Efficient Access to Triarylmethanes through Decarboxylation

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Supporting Information

EXPERIMENTAL SECTION

General. All the reactions were carried out in oven-dried glassware under Argon atmosphere. Carboxylic acids were purchased from Sigma Aldrich. Palladium catalysts were purchased from Sigma Aldrich. Phosphine ligands were bought from Sigma-Aldrich or Alfa Aesar and used as such. DMSO and DMF was dried according to standard procedure and stored on molecular sieves 4 Å. All the other reagents and solvents mentioned in this text were bought from Sigma Aldrich or Alfa Aesar or spectrochem and purified if necessary. NMR spectra were recorded on 300, 400 or 500 MHz spectrometer for ^{1}H NMR, 75 or 100 or 125 MHz for ^{13}C NMR spectroscopy. Chemical shifts are reported in parts per million (δ) downfield relative to the residual signals of tetramethylsilane in $CDCl_3$. Spin multiplicities are indicated by the following symbol: s (singlet), d (doublet), t (triplet) and m (multiplet).

General procedure for synthesis of Diphenyl carbinols

A 100 ml double-necked round bottom flask was charged with magnesium (2 equiv), pinch of iodine and after flushing with nitrogen, dried THF was added and stirred. Then corresponding bromoarene (1.5equiv) was added, the colour of iodine disappeared slowly on time, indicating the generation of Grignard reagent. It was stirred for one hour followed by addition of corresponding carbaldehye (1 equiv). Completion of reaction was determined by TLC. Reaction was quenched by NH_4Cl solution. Organic layer was extracted thrice with ethyl acetate and concentrated *in vacuo*. Compound was purified by column chromatography.

General procedure for synthesis of Diphenyl methyl iodide

To oven dried round bottom flask under nitrogen, the alcohol (1 equiv) and KI (1 equiv) were added and dissolved in dry 1,4-dioxane and stirred for 5 min. Then $BF_3.Et_2O$ (1equiv) was added and

stirred at room temperature. Completion of reaction was monitored by TLC. The reaction mixture was poured in cold water and extracted with dichloromethane. The organic layer was washed with water and dried by sodium sulphate; solvent was evaporated under reduced pressure. The crude was purified by quickly passing through short pad of silica using hexane /ethyl acetate (8:2) as eluent to obtain diphenylmethyl iodides and should be used immediately.

General procedure A for synthesis of various substituted triarylmethanes by Decarboxylative Cross Coupling

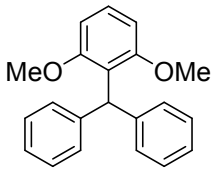
To the oven dried round bottom flask was charged with aryl carboxylic acid (1equiv), Ag_2CO_3 (1.5 equiv), $PdCl_2$ (0.1 equiv), Xantphos (0.2 equiv), diphenyl methyl iodides (1 equiv) were dissolved in DMSO. The reaction mixture was degassed with argon thrice and stirred in preheated oil bath. Reaction as monitored by TLC after completion, the reaction mixture was cooled to room temperature and filtered through celite bed. The organic layer was washed with NH_4Cl solution and dried on sodium sulphate and concentrated under reduced pressure. It was later purified by column chromatography on silica gel to obtain corresponding triarylmethanes.

General procedure B for synthesis of substituted triarylmethanes by Decarboxylative Cross Coupling

To the oven dried round bottom flask was charged with aryl carboxylic acid (1equiv), Ag_2CO_3 (1.5 equiv), $PdCl_2$ (0.2 equiv), Xantphos (0.4 equiv), diphenyl methyl iodides (1 equiv) were dissolved in DMSO:DMF 90:10. The reaction mixture was degassed with argon thrice and stirred in preheated oil bath at requisite temperature and was monitored by TLC. After the completion of reaction, mixture was cooled to room temperature and filtered through a bed celite with ethyl acetate. The organic layer was washed with NH_4Cl solution and dried on sodium sulphate and concentrated *in vacuo*. It was later purified by column chromatography on silica gel to obtain corresponding triarylmethanes.

Spectroscopic data of synthesized compounds

((2,6-dimethoxyphenyl)methylene)dibenzene (3aa)

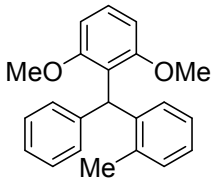


3aa

Prepared following the general procedure A. Rf: 0.35 (2% EtOAc in Hex). Isolated as pale brown, oily liquid (yield: 68%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.27-7.06 (m, 10H), 6.73-6.71 (m, 1H), 6.46-6.37 (m, 2H), 5.82 (s,

1H), 3.78 (s, 3H), 3.68 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): 158.2 (2C), 143.0 (2C), 129.5 (4C), 128.1 (4C), 126.8 (2C), 124.6, 102.5 (2C), 97.4, 54.3, 54.0, 47.9 ppm. IR (film, cm^{-1}): 2817, 1726, 1582, 1231, 814, 770, 637. MS (ESI): m/z 305 $(M+H)^+$. HRMS (ESI): m/z calcd for $C_{21}H_{21}O_2$ $[M+H]^+$ 305.1536, found 305.1542.

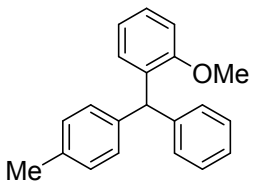
1,3-dimethoxy-2-(phenyl(o-tolyl)methyl)benzene (3ab)



3 ab

Prepared following the general procedure A. Rf: 0.4 (2% EtOAc in Hex). Isolated as yellow oily liquid (yield: 70%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.22-7.02 (m, 8H), 6.79-6.35 (m, 4H), 5.89 (s, 1H), 3.78 (s, 3H), 3.68 (s, 3H), 2.18 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): 159.3, 158.0, 143.6, 142.6, 136.7, 130.6, 130.1, 129.5 (2C), 128.9, 128.0 (2C), 126.0, 125.8, 125.4, 124.7, 103.7, 98.6, 55.6, 55.2, 45.9, 19.6 ppm. IR (film, cm^{-1}): 2828, 1631, 1536, 1483, 1284, 1171, 823, 772, 656. MS (ESI): m/z 319 $(M+H)^+$. HRMS (ESI): m/z calcd for $C_{22}H_{23}O_2$ $[M+H]^+$ 319.1693, found 319.1691.

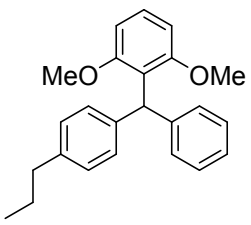
1-methoxy-2-(phenyl(p-tolyl)methyl)benzene (3bf)



3bf

Prepared following the general procedure A. Rf: 0.6 (1.5% EtOAc in Hex). Yield: 72%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23-6.80 (m, 11H), 6.43-6.34 (m, 2H), 5.69 (s, 1H), 3.70 (s, 3H), 2.27 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): 156.2, 144.4, 141.1, 141.0, 135.1, 135.0, 132.7, 132.6, 129.1, 129.0, 129.0, 128.6, 127.8, 125.6, 125.5, 124.1, 103.7, 98.6, 55.8, 48.6, 21.0 ppm. IR (film, cm^{-1}): 1689, 1276, 948, 879, 847, 716. MS (ESI): m/z 289 $(M+H)^+$. HRMS (ESI): m/z calcd for $C_{21}H_{21}O$ $[M+H]^+$ 289.1587, found 289.1594.

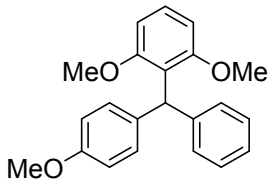
1,3-dimethoxy-2-(phenyl(4-propylphenyl)methyl)benzene (3ac)



3 ac

Prepared following the general procedure A. Rf: 0.3 (2% EtOAc in Hex). Yield: 80%, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.25-6.96 (m, 9H), 6.75-6.73 (m, 1H), 6.45-6.37 (m, 2H), 5.79 (s, 1H), 3.77 (s, 3H), 3.68 (s, 3H), 2.54 (t, 2H, J=7.83Hz), 1.64-1.59 (m, 2H), 0.92 (t, 3H, J=7.32Hz) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): 159.3, 157.9, 144.5, 141.3, 140.1, 130.7, 129.3 (2C), 129.1 (2C), 128.1 (2C), 128.0 (2C), 125.8, 125.5, 103.7, 98.6, 55.5, 55.2, 48.6, 37.6, 24.4, 13.9 ppm. IR (film, cm^{-1}): 3248, 1692, 1452, 1268, 910, 880, 730, 633. MS (ESI): m/z 347 $(M+H)^+$. HRMS (ESI): m/z calcd for $C_{24}H_{27}O_2$ $(M+H)^+$ 347.2006, found 347.2003.

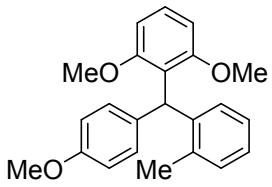
1,3-dimethoxy-2-((4-methoxyphenyl)(phenyl)methyl)benzene (3 ad):



3ad

Prepared following the general procedure A. Rf: 0.2 (3% EtOAc in Hex). Yield: 74%, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35-6.36 (m, 12H), 5.76 (s, 1H), 3.76 (s, 3H), 3.76 (s, 3H), 3.67 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): 159.3, 158.9, 157.9, 144.6, 136.3, 130.6, 130.3, 129.3, 128.5, 128.3, 128.0, 127.1, 127.0, 125.8, 125.5, 113.4, 103.7, 98.6, 55.6, 55.2, 55.2, 48.3 ppm. IR (film, cm^{-1}): 2802, 1664, 1596, 1327, 932, 815, 804, 730, 623. MS (ESI): m/z 335 $(M+H)^+$. HRMS (ESI): m/z calcd for $C_{22}H_{23}O_3$ $(M+H)^+$ 335.1642, found 335.1632.

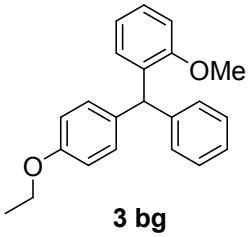
1,3-dimethoxy-2-((4-methoxyphenyl)(o-tolyl)methyl)benzene (3 ae)



3ae

Prepared following the general procedure A. Rf: 0.3 (4% EtOAc in Hex). Yield: 72%, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.17-6.28 (m, 11H), 5.76 (s, 1H), 3.70 (s, 6H), 3.60 (s, 3H), 2.10 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): 159.3, 157.9, 157.7, 143.0, 136.6, 135.6, 130.5, 130.3, 130.1 (2C), 128.7 (2C), 125.9, 125.4, 125.0, 113.4, 103.7, 98.6, 55.6, 55.2, 55.1, 45.1, 19.5 ppm. IR (film, cm^{-1}): 2902, 1685, 1536, 1347, 911, 776. MS (ESI): m/z 349 $(M+H)^+$. HRMS (ESI): m/z calcd for $C_{23}H_{25}O_3$ $(M+H)^+$ 349.1798, found 349.1810.

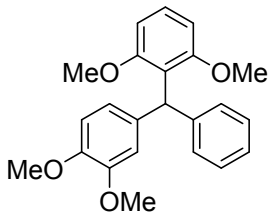
1-((4-ethoxyphenyl)(phenyl)methyl)-2-methoxybenzene (3bg):



3 bg

Prepared following the general procedure A. Rf: 0.3 (2 % EtOAc in Hex). Yield: 68%, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.08-6.29 (m, 13H), 5.62 (s, 1H), 3.90 (m, 2H), 3.62 (s, 3H), 1.31 (t, 3H, J=5.4 Hz) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): 157.0, 156.2, 144.6, 136.2, 136.1, 132.6, 132.5, 130.0 (2C), 129.1 (3C), 127.9 (2C), 125.6, 124.3, 113.9, 95.5, 63.3, 55.8, 48.2, 14.9 ppm. IR (film, cm^{-1}): 2815, 1664, 1592, 1585, 1320, 1104, 932, 812, 730, 623. MS (ESI): m/z 319 $(M+H)^+$. HRMS (ESI): m/z calcd for $C_{22}H_{23}O_2$ $[M+H]^+$ 319.1693, found 319.1693.

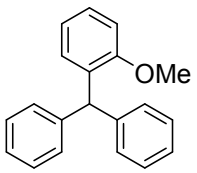
2-((3,4-dimethoxyphenyl)(phenyl)methyl)-1,3-dimethoxybenzene (3ah):



3 ah

Prepared following the general procedure A. Rf: 0.3 (5% EtOAc in Hex). Yield: 79%, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.18-6.29 (m, 11H), 5.68 (s, 1H), 3.76 (s, 3H), 3.70 (s, 3H), 3.67 (s, 3H), 3.61 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): 158.2, 156.8, 147.4, 146.0, 143.3, 135.6, 129.4 (2C), 128.1 (2C), 126.8, 124.7, 124.2, 120.1, 111.7, 109.5, 102.6, 97.5, 54.6, 54.6, 54.4, 54.1, 47.5 ppm. IR (film, cm^{-1}): 2805, 1664, 1587, 1523, 1320, 1104, 897, 816. MS (ESI): m/z 365 $(M+H)^+$, HRMS (ESI): m/z calcd for $C_{23}H_{25}O_3$ $[M+H]^+$ 365.1747, found 365.1756.

((2-methoxyphenyl)methylene)dibenzene (3ba):

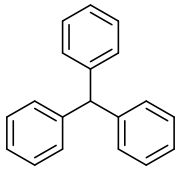


3 ba

Prepared following the general procedure A. Rf: 0.5 (1% EtOAc in Hex). Yield: 71%, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.27-6.79 (m, 14H), 5.48 (s, 1H), 3.75 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): 158.0, 144.3 (2C), 136.1, 130.4 (2C), 129.4 (5C), 128.3 (4C), 126.2 9(2C), 113.7, 56.0, 55.2 ppm. IR (film, cm^{-1}): 2943, 1685,

1576, 1280, 810, 730. MS (ESI): m/z 275 $(M+H)^+$. HRMS (ESI): m/z calcd for $C_{20}H_{19}O$ $[M+H]^+$ 275.1430, found 275.1439.

