Electronic Supplementary Material (ESI) for Green Chemistry

Development of a robust reagent for the two-carbon homologation of aldehydes to (*E*)-α,β-unsaturated aldehydes in water.

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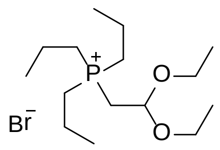
Supplemental Material

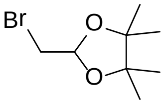
General Considerations. Reactions were carried out under air or dry argon in oven-dried glassware unless otherwise stated. All fine chemicals were obtained from Aldrich. CIMS were run on a Micromass Quattro Ultima spectrometer fitted with a direct injection probe (DIP) with ionization energy set at 70 eV. HRMS (EI) were performed with a Micromass Q-Tof Ultima spectrometer. ^{1}H, ^{13}C and ^{31}P spectra were recorded on Bruker 200, AV 600 spectrometers in $CDCl_3$ or MeOD-d_4 with TMS as internal standard, chemical shifts (δ) are reported in ppm downfield of TMS and coupling constants (J) are expressed in Hz; ^{31}P spectra were calibrated using an external reference of 85% H_3PO_4. Signal assignments were accomplished *via* analysis of HMBC, HMQC, COSY, NOESY experiments where necessary. The (*E*) to (*Z*) ratios were determined from the relative integration of the ^{1}H spectra for the olefinic protons. All melting points are corrected.

(2,2-diethoxyethyl)tributyl-phosphonium bromide (C).



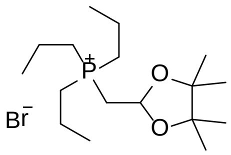
A solution of bromoacetaldehyde diethyl acetal (9.60 g, 48.7 mmol) and tri-*n*-propylphosphine (8.58 g, 53.6 mmol) in THF (27 mL) was heated for 24 hours at 60 °C. After cooling, the solvent and the residual tri-*n*-propylphosphine were removed on the rotary evaporator (75 °C at 5 mbar). The obtained yellow oil was dried under vacuum (0.1 mbar) yielding 16.38 g (95%) of phosphonium bromide as a white solid. No further purification was necessary. MP: 48-50 °C, ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: δ 4.83 (dt, J = 16.6, 3.9 Hz, 1H), 3.62 (dq, J = 9.1, 7.1 Hz, 2H), 3.50 (dq, J = 9.2, 7.0 Hz, 2H), 2.89 (dt, J = 10.8, 5.4 Hz, 2H), 2.33 – 2.20 (m, 6H), 1.60 – 1.46 (m, 6H), 1.12 – 1.03 (m, 6H), 0.98 (td, J = 7.2, 1.4 Hz, 9H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ 99.1 (d, $^{2}J(^{13}C\text{-}^{31}P)$ = 6.7 Hz), 64.1 (d, $^{4}J(^{13}C\text{-}^{31}P)$ = 12.1 Hz), 25.9 (d, $^{1}J(^{13}C\text{-}^{31}P)$ = 47.3 Hz), 22.3 (d, $^{1}J(^{13}C\text{-}^{31}P)$ = 47.0 Hz), 15.4 (d, $^{2}J(^{13}C\text{-}^{31}P)$ = 3.9 Hz), 15.2 ^{3}(d, $J(^{13}C\text{-}^{31}P)$ = 16.9 Hz), 14.8 (s); ^{31}P NMR ($CDCl_3$, 240 MHz,) δ 30.85 (s); HRES MS $(M+H)^+$ calcd. for $C_{15}H_{34}O_2P$ 278.2331; found 278.2296. Storage temperature: 0°C.

2-(bromomethyl)-4,4,5,5-tetramethyl-1,3-dioxolane (3).



The parent compound was prepared using a modified Whiting procedure[1]. To a stirred solution of pinacol (6.94 g, 58.70 mmol, 1.05 eq) and bromoacetaldehyde diethyl acetate (11.02 g, 55.90 mmol) was added *p*-toluenesulfonic acid monohydrate (0.266 g, 1.40 mmol, 0.03 eq). The mixture was heated to 100-110 °C for 15min. Ethanol was then distilled at 100 °C at 32 mbar via a Vigreux column. The yellow residue was purified by Kugelrohr distillation at 75 °C at 0.1 mbar to give 11.3 g of the desired acetal. Yield 90%, colourless oil; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 5.18 (t, J = 4.9 Hz, 1H), 3.28 (d, J = 4.9 Hz, 2H), 1.20 (s, 6H), 1.19 (s, 6H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 99.2, 82.9, 33.8, 23.9, 22.0.

Tripropyl[(4,4,5,5-tetramethyl-1,3-dioxolan-2-yl)methyl]phosphonium bromide (4).

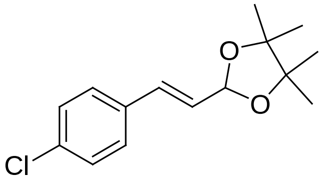


2-(Bromomethyl-4,4,5,5-tetramethyl-1,3-dioxolane (8.41 g, 37.69 mmol) and tri-*n*-propylphosphine (6.64 g, 41.45 mmol) were heated for 24 hours at 125-130 °C. After cooling, the brown solid was suspended in 5 mL of toluene, filtered and dried under vacuum (0.1 mbar) to obtain 13.2 g (91%) of the desired phosphonium bromide. No further purification was necessary. White solid; MP: 111-120 °C; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 5.24 (td, J = 5.6, 2.3 Hz, 1H), 2.68 (dd, J = 13.1, 5.6 Hz, 2H), 2.46 – 2.38 (m, 6H), 1.57 (dt, J = 11.3, 7.6 Hz, 6H), 1.12 (s, 6H), 1.09 (s, 6H), 1.03 (td, J = 7.2, 1.5 Hz, 9H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 94.8 (s), 83.2 (d, $^2J(^{13}C\text{-}^{31}P)$ = 16.0 Hz), 26.7 (d, $^1J(^{13}C\text{-}^{31}P)$ = 46.3 Hz), 23.9 (s), 21.8 (d, $^1J(^{13}C\text{-}^{31}P)$ = 23.4 Hz), 21.8 (s), 15.6 (d, $^2J(^{13}C\text{-}^{31}P)$ = 4.0 Hz), 15.3 (d, $^3J(^{13}C\text{-}^{31}P)$ = 16.8 Hz); ^{31}P NMR [$CDCl_3$, 240 MHz] δ 29.90 (s) ; HRES MS $(M+H)^+$ calcd. for $C_{17}H_{36}O_2P$ 304.2487; found 304.2515.

General Experimental Procedure

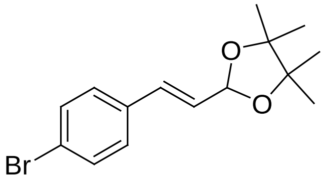
To a flask containing a magnetic stir bar was added phosphonium salt # (1.05 mmol, 1.05 equiv.) and water (1 mL) added to make a 1 *M* solution. Sodium hydroxide (3 mmol, 3 equiv.) was added followed by the corresponding aldehyde (1 mmol, 1 equiv.). The flask was then sealed and heated to 100 °C in an oil bath with vigourous stirring for 1-3 days. The contents of the flask were subsequently transferred to an Erlenmeyer flask and water added to the mixture. The slurry was stirred for 10 minutes under open air before extraction with ethyl acetate (3 x 5 mL). The combined organic layers were then dried over sodium sulphate anhydrous and concentrated under reduced pressure. The crude mixture was then filtered through a silica plug to afford the title compound which was used without further purification.

4,4,5,5-Tetramethyl-2-[(1*E*)-2-(4-chlorophenyl)ethenyl]-1,3-dioxolane (6a).



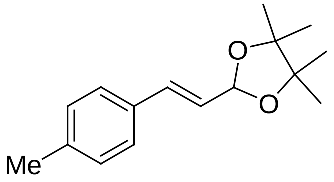
Yield 87%, Colourless oil; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 7.35 (d, J = 8.6 Hz, 2H), 7.29 (d, J = 8.6 Hz, 2H), 6.68 (d, J = 15.9 Hz, 1H), 6.14 (dd, J = 15.9, 6.6 Hz, 1H), 5.54 (d, J = 6.6 Hz, 1H), 1.29 (s, 6H), 1.28 (s, 6H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 134.5, 133.8, 132.9, 128.6, 128.4, 128.1, 100.3, 82.4, 23.9, 21.9; HREI MS $(M+H)^+$ calcd. for $C_{15}H_{19}ClO_2$: 266.1074, found 266.1075

4,4,5,5-Tetramethyl-2-[(1*E*)-2-(4-bromophenyl)ethenyl]-1,3-dioxolane (6b).



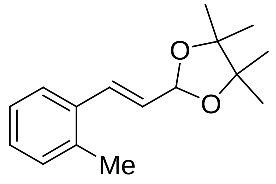
Yield 91%, Colourless oil; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 7.45 (d, J = 8.5 Hz, 4H), 7.29 (d, J = 8.5 Hz, 4H), 6.67 (d, J = 15.9 Hz, 2H), 6.16 (dd, J = 15.9, 6.7 Hz, 2H), 5.54 (d, J = 6.7 Hz, 2H), 1.29 (s, 13H), 1.28 (s, 12H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 134.9, 133.1, 131.6, 128.5, 128.4, 122.00, 100.3, 82.4, 23.9, 22.0; HREI MS $(M-H)^+$ calcd. for $C_{15}H_{18}BrO_2$: 309.0490, found 309.0490

4,4,5,5-tetramethyl-2-[(1*E*)-2-(4-Methylphenyl)ethenyl]-1,3-dioxolane (6c).



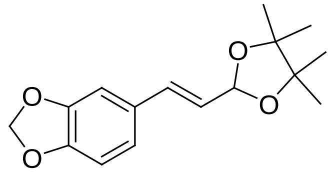
Yield 89%, Colourless oil; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 7.31 (d, *J* = 8.1 Hz, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 6.69 (d, *J* = 15.9 Hz, 1H), 6.11 (dd, *J* = 15.9, 6.8 Hz, 1H), 5.54 (d, *J* = 6.8 Hz, 1H), 2.34 (s, 3H), 1.28 (s, 6H), 1.27 (s, 6H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 137.9, 134.3, 133.2, 129.1, 126.8, 126.6, 100.8, 82.2, 24.00, 21.9, 21.2; HRES MS $(M+H)^+$ calcd. for $C_{16}H_{23}O_2$: 247.1698, found 247.1703

4,4,5,5-tetramethyl-2-[(1*E*)-2-(4-Methylphenyl)ethenyl]-1,3-dioxolane (6d).



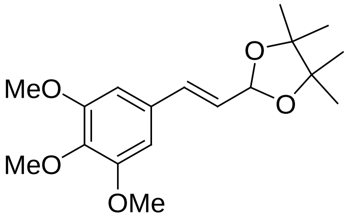
Yield 90%, Colourless oil; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 7.52 – 7.47 (m, 6H), 7.22 – 7.11 (m, 22H), 6.96 (d, *J* = 15.7 Hz, 6H), 6.06 (dd, *J* = 15.7, 6.7 Hz, 6H), 5.58 (d, *J* = 6.7 Hz, 7H), 2.35 (s, 18H), 1.29 (s, 38H), 1.28 (s, 35H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 135.7, 135.1, 132.1, 130.1, 129.0, 127.9, 126.1, 125.9, 100.7, 82.2, 23.9, 21.9, 19.6; HRES MS $(M+H)^+$ calcd. for $C_{16}H_{23}O_2$: 247.1698, found 247.1687

4,4,5,5-Tetramethyl-2-[(1*E*)-2-(1,3-benzodioxol-5-yl)ethenyl]-1,3-dioxolane (6e).



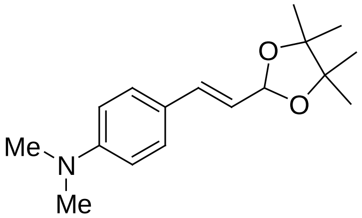
Yield 96%, Beige solid; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 6.94 (d, *J* = 1.6 Hz, 1H), 6.83 (dd, *J* = 8.0, 1.5 Hz, 1H), 6.74 (d, *J* = 8.0 Hz, 1H), 6.61 (d, *J* = 15.8 Hz, 1H), 5.97 (dd, *J* = 15.97, 6.84 Hz, 1H), 5.95 (s, 2H), 5.50 (d, *J* = 6.8 Hz, 1H), 1.27 (s, 6H), 1.25 (s, 6H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 147.9, 147.7, 134.1, 130.5, 125.8, 121.8, 108.2, 106.1, 101.1, 100.8, 82.3, 24.0, 22.0. HRES MS $(M+H)^+$ calcd. for $C_{16}H_{21}O_4$: 276.1964, found 276.1963.

4,4,5,5-Tetramethyl-2-[(1*E*)-2-(3,4,5-trimethoxyphenyl)ethenyl]-1,3-dioxolane (6f).



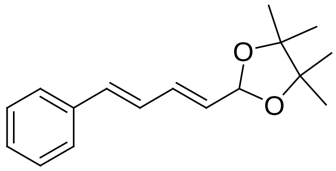
Yield 91%, Colourless Oil; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 6.63 (d, *J* = 15.6 Hz, 1H), 6.62 (s, 2H), 6.04 (dd, *J* = 15.8, 6.6 Hz, 1H), 5.52 (d, *J* = 6.6 Hz, 1H), 3.86 (s, 6H), 3.84 (s, 3H), 1.27 (s, 6H), 1.26 (s, 6H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 153.2, 138.2, 134.3, 131.8, 127.1, 104.0, 100.4, 82.3, 56.1, 24.0, 22.0; HRES MS $(M+H)^+$ calcd. for $C_{18}H_{27}O_5$: 324.1893, found 324.1898.

4,4,5,5-Tetramethyl-2-[2-(4-bromophenyl)ethenyl]-1,3-dioxolane (6g).



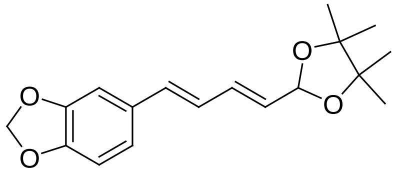
Yield 90%, Yellow oil; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 7.30 (d, *J* = 8.8 Hz, 2H), 6.65 (d, *J* = 8.8 Hz, 2H), 6.62 (d, *J* = 15.8 Hz, 1H), 5.94 (dd, *J* = 7.0, 15.8 Hz, 1H), 5.52 (d, *J* = 7.0 Hz, 1H), 2.95 (s, 6H), 1.28 (s, 6H), 1.26 (s, 6H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 150.5, 134.9, 128.2, 124.5, 123.1, 112.2, 101.5, 82.2, 40.5, 24.2, 22.1; HRES MS $(M+H)^+$ calcd. for $C_{17}H_{26}NO_2$: 278.1474, found 278.1482.

4,4,5,5-Tetramethyl-2-[(1*E*,3*E*)-4-penyl-1,3-butadien-1-yl]-1,3-dioxolane (6h).



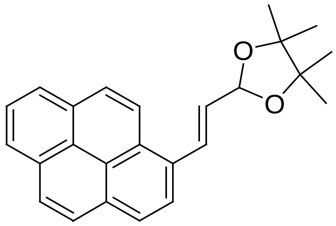
Yield 36%, yellow oil; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 7.45 – 7.31 (m, 5H), 6.83 (dd, *J* = 15.6, 10.6 Hz, 1H), 6.64 (d, *J* = 15.7 Hz, 1H), 6.54 (dd, *J* = 15.2, 10.6 Hz, 1H), 5.82 (dd, *J* = 15.2, 6.8 Hz, 1H), 5.50 (d, *J* = 6.8 Hz, 1H), 1.29 (s, 6H), 1.29 (s, 6H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 136.8, 134.3, 134.3, 131.3, 128.5, 127.8, 127.5, 126.5, 100.2, 82.2, 23.9, 21.9; HREI MS $(M)^+$ calcd. for $C_{17}H_{22}O_2$: 258.1620, found 258.1621.

4,4,5,5-Tetramethyl-2-[(1*E*,3*E*)-4-(1,3-benzodioxol-5-yl)-1,3-butadien-1-yl]-1,3-dioxolane (6i).



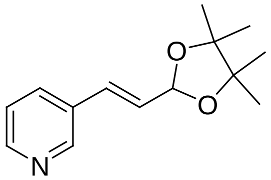
Yield 53%, yellow; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 6.94 (d, *J* = 1.5 Hz, 1H), 6.82 (dd, *J* = 8.0, 1.5 Hz, 1H), 6.75 (d, *J* = 8.0 Hz, 1H), 6.61 (dd, *J* = 15.5, 10.5 Hz, 1H), 6.50 (d, *J* = 15.6 Hz, 1H), 6.46 (dd, *J* = 15.1, 10.5 Hz, 1H), 5.95 (s, 2H), 5.72 (dd, *J* = 15.1, 6.9 Hz, 1H), 5.44 (d, *J* = 6.9 Hz, 1H), 1.25 (s, 7H), 1.24 (s, 6H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 148.0, 147.5, 134.5, 134.0, 131.5, 130.5, 126.0, 121.6, 108.4, 105.5, 101.1, 100.3, 82.2, 24.0, 22.0; HRES MS $(M+H)^+$ calcd. for $C_{18}H_{23}O_4$: 304.1631; found 304.1656.

4,4,5,5-Tetramethyl-2-[(1*E*)-2-(1-pyrenyl)ethenyl]-1,3-dioxolane (6j).



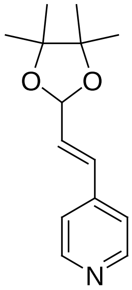
Yield 92%, Yellow solid; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 8.37 (d, *J* = 9.0 Hz, 1H), 8.22 (d, *J* = 8.0 Hz, 1H), 8.17 (d, *J* = 7.6 Hz, 2H), 8.13 (d, *J* = 8.0 Hz, 1H), 8.09 (d, *J* = 9.3 Hz, 1H), 8.04 (d, *J* = 9.0 Hz, 1H), 8.02 (d, *J* = 8.9 Hz, 1H), 7.99 (t, *J* = 7.4 Hz, 1H), 7.81 (d, *J* = 15.8 Hz, 1H), 6.42 (dd, *J* = 6.7, 15.7 Hz, 1H), 5.79 (d, *J* = 6.7 Hz, 1H), 1.37 (s, 6H), 1.36 (s, 6H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 131.7, 131.5, 131.3, 131.1, 131.0, 130.7, 128.5, 127.8, 127.6, 127.5, 126.1, 125.4, 125.2, 125.1, 125.0, 124.5, 123.1, 101.0, 82.6, 24.2, 22.2; HRES MS $(M+H)^+$ calcd. for $C_{25}H_{25}O_2$: 357.1855, found 357.1867.

4,4,5,5-Tetramethyl-2-[(1*E*)-2-(3-pyridyl)ethenyl]-1,3-dioxolane (6k).



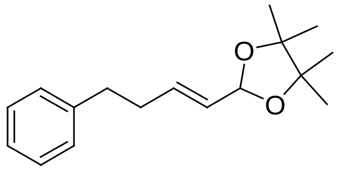
Yield 75%, Beige solid; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 8.61 (d, *J* = 2.2 Hz, 1H), 8.49 (dd, *J* = 1.7, 4.8 Hz, 1H), 7.73 (dt, *J* = 1.9, 7.9 Hz, 1H), 7.25 (dd, *J* = 4.7, 7.9 Hz, 1H), 6.71 (d, *J* = 16.0 Hz, 1H), 6.23 (dd, *J* = 6.5, 16.0 Hz, 1H), 5.54 (d, *J* = 6.5 Hz, 1H), 1.28 (s, 6H), 1.27 (s, 6H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 149.4, 148.9, 133.4, 131.8, 130.7, 130.3, 123.6, 100.2, 82.7, 24.1, 22.2; HRES MS $(M+H)^+$ calcd. for $C_{14}H_{20}NO_2$: 234.1493, found 234.1499.

4,4,5,5-Tetramethyl-2-[(1*E*)-2-(4-pyridyl)ethenyl]-1,3-dioxolane (6l).



Yield 83%, Beige solid; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 8.56 (dd, *J* = 1.6, 4.6 Hz, 2H), 7.27 (dd, *J* = 1.6, 4.6 Hz, 2H), 6.67 (d, *J* = 16.0 Hz, 1H), 6.36 (dd, *J* = 6.2, 16.0 Hz, 1H), 5.55 (d, *J* = 6.2 Hz, 1H), 1.29-1.27 (m, 12H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 150.3, 143.6, 132.7, 131.6, 121.4, 99.8, 82.8, 24.07, 22.1; HRES MS $(M+H)^+$ calcd. for $C_{14}H_{20}NO_2$: 234.1494, found 234.1499.

4,4,5,5-Tetramethyl-2-[(1*E*)-4-phenyl-1-buten-1-yl]-1,3-dioxolane (6m).



The parent compound was prepared using the general procedure. Yield 54%, Yellow oil; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 7.39 – 7.35 (m, 6H), 7.27 (s, 4H), 6.01 (dt, *J* = 15.3, 6.6 Hz, 2H), 5.64 (dd, *J* = 15.4, 7.2 Hz, 2H), 5.42 (d, *J* = 7.2 Hz, 2H), 2.82 – 2.79 (m, 5H), 2.51 – 2.44 (m, 4H), 1.32 (s, 12H), 1.32 (s, 12H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 141.6, 136.1, 129.2, 128.3, 125.8, 100.8, 82.1, 35.1, 33.8, 24.0, 21.9; HREI MS $(M)^+$ calcd. for $C_{17}H_{24}O_2$: 260.1776, found 260.1777.

General Experimental Procedure

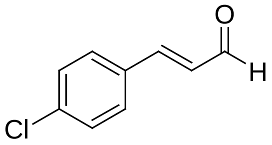
General Procedure 1:

The reactions were performed in deionised water unless otherwise indicated. No special precautions were taken to exclude oxygen and standard vials were used. To 0.15 mmol of the acetal were added 1 mL of deionised water and 80 mg of Amberlite® IR120 hydrogen form. The vial was stirred at 700 rpm and heated to 60-70 °C for 12 – 18 hr. After that a filtration was performed to remove the Amberlite®, 1 mL of water was used to rinse the flask. Alternatively, if the product is a solid, ethyl acetate 1-2 mL could be used to dissolve and extract the product from the aqueous layer. Treatment with activated charcoal was performed at 40 °C during 3 hours to remove color impurities, filtration and evaporation of the solvent gives the product. When necessary, the crude product was purified by chromatography using ethyl acetate and hexane as the eluent.

