Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting Information

A Well-defined Low-Valent Cobalt Catalyst $Co(PMe_3)_4$ with Dimethylzinc : A Simple Catalytic Approach for the Reductive Dimerization of Benzyl Halides

Brendan J. Fallon, Vincent Corcé, Muriel Amatore, Corinne Aubert, Fabrice Chemla, Franck Ferreira, Alejandro Perez-Luna, Marc Petit*

Institut Parisien de Chimie Moléculaire, UMR 8232, Université Pierre et Marie Curie-Paris 6, Case 229, 4 Place Jussieu, 75252 Paris, France

List of the Contents of the Supporting Information

I. General information:

Commercial reagents were purified prior to use following the guidelines of Perrin and Armarego.[1] Tetrahydrofuran was purified by mean of distillation under dry nitrogen atmosphere on benzophenone/sodium and degassed by freeze-pump-thaw technique. Organic solutions were concentrated under reduced pressure on a Büchi rotary evaporatory. Chromatographic purifications of products were accomplished using force-flow chromatography on Davisil (LC60A) SI 60 Å (40 – 63 μm) silica gel according to the method of Still.[2] Thin layer chromatography (TLC) was performed on Merck 60 F254 silica gel plates. TLC visualization was performed by fluorescence quenching (λ = 254 nm), dipping in $KMnO_4$ or *para*-anisaldehyde stains. Filtrations through Celite were performed using Hyflo Super Cel from Fluka. ^{1}H NMR spectra were recorded on a Brucker 400 AVANCE or 300 AVANCE (400 and 300 MHz respectively) and are referenced relative to residual $CDCl_3$ protons signals at δ 7.26 ppm. ^{13}C NMR spectra were recorded on a Brucker 400 AVANCE or 300 AVANCE (100 and 75 MHz respectively) and are referenced relative to $CDCl_3$ at δ 77.00 ppm. ^{19}F NMR spectra were recorded on a Brucker 400 AVANCE (376 MHz) and are referenced relative to $CFCl_3$ at δ 0.00 ppm. Data are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, qt = quintuplet, m = multiplet, bs = broad signal), coupling constant (Hz) and integration. IR spectra were recorded on a Bruker Tensor 27 (ATR diamond) and are reported in terms of frequency of absorption (cm-1). High-resolution mass spectra were obtained from the Laboratoire Structure et Fonction de Molécules Bioactives (Université Pierre et Marie Curie, Paris 6).

II. Mechanistic studies:

Role of Zinc in the reaction: see table 2 in communication for details

A number of control experiments were ran in order to determine the role of dimethyl zinc.

1. **Absence of Me_2Zn:** The reaction of **1a** with $Co(PMe_3)_4$ 10 mol %, 66 ^oC, 10 mins, THF) in the absence of Me_2Zn yields the desired product of homo-coupling **2a** with a conversion of ~20 % as determined by gas chromatography (GC) using decan as internal standard. This suggests $Co(PMe_3)_4$ is capable of performing two single electron transfers and the catalytic activity is likely to be lost due to the formation of $X_2Co(PMe_3)_3$ (where X = Br, Cl)

2. **Use of $ClCo(PMe_3)_3$ as catalyst:** Based on our previous result in the absence of zinc we hypothesised that a two electron transfer process was in operation for catalyst $Co(PMe_3)_4$. Thus in the presence of $ClCo(PMe_3)_3$ we expected that only a single electron transfer could be achieved therefore facilitating a single homo-coupling cycle. Gratifyingly this proved to be the case. The reaction of **1a** under standard reaction conditions $ClCo(PMe_3)_3$ 10 mol %, 66 ^oC, 10 mins, THF) in the absence of Me_2Zn yielded the desired product of homo-coupling with a conversion of ~10 % as determined by GC.

   If our proposed theory was correct and the catalytic activity was loss due to the generation of $X_2Co(PMe_3)_3$ then the introduction of Me_2Zn into the reaction with $ClCo(PMe_3)_3$ should regenerate the active cobalt(0) $Co(PMe_3)_3$ through a transmetalation process followed by loss of ethane gas. Pleasingly this proved to be the case and we could efficiently reactivate the catalyst by introduction of Me_2Zn (0.6 equiv).

3. **Use of $Cl_2Co(PMe_3)_3$ as catalyst:** As further proof of the regeneration of our cobalt catalyst through a transmetalation step followed by elimination of ethane we performed a series of experiments with $Cl_2Co(PMe_3)_3$. As expected no conversion to the desired homo-coupling product was observed through the introduction of $Cl_2Co(PMe_3)_3$ (20 mol %) at 66 ^oC, for 10 mins. However, by introducing Me_2Zn (0.6 equiv) (gaseous evolution observed) we could regenerate the active catalytic species and the desired homo-coupling product was observed.

4. **Use of $ClCo(PPh_3)$ as catalyst.**
   As previously reported the use of stoichiometric amounts of $ClCo(PPh_3)_3$ could generate the homo-coupling product. However, the catalyst could not be efficiently regenerated in the presence of Me_2Zn (1.2 equiv). This suggests an important role for the phosphine ligand.

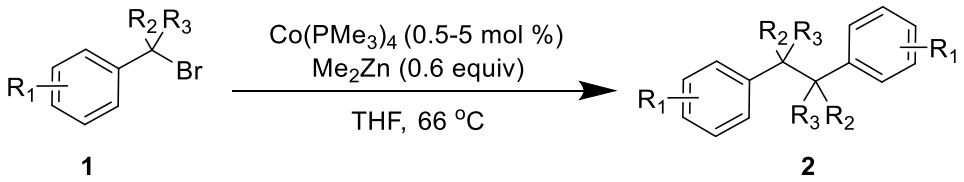
III. Preparation of starting materials:

The following cobalt catalysts were synthesized according to literature methods and showed good agreement with the literature data.

- $Co(PMe_3)_4$ [3]
- $ClCo(PMe_3)_3$ [4]
- $Cl_2Co(PMe_3)_3$ [5]

IV. Characterization of compounds:

General procedure for the cobalt catalyzed homo-coupling of benzyl halides:

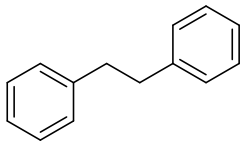


1 **2**

To a solution of $Co(PMe_3)_4$ (0.5-5 mol %) in degassed THF (0.5 mL) was added 1.0 M Me_2Zn in hexane (0.3 mL, 0.3 mmol) followed by benzyl halide (**1**; 0.5 mmol) at ambient temperature. The reaction was then transferred to an oil bath and heated to 66 ^{o}C for between 10-60 mins. The resulting mixture was quenched with 10 % HCl, and extracted with AcOEt. The AcOEt layer was washed with brine and dried over $MgSO_4$. The solvent was removed *in vacuo* and the residue was purified by column chromatography on silica to give the desired product.

Spectroscopic data:

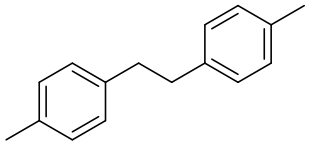
1,2-Diphenylethane (2a) :



Chemical Formula: $C_{14}H_{14}$

Colorless solid; **m.p**. 50.5–51.0 °C. **^{1}H NMR (400 MHz, CDCl3)** δ: 7.25–7.30 (m, 4H) 7.18–7.21 (m, 6H), 2.92 (s, 4H), **^{13}C NMR (100 MHz, $CDCl_3$)** δ 141.8 (2C), 128.5(4C), 128.4 (4C), 125.9 (2C), 38.0 (2C). **IR (neat, cm-1): ν max** 3005, 2900, 2845, 1775, 1505, 1401 Spectral data in good accordance with reported literature values.[6]

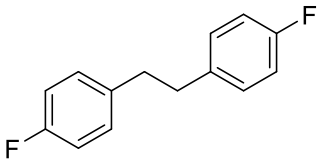
1,2-di-*p*-tolylethane (2b):



Chemical Formula: $C_{16}H_{18}$

Colorless solid; **m.p.** 78.5–79.5 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.12 (s, 8H), 2.90 (s, 4H), 2.36 (s, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 138.8 (2C), 135.2 (2C), 128.9 (4C), 128.2 (4C), 37.6 (2C), 21.0 (2C). **IR (neat, cm-1): ν max** 2901, 2839, 1498, 1412. Spectral data in good accordance with reported literature values.[6]

1,2-*Bis*(4-fluorophenyl)ethane (2c):

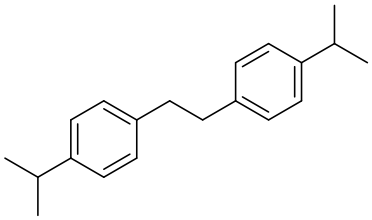


Chemical Formula: $C_{14}H_{12}F_2$

Colorless solid; **m.p.** 91.0-92.0 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.03 – 6.97 (dd, J = 6.1, 3.2 Hz, 4H), 6.91 – 6.83 (app. t, *J* = 8.8 Hz, 4H), 2.79 (s, 4H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 161.3 (d, *J* = 243.7 Hz, 2C), 136.9 (d, *J* = 3.2 Hz, 2C), 129.8 (d, *J* = 7.8 Hz(4C)), 115.1 (d, J = 21.6 Hz,

4C), 37.2 (2C). **IR (neat, cm-1): ν max** 2901, 1530, 1216. Spectral data in good accordance with reported literature values. [6]

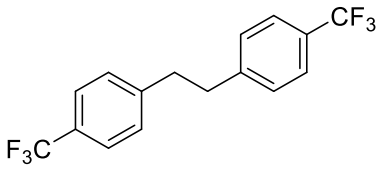
1,2-*Bis*(4-isopropylphenyl)ethane (2d):



Chemical Formula: $C_{20}H_{26}$

Colorless solid; **m.p**. 94–96 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.19 (s, 8H), 2.97 – 2.86 (m, 6H), 1.28 (d, *J* = 6.9 Hz, 12H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 146.4 (2C), 139.4 (2C), 128.2(4C), 126.3(4C), 37.6(2C), 33.7(2C), 24.1(4C). **IR (neat, cm-1): ν max** 2956, 2856, 1513, 1459, 1142, 827. Spectral data in good accordance with reported literature values.[7]

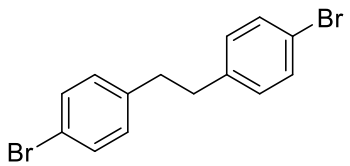
1,2-*Bis*(4-(trifluoromethyl)phenyl)ethane(2e):



Chemical Formula: $C_{16}H_{12}F_6$

Colorless solid; **m.p**. 81-83 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.57 – 7.51 (app. d, J = 7.9 Hz, 4H), 7.28 – 7.23 (app. d, J = 7.5 Hz, 4H), 3.00 (s, 4H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 145.0 (2C) , 128.7 , 128.4 , 125.3 (q, *J* = 7.5 Hz), 37.2 (2C). **^{19}F NMR (376 MHz, $CDCl_3$)** δ -62.3. **IR (neat, cm-1): ν max** 2955, 2866, 1616, 1321, 1173, 1115, 1069, 834. Spectral data in good accordance with reported literature values[8]

1,2-*Bis*(4-bromophenyl)ethane (2f):

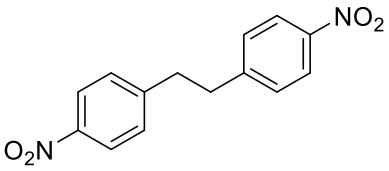


Chemical Formula: $C_{14}H_{12}Br_2$

Colorless solid; **m.p**. 117–118 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.42 – 7.35 (app. d, J = 6.8 Hz, 4H), 7.03 – 6.96 (app. d, J = 6.8 Hz, 4H), 2.85 (s, 4H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 140.0 (2C),

131.4 (4C), 130.2 (4C), 119.8 (2C), 37.0 (2C). **IR (neat, cm-1): ν max** 2922, 1479, 1285. Spectral data in good accordance with reported literature values.[6]

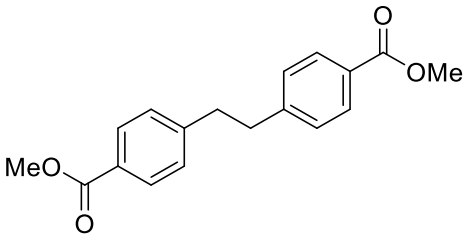
1,2-*Bis*(4-nitrophenyl)ethane (2g):



Chemical Formula: $C_{14}H_{12}N_2O_4$

Yellow solid; **m.p**. 175–177 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.17 – 8.12 (app. d, J = 7.8 Hz, 4H), 7.30 – 7.26 (app. d, J = 7.8 Hz, 4H), 3.08 (s, 4H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 148.0 (2C), 146.7 (2C), 129.2 (4C), 123.8 (4C), 36.9 (2C). **IR (neat, cm-1): ν max** 3007, 2934, 1595, 1512, 1341, 854. Spectral data in good accordance with reported literature values.[7]

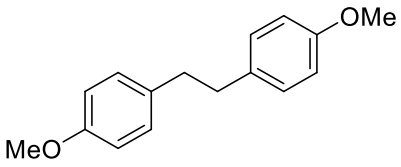
Dimethyl 4,4'-(ethane-1,2-diyl)dibenzoate (2h)



Chemical Formula: $C_{18}H_{18}O_4$

Colorless solid; **m.p.** 120-121 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.97 – 7.90 (app. d, J = 7.5 Hz, 4H), 7.22 – 7.16 (app. d, J = 7.5 Hz, 4H), 3.90 (s, 6H), 2.98 (s, 4H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 167.0 (2C), 146.4 (2C), 129.7 (4C), 128.5 (4C), 128.1 (2C), 52.0 (2C), 37.4 (2C). **IR (neat, cm-1): ν max** 2957, 1702, 1301, 1104. Spectral data in good accordance with reported literature values.[6]

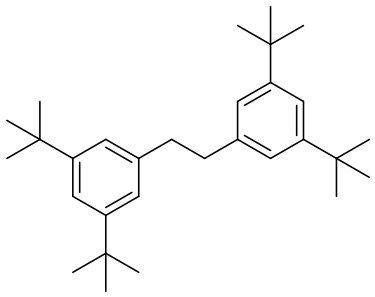
1,2-*Bis*(4-methoxyphenyl)ethane (i)



Chemical Formula: $C_{16}H_{18}O_2$

Colourless solid; **m.p.** 128-130 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.05 – 6.97 (app. d, J = 7.4 Hz, 4H), 6.79 – 6.70 (app. d, J = 7.4 Hz, 4H), 3.72 (s, 6H), 2.76 (s, 4H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 157.7 (2C), 133.9 (4C), 129.3 (4C), 113.7 (2C), 55.2 (2C), 37.2 (2C). **IR (neat, cm-1): ν max** 2964, 2932, 2852, 1610, 1510, 1302, 1030, 832. Spectral data in good accordance with reported literature values.[8]

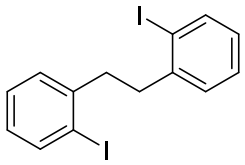
1,2-*Bis*(3,5-di-*tert*-butylphenyl)ethane (2j)



Chemical Formula: $C_{30}H_{46}$

Colorless solid; **m.p.** 94-96 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.26 (m, 2H), 7.05 (d, *J* = 1.9 Hz, 4H), 2.93 (s, 4H), 1.32 (s, 36H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 150.5 (4C), 141.0 (2C), 122.5 (4C), 119.8 (2C), 38.6 (2C), 34.7 (4C), 31.5 (12C). **IR (neat, cm-1): ν max** 1675, 1402, 1248. Spectral data in good accordance with reported literature values.[8]

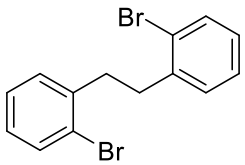
1,2-*Bis*(2-iodophenyl)ethane (2k):



Chemical Formula: $C_{14}H_{12}I_2$

Colorless solid; **m.p.** 100-102°C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.87 (dd, J = 7.9, 1.2 Hz, 2H), 7.35 – 7.22 (m, 4H), 6.94 (ddd, J = 8.0, 7.1, 2.0 Hz, 2H), 3.03 (s, 4H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 143.7 (2C), 139.4 (2C), 129.7 (2C), 128.3 (2C), 128.0 (2C), 100.6 (2C), 41.2 (2C). **IR (neat, cm-1): ν max** 2946, 2861, 1428, 1204, 1048, 951. Spectral data in good accordance with reported literature values.[9]

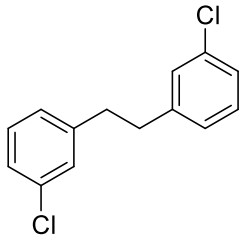
1,2-*Bis*(2-bromophenyl)ethane (2l)



Chemical Formula: $C_{14}H_{12}Br_2$

Colorless solid; **m.p.** 79-81°C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.55 (dd, *J* = 7.9, 1.2 Hz, 2H), 7.24 – 7.16 (m, 4H), 7.08 (ddd, *J* = 7.9, 6.7, 2.4 Hz, 2H), 3.05 (s, 4H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 140.5 (2C), 132.8 (2C), 130.6 (2C), 127.8 (2C), 127.4 (2C), 124.4 (2C), 36.4 (2C). **IR (neat, cm-1): ν max** 3021, 2961, 1528, 1404. Spectral data in good accordance with reported literature values.[6]

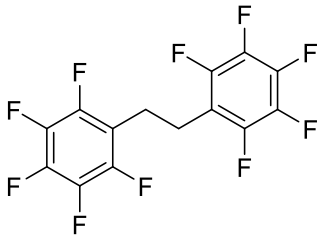
1,2-*Bis*(3-chlorophenyl)ethane (2m)



Chemical Formula: $C_{14}H_{12}Cl_2$

Colorless solid; **m.p.** 54-56°C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.23 – 7.16 (m, 6H), 7.07 – 7.01 (m, 2H), 2.89 (s, 4H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 143.1 (2C), 134.1 (2C), 129.6 (2C), 128.5 (2C), 126.6 (2C), 126.3 (2C), 37.2 (2C). **IR (neat, cm-1): v max** 2948, 2860, 1596, 1573, 1476, 1429, 1077, 817, 747. Spectral data in good accordance with reported literature values.[10]

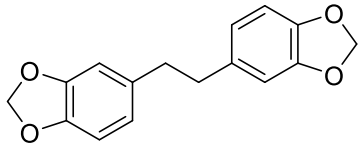
1,2-*Bis*(perfluorophenyl)ethane (2n)



Chemical Formula: $C_{14}H_4F_{10}$

Colorless solid; **m.p.** 102-104 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 3.02 (s, 4H). **^{13}C NMR (100 MHz, Chloroform-d)** δ 146.3 (ddt, *J* = 12.5, 8.3, 4.1 Hz), 143.8 (ddd, *J* = 15.3, 8.3, 4.0 Hz), 141.6 – 141.1 (m), 138.72 (tdd, *J* = 17.5, 12.9, 6.7 Hz), 136.4 – 135.8 (m), 112.7 (td, *J* = 18.7, 4.0 Hz), 21.8. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -144.3 (dd, J = 8.2, 22.6 Hz,), -155.82 (t, *J* = 20.8 Hz), -162.1 (td, J = 9.1, 13.3 Hz). **IR (neat, cm-1): v max** 2959, 2926, 1521, 1184, 1124, 960. Spectral data in good accordance with reported literature values.[6]

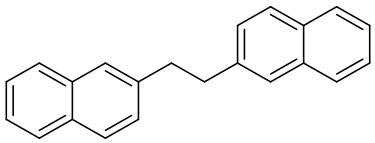
1,2-*Bis*(3,4-methylenedioxyphenyl)ethane (2o)



