Supporting Information

# Catalytic and Enantioselective *oxa*-Piancatelli Reaction Using Chiral Vanadium Complex

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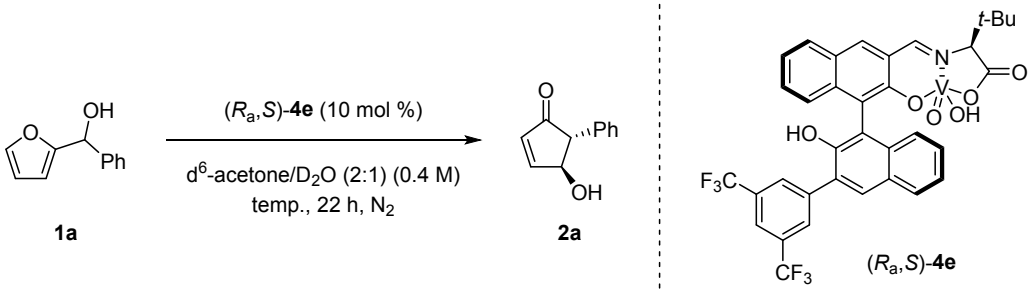
## 1. General information

$^{1}$H-, $^{13}$C-, and $^{19}$F-NMR were recorded with JEOL JMN ECS400 FT NMR, JNM ECA600 FT NMR or Bruker AVANCE II ($^{1}$H-NMR 400, 600 or 700 MHz, $^{13}$C-NMR 100, 150 or 175 MHz, $^{19}$F-NMR 565 MHz.) $^{1}$H-NMR spectra are reported as follows: chemical shift in ppm relative to the chemical shift of tetramethylsilane (TMS) at 0 ppm, integration, multiplicities (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), and coupling constants (Hz). $^{13}$C-NMR spectra reported in ppm relative to the central line of triplet for $CDCl_3$ at 77 ppm. $CF_3CO_2H$ used as external standards for $^{19}$F-NMR. FT-MS spectra were obtained with LTQ Orbitrap XL (Thermo Fisher Scientific). ESI-MS spectra were obtained with JMS-T100LC (JEOL). Optical rotations were measured with JASCO P-1030 polarimeter. HPLC analyses were performed on a JASCO HPLC system (JASCO PU 980 pump and UV-975 UV/Vis detector) using a mixture of hexane and 2-propanol as eluents. FT-IR spectra were recorded on a JASCO FT-IR system (FT/IR4100). Column chromatography on $SiO_2$ was performed with Kanto Silica Gel 60 (63-210 μm). Commercially available organic and inorganic compounds were used without further purification.

## 2. Optimization of reaction conditions

Table S1. Screening of reaction temperature using ($R_a$,*S*)-**4e**

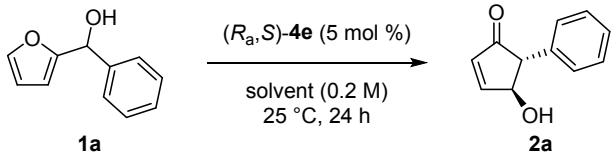


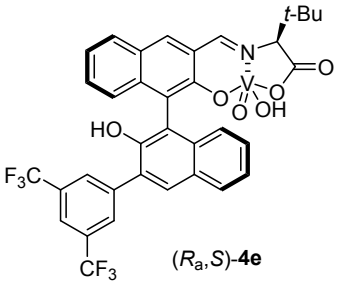

| entry | temp. (°C) | yield (%)[a] | er (%)[b] |
|---|---|---|---|
| 1 | 10 | 11 | 85:15 |
| 2 | 15 | 36 | 85:15 |
| 3 | 20 | 46 | 87.5:12.5 |
| 4 | 25 | 59 | 86:14 |
| 5 | 30 | 60 | 85:15 |
| 6 (under air) | 10 | 10 | 82:18 |

[a]NMR yield using 1,3,5-trimethoxybenzene as an internal standard.

