Advanced Synthesis & Catalysis

Supporting Information

SUPPORTING INFORMATION

A Practical Procedure for Iron Catalyzed Cross Coupling Reactions of Sterically Hindered Aryl-Grignard Reagents with Primary Alkyl Halides

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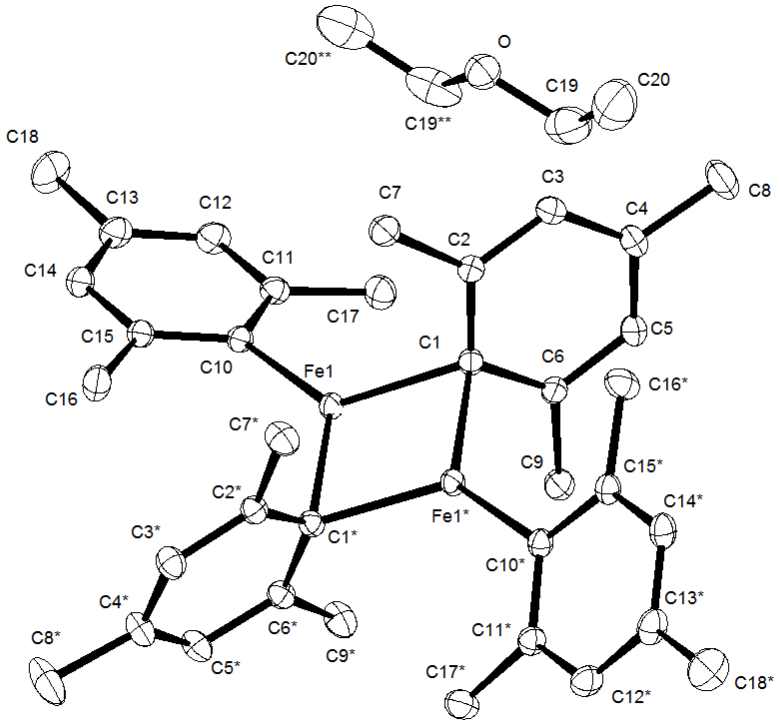

Figure S1. Structure of complex **6** in the solid state.

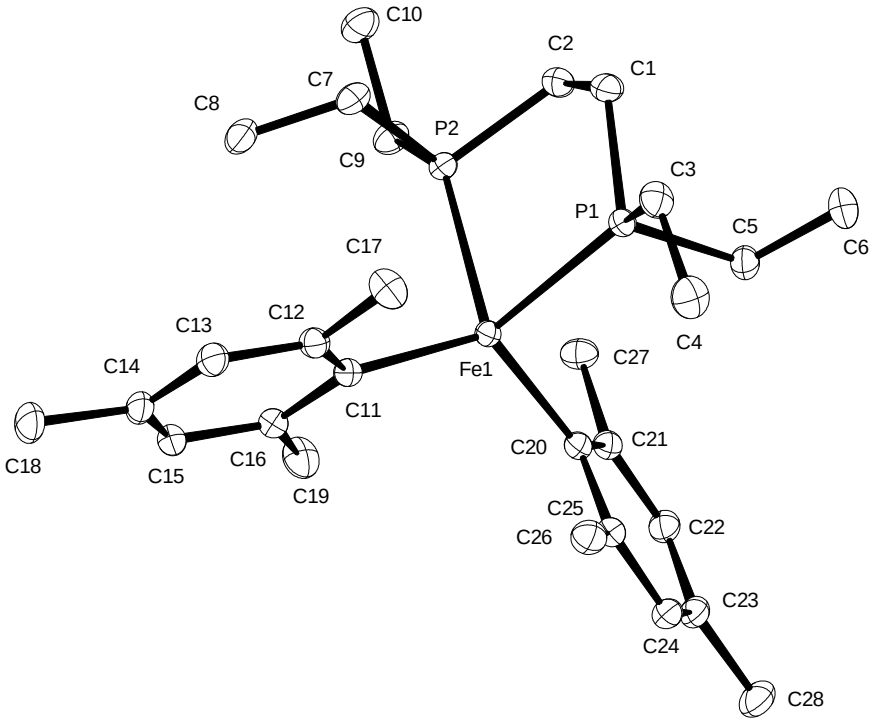

Figure S2. Structure of complex **10** in the solid state.

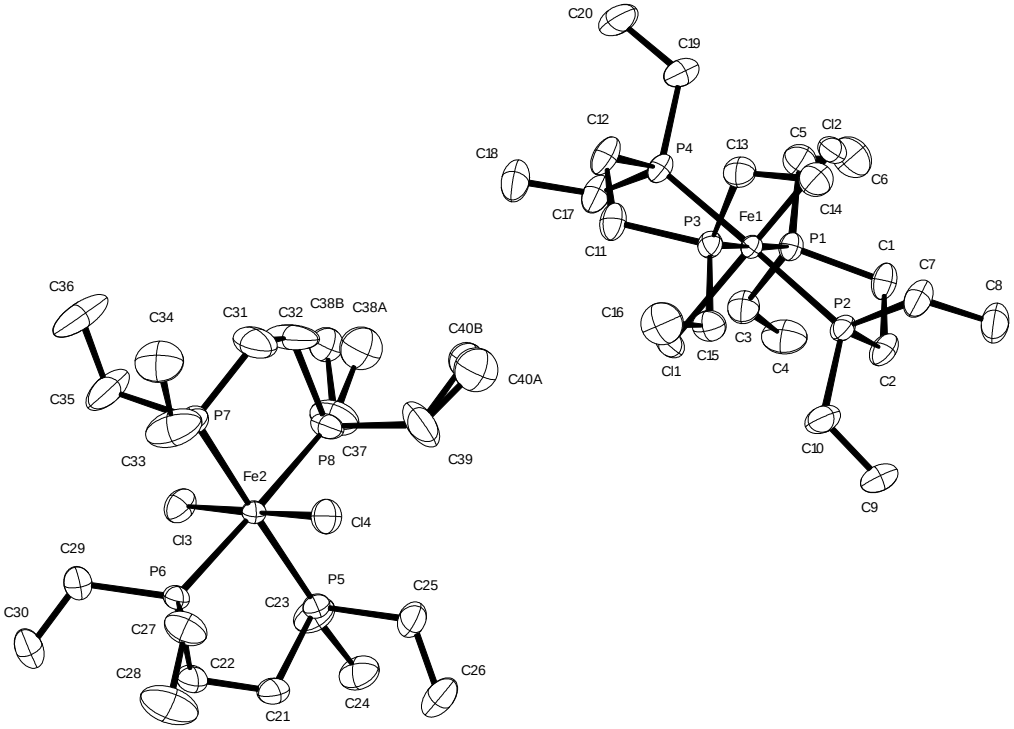

Figure S3. Structure of complex **17** in the solid state.

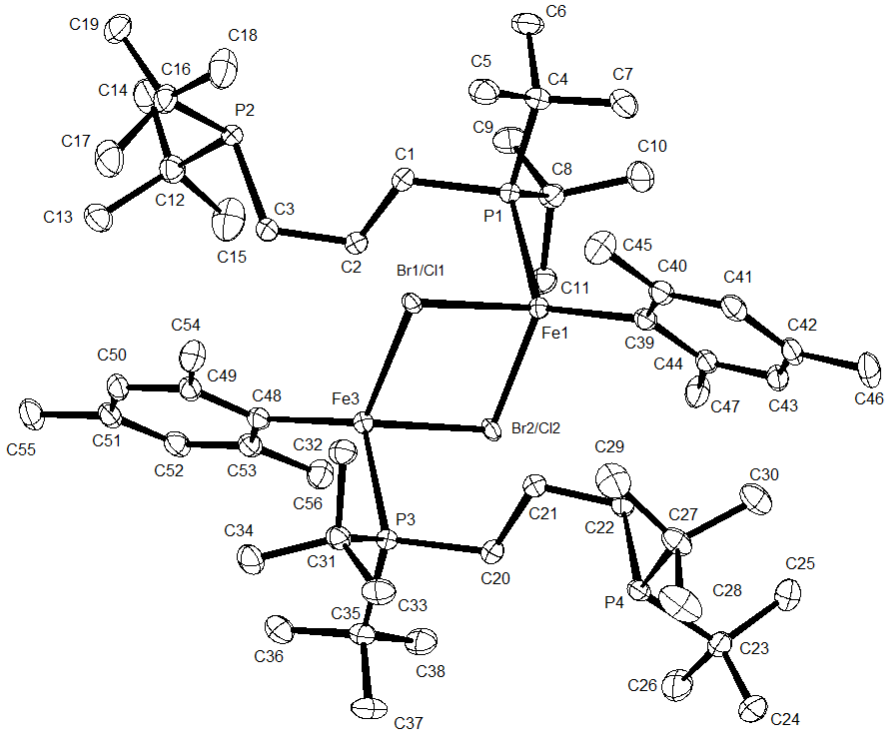

Figure S4. Structure of complex **19** in the solid state.

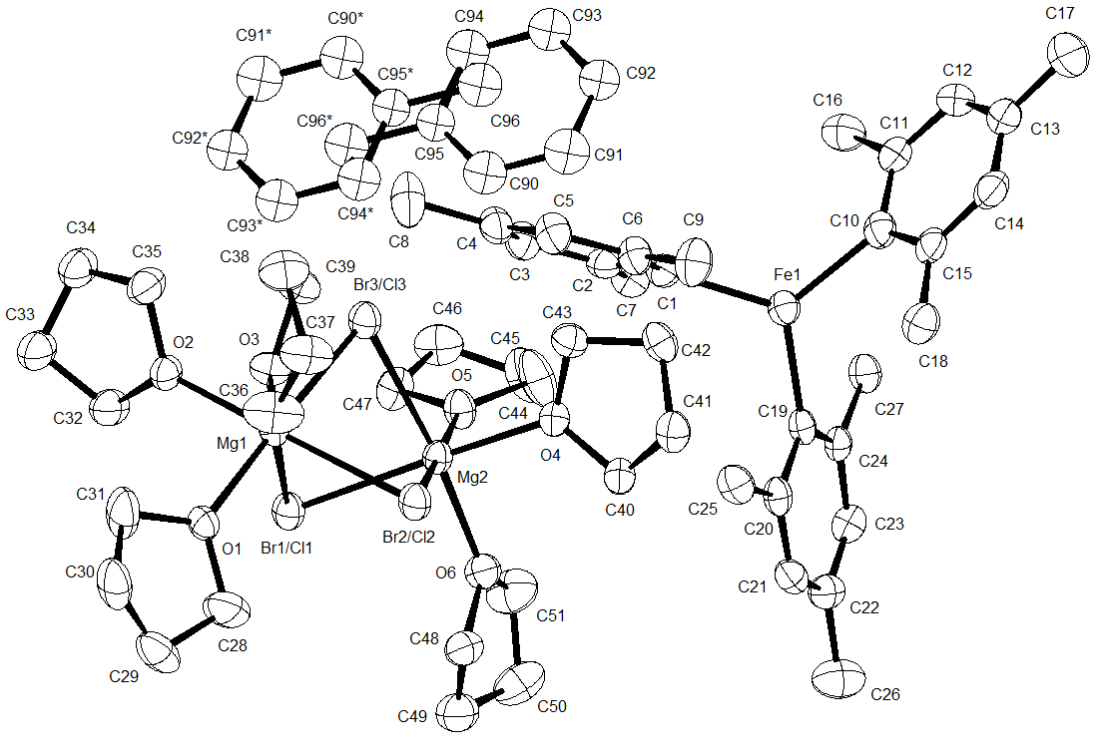

Figure S5. Structure of the ate-complex complex **21** in the solid state.

CCDC-973605 (**21**), **CCDC-973606** (**6**), **CCDC-973607** (**10**), **CCDC-973608** (**17**), and **CCDC-973609** (**19**) contain the supplementary crystallographic data for this paper. This information can be obtained free of charge from The Cambridge Crystallographic Data Centre via

www.ccdc.cam.ac.uk/data_request/cif.

X-ray Crystal Structure Analysis of Complex 6: $C_{40}H_{54}Fe_2O$, M_r = 662.53 g · mol^{-1}, red plate, crystal size 0.254 x 0.153 x 0.060 mm, monoclinic, space group *C2/c*, a = 18.8361(7) Å, b = 18.9509(6) Å, c = 13.1715(6) Å, β = 128.927(6)°, V = 3657.7(2) Å^3, T = 100 K, Z = 4, D_{calc} = 1.203 g·cm^3, λ = 0.71073 Å, μ(*Mo-K*$_\alpha$) = 0.676 mm^{-1}, Empirical absorption correction (T_{min} = 0.71, T_{max} = 0.91), Bruker-AXS Enraf-Nonius KappaCCD diffractometer, 2.78 < θ < 33.10°, 44987 measured reflections, 6945 independent reflections, 5942 reflections with $I > 2\sigma(I)$, Structure solved by direct methods and refined by full-matrix least-squares against F^2 to R_1 = 0.030 [$I > 2\sigma(I)$], wR_2 = 0.076, 202 parameters, H atoms riding, S = 1.089, residual electron density 0.4 / -0.4 e Å^{-3}.

X-ray Crystal Structure Analysis of Complex 10: $C_{28}H_{46}FeP_2$, M_r = 500.44 g · mol^{-1}, yellow plate, crystal size 0.480 x 0.325 x 0.140 mm, orthorombic, space group *Pbca*, a = 17.901(2) Å, b = 15.2534(17) Å, c = 20.199(2) Å, V = 5515.5(11) Å^3, T = 100 K, Z = 8, D_{calc} = 1.205 g·cm^3, λ = 0.71073 Å, μ(*Mo-K*$_\alpha$) = 3.441 mm^{-1}, Empirical absorption correction (T_{min} = 0.74, T_{max} = 0.91), Bruker-AXS Enraf-Nonius KappaCCD diffractometer, 2.02 < θ < 36.32°, 203791 measured reflections, 13326 independent reflections, 11800 reflections with $I > 2\sigma(I)$, Structure solved by direct methods and refined by full-matrix least-squares against F^2 to R_1 = 0.025 [$I > 2\sigma(I)$], wR_2 = 0.077, 290 parameters, H

atoms riding, S = 1.076, absolute structure parameter = 0.026(7), residual electron density 0.6 / -0.3 e Å^{-3}.

X-ray Crystal Structure Analysis of Complex 17: $C_{40}H_{96}Cl_4Fe_2P_8$, M_r = 1078.43 g · mol^{-1}, green plate, crystal size 0.21 x 0.13 x 0.08 mm, triclinic, space group *P1*, a = 10.2085(12) Å, b = 16.3797(19) Å, c = 16.692(2) Å, α = 83.460(2)°, β = 88.817(2)°, γ = 78.306(2)°, V = 2715.4(6) Å^3, T = 200 K, Z = 2, D_{calc} = 1.319 g·cm^3, λ = 0.71073 Å, μ(*Mo-K*$_\alpha$) = 0.994 mm^{-1}, Gaussian absorption correction (T_{min} = 0.80, T_{max} = 0.92), Bruker-AXS Smart APEX-II diffractometer, 1.23 < θ < 31.00°, 80063 measured reflections, 17311 independent reflections, 14555 reflections with $I > 2\sigma(I)$, Structure solved by direct methods and refined by full-matrix least-squares against F^2 to R_1 = 0.040 [$I > 2\sigma(I)$], wR_2 = 0.121, 501 parameters, H atoms riding, S = 1.027, residual electron density 1.4 / -1.3 e Å^{-3}.

X-ray Crystal Structure Analysis of Complex 19: $C_{56}H_{106}Fe_4P_4$, M_r = 563.34 g · mol^{-1}, yellow block, crystal size 0.13 x 0.11 x 0.09 mm, triclinic, space group *P1*, a = 13.5165(17) Å, b = 15.0944(19) Å, c = 17.563(2) Å, α = 84.612(2)°, β = 68.541(2)°, γ = 71.096(2)°, V = 3153.8(7) Å^3, T = 100 K, Z = 2, D_{calc} = 1.186 g·cm^3, λ = 0.71073 Å, μ(*Mo-K*$_\alpha$) = 1.034 mm^{-1}, Gaussian absorption correction (T_{min} = 0.92, T_{max} = 0.95), Bruker-AXS Smart APEX-II diffractometer, 1.43 < θ < 34.34°, 109475 measured reflections, 26318 independent reflections, 18331reflections with $I > 2\sigma(I)$, Structure solved by direct methods and refined by full-matrix least-squares against F^2 to R_1 = 0.037 [$I > 2\sigma(I)$], wR_2 = 0.091, 607 parameters, H atoms riding, S = 1.025, residual electron density 0.6 / -0.8 e Å^{-3}.

X-ray Crystal Structure Analysis of Complex 21: $C_{51}H_{81}Br_{1.29}Cl_{1.71}FeMg_2O_6 \cdot 0.5\ C_7H_8$, M_r = 1104.40 g · mol^{-1}, colorless plate, crystal size 0.239 x 0.157 x 0.062 mm, monoclinic, space group $P2_1/n$, a = 13.546(2) Å, b = 15.160(3) Å, c = 28.029(5) Å, β = 96.233(3)°, V = 5721.8(18) Å^3, T = 100 K, Z = 4, D_{calc} = 1.282 g·cm^3, λ = 0.71073 Å, μ(*Mo-K*$_\alpha$) = 1.311 mm^{-1}, Empirical absorption correction (T_{min} = 0.85, T_{max} = 0.96), Bruker-AXS Enraf-Nonius KappaCCD diffractometer, 1.46 < θ < 27.13°, 122880 measured reflections, 12616 independent reflections, 7765 reflections with $I > 2\sigma(I)$, Structure solved by direct methods and refined by full-matrix least-squares against F^2 to R_1 = 0.060 [$I > 2\sigma(I)$], wR_2 = 0.166, 593 parameters, H atoms riding, S = 1.023, residual electron density 2.8 / -2.2 e Å^{-3}.

General. All reactions were carried out in carefully flame-dried glassware under Argon. All solvents were dried and purified by distillation over the indicated drying agents and were transferred under Argon: THF (Mg-anthracene), diethyl ether (Mg-anthracene), dichloromethane (CaH_2), acetonitrile (CaH_2), triethylamine (CaH_2), hexane (Na/K), toluene (Na/K). HOAc and MeOH were used as received. Flash chromatography: Merck silica gel 60 (230-400 mesh). IR: Nicolet FT-7199 spectrometer, wavenumbers in cm−1. MS (EI): Finnigan MAT 8200, MS (CI): Finnigan MAT 95, MS (ESI) ESQ 3000; accurate mass determinations: Bruker APEX III FT-MS (7 T magnet). NMR: Spectra were recorded on a Bruker DPX 300, AV 400 or AV 500 spectrometer in the solvents indicated; ^{1}H and ^{13}C chemical shifts (δ) are given in ppm relative to TMS, coupling constants (J) in Hz. The solvent signals were used as references ($CDCl_3$: δ_H = 7.24 ppm, δ_C = 77.0 ppm) and the chemical shifts converted to the TMS scale. Elemental analyses: H. Kolbe, Mülheim/Ruhr. Unless stated otherwise, all commercially available compounds (Acros, Sigma-Aldrich, Fluka, Alfa Aesar, Lancaster, Strem) were used as received.

The following complexes were prepared form Fe_2Mes_4 (**6**) according to the cited literature procedures: [Fe(tmeda)(mes)$_2$] (**1**),[1] [$FeCl_2$(3,5-*t*Bu-SciOPP)] (**3**),[2] [Fe(phen)(mes)$_2$] (**7**),[1b] [Fe(2,2'-bipyridin)(mes)$_2$] (**8**),[4] [Fe(2,6-dimethylpyridine)(mes)$_2$] (**9**),[3] [Fe(depe)(mes)$_2$] (**10**),[4] [Fe(dppp)(mes)$_2$] (**11**),[4] *trans*-[Fe(^{n}Bu$_3$P)$_2$(mes)$_2$] (**12**),[4] and *trans*-[Fe[(EtO)$_3$P]$_2$(mes)$_2$] (**13**),[4] and *trans*-[Fe(depe)$_2$Cl$_2$] (**17**); [5] complex [Fe(3,5-*t*Bu-SciOPP)(mes)$_2$] (**14**) was prepared analogously; since no crystals suitable for X-ray diffraction could be grown so far, the assignment remains tentative.

Preparation of [$FeCl_2 \cdot (THF)_n$] (5).[6] A Soxhlet apparatus was charged with anhydrous $FeCl_2$ (10.0 g, 78.7 mmol). The system was degassed in vacuum and refilled with argon. Anhydrous THF (160 mL) was added under argon atmosphere. The mixture was heated to reflux for three days to furnish a suspension of a pale-pink colored solid in a yellow solution. The solid was separated by filtration and dried under a stream of argon. The product was obtained as a pale-pink colored solid (17.3 g). Elemental analysis of different samples prepared according to this protocol showed a varying THF content in the range of $\approx 1.2 - 1.5$ mol per mol of $FeCl_2$.

Preparation of Fe_2Mes_4 (6).[7] Freshly prepared [$FeCl_2 \cdot 1.5$ THF] (10.82 g, 46.0 mmol) was suspended in a mixture of anhydrous THF (200 mL) and 1,4-dioxane (40 mL). The suspension was cooled to −30 °C before MesMgBr (0.88 M in THF, 104.5 mL, 92.0 mmol) was slowly introduced to the stirred suspension (with occasional shaking). The temperature was raised to room temperature and stirring continued for 2 h. For work up, the magnesium salts were filtered off under Ar and the filtrate was evaporated to dryness. The resulting residue was dried under vacuum before Et_2O (200 mL) was added. A second filtration was performed to remove traces of remaining magnesium salts. The

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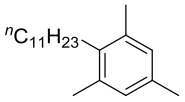
solution was slowly concentrated to a volume of ca. 40 mL, from which the product crystallized. The precipitate was filtered off and dried in vacuo to furnish complex **6** in form of a brick-red solid (12.04 g, 89%). The structure and constitution was confirmed by X-ray diffraction (see above).

Preparation of Fe(depe)Mes_2 (10) from Fe(depe)$_2Cl_2$ (17). MesMgBr solution (1 M in THF, 0.344 mL, 0.344 mmol) was slowly added via syringe to a strirred solution of Fe(depe)$_2Cl_2$ (**17**) (84.2 mg, 0.156 mmol) in THF (3.0 mL). Once the addition was complete, the mixture was stirred at 70 °C for 30 min before it was allowed to cool to ambient temperature. The solvent was removed under vacuum and the residue was dissolved in cold Et_2O (–30 °C). The precipitated magnesium salts were filtered off and the yellow filtrate was evaporated in vacuo. A small amount of cold pentane (about 0.5 mL, –30 °C) was added and the mixture was stirred until a yellow suspension had formed. The solid was collected by filtration and dried under vacuum to give complex **10** as a yellow solid (63.2 mg, 81%). The structure was confirmed by X-ray diffraction (see above).

Complex 19. MesMgBr (1 M in THF, 0.416 mL, 0.416 mmol) was added via syringe to a solution of [(1,3-bis(di-*tert*-butylphosphino)propane)$FeCl_2$] (**18**, R = *t*Bu) (191.1 mg, 0.416 mmol) in THF (5.0 mL). The mixture was stirred at ambient temperature for 3 h to form a yellow-brown solution. The solvent was removed in vacuo and the residue was suspended in cold Et_2O (–30 °C). The precipitated magnesium salts were filtered off and washed with cold Et_2O (–30 °C). The combined filtrates were evaporated to afford the title complex as a yellow-orange solid (118.2 mg, 53%). The structure was confirmed by X-ray diffraction (see above).

Representative Procedure for the Stoichiometric Reaction of 1-Iodoundecane with Various Iron Complexes: Preparation of 1,3,5-Trimethyl-2-undecylbenzene (16). 1-Iodoundecane (44.6 mg, 0.158 mmol) was added to a stirred solution of [Fe(depe)Mes_2] (**10**) (102.9 mg, 0.206 mmol) in THF (4.0 mL). The mixture was then stirred at 70 °C until complete conversion of the starting material was reached (the reaction was monitored by GC-MS; the samples were quenched with MeOD and then diluted by MeO^{t}Bu). For work up, the mixture was allowed to reach ambient temperature, the reaction was quenched with water (20 mL) and the organic phase extracted by MeO^{t}Bu (3 x 20 mL). The combined organic layers were dried over Na_2SO_4 and evaporated, and the residue purified by flash chromatography (silica, hexane) to give the title compound as a colorless liquid (39.4 mg, 91%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.02 (t, *J* = 3.6 Hz, 3H), 1.40-1.54 (m, 18H), 2.36 (s, 3H), 2.40 (s, 6H), 2.68 (t, *J* = 8.0 Hz, 2H), 6.93 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.1, 19.7, 20.8, 22.7, 29.4, 29.5, 29.6, 29.7, 30.3, 32.0, 128.9, 134.7, 135.8, 136.7. IR (film, cm^{-1}): 2921, 2853, 1614, 1578, 1484, 1466, 1376, 1205, 1117, 1029, 1012, 849, 721. MS (EI): *m*/*z* (%) 274 (31), 133 (100), 120 (3), 105 (3), 91 (2), 55 (2), 41 (4). HRMS (EI): *m*/*z*: calcd for $C_{20}H_{34}$ [*M*]: 274.26618, found: 274.26605.

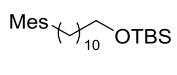


Representative Procedure for the Iron-Catalyzed Cross-Coupling of Alkyl Halides with Aryl Grignard Reagents: Preparation of 1,3,5-Trimethyl-2-undecylbenzene (16). ***Method A***: MesMgBr (1 M in THF, 0.97 mL, 0.974 mmol) was added dropwise over 1 h to a stirred solution of 1-iodoundecane (228.9 mg, 0.812 mmol) and [Fe(depe)Mes_2] (20.3 mg, 5 mol%, 0.041 mmol) in THF (4 mL) at 70°C. Once the addition was complete, stirring was continued at this temperature for 30 min before the mixture was allowed to reach ambient temperature. The reaction was quenched with water (20 mL) and the organic phase extracted by MeO^{t}Bu (3 x 20 mL). The combined organic layers were dried over Na_2SO_4 and evaporated, and the residue purified by flash chromatography (silica, hexane) to give the title compound as a colorless liquid (200.2 mg, 90%). The spectral data are identical with described above. ***Method B***: MesMgBr (1 M in THF, 0.86 mL, 0.86 mmol) was added dropwise over 1 h to a stirred

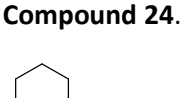
solution of 1-undecyl tosylate (207.5 mg, 0.64 mmol), $[Fe(depe)_2Cl_2]_2$ (17.2 mg, 0.032 mmol) and NaI (9.5 mg, 0.064 mmol) in THF (3 mL) at 70°C. Once the addition was complete, stirring was continued at this temperature until GC control indicated complete conversion of the substrate. The mixture was then allowed to reach ambient temperature before the reaction was quenched with water (20 mL). The organic phase was extracted by MeO^{t}Bu (3 x 20 mL), the combined organic layers were dried over Na_2SO_4 and evaporated, and the residue was purified by flash chromatography (silica, hexane) to give the title compound as a colorless liquid (175.2 mg, 99%). The spectral data are identical with those described above.