Chemical Formula: $C_{16}H_{14}O_4$

Colorless solid; **m.p.** 133-135 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.76 – 6.58 (m, 6H), 5.92 (s, 4H), 2.80 (s, 4H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.5 (2C), 145.6 (2C), 135.4 (2C), 121.2 (2C), 108.9 (2C), 108.1 (2C), 100.7 (2C), 37.8 (2C). **IR (neat, cm-1): v max**. 2901, 1200, 1067. Spectral data in good accordance with reported literature values.[6]

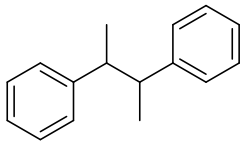
1,2-Di(naphthalen-2-yl)ethane (2p)



Chemical Formula: $C_{22}H_{18}$

Colorless solid; **m.p.** 181-183 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.85 – 7.76 (m, 6H), 7.68 – 7.65 (m, 2H), 7.49 – 7.40 (m, 4H), 7.38 (dd, *J* = 8.4, 1.8 Hz, 2H), 3.20 (s, 4H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 139.2 (2C), 133.6 (2C), 132.0 (2C), 127.9 (2C), 127.6 (2C), 127.4 (2C), 127.3 (2C), 126.5 (2C), 125.9 (2C), 125.1 (2C), 38.0 (2C). **IR (neat, cm-1): ν max** 2935, 2914, 1598, 1452, 862, 775. Spectral data in good accordance with reported literature values.[11]

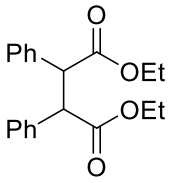
2,3-Diphenylbutane (2q):



Chemical Formula: $C_{16}H_{18}$

Colourless oil, isolated as a mixture of *dl* and *meso* compounds (48/52). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.42 – 7.34 (m, 5H,), 7.32 – 7.12 (m, 12H), 7.10 – 7.05 (m, 3H), 3.01 (m, 2H, *dl* isomer), 2.86 (m, 2H, *meso* isomer), 1.38 – 1.31 (m, 6H, *dl* isomer), 1.12 – 1.06 (m, 6H, *meso* isomer). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 146.5 (2C), 145.8 (2C), 128.3 (4C), 127.8 (4C), 127.8 (4C), 127.6 (4C), 126.0 (2C), 125.7 (2C), 47.3 (2C), 46.5 (2C), 21.0 (2C), 17.9 (2C). **IR (neat, cm-1): ν max** 2970, 2957, 1684, 1601, 1540, 1492, 697. Spectral data in good accordance with reported literature values.[6]

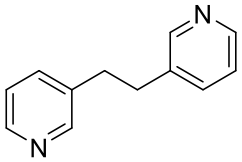
Diethyl 2,3-diphenylsuccinate (2r):



Chemical Formula: $C_{20}H_{22}O_4$

White solid. **mp** 138-139 ^{o}C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.57 – 7.49 (m, 4H), 7.40 – 7.29 (m, 6H), 4.39 (s, 2H), 3.97 – 3.80 (m, 4H), 0.95 (t, J = 7.1 Hz, 6H). **IR (neat, cm-1): ν max** 3000, 1750, 1250, 1020, 750. Spectral data in good accordance with reported literature values.[13]

1,2-Di(pyridin-3-yl)ethane (2s):

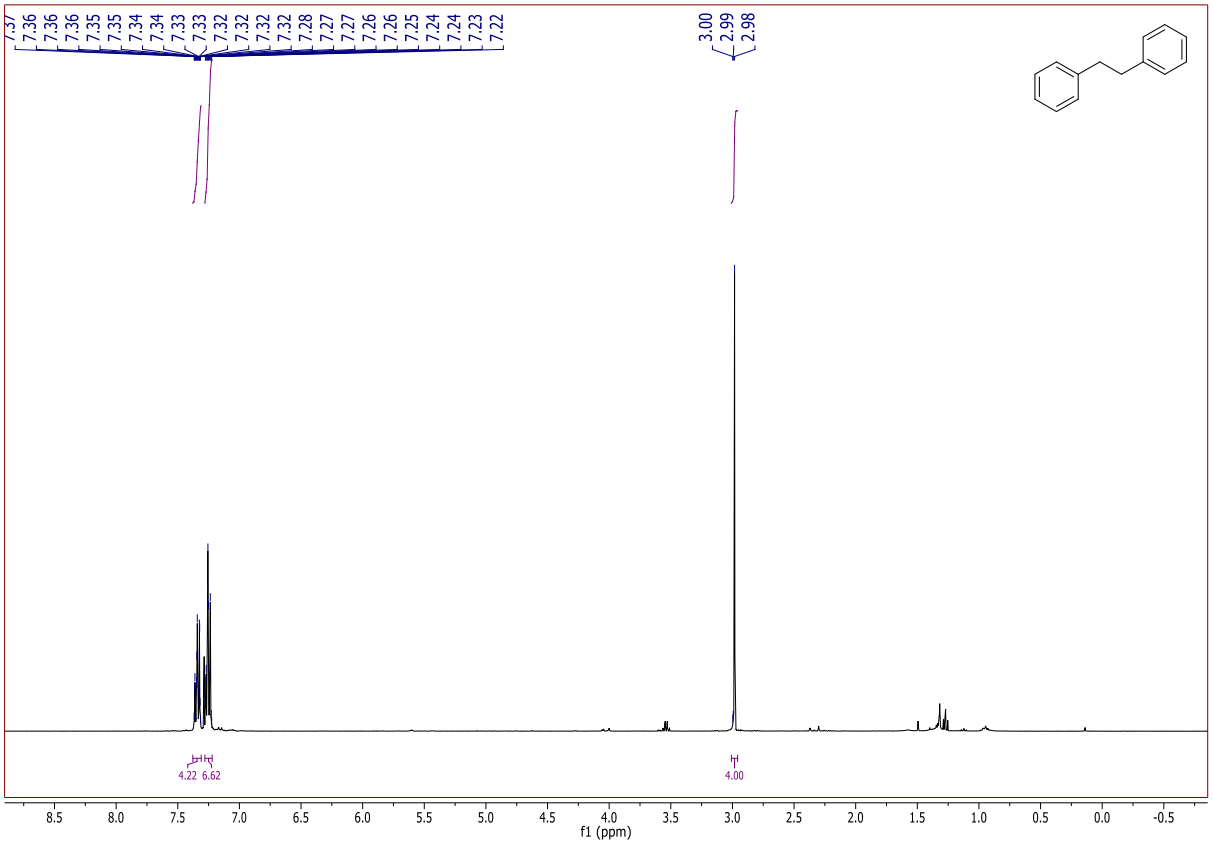


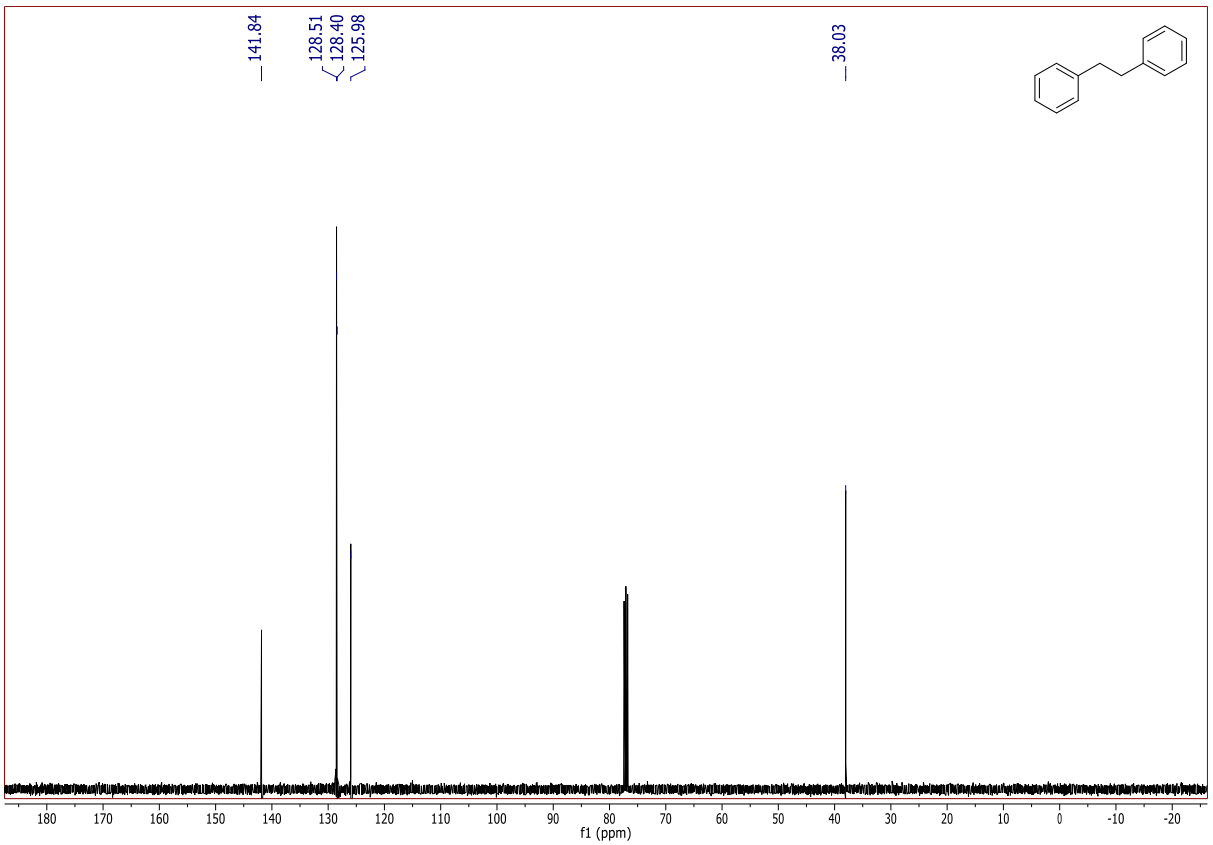
Chemical Formula: $C_{12}H_{12}N_2$

Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.41 – 8.35 (m, 4H), 7.36 (dt, *J* = 7.8, 2.0 Hz, 2H), 7.13 (ddd, *J* = 7.8, 4.8, 0.9 Hz, 2H), 2.87 (s, 4H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 149.9 (2C), 147.7 (2C), 135.9 (2C), 135.9 (2C), 123.3 (2C), 34.5 (2C). **IR (neat, cm-1): ν max** 2994, 2918, 1593, 1479, 1424, 1100, 808, 713. Spectral data in good accordance with reported literature values.[12]

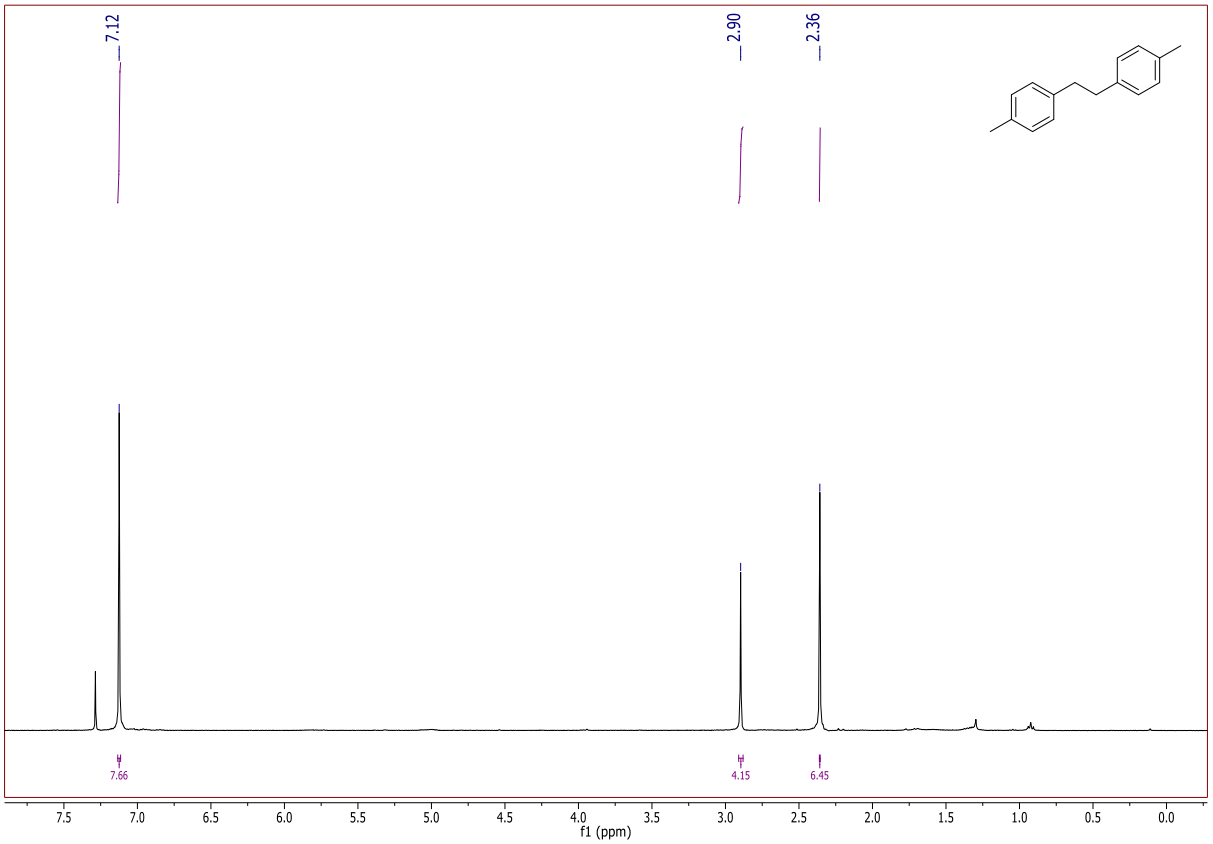
NMR Spectra:

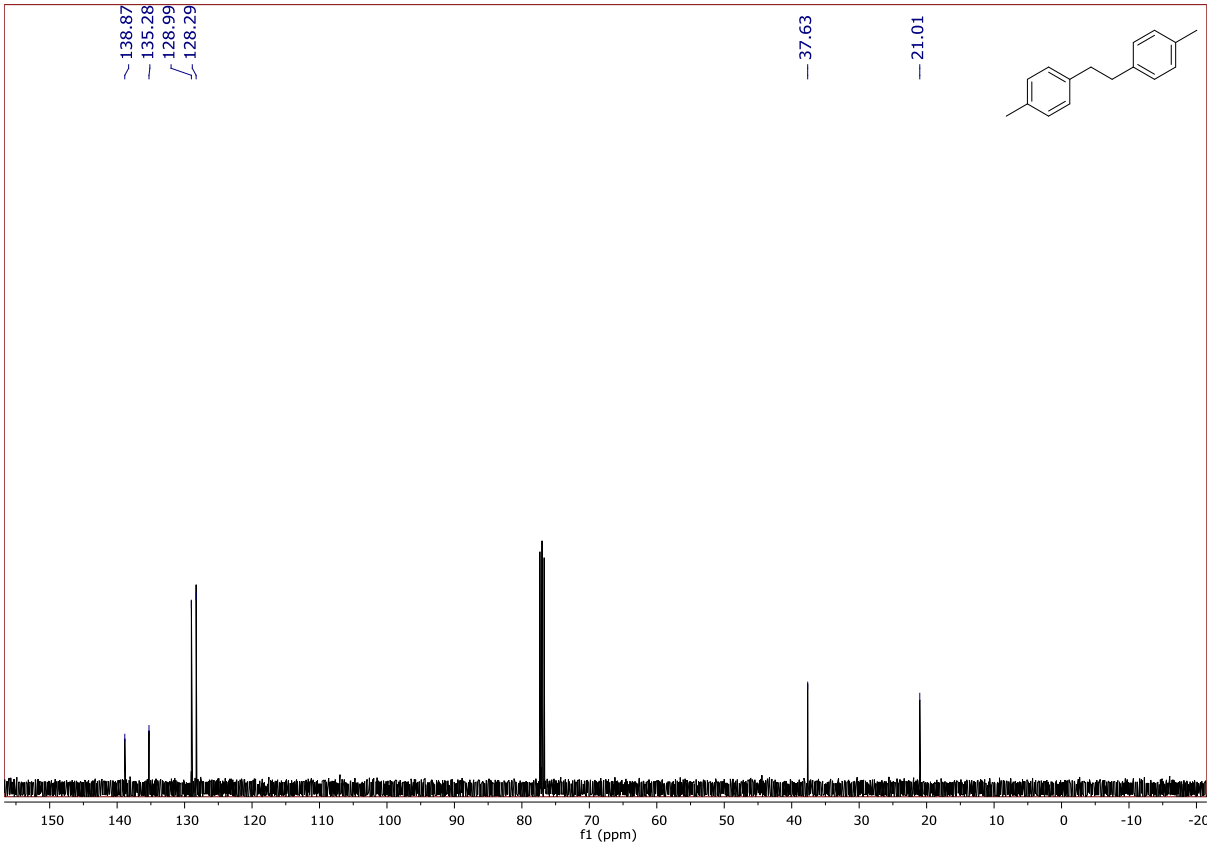
1,2-diphenylethane (2a):



