Supporting Information

Vicinal functionalization of olefins: a facile route to direct synthesis of β-chlorohydrins and β-chloroethers

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General information

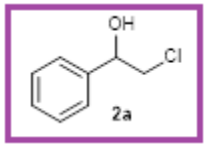
All chemicals used were reagent grade and used as received without further purification. ^{1}H NMR spectra were recorded at 300 or 500 MHz and ^{13}C NMR spectra at 75 or 125 MHz in $CDCl_3$. The chemical shifts (δ) are reported in ppm units relative to TMS as an internal standard for ^{1}H NMR and $CDCl_3$ for ^{13}C NMR spectra. Coupling constants (*J*) are reported in hertz (Hz) and multiplicities are indicated as follows: s (singlet), br s (broad singlet), d (doublet), dd (doublet of doublet), m (multiplet). Column chromatography was carried out using silica gel (100-200 mesh) and TLC inspections were performed on Silica gel 60 F_{254} plates.

Typical procedure for vicinal functionalization of olefins

Oxone® (2.2 mmol) was slowly added to a well stirred solution of NH_4Cl (2.2 mmol) and olefin (2 mmol) in acetone-H_2O (1:1; 10 mL) or methanol (5 mL) and the reaction mixture was allowed to stir at room temperature, until olefin was completely disappeared (monitored by TLC, eluent: *n*-hexane-ethyl acetate). The organic layer was separated, and the aqueous phase was extracted with ethyl acetate (2x15 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by column chromatography on silica gel using *n*-hexane-ethyl acetate as eluent to give desired products.

^{1}H, ^{13}C NMR and Mass Spectral data

2-Chloro-1-phenylethanol (2a)[1]

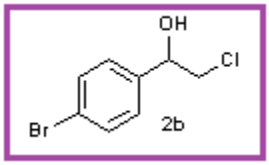


95% yield, colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.40-7.25 (m, 5 H), 4.90-4.76 (m, 1 H), 3.69 (dd, J = 11.33, 3.02 Hz, 1 H), 3.58 (dd, J = 11.33, 9.06 Hz, 1 H), 2.72 (br s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 139.89, 128.52, 128.31, 125.96, 73.92, 50.61.

2-Chloro-1-(4-bromophenyl)ethanol (2b)



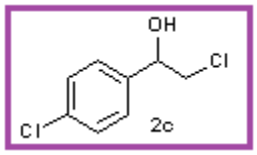
90% yield, white solid, m.p. 59-61 ^{o}C.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.51 (d, J = 8.30 Hz, 2 H), 7.27 (d, J = 8.30 Hz, 2 H) 4.87 (dd, J = 8.30, 2.83 Hz, 1 H), 3.71 (dd, J = 11.14, 3.39 Hz, 1 H), 3.60 (dd, J = 11.33, 8.49 Hz, 1 H), 2.81 (br s, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 138.96, 131.66, 127.71, 122.21, 73.29, 50.36.

HRMS (EI): *m/z* calculated for C_8H_8BrClO $[M]^+$ 235.96036, found: 235.96008.

2-Chloro-1-(4-chlorophenyl)ethanol (2c)[2]

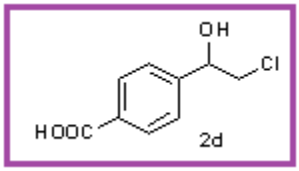


91% yield, colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.41-7.27 (m, 4 H), 4.88 (d, J = 8.49 Hz, 1 H), 3.71 (dd, J = 11.33, 3.58 Hz, 1 H), 3.60 (dd, J = 11.14, 8.68 Hz, 1 H), 2.77 (br s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 138.31, 134.00, 128.66, 127.35, 73.19, 50.32.

2-Chloro-1-(4-carboxyphenyl)ethanol (2d)



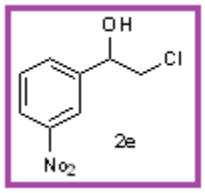
81% yield, colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 8.07 (d, J = 8.30 Hz, 2 H), 7.48 (d, J = 8.30 Hz, 2 H), 4.96 (dd, J = 8.30, 3.77 Hz, 1 H), 3.80-3.61 (m, 2 H), 1.26 (br s, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 168.42, 145.11, 130.48, 130.11, 125.99, 73.50, 50.43.

HRMS (EI): *m/z* calculated for $C_9H_9ClO_3$ $[M]^+$ 200.02402, found: 200.02351.

2-Chloro-1-(3-nitrophenyl)ethanol (2e)



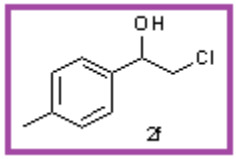
83% yield, colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 8.29 (s, 1 H), 8.18 (d, J = 8.30 Hz, 1 H), 7.75 (d, J = 7.74 Hz, 1 H), 7.57 (t, J = 7.93 Hz, 1 H), 5.05 (t, J = 3.77 Hz, 1 H), 3.86-3.76 (m, 1 H), 3.74-3.62 (m, 1 H), 3.17 (d, J = 2.26 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 148.22, 142.05, 132.16, 129.54, 123.20, 121.13, 72.72, 50.14.

HRMS (EI): *m/z* calculated for $C_8H_8ClNO_3$ $[M]^+$ 201.01927, found: 201.01920.

2-Chloro-1-(4-methylphenyl)ethanol (2f)[2]

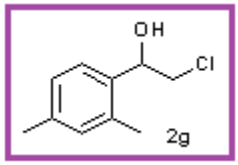


83% yield, colourless liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.27 (d, J = 7.99 Hz, 2 H), 7.18 (d, J = 7.99 Hz, 2 H), 4.86 (dd, J = 8.99, 2.99 Hz, 1 H), 3.72 (dd, J = 10.99, 3.99 Hz, 1 H), 3.64 (dd, J = 10.99, 8.99 Hz, 1 H), 2.62 (br s, 1 H), 2.35 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 138.10, 136.96, 129.21, 125.89, 73.83, 50.66, 21.05.

2-Chloro-1-(2,4-dimethylphenyl)ethanol (2g)



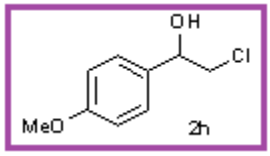
72% yield, colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.39 (d, J = 7.74 Hz, 1 H), 7.05 (d, J = 7.93 Hz, 1 H), 6.98 (s, 1 H), 5.07 (d, J = 8.87 Hz, 1 H), 3.72-3.64 (m, 1 H), 3.62-3.53 (m, 1 H), 2.65 (d, J = 2.45 Hz, 1 H), 2.31 (s, 6 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 137.72, 134.99, 134.53, 131.22, 126.97, 125.40, 70.69, 49.69, 20.89, 18.81.

HRMS (EI): *m/z* calculated for $C_{10}H_{13}ClO$ $[M]^+$ 184.06549, found: 184.06530.

2-Chloro-1-(4-methoxyphenyl)ethanol (2h)[2]

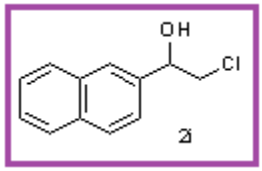


82% yield, colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.31 (d, J = 8.68 Hz, 2 H), 6.90 (d, J = 8.68 Hz, 2 H), 4.85 (d, J = 8.49 Hz, 1 H), 3.81 (s, 3 H), 3.74-3.58 (m, 2 H), 2.65 (d, J = 2.26 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 159.51, 132.09, 127.24, 113.93, 73.58, 55.19, 50.65.

2-Chloro-1-(2-naphthyl)ethanol (2i)[2]

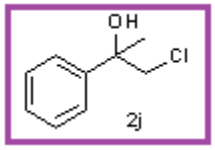


82% yield, white solid, m.p. 65-66 ^{o}C.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.89-7.81 (m, 4 H), 7.53-7.45 (m, 3 H), 5.07 (d, J = 8.69 Hz, 1 H), 3.83 (d, J = 11.29, 3.35 Hz, 1 H), 3.76-3.70 (m, 1 H), 2.80 (d, J = 2.74 Hz, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 137.23, 133.23, 133.13, 128.44, 127.99, 127.67, 126.34, 126.23, 125.21, 123.60, 74.12, 50.70.

1-Chloro-2-phenylpropan-2-ol (2j)[3]

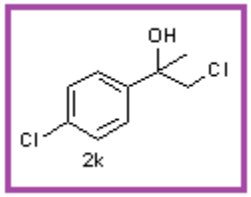


91% yield, colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.50-7.43 (m, 2 H), 7.42-7.33 (m, 2 H), 7.34-7.26 (m, 1 H), 3.84 (d, J = 11.33 Hz, 1 H), 3.76 (d, J = 11.33 Hz, 1 H), 2.63 (br s, 1 H), 1.64 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 144.06, 128.33, 127.44, 124.86, 73.78, 55.26, 27.19.

1-Chloro-2-(4-chlorophenyl)propan-2-ol (2k)



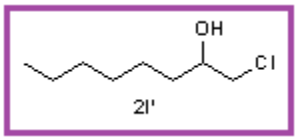
77% yield, light yellow liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.40 (d, J = 8.24 Hz, 2 H), 7.33 (d, J = 8.24 Hz, 2 H), 3.79 (d, J = 10.99 Hz, 1 H), 3.72 (d, J = 10.99 Hz, 1 H), 2.62 (br s, 1 H), 1.61 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 142.63, 133.29, 128.44, 126.46, 73.54, 54.95, 27.20.

HRMS (EI): *m/z* calculated for $C_9H_{10}Cl_2O$ $[M]^+$ 204.01087, found: 204.01070.

1-Chloro-2-octanol (2l')[4]

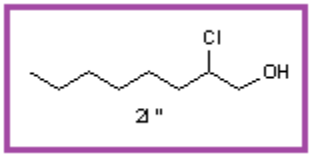


75% yield, colourless liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 3.84-3.76 (m, 1 H), 3.64 (dd, J = 10.98, 3.2 Hz, 1 H), 3.48 (dd, J = 10.98, 7.01 Hz, 1 H), 2.20 (br s, 1 H), 1.59-1.39 (m, 3 H), 1.40-1.23 (m, 7 H), 0.92-0.84 (m, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 71.38, 50.43, 34.15, 31.63, 29.10, 25.41, 22.50, 13.97.

2-Chloro-1-octanol (2l")[4]

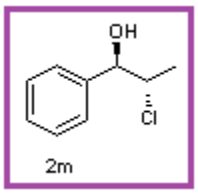


20% yield, colourless liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 4.06-3.99 (m, 1 H), 3.83-3.75 (m, 1 H), 3.71-3.63 (m, 1 H), 2.09-2.03 (m, 1 H), 1.81-1.67 (m, 2 H), 1.58-1.48 (m, 1 H), 1.47-1.23 (m, 7 H), 0.93-0.86 (m, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 66.97, 65.34, 34.22, 31.56, 28.72, 26.24, 22.49, 13.98.

1-Phenyl-2-chloro-1-propanol (**2m**)[5]

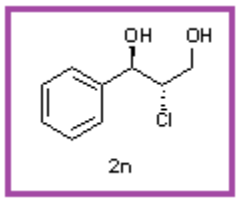


Major diastereomer (anti): 61% yield, colourless liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.54-7.27 (m, 5 H), 4.94 (t, J = 3.02 Hz, 1 H), 4.39-4.25 (m, 1 H), 2.48 (d, J = 3.02 Hz, 1 H), 1.38 (d, J = 6.79 Hz, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 139.55, 128.29, 127.97, 126.34, 77.09, 62.66, 18.03.

2-Chloro-3-phenyl-1,3-propandiol (**2n**)



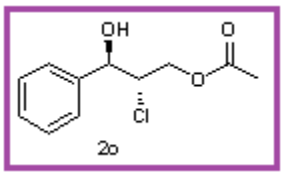
Major diastereomer (anti): 74% yield, light yellow liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.42-7.30 (m, 5 H), 4.97 (d, J = 5.93 Hz, 1 H), 4.24-4.14 (m, 1 H), 3.96 (dd, J = 12.85, 3.95 Hz, 1 H), 3.87 (dd, J = 11.86, 3.95 Hz, 1 H), 2.37 (br s, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 140.10, 128.29, 128.14, 126.55, 76.04, 65.19, 63.74.

HRMS (EI): *m/z* calculated for $C_9H_{11}ClO_2$ $[M]^+$ 186.04476, found: 186.04396.

2-Chloro-3-hydroxy-3-phenylpropyl acetate (**2o**)



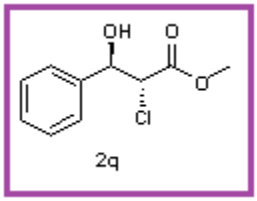
Major diastereomer (anti): 60.5 % yield, colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.47-7.28 (m, 5 H), 4.93 (d, J = 5.28 Hz, 1 H), 4.49-4.38 (m, 1 H), 4.37-4.24 (m, 2 H), 2.85 (br s, 1 H), 2.04 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 170.91, 139.26, 128.35, 128.23, 126.40, 74.62, 64.09, 62.57, 20.53.

HRMS (EI): *m/z* calculated for $C_{11}H_{13}ClO_3$ $[M]^+$ 228.05532, found: 228.05519.

Methyl 2-chloro-3-hydroxy-3-phenylpropanoate (2q)[1]

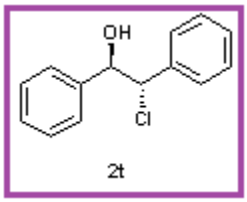


Major diastereomer (anti): 21% yield, light yellow liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.45-7.31 (m, 5 H), 5.03 (dd, J = 7.78, 3.05 Hz, 1 H), 4.38 (d, J = 7.93 Hz, 1 H), 3.79 (s, 3 H), 3.14 (br s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 169.31, 138.62, 128.75, 128.47, 126.87, 75.25, 58.91, 53.06.

2-Chloro-1,2-diphenylethanol (2t)[1]

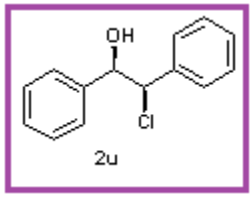


Major diastereomer (anti): 37% yield, colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.36-7.25 (m, 10 H), 5.08 (d, J = 6.79 Hz, 1 H), 5.00 (d, J = 6.79 Hz, 1 H), 2.35 (br s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 139.44, 137.19, 128.65, 128.40, 128.30, 128.10, 127.03, 78.10, 66.83.

2-Chloro-1,2-diphenylethanol (2u)[1]

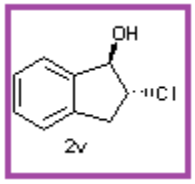


Major diastereomer (syn): 32% yield, colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.27-7.14 (m, 8 H), 7.13-7.06 (m, 2 H), 5.01 (d, J = 8.30 Hz, 1 H), 4.94 (d, J = 8.30 Hz, 1 H), 3.08 (br s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 138.51, 137.68, 128.51, 128.29, 128.12, 127.94, 126.94, 78.74, 70.64.

2-Chloroindan-1-ol (2v)[4]

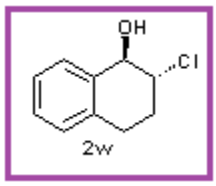


Major diastereomer (anti): 62.33% yield, colourless liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.44-7.37 (m, 1 H), 7.33-7.27 (m, 2 H), 7.24-7.17 (m, 1 H), 5.20 (d, J = 5.00 Hz, 1 H), 4.29 (dd, J = 13.01, 7.00 Hz, 1 H), 3.51 (dd, J = 16.01, 7.00 Hz, 1 H), 3.09 (dd, J = 16.01, 7.00 Hz, 1 H), 2.35 (br s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 141.32, 138.95, 128.96, 127.57, 124.63, 124.10, 82.95, 64.56, 39.75.

2-Chloro-1,2,3,4-tetrahydro-1-naphthol (2w)[6]

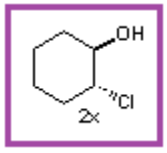


Major diastereomer (anti): 68% yield, white solid, m.p. 91-93 ^{o}C.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.57-7.48 (m, 1 H), 7.30-7.19 (m, 2 H), 7.16-7.07 (m, 1 H), 4.78 (d, J = 6.79 Hz, 1 H), 4.27-4.16 (m, 1 H), 3.06-2.83 (m, 1 H), 2.60 (br s, 1 H), 2.48-2.35 (m, 1 H), 2.24-2.08 (m, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 135.44, 134.95, 128.37, 128.17, 127.88, 126.54, 73.83, 62.75, 29.01, 27.18.

2-Chloro cyclohexanol (2x)[7]

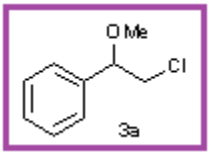


Major diastereomer (anti): 90% yield, colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 3.81-3.67 (m, 1 H), 3.61-3.46 (m, 1 H), 2.67 (br s, 1 H), 2.31-2.03 (m, 2 H), 1.88-1.57 (m, 3 H), 1.44-1.21 (m, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 75.32, 67.49, 35.11, 33.67, 25.63, 23.93.

1-(2-Chloro-1-methoxyethyl)benzene (3a)[8]

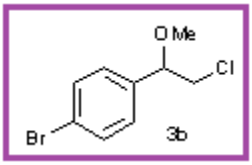


90% yield, colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.43-7.30 (m, 5 H), 4.41-4.33 (m, 1 H), 3.68 (dd, J = 11.33, 8.30 Hz, 1 H), 3.59 (dd, J = 11.33, 4.53 Hz, 1 H), 3.32 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 138.50, 128.57, 128.45, 126.78, 83.62, 57.14, 48.07.

1-Bromo-4-(2-chloro-1-methoxyethyl)benzene (3b)



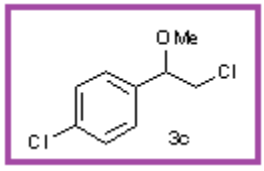
91% yield, colourless liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.52 (d, J = 8.39 Hz, 2 H), 7.22 (d, J = 8.39 Hz, 2 H), 4.35-4.30 (m, 1 H), 3.65 (dd, J = 11.44, 7.47 Hz, 1 H), 3.55 (dd, J = 11.44, 4.73 Hz, 1 H), 3.31 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 137.55, 131.77, 128.56, 122.42, 82.93, 57.27, 47.68.

HRMS (EI): *m/z* calculated for $C_9H_{10}BrClO$ $[M]^+$ 249.97601, found: 249.97589.

1-Chloro-4-(2-chloro-1-methoxyethyl)benzene (3c)[8]

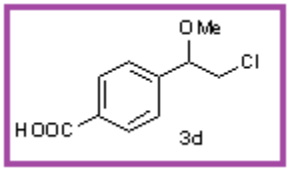


91% yield, colourless liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.36 (d, J = 8.83 Hz, 2 H), 7.27 (d, J = 8.83 Hz, 2 H), 4.37-4.31 (m, 1 H), 3.65 (dd, J = 11.04, 7.72 Hz, 1 H), 3.55 (dd, J =11.04, 4.41 Hz, 1 H), 3.31 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 137.04, 134.22, 128.79, 128.21, 82.86, 57.21, 47.73.

4-(2-Chloro-1-methoxyethyl)benzoic acid (3d)



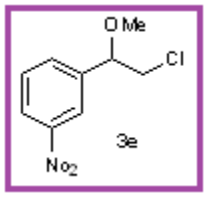
83% yield, white solid, m.p. 98-100 °C.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 8.15 (d, J = 8.39 Hz, 2 H), 7.47 (d, J = 8.24 Hz, 2 H), 4.48-4.43 (m, 1 H), 3.73-3.67 (m, 1 H), 3.65-3.60 (m, 1 H), 3.36 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 171.90, 144.70, 130.56, 127.68, 127.04, 83.17, 57.48, 47.52.

HRMS (EI): *m/z* calculated for $C_{10}H_{12}ClO_3$ $[M + H]^+$ 215.04583, found: 215.04576.

1-(2-Chloro-1-methoxyethyl)-3-nitrobenzene (3e)



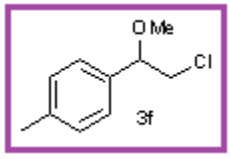
88% yield, white solid, m.p. 54-56 °C.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 8.25-8.19 (m, 2 H), 7.71 (d, J = 7.62 Hz, 1 H), 7.59 (t, J = 7.78 Hz, 1 H), 4.52-4.47 (m, 1 H), 3.75-3.69 (dd, J = 11.59, 6.71 Hz, 1 H), 3.66-3.61 (dd, J = 11.59, 5.18 Hz, 1 H), 3.37 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 148.39, 140.89, 132.98, 129.60, 123.46, 121.92, 82.41, 57.60, 47.24.

HRMS (EI): *m/z* calculated for $C_9H_{11}ClNO_3$ $[M + H]^+$ 216.02715, found: 216.02709.

1-(2-Chloro-1-methoxyethyl)-4-methylbenzene (3f)[8]



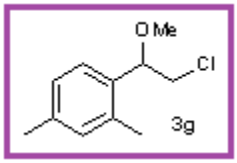
83% yield, colourless liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.24-7.16 (m, 4 H), 4.32 (dd, J = 7.89, 4.30 Hz, 1 H), 3.69-3.63 (m,1 H), 3.56 (dd, J = 11.48, 4.30 Hz, 1 H), 3.30 (s, 3 H), 2.36 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 138.20, 135.45, 129.25, 126.71, 83.44, 56.97, 48.09,

21.08.

1-(2-Chloro-1-methoxyethyl)-2,4-dimethylbenzene (3g)



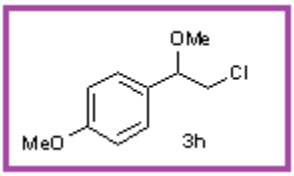
64% yield, colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.27 (d, J = 7.55 Hz, 1 H), 7.05 (d, J = 7.55 Hz, 1 H), 6.99 (s, 1 H), 4.60 (dd, J = 8.30, 3.77 Hz, 1 H), 3.68-3.49 (m, 2 H), 3.29 (s, 3 H), 2.32 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 137.72, 135.56, 133.41, 131.38, 127.05, 126.01, 80.32, 56.93, 47.37, 20.91, 18.88.

HRMS (EI): *m/z* calculated for $C_{11}H_{15}ClO$ $[M]^+$ 198.08114, found: 198.08115.

1-(2-Chloro-1-methoxyethyl)-4-methoxybenzene (3h)



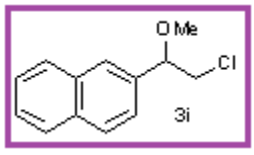
84% yield, light yellow liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.25 (d, J = 9.06 Hz, 2 H), 6.91 (d, J = 8.30 Hz, 2 H), 4.36-4.26 (m, 1 H), 3.81 (s, 3 H), 3.66 (dd, J = 11.33, 8.30 Hz, 1 H), 3.55 (dd, J = 11.33, 4.53 Hz, 1 H), 3.28 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 159.63, 130.40, 127.99, 113.92, 83.09, 56.86, 55.14, 48.10.

HRMS (EI): *m/z* calculated for $C_{10}H_{13}ClO_2$ $[M]^+$ 200.06041, found: 200.06084.

2-(2-Chloro-1-methoxyethyl)naphthalene (3i)



90% yield, yellow liquid.

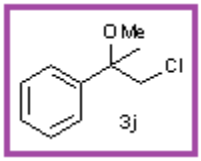
^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.92-7.82 (m, 3 H), 7.80 (s, 1 H), 7.55-7.47 (m, 2 H), 7.44 (dd, J = 8.49, 1.32 Hz, 1 H), 4.57-4.49 (m, 1 H), 3.82-3.62 (m, 2 H), 3.36 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 135.88, 133.37, 133.08, 128.58, 127.88, 127.70, 126.45,

126.33, 126.25, 124.01, 83.76, 57.24, 47.97.

HRMS (EI): *m/z* calculated for $C_{13}H_{13}ClO$ $[M]^+$ 220.06549, found: 220.06502.

(2-Chloro-1-methoxy-1-methylethyl)-benzene (3j)[8]

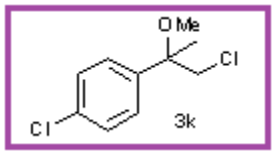


68% yield, colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.45-7.27 (m, 5 H), 3.72 (d, J = 11.33 Hz, 1 H), 3.59 (d, J = 11.33 Hz, 1 H), 3.14 (s, 3 H), 1.69 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 141.69, 128.41, 127.80, 126.44, 78.61, 53.39, 50.89, 20.65.

1-Chloro-4-(2-chloro-1-methoxy-1-methylethyl)benzene (3k)



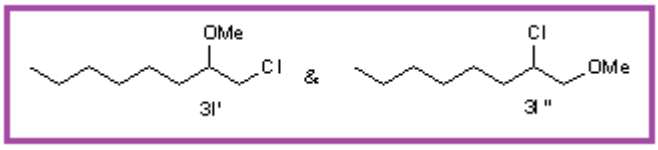
83% yield, colourless liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.41-7.30 (m, 4 H), 3.66 (d, J = 11.04 Hz, 1 H), 3.56 (d, J = 12.14 Hz, 1 H), 3.14 (s, 3 H), 1.66 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 140.28, 133.69, 128.53, 127.94, 78.32, 52.96, 50.85, 20.60.

HRMS (EI): *m/z* calculated for $C_{10}H_{11}Cl_2O$ $[M - H]^+$ 217.01870, found: 217.01861.

1-Chloro-2-methoxyoctane (3l') & 2-chloro-1-methoxyoctane (3l")



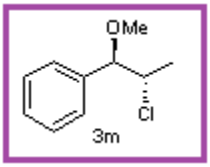
82% yield (obtained as a mixture of regioisomers **3l'** and **3l"**), colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 4.04-3.93 (m, 1 H), 3.61-3.48 (m, 5 H), 3.42 (s, 3 H), 3.40 (s, 3 H), 3.39-3.31 (m, 1 H), 1.90-1.20 (m, 20 H), 0.96-0.82 (m, 6 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 80.67, 76.65, 60.54, 58.99, 57.39, 45.55, 34.70, 31.91, 31.68, 31.59, 29.23, 28.71, 26.03, 25.04, 22.51, 13.99.

HRMS (EI): *m/z* calculated for $C_9H_{19}ClO$ $[M]^+$ 178.11244, found: 178.11234.

2-Chloro-1-methoxy-1-phenylpropane (3m)



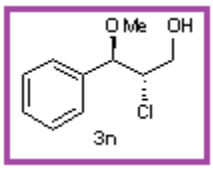
Major diastereomer (anti): 67% yield, light yellow liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.41-7.29 (m, 5 H), 4.26 (d, J = 5.28 Hz, 1 H), 4.21-4.09 (m, 1 H), 3.31 (s, 3 H), 1.48 (d, J = 6.79 Hz, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 138.07, 128.31, 128.13, 127.47, 87.16, 60.08, 57.42, 19.80.

HRMS (EI): *m/z* calculated for $C_{10}H_{14}ClO$ $[M + H]^+$ 185.07332, found: 185.07332.

2-Chloro-3-methoxy-3-phenylpropan-1-ol (3n)



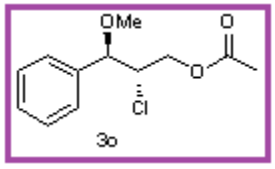
Major diastereomer (anti): 59% yield, colourless liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.42-7.32 (m, 5 H), 4.39 (d, J = 6.99 Hz), 4.12-4.06 (m, 1 H), 3.99-3.88 (m, 2 H), 2.55 (br s, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 137.71, 128.50, 128.40, 127.54, 85.52, 64.55, 64.28, 57.35.

HRMS (EI): *m/z* calculated for $C_{10}H_{13}ClO_2$ $[M]^+$ 200.06041, found: 200.06085.

2-Chloro-3-methoxy-3-phenylpropyl acetate (3o)



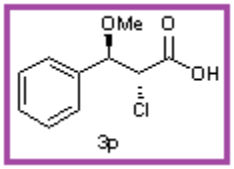
Major diastereomer (anti): 55% yield, colourless liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.42-7.32 (m, 5 H), 4.46-4.41 (m, 1 H), 4.40-4.30 (m, 2 H), 4.25-4.20 (m, 1 H), 3.27 (s, 3 H), 2.08 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 170.50, 137.22, 128.46, 128.32, 127.53, 83.81, 64.61, 61.09, 57.36, 20.53.

HRMS (EI): *m/z* calculated for $C_{11}H_{12}ClO_2$ $[M - OMe]^+$ 211.05258, found: 211.05250.

2-Chloro-3-methoxy-3-phenylpropanoic acid (3p)



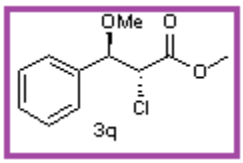
Major diastereomer (anti): 51% yield, white solid, m.p. 162-163 °C.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.44-7.37 (m, 5 H), 4.53 (d, J = 8.85 Hz, 1 H), 4.31 (d, J = 8.69 Hz, 1 H), 3.29 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 169.36, 136.06, 128.06, 127.65, 127.38, 83.42, 58.57, 56.63.

HRMS (EI): *m/z* calculated for $C_9H_8ClO_2$ $[M - OMe]^+$ 183.02128, found: 183.02125.