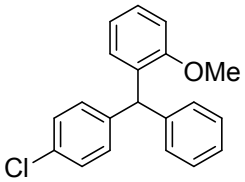
Triphenylmethane (3ca):



3ca

Prepared following the general procedure A. Rf: 0.5 (1% EtOAc in Hex). Yield: 67 %, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33-7.09 (m, 15H), 5.54 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): 143.9 (3C), 129.5, 128.3 (6C), 128.3 (6C), 128.2, 126.3, 56.9 ppm. IR (film, cm^{-1}): 2987, 1642, 1586, 1572, 1121. MS (ESI): m/z 245 $(M+H)^+$. HRMS (ESI): m/z calcd for $C_{19}H_{17}O$ $[M+H]^+$ 245.1325, found 245.1329.

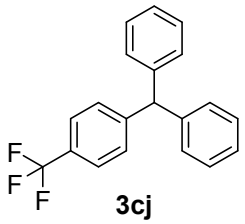
1-((4-chlorophenyl)(phenyl)methyl)-2-methoxybenzene (3bi):



3bi

Prepared following the general procedure A. Rf: 0.3 (2% EtOAc in Hex). Yield: 62%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.25-7.19 (m, 6H), 7.07-6.99 (m, 4H), 6.87- 6.79 (m, 3H), 5.87 (s, 1H), 3.69 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): 157.0, 143.3, 142.5, 132.1, 131.8, 130.7, 130.2, 129.4 (2C), 128.2 (5C), 127.8, 126.2, 120.3, 110.7, 55.5, 49.0 ppm. IR (film, cm^{-1}): 2763, 1675, 1297, 981, 824, 747, 615. MS (ESI): m/z 309 $(M+H)^+$. HRMS (ESI): m/z calcd for $C_{20}H_{18}ClO$ $[M+H]^+$ 309.1041, found 309.1050.

((4-(trifluoromethyl)phenyl)methylene)dibenzene (3cj):

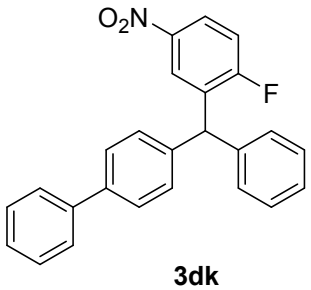


3cj

Prepared following the general procedure A. Rf: 0.5 (1 % EtOAc in Hex). Yield: 56%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54-7.52 (m, 2H), 7.31-7.21 (m, 8H), 7.10-7.08 (m, 4H), 5.59 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): 148.0, 142.9 (2C), 129.7, 129.3 (6C), 128.5 (6C), 126.6, 125.2, 125.2, 56.6. IR (film, cm^{-1}): 2812,

1664, 1543, 1320, 932, 815. MS (ESI): m/z 313 $(M+H)^+$. HRMS (ESI): m/z calcd for $C_{20}H_{16}F_3$ $[M+H]^+$ 313.1199, found 313.1198.

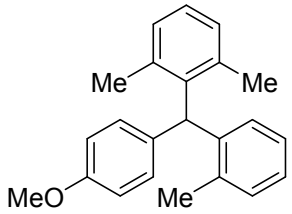
4-((2-fluoro-5-nitrophenyl)(phenyl)methyl)-1,1'-biphenyl (3dk):



3dk

Prepared following the general procedure B. Rf: 0.5 (3% EtOAc in Hex). Yield: 42%, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58-7.54 (m, 4H), 7.45-7.32 (m, 6H), 7.29-7.25 (m, 7H), 5.48 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): 145.2, 142.1, 140.8, 140.0, 137.8, 132.3, 130.7, 130.0, 128.9, 128.7, 128.4, 128.3 (4C), 128.1(2C), 127.6 (2C), 127.3, 127.2, 127.1, 127.0, 126.9, 79.8. IR (film, cm^{-1}): 2883, 1904, 1684, 1892, 1543, 871, 810, 742, 613. MS (ESI): m/z 384 $(M+H)^+$. HRMS (ESI): m/z calcd for $C_{25}H_{19}FNO_2$ $[M+H]^+$ 384.1394, found 384.1402.

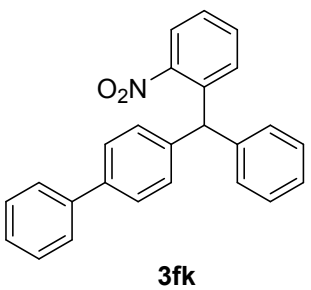
2-((4-methoxyphenyl)(o-tolyl)methyl)-1,3-dimethylbenzene (3ef):



3ef

Prepared following the general procedure A. Rf: 0.5 (2 % EtOAc in Hex). Yield: 74%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42-7.40 (m, 1H), 7.31-7.12 (m, 10H), 5.40 (s, 1H), 3.38 (s, 3H), 2.26 (s, 9H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): 144.6, 141.8, 141.4, 141.0, 139.9, 139.6, 136.4, 136.0, 130.6, 130.5, 128.3, 128.2, 127.9, 127.6, 127.4, 126.2, 126.1 (2C), 57.1, 47.4, 19.5 (2C), 19.3 ppm. IR (film, cm^{-1}): 2984, 1683, 1317, 981, 823, 728, 604. HRMS (ESI): m/z calcd for $C_{23}H_{25}O$ $[M+H]^+$ 317.1900, found 317.1898.

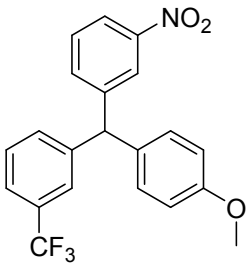
4-((2-nitrophenyl)(phenyl)methyl)-1,1'-biphenyl (3fk):



3fk

Prepared following the general procedure A. Rf : 0.4 (2 % EtOAc in Hex). Yield: 49 %, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58-7.54 (m, 5H), 7.46-7.40 (m, 7H), 7.36-7.28 (m, 6H), 5.48 (s, 1H) ppm. IR (film, cm^{-1}): 2873, 1796, 1676, 1579, 1544, 923, 815, 794, 745, 631. HRMS (ESI): m/z calcd for $C_{25}H_{20}NO_2$ $[M+H]^+$ 366.1489, found 366.1499.

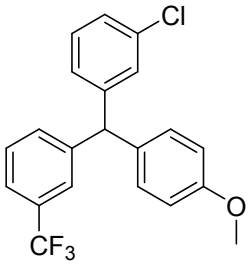
1-((4-methoxyphenyl)(3-(trifluoromethyl)phenyl)methyl)-3-nitrobenzene (3gl):



3gl

Prepared following the general procedure B. Rf: 0.4 (3% EtOAc in Hex). Yield: 51%. ^{1}H NMR (400 MHz, $CDCl_3$): □□7.85-7.79 (m, 1H), 7.56-7.51 (m, 2H), 7.42-7.36 (m, 3H), 7.09-7.06 (m, 2H), 7.01-7.00 (m, 2H), 6.85-6.82(m, 2H), 5.85 (s, 1H), 3.80 (S, 3H) ppm. IR (film, cm^{-1}): 2907, 1886, 1673, 1584, 1543, 1281, 942, 881, 634. HRMS (ESI): m/z calcd for $C_{21}H_{17}F_3NO_3$ $[M+H]^+$ 388.1155, found 388.1149.

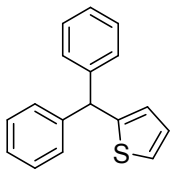
1-chloro-3-((4-methoxyphenyl)(3-(trifluoromethyl)phenyl)methyl)benzene (3hl):



3hl

Prepared following the general procedure B. Rf: 0.2 (2% EtOAc in Hex). Yield: 56%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24-7.22 (m, 1H), 7.04-6.98 (m, 2H), 6.95-6.93 (m, 3H), 6.89-6.88 (m, 2H), 6.85-6.84(m, 1H), 6.82-6.79 (m, 2H), 6.63-6.62 (m, 1H), 5.82 (s, 1H), 3.78 (S, 3H) ppm. IR (film, cm^{-1}): 2928, 1691, 1564, 1298, 911, 828, 804. HRMS (ESI): m/z calcd for $C_{21}H_{17}ClF_3O$ $[M+H]^+$ 377.0915, found 377.0919.

2-benzhydrylthiophene (5):



5

Prepared following the general procedure A. Rf: 0.6 (1% EtOAc in Hex). Yield: 50%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30-7.17 (m, 11H), 6.92 (m, 1H), 6.68 (m, 1H), 5.67 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): 147.9, 143.8 (2C), 128.8 (4C), 128.4 (4C), 126.7, 126.6, 126.4 (2C), 124.5, 52.1. IR (film, cm^{-1}): 1592, 1585,

1365, 623. MS (ESI): m/z 251 $(M+H)^+$. HRMS (ESI): m/z calcd for $C_{17}H_{15}S$ $[M+H]^+$ 251.0889, found 251.0892.

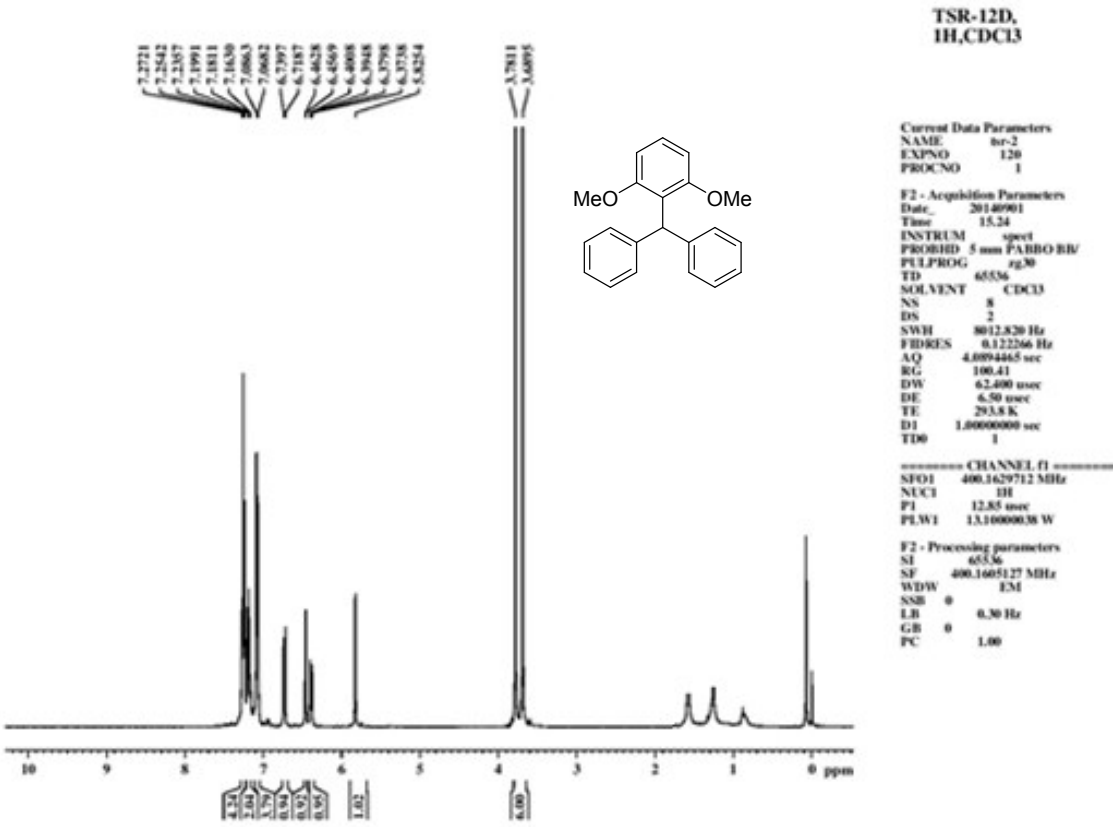


Figure 1. ^{1}H spectrum of compound **3aa**.

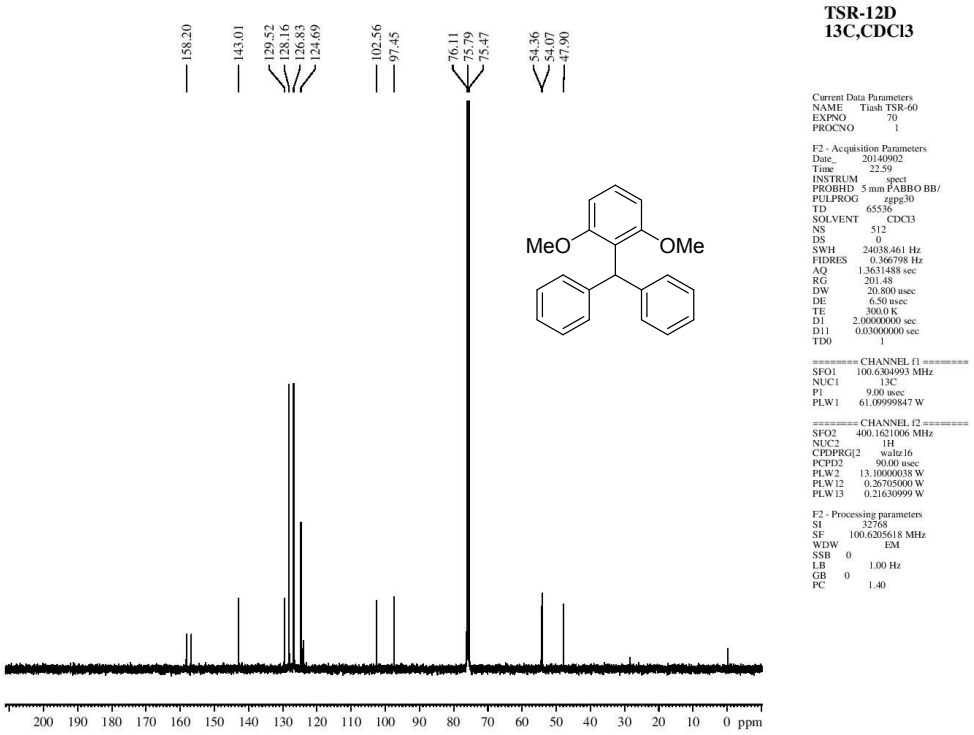


Figure 2. ^{13}C spectrum of compound **3aa**.