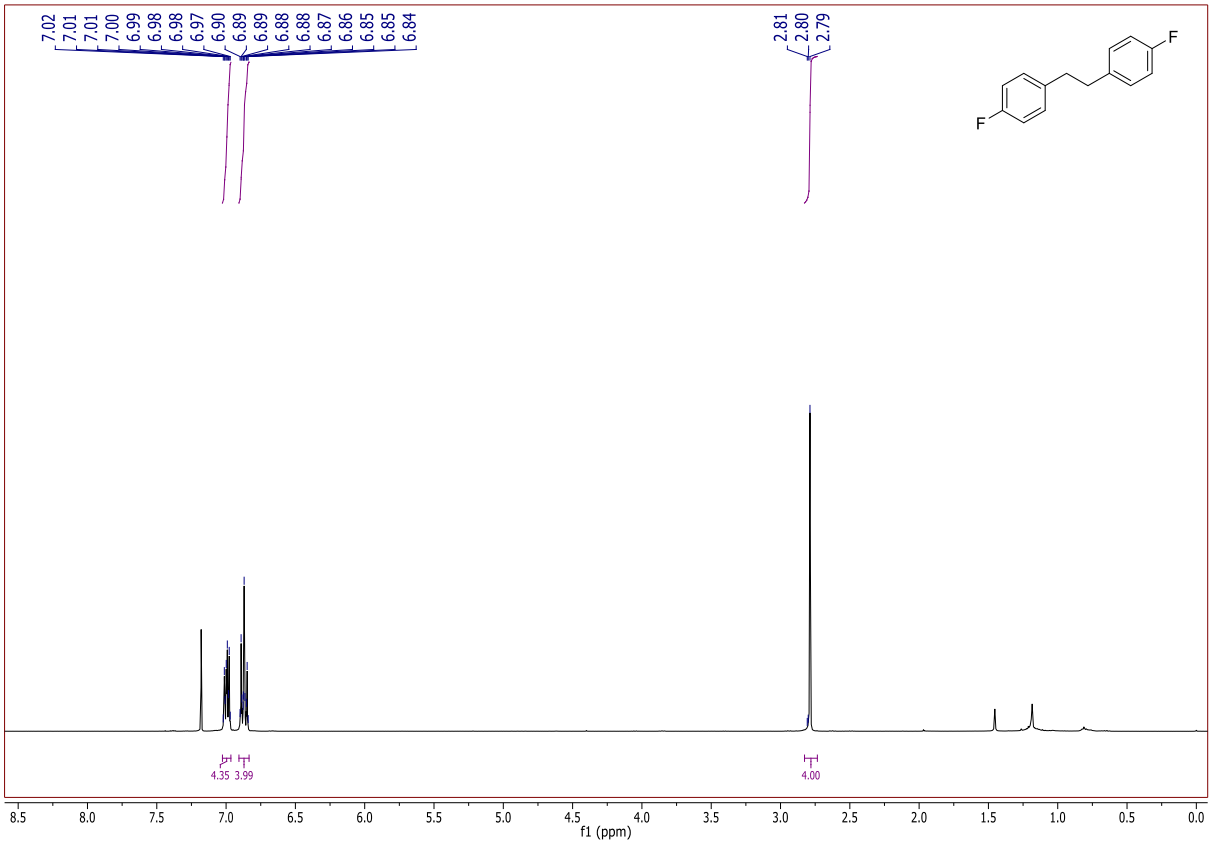


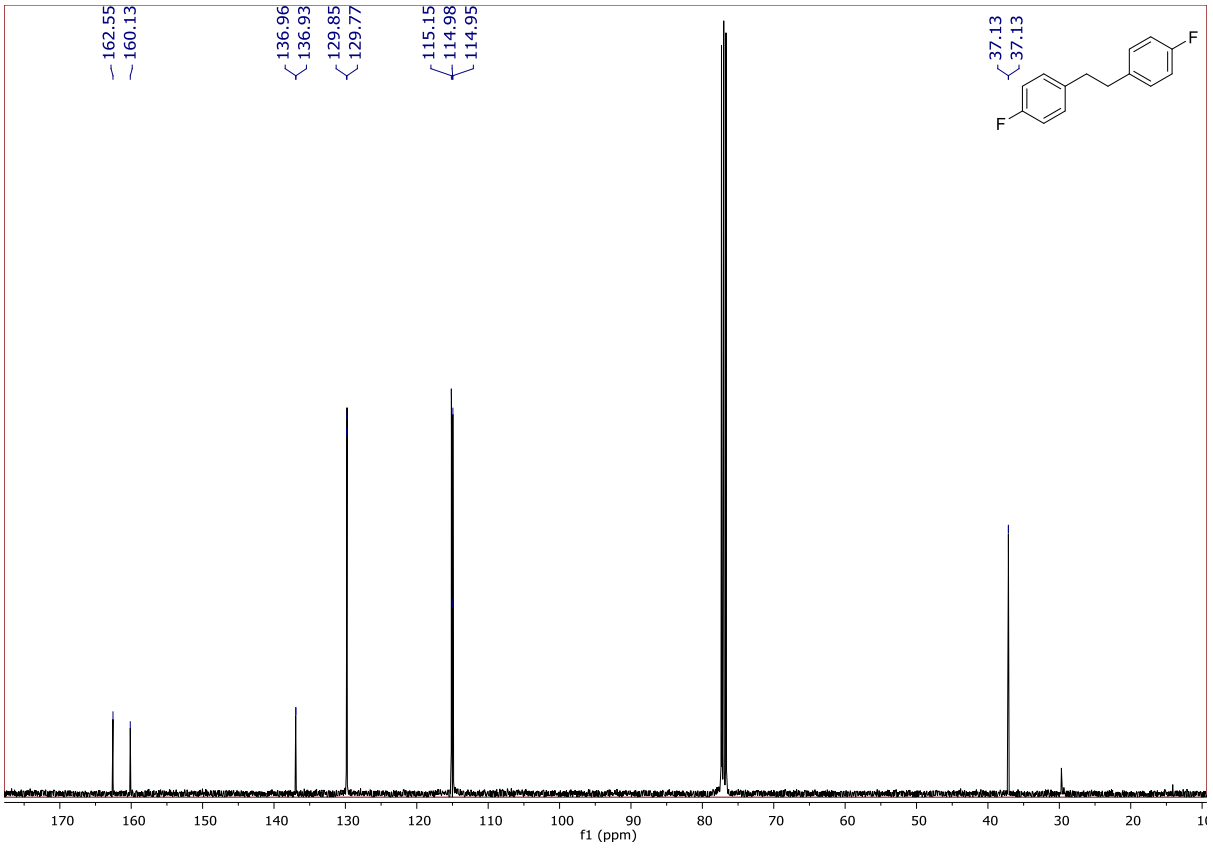
1,2-di-*p*-tolylethane (2b):



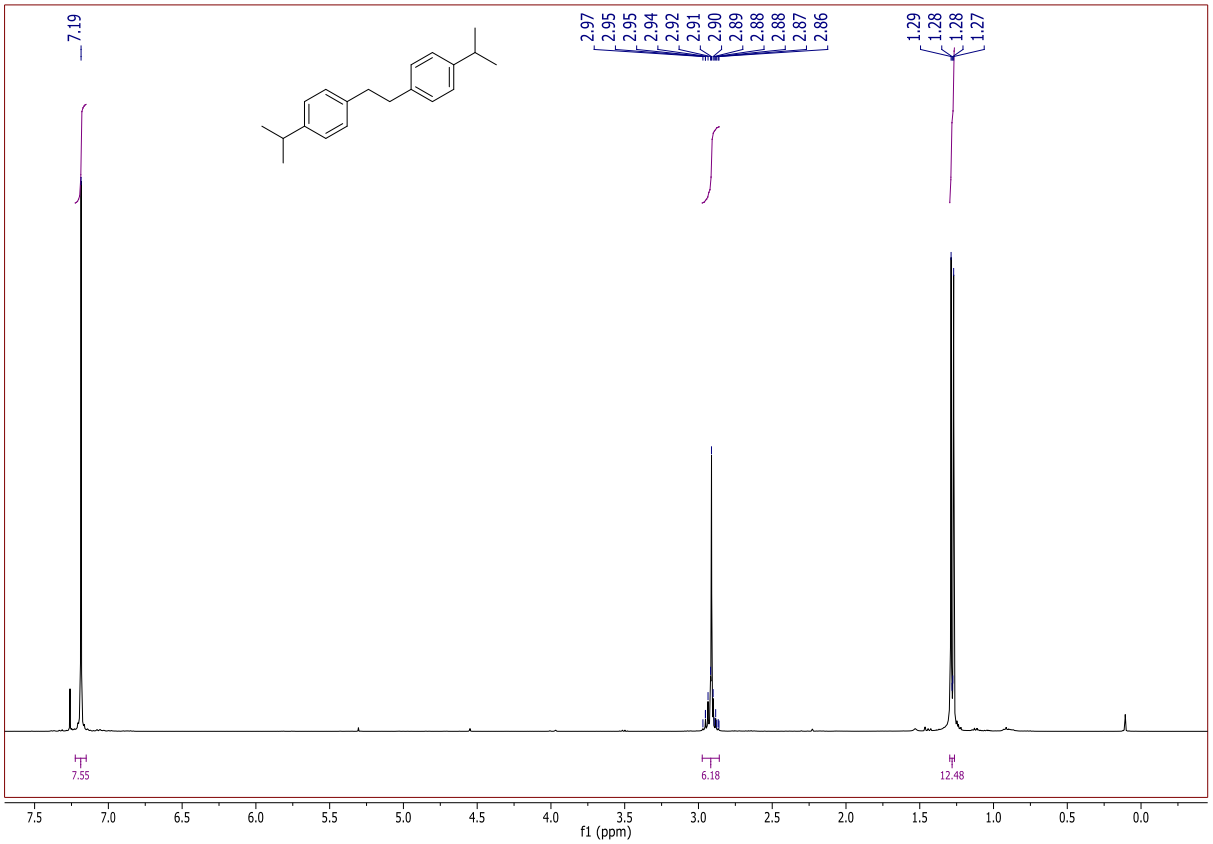


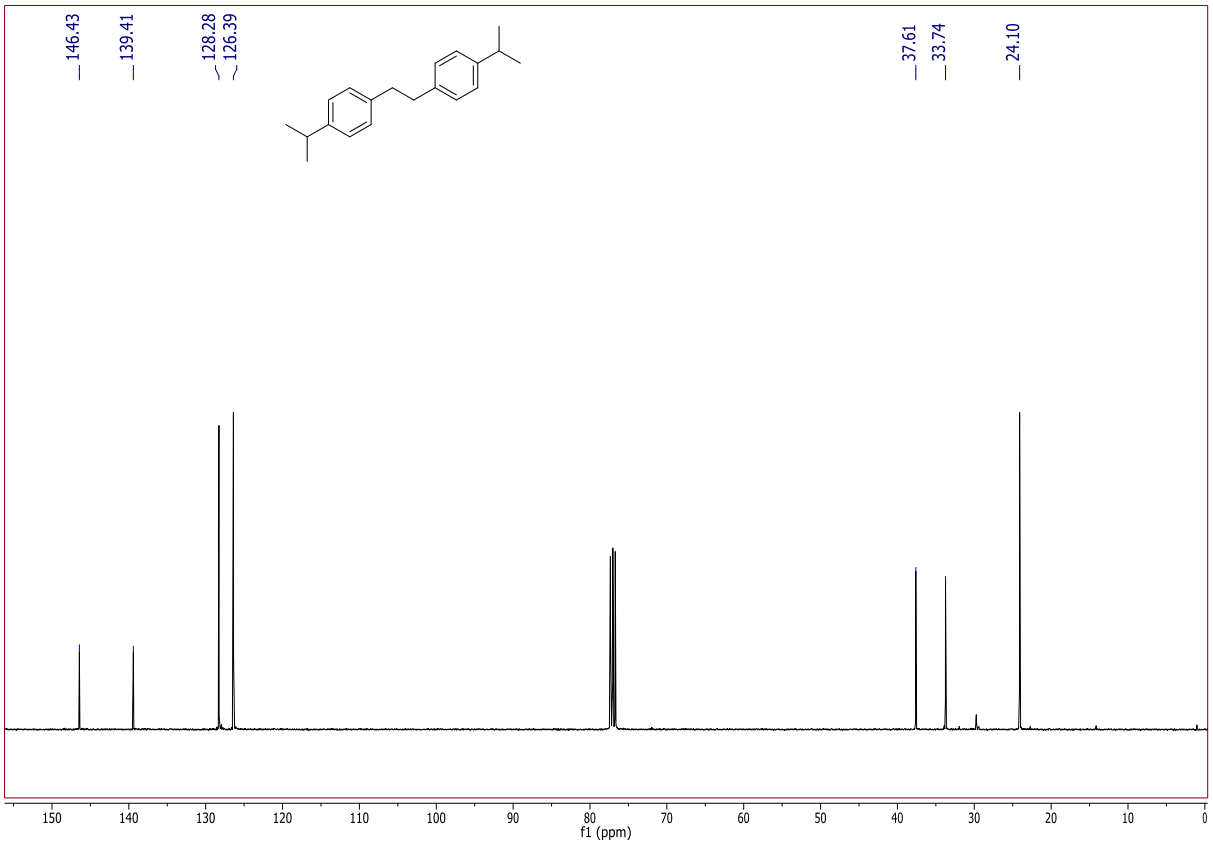
1,2-bis(4-fluorophenyl)ethane (2c):



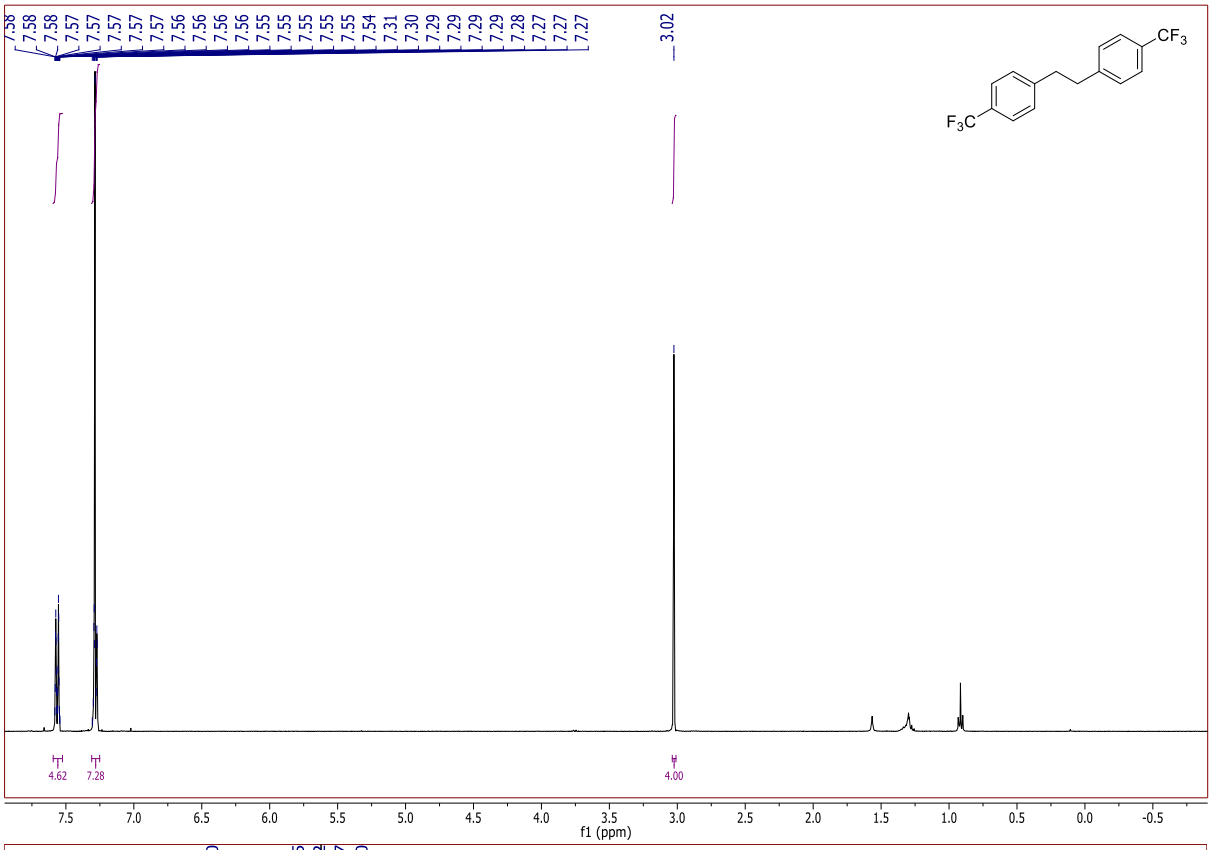


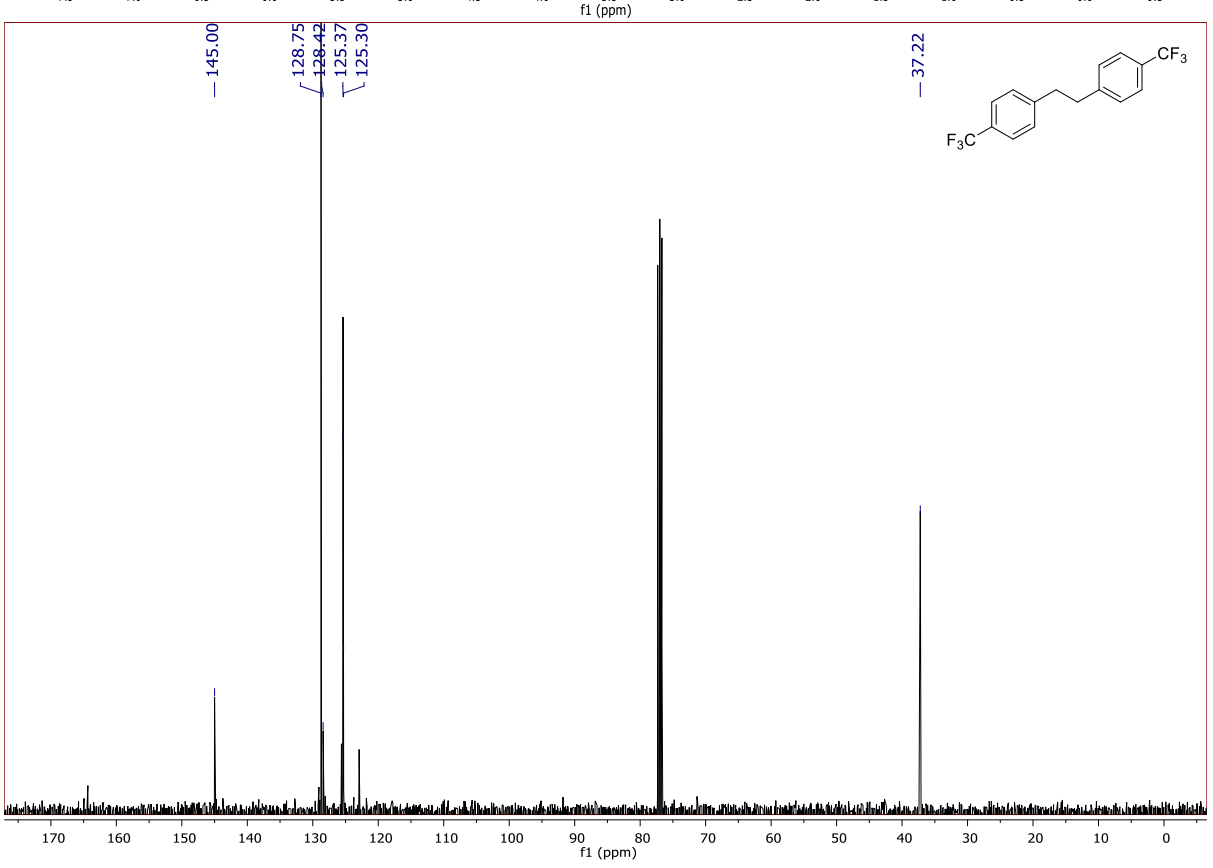
1,2-bis(4-isopropylphenyl)ethane (2d)

