Electronic Supplementary Information

4-Hydroxymethyl-Substituted Oxazolidinone Synthesis by Tetraarylphosphonium Salt-Catalyzed Reactions of Glycidols and Isocyanates

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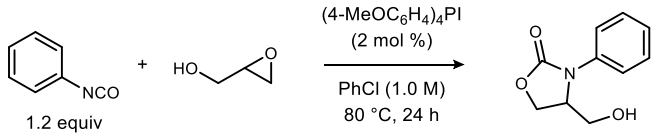
General information

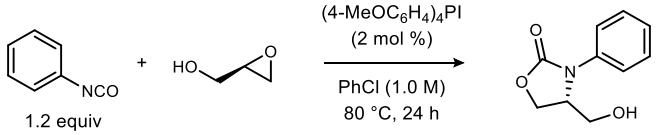
All reagents and solvents were commercial grade and purified prior to use when necessary. Tetrahydrofuran (THF), diethyl ether (Et_2O), and dichloromethane (CH_2Cl_2) were dried by passage through a column of activated alumina as described by Grubbs.[1] Thin layer chromatography (TLC) was performed using TLC aluminum sheets from Merck (silica gel 60 F_{254}, 200 μm), and flash chromatography utilized silica gel from FUJIFILM Wako Pure Chemical Corporation (Wakogel® C-300HG). Products were visualized by ultraviolet (UV) light, iodine (I_2), and/or a stain [phosphomolybdic acid (PMA), 4-anisaldehyde (AA), potassium permanganate ($KMnO_4$) solutions]. High-performance liquid chromatography (HPLC) was performed on a Jasco HPLC system using Daicel chiral columns (25 cm x 4.6 mm). Optical rotations were measured on a Jasco P-1010 polarimeter with a halogen lamp and are reported as follows; $[\alpha]^{T\,^{\circ}C}_{D}$ (c = g/100 mL, solvent). Melting points were measured on a Yanaco micro melting point apparatus and were not corrected. Nuclear magnetic resonance (NMR) spectra were acquired on a Bruker Fourier 300 (300 MHz). Chemical shifts are measured relative to residual solvent peaks as an internal standard set to 7.26 and 77.0 for $CDCl_3$ (or 0.00 for TMS). Data are reported as follows: chemical shift (ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, qui = quintet, br = broad, m = multiplet), coupling constants (Hz), and integration. Infrared (IR) spectra were recorded on a Jasco FT/IR-4200 spectrophotometer and are reported in wavenumbers (cm^{-1}). All compounds were analyzed as neat films on a potassium bromide (KBr) plate. Mass spectra were recorded on a Bruker micrOTOF II mass spectrometer by the ionization method noted. A post-acquisition gain correction was applied using sodium formate (HCO_2Na) as the lock mass.

General procedure for the reaction of glycidols with isocyanates

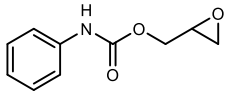
To an oven-dried test tube equipped with a stir bar was added glycidol (**1a**) (111.1 mg, 1.5 mmol, 1.0 equiv), tetrakis(4-methoxyphenyl)phosphonium iodide (17.1 mg, 30 μmol, 2 mol %), PhCl (1.5 mL, 1.0 M), and isocyanate **2** (1.2 equiv). The mixture was stirred at 80 °C for 24 h under argon atmosphere, cooled to rt, and then concentrated. Flash column chromatography (SiO_2) followed by crystallization yielded oxazolidinone **4**.



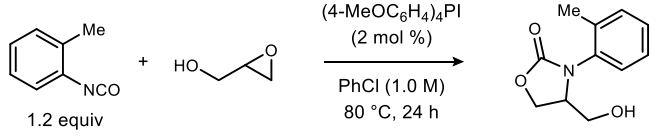
4-(Hydroxymethyl)-3-phenyloxazolidin-2-one (4a). Prepared according to the general procedure using isocyanate **2a** (196 μL, 1.8 mmol). Flash column chromatography (SiO_2: 15 g, Hexane/EtOAc = 2/1) and crystallization (Hexane/$CHCl_3$ = 2/1, 3 mL) yielded a white solid (263.9 mg, 91%). R_f = 0.15 (Hexane/EtOAc = 1/1) visualized with $KMnO_4$; Mp 112-113 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47-7.43 (m, 2H), 7.41-7.35 (m, 2H), 7.23-7.17 (m, 1H), 4.56-4.41 (m, 3H), 3.78-3.63 (m, 2H), 2.35 (t, *J* = 5.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.3 (C), 136.3 (C), 129.3 (CH), 125.6 (CH), 122.1 (CH), 64.5 (CH_2), 60.2 (CH_2), 57.4 (CH); ^{1}H NMR (300 MHz, CD_3OD) δ 7.51 (d, *J* = 8.4 Hz, 2H), 7.41 (dd, *J* = 8.4, 7.2 Hz, 2H), 7.22 (t, *J* = 7.2 Hz, 1H), 4.61-4.52 (m, 2H), 4.47-4.40 (m, 1H), 3.69-3.64 (m, 1H), 3.57-3.53 (m, 1H); ^{13}C NMR (75 MHz, CD_3OD) δ 158.7 (C), 137.9 (C), 130.2 (CH), 126.7 (CH), 124.0 (CH), 66.0 (CH_2), 60.4 (CH_2), 59.3 (CH); IR (KBr) 3366, 1712, 1498, 1421, 1225, 1144, 1051, 768 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{10}H_{11}NNaO_3$ $[M+Na]^+$ 216.0631, found 216.0642.



(*R*)-4a. Prepared according to the general procedure using (*R*)-**1a** and **2a**. Flash column chromatography (SiO_2: 30 g, Hexane/EtOAc = 1/1) yielded a white solid (248.4 mg, 86%). The product was determined to be 92% ee by chiral HPLC analysis (Chiralpak AD-3, Hexane/EtOH = 7/3, 0.5 mL/min, t_r(*major*) = 22.1 min, t_r(*minor*) = 28.1 min, 225 nm, 35 °C). $[\alpha]_D^{24}$ -68.4 (*c* 0.5, $CHCl_3$). The absolute configuration was determined by analogy with **4f**.

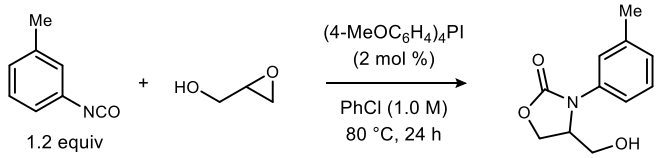


Glycidyl phenylcarbamate (3a). White solid; R_f = 0.20 (Hexane/EtOAc = 4/1) visualized with $KMnO_4$; Mp 55-56 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39-7.28 (m, 4H), 7.08 (t, *J* = 7.2 Hz, 1H), 6.79 (br s, 1H), 4.56 (dd, *J* = 12.3, 3.0 Hz, 1H), 3.98 (dd, *J* = 12.3, 6.4 Hz, 1H), 3.28 (dddd, *J* = 6.4, 4.2, 3.0, 2.7 Hz, 1H), 2.88 (dd, *J* = 4.8, 4.2 Hz, 1H), 2.70 (dd, *J* = 4.8, 2.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.0 (C), 137.5 (C), 129.1 (CH), 123.7 (CH), 118.8 (CH), 65.7 (CH_2), 49.6 (CH), 44.6 (CH_2); IR (KBr) 3274, 1742, 1315, 1224, 1065, 754 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{10}H_{11}NNaO_3$ $[M+Na]^+$ 216.0631, found 216.0653.

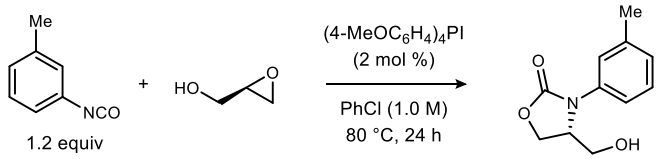


4-(Hydroxymethyl)-3-*o*-tolyloxazolidin-2-one (4b). Prepared according to the general procedure using isocyanate **2b** (223 μL, 1.8 mmol). Flash column chromatography (SiO_2: 15 g, Toluene/EtOAc = 3/1) yielded a colorless oil (229.4 mg, 74%). R_f = 0.15 (Hexane/EtOAc = 1/1) visualized with $KMnO_4$; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30-7.18 (m, 4H), 4.57 (t, *J* = 8.7 Hz, 1H), 4.46 (dd, *J* = 8.7, 6.0 Hz, 1H), 4.27-4.20 (m, 1H), 3.62

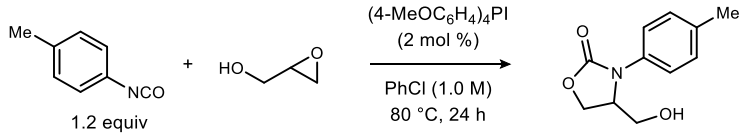
(dd, J = 11.4, 4.5 Hz, 1H), 3.57 (dd, J = 11.4, 3.3 Hz, 1H), 2.30 (s, 3H), 2.15 (br s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.9 (C), 136.5 (C), 134.2 (C), 131.5 (CH), 128.5 (CH), 127.7 (CH), 127.0 (CH), 65.0 (CH_2), 60.9 (CH_2), 59.0 (CH), 17.8 (CH_3); IR (KBr) 3431, 1730, 1496, 1419, 1220, 1146, 1043, 765, 723 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_{13}NNaO_3$ $[M+Na]^+$ 230.0788, found 230.0788.



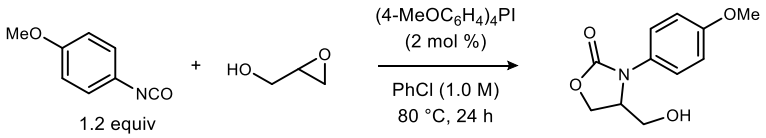
4-(Hydroxymethyl)-3-*m*-tolyloxazolidin-2-one (4c). Prepared according to the general procedure using isocyanate **2c** (232 µL, 1.8 mmol). Flash column chromatography (SiO_2: 15 g, Hexane/EtOAc = 2/1) and crystallization (Hexane/Et_2O = 1/1, 2 mL) yielded a white solid (273.4 mg, 88%). R_f = 0.15 (Hexane/EtOAc = 1/1) visualized with $KMnO_4$; Mp 108-109 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30 (s, 1H), 7.28-7.20 (m, 2H), 7.03 (d, J = 7.2 Hz, 1H), 4.56-4.44 (m, 3H), 3.82-3.77 (m, 1H), 3.68 (dd, J = 11.7, 6.0 Hz, 1H), 2.36 (s, 3H), 1.86 (br s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.4 (C), 139.4 (C), 136.2 (C), 129.1 (CH), 126.6 (CH), 123.0 (CH), 119.2 (CH), 64.5 (CH_2), 60.4 (CH_2), 57.6 (CH), 21.5 (CH_3); IR (KBr) 3328, 1712, 1497, 1420, 1240, 1134, 1054, 786, 756 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_{13}NNaO_3$ $[M+Na]^+$ 230.0788, found 230.0785.



(*R*)-4c. Prepared according to the general procedure using (*R*)-**1a** and **2c**. Flash column chromatography (SiO_2: 30 g, Hexane/EtOAc = 2/1) yielded a white solid (274.6 mg, 88%). The product was determined to be 98% ee by chiral HPLC analysis (Chiralpak AD-3, Hexane/EtOH = 9/1, 0.5 mL/min, t_r(*major*) = 71.4 min, t_r(*minor*) = 67.9 min, 225 nm, 35 °C). $[\alpha]_D^{24}$ -62.1 (*c* 0.5, $CHCl_3$). The absolute configuration was determined by analogy with **4f**.

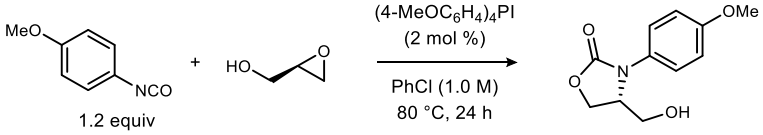


4-(Hydroxymethyl)-3-*p*-tolyloxazolidin-2-one (4d). Prepared according to the general procedure using isocyanate **2d** (227 µL, 1.8 mmol). Flash column chromatography (SiO_2: 15 g, Hexane/EtOAc = 2/1 (run 1), Toluene/EtOAc = 2/1 (run 2)) yielded a white solid (247.4 mg, 80%). R_f = 0.15 (Hexane/EtOAc = 1/1) visualized with $KMnO_4$; Mp 109-110 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31 (d, J = 8.4 Hz, 2H), 7.18 (d, J = 8.4 Hz, 2H), 4.53-4.38 (m, 3H), 3.75-3.60 (m, 2H), 2.42 (t, J = 5.7 Hz, 1H), 2.33 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.6 (C), 135.6 (C), 133.6 (C), 129.9 (CH), 122.5 (CH), 64.6 (CH_2), 60.3 (CH_2), 57.7 (CH), 20.8 (CH_3); IR (KBr) 3374, 1711, 1514, 1425, 1227, 1145, 1051, 826, 758 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_{11}NNaO_3$ $[M+Na]^+$ 230.0788, found 238.0797.

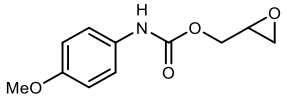


4-(Hydroxymethyl)-3-(4-methoxyphenyl)oxazolidin-2-one (4e). Prepared according to the general procedure using isocyanate **2e** (233 µL, 1.8 mmol). Flash column chromatography (SiO_2: 15 g, Hexane/EtOAc = 2/1) and crystallization (Hexane/$CHCl_3$ = 2/1, 3 mL) yielded a white solid (283.2 mg, 85%). R_f = 0.15

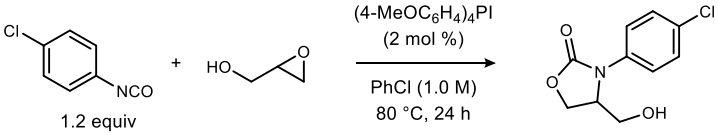
(Hexane/EtOAc = 1/1) visualized with $KMnO_4$; Mp 113-115 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34 (d, *J* = 9.0 Hz, 2H), 6.93 (d, *J* = 9.0 Hz, 2H), 4.55 (t, *J* = 8.1 Hz, 1H), 4.46-4.36 (m, 2H), 3.81 (s, 3H), 3.81-3.76 (m, 1H), 3.66 (d, *J* = 11.7 Hz, 1H), 1.65 (br s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.8 (C), 156.9 (C), 128.9 (C), 125.0 (CH), 114.6 (CH), 64.5 (CH_2), 60.4 (CH_2), 58.3 (CH), 55.5 (CH_3); IR (KBr) 3398, 1705, 1513, 1250, 1139, 836, 758 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_{13}NNaO_4$ $[M+Na]^+$ 246.0737, found 246.0748.



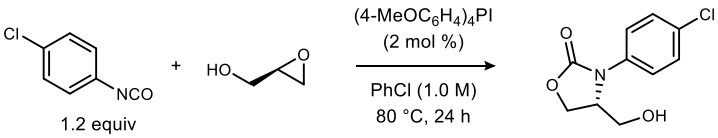
(*R*)-4e. Prepared according to the general procedure using (*R*)-**1a** and **2e**. Flash column chromatography (SiO_2: 40 g, CH_2Cl_2/MeOH = 200/1) yielded a white solid (270.2 mg, 81%). The product was determined to be 93% ee by chiral HPLC analysis (Chiralpak AD-3, Hexane/EtOH = 7/3, 0.5 mL/min, t_r(*major*) = 22.6 min, t_r(*minor*) = 36.3 min, 225 nm, 35 °C). $[\alpha]_D^{20}$ -55.1 (*c* 0.1, $CHCl_3$). The absolute configuration was determined by analogy with **4f**.



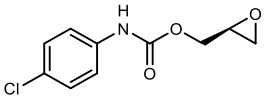
Oxiran-2-ylmethyl-4-methoxyphenylcarbamate (3e). To an oven-dried test tube equipped with a stir bar was added glycidol (**1a**) (444.6 mg, 6.0 mmol, 1.2 equiv), PhCl (5.0 mL, 1.0 M), and isocyanate **2e** (648 μL, 5.0 mmol, 1.0 equiv). The reaction mixture was stirred at 35 °C for 24 h under argon atmosphere, and then concentrated. Flash column chromatography (SiO_2: 25 g, Hexane/EtOAc = 1/1) yielded carbamate **3e** as a white solid (804.7 mg, 80%). R_f = 0.20 (Hexane/EtOAc = 4/1) visualized with $KMnO_4$; Mp 59-60 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30-7.26 (m, 2H), 6.86 (d, *J* = 9.0 Hz, 2H), 6.66 (br s, 1H), 4.54 (dd, *J* = 12.3, 3.0 Hz, 1H), 3.97 (dd, *J* = 12.3, 6.3 Hz, 1H), 3.79 (s, 3H), 3.27 (dddd, *J* = 6.3, 4.2, 3.0, 2.7 Hz, 1H), 2.87 (dd, *J* = 4.8, 4.2 Hz, 1H), 2.69 (dd, *J* = 4.8, 2.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.2 (C), 153.4 (C), 130.6 (C), 120.8 (CH), 114.3 (CH), 65.6 (CH_2), 55.5 (CH_3), 49.7 (CH), 44.6 (CH_2); IR (KBr) 3333, 1703, 1526, 1233, 1065, 768 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_{13}NNaO_4$ $[M+Na]^+$ 246.0737, found 246.0733.



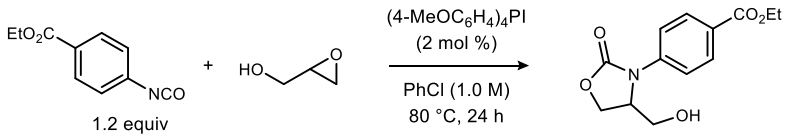
3-(4-Chlorophenyl)-4-(hydroxymethyl)oxazolidin-2-one (4f). Prepared according to the general procedure using isocyanate **2f** (230 μL, 1.8 mmol). Flash column chromatography (SiO_2: 15 g, Hexane/EtOAc = 2/1) and crystallization (Et_2O/Hexane = 2/1, 3 mL) yielded a white solid (304.2 mg, 89%). R_f = 0.20 (Hexane/EtOAc = 1/1) visualized with $KMnO_4$; Mp 90-92 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42 (d, *J* = 9.0 Hz, 2H), 7.35 (d, *J* = 9.0 Hz, 2H), 4.57-4.44 (m, 3H), 3.82-3.77 (m, 1H), 3.71-3.66 (m, 1H), 2.11 (br s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.0 (C), 135.0 (C), 130.9 (C), 129.4 (CH), 123.1 (CH), 64.5 (CH_2), 60.3 (CH_2), 57.3 (CH); IR (KBr) 3378, 1713, 1496, 1425, 1223, 1143, 1051, 835, 758 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{10}H_{10}ClNNaO_3$ $[M+Na]^+$ 250.0241, found 250.0245.



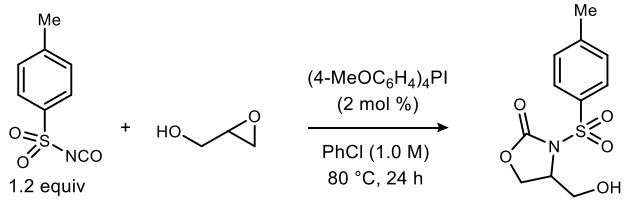
(*R*)-4f. Prepared according to the general procedure using (*R*)-**1a** and **2f**. Flash column chromatography (SiO_2: 20 g, Toluene/EtOAc = 2/1) yielded a white solid (308.6 mg, 90%). The product was determined to be 91% ee by chiral HPLC analysis (Chiralpak AD-3, Hexane/EtOH = 7/3, 0.5 mL/min, t_r(*major*) = 16.7 min, t_r(*minor*) = 21.5 min, 225 nm, 35 °C). $[\alpha]_D^{24}$ -53.0 (*c* 0.1, $CHCl_3$). The absolute configuration was determined by X-ray crystallographic analysis (CCDC 1898009). A single crystal of **4f** was grown in toluene.



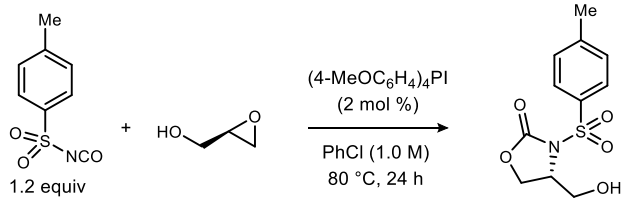
Oxiran-2-ylmethyl-4-chlorophenylcarbamate ((*S*)-3f). To an oven-dried test tube equipped with a stir bar was added (*R*)-glycidol ((*R*)-**1a**) (226.7 mg, 3.6 mmol, 1.2 equiv), PhCl (3.0 mL, 1.0 M), and isocyanate **2f** (384 μL, 3.0 mmol, 1.0 equiv). The reaction mixture was stirred at 35 °C for 24 h under argon atmosphere, and then concentrated. Flash column chromatography (SiO_2: 25 g, Hexane/EtOAc = 1/1) yielded carbamate (*S*)-**3f** as a white solid (612.7 mg, 90%). The product was determined to be 99% ee by chiral HPLC analysis (Chiralcel OD-3, Hexane/EtOH = 9/1, 0.5 mL/min, t_r(*major*) = 18.6 min, t_r(*minor*) = 19.6 min, 225 nm, 35 °C). $[\alpha]_D^{20}$ +42.8 (*c* 0.05, $CHCl_3$); R_f = 0.25 (Hexane/EtOAc = 4/1) visualized with $KMnO_4$; Mp 53-54 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35-7.25 (m, 4H), 6.81 (br s, 1H), 4.56 (dd, *J* = 12.3, 2.7 Hz, 1H), 3.97 (dd, *J* = 12.3, 6.6 Hz, 1H), 3.27 (ddt, *J* = 6.6, 4.2, 2.7 Hz, 1H), 2.88 (dd, *J* = 4.8, 4.2 Hz, 1H), 2.69 (dd, *J* = 4.8, 2.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 152.9 (C), 136.2 (C), 129.1 (CH), 128.7 (C), 120.0 (CH), 65.9 (CH_2), 49.6 (CH), 44.6 (CH_2); IR (KBr) 3281, 1732, 1605, 1546, 1232, 1065, 858, 825, 731 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{10}H_{10}ClNNaO_3$ $[M+Na]^+$ 250.0241, found 250.0229.



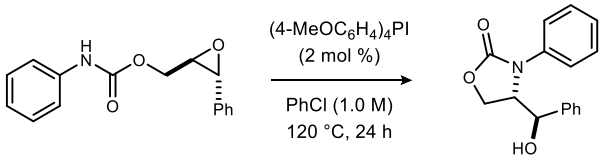
Ethyl 4-(4-(hydroxymethyl)-2-oxooxazolidin-3-yl)benzoate (4g). Prepared according to the general procedure using isocyanate **2g** (358.5 mg, 1.8 mmol). Flash column chromatography (SiO_2: 15 g, Hexane/EtOAc = 2/1) yielded a white solid (337.4 mg, 82%). R_f = 0.25 (Hexane/EtOAc = 1/1) visualized with $KMnO_4$; Mp 121-122 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.03 (d, *J* = 8.7 Hz, 2H), 7.60 (d, *J* = 8.7 Hz, 2H), 4.63-4.48 (m, 3H), 4.36 (q, *J* = 7.2 Hz, 2H), 3.87-3.74 (m, 2H), 2.40 (br s, 1H), 1.39 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.0 (C), 155.3 (C), 140.7 (C), 130.8 (CH), 126.6 (C), 119.7 (CH), 64.5 (CH_2), 61.1 (CH_2), 60.4 (CH_2), 56.7 (CH), 14.3 (CH_3); IR (KBr) 3483, 1746, 1608, 1516, 1410, 1284, 1211, 1131, 1044, 855, 768 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_{15}NNaO_5$ $[M+Na]^+$ 288.0842, found 288.0837.



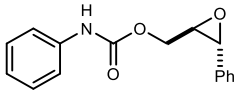
4-(Hydroxymethyl)-3-tosyloxazolidin-2-one (4h). Prepared according to the general procedure using isocyanate **2h** (274 μL, 1.8 mmol). Flash column chromatography (SiO_2: 15 g, Toluene/EtOAc = 2/1) and methanol wash yielded a white solid (359.7 mg, 88%). R_f = 0.30 (Toluene/EtOAc = 1/1) visualized with $KMnO_4$; Mp 122-123 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.4 Hz, 2H), 7.37 (d, *J* = 8.4 Hz, 2H), 4.49 (dddd, *J* = 8.4, 4.8, 4.5, 2.7 Hz, 1H), 4.39 (t, *J* = 8.4 Hz, 1H), 4.35 (dd, *J* = 8.4, 4.8 Hz, 1H), 4.08 (ddd, *J* = 12.0, 5.1, 4.5 Hz, 1H), 3.85 (ddd, *J* = 12.0, 5.1, 2.7 Hz, 1H), 2.46 (s, 3H), 2.14 (t, *J* = 5.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 152.4 (C), 145.9 (C), 134.4 (C), 129.9 (CH), 128.4 (CH), 65.4 (CH_2), 62.6 (CH_2), 57.9 (CH), 21.7 (CH_3); IR (KBr) 3506, 1756, 1362, 1201, 1173, 755, 669 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_{13}NNaO_5S$ $[M+Na]^+$ 294.0407, found 294.0396.



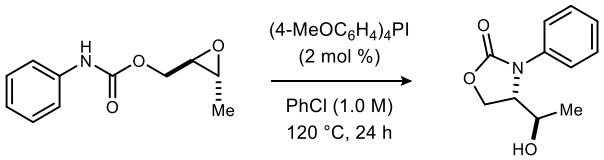
(*R*)-4h. Prepared according to the general procedure using (*R*)-**1a** and **2h**. Flash column chromatography (SiO_2: 15 g, Toluene/EtOAc = 4/1) yielded a white solid (348.7 mg, 86%). The product was determined to be 98% ee by chiral HPLC analysis (Chiralpak IC, Hexane/EtOH = 8/2, 0.5 mL/min, t_r(*major*) = 26.8 min, t_r(*minor*) = 28.8 min, 225 nm, 35 °C). $[\alpha]_D^{23}$ -38.1 (*c* 0.1, $CHCl_3$). The absolute configuration was determined by analogy with **4f**.



***anti*-4-Hydroxy(phenyl)methyl-3-phenyloxazolidin-2-one (4i).** To an oven-dried test tube equipped with a stir bar was added carbamate **3i** (80.8 mg, 0.30 mmol, 1.0 equiv), tetrakis(4-methoxyphenyl)phosphonium iodide (3.4 mg, 6.0 μmol, 2 mol %), and PhCl (0.3 mL, 1.0 M). The reaction mixture was stirred at 120 °C for 24 h under argon atmosphere, cooled to rt, and then concentrated. Flash column chromatography (SiO_2: 8 g, Hexane/EtOAc = 3/1) yielded a white solid (77.7 mg, 96%). R_f = 0.30 (Hexane/EtOAc = 1/1) visualized with $KMnO_4$; Mp 145-147 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.53-7.49 (m, 2H), 7.44-7.26 (m, 7H), 7.25-7.19 (m, 1H), 5.01 (dd, *J* = 3.6, 2.4 Hz, 1H), 4.60 (ddd, *J* = 8.7, 5.1, 2.4 Hz, 1H), 4.51 (dd, *J* = 8.7, 5.1 Hz, 1H), 4.13 (t, *J* = 8.7 Hz, 1H), 2.62 (d, *J* = 3.6 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.4 (C), 138.8 (C), 136.3 (C), 129.4 (CH), 128.8 (CH), 128.1 (CH), 125.7 (CH), 125.4 (CH), 122.6 (CH), 69.3 (CH), 61.9 (CH), 61.5 (CH_2); IR (KBr) 3423, 1754, 1500, 1430, 1233, 1144, 762, 736, 694 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{16}H_{15}NNaO_3$ $[M+Na]^+$ 292.0944, found 292.0958.

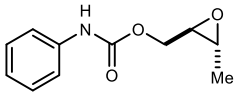


(*trans*-2,3-3-Phenyloxiran-2-yl)methyl phenylcarbamate (3i). To an oven-dried test tube equipped with a stir bar was added *trans*-3-phenylglycidol[2] (150.2 mg, 1.0 mmol, 1.0 equiv), PhCl (1.0 mL, 1.0 M), and phenyl isocyanate (**2a**, 131 μL, 1.2 mmol, 1.2 equiv). The reaction mixture was stirred at 80 °C for 24 h under argon atmosphere, and then concentrated. Flash column chromatography (SiO_2: 10 g, Hexane/EtOAc = 4/1) yielded carbamate **3i** as a white solid (236.1 mg, 88%). R_f = 0.10 (Hexane/EtOAc = 4/1) visualized with $KMnO_4$; Mp 83-85 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.25 (m, 9H), 7.08 (t, *J* = 7.2 Hz, 1H), 6.80 (br s, 1H), 4.61 (dd, *J* = 12.3, 3.3 Hz, 1H), 4.18 (dd, *J* = 12.3, 6.0 Hz, 1H), 3.85 (d, *J* = 2.1 Hz, 1H), 3.33 (ddd, *J* = 6.0, 3.3, 2.1 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.0 (C), 137.5 (C), 136.1 (C), 129.1 (CH), 128.6 (CH), 128.5 (CH), 125.7 (CH), 123.7 (CH), 118.8 (CH), 64.9 (CH_2), 59.5 (CH), 56.4 (CH); IR (KBr) 3301, 1703, 1603, 1550, 1526, 1447, 1253, 762, 719, 695 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{16}H_{15}NNaO_3$ $[M+Na]^+$ 292.0944, found 292.0961.

