SUPPORTING INFORMATION

Additive Effects on Palladium-Catalyzed Deprotonative-Cross-Coupling Processes (DCCP) of sp^3 C–H Bonds in Diarylmethanes

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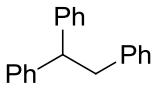
General Methods. All reactions were performed under nitrogen using oven-dried glassware and standard Schlenk or vacuum line techniques. Air- and moisture sensitive solutions were handled under nitrogen and transferred *via* syringe. Anhydrous CPME, DME and diglyme were purchased from Sigma-Aldrich and used as solvent without further purification. Unless otherwise stated, reagents were commercially available and used as purchased without further purification. Chemicals were obtained from Sigma-Aldrich, Acros, TCI America or Matrix Scientific. The progress of the reactions was monitored by thin-layer chromatography using Whatman Partisil K6F 250 μm precoated 60 Å silica gel plates and visualized by short-wave ultraviolet light as well as by treatment with ceric ammonium molybdate (CAM) stain or iodine. Silica gel (230–400 mesh, Silicycle) was used for flash chromatography. The ^{1}H NMR and ^{13}C{^{1}H} NMR spectra were obtained using a Brüker AM-500 Fourier-transform NMR spectrometer at 500 and 125 MHz, respectively. Chemical shifts are reported in units of parts per million (ppm) downfield from tetramethylsilane (TMS), and all coupling constants are reported in hertz. The infrared spectra were obtained on KBr plates using a Perkin-Elmer Spectrum 100 Series FTIR spectrometer. High resolution mass spectrometry (HRMS) data were obtained on a Waters LC-TOF mass spectrometer (model LCT-XE Premier) using chemical ionization (CI) or electrospray ionization (ESI) in positive or negative mode, depending on the analyte. Melting points were determined on a Unimelt Thomas-Hoover melting point apparatus and are uncorrected.

Procedure and Characterization for the Room-Temperature Deprotonation/Benzylation of Diphenylmethane (1a) and 2-Benzylpyridine (1b).

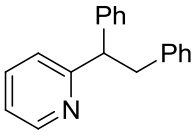
An oven-dried reaction vial equipped with a stir bar was charged with 3 equiv of the base (0.3 mmol) under a nitrogen atmosphere followed by 1 mL of dry CPME, and the reaction mixture was stirred for 5 min. Substrate **1a** or **1b** (0.3 mmol) was then added to the reaction followed by benzyl chloride (11.5 μL, 0.1 mmol). The reaction mixture was stirred for 12 h at rt and then quenched with two drops of H_2O, diluted with 3 mL of ethyl acetate, and filtered over a pad of $MgSO_4$ and silica. The pad was rinsed with additional ethyl acetate and the solution was concentrated under vacuum. The crude material was loaded onto a silica gel column and purified by flash chromatography. In the case of the use of 12-crown-4, the additive was added along with $LiN(SiMe_3)_2$.

1,1,2-triphenylethane



The reaction was performed as described above using $KN(SiMe_3)_2$ as base. The crude material was purified by flash chromatography on silica gel (eluted with hexanes to EtOAc:hexanes = 2:98) to give the product (24.7 mg, 96% yield) as a colorless oil. R_f = 0.25 (hexanes). The NMR spectral data match the previously published data.[1]

2-(1,2-diphenylethyl)pyridine

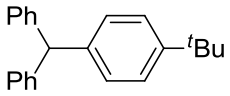


The reaction was performed as described above using $KN(SiMe_3)_2$ as base. The crude material was purified by flash chromatography on silica gel (eluted with EtOAc:hexanes = 1:9) to give the product (25.9 mg, 99% yield) as a colorless oil. R_f = 0.33 (EtOAc:hexanes = 1:9). The NMR spectral data match the previously published data.[2]

General Procedure and Characterization for the Pd-Catalyzed DCCP of Diarylmethanes in the presence of Additives.

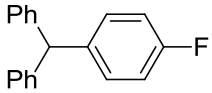
An oven-dried reaction vial charged with a solution (from a stock solution) of $Pd(OAc)_2$ (1.1 mg, 5 mol %) and NiXantphos (5.1 mg, 10 mol %) in 1 mL THF and the solvent was evaporated at rt under reduced pressure. The vial was equipped with a stir bar, taken inside the glovebox and it was charged with the corresponding base (0.3 mmol), additive (0.6 mmol 12-crown-4 or 0.03 mmol 15-crown-5 or 0.6 mmol HMTETA or 0.6 mmol diglyme) and 1 mL of dry CPME. After stirring for 30 min., the diarylmethane (0.3 mmol when using $NaN(SiMe_3)_2$/15-crown-5 and 0.12 mmol for all other cases) was added to the reaction followed by the aryl bromide (0.1 mmol). The vial was sealed and the reaction mixture was stirred for 12 h at rt. The reaction was quenched with two drops of H_2O, diluted with 3 mL of ethyl acetate, and filtered over a pad of $MgSO_4$ and silica. The pad was rinsed with additional ethyl acetate, and the solution was concentrated under vacuum. The crude material was loaded onto a silica gel column and purified by flash chromatography.

3aa – (4-*tert*-butylphenyl)diphenylmethane



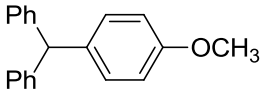
The reaction was performed following the General Procedure described above with **1a** (50.2 μL, 0.3 mmol), $NaN(SiMe_3)_2$ (55.0 mg, 0.3 mmol), 15-crown-5 (5.9 μL, 0.03 mmol) and **2a** (17.3 μL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with hexanes to EtOAc:hexanes = 2:98) to give the product (30.5 mg, 99% yield) as a white solid. R_f = 0.33 (hexanes). The NMR spectral data match the previously published data.[3]

3ab – (4-fluorophenyl)diphenylmethane



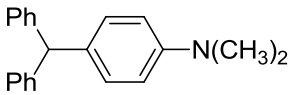
The reaction was performed following the General Procedure described above with **1a** (50.2 μL, 0.3 mmol), $NaN(SiMe_3)_2$ (55.2 mg, 0.3 mmol), 15-crown-5 (5.9 μL, 0.03 mmol) and **2b** (11.0 μL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (16.6 mg, 63% yield) as a white solid. R_f = 0.33 (hexanes). The NMR spectral data match the previously published data.[3]

3ad – (4-methoxyphenyl)diphenylmethane



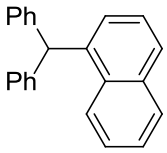
The reaction was performed following the General Procedure described above with **1a** (20.1 μL, 0.12 mmol), $LiN(SiMe_3)_2$ (50.2 mg, 0.3 mmol), 12-crown-4 (97.1 μL, 0.6 mmol) and **2d** (12.5 μL, 0.1 mmol) or **1a** (50.2 μL, 0.3 mmol), $NaN(SiMe_3)_2$ (55.0 mg, 0.3 mmol), 15-crown-5 (5.9 μL, 0.03 mmol) and **2d** (12.5 μL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with hexanes to EtOAc:hexanes = 5:95) to give the product (27.1 mg, 99% yield and 26.7 mg, 97% yield respectively) as a colorless oil. R_f = 0.25 (hexanes). The NMR spectral data match the previously published data.[3]

3ae – (*N*,*N*-dimethylaminophenyl)diphenylmethane



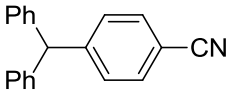
The reaction was performed following the General Procedure described above with **1a** (50.2 μL, 0.3 mmol), $NaN(SiMe_3)_2$ (55.0 mg, 0.3 mmol), 15-crown-5 (5.9 μL, 0.03 mmol) and **2e** (20.0 mg, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with EtOAc:hexanes = 5:95) to give the product (29.0 mg, 99% yield) as a white solid. R_f = 0.3 (EtOAc:hexanes = 5:95). The NMR spectral data match the previously published data.[3]

3af – (1-naphthyl)diphenylmethane



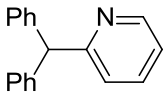
The reaction was performed following the General Procedure described above with **1a** (20.1 μL, 0.12 mmol), $NaN(SiMe_3)_2$ (54.9 mg, 0.3 mmol), 15-crown-5 (5.9 μL, 0.03 mmol) and **2f** (14.0 μL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with hexanes to EtOAc:hexanes = 3:97) to give the product (22.8 mg, 77% yield) as a white solid. R_f = 0.33 (hexanes); m.p. = 136–139 ºC; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.98 (d, *J* = 8.5 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.74 (d, *J* = 8.0 Hz, 1H), 7.45 – 7.32 (m, 3H), 7.30 – 7.24 (m, 4H), 7.24 – 7.18 (m, 2H), 7.14 – 7.07 (m, 4H), 6.94 (d, *J* = 7.0 Hz, 1H), 6.27 (s, 1H) ppm; $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$): δ 143.9, 140.1, 134.1, 132.1, 129.9, 128.9, 128.6, 127.8, 127.5, 126.6, 126.3, 125.6, 125.4, 124.5, 53.4 ppm; IR (thin film): 3058, 3025, 1598, 1493, 1449, 1395, 1030, 787, 725, 699 cm^{-1}; HRMS calcd. for $C_{23}H_{18}^+$ 294.1409, observed 294.1396 $[M]^+$.

3ag – (4-cyanophenyl)diphenylmethane



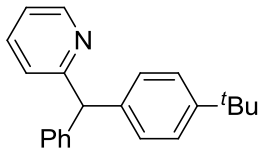
The reaction was performed following the General Procedure described above with **1a** (20.1 μL, 0.12 mmol), $LiN(SiMe_3)_2$ (50.1 mg, 0.3 mmol), 12-crown-4 (97.1 μL, 0.6 mmol) and **2g** (18.2 mg, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with hexanes to EtOAc:hexanes = 5:95) to give the product (18.1 mg, 67% yield) as a colorless oil. R_f = 0.25 (hexanes). The NMR spectral data match the previously published data.[4]

3ai – (2-pyridyl)diphenylmethane



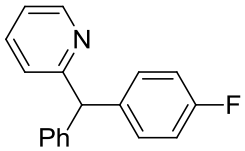
The reaction was performed following the General Procedure described above with **1a** (20.1 μL, 0.12 mmol), $LiN(SiMe_3)_2$ (50.2 mg, 0.3 mmol), 12-crown-4 (97.1 μL, 0.6 mmol) and **2i** (9.5 μL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with hexanes to EtOAc:hexanes = 2:8) to give the product (18.3 mg, 75% yield) as a white solid. R_f = 0.40 (EtOAc:hexanes = 2:8). The NMR spectral data match the previously published data.[5]

3ba – (4-*tert*-butylphenyl)(2-pyridyl)phenylmethane



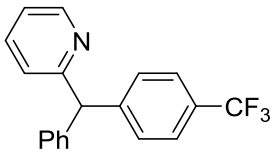
The reaction was performed following the General Procedure described above with **1b** (19.3 μL, 0.12 mmol), $LiN(SiMe_3)_2$ (50.0 mg, 0.3 mmol), 12-crown-4 (97.1 μL, 0.6 mmol) and **2a** (17.3 μL, 0.1 mmol) or **1b** (50.8 μL, 0.3 mmol), $NaN(SiMe_3)_2$ (55.0 mg, 0.3 mmol), diglyme (85.9 μL, 0.6 mmol) and **2a** (17.3 μL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with hexanes to EtOAc:hexanes = 1:9) to give the product (28.9 mg, 96% yield and 30.3 mg, 99% yield respectively) as a colorless oil. R_f = 0.3 (EtOAc:hexanes = 1:9). The NMR spectral data match the previously published data.[3]

3bb – (4-fluorophenyl)(2-pyridyl)phenylmethane



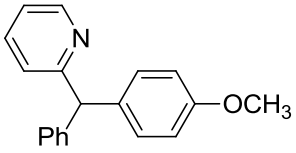
The reaction was performed following the General Procedure described above with **1b** (50.8 μL, 0.3 mmol), $NaN(SiMe_3)_2$ (55.1 mg, 0.3 mmol), 15-crown-5 (5.9 μL, 0.03 mmol) and **2b** (11.0 μL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with hexanes to EtOAc:hexanes = 1:9) to give the product (26.0 mg, 99% yield) as a colorless oil. R_f = 0.50 (EtOAc:hexanes = 2:8); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.62 – 8.56 (m, 1H), 7.59 (td, *J* = 7.8, 1.5 Hz, 1H), 7.32 – 7.26 (m, 2H), 7.24 – 7.19 (m, 1H), 7.17 – 7.09 (m, 5H), 7.07 (d, *J* = 8.0 Hz, 1H), 7.00 – 6.93 (m, 2H), 5.65 (s, 1H) ppm; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 163.2, 161.8 (d, *J* = 244 Hz), 149.9, 142.9, 138.8 (d, *J* = 3 Hz), 136.7, 131.0 (d, *J* = 8 Hz), 129.5, 128.7, 126.9, 123.9, 121.7, 115.4 (d, *J* = 21 Hz), 58.8 ppm; IR (thin film): 3060, 3028, 1603, 1587, 1507, 1433, 1223, 1159, 817, 748, 699 cm^{-1}; HRMS calcd. for $C_{18}H_{15}NF^+$ 264.1189, observed 264.1184 $[MH]^+$.

3bc – (4-trifluoromethylphenyl)(2-pyridyl)phenylmethane



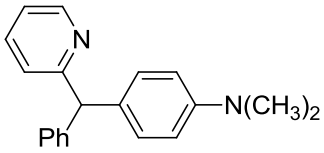
The reaction was performed following the General Procedure described above with **1b** (50.8 μL, 0.3 mmol), $LiN(SiMe_3)_2$ (33.5 mg, 0.2 mmol), 12-crown-4 (97.1 μL, 0.6 mmol) and **2c** (14.0 μL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with EtOAc:hexanes = 5:95 to 2:8) to give the product (24.5 mg, 78% yield) as a colorless oil. R_f = 0.5 (EtOAc:hexanes = 2:8); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.65 – 8.57 (m, 1H), 7.62 (td, *J* = 7.5, 2.0 Hz, 1H), 7.34 – 7.27 (m, 4H), 7.27 – 7.22 (m, 1H), 7.19 – 7.13 (m, 3H), 7.09 (d, *J* = 8.0 Hz, 1H), 5.72 (s, 1H) ppm; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 162.5, 150.0, 147.1, 142.1, 136.8, 129.9, 129.5, 128.8, 127.1, 125.5 (q, *J* = 4 Hz), 124.0, 121.9, 59.3 ppm; IR (thin film): 1326, 1164, 1122, 1068, 700 cm^{-1}; HRMS calcd. for $C_{19}H_{15}NF_3^+$ 314.1157, observed 314.1158 $[MH]^+$.

3bd – (4-methoxyphenyl)(2-pyridyl)phenylmethane



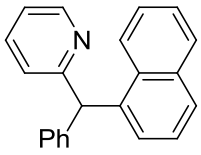
The reaction was performed following the General Procedure described above with **1b** (50.8 μL, 0.3 mmol), $NaN(SiMe_3)_2$ (55.1 mg, 0.3 mmol), 15-crown-5 (5.9 μL, 0.03 mmol) and **2d** (12.5 μL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with CH_2Cl_2, then with EtOAc:hexanes = 2:8) to give the product (27.6 mg, 99% yield) as a colorless oil. R_f = 0.33 (EtOAc:hexanes = 2:8). The NMR spectral data match the previously published data.[6]

3be – (4-*N*,*N*-dimethylaminophenyl)(2-pyridyl)phenylmethane



The reaction was performed following the General Procedure described above with **1b** (50.8 μL, 0.3 mmol), $NaN(SiMe_3)_2$ (55.0 mg, 0.3 mmol), 15-crown-5 (5.9 μL, 0.03 mmol) and **2e** (20.0 mg, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with EtOAc:hexanes = 1:9 to 2:8) to give the product (27.0 mg, 94% yield) as a white solid. R_f = 0.3 (EtOAc:hexanes = 2:8); m.p. = 94–96 ºC; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.60 – 8.54 (m, 1H), 7.56 (td, *J* = 7.5, 2.0 Hz, 1H), 7.30 – 7.24 (m, 2H), 7.22 – 7.13 (m, 3H), 7.12 – 7.06 (m, 2H), 7.03 (d, *J* = 9.0 Hz, 2H), 6.67 (d, *J* = 8.5 Hz, 2H), 5.61 (s, 1H) , 2.90 (s, 6H) ppm; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 164.2, 149.6, 149.4, 143.7, 136.5, 130.9, 130.1, 129.5, 128.5, 126.4, 123.8, 121.4, 112.8, 58.7, 40.8 ppm; IR (thin film): 2885, 2800, 1614, 1587, 1520, 1432, 1349, 748, 699 cm^{-1}; HRMS calcd. for $C_{20}H_{21}N_2^+$ 289.1705, observed 289.1711 $[MH]^+$.

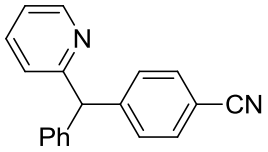
3bf – (1-naphthyl)(2-pyridyl)phenylmethane



The reaction was performed following the General Procedure described above with **1b** (50.8 μL, 0.3 mmol), $NaN(SiMe_3)_2$ (55.1 mg, 0.3 mmol), 15-crown-5 (5.9 μL, 0.03 mmol) and **2f** (14.0 μL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with hexanes to EtOAc:hexanes = 1:9) to give the product (29.3 mg, 99% yield) as a white solid. R_f = 0.25 (EtOAc:hexanes = 1:9); m.p. = 119–121 ºC; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.65 – 8.56 (m, 1H), 7.99 (d, *J* = 8.5 Hz, 1H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.74 (d, *J* = 8.0 Hz, 1H), 7.52 (td, *J* = 7.8, 2.0 Hz, 1H), 7.44 – 7.32 (m, 3H), 7.32 – 7.25 (m, 2H), 7.25 – 7.18 (m, 1H), 7.17 – 7.07 (m, 3H), 6.98 (d, *J* = 7.5 Hz, 2H), 6.45 (s, 1H) ppm; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 163.5, 149.9, 142.7, 139.2, 136.6, 134.3, 132.3, 129.9, 128.9, 128.7, 127.7, 127.5, 126.8, 126.4, 125.7, 125.5, 124.5, 124.2, 121.6, 56.2 ppm; IR (thin

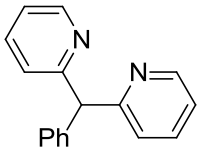
film): 3059, 1587, 1466, 1432, 789, 775, 728, 700 cm^{-1}; HRMS calcd. for $C_{22}H_{18}N^+$ 296.1439, observed 296.1443 $[MH]^+$.

3bg – (4-cyanophenyl)(2-pyridyl)phenylmethane



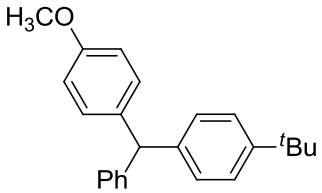
The reaction was performed following the General Procedure described above with **1b** (19.3 µL, 0.12 mmol), $LiN(SiMe_3)_2$ (50.2 mg, 0.3 mmol), 12-crown-4 (97.1 µL, 0.6 mmol) and **2g** (18.2 mg, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with EtOAc:hexanes = 1:9 to 2:8) to give the product (23.1 mg, 86% yield) as a colorless oil. R_f = 0.2 (EtOAc:hexanes = 2:8); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.63 – 8.58 (m, 1H), 7.64 (td, *J* = 7.8, 2.0 Hz, 1H), 7.60 – 7.56 (m, 2H), 7.35 – 7.24 (m, 5H), 7.20 – 7.13 (m, 3H), 7.09 (d, *J* = 8.0 Hz, 1H), 5.70 (s, 1H) ppm; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 161.9, 150.0, 148.6, 141.5, 137.0, 132.4, 130.4, 129.4, 128.9, 127.3, 124.0, 122.1, 119.1, 110.6, 59.4 ppm; IR (thin film): 3062, 2227, 1587, 1433 cm^{-1}; HRMS calcd. for $C_{19}H_{15}N_2^+$ 271.1235, observed 271.1233 $[MH]^+$.

3bi – 2,2'-(phenylmethylene)dipyridine



The reaction was performed following the General Procedure described above with **1b** (19.3 µL, 0.12 mmol), $NaN(SiMe_3)_2$ (55.0 mg, 0.3 mmol), HMTETA (163.2 µL, 0.6 mmol) and **2i** (9.5 µL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with hexanes to EtOAc:hexanes = 6:4 to EtOAc) to give the product (23.7 mg, 96% yield) as a white solid. R_f = 0.45 (EtOAc). The NMR spectral data match the previously published data.[7]

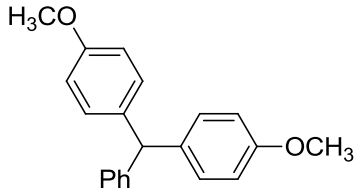
3ca – (4-*tert*-butylphenyl)(4-methoxyphenyl)phenylmethane



The reaction was performed following the General Procedure described above with **1c** (19.8 µL, 0.12 mmol), $LiN(SiMe_3)_2$ (50.2 mg, 0.3 mmol), 12-crown-4 (97.1 µL, 0.6 mmol) and **2a** (17.3 µL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with CH_2Cl_2:hexanes = 4:96 to 15:85) to give the product (17.3 mg, 52% yield) as a light yellow oil. R_f = 0.25 (EtOAc:hexanes = 2:98); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.31 –

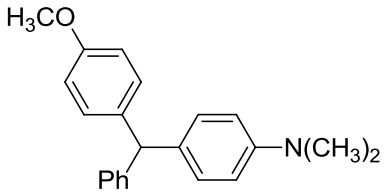
7.23 (m, 4H), 7.22 – 7.16 (m, 1H), 7.14 – 7.09 (m, 2H), 7.06 – 7.00 (m, 4H), 6.84 – 6.78 (m, 2H), 5.45 (s, 1H), 3.77 (s, 3H), 1.30 (s, 9H) ppm; $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$): δ 158.2, 149.2, 144.8, 141.3, 136.6, 130.6, 129.6, 129.1, 128.4, 126.3, 125.3, 113.9, 55.8, 55.4, 34.6, 31.6 ppm; IR (thin film): 2961, 1608, 1509, 1248, 1177, 1036, 822, 701 cm^{-1}; HRMS calcd. for $C_{24}H_{26}O^{+}$ 330.1984, observed 330.1980 $[M]^{+}$.

3cd – (4-methoxyphenyl)(4-methoxyphenyl)phenylmethane



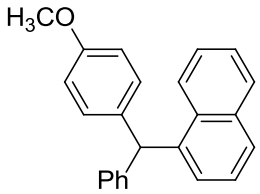
The reaction was performed following the General Procedure described above with **1c** (19.8 μL, 0.12 mmol), $LiN(SiMe_3)_2$ (50.2 mg, 0.3 mmol), 12-crown-4 (97.1 μL, 0.6 mmol) and **2d** (12.5 μL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with hexanes to EtOAc:hexanes = 2:98) to give the product (14.4 mg, 55% yield) as a colorless oil. R_f = 0.25 (EtOAc:hexanes = 5:95). The NMR spectral data match the previously published data.[8]

3ce – (4-*N*,*N*-dimethylaminophenyl)(4-methoxyphenyl)phenylmethane



The reaction was performed following the General Procedure described above with **1c** (19.8 μL, 0.12 mmol), $LiN(SiMe_3)_2$ (50.0 mg, 0.3 mmol), 12-crown-4 (97.1 μL, 0.6 mmol) and **2e** (20.0 mg, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with hexanes to EtOAc:hexanes = 5:95) to give the product (19.5 mg, 61% yield) as a colorless oil. R_f = 0.25 (EtOAc:hexanes = 5:95). The NMR spectral data match the previously published data.[3]

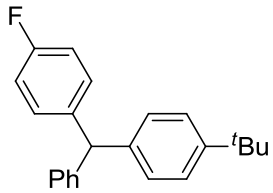
3cf – (1-naphthyl)(4-methoxyphenyl)phenylmethane



The reaction was performed following the General Procedure described above with **1c** (19.8 μL, 0.12 mmol), $LiN(SiMe_3)_2$ (50.2 mg, 0.3 mmol), 12-crown-4 (97.1 μL, 0.6 mmol) and **2f** (14.0 μL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with hexanes to EtOAc:hexanes = 2:98) to give the product (20.3 mg, 63% yield) as a colorless oil. R_f = 0.16 (EtOAc:hexanes = 2:98); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.98 (d, *J* = 8.5 Hz, 1H), 7.85 (d, *J* = 7.5 Hz, 1H), 7.74 (d, *J* = 8.5 Hz, 1H), 7.45 – 7.32 (m, 3H), 7.30 – 7.26 (m, 2H),

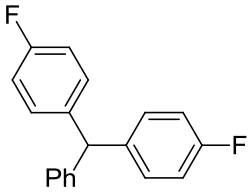
7.24 – 7.18 (m, 1H), 7.11 (d, *J* = 7.5 Hz, 2H), 7.02 (d, *J* = 8.5 Hz, 2H), 6.94 (d, *J* = 7.0 Hz, 1H), 6.82 (d, *J* = 9.0 Hz, 2H), 6.22 (s, 1H), 3.78 (s, 3H) ppm; $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$): δ 158.3, 144.3, 140.4, 136.1, 134.1, 132.1, 130.8, 129.8, 128.9, 128.6, 127.7, 127.4, 126.5, 126.3, 125.6, 125.4, 124.6, 114.0, 55.4, 52.5 ppm; IR (thin film): 3058, 2931, 2834, 1609, 1509, 1248, 1178, 1032, 785, 731 cm^{-1}; HRMS calcd. for $C_{24}H_{20}O^{+}$ 324.1514, observed 324.1520 $[M]^{+}$.

