

SUPPORTING INFORMATION

Solvent-free reduction of carboxylic acids to alcohols with NaBH₄ promoted by 2,4,6-trichloro-1,3,5-triazine and PPh₃ in the presence of K₂CO₃

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Experimental Procedure

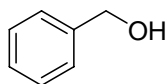
1. Material and methods

All reagents were purchased from Fluka and Aldrich and were used without further purification. The reaction was monitored by thin-layer chromatography carried out on silica gel plates (60F₂₅₄, MERCK, Germany) and visualized under UV light (245 nm). Melting points were determined using Mettler Toledo DSC equipment at a heating rate of 10 °C/min and are uncorrected. NMR spectra were determined using a Bruker AVANCE™ (400 MHz for ¹H). Chemical shifts were reported in parts per million (ppm, δ) downfield from TMS. Splitting patterns are described as singlet (*s*), doublet (*d*), triplet (*t*), quartet (*q*), multiplet (*m*), broad (*br*), and doublet of doublet (*dd*). Gas Chromatography–Mass Spectrometry (GC-MS) analysis was performed with an HP model 6850 gas chromatograph equipped with an HP-5MS (5% phenyl-polymethylsiloxane) capillary column (30 m x 0.25 mm i.d., film thickness 0.25 μ m, Agilent Technologies, USA) interfaced to an HP model 5973 mass-selective detector. EI mass spectra were collected at 70 eV ionization voltages over the range of *m/z* 30–400 and electron multiplier voltage was 2000 V. The mass spectra were compared with mass spectra of individual components with the reference mass spectra in the Wiley 275 and NIST 98 databases. High resolution mass spectra (HRMS) were recorded using the LC-DAD-ESI-MS/MS system consisted of a Waters Alliance 2695 LC-DAD and a Q-TOF 2 (quadrupole mass filter-time-of-flight) mass spectrometer with a Z-spray ES source.

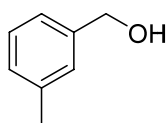
2. General procedure for solvent-free reduction of carboxylic acid

Unless otherwise specified, carboxylic acid (0.271 mmol), TCT (0.0500 g, 0.271 mmol), PPh₃ (0.0142 g, 0.054 mmol) and K₂CO₃ (0.0561 g, 0.406 mmol) were mixed together and ground for 5 min, during which a few drops of CH₂Cl₂ were added to aid the mixing and grinding. After addition of NaBH₄ (0.0202 g, 0.541 mmol), the mixture was ground for further 5 minute. The crude material was purified by short column chromatography using ethyl acetate/hexane as the eluent to afford pure product. All known products were characterized by ¹H-¹³C-NMR and GC-MS, and their spectroscopic data were compared with those reported in the literature.¹⁹

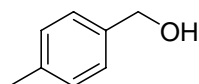
Spectroscopic data of the representative products



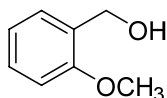
Benzyl alcohol (Table 2, entry 1) Following the general procedure with benzoic acid (0.0331 g, 0.271 mmol), the product was purified by column chromatography using 10% ethyl acetate/hexane as the eluent. Benzyl alcohol was obtained as a colorless oil (0.0264 g, 90% yield); R_f 0.41 (10% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.41-7.32 (m, 5H), 4.62 (s, 2H), 3.47 (br, s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 141.0, 128.5, 127.55, 127.08, 64.9; MS (EI) m/z (rel intensity) 108 (M^+ , 86), 79 (100), 77 (59), 51 (24).



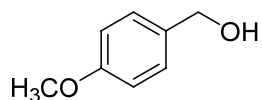
***m*-Tolylmethanol** (Table 2, entry 2) Following the general procedure with 3-methylbenzoic acid (0.0369 g, 0.271 mmol), the product was purified by column chromatography using 10% ethyl acetate/hexane as the eluent. *m*-Tolylmethanol was obtained as a colorless oil (0.0302 g, 91% yield); R_f 0.37 (10% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.19 (t, $J = 7.2$ Hz, 1H), 7.11 (d, $J = 7.2$ Hz, 1H), 7.09 (d, $J = 7.2$ Hz, 1H), 7.04 (d, $J = 7.2$ Hz, 1H), 4.59 (s, 2H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 140.8, 138.3, 128.5, 128.4, 127.8, 124.1, 65.4, 21.4.; MS (EI) m/z (rel intensity) 122 (M^+ , 100), 107 (79), 91 (60), 79 (52).



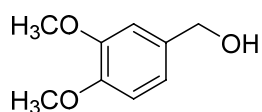
***p*-Tolylmethanol** (Table 2, entry 3) Following the general procedure with 4-methylbenzoic acid (0.0369 g, 0.271 mmol), the product was purified by column chromatography using 10% ethyl acetate/hexane as the eluent. *p*-Tolylmethanol was obtained as a white solid (0.0300 g, 91% yield); mp 60-61 °C; R_f 0.43 (10% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.18 (d, $J = 8.0$ Hz, 2H), 7.11 (d, $J = 8.0$ Hz, 2H), 4.52 (s, 2H), 2.74 (s, 1H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 138.0, 137.3, 129.2, 127.2, 65.0, 21.2.; MS (EI) m/z (rel intensity) 122 (M^+ , 95), 107 (100), 91 (65), 79 (65).



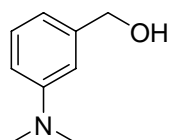
(2-Methoxyphenyl)methanol (Table 2, entry 4) Following the general procedure with 2-methoxybenzoic acid (0.0412 g, 0.271 mmol), the product was purified by column chromatography using 20% ethyl acetate/hexane as the eluent. (2-Methoxyphenyl)methanol was obtained as a colorless oil (0.0333 g, 89% yield); R_f 0.43 (20% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.34 (dd, $J = 8.0, 0.8$ Hz, 1H), 7.30 (td, $J = 8.0, 1.6$ Hz, 1H), 6.98 (td, $J = 8.0, 1.6$ Hz, 1H), 6.88 (d, $J = 8.0$ Hz, 1H), 4.70 (s, 2H), 3.82 (s, 3H), 3.32 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 157.2, 129.3, 128.7, 128.5, 120.6, 110.2, 61.2, 55.2; MS (EI) m/z (rel intensity) 138 (M^+ , 100), 121 (25), 105 (61), 77 (58).



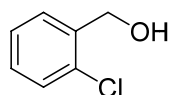
(4-Methoxyphenyl)methanol (Table 2, entry 5) Following the general procedure with 4-methoxybenzoic acid (0.0412 g, 0.271 mmol), the product was purified by column chromatography using 30% ethyl acetate/hexane as the eluent. (4-Methoxyphenyl)methanol was obtained as a colorless oil (92%); R_f 0.50 (30% EtOAc/hexanes); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.20 (d, $J = 8.6$ Hz, 2H), 6.83 (d, $J = 8.6$ Hz, 2H), 4.49 (s, 2H), 3.75 (s, 3H), 3.02 (s, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 159.0, 133.2, 128.6, 113.9, 64.6, 55.3; MS (EI) m/z (rel intensity) 138 (M^+ , 100), 121 (52), 109 (72), 77 (46).



(3,4-Dimethoxyphenyl)methanol (Table 2, entry 6) Following the general procedure with 3,4-dimethoxybenzoic acid (0.0494 g, 0.271 mmol), the product was purified by column chromatography using 30% ethyl acetate/hexane as the eluent. (3,4-Dimethoxyphenyl)methanol was obtained as a yellow oil (0.0434 g, 95% yield); R_f 0.41 (40% EtOAc/hexanes); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 6.92 (d, $J = 1.6$ Hz, 1H), 6.88 (dd, $J = 8.0, 1.6$ Hz, 1H), 6.83 (d, $J = 8.0$ Hz, 1H), 4.61 (s, 2H), 3.88 (s, 3H), 3.87 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 119.1, 148.6, 133.6, 119.4, 111.1, 110.5, 65.3, 55.95, 55.84.; MS (EI) m/z (rel intensity) 168 (M^+ , 100), 151 (26), 139 (33), 109 (17).

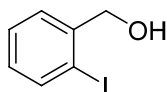


(3-(Dimethylamino)phenyl)methanol (Table 2, entry 7) Following the general procedure with 3-(dimethylamino)benzoic acid (0.0448 g, 0.271 mmol), the product was purified by column chromatography using 20% ethyl acetate/hexane as the eluent. (3-(Dimethylamino)phenyl)methanol was obtained as a yellow oil (0.0307 g, 75% yield); R_f 0.50 (30% EtOAc/hexanes); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.26 (t, $J = 8.0$ Hz, 1H), 6.78 (d, $J = 1.6$ Hz, 1H), 6.74 (d, $J = 8.0$ Hz, 1H), 6.72 (dd, $J = 8.0, 1.6$ Hz, 1H), 4.62 (s, 2H), 2.97 (s, 6H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 150.9, 142.0, 129.2, 115.5, 112.1, 111.4, 65.62, 65.59, 40.8.; MS (EI) m/z (rel intensity) 151 (M^+ , 85), 150 (100), 120 (12), 77 (14).

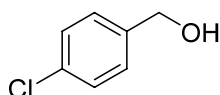


(2-Chlorophenyl)methanol (Table 2, entry 8) Following the general procedure with 2-chlorobenzoic acid (0.0424 g, 0.271 mmol), the product was purified by column chromatography using 20% ethyl acetate/hexane as the eluent. (2-chlorophenyl)methanol was obtained as a white solid (0.0340 g, 88% yield); mp 68-69 °C; R_f 0.41 (10% EtOAc/hexanes); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.47 (d, $J = 8.0$ Hz, 1H), 7.36 (dd, $J = 8.0, 1.2$ Hz, 1H), 7.30-7.21 (m, 2H), 4.75 (s, 2H), 2.84 (br, s, 1H); $^{13}\text{C NMR}$ (100

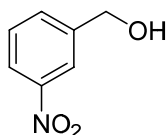
MHz, CDCl₃) δ 138.2, 132.6, 129.3, 128.76, 128.63, 62.6; MS (EI) m/z (rel intensity) 142 (M⁺, 57), 107 (65), 77 (100), 51 (22).



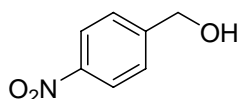
(2-Iodophenyl)methanol (Table 2, entry 9) Following the general procedure with 2-iodobenzoic acid (0.0672 g, 0.271 mmol), the product was purified by column chromatography using 20% ethyl acetate/hexane as the eluent. (2-Iodophenyl)methanol was obtained as a white solid (0.0539 g, 85% yield); mp 92-93 °C; *R_f* 0.50 (20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.81 (d, *J* = 8.0 Hz, 1H), 7.44 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.35 (d, *J* = 8.0 Hz, 1H), 6.98 (td, *J* = 8.0, 0.8 Hz, 1H), 4.65 (s, 2H), 2.41 (br, s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 142.6, 139.2, 129.3, 128.51, 128.44, 97.5, 69.3; MS (EI) m/z (rel intensity) 234 (M⁺, 100), 105 (35), 79 (68), 51 (22).



(4-Chlorophenyl)methanol (Table 2, entry 10) Following the general procedure with 4-chlorobenzoic acid (0.0424 g, 0.271 mmol), the product was purified by column chromatography using 20% ethyl acetate/hexane as the eluent. (4-Chlorophenyl)methanol was obtained as a white solid (0.0344 g, 89% yield); mp 70-71 °C; *R_f* 0.50 (20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.34-7.29 (m, 4H), 4.67 (s, 2H), 1.79 (br, s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 139.2, 133.3, 128.6, 128.3, 64.3.; MS (EI) m/z (rel intensity) 142 (M⁺, 60), 107 (70), 77 (100), 51 (20).

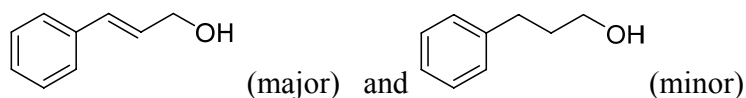


(3-Nitrophenyl)methanol (Table 2, entry 11) Following the general procedure with 3-Nitrobenzoic acid (0.0453 g, 0.271 mmol), the product was purified by column chromatography using 30% ethyl acetate/hexane as the eluent. (3-Nitrophenyl)methanol was obtained as a yellow oil (0.0295 g, 71% yield); *R_f* 0.43 (30% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 8.25 (d, *J* = 0.8 Hz, 1H), 8.16-8.14 (m, 1H), 7.71 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.55 (t, *J* = 8.0 Hz, 1H), 4.84 (s, 2H), 2.23 (br, s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 142.9, 132.62, 131.97, 129.4, 122.5, 121.5, 63.9; MS (EI) m/z (rel intensity) 153 (M⁺, 22), 136 (25), 107 (31), 77 (100).

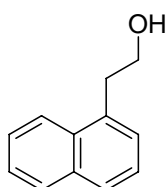


(4-Nitrophenyl)methanol (Table 2, entry 12) Following the general procedure with 4-Nitrobenzoic acid (0.0453 g, 0.271 mmol), the product was purified by column chromatography using 20% ethyl acetate/hexane as the eluent. (4-Nitrophenyl)methanol was obtained as a pale brown (0.0291 g, 70% yield); mp 91-92 °C; *R_f* 0.46 (20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 8.20 (d, *J* = 8.8 Hz,

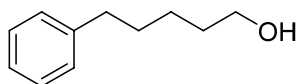
2H), 7.52 (d, $J = 8.8$ Hz, 2H), 4.82 (s, 2H), 1.58 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 148.4, 147.3, 127.0, 123.7, 63.9; MS (EI) m/z (rel intensity) 153 (M^+ , 30), 107 (41), 77 (100), 51 (31).



