Supporting Information

A quick Chan-Lam C-N and C-S cross coupling at room temperature in presence of square pyramidal $[Cu(DMAP)_4I]I$ as catalyst

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CONTENTS

General:

All the chemicals used were of analytical grade. IR spectra were recorded by using Perkin Elmer Spectrum RX I FT-IR Spectrometer. ^{1}H and ^{13}C-NMR spectra were obtained in $CDCl_3$ using Bruker 300 MHz instrument. TGA and DSC data were recorded on a Mettler Toledo instrument. The X-Band Electron Paramagnetic Resonance (EPR) spectrum of the complex was recorded on a JES-FA200 ESR spectrometer.

General procedure for the synthesis of $[Cu(DMAP)_4I]I$ complex:

A Solution of CuI (4.0 mmol) in DMSO (5 mL) was stirred at room temperature. To this solution 4-(Dimethylamino)pyridine (DMAP) (16.4 mmol) was added and the mixture was allowed to stirred at room temperature (25 ºC) for 3 hours. Now the reaction mixture was filtered and the eluent is allowed to recrystallize. After two days pure bluish green crystal of $[Cu(DMAP)_4I]I$ separated out. The complex was characterized by UV-Vis spectroscopy, FT-IR, ESR analysis, DSC analysis and Single crystal XRD . DSC study shows that the compound is stable up to 131°C, UV-Vis spectroscopy shows peak at 644 nm, ESR spectram at room temperature shows the presence of X-band, which shows that in the complex copper atom is in +2 oxidation state and the complex has symmetry C_{4V}. Again single crystal X-ray diffraction studies shows that the complex is square pyramidal in shape, where four DMAP ligand occupied the equatorial positions and one iodine atom occupied the axial position and another iodine atom is in free state and outside the coordination sphere.

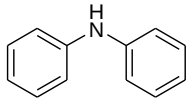
General procedure for Chan-Lam coupling:

To a stirred solution of aryl boronic acid (1.0 mmol), substrate (amine, amide or thiol; 1.0 mmol) in 2 ml MeOH was added $[Cu(DMAP)_4I]I$ complex (0.002 mmol, 2 mol %) under air atmosphere. The reaction mixture was stirred at room temperature for required time until its total conversion (monitored by TLC). After completion of reaction, the reaction mixture was filtered and the filtrate part was evaporated under vacuum to get the crude product. The crude was purified by flash column chromatography in 230-400 silica mesh using ethyl acetate / petroleum ether as eluent.

In case of morpholine, 1.2 equivalent of boronic acid was used. Moreover, the crude produce was re dissolved in ethyl acetate and washed with saturated $NaHCO_3$ solution (3ml x 3). Finally the organic part was dried over Na_2SO_4 and evaporated under vacuum to get the crude product. The crude was purified by flash column chromatography in 230-400 silica mesh using ethyl acetate / petroleum ether as eluent.

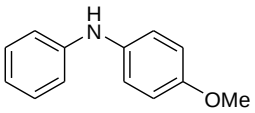
SPECTRAL DATA:

***N*-phenylaniline (1a)** [1]:



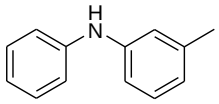
White solid (0.147 g, 87% yield); IR (KBr, cm^{-1}): υ 3357, 3013, 1611, 1527, 1486, 1325, 755; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.32-7.27 (m, 4H), 7.10 (d, *J* = 6 Hz, 4H), 6.98-6.93 (m, 2H) , 5.73 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 143, 129.3, 120.9, 117.7

4-methoxy-*N*-phenylaniline (1b) [1]:



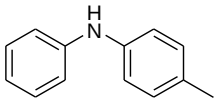
Yellow oil (0.162 g, 81% yield); IR (KBr, cm^{-1}): υ 3376, 3012, 2944, 2846, 2544, 1825, 1583, 1524, 1411, 1230, 1121, 1023, 766; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.28-7.25 (m, 2H), 7.1 (d, *J* = 9 Hz, 2H), 6.96-6.88 (m, 5H), 5.54 (s, 1H), 3.83 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 155.1, 145.1, 135.6, 129.2, 122.1, 119.5, 115.5, 114.6, 55.5.

3-methyl-*N*-phenylaniline, 1c [2]:



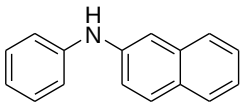
Yellow oil (0.150 g, 82% yield); IR (KBr, cm^{-1}): υ 3387, 3024, 2933, 2867, 2371, 1930, 1596, 1379, 1331, 1190, 1012, 878, 742; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.3-7.25 (m, 2H), 7.16 - 7.06 (m, 3H), 6.95-6.89 (m, 3H), 6.77 (d, *J* = 6 Hz, 1H), 5.67 (br, 1H), 2.32 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.2, 143, 129.3, 129.1, 120.8, 118.4, 117.8, 114.9, 29.7.

4-methyl-*N*-phenylaniline, 1d [1]:



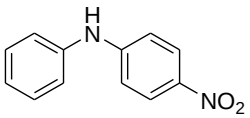
Yellow oil (0.156 g, 85% yield); IR (KBr, cm^{-1}): υ 3387, 3022, 2345, 1591, 1388, 1100, 868, 734; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.25 (t, *J* = 6 Hz, 2H), 7.09-7.03 (m, 6H), 6.90 (d, *J* = 6 Hz, 1H), 5.67 (br, 1H), 2.32 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.9, 140.2, 130.8, 129.8, 129.3, 120.2, 118.8, 116.8, 29.7.

***N*-phenylnaphthalen-2-amine,** 1e [2]:



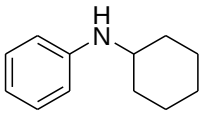
White solid (0.153 g, 70% yield); IR (KBr, cm^{-1}): υ 3375, 2932, 2849, 1589, 1497, 1310, 732; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.76 (d, *J* = 8.7 Hz, 2H), 7.66 (d, *J* = 7.8 Hz, 1H), 7.49 (s, 1H), 7.42 (t, *J* = 7.5 Hz, 1H), 7.36-7.30 (m, 3H), 7.25-7.19 (m, 3H), 7.01 (t, *J* =7.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 147.0, 140.3, 134.6, 130.4, 129.8, 127.7, 126.5, 123.5, 122.5, 119.7, 117.8.

4-nitro-*N*-phenylaniline, 1f [17]:



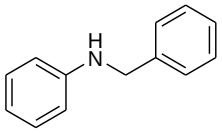
Yellow solid (0.188 g, 88% yield); IR (KBr, cm^{-1}): υ 3317, 3042, 1577, 1292; ^1H NMR (300 MHz, $CDCl_3$): δ 8.11 (d, *J* = 8.5 Hz, 2H), 7.42-7.39 (m, 2H), 7.26-7.14 (m, 3H), 6.96 (d, *J* = 8.7 Hz, 2H), 5.86 (br, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 150.2; 139.6, 139.4, 129.7, 126.2, 124.6, 121.9, 113.6.

***N*-cyclohexylaniline,** 1g [15]:



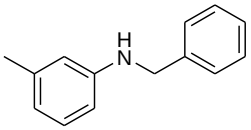
Colorless oil (0.131 g, 75% yield); IR (KBr, cm^{-1}): υ 3385, 3038, 2933, 2852, 1907, 1738, 1597, 1519, 1447, 1327, 1249, 1169, 1142, 1123, 882, 742, 697; ^1H NMR (300 MHz, $CDCl_3$): δ 7.18 (t, *J* = 7.8 Hz, 2H), 6.73-6.64 (m, 3H), 3.31-3.22 (m, 1H), 2.07 (d, *J* = 12.3 Hz, 2H), 1.81-1.75 (m, 2H), 1.68-1.63 (m, 1H), 1.44-1.11 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 146.2, 129.1, 117.9, 114.1, 52.6, 33.1, 25.8, 25.

***N*-benzylaniline,** 1h [1]:



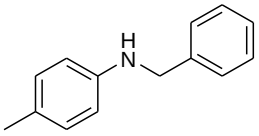
Colorless oil (0.152 g, 83% yield); IR (KBr, cm^{-1}): υ 3450, 3087, 3010, 1624, 1500, 1388, 694; ^1H NMR (300 MHz, $CDCl_3$): δ 7.41-7.32 (m, 5H), 7.25-7.20 (m, 2H), 6.79-6.74 (m, 1H), 6.68 (d, *J* = 9 Hz, 2H), 4.37 (s, 2H), 4.06 (br, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 148.1, 139.4, 129.2, 128.6, 127.4, 127.2, 117.5, 112.8, 48.2.

***N*-benzyl-3-methylaniline,** 1i [11]:



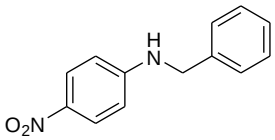
Colorless oil (0.160 g, 81% yield); IR (KBr, cm^{-1}): υ 3417, 3022, 2929, 2854, 1610, 1525, 1467, 1341, 1234, 1171, 801, 736; ^1H NMR (300 MHz, $CDCl_3$): δ 7.37-7.27 (m, 5H), 7.10-7.05 (m, 1H), 6.56 (d, *J* = 6 Hz, 1H) , 6.49-6.45 (m, 2H), 4.33 (s, 2H), 2.28 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 148.2, 139.5, 139.0, 129.8, 129.1, 128.6, 128.2, 127.5, 127.2, 118.5, 113.6, 109.9, 48.3, 21.2.

***N*-benzyl-4-methylaniline,** 1j [3]:



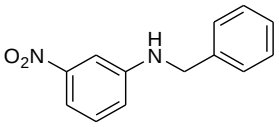
Yellow Oil (0.158 g, 80% yield); IR (KBr, cm^{-1}): υ 3412, 3068, 3011, 1535, 1397, 713; ^1H NMR (300 MHz, $CDCl_3$): δ 7.36-7.34 (d, *J* = 6 Hz, 5H), 6.99 (d, *J* = 6 Hz, 2H), 6.57 (d, *J* = 9 Hz, 2H), 4.32 (s, 2H), 2.24(s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 145.8, 139.6, 136.3, 134.4, 129.8, 129.7, 128.9, 128.2, 127.4, 127, 126.6, 112.9, 48.5, 30.8.

***N*-benzyl-4-nitroaniline,** 1k [3]:



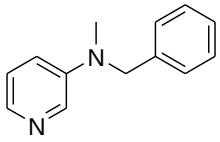
Yellow Solid (0.189 g, 83% yield); IR (KBr, cm^{-1}): υ 3363, 3062, 3024, 2924, 1604, 1539, 1469, 1280, 1103, 833, 748; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.08 (d, *J* = 9Hz, 2H), 7.35-7.33(m, 5H), 6.58 (d, *J* = 9Hz, 2H), 4.93 (br, 1H), 4.44-4.43 (m, 2H); ^{13}C NMR(75 MHz, $CDCl_3$): δ 153, 138.2, 137.3, 128.9, 127.8, 127.3, 126.4, 111.3, 47.6.

***N*-benzyl-3-nitroaniline,** 1l [3]:



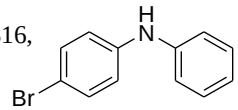
Yellow Solid (0.178 g, 78% yield); IR (KBr, cm^{-1}): υ 3378, 3078, 3012, 1613, 1478, 1200, 1110, 825; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.54 (d, *J* = 6 Hz, 1H), 7.45 (br, 1H), 7.38-7.24 (m, 6H), 6.88 (d, *J* = 9 Hz, 1H), 4.40 (s, 2H); ^{13}C NMR(75 MHz, $CDCl_3$): δ 148.8, 138.1, 129.8, 128.9, 127.7, 127.5, 118.8, 112.1, 106.6, 48.

***N*-benzyl-*N*-methylpyridin-3-amine,** 1m [24]:



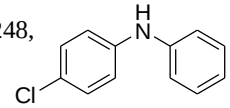
Light yellow Solid (0.133 g, 67%)); IR (KBr, cm^{-1}):υ 3019, 2890, 1578, 1483, 1447, 1367, 1342, 1232, 1047, 1012, 945, 927, 724, 705, 685; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.56-7.27 (m, 6H), 7.24-7.18 (m, 3H), 7.06 (d, *J* = 6.9 Hz, 1H), 4.58 (s, 2H), 3.10 (s, 3H) ^{13}C NMR(75 MHz, $CDCl_3$): δ 162.8, 137.1, 134.6, 131.7, 130.5, 128.9, 127.7, 127.6, 126.7, 124.7, 121, 56.3, 33.9

4-bromo-*N*-phenylaniline, 1n [21]:



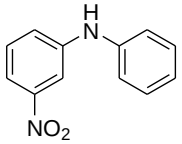
Light yellow Solid (0.198 g, 80% yield); IR (KBr, cm^{-1}):υ 3395, 3051, 3019, 1582, 1502, 1482, 1316, 1065, 781; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.37-7.27 (m, 4H), 7.07 (d, *J* = 8.4 Hz, 2H), 7.0-6.91 (m, 3H); ^{13}C NMR(75 MHz, $CDCl_3$): δ 142.4, 132.2, 129.5, 121.7, 119, 118.3, 112.6.

4-chloro-*N*-phenylaniline, 1o [21]:



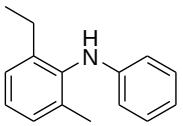
Light yellow Solid (0.166 g, 82%); IR (KBr, cm^{-1}):υ 3401, 3045, 3027, 1593, 1492, 1478, 1315, 1248, 1165, 1030, 773; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.39-7.22 (m, 4H), 7.16-6.93 (m, 5H)) ^{13}C NMR(75 MHz, $CDCl_3$): δ 142.6, 141.8, 129.7, 129.3, 125.5, 121.5, 118.9, 117.6

3-nitro-*N*-phenylaniline, 1p [25]:



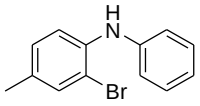
Red Solid (0.161 g, 75% yield); IR (KBr, cm^{-1}):υ 3399, 3047, 3025, 1598, 1532, 1479, 1325, 1255, 1176; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.40-7.24 (m, 4H), 7.17-7.07 (m, 1H), 6.95 (t, *J* = 7.5 Hz, 1H), 6.86 (d, *J* = 7.8 Hz, 2H) ^{13}C NMR(75 MHz, $CDCl_3$): δ 155.6, 145.1, 141, 129.7, 129.6, 123.2, 121.9, 120.7, 114.7, 110.3

2-ethyl-6-methyl-*N*-phenylaniline, 1q:



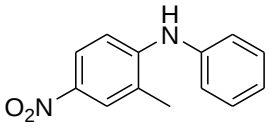
Light yellow gum (0.143 g, 68% yield); IR (KBr, cm^{-1}):υ 3397, 3065, 3031, 2927, 2875, 1582, 1039, 765; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.39-7.25 (m, 1H), 7.20-6.92 (m, 6H), 6.76 (t, *J* = 7.2 Hz, 1H), 6.52 (d, *J* = 8.1 Hz, 2H), 3.84 (s, 1H), 2.66-2.59 (m, 2H), 2.23 (s, 3H), 1.17 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR(75 MHz, $CDCl_3$): δ 143.7, 142.5, 128, 126.8, 122.2, 121.6, 120.6, 120.5, 118.9, 116.7, 21.1, 14.2, 13.4; MS (ES) m/z 212 [M+H, 100]; Anal. Calcd. for $C_{15}H_{17}N$ (%): C, 85.26; H, 8.11; N, 6.63 Found: C, 85.45; H, 8.01; N, 6.76.

2-bromo-4-methyl-*N*-phenylaniline, 1r [22]:



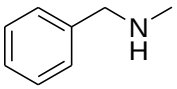
Light Yellow Oil (0.194 g, 74% yield); IR (KBr, cm^{-1}):υ 3387, 3057, 3022, 2924, 2867, 1589, 1034, 754; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.39-7.27 (m, 3H), 7.21 (d, J = 8.4 Hz, 1H), 7.15-7.11 (m, 2H), 7.05-6.95 (m, 3H), 2.30 (s, 3H); NMR(100 MHz, $CDCl_3$): δ 141.5,132.7, 129.5, 129.4, 129, 121.6, 119.1, 118.3, 117.6, 115.8, 109.3, 20.1

2-methyl-4-nitro-*N*-phenylaniline, 1s:



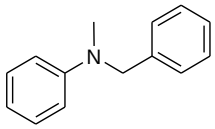
Light yellow gum (0.178 g, 78% yield); IR (KBr, cm^{-1}):υ 3382, 3052, 3027, 2934, 2865, 1592, 1575, 1045, 778; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.42-7.27 (m, 5H), 7.05-6.99 (m, 3H), 2.37 (s, 3H); ^{13}C NMR(75 MHz, $CDCl_3$): δ 143.3, 142.1, 129.3, 129.2, 126.4, 121.8, 121.2, 120.2, 118.6, 116.4, 13.0 MS (ES) m/z 229 [M+H, 100]; Anal. Calcd. for $C_{13}H_{12}N_2O_2$ (%): C, 68.41; H, 5.3; N, 12.27 Found: C, 68.28; H, 5.39; N, 12.39.

N-methylbenzylamine, 1t



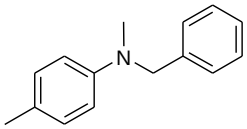
Colourless oil (0.097 g, 80%); IR 3027, 2978, 2855, 1658, 1562, 1475, 1320,1272, 1047, 892, 743; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.37-7.27 (m, 5H), 3.75 (s, 2H), 2.46 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$):δ 140, 128.4, 128.2, 127, 56, 35.9

***N*-benzyl-*N*-methylaniline,** 2a [4]:



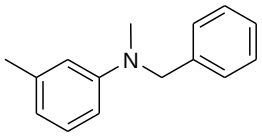
Light Yellow Oil (0.183 g, 93% yield); IR (KBr, cm^{-1}):υ 3012, 2987, 2863, 1788, 1578, 1465, 1319, 1282, 1056, 933, 747; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.35-7.23 (m, 8H), 6.78-6.7 (m, 2H), 4.55 (s, 2H), 3.03 (s, 3H); ^{13}C NMR(75 MHz, $CDCl_3$):δ 136.3, 134.4, 129.7, 129, 128.4, 126.7, 116.4, 112.3, 56.5, 38.5.

***N*-benzyl-*N*,4-dimethylaniline,** 2b [4]:



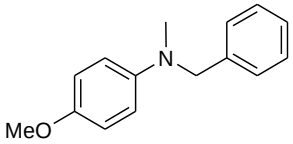
Light Yellow Oil (0.190 g, 90% yield); IR (KBr, cm^{-1}): υ 3062, 2911, 2853, 1627, 1564, 1487, 1472, 1325, 1277, 1131, 1029, 801, 743; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.35-7.27 (m, 5H), 7.18-7.06 (m, 2H), 6.74-6.58 (m, 2H), 4.53 (s, 2H), 3.01 (s, 3H), 2.29 (s, 3H); ^{13}C NMR(75 MHz, $CDCl_3$): δ 147.7, 139.2, 129.6, 128.5, 126.8, 125.7, 117.4, 112.6, 56.9, 38.6, 20.2.

***N*-benzyl-*N*,3-dimethylaniline,** 2c [4]:



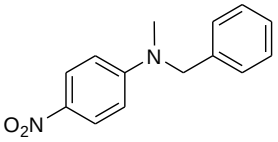
Light Yellow Oil (0.184 g, 87% yield); IR (KBr, cm^{-1}): υ 2931, 2850, 2822, 2776, 1621, 1534, 1456, 1366, 1250, 1181, 961, 802; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.34-7.25 (m, 6H), 6.61-6.56 (m, 3H), 4.55 (s, 2H), 3.02 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 149.9, 139.2, 129.7, 129.0, 128.5, 126.8, 117.5, 113.0, 112.6, 109.6, 56.9, 38.6, 21.9.

***N*-benzyl-4-methoxy-*N*-methylaniline,** 2d [4]:



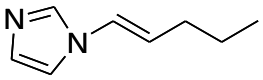
Yellow Oil (0.191 g, 84% yield); IR (KBr, cm^{-1}): υ 2952, 2837, 1588, 1516, 1332, 1234, 1179, 1140, 1041, 825, 783, 752, 687; ^{1}H NMR (300 MHz, $CDCl_3$):δ 7.34-7.21 (m, 7H), 6.29 (d, *J* = 6 Hz, 2H), 4.53 (s, 2H), 3.77 (s, 3H), 3.01 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$):δ 160.7, 151.1, 138.9, 129.8, 128.5, 126.8, 126.6, 114.6, 56.5, 55.1, 38.6.

***N*-benzyl-*N*-methyl-4-nitroaniline,** 2e [14]:



Yellow Solid (0.218 g, 90% yield); IR (KBr, cm^{-1}): υ 2910, 2817, 1570, 1538, 1359, 1243, 1169, 1097, 817, 793, 743, 674; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.11(d, *J* = 9 Hz, 2H), 7.35-7.18 (m, 5H), 6.66 (d, *J* = 9 Hz, 2H), 4.68 (s, 2H), 3.2 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 153.76, 137.30, 137.56, 128.94, 128.56, 127.52, 126.22, 110.57, 56.12, 39.19.

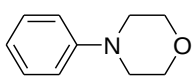
1-pent-1-enyl-1H-imidazole, 2f:



Colourless Liquid (0.122 g, 90%); E/Z isomeric ratio 3:1; IR (KBr, cm^{-1}): υ 2968, 2934, 1655, 1511, 1334, 1094, 745, 610; ^{1}H NMR (300 MHz, $CDCl_3$) for major isomer: δ 7.51-7.46 (m, 1H), 7.02-6.9 (m, 2 H), 6.61-6.48 (m, 1H), 5.76-5.67 (m, 1H), 2.10-1.99 (m, 2H), 1.45-1.32 (m, 2H), 0.88-0.83 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 136.7, 135.4, 129.3, 128.7, 125.6, 123, 122.1, 119.7, 118.9, 115.9, 31.3, 28.5, 22.1, 13.2; ; HRMS (ESI) calcd for $[M+H]^+$: 137.1079; found 137.1084.

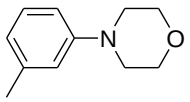
4-phenylmorpholine, 3a [4]:

Light brown solid (0.127 g, 78% yield); IR (KBr, cm^{-1}): υ 2955, 2842, 2817, 1610, 1512, 1452, 1267, 1229, 1118, 922, 745, 697; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.37-7.24 (m, 2H), 6.99-6.89 (m, 3H), 3.92 (t, *J* = 4.5 Hz, 4H), 3.20 (t, *J* = 4.5 Hz, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 150.96, 129.17, 120.35, 115.88, 66.80, 49.43.



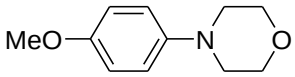
4-(3-methylphenyl)morpholine, 3b [4]:

Light brown solid (0.133 g, 75% yield); IR (KBr, cm^{-1}): υ 2959, 2856, 2816, 2759, 1609, 1495, 1449, 1243, 1180, 1118, 951, 871, 775, 688; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.24 (t, *J* = 7.8 Hz, 1H), 6.82-6.75 (m, 3H), 3.92 (t, *J* = 4.8 Hz, 4H), 3.20 (t, *J* = 4.8 Hz, 4H), 2.40 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 151.3, 139, 129.1, 121.2, 116.7, 113, 67, 49.6, 21.8



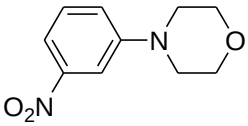
4-(4-methoxyphenyl)morpholine, 3c [4]:

white solid (0.139 g, 72% yield); IR (KBr, cm^{-1}): υ 2965, 2851, 2822, 2742, 1570, 1512, 1456, 1267, 1239, 1187, 1113, 1026, 924, 815, 706; ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.93-6.84 (m, 4H), 3.87 (t, *J* = 4.8 Hz, 4H), 3.78 (s, 3H), 3.07 (t, *J* = 4.8 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.1, 145.5, 117.9, 114.6, 67.0, 55.6, 50.9



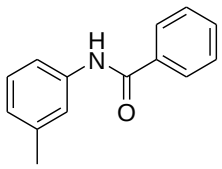
4-(3-nitrophenyl)morpholine, 3d [19]:

Yellow solid (0.150 g, 72% yield); IR (KBr, cm^{-1}): υ 2958, 2832, 2770, 1626, 1591, 1506, 1261, 1214, 1118, 968, 831, 745, 625; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.52-7.47 (m, 1H), 7.41-7.36 (m, 1H), 7.31-7.27 (m, 1H), 7.17 (s, 1H), 3.95 (t, *J* = 4.8 Hz, 4H), 3.27 (t, *J* = 4.8 Hz, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 149.1, 128.9, 123.6, 119, 110.2, 66.9, 49.8



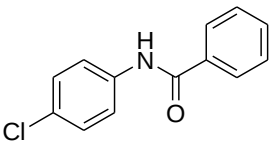
***N*-(3-methylphenyl)benzamide,** 4a [5]:

Light yellow solid (0.158 g, 75% yield); IR (KBr, cm^{-1}): υ 3275, 1640, 1535, 1302, 1255, 715 ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.93 (br, 1H) 7.87 (d, *J* = 6.9 Hz, 2H), 7.41-7.57 (m, 5H), 7.22-7.28 (m, 1H), 6.97 (d, *J* = 7.5 Hz, 1H), 2.36 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.7, 139.0, 137.8, 135.0, 131.8, 128.8, 128.7, 127.0, 125.3, 120.8, 117.2, 21.5.



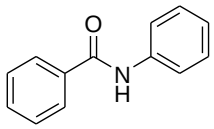
***N*-(4-chlorophenyl)benzamide,** 4b [5]:

Light yellow solid (0.169 g, 73% yield); IR (KBr, cm^{-1}): υ 3345, 1653, 1592, 1522, 1490, 1395, 820, 715; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.96 (br, 1H), 7.88 (d, *J* = 8.4Hz, 2H), 7.47-7.64 (m, 5H), 7.34



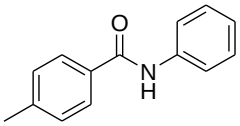
(d, *J* = 9 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 172.3, 136.5, 134.5, 132.0, 129.1, 128.8, 127.0, 121.4.

***N*-phenylbenzamide,** 4c [5]:



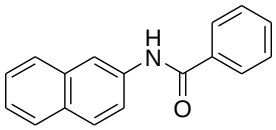
White solid (0.154 g, 78% yield); IR (KBr, cm^{-1}): υ 3340, 1649, 1536, 754; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.89 (d, *J* = 7.2Hz, 1H), 7.65 (d, *J* = 7.8Hz, 1H), 7.48-7.57 (m, 1H), 7.37-7.42 (m, 1H), 7.18-7.27 (m, 3H), 6.83-6.95 (m, 3H), 5.56 (br, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 155.6, 134.7, 132.0, 129.6, 129.3, 128.8, 127.0, 124.8, 120.5, 120.4, 115.3.

4-methyl-*N*-phenylbenzamide, 4d [5]:



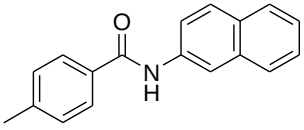
White solid (0.152 g, 72% yield); IR (KBr, cm^{-1}): υ 3345, 1654, 1528, 759; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.88 (br, 1H), 7.78 (d, *J* = 7.8 Hz, 2H), 7.65 (d, *J* = 8 Hz, 2H), 7.37 (t, *J* = 7.5 Hz, 2H), 7.28 (d, *J* = 8.1 Hz, 2H), 7.15 (t, *J* = 7.5 Hz, 1H), 2.43 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.7, 142.4, 138.0, 132.0, 129.4, 129.1, 127.0, 124.4, 120.1, 22.7,

***N*-(naphthalen-2-yl)benzamide,** 4e [10]:



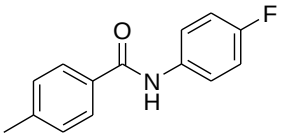
Brown solid (0.170 g, 69% yield); IR (KBr, cm^{-1}): υ 3065, 2975, 1765, 1705, 1545, 1462, 1378, 1019, 863, 735; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.37 (s, 1H), 8.13-8.18 (m, 1H), 7.94 (d, *J* = 7.2Hz, 2H), 7.8-7.86 (m, 3H), 7.41-7.62 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.9, 135.3, 134.9, 133.8, 131.9, 130.7, 128.8, 127.7, 127.6, 127.0, 126.5, 125.1, 120.1, 117.0.

4-methyl-*N*-(naphthalen-2-yl)benzamide, 4f [20]:



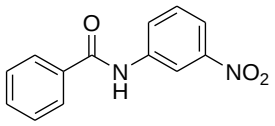
White soild (0.170 g, 65% yield); IR (KBr, cm^{-1}): υ 3285,3086, 2919, 1649, 1586, 1501, 1352, 1283, 1026, 866, 820; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.08 (s, 1H), 7.84-7.75 (m, 4H), 7.62-7.58 (m, 1H), 7.50-7.38 (m, 3H), 7.32-7.23 (m, 3H), 2.44 (s, 3H); ^{13}C NMR (100 MHz,$CDCl_3$): δ 165.8, 142.5, 135.4, 132, 130.7, 129.5, 128.8, 128.1, 127.7, 127.5, 127.1, 126.5, 125.1, 120.1, 117, 21.5

***N*-(4-fluorophenyl)-4-methylbenzamide,** 4g [16]:



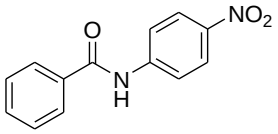
Light yellow solid (0.172 g, 75% yield); IR (KBr, cm^{-1}): υ 3252, 3095, 3017, 1680, 1522; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.89 (br, 1H), 7.76 (d, *J* = 8.1 Hz, 2H), 7.62-757 (m, 2H), 7.28 (d, *J* = 7.8 Hz, 2H), 7.05 (t, *J* = 8.7 Hz), 2.43 (s, 3H); ^{13}C NMR (75 MHz,$CDCl_3$): δ 165.7, 158.5, 142.5, 133.9, 131.8, 129.5, 126.9, 122.1, 115.8, 21.5.

***N*-(3-nitrophenyl)benzamide,** 4h [2]:



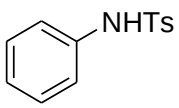
White solid (0.194 g, 80% yield); IR (KBr, cm^{-1}): υ 2959, 2919, 2856, 1747, 1616, 1529, 1484, 1352, 1312, 1273, 1113, 1078; ^{1}H NMR (300 MHz, $CDCl_3$):δ 8.51 (s, 2H), 8.49-8.30 (m, 3H), 7.99 (d, *J* = 7.5 Hz, 2H), 7.72 (d, *J* = 7.5 Hz, 2H). ^{13}C NMR (75 MHz,$CDCl_3$): δ 156.3, 140.4, 133.1, 130.3, 124.5, 123.3, 122.1, 121.9, 115.9, 110.5

***N*-(4-nitrophenyl)benzamide,** 4i [18]:



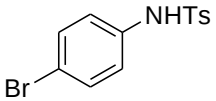
White solid (0.193 g, 80% yield); IR (KBr, cm^{-1}): υ 2952, 2915, 2847, 1649, 1620, 1589, 1526, 1501, 1479, 1388,1299. 1249, 1167, 1087, 1017; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.37 (d, *J* = 8.7 Hz, 2H), 8.17 (d, *J* = 9.3 Hz 2H), 7.92-7.79 (m, 3H), 6.93 (d, *J* = 9 Hz, 2H), ^{13}C NMR (100 MHz,$CDCl_3$): δ 161.9, 145, 141.4, 132.8, 129.1, 128.4, 127.2, 126.2, 125.2, 124.4, 119.7.

4-methyl-*N*-phenylbenzenesulfonamide, 4k [23]:



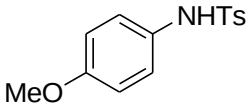
White solid (0.161 g, 65% yield); IR (KBr, cm^{-1}): υ 3297, 1377, 1185; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.69 (d, *J* = 7.8 Hz, 2H), 7.35 (s, 1H), 7.27-7.20 (m, 4H), 7.11-7.06 (m, 3H), 2.36 (s, 3H); ^{13}C NMR (100 MHz,$CDCl_3$): δ 144.1, 136.8, 136.2, 132.5, 129.9, 129.5, 127.5, 125.4, 123.1, 121.6, 21.7

***N*-(4-bromophenyl)-4-methylbenzenesulfonamide,** 4l [23]:



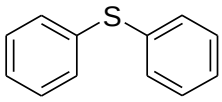
White solid (0.206 g, 63% yield); IR (KBr, cm^{-1}): υ 3301, 1346, 1180; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (s, 1H), 7.81 (d, *J* = 8.1 Hz, 2H), 7.39 (d, *J* = 8.7 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 2H), 7.11 (d, *J* = 8.7 Hz, 2H), 244 (s, 3H); ^{13}C NMR (100 MHz,$CDCl_3$): δ 144.4, 136.0, 135.6, 132.4, 130.0,127.4, 123.0, 118.4, 21.7

***N*-(4-methoxyphenyl)-4-methylbenzenesulfonamide,** 4m [23]:



White solid (0.167 g, 60% yield); IR (KBr, cm^{-1}): υ 3321, 1327, 1175,;NMR (400 MHz, $CDCl_3$): δ 7.58 (d, J = 8.2 Hz, 2H) 7.21 (d, J = 8.1 Hz, 2H), 6.97 (d, J = 8.9 Hz, 2H), 6.75 (d, J = 8.9 Hz, 2H), 6.51 (s, 1H), 3.75 (s, 3H), 2.38 (s, 3H) ^{13}C NMR (100 MHz,$CDCl_3$): δ 158.2, 143.9, 129.8, 129.1, 127.6, 125.7, 114.6, 55.6, 21.8.

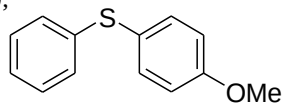
diphenyl sulfide, 5a [6]:



Colourless oil (0.149 g, 80% yield); IR (KBr, cm^{-1}): υ 3143, 3100, 1600, 1492, 1426, 1051, 612; ^{1}H

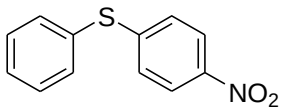
NMR (300 MHz,$CDCl_3$): δ 7.40- 7.30 (m, 10 H); ^{13}C NMR (75 MHz,$CDCl_3$): δ 135.7, 131, 129.1, 127.0.

1-methoxy-4-(phenylsulfanyl)benzene, 5b [7]:



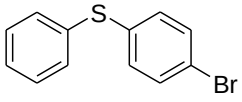
Colourless oil (0.168 g, 78% yield); IR (KBr, cm^{-1}): υ 3067, 3013, 2901, 2871, 1565, 1217, 1222, 1100; ^1H NMR (300 MHz,$CDCl_3$): δ 7.43 (d, *J* = 9 Hz, 2H), 7.27-7.15 (m, 5H), 6.91 (d, *J* = 9 Hz, 2H), 3.84(s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 159.8, 138.6, 135.4, 128.9, 128.1, 125.7, 124.2, 114.9, 55.3.

4-nitrophenyl phenyl sulfide, 5c [6]:



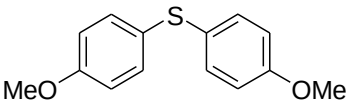
Yellow Solid (0.196 g, 85% yield); IR (KBr, cm^{-1}): υ 3120, 3019, 1592, 1489, 1293, 1118, 1854, 729; ^1H NMR (300 MHz, $CDCl_3$): δ 8.06(d, *J* = 9 Hz, 2H), 7.57-7.54(m, 2H), 7.47-7.46 (m, 3H), 7.17 (d, *J* = 9 Hz, 2H) ; ^{13}C NMR(75 MHz, $CDCl_3$): δ 148.4, 145.2, 134.6, 130.2, 129.9, 129.6, 126.5, 113.9.

4-bromophenyl phenyl sulfide, 5d, 5i[7]:



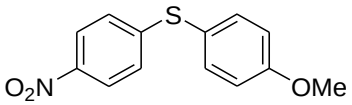
Colorless oil (0.212 g, 80% yield) (0.207 g 78% yield); IR (KBr, cm^{-1}): υ 2919, 2844, 1706, 1604,1489, 1266, 1203, 764; ^1H NMR (300 MHz, $CDCl_3$): δ 7.29-7.24 (m, 4H), 6.98-6.87 (m, 5H); ^{13}C NMR(75 MHz, $CDCl_3$): δ 137.8,135.1, 133.8, 132.9, 132.3, 128.9, 127.0, 126.5.

1,1'-sulfanediylbis(4-methoxybenzene), 5e [8]:



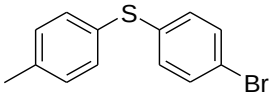
Colorless oil (0.187 g, 76% yield); IR (KBr, cm^{-1}): υ 3045, 1276, 1132, 1015, 780; ^1H NMR (300 MHz, $CDCl_3$): δ 7.31(d, *J* = 9 Hz, 4H), 6.86 (d, *J* = 9 Hz, 4H), 3.8(s, 6H); ^{13}C NMR(75 MHz, $CDCl_3$): δ 158.8, 132.6, 127.2, 114.6, 55.2.

1-methoxy-4-[(4-nitrophenyl)sulfanyl]benzene, 5f [12]:



Yellow Solid (0.211 g, 81% yield); IR (KBr, cm^{-1}): υ 3100, 2988, 2804, 1545, 1511, 1310, 1217, 1189, 1015, 867; ^1H NMR (300 MHz, $CDCl_3$): δ 8.04(d, *J* = 9 Hz, 2H), 7.49 (d, *J* = 9 Hz, 2H), 7.09 (d, *J* = 9 Hz, 2H), 7.0 (d, *J* = 9 Hz, 2H), 3.88(s, 3H); ^{13}C NMR(75 MHz, $CDCl_3$): δ 160.95, 150, 144.8, 137, 125.4, 123.8, 119.9, 115.5, 55.3.

1-bromo-4-[(4-methylphenyl)sulfanyl]benzene, 5g [9]:

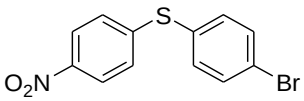


White Solid (0.349 g, 80% yield); IR (KBr, cm^{-1}): υ 3061, 1614, 1457, 1418, 1123, 1071, 1037, 717; ^1H NMR (300 MHz, $CDCl_3$): δ 7.37 (d, *J* = 9 Hz, 2H), 7.31 (d, *J* = 6 Hz, 2H), 7.16 (d, *J* = 9

Hz, 2H), 7.10 (d, *J* = 6 Hz, 2H), 2.36 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 138.05, 137.78, 136.64, 132.56, 131.9, 130.71, 130.11, 119.95, 21.05.

1-bromo-4-[(4-nitrophenyl)sulfanyl]benzene, 5h [13]:

Yellow Solid (0.257 g, 83% yield); IR (KBr, cm^{-1}): υ 3075, 2365, 1655, 1564, 1514, 1322, 1068, 1018, 853, 745; ^{1}H NMR (300 MHz, $CDCl_3$): δ: 8.06 (d, *J* = 9 Hz, 2H), 7.56 (d, *J* = 9 Hz, 2H), 7.38 (d, *J* = 9 Hz, 2H), 7.18 (d, *J* = 9 Hz, 2H); ^{13}C NMR(75 MHz, $CDCl_3$): δ 147.31, 145.45, 135.88, 133.11, 129.70, 126.91, 124.05.



Reference:

1. S. M. Islam, N. Salam, P. Mondal, A. S.Roy, K. Ghosh and K. Tuhina, *J. Mol. Catal. A: Chem.* 2014, **387**, 7.
2. Z.-J. Quan, H.-D. Xia, Z. Zhang, Y.-X. Da and X.-C. Wang, *Appl. Organometal. Chem.* 2014, **28,** 81.
3. M. M. Reddy, M. A. Kumar, P. Swamy, M. Naresh, K. Sru*J*ana, L. Satyanarayana, A. Venugopal and N. Narender, *Green Chem.* 2013, **15,** 3474.
4. L. Zhu, T.-T. Gao and L.-X. Shao, *Tetrahedron* 2011, **67**, 5150.
5. B. Y.-H. Tan and Y.-C. Teo, *Org. Biomol. Chem.* 2014, **12,** 7478.
6. A. Correa, M. Carril and C. Bolm, *Angew. Chem., Int. Ed*. 2008, **47**, 2880.
7. L. Rout, T. K. Sen and T. Punniyamurthy, *Angew. Chem. Int. Ed*. 2007, **46**, 5583.
8. M. Kuhn, F. C. Falk and *J*. Paradies, *Org. Lett.* 2011, **13**, 4100.
9. P. Malik and D. Chakraborty, *Appl. Organometal. Chem*. 2012, **26**, 557.
10. T. Kaicharla, M. Thangara*J* and A. T. Biju, *Org. Lett.* 2014, **16,** 1728.
11. C. Salome, P. Wagner, M. Bollenbach, F. Bihel, J.-J. Bourguignon and M. Schmitt, *Tetrahedron* 2014, **70**, 3413.
12. N. Singh, R. Singh, D. S. Raghuvanshi and K. N. Singh, *Org. Lett*. 2013, **15**, 5874.
13. R. M. López-Nicolás, J. A. Abad and J. Vicente, *Tetrahedron Lett.* 2005, **46,** 5839.
14. A. R. Hajipour, F. Dordahan and F. Rafiee, *Appl. Organometal. Chem*. 2013, **27,** 704.

15. D. B. Bagal, R. A. Watile, M. V. Khedkar, K. P. Dhake, and B. M. Bhanage, *Catal. Sci. Technol.* 2012, ***2***, 354.

16. K. N. Kumar, K. Sreeramurthy, S. Palle, K. Mukkanti and P. Das, *Tetrahedron Lett.* 2010, **51**, 899.

17. T. Kubo, C. Katoh, K. Yamada, K. Okano, H. Tokuyama and T. Fukuyama, *Tetrahedron* 2008, **64**, 1120.

18. S. Jammi, S. Sakthivel, L. Rout, T. Mukherjee, S. Mandal, R. Mitra, P. Saha and T. Punniyamurthy, J. *Org. Chem.* 2009, **74**, 1971.

19. D. Jiang, H. Fu, Y. Jiang and Y. Zhao, *J. Org. Chem.* 2007, **72**, 672.

20. T. T. Dang, Y. Zhu, S. C. Ghosh, A. Chen, C. L. L. Chai and A. M. Seayad, *Chem. Commun.* 2012, **48**, 1805.

21. M. A. Carroll and R. A. Wood *Tetrahedron* 2007, **63**, 11349.

22. M. T. Barros, S. S. Dey and C. D. Maycock, *Eur. J. Org.* 2013, 742.

23. S.-Y. Moon, J. Nam, K. Rathwell and W.-S. Kim, *Org. Lett.* 2014, **16**, 338.

24. L. Zhu, T.-T. Gao and L.-X. Shao, *Tetrahedron* 2011, **67**, 5150

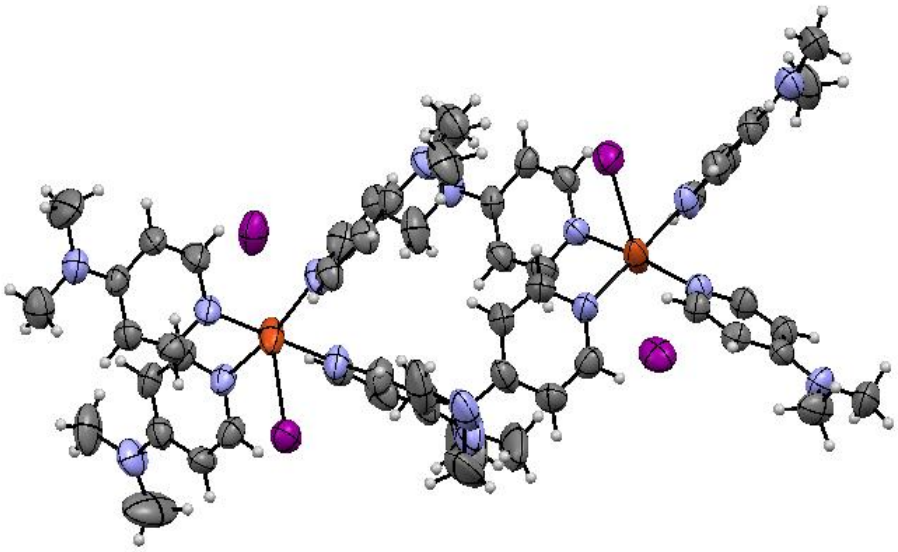
25. T. Kubo, C. Katoh, K. Yamada, K. Okano, H. Tokuyama and T. Fukuyama Tetrahedron 2008, 64, 11230

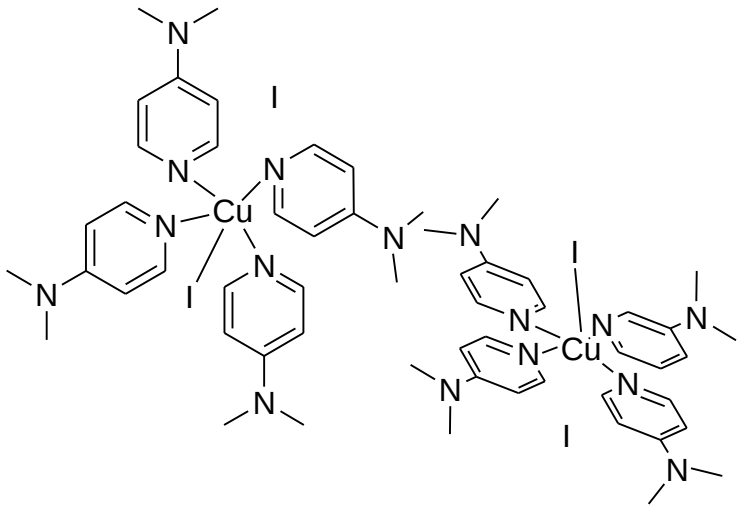
Single-Crystale Data Parameters of the catalyst

Chemical formula	$C_{28}H_{40}CuI_2N_8$
Formula weight	806.02
Crystal system	Monoclinic
Space group	$P2_1/c$
T/K	296(2)
a/Å	18.1909(3)
b/Å	19.1754(4)
c/Å	20.7590(4)
α/°	90
β/°	112.8500(10)
γ/°	90
Z	8
V/Å3	6672.9(2)
Dcalc/g cm−3	1.605
μ/mm−1	2.535
Reflns. Collected	16166
Unique reflns.	8300
R1 [I > 2(I)]	0.0452
wR2 (all)	0.1283
Goodness-of-fit	0.979
Structure refinement	SHELXL-97
Data collection	Bruker SMART

$C_{28}H_{40}CuI_2N_8$	Z=8
M_r = 806.02	D_x =1.605 Mgm^{-3}
$P2_1/c$	Mo *Kα* radiation
a=18.1909(3)°A	μ = 2.53 mm^{-1}
b=19.1754(4)°A	T = 296(2) K
c=20.7590(4)°A	Needle, brown
V=6672.86°A^3	0.35 x 0.28 x 0.25 mm

Absorption Correction:
T_{min} = 0.4707, T_{max} = 0.5698
61812 measured reflections
16166 independent reflections
8300 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 28.1°
h = -24→23
k = -22→25
l =-23→27

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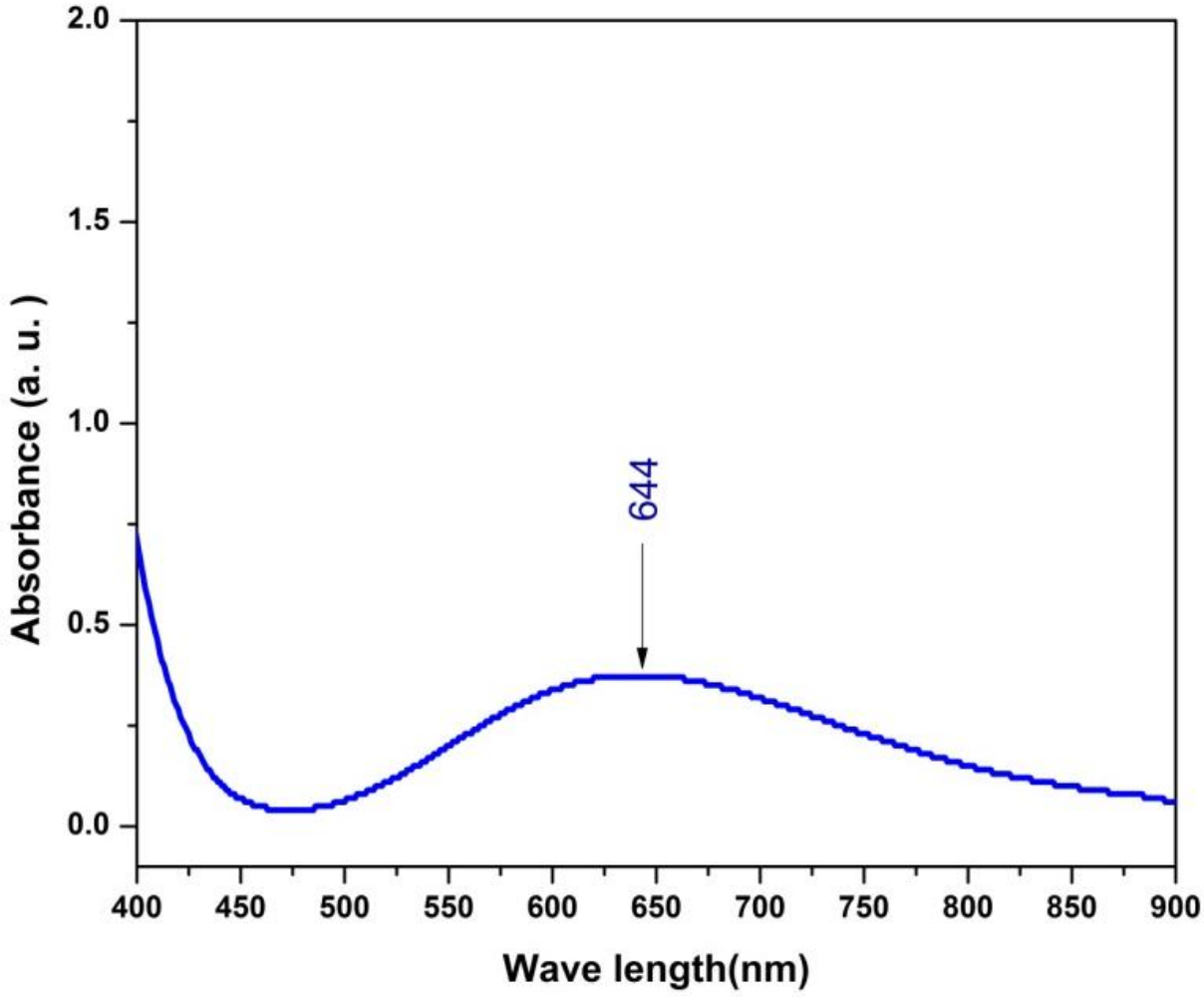
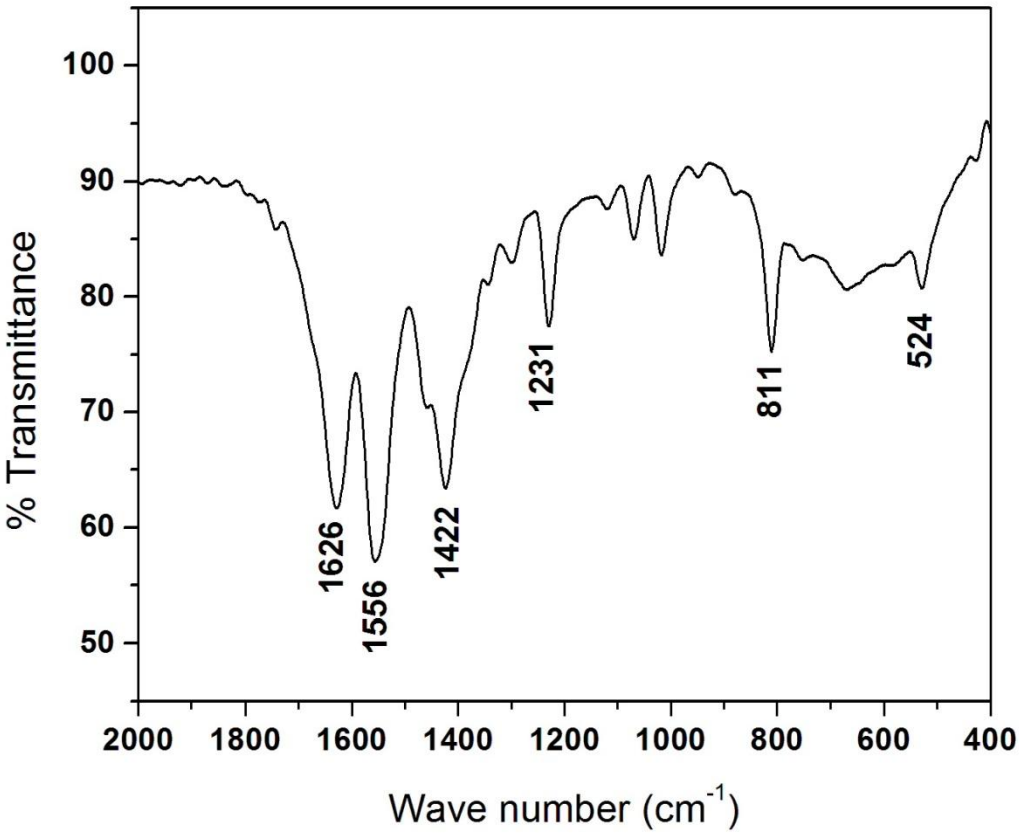
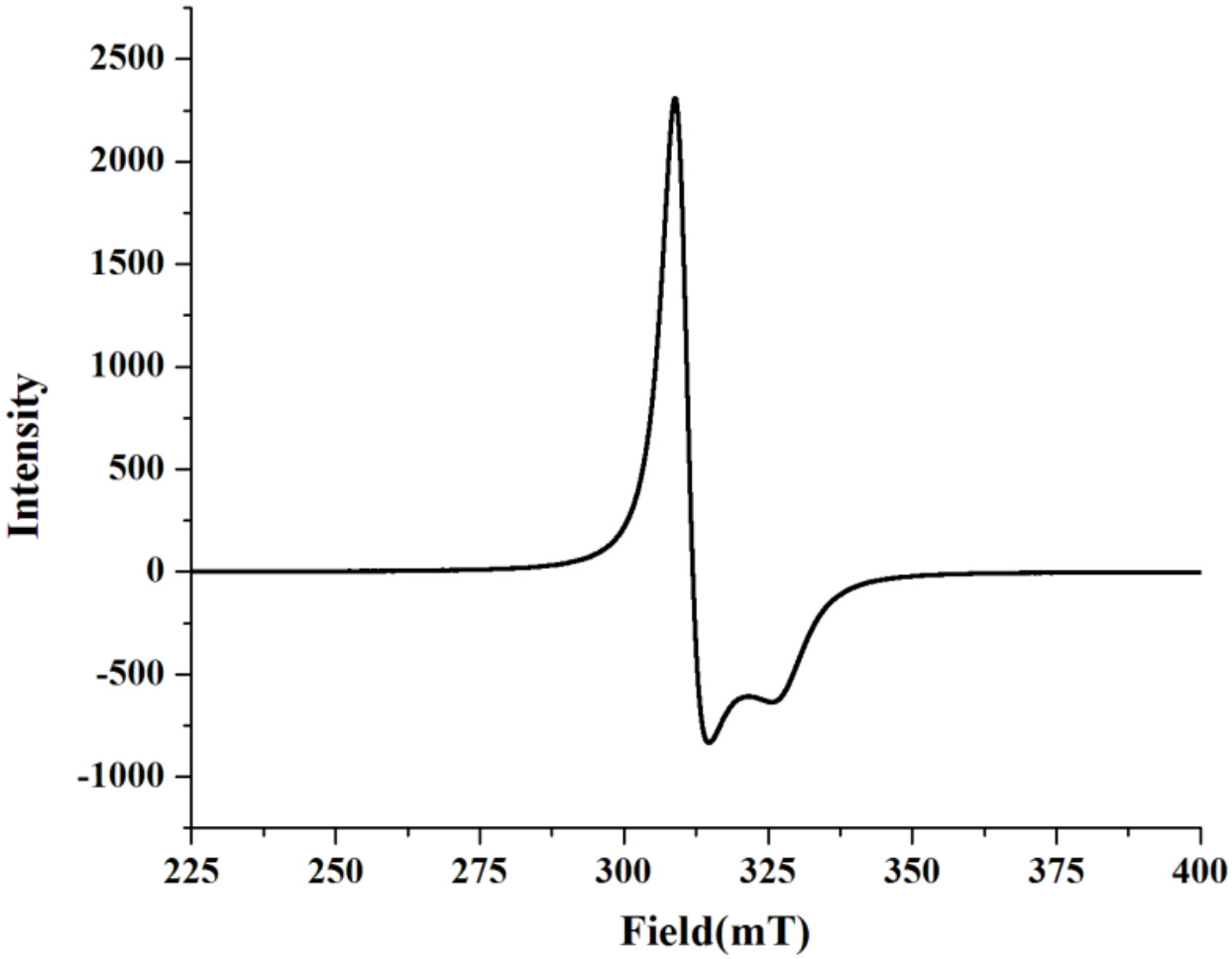
UV-visible spectrum of [Cu (DMAP)$_4$I]I



