Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

$ZnI_2/Zn(OTf)_2$–TsOH: A Versatile Combined-Acid System for Catalytic Intramolecular Hydrofunctionalization and Polyene Cyclization

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Table of Contents

1. General Information

The reactions were monitored by TLC (glass plates precoated with silica gel 60 F254, Merck). Column chromatography was performed on silica gel Geduran® Si 60 (Merck). ^{1}H and ^{13}C NMR spectra were recorded with Bruker AV-III 400 MHz, Bruker AV-400, Bruker AV-500, or N600 MHz spectrometers and chemical shifts were measured in δ (ppm) with residual solvent peaks as internal standards ($CDCl_3$, δ 7.26 ppm in ^{1}H NMR, δ 77 ppm in ^{13}C NMR). IR spectra were recorded with Thermo Nicolet iS-5 FT-IR spectrophotometer, max in cm^{-1}. Commercial grade reagents and solvents were used without further purification except as indicated below.

2. General Procedures

General Procedure A

To a 4-mL vial equipped with a stirring bar was added 1a (100 mg, 0.567 mmol), ZnI_2 (4.5 mg, 0.014 mmol), $TsOH{\cdot}H_2O$ (2.7 mg, 0.014 mmol) and CH_2Cl_2 (1.1 ml). After stirring at room temperature for the reaction time given in Table 2 or Table 3, the reaction mixture was filtered through a short pad of silica gel, washed with DCM, and then concentrated to give the product.

General Procedure B

To a 4-mL vial equipped with a stirring bar was added 1f (100 mg, 0.475 mmol), $Zn(OTf)_2$ (4.3 mg, 0.012 mmol), $TsOH{\cdot}H_2O$ (2.3 mg, 0.012 mmol) and CH_2Cl_2 (0.95 ml). After stirring at room temperature for the reaction time given in Table 2 or Table 3, the reaction mixture was filtered through a short pad of silica gel, washed with DCM, and then concentrated to give the product.

General Procedure C

To a 10-mL sealed tube equipped with a stirring bar was added 1h (100 mg, 0.999 mmol), $Zn(OTf)_2$ (9.1 mg, 0.025 mmol), $TsOH{\cdot}H_2O$ (4.8 mg, 0.025 mmol) and DCE (2 ml). After heated to reflux for the reaction time given in Table 2 or Table 3, the reaction mixture was filtered through a short pad of silica ge, washed with DCM, and then concentrated to give the product.

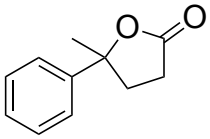
General Procedure D

To a 4-mL vial equipped with a stirring bar was added 3a (100 mg, 0.624 mmol), $Zn(OTf)_2$ (11.3 mg, 0.031 mmol), $TsOH{\cdot}H_2O$ (5.9 mg, 0.031 mmol) and CH_2Cl_2 (2.5 ml). After stirring at room temperature for the reaction time given in Table 2 or Table 3, the reaction mixture was filtered through a short pad of silica gel, washed with DCM, and then concentrated to give the product.

General Procedure E

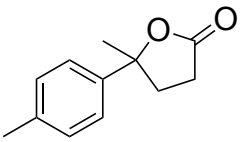
To a 4-mL vial equipped with a stirring bar was added 3e (50 mg, 0.274 mmol), ZnI_2 (8.7 mg, 0.027 mmol), $TsOH{\cdot}H_2O$ (5.1 mg, 0.027 mmol) and CH_2Cl_2 (1.1 ml). After heated to reflux for the reaction time given in Table 2 or Table 3, the reaction mixture was filtered through a short pad of silica gel, washed with DCM, and then concentrated to give the product.

5-methyl-5-phenyldihydrofuran-2(3*H*)-one (2a)[1]



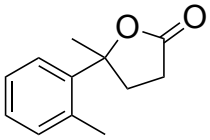
Procedure A, after 16 h to afford 98% yield. Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.41-7.35 (m, 4H), 7.33-7.28 (m, 1H), 2.60-2.69 (m, 1H), 2.54-2.40 (m, 3H), 1.72 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 176.6, 144.3, 128.6, 127.6, 124.1, 87.0, 36.2, 29.4, 29.0.

5-methyl-5-(*p*-tolyl)dihydrofuran-2(3*H*)-one (2b)[1]



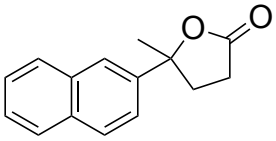
Procedure A, after 16 h to afford 99% yield. Colorless solid; mp 48ºC; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.27 (d, J = 8.6 Hz , 2H), 7.19 (d, J = 8.1 Hz , 2H), 2.66-2.60 (m, 1H), 2.55-2.46 (m, 2H), 2.45-2.38 (m, 1H), 2.36 (s, 3H), 1.72 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 176.7, 141.4, 137.3, 129.3, 124.1, 87.1, 36.2, 29.4, 29.0, 21.0.

5-methyl-5-(o-tolyl)dihydrofuran-2(3H)-one (2c)[1]



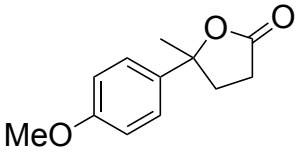
Procedure A, after 16 h to afford 94% yield. Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.45 (d, J = 7.7 Hz , 1H), 7.22-7.16 (m, 3H), 2.72-2.64 (m, 1H), 2.62-2.48 (m, 3H), 2.44 (s, 3H), 1.74 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 176.2, 142.0, 133.8, 132.5, 127.8, 126.1, 124.7, 87.9, 35.0, 28.8, 27.8, 21.6.

5-methyl-5-(naphthalen-2-yl)dihydrofuran-2(3*H*)-one (2d)[2]



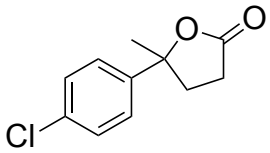
Procedure A, after 16 h to afford 98% yield. Colorless solid; mp 78ºC ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.89-7.81 (m, 4H), 7.53-7.47 (m, 2H), 7.43 (dd, J = 8.6 Hz , J = 1.9 Hz, 1H), 2.69-2.62 (m, 1H), 2.60-2.43 (m, 3H), 1.80 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 176.7, 141.5, 133.0, 132.6, 128.7, 128.2, 127.6, 126.6, 126.4, 122.7, 122.5, 87.1, 36.1, 29.3, 29.0.

5-(4-methoxyphenyl)-5-methyldihydrofuran-2(3*H*)-one (2e)[1]



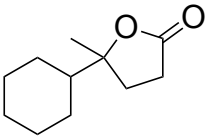
Procedure A, after 16 h to afford 94% yield. Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.30 (d, J = 8.9 Hz , 2H), 6.90 (d, J = 8.6 Hz , 2H), 3.80 (s, 3H), 2.62-2.59 (m, 1H), 2.55-2.44 (m, 2H), 2.43-2.35 (m, 1H), 1.70 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 176.6, 159.0, 136.3, 125.4, 113.9, 87.0, 55.3, 36.1, 29.5, 29.1.

5-(4-chlorophenyl)-5-methyldihydrofuran-2(3*H*)-one (2f)[1]



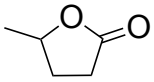
Procedure B, after 16 h to afford 99% yield. Colorless solid; mp 50ºC; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.33-7.27 (m, 4H), 2.66-2.59 (m, 1H), 2.51-2.36 (m, 3H), 1.67 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 176.2, 142.9, 133.5, 128.8, 125.7, 86.5, 36.0, 29.3, 28.9.

5-cyclohexyl-5-methyldihydrofuran-2(3*H*)-one (2g)



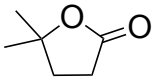
Procedure A, after 16 h to afford 98% yield. Colorless solid; mp 57ºC; ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.61-2.53 (m, 1H), 2.51-2.43 (m, 2H), 2.11-2.04 (m, 1H), 1.88-1.81 (m, 1H), 1.81-1.71 (m, 3H), 1.65 (t, J = 15.0 Hz, 1H), 1.48 (tt, J = 12.1 Hz, J = 2.9 Hz, 1H), 1.25 (s, 3H), 1.23-1.13 (m, 1H), 1.09 (tt, J = 12.6 Hz, J = 3.2 Hz, 1H), 1.05-0.97 (m, 1H), 0.97-0.89 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 176.9, 89.3, 47.6, 31.3, 29.1, 27.2, 27.1, 26.3, 26.2, 26.1, 22.6; IR (film) ν_{max} 2922, 2850, 1756, 1385, 1207, 1164, 970, 933 cm^{-1}; HRMS-EI (*m/z*): calculated for $C_{11}H_{18}O_2$ [M^+] 182.1307, found 182.1309

5-methyldihydrofuran-2(3*H*)-one (2h)[3]



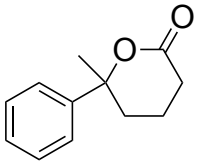
Procedure C, after 16 h to afford 98% yield (GC yield). Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.67-4.60 (m, 1H), 2.60-2.49 (m, 2H), 2.39-2.32 (m, 1H), 1.87-1.79 (m, 1H), 1.41 (d, *J* = 6.6 Hz, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 177.3, 77.2, 29.6, 29.0, 21.0

5,5-dimethyldihydrofuran-2(3*H*)-one (2i)[3]



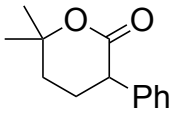
Procedure A, after 16 h to afford 94% yield. Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.61 (t, *J* = 8.3 Hz, 4H), 2.04 (t, *J* = 8.2 Hz, 1H), 1.42 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 176.7, 84.6, 34.6, 29.3, 27.7.

6-methyl-6-phenyltetrahydro-2*H*-pyran-2-one (2k)[4]



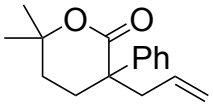
Procedure B, after 24 h to afford 92% yield. White solid; mp 72ºC; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 (m, 5H); 2.44 (m, 2H); 2.30 (m, 1H); 1.99 (m, 1H); 1.77 (m, 1H); 1.66 (s, 3H); 1.57 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.27, 144.40, 128.47, 127.14, 124. 21, 34.13, 31.06, 28.85, 16.35

6,6-dimethyl-3-phenyltetrahydro-2*H*-pyran-2-one (2l)



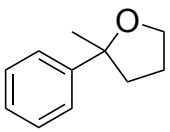
Procedure B, after 16 h to afford 94% yield. Colorless solid; mp 116ºC; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.35 (t, *J* = 7.6 Hz, 2H), 7.28 (t, *J* = 7.5 Hz, 1H), 7.23 (d, *J* = 7.2 Hz, 2H), 3.67 (dd, *J* = 9.7 Hz, *J* = 7.0 Hz, 1H), 2.24-2.18 (m, 1H), 2.16-2.08 (m, 1H), 1.95-1.84 (m, 2H), 1.51 (d, *J* = 9.7 Hz, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.2, 140.0, 128.8, 128.1, 127.1, 83.0, 47.2, 33.4, 29.9, 28.4, 26.7; IR (film) ν_{max} 2974, 1708, 1206, 1110, 702 cm^{-1}; HRMS-EI (*m/z*): calculated for $C_{13}H_{16}O_2$ [M$^+$] 204.1150, found 204.1146

3-allyl-6,6-dimethyl-3-phenyltetrahydro-2*H*-pyran-2-one (2m)



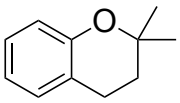
Procedure A, after 16 h, the reaction mixture was filter through short pad of Al_2O_3 with CH_2Cl_2 and concentrated to afford 94% yield. Yellow gummy oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.34-7.28 (m, 4H), 7.25-7.21 (m, 1H), 5.80-5.71 (m, 1H), 5.13 (d, *J* = 6.2 Hz, 1H), 5.10 (s, 1H), 2.89 (dd, *J* = 13.8 Hz, *J* = 5.6 Hz, 1H), 2.49 (dd, *J* = 13.4 Hz, *J* = 8.6 Hz, 1H), 2.28 (td, *J* = 13.6 Hz, *J* = 3.9 Hz, 1H), 2.06 (dt, *J* = 14.8 Hz, *J* = 3.5 Hz, 1H), 1.66 (td, *J* = 14.0 Hz, *J* = 3.3 Hz, 1H), 1.59 (dt, *J* = 14.3 Hz, *J* = 4.3 Hz, 1H), 1.37 (s, 3H), 1.28 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 173.6, 142.2, 134.1, 128.7, 127.0, 126.3, 119.1, 83.3, 50.9, 45.4, 31.2, 30.5, 28.2, 28.0; IR (film) ν_{max} 2978, 1718, 1276, 1114, 933, 761, 700 cm^{-1}; HRMS (EI) *(m/z)*: calculated for $C_{16}H_{20}O_2$ [M$^+$] 244.1463, found 244.1470.

2-methyl-2-phenyltetrahydrofuran (2n)[1]



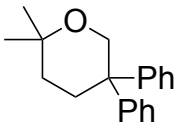
Procedure B, after 14 h to afford 94% yield. Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.6 Hz, 2H), 7.34 (t, *J* = 7.6 Hz, 2H), 7.23 (d, *J* = 7.2 Hz, 1H), 4.07-4.02 (m, 1H), 3.97-3.91 (m, 1H), 2.26-2.21 (m, 1H), 2.07-1.96 (m, 2H), 1.87-1.78 (m, 1H), 1.55 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 148.2, 128.1, 126.4, 124.7, 84.3, 67.6, 39.5, 29.8, 25.8.

2,2-dimethylchromane (2o)[5]



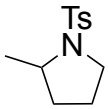
Procedure B, after 16 h to afford 94% yield. Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.06 (m, 2H); 6.79 (m, 2H); 2.77 (t, *J* = 6.8 Hz, 2H); 2.83 (t, *J* = 6.8 Hz, 2H); 1.33 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.05, 129.50, 127.30, 120.96, 119.66, 117.30, 74.13, 32.88, 26.95, 22.52

2,2-dimethyl-5,5-diphenyltetrahydro-2*H*-pyran (2p)[6]



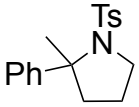
Procedure B, after 24 h to afford 93% yield. Colorless solid; mp 88ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (m, 8H); 7.18 (m, 2H); 4.07 (s, 2H); 2.44 (m, 2H); 1.41 (m, 2H); 1.24 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.52, 128.16, 128.00, 126.00, 71.27, 69.04, 45.94, 32.63, 30.90, 26.42

2-methyl-1-tosylpyrrolidine (2q)[7]



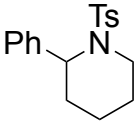
Procedure C, after 16 h to afford 92% yield. Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.68 (d, *J* = 12.6 Hz, 2H), 7.27 (d, *J* = 12 Hz, 2H); 3.66 (m, 1H); 3.39 (m, 1H); 3.22 (m, 1H); 1.79 (m, 1H); 1.64 (m, 1H); 1.48 (m, 2H); 1.27 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 142.97, 134.76, 129.38, 127.22, 55.989, 48.85, 33.28, 23.69, 22.62, 21.26

2-methyl-2-phenyl-1-tosylpyrrolidine (2r)[7]



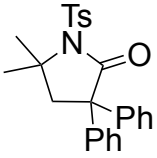
Procedure B, after 1 h to afford 92% yield. Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.46 Hz, 2H); 7.40 (d, *J* = 8.46 Hz, 2H); 7.29 (m, 2H); 7.22 (m, 3H) ^{13}C NMR (100 MHz, $CDCl_3$): δ 129.21, 128.00, 127.06, 126.56, 125.79, 49.75, 45.77, 26.39, 22.41, 21.41

2-phenyl-1-tosylpiperidine (2s)[8]



Procedure C, after 3 h to afford 93% yield. Colorless solid; mp 132ºC; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.3 Hz, 2H), 7.36-7.28 (m, 6H), 7.25-7.21 (m, 1H), 5.27 (d, *J* = 4.3 Hz, 1H), 3.84 (d, *J* = 15.0 Hz, 1H), 3.01 (t, *J* = 14.6 Hz, 1H), 2.42 (s, 3H), 2.21 (d, *J* = 13.3 Hz, 1H), 1.70-1.62 (m, 1H), 1.53-1.47 (m, 1H), 1.45-1.36 (m, 2H), 1.34-1.25 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 143.0, 138.9, 138.7, 129.7, 128.6, 127.02, 127.00, 126.8, 55.3, 41.9, 27.3, 24.3, 21.5, 19.0.

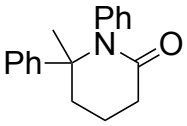
5,5-dimethyl-3,3-diphenyl-1-tosylpyrrolidin-2-one (2t)



Procedure B, after 1 h to afford 99% yield. Colorless solid; mp 186ºC; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.6 Hz, 2H), 7.31-7.22 (m, 12H), 2.92 (s, 2H), 2.40 (m, 3H), 1.30 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 175.3, 143.0, 142.4, 138.8, 128.9, 128.5, 127.8, 127.5, 127.4, 90.4, 61.9, 50.1, 28.4, 21.5; IR (film) ν_{max} 2978, 1620, 1322, 1160, 1140, 1086, 780, 543 cm^{-1}; HRMS-ESI (*m/z*): calculated for $C_{25}H_{25}NO_3NaS$ $[M + Na]^+$ 442.1447, found 442.1443

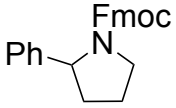
6-methyl-1,6-diphenylpiperidin-2-one (2u)

Procedure C, after 24 h to afford 91% yield. Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 ~6.97 (m, 10H), 2.68 (m, 2H), 2.08 (m, 2H), 1.71 (m, 2H), 1.52 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 172.0, 145.4, 140.1, 129.2, 128.6, 128.4, 127.3,



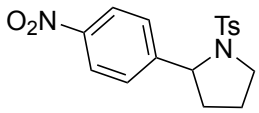
127.2, 126.6, 64.6, 40.8, 32.0, 28.7, 16.7; IR (film) ν_{max} 2950, 1647, 1494, 1443, 1381, 762, 697 cm^{-1}; HRMS-ESI (*m/z*): calculated for $C_{18}H_{19}NONa$ $[M+Na]^+$ 288.1359, found 288.1357

(9*H*-fluoren-9-yl)methyl 2-phenylpyrrolidine-1-carboxylate (2v)[7]



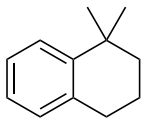
To a 10-mL sealed tube equipped with a stirring bar was added **1v** (50 mg, 0.135 mmol), $Zn(OTf)_2$ (5 mg, 0.014 mmol), $TsOH{\cdot}H_2O$ (2.5 mg, 0.014 mmol) and in DCE (0.27 ml). After heated to reflux for 36 h, the reaction mixture was filtered through a short pad of silica gel, washed with DCM, and then concentrated to give crude mixture. The crude mixture was further purified by flash chromatography (SiO_2) and concentrated to give **2v** (46 mg, 92%). Yellow oil; ^1H NMR (500 MHz, $CDCl_3$) δ 7.81~7.62 (m, 3H), 7.50~6.96 (m, 10H), 5.02 (m, 1H), 4.51~3.94 (m, 3H), 3.86~3.52 (m, 2H), 2.42~2.29 (m, 1H), 2.09~1.81 (m, 3H) ; ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.3, 154.8, 144.1, 144.0, 143.9, 141.3, 141.1, 141.0, 128.6, 128.4, 127.6, 127.4, 127.3, 127.0, 126.9, 125.4, 125.1, 125.0, 119.9, 119.7, 119.6, 67.4, 66.9, 61.2, 61.0, 47.7, 47.5, 47.1, 35.8, 34.7, 23.5, 22.5

2-(4-nitrophenyl)-1-tosylpyrrolidine (2w)



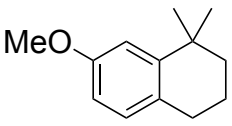
To a 10-mL sealed tube equipped with a stirring bar was added **1w** (50 mg, 0.144 mmol), $Zn(OTf)_2$ (10.5 mg, 0.029 mmol), $TsOH{\cdot}H_2O$ (32.9 mg, 0.173 mmol) and in DCE (0.29 ml). After heated to reflux for 36 h, the reaction mixture was filtered through a short pad of silica gel, washed with EA, and then concentrated to give crude mixture. The crude mixture was further purified by flash chromatography (SiO_2) and concentrated to give **2w** (49mg, 91%). White solid; mp 179 ºC; ^1H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, *J* = 8.51 Hz, 2H), 7.69 (d, *J* = 7.66 Hz, 2H), 7.50 (d, *J* = 8.08 Hz, 2H), 7.32 (d, *J* = 7.66 Hz, 2H), 4.84~4.78 (m, 1H), 3.70~3.62 (m, 1H), 3.48~3.39 (m, 1H), 2.44 (s, 3H), 2.15~2.04 (m, 1H), 1.91~1.75 (m, 2H), 1.75~1.63 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.8, 147.1, 143.9, 134.3, 129.8, 127.5, 127.1, 123.7, 62.8, 49.6, 35.8, 24.1, 21.6 IR (film) ν_{max} 3650, 3447, 1654, 1637, 1341, 1156, 1085 cm^{-1}; HRMS-ESI (*m/z*): calculated for $C_{17}H_{18}N_2O_4NaS$ $[M+Na]^+$ 369.0879, found 369.0870.

1,1-dimethyl-1,2,3,4-tetrahydronaphthalene (4a)[9]



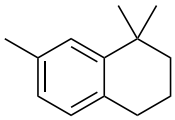
Procedure D, after 1 h to afford 99% yield. Colorless oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.40 (m, 1H); 7.21 (m, 1H); 7.13 (m, 2H) 2.84 (m, 2H); 1.88 (m, 2H); 1.75 (m, 2H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 128.94, 126.51, 125.70, 125.14, 39.26, 31,78, 30,66, 19.65

7-methoxy-1,1-dimethyl-1,2,3,4-tetrahydronaphthalene (4b)[9]



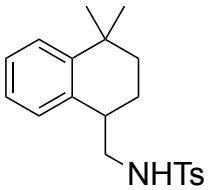
Procedure D, after 1 h to afford 99% yield. Colorless oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.02 (d, *J* = 8.28 Hz, 1H); 6.94 (m, 1H); 6.72 (dd, *J* = 8.31 Hz, 1H) 3.84 (s, 3H); 2.76 (t, *J* = 12.2 Hz, 2H); 1.85 (m, 2H); 1.71 (m, 2H); 1.35 (s, 6H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.70, 147.00, 129.77, 128.33, 112.13, 110.91, 55.15, 39.23, 34.05, 31.83, 29.87, 19.85

1,1,7-trimethyl-1,2,3,4-tetrahydronaphthalene (4c)



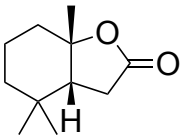
Procedure D, after 1 h to afford 95% yield. Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.99 (m, 3H); 2.72 (t, *J* = 12.9 Hz, 2H); 2.31 (s, 3H) 1.79 (m, 2H); 1.65 (m, 2H); 1.28 (s, 6H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.58, 135.01, 133.00, 128.93, 127.12, 126.17, 39.45, 33.75, 31.86, 30.35, 21.21, 19.8; IR (film) ν_{max} 3446, 2957, 2927, 1505, 1456 cm^{-1} ;HRMS-EI (*m/z*): calculated for $C_{13}H_{18}$ [M^+] 174.1409, found 174.1413

***N*-((4,4-dimethyl-1,2,3,4-tetrahydronaphthalen-1-yl)methyl)-4-methylbenzenesulfonamide (4d)**



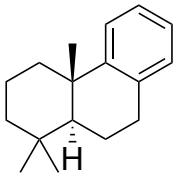
Procedure D, after 16 h to afford 95% yield. White solid; mp 132ºC;^{1}H NMR (600 MHz, $CDCl_3$): δ 7.78 (d, *J* = 7.49 Hz, 2H); 7.31 (m, 3H); 7.17 (t, *J* = 7.3 Hz, 1H); 7.05 (m, 2H); 5.03 (t, *J* = 6.6 Hz; 1H); 3.16 (m, 2H); 2.93 (m, 1H), 2.43 (s, 3H); 1.87 (m, 2H); 1.67 (m, 1H); 1.53 (m, 1H); 1.27 (s, 3H); 1.25 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.45, 143.27, 138.93, 129.64, 128.46, 127.00, 126.85, 126.69, 125.55, 47.85, 38.39, 34.52, 33.71, 31.86, 31.41, 21.43; IR (film) ν_{max} 3285, 2957, 2930, 1756, 1323, 1158, 1093, 662, 550 cm^{-1}; HRMS-ESI (*m/z*): Calcd. for $C_{20}H_{25}NO_2S$ [M^+] 343.16, found [M + Na]$^+$ 366.1496

(3a*R*,7a*S*)-4,4,7a-trimethylhexahydrobenzofuran-2(3*H*)-one (4e)[10]



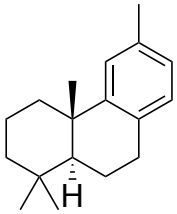
Procedure E, after 70 h, the crude mixture was further purified by flash chromatography (SiO_2) to afford 94% yield. Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.49 (dd, *J* = 17.2 Hz, *J* = 12.6 Hz, 1H), 2.41 (dd, *J* = 17.5 Hz, *J* = 8.4 Hz, 1H), 2.05 (dd, *J* = 12.7 Hz, *J* = 8.1 Hz, 1H), 1.85 (d, *J* = 13.4 Hz, 1H), 1.63-1.57 (m, 1H), 1.51 (s, 3H), 1.50-1.23 (m, 4H), 1.04 (s, 3H), 0.90 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 175.8, 86.1, 51.9, 34.7, 33.6, 33.3, 32.2, 30.1, 28.4, 26.9, 18.9.

(4a*S*,10a*S*)-1,1,4a-trimethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthrene (4f)[11]



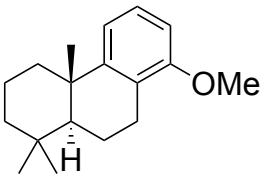
Procedure D, after 44 h to afford 92% yield. Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.29 (d, *J* = 7.7 Hz, 1H), 7.16 (t, *J* = 7.3 Hz, 1H), 7.10 (t, *J* = 7.6 Hz, 1H), 7.07 (d, *J* = 7.5 Hz, 1H), 2.98 (dd, *J* = 17.0 Hz, *J* = 6.9 Hz, 1H), 2.94-2.86 (m, 1H), 2.33 (d, *J* = 12.9 Hz, 1H), 1.95-1.89 (m, 1H), 1.84 -1.71 (m, 2H), 1.68-1.61 (m, 1H), 1.52 (d, *J* = 13.3 Hz, 1H), 1.44 (td, *J* = 13.4 Hz, *J* = 3.4 Hz, 1H), 1.38 (dd, *J* = 12.3 Hz, *J* = 2.4 Hz, 1H), 1.31-1.25 (m, 1H), 1.23 (s, 3H), 0.99 (s, 3H), 0.97 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 150.2, 135.3, 129.1, 125.6, 125.2, 124.4, 50.3, 41.7, 38.9, 37.9, 33.5, 33.4, 30.5, 24.9, 21.7, 19.4, 19.1.

(4a*S*,10a*S*)-1,1,4a,6-tetramethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthrene (4g)[12]



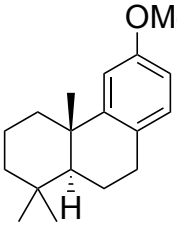
Procedure D, after 14 h to afford 98% yield. Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.11 (s, 1H), 6.98 (d, *J* = 7.8 Hz, 1H), 6.94 (t, *J* = 7.8 Hz, 1H), 2.95 (dd, *J* = 16.7 Hz, *J* = 6.7 Hz,1H), 2.91-2.83 (m, 1H), 2.39-2.31 (m, 1H), 2.34(s, 3H), 1.94-1.89 (m, 1H), 1.83 -1.70 (m, 2H), 1.68-1.63 (m, 1H), 1.53 (d, *J* = 13.1 Hz, 1H), 1.44 (td, *J* = 13.3 Hz, *J* = 3.3 Hz, 1H), 1.38 (dd, *J* = 12.7 Hz, *J* = 2.2 Hz, 1H), 1.31-1.25 (m, 1H), 1.23 (s, 3H), 0.99 (s, 3H), 0.97 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 150.0, 134.8, 132.1, 129.0, 126.1, 125.0, 50.5, 41.8, 38.9, 37.8, 33.5, 33.4, 30.1, 24.9, 21.7, 21.4, 19.4, 19.2.