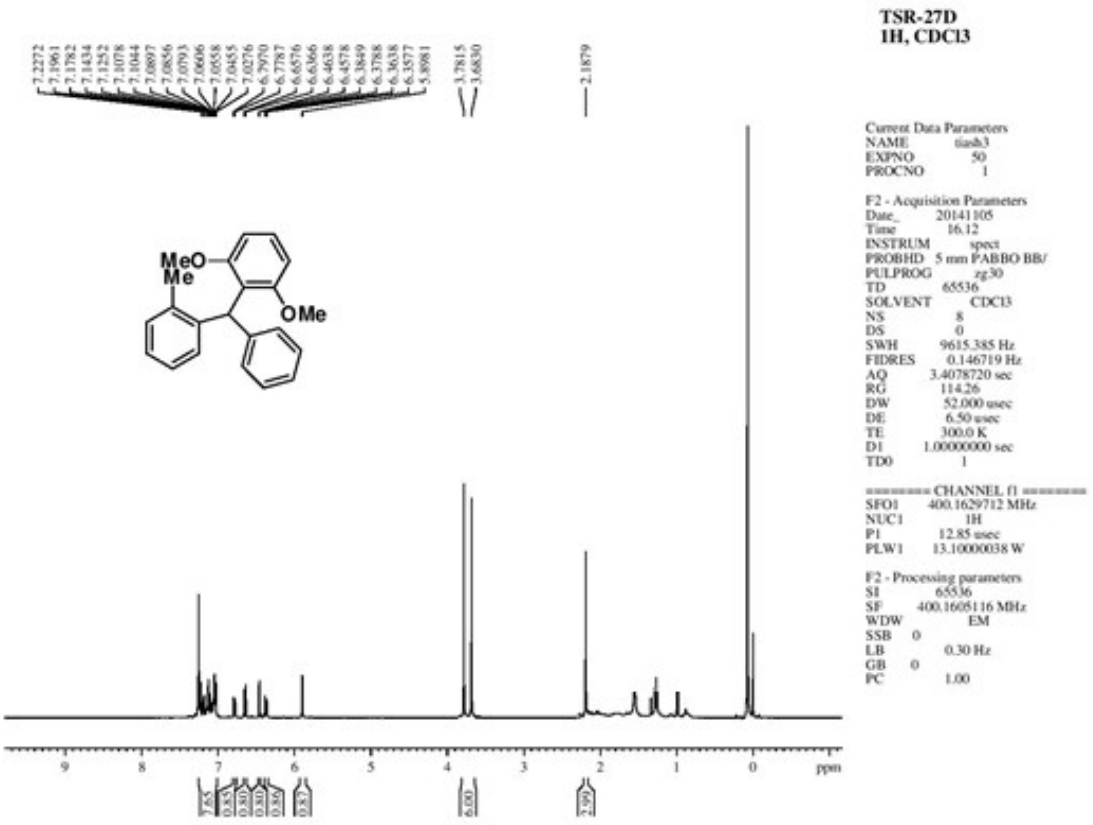


Figure 3. ^{1}H spectrum of compound **3ab**

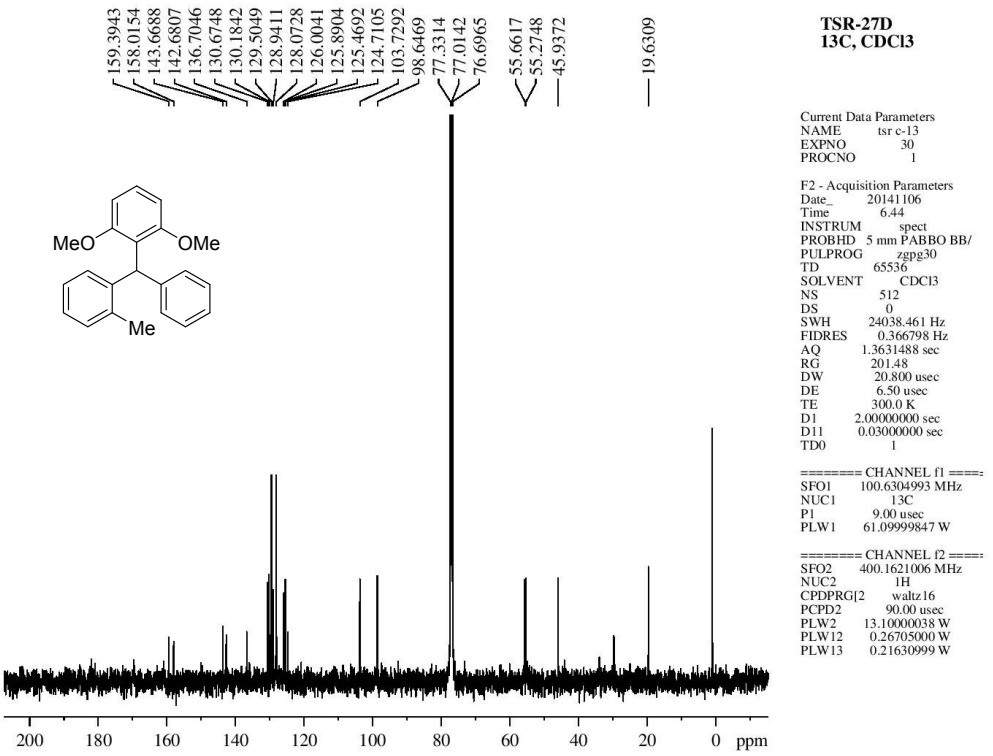


Figure 4. ^{13}C spectrum of compound **3ab**.

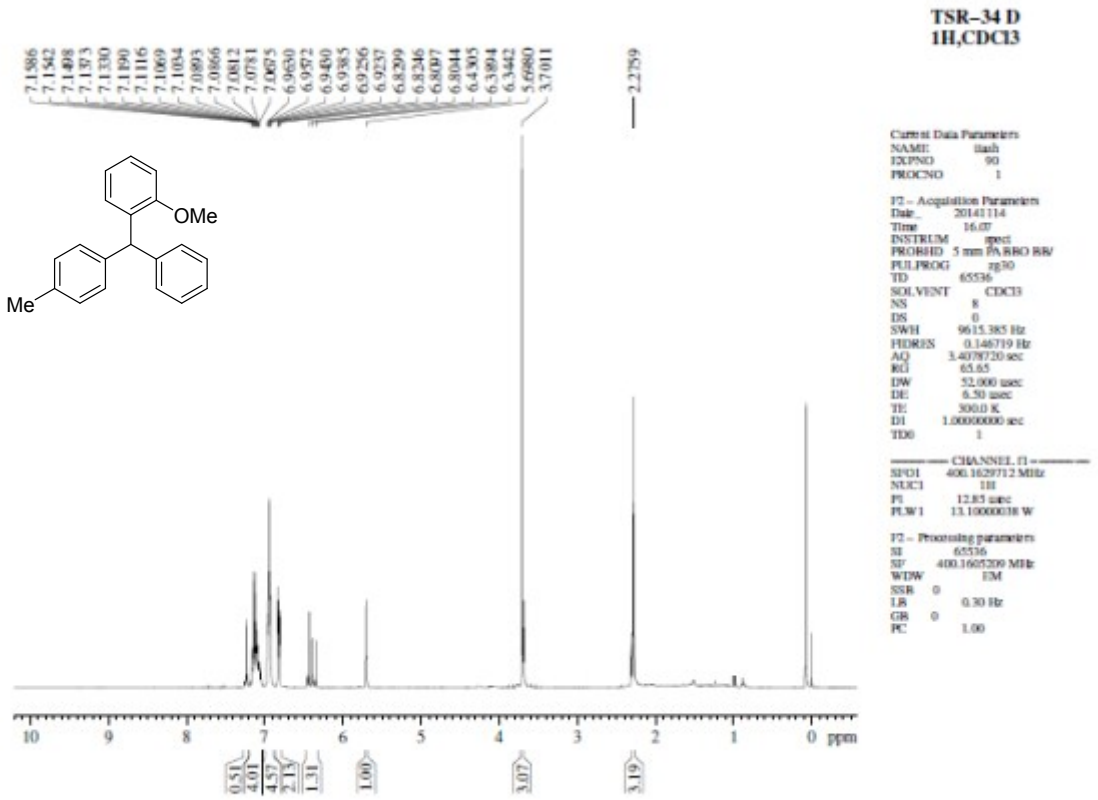


Figure 5. ^{1}H spectrum of compound **3bf**.