[b]Determined by HPLC

Table S2. Screening of various reaction solvents

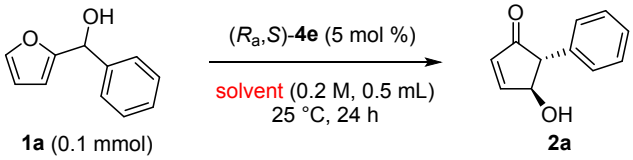


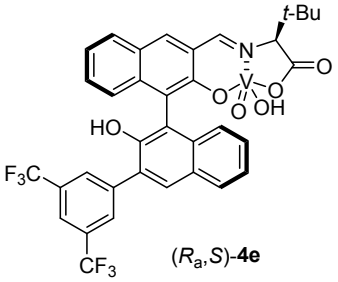




| entry | solvent | conv. (%) | yield (%)[a] | er (%)[b] | entry | solvent | conv. (%) | yield (%)[a] | er (%)[b] |
|---|---|---|---|---|---|---|---|---|---|
| 1 | $Et_2O$ | 93 | 41 | 83:17 | 6 | acetone | 71 | 13 | 73.5:26.5 |
| 2 | MeO*t*-Bu | 79 | 23 | 72.5:27.5 | 7 | toluene | 90 | 7 | 77:23 |
| 3 | CPME | 70 | 14 | 71:29 | 8 | MeCN | full | 8 | 75.5:24.5 |
| 4 | 1,4-dioxane | 76 | 8 | 66.5:33.5 | 9 | EtOAc | 54 | 9 | 66.5-33.5 |
| 5 | THF | 51 | 5 | 67:33 | 10 | $CH_2Cl_2$ | full | 19 | 82.5-17.5 |

[a]NMR yield using 1,3,5-trimethoxybenzene as an internal standard.
[b]Determined by HPLC

Table S3. Screening of ether-type reaction solvents

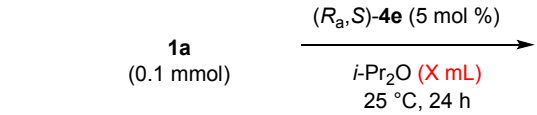



| entry | solvent | conv. (%) | yield (%)[a] | er (%)[b] | entry | solvent | conv. (%) | yield (%)[a] | er (%)[b] |
|---|---|---|---|---|---|---|---|---|---|
| 1[c] | $Et_2O$ | 93 (80) | 41 (30) | 83:17 (79.5:20.5) | 8 | MeO*t*-Bu | 79 | 23 | 72.5:27.5 |
| 2 | *n*-$Pr_2O$ | 79 | 25 | 80.5:19.5 | 9 | CPME | 70 | 14 | 71:29 |
| 3[c] | *i*-$Pr_2O$ | 95 (89) | 37 (32) | 83.5:16.5 (84:16) | 10 | THF | 51 | 5 | 67:33 |
| 4 | *n*-$Bu_2O$ | 82 | 14 | 75.5:24.5 | 11 | 1,4-dioxane | 76 | 8 | 66.5:33.5 |
| 5 | *n*-$Oct_2O$ | – | – | 73.5:26.5 | 12 | $MeOCH_2CH_2OMe$ | 50 | 6 | 63.5:36.5 |
| 6 | $Bn_2O$ | – | – | 66:34 | 13 | $(MeOCH_2CH_2)_2O$ | – | trace | – |
| 7 | $(ClCH_2CH_2)_2O$ | complex mixture | | | | | | | |

[a]NMR yield using 1,3,5-trimethoxybenzene as an internal standard.
[b]Determined by HPLC
[c]Results of 2nd trial in the parentheses.

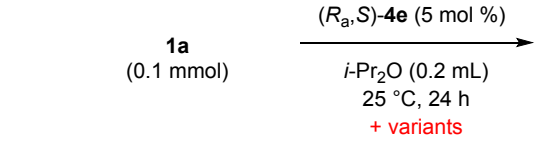
Table S4. Screening of concentration




| entry | *i*-$Pr_2O$ | conv. (%) | yield (%)[a] | er (%)[b] |
|---|---|---|---|---|
| 1 | 0.5 mL (0.2 M) | 95 | 37 | 83.5:16.5 |
| 2 | 0.4 mL (0.25 M) | 92 | 33 | 87.5:12.5 |
| 3 | 0.3 mL (0.33 M) | 97 | 31 | 88::12 |
| 4 | 0.2 mL (0.5 M) | full | 34 | 89.5:10.5 |

[a]NMR yield using 1,3,5-trimethoxybenzene as an internal standard.
[b]Determined by HPLC