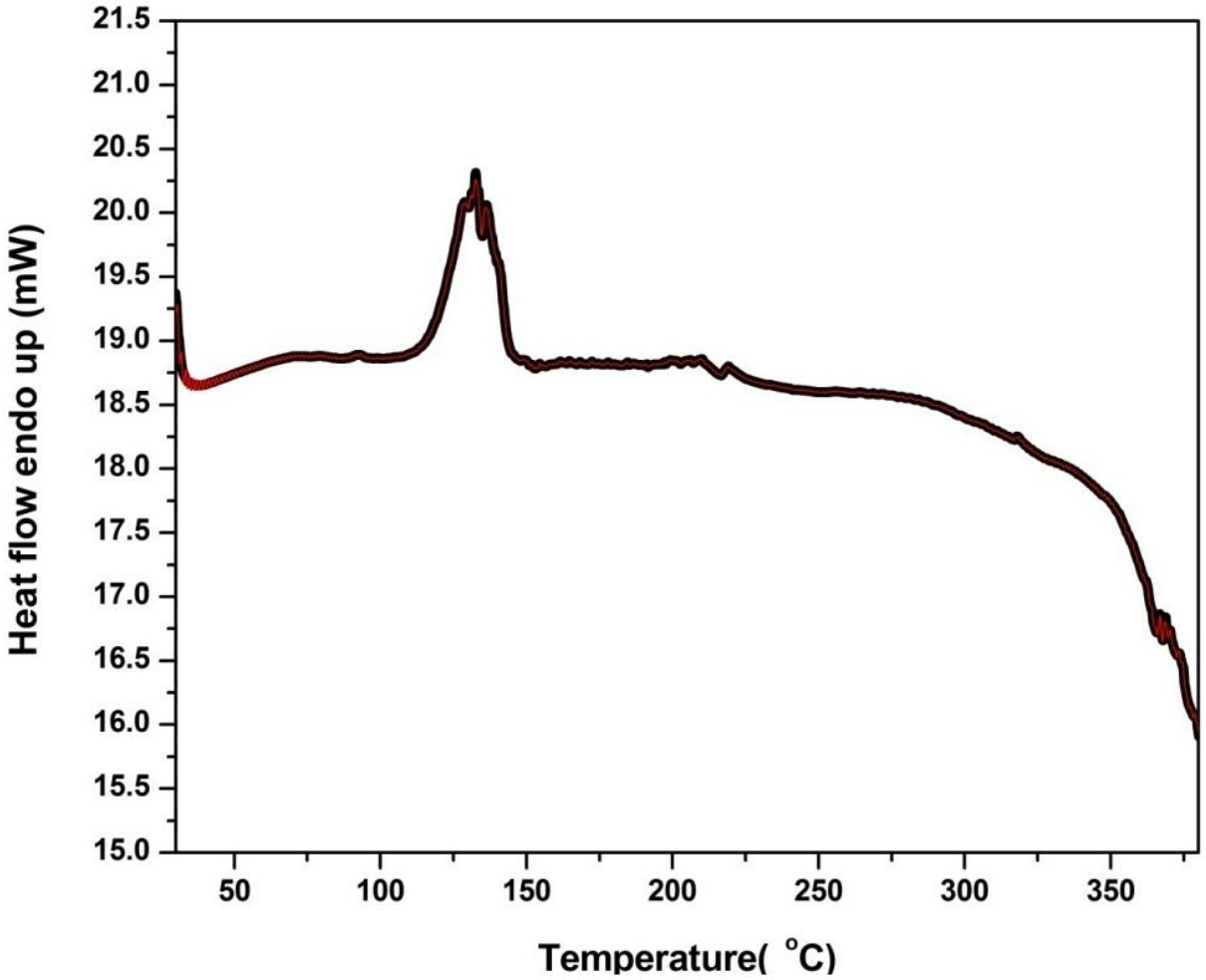
FT-IR spectrum of [Cu (DMAP)$_4$I]I



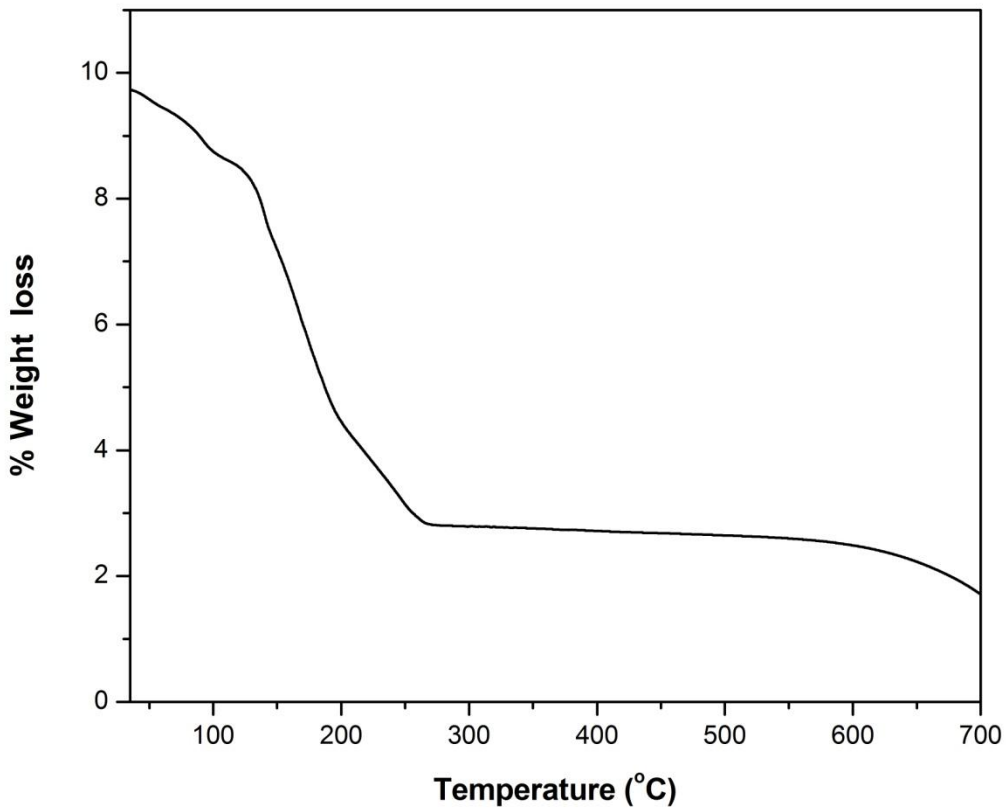
X –band EPR of [Cu (DMAP)$_4$I]I complex at room temperature

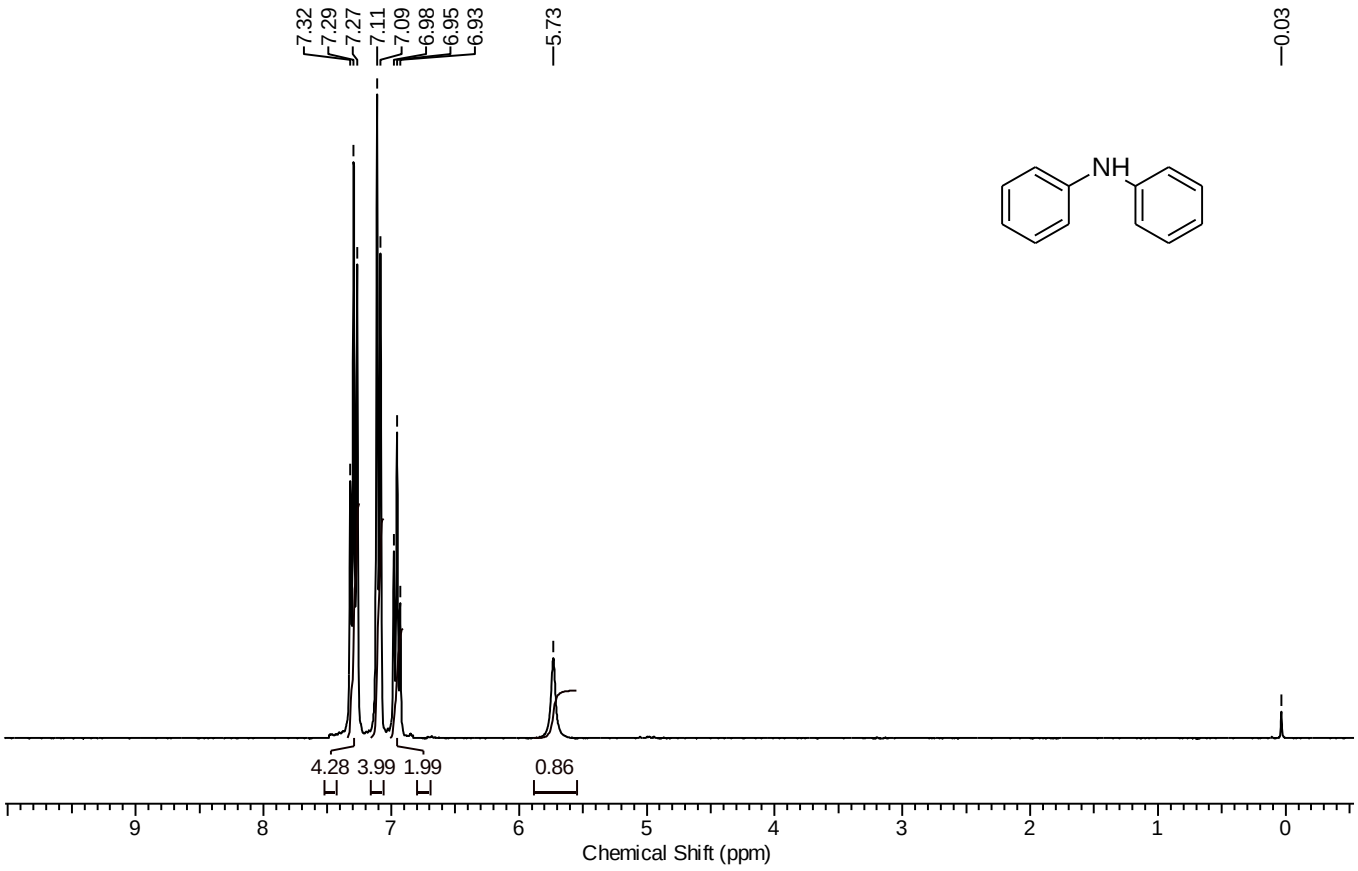


DSC analysis of [Cu (DMAP) $_4$I] I complex



TGA of [Cu (DMAP)$_4$I]I

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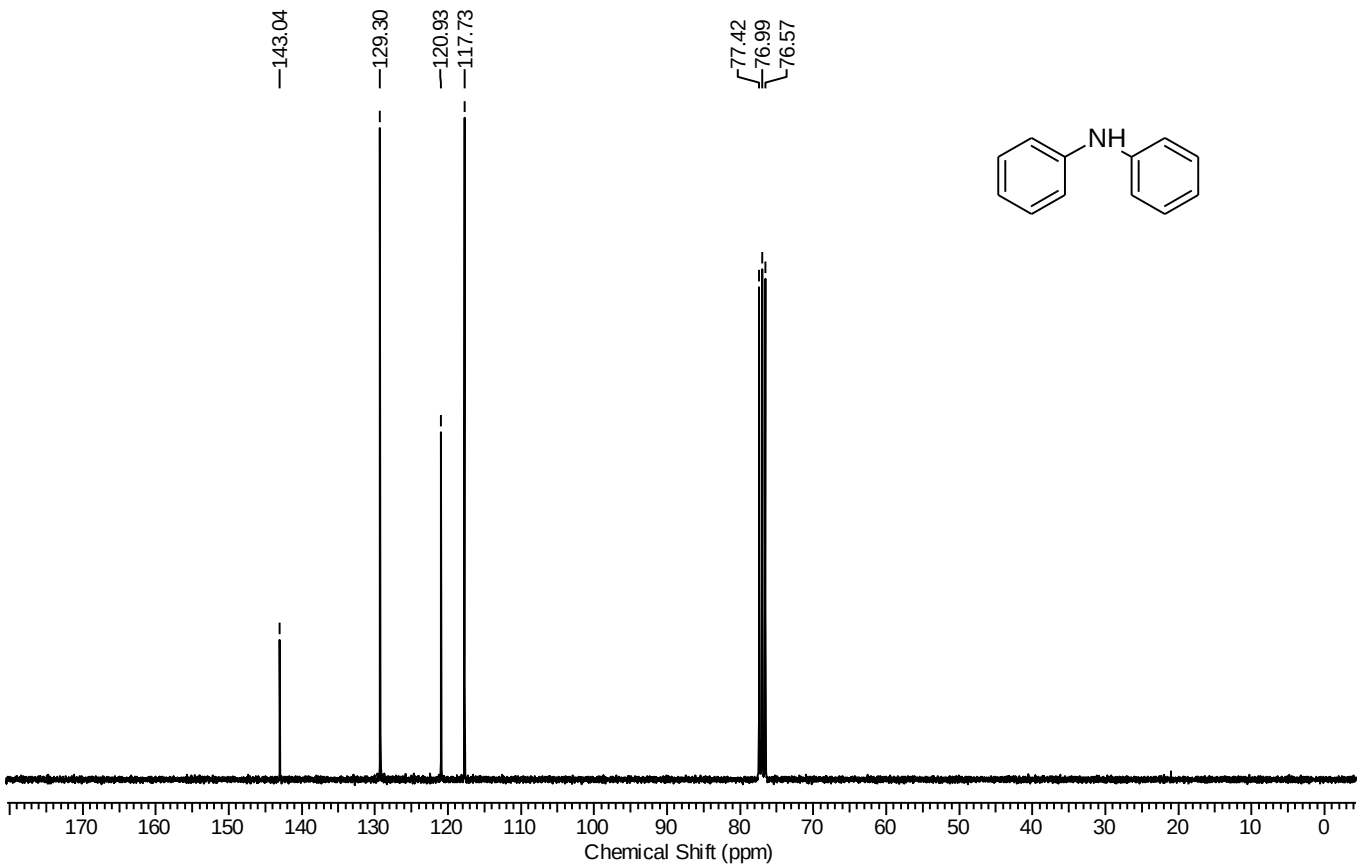
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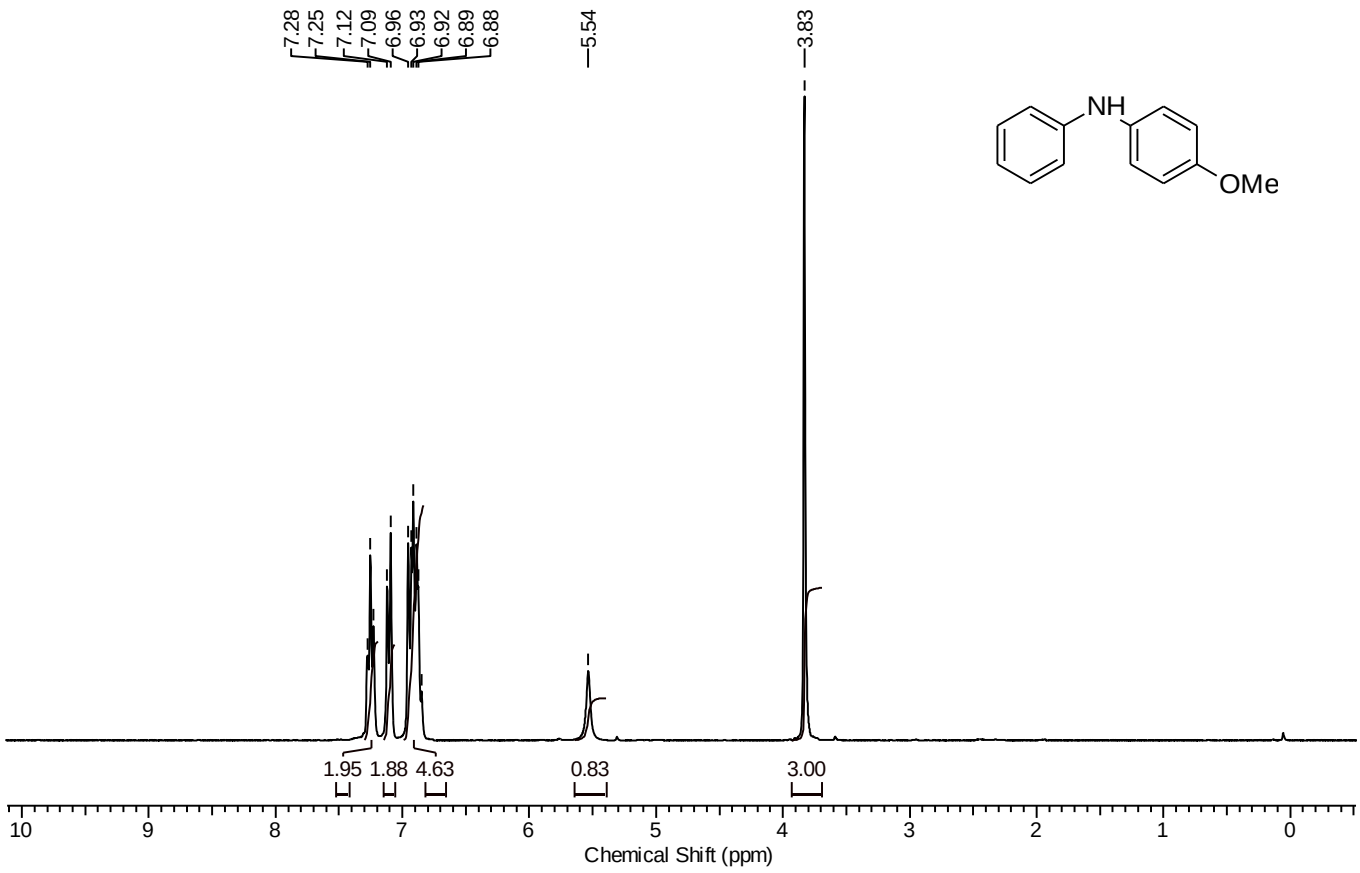
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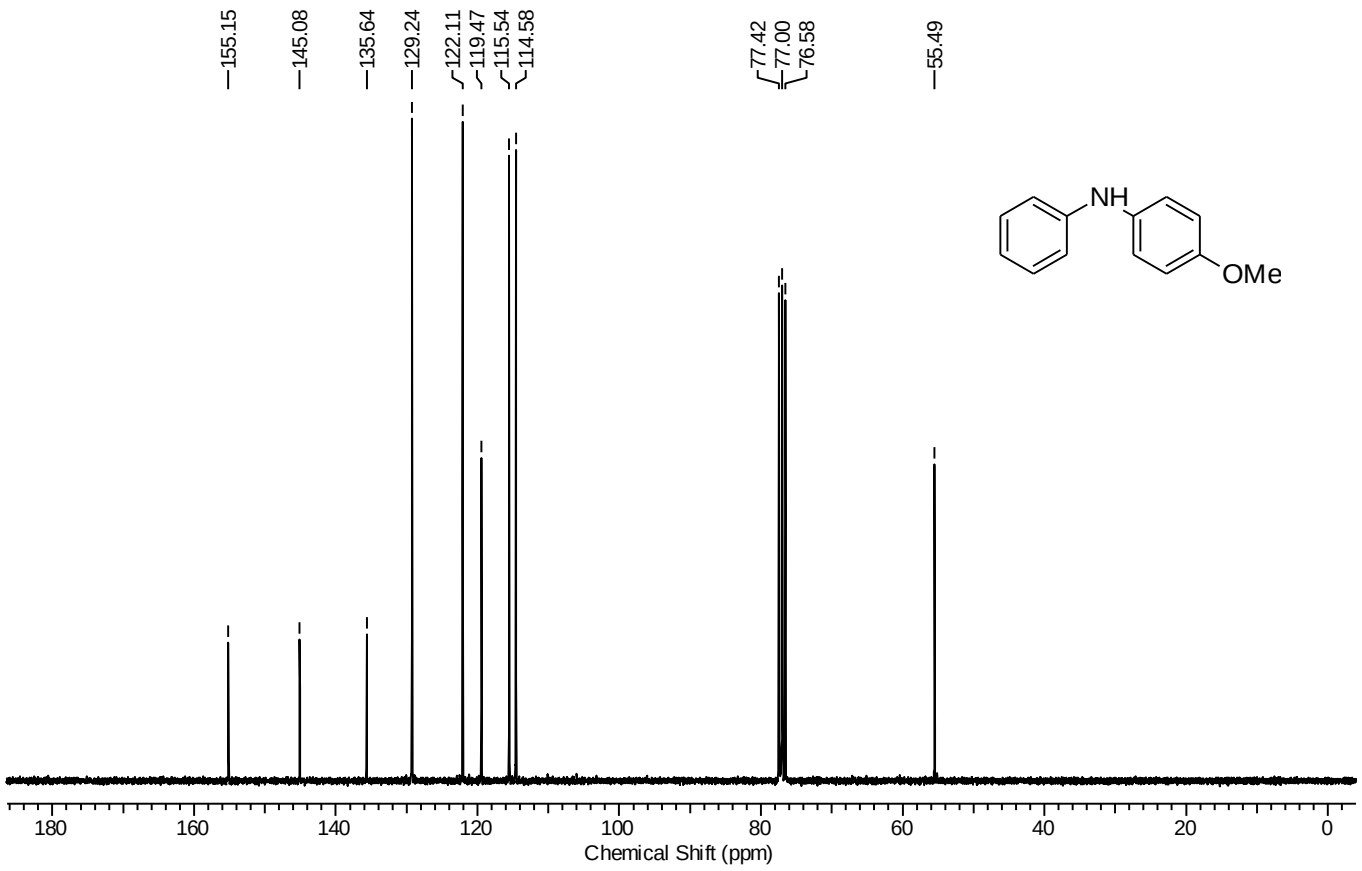
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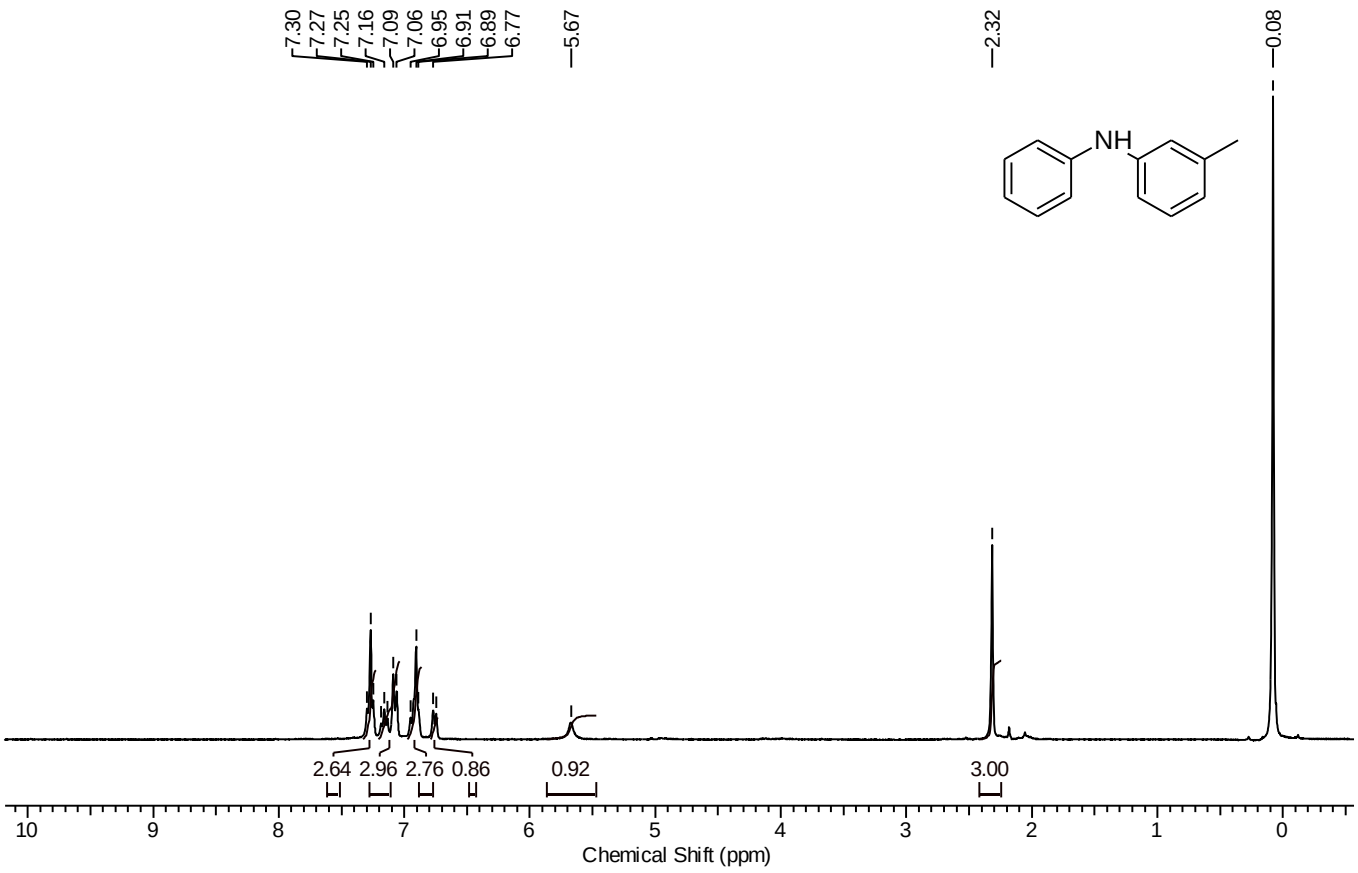
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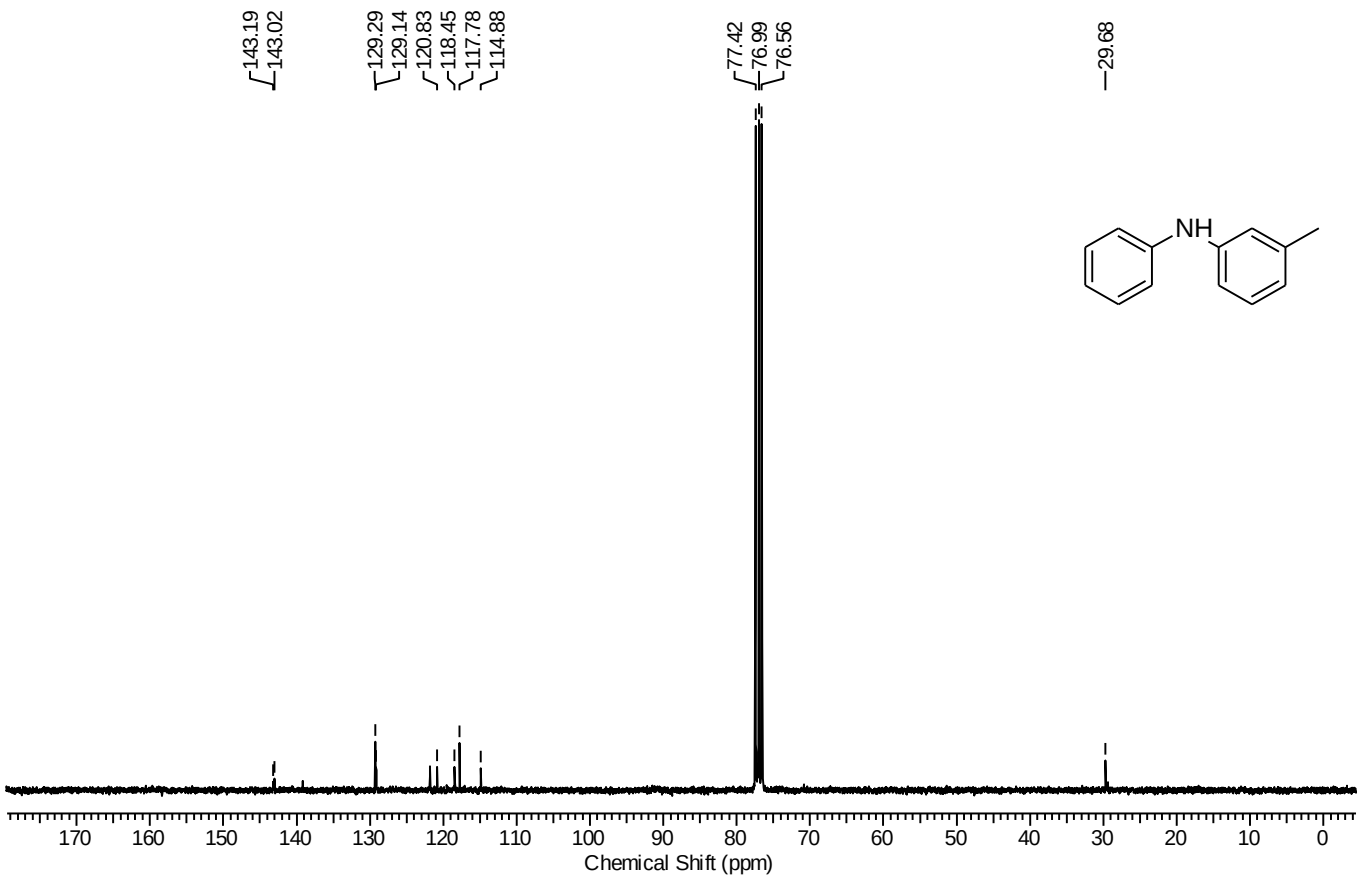
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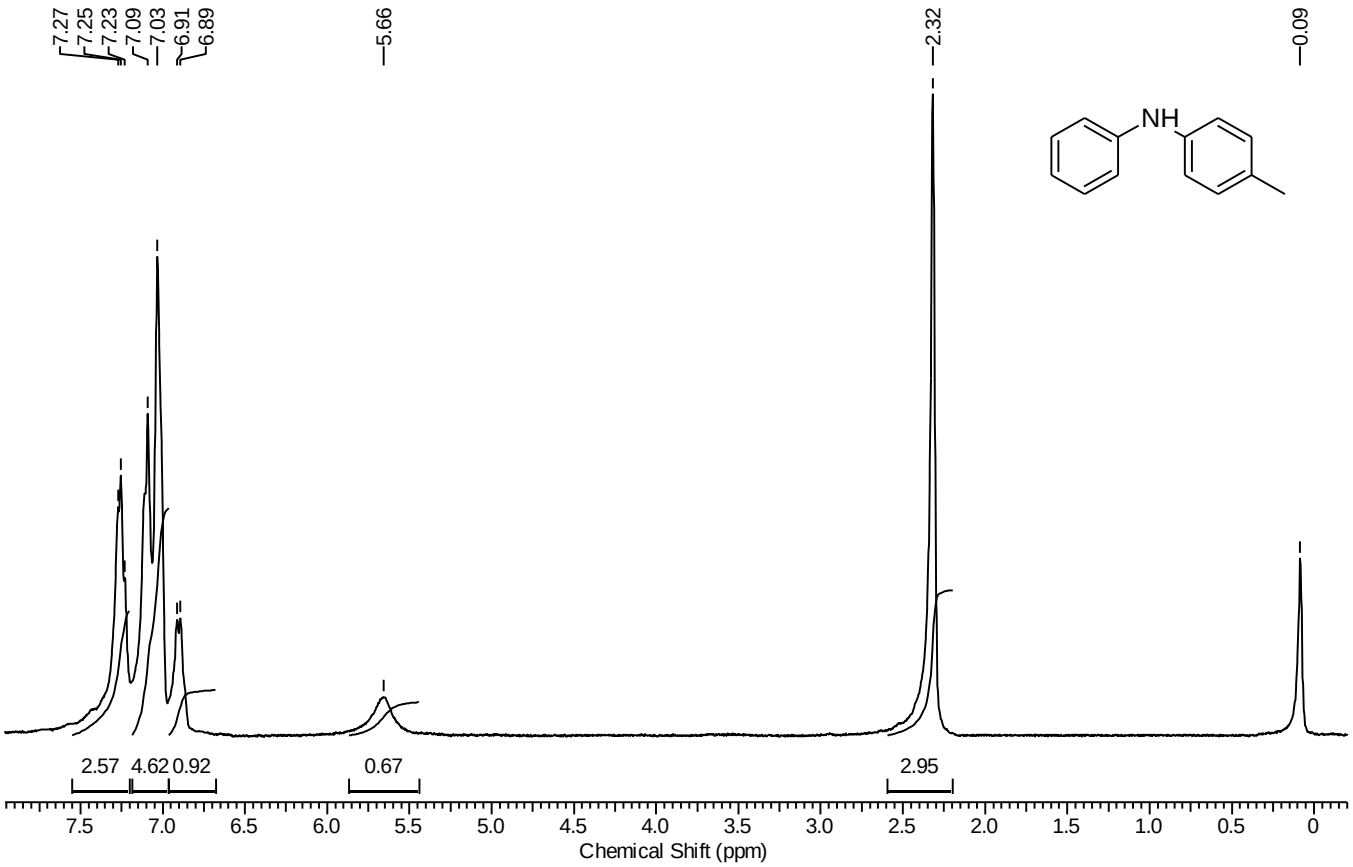
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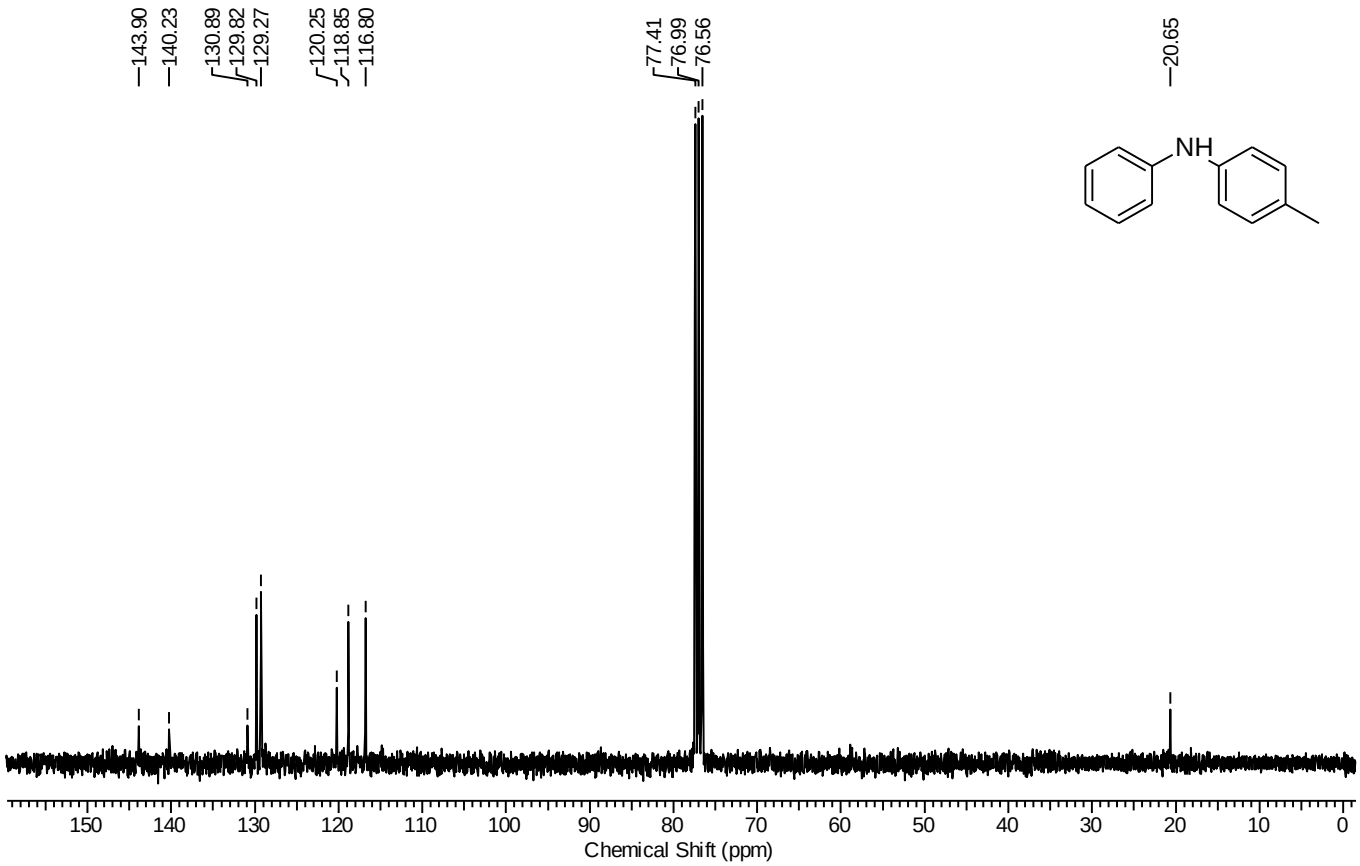
Chemical Shift (ppm)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50
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10
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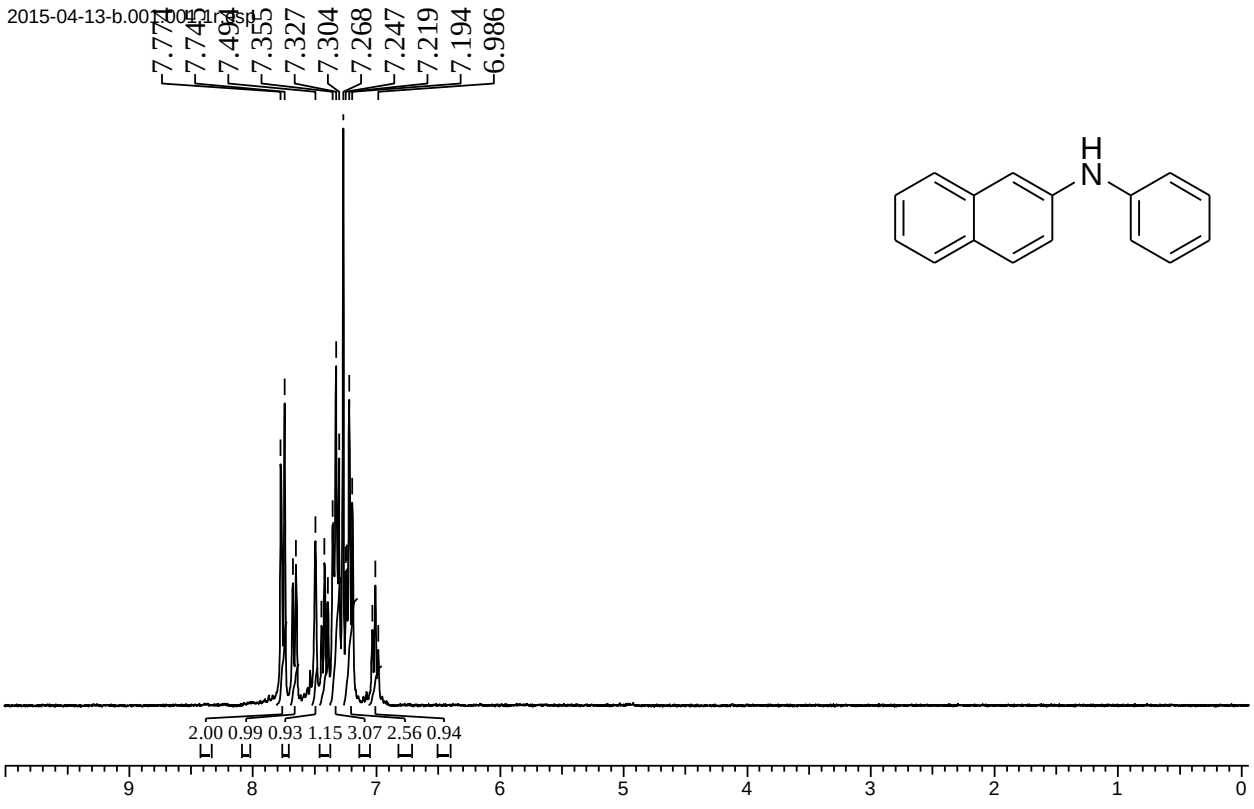
Chemical Shift (ppm)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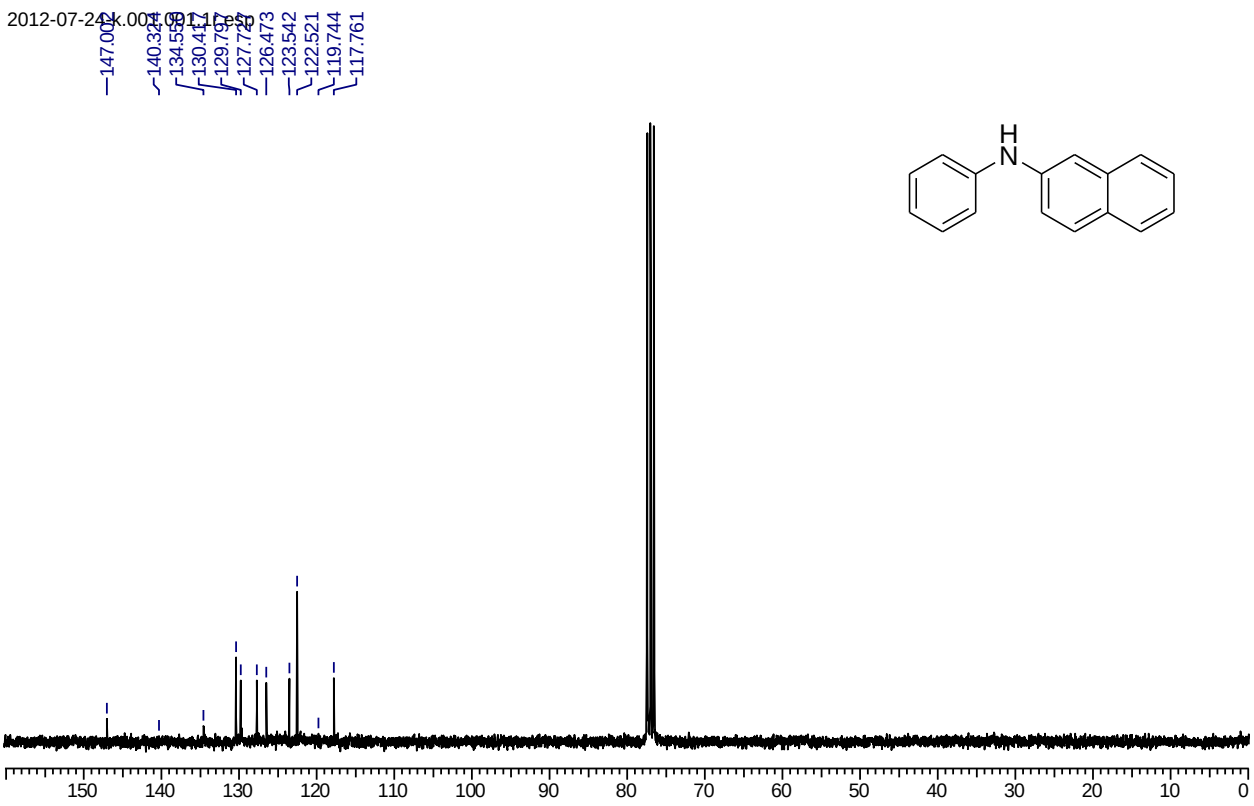
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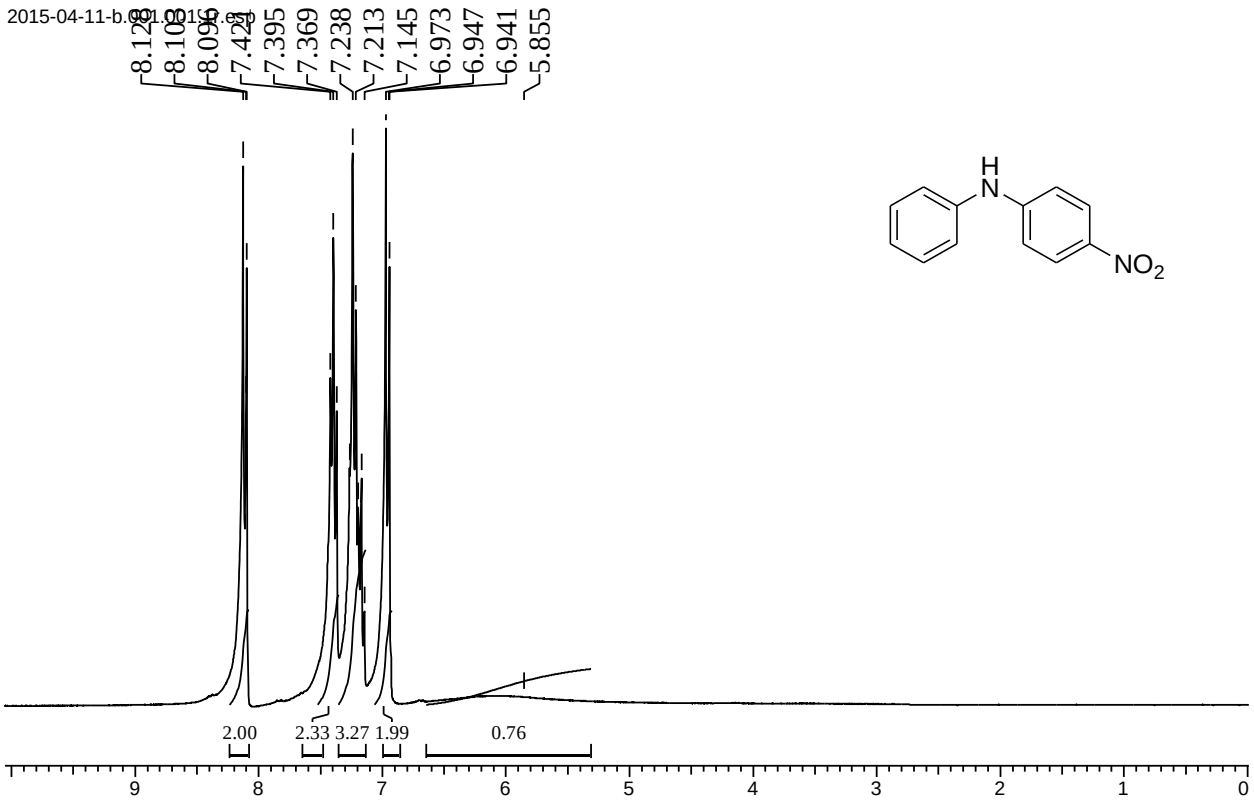
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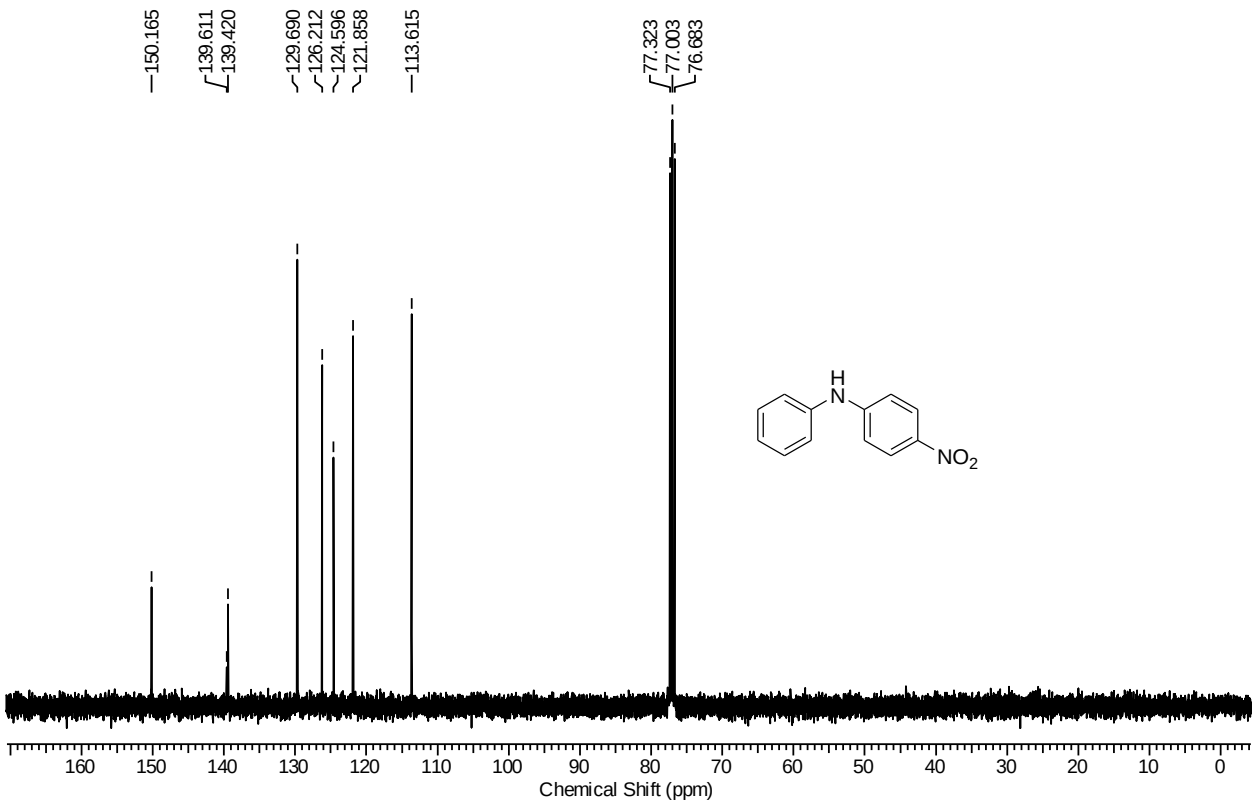
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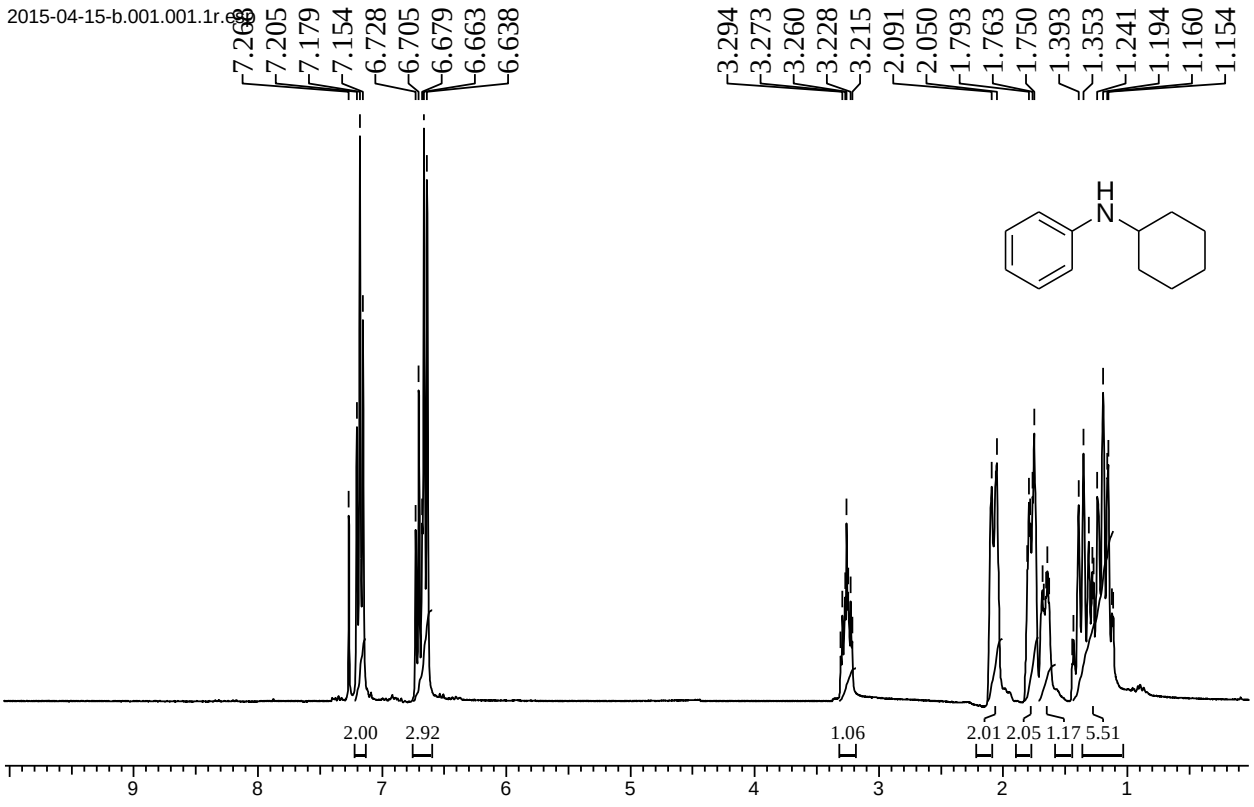
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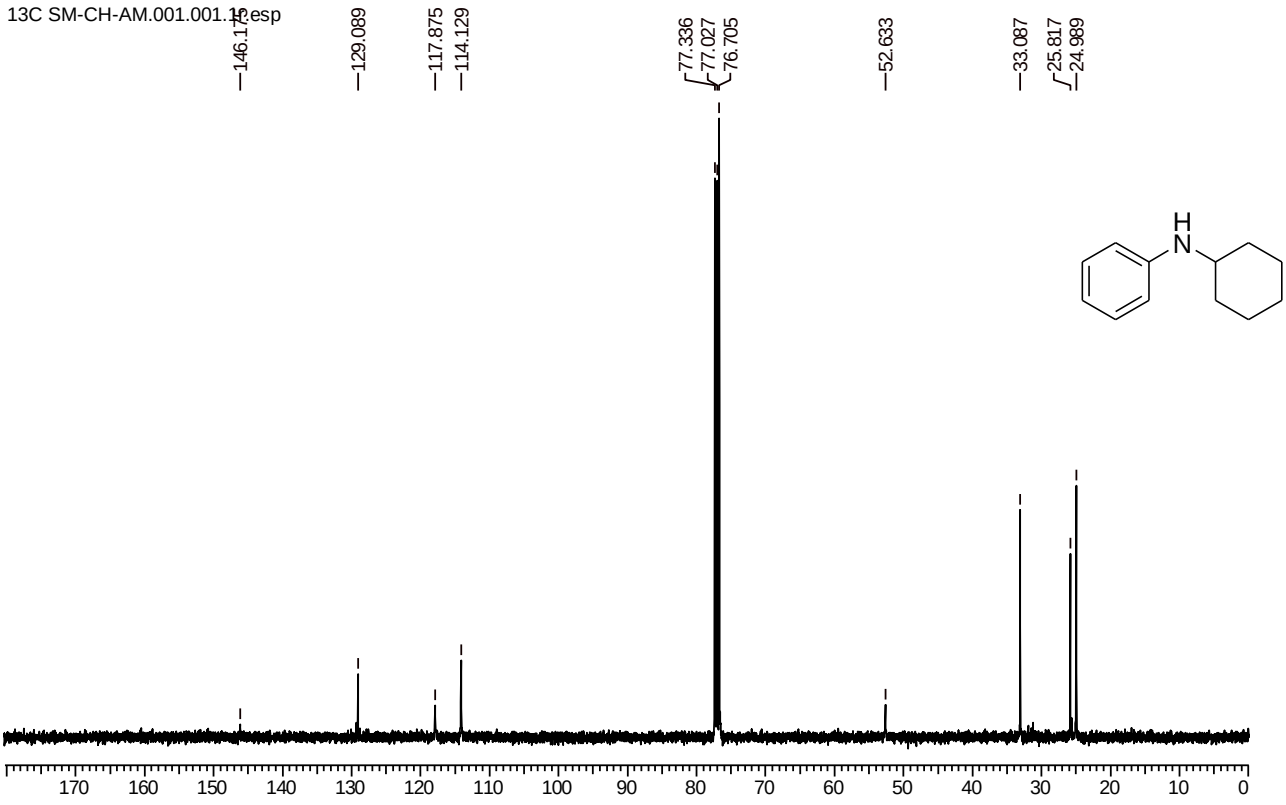
13C SM-CH-AM.001.001.1r.esp
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117.875
114.129
77.336
77.027
76.705
52.633
33.087
25.817
24.989
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N
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150
140
130
120
110
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70
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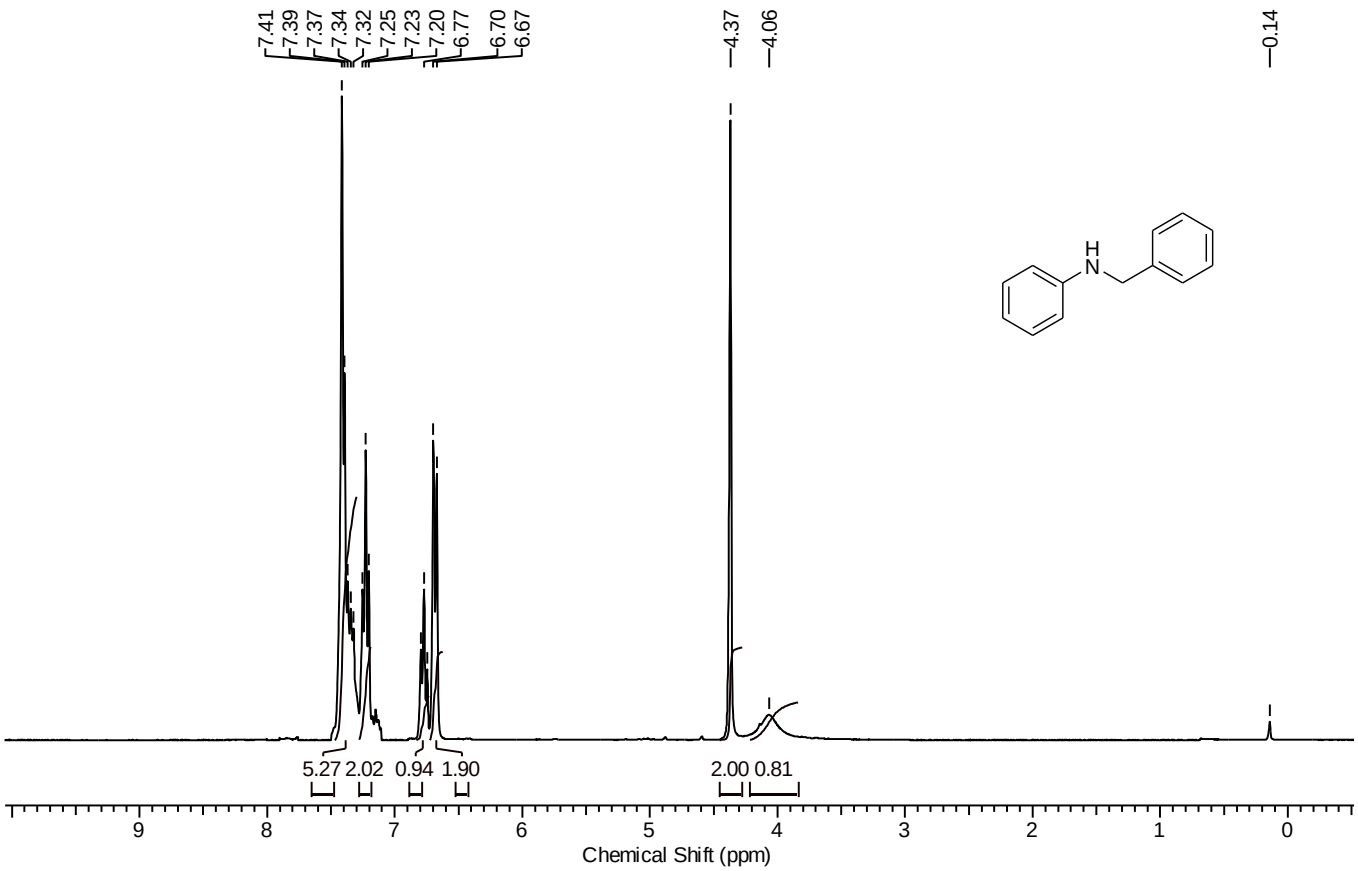
50
40
30
20
10
0


