Supporting Information

Ligand- and copper-free Sonogashira and Heck couplings of (Het)aryl chlorides and bromides catalyzed by palladium nanoparticles supported on *in situ* generated $Al(OH)_3$

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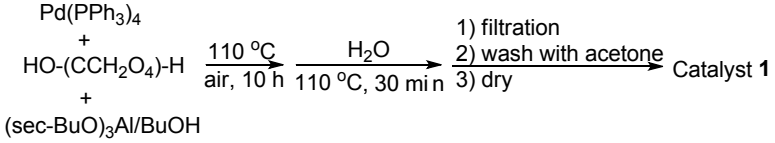
1. General

Unless otherwise indicated, all reagents were purchased from commercial sources and used without further purification. And deuterated solvents were purchased from Aldrich. Refinement of the mixed system through Column chromatography which was performed on silica gel (200-300 mesh) with petroleum ether (solvent A)/ethyl acetate (solvent B) gradients as elution. In addition, all yields were referred to isolated yields (average of two runs) of compounds unless otherwise specified. The known compounds were partly characterized by melting points (for solid samples), ^{1}H NMR, and compared to authentic samples or the literature data. Melting points were measured with a RD-II digital melting point apparatus and were uncorrected. ^{1}H NMR data were acquired at 300 K on a Bruker Advance 600 MHz spectrometer using $CDCl_3$ as solvent. Chemical shifts are reported in ppm from tetramethylsilane with the solvent $CDCl_3$ resonance as the internal standard ($CDCl_3$ = 7.26). Spectra are reported as follows: chemical shift (δ = ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration, and assignment.

2. Preparation of the Pd catalyst 1

A mixture of $Pd(PPh_3)_4$ (260 mg, 0.225 mmol), tetra (ethylene glycol) (418 mg, 2.20 mmol), 1-butanol (3 mL, 32.7 mmol), and aluminum tri-sec-butoxide (9.50 g, 38.5 mmol) was stirred at 110 ^{o}C for 10 h. Then water was dropwise added and the reaction system was heated to 110 ^{o}C and stirred constantly for another 0.5 h to form a black gel. Subsequently, filtering, washing with acetone, and drying the gel gave the catalyst **1** (2.75 g) as dark greyish-green powder under air atmosphere at room temperature (See Scheme 1).



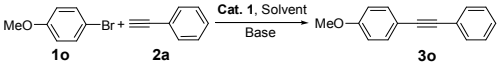
Scheme 1. Palladium nanoparticles catalyst **1**

3. Optimization of Sonogashira reaction of (Het)aryl halides

We first investigated the Sonogashira reaction between 4-methoxybromobenzene (**1o**) with phenylacetylene (**2a**) to optimize the reaction conditions (Table 1). Screening of common solvents showed that DMSO was the best choice over DMF, NMP, THF, toluene, and H_2O (Table 1, entries

1−5). The bases have a strong effect on the reaction yield, and NaOAc was the best among the bases screened, including K_2CO_3, Et_3N, K_3PO_4, and KOAc (Table 1, entries 2, 6−8 and 10). The reaction did not give **3o** at all when KOH was used (Table 1, entry 9). Of solvent dosage and the amount of bases examined, it turned out that 1.0 mL DMSO and 1.5 equiv. of NaOAc were perfect for the reaction, respectively (Table 1, entries 10−14). Subsequently, the evaluation of both catalyst loading and the amount of phenylacetylene revealed that 0.1 mol% nano-pd catalyst **1** gave the best results in the presence of 1.5 equiv of phenylacetylene (Table 1, entries 10 and 15−18). It is noteworthy that the addition of TBAB improved the result further, providing **3o** in 91% yield (Table 1, entry 19).

Table 1. Optimization of reaction conditions for the Sonogashira reaction of 4-methoxybromobenzene with phenylacetylene[a]



1o 2a 3o

Entry	Solvent (mL)	Base (equiv.)	Catalyst loading (mol% Pd)	**2a** (equiv.)	Yield (%)[b]
1	DMF (1.0)	K_2CO_3 (1.5)	0.1	1.5	21
2	DMSO (1.0)	K_2CO_3 (1.5)	0.1	1.5	36
3	NMP (1.0)	K_2CO_3 (1.5)	0.1	1.5	trace
4	H_2O (1.0)	K_2CO_3 (1.5)	0.1	1.5	Trace
5	PhMe (1.0)	K_2CO_3 (1.5)	0.1	1.5	N.D.[c]
6	DMSO (1.0)	Et_3N (1.5)	0.1	1.5	22
7	DMSO (1.0)	K_3PO_4 (1.5)	0.1	1.5	13
8	DMSO (1.0)	KOAc (1.5)	0.1	1.5	37
9	DMSO (1.0)	KOH (1.5)	0.1	1.5	N.D.
10	DMSO (1.0)	NaOAc (1.5)	0.1	1.5	62
11	DMSO (0.8)	NaOAc (1.5)	0.1	1.5	53
12	DMSO (1.2)	NaOAc (1.5)	0.1	1.5	49
13	DMSO (1.0)	NaOAc (1.0)	0.1	1.5	47
14	DMSO (1.0)	NaOAc (2.0)	0.1	1.5	51
15	DMSO (1.0)	NaOAc (1.5)	0.2	1.5	76
16	DMSO (1.0)	NaOAc (1.5)	0.3	1.5	75
17	DMSO (1.0)	NaOAc (1.5)	0.2	1.2	48
18	DMSO (1.0)	NaOAc (1.5)	0.2	1.8	71
19[d]	DMSO (1.0)	NaOAc (1.5)	0.2	1.5	91

a) Reactions were performed on a 0.2 mmol scale of 4-methoxybromobenzene under N_2 atmosphere at 120 °C for 40 h. b) Isolated yield. c) N.D. = Not detected. d) 0.5 equiv. of TBAB was added.

4. General procedure for Sonogashira reaction of (Het)aryl bromides and chlorides

To a tube equipped with a magnetic stir bar were added the nano-Pd catalyst **1** (8.8 mg, 0.2 mol% Pd), 1.5 equiv. of NaOAc (24.5 mg, 0.3 mmol), and 0.5 equiv. of TBAB (32.3 mg, 0.1 mmol) under N_2 atmosphere. Subsequently, aryl bromide (0.2 mmol), 1.5 equiv. of phenylacetylene (0.3 mmol) and the solvent (DMSO, 1.0 mL) in turn were added. The reaction system was then heated to 120 °C and stirred until aryl bromide was completely consumed as determined by TLC. At last, the reaction mixture was purified by silica gel column chromatography to afford the desired pure coupling product.

5. General procedure for Heck reaction of aryl bromides and chlorides

To a tube equipped with a magnetic stirring bar were added nano-Pd catalyst **1** (0.1 mol% Pd), K_3PO_4 (0.2 mmol), olefin (0.26 mmol) and aryl halide (0.2 mmol) in TBAB (0.3 g) or TBAB (0.3 g)+DMF (0.2 mL). The mixture under nitrogen was heated at 130 ^oC using an oil bath and the progress was monitored by TLC (petroleum ether/ethyl acetate, 50:1). After completing the reaction, the mixture was diluted with dichloromethane (30 mL) and H_2O. The organic layer was washed with brine (20 mL), dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The residue was purified by column chromatography.

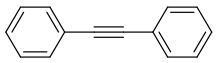
6. The procedure for recycled Sonogashira reaction

To a tube equipped with a magnetic stir bar were added NaOAc (24.5 mg, 0.3 mmol, 1.5 equiv.), TBAB (32.3 mg, 0.1 mmol, 0.5 equiv.) and the Pd catalyst **1** (19.6 mg, 0.7 mol % Pd) in turn. Subsequently, the solvent (DMSO, 1.0 mL), 4-methoxybromobenzene **1o** (1.0 equiv), and phenylacetylene **2a** (1.5 equiv.) were in turn added under N_2 atmosphere. The reaction mixture was then stirred at 120 ^oC for the specified time. The corresponding recycling reactions were carried out with the recovered Pd catalyst **1** that determined the amount of the substrate and reagents as reference at 120 ^oC. Reaction times were 40, 41, 42, 42, 43 and 45 h for cycles 1, 2, 3, 4, 5, and 6, respectively. After the reactions were fully performed as determined by TLC, 3 mL H_2O and 4 mL CH_2Cl_2 were added, and the reaction system was shaken well. Subsequently, the Pd catalyst **1** was recovered through membrane filtration and reused in the next reaction. After the organic phase was separated from the reaction system, the water phase was extracted with CH_2Cl_2 (3×4 mL). The organic layer was then combined and dried over anhydrous Na_2SO_4. At last, the solvent was removed in vacuum and the residue was purified by silica gel column chromatography

to afford the pure product **3o**.

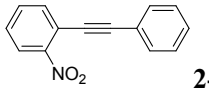
7. Characterization data for the coupling products

7.1 Characterization data for the Sonogashira-coupling products



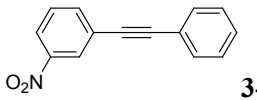
Diphenylacetylene (3a) [8m]

Following the general procedure, the 33.2 mg (0.186 mmol, 93% yield) of coupling product was provided after purification by column chromatography; White solid; mp. : 59-61 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.31-7.39 (m, 6H), 7.51-7.54 (m, 4H) ppm.



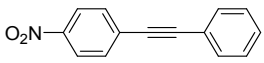
2-Nitro-diphenylacetylene (3b) [8h,10k]

Following the general procedure, 1-bromo-2-nitrobenzene provided the 41.5 mg (0.186 mmol, 93% yield) of coupling product after purification by column chromatography. 1-Chloro-2-nitrobenzene offered 30.3 mg (0.136 mmol, 68% yield) of coupling product. Orange solid; mp. : 51-52 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.36-7.39 (dd, J = 5.2, 1.9 Hz, 3H), 7.43-7.47 (m, 1H), 7.58-7.61(m, 3H), 7.69-7.72 (dd, J = 7.7, 1.4 Hz, 1H), 8.05-8.08 (dd, J = 8.3, 1.1 Hz, 1H) ppm.



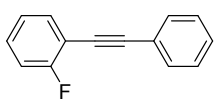
3-Nitro-diphenylacetylene (3c) [8h]

Following the general procedure, 1-bromo-3-nitrobenzene provided the 42.8 mg (0.192 mmol, 96% yield) of coupling product after purification by column chromatography. 1-Chloro-3-nitrobenzene offered 33.5 mg (0.15 mmol, 75% yield) of coupling product. Yellow solid; mp. : 68-70 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.36-7.40 (m, 3H), 7.50-7.53 (t, J = 8.0 Hz, 1H), 7.54-7.56 (m, 2H), 7.80-7.82 (dt, J = 7.6, 1.2 Hz, 1H), 8.15-8.18 (dq, J = 8.3, 1.0 Hz, 1H), 8.35-8.37 (t, J = 1.9 Hz, 1H) ppm.



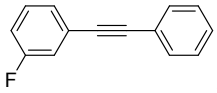
3-Nitro-diphenylacetylene (3d) [6f,10k]

Following the general procedure, 1-bromo-4-nitrobenzene provided the 42.8 mg (0.192 mmol, 96% yield) of coupling product after purification by column chromatography. 1-Chloro-4-nitrobenzene offered 36.1 mg (0.162 mmol, 81% yield) of coupling product. Yellow solid; mp. : 121-122 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.37-7.41 (m, 3H), 7.55-7.58 (m, 2H), 7.66-7.68(d, J = 8.9 Hz, 2H), 8.21-8.24 (d, J = 8.8 Hz, 2H) ppm.



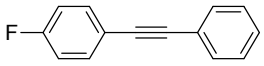
2-Fluoro-diphenylacetylene (3e) [16]

Following the general procedure, 1-bromo-2-fluorobenzene provided the 34.9 mg (0.178 mmol, 89% yield) of coupling product after purification by column chromatography. 1-Ethynyl-2-fluorobenzene offered 31.4 mg (0.16 mmol, 80% yield) of coupling product. Pale yellow solid; mp. : 45-46 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ =7.09-7.15 (m, 2H), 7.29-7.34 (m, 1H), 7.35-7.37 (dd, J = 4.7, 1.7 Hz, 3H), 7.50-7.54 (td, J = 7.4, 1.7 Hz, 1H), 7.55-7.58 (m, 2H) ppm.



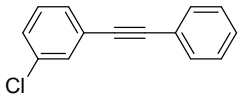
3-Fluoro-diphenylacetylene (3f) [17]

Following the general procedure, 1-bromo-3-fluorobenzene provided the 36.9 mg (0.188 mmol, 94% yield) of coupling product after purification by column chromatography. 1-Ethynyl-3-fluorobenzene offered 32.5 mg (0.166 mmol, 83% yield) of coupling product. Yellow solid; mp. : 36 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ =7.02-7.06 (m, 1H), 7.21-7.24 (dd, J = 8.9, 2.0 Hz, 1H), 7.30-7.33 (dd, J = 6.2, 5.2 Hz, 2H), 7.34-7.37 (dt, J = 7.7, 3.4 Hz, 3H), 7.52-7.55 (dd, J = 7.4, 3.8,Hz, 2H) ppm.



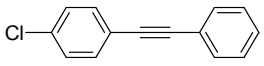
4-Fluoro-diphenylacetylene (3g) [10i]

Following the general procedure, 1-bromo-4-fluorobenzene provided the 37.3 mg (0.19 mmol, 95% yield) of coupling product after purification by column chromatography. 1-Ethynyl-4-fluorobenzene offered 34.5 mg (0.176 mmol, 88% yield) of coupling product. White solid; mp. : 109-110 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ =7.02-7.07 (tt, J = 9.7, 2.8 Hz, 2H), 7.33-7.37 (m, 3H), 7.49-7.53 (tt, J = 7.9, 2.6,Hz, 4H) ppm.



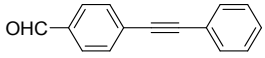
3-Chloro-diphenylacetylene (3h) [10j]

Following the general procedure, the 39.4 mg (0.186 mmol, 93% yield) of coupling product was provided after purification by column chromatography; Yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.27-7.30 (d, J = 7.7 Hz, 1H), 7.30-7.33 (ddd, J = 8.0, 1.7, 1.3 Hz, 1H), 7.34-7.38(m, 3H), 7.40-7.43 (dt, J = 7.4, 1.4 Hz, 1H) ppm.



4-Chloro-diphenylacetylene (3i) [8a,8p,12d]

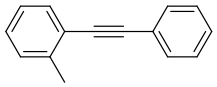
Following the general procedure, the 41.5 mg (0.196 mmol, 98% yield) of coupling product was provided after purification by column chromatography; White solid; mp: 109-110 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.30-7.33 (dt, J = 9.0, 2.3 Hz, 2H), 7.32-7.36 (m, 3H), 7.43-7.46(dt, J = 9.0, 2.4 Hz, 2H), 7.50-7.53 (m, 2H) ppm.



4-Formaldehydediphenylacetylene (3j) [6c]

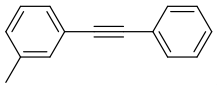
Following the general procedure, the 37.9 mg (0.184 mmol, 92% yield) of coupling product was provided after purification by column chromatography; White solid; mp. : 98-99 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.35-7.40 (m, 3H), 7.55-7.57 (m, 2H), 7.66-7.69 (d, J = 8.2 Hz, 2H), 7.86-

7.88 (d, J = 8.3 Hz, 2H), 10.02 (s, 1H) ppm.



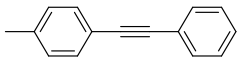
2-Methyl-diphenylacetylene (3k) [8g, 8h]

Following the general procedure, 1-bromo-2-methylbenzene provided the 31.2 mg (0.162 mmol, 81% yield) of coupling product after purification by column chromatography. 1-Ethynyl-2-methylbenzene offered 31.5 mg (0.164 mmol, 82% yield) of coupling product. Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.52 (s, 3H), 7.15-7.19 (dt, J = 12.1, 3.9 Hz, 1H), 7.22-7.24 (d, J = 4.1 Hz, 2H), 7.31-7.37 (m, 3H), 7.48-7.51 (d, J = 7.5 Hz, 1H), 7.52-7.55 (dt, J = 8.0, 1.7 Hz, 2H) ppm.



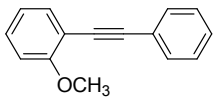
3-Methyl-diphenylacetylene (3l) [8h]

Following the general procedure, 1-bromo-3-methylbenzene provided the 36.9 mg (0.192 mmol, 86% yield) of coupling product after purification by column chromatography. 1-Ethynyl-3-methylbenzene offered 34.2 mg (0.178 mmol, 89% yield) of coupling product. Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.36 (s, 3H), 7.13-7.16 (d, J = 7.6 Hz, 1H), 7.22-7.25 (t, J = 7.7 Hz, 1H), 7.31-7.37 (m, 5H), 7.51-7.54 (dt, J = 8.0, 1.9 Hz, 2H) ppm.



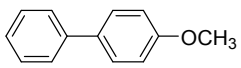
4-Methyl-diphenylacetylene (3m) [8g]

Following the general procedure, 1-bromo-4-methylbenzene provided the 35.3 mg (0.184 mmol, 92% yield) of coupling product after purification by column chromatography. 1-Chloro-4-methylbenzene offered 15.8 mg (0.082 mmol, 41% yield) of coupling product. 1-Ethynyl-4-methylbenzene offered 35.4 mg (0.184 mmol, 92% yield) of coupling product. White solid; mp. : 58-60 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.37 (s, 3H), 7.14-7.16 (d, J = 8.0 Hz, 2H), 7.31-7.36 (m, 3H), 7.41-7.44 (d, J = 7.9 Hz, 2H), 7.51-7.53 (m, 2H) ppm.



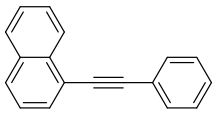
2-Methoxy-diphenylacetylene (3n) [8g, 8h]

Following the general procedure, the 33.7 mg (0.162 mmol, 81% yield) of coupling product was provided after purification by column chromatography; Brown oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 3.9 (s, 3H), 6.90-6.92 (d, J = 8.3 Hz, 1H), 6.92-6.96 (td, J = 7.5, 0.9 Hz, 1H), 7.29-7.36 (m, 4H), 7.49-7.51 (dd, J = 7.5, 1.7 Hz, 1H), 7.54-7.57 (dt, J = 8.2, 1.8 Hz, 1H) ppm.



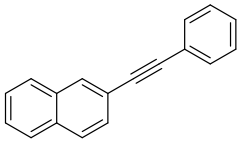
4-Methoxybiphenyl (3o) [8g, 6e]

Following the general procedure, the 37.9 mg (0.182 mmol, 91% yield) of coupling product was provided after purification by column chromatography; White solid; M.p. = 86-87 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.82 (s, 3H), 6.86-6.90 (m, 2H), 7.30-7.36 (m, 3H), 7.45-7.49 (m, 2H), 7.49-7.53 (m, 2H) ppm.



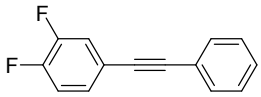
1-Naphthylphenylacetylene (3p) [6e]

Following the general procedure, 1-bromonaphthalene provided the 37.8 mg (0.166 mmol, 83% yield) of coupling product after purification by column chromatography. 1-Chloronaphthalene offered 24.2 mg (0.106 mmol, 53% yield) of coupling product. Colourless liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.36-7.41 (m, 3H), 7.45-7.48 (t, J = 7.9 Hz, 1H), 7.52-7.55(t, J = 7.2 Hz, 1H), 7.58-7.62 (t, J = 7.1 Hz, 1H),7.64-7.66 (d, J = 7.7 Hz, 2H), 7.76-7.78 (d, J = 7.1 Hz, 1H), 7.83-7.88 (dd, J = 13.9, 8.2 Hz, 2H), 8.43-8.46 (d, J = 8.3 Hz, 1H) ppm.



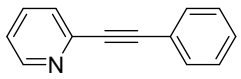
2-Naphthylphenylacetylene (3q) [10i]

Following the general procedure, the 39.3 mg (0.172 mmol, 86% yield) of coupling product was provided after purification by column chromatography; White solid; mp.: 115 - 116 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.33-7.40 (m, 3H), 7.48-7.52 (m, 2H), 7.57-7.60 (m, 3H), 7.80-7.84 (m, 3H), 8.06 (s, 1H) ppm.



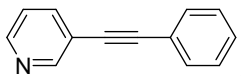
3,4-Difluoro-diphenylacetylene (3r) [18]

Following the general procedure, the 42.0 mg (0.196 mmol, 98% yield) of coupling product was provided after purification by column chromatography; White solid; mp.: 59-60^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.11-7.17 (m, 1H), 7.26-7.28 (dd, J = 4.0, 1.7 Hz, 1H), 7.32-7.35(m, 1H), 7.35-7.37 (t, J = 3.4 Hz, 3H), 7.51-7.53 (m, 2H) ppm.



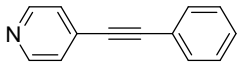
2-Phenylethynylpyridine (3s) [10i, 12b]

Following the general procedure, 2-bromopyridine provided the 25.1 mg (0.14 mmol, 70% yield) of coupling product after purification by column chromatography. 2-Ethynylpyridine offered 26.5 mg (0.148 mmol, 74% yield) of coupling product. White solid; mp.: 78-80^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.23-7.26 (ddd, J = 5.8, 4.9, 0.8 Hz, 1H), 7.34-7.39 (m, 3H), 7.52-7.55 (d, J = 7.8 Hz, 1H), 7.60-7.62 (m, 2H), 7.67-7.71 (td, J = 7.7, 1.8 Hz, 1H), 8.62-8.64 (d, J = 4.6 Hz, 1H) ppm.



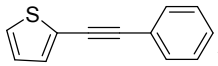
3-Phenylethynylpyridine (3t) [8m, 6e]

Following the general procedure, the 29.7 mg (0.166 mmol, 83% yield) of coupling product was provided after purification by column chromatography; White solid; mp.: 50-52^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.27-7.30 (dd, J = 7.7, 4.9 Hz, 1H), 7.36-7.38 (t, J = 3.3 Hz, 3H), 7.54-7.56 (m, 2H), 7.80-7.83 (dt, J = 7.9, 1.7 Hz, 1H), 8.55-8.56 (d, J = 3.2 Hz, 1H), 8.78 (s, 1H) ppm.



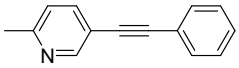
4-Phenylethynylpyridine (3u) [19]

Following the general procedure, the 32.6 mg (0.182 mmol, 91% yield) of coupling product was provided after purification by column chromatography; Yellow solid; mp.: 94-95 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.37-7.41 (m, 5H), 7.54-7.57 (m, 2H), 8.60-8.62 (d, J = 5.0 Hz, 2H) ppm.



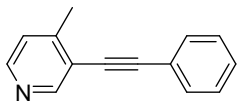
2-Phenylethynylthiophene (3v) [8m, 10i]

Following the general procedure, the 27.6 mg (0.15 mmol, 75% yield) of coupling product was provided after purification by column chromatography; White solid; mp.: 49-51 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 6.99-7.01 (dd, J = 4.8, 4.1 Hz, 1H), 7.27-7.29 (dd, J = 2.0, 1.0 Hz, 1H), 7.31-7.36 (m, 3H), 7.50-7.52 (m, 2H) ppm.



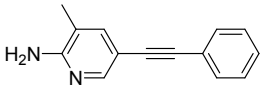
2-Methyl-5- Phenylethynylpyridine (3w) [20]

Following the general procedure, the 27.8 mg (0.144 mmol, 72% yield) of coupling product was provided after purification by column chromatography; Yellow solid; mp.: 47-48 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.58 (s, 3H), 7.13-7.16 (d, J = 8.0 Hz, 1H), 7.34-7.37 (m, 3H), 7.52-7.55 (m, 2H), 7.68-7.71 (dd, J = 8.0, 2.1 Hz, 1H), 8.65-8.67 (d, J = 1.5 Hz, 1H) ppm.



