

Supporting Information

Homocoupling of Hetroaryl/Aryl/Alkyl Grignard Reagents: I₂-Promoted, or Ni- or Pd- or Cu- or Nano-Fe-based salt catalyzed

Xing Li,* Dongjun Li, Yingjun Li, Honghong Chang, Wenchao Gao and Wenlong Wei*

Department of Chemistry and Chemical Engineering, Taiyuan University of Technology, 79 West Yingze Street, Taiyuan 030024, People's Republic of China. E-mail: weiwenlong@tyut.edu.cn and lixing@tyut.edu.cn

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1. General

All reagents were purchased from commercial sources and used without further purification, unless otherwise indicated. Deuterated solvents were purchased from Aldrich. Solvents were distilled over Na, or NaH, or CaH, respectively before use. Phenylmagnesium bromide (1.0 mol/mL in THF), *p*-tolylmagnesium bromide (1.0 mol/mL in THF), 4-anisylmagnesium bromide (1.0 mol/mL in THF), 3-fluorophenylmagnesium bromide (1.0 mol/mL in THF), 4-fluorophenylmagnesium bromide (1.0 mol/mL in THF), phenylmagnesium chloride (1.0 mol/mL in THF), and *p*-tolylmagnesium chloride (1.0 mol/mL in THF) were also purchased from Sigma-Aldrich. *o*-tolylmagnesium bromide (1.0 mol/mL in THF), *m*-tolylmagnesium bromide (1.0 mol/mL in THF), 2-thienylmagnesium bromide (1.0 mol/mL in THF), and *m*-tolylmagnesium chloride (2.0 mol/mL in THF) were purchased from Energy Chemical. 2-pyridinemagnesium bromide (1.0 mol/mL in THF) and 3-pyridinemagnesium bromide (1.0 mol/mL in THF) were prepared according to the general procedure by us. Column chromatography was performed on silica gel (200-300 mesh) with petroleum ether (solvent A)/ethyl acetate (solvent B) gradients, unless otherwise specified. All yields were referred to isolated yields (average of two runs) of compounds. The known compounds were partly characterized by melting points (for solid samples), ¹H NMR, and compared to authentic samples or the literature data. Melting points were measured with a RD-II digital melting point apparatus and are uncorrected. ¹H NMR data were acquired at 300 K on a Bruker Advance 600 MHz spectrometer or Avian Inova 500 MHz spectrometer using CDCl₃ as solvent. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard (CDCl₃ = 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. Optimization of reaction conditions for other four reaction systems

2.1 Optimization of reaction conditions for Pd(OAc)₂

In the study on the Pd(OAc)₂-catalyzed homo-coupling reaction using phenyl magnesium bromide as a model substrate under N₂ atmosphere, the exploration of several frequently-used anhydrous solvents in cross-coupling reactions such as toluene, THF, ethyl ether and CH₂Cl₂ revealed that toluene was appropriate because of higher boiling point (Table 1, entry 7 vs. 2-5). Toluene provided lower yield at 25 °C (Table 1, entry 1 vs. 2-4). 75% yield was obtained when the temperature was increased to 100 °C (Table 1, entry 7). Several oxidants were then screened. It was found that inexpensive LiClO₄ was excellent oxidant for the homocoupling, giving biphenyl in 98% yield (Table 1, entry 8 vs. 9 and 10). Thus, in the presence of 10 mol% Pd(OAc)₂ and 1.0 equiv. of LiClO₄, the homo-coupling was highly efficient in toluene at 100 °C for 15 h.

Table 1 Screening of reaction conditions on the homocoupling of PhMgBr with Pd(OAc)₂^[a]

| Entry | Solvent | Oxidant | T (°C) | Yield ^[b] (%) |
|-------|---------|---------|--------|--------------------------|
|-------|---------|---------|--------|--------------------------|

| | | | | |
|----|---------------------------------|---|-----|----|
| 1 | Toluene | - | 25 | 31 |
| 2 | THF | - | 25 | 55 |
| 3 | Et ₂ O | - | 25 | 40 |
| 4 | CH ₂ Cl ₂ | - | 25 | 32 |
| 5 | THF | - | 65 | 68 |
| 6 | Toluene | - | 65 | 53 |
| 7 | Toluene | - | 100 | 75 |
| 8 | Toluene | LiClO ₄ | 100 | 98 |
| 9 | Toluene | K ₂ Cr ₂ O ₇ | 100 | 46 |
| 10 | Toluene | Cu(OAc) ₂ | 100 | 39 |
| 11 | THF | LiClO ₄ | 65 | 81 |

^[a] Reaction conditions: phenylmagnesium bromide (0.3 mmol), Pd(OAc)₂ (10 mol%), oxidant (1 equiv.), solvent (2.0 mL), time (15 h), N₂. ^[b] Isolated yield.

2.2 Optimization of reaction conditions for Ni(OAc)₂

First, several solvents were examined for the Ni(OAc)₂·4H₂O-catalyzed homo-coupling of phenylmagnesium bromide. It was found that CH₂Cl₂ was a good solvent for the homo-coupling, giving biphenyl in 56% yield at 25 °C in the presence of 10 mol% Ni(OAc)₂·4H₂O (Table 2, entry 4 vs. 1-3). Several oxidants were then screened and Ag₂O provided the higher yield (Table 2, entry 7 vs. 5-6, 8). Fortunately, 95% yield was obtained when the Ni(OAc)₂ was used (Table 2, entry 9).