3da – (4-*tert*-butylphenyl)(4-fluorophenyl)phenylmethane



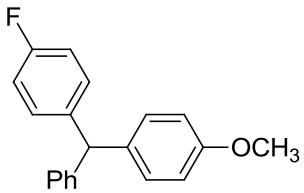
The reaction was performed following the General Procedure described above with **1d** (18.6 μL, 0.12 mmol), $NaN(SiMe_3)_2$ (55.1 mg, 0.3 mmol), HMTETA (163.2 μL, 0.6 mmol) and **2a** (17.3 μL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (30.3 mg, 99% yield) as a colorless oil. R_f = 0.33 (hexanes). The NMR spectral data match the previously published data.[3]

3db – (4-fluorophenyl)(4-fluorophenyl)phenylmethane



The reaction was performed following the General Procedure described above with **1d** (18.6 μL, 0.12 mmol), $LiN(SiMe_3)_2$ (50.2 mg, 0.3 mmol), 12-crown-4 (97.1 μL, 0.6 mmol) and **2b** (11.0 μL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with hexanes) to give the product (21.1 mg, 76% yield) as a colorless oil. R_f = 0.33 (hexanes); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32 – 7.26 (m, 2H), 7.26 – 7.20 (m, 1H), 7.09 – 7.01 (m, 6H), 7.00 – 6.93 (m, 4H), 5.51 (s, 1H) ppm; $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$): δ 161.7 (d, *J* = 244 Hz), 143.7, 139.7 (d, *J* = 3 Hz), 131.0 (d, *J* = 8 Hz), 129.4, 128.7, 126.8, 115.4 (d, *J* = 21 Hz), 55.4 ppm; IR (thin film): 3029, 1603, 1508, 1224, 1157, 826, 700 cm^{-1}; HRMS calcd. for $C_{19}H_{14}F_2^{+}$ 280.1064, observed 280.1059 $[M]^{+}$.

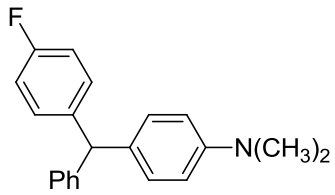
3dd (= 3cb) – (4-methoxyphenyl)(4-fluorophenyl)phenylmethane



The reaction was performed following the General Procedure described above with **1d** (55.8 μL, 0.3 mmol), $NaN(SiMe_3)_2$ (54.9 mg, 0.3 mmol), 15-crown-5 (5.9 μL, 0.03 mmol) and **2d** (12.5 μL, 0.1 mmol) or **1c** (19.8 μL, 0.12 mmol), $LiN(SiMe_3)_2$ (50.2 mg, 0.3 mmol), 12-crown-4 (97.1 μL, 0.6 mmol) and **2b** (11.0 μL, 0.1 mmol). The

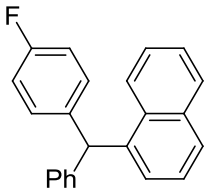
crude material was purified by flash chromatography on silica gel (eluted with hexanes to EtOAc:hexanes = 2:98) to give the product (28.9 mg, 99% yield and 14.8 mg, 51% yield respectively) as a colorless oil. R_f = 0.33 (EtOAc:hexanes = 2:98); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32 – 7.26 (m, 2H), 7.24 – 7.18 (m, 1H), 7.11 – 7.03 (m, 4H), 7.03 – 6.93 (m, 4H), 6.85 – 6.80 (m, 2H), 5.48 (s, 1H), 3.78 (s, 3H) ppm; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 161.6 (d, *J* = 243 Hz), 158.3, 144.3, 140.2 (d, *J* = 3 Hz), 136.1, 131.0 (d, *J* = 8 Hz), 130.5, 129.5, 128.6, 126.6, 115.3 (d, *J* = 21 Hz), 113.9, 55.45, 55.41 ppm; IR (thin film): 3028, 2836, 1606, 1506, 1248, 1034, 824, 700 cm^{-1}; HRMS calcd. for $C_{20}H_{16}FO^+$ 291.1185, observed 291.1189 [M-H]$^+$.

3de – (4-methoxyphenyl)(4-fluorophenyl)phenylmethane



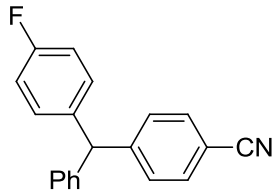
The reaction was performed following the General Procedure described above with **1d** (55.8 μL, 0.3 mmol), $NaN(SiMe_3)_2$ (54.9 mg, 0.3 mmol), 15-crown-5 (5.9 μL, 0.03 mmol) and **2e** (20.0 mg, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with EtOAc:hexanes = 2:98 to 5:95) to give the product (30.2 mg, 99% yield) as a colorless oil. R_f = 0.33 (EtOAc:hexanes = 5:95); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.30 – 7.24 (m, 2H), 7.22 – 7.16 (m, 1H), 7.12 – 7.04 (m, 4H), 6.97 – 6.91 (m, 4H), 6.69 – 6.64 (m, 2H), 5.43 (s, 1H), 2.91 (s, 6H) ppm; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 161.5 (d, *J* = 243 Hz), 149.3, 144.7, 140.6 (d, *J* = 3 Hz), 131.9, 131.0 (d, *J* = 8 Hz), 130.1, 129.5, 128.5, 126.4, 115.1 (d, *J* = 21 Hz), 112.7, 55.3, 40.8 ppm; IR (thin film): 3026, 2883, 2800, 1614, 1520, 1506, 1350, 1222, 816, 701 cm^{-1}; HRMS calcd. for $C_{21}H_{20}NF^+$ 305.1580, observed 305.1577 [M]$^+$.

3df – (1-naphthyl)(4-fluorophenyl)phenylmethane



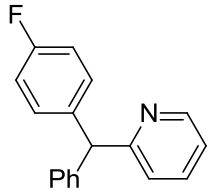
The reaction was performed following the General Procedure described above with **1d** (18.6 μL, 0.12 mmol), $LiN(SiMe_3)_2$ (50.1 mg, 0.3 mmol), 12-crown-4 (97.1 μL, 0.6 mmol) and **2e** (14.0 μL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with hexanes to EtOAc:hexanes = 2:98) to give the product (27.4 mg, 88% yield) as a yellow oil. R_f = 0.33 (EtOAc:hexanes = 2:98); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.94 (d, *J* = 8.5 Hz, 1H), 7.85 (d, *J* = 8.0 Hz, 1H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.46 – 7.33 (m, 3H), 7.31 – 7.25 (m, 2H), 7.25 – 7.19 (m, 1H), 7.12 – 7.03 (m, 4H), 6.99 – 6.88 (m, 3H), 6.24 (s, 1H) ppm; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 161.7 (d, *J* = 243 Hz), 143.8, 139.9, 139.6 (d, *J* = 3 Hz), 134.2, 132.0, 131.2 (d, *J* = 8 Hz), 129.8, 129.0, 128.7, 127.72, 127.68, 126.7, 126.4, 125.7, 125.4, 124.4, 115.4 (d, *J* = 21 Hz), 52.6 ppm; IR (thin film): 3059, 1600, 1506, 1223, 1158, 780, 733, 699 cm^{-1}; HRMS calcd. for $C_{23}H_{17}F^+$ 312.1314, observed 312.1313 [M]$^+$.

3dg– (4-cyanophenyl)(4-fluorophenyl)phenylmethane



The reaction was performed following the General Procedure described above with **1d** (55.8 μL, 0.3 mmol), $NaN(SiMe_3)_2$ (55.1 mg, 0.3 mmol), 15-crown-5 (5.9 μL, 0.03 mmol) and **2g** (18.2 mg, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with EtOAc:hexanes = 5:95) to give the product (7.5 mg, 26% yield) as a colorless oil. R_f = 0.2 (EtOAc:hexanes = 5:95); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.58 (d, *J* = 8.5 Hz, 2H), 7.32 (t, *J* = 7.5 Hz, 2H), 7.28 – 7.24 (m, 1H), 7.21 (d, *J* = 8.5 Hz, 2H), 7.08 – 6.96 (m, 6H), 5.56 (s, 1H) ppm; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 161.9 (d, *J* = 245 Hz), 149.5, 142.4, 138.4 (d, *J* = 3 Hz), 132.5, 131.0 (d, *J* = 8 Hz), 130.3, 129.4, 128.9, 127.2, 119.0, 115.7 (d, *J* = 21 Hz), 110.7, 56.2 ppm; IR (thin film): 3030, 2228, 1607, 1507, 1224, 1159, 826, 738, 701 cm^{-1}; HRMS calcd. for $C_{20}H_{15}NF^+$ 288.1189, observed 288.1194 $[MH]^+$.

3di– (2-pyridyl)(4-fluorophenyl)phenylmethane



The reaction was performed following the General Procedure described above with **1d** (18.6 μL, 0.12 mmol), $LiN(SiMe_3)_2$ (50.2 mg, 0.3 mmol), 12-crown-4 (97.1 μL, 0.6 mmol) and **2i** (9.5 μL, 0.1 mmol). The crude material was purified by flash chromatography on silica gel (eluted with EtOAc:hexanes = 5:95 to 1:9) to give the product (22.4 mg, 85% yield) as a colorless oil. R_f = 0.45 (EtOAc:hexanes = 2:8); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.62 – 8.56 (m, 1H), 7.60 (td, *J* = 7.8, 2.0 Hz, 1H), 7.30 (t, *J* = 7.5 Hz, 2H), 7.23 (t, *J* = 7.5 Hz, 1H), 7.16 – 7.10 (m, 5H), 7.07 (d, *J* = 8.0 Hz, 1H), 7.01 – 6.94 (m, 2H), 5.67 (s, 1H) ppm; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 163.2, 161.8 (d, *J* = 244 Hz), 149.8, 142.8, 138.7 (d, *J* = 3 Hz), 136.7, 131.0 (d, *J* = 8 Hz), 129.5, 128.7, 126.9, 123.9, 121.7, 115.4 (d, *J* = 21 Hz), 58.7 ppm; IR (thin film): 3060, 2895, 1587, 1507, 1432, 1222, 1158, 1098, 817, 748, 699 cm^{-1}; HRMS calcd. for $C_{18}H_{15}NF^+$ 264.1189, observed 264.1183 $[MH]^+$.

High-throughput Experimentation Screenings.

General Experimental:

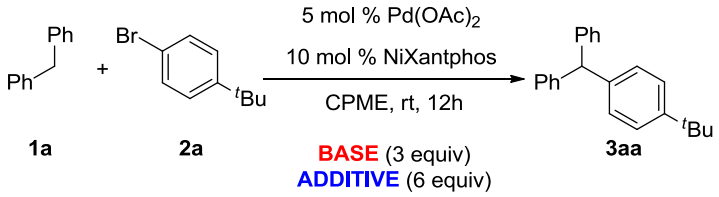
Set up:

Experiments were set up inside a glovebox under a nitrogen atmosphere. A 96-well aluminum block containing 1 mL glass vials was predosed with $Pd(OAc)_2$ (0.5 μmol) and NiXantphos (1 μmol) in THF. The solvent was removed to dryness using a J-Kem blow down block and the corresponding base (30 μmol) and additive (60 μmol for 12-crown-4, HMTETA and dilgyme and 3 μmol for 15-crown-5) in THF were added to the ligand/catalyst mixture. The solvent was removed on the J-Kem blow down block and a parylene stir bar was then added to each reaction vial. The aryl bromide (10 μmol/reaction), diarylmethane (30 μmol/reaction when using $NaN(SiMe_3)_2$/15-crown-5 and 12 μmol/reaction for all other cases) and biphenyl (1 μmol/reaction) (used as an internal standard to measure HPLC yields) were then dosed together into each reaction vial as a solution in CPME (100 μL, 0.1 M). The 96-well plate was then sealed and stirred for 18 h at rt.

Work up:

Upon opening the plate to air, 500 μL of acetonitrile was added into each vial. The plate was covered again and the vials stirred for 10 min. to ensure good homogenization. Into a separate 96-well LC block was added 700 μL of acetonitrile, followed by 40 μL of the diluted reaction mixtures. The LC block was then sealed with a silicon-rubber storage mat and mounted on an automated HPLC instrument for analysis.

(1) Additive and Base Screening:



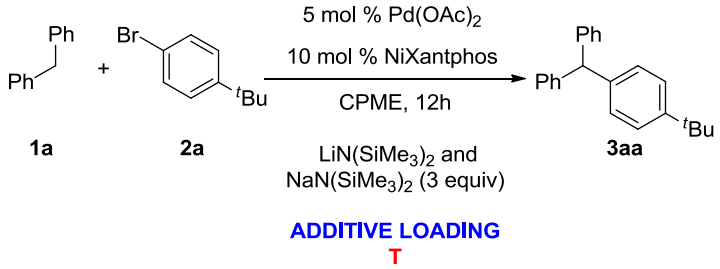
Bases: $KN(SiMe_3)_2$, $LiN(SiMe_3)_2$, $NaN(SiMe_3)_2$, KO*t*Bu, LiO*t*Bu, NaO*t*Bu.

Additives: 12-crown-4, 15-crown-5, 18-crown-6, NEt_3, TMEDA, *N*,*N*,*N'*,*N'*-tetramethyl-1,3-propanediamine (TMPDA), *N*,*N'*-diisopropylethylenediamine, *N*,*N*-diethylethylenediamine, *N*,*N'*-dibenzylethylenediamine (DBED), *N*,*N*,*N'*,*N''*,*N''*-pentamethyldiethylenetriamine (PMDTA), 2,2'-bipyridine (bipy), 1,10-phenanthroline and 1,4-diazabicyclo[2.2.2]octane (DABCO).

Additives	Bases (AY % 3aa)					
	$KN(SiMe_3)_2$	$LiN(SiMe_3)_2$	$NaN(SiMe_3)_2$	KO*t*Bu	LiO*t*Bu	NaO*t*Bu
Control	95	0	0	0	0	0
crown ether	25	97	91	0	0	5
NEt_3	80	0	0	0	0	0
TMEDA	89	6	10	0	0	2
TMPDA	79	0	0	0	0	0
N,*N*′-diisopropylethylenediamine	40	2	10	0	0	3
N,*N*-diethylethylenediamine	0	0	0	0	0	0
DBED	0	0	0	0	0	0
PMDTA	68	8	12	0	0	5
Bipy	70	0	0	0	0	0
1,10-phenanthroline	71	0	0	0	0	0
DABCO	71	0	0	0	0	0

The lead hits from the screening were $LiN(SiMe_3)_2$/12-crown-4 giving 97% assay yield **3aa**, $NaN(SiMe_3)_2$/15-crown-5 rendering product in 91% assay yield and $NaN(SiMe_3)_2$/TMEDA, $NaN(SiMe_3)_2$/*N*,*N*′-diisopropylethylenediamine and $NaN(SiMe_3)_2$/PMDTA generating the triarylmethane compound in 10-12% assay yield.

(2) Additive Loading and Temperature Screening



Bases: $LiN(SiMe_3)_2$, $NaN(SiMe_3)_2$.

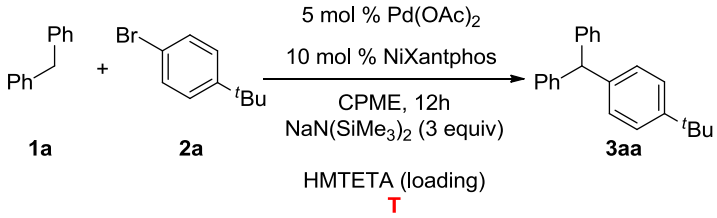
Additives/Additives loading: 12-crown-4, 15-crown-5, TMEDA, *N*,*N*′-diisopropylethylenediamine and PMDTA using 1, 2, 3, 6 and 8 equiv.

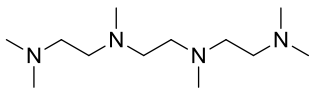
Base	Additive, loading	T (°C)	AY (%) 3aa
$NaN(SiMe_3)_2$	15-crown-5 6 equiv	23	91
	15-crown-5 1 equiv	23	68
	N,*N*′-diisopropylethylenediamine 6 equiv	23	2
		110	15
	N,*N*′-diisopropylethylenediamine 2 equiv	23	2
		110	11
	TMEDA 8 equiv	23	5
		110	24
	TMEDA 6 equiv	23	7
		110	32
	TMEDA 3 equiv	23	4

		110	28
	TMEDA 2 equiv	23	3
		110	27
	TMEDA 1 equiv	23	2
		110	27
	PMDTA 6 equiv	23	4
		110	31
	PMDTA 3 equiv	23	9
		110	32
	PMDTA 1 equiv	23	3
		110	45
$LiN(SiMe_3)_2$	12-crown-4 6 equiv	23	97
	12-crown-4 1 equiv	23	79

Conclusions from the screening: loading of the crown ethers could be reduced from 6 equiv to 1 equiv without a dramatic impact in the reaction yield at rt although yields obtained were lower. PMDTA was more promising than TMEDA and *N,N'*-diisopropylethylenediamine at 110 °C. 1 equiv PMDTA rendered **3aa** in 45% assay yield while 6 equiv TMEDA generated the product in 32% assay yield.

(3) Tetramine Loading and Temperature Screening





HMTETA

Tetramines: HMTETA (30 mol %, 1 equiv, 3 equiv and 6 equiv).

Base	Additive, loading	T (°C)	1a loading	AY (%) 3aa
$NaN(SiMe_3)_2$	HMTETA 6 equiv	23	1.2 equiv	80
		110	1.2 equiv	73
		23	3 equiv	97
		110	1.2 equiv	83
	HMTETA 3 equiv	23	1.2 equiv	23
		110	1.2 equiv	73
	HMTETA 1 equiv	23	3 equiv	15
	HMTETA 30 mol%	23	3 equiv	4

The lead hit from the screening was the combination of $NaN(SiMe_3)_2$/6 equiv HMTETA at rt affording 80% assay yield of the desired DCCP product **3aa** when 1.2 equiv **1a** were used and 97% assay yield when 3 equiv **1a** were used.

(4) Base, Solvent, Temperature and Additive loading Screening

Base: $KN(SiMe_3)_2$, $LiN(SiMe_3)_2$, $NaN(SiMe_3)_2$, KO*t*Bu, LiO*t*Bu, NaO*t*Bu.

Solvent: CPME, DME, diglyme.

Temperature: rt, 50 °C, 110 °C.

Additives: 12-crown-4, 15-crown-5, 18-crown-6, TMEDA, PMDTA, HMTETA, diglyme.

$NaN(SiMe_3)_2$

No additive:

Base	Solvent	T (°C)	AY (%) 3aa
$NaN(SiMe_3)_2$	CPME	23	NR
		50	NR
		110	35
	DME	23	40
		110	70
	diglyme	23	54
		110	92

Although diglyme as coordinating solvent rendered good results at high temperature, diglyme's toxicity and high boiling point made us consider its role as an additive.

In the presence of additives:

15-crown-5 in CPME:

	Loading (equiv)	T (°C)	1a loading (equiv)	AY (%) 3aa
$NaN(SiMe_3)_2$	6 equiv	23	1.2	91
	1 equiv	23	1.2	68
		23	3	79
		110	1.2	83
		110	3	87
	0.03 equiv	23	1.2	54
		23	3	102
		50	1.2	90
		110	3	74
	0.015 equiv	23	1.2	41
		23	3	54
		50	1.2	74
		110	3	79
	0.003 equiv	23	1.2	NR
		23	3	3
		50	1.2	17
		110	3	67

Best hit: $NaN(SiMe_3)_2$/0.03 equiv 15-crown-5, 3 equiv **1a** in CPME at rt generated the desired product in quantitative assay yield.

HMTETA in different solvents:

Base	Loading (equiv)	Solvent	T (°C)	1a loading (equiv)	AY (%) 3aa
$NaN(SiMe_3)_2$	6 equiv	CPME	23	1.2	80
			23	3	97
		diglyme	23	1.2	56
	3 equiv	CPME	23	1.2	23
			50	1.2	76
		DME	23	1.2	42
	1 equiv	CPME	23	3	15
			23	3	4
	0.03 equiv	CPME	110	1.2	65

Best hit: $NaN(SiMe_3)_2$/6 equiv HMTETA, 1.2 equiv **1a** in CPME at rt generated the desired product in 80% assay yield.

Diglyme in CPME:

Base	Loading (equiv)	T (°C)	1a loading (equiv)	AY (%) 3aa
$NaN(SiMe_3)_2$	6 equiv	23	1.2	88
		23	3	97
	3 equiv	23	1.2	41
		50	1.2	89
	1 equiv	23	3	40
	0.03 equiv	23	3	6
		110	1.2	41

Best hit: $NaN(SiMe_3)_2$/6 equiv diglyme, 1.2 equiv **1a** in CPME at rt generated the desired product in 88% assay yield.

$LiN(SiMe_3)_2$

Additive, loading	Solvent	T (°C)	1a loading (equiv)	AY (%) 3aa
-	CPME	23	1.2	NR
-		50	1.2	NR
-		50	3	NR
12-crown-4 6 equiv		23	1.2	97
12-crown-4 1 equiv		23	1.2	79
		50	1.2	78
HMTETA 6 equiv	CPME	23	1.2	33

Best hit: $LiN(SiMe_3)_2$/6 equiv 12-crown-4, 1.2 equiv **1a** in CPME at rt generated the desired product in 97% assay yield.