The following compounds were prepared analogously:

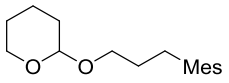
Compound 22. Colorless syrup (95%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.20 (s, 6H), 1.05 (s, 9H), 1.44-1.68 (m, 18H), 2.38 (s, 3H), 2.42 (s, 6H), 2.70 (t, J = 7.6 Hz, 2H), 3.75 (t, J = 6.1 Hz, 2H), 6.96 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = -5.3, 18.3, 19.7, 20.8, 25.8, 26.0, 29.38, 29.46, 29.54, 29.59, 29.63, 29.7, 30.3, 32.9, 63.3, 128.8, 134.6, 135.7, 136.6. IR (film, cm^{-1}): 2925, 2854, 1717, 1614, 1578, 1470, 1463, 1387, 1360, 1253, 1097, 1030, 1006, 959, 938, 833, 773, 719, 661. MS (EI): *m*/*z* (%) 404 (1), 347 (100), 199 (2), 159 (6), 133 (99), 115 (3), 89 (5), 75 (30), 59 (3), 41 (3). HRMS (ESI$^+$): *m*/*z*: calcd for $C_{26}H_{48}OSiNa$ [*M+Na*]: 427.33636, found: 427.33666.



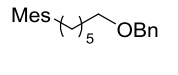
Compound 23. Colorless syrup (94%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.30 (s, 9H), 1.39-1.57 (m, 16H), 1.68-1.75 (m, 2H), 2.33 (s, 3H), 2.37 (s, 6H), 2.66 (t, J = 7.2 Hz, 2H), 4.15 (t, J = 6.0 Hz, 2H), 6.91 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 19.6, 20.7, 25.8, 27.1, 28.5, 29.1, 29.29, 29.35, 29.46, 29.52, 30.2, 38.6, 64.3, 128.7, 134.5, 135.6, 136.5, 178.4. IR (film, cm^{-1}): 2924, 2854, 1728, 1614, 1578, 1480, 1460, 1397, 1365, 1283, 1151, 1033, 978, 939, 849, 770, 722. MS (EI): *m*/*z* (%) 374 (35), 272 (6), 159 (1), 133 (100), 120 (6), 105 (2), 85 (2), 69 (1), 57 (15), 41 (5). HRMS (EI): *m*/*z*: calcd for $C_{25}H_{42}O_2$ [*M*]: 374.31865, found: 374.31848.



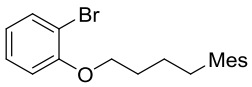
Compound 24. Colorless syrup (67%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.53-1.81 (m, 4H), 1.85-1.91 (m, 4H), 2.27 (s, 3H), 2.32 (s, 6H), 2.64-2.77 (m, 2H), 3.47-3.57 (m, 2H), 3.82-3.96 (m, 2H), 4.65 (t, J = 3.7 Hz, 1H), 6.85 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 19.6, 20.7, 25.5, 26.0, 29.4, 30.8, 62.3, 67.4, 98.8, 128.8, 134.8, 135.87, 135.95. IR (film, cm^{-1}): 2942, 2868, 1614, 1577, 1485, 1465, 1452, 1377, 1352, 1322, 1260, 1200, 1187, 1156, 1136, 1118, 1072, 1033, 1020, 988, 969, 906, 868, 849, 814, 718. MS (EI): *m*/*z* (%) 262 (14), 178 (19), 160 (24), 145 (25), 133 (53), 119 (6), 105 (6), 91 (6), 85 (100), 67 (7), 57 (7), 41 (13), 29 (7). HRMS (EI): *m*/*z*: calcd for $C_{17}H_{26}O_2$ [*M*]: 262.19310, found: 262.19328.



Compound 25. Colorless syrup (86%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.47-1.51 (m, 6H), 1.65-1.72 (m, 2H), 2.28 (s, 3H), 2.31 (s, 6H), 2.59 (t, J = 8.2 Hz, 2H), 3.51 (t, J = 6.8 Hz, 2H), 4.54 (s, 2H), 6.86 (s, 2H), 7.29-7.38 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 19.7, 20.7, 26.1, 29.3, 29.4, 29.8, 30.1, 70.4, 72.8, 127.4, 127.6, 128.3, 128.8, 134.7, 135.8, 136.5, 138.7. IR (film, cm^{-1}): 3850, 3735, 3672, 3650, 3030, 2934, 2856, 2343, 1719, 1614, 1578, 1484, 1454, 1361, 1308, 1265, 1204, 1098, 1028, 908, 850, 804, 731, 696. MS (EI): *m*/*z* (%) 310 (18), 219 (8), 201 (6), 159 (5), 133 (100), 121 (5), 99 (7), 91 (26), 81 (6), 41 (3). HRMS (EI): *m*/*z*: calcd for $C_{22}H_{30}O$ [*M*]: 310.22989, found: 310.22966.

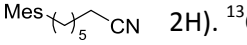


Compound 26. Colorless syrup (76%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.56-1.64 (m, 2H), 1.83-1.90 (m, 2H), 2.15 (s, 3H), 2.20 (s, 6H), 2.57 (t, J = 8.5 Hz, 2H), 3.96 (t, J = 6.0 Hz, 2H), 6.69-6.79 (m, 4H), 7.11-7.16 (m, 1H), 7.42-7.44 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 19.7, 20.8, 25.7, 29.0, 29.6, 68.7, 112.3, 113.1, 121.6, 128.4, 128.9,

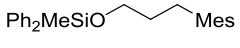


133.3, 134.9, 135.8, 136.0, 155.4. IR (film, cm^{-1}): 3063, 2945, 2873, 1613, 1587, 1573, 1482, 1466, 1442, 1377, 1292, 1277, 1247, 1161, 1125, 1051, 1030, 984, 964, 924, 881, 851, 746, 708, 691, 665. MS (EI): *m*/*z* (%) 348 (11), 346 (12), 175 (71), 159 (4), 133 (100), 117 (6), 91 (7), 77 (3), 55 (3), 41 (4). HRMS (EI): *m*/*z*: calcd for $C_{19}H_{23}OBr$ [*M*]: 346.09322, found: 346.09324.

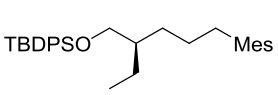
Compound 27. Colorless syrup (70%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.55-1.63 (m, 6H), 1.72-1.79 (m, 2H), 2.35 (s, 3H), 2.38 (s, 6H), 2.40 (t, *J* = 7.0 Hz, 2H), 2.68 (t, *J* = 7.0 Hz, 2H), 6.92 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 16.8, 19.5, 20.6, 25.2, 28.4, 28.9, 29.0, 29.1, 119.6, 128.7, 134.6, 135.5, 136.0. IR (film, cm^{-1}): 2935, 2857, 2246, 1733, 1613, 1578, 1484, 1459, 1425, 1377, 1206, 1141, 1078, 1029, 1013, 851, 722. MS (EI): *m*/*z* (%) 229 (17), 133 (100), 117 (3), 105 (3), 91 (4), 77 (2), 41 (4). HRMS (EI): *m*/*z*: calcd for $C_{16}H_{23}N$ [*M*]: 229.18292, found: 229.18305.



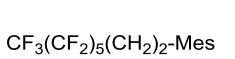
Compound 28. Colorless syrup (59%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.69 (s, 3H), 1.70-1.77 (m, 2H), 2.27 (s, 3H), 2.28 (s, 6H), 2.65-2.69 (m, 2H), 3.83 (t, *J* = 6.3 Hz, 2H), 6.85 (s, 2H), 7.39-7.45 (m, 6H), 7.61-7.66 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = –3.0, 19.6, 20.8, 25.7, 32.1, 63.6, 127.8, 128.8, 129.8, 134.3, 134.8, 135.9, 136.0, 136.2. IR (film, cm^{-1}): 3069, 3048, 3001, 2949, 2918, 2865, 1974, 1956, 1884, 1827, 1614, 1590, 1485, 1468, 1428, 1379, 1303, 1253, 1178, 1111, 1081, 1028, 1011, 998, 969, 914, 849, 818, 788, 766, 729, 696, 655. MS (EI): *m*/*z* (%) 374 (26), 296 (12), 197 (27), 183 (11), 160 (100), 145 (61), 133 (50), 117 (8), 105 (9), 91 (14), 77 (7), 41 (4). HRMS (EI): *m*/*z*: calcd for $C_{25}H_{30}OSi$ [*M*]: 374.20627, found: 374.20659.



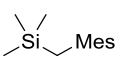
Compound 29. Colorless syrup (80%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.11 (t, *J* = 7.4 Hz, 3H), 1.35 (s, 9H), 1.66-1.77 (m, 6H), 1.82-1.87 (m, 1H), 2.50 (s, 3H), 2.52 (s, 6H), 2.82 (t, *J* = 2.7 Hz, 2H), 3.85-3.86 (m, 2H), 7.08 (s, 2H), 7.58-7.66 (m, 6H), 7.94-7.96 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 11.3, 19.4, 19.7, 20.9, 23.7, 26.7, 27.0, 29.9, 31.2, 42.0, 65.7, 127.6, 128.8, 129.5, 134.1, 134.6, 135.6, 135.7, 136.5. IR (film, cm^{-1}): 3071, 3050, 3000, 2957, 2930, 2857, 1900, 1825, 1614, 1589, 1484, 1471, 1462, 1427, 1389, 1377, 1361, 1306, 1260, 1187, 1106, 1029, 1007, 998, 970, 938, 908, 850, 823, 793, 736, 699, 689. MS (EI): *m*/*z* (%) 415 (74), 215 (5), 199 (58), 183 (11), 159 (10), 133 (100), 91 (5), 77 (6), 57 (4), 41 (6). HRMS (ESI^+): *m*/*z*: calcd for $C_{32}H_{44}OSiNa$ [*M+Na*]: 495.30520, found: 495.30536.



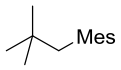
Compound 30. Colorless syrup (81%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.19-2.28 (m, 2H), 2.33 (s, 3H), 2.37 (s, 6H), 2.98 (t, *J* = 2.7 Hz, 2H), 6.94 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 19.3, 19.8 (t, *J* = 16.6 Hz), 20.7, 30.5 (t, *J* = 90.4 Hz), 105-125 (m), 129.3, 132.7, 136.1, 136.2. IR (film, cm^{-1}): 2975, 2926, 1615, 1581, 1486, 1472, 1451, 1365, 1317, 1233, 1188, 1142, 1119, 1070, 1027, 979, 945, 903, 853, 807, 778, 746, 736, 722, 706, 692. MS (EI): *m*/*z* (%) 466 (29), 447 (5), 133 (100), 119 (6), 91 (5), 69 (7). HRMS (EI): *m*/*z*: calcd for $C_{17}H_{15}F_{13}$ [*M*]: 466.09643, found: 466.09662.



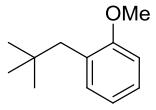
Compound 31. Colorless syrup (58%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.06 (s, 9H), 2.14 (s, 2H), 2.23 (s, 6H), 2.27 (s, 3H), 6.84 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = -0.1, 19.6, 20.7, 21.1, 128.6, 132.7, 134.6, 134.8. IR (film, cm^{-1}): 2953, 2917, 2731, 1723, 1613, 1575, 1480, 1445, 1417, 1375, 1289, 1248, 1206, 1164, 1027, 1011, 922, 838, 782, 760, 726, 690, 662. MS (EI): *m*/*z* (%) 206 (28), 191 (5), 175 (5), 133 (7), 117 (3), 105 (2), 91 (4), 73 (100), 59 (3), 45 (6). HRMS (EI): *m*/*z*: calcd for $C_{13}H_{22}Si$ [*M*]: 206.14889, found: 206.14908.



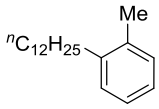
Compound 32. Colorless syrup (72%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.98 (s, 9H), 2.27 (s, 3H), 2.32 (s, 6H), 2.66 (s, 2H), 6.86 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 20.7, 21.6, 30.6, 34.7, 40.7, 126.9, 129.0, 134.1, 134.5, 137.7. IR (film, cm^{-1}): 2949, 2867, 1725, 1613, 1477, 1391, 1376, 1362, 1230, 1199, 1156, 1033, 850, 790, 751, 697, 583, 564, 515, 468. MS (EI): *m*/*z* (%) 190 (19), 175 (4), 133 (100), 119 (19), 105 (5), 91 (7), 77 (4), 57 (10), 41 (6). HRMS (EI): *m*/*z*: calcd for $C_{14}H_{22}$ [*M*]: 190.17198, found: 190.17215.



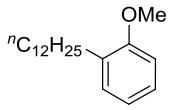
Compound 33. Colorless syrup (55%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.92 (s, 9H), 2.57 (s, 2H), 3.80 (s, 3H), 6.85-6.90 (m, 2H), 7.08-7.10 (m, 1H), 7.17-7.21 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 29.5, 32.4, 42.7, 55.0, 110.3, 119.7, 127.0, 128.4, 132.4, 158.1. IR (film, cm^{-1}): 2949, 2905, 2865, 2835, 1600, 1585, 1493, 1462, 1438, 1392, 1363, 1330, 1290, 1240, 1202, 1178, 1153, 1107, 1053, 1036, 930, 906, 833, 744, 709, 611, 572, 537, 482, 435. MS (EI): *m*/*z* (%) 178 (33), 163 (10), 135 (5), 122 (100), 115 (4), 107 (10), 91 (51), 77 (11), 65 (11), 57 (29), 51 (6), 41 (16), 29 (13). HRMS (EI): *m*/*z*: calcd for $C_{12}H_{18}O$ [*M*]: 178.13566, found: 178.13577.



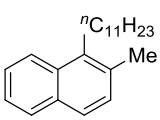
Compound 34. Colorless oily liquid (77%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.90 (t, *J* = 6.2 Hz, 3H), 1.28-1.42 (m, 18H), 1.53-1.62 (m, 2H), 2.32 (s, 3H), 2.60 (t, *J* = 7.7 Hz, 2H), 7.08-7.15 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.1, 19.3, 22.7, 29.4, 29.57, 29.63, 29.65, 29.68, 29.73, 30.3, 31.9, 33.3, 125.7, 125.8, 128.8, 130.1, 135.8, 141.1. IR (film, cm^{-1}): 2922, 2852, 1604, 1493, 1463, 1378, 739, 450. MS (EI): *m*/*z* (%) 260 (33), 118 (3), 105 (100), 91 (9), 79 (5), 57 (6), 43 (13), 29 (9). HRMS (EI): *m*/*z*: calcd for $C_{19}H_{32}$ [*M*]: 260.25038, found: 260.25040.



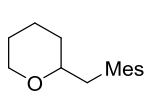
Compound 35. Colorless syrup (70%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.89 (t, *J* = 6.7 Hz, 3H), 1.27-1.37 (m, 18H), 1.55-1.62 (m, 2H), 2.61 (t, *J* = 7.6 Hz, 2H), 3.83 (s, 3H), 6.84-6.90 (m, 2H), 7.13-7.19 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.1, 22.7, 29.4, 29.55, 29.63, 29.65, 29.70, 29.87, 30.1, 31.9, 55.2, 110.2, 120.3, 126.7, 129.7, 131.4, 157.5. IR (film, cm^{-1}): 2922, 2852, 1601, 1588, 1493, 1464, 1438, 1377, 1326, 1289, 1241, 1176, 1128, 1104, 1051, 1033, 742, 730, 569, 465. MS (EI): *m*/*z* (%) 276 (44), 121 (100), 108 (4), 91 (42), 77 (4), 65 (4), 55 (5), 43 (17), 41 (17), 29 (14). HRMS (EI): *m*/*z*: calcd for $C_{19}H_{32}O$ [*M*]: 276.24507, found: 276.24532.



Compound 36. Colorless syrup (78% (GC); because of the low polarity of the compound, trace impurities had to be removed by HPLC). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.90 (t, *J* = 6.1 Hz, 3H), 1.29-1.41 (m, 14H), 1.48-1.55 (m, 2H), 1.60-1.68 (m, 2H), 2.51 (s, 3H), 3.06 (t, *J* = 7.2 Hz, 2H), 7.30 (d, *J* = 8.5 Hz, 1H), 7.39-7.42 (m, 1H), 7.47-7.51 (m, 1H), 7.62 (d, *J* = 8.2 Hz, 1H), 7.80 (d, *J* = 7.7 Hz, 1H), 8.03 (d, *J* = 8.5 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.1, 20.1, 22.7, 28.8, 29.3, 29.58, 29.65, 29.69, 30.1, 30.3, 31.9, 123.7, 124.4, 125.7, 125.8, 128.5, 129.2, 132.1, 132.58, 132.63, 136.2. IR (film, cm^{-1}): 2930, 2858, 1282, 1174, 1100, 744, 563. MS (EI): *m*/*z* (%) 296 (59), 167 (3), 155 (100), 129 (3), 115 (3), 41 (4). HRMS (EI): *m*/*z*: calcd for $C_{22}H_{32}$ [*M*]: 296.25029, found: 296.25040.

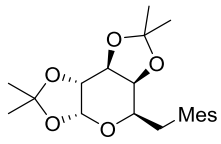


Compound 37. Colorless syrup (78%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.33-1.83 (m, 6H), 2.26 (s, 3H), 2.32 (s, 6H), 2.26-2.91 (m, 2H), 3.35-3.47 (m, 2H), 3.95-3.99 (m, 1H), 6.84 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 20.5, 20.8, 23.8, 26.1, 32.0, 36.3, 68.7, 78.4, 128.9, 132.8, 135.3, 137.0. IR (film, cm^{-1}): 2934, 2844, 2737, 1957, 1614, 1577, 1484, 1440, 1377,

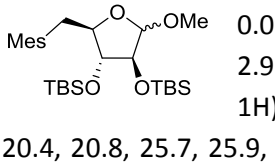


1365, 1350, 1286, 1261, 1219, 1195, 1173, 1086, 1067, 1045, 979, 946, 901, 849, 825, 800, 774, 732. MS (EI): *m/z* (%) 218 (12), 134 (22), 117 (4), 105 (3), 85 (100), 67 (12), 57 (12), 43 (13), 41 (11), 29 (8). HRMS (EI): *m/z*: calcd for $C_{15}H_{22}O$ [*M*]: 218.16691, found: 218.16707.

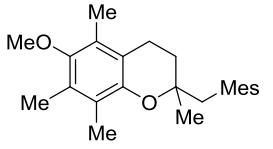
Compound 38. Colorless syrup (80%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.32 (s, 3H), 1.39 (s, 3H), 1.47 (s, 3H), 1.57 (s, 3H), 2.28 (s, 3H), 2.39 (s, 6H), 2.95-3.00 (m, 1H), 3.07-3.12 (m, 1H), 3.95 (t, *J* = 7.3 Hz, 1H), 4.09 (dd, *J* = 8.0, 1.4 Hz, 1H), 4.27 (dd, *J* = 5.0, 2.0 Hz, 1H), 4.55 (dd, *J* = 8.0, 2.0 Hz, 1H), 5.57 (d, *J* = 4.3 Hz, 1H), 6.87 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 20.1, 20.7, 24.2, 24.7, 26.0, 26.1, 30.0, 67.6, 70.7, 70.9, 71.8, 96.6, 107.9, 108.7, 128.9, 131.8, 135.3, 137.1. IR (film, cm^{-1}): 2986, 2918, 2732, 1732, 1614, 1578, 1485, 1456, 1380, 1371, 1310, 1254, 1209, 1166, 1142, 1101, 1066, 1037, 1019, 996, 956, 917, 901, 883, 864, 849, 807, 756, 733, 677. MS (EI): *m/z* (%) 362 (33), 347 (14), 229 (17), 171 (46), 133 (70), 113 (8), 100 (10), 85 (9), 81 (9), 71 (100), 59 (16), 43 31). HRMS (ESI$^+$): *m/z*: calcd for $C_{21}H_{30}O_5Na$ [*M+Na*]: 385.19848, found: 385.19854.



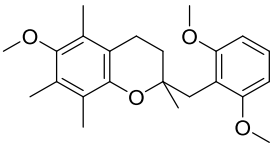
Compound 39. Colorless syrup (89%). For analytical purposes, the two anomers were separated by HPLC: ***major anomer***: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.04 (s, 3H), 0.07 (s, 3H), 0.09 (s, 3H), 0.10 (s, 3H), 0.88 (s, 9H), 0.92 (s, 9H), 2.24 (s, 3H), 2.31 (s, 6H), 2.82-2.95 (m, 2H), 3.20 (s, 3H), 3.79-3.82 (m, 1H), 3.96-4.02 (m, 2H), 4.66 (d, *J* = 1.6 Hz, 1H), 6.82 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = -4.8, -4.61, -4.58, -4.2, 17.8, 18.0, 20.4, 20.8, 25.7, 25.9, 32.9, 54.8, 83.1, 83.3, 84.2, 109.2, 128.8, 132.8, 135.2, 136.9. IR (film, cm^{-1}): 2954, 2929, 2858, 1614, 1485, 1472, 1463, 1408, 1388, 1362, 1321, 1251, 1189, 1105, 1081, 1044, 1006, 954, 917, 837, 776, 670; MS (EI): *m/z* (%) 463 (2), 437 (21), 405 (4), 377 (5), 331 (46), 301 (42), 275 (9), 231 (4), 201 (9), 171 (9), 147 (9), 133 (100), 89 (28), 73 (76), 57 (14), 41 (7). HRMS (ESI$^+$): *m/z*: calcd for $C_{27}H_{50}O_4Si_2Na$ [*M+Na*]: 517.31420, found: 517.31399; ***minor anomer***: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.08 (s, 3H), 0.09 (s, 3H), 0.10 (s, 3H), 0.13 (s, 3H), 0.91 (s, 9H), 0.93 (s, 9H), 2.23 (s, 3H), 2.33 (s, 6H), 2.86-2.98 (m, 2H), 3.45 (s, 3H), 3.84-3.89 (m, 1H), 3.97 (dd, *J* = 7.0, 4.5 Hz, 1H), 4.07 (t, *J* = 7.0 Hz, 1H), 4.65 (d, *J* = 4.4 Hz, 1H), 6.83 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = -4.53, -4.49, -4.37, -3.8, 17.8, 18.2, 20.4, 20.8, 25.8, 26.0, 34.0, 56.1, 78.3, 79.8, 81.4, 103.1, 129.0, 132.5, 135.5, 136.7. IR (film, cm^{-1}): 2953, 2928, 2857, 1614, 1578, 1472, 1463, 1445, 1407, 1388, 1374, 1361, 1331, 1250, 1226, 1189, 1165, 1146, 1100, 1084, 1047, 1003, 939, 918, 895, 836, 776, 739, 721, 671.



Compound 40. White solid (77%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.21 (s, 3H), 1.80-1.90 (m, 2H), 2.08 (s, 3H), 2.14 (s, 3H), 2.19 (s, 3H), 2.26 (s, 3H), 2.39 (s, 6H), 2.60-2.66 (m, 2H), 3.11 (d, *J* = 3.2 Hz, 2H), 3.63 (s, 3H), 6.86 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 11.6, 12.2, 12.6, 20.7, 20.8, 21.5, 23.4, 32.0, 40.2, 53.4, 60.4, 117.4, 123.1, 125.7, 127.9, 128.9, 132.0, 135.3, 138.0, 147.7, 149.5. IR (film, cm^{-1}): 3747, 3651, 2978, 2938, 2915, 2824, 2730, 1724, 1611, 1573, 1455, 1398, 1376, 1347, 1327, 1304, 1253, 1205, 1188, 1162, 1116, 1097, 1086, 1059, 1034, 1007, 988, 924, 864, 849, 812, 789, 762, 744, 720, 680, 670. MS (EI): *m/z* (%) 352 (12), 219 (100), 179 (7), 133 (8), 105 (3), 91 (4), 43 (5). HRMS (EI): *m/z*: calcd for $C_{24}H_{32}O_2$ [*M*]: 352.23997, found: 352.24023.

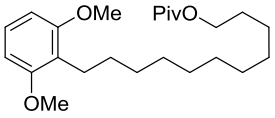


Compound 41. An extended reaction time (20 h) and 1.5 equiv. of the Grignard reagent were used to form this product as a pale yellow syrup (62%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.21 (s, 3H), 1.75-1.94 (m, 2H), 2.08 (s, 3H), 2.15 (s, 3H), 2.19 (s, 3H), 2.51-2.74 (m, 2H), 3.00-3.13 (m, 2H), 3.64 (s, 3H), 3.75 (s, 6H), 6.53 (d, *J* = 8.3 Hz, 2H), 7.16 (t, *J* = 8.3 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 11.66, 11.73, 12.5, 20.7, 23.9, 31.2, 32.8, 55.2, 60.4, 76.6, 103.2, 114.6, 117.4, 112.9,

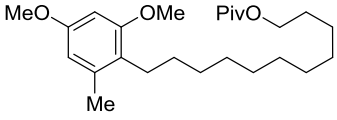


125.5, 127.3, 127.4, 148.3, 149.1, 159.2. IR (film, cm^{-1}): 2934, 2835, 1593, 1473, 1457, 1403, 1376, 1332, 1280, 1251, 1188, 1172, 1140, 1120, 1104, 1086, 1061, 1040, 1013, 909, 857, 803, 782, 730, 702, 681, 648, 615, 587. MS (ESI^+): *m*/*z* (%) 393.2 (100). HRMS (ESI^+): *m*/*z*: calcd for $C_{23}H_{30}O_4Na$ $[M+Na]^+$: 393.20347, found: 393.20363.

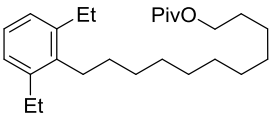
Compound 42. Colorless syrup (88%). ^1H NMR (400 MHz, $CDCl_3$): δ = 1.21 (s, 9H), 1.27-1.37 (m, 20H), 1.44-1.51 (m, 2H), 1.59-1.66 (m, 2H), 2.54 (t, *J* = 7.5 Hz, 2H), 4.06 (t, *J* = 6.5 Hz, 2H), 6.44 (d, *J* = 8.4 Hz, 2H), 7.02 (t, *J* = 8.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 22.8, 25.9, 27.2, 28.6, 29.2, 29.5, 29.56, 29.58, 29.79, 38.7, 55.6, 64.4, 103.7, 119.7, 126.3, 158.3, 178.6. IR (film, cm^{-1}): 2925, 2854, 1726, 1594, 1473, 1435, 1397, 1365, 1328, 1282, 1254, 1151, 1126, 1091, 1041, 979, 939, 773, 723, 698, 589. MS (EI): *m*/*z* (%) 392 (25), 290 (3), 151 (100), 138 (3), 121 (5), 91 (13), 85 (3), 57 (20), 41 (7). HRMS (ESI^+): *m*/*z*: calcd for $C_{24}H_{40}O_4Na$ $[M+Na]^+$: 415.28167, found: 415.28188.



