Tropylium Salts as Efficient Organic Lewis Acid Catalysts for Acetalization and Transacetalization Reactions in Batch and Flow

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General Methods

Reactions, unless otherwise stated, were conducted under a positive pressure of argon in oven-dried glassware. Commercially available reagents were used as purchased unless otherwise noted. Solvents were pre-distilled according to standard laboratory methods. Analytical thin layer chromatography was performed using Merck aluminium plates pre-coated with silica gel 60 F_{254} (0.2 mm) pre-treated with a solution mixture of *n*-pentane/diethyl ether/triethylamine (92/6/2). Visualization of the developed TLC plates was performed with ultraviolet irradiation (254 nm). Products were purified by flash chromatography, unless otherwise stated, using Merck silica gel 60, particle size 0.040-0.063 mm (230-240 mesh, flash). Solvents used for chromatography are quoted as volume/volume ratios.

NMR spectroscopy was performed at 298 K using an Agilent VNMRS 600 (600.13 MHz, ^{1}H; 151.0 MHz, ^{13}C) or Inova 400 (400.1 MHz, ^{1}H; 100.6 MHz, ^{13}C) spectrometers. ^{1}H NMR data is expressed in parts per million (ppm) downfield shift from tetramethylsilane with residual solvent as an internal reference (δ 7.26 ppm for chloroform) and is reported as position (δ in ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, ddq, doublet doublet quartet, m = multiplet), coupling constant (J in Hz) and integration (number of protons). ^{13}C NMR spectra were recorded at 298 K with complete proton decoupling. Data is expressed in parts per million (ppm) downfield shift relative to the internal reference (δ 77.2 ppm for the central peak of deuterated chloroform). Abbreviations are as follows: s (singlet), d (doublet), dd (doublet of doublet) t (triplet), q (quartet), m (multiplet), bs (broad singlet).

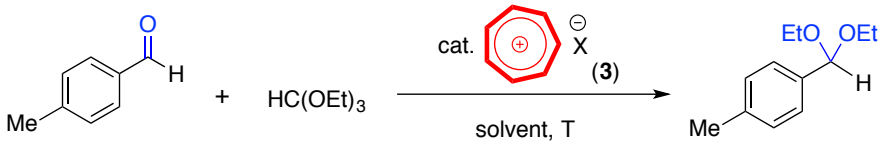
Infrared spectra were obtained on a PerkinElmer@100 FT-IR spectrometer and are reported in wavenumbers (cm^{-1}).

LR-MS data were recorded on a Finnigan SSQ 7000 or Finnigan MAT 95 using EI ionization method. HRMS data were recorded on a ThermoFisher Scientific LTQ Orbitrap XL using ESI ionization method.

Optimization of Tropylium-Catalyzed Diethyl Acetalization Reaction of *p*-Tolualdehyde

General procedure for Table 1: A mixture of *p*-tolualdehyde **1a** (0.5 mmol), triethylorthoformate **2a** (1.0 mmol) and tropylium salt **3** (cat.) was taken up in dry acetonitrile (0.6 mL) in a vial loaded with a stirrer bar under argon atmosphere. The reaction mixture was heated to 70°C for five hours then cooled to room temperature. The resulting crude reaction mixture was concentrated under reduced pressure. The product was purified from the residues by column chromatography (silica-gel, pentane/diethylether/triethylamine = 92/6/2).

Table 1. Optimization of tropylium-promoted acetalization reaction[a]

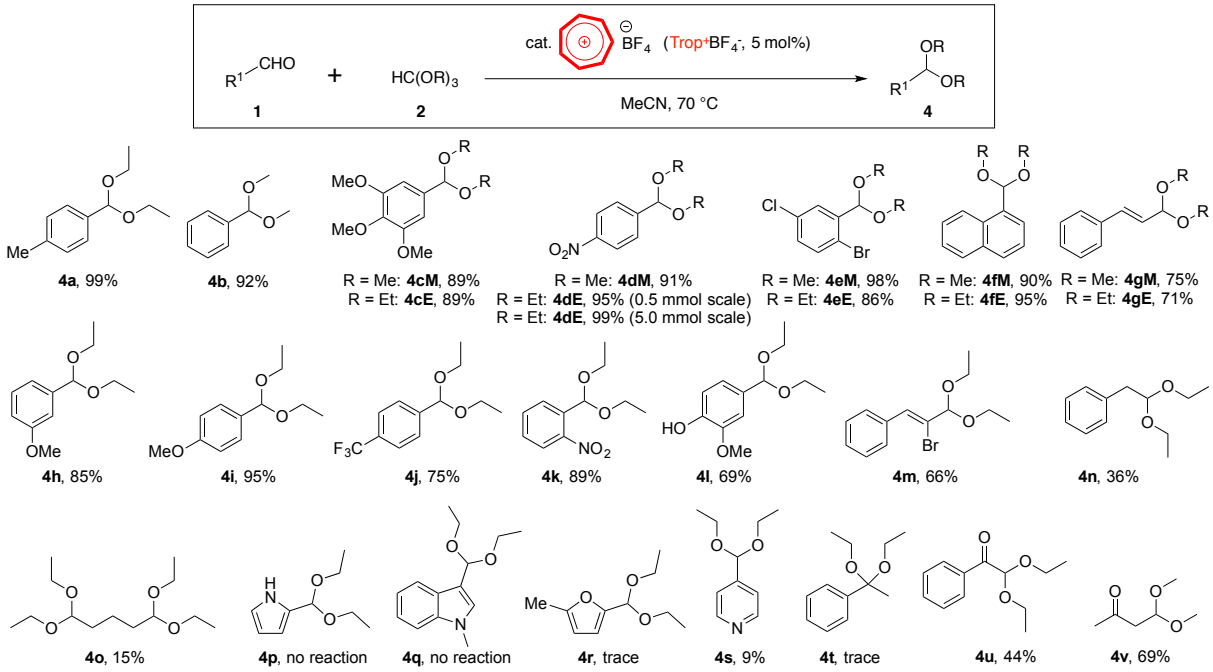


1a **2a** **4a**

Entry	X	mol% cat.	solvent	T (°C)	time[b]	Yield[c]
1	BF_4	5	DCM	rt/70	24	83/74
2	BF_4	5	toluene	rt/70	24	56/69
3	BF_4	5	neat	70	24	46
4	BF_4	2.5	neat	70	5	80
5	BF_4	5	MeCN	rt/reflux	24/5	63/61
6	**BF_4**	**5**	**MeCN**	**70**	**5**	**99**
7[d]	BF_4	5	MeCN	70	5	89
8	BF_4	2.5	MeCN	70	5	84
9	BF_4	1	MeCN	70	5	75
10	-	0	MeCN	70	24	12
11	Br	5	MeCN	70	5	81
12	OTf	5	MeCN	70	5	88
13	BPh_4	5	MeCN	70	5	90
14[e]	BF_4	5	MeCN	70	5	96

[a] Conditions: aldehyde **1a** (0.5 mmol), triethyl orthoformate **2a** (1.0 mmol), cat. **3** in dry solvent (0.6 mL) under Ar atmosphere. [b] Reaction time (hour) for total consumption of aldehyde **1a**. [c] Yield of the isolated product. [d] Reaction flask opened to air. [e] 10 mol% of 2,6-di-*tert*-butyl-4-methylpyridine added in extra-dry MeCN solvent.[9]

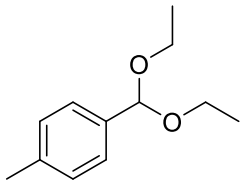
Substrate Scope (Scheme 2)



General procedure: A mixture of aldehyde **1** (0.5 mmol), triethylorthoformate or trimethylorthoformate **2** (1.0 mmol) and tropylium tetrafluoroborate **3** (0.025 mmol) was taken up in dry acetonitrile (0.6 mL) in a vial loaded with a stirrer bar under argon atmosphere. The reaction mixture was heated to 70°C for five hours then cooled to room temperature. The resulting crude reaction mixture was concentrated under reduced pressure. The product was purified from the residues by column chromatography (silica-gel, *n*-pentane/diethylether/triethylamine = 92/6/2).

Characterization Data of Acetal Products (Scheme 2)

1-(Diethoxymethyl)-4-methylbenzene (4a): Prepared according to the general procedure from *p*-tolualdehyde and triethylorthoformate to yield the title compound as a colourless oil (99 mg, 0.50 mmol, quant. yield).



4a

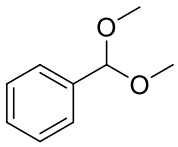
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 8.1 Hz, 2H), 7.18 (d, *J* = 7.9 Hz, 2H), 5.50 (s, 1H), 3.59 (ddq, *J* = 35.4, 9.5, 7.1 Hz, 4H), 2.37 (s, 3H), 1.25 (t, *J* = 7.1 Hz, 6H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$) δ 138.0, 136.3, 128.9, 126.64, 101.7, 61.0, 21.3, 15.3 ppm;

IR (KBr) 2973, 2880, 2327, 2102, 1909, 1740, 1448 cm^{-1};

ESI-MS Anal. Calcd. for 217.1199 $C_{12}H_{18}O_2Na$, found 217.1195.

(Dimethoxymethyl)benzene[1] (4b): Prepared according to the general procedure from benzaldehyde and trimethylorthoformate to yield the title compound as a colourless oil (83 mg, 0.46 mmol, 92% yield).



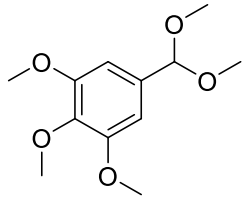
4b

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.50 – 7.42 (m, 2H), 7.42 – 7.29 (m, 3H), 5.40 (s, 1H), 3.34 (s, 6H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$) *δ* 138.18, 128.56, 128.31, 126.80, 103.30, 52.83 ppm;

[1] M. Baxter, Y. Bolshan, *Chem. Eur. J.* **2015**, *21*, 13535-13538.

5-(Dimethoxymethyl)-1,2,3-trimethoxybenzene (4cM): Prepared according to the general procedure from 3,4,5-trimethoxybenzaldehyde and trimethylorthoformate to yield the title compound as a colourless oil (108 mg, 0.45 mmol, 89% yield).



4cM

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.67 (s, 2H), 5.28 (s, 1H), 3.85 (s, 6H), 3.82 (s, 3H), 3.32 (s, 6H) ppm;

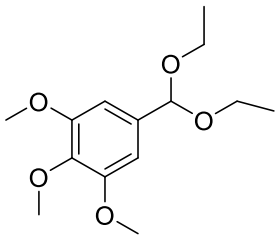
^{13}C NMR (101 MHz, $CDCl_3$) δ 153.2, 138.0, 133.9, 103.6, 103.3, 60.9, 56.2, 53.0 ppm;

IR (KBr) 2940, 2833, 2322, 2100, 1904, 1741, 1591 cm^{-1};

EI-MS: *m/z* (%) = 242.1 (29, [M^+]), 211.1 (71, [M - CH_3O]);

ESI-MS Anal. Calcd. for 265.1046 $C_{12}H_{18}O_5Na$, found 265.1039.

5-(Diethoxymethyl)-1,2,3-trimethoxybenzene (4cE): Prepared according to the general procedure from 3,4,5-trimethoxybenzaldehyde and triethylorthoformate to yield the title compound as a colourless oil (122 mg, 0.45 mmol, 89% yield).



4cE

^{1}H NMR (600 MHz, $CDCl_3$) δ 6.66 (s, 2H), 5.34 (s, 1H), 3.82 (s, 6H), 3.78 (s, 3H), 3.53 (ddq, *J* = 62.8, 9.5, 7.1 Hz, 4H), 1.20 (t, *J* = 7.2 Hz, 6H) ppm;

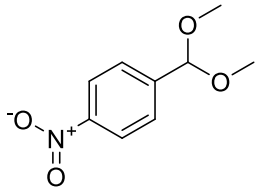
^{13}C NMR (151 MHz, $CDCl_3$) δ 153.1, 137.8, 134.8, 103.4, 101.7, 61.3, 60.7, 56.0, 15.2 ppm;

IR (KBr) 2973, 2935, 2882, 2838, 2112, 1739, 1592 cm^{-1};

EI-MS: *m/z* (%) = 270.2 (33, [M^+]), 225.2 (66, [M - CH_3CH_2O]);

ESI-HRMS Anal. Calcd. for 293.1359 $C_{14}H_{22}O_5Na$, found 293.1357.

1-(Dimethoxymethyl)-4-nitrobenzene[1] (4dM): Prepared according to the general procedure from 4-nitrobenzaldehyde and trimethylorthoformate to yield the title compound as a light-yellow oil (93 mg, 0.47 mmol, 91% yield).



4dM

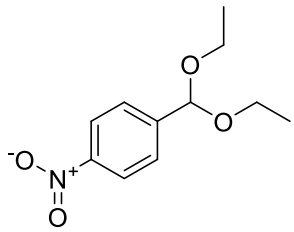
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, *J* = 8.8 Hz, 2H), 7.63 (d, *J* = 8.2 Hz, 2H), 5.46 (s, 1H), 3.32 (s, 6H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$) δ 148.1, 145.2, 127.9, 123.5, 101.7, 52.8 ppm;

IR (KBr) 2942, 2835, 2310, 2091, 1740, 1606, 1520 cm^{-1};

EI-MS: *m/z* (%) = 166.1 (100, [M - CH_3O]);

1-(Diethoxymethyl)-4-nitrobenzene (4dE): Prepared according to the general procedure from 4-nitrobenzaldehyde and triethylorthoformate to yield the title compound as a light-yellow oil (108 mg, 0.48 mmol, 95% yield).



4dE

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.17 (d, *J* = 8.8 Hz, 2H), 7.63 (d, *J* = 8.9 Hz, 2H), 5.54 (s, 1H), 3.64 – 3.45 (m, 4H), 1.21 (t, *J* = 7.2 Hz, 6H) ppm;

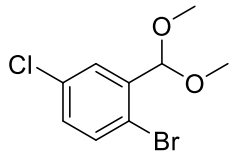
^{13}C NMR (151 MHz, $CDCl_3$) δ 147.9, 146.2, 127.8, 123.4, 100.2, 61.4, 15.2 ppm;

IR (KBr) 2977, 2929, 2882, 2114, 1737, 1606, 1522 cm^{-1};

EI-MS: *m/z* (%) = 226.1 (20, [M^+]), 180.1 (80, [M - CH_3CH_2OH]);

ESI-MS Anal. Calcd. for 248.0893 $C_{11}H_{15}O_4NNa$, found 248.0891.

1-Bromo-4-chloro-2-(dimethoxymethyl)benzene (4eM): Prepared according to the general procedure from 2-bromo-5-chlorobenzaldehyde and trimethylorthoformate to yield the title compound as a light-yellow oil (135 mg, 0.5 mmol, 98% yield).



4eM

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 2.6 Hz, 1H), 7.47 (d, *J* = 8.5 Hz, 1H), 7.17 (dd, *J* = 8.5, 2.6 Hz, 1H), 5.50 (s, 1H), 3.37 (s, 6H) ppm;

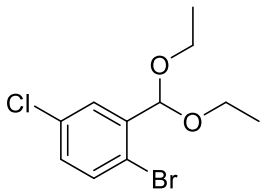
^{13}C NMR (101 MHz, $CDCl_3$) δ 138.7, 134.1, 133.6, 130.2, 128.8, 120.7, 102.3, 53.9 ppm;

IR (KBr) 2938, 2829, 2326, 2095, 1895, 1743 cm^{-1};

EI-MS: *m/z* (%) = 265.9 (12, [M^+]), 234.9 (88, [M - CH_3O]);

ESI-HRMS Anal. Calcd. for 263.9547 $C_9H_{10}O_2{}^{79}Br^{35}Cl$, found 263.9549.

1-Bromo-4-chloro-2-(diethoxymethyl)benzene (4eE): Prepared according to the general procedure from 2-bromo-5-chlorobenzaldehyde and triethylorthoformate to yield the title compound as a light-yellow oil (127 mg, 0.43 mmol, 86% yield).



4eE

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.63 (s, 1H), 7.45 (d, *J* = 9.5 Hz, 1H), 7.15 (d, *J* = 8.6 Hz, 1H), 5.59 (s, 1H), 3.62 (dt, *J* = 46.4, 7.9 Hz, 4H), 1.24 (t, *J* = 7.1 Hz, 6H) ppm;

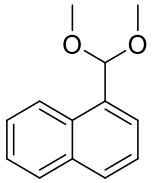
^{13}C NMR (151 MHz, $CDCl_3$) δ 139.8, 134.0, 133.7, 130.0, 128.6, 120.7, 100.8, 62.6, 15.3 ppm;

IR (KBr) 2975, 2883, 2320, 2096, 1888 cm^{-1};

EI-MS: *m/z* (%) = 294.0 (6, [^{81}Br M^+]), 292.0 (4, [^{79}Br M^+]), 249.0 (52, [^{81}Br M - CH_3CH_2O]) 247.0 (38, [^{79}Br M - CH_3CH_2O]);

ESI-HRMS Anal. Calcd. for 314.9758 $C_{11}H_{14}O_2BrClNa$, found 314.9759.

1-(Dimethoxymethyl)naphthalene (4fM): Prepared according to the general procedure from 1-naphthaldehyde and trimethylorthoformate to yield the title compound as a colourless oil (91 mg, 0.45 mmol, 90% yield).



4fM

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (d, *J* = 8.1 Hz, 1H), 7.88 (t, *J* = 9.5 Hz, 2H), 7.76 (d, *J* = 6.9 Hz, 1H), 7.60 – 7.45 (m, 3H), 5.95 (s, 1H), 3.41 (s, 6H) ppm;

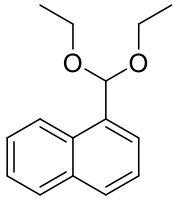
^{13}C NMR (101 MHz, $CDCl_3$) δ 133.9, 133.1, 130.9, 129.4, 128.6, 126.3, 125.8, 125.1, 124.9, 124.3, 102.5, 53.3 ppm;

IR (KBr) 2939, 2828, 2321, 2101, 1915, 1741 cm^{-1};

EI-MS: *m/z* (%) = 202.1 (40, [M^+]), 171.1 (60, [M - CH_3O]);

ESI-MS Anal. Calcd. for 225.0886 $C_{13}H_{14}O_2Na$, found 225.0884.

1-(Diethoxymethyl)naphthalene (4fE): Prepared according to the general procedure from 1-naphthaldehyde and triethylorthoformate to yield the title compound as a colourless oil (109 mg, 0.47 mmol, 95% yield).



4fE

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.39 (d, *J* = 8.6 Hz, 1H), 7.95 – 7.77 (m, 3H), 7.62 – 7.45 (m, 3H), 6.10 (d, *J* = 2.0 Hz, 1H), 3.87 – 3.48 (m, 4H), 1.30 (t, *J* = 7.9 Hz, 6H) ppm;

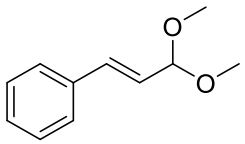
^{13}C NMR (151 MHz, $CDCl_3$) δ 134.1, 133.9, 130.9, 129.1, 128.5, 126.1, 125.6, 124.9, 124.7, 124.4, 100.6, 61.5, 15.4 ppm;

IR (KBr) 3051, 2973, 2881, 2325, 2104, 1913, 1741 cm^{-1};

EI-MS: *m/z* (%) = 230.2 (22, [M^+]), 185.2 (78, [M - CH_3CH_2O]);

ESI-HRMS Anal. Calcd. for 253.1199 $C_{15}H_{18}O_2Na$, found 253.1194.

(*E*)-(3,3-Dimethoxyprop-1-en-1-yl)benzene (4gM): Prepared according to the general procedure from cinnamaldehyde and trimethylorthoformate to yield the title compound as a colourless oil (66 mg, 0.37 mmol, 75% yield).



4gM

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.38 (m, 2H), 7.35 – 7.31 (m, 2H), 7.29 – 7.20 (m, 1H), 6.74 (d, *J* = 16.3 Hz, 1H), 6.15 (dd, *J* = 16.2, 4.9 Hz, 1H), 4.96 (dd, *J* = 4.9, 1.2 Hz, 1H), 3.38 (s, 6H) ppm;

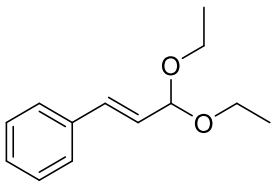
^{13}C NMR (101 MHz, $CDCl_3$) δ 136.2, 133.7, 128.7, 128.2, 126.9, 125.8, 103.0, 52.9 ppm;

IR (KBr) 2937, 2829, 2322, 2102, 1889, 1743 cm^{-1};

EI-MS: *m/z* (%) = 178.0 (21, [M^+]), 147.0 (79, [M - CH_3O]);

ESI-MS Anal. Calcd. for 201.0886 $C_{11}H_{14}O_2Na$, found 201.0882.

(*E*)-(3,3-Diethoxyprop-1-en-1-yl)benzene (4gE): Prepared according to the general procedure from cinnamaldehyde and triethylorthoformate to yield the title compound as a colourless oil (75 mg, 0.37 mmol, 71% yield).



4gE

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.38 (m, 2H), 7.36 – 7.28 (m, 2H), 7.27 – 7.20 (m, 1H), 6.70 (d, *J* = 16.1 Hz, 1H), 6.20 (dd, *J* = 16.1, 5.2 Hz, 1H), 5.06 (dd, *J* = 5.2, 1.2 Hz, 1H), 3.70 (ddq, *J* = 56.8, 9.4, 7.1 Hz, 4H), 1.25 (t, *J* = 7.1 Hz, 6H) ppm;

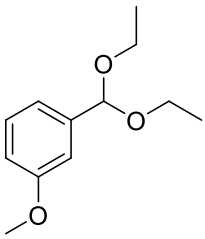
^{13}C NMR (101 MHz, $CDCl_3$) δ 136.3, 133.0, 128.7, 128.1, 126.9, 126.8, 101.6, 61.2, 15.4 ppm;

IR (KBr) 3455, 3104, 2240, 2182, 2121, 1978, 1739 cm^{-1};

EI-MS: *m/z* (%) = 206.2 (25, [M^+]), 161.1 (75, [M - CH_3CH_2O]);

ESI-MS Anal. Calcd. for 229.1199 $C_{13}H_{18}O_2Na$, found 229.1197.

1-(Diethoxymethyl)-3-methoxybenzene (4h): Prepared according to the general procedure from *m*-anisaldehyde and triethylorthoformate to yield the title compound as a colourless oil (90 mg, 0.43 mmol, 85% yield).



4h

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.27 (t, *J* = 7.5 Hz, 1H), 7.06 (s, 1H), 7.04 (s, 1H), 6.85 (d, *J* = 8.1 Hz, 1H), 5.47 (s, 1H), 3.81 (s, 3H), 3.58 (ddq, *J* = 53.2, 8.2, 7.2 Hz, 4H), 1.23 (t, *J* = 7.2 Hz, 6H) ppm;

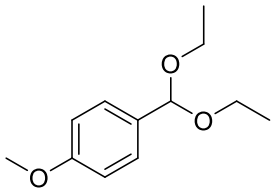
^{13}C NMR (151 MHz, $CDCl_3$) δ 159.7, 140.8, 129.3, 119.1, 114.1, 112.0, 101.5, 61.2, 55.3, 15.3 ppm;

IR (KBr) 2973, 2883, 2320, 2102, 1916, 1743, 1597 cm^{-1};

EI-MS: *m/z* (%) = 210.2 (10, [M^+]), 165.1 (90, [M - CH_3CH_2O]);

ESI-MS Anal. Calcd. for 233.1148 $C_{12}H_{18}O_3Na$, found 233.1143.

1-(Diethoxymethyl)-4-methoxybenzene (4i): Prepared according to the general procedure from *p*-anisaldehyde and triethylorthoformate to yield the title compound as a colourless oil (103 mg, 0.49 mmol, 95% yield).



4i

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.42 – 7.36 (m, 2H), 6.90 – 6.85 (m, 2H), 5.46 (s, 1H), 3.79 (s, 3H), 3.56 (ddq, *J* = 55.0, 9.6, 7.1 Hz, 4H), 1.26 (t, *J* = 7.2, 6H) ppm;

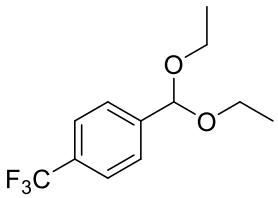
^{13}C NMR (151 MHz, $CDCl_3$) δ 159.6, 131.4, 127.9, 113.5, 101.4, 60.9, 55.3, 15.2 ppm;

IR (KBr) 2971, 2887, 2314, 2098, 1905, 1693, 1600, 1510 cm^{-1};

EI-MS: *m/z* (%) = 210.2 (10, [M^+]), 165.1 (90, [M - CH_3CH_2O]);

ESI-MS Anal. Calcd. for 233.1148 $C_{12}H_{18}O_3Na$, found 233.1145.

1-(Diethoxymethyl)-4-(trifluoromethyl)benzene (4j): Prepared according to the general procedure from 4-(trifluoromethyl)benzaldehyde and triethylorthoformate to yield the title compound as a colourless oil (94 mg, 0.38 mmol, 75% yield).



4j

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.71 – 7.47 (m, 4H), 5.54 (s, 1H), 3.58 (ddq, *J* = 37.5, 9.6, 7.0 Hz, 4H), 1.25 (t, *J* = 7.1 Hz, 6H) ppm;

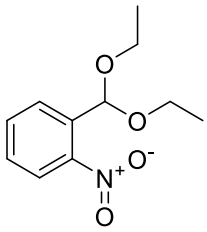
^{13}C NMR (151 MHz, $CDCl_3$) δ 143.0, 130.6 (q, *J* = 31.9 Hz, 1C), 127.1, 125.3 (q, *J* = 3.8 Hz, 1C), 123.2, 100.6, 61.1, 15.1 ppm;

IR (KBr) 2976, 2885, 2321, 2097, 1915 cm^{-1};

EI-MS: *m/z* (%) = 203.1 (100, [M - CH_3CH_2O]);

ESI-MS Anal. Calcd. for 271.0916 $C_{12}H_{15}O_2F_3Na$, found 271.0910.

1-(Diethoxymethyl)-2-nitrobenzene (4k): Prepared according to the general procedure from 2-nitrobenzaldehyde and triethylorthoformate to yield the title compound as a light-yellow oil (99 mg, 0.44 mmol, 89% yield).



4k

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.79 (ddd, *J* = 19.6, 7.9, 1.4 Hz, 2H), 7.57 (td, *J* = 7.6, 1.3 Hz, 1H), 7.43 (td, *J* = 7.7, 1.5 Hz, 1H), 6.01 (s, 1H), 3.64 (ddq, *J* = 66.3, 9.4, 7.0 Hz, 4H), 1.21 (t, *J* = 7.1 Hz, 6H) ppm;

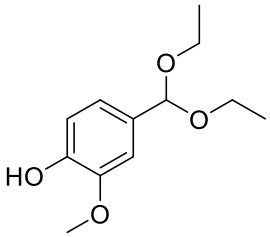
^{13}C NMR (151 MHz, $CDCl_3$) δ 149.1, 133.7, 132.5, 129.2, 128.0, 124.2, 98.4, 63.5, 15.1 ppm;

IR (KBr) 2976, 2886, 2326, 2097, 1905, 1739, 1529 cm^{-1};

EI-MS: *m/z* (%) = 180.0 (100, [M - CH_3CH_2O]);

ESI-MS Anal. Calcd. for 248.0893 $C_{11}H_{15}O_4NNa$, found 248.0892.