Other bases:

Base	Additive, loading	Solvent	T (°C)	1a loading (equiv)	AY (%) 3aa
KO*t*Bu	-	CPME	23	1.2	NR
	-	diglyme	23	1.2	10
	-	DME	110	3	11
	18-crown-6 6 equiv	CPME	23	1.2	NR
	18-crown-6 2equiv	DME	50	1.2	5
			50	3	9
	18-crown-6 0.03 equiv		50	1.2	6
			50	3	13
	18-crown-6 0.003 equiv		50	1.2	5
			50	3	11
	TMEDA 3 equiv	CPME	110	3	9
	TMEDA 1 equiv		110	3	14
	TMEDA 0.03 equiv		110	3	16
	PMDTA 3 equiv		110	3	13
	PMDTA 1 equiv		110	3	14
	PMDTA 0.03 equiv		110	3	20
	HMTETA 6 equiv	diglyme	23	1.2	15
LiO*t*Bu	-	CPME	23	1.2	NR
	-	diglyme	23	1.2	NR
	12-crown-4 6 equiv	CPME	23	1.2	NR
NaO*t*Bu	-	CPME	23	1.2	NR
	-	diglyme	23	1.2	NR
	15-crown-5 6 equiv	CPME	23	1.2	NR
		CPME	110	1.2	12

(5) Substrate Scope Screening

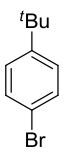
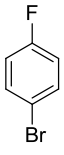
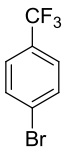
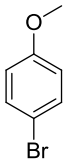
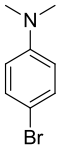
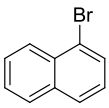
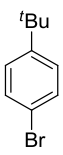
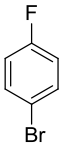
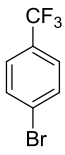
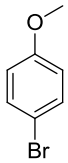
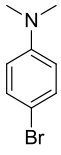
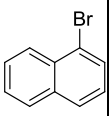
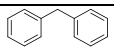
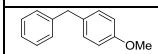
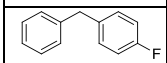
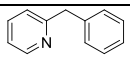
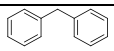
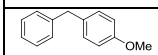
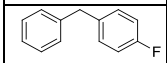
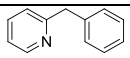
Well	Substrates	Base	Additive, loading	Prod/IS[a]	Prod/IS[a] ($KN(SiMe_3)_2$)
A01	**1a/2a**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	5.28 (95%)[b]	6.36
A07		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	6.14 (99%)[b]	
E01		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	5.13 (86)[b]	
E07		$NaN(SiMe_3)_2$	diglyme, 6 equiv	4.77 (78)[b]	
A02	**1a/2b**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	0.47	4.08
A08		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	3.96 (63%)[b]	
E02		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	1.86	
E08		$NaN(SiMe_3)_2$	diglyme, 6 equiv	0.86	
A03	**1a/2c**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	0.31	3.22
A09		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	0.37	
E03		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	0	
E09		$NaN(SiMe_3)_2$	diglyme, 6 equiv	0.18	
A04	**1a/2d**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	6.90 (99%)[b]	7.90
A10		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	7.65 (97%)[b]	
E04		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	0.12	
E10		$NaN(SiMe_3)_2$	diglyme, 6 equiv	1.85	
A05	**1a/2e**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	6.52	10.51
A11		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	9.96 (99%)[b]	
E05		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	0.18	
E11		$NaN(SiMe_3)_2$	diglyme, 6 equiv	2.08	
A06	**1a/2f**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	0.74	7.97
A12		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	7.77 (77%)[b]	
E06		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	4.34	
E12		$NaN(SiMe_3)_2$	diglyme, 6 equiv	1.65	

B01	**1c/2a**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	5.46 (52%)[b]	7.78
B07		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	2.68	
F01		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	0.68	
F07		$NaN(SiMe_3)_2$	diglyme, 6 equiv	0.07	
B02	**1c/2b**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	3.51 (51%)[b]	1.25 (17%)[c]
B08		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	0.90	
F02		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	0.18	
F08		$NaN(SiMe_3)_2$	diglyme, 6 equiv	0	
B03	**1c/2c**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	0	0
B09		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	0	
F03		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	0	
F09		$NaN(SiMe_3)_2$	diglyme, 6 equiv	0	
B04	**1c/2d**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	5.20 (48%)[b]	7.02
B10		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	2.64	
F04		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	0.55	
F10		$NaN(SiMe_3)_2$	diglyme, 6 equiv	0.24	
B05	**1c/2e**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	5.27 (52%)[b]	8.57
B11		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	3.01	
F05		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	0	
F11		$NaN(SiMe_3)_2$	diglyme, 6 equiv	0.42	
B06	**1c/2f**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	6.00 (63%)[b]	8.41
B12		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	2.71	
F06		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	0	
F12		$NaN(SiMe_3)_2$	diglyme, 6 equiv	0.15	
C01	**1d/2a**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	3.66	6.68
C07		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	0.35	
G01		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	4.46 (99%)[b]	
G07		$NaN(SiMe_3)_2$	diglyme, 6 equiv	2.98	
C02	**1d/2b**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	5.63 (76%)[b]	3.36 (50%)[c]
C08		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	3.33	
G02		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	3.56	
G08		$NaN(SiMe_3)_2$	diglyme, 6 equiv	0.16	
C03	**1d/2c**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	0	0
C09		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	0	
G03		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	0	
G09		$NaN(SiMe_3)_2$	diglyme, 6 equiv	0	
C04	**1d/2d**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	7.67	7.99
C10		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	7.66 (99%)[b]	
G04		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	4.41	
G10		$NaN(SiMe_3)_2$	diglyme, 6 equiv	3.13	
C05	**1d/2e**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	0	10.46
C11		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	10.01 (99%)[b]	
G05		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	7.63	
G11		$NaN(SiMe_3)_2$	diglyme, 6 equiv	2.90	
C06	**1d/2f**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	7.73 (88%)[b]	8.91
C12		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	8.70	
G06		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	5.95	
G12		$NaN(SiMe_3)_2$	diglyme, 6 equiv	3.35	
D01	**1b/2a**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	6.23	6.45
D07		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	1.68	
H01		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	2.39	
H07		$NaN(SiMe_3)_2$	diglyme, 6 equiv	6.13 (99%)[b]	
D02	**1b/2b**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	4.32	1.46 (68%)[c]
D08		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	4.57 (99%)[b]	
H02		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	1.79	
H08		$NaN(SiMe_3)_2$	diglyme, 6 equiv	4.96	
D03		$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	multiple	

	1b/**2c**			products	1.17 (0%)[c]
D09		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	4.50 (multiple products)[b]	
H03		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	multiple products	
H09		$NaN(SiMe_3)_2$	diglyme, 6 equiv	multiple products	
D04	**1b**/**2d**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	6.21	5.91 (quant.)[c]
D10		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	7.56 (116%)[b]	
H04		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	6.04	
H10		$NaN(SiMe_3)_2$	diglyme, 6 equiv	1.11	
D05	**1b**/**2e**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	0.77	5.95 (quant.)[c]
D11		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	7.27 (110%)[b]	
H05		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	7.13	
H11		$NaN(SiMe_3)_2$	diglyme, 6 equiv	7.10	
D06	**1b**/**2f**	$LiN(SiMe_3)_2$	12-crown-4, 6 equiv	0	7.87
D12		$NaN(SiMe_3)_2$	15-crown-5, 0.03 equiv	7.44 (99%)[b]	
H06		$NaN(SiMe_3)_2$	HMTETA, 6 equiv	2.72	
H12		$NaN(SiMe_3)_2$	diglyme, 6 equiv	0	

[a]Ratio product/internal standard obtained by high-throughput screening; [b]isolated yield on laboratory scale (0.1 mmol); [c]yield determined by ^{1}H NMR spectroscopy of the crude reaction mixture on laboratory scale.

Representative 96 well plate for screening (5).

		LiN(SiMe$_3$)$_2$, 6 equiv 12-crown-4, CPME, rt						NaN(SiMe$_3$)$_2$, 30 mol % 15-crown-5, CPME, rt					
		2a 	**2b** 	**2c** 	**2d** 	**2e** 	**2f** 	**2a** 	**2b** 	**2c** 	**2d** 	**2e** 	**2f** 
		1	***2***	***3***	***4***	***5***	***6***	***7***	***8***	***9***	***10***	***11***	***12***
 **1a**	***A***	87 (97) **3aa**	6 **3ab**	25 **3ac**	106 (99) **3ad**	66 **3ae**	9 **3af**	96 (99) **3aa**	54 (63) **3ab**	18 **3ac**	118 (97) **3ad**	105 (99) **3ae**	91 (77) **3af**
 **1c**	***B***	61 (52) **3ca**	39 (51) **3cb**	mult. prod. **3cc**	59 (48) **3cd**	48 (61) **3ce**	55 (63) **3cf**	30 **3ca**	10 **3cb**	mult. prods. **3cc**	30 **3cd**	27 **3ce**	25 **3cf**
 **1d**	***C***	60 **3da**	74 (76) **3db**	12 **3dc**	101 **3dd**	0 **3de**	82 (88) **3df**	6 **3da**	44 **3db**	8 **3dc**	101 (99) **3dd**	109 (99) **3de**	103 **3df**
 **1b**	***D***	82 **3ba**	76 **3bb**	81 (78) **3bc**	92 **3bd**	10 **3be**	0 **3bf**	26 **3ba**	73 (99) **3bb**	21 **3bc**	112 (99) **3bd**	99 (94) **3be**	73 (99) **3bf**
 **1a**	***E***	80 (86) **3aa**	25 **3ab**	4 **3ac**	2 **3ad**	2 **3ae**	36 **3af**	88 (78) **3aa**	12 **3ab**	0 **3ac**	28 **3ad**	23 **3ae**	19 **3af**
 **1c**	***F***	8 **3ca**	2 **3cb**	0 **3cc**	6 **3cd**	0 **3ce**	0 **3cf**	1 **3ca**	0 **3cb**	0 **3cc**	3 **3cd**	4 **3ce**	1 **3cf**
 **1d**	***G***	73 (99) **3da**	47 **3db**	0 **3dc**	58 **3dd**	83 **3de**	56 **3df**	49 **3da**	2 **3db**	0 **3dc**	41 **3dd**	32 **3de**	32 **3df**
 **1b**	***H***	37 **3ba**	31 **3bb**	19 **3bc**	66 **3bd**	83 **3be**	27 **3bf**	95 (99) **3ba**	87 **3bb**	mult. prods. **3bc**	16 **3bd**	97 **3be**	0 **3bf**
		NaN(SiMe$_3$)$_2$, 6 equiv HMTETA, CPME, rt						NaN(SiMe$_3$)$_2$, 6 equiv diglyme, CPME, rt					

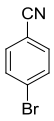
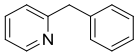
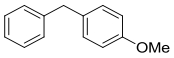
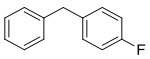
Numbers in black correspond to yield based on ^{1}H NMR of the crude mixture; numbers in blue correspond to HPLC calibrated yields from ^{1}H NMR; numbers in brackets correspond to isolated yields on laboratory scale.

(6) Challenging Ar-Br Screening with 1a

Well	Condition	Substrate	Prod/IS[a]
A01	$LiN(SiMe_3)_2$/12-crown-4, 6 equiv	**2g**	4.18 (67%)[b]
A02		**2h**	0
A03		**2i**	3.35 (75%)[b]
A04		**2j**	0
A05		**2k**	0
A06		**2l**	0
B01	$NaN(SiMe_3)_2$/15-crown-5, 0.03 equiv	**2g**	2.45 (34%)[b]
B02		**2h**	0
B03		**2i**	1.16 (37%)[b]
B04		**2j**	0
B05		**2k**	0
B06		**2l**	0
C01	$NaN(SiMe_3)_2$/HMTETA, 6 equiv	**2g**	0
C02		**2h**	0
C03		**2i**	0.71
C04		**2j**	0
C05		**2k**	0
C06		**2l**	0
D01	$NaN(SiMe_3)_2$/diglyme, 6 equiv	**2g**	0
D02		**2h**	0
D03		**2i**	0.74
D04		**2j**	0
D05		**2k**	0
D06		**2l**	0
Control	$KN(SiMe_3)_2$	**2g**	1.15 (16%)[b]
		2h	0
		2i	0
		2j	0
		2k	0
		2l	0

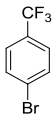
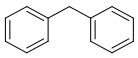
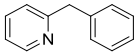
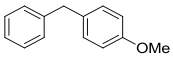
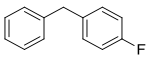
[a]Ratio product/internal standard obtained by high-throughput screening; [b]isolated yield on laboratory scale.

(7) Screening of compounds 2g and 2i with diarylmethanes 1b-1d

	Ar-Br	**2g** 		**2i** 	
Diarylmethanes	**Conditions**	**Prod/IS[a]**	**AY (%)[b]**	**Prod/IS[a]**	**AY (%)[b]**
 **1b**	$LiN(SiMe_3)_2$, 6 equiv 12-C-4	4.86	92 (86)	3.76	95
	$NaN(SiMe_3)_2$, 30 mol % 15-C-5	4.38	83	3.58	83
	$NaN(SiMe_3)_2$, 6 equiv HMTETA	4.03	69	4.29	107 (96)
	$NaN(SiMe_3)_2$, 6 equiv diglyme	3.68	54	1.69	44
	CONTROL - $KN(SiMe_3)_2$	2.88	20	2.80	74
	Product	**3bg**		**3bi**	
 **1c**	$LiN(SiMe_3)_2$, 6 equiv 12-C-4	0.88	0	0.67	<6
	$NaN(SiMe_3)_2$, 30 mol % 15-C-5	0.86	0	0.64	0
	$NaN(SiMe_3)_2$, 6 equiv HMTETA	NR[c]	0	NR[c]	0
	$NaN(SiMe_3)_2$, 6 equiv diglyme	NR[c]	0	NR[c]	0
	CONTROL - $KN(SiMe_3)_2$	2.35	0	0.29	0
	Product	**3cg**		**3ci**	
 **1d**	$LiN(SiMe_3)_2$, 6 equiv 12-C-4	0	0	3.07	83 (85)
	$NaN(SiMe_3)_2$, 30 mol % 15-C-5	4.48	18 (26)	0.78	60
	$NaN(SiMe_3)_2$, 6 equiv HMTETA	0.74	3	0.23	0
	$NaN(SiMe_3)_2$, 6 equiv diglyme	NR[c]	0	NR[c]	0
	CONTROL - $KN(SiMe_3)_2$	3.82	14	0.23	0
	Product	**3dg**		**3di**	

[a]Ratio product/internal standard obtained by high-throughput screening; [b]results correspond to assay yields based on ^{1}H NMR of the crude mixture in the high-throughput screening. Control results correspond to ^{1}H NMR yields from the crude mixture on laboratory scale. Numbers in brackets correspond to isolated yields on laboratory scale; [c]NR = no reaction, starting material unreacted.

(8) Screening of compounds 2c with diarylmethanes 1a-1d using 2 equivalents of base and 3 equivalents of diarylmethane

	Ar-Br	**2c** 	
Diarylmethanes	**Conditions**	**Prod/IS[a]**	**AY (%)[b]**
 **1a**	$LiN(SiMe_3)_2$, 6 equiv 12-C-4	2.85	25
	$NaN(SiMe_3)_2$, 30 mol % 15-C-5	2.10	18
	$NaN(SiMe_3)_2$, 6 equiv HMTETA	0.50	4
	$NaN(SiMe_3)_2$, 6 equiv diglyme	NR	0
	CONTROL - $KN(SiMe_3)_2$	6.90	66
	Product	**3ac**	
 **1b**	$LiN(SiMe_3)_2$, 6 equiv 12-C-4	5.06	81 (78)
	$NaN(SiMe_3)_2$, 30 mol % 15-C-5	1.29	21
	$NaN(SiMe_3)_2$, 6 equiv HMTETA	1.24	19
	$NaN(SiMe_3)_2$, 6 equiv diglyme	multiple products	-[c]
	CONTROL - $KN(SiMe_3)_2$	NR	<5
	Product	**3bc**	
 **1c**	$LiN(SiMe_3)_2$, 6 equiv 12-C-4	multiple products	-[c]
	$NaN(SiMe_3)_2$, 30 mol % 15-C-5	multiple products	-[c]
	$NaN(SiMe_3)_2$, 6 equiv HMTETA	NR[d]	-[c]
	$NaN(SiMe_3)_2$, 6 equiv diglyme	NR[d]	-[c]
	CONTROL - $KN(SiMe_3)_2$	multiple products	-[c]
	Product	**3cc**	
 **1d**	$LiN(SiMe_3)_2$, 6 equiv 12-C-4	1.24	12
	$NaN(SiMe_3)_2$, 30 mol % 15-C-5	0.85	8
	$NaN(SiMe_3)_2$, 6 equiv HMTETA	NR[d]	0
	$NaN(SiMe_3)_2$, 6 equiv diglyme	NR[d]	0
	CONTROL - $KN(SiMe_3)_2$	2.84	40
	Product	**3dc**	

[a]Ratio product/internal standard obtained by high-throughput screening; [b]results correspond o assay yields based on ^{1}H NMR of the crude mixture in the high-throughput screening. Control results correspond to ^{1}H NMR yields from the crude mixture on laboratory scale. Numbers in brackets correspond to isolated yields on laboratory scale; [c]not determined; [d]NR = no reaction, starting material unreacted.

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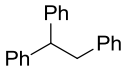
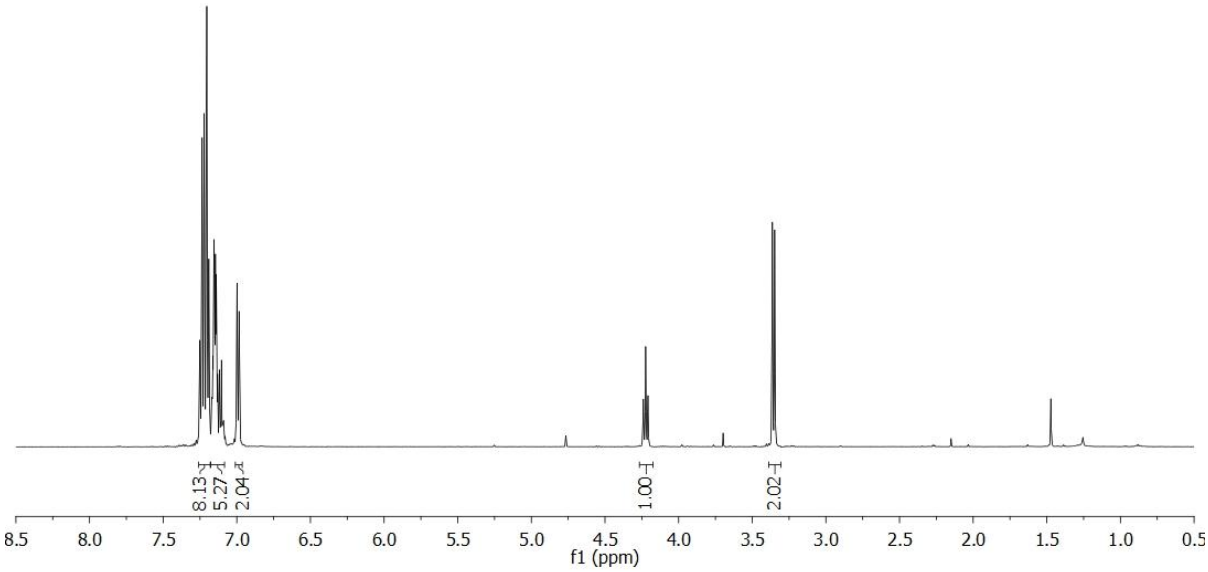
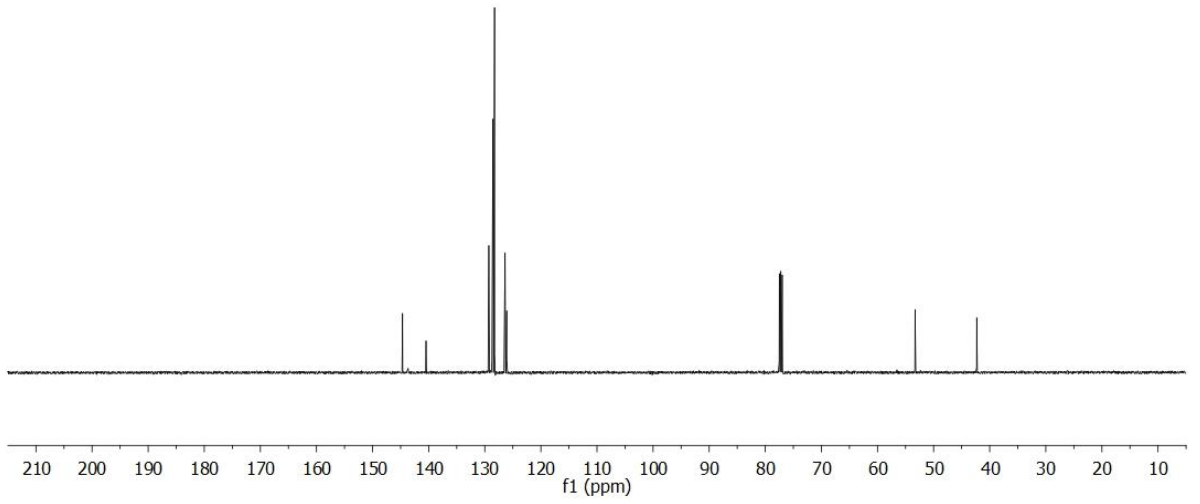
NMR Spectra.