Methyl 2-chloro-3-methoxy-3-phenylpropanoate (3q)

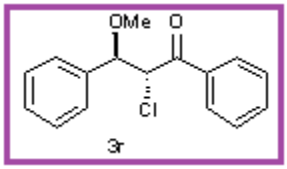


Major diastereomer (anti): 74% yield, colourless liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.41-7.36 (m, 5 H), 4.51 (d, J = 9.30 Hz, 1 H), 4.27 (d, J = 9.15 Hz, 1 H), 3.84 (s, 3 H), 3.23 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 169.11, 136.38, 128.87, 128.38, 127.91, 84.13, 61.41, 58.30, 52.94.

2-Chloro-3-methoxy-1,3-diphenylpropan-1-one (3r)[9]

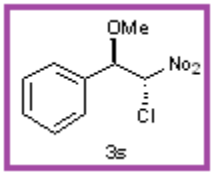


Major diastereomer (anti): 60% yield, white solid, m.p. 61-63 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 8.09-8.01 (m, 2 H), 7.66-7.57 (m, 1 H), 7.56-7.28 (m, 7H), 5.09 (d, J = 9.06 Hz, 1 H), 4.76 (d, J = 9.06 Hz, 1 H), 3.18 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 193.37, 137.34, 133.73, 128.84, 128.73, 128.35, 128.09, 83.55, 57.36, 56.81.

(2-Chloro-1-methoxy-2-nitroethyl)benzene (3s)



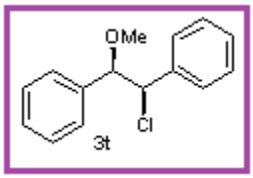
Major diastereomer (anti): 42% yield, colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.58-7.33 (m, 5 H), 5.76 (d, J = 9.06 Hz, 1 H), 4.70 (d, J = 9.06 Hz, 1 H), 3.25 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 133.47, 129.81, 128.87, 128.17, 91.28, 84.52, 57.65.

HRMS (EI): *m/z* calculated for $C_9H_9ClNO_3$ $[M - H]^+$ 214.02710, found: 214.02700.

1-Methoxy-1,2-diphenyl-2-chloroethane (3t)[8]

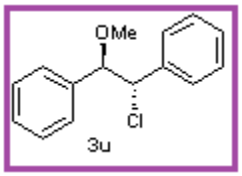


Major diastereomer (anti): 54% yield, white solid, m.p. 69-70 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.24-7.01 (m, 10 H), 4.95 (d, J = 8.30 Hz, 1 H), 4.46 (d, J = 7.55 Hz, 1 H), 3.32 (s, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 137.51, 128.09, 127.65, 128.03, 87.74, 66.82, 57.28.

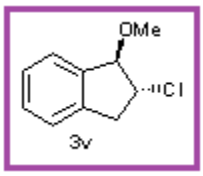
1-Methoxy-1,2-diphenyl-2-chloroethane (3u)[8]



Major diastereomer (anti): 50% yield, white solid, m.p. 83-84 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.38-7.28 (m, 8 H), 7.24-7.18 (m, 2 H), 4.98 (d, J = 6.61 Hz, 1 H), 4.53 (d, J = 6.61 Hz, 1 H), 3.19 (s, 3 H).

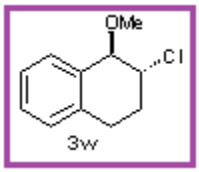
2-Chloro-1-methoxy-2,3-dihydro-1*H*-indene (3v)[8]



Major diastereomer (anti): 63% yield, yellow liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.39 (d, J = 7.17 Hz, 1 H), 7.36-7.31 (m, 2 H), 7.23 (d, J = 7.17 Hz, 1 H), 4.85 (d, J = 3.81 Hz, 1 H), 4.49-4.44 (m, 1 H), 3.64-3.54 (m, 4 H), 3.10 (dd, J = 16.47, 5.01 Hz, 1 H).

2-Chloro-1-methoxy-1,2,3,4-tetrahydronaphthalene (3w)



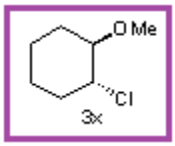
Major diastereomer (anti): 64% yield, colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.36-7.29 (m, 1 H), 7.28-7.17 (m, 2 H), 7.16-7.10 (m, 1 H), 4.53-4.46 (m, 1 H), 4.35 (d, J = 4.53 Hz, 1 H), 3.53 (s, 3 H), 3.11-2.95 (m, 1 H), 2.88-2.74 (m, 1 H), 2.50-2.36 (m, 1 H), 2.18-2.03 (m, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 135.66, 133.01, 130.03, 128.69, 128.13, 126.07, 81.32, 57.53, 56.99, 26.84, 24.93.

HRMS (EI): *m/z* calculated for $C_{11}H_{13}ClO$ $[M]^+$ 196.06549, found: 196.06539.

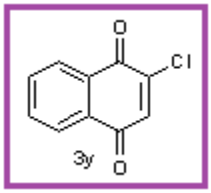
1-Chloro-2-methoxycyclohexane (3x)[8]



Major diastereomer (anti): 62.5% yield, colourless liquid.

^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 3.90-3.77 (m, 1 H), 3.45 (s, 3 H), 3.24-3.10 (m, 1 H), 2.28-2.07 (m, 2 H), 1.83-1.58 (m, 3 H), 1.42-1.21 (m, 3 H).

2-Chloro-1,4-naphthoquinone (3y)[10]

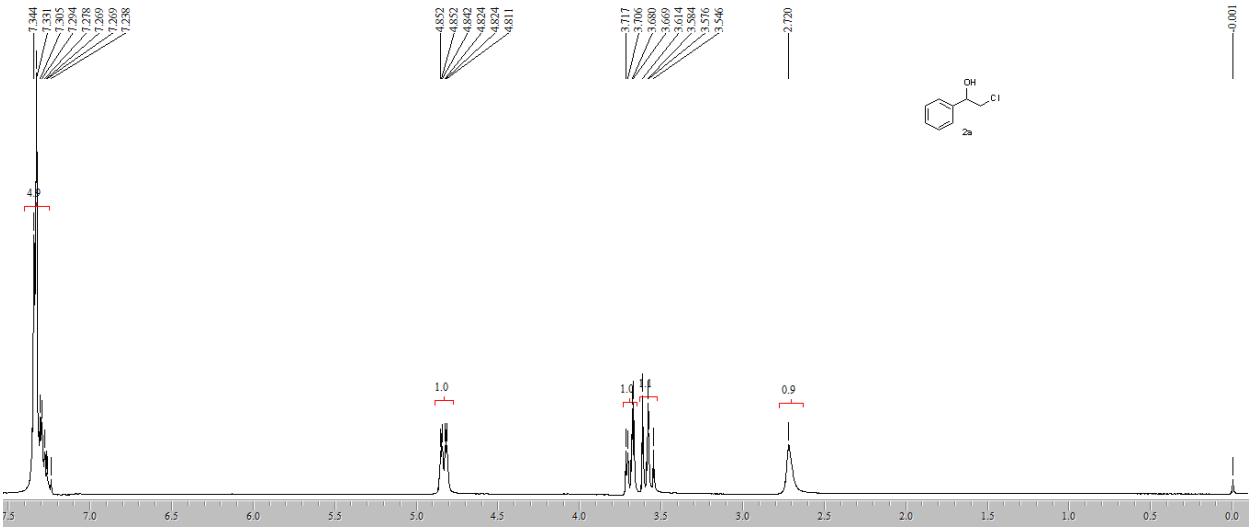


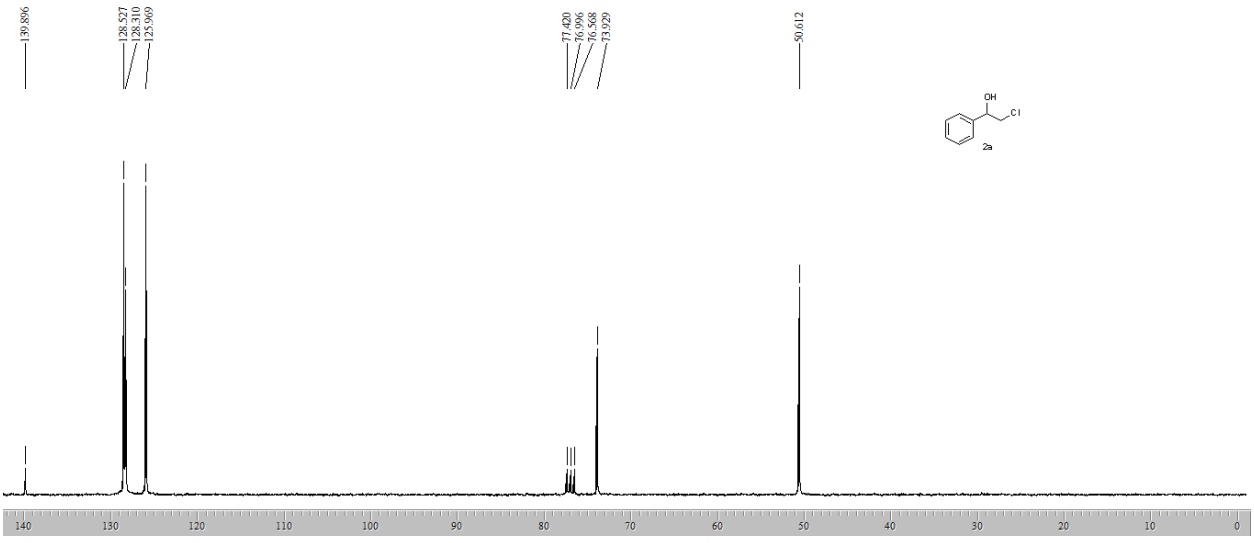
57% yield, yellow solid, m.p. 108-110 ^{o}C.

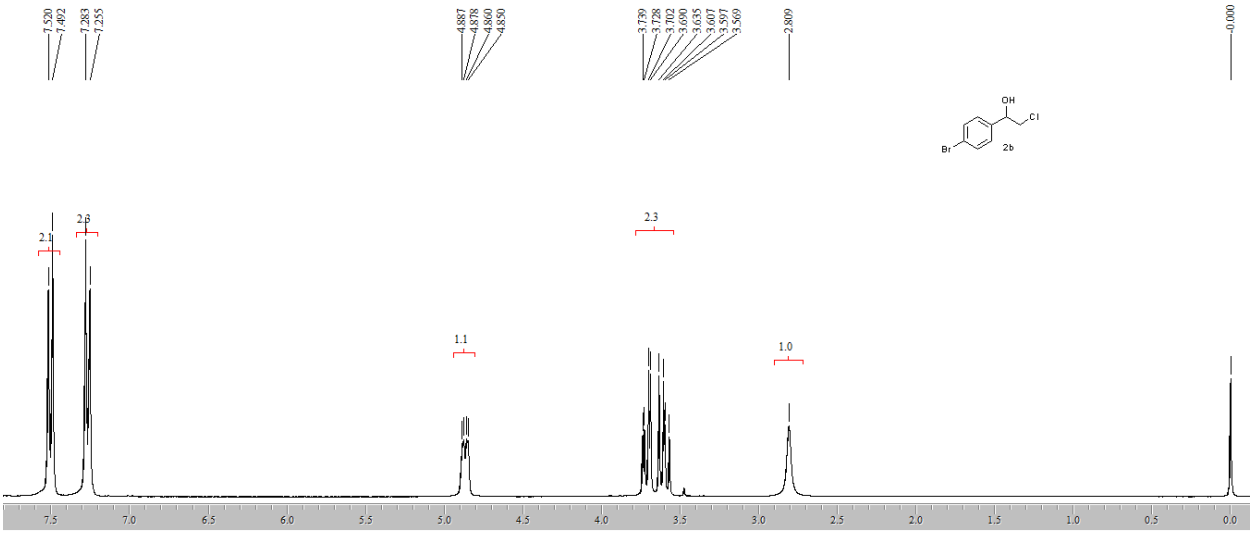
^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 8.23-8.15 (m, 1 H), 8.14-8.07 (m, 1 H), 7.85-7.75 (m, 2 H), 7.24 (s, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) = 182.61, 177.89, 146.27, 135.86, 134.46, 134.11, 131.69, 131.23, 127.47, 126.71.

Copies of ^{1}H and ^{13}C NMR Spectra



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7.492
7.283
7.255
4.887
4.878
4.860
4.850
3.739
3.728
3.702
3.690
3.635
3.607
3.597
3.569
2.809
0.000
OH
Cl
Br
2b
2.1
2.0
1.1
2.3
1.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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3.0
2.5
2.0
1.5
1.0
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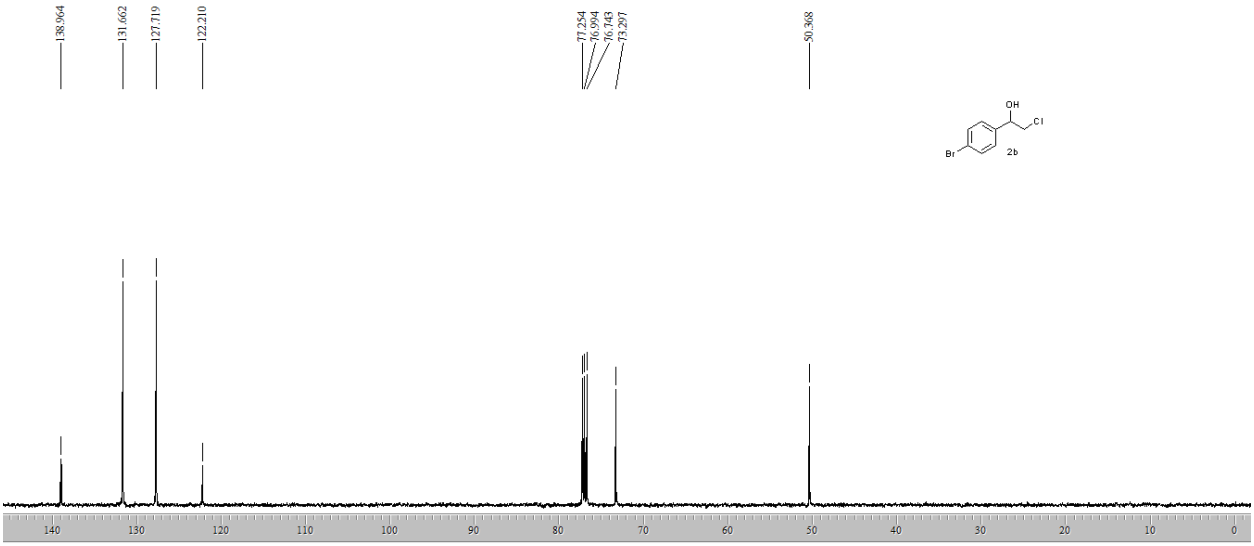
0.0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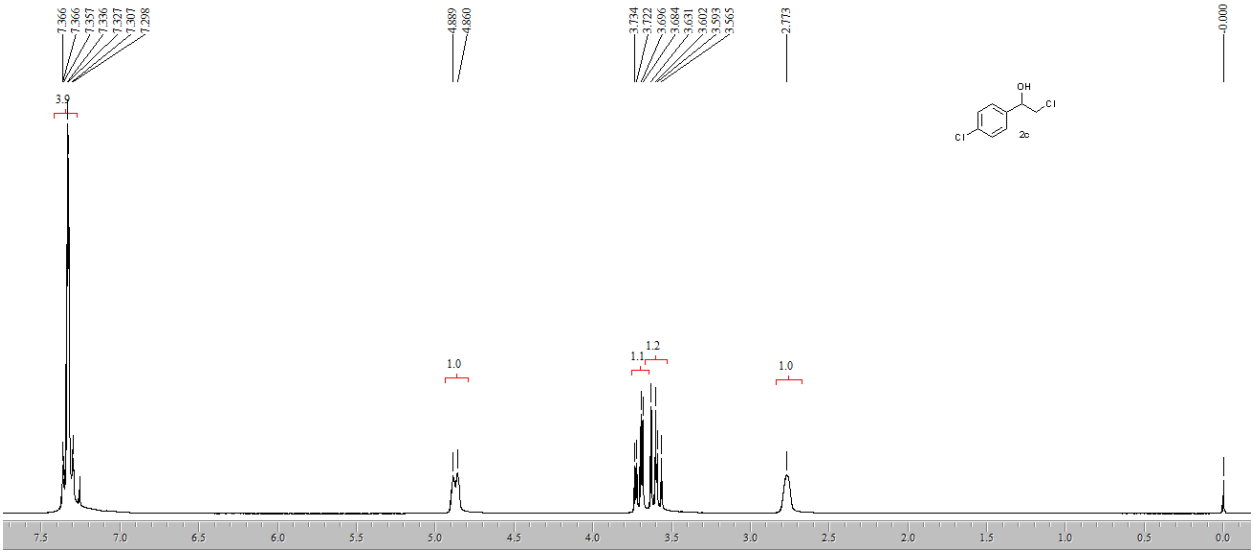
131.662
127.719
122.210
77.254
76.994
76.743
73.297
50.368
OH
Cl
Br
2b
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0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7.366
7.357
7.336
7.327
7.307
7.298
4.889
4.860
3.734
3.722
3.696
3.684
3.631
3.602
3.593
3.565
2.773
0.000
3.9
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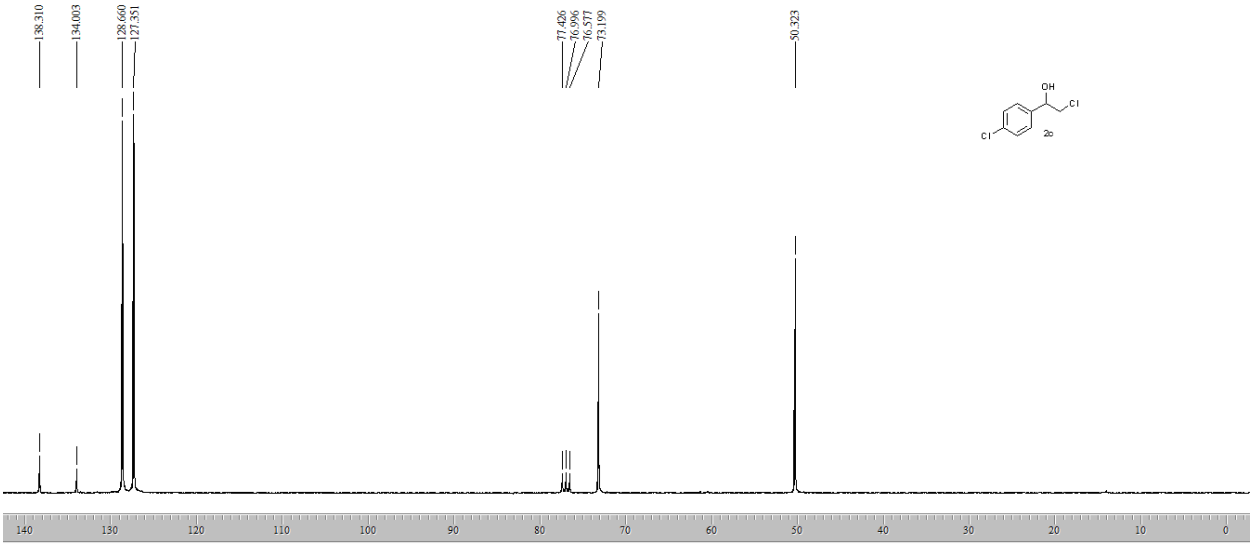
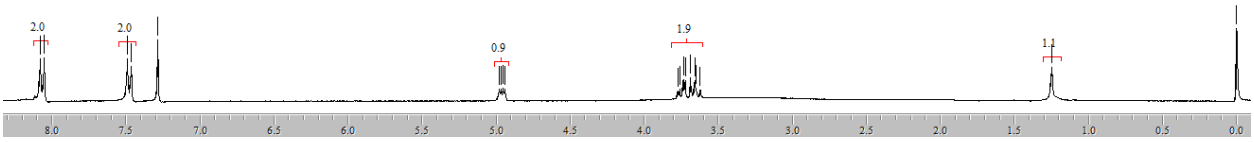
1.1
1.2
1.0
OH
Cl
Cl
2o