7.41
7.39
7.37
7.34
7.32
7.25
7.23
7.20
6.77
6.70
6.67
4.37
4.06
0.14
H
N
5.27
2.02
0.94
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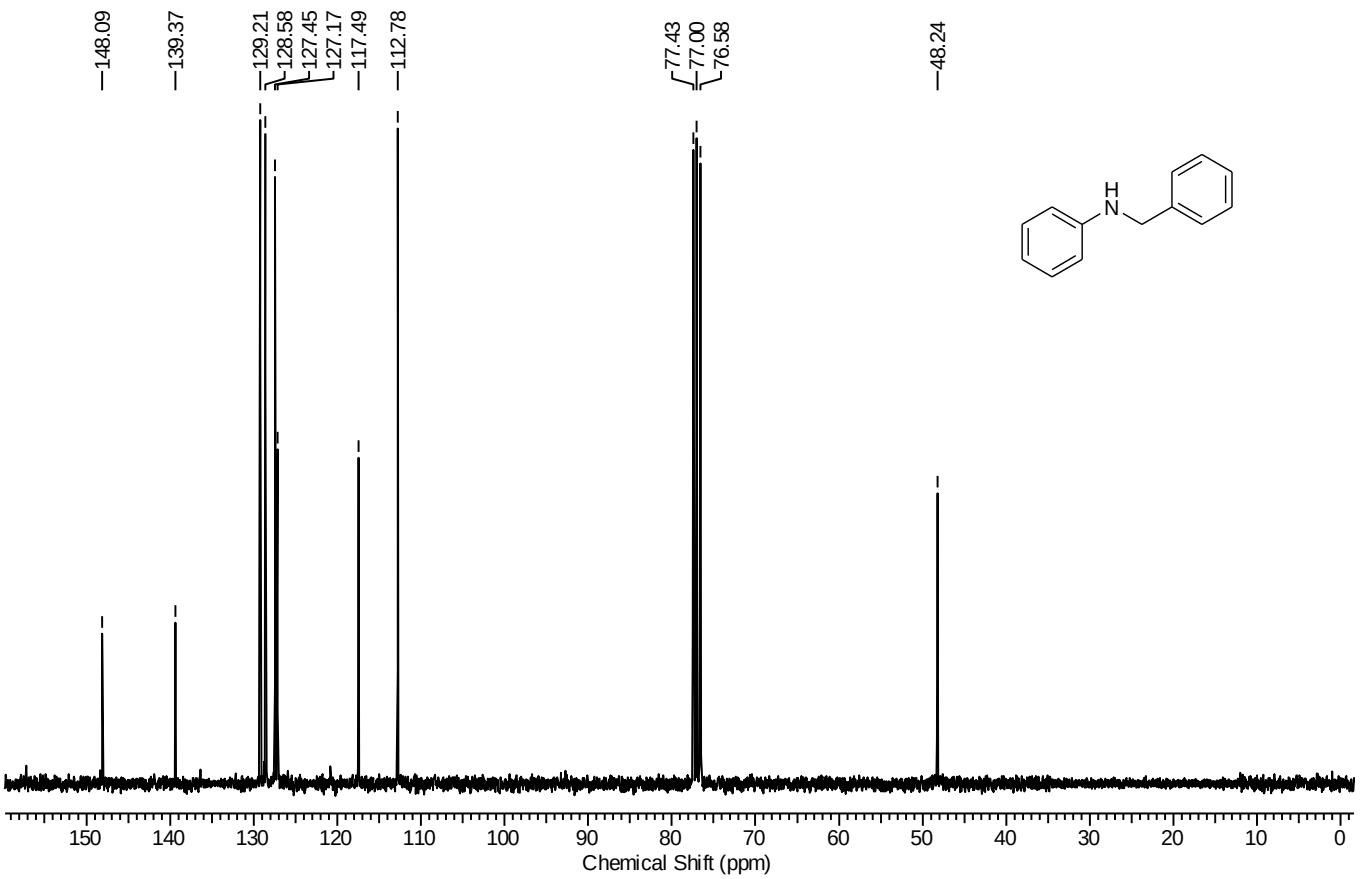
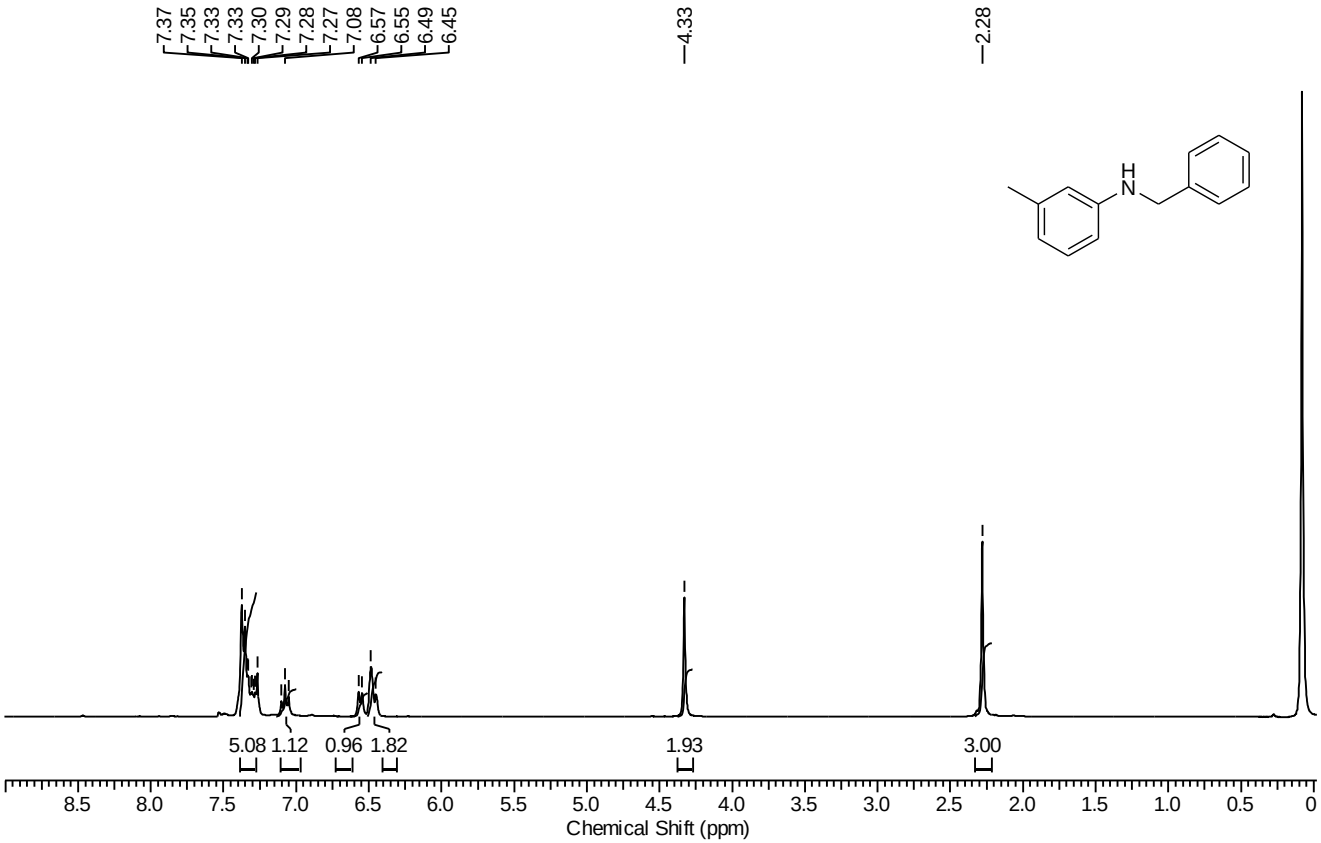
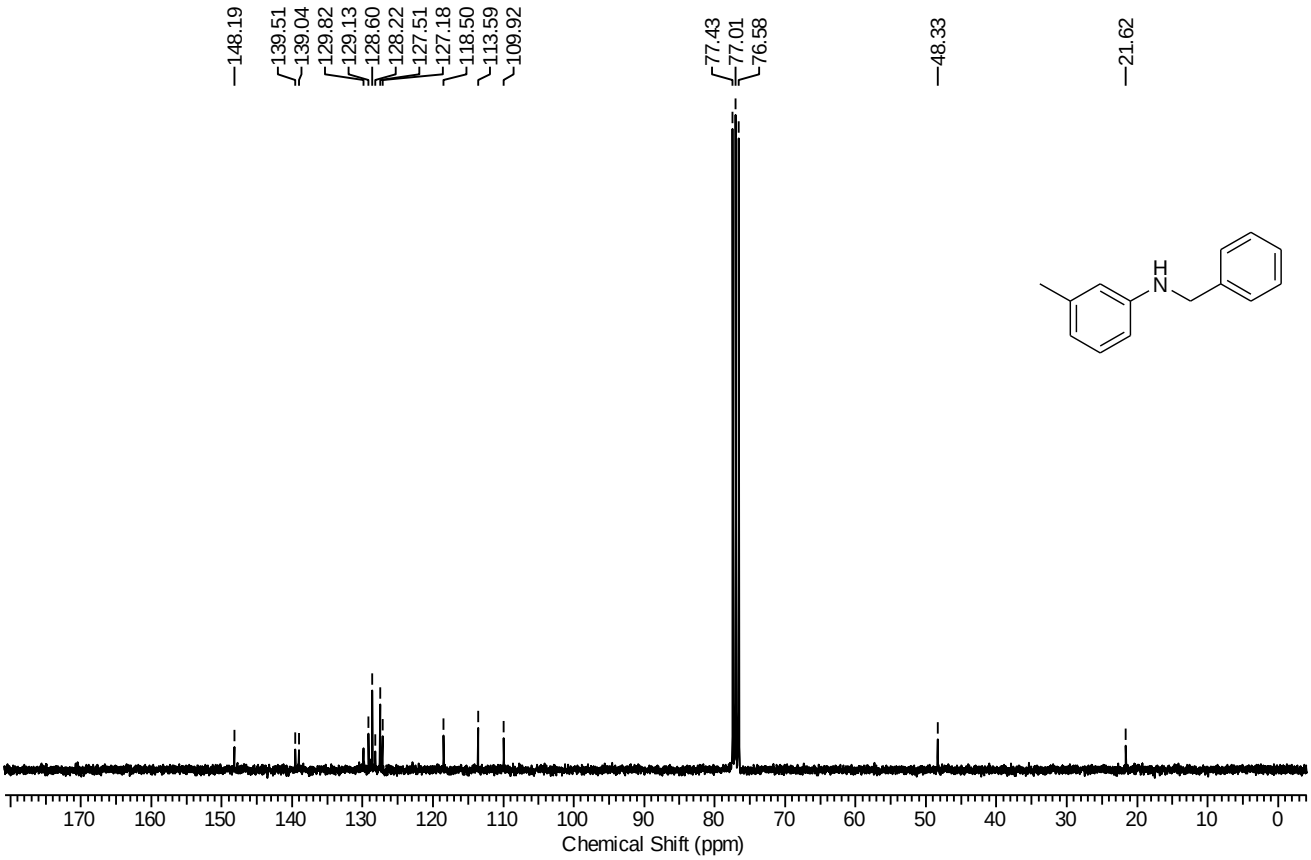
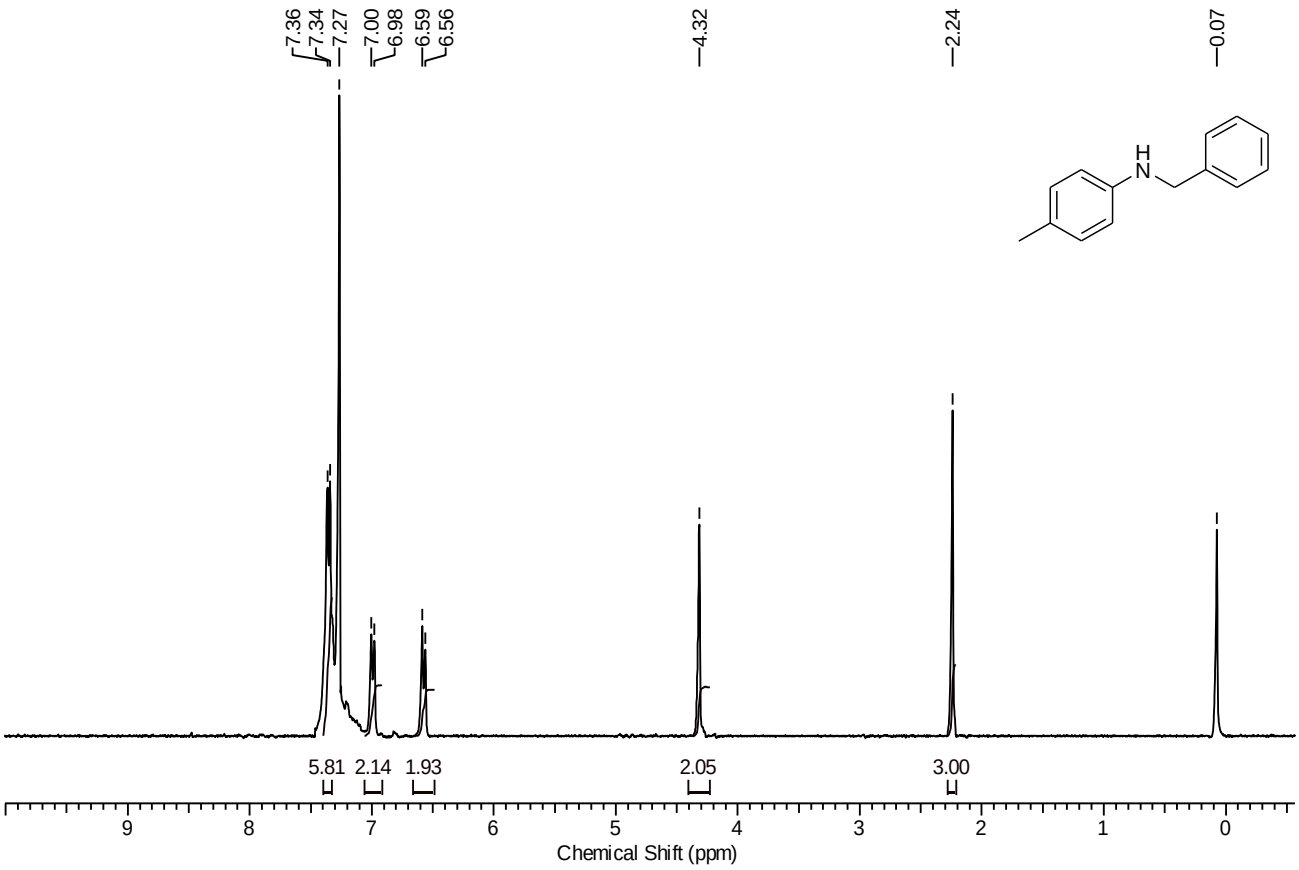
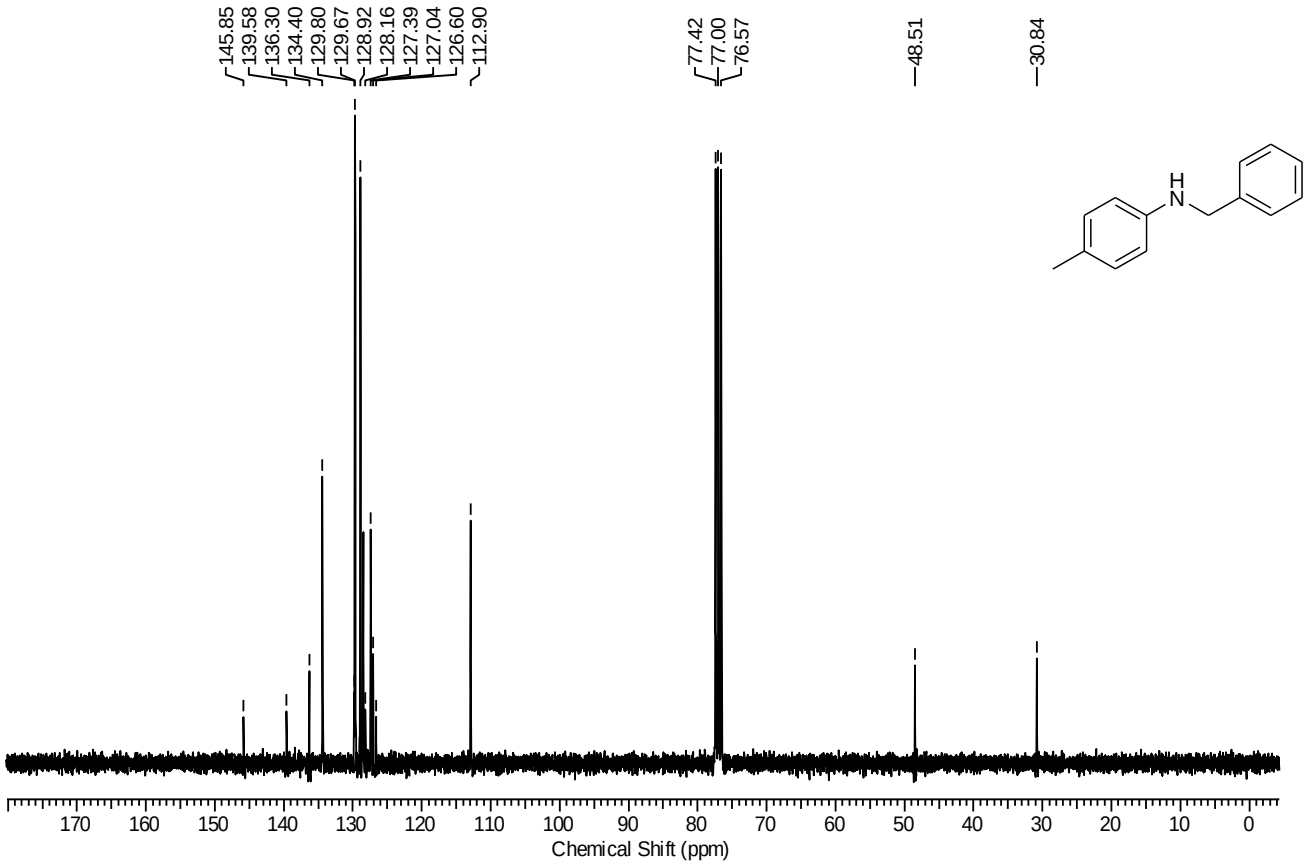
2.00
0.81
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

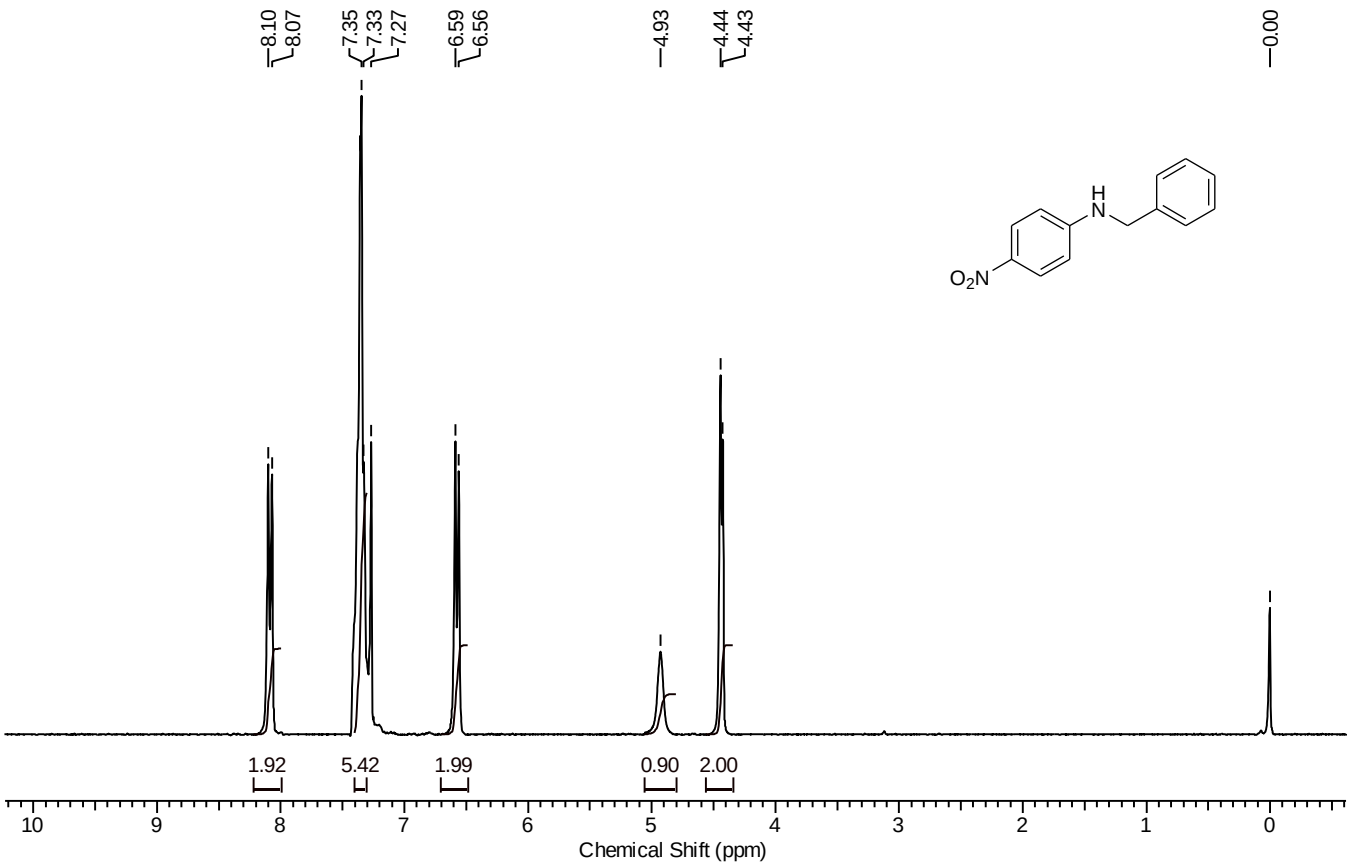

148.09
139.37
129.21
128.58
127.45
127.17
117.49
112.78
77.43
77.00
76.58
48.24
H
N
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

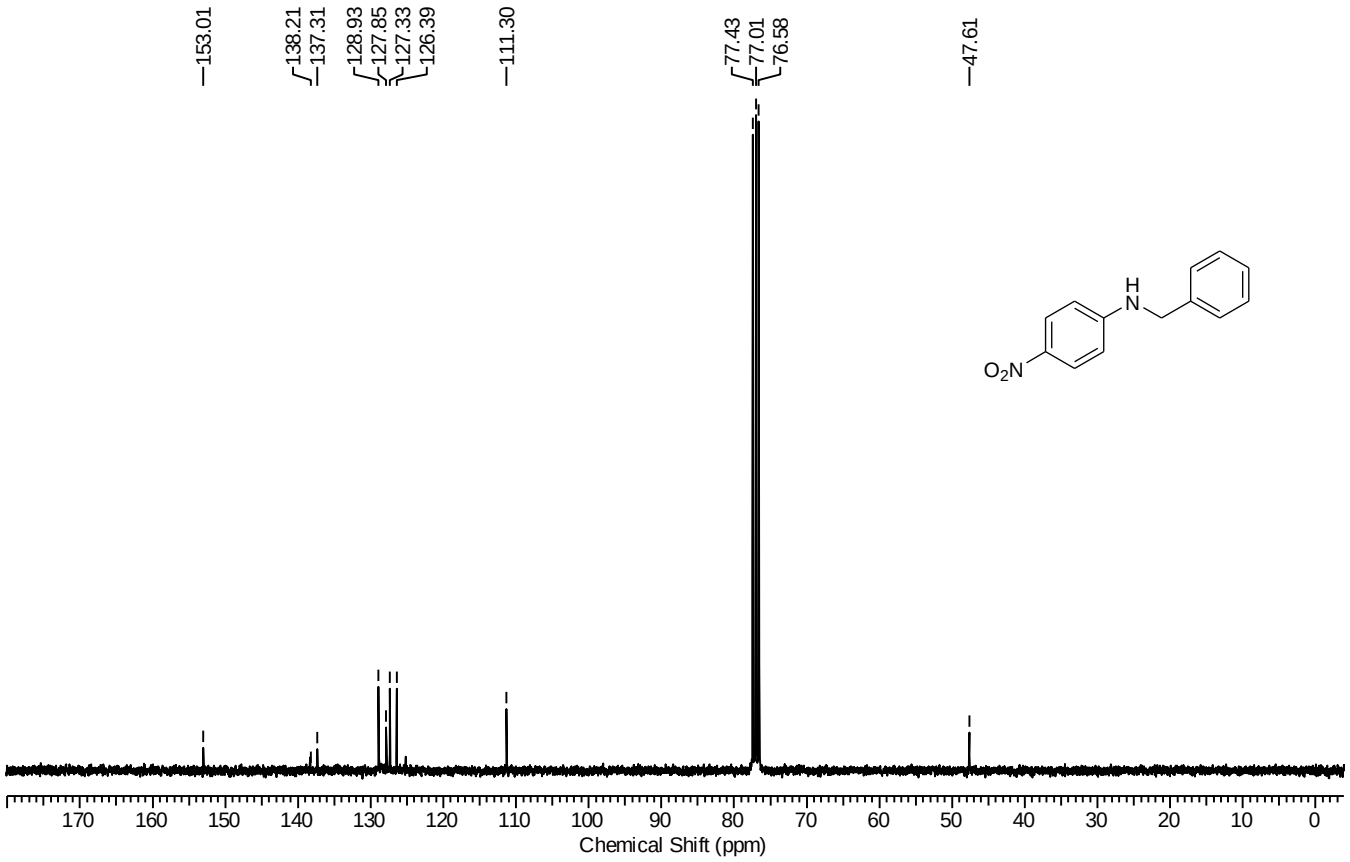

7.37
7.35
7.33
7.33
7.30
7.29
7.28
7.27
7.08
6.57
6.55
6.49
6.45
4.33
2.28
H
N
5.08
1.12
0.96
1.82
1.93
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
Chemical Shift (ppm)

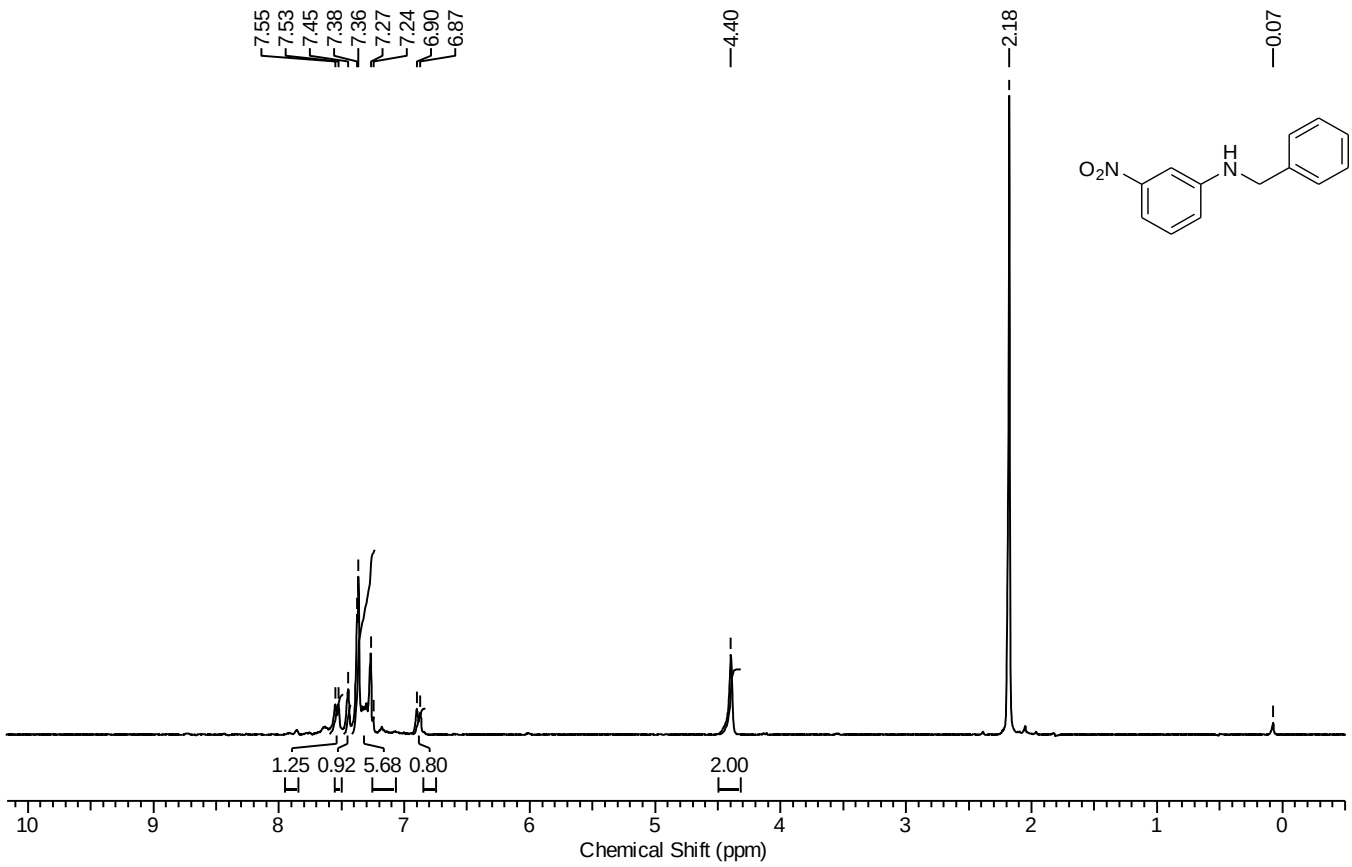

148.19
139.51
139.04
129.82
129.13
128.60
128.22
127.51
127.18
118.50
113.59
109.92
77.43
77.01
76.58
48.33
21.62
H
N
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
Chemical Shift (ppm)

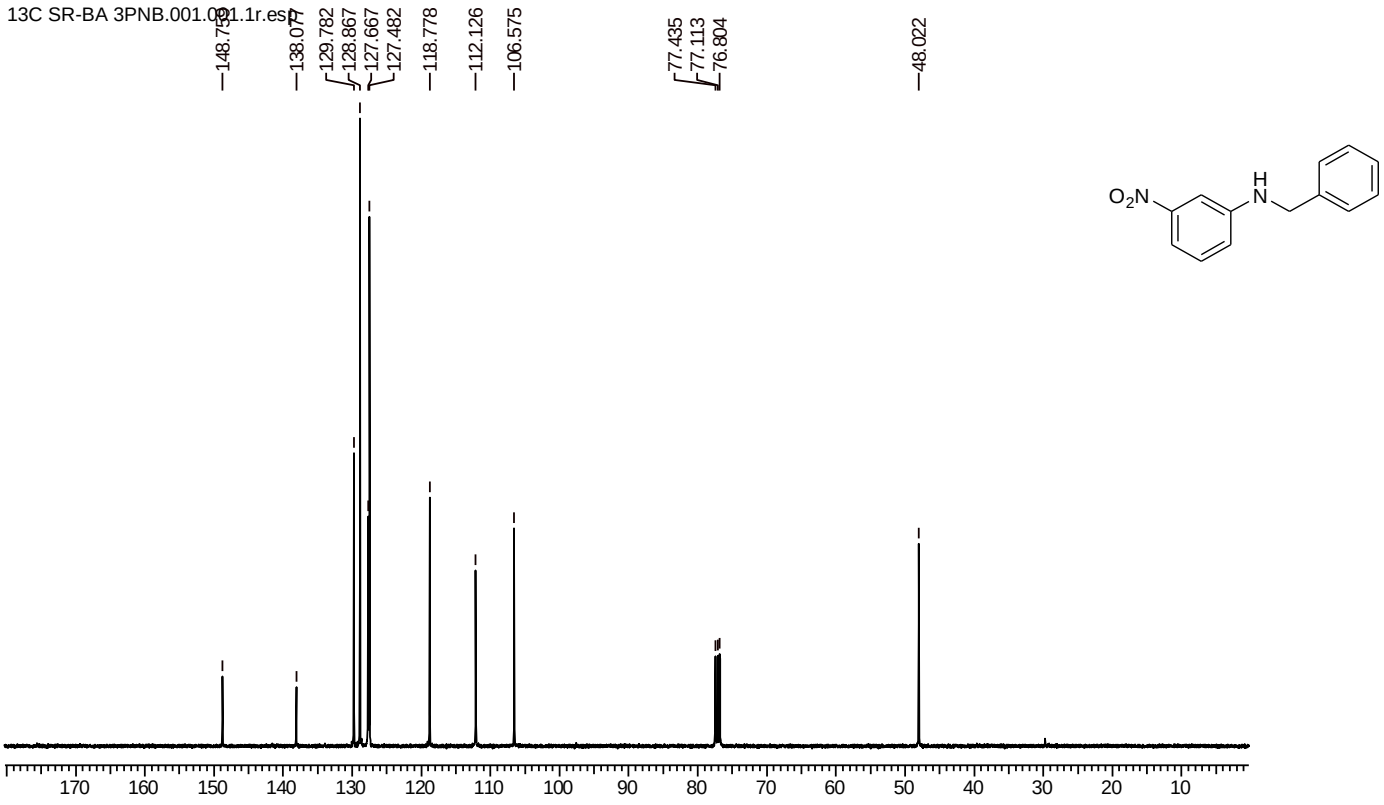

7.36
7.34
7.27
7.00
6.98
6.59
6.56
4.32
2.24
0.07
H
N
5.81
2.14
1.93
2.05
3.00
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

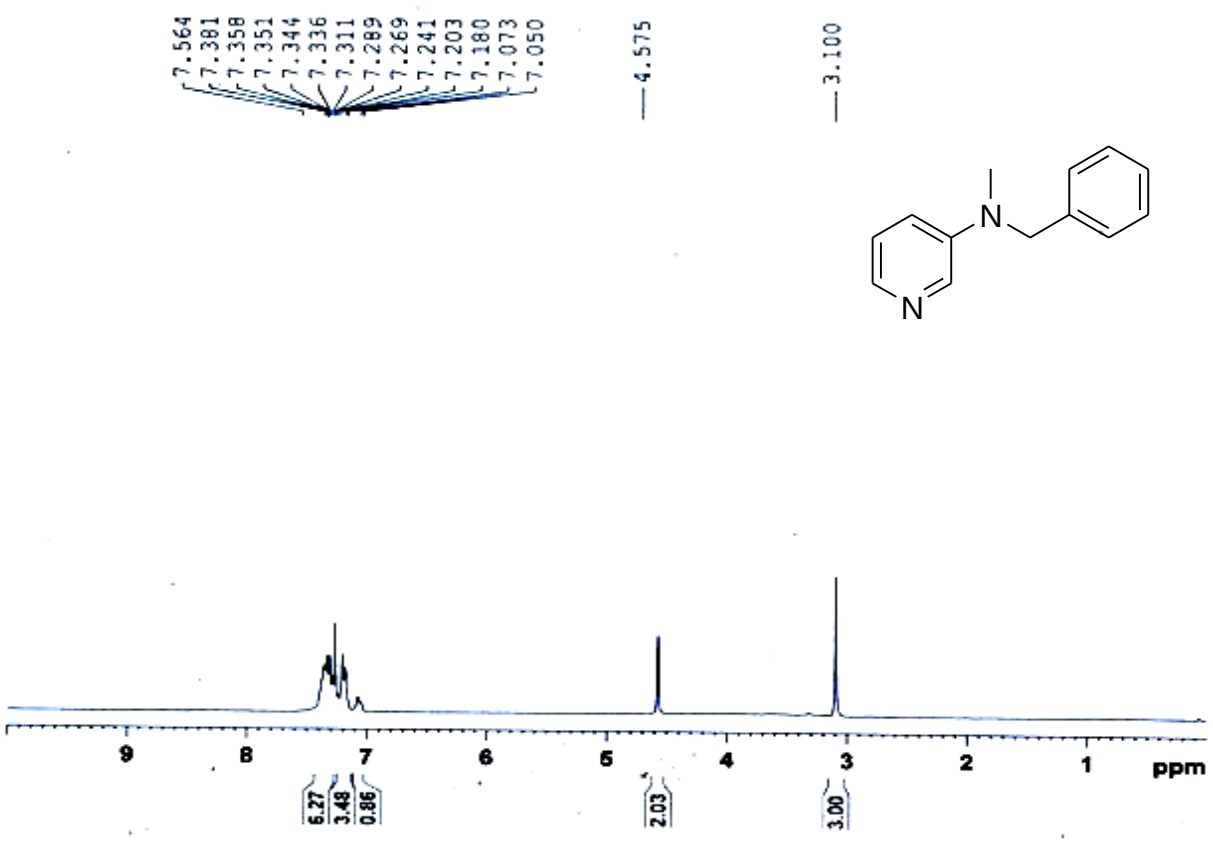

145.85
139.58
136.30
134.40
129.80
129.67
128.92
128.16
127.39
127.04
126.60
112.90
77.42
77.00
76.57
48.51
30.84
H
N
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

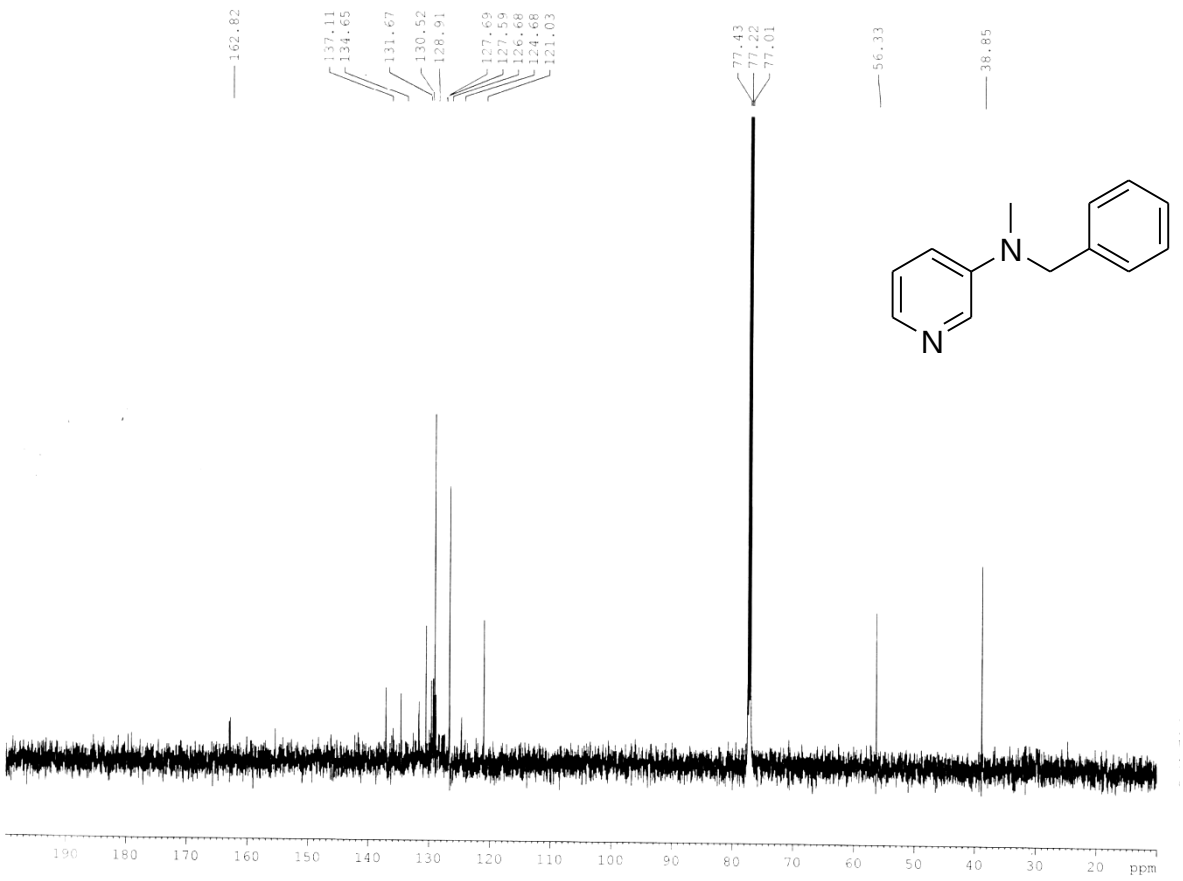

8.10
8.07
7.35
7.33
7.27
6.59
6.56
4.93
4.44
4.43
0.00
H
N
O2N
1.92
5.42
1.99
0.90
2.00
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