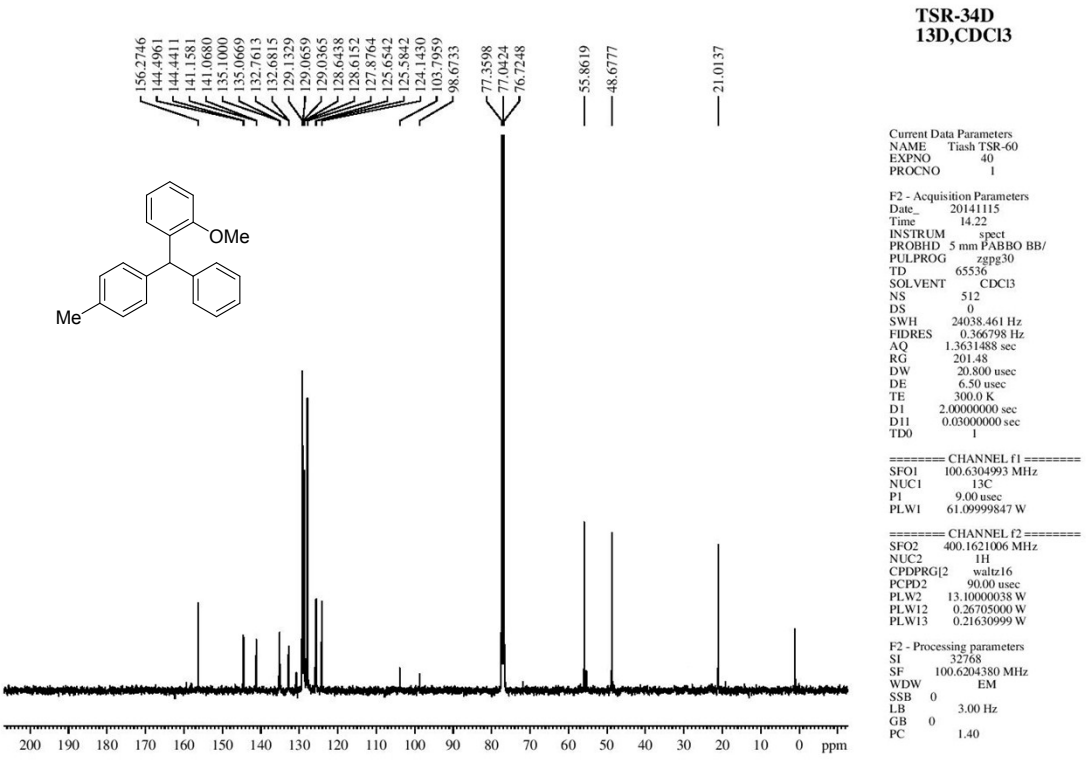


Figure 6. ^{13}C spectrum of compound **3bf**

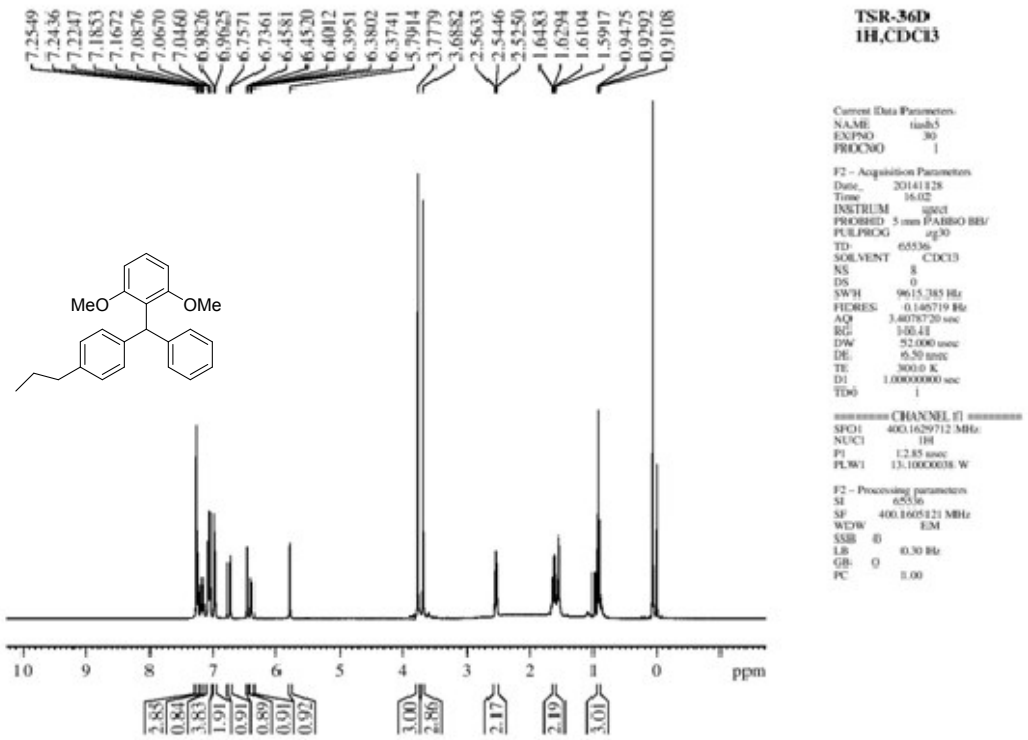


Figure 7. [1]H spectrum of compound **3ac**

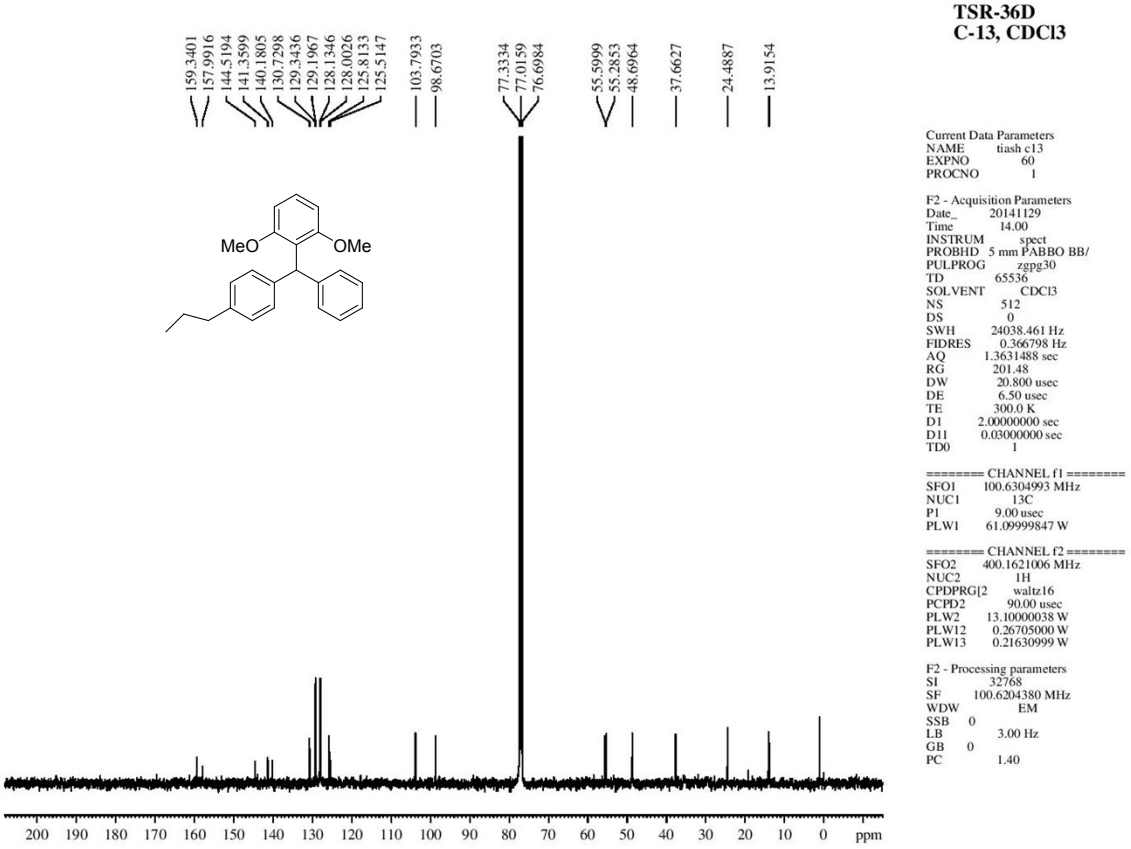


Figure 8: ^{13}C spectrum of compound **3ac**

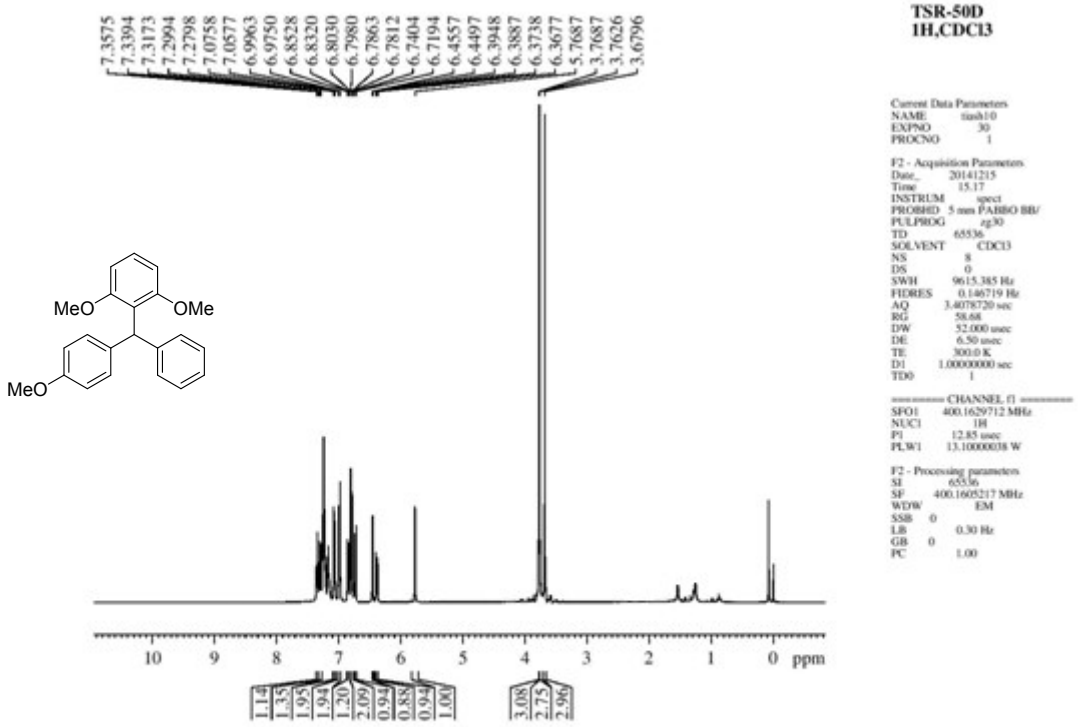


Figure 9. [1]H spectrum of compound **3ad**

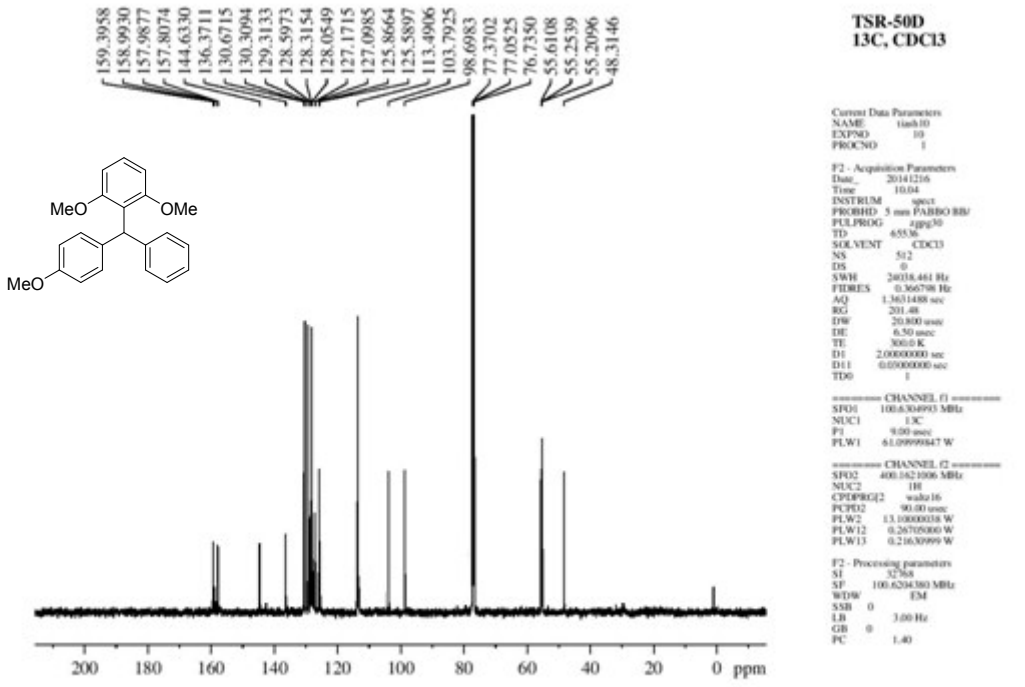


Figure 10. [13]C spectrum of compound **3ad**

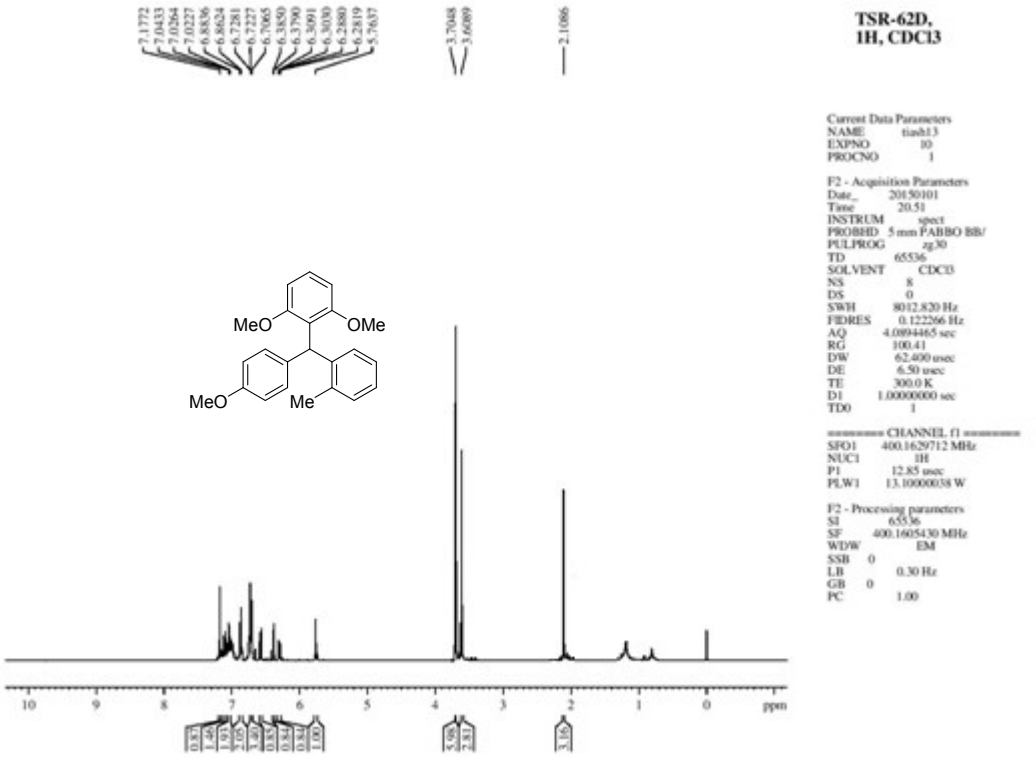