138.310
134.003
128.660
127.351
77.426
76.996
76.577
73.199
50.323
OH
Cl
Cl
2o


8.082
8.054
7.492
7.464
7.286
4.980
4.967
4.955
4.942
3.775
3.762
3.736
3.724
3.687
3.659
3.649
3.623
1.250
0.001
OH
Cl
HOOC
2d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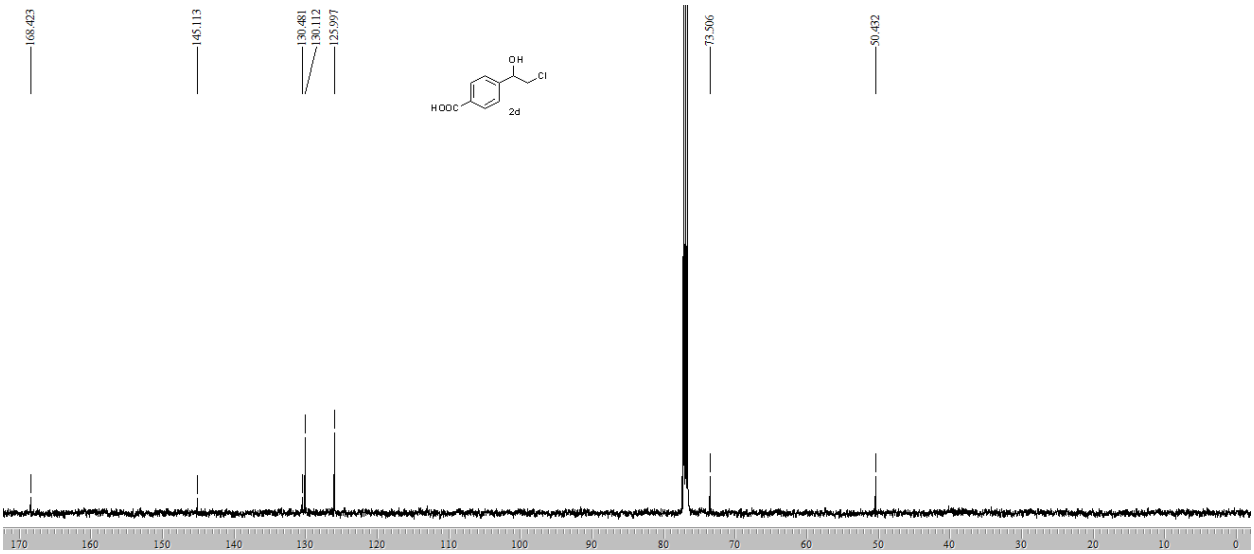
2.0
0.9
1.9
1.1
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0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145.113
130.481
130.112
125.997
73.506
50.432
OH
Cl
HOOC
2d
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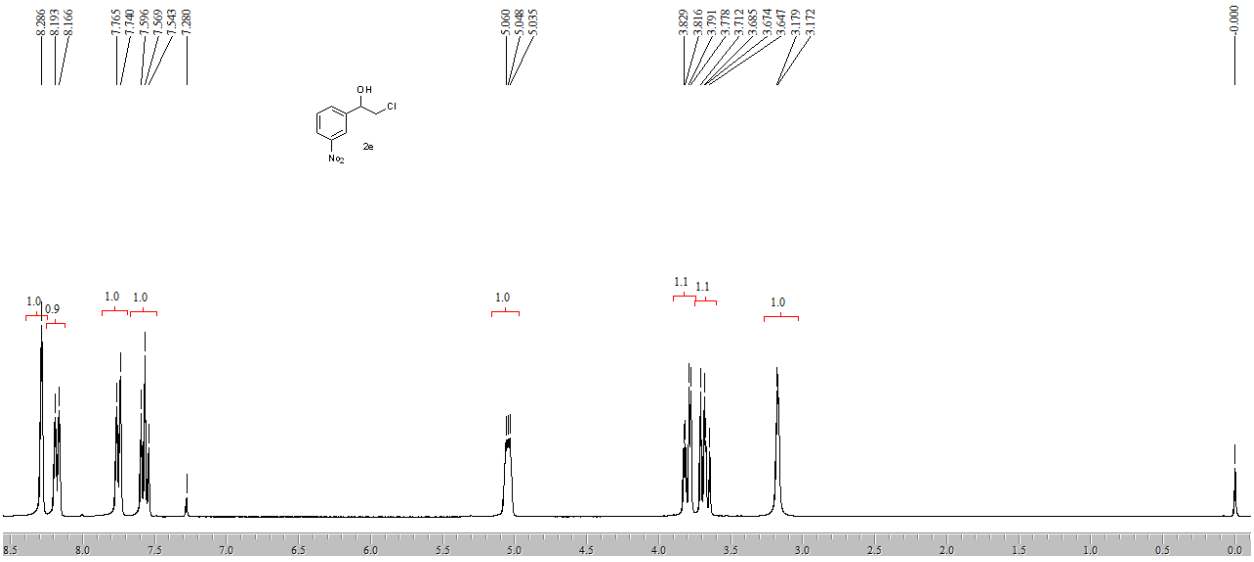
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0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8.193
8.166
7.765
7.740
7.596
7.569
7.543
7.280
5.060
5.048
5.035
3.829
3.816
3.791
3.778
3.712
3.685
3.674
3.647
3.179
3.172
0.000
OH
Cl
2e
NO2
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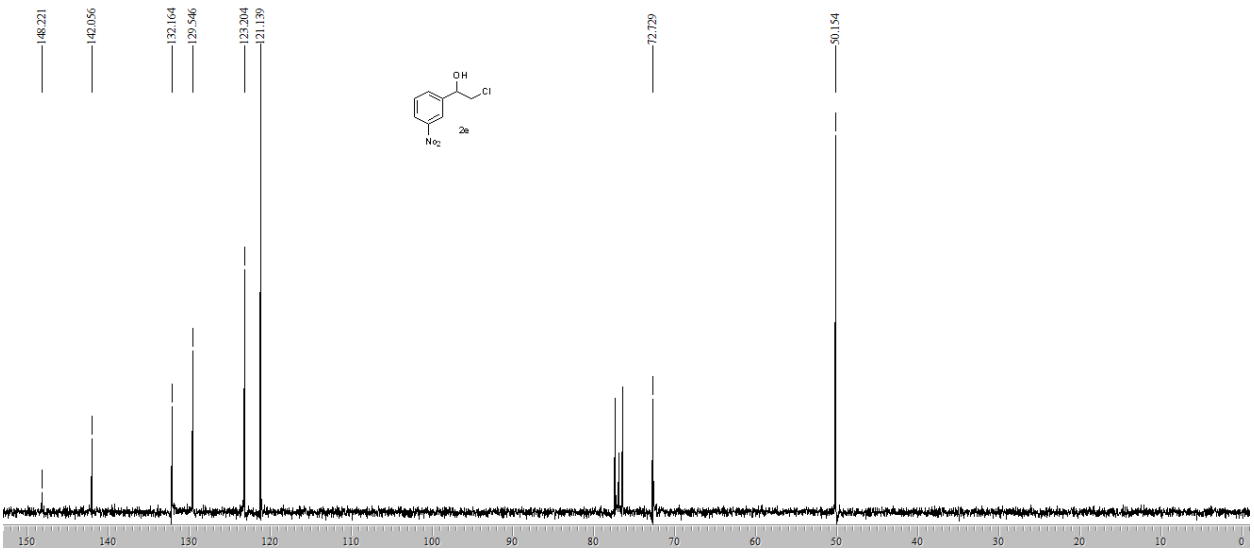
0.9
1.0
1.0
1.0
1.1
1.1
1.0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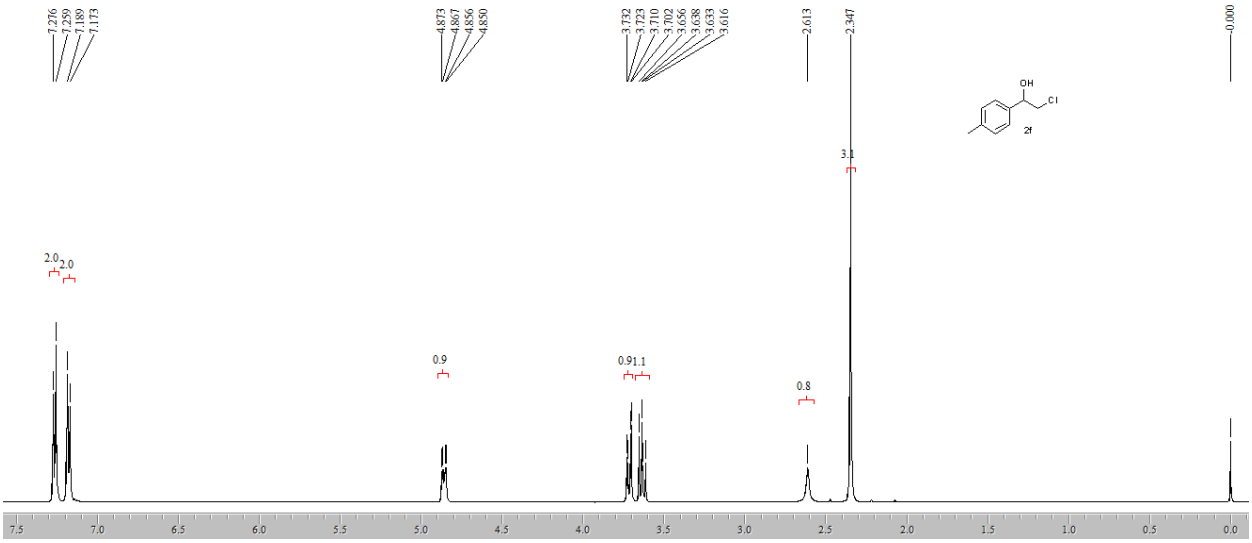
132.164
129.546
123.204
121.139
72.729
50.154
OH
Cl
2e
NO2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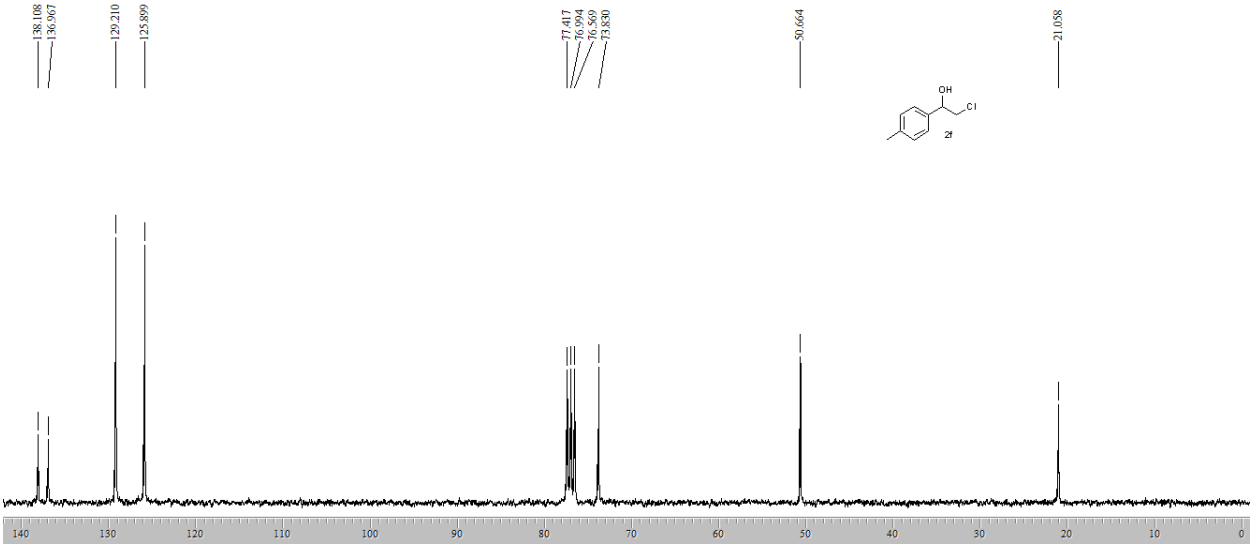
7.259
7.189
7.173
4.873
4.867
4.856
4.850
3.732
3.723
3.710
3.702
3.656
3.638
3.633
3.616
2.613
2.347
0.000
OH
Cl
2f
3.1
2.0
2.0
0.9
0.9
1.1
0.8
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0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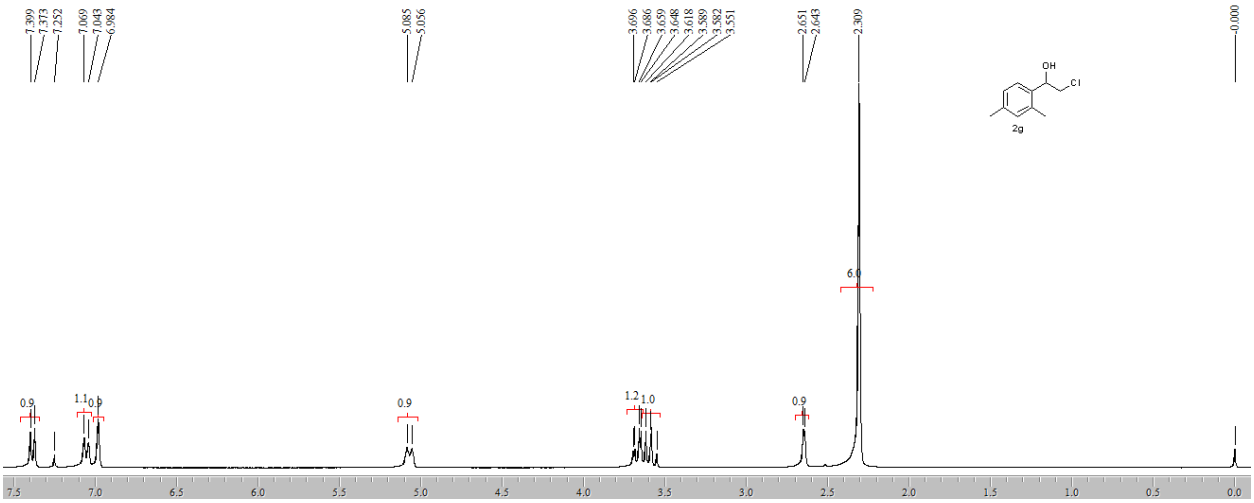
125.899
77.417
76.994
76.569
73.830
50.664
21.058
OH
Cl
2f
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0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7.252
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6.984
5.085
5.056
3.696
3.686
3.659
3.648
3.618
3.589
3.582
3.551
2.651
2.643
2.309
0.000
OH
Cl
2g
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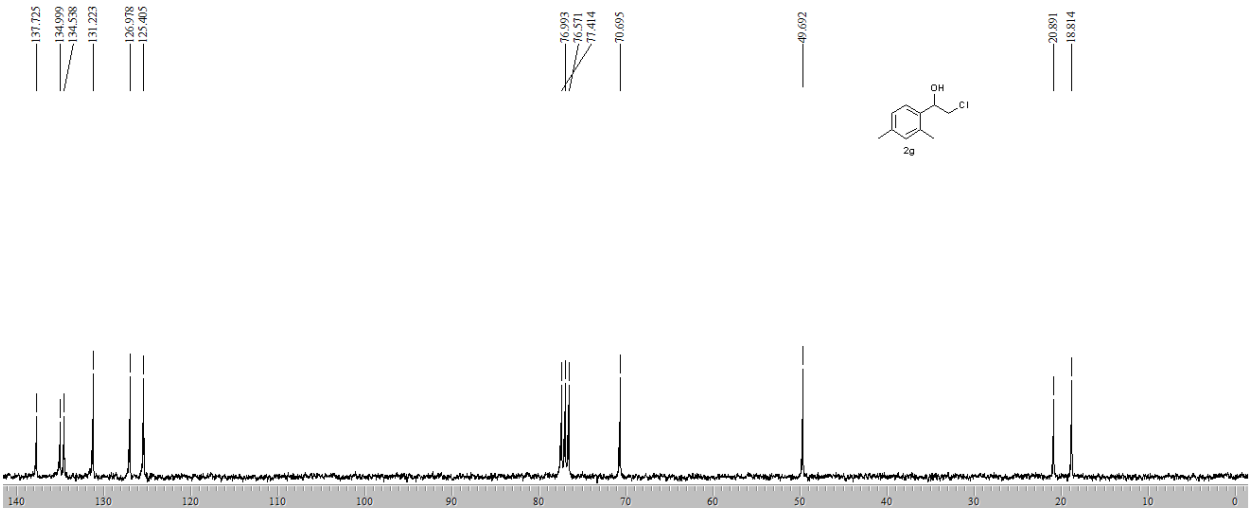
0.9
1.2
1.0
0.9
6.0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125.405
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49.692
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18.814
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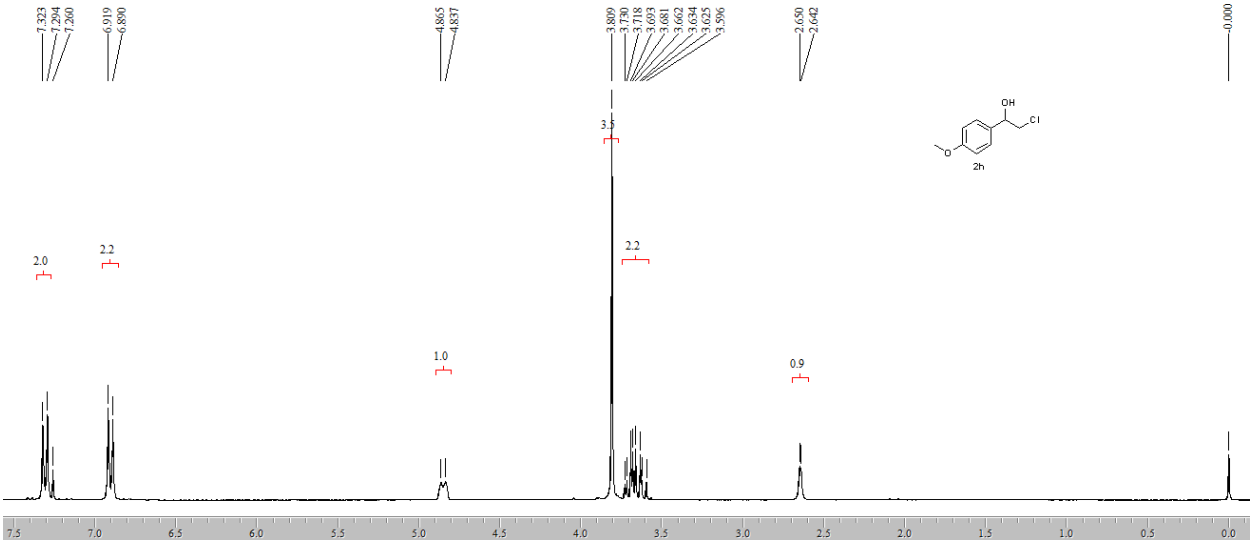
Cl
2g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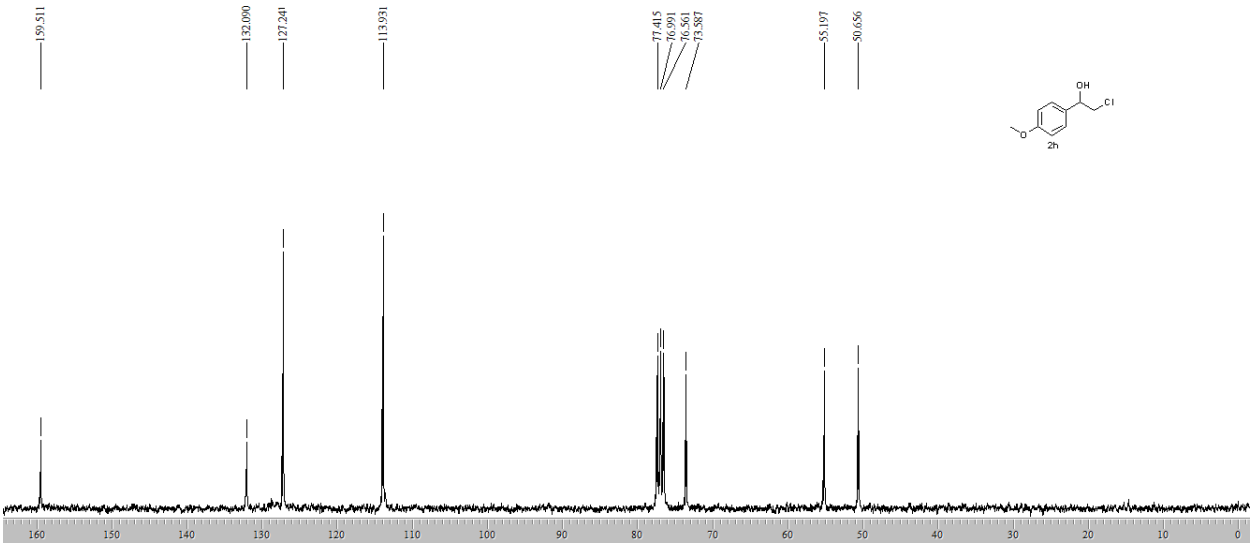
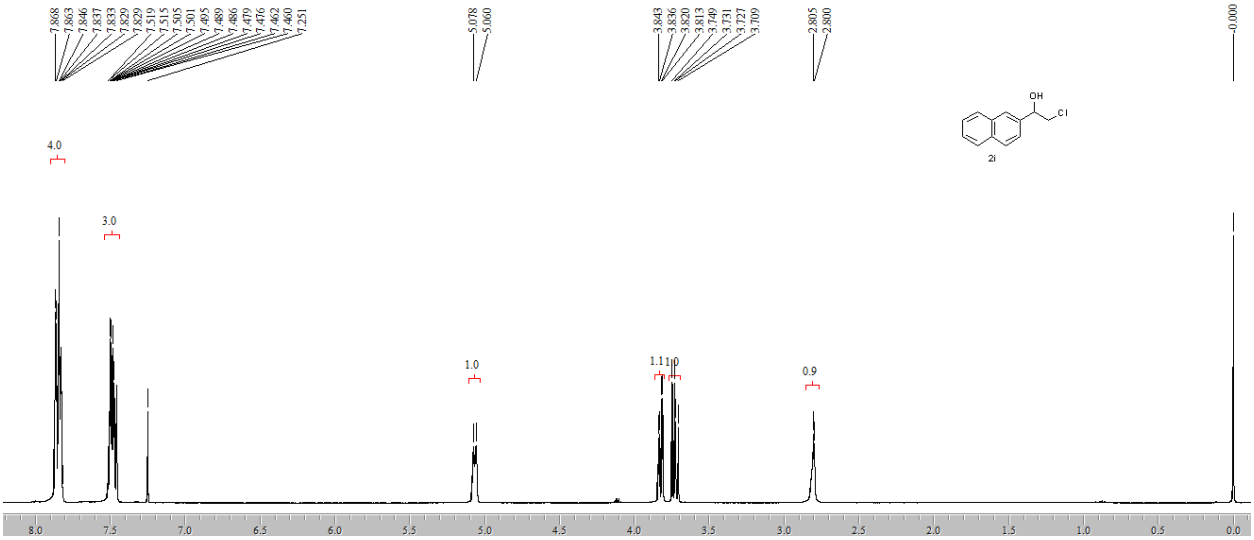
6.919
6.890
4.865
4.837
3.809
3.790
3.718
3.693
3.681
3.662
3.654
3.625
3.596
2.650
2.642
0.000
OH
Cl
O
2h
2.0
2.2
3.5
2.2
1.0
0.9
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


159.511
132.090
127.241
113.931
77.415
76.991
76.561
73.587
55.197
50.656
OH
Cl
O
2h
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0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7.833
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7.515
7.505
7.501
7.495
7.489
7.486
7.479
7.476
7.462
7.460
7.251
5.078
5.060
3.843
3.836
3.820
3.813
3.749
3.731
3.727
3.709
2.805
2.800
0.000
OH
Cl
2i
4.0
3.0
1.0
1.1
1.0
0.9
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
4.0
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3.0
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2.0
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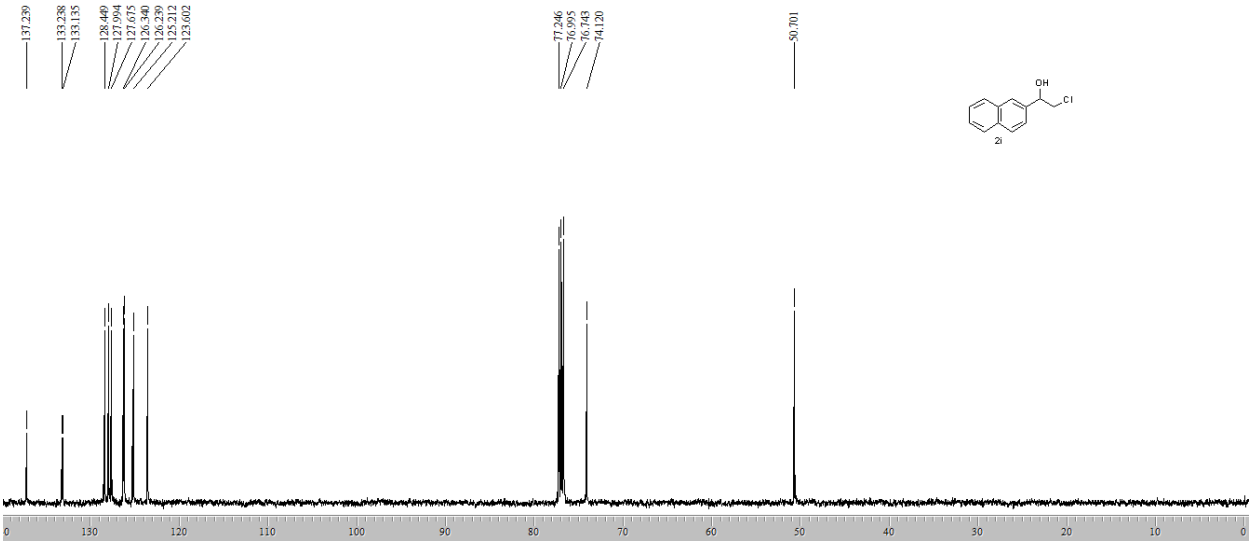
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123.602
77.246
76.995
76.743
74.120
50.701
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2i
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110
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30
20
10
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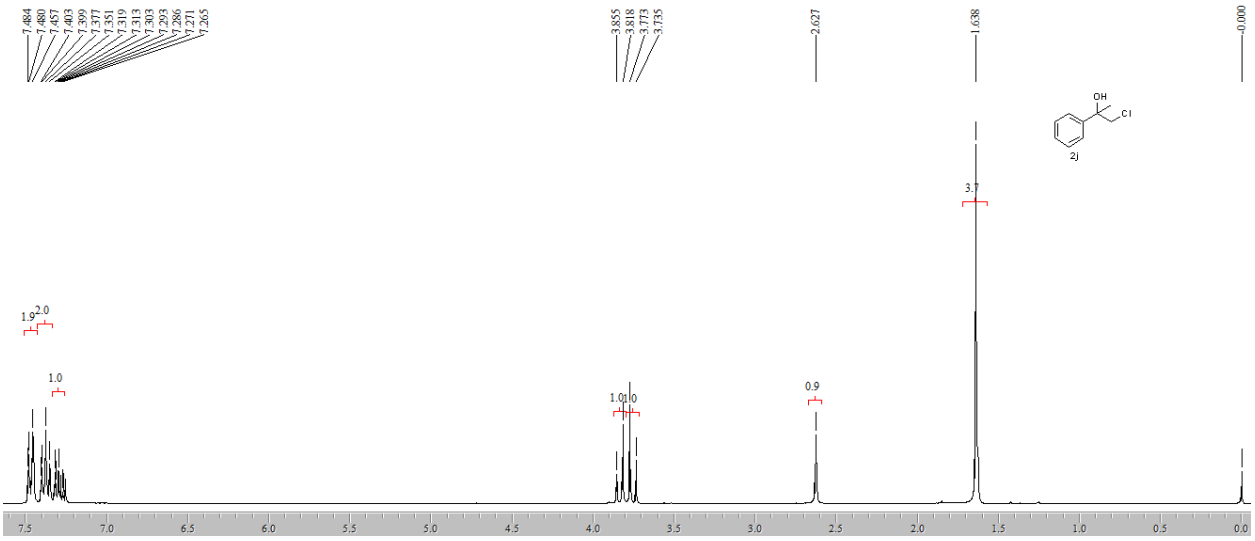
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7.377
7.351
7.319
7.313
7.303
7.293
7.286
7.271
7.265
3.855
3.818
3.773
3.735
2.627
1.638
0.000
OH
Cl
2j
1.9
2.0
1.0
1.0
1.0
0.9
3.7
7.5
7.0
6.5
6.0
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3.5
3.0
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2.0
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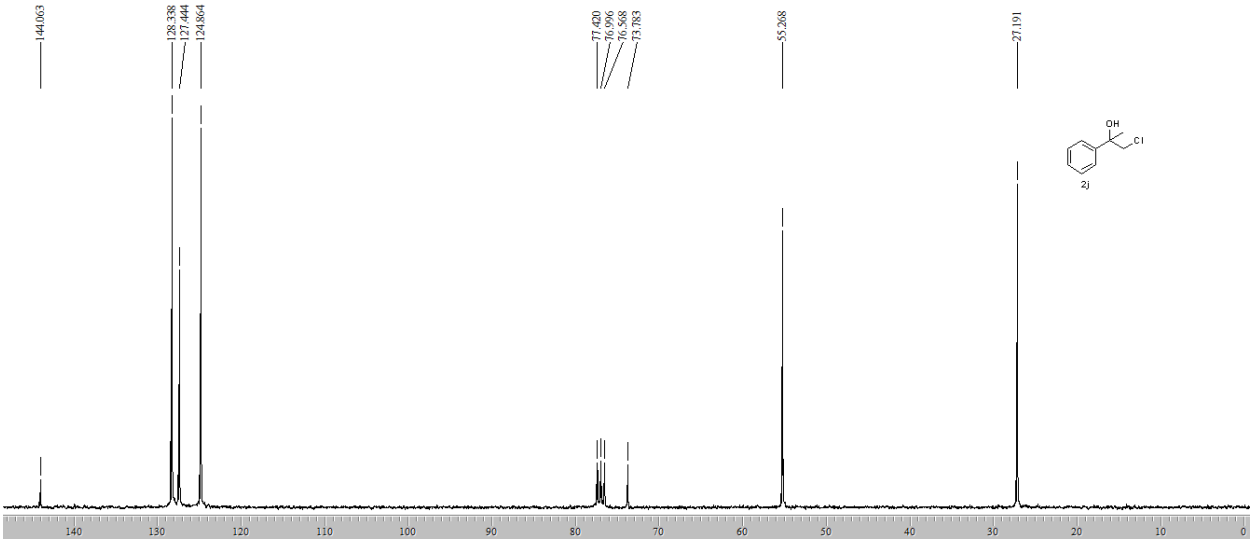
0.0