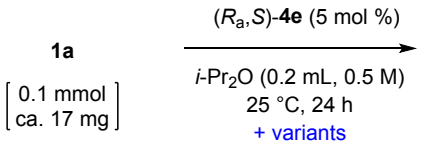
Table S5. Screening of reaction conditions I




| entry | variants | conv. (%) | yield (%)[a] | er (%)[b] |
|---|---|---|---|---|
| 1 | none | 95 | 34 | 89.5:10.5 |
| 2 | + $H_2O$ (0.1 mL, ca. 56 equiv) | 95 | 43 | 87.5:12.5 |
| 3 | + $H_2O$ (0.05 mL, ca. 28 equiv) | 90 | 53 | 87.5:12.5 |
| 4 | ($R_a$,*S*)-**4e** (10 mol %) | full | 38 | 89:11 |
| 5 | $Na_2SO_4$•$10H_2O$ (0.6 equiv) | full | 42 | 92:8 |
| 6 | NaOAc•$3H_2O$ (2.0 equiv) | 37 | 8 | 77:23 |
| 7 | unactivated MS3A (20 mg) | 77 | 33 | 83.5:16.5 |

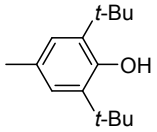
[a]NMR yield using 1,3,5-trimethoxybenzene as an internal standard.
[b]Determined by HPLC

Table S6. Screening of reaction conditions II



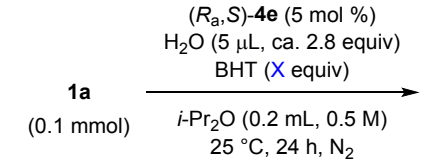

| entry | variants | conv. (%) | yield (%)[a] | er (%)[b] |
|---|---|---|---|---|
| 1 | none | 95 | 34 | 89.5:10.5 |
| 2 | + $H_2O$ (100 μL, ca. 56 equiv) | 95 | 43 | 87.5:12.5 |
| 3a | + $H_2O$ (50 μL, ca. 28 equiv) | 90 | 53 | 87.5:12.5 |
| 3b | + $H_2O$ (50 μL, ca. 28 equiv) | 93 | 64 | 88.5:11.5 |
| 4 | + $H_2O$ (25 μL, ca. 14 equiv) | 95 | 58 | 90.5 9.5 |
| 5 | + $H_2O$ (5 μL, ca. 2.8 equiv) | 98 | 60 | 91.5:8.5 |
| 6 | + $H_2O$ (5 μL, ca. 2.8 equiv)<br>2,6-di-*t*-Bu-*p*-cresol (0.5 equiv) | full | 78 (75)[c] | 91.5:8.5 |
| 7 | + 2,6-di-*t*-Bu-*p*-cresol (0.5 equiv) | full | 56 | 90:10 |
| 8 | + $Na_2SO_4$•10$H_2O$ (0.6 equiv) | full | 42 | 92:8 |
| 9 | + $Na_2SO_4$•10$H_2O$ (0.4 equiv) | full | 41 | 91.5:8.5 |
| 10 | + $Na_2SO_4$•10$H_2O$ (0.2 equiv) | full | 37 | 91.5:8.5 |

[a]NMR yield using 1,3,5-trimethoxybenzene as an internal standard.
[b]Determined by HPLC
[c]Isolated yield