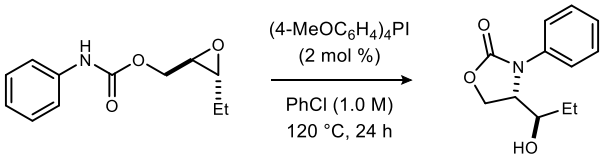


***anti*-4-(1-Hydroxyethyl)-3-phenyloxazolidin-2-one (4j).** To an oven-dried test tube equipped with a stir bar was added carbamate **3j** (62.3 mg, 0.30 mmol, 1.0 equiv), tetrakis(4-methoxyphenyl)phosphonium iodide (3.4 mg, 6.0 μmol, 2 mol %), and PhCl (0.3 mL, 1.0 M). The reaction mixture was stirred at 120 °C for 24 h under argon atmosphere, cooled to rt, and then concentrated. Flash column chromatography (SiO_2: 8 g, Hexane/EtOAc = 3/1) yielded a white solid (58.2 mg, 94%). R_f = 0.20 (Hexane/EtOAc = 1/1) visualized with $KMnO_4$; Mp 118-120 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45-7.37 (m, 4H), 7.24-7.19 (m, 1H), 4.53 (dd, *J* = 8.7, 5.7 Hz, 1H), 4.44 (t, *J* = 8.7 Hz, 1H), 4.35 (ddd, *J* = 8.7, 5.7, 2.1 Hz, 1H), 4.11-4.05 (m, 1H), 2.02 (br s, 1H), 1.13 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.4 (C), 136.4 (C), 129.3 (CH), 125.8 (CH), 122.7 (CH), 63.9 (CH), 61.8 (CH_2), 61.2 (CH), 18.2 (CH_3); IR (KBr) 3400, 1713, 1500, 1424, 1289, 1227, 1146, 759, 693 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_{13}NNaO_3$ $[M+Na]^+$ 230.0788, found 230.0797.

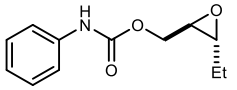
The relative configuration was determined by X-ray crystallographic analysis (CCDC 1898013). A single crystal of **4i** was grown in $CHCl_3$.



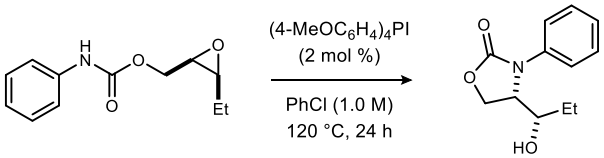
***trans*-1-((*N*-Phenylcarbamoyl)oxy)-2,3-epoxybutane (3j).**[3] To a solution of crotyl alcohol (649.1 mg, 9.0 mmol, 1.0 equiv) in CH_2Cl_2 (25.7 mL, 0.35 M) was added *m*-CPBA (contains ca. 30% H_2O, 4.44 g, 18 mmol, 2.0 equiv) at 0 °C. The mixture was stirred overnight at rt, filtered through a pad of silica gel with CH_2Cl_2, and then concentrated (30 °C, ca. 300 mmHg) to remove CH_2Cl_2. The residue was treated with hexane (30 mL), and the precipitates were removed by filtration with Celite®. Phenyl isocyanate (**2a**, 2.95 mL, 27 mmol) was added to the hexane solution, and the mixture was stirred at 35 °C for 3 days. The resulting mixture was concentrated, and purified by flash column chromatography (SiO_2: 30 g, Toluene/EtOAc = 25/1 (run 1), 40 g, $CHCl_3$ (run 2)) to give carbamate **3j** as a white solid (652.6 mg, 35%). R_f = 0.20 ($CHCl_3$) visualized with $KMnO_4$; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 (d, *J* = 7.5 Hz, 2H), 7.30 (t, *J* = 7.5 Hz, 2H), 7.07 (t, *J* = 7.5 Hz, 1H), 6.85 (br s, 1H), 4.50 (dd, *J* = 12.0, 2.7 Hz, 1H), 3.99 (dd, *J* = 12.0, 6.3 Hz, 1H), 3.02-2.95 (m, 2H), 1.36 (d, *J* = 5.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.1 (C), 137.6 (C), 129.0 (CH), 123.6 (CH), 118.8 (CH), 65.3 (CH_2), 56.5 (CH), 52.5 (CH), 17.2 (CH_3). Characterization data matched the literature.



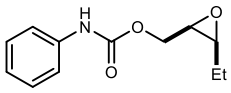
***anti*-4-(1-Hydroxypropyl)-3-phenyloxazolidin-2-one (4k).** To an oven-dried test tube equipped with a stir bar was added carbamate **3k** (66.4 mg, 0.30 mmol, 1.0 equiv), tetrakis(4-methoxyphenyl)phosphonium iodide (3.4 mg, 6.0 μmol, 2 mol %), and PhCl (0.3 mL, 1.0 M). The reaction mixture was stirred at 120 °C for 24 h under argon atmosphere, cooled to rt, and then concentrated. Flash column chromatography (SiO_2: 10 g, Hexane/EtOAc = 3/1) yielded a white solid (60.4 mg, 91%). R_f = 0.30 (Hexane/EtOAc = 1/1) visualized with $KMnO_4$; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44-7.35 (m, 4H), 7.24-7.18 (m, 1H), 4.54-4.36 (m, 3H), 3.79-3.74 (m, 1H), 2.27 (d, *J* = 3.9 Hz, 1H), 1.49-1.29 (m, 2H), 0.96 (d, *J* = 7.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.5 (C), 136.3 (C), 129.3 (CH), 125.7 (CH), 122.6 (CH), 69.0 (CH), 61.8 (CH_2), 60.2 (CH), 25.4 (CH_2), 10.2 (CH_3); IR (KBr) 3443, 1712, 1504, 1421, 1220, 1143, 759 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{12}H_{15}NNaO_3$ $[M+Na]^+$ 244.0944, found 244.0963.



(*trans*-2,3-3-Ethyloxiran-2-yl)methyl phenylcarbamate (3k). To a solution of (*E*)-pent-2-enyl phenyl carbamate (656.8 mg, 3.2 mmol) in CH_2Cl_2 (6.4 mL) and satd aq $NaHCO_3$ (0.8 mL) was added *m*-CPBA (contains ca. 30% H_2O, 986.1 mg, 4.0 mmol). The mixture was stirred overnight at rt, filtered through a pad of N-H silica gel/$MgSO_4$ with CH_2Cl_2, and then concentrated. Flash column chromatography (SiO_2: 30 g, Hexane/EtOAc = 10/1) to give carbamate **3k** as a white solid (536.9 mg, 76%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39-7.37 (m, 2H), 7.33-7.28 (m, 2H), 7.10-7.04 (m, 1H), 6.78 (br s, 1H), 4.51 (dd, *J* = 12.0, 2.7 Hz, 1H), 3.99 (dd, *J* = 12.0, 6.3 Hz, 1H), 3.05 (ddd, *J* = 6.3, 2.7, 2.1 Hz, 1H), 2.89 (td, *J* = 5.4, 2.1 Hz, 1H), 1.73-1.53 (m, 2H), 1.01 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.1 (C), 137.6 (C), 129.1 (CH), 123.7 (CH), 118.8 (CH), 65.5 (CH_2), 57.6 (CH), 55.3 (CH), 24.6 (CH_2), 9.7 (CH_3); HRMS (ESI): Exact mass calcd for $C_{12}H_{15}NNaO_3$ $[M+Na]^+$ 244.0944, found 244.0917.



***syn*-4-(1-Hydroxypropyl)-3-phenyloxazolidin-2-one (4l).** To an oven-dried test tube equipped with a stir bar was added carbamate **3l** (66.4 mg, 0.30 mmol, 1.0 equiv), tetrakis(4-methoxyphenyl)phosphonium iodide (3.4 mg, 6.0 μmol, 2 mol %), and PhCl (0.3 mL, 1.0 M). The reaction mixture was stirred at 120 °C for 24 h under argon atmosphere, cooled to rt, and then concentrated. Flash column chromatography (SiO_2: 10 g, Hexane/EtOAc = 3/1) yielded a white solid (59.0 mg, 89%). R_f = 0.30 (Hexane/EtOAc = 1/1) visualized with $KMnO_4$; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51-7.47 (m, 2H), 7.41-7.34 (m, 2H), 7.22-7.16 (m, 1H), 4.63-4.57 (m, 1H), 4.48-4.41 (m, 2H), 3.76 (dq, *J* = 8.7, 4.5 Hz, 1H), 2.29 (d, *J* = 4.5 Hz, 1H), 1.50-1.28 (m, 2H), 0.90 (d, *J* = 7.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.2 (C), 137.1 (C), 129.2 (CH), 125.5 (CH), 121.8 (CH), 71.9 (CH), 63.6 (CH_2), 59.5 (CH), 23.4 (CH_2), 18.2 (CH_3); IR (KBr) 3387, 1708, 1505, 1428, 1225, 1144, 761 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{12}H_{15}NNaO_3$ $[M+Na]^+$ 244.0944, found 244.0950.



(*cis*-2,3-3-Ethyloxiran-2-yl)methyl phenylcarbamate (3l). To a solution of (*Z*)-pent-2-enyl phenyl carbamate (656.8 mg, 3.2 mmol) in CH_2Cl_2 (6.4 mL) and satd aq $NaHCO_3$ (0.8 mL) was added *m*-CPBA (contains ca. 30% H_2O, 986.1 mg, 4.0 mmol). The mixture was stirred overnight at rt, filtered through a pad of N-H silica gel/$MgSO_4$ with CH_2Cl_2, and then concentrated. Flash column chromatography (SiO_2: 30 g, Hexane/EtOAc = 10/1) to give carbamate **3l** as a white solid (531.0 mg, 75%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.37 (m, 2H), 7.34-7.29 (m, 2H), 7.11-7.05 (m, 1H), 6.71 (br s, 1H), 4.48 (dd, *J* = 12.0, 3.9 Hz, 1H), 4.10 (dd, *J* = 12.0, 7.2 Hz, 1H), 3.25 (dt, *J* = 7.2, 4.2 Hz, 1H), 3.02 (ddd, *J* = 6.6, 6.0, 4.2 Hz, 1H), 1.73-1.51 (m, 2H), 1.08 (d, *J* = 7.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.1 (C), 137.6 (C), 129.1 (CH), 123.7 (CH), 118.7 (CH), 63.6 (CH_2), 57.8 (CH), 54.1 (CH), 21.4 (CH_2), 10.9 (CH_3); HRMS (ESI): Exact mass calcd for $C_{12}H_{15}NNaO_3$ $[M+Na]^+$ 244.0944, found 244.0957.

Catalyst synthesis

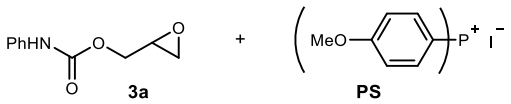
Phosphonium salts were prepared using a modified procedure of the literature.[4]

Tetrakis(4-methoxyphenyl)phosphonium iodide. Under argon atmosphere in a sealed tube equipped with a stir bar, 4-iodoanisole (234.1 mg, 1.0 mmol, 1.0 equiv), tris(4-methoxyphenyl)phosphine (334.4 mg, 1.0 mmol, 1.0 equiv), and $Pd_2(dba)_3$ (18.3 mg, 20 μmol, 2 mol %) were reacted in toluene (4.0 mL) at 145 °C for 8 h. After cooling to room temperature, the mixture was filtered over Celite®, rinsed with MeOH, and then concentrated. The residue was triturated with MeOH/THF (0.3 mL/10 mL) to afford the product as a white solid (435.1 mg, 77%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50 (dd, *J* = 12.3, 9.0 Hz, 8H), 7.23 (dd, *J* = 9.0, 2.7 Hz, 8H), 3.96 (s, 12H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.8 (d, *J* = 3.3 Hz, C), 136.0 (d, *J* = 12.0 Hz, CH), 116.3 (d, *J* = 13.8 Hz, CH), 109.0 (d, *J* = 98.4 Hz, C), 56.3 (CH_3); ^{31}P NMR (121 MHz, $CDCl_3$) δ 20.5.

Tetrakis(4-fluorophenyl)phosphonium iodide. Under argon atmosphere in a sealed tube equipped with a stir bar, 4-fluoroiodobenzene (66.6 mg, 0.3 mmol, 1.0 equiv), tris(4-fluorophenyl)phosphine (94.9 mg, 0.3 mmol, 1.0 equiv), and $Pd_2(dba)_3$ (5.5 mg, 6.0 μmol, 2 mol %) were reacted in toluene (1.2 mL) at 145 °C for 24 h. After cooling to room temperature, the mixture was filtered over Celite®, rinsed with MeOH, and then concentrated. The residue was wash with THF (5 mL x 3) to afford the product as a white solid (128.3 mg, 80%). ^{1}H NMR (300 MHz, CD_3OD) δ 7.90-7.79 (m, 8H), 7.62-7.53 (m, 8H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.8 (dd, *J* = 259.2, 3.6 Hz, C), 139.3 (dd, *J* = 12.6, 9.9 Hz, CH), 119.6 (dd, *J* = 22.5, 15.0 Hz, CH), 115.4 (dd, *J* = 95.7, 3.6 Hz, C); ^{19}F NMR (282 MHz, CD_3OD) δ -102.2 (d, *J* = 2.0 Hz); ^{31}P NMR (121 MHz, CD_3OD) δ 21.7.

Appendix

Table S1. Δδ (NH) of **3a** for each molar ratio.



3a:**PS**	**3a**	Phosphonium Salt (**PS**)	$CDCl_3$	δ (NH)	Δδ
1:1	8.7 mg (45 μmol)	25.6 mg (45 μmol)	600 μL	6.98	0.23
1:0.8	8.7 mg (45 μmol)	20.5 mg (36 μmol)	600 μL	6.94	0.19
1:0.6	8.7 mg (45 μmol)	15.3 mg (27 μmol)	600 μL	6.89	0.14
1:0.4	8.7 mg (45 μmol)	10.2 mg (18 μmol)	600 μL	6.85	0.10
1:0.2	8.7 mg (45 μmol)	5.1 mg (9 μmol)	600 μL	6.79	0.04
1:0	8.7 mg (45 μmol)	-	600 μL	6.75	-

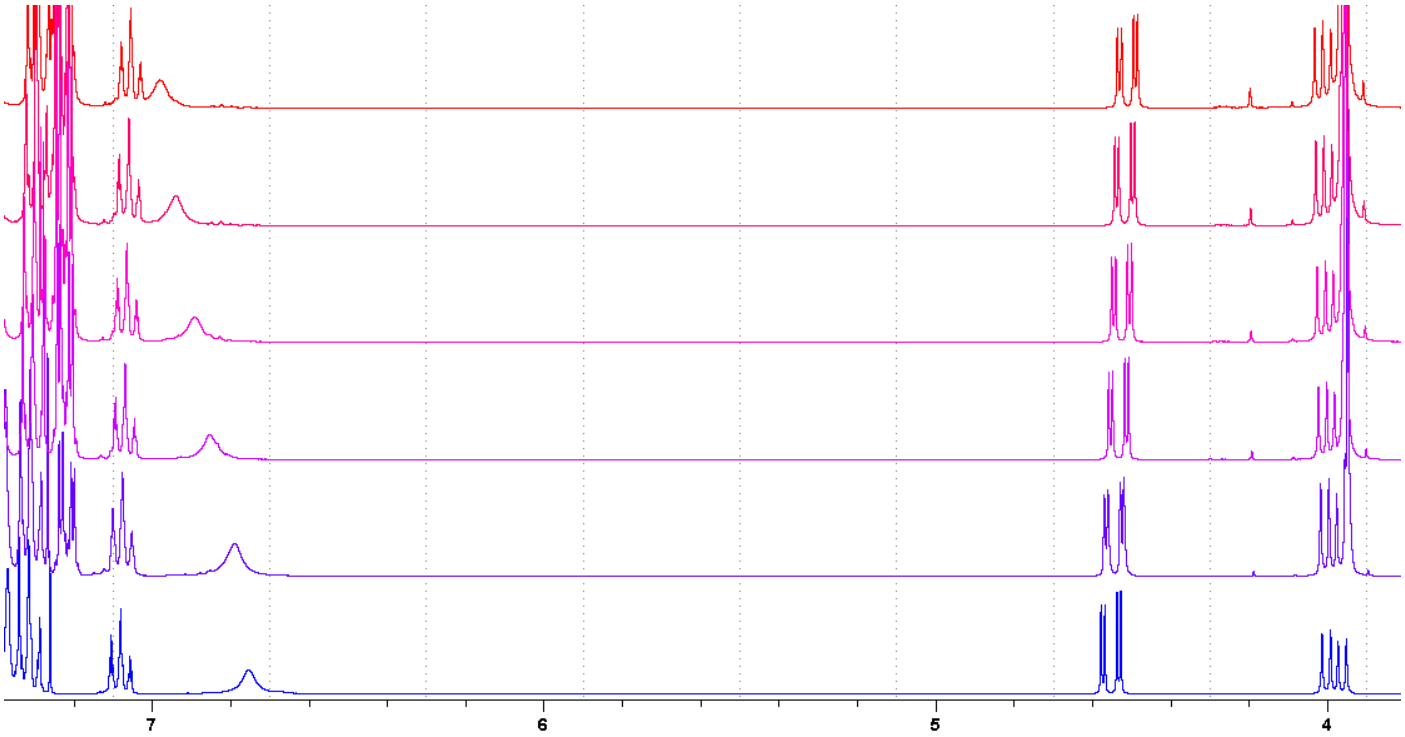
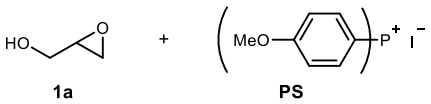


Figure S1. ^{1}H NMR spectra of a mixture of **3a** and **PS** recorded in $CDCl_3$.

Table S2. Δδ (OH) of **1a** for each molar ratio.



1a:**PS**	**1a**	Phosphonium Salt (**PS**)	$CDCl_3$	δ (OH)	Δδ
1:1	3.3 mg (45 μmol)	25.6 mg (45 μmol)	600 μL	2.10	0.38
1:0.8	3.3 mg (45 μmol)	20.5 mg (36 μmol)	600 μL	2.05	0.33
1:0.6	3.3 mg (45 μmol)	15.3 mg (27 μmol)	600 μL	1.97	0.25
1:0.4	3.3 mg (45 μmol)	10.2 mg (18 μmol)	600 μL	1.89	0.17
1:0.2	3.3 mg (45 μmol)	5.1 mg (9 μmol)	600 μL	1.83	0.11
1:0	3.3 mg (45 μmol)	-	600 μL	1.72	-

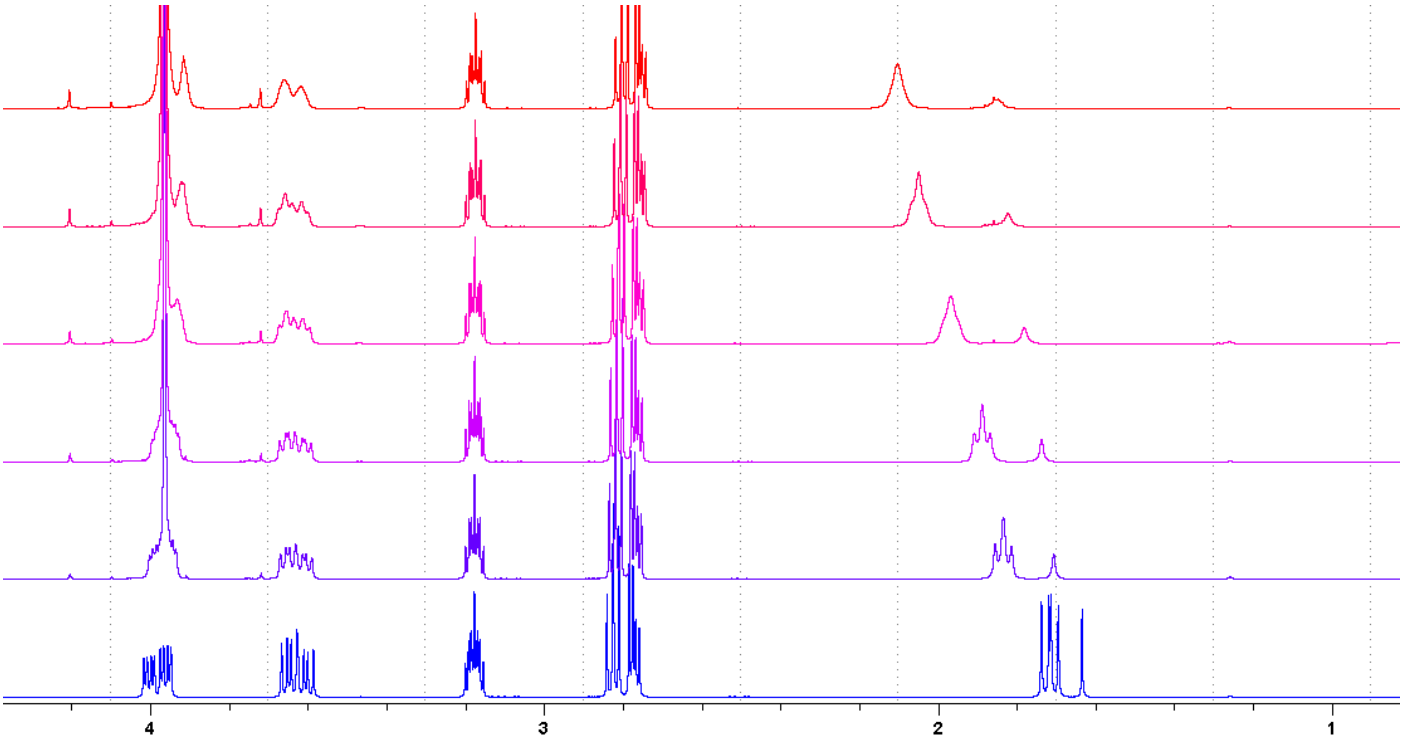
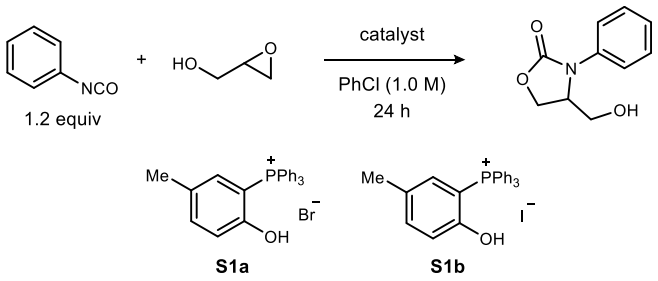


Figure S2. ^{1}H NMR spectra of a mixture of **1a** and **PS** recorded in $CDCl_3$.

Table S3. Optimization of reaction conditions[a]



Catalyst (mol %)	Temp. (°C)	Yield (%)[b]
S1a (2)[c]	80	52
S1b (2)[c]	80	67
Ph_4PI (2)	80	86
Ph_4PI (2)	60	76
Ph_4PI (2)	35	32
Ph_4PI (5)	35	47
Ph_4PI (10)	35	60

[a]All reactions were performed out on a 0.60 mmol scale. [b]Determined by ^1H NMR analysis. [c]See ref 5 for details.

References

(1) Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518-1520.
(2) Pettersson, H.; Gogoll, A.; Bäckvall, J.-E. *J. Org. Chem.* **1992**, *57*, 6025-6031.
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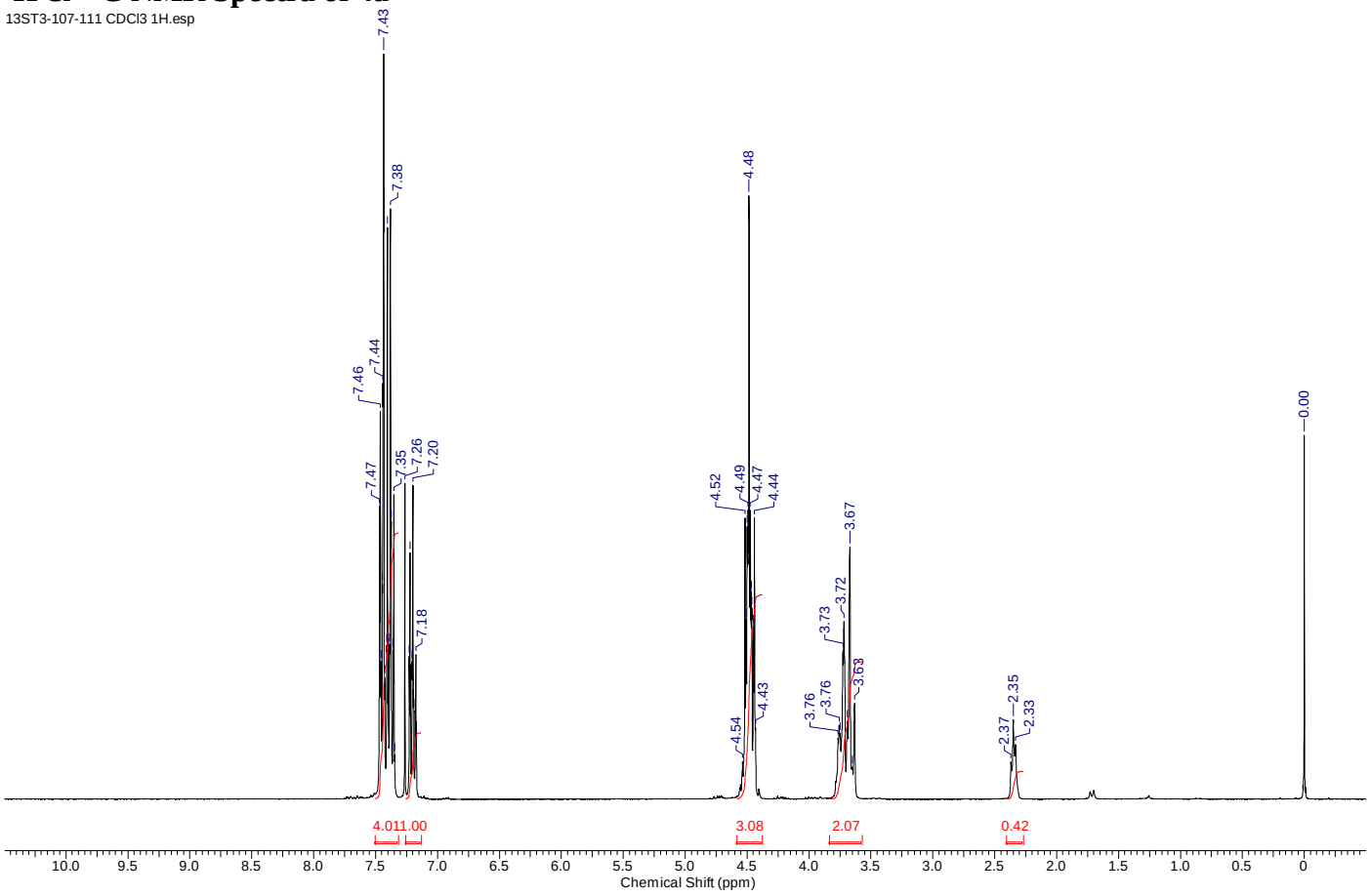
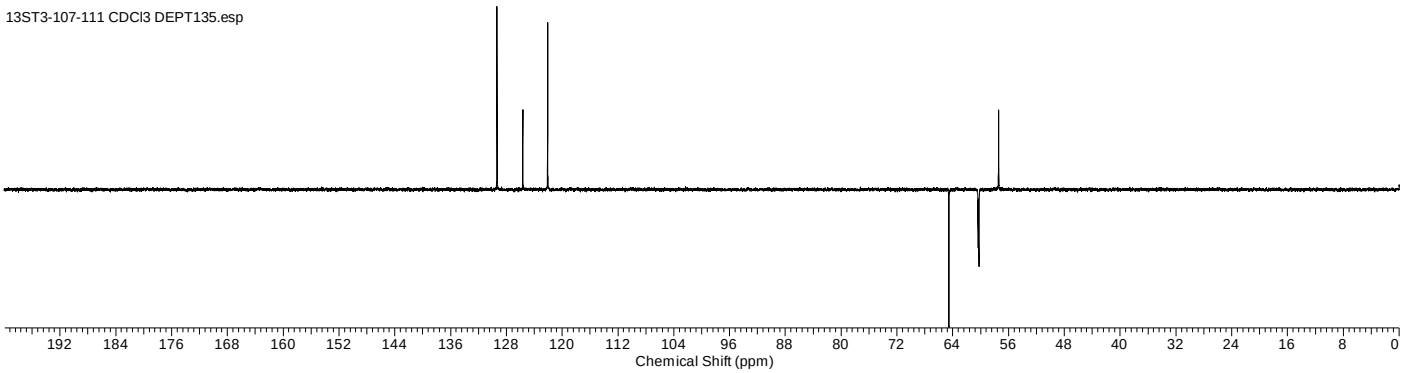
(5) (a) Toda, Y.; Komiyama, Y.; Kikuchi, A.; Suga, H. *ACS Catal.* **2016**, *6*, 6906-6910. (b) Toda, Y.; Sakamoto, T.; Komiyama, Y.; Kikuchi, A.; Suga, H. *ACS Catal.* **2017**, *7*, 6150-6154.

