

Observations on transition metal free biaryl coupling: Potassium *tert*-butoxide alone promotes the reaction without diamine or phenanthroline catalysts.

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SUPPLEMENTARY INFORMATION

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General Information

Potassium *tert*-butoxide (98+%) purchased from Acros Organics was used without further purification. Anhydrous benzene (99.8%, SureSealTM) was purchased from Sigma-Aldrich and used without further purification using standard Schlenk techniques.

1,10-Phenanthroline (anhydrous, 99%) was purchased from Alfa Aesar. Ammonia solution (28% solution in water) was purchased from VWR International. All other commercially available aromatic iodides were used without further purification. Analysis by thin layer chromatography (TLC) was conducted using aluminium-backed plates pre-coated (250 µm) with silica (Merck, TLC silica gel 60 F₂₅₄). TLC plates were visualised using ultraviolet light (254 nm), and then using KMnO₄ solution and heating. Flash chromatography was performed using silica gel (Merck Kieselgel 60) 0.04/0.063mm (230-400 mesh) silica gel.

¹H and ¹³C NMR spectra were recorded on a Bruker AMX500 or AMX600 using CDCl₃ as the deuterated solvent. Coupling constants are reported in Hertz (Hz). ¹³C NMR spectra were recorded at 125 MHz or 150 MHz on either a Bruker AMX500 or AMX600 MHz spectrometer, and are reported in ppm. Mass spectra were measured on a Thermo Finnigan MAT900 XP operating in EI and CI mode. Melting points were measured using Gallenkamp apparatus and are uncorrected.

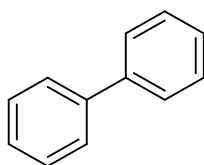
Purification procedure for commercially-available 1,10-phenanthroline

1,10-phenanthroline (anhydrous, 99%) purchased from Alfa Aesar (3.0 g, 16.6 mmol, 1.0 eq.) was dissolved in CH₂Cl₂ (100 mL) and methanesulfonic acid (3.2 g, 33.3 mmol, 2.0 eq.) was added. This mixture was then transferred to a separating funnel and washed with ammonia (28% sol'n in water) (3 x 100 mL) in successive portions. The organic layer was then separated, dried over MgSO₄, filtered and concentrated *in vacuo* to afford a white solid. This solid was consequently recrystallised from chloroform/petrol to afford the product as colourless plates which were filtered and dried in a vacuum oven overnight (2.3 g, 77%); m.p. 115–116 °C (CH₃Cl/PE) (lit. 118 °C from PE).¹ M.p. without purification was 90–99 °C.

General procedure for the arylation of benzene with aryl iodides.

Potassium *tert*-butoxide (101 mg, 0.90 mmol, 4.0 eq), aryl iodide (0.225 mmol, 1.0 eq) and benzene (2.4 mL, 120 eq.) were stirred in a flame-dried, pressure resistant tube for 6 h under an argon atmosphere. The external bath temperature was set at 170 °C. After cooling to rt, the reactions were quenched with 1 M aqueous HCl solution (5 mL), and diluted with Et₂O (5 mL). The organic layer was separated, and the aqueous layer extracted three times with Et₂O (3 x 10 mL). The organic layers were combined, washed with brine (10 mL) and dried over MgSO₄. The organic portion was filtered, concentrated *in vacuo* and purified *via* flash chromatography (100% petroleum ether 40-60 °C).²

Biphenyl 2a



Synthesised according to the general procedure using iodobenzene as the limiting reagent. White solid, 77%; m.p.; 69–70 °C (lit. 69-70 °C)³; ¹H NMR (600 MHz, CDCl₃) δ_H 7.60 (d, *J* = 7.8 Hz, 4H), 7.45 (t, *J* = 7.6 Hz, 4H), 7.35 (t, *J* = 7.1 Hz, 2H); ¹³C NMR (150 MHz, CDCl₃) δ_C 141.3 (C_q), 128.9 (CH), 127.4 (CH), 127.3 (CH); LRMS (EI) 154 (100), 128 (4), 115 (4) 76 (5). Data in agreement with literature values.⁴

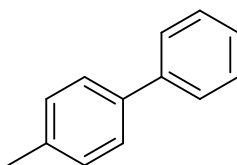
¹ C.W.N. Cumper, D.G. Redford, A.I. Vogel, *J. Chem. Soc.*, **1962**, 1158.

² 3-Phenyl pyridine purified *via* column chromatography using 0-2% MeOH/CH₂Cl₂.

³ K.C. Chan, R.L. Ruang, *J. Chem. Soc.*, **1965**, 2649.

⁴ M.E. Buden, J.F. Guastavino, R.A. Rossi, *Org. Lett.*, **2013**, 1174.

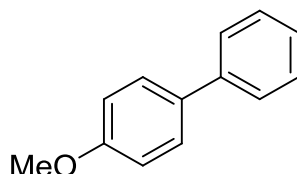
4-Methylbiphenyl 2b



Synthesised according to the general procedure using 4-iodotoluene as the limiting reagent. White solid, 66%; m.p.; 45–46 °C (lit. 46 °C)⁵; ¹H NMR (600 MHz, CDCl₃) δ_H 7.60 (d, *J* = 8.3 Hz, 2H), 7.51 (d, *J* = 8.1 Hz, 2H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.34 (t, *J* = 7.5 Hz, 1H), 7.27 (d, *J* = 7.9 Hz, 2H), 2.41 (s, 3H); ¹³C NMR (150 MHz, CDCl₃)* δ_C 141.3 (C_q), 138.5 (C_q), 137.2 (C_q), 129.6 (CH), 128.8 (CH), 127.1 (CH), 127.1 (CH), 21.2 (CH₃); LRMS (EI) 168 (100), 167 (52), 152 (18), 115 (6), 91 (4). Data in agreement with literature values.⁴

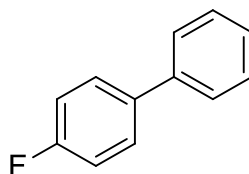
*Two signals are coincident.

4-Methoxybiphenyl 2c



Synthesised according to the general procedure using 4-iodoanisole as the limiting reagent. White solid, 48%; m.p.; 84–85 °C (lit. 84-85 °C)⁶; ¹H NMR (600 MHz, CDCl₃) δ_H 7.57-7.53 (m, 4H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 7.4 Hz, 1H), 6.99 (d, *J* = 8.8 Hz, 2H), 3.86 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ_C 159.2 (C_q), 140.9 (C_q), 133.9 (C_q), 128.8 (CH), 128.3 (CH), 126.9 (CH), 126.8 (CH), 114.3 (CH), 55.5 (CH₃); LRMS (EI) 184 (100), 169 (31), 152 (7), 141 (37), 115 (29). Data in agreement with literature values.⁴

4-Fluorobiphenyl 2d



Synthesised according to the general procedure using 4-fluoroiodobenzene as the limiting reagent. White solid, 64%; m.p.; 69–70 °C (lit. 69 °C)⁷; ¹H NMR (600 MHz, CDCl₃) δ_H 7.56-7.54 (m, 4H), 7.44 (t, *J* = 7.5 Hz, 2H), 7.35 (t, *J* = 7.4 Hz, 1H), 7.13 (t, *J* = 8.6 Hz, 2H); ¹³C NMR (150 MHz, CDCl₃) δ_C 163.4 (C_q), 161.8 (C_q), 140.4 (C_q), 137.4 (d, *J* = 3.2 Hz) (C_q), 128.9 (CH), 128.8 (d, *J* = 8.0 Hz) (CH), 127.4 (CH),

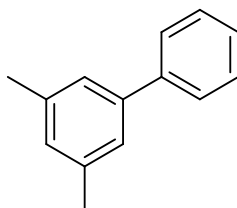
⁵ T. Mino, Y. Shirae, M. Sakamoto, T. Fujita, *J. Org. Chem.*, **2005**, 2191.

⁶ M. Kuriyama, S. Matsuo, O. Onomura, M. Shinozawa, *Org. Lett.*, **2013**, 2716.

⁷ A.L. Allred, L.W. Bush, *Tetrahedron*, **1968**, 6883.