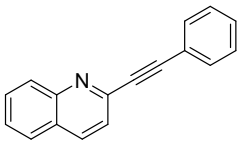
4-Methyl-3-Phenylethynylpyridine (3x) [21]

Following the general procedure, the 26.6 mg (0.138 mmol, 69% yield) of coupling product was provided after purification by column chromatography; Colourless liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.50 (s, 3H), 7.15-7.16 (d, J = 5.0 Hz, 1H), 7.35-7.39 (m, 3H), 7.54-7.57 (m, 2H), 8.39-8.41 (d, J = 5.0 Hz, 1H), 8.68 (s, 1H) ppm.



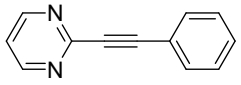
2-Amino-3-Methyl-5- Phenylethynylpyridine (3y) [21]

Following the general procedure, the 30.4 mg (0.146 mmol, 73% yield) of coupling product was provided after purification by column chromatography; Yellow solid; mp.: 97-98^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.14 (s, 3H), 4.64 (s, 2H), 7.30-7.36 (m, 3H), 7.44-7.45 (d, J = 0.9 Hz, 1H), 7.48-7.51 (m, 2H), 8.17 (s, 1H) ppm.



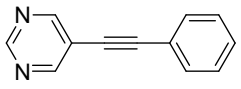
2-Phenylethynylquinoline (3z) [21]

Following the general procedure, the 32.5 mg (0.142 mmol, 71% yield) of coupling product was provided after purification by column chromatography; Yellow solid; mp.: 63-64 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.38-7.41 (m, 3H), 7.55-7.58 (m, 1H), 7.61-7.64 (d, J = 8.4 Hz, 1H), 7.66-7.69 (m, 2H), 7.73-7.76 (m, 1H), 7.81-7.83 (d, J = 7.9 Hz, 1H) ppm.



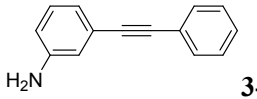
2-Phenylethynylpyrimidine (3za) [12b]

Following the general procedure, the 29.2 mg (0.162 mmol, 81% yield) of coupling product was provided after purification by column chromatography; Yellow solid; mp.: 84-85 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.24-7.27 (dd, J = 8.0, 3.1 Hz, 1H), 7.36-7.44 (m, 3H), 7.66-7.69 (dd, J = 7.8, 1.0 Hz, 2H), 8.76-8.77 (d, J = 4.9 Hz, 2H) ppm.



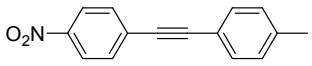
5-Phenylethynylpyrimidine (3zb) [12b]

Following the general procedure, the 33.5 mg (0.186 mmol, 93% yield) of coupling product was provided after purification by column chromatography; Yellow solid; mp.: 52-53 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.37-7.43 (m, 3H), 7.54-7.58 (m, 2H), 8.86 (s, 2H), 9.15 (s, 1H) ppm.



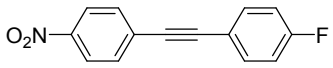
3-Aminodiphenylacetylene (3zc) [22]

Following the general procedure, the 31.3 mg (0.162 mmol, 81% yield) of coupling product was provided after purification by column chromatography; Pale yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 3.68 (s, 2H), 6.64-6.67 (ddd, J = 3.3, 2.5, 1.0 Hz, 1H), 6.85-6.87 (t, J = 2.1 Hz, 1H), 6.93-6.96 (dt, J = 7.5, 1.1 Hz, 1H), 7.11-7.15 (t, J = 7.7 Hz, 1H), 7.30-7.36 (m, 3H), 7.50-7.53 (m, 2H) ppm.



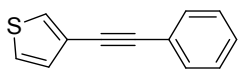
1-methyl-4-(2-(4-nitrophenyl)ethynyl)benzene (3zd) [6d]

Following the general procedure, the 40.8 mg (0.172 mmol, 86% yield) of coupling product was provided after purification by column chromatography; Yellow solid; mp.: 157-158 ^{o}C; ^{1}H NMR (600 MHZ, $CDCl_3$): δ= 8.21-8.23 (m, 2H), 7.64-7.67 (m, 2H), 7.45-7.47 (d, J = 8.1Hz, 2H), 7.19-7.21 (d, J=7.8Hz, 2H), 2.40 (s, 3H).



1-fluoro-4-(2-(4-nitrophenyl)ethynyl)benzene (3ze) [6f]

Following the general procedure, the 39.6 mg (0.164 mmol, 82% yield) of coupling product was provided after purification by column chromatography; Pale yellow solid, m.p.: 111-113 ^{o}C; ^{1}H NMR (600 MHZ, $CDCl_3$): δ = 8.22-8.24 (d, J = 8.8 Hz, 2H), 7.65-7.67 (d, J = 8.7 Hz, 2H), 7.54-7.57 (m, 2H), 7.07-7.11 (t, J = 8.7 Hz, 2H).

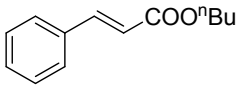


3-Phenylethynylthiophene (3zf) [12a, 12b]

Following the general procedure, the 11.4 mg (0.062 mmol, 31% yield) of coupling product was provided after purification by column chromatography; White solid, mp.: 48-50 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.16-7.18 (dd, J = 5.0, 1.0 Hz, 2H), 7.28-7.30 (dd, J =5.0, 3.0 Hz, 2H), 7.33-

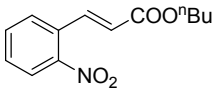
7.35 (m, 1H), 7.51-7.53(m, 1H), 7.58-7.60 (dd, J = 2.9, 1.0 Hz, 2H) ppm.

7.2 Characterization data for the Heck coupling products



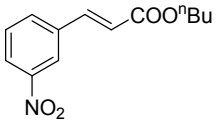
***trans*-Cinnamic Acid Butyl Ester (5a)** [7c, 8p]

Following the general procedure, the 40.4 mg (0.198 mmol, 99% yield) of coupling product was provided after purification by column chromatography; Colourless oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.95-0.98 (t, J = 7.4 Hz, 3H), 1.40-1.47 (m, 2H), 1.66-1.72 (m, 2H), 4.19-4.23 (t, J = 6.7 Hz, 2H), 6.43-6.46 (d, J = 16.0 Hz, 1H), 7.36-7.40 (m, 3H), 7.51-7.54 (m, 2H), 7.66-7.70 (d, J = 16.0 Hz, 1H) ppm.



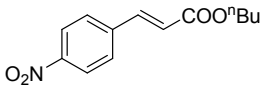
***trans*-2-nitro-Cinnamic Acid Butyl Ester (5b)** [9g]

Following the general procedure, 1-bromo-2-nitrobenzene provided the 45.3 mg (0.182 mmol, 91% yield) of coupling product after purification by column chromatography. 1-Chloro-2-nitrobenzene offered 46.4 mg (0.186 mmol, 93% yield) of coupling product. Pale yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.95-0.99 (t, J = 7.4 Hz, 3H), 1.41-1.48 (m, 2H), 1.68-1.73 (m, 2H), 4.22-4.25 (t, J = 6.7 Hz, 2H), 6.36-6.39 (d, J = 15.8 Hz, 1H), 7.53-7.57 (m, 1H), 7.64-7.68 (m, 2H), 8.04-8.06 (d, J = 8.5 Hz, 1H), 8.09-8.13 (d, J = 15.8Hz, 1H) ppm.



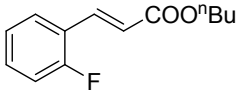
***trans*-3-nitro-Cinnamic Acid Butyl Ester (5c)** [23]

Following the general procedure, 1-bromo-3-nitrobenzene provided the 44.9 mg (0.18 mmol, 90% yield) of coupling product after purification by column chromatography. 1-Chloro-3-nitrobenzene offered 42.4 mg (0.17 mmol, 85% yield) of coupling product. Yellow solid; M.p. = 53-55 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.96-1.0 (t, J = 7.4 Hz, 3H), 1.41-1.49 (m, 2H), 1.68-1.74 (m, 2H), 4.23-7.26 (t, J = 6.6 Hz, 2H), 6.55-6.59 (d, J = 16.0 Hz, 1H), 7.57-7.61 (t, J = 8.0 Hz, 1H), 7.70-7.73 (d, J = 16.1 Hz, 1H), 7.82-7.84 (d, J = 7.6 Hz, 1H), 8.22-8.25 (m, 1H), 8.38-8.40(t, J = 1.9 Hz, 1H) ppm.



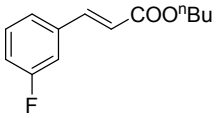
***trans*-4-nitro-Cinnamic Acid Butyl Ester (5d)** [7c, 7f]

Following the general procedure, 1-bromo-4-nitrobenzene provided the 42.4 mg (0.17 mmol, 85% yield) of coupling product after purification by column chromatography. 1-Chloro-4-nitrobenzene offered 37.9 mg (0.152 mmol, 76% yield) of coupling product. Yellow solid; M.p. = 63-65 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.96-0.99 (t, J = 7.4 Hz, 3H), 1.41-1.48 (m, 2H), 1.68-1.74 (m, 2H), 4.23-4.26 (t, J = 6.7 Hz, 2H), 6.55-6.59 (d, J = 16.1 Hz, 1H), 7.67-7.69 (d, J = 8.8 Hz, 2H), 7.69-7.73 (d, J = 16.1 Hz, 1H), 8.25-8.27 (d, J = 8.8 Hz, 2H) ppm.



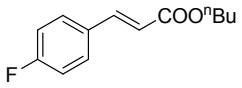
***trans*-2-fiuo-Cinnamic Acid Butyl Ester (5e)** [7c, 7f]

Following the general procedure, the 40.9 mg (0.184 mmol, 92% yield) of coupling product was provided after purification by column chromatography; Pale yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.95-0.98 (t, J = 7.4 Hz, 3H), 1.40-1.48 (m, 2H), 1.67-1.72 (m, 2H), 4.20-4.24 (t, J = 6.7 Hz, 2H), 6.52-6.56 (d, J = 16.2 Hz, 1H), 7.07-7.12 (m, 1H), 7.14-7.18 (m, 1H), 7.33-7.37 (m, 1H), 7.52-7.56 (m, 1H), 7.79-7.83 (d, J = 16.2 Hz, 1H) ppm.



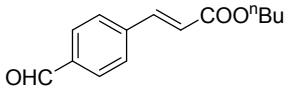
***trans*-3-fiuo-Cinnamic Acid Butyl Ester (5f)** [24]

Following the general procedure, the 39.6 mg (0.178 mmol, 89% yield) of coupling product was provided after purification by column chromatography; Pale yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.95-0.99 (t, J = 7.4 Hz, 3H), 1.40-1.47 (m, 2H), 1.66-1.72 (m, 2H), 4.20-4.23 (t, J = 6.7 Hz, 2H), 6.42-6.45 (d, J = 16.0 Hz, 1H), 7.06-7.10 (m, 1H), 7.21-7.24 (m, 1H), 7.28-7.31 (d, J = 7.8 Hz, 1H), 7.33-7.38 (m, 1H), 7.61-7.65 (d, J = 16.0 Hz, 1H) ppm.



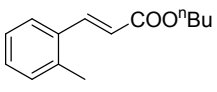
***trans*-4-fiuoro-Cinnamic Acid Butyl Ester (5g)** [25]

Following the general procedure, the 36.5 mg (0.164 mmol, 82% yield) of coupling product was provided after purification by column chromatography; Pale yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.95-0.98 (t, J = 7.4 Hz, 3H), 1.40-1.47 (m, 2H), 1.66-1.72 (m, 2H), 4.19-4.23 (t, J = 6.7 Hz, 2H), 6.35-6.38 (d, J = 16.1 Hz, 1H), 7.06-7.10 (m, 2H), 7.50-7.53 (m, 2H), 7.62-7.66 (d, J = 16.0 Hz, 1H) ppm.



***trans*-butyl 3-(4-formylphenyl)acrylate (5h)** [7c, 7f]

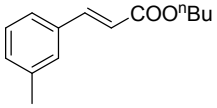
Following the general procedure, the 39.5 mg (0.17 mmol, 85% yield) of coupling product was provided after purification by column chromatography; Yellow solid; M.p. = 35-36 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.96-0.99 (t, J = 7.4 Hz, 3H), 1.41-1.48 (m, 2H), 1.67-1.73 (m, 2H), 4.22-4.25 (t, J = 6.7 Hz, 2H), 6.54-6.58 (d, J = 16.0 Hz, 1H), 7.67-7.69 (d, J = 8.2 Hz, 2H), 7.69-7.72 (d, J = 16.2 Hz, 1H), 7.89-7.92 (d, J = 8.2 Hz, 2H), 10.03 (s, 1H) ppm.



***trans*-2-Methyl-Cinnamic Acid Butyl Ester (5i)** [9g]

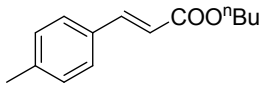
Following the general procedure, 1-bromo-2-methylbenzene provided the 38.0 mg (0.174 mmol, 87% yield) of coupling product after purification by column chromatography. 1-Chloro-2-methylbenzene offered 31.4 mg (0.144 mmol, 72% yield) of coupling product. Pale yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.95-0.99 (t, J = 7.4 Hz, 3H), 1.41-1.48 (m, 2H), 1.67-1.72 (m, 2H),

2.44 (s, 3H), 4.20-4.23 (t, J = 6.7 Hz, 2H), 6.34-6.38 (d, J = 15.9 Hz, 1H), 7.19-7.22 (t, J = 7.4 Hz, 2H), 7.25-7.29 (m, 1H), 7.54-7.56 (d, J = 7.6 Hz, 1H), 7.96-7.99 (d, J = 15.9 Hz, 1H) ppm.



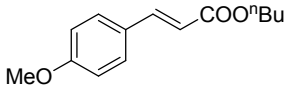
trans-3-Methyl-Cinnamic Acid Butyl Ester (5j) [9g]

Following the general procedure, 1-bromo-3-methylbenzene provided the 36.7 mg (0.168 mmol, 84% yield) of coupling product after purification by column chromatography. 1-Chloro-3-methylbenzene offered 25.3 mg (0.116 mmol, 58% yield) of coupling product. Pale yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.95-0.98 (t, J = 7.4 Hz, 3H), 1.40-1.47 (m, 2H), 1.66-1.72 (m, 2H), 2.37 (s, 3H), 4.19-4.22 (t, J = 6.7 Hz, 2H), 6.41-6.45 (d, J = 16.0 Hz, 1H), 7.18-7.20 (d, J = 7.4 Hz, 1H), 7.25-7.29 (t, J = 15.3 Hz, 1H), 7.32-7.34 (d, J = 8.5 Hz, 2H), 7.63-7.67 (d, J = 16.0 Hz, 1H) ppm.



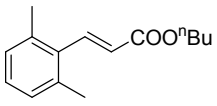
trans-4-Methyl-Cinnamic Acid Butyl Ester (5k) [7c,9g]

Following the general procedure, 1-bromo-4-methylbenzene provided the 34.9 mg (0.16 mmol, 80% yield) of coupling product after purification by column chromatography. 1-Chloro-4-methylbenzene offered 17.9 mg (0.082 mmol, 41% yield) of coupling product. Pale yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.95-0.98 (t, J = 7.4 Hz, 3H), 1.40-1.47 (m, 2H), 1.66-1.71 (m, 2H), 2.37 (s, 3H), 4.18-4.22 (t, J = 6.7 Hz, 2H), 6.38-6.41 (d, J = 16.0 Hz, 1H), 7.17-7.20 (d, J = 8.0 Hz, 2H), 7.41-7.43 (d, J = 8.1 Hz, 2H), 7.64-7.67 (d, J = 16.0 Hz, 1H) ppm.



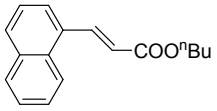
trans-4-methoxyl-Cinnamic Acid Methyl Ester (5l) [7c,9g]

Following the general procedure, the 33.7 mg (0.144 mmol, 72% yield) of coupling product was provided after purification by column chromatography; Pale yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.95-0.98 (t, J = 7.4 Hz, 3H), 1.40-1.47 (m, 2H), 1.66-1.71 (m, 2H), 3.83 (s, 3H), 4.18-4.21 (t, J = 6.7 Hz, 2H), 6.29-6.33 (d, J = 16.0 Hz, 1H), 6.89-6.91 (dt, J = 9.7, 2.9 Hz, 2H), 7.46-7.49 (dt, J = 9.7, 2.9 Hz, 2H), 7.62-7.65 (d, J = 16.0 Hz, 1H) ppm.



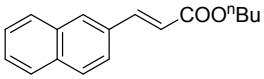
(_E_)-butyl 3-(2,6-dimethylphenyl)acrylate (5m) [26]

Following the general procedure, the 26.5 mg (0.114 mmol, 57% yield) of coupling product was provided after purification by column chromatography; Yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.95-0.99 (t, J = 7.4 Hz, 3H), 1.41-1.48 (m, 2H), 1.67-1.73 (m, 2H), 2.35 (s, 6H), 4.21-4.24 (t, J = 6.7 Hz, 2H), 6.05-6.09 (d, J = 16.4 Hz, 1H), 7.05-7.07 (d, J = 7.5 Hz, 2H), 7.11-7.15 (m, 1H), 7.82-7.86 (d, J = 16.4 Hz, 1H) ppm.



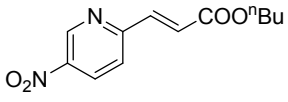
(*E*)-butyl 3-(naphthalen-1-yl)acrylate (5n) [7f,9g]

Following the general procedure, the 43.7 mg (0.172 mmol, 86% yield) of coupling product was provided after purification by column chromatography; Pale yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.97-1.01 (t, *J* = 7.4 Hz, 3H), 1.43-1.51 (m, 2H), 1.70-1.76 (m, 2H), 4.25-4.28 (t, *J* = 6.7 Hz, 2H), 6.51-6.55 (d, *J* = 15.8 Hz, 1H), 7.46-7.50 (t, *J* = 7.7 Hz, 1H), 7.51-7.54 (td, *J* = 8.1, 1.1 Hz, 1H), 7.55-7.59 (m, 1H), 7.74-7.76 (d, *J* = 7.2 Hz, 1H), 7.86-7.90 (t, *J* = 8.9 Hz, 2H), 8.18-8.21 (d, *J* = 8.5 Hz, 1H), 8.50-8.54 (d, *J* = 15.8 Hz, 1H) ppm.



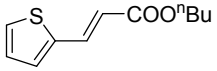
(*E*)-butyl 3-(naphthalen-2-yl)acrylate (5o) [27]

Following the general procedure, the 41.2 mg (0.162 mmol, 81% yield) of coupling product was provided after purification by column chromatography; Yellow solid; M.p. = 60-61 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.96-1.00 (t, *J* = 7.4 Hz, 3H), 1.43-1.50 (m, 2H), 1.68-1.74 (m, 2H), 4.22-4.25 (t, *J* = 6.7 Hz, 2H), 6.54-6.57 (d, *J* = 15.9 Hz, 1H), 7.49-7.54 (m, 2H), 7.66-7.69 (dd, *J* = 8.5, 1.6 Hz, 1H), 7.82-7.87 (m, 4H), 7.93 (s, 1H) ppm.



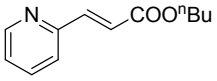
(*E*)-butyl 3-(5-nitropyridin-2-yl)acrylate (5p) [9n]

Following the general procedure, the 38.1 mg (0.152 mmol, 76% yield) of coupling product was provided after purification by column chromatography; Yellow solid; M.p. = 55-57 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.96-0.99 (t, *J* = 7.4 Hz, 3H), 1.41-1.48 (m, 2H), 1.68-1.74 (m, 2H), 4.24-4.27 (t, *J* = 6.7 Hz, 2H), 6.64-6.67 (d, *J* = 16.1 Hz, 1H), 7.71-7.75 (d, *J* = 16.1 Hz, 1H), 8.14-8.17 (dd, *J* = 8.5, 2.2 Hz, 1H), 8.29-8.31 (d, *J* = 8.4 Hz, 1H), 8.75-8.77 (d, *J* = 2.0 Hz, 1H) ppm.



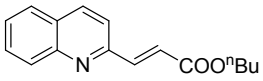
(*E*)-butyl 3-(thiophen-2-yl)acrylate (5q) [9q, 9u]

Following the general procedure, the 34.5 mg (0.164 mmol, 82% yield) of coupling product was provided after purification by column chromatography; Pale yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.94-0.98 (t, *J* = 7.4 Hz, 3H), 1.39-1.47 (m, 2H), 1.65-1.71 (m, 2H), 4.18-4.21 (t, *J* = 6.7 Hz, 2H), 6.22-6.26 (d, *J* = 15.7 Hz, 1H), 7.03-7.06 (dd, J = 5.0, 3.5 Hz, 1H), 7.24-7.25 (d, *J* = 3.3 Hz, 1H), 7.36-7.37 (d, *J* = 5.0 Hz, 1H), 7.76-7.79 (d, *J* = 15.8 Hz, 1H) ppm.



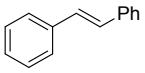
(*E*)-butyl 3-(pyridin-2-yl)acrylate (5r) [9n]

Following the general procedure, the 17.7 mg (0.086 mmol, 43% yield) of coupling product was provided after purification by column chromatography; Pale yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.95-0.98 (t, *J* = 7.4 Hz, 3H), 1.41-1.48 (m, 2H), 1.66-1.72 (m, 2H), 4.21-4.24 (t, *J* = 6.7 Hz, 2H), 6.91-6.94 (d, *J* = 15.7 Hz, 1H), 7.25-7.29 (m, 1H), 7.42-7.45 (d, *J* = 7.7 Hz, 1H), 7.67 (s, 1H), 7.70 (s, 1H), 7.70-7.74 (td, *J* = 7.7, 5.5 Hz, 1H), 8.64-8.66 (m, 1H) ppm.



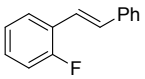
(*E*)-butyl 3-(quinolin-2-yl)acrylate (5s) [28]

Following the general procedure, the 38.3 mg (0.15 mmol, 75% yield) of coupling product was provided after purification by column chromatography; Yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 0.96-0.99 (t, J = 7.4 Hz, 3H), 1.43-1.50 (m, 2H), 1.69-1.74 (m, 2H), 4.24-4.27 (t, J = 6.7 Hz, 2H), 6.97-6.71 (d, J = 15.8 Hz, 1H), 7.54-7.58 (m, 1H), 7.60-7.63 (d, J = 8.5 Hz, 1H), 7.72-7.76 (m, 1H), 7.80-7.82 (d, J = 8.0 Hz, 1H), 7.87-7.91 (d, J = 15.9 Hz, 1H), 8.09-8.12 (d, J = 8.7 Hz, 1H), 8.16-8.19 (d, J = 8.5 Hz, 1H) ppm.



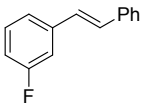
***trans*-Stilbene (5t)** [10a]

Following the general procedure, the 30.6 mg (0.17 mmol, 85% yield) of coupling product was provided after purification by column chromatography; White solid; M.p. = 121-123 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.12 (s, 2H), 7.24-7.28 (m, 2H), 7.34-7.38 (m, 4H), 7.51-7.53 (dt, J = 8.4, 1.3 Hz, 4H) ppm.



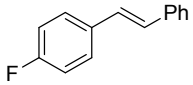
***trans*-2-florostilbene (5u)** [29]

Following the general procedure, the 35.3 mg (0.178 mmol, 89% yield) of coupling product was provided after purification by column chromatography; White solid; M.p. = 94-95 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.05-7.09 (m, 1H), 7.12-7.16 (t, J = 7.4 Hz, 1H), 7.19 (s, 1H), 7.21-7.25 (m, 1H), 7.26-7.30 (m, 2H), 7.35-7.39 (t, J = 7.6 Hz, 2H), 7.53-7.55 (d, J = 7.5 Hz, 2H), 7.59-7.63 (td, J = 7.7, 1.5 Hz, 1H) ppm.