(4a*S*,10a*S*)-8-methoxy-1,1,4a-trimethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthrene (4h)[13]



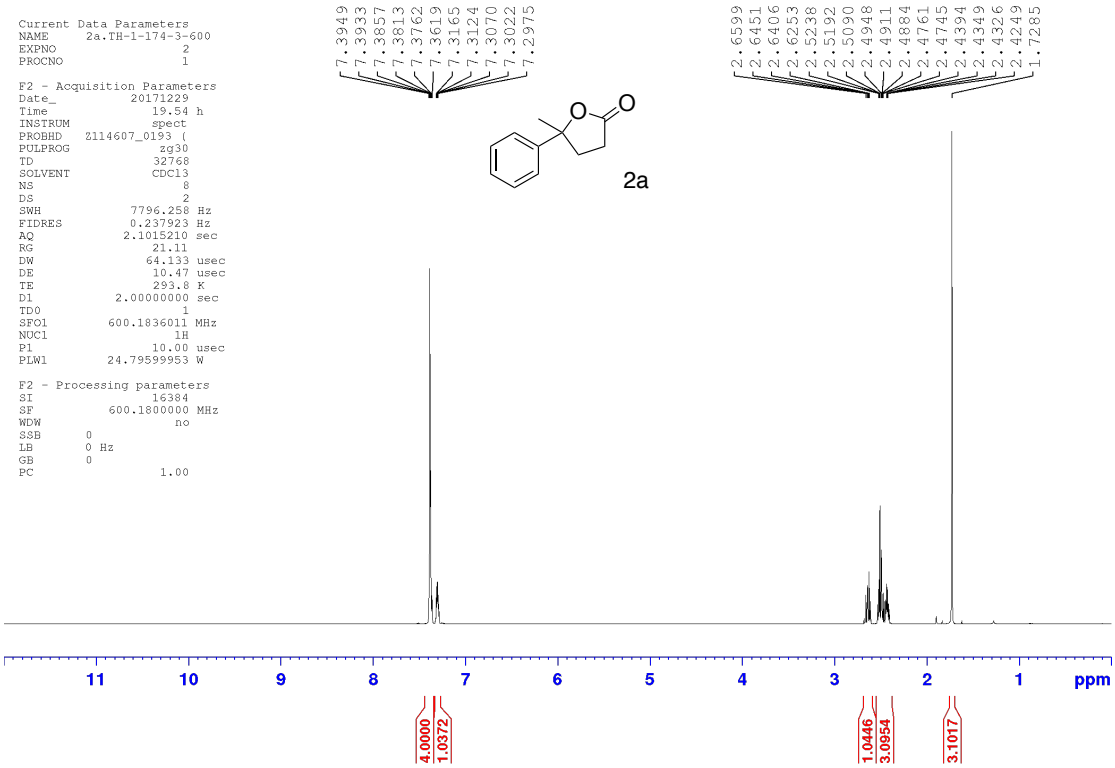
Procedure D, after 23 h to afford 98% yield. Colorless solid; mp 109ºC; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.15 (t, *J* = 7.8 Hz, 1H), 6.94 (d, *J* = 8.0 Hz, 1H), 6.67 (d, *J* = 7.8 Hz, 1H), 3.83 (s, 3H), 2.92 (dd, *J* = 17.8 Hz, *J* = 6.7 Hz, 1H), 2.67-2.59 (m, 1H), 2.31 (d, *J* = 12.6 Hz, 1H), 1.98-1.92 (m, 1H), 1.80 -1.73 (m, 1H), 1.70-1.61 (m, 2H), 1.51 (d, *J* = 13.8 Hz, 1H), 1.41 (td, *J* = 13.2 Hz, *J* = 3.5 Hz, 1H), 1.35 (dd, *J* = 12.5 Hz, *J* = 1.8 Hz, 1H), 1.28-1.21 (m, 1H), 1.23 (s, 3H), 0.98 (s, 3H), 0.96 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 157.1, 151.6, 126.0, 124.3, 116.6, 106.3, 55.2, 49.9, 41.7, 39.1, 37.8, 33.5, 33.4, 24.8, 24.6, 21.7, 19.4, 18.5.

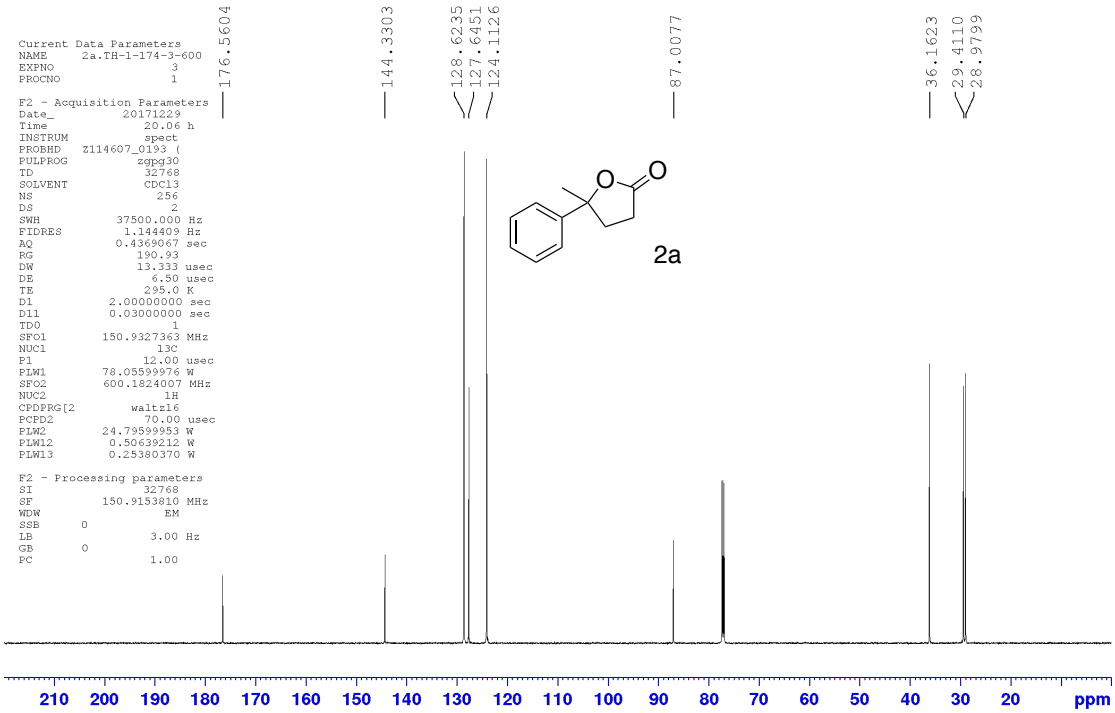
(4a*S*,10a*S*)-6-methoxy-1,1,4a-trimethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthrene (4i)[11]

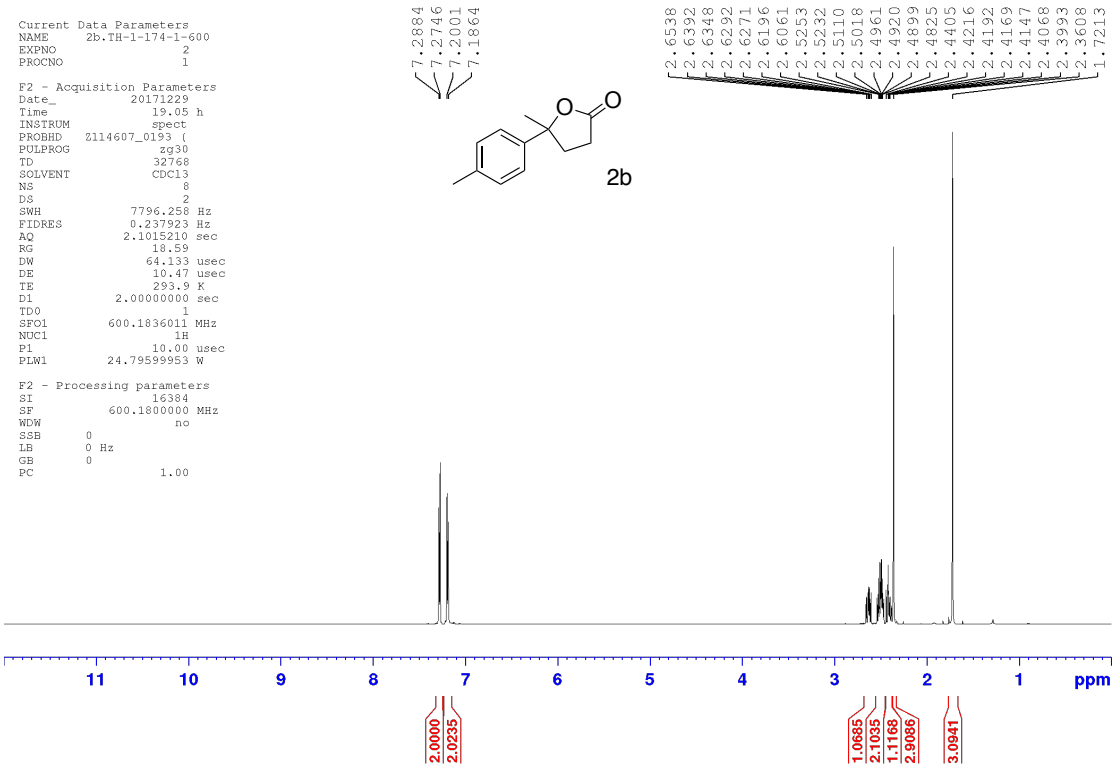


Procedure D, after 14 h to afford 99% yield. Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 6.98 (d, *J* = 8.6 Hz, 1H), 6.84 (d, *J* = 2.4 Hz, 1H), 6.68 (dd, *J* = 8.6 Hz, *J* = 2.9 Hz, 1H), 3.79 (s, 3H), 2.91 (dd, *J* = 16.7 Hz, *J* = 6.8 Hz,1H), 2.85-2.78 (m, 1H), 2.27 (d, *J* = 13.0 Hz, 1H), 1.92-1.86 (m, 1H), 1.80 -1.67 (m, 2H), 1.66-1.61 (m, 1H), 1.50 (d, *J* = 13.3 Hz, 1H), 1.43 (td, *J* = 13.0 Hz, *J* = 3.2 Hz, 1H), 1.35 (dd, *J* = 12.3 Hz, *J* = 2.0 Hz, 1H), 1.29-1.23 (m, 1H), 1.21 (s, 3H), 0.97 (s, 3H), 0.95 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 157.7, 151.5, 129.8, 127.5, 110.7, 110.2, 55.3, 50.3, 41.7, 38.9, 38.0, 33.5, 33.4, 29.6, 24.8, 21.7, 19.4, 19.2.

3. ^{1}H &^{13}C NMR Spectra



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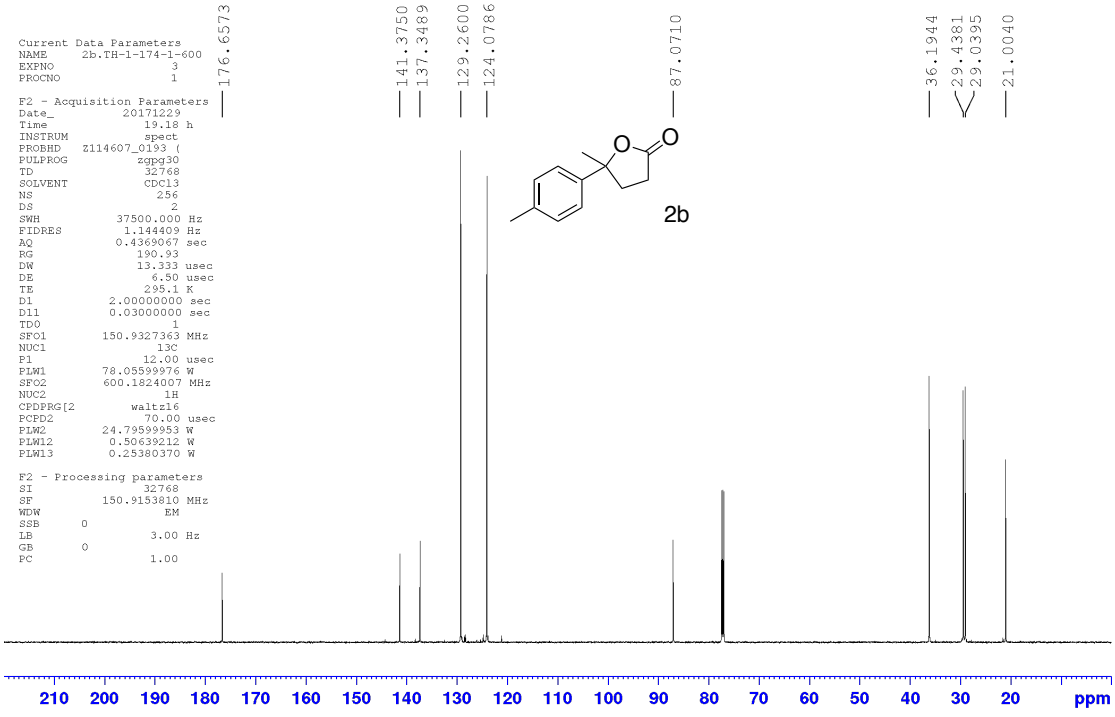
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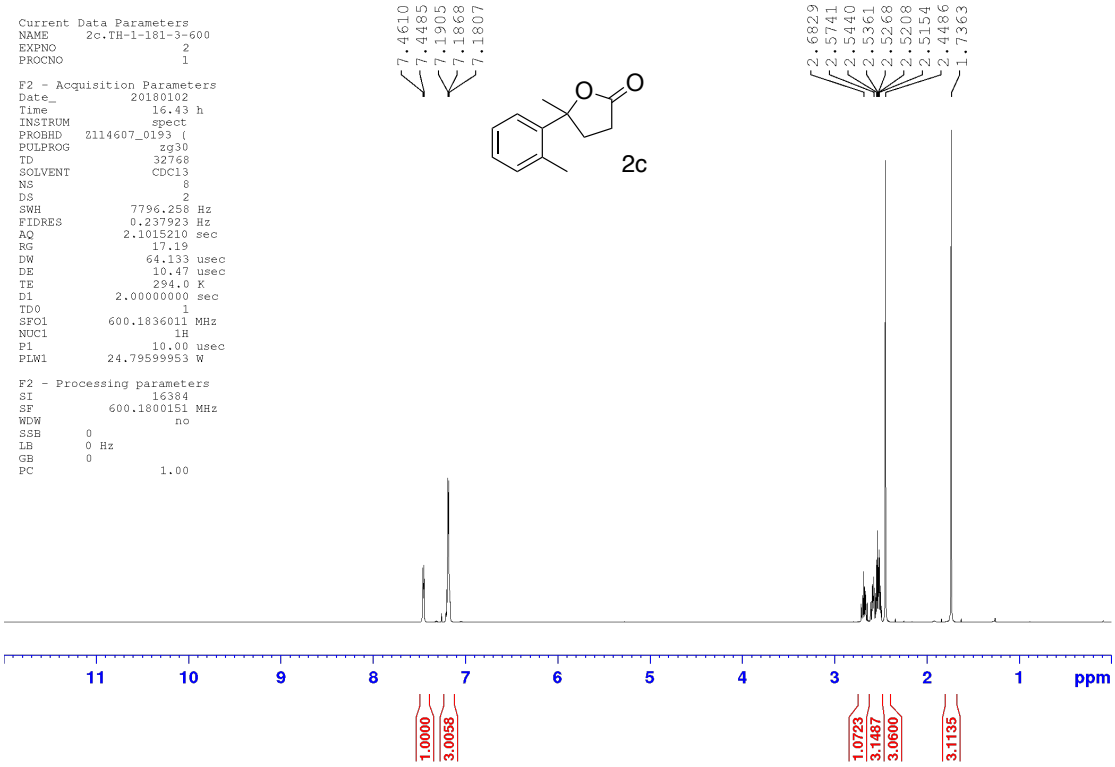
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FIDRES 0.237923 Hz
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TD0 1
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NUC1 1H
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GB 0
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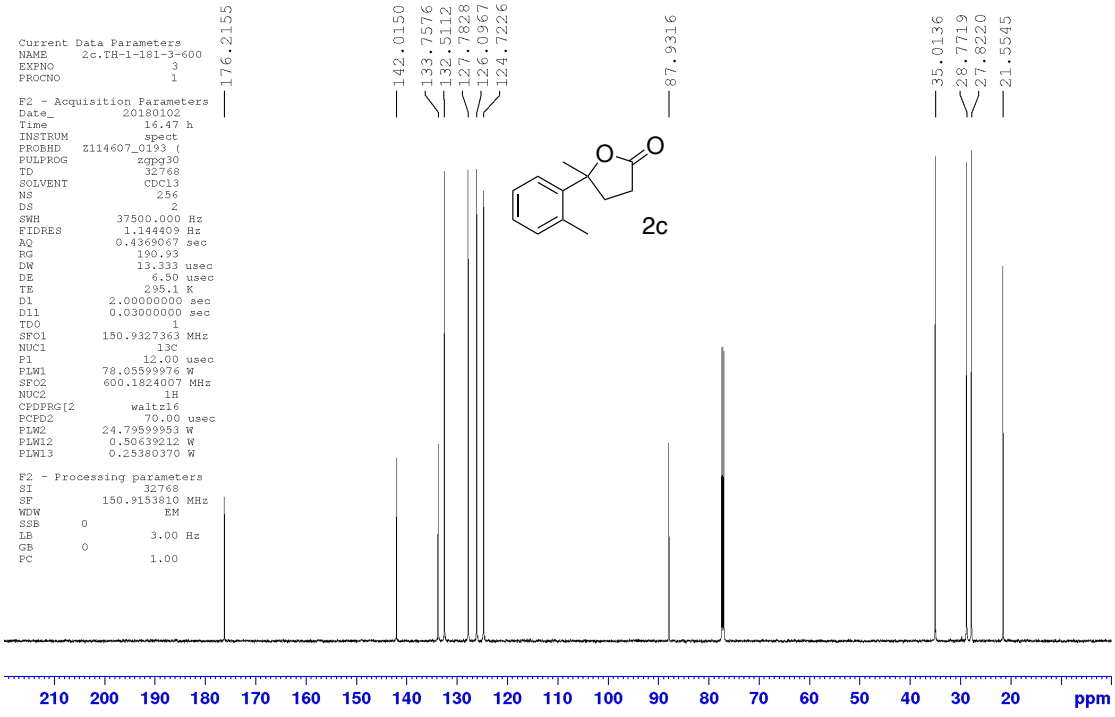
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PROCNO 1
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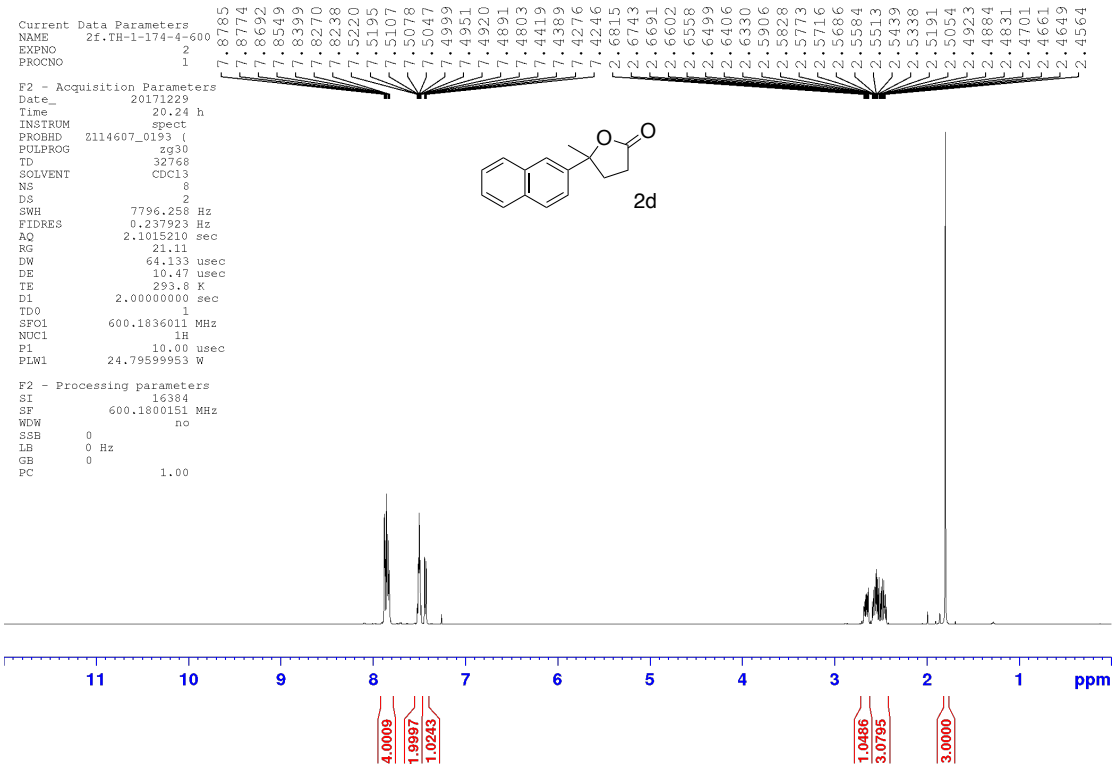
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369067 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9327363 MHz
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO2 600.1824007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
F2 - Processing parameters
SI 32768
SF 150.9153810 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
176.2155
142.0150
133.7576
132.5112
127.7828
126.0967
124.7226
87.9316
35.0136
28.7719
27.8220
21.5545
O
O
2c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Current Data Parameters
NAME 2f.TH-1-174-4-600
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171229
Time 20.24 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015210 sec
RG 21.11
DW 64.133 usec
DE 10.47 usec
TE 293.8 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800151 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
7.8785
7.8774
7.8692
7.8549
7.8399
7.8270
7.8238
7.5220
7.5195
7.5107
7.5078
7.5047
7.4999
7.4951
7.4920
7.4891
7.4803
7.4419
7.4389
7.4276
7.4246
2.6815
2.6743
2.6691
2.6602
2.6558
2.6499
2.6406
2.6330
2.5906
2.5828
2.5773
2.5716
2.5686
2.5584
2.5513
2.5439
2.5338
2.5191
2.5054
2.4923
2.4884
2.4831
2.4701
2.4661
2.4649
2.4564
O
O
2d
11
10
9
8
7
6
5
4
3
2
1
ppm
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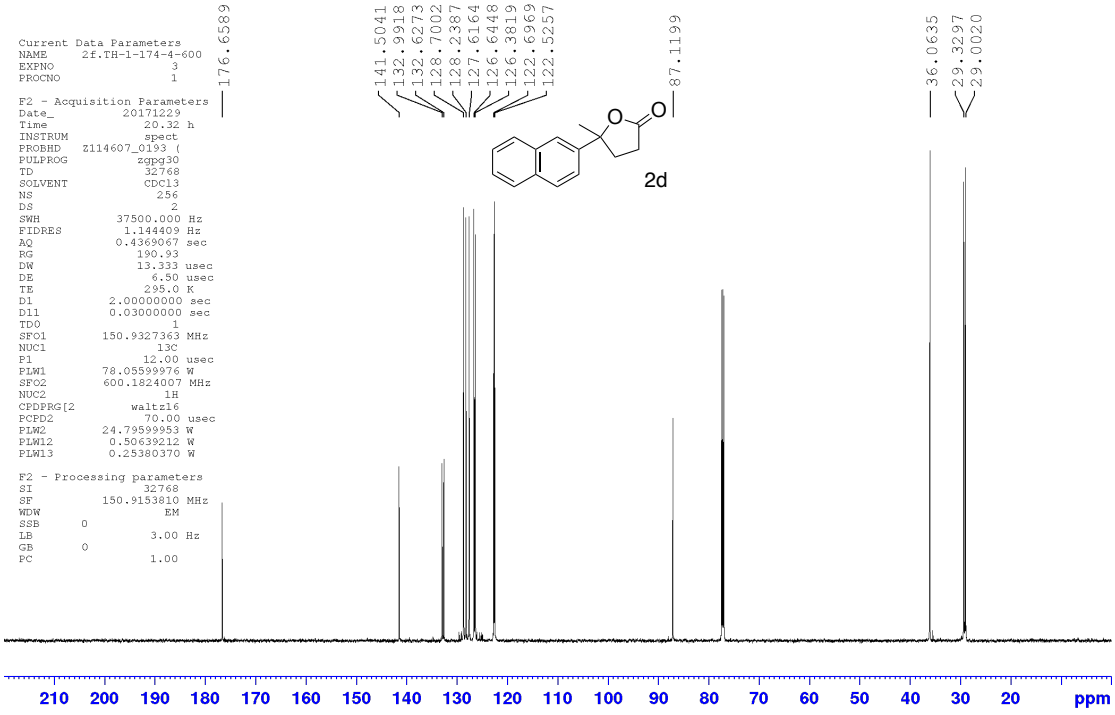
1.9997
1.0243
1.0486
3.0795
3.0000


Current Data Parameters
NAME 2f.TH-1-174-4-600
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171229
Time 20.32 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369067 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9327363 MHz
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO2 600.1824007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
F2 - Processing parameters
SI 32768
SF 150.9153810 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
176.6589
141.5041
132.9918
132.6273
128.7002
128.2387
127.6164
126.6448
126.3819
122.6969
122.5257
87.1199
36.0635
29.3297
29.0020
O
O
2d
210
200
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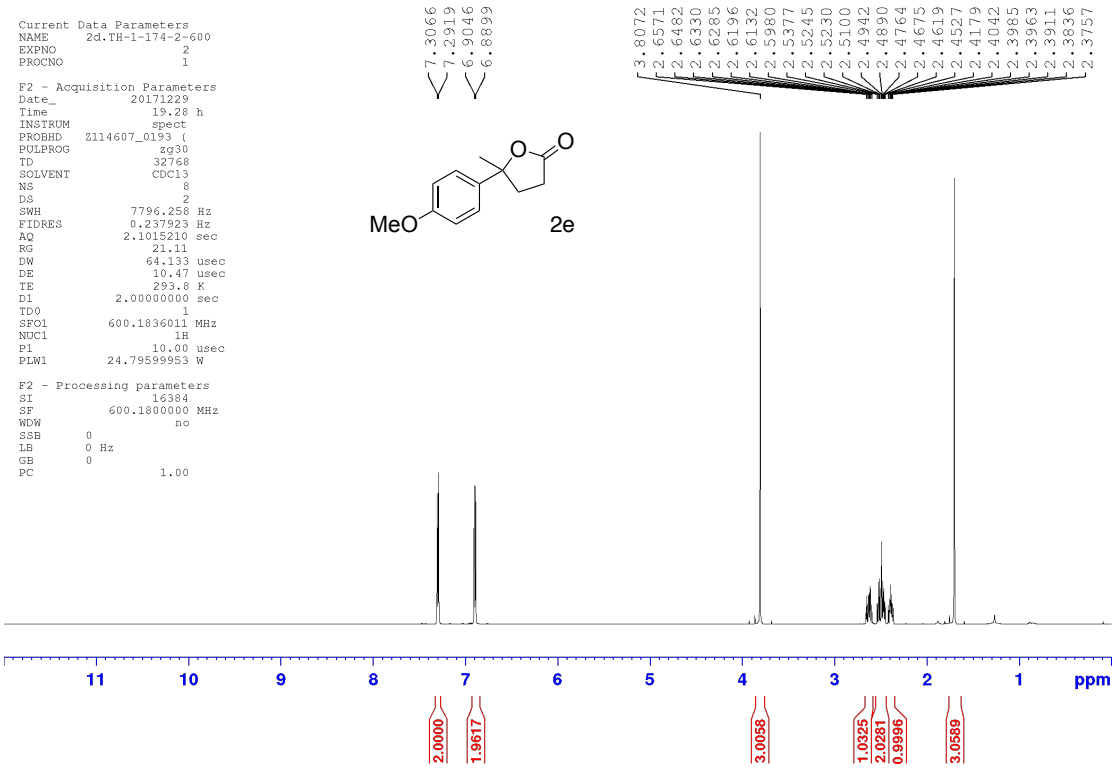
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Current Data Parameters
NAME 2d.TH-1-174-2-600
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171229
Time 19.28 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015210 sec
RG 21.11
DW 64.133 usec
DE 10.47 usec
TE 293.8 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800000 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
7.3066
7.2919
6.9046
6.8899
3.8072
2.6571
2.6482
2.6330
2.6285
2.6196
2.6132
2.5980
2.5377
2.5245
2.5230
2.5100
2.4942
2.4890
2.4764
2.4675
2.4619
2.4527
2.4179
2.4042
2.3985
2.3963
2.3911
2.3836
2.3757
MeO
O
O
2e
11
10
9
8
7
6
5
4
3
2
1
ppm
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3.0058
1.0325
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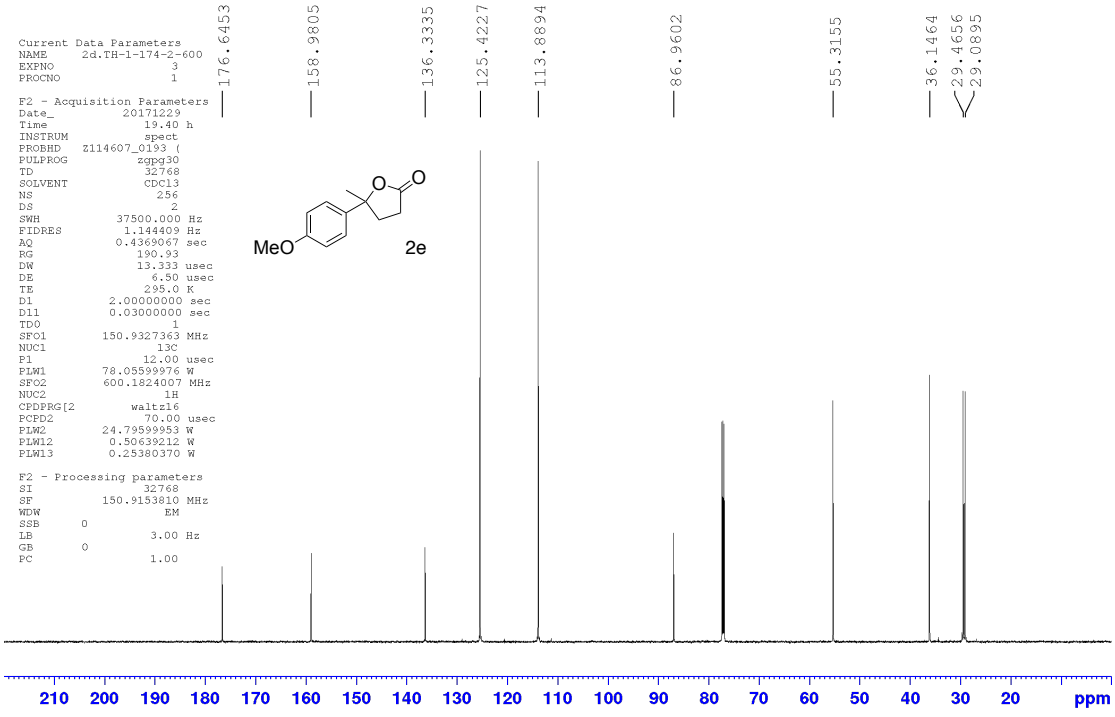
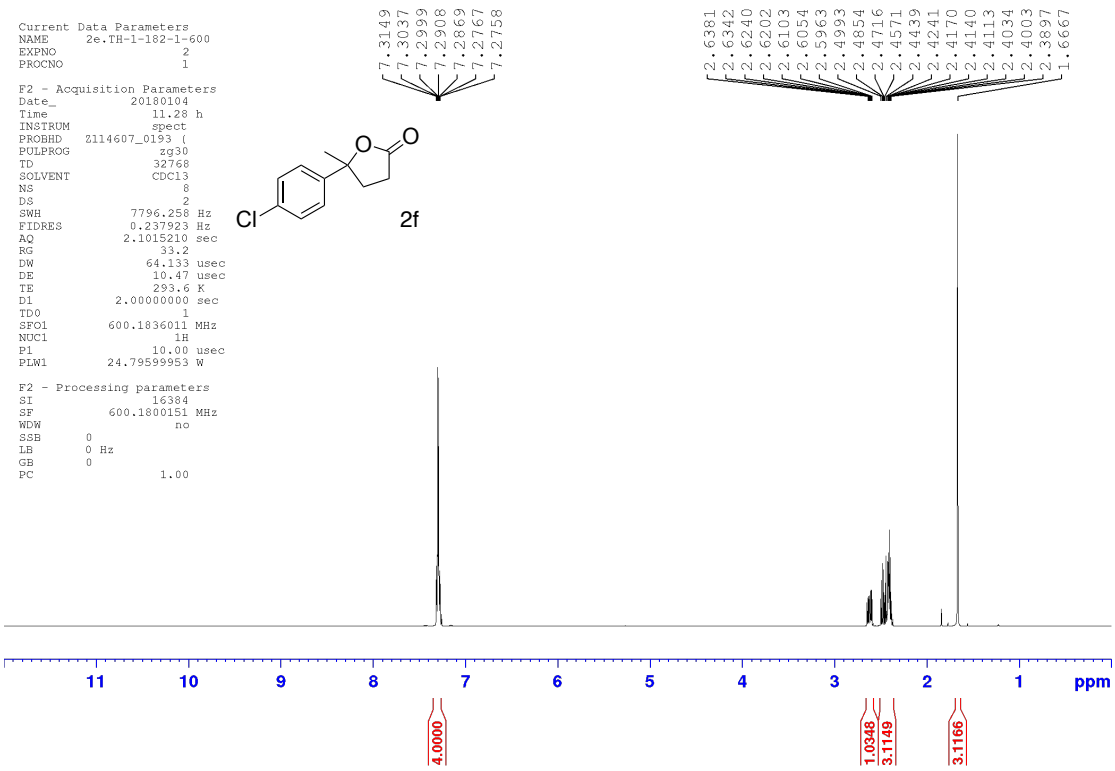
0.9996
3.0589


