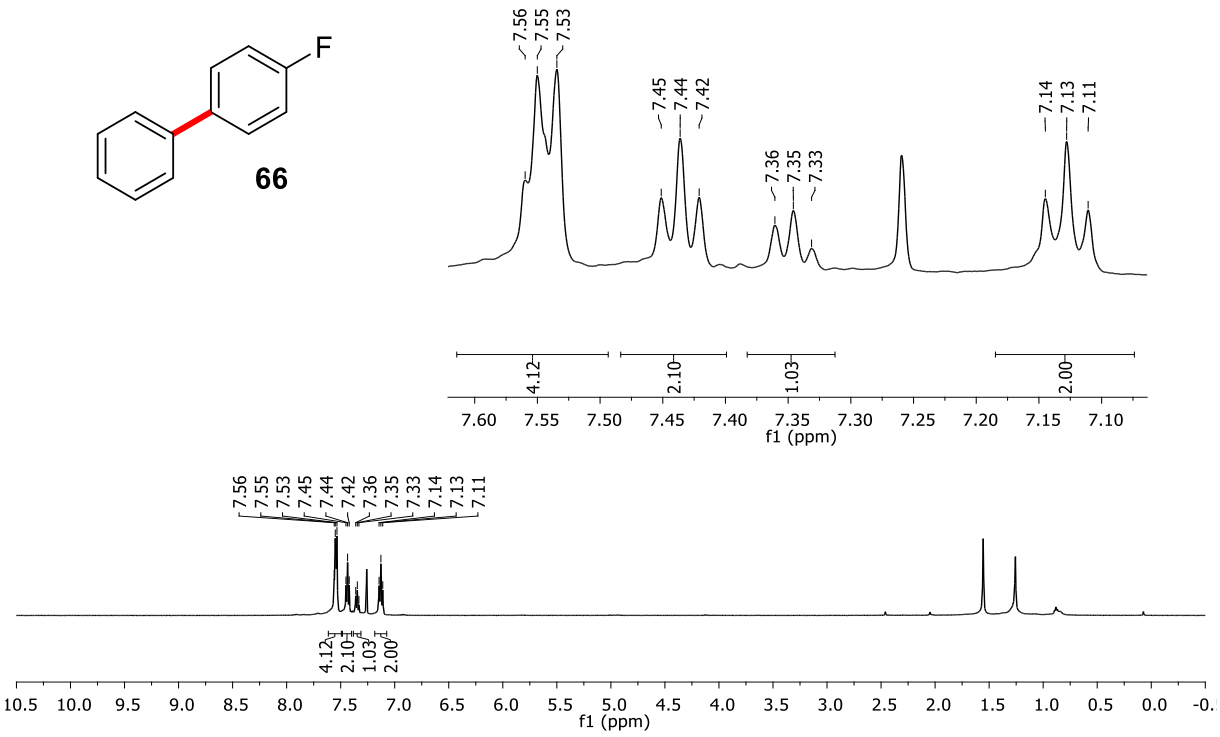

Fig. 160. ^{13}C NMR of 4-Fluoro-1,1'-biphenyl in $CDCl_3$ **(66)**[58].

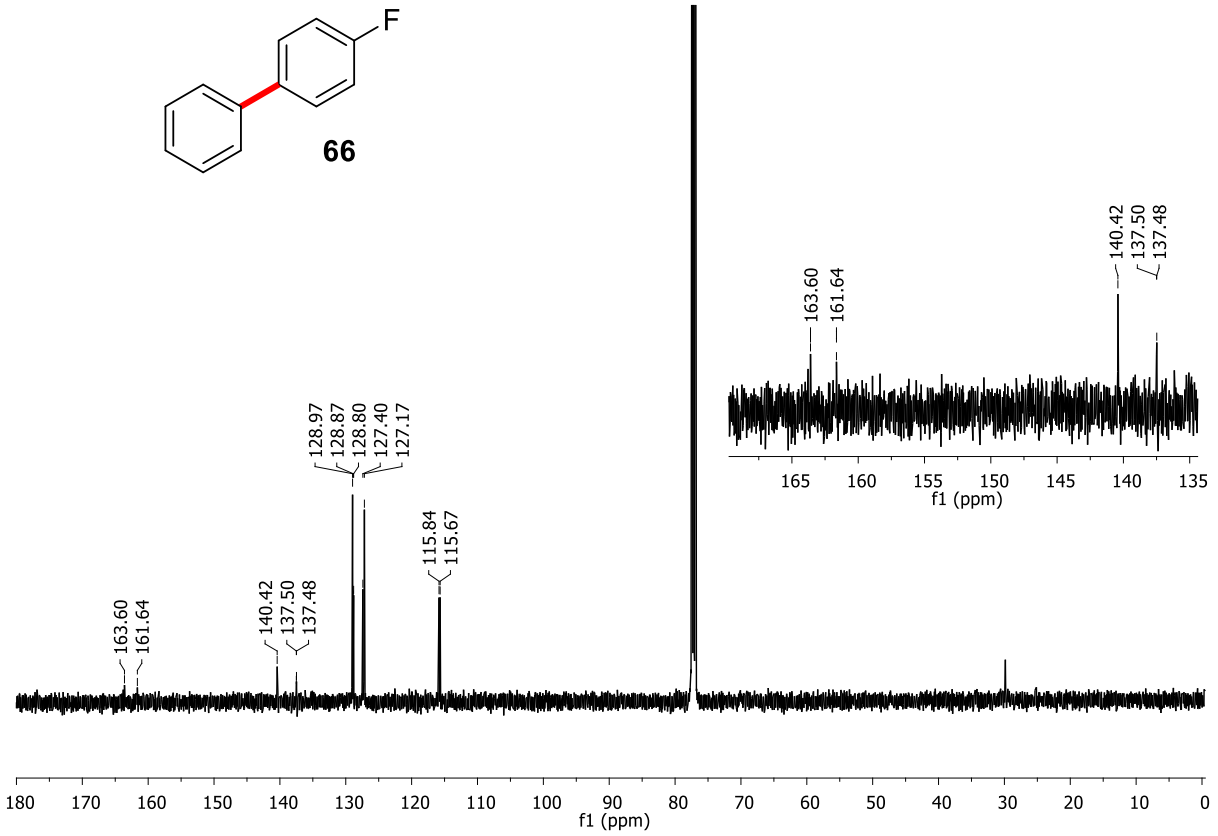

Fig. 161. ^{1}H NMR of 4-(Trifluoromethyl)-1,1'-biphenyl in $CDCl_3$ **(67)**[59].

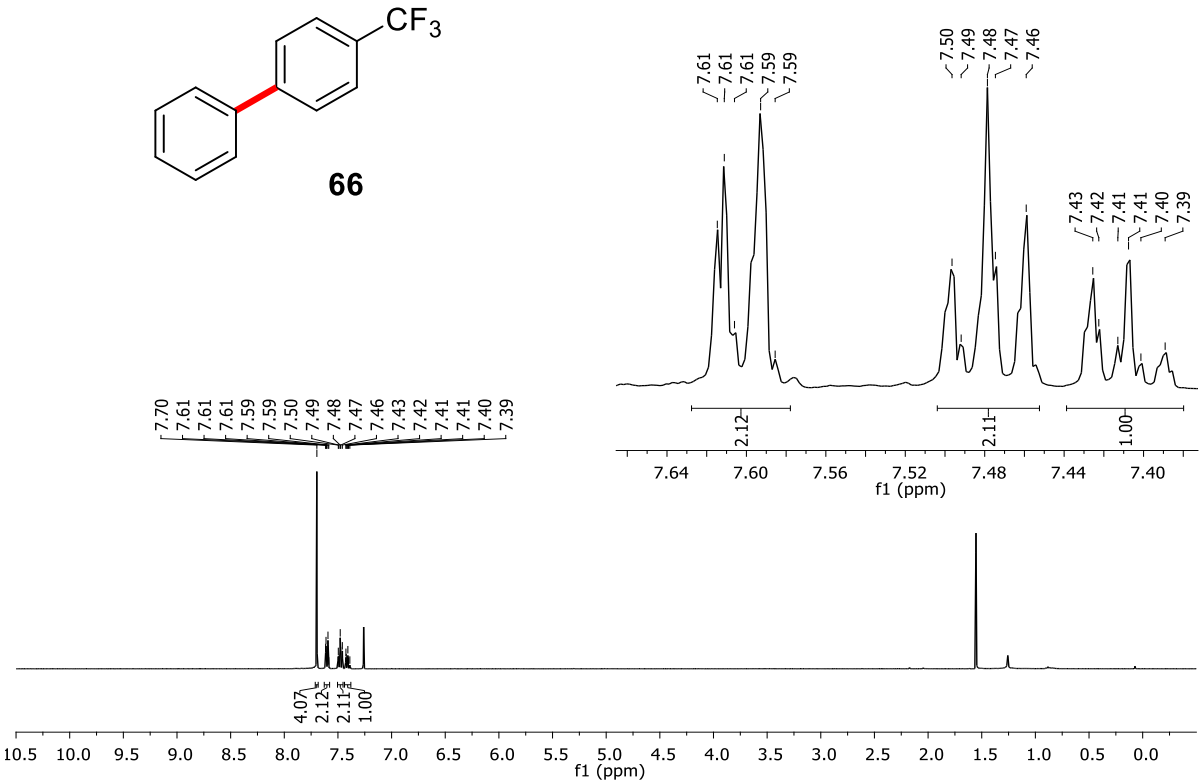

Fig. 162. ^{13}C NMR of 4-(Trifluoromethyl)-1,1'-biphenyl in $CDCl_3$ **(11)**[59].

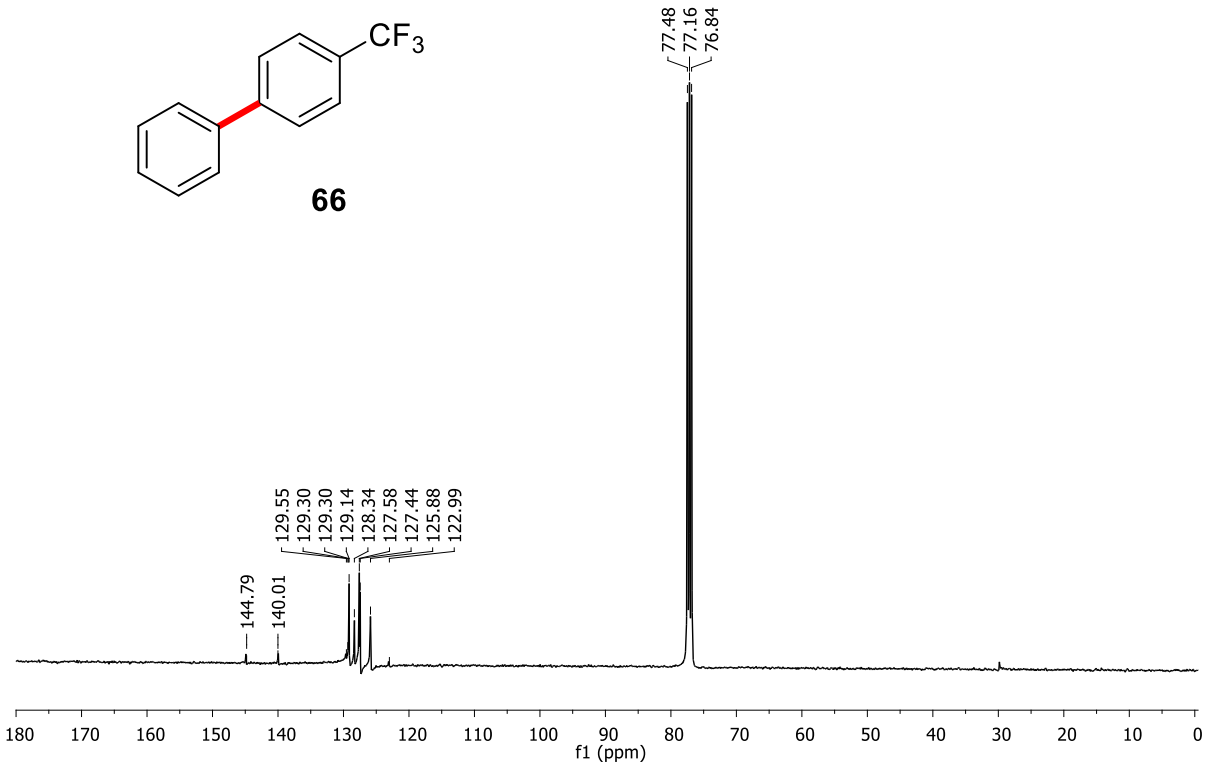

Fig. 163. ^{1}H NMR of 1,1'-Biphenyl in $CDCl_3$ **(67)**[60].