4-(Diethoxymethyl)-2-methoxyphenol (4l): Prepared according to the general procedure from vanillin and triethylorthoformate to yield the title compound as a colourless oil (80 mg, 0.35 mmol, 69% yield).



4l

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.01 (d, *J* = 1.9 Hz, 1H), 6.95 (dd, *J* = 8.1, 1.9 Hz, 1H), 6.88 (d, *J* = 8.1 Hz, 1H), 5.74 (s, 1H), 5.41 (s, 1H), 3.89 (s, 3H), 3.57 (ddq, *J* = 63.9, 9.4, 7.0 Hz, 4H), 1.23 (t, *J* = 7.2 Hz, 6H) ppm;

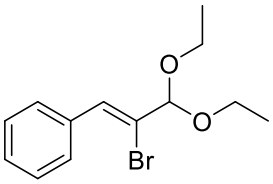
^{13}C NMR (151 MHz, $CDCl_3$) δ 146.6, 145.8, 131.4, 119.9, 113.9, 108.9, 101.8, 61.2, 56.0, 15.3 ppm;

IR (KBr) 3388, 2971, 2885, 2309, 2100, 1917, 1678, 1593, 1514 cm^{-1};

EI-MS: *m/z* (%) = 226.2 (20, [M^+]), 181.1 (80, [M - CH_3CH_2O]);

ESI-MS Anal. Calcd. for 249.1097 $C_{12}H_{18}O_4Na$, found 249.1095.

(2-Bromo-3,3-diethoxyprop-1-en-1-yl)benzene (4m): Prepared according to the general procedure from *α*-bromocinnamaldehyde and triethylorthoformate to yield the title compound as a light-yellow oil (94 mg, 0.33 mmol, 66% yield).



4m

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.4 Hz, 2H), 7.37 (t, *J* = 7.5 Hz, 2H), 7.33 (d, *J* = 7.3 Hz, 1H), 7.30 (s, 1H), 4.98 (s, 1H), 3.66 (ddq, *J* = 61.1, 9.4, 7.0 Hz, 4H), 1.29 (t, *J* = 7.1 Hz, 6H) ppm;

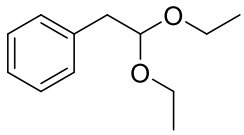
^{13}C NMR (151 MHz, $CDCl_3$) δ 134.8, 130.0, 129.4, 128.5, 128.2, 122.8, 103.2, 62.1, 15.2 ppm;

IR (KBr) 3058, 3025, 2975, 2927, 2882, 2110, 1737, 1641 cm^{-1};

EI-MS: *m/z* (%) = 286.1 (10, [^{81}Br M^+]), 284.1 (9, [^{79}Br M^+]), 241.1 (41, [^{81}Br M - CH_3CH_2O]), 239.1 (40, [^{79}Br M - CH_3CH_2O]);

ESI-HRMS Anal. Calcd. for 307.0304 $C_{13}H_{17}O_2BrNa$, found 307.0302.

(2,2-Diethoxyethyl)benzene (4n): Prepared according to the general procedure from 2-phenylacetaldehyde and triethylorthoformate to yield the title compound as a colourless oil (33 mg, 0.17 mmol, 36% yield).



4n

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.30 – 7.24 (m, 3H), 7.23 – 7.19 (m, 2H), 4.63 (t, *J* = 5.7 Hz, 1H), 3.56 (ddq, *J* = 135.0, 9.3, 7.0 Hz, 4H), 2.92 (d, *J* = 5.7 Hz, 2H), 1.17 (t, *J* = 7.1 Hz, 6H) ppm;

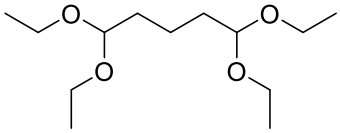
^{13}C NMR (151 MHz, $CDCl_3$) δ 137.5, 129.7, 128.3, 126.4, 104.0, 62.0, 41.0, 15.4 ppm;

IR (KBr) 3028, 2973, 2883, 2323, 2103, 1900, 1739, 1601 cm^{-1};

EI-MS: *m/z* (%) = 194.1 (66, [M^+]), 148.1 (34, [M - CH_3CH_2OH]);

ESI-HRMS Anal. Calcd. for 217.1199 $C_{12}H_{18}O_2Na$, found 217.1194.

1,1,5,5-Tetraethoxypentane (4o): Prepared according to the general procedure from glutaraldehyde and triethylorthoformate to yield the title compound as a colourless oil (10 mg, 0.04 mmol, 15% yield).



4o

^{1}H NMR (600 MHz, $CDCl_3$) δ 4.47 (t, *J* = 5.7 Hz, 2H), 3.55 (ddq, *J* = 91.0, 9.3, 7.0 Hz, 8H), 1.65 – 1.59 (m, 4H), 1.45 – 1.38 (m, 2H), 1.19 (t, *J* = 7.1 Hz, 12H) ppm;

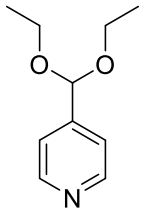
^{13}C NMR (151 MHz, $CDCl_3$) δ 102.9, 61.1, 33.5, 20.2, 15.5 ppm;

IR (KBr) 2968, 2879, 2328, 2105, 1901, 1737 cm^{-1};

EI-MS: *m/z* (%) = 103.1 (100, [M – $(CH_3CH_2O)_2CHCH_2CH_2CH_2$]);

ESI-MS Anal. Calcd. for 271.1880 $C_{13}H_{28}O_4Na$, found 271.1877.

4-(diethoxymethyl)pyridine (4s): Prepared according to the general procedure from 4-pyridinecarboxaldehyde and triethylorthoformate to yield the title compound as a colourless oil (8 mg, 0.05 mmol, 9% yield).



4s

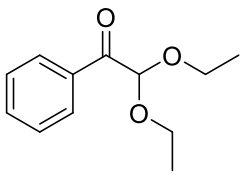
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.61 (d, *J* = 6.6 Hz, 2H), 7.39 (d, *J* = 6.7 Hz, 2H), 5.49 (s, 1H), 3.68 – 3.50 (m, 4H), 1.25 (t, *J* = 6.9 Hz, 6H) ppm;

^{13}C NMR (151 MHz, $CDCl_3$) δ 150.0, 147.7, 121.8, 99.9, 61.3, 15.3 ppm;

IR (KBr) 2975, 2924, 2883, 1736, 1601 cm^{-1};

ESI-MS Anal. Calcd. for 182.1176 $C_{10}H_{16}NO_2$, found 182.1173.

2,2-Diethoxy-1-phenylethan-1-one[2] (4u): Prepared according to the general procedure from phenylglyoxal hydrate and triethylorthoformate to yield the title compound as a light-yellow oil (46 mg, 0.24 mmol, 44% yield).



4u

[2] F. Malmedy, T. Wirth, *Chem. Eur. J.* **2016**, *22*, 16072-16077.

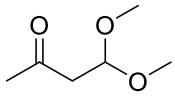
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.17 – 8.13 (m, 2H), 7.59 – 7.52 (m, 1H), 7.47 – 7.42 (m, 2H), 5.27 (s, 1H), 3.70 (ddq, *J* = 62.6, 9.5, 7.1 Hz, 4H), 1.24 (t, *J* = 7.1 Hz, 6H) ppm;

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.1, 133.9, 133.6, 129.9, 128.5, 102.5, 63.3, 15.3 ppm;

IR (KBr) 2976, 2885, 2322, 2108, 1912, 1692, 1594 cm^{-1};

EI-MS: *m/z* (%) = 163.2 (100, [M - CH_3CH_2O]);

4,4-Dimethoxybutan-2-one[3] (4v): Prepared according to the general procedure from 3-oxobutanal and trimethylorthoformate to yield the title compound as a colourless oil (46 mg, 0.35 mmol, 69% yield).



4v

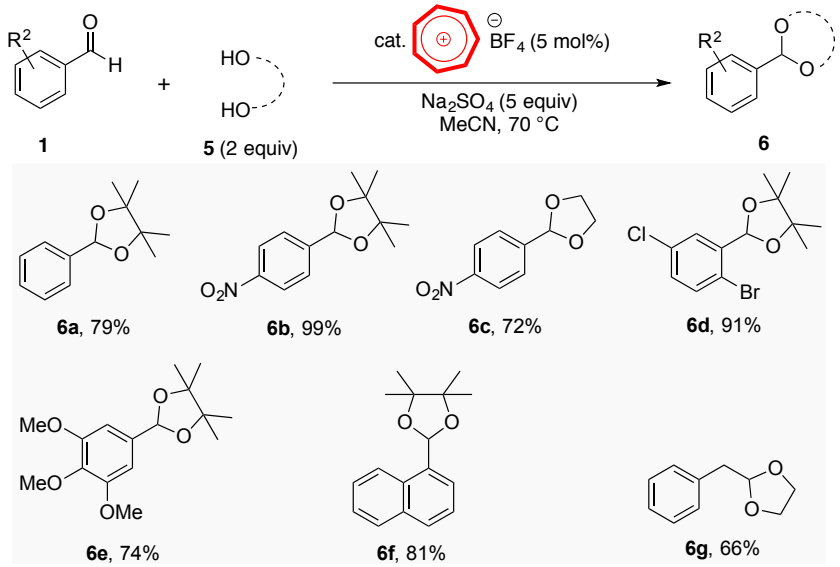
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.78 (t, *J* = 5.6 Hz, 1H), 3.35 (s, 6H), 2.75 – 2.69 (d, *J* = 5.6 Hz, 2H), 2.18 (s, 3H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$) δ 205.67, 101.62, 53.95, 47.42, 31.21 ppm.

[3] Z.-Y. Wang, H.-F. Jiang, C.R. Qi, Y.G. Wang, Y.S. Dong, H.L. Liu, *Green Chem.* **2005**, *7*, 582-585.

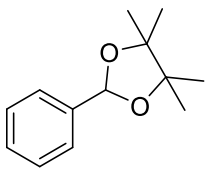
Cyclic Acetalization Reactions and Transacetalization Reactions (Scheme 3)

General procedure for cyclic acetalization reaction:



A mixture of aldehyde **1** (0.5 mmol), diol **5** (1.0 mmol), tropylium tetrafluoroborate **3** (0.025 mmol) and sodium sulphate (2.5 mmol) was taken up in dry acetonitrile (0.6 mL) in a vial loaded with a stirrer bar under an argon atmosphere. The reaction mixture was heated to 70 °C for five hours then cooled to room temperature. The crude reaction mixture was and concentrated under reduced pressure. The product was purified from the residues by column chromatography (silica-gel, pentane/diethylether/triethylamine = 78/20/2) unless otherwise noted.

4,4,5,5-Tetramethyl-2-phenyl-1,3-dioxolane (6a): Prepared according to the general procedure from benzaldehyde and pinacol to yield the title compound as a white solid (88 mg, 0.43 mmol, 79% yield).



6a

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.48 (m, 2H), 7.41 – 7.29 (m, 3H), 6.00 (s, 1H), 1.34 (s, 6H), 1.29 (s, 6H) ppm;

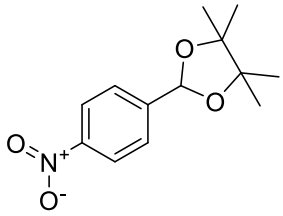
^{13}C NMR (101 MHz, $CDCl_3$) δ 139.8, 128.7, 128.4, 126.4, 100.0, 82.8, 24.5, 22.3 ppm;

IR (KBr) 3035, 2981, 2932, 2869, 1740 cm^{-1};

EI-MS: *m/z* (%) = 206.2 (100, $[M^+]$);

ESI-MS Anal. Calcd. for 229.1199 $C_{13}H_{18}O_2Na$, found 229.1196.

4,4,5,5-Tetramethyl-2-(4-nitrophenyl)-1,3-dioxolane (6b): Prepared according to the general procedure from 4-nitrobenzaldehyde and pinacol to yield the title compound as a white solid (124 mg, 0.49 mmol, 99% yield).



6b

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 – 8.18 (m, 2H), 7.71 – 7.61 (m, 2H), 6.01 (s, 1H), 1.33 (s, 6H), 1.22 (s, 6H) ppm;

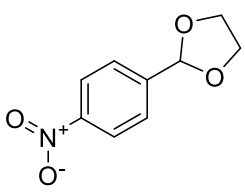
^{13}C NMR (101 MHz, $CDCl_3$) δ 147.4, 127.1, 123.6, 98.6, 83.4, 24.2, 22.3 ppm (C4 not observed);

IR (KBr) 3113, 3084, 2979, 2936, 2863, 2072, 1993, 1738, 1605, 1516 cm^{-1};

EI-MS: *m/z* (%) = 251.1 (100, $[M^+]$);

ESI-MS Anal. Calcd. for 274.1050 $C_{13}H_{17}O_4NNa$, found 274.1046.

2-(4-Nitrophenyl)-1,3-dioxolane (6c): Prepared according to the general procedure from 4-nitrobenzaldehyde and ethylene glycol to yield the title compound as a white solid (72% conversion calculated by ^{1}H NMR).



6c

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 – 8.17 (m, 2H), 7.69 – 7.57 (m, 2H), 5.86 (s, 1H), 4.17 – 4.01 (m, 4H) ppm;

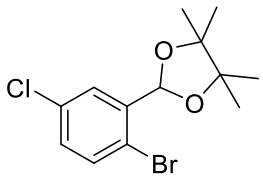
^{13}C NMR (101 MHz, $CDCl_3$) δ 148.5, 145.1, 127.5, 123.7, 102.3, 65.6 ppm;

IR (KBr) 3086, 2973, 2891, 2096, 1923, 1736, 1608, 1514 cm^{-1};

EI-MS: *m/z* (%) = 194.0 (100, [M^+]);

ESI-MS Anal. Calcd. for 196.0604 $C_9H_{10}O_4N$, found 196.0601.

2-(2-Bromo-5-chlorophenyl)-4,4,5,5-tetramethyl-1,3-dioxolane (6d): Prepared according to the general procedure from 2-bromo-5-chlorobenzaldehyde and pinacol to yield the title compound as a white solid (145 mg, 0.45 mmol, 91% yield).



6d

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.65 (d, *J* = 2.7 Hz, 1H), 7.46 (dd, *J* = 8.5, 1.1 Hz, 1H), 7.16 (ddd, *J* = 8.5, 2.6, 1.1 Hz, 1H), 6.11 (s, 1H), 1.34 (s, 6H), 1.29 (s, 6H) ppm;

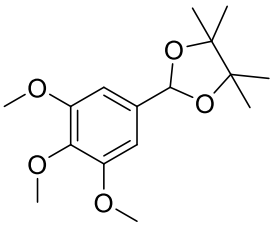
^{13}C NMR (101 MHz, $CDCl_3$) δ 140.1, 134.1, 133.7, 130.2, 128.1, 121.1, 98.4, 83.3, 24.5, 22.3 ppm;

IR (KBr) 3083, 2981, 2932, 2884, 2325, 2111, 1897, 1742 cm^{-1};

EI-MS: *m/z* (%) = 318.0 (100, [M^+]), (, [M - CH_3CH_2O]);

ESI-MS Anal. Calcd. for 340.9914 $C_{13}H_{16}O_2BrClNa$, found 340.9910.

4,4,5,5-Tetramethyl-2-(3,4,5-trimethoxyphenyl)-1,3-dioxolane (6e): Prepared according to the general procedure from 3,4,5-trimethoxybenzaldehyde and pinacol to yield the title compound as a white solid (109 mg, 0.37 mmol, 74% yield).



6e

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.74 (s, 2H), 5.92 (s, 1H), 3.87 (s, 6H), 3.83 (s, 3H), 1.33 (s, 6H), 1.29 (s, 6H) ppm;

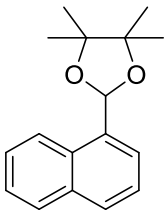
^{13}C NMR (101 MHz, $CDCl_3$) δ 153.4, 138.4, 135.5, 103.4, 100.1, 82.9, 61.0, 56.3, 24.6, 22.4 ppm;

IR (KBr) 2981, 2325, 2103, 1589, 1503 cm^{-1};

EI-MS: *m/z* (%) = 196.2 (100, [M^+]);

ESI-MS Anal. Calcd. for 319.1516 $C_{16}H_{24}O_5Na$, found 319.1513.

4,4,5,5-Tetramethyl-2-(naphthalen-1-yl)-1,3-dioxolane (6f): Prepared according to the general procedure from 1-naphthaldehyde and pinacol to yield the title compound as a white solid (105 mg, 0.41 mmol, 81% yield).



6f

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, *J* = 8.4, Hz, 1H), 7.86 (d, *J* = 7.45 Hz, 2H), 7.83 (d, *J* = 8.2 Hz, 1H), 7.54 (dd, (*J* = 6.8, 1.5 Hz, 1H), 7.53 – 7.44 (m, 2H), 6.67 (s, 1H), 1.43 (s, 6H), 1.30 (s, 6H) ppm;

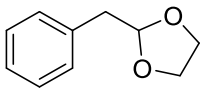
^{13}C NMR (101 MHz, $CDCl_3$) δ 135.6, 133.8, 131.2, 128.9, 128.6, 126.3, 125.7, 125.3, 124.2, 122.6, 97.7, 82.8, 24.3, 22.4 ppm;

IR (KBr) 3055, 2977, 2868, 2314, 2103, 1925, 1596, 1510 cm^{-1};

EI-MS: *m/z* (%) = 256.2 (100, [M^+]);

ESI-MS Anal. Calcd. for 279.1356 $C_{17}H_{20}O_2Na$, found 279.1356.

2-Benzyl-1,3-dioxolane[4] (6g): Prepared according to the general procedure from 2-phenylacetaldehyde and ethylene glycol to yield the title compound as a colorless oil (54 mg, 0.33 mmol, 66% yield).

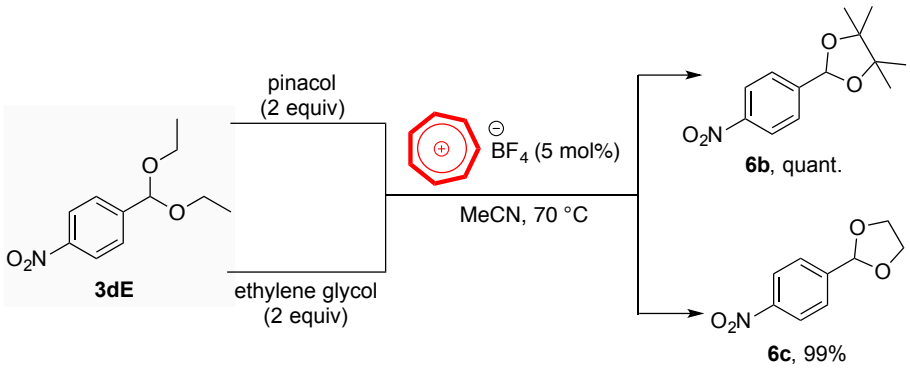


6g

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.15 (m, 5H), 5.05 (t, *J* = 4.8 Hz, 1H), 3.98 – 3.76 (m, 4H), 2.95 (d, *J* = 4.8 Hz, 2H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$) δ 136.28, 129.79, 128.47, 126.73, 104.82, 65.12, 40.93 ppm;

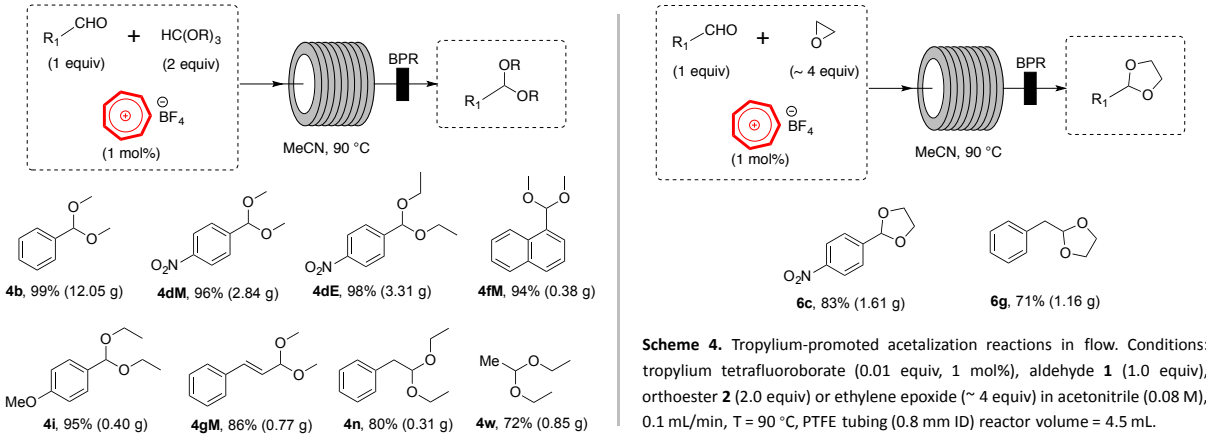
General procedure for transacetalization reaction:



A mixture of 1-(diethoxymethyl)-4-nitrobenzene **4dE** (0.5 mmol), diol **5** (1.0 mmol) and tropylium tetrafluoroborate (0.025 mmol) was taken up in dry acetonitrile (0.6 mL) in a vial loaded with a stirrer bar under argon atmosphere. The reaction mixture was heated to 70 °C for five hours then cooled to room temperature. ^{1}H NMR spectra confirmed that no acyclic acetal was present in the crude reaction mixture. It was concentrated under reduced pressure and purified by column chromatography.

[4] I. Kondolff, H. Doucet, M. Santelli, Eur. *J. Org. Chem.* **2006**, 765-774.

General Settings for Acetalization Reactions in Flow



Scheme 4. Tropylium-promoted acetalization reactions in flow. Conditions: tropylium tetrafluoroborate (0.01 equiv, 1 mol%), aldehyde **1** (1.0 equiv), orthoester **2** (2.0 equiv) or ethylene epoxide (~ 4 equiv) in acetonitrile (0.08 M), 0.1 mL/min, T = 90 °C, PTFE tubing (0.8 mm ID) reactor volume = 4.5 mL.

Optimization of catalyst loading for flow chemistry: The acetalization reactions of *p*-nitrobenzaldehyde to form 3dE with 2.5 and 1 mol% tropylium tetrafluoroborate in flow gave comparable results to the reaction with 5 mol% catalyst loading so we carried out all other reactions using only 1 mol% catalyst.

Catalyst loading	Yield of **3dE**
5 mol%	99%
2.5 mol%	99%
1 mol%	98%

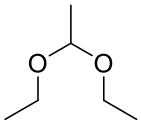
Reaction conditions: A solution of tropylium tetrafluoroborate (0.01 equiv, 1 mol%), aldehyde **1** (1.0 equiv), orthoester **2** (2.0 equiv) or ethylene epoxide (~ 4 equiv, concentration determined by ^{1}H NMR) in acetonitrile (0.08 M) was pumped through a coil of tubular reactor submerged in an oil bath maintained at T = 90 °C. The reaction mixture at the output was collected and worked up in similar fashion to batch reaction.

Equipment: Pumps = Little Things Factory GmbH Mr. Q continuous syringe pump (0.1 mL/min); PTFE tubing (0.8 mm ID) reactor volume = 4.5 mL. Pressure regulator = Upchurch Scientific™ 100 psi.

Characterization of Flow Chemistry Acetal Products

Characterization data of compounds **4b-4n**, **6c** and **6g** (Scheme 4) synthesized in flow are similar to data reported above for these compounds synthesized in batch.

1,1-Diethoxyethane[5] (4w): Prepared according to the general procedure from acetaldehyde and triethylorthoformate to yield the title compound as a colourless oil after purification by distillation (0.85 g, 7.19 mmol, 72% yield).



4w

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.67 (q, *J* = 5.4 Hz, 1H), 3.54 (ddq, *J* = 62.7, 9.4, 7.1 Hz, 4H), 1.29 (d, *J* = 5.3 Hz, 3H), 1.18 (t, *J* = 7.1 Hz, 6H) ppm;

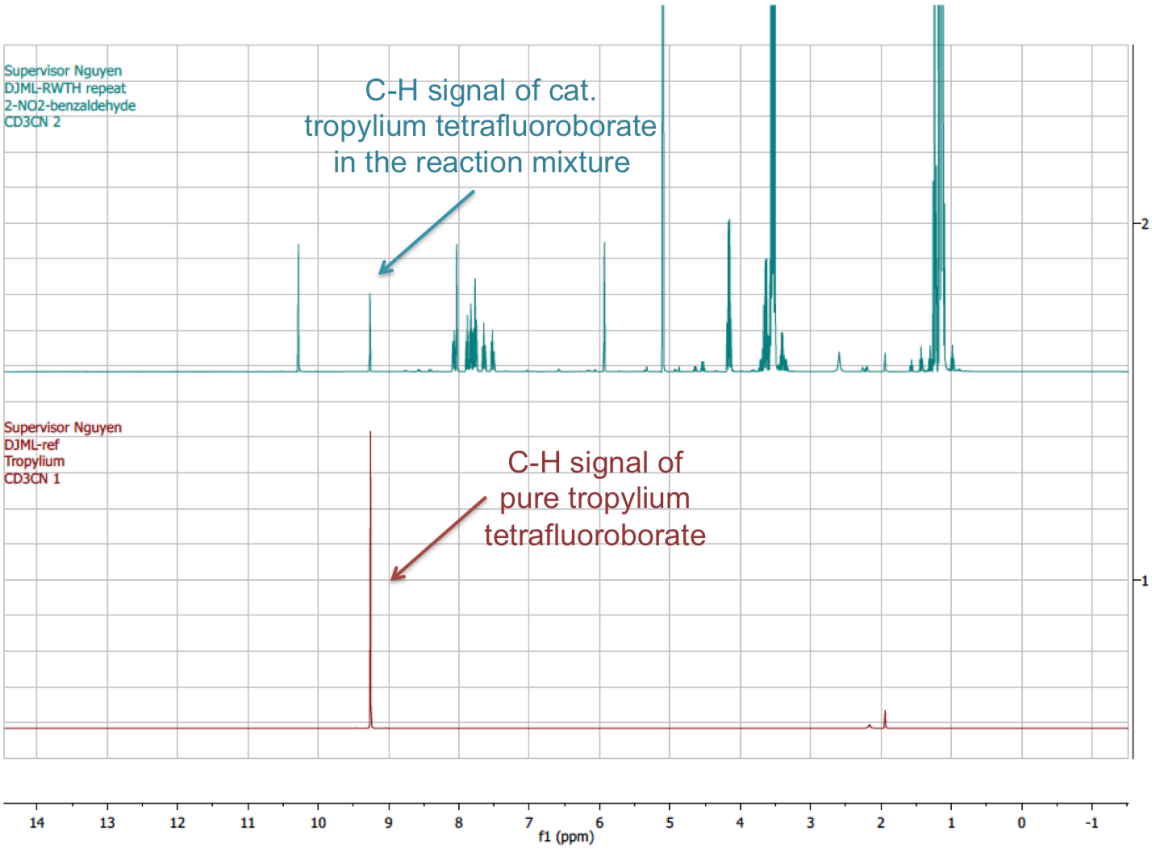
^{13}C NMR (101 MHz, $CDCl_3$) δ 99.52, 60.73, 20.05, 15.43 ppm.