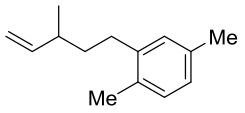
Compound 43. Colorless syrup (85%). ^1H NMR (400 MHz, $CDCl_3$): δ = 1.21 (s, 9H), 1.29-1.47 (m, 16H), 1.59-1.66 (m, 2H), 2.29 (s, 3H), 2.55 (t, *J* = 7.4 Hz, 2H), 3.78 (s, 6H), 4.06 (t, *J* = 6.7 Hz, 2H), 6.32 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 25.7, 25.9, 27.2, 28.6, 29.2, 29.48, 29.51, 29.53, 29.57, 29.90, 38.7, 55.1, 55.4, 64.4, 96.0, 106.4, 122.4, 137.6, 158.0, 158.4, 178.6. IR (film, cm^{-1}): 2924, 2853, 1727, 1606, 1590, 1492, 1480, 1463, 1419, 1342, 1315, 1284, 1201, 1146, 1123, 1085, 1062, 1035, 1005, 952, 827. MS (EI): *m*/*z* (%) 406 (29), 304 (3), 165 (100), 135 (12), 85 (3), 57 (15), 41 (5). HRMS (ESI^+): *m*/*z*: calcd for $C_{25}H_{42}O_4Na$ $[M+Na]^+$: 429.29731, found: 429.29753.



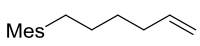
Compound 44. Colorless syrup (83%). ^1H NMR (400 MHz, $CDCl_3$): δ = 1.22 (s, 9H), 1.25 (t, *J* = 7.3 Hz, 6H), 1.29-1.35 (m, 12H), 1.38-1.52 (m, 4H), 1.61-1.68 (m, 2H), 2.61-2.68 (m, 2H), 2.67 (q, *J* = 7.7 Hz, 4H), 4.07 (t, *J* = 6.6 Hz, 2H), 7.01-7.13 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 15.7, 25.8, 25.9, 27.2, 28.6, 29.2, 29.45, 29.49, 29.51, 29.56, 30.4, 31.1, 38.7, 64.4, 125.8, 126.1, 138.3, 142.0, 178.6. IR (film, cm^{-1}): 2963, 2925, 2854, 1729, 1479, 1459, 1397, 1365, 1283, 1151, 1059, 1035, 977, 939, 799, 770, 751, 722. MS (EI): *m*/*z* (%) 388 (22), 286 (8), 257 (8), 173 (4), 159 (11), 147 (100), 133 (41), 131 (12), 119 (54), 117 (19), 105 (13), 103 (12), 91 (14), 85 (15), 57 (92), 55 (13), 41 (32), 29 (15). HRMS (ESI^+): *m*/*z*: calcd for $C_{26}H_{44}O_2Na$ $[M+Na]^+$: 411.32322, found: 411.32335.



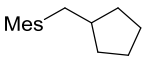
Compound 46. Colorless syrup (73%). ^1H NMR (400 MHz, $CDCl_3$): δ = 1.09 (d, *J* = 6.7 Hz, 3H), 1.54-1.60 (m, 2H), 2.20-2.26 (m, 1H), 2.28 (s, 3H), 2.32 (s, 3H), 2.50-2.64 (m, 2H), 4.99-5.08 (m, 2H), 5.75-5.84 (m, 1H), 6.93 (d, *J* = 7.6 Hz, 1H), 6.97 (s, 1H), 7.04 (d, *J* = 7.7 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 18.7, 20.2, 20.9, 31.0, 37.3, 38.0, 112.9, 126.4, 129.6, 130.0, 132.6, 135.2, 140.8, 144.4. IR (film, cm^{-1}): 3078, 2956, 2923, 2866, 1828, 1751, 1638, 1616, 1504, 1455, 1418, 1374, 1156, 1037, 994, 909, 879, 805, 714, 679. MS (EI): *m*/*z* (%) 188 (55), 173 (4), 159 (2), 145 (3), 132 (28), 119 (100), 105 (37), 91 (25), 77 (12), 65 (5), 53 (5), 41 (15), 27 (7). HRMS (EI): *m*/*z*: calcd for $C_{14}H_{20}$ [*M*]: 188.15636, found: 188.15650.



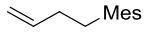
Compound 52. Colorless syrup (53%). ^1H NMR (400 MHz, $CDCl_3$): δ = 1.34-1.45 (m, 4H), 2.00-2.05 (m, 2H), 2.14 (s, 3H), 2.18 (s, 6H), 2.48 (t, *J* = 8.8 Hz, 2H), 4.84-4.95 (m, 2H), 5.68-5.78 (m, 1H), 6.72 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 19.7, 20.7, 28.8, 29.2, 29.4, 33.6, 114.4, 128.8, 134.7, 135.8, 136.4, 138.8. IR (film, cm^{-1}): 3076, 2920, 2857, 2731, 1823, 1728, 1640, 1614, 1579, 1484, 1468, 1455, 1376, 1255, 1206, 1176, 1108, 1071, 1029, 1012, 991, 908, 849, 723, 627. MS (EI): *m*/*z* (%) 202 (28), 159 (6), 147 (4), 133 (100), 120 (12), 105 (5), 91 (7), 82 (7), 41 (5). HRMS (EI): *m*/*z*: calcd for $C_{15}H_{22}$ [*M*]: 202.17206, found: 202.17215.

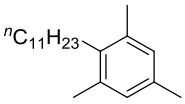


Compound 53. Colorless syrup (16%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.26-1.32 (m, 2H), 1.54-1.57 (m, 2H), 1.68-1.77 (m, 4H), 2.01-2.09 (m, 1H), 2.29 (s, 3H), 2.34 (s, 6H), 2.67 (d, J = 7.2 Hz, 2H), 6.87 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 20.4, 20.8, 24.7, 32.9, 34.3, 41.4, 128.9, 134.6, 136.2, 136.3. IR (film, cm^{-1}): 2949, 2866, 1733, 1614, 1578, 1482, 1452, 1376, 1205, 1143, 1028, 1012, 850, 721. MS (EI): *m*/*z* (%) 202 (17), 145 (2), 133 (100), 117 (6), 105 (7), 91 (8), 77 (4), 41 (12). HRMS (EI): *m*/*z*: calcd for $C_{15}H_{22}$ [*M*]: 202.17197, found: 202.17215.



Compound 55. Colorless syrup (64%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.27-2.39 (m, 2H), 2.35 (s, 3H), 2.39 (s, 6H), 2.76-2.80 (m, 2H), 5.08-5.12 (m, 1H), 5.16-5.22 (m, 1H), 5.98-6.08 (m, 1H), 6.93 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 19.7, 20.8, 28.9, 33.3, 114.5, 128.9, 135.0, 135.6, 135.9, 138.5. MS (EI): *m*/*z* (%) 174 (13), 133 (100), 117 (5), 115 (5), 105 (4), 91 (6), 77 (3), 41 (4). HRMS (EI): *m*/*z*: calcd for $C_{13}H_{18}$ [*M*]: 174.14100, found: 174.14085.

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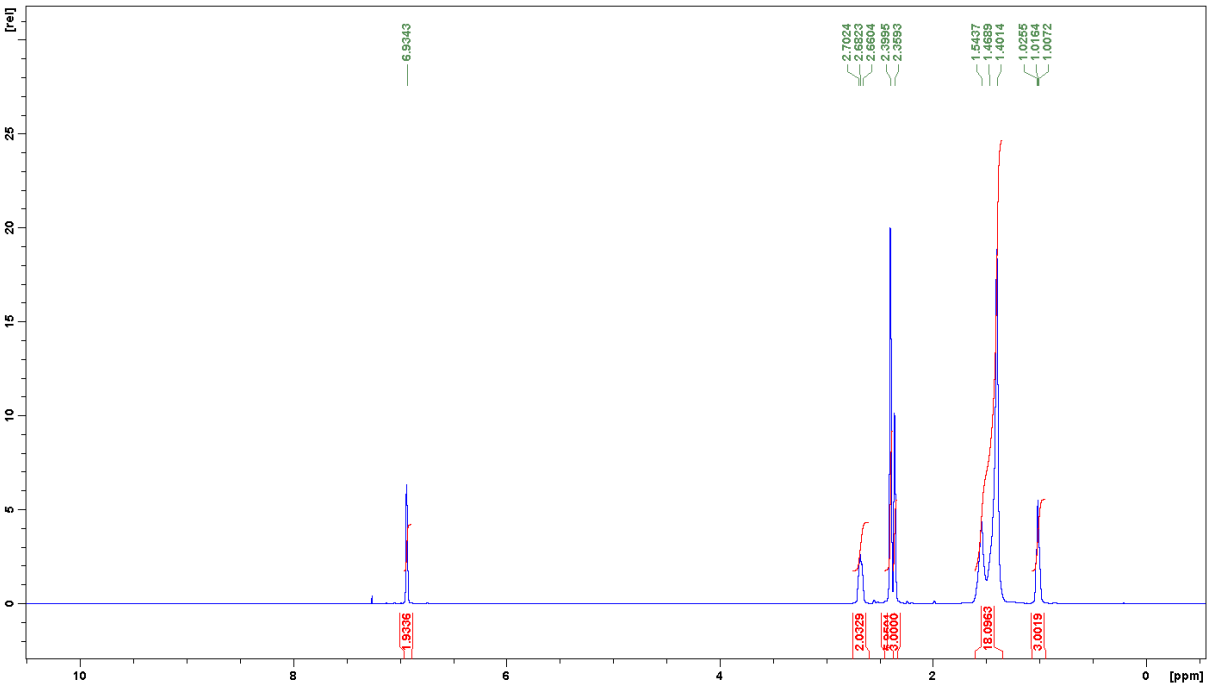
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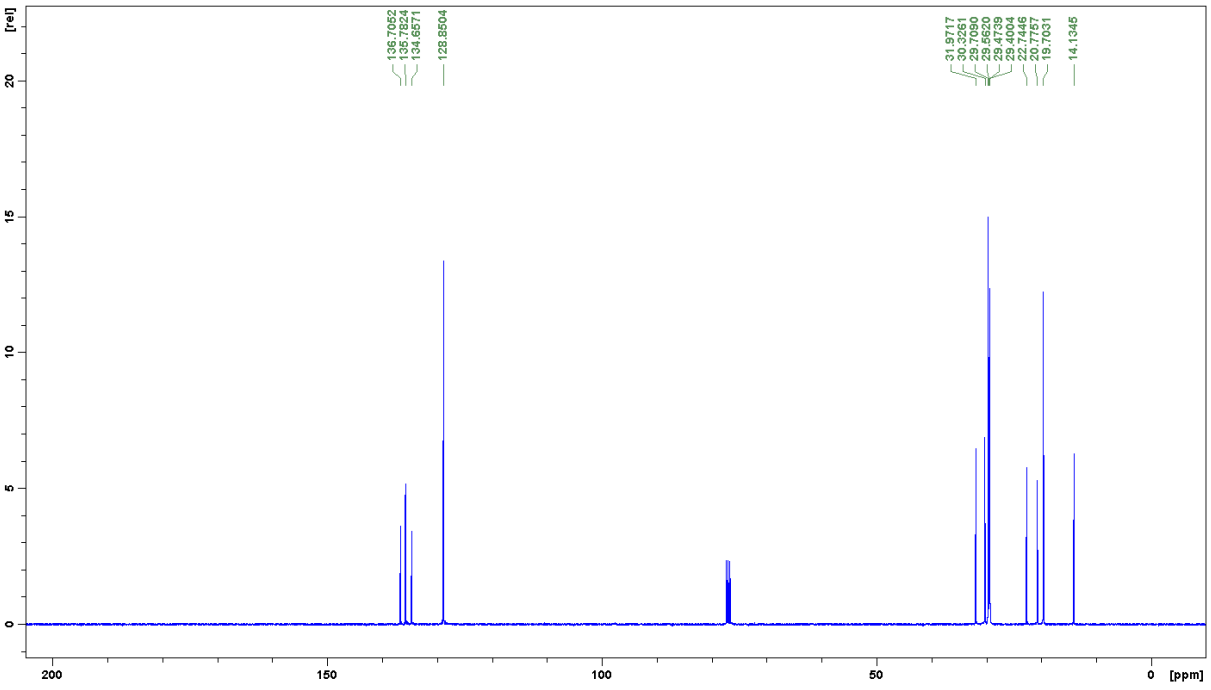
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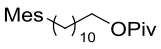
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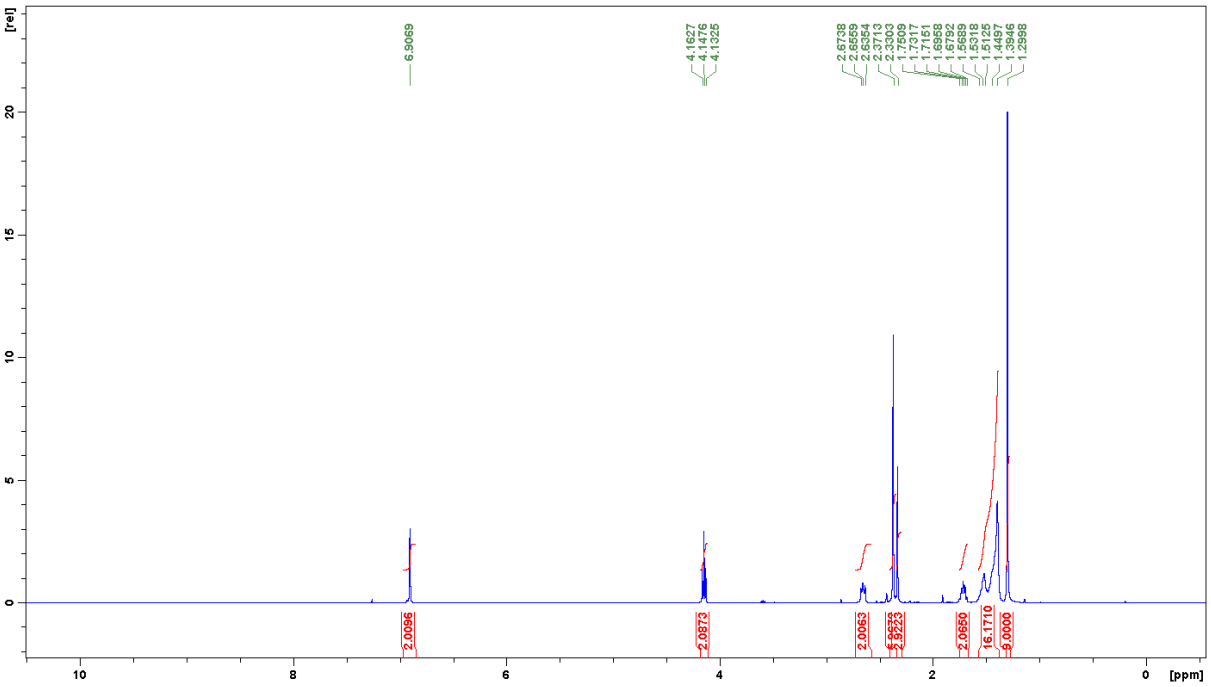
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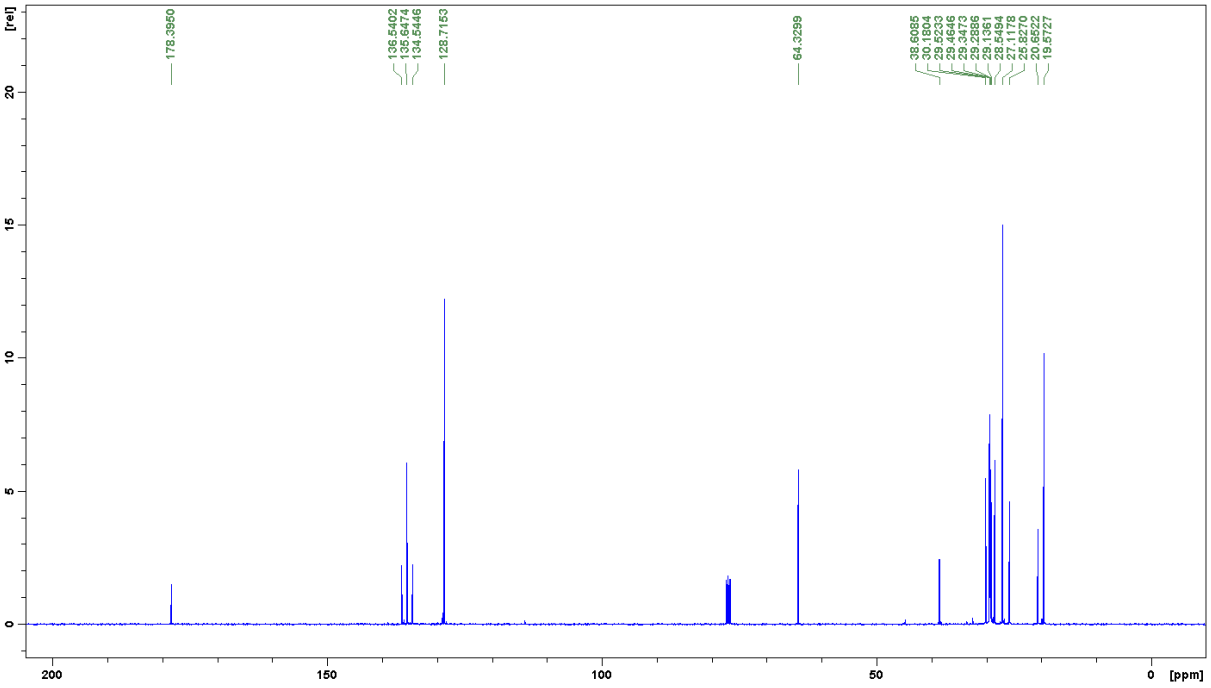
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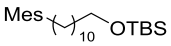
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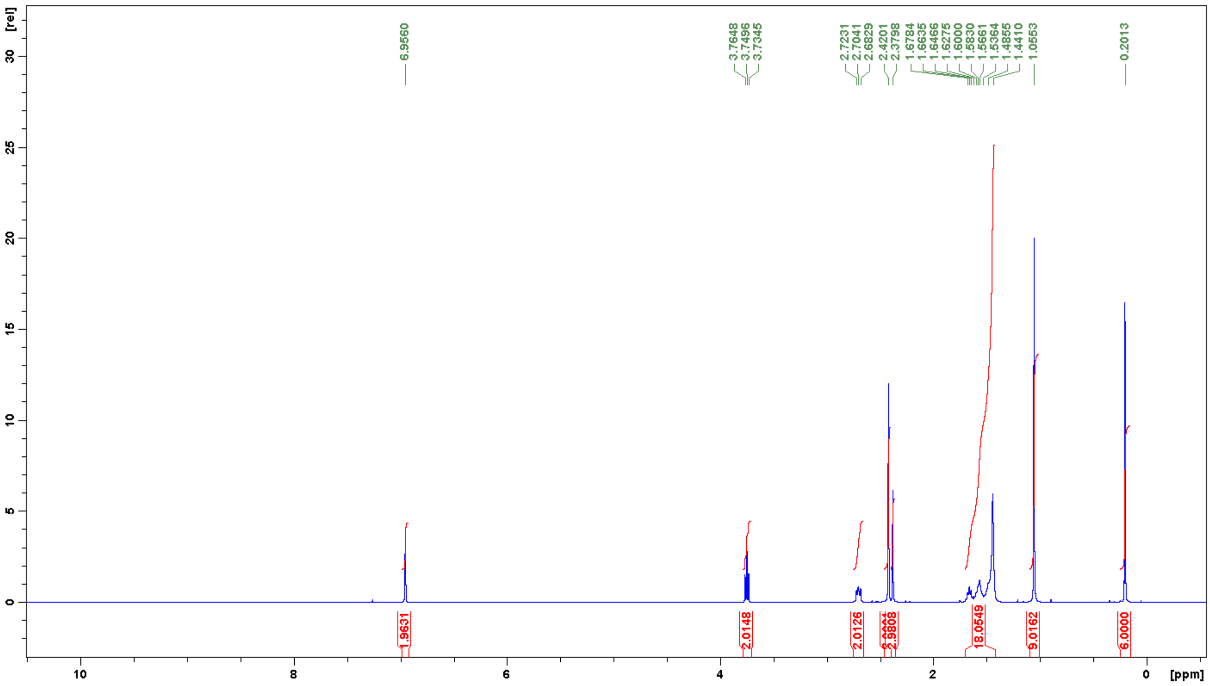
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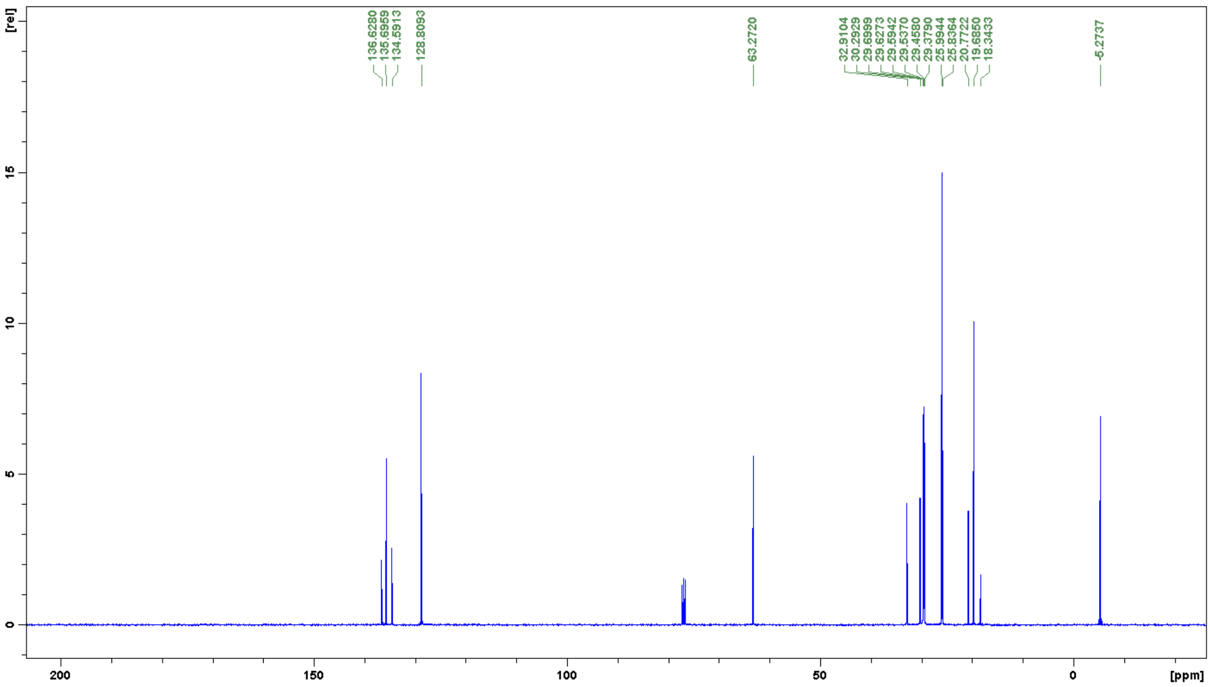
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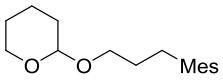
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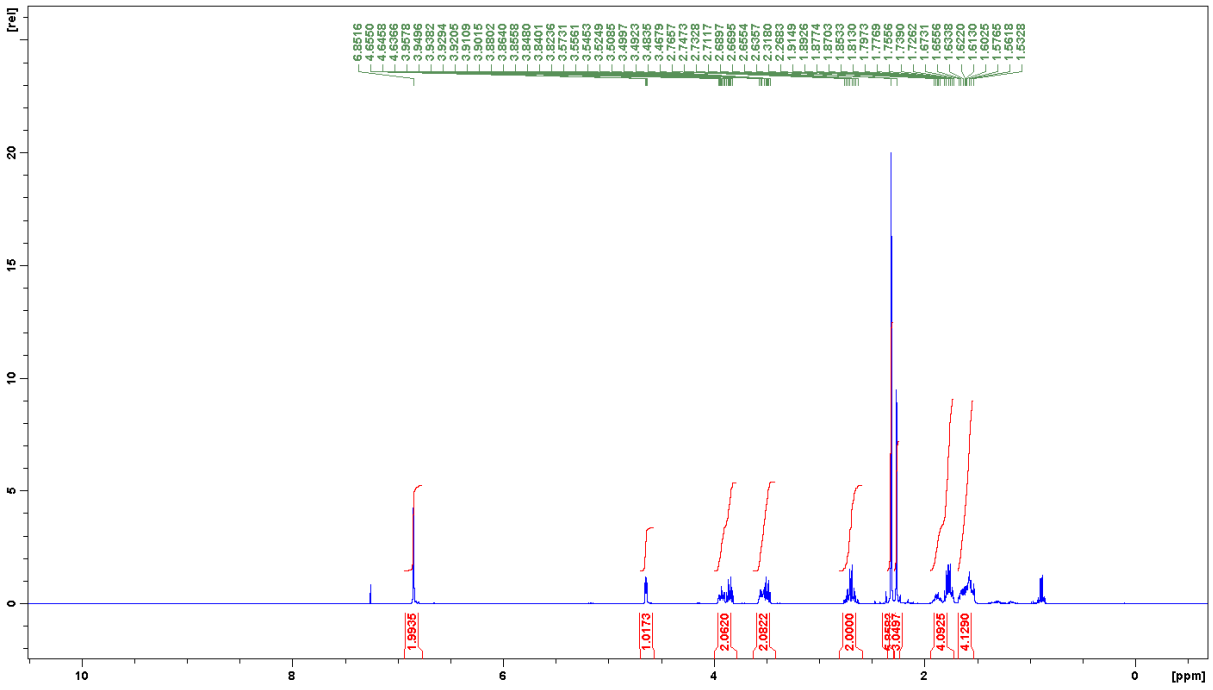
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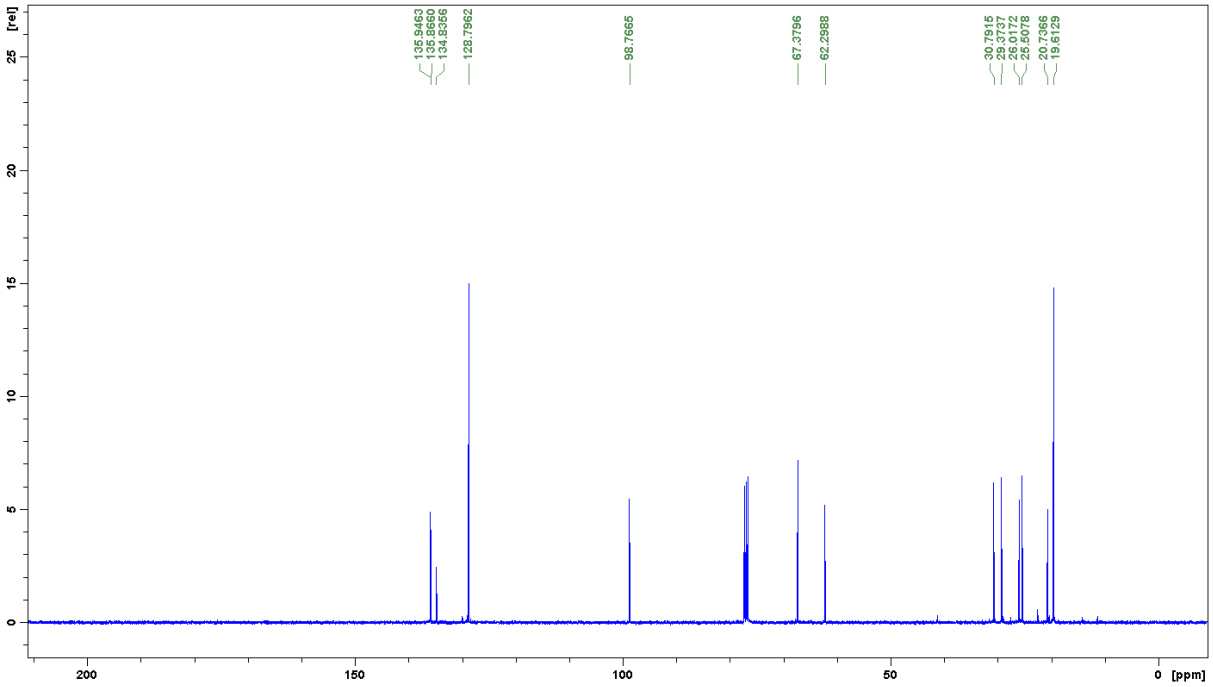
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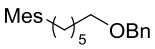
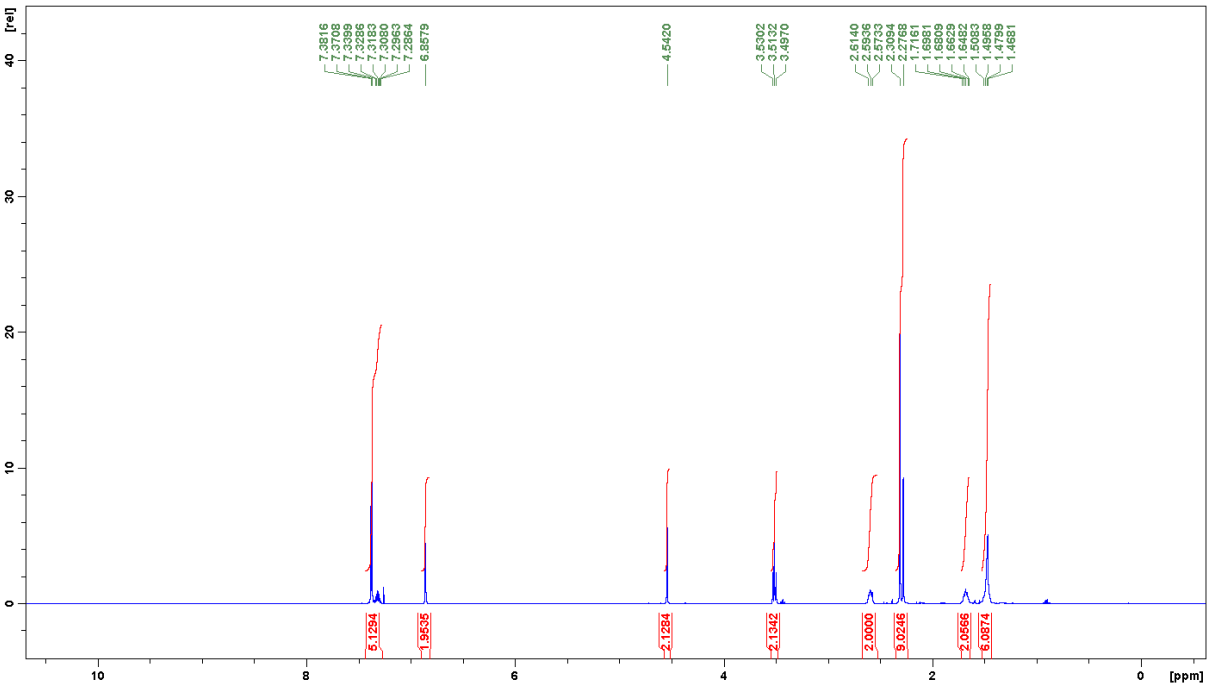
20.7366
19.6129