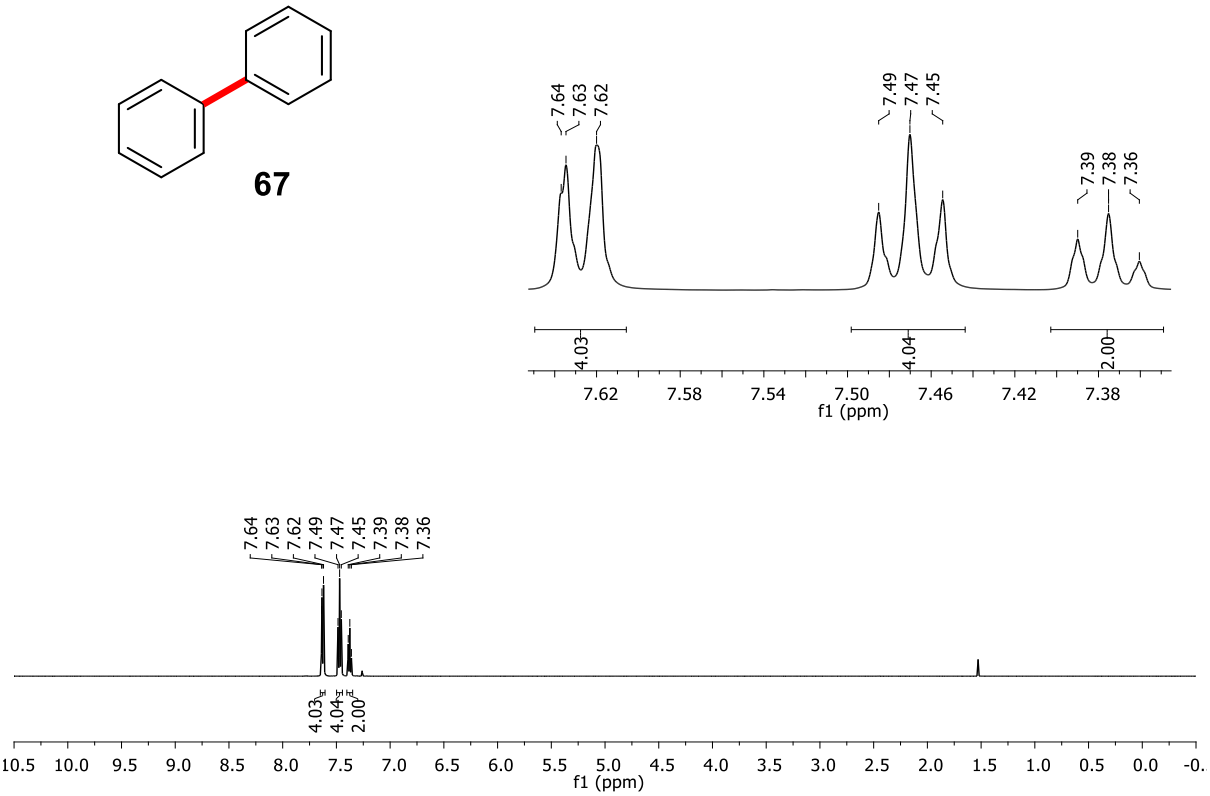

Fig. 164. ^{13}C NMR of 1,1'-Biphenyl in $CDCl_3$ **(67)**[60].

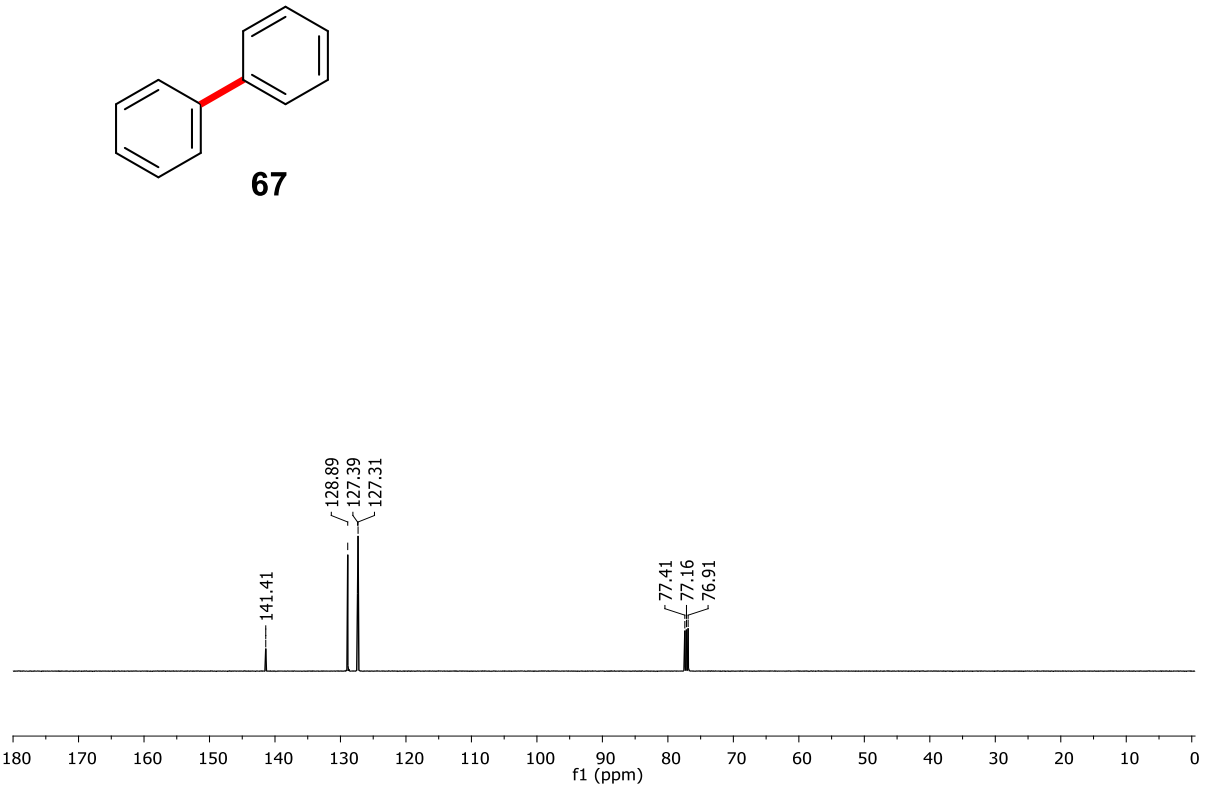

Fig. 165. ^{1}H NMR of 4-(*tert*-Butyl)-1,1'-biphenyl in $CDCl_3$ **(68)**[61].

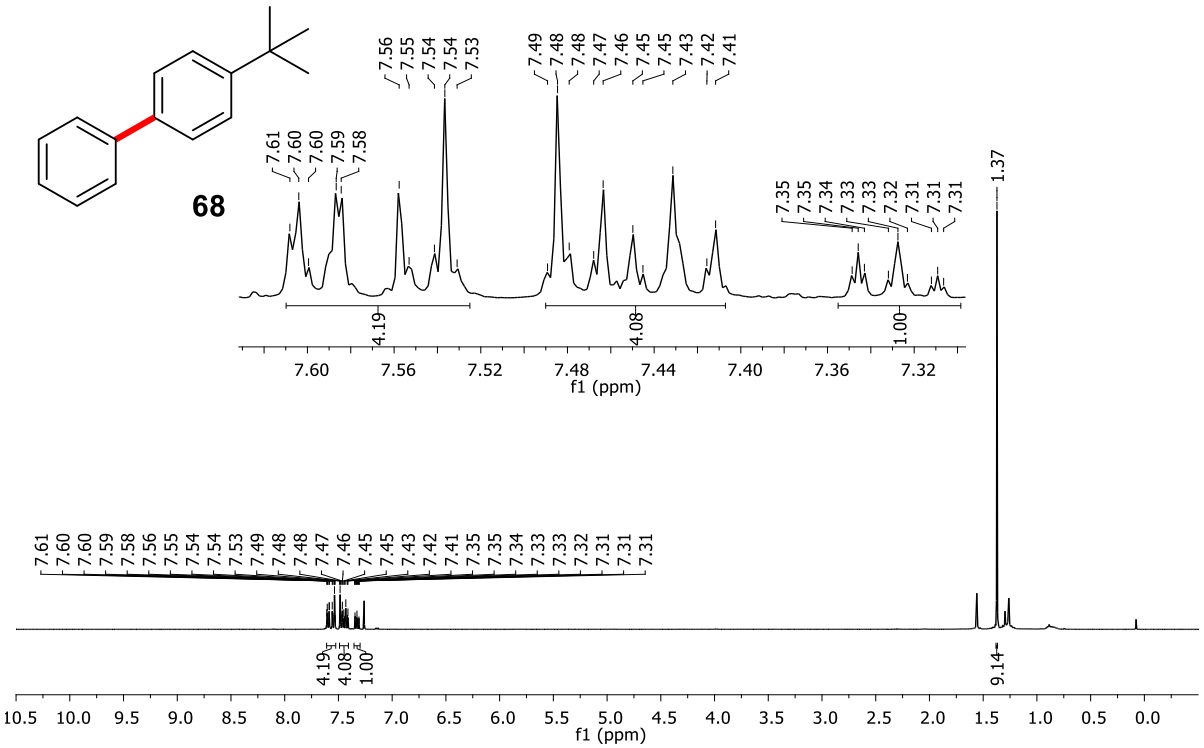

Fig. 166. ^{13}C NMR of 4-(*tert*-Butyl)-1,1'-biphenyl in $CDCl_3$ **(68)**[61]

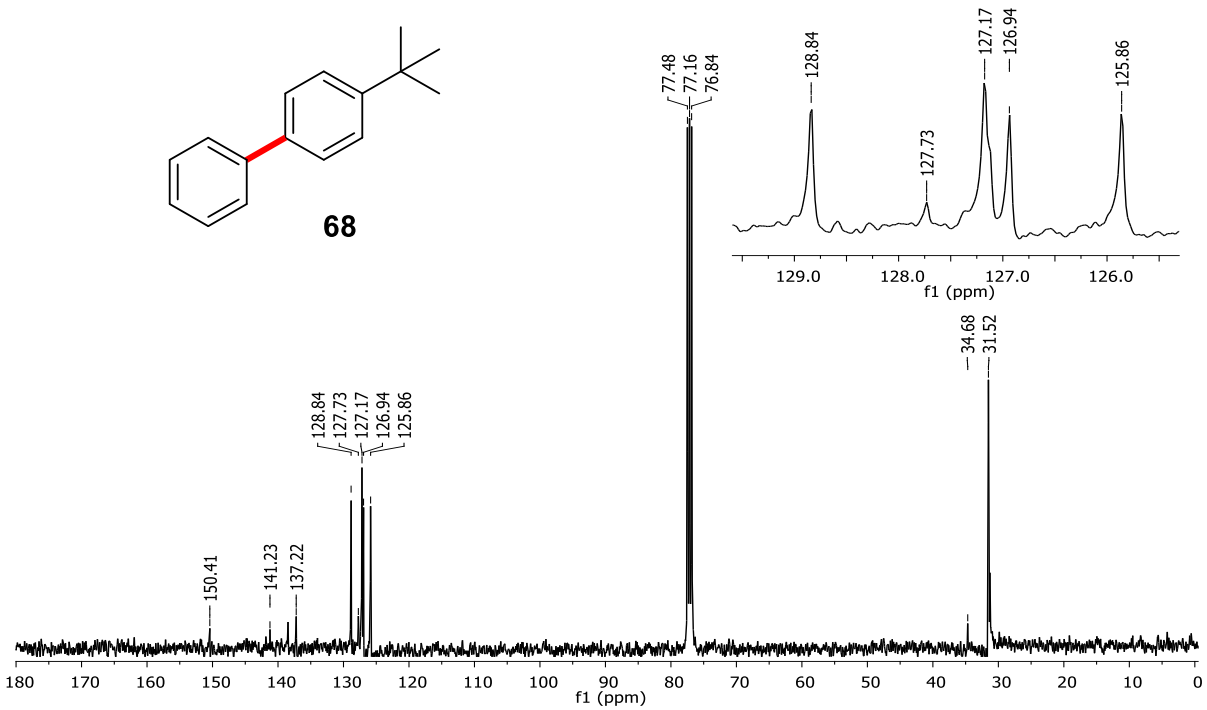

Fig. 167. ^{1}H NMR of 3, 5-Dimethyl-1, 1'-biphenyl in $CDCl_3$ **(69)**[62].