[5] H. Zhang, Z. Zhu, Y. Wu, T. Zhao, L. Li, *Green Chem.* **2014**, *16*, 4076-4-80.

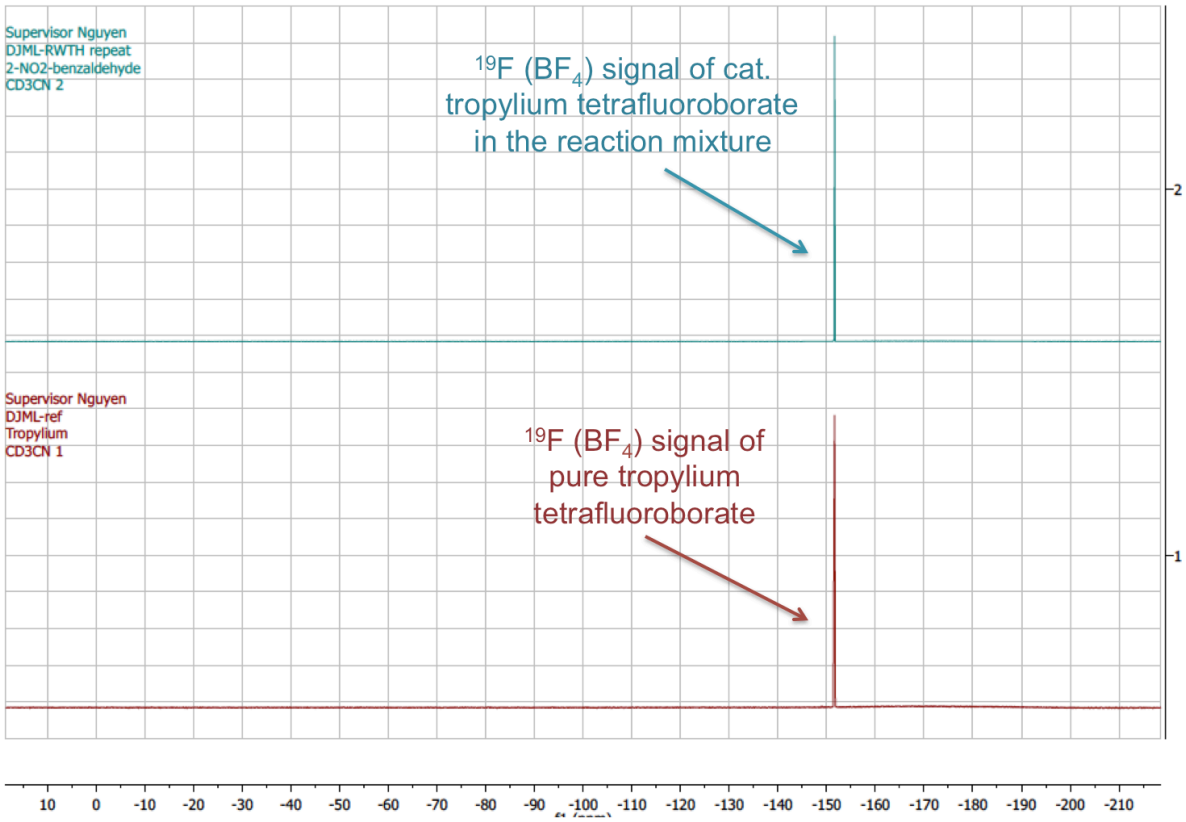
Possibility of Recycling Tropylium Tetrafluoroborate Catalyst after Reaction

In both batch and flow conditions, we could still observe the characteristic signals of tropylium tetrafluoroborate catalyst after reactions by ^{1}H and ^{19}F NMR spectroscopy. Integration of ^{1}H NMR signals confirmed that the tropylium catalyst remained at ~ 90-100% of the initial catalyst loading. The tropylium catalyst could be recollected from the reaction mixture by column chromatography or recrystallization.

An example of the tropylium tetrafluoroborate catalyst remained in the reaction mixture can be observed in the ^{1}H and ^{19}F NMR spectra (in CD_3CN at 298K) of the tropylium-catalyzed (5 mol%) between 2-nitrobenzaldehyde and triethyl orthoformate to form acetal **4k**.

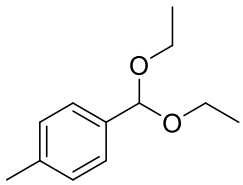


^{1}H NMR (400 MHz, CD_3CN, 298K): (top) Crude reaction mixture of the tropylium-catalyzed acetalization reaction of 2-nitrobenzaldehyde with triethyl orthoformate; (bottom) Pure tropylium tetrafluoroborate

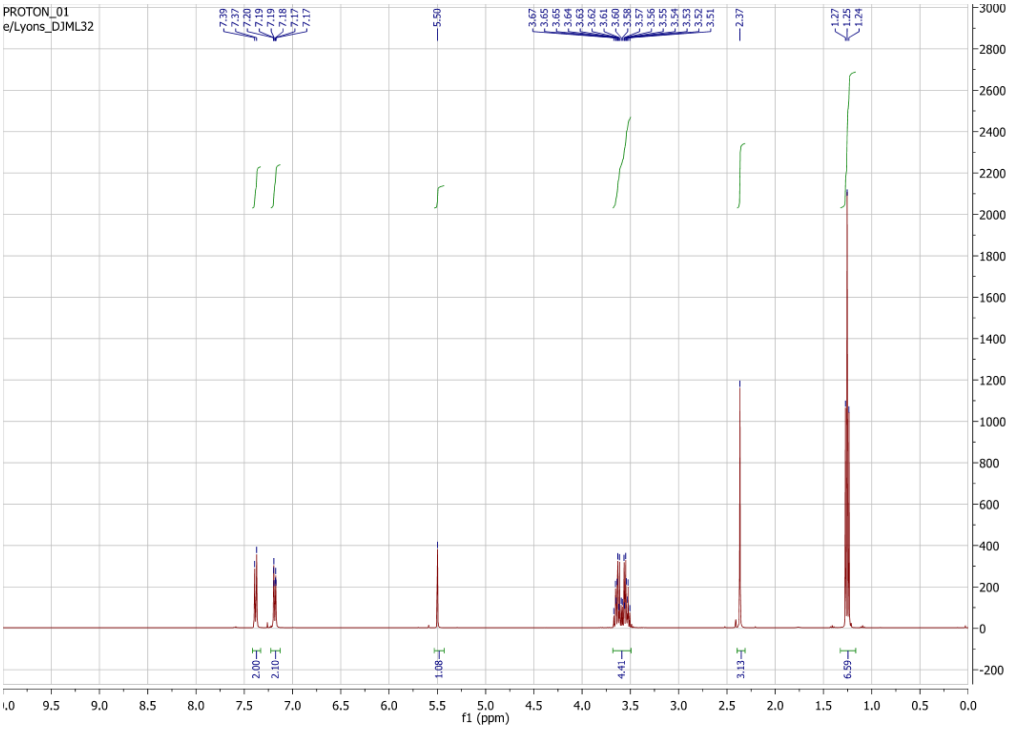


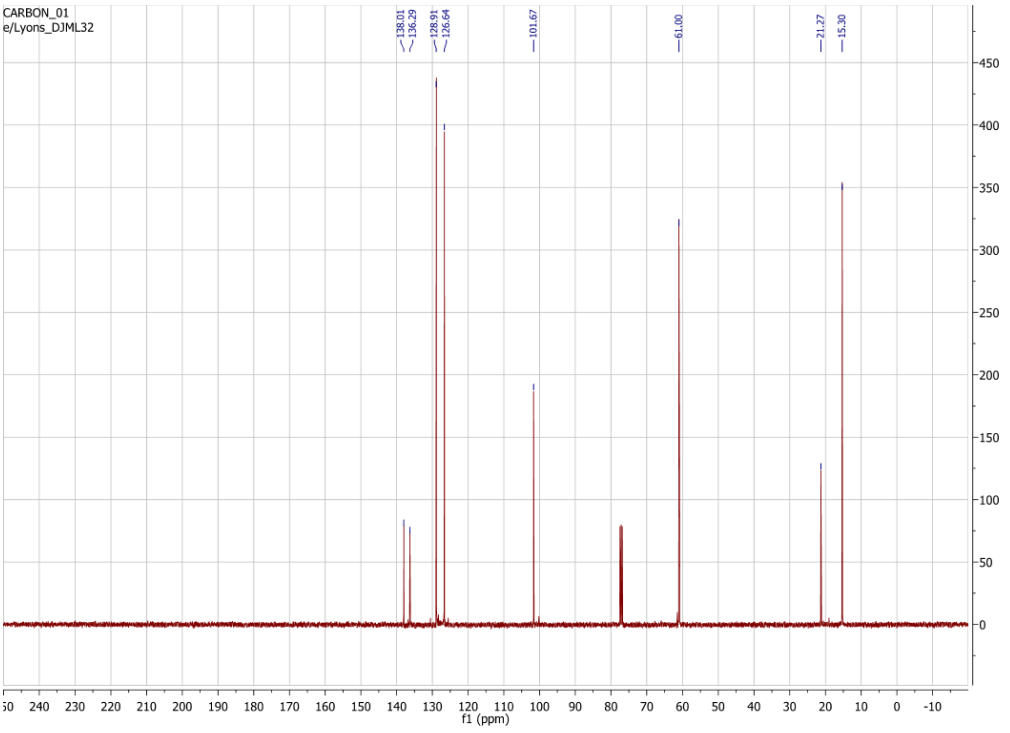
^{19}F NMR (376.5 MHz, CD_3CN, 298K): (top) Crude reaction mixture of the tropylium-catalyzed acetalization reaction of 2-nitrobenzaldehyde with triethyl orthoformate; (bottom) Pure tropylium tetrafluoroborate

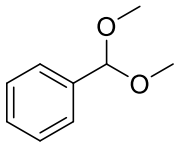
NMR Spectra of Acetal Products



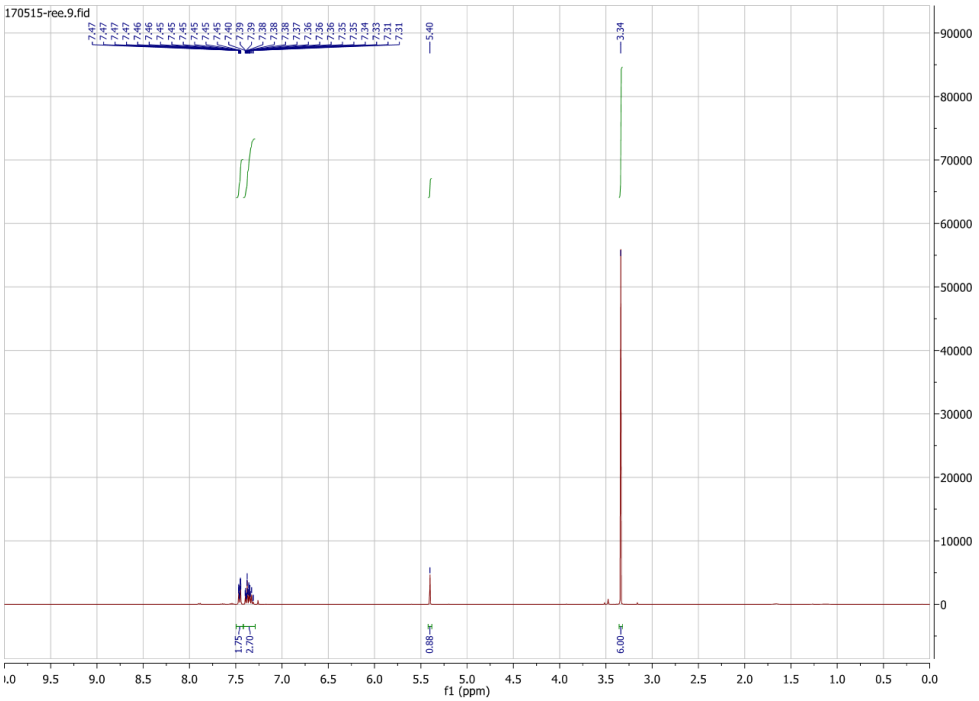
4a







4b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7.47
7.47
7.47
7.46
7.46
7.45
7.45
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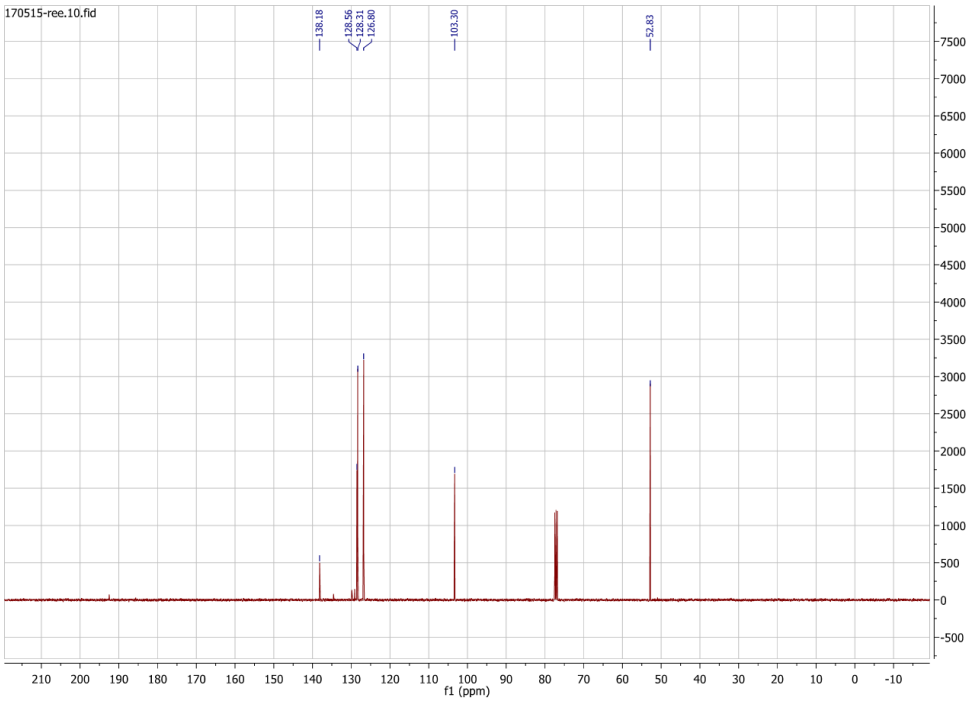
7.45
7.45
7.40
7.39
7.38
7.38
7.38
7.37
7.36
7.36
7.35
7.35
7.34
7.33
7.31
7.31
5.40
3.34
1.75
2.70
0.88
6.00
f1 (ppm)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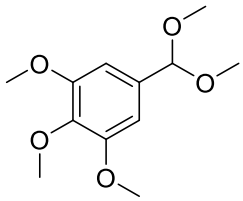
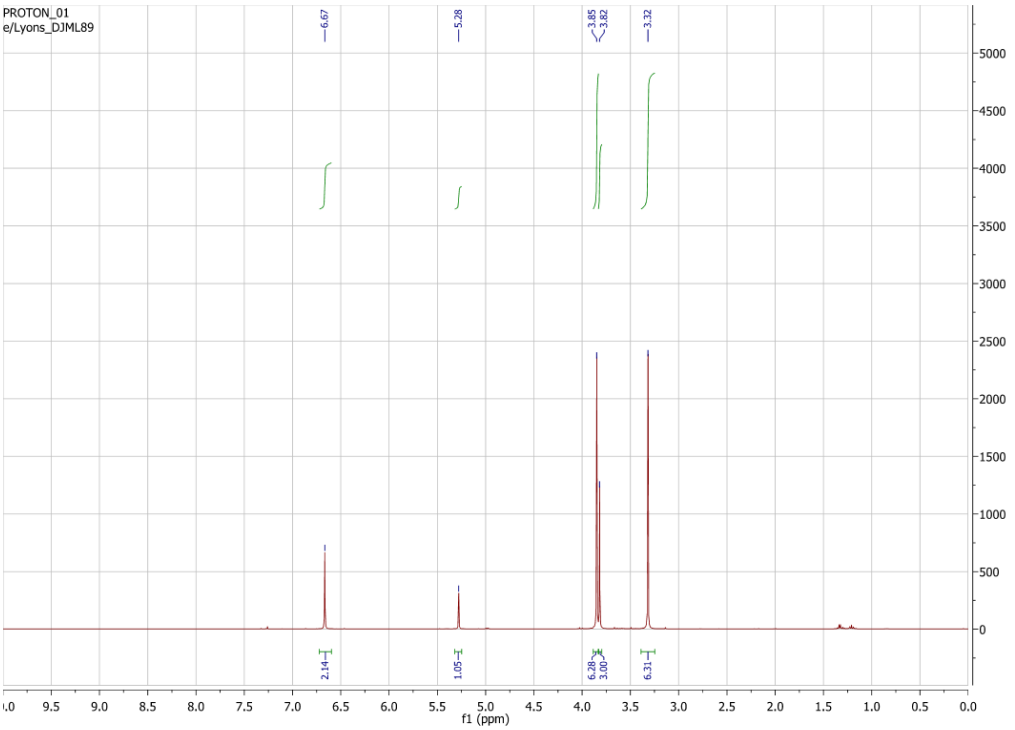
126.80
103.30
52.83
f1 (ppm)



4cM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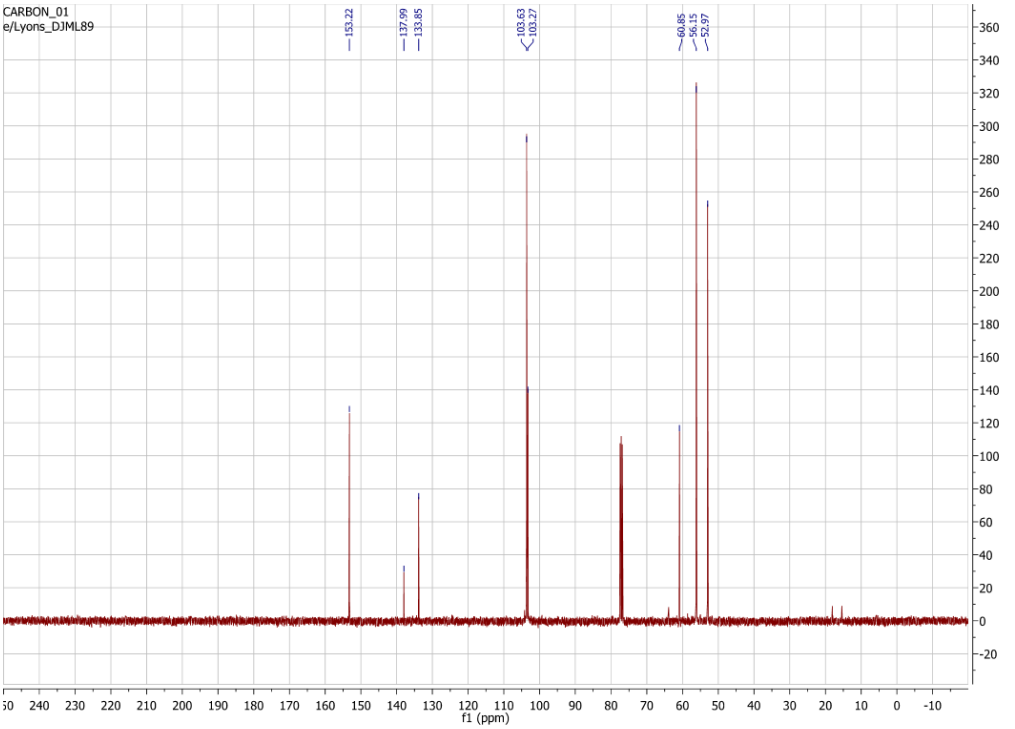
3.32
2.14
1.05
6.28
3.00
6.31
f1 (ppm)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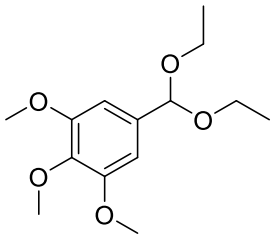
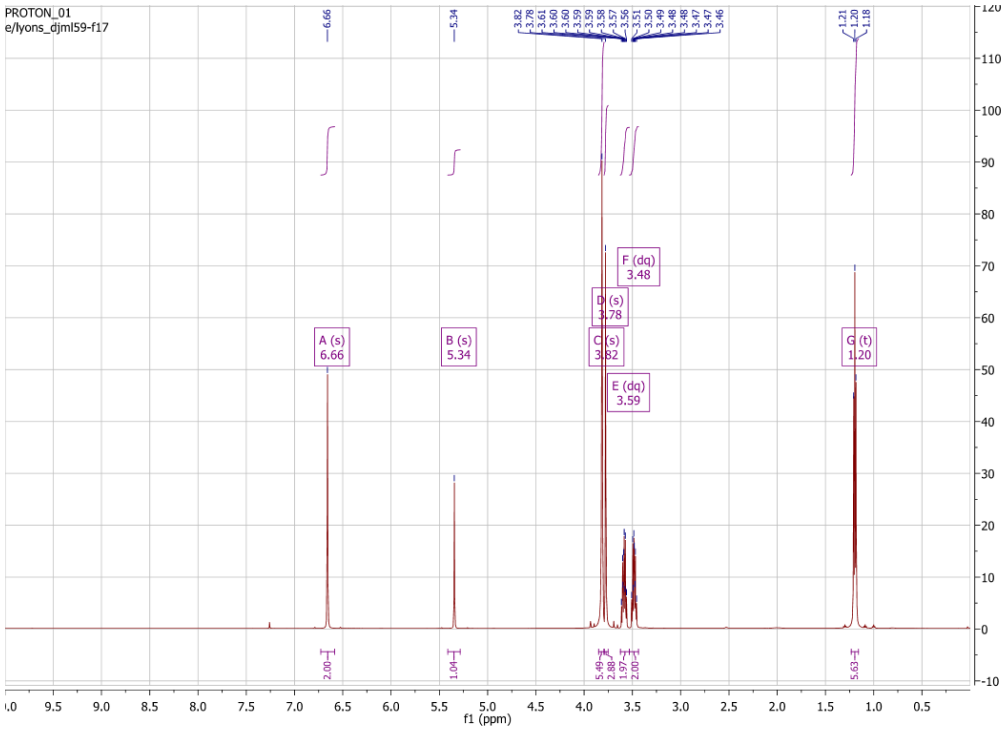
103.63
103.27
60.85
56.15
52.57
f1 (ppm)



4cE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D (s)
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E (dq)
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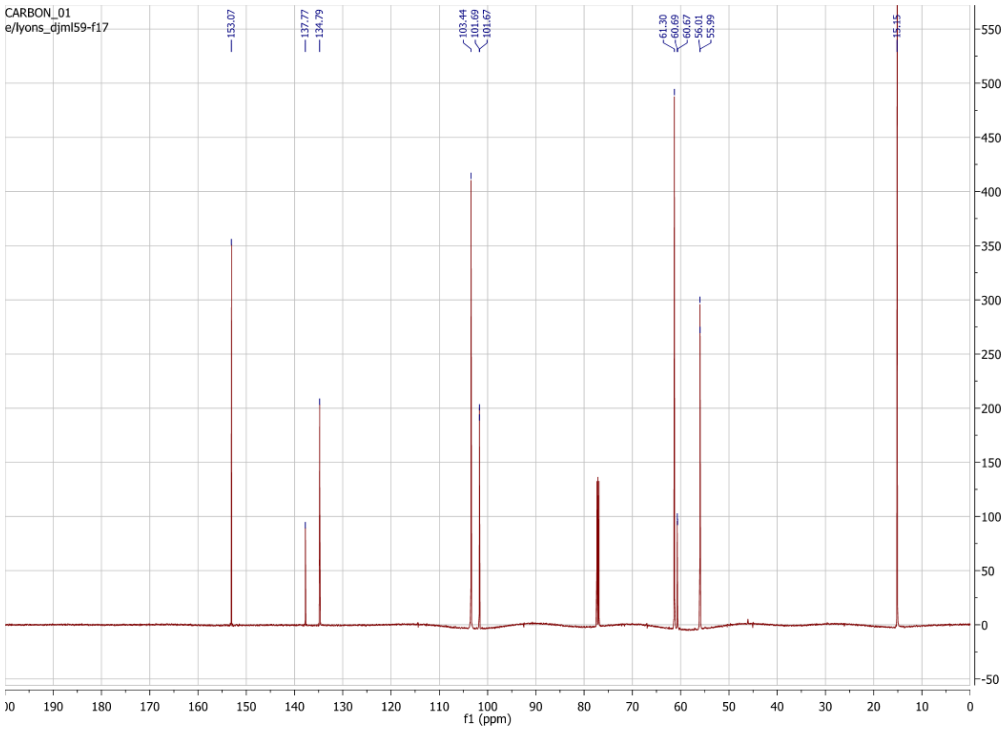
F (dq)
3.48
G (t)
1.20
f1 (ppm)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60.69
60.67
56.01
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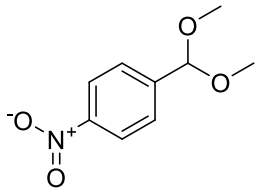
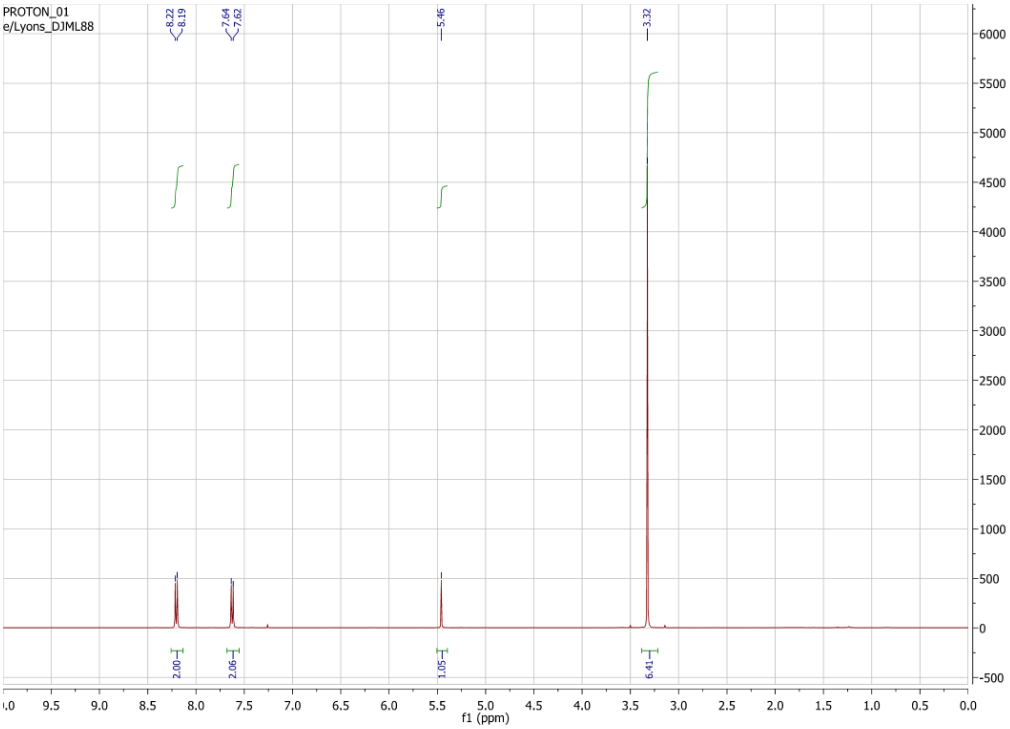
15.15
f1 (ppm)