Figure 11. ^{1}H spectrum of compound **3ae**

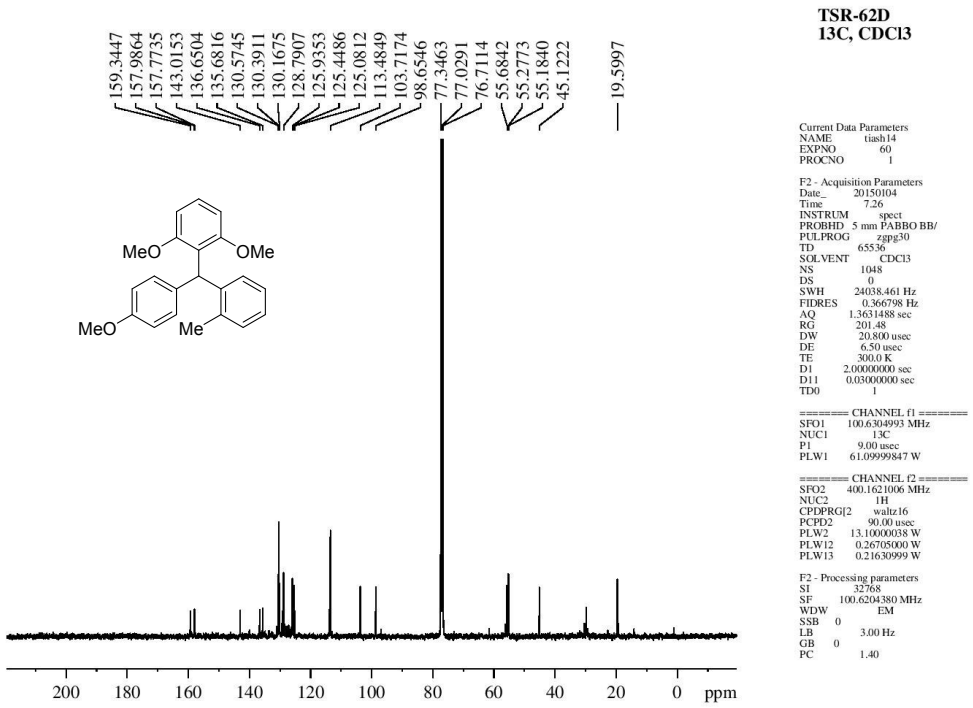


Figure 12. ^{13}C spectrum of compound **3ae**

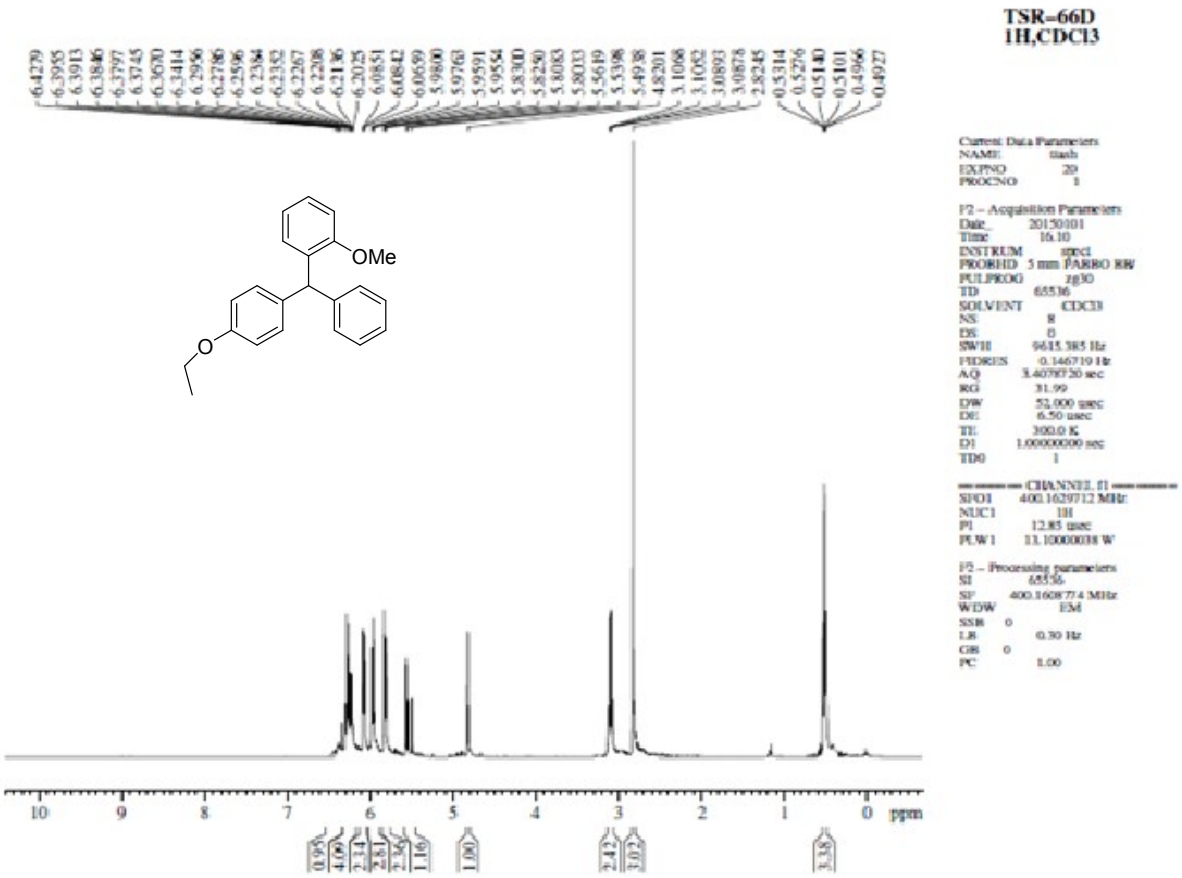


Figure 13. ^{1}H spectrum of compound **3bg**

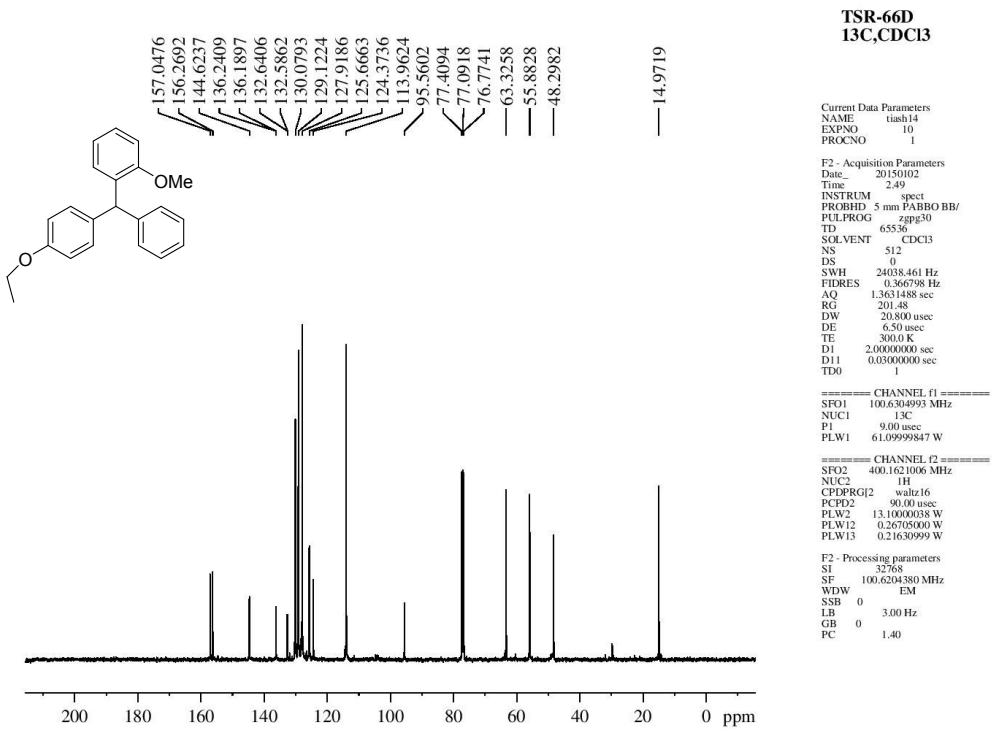


Figure 14. ^{13}C spectrum of compound **3bg**

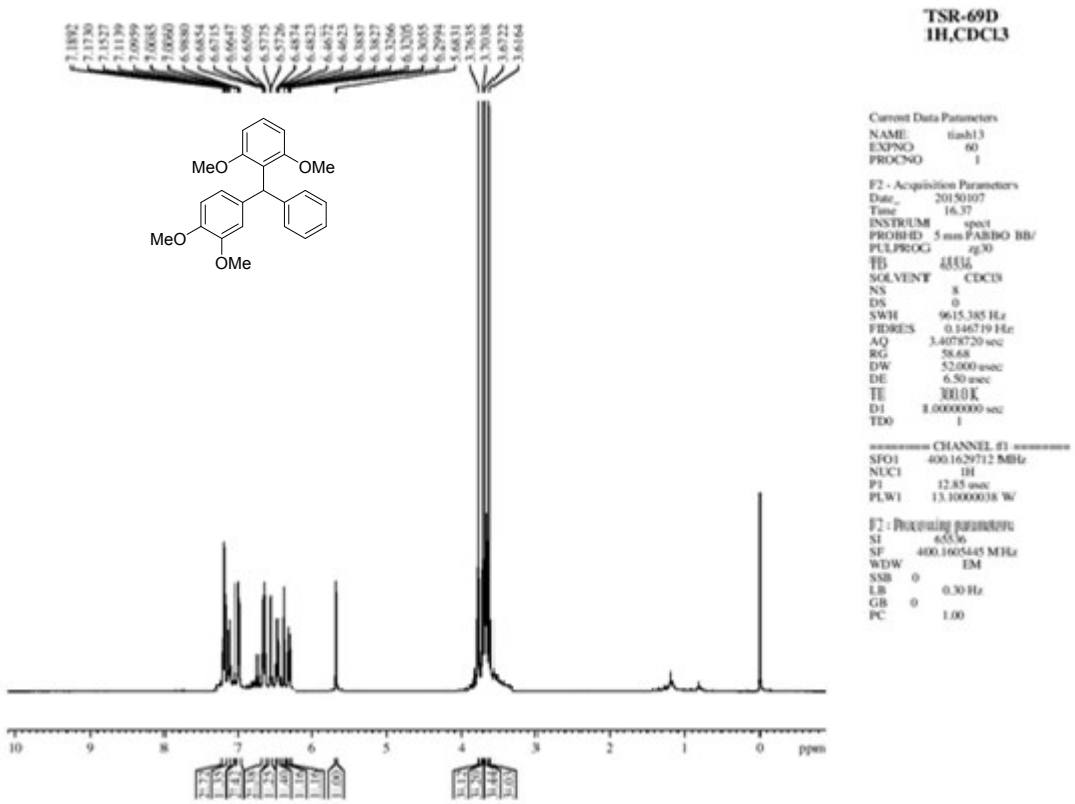


Figure 15. ^{1}H spectrum of compound **3ah**

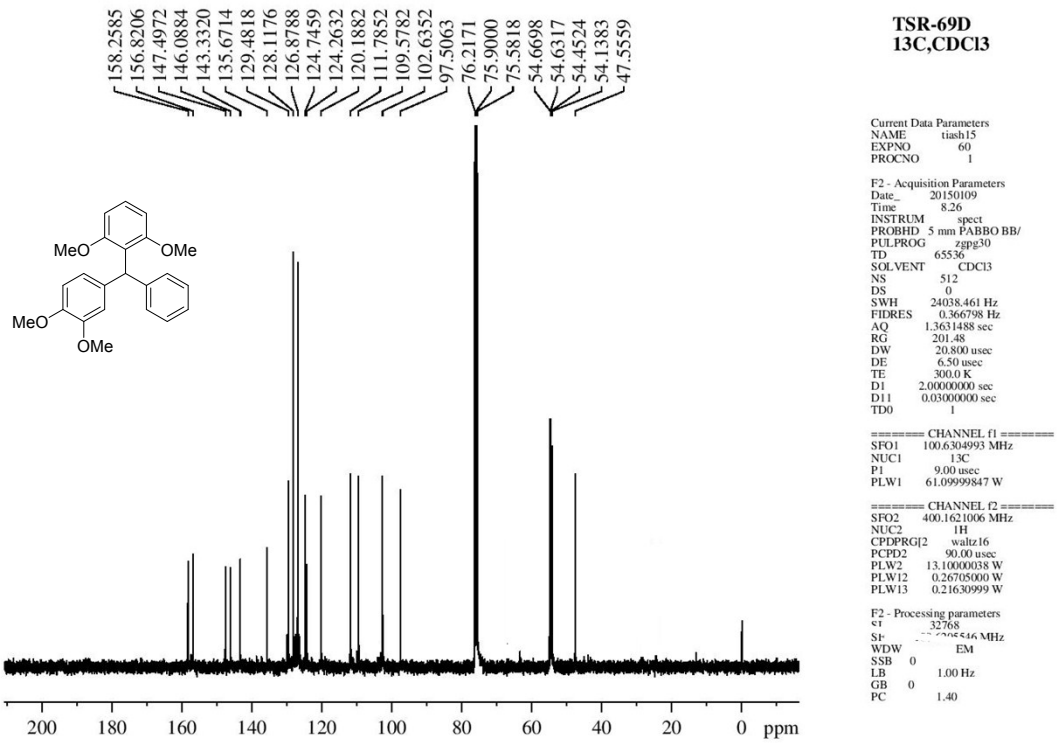


Figure 16. ^{13}C spectrum of compound **3ah**