2,6-di-*t*-Bu-*p*-cresol

Table S7. Screening of the amount of BHT

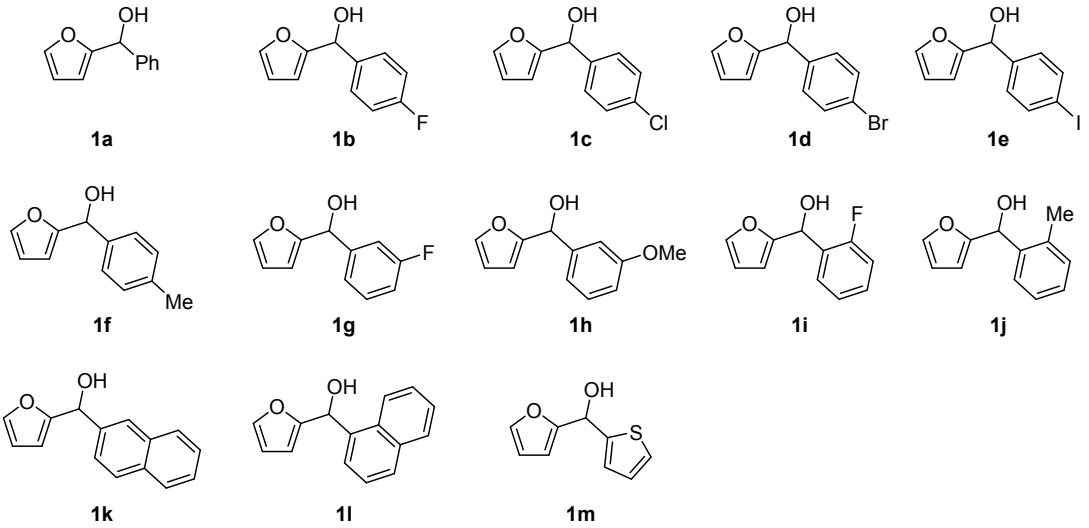



| entry | BHT (X eq) | yield (%)[a] | ee (%)[b] |
|---|---|---|---|
| 1 | 0.1 | 52 | 91.5:8.5 |
| 2 | 0.5 | 78 | 91.5:8.5 |
| 3 | 1.0 | 84[c] | 93:7 |
| 4 | 2.0 | 62 | 91.5:8.5 |

[a]NMR yield using 1,3,5-trimethoxybenzene
[b]IE, H/IPA = 7/1, 1.0 mL/min, 210 nm, $t_R$ = 13, 15 min
[c]Isolated yield, 48 h

## 3. Experimental procedures

### 3-1. Preparation of starting materials




Compounds **1a-d** were prepared according to the literature procedure.[1)]

Compound **1e** was prepared according to the literature procedure.[2)]

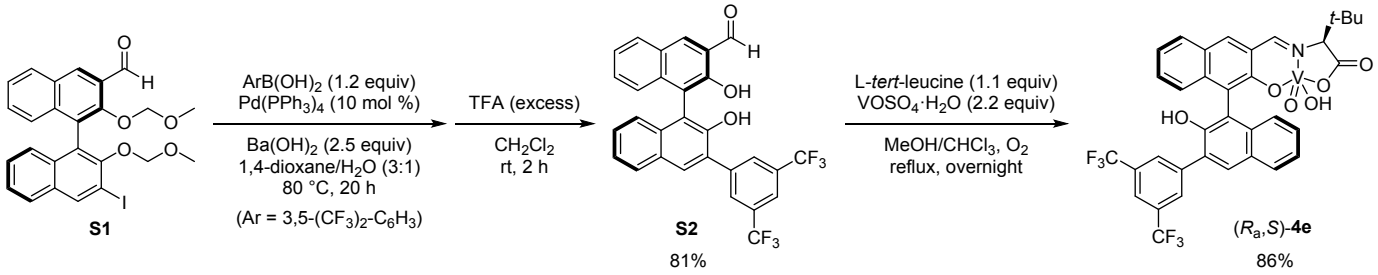
Compounds **1f-l** were prepared according to the literature procedure.[3)]

Compound **1m** was prepared according to the literature procedure.[4)]

### 3-2. Preparation of vanadium complexes

Dinuclear and mononuclear vanadium complexes were prepared according to literature.[5,6)]