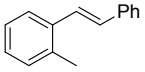
***trans*-3-florostilbene (5v)** [30]

Following the general procedure, the 34.5 mg (0.174 mmol, 87% yield) of coupling product was provided after purification by column chromatography; White solid; M.p. = 66-68 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 6.93-6.97 (m, 1H), 7.04-7.13 (q, J = 16.3 Hz, 2H), 7.20-7.23 (dt, J = 10.3, 2.2 Hz, 1H), 7.25-7.33 (m, 3H), 7.35-7.39 (t, J = 7.5 Hz, 2H), 7.50-7.53 (m, 2H) ppm.



***trans*-4-florostilbene (5w)** [9u]

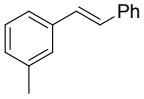
Following the general procedure, the 33.7 mg (0.17 mmol, 85% yield) of coupling product was provided after purification by column chromatography; White solid; M.p. = 121-123 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.00-7.09 (m, 4H), 7.25-7.28 (m, 1H), 7.34-7.38 (m, 2H), 7.46-7.51 (m, 4H) ppm.



***trans*-2-methylstilbene (5x)** [7a]

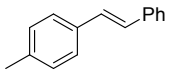
Following the general procedure, the 31.9 mg (0.164 mmol, 82% yield) of coupling product was

provided after purification by column chromatography; White solid; M.p. = 30-32 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.44 (s, 3H), 6.99-7.03 (d, J = 16.1 Hz, 1H), 7.18-7.20 (d, J = 4.0 Hz, 2H), 7.20-7.24 (m, 1H), 7.26-7.29 (t, J = 7.3 Hz, 1H), 7.33 (s, 1H), 7.36-7.39 (m, 2H), 7.52-7.55 (d, J = 7.4 Hz, 2H), 7.59-7.62 (d, J = 7.4 Hz, 1H) ppm.



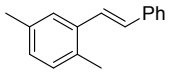
***trans*-3-methylstilbene (5y)** [14b]

Following the general procedure, the 32.6 mg (0.168 mmol, 84% yield) of coupling product was provided after purification by column chromatography; White solid; M.p. = 47-49 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.38 (s, 3H), 7.08 (s, 1H), 7.09-7.10 (d, J = 2.3 Hz, 2H), 7.23-7.27 (m, 2H), 7.31-7.33(d, J = 7.9 Hz, 1H), 7.34-7.37 (m, 3H), 7.50-7.52 (d, J = 7.4 Hz, 2H) ppm.



***trans*-4-methylstilbene (5z)** [7a, 10a]

Following the general procedure, the 34.2 mg (0.176 mmol, 88% yield) of coupling product was provided after purification by column chromatography; White solid; M.p. = 119-120 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.38 (s, 3H), 7.09-7.11 (d, J = 5.3 Hz, 2H), 7.18-7.20 (d, J = 7.9 Hz, 2H), 7.26-7.29 (m, 1H), 7.35-7.39 (m, 2H), 7.43-7.45 (d, J = 8.1 Hz, 2H), 7.51-7.54 (m, 2H) ppm.



***trans*-2,5-dimethylstilbene (5za)** [31]

Following the general procedure, the 33.3 mg (0.16 mmol, 80% yield) of coupling product was provided after purification by column chromatography; White solid; M.p. = 42-43 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.36 (s, 3H), 2.39 (s, 3H), 6.98-7.00 (d, J = 9.1 Hz, 1H), 7.01 (s, 1H), 7.06-7.09 (d, J = 7.6 Hz, 1H), 7.26-7.28 (m, 1H), 7.30-7.34 (d, J = 16.1 Hz, 1H), 7.35-7.38 (t, J = 7.6 Hz, 2H), 7.42 (s, 1H), 7.52-7.54 (d, J = 7.4 Hz, 2H) ppm.

8. References

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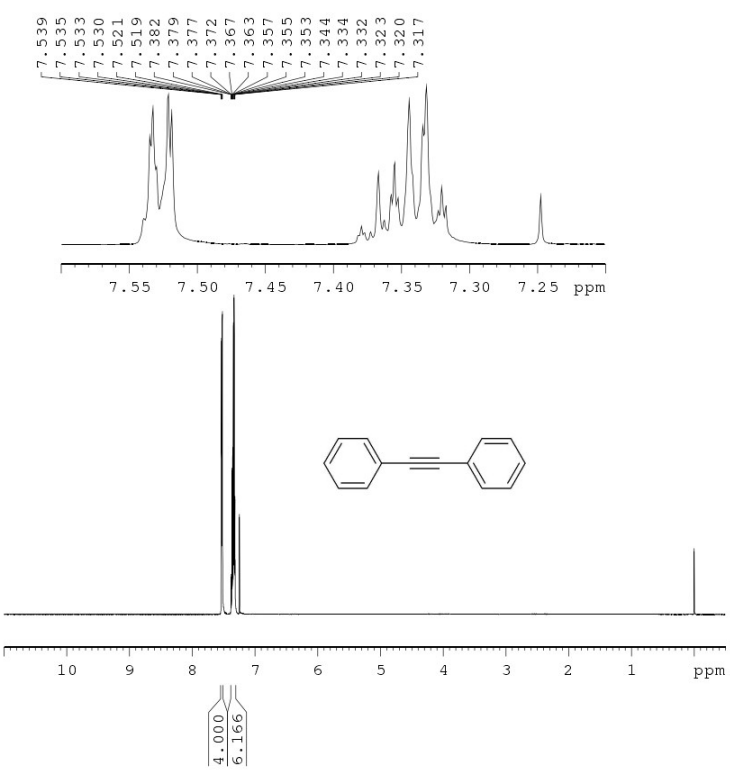
10. ^{1}H-NMR spectra for products

10.1 ^{1}H-NMR spectra for the Sonogashira coupling products

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PC                 1.00
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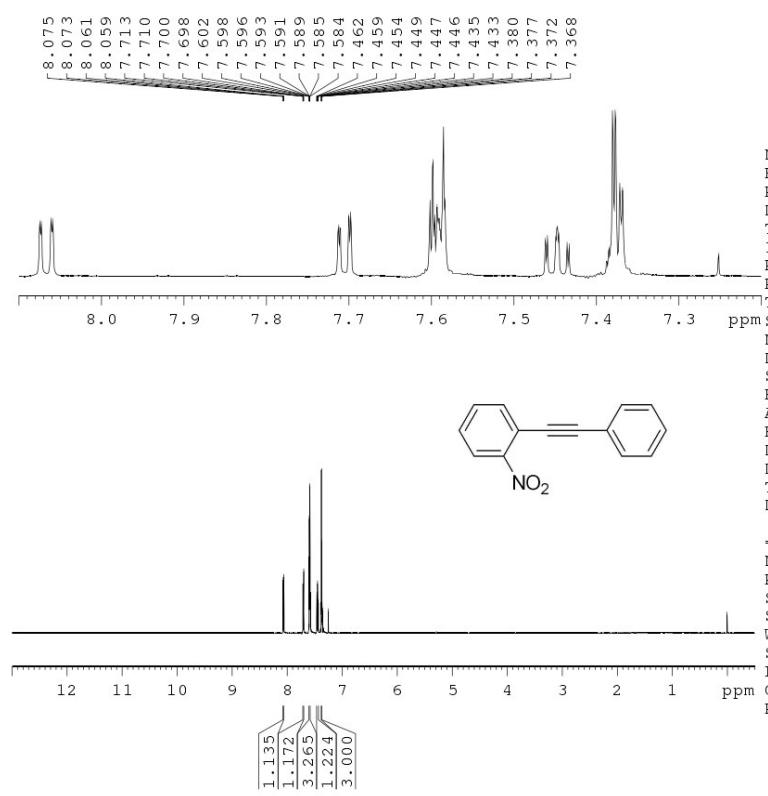
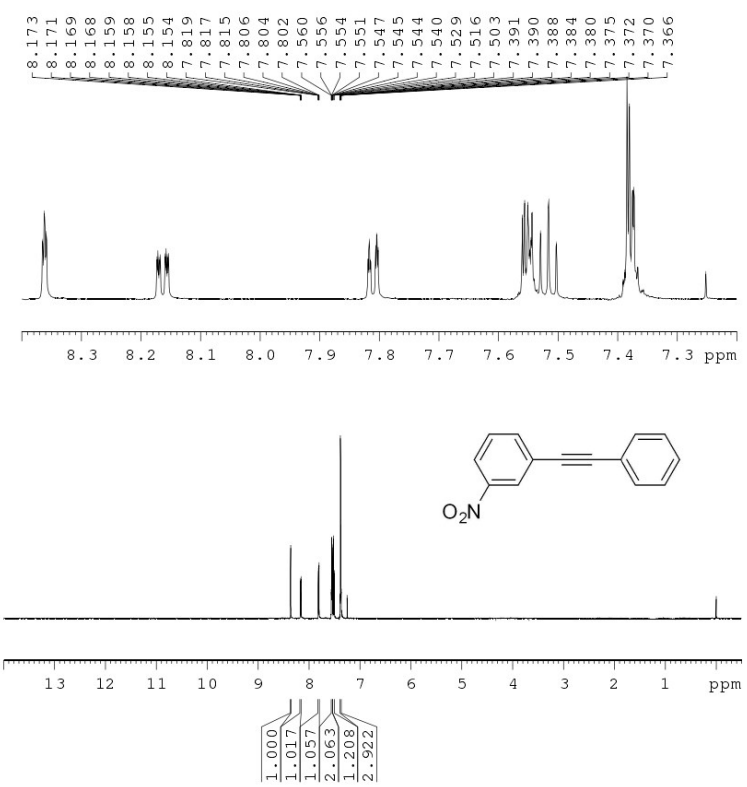
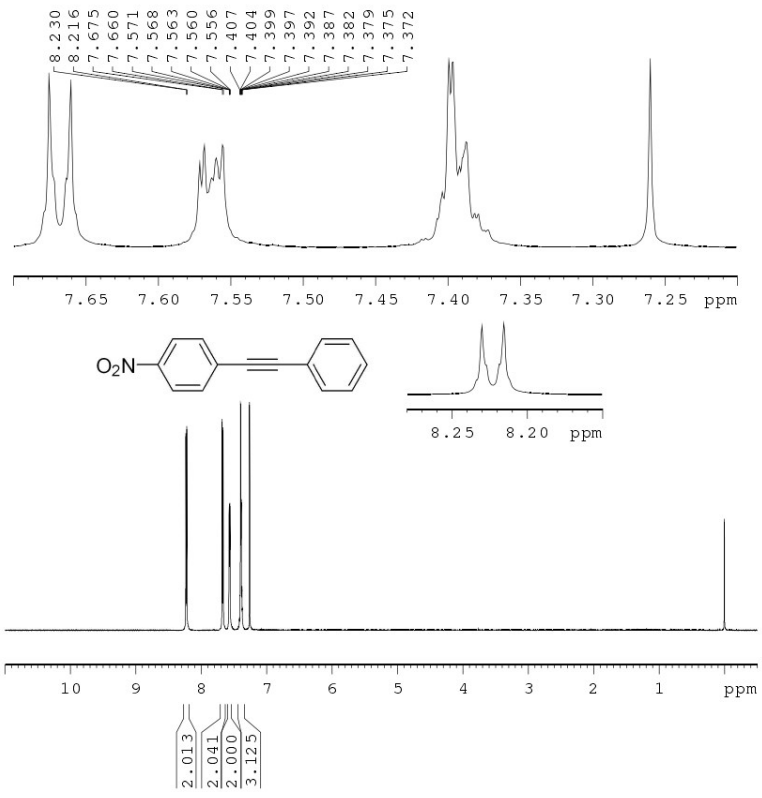



```
NAME           lizhipeng
EXPNO                 14
PROCNO                 1
Date_           20140318
Time               16.57
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     2
DS                     2
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                  90.5
DW                40.533 usec
DE                 6.50 usec
TE                296.9 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
SI                32768
SF          600.1300222 MHz
WDW                  GM
SSB                   0
LB                -0.10 Hz
GB                  0.1
PC                 1.00
```



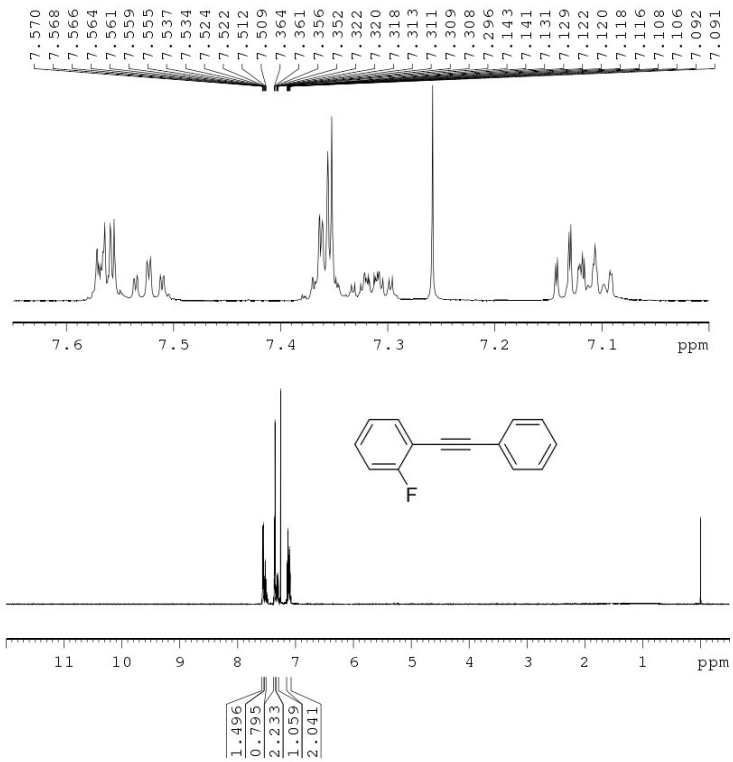
BRUKER

```
NAME            lizhipeng
EXPNO                  15
PROCNO                  1
Date_            20140318
Time                17.02
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      2
DS                      2
SWH             12335.526 Hz
FIDRES          0.188225 Hz
AQ              2.6564426 sec
RG                    101
DW                 40.533 usec
DE                  6.50 usec
TE                 296.9 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
SI                    32768
SF           600.1300222 MHz
WDW                    EM
SSB                     0
LB                   0.10 Hz
GB                      0
PC                   1.00
```

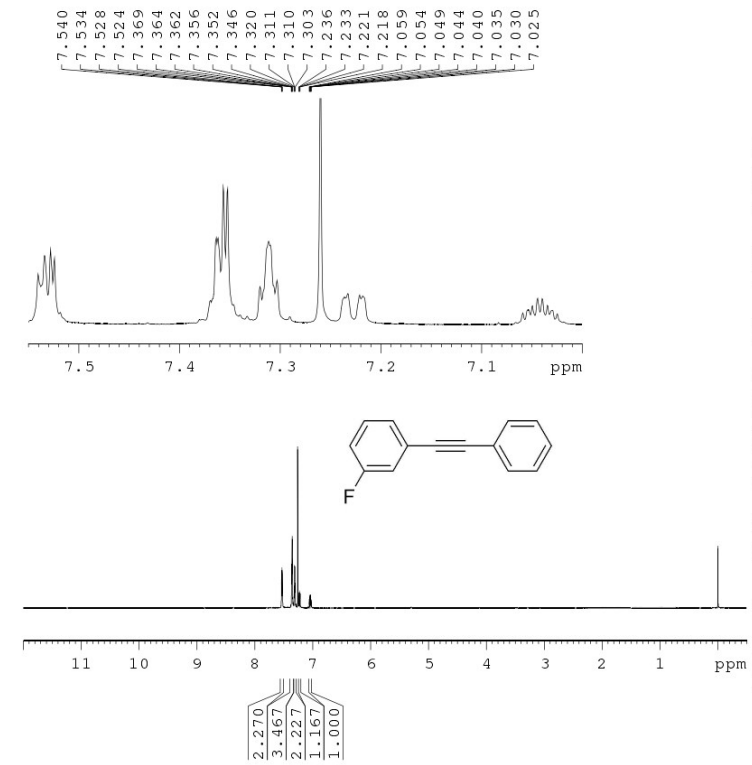


BRUKER

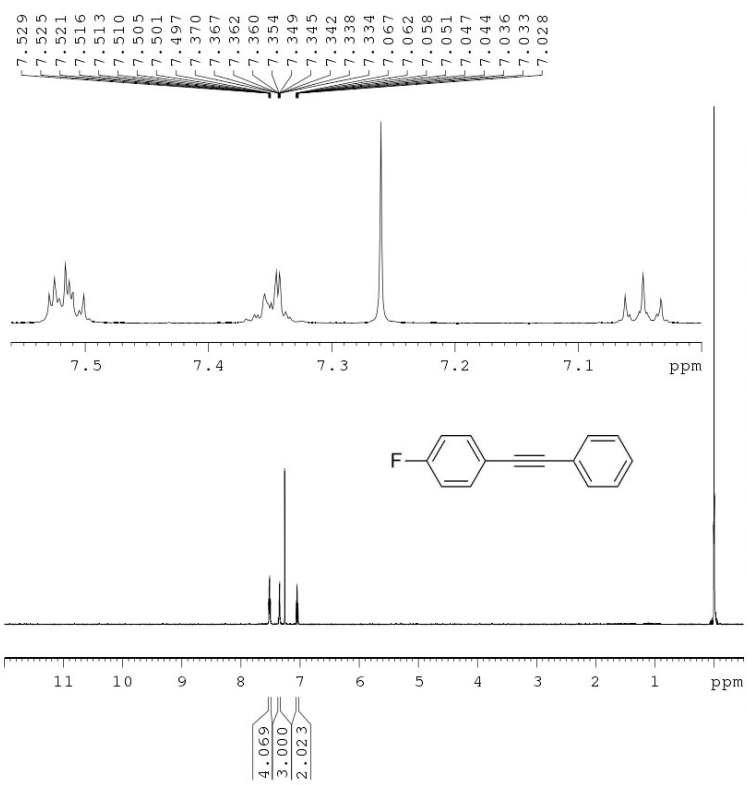
```
NAME            lizhipeng
EXPNO                  16
PROCNO                  1
Date_            20130619
Time                15.16
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             12335.526 Hz
FIDRES          0.188225 Hz
AQ              2.6564426 sec
RG                    203
DW                 40.533 usec
DE                  6.50 usec
TE                 302.4 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 12.60 usec
SI                    32768
SF           600.1300163 MHz
WDW                    EM
SSB                     0
LB                   0.10 Hz
GB                      0
PC                   1.00
```




```
NAME           lizhipeng
EXPNO                  7
PROCNO                 1
Date_           20140327
Time               10.31
INSTRUM            spect
PROBHD    5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                   203
DW              40.533 usec
DE               6.50 usec
TE               295.3 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
SI                 32768
SF          600.1300188 MHz
WDW                   EM
SSB                    0
LB                -0.10 Hz
GB                     0
PC                  1.00
```

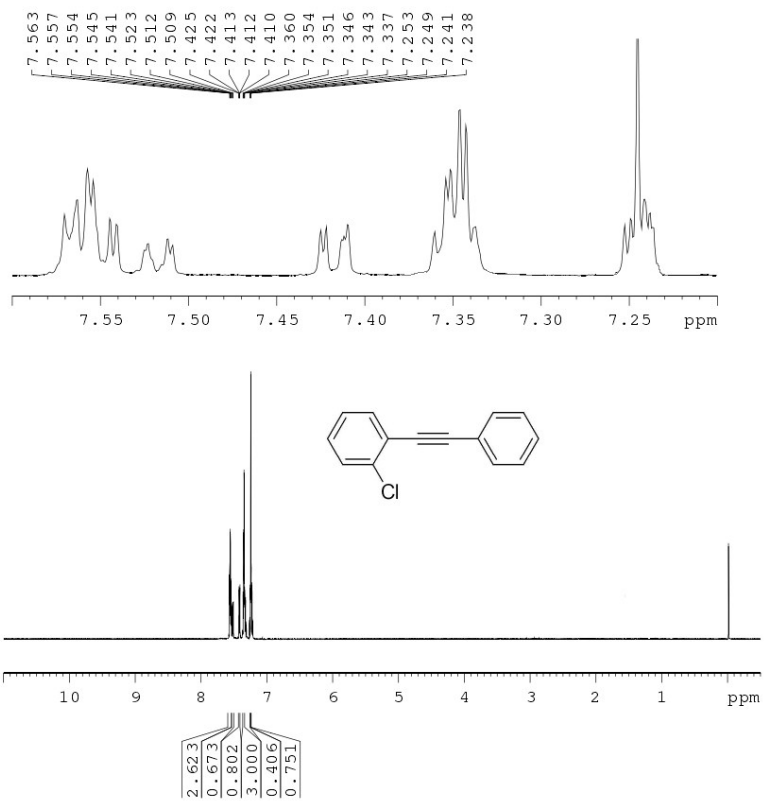



```
NAME           lizhipeng
EXPNO                  8
PROCNO                 1
Date_           20130425
Time               15.31
INSTRUM            spect
PROBHD    5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                   203
DW              40.533 usec
DE               6.50 usec
TE               295.6 K
D1           1.00000000 sec

-------- CHANNEL f1 --------
NUC1                  1H
P1                14.00 usec
SI                 32768
SF          600.1300173 MHz
WDW                   EM
SSB                    0
LB                 0.10 Hz
GB                     0
PC                  1.00
```



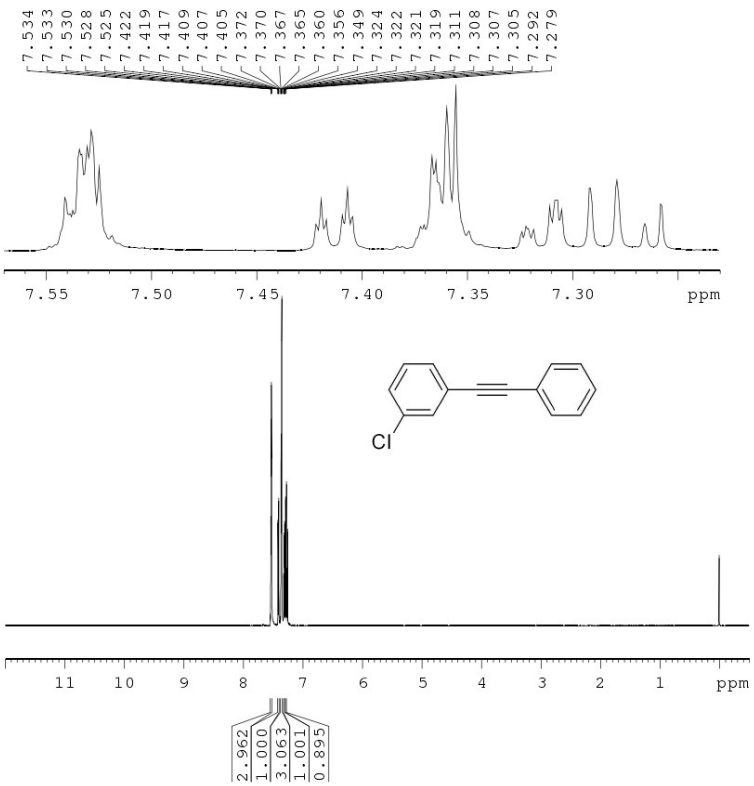
BRUKER

NAME	lizhipeng
EXPNO	9
PROCNO	1
Date_	20140303
Time	16.43
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	4
DS	2
SWH	12335.526 Hz
FIDRES	0.188225 Hz
AQ	2.6564426 sec
RG	203
DW	40.533 usec
DE	6.50 usec
TE	294.2 K
D1	1.00000000 sec
-------- CHANNEL f1 --------	
NUC1	1H
P1	14.00 usec
SI	32768
SF	600.1300176 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