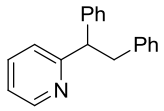
1,1,2-triphenylethane

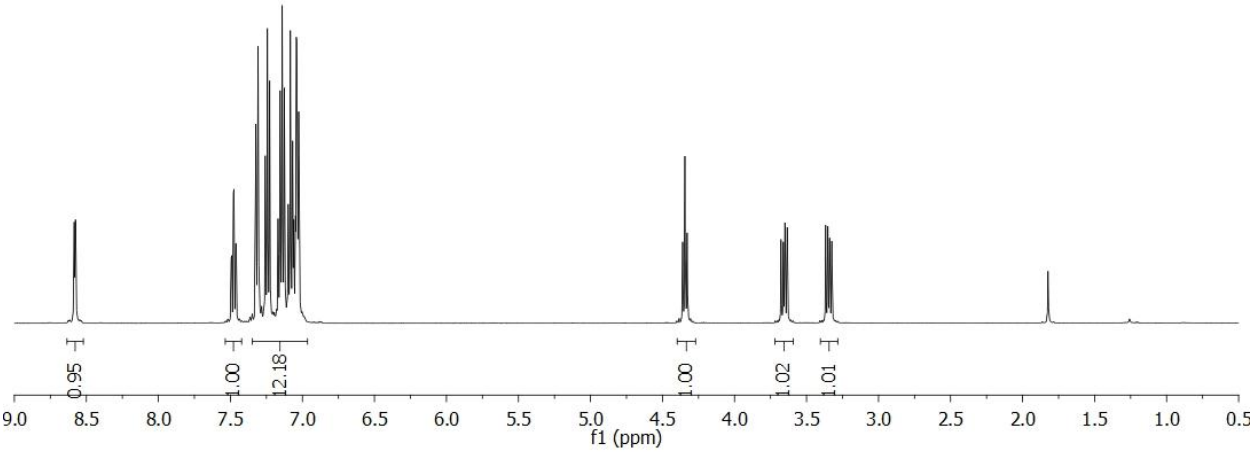


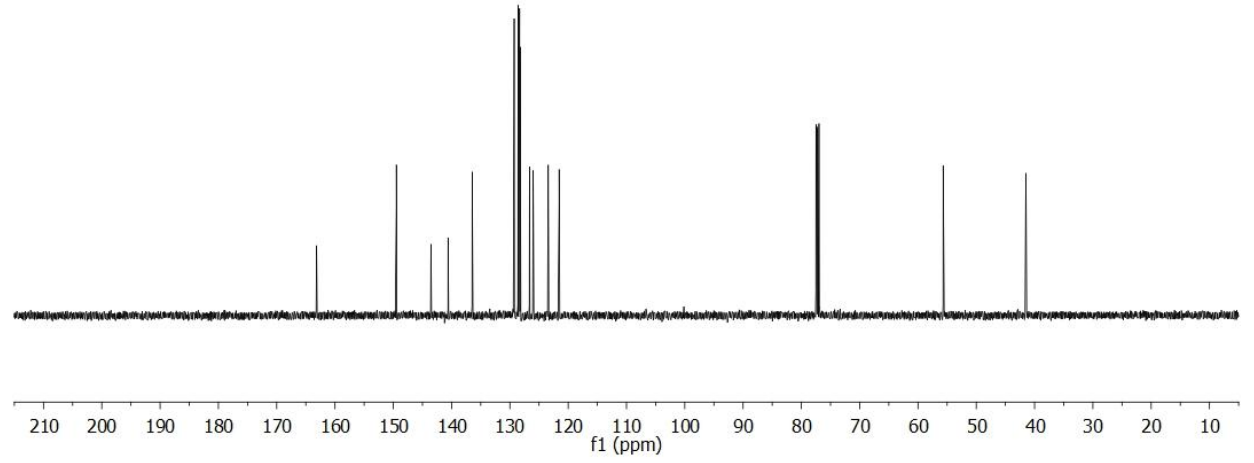




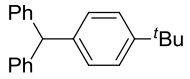
2-(1,2-diphenylethyl)pyridine

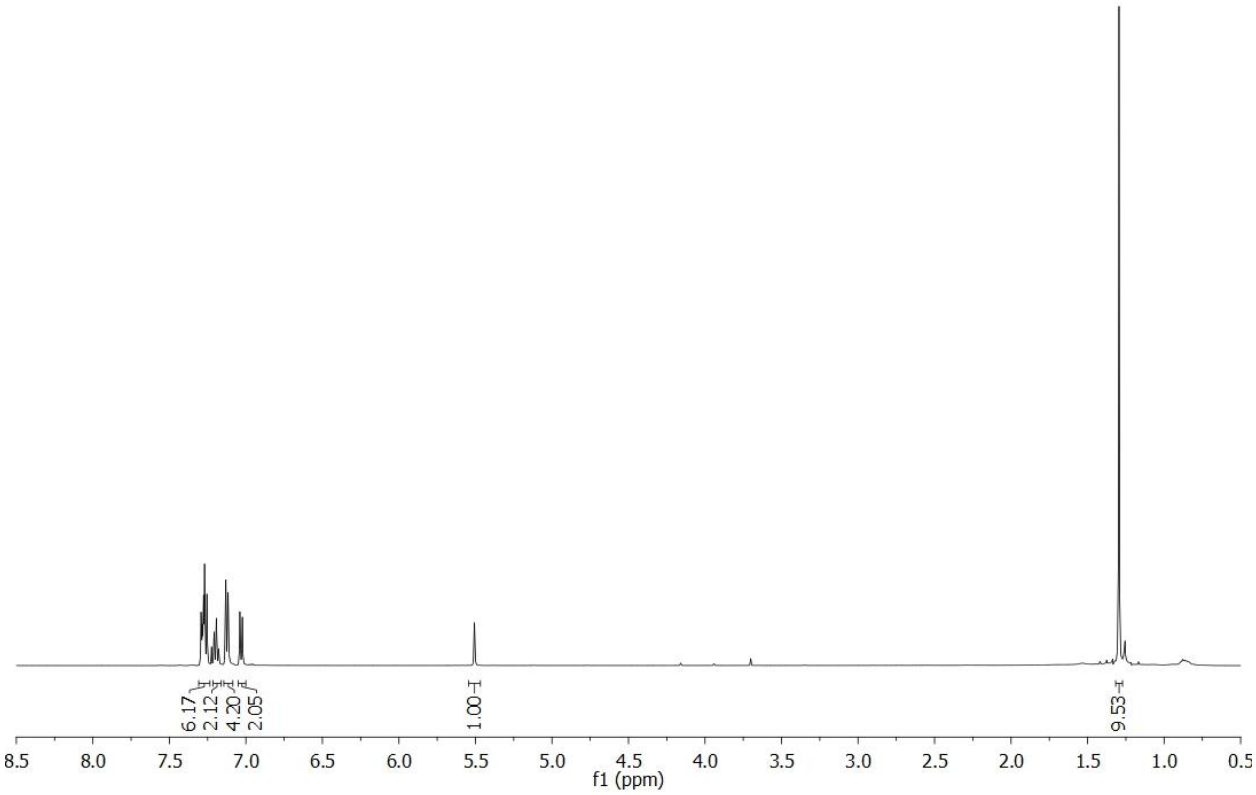


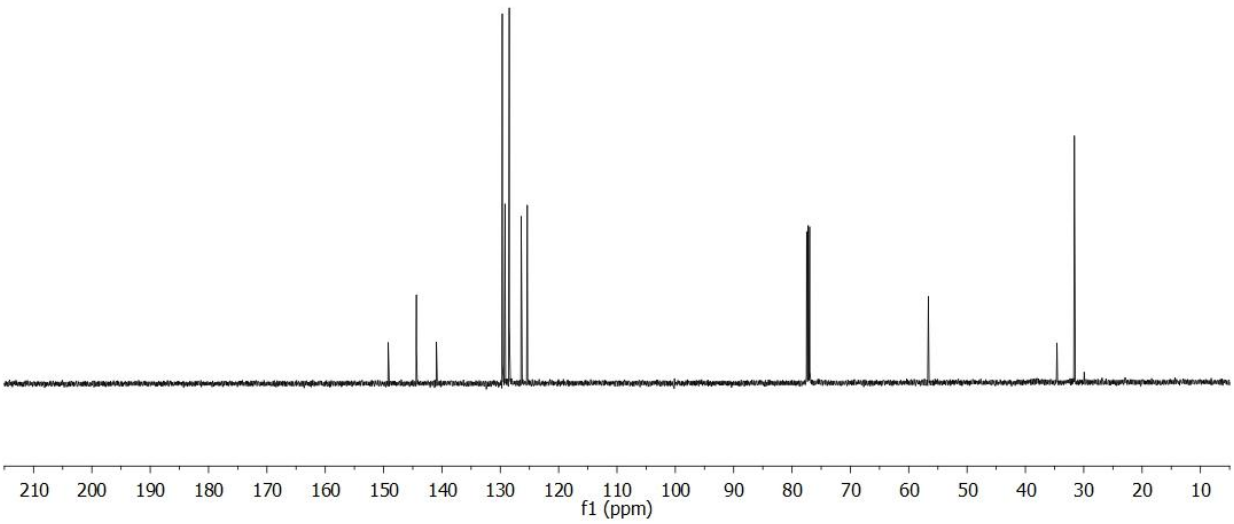




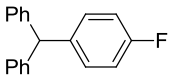
3aa – (4-*tert*-butylphenyl)diphenylmethane

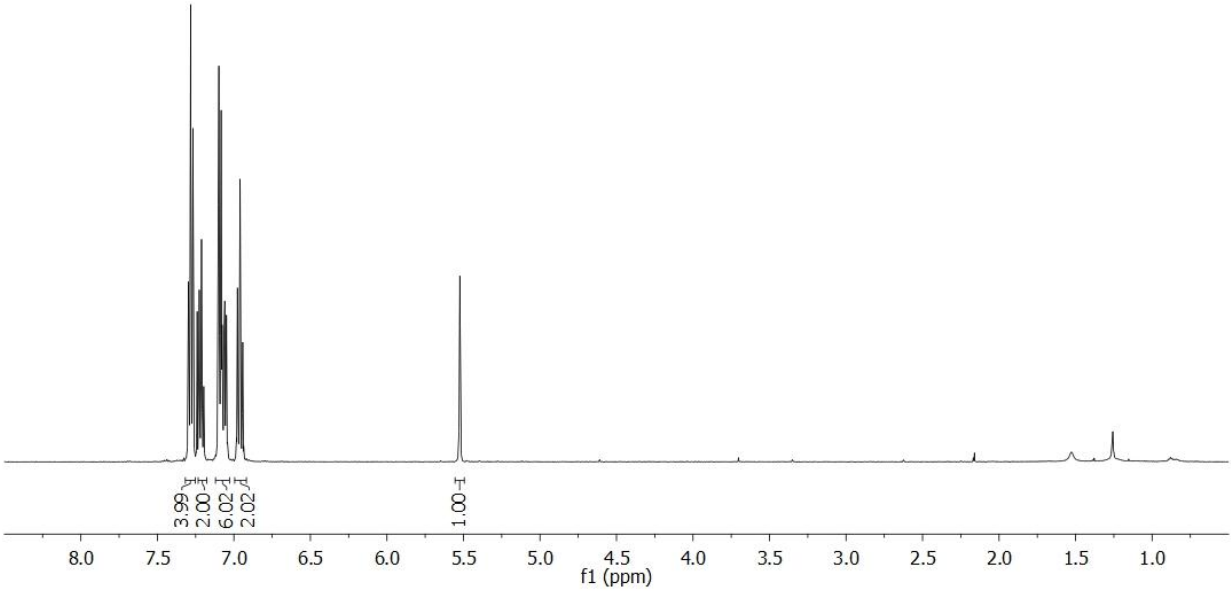


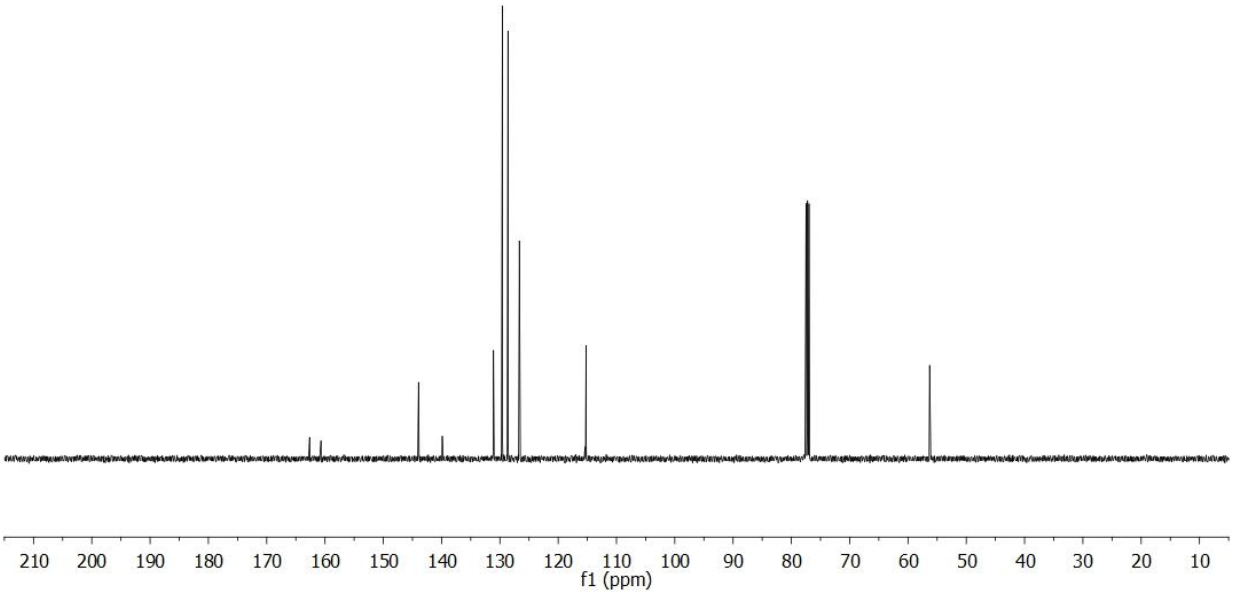




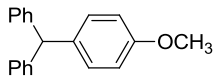
3ab – (4-fluorophenyl)diphenylmethane

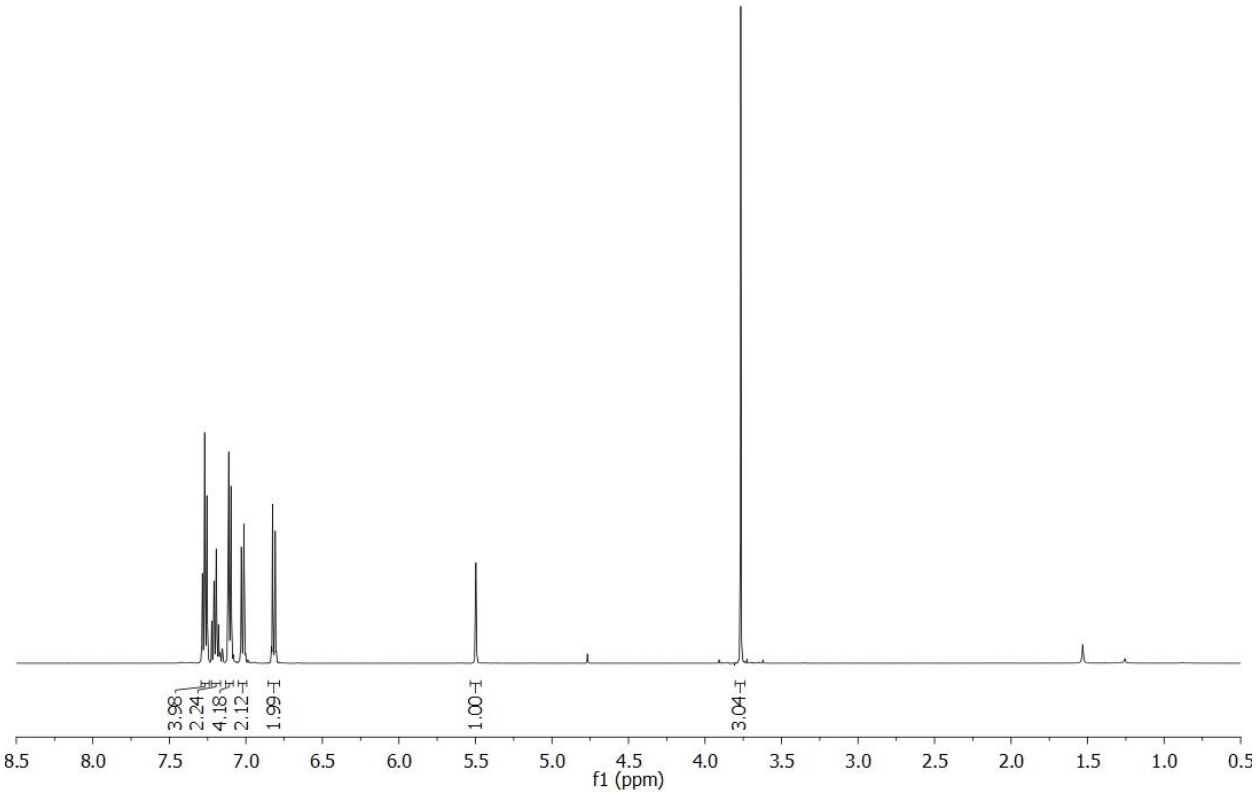


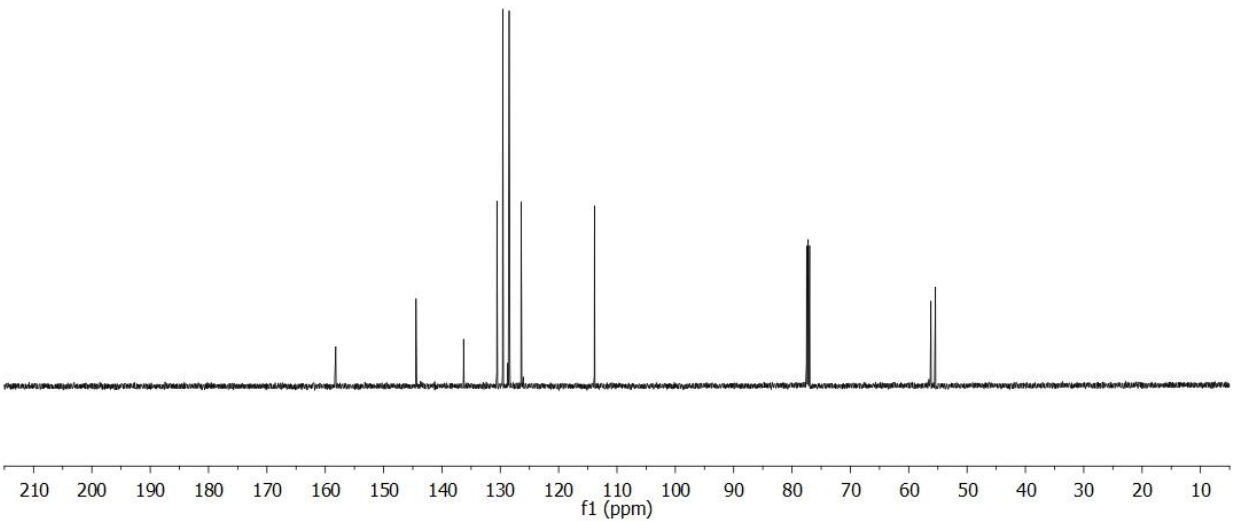




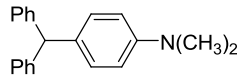
3ad – (4-methoxyphenyl)diphenylmethane

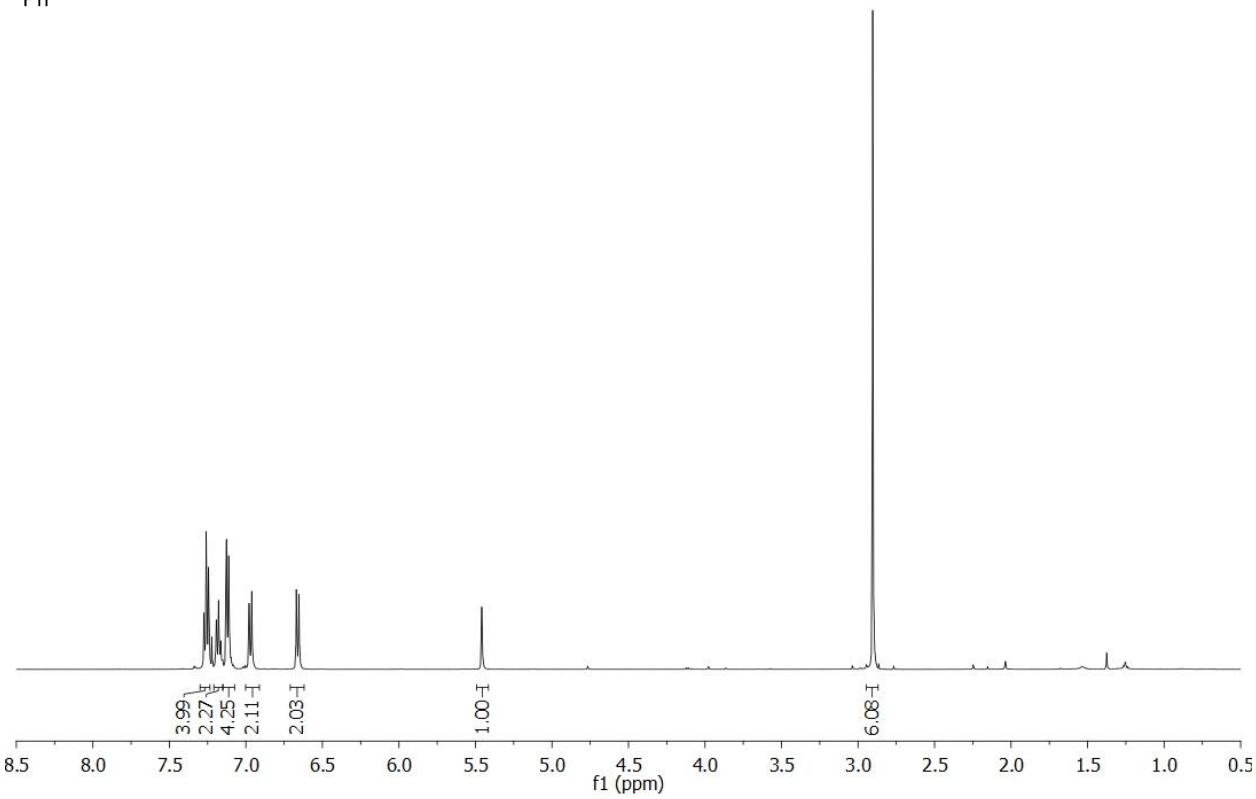


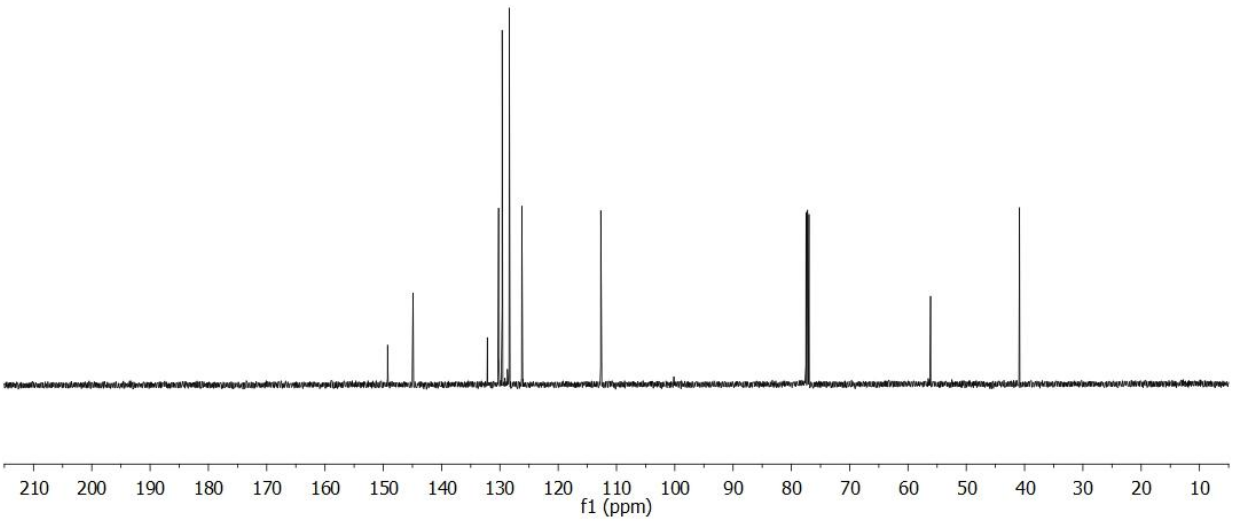




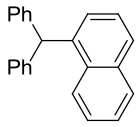
3ae – (*N*,*N*-dimethylaminophenyl)diphenylmethane