^{1}H & ^{13}C NMR Spectra of 4a

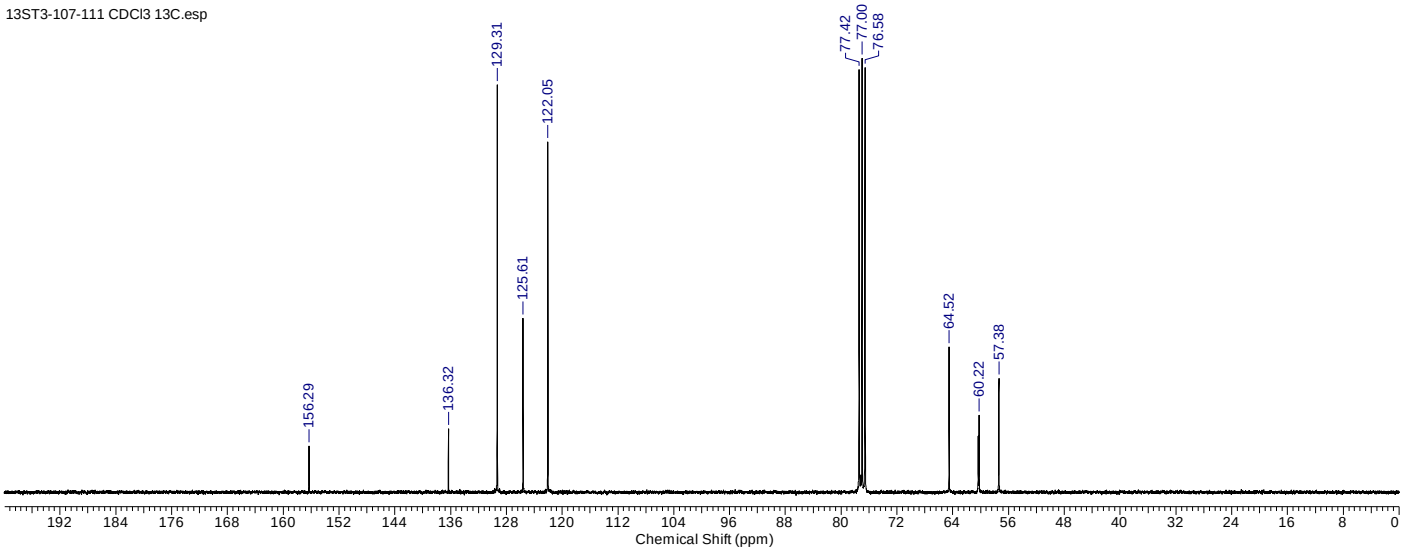
13ST3-107-111 CDCl3 1H.esp



13ST3-107-111 CDCl3 DEPT135.esp

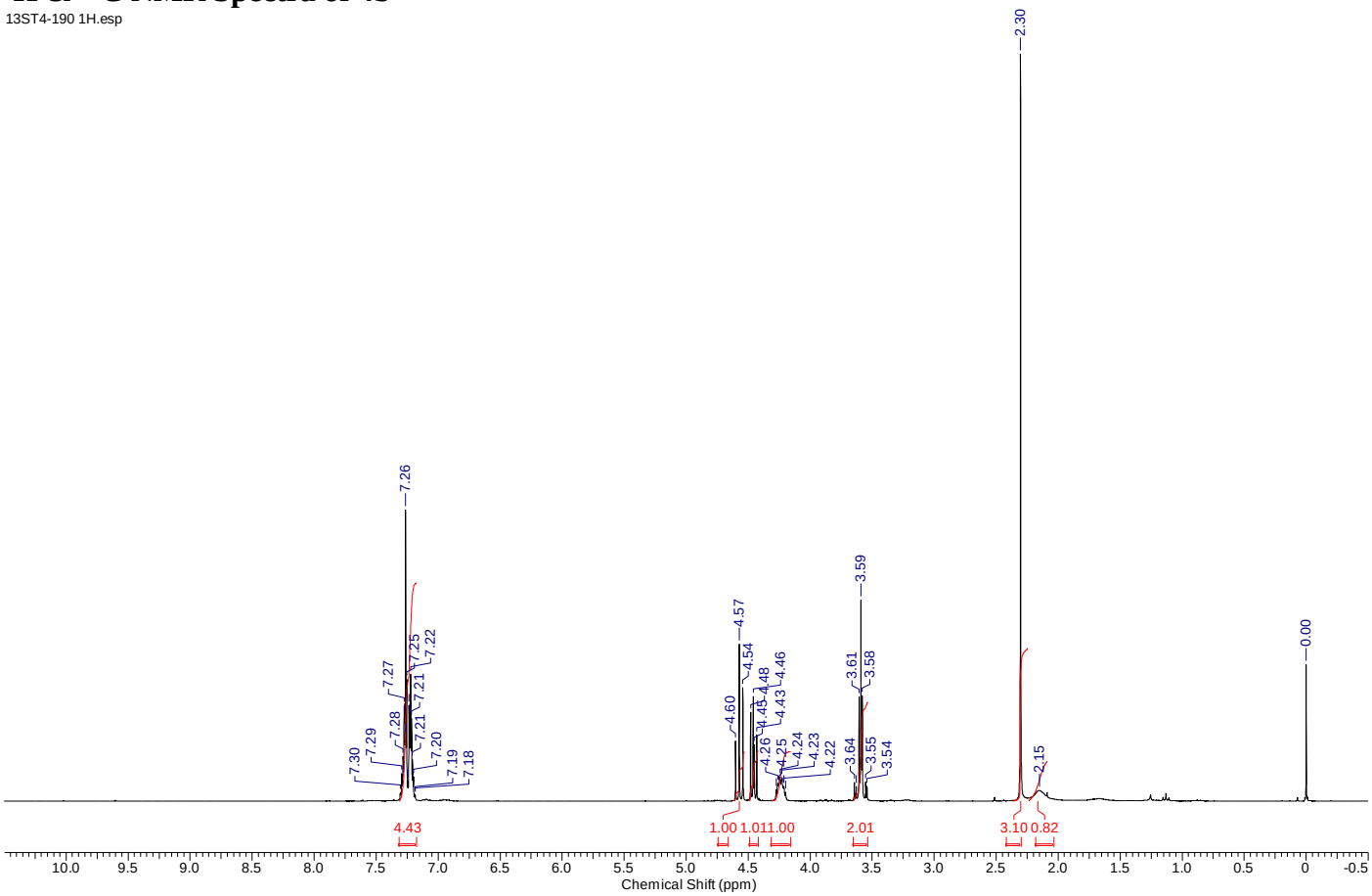


13ST3-107-111 CDCl3 13C.esp

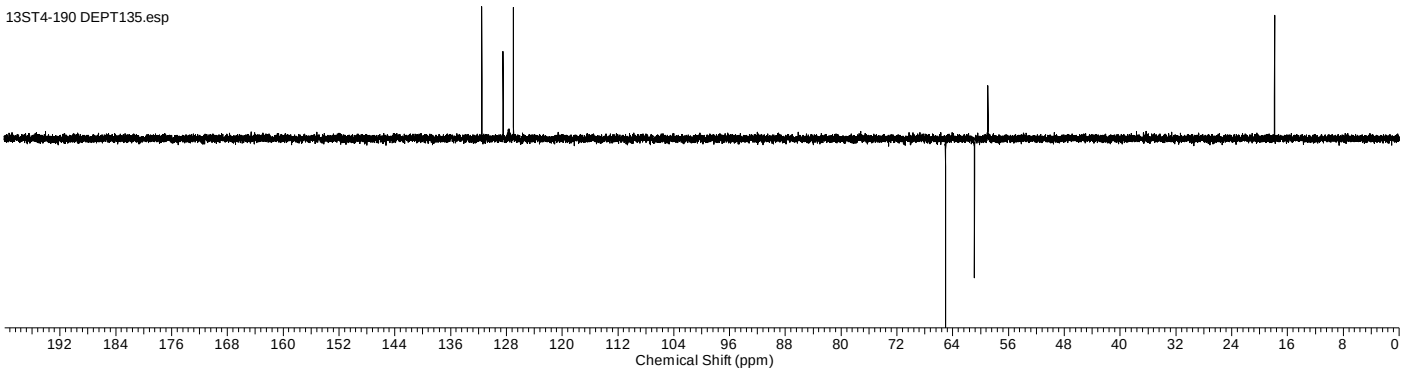


^{1}H & ^{13}C NMR Spectra of 4b

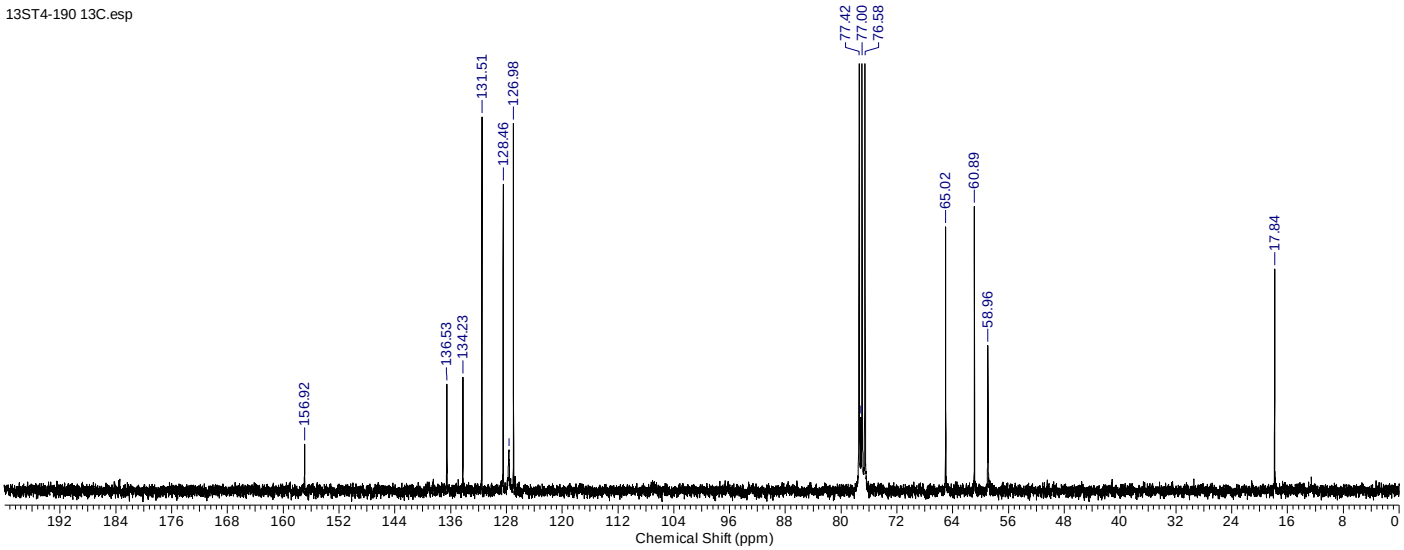
13ST4-190 1H.esp



13ST4-190 DEPT135.esp



13ST4-190 13C.esp



^{1}H & ^{13}C NMR Spectra of 4c

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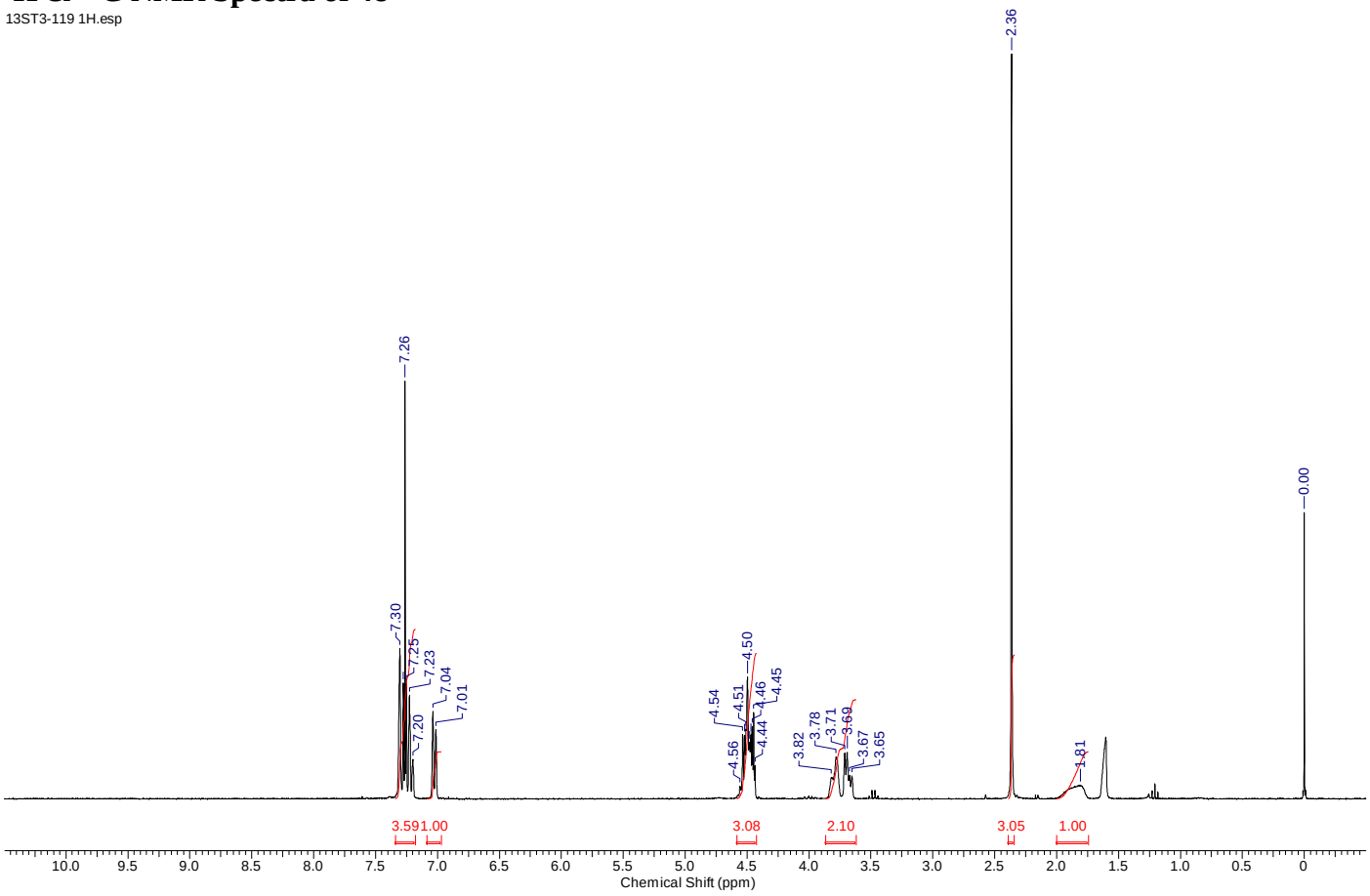
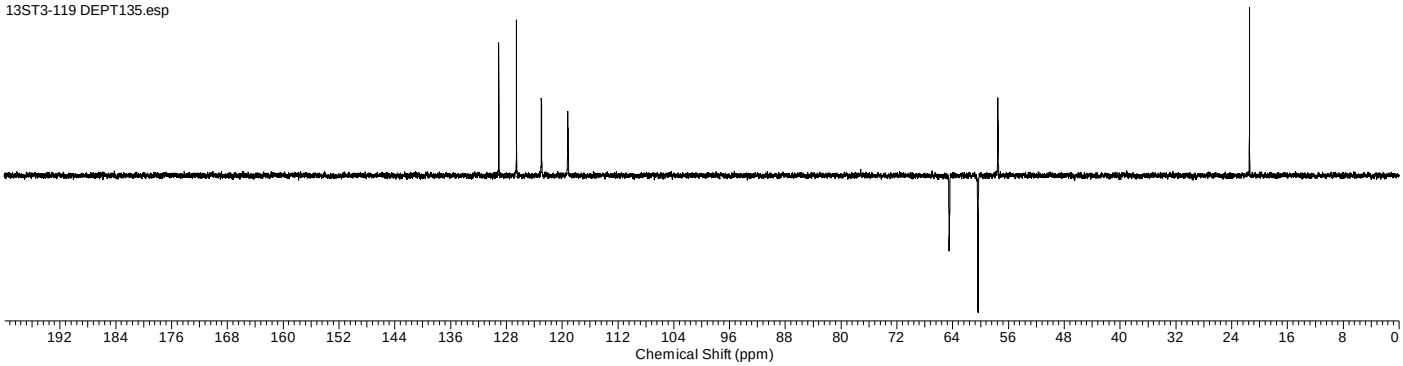
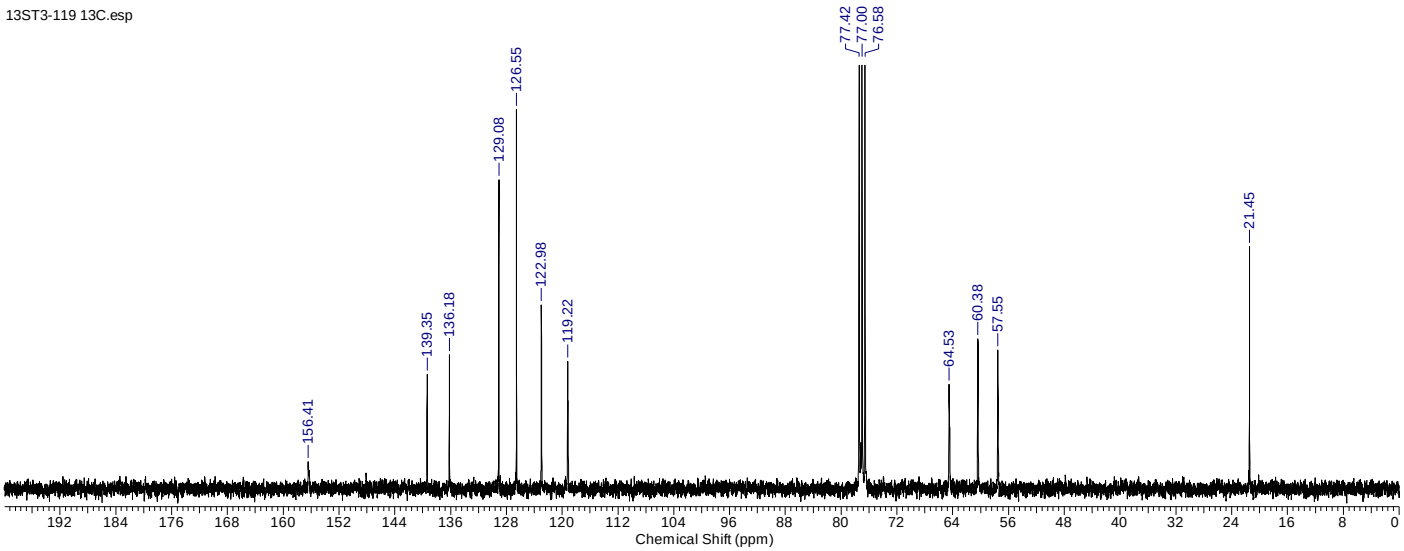