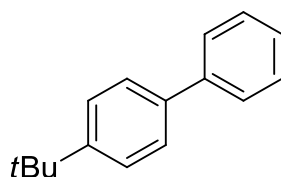
127.1 (CH), 115.7 (d, $J = 21.3$ Hz) (CH); LRMS (EI) 172 (100), 152 (5). Data in agreement with literature values.⁴

3,5-Dimethylbiphenyl 2e



Synthesised according to the general procedure using 5-iodo-*m*-xylene as the limiting reagent. Colourless oil, 48%; ¹H NMR (600 MHz, CDCl₃) δ_{H} 7.58 (dd, $J = 8.3, 1.3$ Hz, 2H), 7.42 (t, $J = 7.5$ Hz, 2H), 7.33 (t, $J = 7.4$ Hz, 1H), 7.21 (br s, 2H), 7.00 (br s, 1H), 2.38 (s, 6H); ¹³C NMR (150 MHz, CDCl₃) δ_{C} 141.6 (C_q), 141.4 (C_q), 138.4 (C_q), 129.0 (CH), 128.7 (CH), 127.3 (CH), 127.2 (CH), 125.2 (CH), 21.5 (CH₃); LRMS (EI) 182 (100), 167 (36), 152 (11), 115 (5). Data in agreement with literature values.⁸

4-*tert*-butylbiphenyl 2f

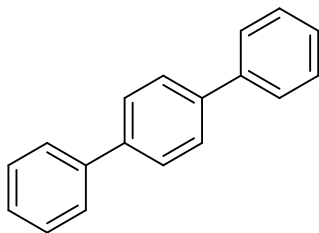


Synthesised according to the general procedure using 1-*tert*-butyl-4-iodobenzene as the limiting reagent. White solid, 30%; m.p.; 48–49 °C (lit. 48-49 °C); ¹H NMR (500 MHz, CDCl₃) δ_{H} 7.60 (d, $J = 7.2$ Hz, 2H), 7.55 (d, $J = 8.4$ Hz, 2H), 7.47 (d, $J = 8.4$ Hz, 2H), 7.43 (t, $J = 7.5$ Hz, 2H), 7.33 (t, $J = 7.3$ Hz, 1H), 1.37 (s, 9H); ¹³C NMR (125 MHz, CDCl₃) δ_{C} 150.3 (C_q), 141.1 (C_q), 138.4 (C_q), 128.7 (CH), 127.1 (CH), 127.0 (CH), 126.8 (CH), 125.8 (CH), 34.6 (C_q), 31.4 (CH₃); LRMS (EI) 210 (36), 195 (100), 178 (11), 167 (19), 152 (9), 115 (4). Data in agreement with literature values.⁹

⁸ X. Li, X-Y. Yan, H-H. Chang, L-C. Wang, Y. Zhang, W-W. Chen, Y-W. Li, W-L. Wei, *Org. Biomol. Chem.* **2012**, *10*, 495.

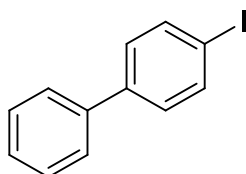
⁹ (a) M.J. Inglesias, A. Prieto, M.C. Nicasio, *Org. Lett.*, **2012**, 4318. (b) X-H. Fan, L-M. Yang, *Eur. J. Org. Chem.*, **2010**, 2457.

***p*-Terphenyl 2g**



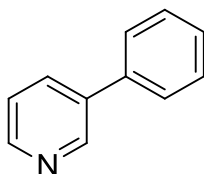
Synthesised according to the general procedure using 1,4-diiodobenzene as the limiting reagent. White solid, 21%; m.p.; 212–213 °C (lit. 212-213 °C)⁵; ¹H NMR (500 MHz, CDCl₃) δ_H 7.68 (s, 4H), 7.65 (d, *J* = 7.1 Hz, 4H), 7.46 (t, *J* = 7.4 Hz, 4H), 7.36 (t, *J* = 7.4 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ_C 140.8 (C_q), 140.2 (C_q), 128.9 (CH), 127.6 (CH), 127.4 (CH), 127.1 (CH); LRMS (EI) 230 (100), 195 (15), 152 (13), 115 (9). Data in agreement with literature values.⁴

4-Iodobiphenyl 2h



Synthesised according to the general procedure using 1,4-diiodobenzene as the limiting reagent. White solid, 30%; m.p.; 107–108 °C (lit. 109-110 °C)¹⁰; ¹H NMR (500 MHz, CDCl₃) δ_H 7.77 (m, 2H), 7.55 (m, 2H), 7.44 (m, 2H), 7.38-7.32 (m, 3H); ¹³C NMR (125 MHz, CDCl₃) δ_C 140.8 (C_q), 140.1 (C_q), 137.9 (CH), 129.1 (CH), 129.0 (CH), 127.8 (CH), 127.0 (CH), 93.1 (C_q); LRMS (EI) 280 (100), 152 (55). Data in agreement with literature values.¹¹

3-Phenylpyridine 2i

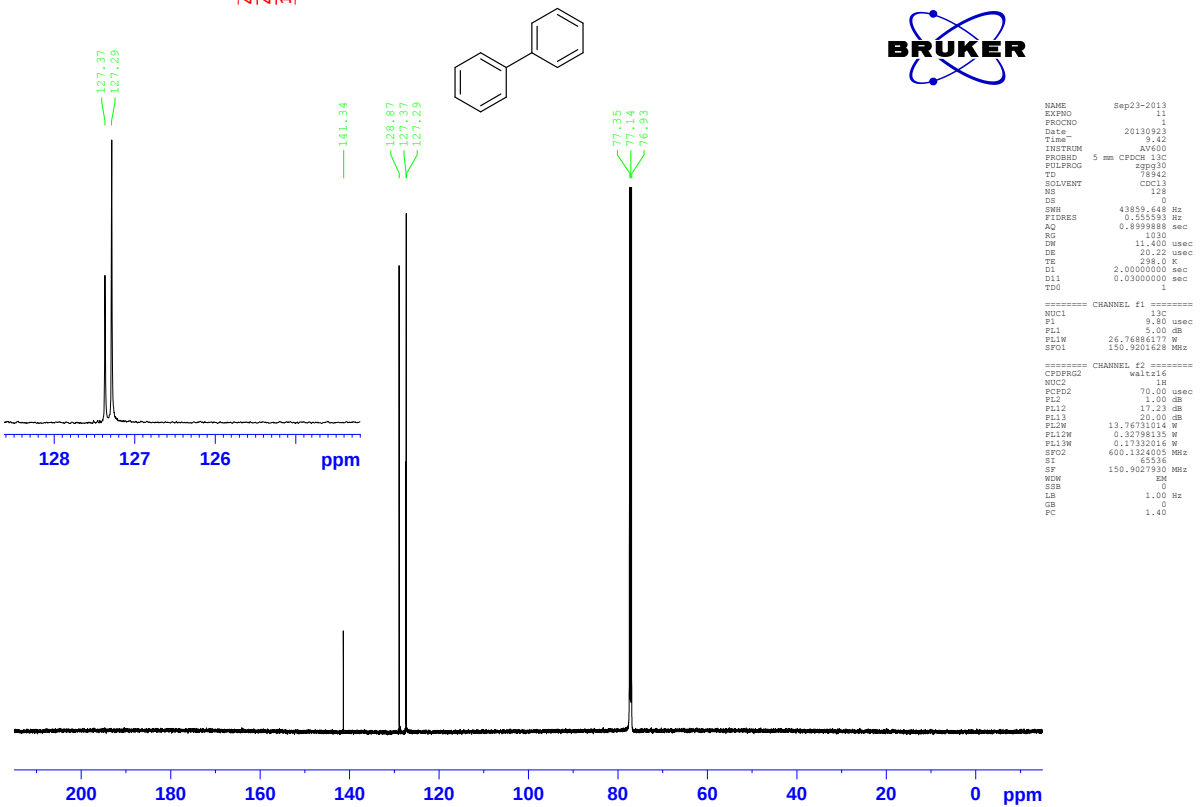
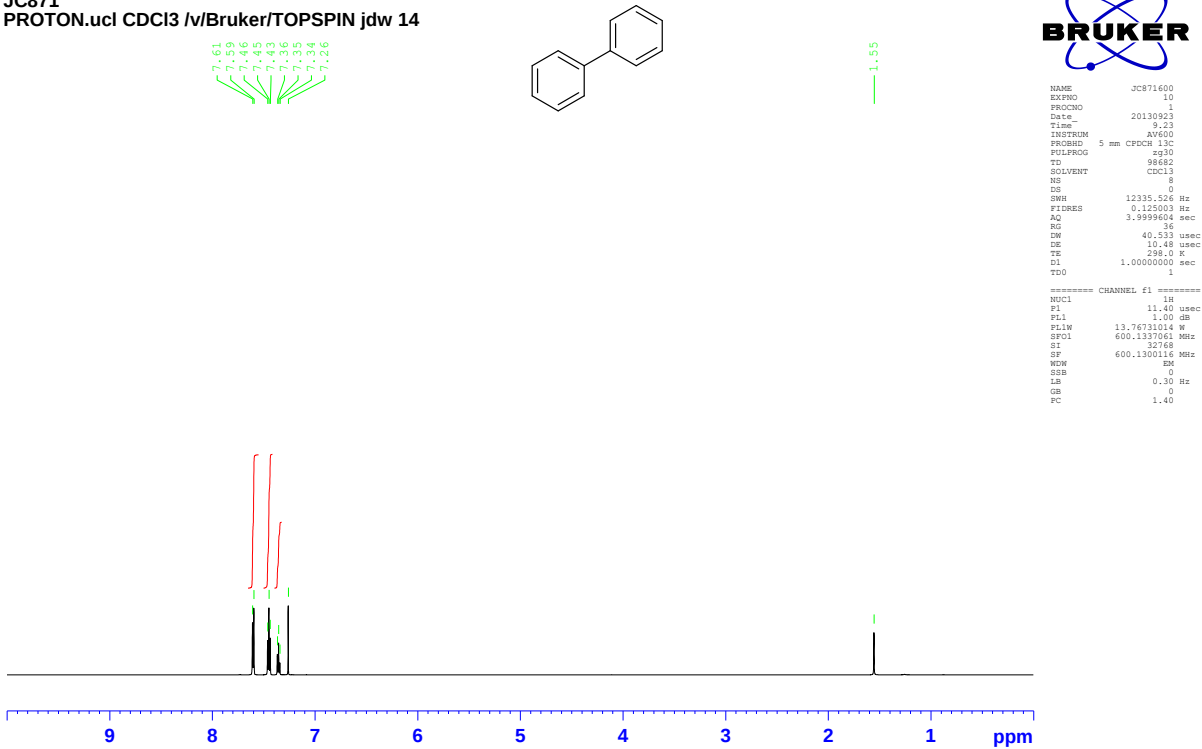