Current Data Parameters
NAME 2d.TH-1-174-2-600
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171229
Time 19.40 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369067 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9327363 MHz
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO2 600.1824007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
F2 - Processing parameters
SI 32768
SF 150.9153810 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
176.6453
158.9805
136.3335
125.4227
113.8894
86.9602
55.3155
36.1464
29.4656
29.0895
MeO
O
O
2e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Current Data Parameters
NAME 2e.TH-1-182-1-600
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180104
Time 11.28 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015210 sec
RG 33.2
DW 64.133 usec
DE 10.47 usec
TE 293.6 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800151 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
7.3149
7.3037
7.2999
7.2908
7.2869
7.2767
7.2758
2.6381
2.6342
2.6240
2.6202
2.6103
2.6054
2.5963
2.4993
2.4854
2.4716
2.4571
2.4439
2.4241
2.4170
2.4140
2.4113
2.4034
2.4003
2.3897
1.6667
Cl
O
O
2f
11
10
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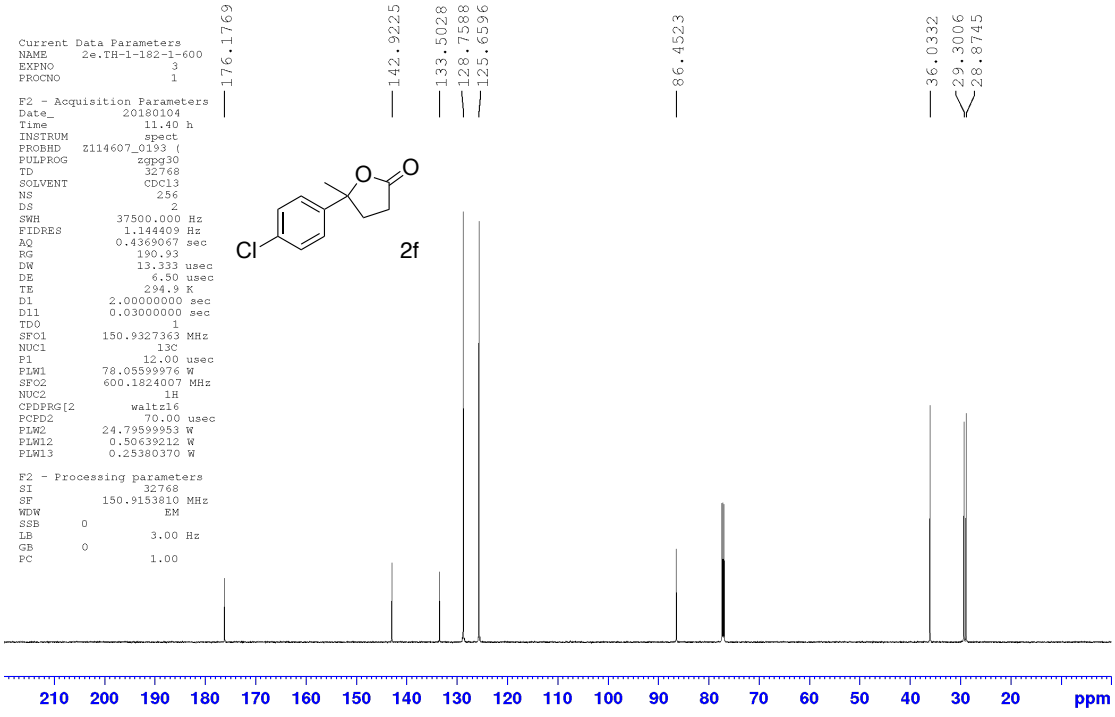
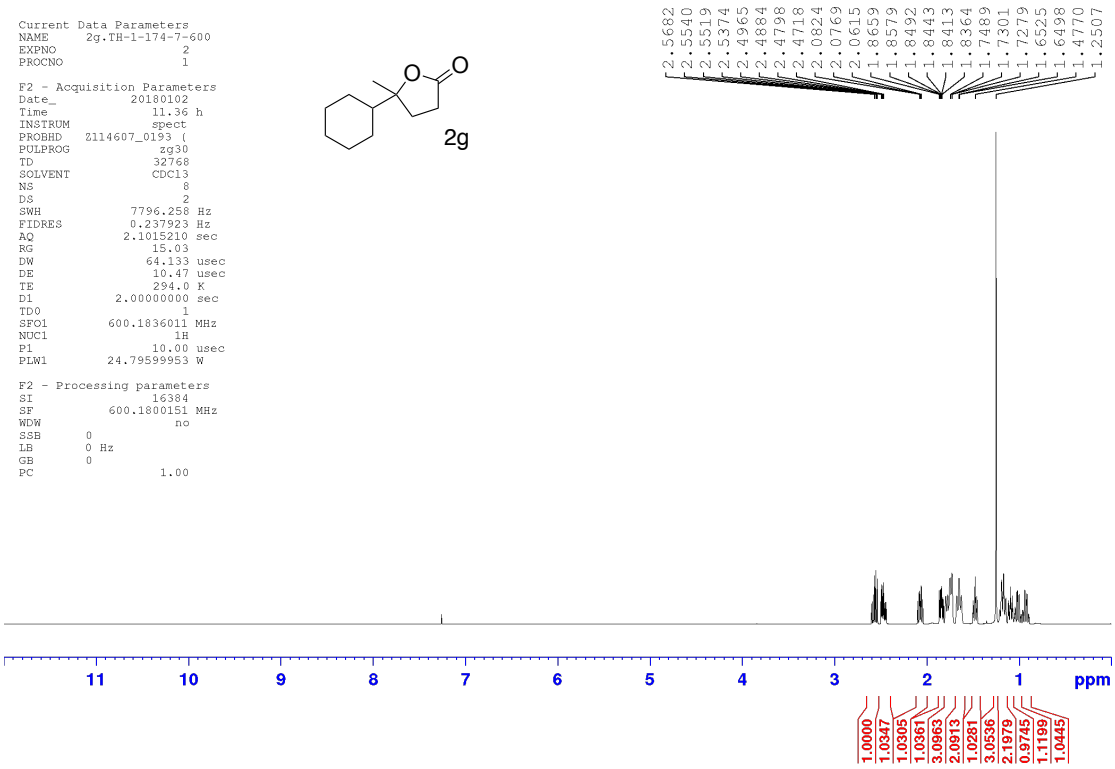
8
7
6
5
4
3
2
1
ppm
4.0000
1.0348
3.1149
3.1166


Current Data Parameters
NAME 2e.TH-1-182-1-600
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180104
Time 11.40 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369067 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 294.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9327363 MHz
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO2 600.1824007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
F2 - Processing parameters
SI 32768
SF 150.9153810 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
176.1769
142.9225
133.5028
128.7588
125.6596
86.4523
36.0332
29.3006
28.8745
Cl
O
O
2f
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Current Data Parameters
NAME 2g.TH-1-174-7-600
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180102
Time 11.36 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015210 sec
RG 15.03
DW 64.133 usec
DE 10.47 usec
TE 294.0 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800151 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
O
O
2g
2.5682
2.5540
2.5519
2.5374
2.4965
2.4884
2.4798
2.4718
2.0824
2.0769
2.0615
1.8659
1.8579
1.8492
1.8443
1.8413
1.8364
1.7489
1.7301
1.7279
1.6525
1.6498
1.4770
1.2507
11
10
9
8
7
6
5
4
3
2
1
ppm
1.0000
1.0347
1.0305
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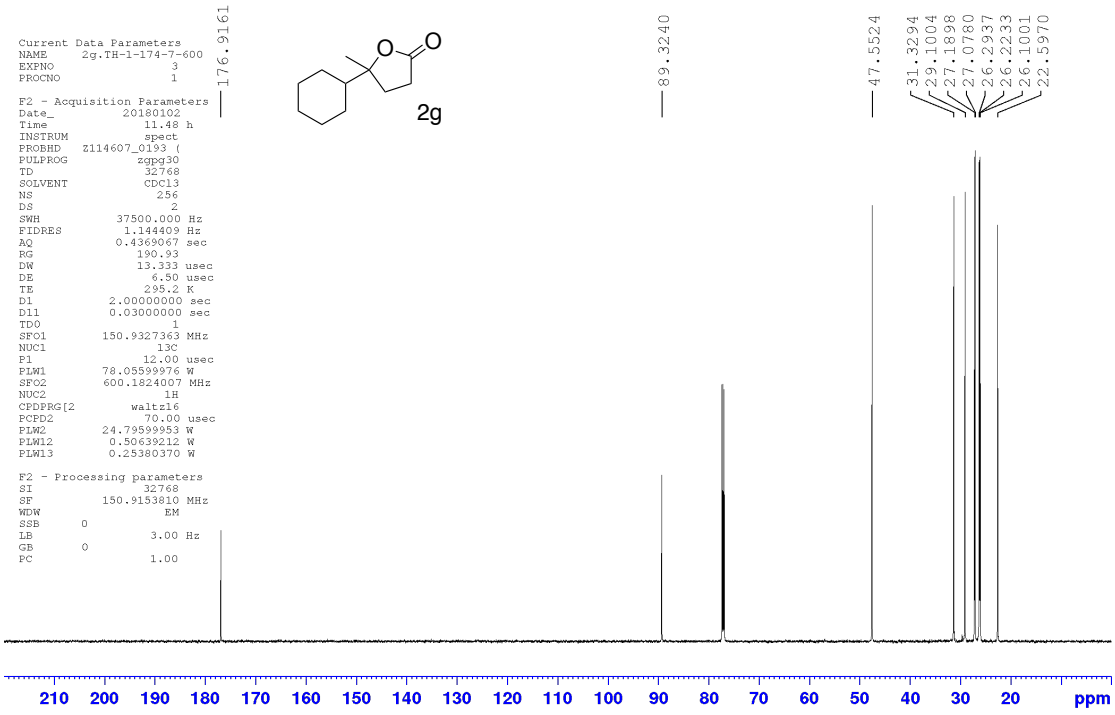
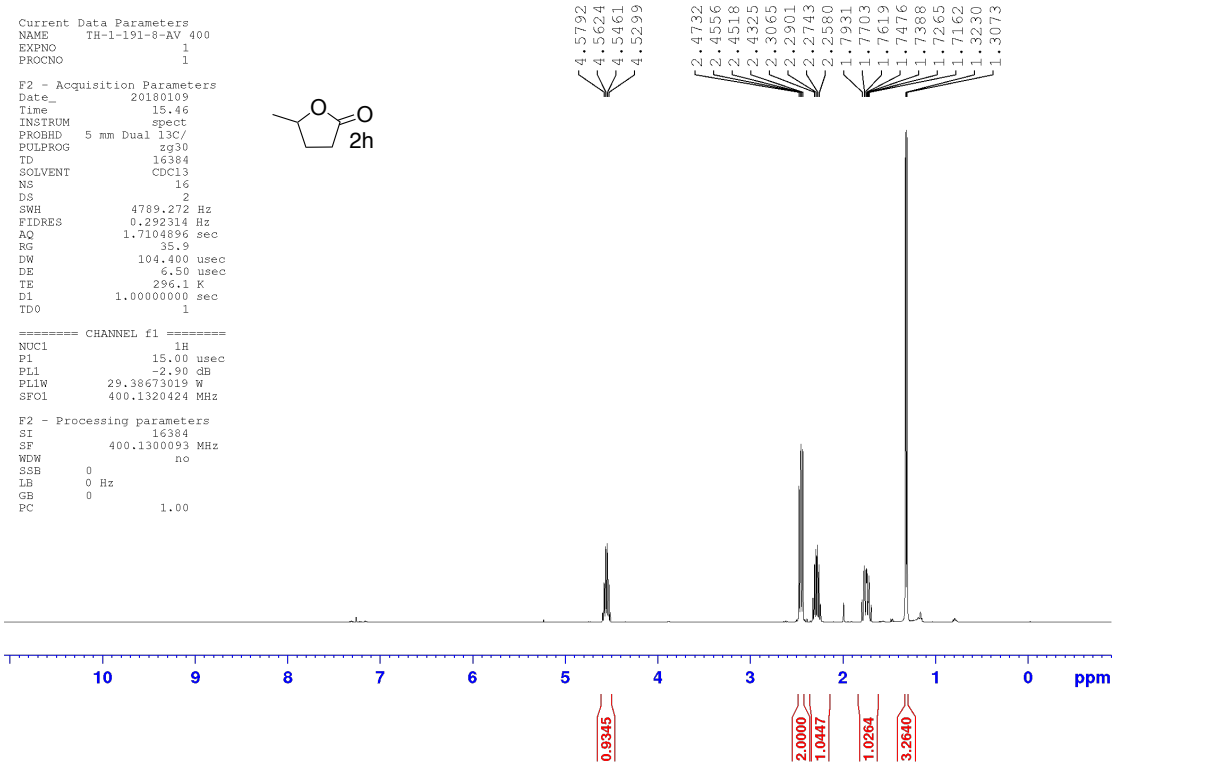
3.0963
2.0913
1.0281
3.0536
2.1979
0.9745
1.1199
1.0445


Current Data Parameters
NAME 2g.TH-1-174-7-600
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180102
Time 11.48 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369067 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9327363 MHz
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO2 600.1824007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
F2 - Processing parameters
SI 32768
SF 150.9153810 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
176.9161
O
O
2g
89.3240
47.5524
31.3294
29.1004
27.1898
27.0780
26.2937
26.2233
26.1001
22.5970
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Current Data Parameters
NAME TH-1-191-8-AV 400
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180109
Time 15.46
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7104896 sec
RG 35.9
DW 104.400 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.90 dB
PL1W 29.38673019 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300093 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
2h
4.5792
4.5624
4.5461
4.5299
2.4732
2.4556
2.4518
2.4325
2.3065
2.2901
2.2743
2.2580
1.7931
1.7703
1.7619
1.7476
1.7388
1.7265
1.7162
1.3230
1.3073
10
9
8
7
6
5
4
3
2
1
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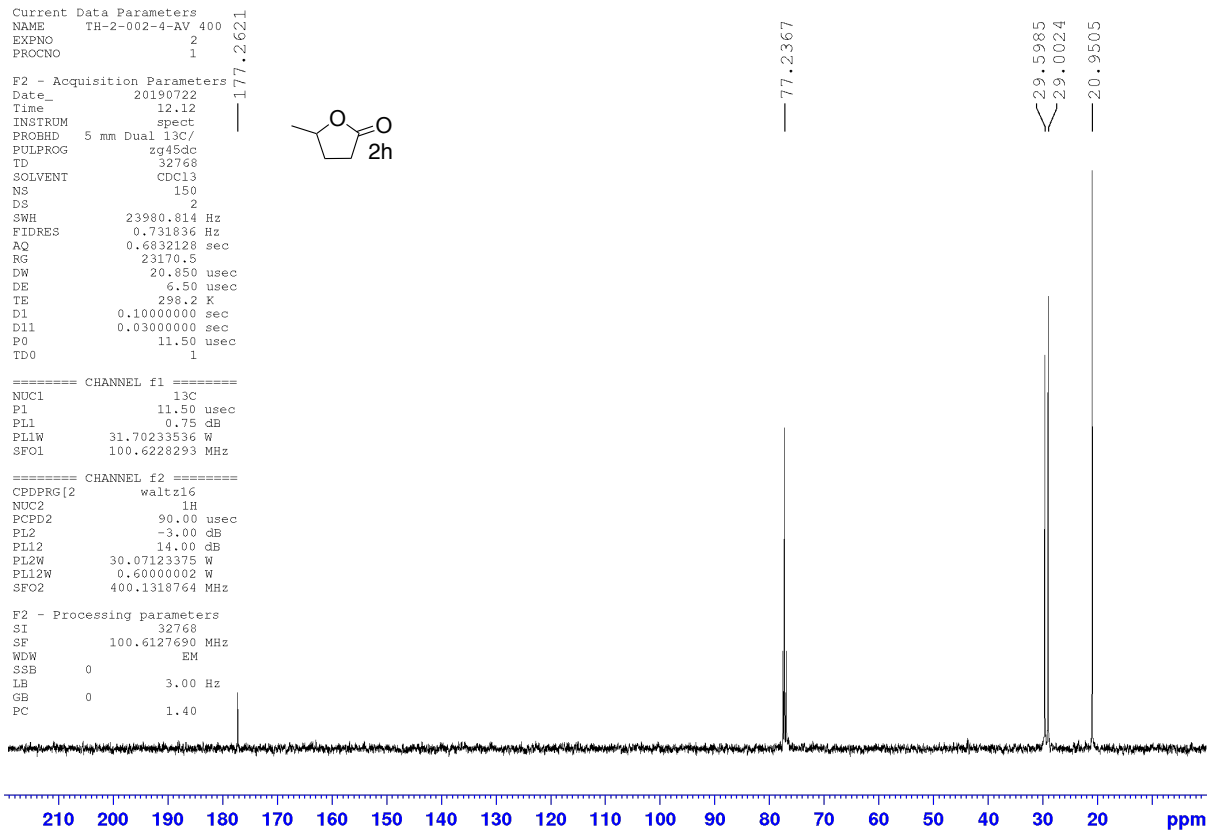
2.0000
1.0447
1.0264
3.2640


Current Data Parameters
NAME TH-2-002-4-AV 400
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190722
Time 12.12
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg45dc
TD 32768
SOLVENT CDCl3
NS 150
DS 2
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832128 sec
RG 23170.5
DW 20.850 usec
DE 6.50 usec
TE 298.2 K
D1 0.10000000 sec
D11 0.03000000 sec
P0 11.50 usec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.50 usec
PL1 0.75 dB
PL1W 31.70233536 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL2W 30.07123375 W
PL12W 0.60000002 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
2h
177.2621
77.2367
29.5985
29.0024
20.9505
210
200
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180
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160
150
140
130
120
110
100
90
80
70
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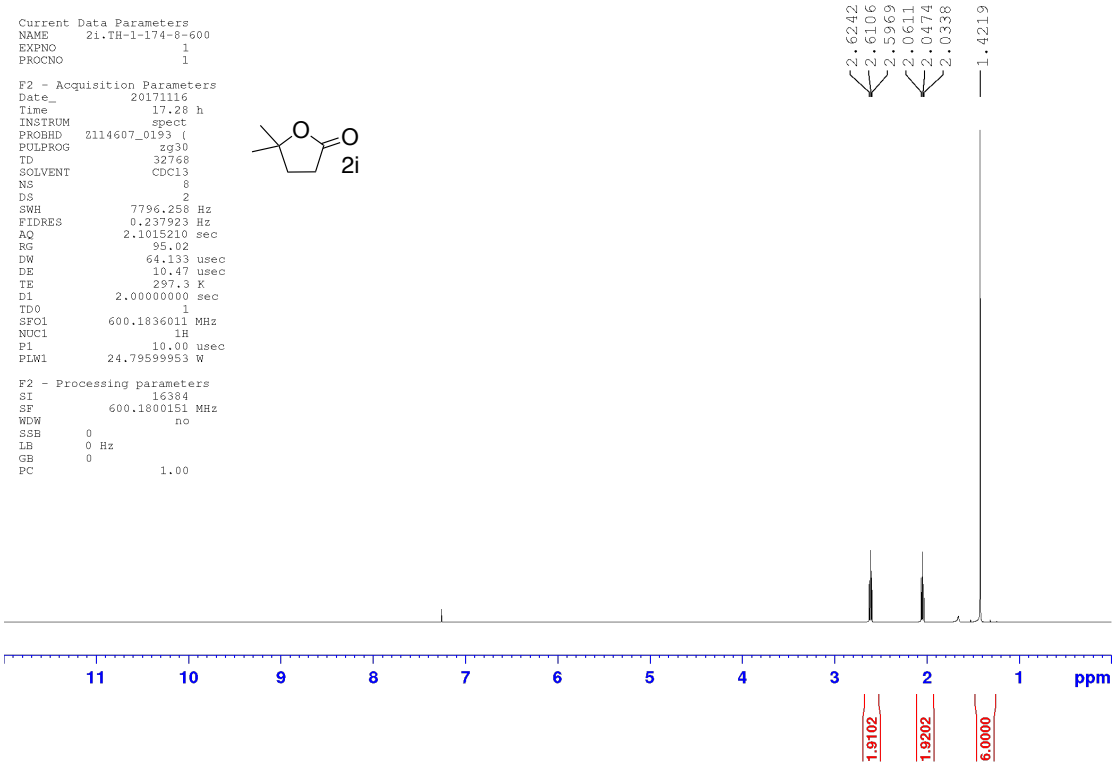
50
40
30
20
ppm


Current Data Parameters
NAME 2i.TH-1-174-8-600
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171116
Time 17.28 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015210 sec
RG 95.02
DW 64.133 usec
DE 10.47 usec
TE 297.3 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800151 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
O
O
2i
2.6242
2.6106
2.5969
2.0611
2.0474
2.0338
1.4219
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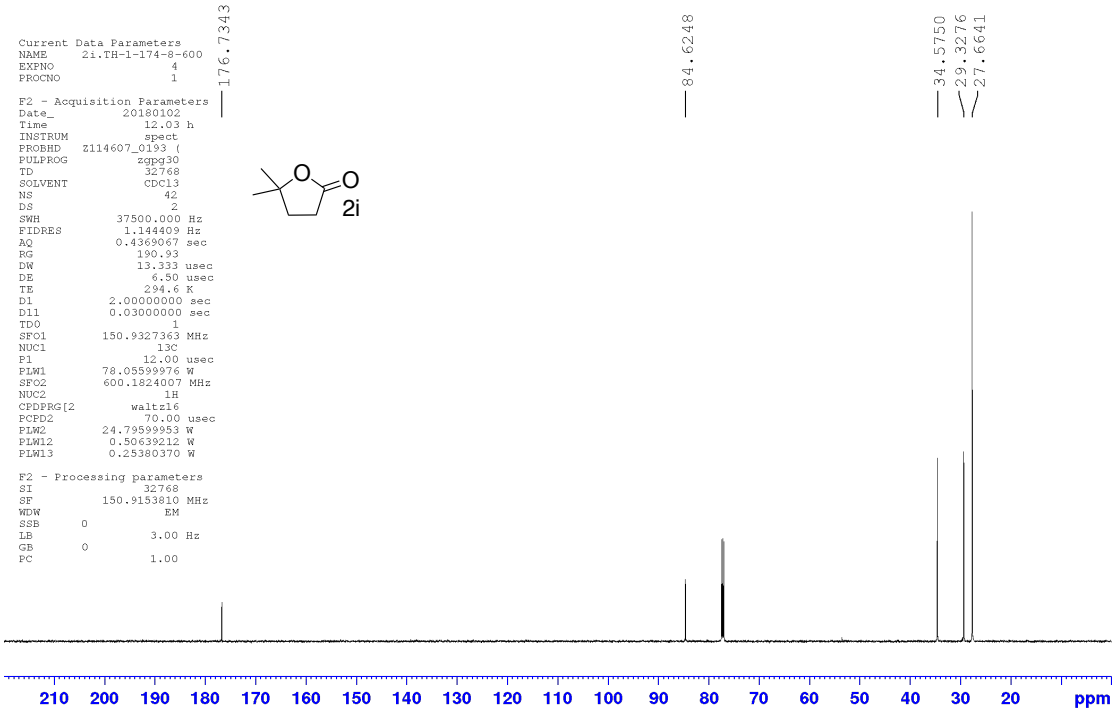
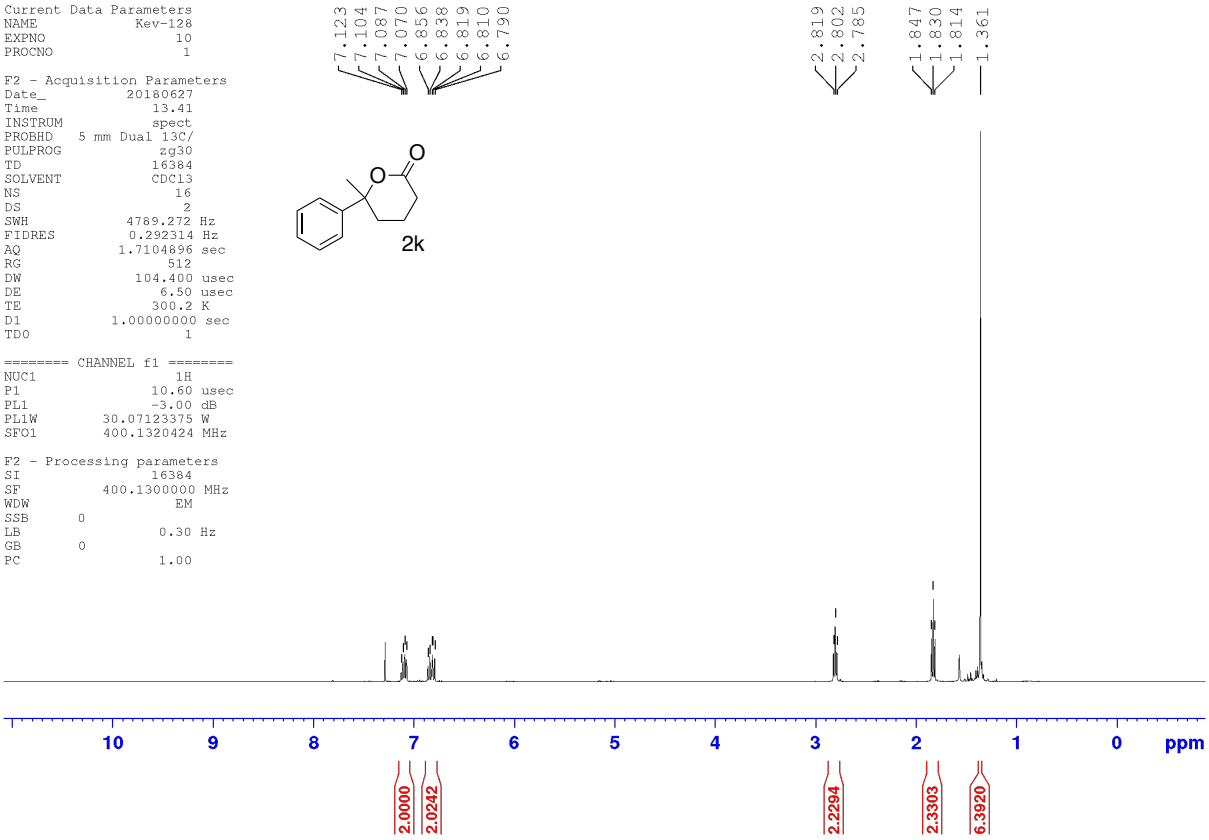
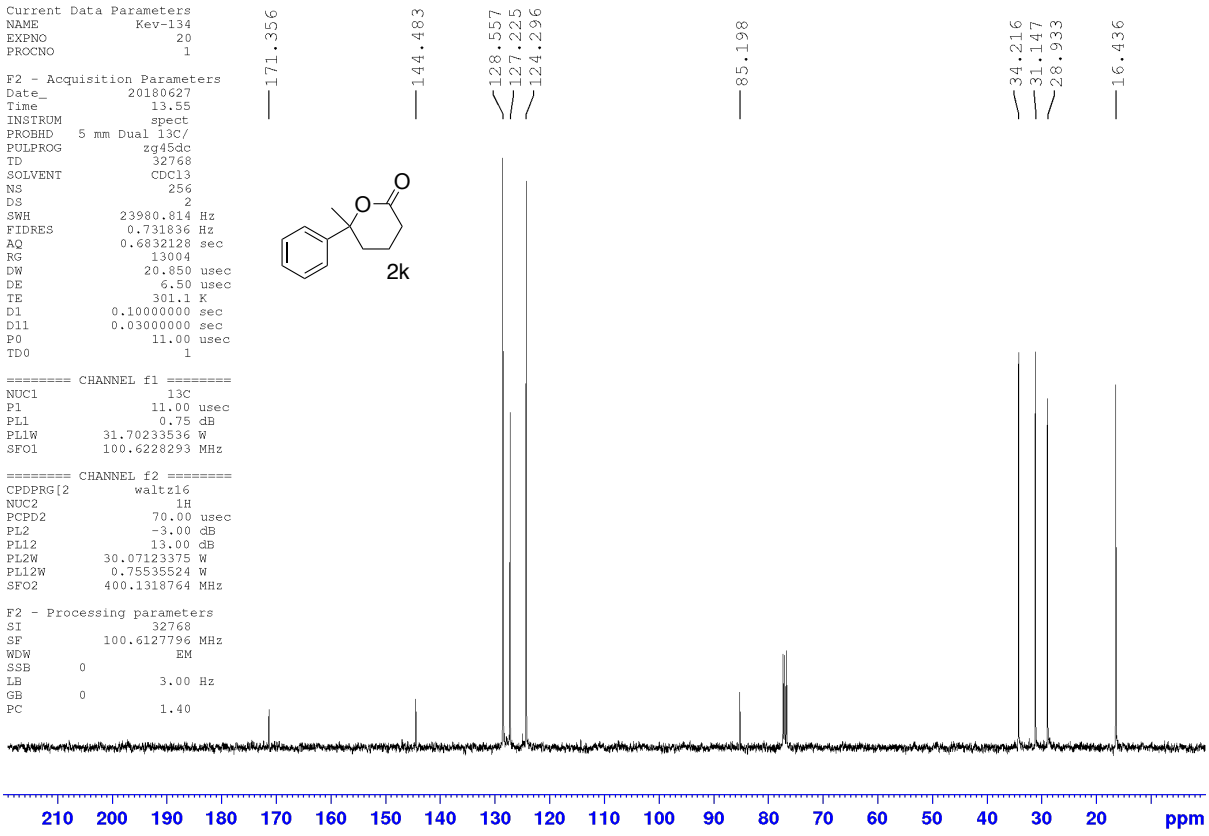
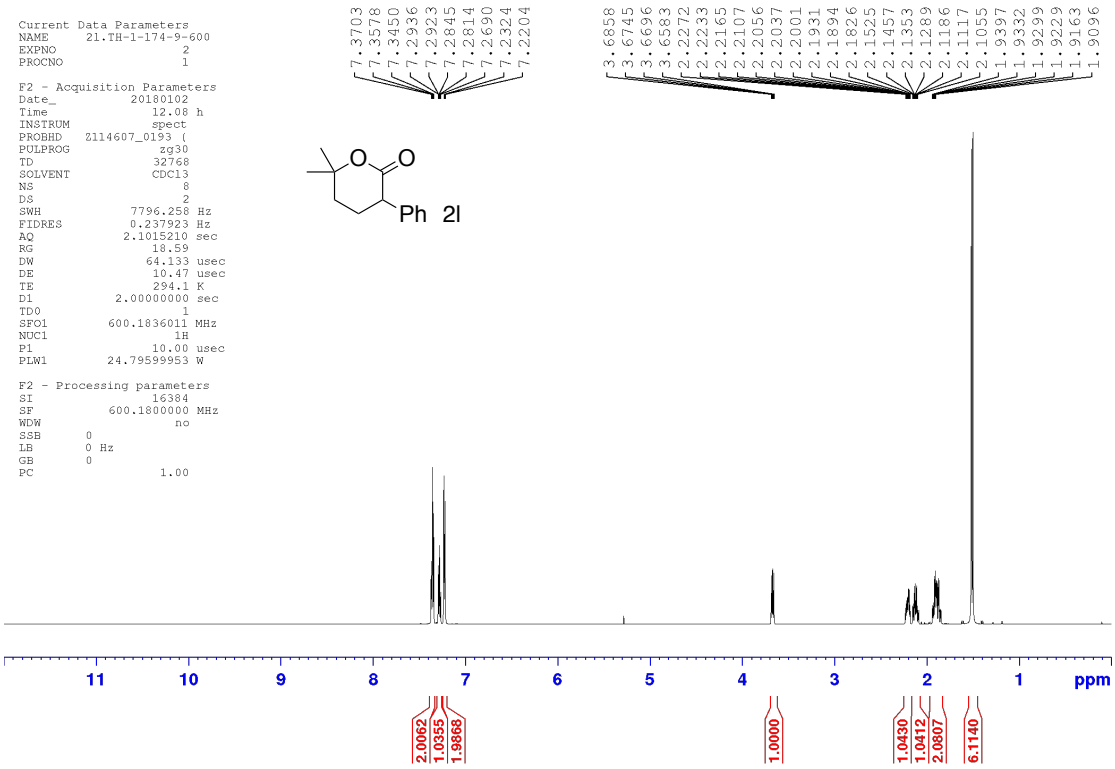
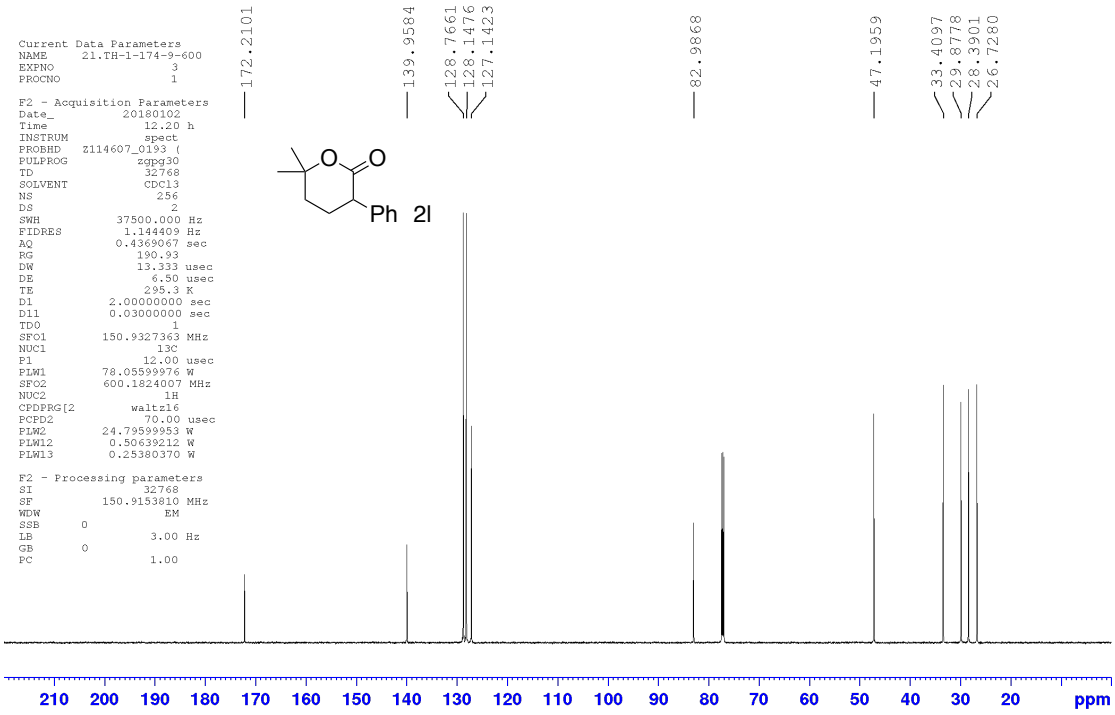
9
8
7
6
5
4
3
2
1
ppm
1.9102
1.9202
6.0000