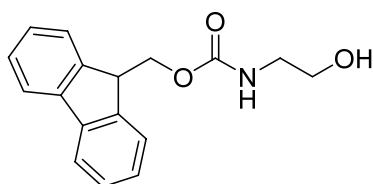
(*E,Z*)-3-phenylprop-2-en-1-ol (Table 2, entry 13) Following the general procedure with Cinnamic acid (0.0402 g, 0.271 mmol), the product was purified by column chromatography using 20% ethyl acetate/hexane as the eluent. (*E,Z*)-3-Phenylprop-2-en-1-ol was obtained as a yellow solid (0.0295 g, 81% yield); R_f 0.45 (20% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) (major) δ 7.40-6.33 (m, 5H), 6.62 (d, $J = 16.0$ Hz, 1H), 6.37 (dt, $J = 16.0, 5.6$ Hz, 1H), 4.32 (dd, $J = 5.8, 1.6$ Hz, 2H), 1.69 (br, s, 1H); (minor) δ 7.40-6.33 (m, 5H), 3.68 (t, $J = 6.8$ Hz, 2H), 2.71 (t, $J = 6.8$ Hz, 2H), 1.93-1.86 (m, 2H), 1.69 (br, s, 1H); MS (EI) m/z (rel intensity) 134 (M^+ , 70), 115 (52), 92 (100), 78 (55).



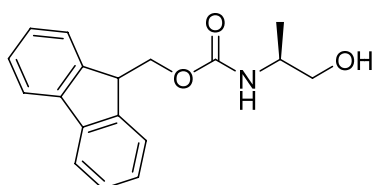
2-(Naphthalen-1-yl)ethanol (Table 2, entry 14) Following the general procedure with 2-(Naphthalen-1-yl)acetic acid (0.0505 g, 0.271 mmol), the product was purified by column chromatography using 20% ethyl acetate/hexane as the eluent. 2-(Naphthalen-1-yl)ethanol was obtained as a yellow oil (0.0355 g, 76% yield); R_f 0.40 (20% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 8.05 (d, $J = 8.2$ Hz, 1H), 7.86 (dd, $J = 8.2, 1.6$ Hz, 1H), 7.75 (d, $J = 7.6$ Hz, 1H), 7.54 – 7.46 (m, 2H), 7.43 – 7.37 (m, 2H), 3.99 (t, $J = 6.8$ Hz, 2H), 3.35 (t, $J = 6.8$ Hz, 2H), 1.60 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 134.4, 134.0, 132.1, 128.9, 127.4, 127.2, 126.1, 125.7, 125.5, 123.7, 63.1, 36.2; MS (EI) m/z (rel intensity) 172 (M^+ , 23), 141 (100), 128 (6), 115 (23).



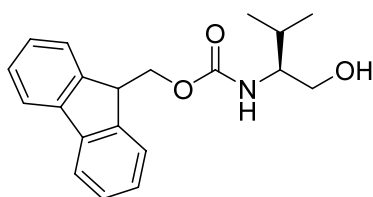
5-Phenylpentan-1-ol (Table 2, entry 15). Following the general procedure with 5-Phenylpentanoic acid (0.0483 g, 0.271 mmol), the product was purified by column chromatography using 10% ethyl acetate/hexane as the eluent. 5-Phenylpentan-1-ol was obtained as a colorless oil (0.0330 g, 74% yield); R_f 0.43 (10% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.28 – 7.15 (m, 5H), 3.59 (t, $J = 6.8$ Hz, 2H), 2.60 (t, $J = 6.8$ Hz, 2H), 2.14 (s, 1H), 1.67 – 1.53 (m, 4H), 1.42 – 1.34 (m, 2H); ^{13}C NMR. (100 MHz, CDCl_3) δ 142.6, 128.4, 128.3, 125.7, 62.8, 36.0, 32.6, 31.3, 25.5; MS (EI) m/z (rel intensity) 164 (M^+ , 5), 146 (30), 117 (50), 91 (100).



(9H-Fluoren-9-yl)methyl 2-hydroxyethylcarbamate (Table 2, entry 16) Following the general procedure with 2-(((9H-fluoren-9-yl)methoxy)carbonylamino)acetic acid (0.0806 g, 0.271 mmol), the product was purified by column chromatography using 40% ethyl acetate/hexane as the eluent. (9H-Fluoren-9-yl)methyl 2-hydroxyethylcarbamate was obtained as a white solid (0.0669 g, 87% yield); mp 137-138 °C; R_f 0.42 (10% Acetone/DCM); ^1H NMR (400 MHz, CDCl_3) δ 7.71 (d, $J=7.2$ Hz, 2H), 7.56 (d, $J=7.2$ Hz, 2H), 7.35 (t, $J=7.2$ Hz, 2H), 7.26 (t, $J=7.2$ Hz, 2H), 5.94 (br, s, 1H), 4.35 (d, $J=6.4$ Hz, 2H), 4.15 (t, $J=6.4$ Hz, 1H), 3.60 (br, s, 4H), 3.25 (br, s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 157.3, 143.8, 141.2, 127.7, 127.0, 125.0, 120.0, 66.7, 61.3, 47.1, 43.2; HRMS (ESI): MNa^+ found 306.1104. $[\text{C}_{17}\text{H}_{17}\text{NO}_3\text{Na}]^+$ requires 306.1106.

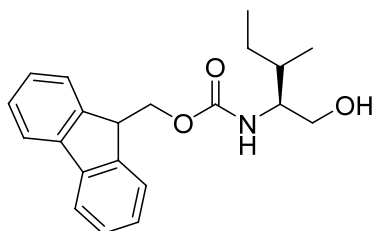


(9H-Fluoren-9-yl)methyl 1-hydroxypropan-2-ylcarbamate (Table 2, entry 17) Following the general procedure with 2-(((9H-fluoren-9-yl)methoxy)carbonylamino)propanoic acid (0.0844 g, 0.271 mmol), the product was purified by column chromatography using 40% ethyl acetate/hexane as the eluent. (9H-Fluoren-9-yl)methyl 1-hydroxypropan-2-ylcarbamate was obtained as a yellow solid (0.0645 g, 80% yield); mp 117-118 °C; R_f 0.38 (5% Acetone/DCM); $[\alpha]_D^{26} = -11.7$ ($c=1.09$, MeOH); ^1H NMR (400 MHz, CDCl_3) δ 7.66 (d, $J=7.2$ Hz, 2H), 7.50 (d, $J=7.2$ Hz, 2H), 7.30 (t, $J=7.2$ Hz, 2H), 7.21 (t, $J=7.2$ Hz, 2H), 5.43 (br, s, 1H), 4.30 (d, $J=5.6$ Hz, 2H), 4.11 (t, $J=5.6$ Hz, 1H), 3.66-3.48 (br, m, 2H), 3.40 (br, s, 1H), 2.93 (br, s, 1H), 1.06 (d, $J=5.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 156.7, 143.9, 141.3, 127.67, 127.05, 125.0, 119.9, 66.6, 49.3, 47.2, 17.2.; HRMS (ESI): MNa^+ found 320.1266. $[\text{C}_{18}\text{H}_{19}\text{NO}_3\text{Na}]^+$ requires 320.1263.

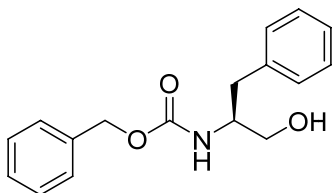


(9H-Fluoren-9-yl)methyl 1-hydroxy-3-methylbutan-2-ylcarbamate (Table 2, entry 18) Following the general procedure with 2-(((9H-fluoren-9-yl)methoxy)carbonylamino)-3-methylbutanoic acid (0.0920 g, 0.271 mmol), the product was purified by column chromatography using 40% ethyl acetate/hexane as the eluent. (9H-Fluoren-9-yl)methyl 1-hydroxy-3-methylbutan-2-ylcarbamate was obtained as a white solid (0.0732 g, 83% yield); mp 108-110 °C.; R_f 0.41 (3% Acetone/DCM); $[\alpha]_D^{26} = -21.4$ ($c=1.02$, MeOH.); ^1H NMR (400 MHz, CDCl_3) δ 7.74 (d, $J=7.2$ Hz, 2H), 7.57 (d, $J=7.2$ Hz, 2H), 7.38 (t, $J=7.2$ Hz, 2H), 7.29 (t, $J=7.2$ Hz, 2H), 5.05 (d, $J=8.8$ Hz, 1H), 4.47 – 4.35 (m, 2H), 4.19 (t, $J=6.4$ Hz,

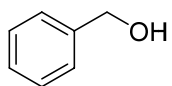
1H), 3.68 – 3.58 (m, 2H), 3.46 (s, 1H), 2.45 (s, 1H), 1.86 – 1.81 (m, 1H), 0.94 – 0.89 (m, 6H).; ¹³C NMR (100 MHz, CDCl₃) δ 157.1, 143.9, 141.4, 127.7, 127.1, 125.1, 120.0, 66.6, 63.6, 58.6, 47.4, 29.2, 19.6, 18.7; HRMS (ESI): MNa⁺ found 348.1573. [C₂₀H₂₃NO₃Na]⁺ requires 348.1576.



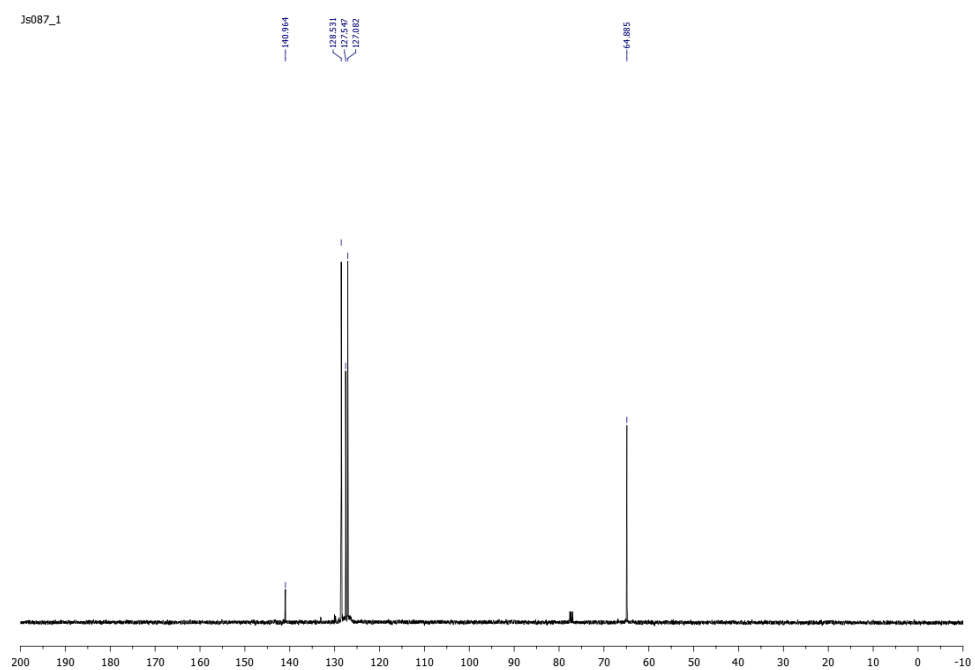
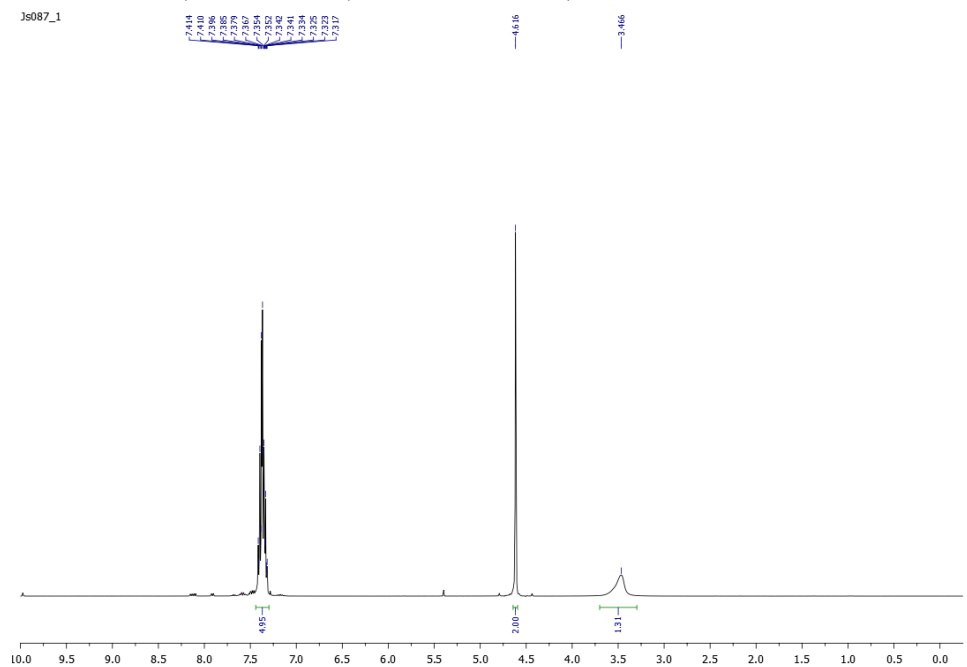
(9H-Fluoren-9-yl)methyl 1-hydroxy-3-methylpentan-2-ylcarbamate (Table 2, entry 19) Following the general procedure with 2-(((9H-fluoren-9-yl)methoxy)carbonylamino)-3-methylpentanoic acid (0.0958 g, 0.271 mmol), the product was purified by column chromatography using 40% ethyl acetate/hexane as the eluent. (9H-Fluoren-9-yl)methyl 1-hydroxy-3-methylpentan-2-ylcarbamate was obtained as a white solid (0.0717 g, 78% yield); mp 118-119 °C; *R_f* 0.44 (5% Acetone/DCM); [α]_D²⁶ = -25.9 (*c* = 1.15, MeOH); ¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, *J* = 7.6 Hz, 2H), 7.59 (d, *J* = 7.6 Hz, 2H), 7.39 (t, *J* = 7.6 Hz, 2H), 7.20 (td, *J* = 7.6, 0.8 Hz, 2H), 5.11 (d, *J* = 8.8 Hz, 1H), 4.48 – 4.37 (m, 2H), 4.20 (t, *J* = 6.4 Hz, 1H), 3.71 – 3.59 (m, 2H), 3.54 (br, s, 1H), 1.61-1.46 (br, m, 2H), 1.14-1.09 (br, m, 1H), 0.92 – 0.88 (m, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 157.1, 143.9, 141.4, 127.7, 127.1, 125.1, 120.0, 66.6, 63.3, 57.4, 47.4, 35.9, 25.4, 15.5, 11.4; HRMS (ESI): MNa⁺ found 362.1737. [C₂₁H₂₅NO₃Na]⁺ requires 362.1732