144.063
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110
100
90
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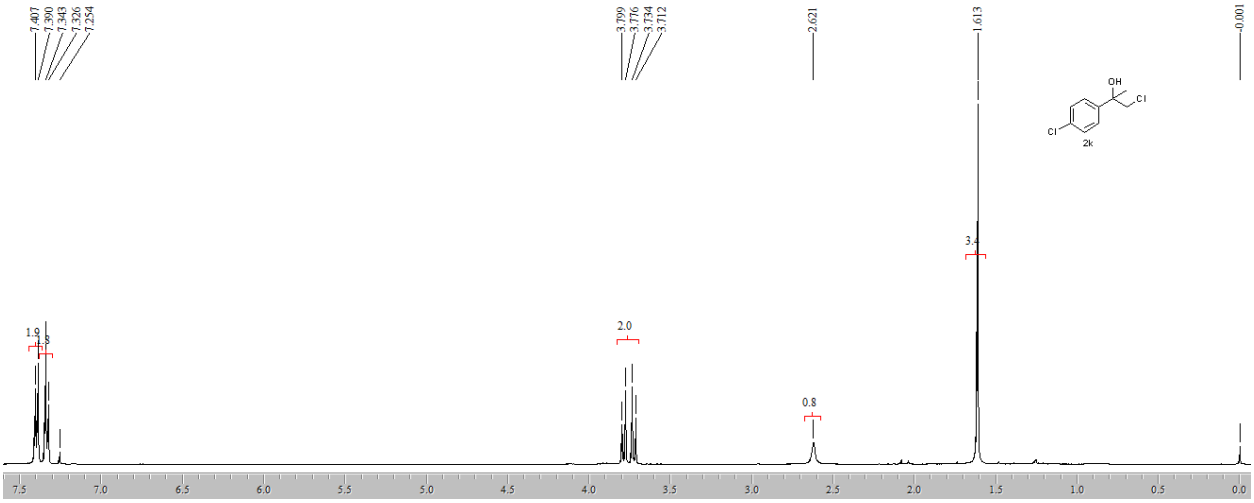
70
60
50
40
30
20
10
0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7.343
7.326
7.254
3.799
3.776
3.734
3.712
2.621
1.613
0.001
OH
Cl
Cl
2k
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1.8
2.0
0.8
3.4
7.5
7.0
6.5
6.0
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3.0
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2.0
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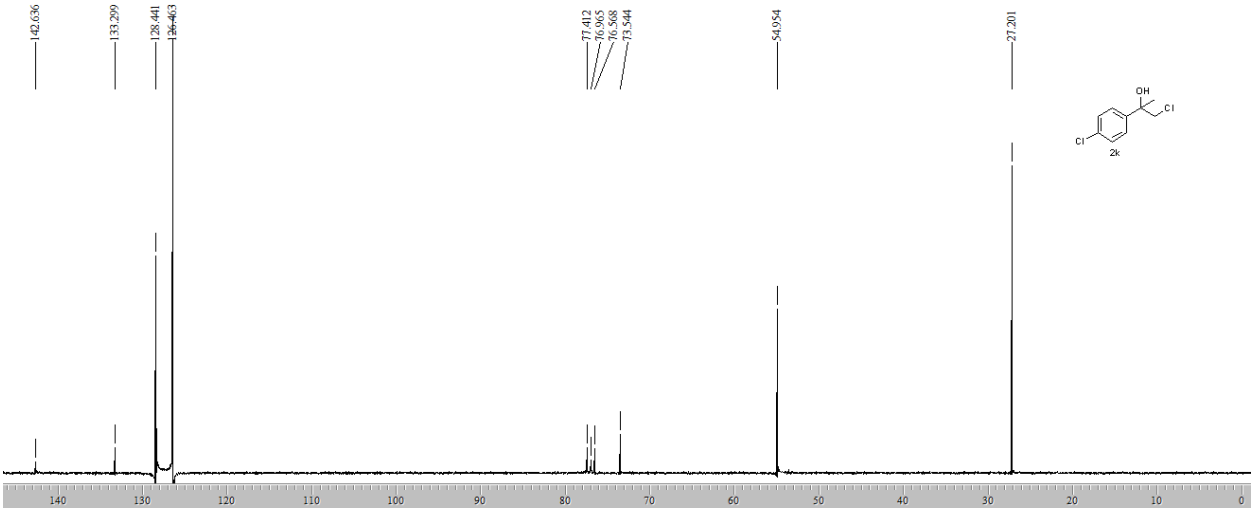
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73.544
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110
100
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70
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50
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30
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10
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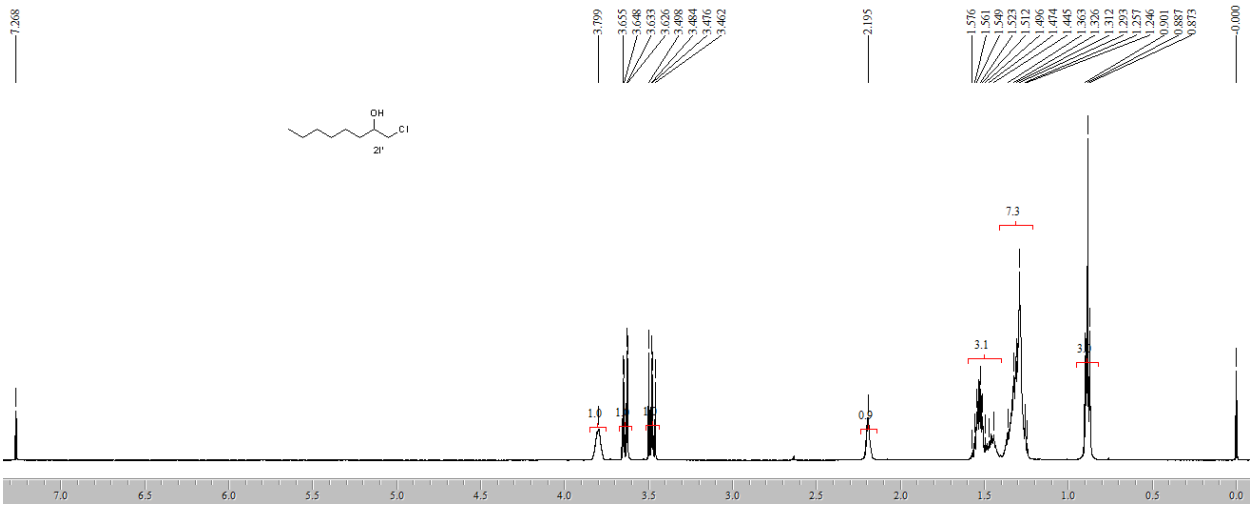
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3.626
3.488
3.484
3.476
3.462
2.195
1.576
1.561
1.549
1.523
1.512
1.496
1.474
1.445
1.363
1.326
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1.293
1.257
1.246
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0.887
0.873
0.000
OH
Cl
2l'
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1.0
0.9
7.0
6.5
6.0
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4.0
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2.0
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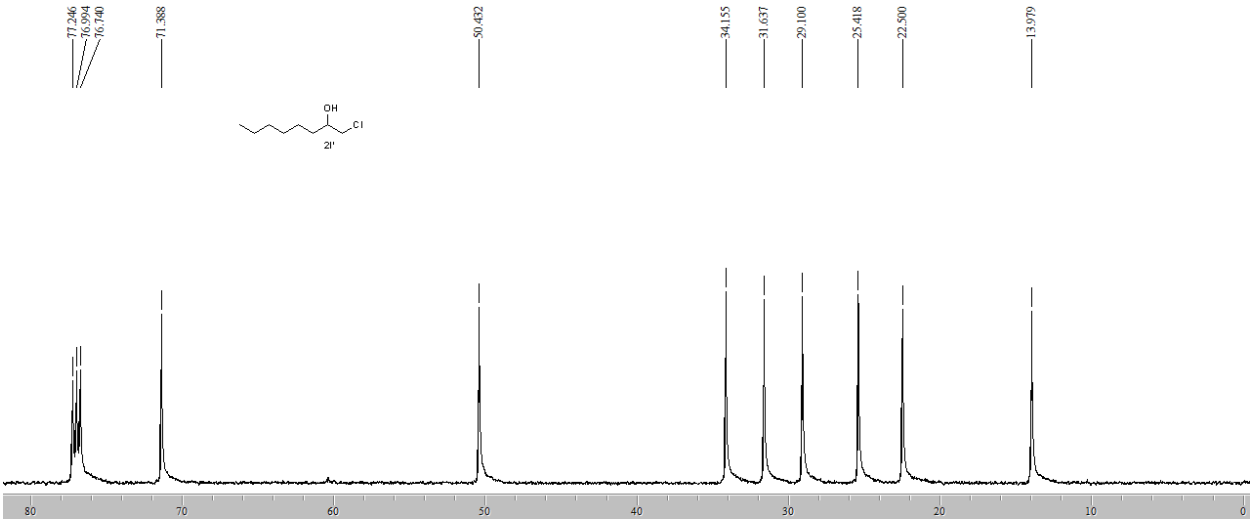
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22.500
13.979
OH
Cl
2l'
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70
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10
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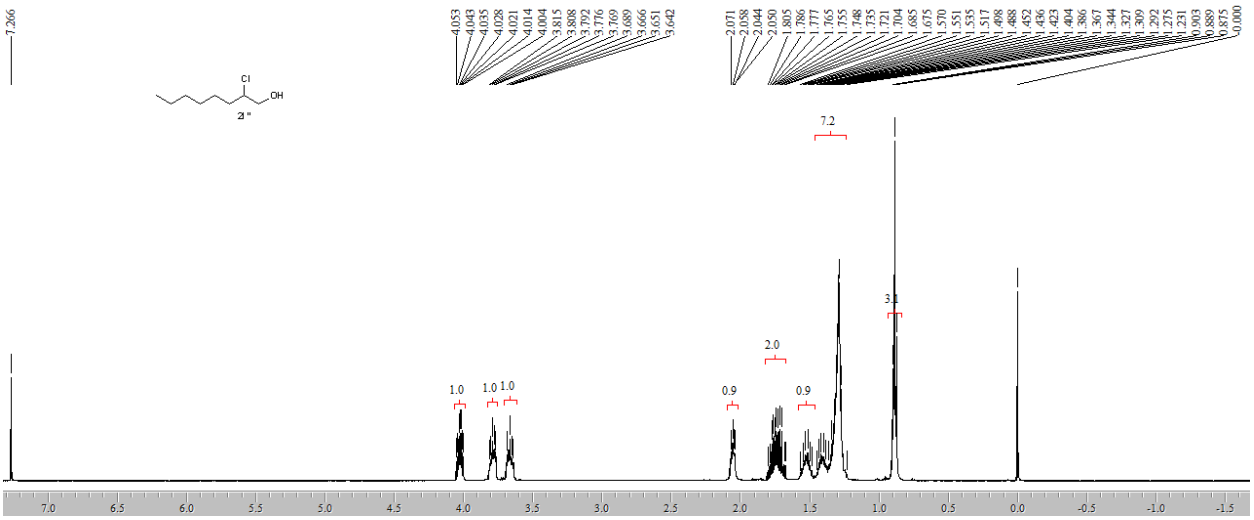
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6.0