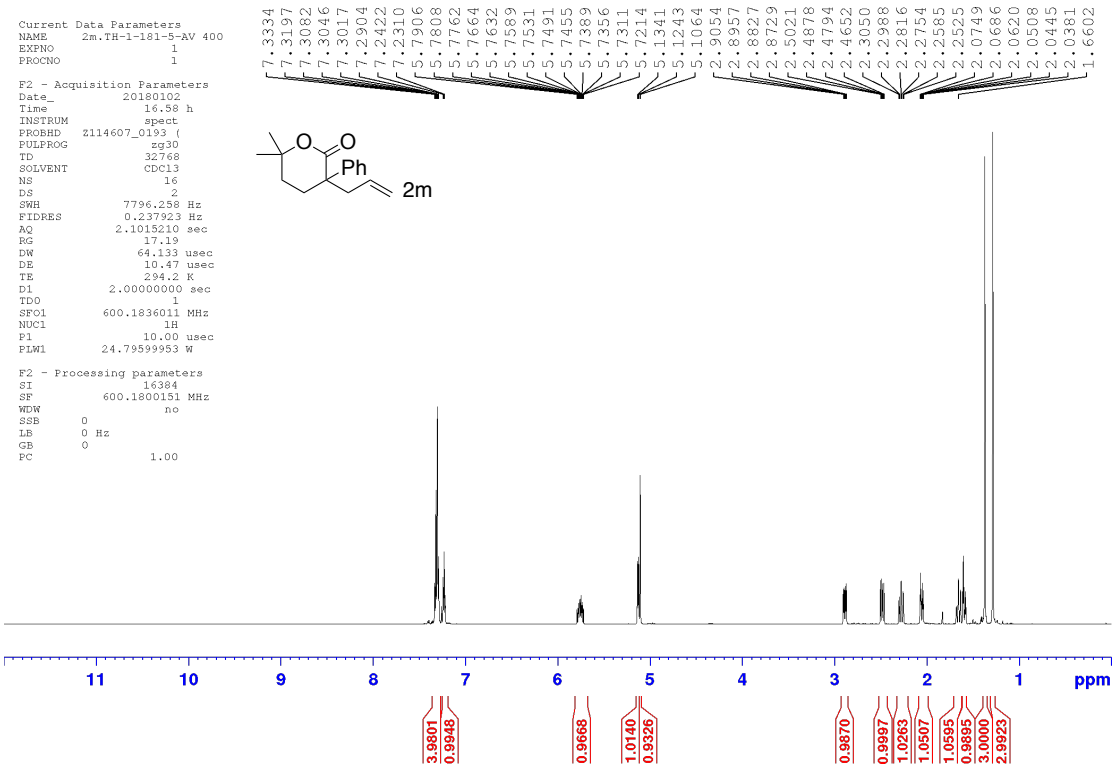

Current Data Parameters
NAME 2i.TH-1-174-8-600
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180102
Time 12.03 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 42
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369067 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 294.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9327363 MHz
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO2 600.1824007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
F2 - Processing parameters
SI 32768
SF 150.9153810 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
176.7343
84.6248
34.5750
29.3276
27.6641
O
O
2i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

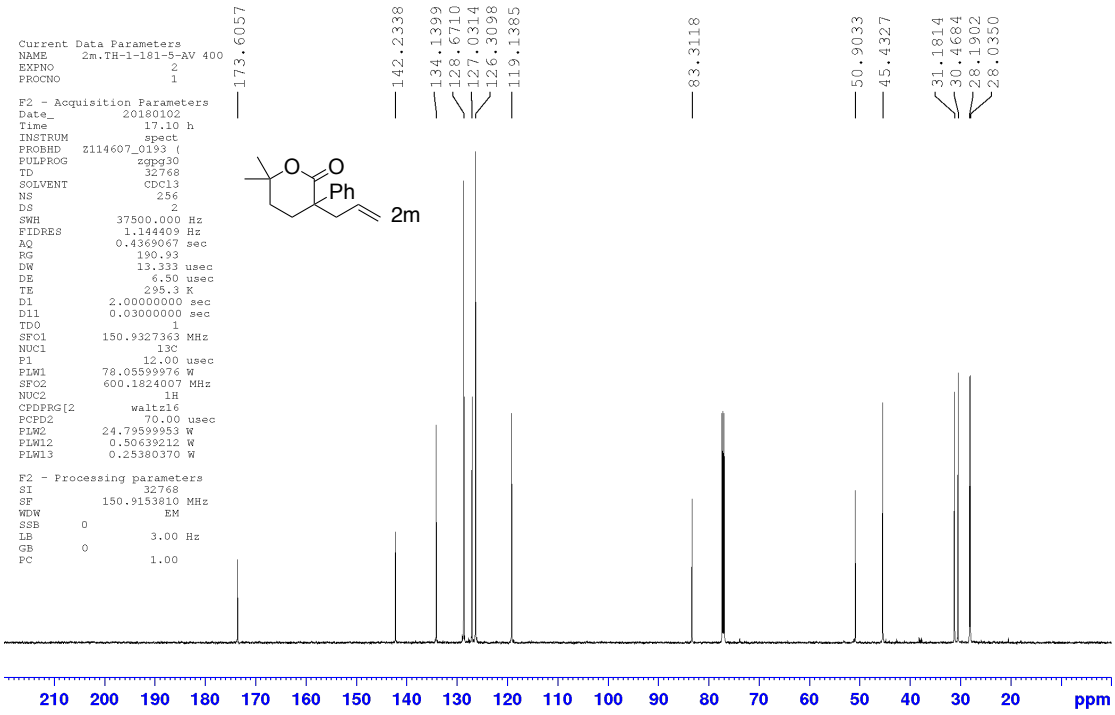

Current Data Parameters
NAME Kev-128
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180627
Time 13.41
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7104896 sec
RG 512
DW 104.400 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.60 usec
PL1 -3.00 dB
PL1W 30.07123375 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.123
7.104
7.087
7.070
6.856
6.838
6.819
6.810
6.790
2.819
2.802
2.785
1.847
1.830
1.814
1.361
2k
10
9
8
7
6
5
4
3
2
1
0
ppm
2.0000
2.0242
2.2294
2.3303
6.3920

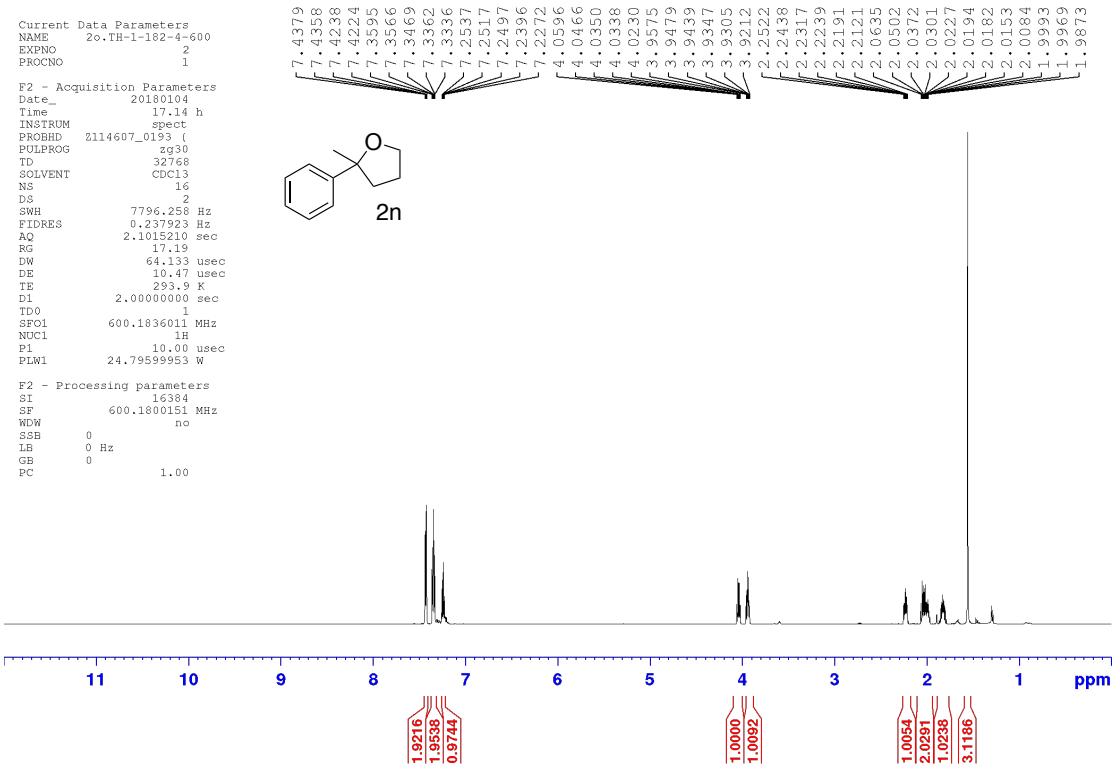

Current Data Parameters
NAME Kev-134
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180627
Time 13.55
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg45dc
TD 32768
SOLVENT CDCl3
NS 256
DS 2
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832128 sec
RG 13004
DW 20.850 usec
DE 6.50 usec
TE 301.1 K
D1 0.10000000 sec
D11 0.03000000 sec
P0 11.00 usec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0.75 dB
PL1W 31.70233536 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 13.00 dB
PL2W 30.07123375 W
PL12W 0.75535524 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127796 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
171.356
144.483
128.557
127.225
124.296
85.198
34.216
31.147
28.933
16.436
2k
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Current Data Parameters
NAME 21.TH-1-174-9-600
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180102
Time 12.08 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015210 sec
RG 18.59
DW 64.133 usec
DE 10.47 usec
TE 294.1 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800000 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
7.3703
7.3578
7.3450
7.2936
7.2923
7.2845
7.2814
7.2690
7.2324
7.2204
3.6858
3.6745
3.6696
3.6583
2.2272
2.2233
2.2165
2.2107
2.2056
2.2037
2.2001
2.1931
2.1894
2.1826
2.1525
2.1457
2.1353
2.1289
2.1186
2.1117
2.1055
1.9337
1.9332
1.9299
1.9229
1.9163
1.9096
O
O
Ph 2l
11
10
9
8
7
6
5
4
3
2
1
ppm
2.0062
1.0355
1.9868
1.0000
1.0430
1.0412
2.0807
6.1140


Current Data Parameters
NAME 21.TH-1-174-9-600
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180102
Time 12.20 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369067 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9327363 MHz
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO2 600.1824007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
F2 - Processing parameters
SI 32768
SF 150.9153810 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
172.2101
139.9584
128.7661
128.1476
127.1423
82.9868
47.1959
33.4097
29.8778
28.3901
26.7280
O
O
Ph 2l
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Current Data Parameters
NAME 2m.TH-1-181-5-AV 400
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180102
Time 16.58 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015210 sec
RG 17.19
DW 64.133 usec
DE 10.47 usec
TE 294.2 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800151 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
7.3334
7.3197
7.3082
7.3046
7.3017
7.2904
7.2422
7.2310
5.7906
5.7808
5.7762
5.7664
5.7632
5.7589
5.7531
5.7491
5.7455
5.7389
5.7356
5.7311
5.7214
5.1341
5.1243
5.1064
2.9054
2.8957
2.8827
2.8729
2.5021
2.4878
2.4794
2.4652
2.3050
2.2988
2.2816
2.2754
2.2585
2.2525
2.0749
2.0686
2.0620
2.0508
2.0445
2.0381
1.6602
O
O
Ph
2m
11
10
9
8
7
6
5
4
3
2
1
ppm
3.9801
0.9948
0.9668
1.0140
0.9326
0.9870
0.9997
1.0263
1.0507
1.0595
0.9895
3.0000
2.9923


Current Data Parameters
NAME 2m.TH-1-181-5-AV 400
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180102
Time 17.10 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 256
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369067 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9327363 MHz
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO2 600.1824007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
F2 - Processing parameters
SI 32768
SF 150.9153810 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
173.6057
142.2338
134.1399
128.6710
127.0314
126.3098
119.1385
83.3118
50.9033
45.4327
31.1814
30.4684
28.1902
26.0350
O
O
Ph
2m
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Current Data Parameters
NAME 2o.TH-1-182-4-600
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180104
Time 17.14 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015210 sec
RG 17.19
DW 64.133 usec
DE 10.47 usec
TE 293.9 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800151 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
7.4379
7.4358
7.4238
7.4224
7.3595
7.3566
7.3469
7.3362
7.3336
7.2537
7.2517
7.2497
7.2396
7.2272
4.0596
4.0466
4.0350
4.0338
4.0230
3.9575
3.9479
3.9439
3.9347
3.9305
3.9212
2.2522
2.2438
2.2317
2.2239
2.2191
2.2121
2.0635
2.0502
2.0372
2.0301
2.0227
2.0194
2.0182
2.0153
2.0084
1.9993
1.9969
1.9873
O
2n
11
10
9
8
7
6
5
4
3
2
1
ppm
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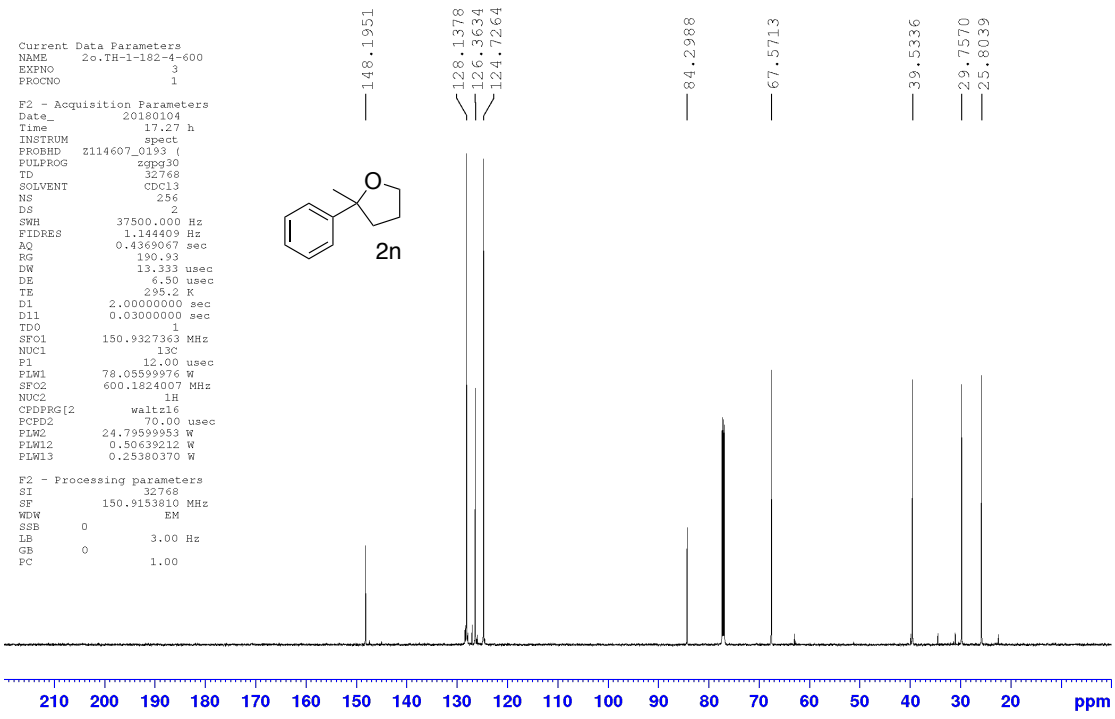
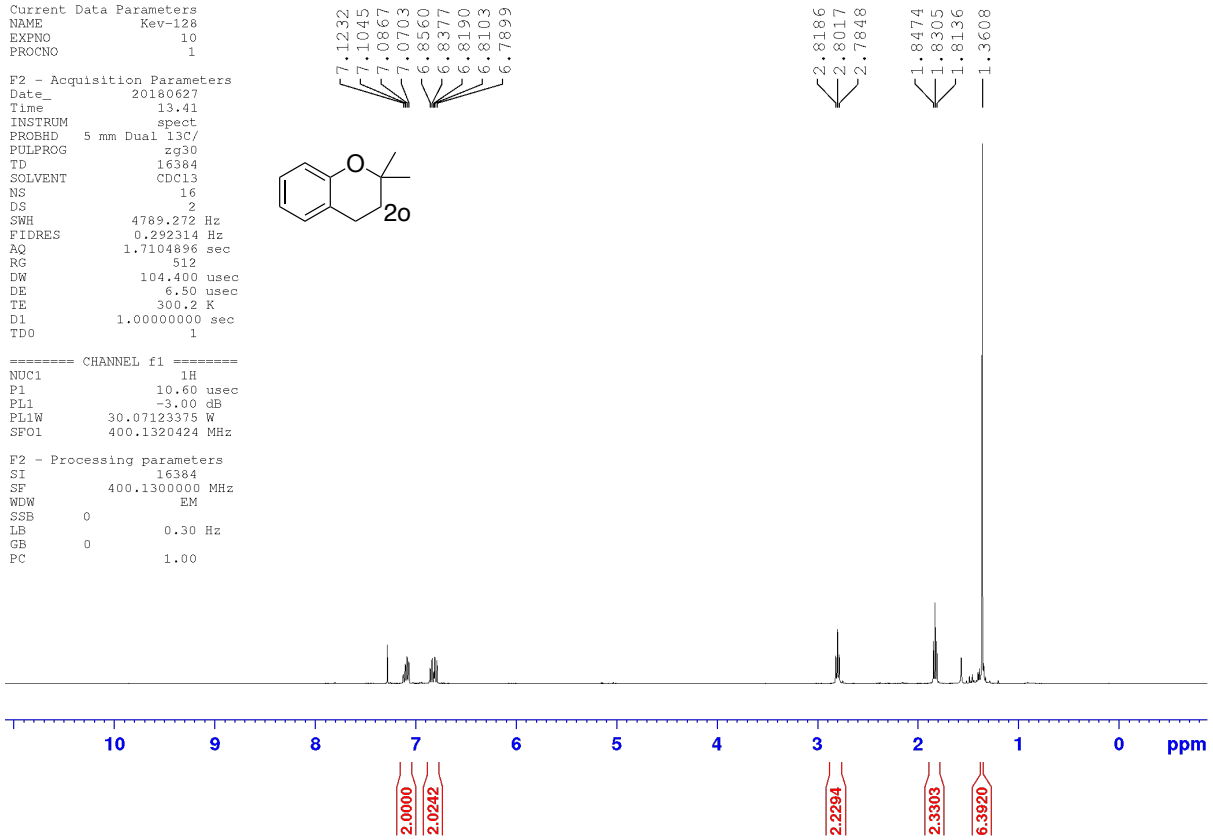
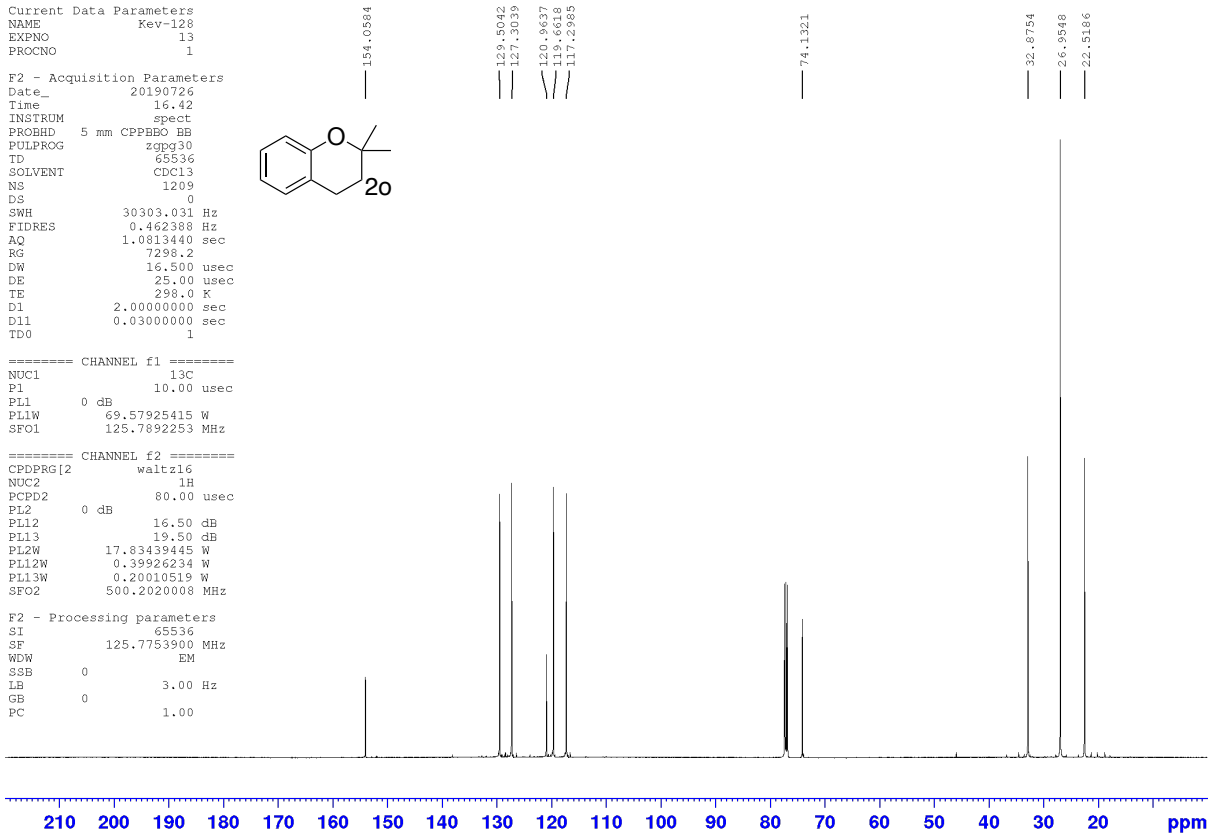
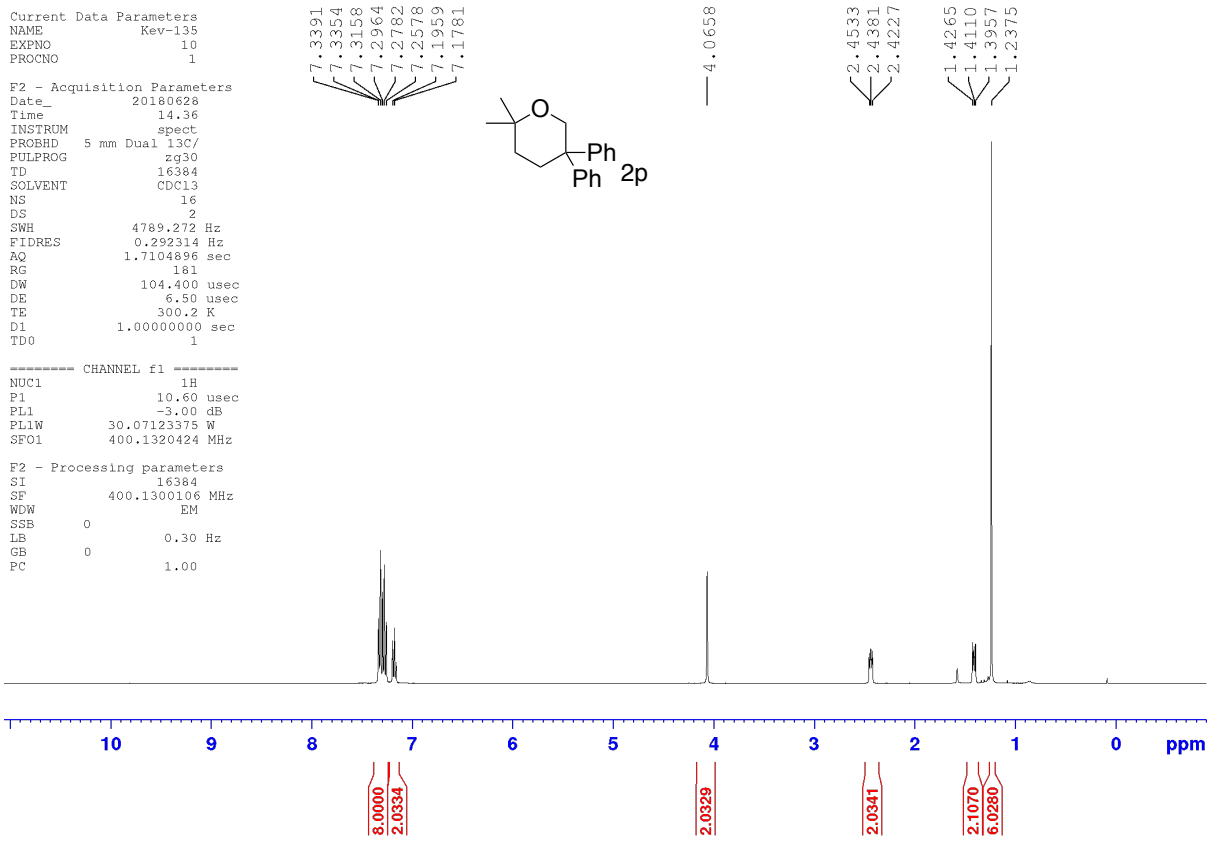
1.9538
0.9744
1.0000
1.0092
1.0054
2.0291
1.0238
3.1186