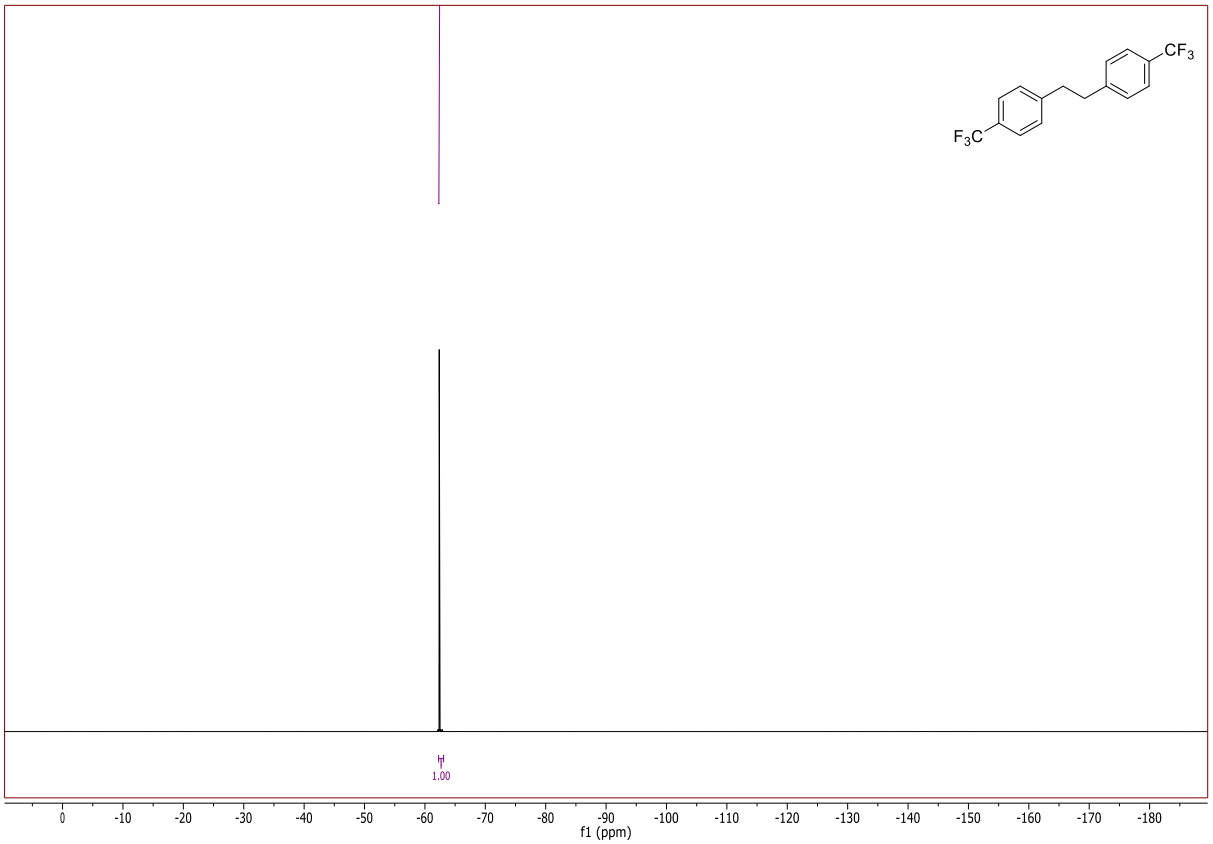




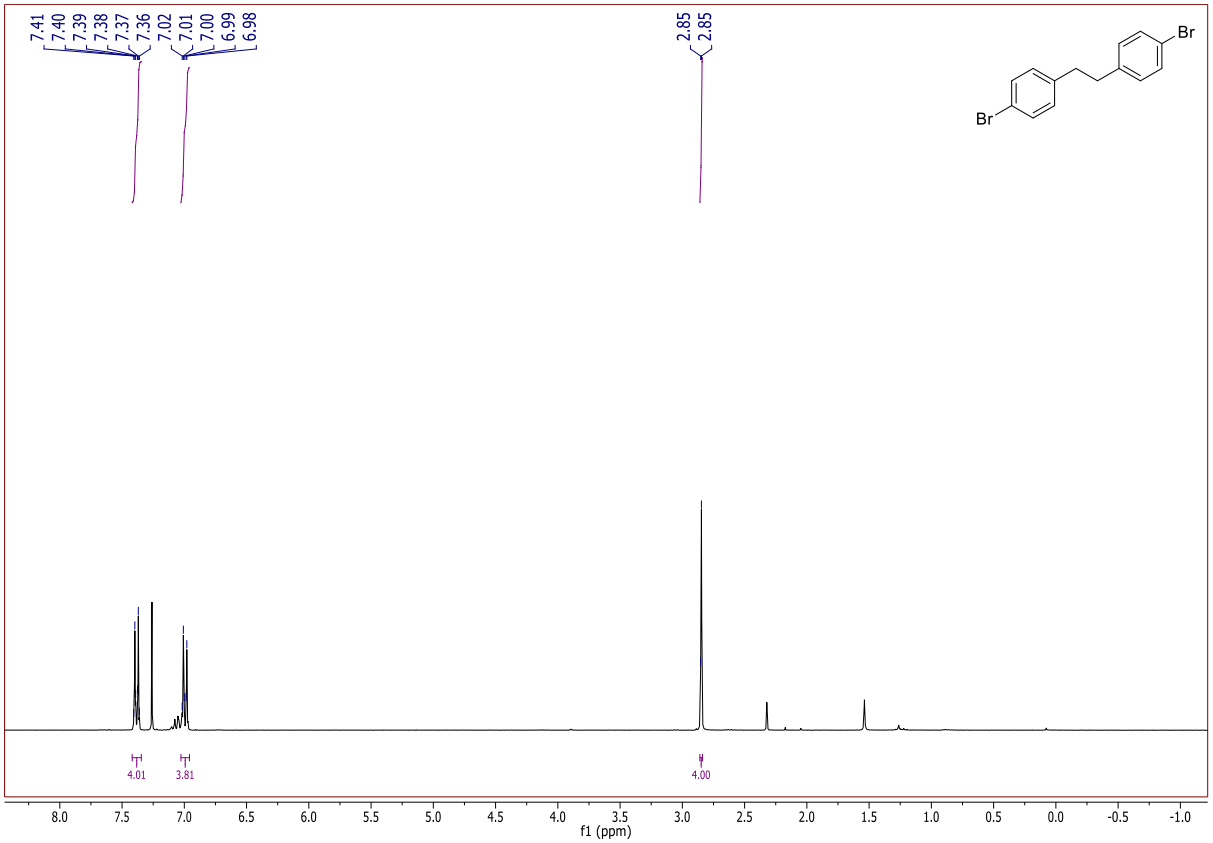
1,2-bis(4-(trifluoromethyl)phenyl)ethane (2e):

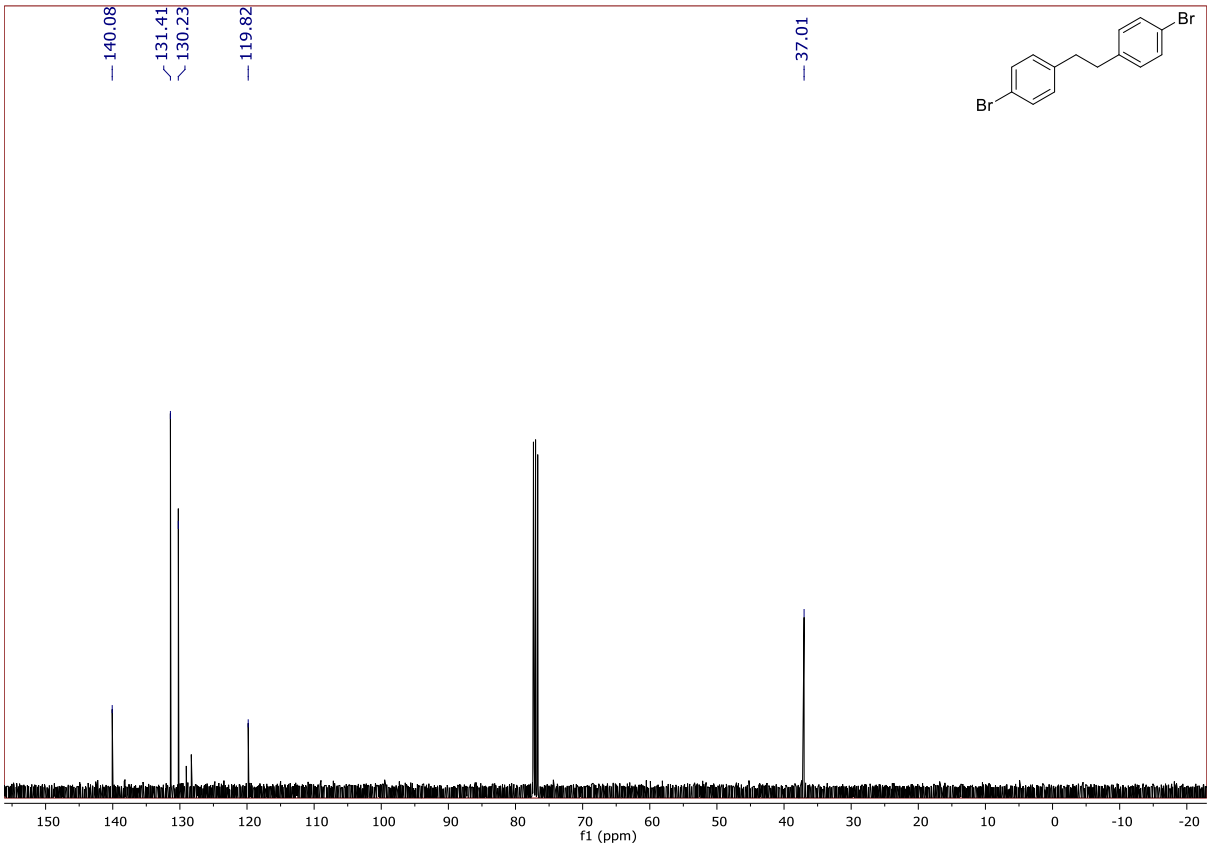




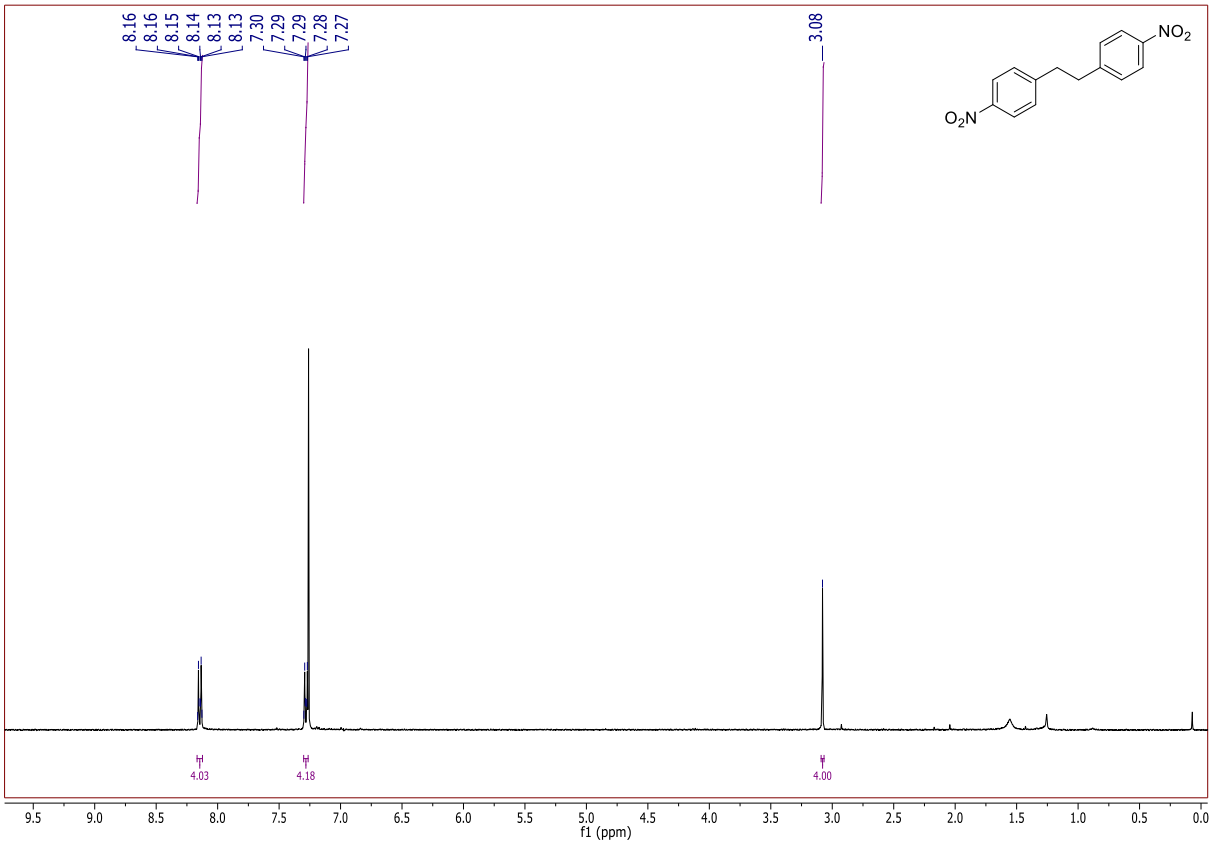

CF3
F3C
1.00
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
f1 (ppm)

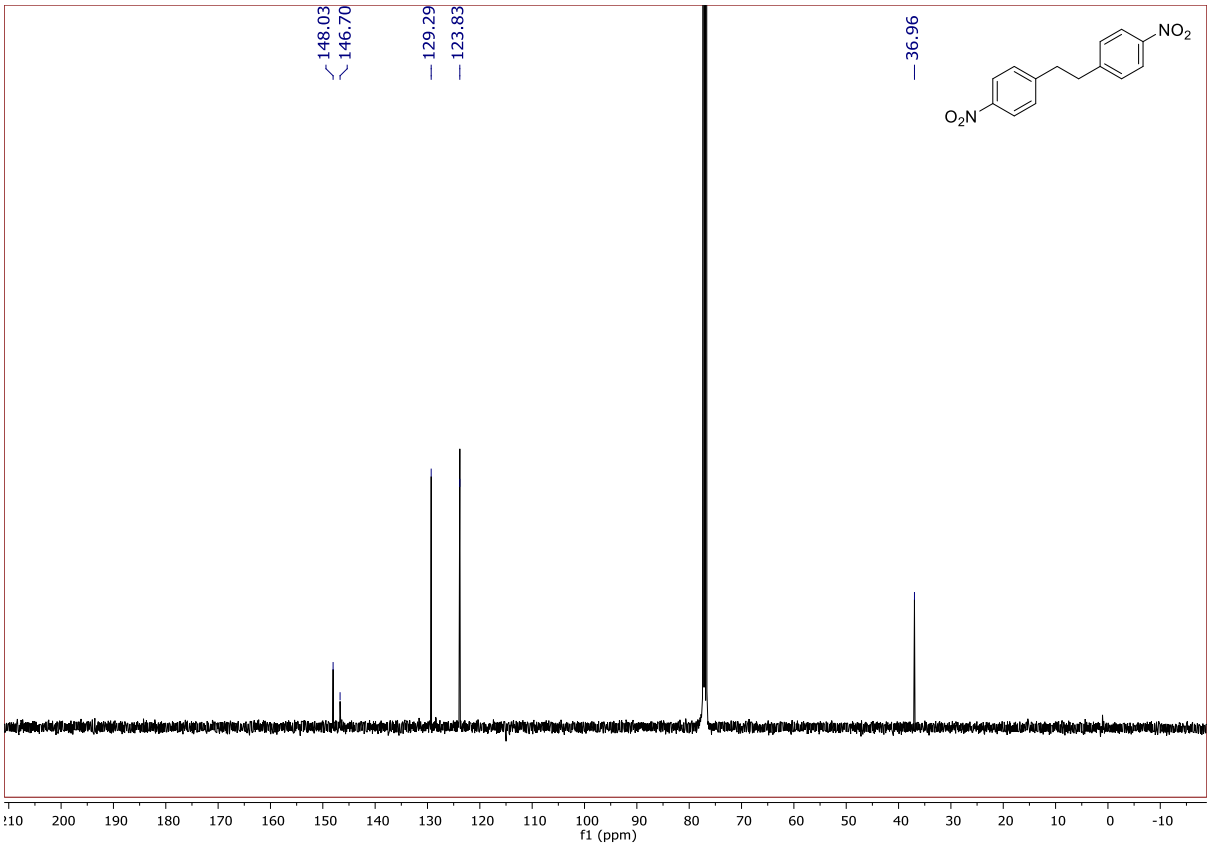
1,2-bis(4-bromophenyl)ethane (2f)



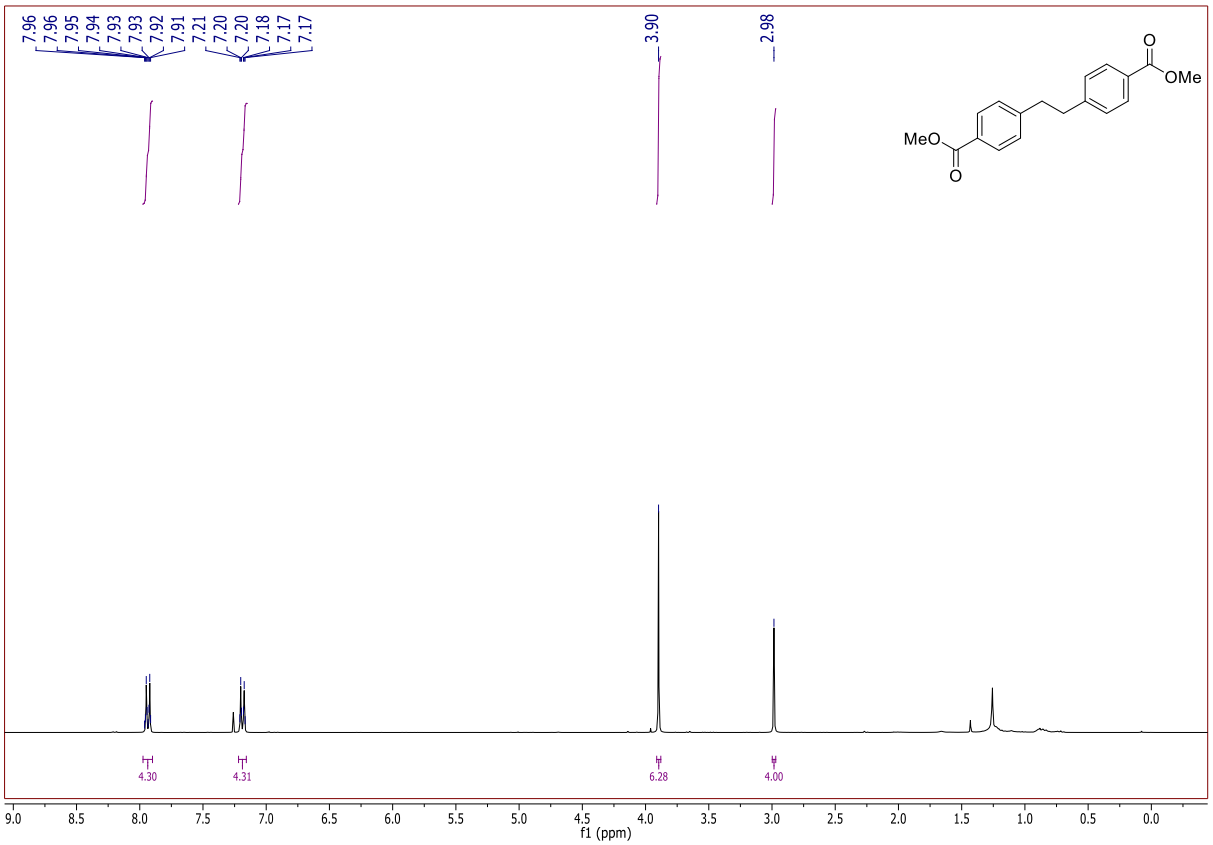


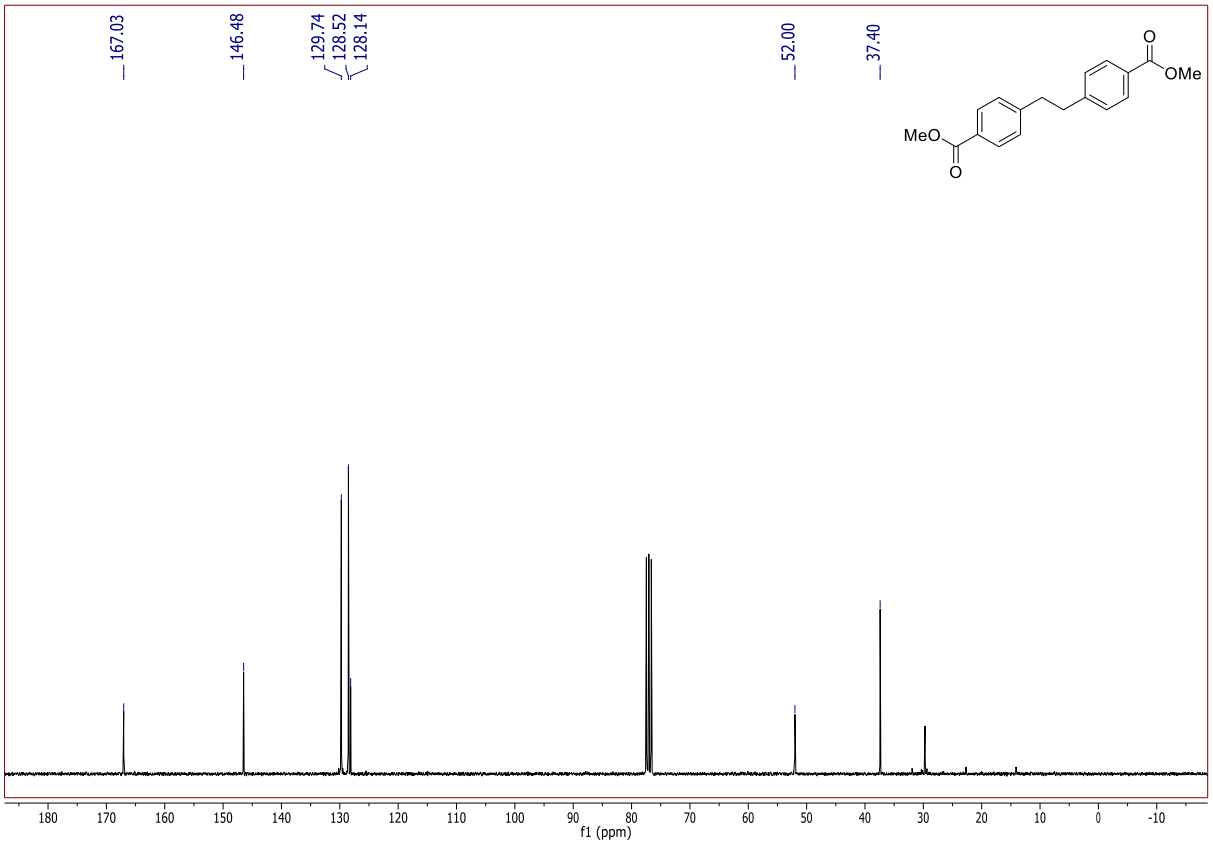
1,2-bis(4-nitrophenyl)ethane (2g)