General Procedure 2:

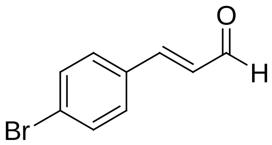
In a 1.5 mL vial equipped with a stirring bar, 0.15 mmol of the acetal were added to 1 mL of 25% H_3PO_4. Then the vial was closed and heated up to 60-70 °C for 12 – 18 hr. When the reaction was completed, the reaction mixture was cooled to room temperature. The pure product was isolated after a simple filtration. When necessary the crude product was purified by chromatography using ethyl acetate and hexane as the eluent.

(2*E*)-3-(4-Chlorophenyl)-2-propenal (7a).



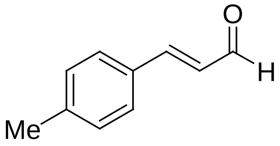
The parent compound was prepared using the general procedure 2. This compound is known and matches the reported spectroscopic data[2]. Yield 95%, white solid; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 9.70 (d, *J* = 7.6 Hz, 1H), 7.50 (d, *J* = 8.5 Hz, 2H), 7.43 (d, *J* = 15.6 Hz, 1H), 7.41 (d, *J* = 8.4 Hz, 2H), 6.69 (dd, *J* = 16.0, 7.6 Hz, 2H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 193.3, 151.0, 137.3, 132.5, 129.6, 129.4, 129.0.

(2*E*)-3-(4-Bromophenyl)-2-propenal (7b).



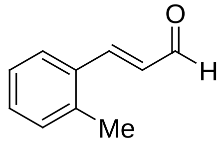
The parent compound was prepared using the general procedure 2. This compound is known and matches the reported spectroscopic data[3,4]. Yield 93%, white solid; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 9.71 (d, *J* = 7.6 Hz, 2H), 7.57 (d, *J* = 8.5 Hz, 4H), 7.43 (d, *J* = 8.5 Hz, 5H), 7.42 (d, *J* = 15.6 Hz, 1H), 6.70 (dd, *J* = 16.0, 7.6 Hz, 2H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 193.3, 151.1, 132.9, 132.4, 129.8, 129.0, 125.7.

(2*E*)-3-(4-Methylphenyl)-2-propenal (7c).



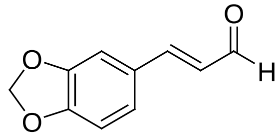
The parent compound was prepared using the general procedure 2. This compound is known and matches the reported spectroscopic data[5,6]. Yield 94%, yellow solid; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 9.69 (d, *J* = 7.7 Hz, 2H), 7.47 (d, *J* = 8.1 Hz, 5H), 7.46 (d, *J* = 15.7 Hz, 1H), 7.24 (d, *J* = 8.0 Hz, 4H), 6.69 (dd, *J* = 15.9, 7.7 Hz, 2H), 2.40 (s, 6H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 193.8, 152.9, 142.0, 131.3, 129.8, 128.5, 127.7, 21.6.

(2*E*)-3-(2-Methylphenyl)-2-propenal (7d).



The parent compound was prepared using the general procedure 2. This compound is known and matches the reported spectroscopic data[3,5]. Yield 83%, yellow solid; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 9.73 (d, *J* = 7.7 Hz, 1H), 7.78 (d, *J* = 15.8 Hz, 1H), 7.59 (d, *J* = 7.7 Hz, 1H), 7.35 – 7.31 (m, 1H), 7.27 – 7.24 (m, 1H), 6.67 (dd, *J* = 15.8, 7.7 Hz, 1H), 2.48 (s, 3H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 193.9, 150.3, 137.9, 132.9, 131.1, 131.0, 129.6, 126.9, 126.6, 19.8.

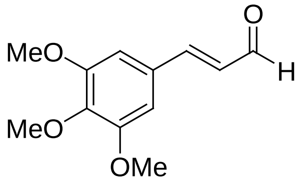
(2*E*)-3-(1,3-benzodioxol-5-yl)-2-propenal (7e).



The parent compound was prepared using the general procedure 2. This compound is known and matches the reported spectroscopic data[7,8].

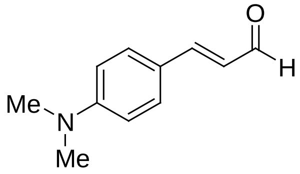
Yield 95%, beige solid; ^{1}H-NMR [CDCl$_3$, 600 MHz] δ: 9.59 (d, *J* = 7.7 Hz, 1H), 7.32 (d, *J* = 15.8 Hz, 1H), 7.04 – 6.95 (m, 2H), 6.82 – 6.79 (m, 1H), 6.50 (dd, *J* = 15.8, 7.7 Hz, 1H), 5.99 (s, 2H); ^{13}C-NMR [CDCl$_3$, 150 MHz] δ: 193.6, 152.7, 150.6, 148.7, 128.6, 127.0, 125.4, 108.9, 106.9, 102.0.

(2*E*)-3-(3,4,5-trimethoxyphenyl)-2-propenal (7f).



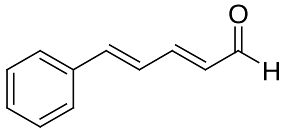
The parent compound was prepared using the general procedure 2. This compound is known and matches the reported spectroscopic data[9,10]. Yield 93%, translucent liquid; ^{1}H-NMR [CDCl$_3$, 600 MHz] δ: 9.68 (d, *J* = 7.7 Hz, 1H), 7.40 (d, *J* = 15.8 Hz, 1H), 6.79 (s, 2H), 6.64 (dd, *J* = 15.8, 7.7 Hz, 1H), 3.90 (s, 9H); ^{13}C-NMR [CDCl$_3$, 150 MHz] δ: 193.4, 153.6, 152.7, 129.5, 128.0, 105.7, 61.0, 56.2.

(2*E*)-3-(4-(dimethylamino)phenyl)-2-propenal (7g).



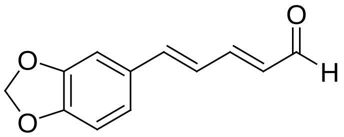
The parent compound was prepared using the general procedure 1. This compound is known and matches the reported spectroscopic data[11]. Yield 88%, yellow oil; ^{1}H-NMR [CDCl$_3$, 600 MHz] δ: 9.59 (d, *J* = 7.9 Hz, 1H), 7.45 (d, *J* = 8.9 Hz, 2H), 7.37 (d, *J* = 15.6 Hz, 1H), 6.68 (d, *J* = 8.9 Hz, 2H), 6.54 (dd, *J* = 15.6, 7.9 Hz, 1H), 3.05 (s, 6H); ^{13}C-NMR [CDCl$_3$, 150 MHz] δ: 193.67, 153.9, 152.4, 130.5, 123.8, 121.7, 111.7, 40.0.

(2*E*,4*E*)-5-Phenyl-2,4-pentadienal (7h).



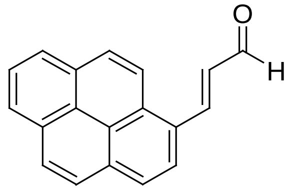
The parent compound was prepared using the general procedure 1. This compound is known and matches the reported spectroscopic data[4,5]. Yield 68%, yellow oil; ^{1}H-NMR [CDCl$_3$, 600 MHz] δ: 9.56 (d, *J* = 7.9 Hz, 1H), 7.45 – 7.42 (m, 2H), 7.34 – 7.27 (m, 3H), 7.23 – 7.19 (m, 1H), 6.96 – 6.93 (m, 2H), 6.21 (dd, *J* = 15.2, 7.9 Hz, 1H); ^{13}C-NMR [CDCl$_3$, 150 MHz] δ: 193.6, 152.0, 142.4, 135.6, 131.7, 129.7, 129.0, 127.5, 126.2.

(2*E*,4*E*)-5-(1,3-Benzodioxol-5-yl)-2,4-pentadienal (7i).



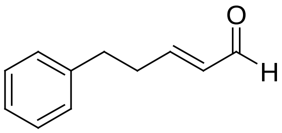
The parent compound was prepared using the general procedure 1. This compound is known and matches the reported spectroscopic data[12]. Yield 76%, yellow oil; ^{1}H-NMR [CDCl$_3$, 600 MHz] δ: 9.60 (d, *J* = 8.0 Hz, 1H), 7.23 (dd, *J* = 15.2, 10.8 Hz, 1H), 7.03 (d, *J* = 1.6 Hz, 1H), 6.97 (dd, *J* = 8.0, 1.6 Hz, 1H), 6.93 (d, *J* = 15.5 Hz, 1H), 6.85 (d, *J* = 10.7 Hz, 1H), 6.81 (d, *J* = 8.0 Hz, 1H), 6.23 (dd, *J* = 15.2, 7.9 Hz, 1H), 6.01 (s, 2H); ^{13}C-NMR [CDCl$_3$, 150 MHz] δ: 193.5, 152.3, 149.1, 148.4, 142.2, 130.9, 130.2, 124.5, 123.6, 108.6, 106.1, 101.5.

(2*E*)-3-(1-Pyrenyl)-2-propenal (7j).



The parent compound was prepared using the general procedure 1. This compound is known and matches the reported spectroscopic data[13]. Yield 81%, yellow solid; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 9.95 (d, *J* = 7.6 Hz, 1H), 8.65 (d, *J* = 15.7 Hz, 1H), 8.50 (d, *J* = 9.3 Hz, 1H), 8.33 (d, *J* = 8.1 Hz, 1H), 8.27 (d, *J* = 7.1 Hz, 2H), 8.25 (d, *J* = 9.2 Hz, 1H), 8.21 (d, *J* = 8.1 Hz, 1H), 8.18 (d, *J* = 8.8 Hz, 1H), 8.10 (d, *J* = 8.8 Hz, 1H), 8.07 (t, *J* = 7.6 Hz, 1H), 7.04 (dd, *J* = 15.6, 7.6 Hz, 1H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 192.4, 148.1, 146.8, 130.8, 129.3, 128.2, 127.8, 127.6, 126.4, 126.2, 125.5, 125.2, 125.1, 125.0, 124.2, 123.5, 123.3, 122.5, 120.8.

(2*E*)-5-Phenyl-2-pentenal (7m).



The parent compound was prepared using the general procedure 1. This compound is known and matches the reported spectroscopic data[14,15]. Dichloromethane–hexane (1 : 1); Yield 82%, Yellow oil; ^{1}H-NMR [$CDCl_3$, 600 MHz] δ: 9.50 (d, *J* = 7.9 Hz, 1H), 7.32 – 7.29 (m, 2H), 7.22 – 7.18 (m, 3H), 6.86 (dt, *J* = 15.6, 6.7 Hz, 1H), 6.14 (ddt, *J* = 15.6, 7.8, 1.4 Hz, 1H), 2.84 (t, *J* = 7.6 Hz, 2H), 2.70 – 2.65 (m, 2H); ^{13}C-NMR [$CDCl_3$, 150 MHz] δ: 194.0, 157.3, 133.4, 129.4, 128.6, 128.3, 126.4, 34.2, 34.1.

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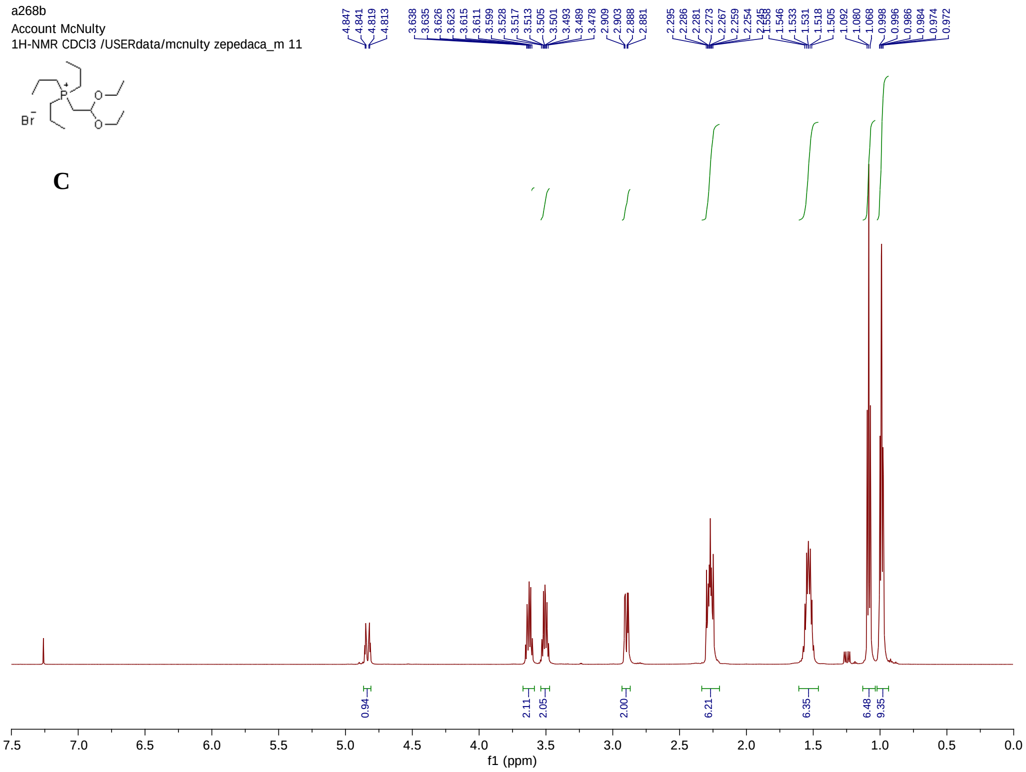

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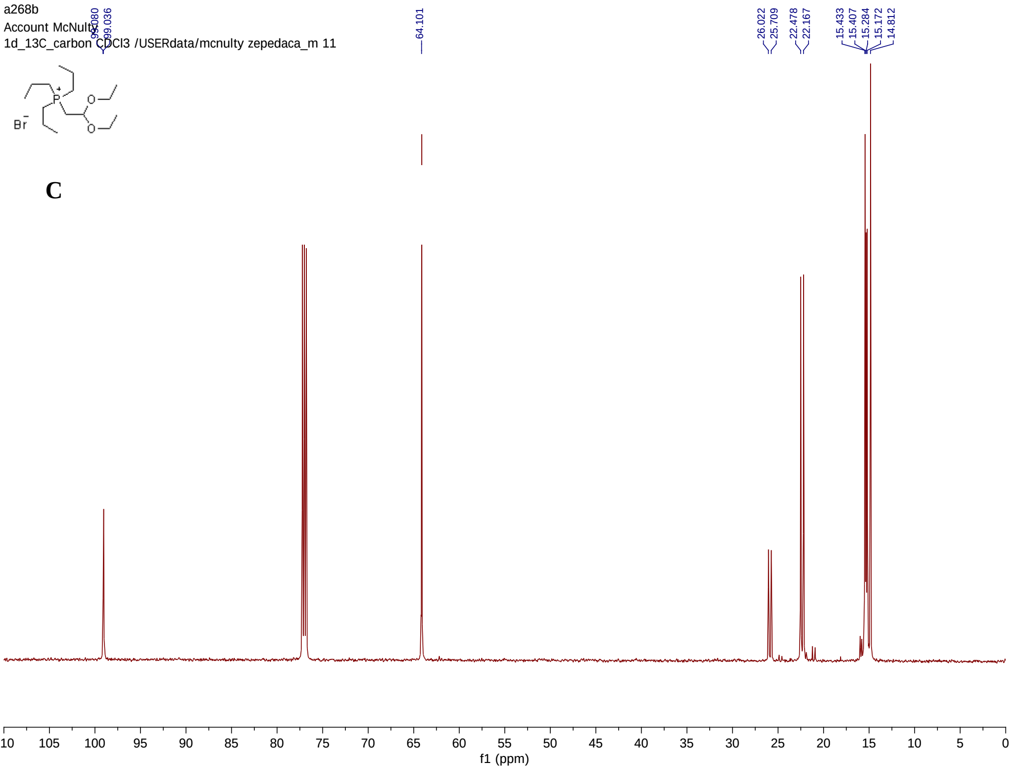