Mes
5
OBn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3.5132
3.4970
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2.5936
2.5733
2.3094
2.2768
2.1611
1.6981
1.6809
1.6629
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1.5083
1.4958
1.4799
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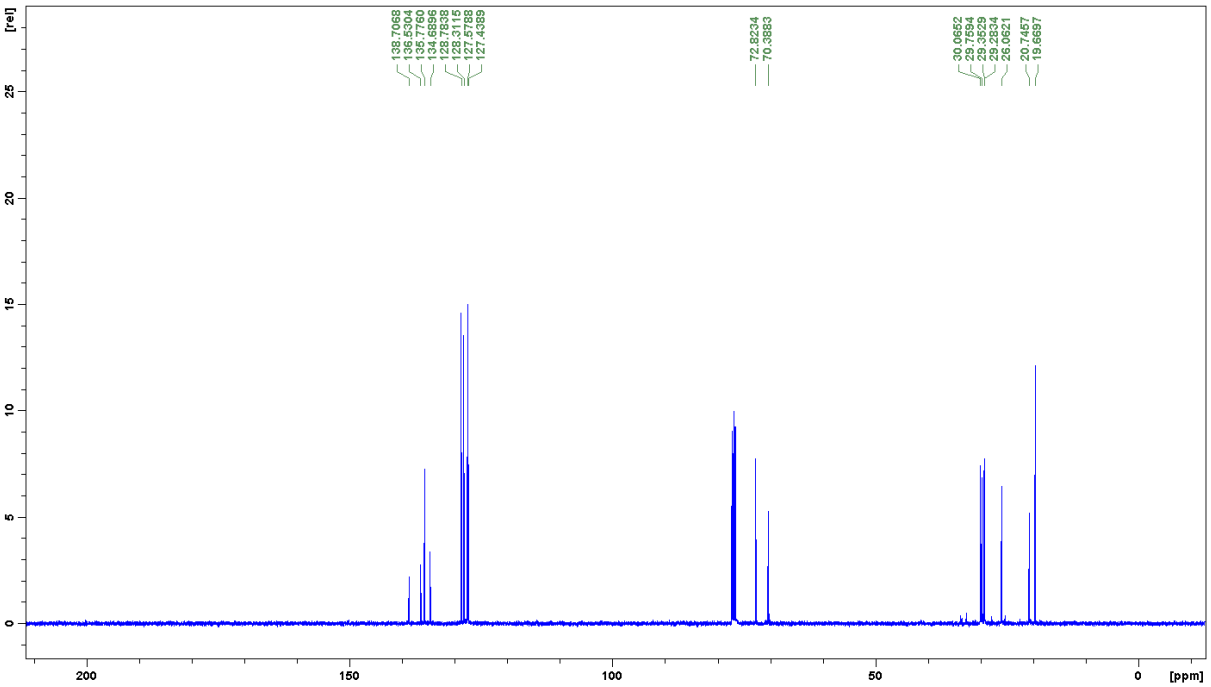
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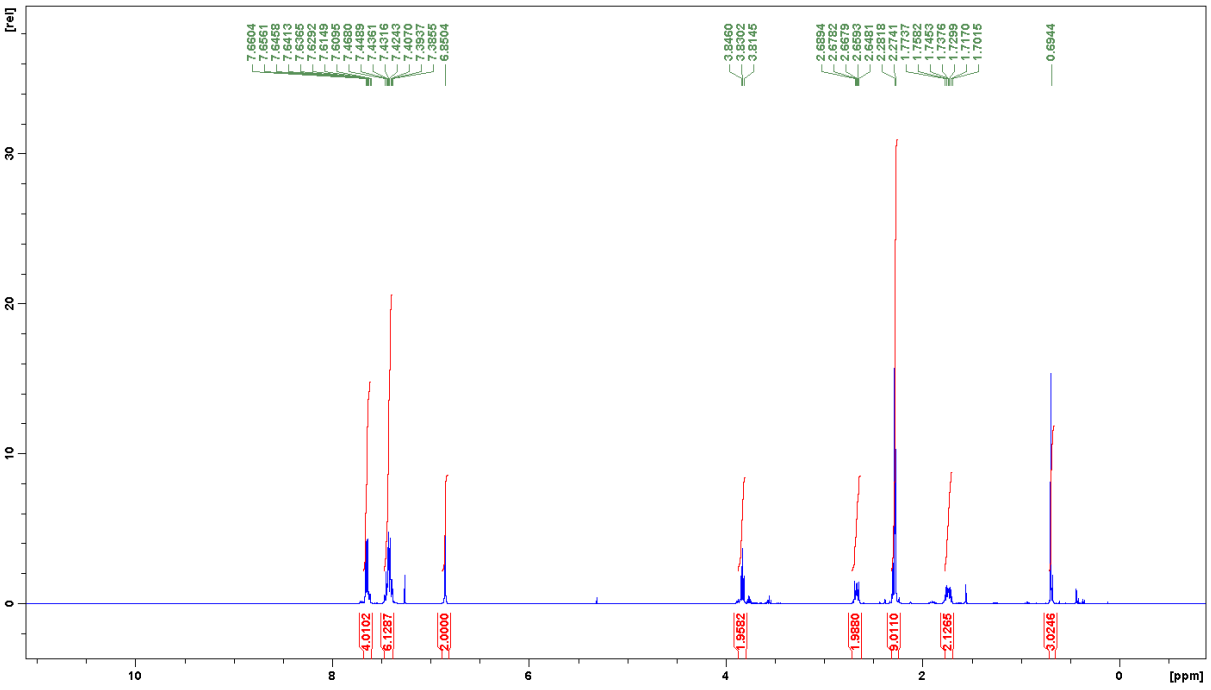
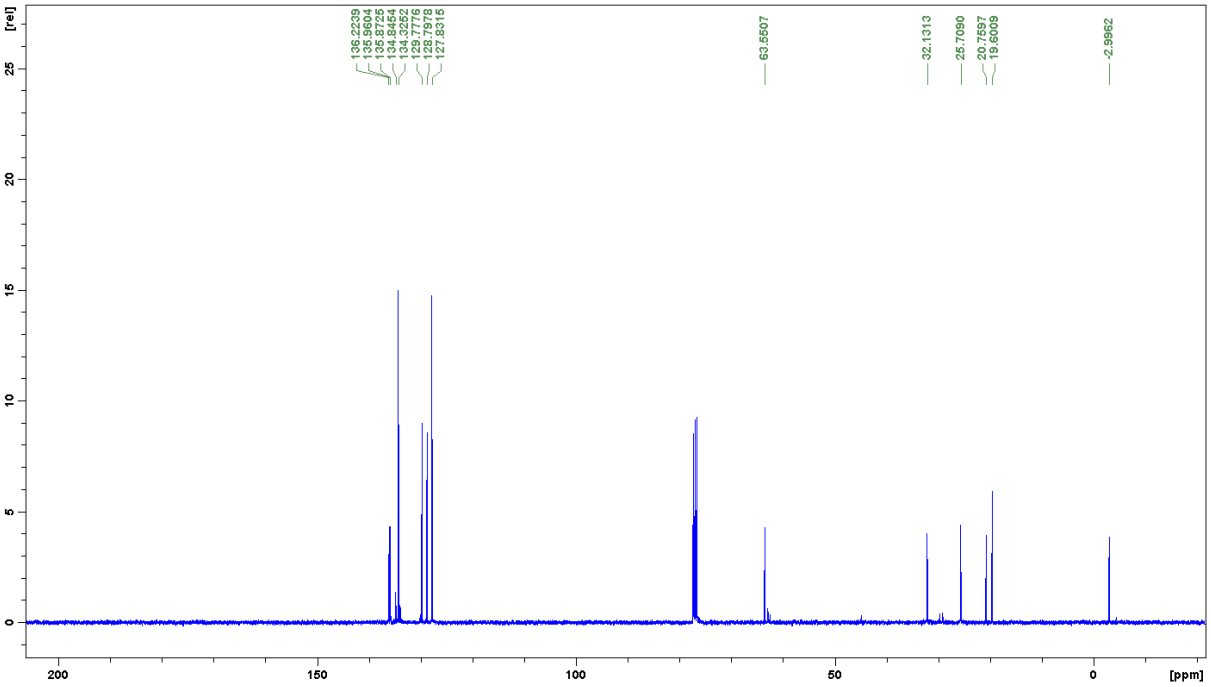
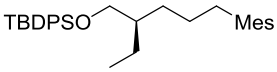
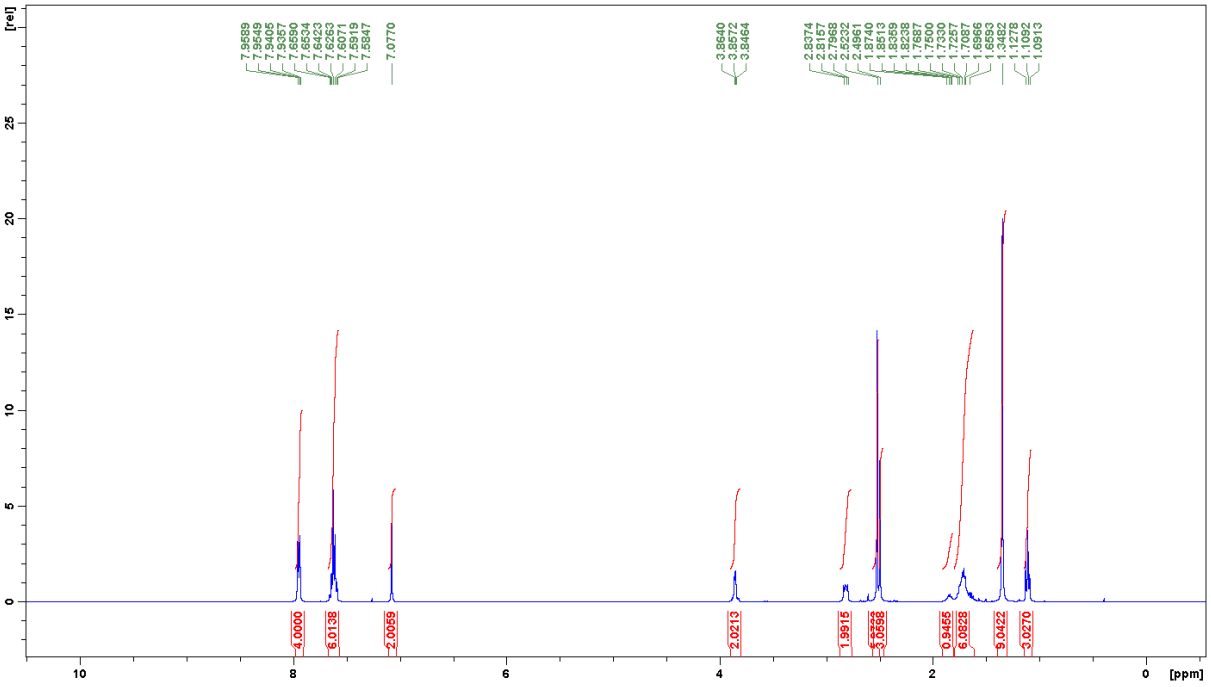
Ph_2MeSiO Mes






TBDPSO
Mes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7.0770
3.9640
3.8572
3.8464
2.8374
2.8157
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2.7832
2.4961
1.8740
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1.7330
1.7257
1.7087
1.6966
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1.1278
1.1092
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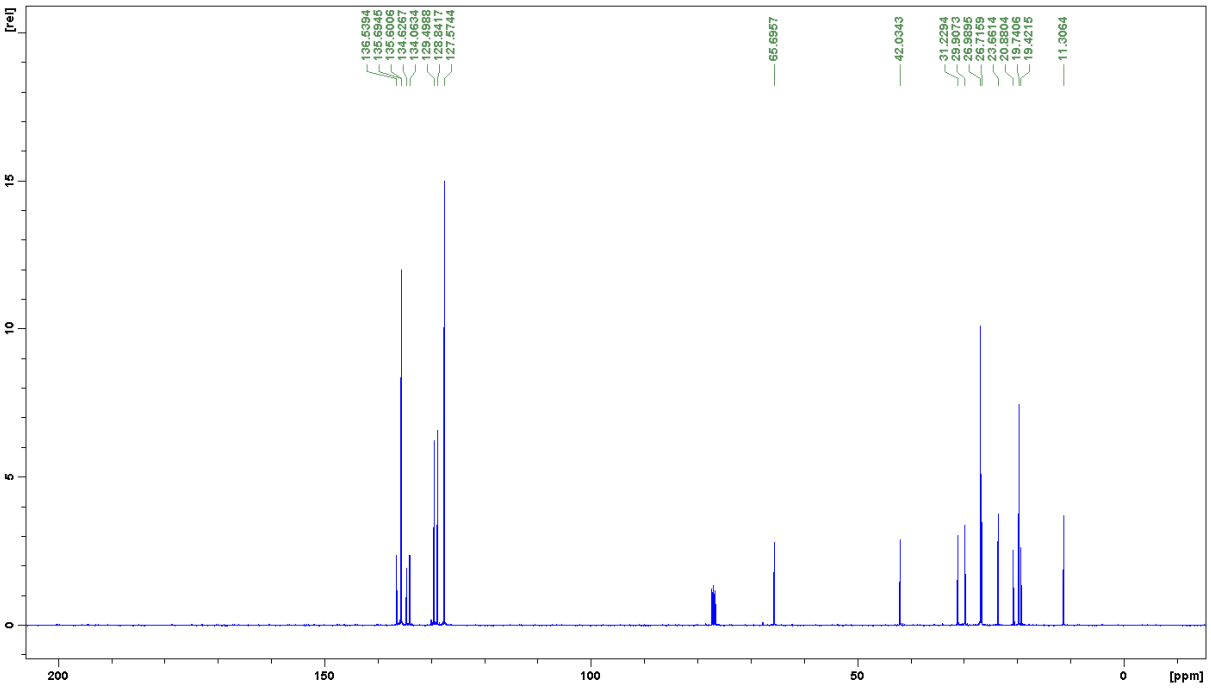
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19.4215
11.3064
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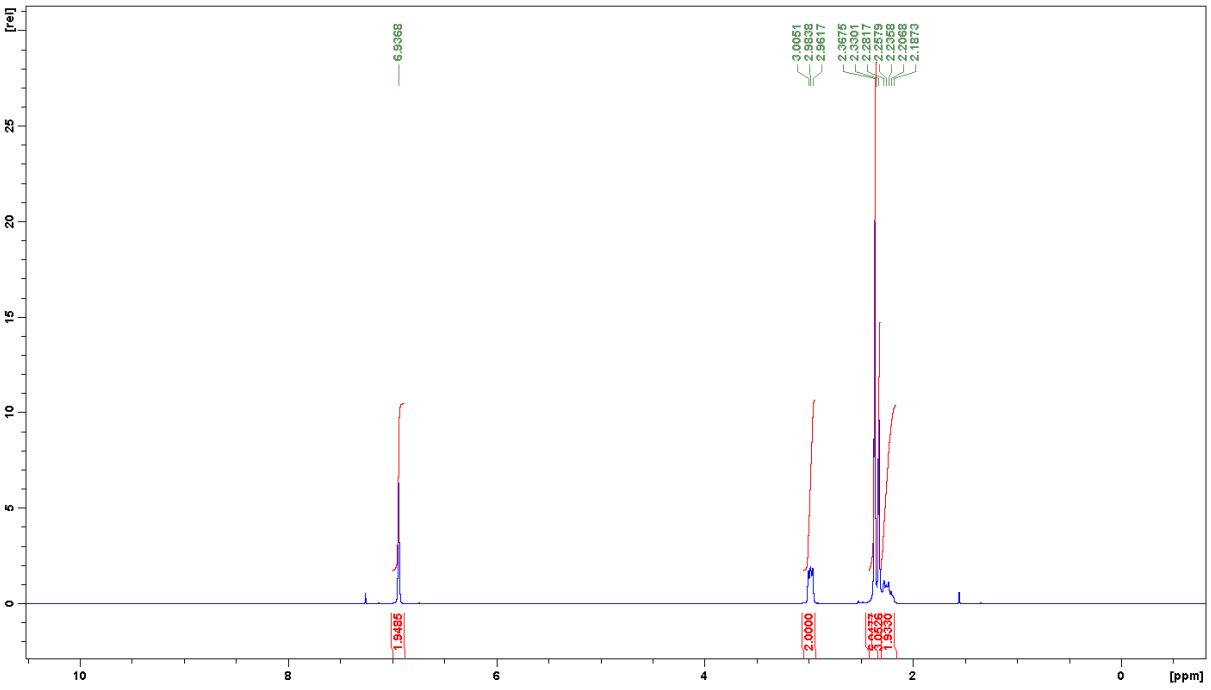
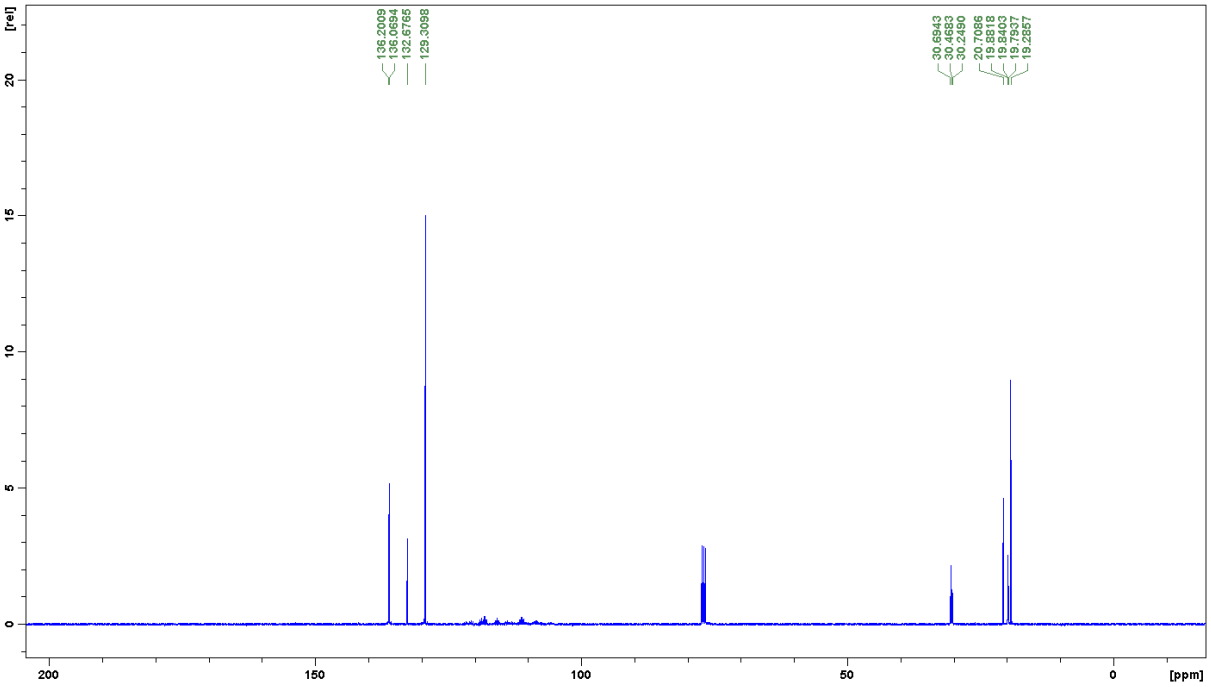
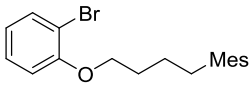
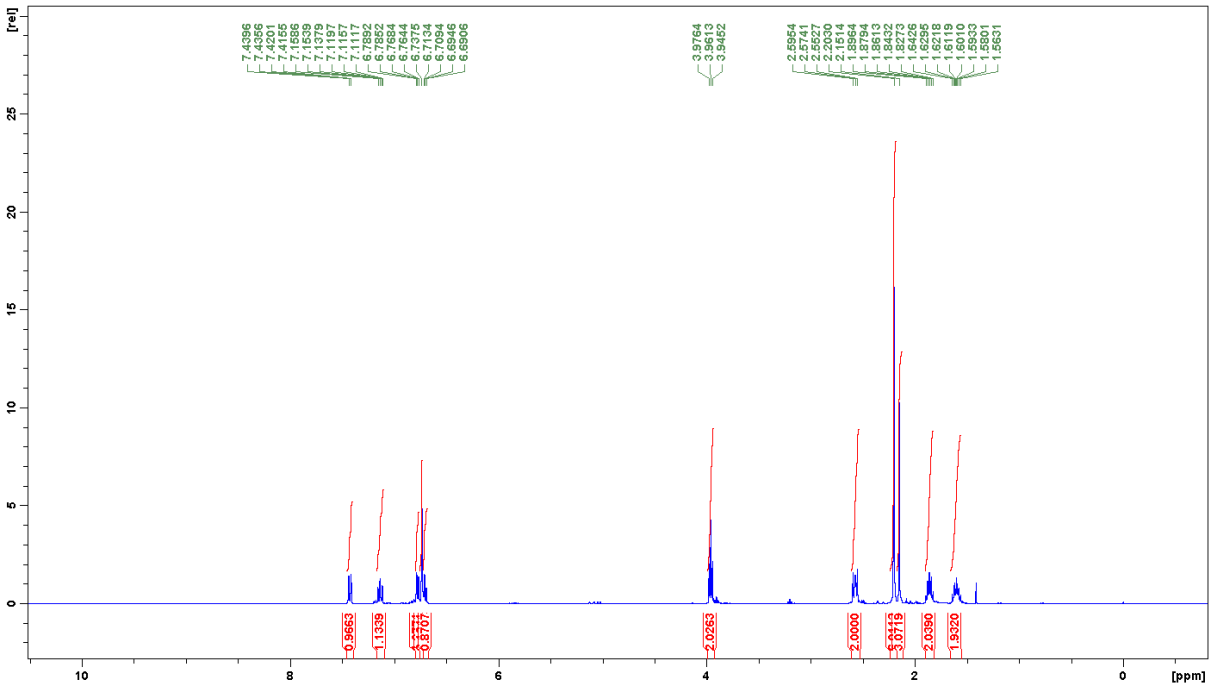
$CF_3(CF_2)_5(CH_2)_2$-Mes