Synthesised according to the general procedure using 3-iodopyridine as the limiting reagent. Colourless oil, 37%; NMR (600 MHz, CDCl₃) δ_H 8.87 (br s, 1H), 8.61 (br s, 1H), 7.89 (dt, *J* = 7.9, 1.7 Hz, 1H), 7.59 (d, *J* = 7.1 Hz, 2H), 7.49 (t, *J* = 7.5 Hz, 2H), 7.43-7.37 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ_C 148.5 (CH), 148.3 (CH), 137.9 (C_q), 136.9 (C_q), 134.6 (CH), 129.2 (CH), 128.2 (CH), 127.3 (CH), 123.8 (CH); LRMS (EI) 155 (100), 127 (8), 115 (3). Data in agreement with literature values.⁴

¹⁰ Y. Qin, W. Wei, M. Luo, *Synlett*, **2007**, 2410

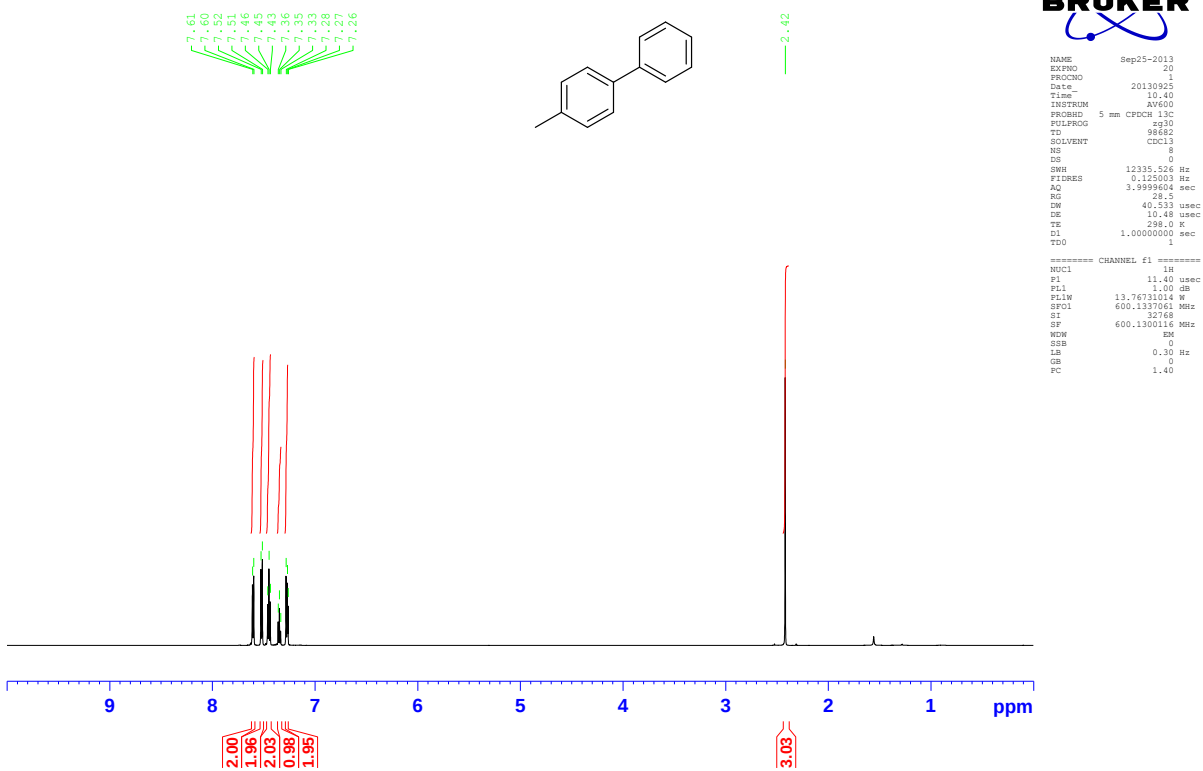
¹¹ K. Kulbitski, G. Nisnevich, M. Gandelman, *Adv. Synth. Catal.*, **2011**, 353, 1438.

Biphenyl 2a
JC871
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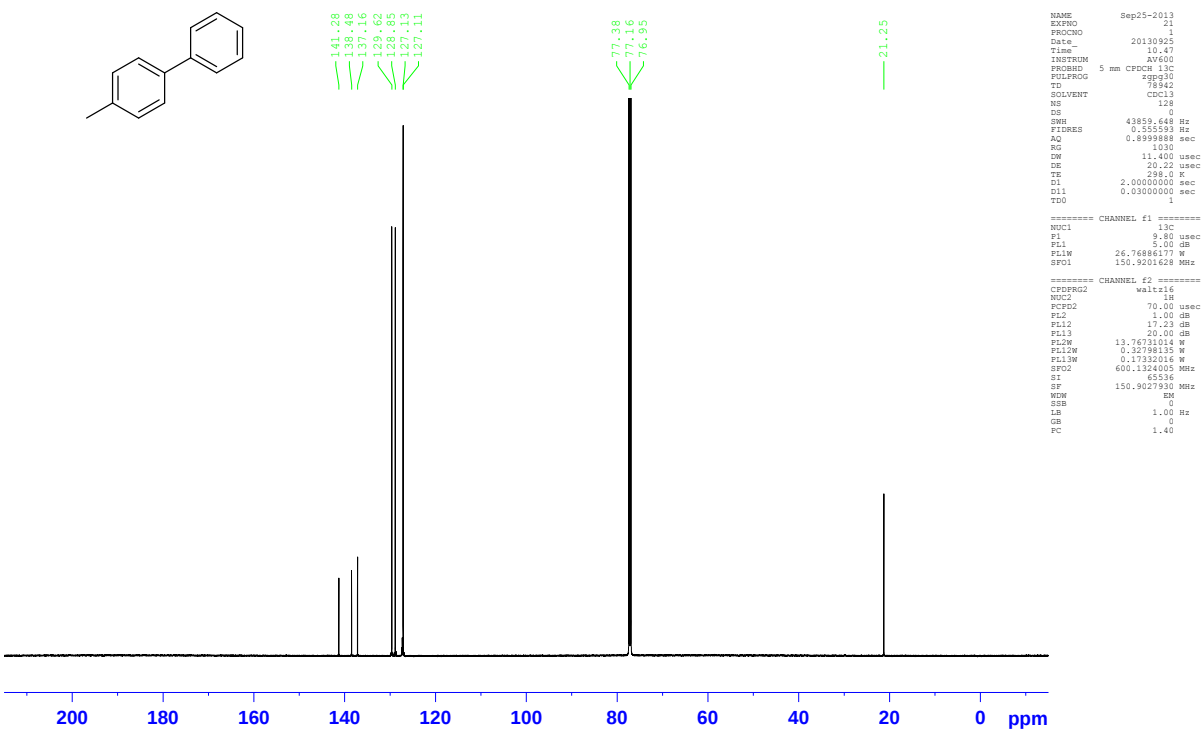