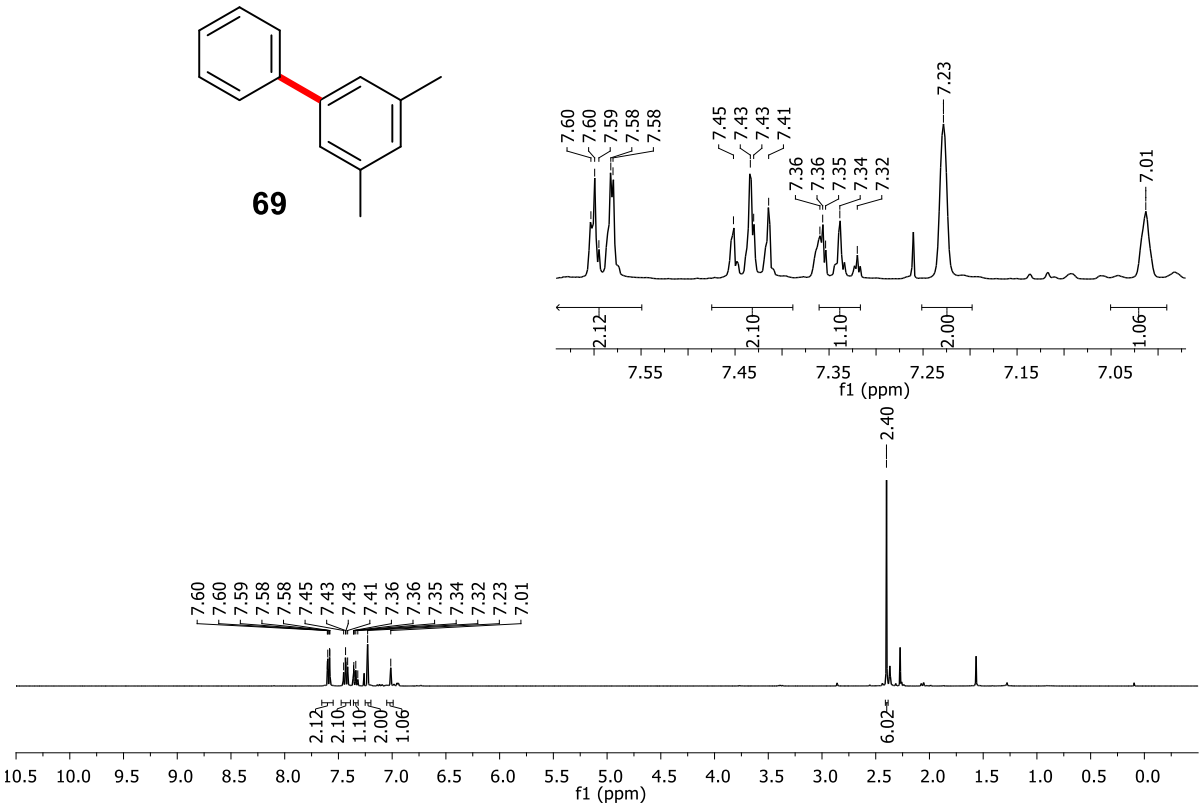

Fig. 168. ^{13}C NMR of 3, 5-Dimethyl-1, 1'-biphenyl in $CDCl_3$ **(69)**[62].

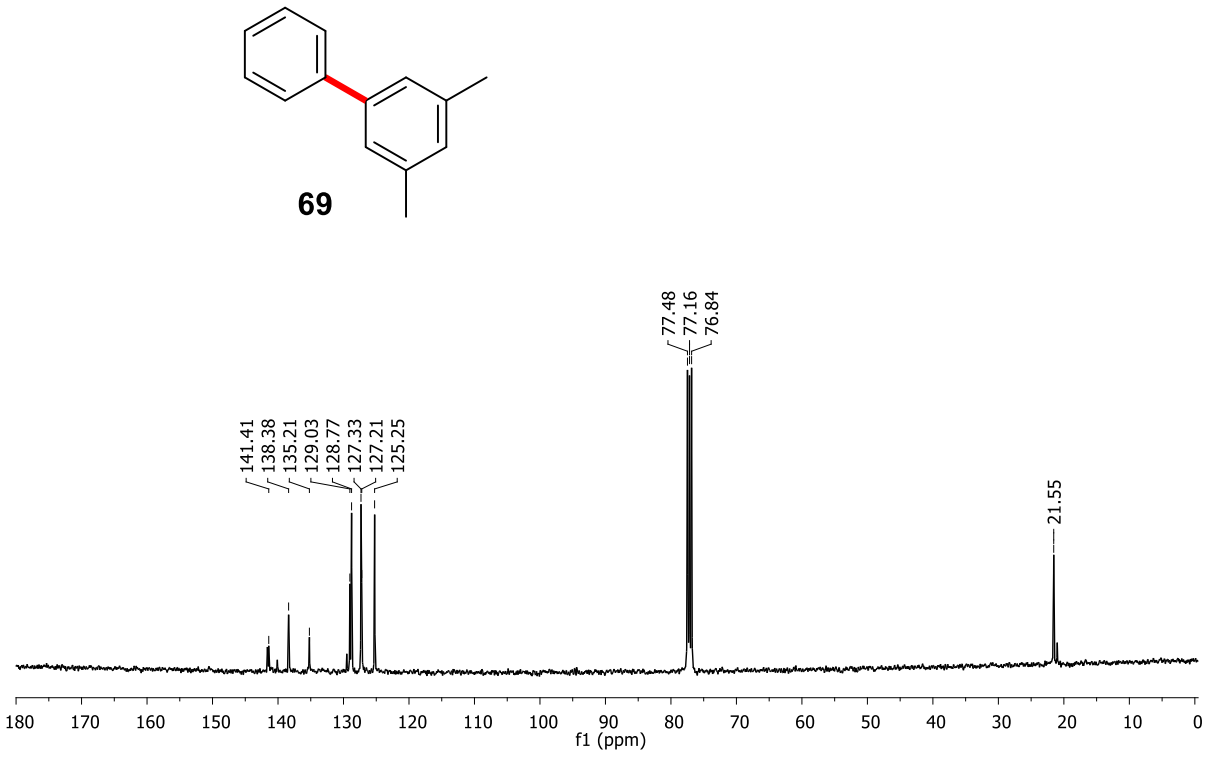

Fig. 169. ^{1}H NMR of 3-Fluoro-1,1'-biphenyl in $CDCl_3$ **(70)**[63].

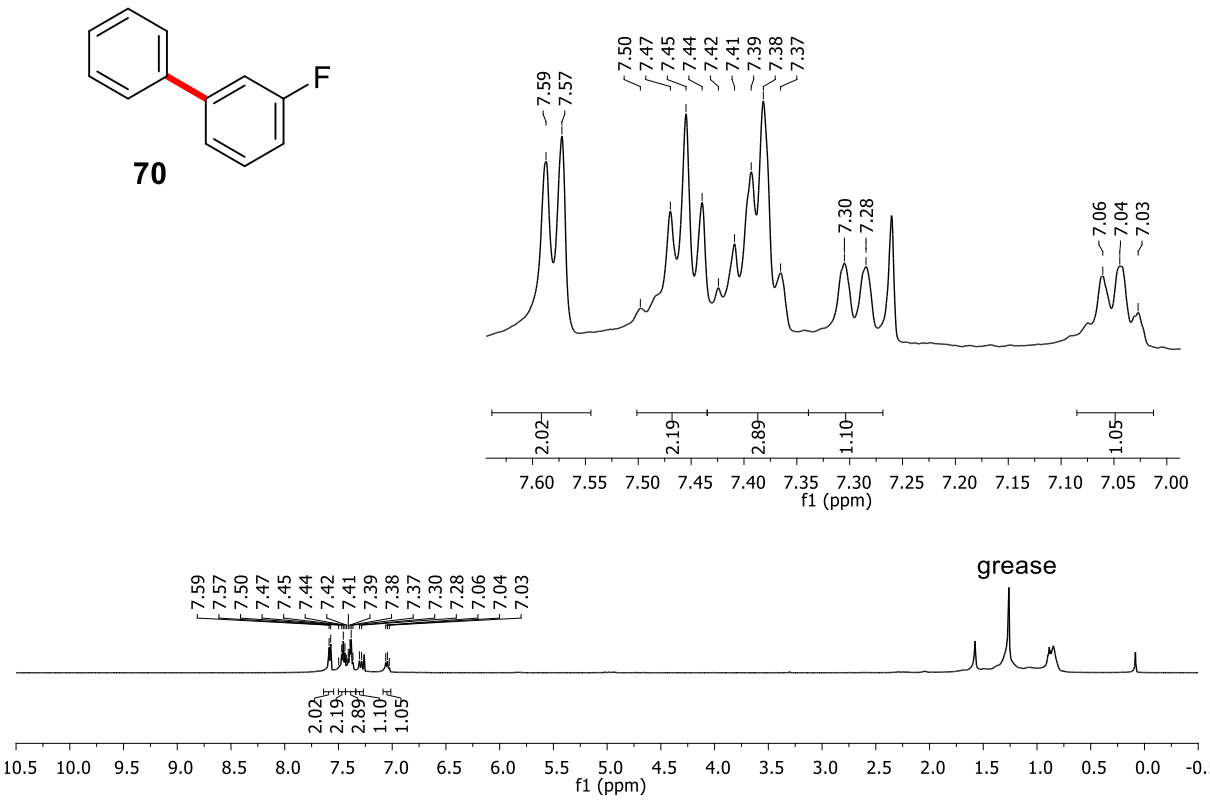

Fig. 170. ^{13}C NMR of 3-Fluoro-1,1'-biphenyl in $CDCl_3$ **(70)**[63].

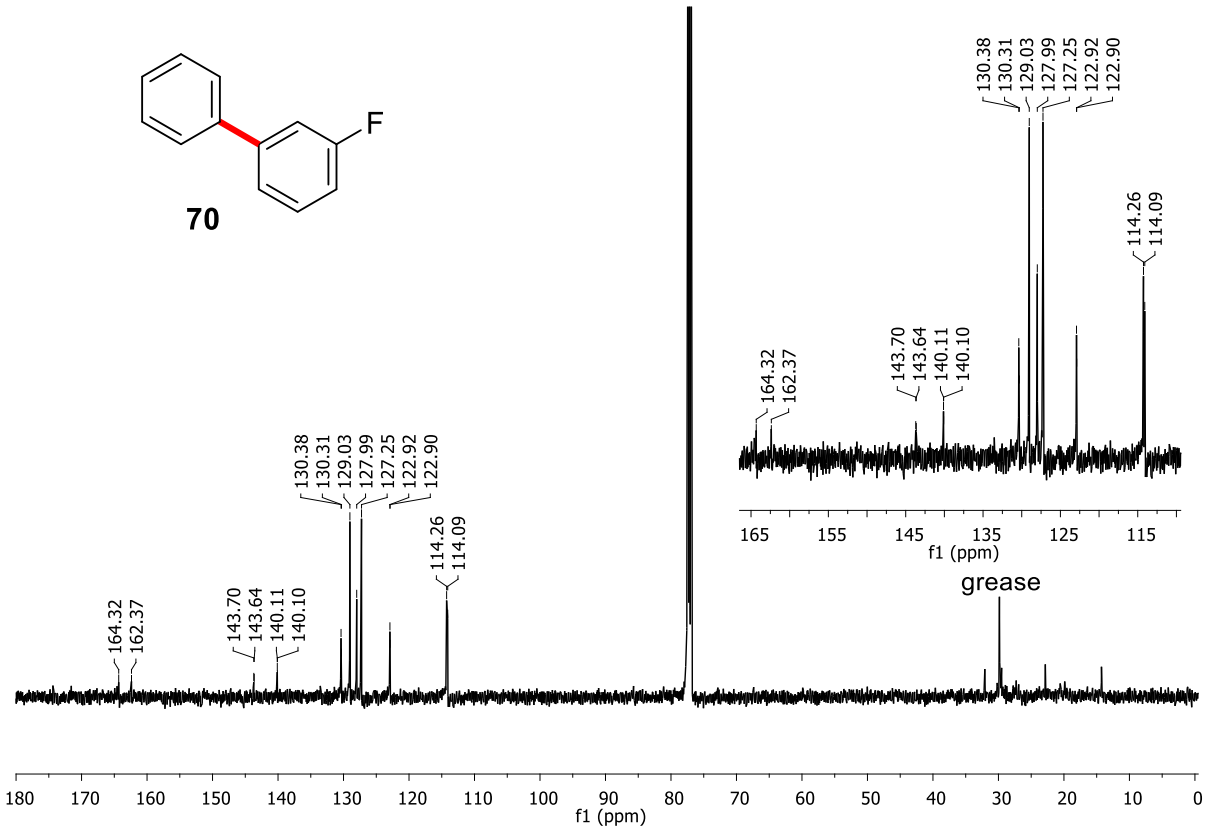

Fig. 171. ^{1}H NMR of 2, 4, 6-Trimethyl-1,1'-biphenyl in $CDCl_3$ **(71)**[64] **.**

