Functionalization of the Methylene Groups of *p*-tert-Butylcalix[4]arene: S-C, N-C and C-C**Bond Formation**

Samah Simaan, Kasim Agbaria and Silvio E. Biali*

Supporting Information

Page S2. ¹H NMR spectrum of **10** (400 MH, CDCl₃, rt)

Page S3. ¹H NMR spectrum of **11** (400 MH, CDCl₃, rt)

Page S4. ¹H NMR spectrum of **12** (400 MH, CDCl₃, rt)

Page S5. ¹H NMR spectrum of **13** (400 MH, CDCl₃, rt)

Page S6. ¹H NMR spectrum of **14** (400 MH, CDCl₃, rt)

Page S7. ¹H NMR spectrum of **15** (400 MH, CDCl₃, rt)

Page S8. ¹H NMR spectrum of **16** (400 MH, CDCl₃, rt)

Page S9. ¹H NMR spectrum of **17** (400 MH, CDCl₃, 230 K)

Page S10. ¹H NMR spectrum of **18** (400 MH, DMSO-d₆, 430 K)

Page S11. ¹H NMR spectrum of **21b** (400 MH, CDCl₃, rt)

Page S12. ¹H NMR spectrum of **22** (400 MH, CDCl₂CDCl₂, rt)

Page S13. ¹H NMR spectrum of **23** (400 MH, CDCl₃, rt)

Page S14. ¹H NMR spectrum of **24** (400 MH, CDCl₃, rt)

Page S15. ¹H NMR spectrum of **25** (400 MH, CDCl₃, rt)

Current Data Parameters

NAME samah-ANPh
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20010722
Time 14.39
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 8
DS 0
SWH 8250.825 Hz
FIDRES 0.503590 Hz
AQ 0.9929204 sec
RG 362
DW 60.600 usec
DE 10.00 usec
TE 300.0 K
D1 2.0000000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 5.59 usec
PL1 -6.00 dB
SF01 400.1320007 MHz

F2 - Processing parameters

SI 16384
SF 400.1300097 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
FIP 8.275 ppm
F1 3311.06 Hz
F2 -0.523 ppm
F2 -209.29 Hz
PPMCM 0.43990 ppm/cm
HZCM 176.01759 Hz/cm

11

S

[Integral]

Current Data Parameters

NAME fgr
EXPNO 33
PROCNO 1

F2 - Acquisition Parameters

Date_ 20010314
Time 22.08
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 32
DS 0
SMH 3894.081 Hz
FIDRES 0.237676 Hz
AQ 2.1037557 sec
RG 2048
DM 128.400 usec
DE 10.00 usec
TE 300.0 K
D1 2.0000000 sec

===== CHANNEL f1 =====

NUC1 ¹H
P1 5.59 usec
PL1 -6.00 dB
SF01 400.1315266 MHz

F2 - Processing parameters

SI 16384
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 7.865 ppm
F1 3147.13 Hz
F2P 0.551 ppm
F2 220.52 Hz
PPMCM 0.36571 ppm/cm
HZCM 146.33035 Hz/cm

1H-NMR

Current Data Parameters
 NAME SMAF-132
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20010807
 Time 18.52
 INSTRUM cir400
 PROBED 5 mm Multinu
 PULPROG zg30
 TD 16384
 SOLVENT CDCl3
 NS 1
 DS 0
 SWH 8250.825 Hz
 FIDRES 0.503590 Hz
 AQ 0.9929204 sec
 RG 362
 DW 60.600 usec
 DE 10.00 usec
 TE 300.0 K
 D1 2.00000000 sec

===== CHANNEL f1 =====

NUC1 1H
 P1 5.59 usec
 PL1 -6.00 dB
 SFO1 400.1320307 MHz

F2 - Processing parameters

SI 16384
 SF 400.13300155 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 FIP 8.215 ppm
 F1 3286.90 Hz
 F2P -0.620 ppm
 F2 -248.05 Hz
 PPMCM 0.44172 ppm/cm
 HZCM -76.74733 Hz/cm

1.55581
 1.31758
 1.29499
 1.28230
 1.25575
 1.22176
 1.12626
 1.11494
 1.03921
 0.00064

3.71368
 3.67513
 3.08384
 3.04548

7.54668
 7.23842
 7.21552
 7.13481
 6.99387
 6.97279
 6.44847
 5.92537

13

8.9927
 9.0015
 8.8588

1.0000
 1.0066

1.0214
 1.0039

3.0358
 1.0755
 2.6038
 1.0493

Integral

1H-NMR

¹H-NMR

NAME samah-SCH3-220
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20011016
Time 14.50
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 8
DS 0
SWH 8250.825 Hz
FIDRES 0.503590 Hz
AQ 0.9929204 sec
RG 20.2
DM 60.600 usec
DE 10.00 usec
TE 300.0 K
D1 2.00000000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 5.59 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters

SI 16384
SF 400.1300102 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
ID NMR plot parameters
CX 20.00 cm
FIP 9.031 ppm
F1 3613.75 Hz
F2P -0.330 ppm
F2 -131.91 Hz
PPMCM 0.46805 ppm/cm
HZCM 187.28275 Hz/cm