4-Methylbiphenyl 2b

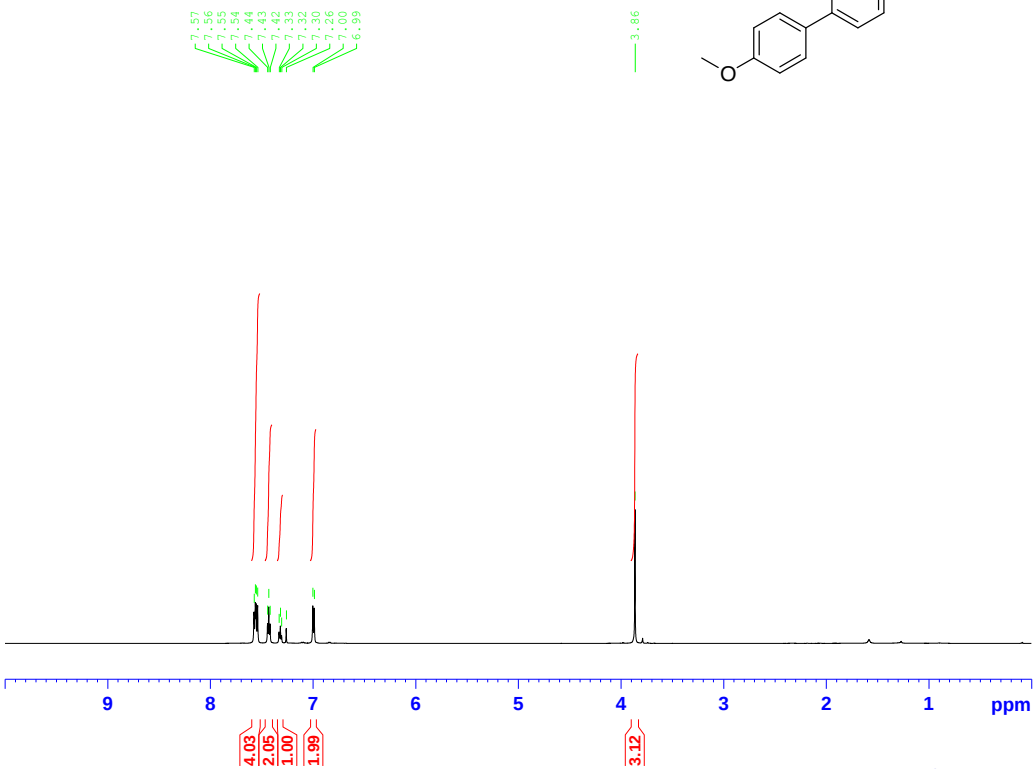
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JC819
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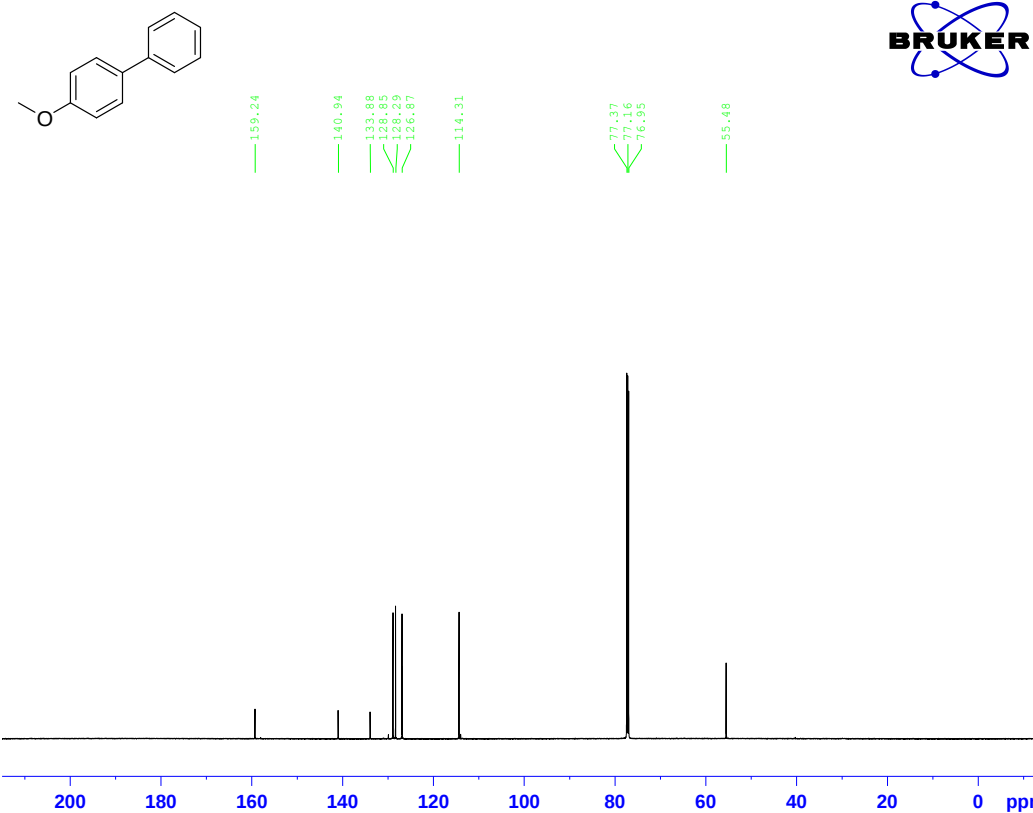