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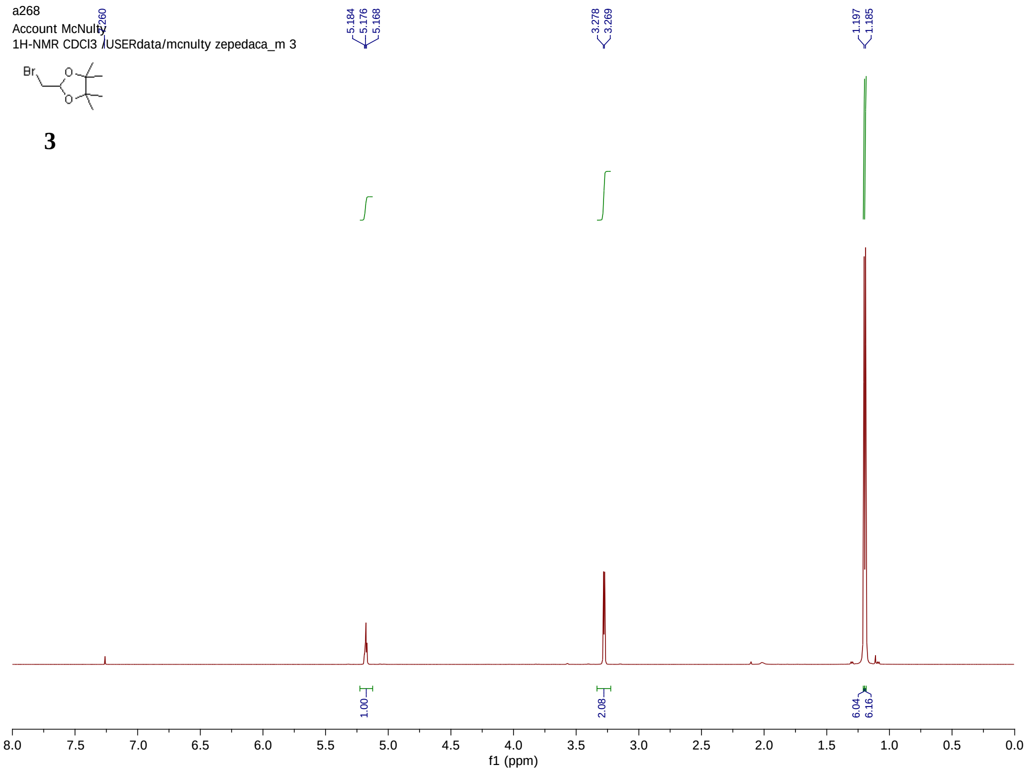
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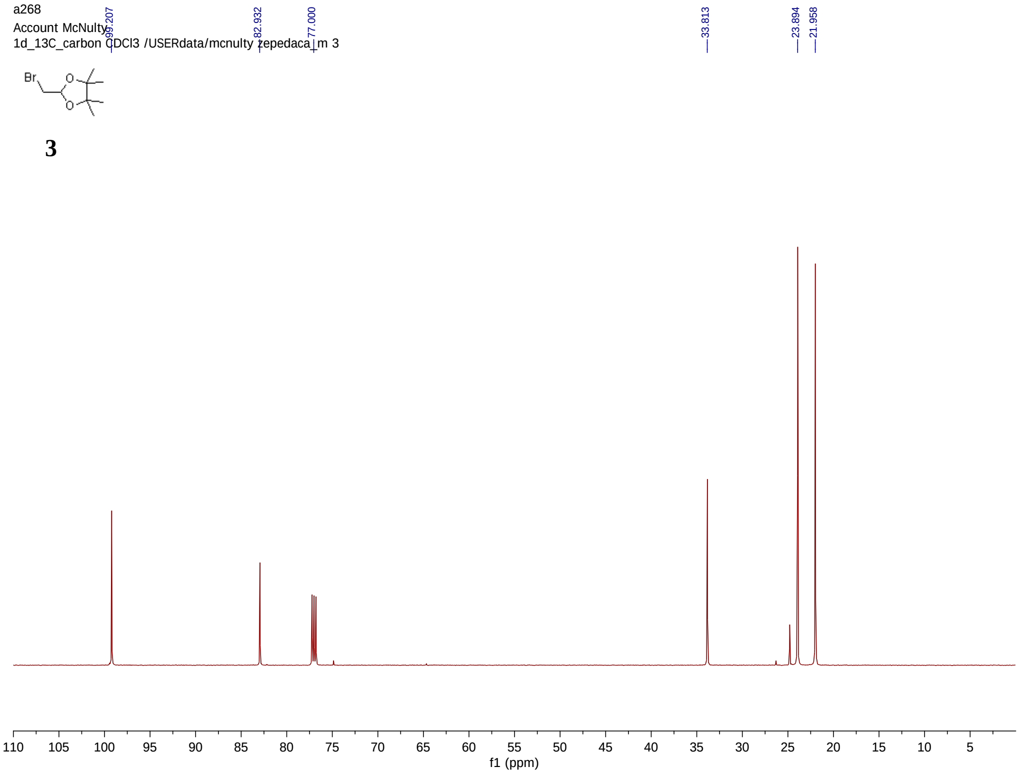
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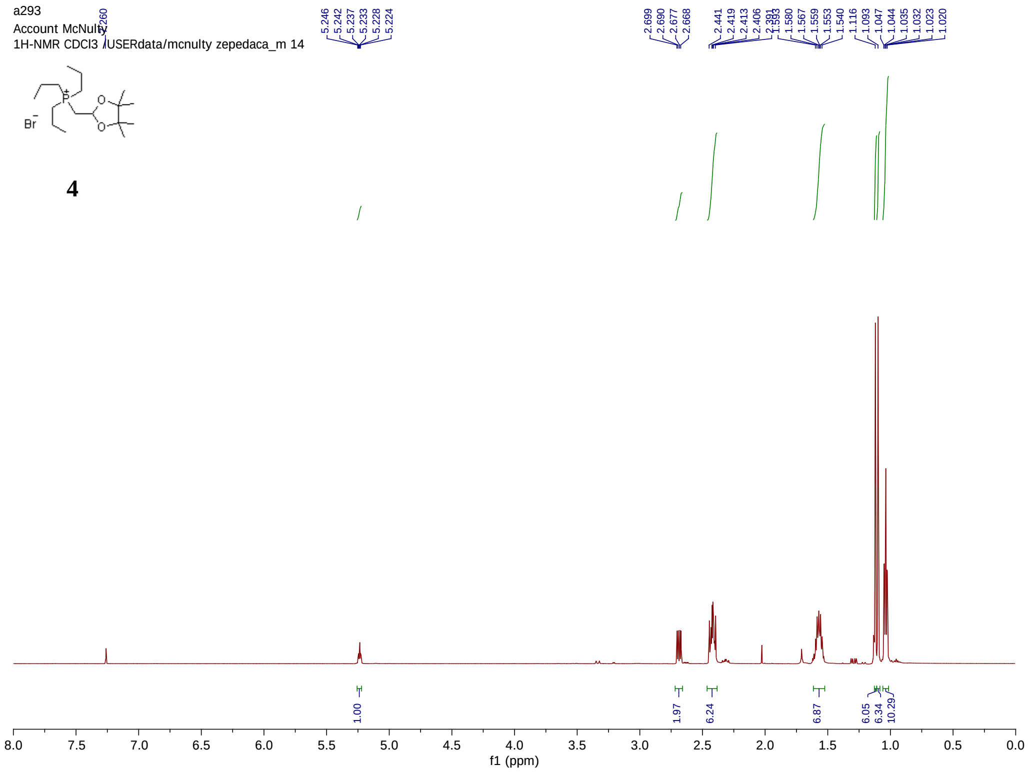
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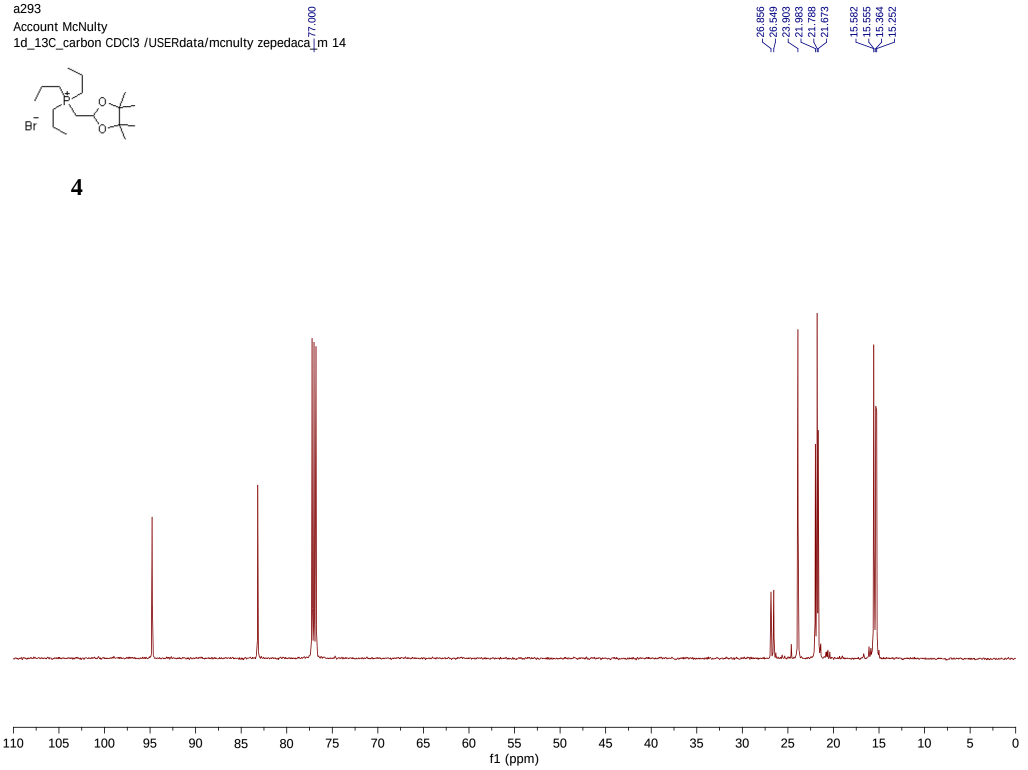
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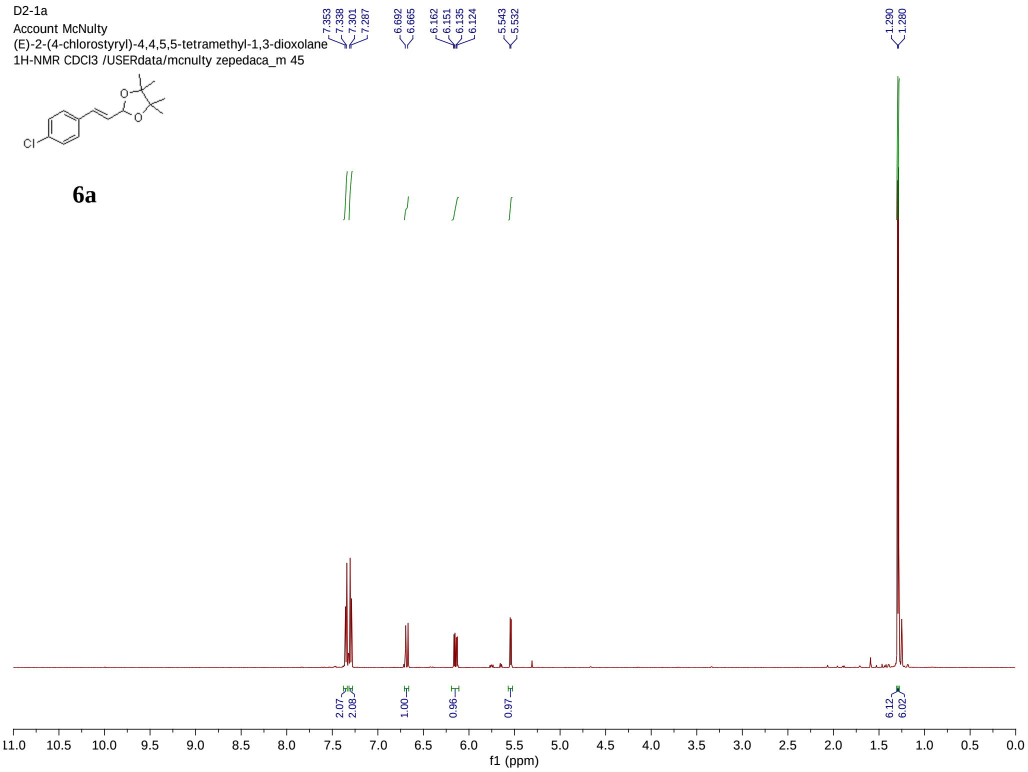
4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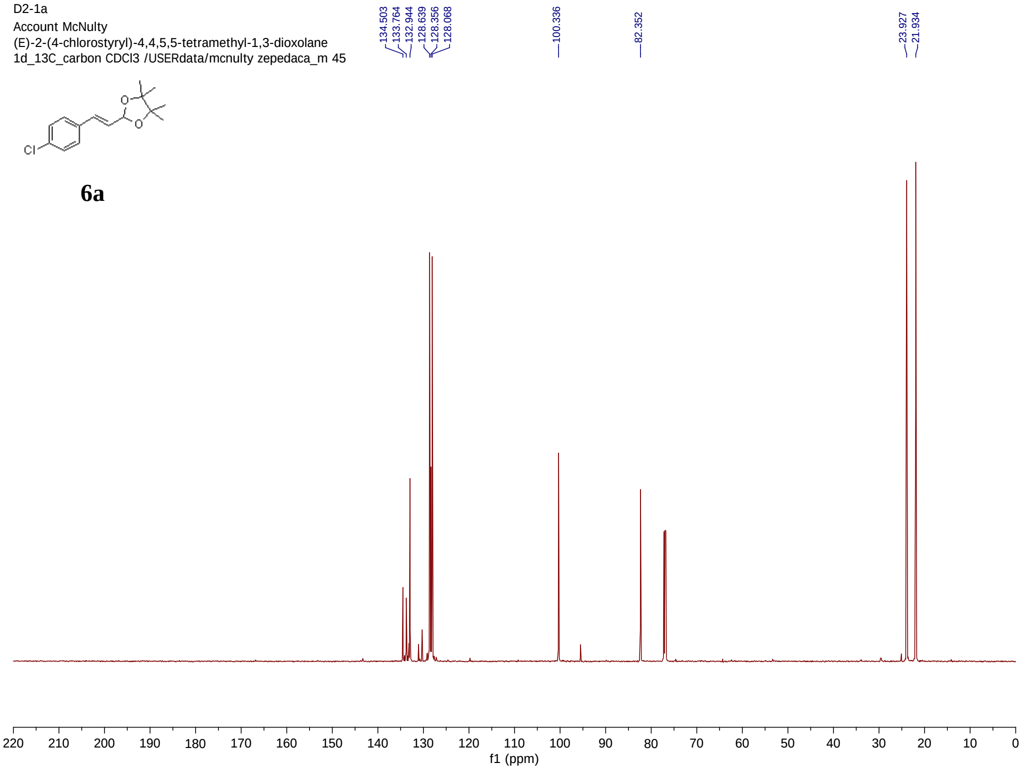
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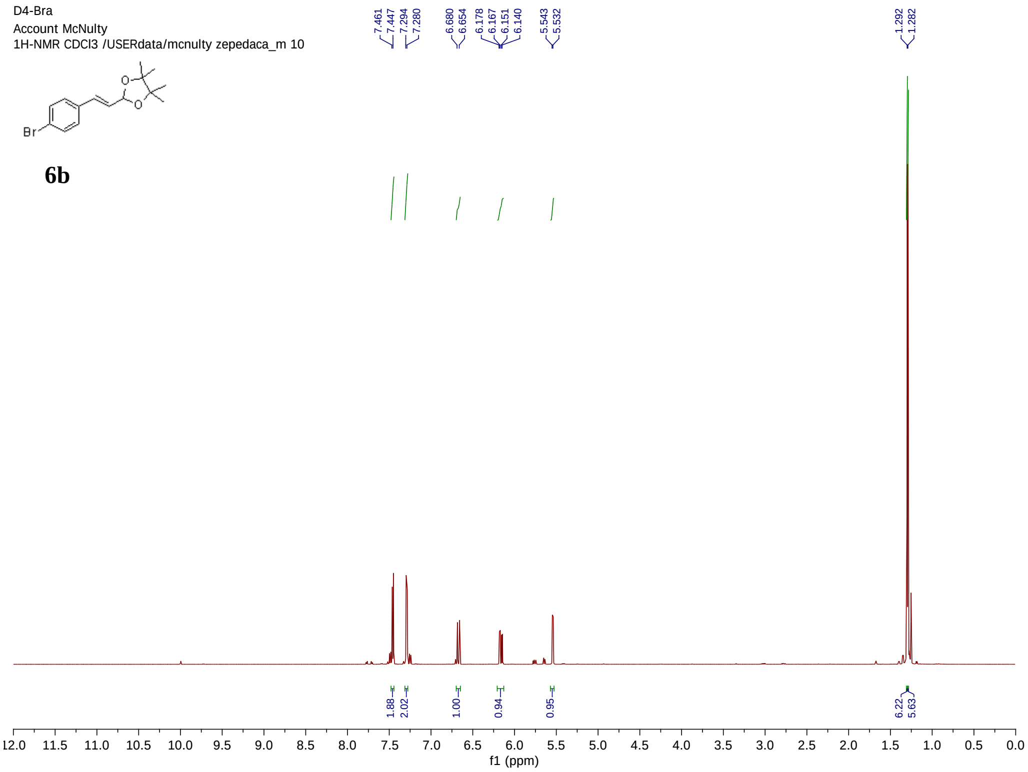
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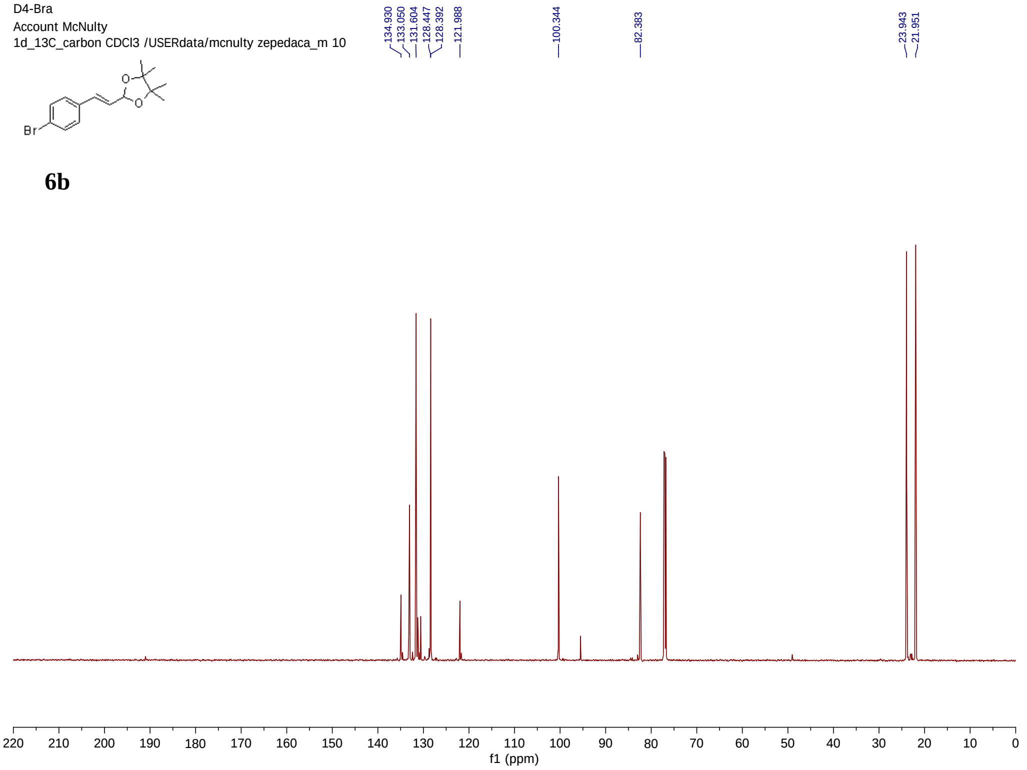
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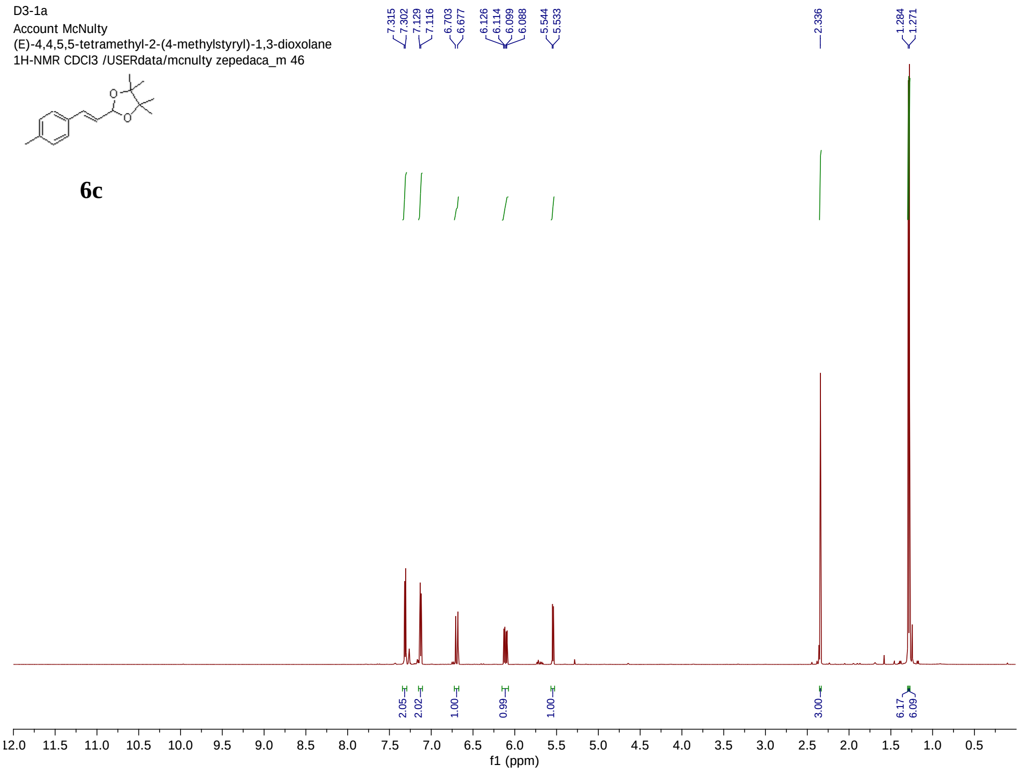
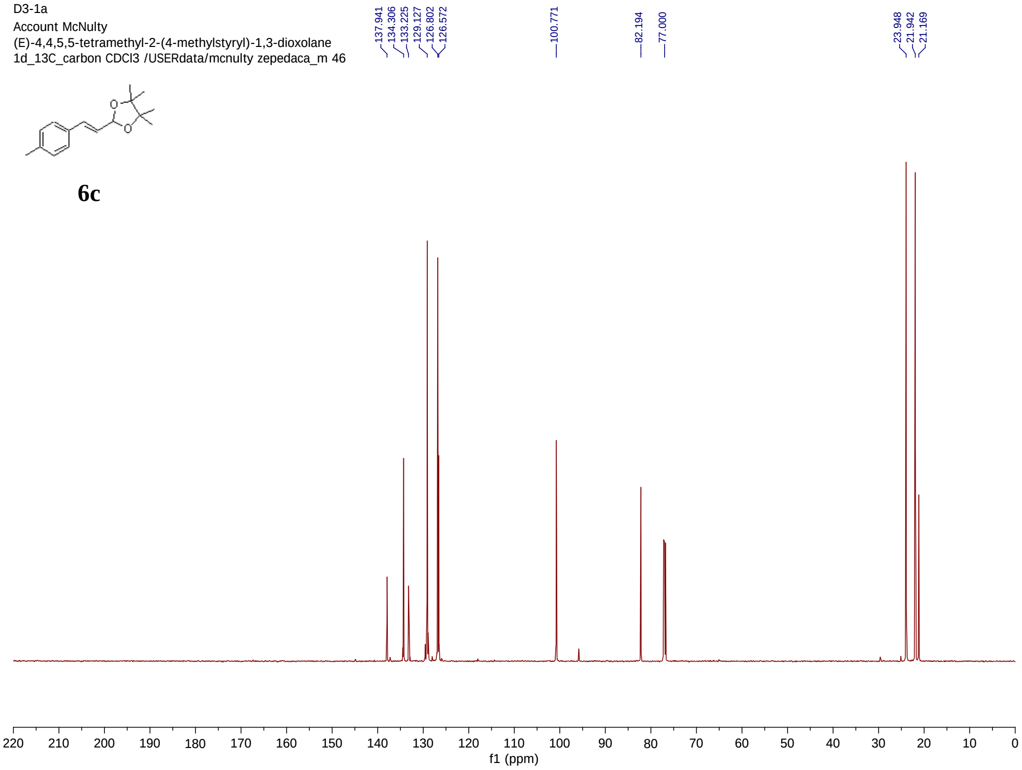
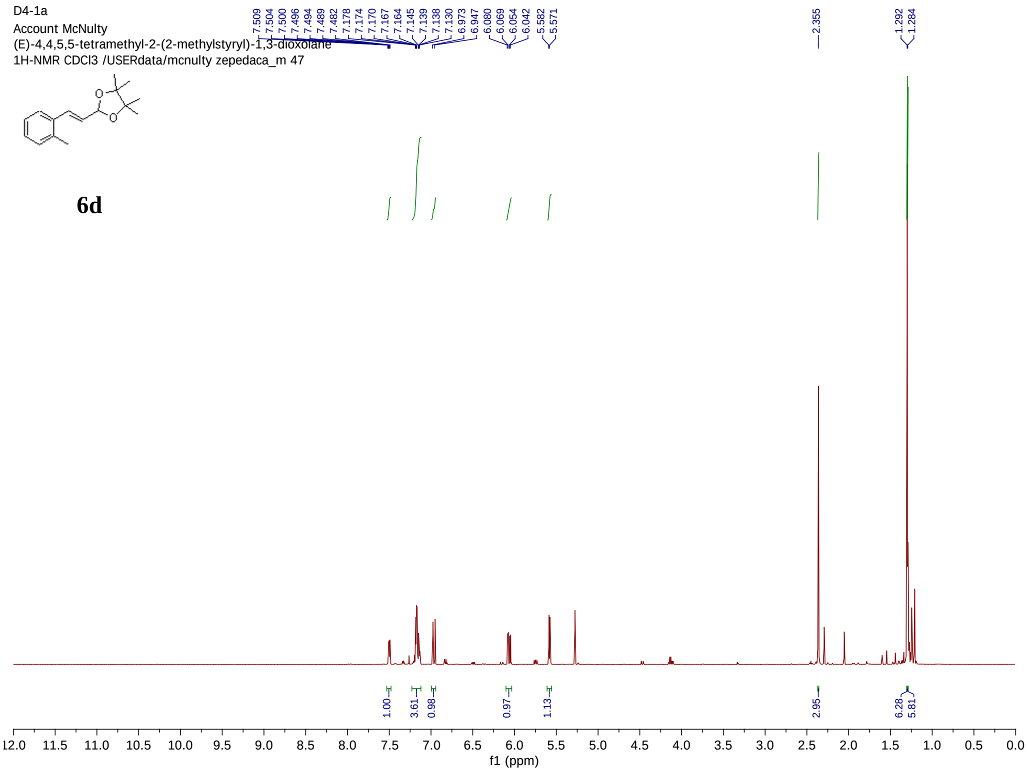
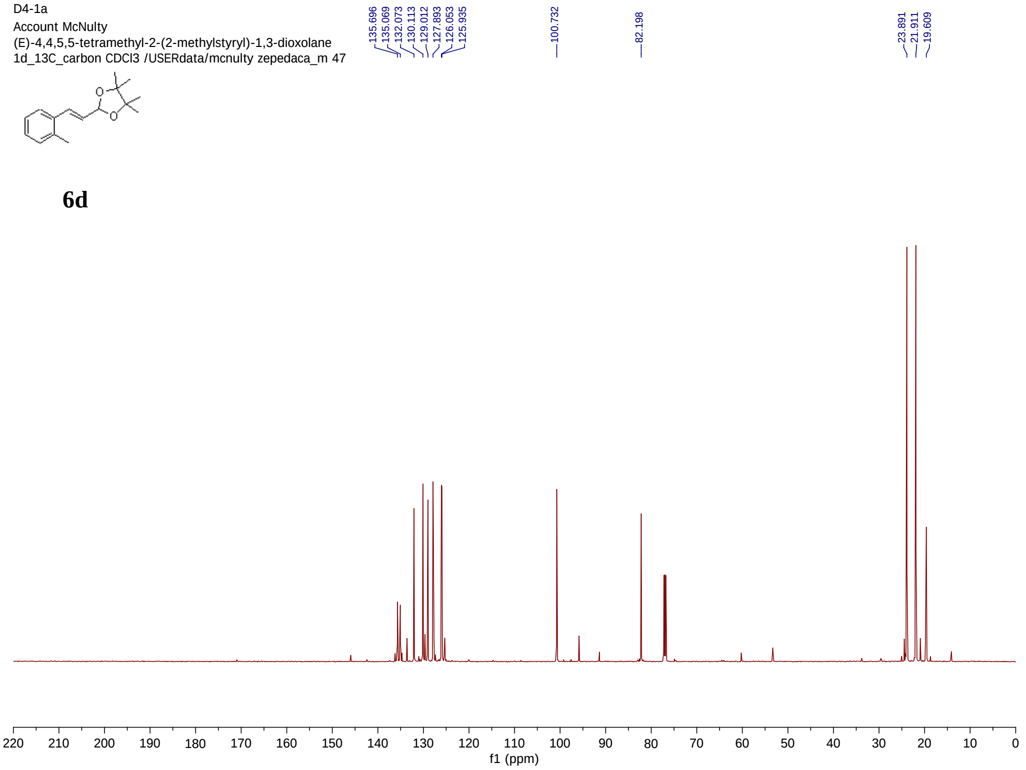
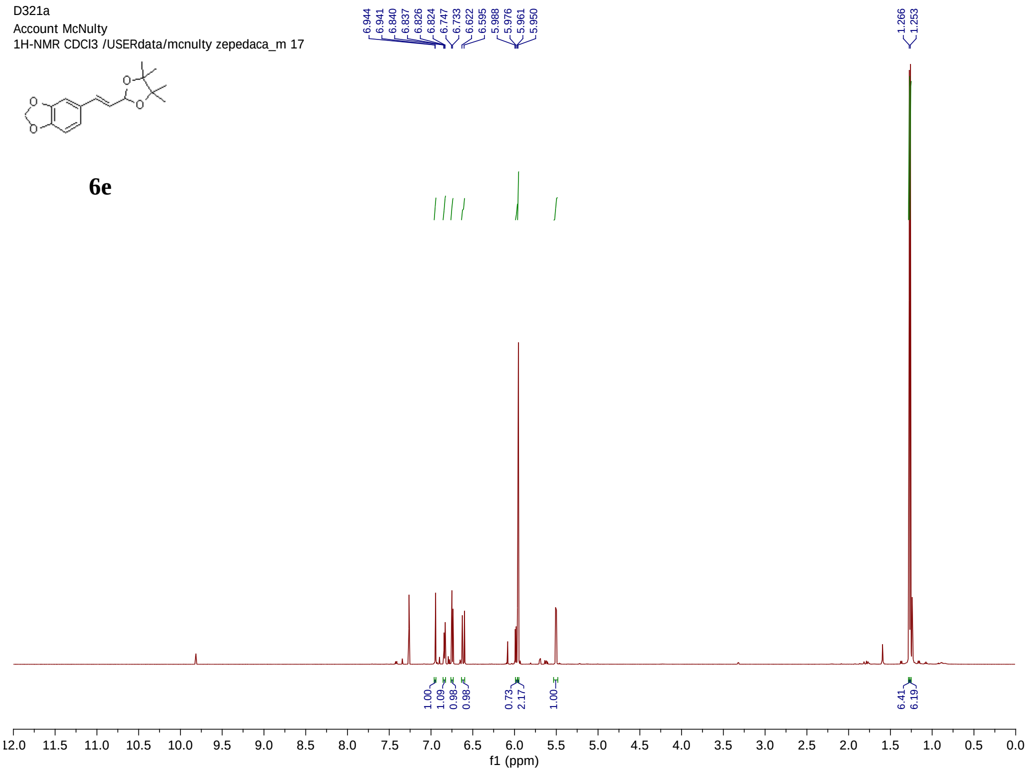
f1 (ppm)

