SUPPORTING INFORMATION FOR

Pd-catalysed amination on soluble polymer support: arylation of anilines with PEG-supported aryl halides

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General Information:

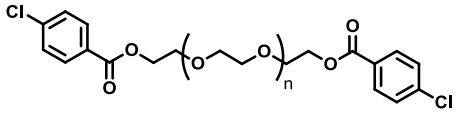
All commercially obtained reagents were used without further purification unless specified. PEG 2000 was purchased from Fluka as white flakes. Pyrrolidine, aniline, 2,5-dimethylaniline were previously distilled. Isopropanol was dried by usual methods.

NMR spectra (400 MHz for ^{1}H and 100 MHz for ^{13}C) were recorded with a Brucker ARX 400 spectrometer using $CDCl_3$ as solvent using the corresponding $CHCl_3$ signal as reference. IR spectra were recorded using a Perkin-Elmer Spectrum 1000 FT-IR.

General procedure for PEG supported aryl halides:

To the PEG 2000 (1 equiv.) in DMF (0,25 M) was added DIC (4 equiv.) and the solution was stirred for 30 min. at room temperature. Then it was added the corresponding benzoic acid halide (4 equiv.) and DMAP (0,4 equiv.). The mixture was stirred at 50ºC overnight. The mixture was filtered over a celite pad in order to remove the formed urea and the solution was placed in an ice bath. The product was precipitated by the addition of cold diethyl ether. The product was filtered and washed several times with cold diethyl ether in order to remove the DMF. The products were obtained as white solids and were vacuum dried and stored in a desiccator. The products were obtained in quantitative yield, and characterized by IR and NMR spectra.

PEG-bis(4-chlorobenzoate)

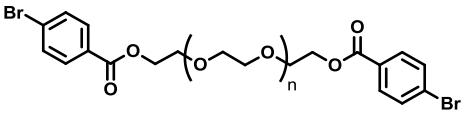


IR (KBr) ν_{max} (cm^{-1}): 2875, 1718, 1638, 1104.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.98 (d, *J* = 8.5 Hz, Ar-*H*, 4H), 7.41 (d, *J* = 8.5 Hz, Ar-*H*, 4H), 4.47 – 4.44 (m, CH_2 PEG, 4H), 3.83 – 3.45 (m, CH_2 PEG, 216H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.8 (*C*=O), 139.5 (*C*-q), 131.2 (*C*-Ar), 128.8 (*C*-Ar), 128.7 (C-q), 70.8 (*C*H_2 PEG), 70.7 (*C*H_2 PEG), 70.7 (*C*H_2 PEG), 69.3 (*C*H_2 PEG), 64.4 (*C*H_2 PEG).

PEG-bis(4-bromobenzoate)

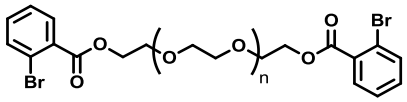


IR (KBr) ν_{max} (cm^{-1}): 2872, 1720, 1105.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.91 (d, *J* = 8.5 Hz, Ar-*H*, 4H), 7.57 (d, *J* = 8.5 Hz, Ar-*H*, 4H), 4.47 – 4.44 (m, CH_2 PEG, 4H), 3.83 – 3.63 (m, CH_2 PEG, 192H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.9 (*C*=O), 131.8 (*C*-Ar), 131.4 (*C*-Ar), 129.1 (*C*-q), 128.2 (*C*-q), 70.8 (*C*H_2 PEG), 70.8 (*C*H_2 PEG), 70.7 (*C*H_2 PEG), 69.2 (*C*H_2 PEG), 64.5 (*C*H_2 PEG).

PEG-bis(4-chlorobenzoate)

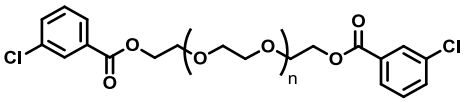


IR (KBr) ν_{max} (cm^{-1}): 2836, 1730, 1655, 1114.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.79 (dd, *J* = 7.5, 1.9 Hz, Ar-*H*, 2H), 7.63 (dd, *J* = 7.7, 1.4 Hz, Ar-*H*, 2H), 7.36 – 7.28 (m, Ar-*H*, 4H), 4.47 – 4.44 (m, PEG, 4H), 3.81 – 3.43 (m, PEG, 190H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.1 (C=O), 134.3 (C-Ar), 132.6 (C-Ar), 132.1 (C-Ar), 131.5 (C-Ar), 127.2 (C-Ar), 121.7 (C-Ar), 70.7 (CH_2 PEG), 70.6 (CH_2 PEG), 70.6 (CH_2 PEG), 69.0 (CH_2 PEG), 64.6 (CH_2 PEG).

PEG-bis(3-chlorobenzoate)



IR (KBr) ν_{max} (cm^{-1}): 2882, 1723, 1467, 1115.

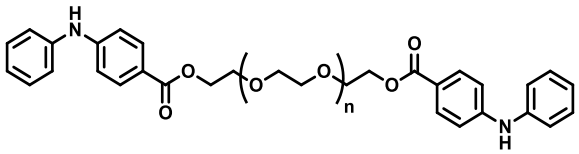
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.90 (m, Ar-*H*, 2H), 7.88 (d, *J* = 7.8 Hz, Ar-*H*, 2H), 7.49 – 7.47 (m, Ar-*H*, 2H), 7.34 (t, *J* = 7.9 Hz, Ar-*H*, 2H), 4.48 – 4.46 (m, C*H*$_2$ PEG, 4H), 3.78 – 3.59 (m, PEG, 208H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.3 (*C*=O), 134.4 (*C*-q), 133.0 (*C*-Ar), 131.8 (*C*-q), 129.7 (*C*-Ar), 129.7 (*C*-Ar), 127.8 (*C*-Ar), 70.6 (*C*H_2 PEG), 70.6 (*C*H_2 PEG), 70.5 (*C*H_2 PEG), 69.0 (*C*H_2 PEG), 64.4 (*C*H_2 PEG).

General procedure for Pd-catalyzed amination on PEG:

To a screw-cap test-tube equipped with a magnetic stir bar, was added the PEG supported aryl halide (0,1 mmol), Pd_2dba_3 (1 mol %), ligand (3 mol %) and base (1,2 equiv.). The vial was sealed with a suba-seal, evacuated and backfilled with argon. Then was added the amine (1,2 equiv.) and the suba-seal was replaced by the teflon screw-cap. The reaction mixture was heated at the desired temperature for 8 h (aryl amines) or 20 h (alkyl amines) (see tables). The solution was allowed to cool to room temperature, dissolved with dried isopropanol and filtered over a celite pad. The solution was placed in an ice bath and the product was precipitated by the addition of cold diethyl ether. The product was washed several times with cold diethyl ether. The obtained product was dried over vacuum and stored in a desiccator.

PEG-bis(4-(phenylamino)benzoate) LC 177.13

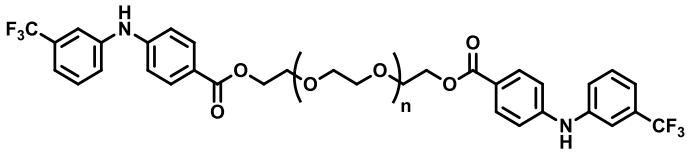


IR (KBr) ν_{max} (cm^{-1}): 2873, 1705, 1591, 1104.

^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.91 (d, *J* = 8.8 Hz, Ar-*H*, 4H), 7.31 (t, *J* = 7.9 Hz, Ar-*H*, 4H), 7.17 (d, *J* = 7.6 Hz, Ar-*H*, 4H), 7.03-7.00 (m, Ar-*H*, 6H), 4.43 – 4.41 (m, CH_2 PEG, 4H), 3.82 – 3.63 (m, CH_2 PEG, 218H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.5 (*C*=O), 148.2 (*C*-q), 141.1 (*C*-q), 131.7 (*C*-Ar), 129.5 (*C*-Ar), 123.1 (*C*-Ar), 120.9 (*C*-Ar), 120.4 (*C*-Ar), 114.8 (*C*-Ar), 70.8 (*C*H_2 PEG), 70.7 (CH_2 PEG), 70.6 (*C*H_2 PEG), 69.5 (*C*H_2 PEG), 63.8 (*C*H_2 PEG).

PEG-bis(4-(3-(trifluoromethyl)phenylamino)benzoate)

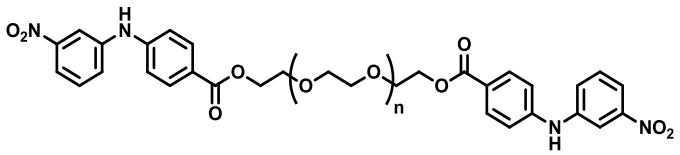


IR (KBr) ν_{max} (cm^{-1}): 2873, 1706, 1596, 110.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.96 (d, *J* = 8.6 Hz, Ar-*H*, 4H), 7.40 – 7.39 (m, Ar-*H*, 6H), 7.23 (d, *J* = 7.5 Hz, Ar-*H*, 2H), 7.07 (d, *J* = 8.7 Hz, Ar-*H*, 4H), 4.45 – 4.42 (m, CH_2 PEG, 4H), 3.82 – 3.60 (m, CH_2 PEG, 215H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.3 (*C*=O), 147.2 (*C*-q), 142.2 (*C*-q), 131.8 (*C*-Ar), 130.1 (*C*-Ar), 122.3 (*C*-Ar), 122.1 (C-q), 118.8 (*C*-Ar), 115.8 (*C*-Ar), 115.6 (*C*-Ar), 70.9 (*C*H_2 PEG), 70.8 (*C*H_2 PEG), 70.7 (*C*H_2 PEG), 69.5 (*C*H_2 PEG), 63.9 (*C*H_2 PEG).

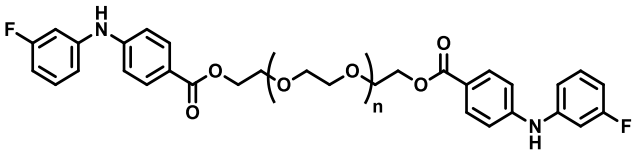
PEG-bis(4-(3-nitrophenylamino)benzoate)



^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.00 – 7.97 (m, Ar-*H*, 6H), 7.79 (d, *J* = 7.8 Hz, Ar-*H*, 2H), 7.49 – 7.39 (m, Ar-*H*, 4H), 7.13 (d, *J* = 8.7 Hz, Ar-*H*, 4H), 4.46 – 4.43 (m, CH_2 PEG, 4H), 3.83 – 3.59 (m, CH_2 PEG, 192H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.2 (*C*=O), 149.4 (*C*-Ar), 146.5 (*C*-Ar), 143.2 (*C*-Ar), 131.8 (*C*-Ar), 130.3 (*C*-Ar), 124.2 (*C*-Ar), 122.8 (*C*-Ar), 116.3 (*C*-Ar), 116.2 (*C*-Ar), 112.8 (*C*-Ar), 70.9 (*C*H_2 PEG), 70.8 (*C*H_2 PEG), 70.7 (*C*H_2 PEG), 69.4 (*C*H_2 PEG), 64.0 (*C*H_2 PEG).

PEG-bis(4-(3-fluorophenylamino)benzoate)

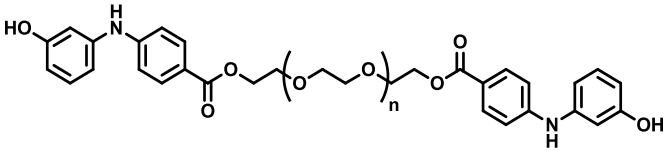


IR (KBr) ν_{max} (cm^{-1}): 2871, 1705, 1597, 1106.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.94 (d, *J* = 8.7 Hz, Ar-*H*, 4H), 7.26 – 7.21 (m, Ar-*H*, 2H), 7.06 (d, *J* = 8.7 Hz, Ar-*H*, 4H), 6.93 – 6.87 (m, Ar-*H*, 4H), 6.68 (td, *J* = 8.5, 2.1 Hz, Ar-*H*, 2H), 4.44 – 4.42 (m, CH_2 PEG, 4H), 3.82 – 3.60 (m, CH_2 PEG, 210H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.4 (*C*=O), 163.7 (d, *J* = 245.0 Hz, *C*-q), 147.3 (*C*-q), 143.2 (d, *J* = 10.3 Hz, *C*-q), 131.7 (2x*C*-Ar), 130.6 (d, *J* = 9.8 Hz, *C*-Ar), 121.8 (*C*-q), 115.6 (2x*C*-Ar), 114.8 (d, *J* = 2.7 Hz, *C*-Ar), 109.0 (d, *J* = 21.3 Hz, *C*-Ar), 106.2 (d, *J* = 24.5 Hz, *C*-Ar), 70.9 (*C*H_2 PEG), 70.8 (*C*H_2 PEG), 70.7 (*C*H_2 PEG), 69.5 (*C*H_2 PEG), 63.9 (*C*H_2 PEG).

PEG-bis(4-(3-hydroxyphenylamino)benzoate)

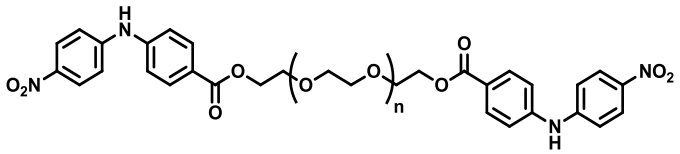


IR (KBr) ν_{max} (cm^{-1}): 2873, 1702, 1596, 1105.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.89 (d, *J* = 8.7 Hz, Ar-*H*, 4H), 7.11 (t, *J* = 8.0 Hz, Ar-*H*, 2H), 7.03 (d, *J* = 8.7 Hz, Ar-*H*, 4H), 6.73 – 6.68 (m, Ar-*H*, 4H), 6.52 (dd, *J* = 8.1, 1.7 Hz, Ar-*H*, 2H), 4.43 – 4.41 (m, CH_2 PEG, 4H), 3.81 – 3.58 (m, CH_2 PEG, 220H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.6 (*C*=O), 158.0 (*C*-q), 148.5 (*C*-q), 142.4 (*C*-q), 131.6 (2x*C*-Ar), 130.2 (*C*-Ar), 120.5 (*C*-q), 114.9 (*C*-Ar), 111.4 (*C*-Ar), 110.2 (*C*-Ar), 107.4 (*C*-Ar), 71.0 (*C*H_2 PEG), 70.8 (*C*H_2 PEG), 70.6 (*C*H_2 PEG), 69.5 (*C*H_2 PEG), 63.8 (*C*H_2 PEG).

PEG-bis(4-chlorobenzoate)

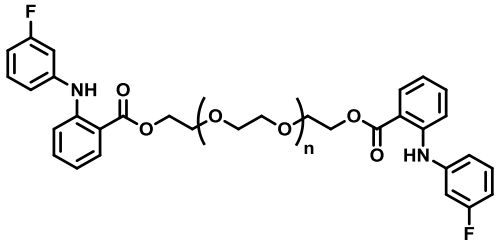


Obs. Product did not precipitate with diethyl ether. It was only observed by ^{1}H NMR along with the *p*-nitro aniline and unreacted starting material.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.06 (d, *J* = 9.1 Hz, Ar-*H*, 4H), 7.93 (d, *J* = 8.6 Hz, Ar-*H*, 4H), 7.31 (d, *J* = 8.7 Hz, Ar-*H*, 4H), 7.27 (m, Ar-*H*, 4H), 4.42 – 4.40 (m, CH_2 PEG, 4H), 3.80 – 3.57 (m, CH_2 PEG, 218H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.2 (*C*=O), 131.3 (*C*-Ar), 131.2 (*C*-Ar), 125.8 (*C*-Ar), 125.8 (*C*-Ar), 118.2 (*C*-Ar), 115.7 (*C*-Ar), 70.6 (*C*H$_2$ PEG), 70.3 (*C*H$_2$ PEG), 69.3 (*C*H$_2$ PEG), 63.8 (*C*H$_2$ PEG).