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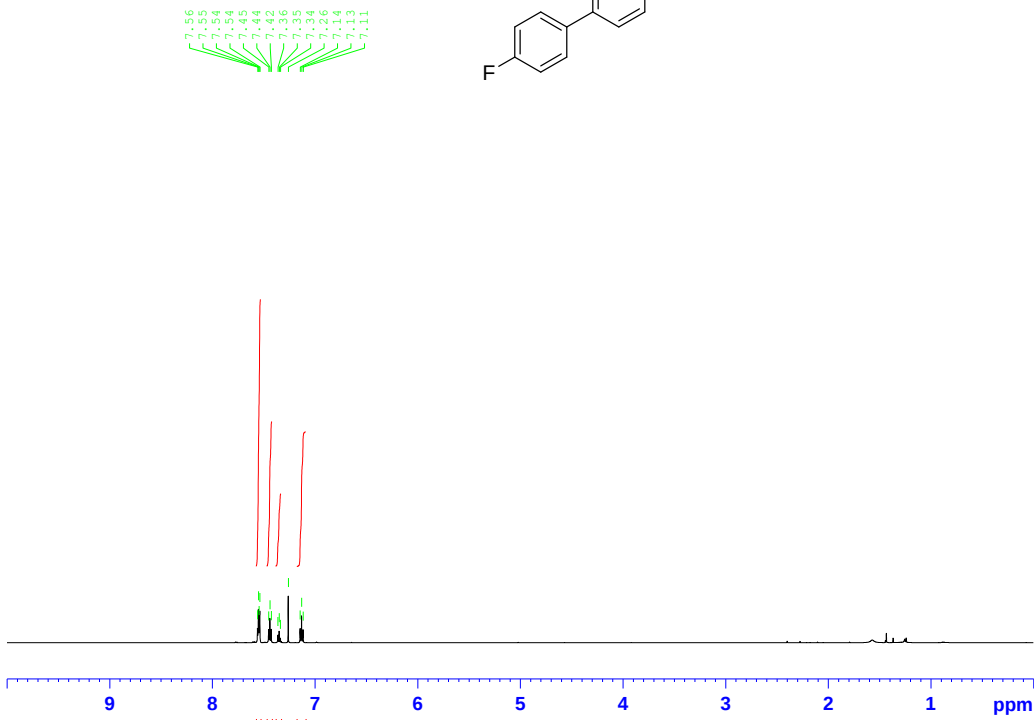
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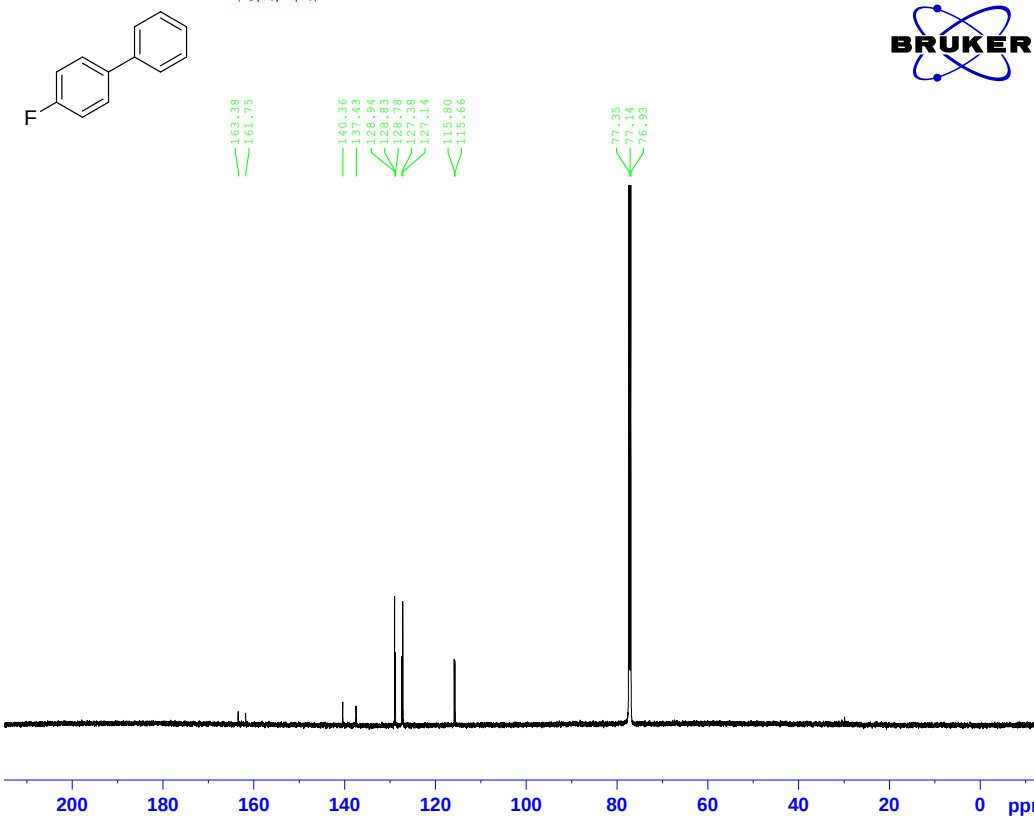
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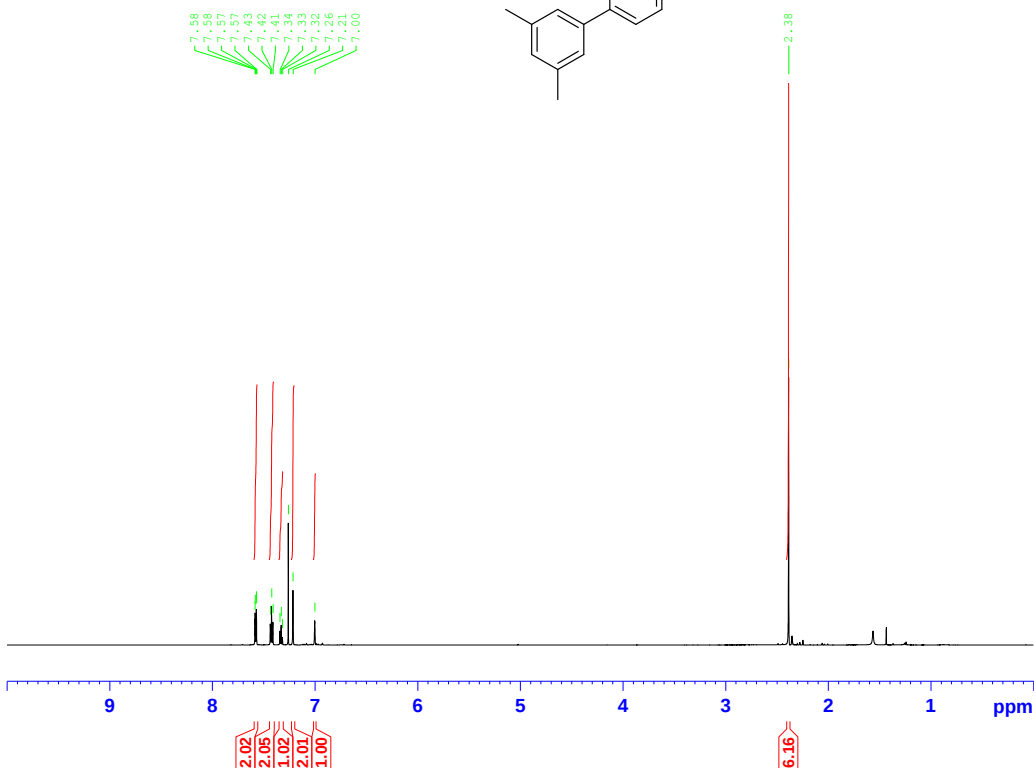
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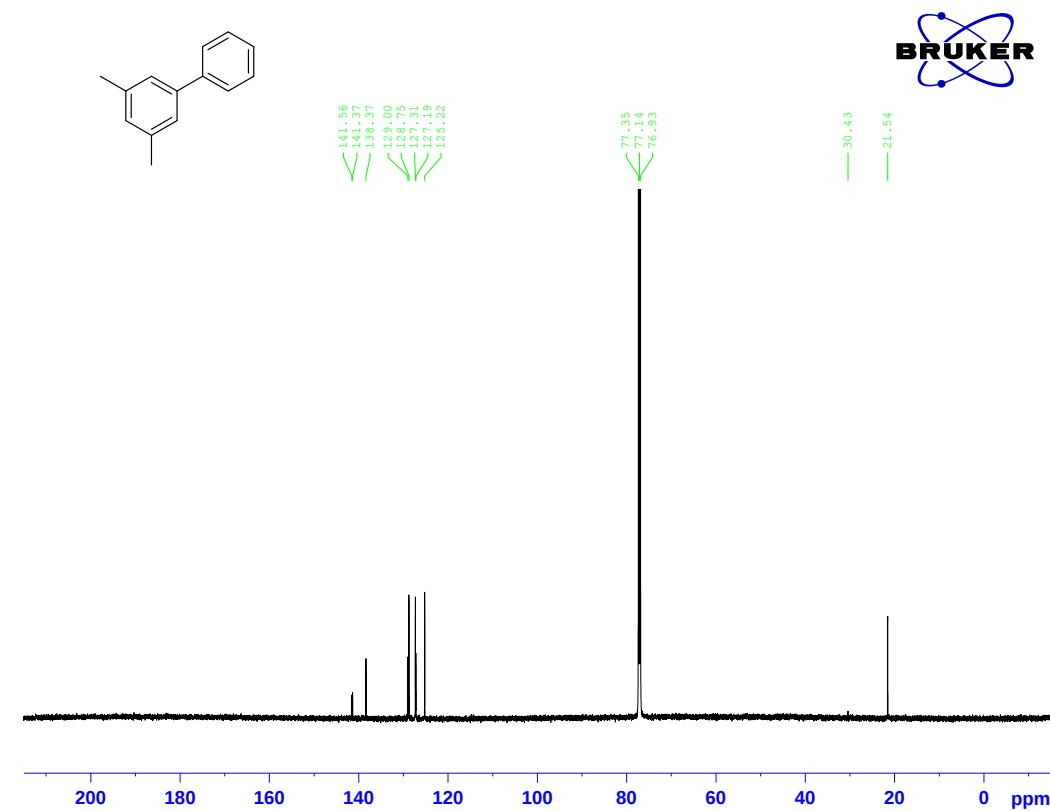
3,5-Dimethylbiphenyl 2e