Table 2 Screening of reaction conditions on the homocoupling of PhMgBr with Ni(OAc)₂^[a]

| Entry | Solvent | Oxidant (equiv.) | Yield ^[b] (%) |
|-------|---------------------------------|---------------------------------------|--------------------------|
| 1 | Toluene | - | 30 |
| 2 | THF | - | 15 |
| 3 | Et ₂ O | - | 44 |
| 4 | CH ₂ Cl ₂ | - | 56 |
| 5 | CH ₂ Cl ₂ | LiClO ₄ (1.0) | 66 |
| 6 | CH ₂ Cl ₂ | AgNO ₃ (1.0) | 73 |
| 7 | CH ₂ Cl ₂ | Ag ₂ O (1.0) | 87 |
| 8 | CH ₂ Cl ₂ | Ag ₂ SO ₄ (1.0) | 85 |
| 9 | CH ₂ Cl ₂ | Ag ₂ O (1.0) | 95 ^[c] |
| 10 | CH ₂ Cl ₂ | Ag ₂ O (1.2) | 95 ^[c] |

^[a] Reaction conditions: phenylmagnesium bromide (0.3 mmol), Ni(OAc)₂·4H₂O or Ni(OAc)₂ (10 mol%), solvent (1.5 mL), time (18 h), N₂. ^[b] Isolated yield. ^[c] Ni(OAc)₂ was used.

2.3 Optimization of reaction conditions for CuI

Subsequently, CuI-catalyzed homo-coupling of phenylmagnesium bromide was investigated under air atmosphere. The examination of solvents showed that toluene was suitable for the homo-coupling because of its higher boiling point, giving biphenyl in 79% yield at 100 °C in the presence of 5 mol% CuI (Table 3, entry 6 vs. 1-5). Then we tested the influence of the amount of CuI on the yield. When the catalyst loading increased to 15 mol%, 95% yield was obtained (Table 3, entry 8 vs. 6 and 7).

Table 3 Screening of reaction conditions on the homocoupling of PhMgBr with CuI^[a]

| Entry | Solvent | CuI (mol%) | T (°C) | Yield ^[b] (%) |
|-------|---------------------------------|------------|--------|--------------------------|
| 1 | Toluene | 5 | 25 | 42 |
| 2 | THF | 5 | 25 | 60 |
| 3 | Et ₂ O | 5 | 25 | 33 |
| 4 | CH ₂ Cl ₂ | 5 | 25 | 45 |
| 5 | THF | 5 | 65 | 63 |
| 6 | Toluene | 5 | 100 | 79 |
| 7 | Toluene | 10 | 100 | 87 |
| 8 | Toluene | 15 | 100 | 95 |
| 9 | Toluene | 20 | 100 | 95 |

^[a] Reaction conditions: phenylmagnesium bromide (0.3 mmol), CuI, solvent (2 mL), time (15 h), air. ^[b] Isolated yield.

2.4 Optimization of reaction conditions for nano-Fe₃O₄

The investigation was conducted using nano-Fe₃O₄ as a catalyst and the results are summarized in Table 4. Toluene was still found to be a good solvent for the homo-coupling, giving biphenyl in 65% yield in the presence of 10 mol% nano-sized Fe₃O₄ at 100 °C (Table 4, entry 7 vs. 1-6). Then the effect of oxidants was tested. 84 % yield was obtained when AgNO₃ was used as an oxidant (Table 4, entry 10 vs. 8-9 and 11-12). Moreover, 93% yield was provided when 1.2 equiv. of AgNO₃ was adopted (Table 4, entry 13).

Table 4 Screening of reaction conditions on the homocoupling of PhMgBr with nano-Fe₃O₄^[a]

| Entry | Solvent | Oxidant (equiv.) | T (°C) | Yield ^[b] (%) |
|-------|---------------------------------|------------------|--------|--------------------------|
| 1 | Toluene | - | 25 | 29 |
| 2 | THF | - | 25 | 28 |
| 3 | Et ₂ O | - | 25 | Trace |
| 4 | CH ₂ Cl ₂ | - | 25 | 8 |
| 5 | Toluene | - | 60 | 44 |

| | | | | |
|----|-------------|---------------------------------------|-----|----|
| | e | | | |
| 6 | Toluen e | - | 80 | 50 |
| 7 | Toluen e | - | 100 | 65 |
| 8 | Toluen e | LiClO ₄ (1.0) | 100 | 9 |
| 9 | Toluen e | CuCl ₂ (1.0) | 100 | 71 |
| 10 | Toluen e | AgNO ₃ (1.0) | 100 | 84 |
| 11 | Toluen e | Ag ₂ O (1.0) | 100 | 73 |
| 12 | Toluen e | Ag ₂ SO ₄ (1.0) | 100 | 67 |
| 13 | Toluen e | AgNO ₃ (1.2) | 100 | 93 |
| 14 | Toluen e | AgNO ₃ (1.5) | 100 | 91 |

^[a] Reaction conditions: phenylmagnesium bromide (0.3 mmol), nano-sized Fe₃O₄ (10 mol%), solvent (2 mL), time (20 h), N₂. ^[b] Isolated yield.