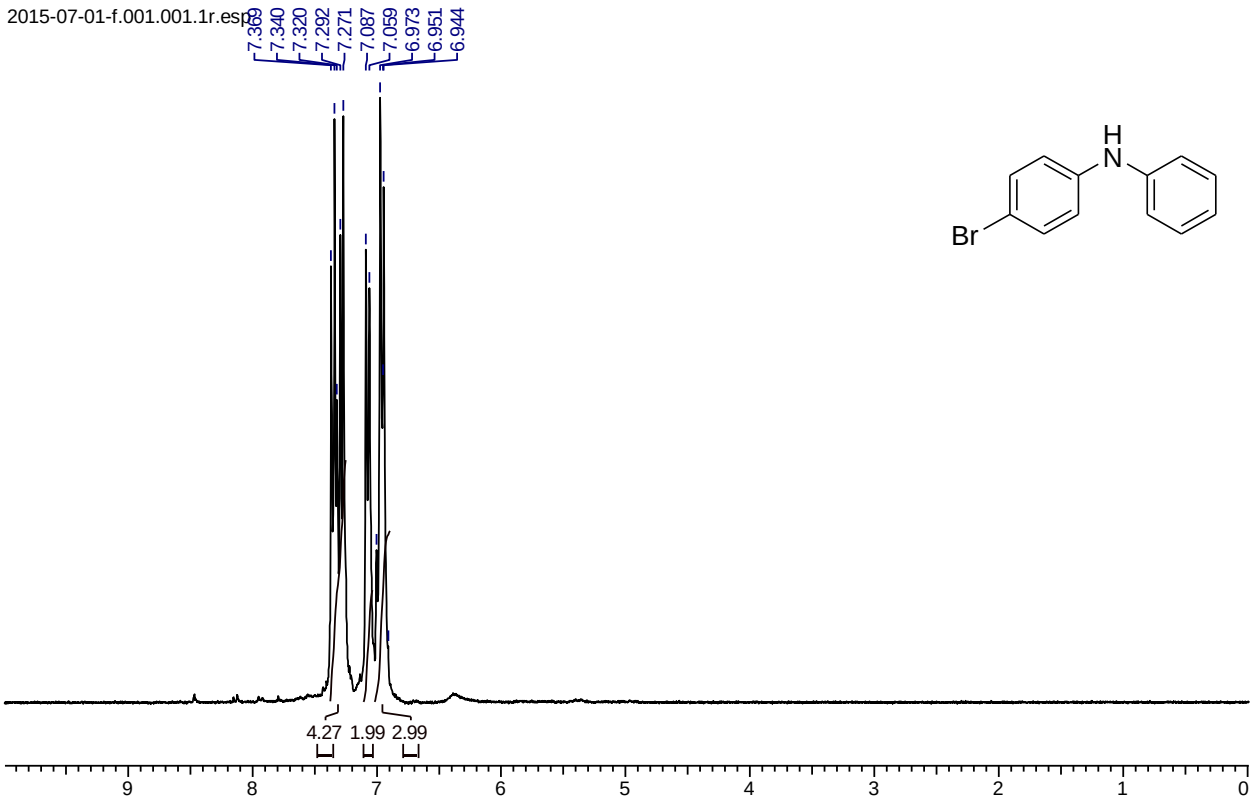

153.01
138.21
137.31
128.93
127.85
127.33
126.39
111.30
77.43
77.01
76.58
47.61
H
N
O2N
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)


7.55
7.53
7.45
7.38
7.36
7.27
7.24
6.90
6.87
4.40
2.18
0.07
O2N
H
N
1.25
0.92
5.68
0.80
2.00
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)


13C SR-BA 3PNB.001.001.1r.esp
148.759
138.077
129.782
128.867
127.667
127.482
118.778
112.126
106.575
77.435
77.113
76.804
48.022
O2N
H
N
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


7.564
7.381
7.358
7.351
7.344
7.336
7.311
7.289
7.269
7.241
7.203
7.180
7.073
7.050
4.575
3.100
N
N
9
8
7
6
5
4
3
2
1
ppm
6.27
3.48
0.86
2.03
3.00


162.82
137.11
134.65
131.67
130.52
128.91
127.69
127.59
126.68
124.68
121.03
77.43
77.22
77.01
56.33
38.85
N
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


2015-07-01-f.001.001.1r.esp
7.369
7.340
7.320
7.292
7.271
7.087
7.059
6.973
6.951
6.944
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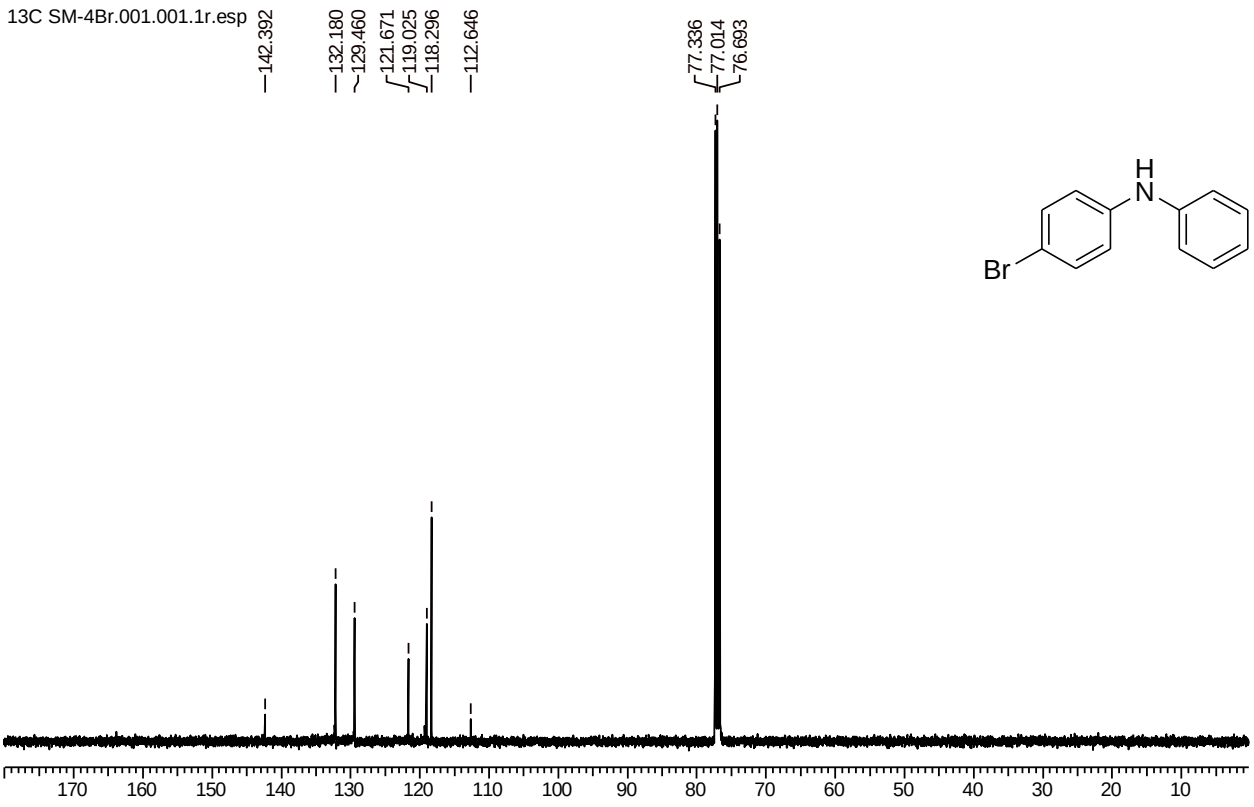
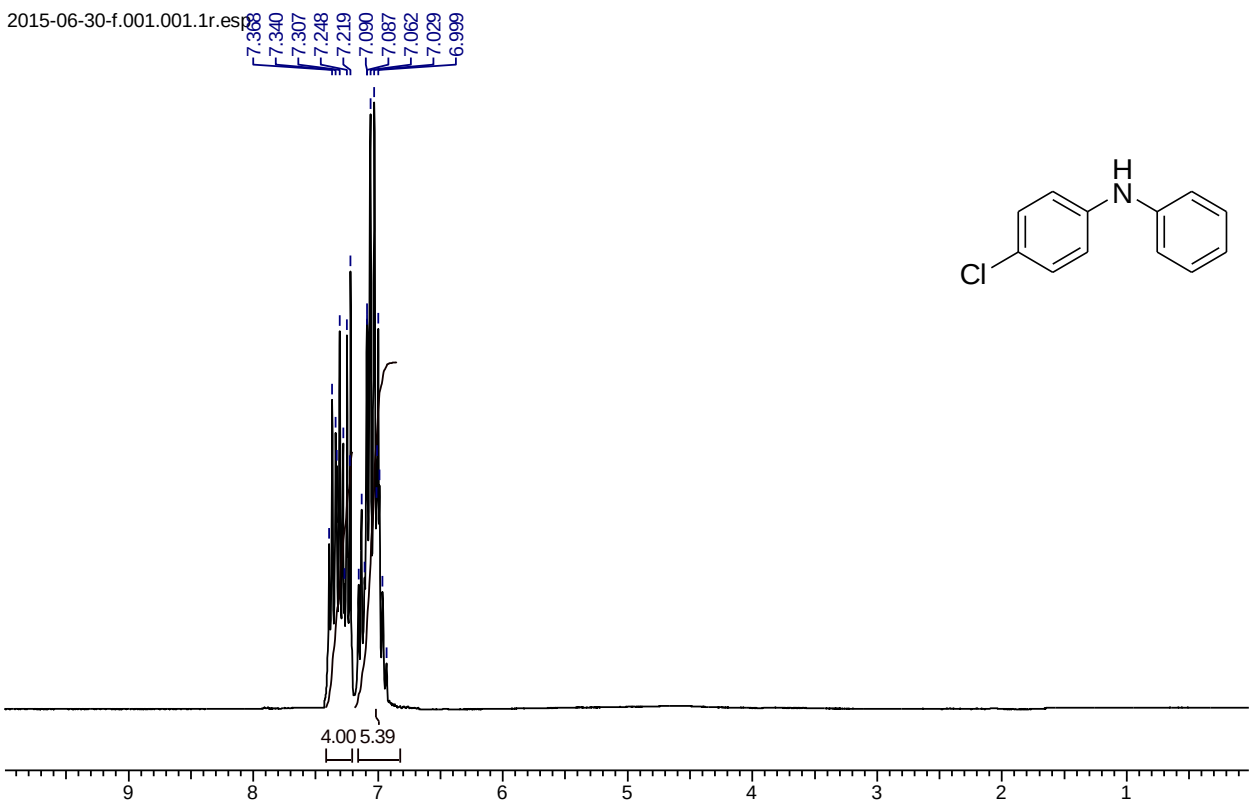
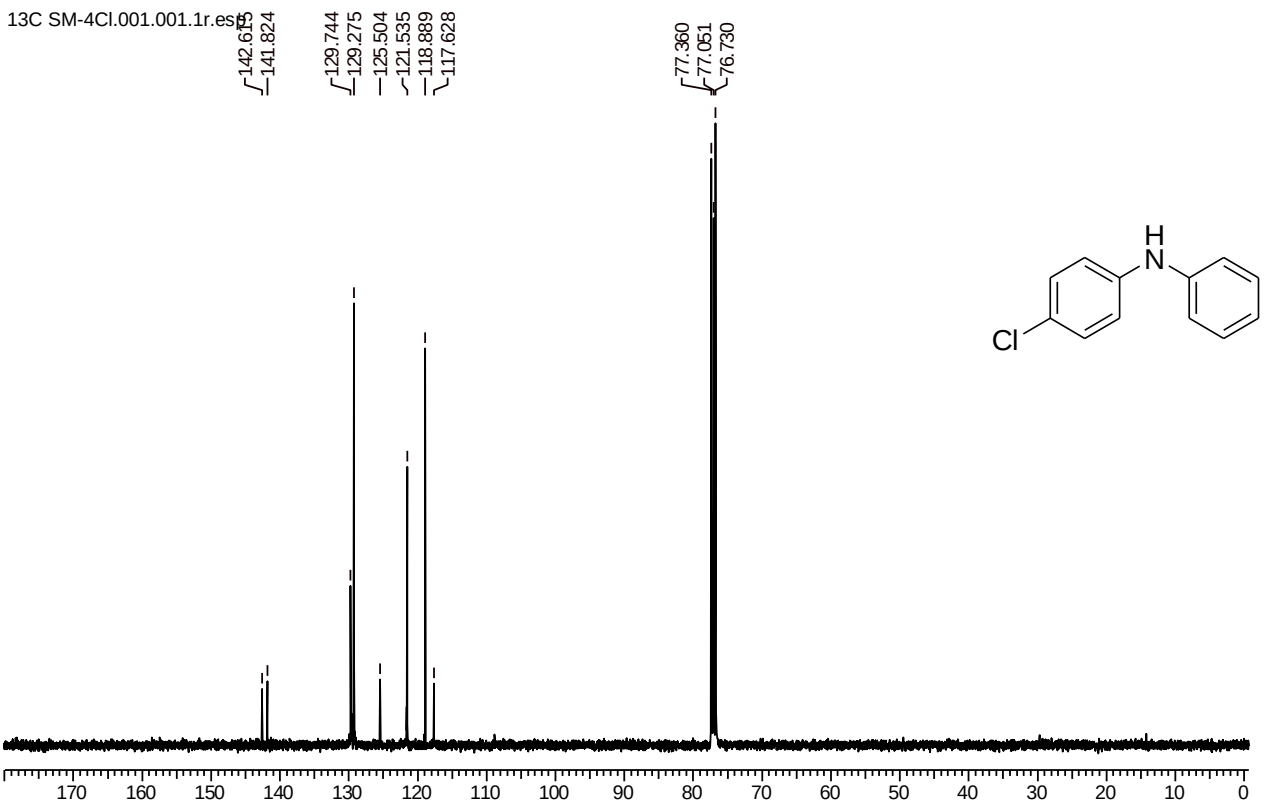
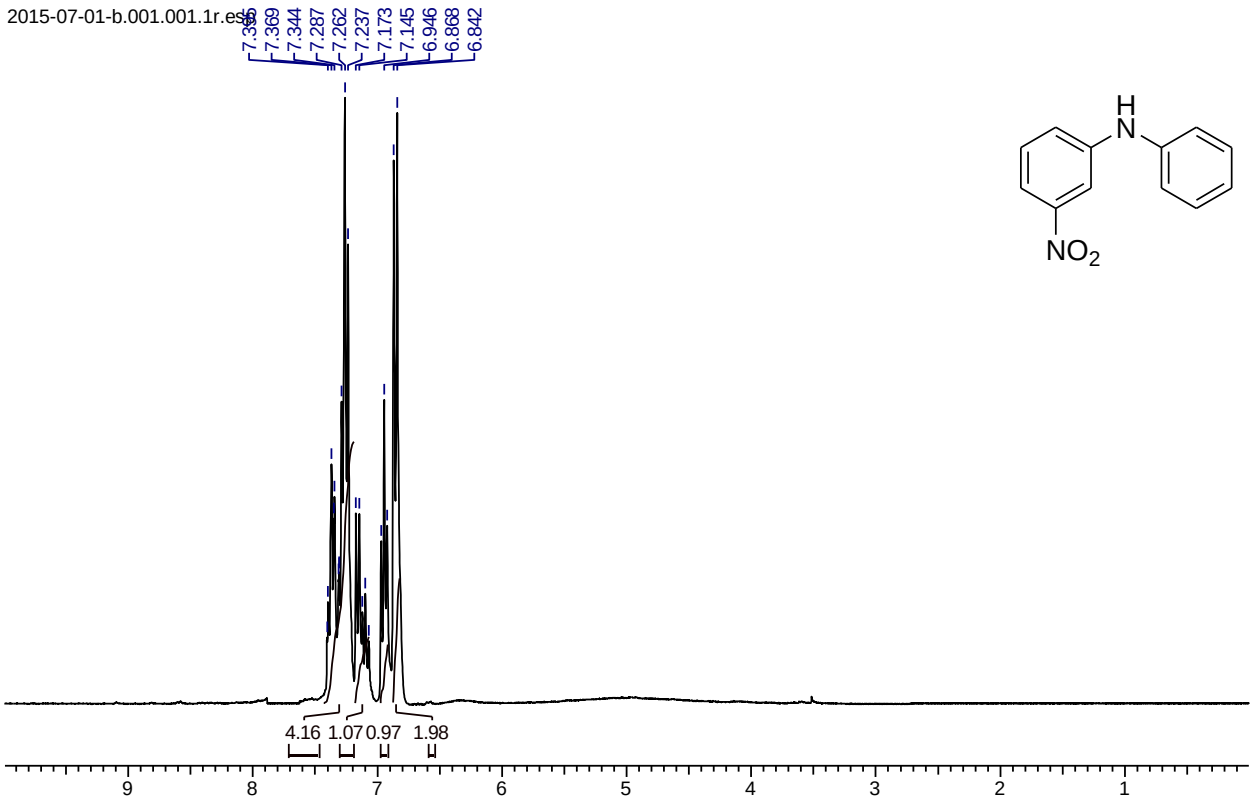
N
Br
4.27
1.99
2.99
9
8
7
6
5
4
3
2
1
0


13C SM-4Br.001.001.1r.esp
142.392
132.180
129.460
121.671
119.025
118.296
112.646
77.336
77.014
76.693
H
N
Br
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


2015-06-30-f.001.001.1r.esp
7.365
7.340
7.307
7.248
7.219
7.090
7.087
7.062
7.029
6.999
H
N
Cl
4.00
5.39
9
8
7
6
5
4
3
2
1


13C SM-4Cl.001.001.1r.esp
142.615
141.824
129.744
129.275
125.504
121.535
118.889
117.628
77.360
77.051
76.730
H
N
Cl
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


2015-07-01-b.001.001.1r.esp
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7.369
7.344
7.287
7.262
7.237
7.173
7.145
6.946
6.868
6.842
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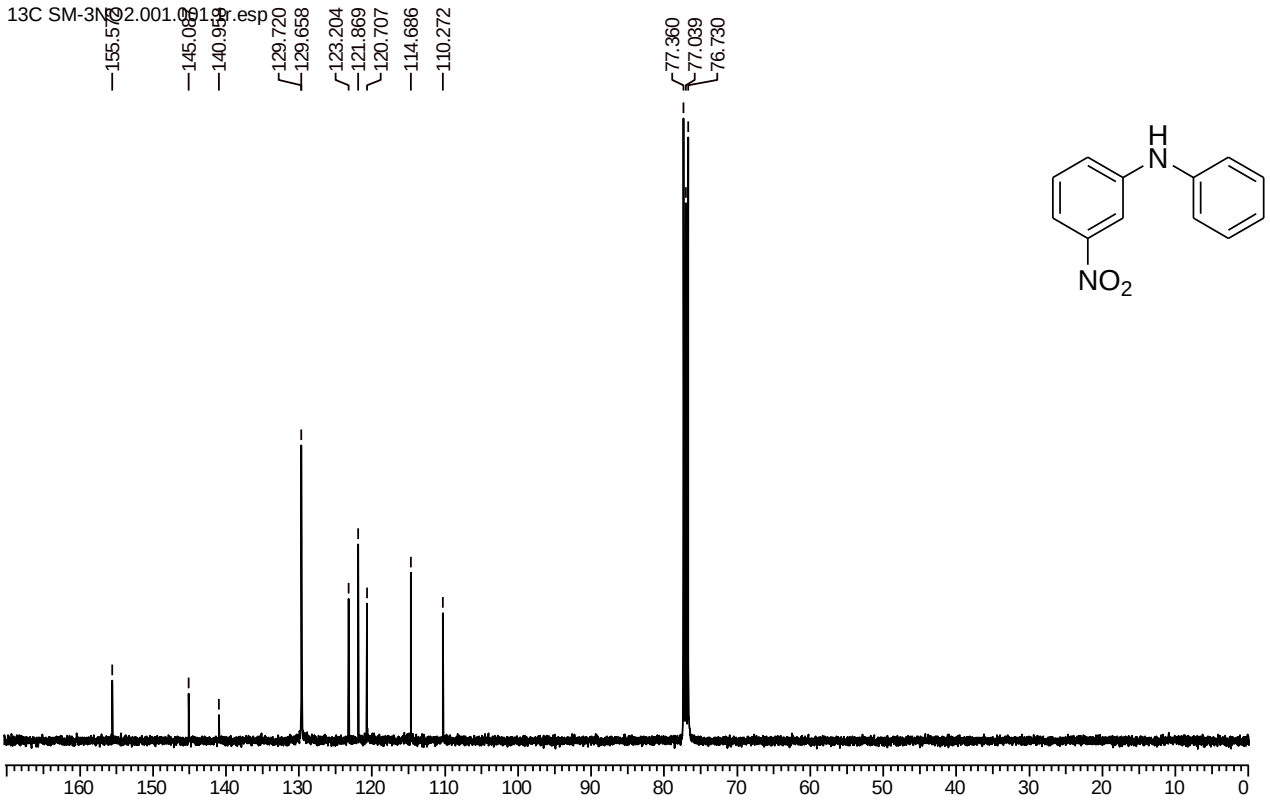
N
NO2
4.16
1.07
0.97
1.98
9
8
7
6
5
4
3
2
1


13C SM-3N-02.001.001r.esp
155.572
145.082
140.959
129.720
129.658
123.204
121.869
120.707
114.686
110.272
77.360
77.039
76.730
H
N
NO2
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
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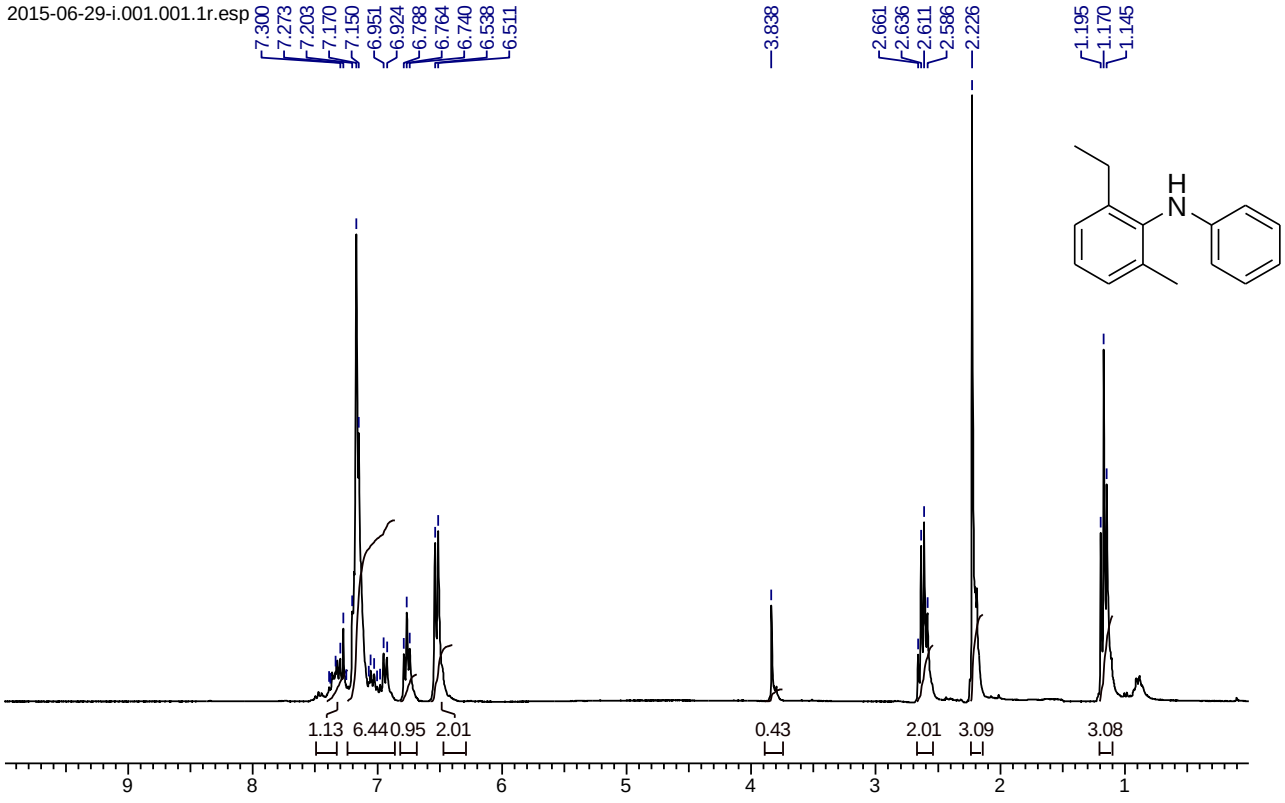
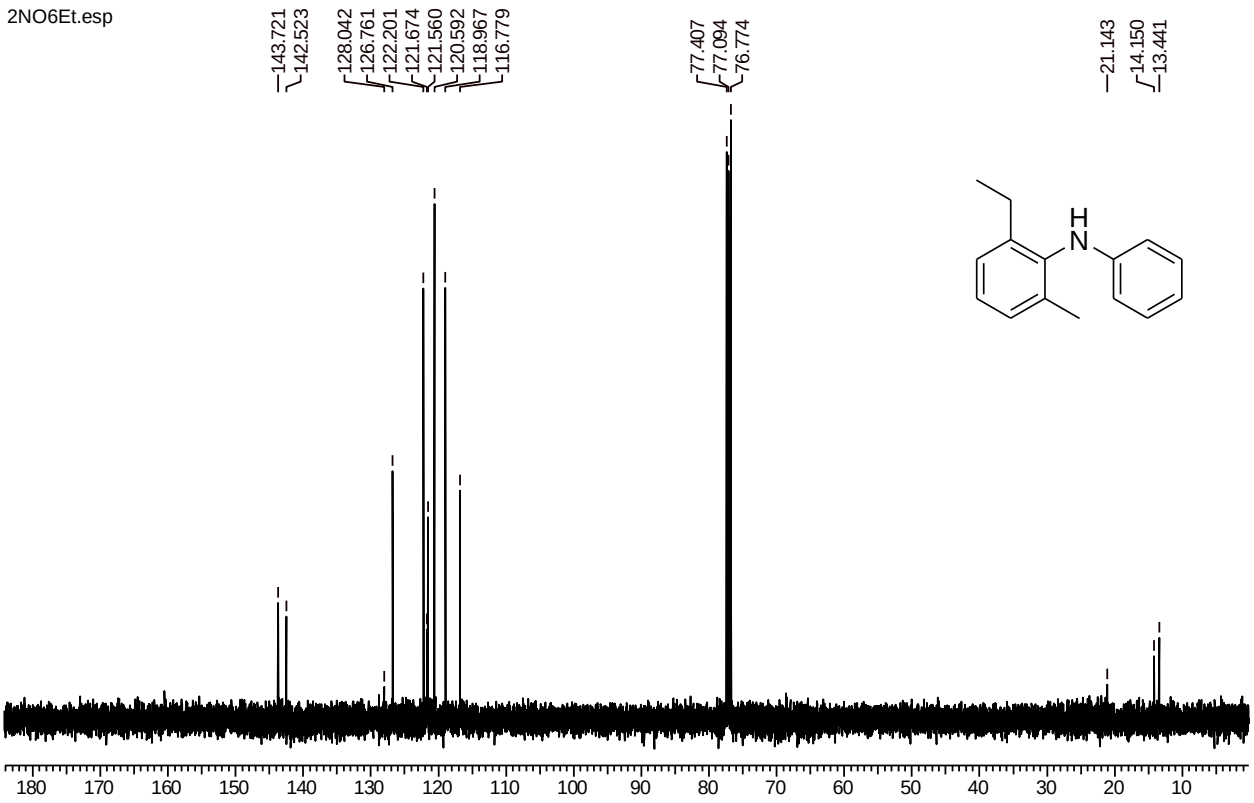

2015-06-29-i.001.001.1r.esp
7.300
7.273
7.203
7.170
7.150
6.951
6.924
6.788
6.764
6.740
6.538
6.511
3.838
2.661
2.636
2.611
2.586
2.226
1.195
1.170
1.145
H
N
1.13
6.44
0.95
2.01
0.43
2.01
3.09
3.08
9
8
7
6
5
4
3
2
1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128.042
126.761
122.201
121.674
121.560
120.592
118.967
116.779
77.407
77.094
76.774
21.143
14.150
13.441
H
N
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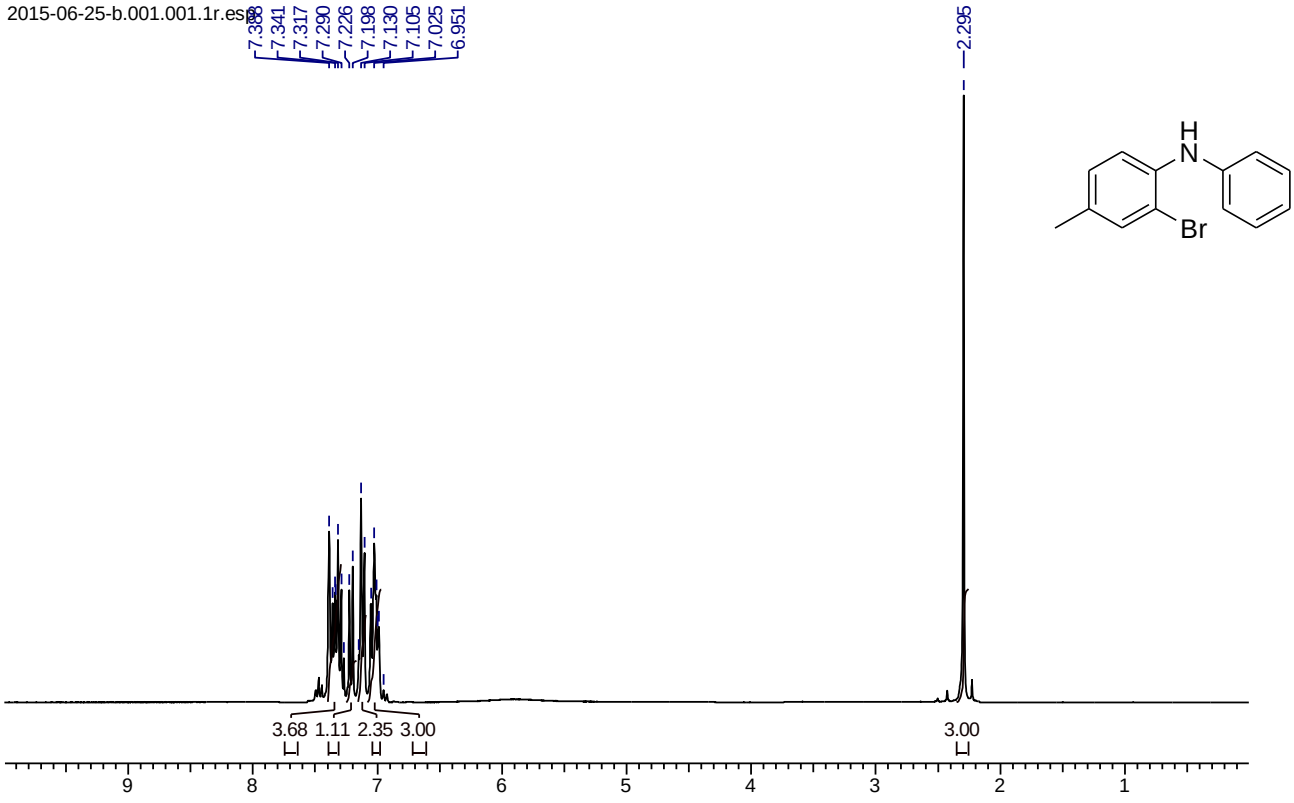
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


2015-06-25-b.001.001.1r.esp
7.341
7.317
7.290
7.226
7.198
7.130
7.105
7.025
6.951
2.295
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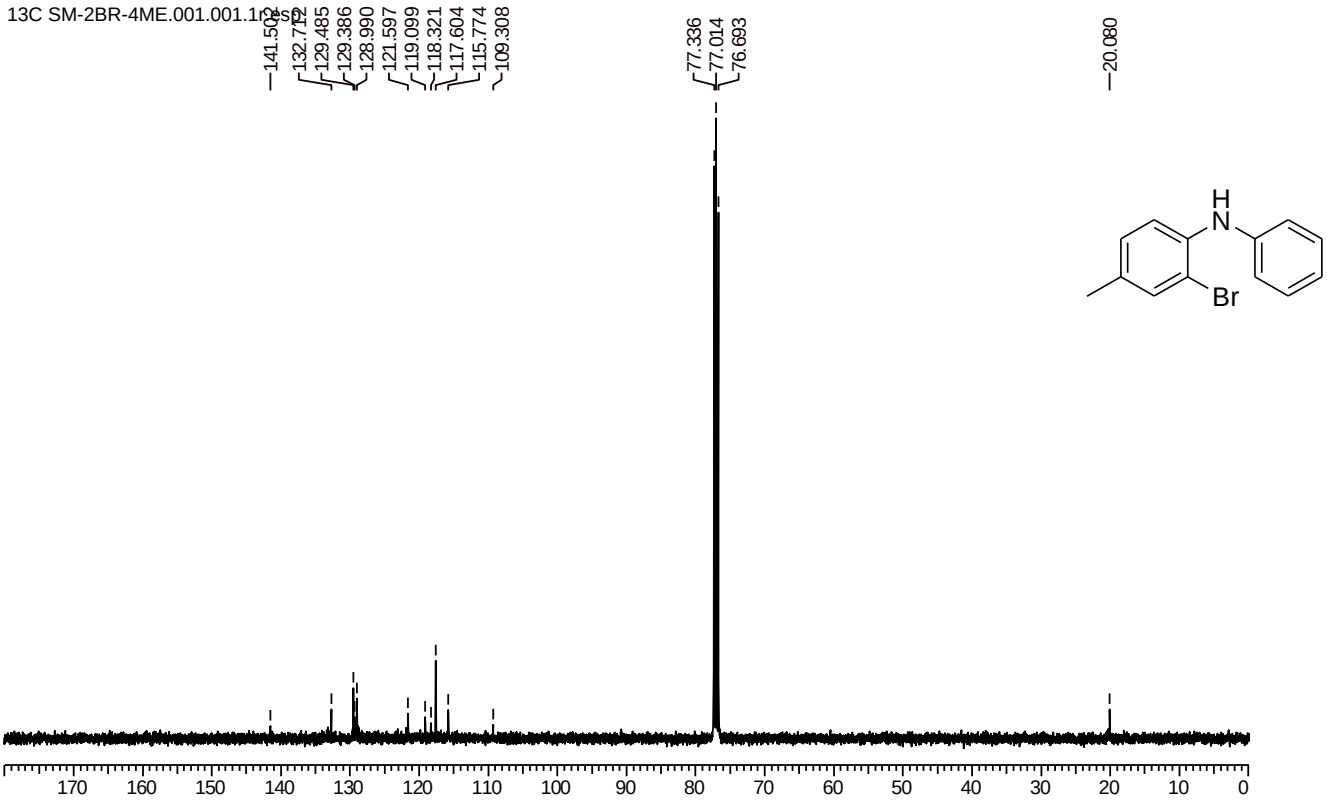
1.11
2.35
3.00
3.00
9
8
7
6
5
4
3
2
1


13C SM-2BR-4ME.001.001.1.2.esp
141.509
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129.386
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121.597
119.099
118.321
117.604
115.774
109.308
77.336
77.014
76.693
20.080
H
N
Br
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160
150
140
130
120
110
100
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80
70
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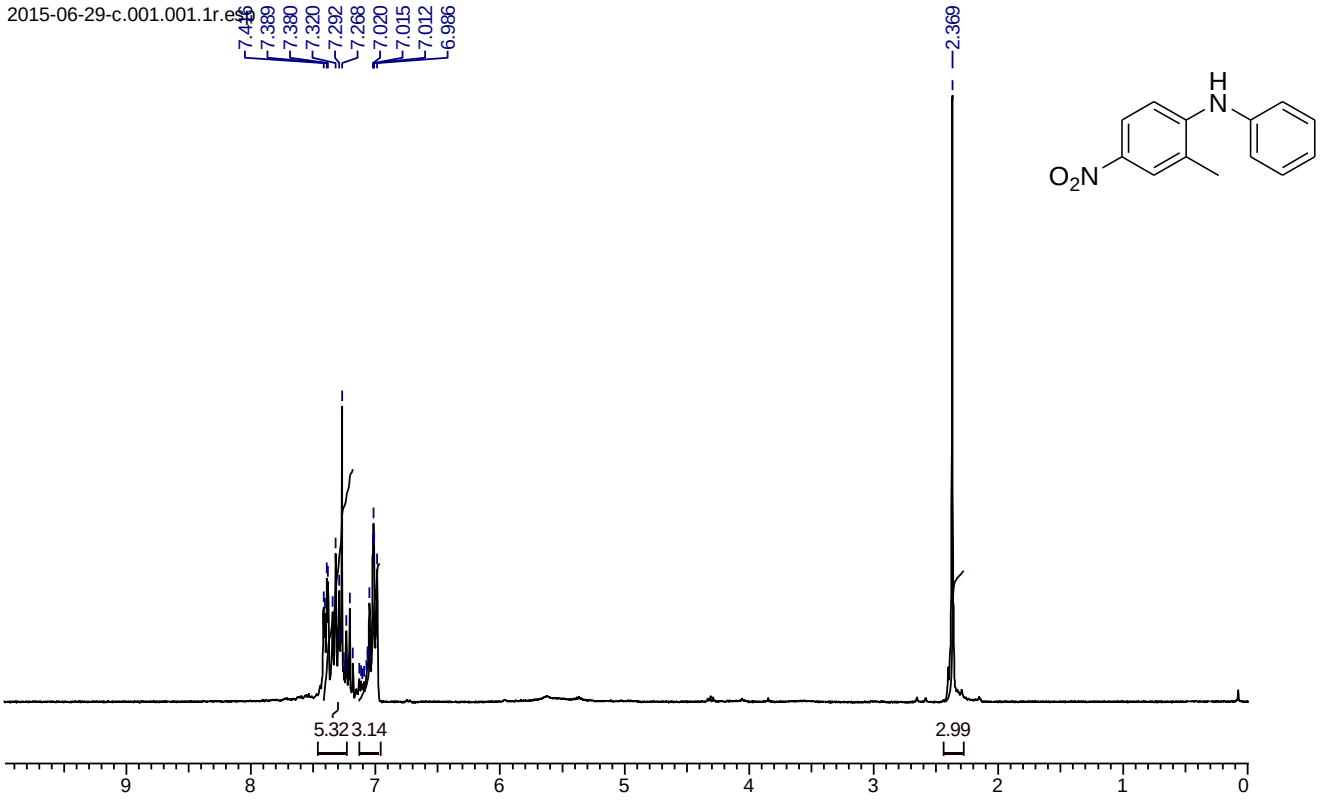
50
40
30
20
10
0


2015-06-29-c.001.001.1r.esp
7.445
7.389
7.380
7.320
7.292
7.268
7.020
7.015
7.012
6.986
2.369
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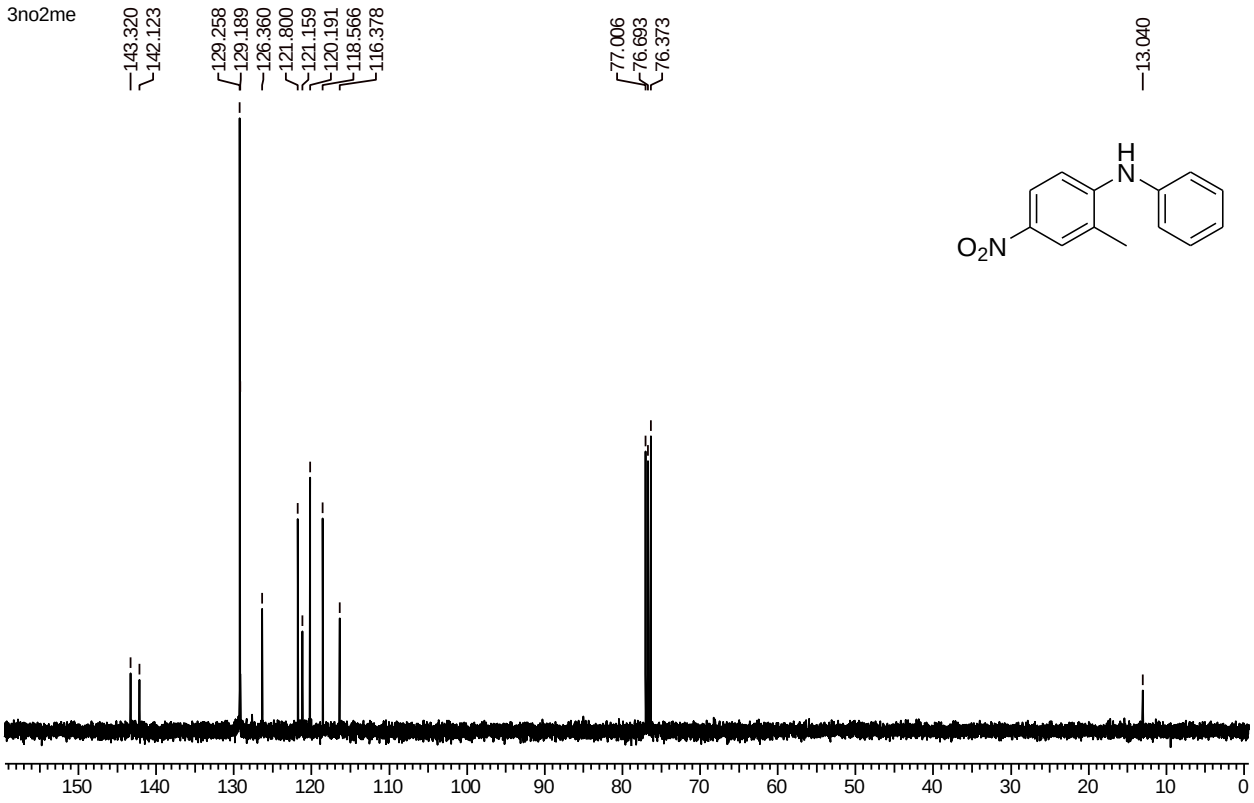
O2N
5.32
3.14
2.99
9
8
7
6
5
4
3
2
1
0


3no2me
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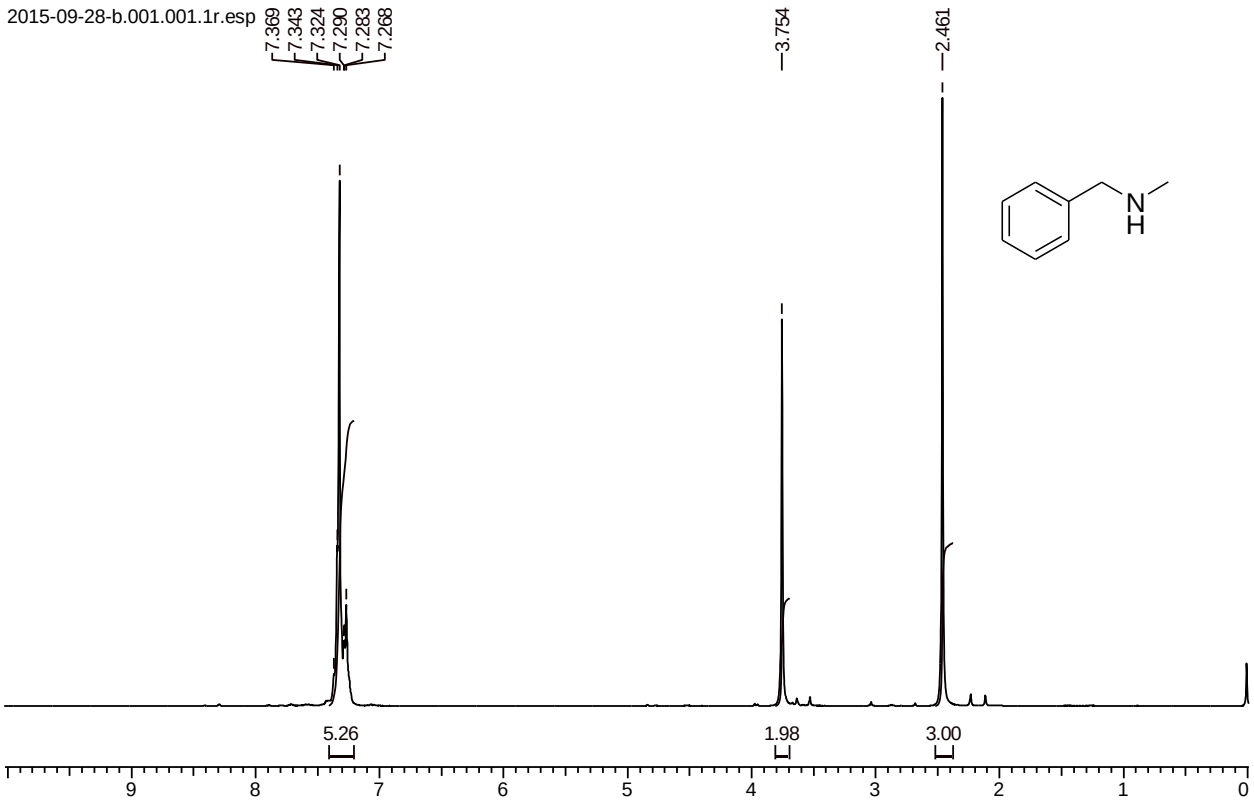
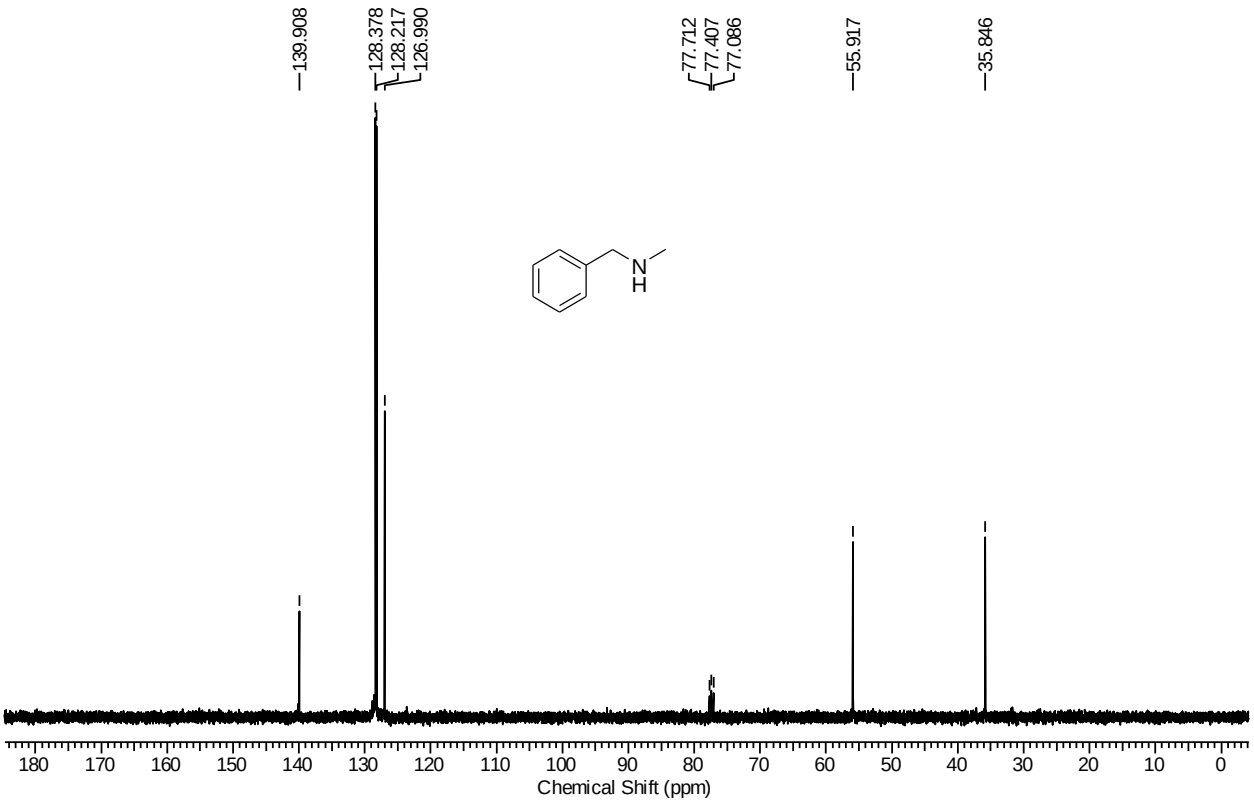
142.123
129.258
129.189
126.360
121.800
121.159
120.191
118.566
116.378
77.006
76.693
76.373
13.040
H
N
O2N
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


2015-09-28-b.001.001.1r.esp
7.369
7.343
7.324
7.290
7.283
7.268
3.754
2.461
N
H
5.26
1.98
3.00
9
8
7
6
5
4
3
2
1
0