Br
O
Mes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6.7852
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6.7644
6.7375
6.7334
6.7094
6.6946
6.6906
3.9764
3.9613
3.9462
2.5954
2.5741
2.5527
2.2030
2.1514
1.8964
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1.8613
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1.8273
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1.6218
1.6119
1.5910
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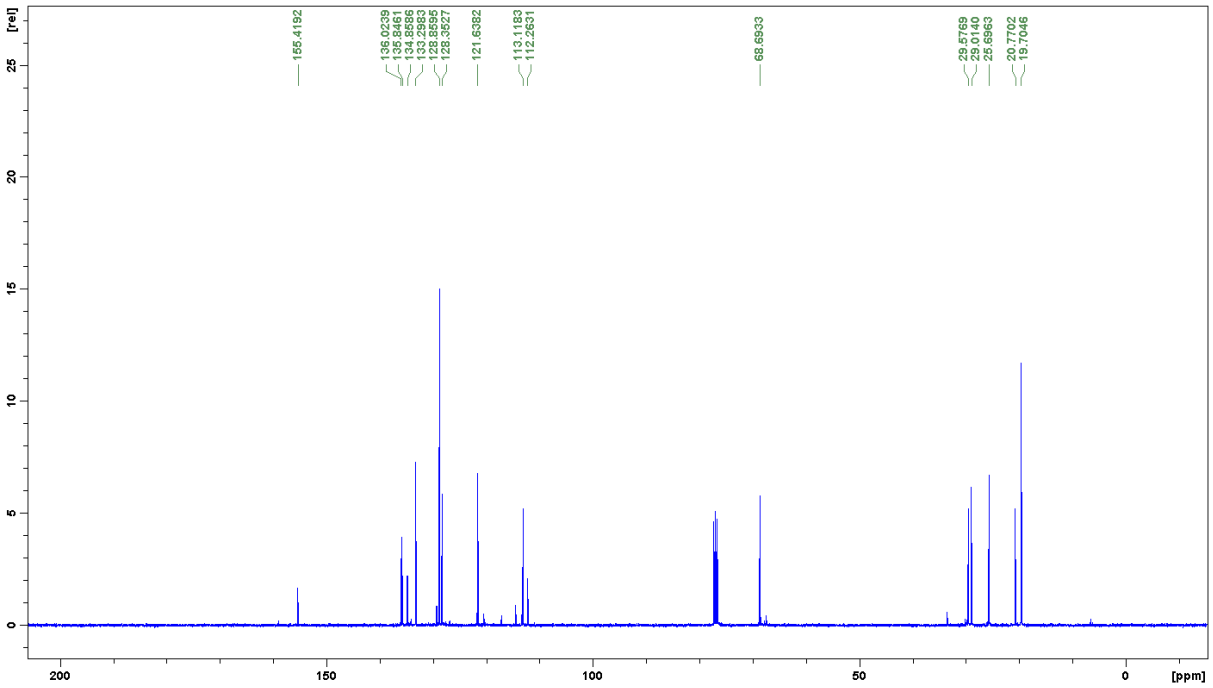
1.9320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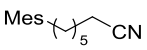
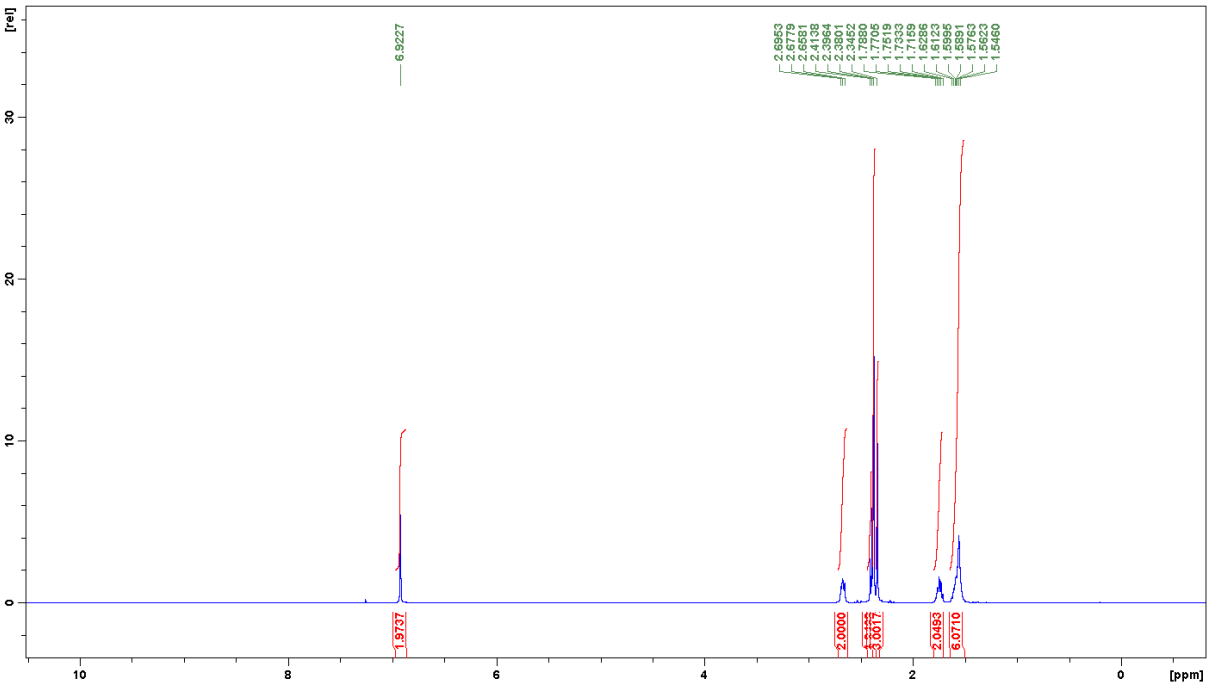
29.0140
25.6963
20.7702
19.7046


Mes
5
CN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2.3462
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1.7705
1.7519
1.7333
1.7159
1.6286
1.6123
1.5995
1.5891
1.5763
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1.5460
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2.0493
6.0710
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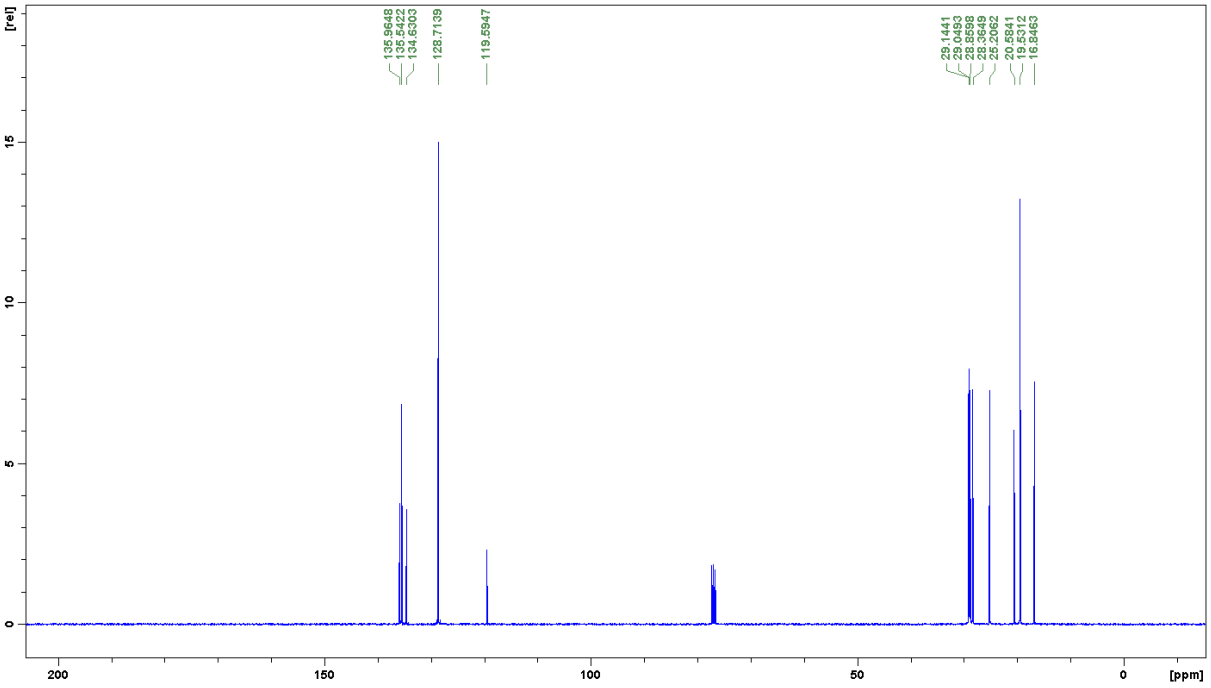
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16.8463
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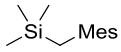
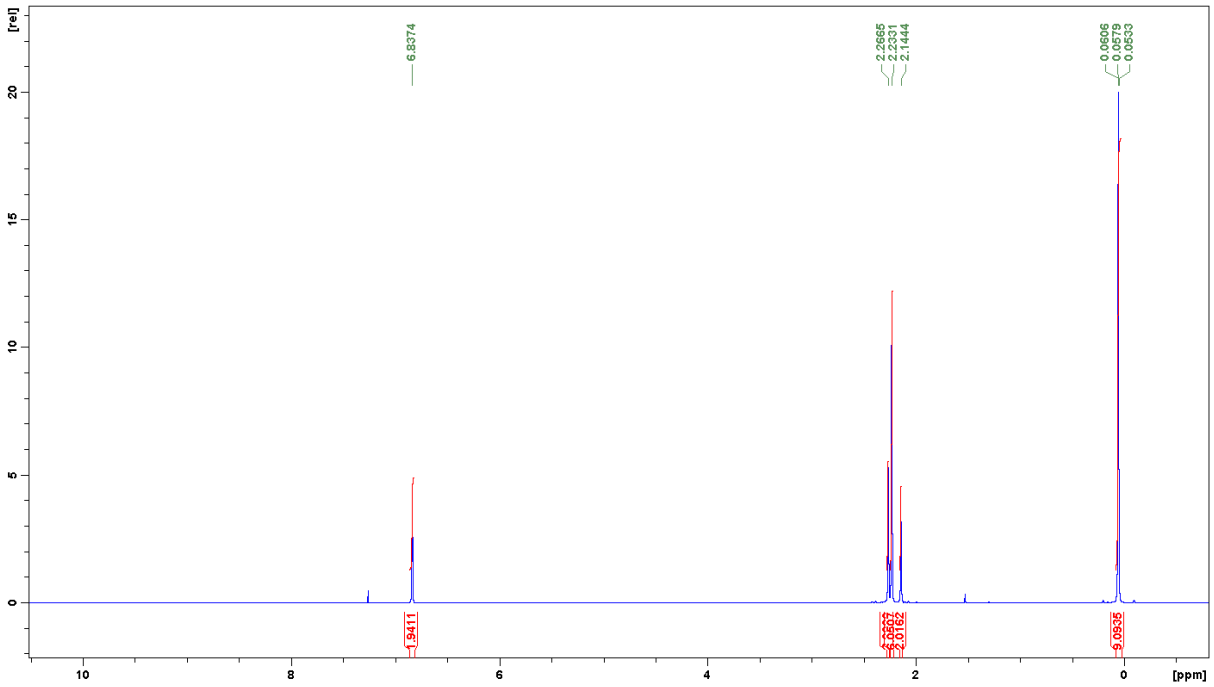

Si
Mes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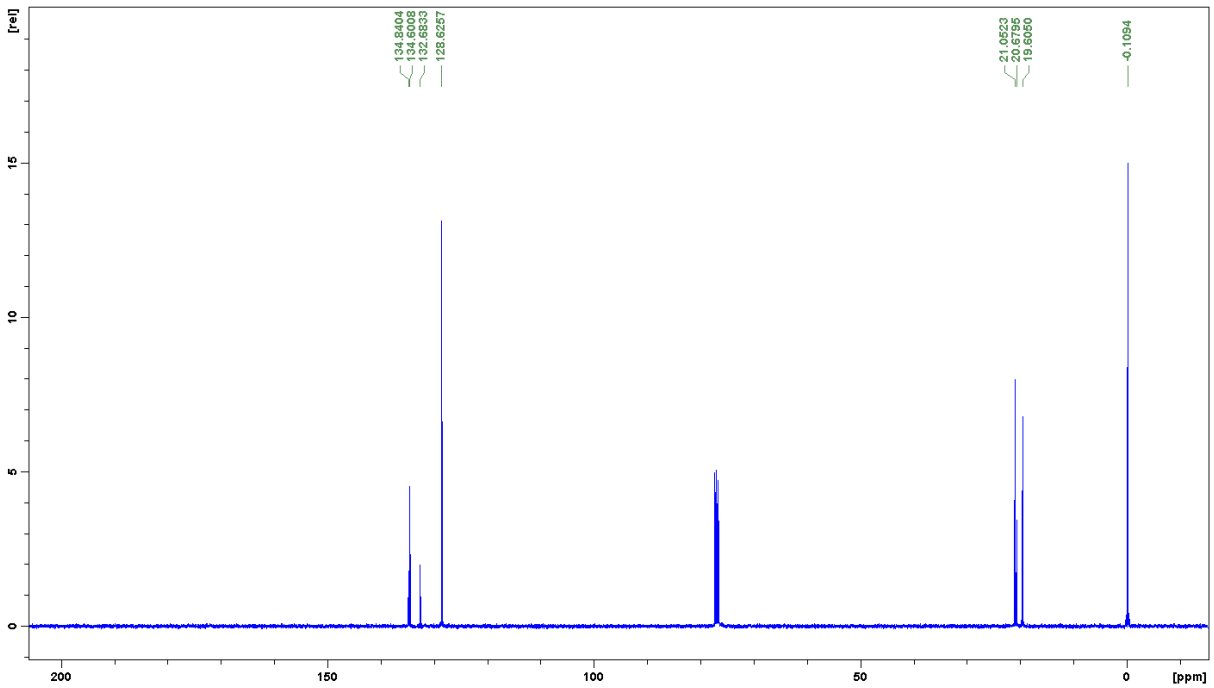
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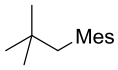
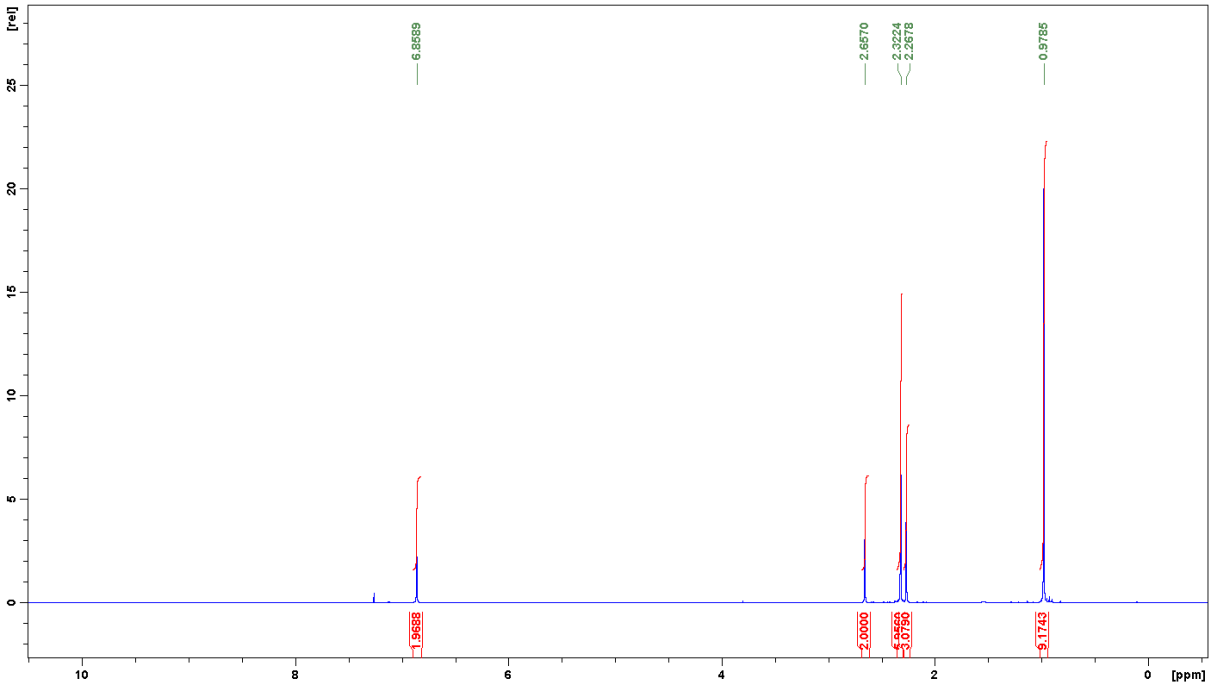

Mes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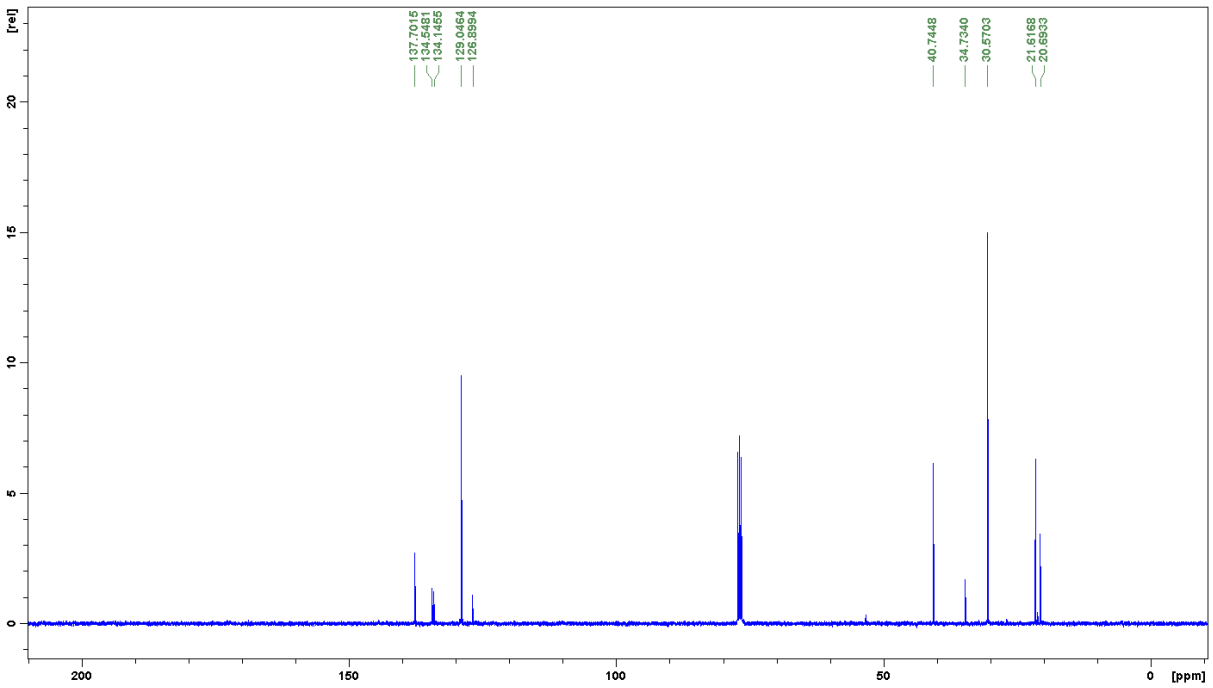
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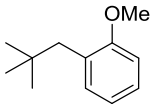
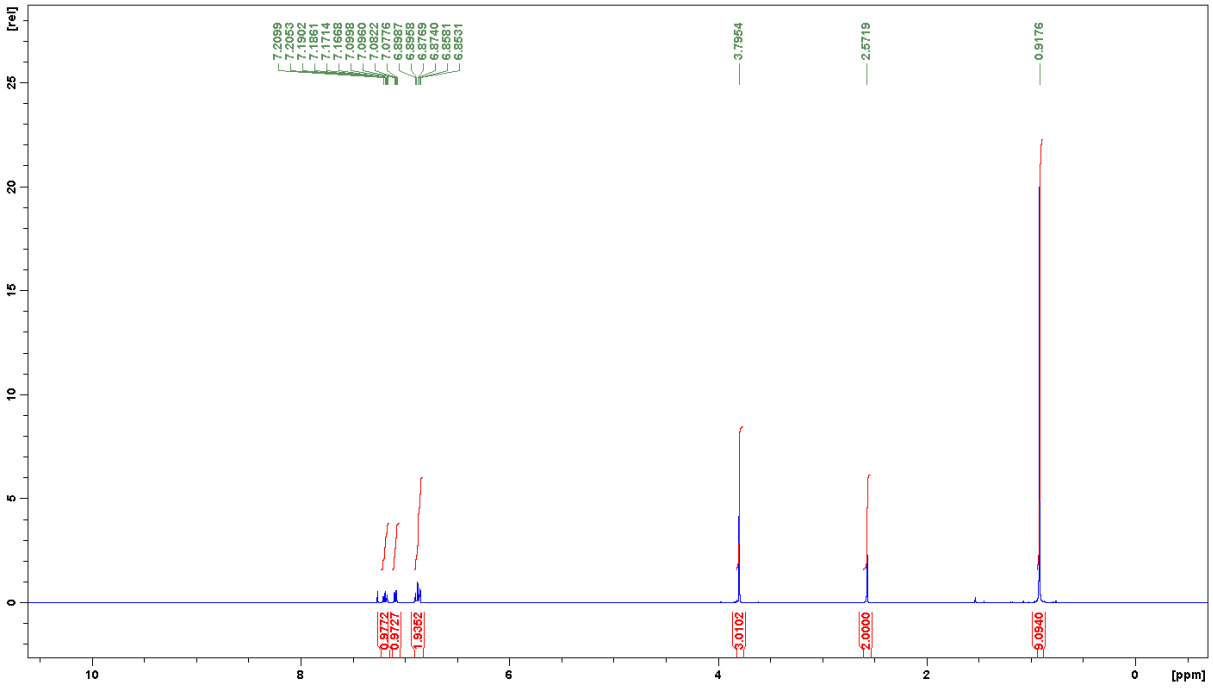

OMe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6.8958
6.8769
6.8740
6.8581
6.8531
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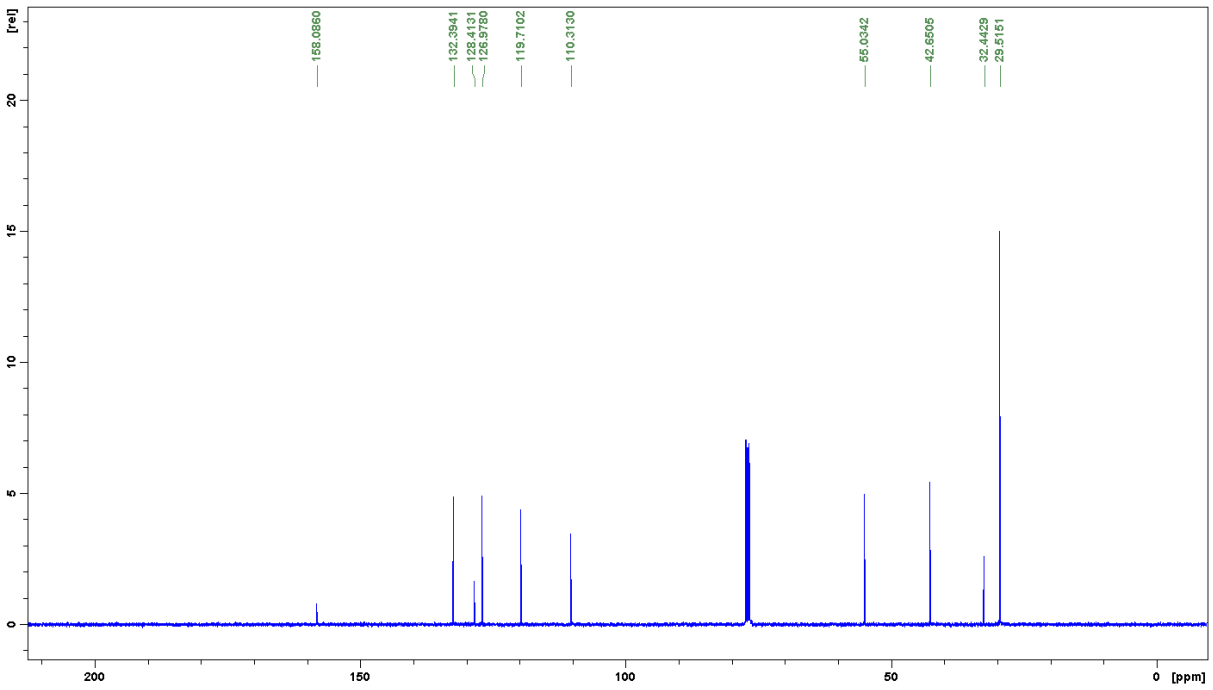
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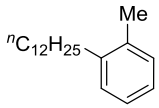
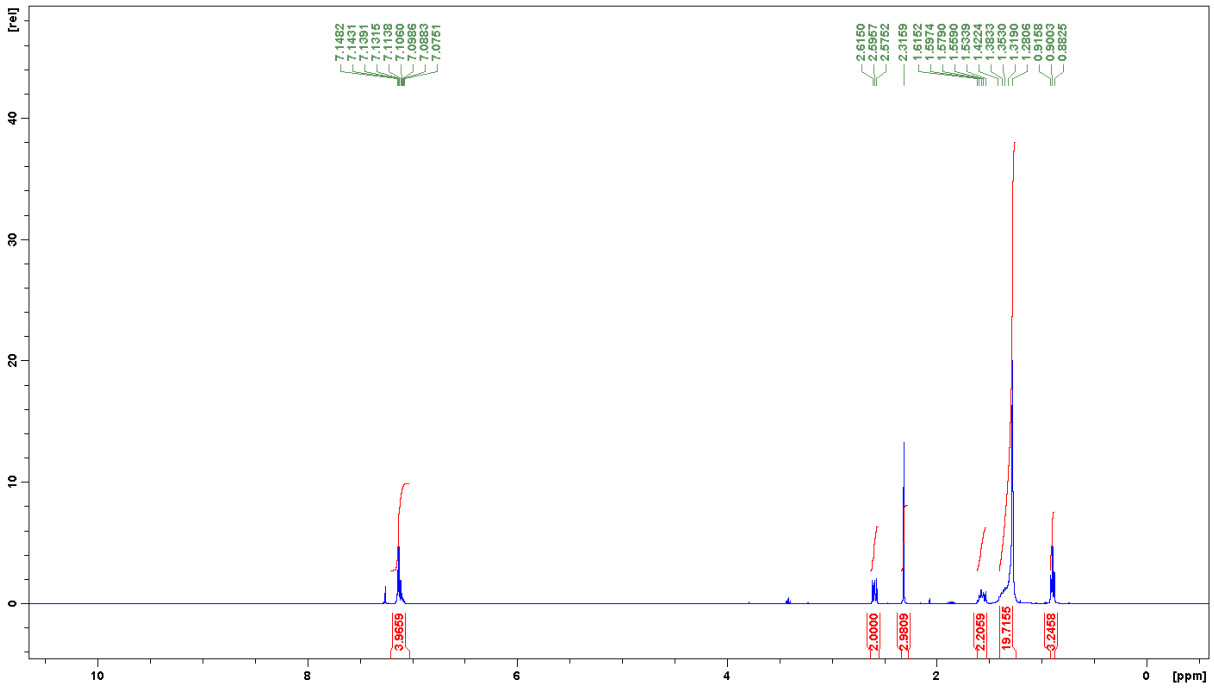