13ST3-119 DEPT135.esp

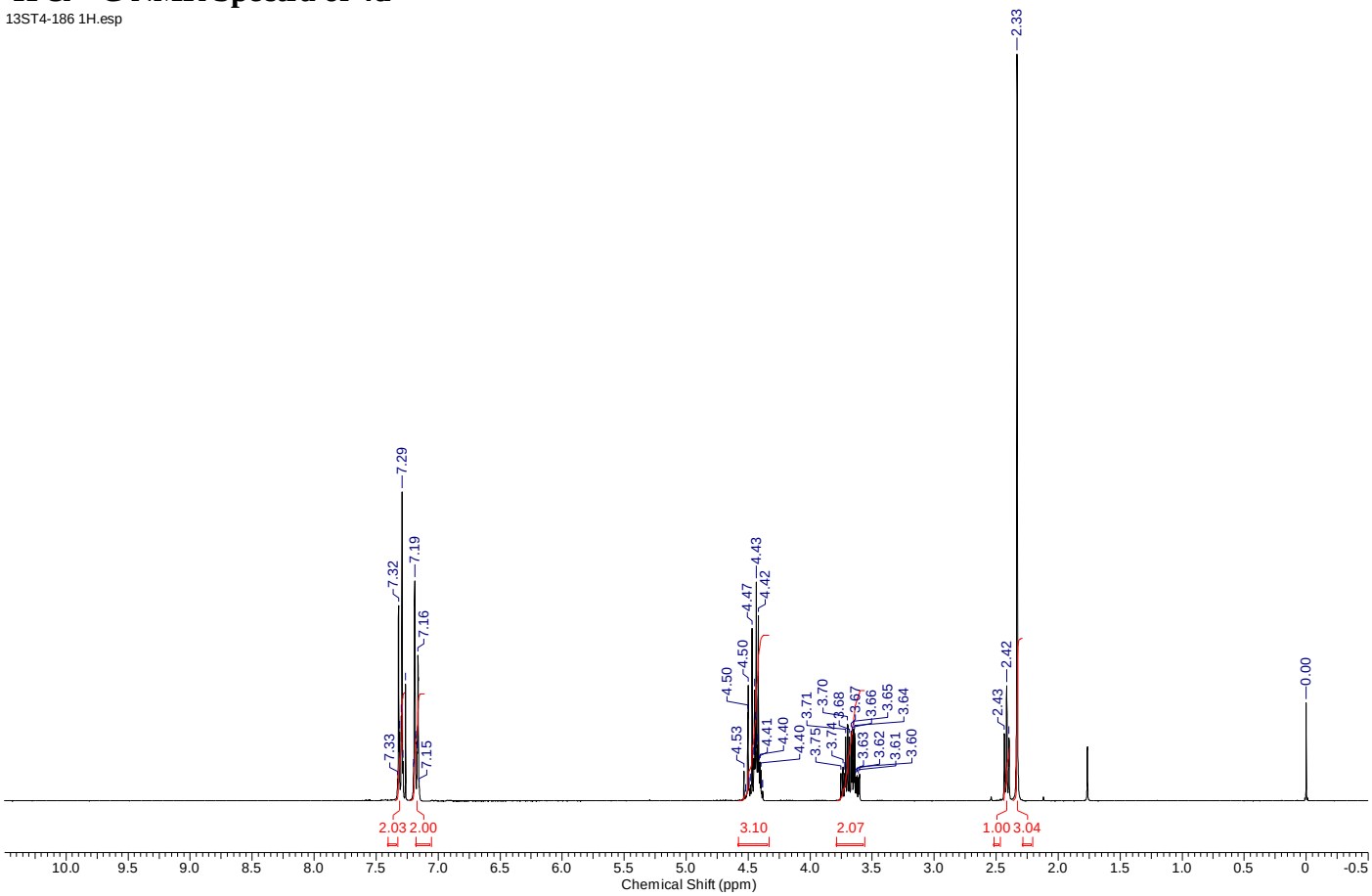


13ST3-119 13C.esp

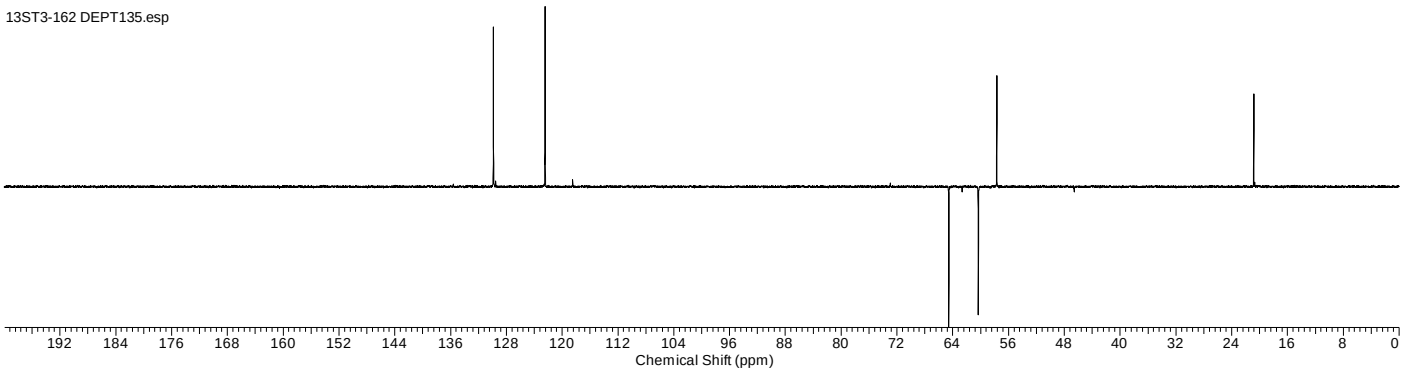


^{1}H & ^{13}C NMR Spectra of 4d

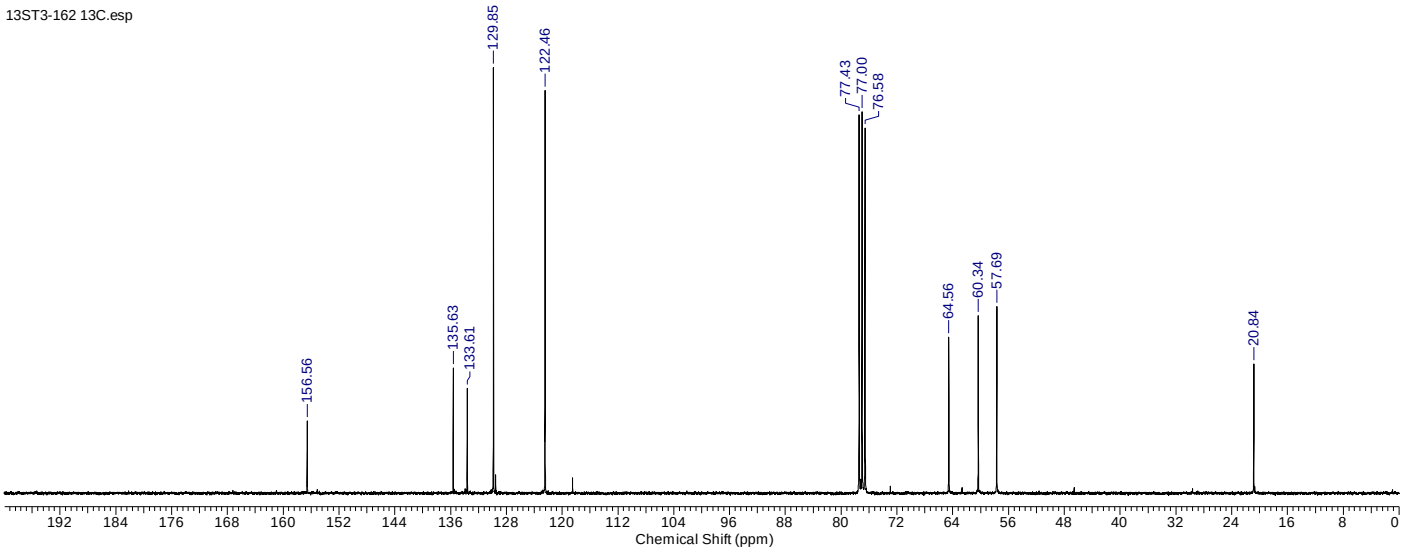
13ST4-186 1H.esp



13ST3-162 DEPT135.esp

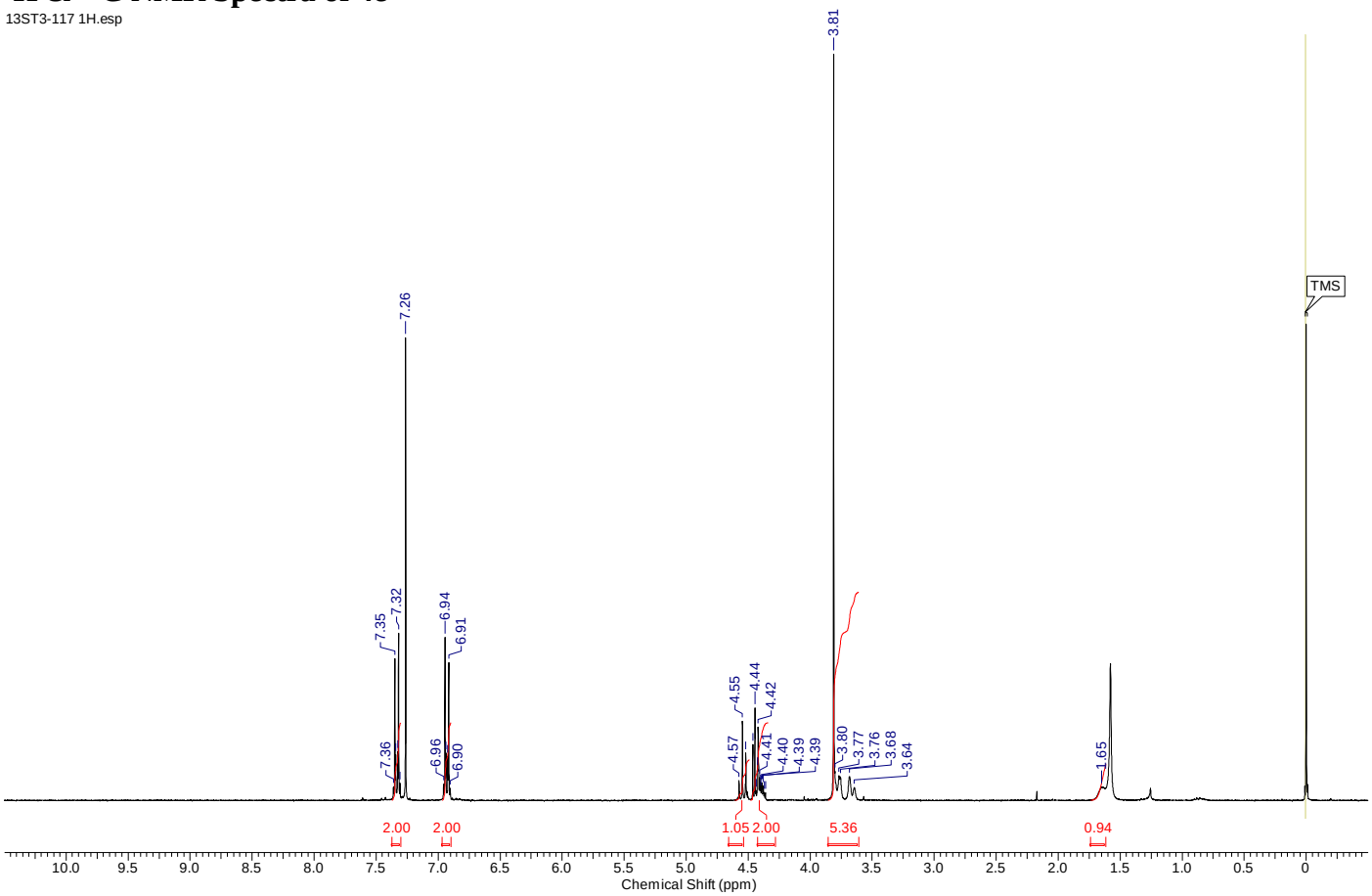


13ST3-162 13C.esp

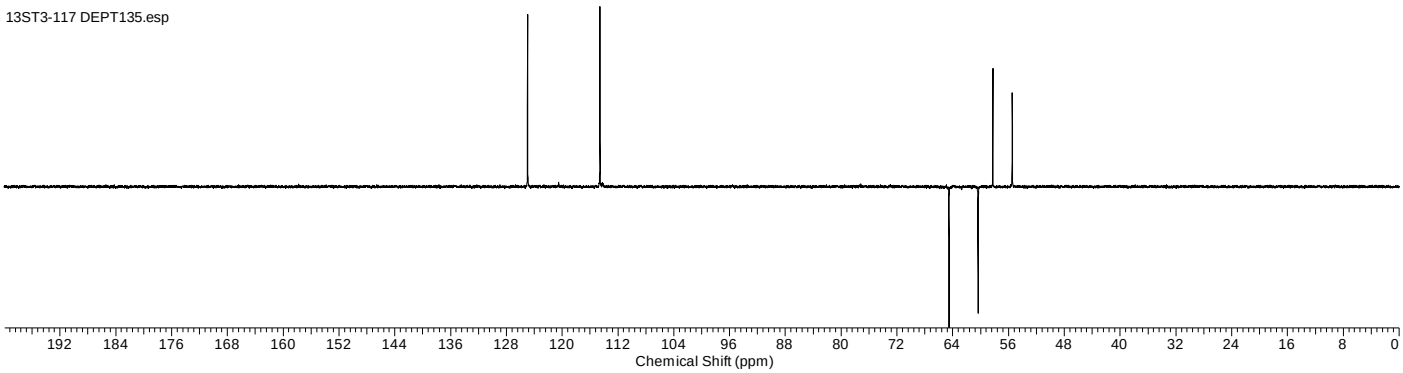


^{1}H & ^{13}C NMR Spectra of 4e

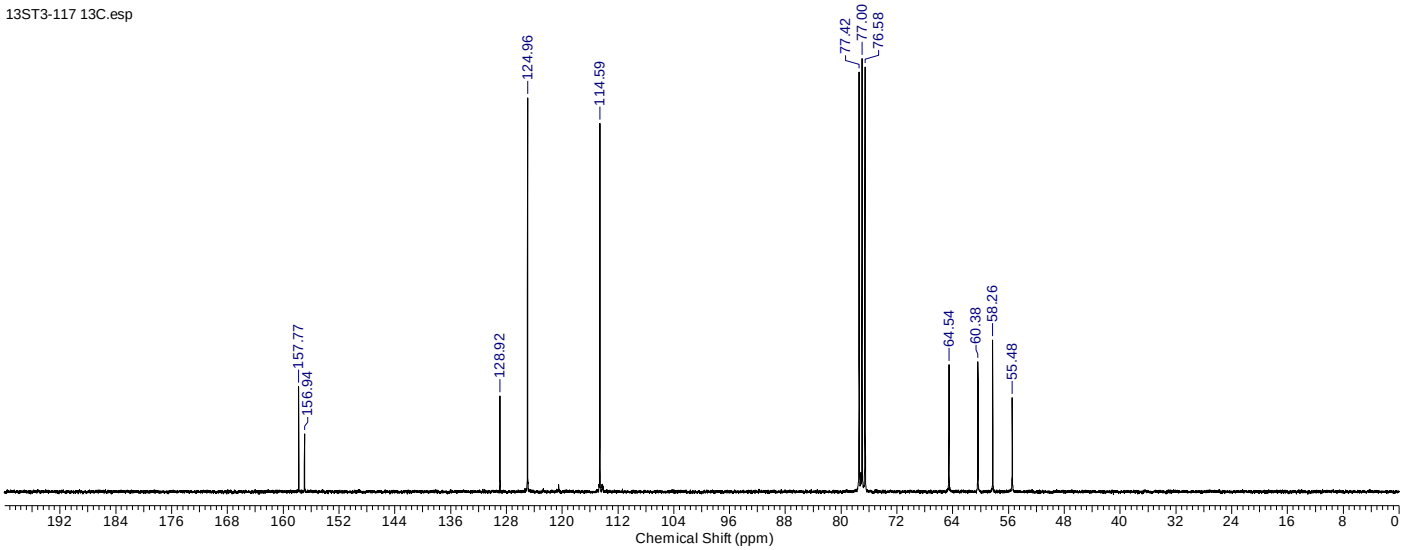
13ST3-117 1H.esp



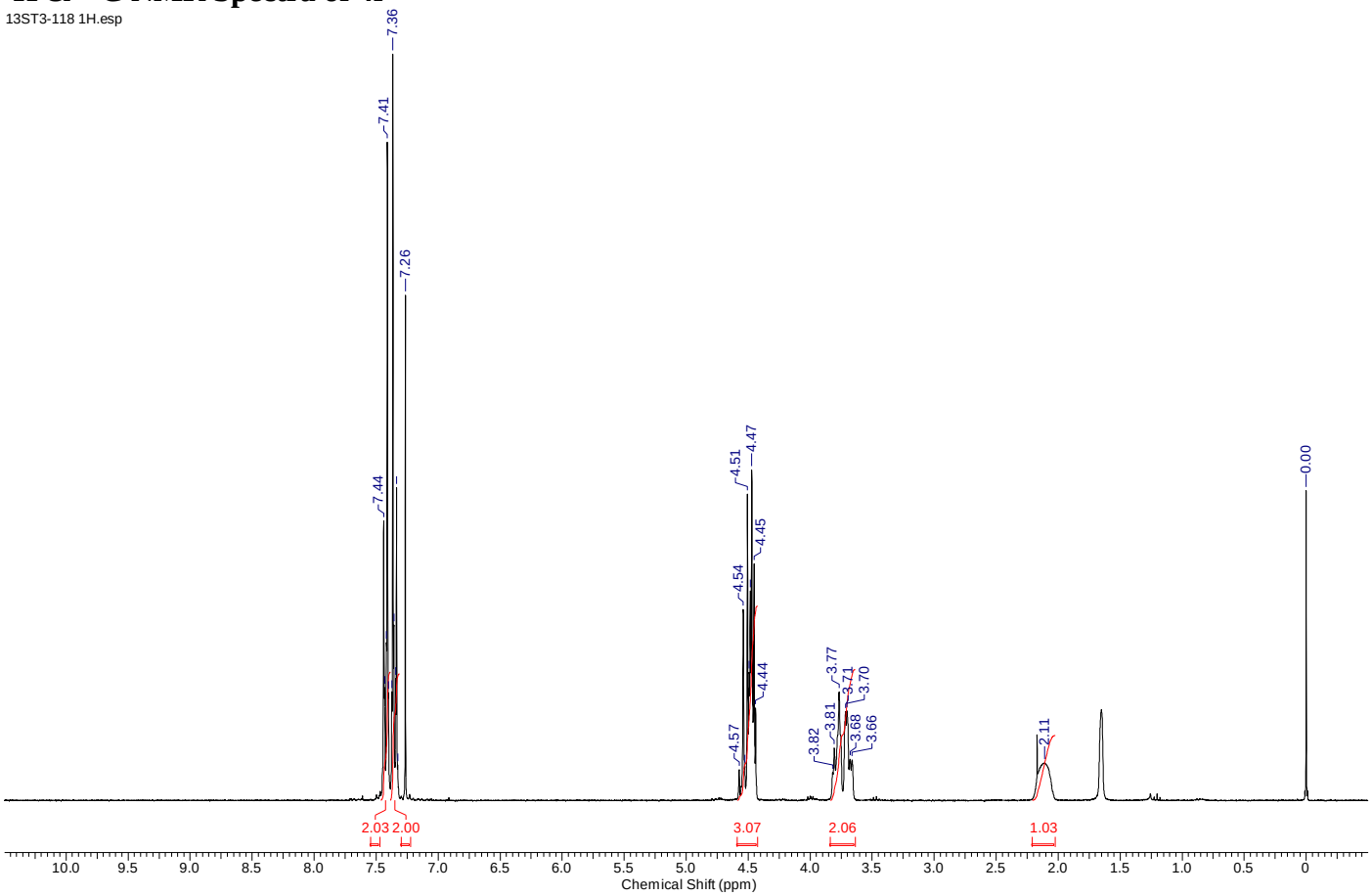
13ST3-117 DEPT135.esp

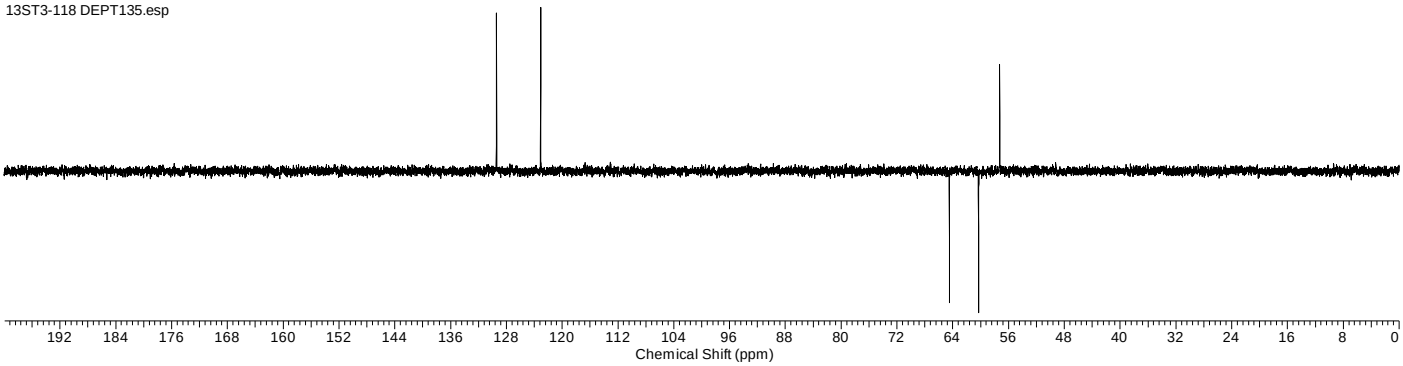


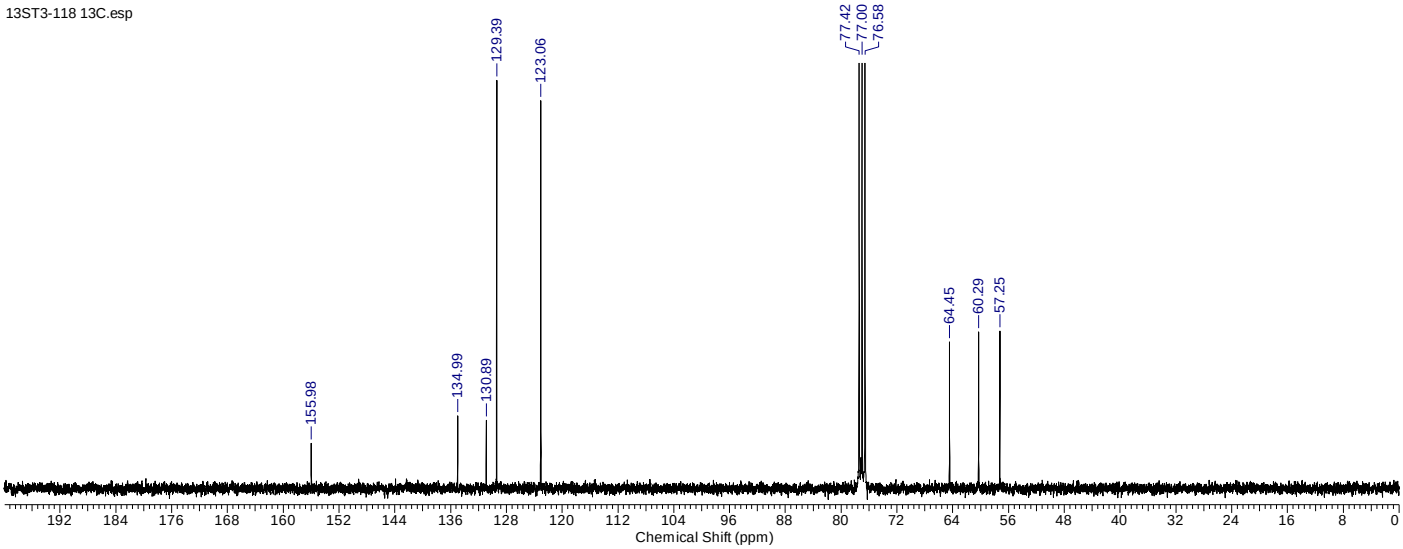
13ST3-117 13C.esp



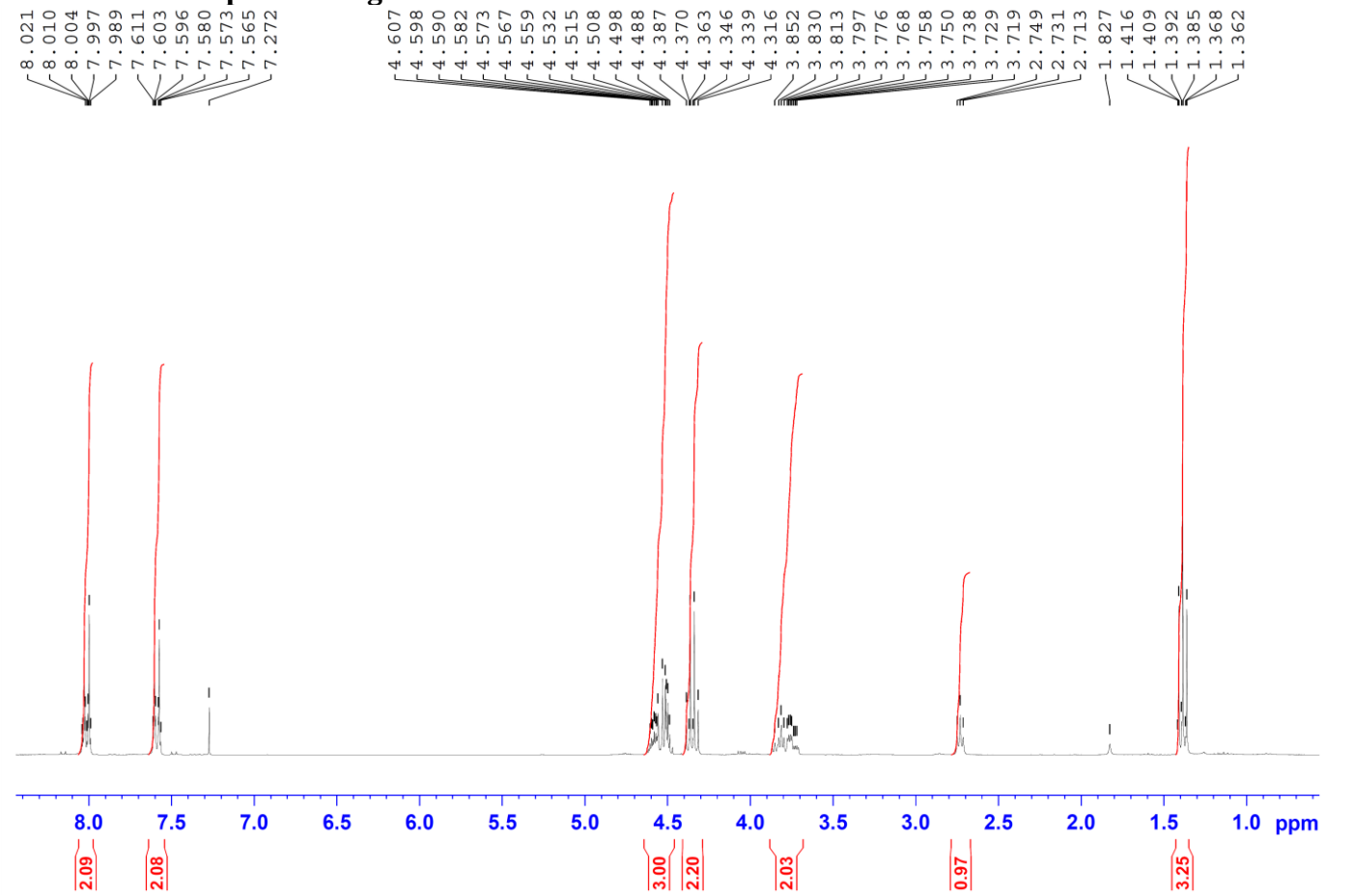
^{1}H & ^{13}C NMR Spectra of 4f

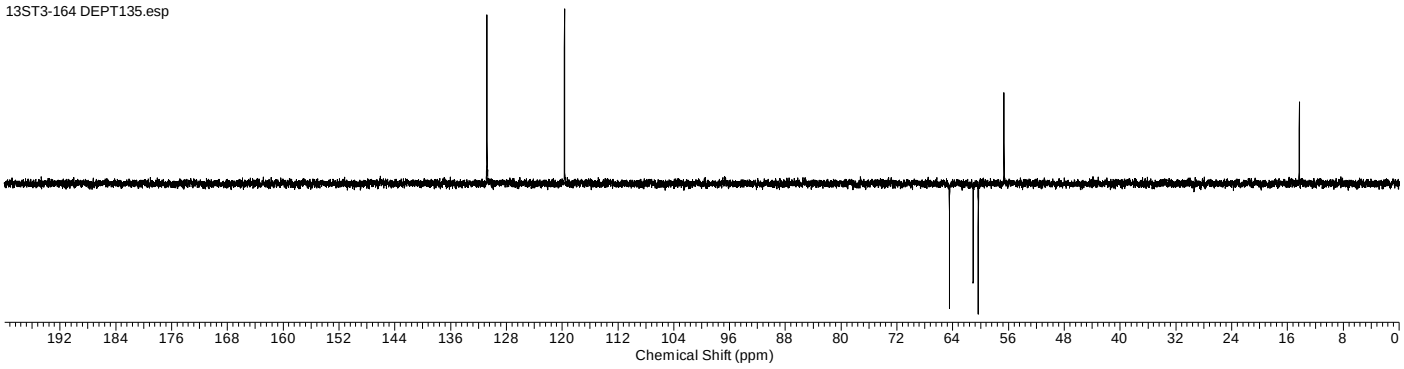


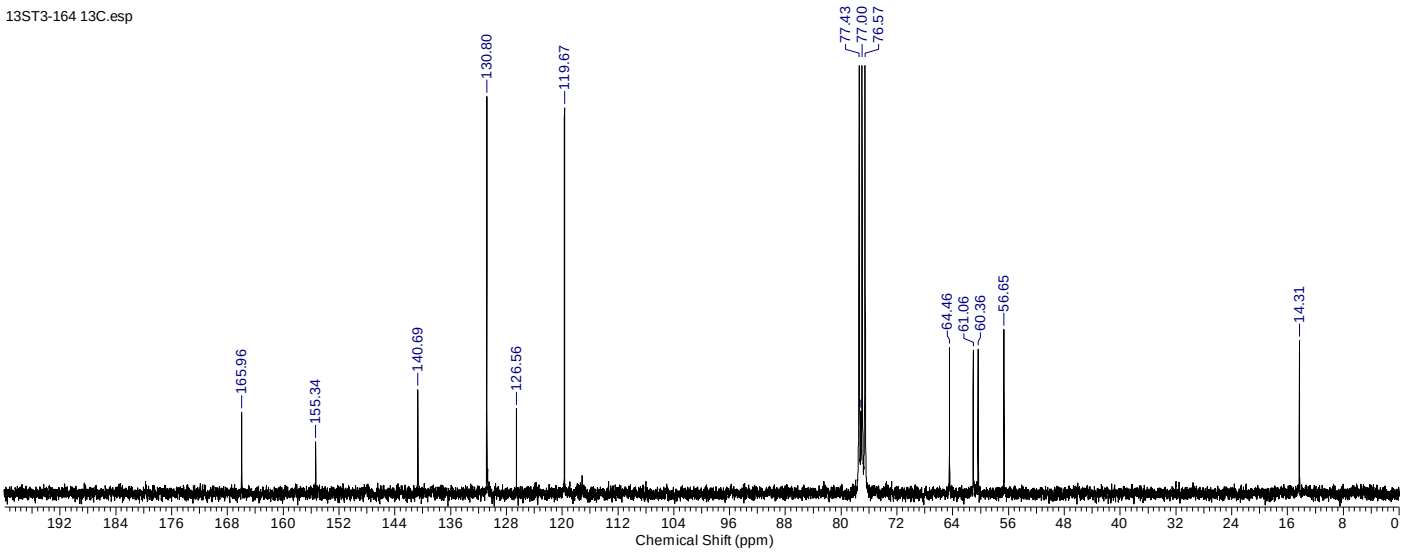




^{1}H & ^{13}C NMR Spectra of 4g

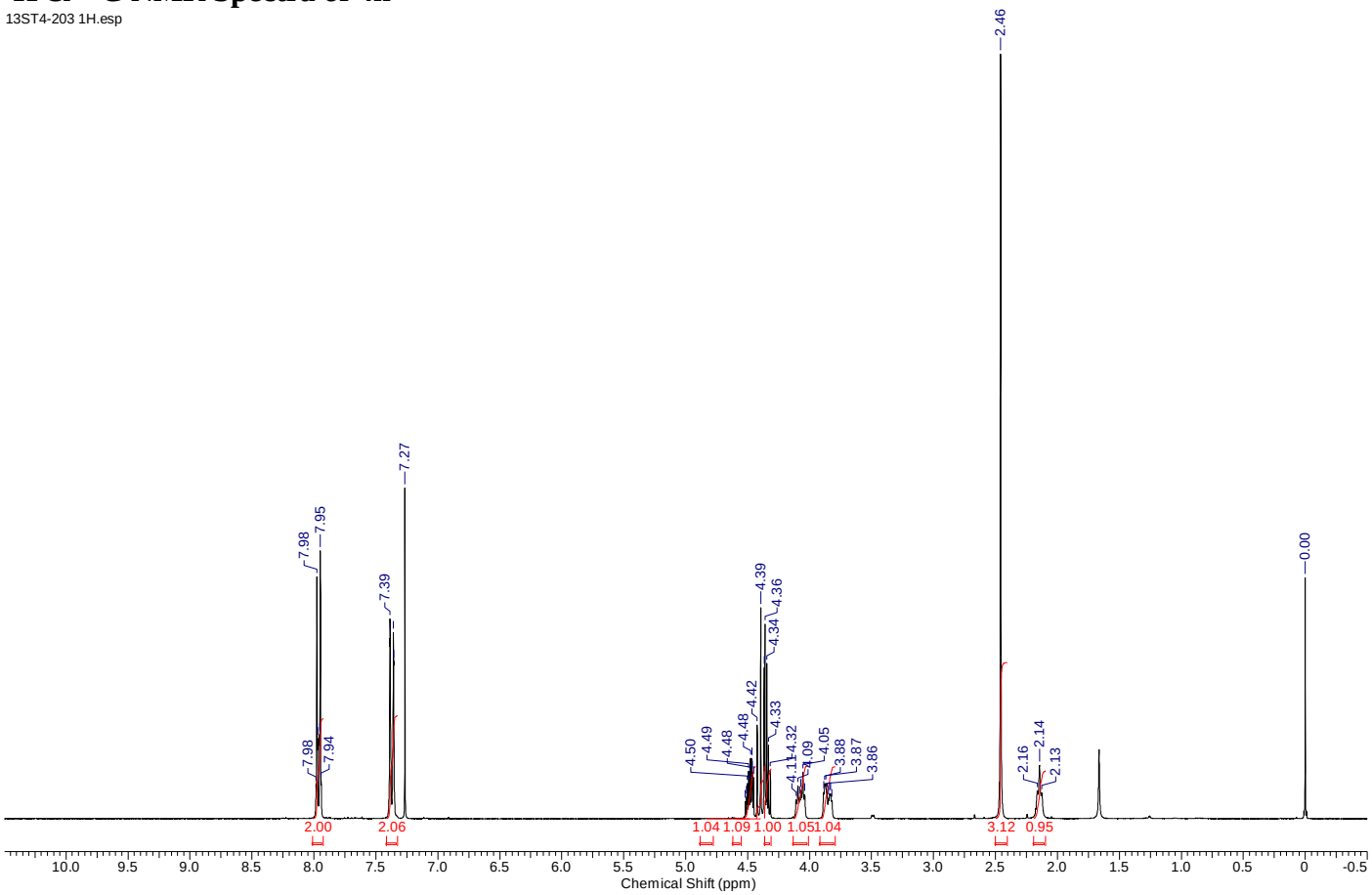




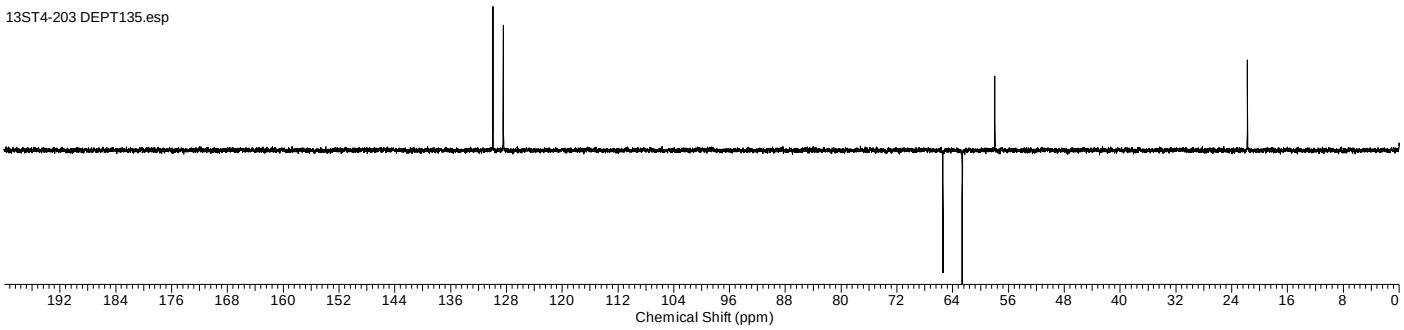


^{1}H & ^{13}C NMR Spectra of 4h

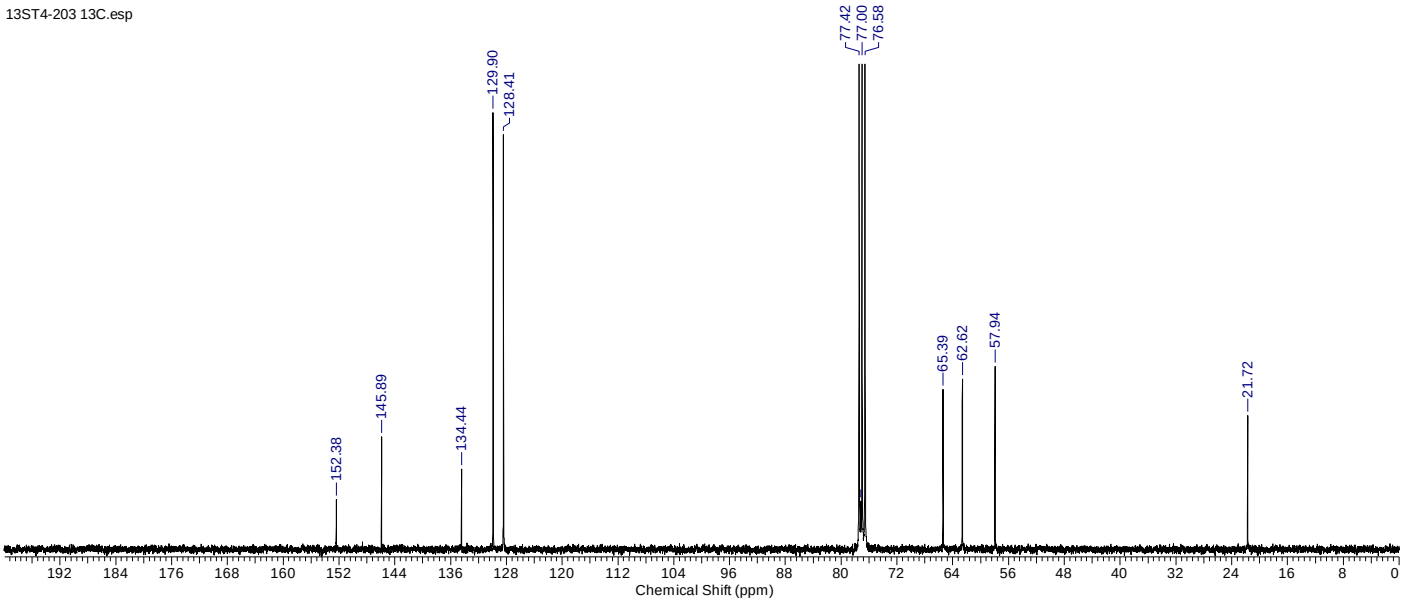
13ST4-203 1H.esp



13ST4-203 DEPT135.esp



13ST4-203 13C.esp



^{1}H & ^{13}C NMR Spectra of 4i

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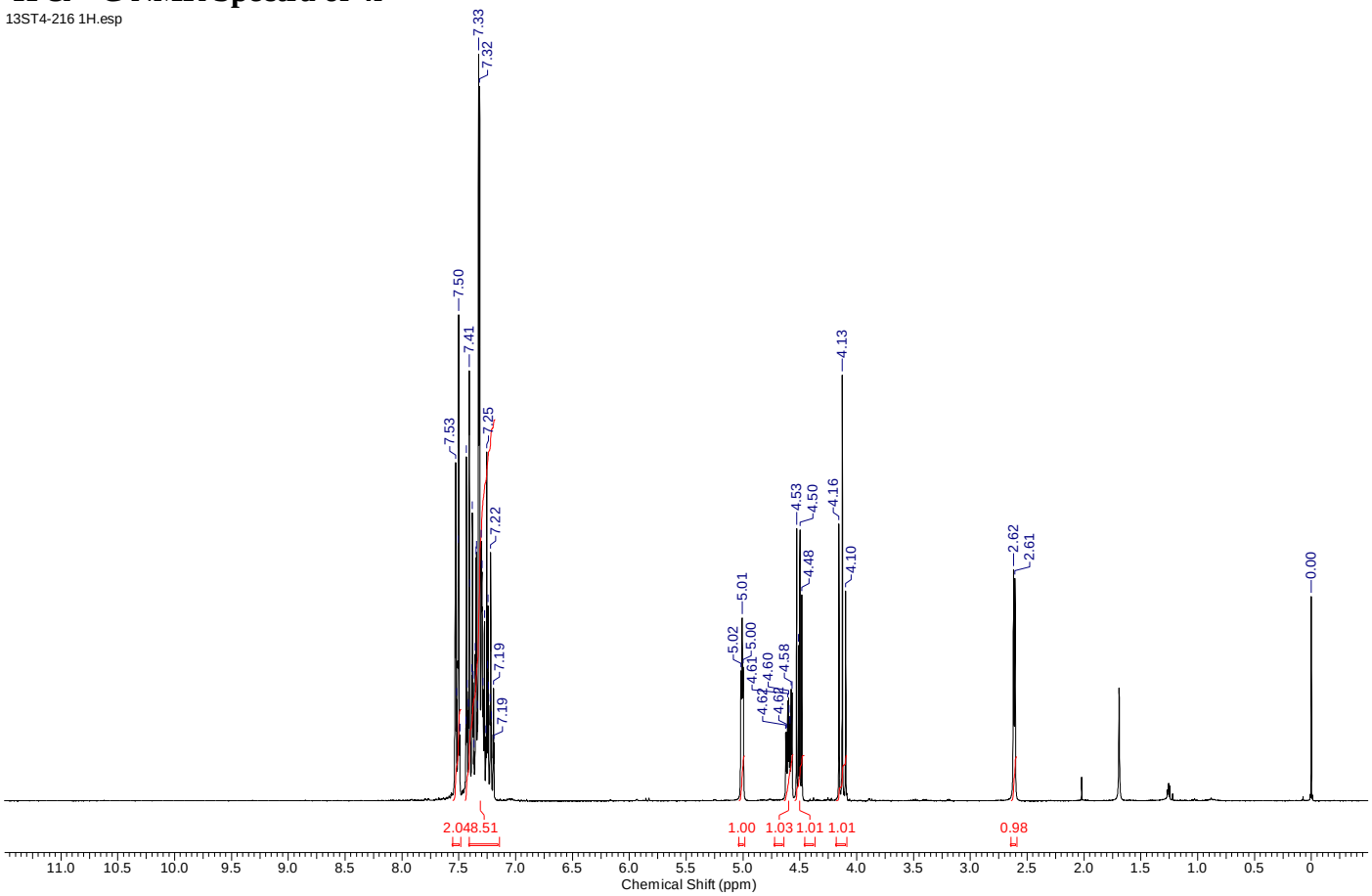