139.908
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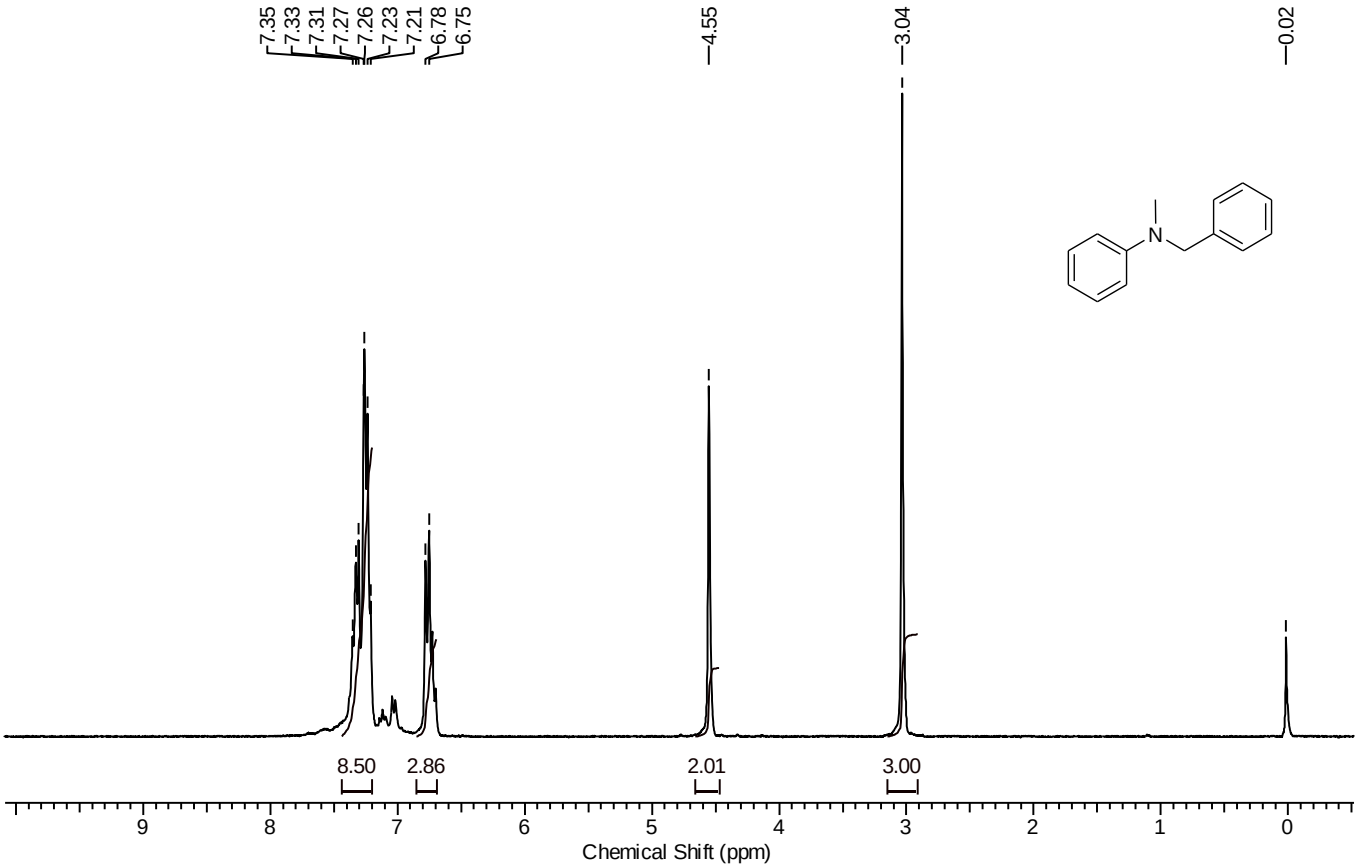
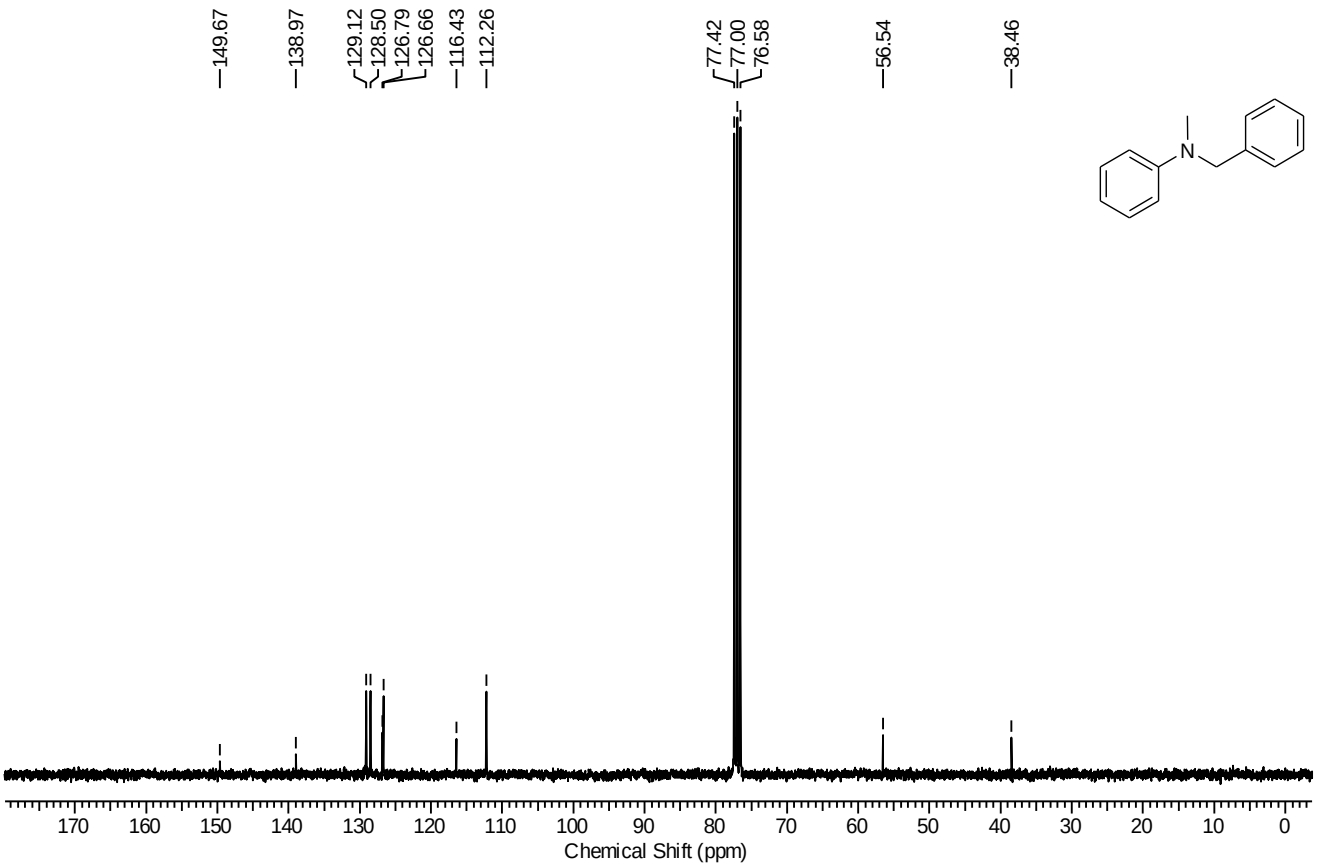
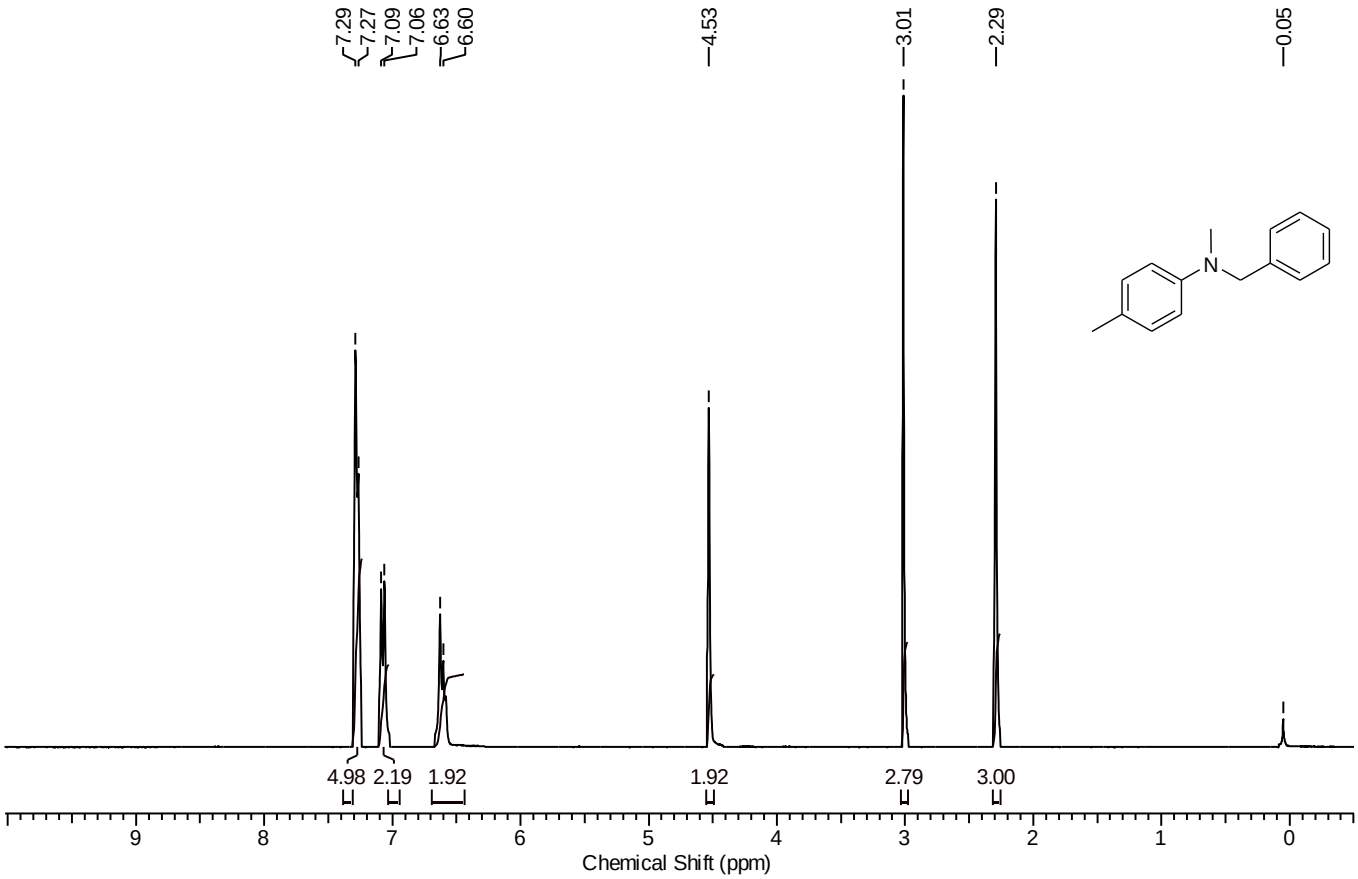
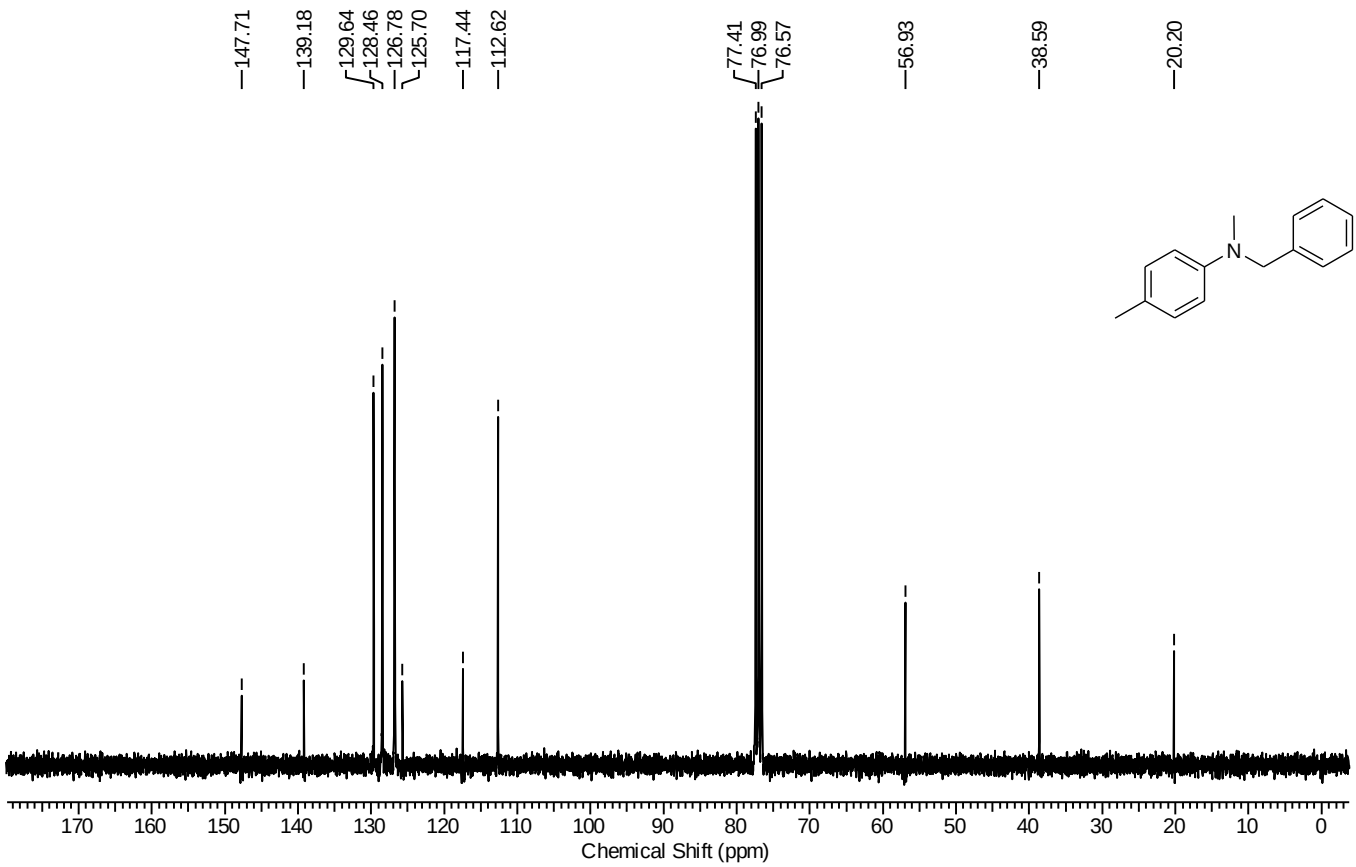
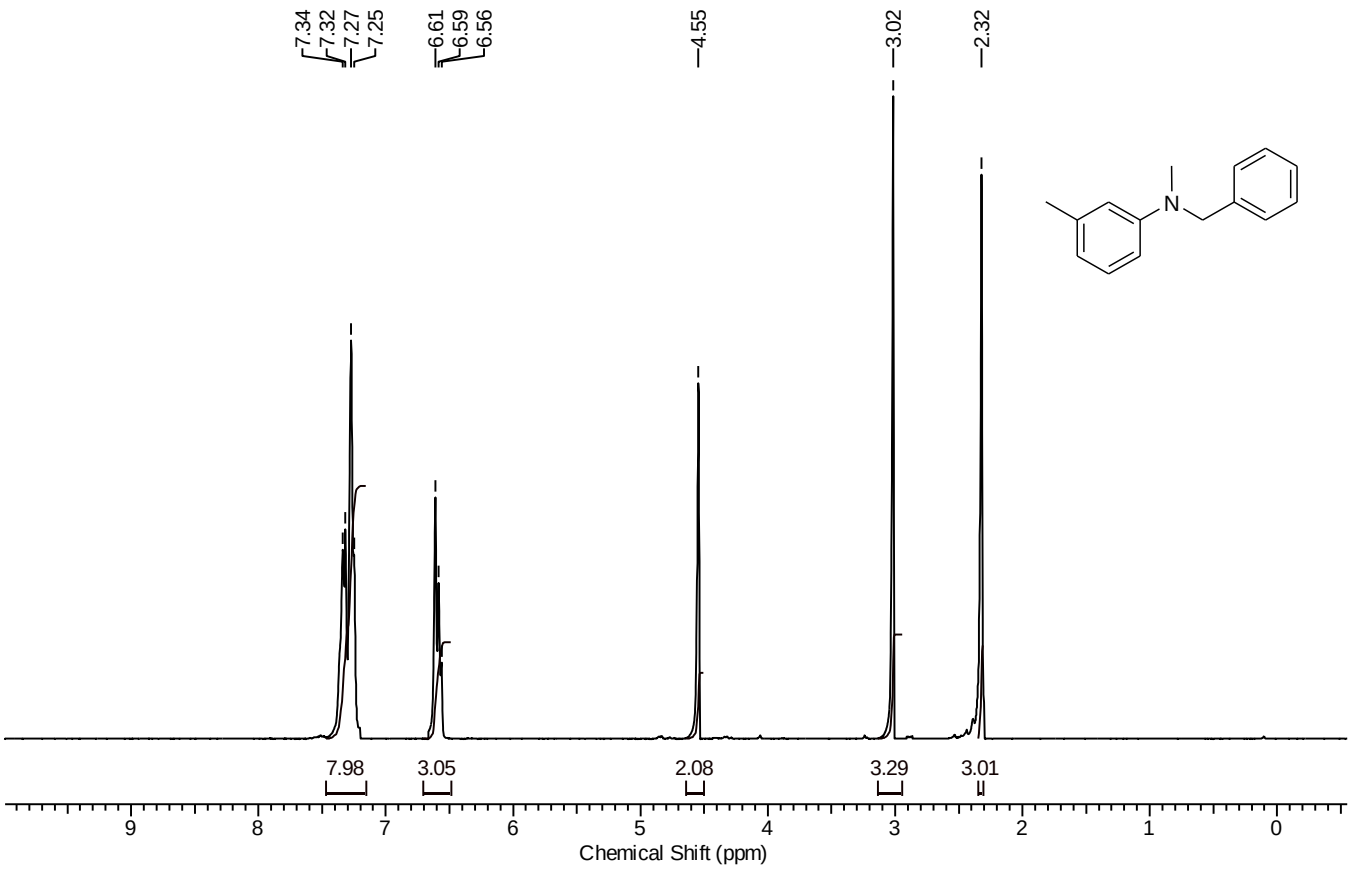
128.217
126.990
77.712
77.407
77.086
55.917
35.846
N
H
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

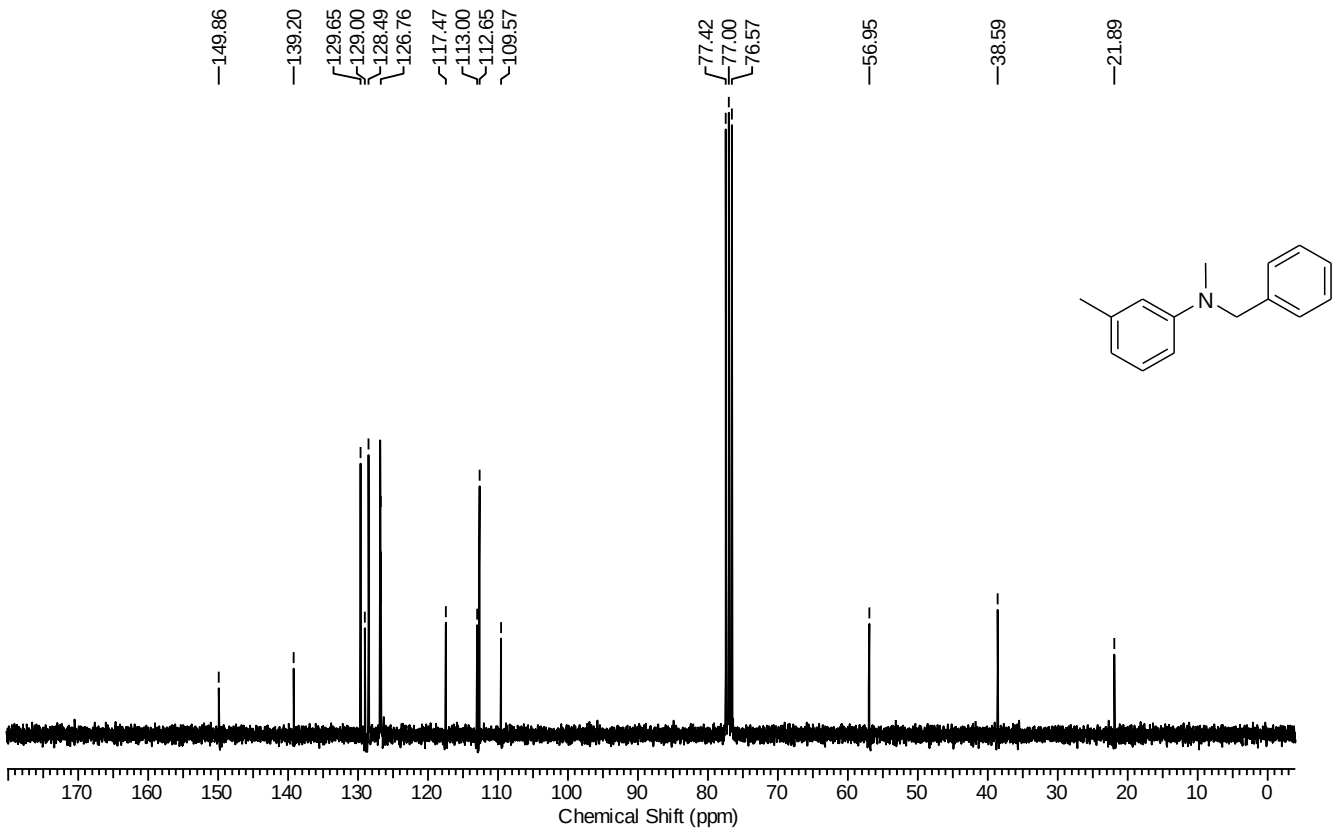

7.35
7.33
7.31
7.27
7.26
7.23
7.21
6.78
6.75
4.55
3.04
0.02
N
8.50
2.86
2.01
3.00
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

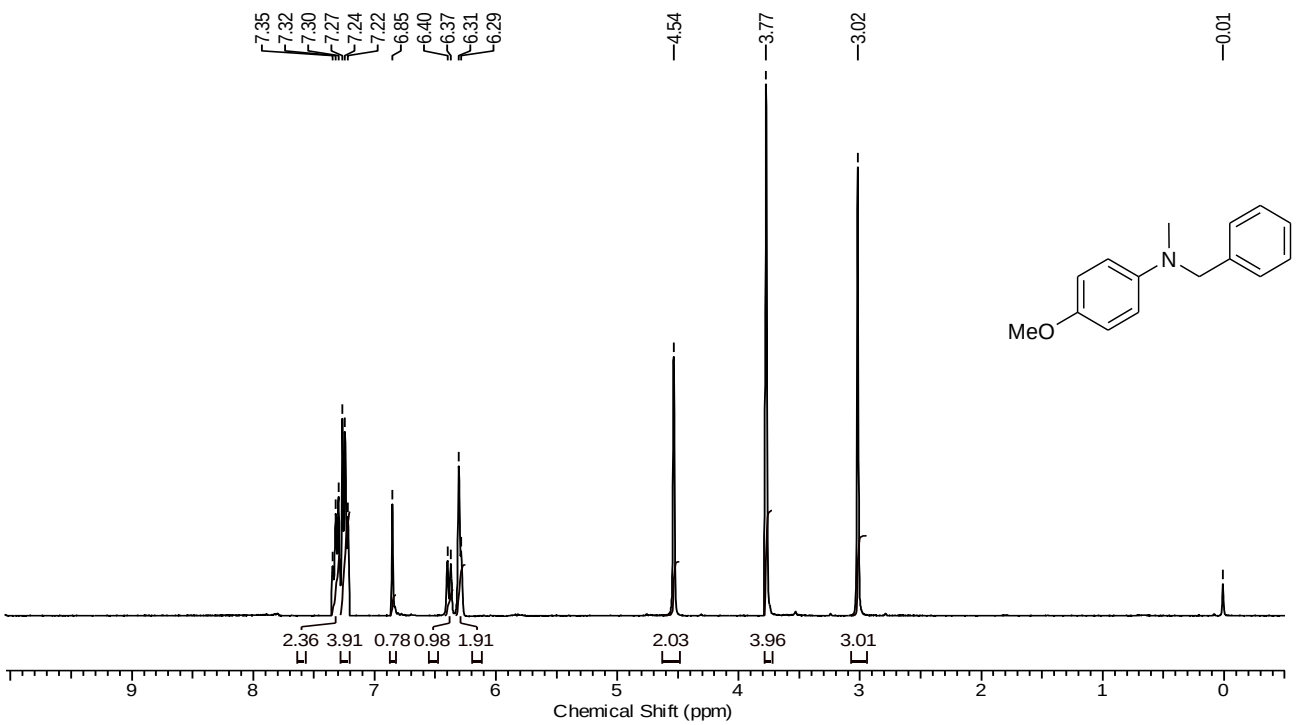

149.67
138.97
129.12
128.50
126.79
126.66
116.43
112.26
77.42
77.00
76.58
56.54
38.46
N
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

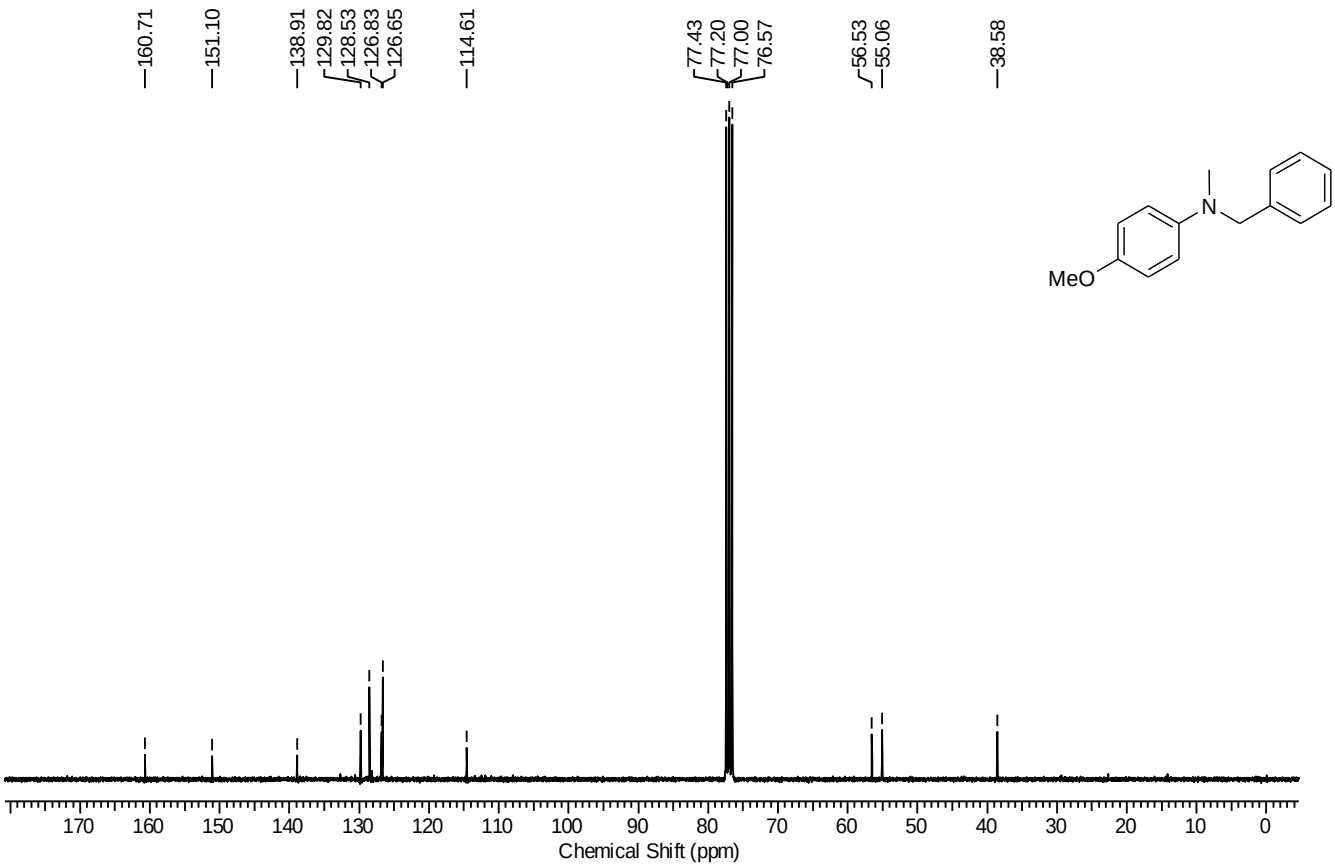

7.29
7.27
7.09
7.06
6.63
6.60
4.53
3.01
2.29
0.05
N
4.98
2.19
1.92
1.92
2.79
3.00
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

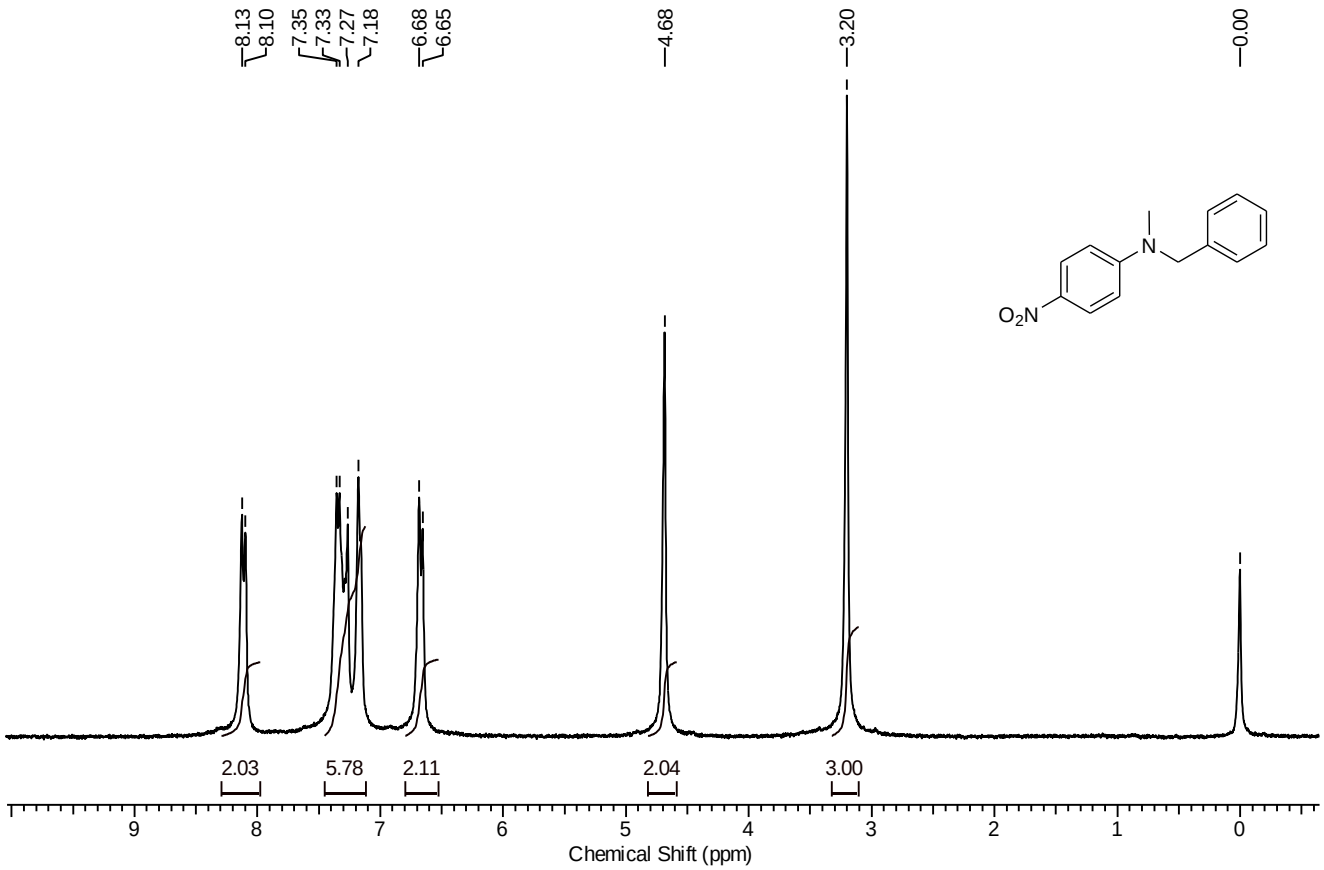

147.71
139.18
129.64
128.46
126.78
125.70
117.44
112.62
77.41
76.99
76.57
56.93
38.59
20.20
N
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

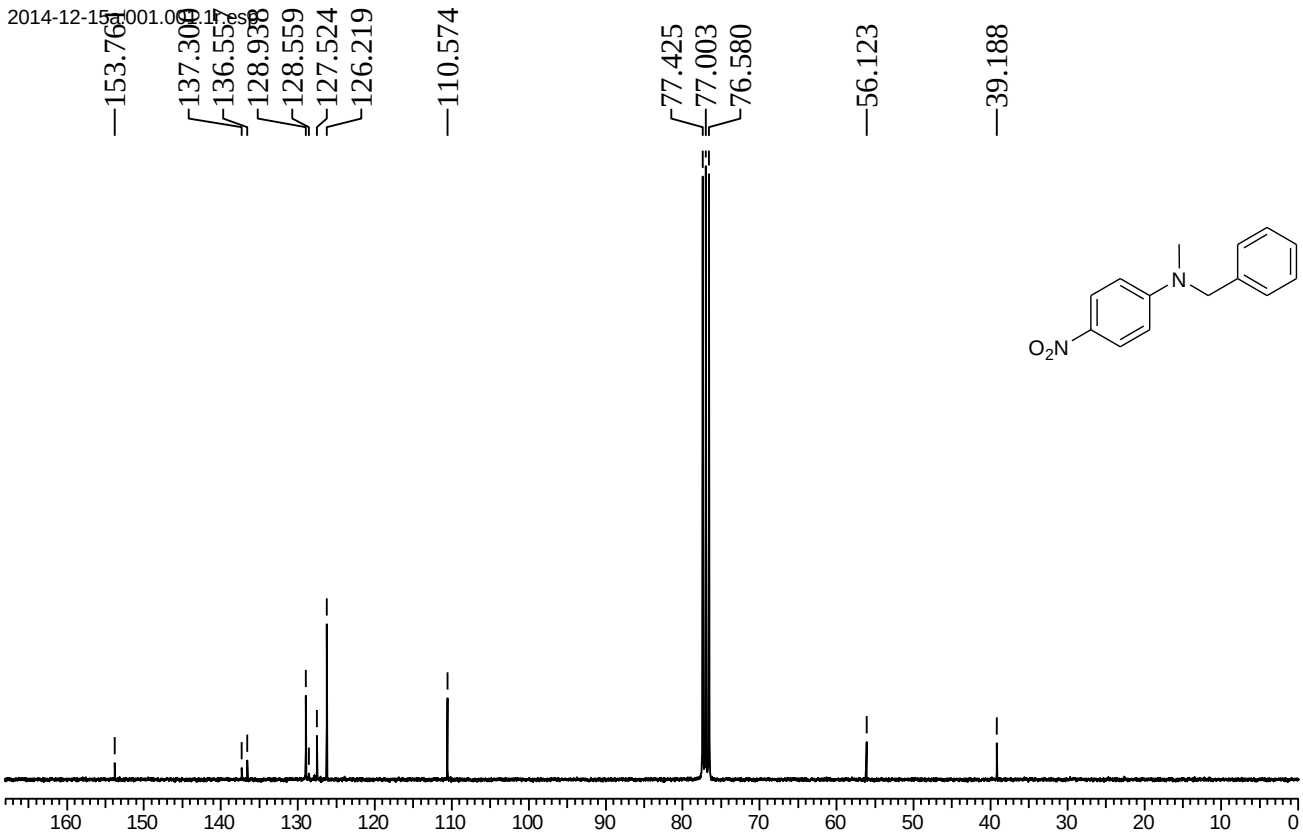

7.34
7.32
7.27
7.25
6.61
6.59
6.56
4.55
3.02
2.32
N
7.98
3.05
2.08
3.29
3.01
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

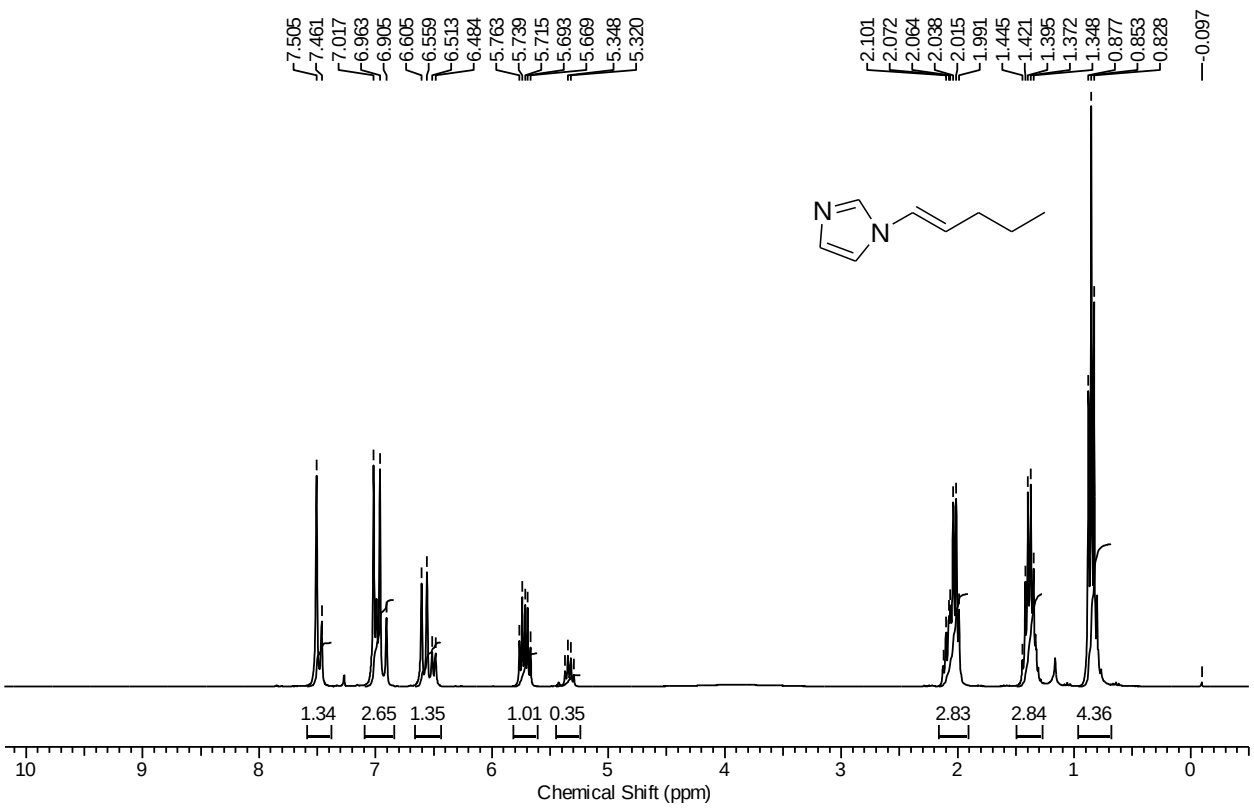

149.86
139.20
129.65
129.00
128.49
126.76
117.47
113.00
112.65
109.57
77.42
77.00
76.57
56.95
38.59
21.89
N
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

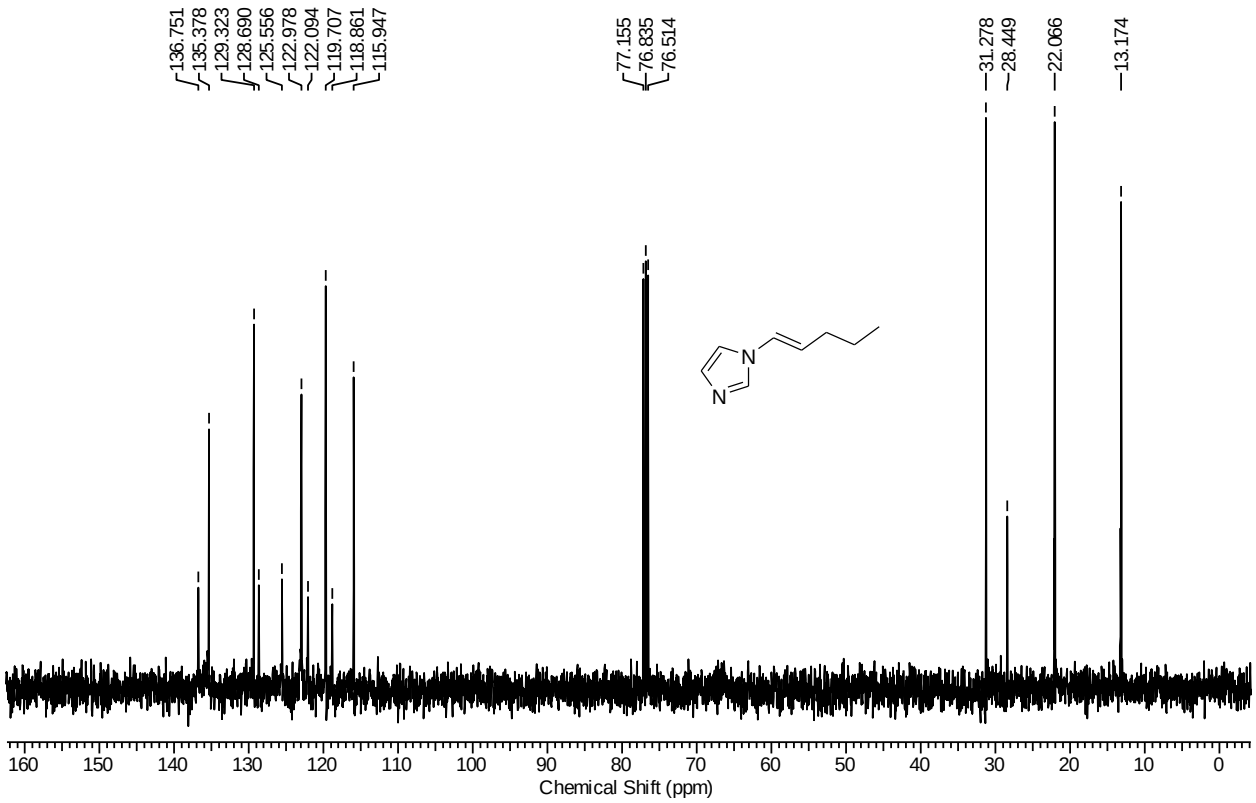

7.35
7.32
7.30
7.27
7.24
7.22
6.85
6.40
6.37
6.31
6.29
4.54
3.77
3.02
0.01
N
MeO
2.36
3.91
0.78
0.98
1.91
2.03
3.96
3.01
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

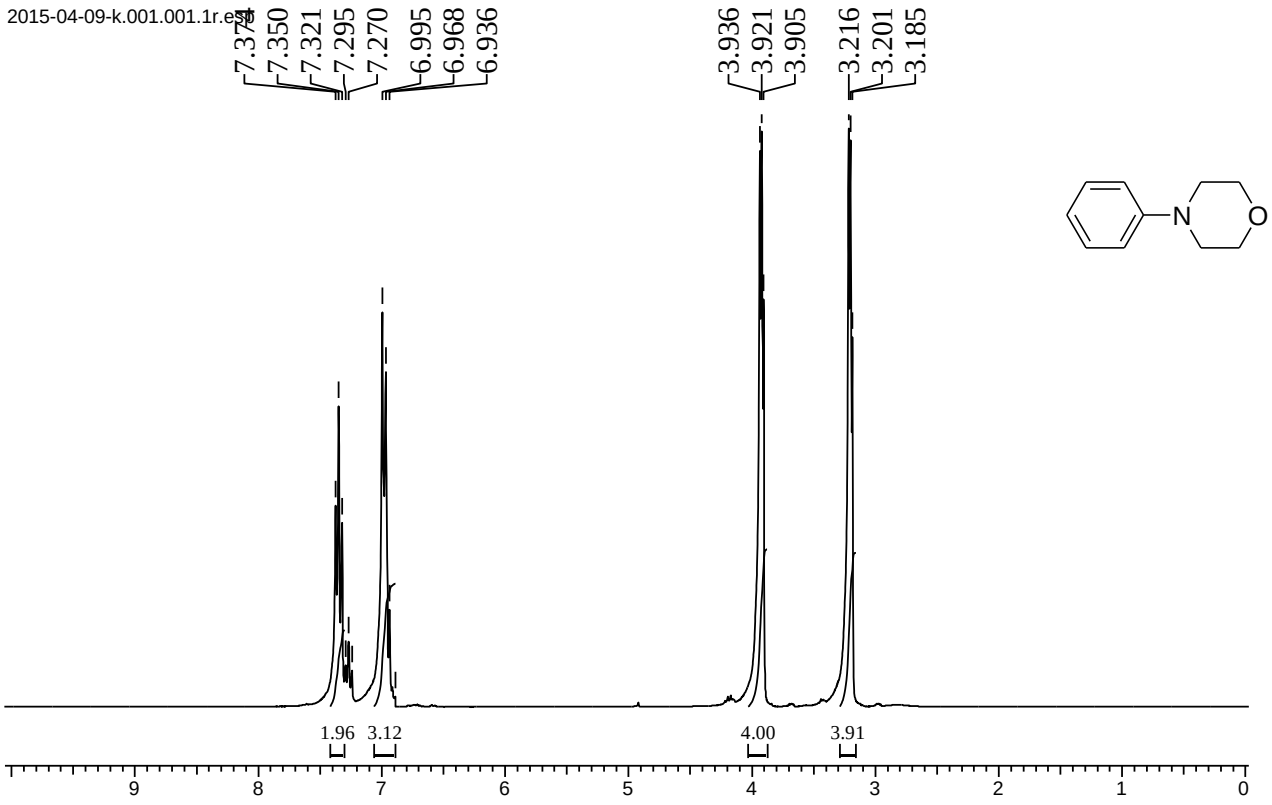

160.71
151.10
138.91
129.82
128.53
126.83
126.65
114.61
77.43
77.20
77.00
76.57
56.53
55.06
38.58
N
MeO
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

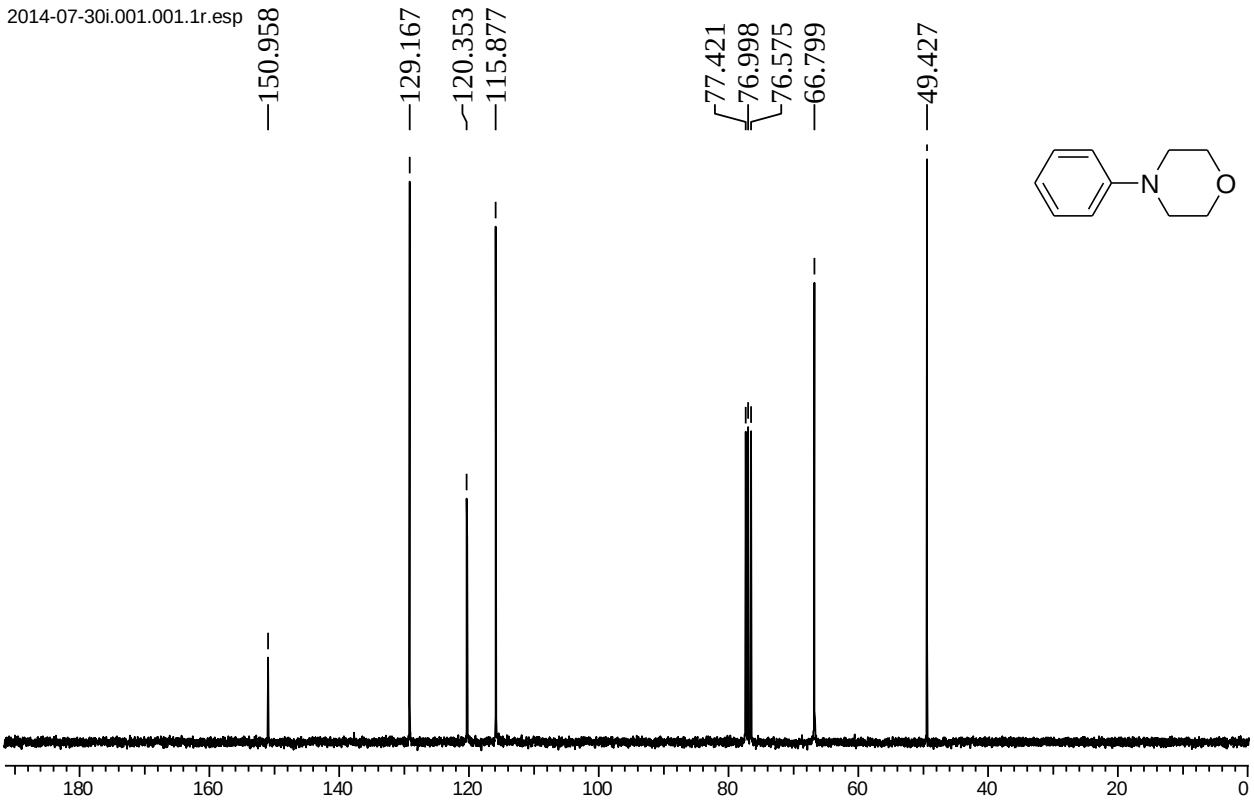

8.13
8.10
7.35
7.33
7.27
7.18
6.68
6.65
4.68
3.20
0.00
2.03
5.78
2.11
2.04
3.00
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)
N
O2N

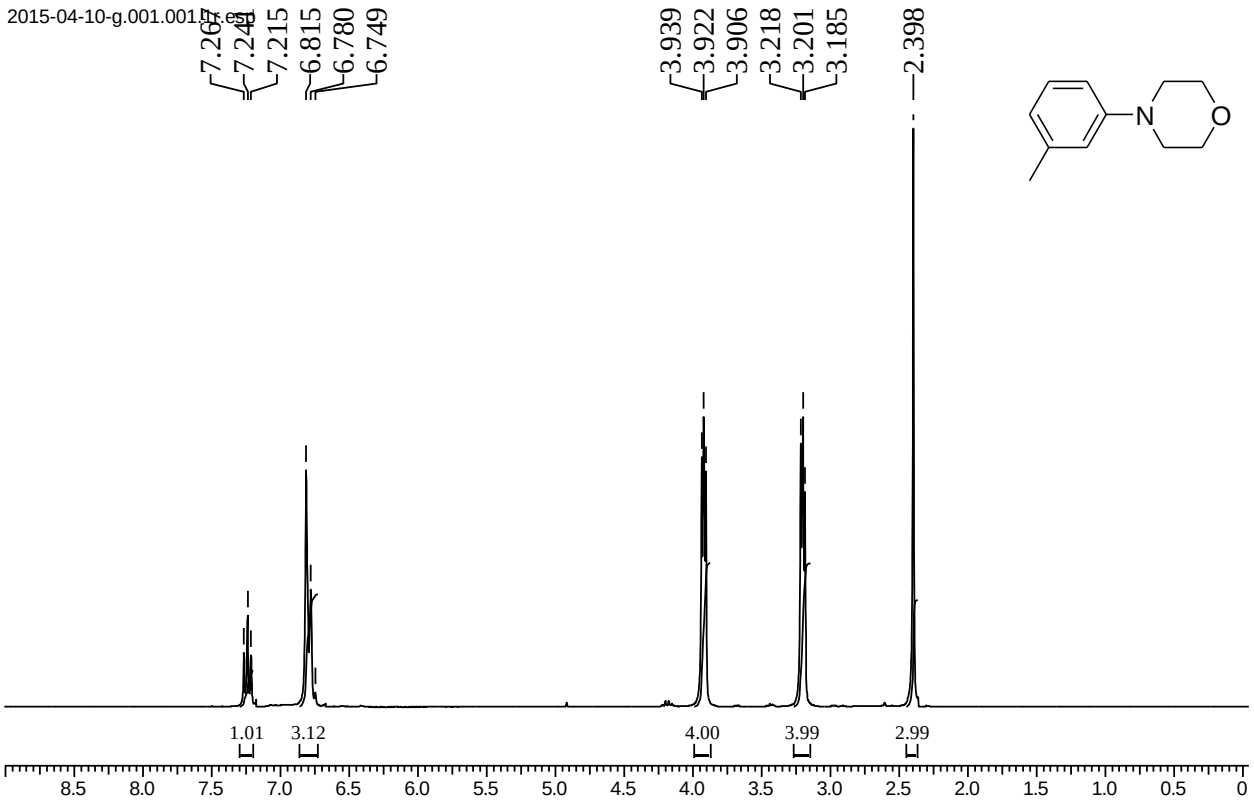

2014-12-15a.001.001.1r.esp
153.764
137.300
136.557
128.938
128.559
127.524
126.219
110.574
77.425
77.003
76.580
56.123
39.188
N
O2N
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

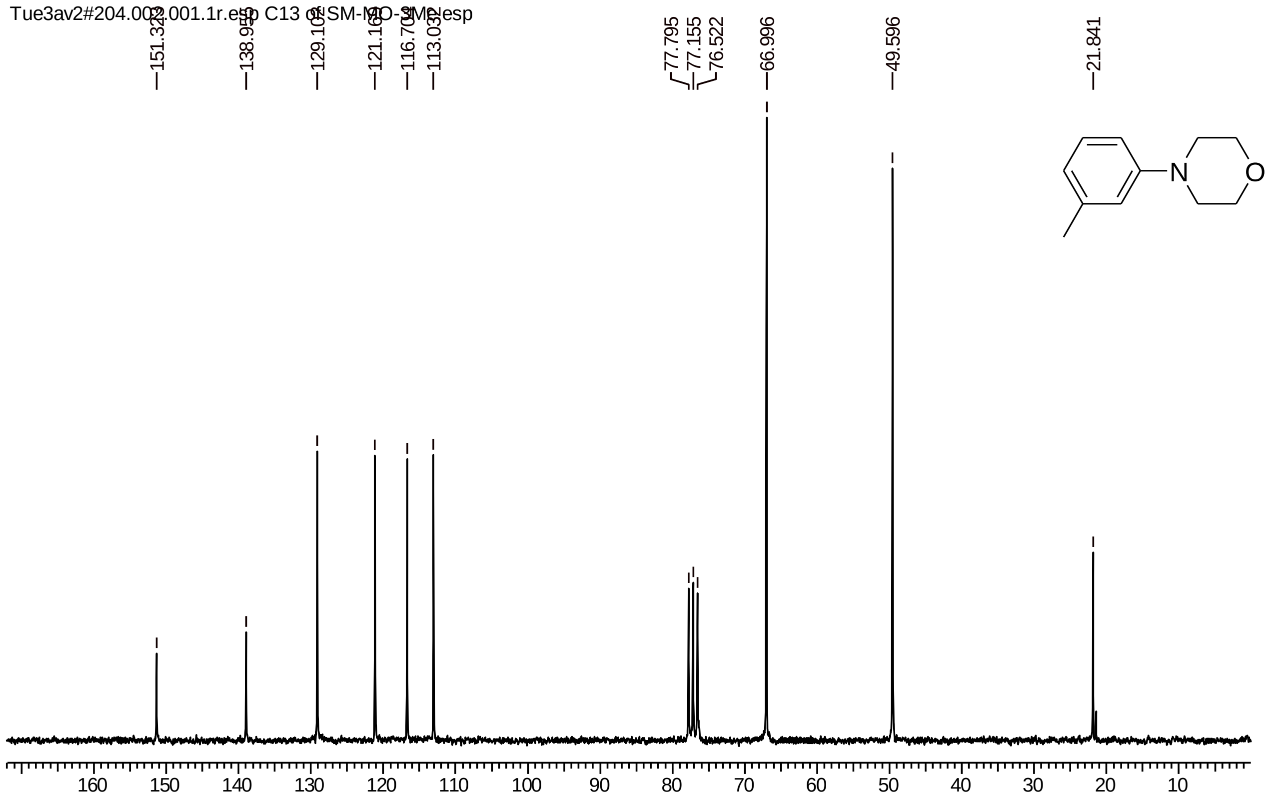

7.505
7.461
7.017
6.963
6.905
6.605
6.559
6.513
6.484
5.763
5.739
5.715
5.693
5.669
5.348
5.320
2.101
2.072
2.064
2.038
2.015
1.991
1.445
1.421
1.395
1.372
1.348
0.877
0.853
0.828
-0.097
N
N
1.34
2.65
1.35
1.01
0.35
2.83
2.84
4.36
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