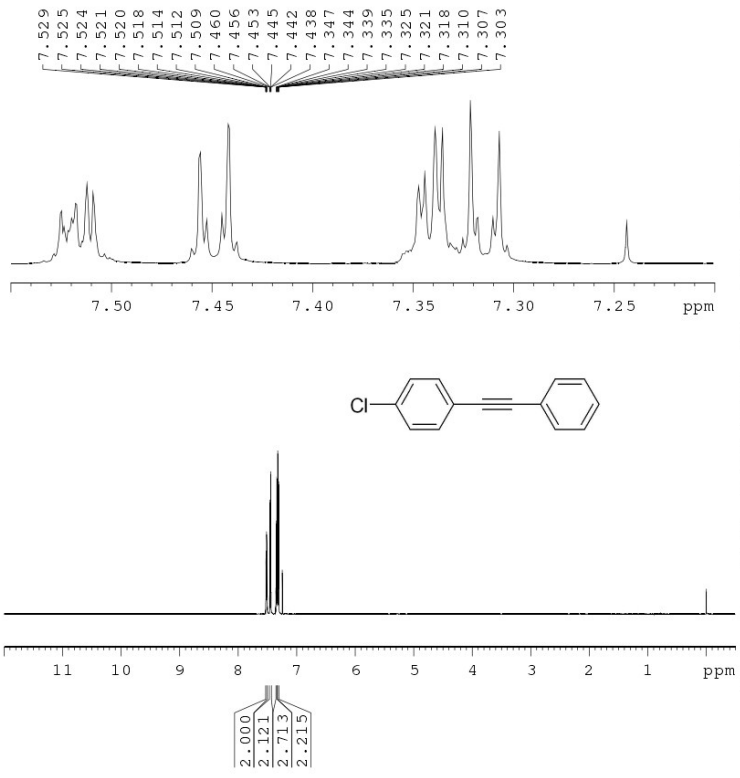


BRUKER

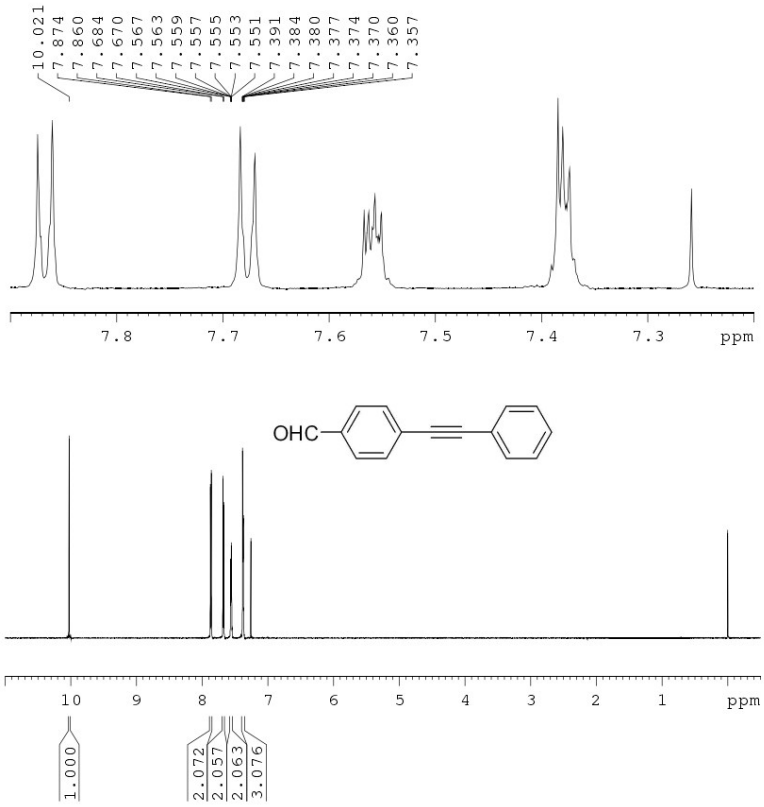
NAME	lizhipeng
EXPNO	11
PROCNO	1
Date_	20130425
Time	15.36
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12335.526 Hz
FIDRES	0.188225 Hz
AQ	2.6564426 sec
RG	203
DW	40.533 usec
DE	6.50 usec
TE	295.6 K
D1	1.00000000 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
SI	32768
SF	600.1300255 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00



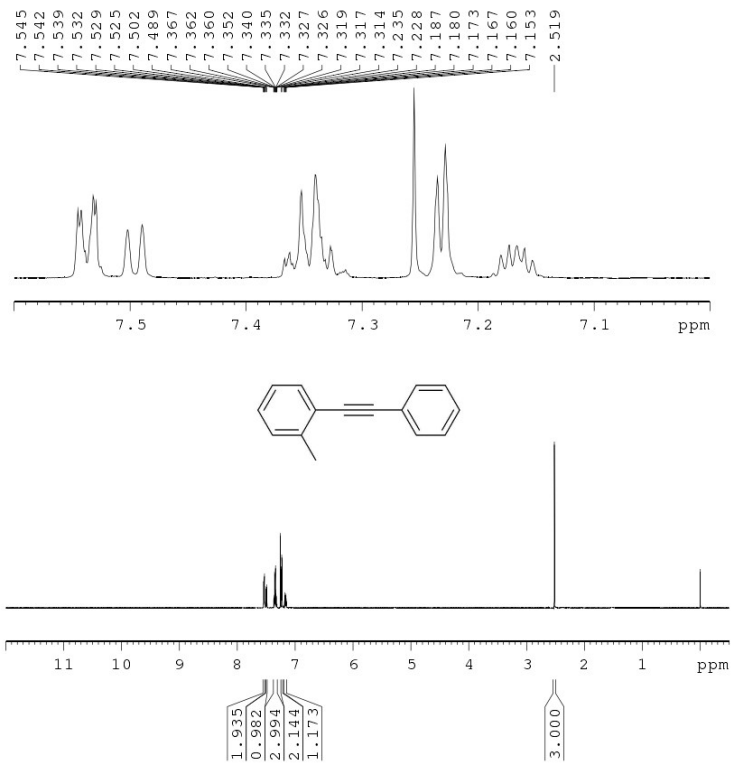

```
NAME            lizhipeng
EXPNO                  12
PROCNO                  1
Date_            20130425
Time                15.46
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                    203
DW                40.533 usec
DE                 6.50 usec
TE                295.6 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
SI                 32768
SF          600.1300179 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```



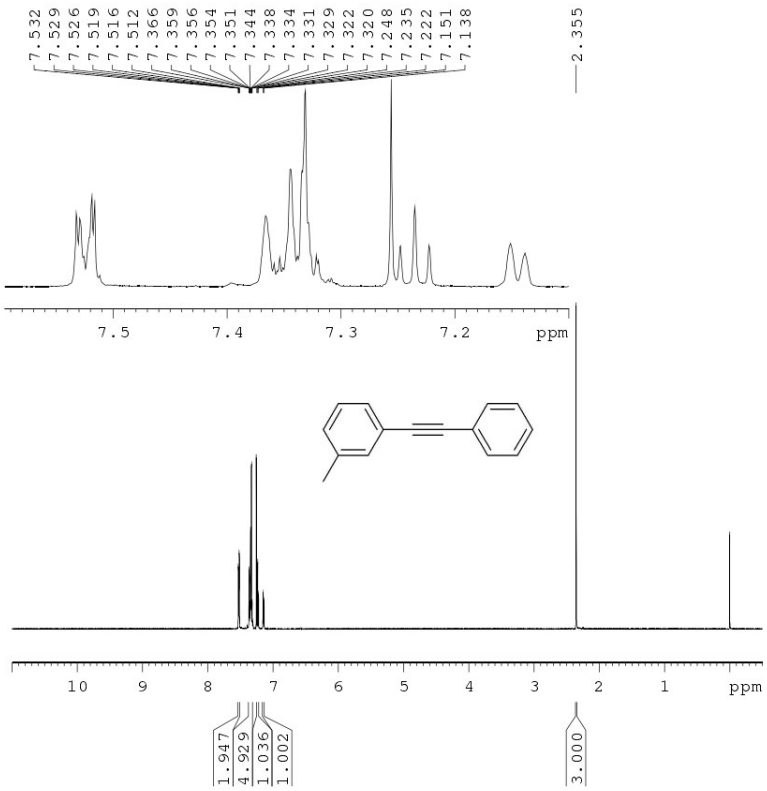

```
NAME            lizhipeng
EXPNO                  13
PROCNO                  1
Date_            20130909
Time                10.23
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                    203
DW                40.533 usec
DE                 6.50 usec
TE                299.3 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
SI                 32768
SF          600.1300274 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```




```
NAME           lizhipeng
EXPNO                 19
PROCNO                 1
Date_           20140318
Time               16.52
INSTRUM            spect
PROBHD    5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     2
DS                     2
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                   203
DW              40.533 usec
DE               6.50 usec
TE               296.9 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
SI                 32768
SF          600.1300183 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```

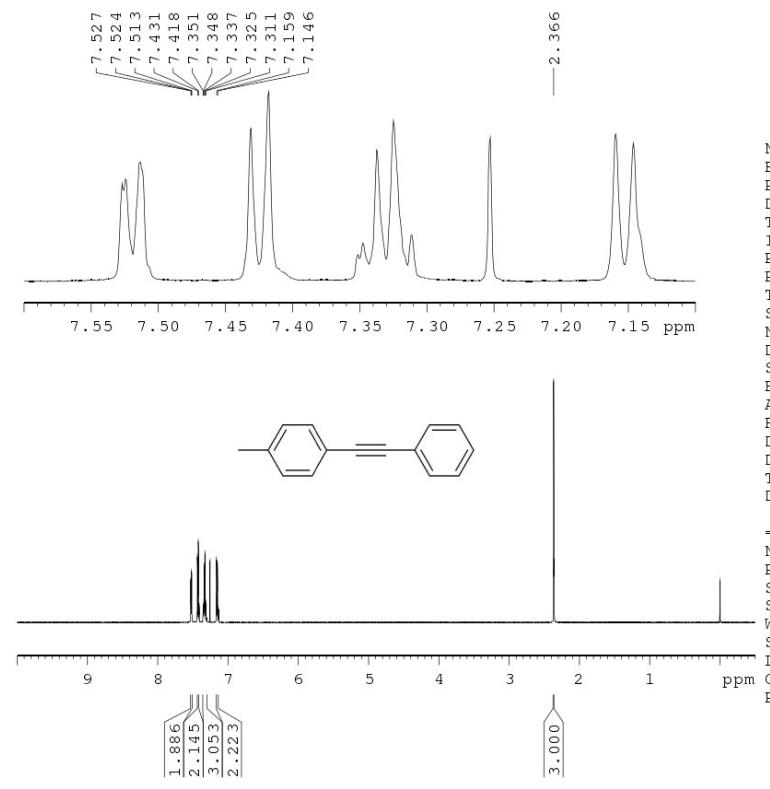



```
NAME           lizhipeng
EXPNO                  4
PROCNO                 1
Date_           20130918
Time               10.17
INSTRUM            spect
PROBHD    5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     7
DS                     2
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                   203
DW              40.533 usec
DE               6.50 usec
TE               300.3 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
SI                 32768
SF          600.1300206 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```



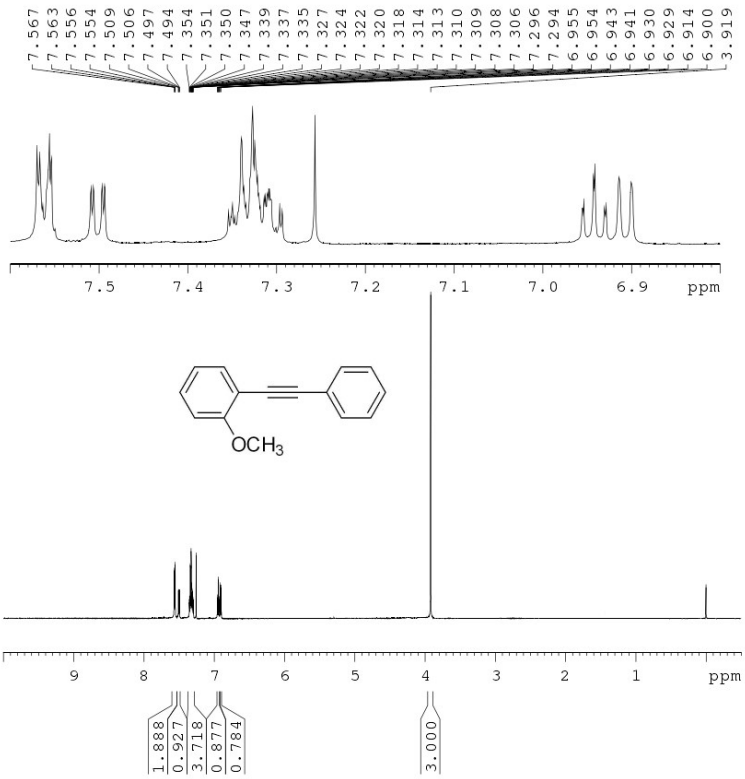
BRUKER

```
NAME            lizhipeng
EXPNO                   5
PROCNO                  1
Date_            20130918
Time                10.21
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       2
SWH             12335.526 Hz
FIDRES           0.188225 Hz
AQ              2.6564426 sec
RG                     203
DW                40.533 usec
DE                 6.50 usec
TE                 300.2 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
SI                 32768
SF           600.1300202 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```



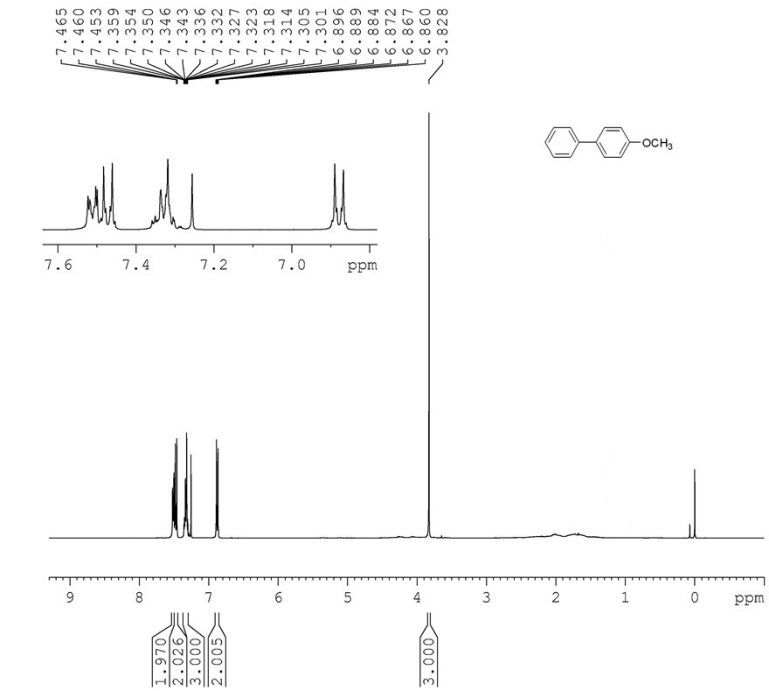
BRUKER

```
NAME            lizhipeng
EXPNO                   6
PROCNO                  1
Date_            20130918
Time                10.25
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       2
SWH             12335.526 Hz
FIDRES           0.188225 Hz
AQ              2.6564426 sec
RG                     203
DW                40.533 usec
DE                 6.50 usec
TE                 300.1 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
SI                 32768
SF           600.1300216 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```



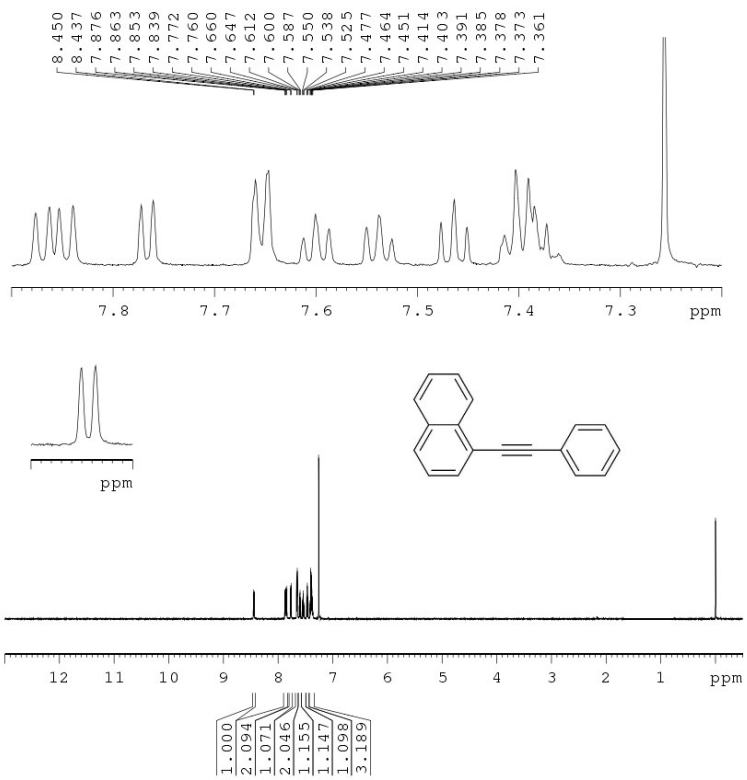
BRUKER

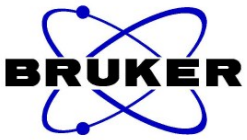
```
NAME            lizhipeng
EXPNO                   2
PROCNO                  1
Date_            20140327
Time                10.27
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             12335.526 Hz
FIDRES          0.188225 Hz
AQ              2.6564426 sec
RG                    181
DW                40.533 usec
DE                 6.50 usec
TE                295.3 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.00 usec
SI                  32768
SF          600.1300196 MHz
WDW                    EM
SSB                     0
LB                  -0.10 Hz
GB                      0
PC                   1.00
```



BRUKER

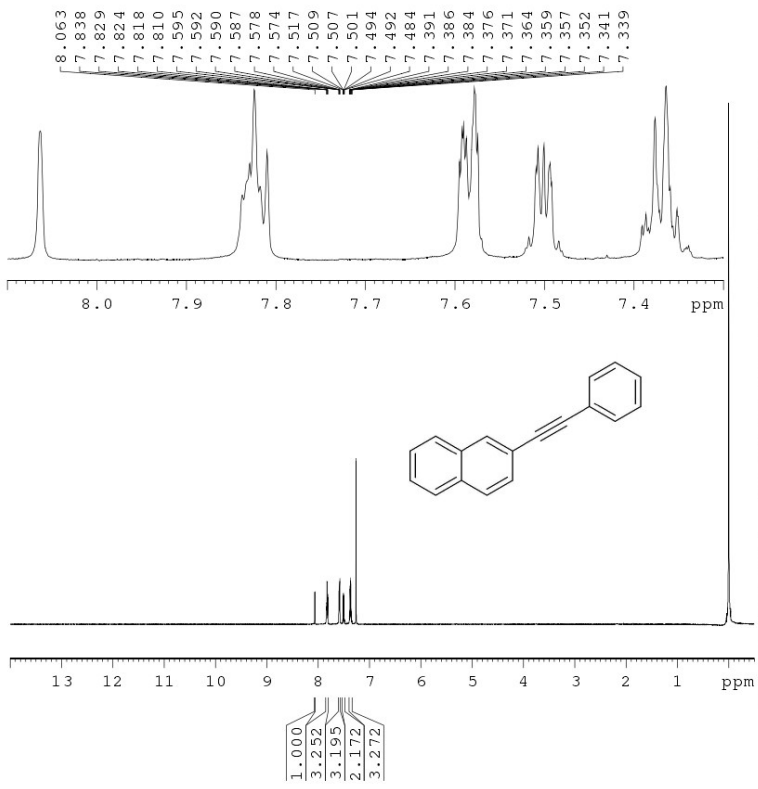
```
NAME      2016-04-01 tyut-chh
EXPNO                  10
PROCNO                  1
Date_            20160401
Time                13.13
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894966 sec
RG                  67.58
DW                62.400 usec
DE                 6.50 usec
TE                296.6 K
D1           1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                   1H
P1                  9.70 usec
SI                  65536
SF          400.1300115 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

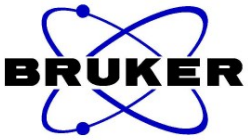




NAME lizhipeng
EXPNO 17
PROCNO 1
Date_ 20130425
Time 15.26
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 203
DW 40.533 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec

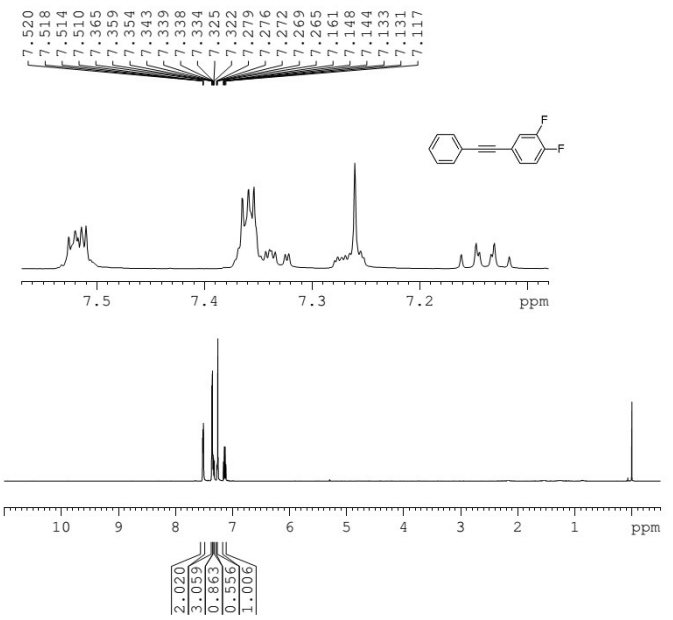
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
SI 32768
SF 600.1300186 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



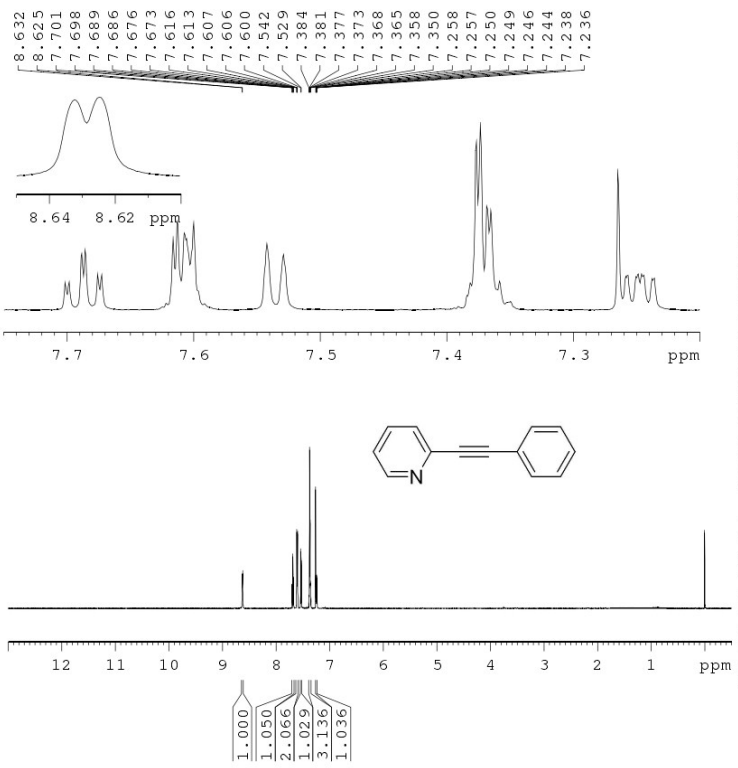


NAME lizhipeng
EXPNO 20
PROCNO 1
Date_ 20140110
Time 14.42
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 203
DW 40.533 usec
DE 6.50 usec
TE 292.4 K
D1 1.00000000 sec

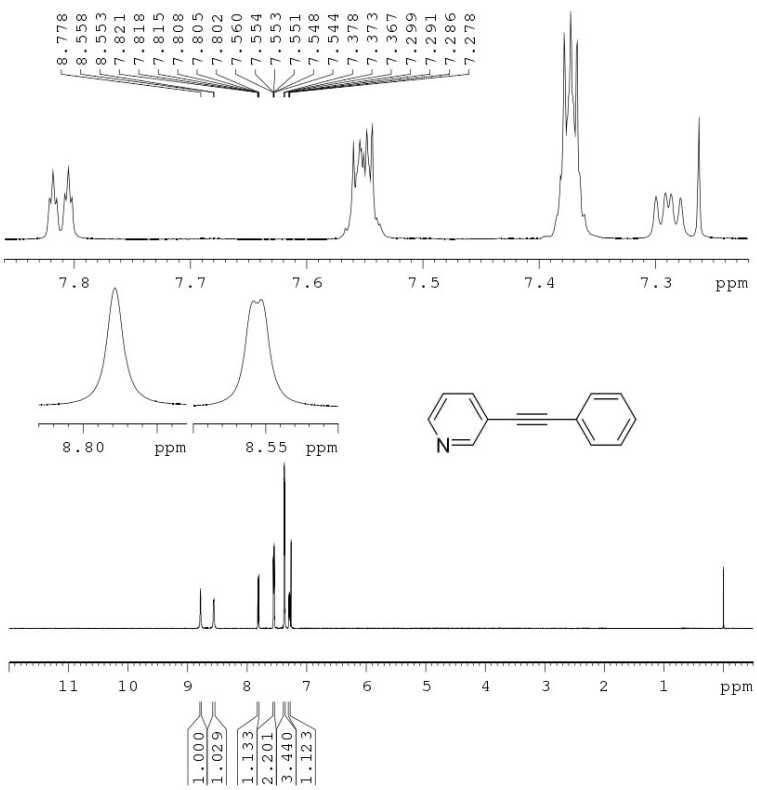
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
SI 32768
SF 600.1300183 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



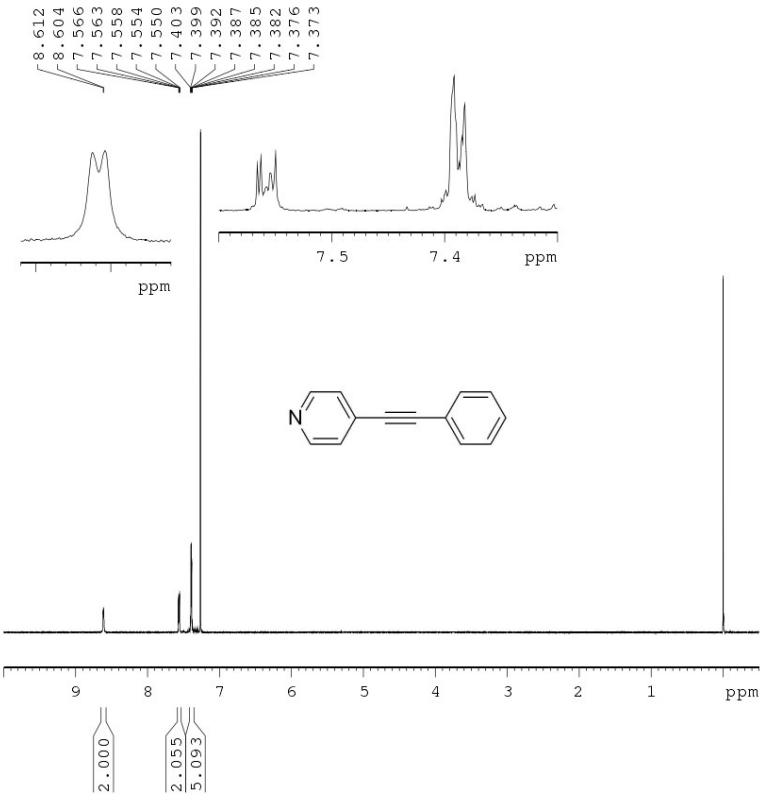