4dM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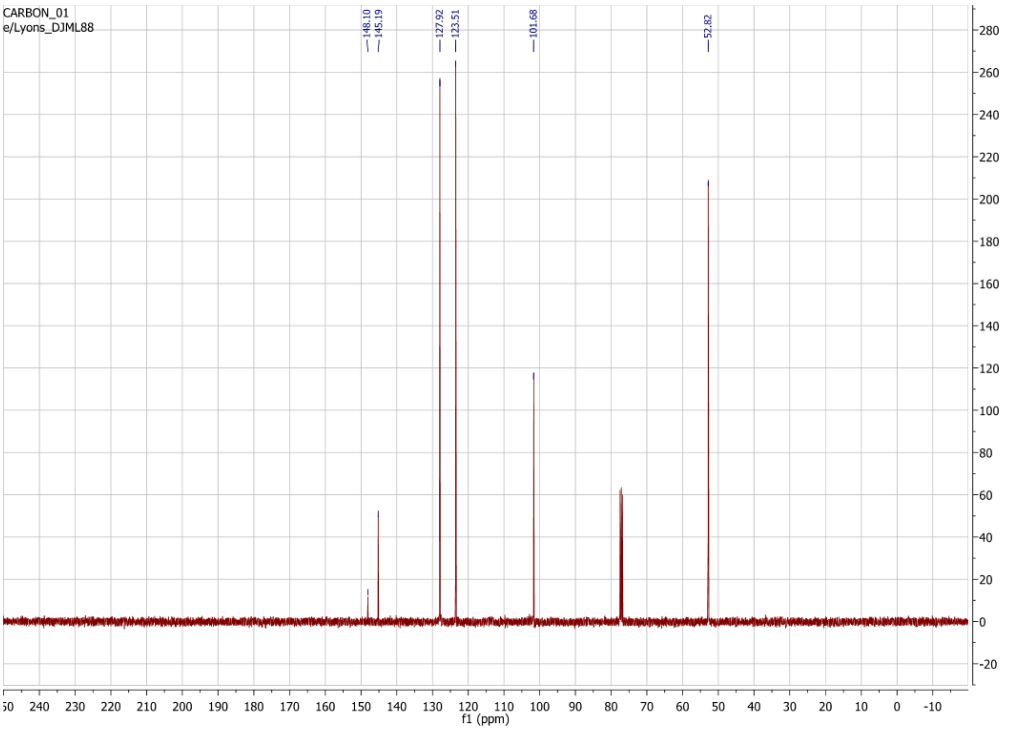
2.00
2.06
1.05
6.41
f1 (ppm)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145.19
127.92
123.51
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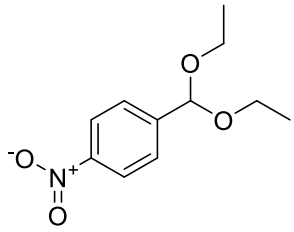
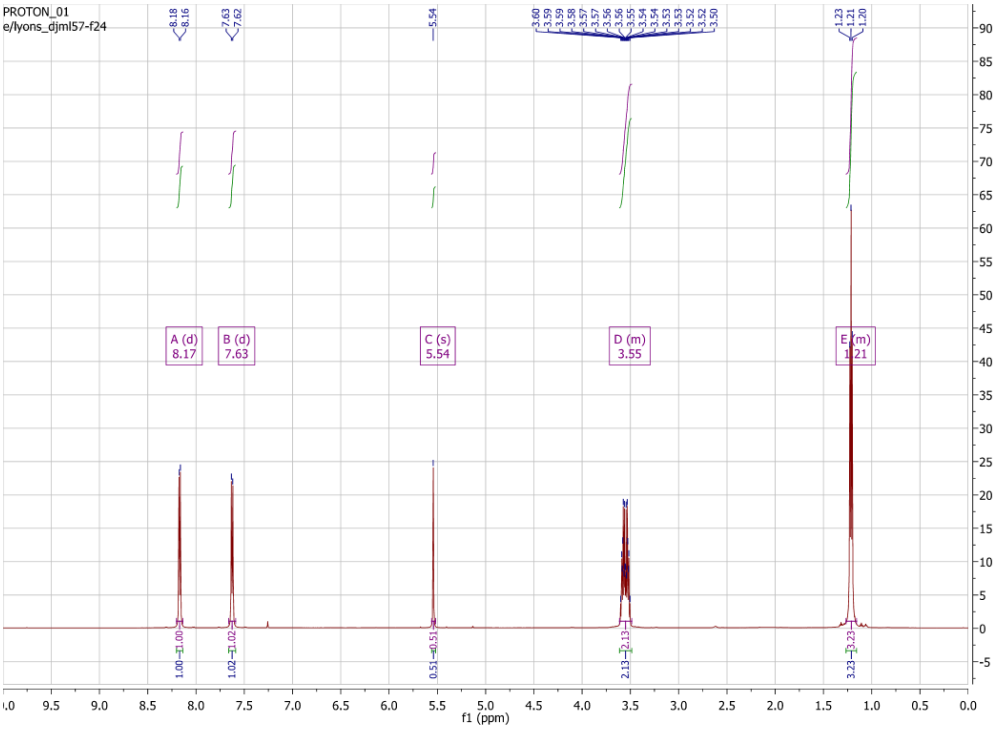
f1 (ppm)



4dE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C (s)
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D (m)
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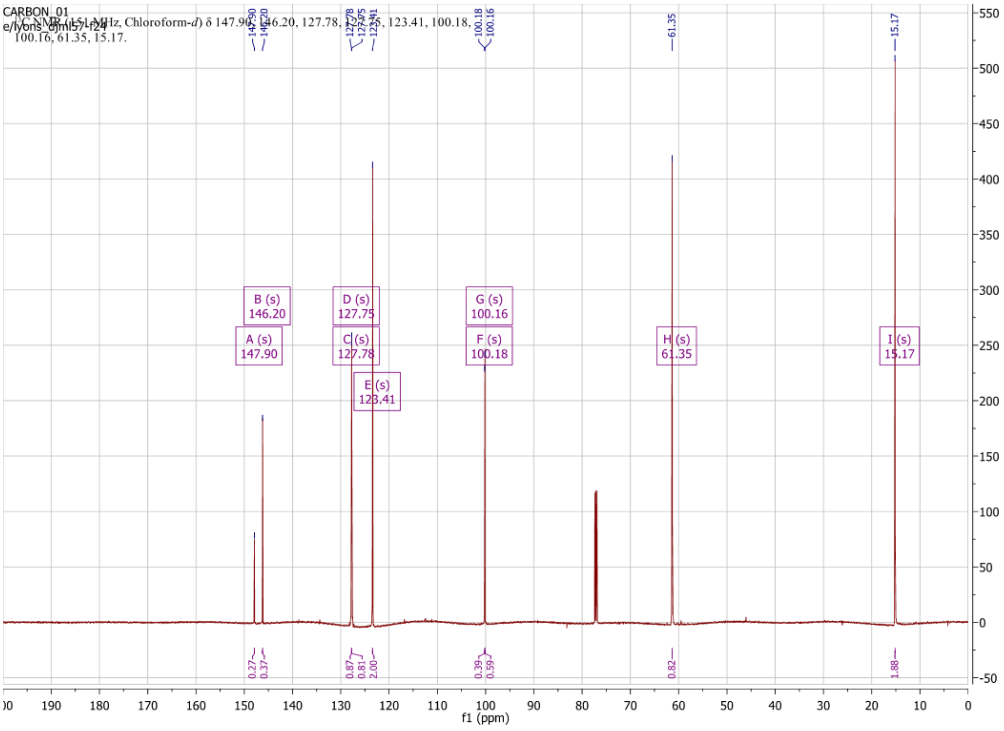
E (m)
1.21
f1 (ppm)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A (s)
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D (s)
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C (s)
127.78
E (s)
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G (s)
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F (s)
100.18
H (s)
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I (s)
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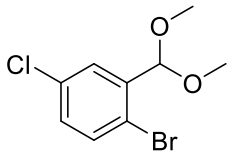
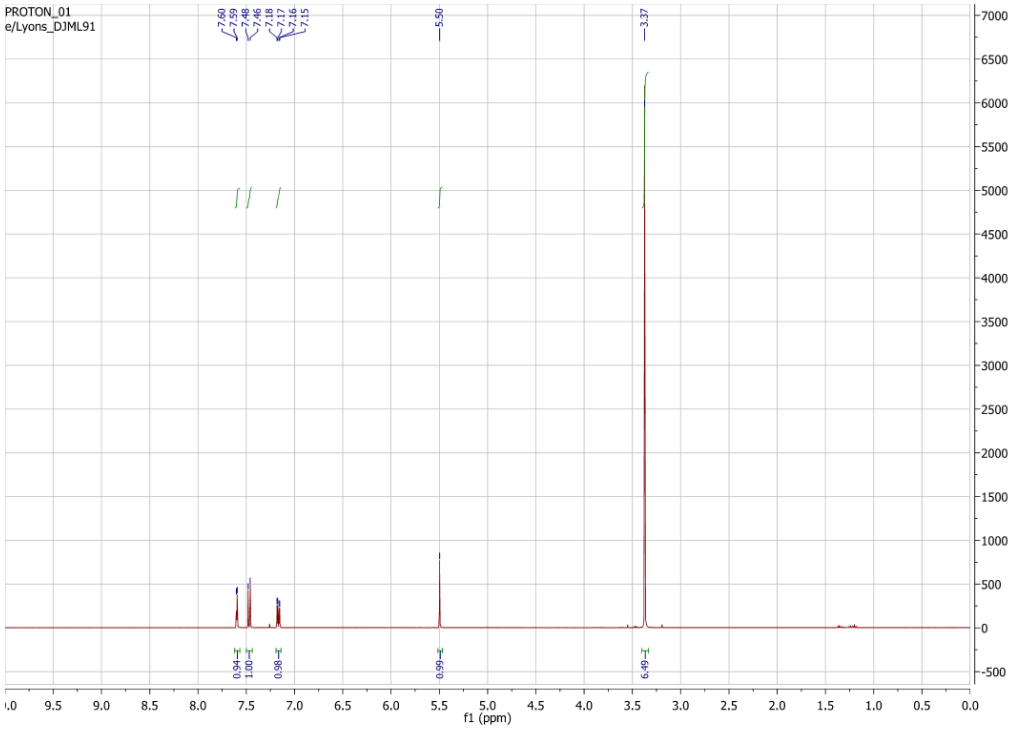
f1 (ppm)


Cl
O
O
Br

4eM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7.48
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7.15
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1.00
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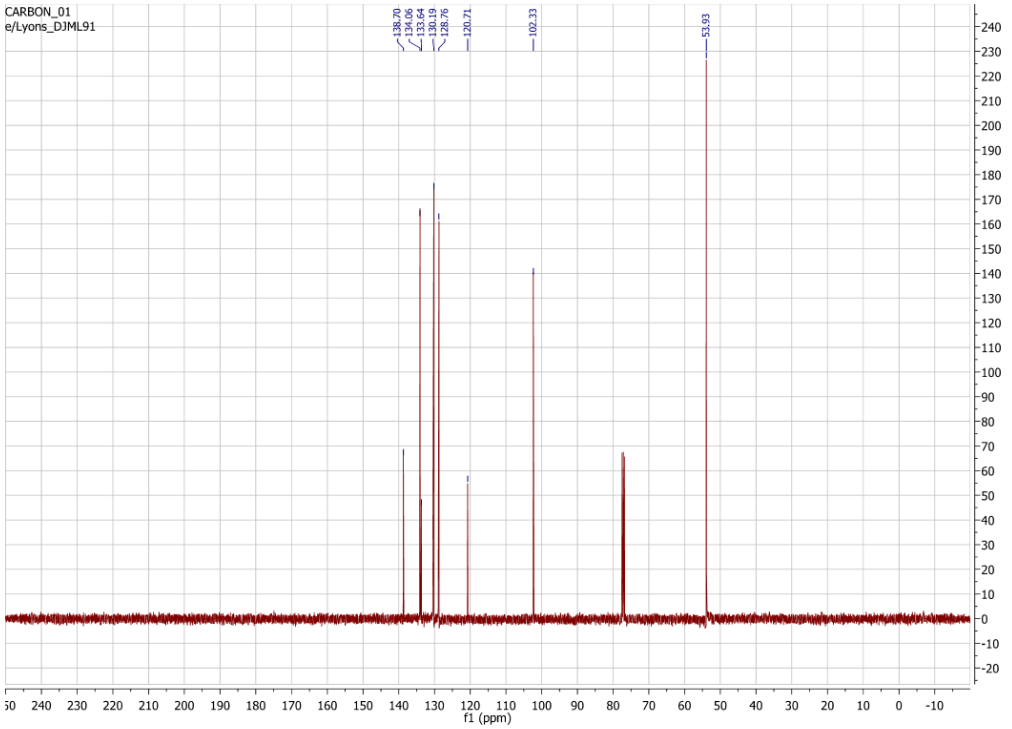
0.99
6.49
f1 (ppm)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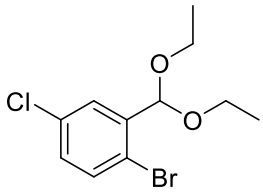
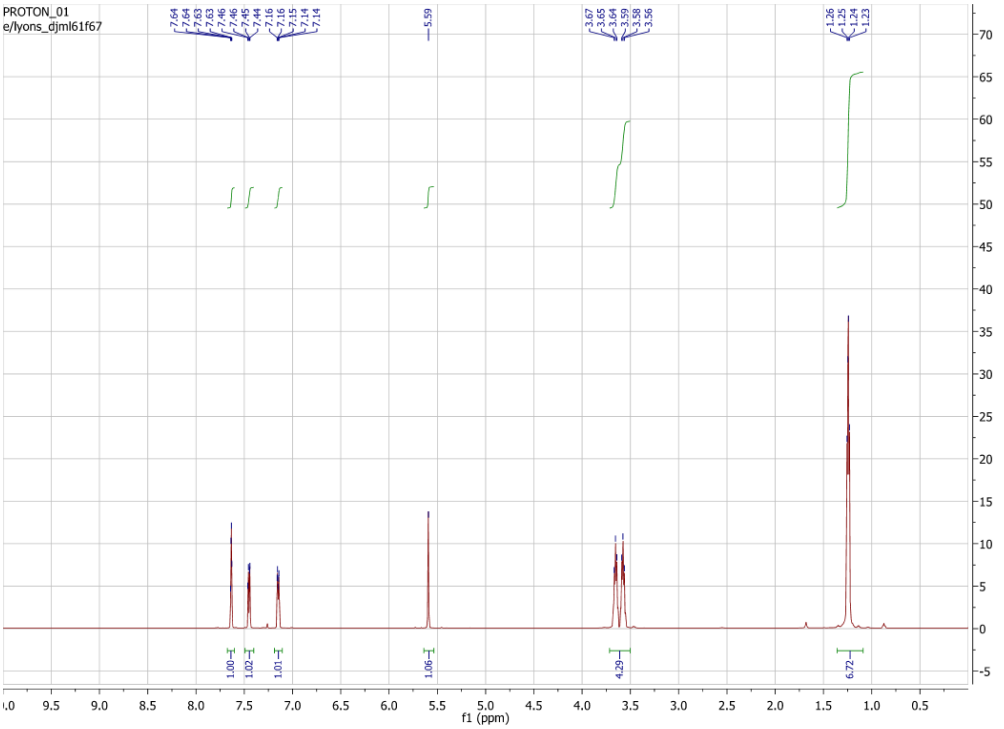
53.93
f1 (ppm)


Cl
O
O
Br

4eE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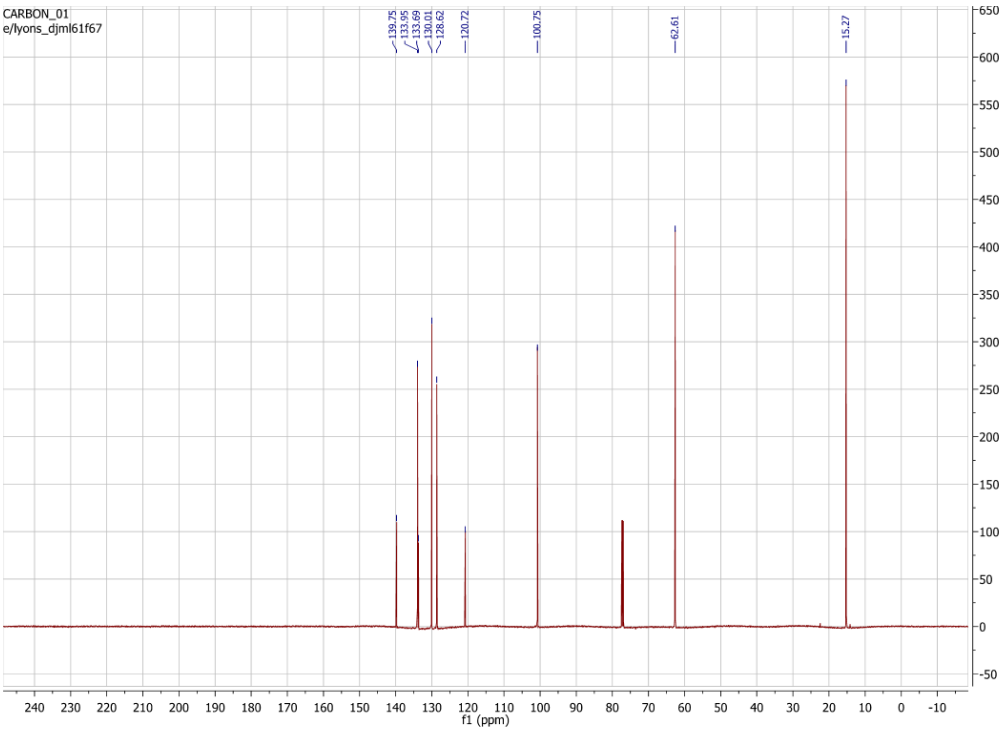
f1 (ppm)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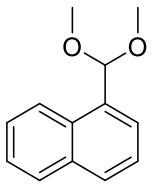
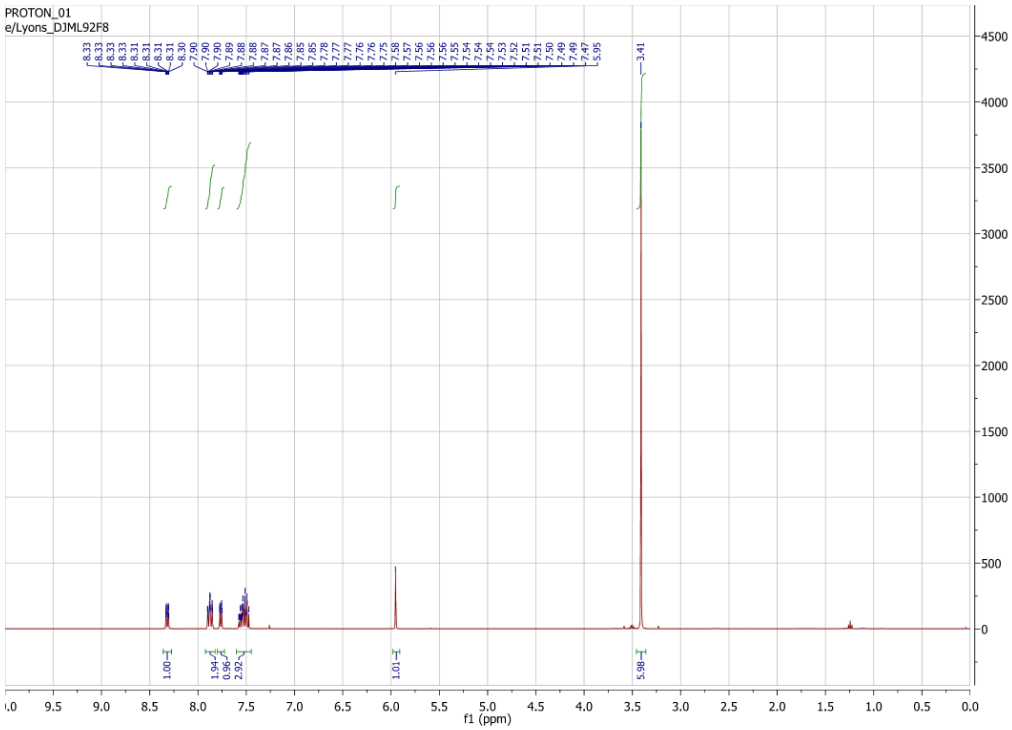


4fM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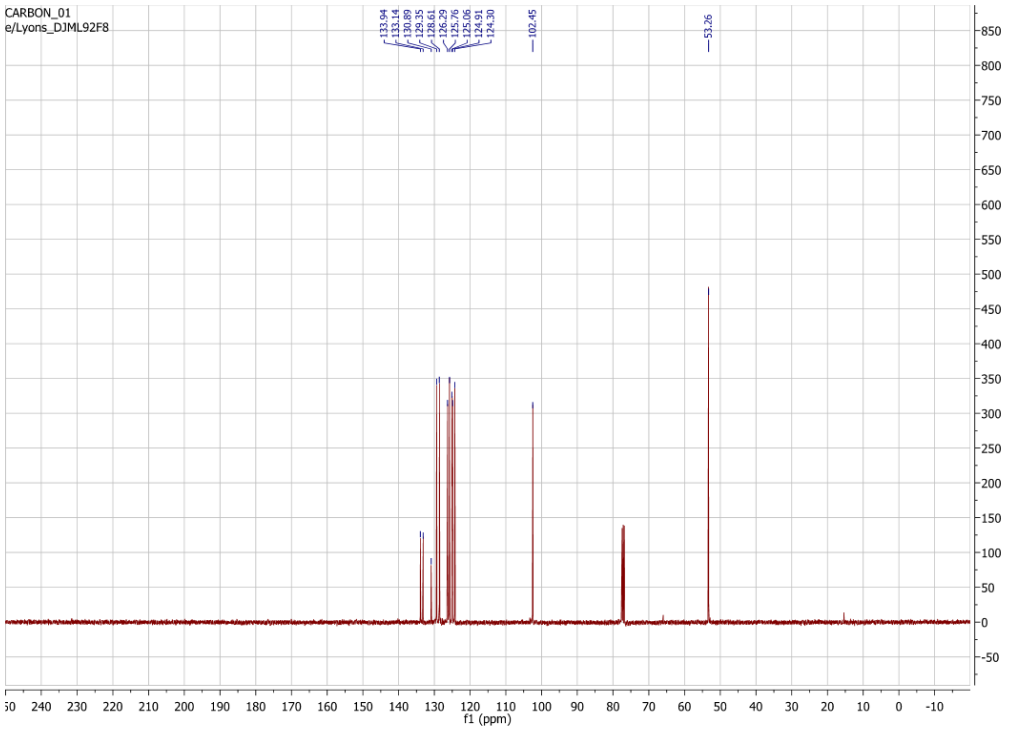
f1 (ppm)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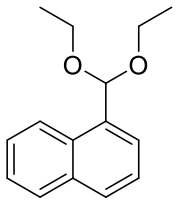
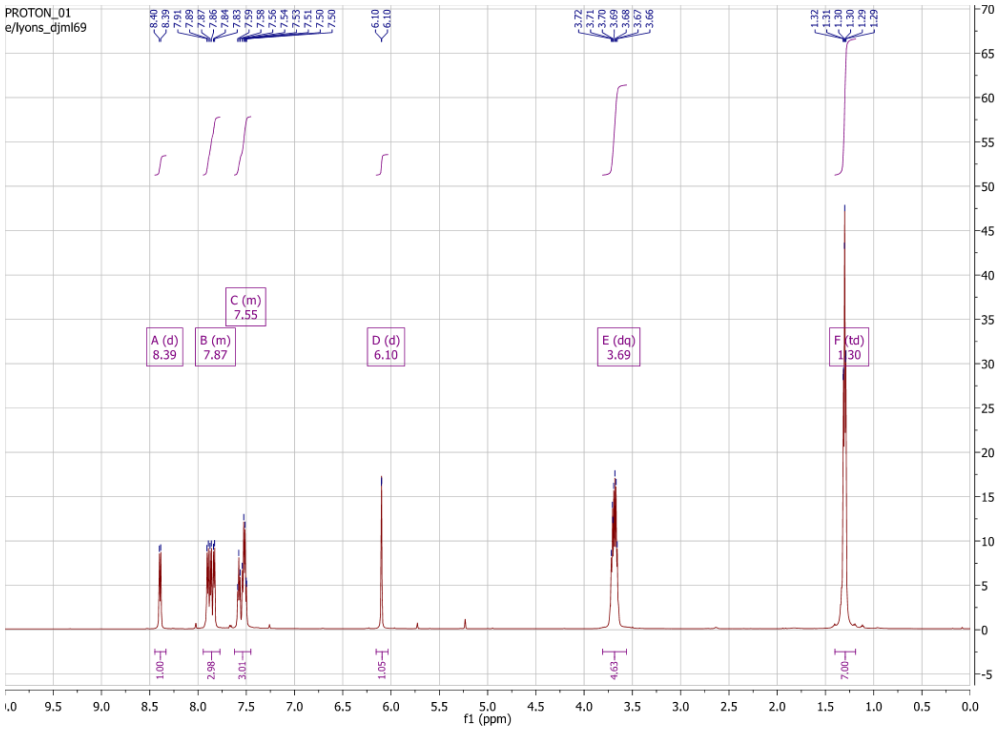
e/Lyons_DJML92F8
f1 (ppm)