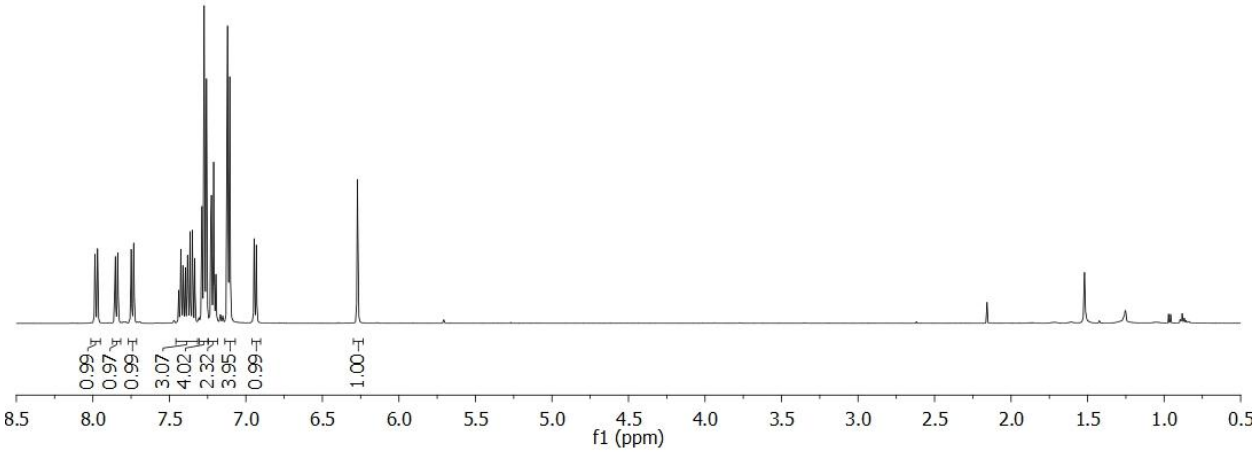


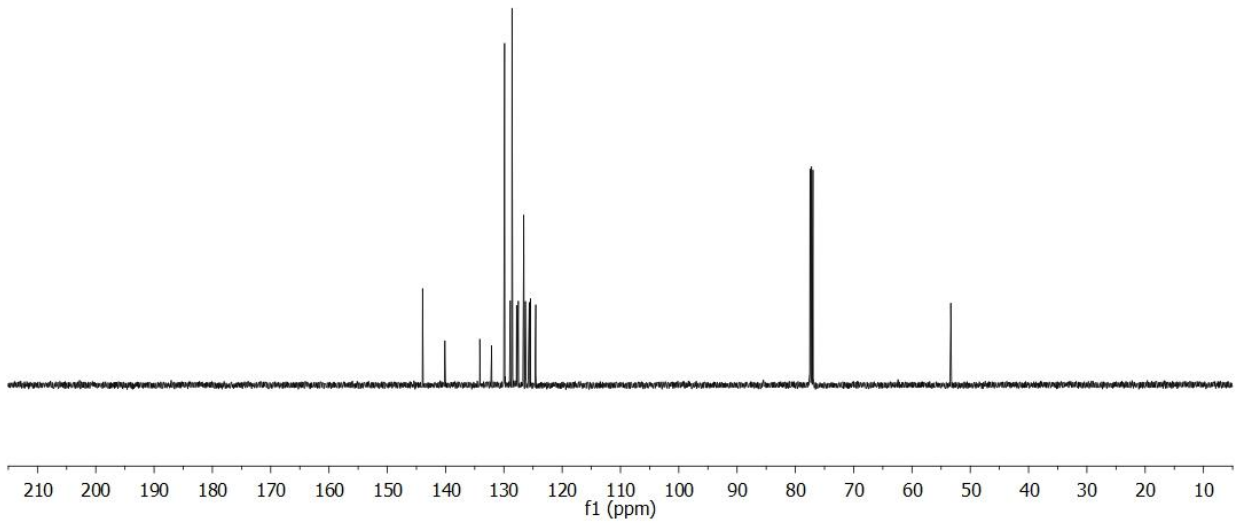




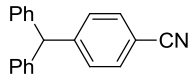
3af – (1-naphthyl)diphenylmethane

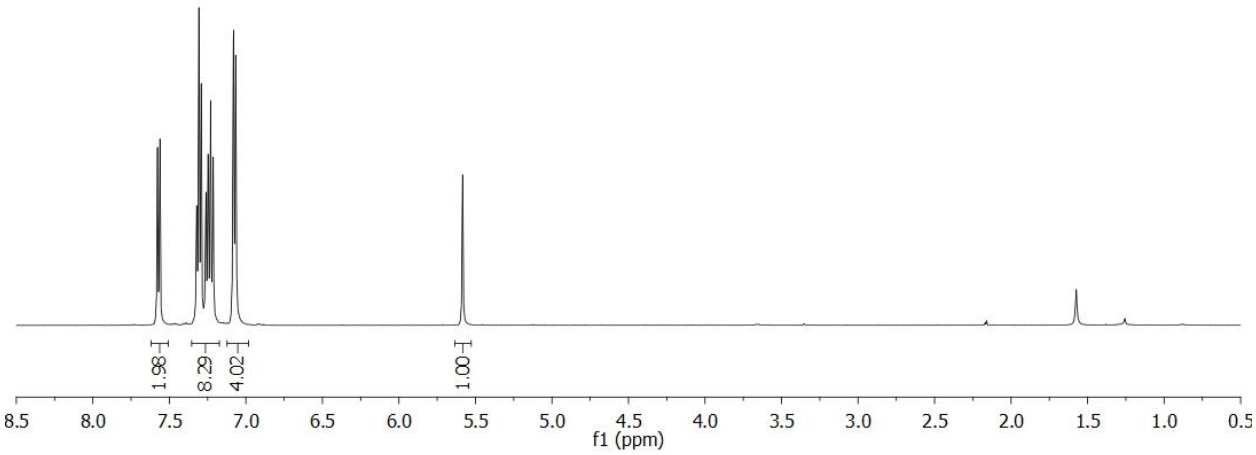


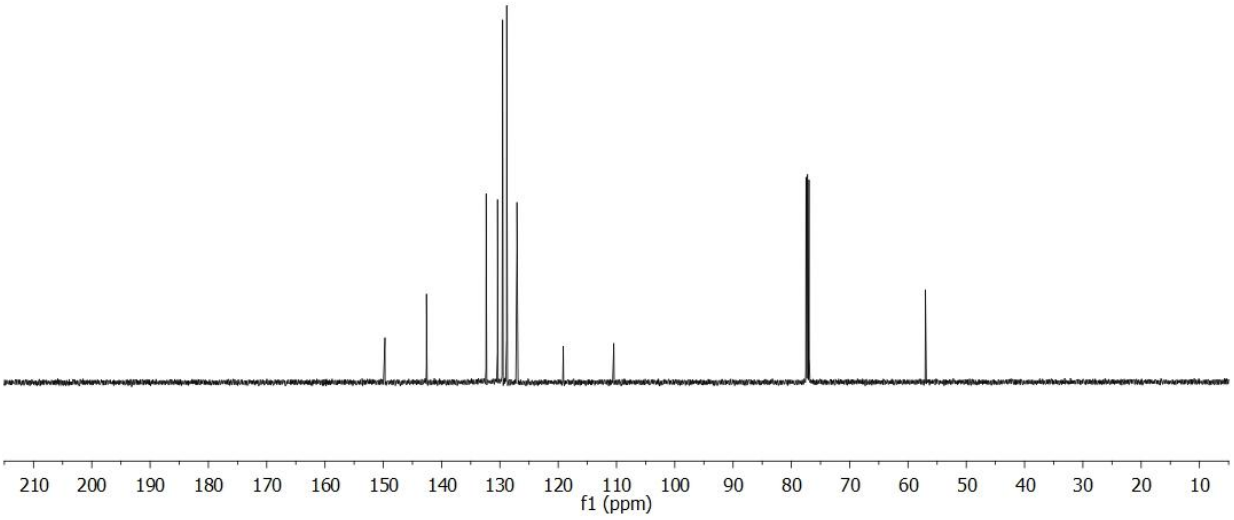




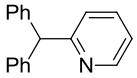
3ag – (4-cyanophenyl)diphenylmethane

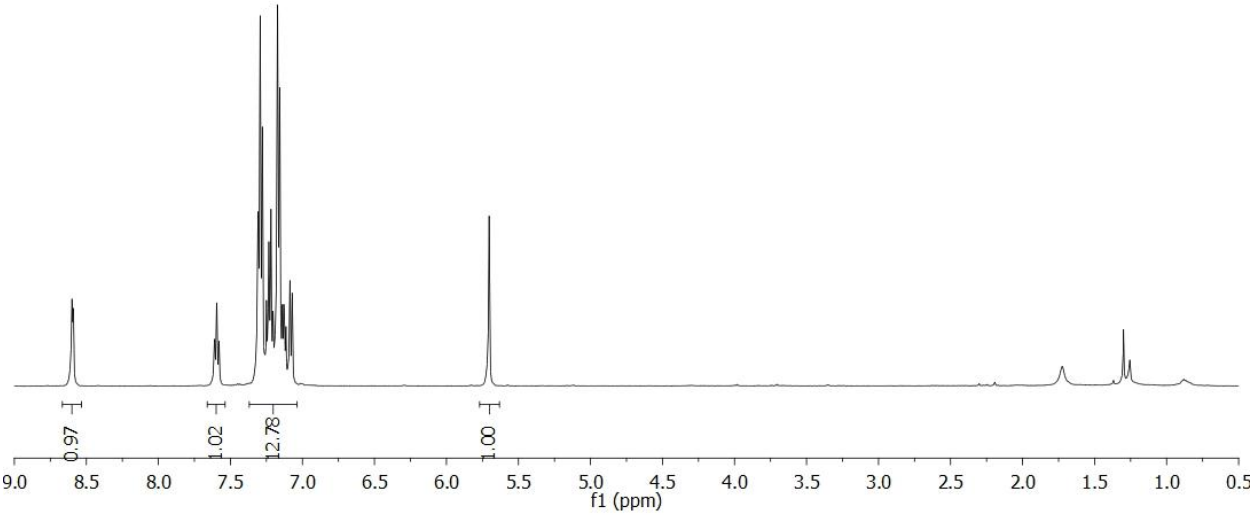


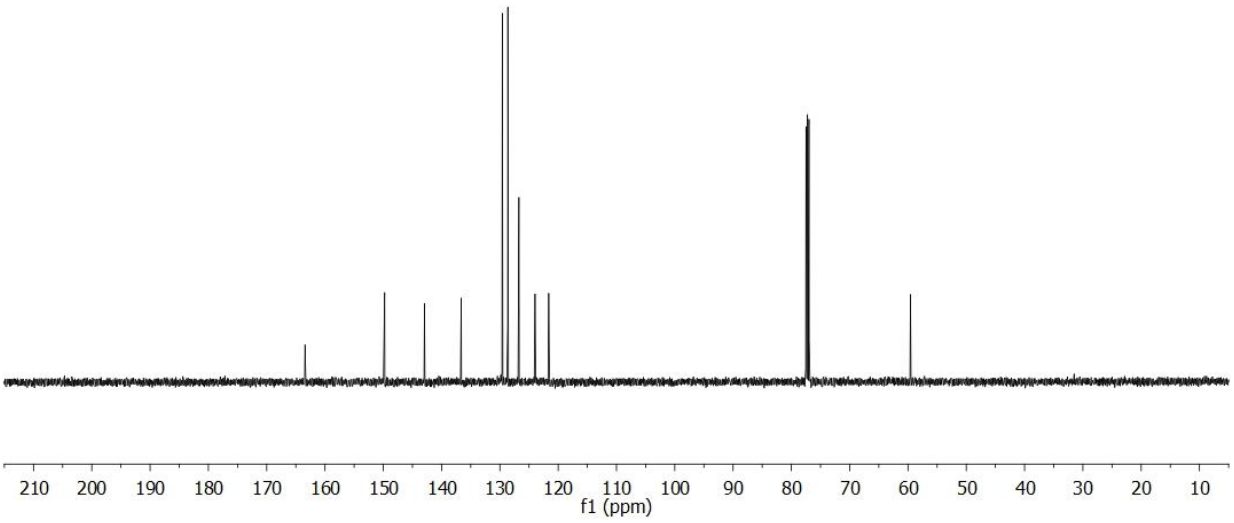




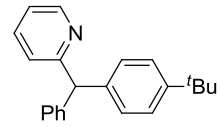
3ai – (2-pyridyl)diphenylmethane

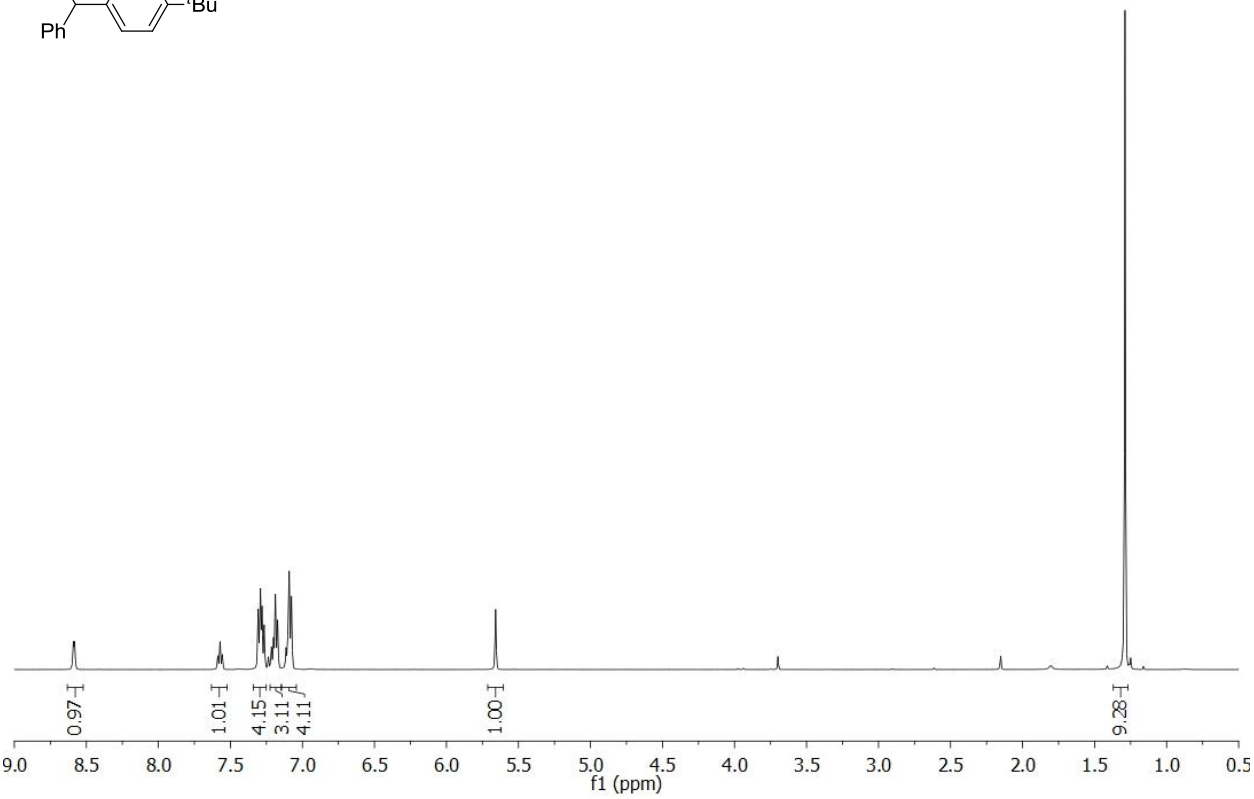


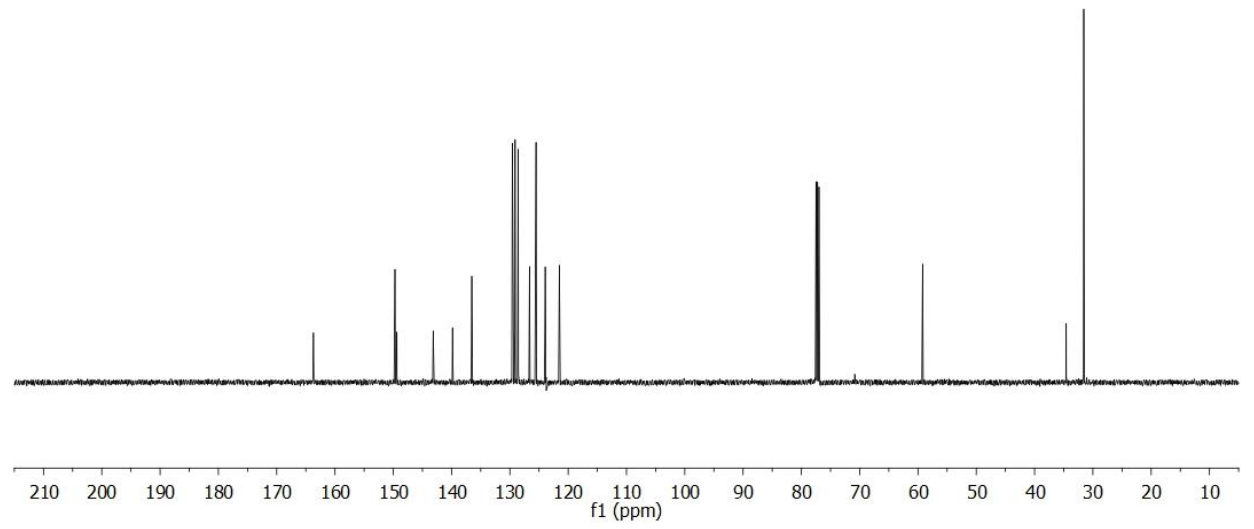




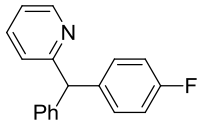
3ba – (4-*tert*-butylphenyl)(2-pyridyl)phenylmethane

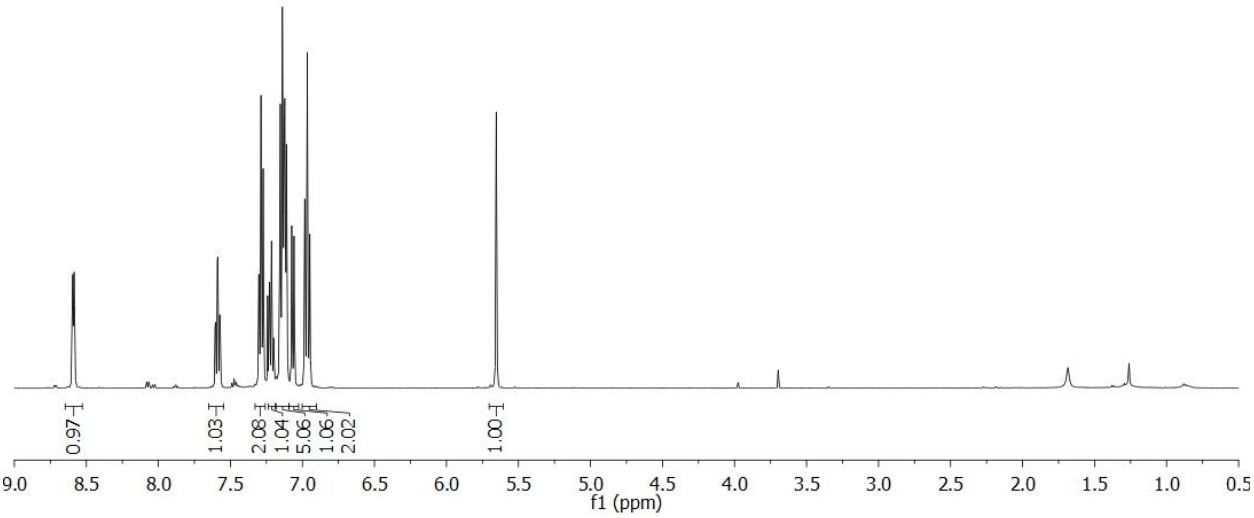


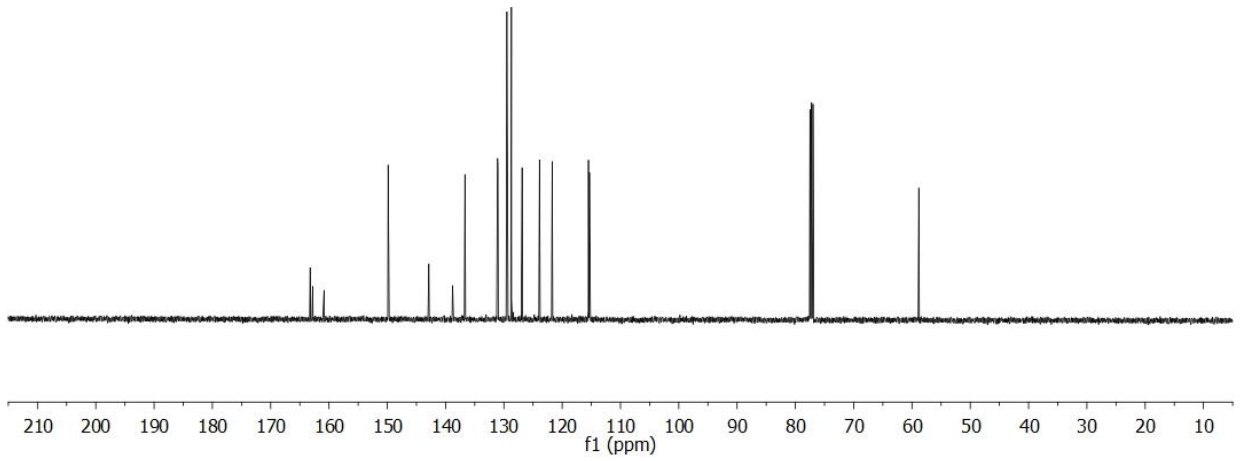




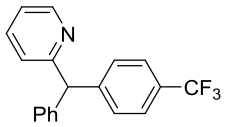
3bb – (4-fluorophenyl)(2-pyridyl)phenylmethane

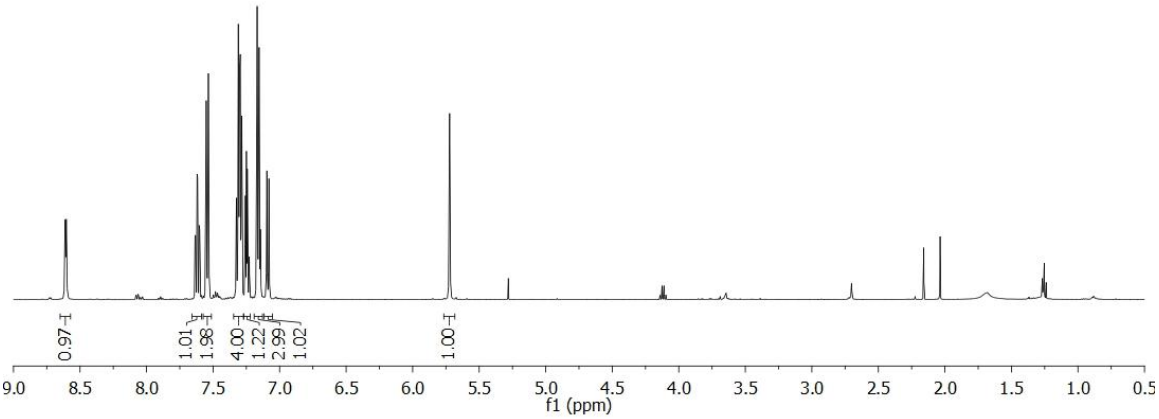


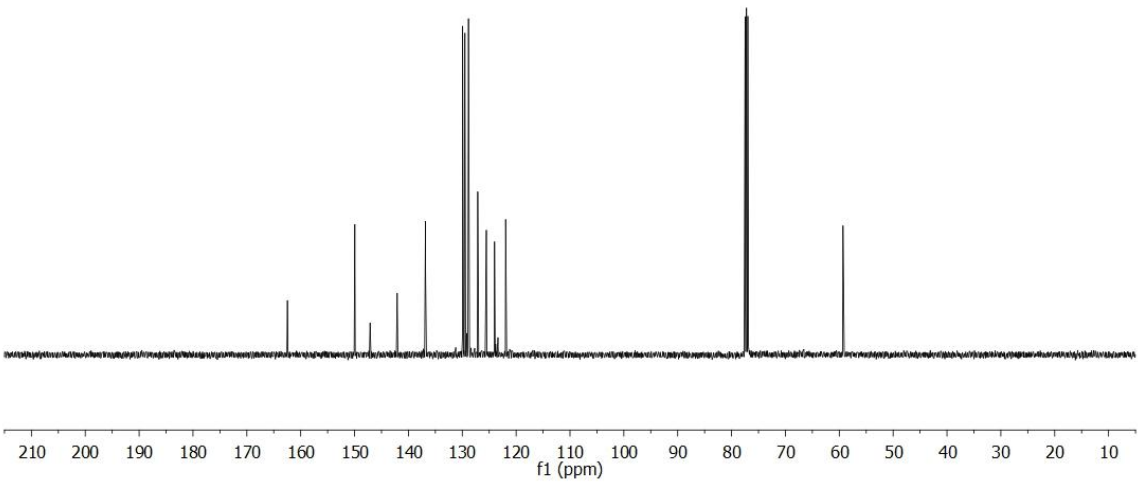




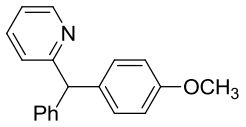
3bc – (4-trifluoromethylphenyl)(2-pyridyl)phenylmethane