```
NAME            20130425
EXPNO                  5
PROCNO                 1
Date_           20130425
Time               15.21
INSTRUM            spect
PROBHD    5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    17
DS                     2
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                   203
DW              40.533 usec
DE               6.50 usec
TE                295.6 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
SI                 32768
SF          600.1300163 MHz
WDW                   EM
SSB                    0
LB                0.10 Hz
GB                     0
PC                  1.00
```



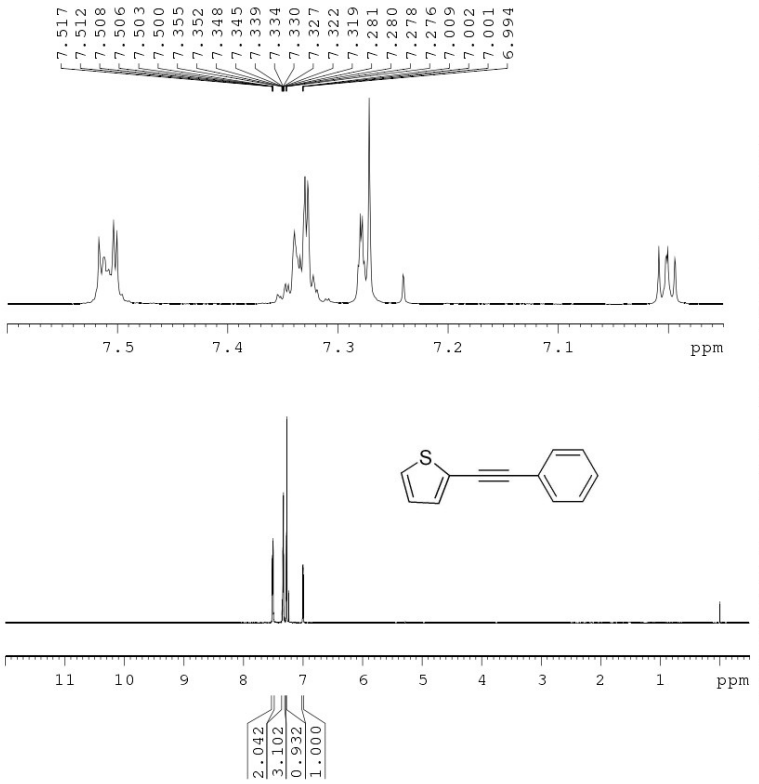

```
NAME          lizhipeng
EXPNO                 21
PROCNO                 1
Date_           20131016
Time               9.49
INSTRUM            spect
PROBHD    5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     2
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                   203
DW              40.533 usec
DE               6.50 usec
TE                294.8 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
SI                 32768
SF          600.1300151 MHz
WDW                   EM
SSB                    0
LB                0.10 Hz
GB                     0
PC                  1.00
```



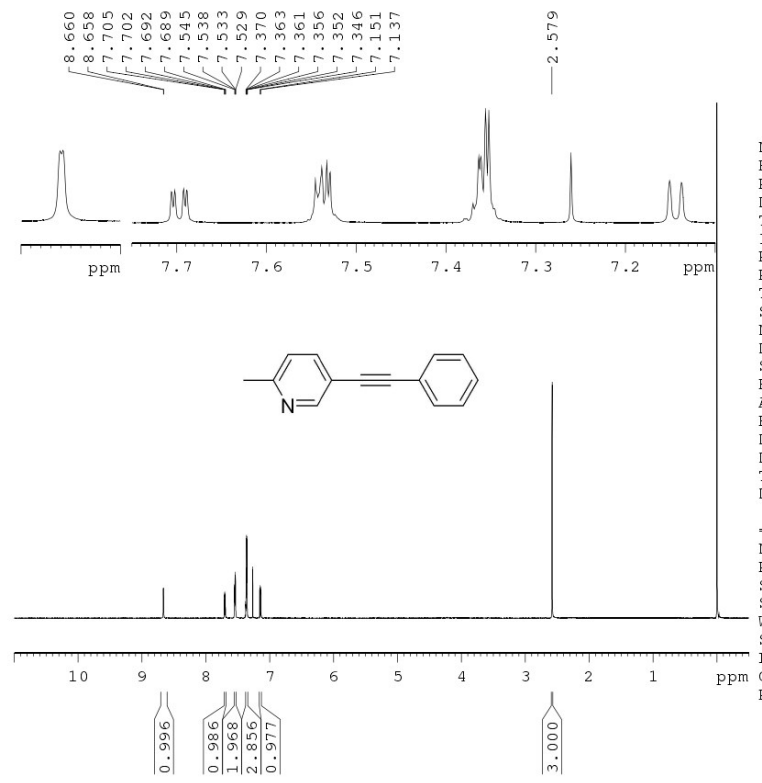
```
NAME            lizhipeng
EXPNO                  18
PROCNO                  1
Date_           20131016
Time                9.53
INSTRUM            spect
PROBHD    5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     9
DS                     2
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                   203
DW              40.533 usec
DE               6.50 usec
TE               294.7 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
SI                 32768
SF           600.1300162 MHz
WDW                   EM
SSB                    0
LB                0.10 Hz
GB                     0
PC                  1.00
```

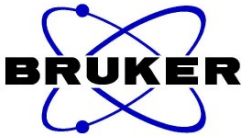


```
NAME            lizhipeng
EXPNO                  22
PROCNO                  1
Date_           20131016
Time                9.57
INSTRUM            spect
PROBHD    5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                   203
DW              40.533 usec
DE               6.50 usec
TE               294.8 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
SI                 32768
SF           600.1300167 MHz
WDW                   EM
SSB                    0
LB                0.10 Hz
GB                     0
PC                  1.00
```

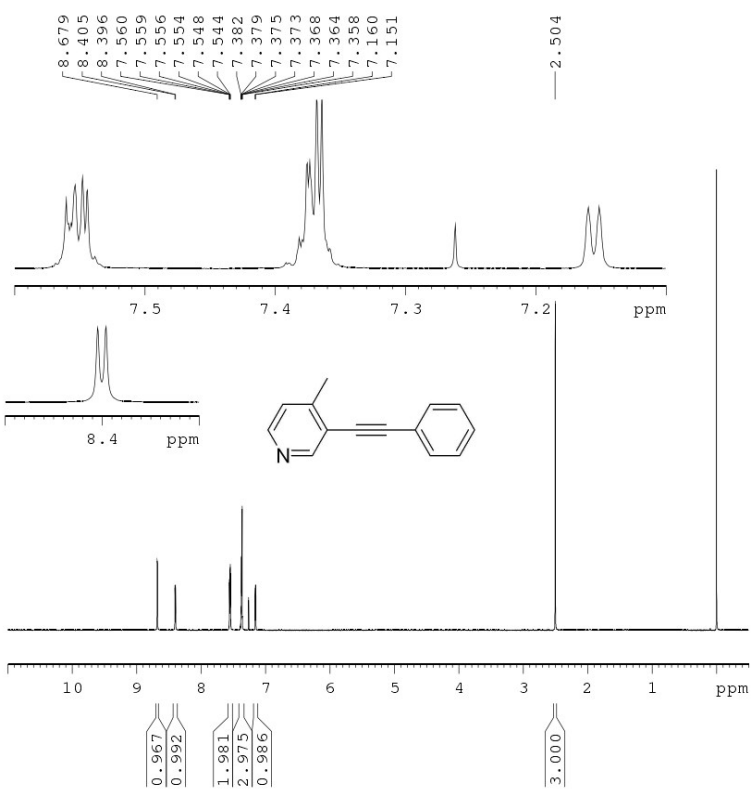


```
NAME            lizhipeng
EXPNO                  23
PROCNO                  1
Date_           20130918
Time               10.29
INSTRUM            spect
PROBHD    5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                   203
DW               40.533 usec
DE                6.50 usec
TE                300.1 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
SI                 32768
SF          600.1300284 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```

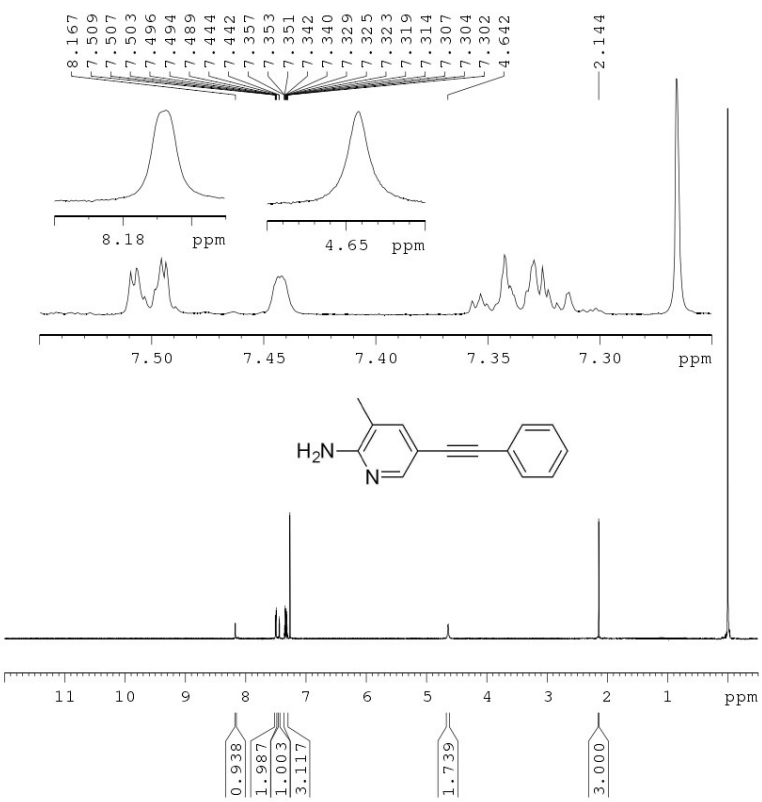




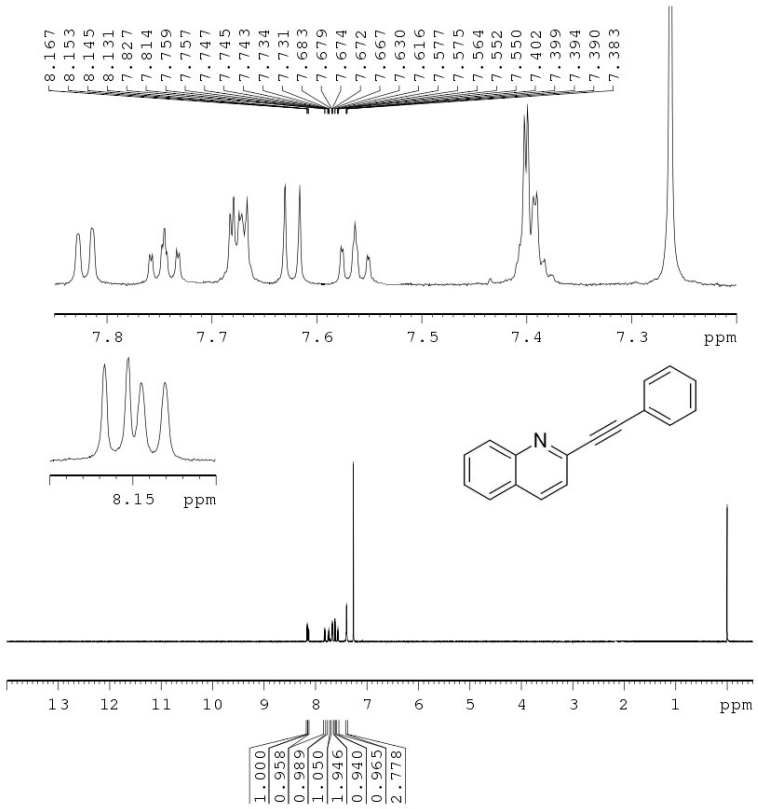
```
NAME            lizhipeng
EXPNO                  24
PROCNO                  1
Date_           20140311
Time               10.39
INSTRUM            spect
PROBHD    5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                   203
DW               40.533 usec
DE                6.50 usec
TE                295.0 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
SI                 32768
SF          600.1300173 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```



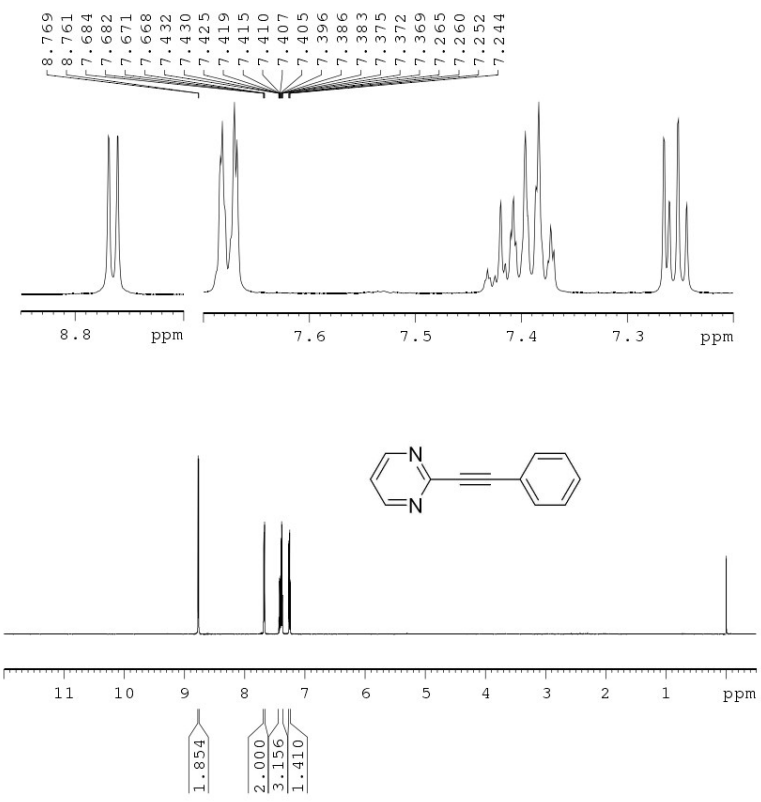

```
NAME            lizhipeng
EXPNO                  26
PROCNO                  1
Date_            20140311
Time                10.48
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      2
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                    101
DW                40.533 usec
DE                 6.50 usec
TE                295.0 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
SI                  32768
SF          600.1300165 MHz
WDW                    EM
SSB                     0
LB                 0.10 Hz
GB                      0
PC                   1.00
```



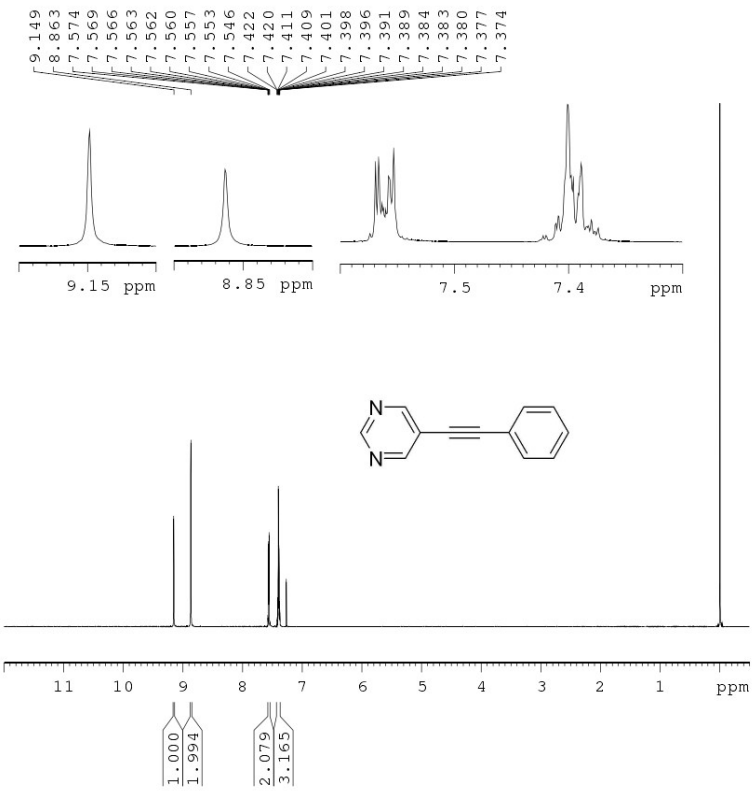

```
NAME            lizhipeng
EXPNO                  25
PROCNO                  1
Date_            20140115
Time                14.45
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                    203
DW                40.533 usec
DE                 6.50 usec
TE                292.0 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
SI                  32768
SF          600.1300144 MHz
WDW                    EM
SSB                     0
LB                -0.10 Hz
GB                      0
PC                   1.00
```



NAME	lizhipeng
EXPNO	29
PROCNO	1
Date_	20140313
Time	14.57
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	12335.526 Hz
FIDRES	0.188225 Hz
AQ	2.6564426 sec
RG	203
DW	40.533 usec
DE	6.50 usec
TE	295.2 K
D1	1.00000000 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
SI	32768
SF	600.1300157 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

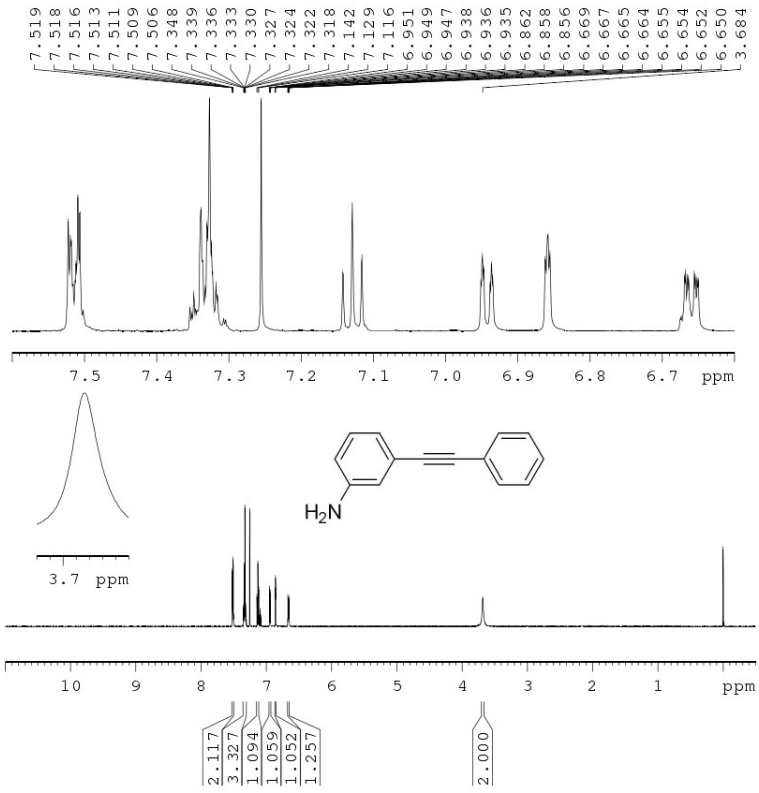


NAME	lizhipeng
EXPNO	27
PROCNO	1
Date_	20140313
Time	14.52
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	12335.526 Hz
FIDRES	0.188225 Hz
AQ	2.6564426 sec
RG	203
DW	40.533 usec
DE	6.50 usec
TE	295.2 K
D1	1.00000000 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
SI	32768
SF	600.1300143 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00



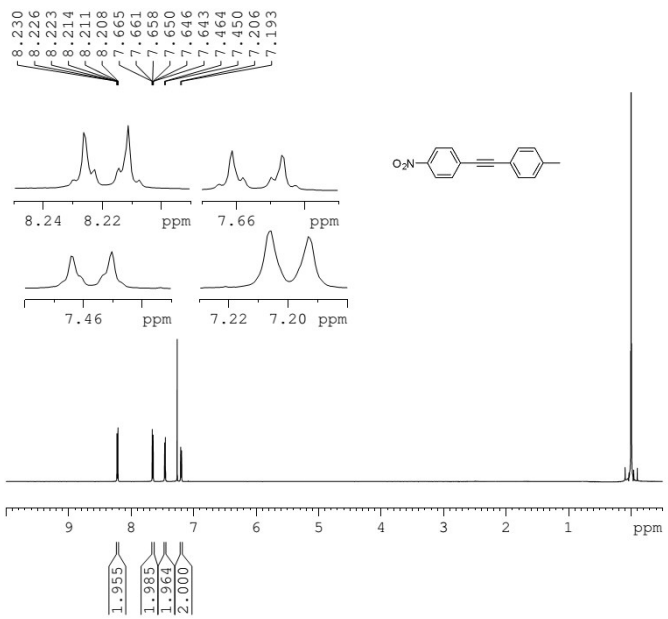
BRUKER