4fE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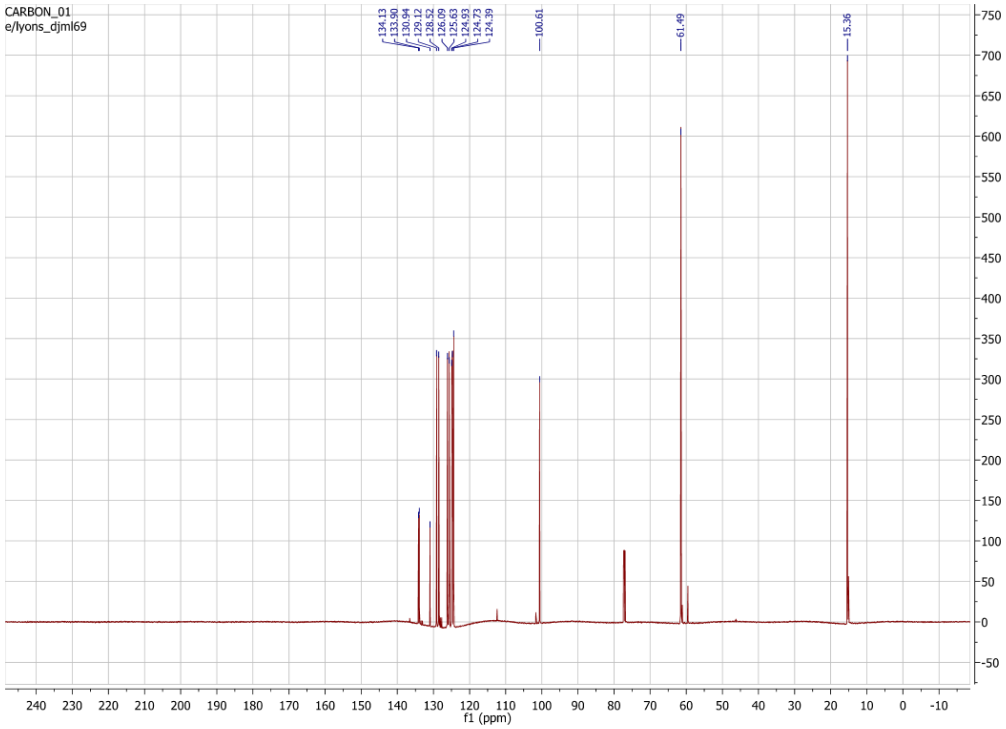
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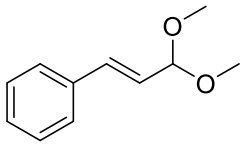
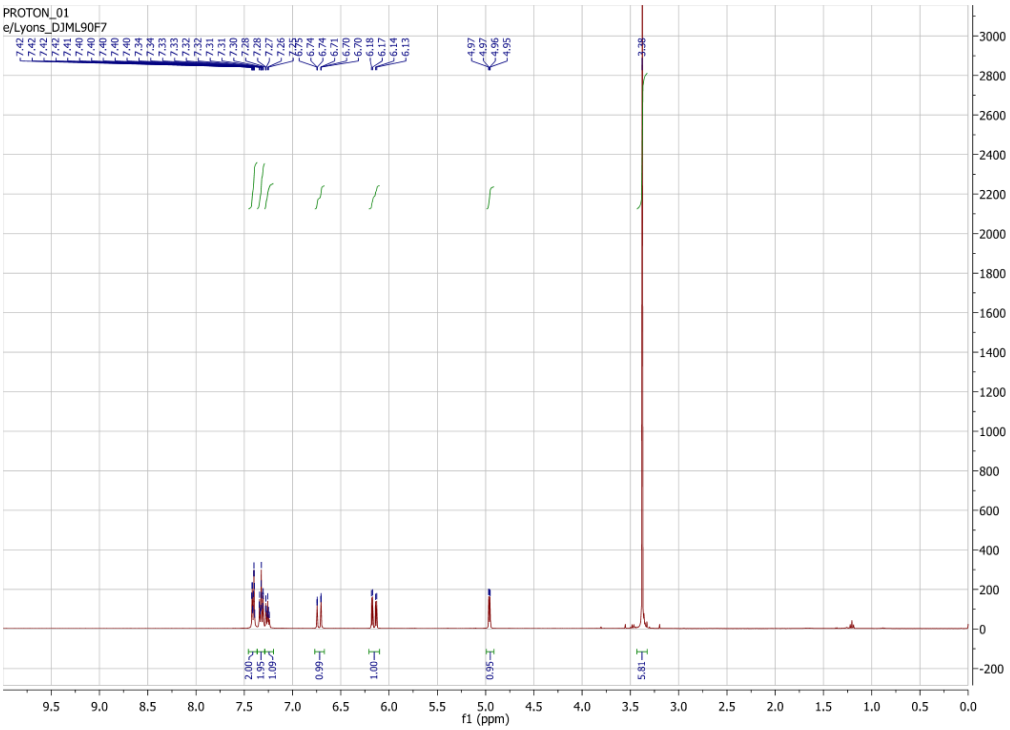
15.36
f1 (ppm)



4gM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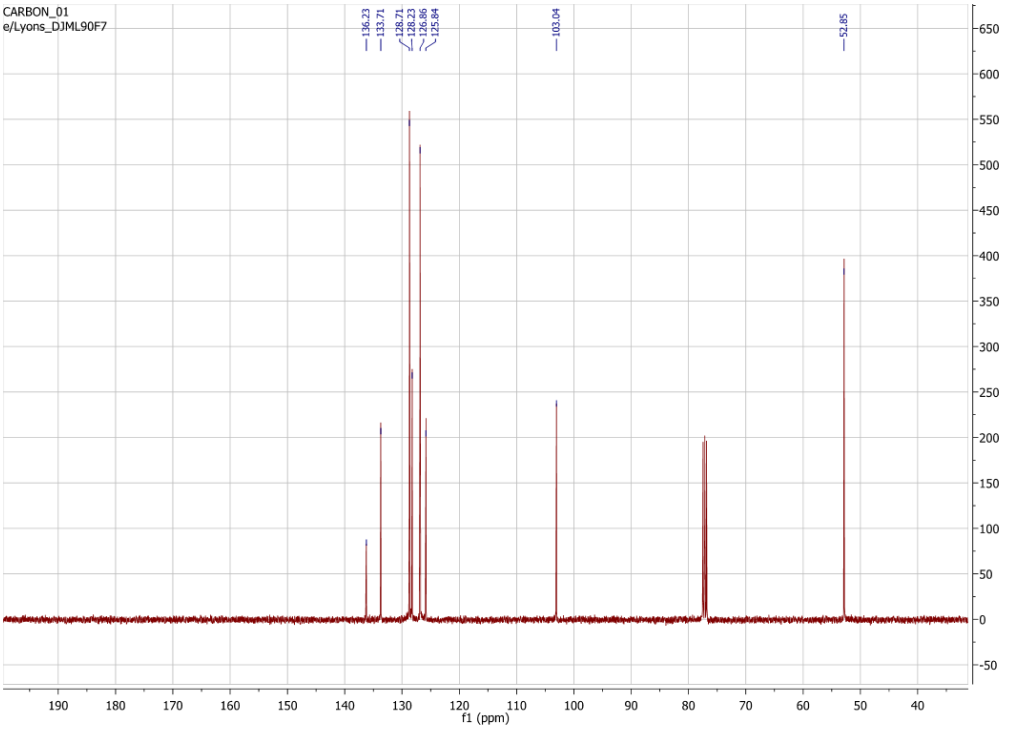
f1 (ppm)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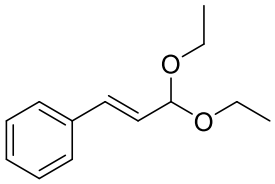
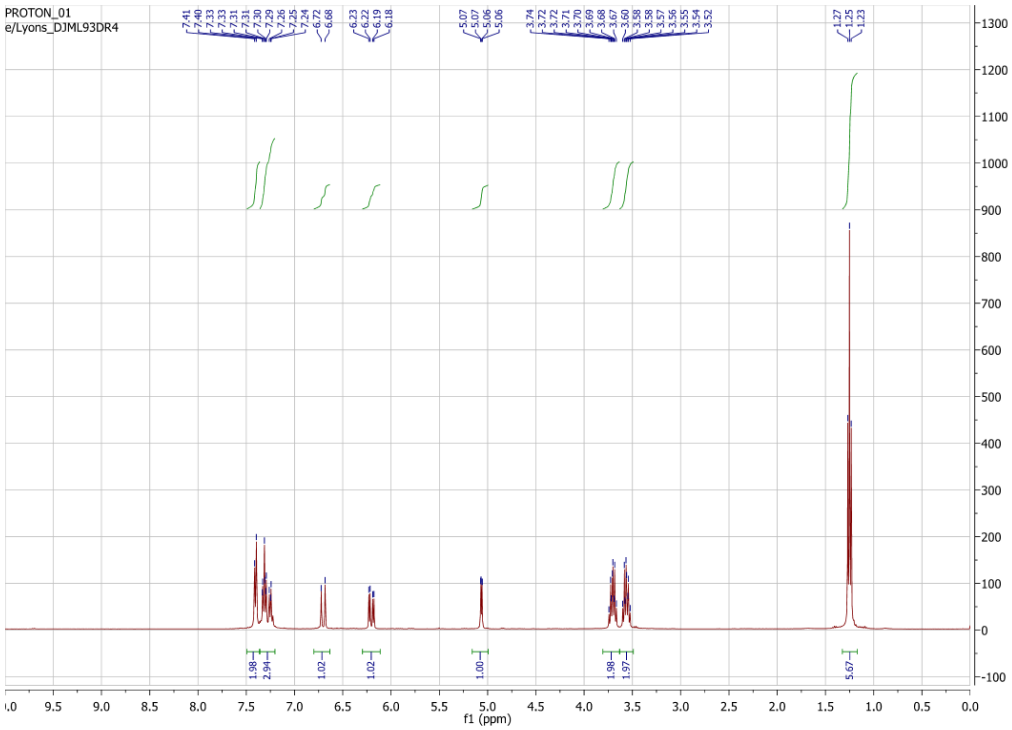


4gE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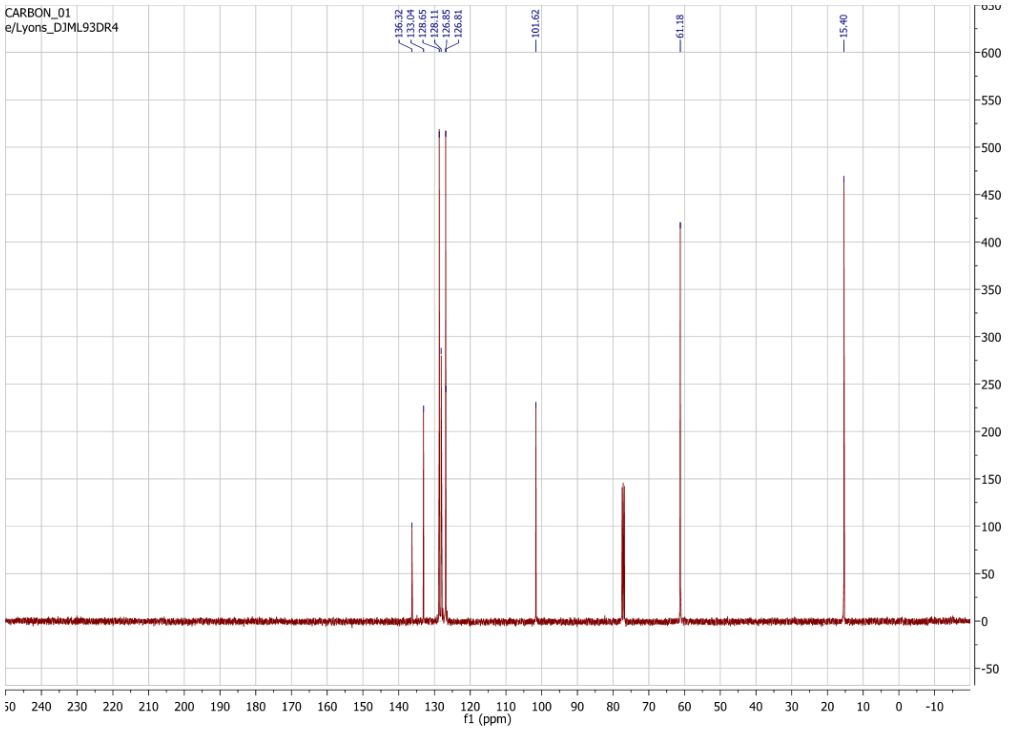
e/Lyons_DJML93DR4
f1 (ppm)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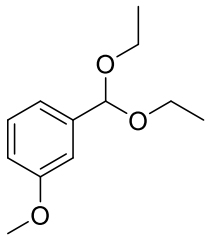

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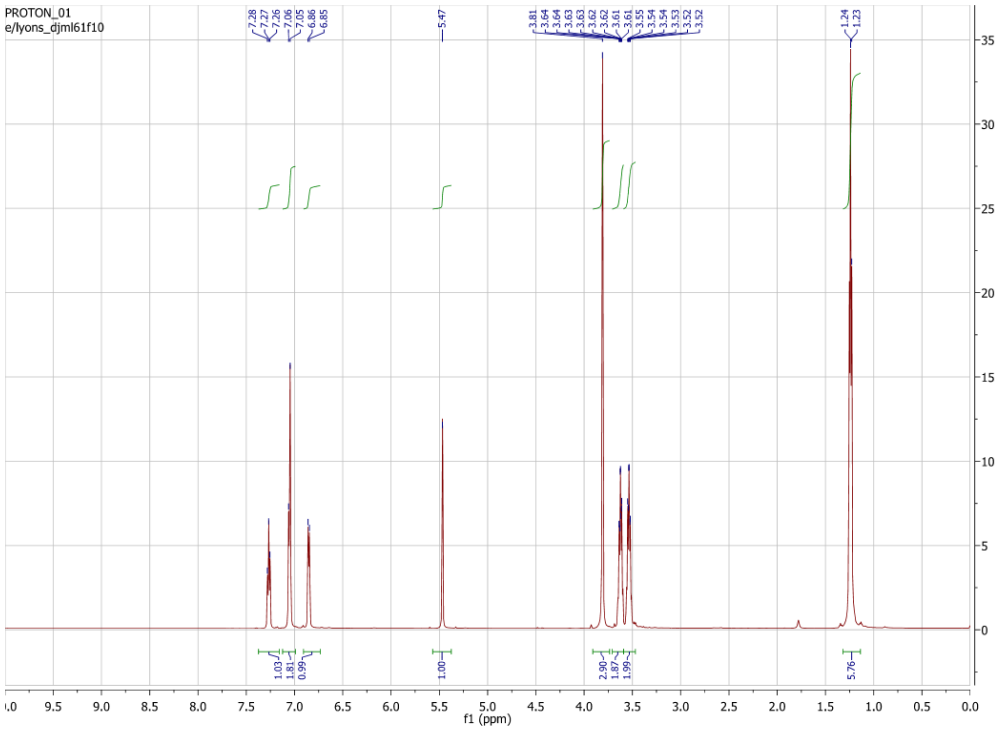
PROTON_01
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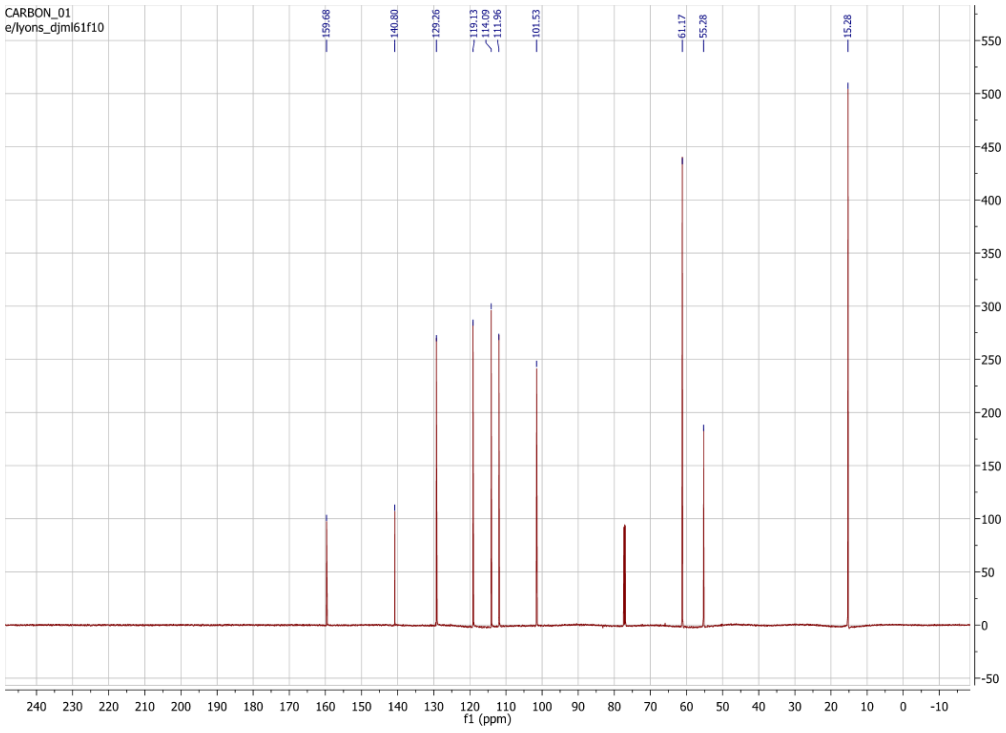
f1 (ppm)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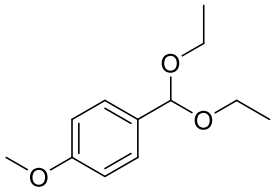

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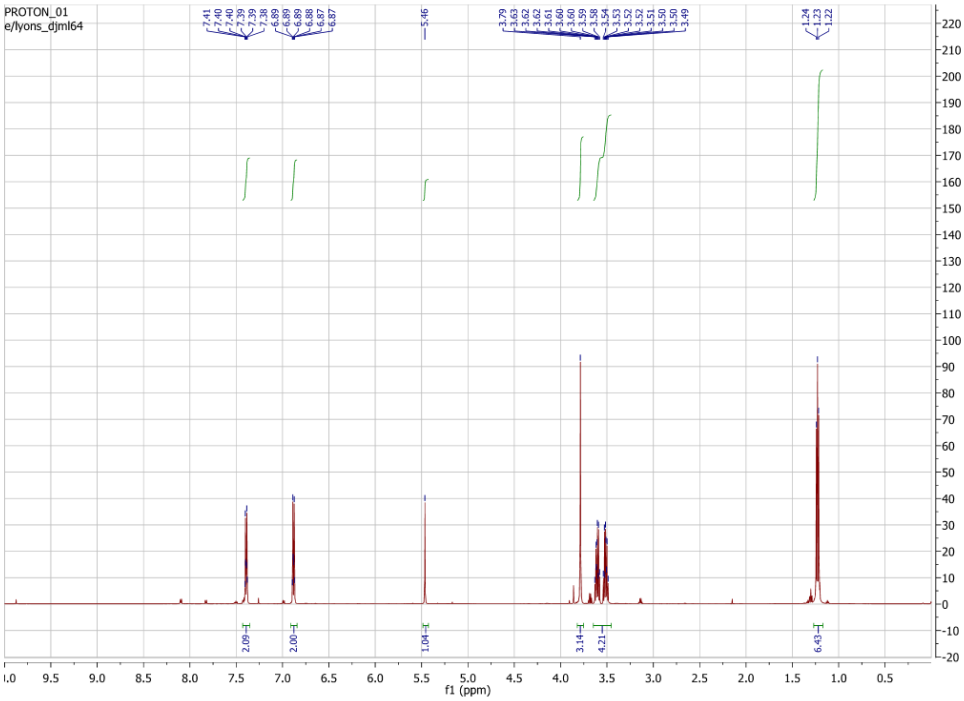
PROTON_01
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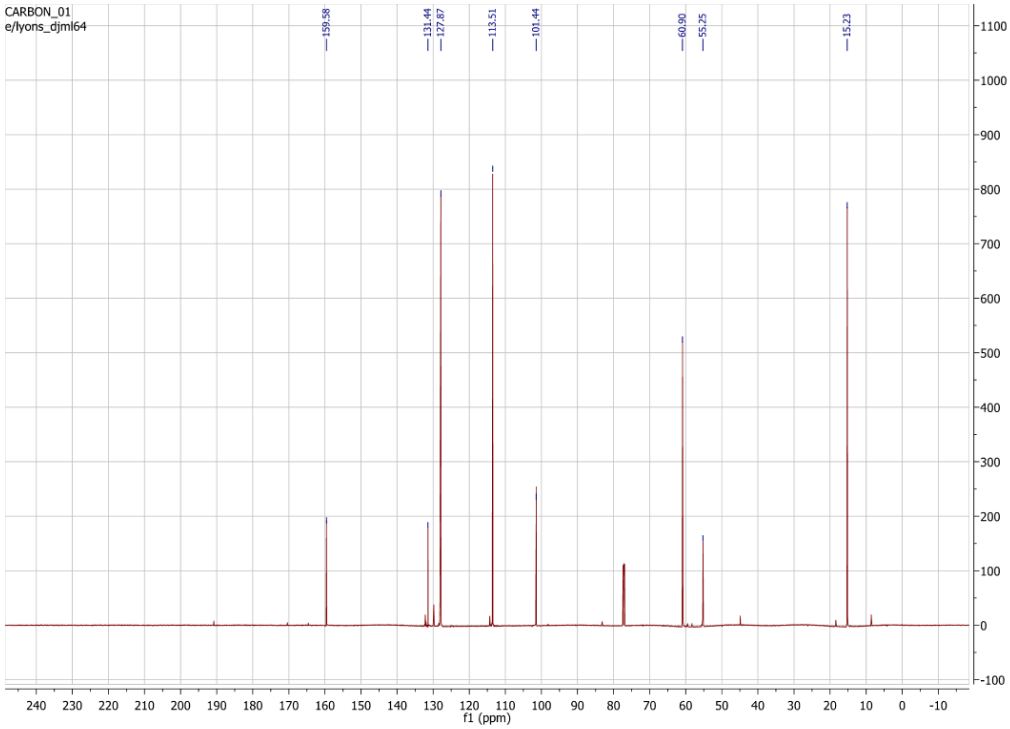
f1 (ppm)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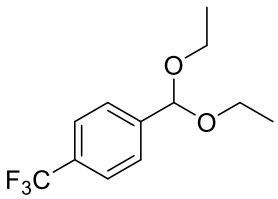
e/lyons_djml64
f1 (ppm)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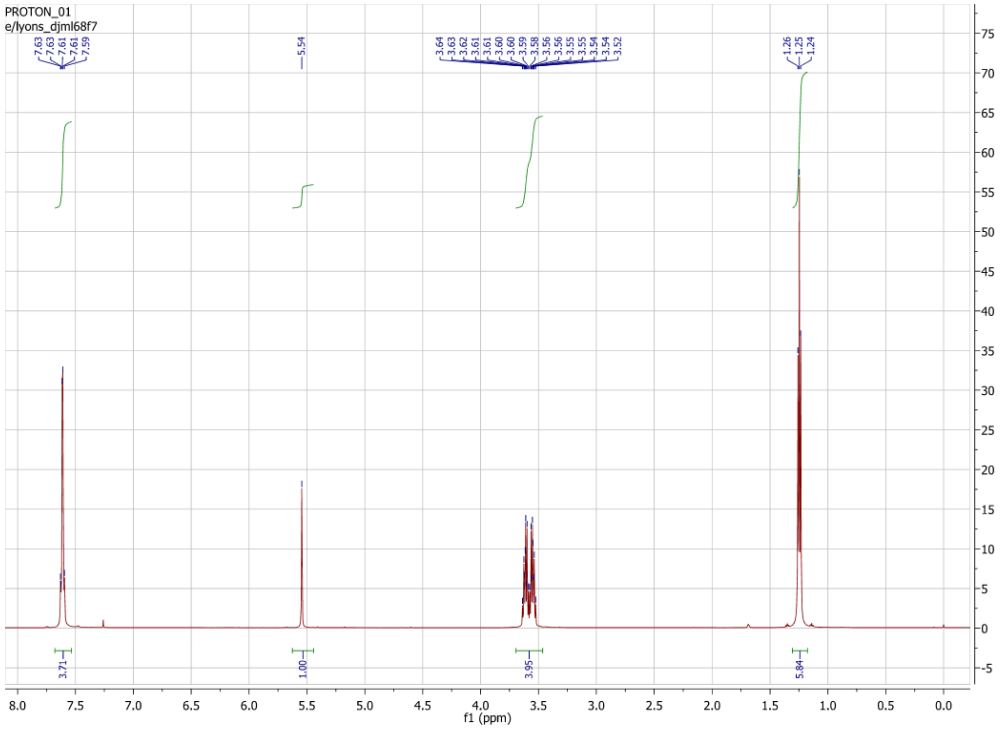
F3C

4j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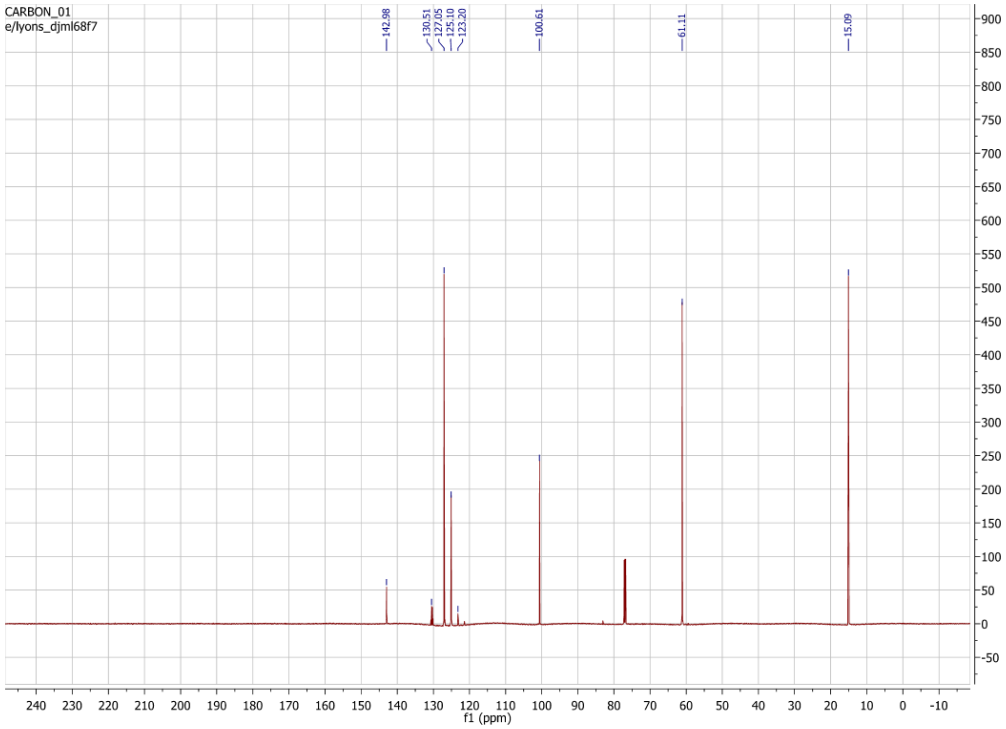
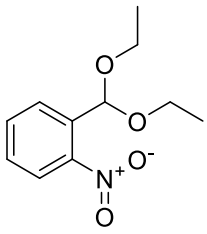
f1 (ppm)


CARBON_01
e/lyons_djml68f7
f1 (ppm)