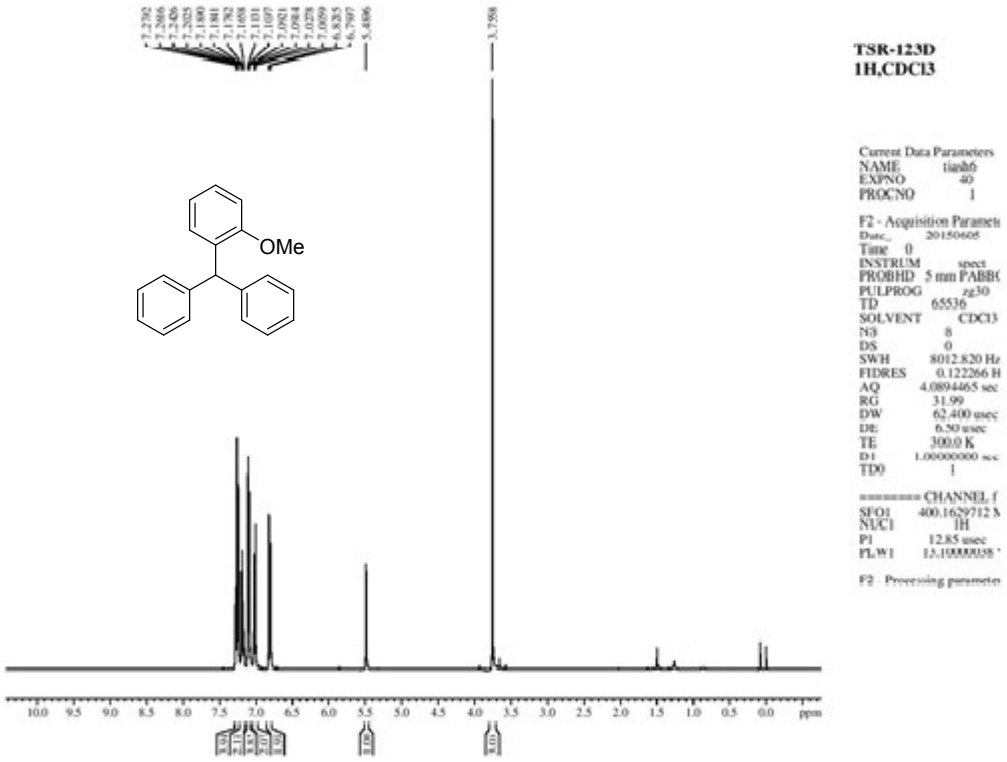


Figure 17. ^{1}H spectrum of compound 3ba

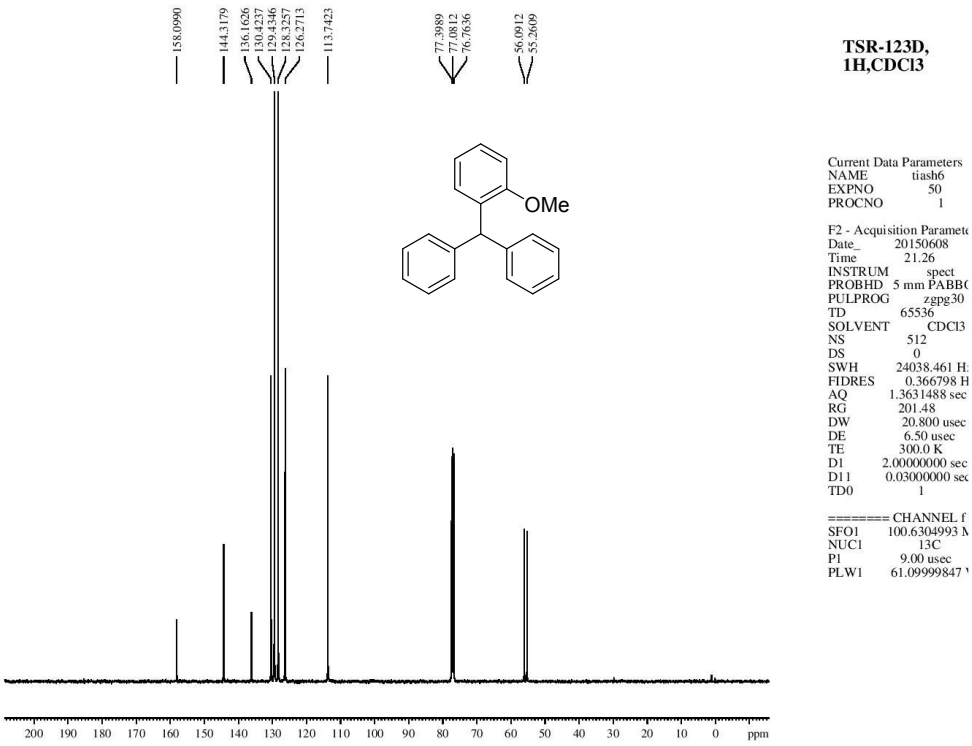


Figure 18. ^{13}C spectrum of compound **3ba**

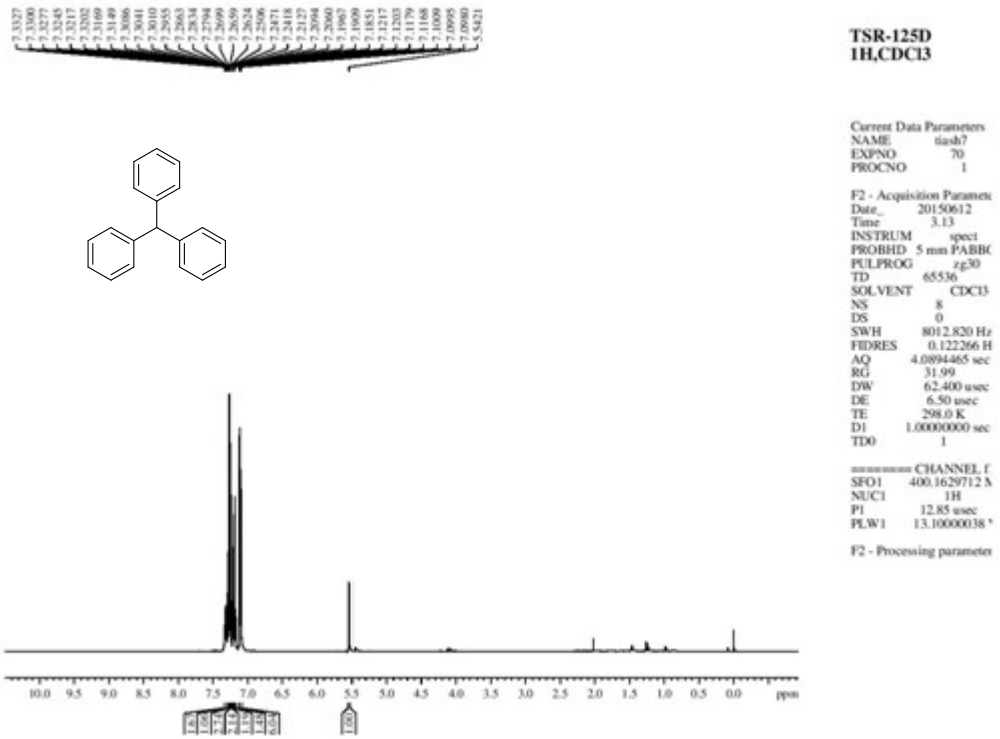


Figure 19. ^{1}H spectrum of compound **3ca**

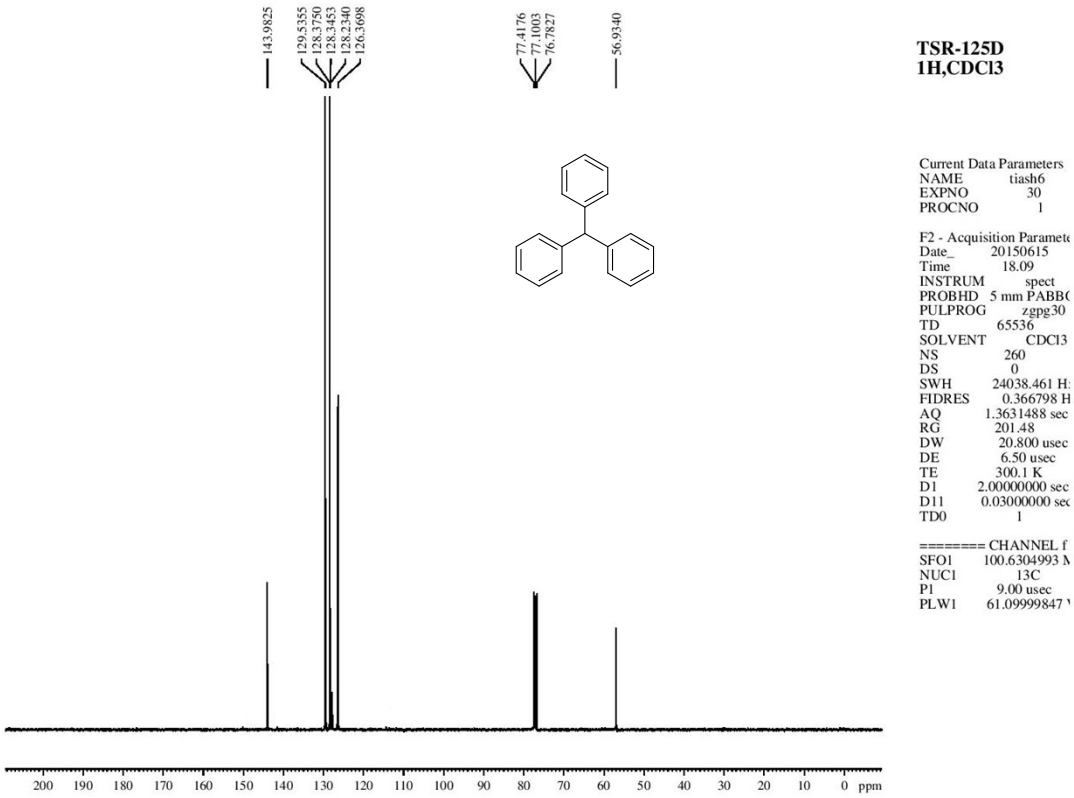


Figure 20. ^{13}C spectrum of compound **3ca**

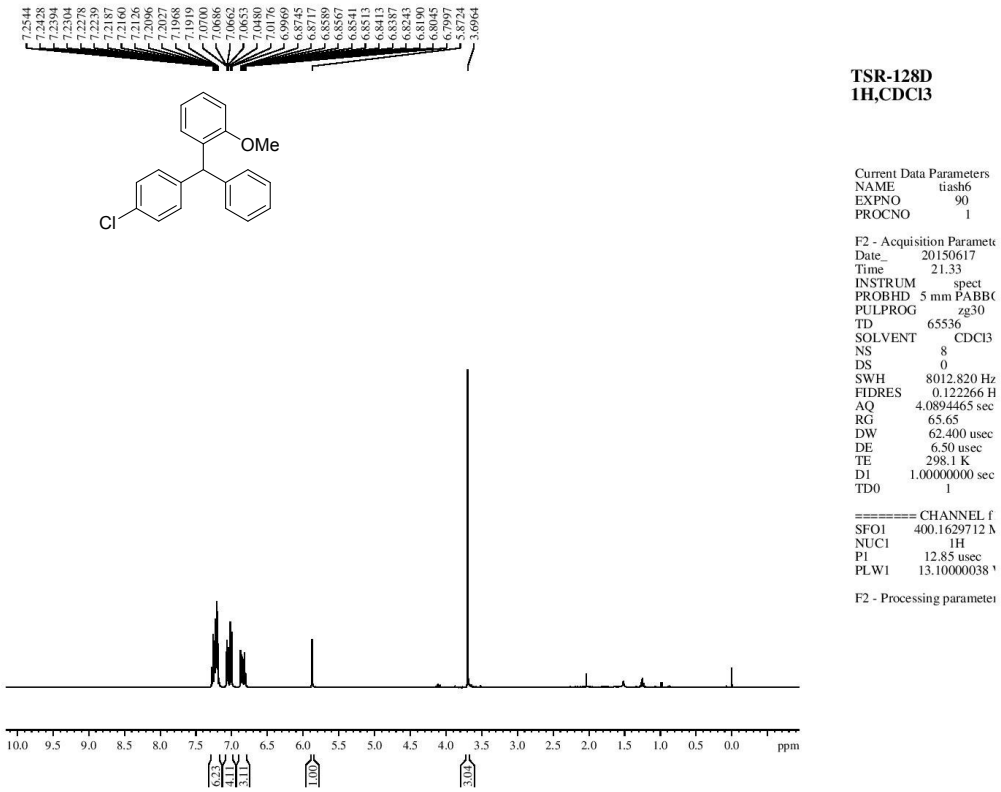


Figure 21. ^{1}H spectrum of compound **3bi**

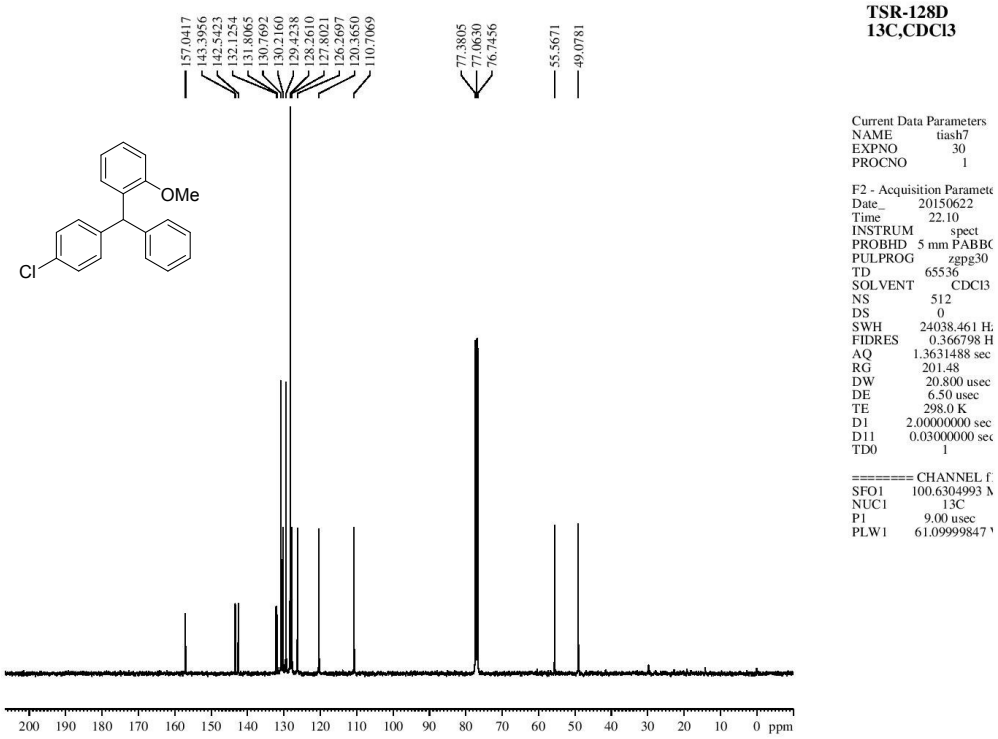


Figure 22. ^{13}C spectrum of compound **3bi**