Current Data Parameters
NAME 2o.TH-1-182-4-600
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180104
Time 17.27 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 256
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369067 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9327363 MHz
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO2 600.1824007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
F2 - Processing parameters
SI 32768
SF 150.9153810 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
148.1951
128.1378
126.3634
124.7264
84.2988
67.5713
39.5336
29.7570
25.8039
O
2n
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Current Data Parameters
NAME Kev-128
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180627
Time 13.41
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7104896 sec
RG 512
DW 104.400 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.60 usec
PL1 -3.00 dB
PL1W 30.07123375 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.1232
7.1045
7.0867
7.0703
6.8560
6.8377
6.8190
6.8103
6.7899
2.8186
2.8017
2.7848
1.8474
1.8305
1.8136
1.3608
2o
10
9
8
7
6
5
4
3
2
1
0
ppm
2.0000
2.0242
2.2294
2.3303
6.3920


Current Data Parameters
NAME Kev-128
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190726
Time 16.42
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1209
DS 0
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813440 sec
RG 7298.2
DW 16.500 usec
DE 25.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 0 dB
PL1W 69.57925415 W
SFO1 125.7892253 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0 dB
PL12 16.50 dB
PL13 19.50 dB
PL2W 17.83439445 W
PL12W 0.39926234 W
PL13W 0.20010519 W
SFO2 500.2020008 MHz
F2 - Processing parameters
SI 65536
SF 125.7753900 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
154.0584
129.5042
127.3039
120.9637
119.6618
117.2985
74.1321
32.8754
26.9548
22.5186
2o
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Current Data Parameters
NAME Kev-135
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180628
Time 14.36
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7104896 sec
RG 181
DW 104.400 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.60 usec
PL1 -3.00 dB
PL1W 30.07123375 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.3391
7.3354
7.3158
7.2964
7.2782
7.2578
7.1959
7.1781
4.0658
2.4533
2.4381
2.4227
1.4265
1.4110
1.3957
1.2375
O
Ph
Ph
2p
10
9
8
7
6
5
4
3
2
1
0
ppm
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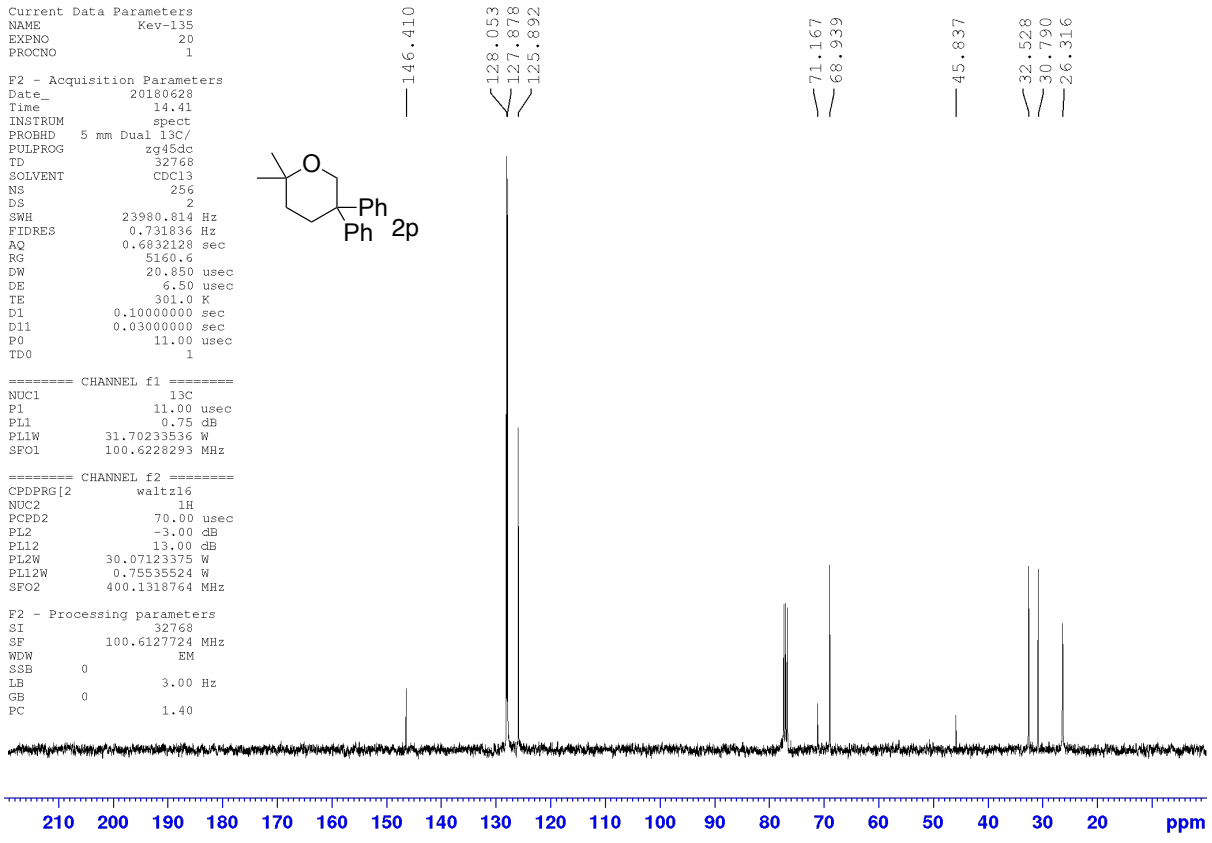
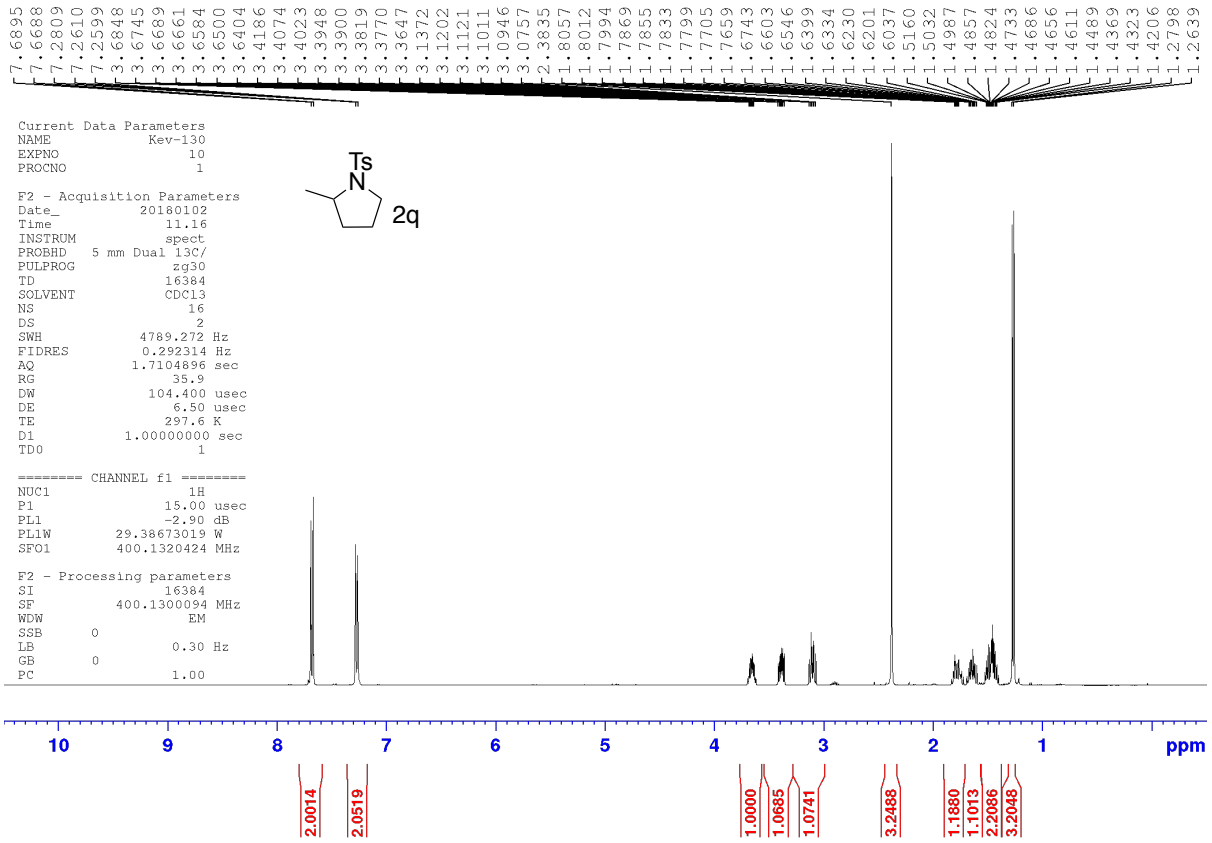
2.0329
2.0341
2.1070
6.0280


Current Data Parameters
NAME Kev-135
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180628
Time 14.41
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg45dc
TD 32768
SOLVENT CDCl3
NS 256
DS 2
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832128 sec
RG 5160.6
DW 20.850 usec
DE 6.50 usec
TE 301.0 K
D1 0.10000000 sec
D11 0.03000000 sec
P0 11.00 usec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0.75 dB
PL1W 31.70233536 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 13.00 dB
PL2W 30.07123375 W
PL12W 0.75535524 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127724 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
146.410
128.053
127.878
125.892
71.167
68.939
45.837
32.528
30.790
26.316
O
Ph
Ph
2p
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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3.6661
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3.6404
3.4186
3.4074
3.4023
3.3948
3.3900
3.3819
3.3770
3.3647
3.1372
3.1202
3.1121
3.1011
3.0946
3.0757
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1.7855
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1.7705
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1.6334
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1.6201
1.6037
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1.4987
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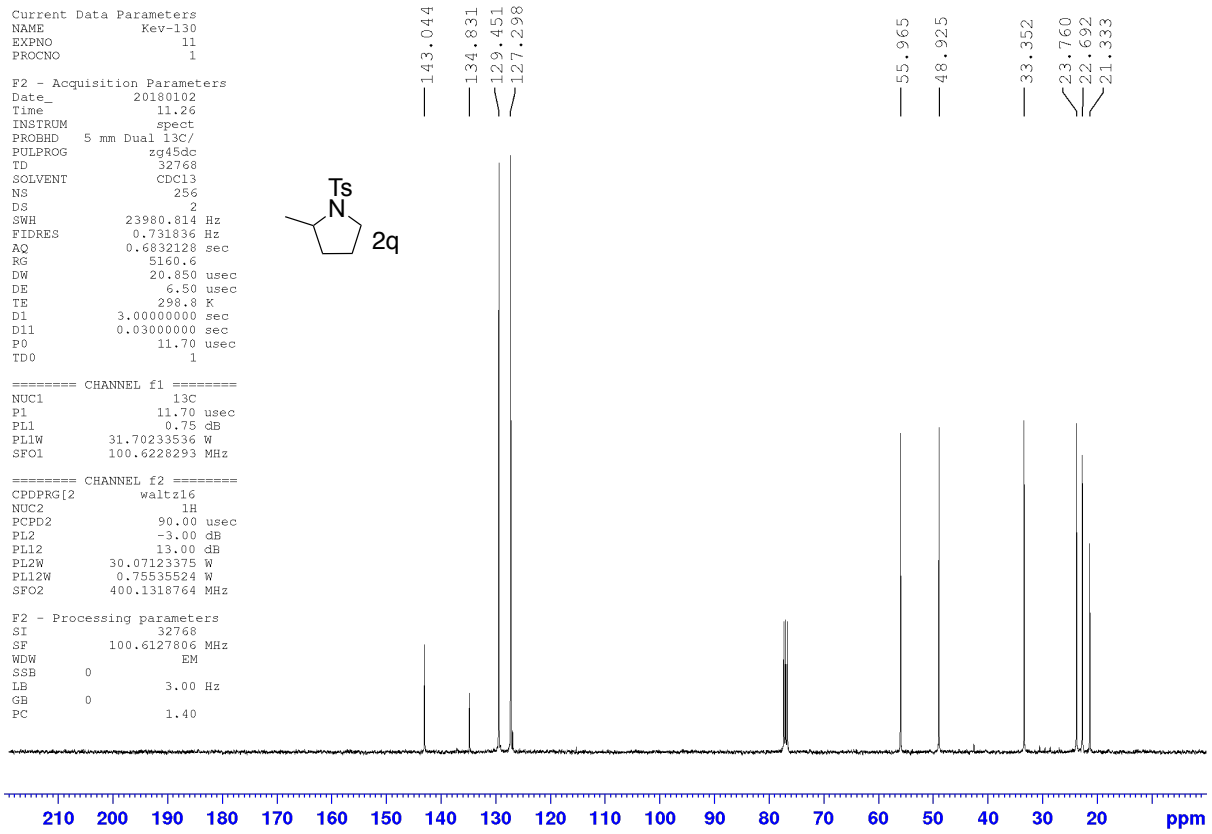
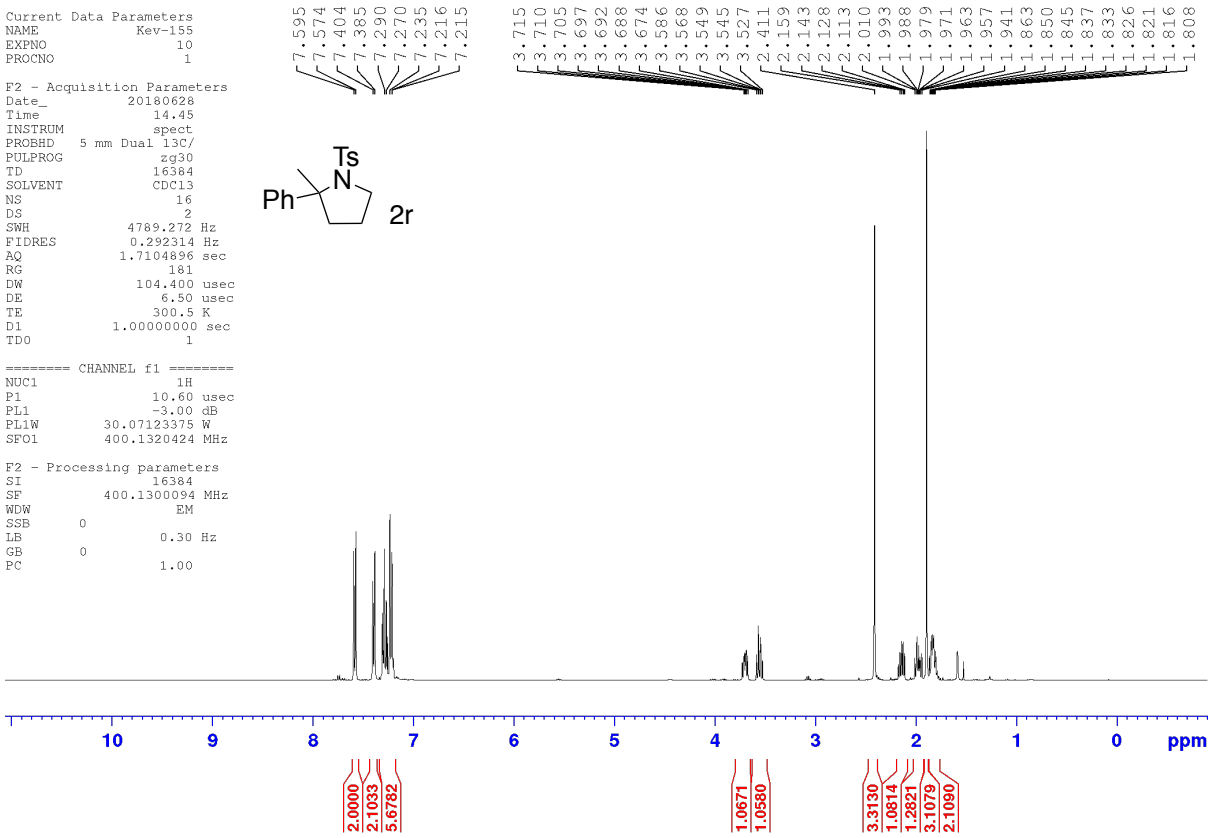
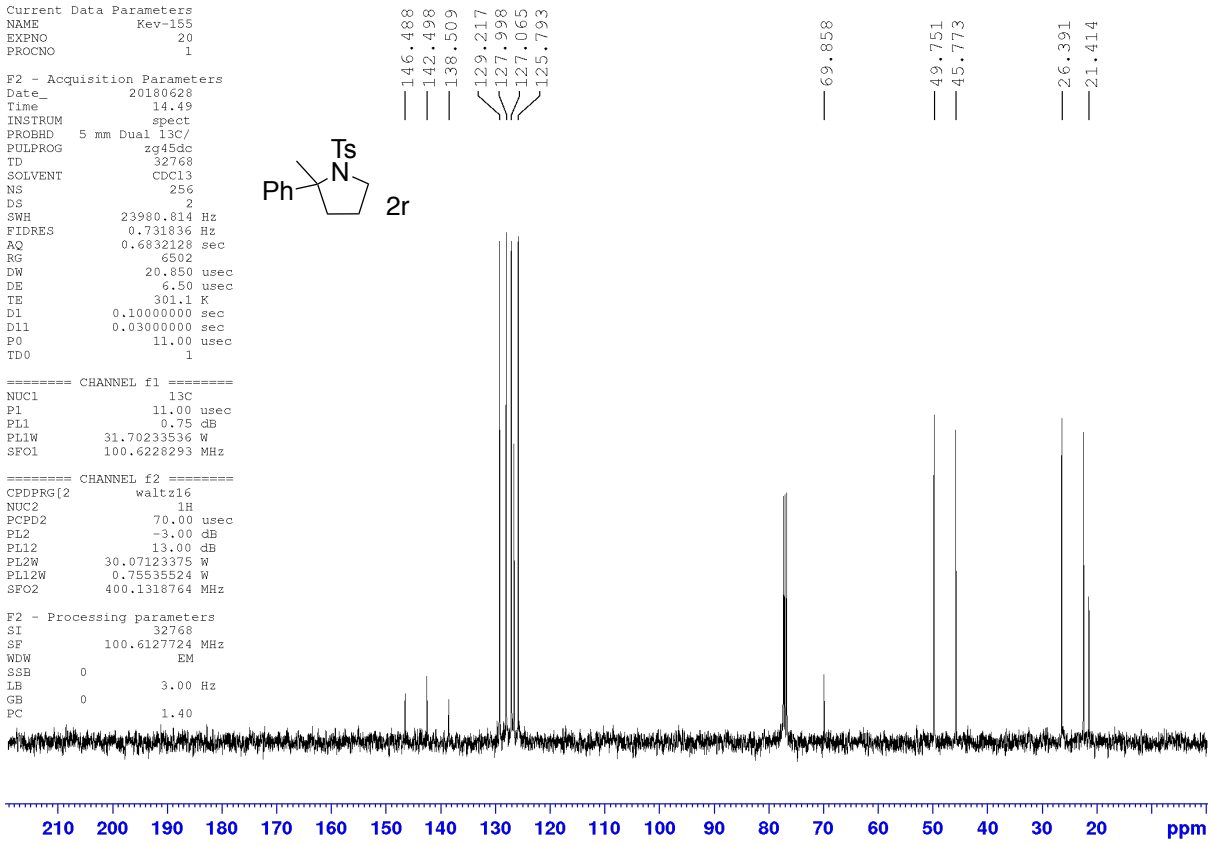
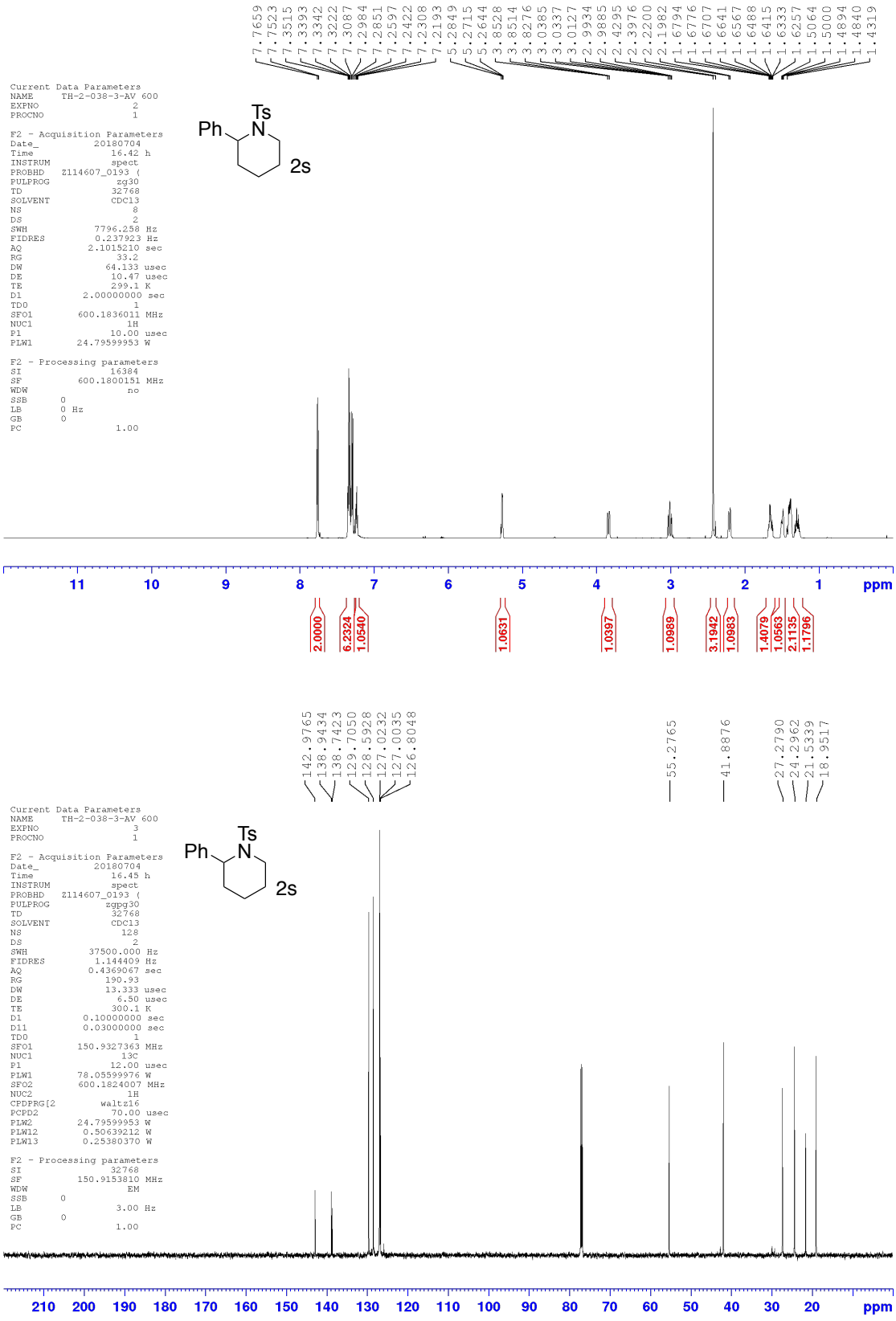
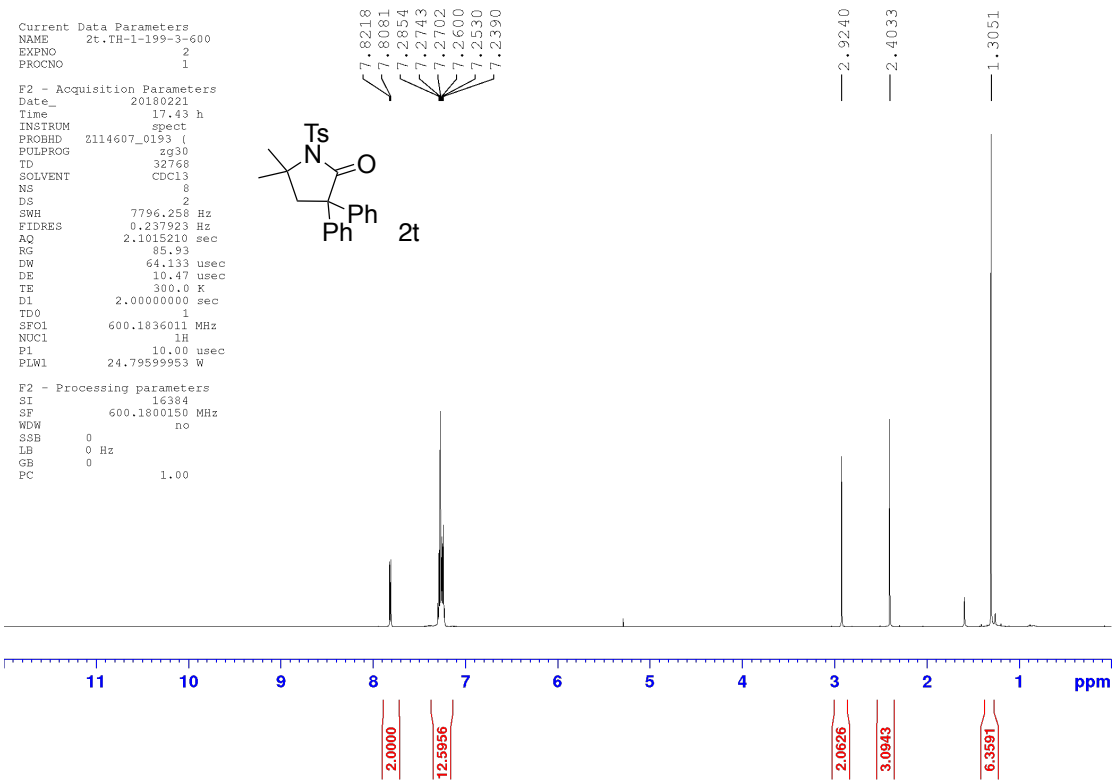
1.4824
1.4733
1.4686
1.4656
1.4611
1.4489
1.4369
1.4323
1.4206
1.2798
1.2639
Current Data Parameters
NAME Kev-130
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180102
Time 11.16
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7104896 sec
RG 35.9
DW 104.400 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.90 dB
PL1W 29.38673019 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ts
N
2q
10
9
8
7
6
5
4
3
2
1
ppm
2.0014
2.0519
1.0000
1.0685
1.0741
3.2488
1.1880
1.1013
2.2086
3.2048

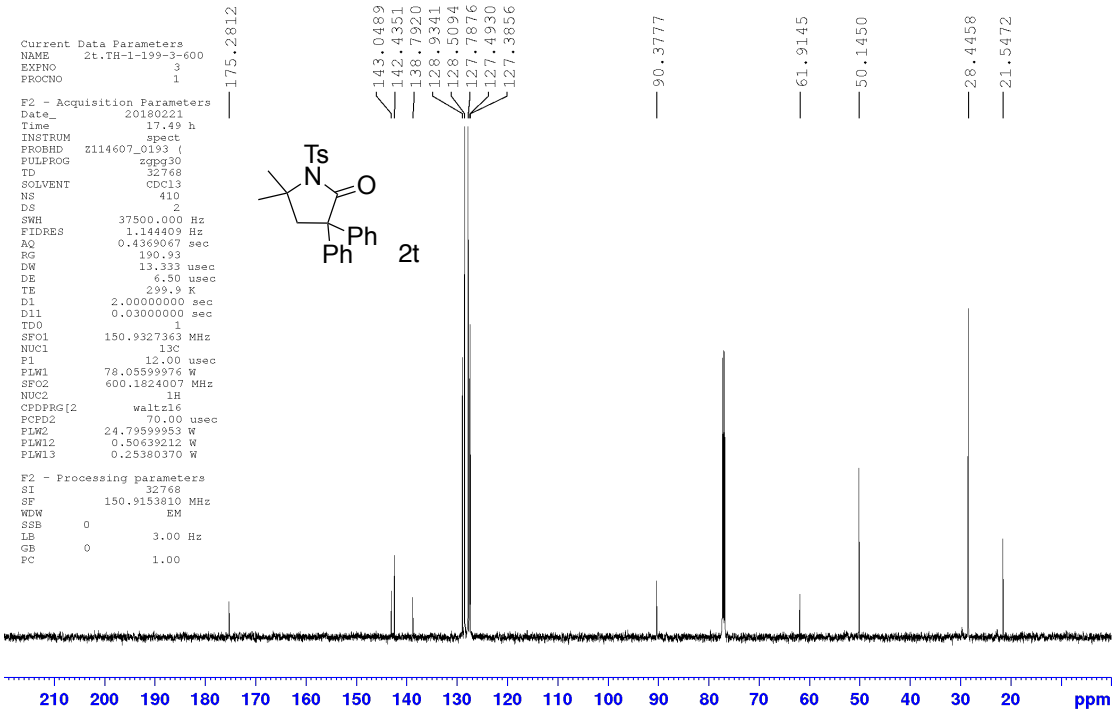

143.044
134.831
129.451
127.298
55.965
48.925
33.352
23.760
22.692
21.333
Current Data Parameters
NAME Kev-130
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180102
Time 11.26
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg45dc
TD 32768
SOLVENT CDCl3
NS 256
DS 2
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832128 sec
RG 5160.6
DW 20.850 usec
DE 6.50 usec
TE 298.8 K
D1 3.00000000 sec
D11 0.03000000 sec
P0 11.70 usec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.70 usec
PL1 0.75 dB
PL1W 31.70233536 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 13.00 dB
PL2W 30.07123375 W
PL12W 0.75535524 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127806 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
Ts
N
2q
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