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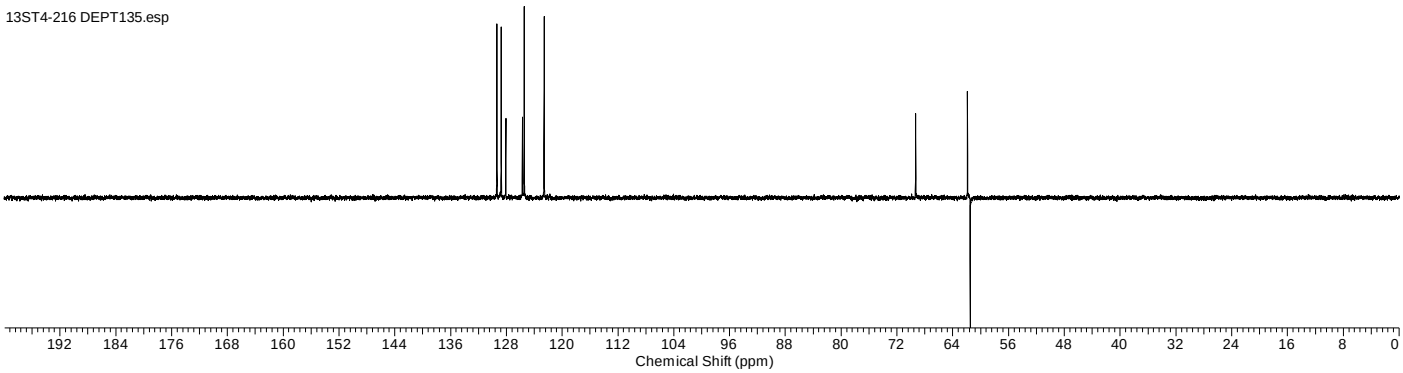

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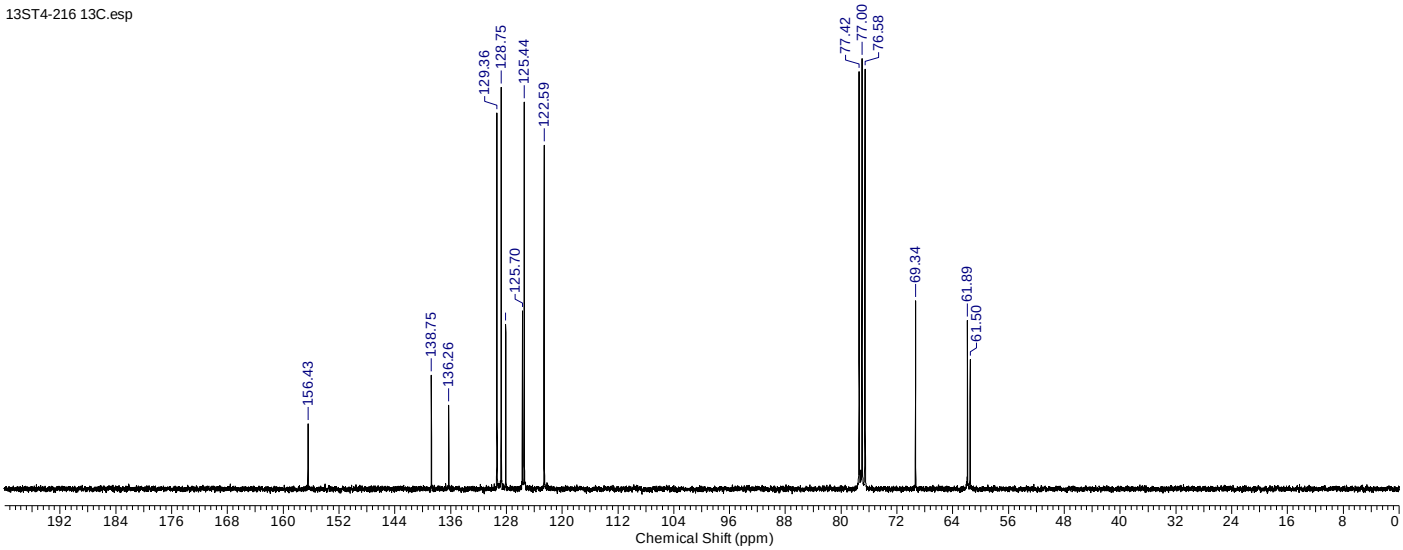
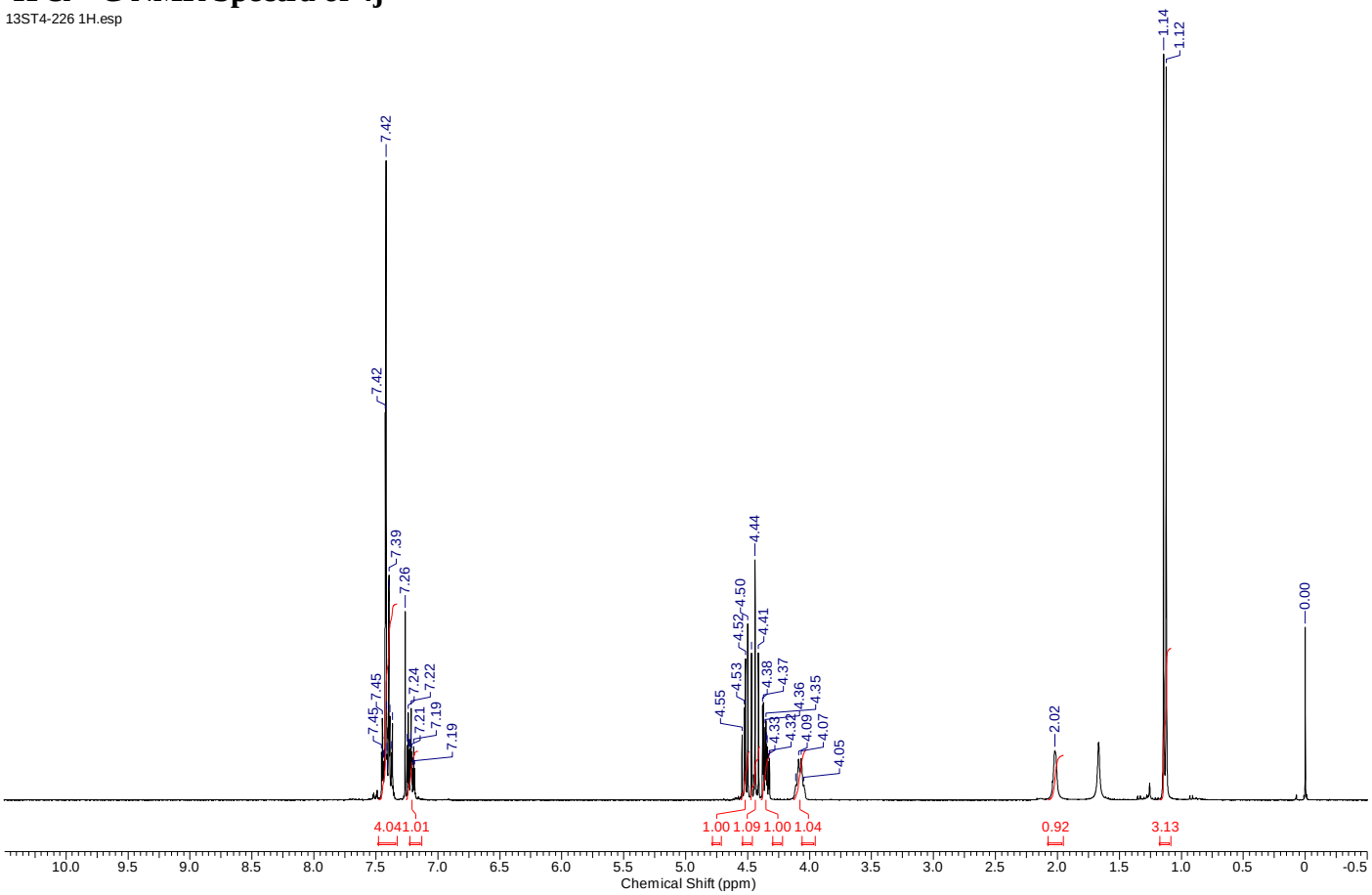


^{1}H & ^{13}C NMR Spectra of 4j

13ST4-226 1H.esp



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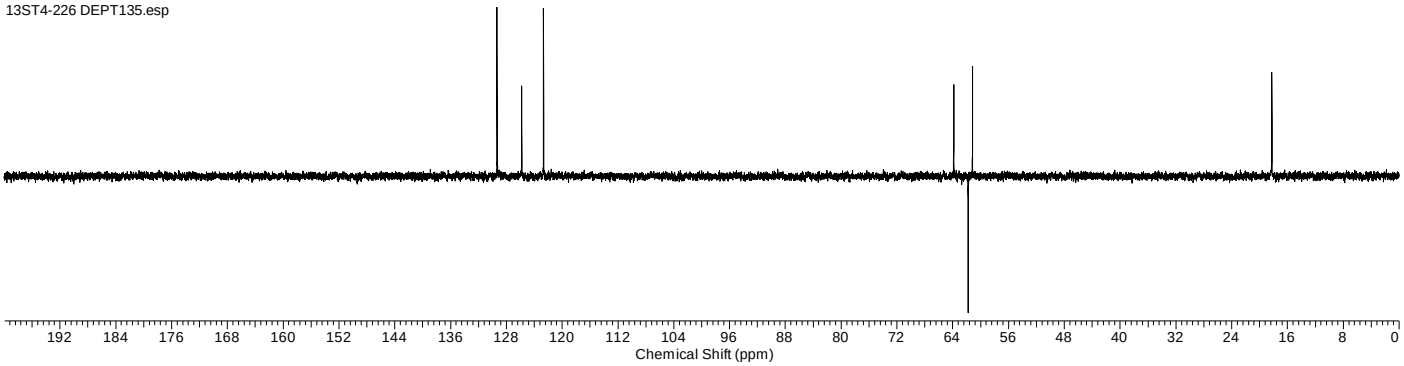
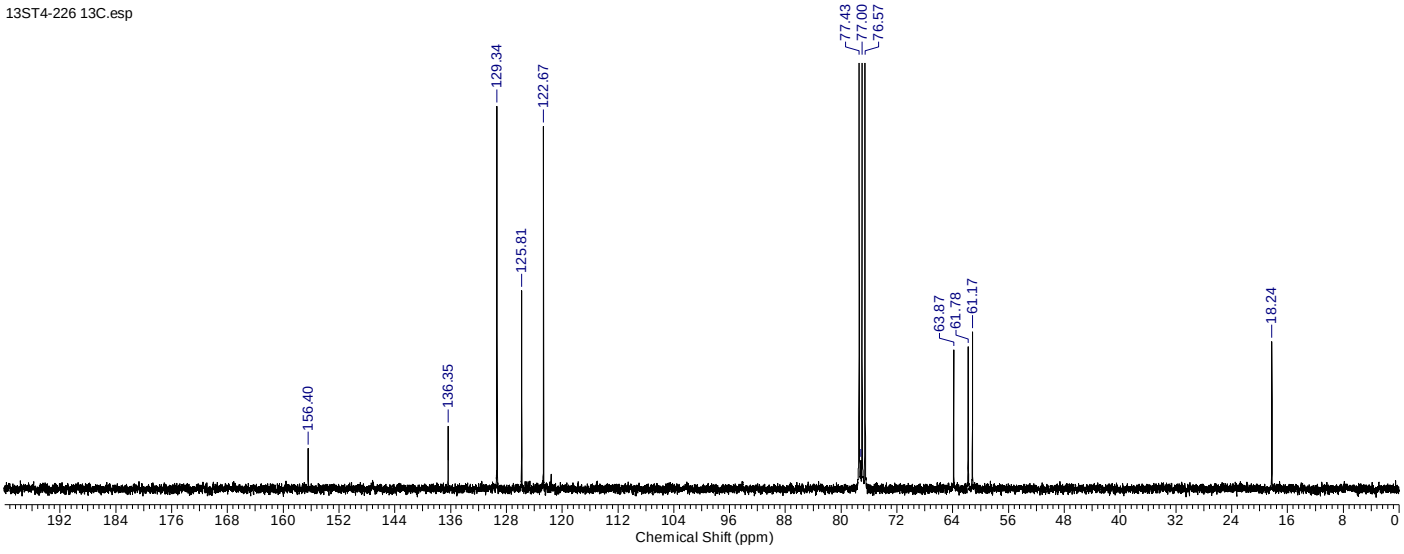


13ST4-226 13C.esp



^{1}H & ^{13}C NMR Spectra of 4k

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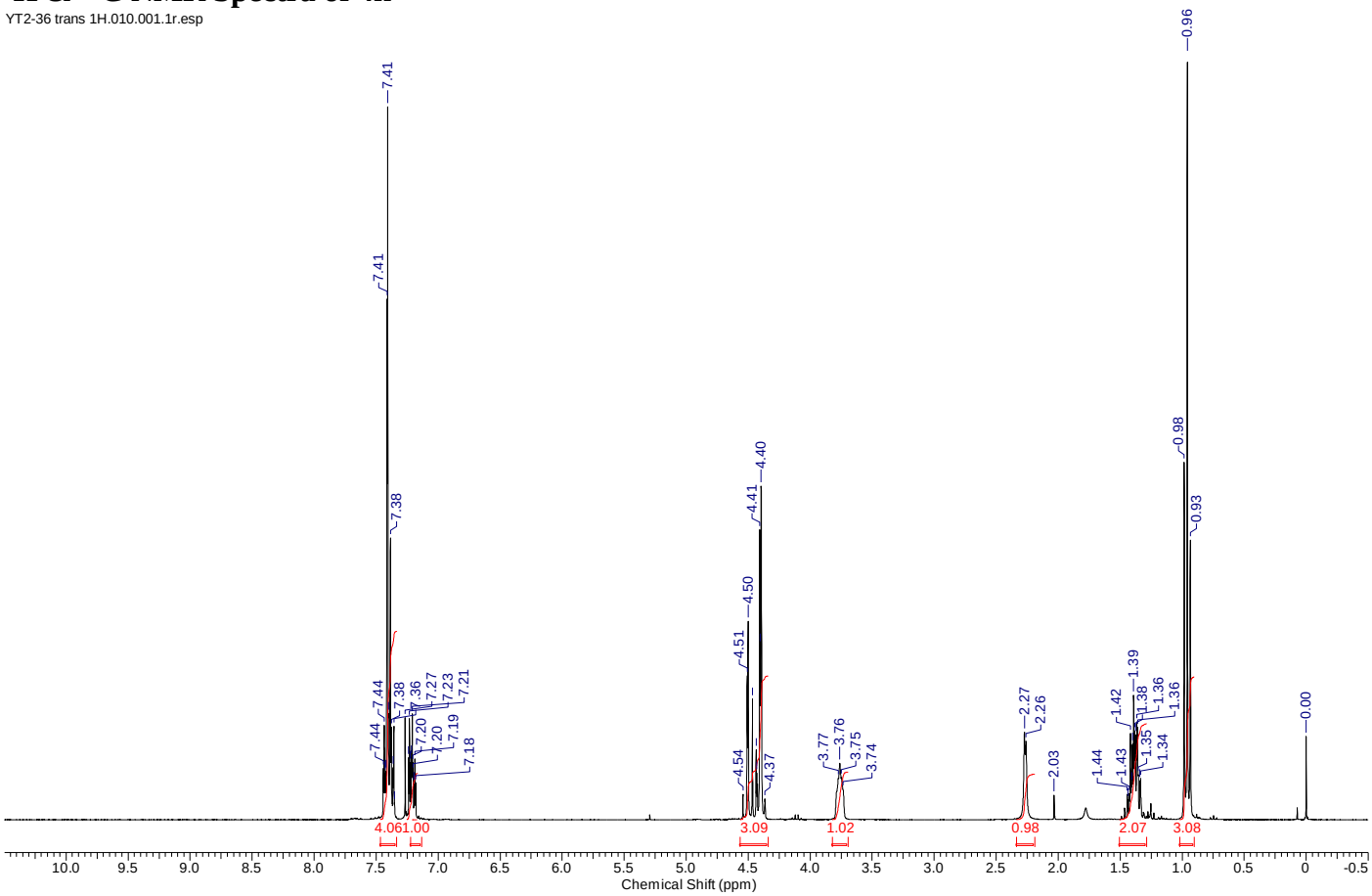

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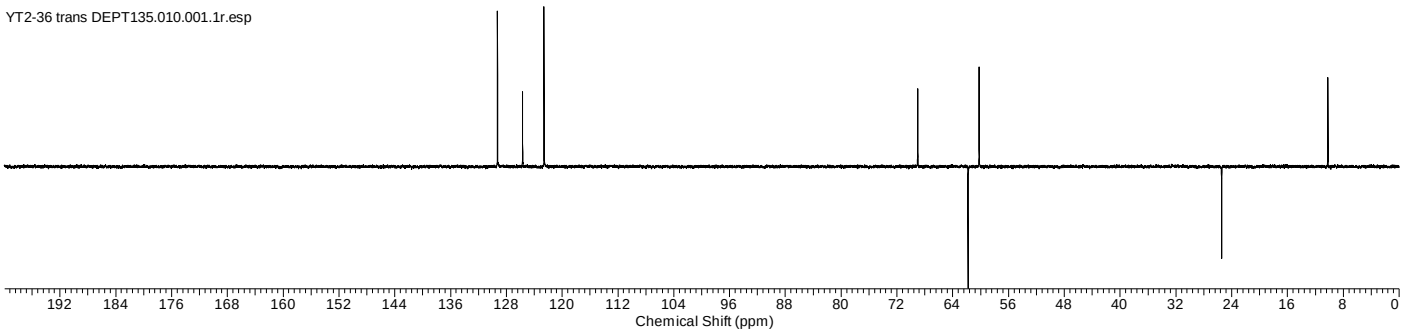
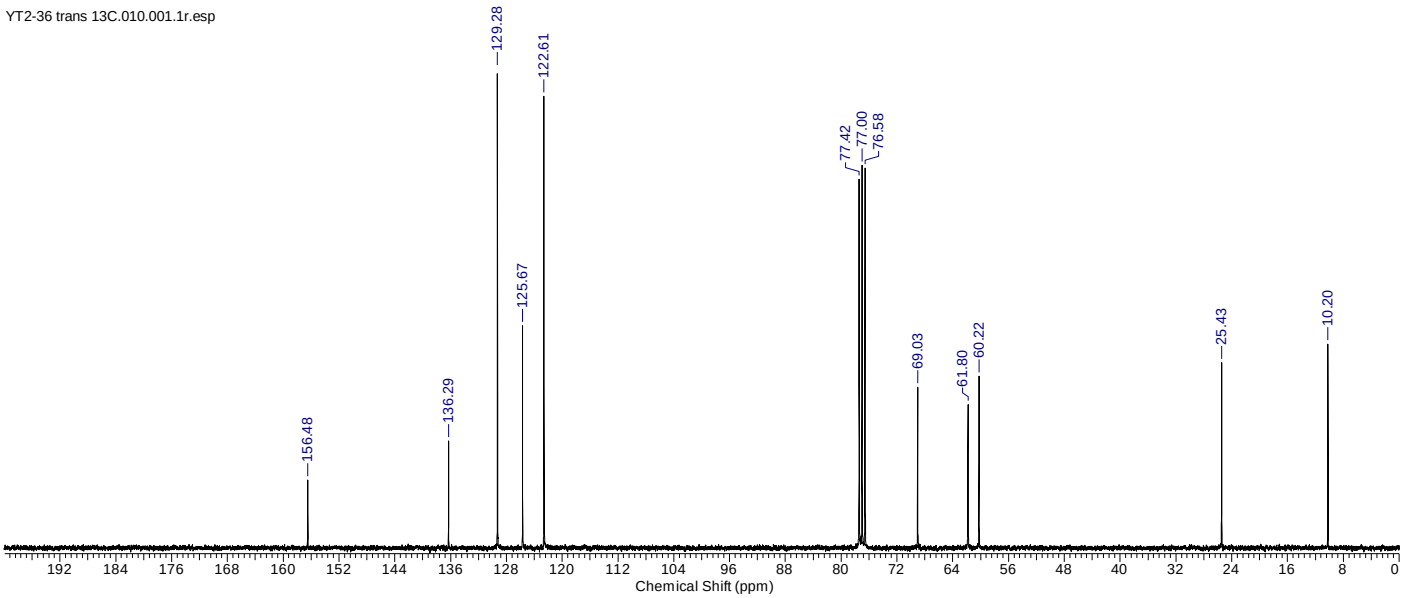


YT2-36 trans 13C.010.001.1r.esp



^{1}H & ^{13}C NMR Spectra of 4l

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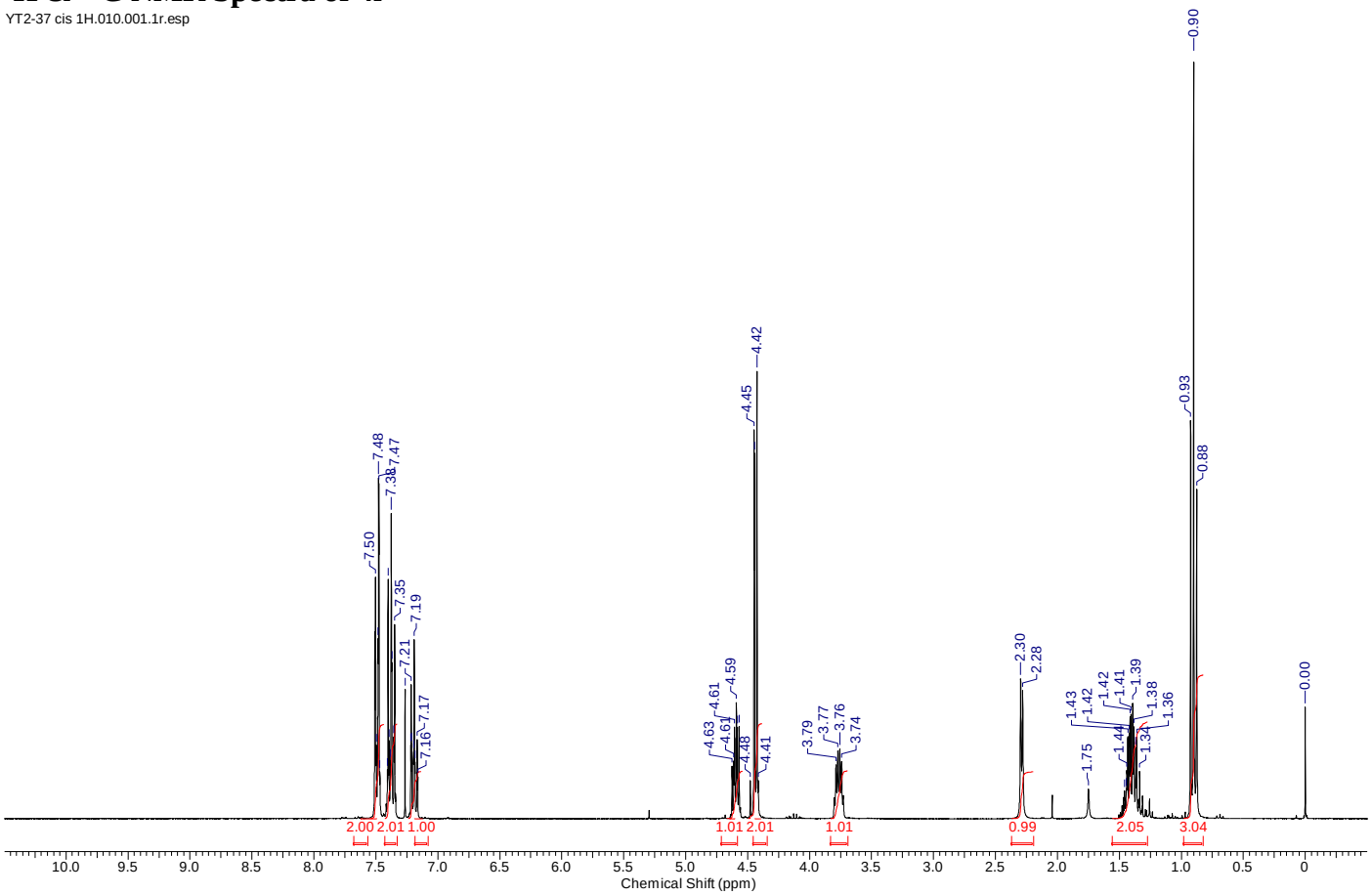

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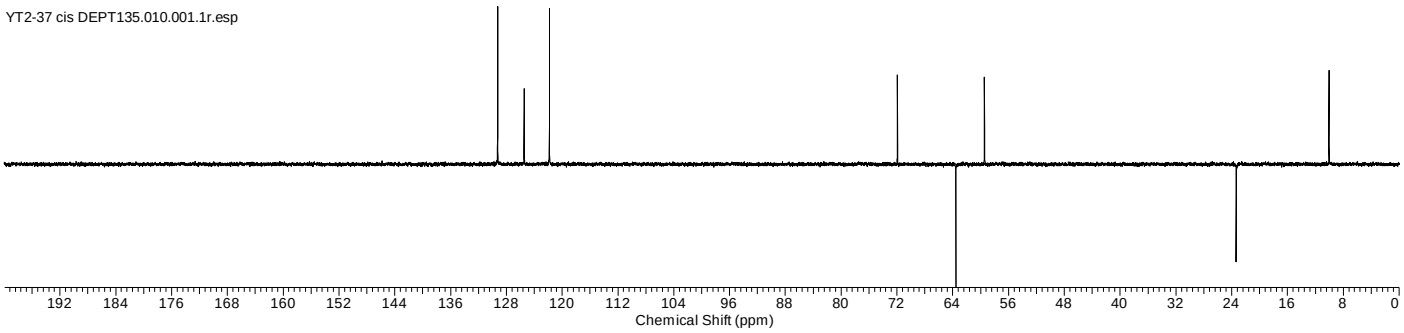