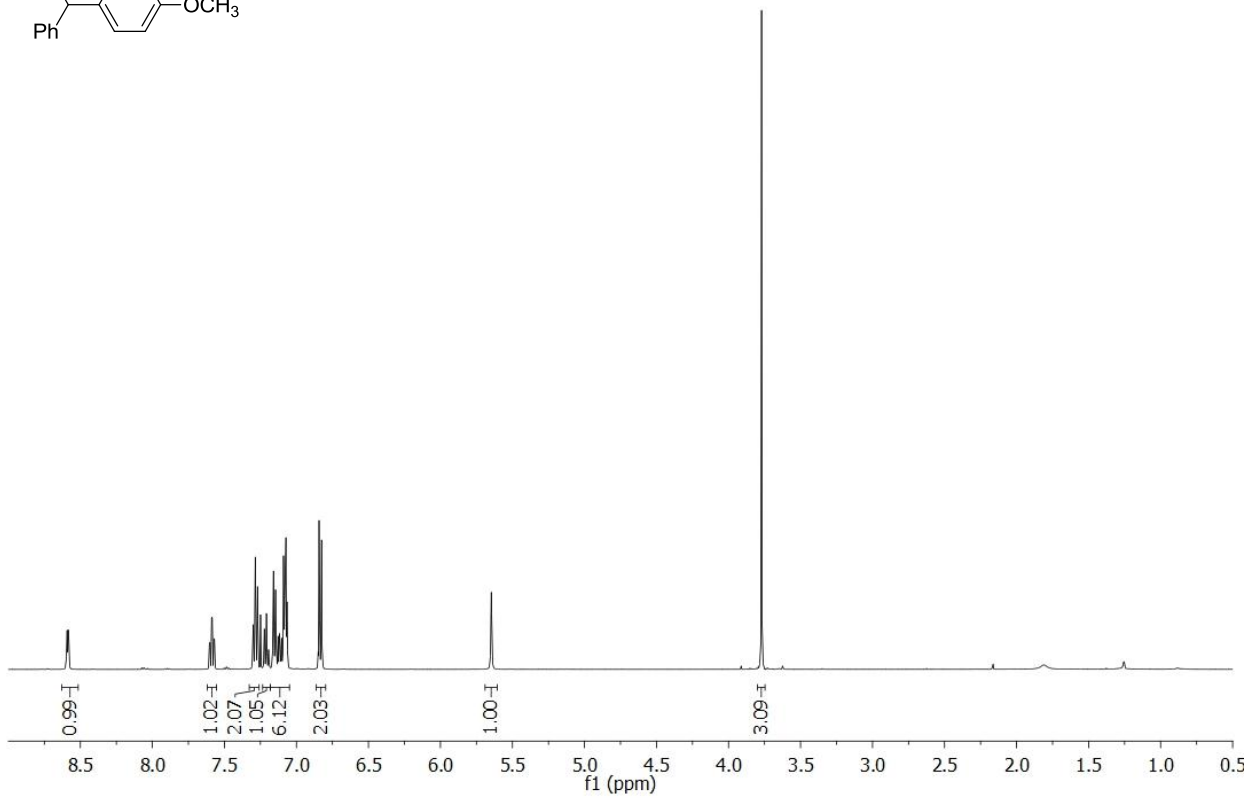


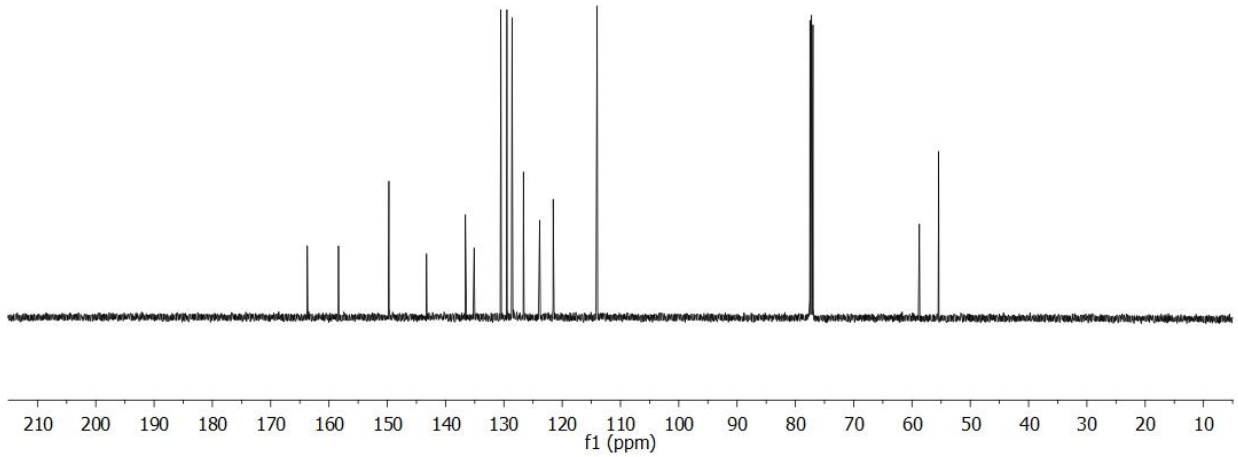




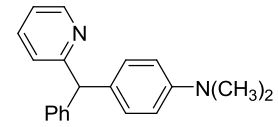
3bd – (4-methoxyphenyl)(2-pyridyl)phenylmethane

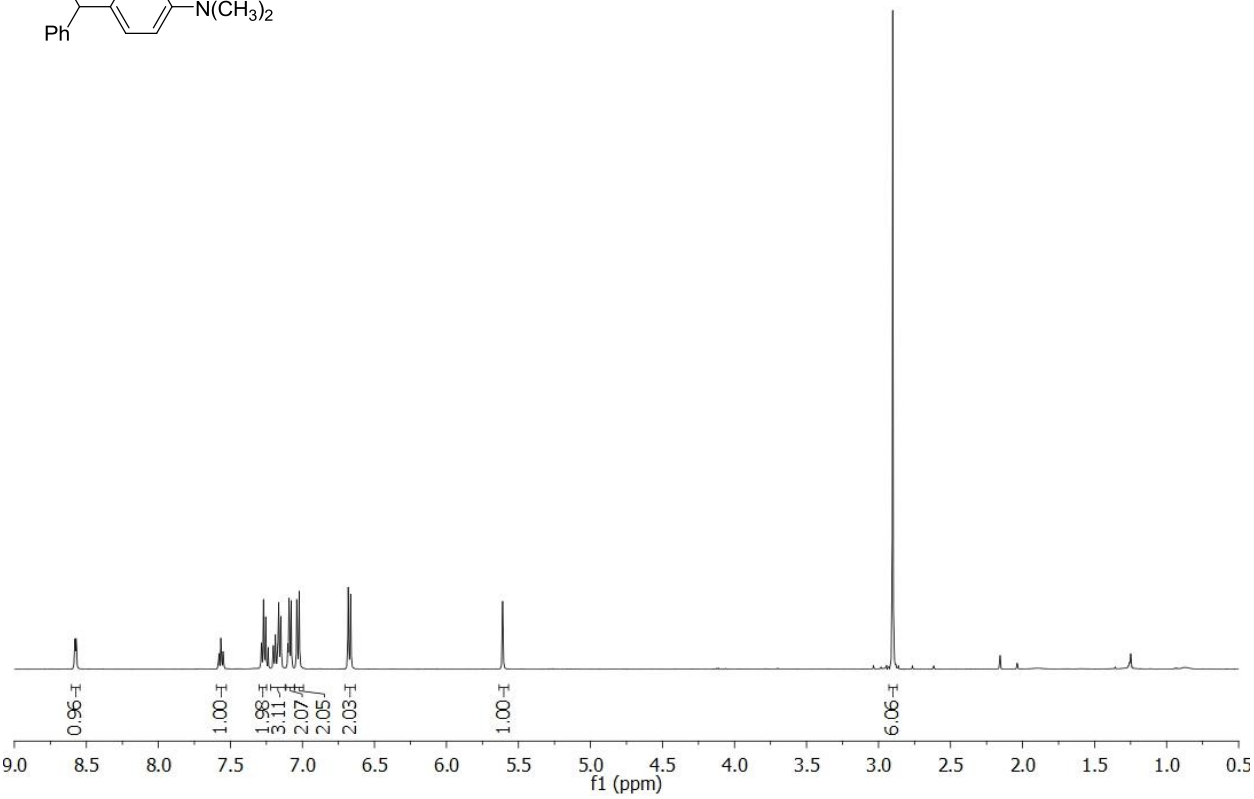


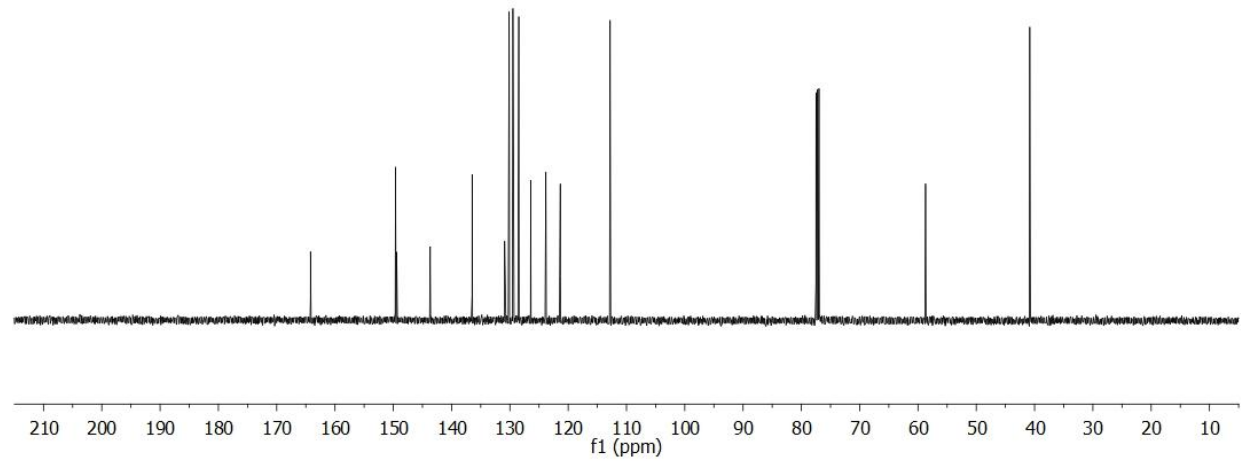




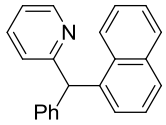
3be – (4-*N*,*N*-dimethylaminophenyl)(2-pyridyl)phenylmethane

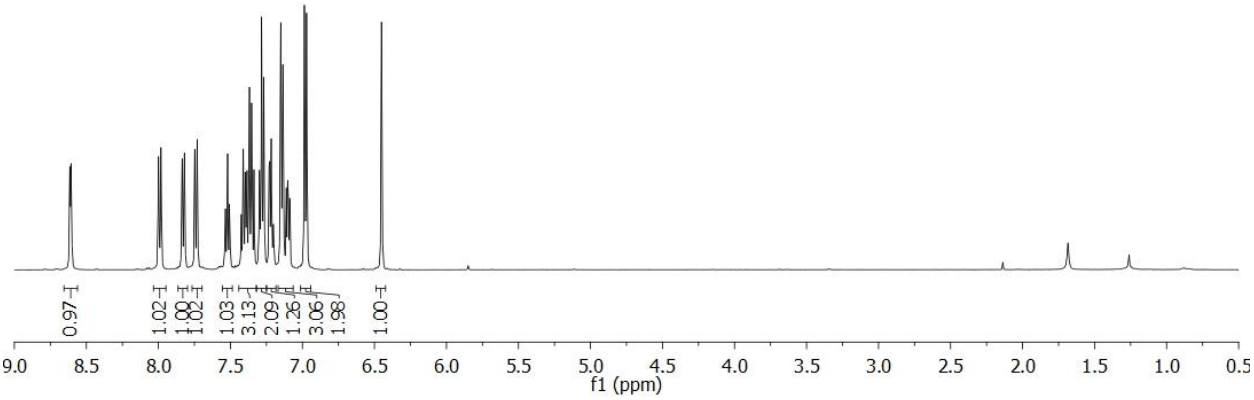


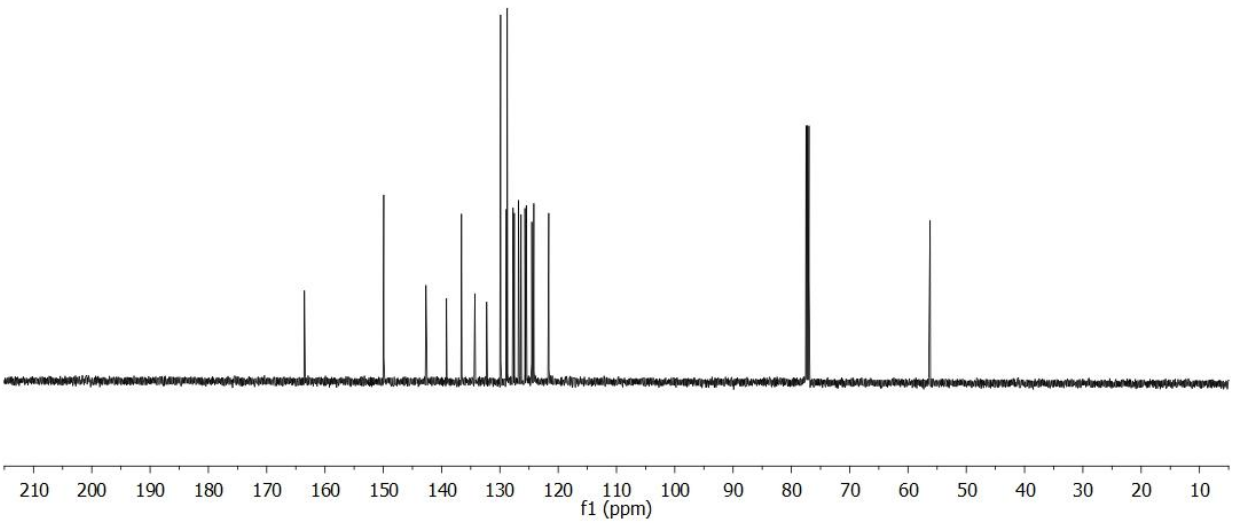




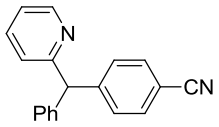
3bf – (1-naphthyl)(2-pyridyl)phenylmethane

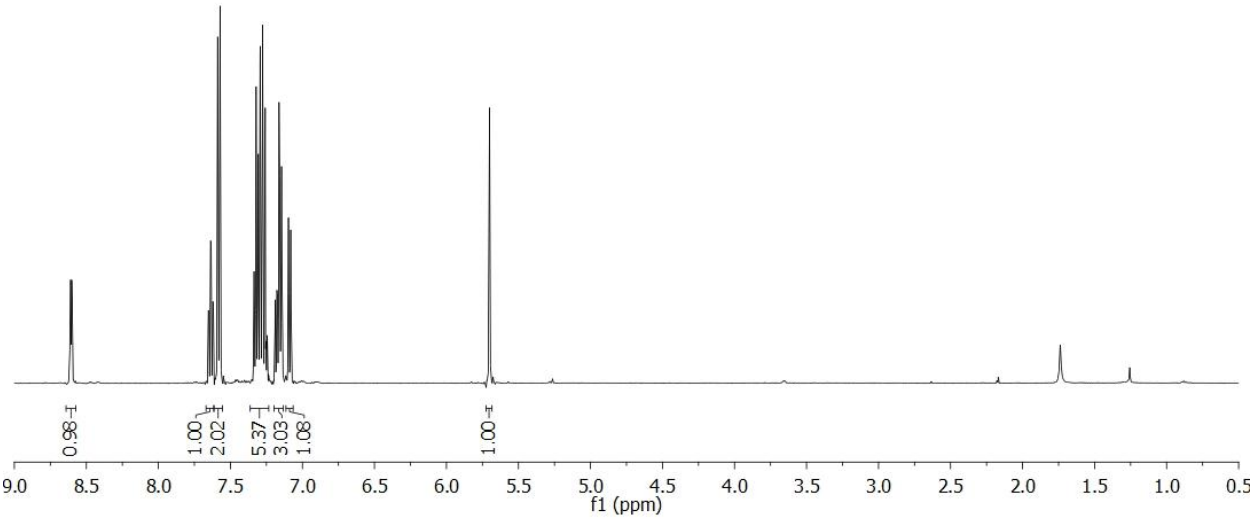


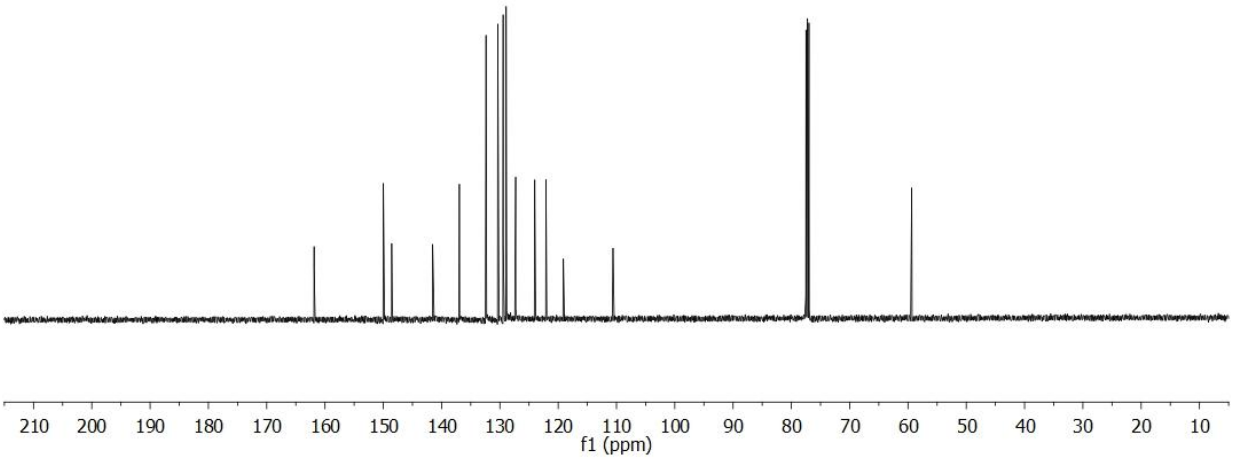




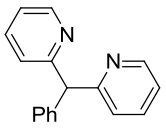
3bg – (4-cyanophenyl)(2-pyridyl)phenylmethane

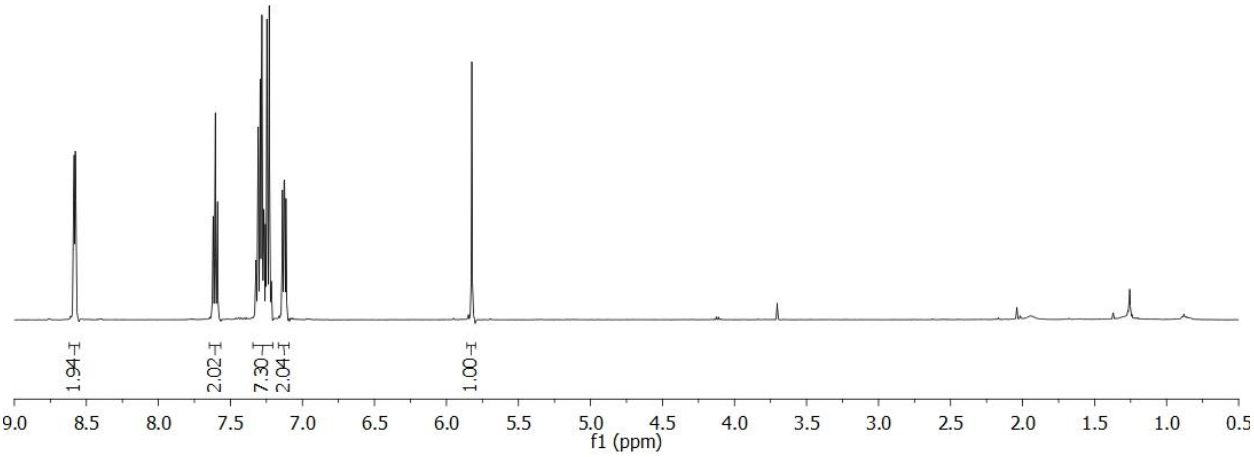


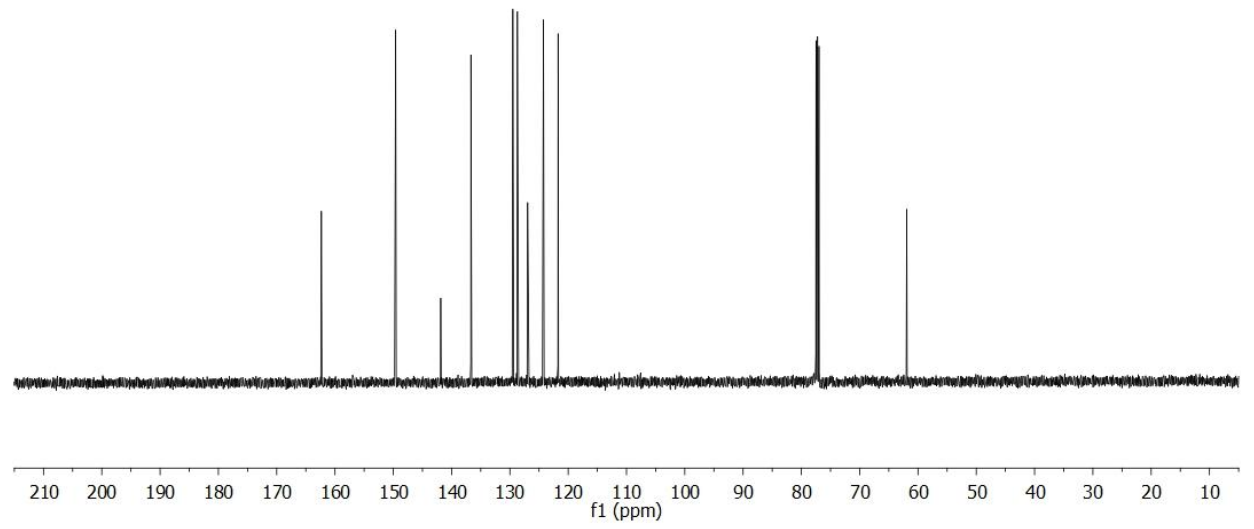




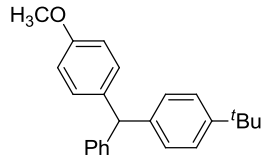
3bi – 2,2'-(phenylmethylene)dipyridine

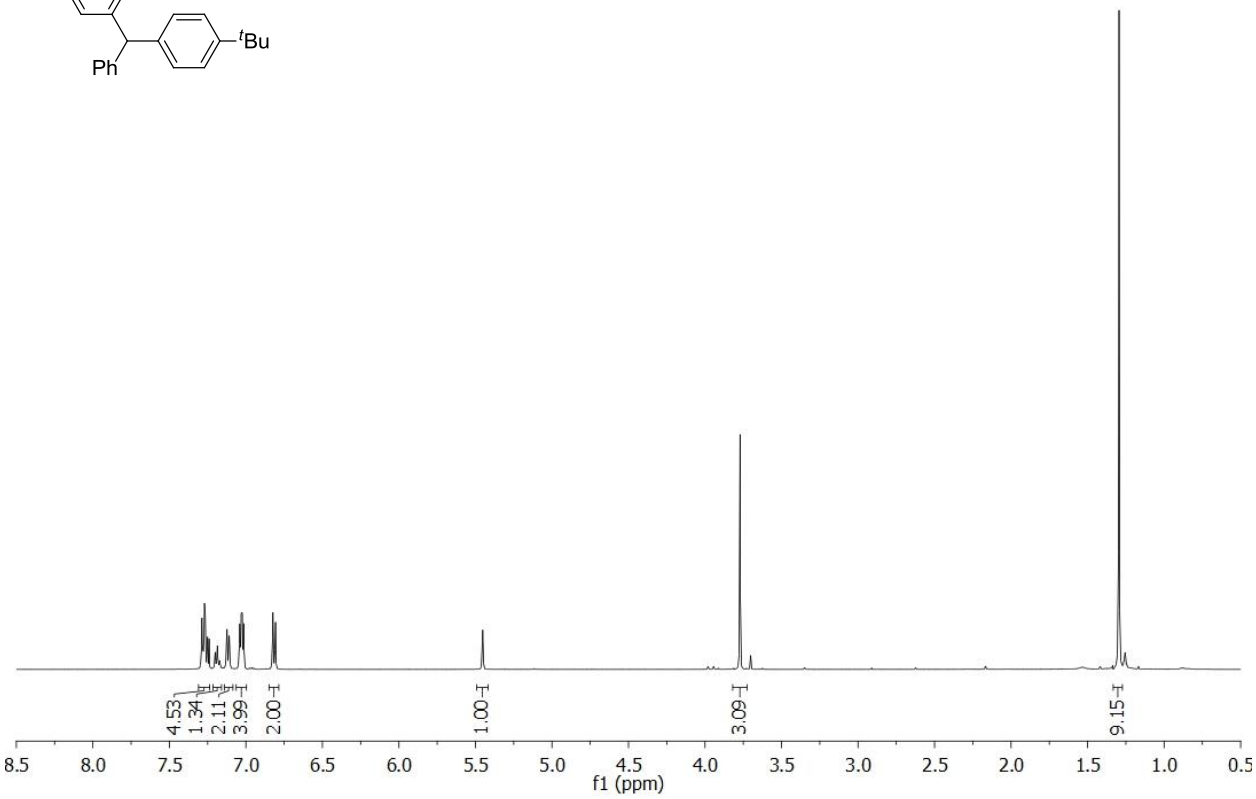


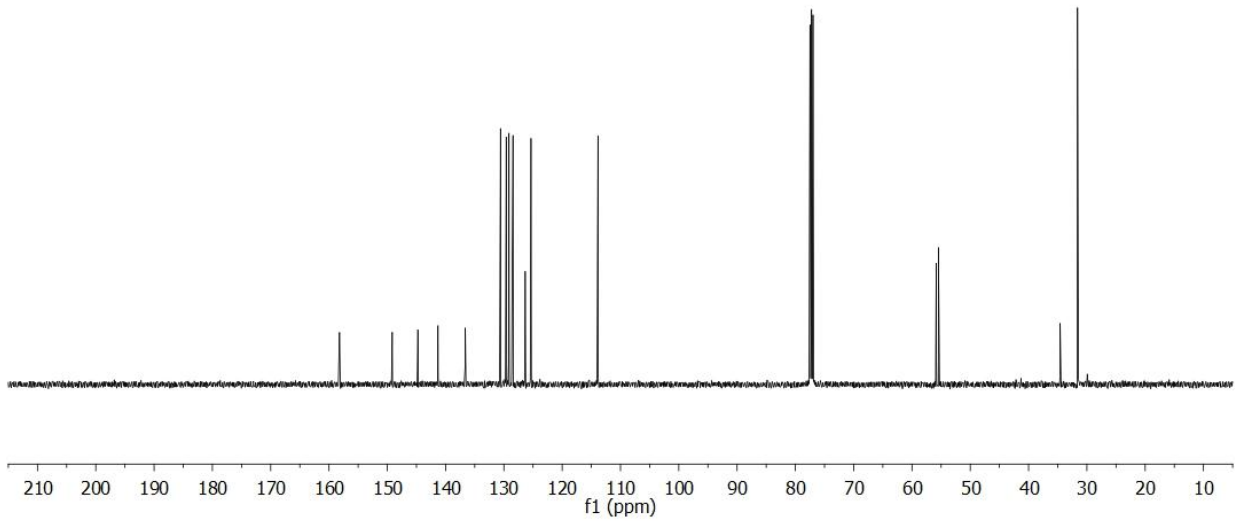




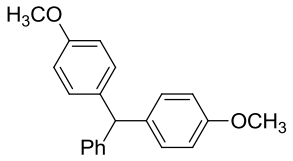
3ca – (4-*tert*-butylphenyl)(4-methoxyphenyl)phenylmethane