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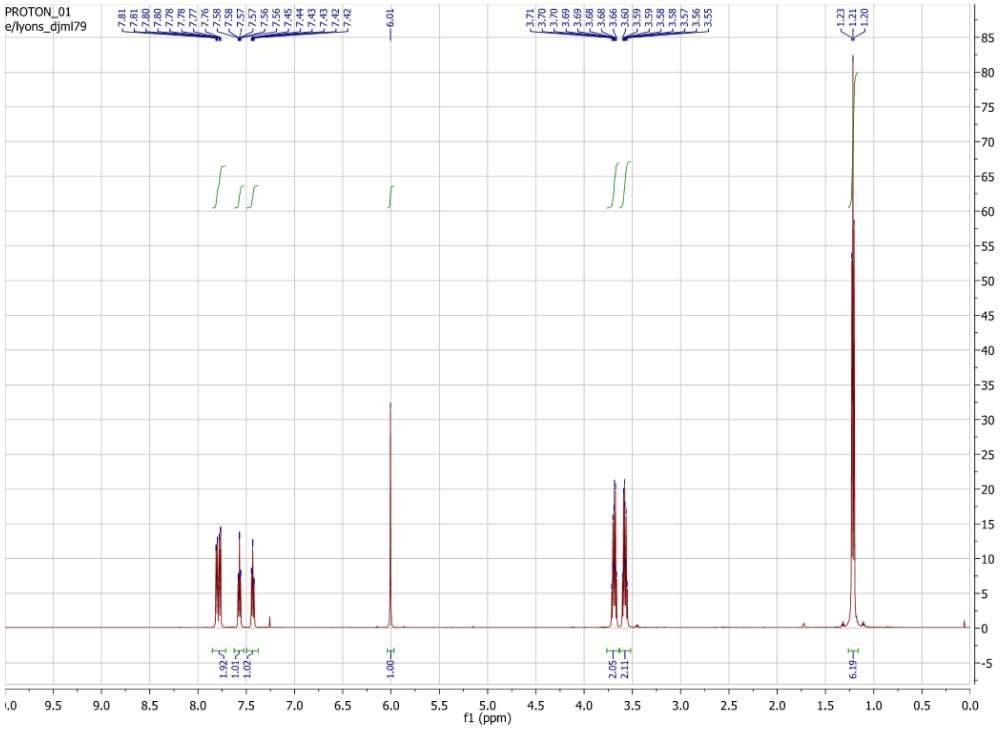
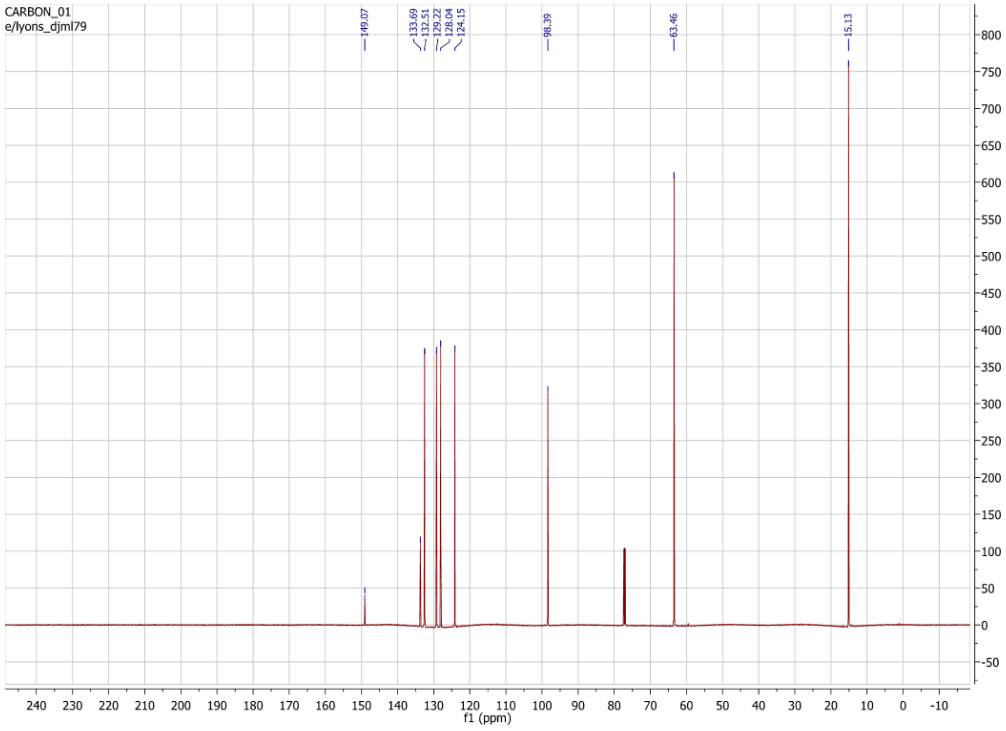
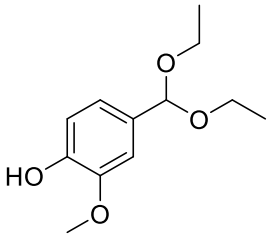

PROTON_01
e/lyons_djml79
f1 (ppm)


CARBON_01
e/lyons_djml79
f1 (ppm)


O
O
HO
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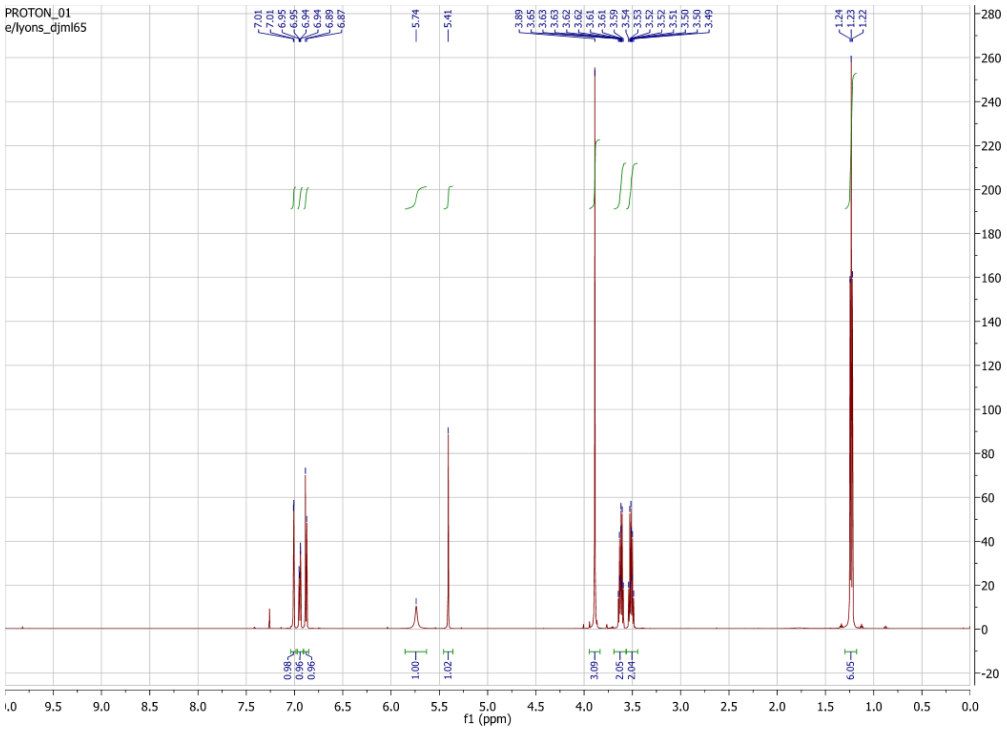
4l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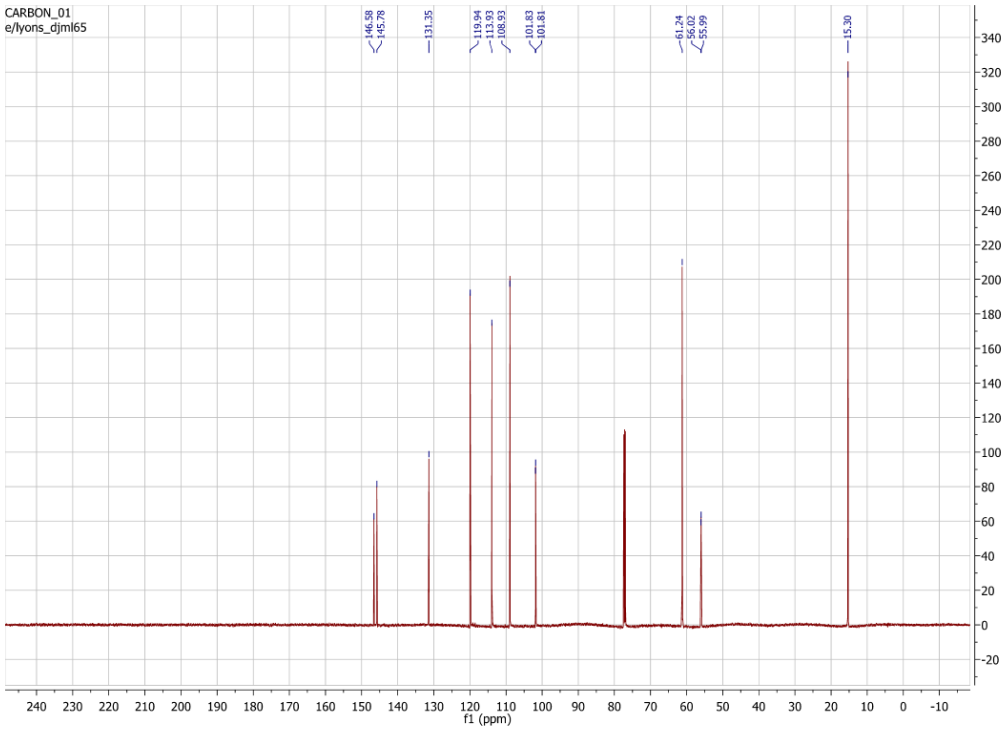
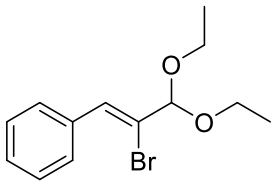
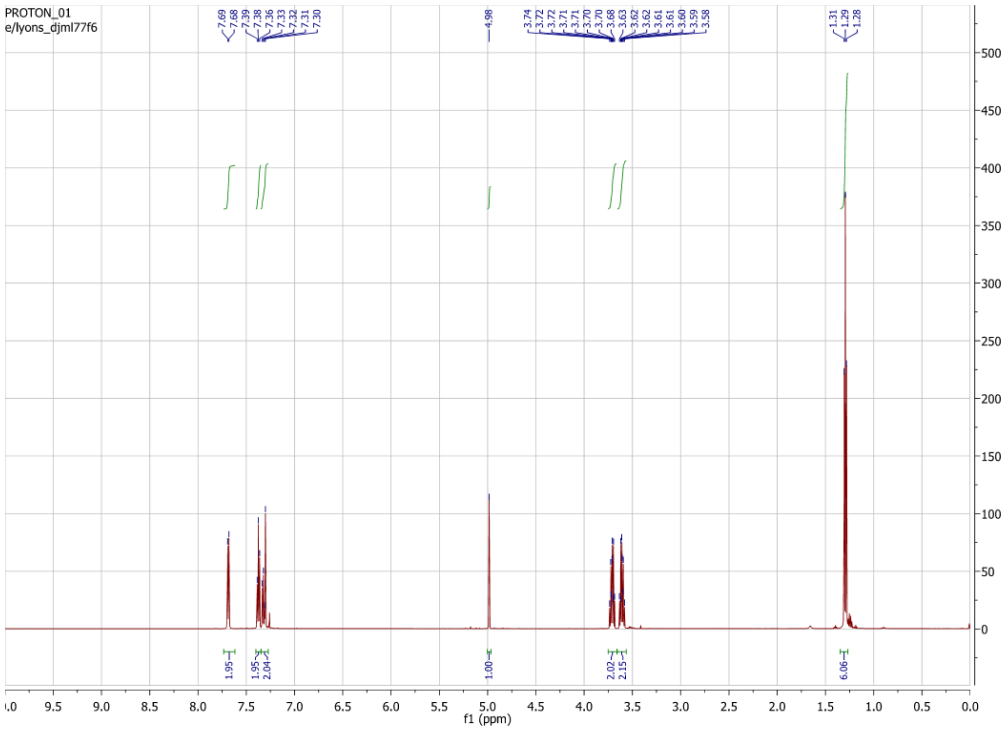
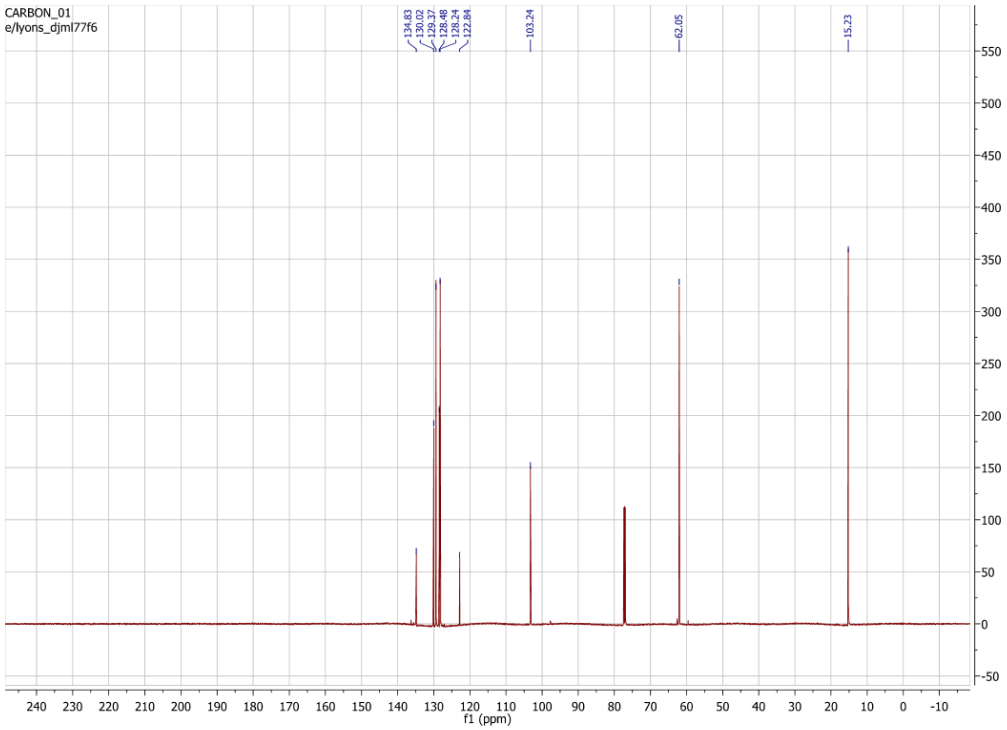
e/lyons_djml65
f1 (ppm)

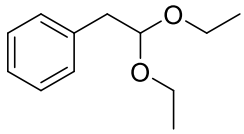

CARBON_01
e/lyons_djml65
f1 (ppm)


O
O
Br

4m


PROTON_01
e/lyons_djml77f6
f1 (ppm)


CARBON_01
e/lyons_djml77f6
f1 (ppm)



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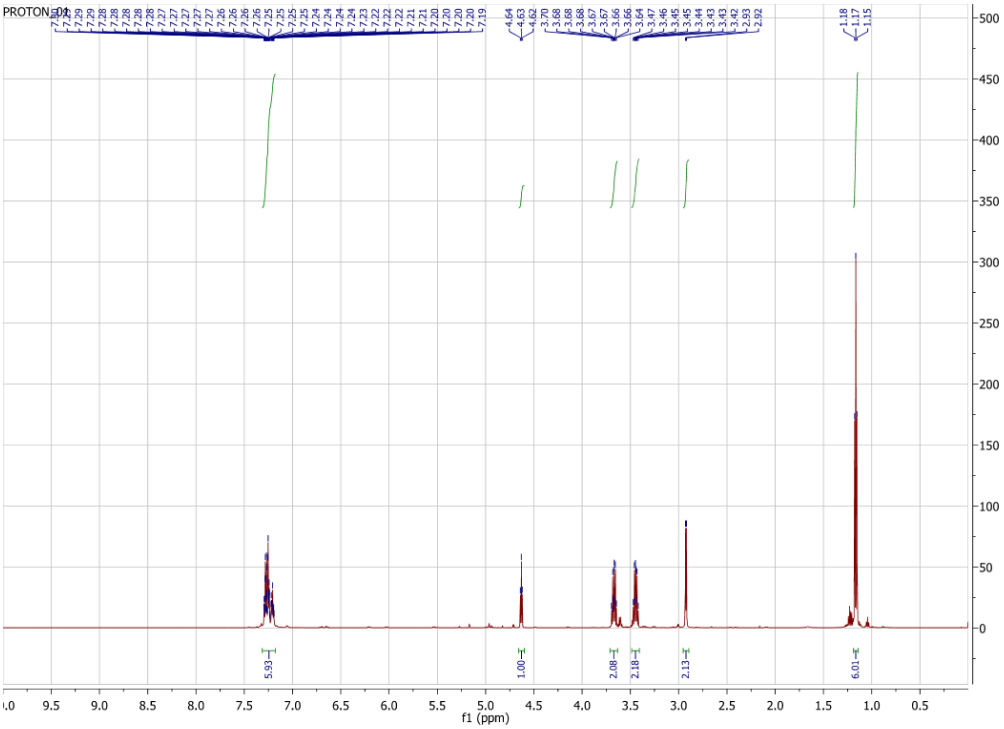
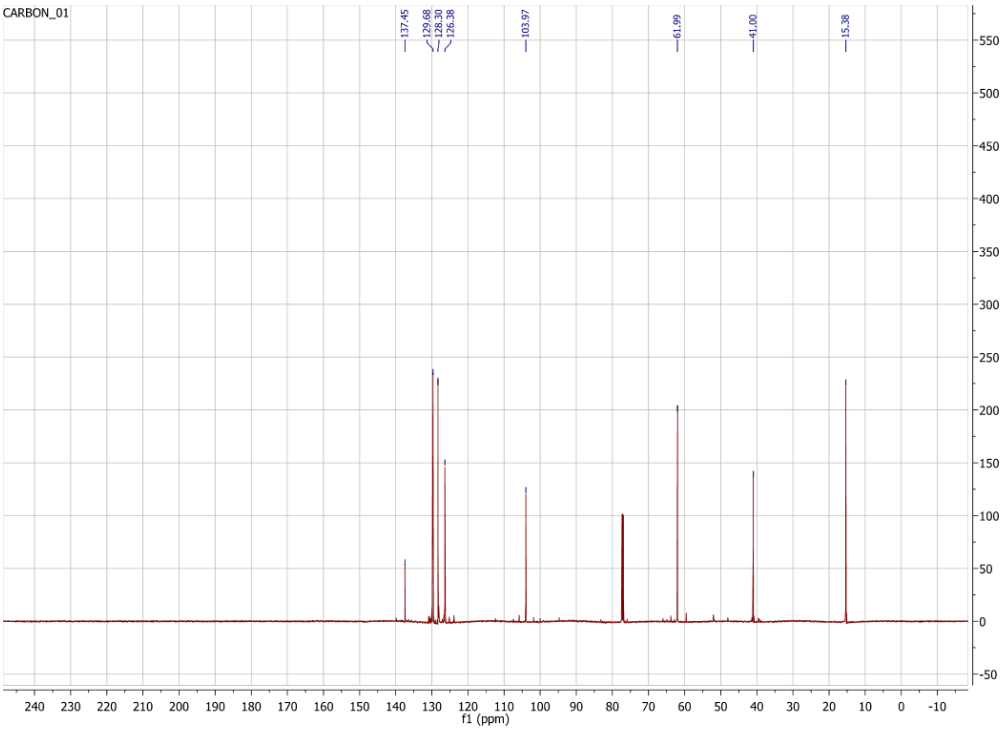
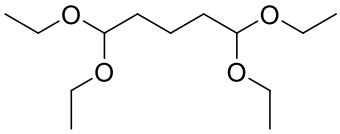
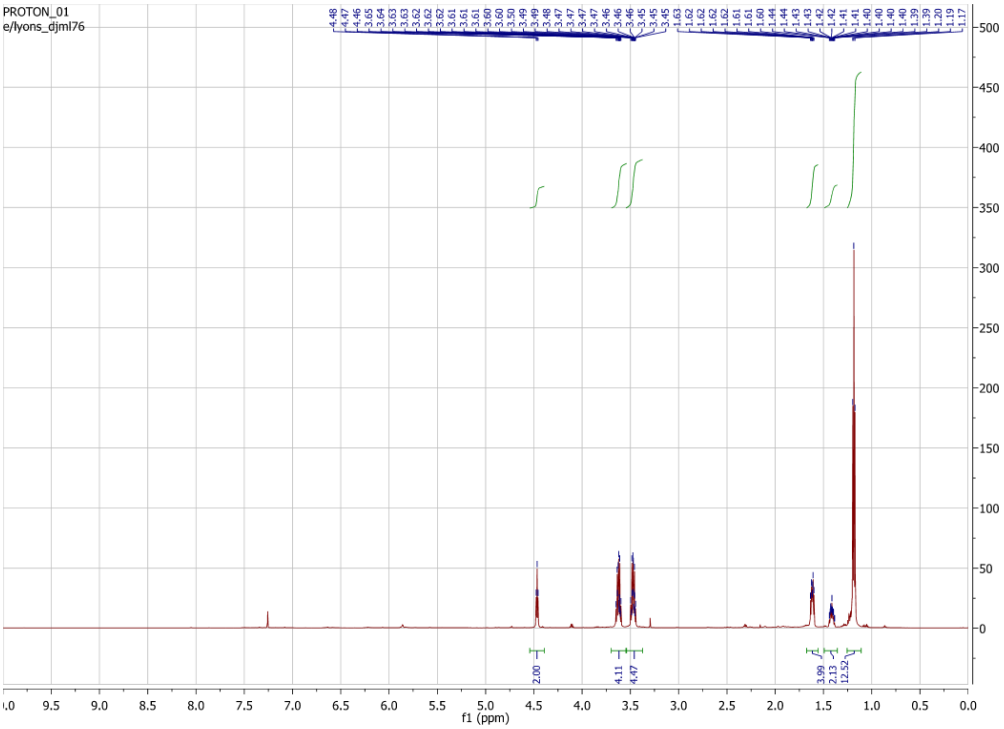
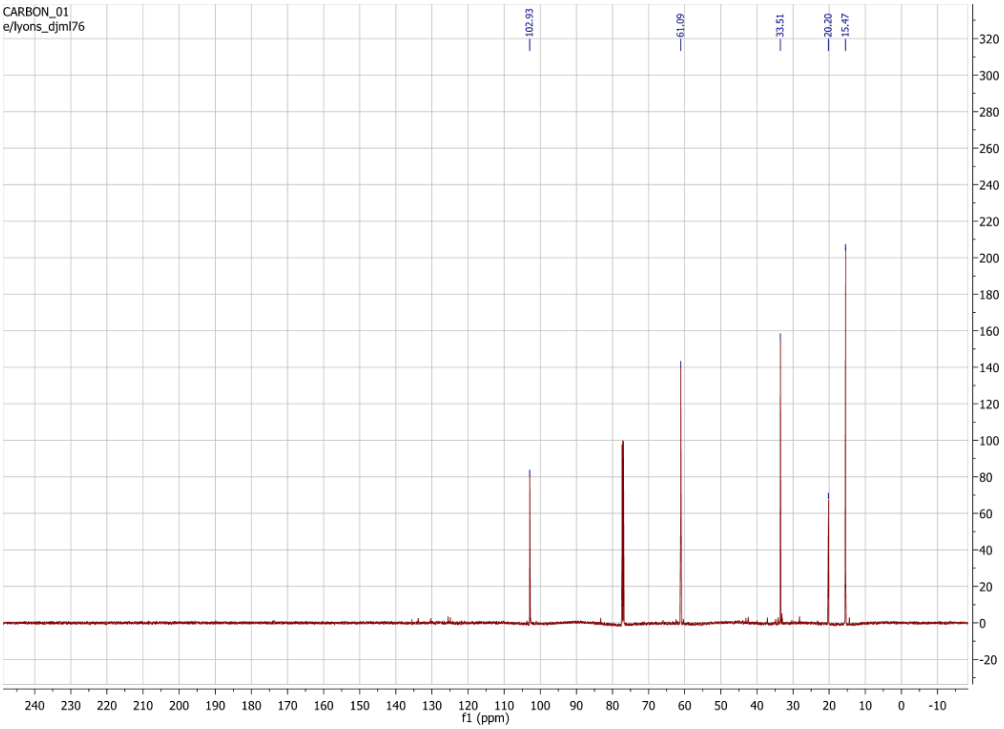

PROTON_01
f1 (ppm)

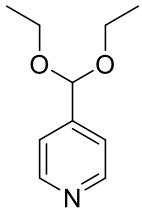

CARBON_01
137.45
120.68
128.30
126.38
103.97
61.99
41.00
15.38
f1 (ppm)



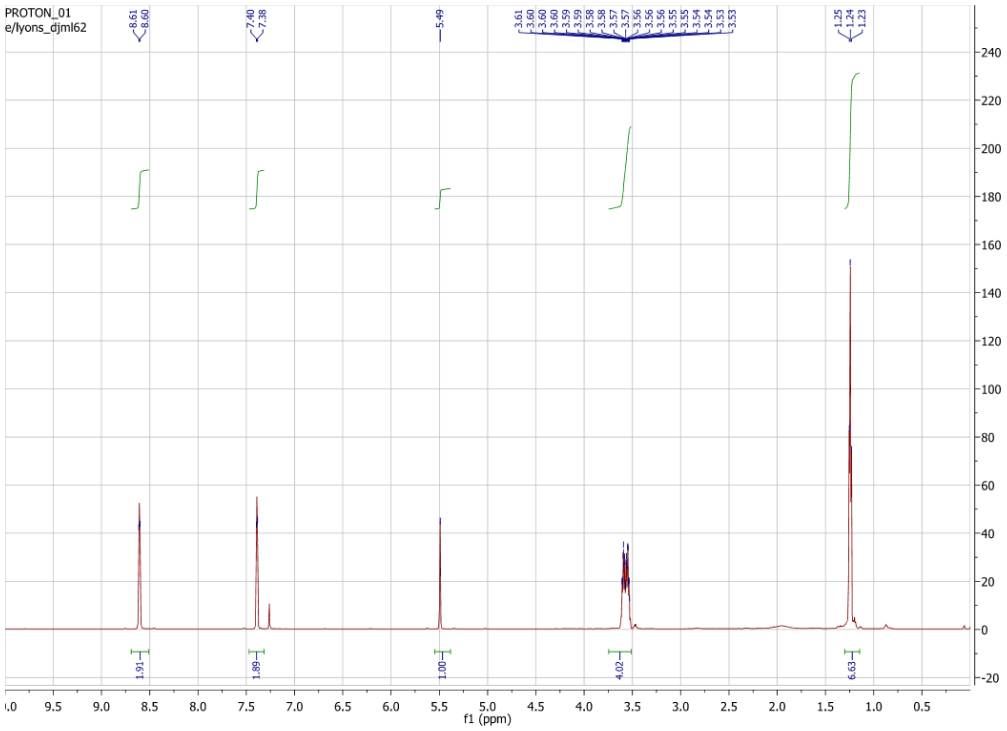
4o

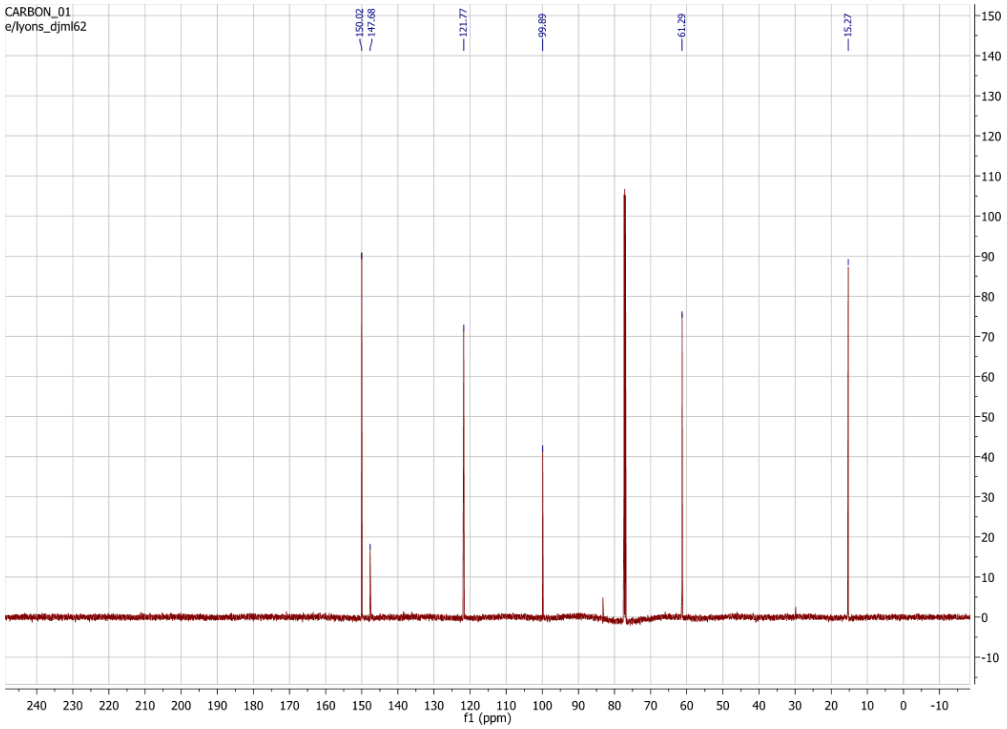

PROTON_01
e/lyons_djml76
f1 (ppm)