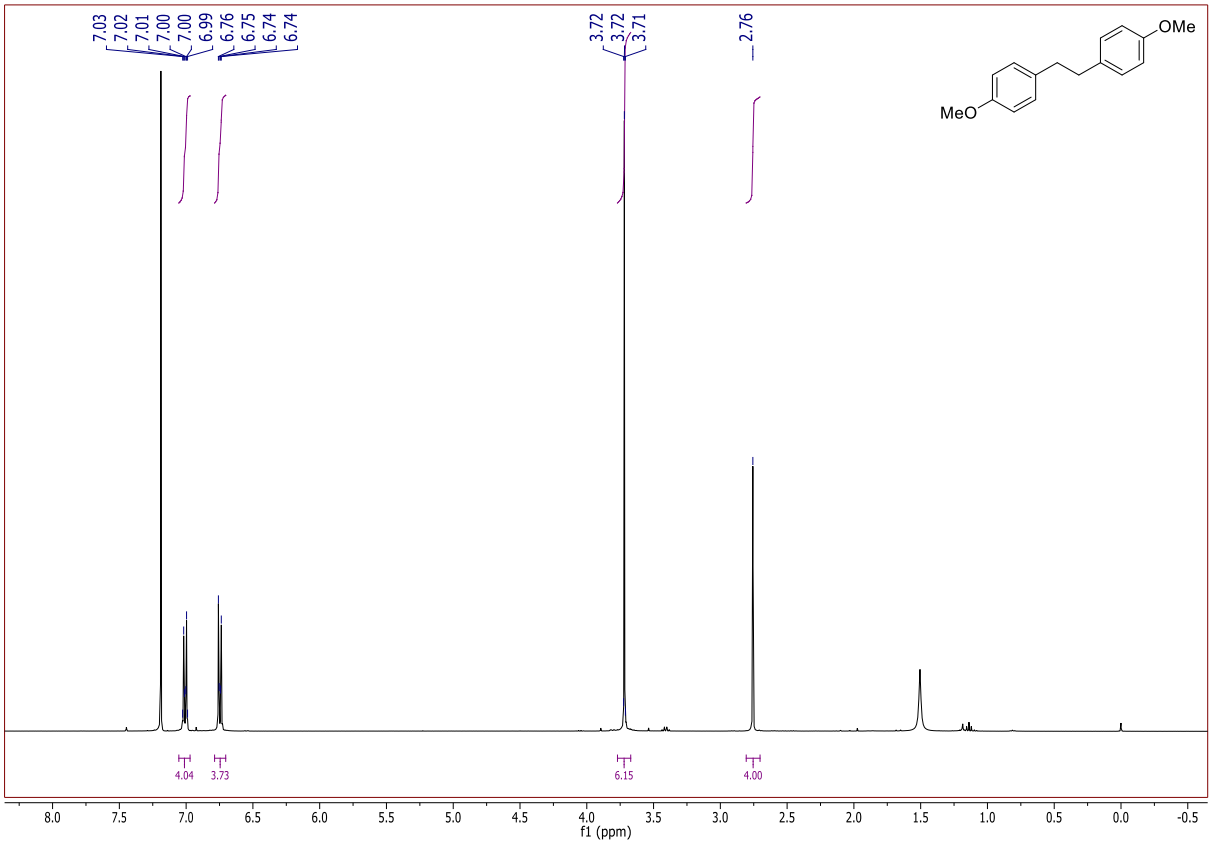


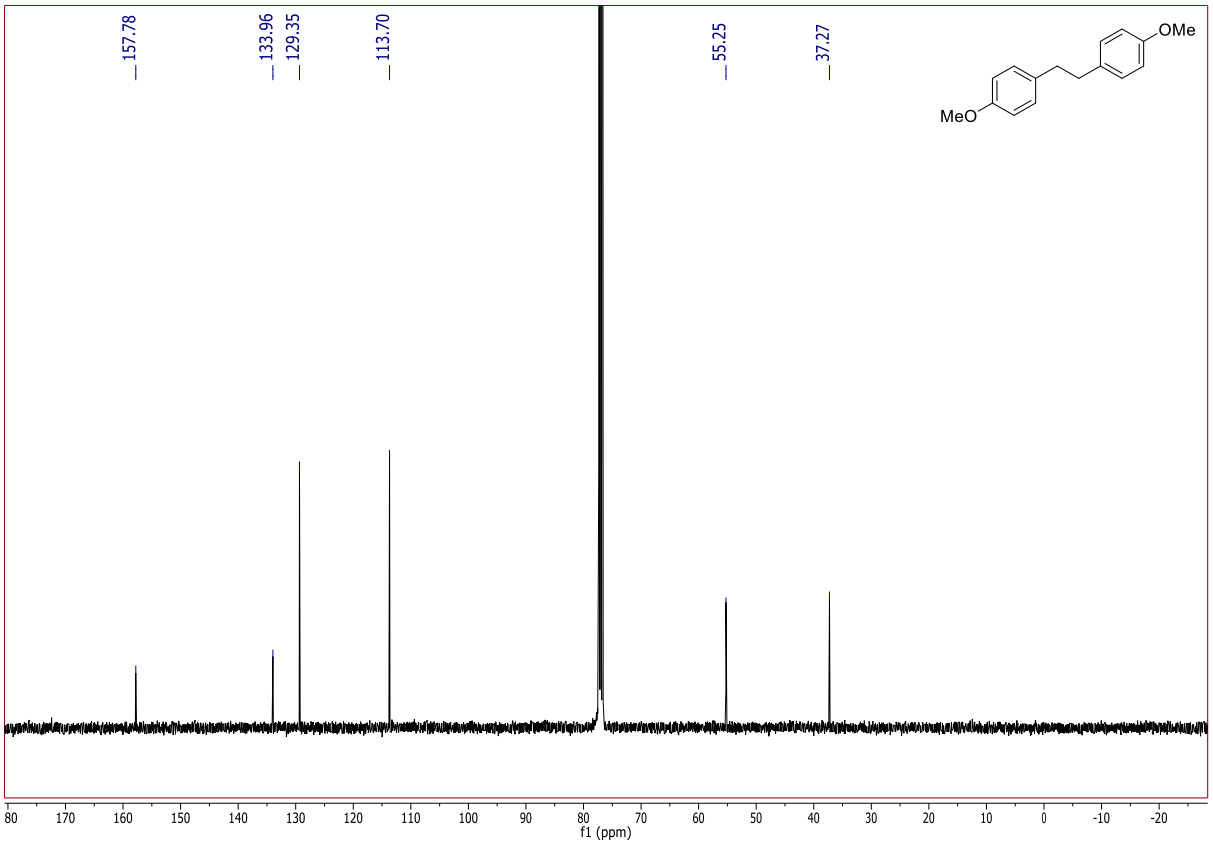
dimethyl 4,4'-(ethane-1,2-diyl)dibenzoate (2h)



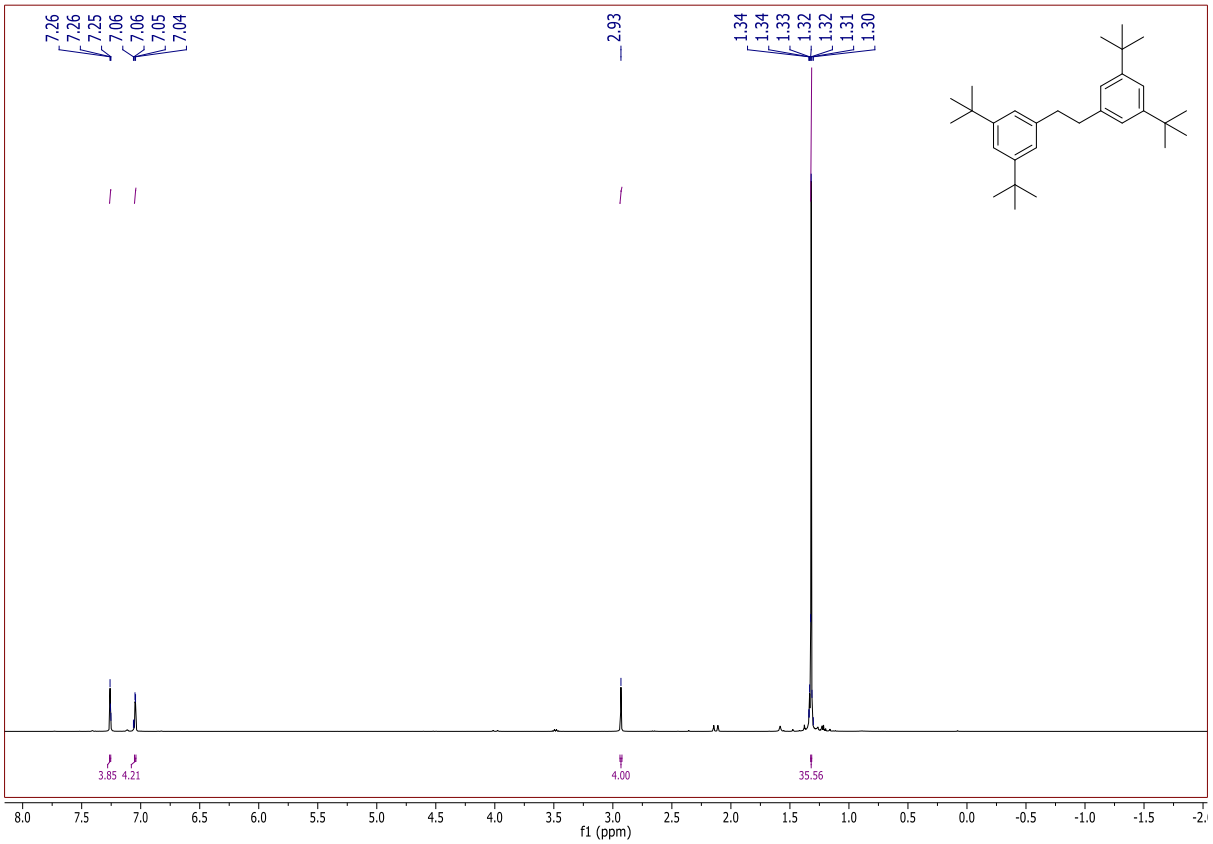


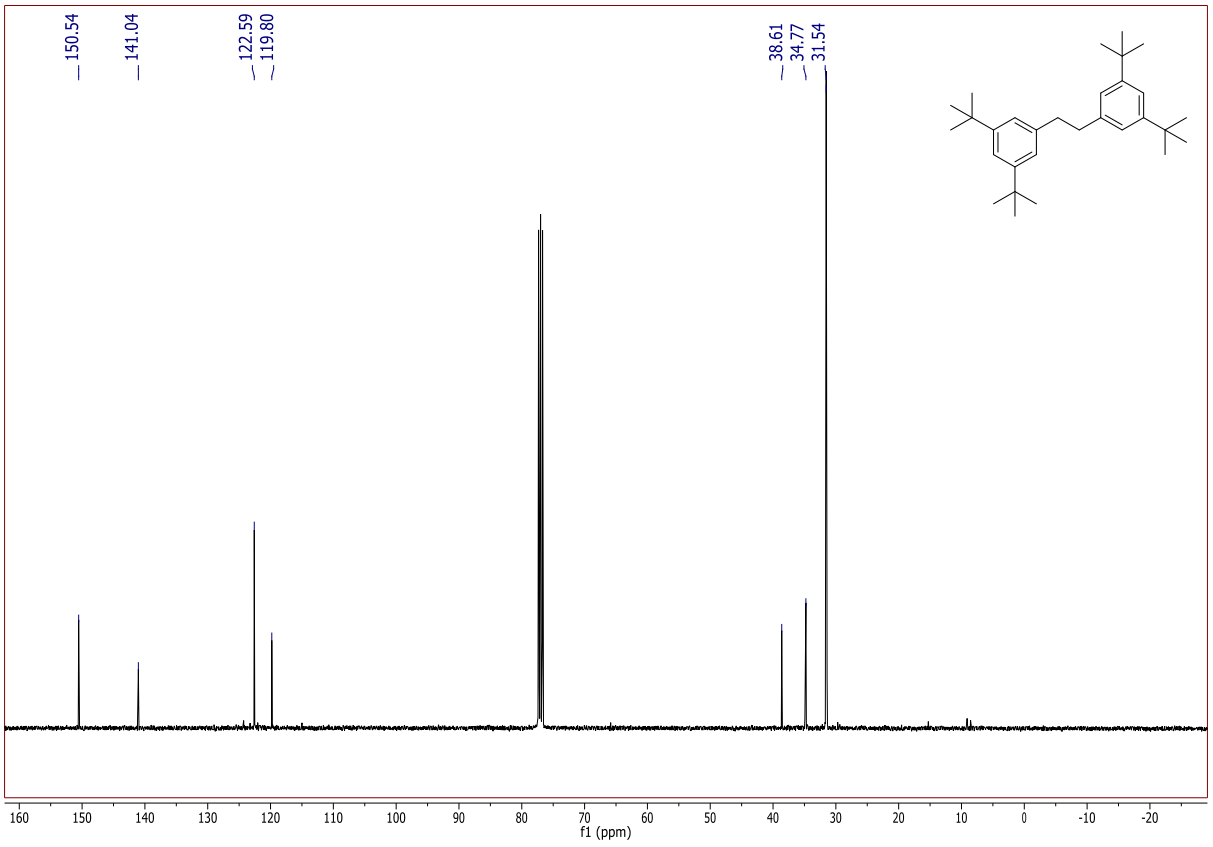
1,2-bis(4-methoxyphenyl)ethane (2i)



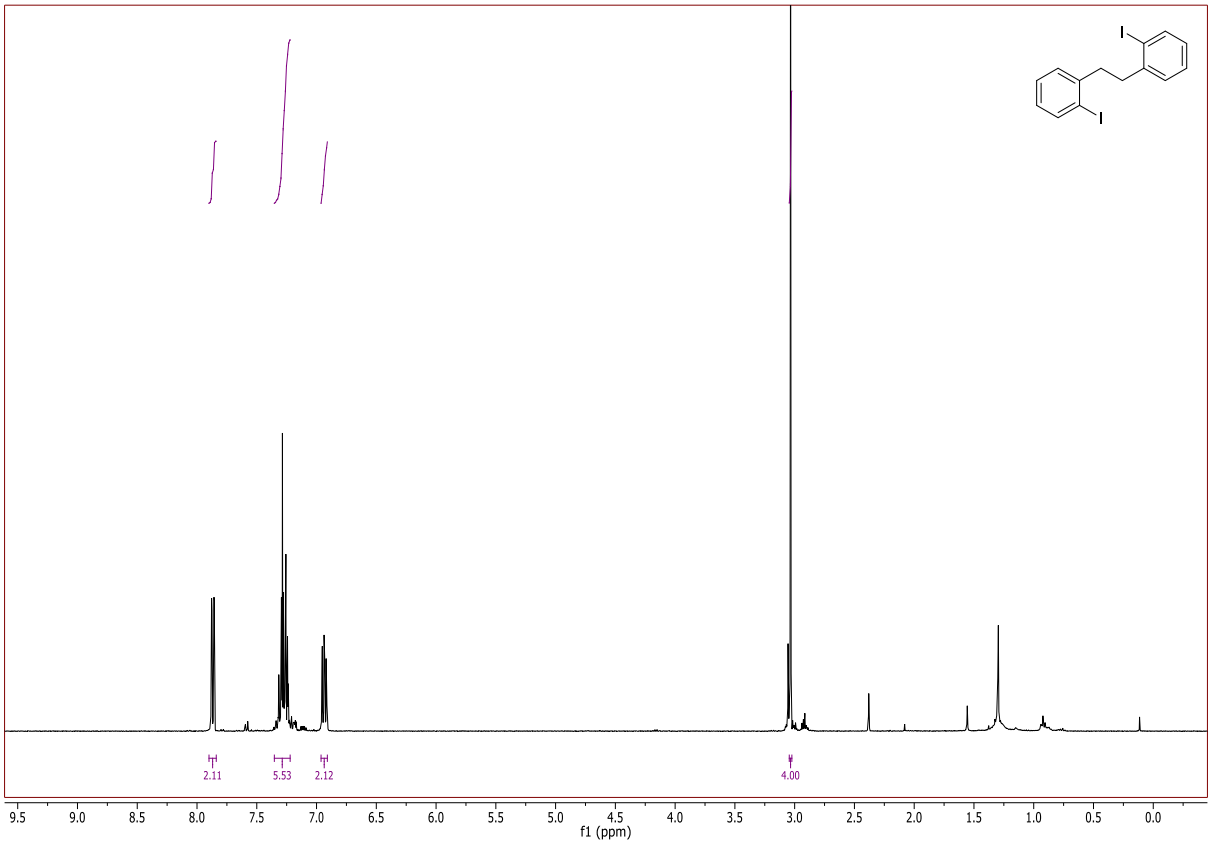


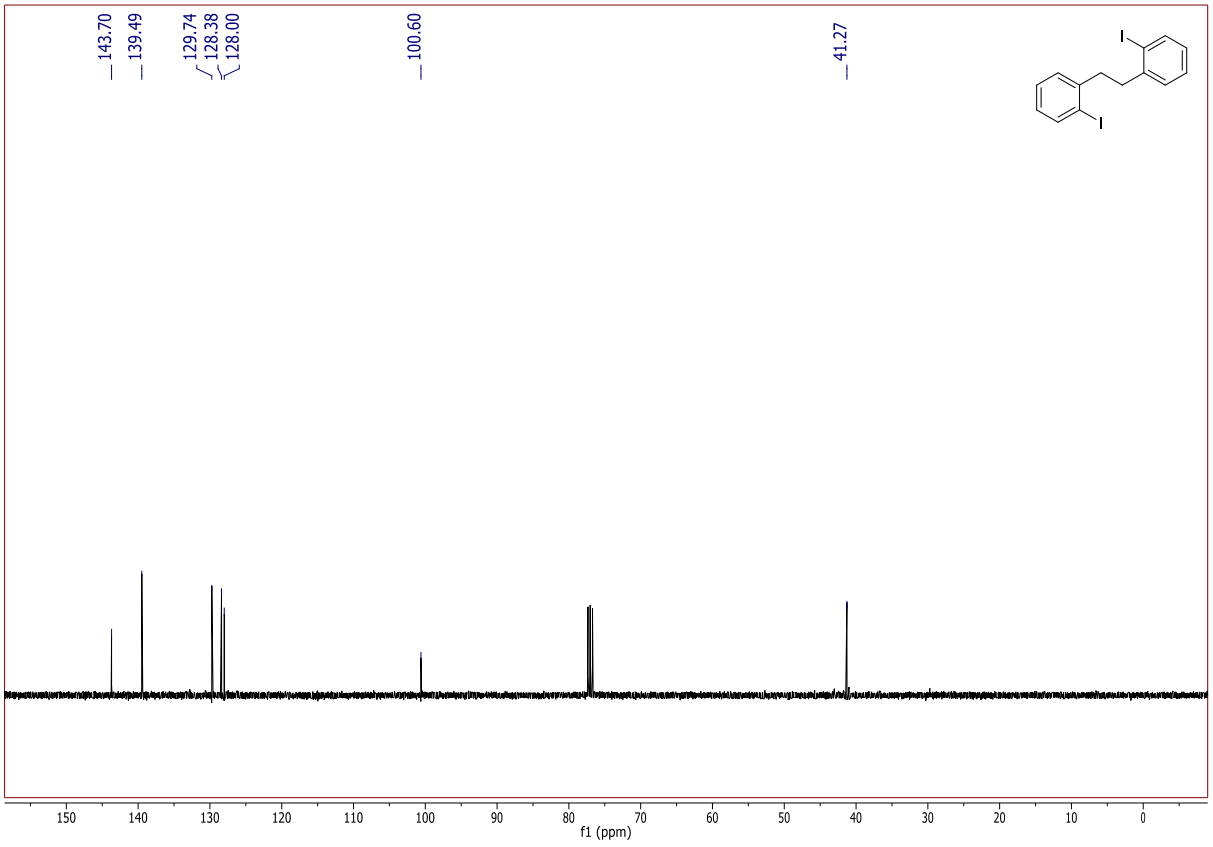
1,2-bis(3,5-di-tert-butylphenyl)ethane (2j)



