Supplementary Material

Synthesis of ethynylated biaryls and asymmetric diethynylated benzene via sequential Sonogashira and Suzuki couplings in water

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$^1$H NMR of compound 4
$^1$H NMR of compound 5
$^1$H NMR of compound 7
$^1$H NMR of compound 8
$^{13}$C NMR of compound 8
$^1$H NMR of compound 9
$^{13}$C NMR of compound 9
$^1$H NMR of compound 10
$^{13}\text{C}$ NMR of compound 10
$^1$H NMR of compound 11
$^{13}$C NMR of compound 11
$^{1}$H NMR of compound 12
$^1$H NMR of compound 13
$^{13}$C NMR of compound 13
$^1$H NMR of compound 14
$^{13}$C NMR of compound 14
$^1$H NMR of compound 15
$^{13}$C NMR of compound 15
$^1$H NMR of compound 16
$^{13}$C NMR of compound 16

Pulse Sequence: s2pu1
Solvent: CDCl3
Temp. 29.8°C / 293.1 K
Field: proton=7.0102 MEGALINES

Relax, delay 1,800 sec
Pulse 75.0 degrees
Acq. time 4.025 sec
Width 0.088 Hz
NMR: 1.00000 ppm

DATA PROCESSING
Line broadening 0.3 Hz
FT 1272 85538
Total time 5 min, 11 sec
$^1$H NMR of compound 17
$^1$H NMR of compound 18

Pulse Sequence: d2pol
Solvent: CDCl3
Temp. 30.4 °C / 303.1 K
file: ProceduresClarity-M45-CDCL3-H1
Mercury-300B "NMR300B"
Relax. delay 1.000 sec
Pulse 75.9 degrees
Acq. time 0.095 sec
Width 6094.0 Hz
32 repetitions
SYSTEM 8.1 300.6730±25 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Total time 3 min, 5 sec
$^1$H NMR of compound 19

MohamedAbdelMaty-CDC13-C13

Archive directory: export/home/vmerl/vmersys/data
Sample directory: D05mm_test_12Mar2014-21:39:40
File: PROTON

Pulse Sequence: zspul
Solvent: CDC13
Temp. 30.6 C / 333.1 K
Mercury-300B5 "AMES88"

Pulse 48.7 degrees
Acq. time 1.797 sec
Width 1878.7 Hz
864 repetitions
OBSERVE C13, 75.4520062 MHz
DECOUPLE H, 390.068856 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 3 hr. 6 min. 43 sec
Date: Nov 30 2014

180 160 140 120 100 80 60 40 20
ppm

132.518 127.647 123.805 114.572
131.990 126.654 114.669 107.087
133.245 128.233 124.195 117.149
132.665 131.018 124.599 113.163
78.748 69.828 67.716 57.067
80.306 79.178 78.328 76.542
80.306 79.178 78.328 76.542

\textbf{13C NMR of compound 19}

- **Steady INH OBSERVE**
- **Pulse Sequence:** 6zpu4
- **Solvent:** CDCl3
- **Temp. 30.0 \textdegree C / 303.1 K**
- **Filter:** NONAMAGNETIC HOST-H-CDCl3-H
- **Mercury-300BB INNM398**

- Relax. delay 1.000 sec
- Pulse 75.9 degrees
- Acq. time 4.885 sec
- Width 6000.0 Hz
- 12 repetitions
- **OBSERVE H1, 300.6673627 MHz**
- **DATA PROCESSING**
  - Line broadening 0.9 Hz
  - FT size 65536
- **Total time 3 min, 5 sec**
1H NMR of compound 20
$^{13}$C NMR of compound 20