PEG-bis(2-(phenylamino)benzoate)



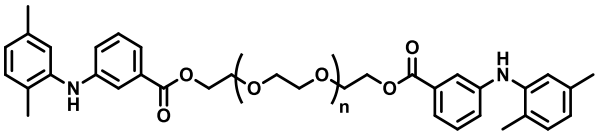
Obs. It was observed by ^{1}H NMR along with the unreacted starting material.

IR *not performed.*

^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.45 (s, N*H*, 2H), 8.00 (d, *J* = 8.0 Hz, Ar-*H*, 2H), 7.34 – 7.26 (m, Ar-*H*, 6H), 6.99–6.76 (m, Ar-*H*, 4H), 6.81–6.72 (m, Ar-*H*, 4H), 4.46 (t, *J* = 11.0, 6.4 Hz, CH_2 PEG, 4H), 3.84 – 3.45 (m, CH_2 PEG, 246 H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 168.3 (*C*=O), 163.6 (d, *J* = 245.3 Hz, *C*-Ar), 146.9 (C-Ar), 142.9 (d, *J* = 10.2 Hz, C-Ar), 134.3 (C-Ar), 132.0 (C-Ar), 130.6 (d, *J* = 9.8 Hz, C-Ar), 118.2 (C-Ar), 117.2 (d, *J* = 2.7 Hz, C-Ar), 114.7 (C-Ar), 113.0 (C-Ar), 109.8 (d, *J* = 21.3 Hz, C-Ar), 108.3 (d, *J* = 23.9 Hz, C-Ar), 70.8 (CH_2 PEG), 70.7 (CH_2 PEG), 70.7 (CH_2 PEG), 69.3 (CH_2 PEG), 64.0 (CH_2 PEG).

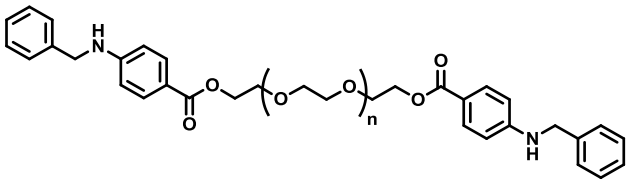
PEG-bis(3-(2,5-dimethylphenylamino)benzoate)



^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.56 – 7.52 (m, Ar-*H*, 4H), 7.26 (m, Ar-*H*, 2H), 7.10 – 7.03 (m, Ar-*H*, 6H), 6.80 (d, *J* = 7.5 Hz, Ar-*H*, 2H), 4.46 – 4.43 (m, C*H*$_2$ PEG, 4H), 3.82 – 3.64 (m, C*H*$_2$ PEG, 250H), 2.27 (s, C*H*$_3$, 6H), 2.19 (s, C*H*$_3$, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.8 (*C*=O), 144.8 (*C*-Ar), 140.3 (*C*-Ar), 136.7 (*C*-Ar), 131.3 (*C*-Ar), 131.0 (*C*-Ar), 129.3 (*C*-Ar), 126.5 (*C*-Ar), 123.9 (*C*-Ar), 121.2 (*C*-Ar), 120.8 (*C*-Ar), 120.8 (*C*-Ar), 118.0 (*C*-Ar), 70.8 (*C*H$_2$ PEG), 70.7 (*C*H$_2$ PEG), 69.4 (*C*H$_2$ PEG), 64.3 (*C*H$_2$ PEG), 21.3 (*C*H$_3$), 17.6 (*C*H$_3$).

PEG-bis(4-(benzylamino)benzoate)

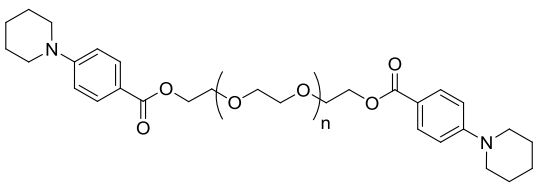


IR (KBr) ν_{max} (cm^{-1}): 2874, 1698, 1604, 1104.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.85 (d, *J* = 8.7 Hz, Ar-*H*, 4H), 7.33 (m, Ar-*H*, 10H), 6.57 (d, *J* = 8.7 Hz, Ar-*H*, 4H), 4.37 (m, C*H*$_2$ benzylic, C*H*$_2$ PEG, 8H), 3.79 – 3.62 (m, C*H*$_2$ PEG, 282H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.7 (*C*=O), 152.0 (*C*-q), 138.5 (*C*-q), 131.7 (*C*-Ar), 128.8 (*C*-Ar), 128.6 (*C*-Ar), 127.5 (*C*-Ar), 127.4 (*C*-Ar), 127.2 (*C*-q), 126.9 (*C*-Ar), 111.7 (*C*-Ar), 70.7 (*C*H$_2$ PEG), 70.6 (*C*H$_2$ PEG), 70.4 (*C*H$_2$ PEG), 69.4 (*C*H$_2$ PEG), 63.5 (*C*H$_2$ PEG), 47.6 (*C*H$_2$).

PEG-bis(4-(piperidin-1-yl)benzoate)

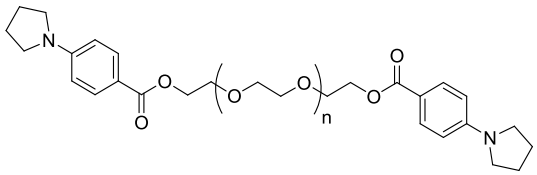


IR (KBr) ν_{max} (cm^{-1}): 2873, 1700, 1603, 1104.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.88 (d, *J* = 8.6 Hz, Ar-*H*, 4H), 6.82 (d, *J* = 8.6 Hz, Ar-*H*, 4H), 4.40 – 4.38 (m, CH_2 PEG, 4H), 3.78 – 3.62 (m, CH_2 PEG, 205H), 3.30 (m, CH_2, 8H), 1.63 (m, CH_2, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.7 (*C*=O), 154.6 (*C*-Ar), 131.4 (*C*-Ar), 118.6 (C-Ar), 113.6 (C-Ar), 70.7 (CH_2 PEG), 70.6 (CH_2 PEG), 69.5 (CH_2 PEG), 63.6 (CH_2 PEG), 48.8 (*C*H_2), 25.4 (*C*H_2), 24.4 (*C*H_2).

PEG-bis(4-(pyrrolidin-1-yl)benzoate)



IR (KBr) ν_{max} (cm^{-1}): 2871, 1696, 1605, 1106.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.89 (d, *J* = 8.6 Hz, Ar-*H*, 4H), 6.49 (d, *J* = 8.6 Hz, Ar-*H*, 4H), 4.40 (m, PEG CH_2, 4H), 3.81 – 3.63 (m, PEG CH_2, 240H), 3.34 (m, CH_2, 8H), 2.02 (m, CH_2, 8H).

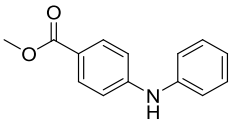
^{13}C NMR (100 MHz, $CDCl_3$) δ: 167.2 (*C*=O), 151.0 (*C*-Ar), 131.6 (*C*-Ar), 116.2 (*C*-Ar), 110.7 (*C*-Ar), 70.8 (*C*H_2 PEG), 70.6 (*C*H_2 PEG), 69.6 (*C*H_2 PEG), 63.5 (*C*H_2 PEG), 47.6 (*C*H_2), 25.6 (*C*H_2).

General procedure for cleavage of compounds from the polymer support:[1]

The PEG bound compound (1 equiv.) was dissolved in methanol and a1 M solution of potassium cyanide (2 equiv.) in methanol

was added. The mixture was stirred at ambient temperature for 12 h. After completion of reaction, the inorganic material was removed by filtration and then filtrate was concentrated under reduced pressure. The polymer was precipitated out with excess of cold ether and removed by filtration. The filtrate was evaporated to dryness to obtain white products in good yields (70 - 90%). The spectral data is according to literature.

Methyl 4-(phenylamino)benzoate [2]

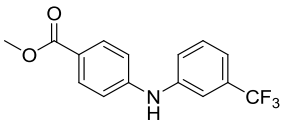


MP: 115-117ºC;

IR (NaCl, cm^{-1}): 3334, 1695, 1590;

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.91 (d, *J* = 8.4 Hz, Ar-*H*, 2H), 7.34 (m, Ar-*H*, 2H), 7.17 (d, *J* = 7.9 Hz, Ar-*H*, 2H), 7.06 (t, *J* = 7.3 Hz, Ar-*H*, 1H), 6.99 (d, *J* = 8.4 Hz, Ar-*H*, 2H), 6.04 (bs, N*H*, 1H), 3.87 (s, CH_3, 3H).

Methyl 4-(3-(trifluoromethyl)phenylamino)benzoate [3]

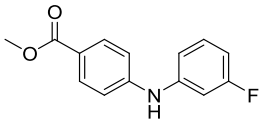


MP: 124-126 ºC;

IR (NaCl, cm^{-1}): 3344, 1694, 1593, 1338;

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.96 (d, *J* = 8.3 Hz, Ar-*H*, 2H), 7.47 – 7.22 (m, Ar-*H*, 4H), 7.03 (d, *J* = 8.5 Hz, Ar-*H*, 2H), 6.21 (bs, N*H*, 1H), 3.89 (s, CH_3, 3H).

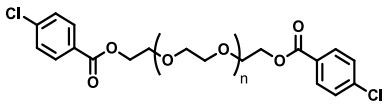
Methyl 4-(3-fluorophenylamino)benzoate [4]

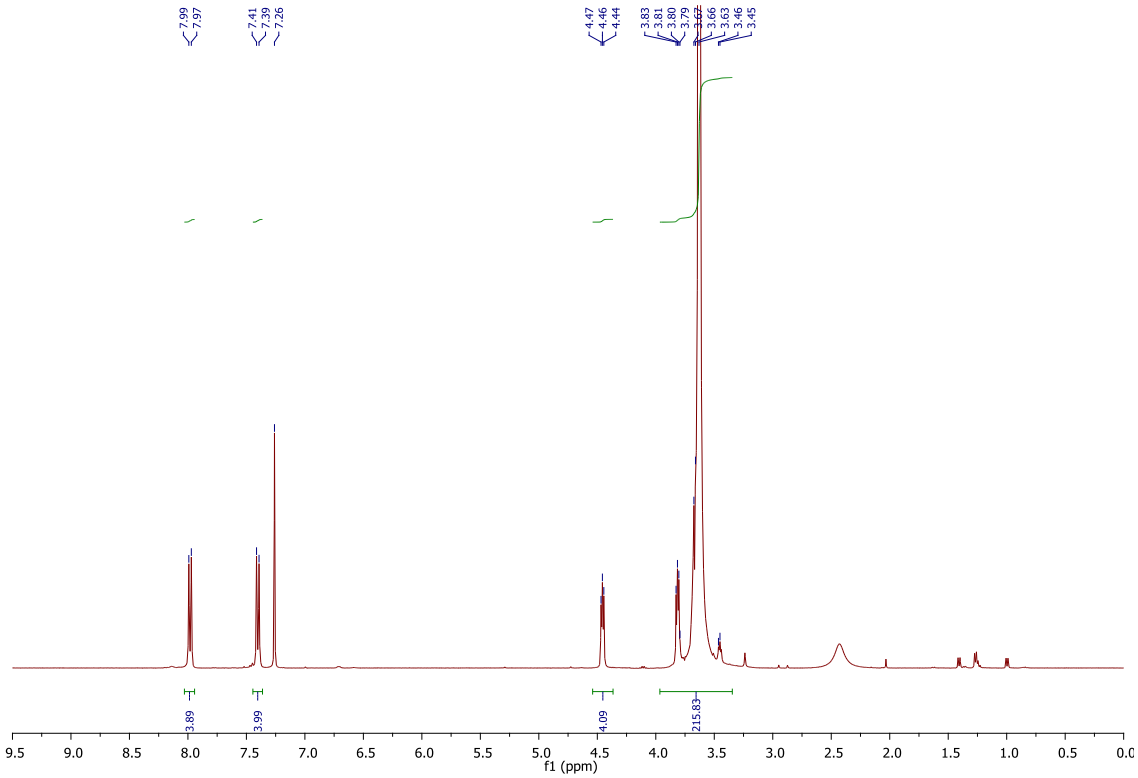


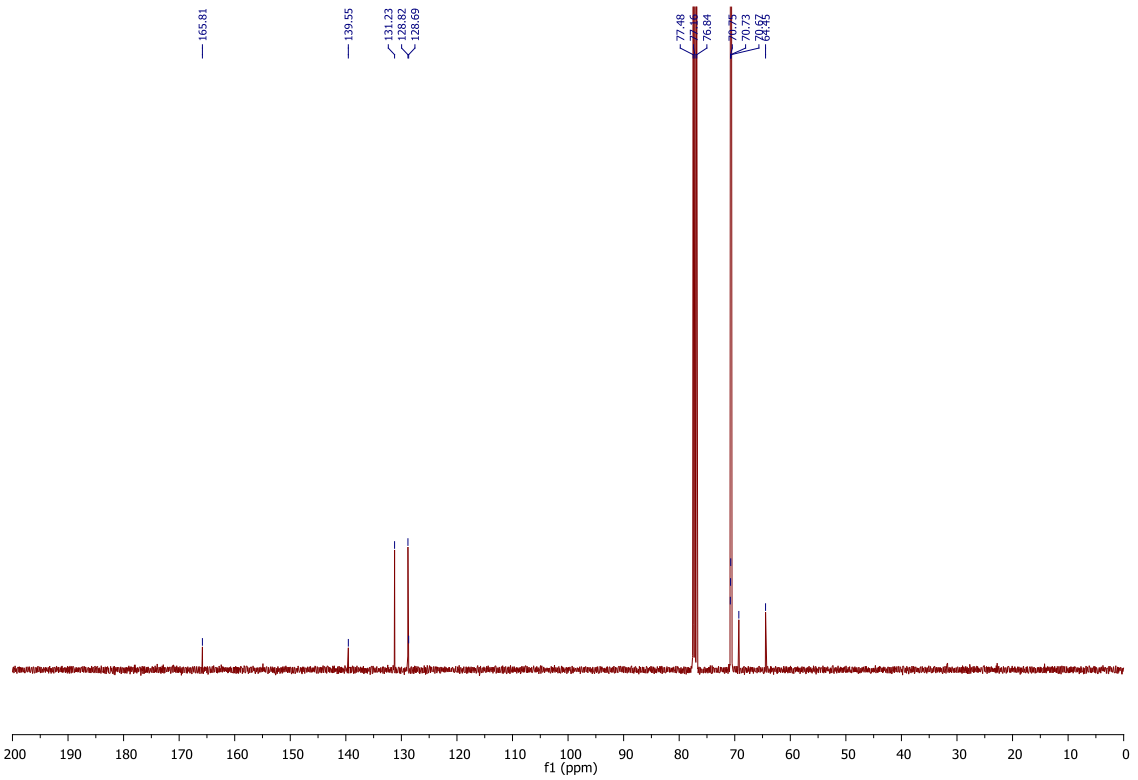
MP: 129-131ºC;

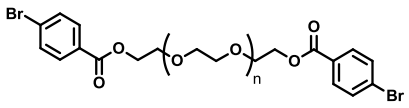
IR (NaCl, cm^{-1}): 3337, 1693, 1597;