3. Proposed mechanism

3.1 A proposed mechanism for the CuI-catalyzed homocoupling

A proposed mechanism is depicted in Figure 1 for the CuI-catalyzed reaction. A low-valent copper species is first generated through reduction by the Grignard reagent which is also a strong reducing agent in the catalytic cycle. The oxidative addition of molecular oxygen to a low-valent copper species **A** then forms a peroxo-copper (II) intermediate **B**, which is the key step for this catalytic cycle. Subsequently, it reacts with two equivalents of RMgX to give intermediate **C** and XMgOOMgX. At last, the intermediate **C** undergoes rapid reductive elimination to provide the homocoupling product R–R and release the low-valent copper species **A**.

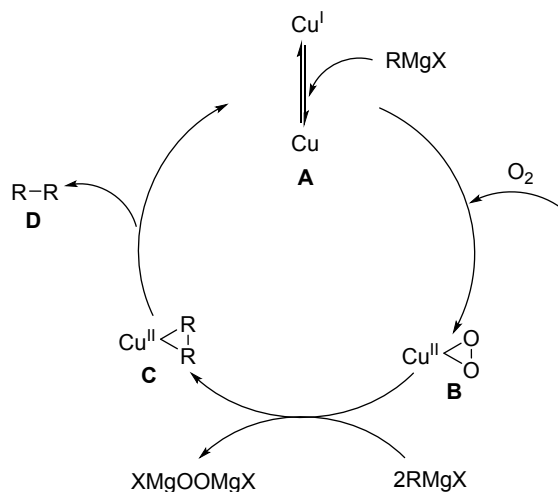


Figure 1. A proposed mechanism for the CuI-catalyzed homocoupling

3.2 A proposed mechanism for the nano-Fe-catalyzed homocoupling

According to the reported literatures concerning iron salts catalysis, a possible mechanism is proposed as follows (Figure 2): iron species ($[\text{Fe}]^n$) can be first rapidly reduced in situ into an iron species **A** by the Grignard reagent. Then this species **A** reacts with Grignard reagent to generate an iron intermediate **B** in the presence of AgNO_3 . This intermediate **B** could then give the homocoupling product **C** by reductive elimination and release the activated iron species ($[\text{Fe}]^n$).

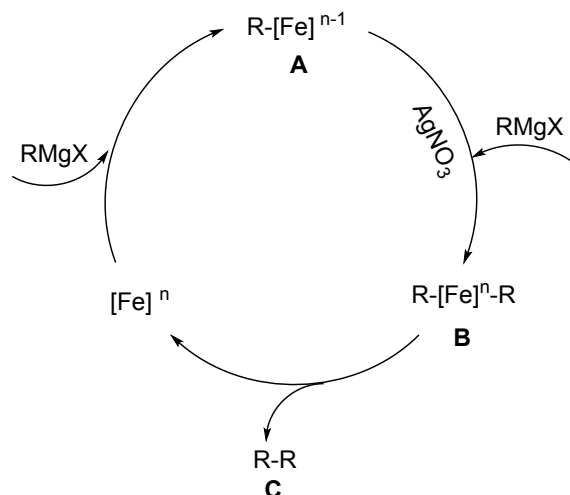


Figure 2. A proposed mechanism for the nano-Fe-catalyzed homocoupling

4. General reaction procedure

4.1 General procedure for homocoupling of Grignard reagents mediated by I_2

To a tube equipped with a magnetic stir bar were added I_2 (61.0 mg, 0.8 equiv.) and 0.3 mmol of phenylmagnesium bromide in turn. Subsequently, the solvent (toluene, 2.0 mL) was added under N_2 atmosphere. The reaction system was then heated to 110 °C and stirred for certain time. At last, the reaction mixture was purified by silica gel column chromatography with petroleum ether or petroleum ether/EtOAc to afford the desired coupling product.

4.2 General procedure for homocoupling of Grignard reagents catalyzed by $\text{Pd}(\text{OAc})_2$

To a tube equipped with a magnetic stir bar were added $\text{Pd}(\text{OAc})_2$ (6.7 mg, 10 mol%), 1.0 equiv. of LiClO_4 (32.0 mg, 0.3 mmol) and 0.3 mmol of phenylmagnesium bromide in turn. Subsequently, the solvent (toluene, 2.0 mL) was added under N_2 atmosphere. The reaction system was then heated to 100 °C and stirred for certain time. At last, the reaction mixture was purified by silica gel column chromatography with petroleum ether or petroleum ether/EtOAc to afford the desired coupling product.

4.3 General procedure for homocoupling of Grignard reagents catalyzed by Ni(OAc)₂

To a tube equipped with a magnetic stir bar were added Ni(OAc)₂ (5.4 mg, 10 mol%), 1.0 equiv. of Ag₂O (37.2 mg, 0.3 mmol) and 0.3 mmol of phenylmagnesium bromide in turn. Subsequently, the solvent (CH₂Cl₂, 1.5 mL) was added under N₂ atmosphere. The reaction system reacted at room temperature and stirred for certain time. At last, the reaction mixture was purified by silica gel column chromatography with petroleum ether or petroleum ether/EtOAc to afford the desired coupling product.