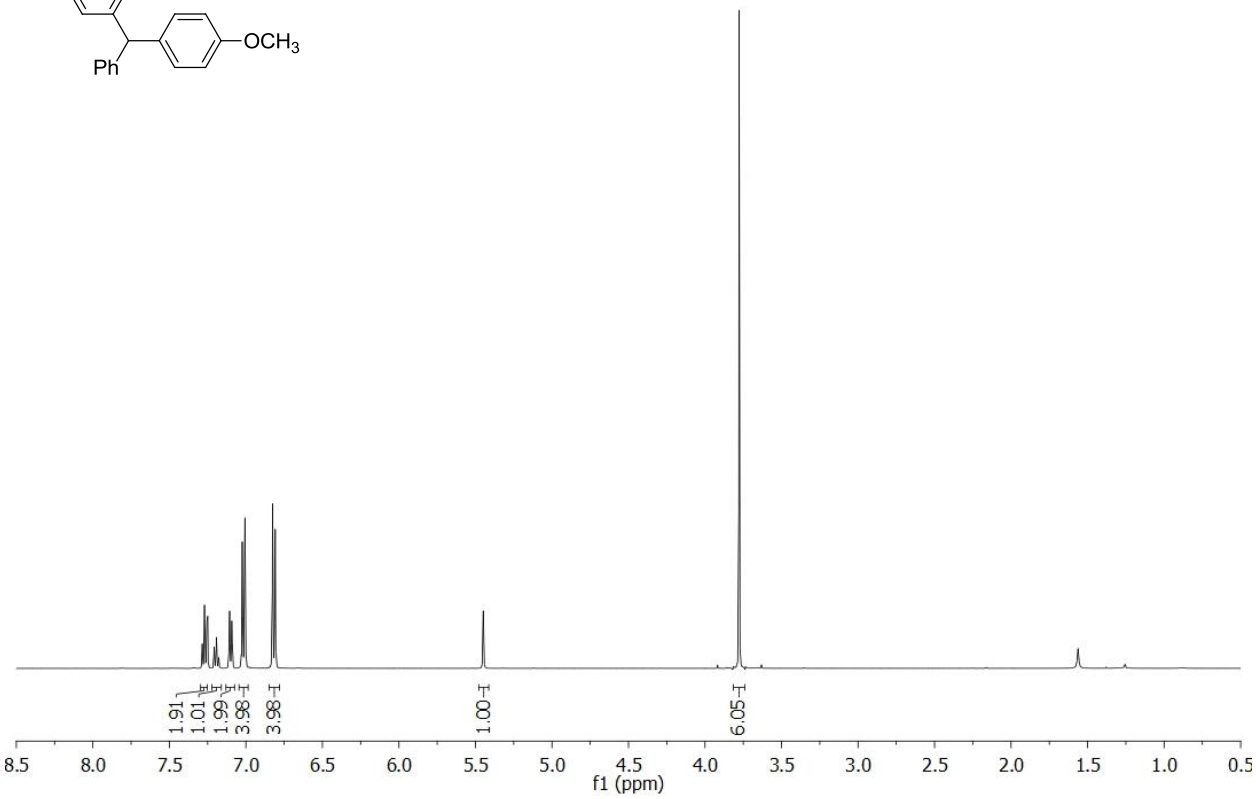


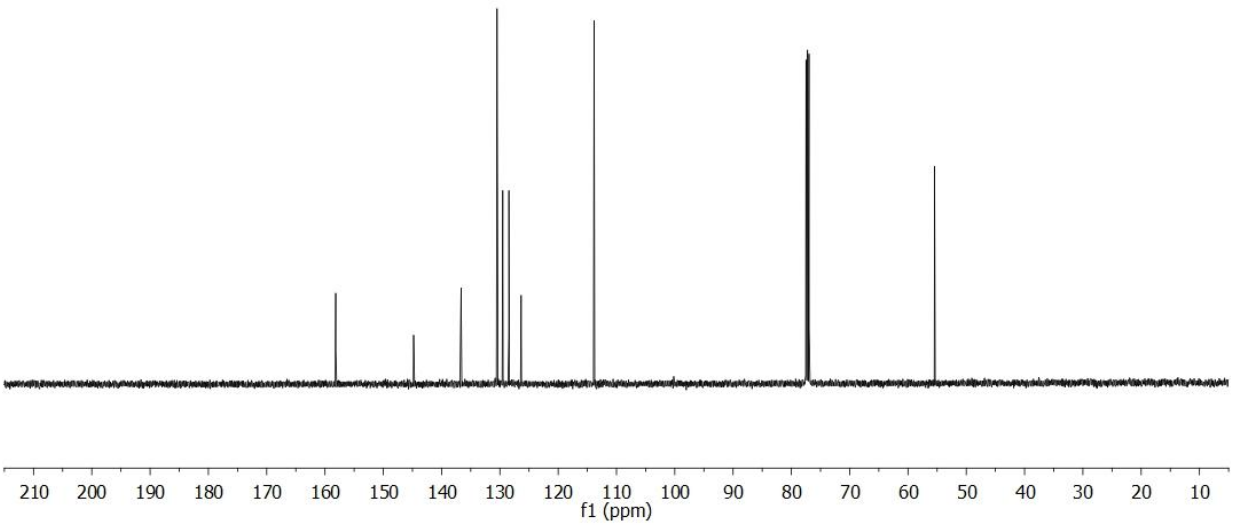




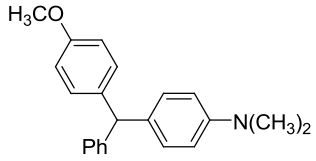
3cd – (4-methoxyphenyl)(4-methoxyphenyl)phenylmethane

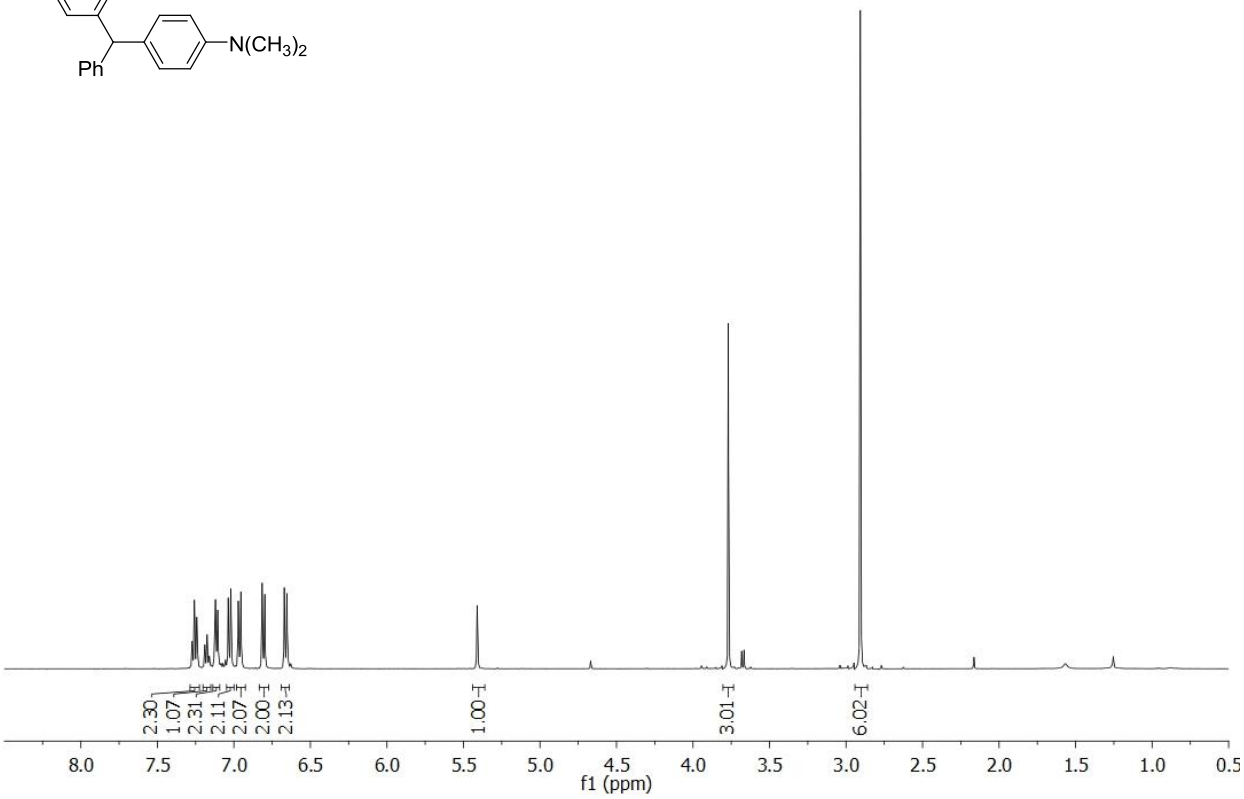


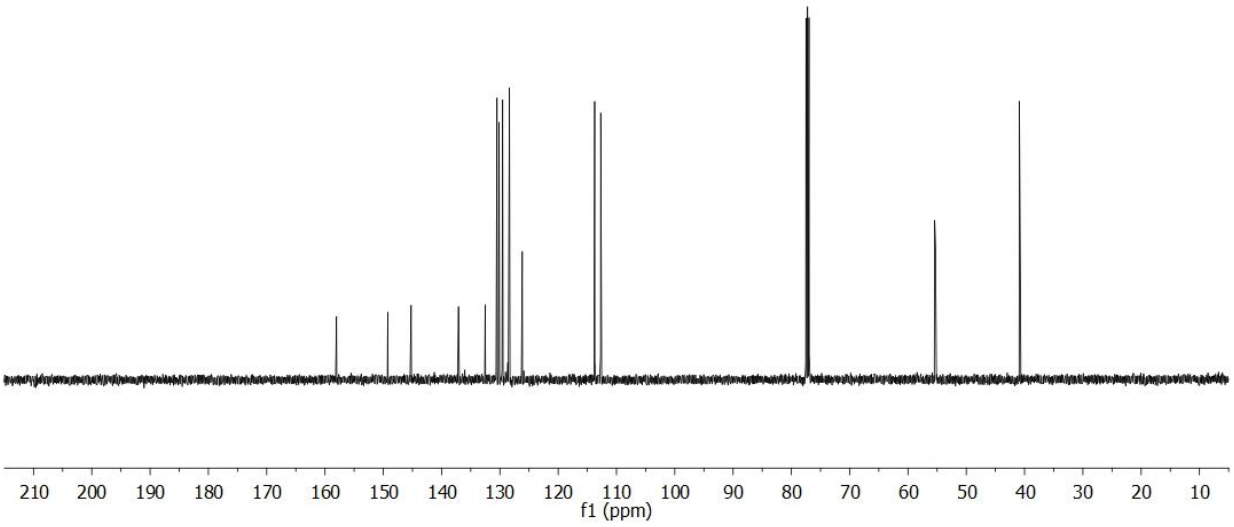




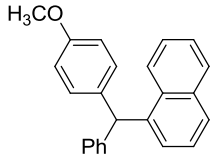
3ce – (4-*N*,*N*-dimethylaminophenyl)(4-methoxyphenyl)phenylmethane

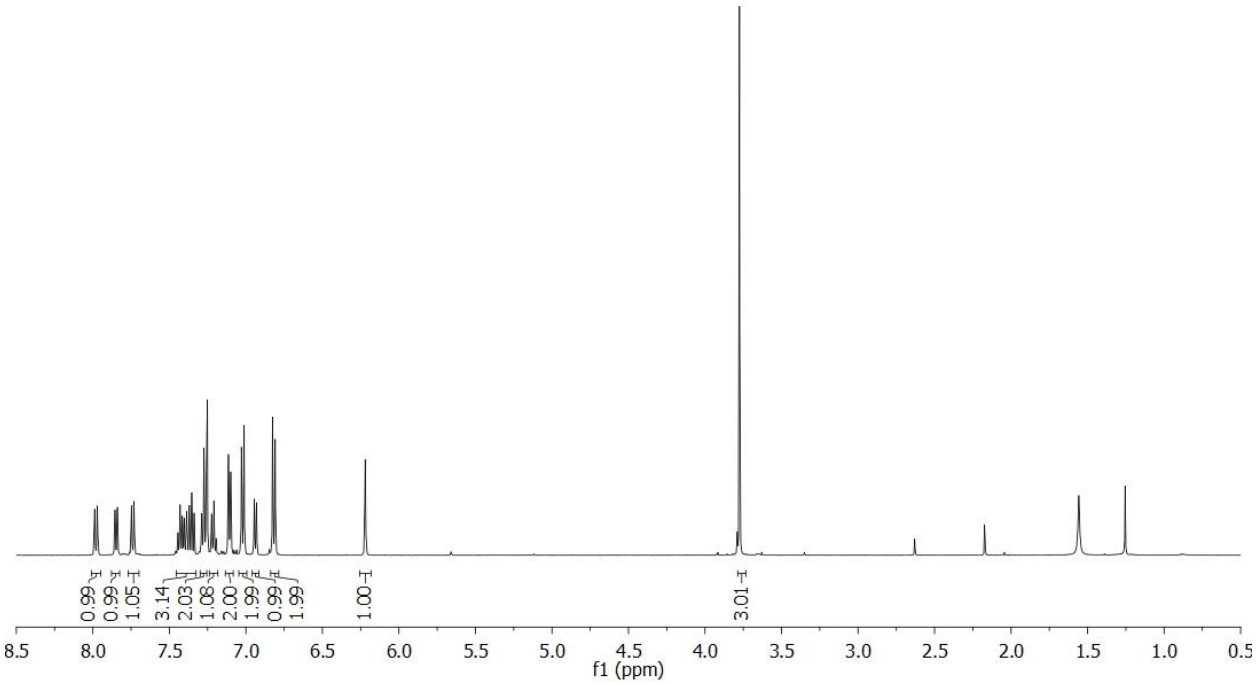


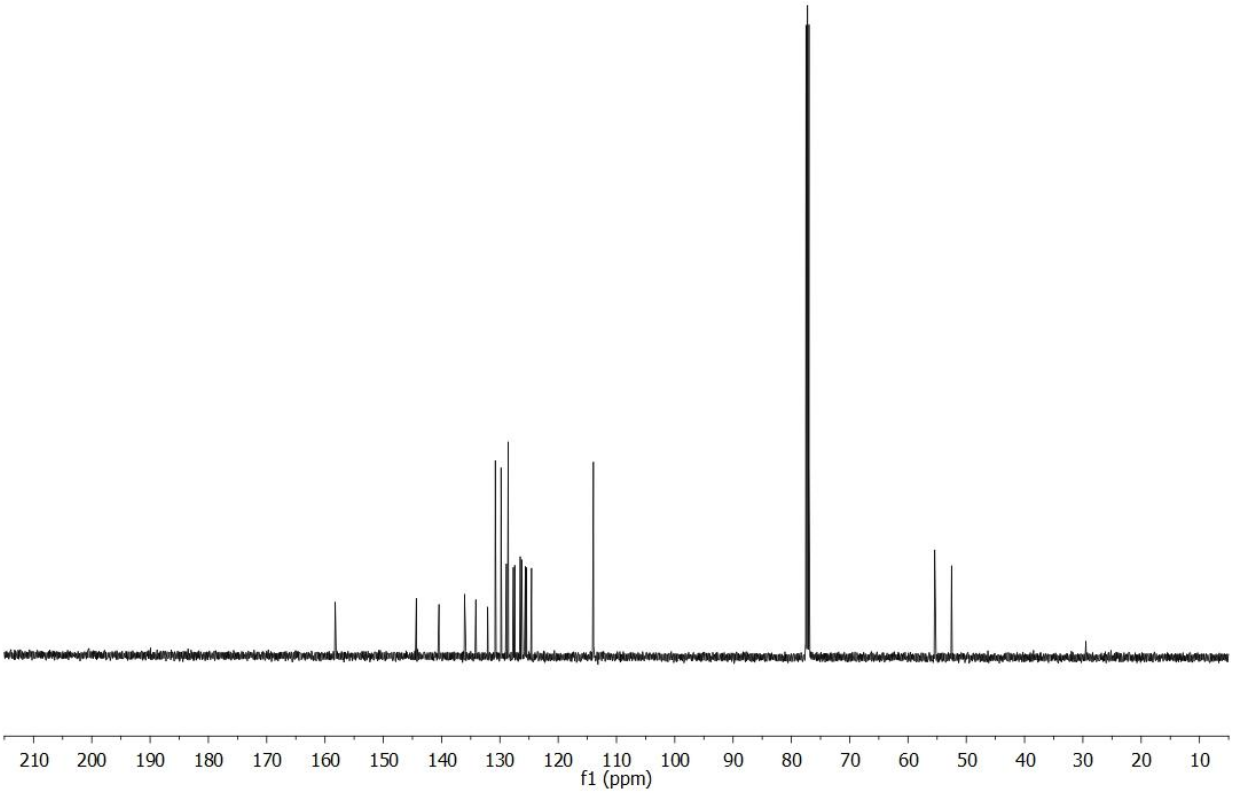




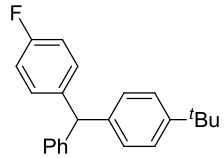
3cf – (1-naphthyl)(4-methoxyphenyl)phenylmethane

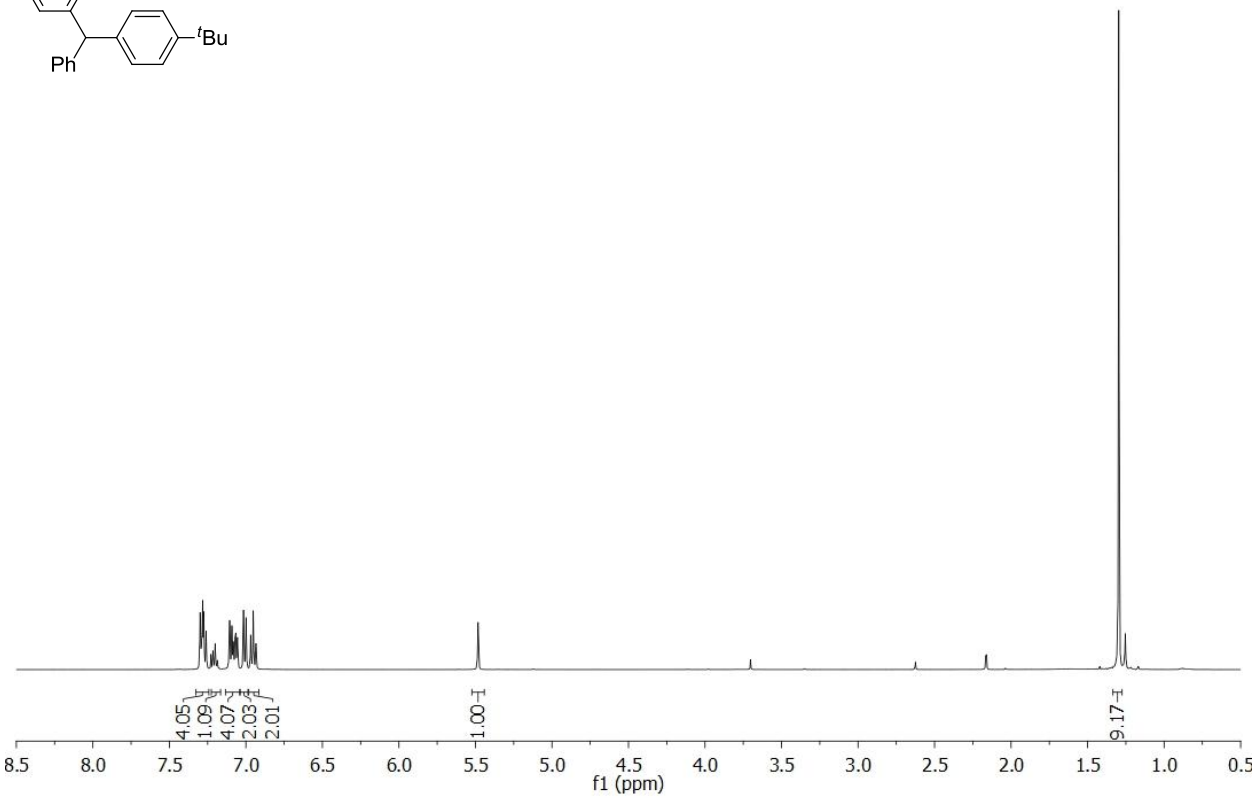


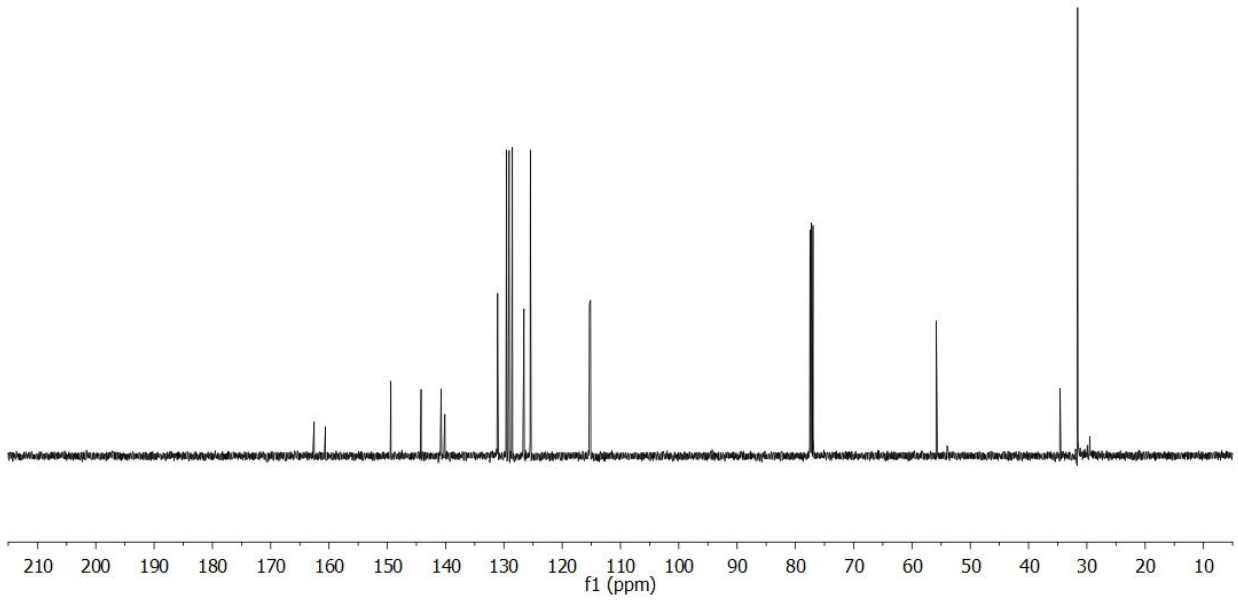




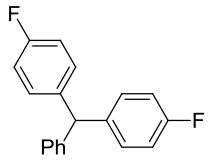
3da – (4-*tert*-butylphenyl)(4-fluorophenyl)phenylmethane