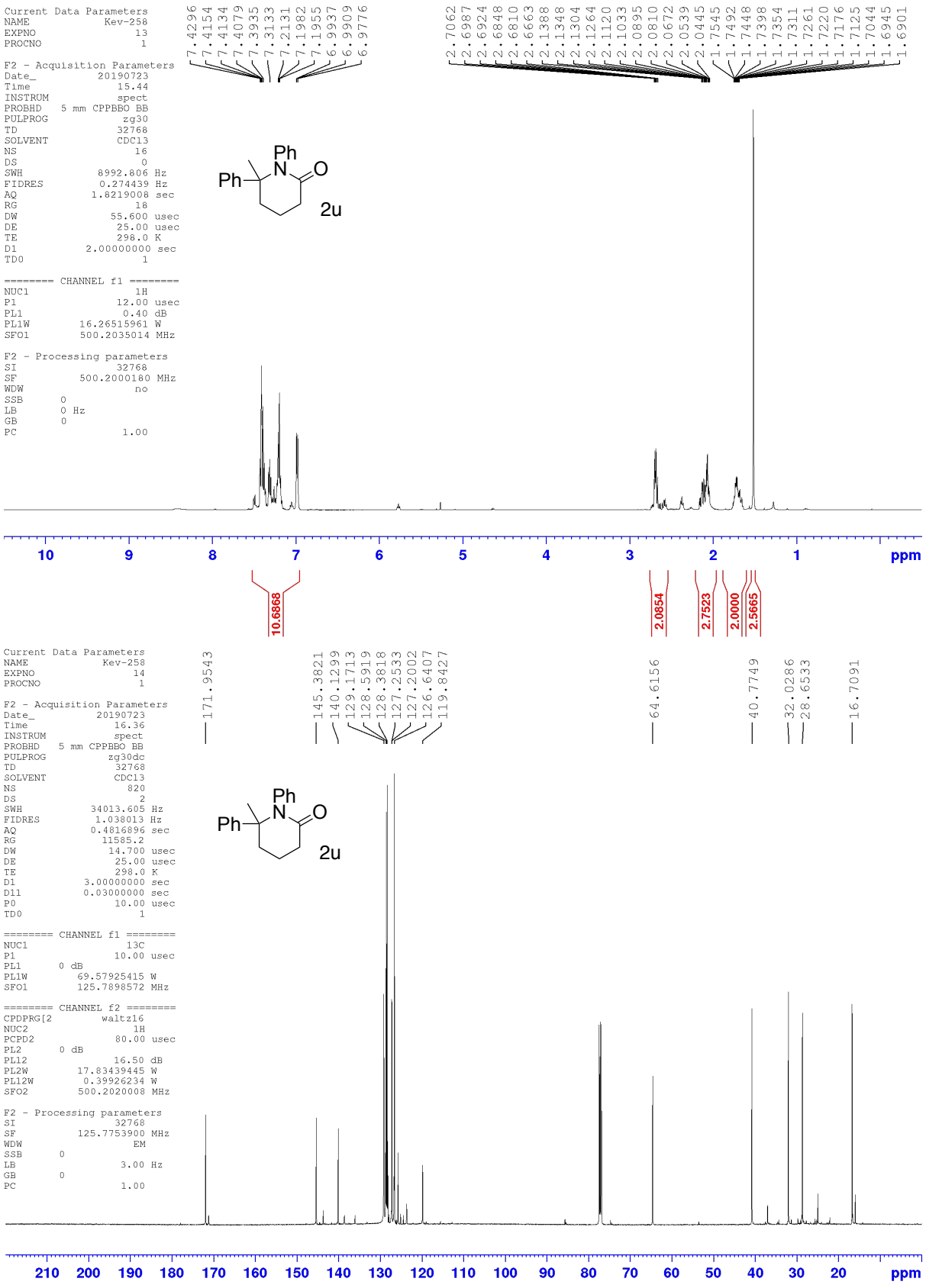

Current Data Parameters
NAME Kev-155
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180628
Time 14.45
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7104896 sec
RG 181
DW 104.400 usec
DE 6.50 usec
TE 300.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.60 usec
PL1 -3.00 dB
PL1W 30.07123375 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.595 7.574 7.404 7.385 7.290 7.270 7.235 7.216 7.215
3.715 3.710 3.705 3.697 3.692 3.688 3.674 3.586 3.568 3.549 3.545 3.527 2.411 2.159 2.143 2.128 2.113 2.010 1.993 1.988 1.979 1.971 1.963 1.957 1.941 1.863 1.850 1.845 1.837 1.833 1.826 1.821 1.816 1.808
Ts
N
Ph
2r
10 9 8 7 6 5 4 3 2 1 0 ppm
2.0000 2.1033 5.6762 1.0671 1.0580 3.3130 1.0614 1.2821 3.1079 2.1090

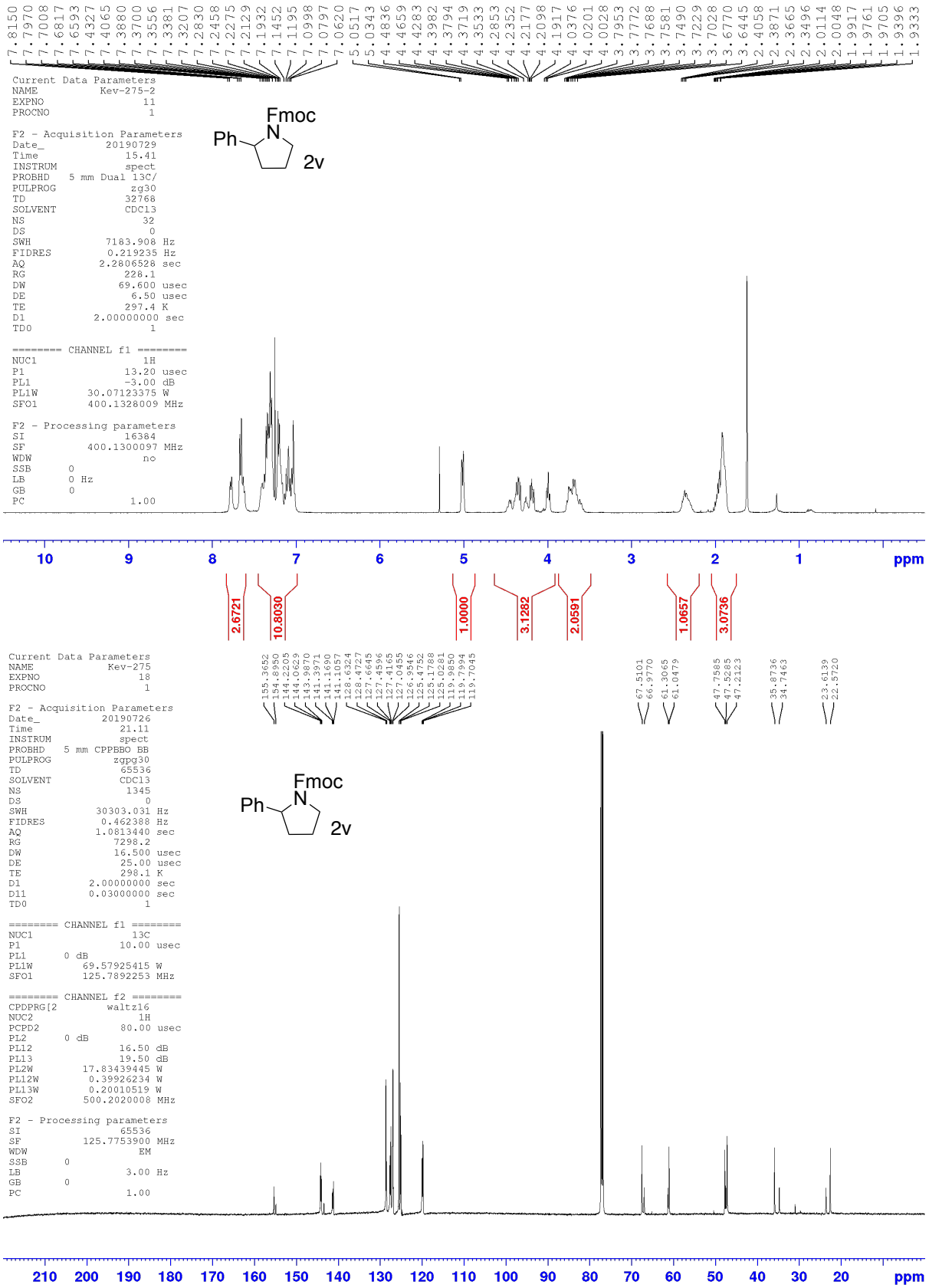

Current Data Parameters
NAME Kev-155
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180628
Time 14.49
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg45dc
TD 32768
SOLVENT CDCl3
NS 256
DS 2
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832128 sec
RG 6502
DW 20.850 usec
DE 6.50 usec
TE 301.1 K
D1 0.10000000 sec
D11 0.03000000 sec
P0 11.00 usec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0.75 dB
PL1W 31.70233536 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 13.00 dB
PL2W 30.07123375 W
PL12W 0.75535524 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127724 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
146.488 142.498 138.509 129.217 127.998 127.065 125.793 69.858 49.751 45.773 26.391 21.414
Ts
N
Ph
2r
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

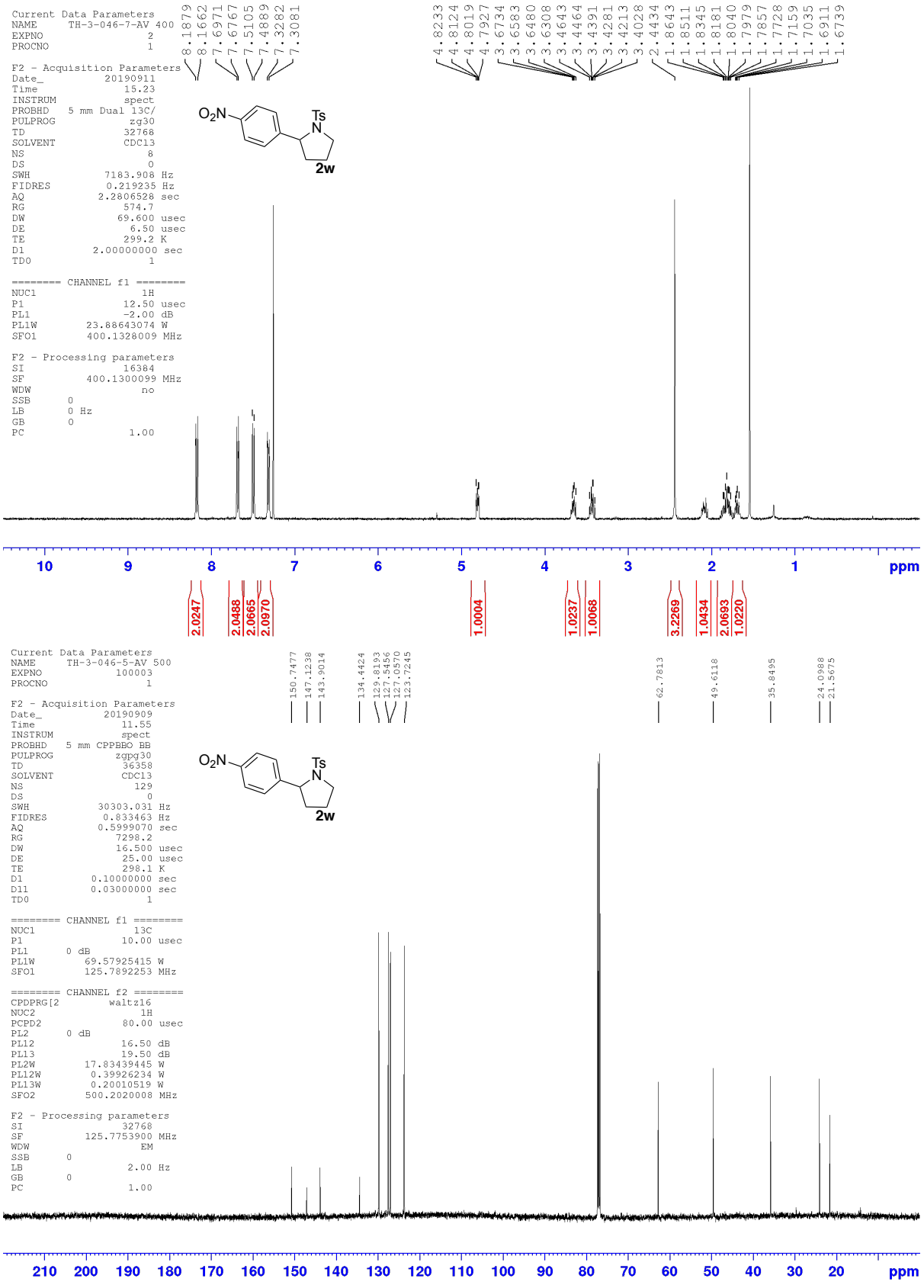

7.7659
7.7523
7.3515
7.3393
7.3342
7.3222
7.3087
7.2984
7.2851
7.2597
7.2422
7.2308
7.2193
5.2849
5.2715
5.2644
3.8528
3.8514
3.8276
3.0385
3.0337
3.0127
2.9934
2.9885
2.4295
2.3976
2.2200
2.1982
1.6794
1.6776
1.6707
1.6641
1.6567
1.6488
1.6415
1.6333
1.6257
1.5064
1.5000
1.4894
1.4840
1.4319
Current Data Parameters
NAME TH-2-038-3-AV 600
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180704
Time 16.42 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015210 sec
RG 33.2
DW 64.133 usec
DE 10.47 usec
TE 299.1 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800151 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
Ts
Ph
N
2s
11
10
9
8
7
6
5
4
3
2
1
ppm
2.0000
6.2324
1.0540
1.0631
1.0397
1.0989
3.1942
1.0983
1.4079
1.0563
2.1135
1.1796
142.9765
138.9434
138.7423
129.7050
128.5928
127.0232
127.0035
126.8048
55.2765
41.8876
27.2790
24.2962
21.5339
18.9517
Current Data Parameters
NAME TH-2-038-3-AV 600
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180704
Time 16.45 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 128
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369067 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 300.1 K
D1 0.10000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9327363 MHz
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO2 600.1824007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
F2 - Processing parameters
SI 32768
SF 150.9153810 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
Ts
Ph
N
2s
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Current Data Parameters
NAME 2t.TH-1-199-3-600
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180221
Time 17.43 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015210 sec
RG 85.93
DW 64.133 usec
DE 10.47 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800150 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
7.8218
7.8081
7.2854
7.2743
7.2702
7.2600
7.2530
7.2390
2.9240
2.4033
1.3051
Ts
N
O
Ph
Ph
2t
11
10
9
8
7
6
5
4
3
2
1
ppm
2.0000
12.5956
2.0626
3.0943
6.3591


Current Data Parameters
NAME 2t.TH-1-199-3-600
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180221
Time 17.49 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 410
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369067 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9327363 MHz
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO2 600.1824007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
F2 - Processing parameters
SI 32768
SF 150.9153810 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
175.2812
143.0489
142.4351
138.7920
128.9341
128.5094
127.7876
127.4930
127.3856
90.3777
61.9145
50.1450
28.4458
21.5472
Ts
N
O
Ph
Ph
2t
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Current Data Parameters
NAME Kev-258
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190723
Time 15.44
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 18
DW 55.600 usec
DE 25.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.40 dB
PL1W 16.26515961 W
SFO1 500.2035014 MHz
F2 - Processing parameters
SI 32768
SF 500.2000180 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
7.4296
7.4154
7.4134
7.4079
7.3935
7.3133
7.2131
7.1982
7.1955
6.9937
6.9909
6.9776
2.7062
2.6987
2.6924
2.6848
2.6810
2.6663
2.1388
2.1348
2.1304
2.1264
2.1120
2.1033
2.0895
2.0810
2.0672
2.0539
2.0445
1.7545
1.7492
1.7448
1.7398
1.7354
1.7311
1.7261
1.7220
1.7176
1.7125
1.7044
1.6945
1.6901
Ph
N
O
Ph
2u
10
9
8
7
6
5
4
3
2
1
ppm
10.6868
2.0854
2.7523
2.0000
2.5665
Current Data Parameters
NAME Kev-258
EXPNO 14
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190723
Time 16.36
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30dc
TD 32768
SOLVENT CDCl3
NS 820
DS 2
SWH 34013.605 Hz
FIDRES 1.038013 Hz
AQ 0.4816896 sec
RG 11585.2
DW 14.700 usec
DE 25.00 usec
TE 298.0 K
D1 3.00000000 sec
D11 0.03000000 sec
P0 10.00 usec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 0 dB
PL1W 69.57925415 W
SFO1 125.7898572 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0 dB
PL12 16.50 dB
PL2W 17.83439445 W
PL12W 0.39926234 W
SFO2 500.2020008 MHz
F2 - Processing parameters
SI 32768
SF 125.7753900 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
171.9543
145.3821
140.1299
129.1713
128.5919
128.3818
127.2533
127.2002
126.6407
119.8427
64.6156
40.7749
32.0286
28.6533
16.7091
Ph
N
O
Ph
2u
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


7.8150 7.7970 7.7008 7.6817 7.6593 7.4327 7.4065 7.3880 7.3700 7.3556 7.3381 7.3207 7.2830 7.2458 7.2275 7.2129 7.1932 7.1452 7.1195 7.0998 7.0797 7.0620 5.0517 5.0343 4.4836 4.4659 4.4283 4.3982 4.3794 4.3719 4.3533 4.2853 4.2352 4.2177 4.2098 4.1917 4.0376 4.0201 4.0028 3.7953 3.7772 3.7688 3.7581 3.7490 3.7229 3.7028 3.6770 3.6445 2.4058 2.3871 2.3665 2.3496 2.0114 2.0048 1.9917 1.9761 1.9705 1.9396 1.9333
Current Data Parameters
NAME Kev-275-2
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190729
Time 15.41
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 7183.908 Hz
FIDRES 0.219235 Hz
AQ 2.2806528 sec
RG 228.1
DW 69.600 usec
DE 6.50 usec
TE 297.4 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.20 usec
PL1 -3.00 dB
PL1W 30.07123375 W
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 16384
SF 400.1300097 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
Fmoc
Ph
N
2v
10 9 8 7 6 5 4 3 2 1 ppm
2.6721 10.8030 1.0000 3.1282 2.0591 1.0657 3.0736
Current Data Parameters
NAME Kev-275
EXPNO 18
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190726
Time 21.11
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1345
DS 0
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813440 sec
RG 7298.2
DW 16.500 usec
DE 25.00 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 0 dB
PL1W 69.57925415 W
SFO1 125.7892253 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0 dB
PL12 16.50 dB
PL13 19.50 dB
PL2W 17.83439445 W
PL12W 0.39926234 W
PL13W 0.20010519 W
SFO2 500.2020008 MHz
F2 - Processing parameters
SI 65536
SF 125.7753900 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
155.3652 154.8950 144.2205 144.0629 143.3970 141.3971 141.1690 141.1057 128.6324 128.4727 127.6645 127.4596 127.4165 127.0455 126.9546 125.4752 125.1788 125.0281 119.9850 119.7994 119.7045
67.5101 66.9770 61.3065 61.0479 47.7585 47.5285 47.2123 35.8736 34.7463 23.6139 22.5720
Fmoc
Ph
N
2v
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


Current Data Parameters
NAME TH-3-046-7-AV 400
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190911
Time 15.23
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 7183.908 Hz
FIDRES 0.219235 Hz
AQ 2.2806528 sec
RG 574.7
DW 69.600 usec
DE 6.50 usec
TE 299.2 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 16384
SF 400.1300099 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
8.1879
8.1662
7.6971
7.6767
7.5105
7.4889
7.3282
7.3081
4.8233
4.8124
4.8019
4.7927
3.6734
3.6583
3.6480
3.6308
3.4643
3.4464
3.4391
3.4281
3.4213
3.4028
2.4434
1.8643
1.8511
1.8345
1.8181
1.8040
1.7979
1.7857
1.7728
1.7159
1.7035
1.6911
1.6739
O2N
Ts
N
2w
10
9
8
7
6
5
4
3
2
1
ppm
2.0247
2.0488
2.0665
2.0970
1.0004
1.0237
1.0068
3.2269
1.0434
2.0693
1.0220
Current Data Parameters
NAME TH-3-046-5-AV 500
EXPNO 100003
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190909
Time 11.55
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 36358
SOLVENT CDCl3
NS 129
DS 0
SWH 30303.031 Hz
FIDRES 0.833463 Hz
AQ 0.5999070 sec
RG 7298.2
DW 16.500 usec
DE 25.00 usec
TE 298.1 K
D1 0.10000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 0 dB
PL1W 69.57925415 W
SFO1 125.7892253 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0 dB
PL12 16.50 dB
PL13 19.50 dB
PL2W 17.83439445 W
PL12W 0.39926234 W
PL13W 0.20010519 W
SFO2 500.2020008 MHz
F2 - Processing parameters
SI 32768
SF 125.7753900 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
150.7477
147.1238
143.9014
134.4424
129.8193
127.5456
127.0570
123.7245
62.7813
49.6118
35.8495
24.0988
21.5675
O2N
Ts
N
2w
210
200
190
180
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160
150
140
130
120
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100
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80
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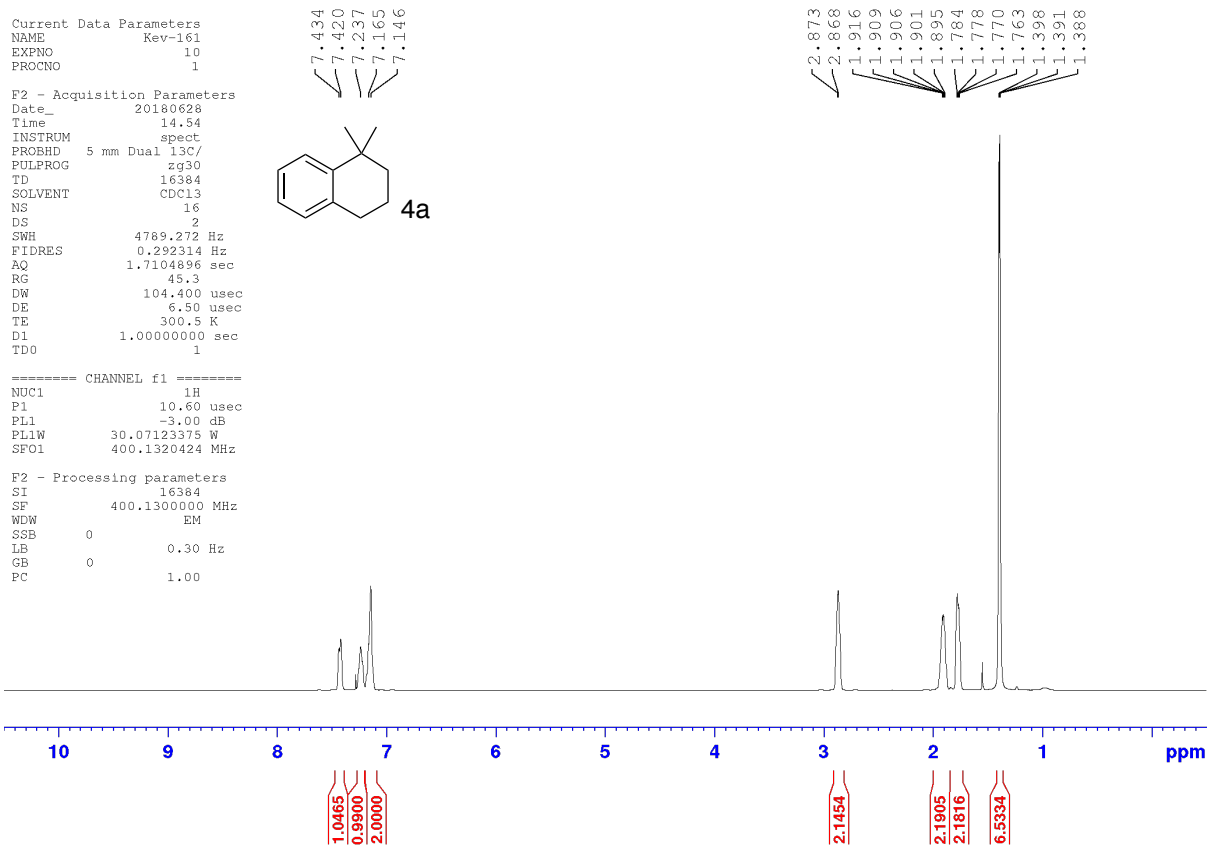
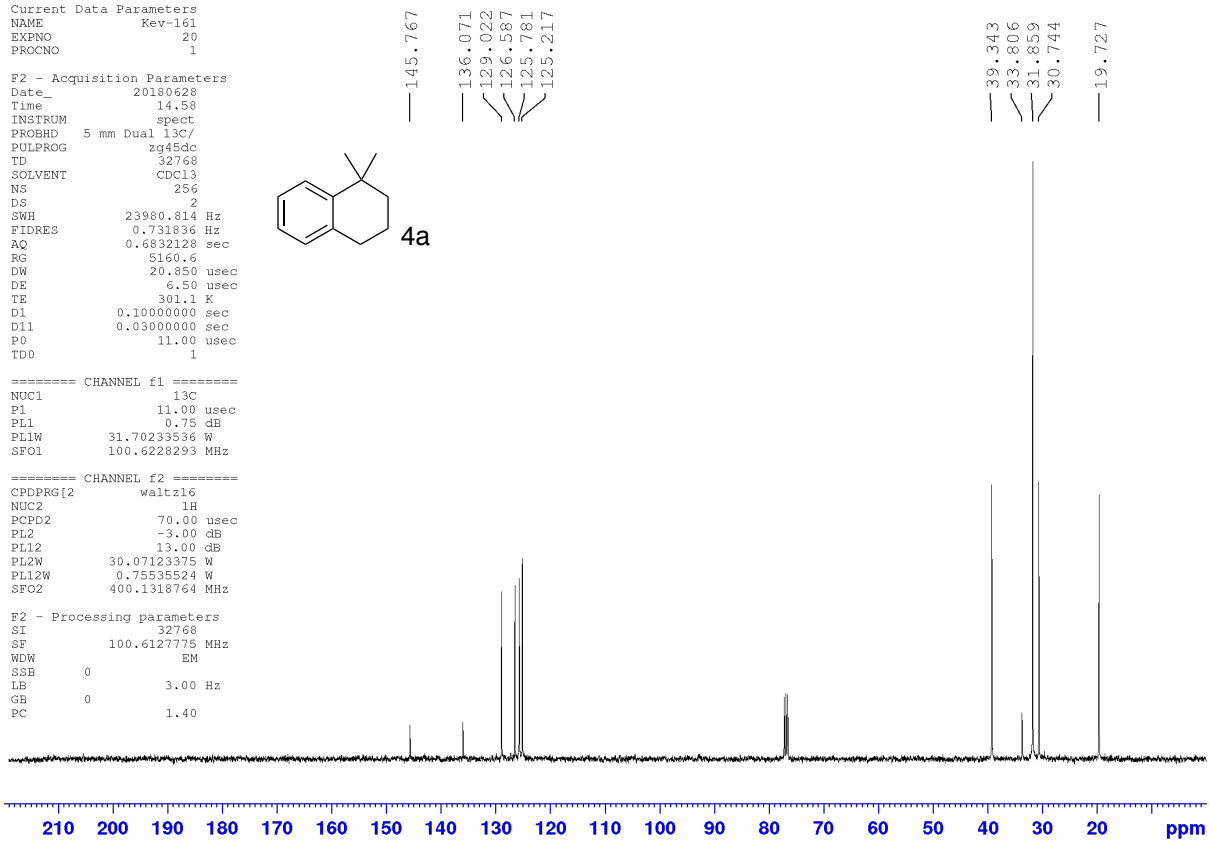
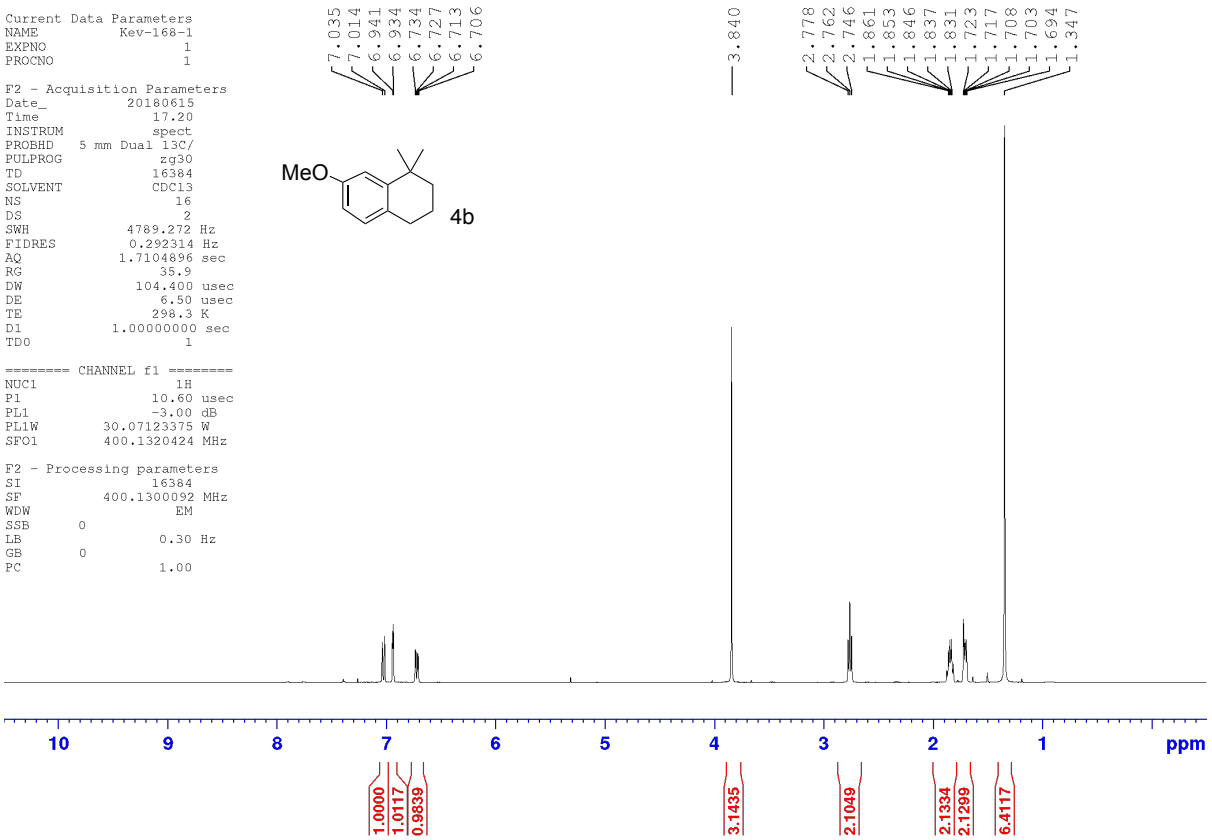
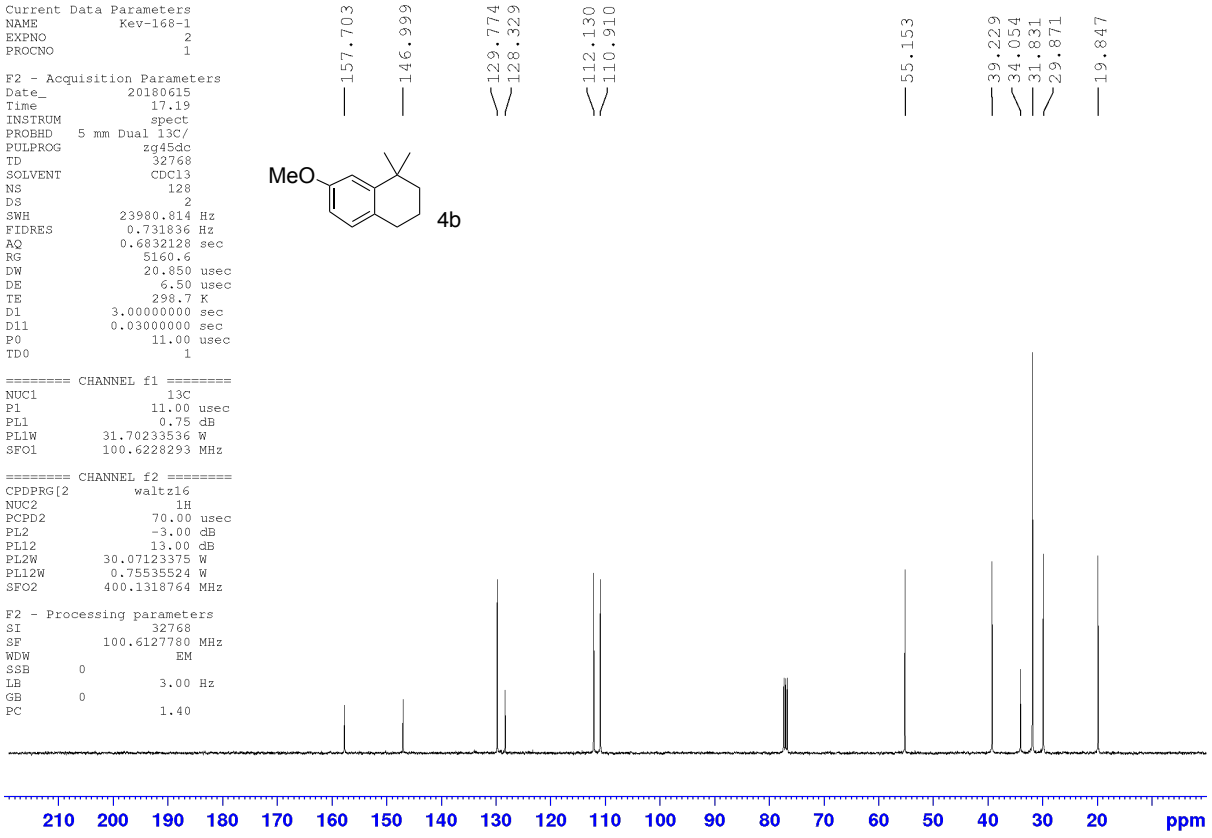
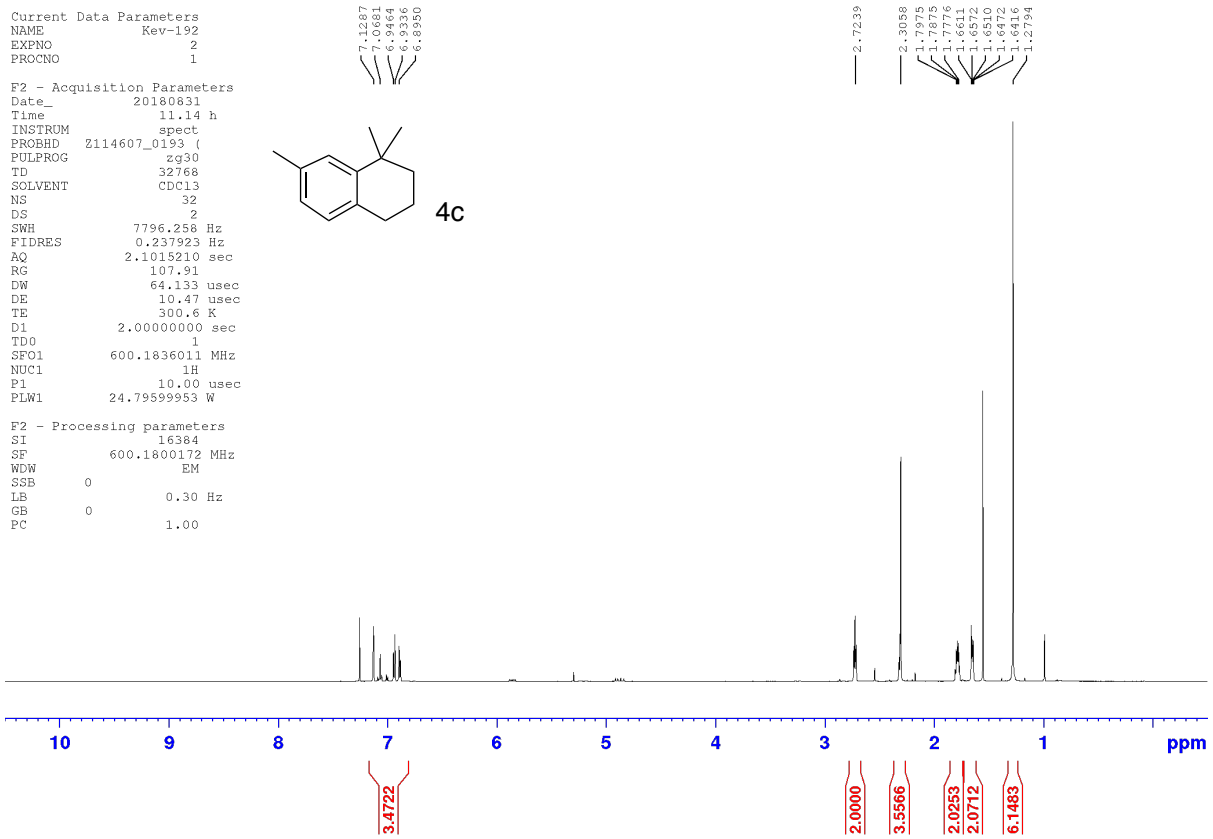
60
50
40
30
20
ppm