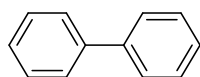
4.4 General procedure for homocoupling of Grignard reagents catalyzed by CuI

To a tube equipped with a magnetic stir bar were added CuI (8.6 mg, 15 mol%) and 0.3 mmol of phenylmagnesium bromide in turn. Subsequently, the solvent (toluene, 2.0 mL) was added under air atmosphere. The reaction system was then heated to 100 °C and stirred for certain time. At last, the reaction mixture was purified by silica gel column chromatography with petroleum ether or petroleum ether/EtOAc to afford the desired coupling product.

4.5 General procedure for homocoupling of Grignard reagents catalyzed by nano-Fe₃O₄

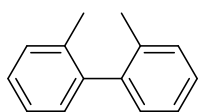
To a tube equipped with a magnetic stir bar were added nano-Fe₃O₄ (6.9 mg, 10 mol%), 1.2 equiv. of AgNO₃ (61.4 mg, 0.3 mmol) and 0.3 mmol of phenylmagnesium bromide in turn. Subsequently, the solvent (toluene, 2.0 mL) was added under N₂ atmosphere. The reaction system was then heated to 100 °C and stirred for certain time. At last, the reaction mixture was purified by silica gel column chromatography with petroleum ether or petroleum ether/EtOAc to afford the desired coupling product.

5. Characterization data for homocoupling products



Biphenyl^[2]

R_f = 0.6 (Petroleum ether); White solid; M.p. = 65-66 °C^[2]; ¹H NMR (600 MHz, CDCl₃): δ = 7.36-7.39 (m, 2H), 7.46-7.48 (m, 4H), 7.62-7.64 (m, 4H) ppm.



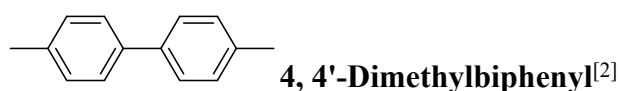
2, 2'-Dimethylbiphenyl^[2]

R_f = 0.6 (Petroleum ether); colorless liquid; ¹H

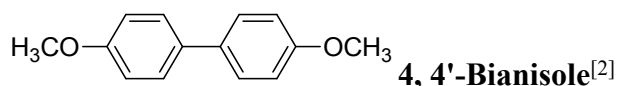
NMR (600 MHz, CDCl₃): δ = 7.24-7.25 (m, 4H), 7.19-7.22 (m, 2H), 7.09 (d, J = 7.2 Hz, 2H), 2.04 (s, 6H) ppm.



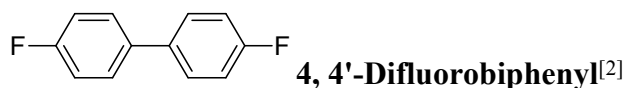
R_f = 0.6 (Petroleum ether); colorless liquid; ¹H NMR (600 MHz, CDCl₃): δ = 7.37 (d, J = 13.8 Hz, 4H), 7.31 (t, J = 10.8 Hz, 2H), 7.14 (d, J = 11.4 Hz, 2H), 2.41 (s, 6H) ppm.



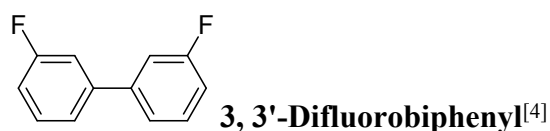
R_f = 0.6 (Petroleum ether); White solid, M.p. = 118-119 °C^[2]; R_f = 0.6 (Petroleum ether); ¹H NMR (400 MHz, CDCl₃): δ = 7.47 (dd, J = 4.8, 1.2 Hz, 4H), 7.22 (d, J = 7.6 Hz, 4H), 2.38 (s, 6H).



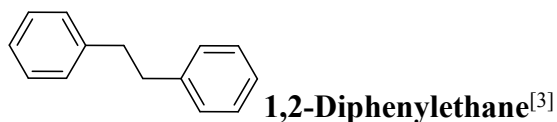
R_f = 0.3 (Petroleum ether/AcOEt = 30/1 v/v); light yellow solid; M.p. = 174-175 °C^[2]; ¹H NMR (600 MHz, CDCl₃): δ = 7.45-7.48 (m, 4H), 6.93-6.96 (m 4H), 3.83 (s, 6H) ppm.



R_f = 0.6 (Petroleum ether); light yellow solid; M.p. = 64-66 °C^[2]; ¹H NMR (600 MHz, CDCl₃): δ = 7.46-7.48 (m, 4H), 7.11 (t, J = 8.4 Hz, 4H) ppm.



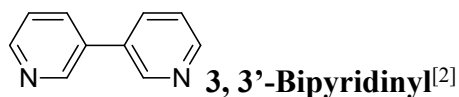
R_f = 0.6 (Petroleum ether); colorless liquid; ¹H NMR (600 MHz, CDCl₃): δ = 7.37 (td, J = 6.0, 1.8 Hz, 2H), 7.33 (dd, J = 5.4, 1.2 Hz, 2H), 7.24 (dt, J = 6.0, 1.8 Hz, 2H), 7.04-7.07 (m, 2H) ppm.