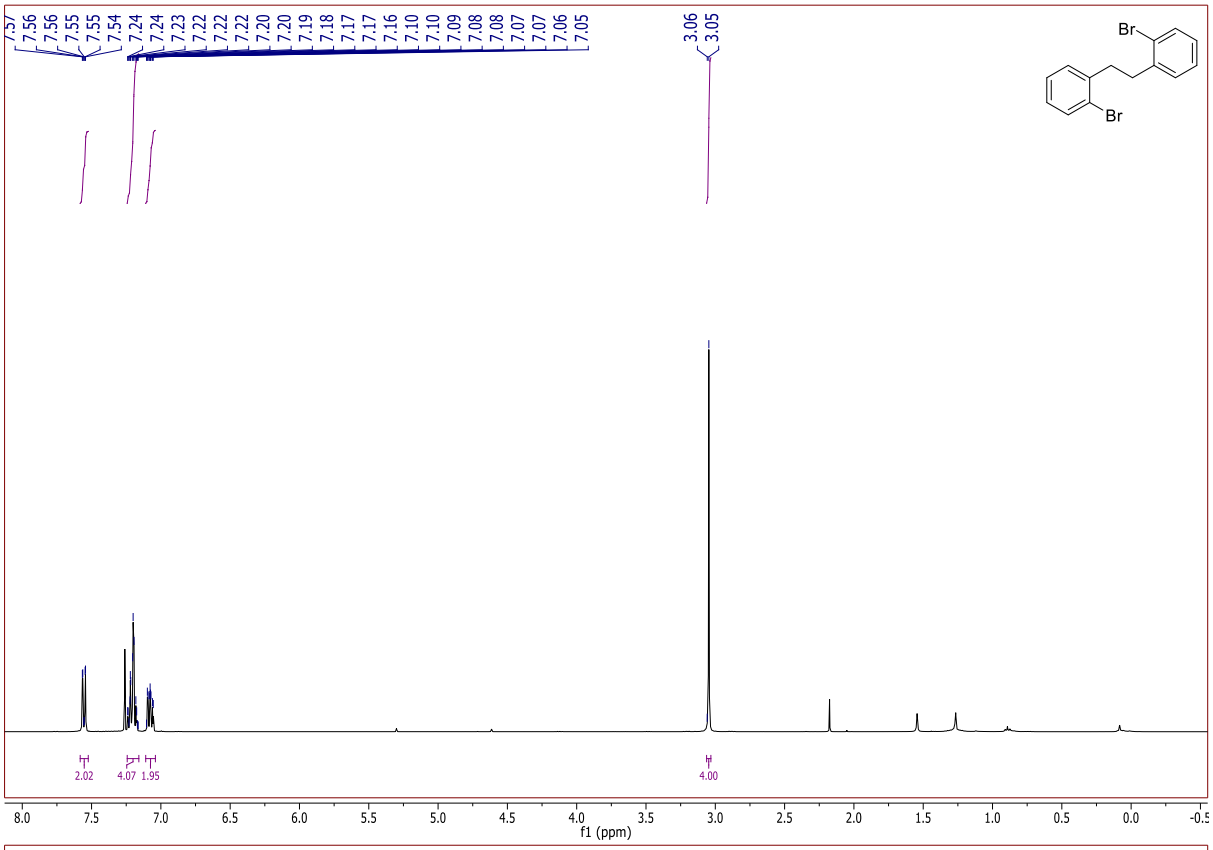


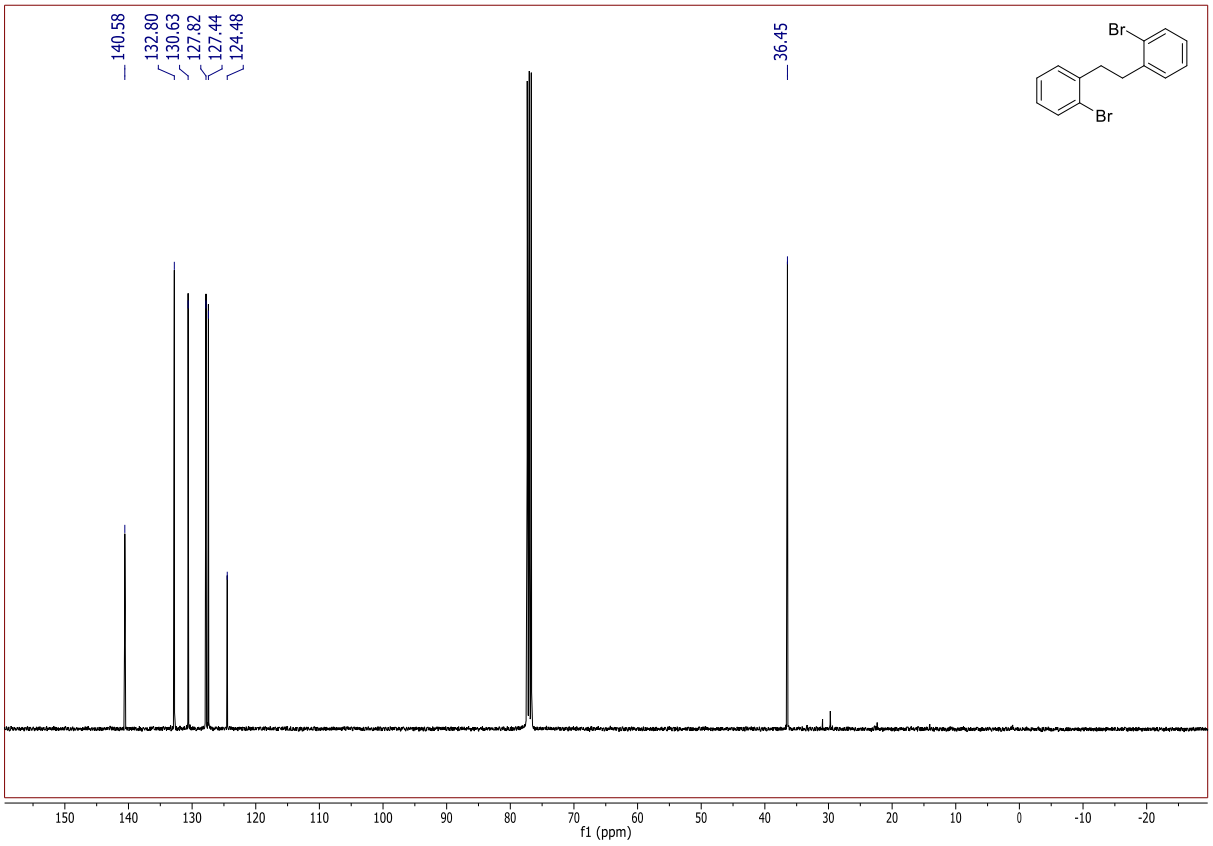
1,2-bis(2-iodophenyl)ethane (2k)



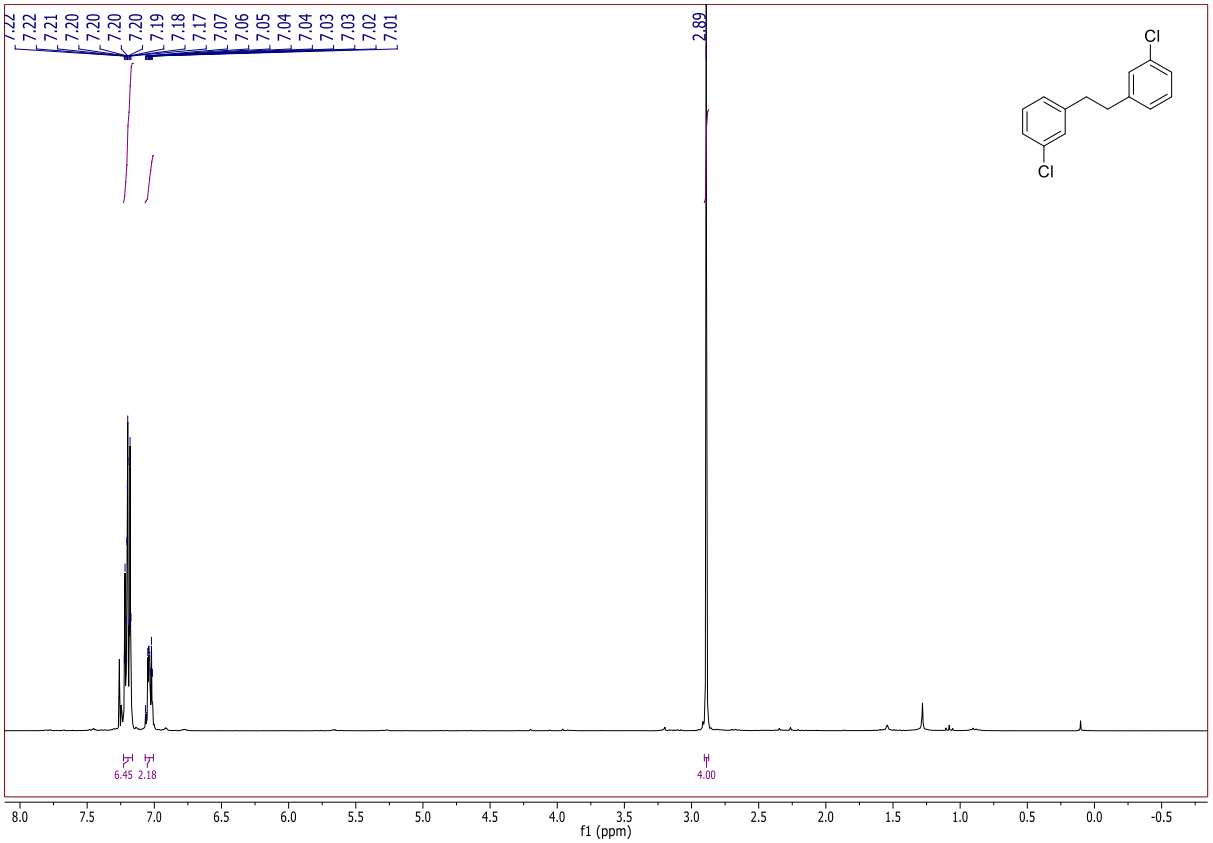


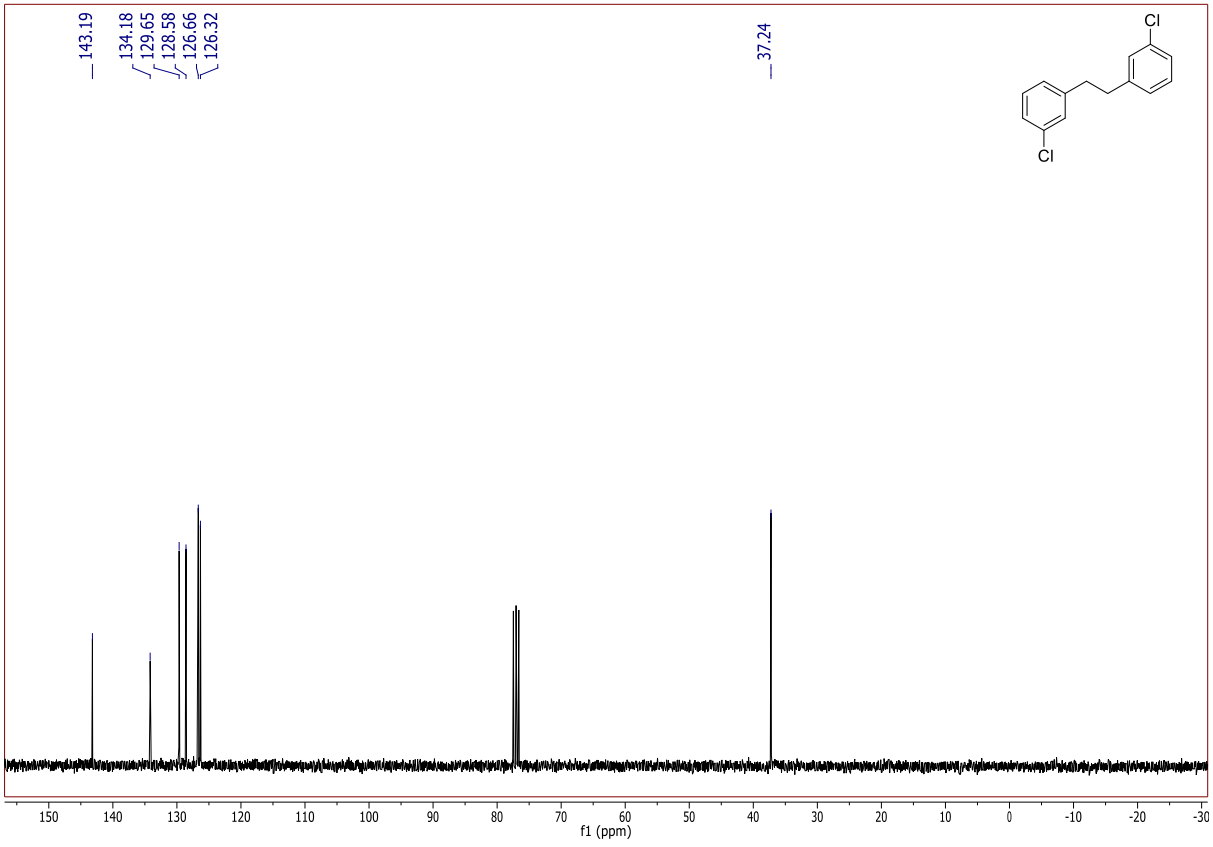
1,2-bis(2-bromophenyl)ethane (2l)



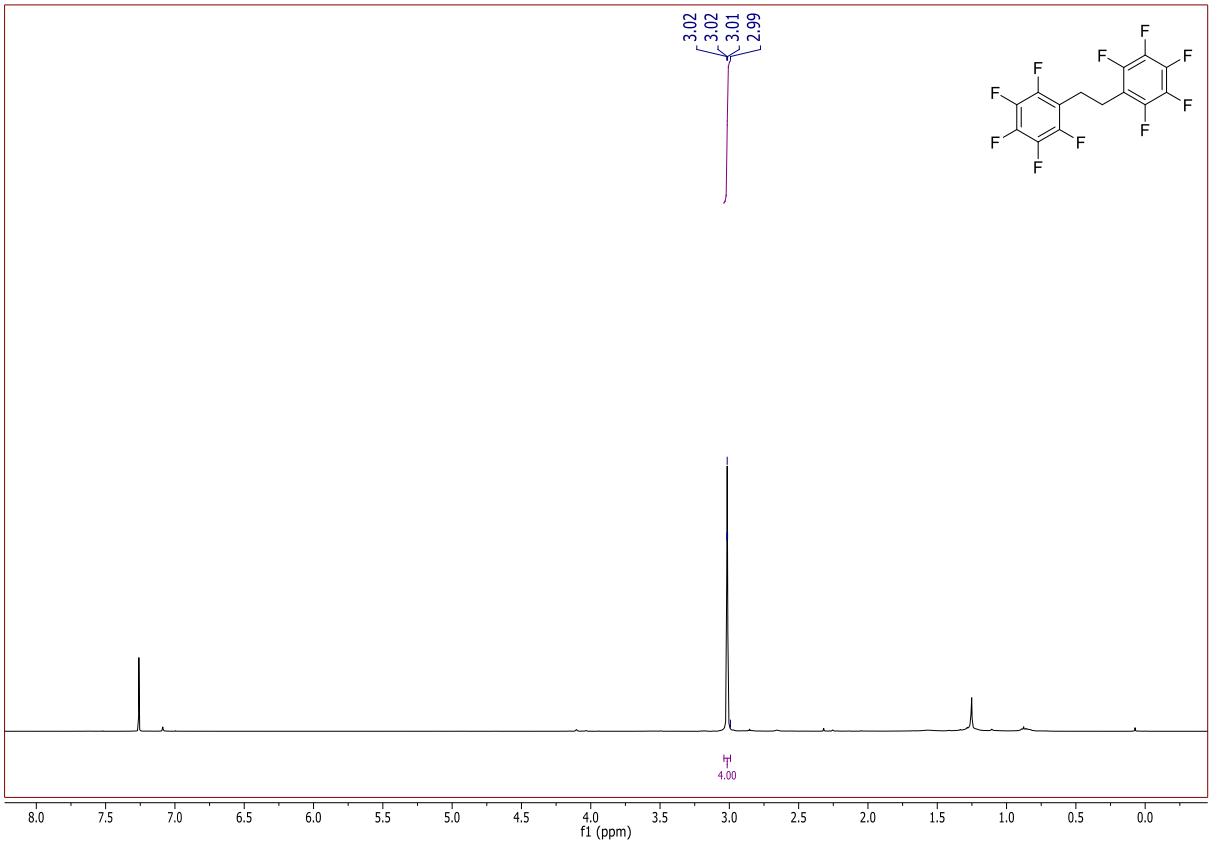


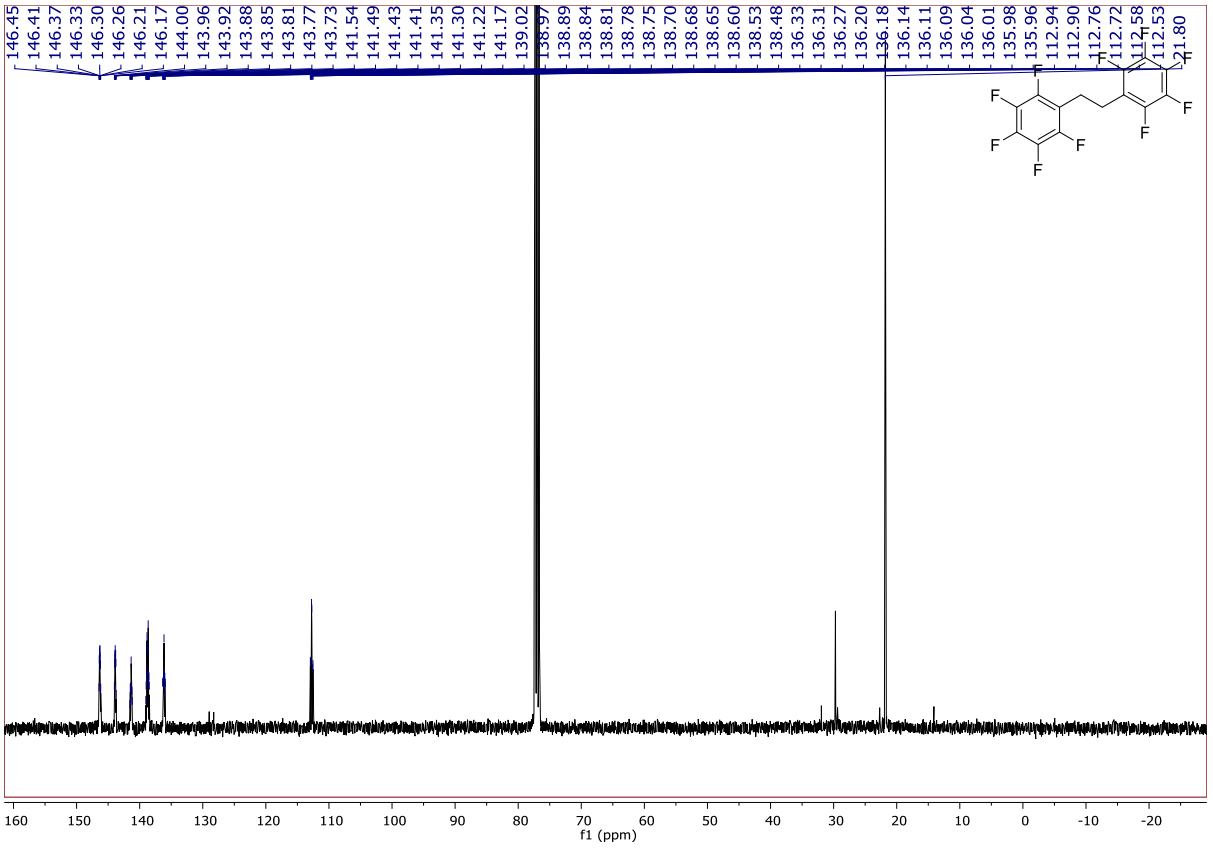
1,2-bis(3-chlorophenyl)ethane (2m)