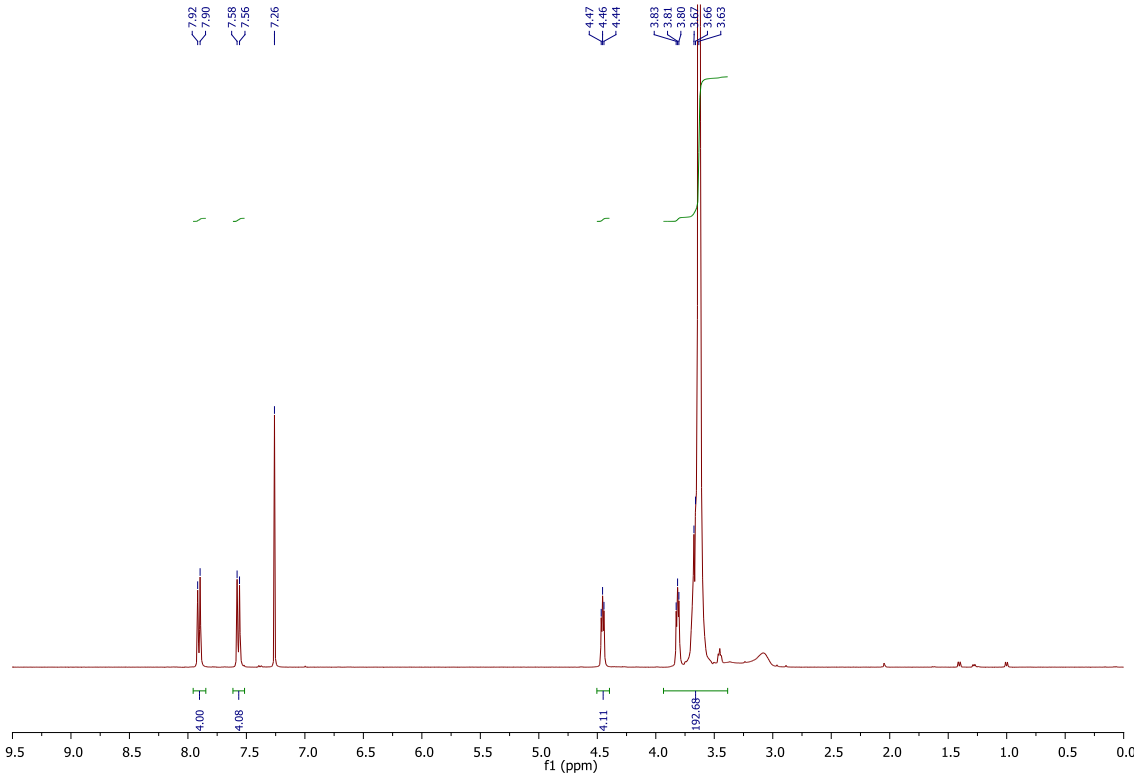
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.94 (d, *J* = 8.5 Hz, Ar-*H*, 2H), 7.31 – 7.22 (m, Ar-*H*, 1H), 7.04 (d, *J* = 8.5 Hz, Ar-*H*, 2H), 6.95 – 6.86 (m, 2H), 6.72 (t, *J* = 8.3 Hz, Ar-*H*, 1H), 3.88 (s, CH_3, 3H).

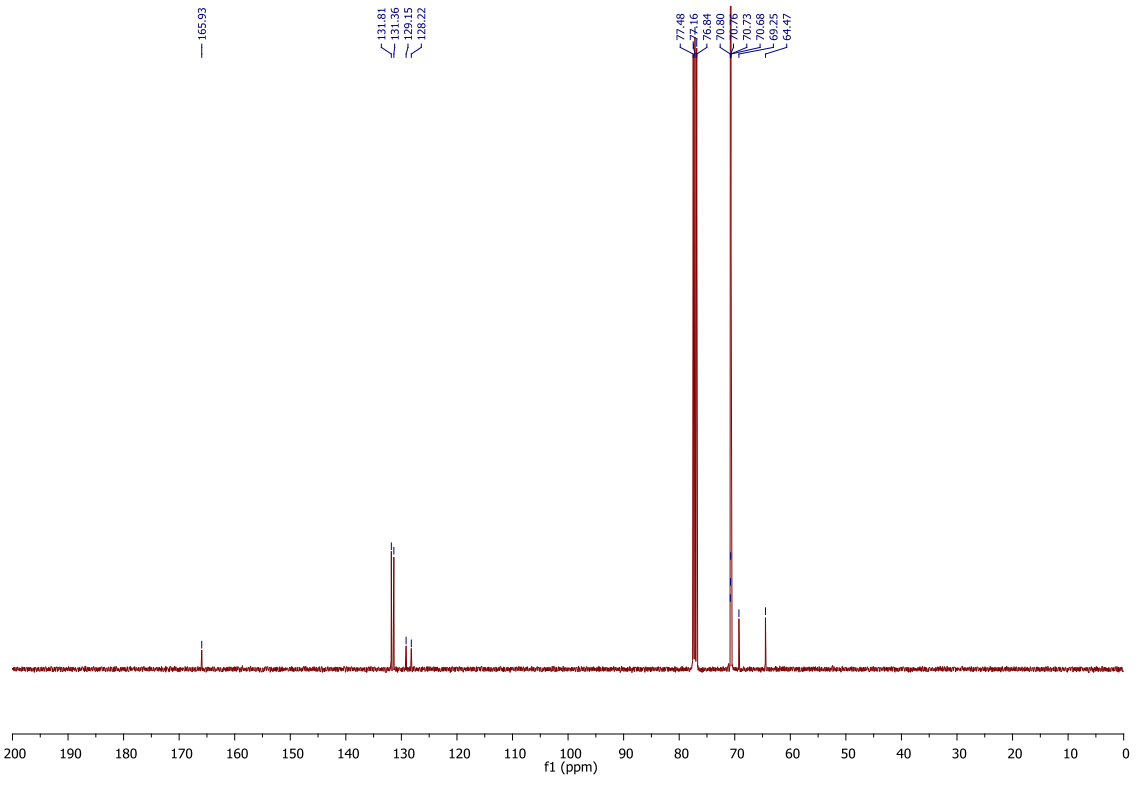


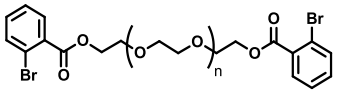


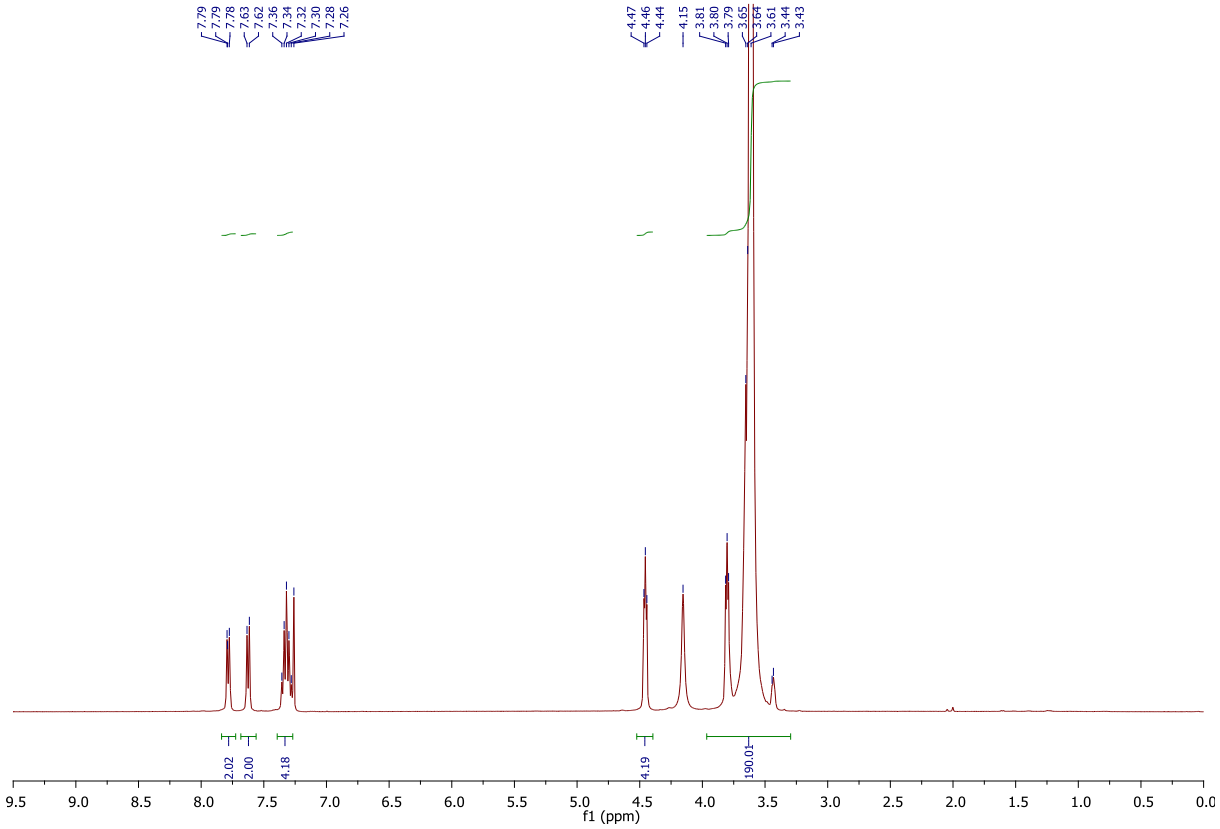


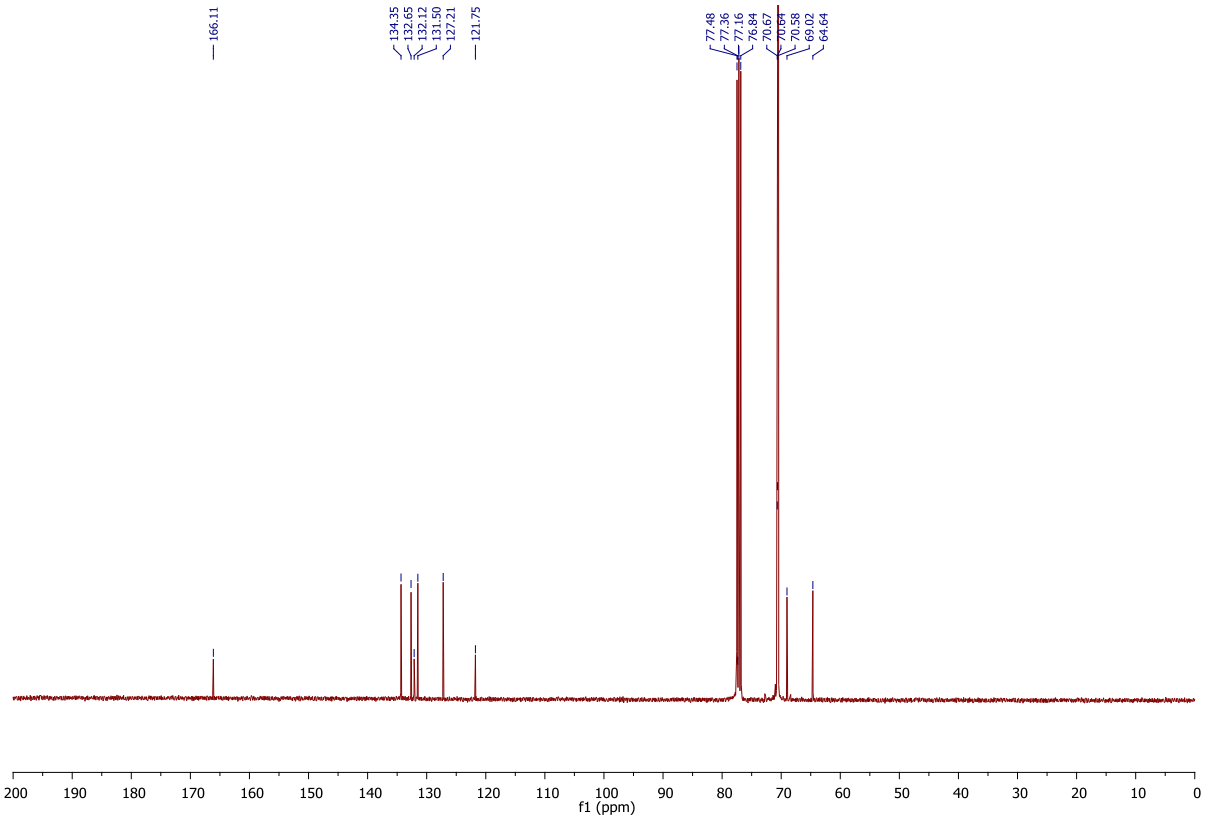


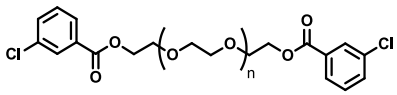


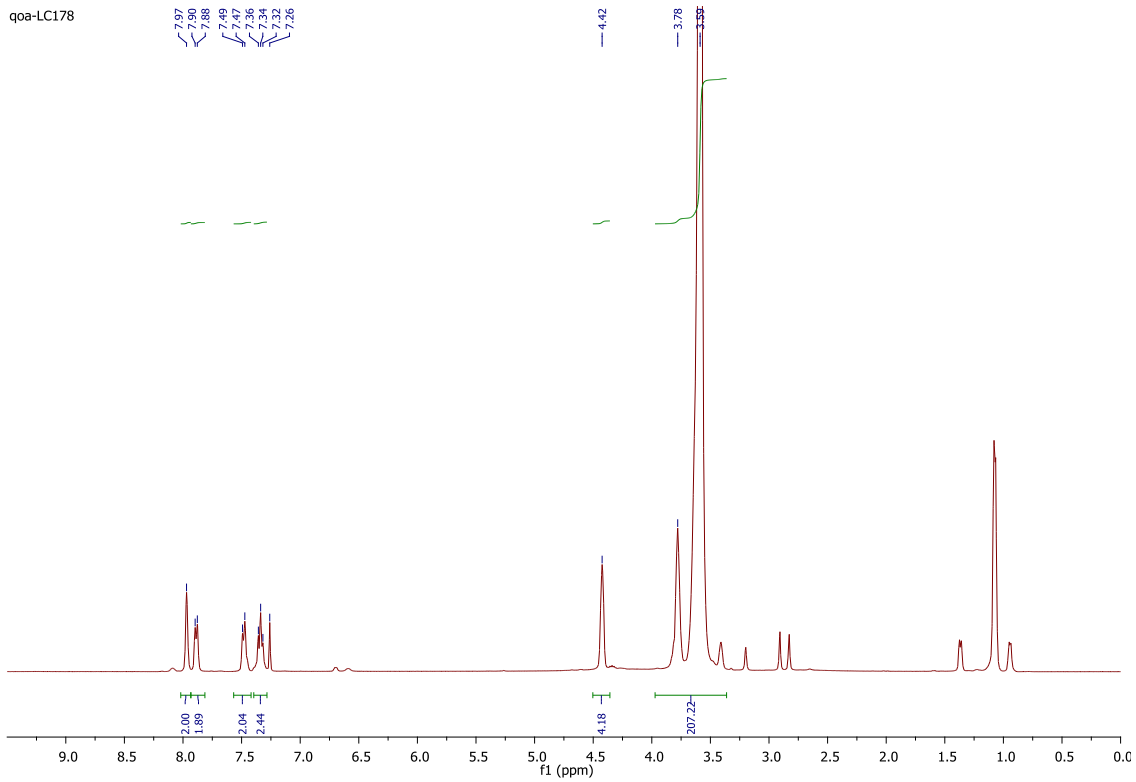


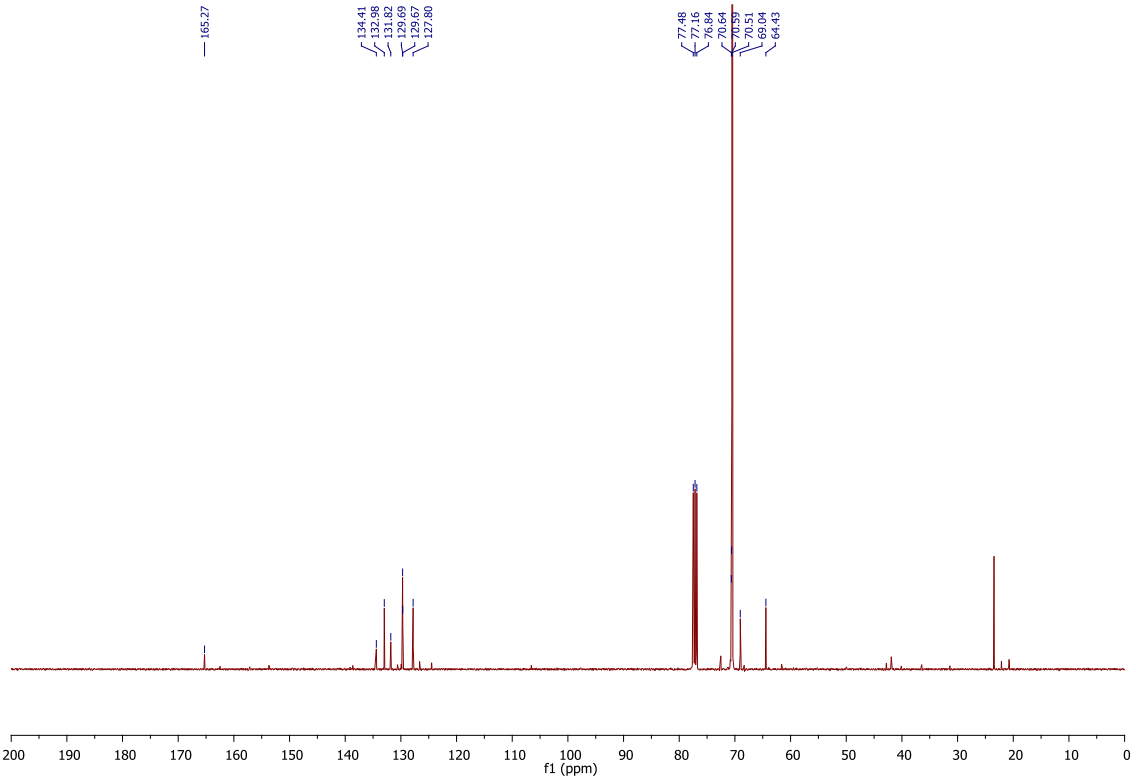

Br
O
O
O
O
n
O
O
Br

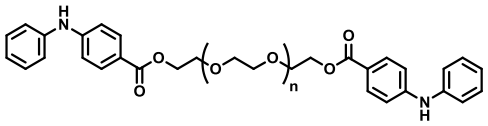

7.79
7.79
7.78
7.63
7.62
7.36
7.34
7.32
7.30
7.28
7.26
4.47
4.46
4.44
4.15
3.81
3.80
3.79
3.65
3.64
3.61
3.44
3.43
2.02
2.00
4.18
4.19
190.01
f1 (ppm)