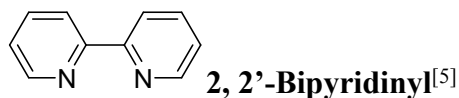
$R_f = 0.6$ (Petroleum ether); colorless liquid; $^1\text{H NMR}$ (600 MHz, CDCl_3): $\delta = 7.28$ (t, $J = 7.2$ Hz, 4H), 7.19 (t, $J = 7.2$ Hz, 6H), 2.92 (s, 4H) ppm.



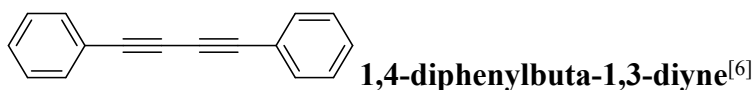
$R_f = 0.6$ (Petroleum ether); colorless liquid; $^1\text{H NMR}$ (600 MHz, CDCl_3): $\delta = 7.20$ (dd, $J = 4.2, 1.2$ Hz, 2H), 7.17 (dd, $J = 3.0, 0.6$ Hz, 2H), 7.00 (dd, $J = 3.6, 1.8$ Hz, 2H) ppm.



$R_f = 0.4$ (Petroleum ether/AcOEt = 20/1 v/v); white solid; M.p. = 67-68 °C^[2]; $^1\text{H NMR}$ (600 MHz, CDCl_3): $\delta = 7.71-7.73$ (m, 4H), 7.51-7.54 (m, 4H) ppm.



$R_f = 0.4$ (Petroleum ether/AcOEt = 20/1 v/v); white solid; M.p. = 72-74 °C^[5]; $^1\text{H NMR}$ (400 MHz, CDCl_3): $\delta = 8.68$ (d, $J = 4.4$ Hz, 2H), 8.38 (d, $J = 8.0$ Hz, 2H), 7.79 (td, $J = 6.0, 1.6$ Hz, 2H), 7.29-7.32 (m, 2H) ppm.



White solid; M.p. = 86-88 °C; $R_f = 0.5$ (Petroleum ether); $^1\text{H NMR}$ (400 MHz, CDCl_3): $\delta = 7.32 - 7.36$ (m, 2H), 7.36-7.39 (m, 4H), 7.53-7.56 (m, 4H) ppm.

6. Reference

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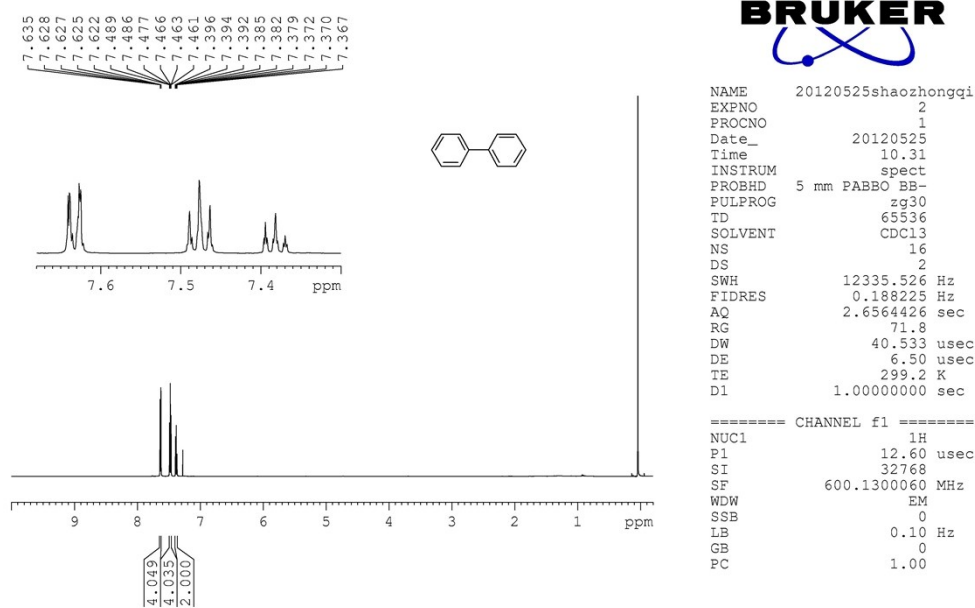
[3] X. Xu, D. Cheng, W. Pei, *J. Org. Chem.* 2006, **71**, 6637-6639.

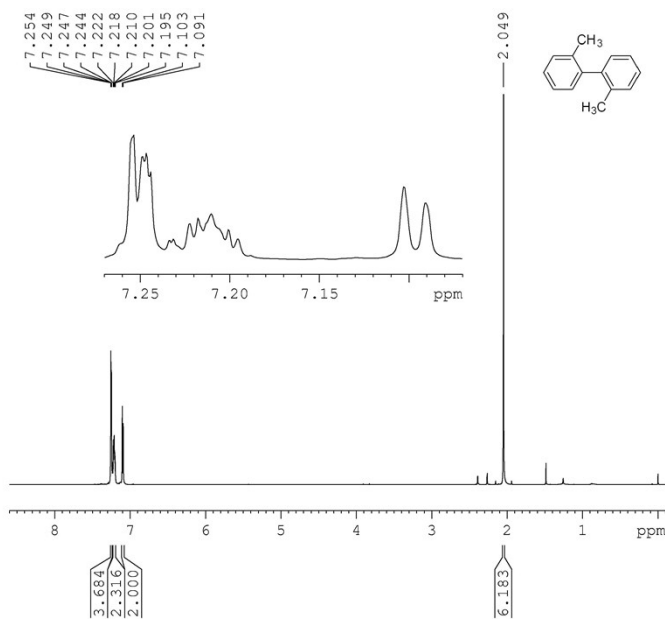
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[6] X. Jia, K. Yin, C. Li, J. Li, H. Bian, *Green Chem.* 2011, **13**, 2175.