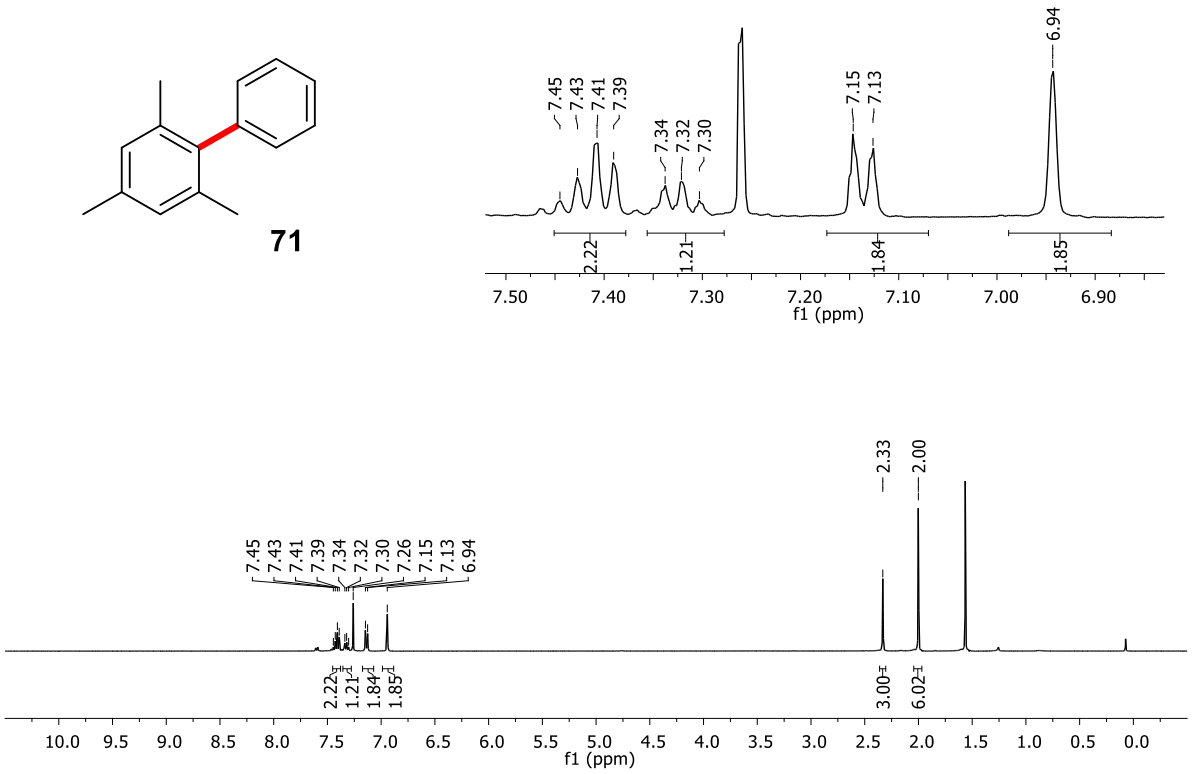

Fig. 172. ^{13}C NMR of 2, 4, 6-Trimethyl-1,1'-biphenyl in $CDCl_3$ **(71)**[64] **.**

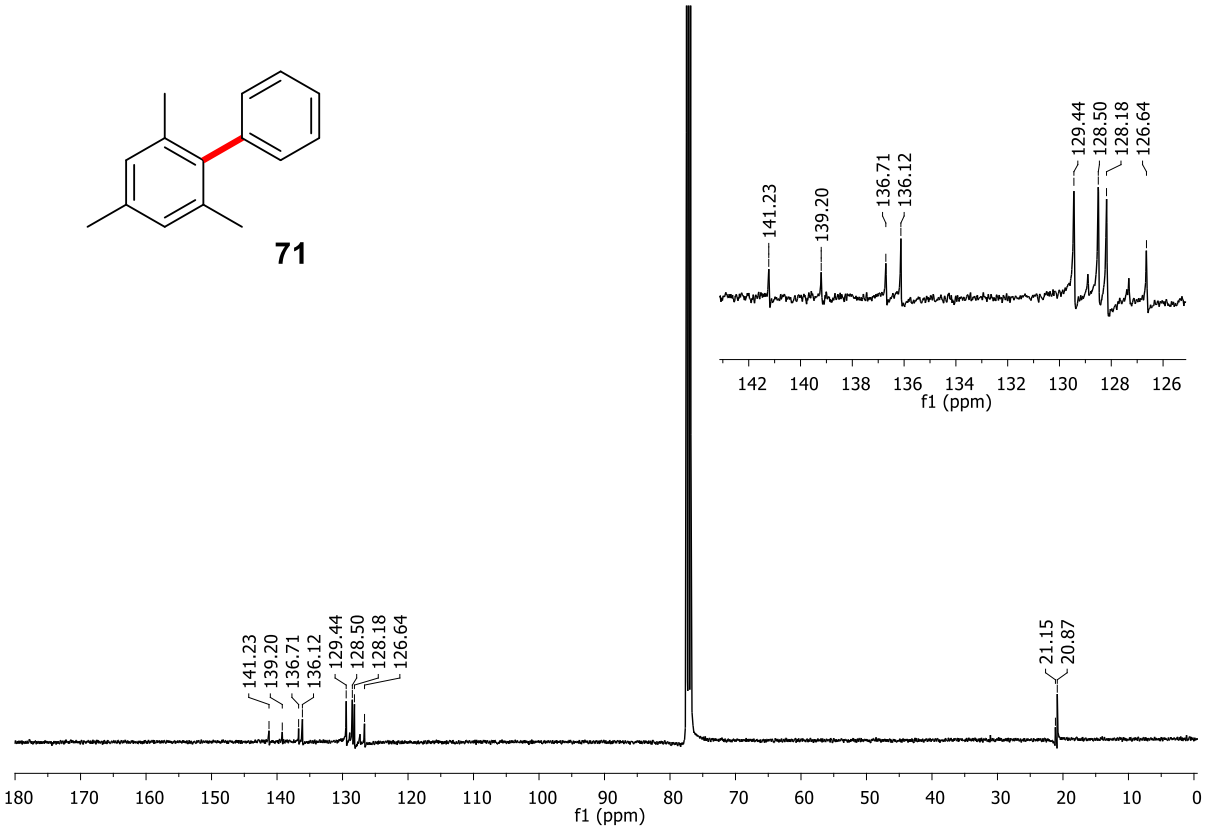

Fig. 173. ^{1}H NMR of 2, 4,6 Ttrimethyl-4'-(trifluoromethyl)-1,1'-biphenyl in $CDCl_3$ **(72)**[65]

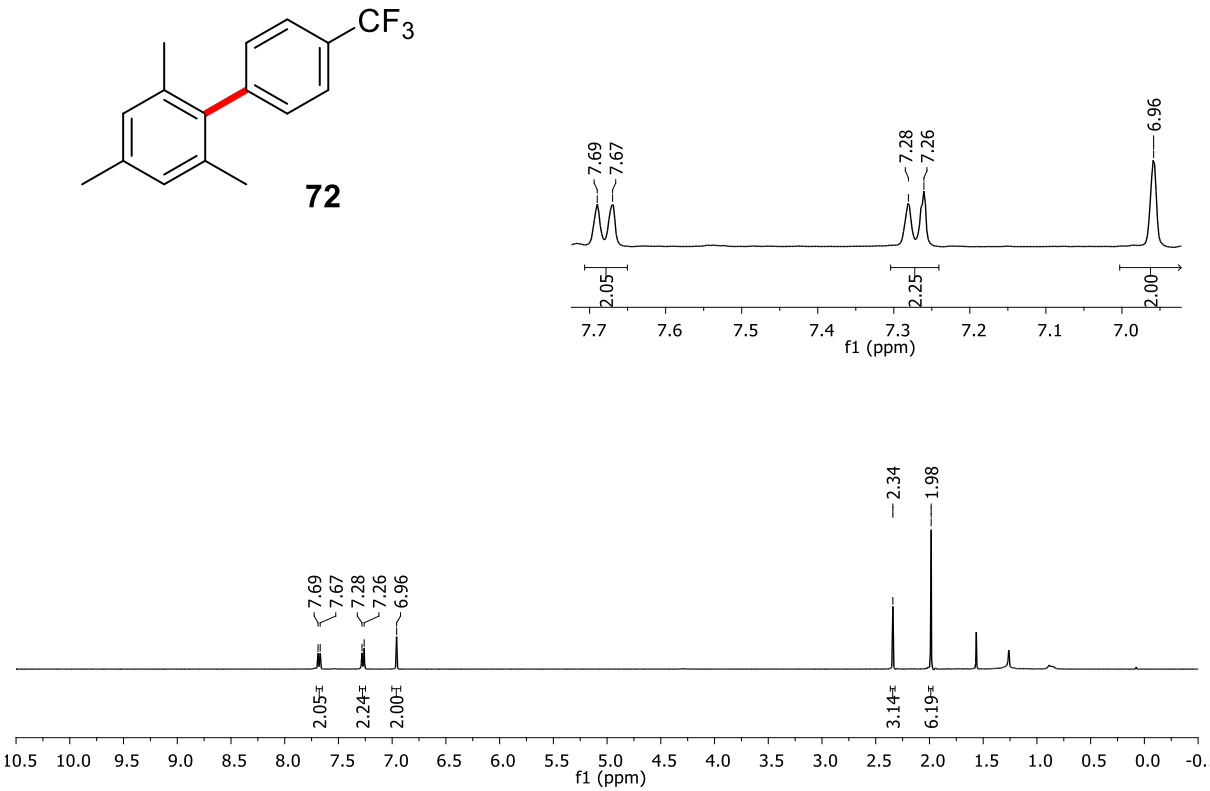

Fig. 174. ^{13}C NMR of 2, 4,6 Ttrimethyl-4'-(trifluoromethyl)-1,1'-biphenyl in $CDCl_3$ **(72)**[65]

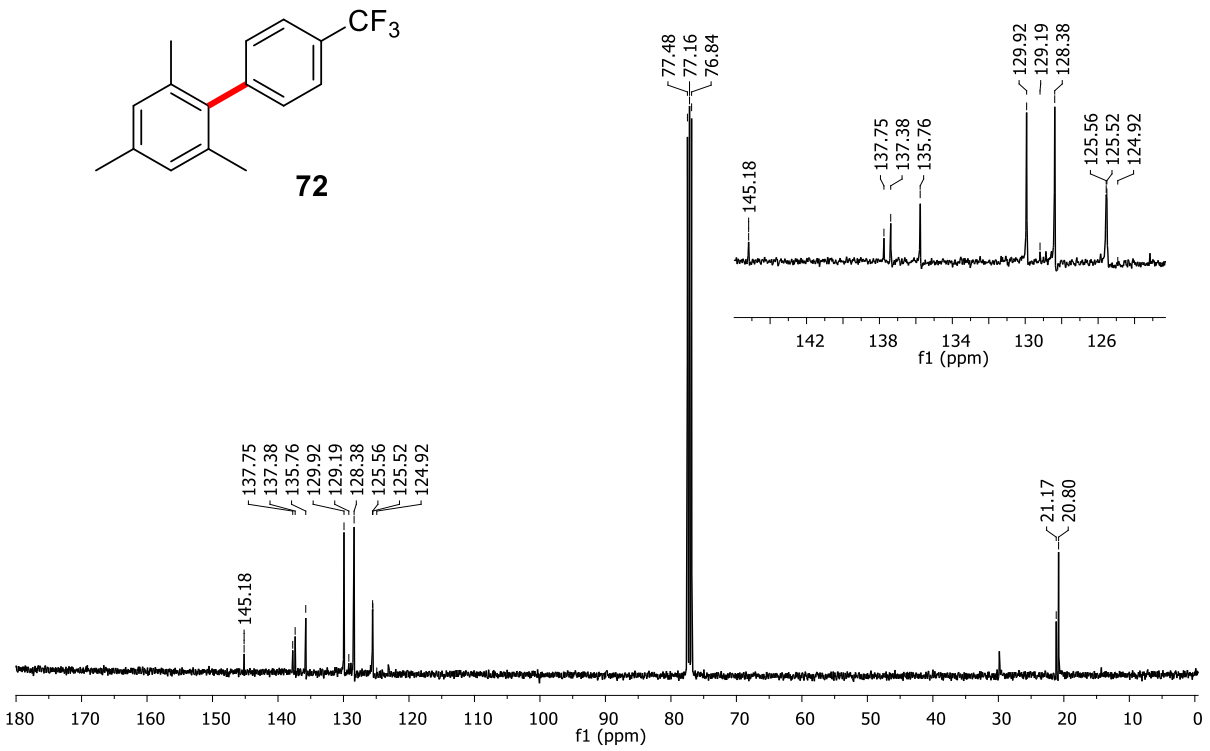

Fig. 175. ^{1}H NMR of 2-Methoxy-1,1'-biphenyl & 3-Methoxy-1,1'-biphenyl & 4-Methoxy-1,1'-biphenyl in $CDCl_3$ **(73)**[66]

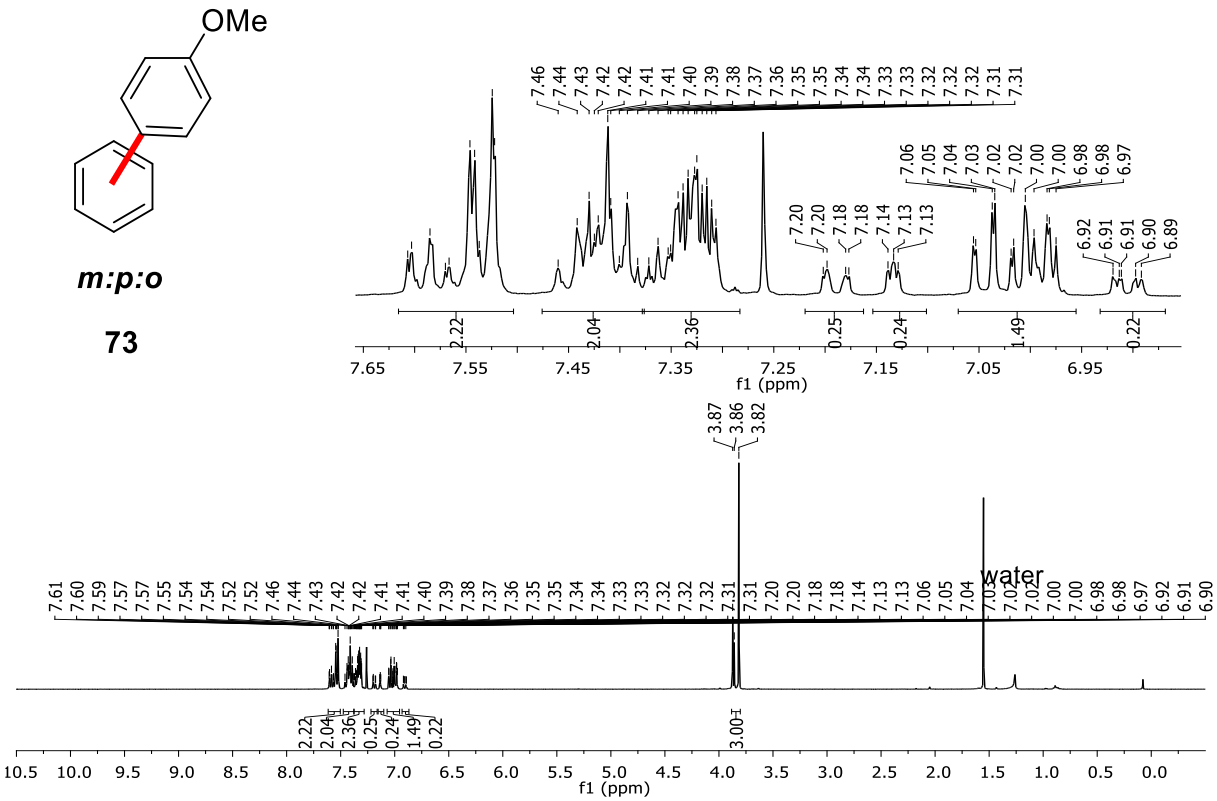

Fig. 176. ^{1}H NMR of 2-Methoxy-1,1'-biphenyl & 3-Methoxy-1,1'-biphenyl & 4-Methoxy-1,1'-biphenyl in $CDCl_3$ **(73)**[66]