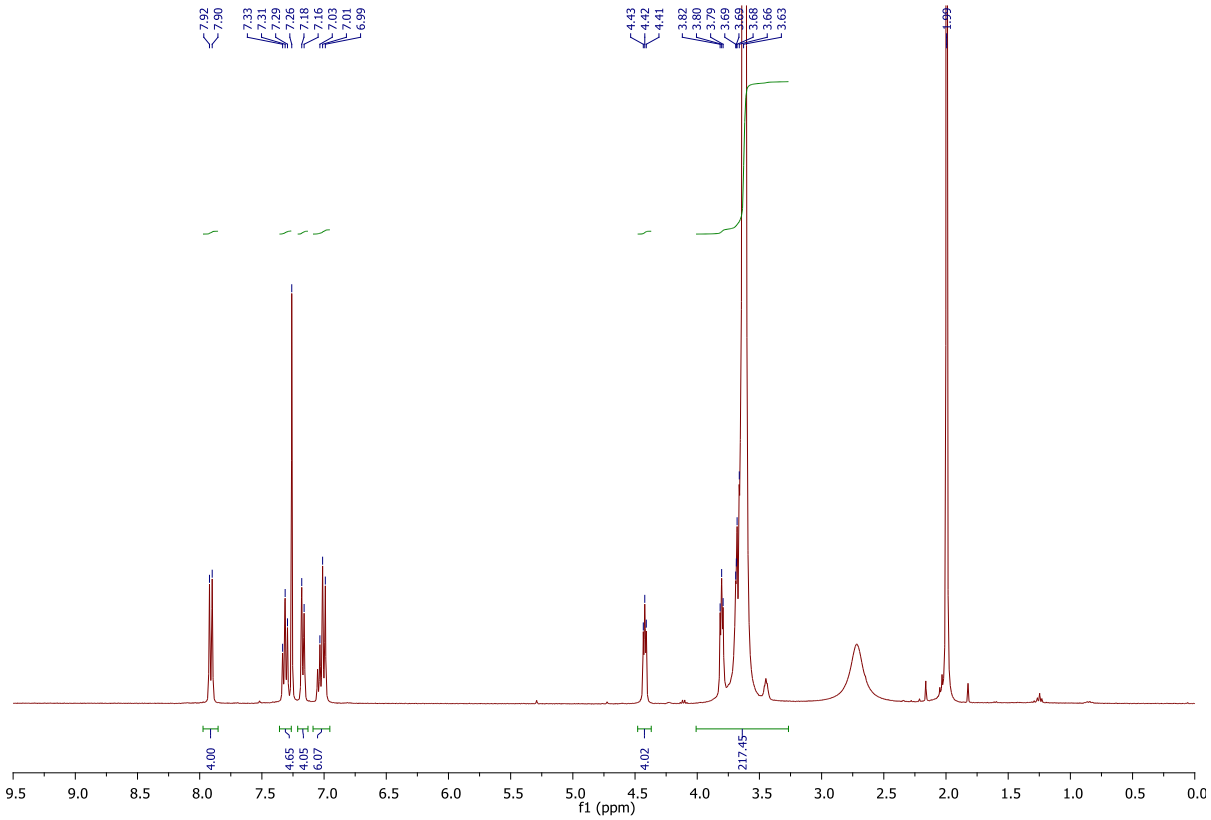

166.11
134.35
132.65
132.12
131.50
127.21
121.75
77.48
77.36
77.16
76.84
70.67
70.64
70.58
69.02
64.64
f1 (ppm)

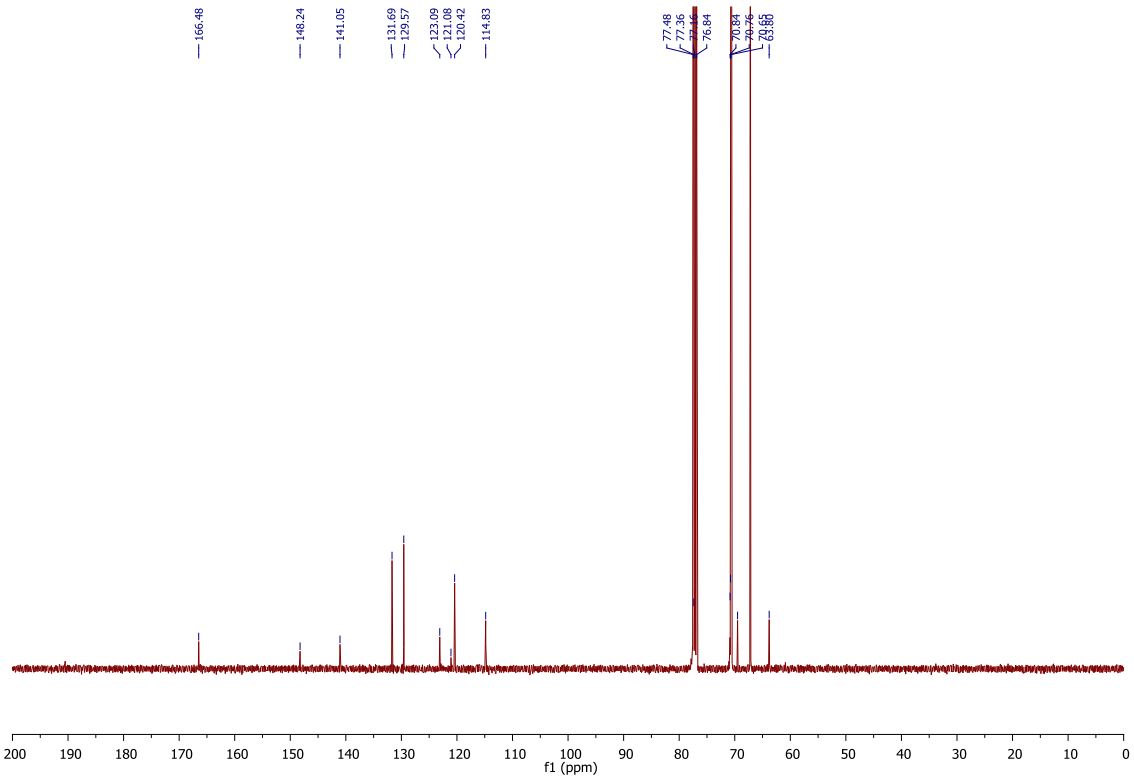

Cl
O
O
O
O
n
O
O
Cl

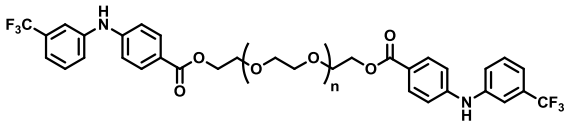

qoa-LC178
7.97
7.90
7.88
7.49
7.47
7.36
7.34
7.32
7.26
4.42
3.78
3.57
2.00
1.89
2.04
2.44
4.18
207.22
f1 (ppm)

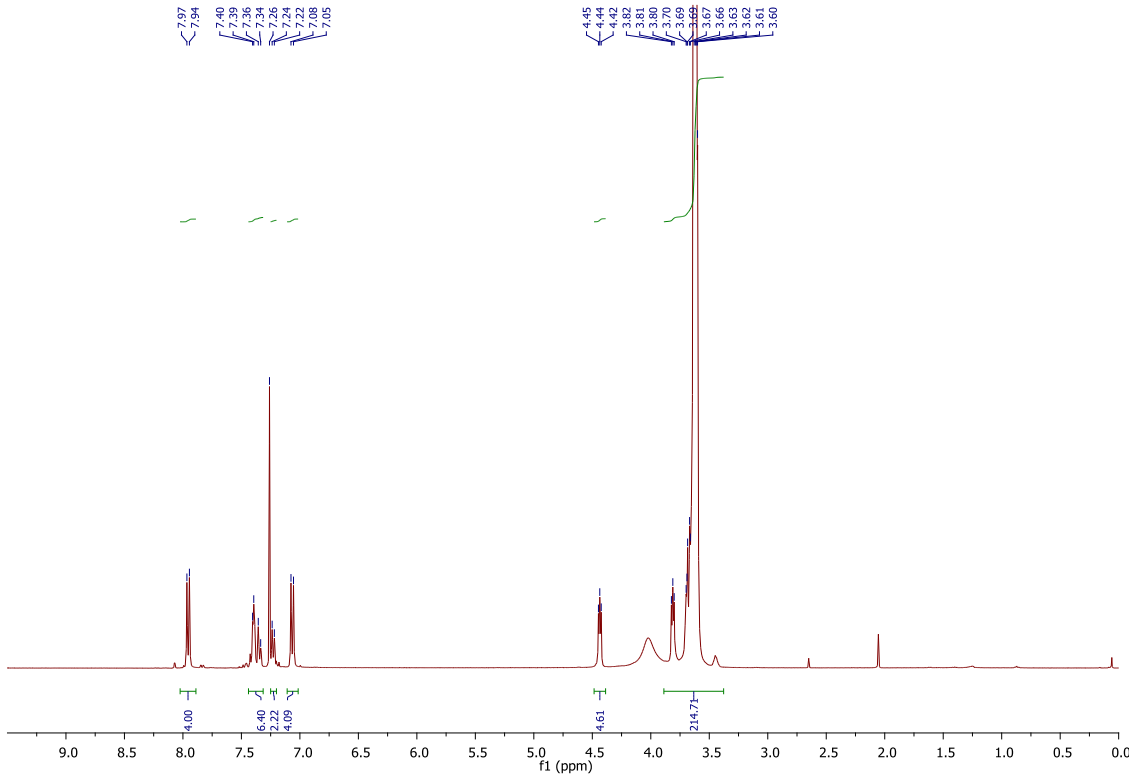

165.27
134.41
132.98
131.82
129.69
129.67
127.80
77.48
77.16
76.84
70.64
70.59
70.51
69.04
64.43
f1 (ppm)