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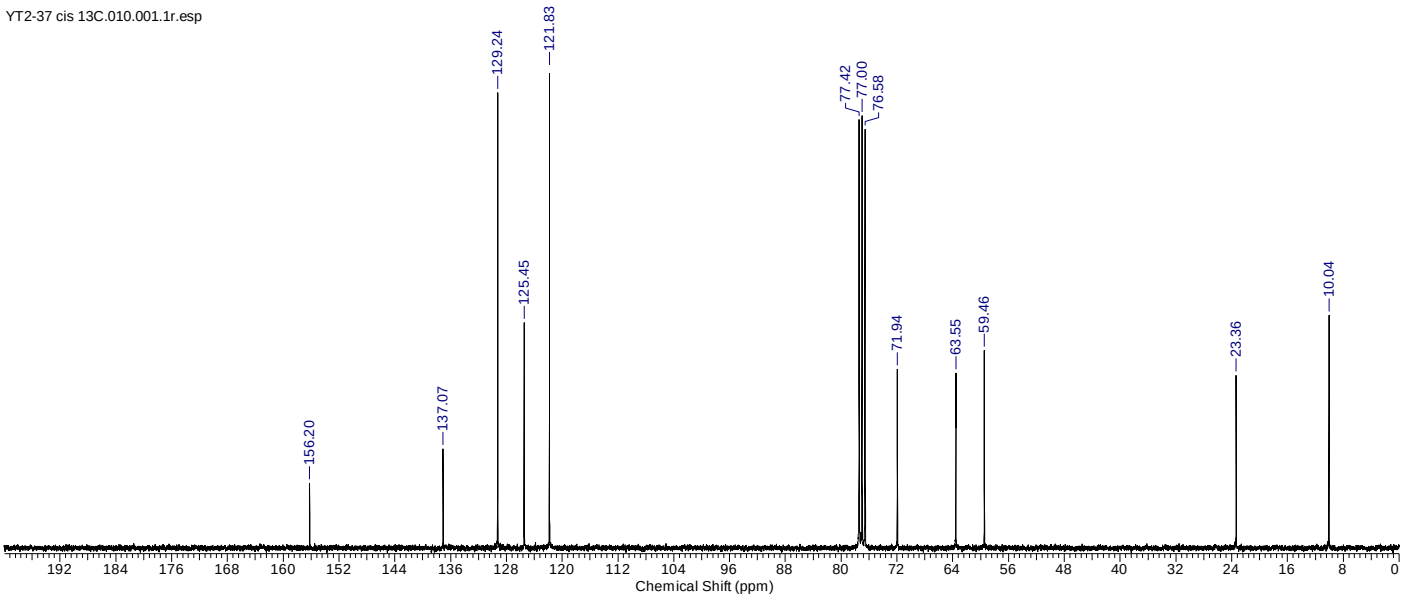
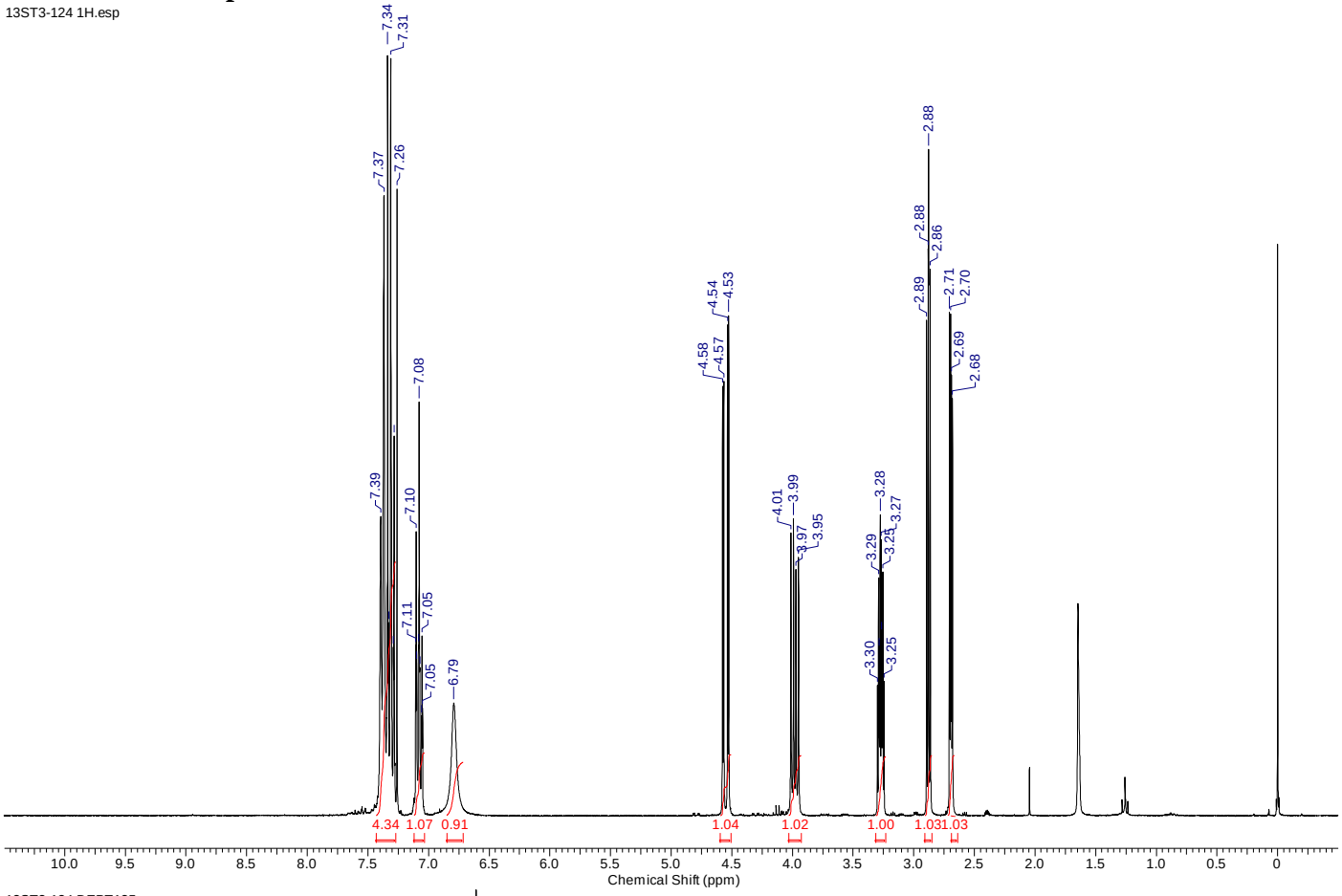
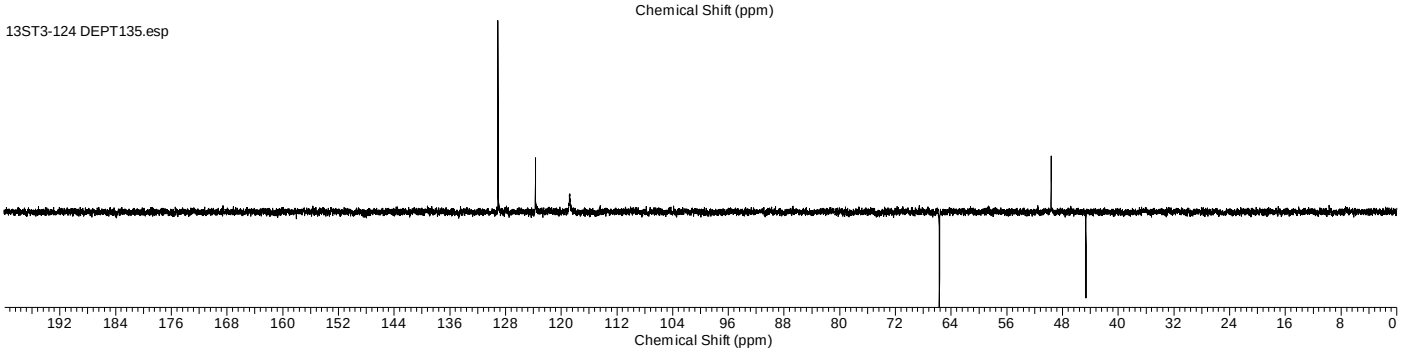


^{1}H & ^{13}C NMR Spectra of 3a

13ST3-124 1H.esp



13ST3-124 DEPT135.esp



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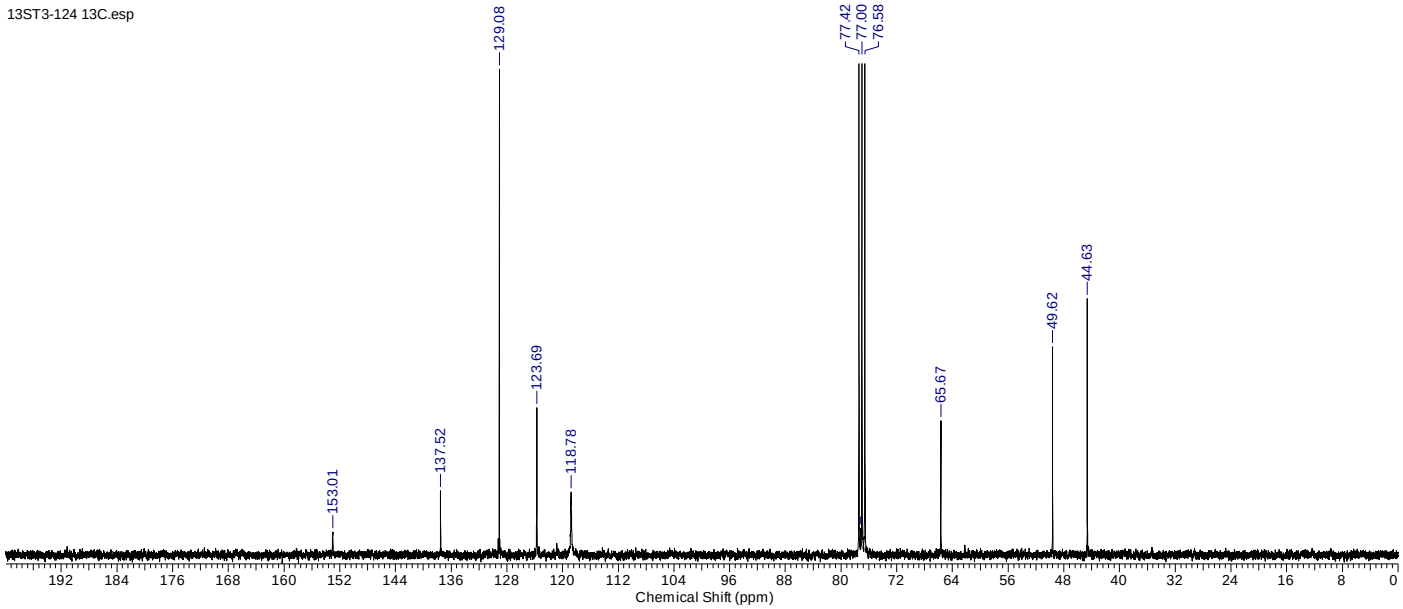
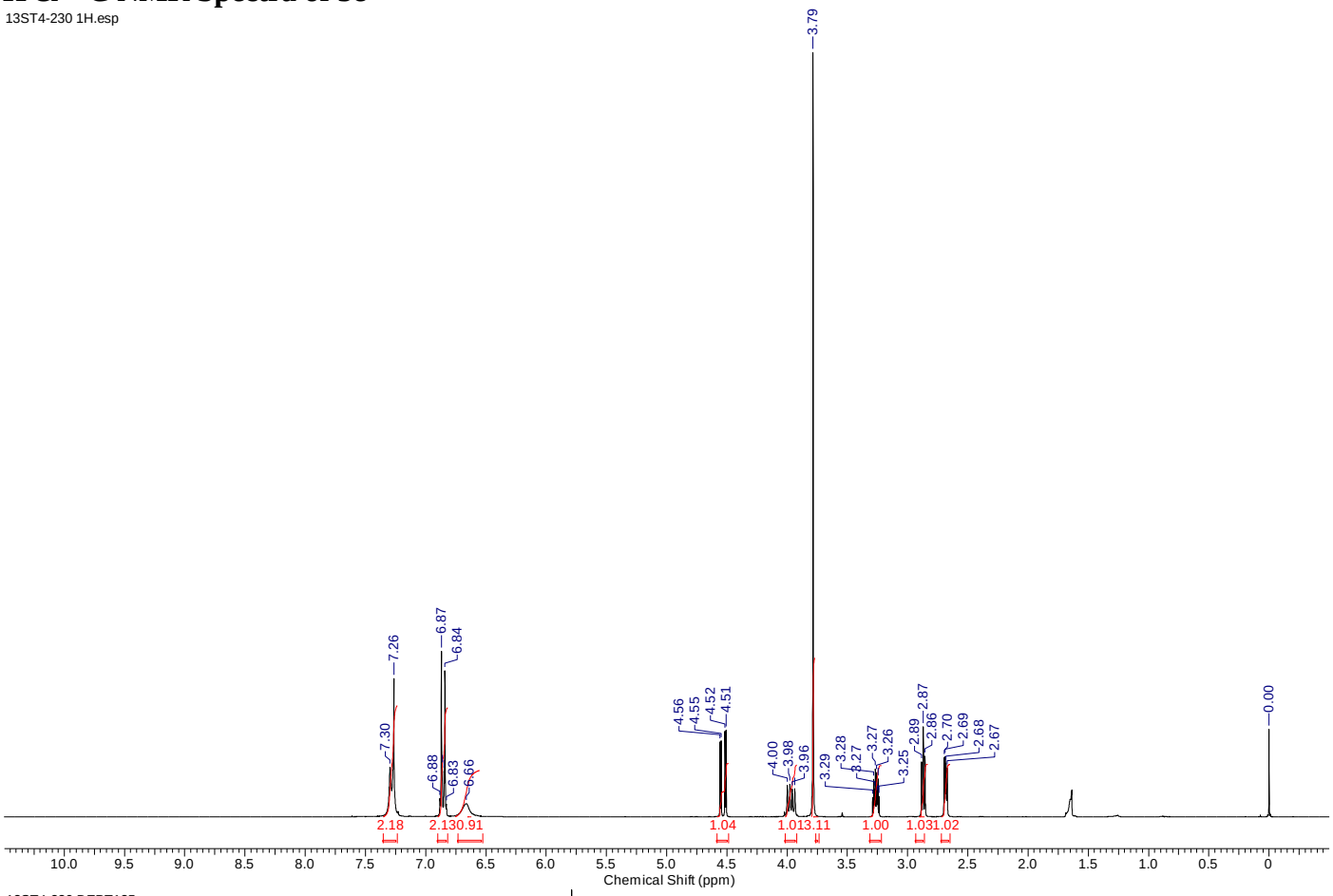
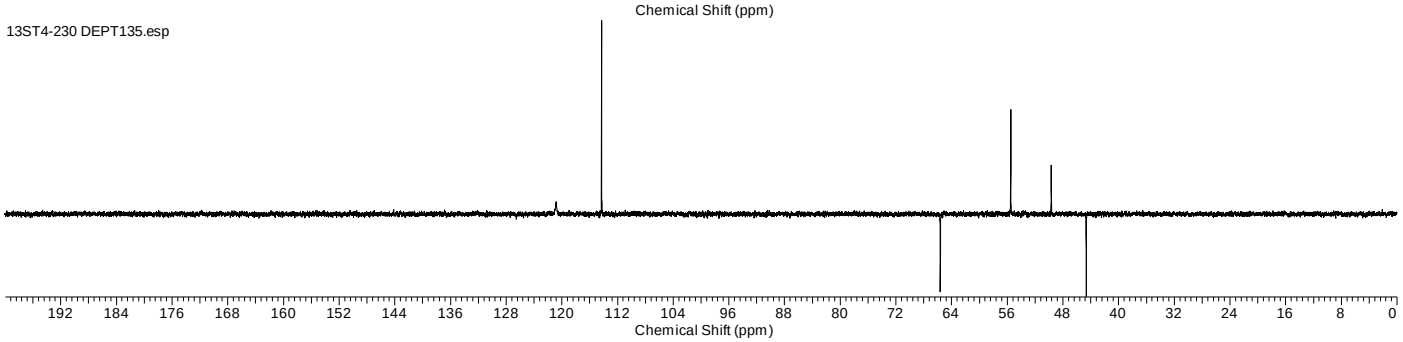
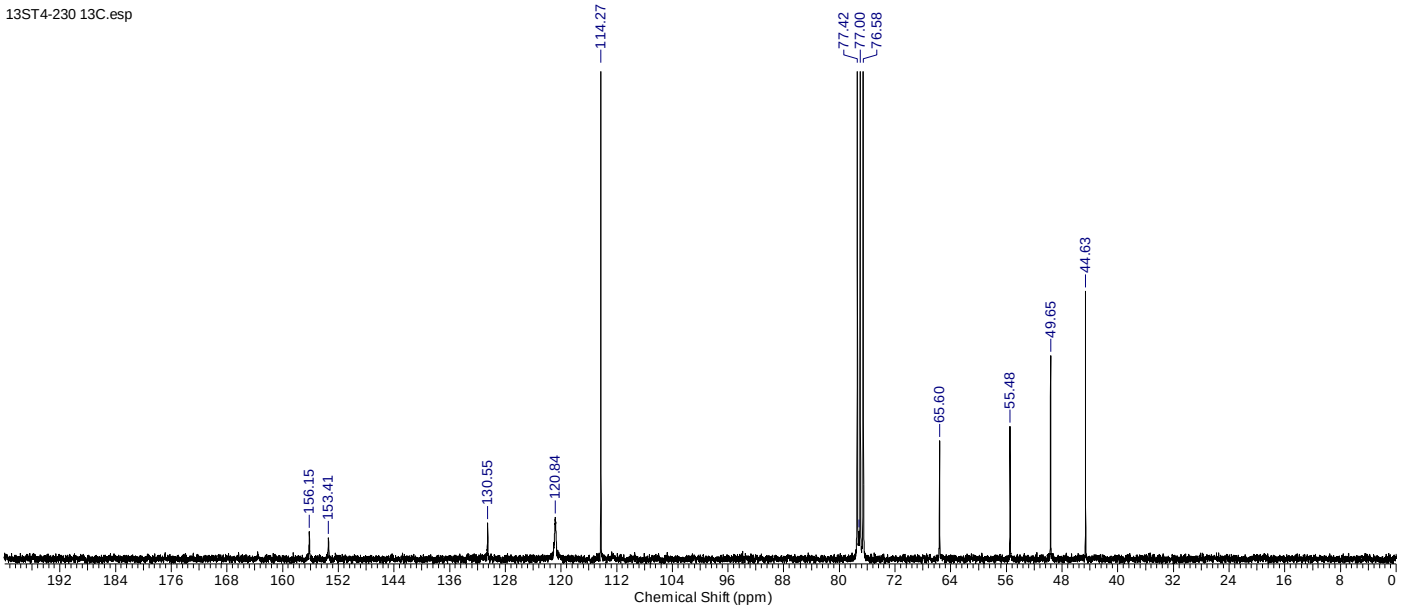