7. Spectra



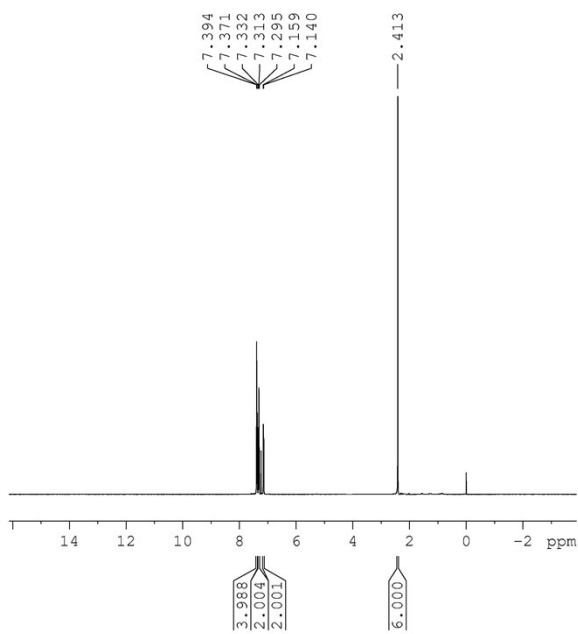


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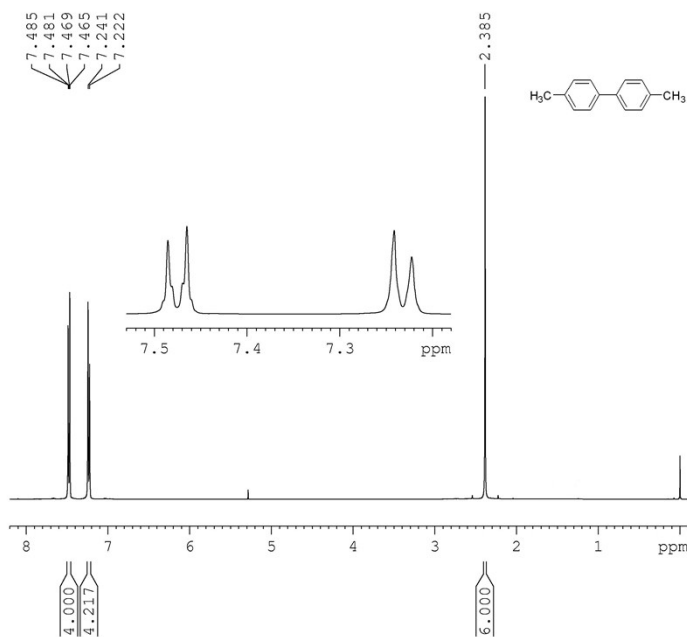


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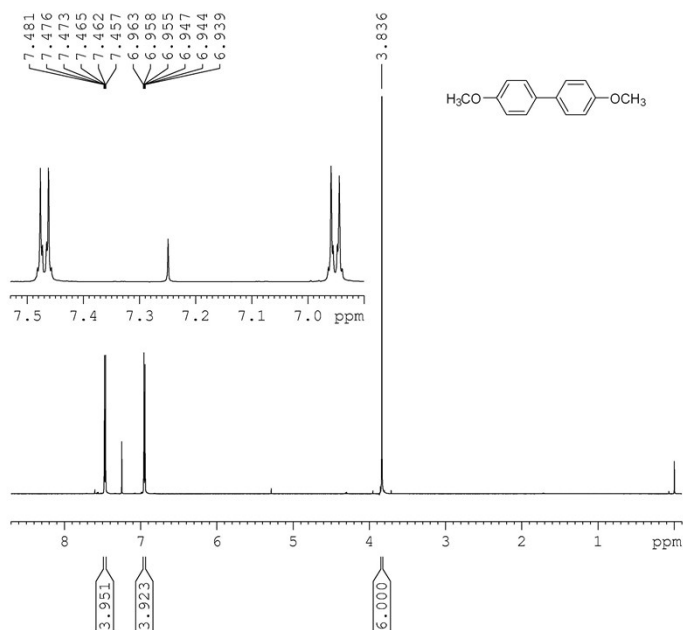
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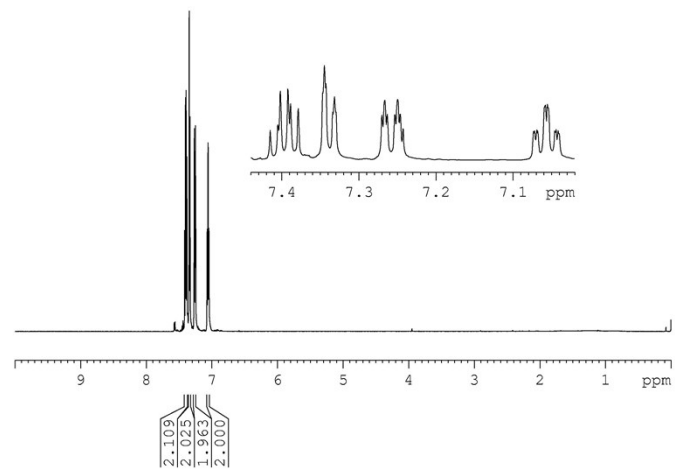
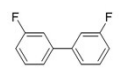
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 LB 0.30 Hz
 GB 0
 PC 1.00