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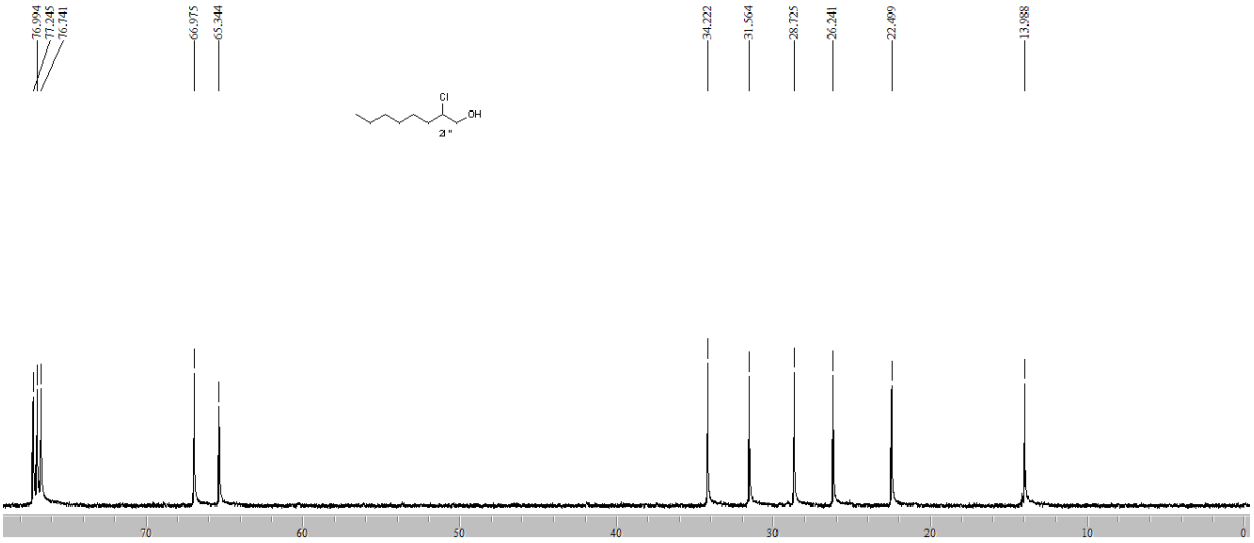
-1.5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34.222
31.564
28.725
26.241
22.499
13.988
Cl
OH
2a
70
60
50
40
30
20
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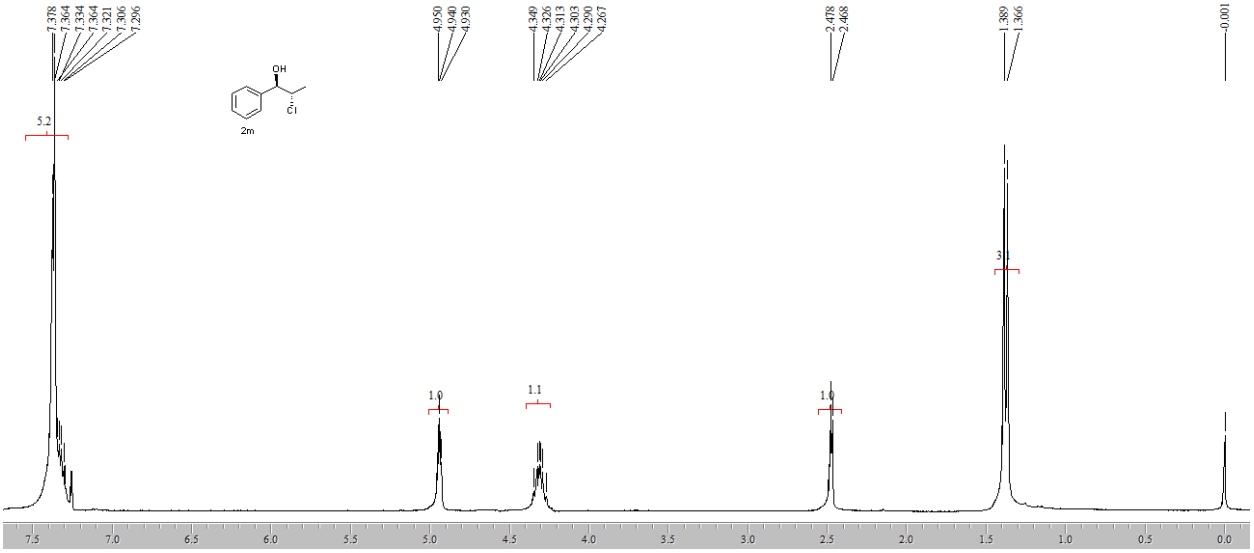
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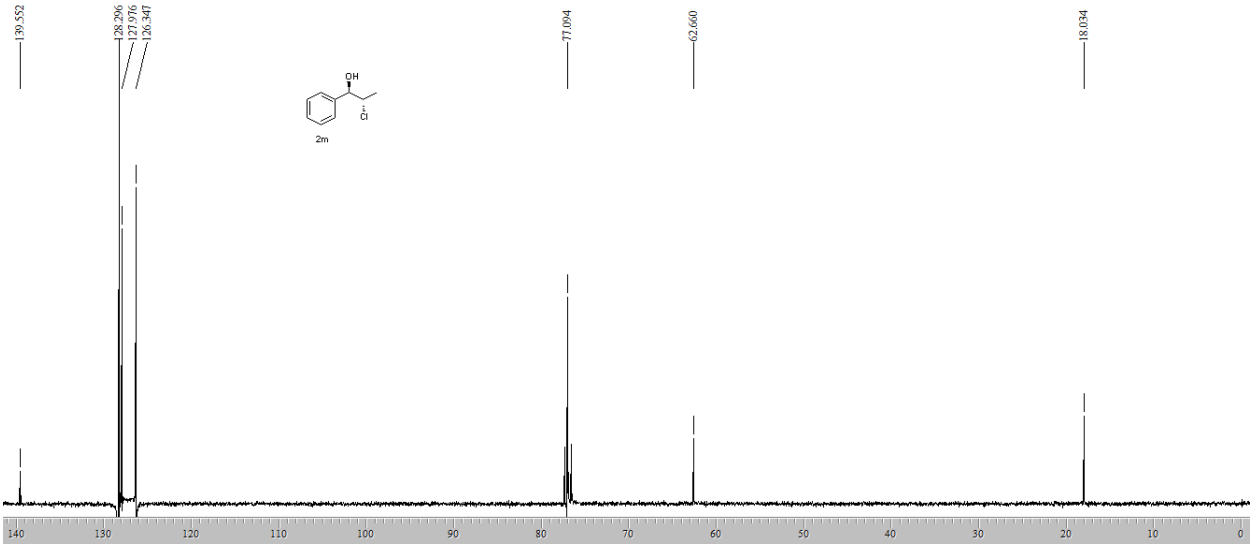
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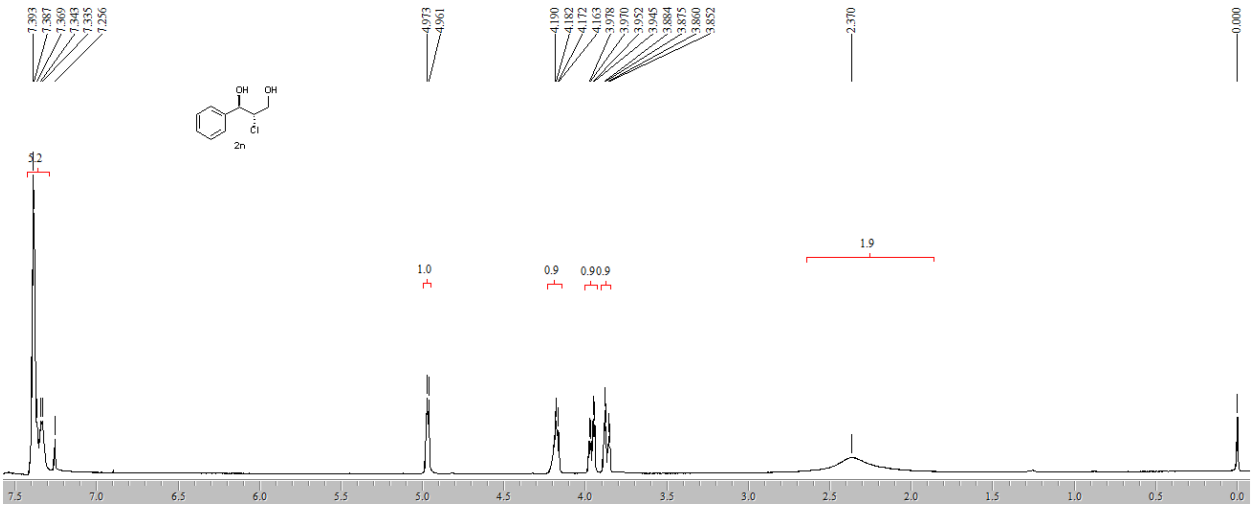
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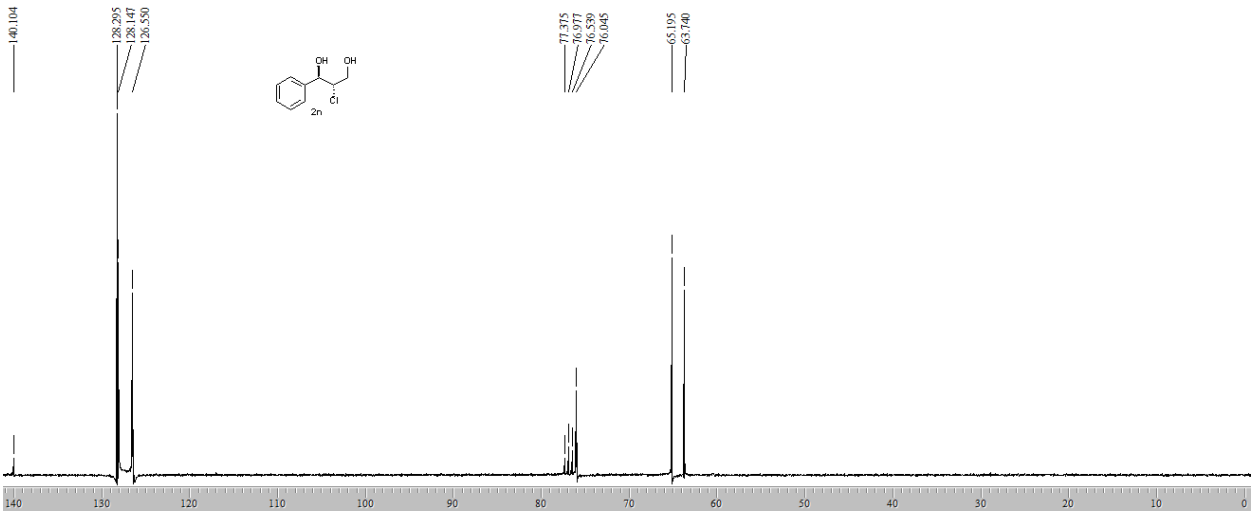
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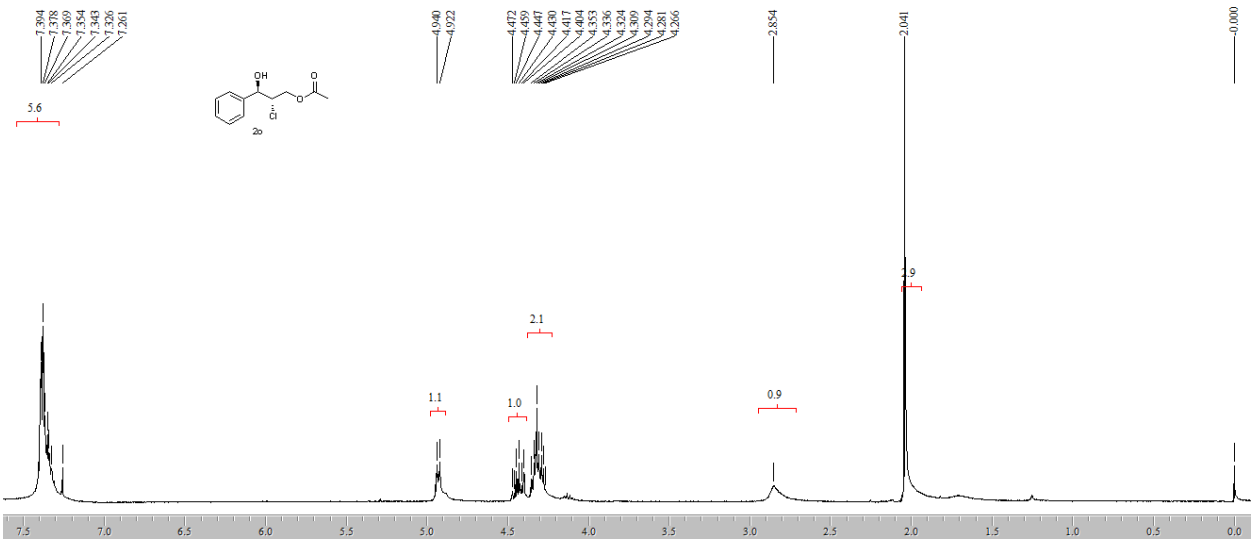
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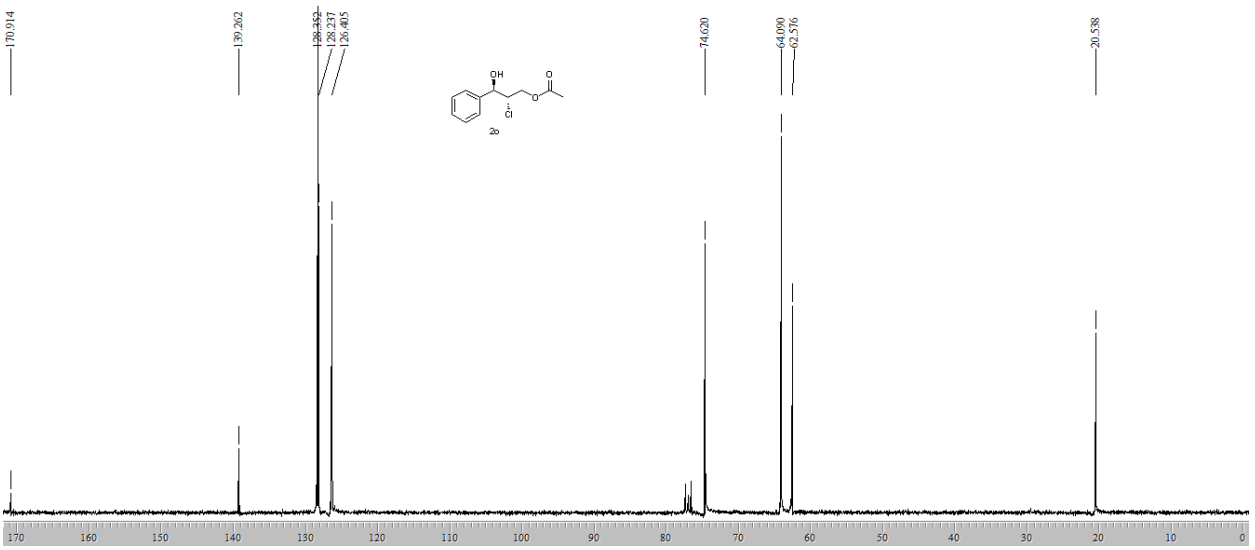
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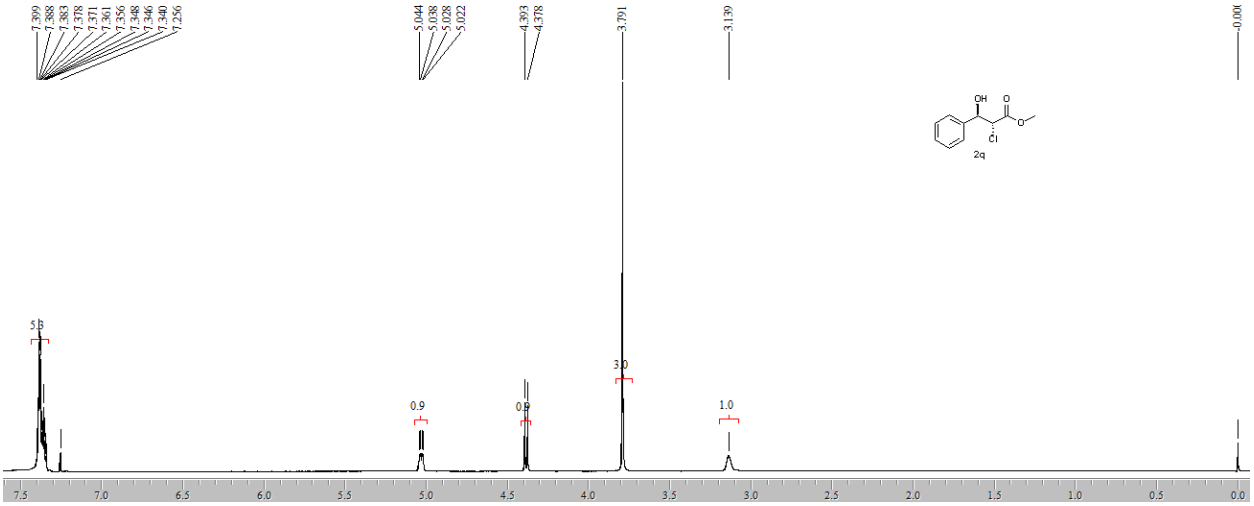
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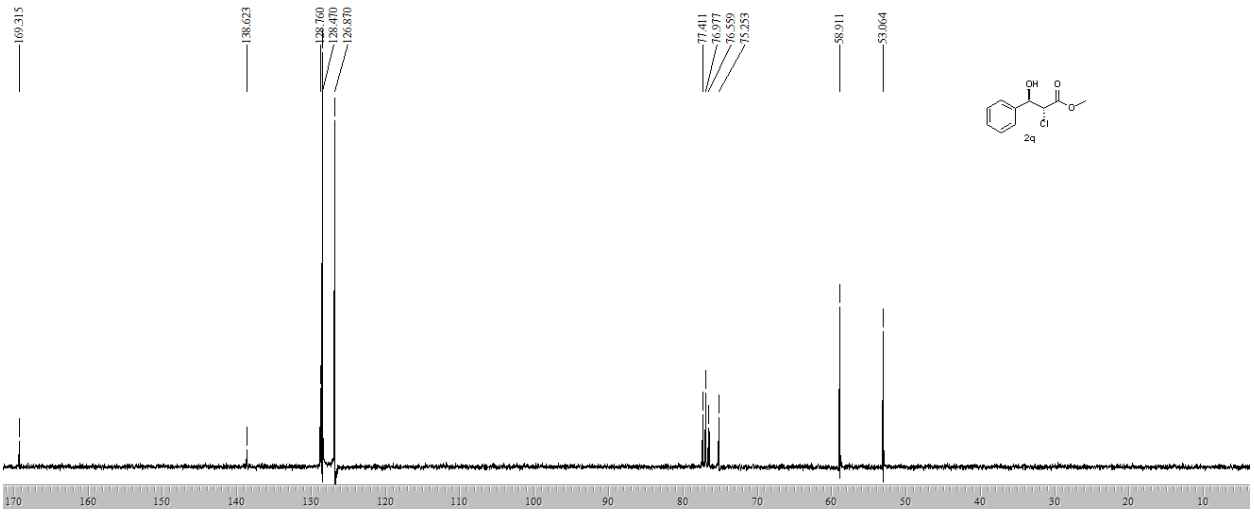
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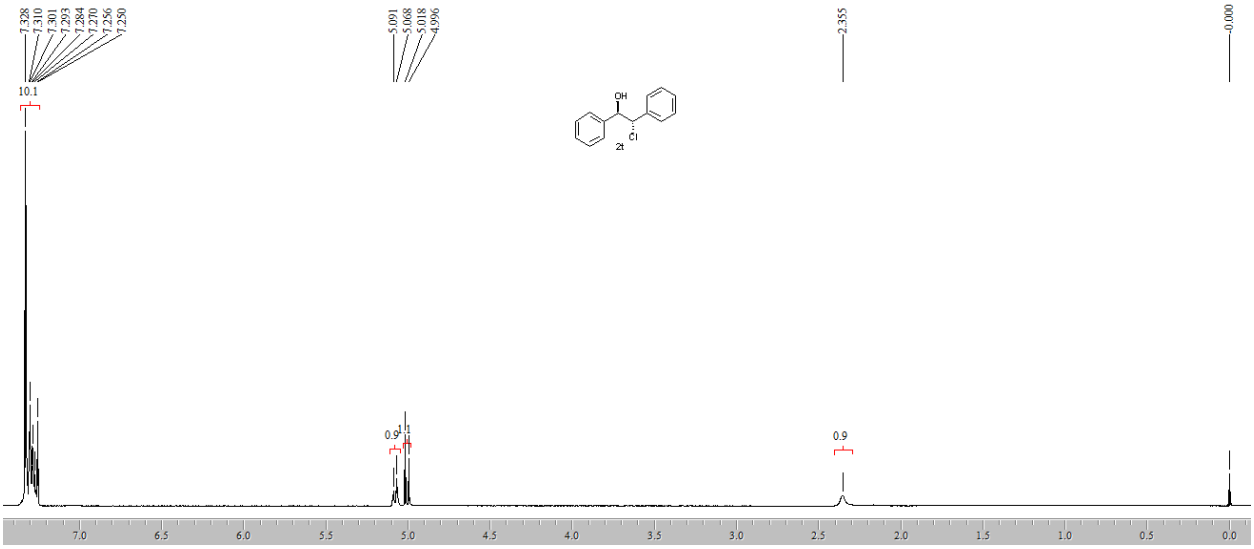
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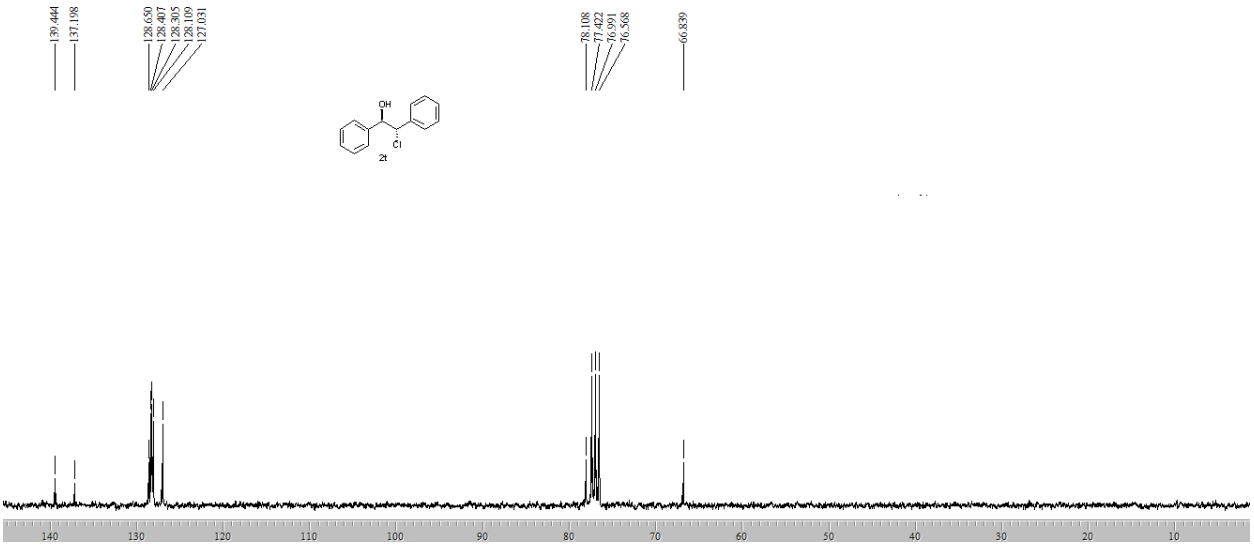
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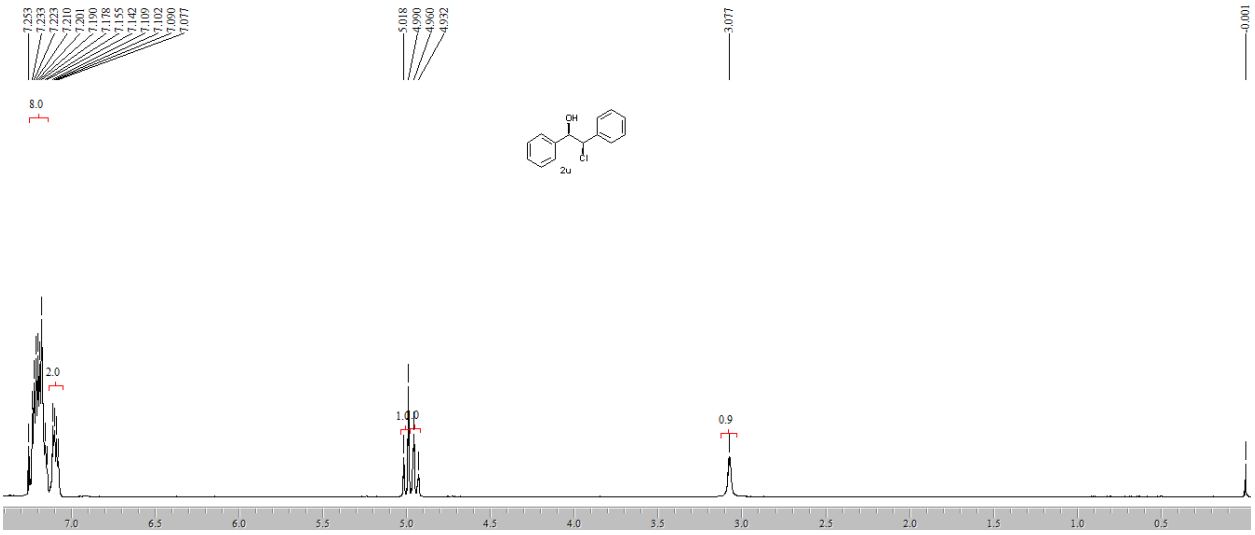
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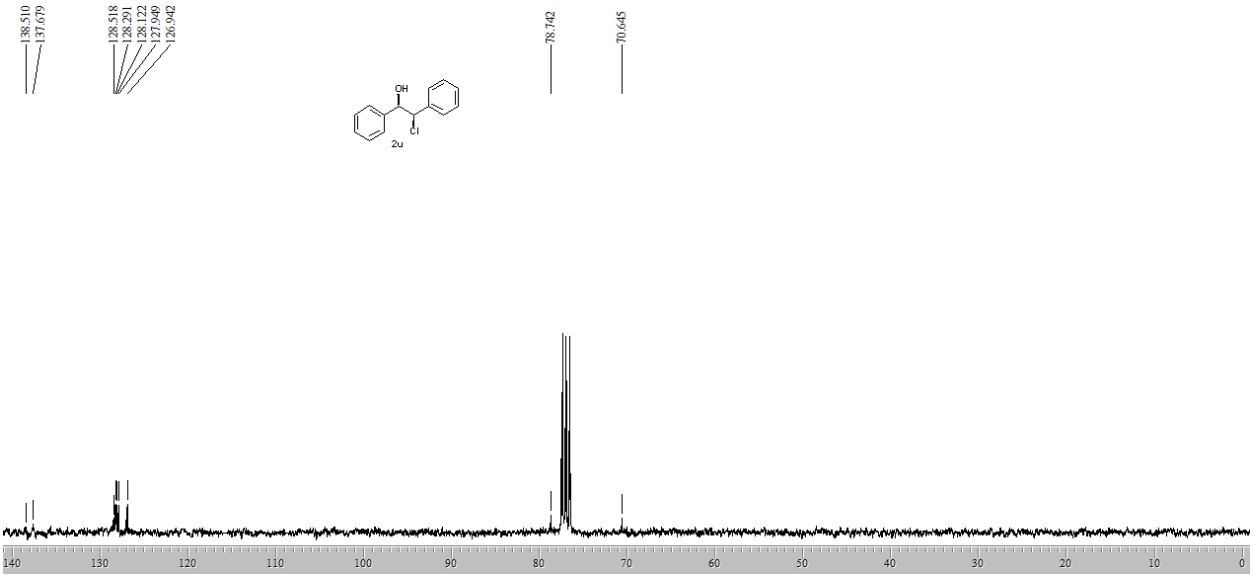
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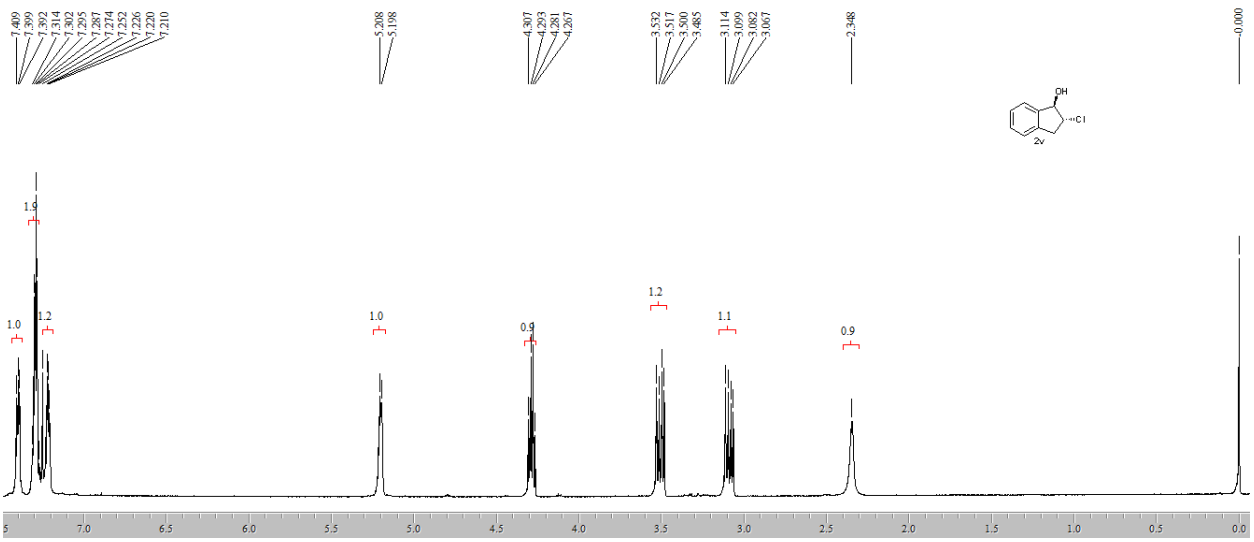
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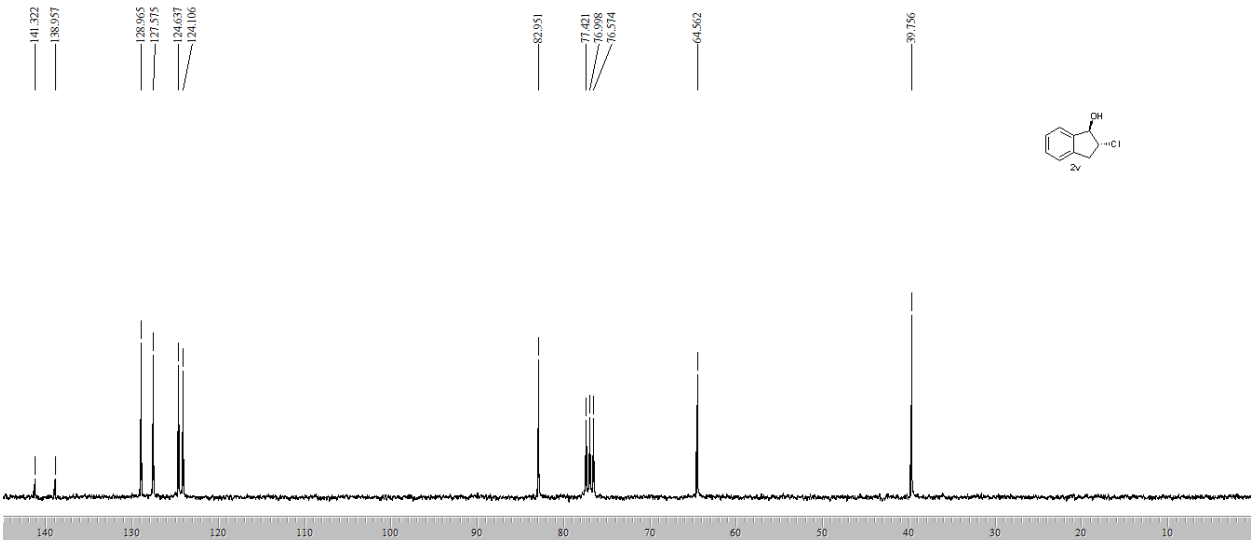
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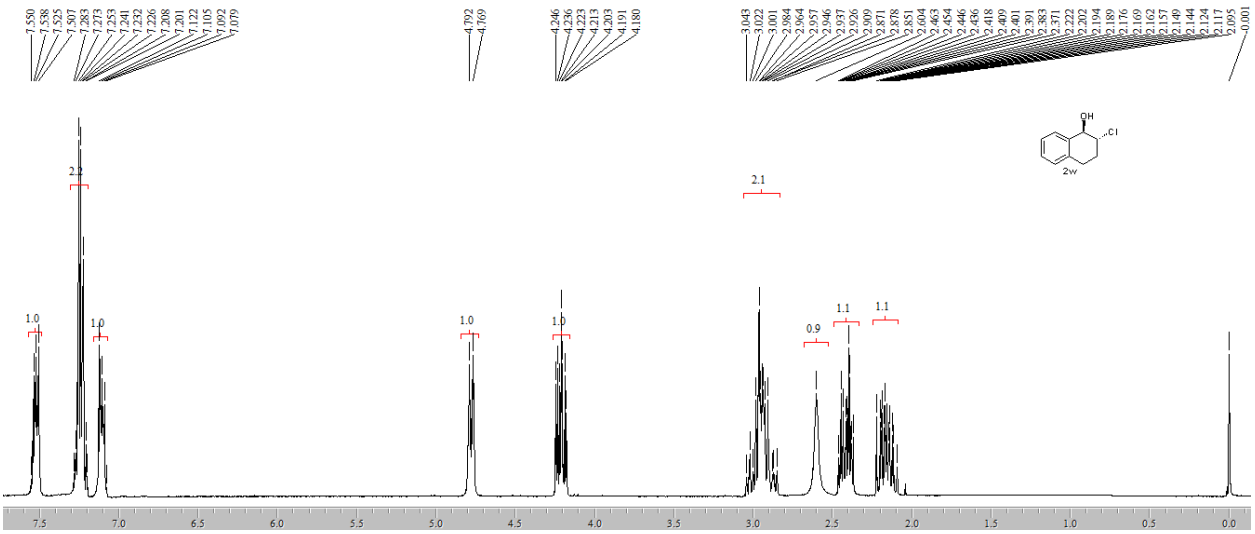
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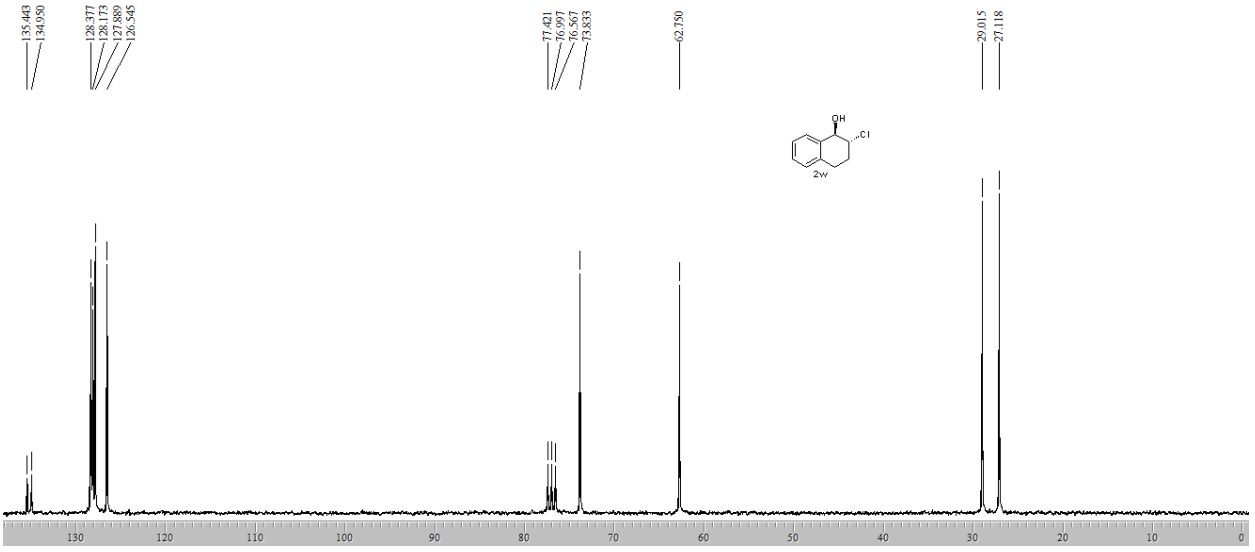
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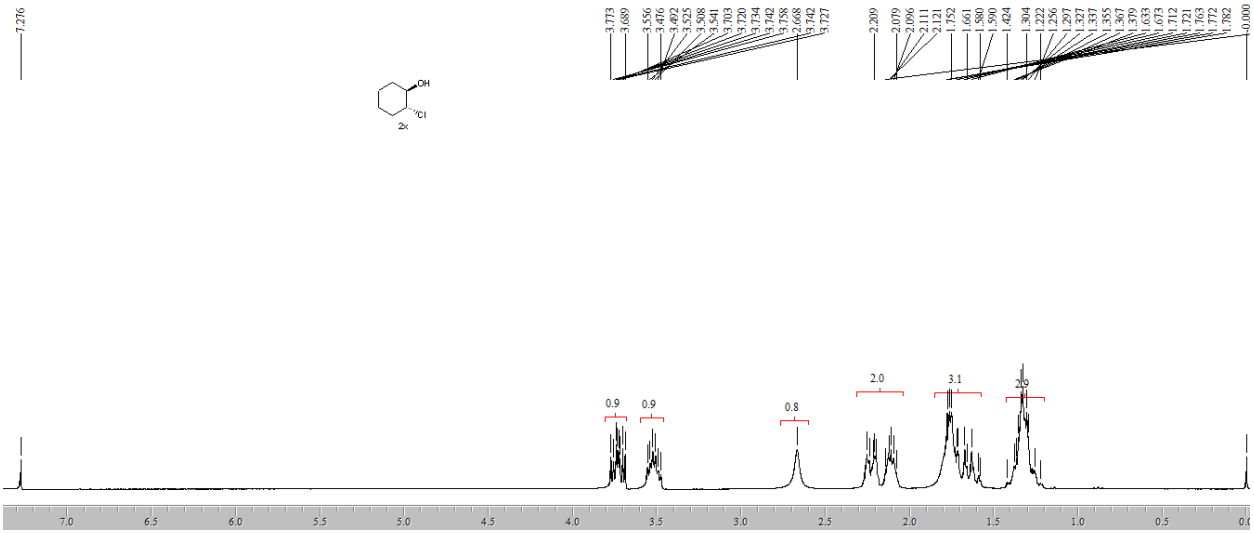
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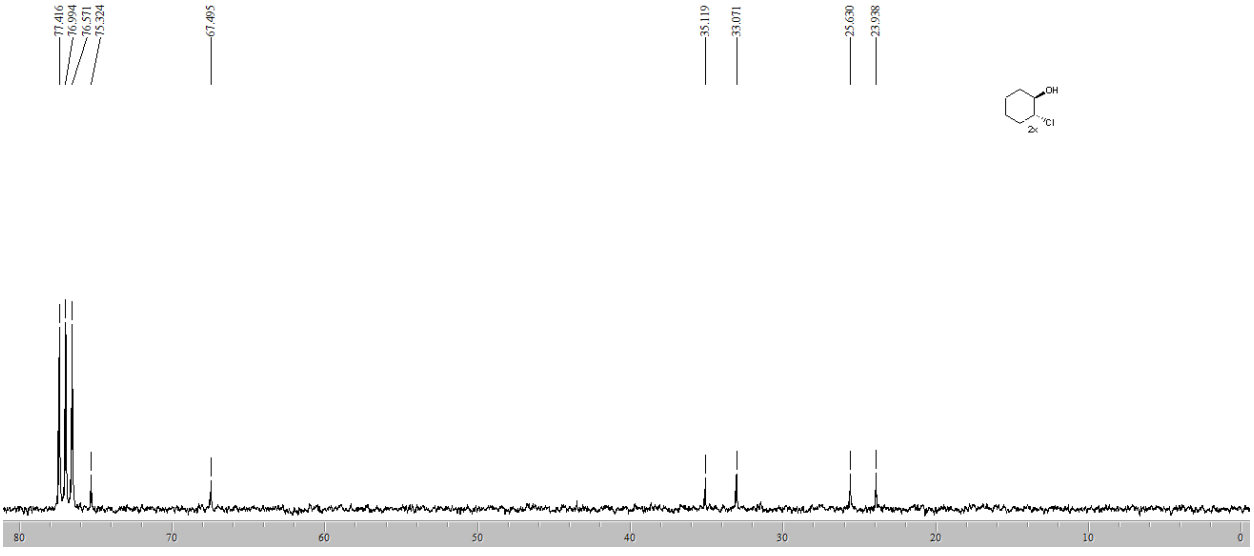
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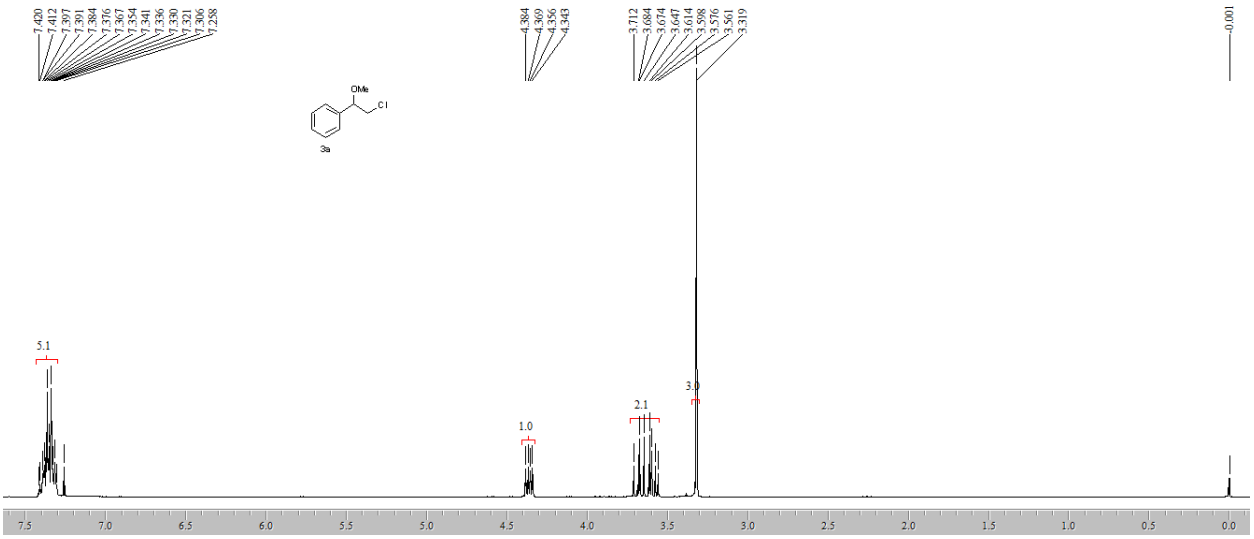