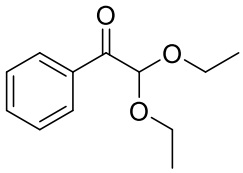

CARBON_01
e/lyons_djml76
102.93
61.09
33.51
20.20
15.47
f1 (ppm)



4s


PROTON_01
e/lyons_djml62
f1 (ppm)


CARBON_01
e/lyons_djml62
f1 (ppm)



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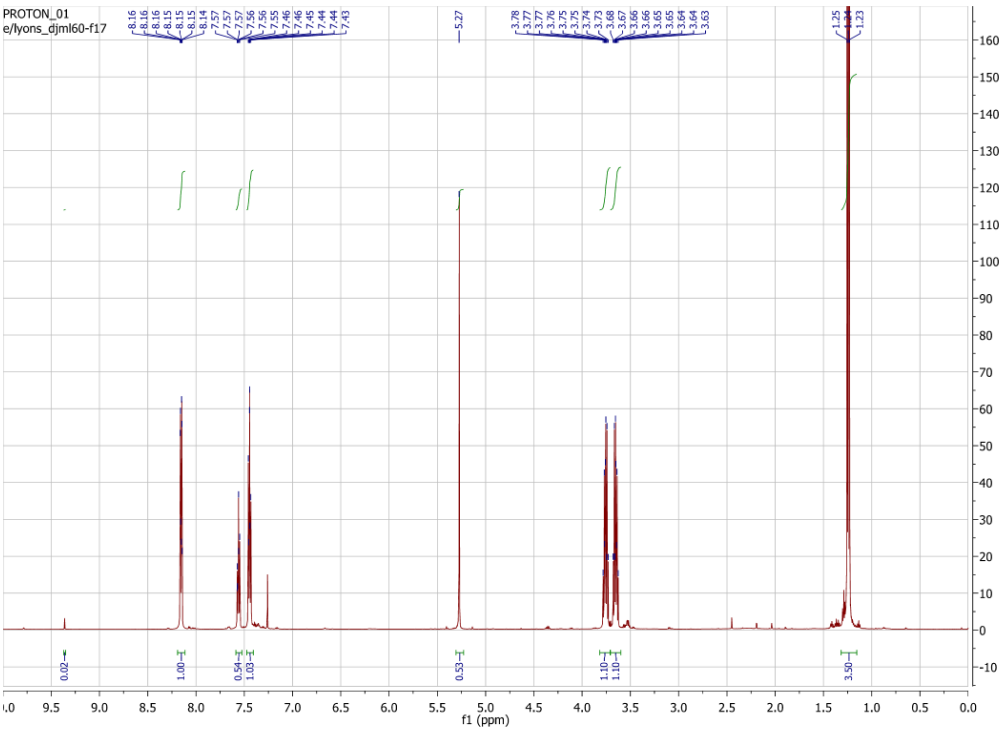
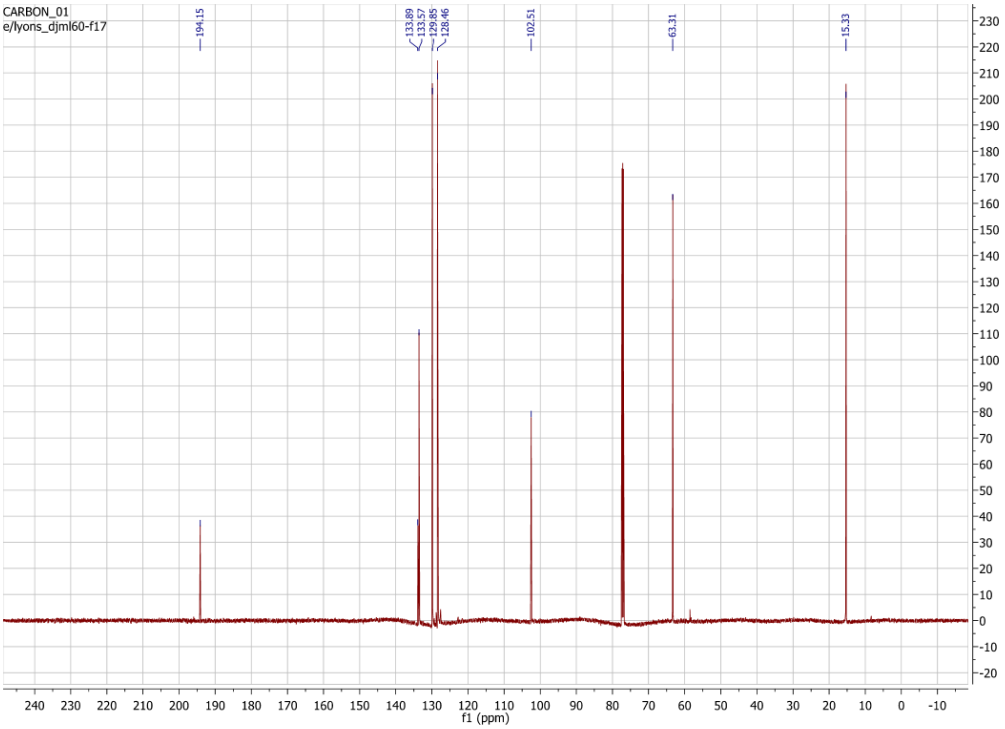
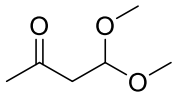
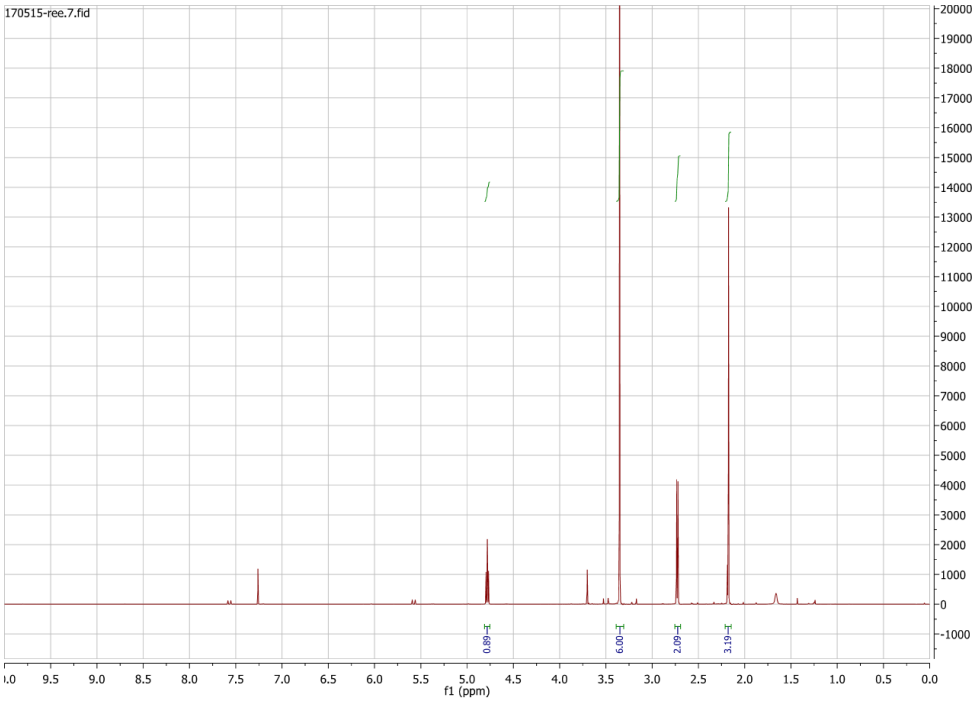
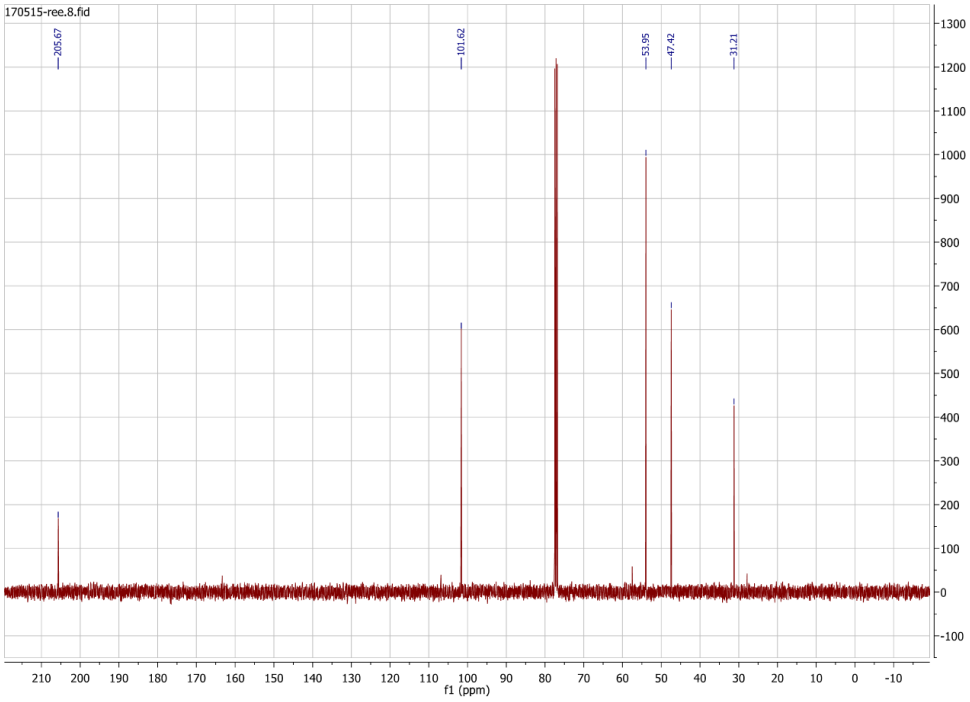

PROTON_01
e/lyons_djml60-f17
f1 (ppm)

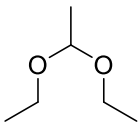

CARBON_01
e/lyons_djml60-f17
194.15
133.89
133.57
129.85
128.46
102.51
63.31
15.33
f1 (ppm)



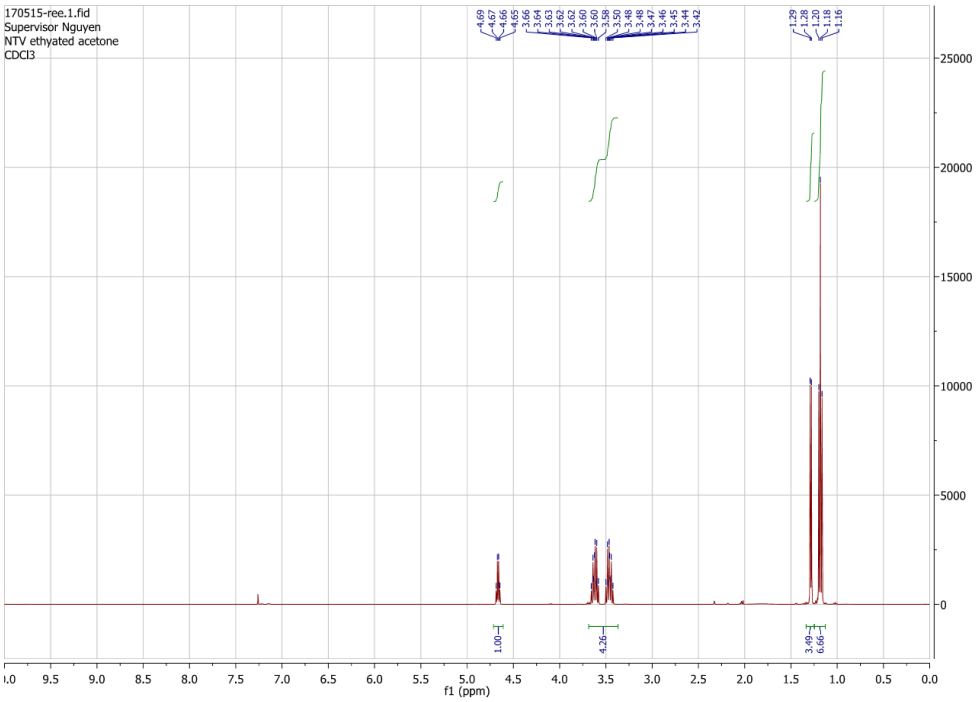
4v

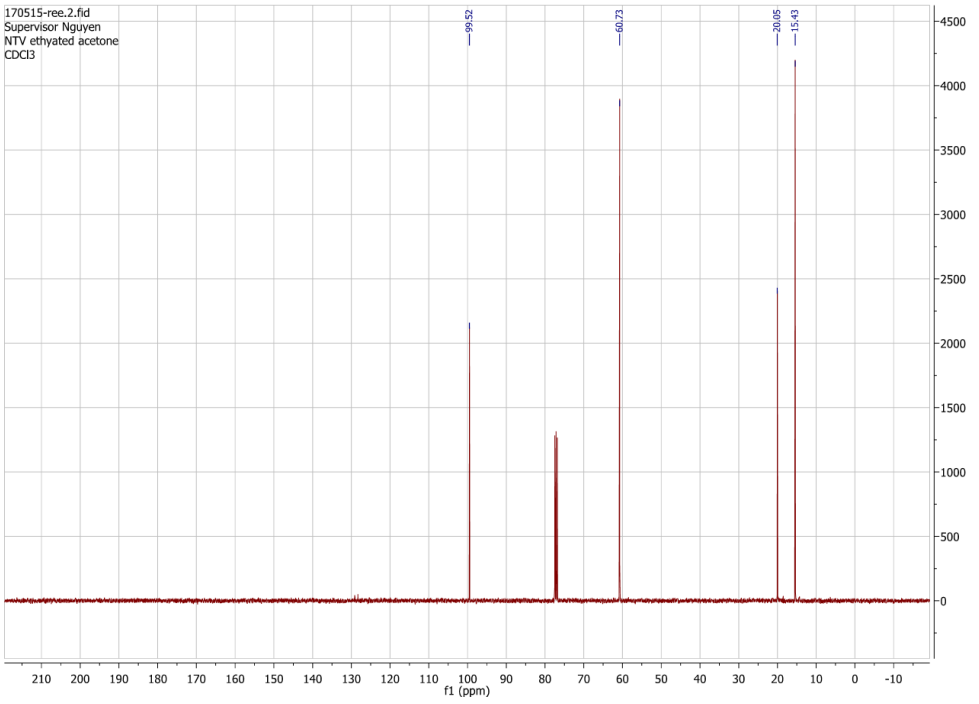

170515-ree.7.fid
f1 (ppm)
0.89
6.00
2.09
3.19

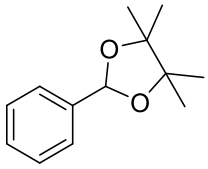

170515-ree.8.fid
205.67
101.62
53.95
47.42
31.21
f1 (ppm)



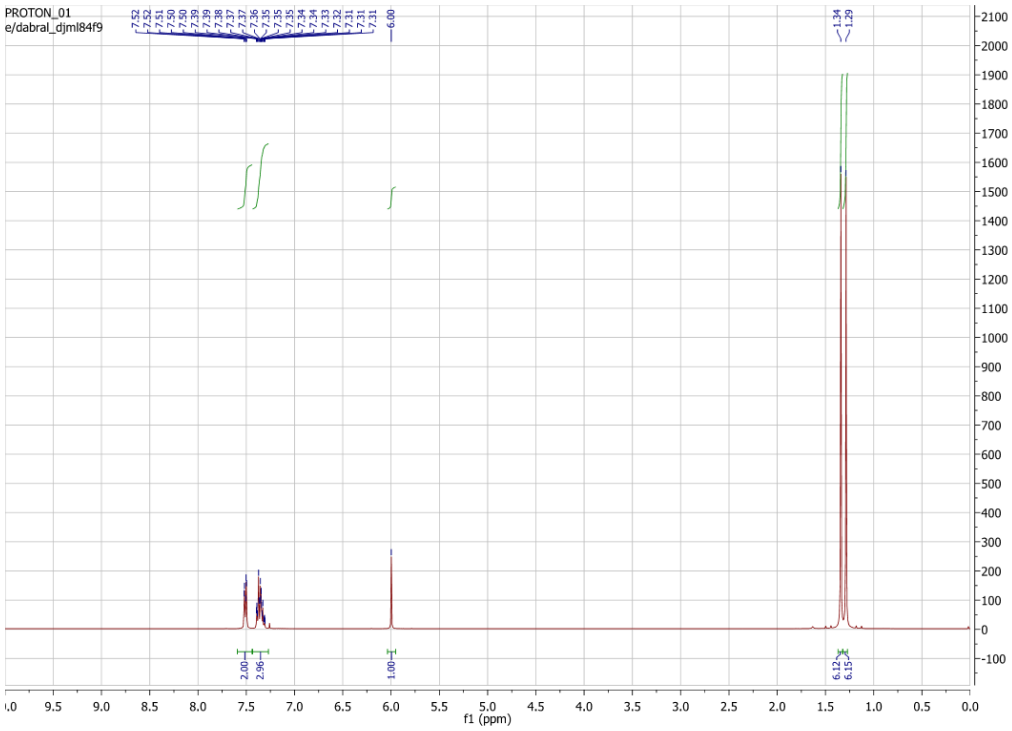
4w

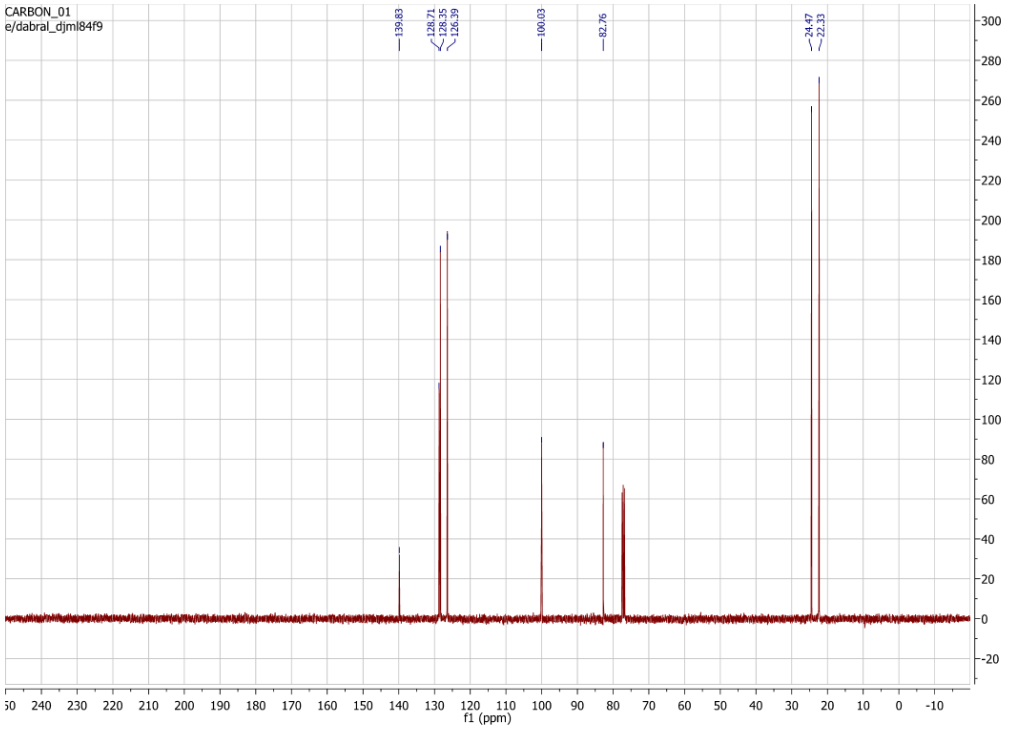

170515-ree.1.fid
Supervisor Nguyen
NTV ethyated acetone
CDCl3
4.69
4.67
4.66
4.65
3.66
3.64
3.63
3.62
3.62
3.60
3.60
3.58
3.50
3.48
3.48
3.47
3.46
3.45
3.44
3.42
1.29
1.28
1.20
1.18
1.16
1.00
4.26
3.49
6.66
f1 (ppm)

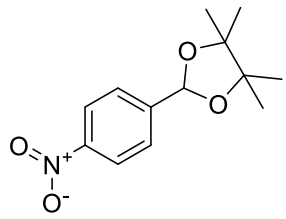

170515-ree.2.fid
Supervisor Nguyen
NTV ethyated acetone
CDCl3
99.52
60.73
20.05
15.43
f1 (ppm)



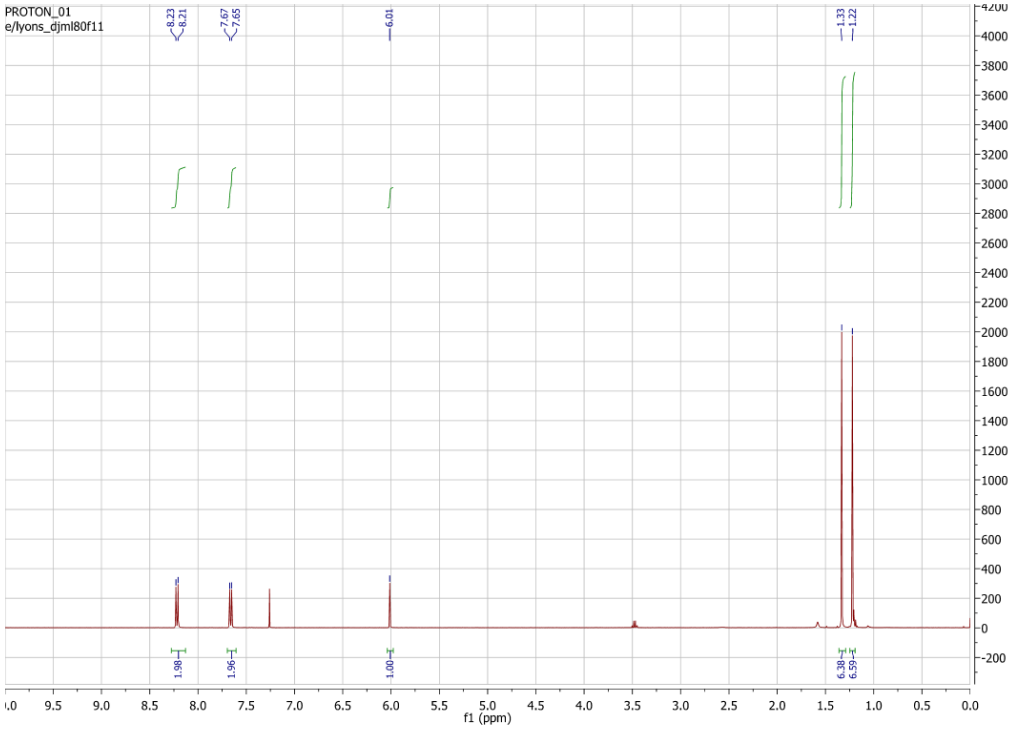
6a

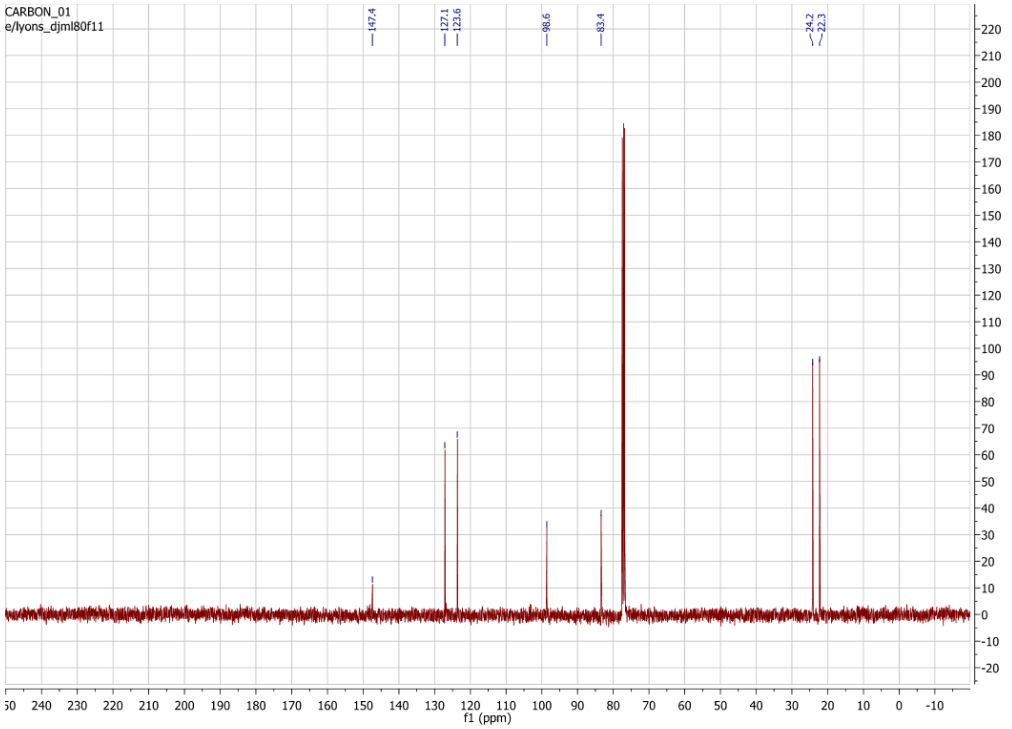

PROTON_01
e/dabral_djml84f9
7.52
7.52
7.51
7.50
7.50
7.39
7.38
7.37
7.37
7.36
7.35
7.35
7.35
7.34
7.34
7.33
7.32
7.31
7.31
7.31
6.00
1.34
1.29
2.00
2.96
1.00
6.12
6.15
f1 (ppm)