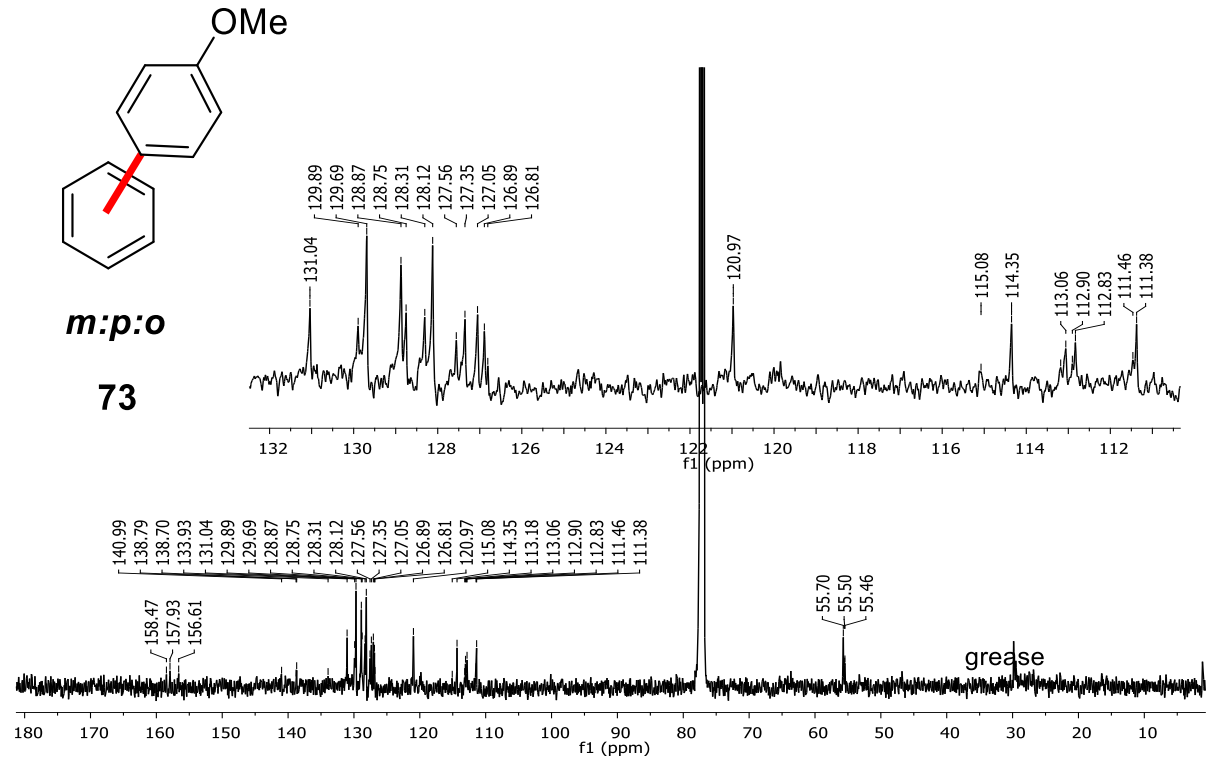

Fig. 177. [1] H NMR of (*E*)-1-Styryl-4-(trifluoromethyl)benzene in $CDCl_3$ **(74)**[67].

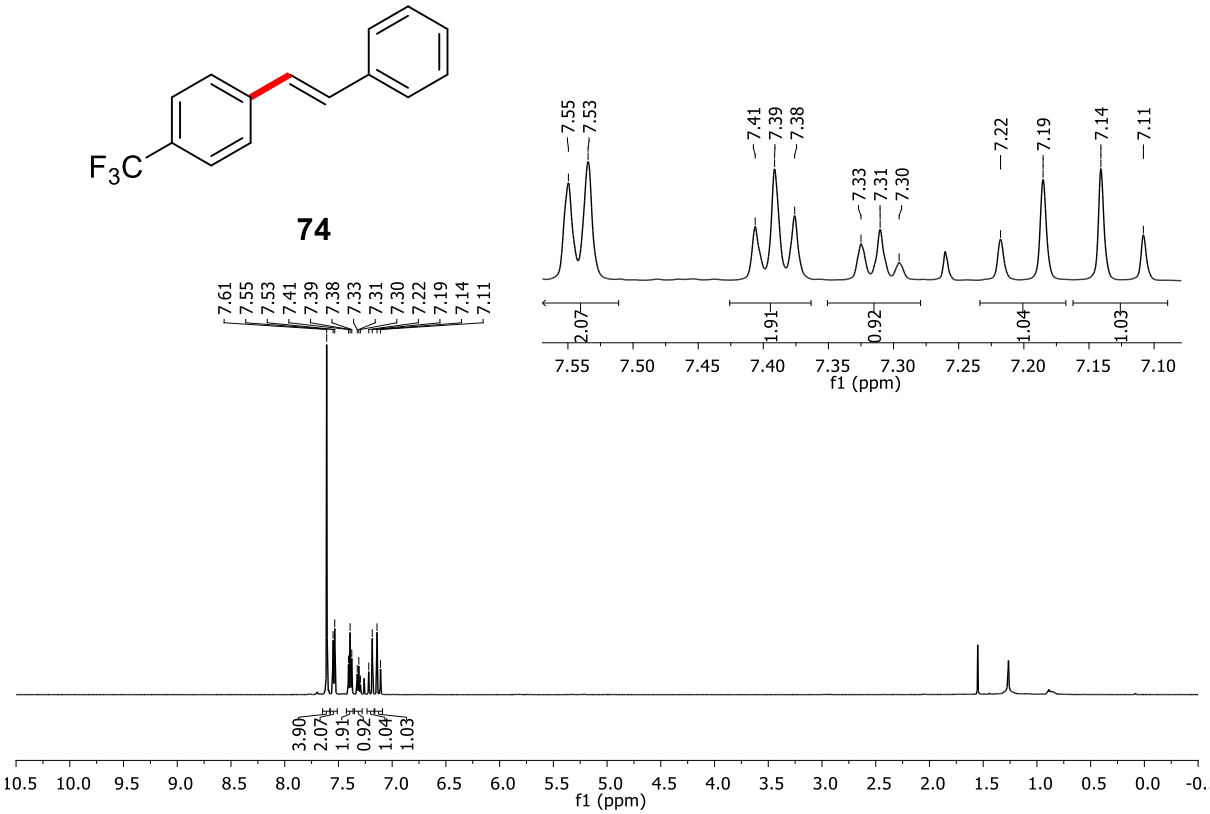

Fig. 178. [13]C NMR of (*E*)-1-Styryl-4-(trifluoromethyl)benzene in $CDCl_3$ **(74)**[67]

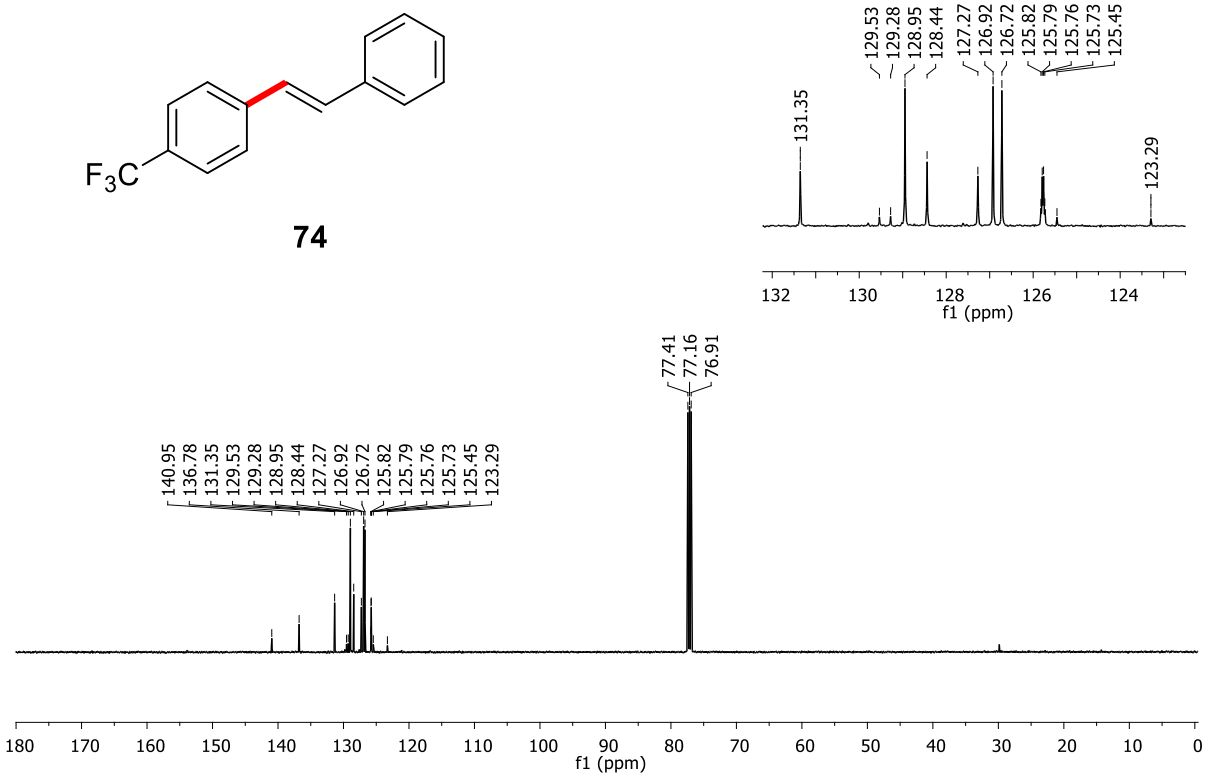

Fig. 179. [1] H NMR of (*E*)-1-Methyl-4-(4-(trifluoromethyl)styryl)benzene in $CDCl_3$ **(75)**[68]

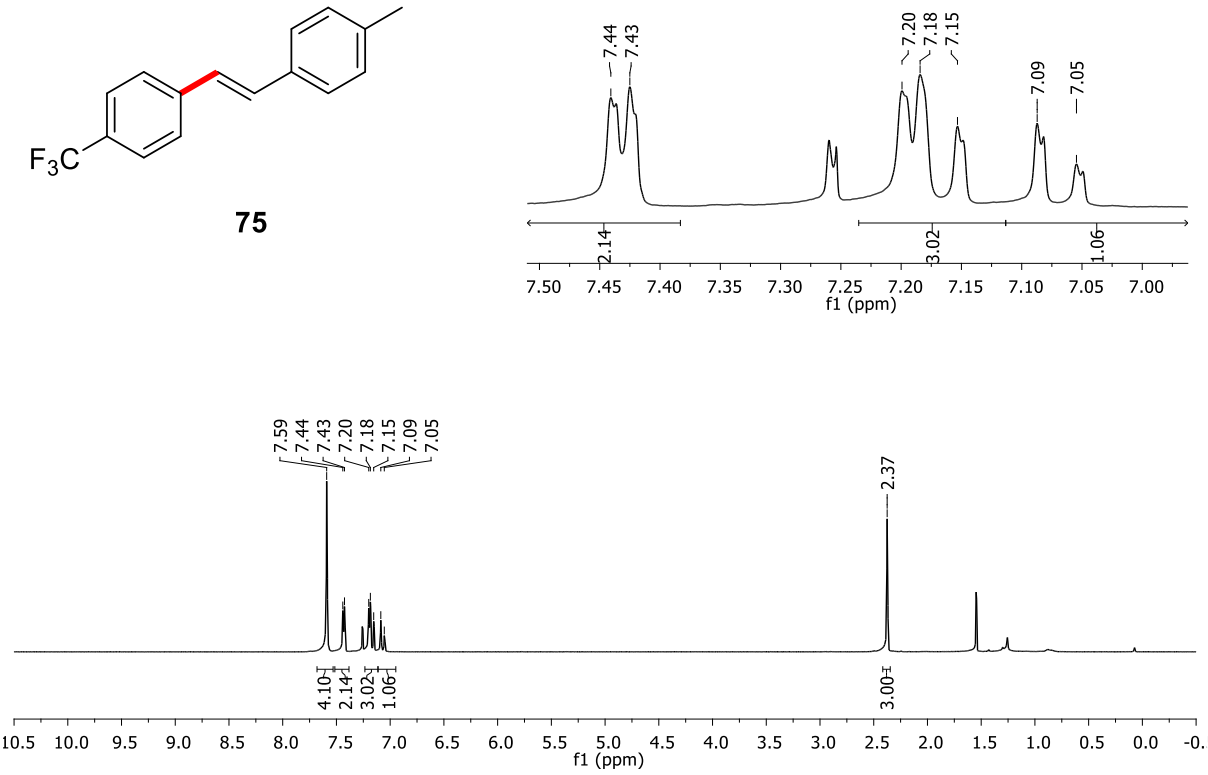

Fig. 180. [13]C NMR of (*E*)-1-Methyl-4-(4-(trifluoromethyl)styryl)benzene in $CDCl_3$ **(75)**[68]