```
NAME            lizhipeng
EXPNO                  28
PROCNO                  1
Date_            20140115
Time                14.51
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH             12335.526 Hz
FIDRES           0.188225 Hz
AQ              2.6564426 sec
RG                     114
DW                40.533 usec
DE                 6.50 usec
TE                292.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
SI                    32768
SF           600.1300144 MHz
WDW                    EM
SSB                     0
LB                  -0.10 Hz
GB                      0
PC                   1.00
```

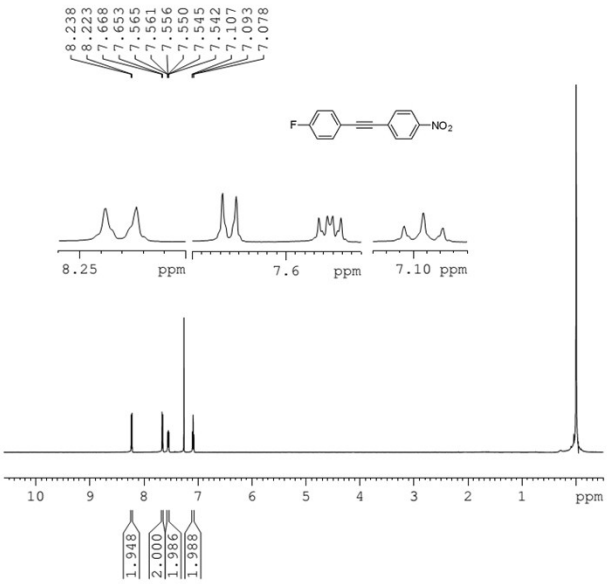


BRUKER

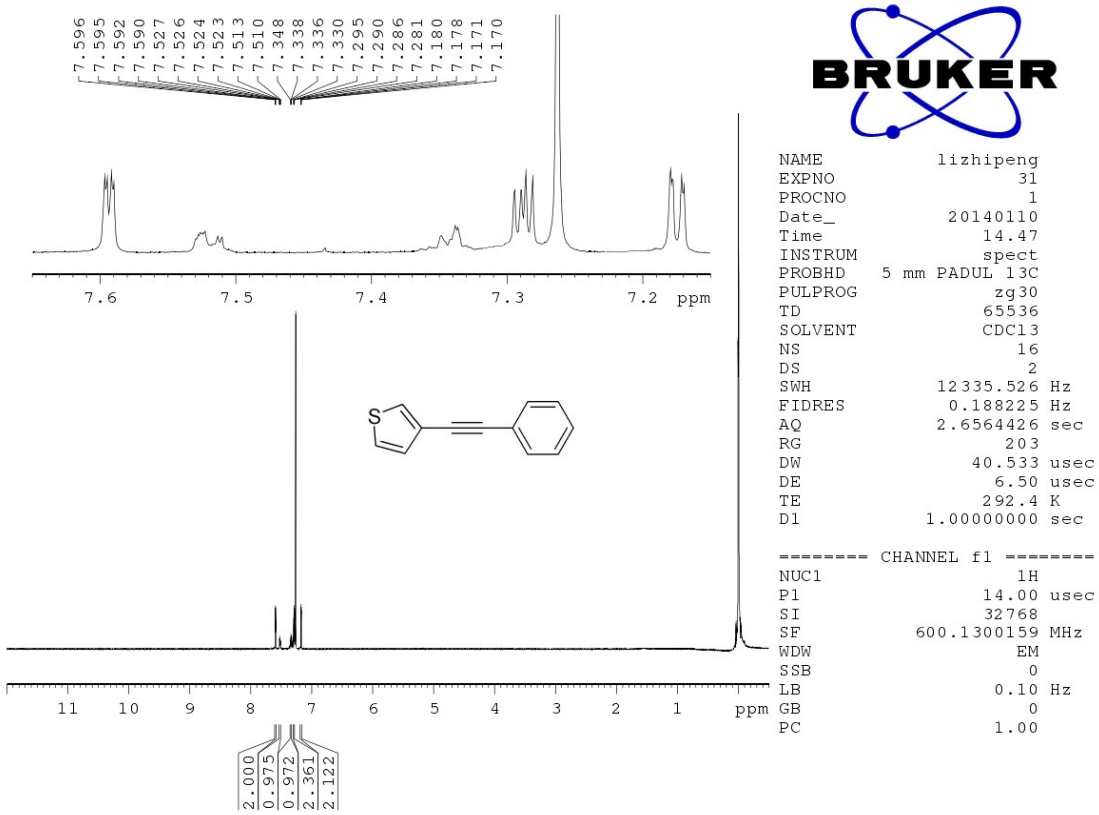
```
NAME            lizhipeng
EXPNO                  30
PROCNO                  1
Date_            20131016
Time                10.05
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       2
SWH             12335.526 Hz
FIDRES           0.188225 Hz
AQ              2.6564426 sec
RG                     203
DW                40.533 usec
DE                 6.50 usec
TE                294.8 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
SI                    32768
SF           600.1300206 MHz
WDW                    EM
SSB                     0
LB                   0.10 Hz
GB                      0
PC                   1.00
```



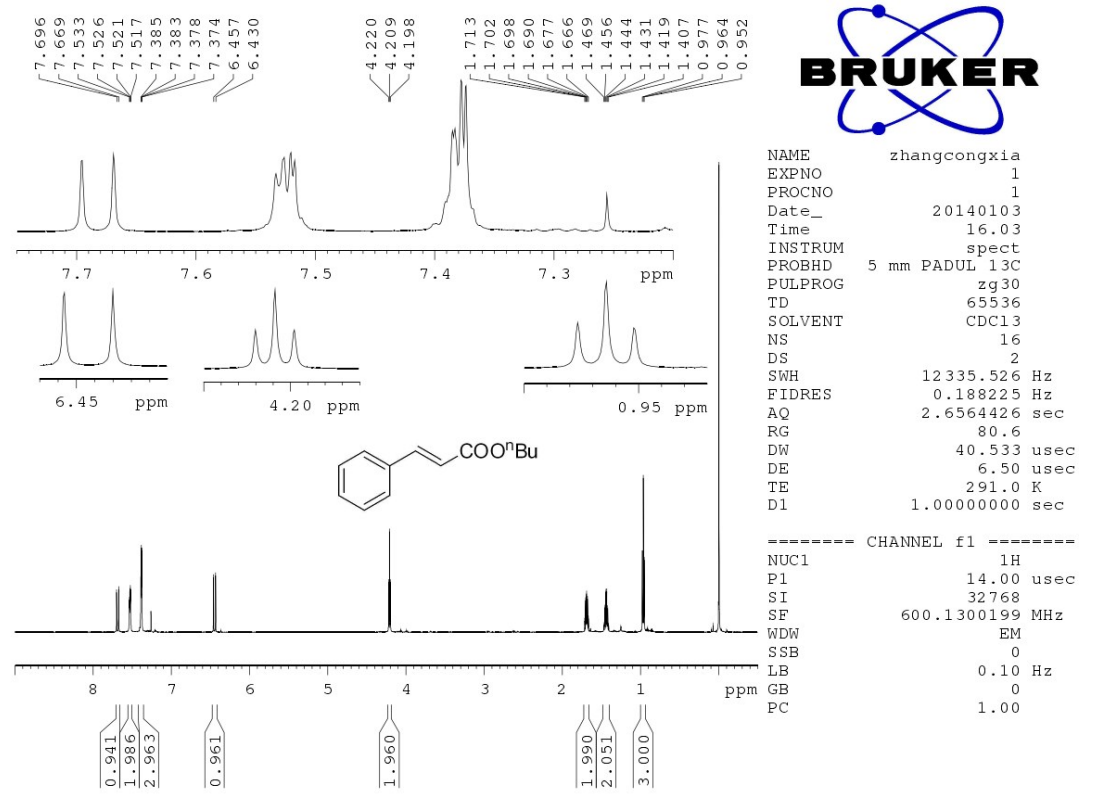

```
NAME            20140110
EXPNO                  3
PROCNO                 1
Date_           20140110
Time               14.53
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                   203
DW                40.533 usec
DE                 6.50 usec
TE                292.4 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
SI                32768
SF          600.1300159 MHz
WDW                   EM
SSB                    0
LB                0.10 Hz
GB                     0
PC                1.00
```

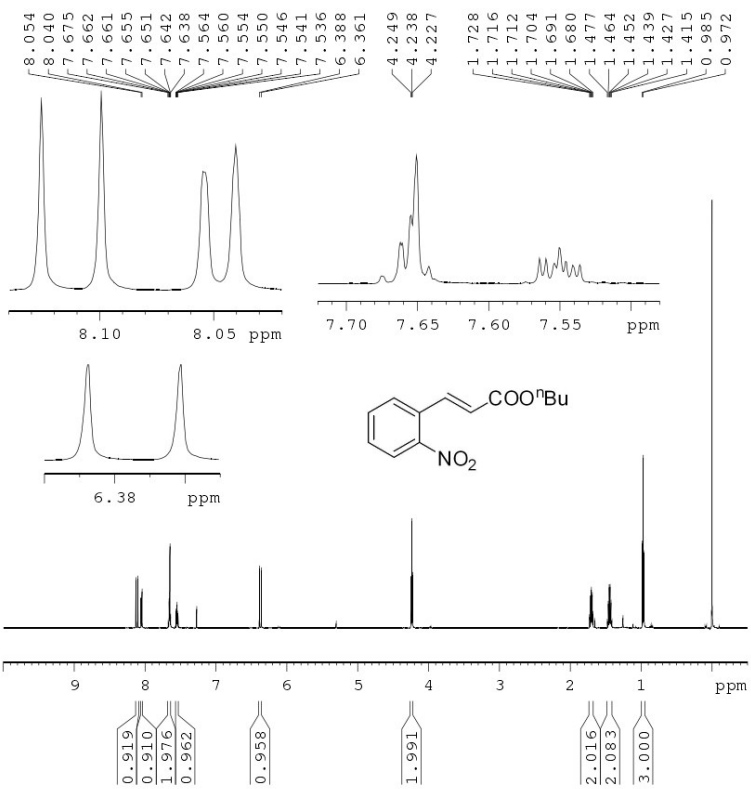



```
NAME            20140110
EXPNO                  4
PROCNO                 1
Date_           20140110
Time               14.59
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                   203
DW                40.533 usec
DE                 6.50 usec
TE                292.4 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
SI                32768
SF          600.1300159 MHz
WDW                   EM
SSB                    0
LB                0.10 Hz
GB                     0
PC                1.00
```



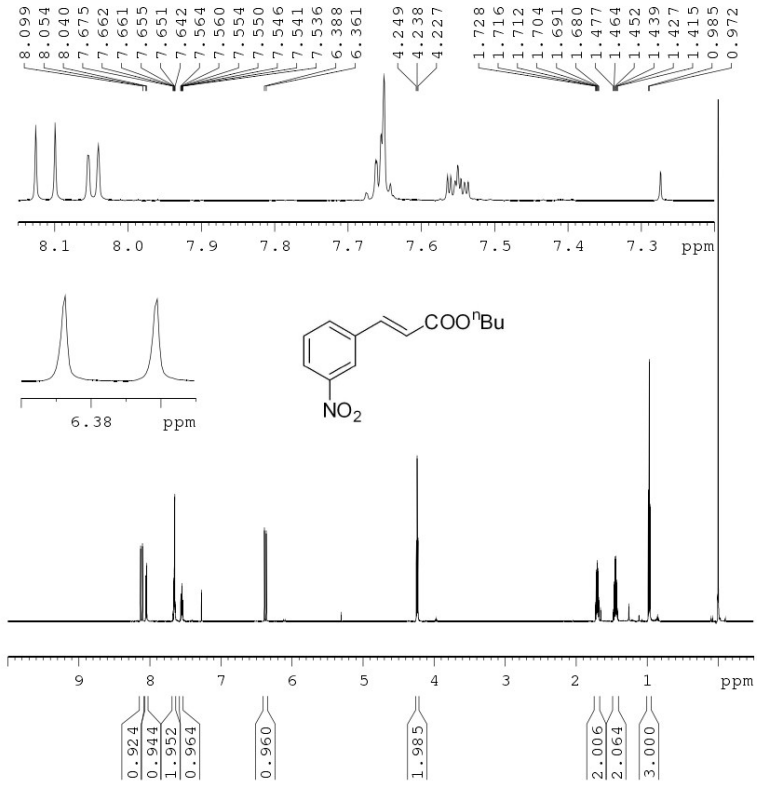
10.2 ^{1}H-NMR spectra for the Heck coupling products





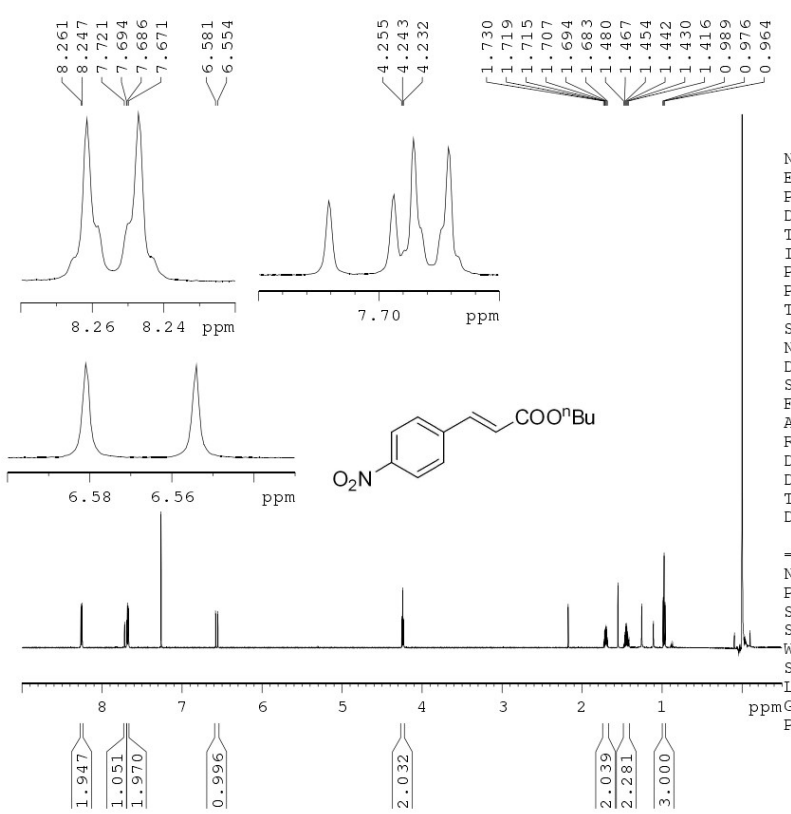
NAME zhangcongxia
EXPNO 7
PROCNO 1
Date_ 20140311
Time 9.36
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 90.5
DW 40.533 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
SI 32768
SF 600.1300093 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

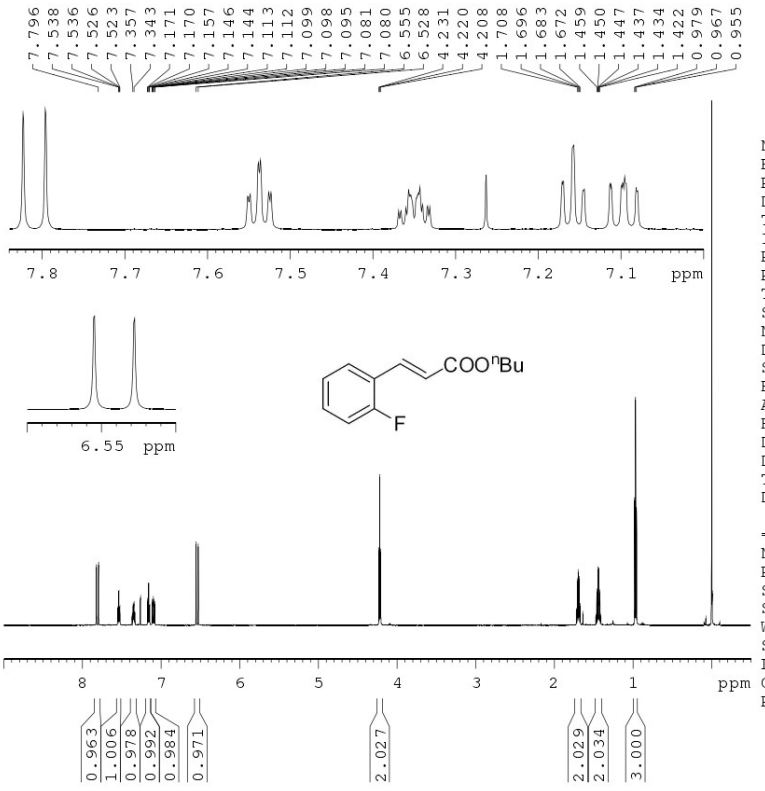


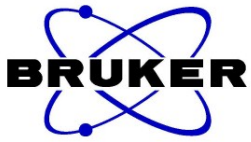
NAME zhangcongxia
EXPNO 8
PROCNO 1
Date_ 20140311
Time 9.36
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 90.5
DW 40.533 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
SI 32768
SF 600.1300093 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

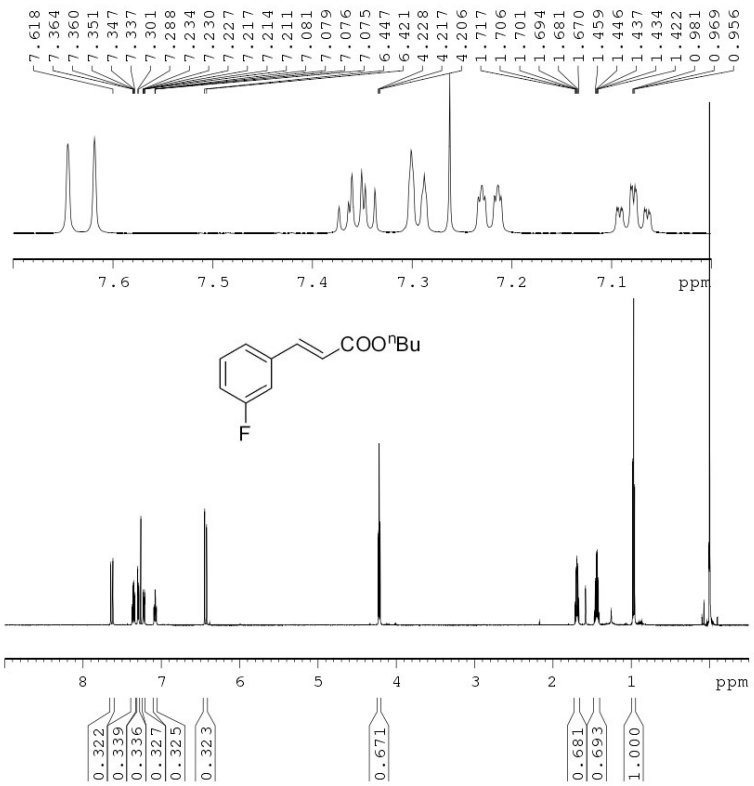


```
NAME          zhangcongxia
EXPNO                    9
PROCNO                   1
Date_             20140311
Time                 9.47
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG              zg30
TD                  65536
SOLVENT             CDC13
NS                      8
DS                      2
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                    203
DW                 40.533 usec
DE                  6.50 usec
TE                 295.0 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
SI                 32768
SF          600.1300155 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```