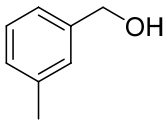


Benzyl 1-hydroxy-3-phenylpropan-2-ylcarbamate (Table 2, entry 20) Following the general procedure with 2-(benzyloxycarbonylamino)-3-phenylpropanoic acid (0.0811 g, 0.271 mmol), the product was purified by column chromatography using 40% ethyl acetate/hexane as the eluent. Benzyl 1-hydroxy-3-phenylpropan-2-ylcarbamate was obtained as a white solid (0.0650 g, 84% yield): mp 91-92 °C.; *R_f* 0.46 (3% Acetone/DCM).; [α]_D²⁶ = +45.7 (*c* = 1.05, MeOH).; ¹H NMR (400 MHz, CDCl₃) δ 7.31 – 7.19 (m, 10H), 5.29 (d, *J* = 6.8 Hz, 1H), 5.03 (s, 2H), 3.92 (br s, 1H), 3.62 – 3.50 (m, 2H), 3.02 (s, 1H), 2.81 (d, *J* = 6.4 Hz, 2H).; ¹³C NMR (100 MHz, CDCl₃) δ 156.6, 137.7, 136.4, 129.4, 128.59, 128.57, 128.2, 128.1, 126.6, 66.8, 63.7, 54.2, 37.3.; HRMS (ESI): MNa⁺ found 308.1260. [C₁₇H₁₉NO₃Na]⁺ requires 308.1263.

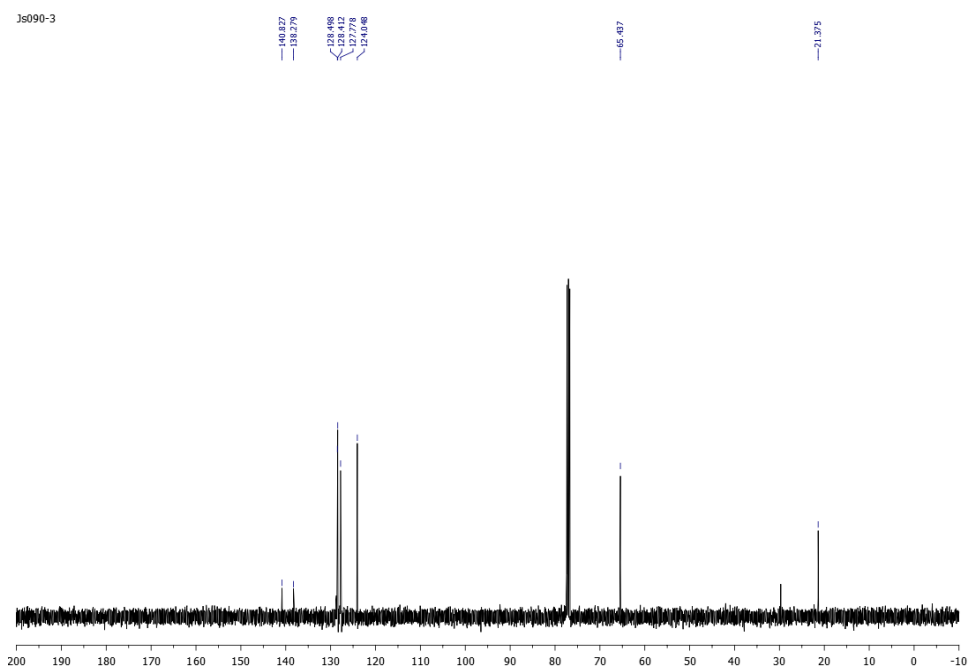
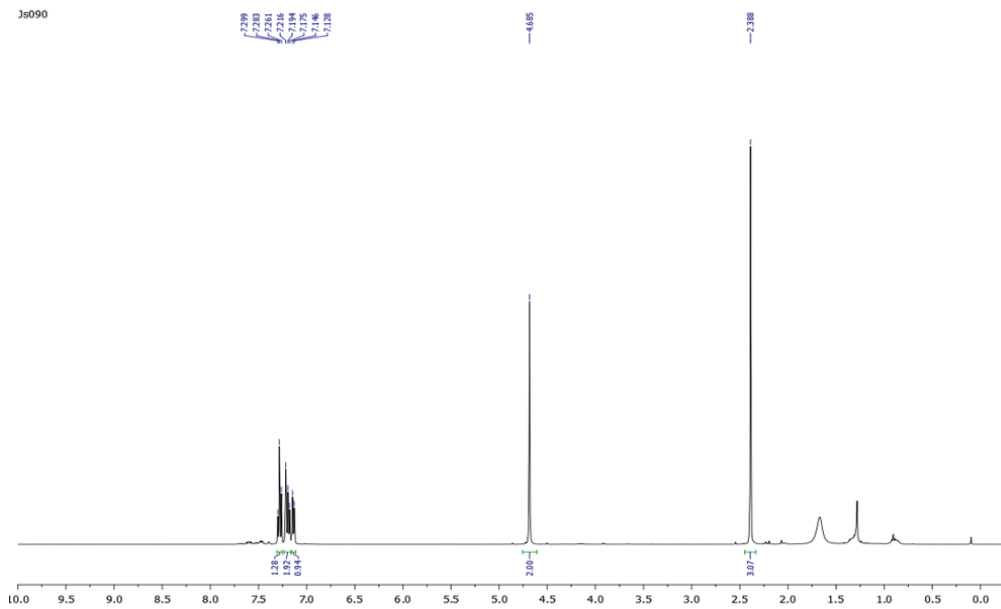


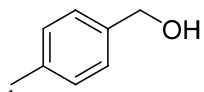
^1H NMR (400 MHz, CDCl_3); ^{13}C NMR (100 MHz, CDCl_3)





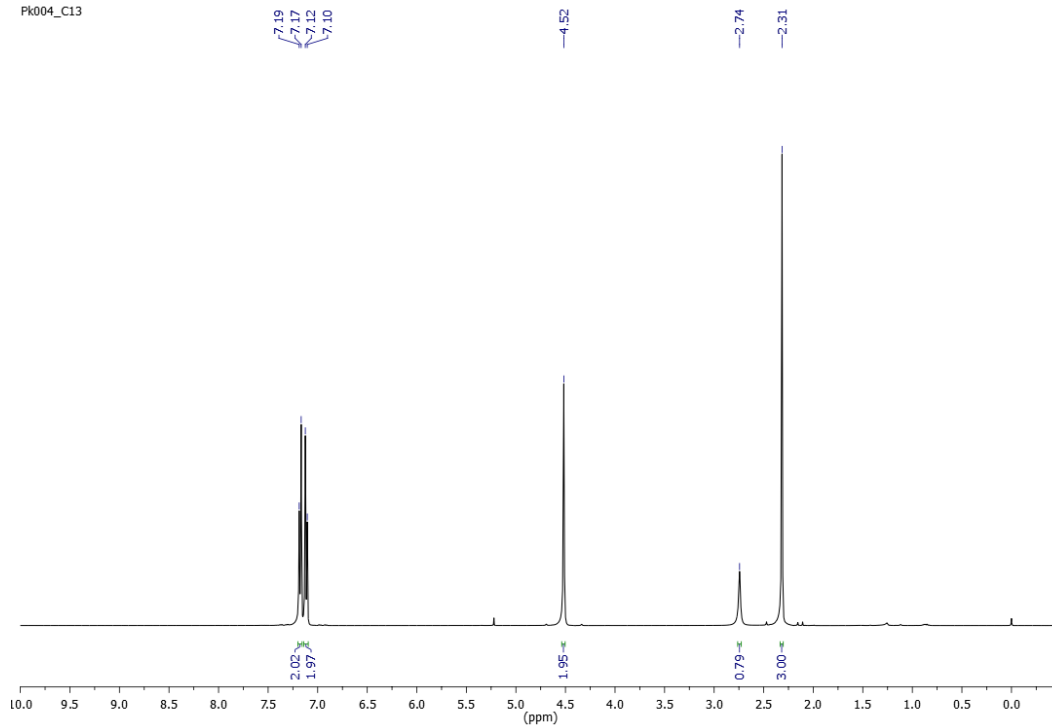
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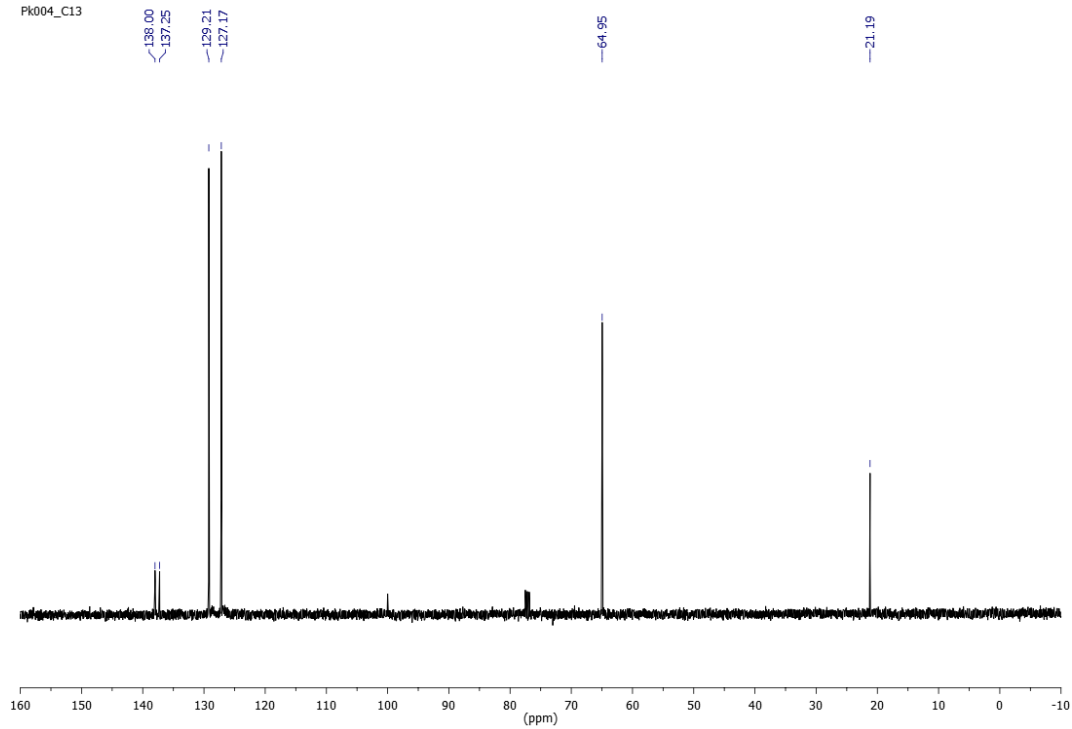


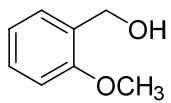
^1H NMR (400 MHz, CDCl_3); ^{13}C NMR (100 MHz, CDCl_3)