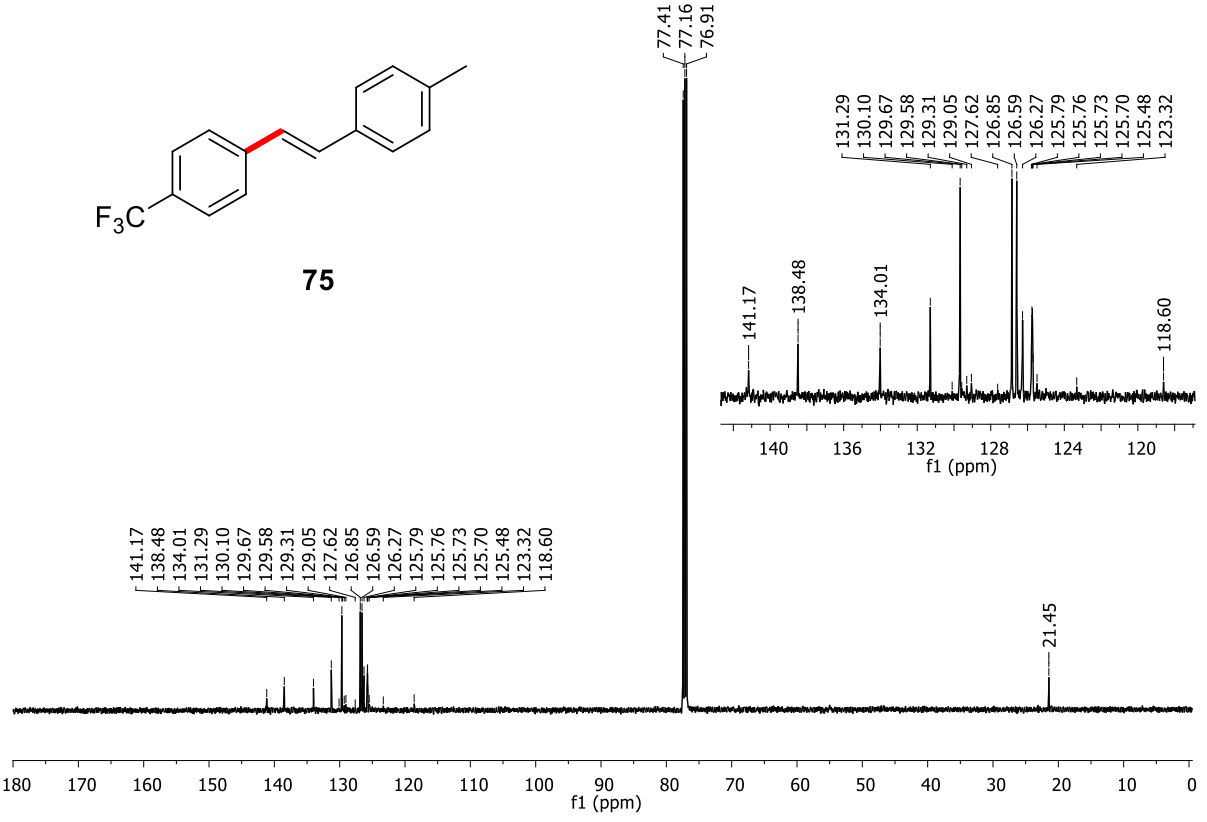

Fig. 181. [1] H NMR of (*E*)-1-Methoxy-4-(4-(trifluoromethyl)styryl)benzene in $CDCl_3$ **(76)**[69]

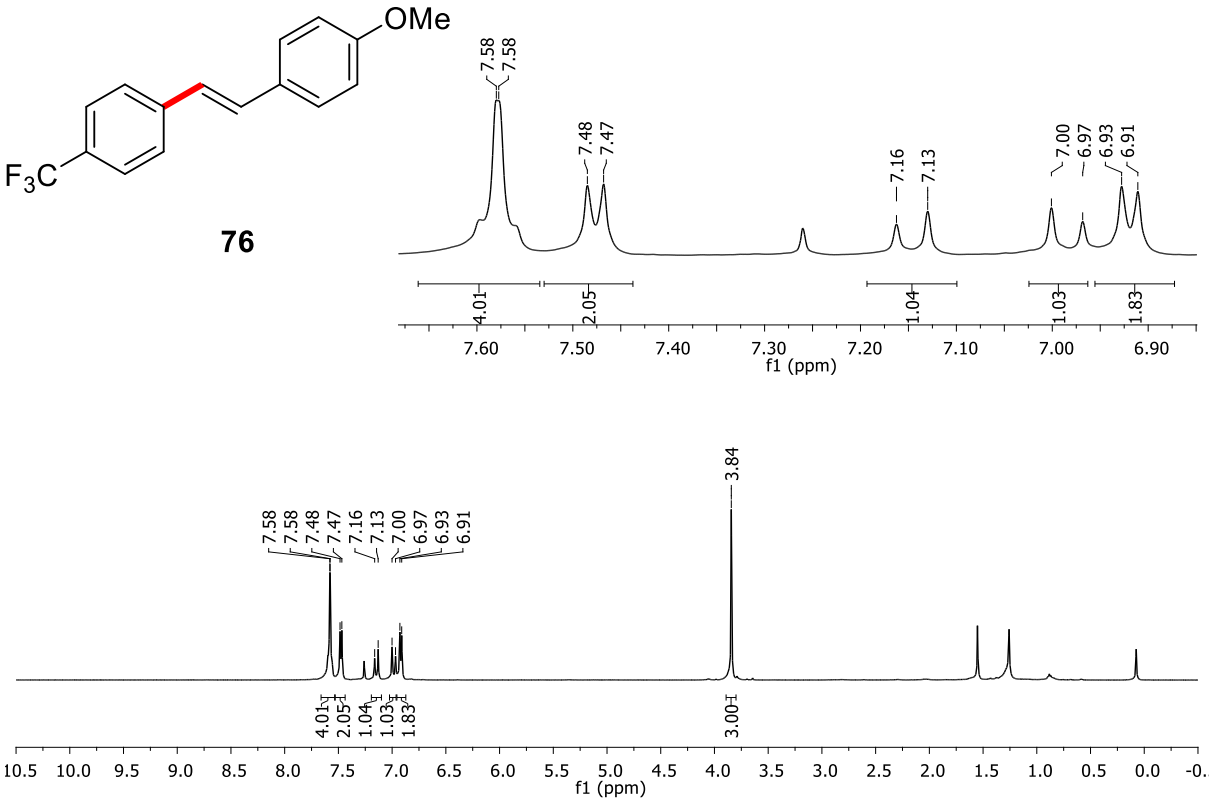

Fig. 182. [13]C NMR of (*E*)-1-Methoxy-4-(4-(trifluoromethyl)styryl)benzene in $CDCl_3$ **(76)**[69]

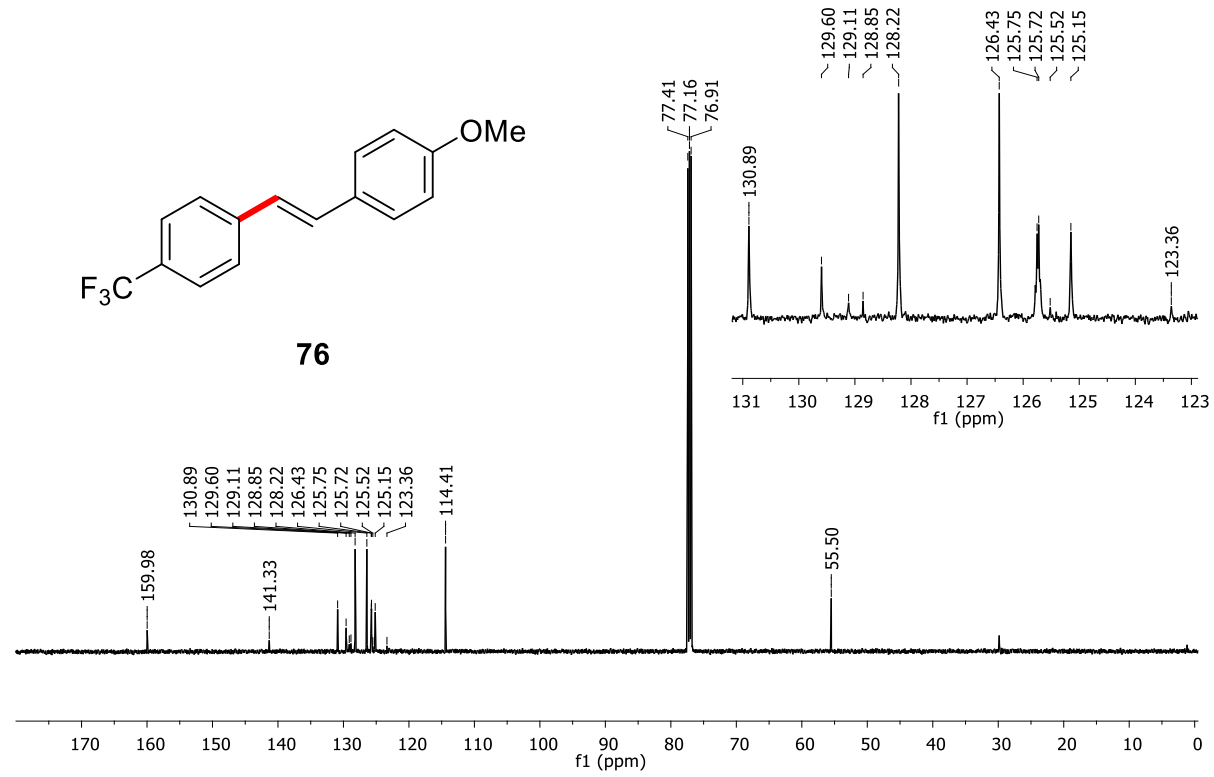

Fig. 183. [1] H NMR of (*E*)-1-Chloro-4-styrylbenzene in $CDCl_3$ **(77)**[70]

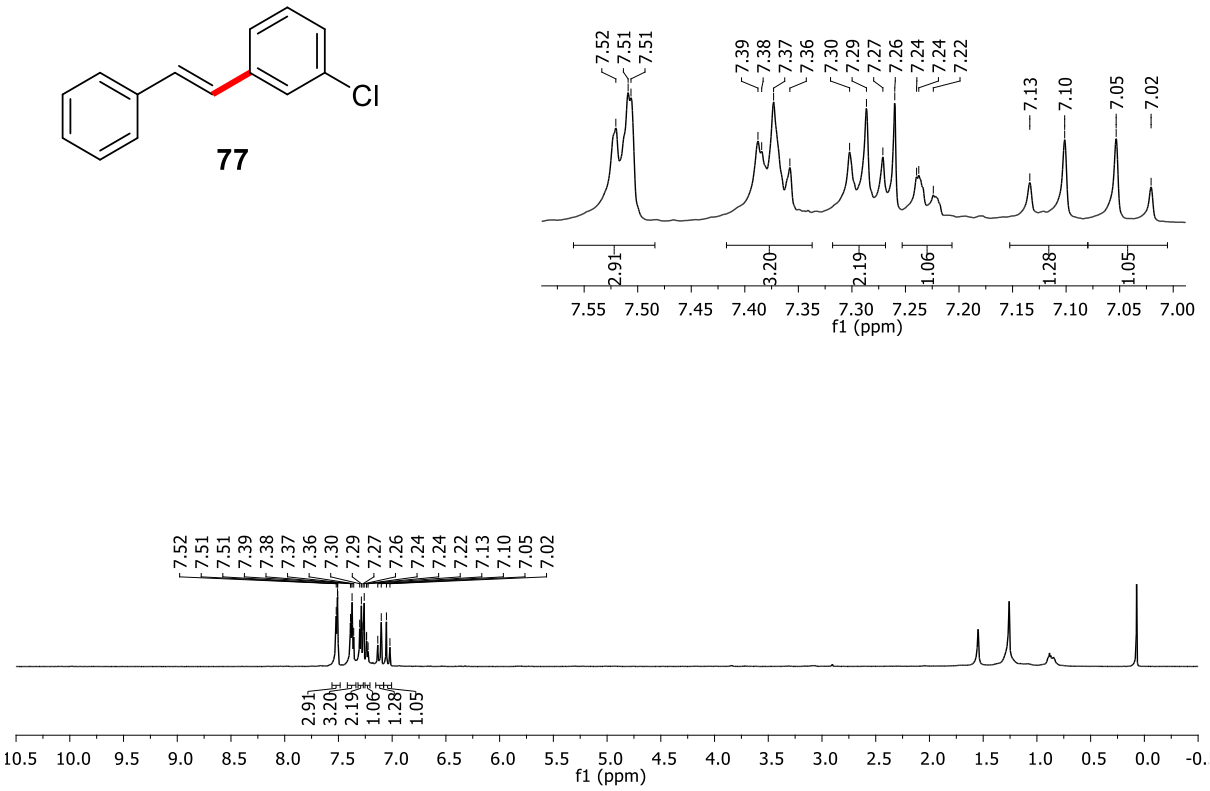

Fig. 184. [13]C NMR of (*E*)-1-Chloro-4-styrylbenzene in $CDCl_3$ **(77)**[70]