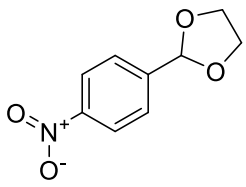

CARBON_01
e/dabral_djml84f9
139.83
128.71
128.35
126.39
100.03
82.76
24.47
22.33
f1 (ppm)



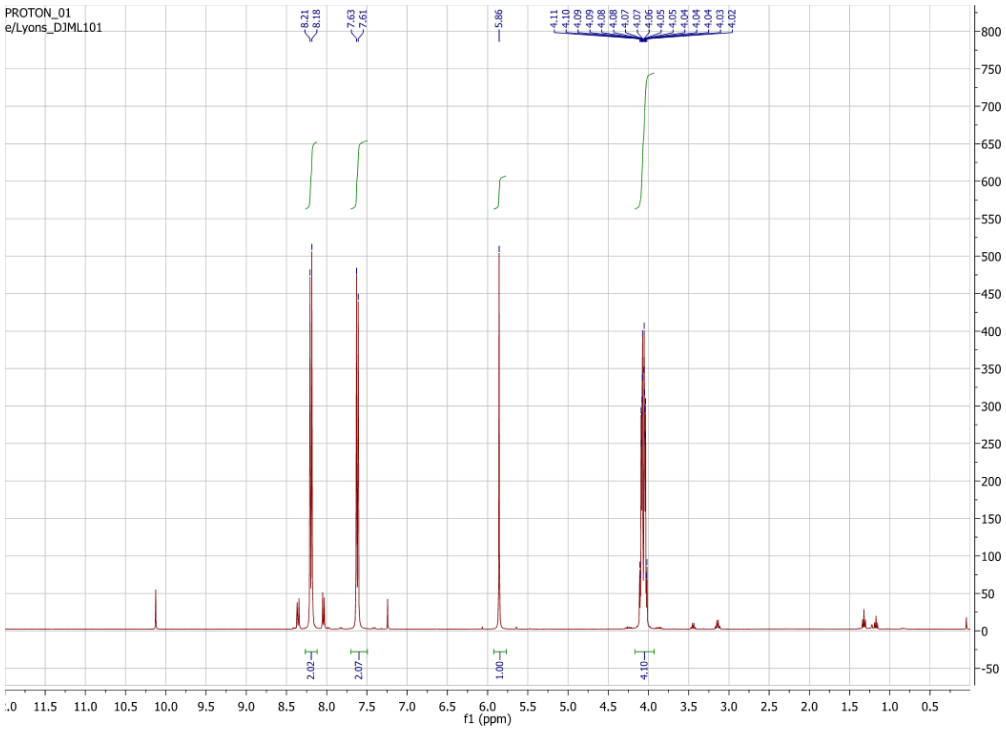
6b

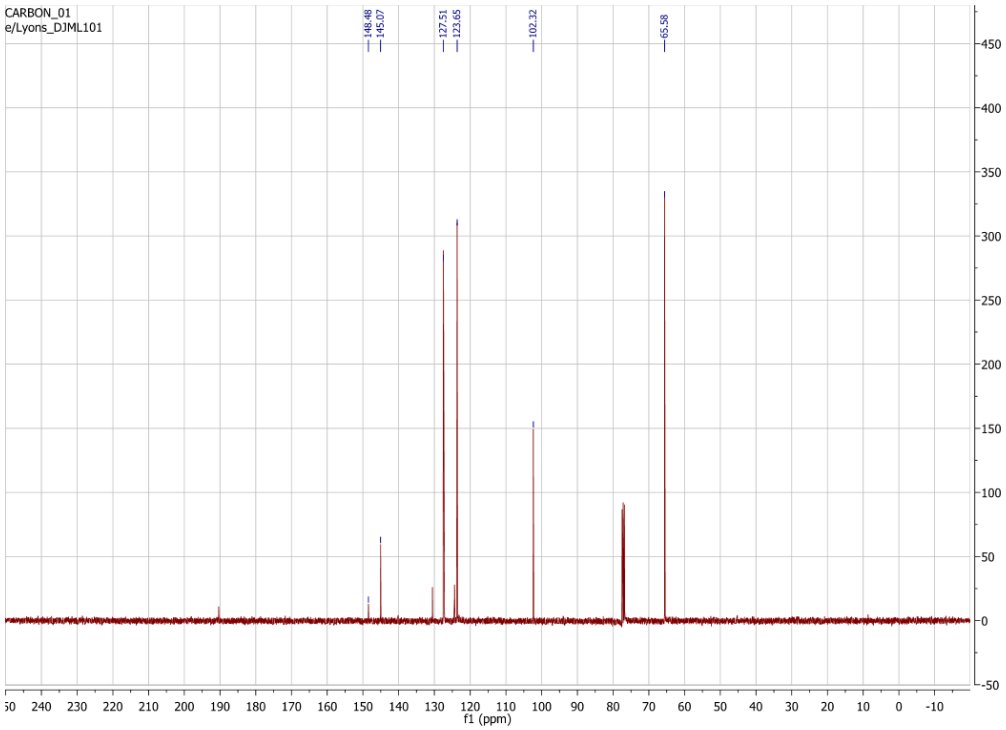

PROTON_01
e/lyons_djml80f11
8.23
8.21
7.67
7.65
6.01
1.33
1.22
1.98
1.96
1.00
6.38
6.59
f1 (ppm)

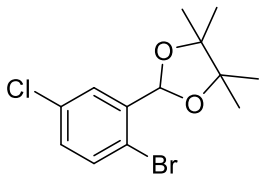

CARBON_01
e/lyons_djml80f11
147.4
127.1
123.6
98.6
83.4
24.2
22.3
f1 (ppm)



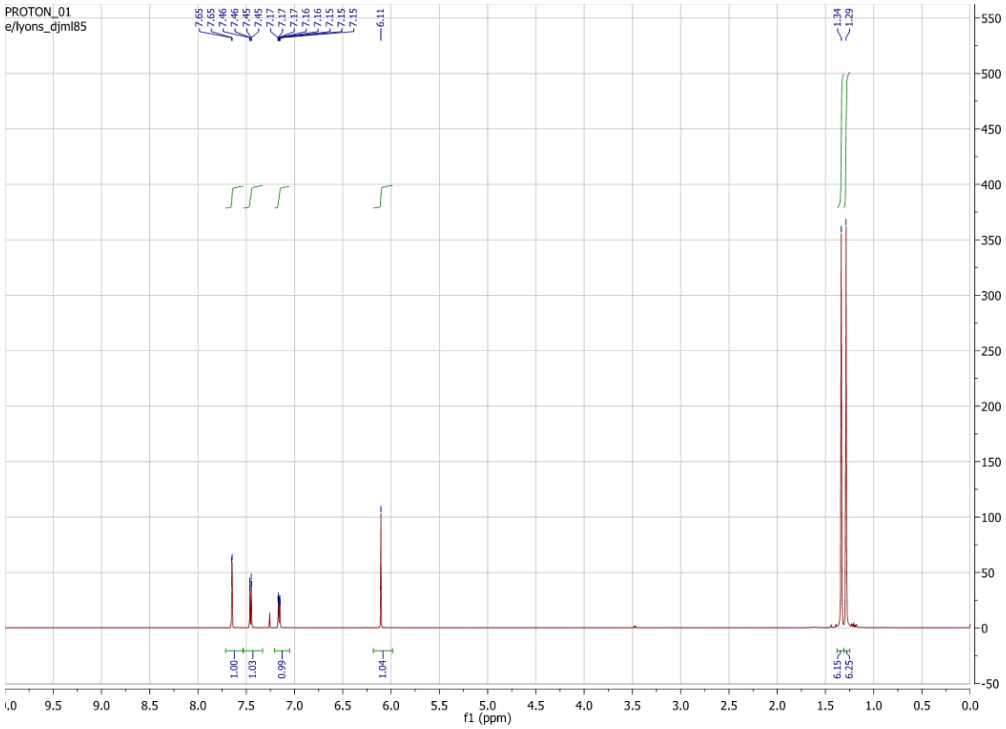
6c

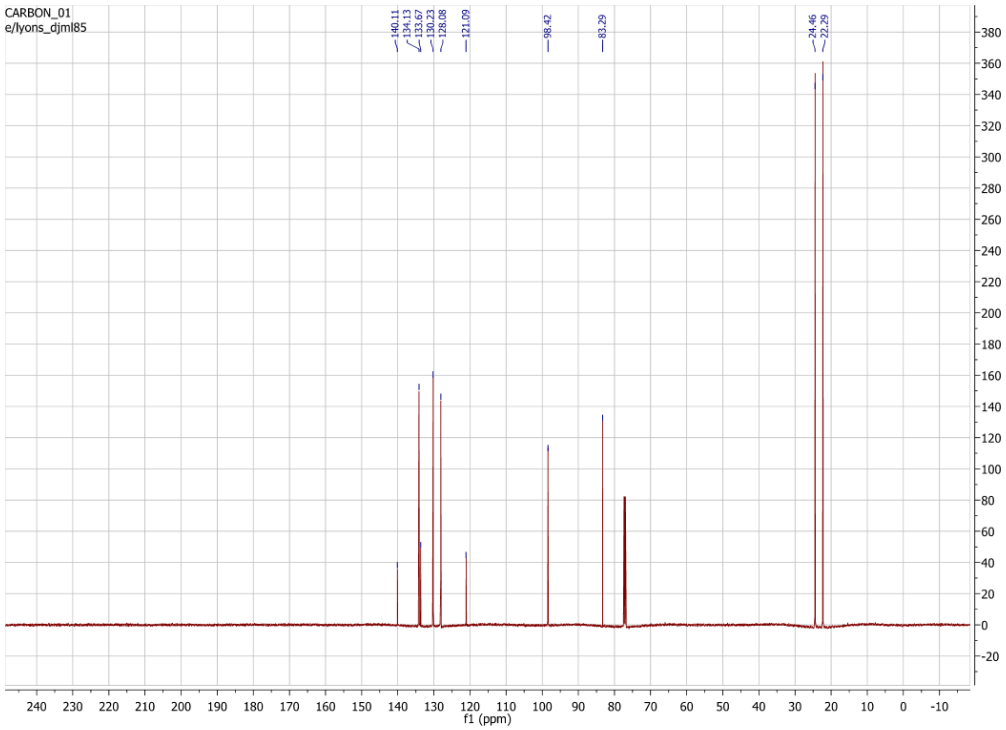

PROTON_01
e/Lyons_DJML101
8.21
8.18
7.63
7.61
5.86
4.11
4.10
4.09
4.09
4.08
4.08
4.07
4.07
4.06
4.05
4.05
4.04
4.04
4.04
4.03
4.02
2.02
2.07
1.00
4.10
f1 (ppm)

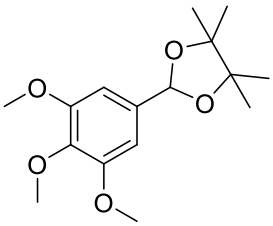

CARBON_01
e/Lyons_DJML101
148.48
145.07
127.51
123.65
102.32
65.58
f1 (ppm)


Cl
O
O
Br

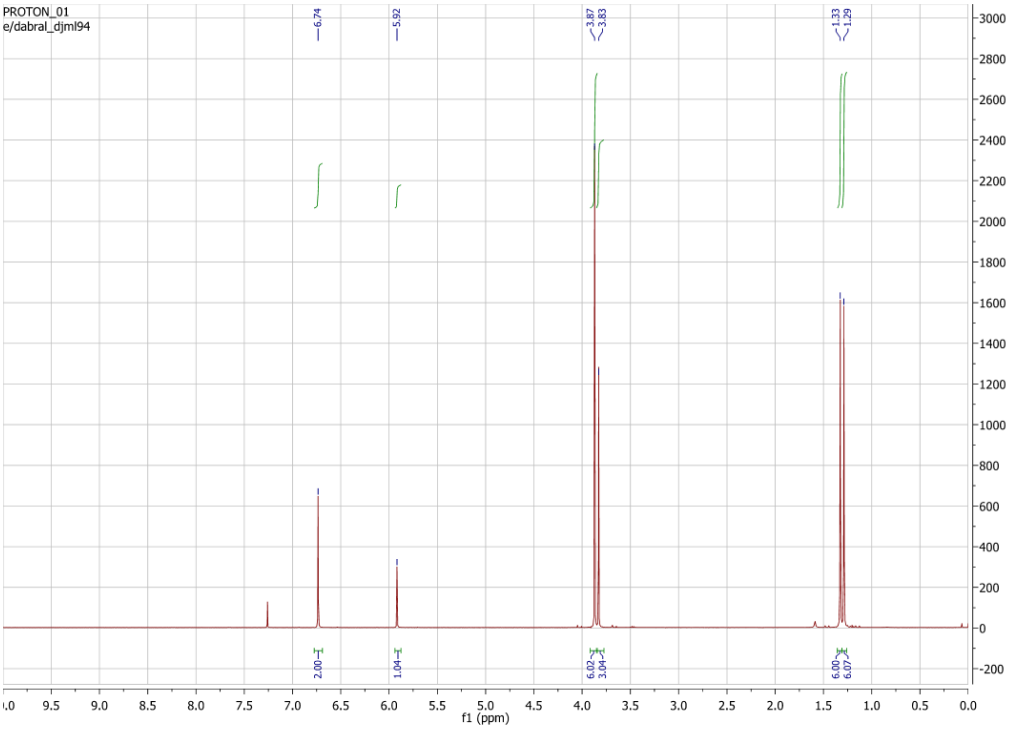
6d

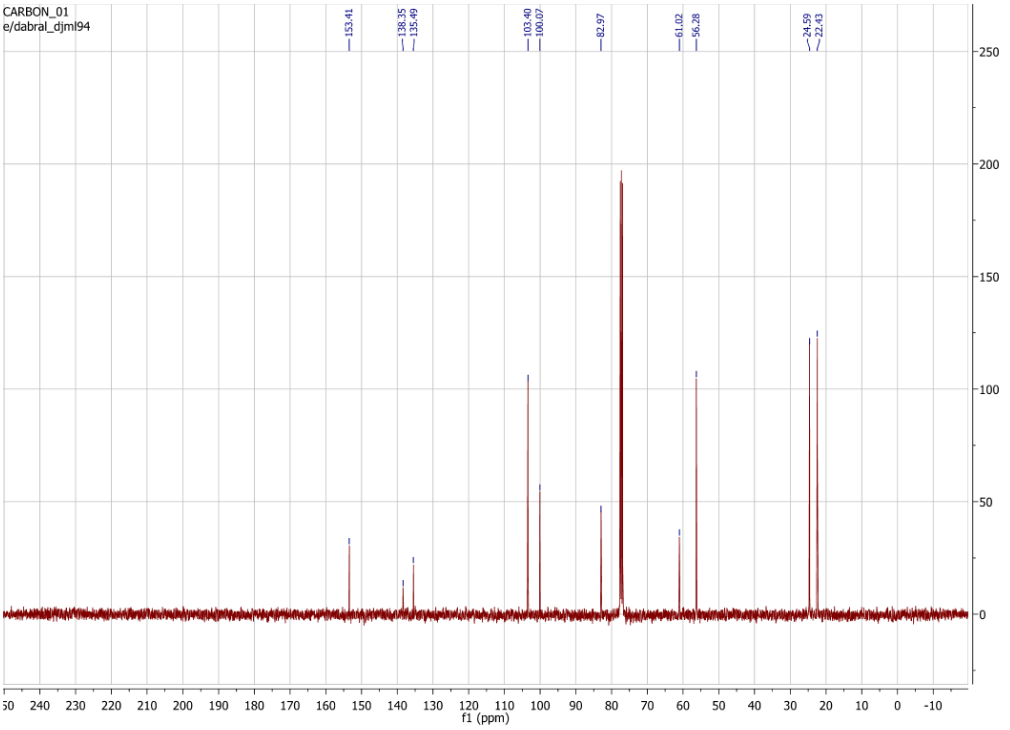

PROTON_01
e/lyons_djml85
7.65
7.65
7.46
7.46
7.45
7.45
7.17
7.17
7.17
7.16
7.16
7.15
7.15
7.15
6.11
1.34
1.29
1.00
1.03
0.99
1.04
6.15
6.25
f1 (ppm)

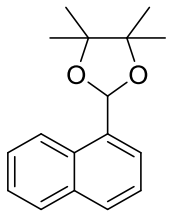

CARBON_01
e/lyons_djml85
140.11
134.13
133.67
130.23
128.08
121.09
98.42
83.29
24.46
22.29
f1 (ppm)



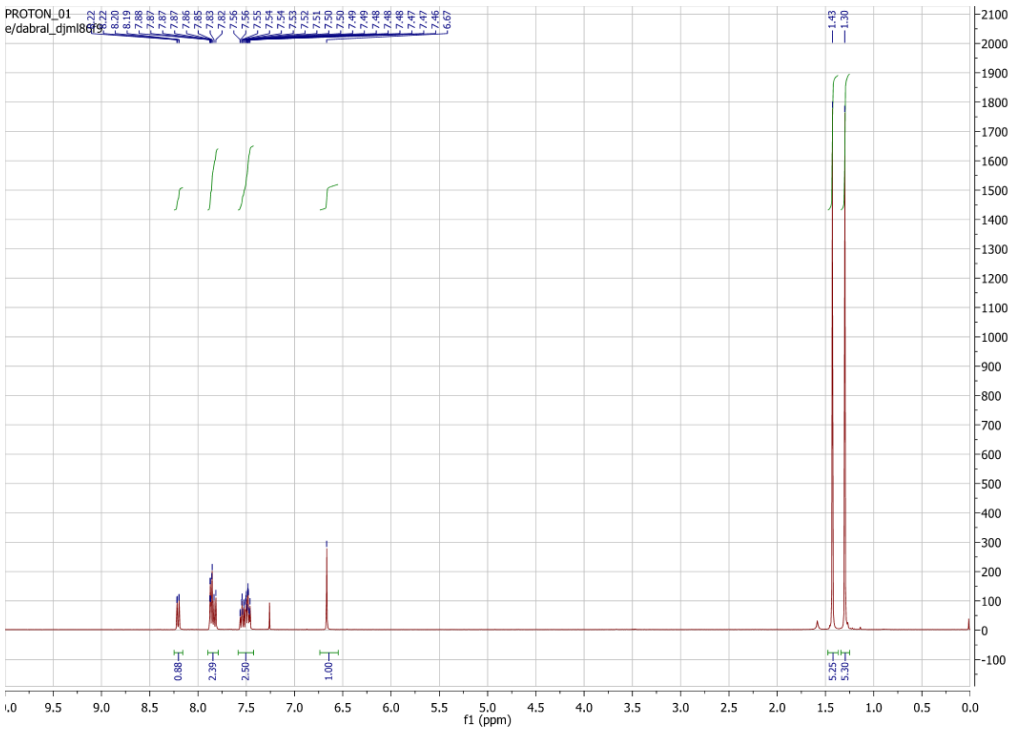
6e

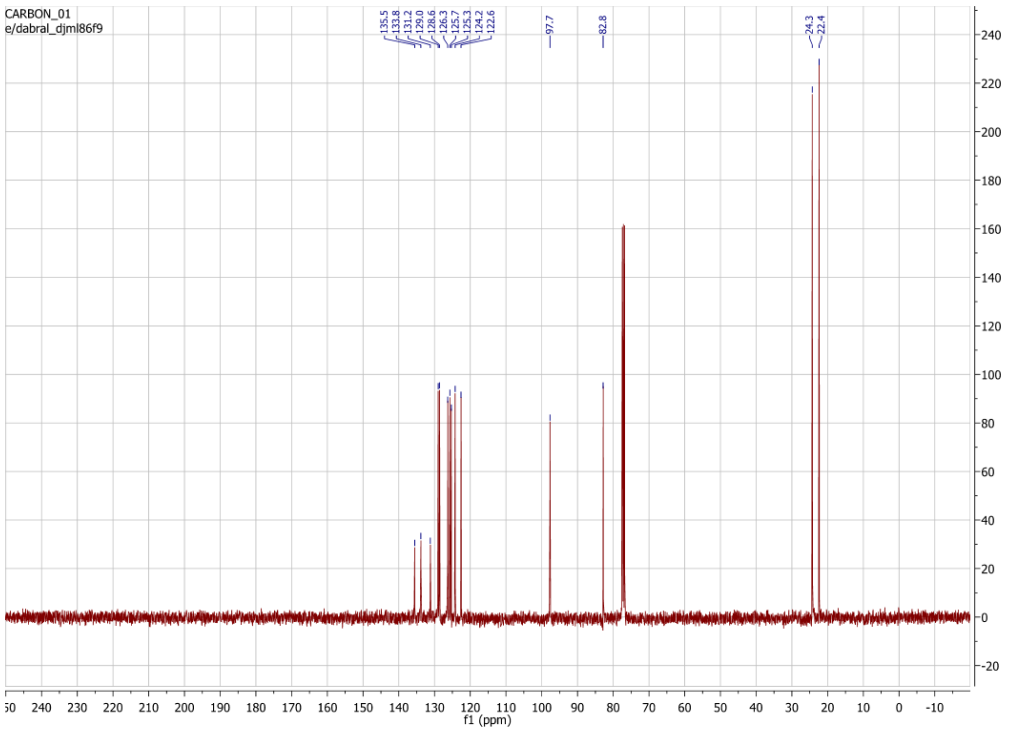

PROTON_01
e/dabral_djml94
6.74
5.92
3.87
3.83
1.33
1.29
2.00
1.04
6.02
3.04
6.00
6.07
f1 (ppm)

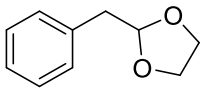

CARBON_01
e/dabral_djml94
153.41
138.35
135.49
103.40
100.07
82.97
61.02
56.28
24.59
22.43
f1 (ppm)



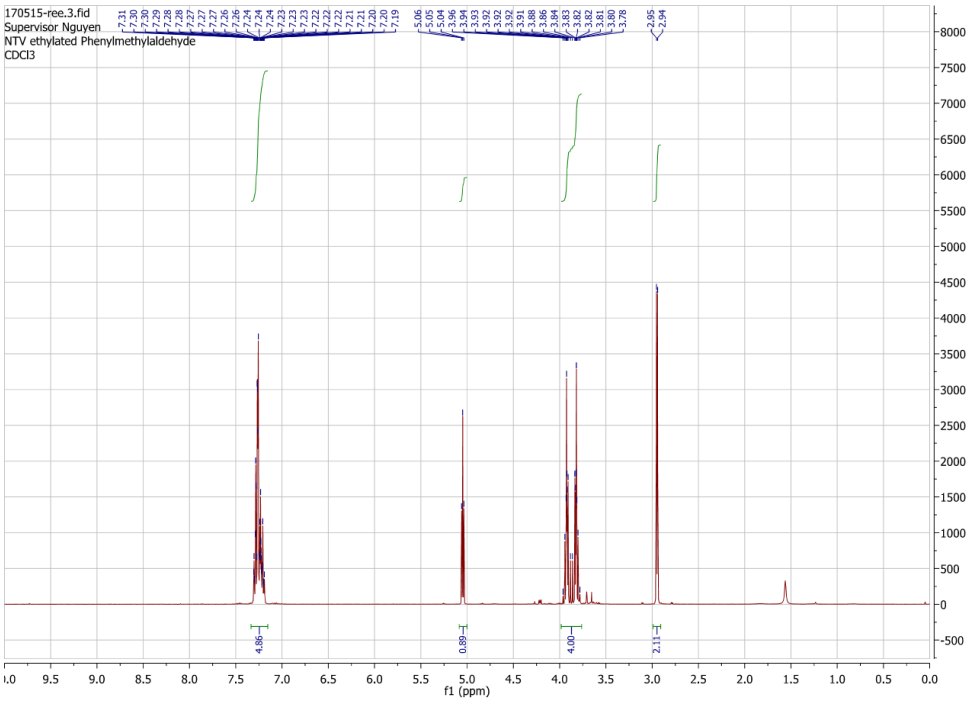
6f

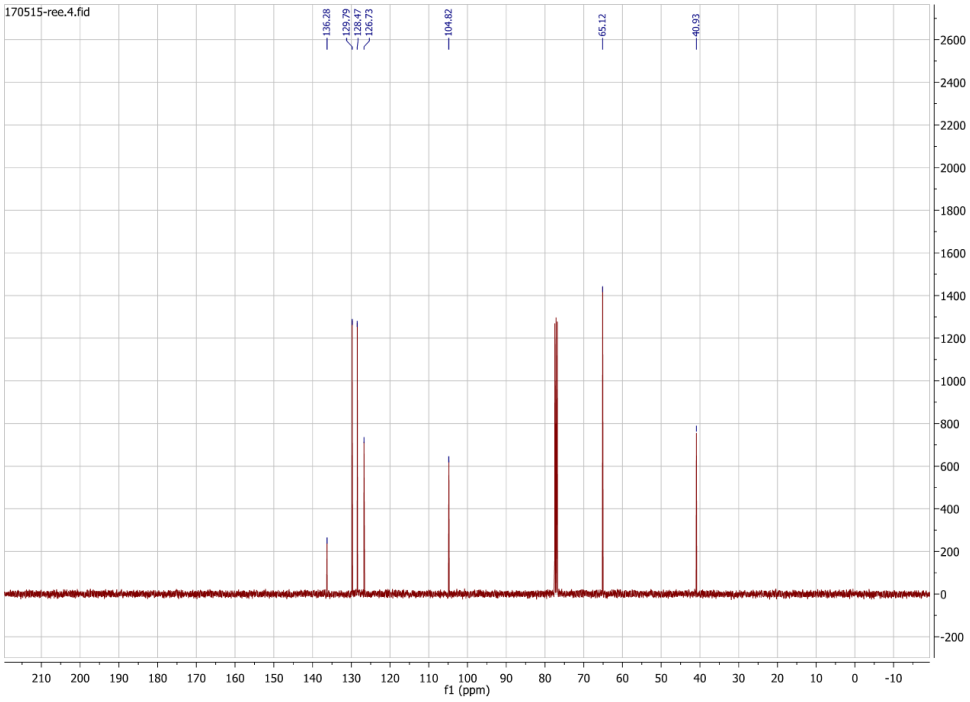

PROTON_01
e/dabral_djml86f9
1.43
1.30
0.88
2.39
2.50
1.00
5.25
5.30
f1 (ppm)


CARBON_01
e/dabral_djml86f9
135.5
133.8
131.2
129.0
126.6
126.3
125.7
125.3
124.2
122.6
97.7
82.8
24.3
22.4
f1 (ppm)



6g


170515-ree.3.fid
Supervisor Nguyen
NTV ethylated Phenylmethylaldehyde
CDCl3
f1 (ppm)


170515-ree.4.fid
136.28
129.79
128.47
126.73
104.82
65.12
40.93
f1 (ppm)