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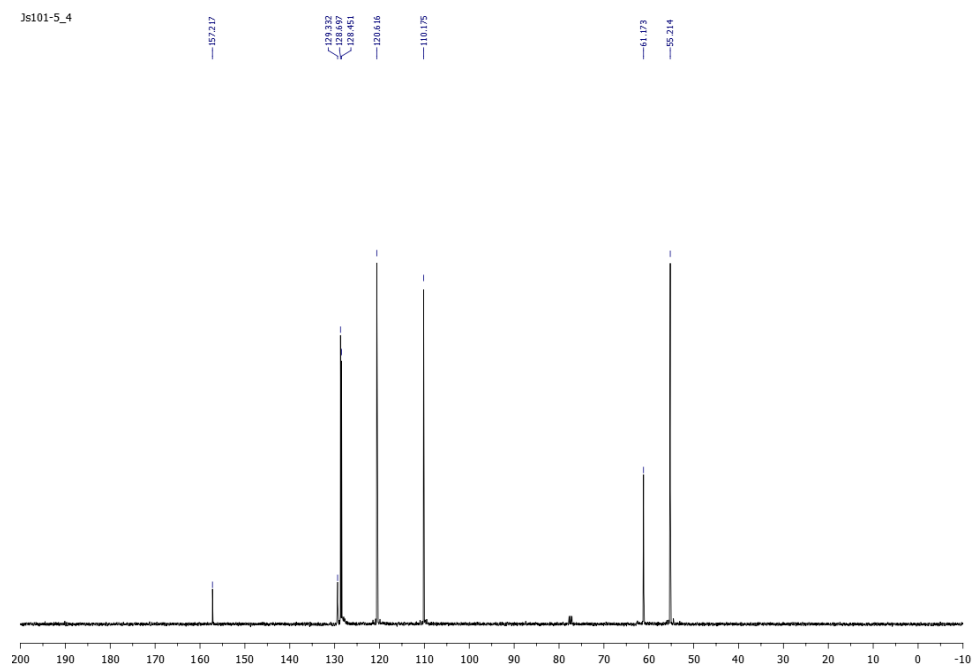
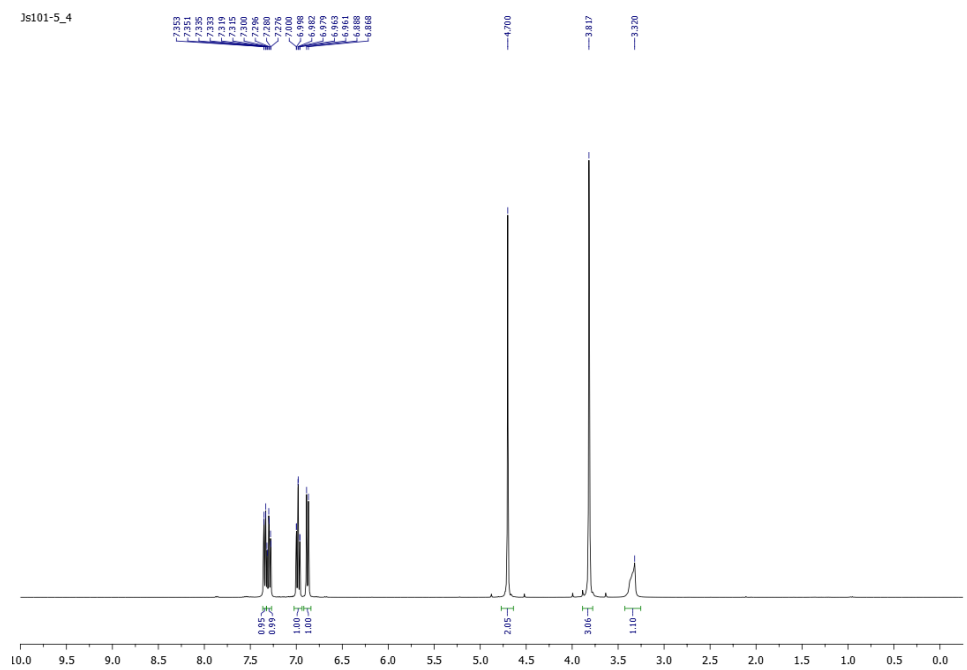


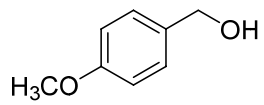
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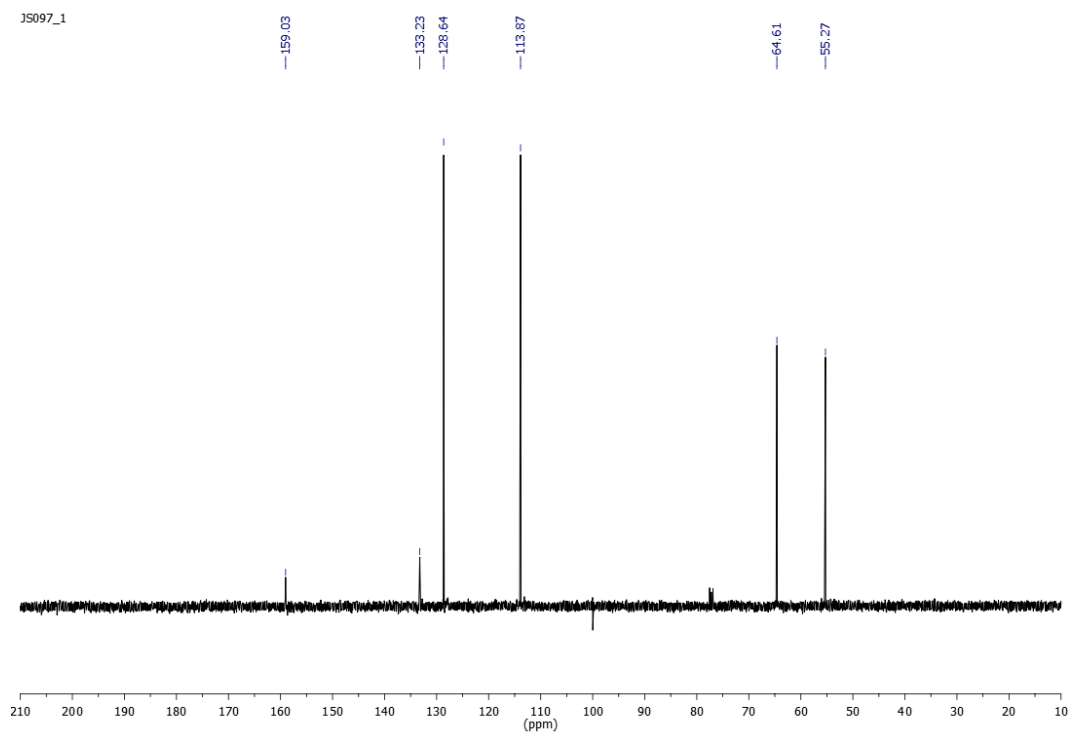
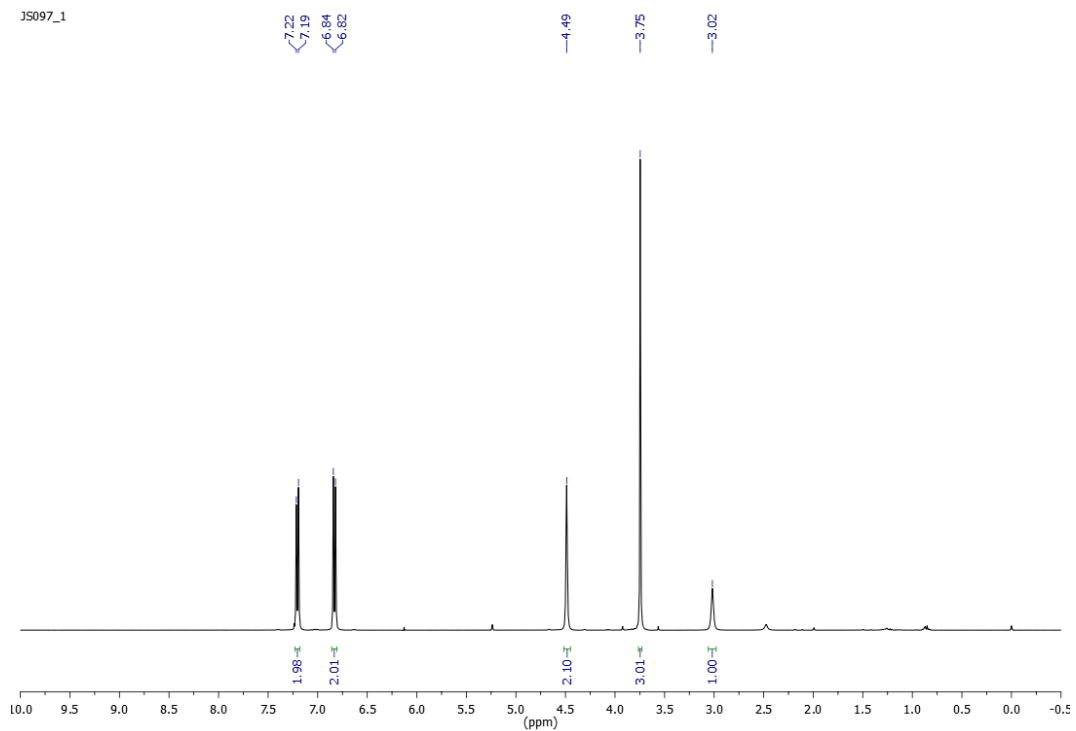


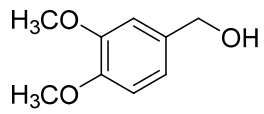
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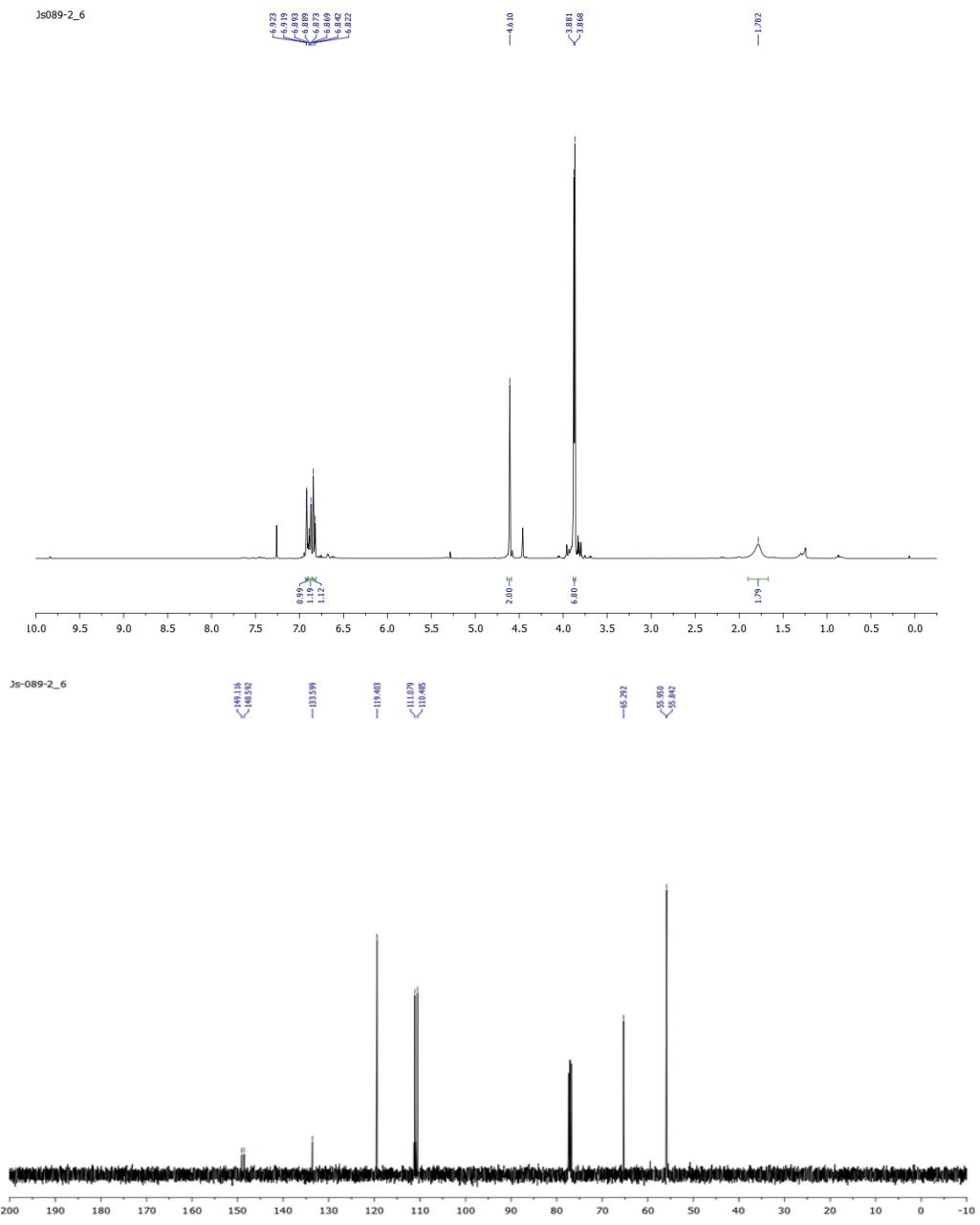