Me
nC12H25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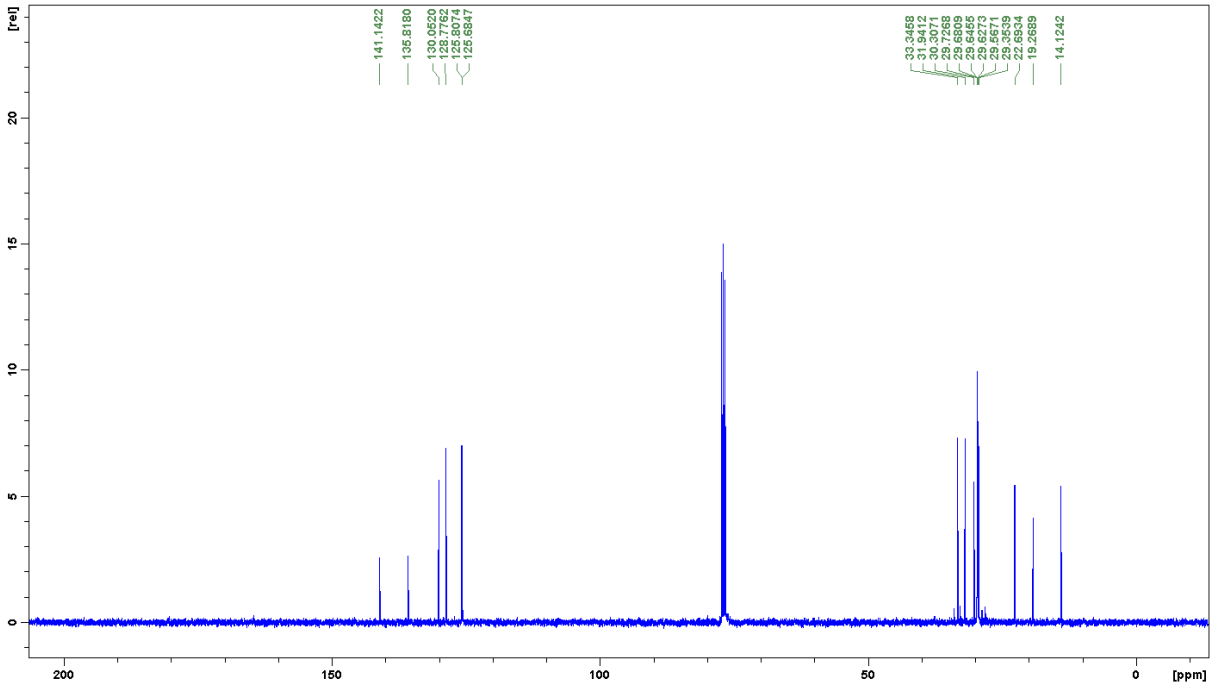
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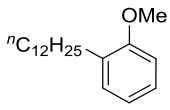
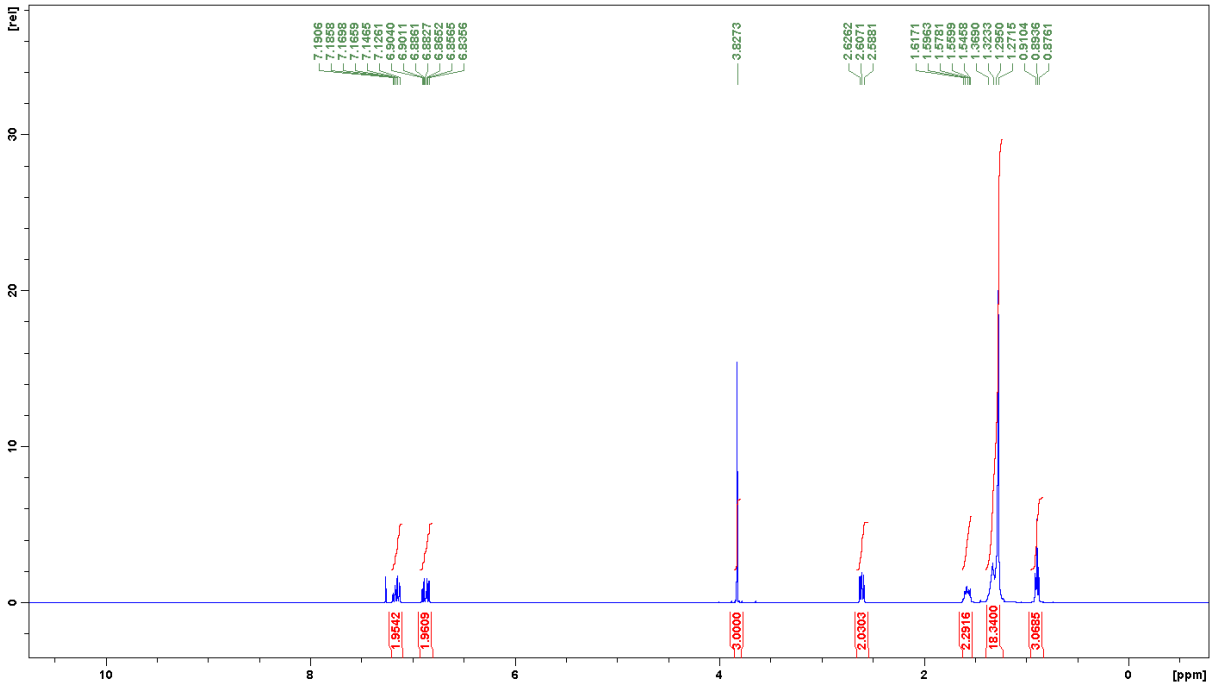

OMe
$^{n}C_{12}H_{25}$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6.8356
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1.5781
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1.3353
1.2950
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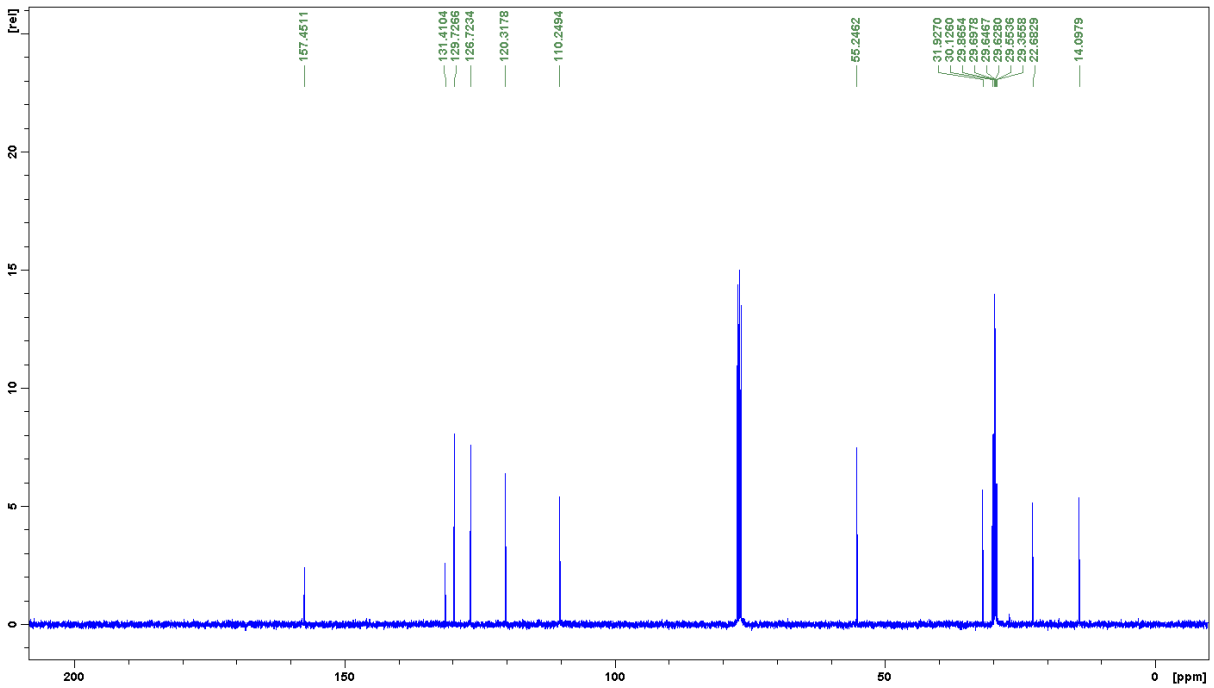
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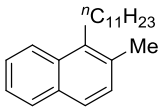
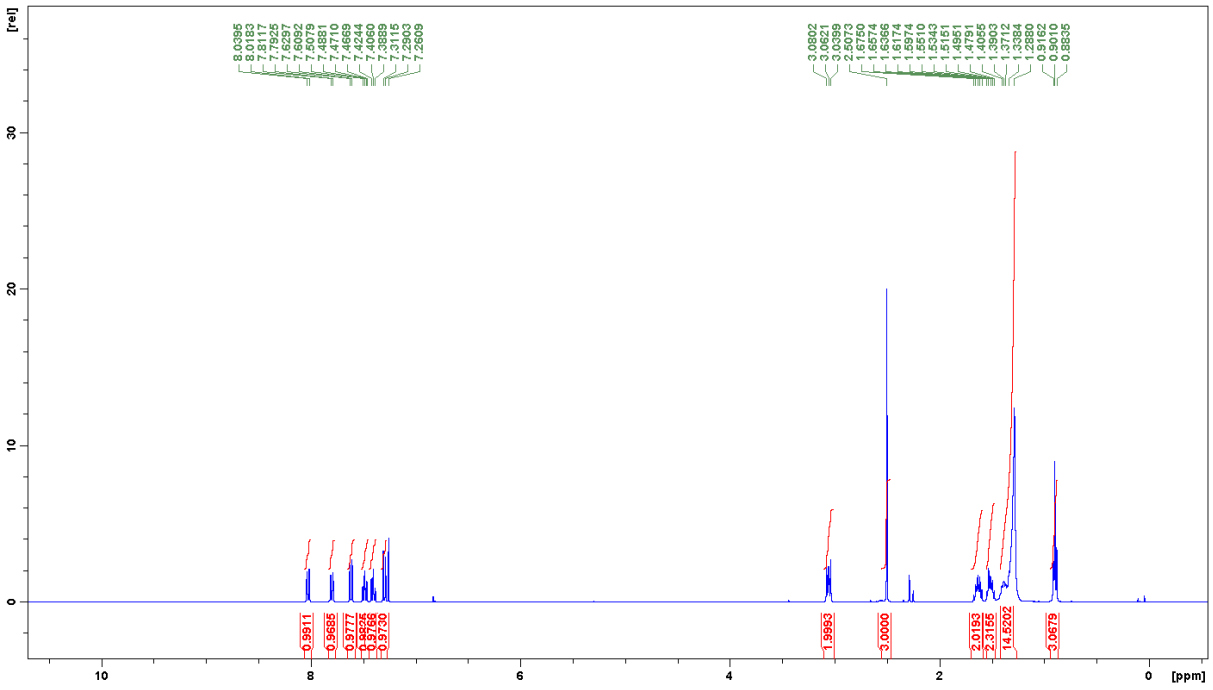
29.6280
29.5536
29.3558
22.6829
14.0979
[ppm]


$^{n}C_{11}H_{23}$
Me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7.3115
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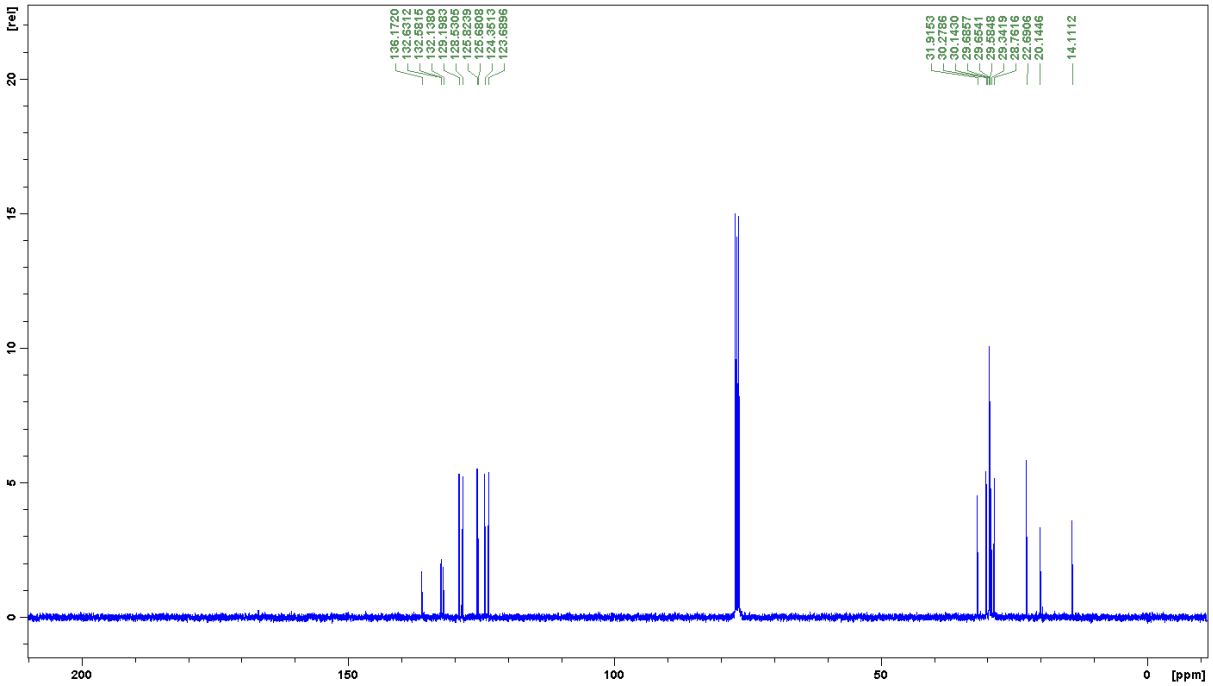
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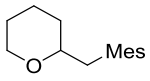
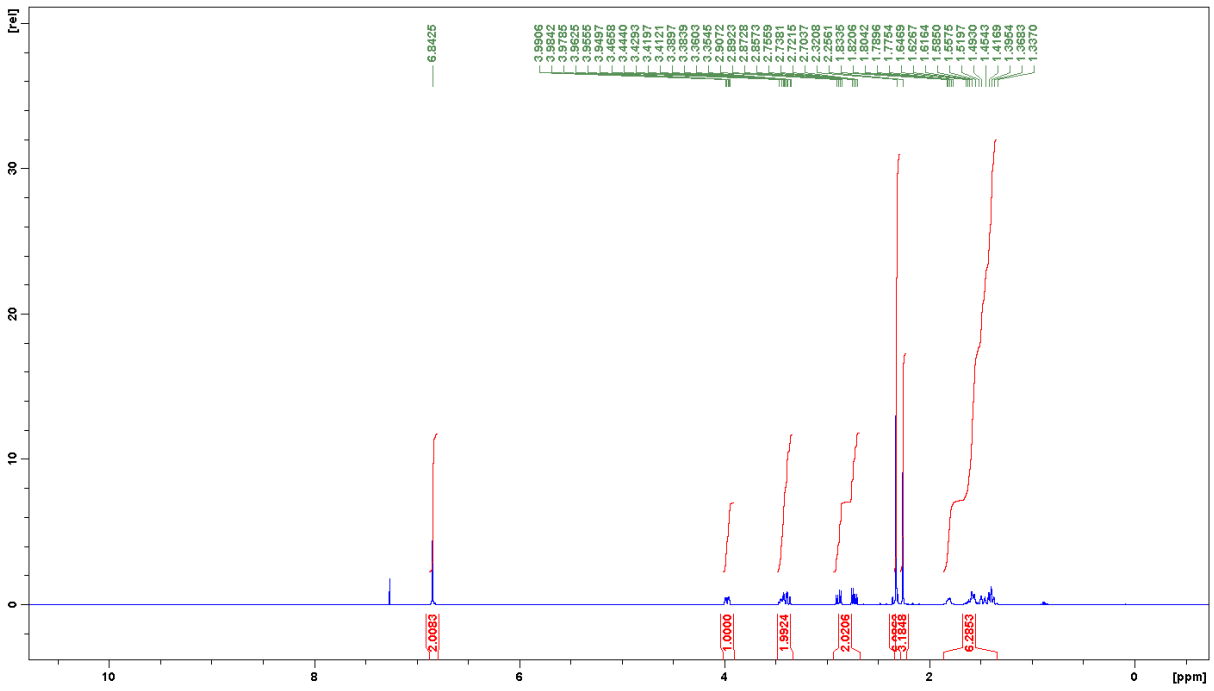

Mes
O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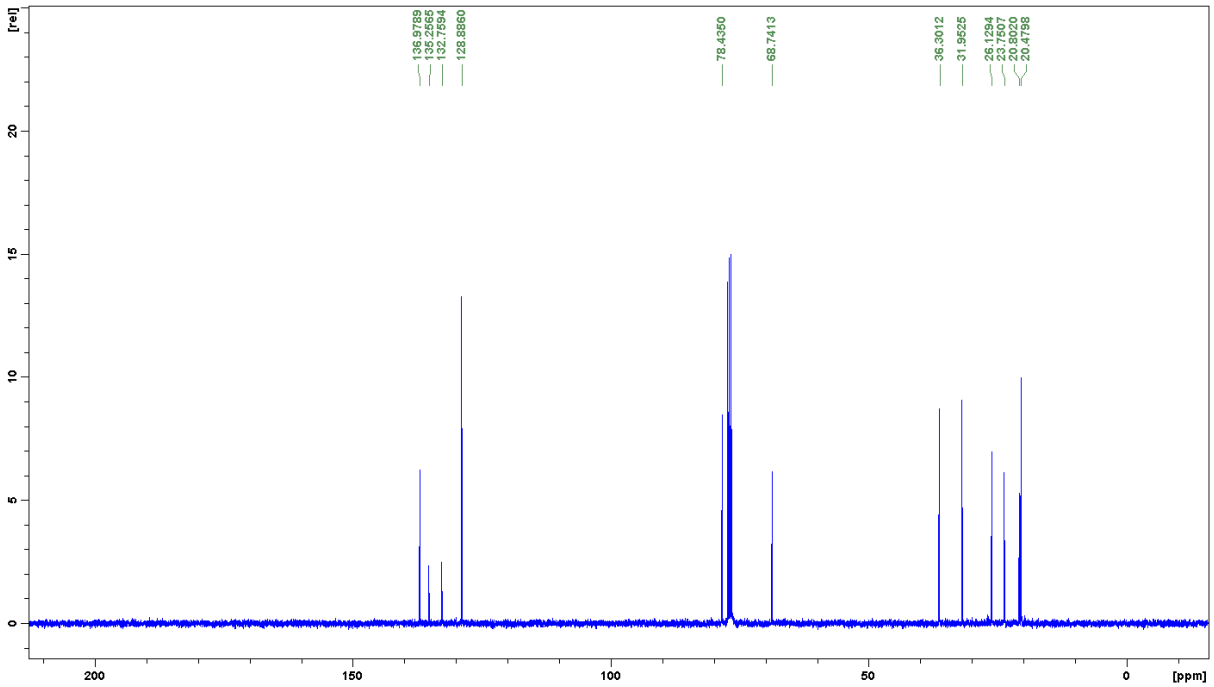
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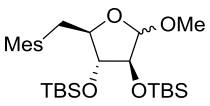
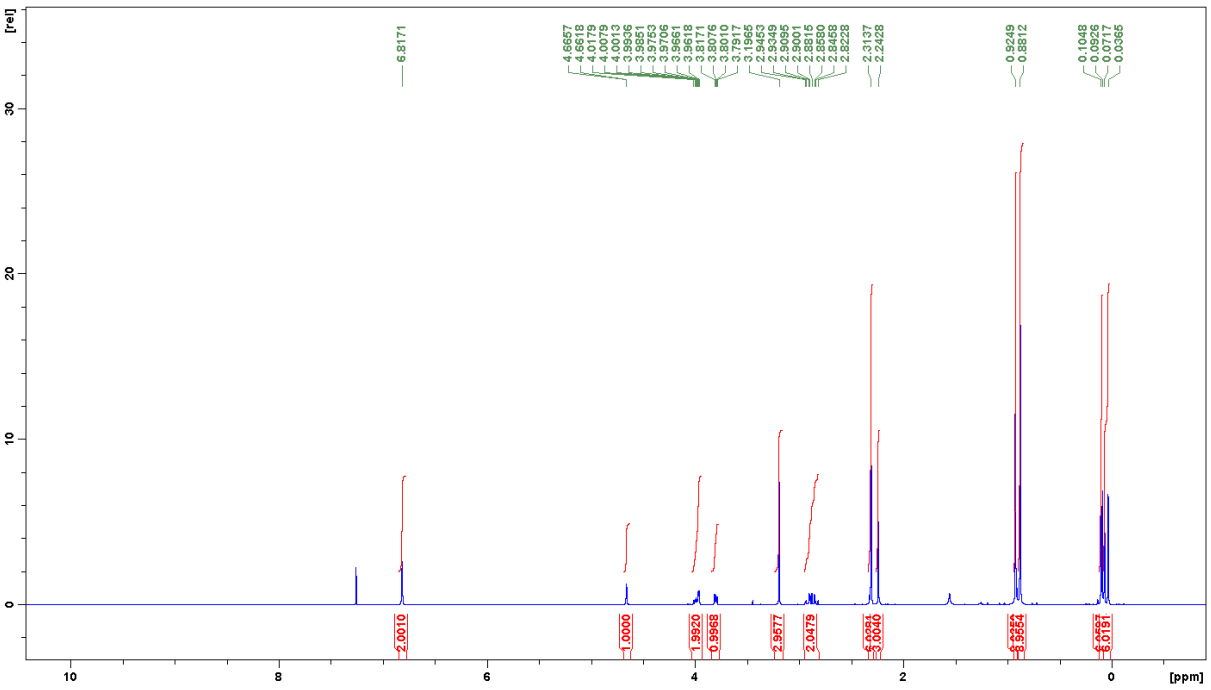
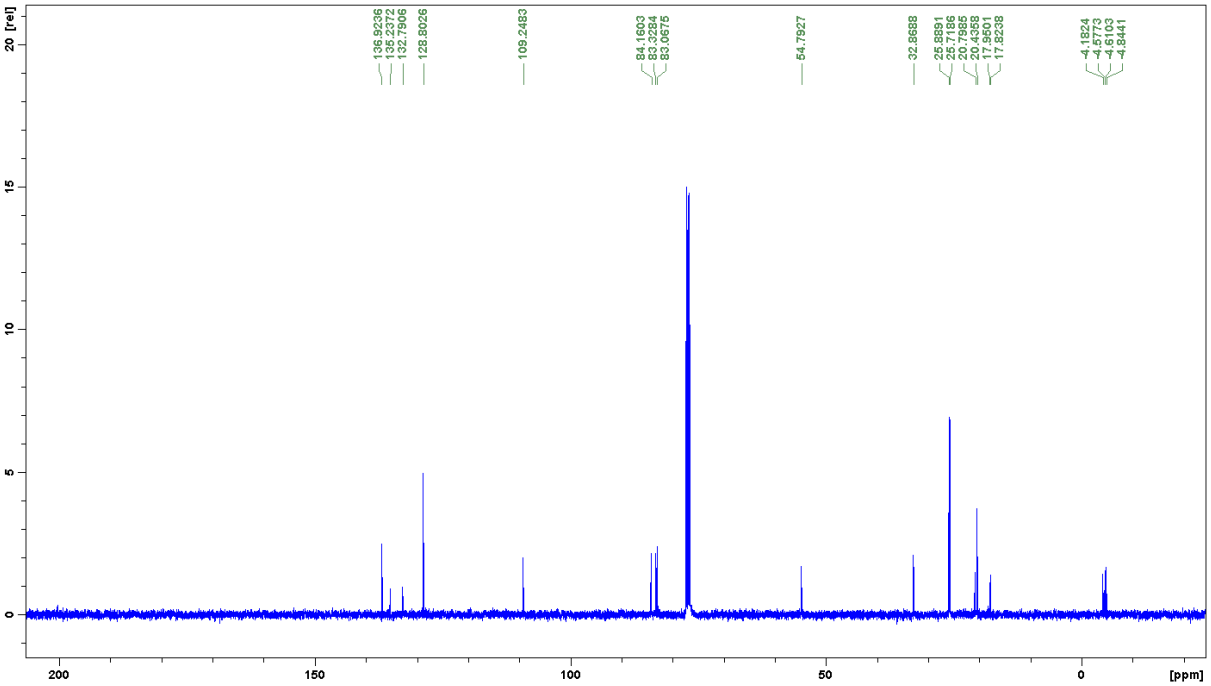
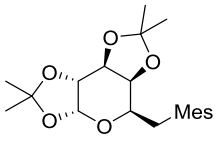


(major anomer)