NAME 20130523liyingjun
 EXPNO 4
 PROCNO 1
 Date_ 20130523
 Time 10.43
 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 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 600.1300228 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

7.932
7.938
7.979
7.945
7.943
7.934
7.932
7.970
7.966
7.963
7.953
7.950
7.946
7.973
7.972
7.969
7.968
7.958
7.955
7.954
7.946
7.944
7.940

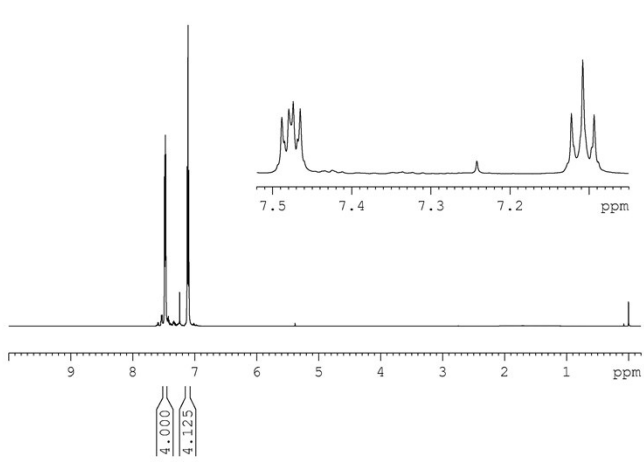
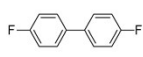


```

NAME      20131008zhangrui
EXPNO     5
PROCNO    1
Date_     20131008
Time      15.46
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         296.1 K
D1         1.00000000 sec

===== CHANNEL f1 =====
NUC1      1H
P1        14.00 usec
SI        32768
SF        600.1300283 MHz
WDW       EM
SSB       0
LB        0.10 Hz
GB        0
PC        1.00
  
```

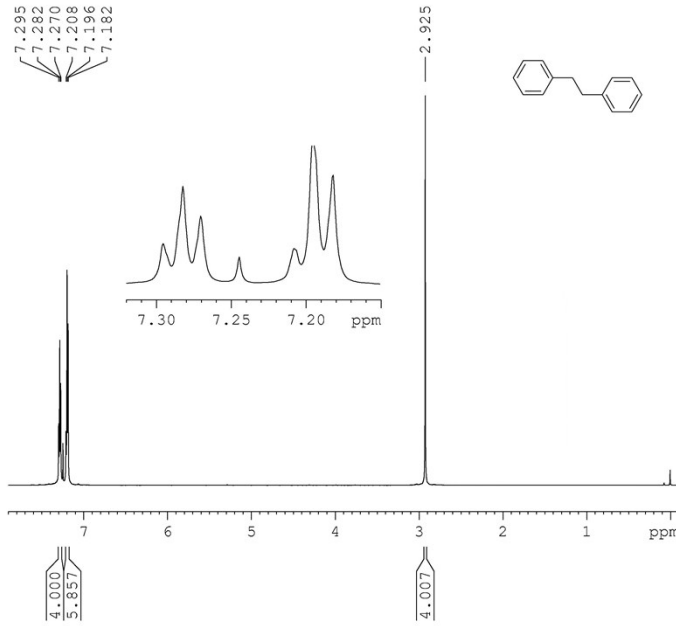
7.488
7.485
7.479
7.476
7.474
7.468
7.485
7.122
7.118
7.104



```

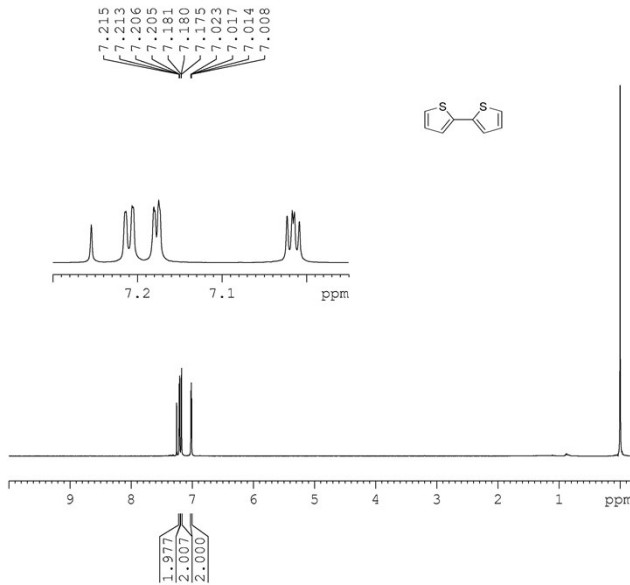
NAME      20130523liyingjun
EXPNO     5
PROCNO    1
Date_     20130523
Time      10.48
INSTRUM   spect
PROBHD    5 mm PADUL 13C
PULPROG   zg30
TD         65536
SOLVENT   CDC13
NS         8
DS         2
SWH       12335.526 Hz
FIDRES    0.188225 Hz
AQ         2.6564426 sec
RG         203
DW         40.533 usec
DE         6.50 usec
TE         296.1 K
D1         1.00000000 sec

===== CHANNEL f1 =====
NUC1      1H
P1        14.00 usec
SI        32768
SF        600.1300283 MHz
WDW       EM
SSB       0
LB        0.10 Hz
GB        0
PC        1.00
  