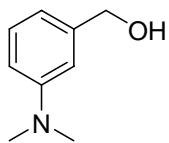
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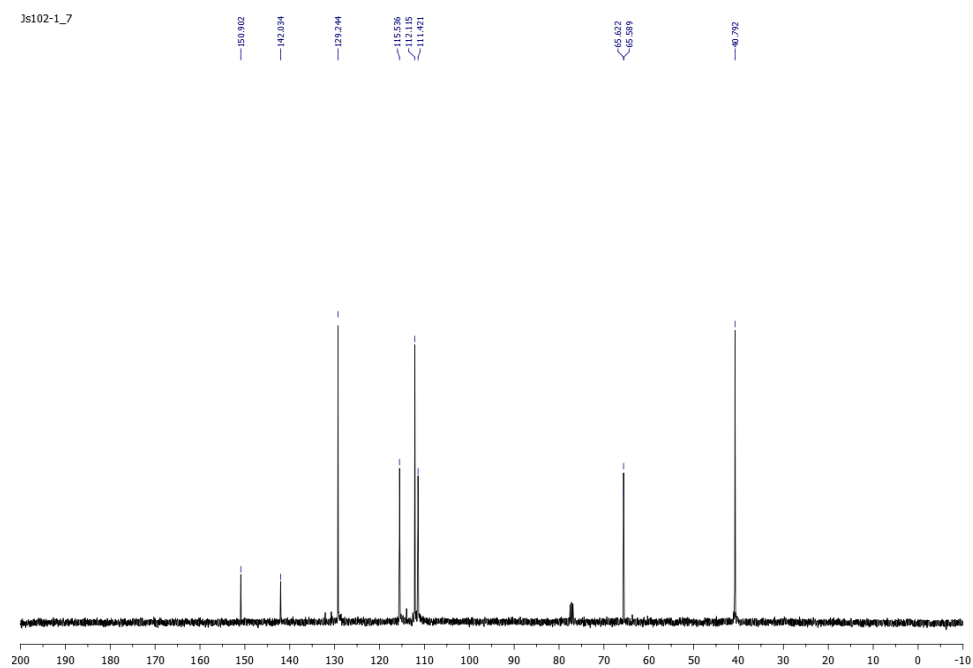
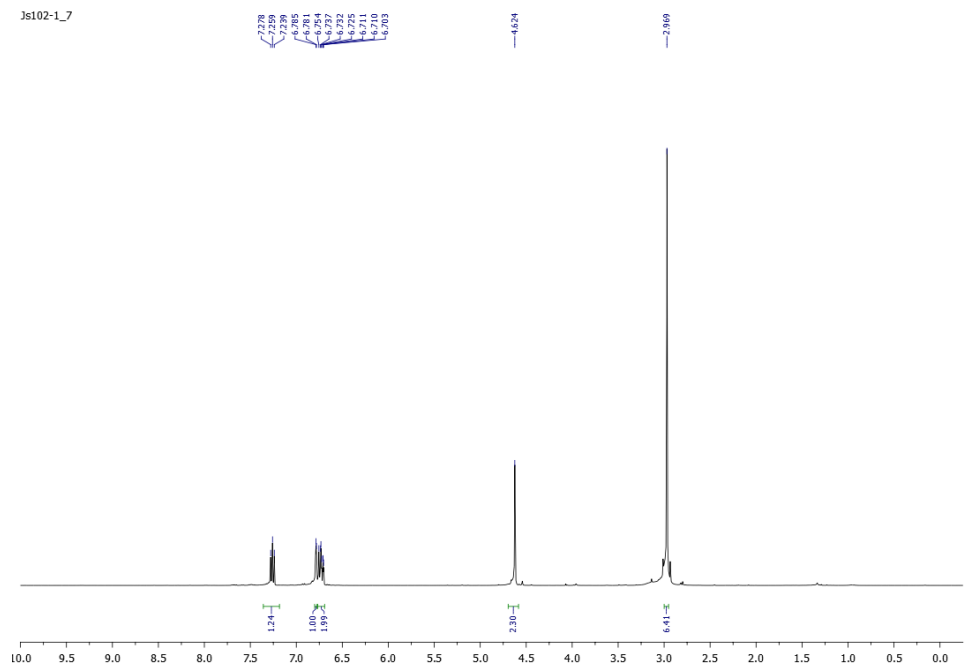


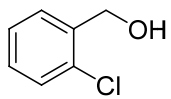
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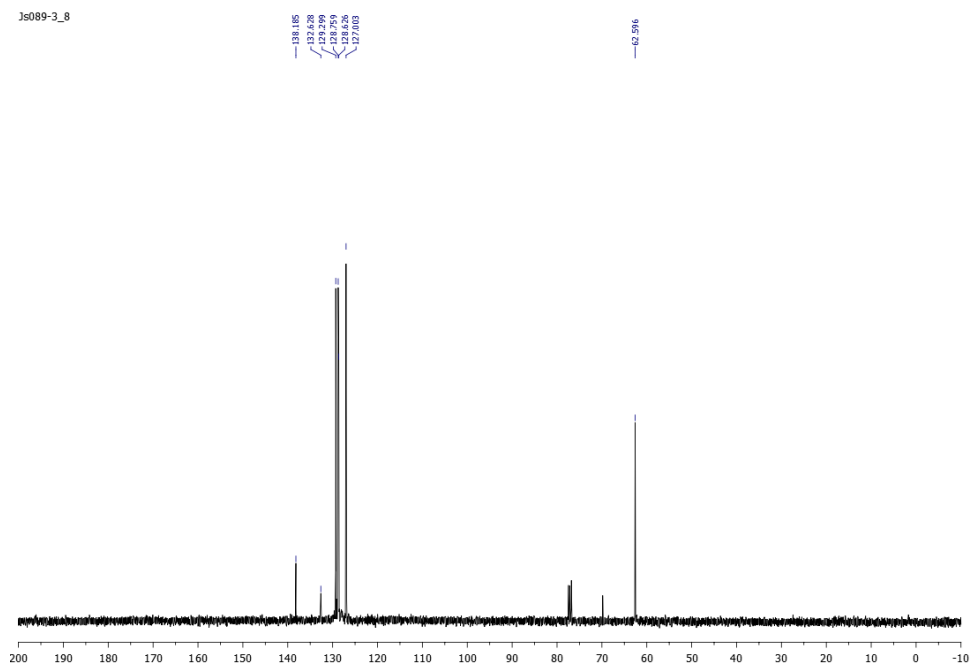
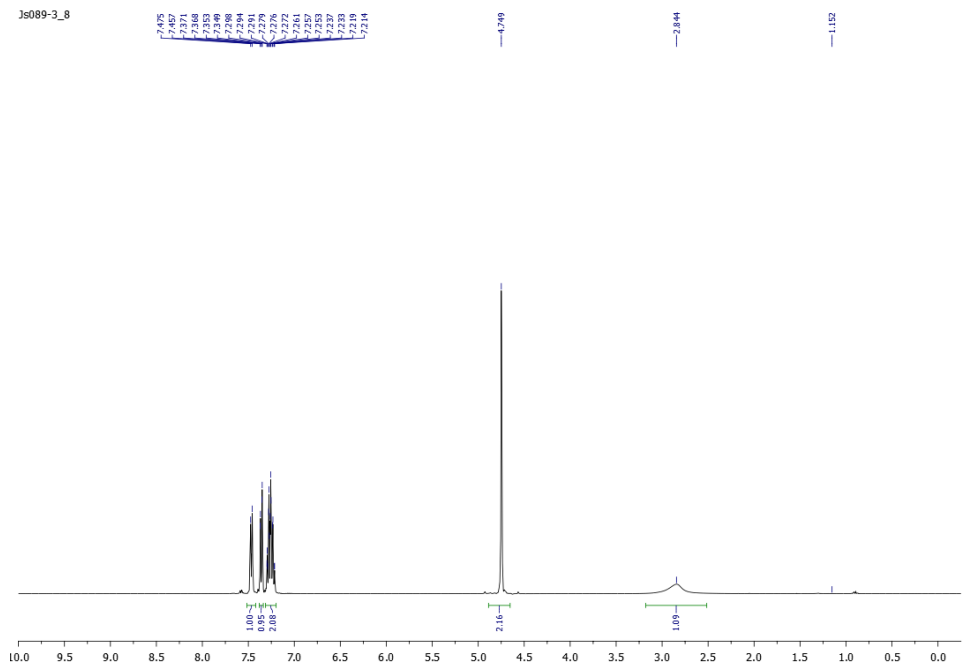


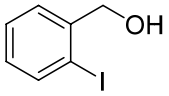
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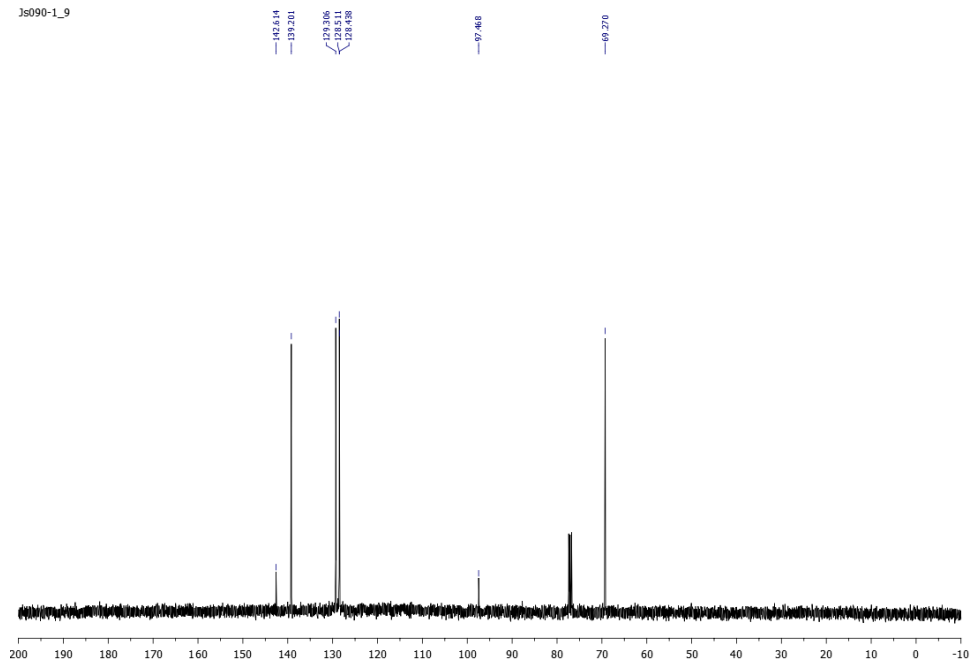
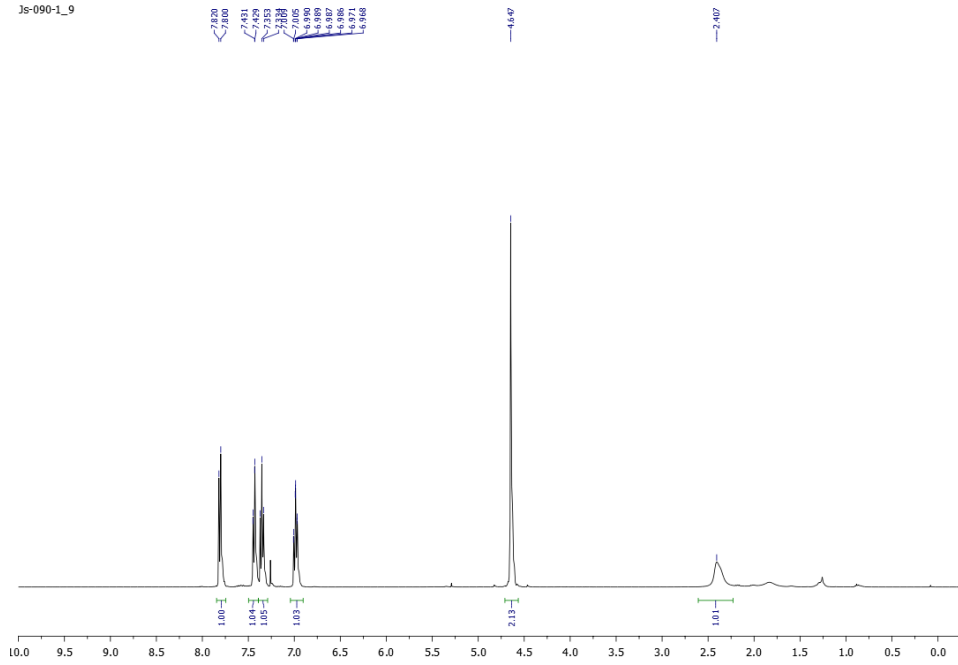


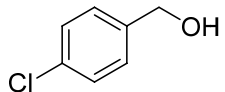
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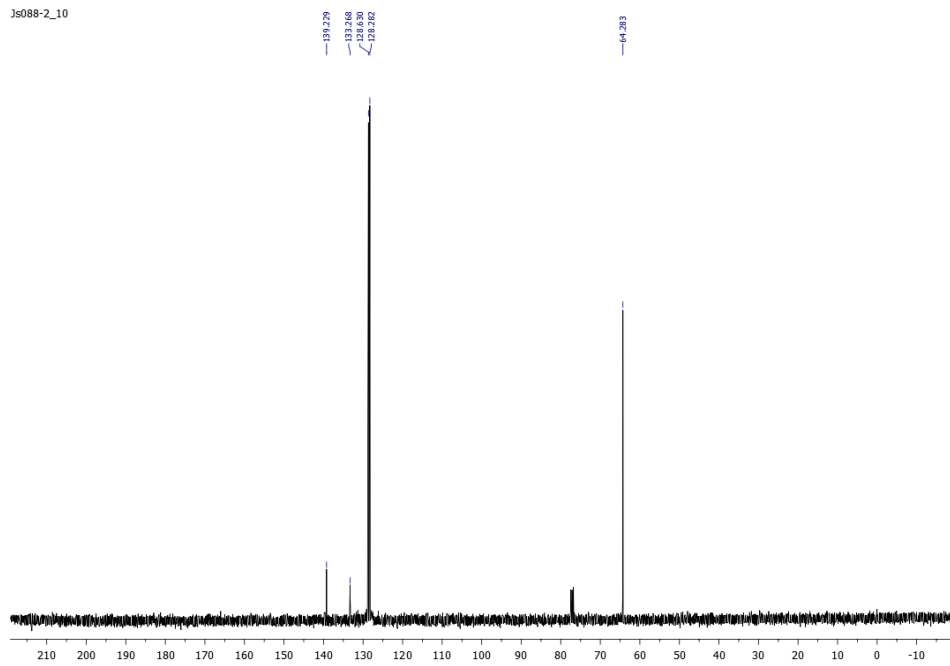
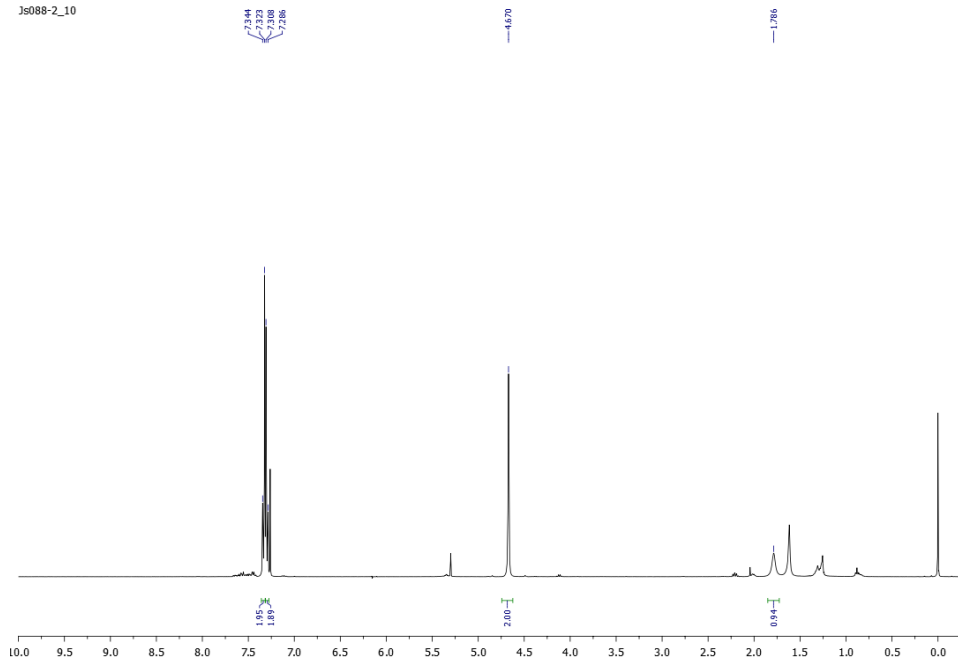