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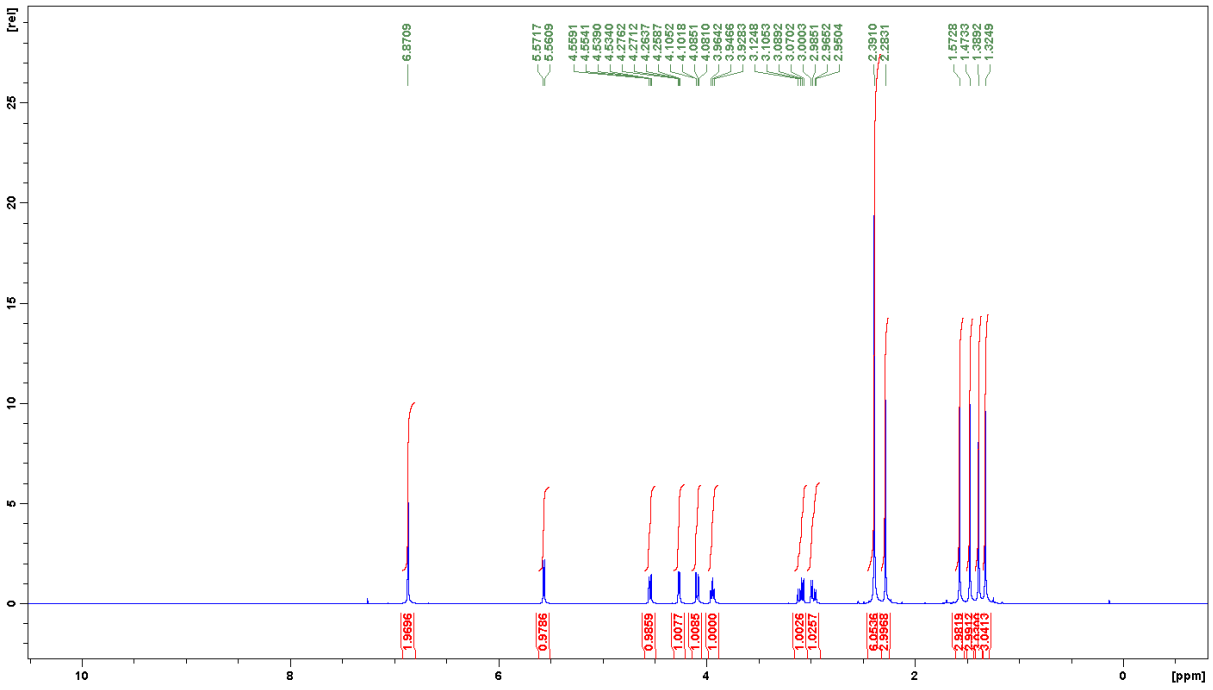
O
O
O
Mes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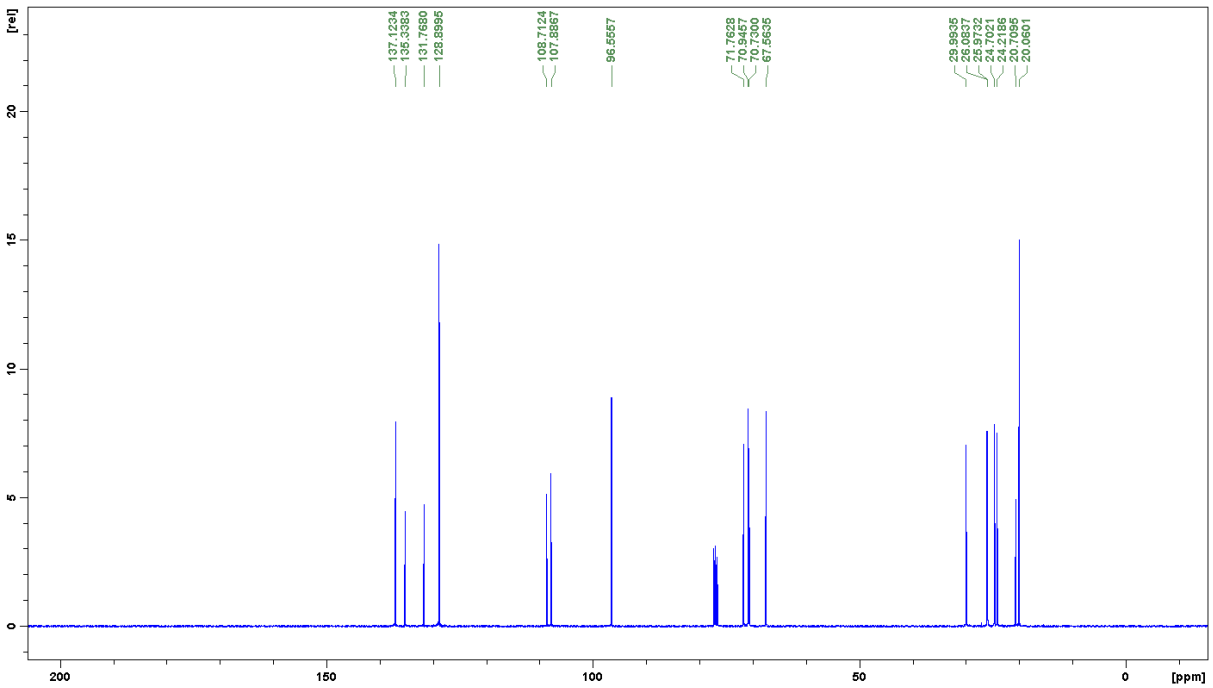
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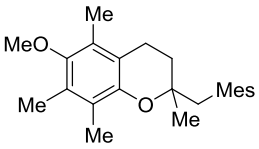
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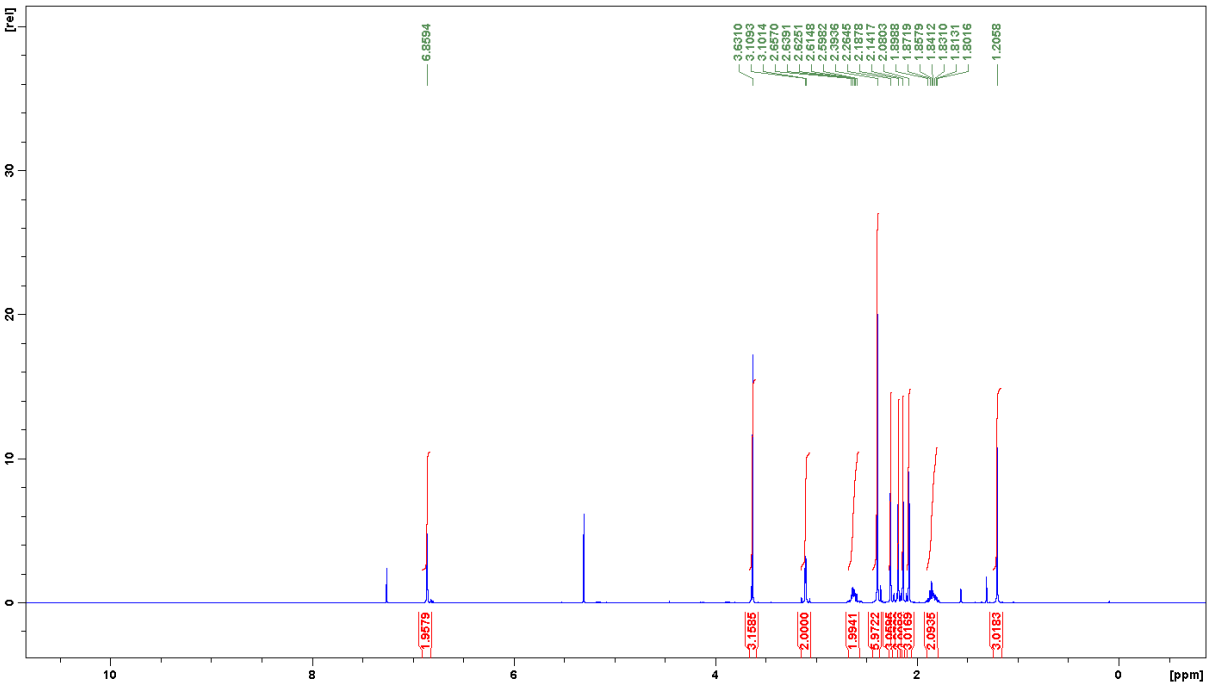
Me
O
Mes
Me
Me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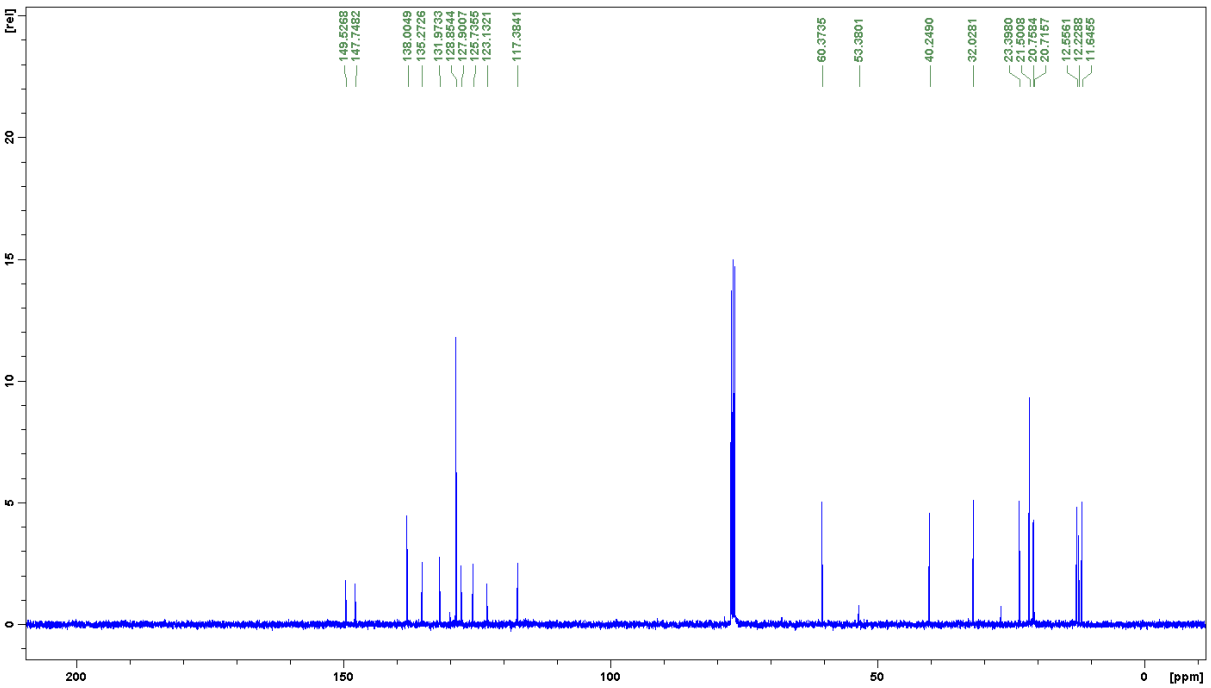
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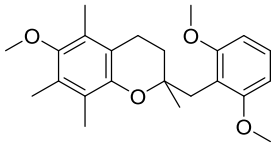

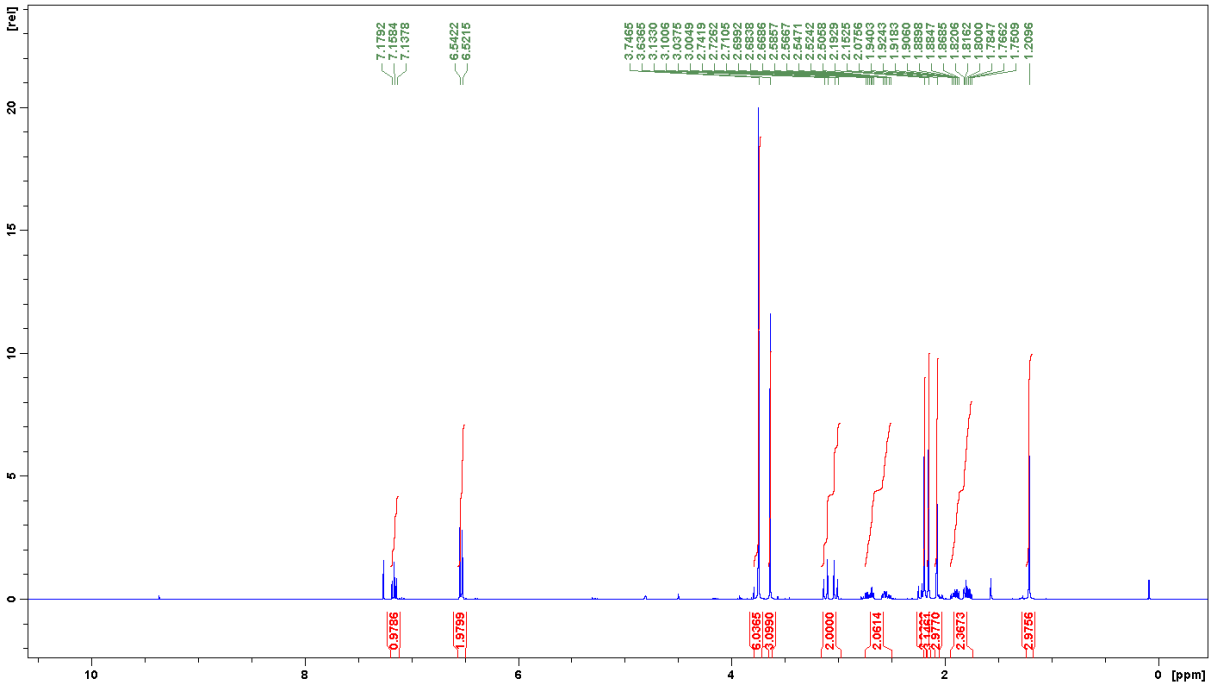
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2.6838
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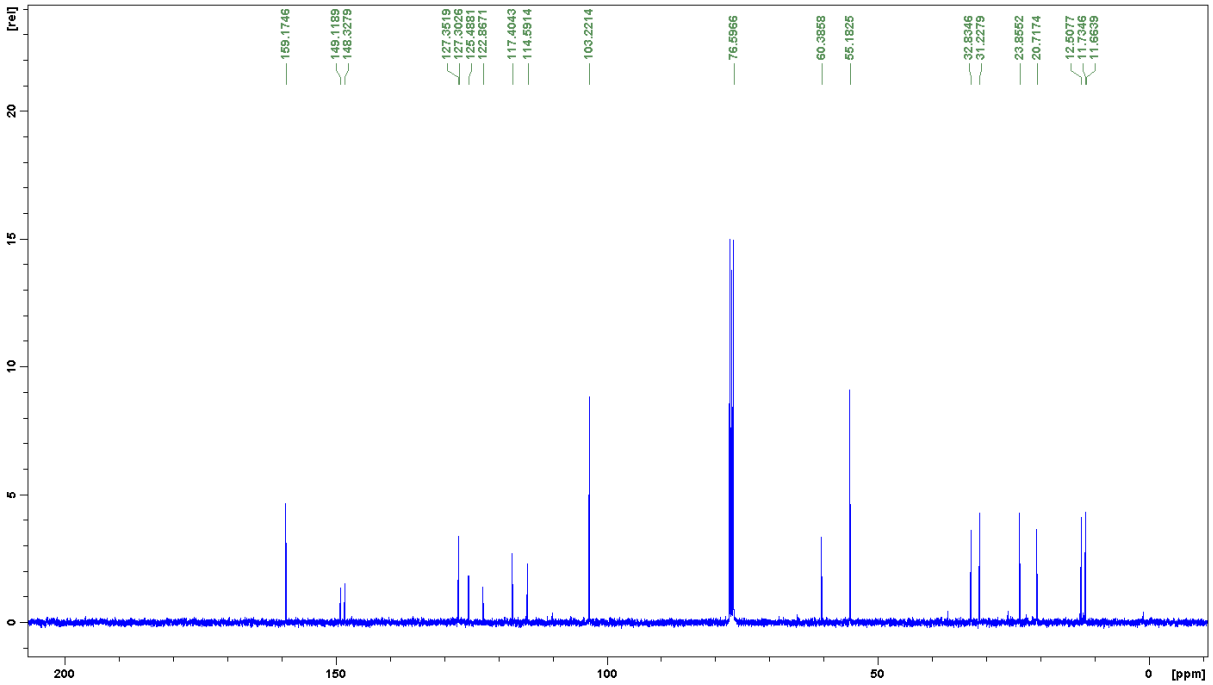
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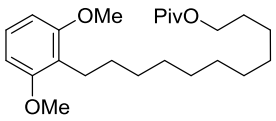
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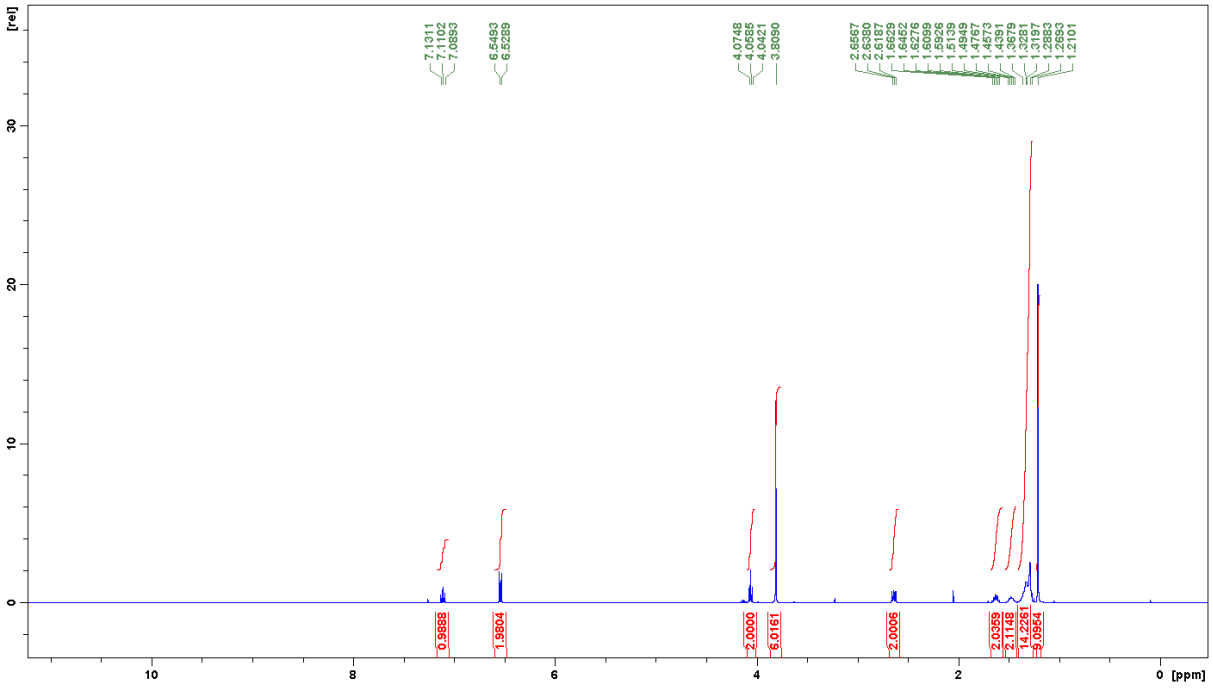
PivO
OMe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1.6099
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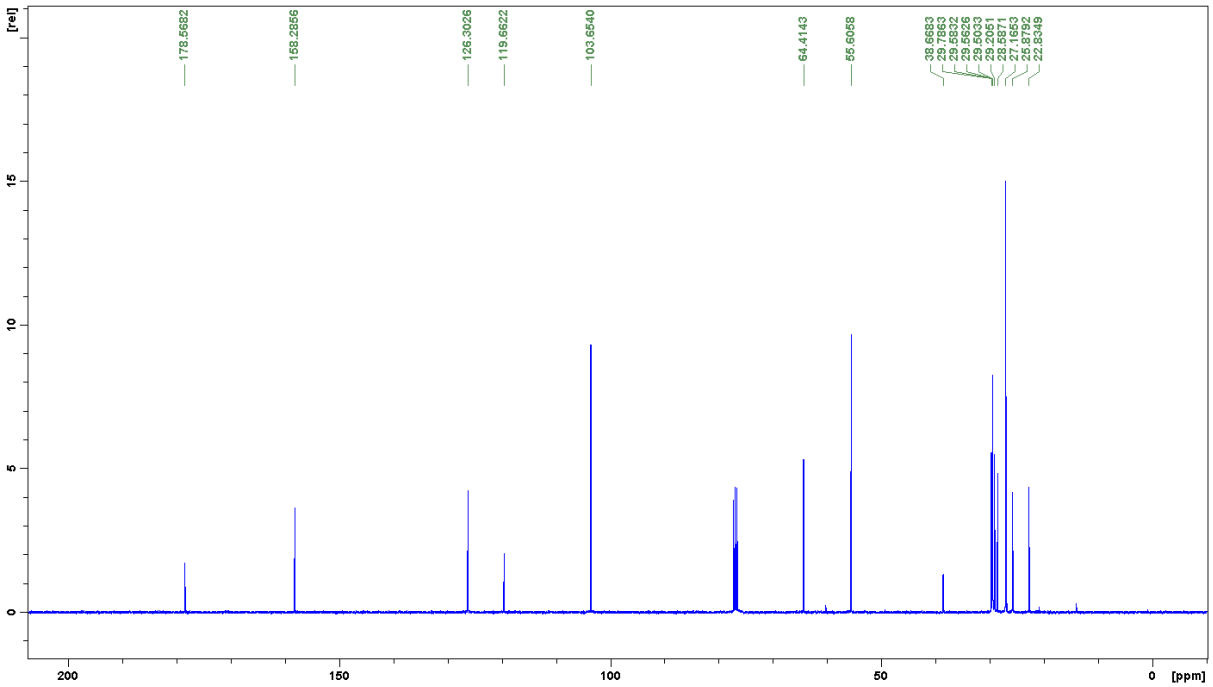
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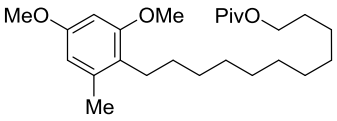
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OMe
PivO
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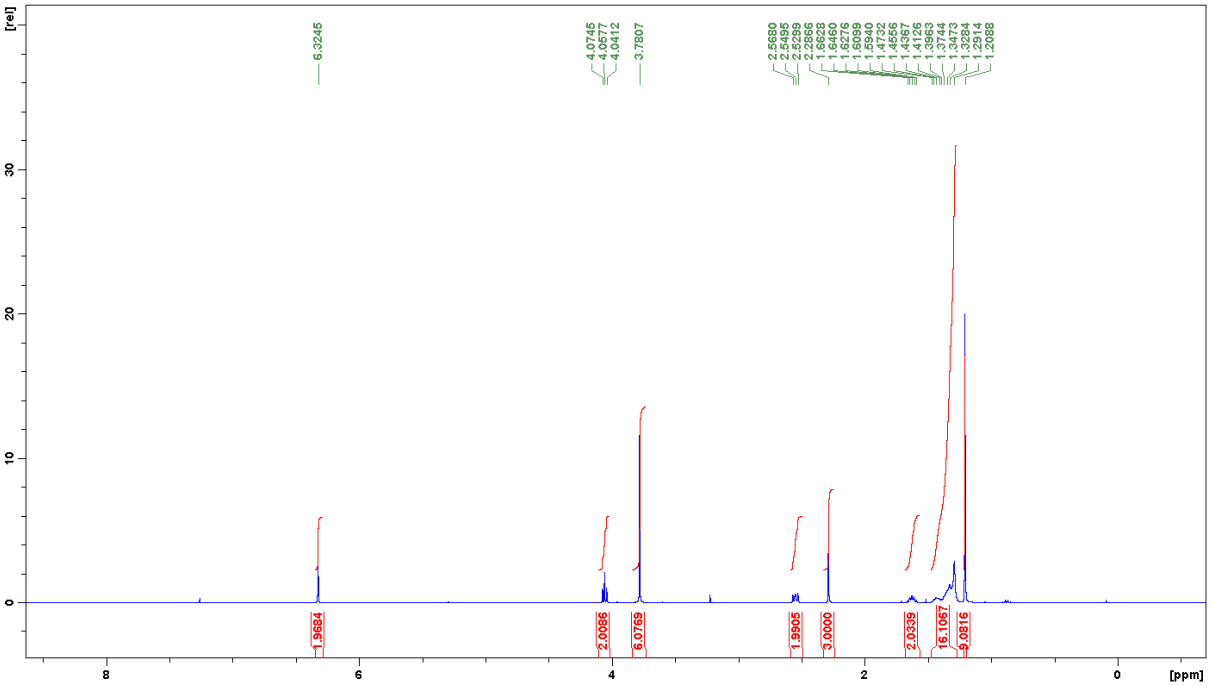
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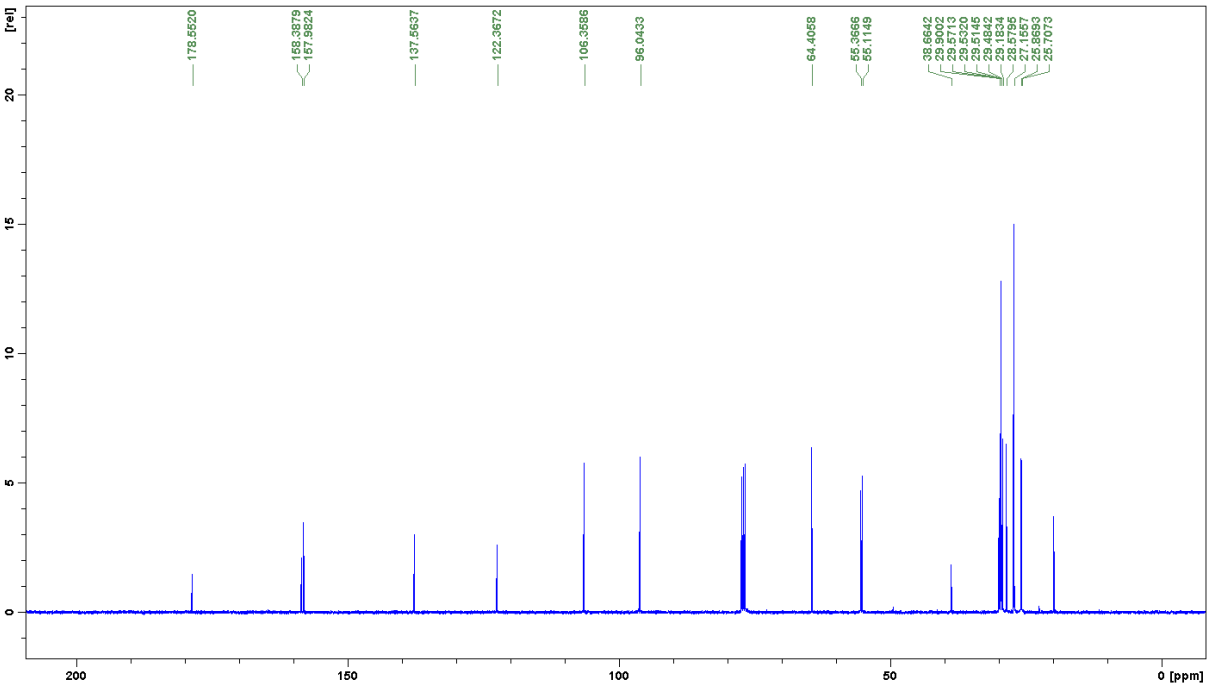
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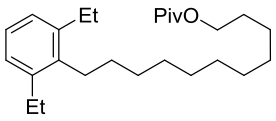
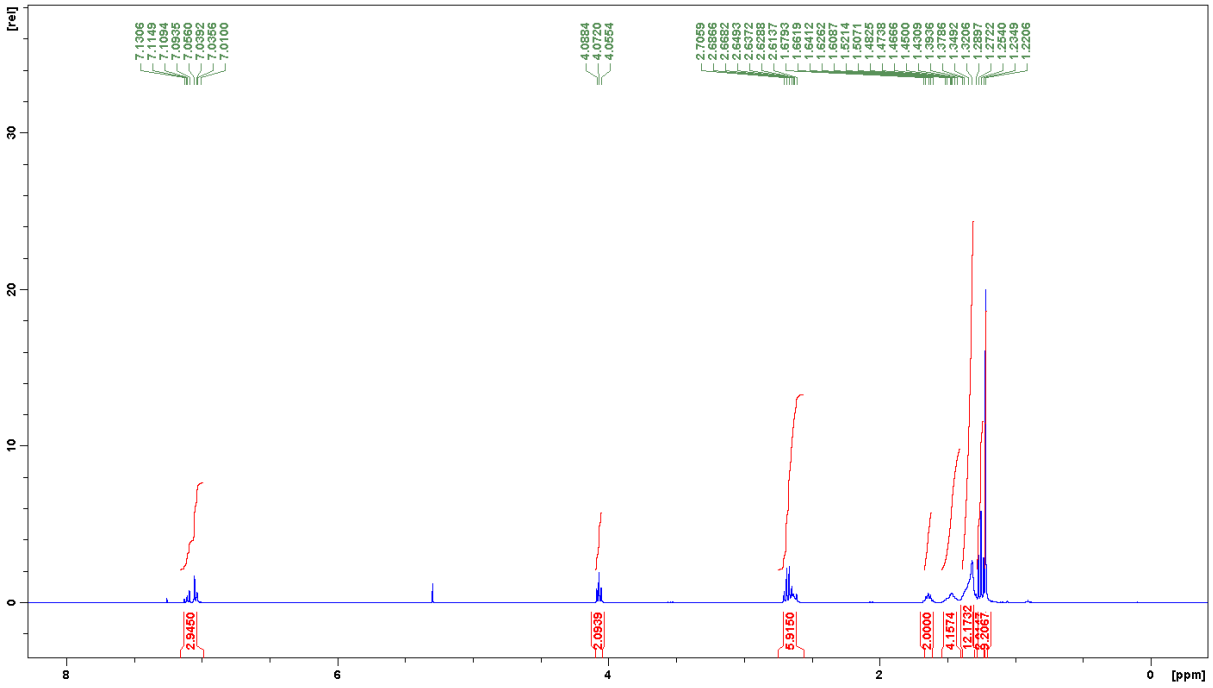