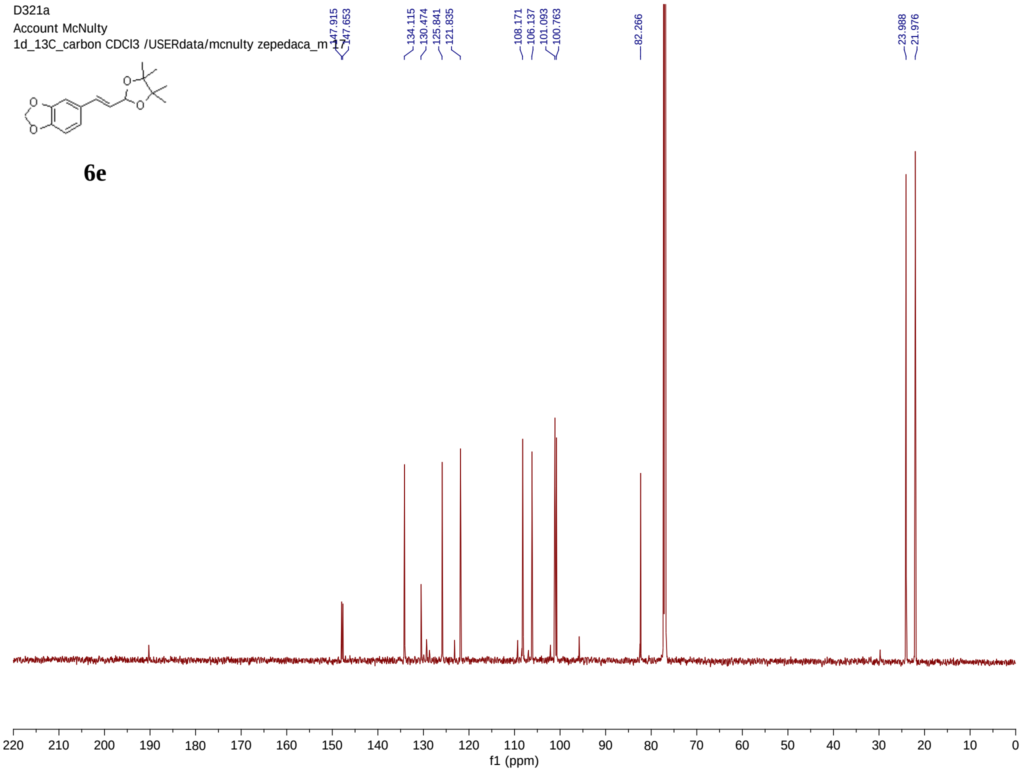

D3-1a
Account McNulty
(E)-4,4,5,5-tetramethyl-2-(4-methylstyryl)-1,3-dioxolane
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 46
6c
7.315
7.302
7.129
7.116
6.703
6.677
6.126
6.114
6.099
6.088
5.544
5.533
2.336
1.284
1.271
2.05
2.02
1.00
0.99
1.00
3.00
6.17
6.09
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

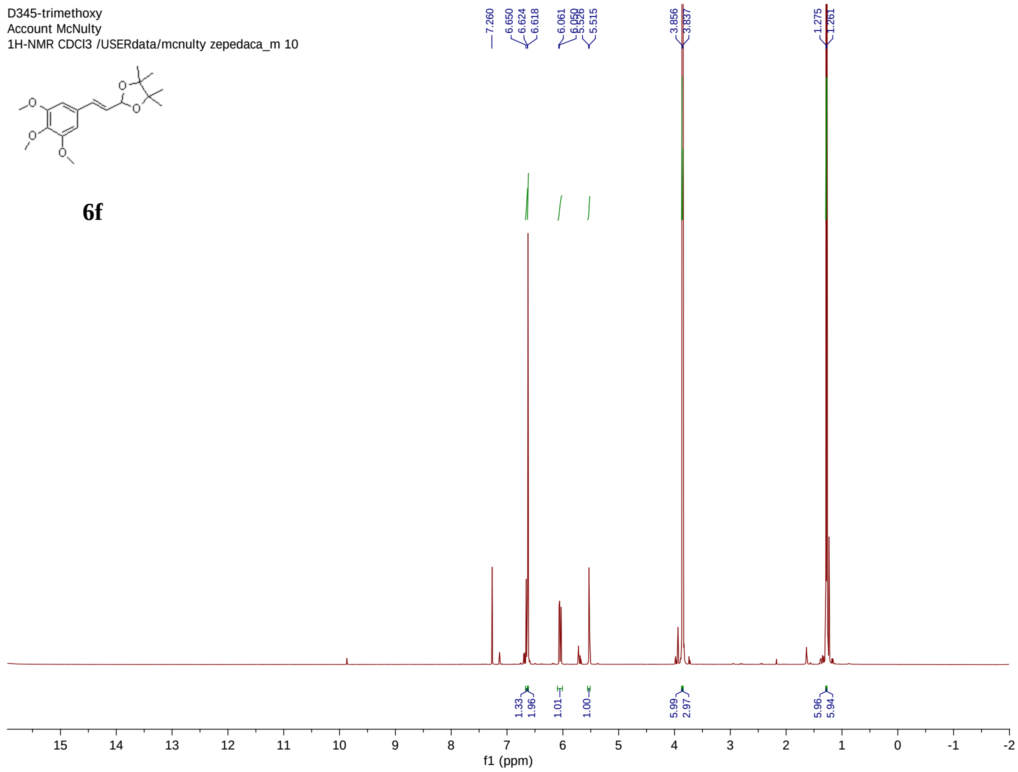

D3-1a
Account McNulty
(E)-4,4,5,5-tetramethyl-2-(4-methylstyryl)-1,3-dioxolane
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 46
6c
137.941
134.306
133.225
129.127
126.802
126.572
100.771
82.194
77.000
23.948
21.942
21.169
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

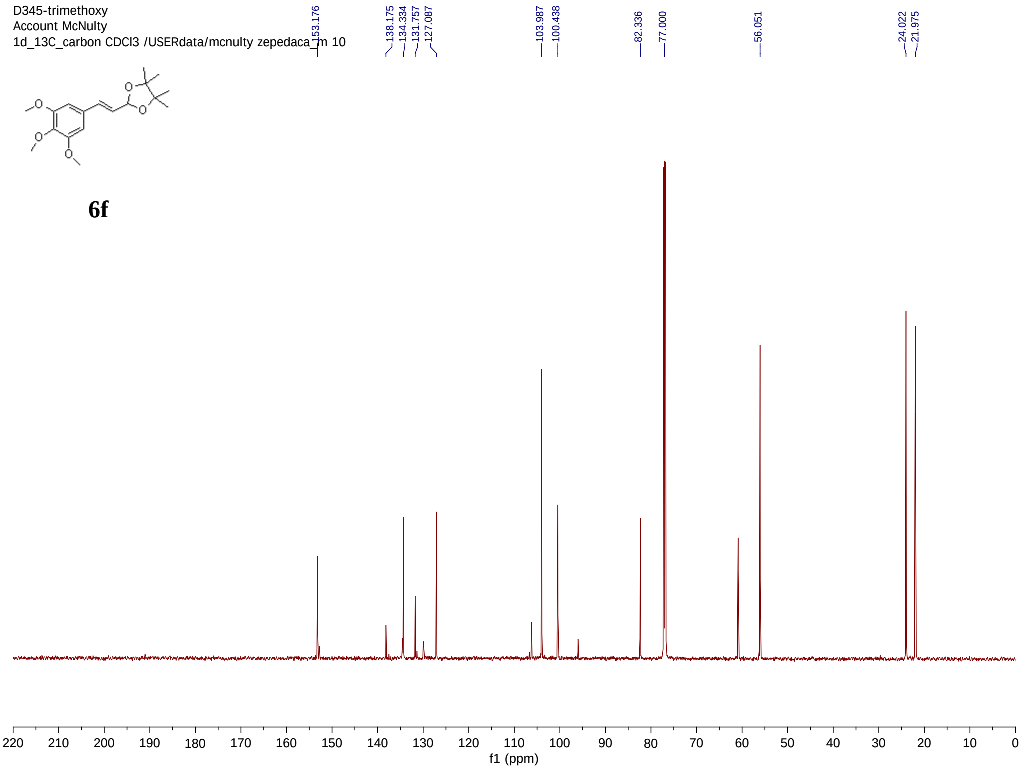

D4-1a
Account McNulty
(E)-4,4,5,5-tetramethyl-2-(2-methylstyryl)-1,3-dioxolane
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 47
6d
7.509
7.504
7.500
7.496
7.494
7.489
7.482
7.178
7.174
7.170
7.167
7.164
7.145
7.139
7.138
7.130
6.973
6.947
6.080
6.069
6.054
6.042
5.582
5.571
2.355
1.292
1.284
1.00
3.61
0.98
0.97
1.13
2.95
6.28
5.81
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

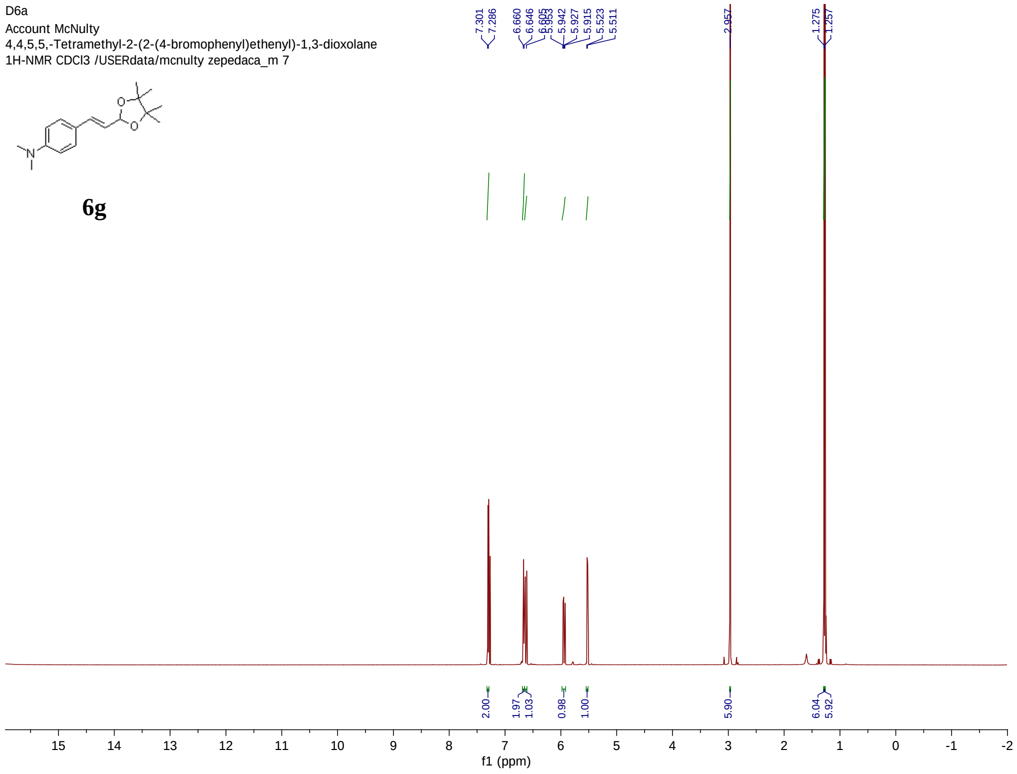

D4-1a
Account McNulty
(E)-4,4,5,5-tetramethyl-2-(2-methylstyryl)-1,3-dioxolane
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 47
6d
135.696
135.069
132.073
130.113
129.012
127.893
126.053
125.935
100.732
82.198
23.891
21.911
19.609
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


D321a
Account McNulty
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 17
6e
6.944
6.941
6.840
6.837
6.826
6.824
6.747
6.733
6.622
6.595
5.988
5.976
5.961
5.950
1.266
1.253
1.00
1.09
0.98
0.98
0.73
2.17
1.00
6.41
6.19
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

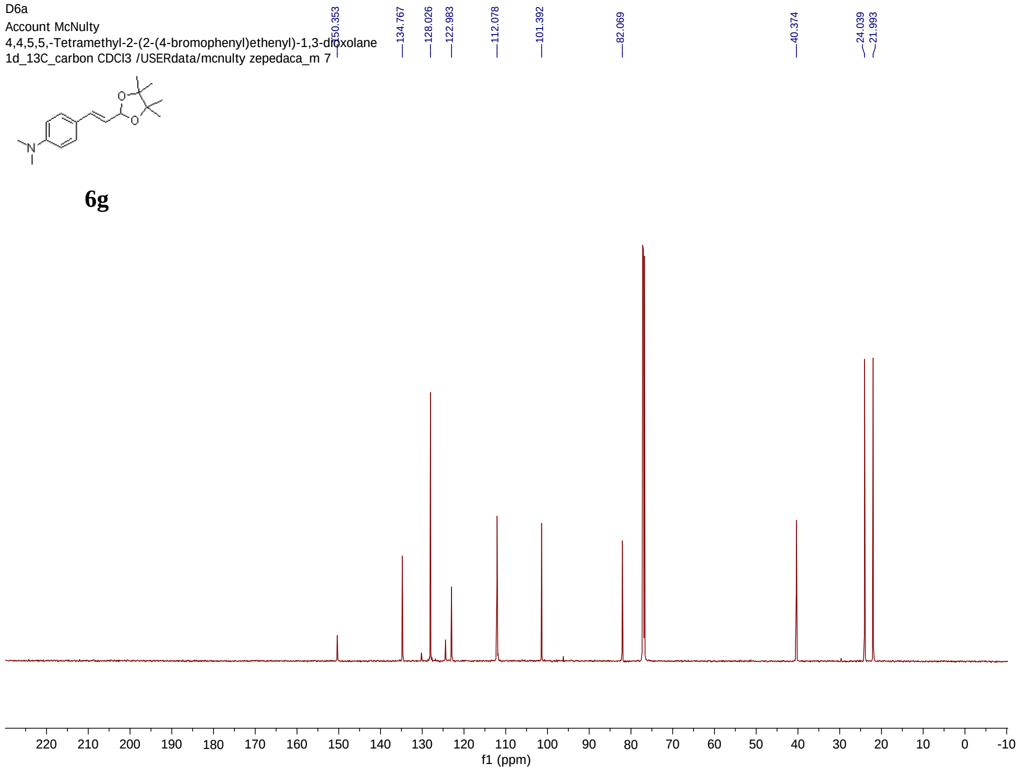

D321a
Account McNulty
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 17
6e
147.915
147.653
134.115
130.474
125.841
121.835
108.171
106.137
101.093
100.763
82.266
23.988
21.976
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


D345-trimethoxy
Account McNulty
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 10
6f
7.260
6.650
6.624
6.618
6.061
6.050
5.526
5.515
3.856
3.837
1.275
1.261
1.33
1.96
1.01
1.00
5.99
2.97
5.96
5.94
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

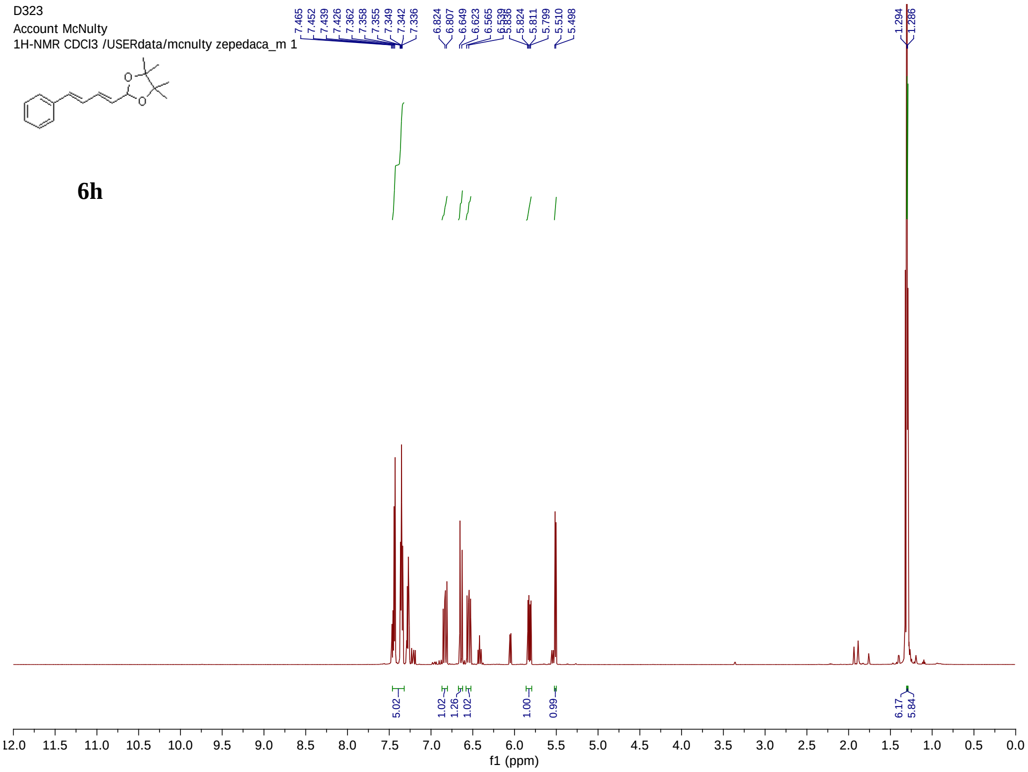

D345-trimethoxy
Account McNulty
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 10
6f
153.176
138.175
134.334
131.757
127.087
103.987
100.438
82.336
77.000
56.051
24.022
21.975
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

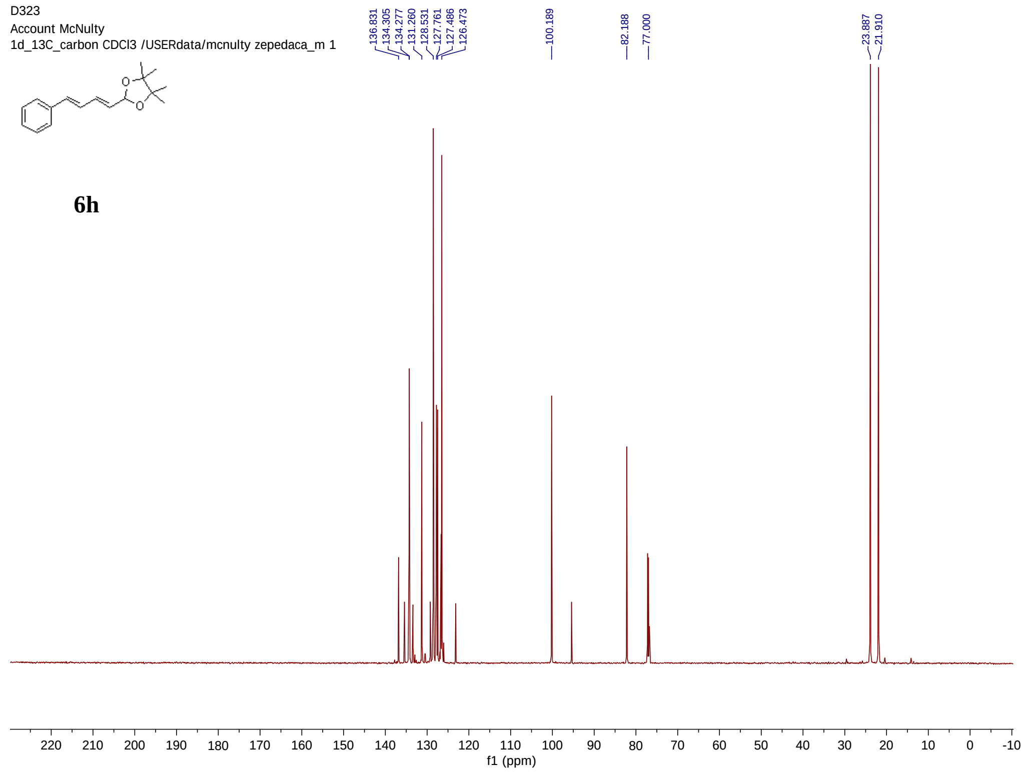

D6a
Account McNulty
4,4,5,5,-Tetramethyl-2-(2-(4-bromophenyl)ethenyl)-1,3-dioxolane
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 7
N
O
O
7.301
7.286
6.660
6.646
6.605
5.953
5.942
5.927
5.915
5.523
5.511
2.957
1.275
1.257
2.00
1.97
1.03
0.98
1.00
5.90
6.04
5.92
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