```



NAME 201312161iyijun
EXPNO 3
PROCNO 1
Date_ 20131216
Time 15.35
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 295.5 K
D1 1.0000000 sec

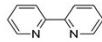
===== CHANNEL f1 =====
NUC1 1H
P1 14.00 usec
SI 32768
SF 600.1300268 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



NAME 201401031iyijun
EXPNO 1
PROCNO 1
Date_ 20140103
Time 15.45
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 291.0 K
D1 1.0000000 sec

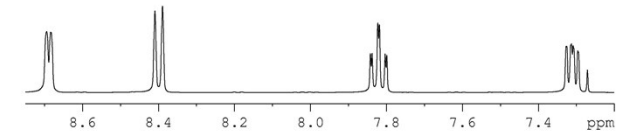
===== CHANNEL f1 =====
NUC1 1H
P1 14.00 usec
SI 32768
SF 600.1300205 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

8.683
8.409
8.389
7.842
7.838
7.823
7.819
7.804
7.799
7.727
7.725
7.715
7.713
7.709
7.706
7.697
7.695



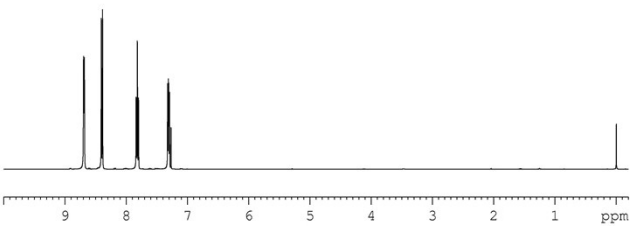
```

NAME      2015-10-29 tylg-chh
EXPNO     1
PROCNO    1
Date_     20151029
Time      17.33
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD        65536
SOLVENT   CDC13
NS        16
DS        2
SWH       8012.820 Hz
FIDRES    0.122266 Hz
AQ        4.0894966 sec
RG        60.71
DW        62.400 usec
DE        6.50 usec
TE        293.4 K
D1        1.00000000 sec
TD0       1
  
```

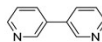


```

===== CHANNEL f1 =====
SF01    400.1324710 MHz
NUC1     1H
P1       9.70 usec
SI       65536
SF       400.1300055 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```

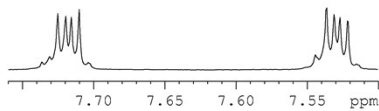


7.736
7.731
7.725
7.720
7.716
7.710
7.704
7.700
7.694
7.687
7.681
7.675
7.669
7.663



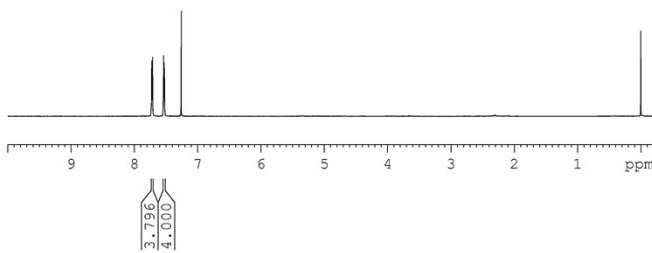
```

NAME      20140318liyingjun
EXPNO     6
PROCNO    1
Date_     20140318
Time      15.56
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        296.9 K
D1        1.00000000 sec
  
```



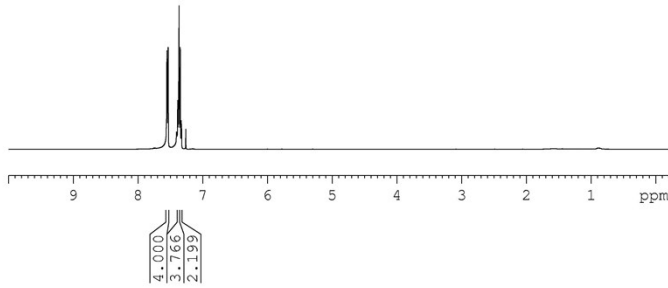
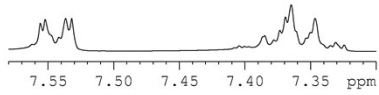
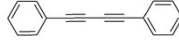
```

===== 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
  
```





7.556
7.552
7.542
7.537
7.532
7.386
7.385
7.378
7.373
7.369
7.365
7.355
7.350
7.346
7.335
7.331
7.324



NAME 2016-08-20 tyut-chh
EXPNO 10
PROCNO 1
Date_ 20160820
Time 21.43
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 60.71
DW 62.400 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.1324710 MHz
NUC1 1H
P1 9.70 usec
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00