H & ^{13}C NMR Spectra of 3e

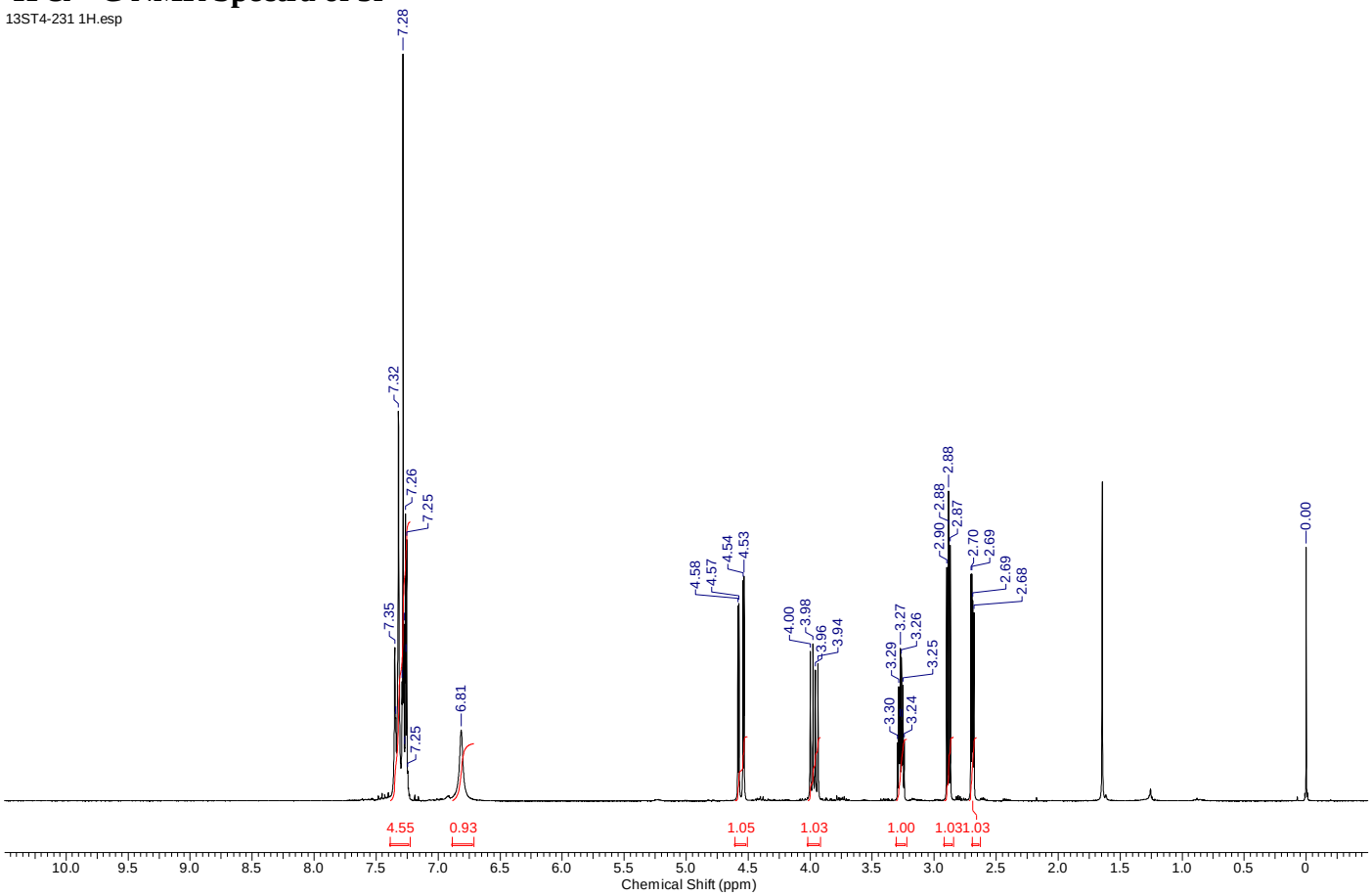




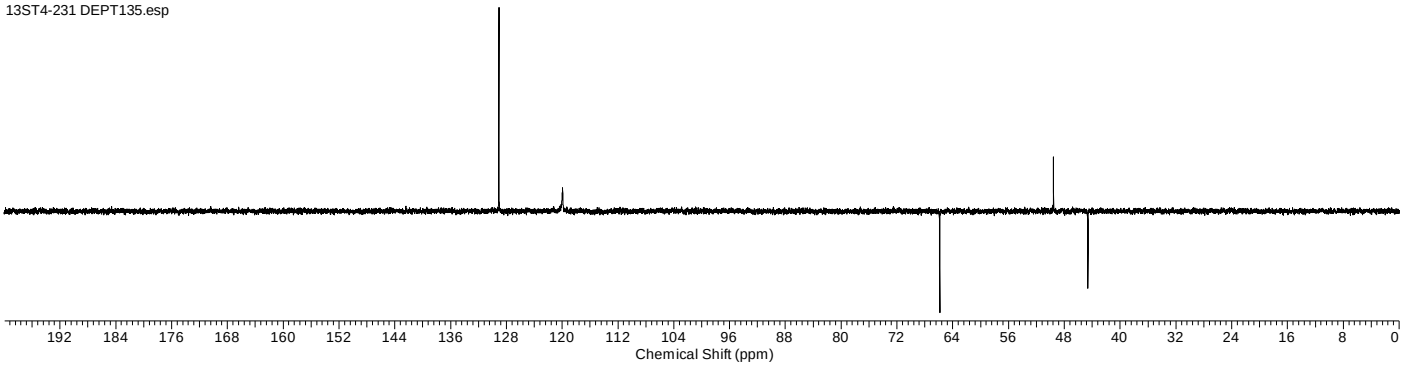


^{1}H & ^{13}C NMR Spectra of 3f

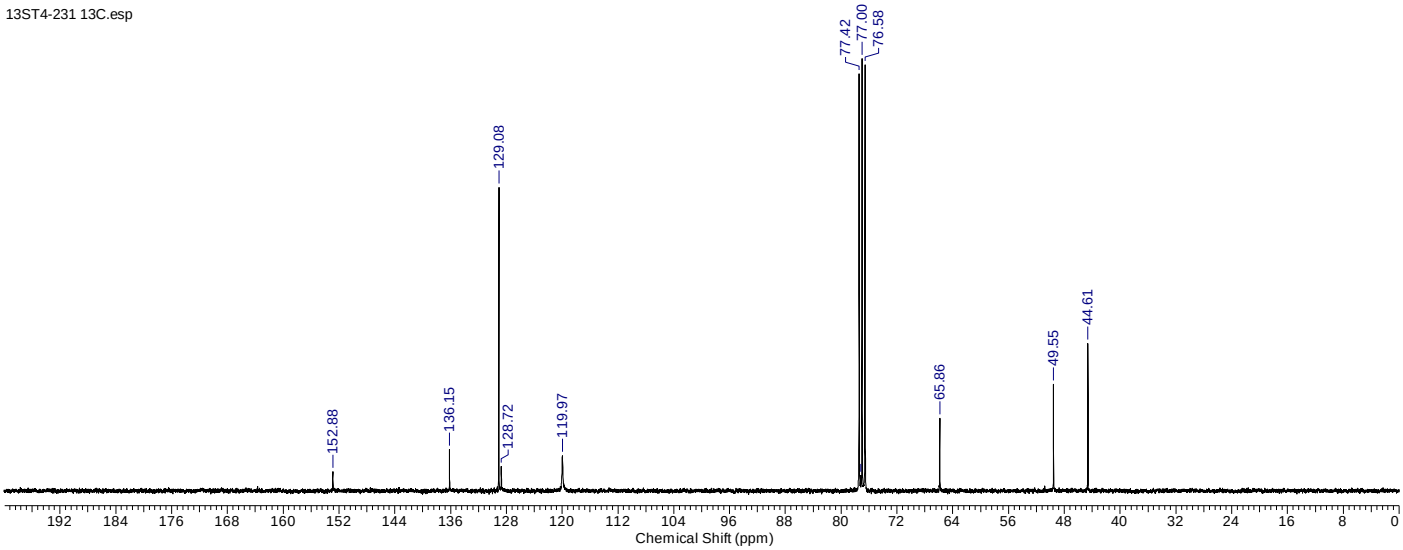
13ST4-231 1H.esp



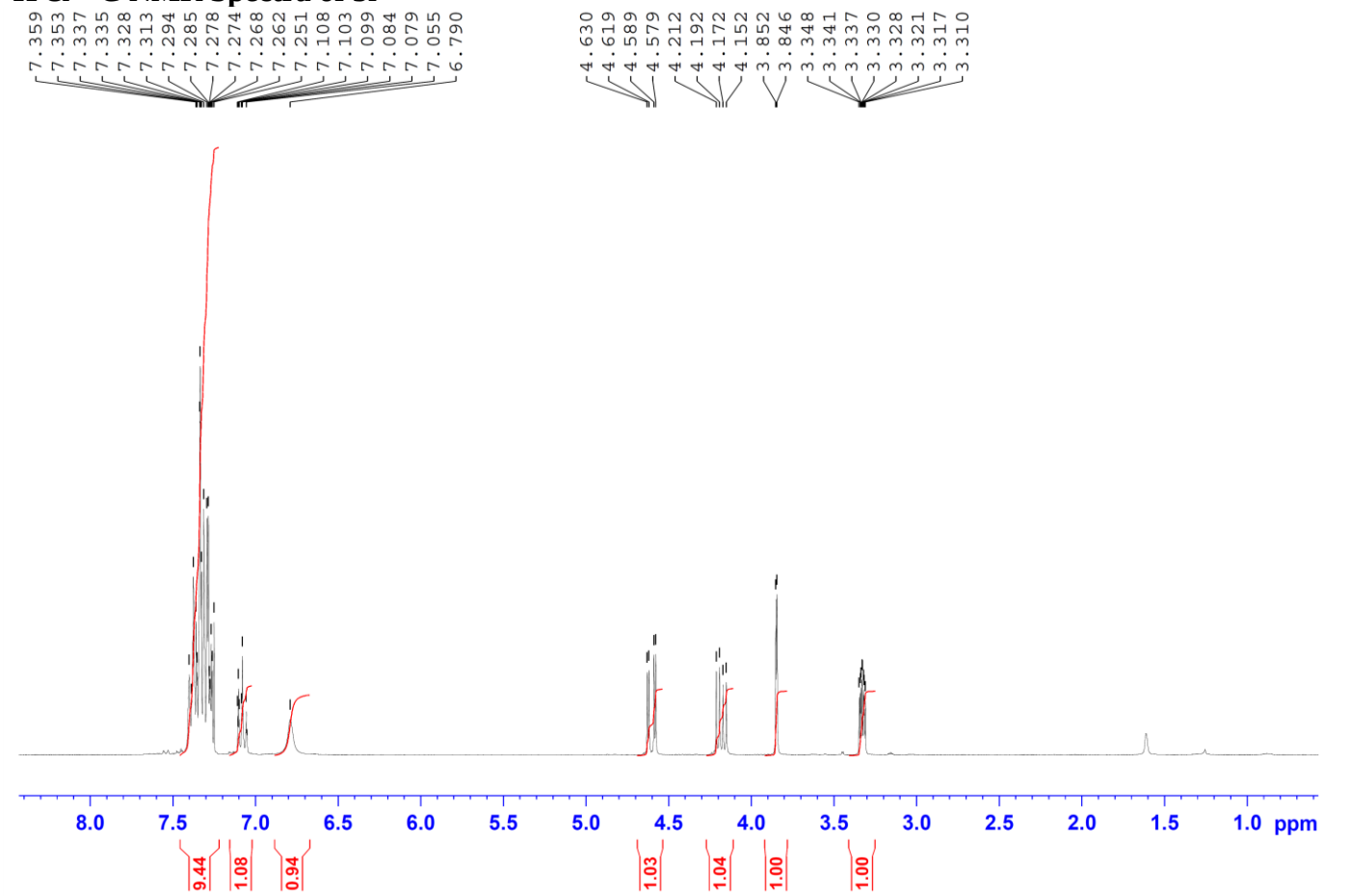
13ST4-231 DEPT135.esp

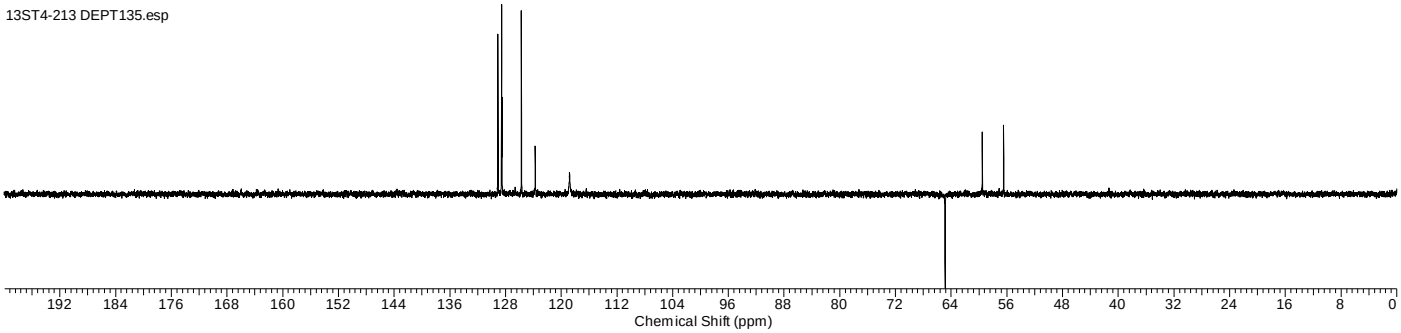


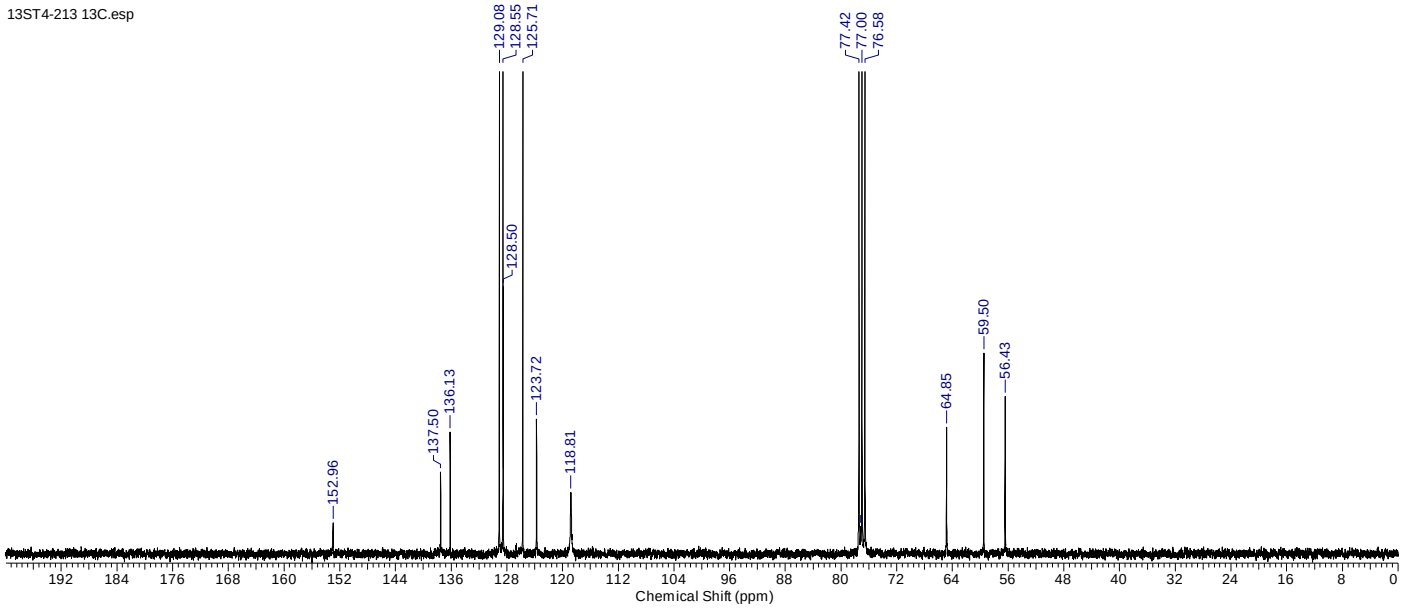
13ST4-231 13C.esp



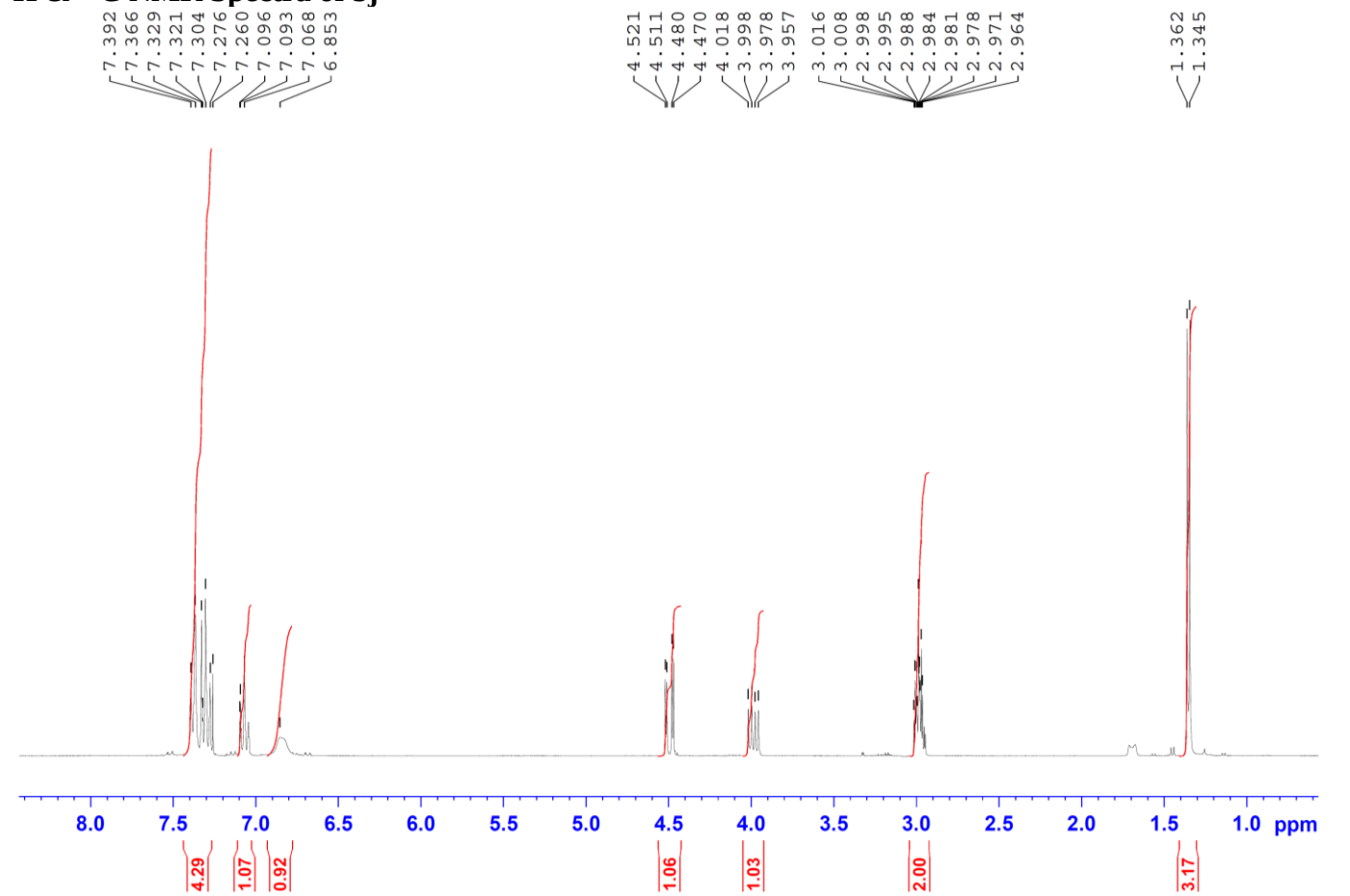
^{1}H & ^{13}C NMR Spectra of 3i

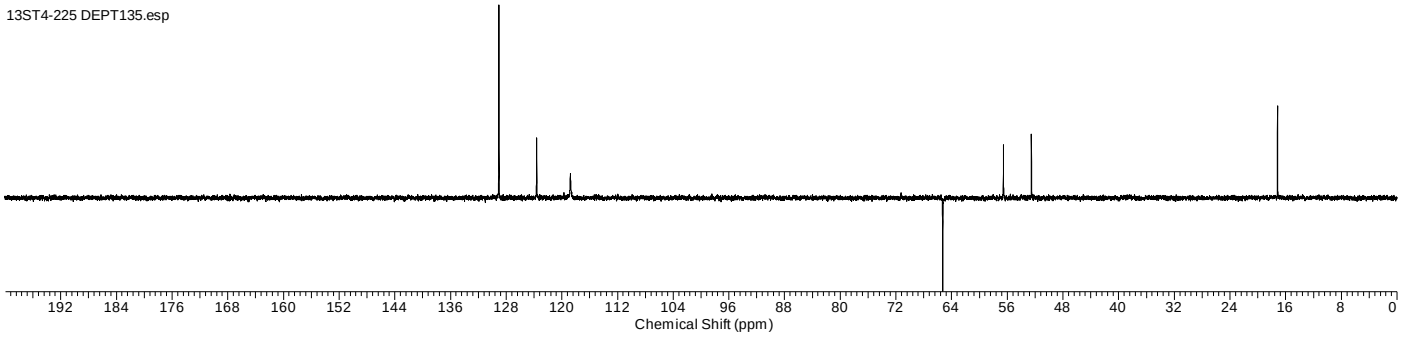


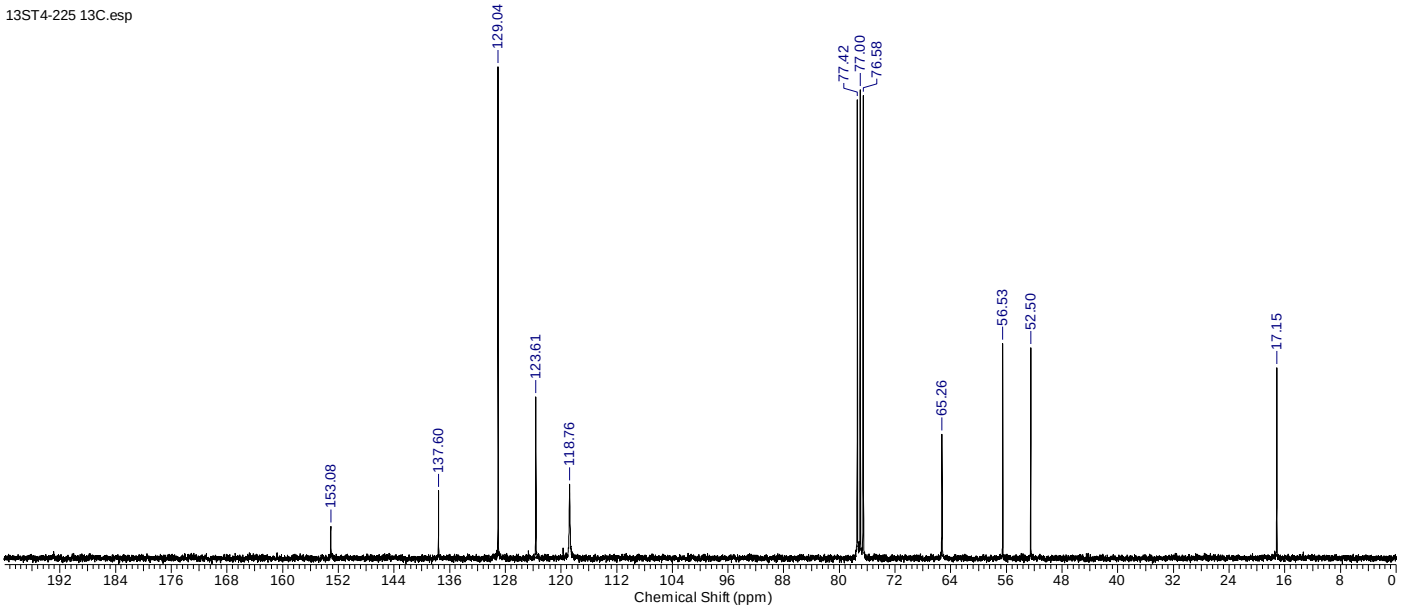




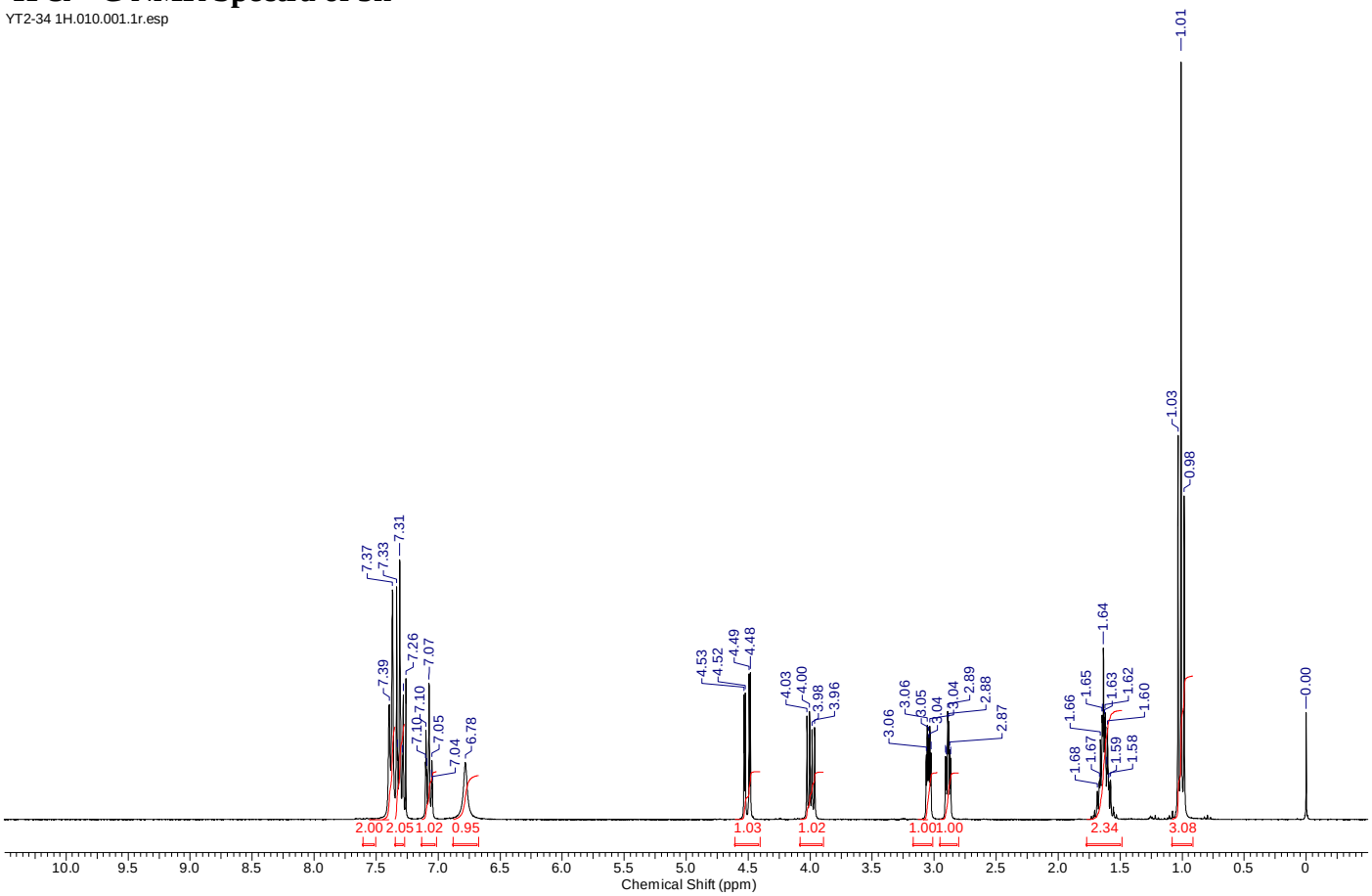
^{1}H & ^{13}C NMR Spectra of 3j

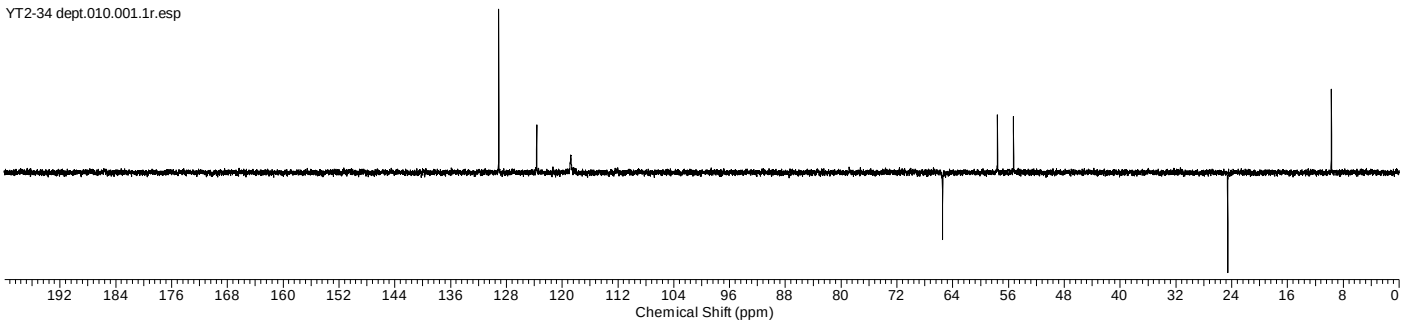


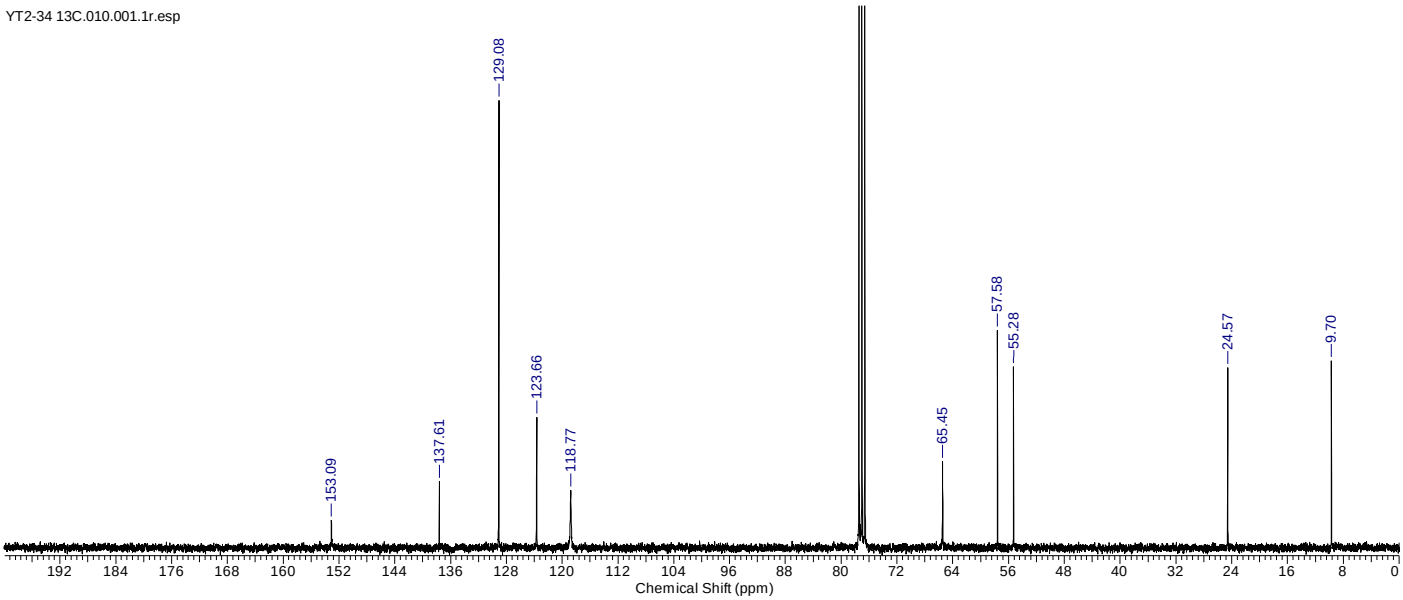




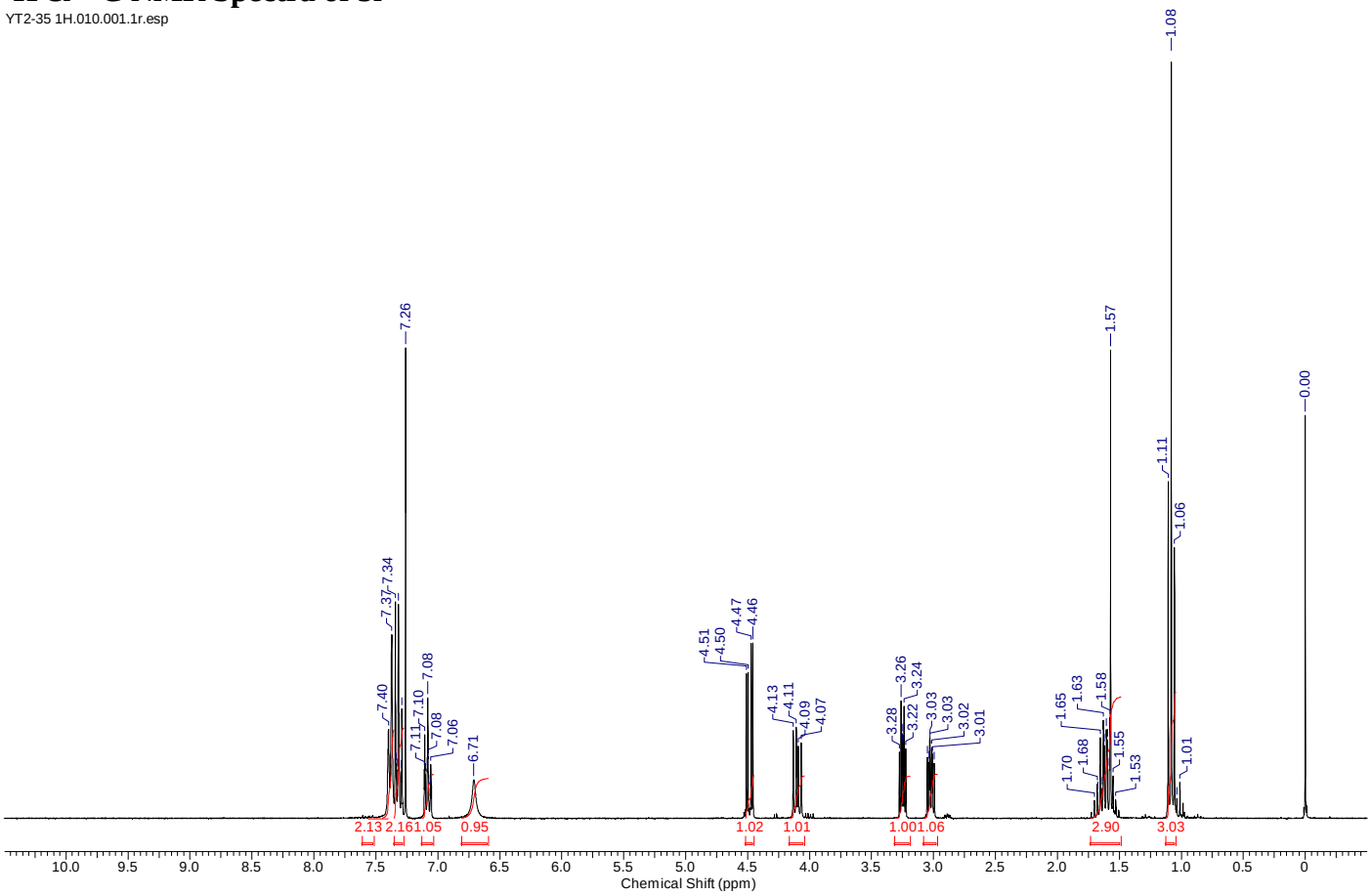
^{1}H & ^{13}C NMR Spectra of 3k

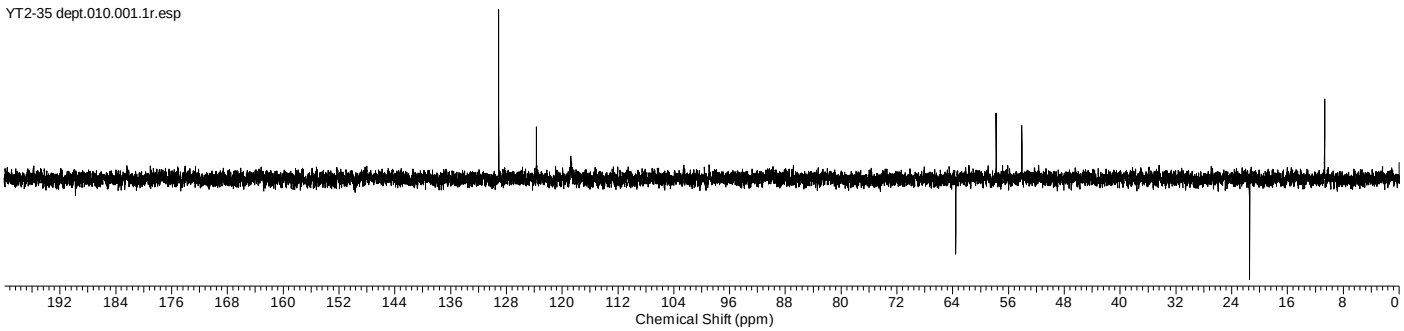


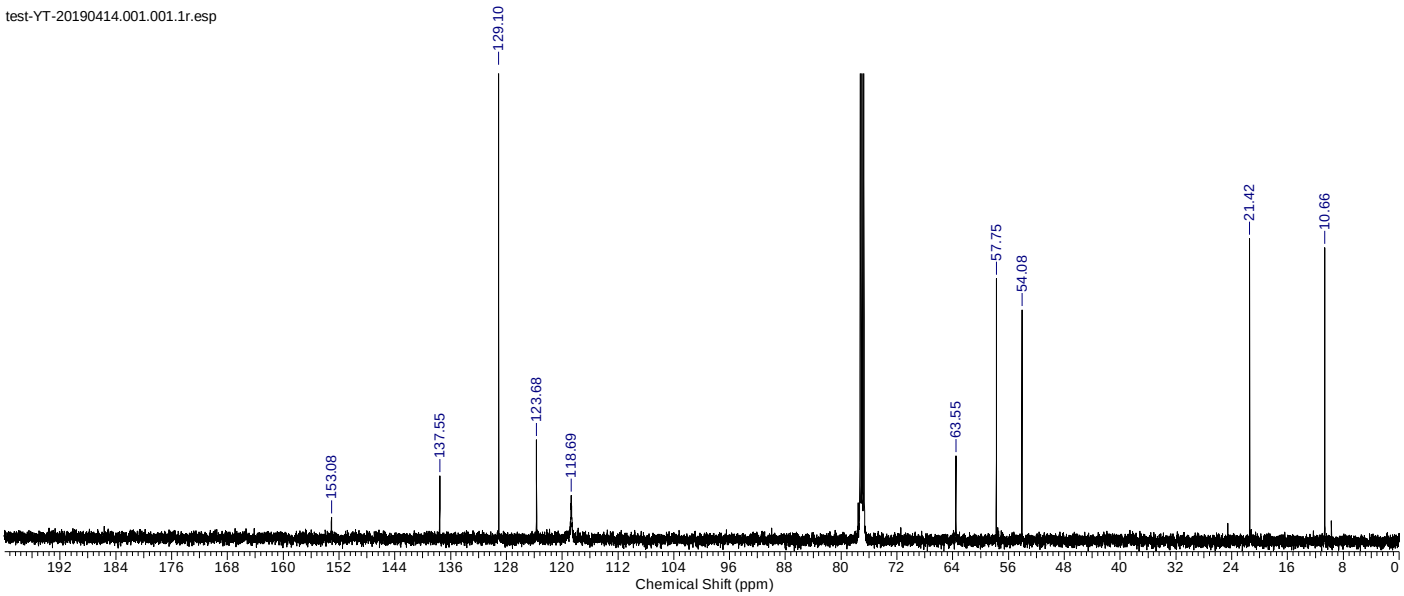




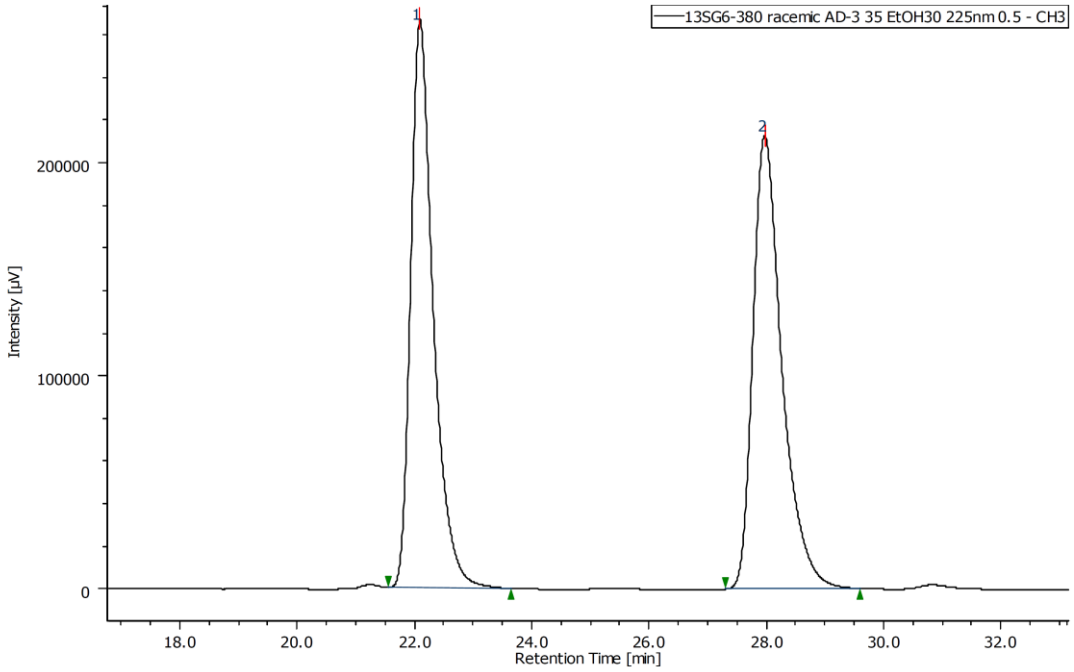
^{1}H & ^{13}C NMR Spectra of 3l





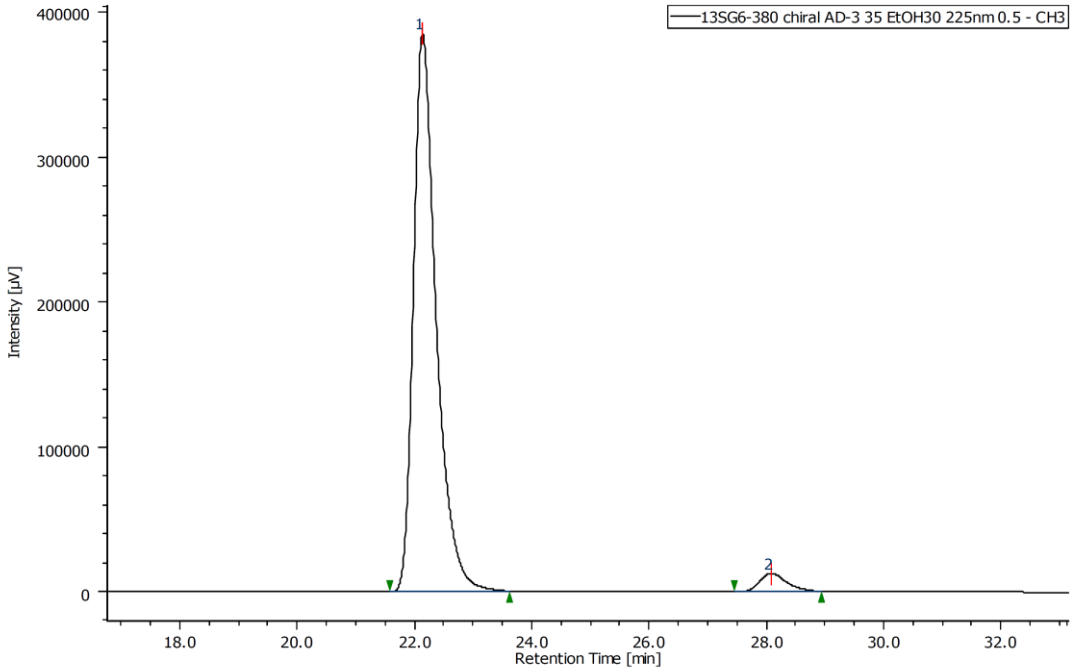


HPLC Trace of 4a



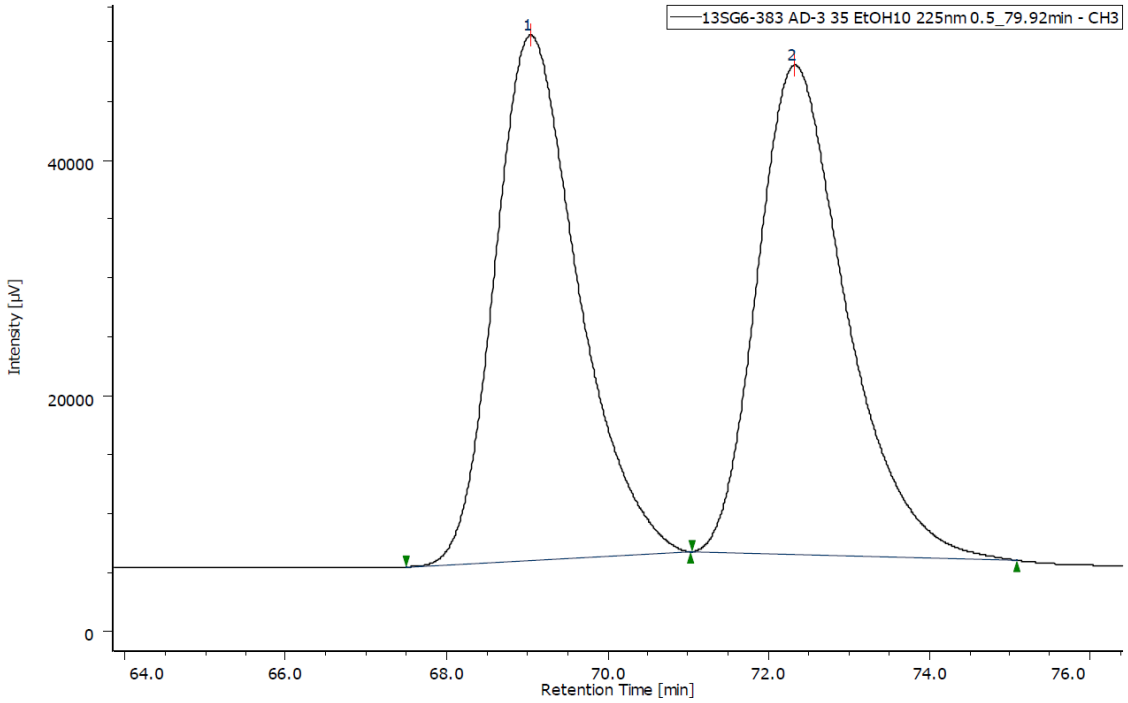
# Peak	CH	tR (min)	Area	Height	Area%
1	3	22.092	7186621	266415	49.629
2	3	27.958	7294112	212348	50.371

Racemate



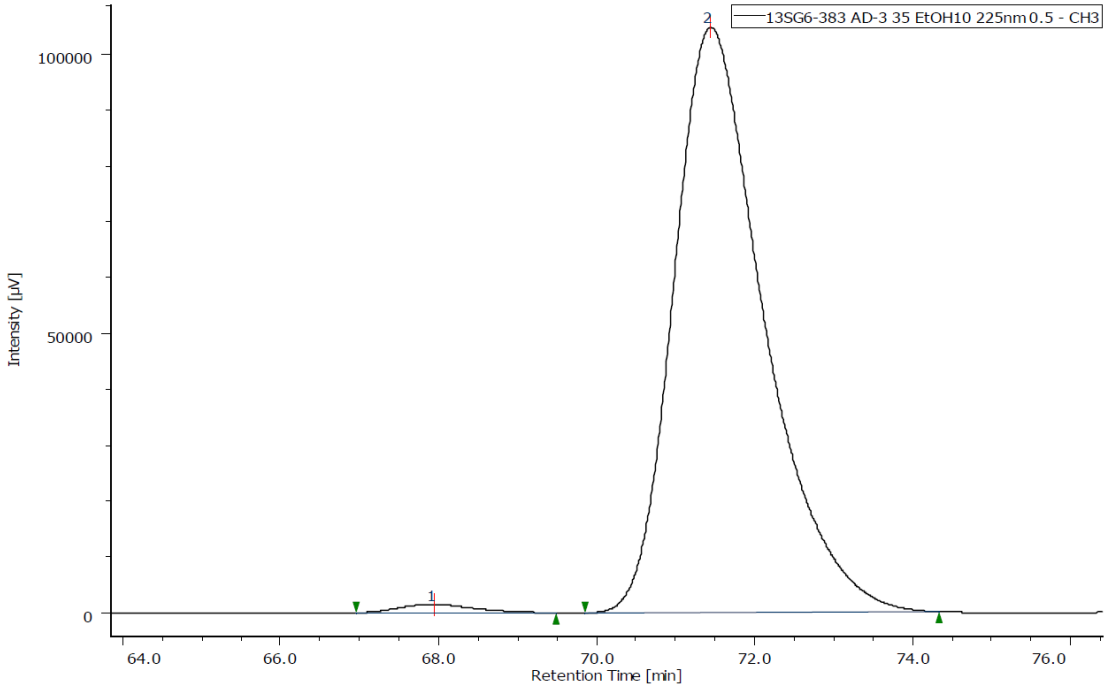
# Peak	CH	tR (min)	Area	Height	Area%
1	3	22.133	10447209	384641	96.229
2	3	28.075	409404	12710	3.771

HPLC Trace of 4c



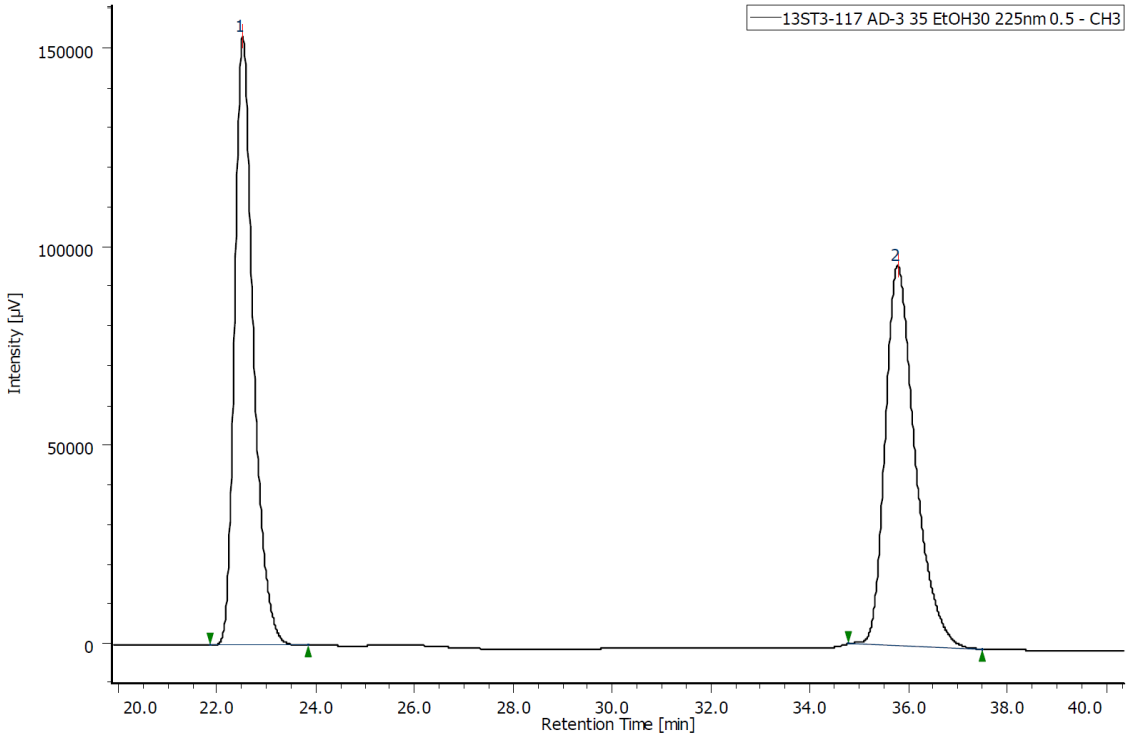