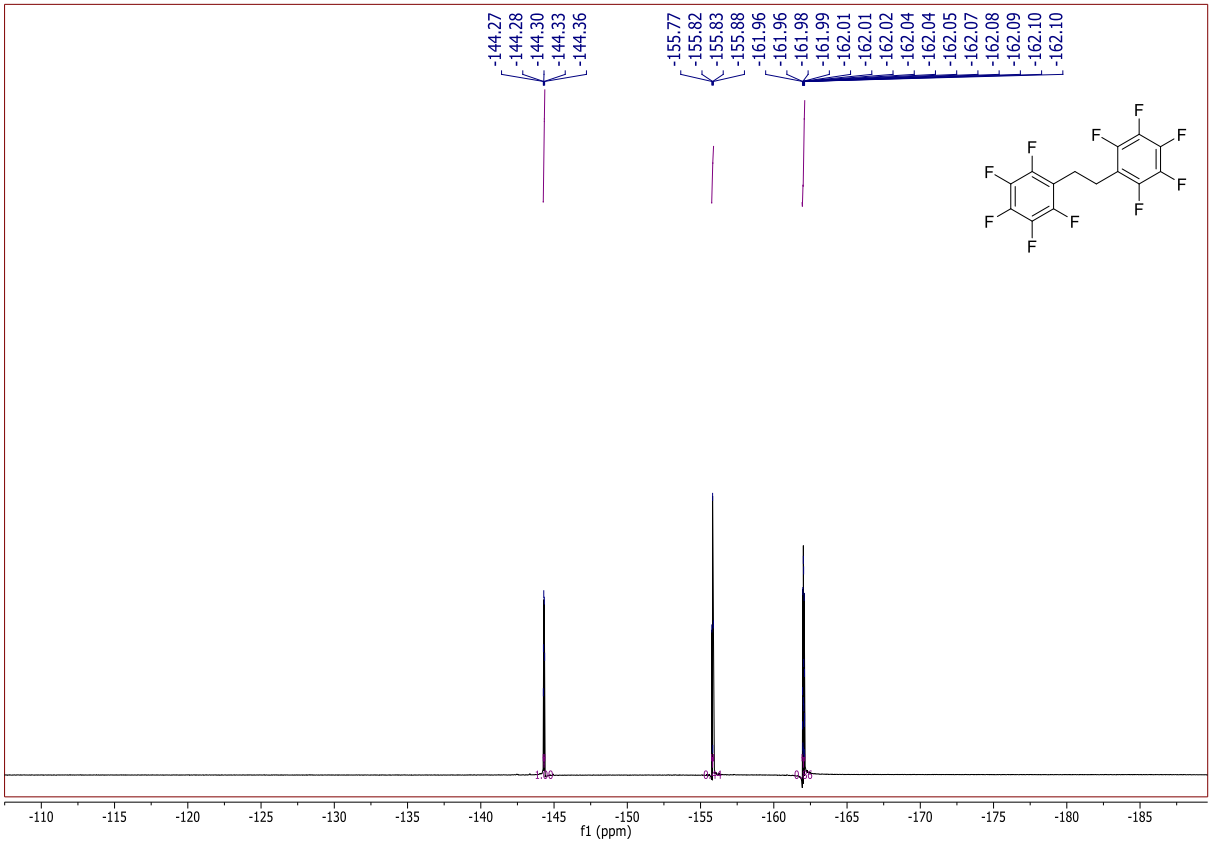




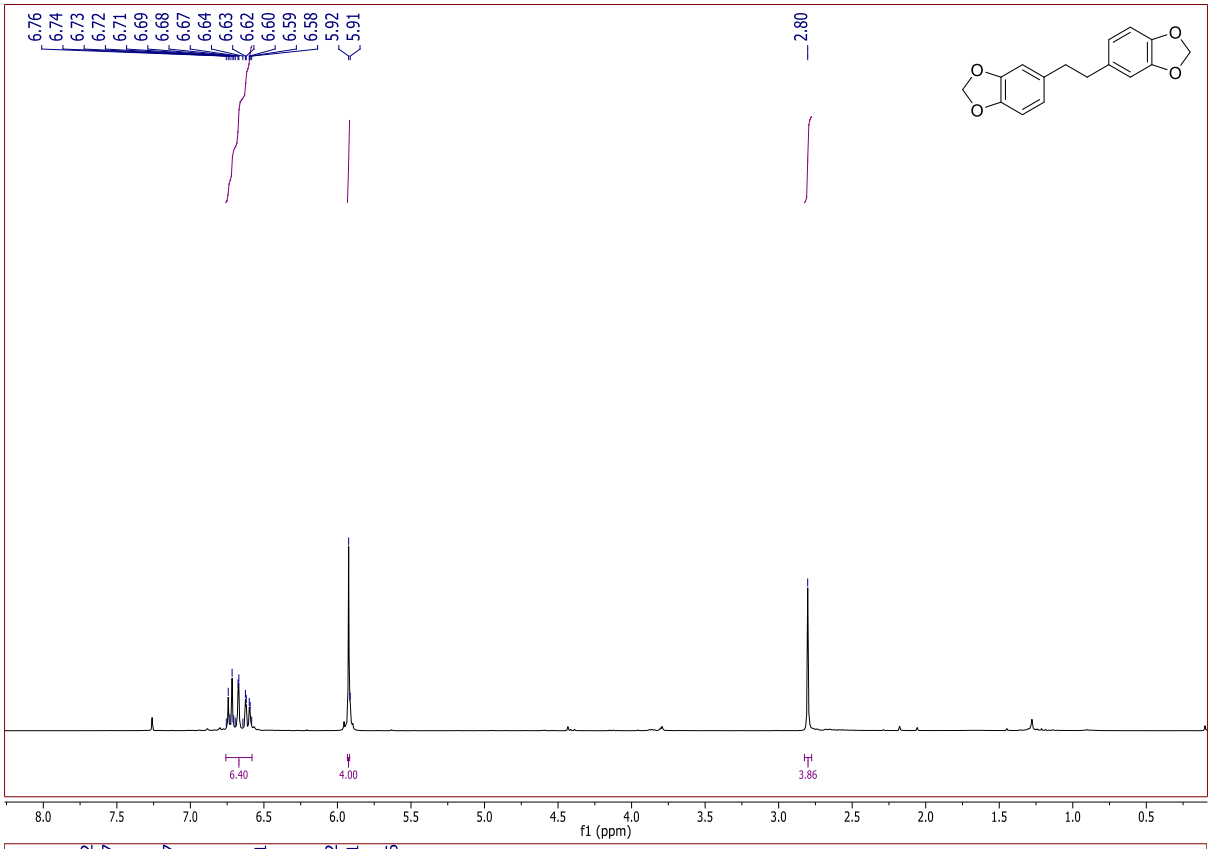
1,2-bis(perfluorophenyl)ethane (2n)

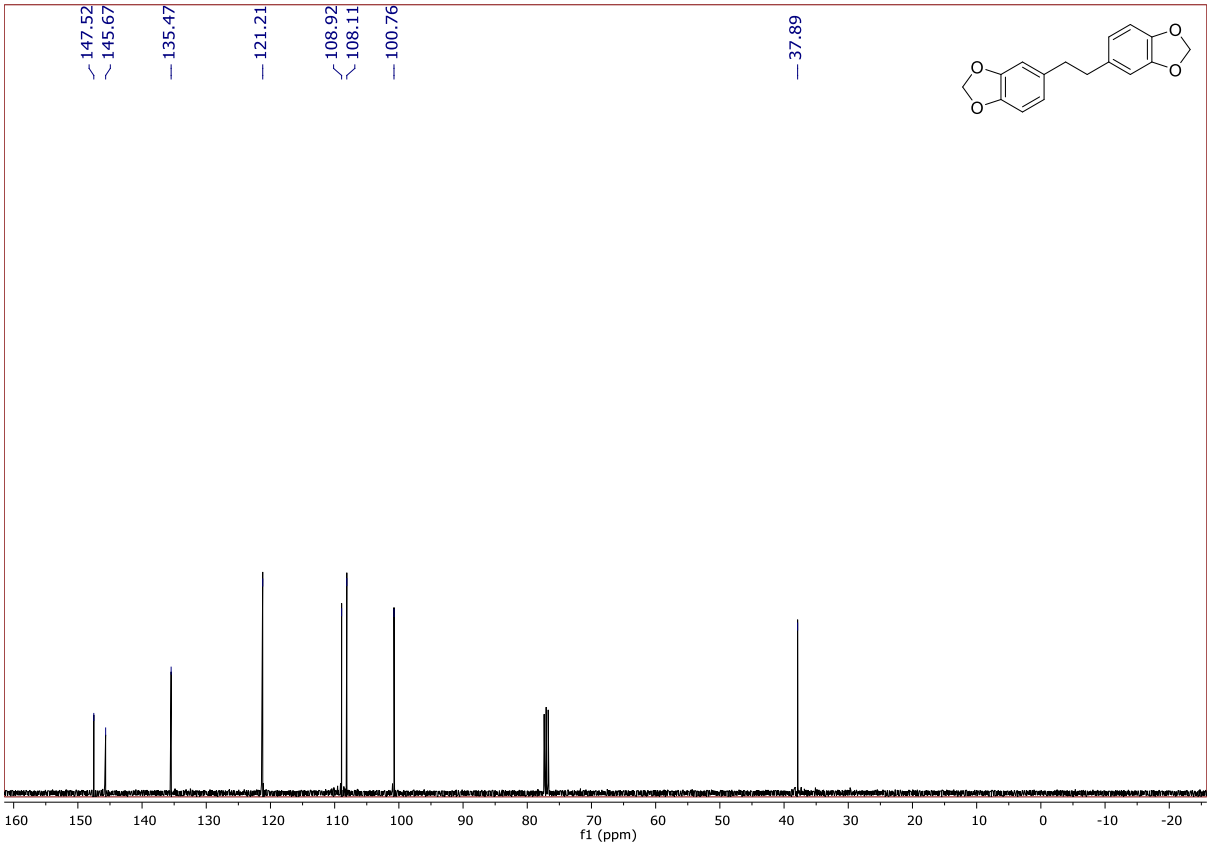




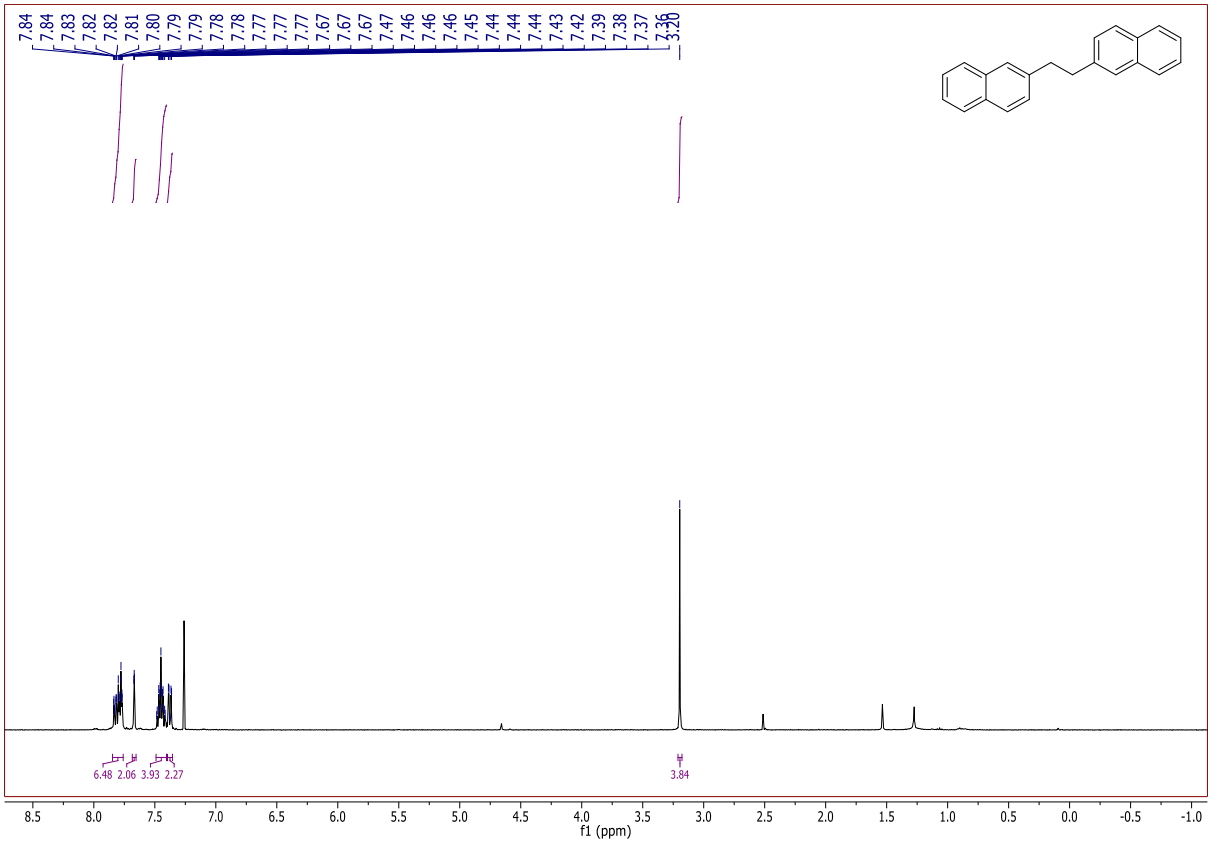

-144.27
-144.28
-144.30
-144.33
-144.36
-155.77
-155.82
-155.83
-155.88
-161.96
-161.96
-161.98
-161.99
-162.01
-162.01
-162.02
-162.04
-162.04
-162.05
-162.07
-162.08
-162.09
-162.10
-162.10
F
F
F
F
F
F
F
F
F
F
1.00
0.94
0.98
-110
-115
-120
-125
-130
-135
-140
-145
-150
-155
-160
-165
-170
-175
-180
-185
f1 (ppm)

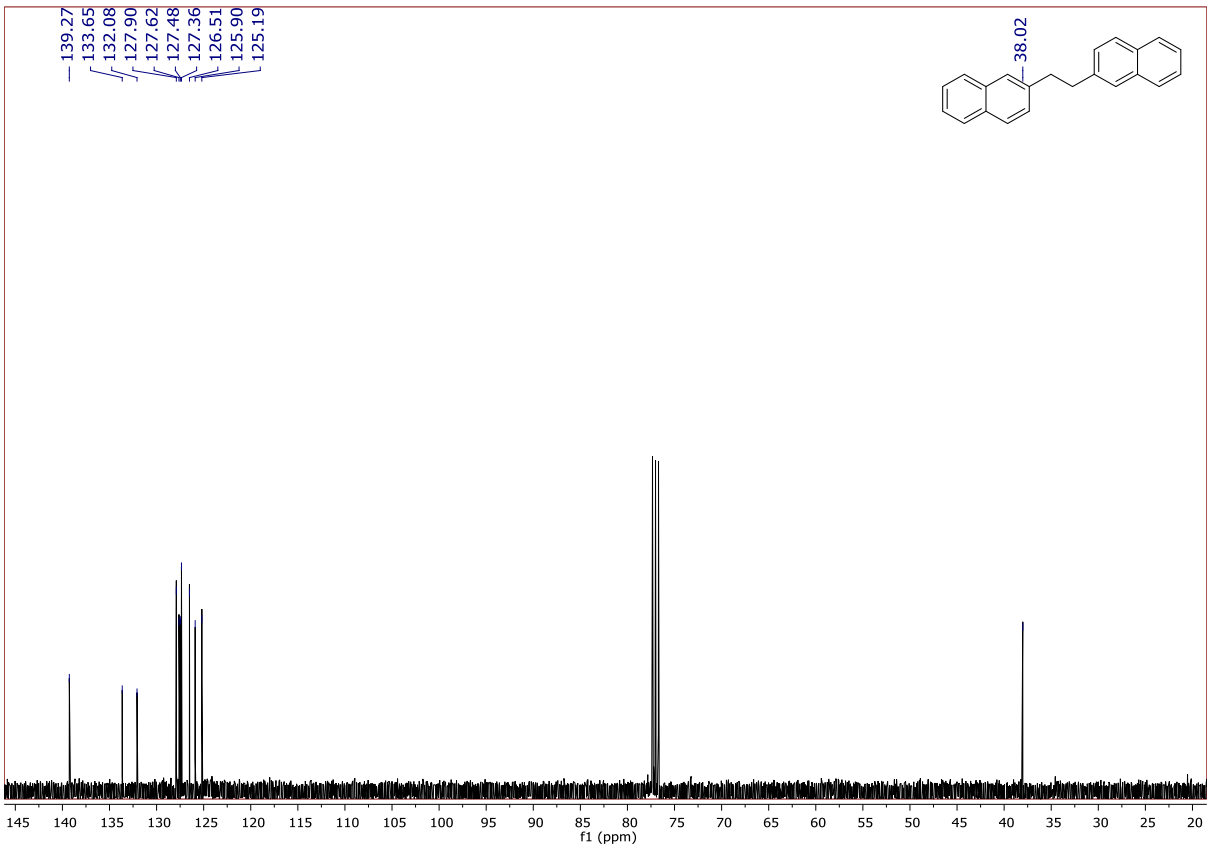
1,2-Bis(3,4-methylenedioxyphenyl)ethane (2o)