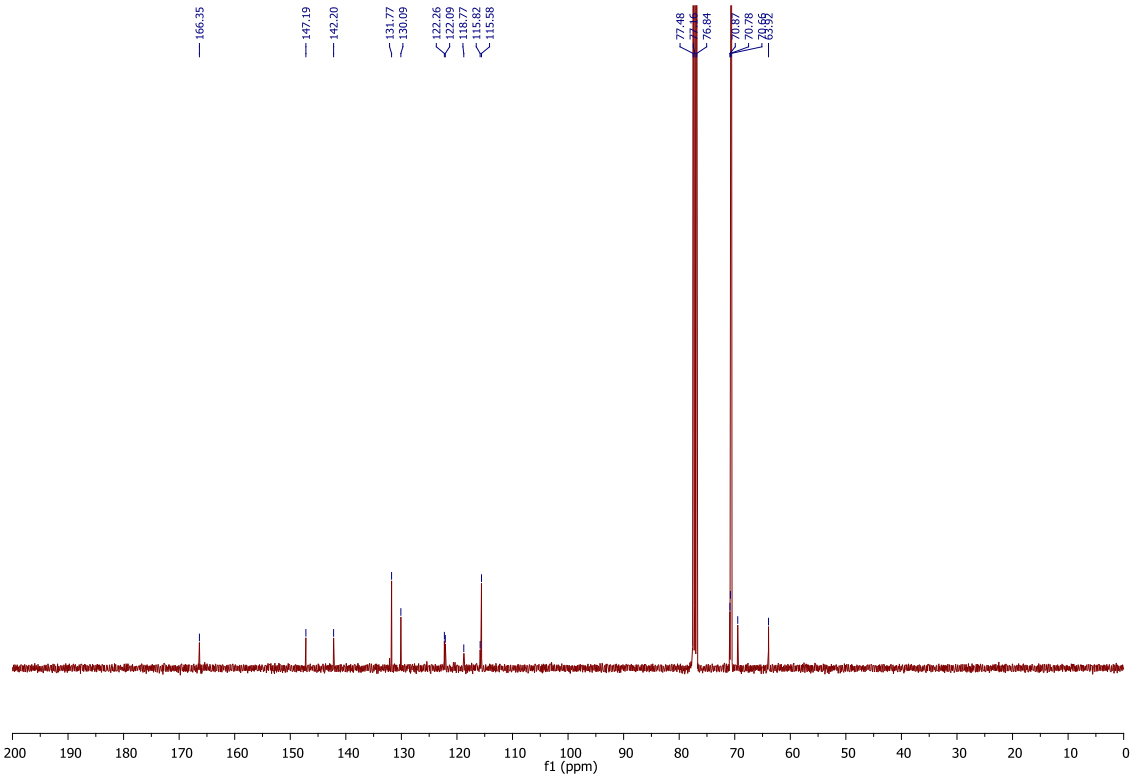


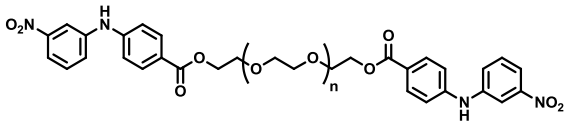


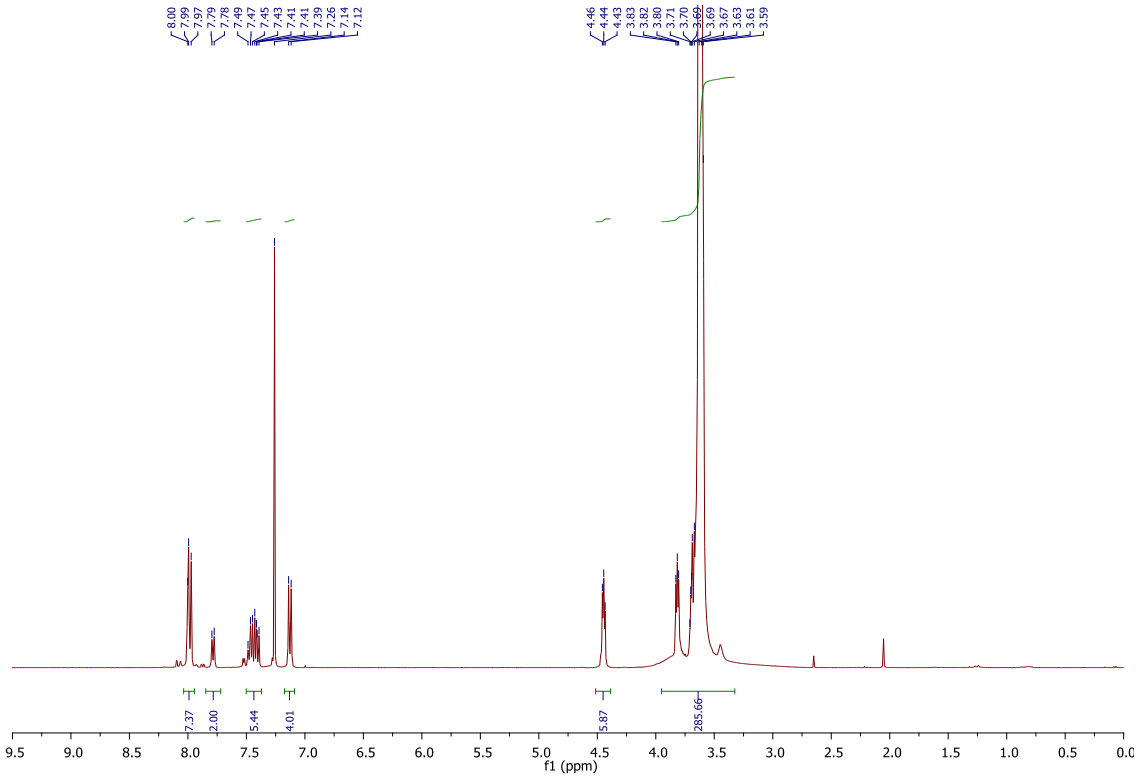


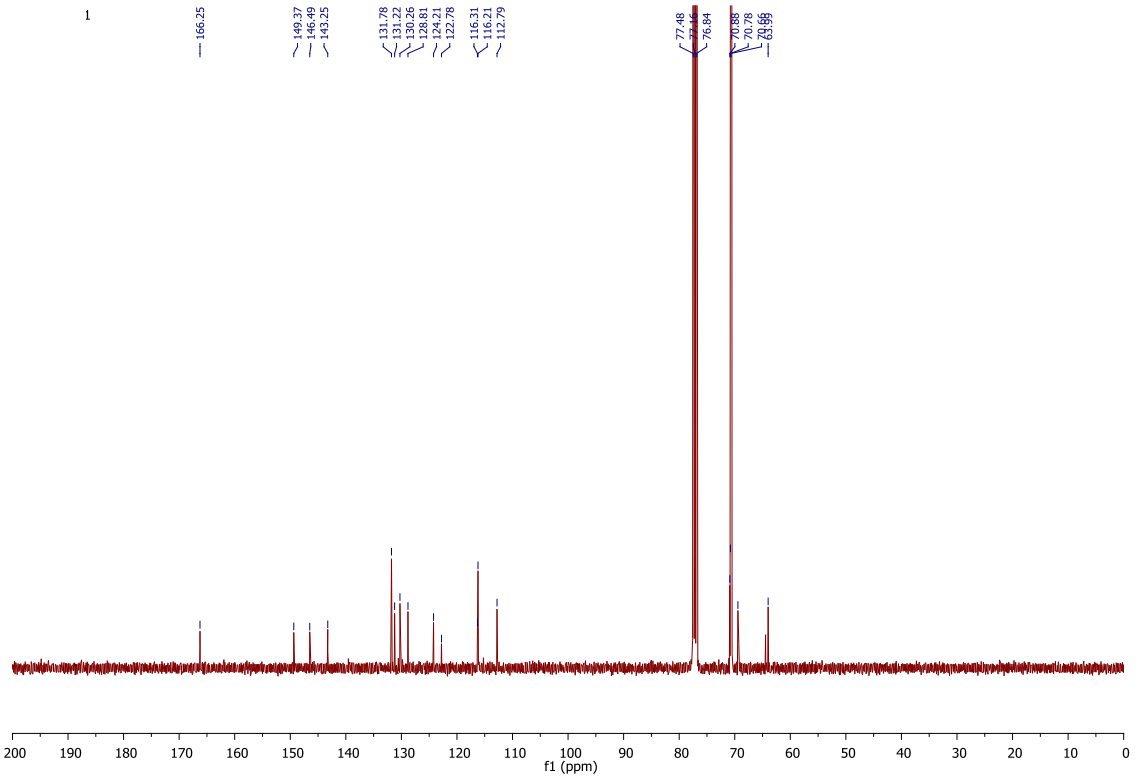

F3C
H
N
O
O
O
O
n
O
O
H
N
CF3

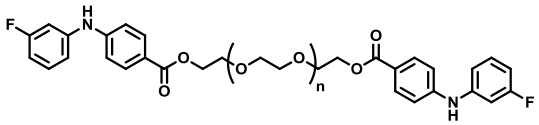

7.97
7.94
7.40
7.39
7.36
7.34
7.26
7.24
7.22
7.08
7.05
4.45
4.44
4.42
3.82
3.81
3.80
3.70
3.69
3.67
3.66
3.63
3.62
3.61
3.60
4.00
6.40
2.22
4.09
4.61
214.71
f1 (ppm)

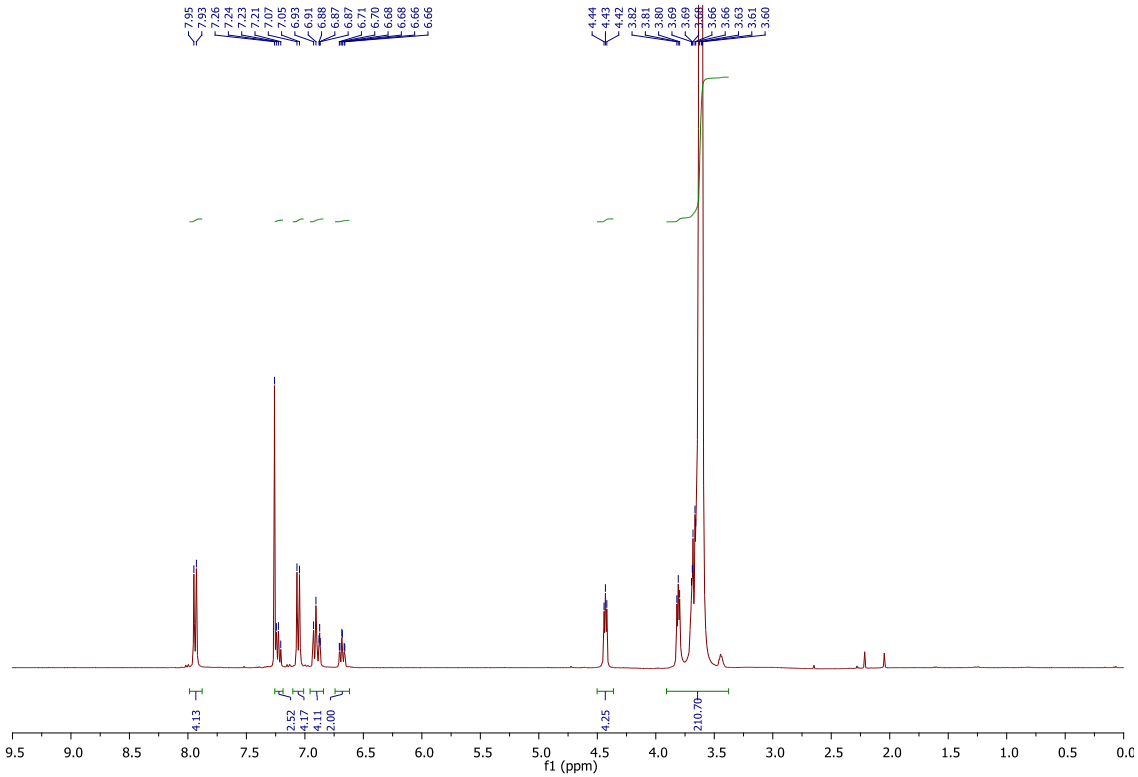

166.35
147.19
142.20
131.77
130.09
122.26
122.09
118.77
115.82
115.58
77.48
77.16
76.84
70.87
70.78
63.92
f1 (ppm)

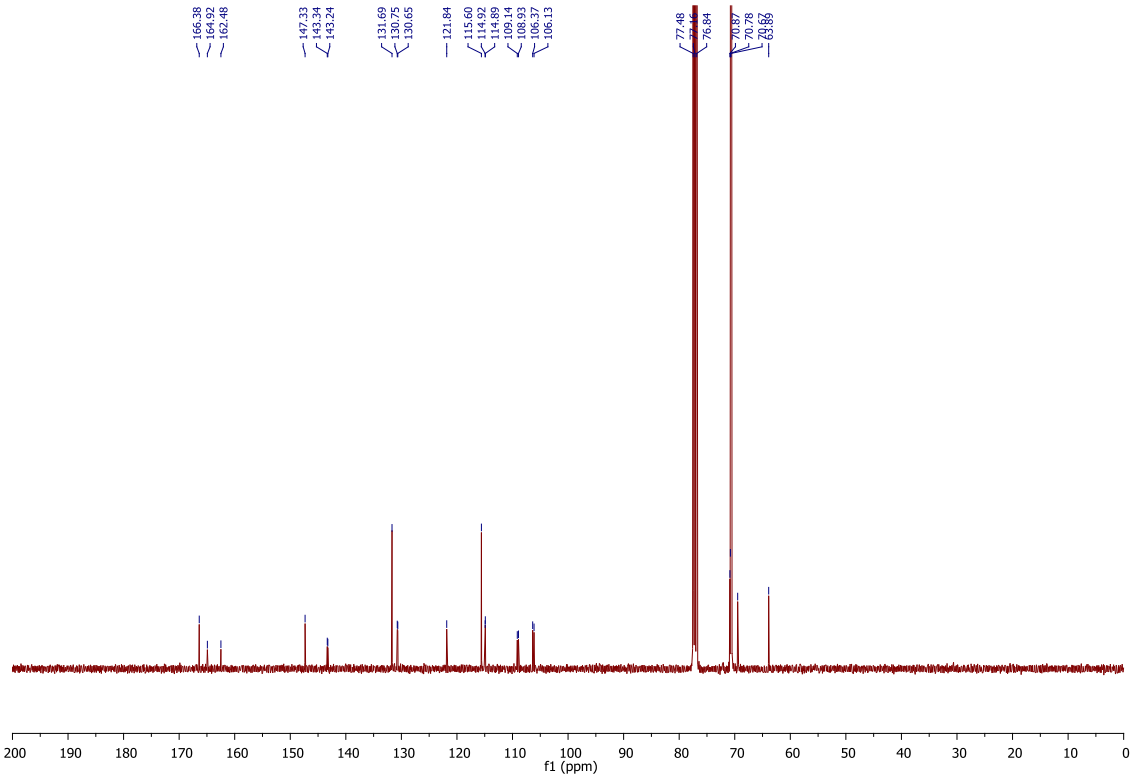


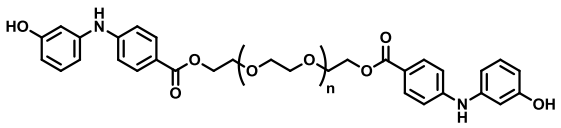


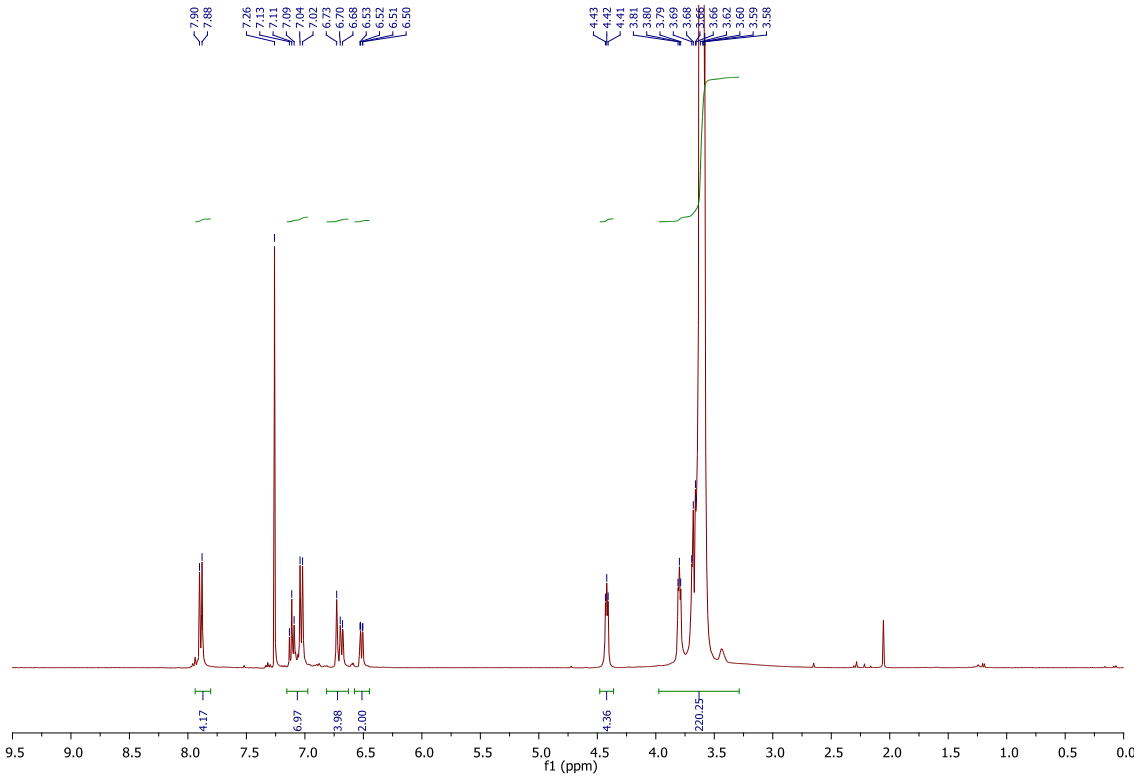





7.95
7.93
7.26
7.24
7.23
7.21
7.07
7.05
6.93
6.91
6.88
6.87
6.71
6.70
6.68
6.68
6.66
6.66
4.44
4.43
4.42
3.82
3.81
3.80
3.69
3.69
3.68
3.65
3.66
3.63
3.61
3.60
4.13
2.52
4.17
4.11
2.00
4.25
210.70
f1 (ppm)


166.38
164.92
162.48
147.33
143.34
143.24
131.69
130.75
130.65
121.84
115.60
114.92
114.89
109.14
108.93
106.37
106.13
77.48
77.16
76.84
70.87
70.78
63.89
f1 (ppm)