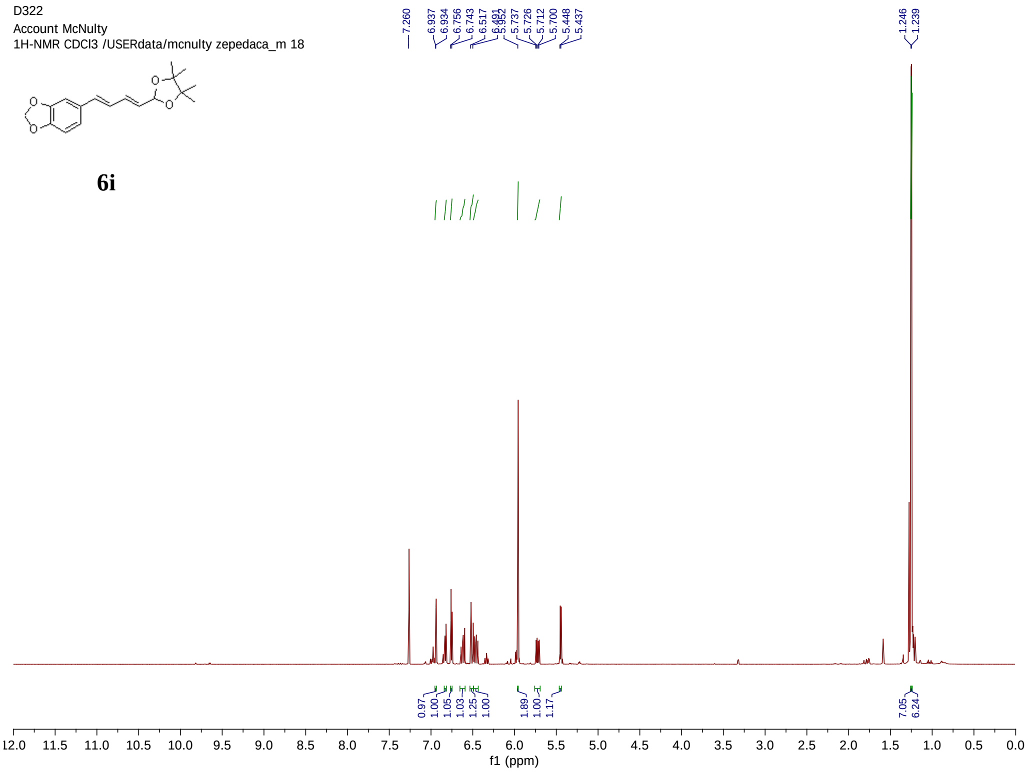
6g

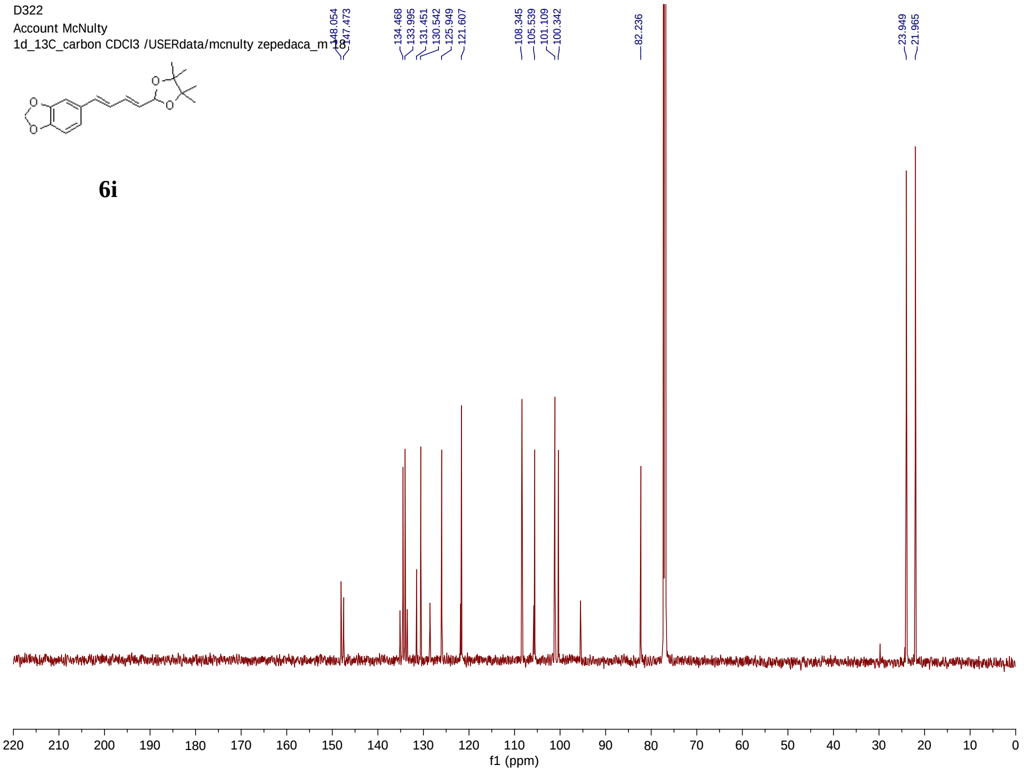

D6a
Account McNulty
4,4,5,5,-Tetramethyl-2-(2-(4-bromophenyl)ethenyl)-1,3-dioxolane
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 7
N
O
O
150.353
134.767
128.026
122.983
112.078
101.392
82.069
40.374
24.039
21.993
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

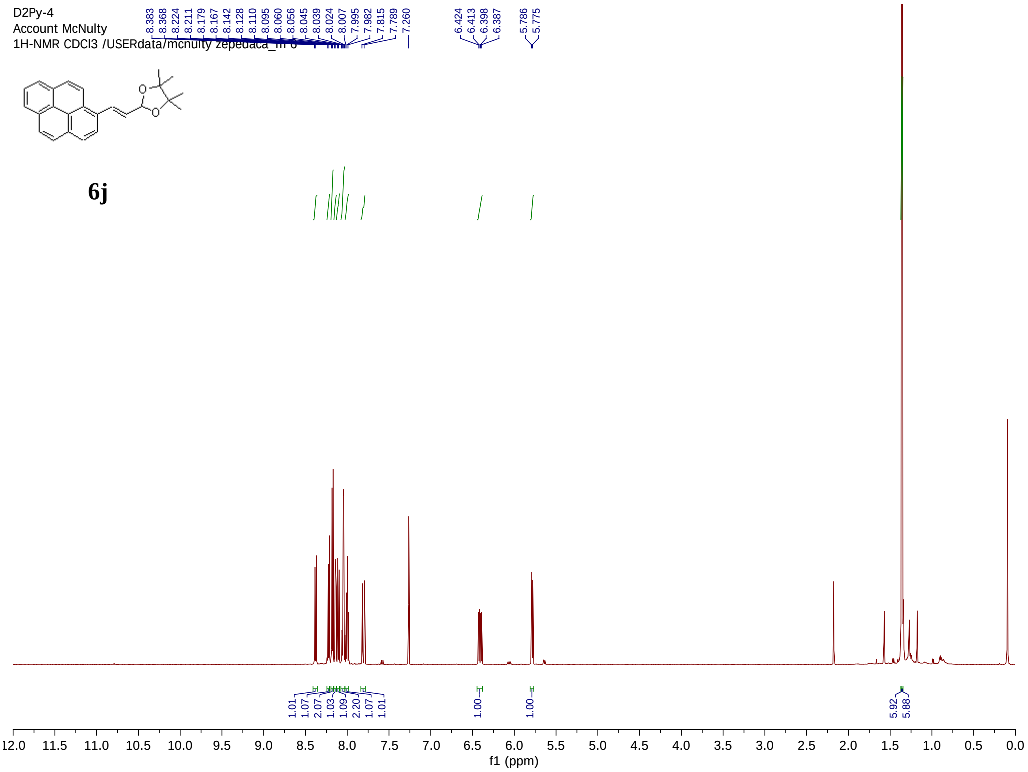
6g

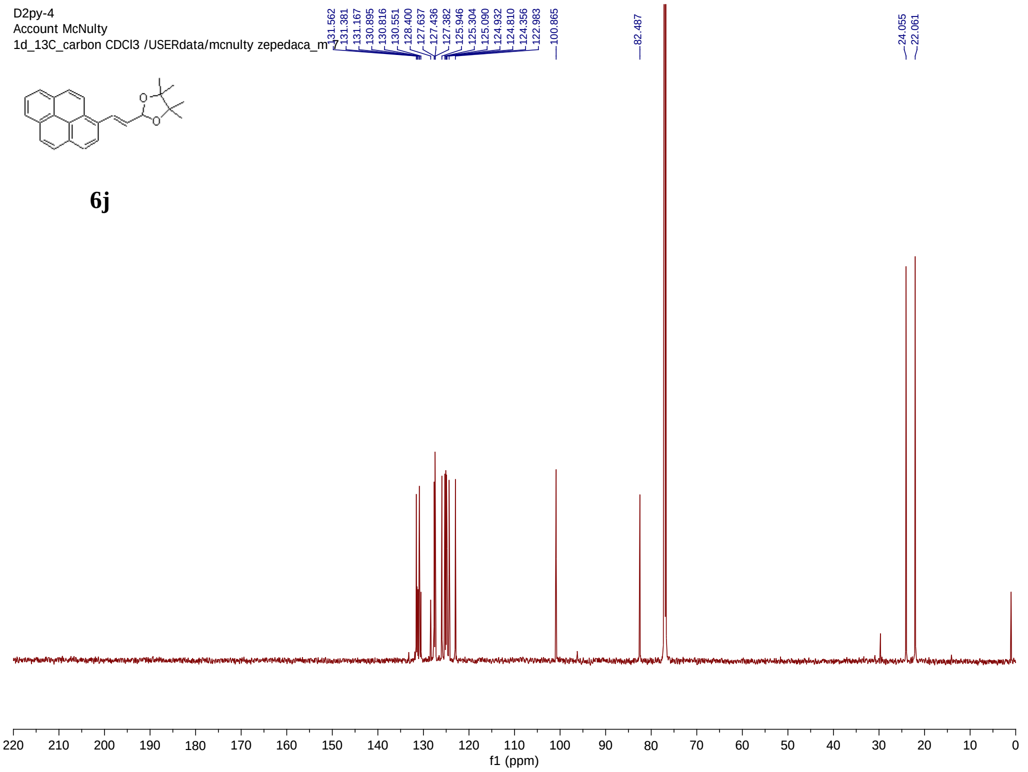

D323
Account McNulty
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 1
6h
7.465
7.452
7.439
7.426
7.362
7.358
7.355
7.349
7.342
7.336
6.824
6.807
6.649
6.623
6.565
6.539
5.836
5.824
5.811
5.799
5.510
5.498
1.294
1.286
5.02
1.02
1.26
1.02
1.00
0.99
6.17
5.84
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

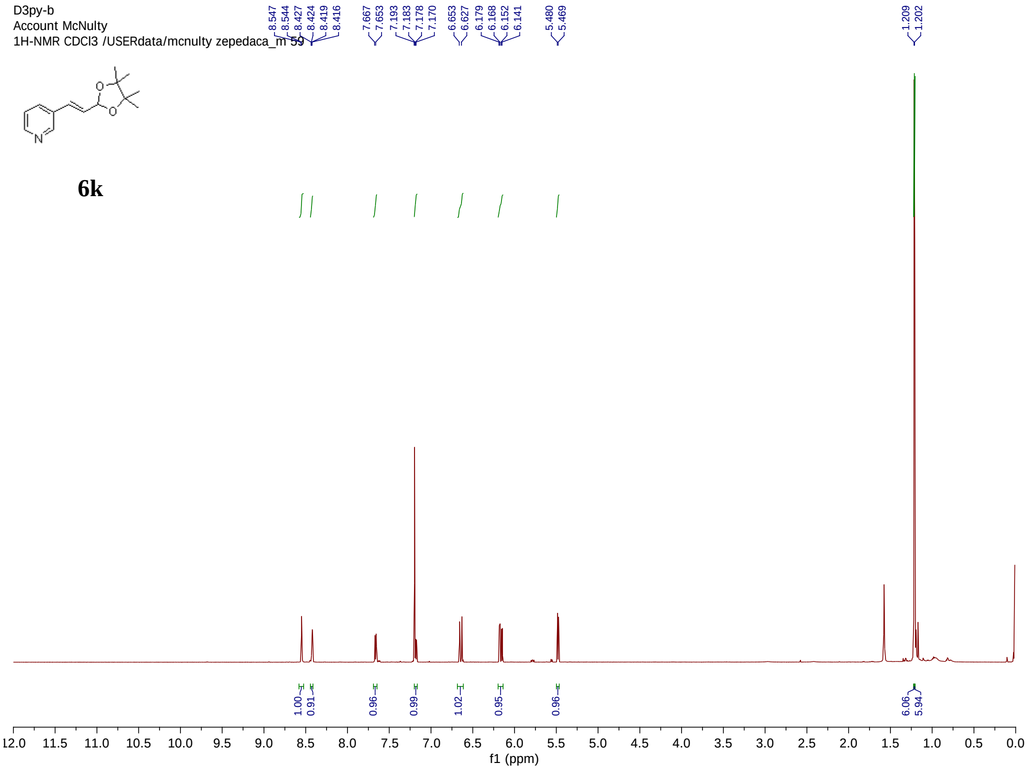

D323
Account McNulty
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 1
6h
136.831
134.305
134.277
131.260
128.531
127.761
127.486
126.473
100.189
82.188
77.000
23.887
21.910
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

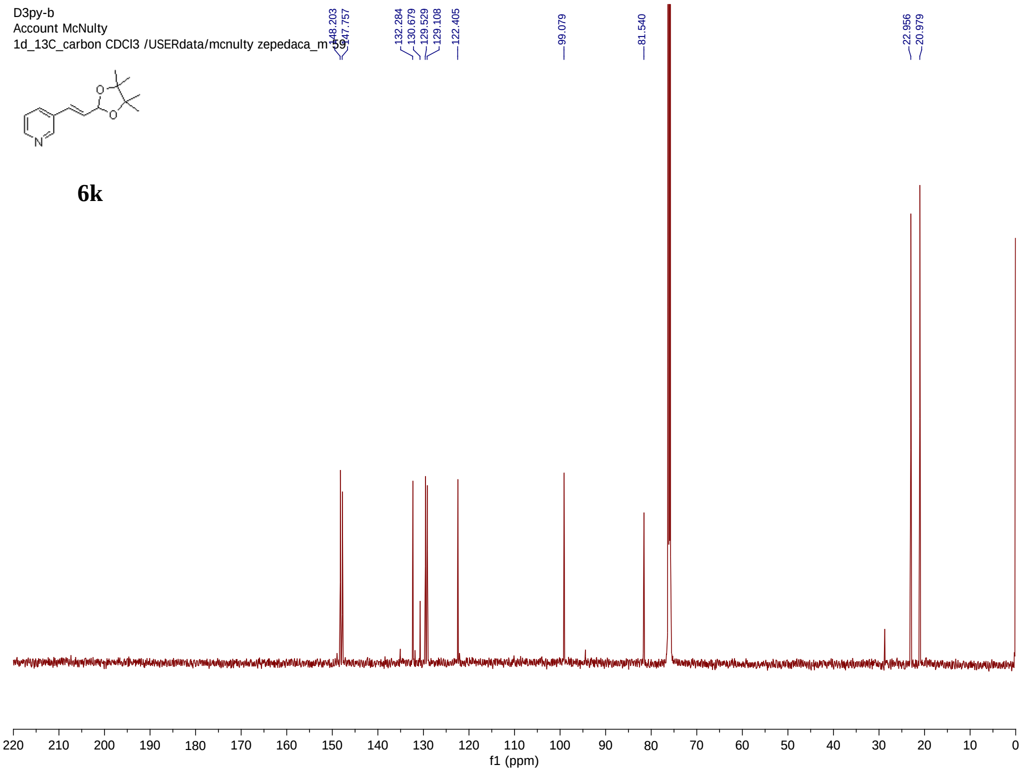

D322
Account McNulty
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 18
O
O
O
O
6i
7.260
6.937
6.934
6.756
6.743
6.517
6.491
5.952
5.737
5.726
5.712
5.700
5.448
5.437
1.246
1.239
0.97
1.00
1.05
1.03
1.25
1.00
1.89
1.00
1.17
7.05
6.24
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

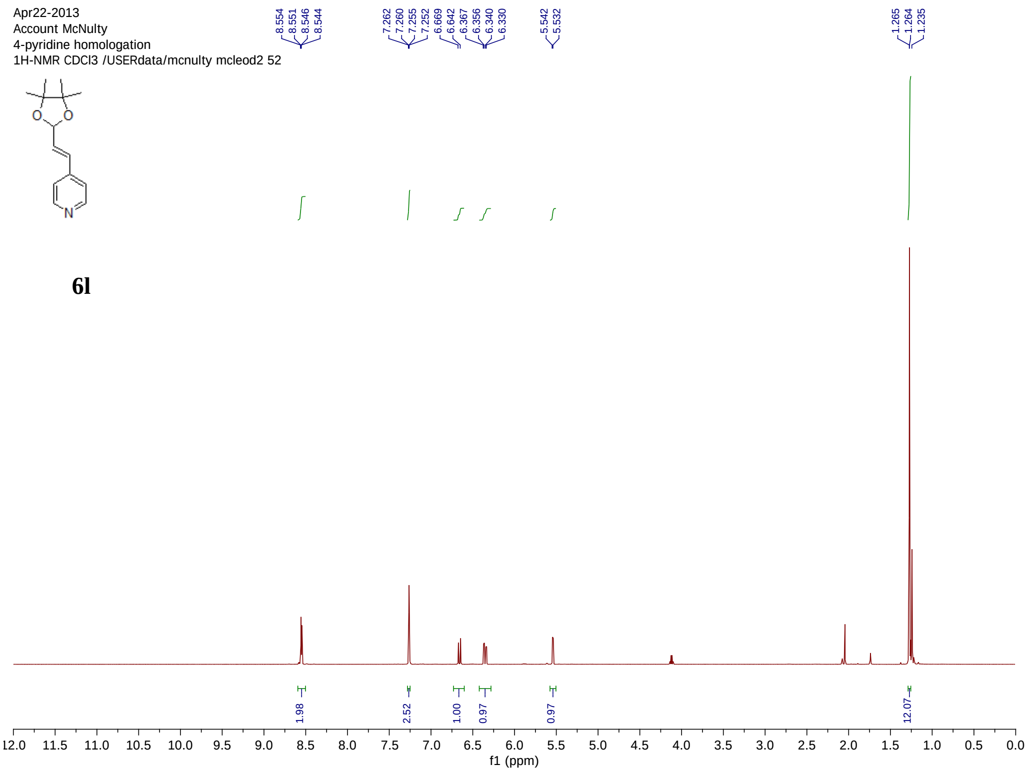

D322
Account McNulty
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 18
O
O
O
O
6i
148.054
147.473
134.468
133.995
131.451
130.542
125.949
121.607
108.345
105.539
101.109
100.342
82.236
23.949
21.965
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

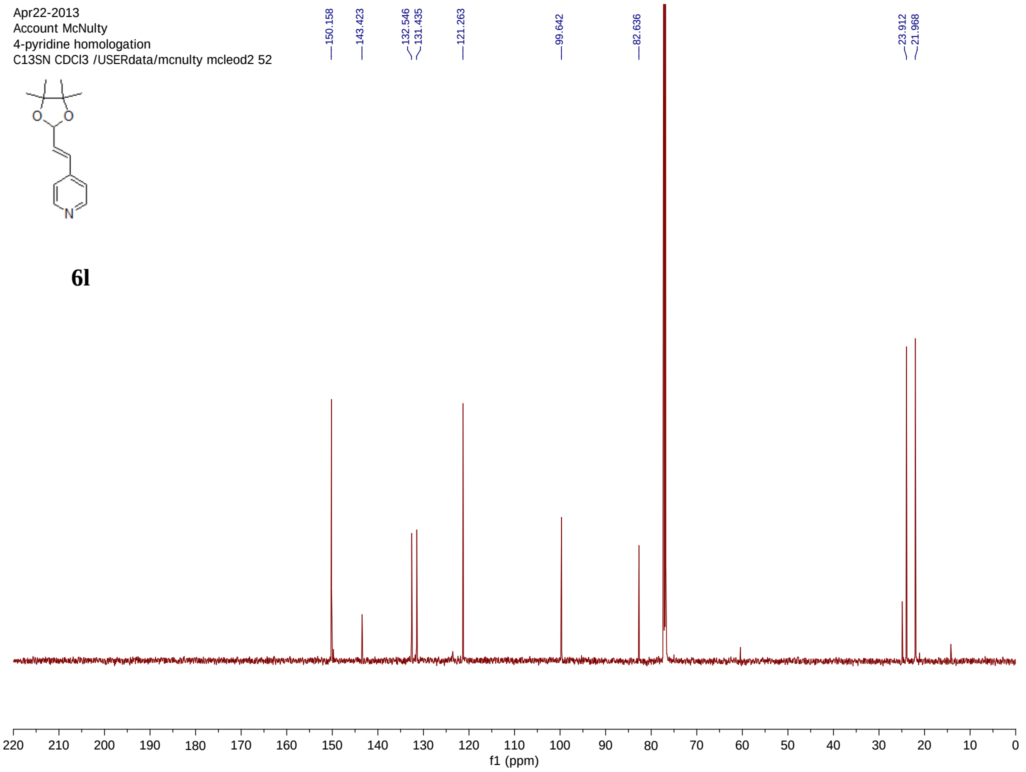

D2Py-4
Account McNulty
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 6
8.383
8.368
8.224
8.211
8.179
8.167
8.142
8.128
8.110
8.095
8.060
8.056
8.045
8.039
8.024
8.007
7.995
7.982
7.815
7.789
7.260
6.424
6.413
6.398
6.387
5.786
5.775
O
O
6j
1.01
1.07
2.07
1.03
1.09
2.20
1.07
1.01
1.00
1.00
5.92
5.88
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