# Peak	CH	tR (min)	Area	Height	Area%
1	3	69.033	3197821	44462	50.105
2	3	72.317	3184468	41435	49.895

Racemate



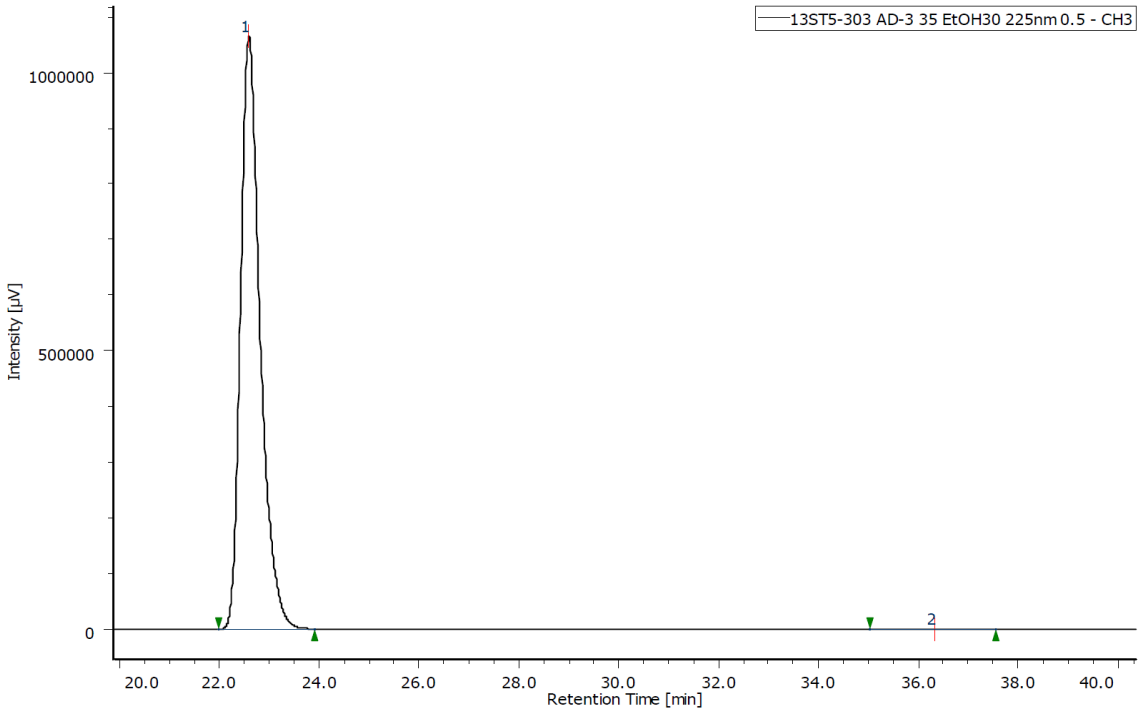
# Peak	CH	tR (min)	Area	Height	Area%
1	3	67.942	100899	1521	1.201
2	3	71.433	8297071	104584	98.799

HPLC Trace of 4e



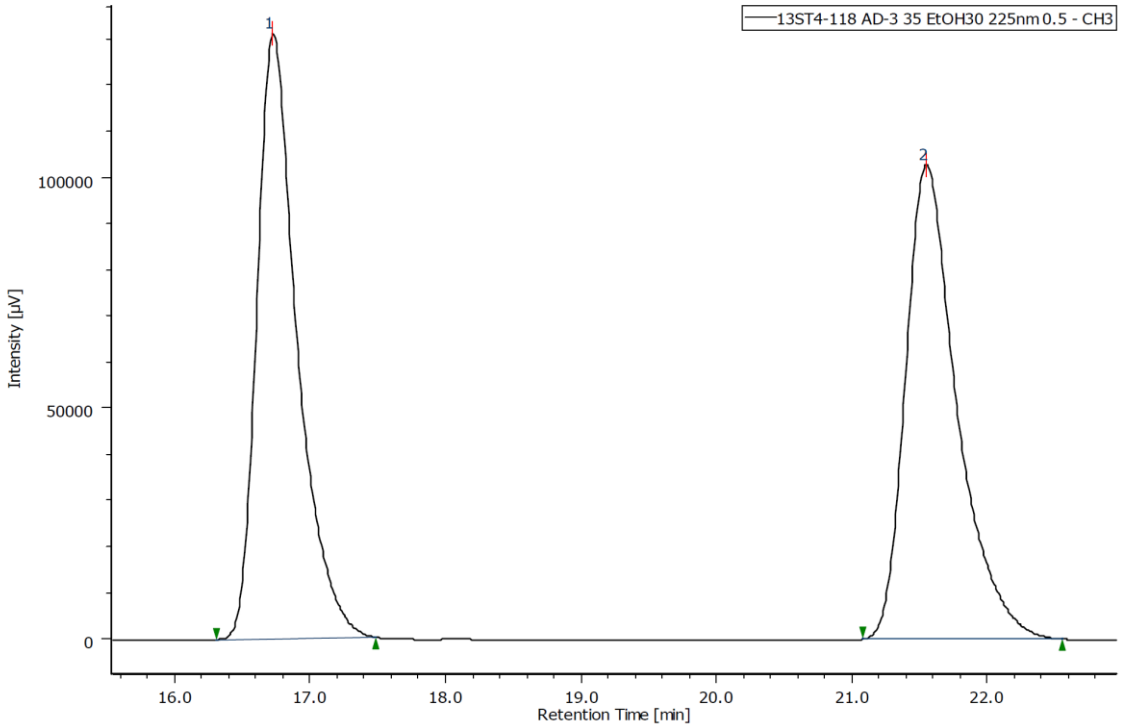
# Peak	CH	tR (min)	Area	Height	Area%
1	3	22.517	4039658	153154	50.17
2	3	35.758	4012243	95500	49.83

Racemate



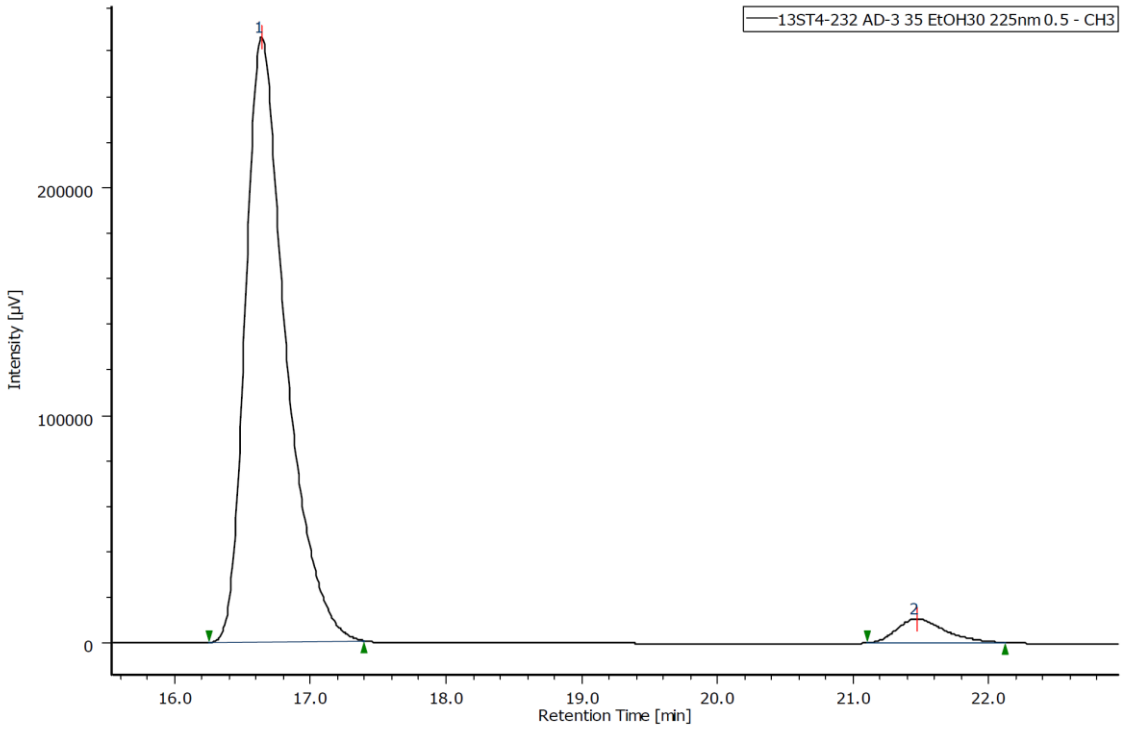
# Peak	CH	tR (min)	Area	Height	Area%
1	3	22.592	29017951	1064591	99.868
2	3	36.317	38322	861	0.132

HPLC Trace of 4f



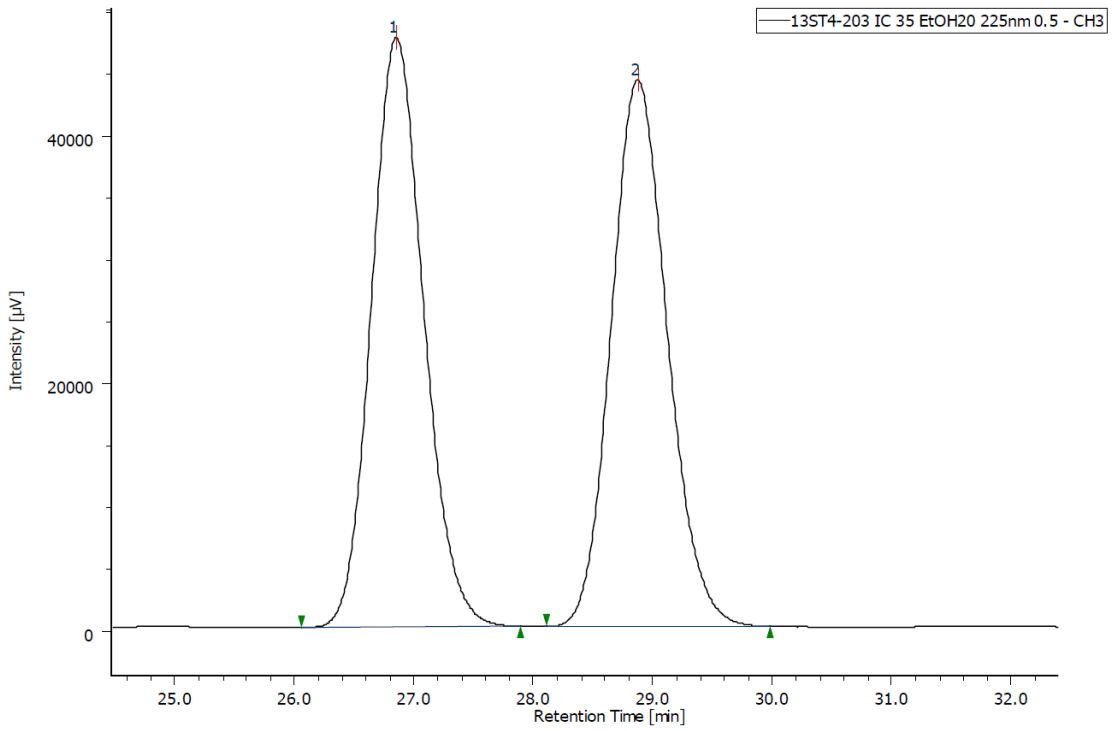
# Peak	CH	tR (min)	Area	Height	Area%
1	3	16.725	3E+06	130880	49.935
2	3	21.542	3E+06	102636	50.065

Racemate



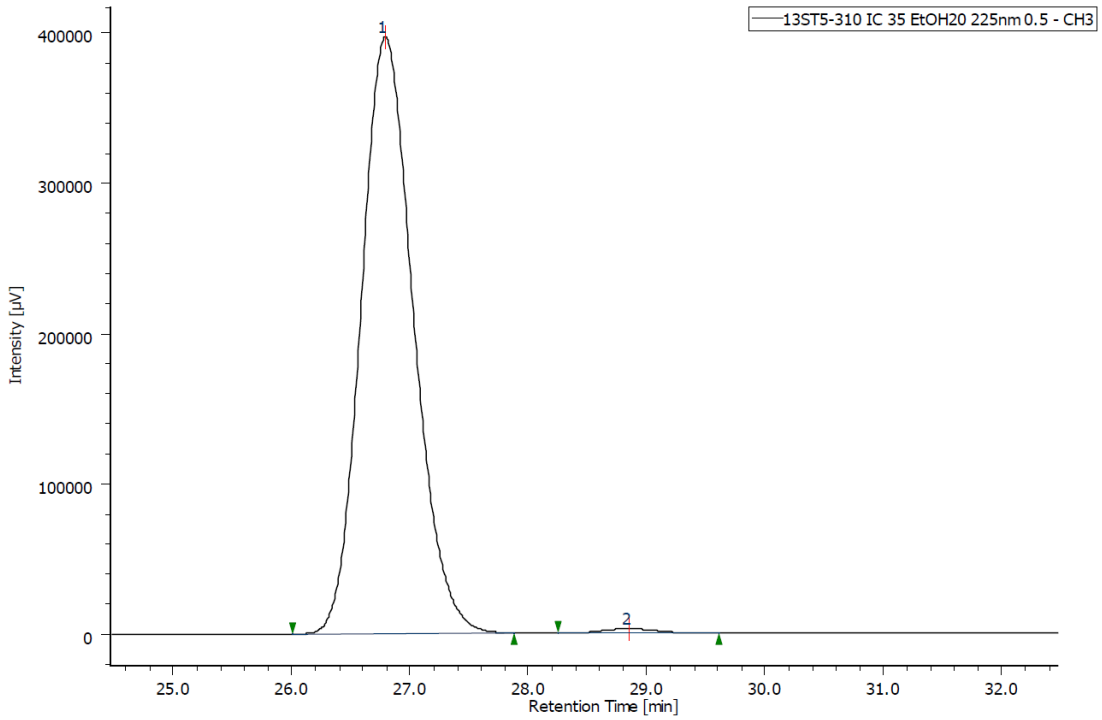
# Peak	CH	tR (min)	Area	Height	Area%
1	3	16.642	5E+06	265160	95.501
2	3	21.467	257925	10515	4.499

HPLC Trace of 4h



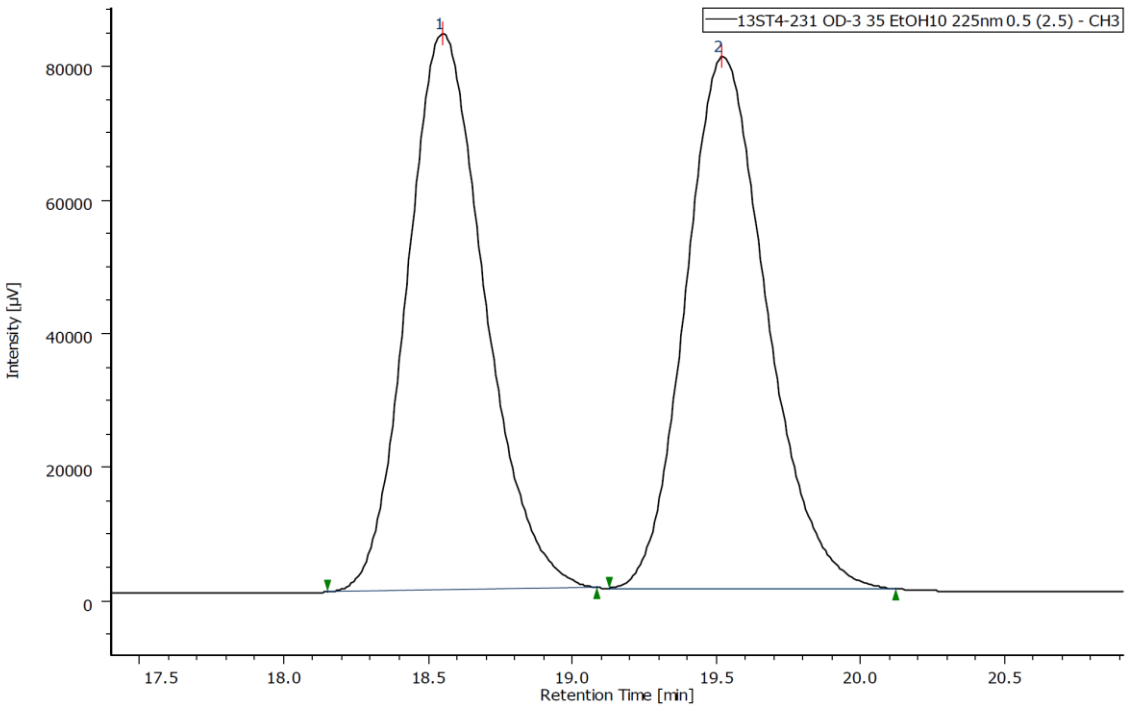
# Peak	CH	tR (min)	Area	Height	Area%
1	3	26.85	1441575	47517	50.046
2	3	28.867	1438902	44064	49.954

Racemate



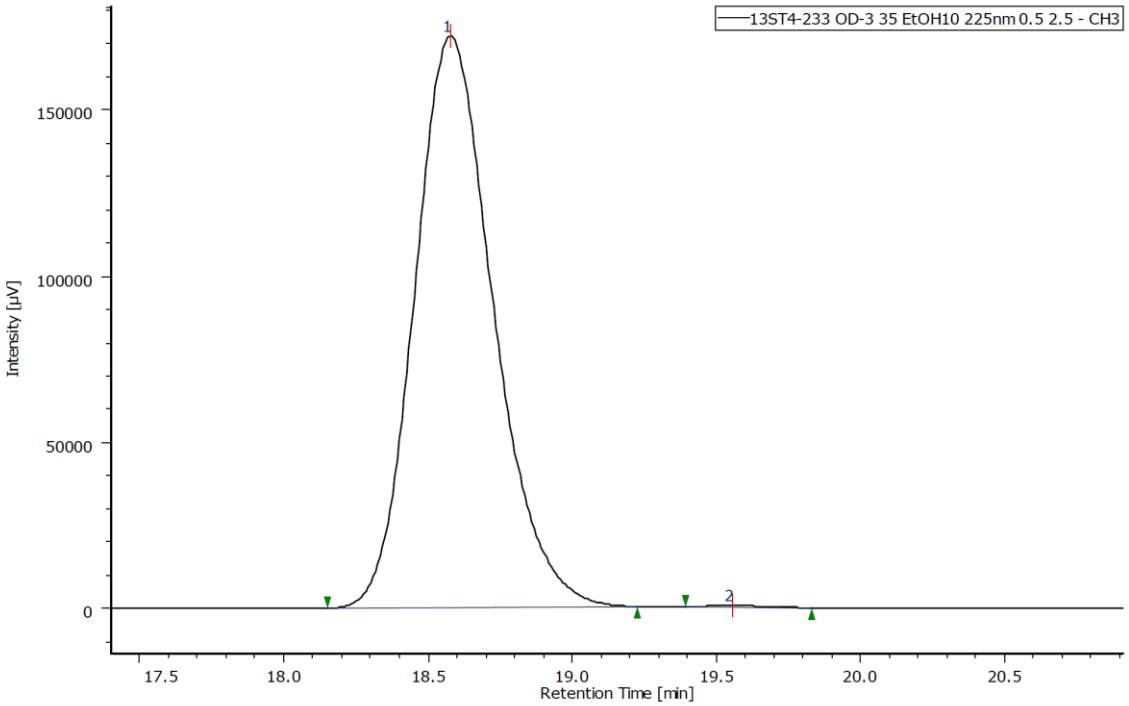
# Peak	CH	tR (min)	Area	Height	Area%
1	3	26.783	12133727	396338	99.168
2	3	28.842	101822	3201	0.832

HPLC Trace of 3f



# Peak	CH	tR (min)	Area	Height	Area%
1	3	18.550	1555227	83216	50.057
2	3	19.517	1551654	79438	49.943

Racemate



# Peak	CH	tR (min)	Area	Height	Area%
1	3	18.575	3255483	6510947	99.800
2	3	19.550	6514	13008	0.200