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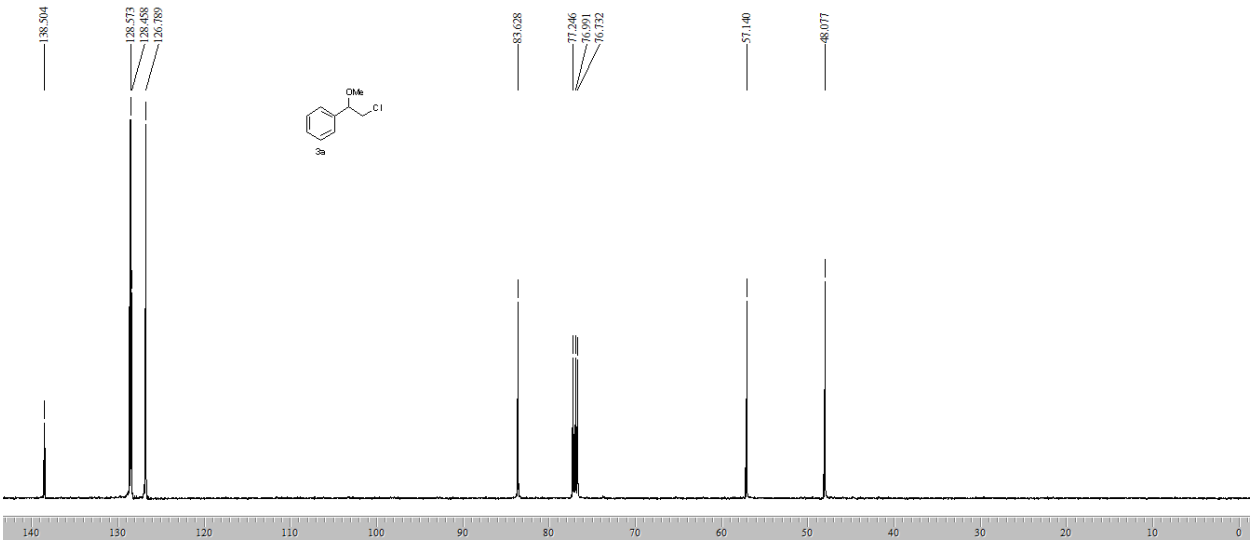
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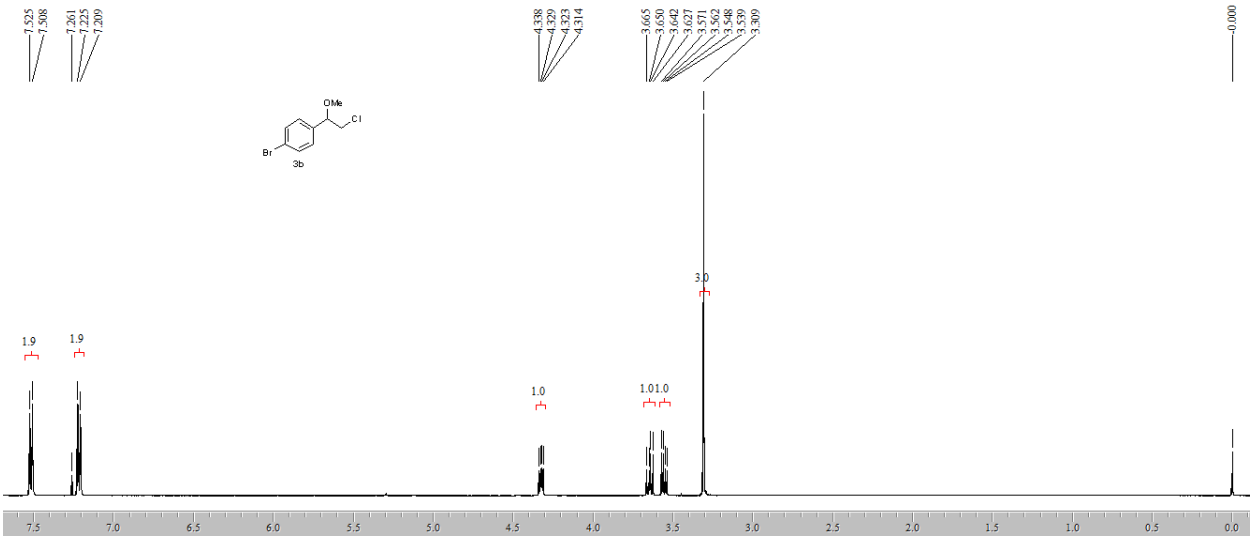
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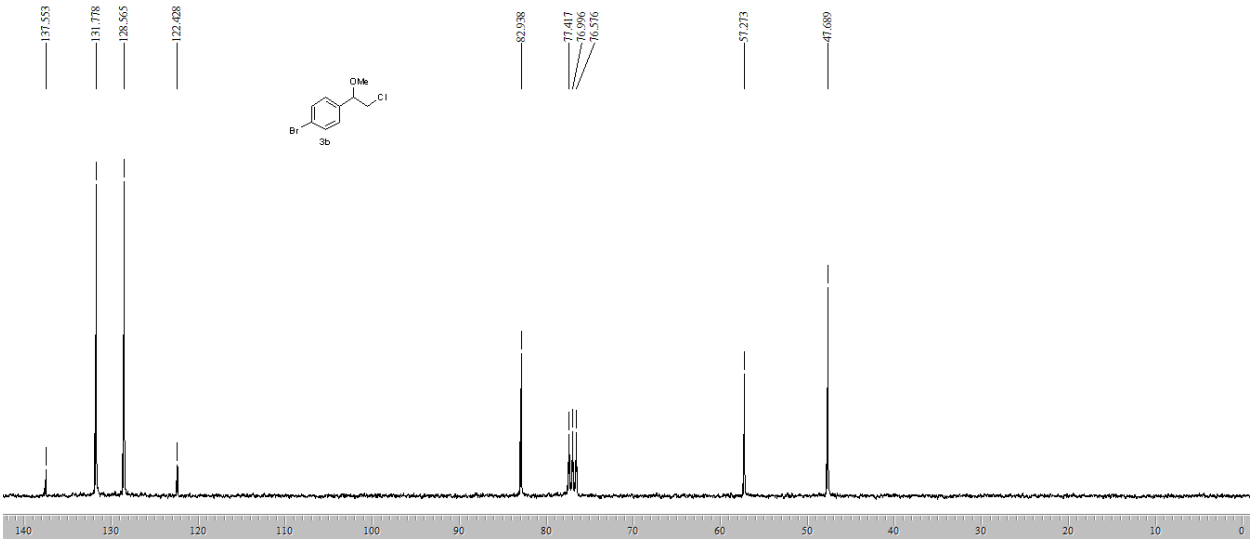
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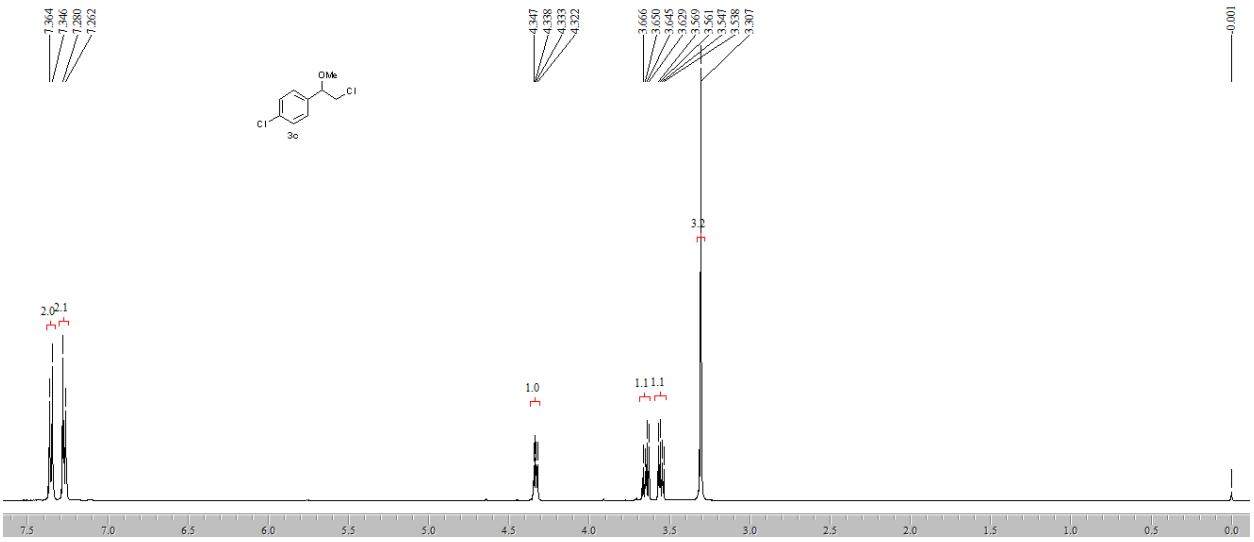
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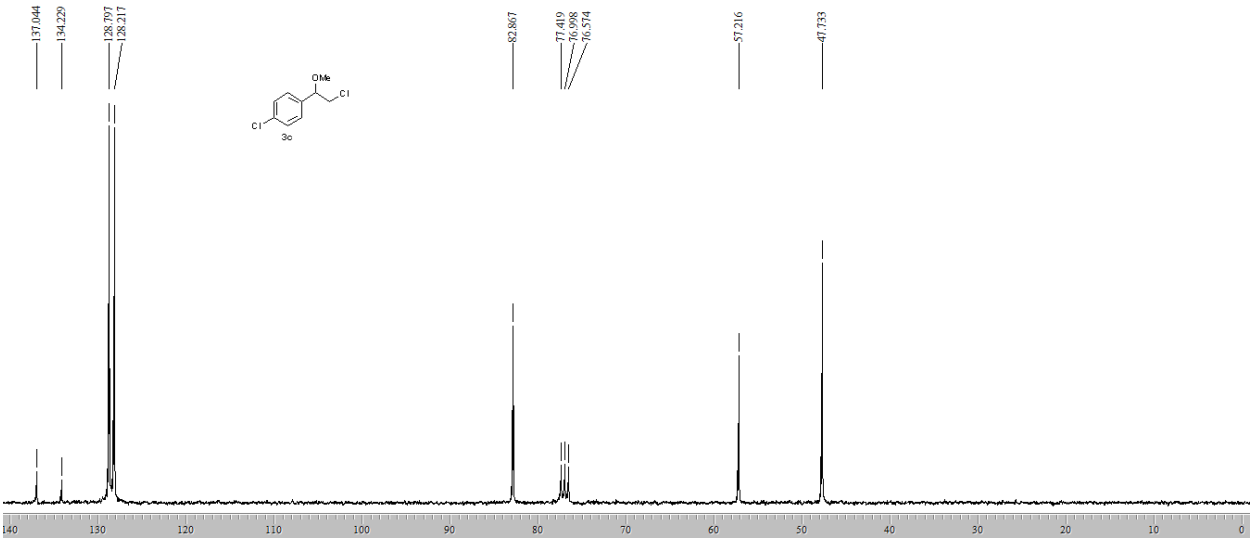
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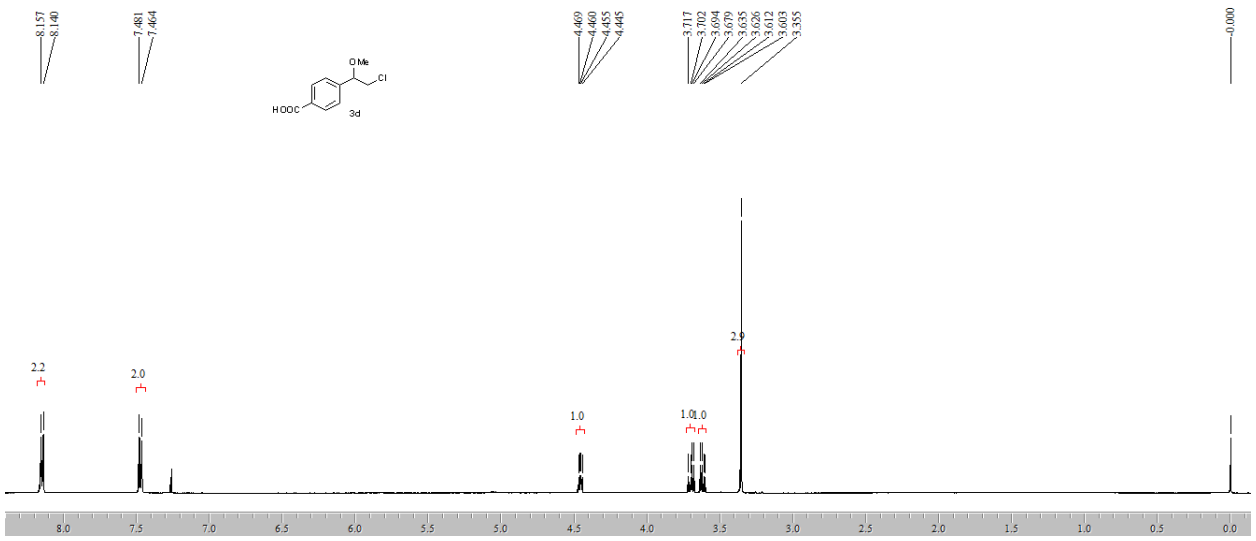
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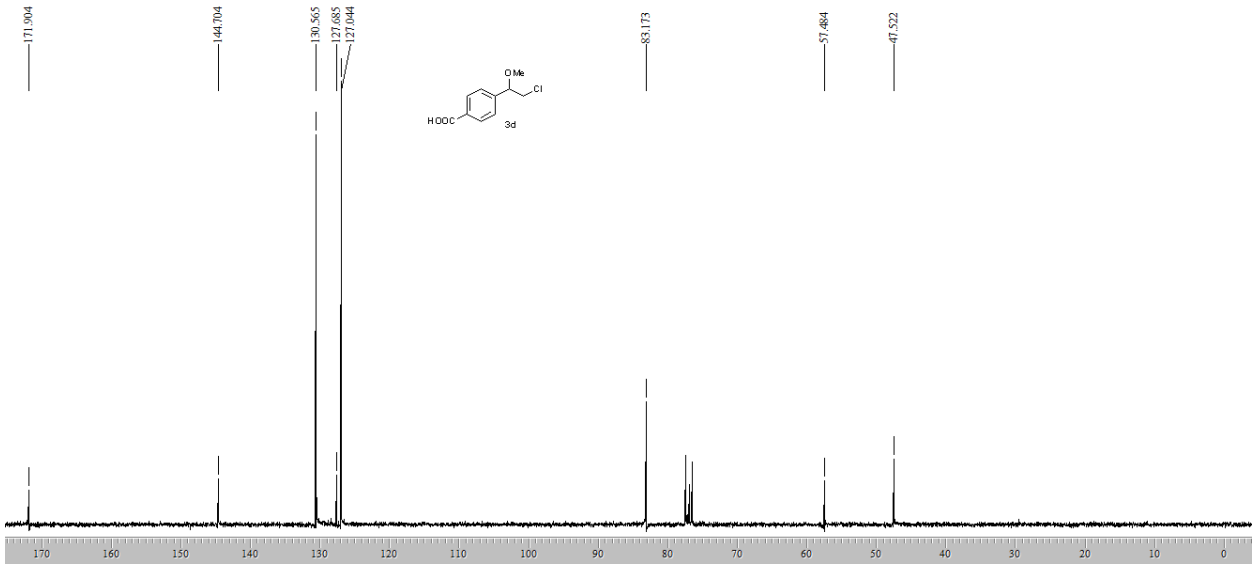
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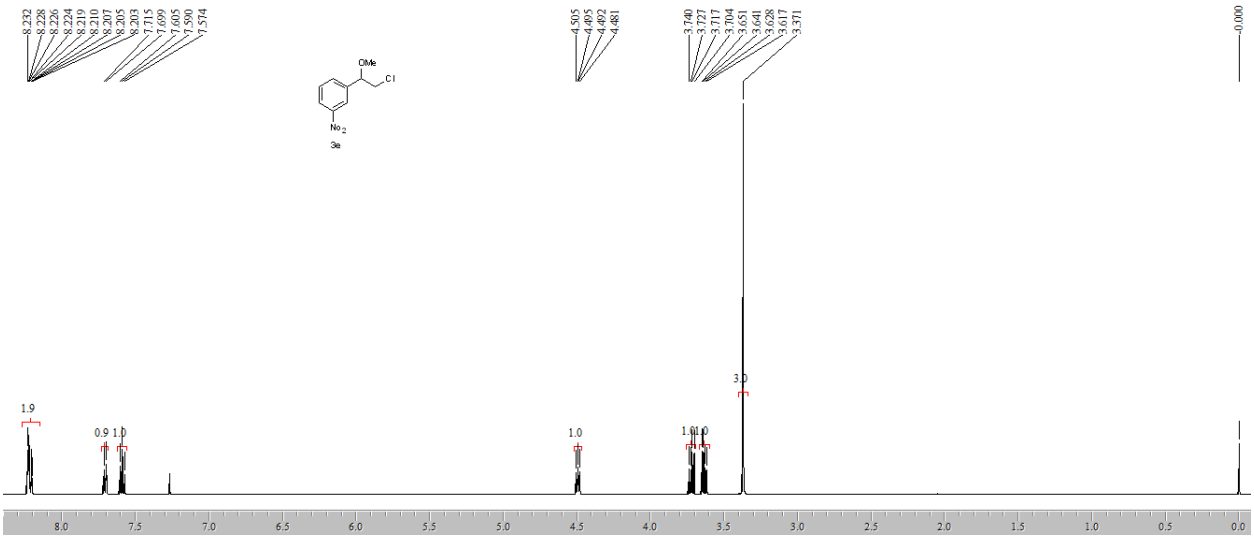
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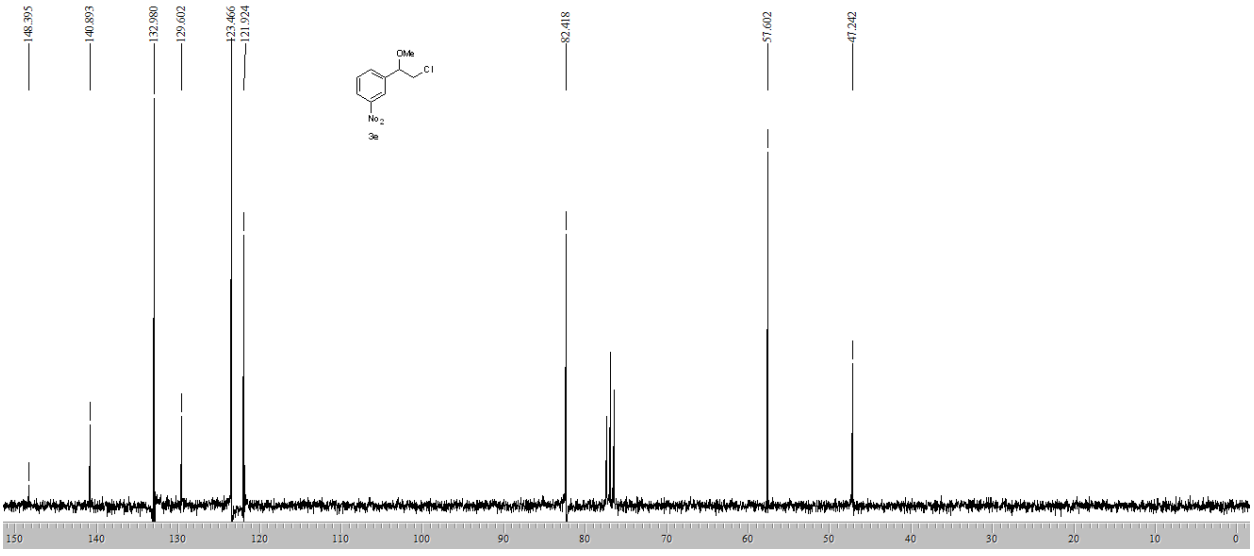
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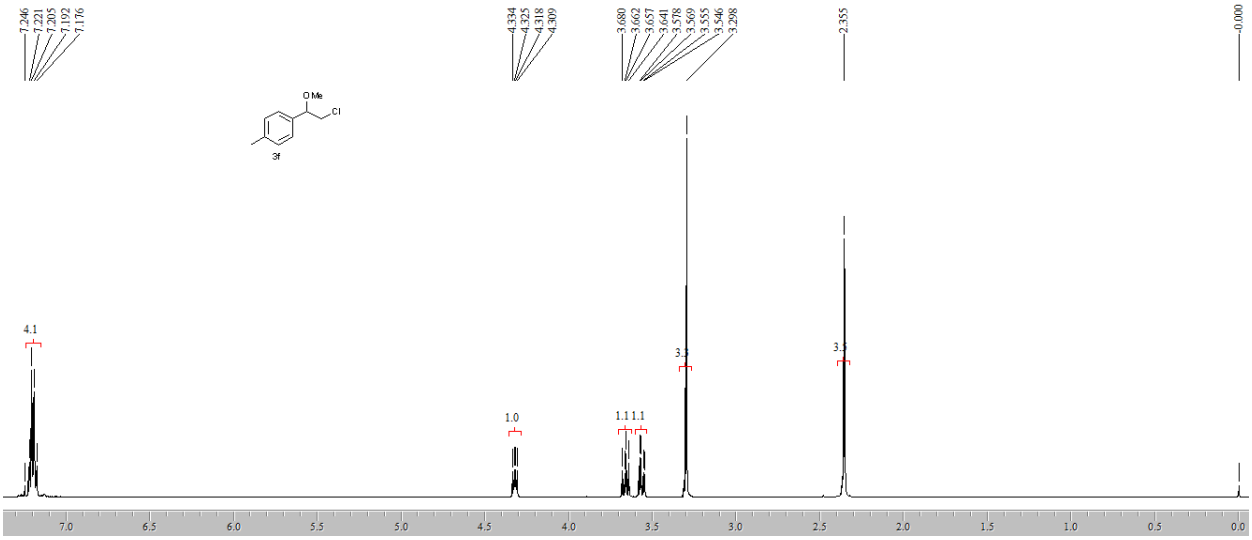
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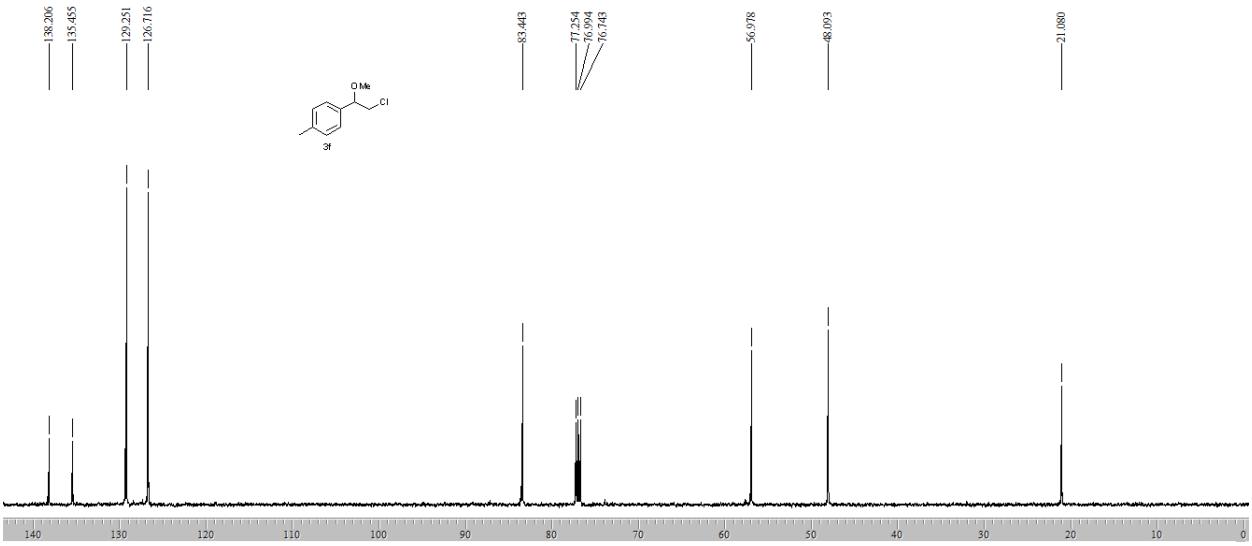
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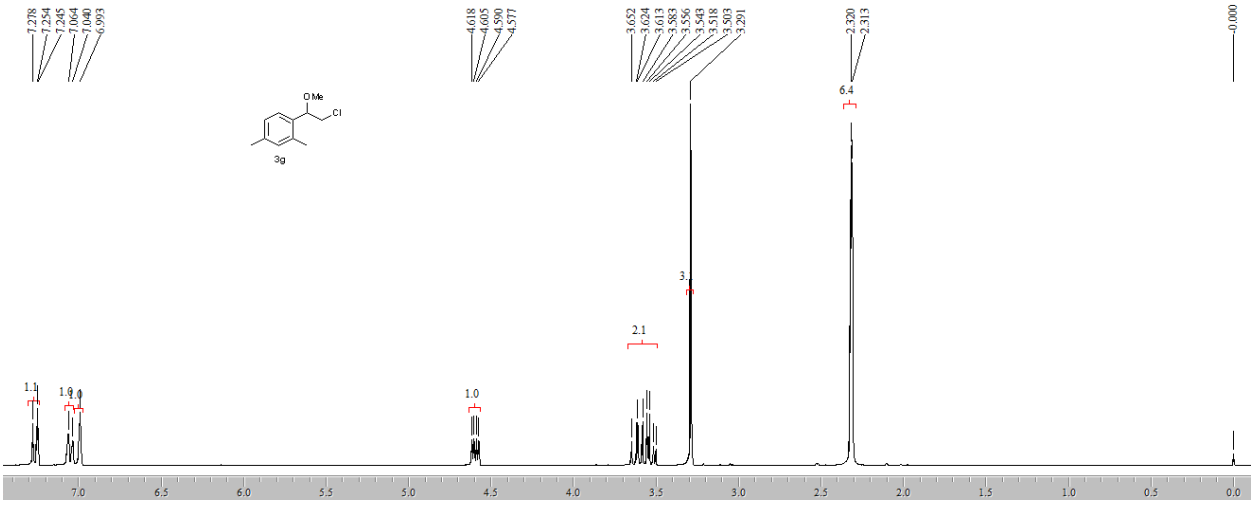
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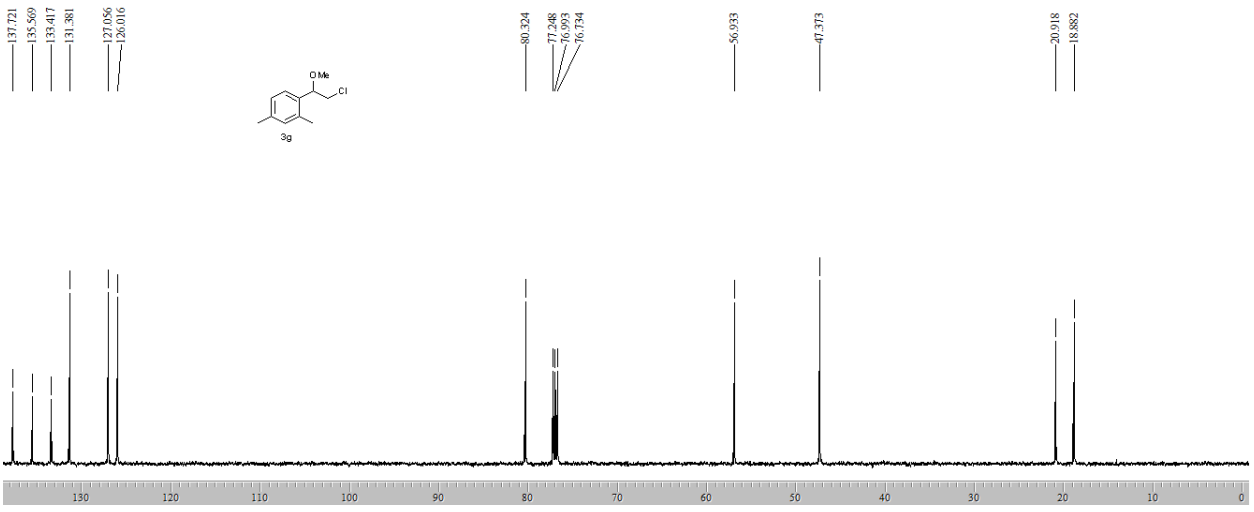
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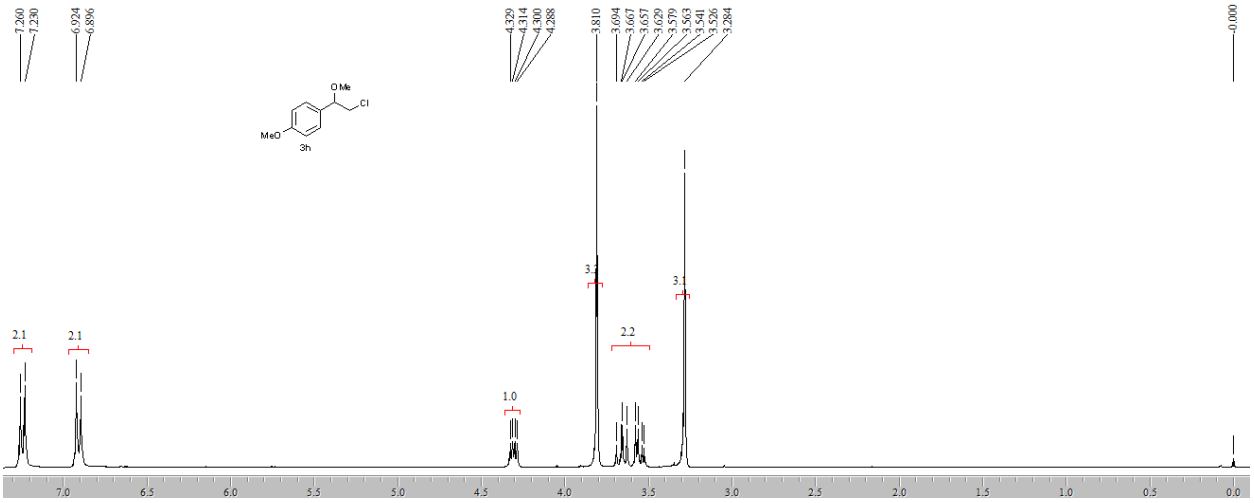
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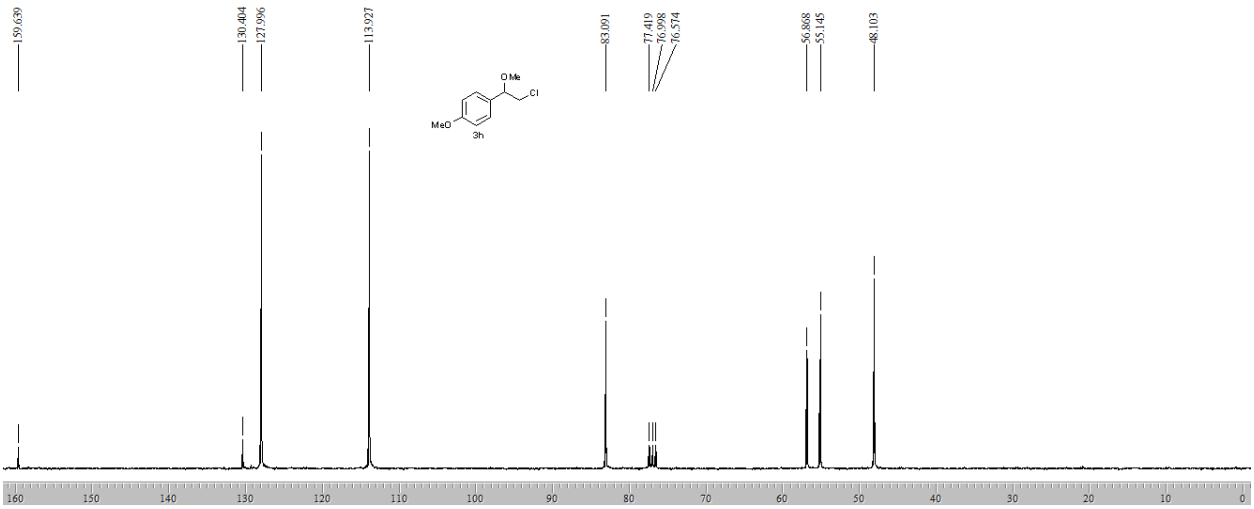
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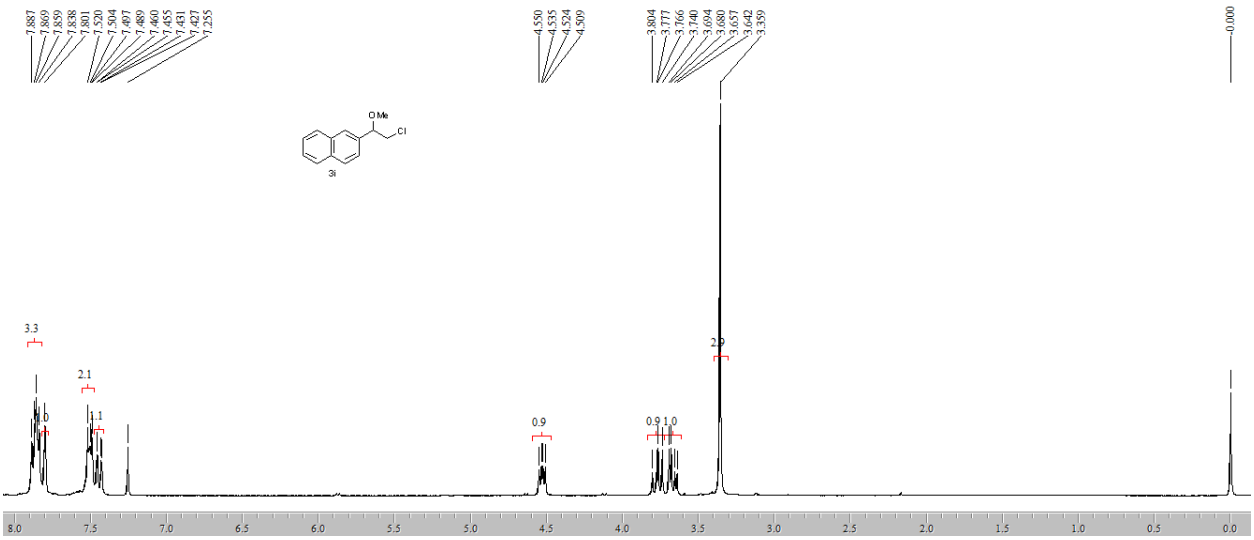
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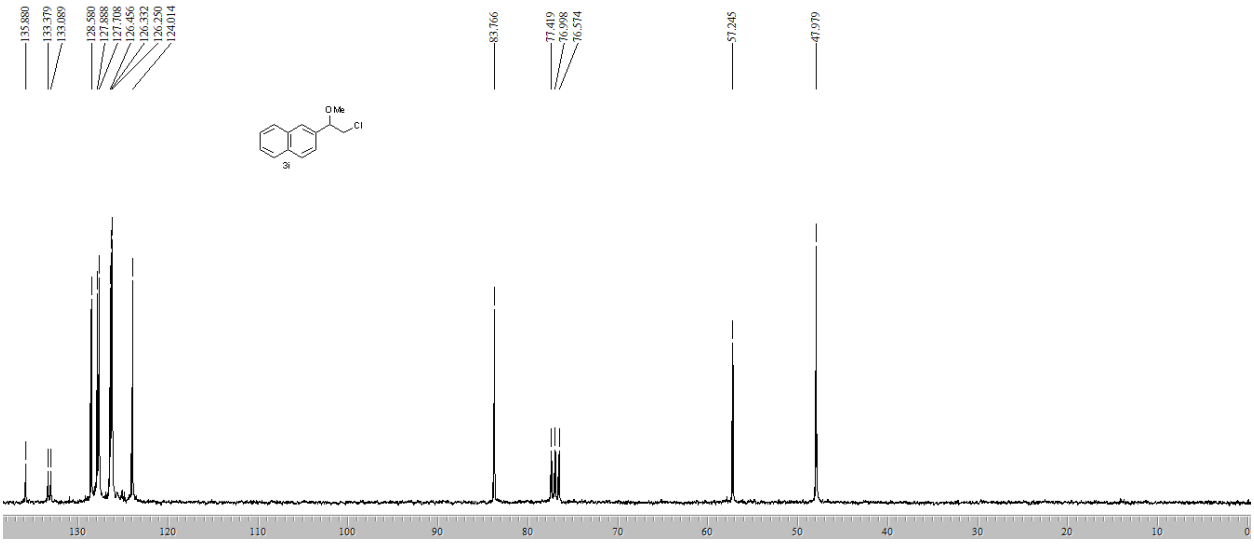
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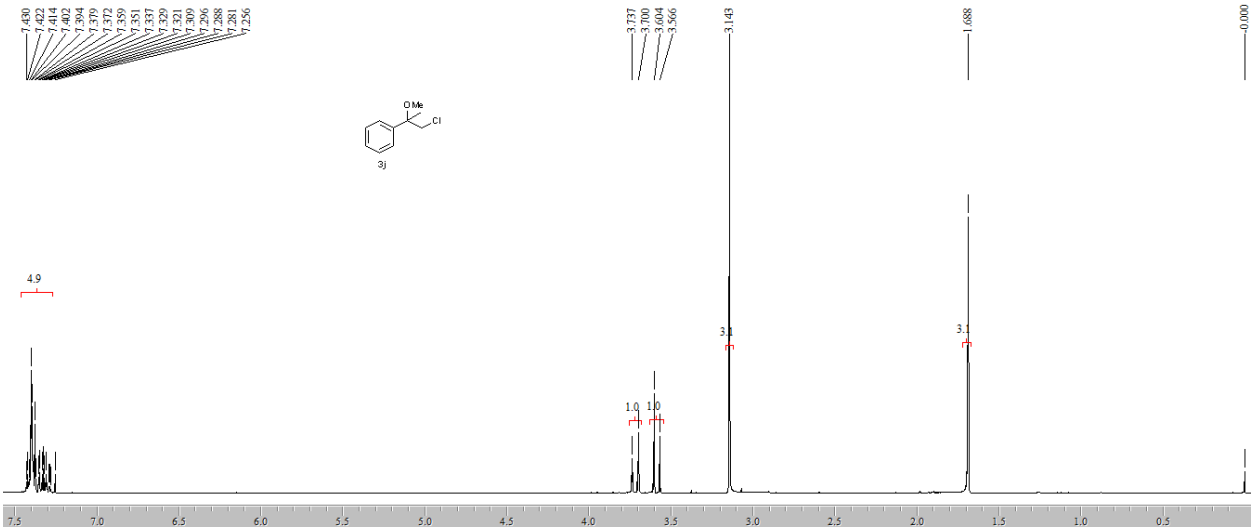
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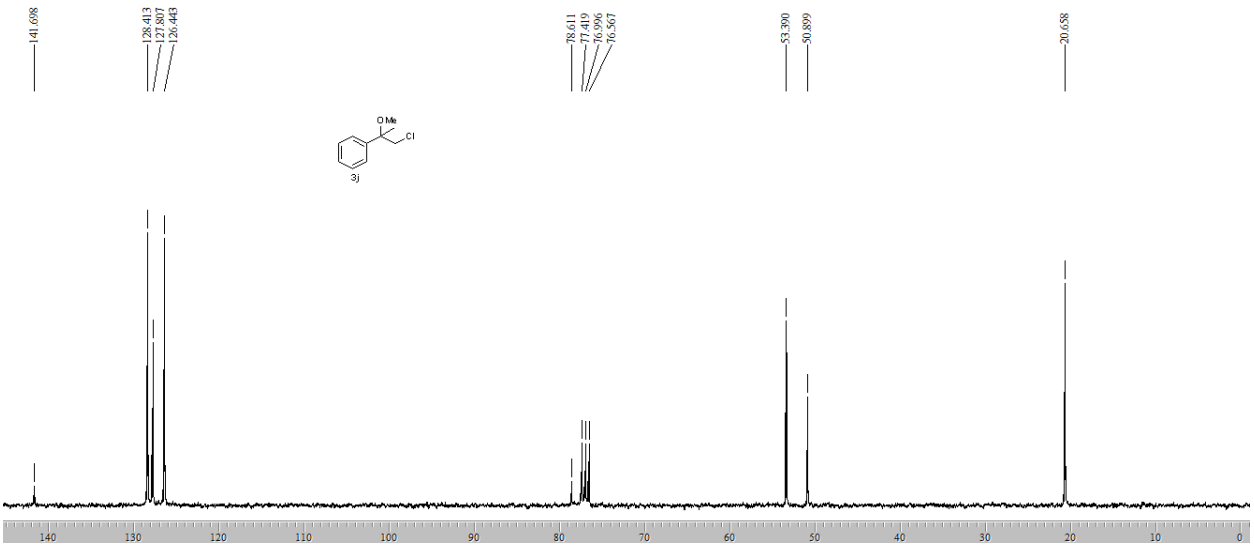
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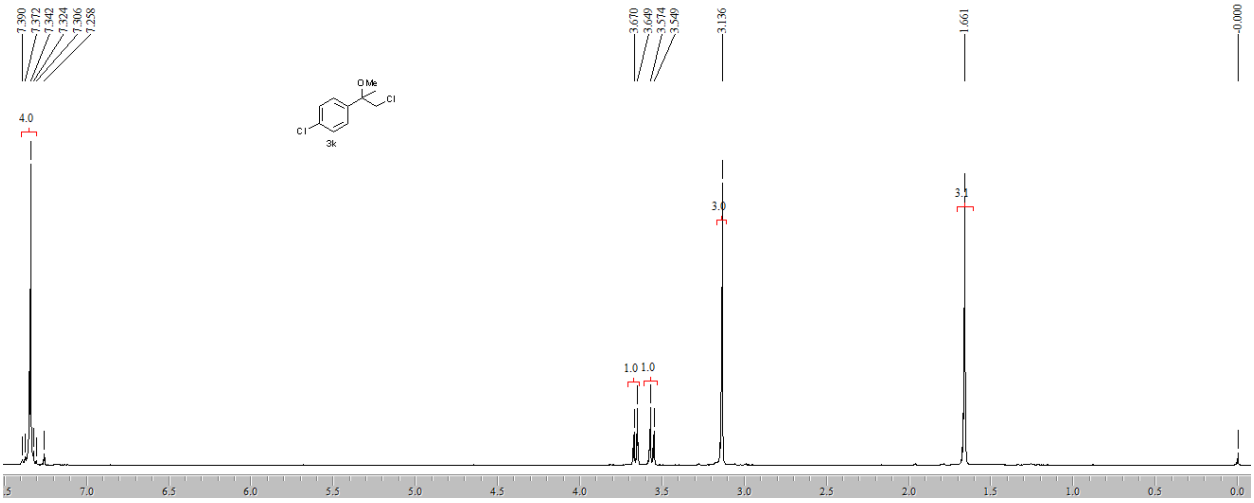
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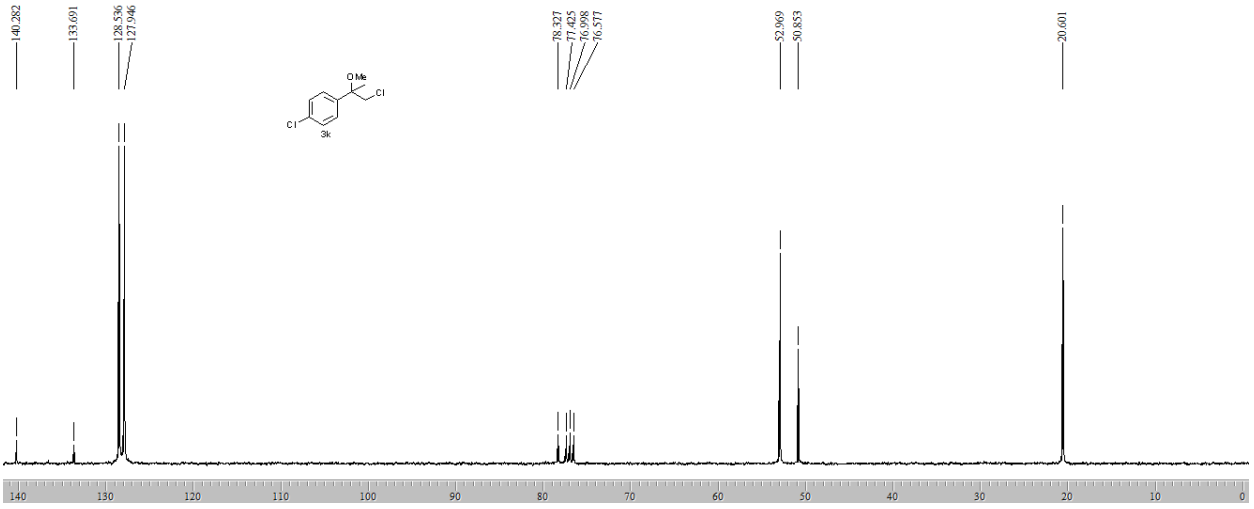
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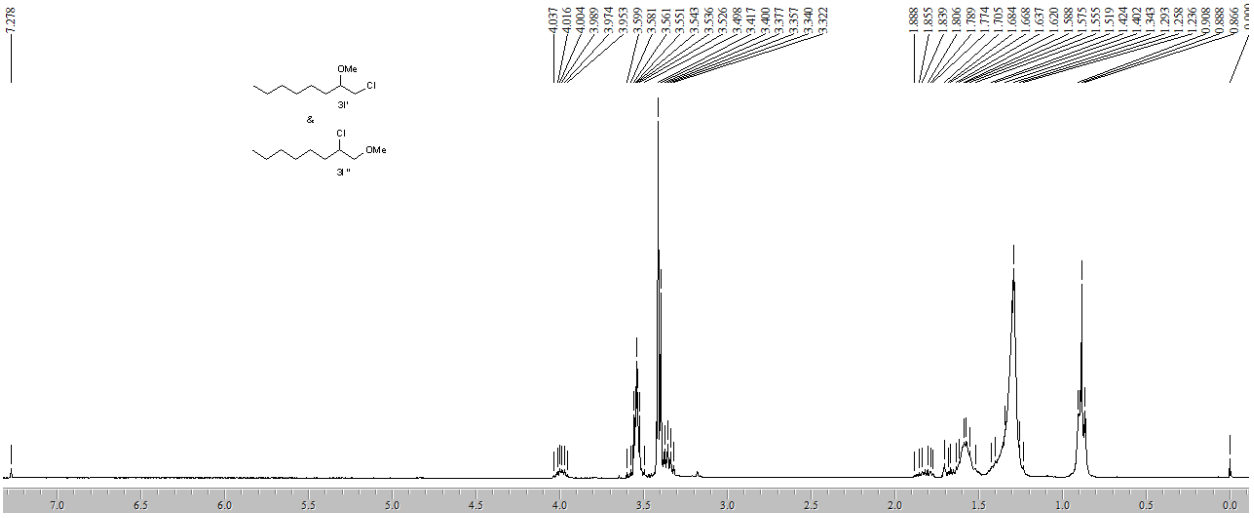