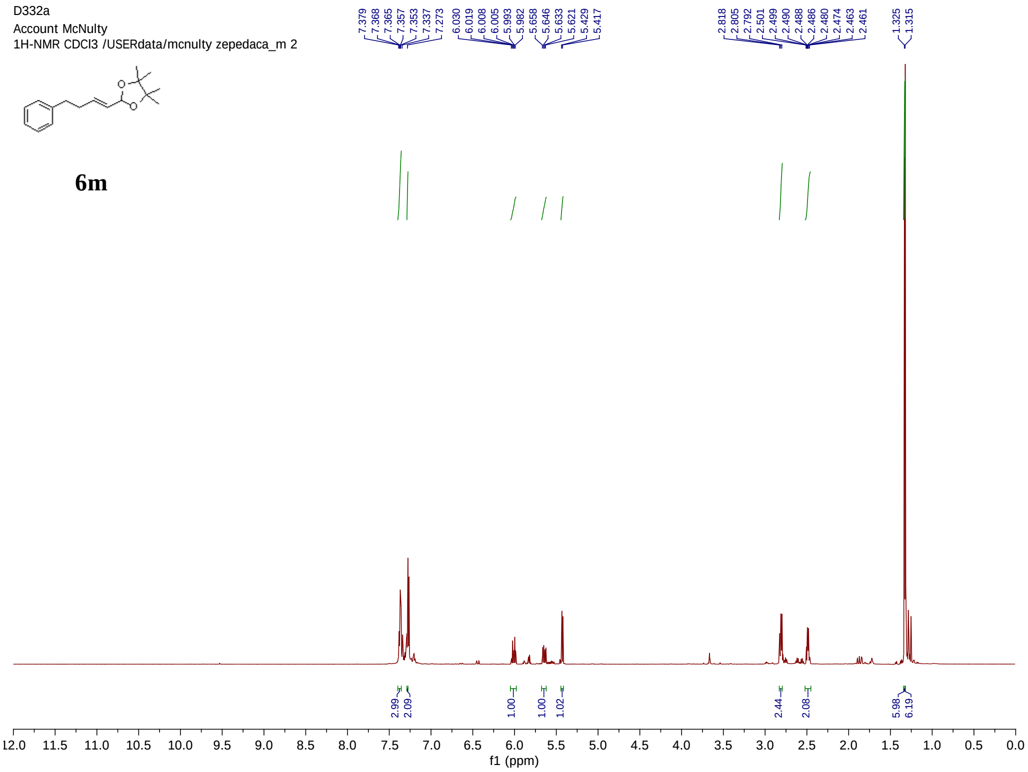

D2py-4
Account McNulty
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 7
131.562
131.381
131.167
130.895
130.816
130.551
128.400
127.637
127.436
127.382
125.946
125.304
125.090
124.932
124.810
124.356
122.983
100.865
82.487
24.055
22.061
O
O
6j
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


D3py-b
Account McNulty
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 59
8.547
8.544
8.427
8.424
8.419
8.416
7.667
7.653
7.193
7.183
7.178
7.170
6.653
6.627
6.179
6.168
6.152
6.141
5.480
5.469
1.209
1.202
N
O
O
6k
1.00
0.91
0.96
0.99
1.02
0.95
0.96
6.06
5.94
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

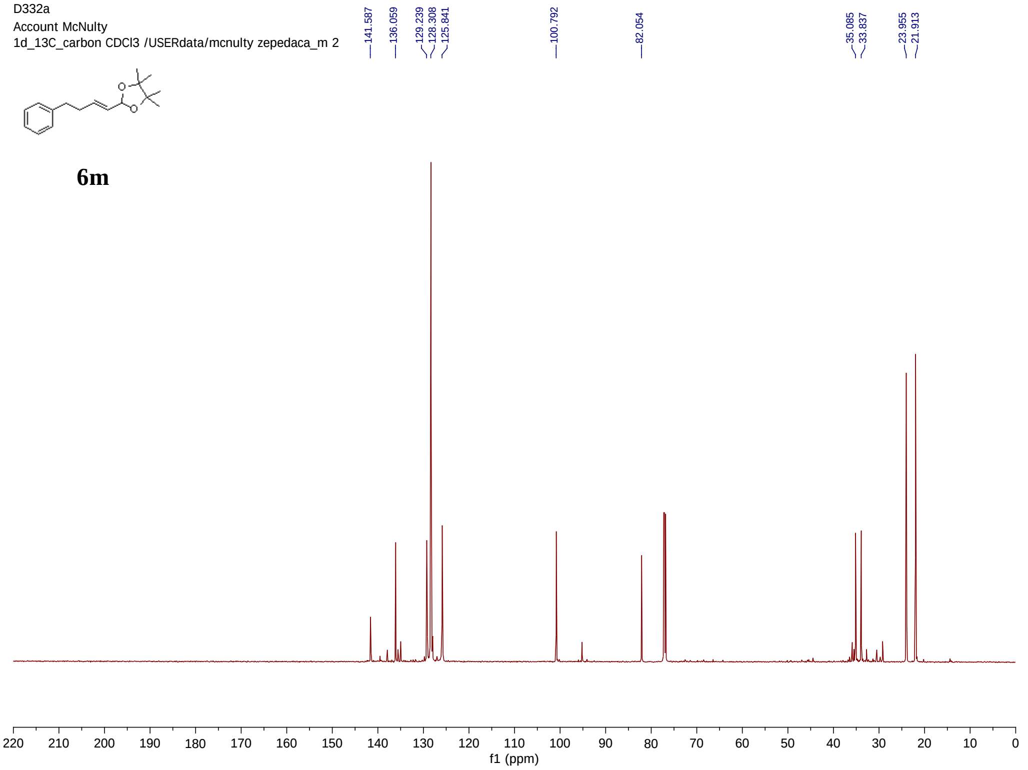

D3py-b
Account McNulty
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 59
148.203
147.757
132.284
130.679
129.529
129.108
122.405
99.079
81.540
22.956
20.979
N
O
O
6k
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Apr22-2013
Account McNulty
4-pyridine homologation
1H-NMR CDCl3 /USERdata/mcnulty mcleod2 52
6l
8.554
8.551
8.546
8.544
7.262
7.260
7.255
7.252
6.669
6.642
6.367
6.356
6.340
6.330
5.542
5.532
1.265
1.264
1.235
1.98
2.52
1.00
0.97
0.97
12.07
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

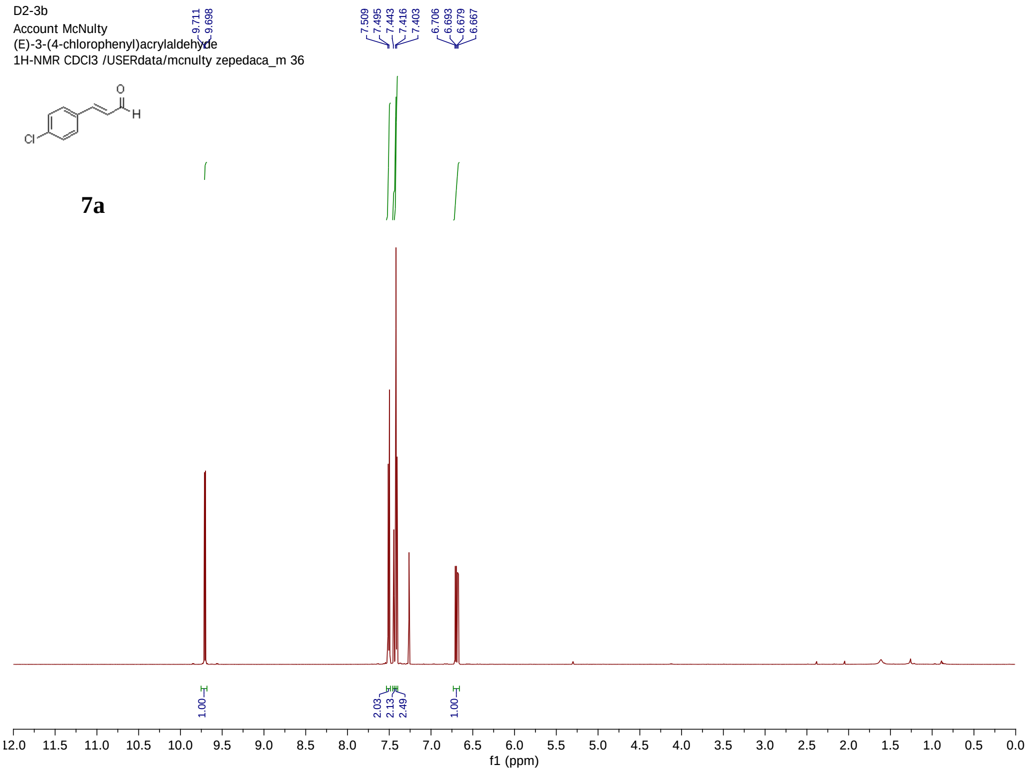

Apr22-2013
Account McNulty
4-pyridine homologation
C13SN CDCl3 /USERdata/mcnulty mcleod2 52
6l
150.158
143.423
132.546
131.435
121.263
99.642
82.636
23.912
21.968
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


D332a
Account McNulty
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 2
7.379
7.368
7.365
7.357
7.353
7.337
7.273
6.030
6.019
6.008
6.005
5.993
5.982
5.658
5.646
5.633
5.621
5.429
5.417
2.818
2.805
2.792
2.501
2.499
2.490
2.488
2.486
2.480
2.474
2.463
2.461
1.325
1.315
2.99
2.09
1.00
1.00
1.02
2.44
2.08
5.98
6.19
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

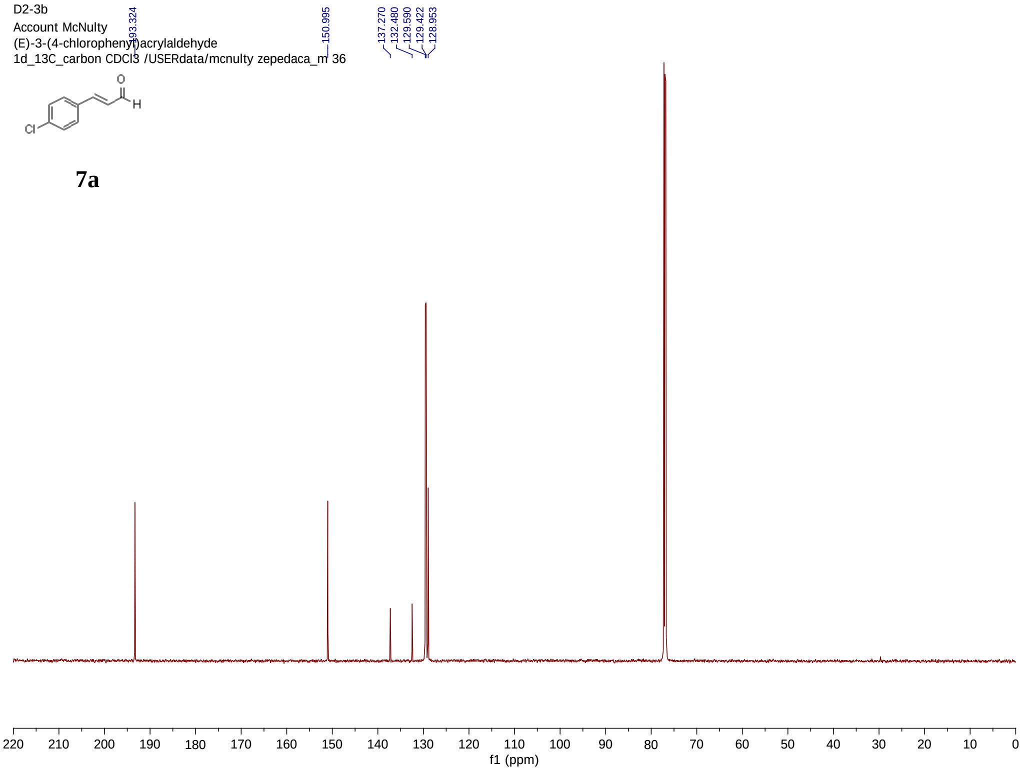
6m


D332a
Account McNulty
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 2
141.587
136.059
129.239
128.308
125.841
100.792
82.054
35.085
33.837
23.955
21.913
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

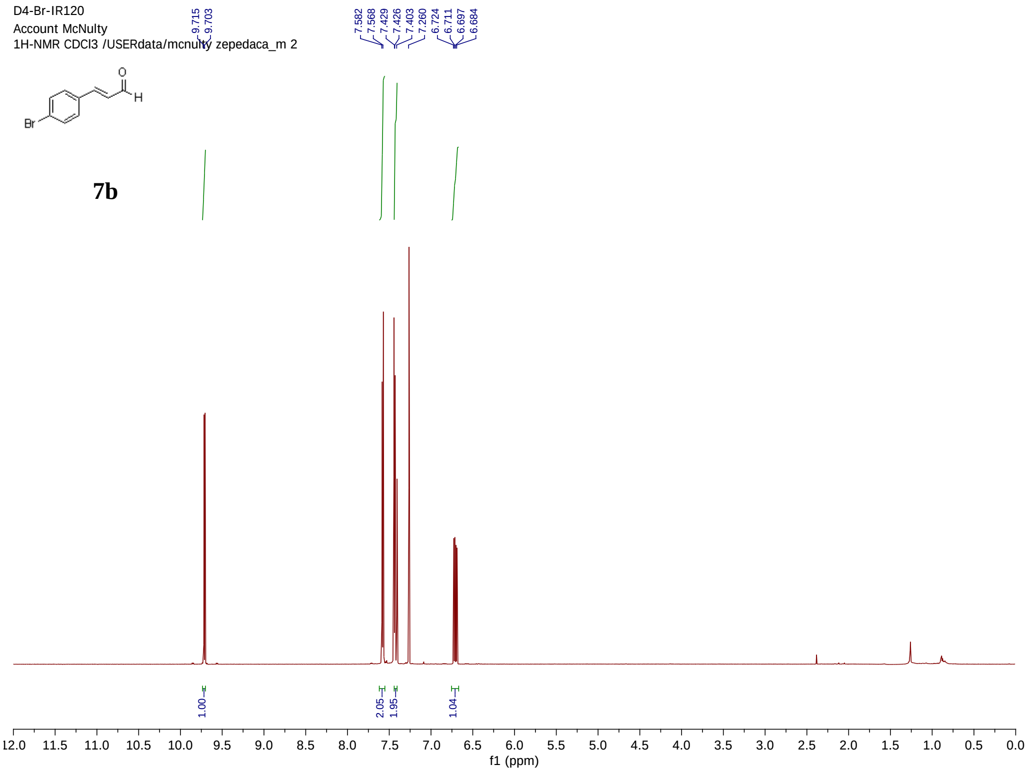
6m

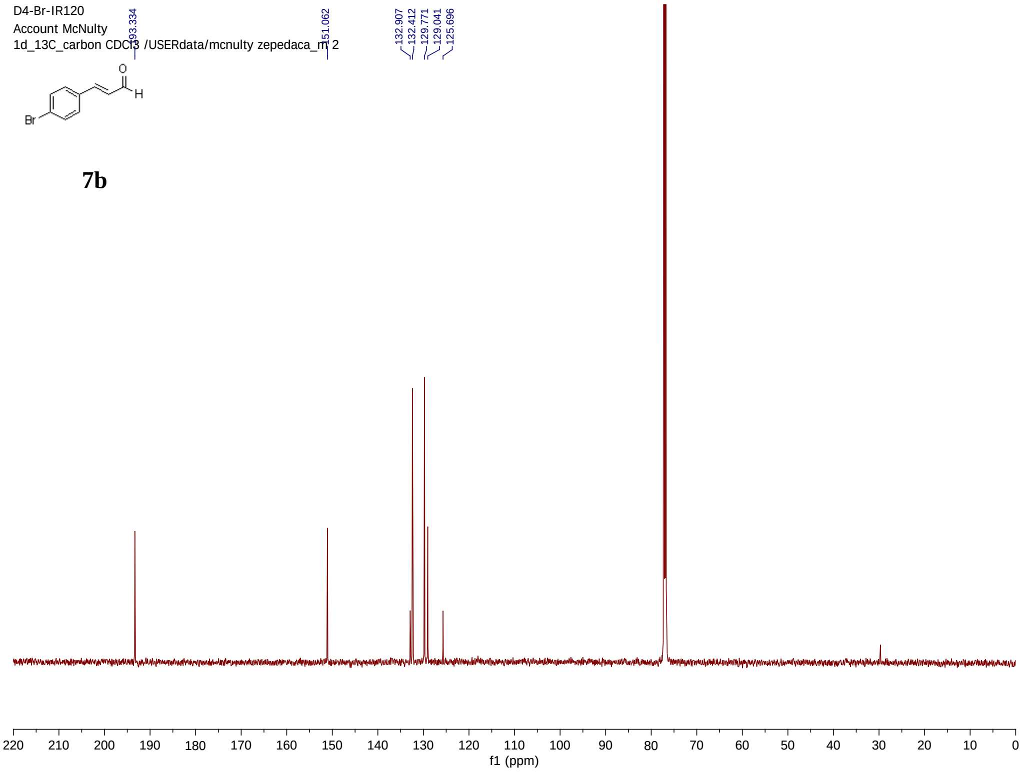

D2-3b
Account McNulty
(E)-3-(4-chlorophenyl)acrylaldehyde
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 36
9.711
9.698
7.509
7.495
7.443
7.416
7.403
6.706
6.693
6.679
6.667
O
H
Cl
1.00
2.03
2.13
2.49
1.00
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

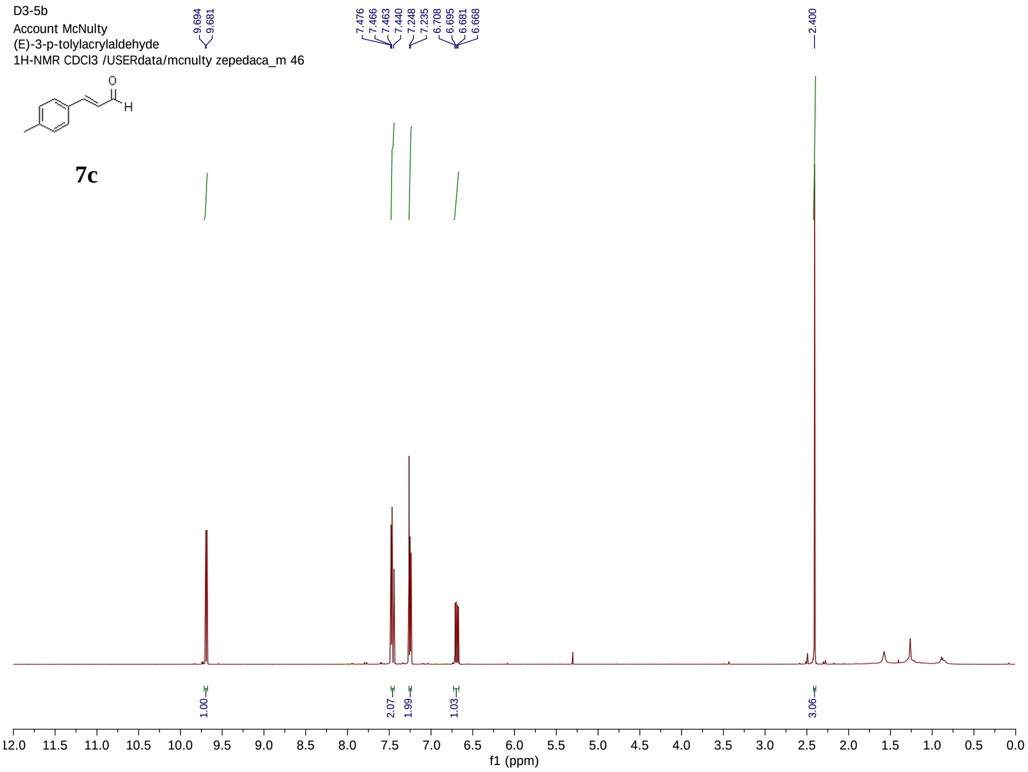
7a


D2-3b
Account McNulty
(E)-3-(4-chlorophenyl)acrylaldehyde
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 36
193.324
150.995
137.270
132.480
129.590
129.422
128.953
O
H
Cl
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

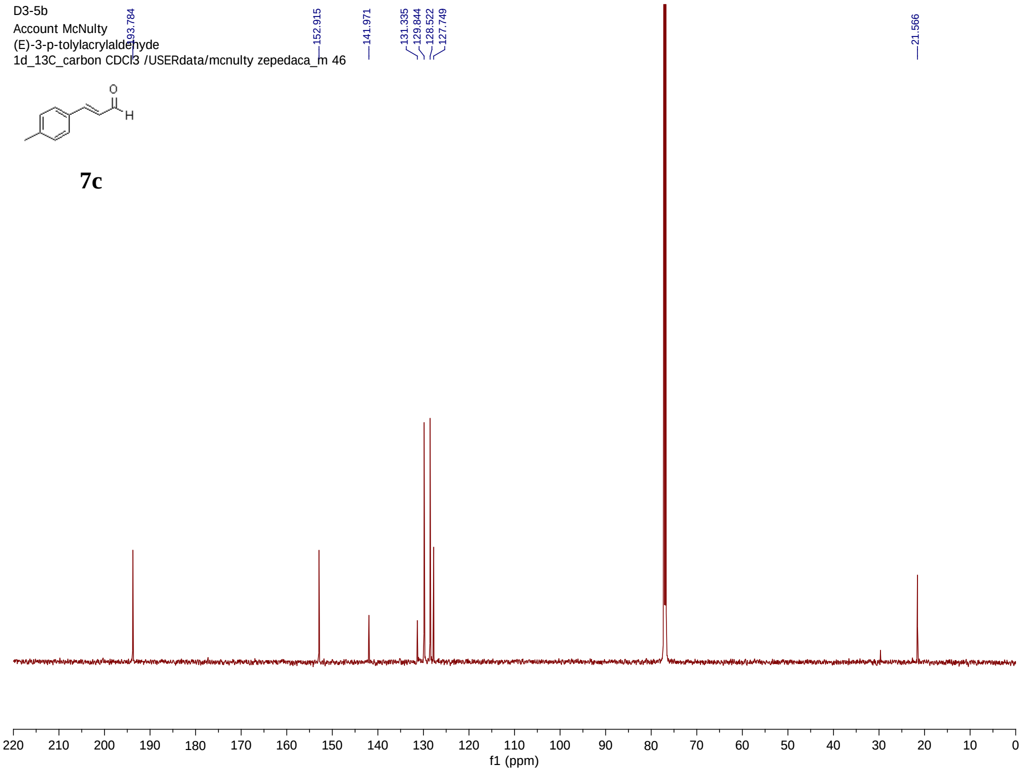
7a


D4-Br-IR120
Account McNulty
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 2
9.715
9.703
7.582
7.568
7.429
7.426
7.403
7.260
6.724
6.711
6.697
6.684
O
H
Br
7b
1.00
2.05
1.95
1.04
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