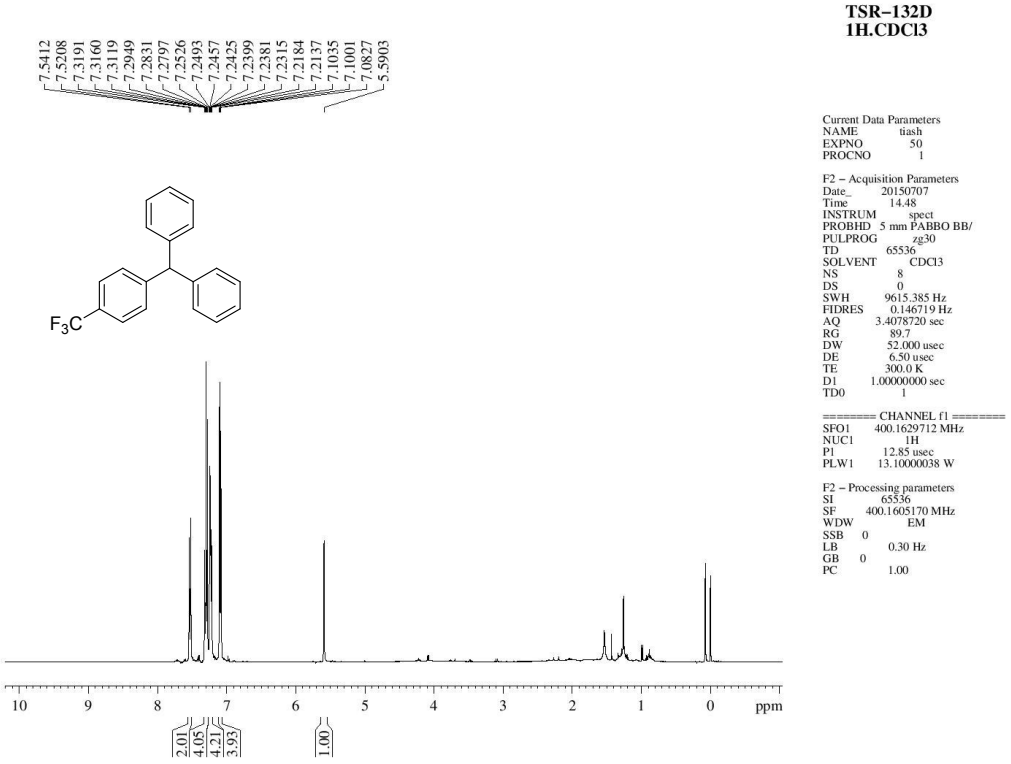


Figure 23. ^{1}H spectrum of compound **3cj**

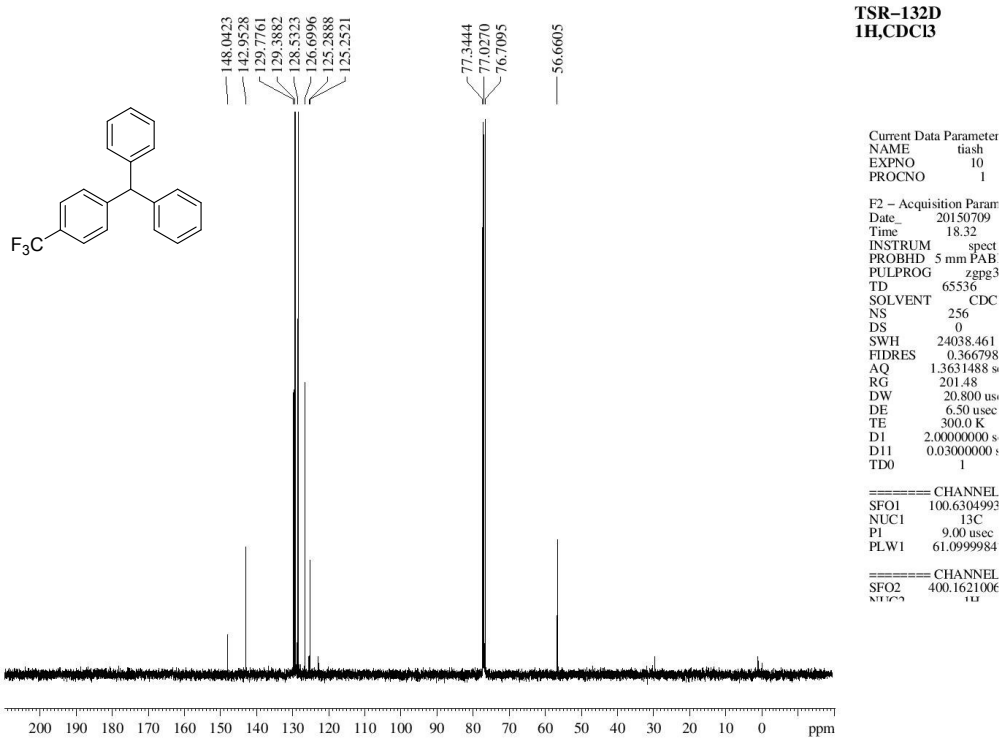


Figure 24. ^{13}C spectrum of compound **3cj**

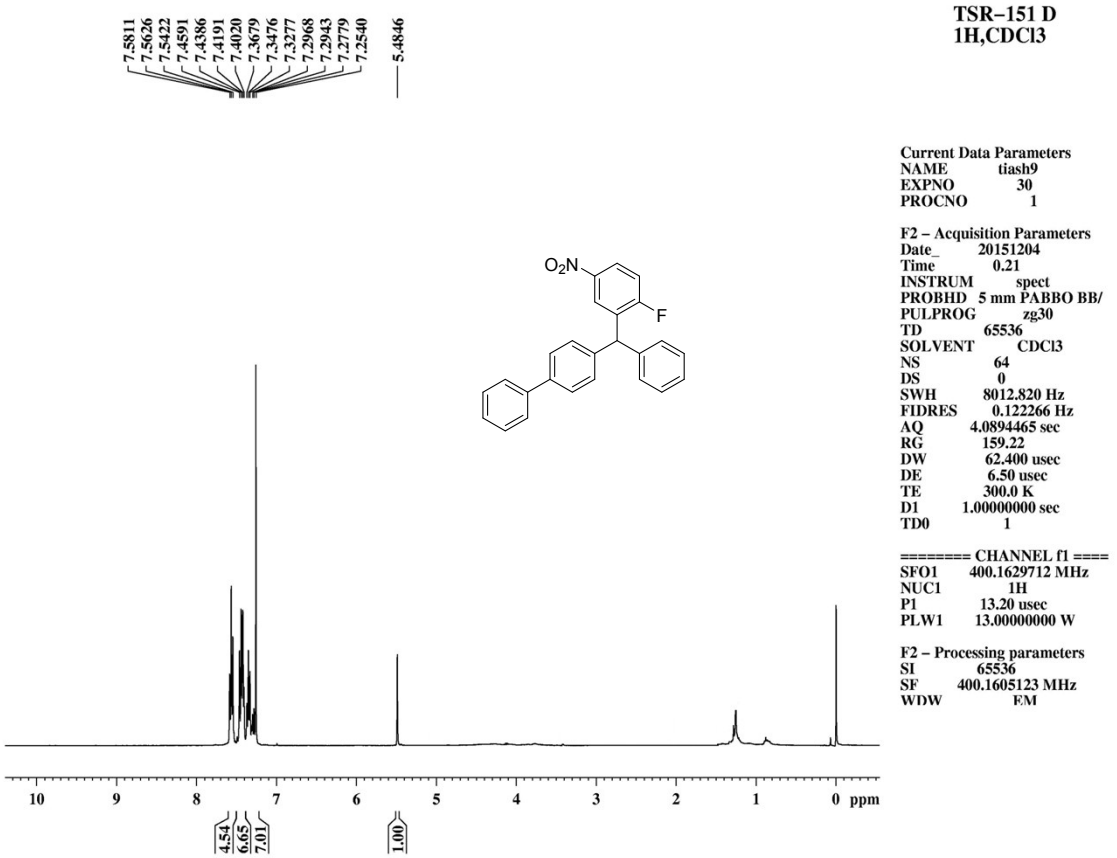


Figure 25. ^{1}H spectrum of compound **3dk**

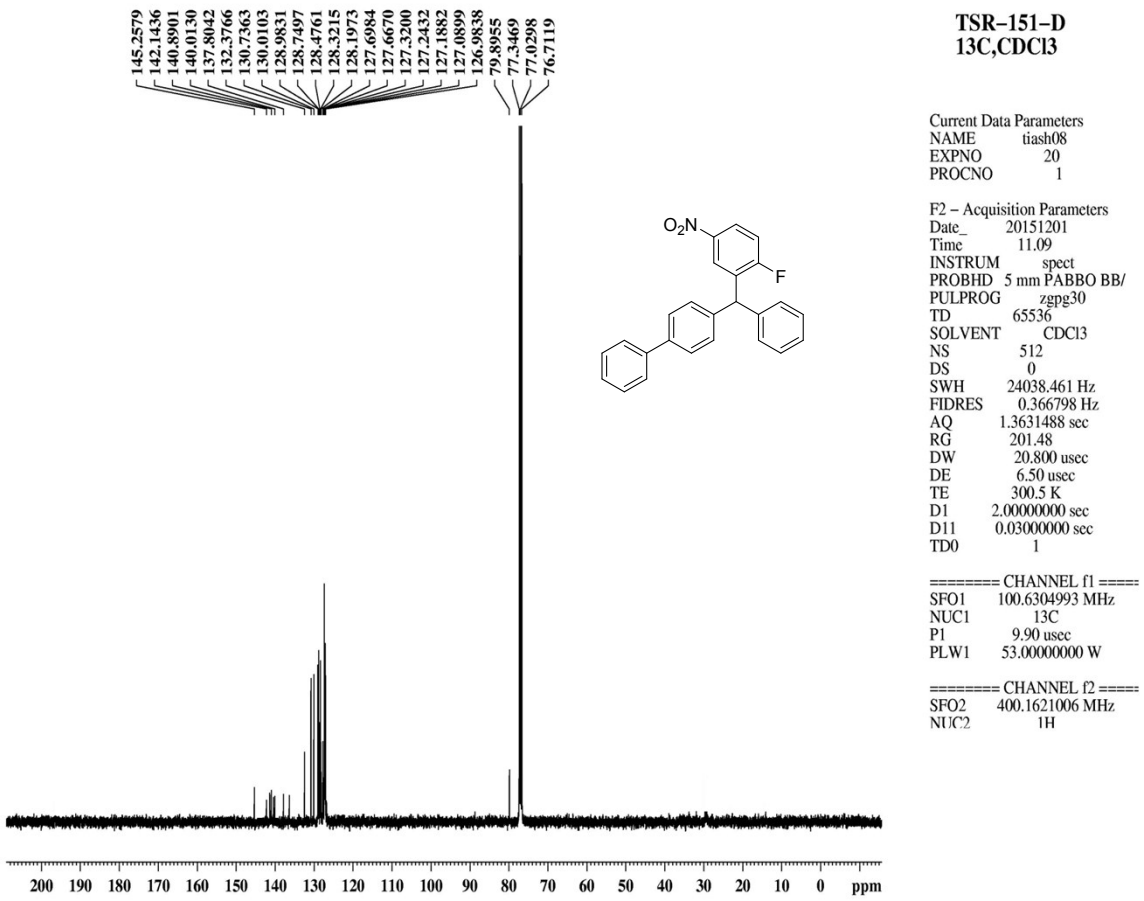


Figure 26. ^{13}C spectrum of compound **3dk**

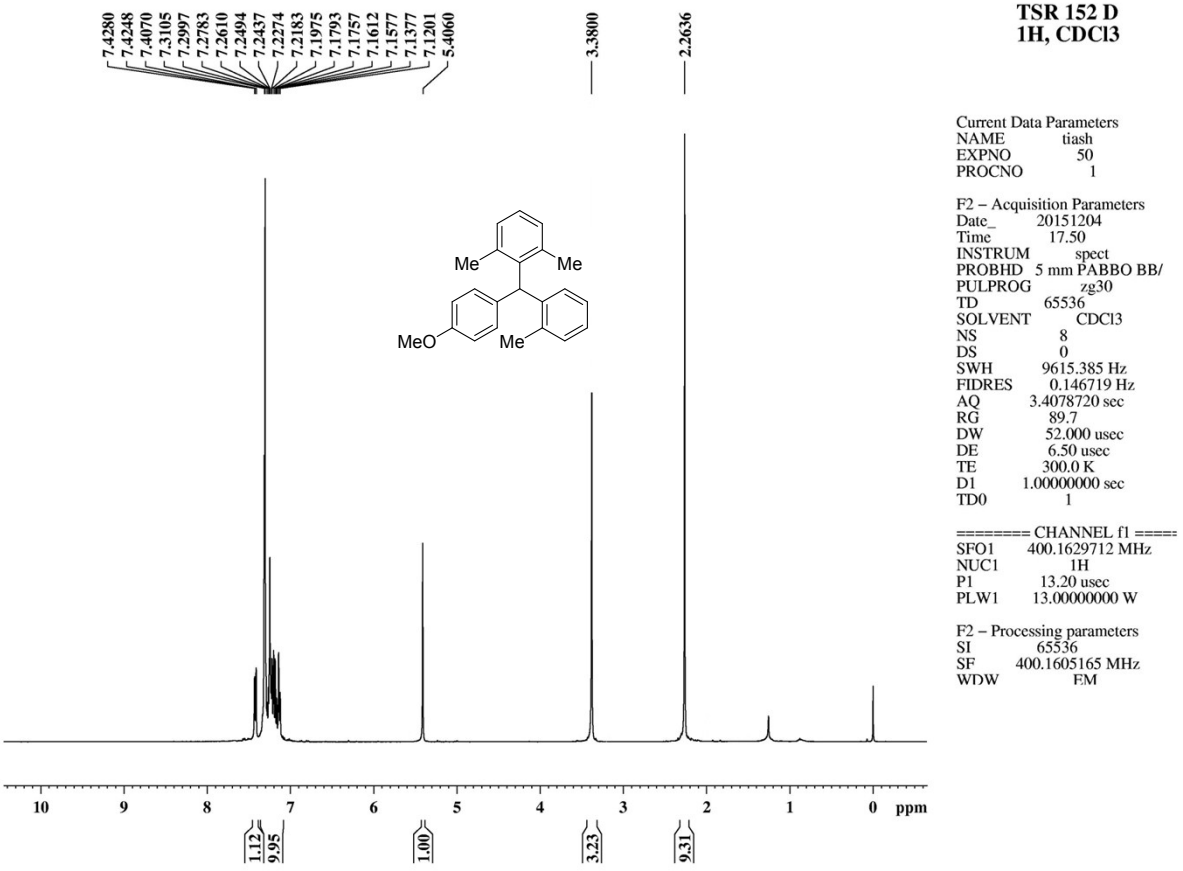


Figure 27. ^{1}H spectrum of compound **3ef**

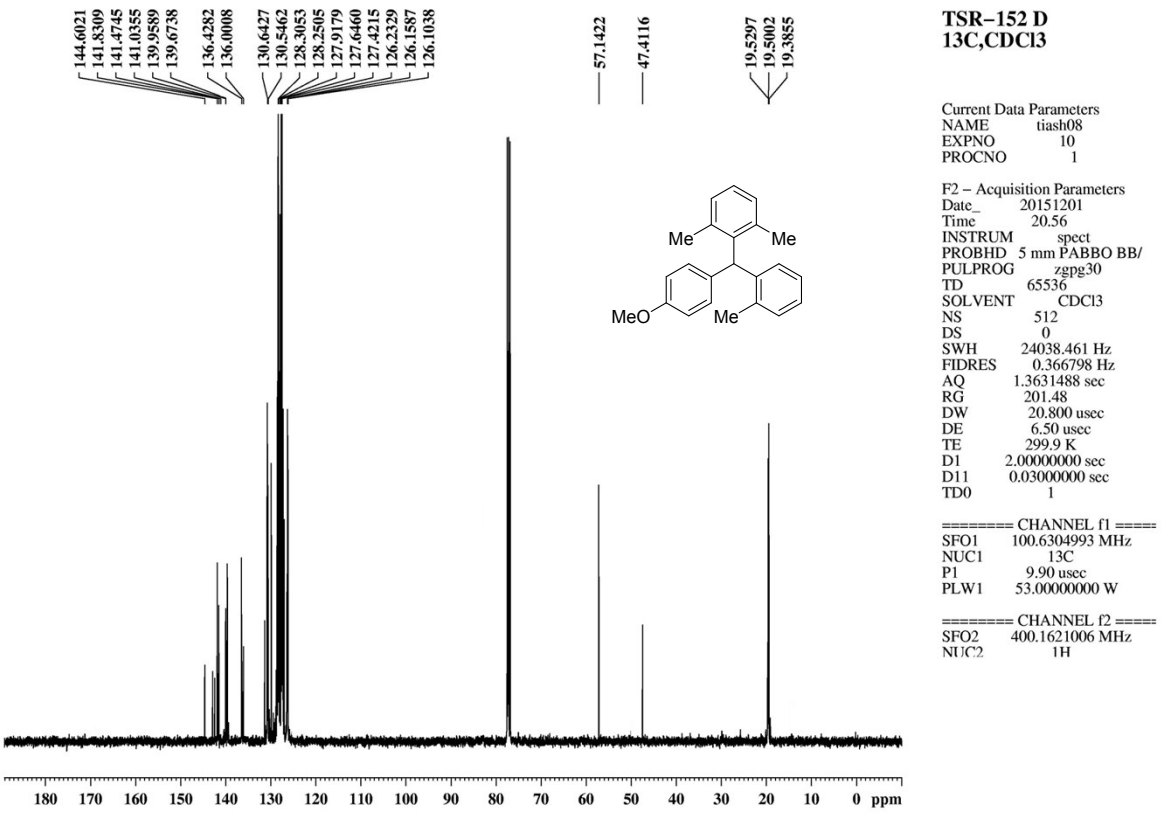