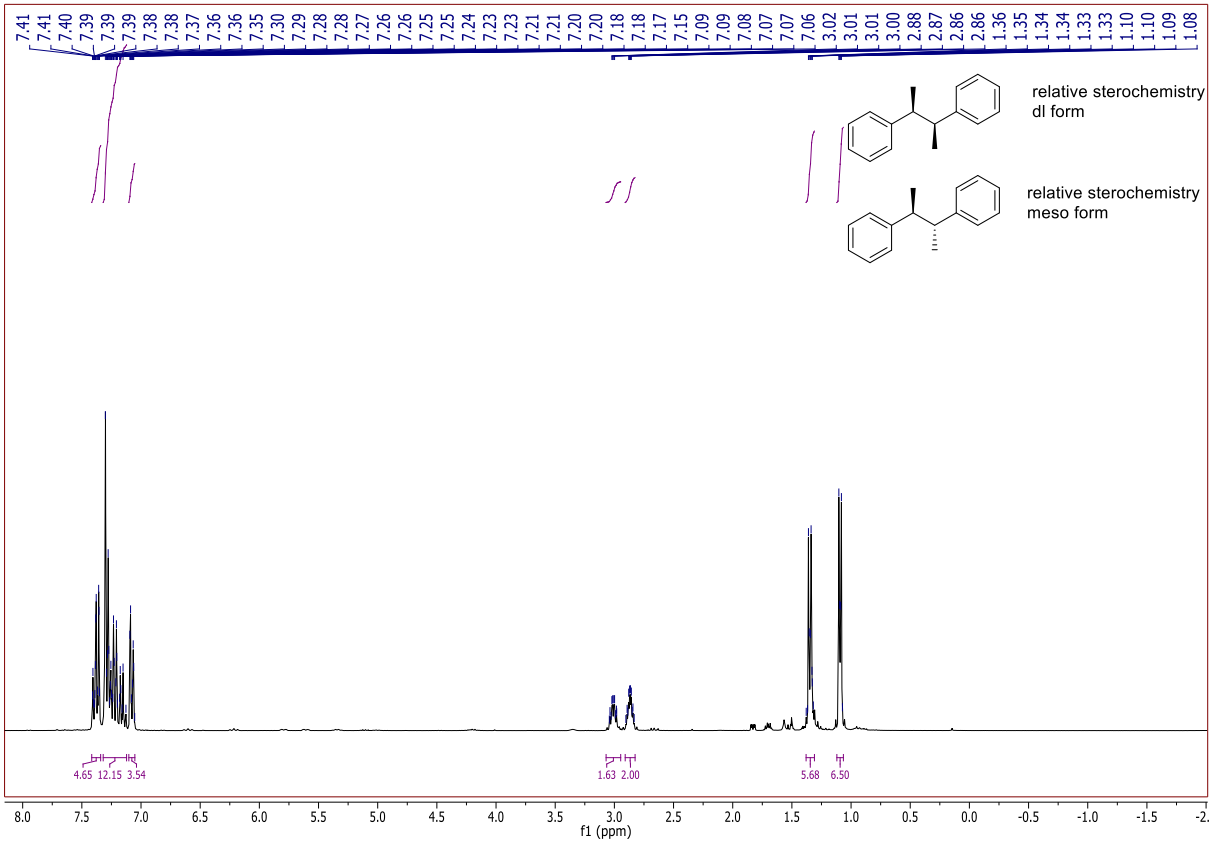


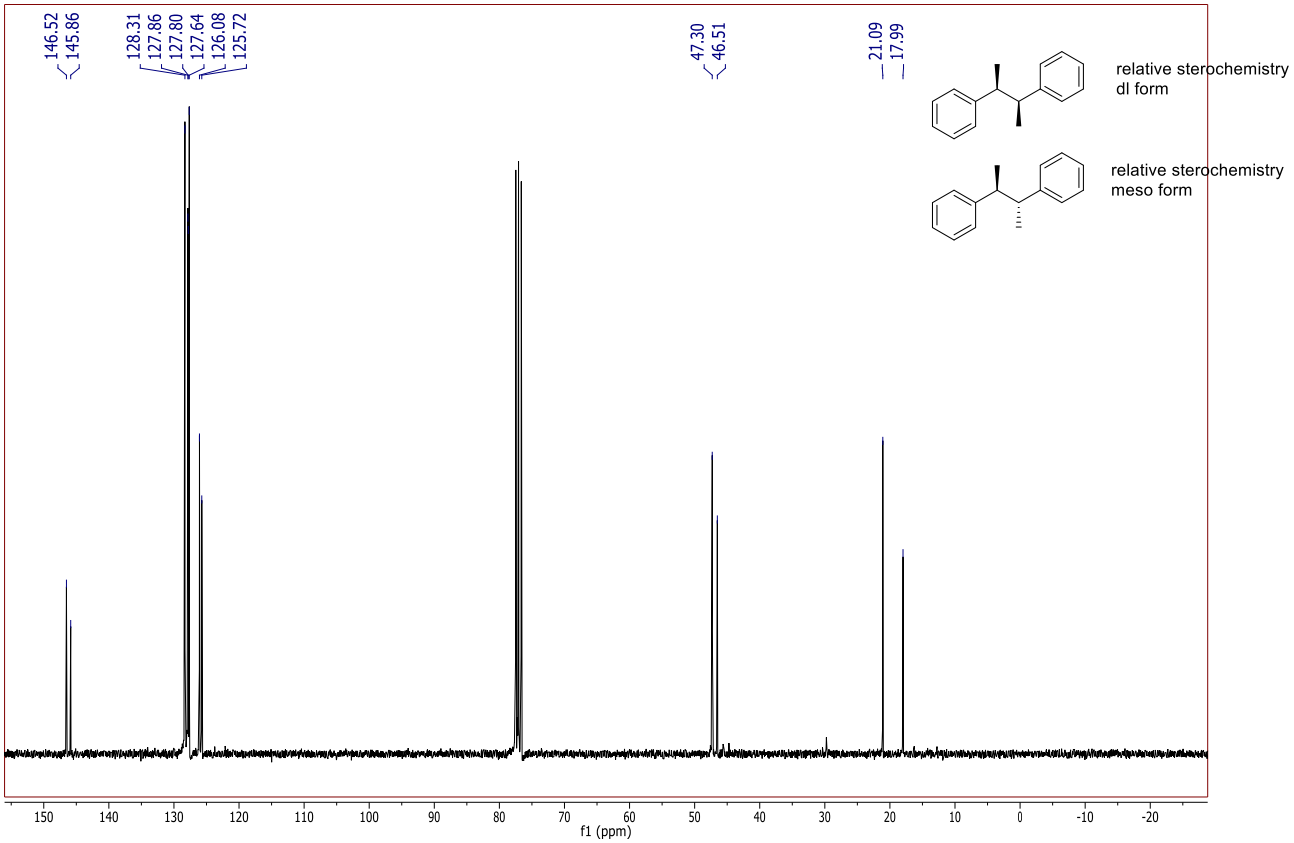
1,2-di(naphthalen-2-yl)ethane (2p)



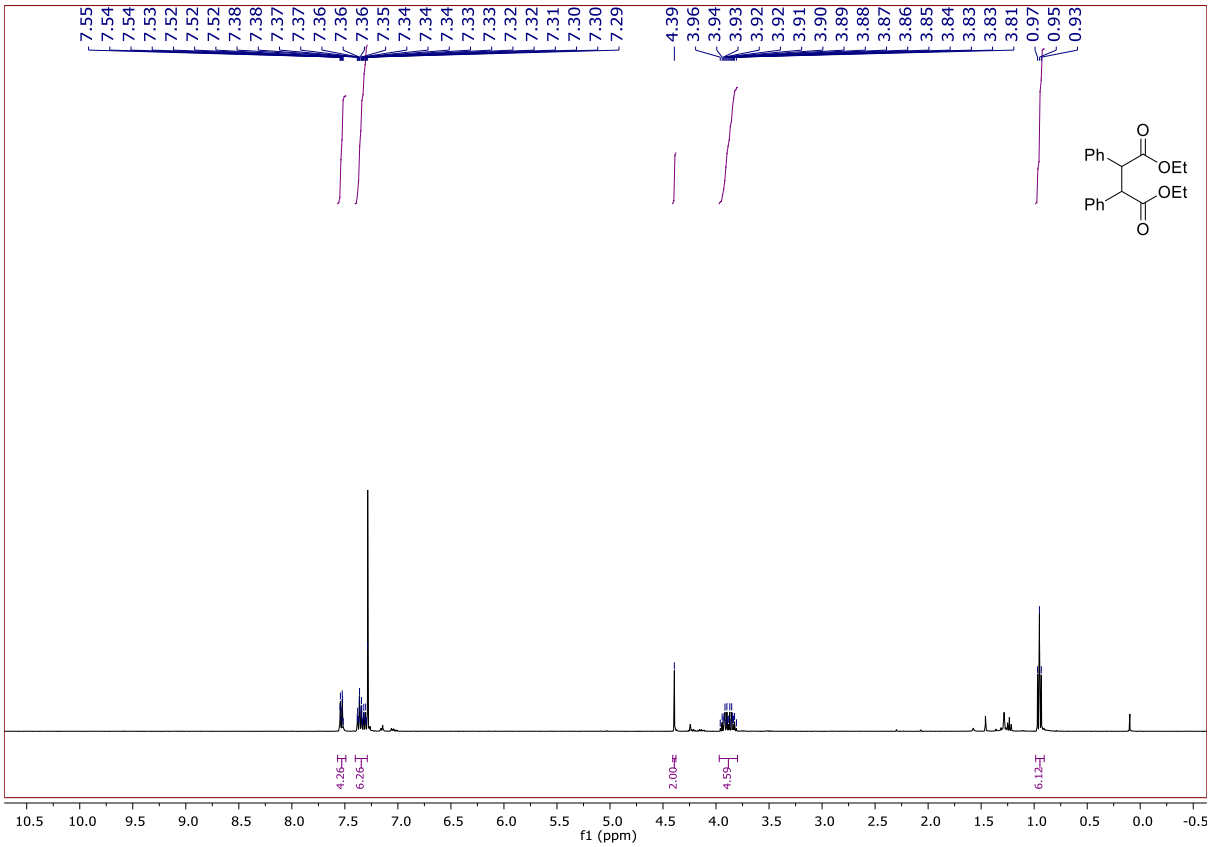


butane-2,3-diyldibenzene (2q)

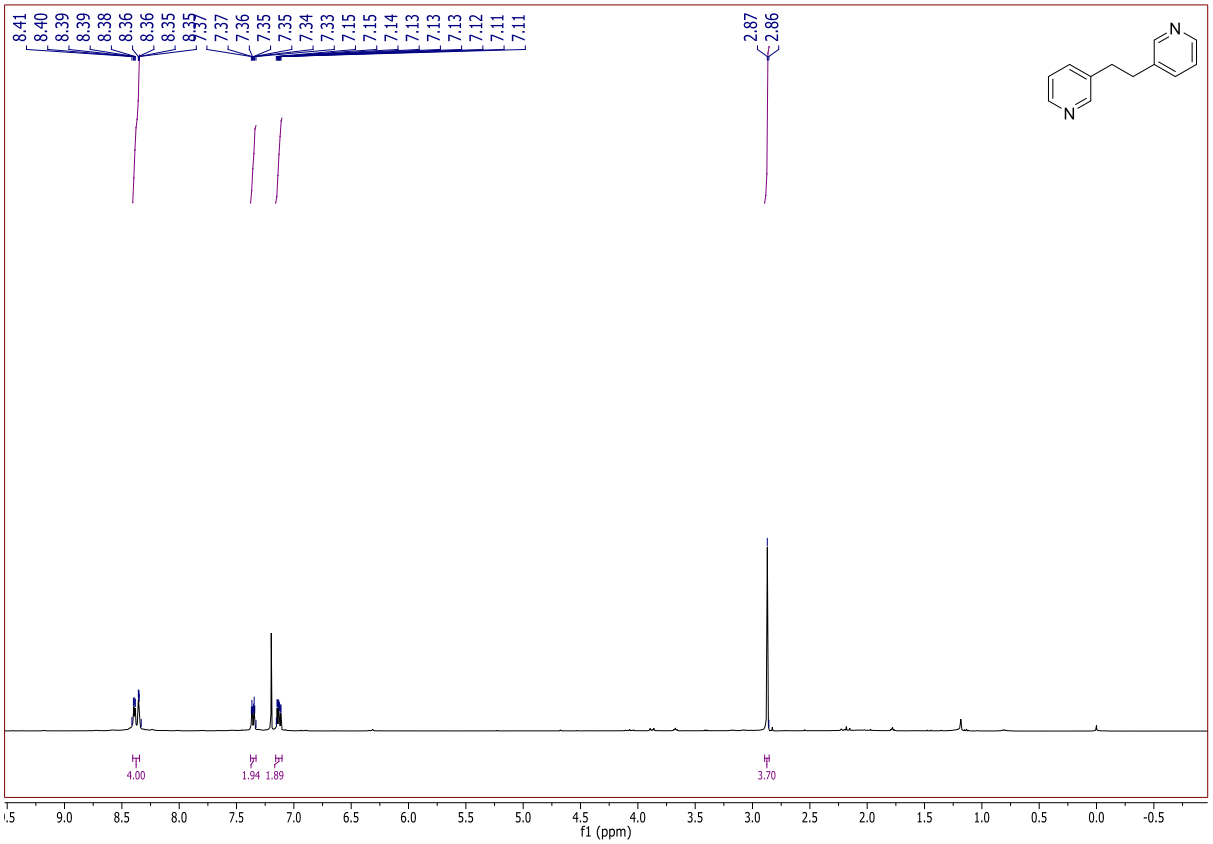

7.41 7.41 7.40 7.39 7.39 7.39 7.38 7.38 7.37 7.36 7.36 7.35 7.30 7.29 7.28 7.28 7.27 7.26 7.26 7.25 7.25 7.24 7.23 7.23 7.21 7.21 7.20 7.20 7.18 7.18 7.17 7.15 7.09 7.09 7.08 7.07 7.07 7.06 3.02 3.01 3.01 3.00 2.88 2.87 2.86 2.86 1.36 1.35 1.34 1.34 1.33 1.33 1.10 1.10 1.09 1.08
relative sterochemistry
dl form
relative sterochemistry
meso form
4.65 12.15 3.54
1.63 2.00
5.68 6.50
f1 (ppm)

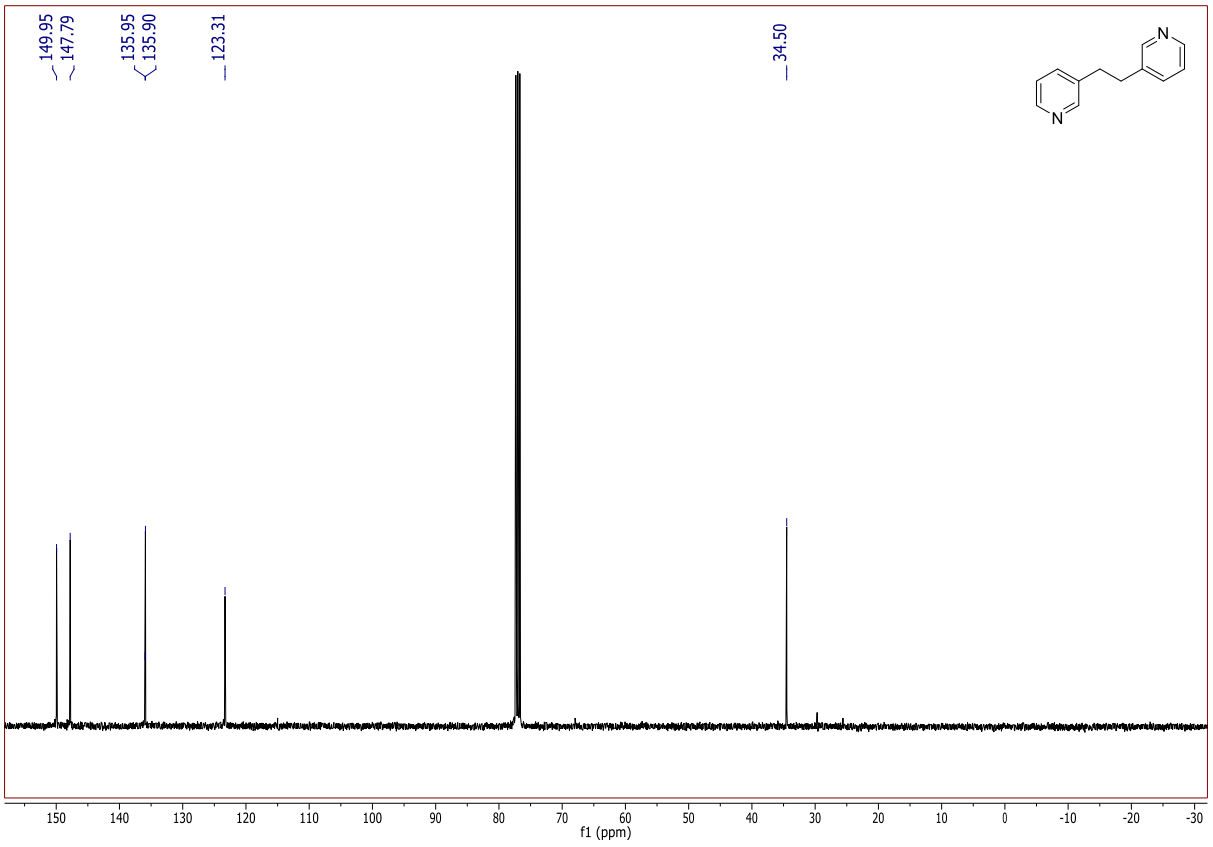

146.52 145.86 128.31 127.86 127.80 127.64 126.08 125.72 47.30 46.51 21.09 17.99
relative sterochemistry
dl form
relative sterochemistry
meso form
f1 (ppm)

Diethyl 2,3-diphenylsuccinate (2r)



1,2-di(pyridin-3-yl)ethane (2s)





References:

1. D. D. Perrin, W. L. F. Armarego, *Purification of Laboratory Chemicals* ; 3rd ed., Pergamon Press, Oxford, 1988.
2. W. C. Still, M. Kahn, A. J. Mitra, *J. Org. Chem*. 1978, **43**, 2923.
3. H. F. Klein, *Angew. Chem. Int. Ed. Engl*., 1971, **10**, 343 (b) H. F. Klein, H. H. Karsch, *Chem. Ber*. 1975, **108**, 944
4. H. F. Klein, H. H. Karsch, *Inorg. Chem*. 1975, **14**, 473.
5. H. F. Klein, H. H. Karsch, *Chem. Ber*. 1976, **109**, 1453.
6. K. Sato, Y. Inoue, T. Mori, A. Sakaue, A. Tarui, M. Omate, I. Kumadaki, A. Ando*, Org. Lett*. 2014, **16**, 3756-3759.
7. Y. L. Hu, F. Li, G. L. Gu, M. Lu, *Catalysis Letters*. 2011, **141**, 467-473.
8. H. G. Aurich, J. Eidel, M. Schmidt, *Chemische Berichte*, 1986, **119**, 18–35.
9. G. Cahiez, C. Chaboche, F. Mahuteau-Betzer, M. Ahr, *Org lett*. 2005, **7**, 1943-1946.
10. W. S. Lindsay , P. Stokes , L. G. Humber , V. Boekelheide, *J. Am. Chem. Soc*., 1961, **83**, 943-949.
11. S. Inaba, H. Matsumoto, R. D. Riek, *J. Org. Chem*., 1984, **49**, 2098-2107.
12. E. C. Taylor, J. S. Driscoll, *J. Org. Chem*., 1961, **26**, 3796–3802
13. H. Horton, R. H. Morris, R. H.Young, C. Hess, T. Sotter, *J. Am. Chem. Soc*., 1961, **79**, 411-414.