HO
H
N
O
O
O
O
n
O
O
N
H
OH


7.90
7.88
7.26
7.13
7.11
7.09
7.04
7.02
6.73
6.70
6.68
6.53
6.52
6.51
6.50
4.43
4.42
4.41
3.81
3.80
3.79
3.69
3.68
3.66
3.62
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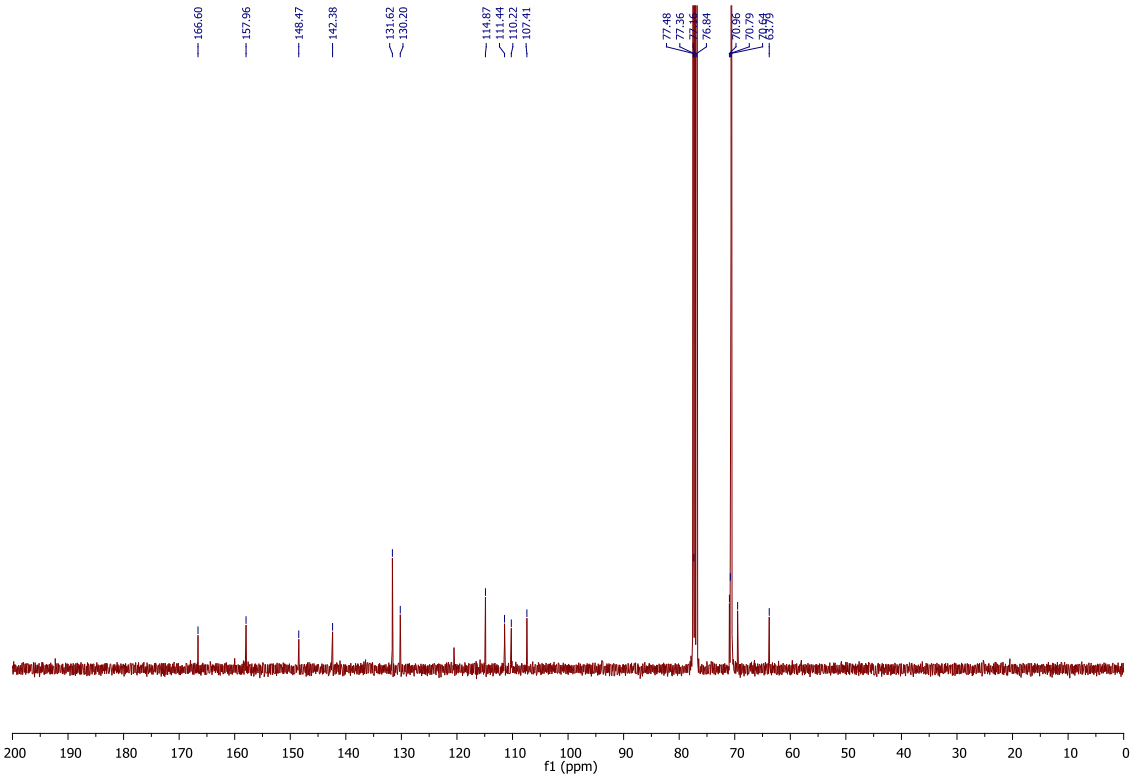
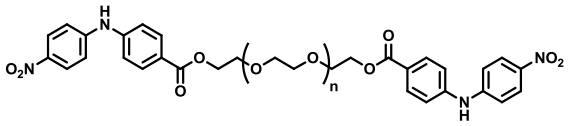
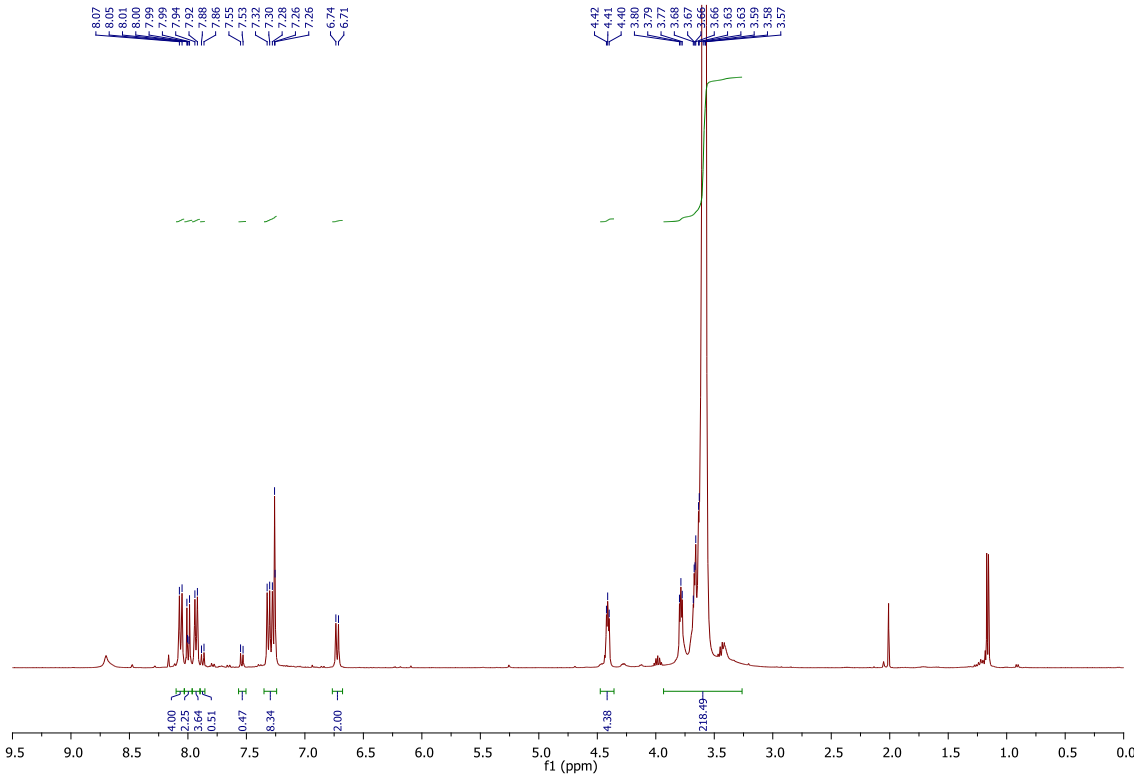
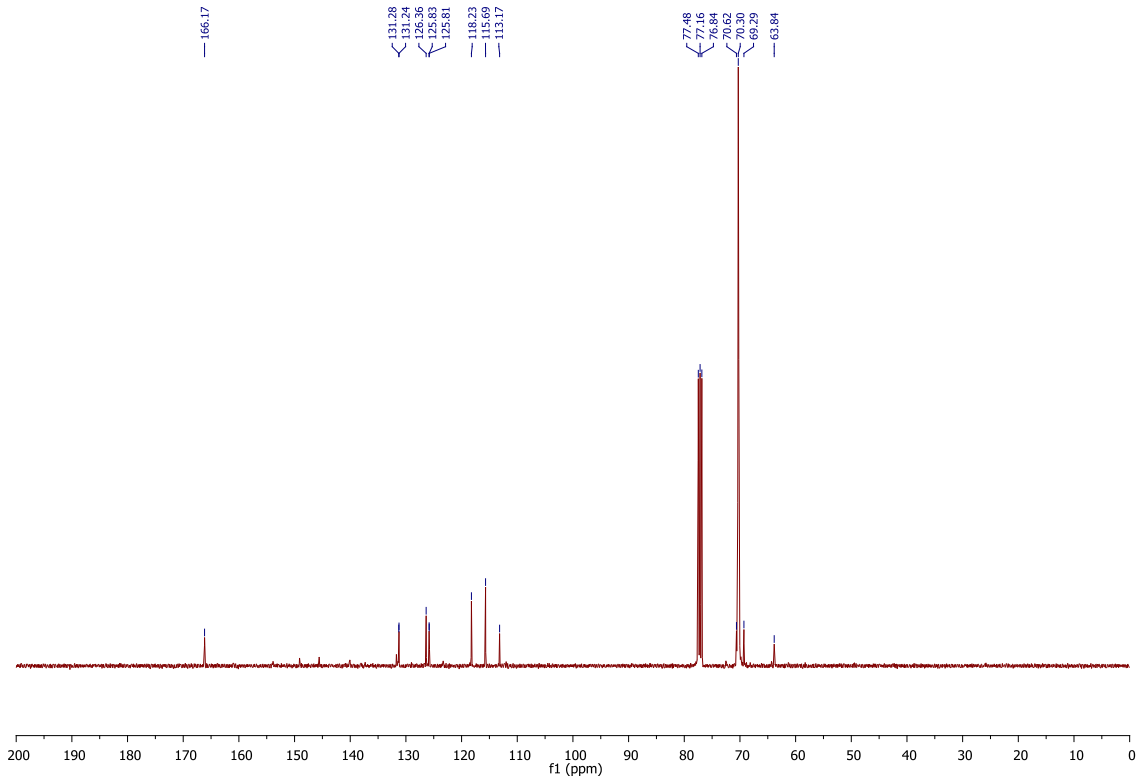
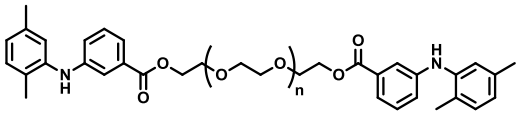
3.98
2.00
4.36
220.25
f1 (ppm)

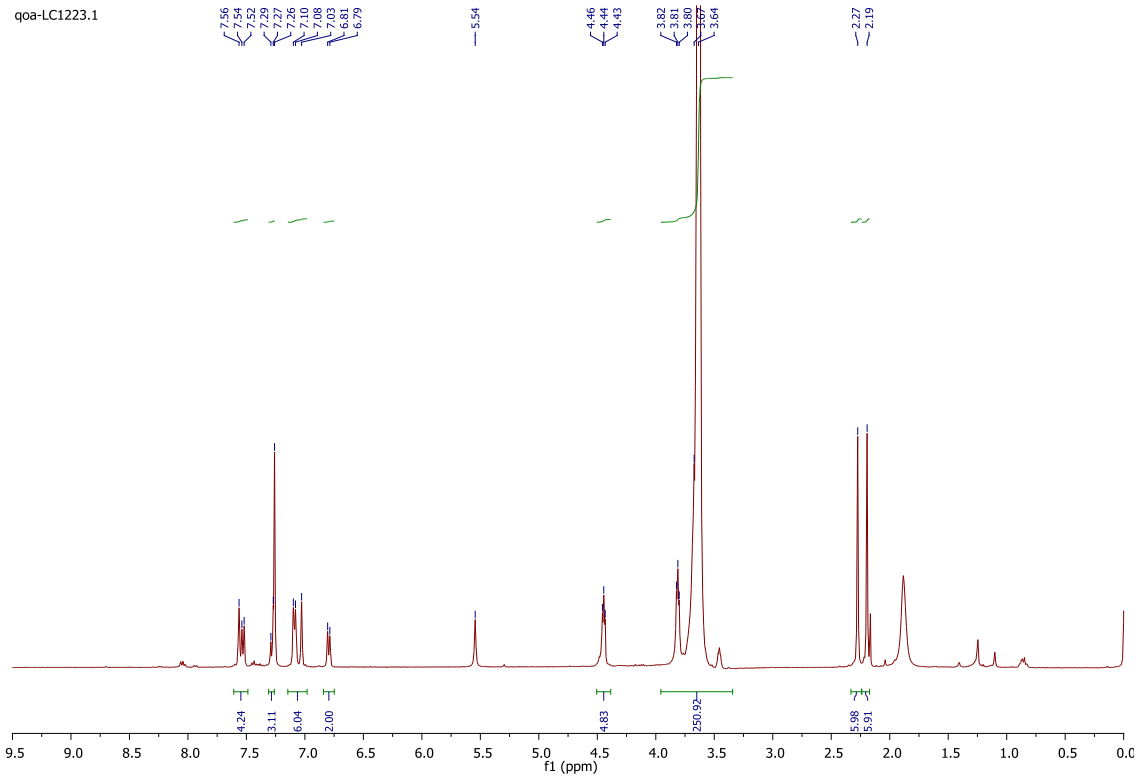

166.60
157.96
148.47
142.38
131.62
130.20
114.87
111.44
110.22
107.41
77.48
77.36
77.16
76.84
70.96
70.79
70.64
63.79
f1 (ppm)