Figure 28. ^{13}C spectrum of compound **3ef**

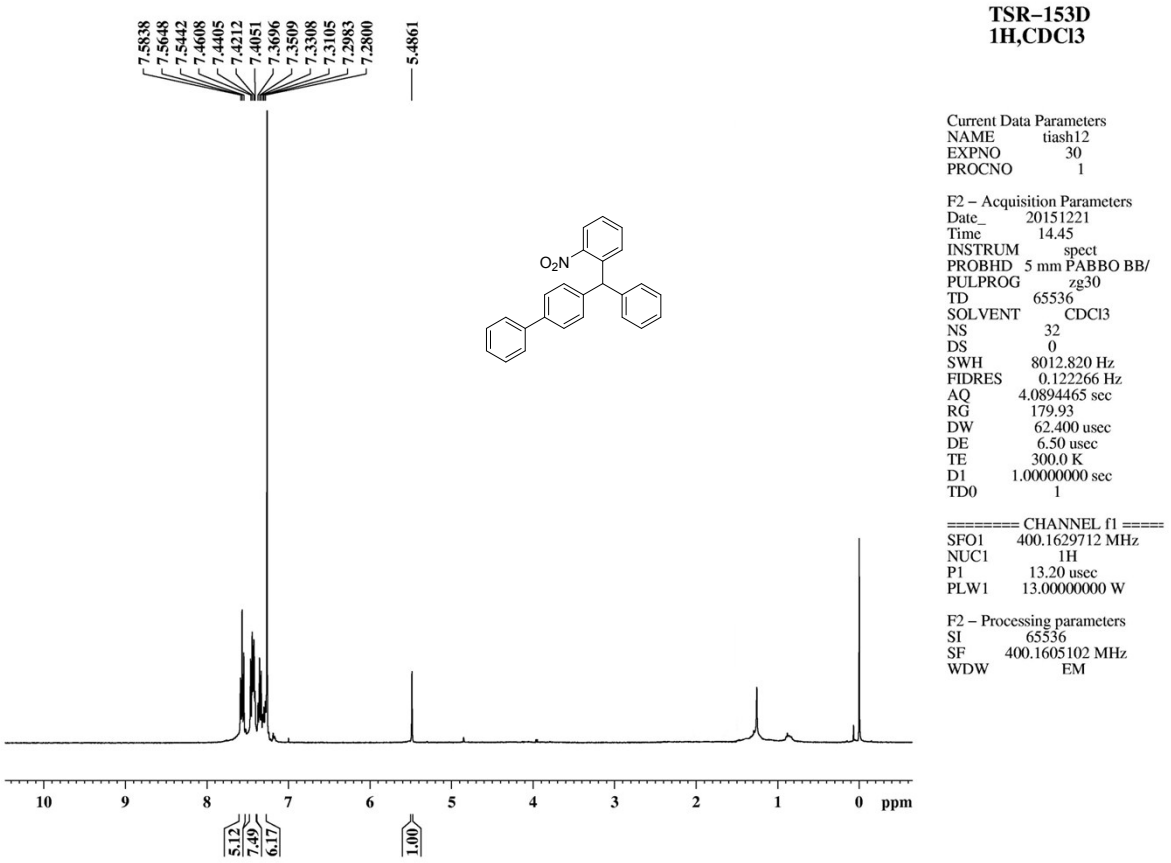


Figure 29. ^{1}H spectrum of compound **3fk**

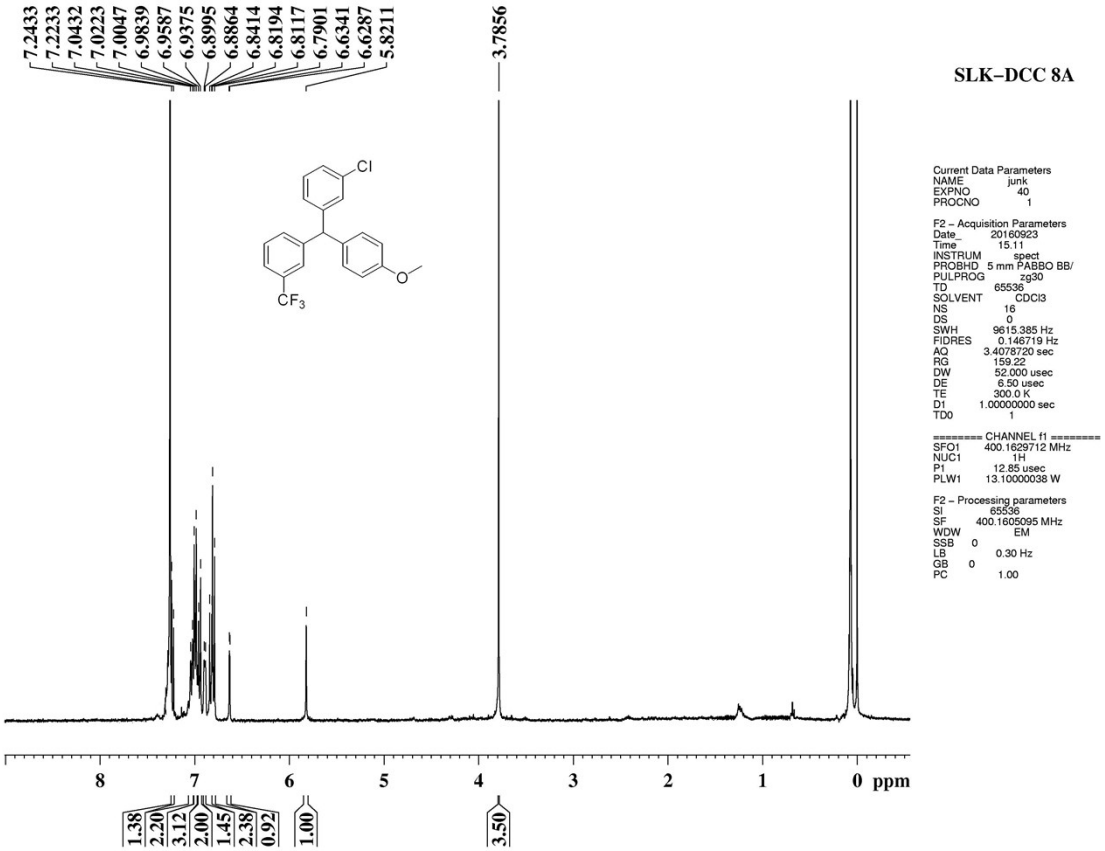


Figure 30. ^{1}H spectrum of compound 3hl

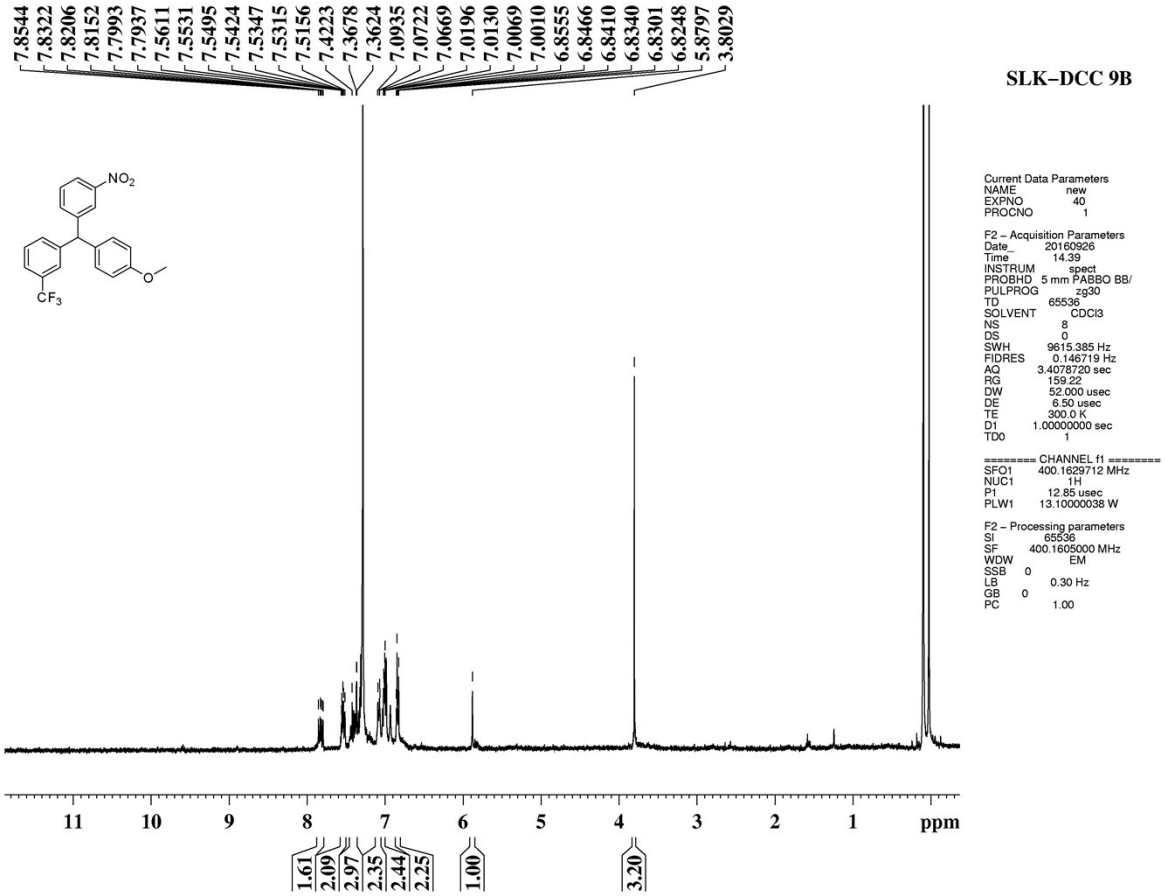


Figure 31. ^{1}H spectrum of compound **3gl**

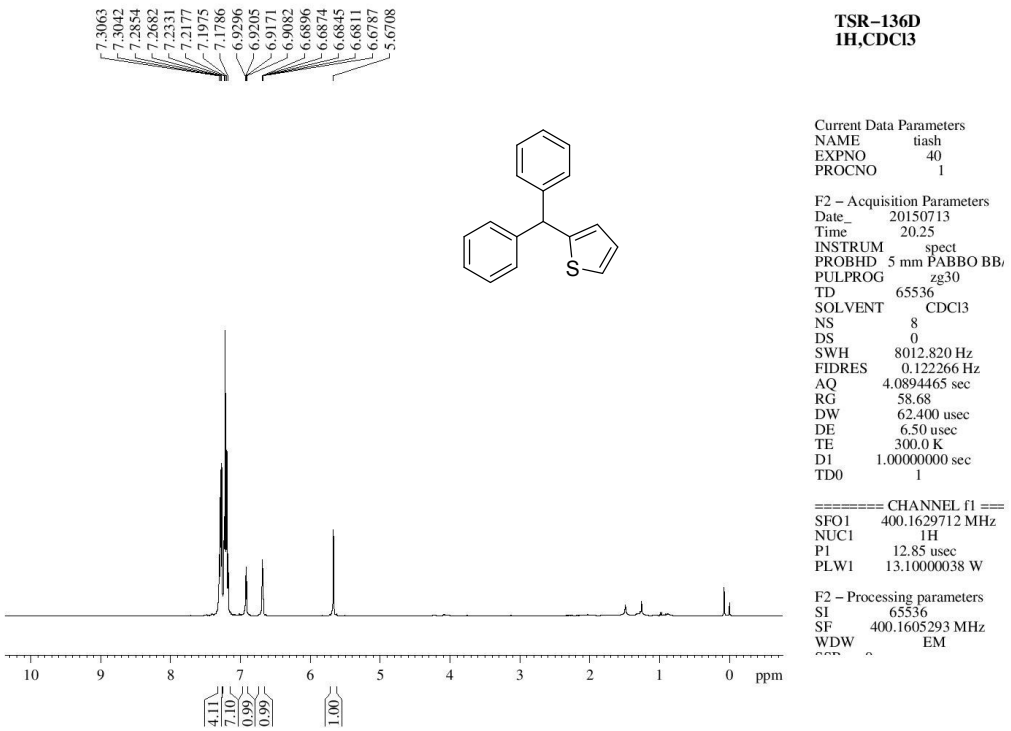


Figure 32. ^{1}H spectrum of compound **5**

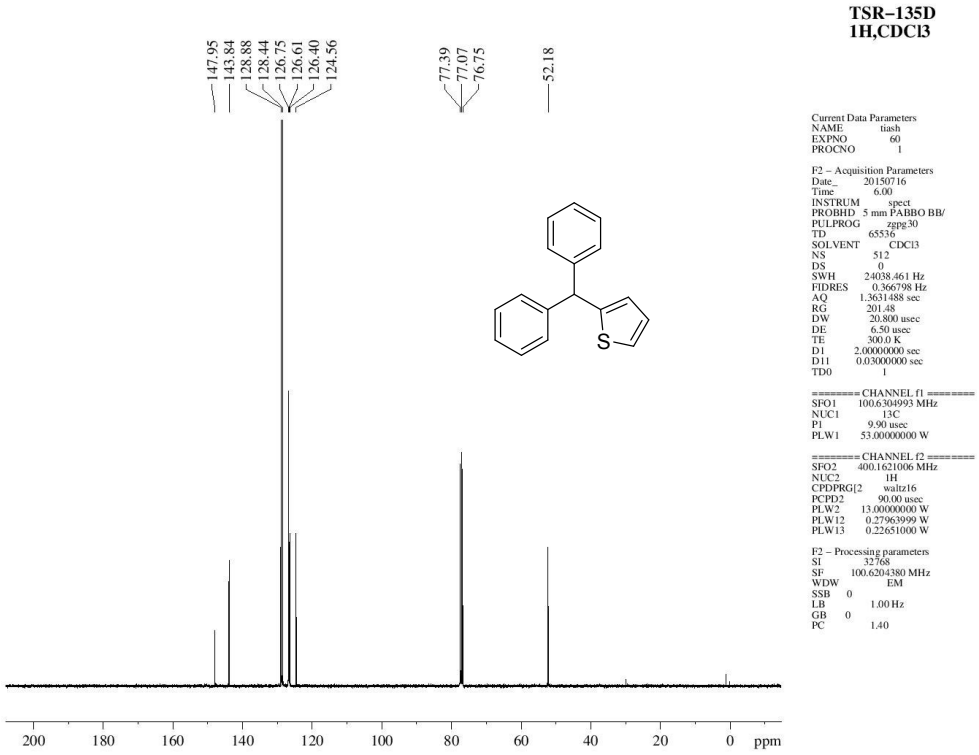


Figure 33. ^{13}C spectrum of compound **5**