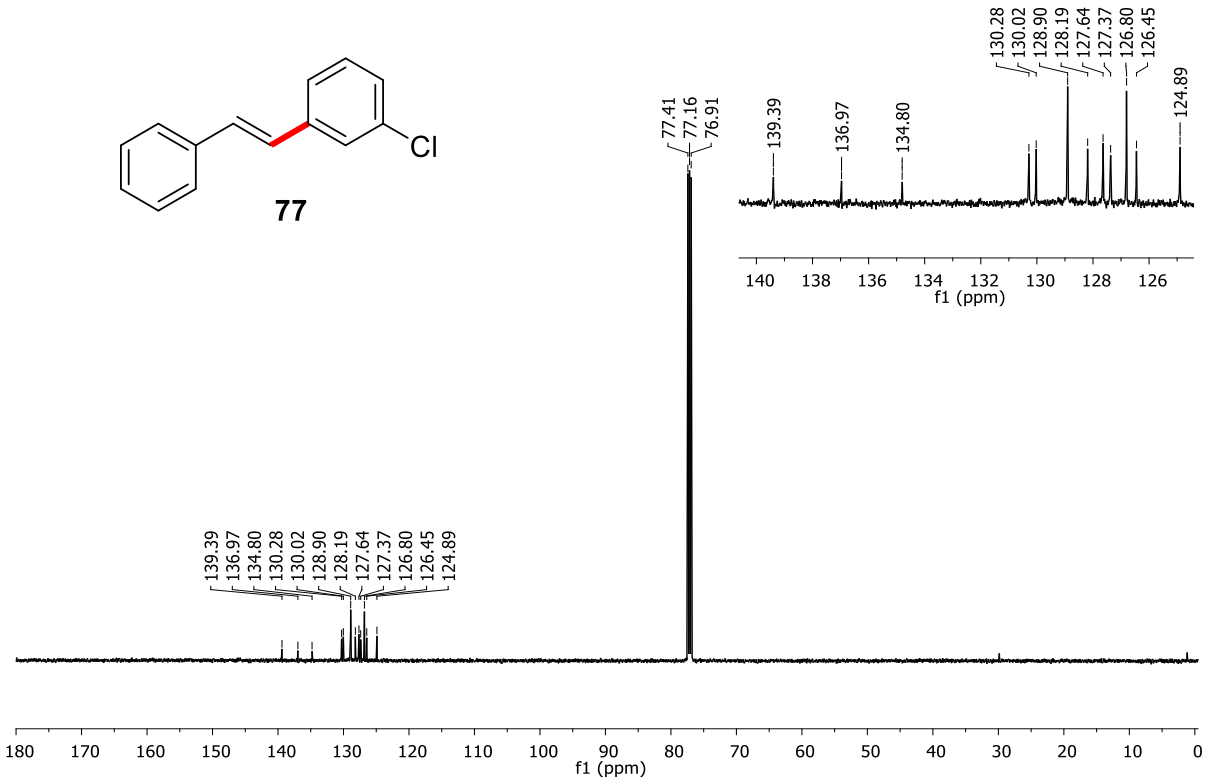

Fig. 185. ^{1}H NMR of (*E*)-1-Fluoro-4-(4-methylstyryl)benzene in $CDCl_3$ **(78)**[71]

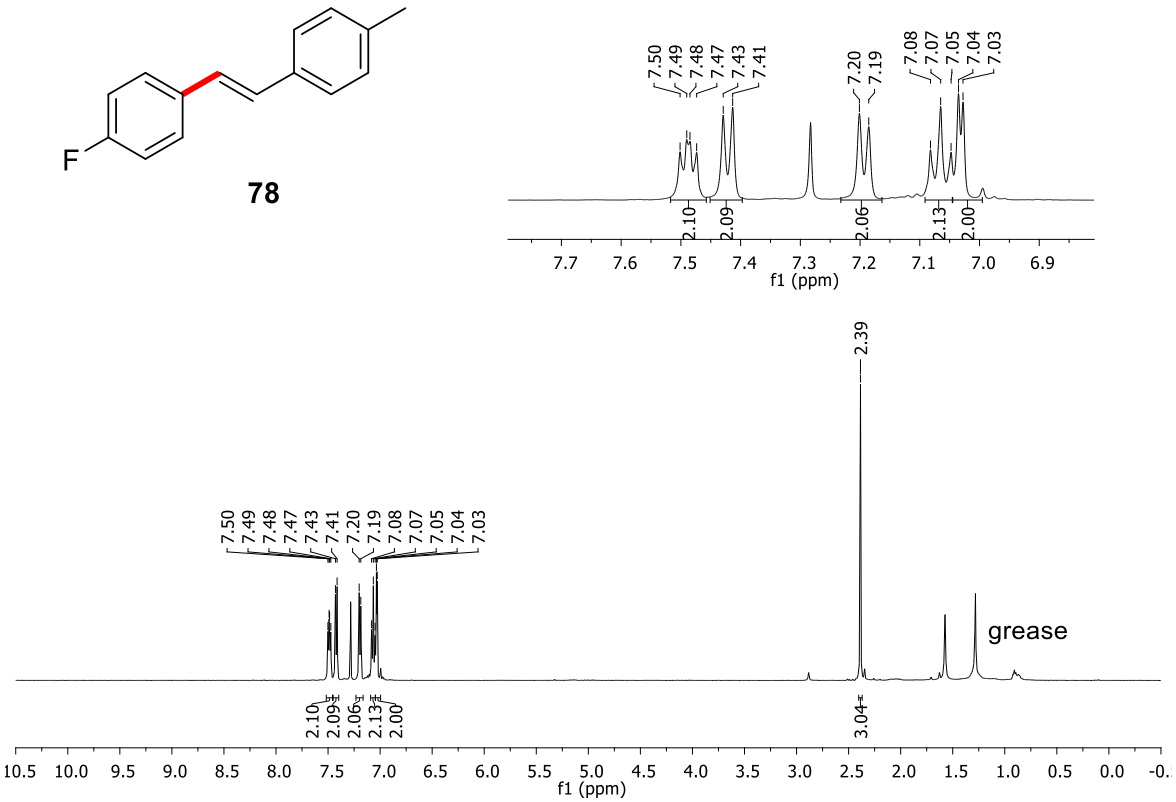

Fig. 186. ^{13}C NMR of (*E*)-1-Fluoro-4-(4-methylstyryl)benzene in $CDCl_3$ **(78)**[71]

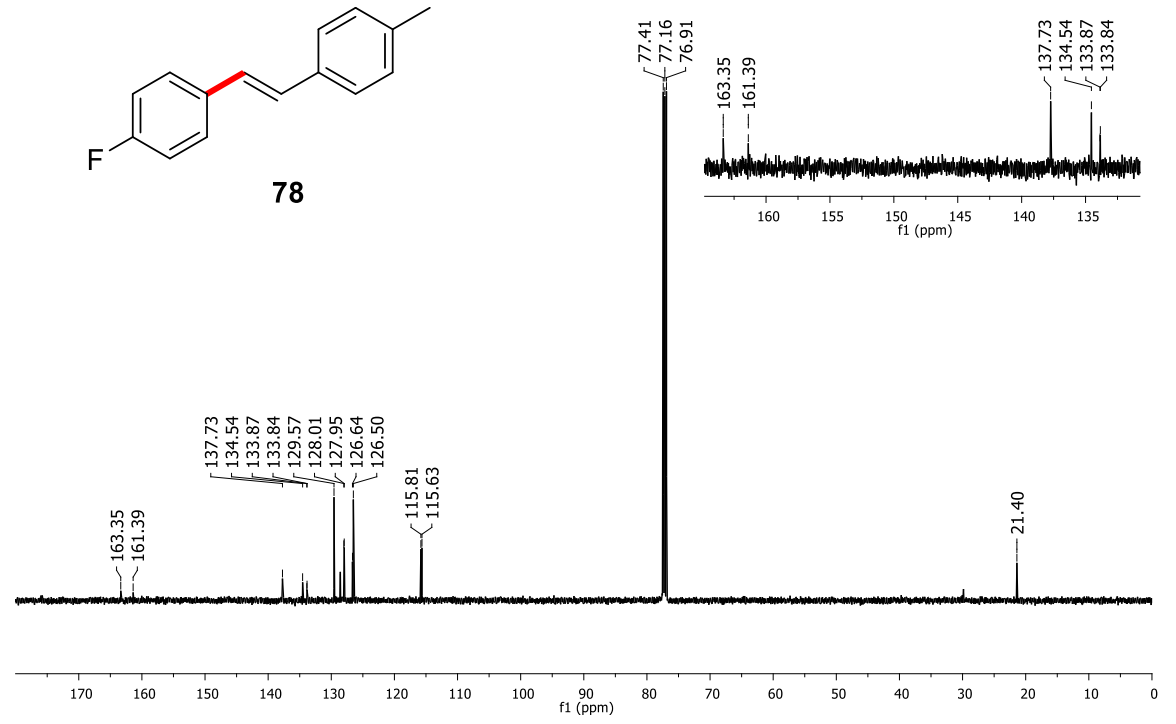

Fig. 187. [1] H NMR of (*E*)-2-(4-Methoxystyryl)pyridine in $CDCl_3$ **(79)**[72]

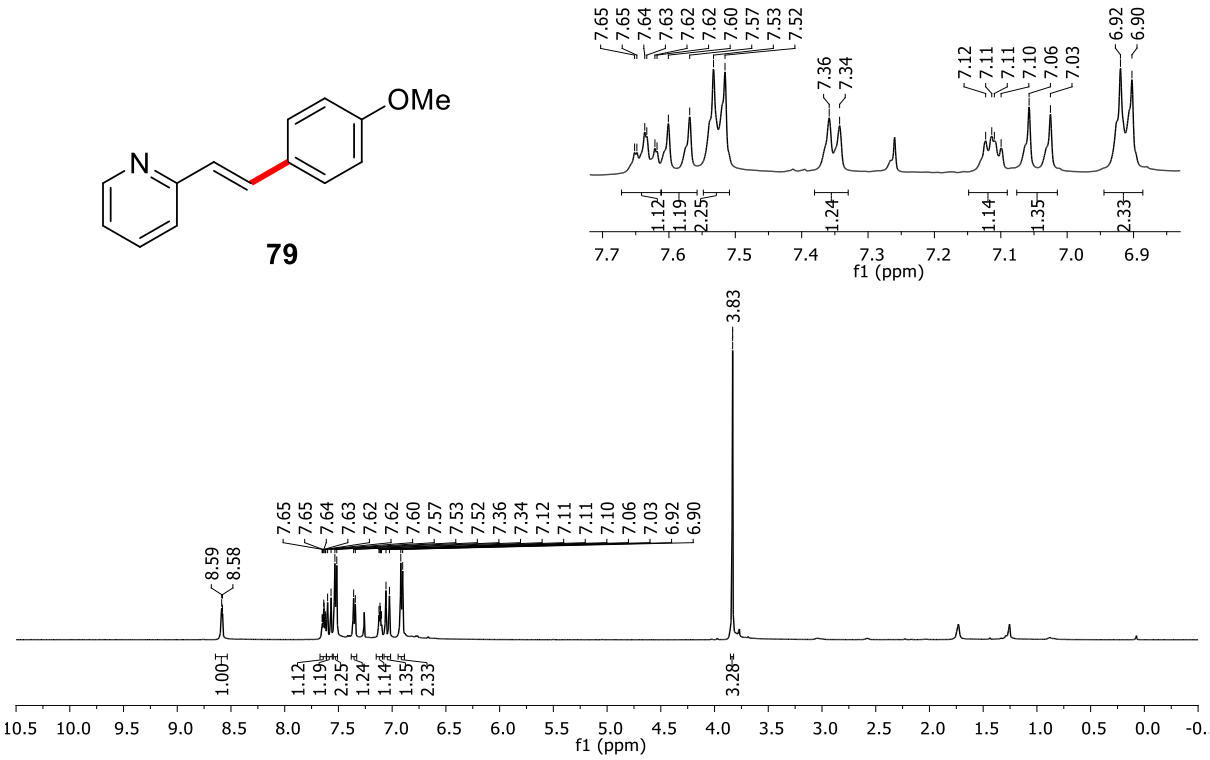

Fig. 188. [13]C NMR of (*E*)-2-(4-Methoxystyryl)pyridine in $CDCl_3$ **(79)**[72]