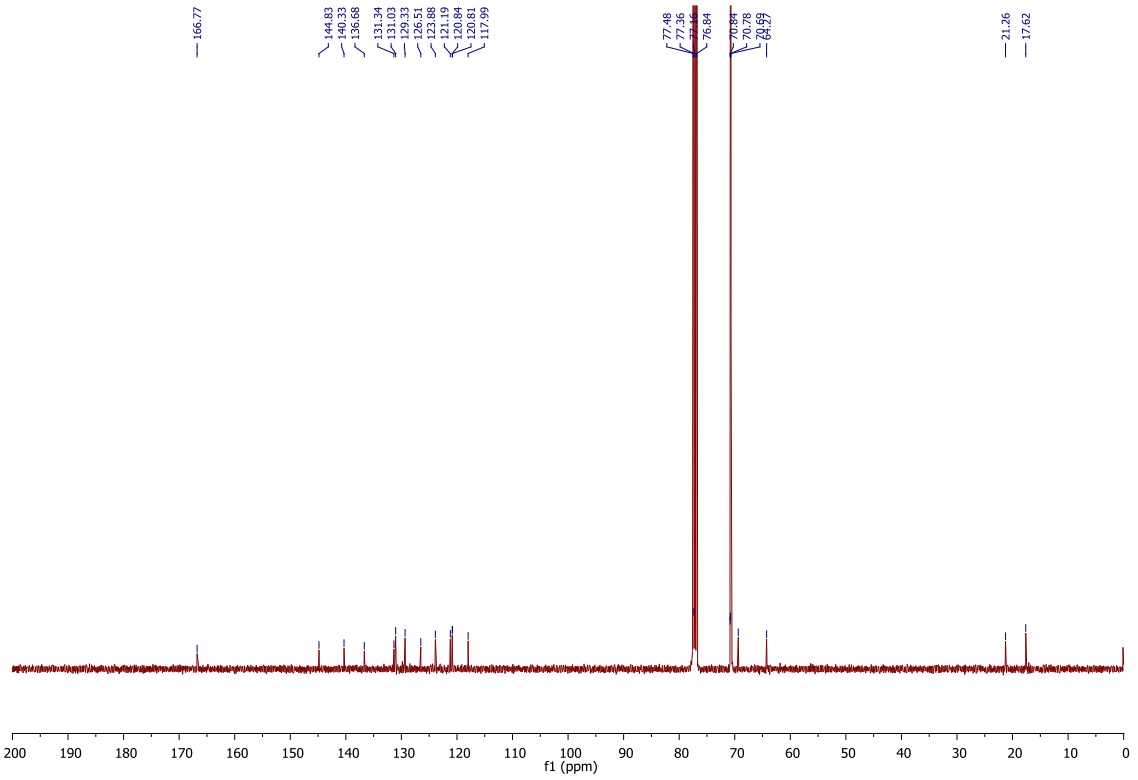









qoa-LC1223.1
7.56
7.54
7.52
7.29
7.27
7.26
7.10
7.08
7.03
6.81
6.79
5.54
4.46
4.44
4.43
3.82
3.81
3.80
3.67
3.64
2.27
2.19
4.24
3.11
6.04
2.00
4.83
250.92
5.98
5.91
f1 (ppm)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140.33
136.68
131.34
131.03
129.33
126.51
123.88
121.19
120.84
120.81
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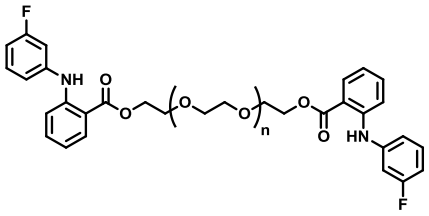
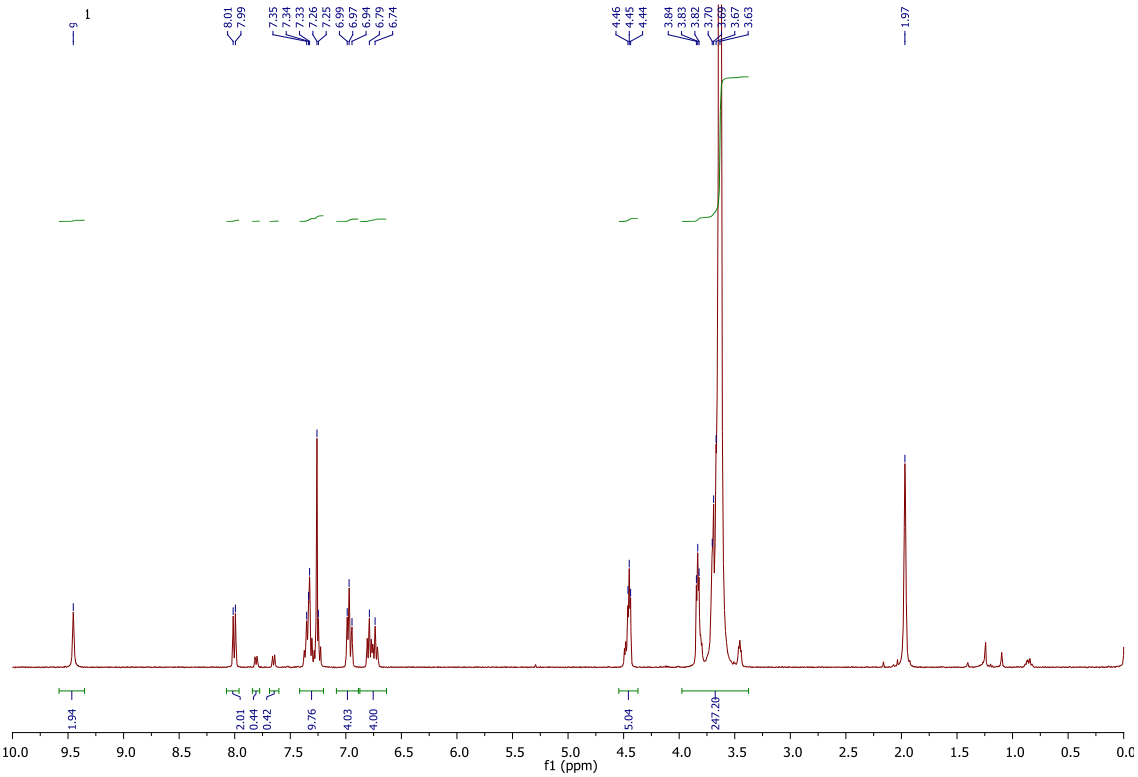
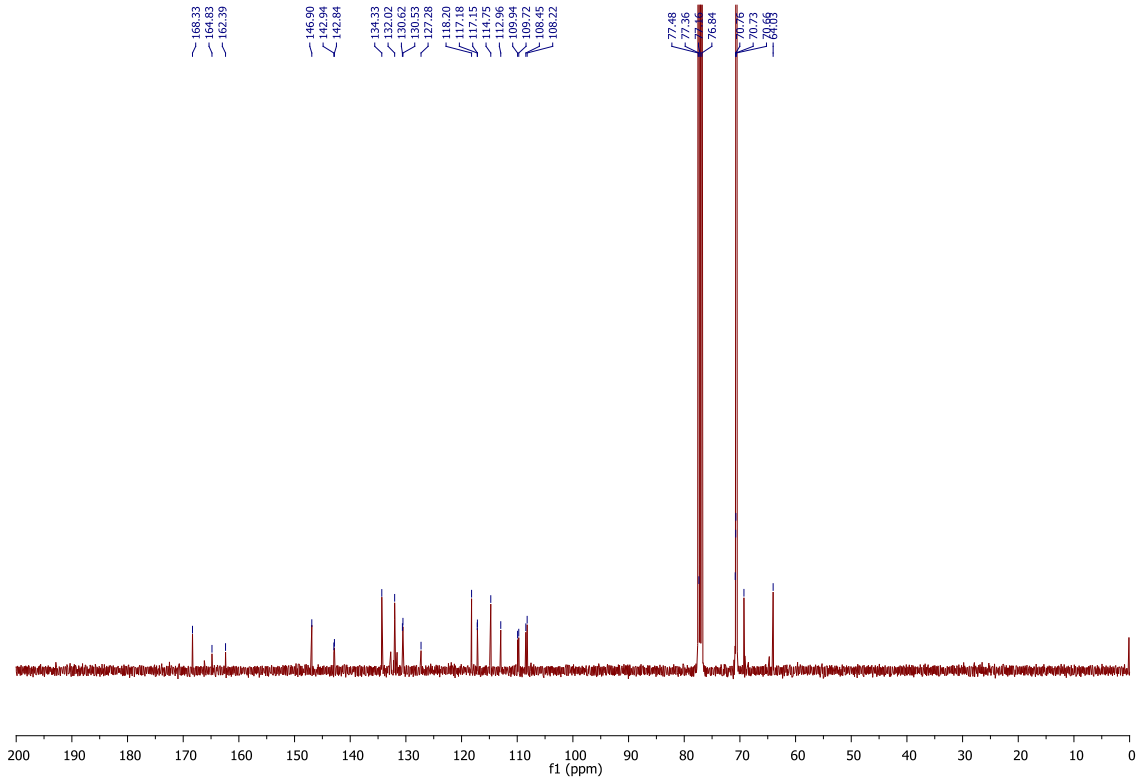
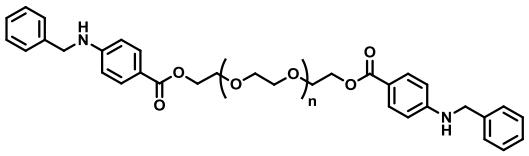
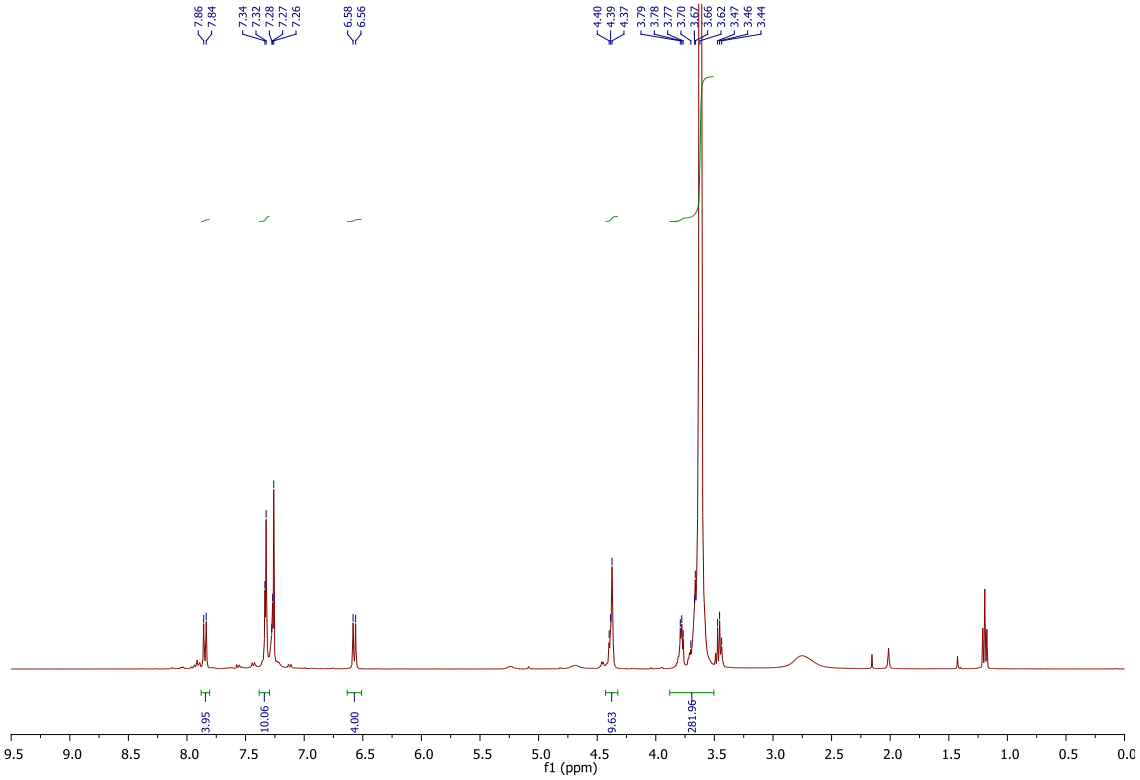
70.59
64.27
21.26
17.62
f1 (ppm)


F
NH
O
O
O
O
O
n
O
O
HN
F


9
1
8.01
7.99
7.35
7.34
7.33
7.26
7.25
6.99
6.97
6.94
6.79
6.74
4.46
4.45
4.44
3.84
3.83
3.82
3.70
3.69
3.67
3.63
1.97
1.94
2.01
0.44
0.42
9.76
4.03
4.00
5.04
247.20
f1 (ppm)


168.33
164.83
162.39
146.90
142.94
142.84
134.33
132.02
130.62
130.53
127.28
118.20
117.18
117.15
114.75
112.96
109.94
109.72
108.45
108.22
77.48
77.36
77.16
76.84
70.76
70.73
70.66
64.03
f1 (ppm)

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7.28
7.27
7.26
6.58
6.56
4.40
4.39
4.37
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3.78
3.77
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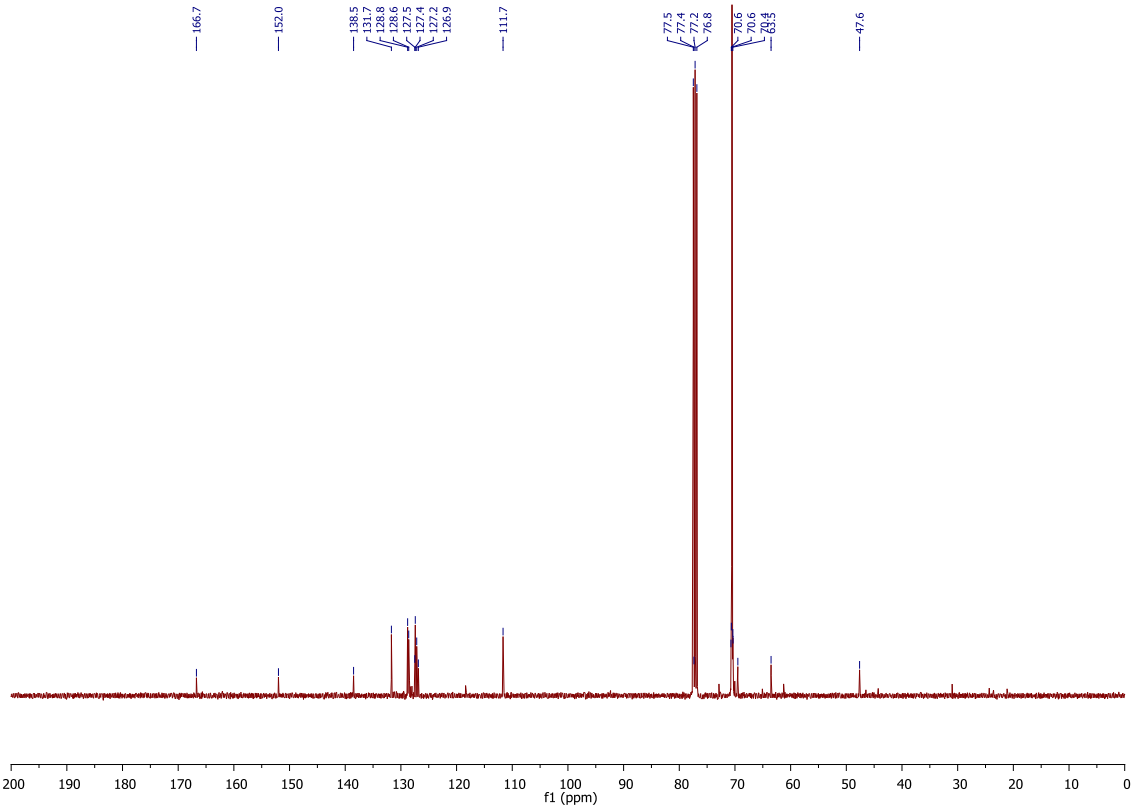
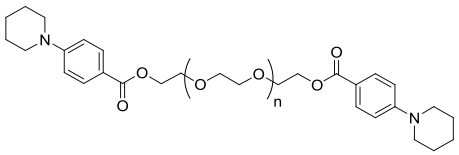
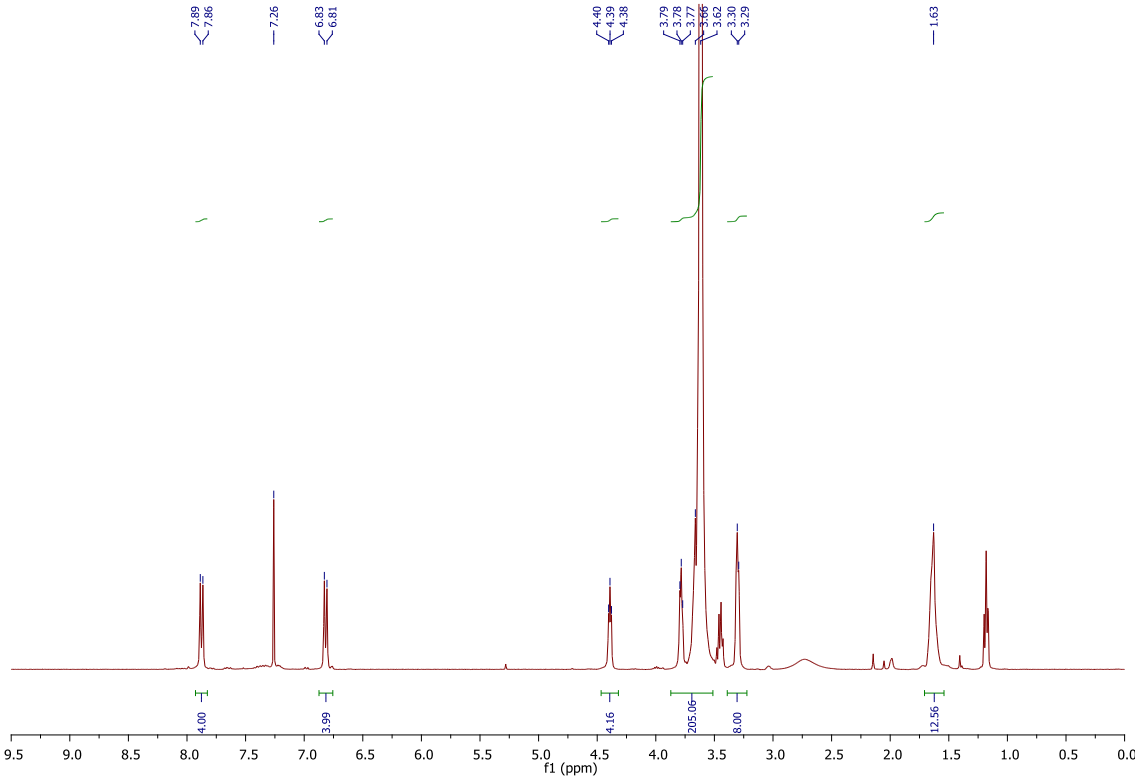
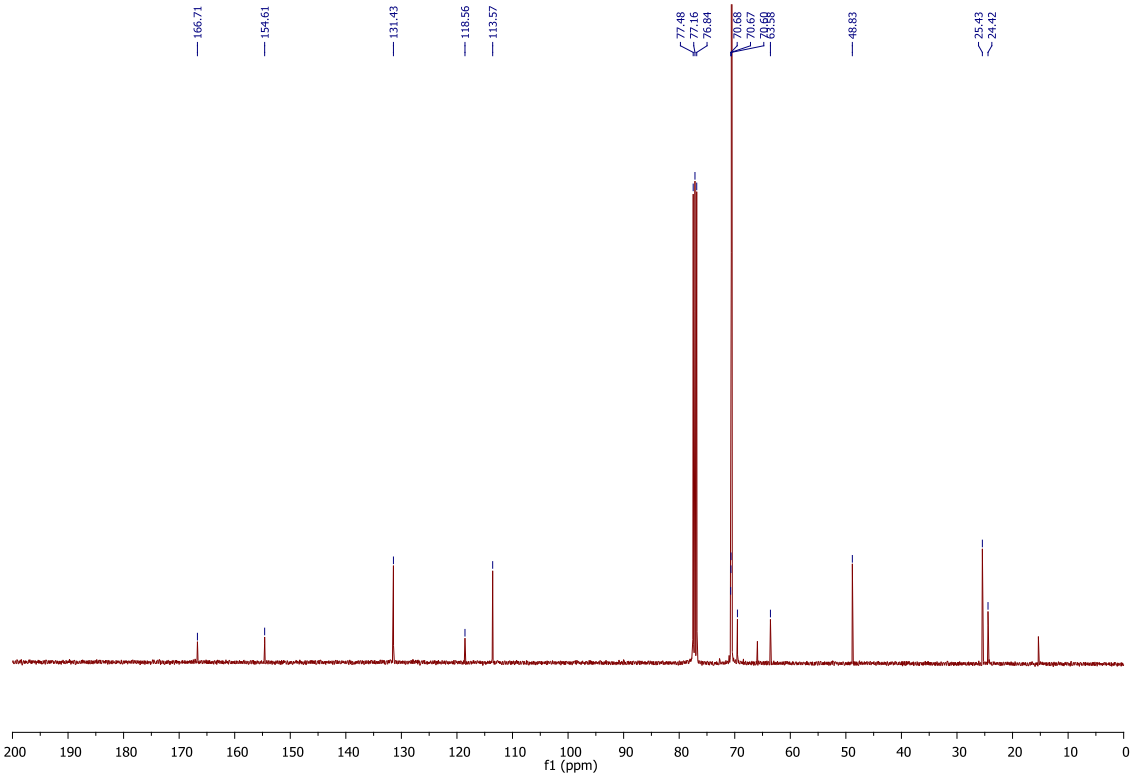
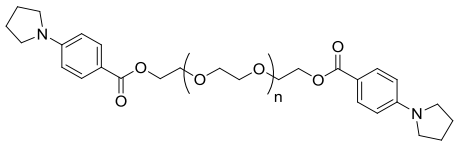
10.06
4.00
9.63
281.96
f1 (ppm)