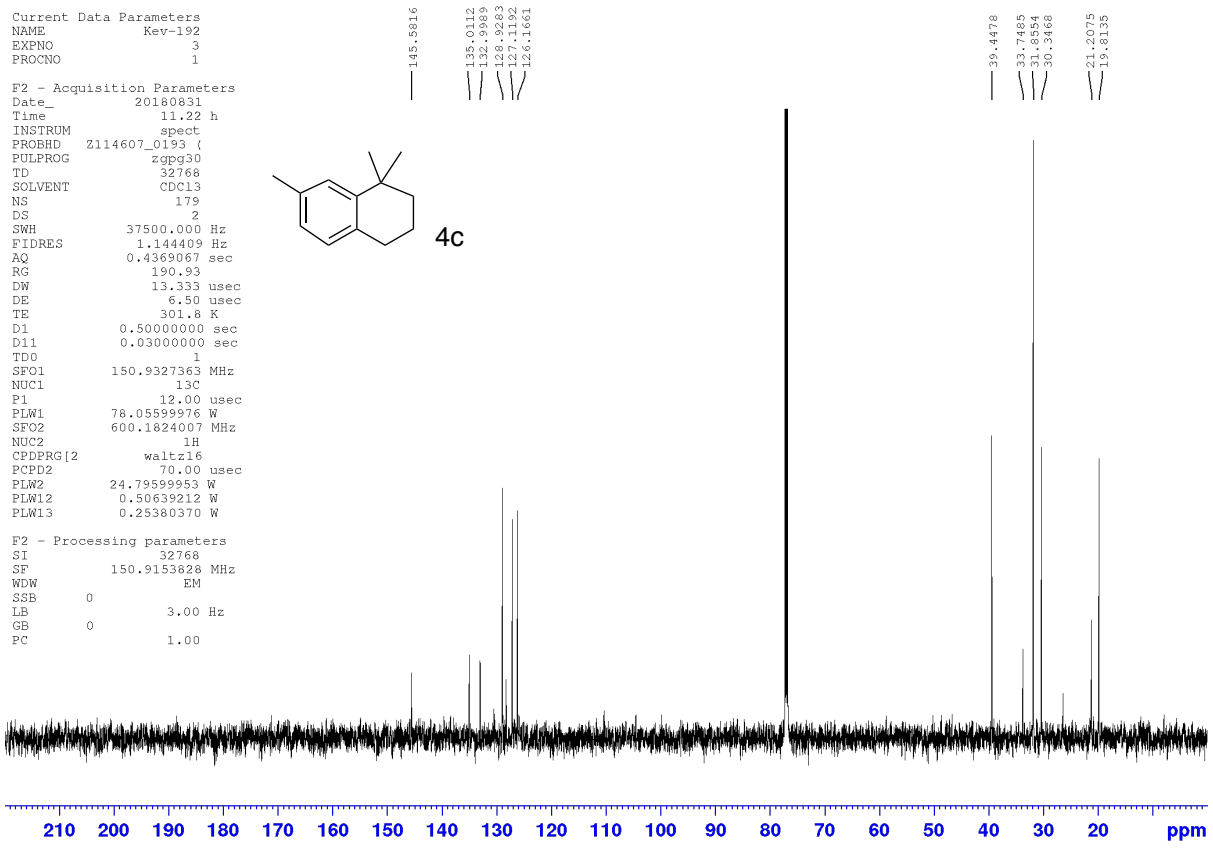

Current Data Parameters
NAME Kev-161
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180628
Time 14.54
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7104896 sec
RG 45.3
DW 104.400 usec
DE 6.50 usec
TE 300.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.60 usec
PL1 -3.00 dB
PL1W 30.07123375 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.434
7.420
7.237
7.165
7.146
2.873
2.868
1.916
1.909
1.906
1.901
1.895
1.784
1.778
1.770
1.763
1.398
1.391
1.388
4a
10
9
8
7
6
5
4
3
2
1
ppm
1.0465
0.9900
2.0000
2.1454
2.1905
2.1816
6.5334

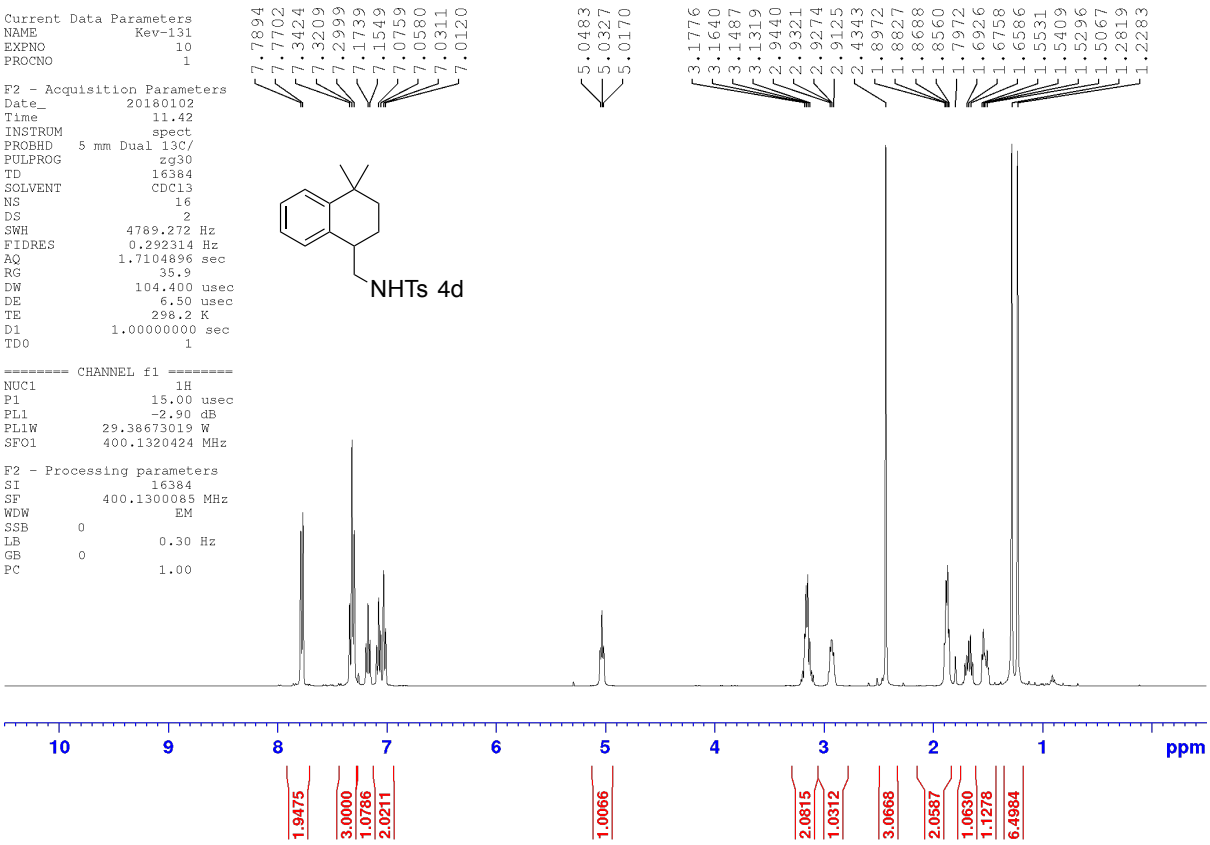

Current Data Parameters
NAME Kev-161
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180628
Time 14.58
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg45dc
TD 32768
SOLVENT CDCl3
NS 256
DS 2
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832128 sec
RG 5160.6
DW 20.850 usec
DE 6.50 usec
TE 301.1 K
D1 0.10000000 sec
D11 0.03000000 sec
P0 11.00 usec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0.75 dB
PL1W 31.70233536 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 13.00 dB
PL2W 30.07123375 W
PL12W 0.75535524 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127775 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
145.767
136.071
129.022
126.587
125.781
125.217
39.343
33.806
31.859
30.744
19.727
4a
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

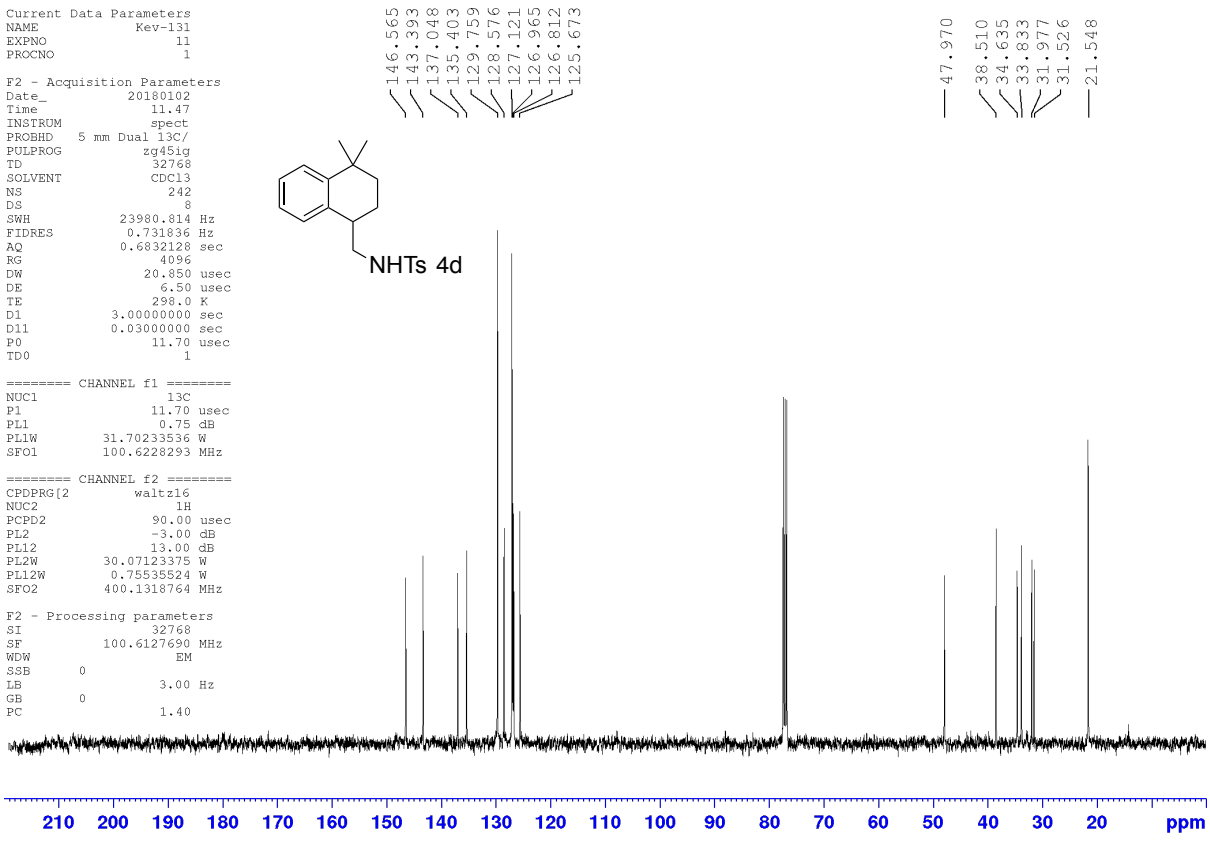

Current Data Parameters
NAME Kev-168-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180615
Time 17.20
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7104896 sec
RG 35.9
DW 104.400 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.60 usec
PL1 -3.00 dB
PL1W 30.07123375 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.035
7.014
6.941
6.934
6.734
6.727
6.713
6.706
3.840
2.778
2.762
2.746
1.861
1.853
1.846
1.837
1.831
1.723
1.717
1.708
1.703
1.694
1.347
MeO
4b
10
9
8
7
6
5
4
3
2
1
ppm
1.0000
1.0117
0.9839
3.1435
2.1049
2.1334
2.1299
6.4117

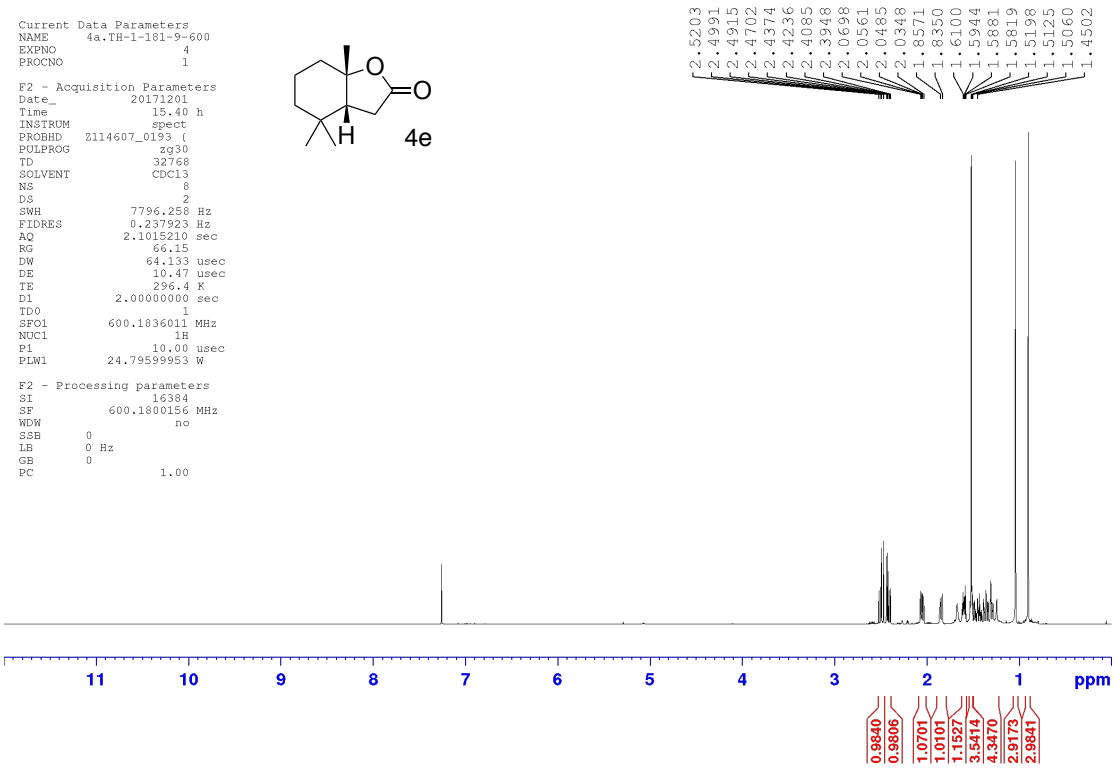

Current Data Parameters
NAME Kev-168-1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180615
Time 17.19
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg45dc
TD 32768
SOLVENT CDCl3
NS 128
DS 2
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832128 sec
RG 5160.6
DW 20.850 usec
DE 6.50 usec
TE 298.7 K
D1 3.00000000 sec
D11 0.03000000 sec
P0 11.00 usec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0.75 dB
PL1W 31.70233536 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 13.00 dB
PL2W 30.07123375 W
PL12W 0.75535524 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127780 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
157.703
146.999
129.774
128.329
112.130
110.910
55.153
39.229
34.054
31.831
29.871
19.847
MeO
4b
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

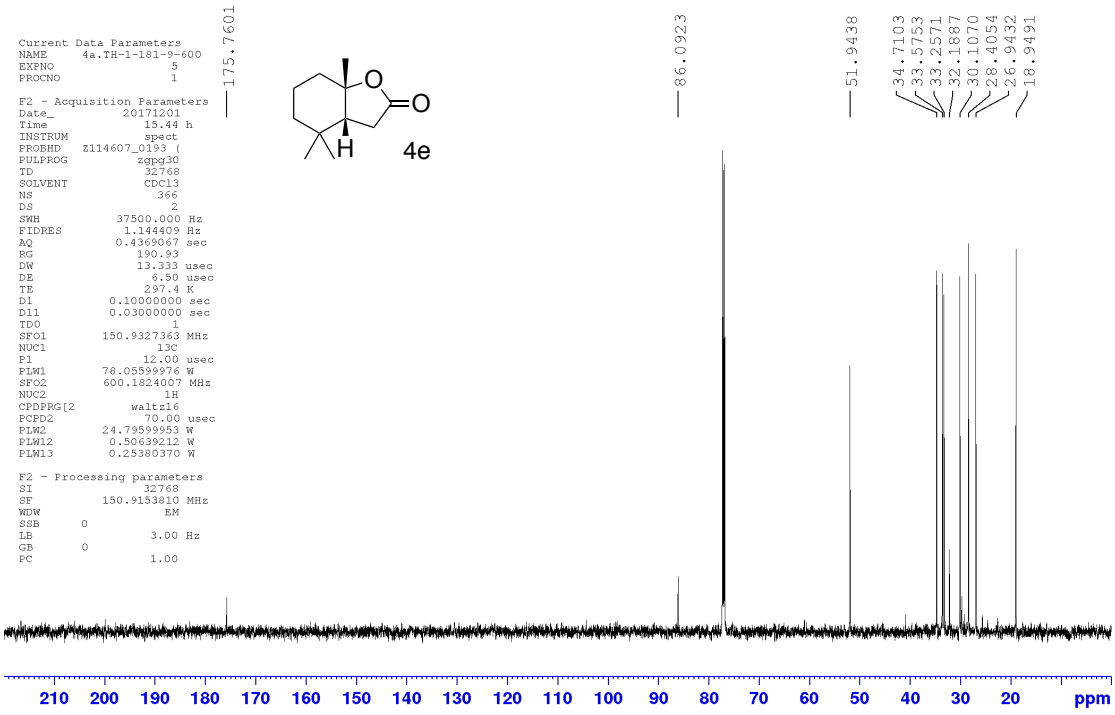

Current Data Parameters
NAME Kev-192
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180831
Time 11.14 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015210 sec
RG 107.91
DW 64.133 usec
DE 10.47 usec
TE 300.6 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.1287
7.0681
6.9464
6.9336
6.8950
2.7239
2.3058
1.7975
1.7873
1.7776
1.7611
1.6571
1.6510
1.6472
1.6416
1.2794
4c
10
9
8
7
6
5
4
3
2
1
ppm
3.4722
2.0000
3.5566
2.0253
2.0712
6.1483

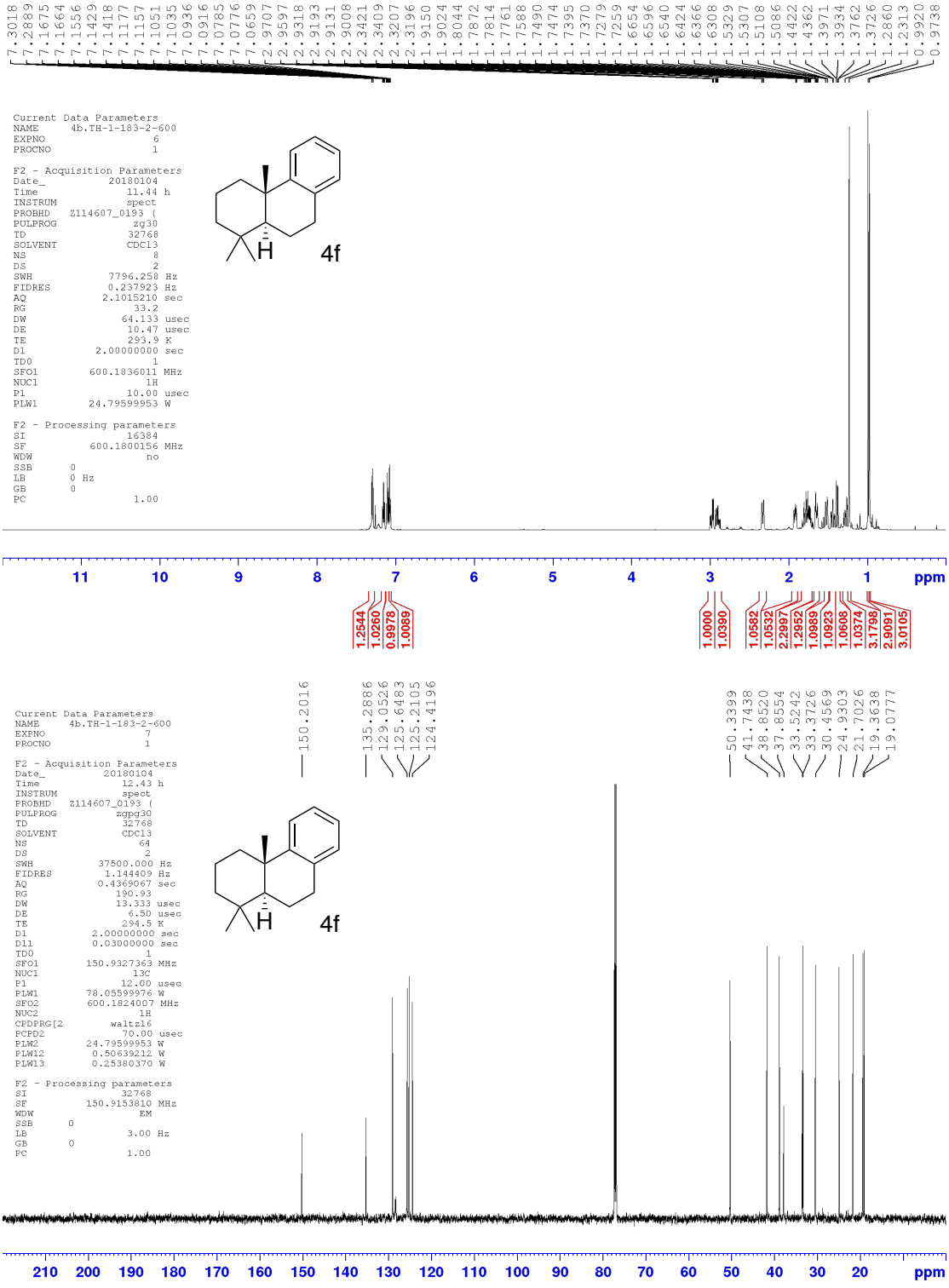

Current Data Parameters
NAME Kev-192
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180831
Time 11.22 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 179
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369067 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 301.8 K
D1 0.50000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9327363 MHz
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO2 600.1824007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
F2 - Processing parameters
SI 32768
SF 150.9153828 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
145.5816
135.0112
132.9989
129.9283
127.1192
126.1661
39.4478
33.7485
31.8554
30.3468
21.2075
19.8135
4c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Current Data Parameters
NAME Kev-131
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180102
Time 11.42
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7104896 sec
RG 35.9
DW 104.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -2.90 dB
PL1W 29.38673019 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300085 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.7894
7.7702
7.3424
7.3209
7.2999
7.1739
7.1549
7.0759
7.0580
7.0311
7.0120
5.0483
5.0327
5.0170
3.1776
3.1640
3.1487
3.1319
2.9440
2.9321
2.9274
2.9125
2.4343
1.8972
1.8827
1.8688
1.8560
1.7972
1.6926
1.6758
1.6586
1.5531
1.5409
1.5296
1.5067
1.2819
1.2283
NHTs 4d
10
9
8
7
6
5
4
3
2
1
ppm
1.9475
3.0000
1.0786
2.0211
1.0066
2.0815
1.0312
3.0668
2.0587
1.0630
1.1278
6.4984