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&
Cl
OMe
3l''
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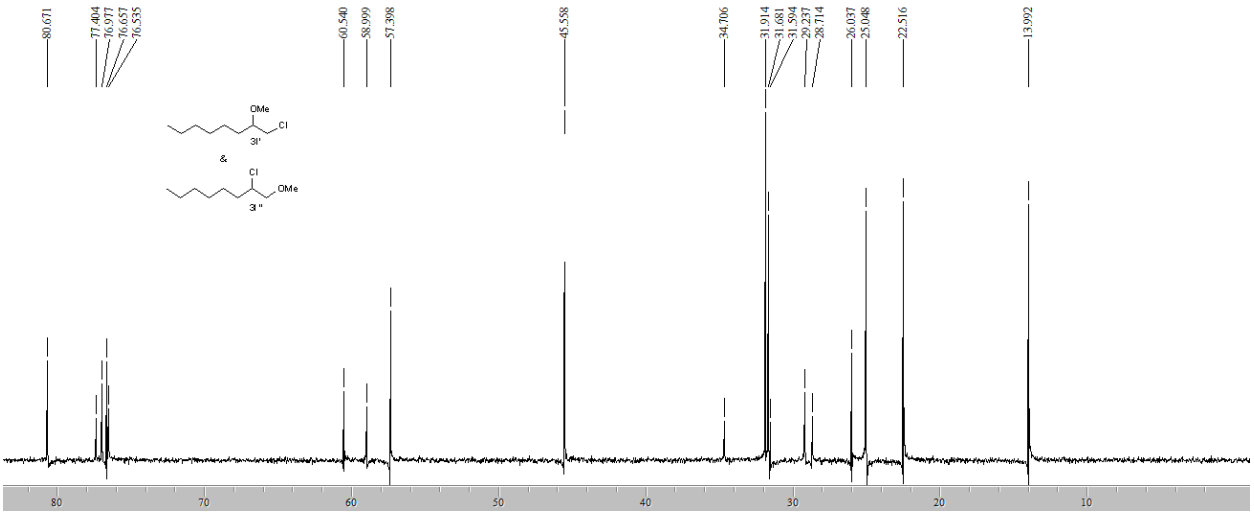
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&
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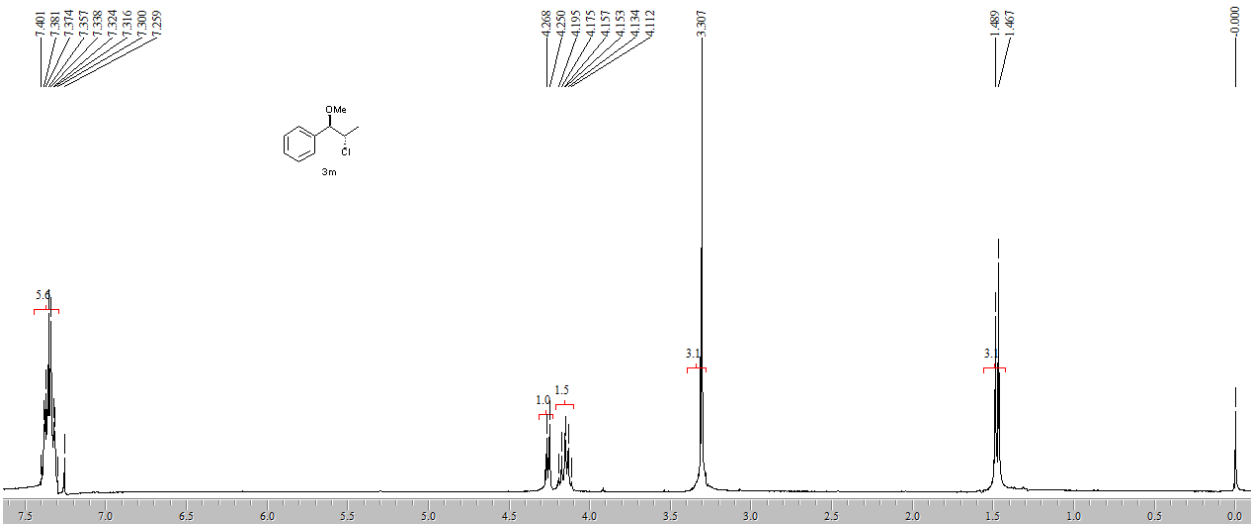
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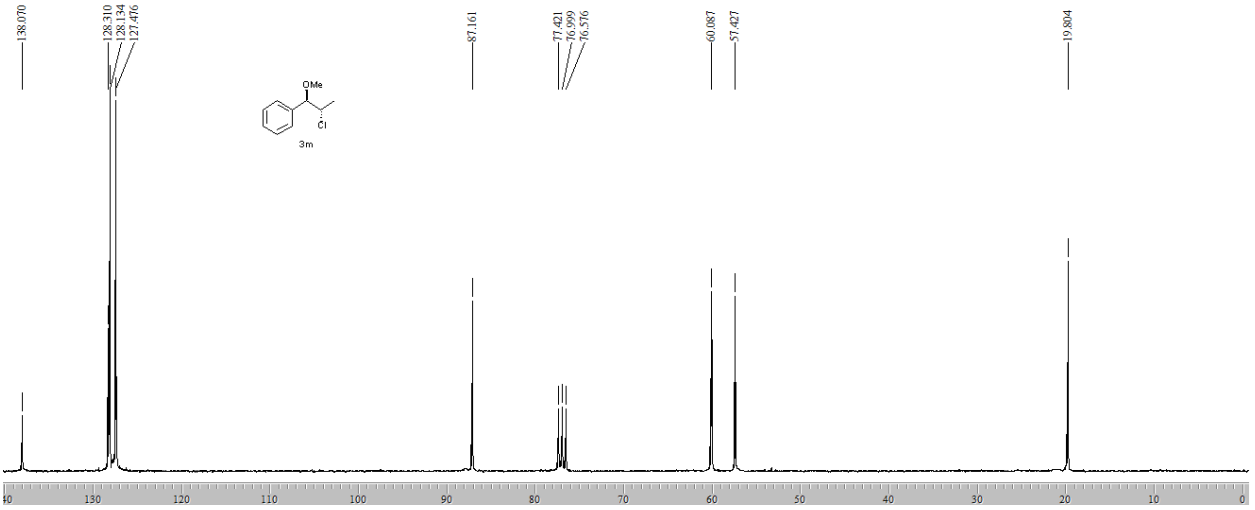
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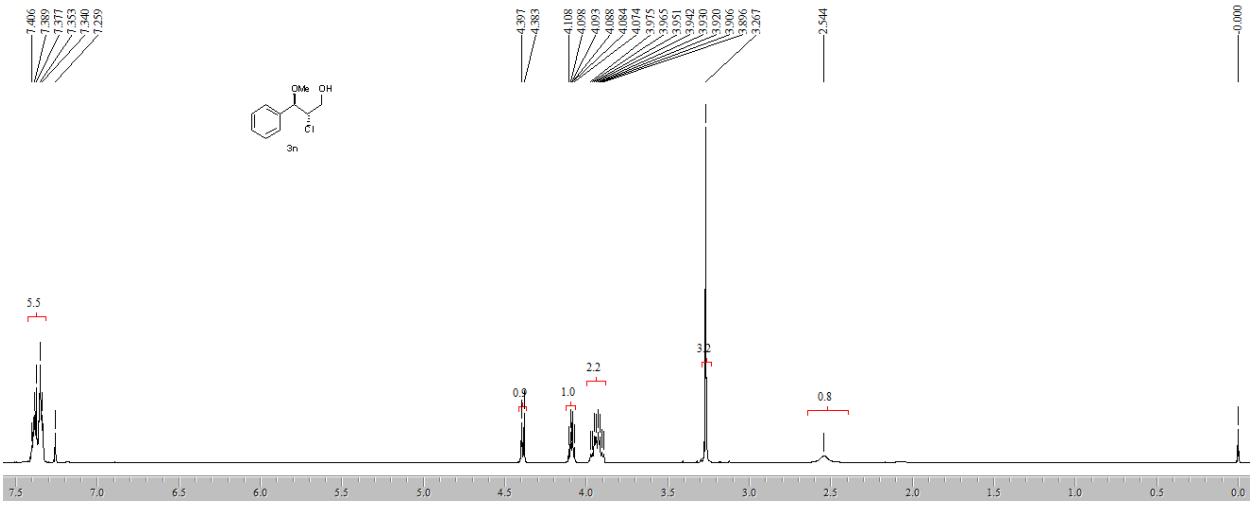
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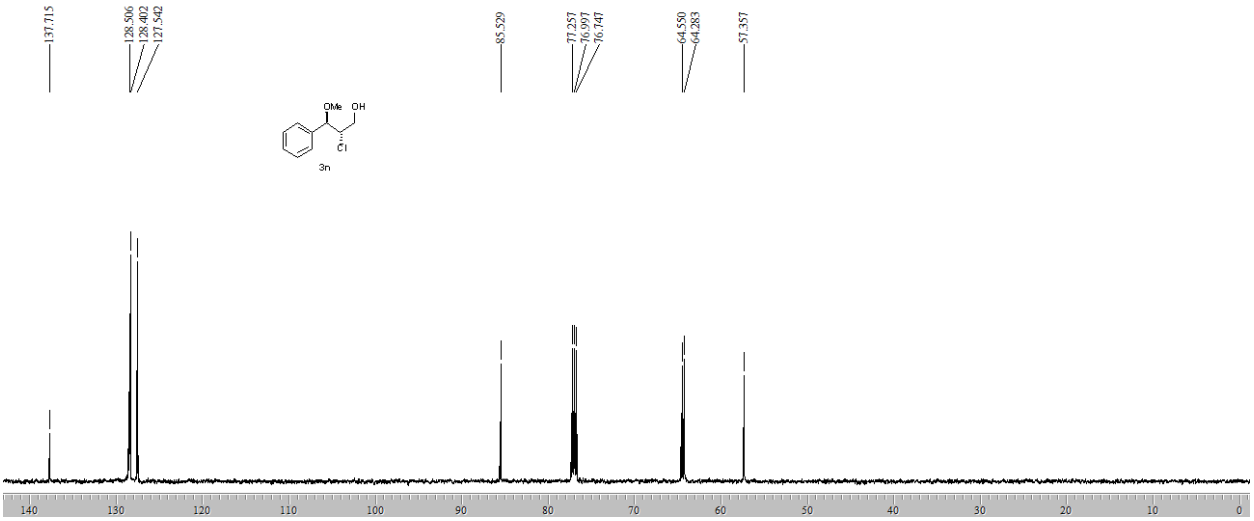
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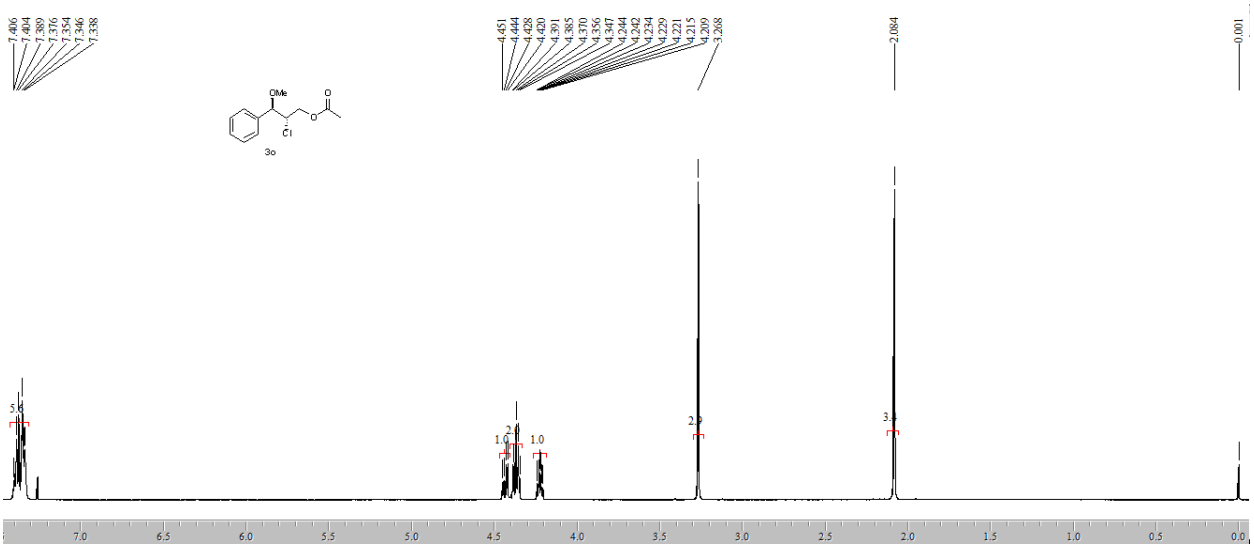
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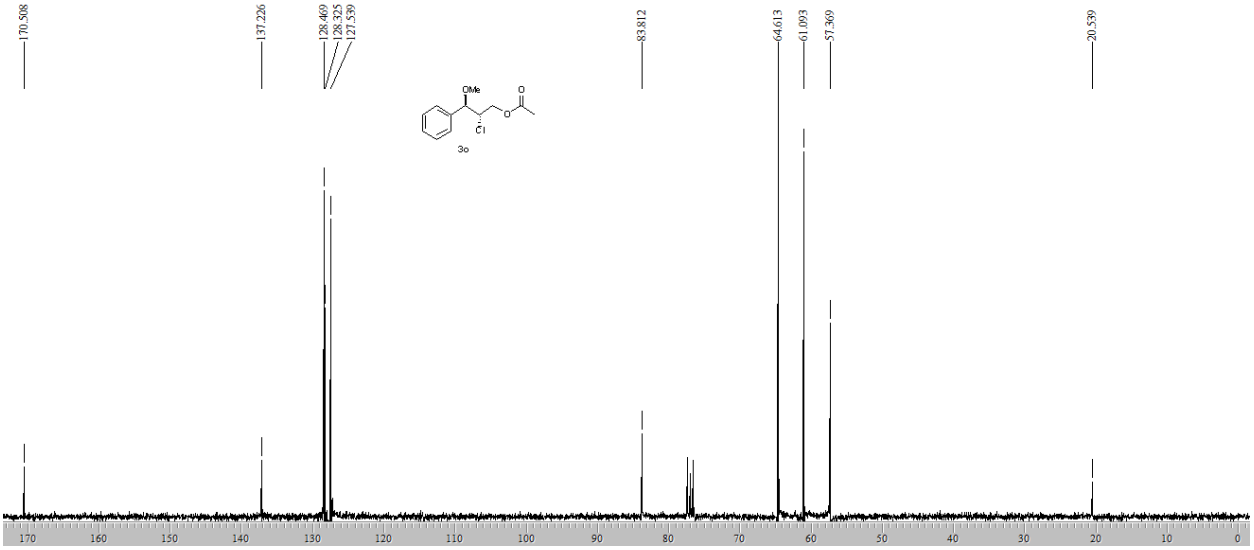
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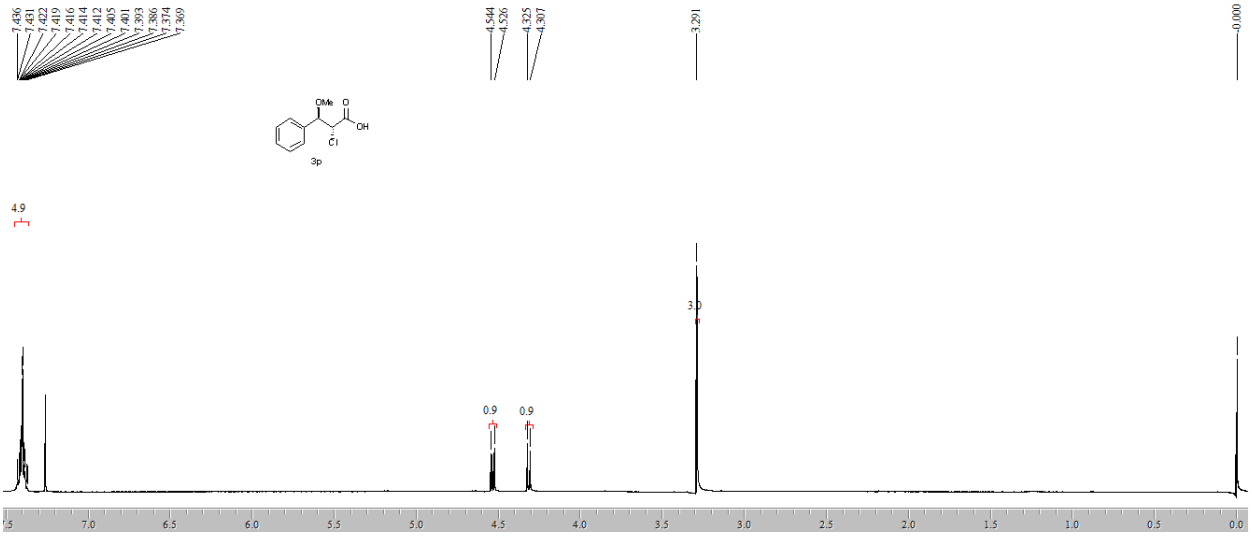
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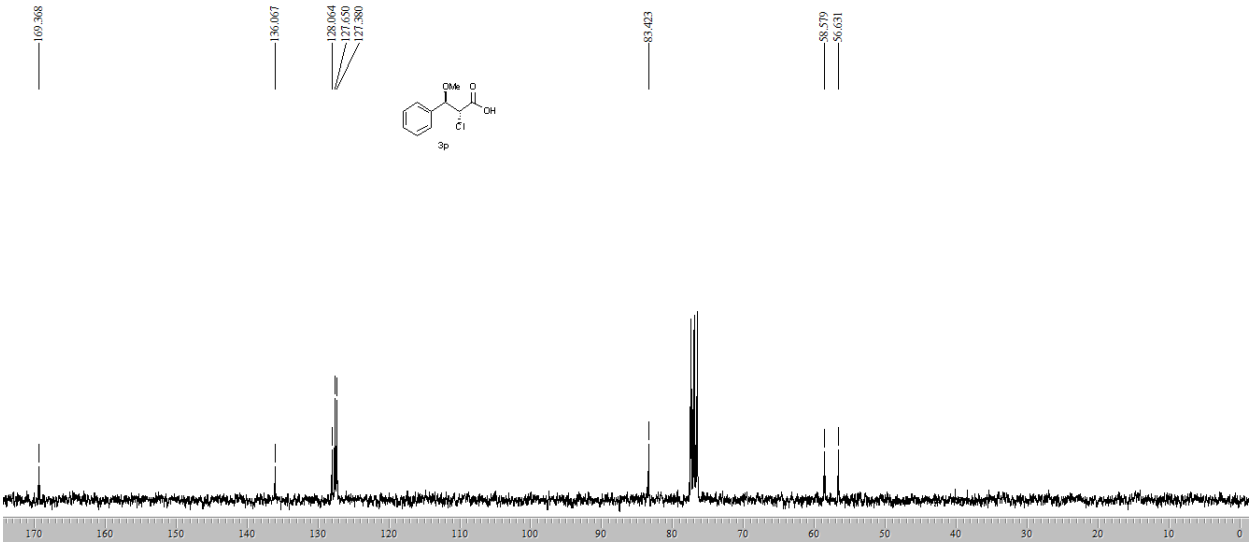
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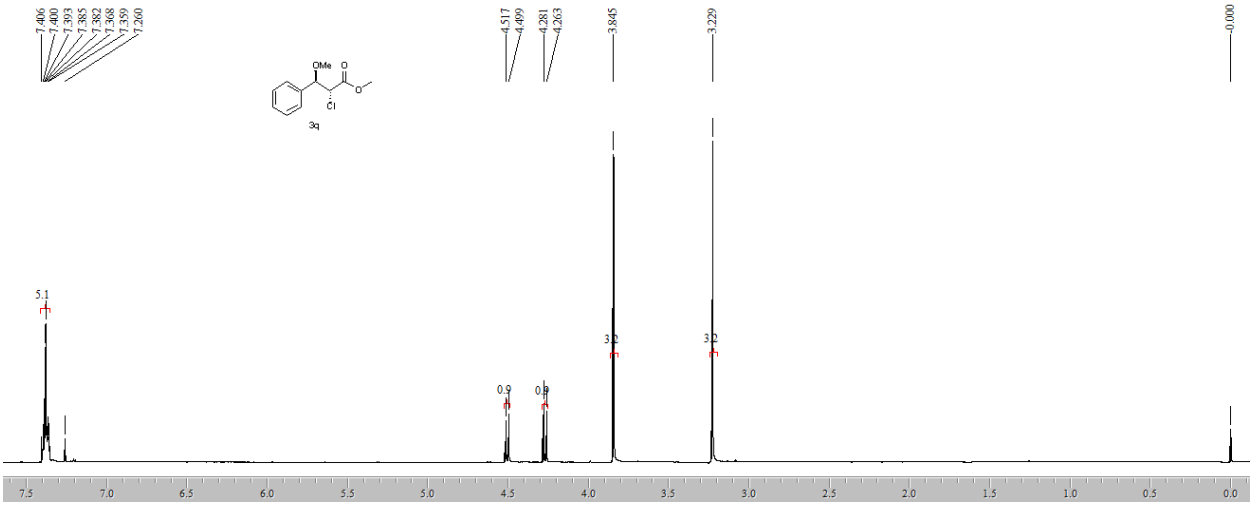
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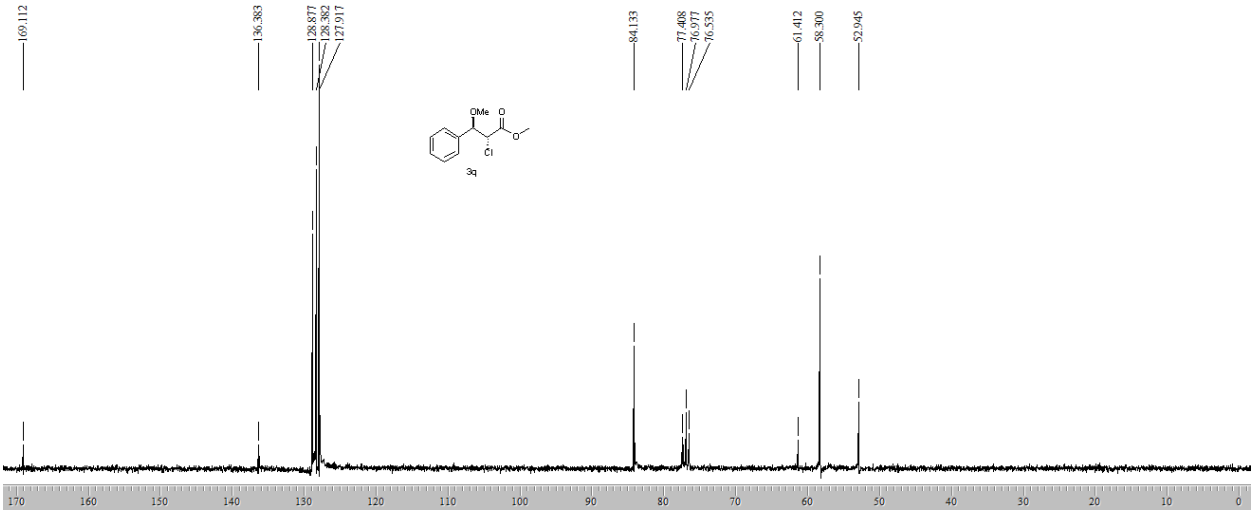
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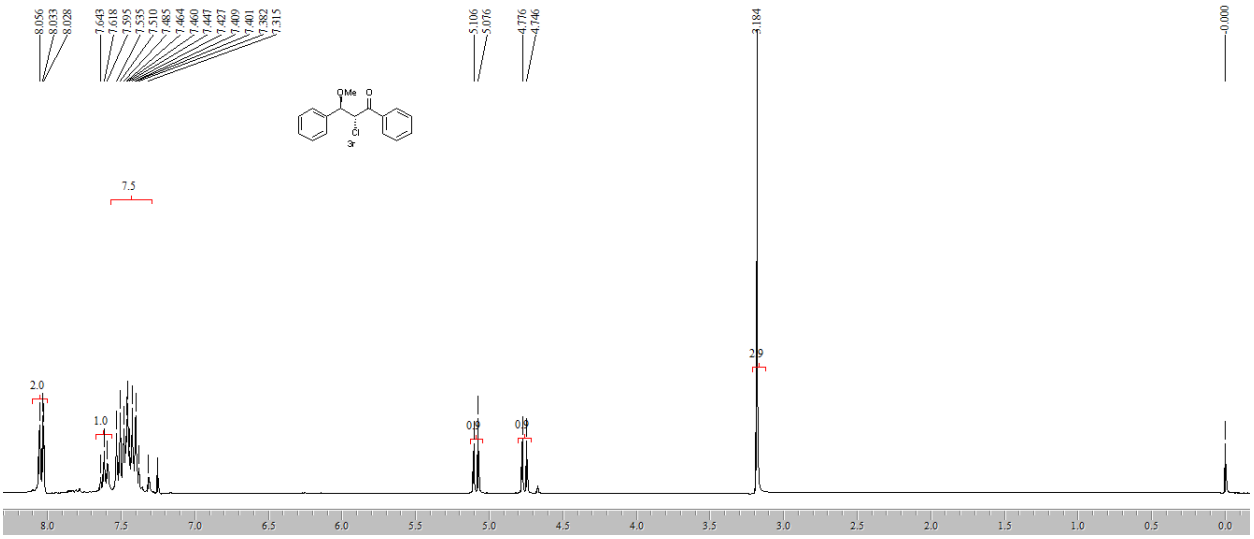
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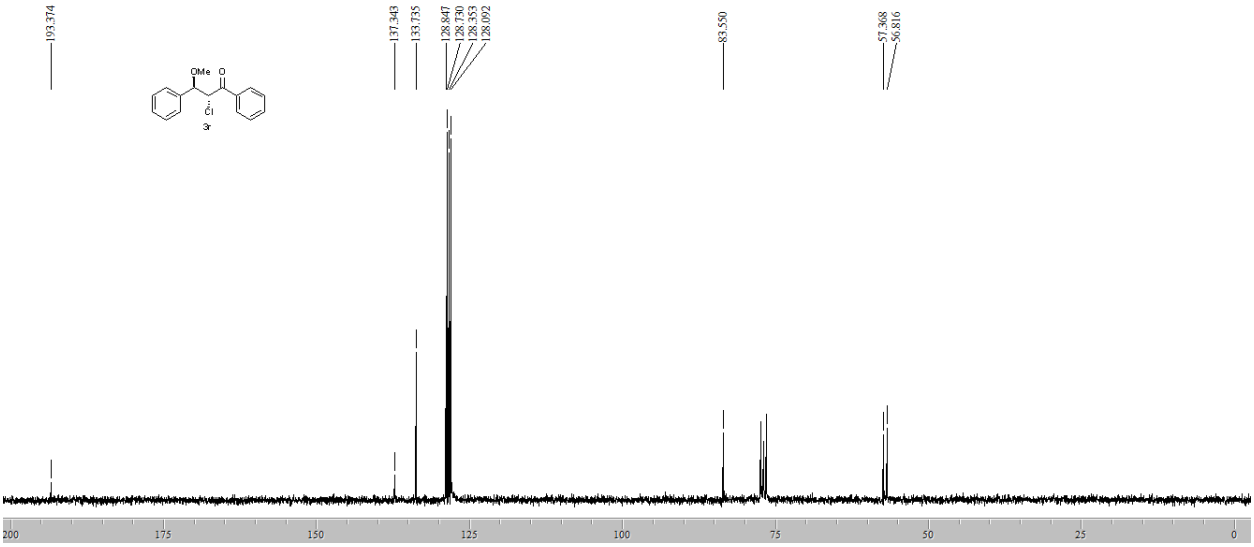
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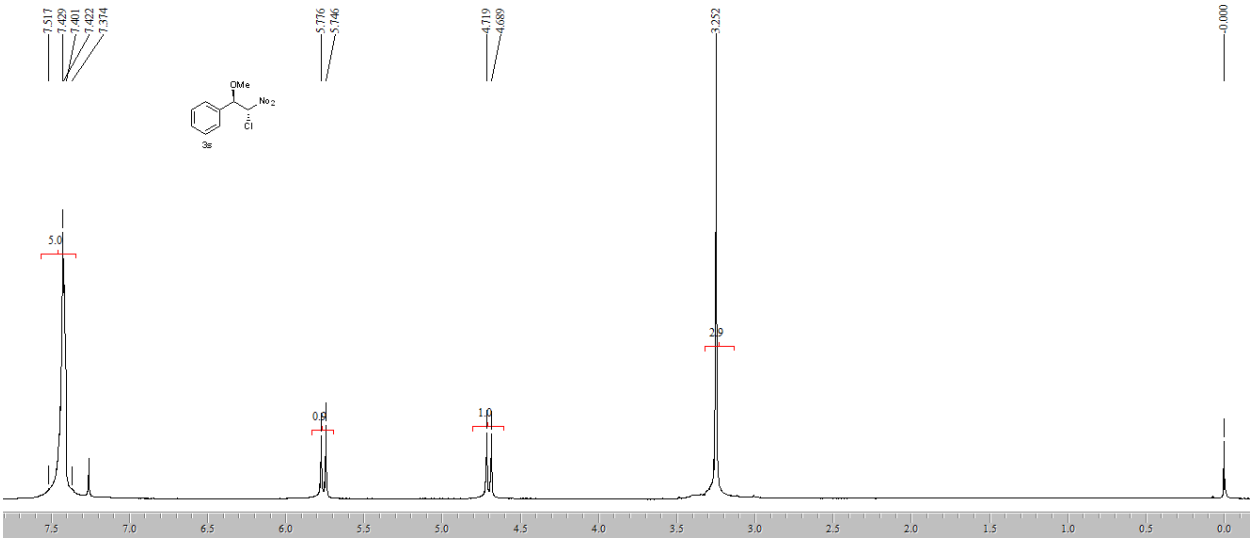
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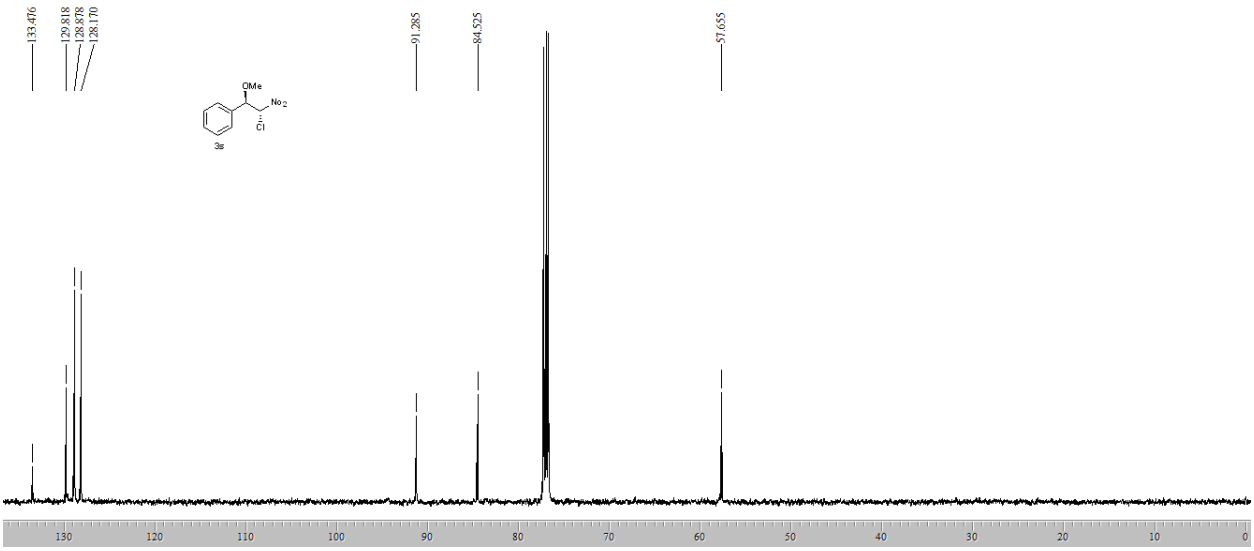
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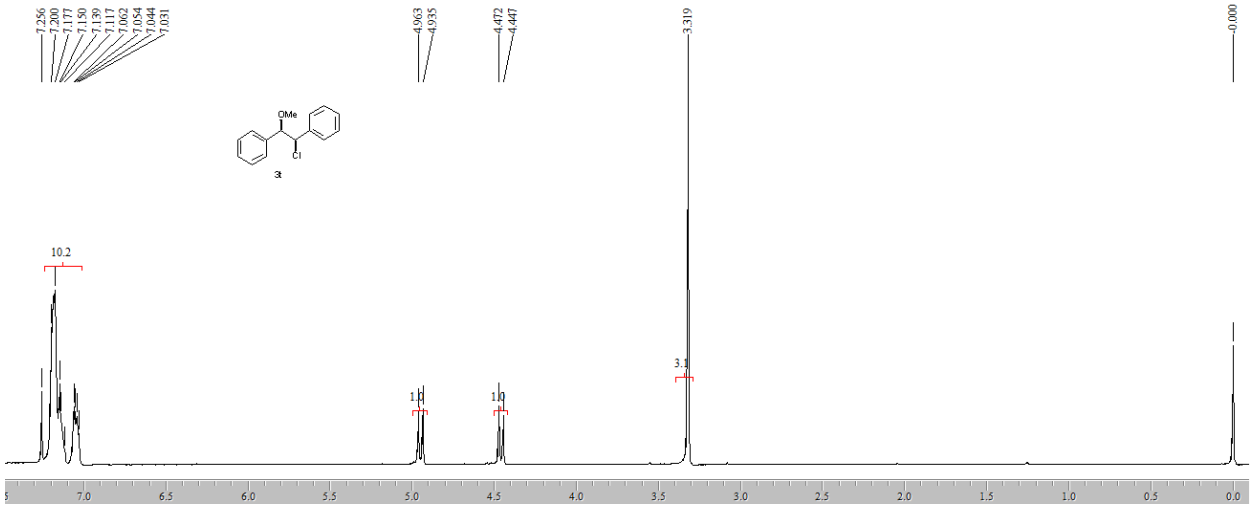
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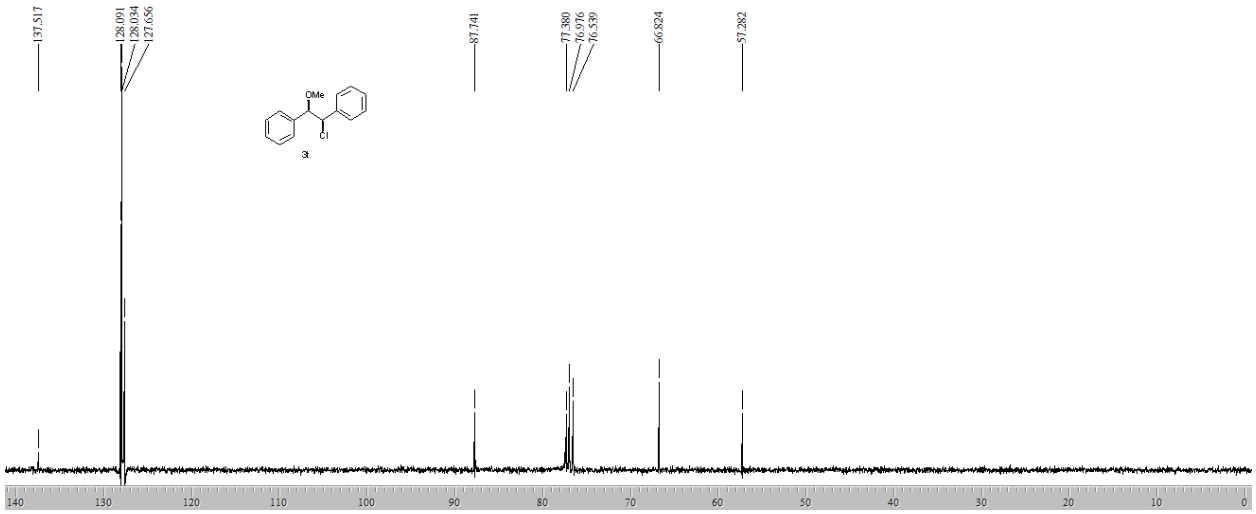
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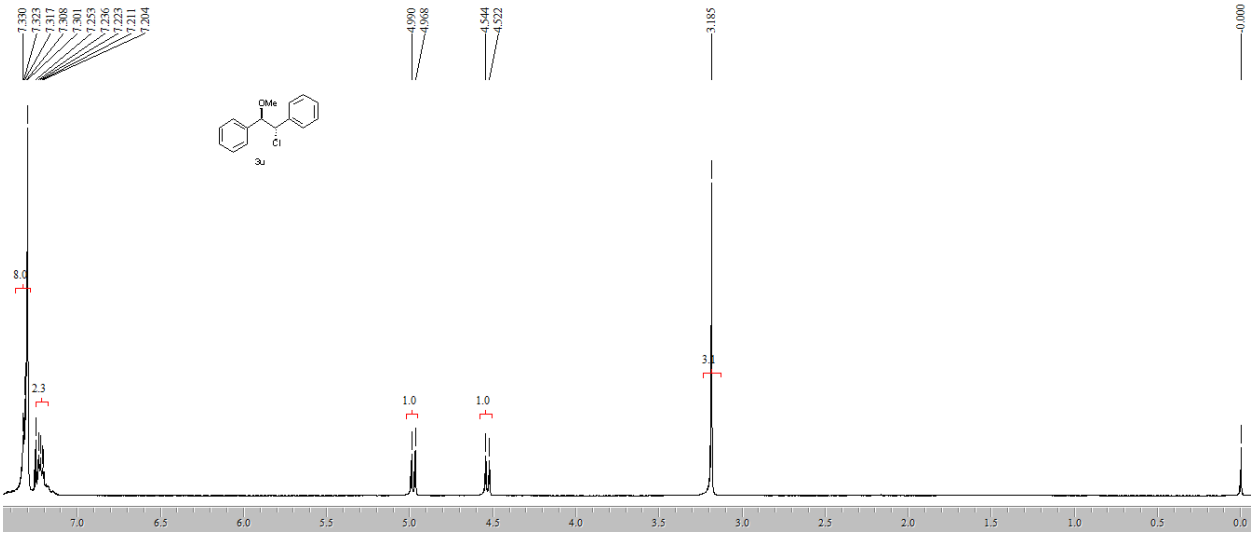
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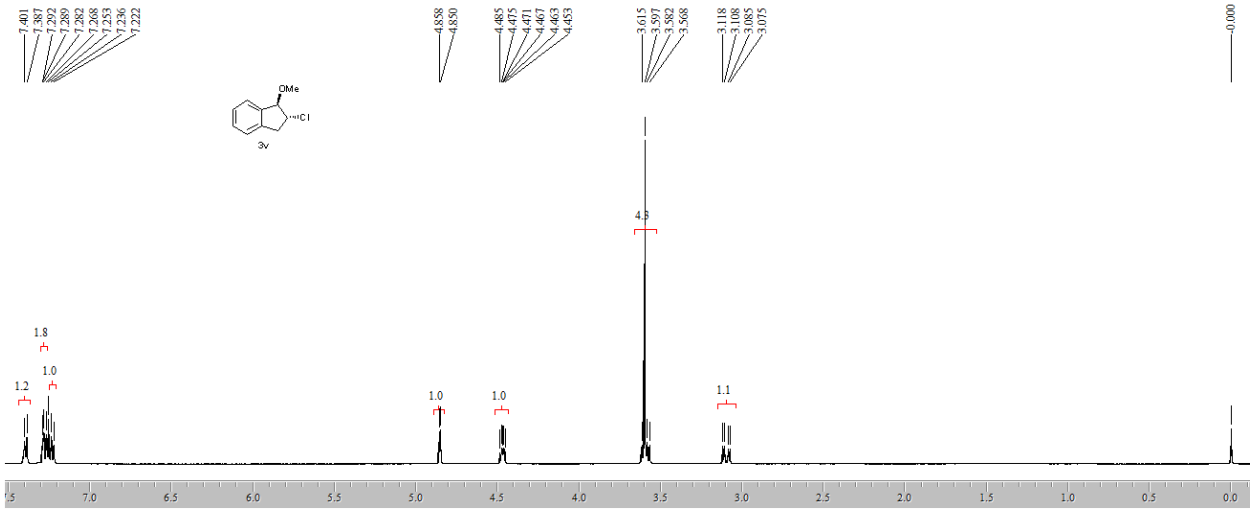
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7.289
7.282
7.268