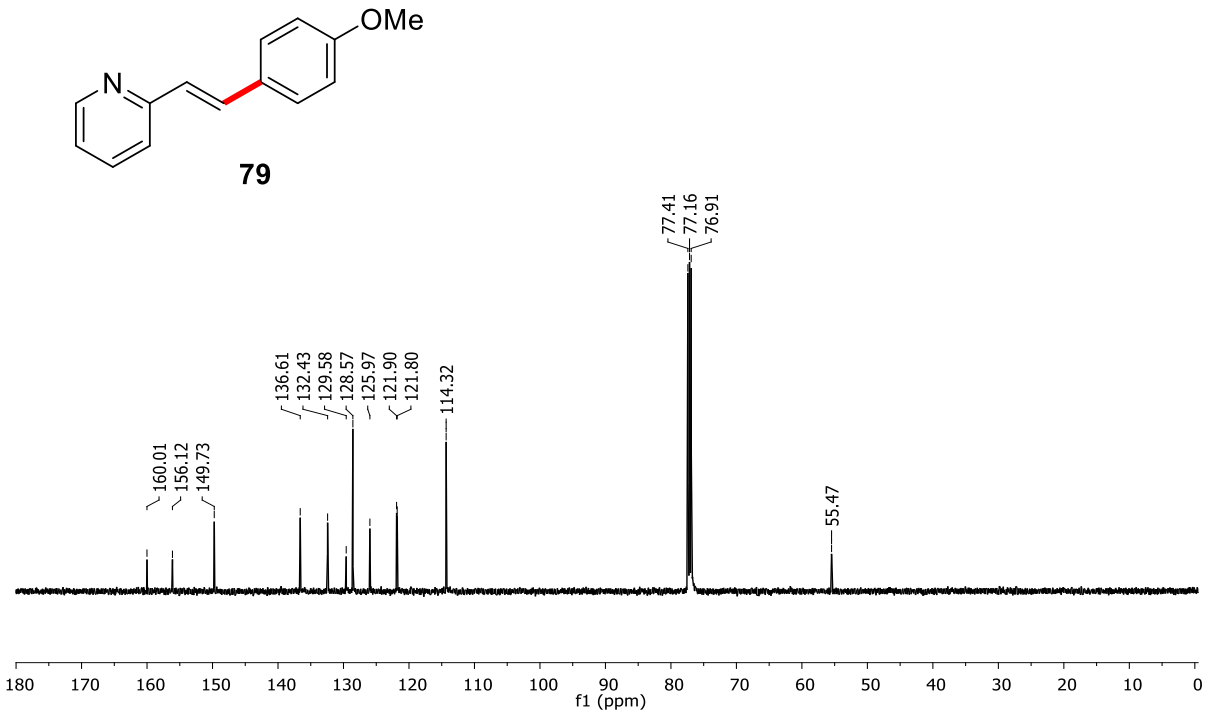

Fig. 189. [1] H NMR of (*E*)-2-(4-Methoxystyryl)-6-methylpyridine in $CDCl_3$ **(80)**[73]

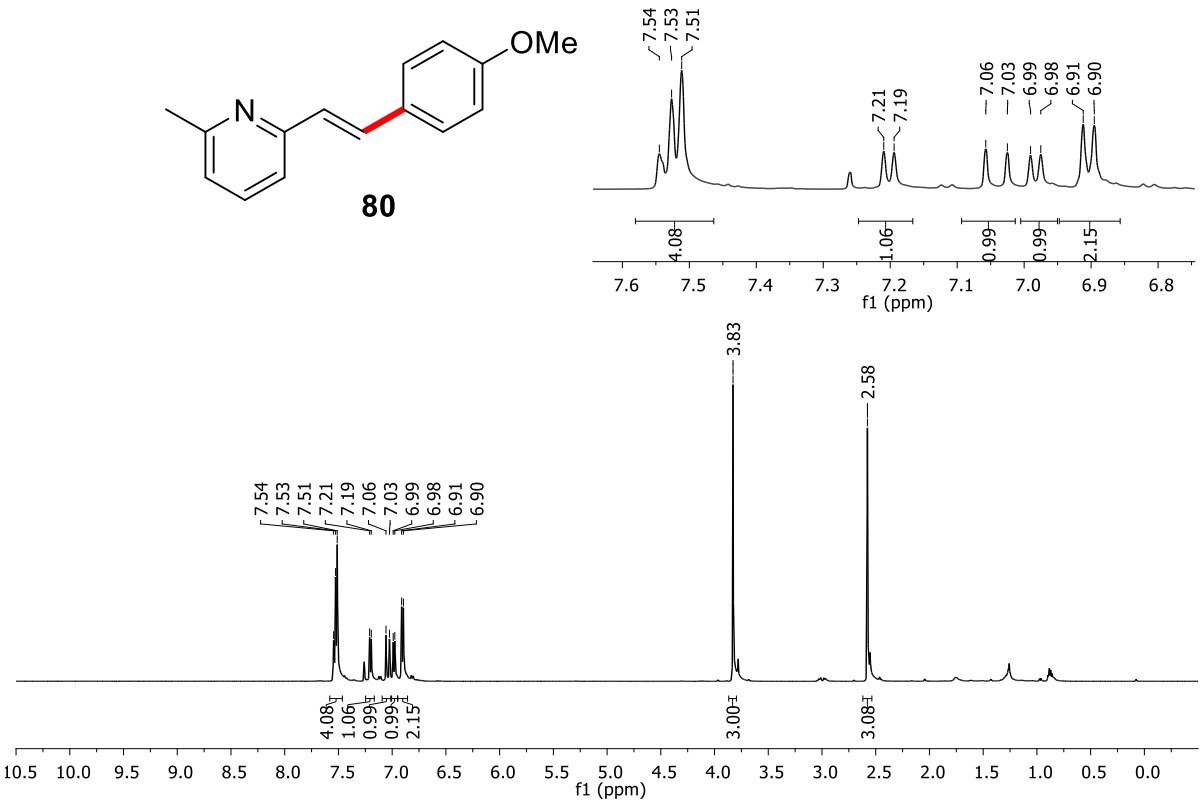

Fig. 190. [13]C NMR of (*E*)-2-(4-Methoxystyryl)-6-methylpyridine in $CDCl_3$ **(80)**[73]

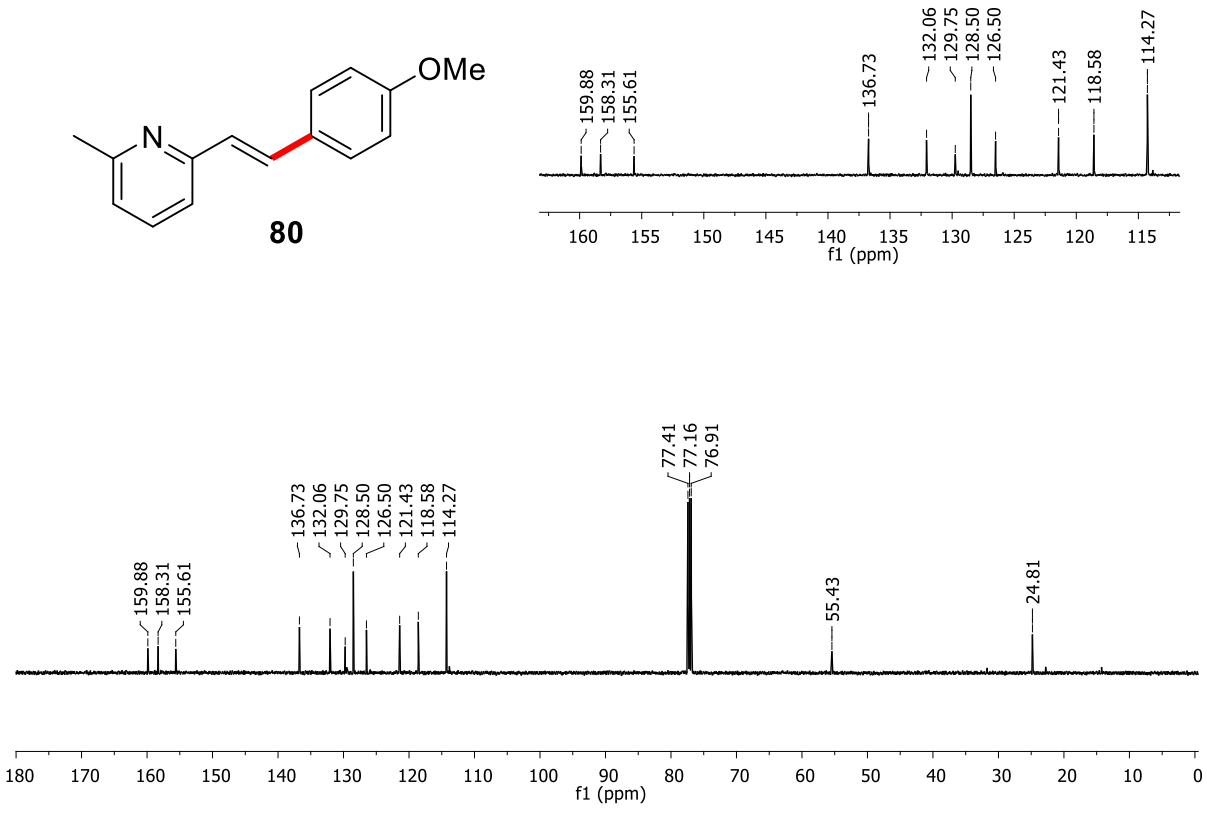

Fig. 191. [1] H NMR of (*E*)-3-(4-Methoxystyryl)thiophene in $CDCl_3$ **(81)**[74]

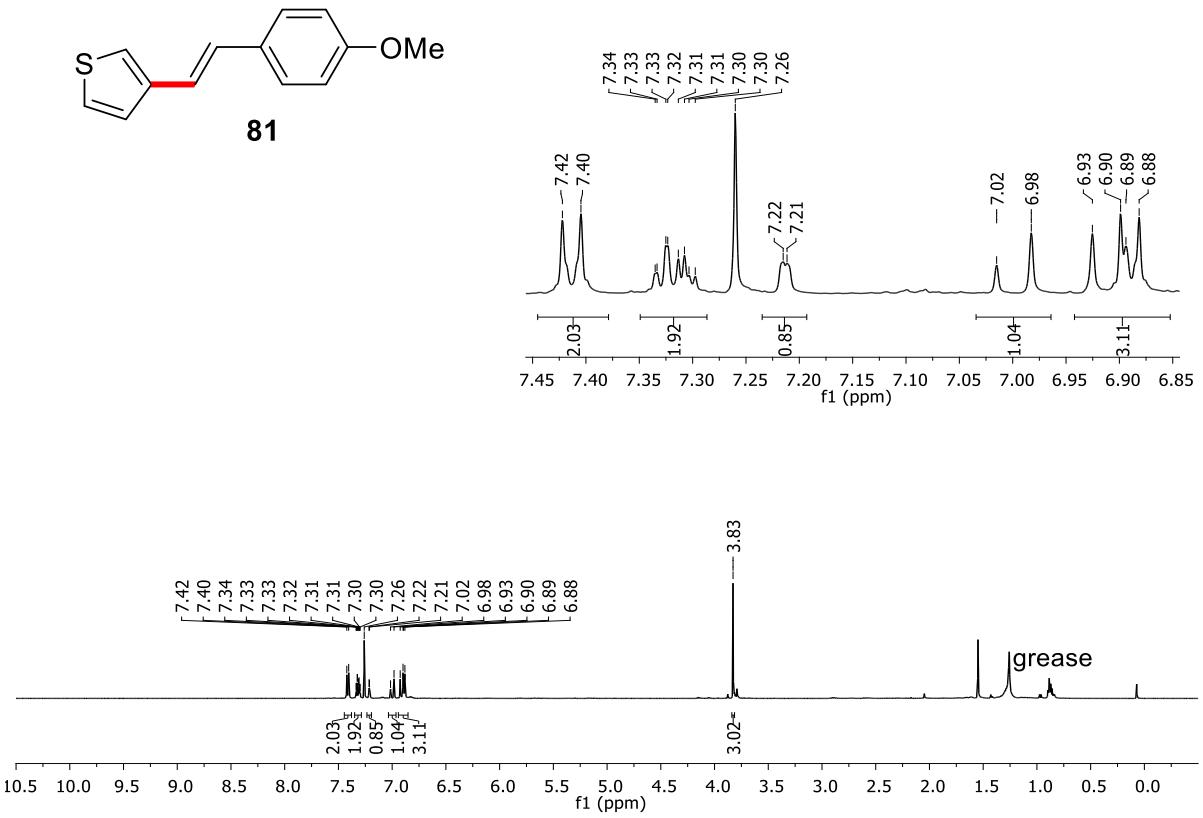

Fig. 192. [13]C NMR of (*E*)-3-(4-Methoxystyryl)thiophene in $CDCl_3$ **(81)**[74]