Et
PivO
Et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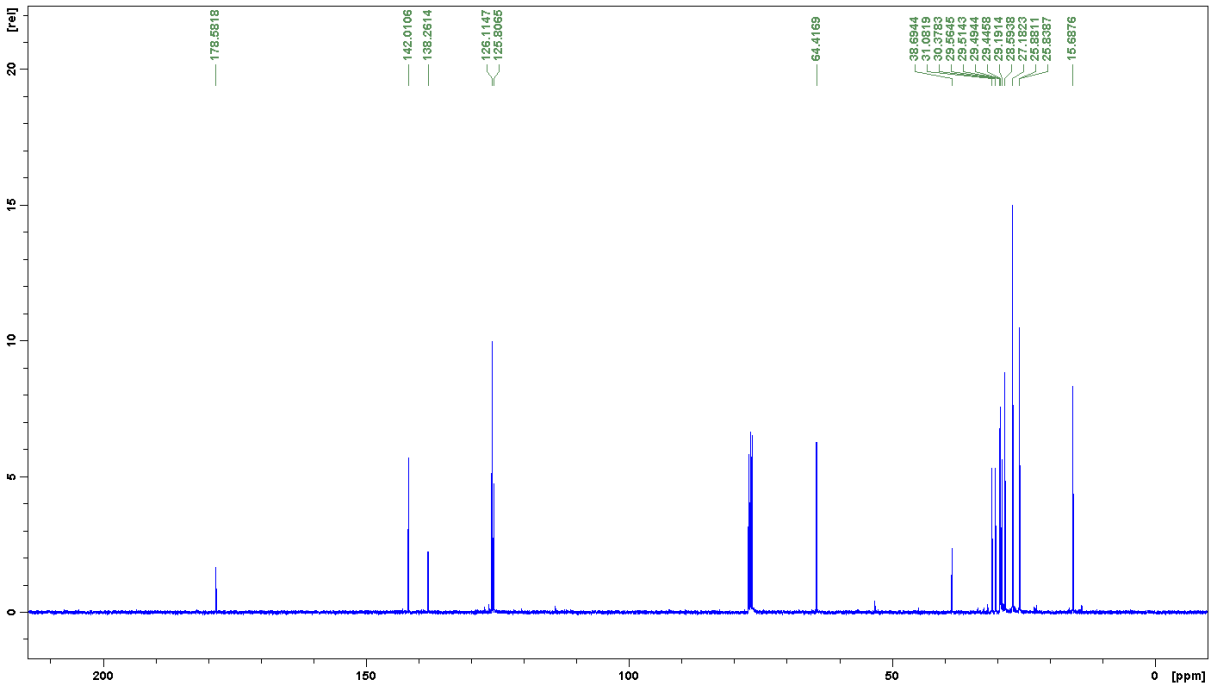
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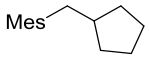
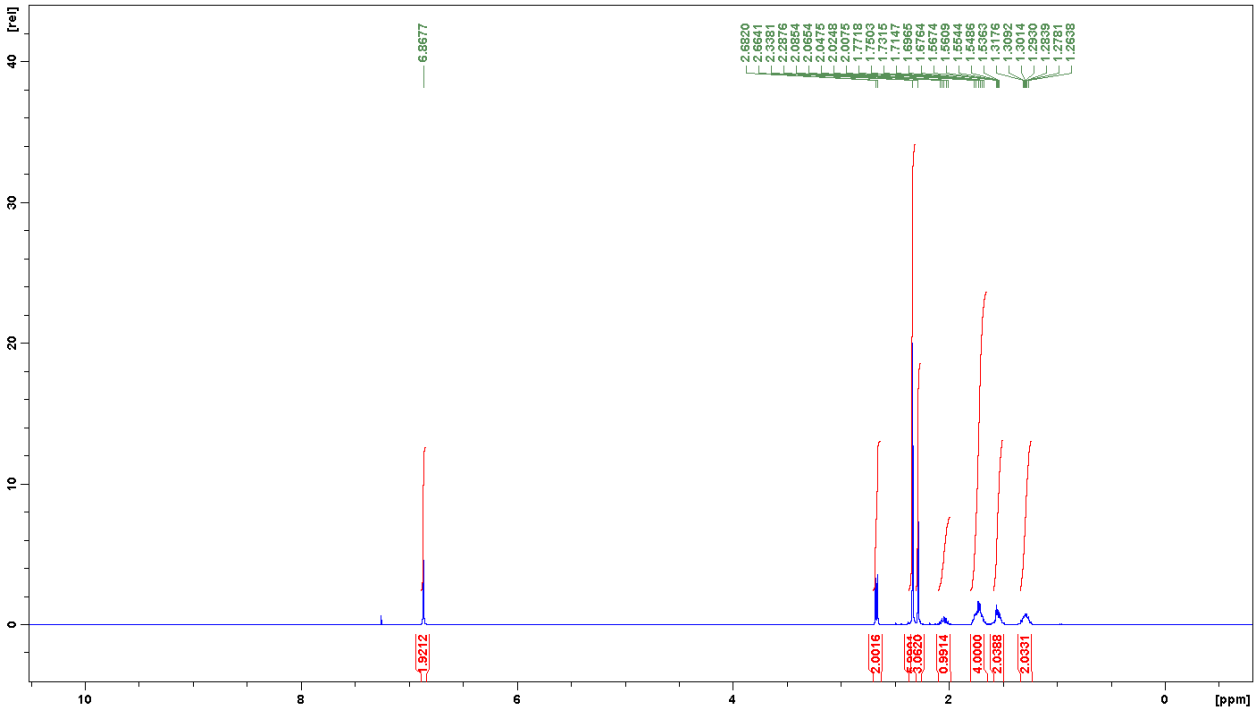

Mes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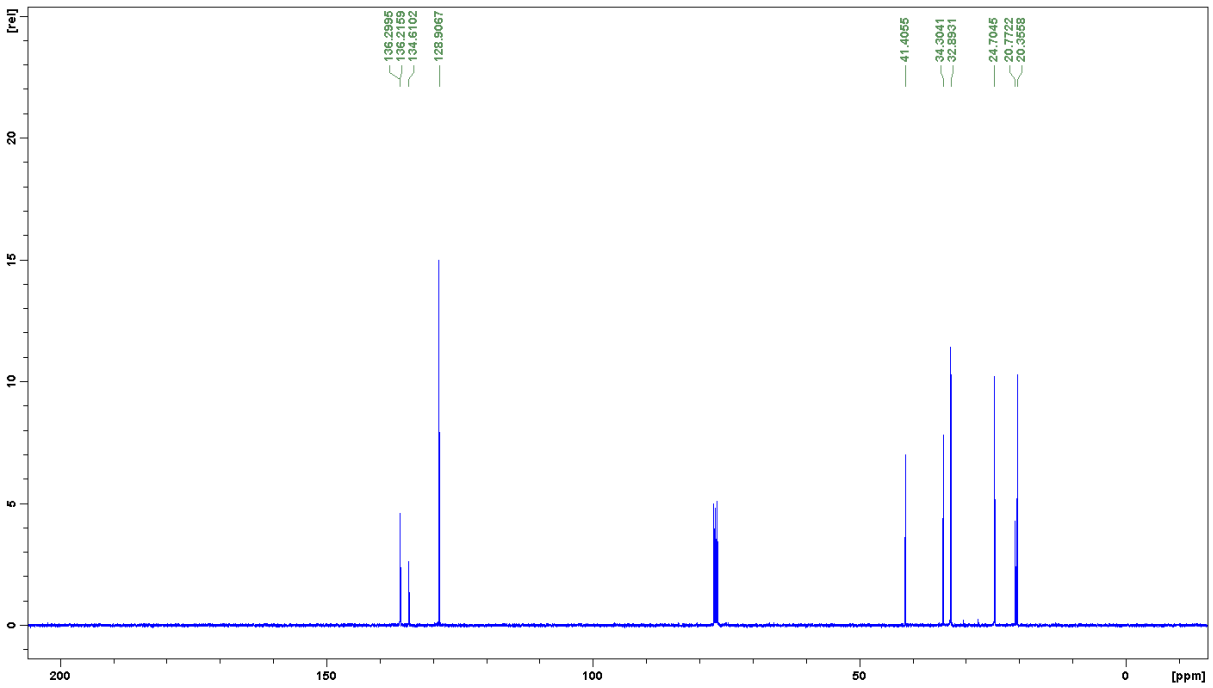
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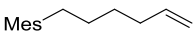
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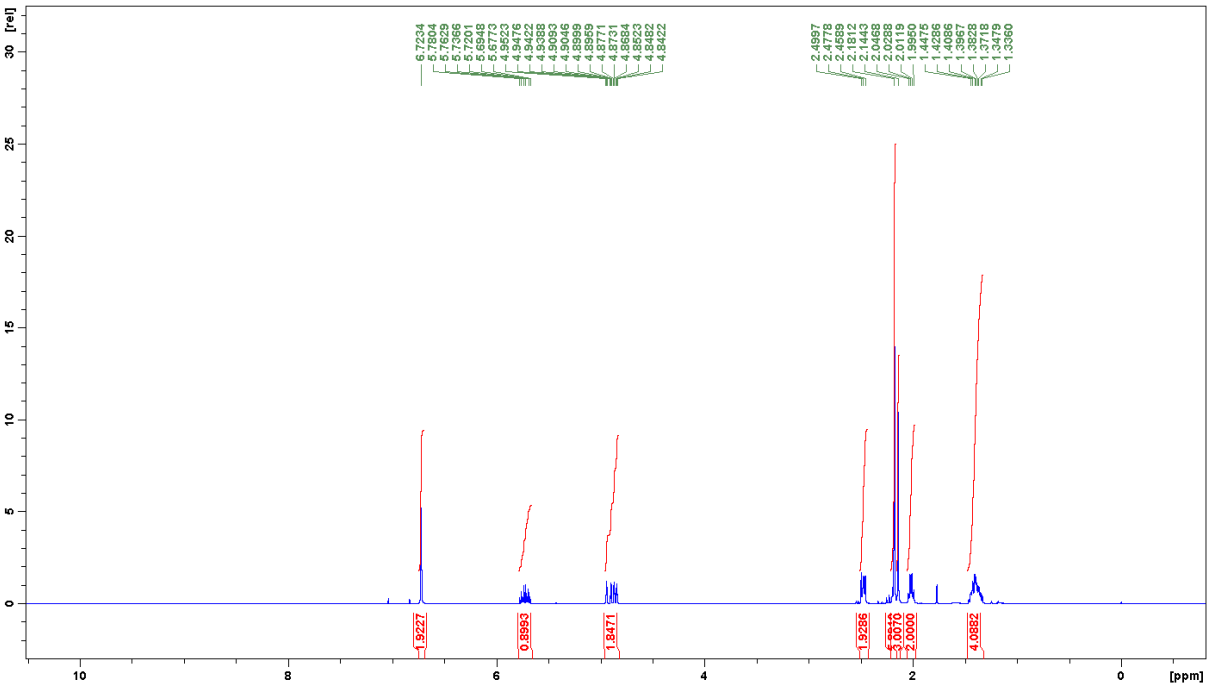
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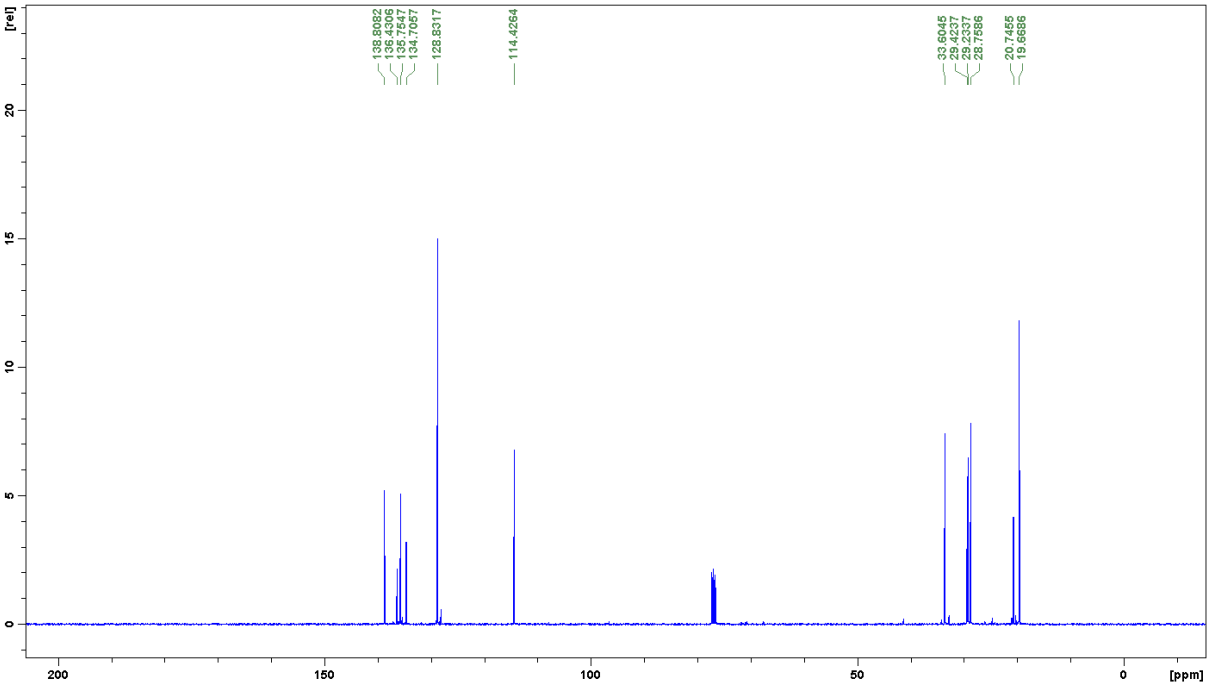
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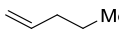
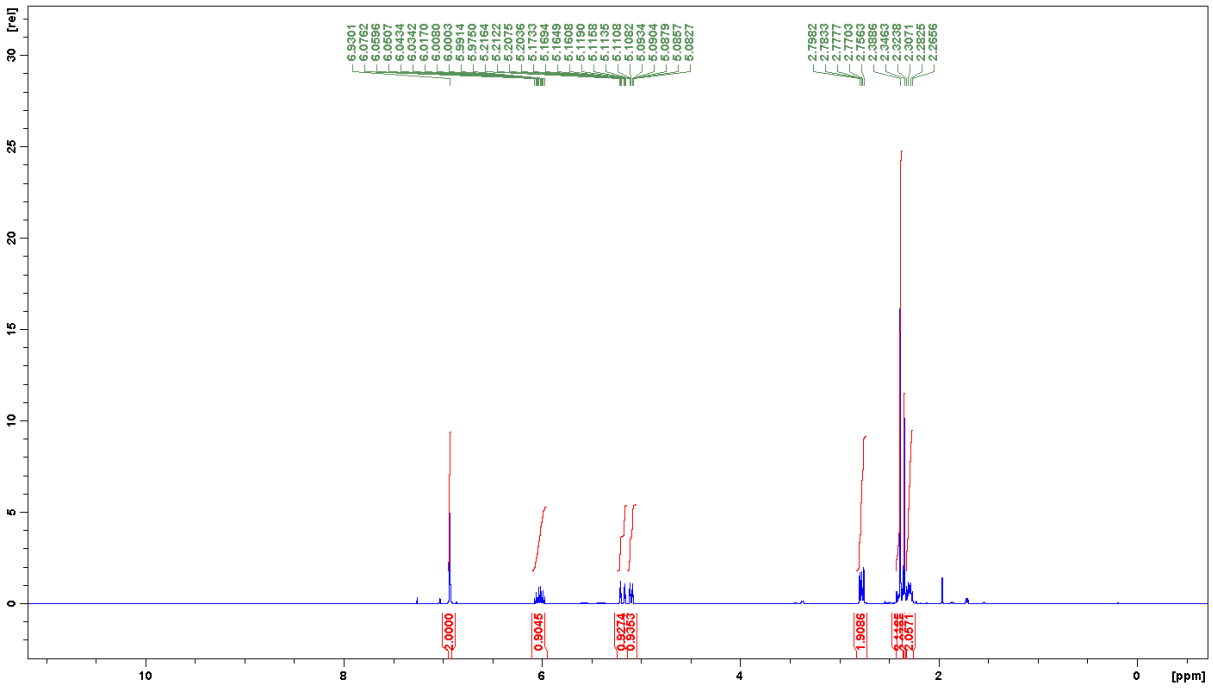

Mes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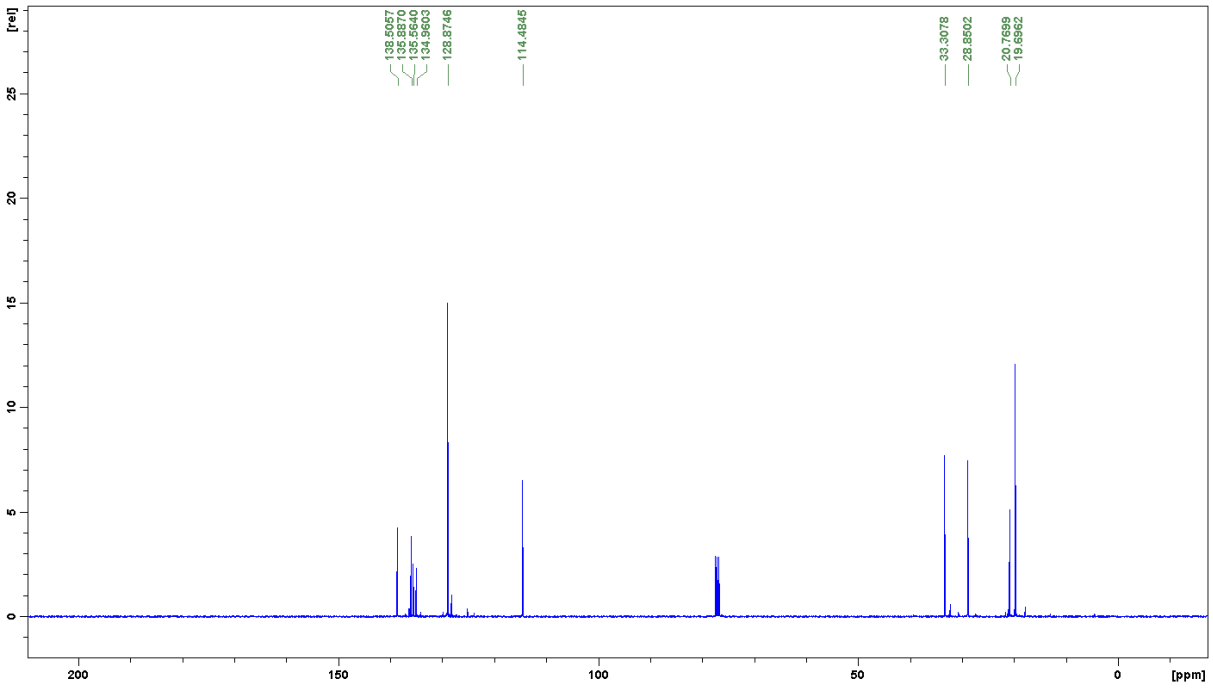
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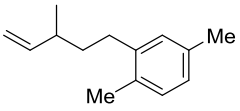
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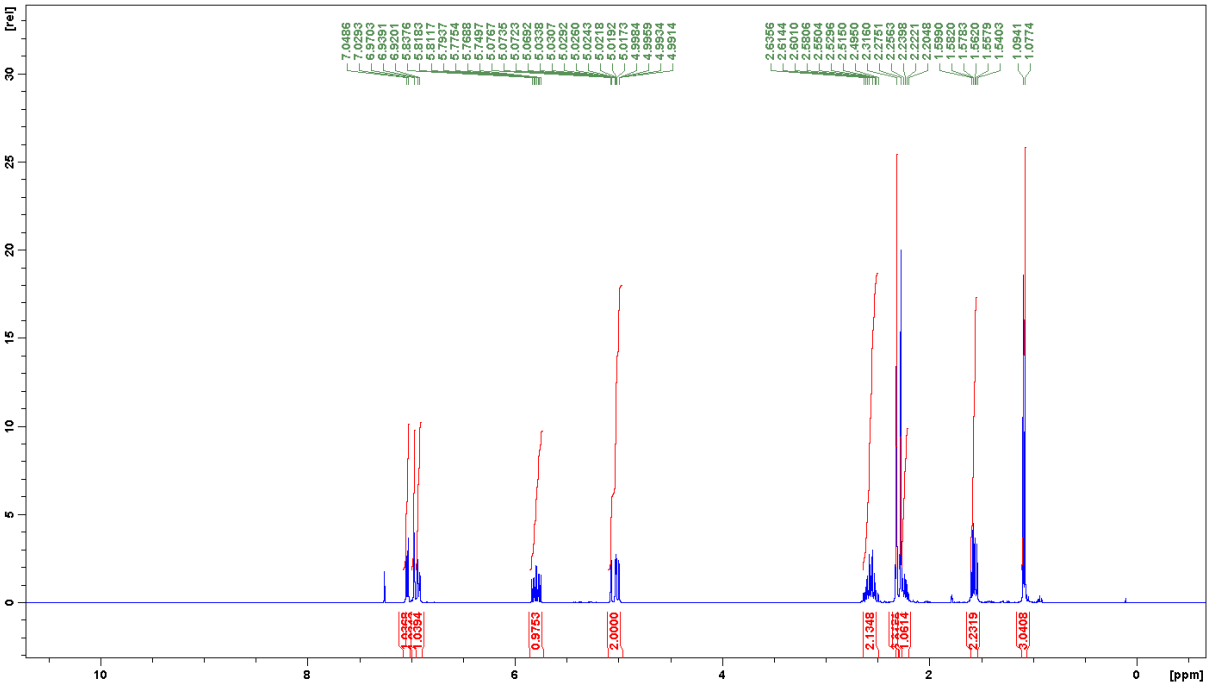
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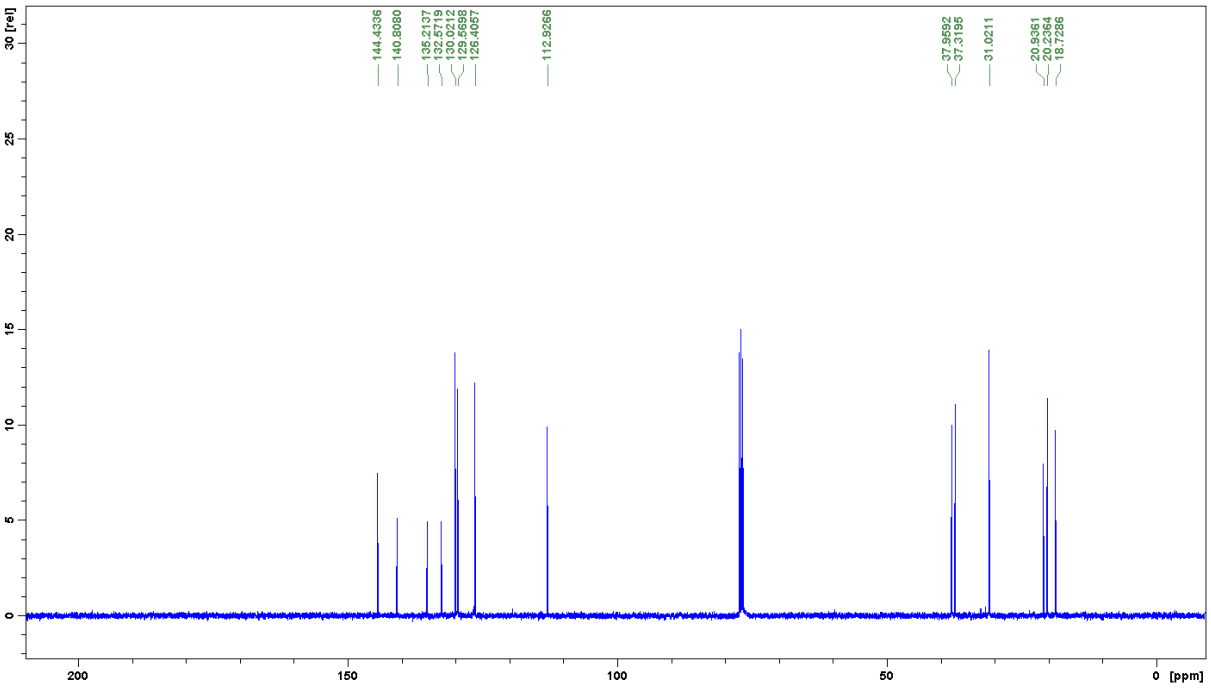
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