7.253
7.236
7.222
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4.453
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Cl
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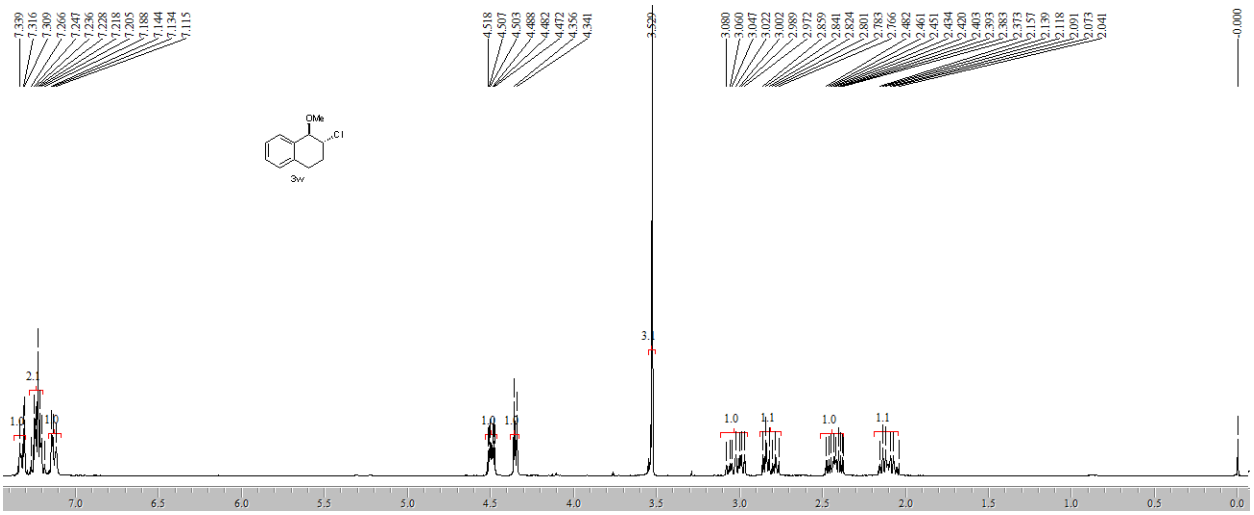
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4.507
4.503
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4.482
4.472
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4.341
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3.060
3.047
3.022
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2.766
2.482
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2.434
2.420
2.403
2.393
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2.373
2.157
2.139
2.118
2.091
2.073
2.041
-0.000
OMe
Cl
Эw
2.1
1.0
1.0
1.0
1.0
3.1
1.0
1.1
1.0
1.1
7.0
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6.0
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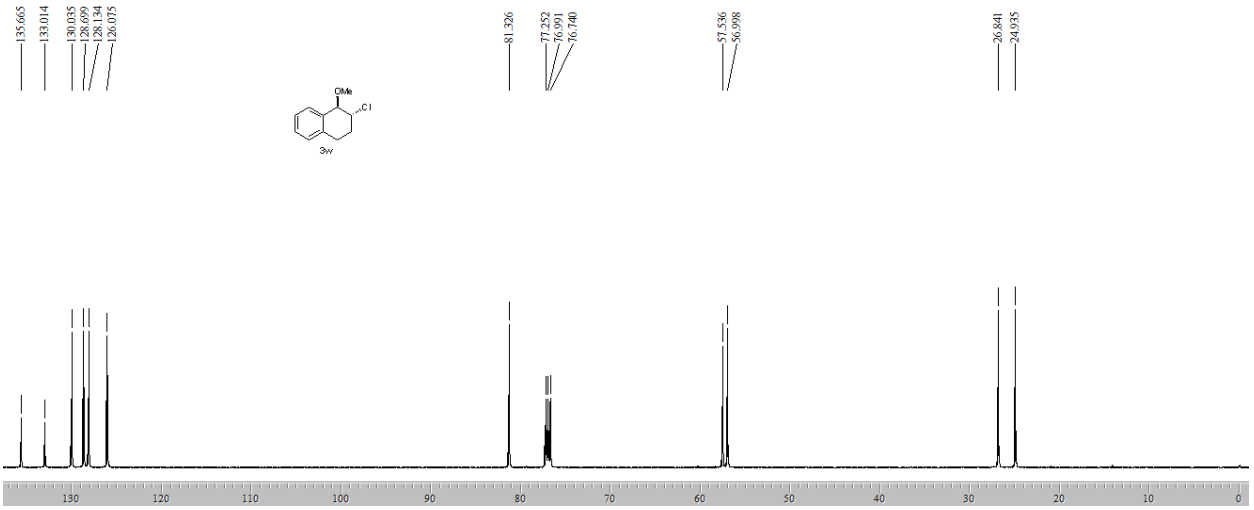
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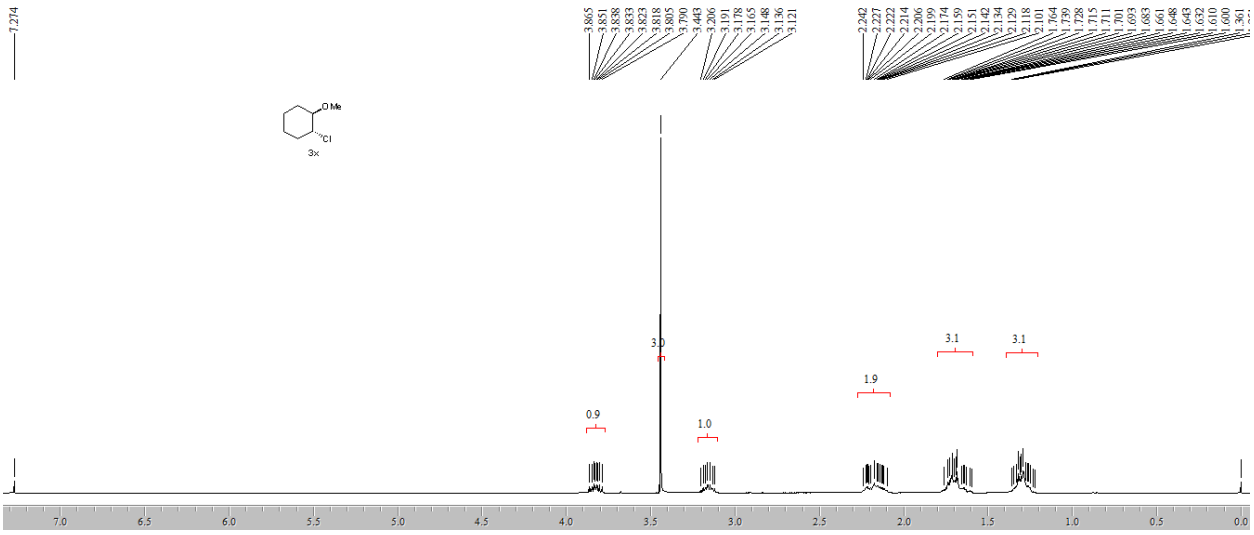
26.841
24.935
OMe
Cl
Эw
130
120
110
100
90
80
70
60
50
40
30
20
10
0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3.165
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3.136
3.121
2.242
2.227
2.222
2.214
2.206
2.199
2.174
2.159
2.151
2.142
2.134
2.129
2.118
2.101
1.764
1.739
1.728
1.715
1.711
1.701
1.693
1.683
1.661
1.648
1.643
1.632
1.610
1.600
1.361
O Me
Cl
3x
0.9
3.0
1.0
1.9
3.1
3.1
7.0
6.5
6.0
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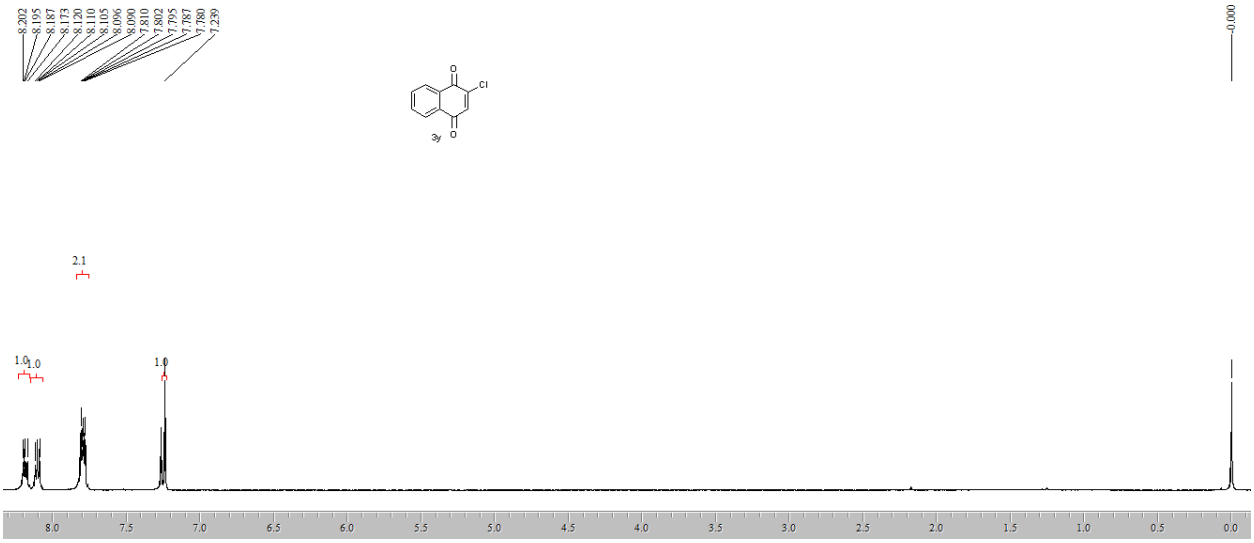
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7.780
7.239
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O
Cl
O
3y
2.1
1.0
1.0
1.0
8.0
7.5
7.0
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6.0
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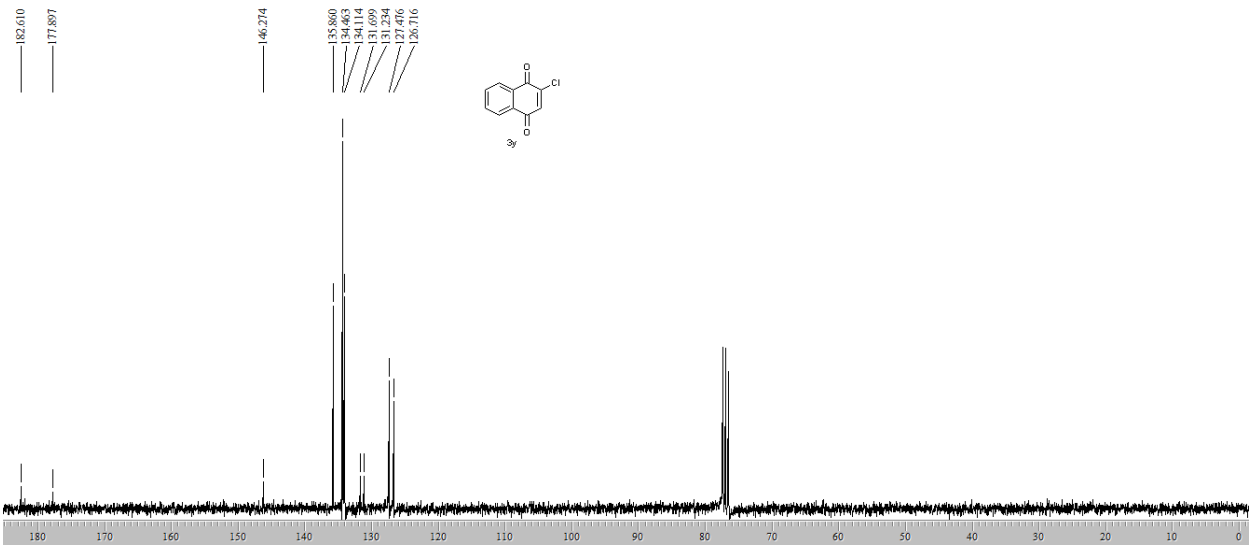
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Эу
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

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