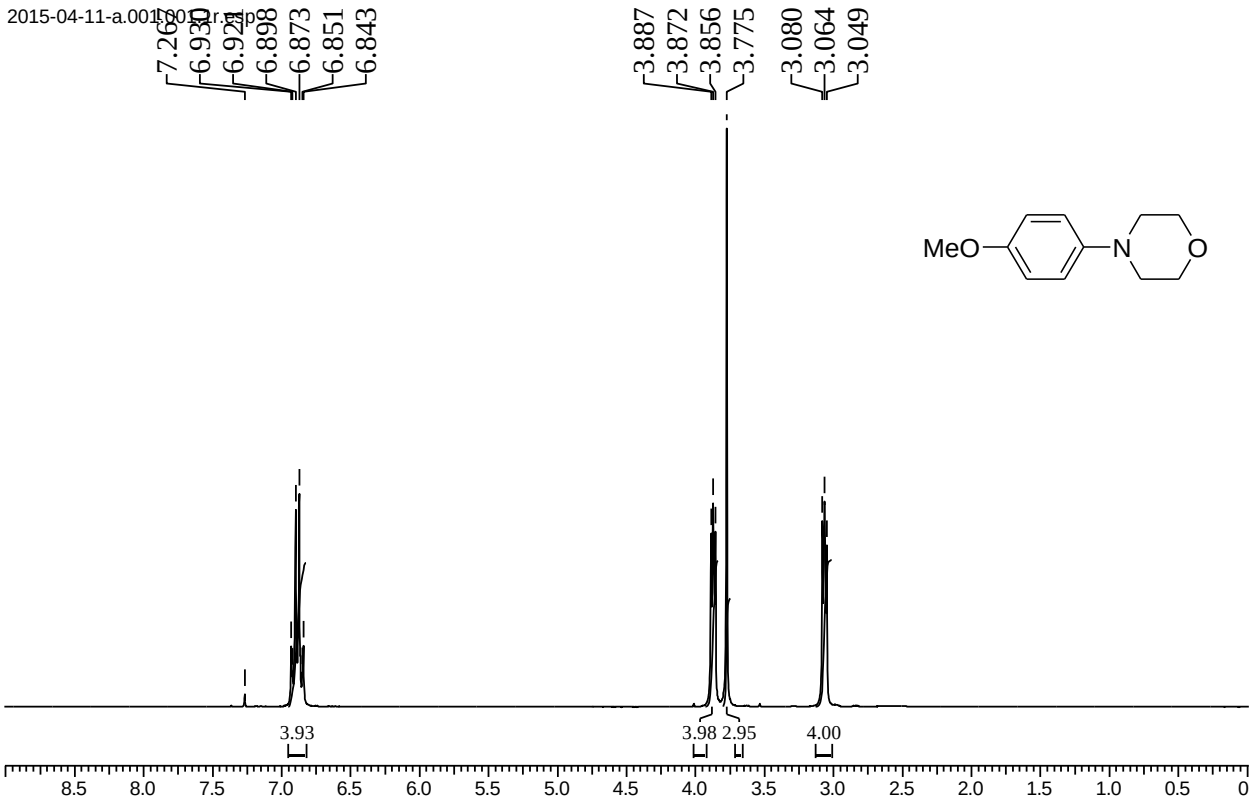

136.751
135.378
129.323
128.690
125.556
122.978
122.094
119.707
118.861
115.947
77.155
76.835
76.514
31.278
28.449
22.066
13.174
N
N
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

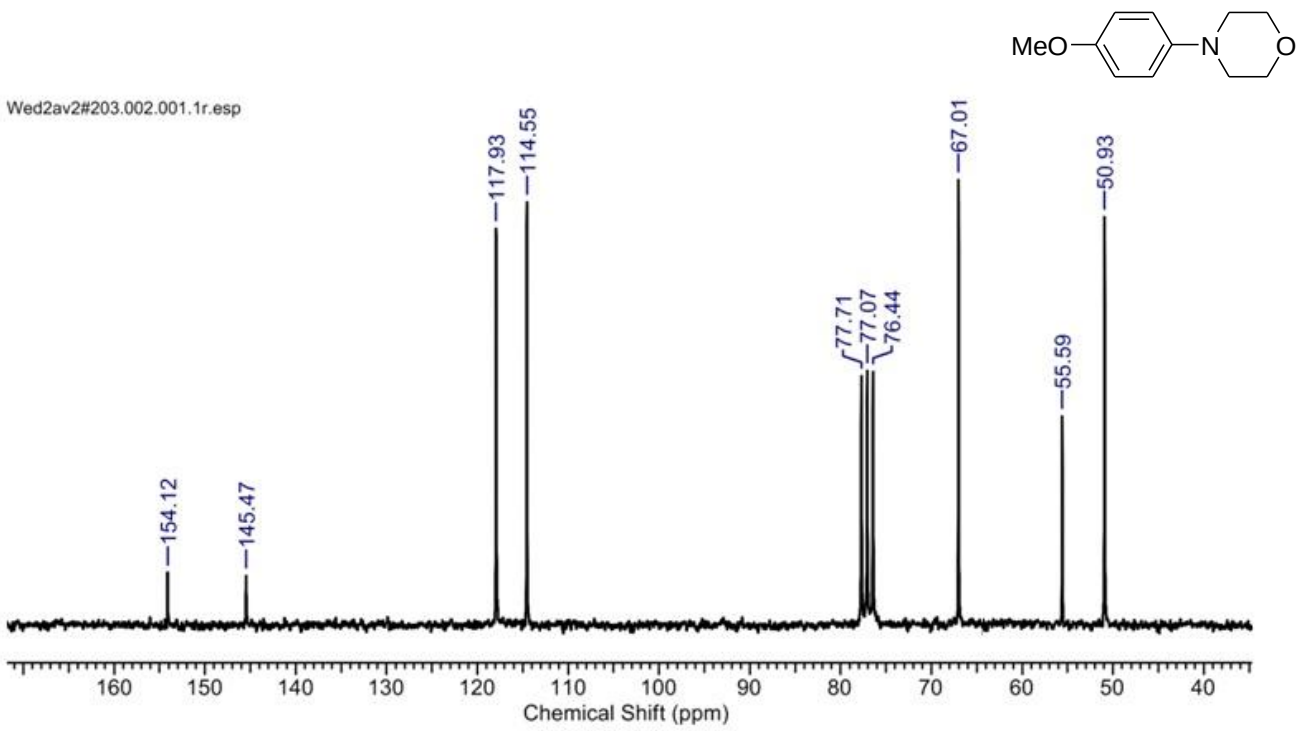

2015-04-09-k.001.001.1r.esp
7.374
7.350
7.321
7.295
7.270
6.995
6.968
6.936
3.936
3.921
3.905
3.216
3.201
3.185
N
O
1.96
3.12
4.00
3.91
9
8
7
6
5
4
3
2
1
0

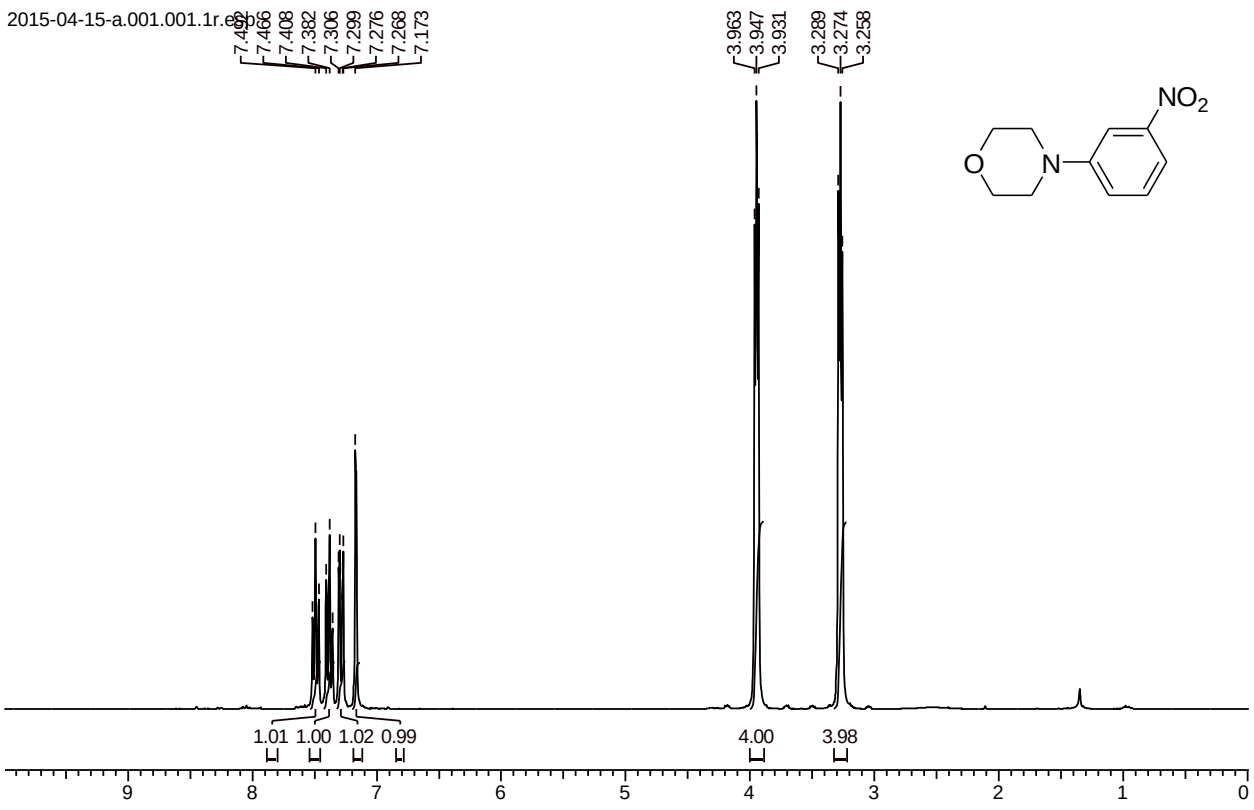

2014-07-30i.001.001.1r.esp
150.958
129.167
120.353
115.877
77.421
76.998
76.575
66.799
49.427
N
O
180
160
140
120
100
80
60
40
20
0

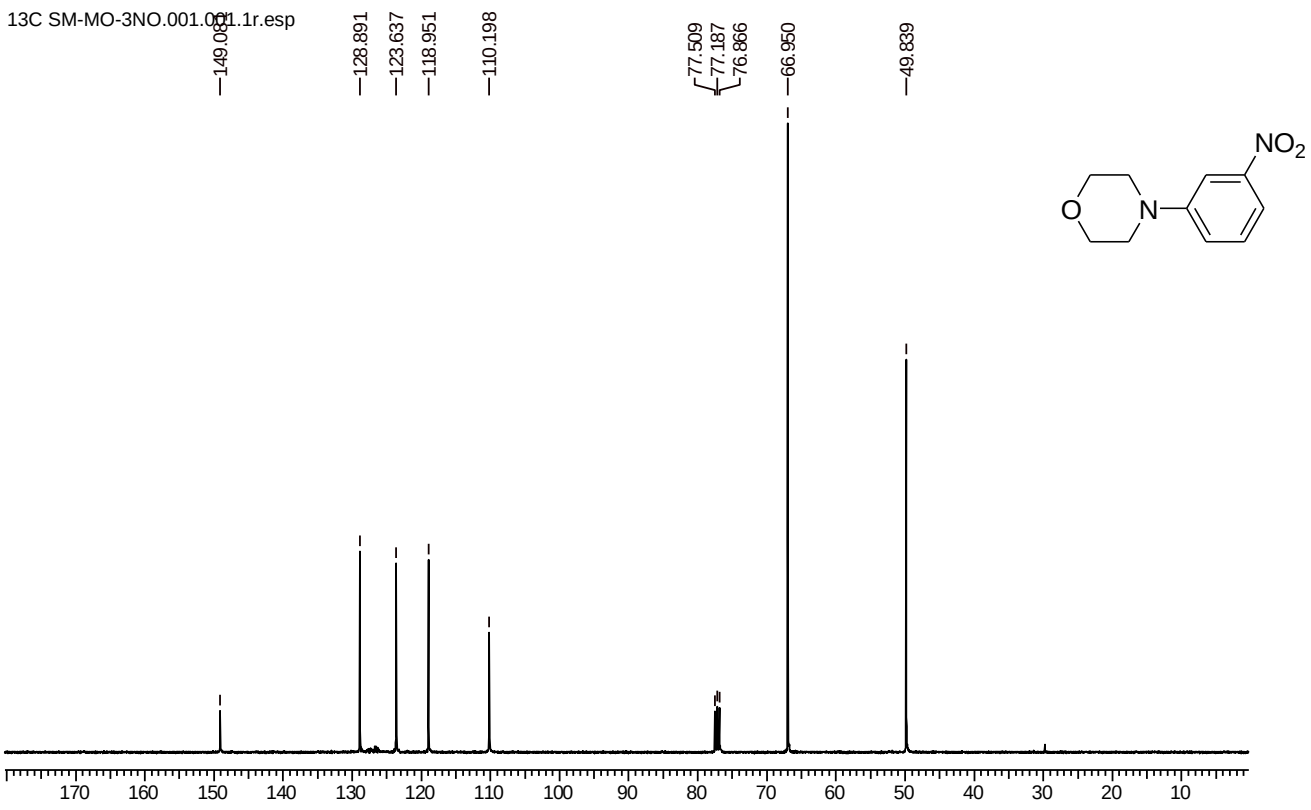

2015-04-10-g.001.001.1.esp
7.267
7.248
7.215
6.815
6.780
6.749
3.939
3.922
3.906
3.218
3.201
3.185
2.398
N
O
1.01
3.12
4.00
3.99
2.99
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0

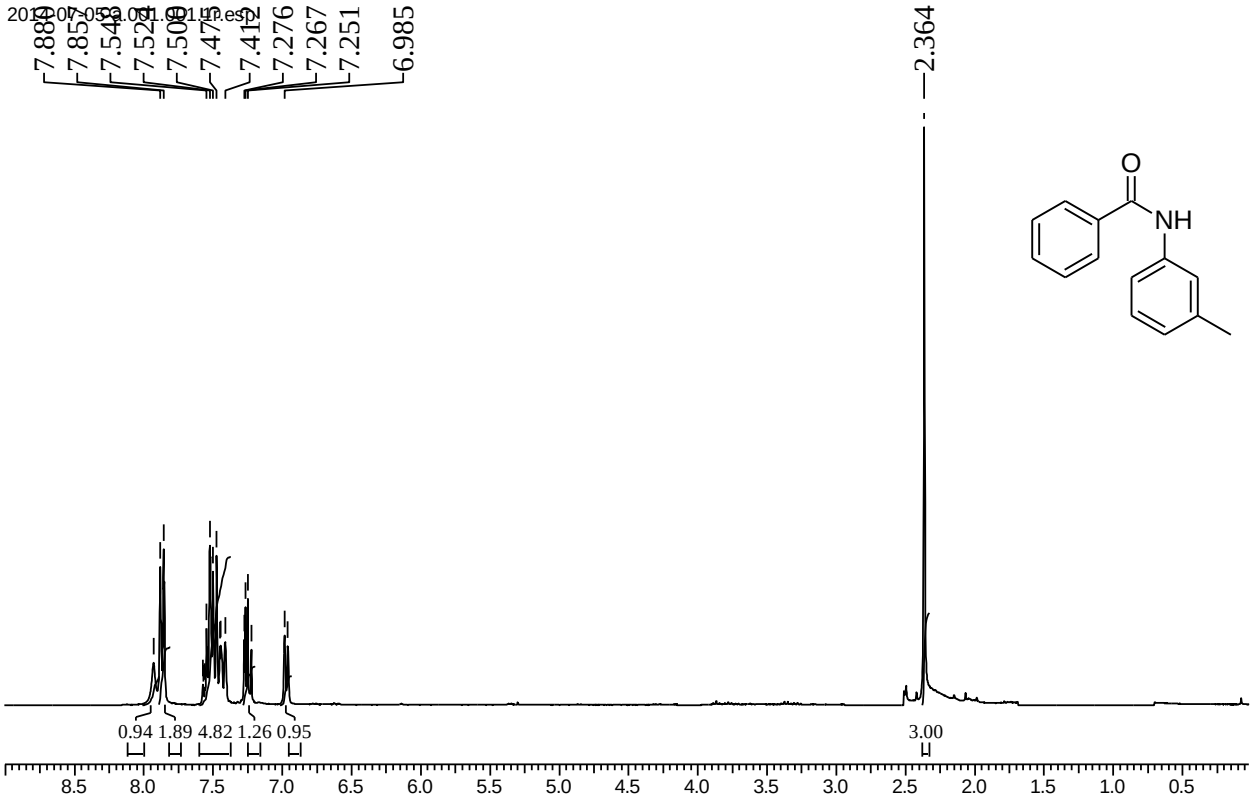

Tue3av2#204.002.001.1r.esp C13 of SM-MO-3Me.esp
151.322
138.950
129.103
121.163
116.709
113.037
77.795
77.155
76.522
66.996
49.596
21.841
N
O
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

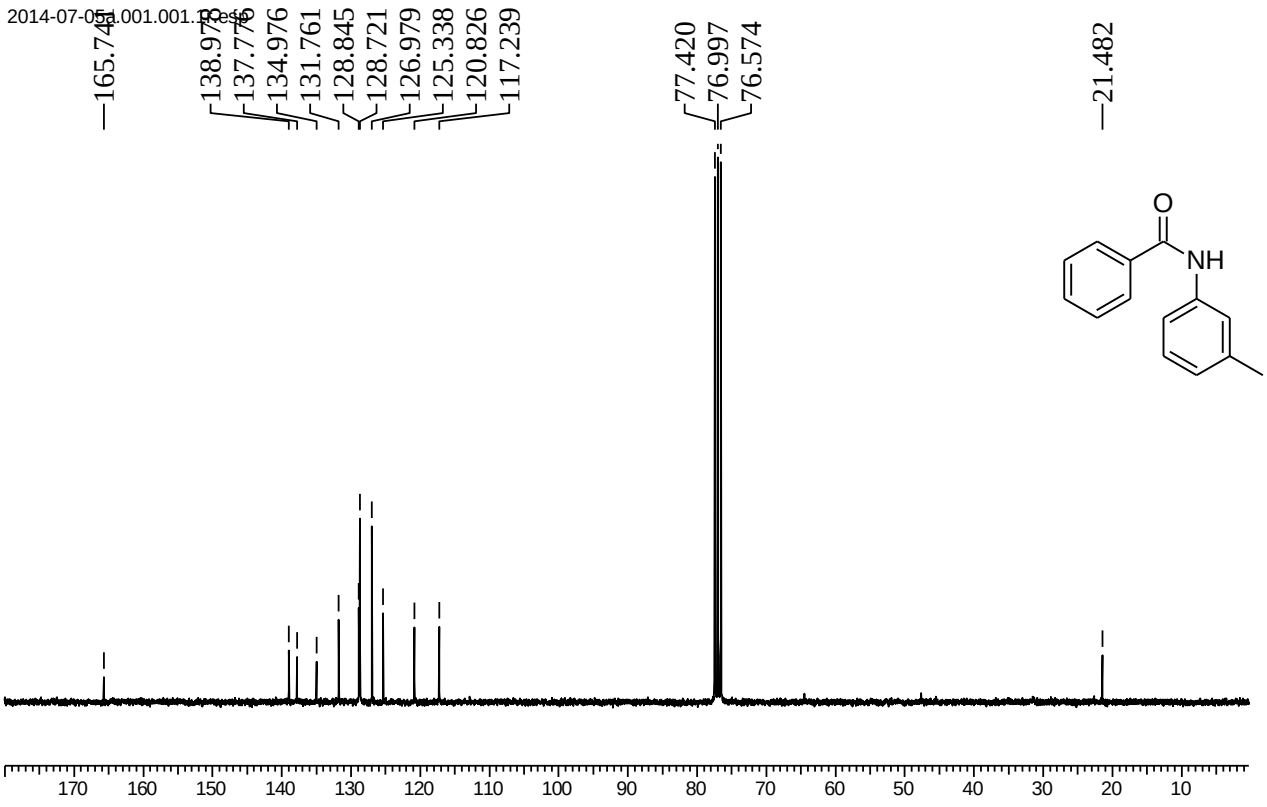

2015-04-11-a.001001.1r.esp
7.260
6.930
6.922
6.898
6.873
6.851
6.843
3.887
3.872
3.856
3.775
3.080
3.064
3.049
MeO
N
O
3.93
3.98
2.95
4.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0


MeO
N
O
Wed2av2#203.002.001.1r.esp
154.12
145.47
117.93
114.55
77.71
77.07
76.44
67.01
55.59
50.93
160
150
140
130
120
110
100
90
80
70
60
50
40
Chemical Shift (ppm)


2015-04-15-a.001.001.1r.esp
7.466
7.408
7.382
7.306
7.299
7.276
7.268
7.173
3.963
3.947
3.931
3.289
3.274
3.258
NO2
O
N
1.01
1.00
1.02
0.99
4.00
3.98
9
8
7
6
5
4
3
2
1
0


13C SM-MO-3NO.001.001.1r.esp
149.082
128.891
123.637
118.951
110.198
77.509
77.187
76.866
66.950
49.839
NO2
O
N
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


7.884
7.853
7.544
7.521
7.508
7.473
7.412
7.276
7.267
7.251
6.985
2.364
O
NH
0.94 1.89 4.82 1.26 0.95
3.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5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165.745
138.978
137.776
134.976
131.761
128.845
128.721
126.979
125.338
120.826
117.239
77.420
76.997
76.574
21.482
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NH
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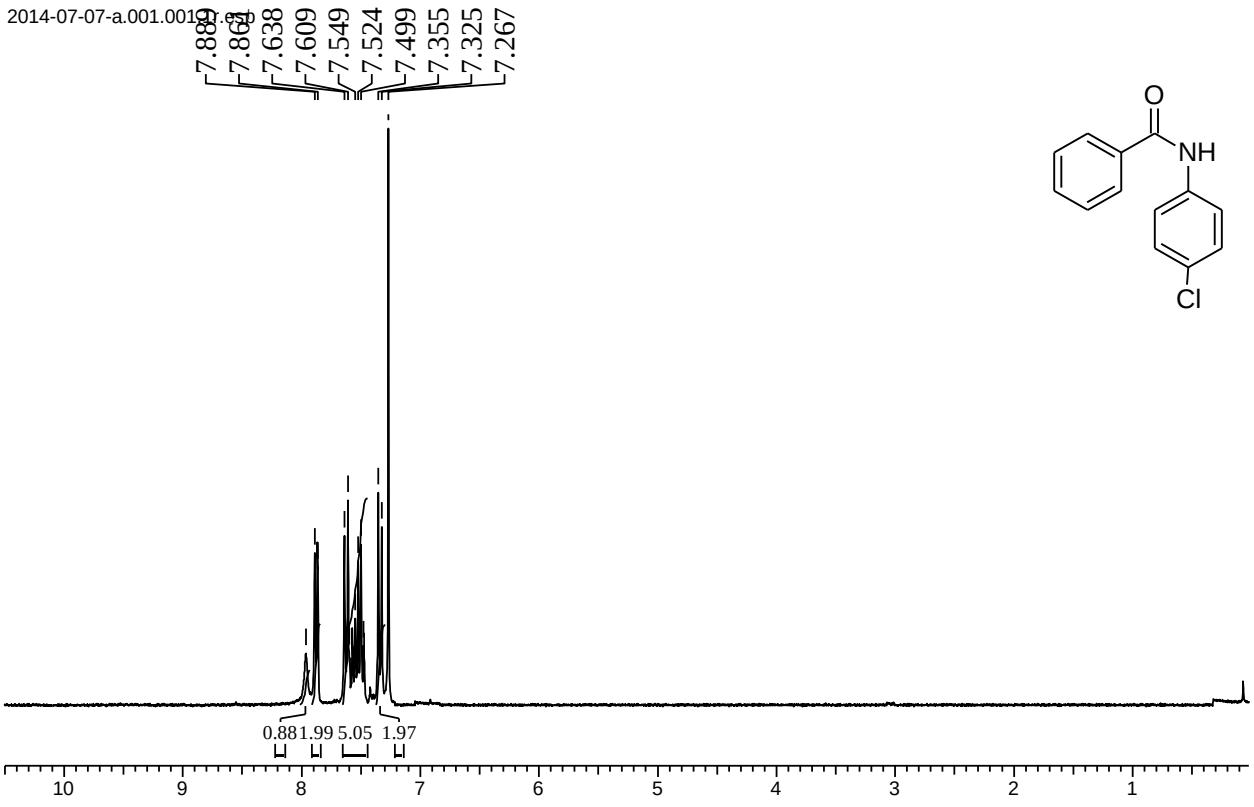
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


2014-07-07-a.001.001.1r.esp
7.889
7.864
7.638
7.609
7.549
7.524
7.499
7.355
7.325
7.267
O
NH
Cl
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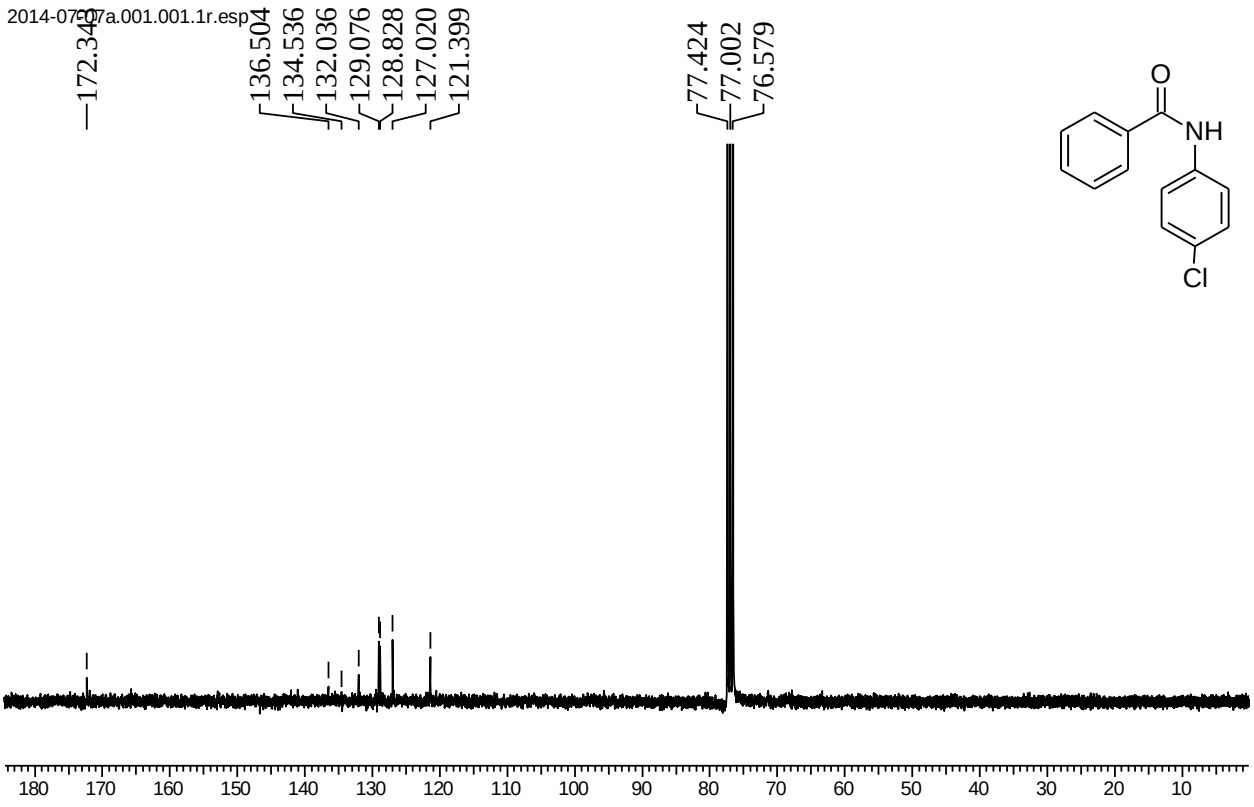
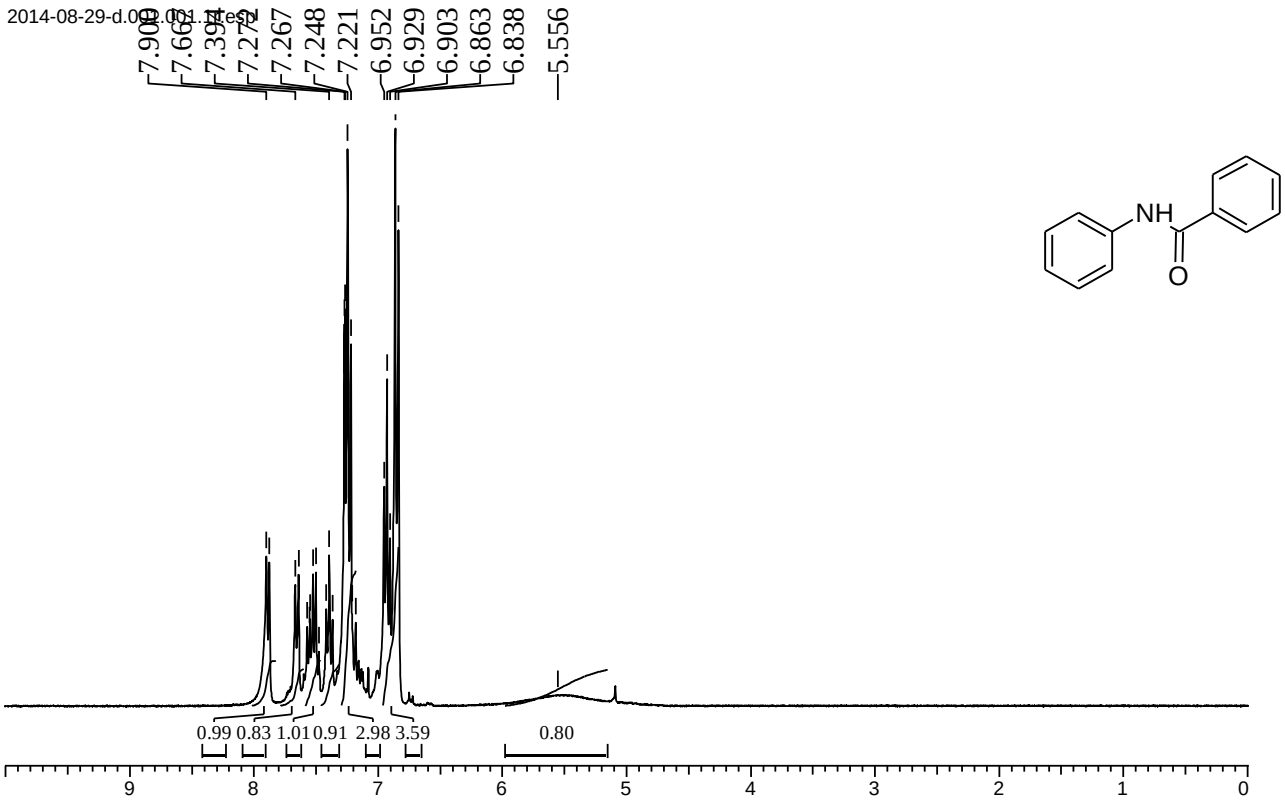
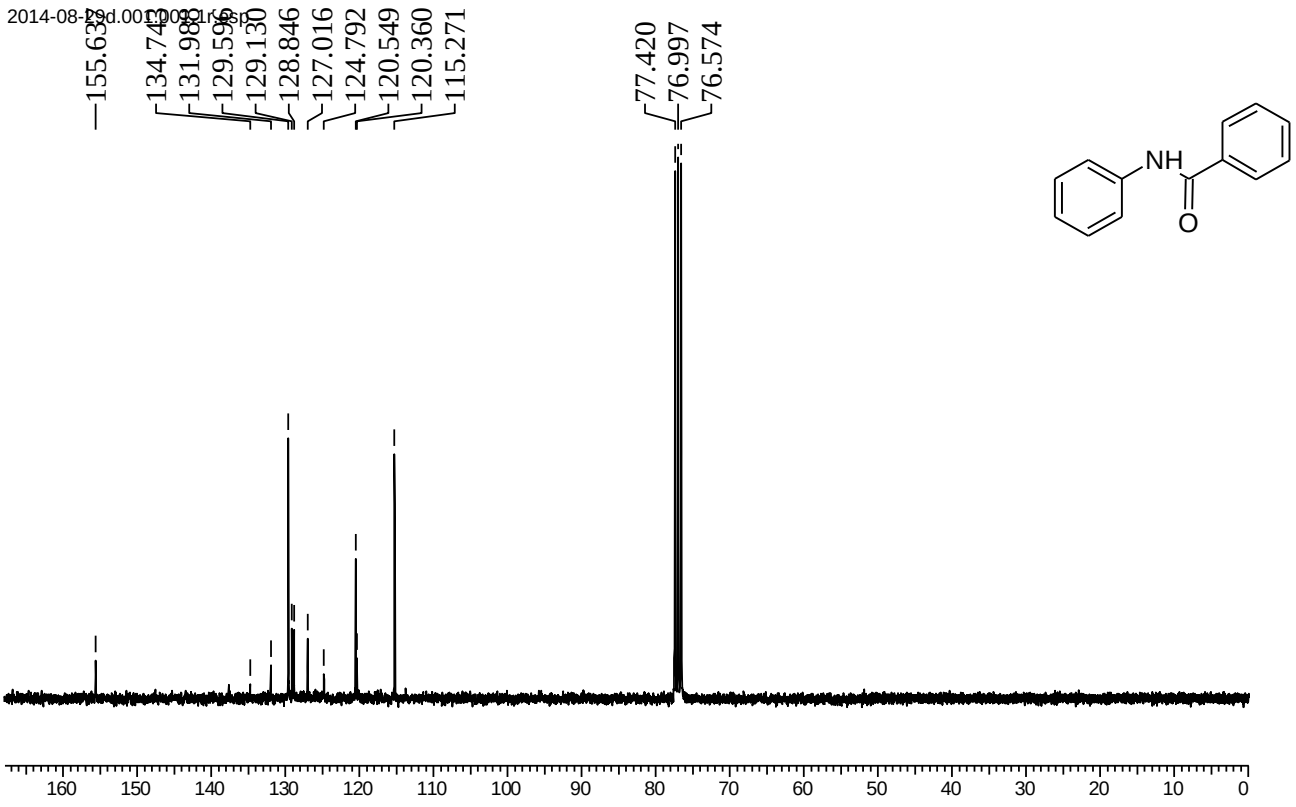
1.99
5.05
1.97
10
9
8
7
6
5
4
3
2
1


2014-07-07a.001.001.1r.esp
172.344
136.504
134.536
132.036
129.076
128.828
127.020
121.399
77.424
77.002
76.579
O
NH
Cl
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


2014-08-29-d.001.01.1H.esp
7.908
7.662
7.394
7.272
7.267
7.248
7.221
6.952
6.929
6.903
6.863
6.838
5.556
NH
O
0.99 0.83 1.01 0.91 2.98 3.59
0.80
9
8
7
6
5
4
3
2
1
0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155.633
134.744
131.982
129.596
129.130
128.846
127.016
124.792
120.549
120.360
115.271
77.420
76.997
76.574
NH
O
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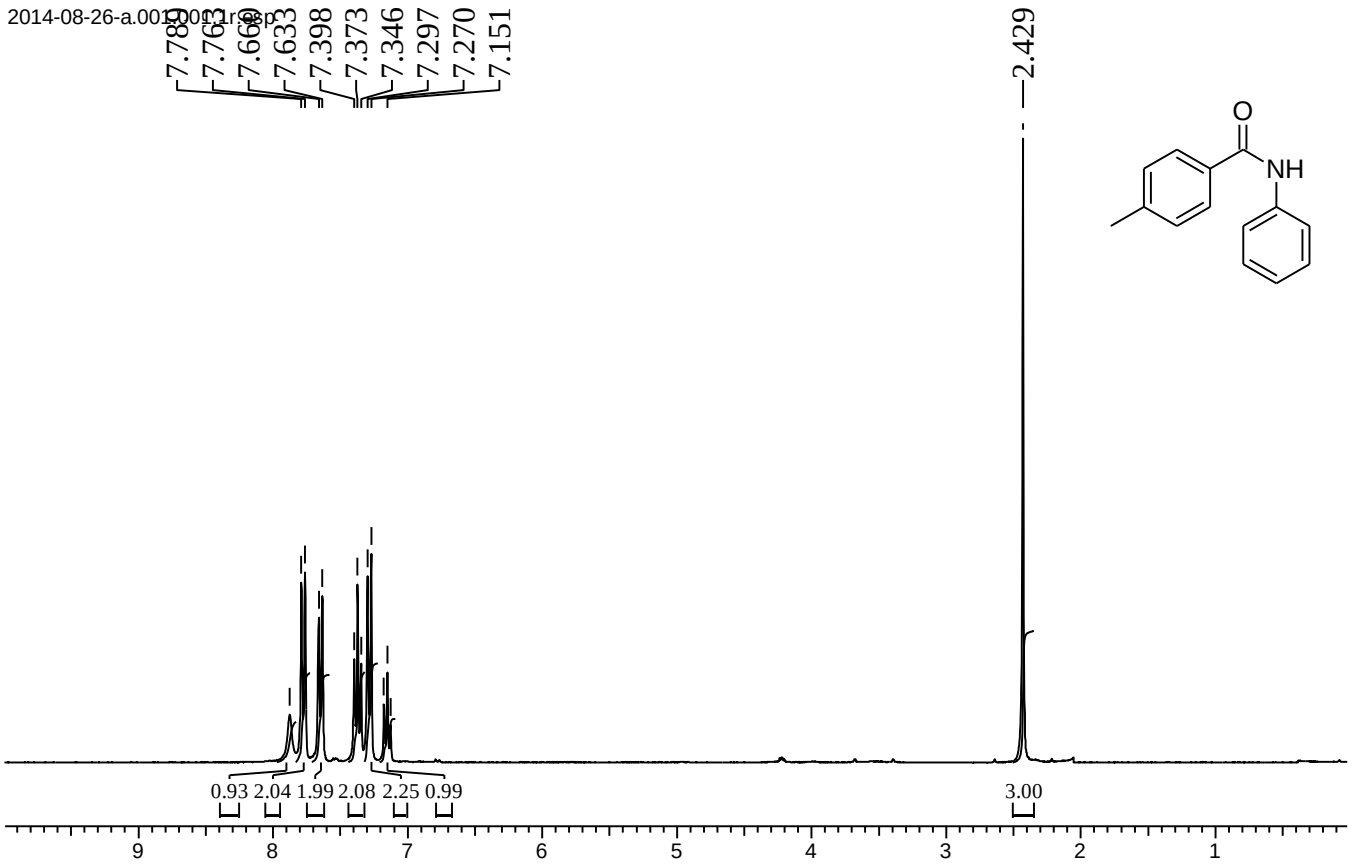
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


2014-08-26-a.001.1001.1r.esp
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7.763
7.660
7.633
7.398
7.373
7.346
7.297
7.270
7.151
2.429
O
NH
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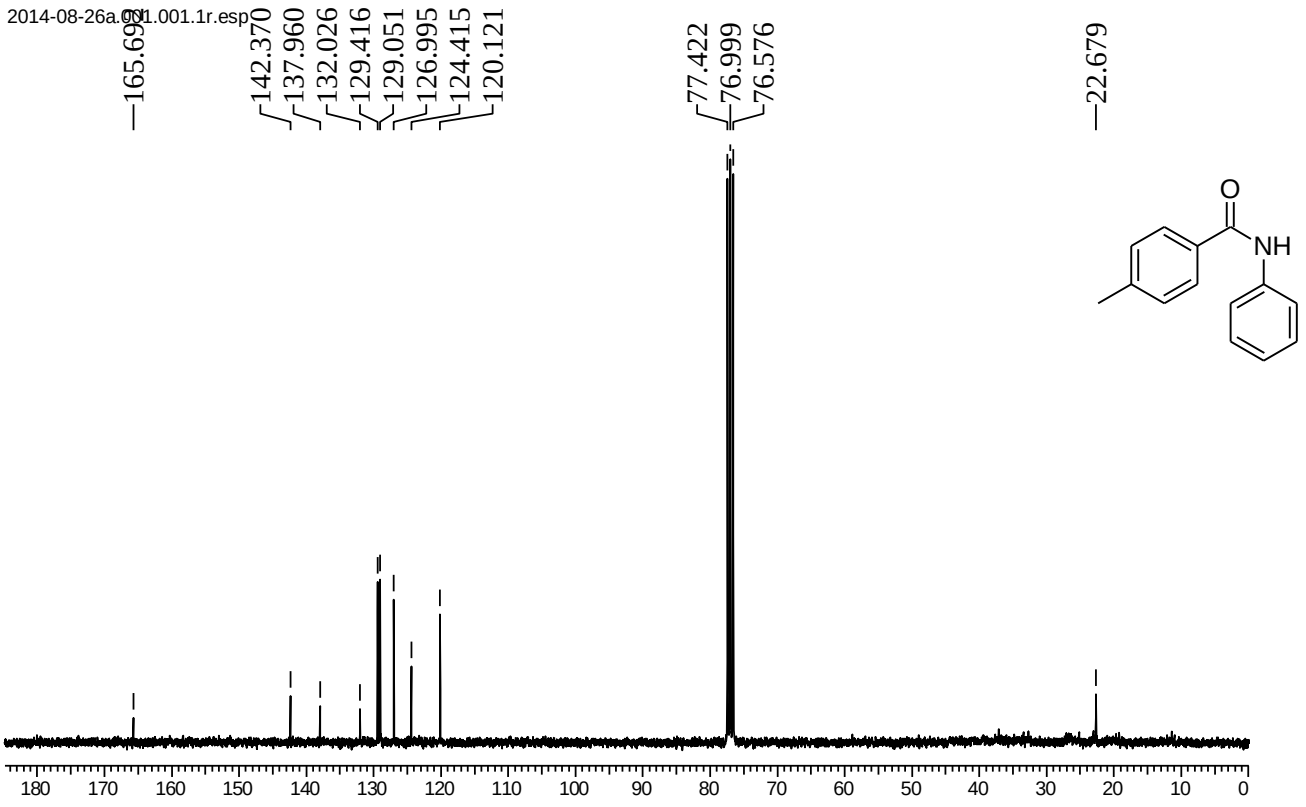
3.00
9
8
7
6
5
4
3
2
1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132.026
129.416
129.051
126.995
124.415
120.121
77.422
76.999
76.576
22.679
O
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150
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130
120
110
100
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80
70
60
50
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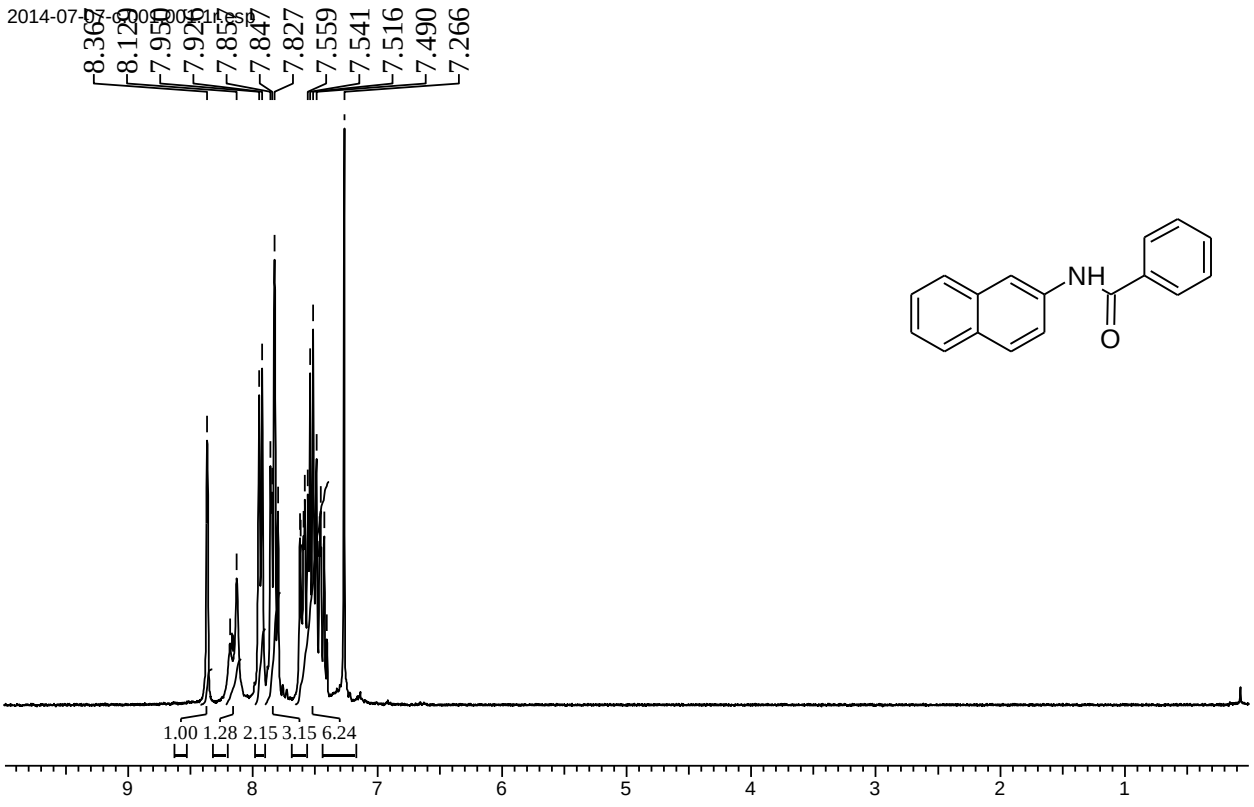
30
20
10
0


2014-07-07-c001_0001.11.esp
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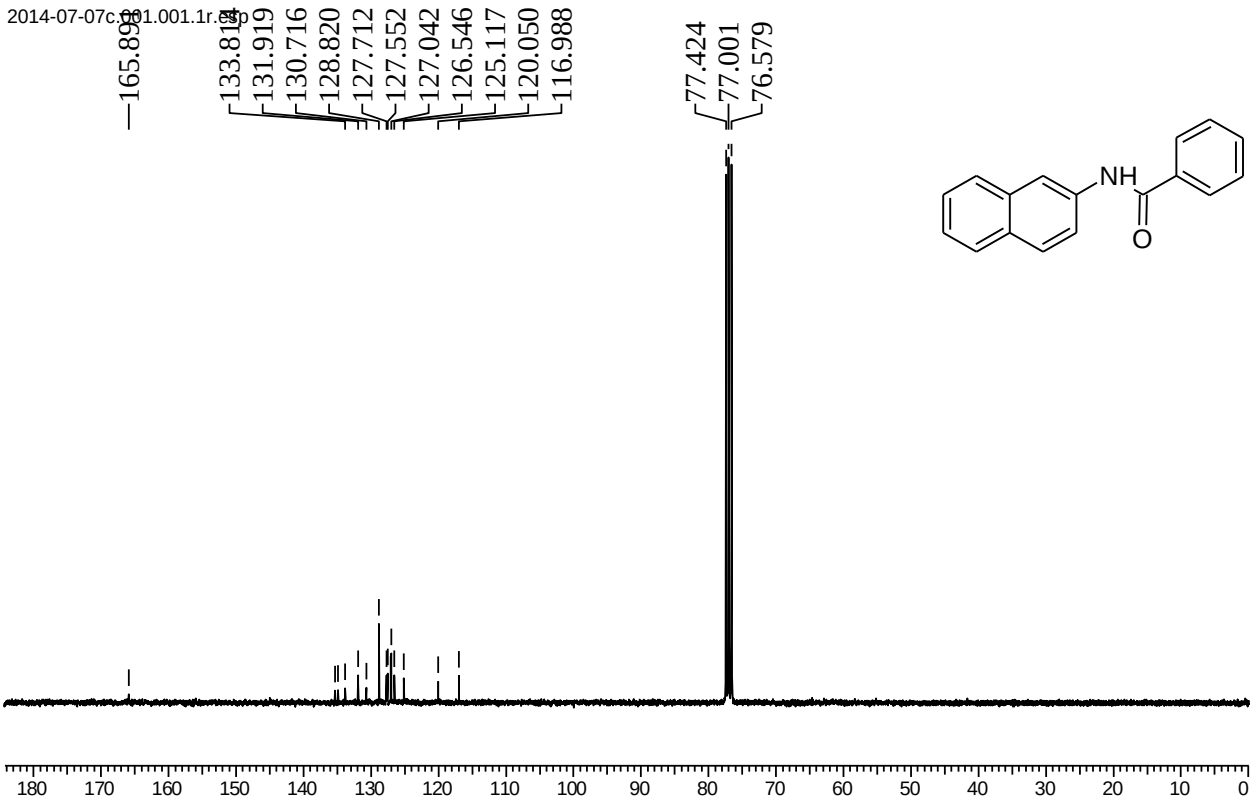
8.129
7.959
7.926
7.855
7.847
7.827
7.559
7.541
7.516
7.490
7.266
NH
O
1.00 1.28 2.15 3.15 6.24
9
8
7
6
5
4
3
2
1


2014-07-07c.001.001.1r.esp
165.893
133.818
131.919
130.716
128.820
127.712
127.552
127.042
126.546
125.117
120.050
116.988
77.424
77.001
76.579
NH
O
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170
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150
140
130
120
110
100
90
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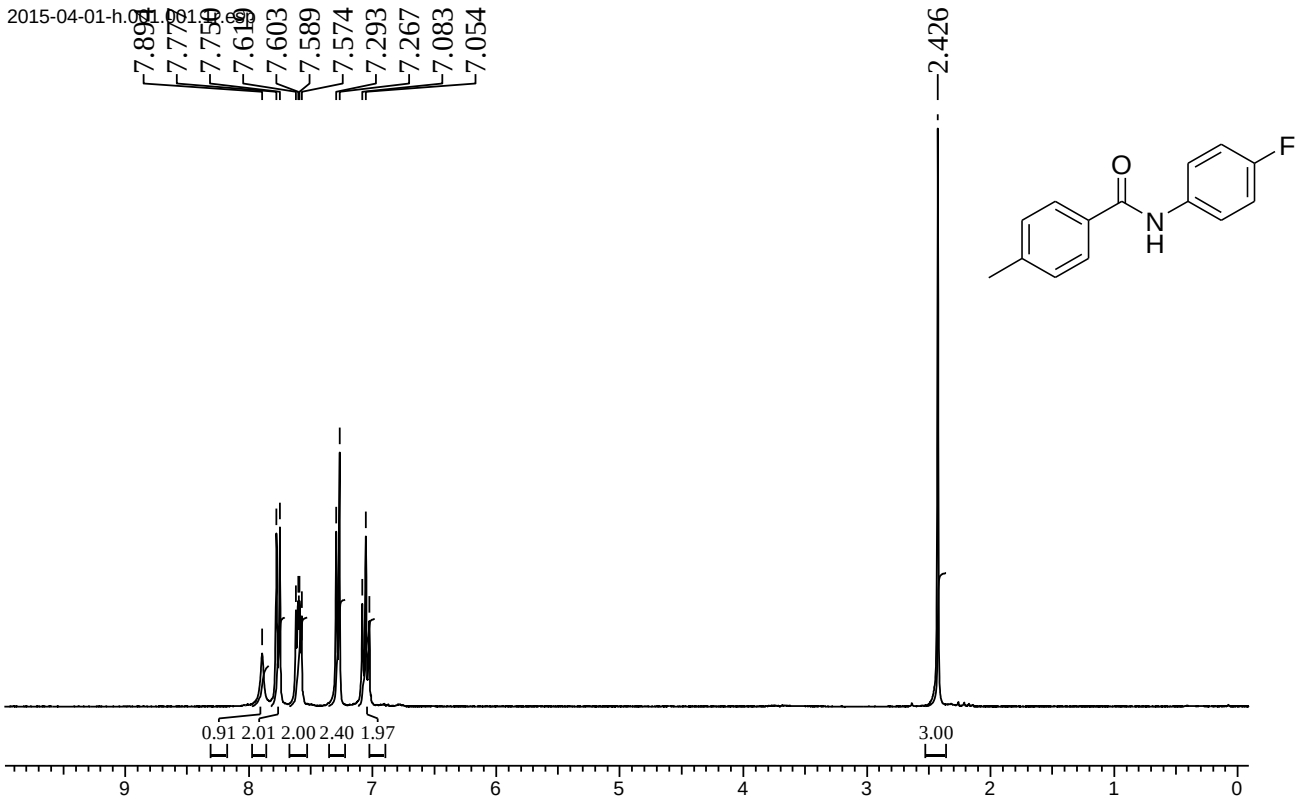
70
60
50
40
30
20
10
0