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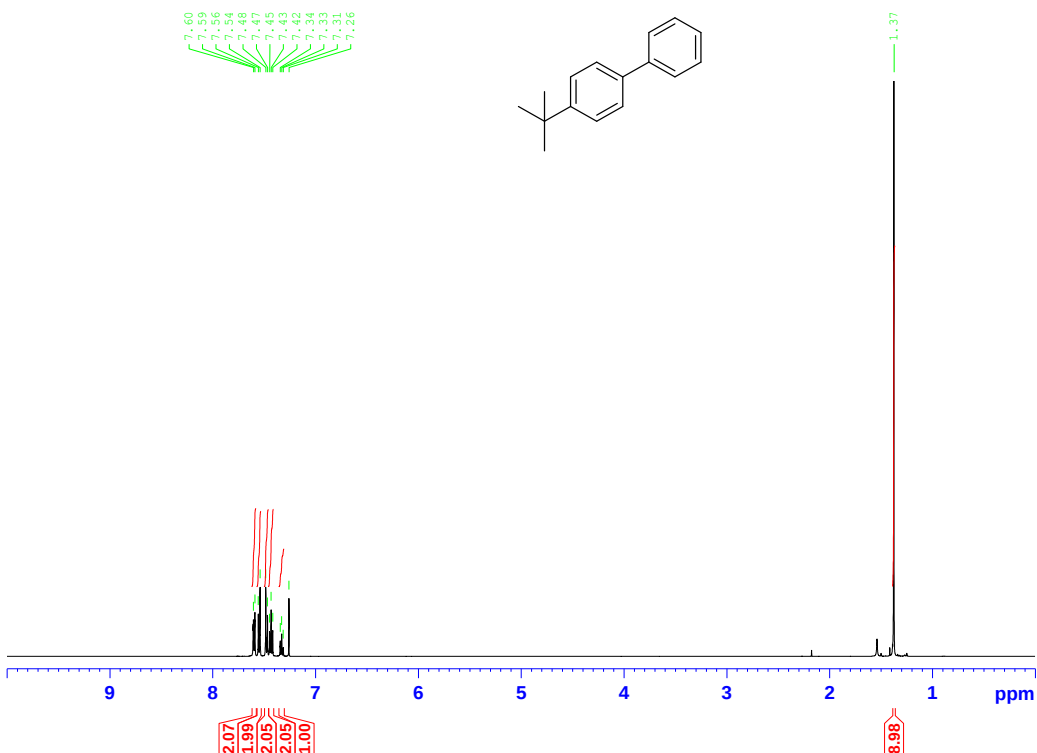
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4-tert-butylbiphenyl 2f

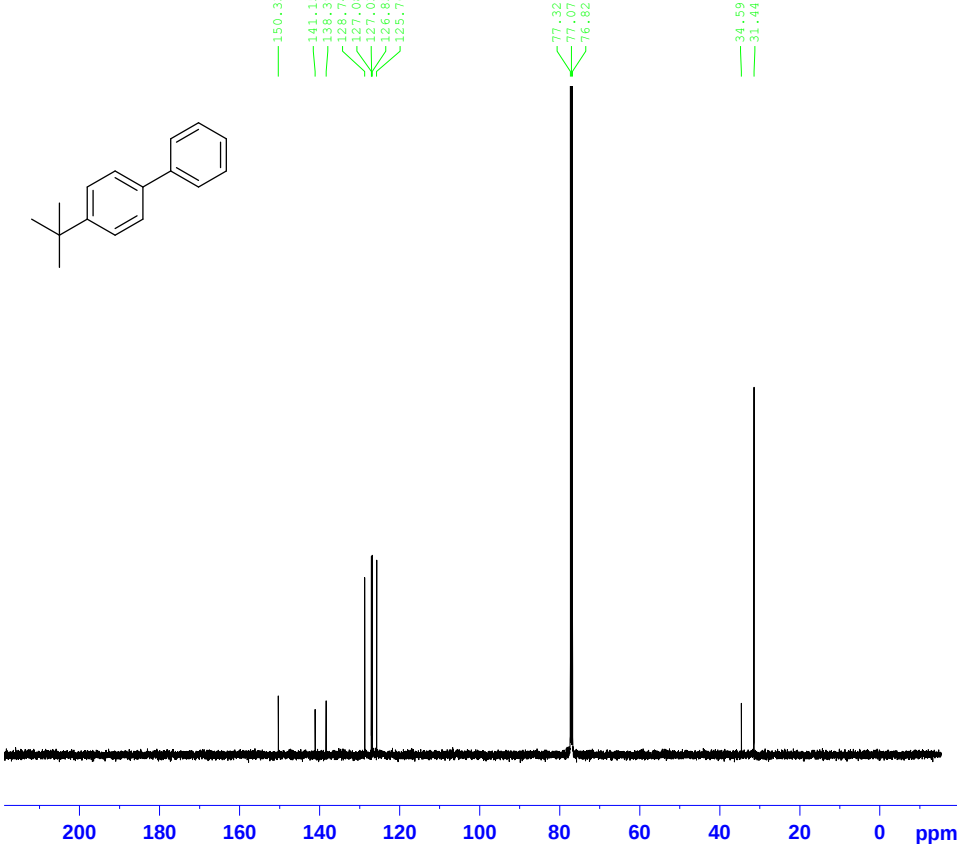
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D12 0.00002000 sec

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JC869
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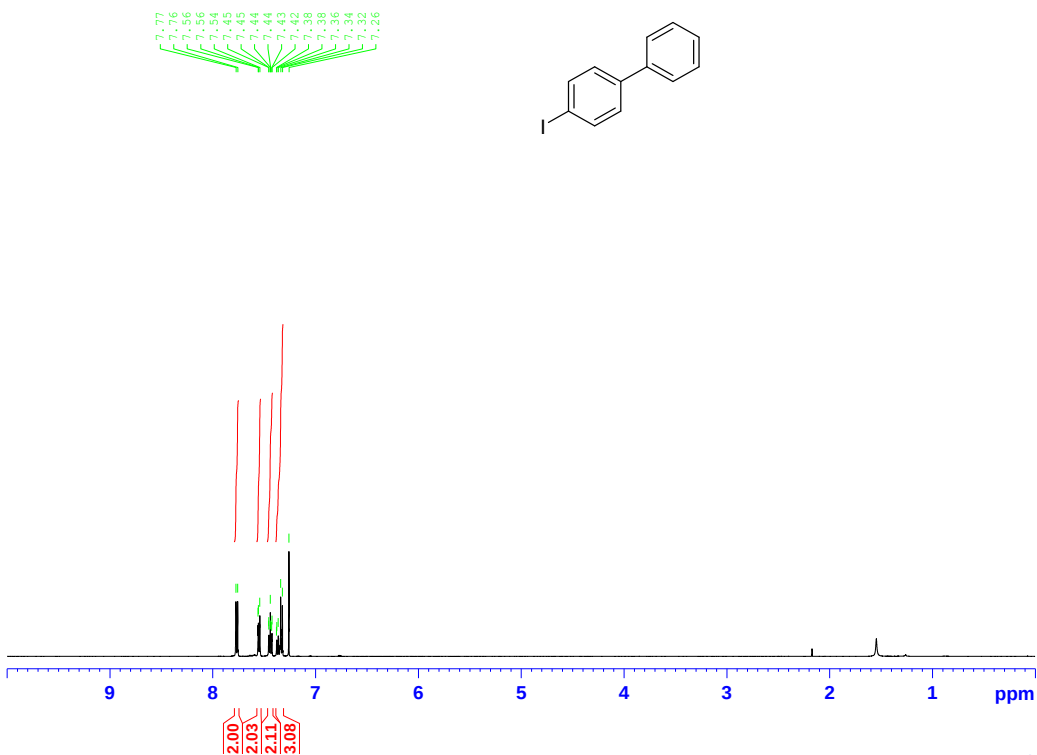
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LB 1.00 Hz
GB 0
PC 1.40