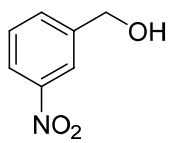
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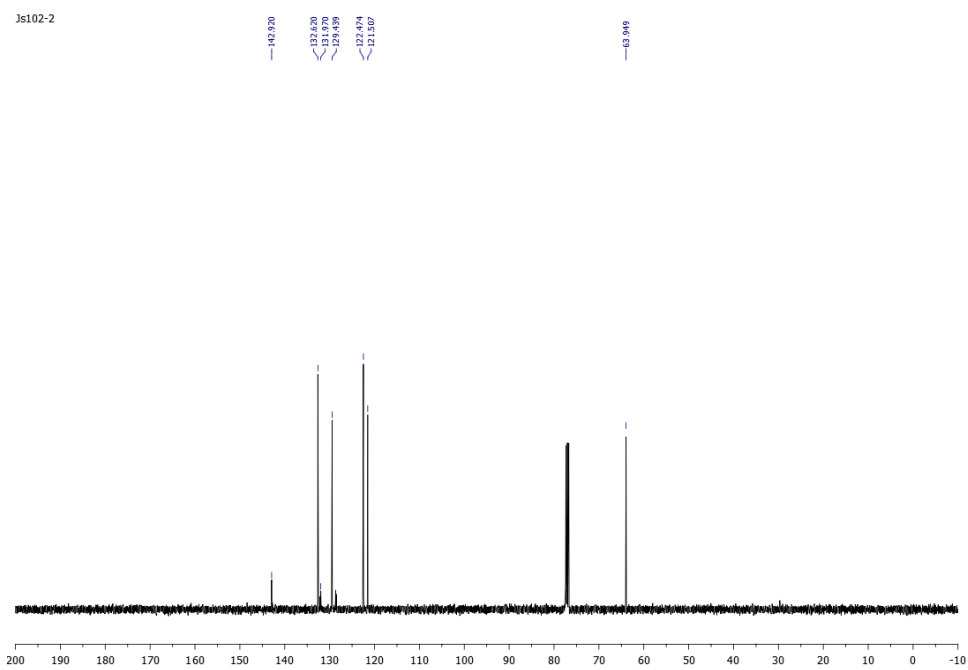
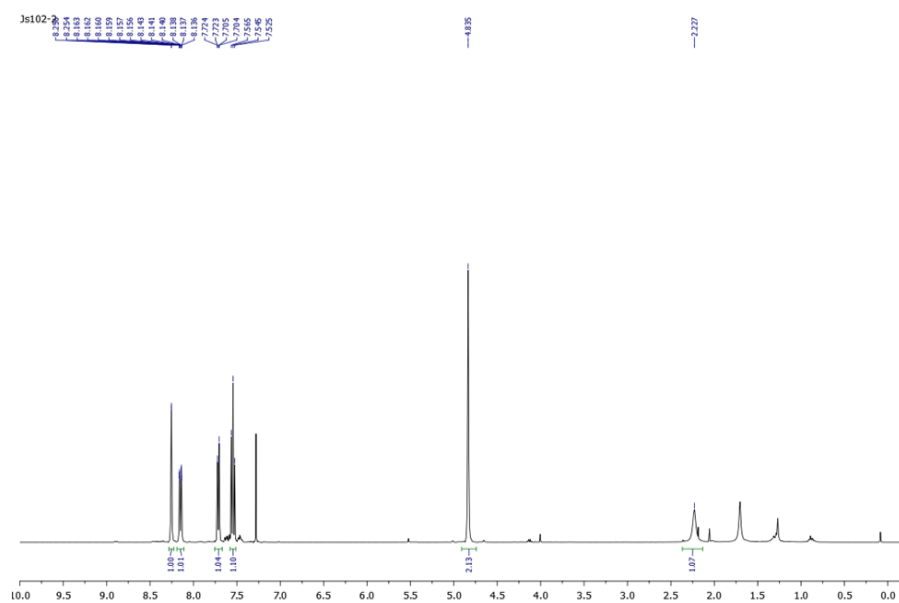


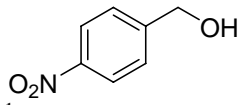
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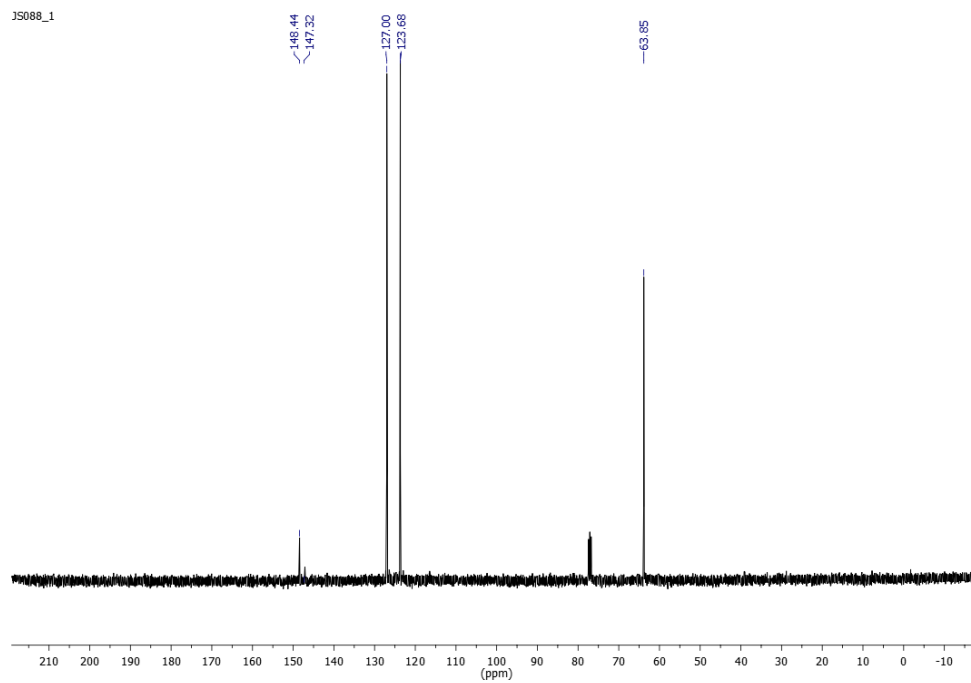
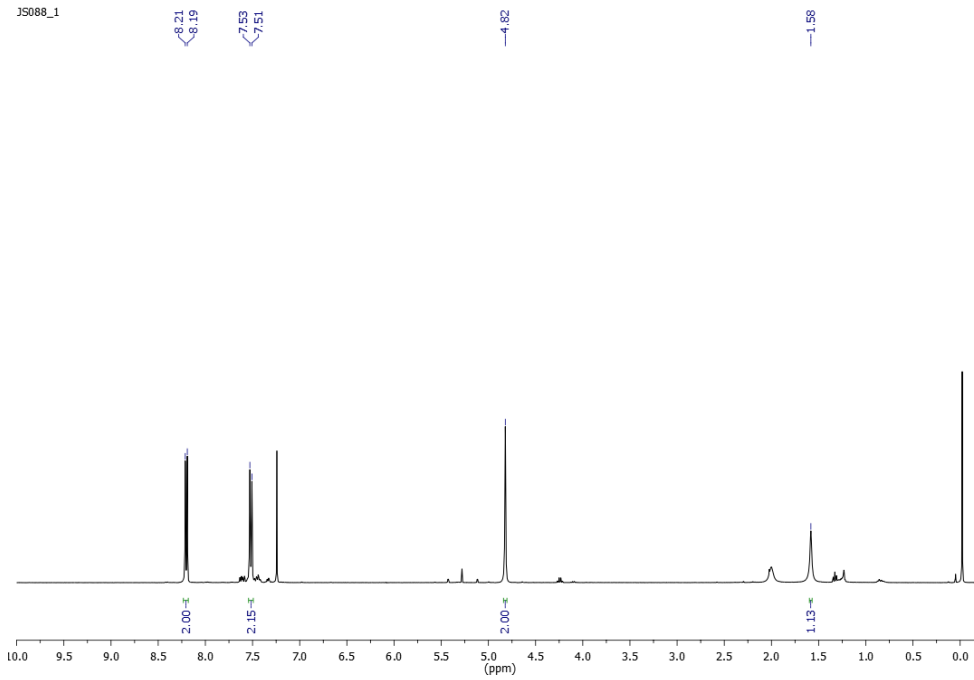


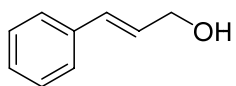
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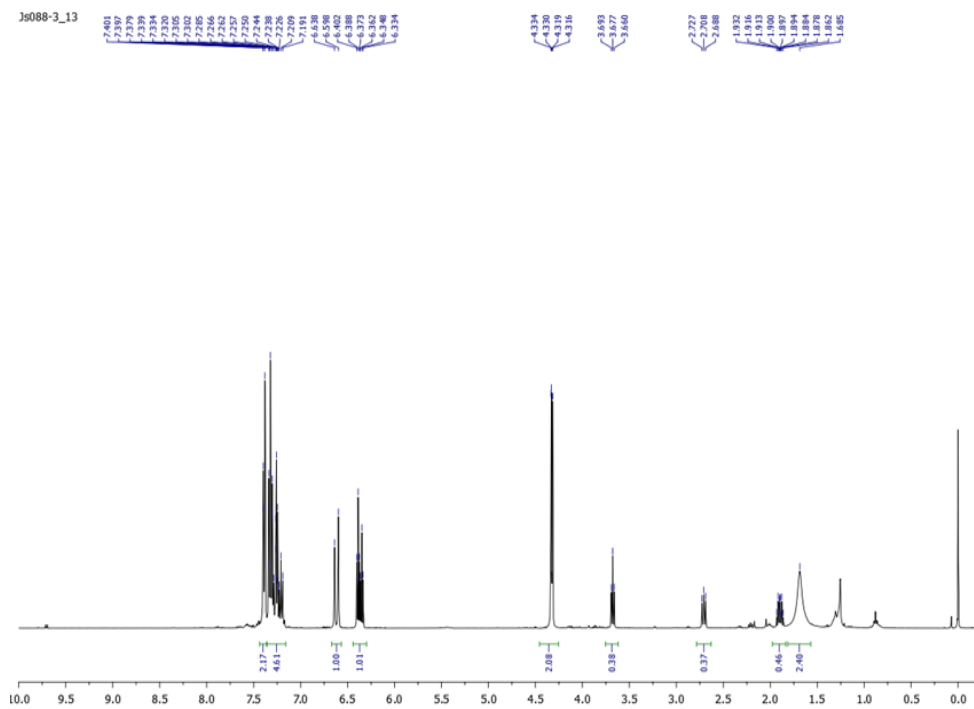