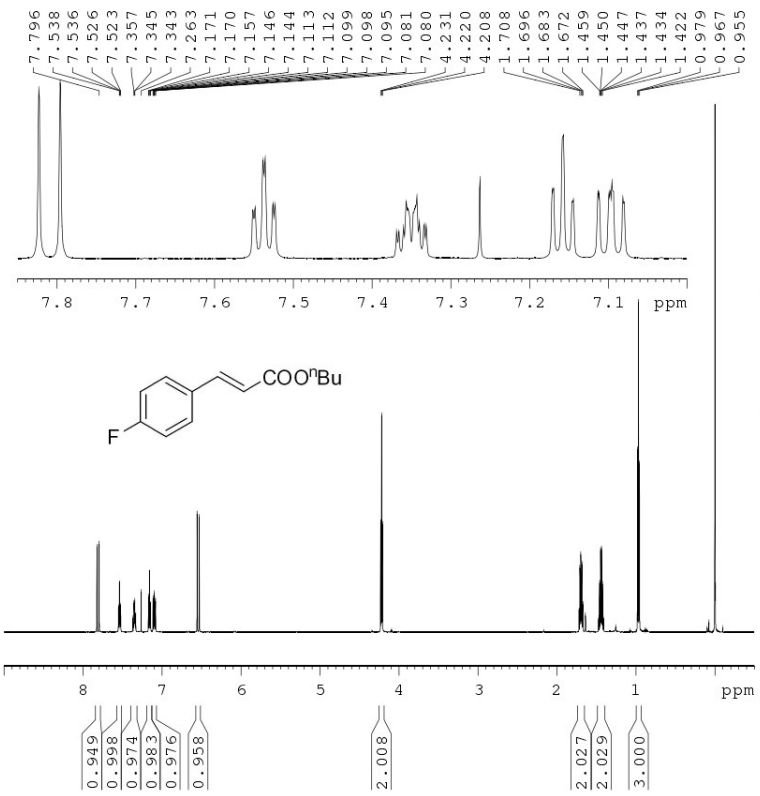


```
NAME          zhangcongxia
EXPNO                   10
PROCNO                   1
Date_             20140303
Time                15.13
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG              zg30
TD                  65536
SOLVENT             CDC13
NS                     16
DS                      2
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                   90.5
DW                 40.533 usec
DE                  6.50 usec
TE                 295.3 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
SI                 32768
SF          600.1300156 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```



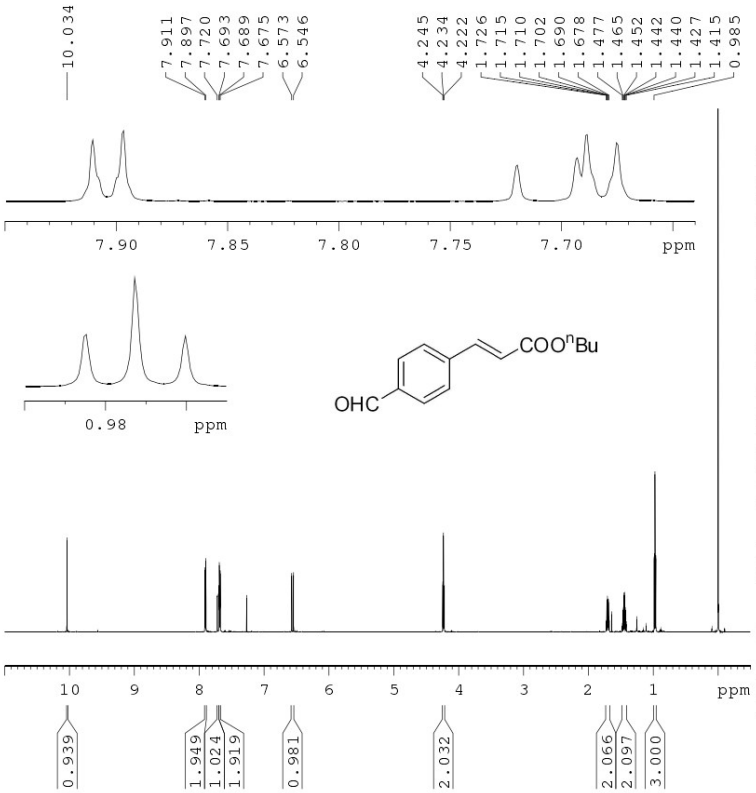
NAME zhangcongxia
EXPNO 11
PROCNO 1
Date_ 20140303
Time 15.17
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 144
DW 40.533 usec
DE 6.50 usec
TE 294.9 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
SI 32768
SF 600.1300163 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

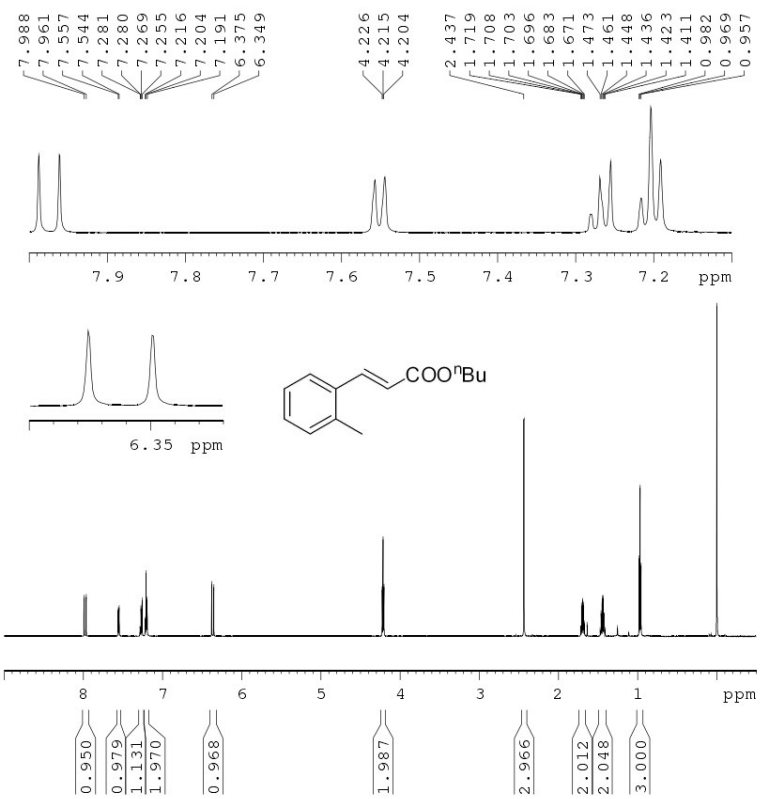


NAME zhangcongxia
EXPNO 12
PROCNO 1
Date_ 20140303
Time 15.13
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 90.5
DW 40.533 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec

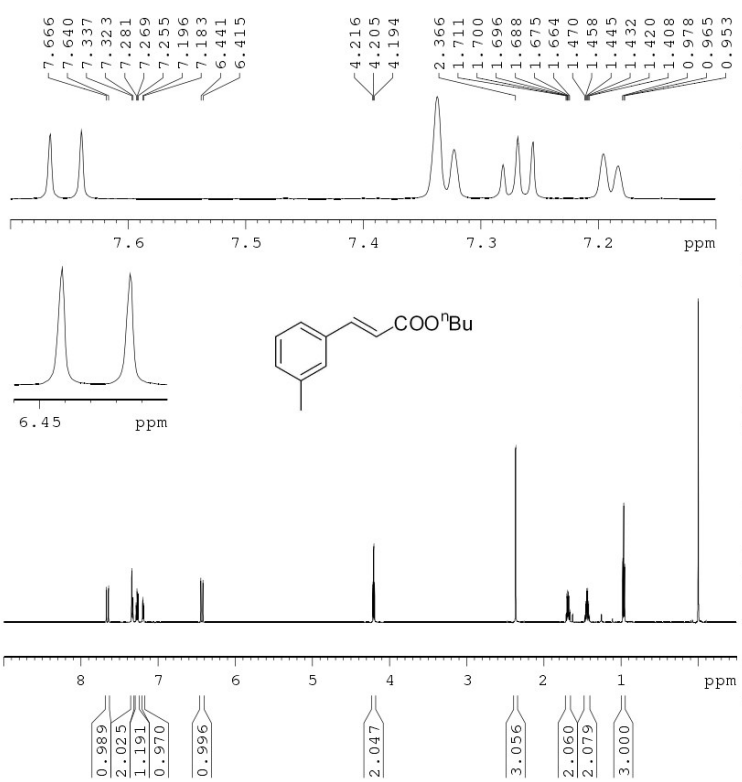
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
SI 32768
SF 600.1300156 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



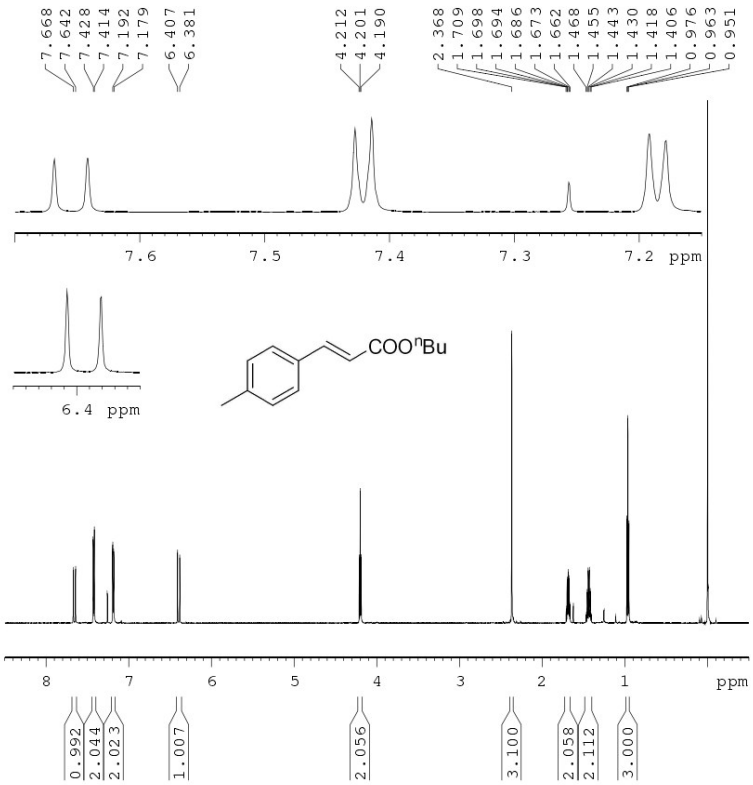
```
NAME          zhangcongxia
EXPNO                   13
PROCNO                   1
Date_             20140303
Time                 16.19
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       2
SWH             12335.526 Hz
FIDRES           0.188225 Hz
AQ              2.6564426 sec
RG                    90.5
DW                40.533 usec
DE                 6.50 usec
TE                 294.2 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.00 usec
SI                   32768
SF           600.1300127 MHz
WDW                     EM
SSB                      0
LB                  0.10 Hz
GB                       0
PC                    1.00
```



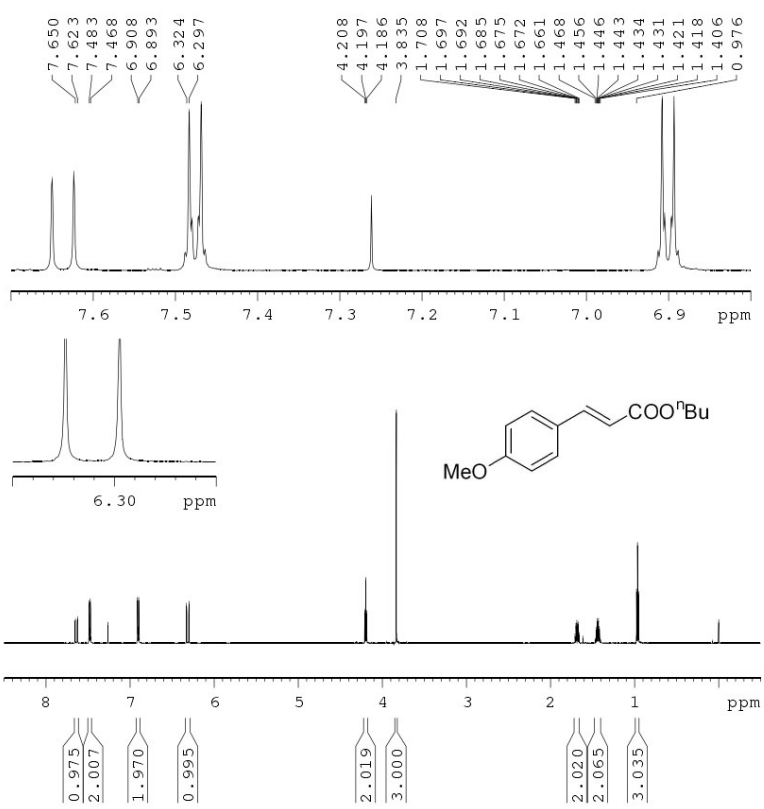
```
NAME          zhangcongxia
EXPNO                    2
PROCNO                   1
Date_             20140303
Time                 15.31
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       2
SWH             12335.526 Hz
FIDRES           0.188225 Hz
AQ              2.6564426 sec
RG                    90.5
DW                40.533 usec
DE                 6.50 usec
TE                 294.5 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.00 usec
SI                   32768
SF           600.1300206 MHz
WDW                     EM
SSB                      0
LB                  0.10 Hz
GB                       0
PC                    1.00
```



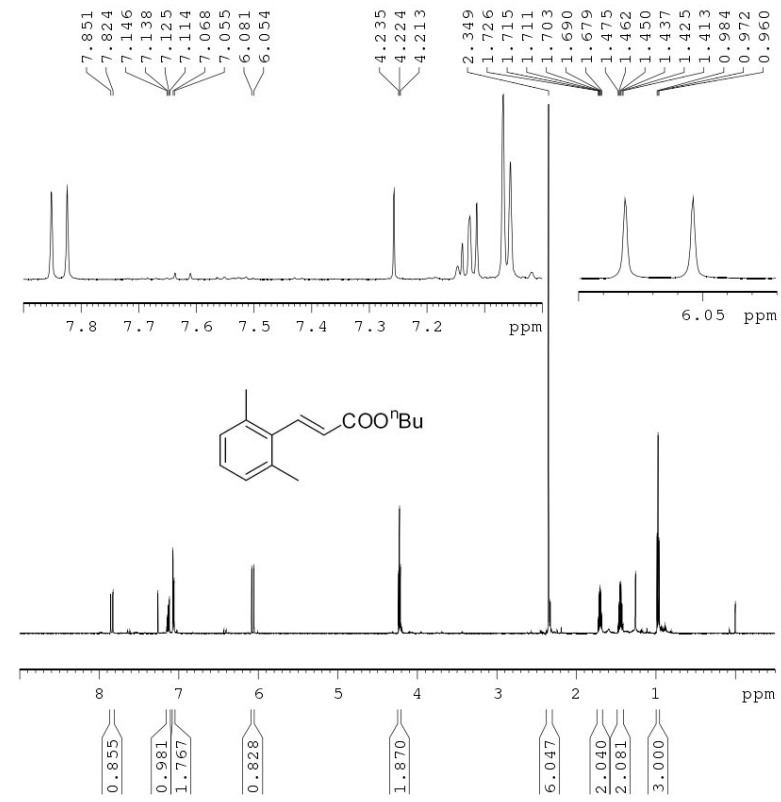

```
NAME          zhangcongxia
EXPNO                    3
PROCNO                   1
Date_             20140303
Time                 15.34
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG               zg30
TD                   65536
SOLVENT              CDC13
NS                       8
DS                       2
SWH             12335.526 Hz
FIDRES           0.188225 Hz
AQ              2.6564426 sec
RG                    90.5
DW                40.533 usec
DE                 6.50 usec
TE                 294.5 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.00 usec
SI                   32768
SF           600.1300201 MHz
WDW                     EM
SSB                      0
LB                  0.10 Hz
GB                       0
PC                    1.00
```

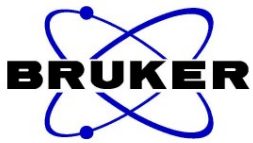



```
NAME          zhangcongxia
EXPNO                    4
PROCNO                   1
Date_             20140303
Time                 15.40
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG               zg30
TD                   65536
SOLVENT              CDC13
NS                       8
DS                       2
SWH             12335.526 Hz
FIDRES           0.188225 Hz
AQ              2.6564426 sec
RG                    90.5
DW                40.533 usec
DE                 6.50 usec
TE                 294.4 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.00 usec
SI                   32768
SF           600.1300199 MHz
WDW                     EM
SSB                      0
LB                  0.10 Hz
GB                       0
PC                    1.00
```

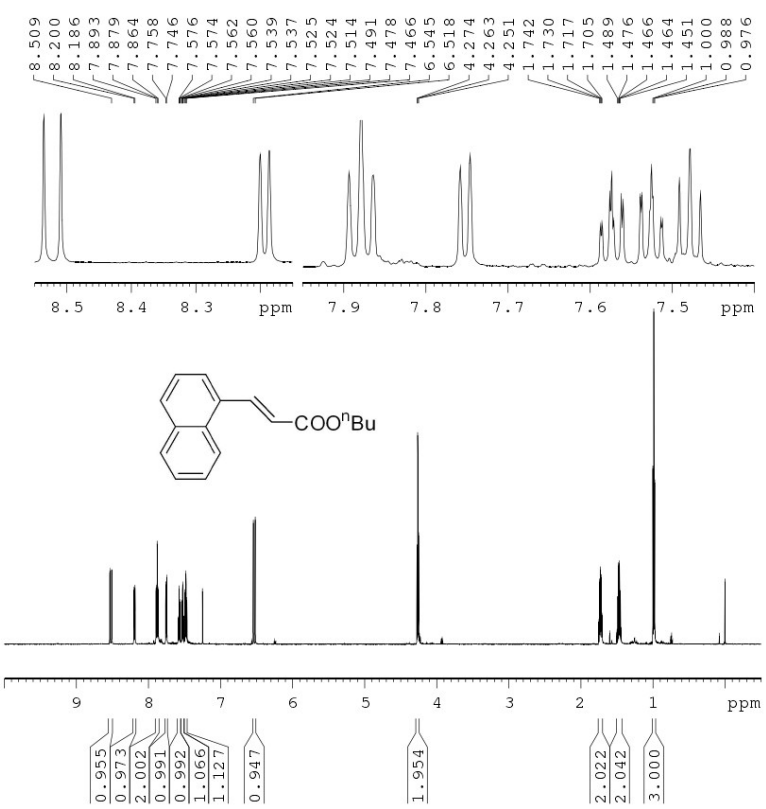


```
NAME          zhangcongxia
EXPNO                    5
PROCNO                   1
Date_             20140318
Time                 16.16
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       4
DS                       2
SWH              12335.526 Hz
FIDRES            0.188225 Hz
AQ                2.6564426 sec
RG                     128
DW                40.533 usec
DE                 6.50 usec
TE                 296.9 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.00 usec
SI                   32768
SF            600.1300168 MHz
WDW                     EM
SSB                      0
LB                  0.10 Hz
GB                       0
PC                    1.00
```



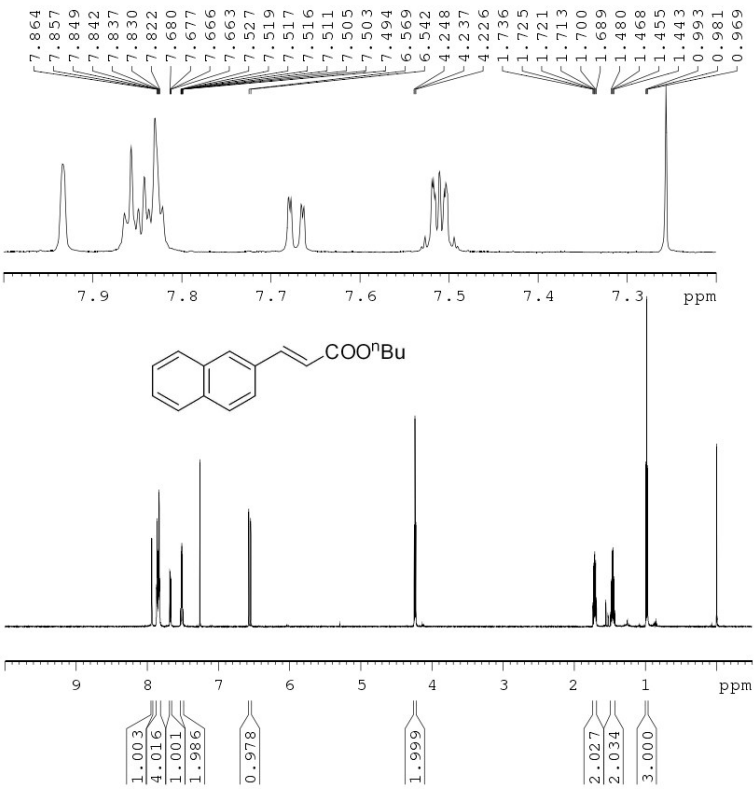


```
NAME          zhangcongxia
EXPNO                    6
PROCNO                   1
Date_             20140408
Time                 15.31
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       2
SWH              12335.526 Hz
FIDRES            0.188225 Hz
AQ                2.6564426 sec
RG                     101
DW                40.533 usec
DE                 6.50 usec
TE                 297.2 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.00 usec
SI                   32768
SF            600.1300191 MHz
WDW                     EM
SSB                      0
LB                 -0.10 Hz
GB                       0
PC                    1.00
```



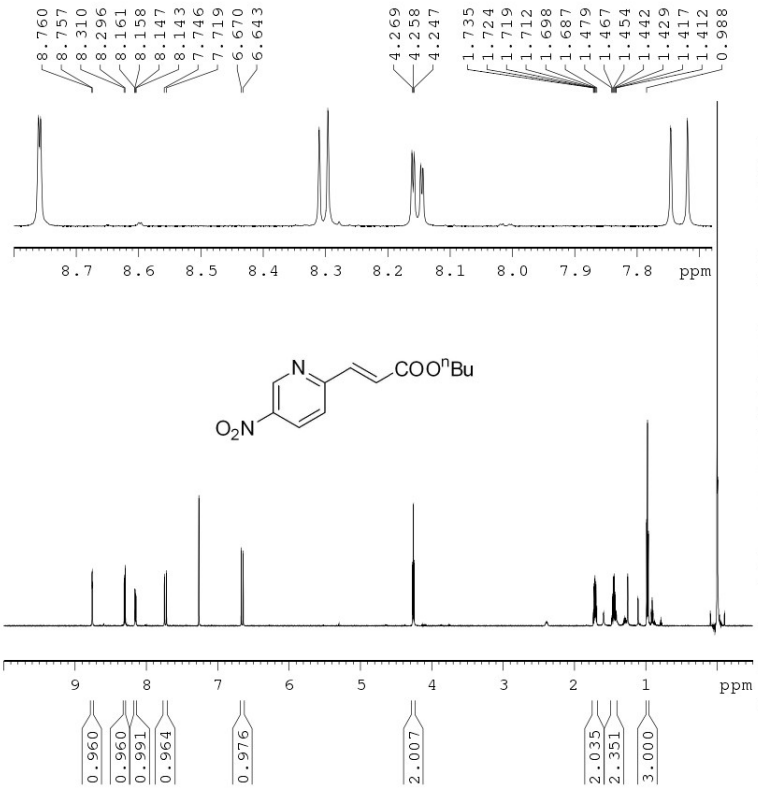
BRUKER

```
NAME          zhangcongxia
EXPNO                   14
PROCNO                   1
Date_             20140318
Time                 16.36
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        4
DS                        2
SWH              12335.526 Hz
FIDRES            0.188225 Hz
AQ               2.6564426 sec
RG                     90.5
DW                40.533 usec
DE                 6.50 usec
TE                 296.9 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.00 usec
SI                      32768
SF            600.1300241 MHz
WDW                     EM
SSB                      0
LB                    0.10 Hz
GB                       0
PC                    1.00
```



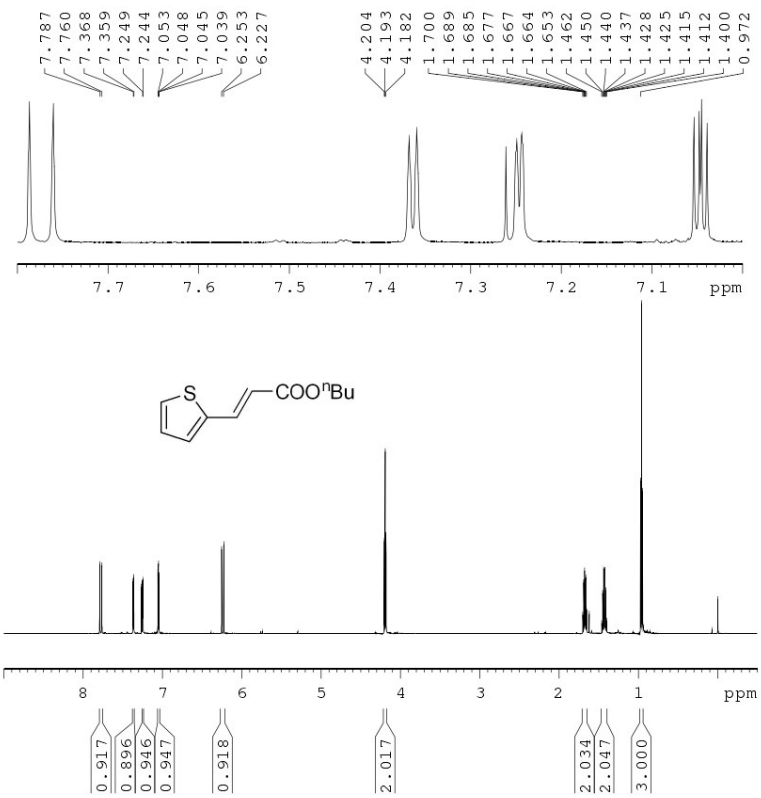
BRUKER