4-Iodobiphenyl 2h

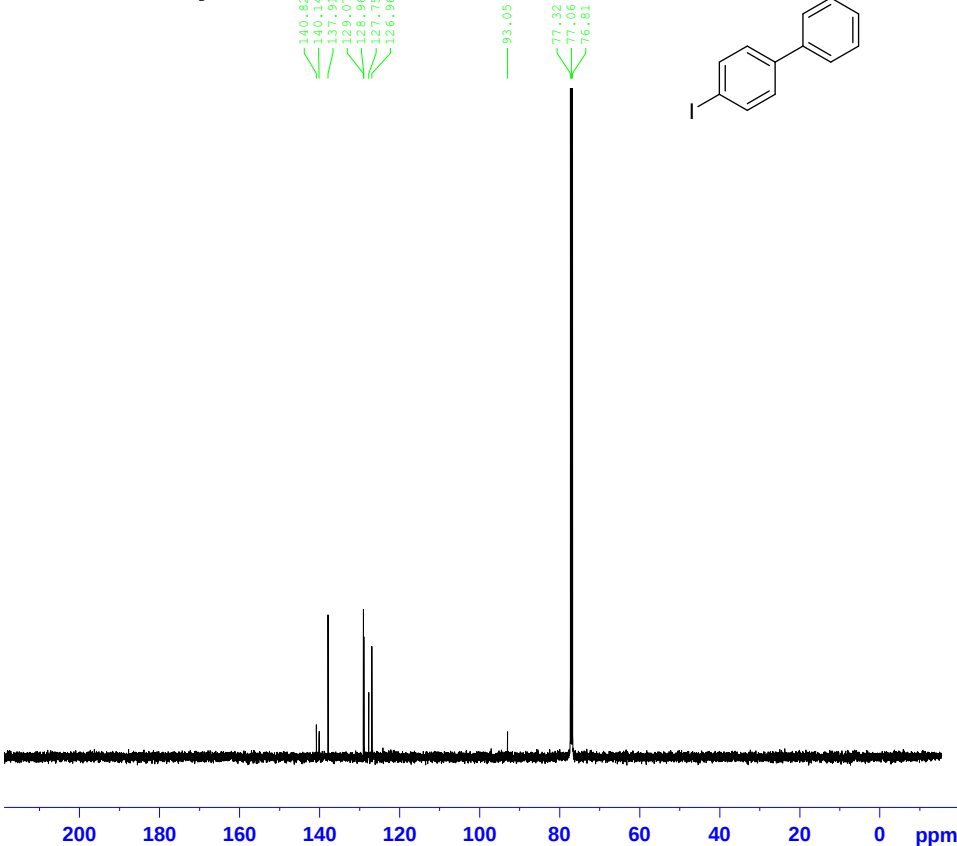
JC871
PROTON.ucl CDC13 v2 jdw 36



NAME JC871
EXPNO 1
PROCNO 1
Date_ 20130920
Time 3.45
INSTRUM drx500
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
F2 32768
SOLVENT CDCl3
NS 512
DS 4
SWH 10330.578 Hz
FIDRES 0.127632 Hz
AQ 0.170247 sec
RG 124
DW 48.460 usec
DE 6.20 usec
TE 300.2 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 19.40 usec
PL1 0.00 dB
SFO1 500.133088 MHz
SI 32768
SF 500.1330148 MHz
WDW EM
LB 0.30 Hz
GB 0
PC 1.00

JC871
C13CPD CDC13 v2 jdw 36

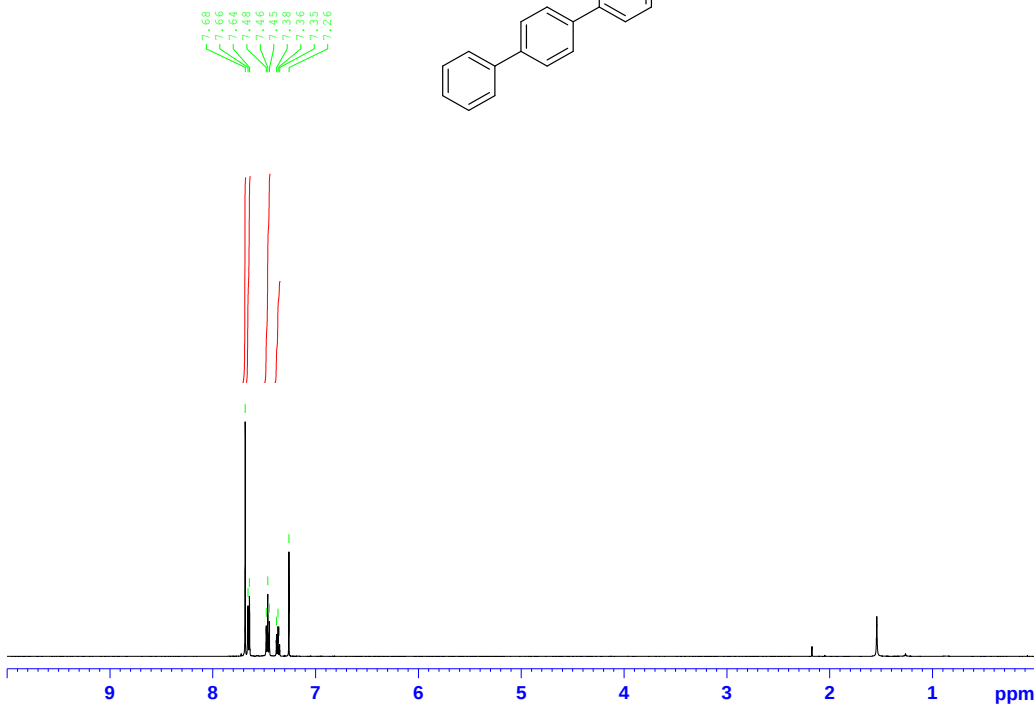
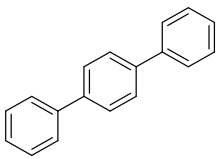


NAME JC871
EXPNO 11
PROCNO 1
Date_ 20130920
Time 4.12
INSTRUM drx500
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
F2 32768
SOLVENT CDCl3
NS 512
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420883 sec
RG 9195.2
DW 15.900 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 6.40 usec
PL1 0.00 dB
SFO1 125.7715719 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577814 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

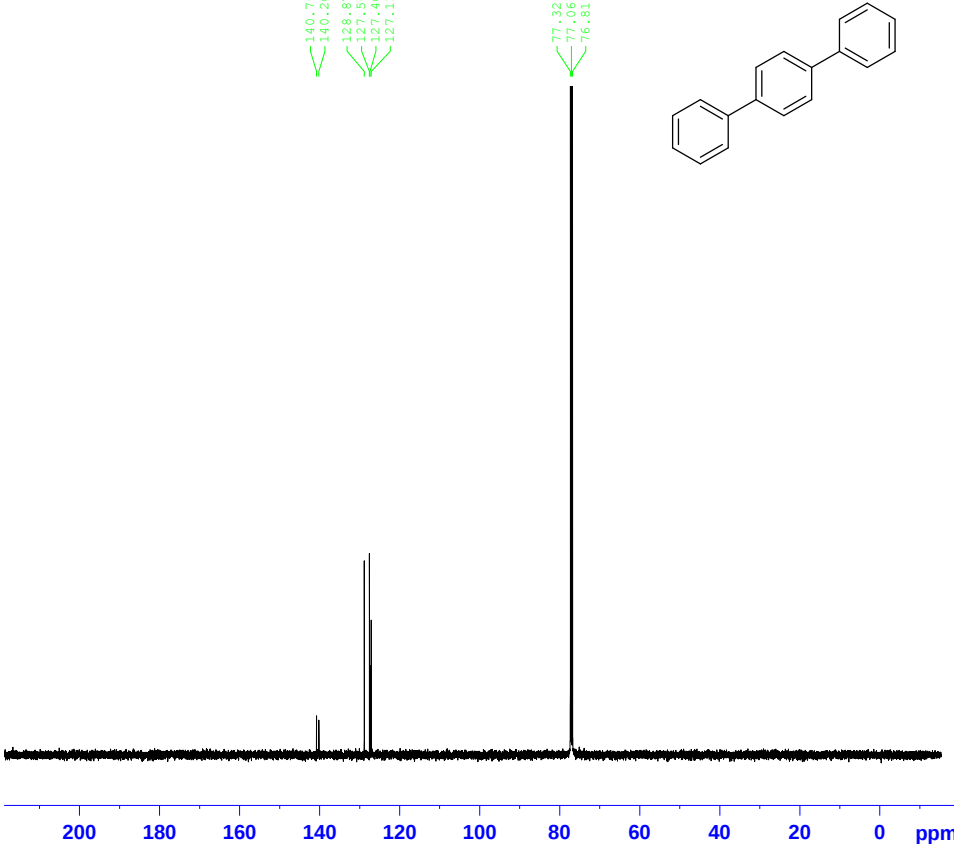
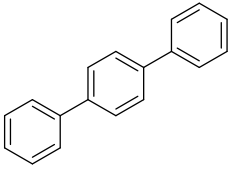
p-Terphenyl 2g
JC8672
PROTON.uc1 CDC13 v2 jdw 39



```
NAME JC8672
EXPNO 4
PROCNO 1
Date_ 20130920
Time 5.23
INSTRUM drx500
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 10330.578 Hz
FIDRES 0.127632 Hz
AQ 0.170247 sec
RG 8192
DW 15.900 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 19.40 usec
PL1 0.00 dB
SFO1 500.133088 MHz
SI 32768
SF 500.1330145 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
```