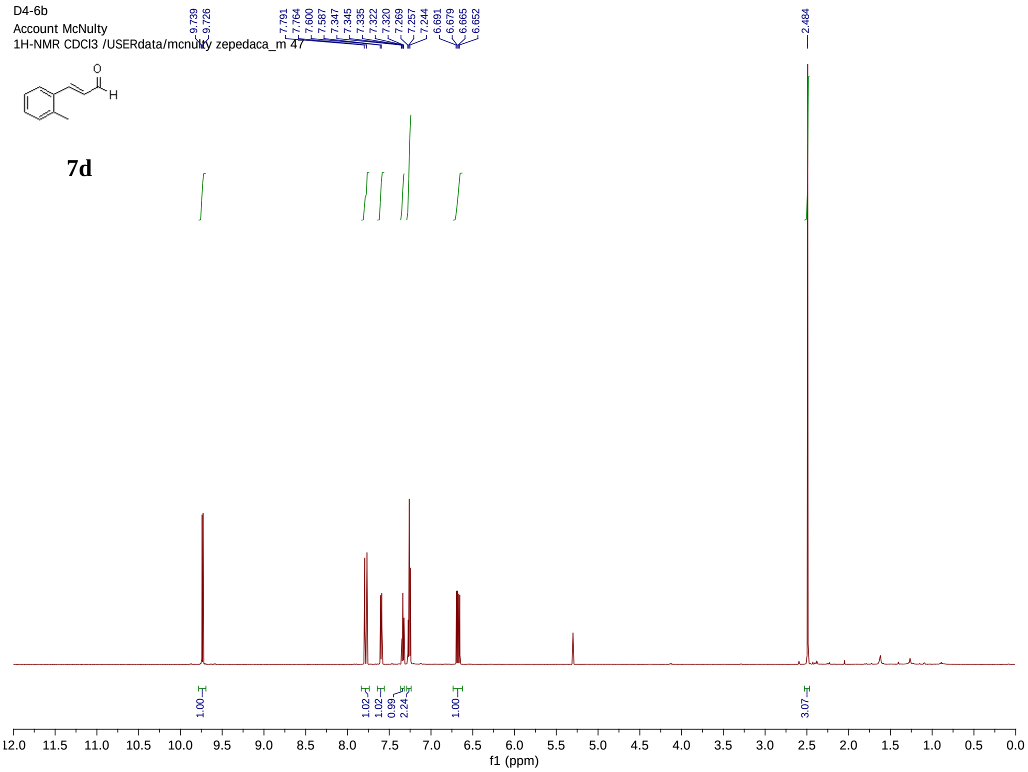

D4-Br-IR120
Account McNulty
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 2
193.334
151.062
132.907
132.412
129.771
129.041
125.696
O
H
Br
7b
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


D3-5b
Account McNulty
(E)-3-p-tolylacrylaldehyde
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 46
O
H
9.694
9.681
7.476
7.466
7.463
7.440
7.248
7.235
6.708
6.695
6.681
6.668
2.400
1.00
2.07
1.99
1.03
3.06
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

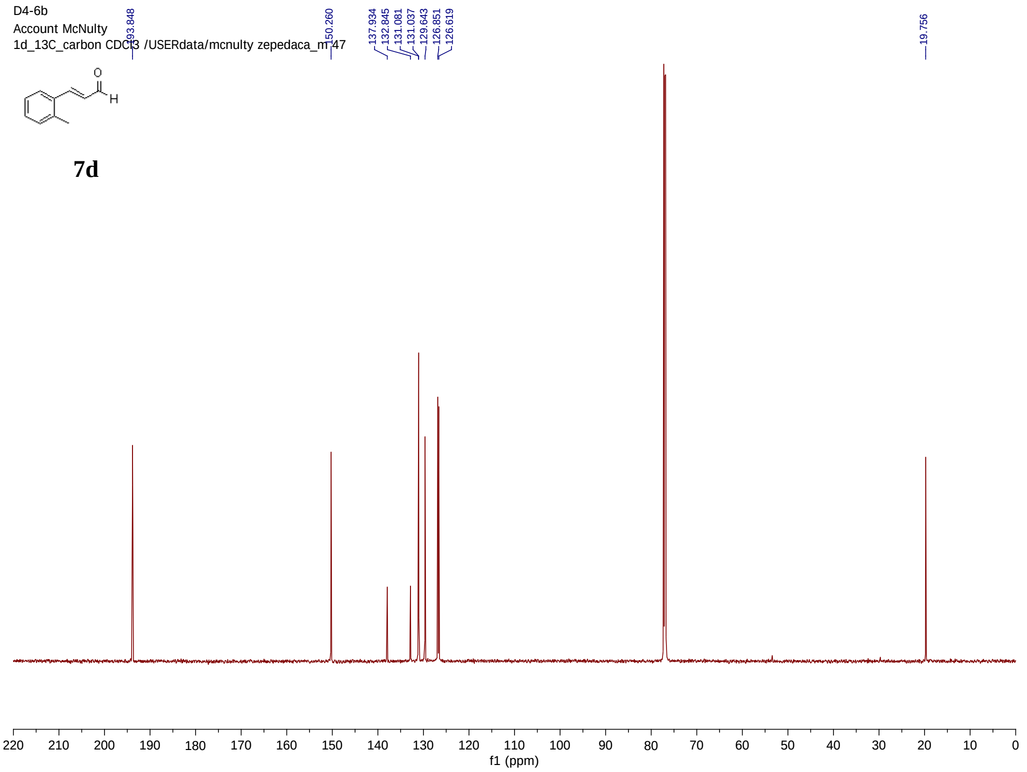
7c


D3-5b
Account McNulty
(E)-3-p-tolylacrylaldehyde
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 46
O
H
193.784
152.915
141.971
131.335
129.844
128.522
127.749
21.566
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

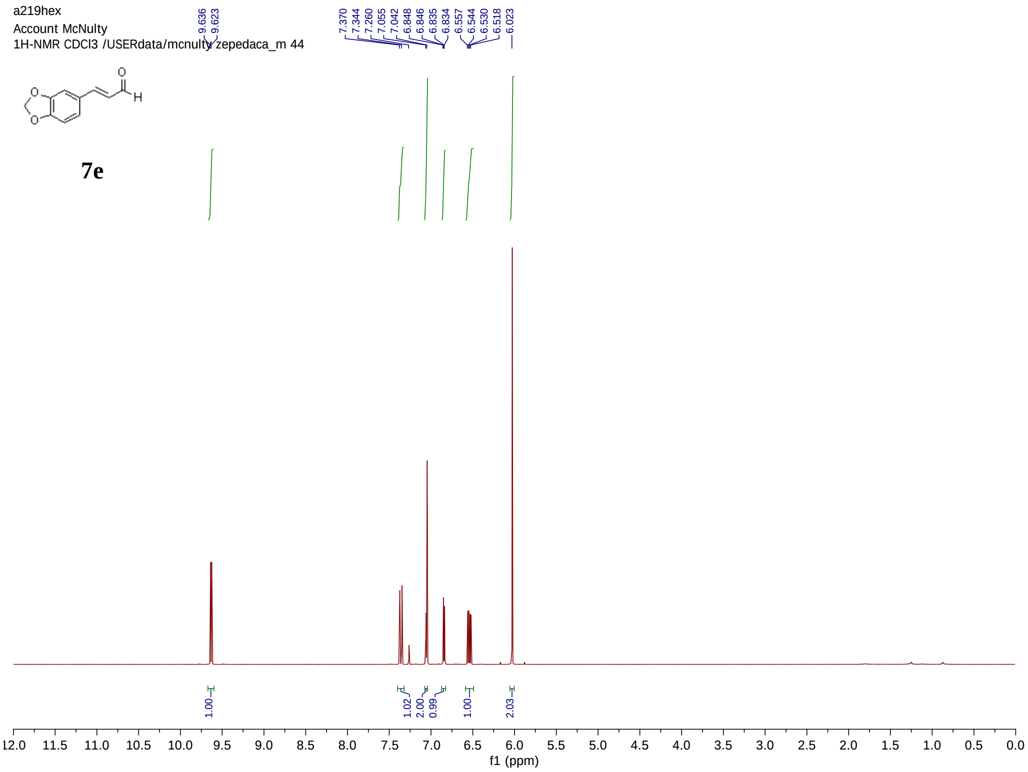
7c


D4-6b
Account McNulty
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 47
9.739
9.726
7.791
7.764
7.600
7.587
7.347
7.345
7.335
7.322
7.320
7.269
7.257
7.244
6.691
6.679
6.665
6.652
2.484
O
H
1.00
1.02
1.02
0.99
2.24
1.00
3.07
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

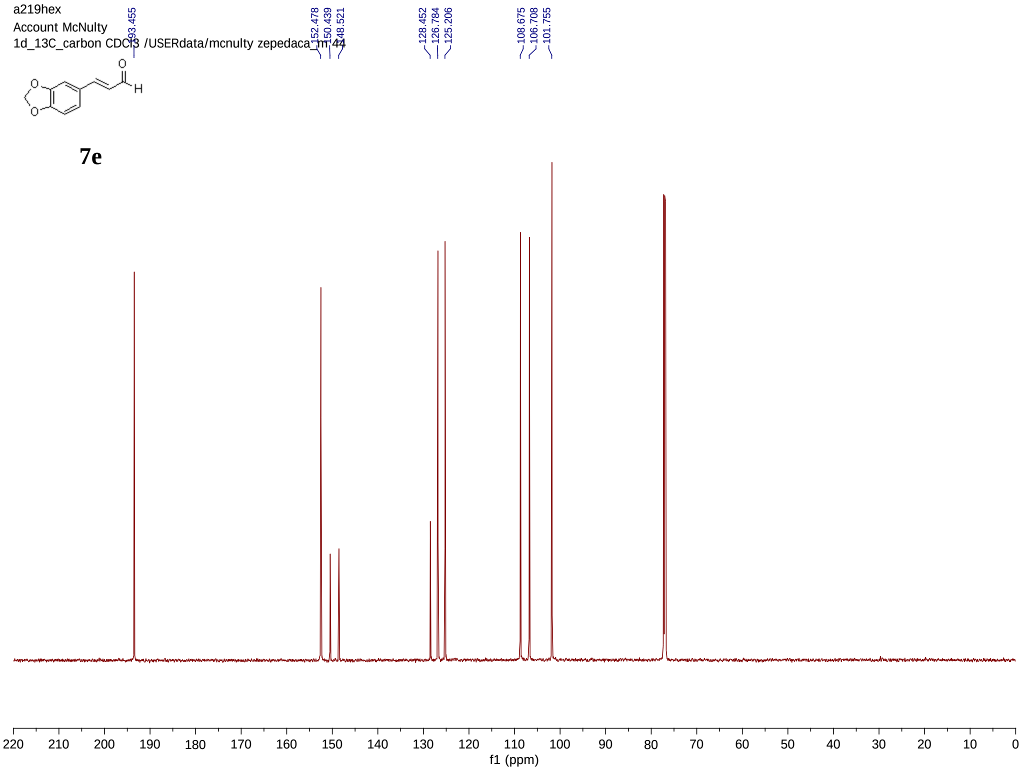
7d


D4-6b
Account McNulty
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 47
193.848
150.260
137.934
132.845
131.081
131.037
129.643
126.851
126.619
19.756
O
H
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

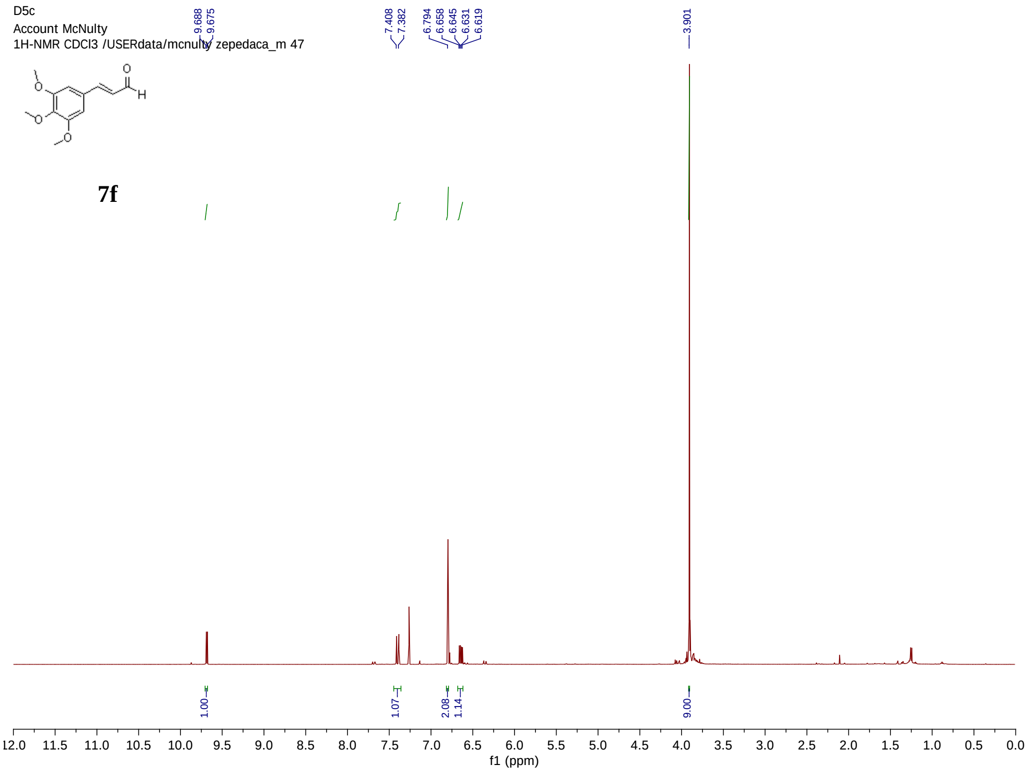
7d

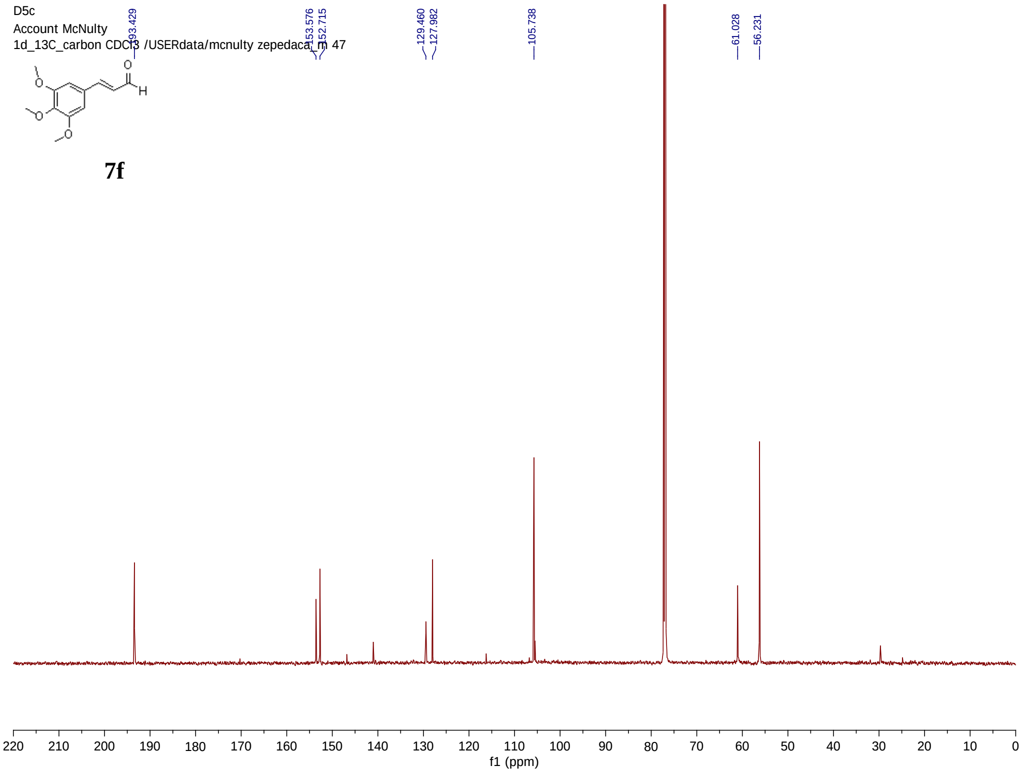

a219hex
Account McNulty
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 44
9.636
9.623
7.370
7.344
7.260
7.055
7.042
6.848
6.846
6.835
6.834
6.557
6.544
6.530
6.518
6.023
O
O
O
H
1.00
1.02
2.00
0.99
1.00
2.03
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

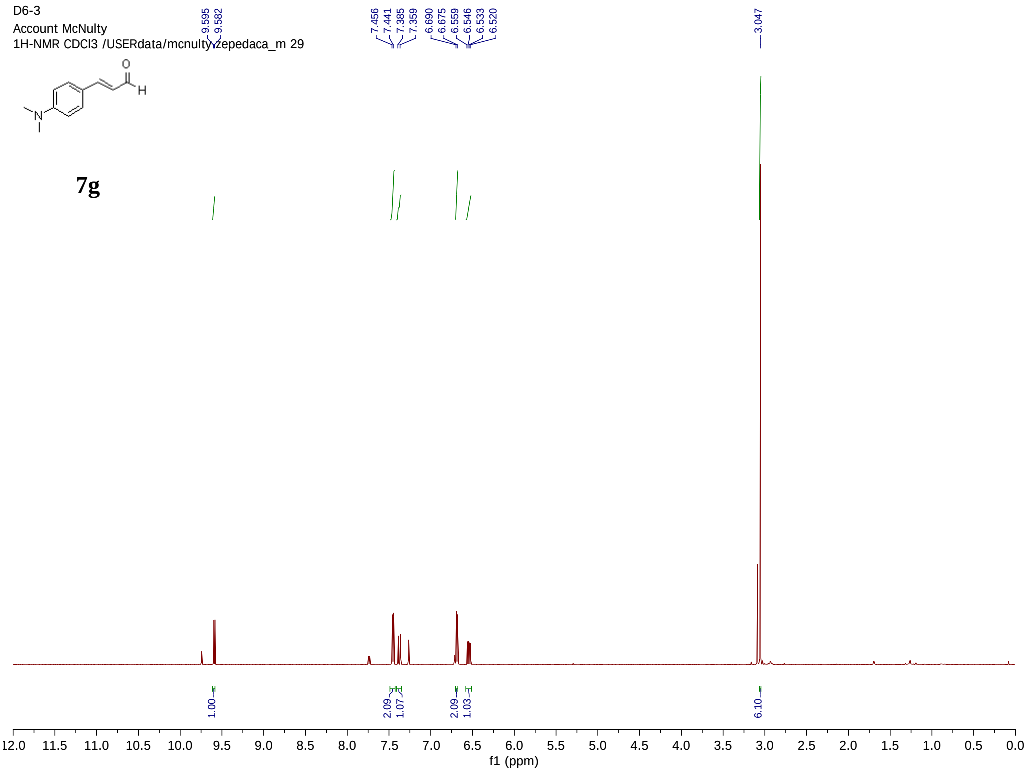
7e


a219hex
Account McNulty
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 44
193.455
152.478
150.439
148.521
128.452
126.784
125.206
108.675
106.708
101.755
O
O
O
H
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

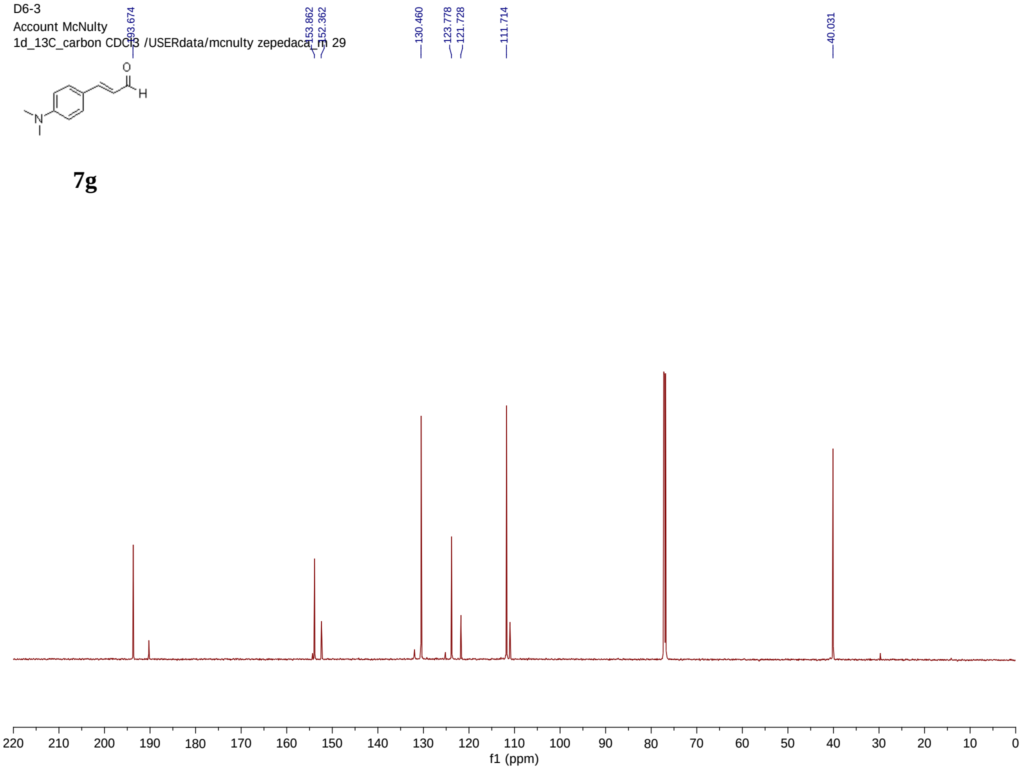
7e


D5c
Account McNulty
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 47
9.688
9.675
7.408
7.382
6.794
6.658
6.645
6.631
6.619
3.901
O
O
O
O
H
7f
1.00
1.07
2.08
1.14
9.00
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