1.06496
1.22352
1.32088
1.37867

2.04243
2.19351

3.54162
3.57641

4.25231
4.28686

4.99988

5.72295

7.02306
7.06980
7.19927
7.26058
7.52722

37.272

3.133
3.299

2.115

2.062

0.950

1.000

3.966
1.920

2.065

Integral

Current Data Parameters

NAME samah-NH13C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20011114
Time 18.31
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 8
DS 0
SWH 8250.825 Hz
FIDRES 0.503590 Hz
AQ 0.9929204 sec
RG 64
DW 60.600 usec
DE 10.00 usec
TE 300.0 K
D1 2.0000000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 5.59 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters

SI 16384
SF 400.1300225 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
FIP 12.001 ppm
F1 4802.05 Hz
F2P -1.163 ppm
F2 -465.22 Hz
PPMCM 0.65819 ppm/cm
HZCM 263.36337 Hz/cm

Current Data Parameters
 NAME samah-3sch3
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20020313
 Time 10.50
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 16384
 SOLVENT CDC13
 NS 8
 DS 0
 SWH 8250.825 Hz
 FIDRES -0.503590 Hz
 AQ 0.9929204 sec
 RG 256
 DW 60.600 usec
 DE 10.00 usec
 TE 300.0 K
 D1 2.00000000 sec

===== CHANNEL f1 =====

NUC1 ¹H
 P1 5.59 usec
 PL1 -6.00 dB
 SF01 400.1320007 MHz

F2 - Processing parameters

SI 16384
 SF 400.1300092 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 F1P 8.821 ppm
 F1 3529.72 Hz
 F2P -1.052 ppm
 F2 -420.89 Hz
 PPMCM 0.49367 ppm/cm
 HZCM 197.53032 Hz/cm

Current Data Parameters
 NAME samah-calSCH3d
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20020106
 Time 14.10
 INSTRUM drx400
 PROBD 5 mm Multinu
 PULPROG zg30
 TD 16384
 SOLVENT CDCl₃
 NS 16
 DS 0
 SWH 8250.825 Hz
 FIDRES 0.503590 Hz
 AQ 0.9929204 sec
 RG 181
 DW 60.600 usec
 DE 10.00 usec
 TE 300.0 K
 D1 2.0000000 sec

===== CHANNEL f1 =====

NUC1 1H
 P1 5.59 usec
 PL1 -6.00 dB
 SFO1 400.132007 MHz

F2 - Processing parameters

SI 16384
 SF 400.1300172 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 F1P 11.953 ppm
 F1 4782.83 Hz
 F2P -0.812 ppm
 F2 -324.83 Hz
 PPMCM 0.63825 ppm/cm
 HZCM 255.38269 Hz/cm

1.1475
 1.1966
 1.2230
 1.2298
 1.2325
 1.6890
 1.9744

3.5097
 3.5449
 4.1560
 4.1909

5.5949
 5.9501

7.0351
 7.0405
 7.4954
 7.4992

10.2946

17.776

2.866

1.006

1.000

0.922

1.920

1.830

1.848

Integral

ppm

Current Data Parameters
NAME gf1
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010617
Time 21.08
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4045.307 Hz
FIDRES 0.246906 Hz
AQ 2.0251124 sec
RG 406.4
DM 123.600 usec
DE 10.00 usec
TE 300.0 K
D1 2.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 5.59 usec
PL1 -6.00 dB
SF01 400.1315829 MHz

F2 - Processing parameters
SI 16384
SF 400.1300078 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 8.443 ppm
F1 3378.49 Hz
F2P -0.500 ppm
F2 -199.87 Hz
PPMCM 0.44715 ppm/cm
HZCM 178.91789 Hz/cm

Current Data Parameters
 NAME fgr
 EXPNO 69
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20010415
 Time 1.32
 INSTRUM drx400
 PROBD 5 mm Multinu
 PULPROG zg30
 TO 16384
 SOLVENT CDC13
 NS 16
 DS 0
 SWH 4045.307 Hz
 FIDRES 0.246906 Hz
 AQ 2.0251124 sec
 RG 574.7
 DM 123.600 usec
 DE 10.00 usec
 TE 300.0 K
 D1 2.0000000 sec

===== CHANNEL f1 =====

NUC1 1H
 P1 5.59 usec
 PL1 -6.00 dB
 SF01 400.1314653 MHz

F2 - Processing parameters

SI 16384
 SF 400.1300079 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 F1P 8.208 ppm
 F1 3284.43 Hz
 F2P -0.358 ppm
 F2 -143.27 Hz
 PPMCM 0.42832 ppm/cm
 HZCM 171.38516 Hz/cm

Current Data Parameters

NAME gfl
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters

Date_ 20010602
Time 4.18
INSTRUM drx400
PROBHD 5 mm Dual 13
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SMH 4045.307 Hz
FIDRES 0.246906 Hz
AQ 2.0251124 sec
RG 812.7
DM 123.600 usec
DE 10.00 usec
TE 300.0 K
D1 2.00000000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 5.59 usec
PL1 -6.00 dB
SF01 400.1315829 MHz

F2 - Processing parameters

SI 16384
SF 400.1300176 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 7.973 ppm
F1 3190.07 Hz
F2P -0.331 ppm
F2 -132.46 Hz
PPMCM 0.41518 ppm/cm
HZCM 166.12656 Hz/cm