JC8672
C13CPD CDC13 v2 jdw 39

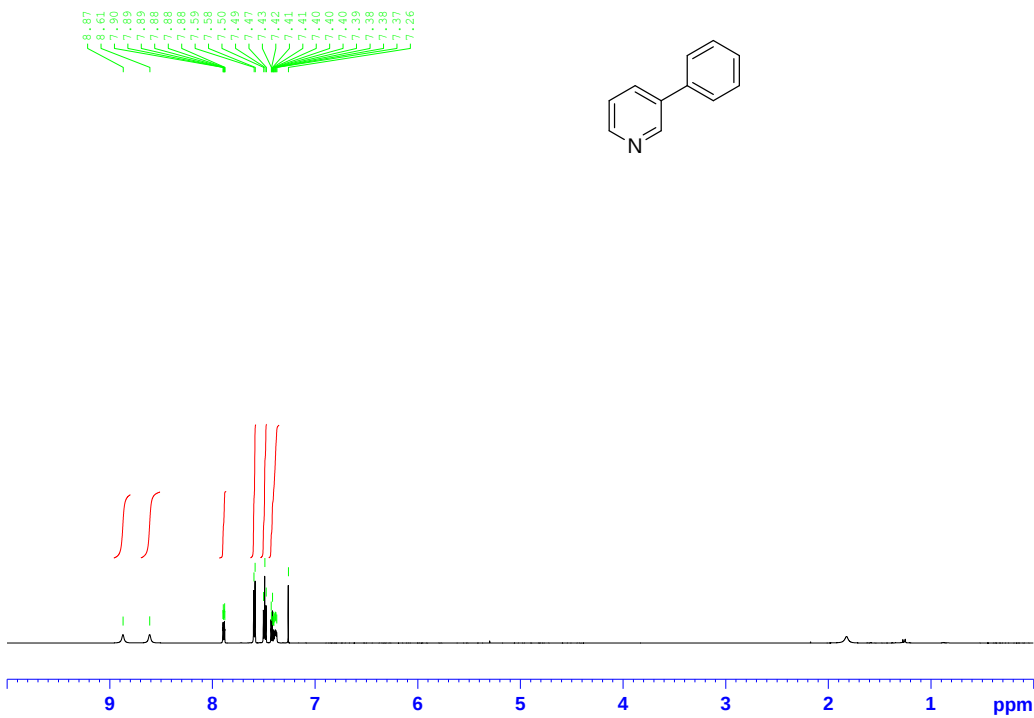


```
NAME JC8672
EXPNO 21
PROCNO 1
Date_ 20130920
Time 5.50
INSTRUM drx500
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420883 sec
RG 8192
DW 15.900 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 6.40 usec
PL1 0.00 dB
SFO1 125.7715719 MHz

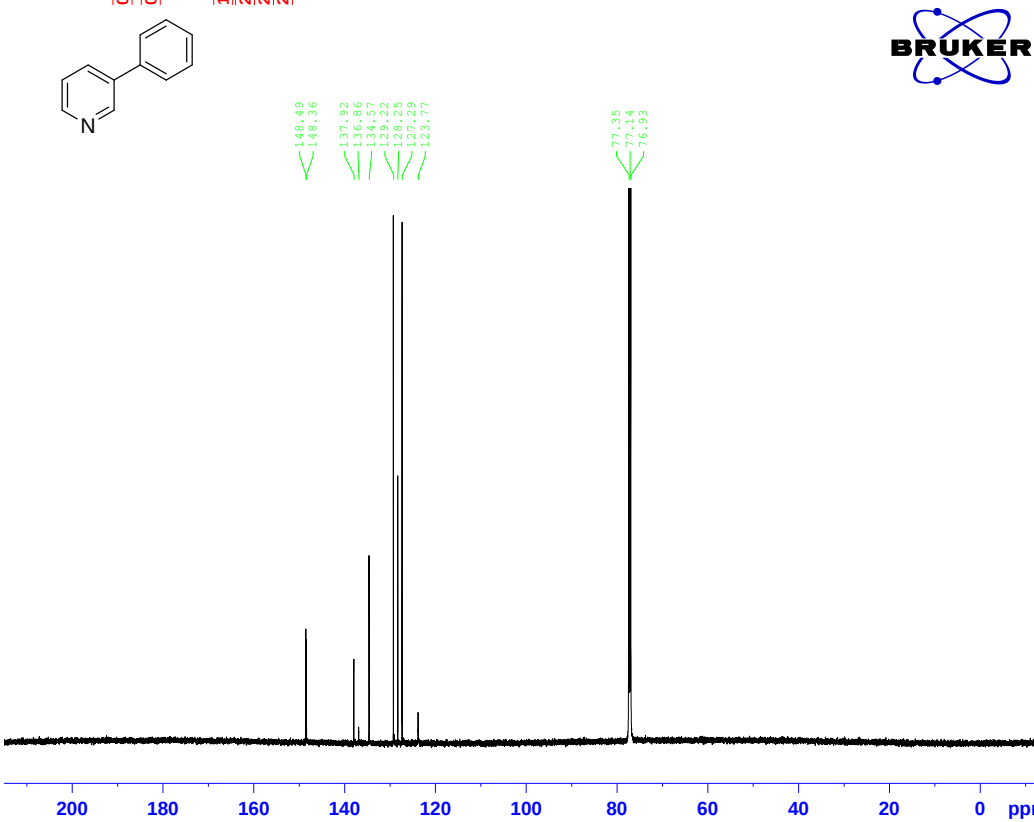
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577814 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
```

3-Phenylpyridine 2i
JC903wo
PROTON.ucl CDCI3 /vBruker/TOPSPIN jdw 11



NAME Oct09-2013
EXPNO 20
PROCNO 1
Date 20131009
Time 10.32
INSTRUM AV600
PROBHD 5 mm CPDCH 13C
PULPROG zgpg30
TD 98682
SOLVENT CDCl3
NS 8
DS 0
SWH 12335.526 Hz
FIDRES 0.115003 Hz
AQ 1.999804 sec
RG 40.3
DW 40.533 usec
DE 10.48 usec
TE 298.0 K
D1 1.00000000 sec
TDO 1

***** CHANNEL f1 *****
NUC1 1H
P1 11.40 usec
PL1 1.00 dB
PL1W 13.76731014 W
SFO1 600.1337061 MHz
SI 32768
SF 600.1300116 MHz
WDM DM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40



NAME Oct09-2013
EXPNO 21
PROCNO 1
Date 20131009
Time 10.45
INSTRUM AV600
PROBHD 5 mm CPDCH 13C
PULPROG zgpg30
TD 98682
SOLVENT CDCl3
NS 128
DS 0
SWH 43859.648 Hz
FIDRES 0.555593 Hz
AQ 0.8999888 sec
RG 1030
DW 11.400 usec
DE 20.22 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

***** CHANNEL f1 *****
NUC1 13C
P1 9.80 usec
PL1 5.00 dB
PL1W 26.76886177 W
SFO1 150.9201428 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 1.00 dB
PL12 17.23 dB
PL13 20.00 dB
PL12W 13.76731014 W
PL12W 0.32798135 W
PL13W 0.1732016 W
SFO2 600.1324005 MHz
SI 65536
SF 150.9027930 MHz
WDM DM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40