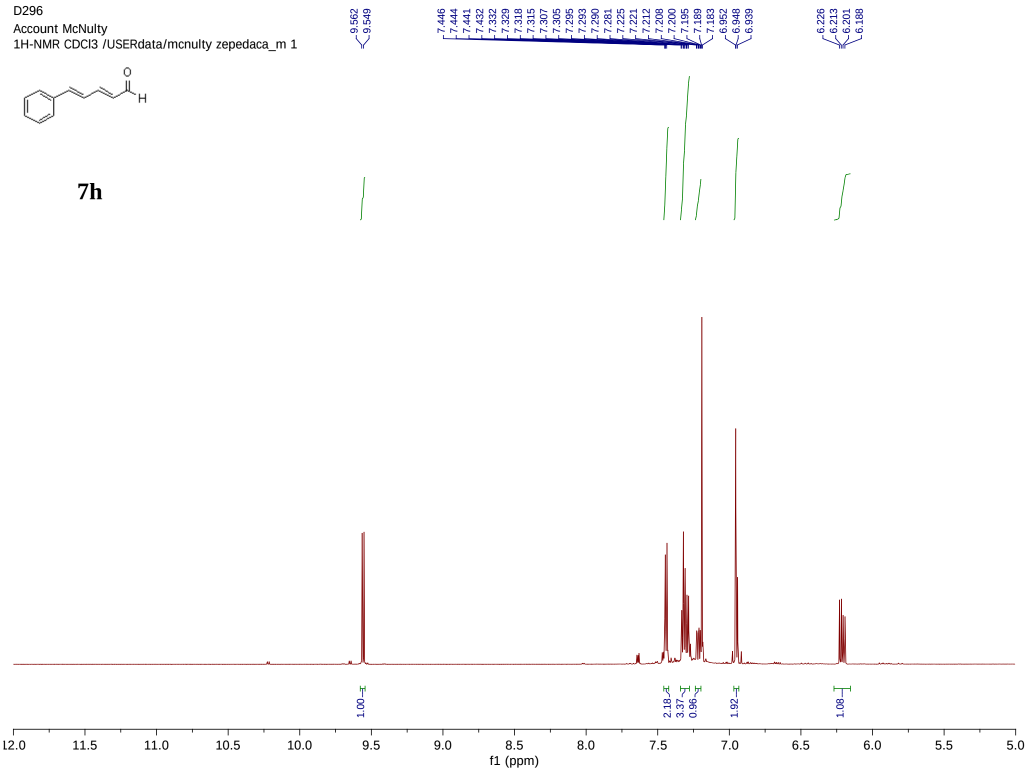

D5c
Account McNulty
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 47
193.429
153.576
152.715
129.460
127.982
105.738
61.028
56.231
O
O
O
O
H
7f
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

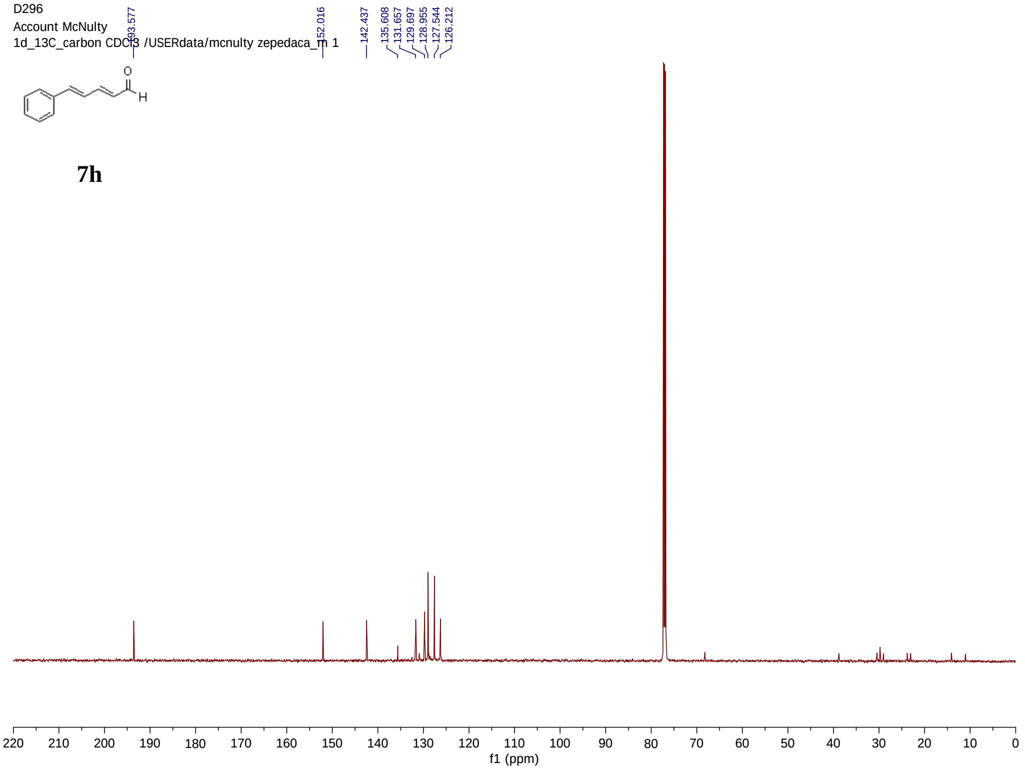

D6-3
Account McNulty
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 29
O
H
N
9.595
9.582
7.456
7.441
7.385
7.359
6.690
6.675
6.559
6.546
6.533
6.520
3.047
1.00
2.09
1.07
2.09
1.03
6.10
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

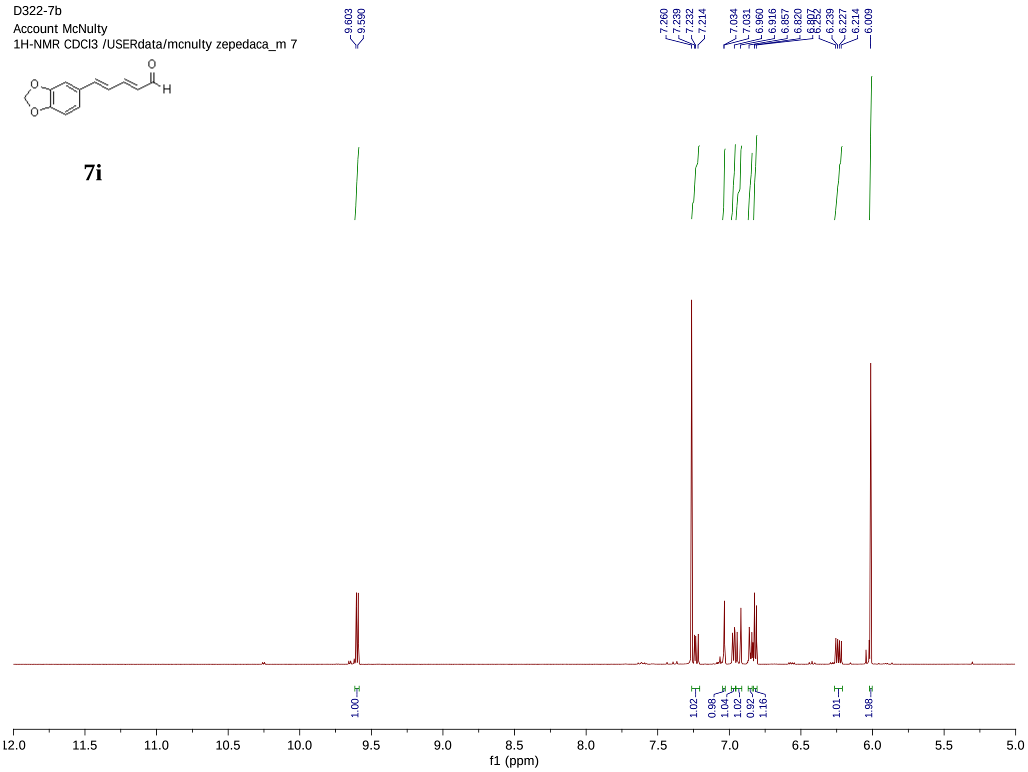
7g


D6-3
Account McNulty
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 29
O
H
N
193.674
153.862
152.362
130.460
123.778
121.728
111.714
40.031
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

7g


D296
Account McNulty
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 1
O
H
7h
9.562
9.549
7.446
7.444
7.441
7.432
7.332
7.329
7.318
7.315
7.307
7.305
7.295
7.293
7.290
7.281
7.225
7.221
7.212
7.208
7.200
7.195
7.189
7.183
6.952
6.948
6.939
6.226
6.213
6.201
6.188
1.00
2.18
3.37
0.96
1.92
1.08
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
f1 (ppm)


D296
Account McNulty
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 1
O
H
7h
193.577
152.016
142.437
135.608
131.657
129.697
128.955
127.544
126.212
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


D322-7b
Account McNulty
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 7
O
O
O
H
7i
9.603
9.590
7.260
7.239
7.232
7.214
7.034
7.031
6.960
6.916
6.857
6.820
6.807
6.252
6.239
6.227
6.214
6.009
1.00
1.02
0.98
1.04
1.02
0.92
1.16
1.01
1.98
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
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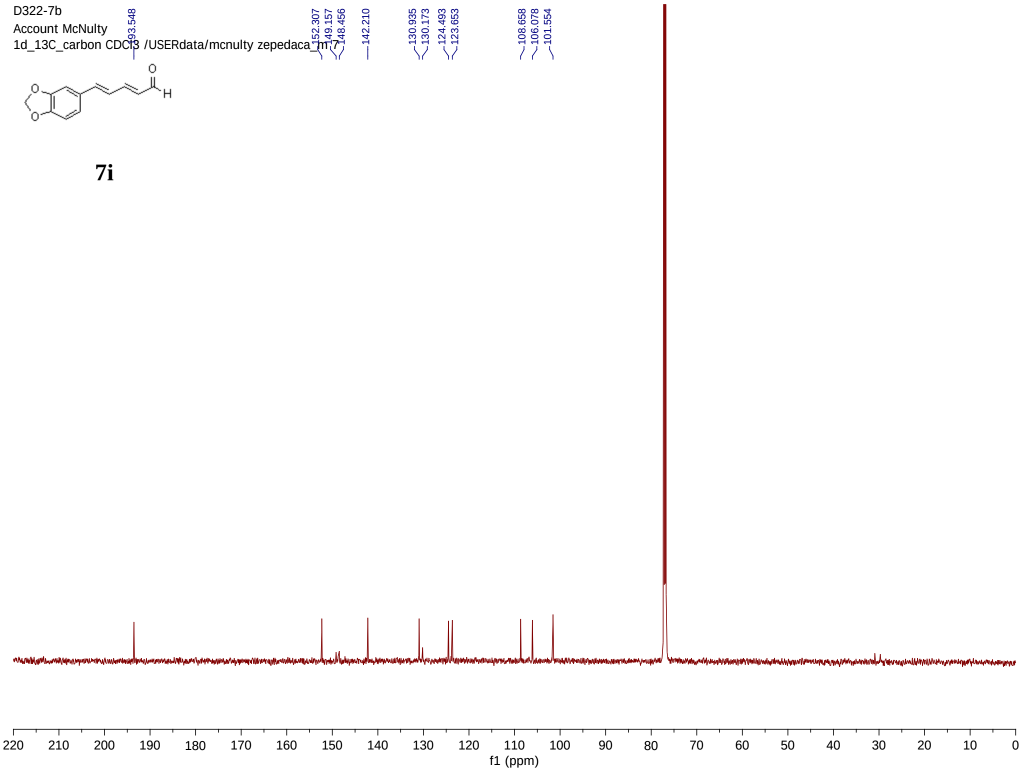
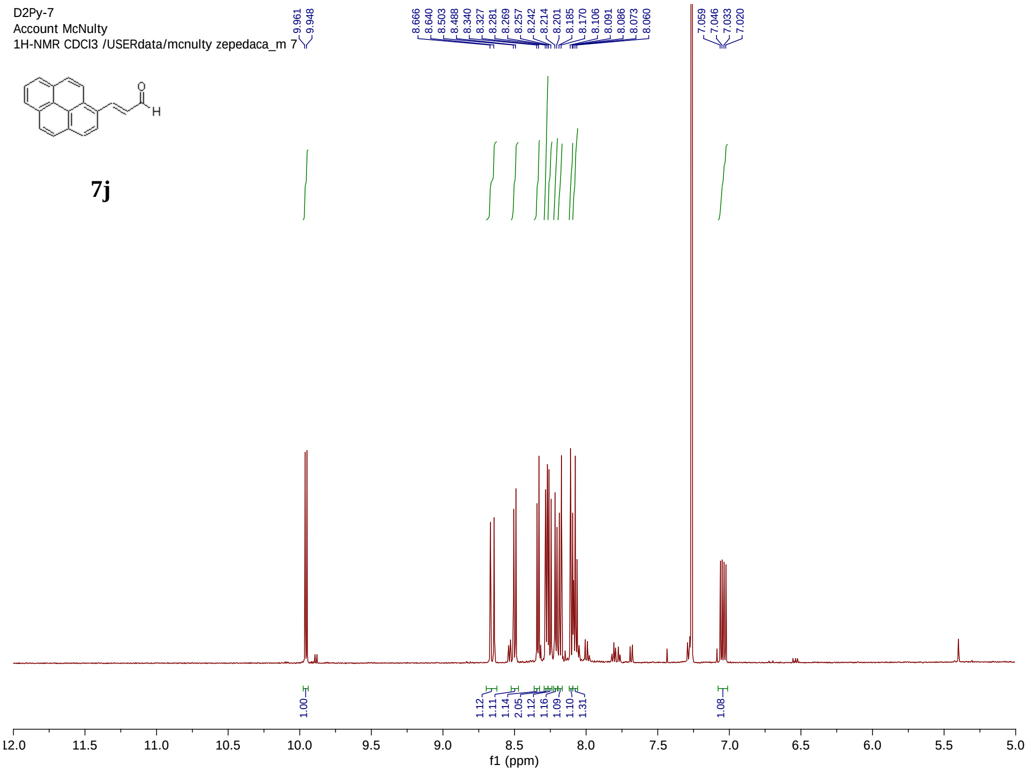
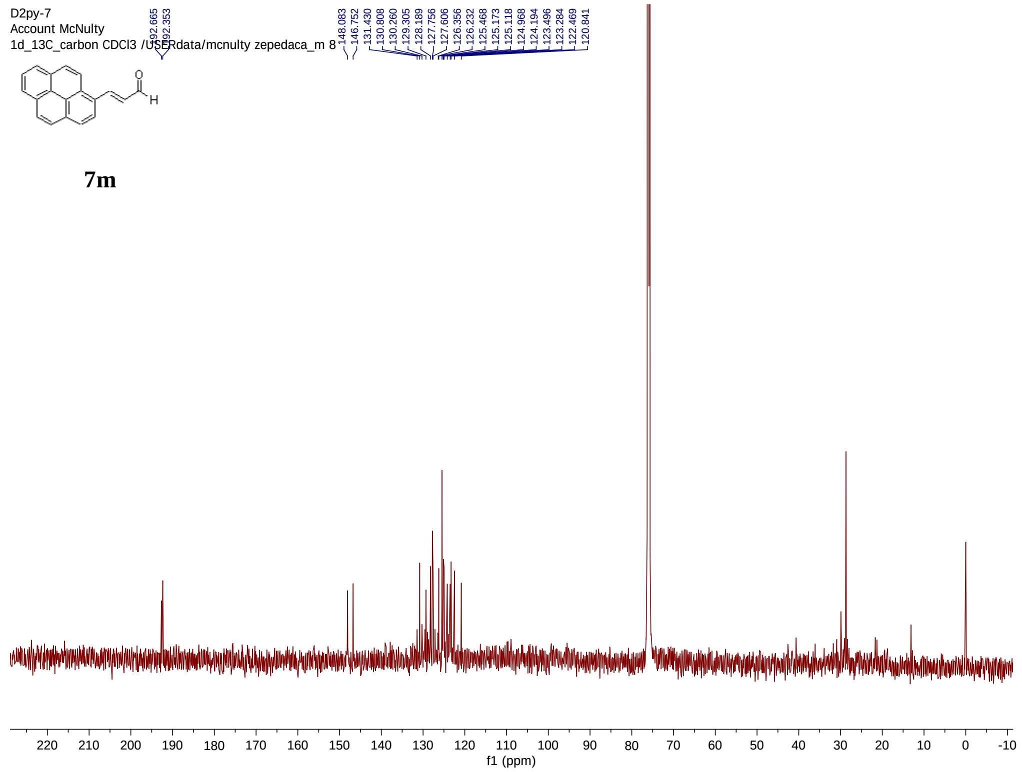
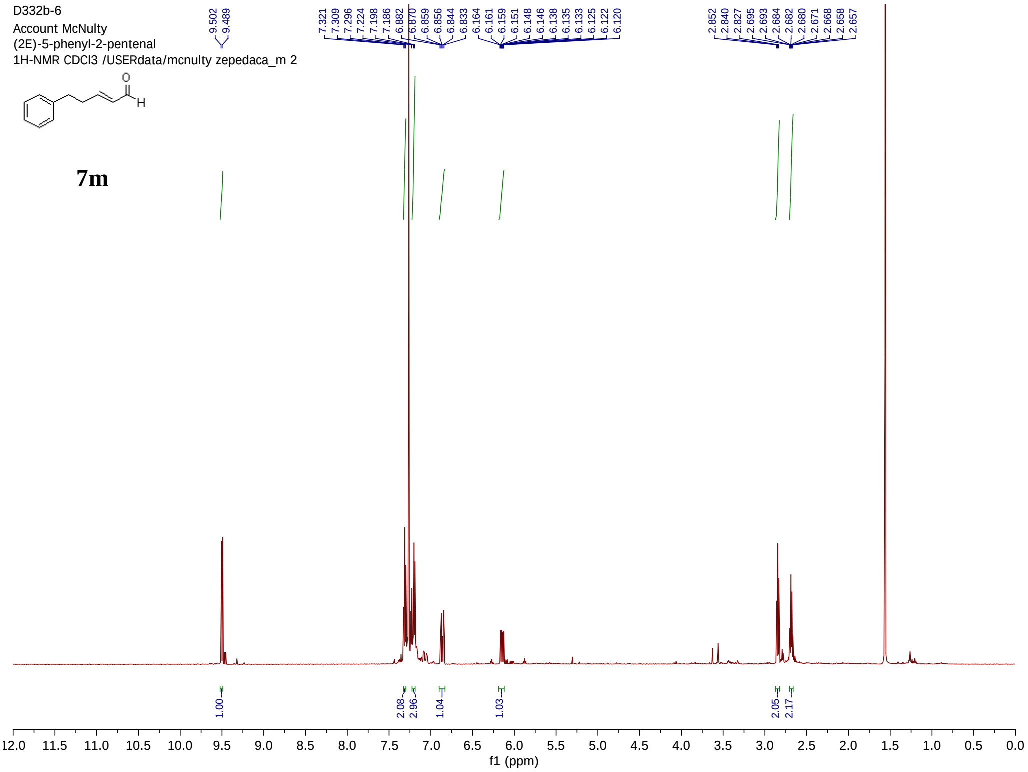
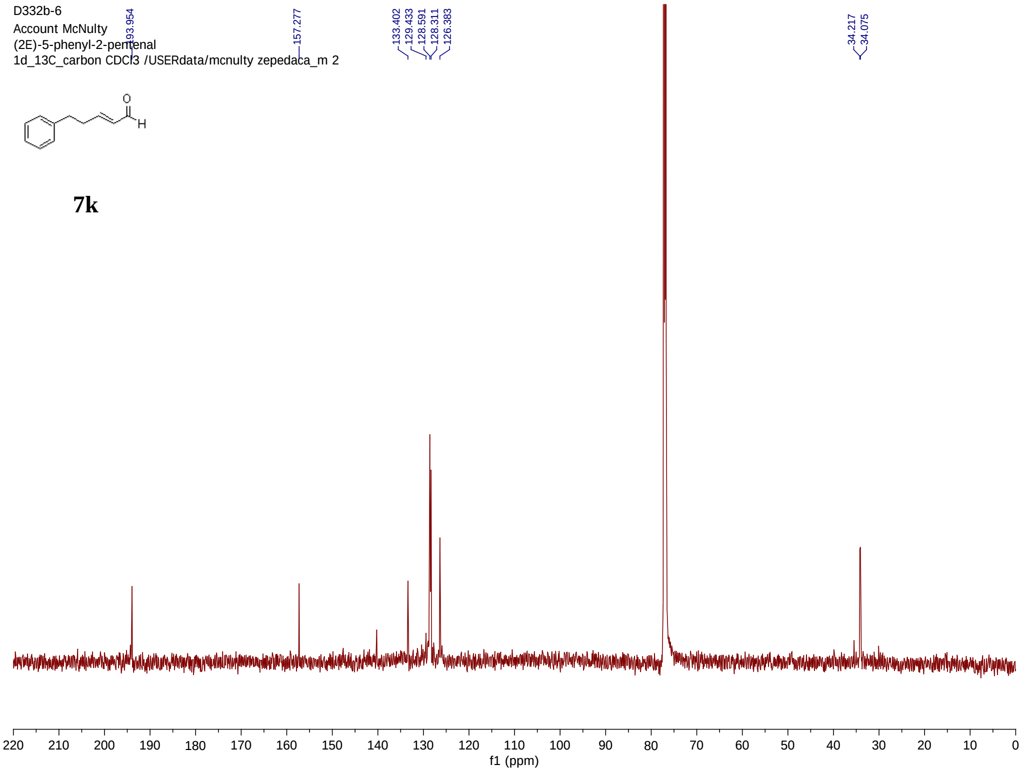
6.0
5.5
5.0
f1 (ppm)


D322-7b
Account McNulty
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 7
O
O
O
H
7i
193.548
152.307
149.157
148.456
142.210
130.935
130.173
124.493
123.653
108.658
106.078
101.554
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


D2Py-7
Account McNulty
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 7
7j
9.961
9.948
8.666
8.640
8.503
8.488
8.340
8.327
8.281
8.269
8.257
8.242
8.214
8.201
8.185
8.170
8.106
8.091
8.086
8.073
8.060
7.059
7.046
7.033
7.020
1.00
1.12
1.11
1.14
2.05
1.12
1.16
1.09
1.10
1.31
1.08
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
f1 (ppm)


D2py-7
Account McNulty
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 8
7m
192.665
192.353
148.083
146.752
131.430
130.808
130.260
129.305
128.189
127.756
127.606
126.356
126.232
125.468
125.173
125.118
124.968
124.194
123.496
123.284
122.469
120.841
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


D332b-6
Account McNulty
(2E)-5-phenyl-2-pentenal
1H-NMR CDCl3 /USERdata/mcnulty zepedaca_m 2
O
H
7m
9.502
9.489
7.321
7.309
7.296
7.224
7.198
7.186
6.882
6.870
6.859
6.856
6.844
6.833
6.164
6.161
6.159
6.151
6.148
6.146
6.138
6.135
6.133
6.125
6.122
6.120
2.852
2.840
2.827
2.695
2.693
2.684
2.682
2.680
2.671
2.668
2.658
2.657
1.00
2.08
2.96
1.04
1.03
2.05
2.17
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


D332b-6
Account McNulty
(2E)-5-phenyl-2-pentenal
1d_13C_carbon CDCl3 /USERdata/mcnulty zepedaca_m 2
O
H
7k
193.954
157.277
133.402
129.433
128.591
128.311
126.383
34.217
34.075
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)