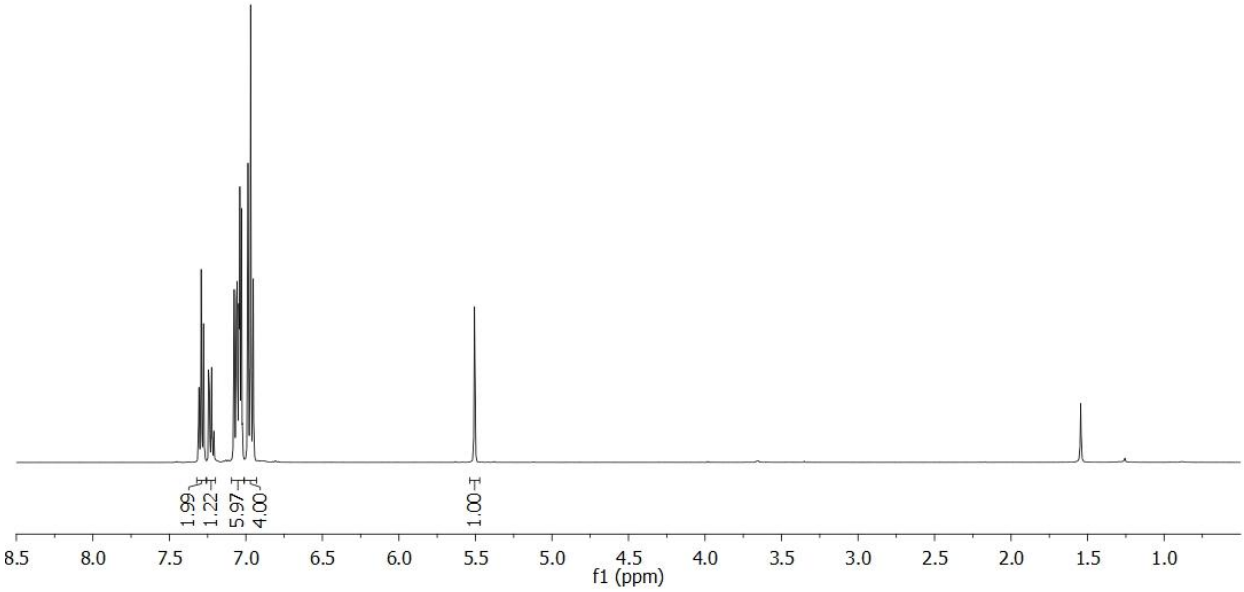


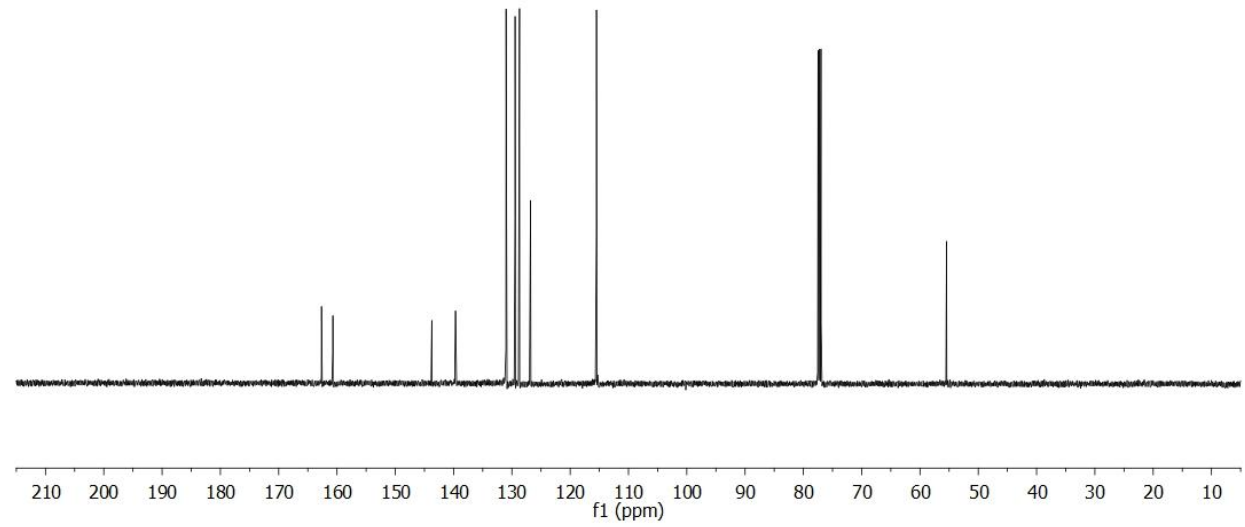




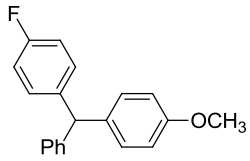
3db – (4-fluorophenyl)(4-fluorophenyl)phenylmethane

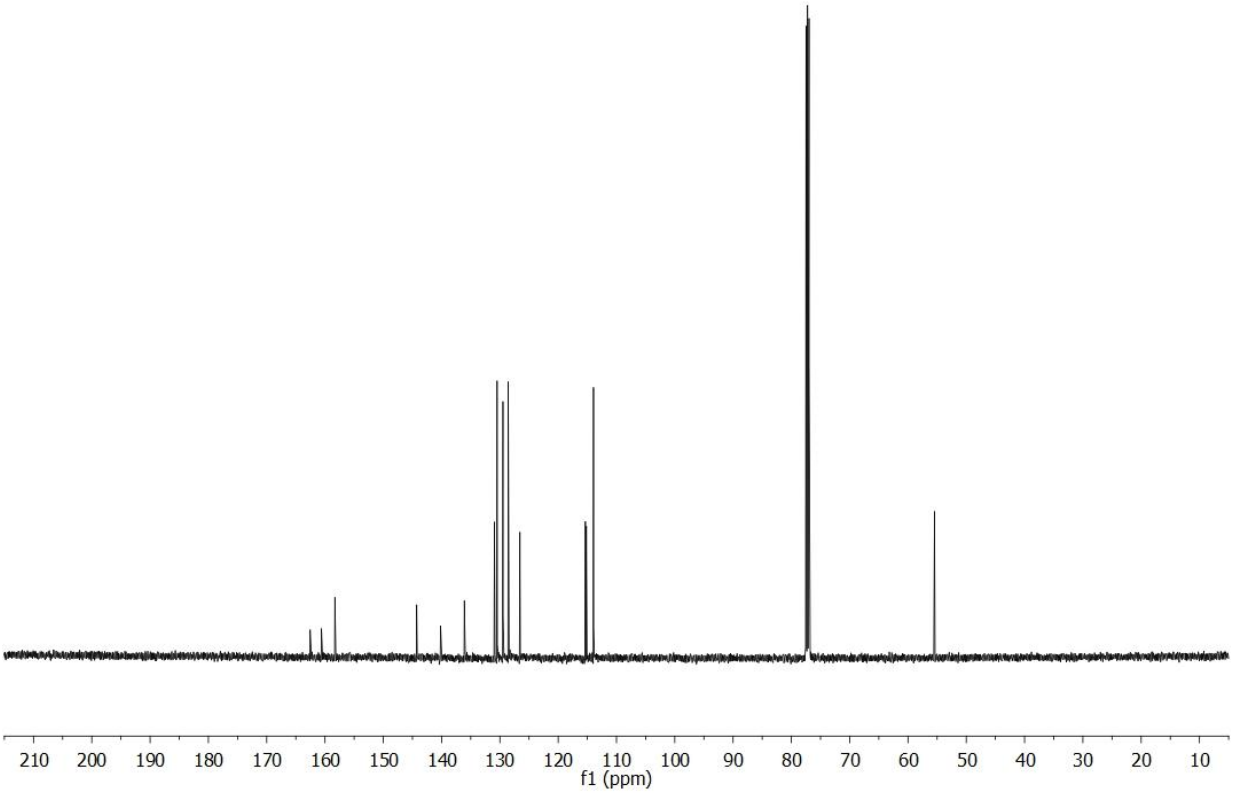




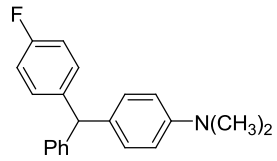


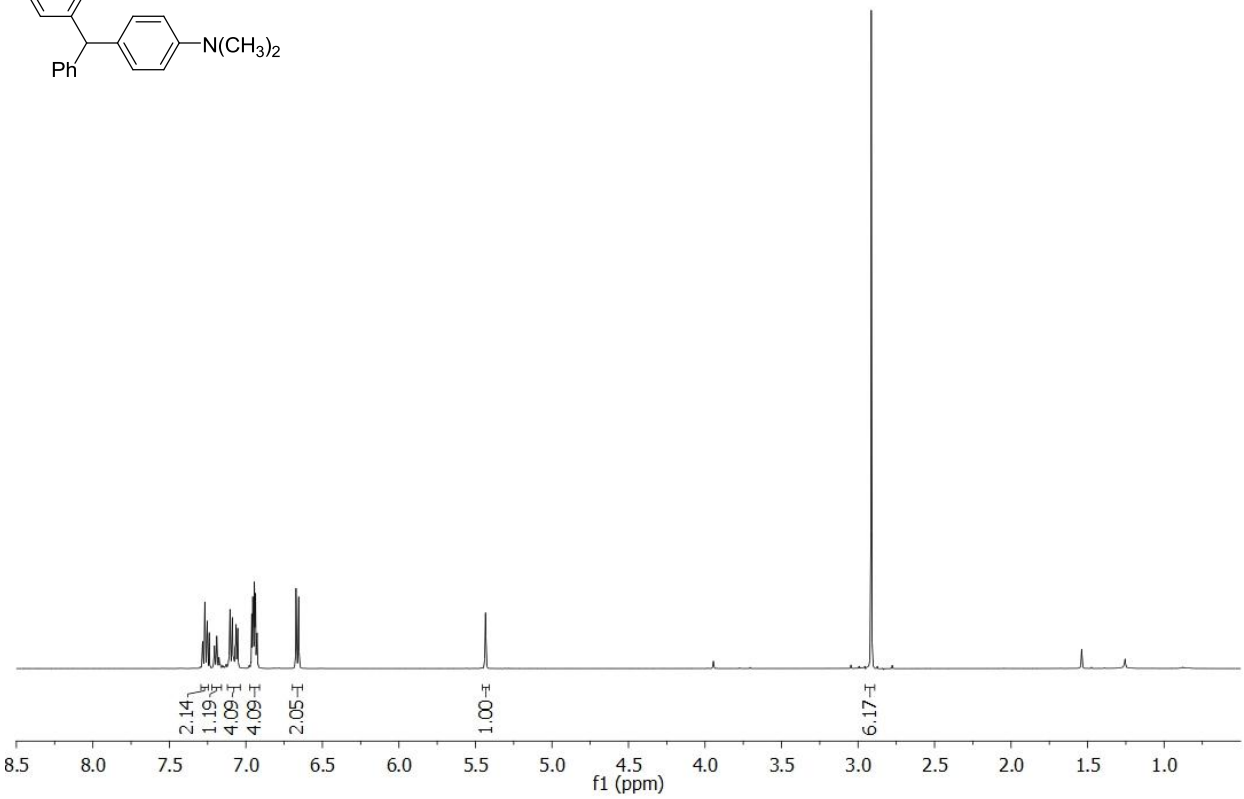
3dd (= 3cb) – (4-methoxyphenyl)(4-fluorophenyl)phenylmethane

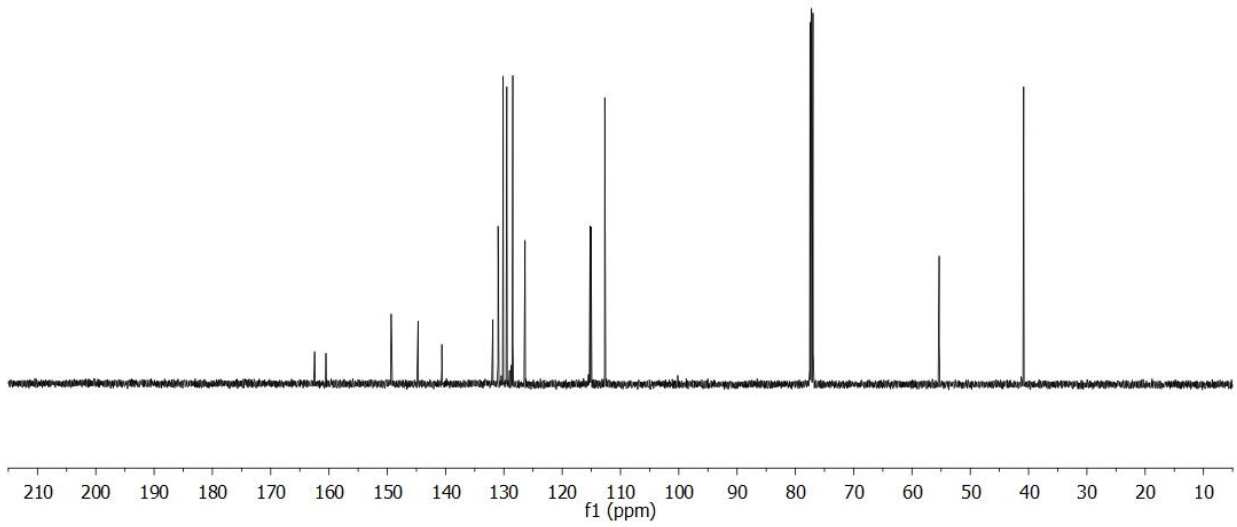




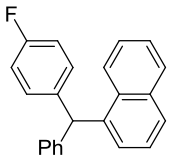
3de – (4-methoxyphenyl)(4-fluorophenyl)phenylmethane

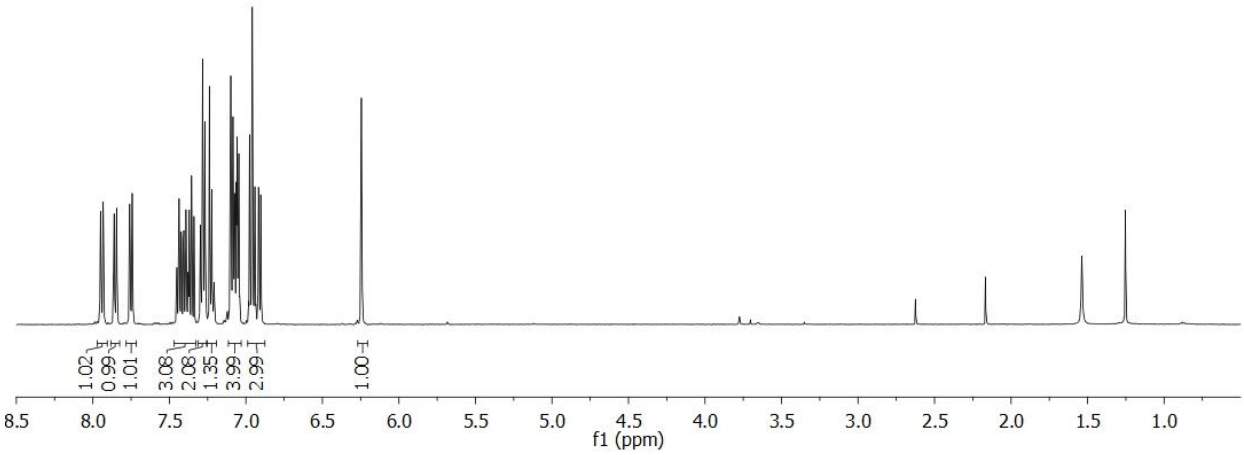


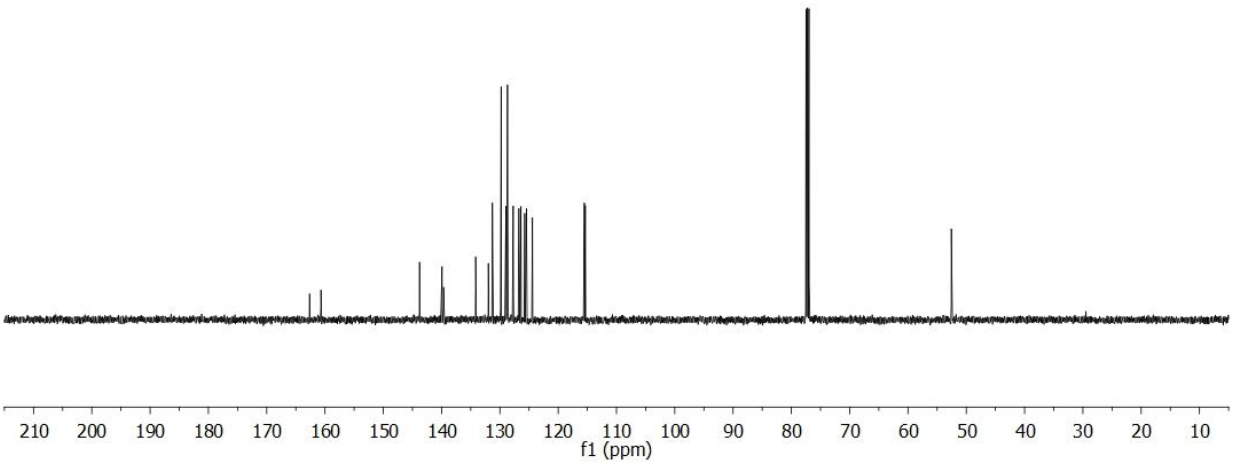




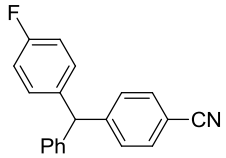
3df – (1-naphthyl)(4-fluorophenyl)phenylmethane

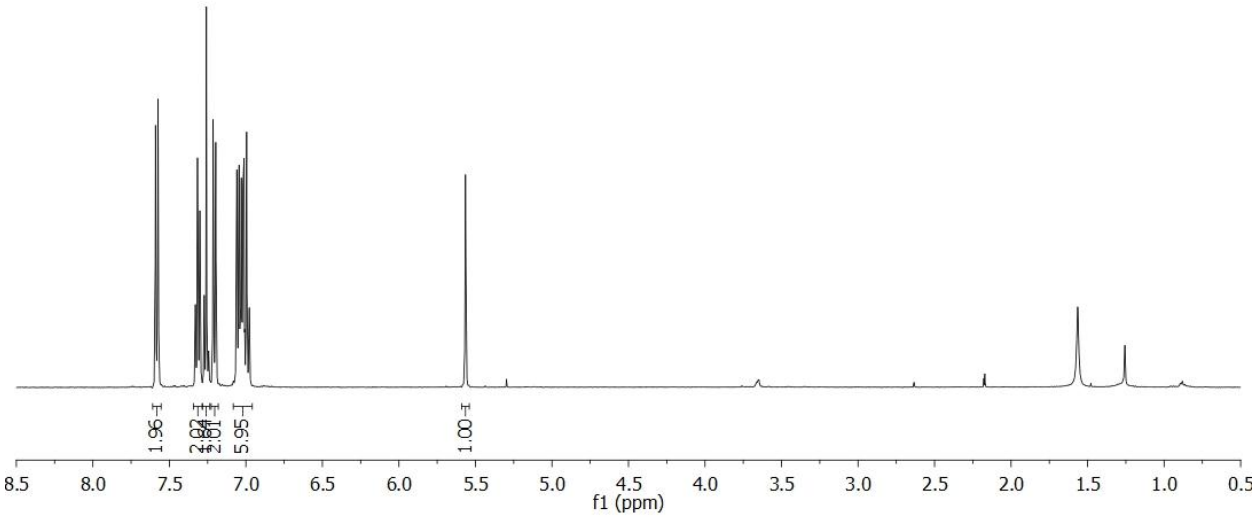


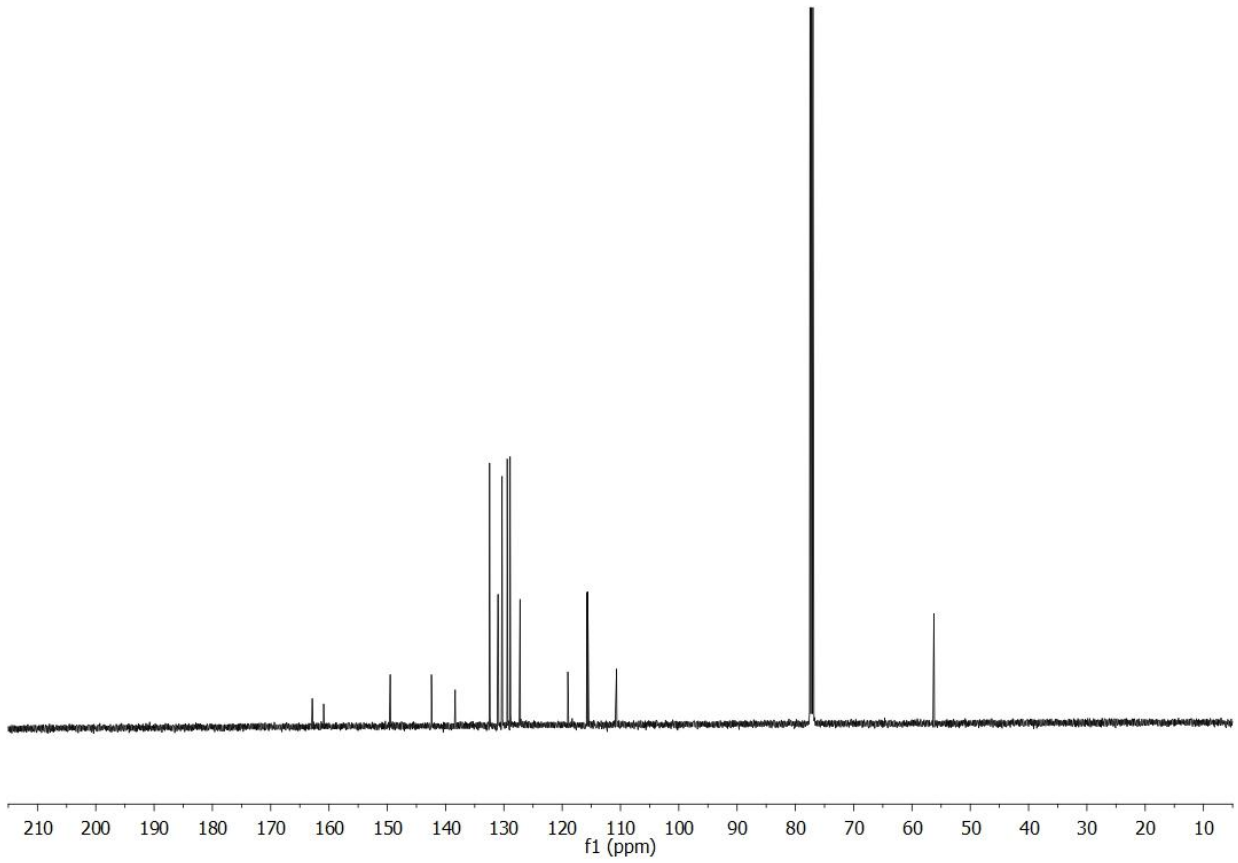




3dg– (4-cyanophenyl)(4-fluorophenyl)phenylmethane







3di– (2-pyridyl)(4-fluorophenyl)phenylmethane