2015-04-01-h.001.001.10.esp
7.895
7.777
7.750
7.619
7.603
7.589
7.574
7.293
7.267
7.083
7.054
2.426
O
N
H
F
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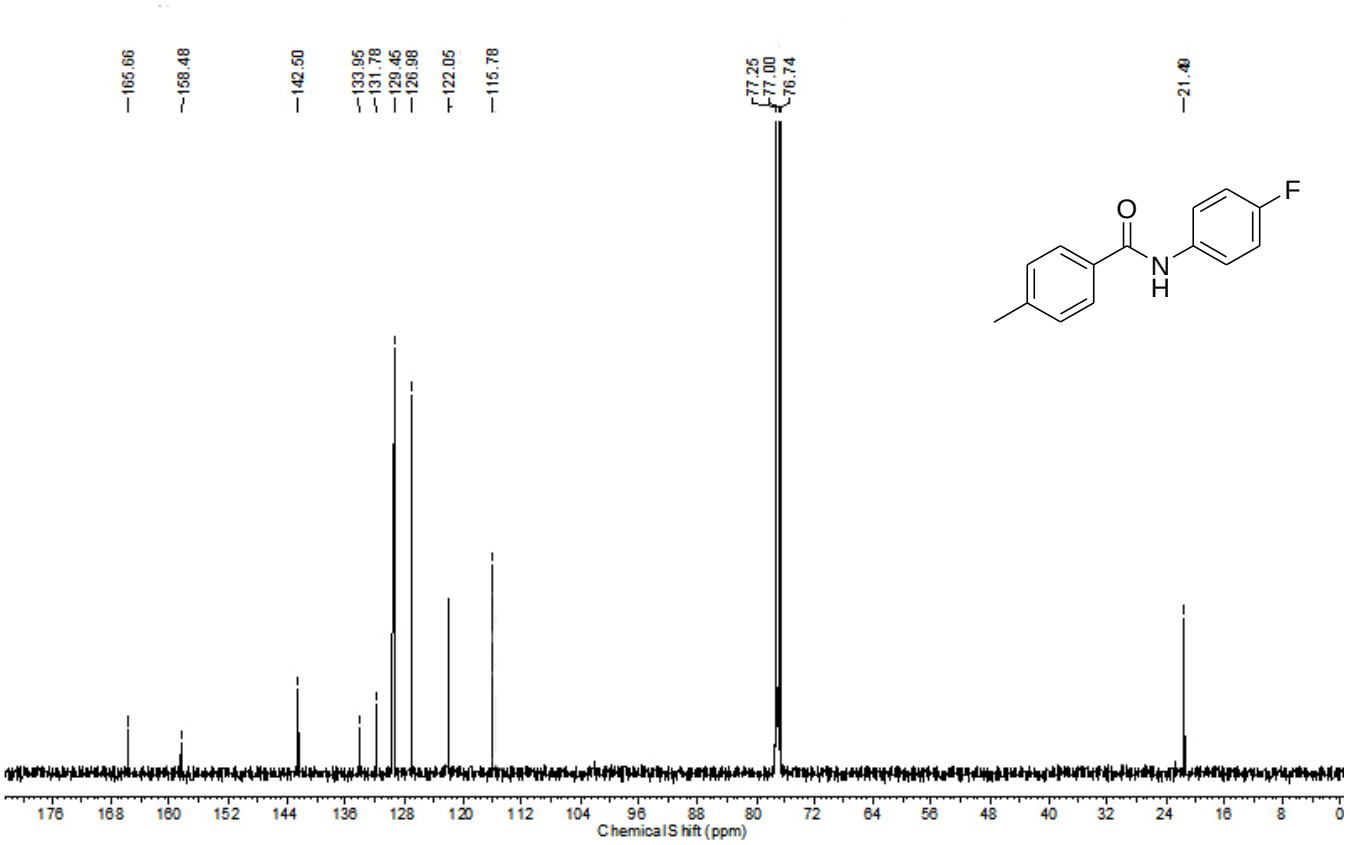
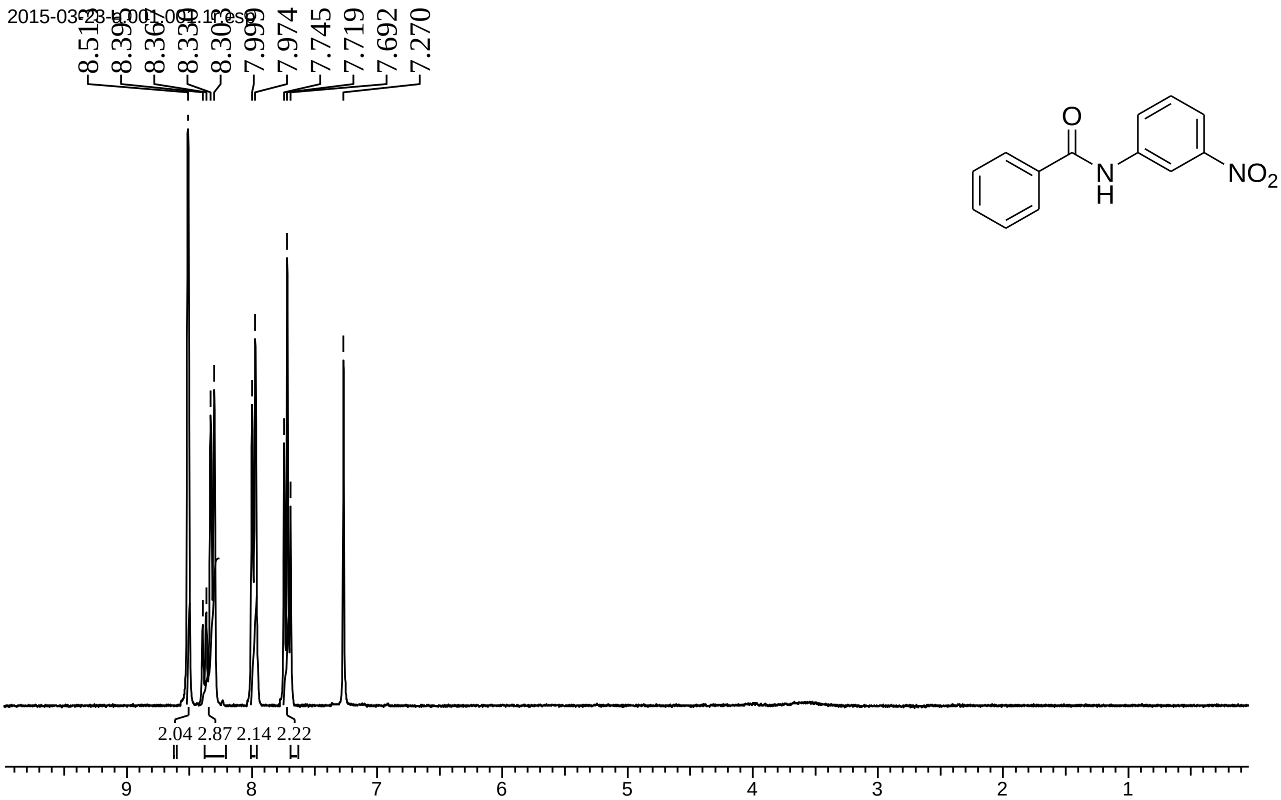
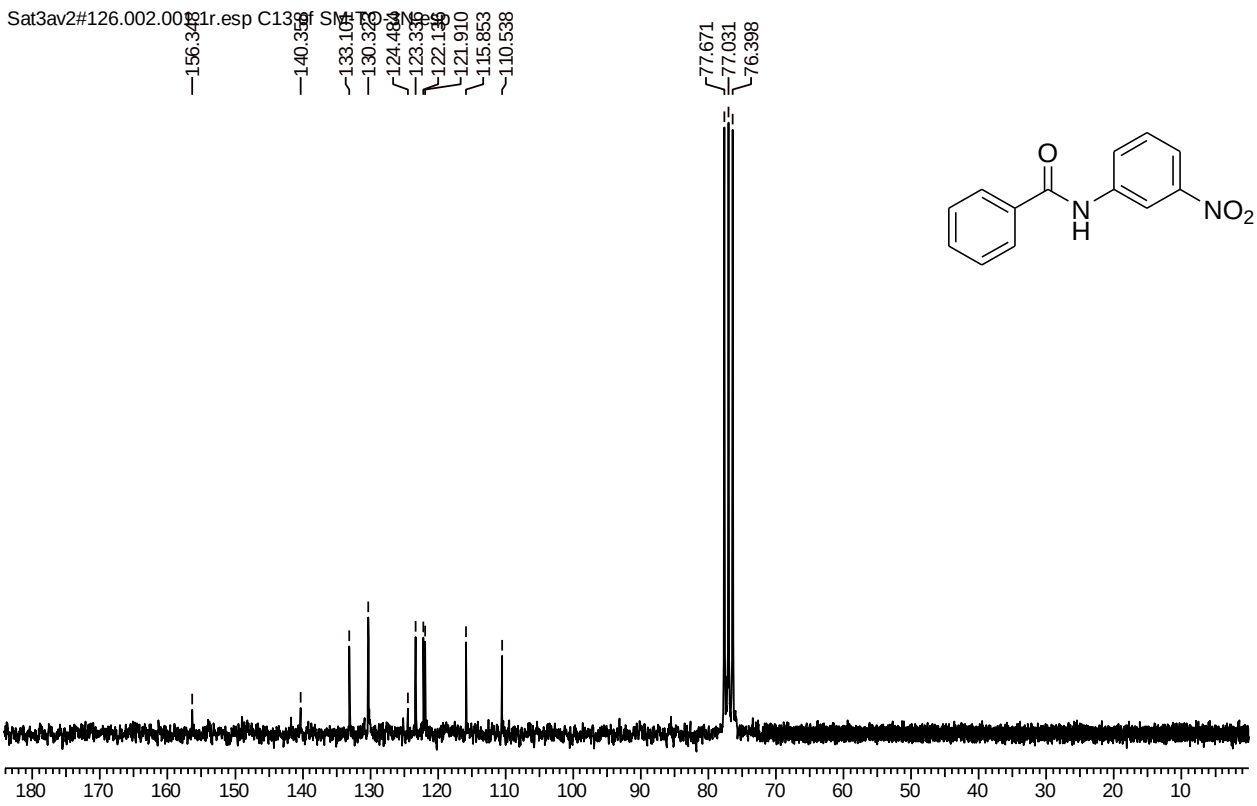
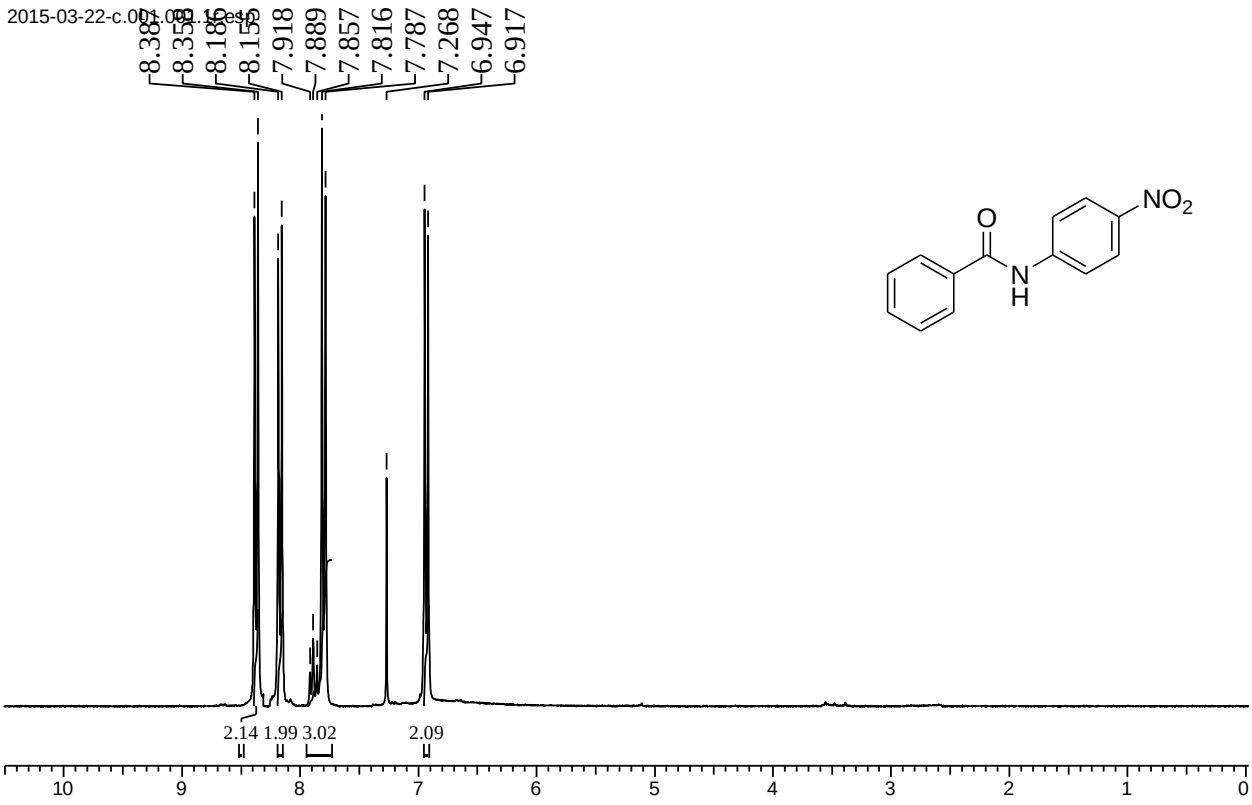
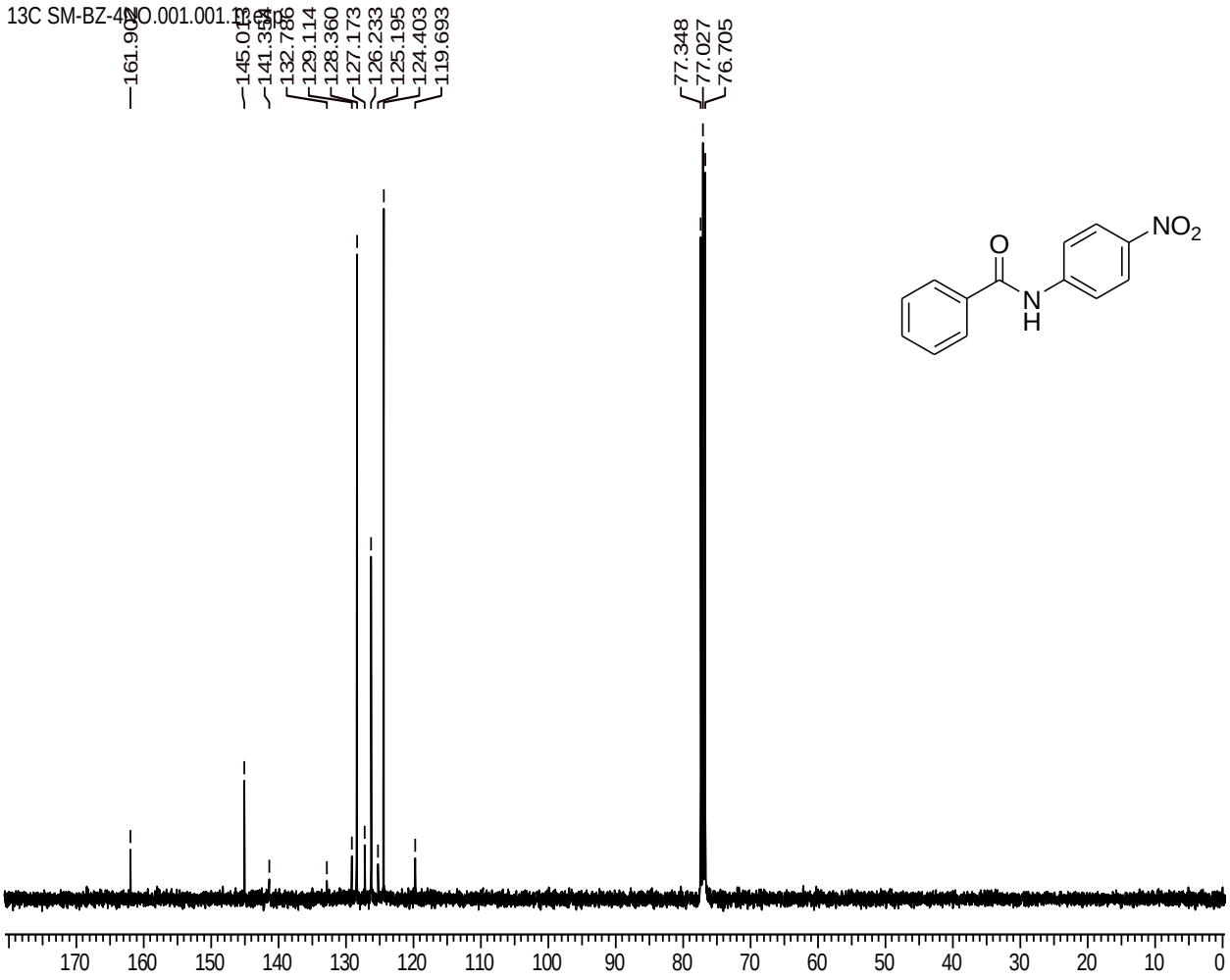
3.00
9
8
7
6
5
4
3
2
1
0

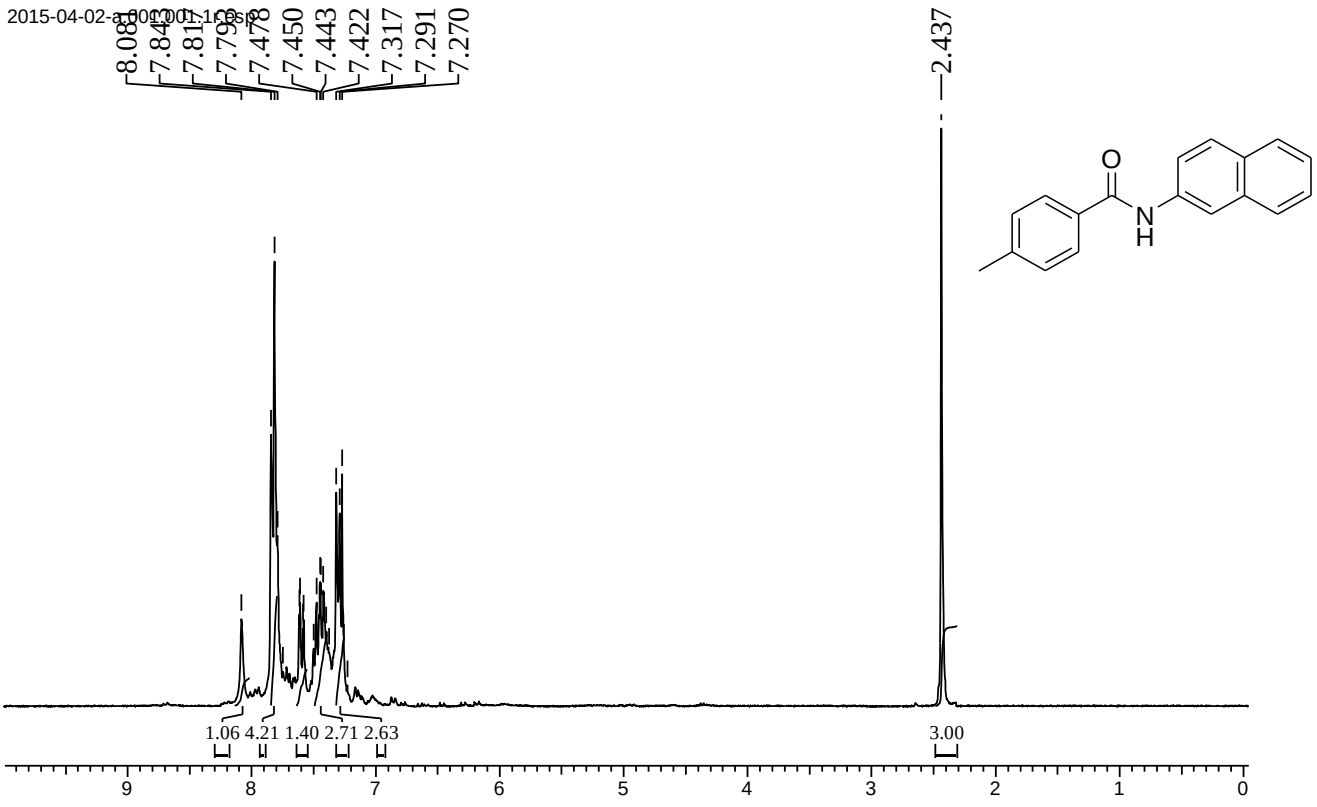

165.66
158.48
142.50
133.95
131.78
129.45
126.98
122.05
115.78
77.25
77.00
76.74
21.49
O
N
H
F
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

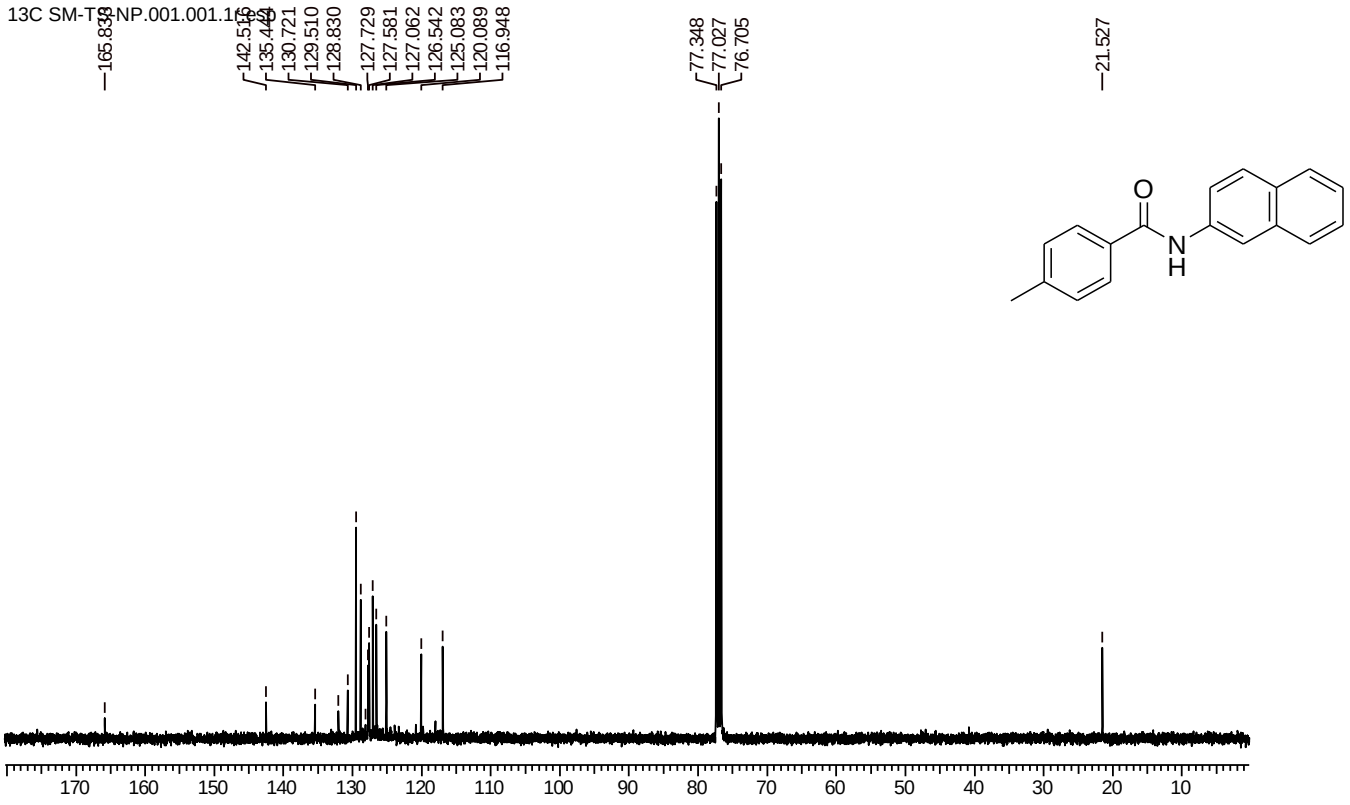

2015-03-23-e001.001.1r.esp
8.512
8.395
8.367
8.330
8.303
7.999
7.974
7.745
7.719
7.692
7.270
2.04
2.87
2.14
2.22
9
8
7
6
5
4
3
2
1
O
N
H
NO2

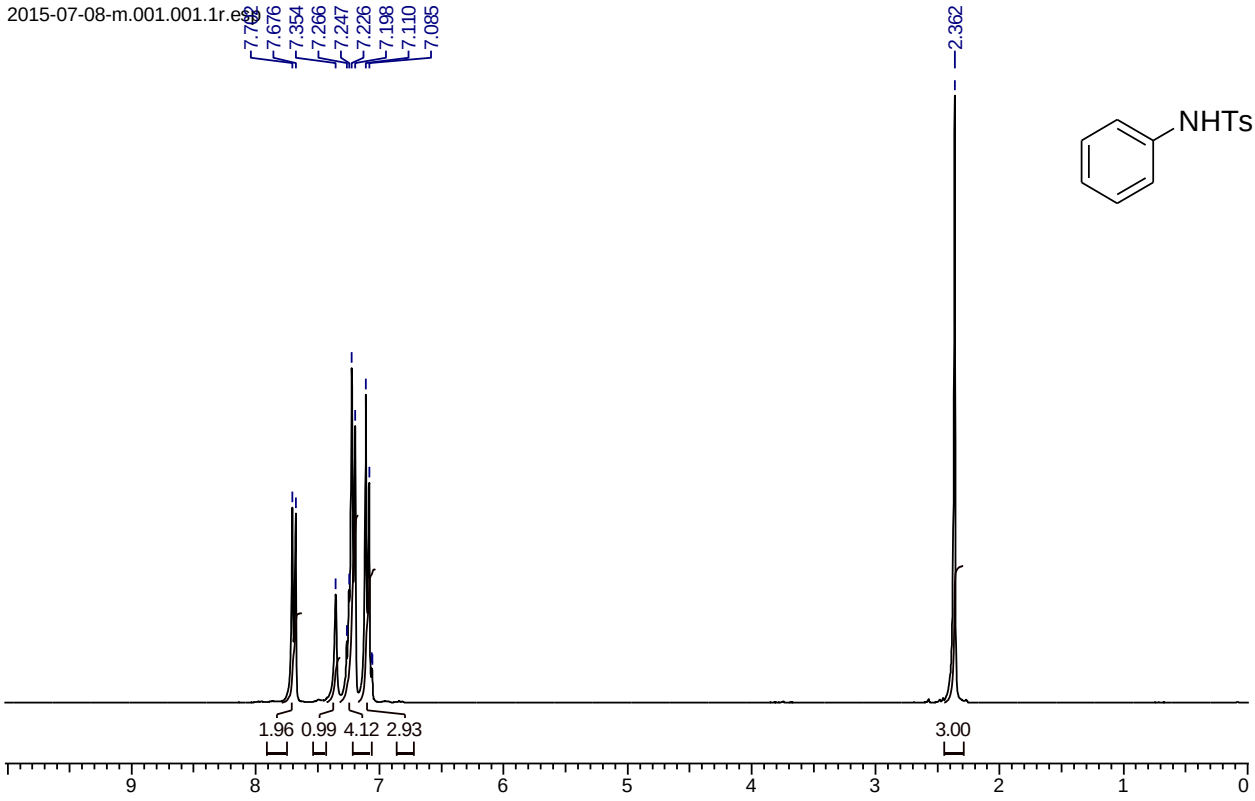

Sat3av2#126.002.001.1r.esp C13 of SM-TO-3N9.esp
156.341
140.354
133.104
130.322
124.464
123.334
122.148
121.910
115.853
110.538
77.671
77.031
76.398
O
N
H
NO2
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

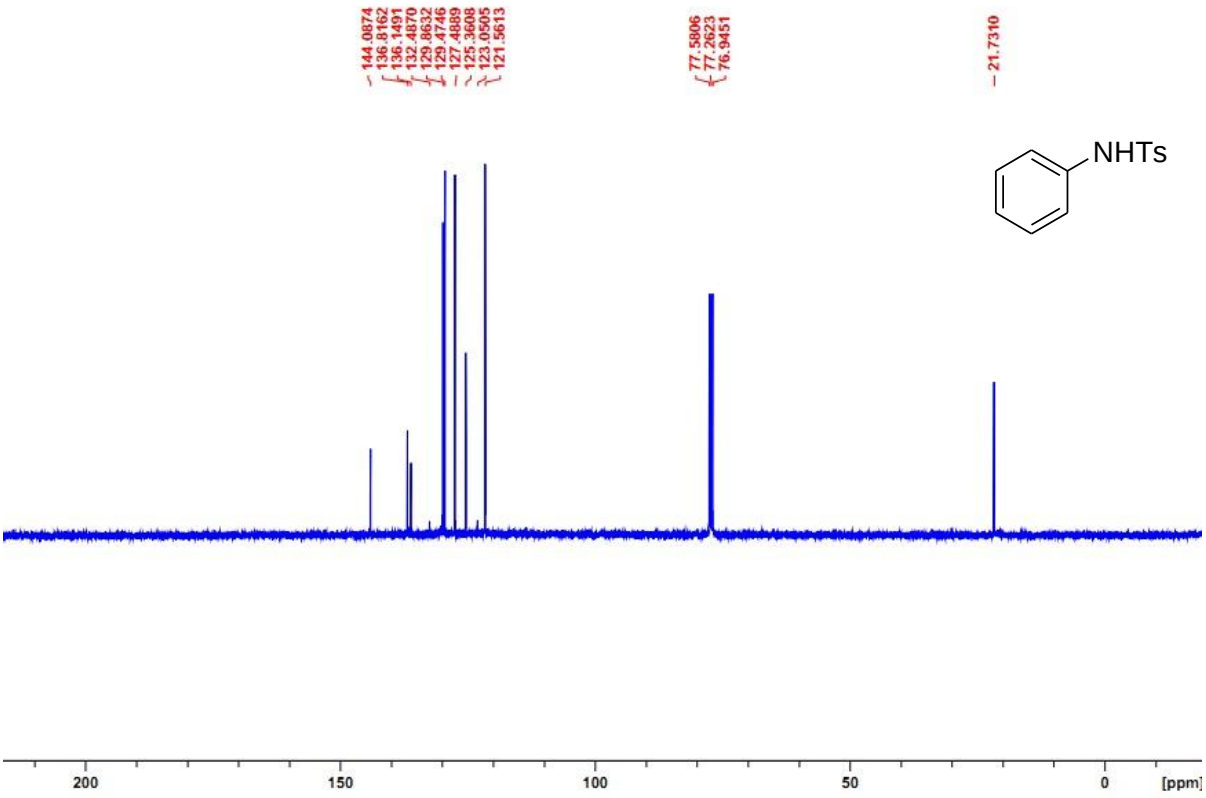

2015-03-22-c.001.001.1r.esp
8.387
8.358
8.185
8.155
7.918
7.889
7.857
7.816
7.787
7.268
6.947
6.917
O
N
H
NO2
2.14
1.99
3.02
2.09
10
9
8
7
6
5
4
3
2
1
0

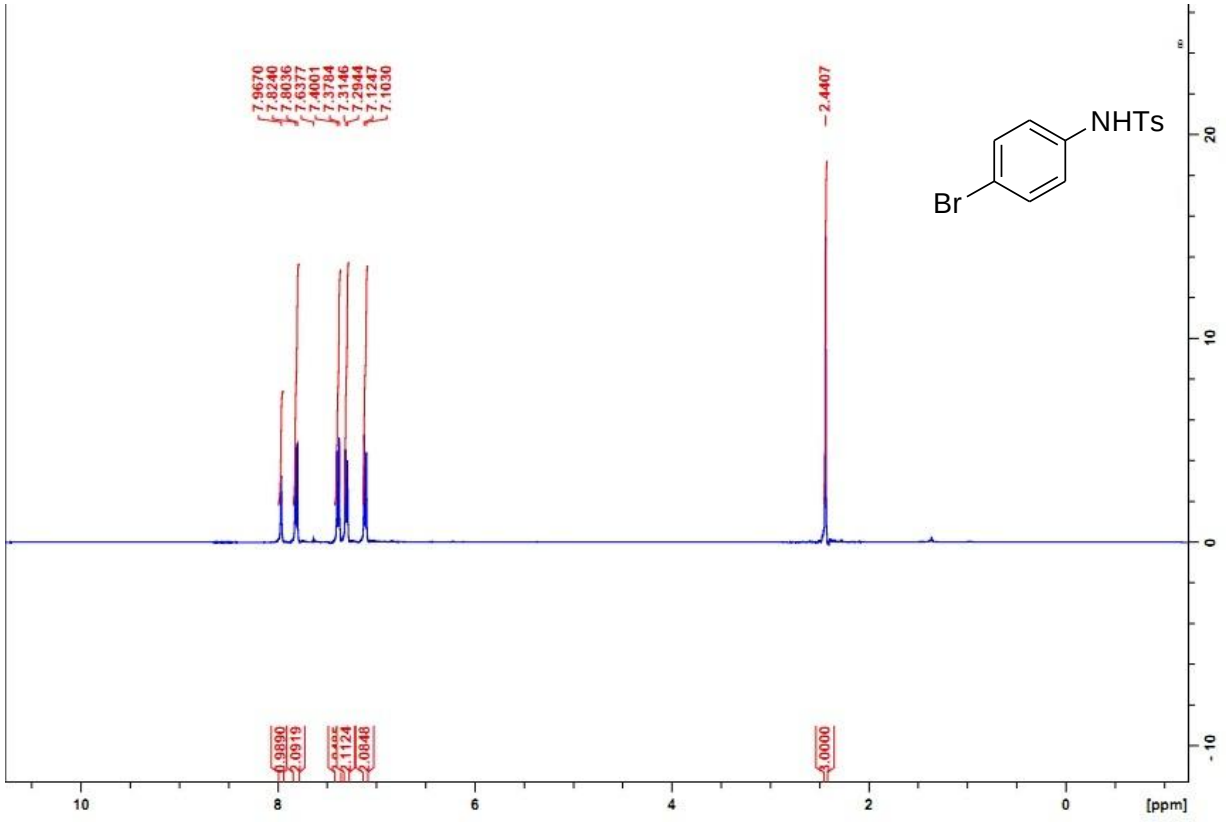

13C SM-BZ-4NO.001.001.1
161.900
145.014
141.350
132.786
129.114
128.360
127.173
126.233
125.195
124.403
119.693
77.348
77.027
76.705
O
N
H
NO2
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

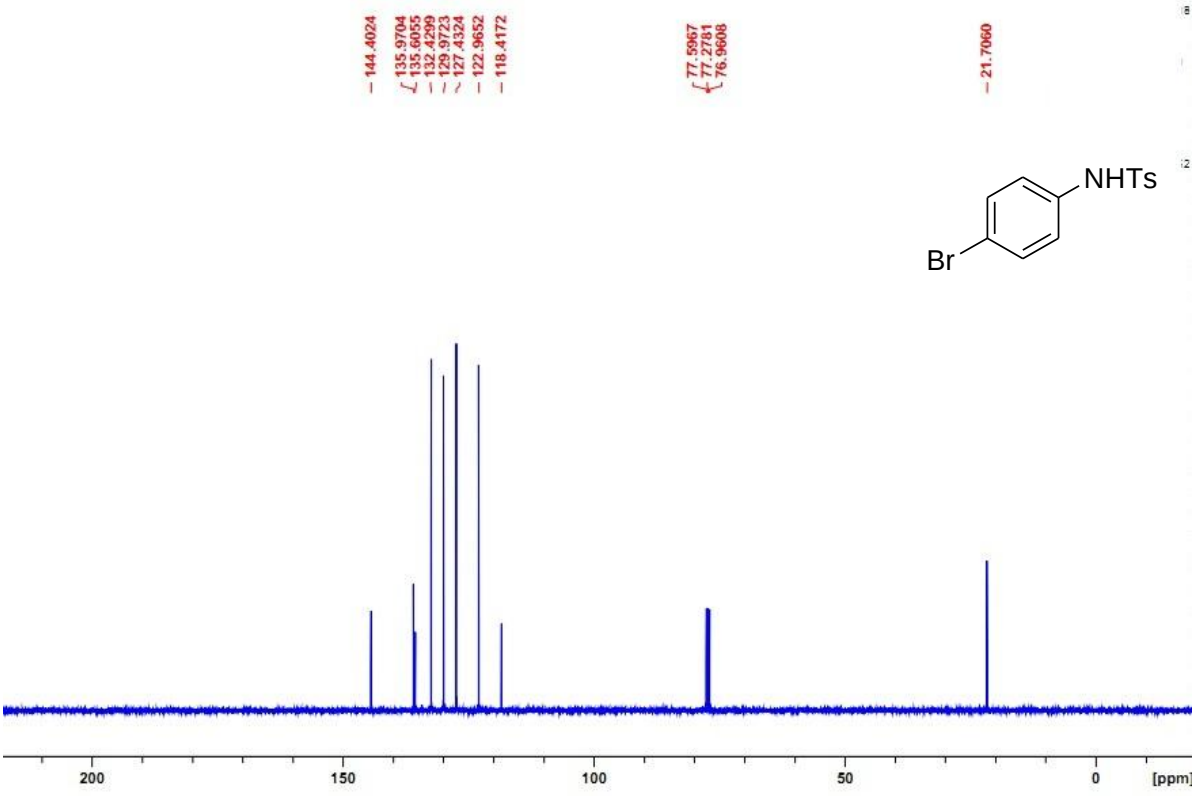

2015-04-02-a_001001_1c.esp
8.088
7.842
7.817
7.795
7.478
7.450
7.443
7.422
7.317
7.291
7.270
2.437
O
N
H
1.06 4.21 1.40 2.71 2.63
3.00
9
8
7
6
5
4
3
2
1
0

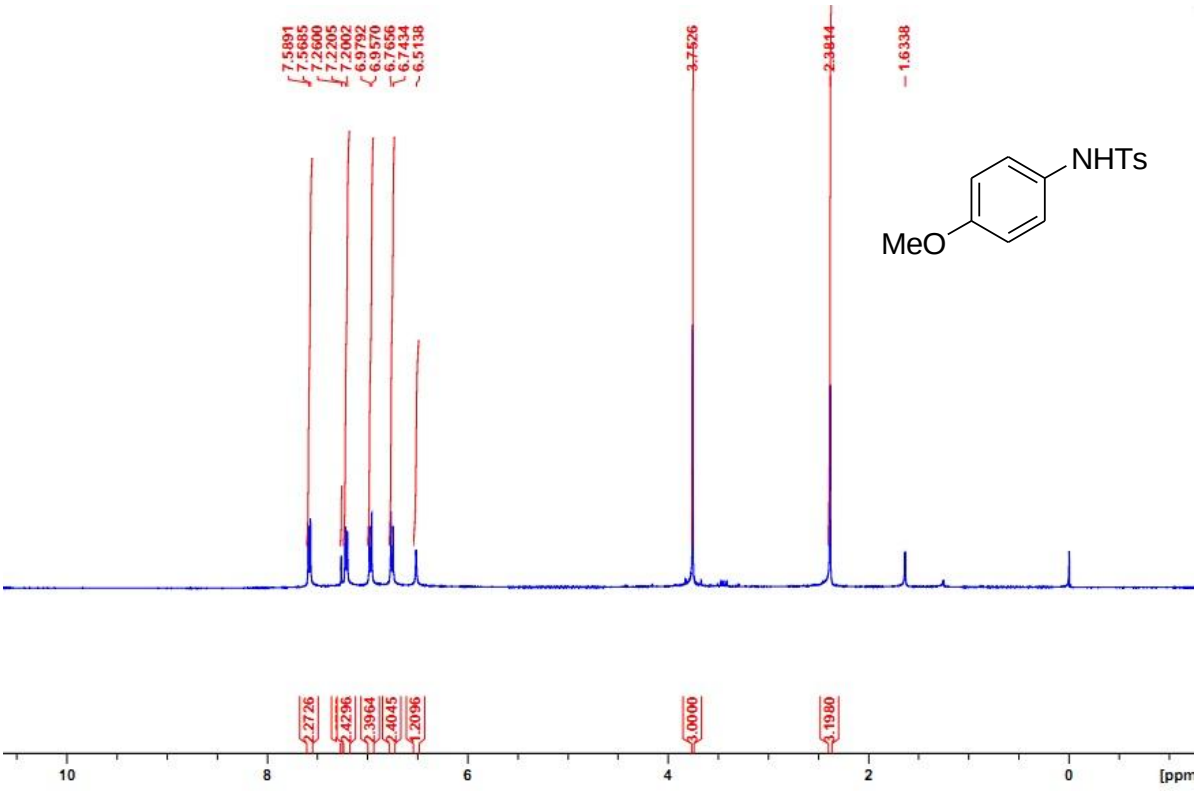

13C SM-TS-NP.001.001.1H3
165.838
142.516
135.446
130.721
129.510
128.830
127.729
127.581
127.062
126.542
125.083
120.089
116.948
77.348
77.027
76.705
21.527
O
N
H
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

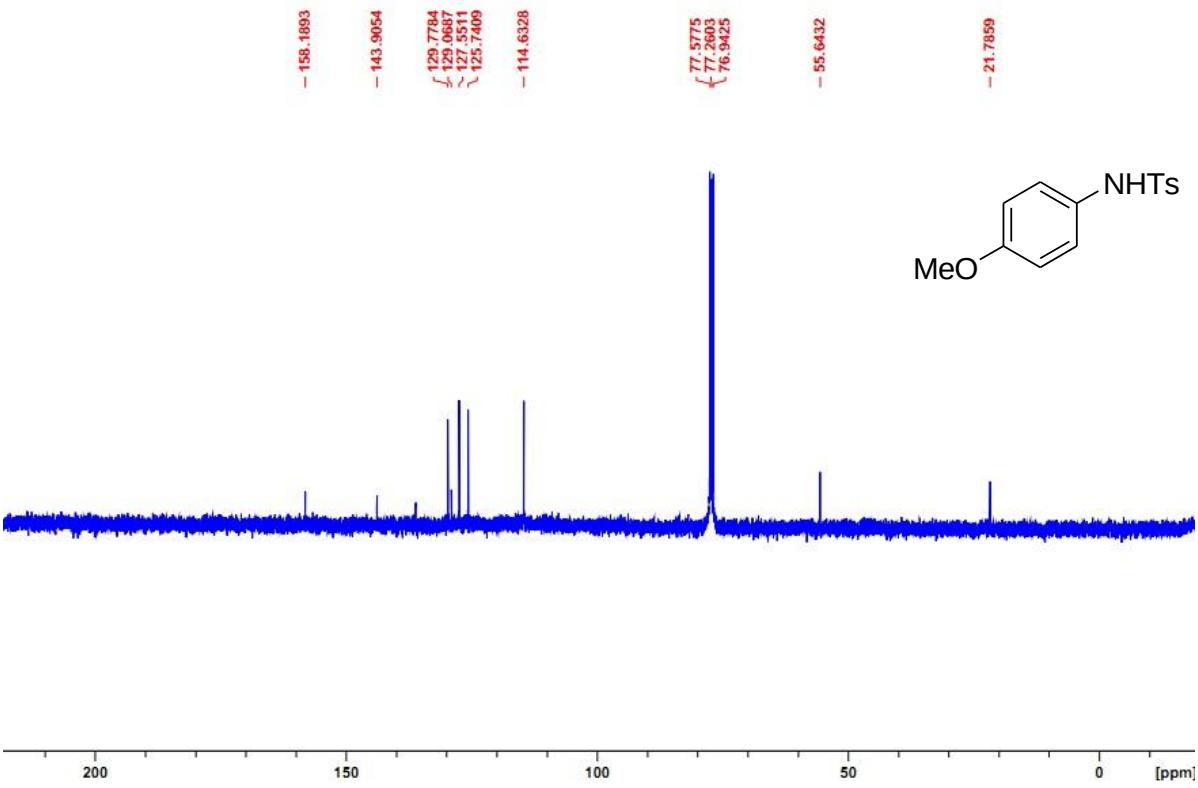

2015-07-08-m.001.001.1r.esp
7.768
7.676
7.354
7.266
7.247
7.226
7.198
7.110
7.085
2.362
NHTs
1.96
0.99
4.12
2.93
3.00
9
8
7
6
5
4
3
2
1
0

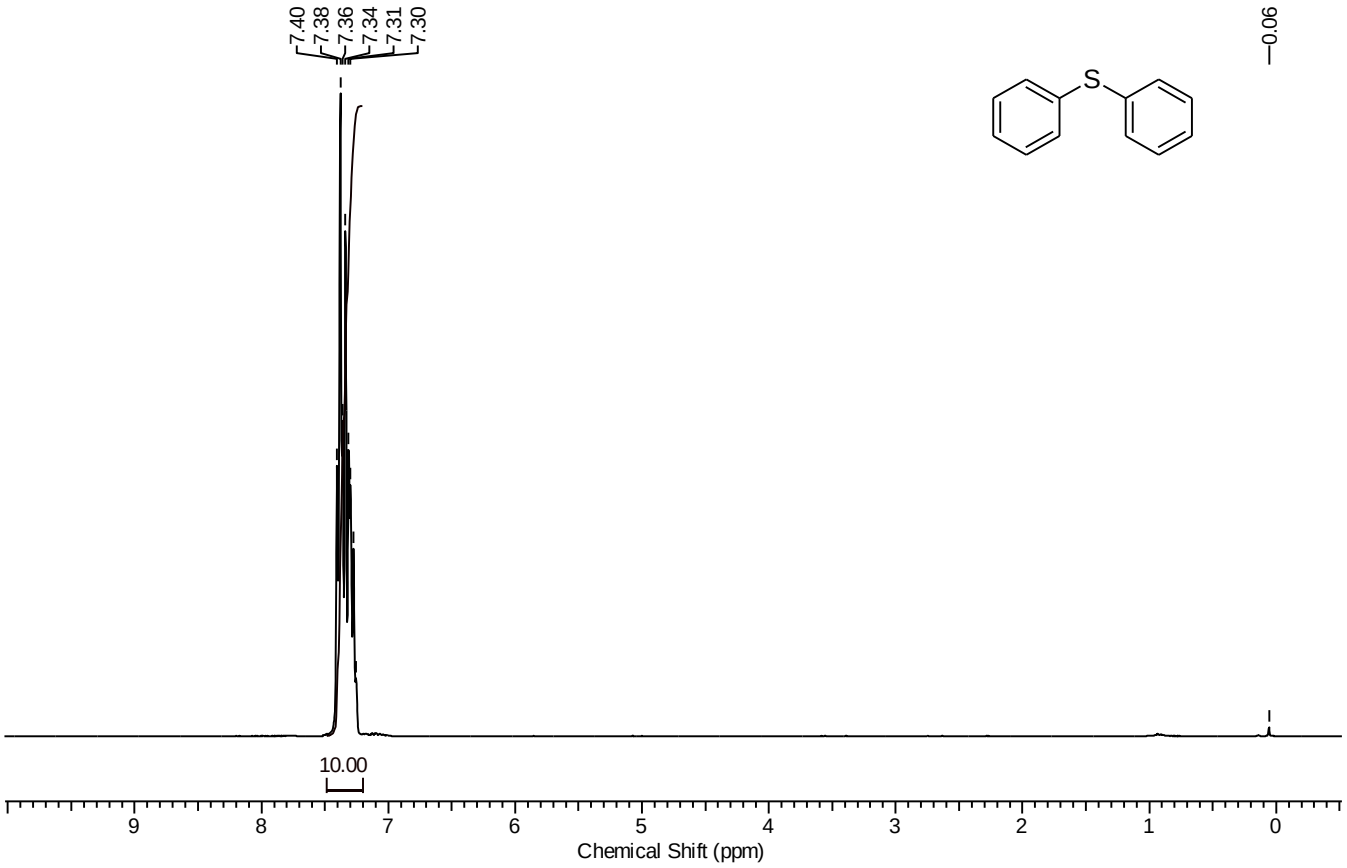

144.0674
136.8162
136.1491
132.4870
129.8632
129.4746
127.4889
125.3608
123.0505
121.5613
77.5806
77.2623
76.9451
21.7310
NHTs
200
150
100
50
0
[ppm]

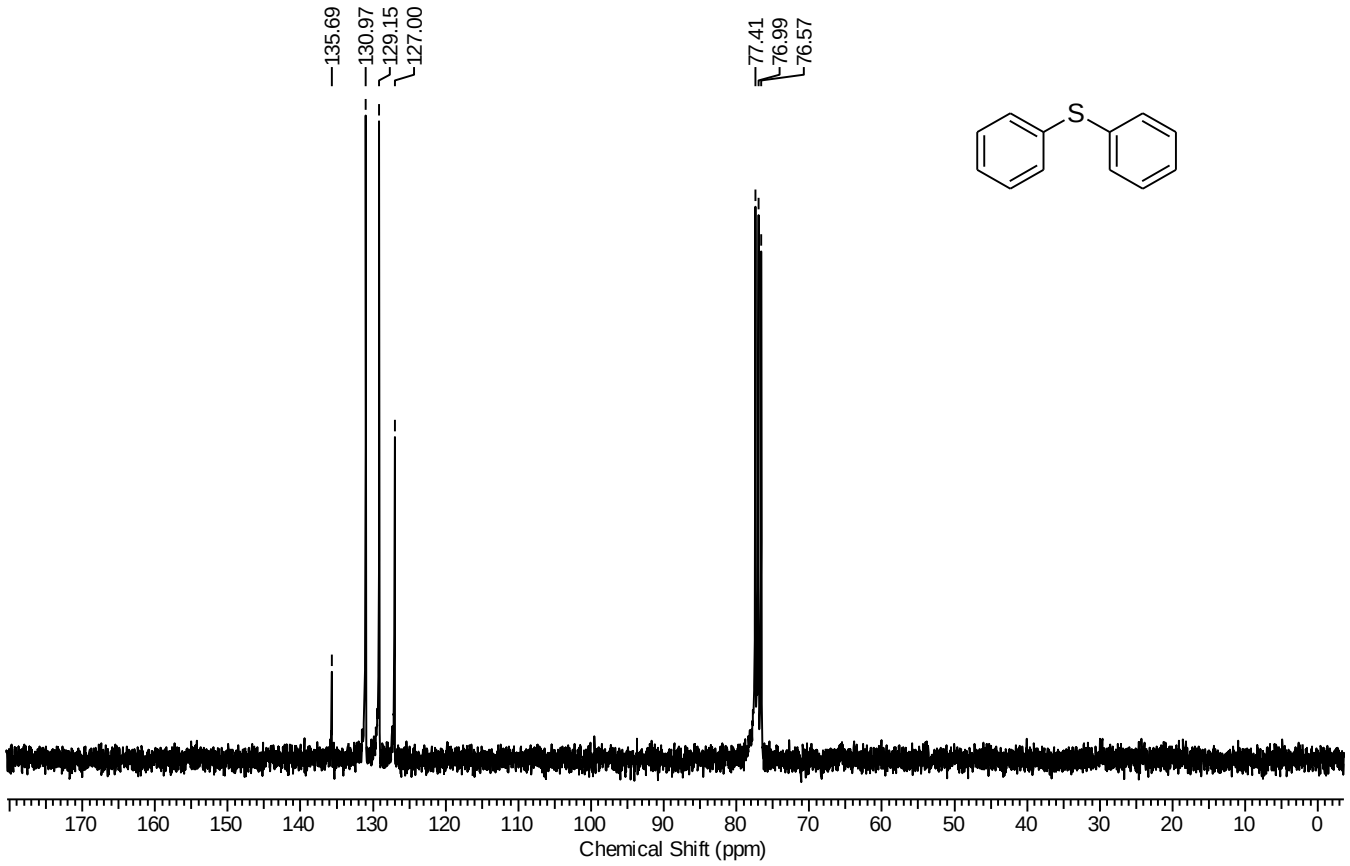

7.9670
7.8240
7.8036
7.6377
7.4001
7.3784
7.3146
7.2944
7.1247
7.1030
2.4407
NHTs
Br
0.9890
2.0919
2.1124
2.0846
3.0000
10
8
6
4
2
0
[ppm]
20
10
0
-10