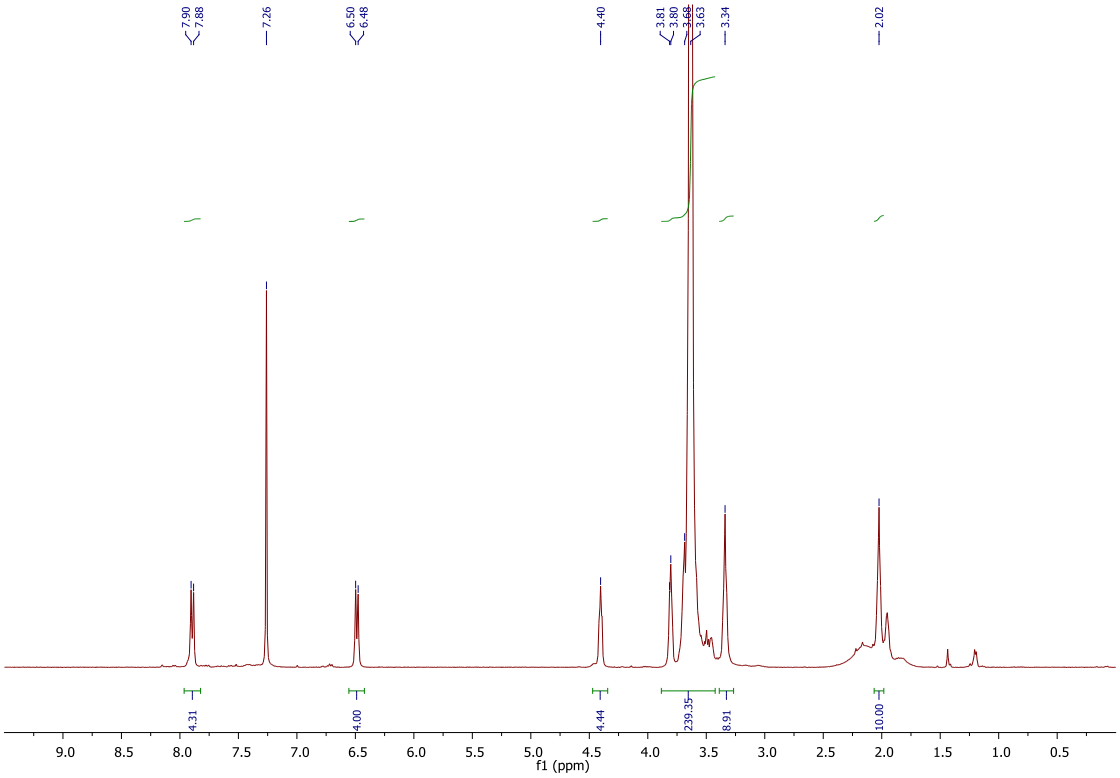

166.7
152.0
138.5
131.7
128.8
128.6
127.5
127.4
127.2
126.9
111.7
77.5
77.4
77.2
76.8
70.6
70.6
70.4
63.4
47.6
f1 (ppm)

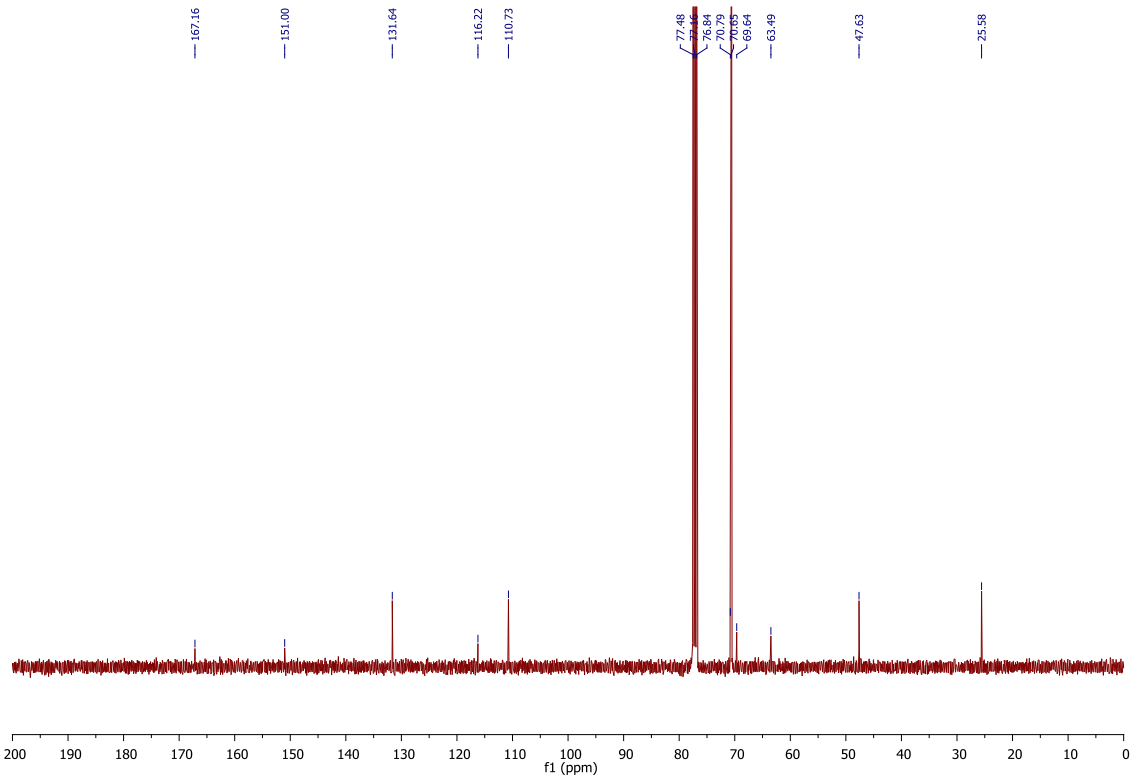


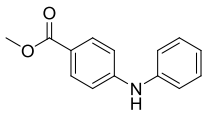


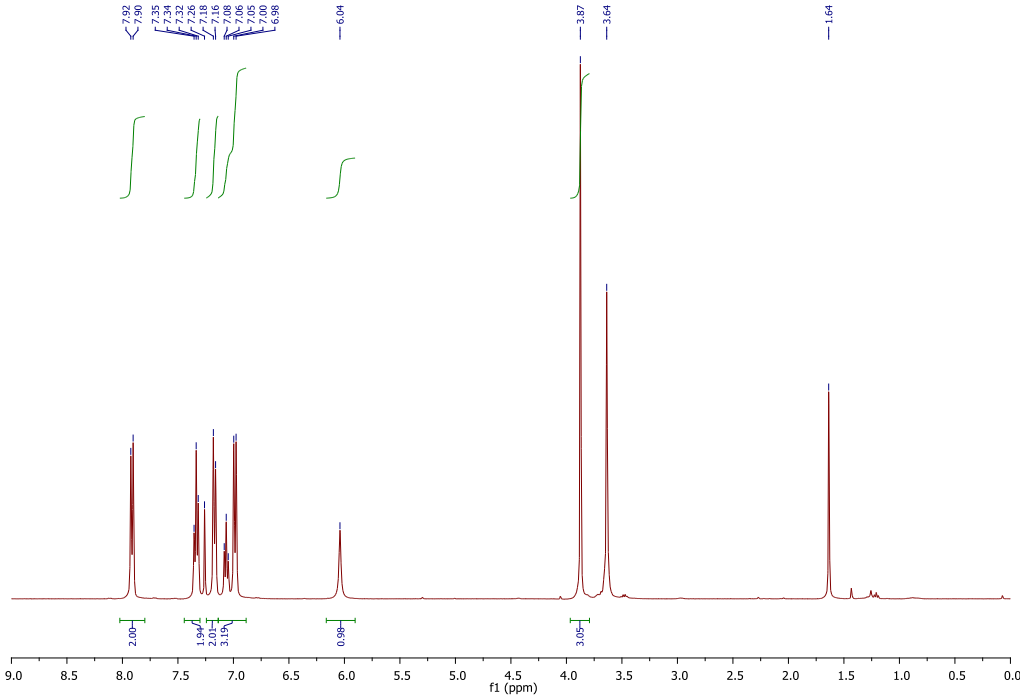


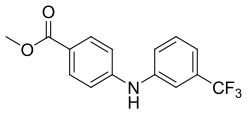


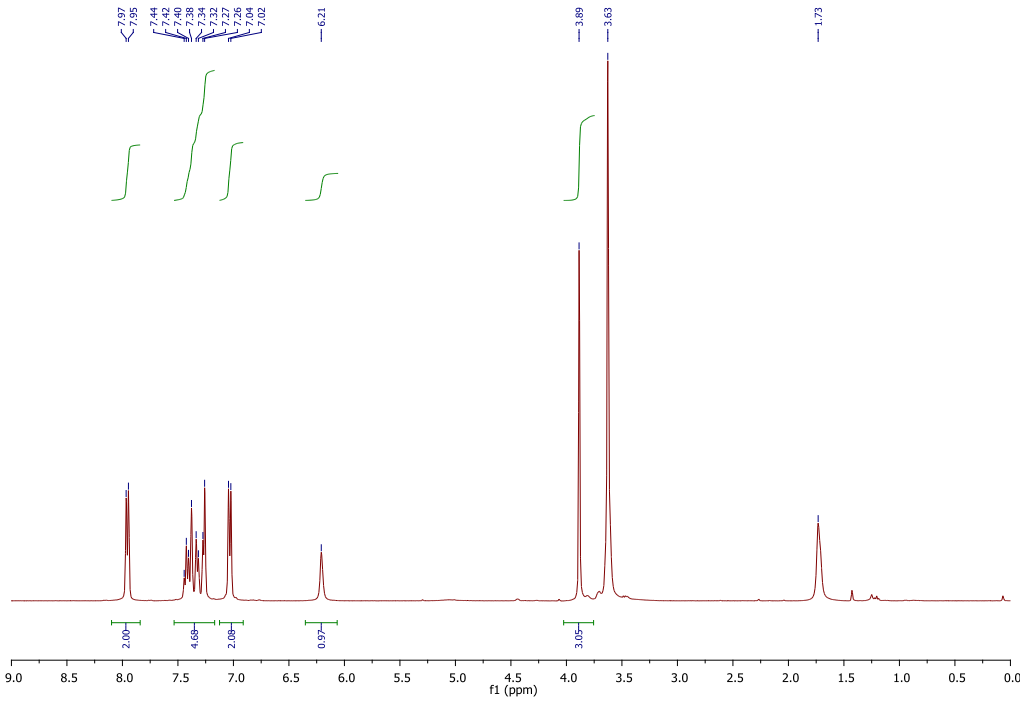


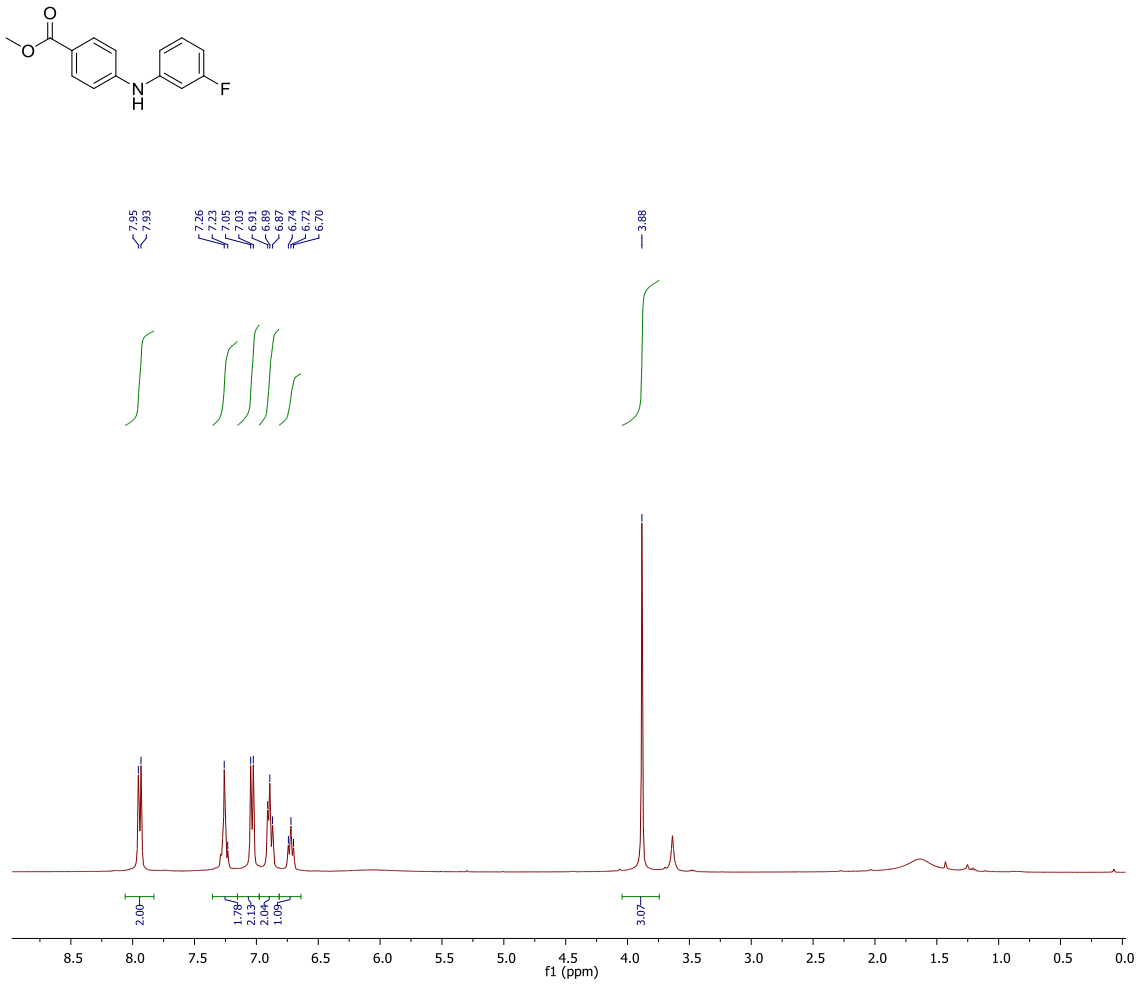












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