```
NAME          zhangcongxia
EXPNO                   15
PROCNO                   1
Date_             20140318
Time                 16.28
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        4
DS                        2
SWH              12335.526 Hz
FIDRES            0.188225 Hz
AQ               2.6564426 sec
RG                      114
DW                40.533 usec
DE                 6.50 usec
TE                 296.8 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.00 usec
SI                      32768
SF            600.1300198 MHz
WDW                     EM
SSB                      0
LB                    0.10 Hz
GB                       0
PC                    1.00
```



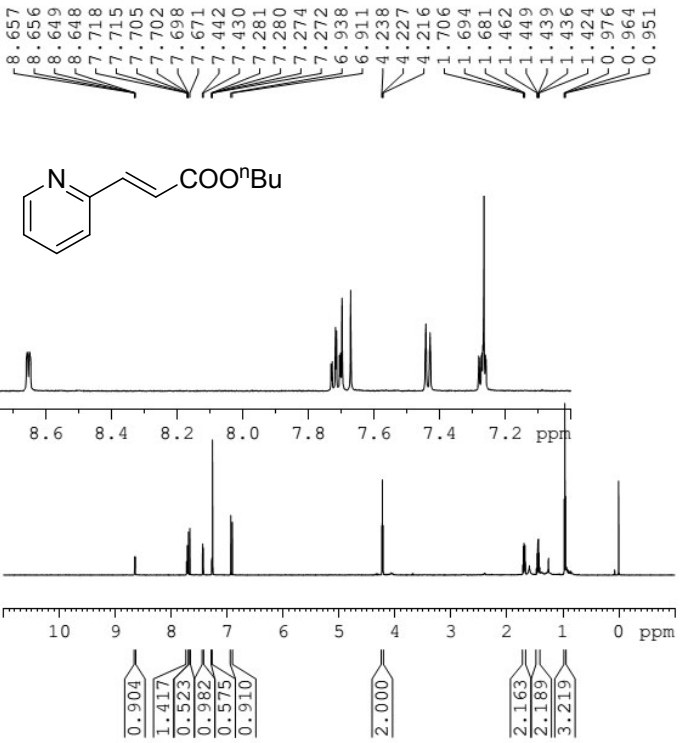
NAME zhangcongxia
EXPNO 16
PROCNO 1
Date_ 20140303
Time 15.43
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 181
DW 40.533 usec
DE 6.50 usec
TE 294.4 K
D1 1.00000000 sec

-------- CHANNEL f1 --------
NUC1 1H
P1 14.00 usec
SI 32768
SF 600.1300151 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



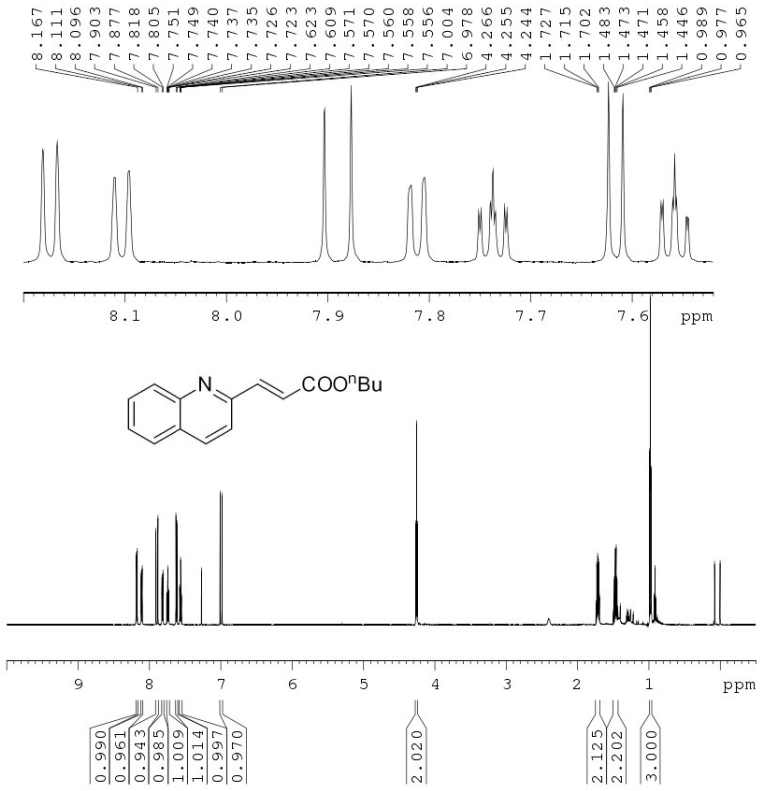
NAME zhangcongxia
EXPNO 17
PROCNO 1
Date_ 20140318
Time 16.41
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 114
DW 40.533 usec
DE 6.50 usec
TE 296.9 K
D1 1.00000000 sec

-------- CHANNEL f1 --------
NUC1 1H
P1 14.00 usec
SI 32768
SF 600.1300170 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



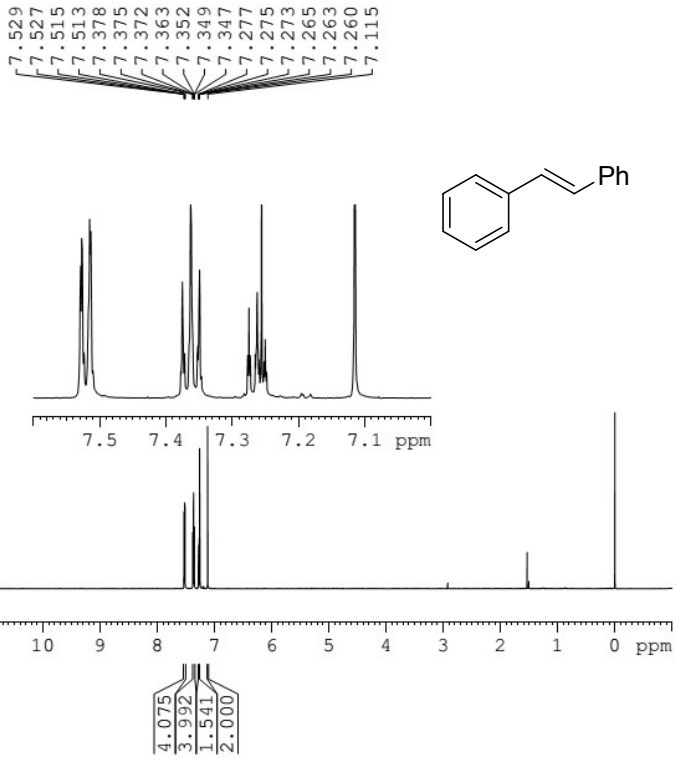
BRUKER

```
NAME          20140327linana
EXPNO                      5
PROCNO                     1
Date_               20140327
Time                   10.36
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                         8
DS                         2
SWH                12335.526 Hz
FIDRES              0.188225 Hz
AQ                 2.6564426 sec
RG                       203
DW                    40.533 usec
DE                     6.50 usec
TE                    295.3 K
D1                 1.00000000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                     14.00 usec
SI                         32768
SF               600.1300143 MHz
WDW                        EM
SSB                         0
LB                     -0.10 Hz
GB                          0
PC                      1.00
```

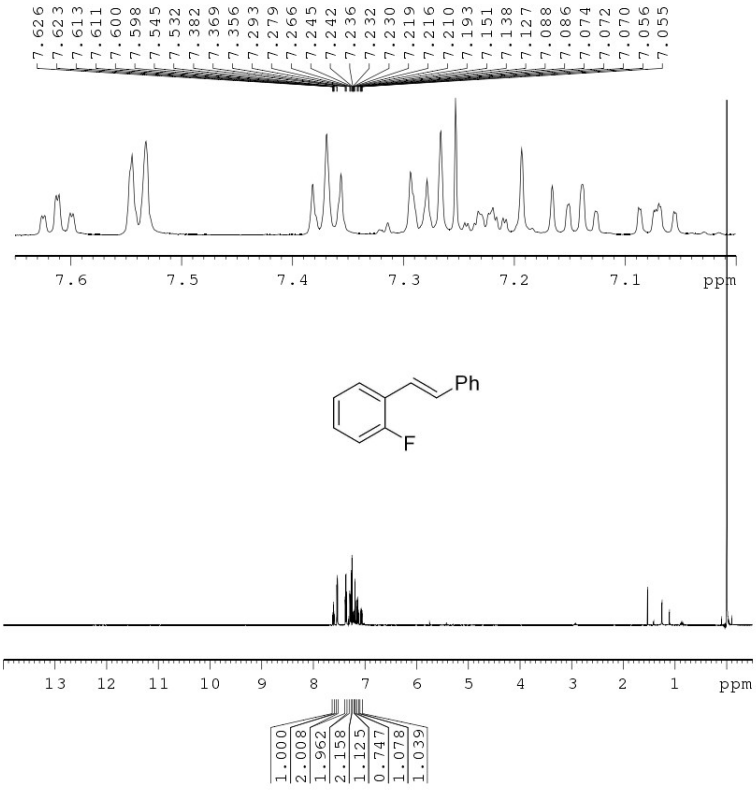


BRUKER

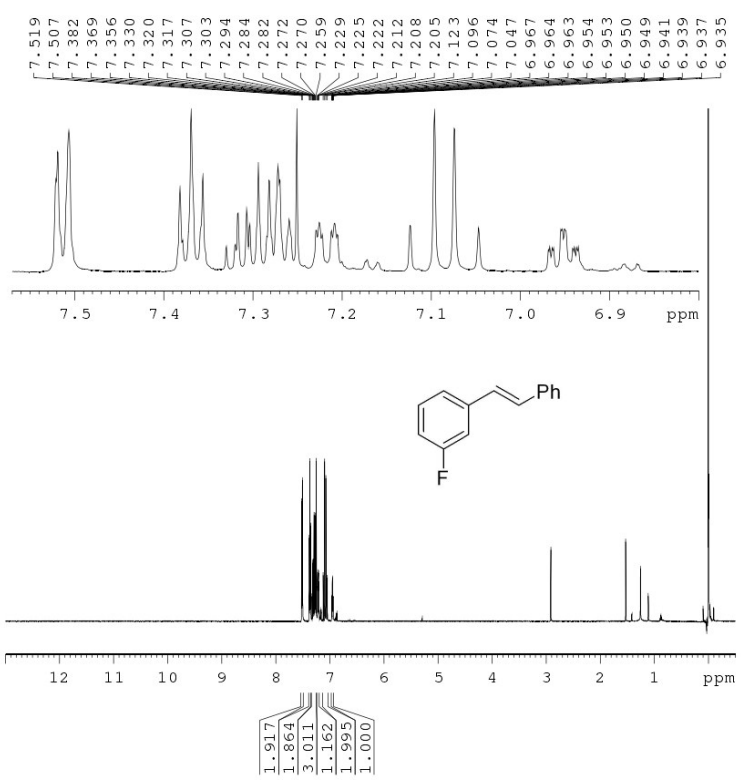
```
NAME          zhangcongxia
EXPNO                    19
PROCNO                    1
Date_              20140318
Time                  16.07
INSTRUM               spect
PROBHD    5 mm PADUL 13C
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        4
DS                        2
SWH               12335.526 Hz
FIDRES             0.188225 Hz
AQ                2.6564426 sec
RG                      114
DW                   40.533 usec
DE                    6.50 usec
TE                   296.9 K
D1                1.00000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                    14.00 usec
SI                        32768
SF              600.1300141 MHz
WDW                       EM
SSB                        0
LB                    0.10 Hz
GB                         0
PC                     1.00
```



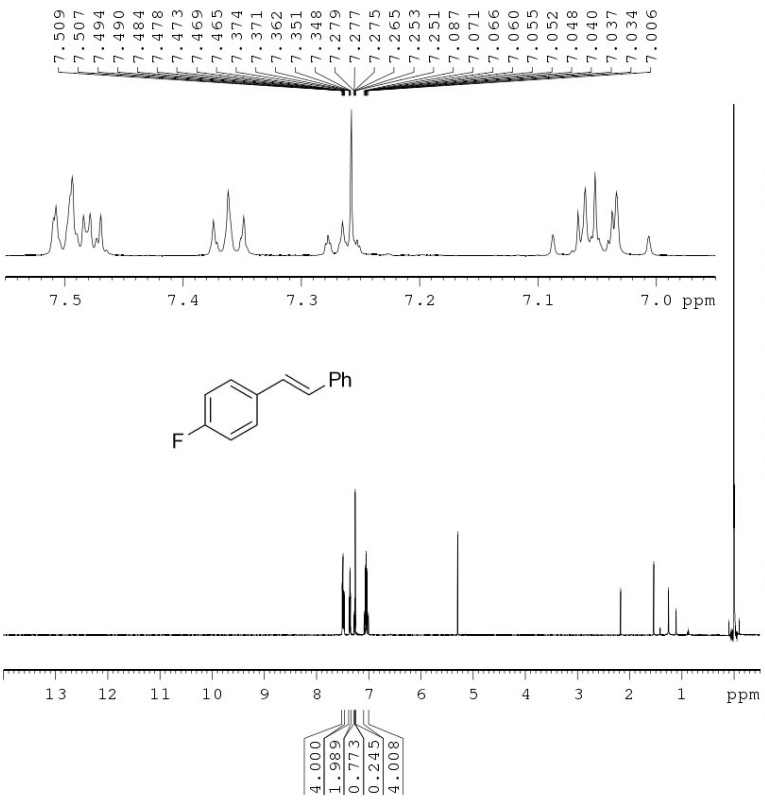

```
NAME        20140327linana
EXPNO                    6
PROCNO                   1
Date_             20140327
Time                 10.40
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH             12335.526 Hz
FIDRES           0.188225 Hz
AQ              2.6564426 sec
RG                     203
DW                40.533 usec
DE                 6.50 usec
TE                 295.4 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.00 usec
SI                   32768
SF           600.1300200 MHz
WDW                     EM
SSB                      0
LB                   -0.10 Hz
GB                       0
PC                    1.00
```



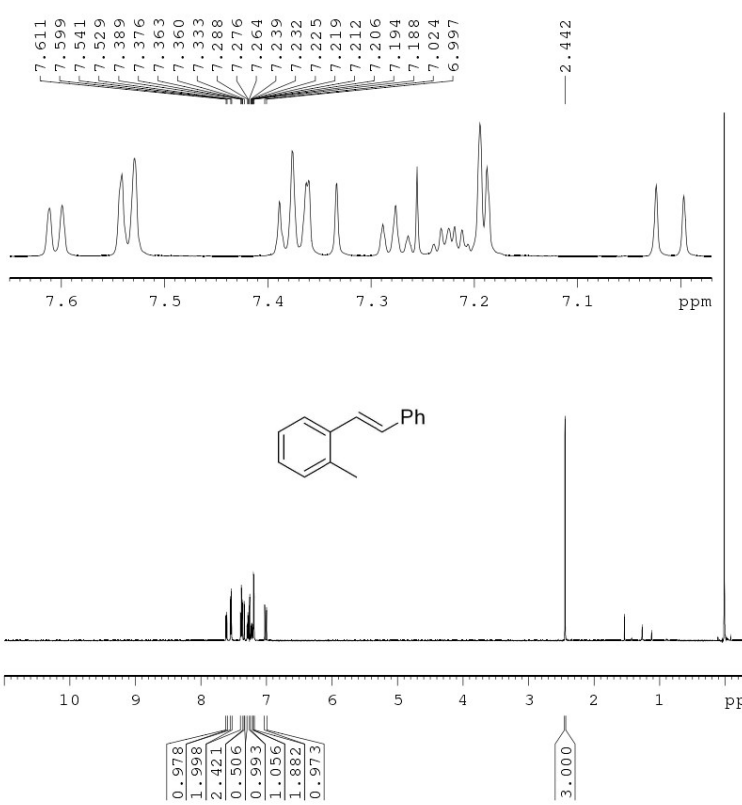

```
NAME        zhangcongxia
EXPNO                  21
PROCNO                  1
Date_           20140311
Time               10.14
INSTRUM            spect
PROBHD    5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                   203
DW              40.533 usec
DE               6.50 usec
TE               294.9 K
D1            1.00000000 sec

-------- CHANNEL f1 --------
NUC1                   1H
P1                14.00 usec
SI                 32768
SF         600.1300212 MHz
WDW                   EM
SSB                    0
LB                0.10 Hz
GB                     0
PC                  1.00
```




```
NAME          zhangcongxia
EXPNO                   22
PROCNO                   1
Date_             20140311
Time                 10.18
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG               zg30
TD                   65536
SOLVENT              CDC13
NS                       8
DS                       2
SWH             12335.526 Hz
FIDRES           0.188225 Hz
AQ              2.6564426 sec
RG                     203
DW                40.533 usec
DE                 6.50 usec
TE                294.9 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.00 usec
SI                   32768
SF           600.1300233 MHz
WDW                     EM
SSB                      0
LB                  0.10 Hz
GB                       0
PC                    1.00
```

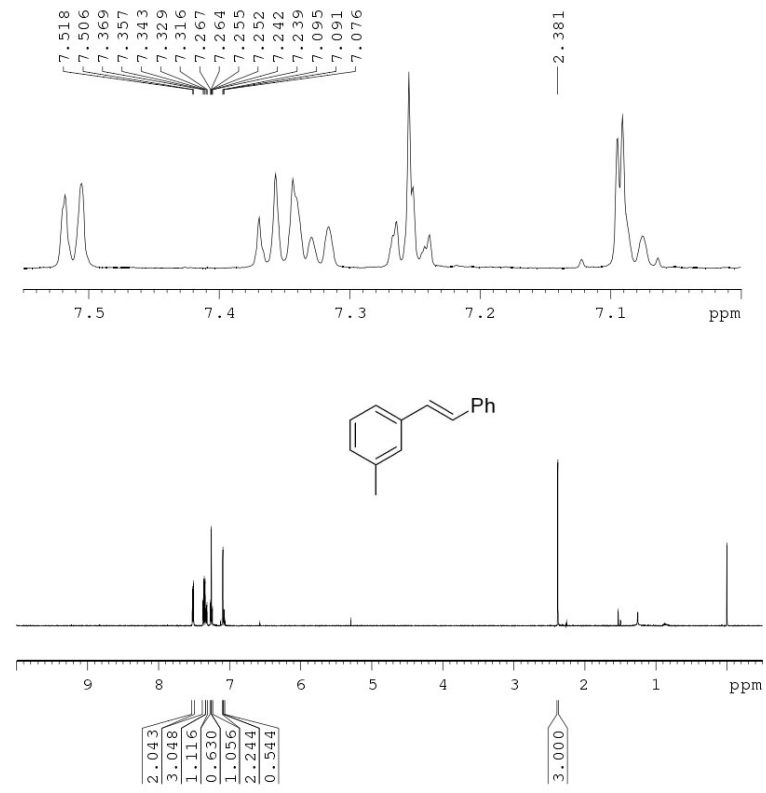



```
NAME          zhangcongxia
EXPNO                   23
PROCNO                   1
Date_             20140311
Time                 10.22
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG               zg30
TD                   65536
SOLVENT              CDC13
NS                       8
DS                       2
SWH             12335.526 Hz
FIDRES           0.188225 Hz
AQ              2.6564426 sec
RG                     203
DW                40.533 usec
DE                 6.50 usec
TE                294.9 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.00 usec
SI                   32768
SF           600.1300191 MHz
WDW                     EM
SSB                      0
LB                  0.10 Hz
GB                       0
PC                    1.00
```



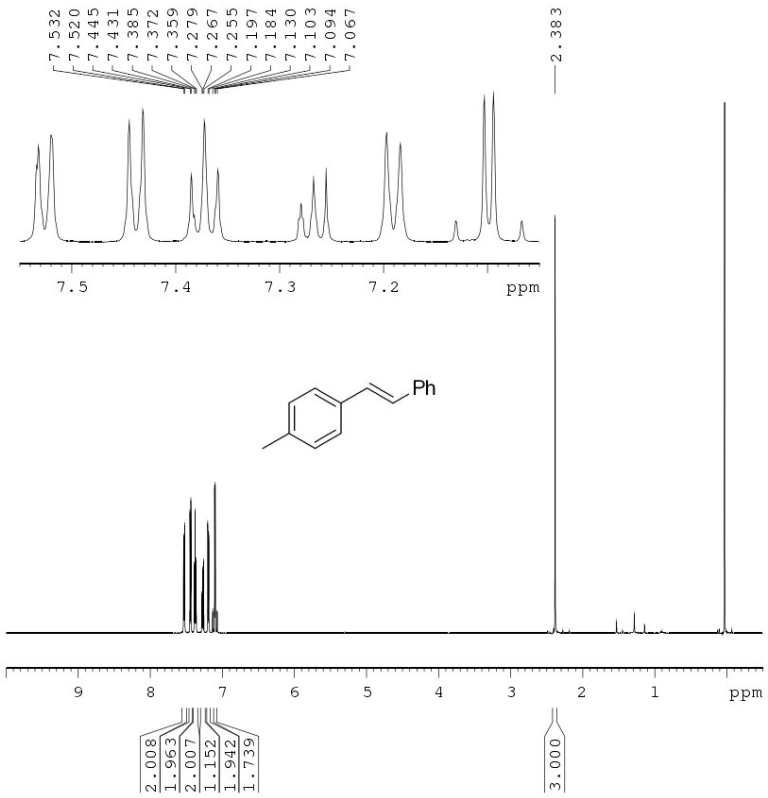
BRUKER

NAME	zhangcongxia
EXPNO	24
PROCNO	1
Date_	20140311
Time	10.26
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	12335.526 Hz
FIDRES	0.188225 Hz
AQ	2.6564426 sec
RG	203
DW	40.533 usec
DE	6.50 usec
TE	294.9 K
D1	1.00000000 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
SI	32768
SF	600.1300205 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00



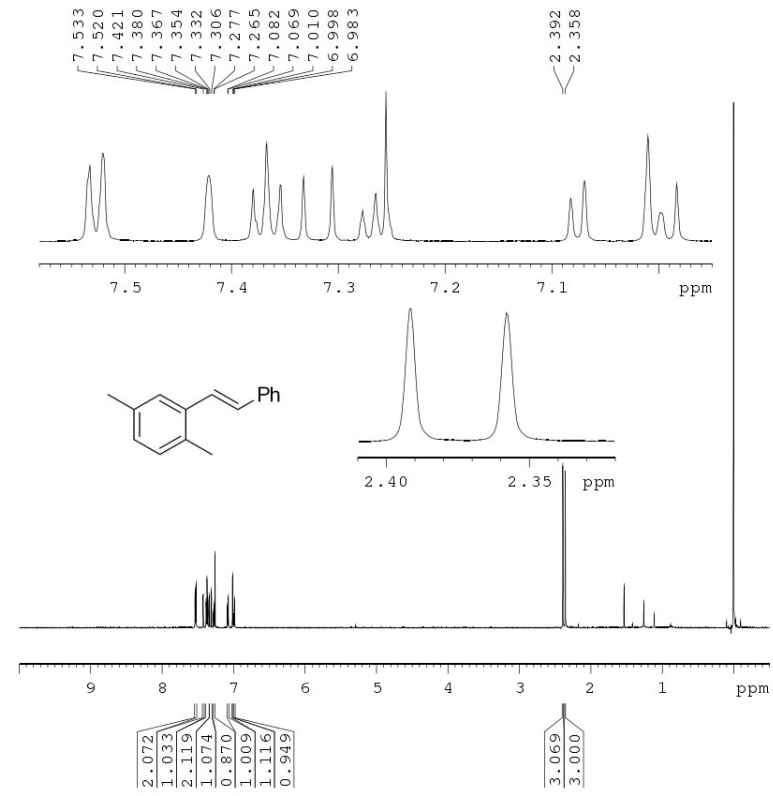
BRUKER

NAME	zhangcongxia
EXPNO	25
PROCNO	1
Date_	20140318
Time	16.44
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	2
DS	2
SWH	12335.526 Hz
FIDRES	0.188225 Hz
AQ	2.6564426 sec
RG	203
DW	40.533 usec
DE	6.50 usec
TE	296.9 K
D1	1.00000000 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
SI	32768
SF	600.1300209 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00



BRUKER

```
NAME          zhangcongxia
EXPNO                   27
PROCNO                   1
Date_             20140311
Time                 10.31
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       2
SWH             12335.526 Hz
FIDRES           0.188225 Hz
AQ              2.6564426 sec
RG                     101
DW                40.533 usec
DE                 6.50 usec
TE                 294.9 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.00 usec
SI                   32768
SF            600.1300205 MHz
WDW                     EM
SSB                      0
LB                  0.10 Hz
GB                       0
PC                    1.00
```



BRUKER

```
NAME          zhangcongxia
EXPNO                   28
PROCNO                   1
Date_             20140311
Time                 10.35
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       2
SWH             12335.526 Hz
FIDRES           0.188225 Hz
AQ              2.6564426 sec
RG                     203
DW                40.533 usec
DE                 6.50 usec
TE                 295.0 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.00 usec
SI                   32768
SF            600.1300205 MHz
WDW                     EM
SSB                      0
LB                  0.10 Hz
GB                       0
PC                    1.00
```