Current Data Parameters
NAME Kev-131
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180102
Time 11.47
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg45ig
TD 32768
SOLVENT CDCl3
NS 242
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832128 sec
RG 4096
DW 20.850 usec
DE 6.50 usec
TE 298.0 K
D1 3.00000000 sec
D11 0.03000000 sec
P0 11.70 usec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.70 usec
PL1 0.75 dB
PL1W 31.70233536 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 13.00 dB
PL2W 30.07123375 W
PL12W 0.75535524 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
146.565
143.393
137.048
135.403
129.759
128.576
127.121
126.965
126.812
125.673
47.970
38.510
34.635
33.833
31.977
31.526
21.548
NHTs 4d
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Current Data Parameters
NAME 4a.TH-1-181-9-600
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171201
Time 15.40 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015210 sec
RG 66.15
DW 64.133 usec
DE 10.47 usec
TE 296.4 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800156 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
O
O
H
4e
2.5203
2.4991
2.4915
2.4702
2.4374
2.4236
2.4085
2.3948
2.0698
2.0561
2.0485
2.0348
1.8571
1.8350
1.6100
1.5944
1.5881
1.5819
1.5198
1.5125
1.5060
1.4502
11
10
9
8
7
6
5
4
3
2
1
ppm
0.9840
0.9806
1.0701
1.0101
1.1527
3.5414
4.3470
2.9173
2.9841


Current Data Parameters
NAME 4a.TH-1-181-9-600
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171201
Time 15.44 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 366
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369067 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 297.4 K
D1 0.10000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9327363 MHz
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO2 600.1824007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
F2 - Processing parameters
SI 32768
SF 150.9153810 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
O
O
H
4e
175.7601
86.0923
51.9438
34.7103
33.5753
33.2571
32.1887
30.1070
28.4054
26.9432
18.9491
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


7.3018
7.2889
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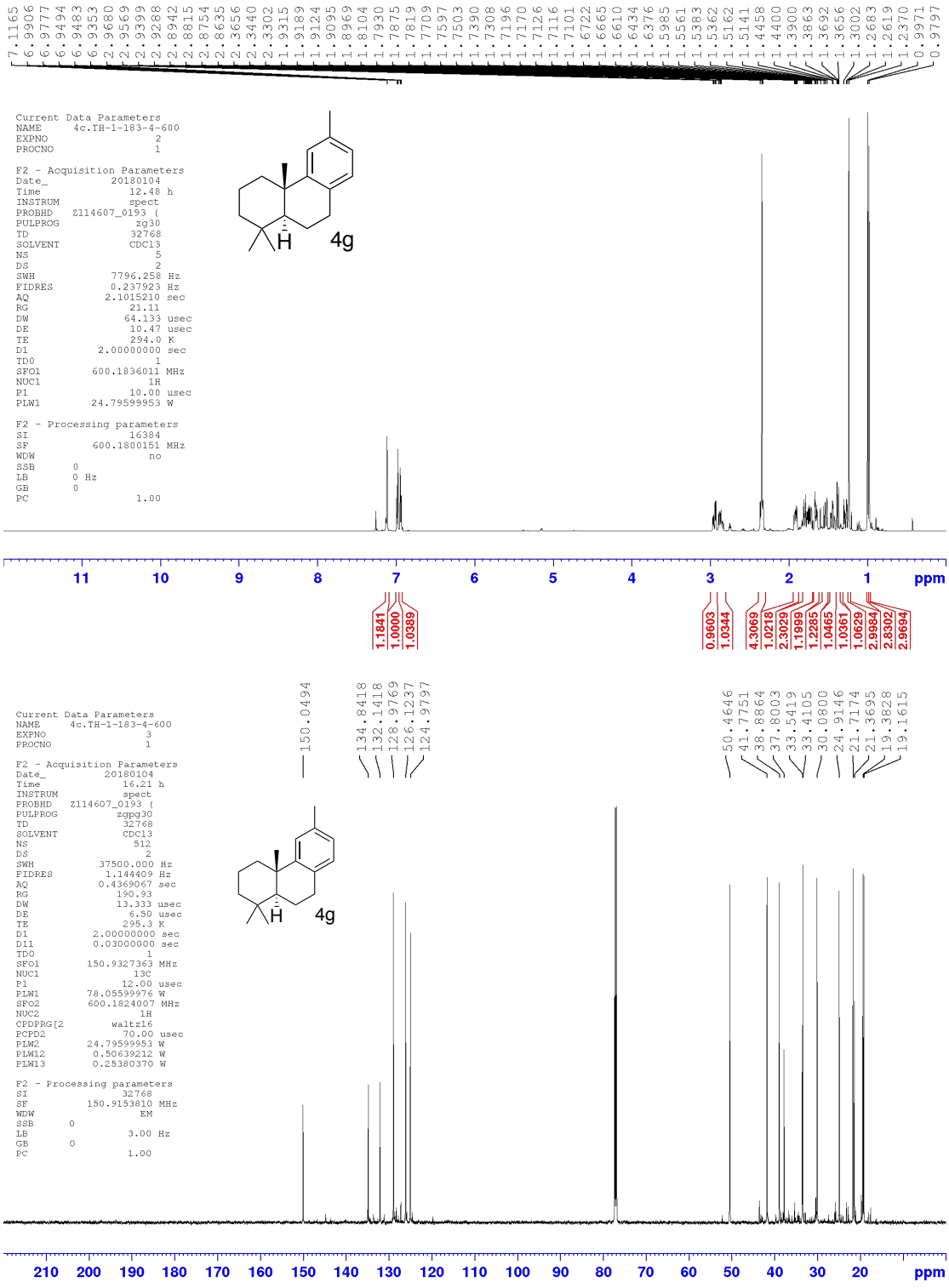
7.1556
7.1429
7.1418
7.1177
7.1157
7.1051
7.1035
7.0936
7.0916
7.0785
7.0776
7.0659
2.9707
2.9597
2.9318
2.9193
2.9131
2.9008
2.3421
2.3409
2.3207
2.3196
1.9150
1.9024
1.8044
1.7872
1.7814
1.7761
1.7588
1.7490
1.7474
1.7395
1.7370
1.7279
1.7259
1.6654
1.6596
1.6540
1.6424
1.6366
1.6308
1.5329
1.5307
1.5108
1.5086
1.4422
1.4362
1.3971
1.3934
1.3762
1.3726
1.2860
1.2313
0.9920
0.9738
Current Data Parameters
NAME 4b.TH-1-183-2-600
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180104
Time 11.44 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015210 sec
RG 33.2
DW 64.133 usec
DE 10.47 usec
TE 293.9 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800156 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
4f
11
10
9
8
7
6
5
4
3
2
1
ppm
1.2544
1.0260
0.9978
1.0089
1.0000
1.0390
1.0582
1.0532
2.2997
1.2952
1.0989
1.0923
1.0608
1.0374
3.1798
2.9091
3.0105
150.2016
135.2886
129.0526
125.6483
125.2105
124.4196
50.3399
41.7438
38.8520
37.8554
37.5242
33.3726
30.4569
24.9303
21.7026
19.3638
19.0777
Current Data Parameters
NAME 4b.TH-1-183-2-600
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180104
Time 12.43 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369067 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 294.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9327363 MHz
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO2 600.1824007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
F2 - Processing parameters
SI 32768
SF 150.9153810 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
4f
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


7.1165
6.9906
6.9777
6.9494
6.9483
6.9353
6.9680
2.9569
2.9399
2.9288
2.8942
2.8815
2.8754
2.8635
2.3656
2.3440
2.3302
2.1315
1.9189
1.9124
1.9095
1.8969
1.8104
1.7930
1.7875
1.7819
1.7709
1.7597
1.7503
1.7390
1.7308
1.7196
1.7170
1.7126
1.7116
1.7101
1.6722
1.6665
1.6610
1.6434
1.6376
1.5985
1.5561
1.5383
1.5362
1.5162
1.5141
1.4458
1.4400
1.3900
1.3863
1.3692
1.3656
1.3002
1.2683
1.2619
1.2370
0.9971
0.9797
Current Data Parameters
NAME 4c.TH-1-183-4-600
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180104
Time 12.48 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 5
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015210 sec
RG 21.11
DW 64.133 usec
DE 10.47 usec
TE 294.0 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800151 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
H
4g
11
10
9
8
7
6
5
4
3
2
1
ppm
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1.0000
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2.3029
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1.0465
1.0361
1.0629
2.9984
2.8302
2.9694
150.0494
134.8418
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EXPNO 3
PROCNO 1
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SOLVENT CDCl3
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FIDRES 1.144409 Hz
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RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 295.3 K
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D11 0.03000000 sec
TD0 1
SFO1 150.9327363 MHz
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO2 600.1824007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
F2 - Processing parameters
SI 32768
SF 150.9153810 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
H
4g
210
200
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180
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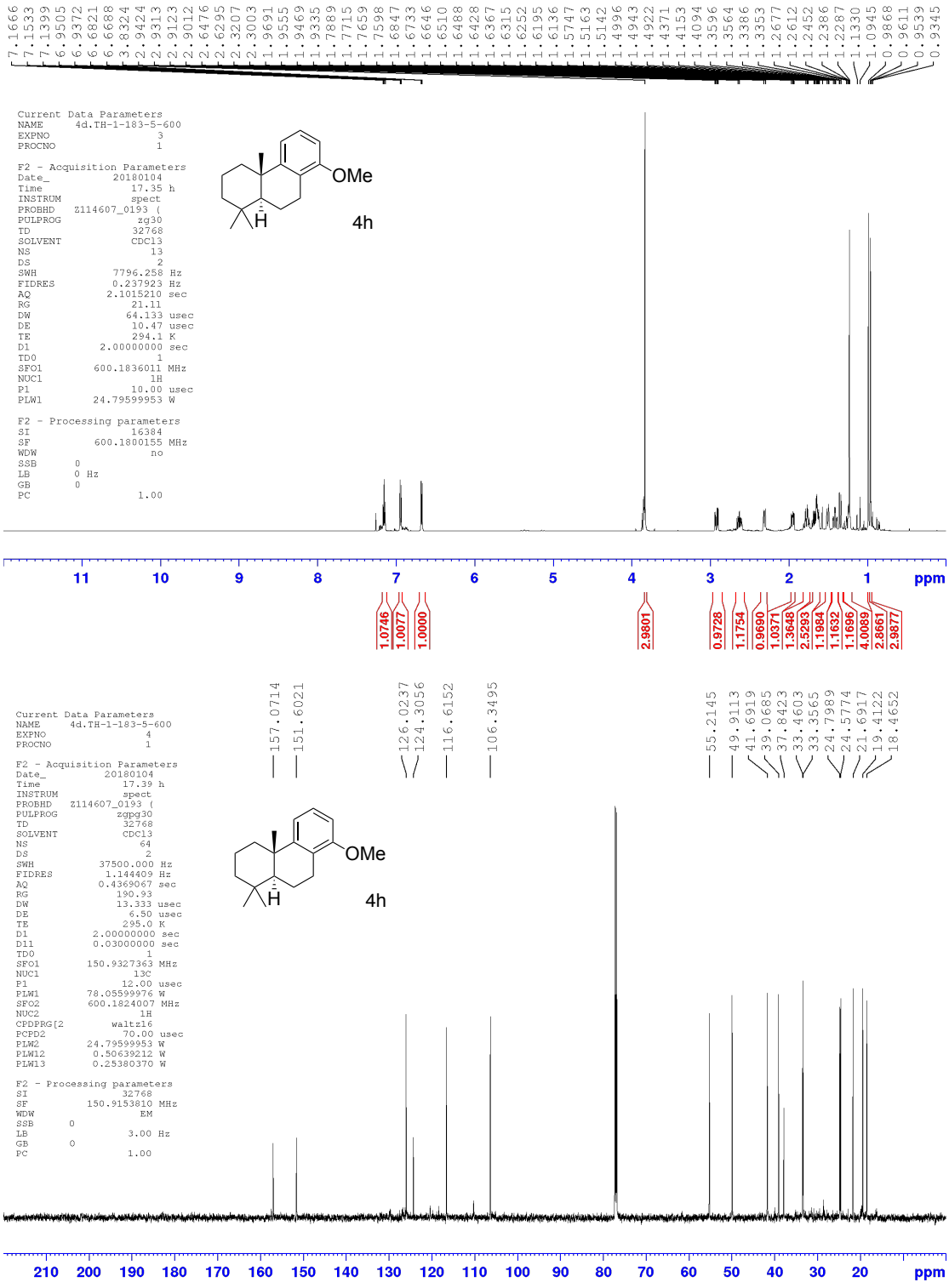
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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2.9012
2.6476
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1.7889
1.7715
1.7659
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1.6136
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1.4922
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NAME 4d.TH-1-183-5-600
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
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TD 32768
SOLVENT CDCl3
NS 13
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015210 sec
RG 21.11
DW 64.133 usec
DE 10.47 usec
TE 294.1 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800155 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
OMe
H
4h
11
10
9
8
7
6
5
4
3
2
1
ppm
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Current Data Parameters
NAME 4d.TH-1-183-5-600
EXPNO 4
PROCNO 1
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Date_ 20180104
Time 17.39 h
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PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369067 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9327363 MHz
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO2 600.1824007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
F2 - Processing parameters
SI 32768
SF 150.9153810 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
OMe
H
4h
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200
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130
120
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80
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50
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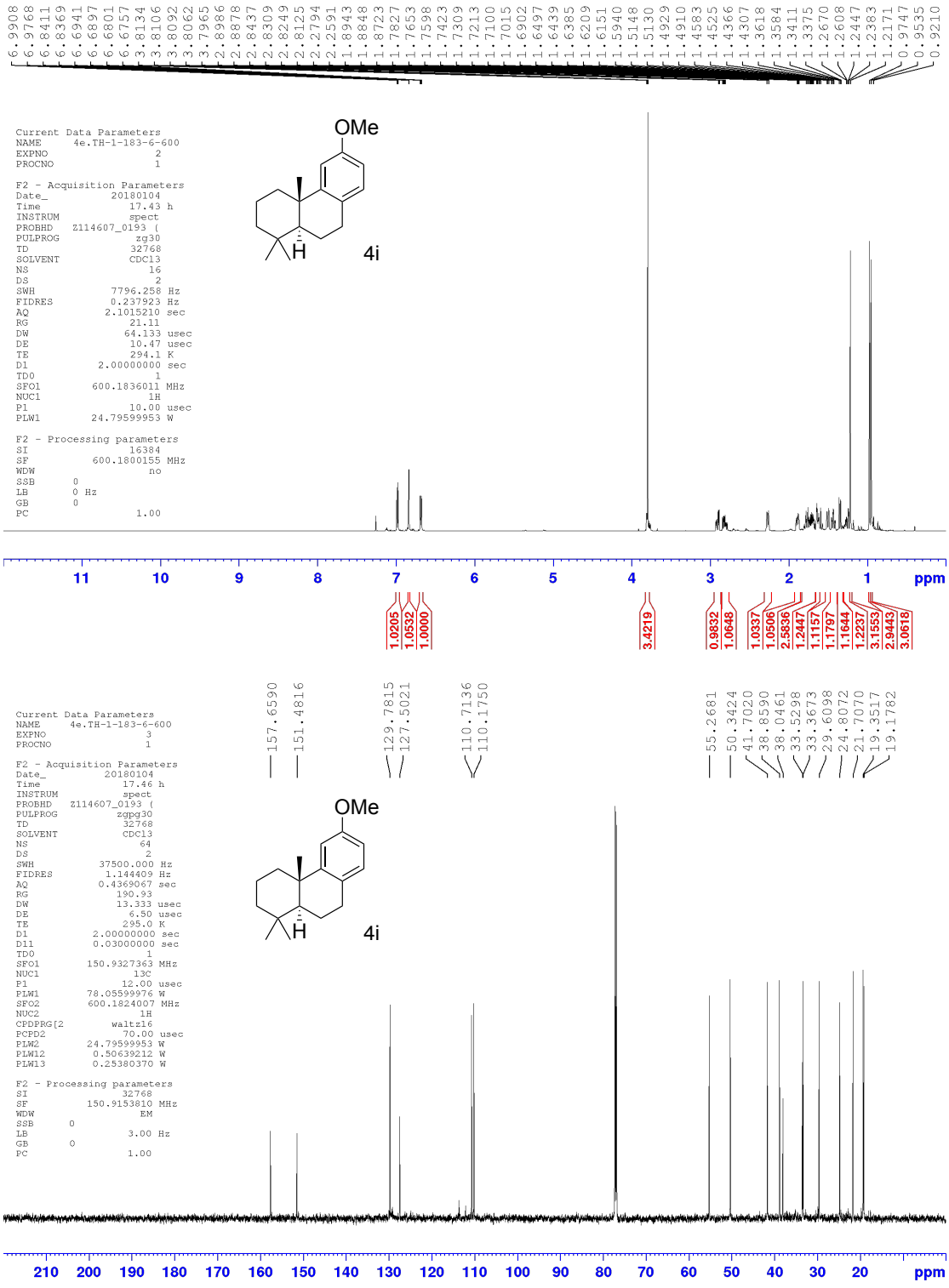
30
20
ppm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NAME 4e.TH-1-183-6-600
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
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PULPROG zg30
TD 32768
SOLVENT CDCl3
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FIDRES 0.237923 Hz
AQ 2.1015210 sec
RG 21.11
DW 64.133 usec
DE 10.47 usec
TE 294.1 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800155 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
OMe
H
4i
11
10
9
8
7
6
5
4
3
2
1
ppm
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1.0532
1.0000
3.4219
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2.5836
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1.1797
1.1644
1.2237
3.1553
2.9443
3.0618
157.6590
151.4816
129.7815
127.5021
110.7136
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55.2681
50.3424
41.7020
38.8590
38.0461
33.5298
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29.6098
24.8072
21.7070
19.3517
19.1782
Current Data Parameters
NAME 4e.TH-1-183-6-600
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180104
Time 17.46 h
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PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369067 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9327363 MHz
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO2 600.1824007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
F2 - Processing parameters
SI 32768
SF 150.9153810 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
OMe
H
4i
210
200
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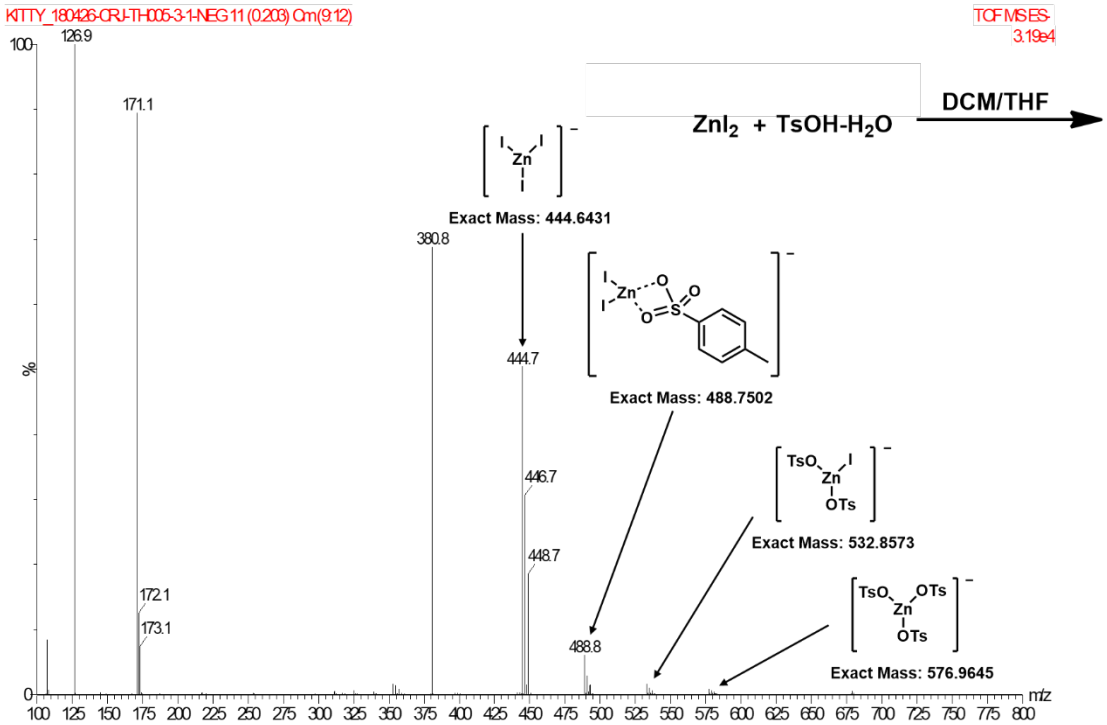
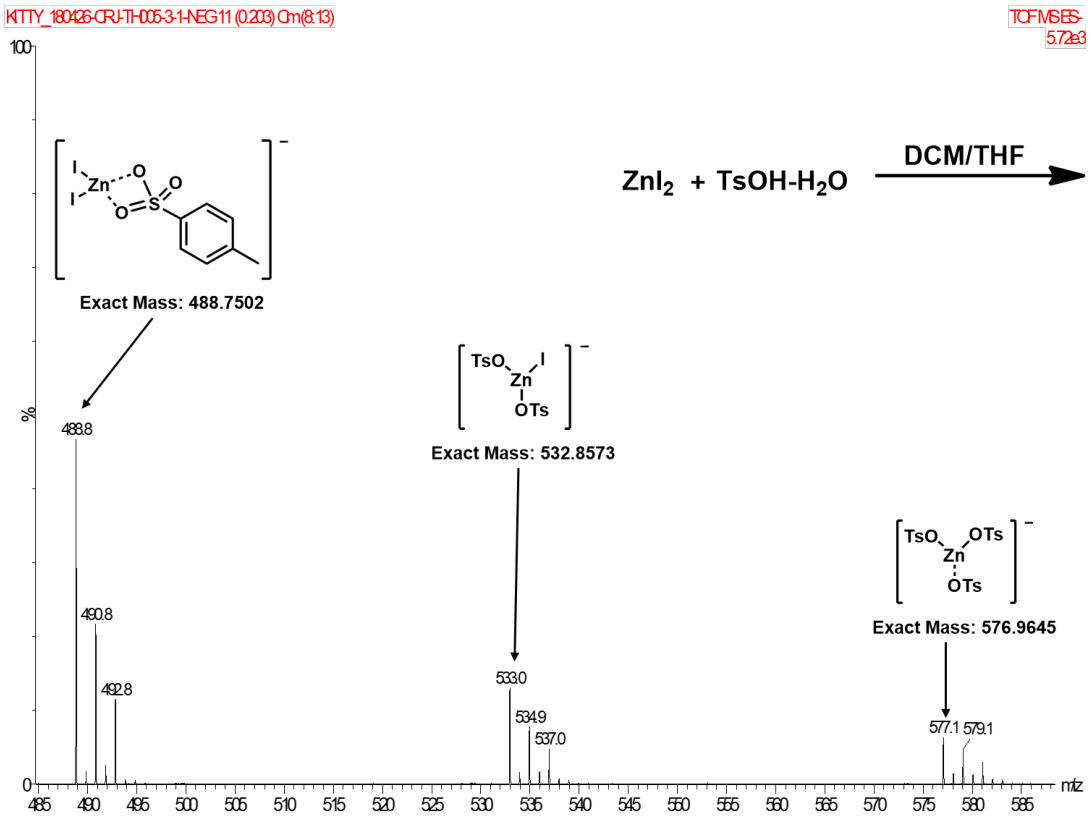
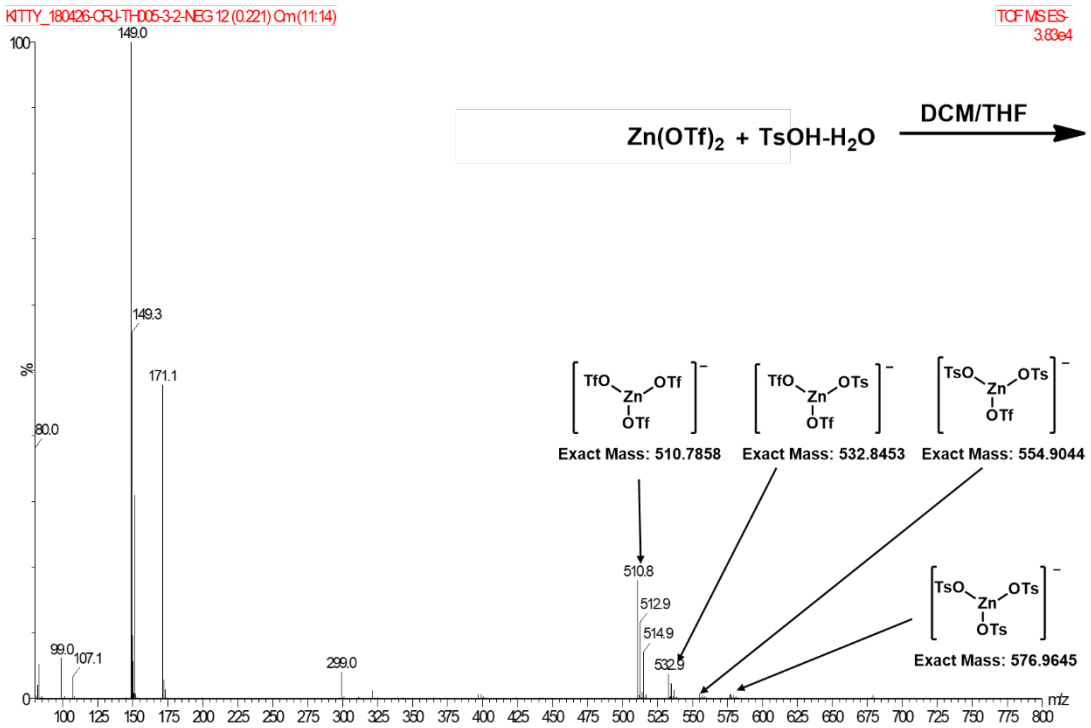
ppm

4. Mass Information



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TOF MS ES-
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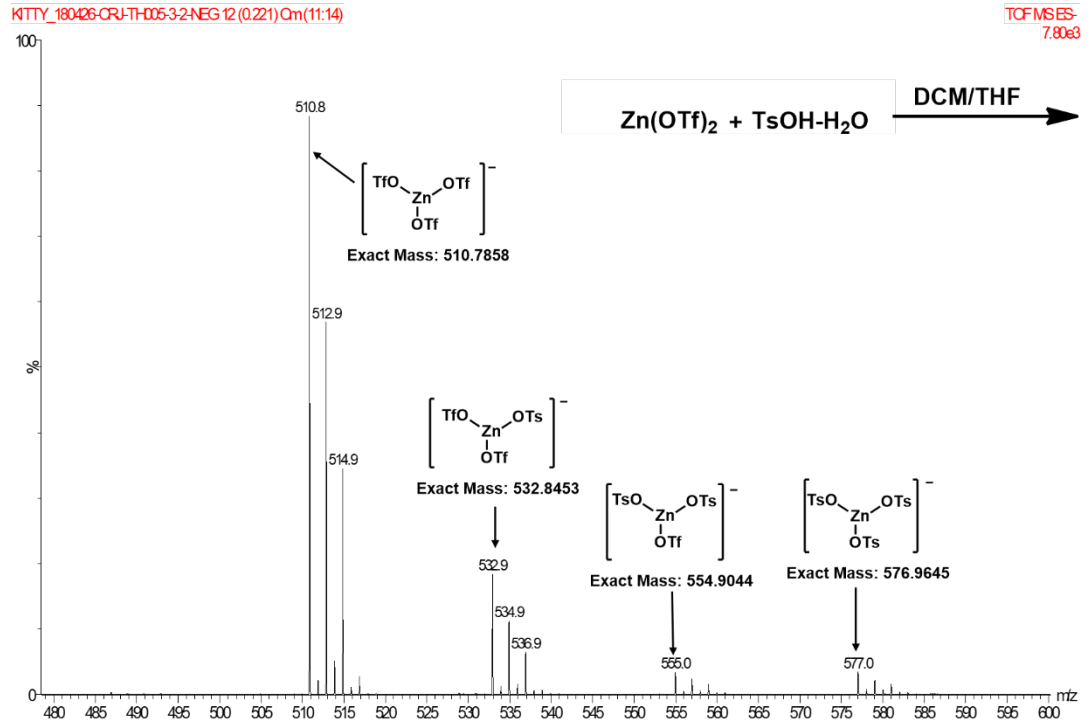
100
%
0
149.0
149.3
171.1
80.0
99.0
107.1
299.0
510.8
512.9
514.9
532.9
Zn(OTf)2 + TsOH-H2O
DCM/THF
TfO Zn OTf OTf
Exact Mass: 510.7858
TfO Zn OTs OTf
Exact Mass: 532.8453
TsO Zn OTs OTf
Exact Mass: 554.9044
TsO Zn OTs OTs
Exact Mass: 576.9645
m/z
100 125 150 175 200 225 250 275 300 325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700 725 750 775 800


KITTY_180426-CRU-TH005-3-2-NEG 12 (0.221) Cm (11:14)
TOF MS ES-
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100
%
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510.8
512.9
514.9
532.9
534.9
536.9
555.0
577.0
Zn(OTf)2 + TsOH-H2O
DCM/THF
TfO Zn OTf OTf
Exact Mass: 510.7858
TfO Zn OTs OTf
Exact Mass: 532.8453
TsO Zn OTs OTf
Exact Mass: 554.9044
TsO Zn OTs OTs
Exact Mass: 576.9645
m/z
480 485 490 495 500 505 510 515 520 525 530 535 540 545 550 555 560 565 570 575 580 585 590 595 600

5. References

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