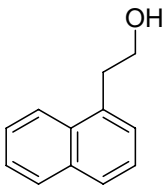
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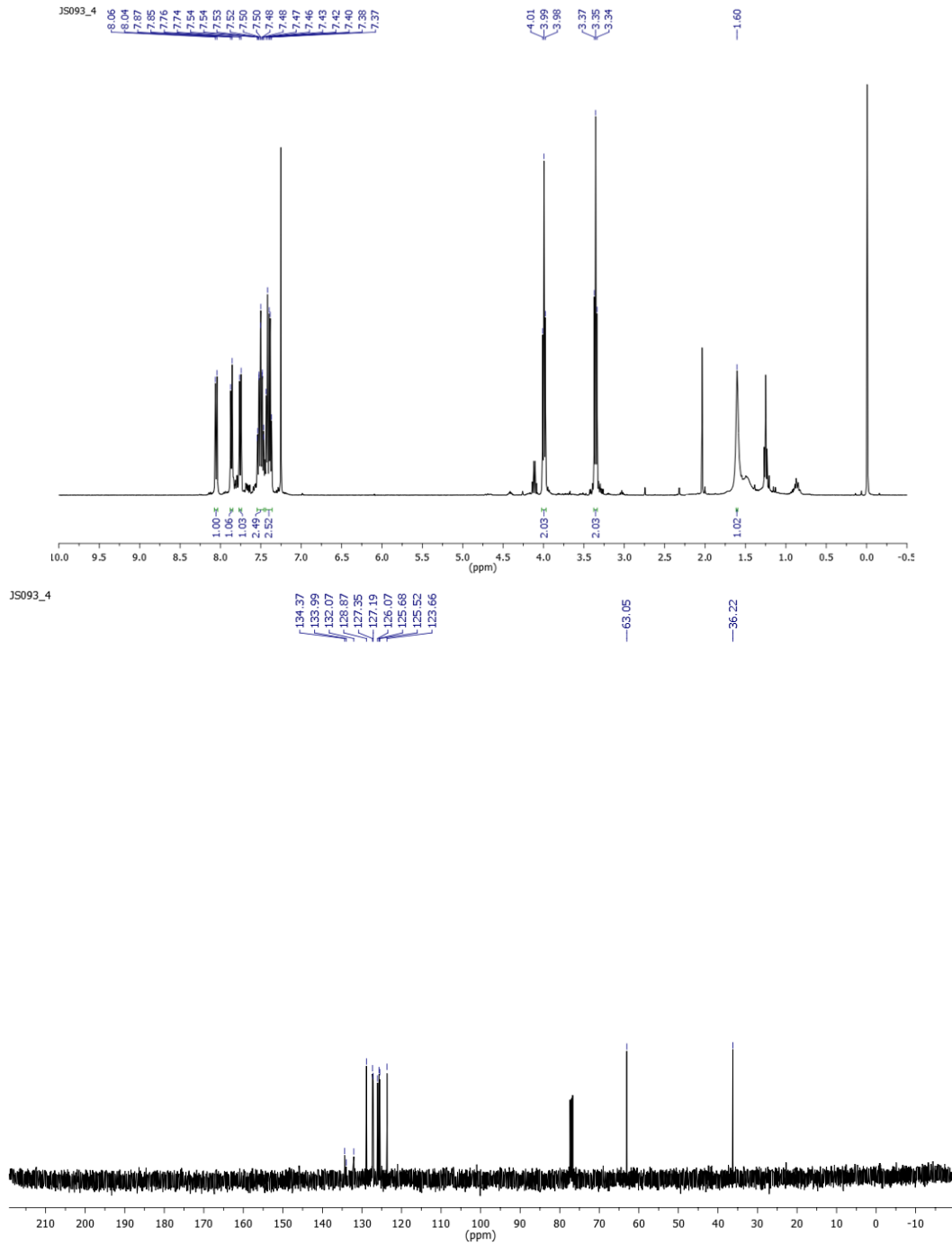


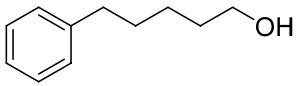
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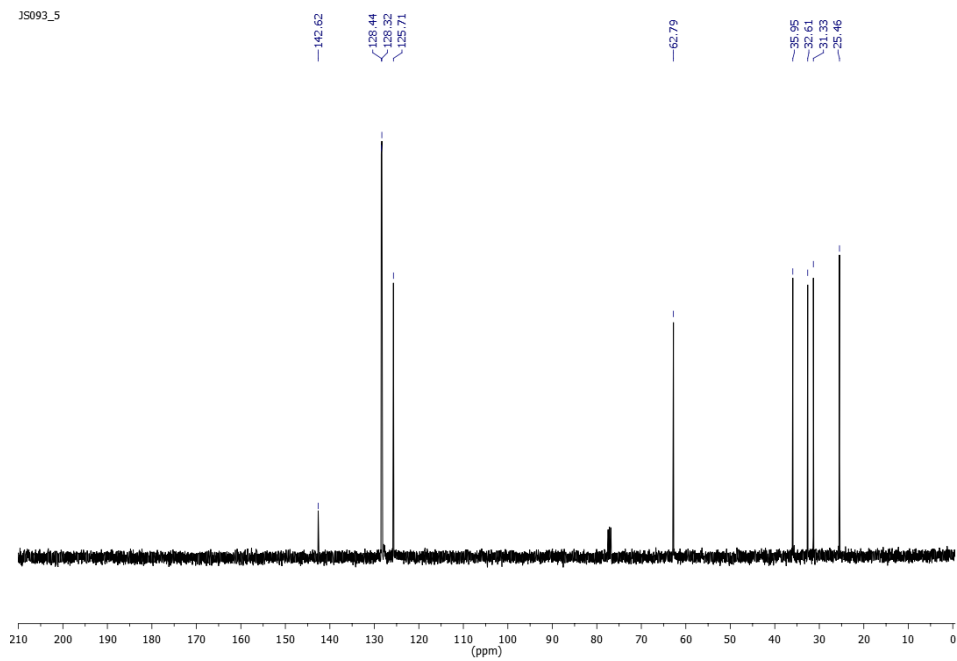
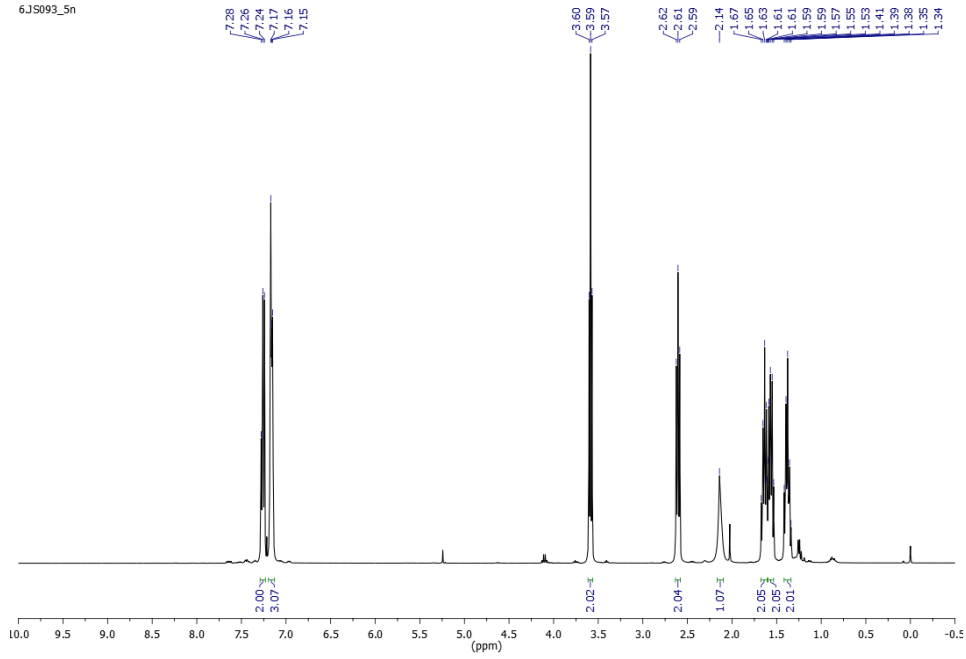


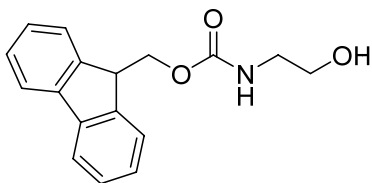
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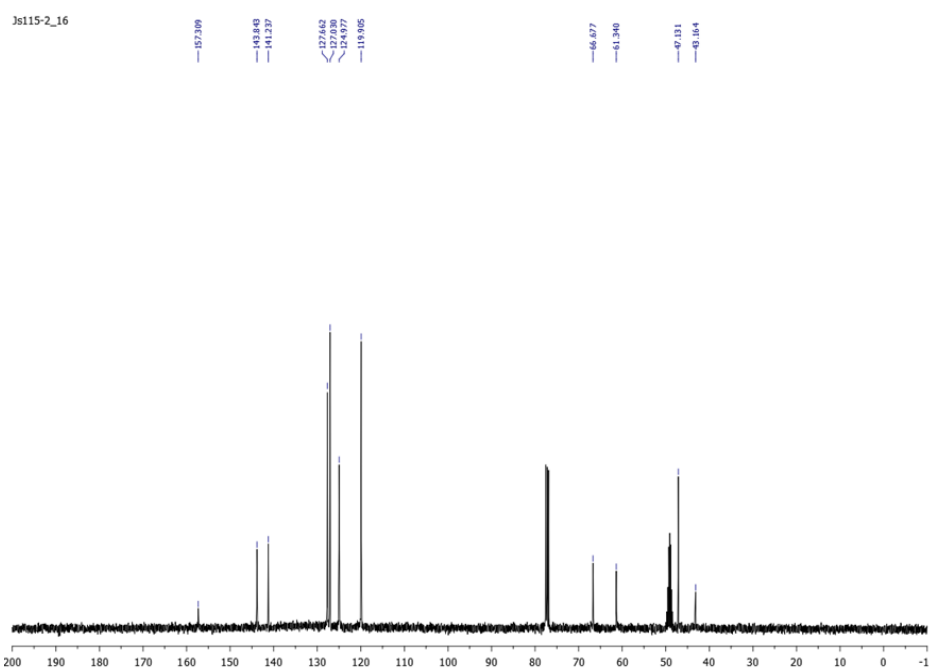
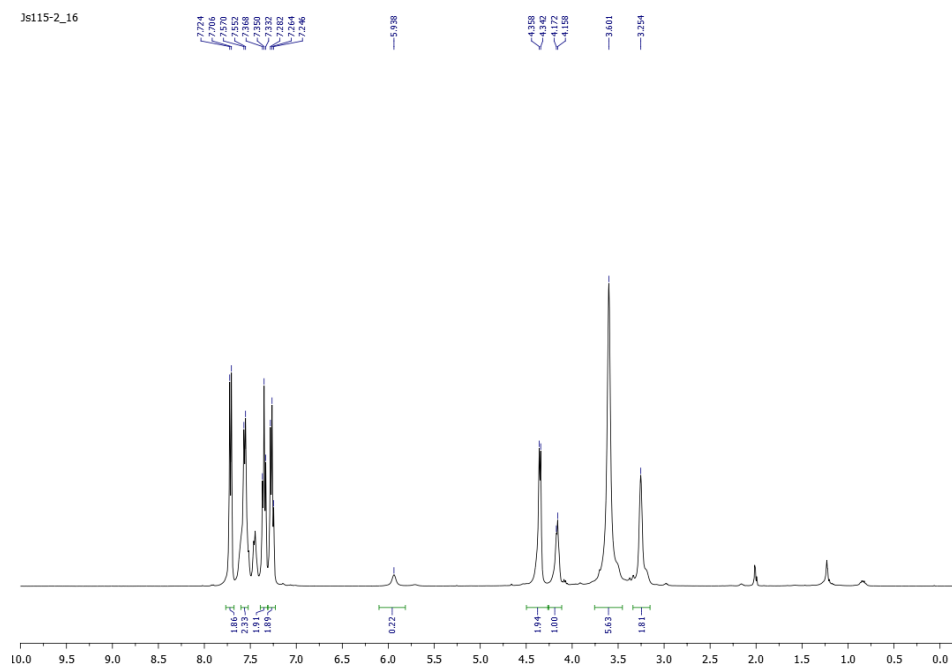


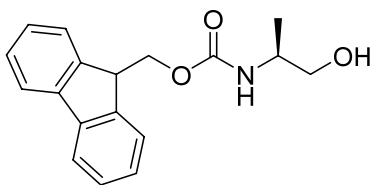
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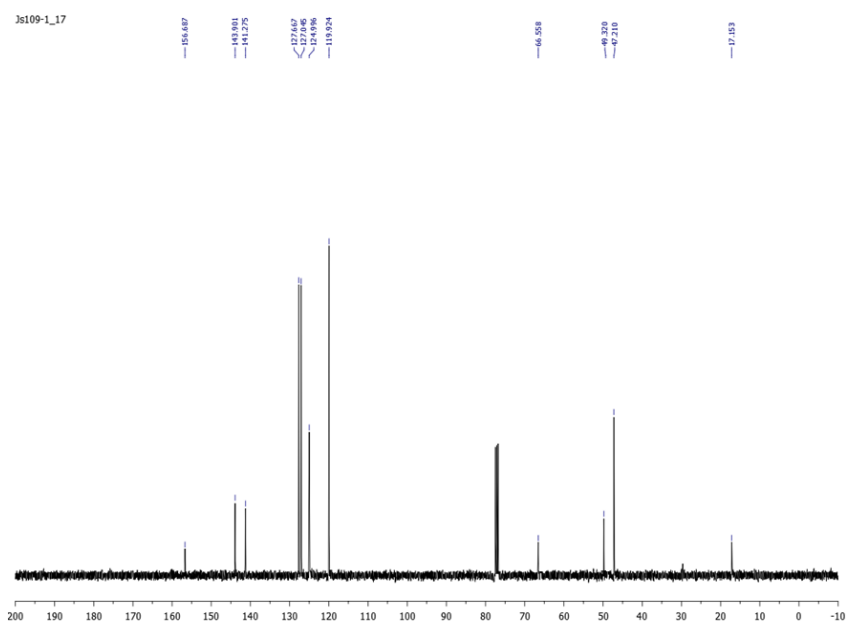
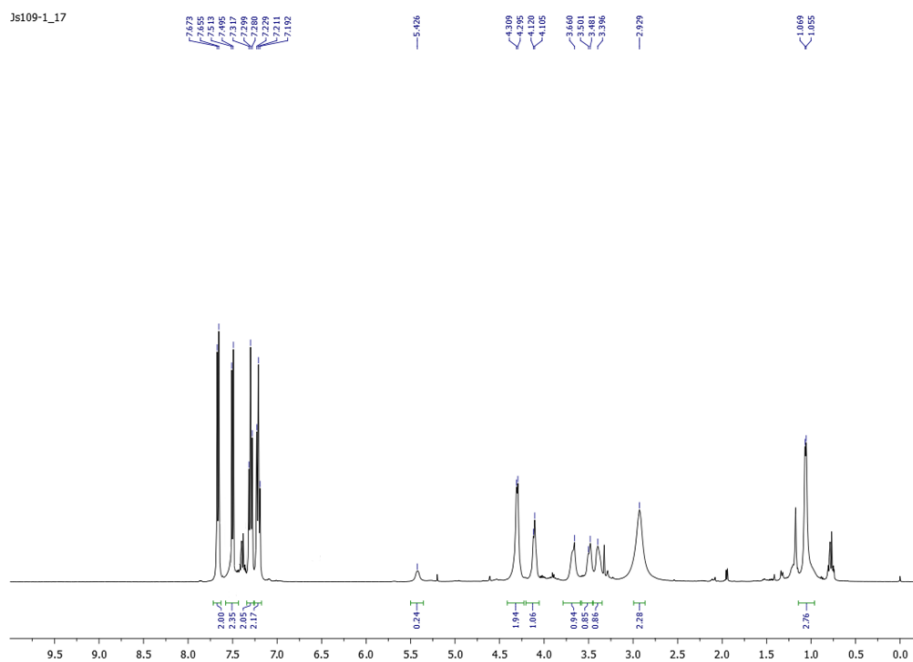