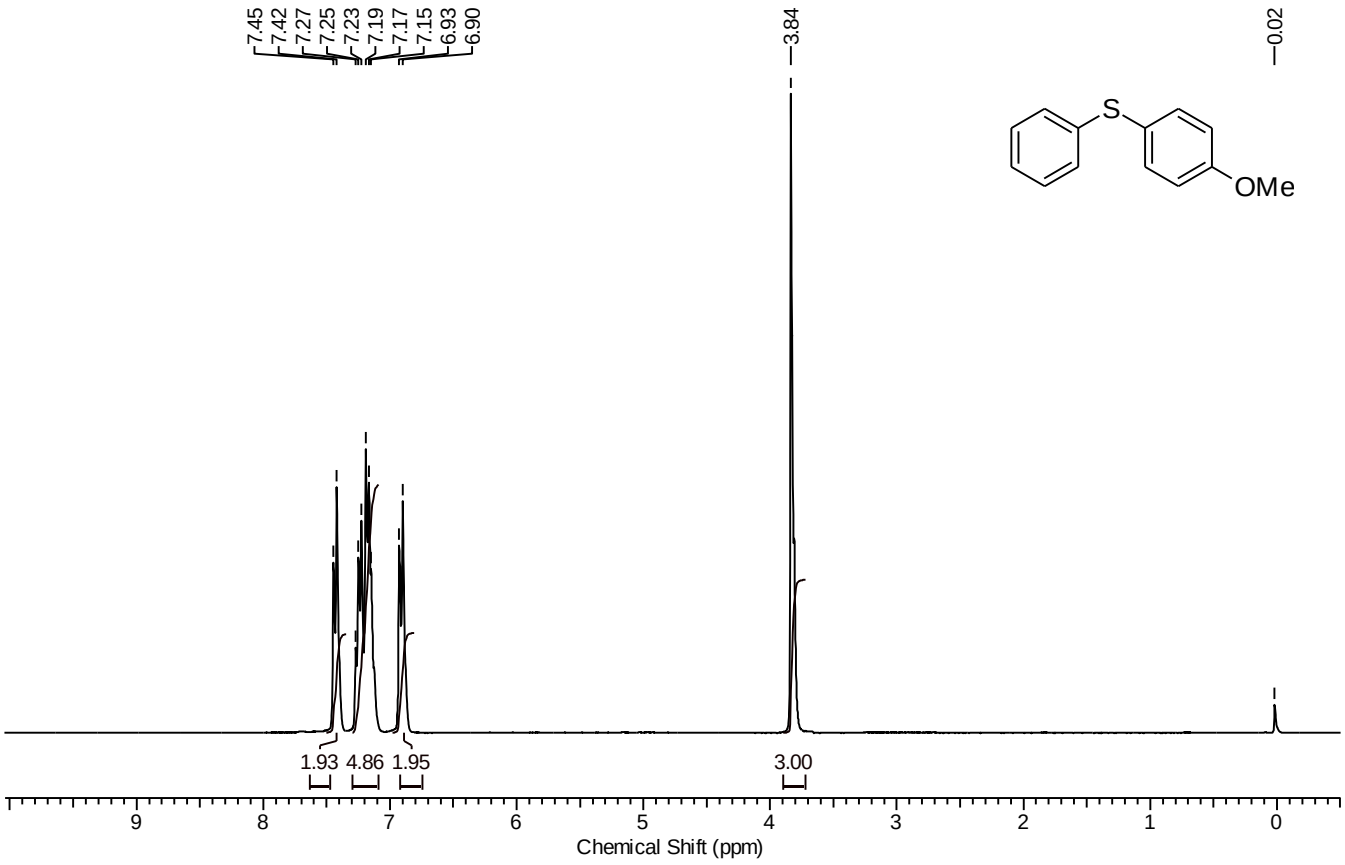

144.4024
135.9704
135.6055
132.4299
129.9723
127.4324
122.9652
118.4172
77.5967
77.2781
76.9608
21.7060
NHTs
Br
200
150
100
50
0
[ppm]

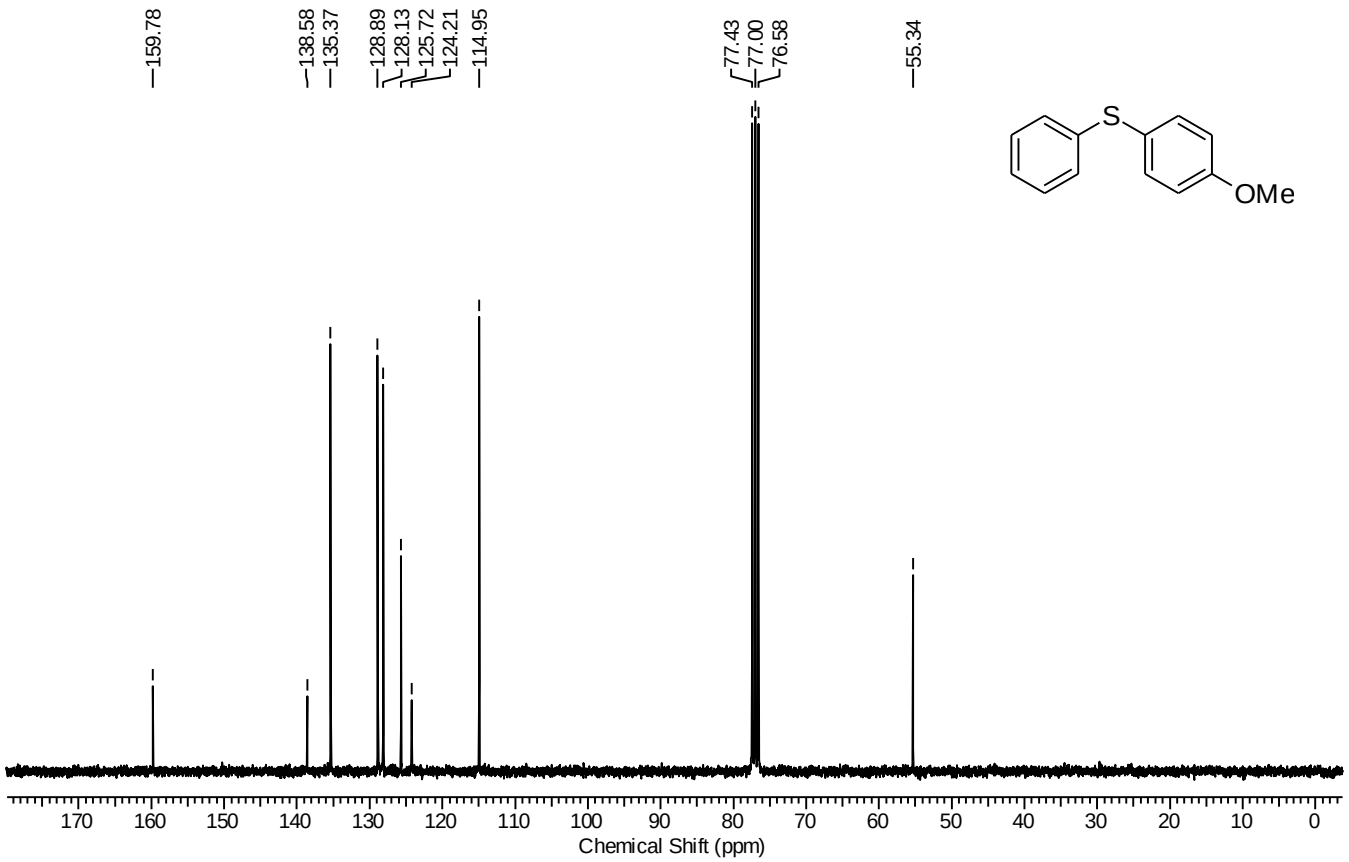

7.5891
7.5685
7.2600
7.2205
7.2002
6.9792
6.9570
6.7656
6.7434
6.5138
3.7526
2.3814
1.6338
NHTs
MeO
2.2726
2.4296
2.3964
2.4045
1.2096
4.0000
3.1980
10
8
6
4
2
0
[ppm

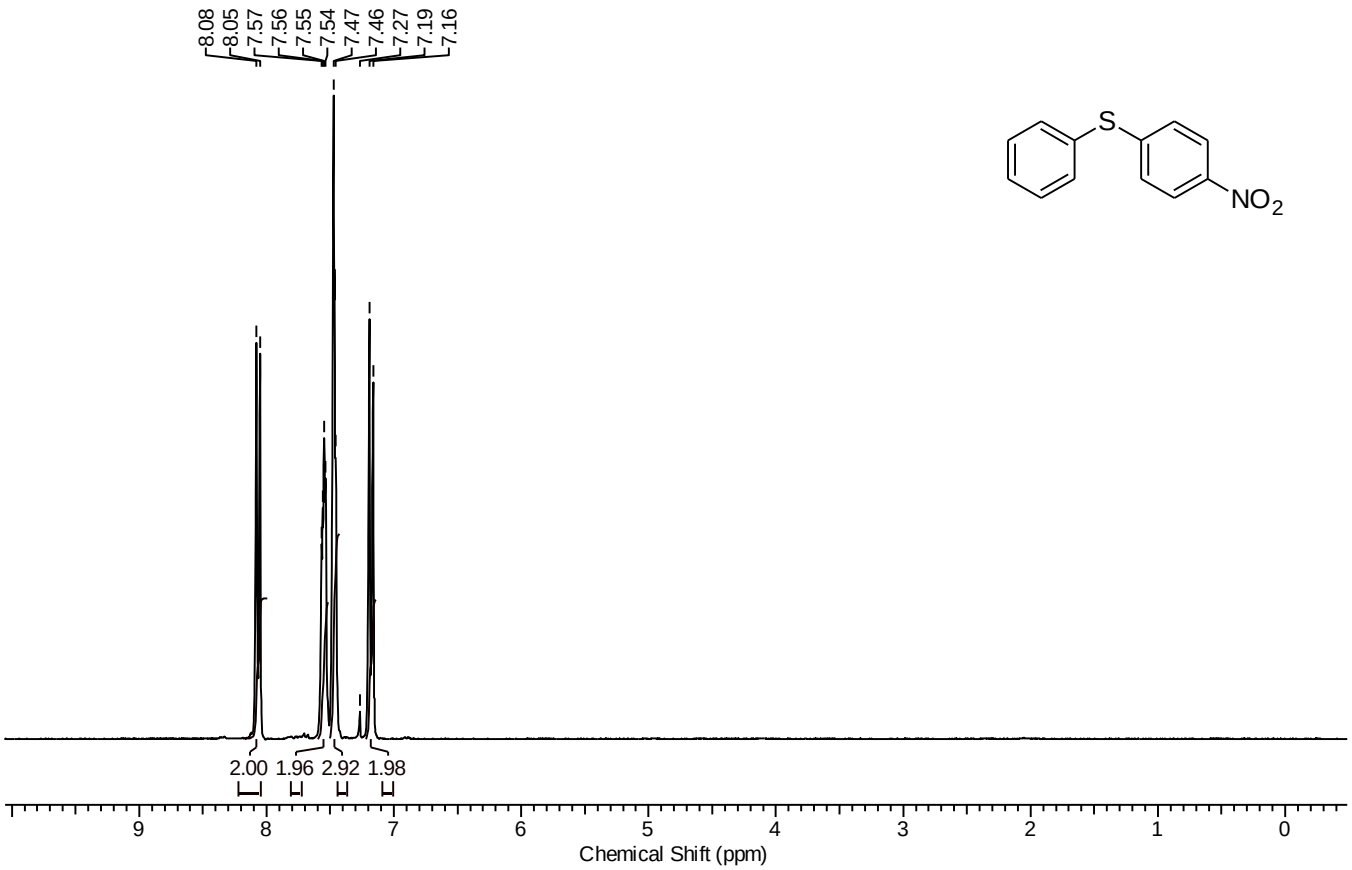

158.1893
143.9054
129.7784
129.0687
127.5511
125.7409
114.6328
77.5775
77.2603
76.9425
55.6432
21.7859
NHTs
MeO
200
150
100
50
0
[ppm]

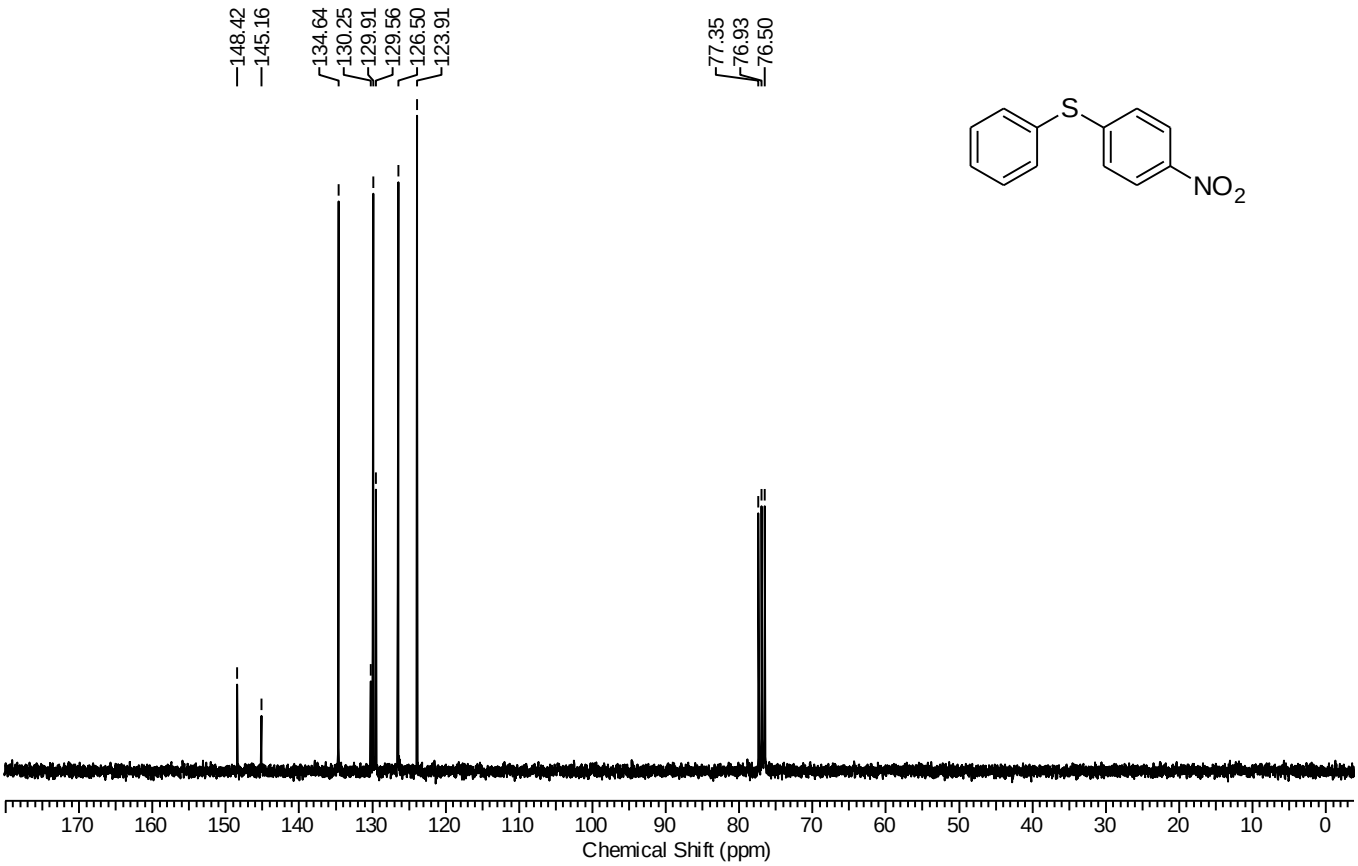

7.40
7.38
7.36
7.34
7.31
7.30
0.06
S
10.00
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

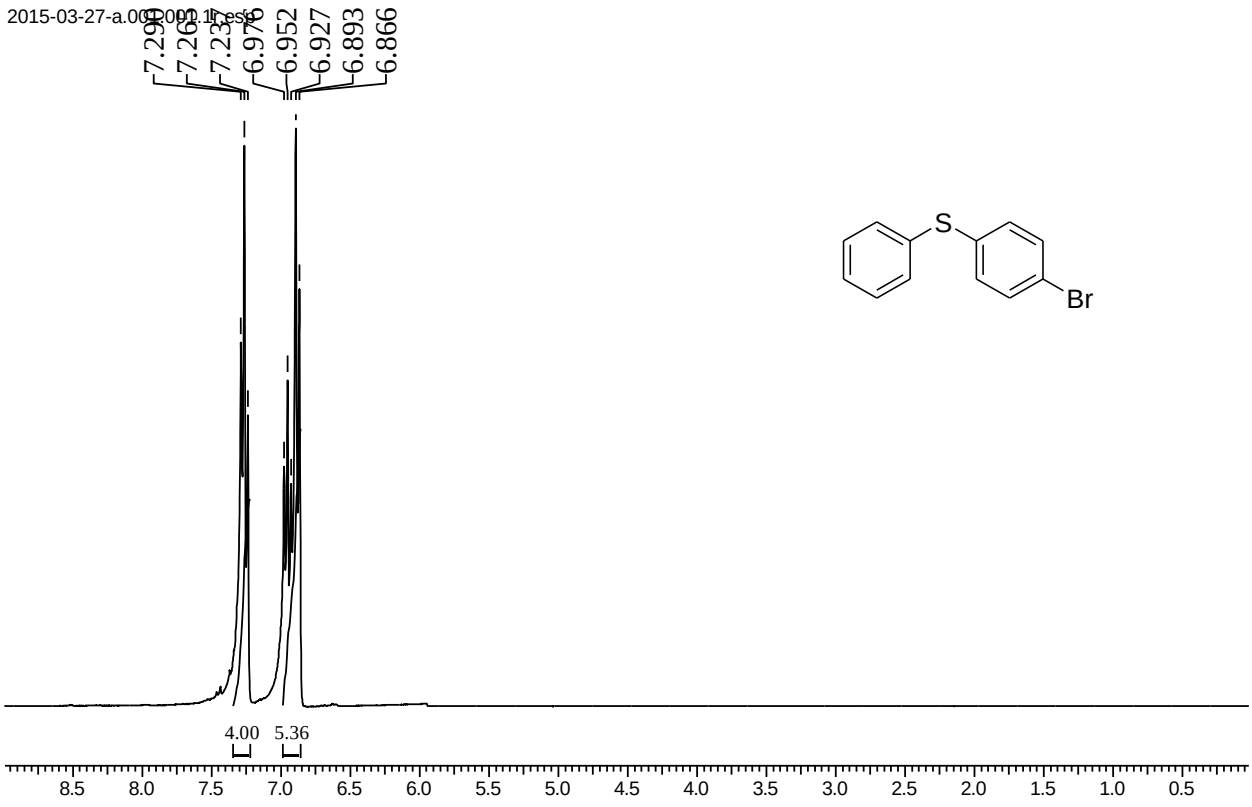

135.69
130.97
129.15
127.00
77.41
76.99
76.57
S
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

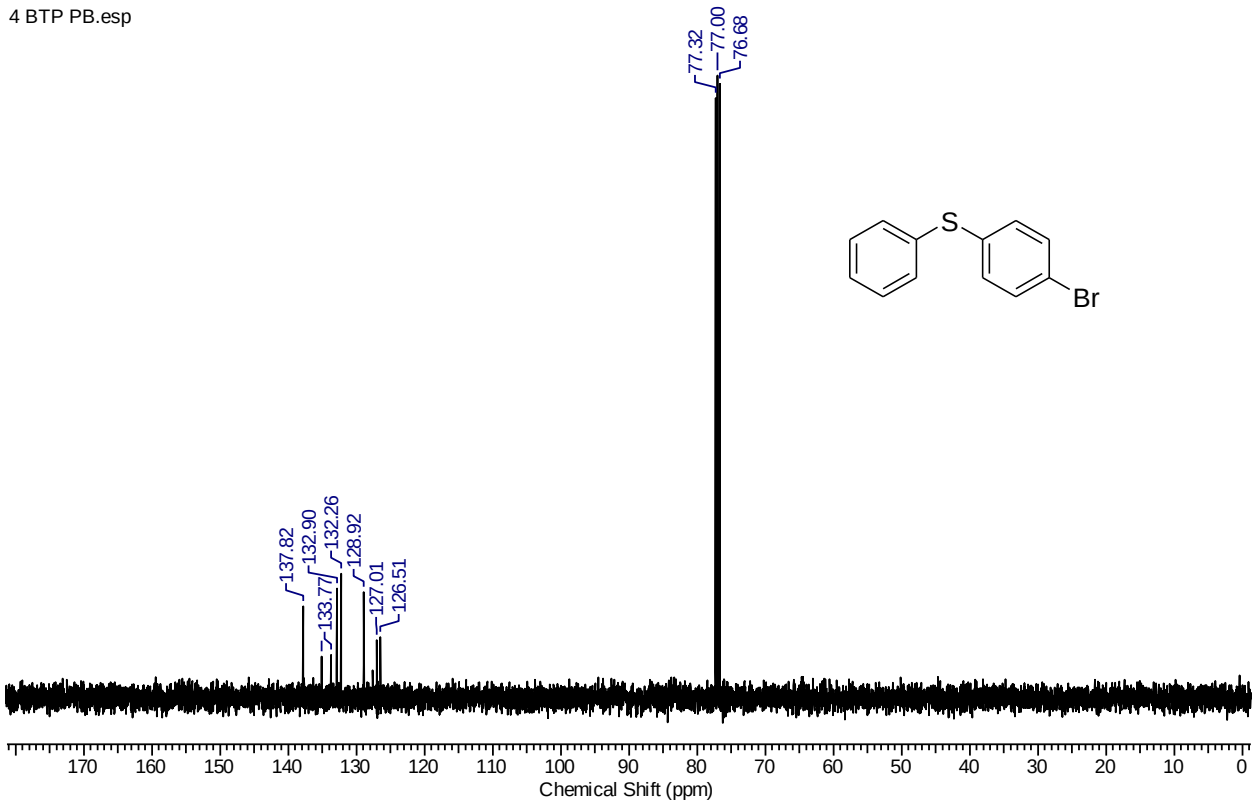

7.45
7.42
7.27
7.25
7.23
7.19
7.17
7.15
6.93
6.90
3.84
0.02
S
OMe
1.93
4.86
1.95
3.00
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

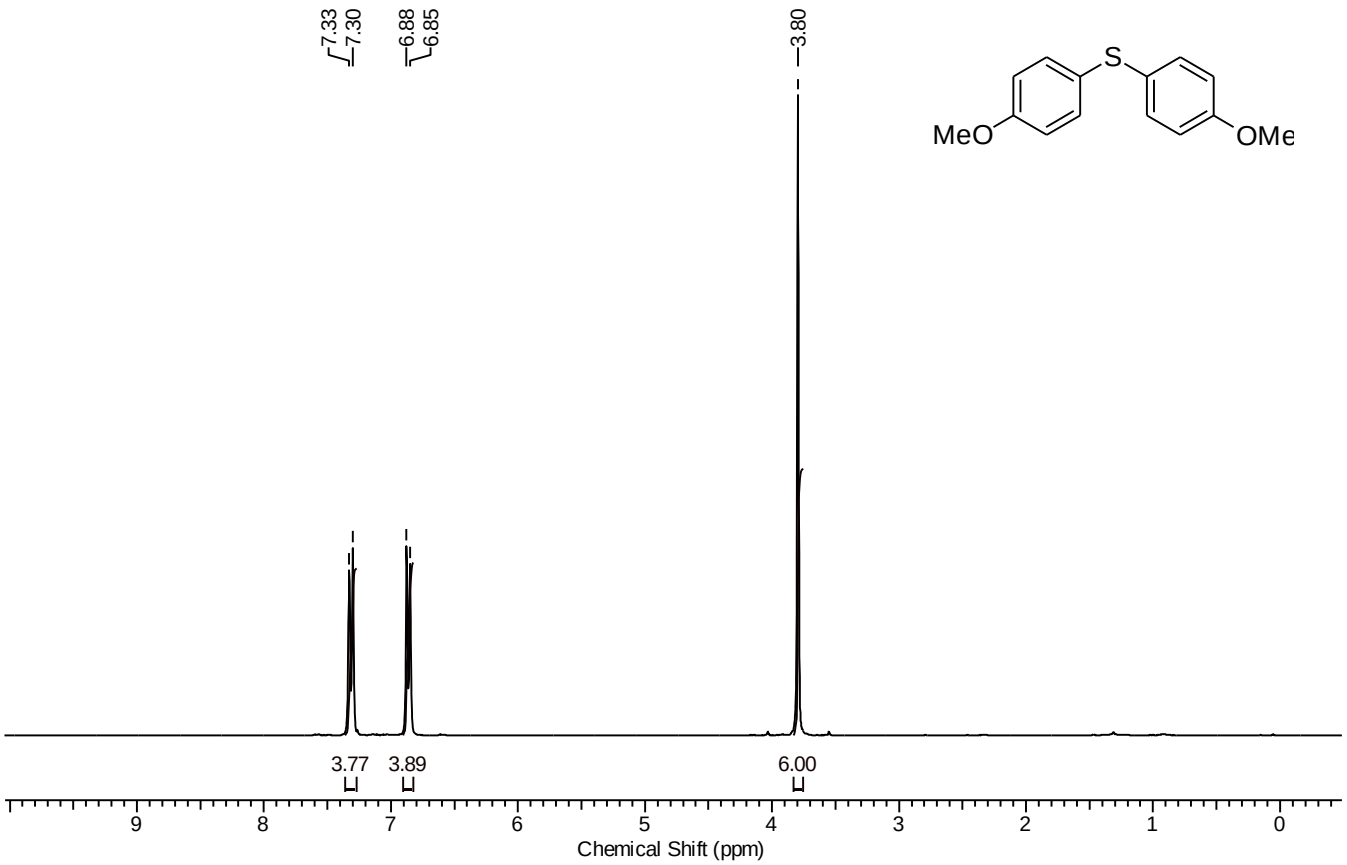

159.78
138.58
135.37
128.89
128.13
125.72
124.21
114.95
77.43
77.00
76.58
55.34
S
OMe
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

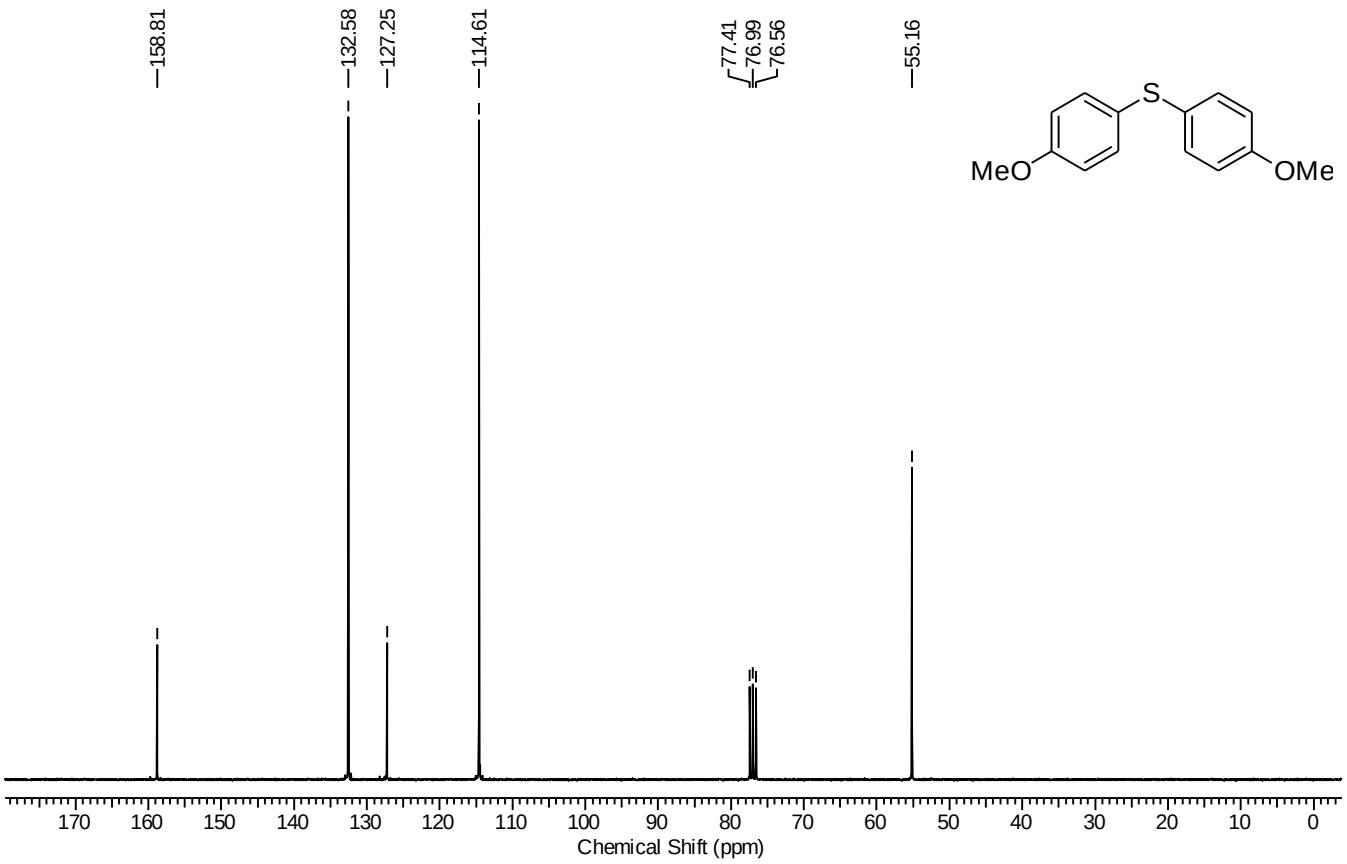

8.08
8.05
7.57
7.56
7.55
7.54
7.47
7.46
7.27
7.19
7.16
S
NO2
2.00 1.96 2.92 1.98
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

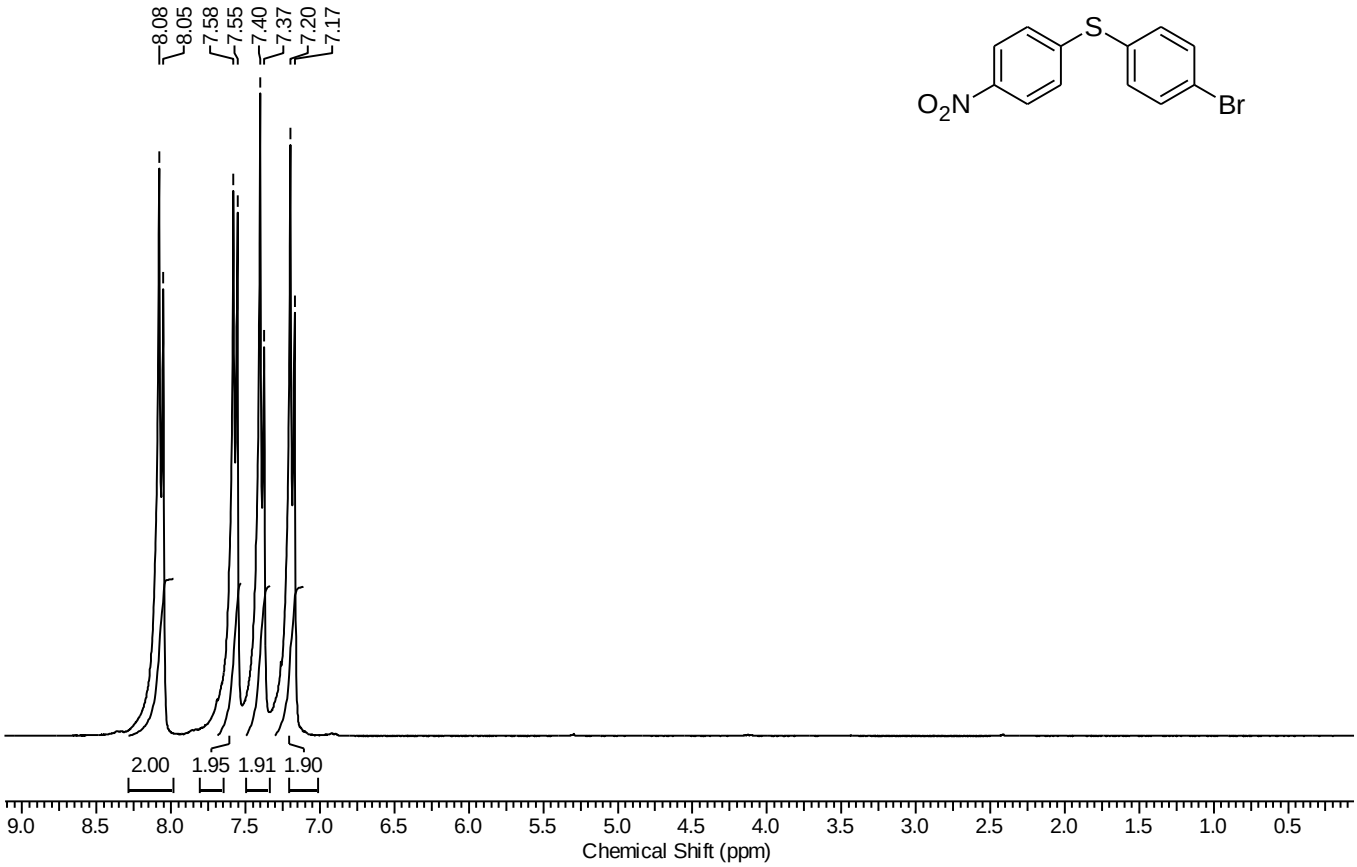

148.42
145.16
134.64
130.25
129.91
129.56
126.50
123.91
77.35
76.93
76.50
S
NO2
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

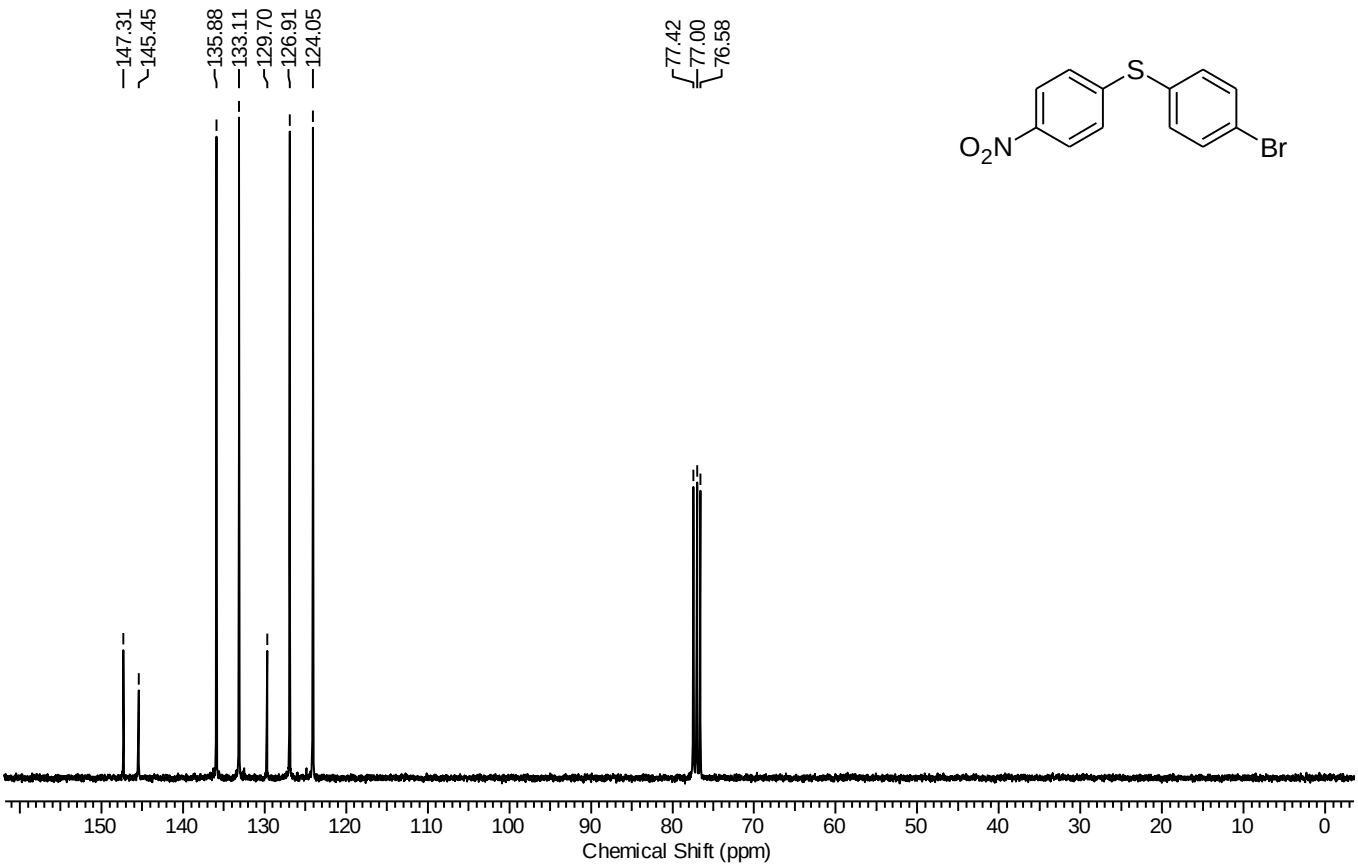

2015-03-27-a.001.2001.1r.esp
7.299
7.266
7.236
6.976
6.952
6.927
6.893
6.866
S
Br
4.00
5.36
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

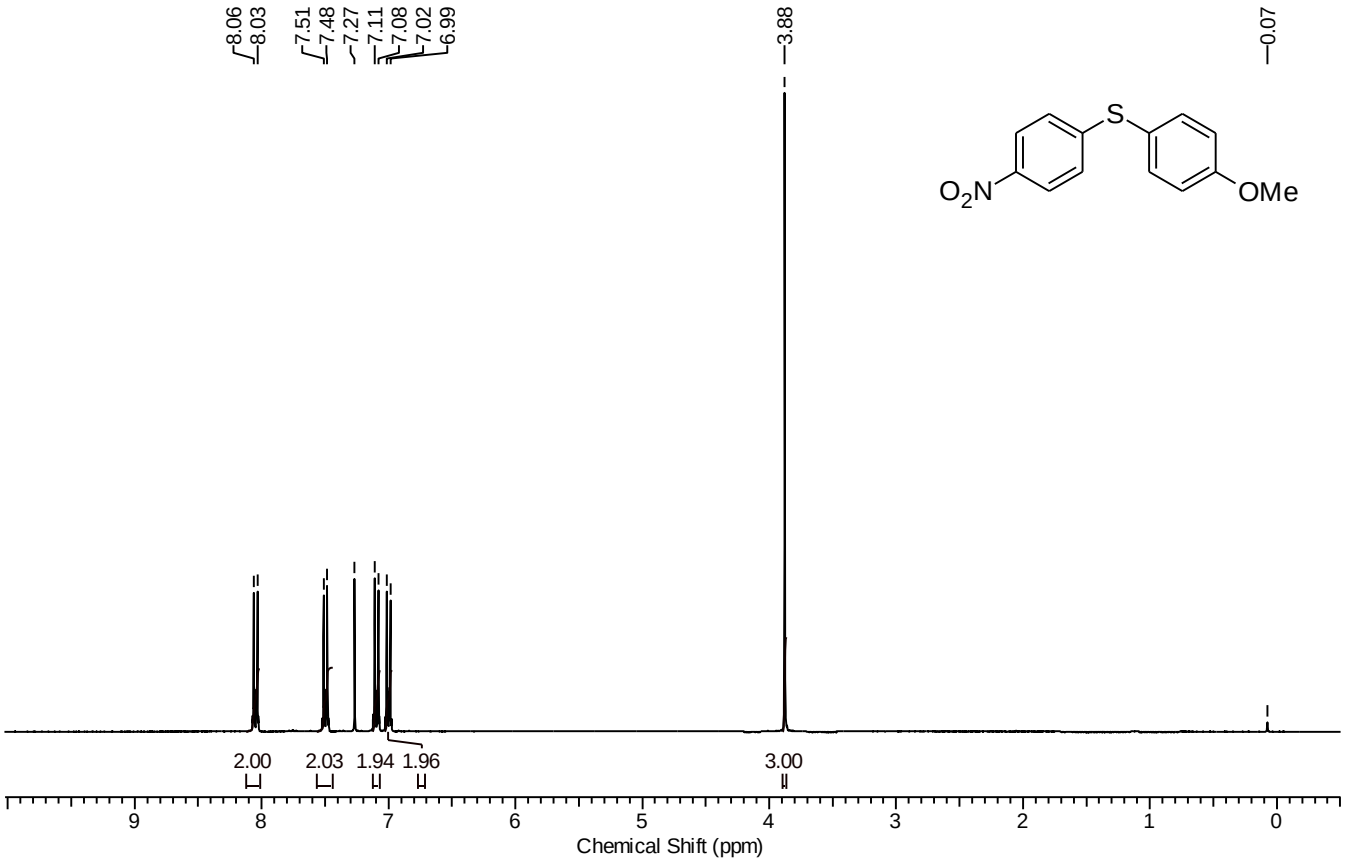

4 BTP PB.esp
77.32
77.00
76.68
137.82
132.90
133.77
132.26
128.92
127.01
126.51
S
Br
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

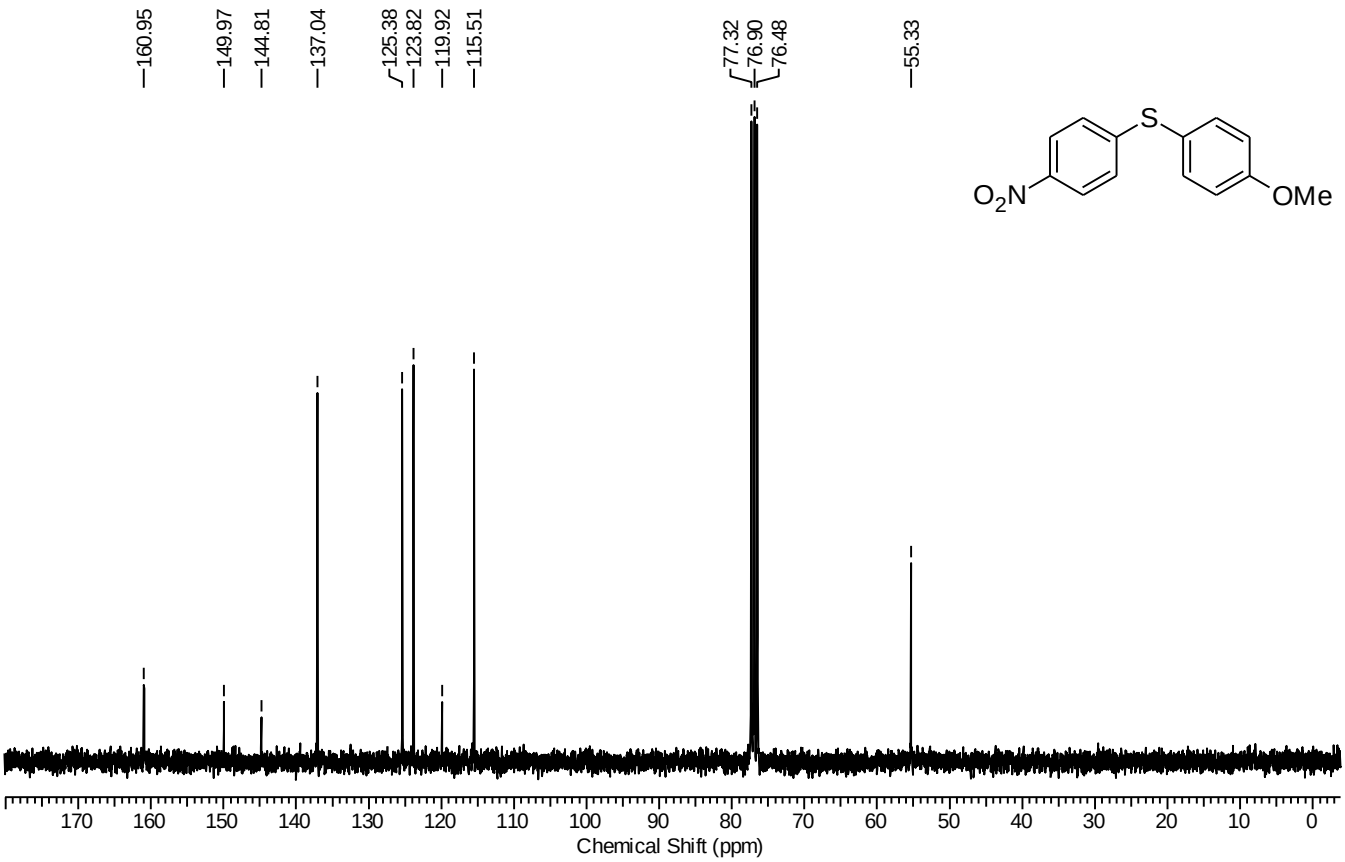

7.33
7.30
6.88
6.85
3.80
S
MeO
OMe
3.77
3.89
6.00
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

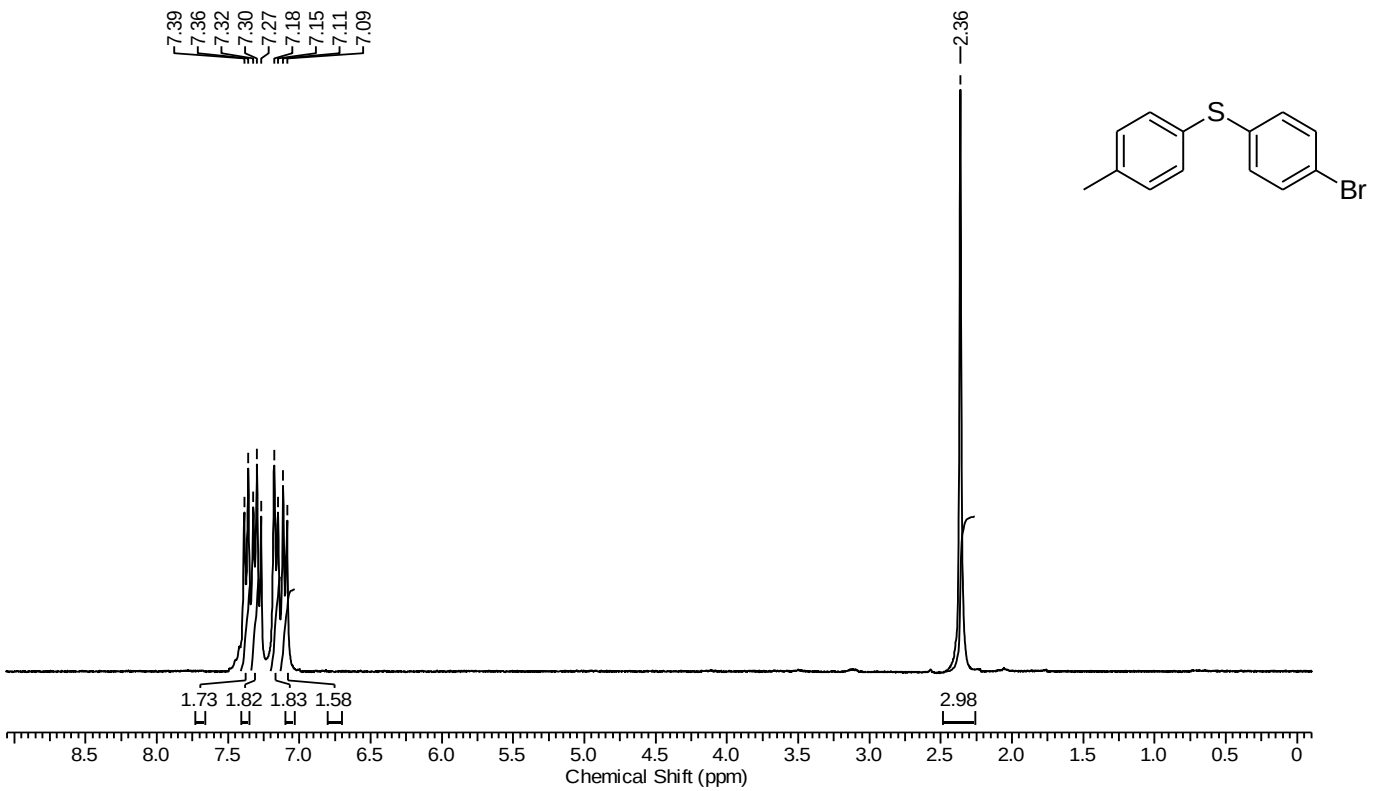

158.81
132.58
127.25
114.61
77.41
76.99
76.56
55.16
S
MeO
OMe
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

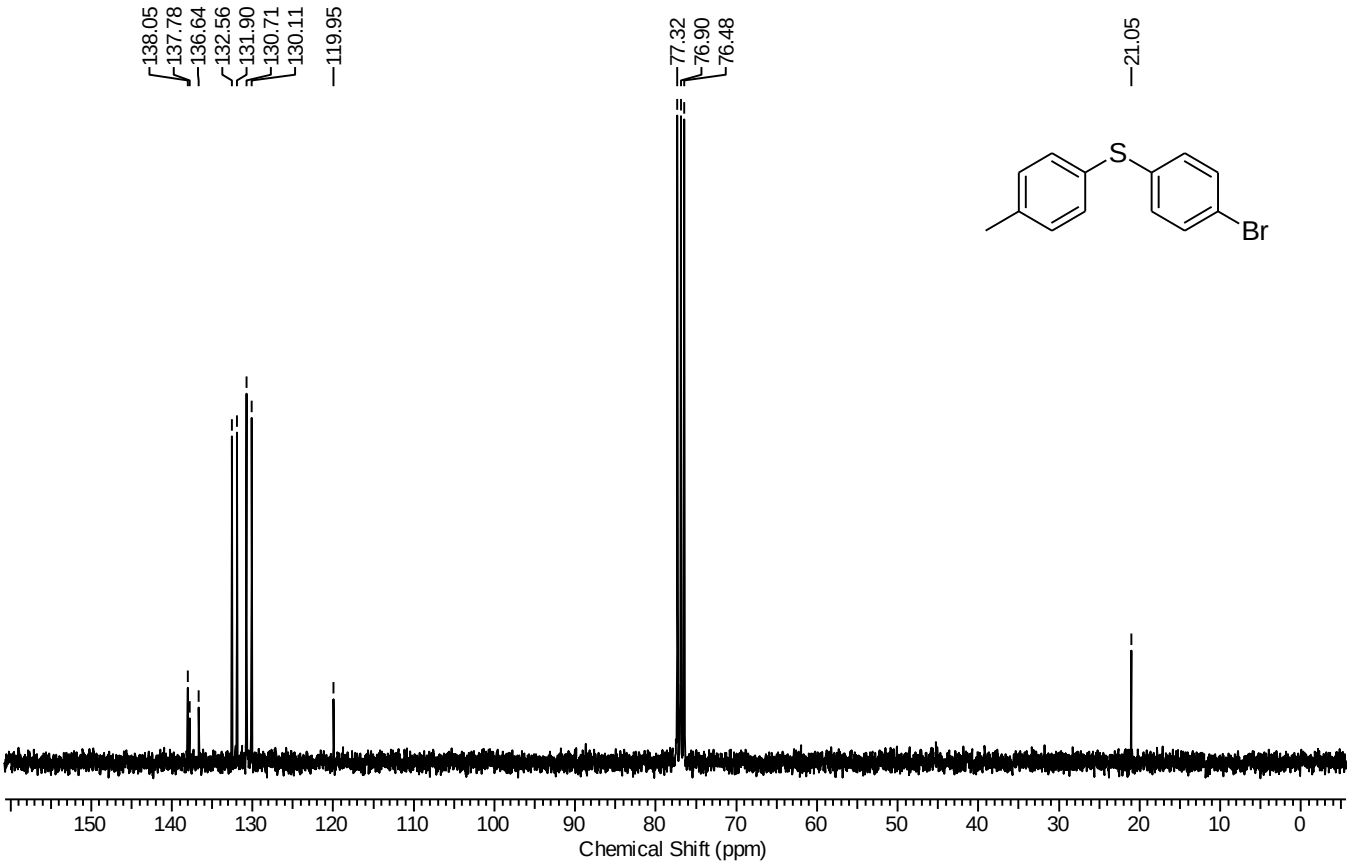

8.08
8.05
7.58
7.55
7.40
7.37
7.20
7.17
S
O2N
Br
2.00
1.95
1.91
1.90
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
Chemical Shift (ppm)


147.31
145.45
135.88
133.11
129.70
126.91
124.05
77.42
77.00
76.58
S
O2N
Br
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)


8.06
8.03
7.51
7.48
7.27
7.11
7.08
7.02
6.99
3.88
0.07
S
O_2N
OMe
2.00
2.03
1.94
1.96
3.00
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)


160.95
149.97
144.81
137.04
125.38
123.82
119.92
115.51
77.32
76.90
76.48
55.33
S
O2N
OMe
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)


7.39
7.36
7.32
7.30
7.27
7.18
7.15
7.11
7.09
2.36
S
Br
1.73
1.82
1.83
1.58
2.98
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
Chemical Shift (ppm)


138.05
137.78
136.64
132.56
131.90
130.71
130.11
119.95
77.32
76.90
76.48
21.05
S
Br
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)