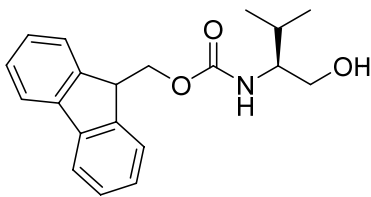
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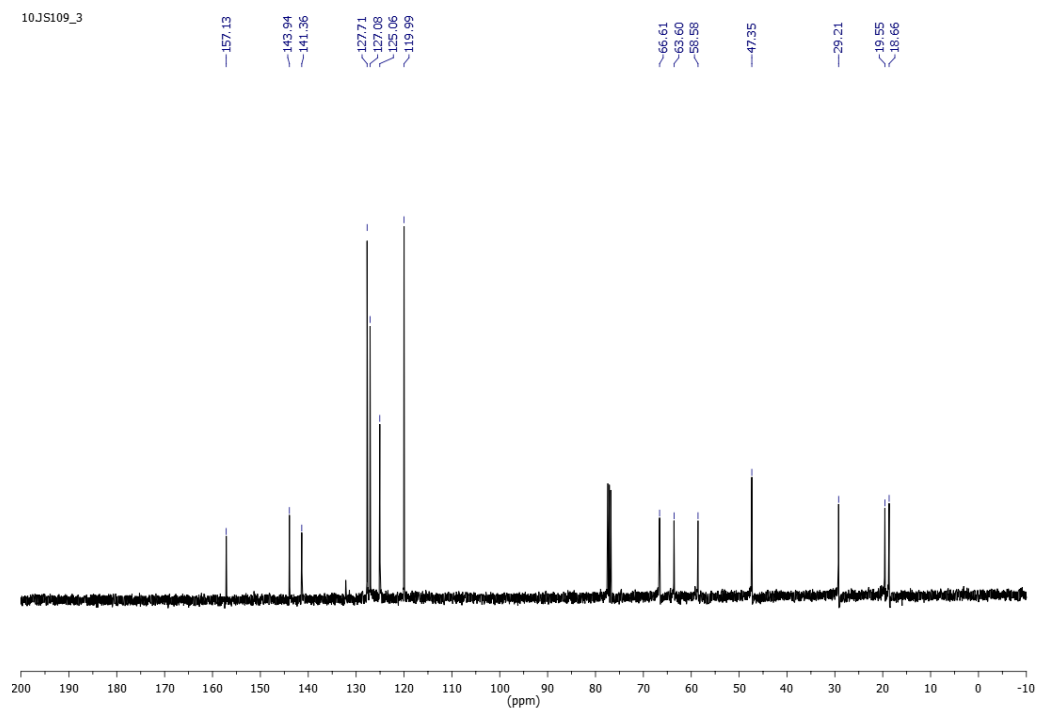
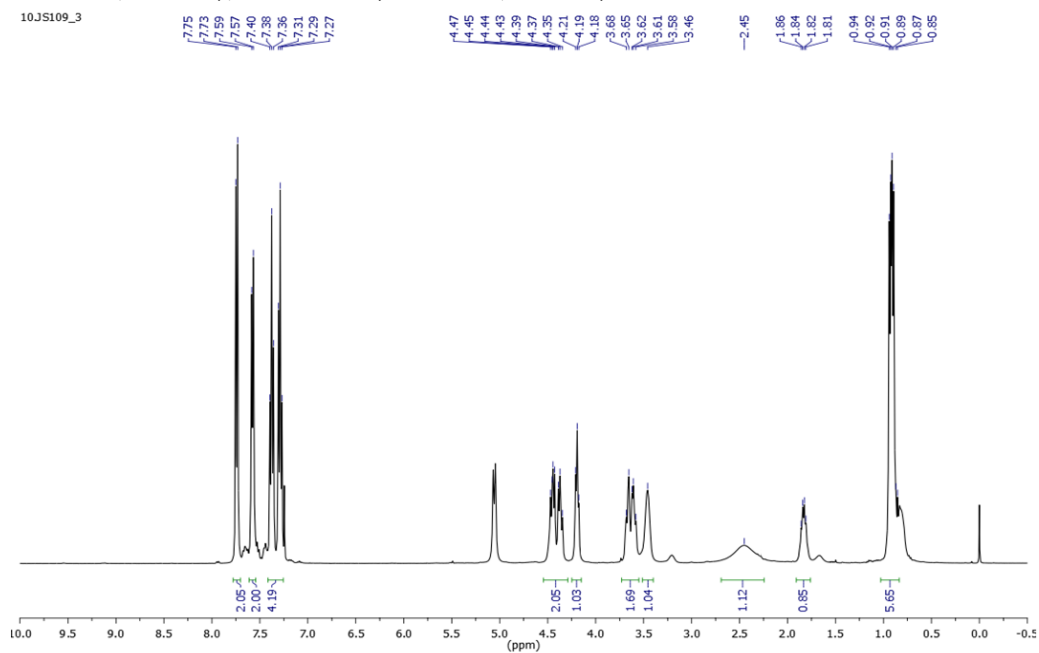


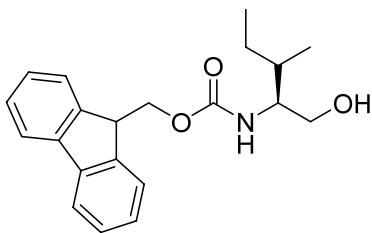
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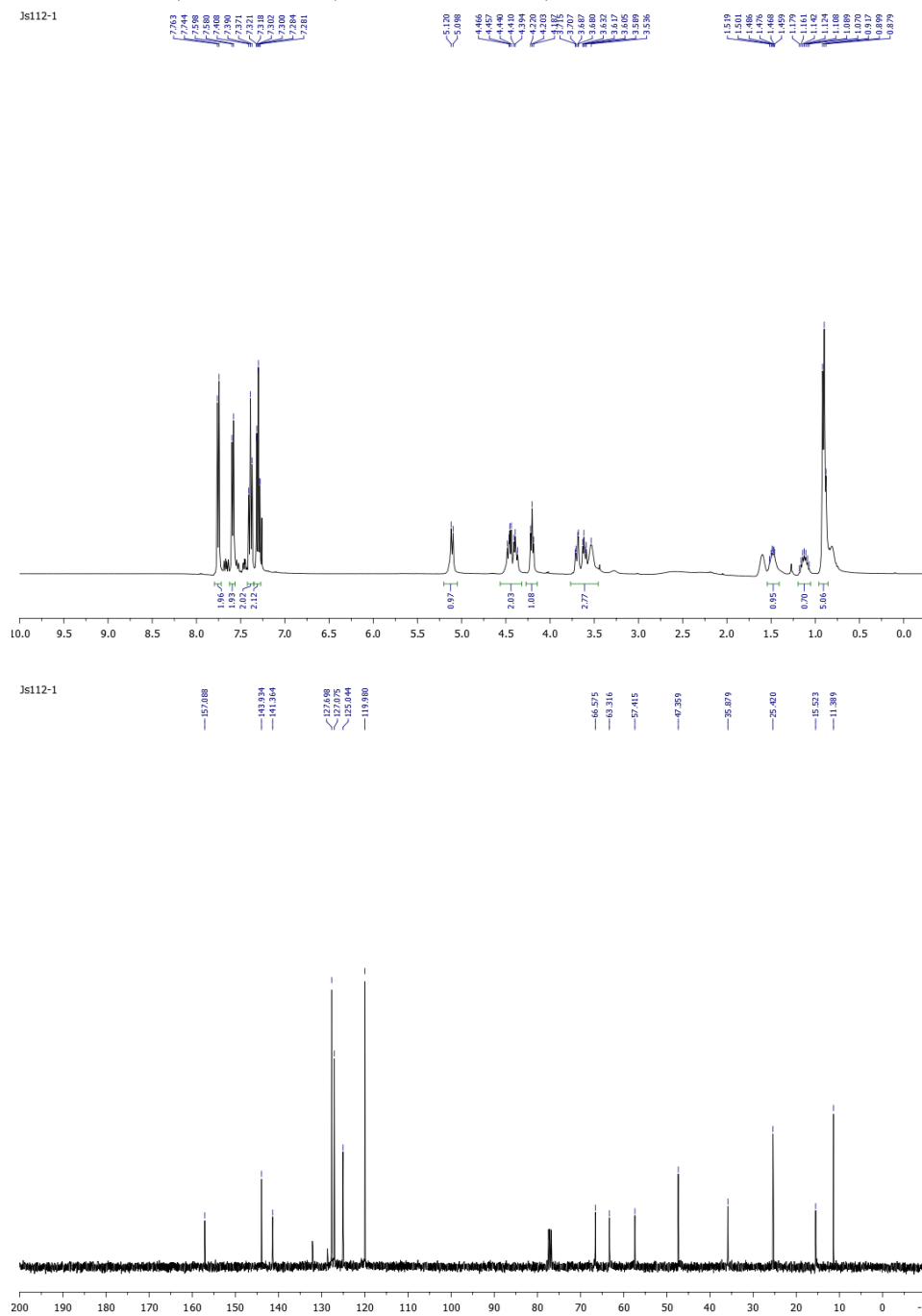


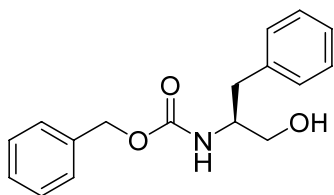
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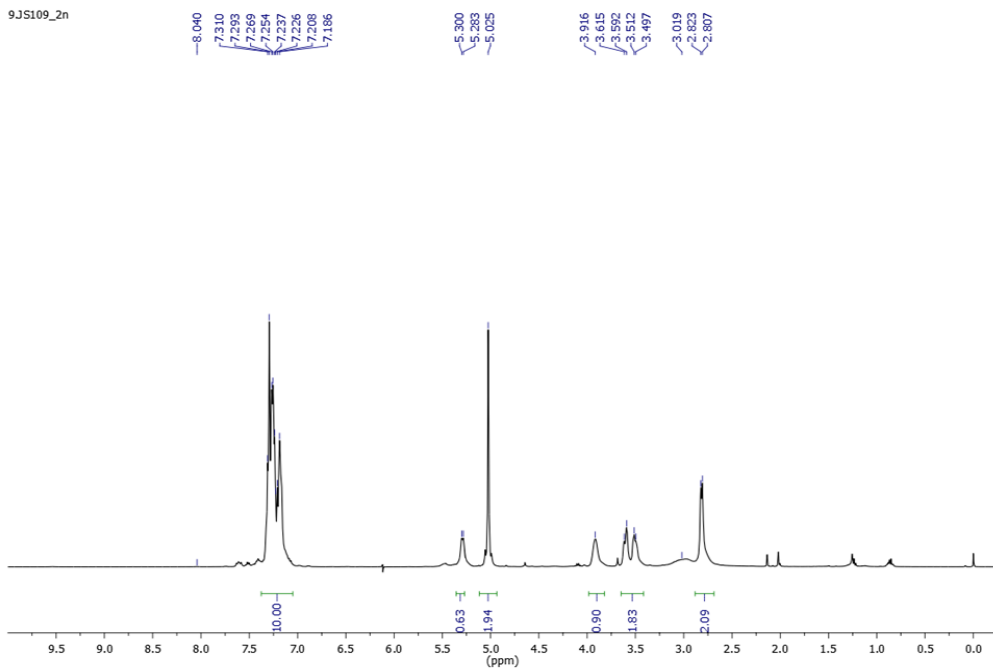
^1H NMR (400 MHz, CDCl_3); ^{13}C NMR (100 MHz, CDCl_3)





^1H NMR (400 MHz, CDCl_3); ^{13}C NMR (100 MHz, CDCl_3)

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