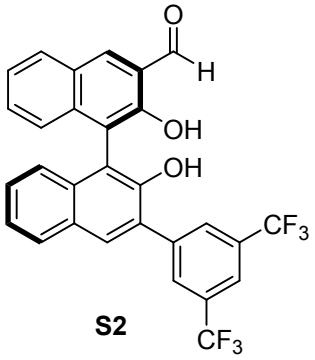
**Preparation of ($R_a$,*S*)-4e**




(*R*)-3'-Iodo-2,2'-bis(methoxymethoxy)-[1,1'-binaphthalene]-3-carbaldehyde[4)] (**S1**) (1.05 g, 1.99 mmol, 1 eq.), 3,5-bis(trifluoromethyl)phenylboronic acid (622 mg, 2.41 mmol, 1.2 eq.), barium hydroxide (1.59 g, 5.05 mmol, 2.5 eq.) and tetrakis(triphenylphosphine)palladium(0) (232 mg, 0.201 mmol, 10 mol%) were dissolved in dioxane/water (3:1, 20 mL) and stirred under inert gas at 80 °C for 20 hours. The reaction was cooled to rt and filtered through $Na_2SO_4$ and silica. The solvent was removed and the crude product was purified *via* automated MPLC (gradient of ethyl acetate in cyclohexane 2% (3 CV), 2-20% (10 CV), 20% (2 CV) to give (*R*)-3'-(3,5-bis(trifluoromethyl)phenyl)-2,2'-bis(methoxymethoxy)-[1,1'-binaphthalene]-3-carbaldehyde (968 mg, 1.84 mmol, 92%) as a yellow solid. 3'-(3,5-Bis(trifluoromethyl)phenyl)-2,2'-bis(methoxymethoxy)-[1,1'-binaphthalene]-3-carbaldehyde (968 mg, 1.575 mmol, 1 eq.) and trifluoroacetic acid (1.7 mL, 22 mmol, 14 eq.) were stirred in $CH_2Cl_2$ (20 mL) at rt for 2 hours. Then, sat. $NaHCO_3$ aq. was added until no more gas evolves and the phases were separated. The organic phase was washed with sat. $NaHCO_3$ aq. again and the aqueous phase was extracted once with $CH_2Cl_2$. The combined organic

phases were washed with water, brine, dried over $NaSO_4$ and filtrated. The solvent was removed and the crude product was purified *via* automated MPLC (gradient of ethyl acetate in cyclohexane 5% (3 CV), 5-40% (12 CV), 40% (2 CV) to give **S2** (727 mg, 1.38 mmol, 88%) as a yellow solid. Compound **S2** (262 mg, 0.499 mmol, 1 eq.), L-*tert*-leucine (74.2 mg, 0.571 mmol, 1.1 eq), $OVSO_4 \cdot H_2O$ (200 mg, 1.11 mmol, 2.2 eq.) and molecular sieves 3A (541 mg) were stirred under oxygen atmosphere in methanol (25 mL) at reflux conditions for 24 hours. The mixture was allowed to cool to rt filtered through celite and the solvent was removed under reduced pressure. The residue was dissolved in $CH_2Cl_2$, washed with water five times, washed with brine two times, dried over $Na_2SO_4$ and filtered. The solvent was removed under reduced pressure and the product was dried in vacuo. Vanadium complex ($R_a$,*S*)-**4e** (312 mg, 0.433 mmol, 86%) was obtained as a black solid.

Compound **S2**



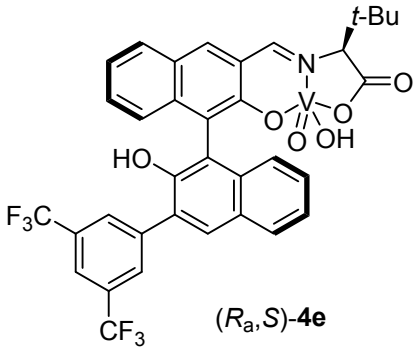

**S2**

**$^1$H-NMR** ($CDCl_3$, 600 MHz): $\delta$ 10.76 (s, 1H), 10.23 (s, 1H), 8.43 (s, 1H), 8.24 (m, 2H), 8.05 (dd, *J* = 2.18 and 7.13 Hz, 1H), 8.02 (s, 1H), 7.95 (d, *J* = 8.09 Hz, 1H), 7.88 (s, 1H), 7.49 (m, 2H), 7.40 (ddd, *J* = 1.21, 6.84 and 8.09 Hz, 1H), 7.28 (m, 2H), 7.08 (dd, *J* = 1.13 and 8.47 Hz, 1H), 5.30 (s, 1H), 5.19 (s, 1H).

**$^{13}$C-NMR** ($CDCl_3$, 125 MHz): $\delta$ 196.7, 154.7, 148.6, 140.3, 139.7, 137.7, 133.6, 131.7, 131.6, 131.4, 131.2, 130.4, 130.1, 129.3, 128.8, 128.0, 127.7, 127.0, 125.3, 124.9, 124.6, 124.5, 122.6, 122.29, 114.8, 114.33.

**HRMS** (ESI, negative ions): calculated for $C_{29}H_{15}O_3F_6$: *m*/*z* 525.09309 [M-H]$^-$, found 525.0931.

Vanadium complex ($R_a$,*S*)-**4e**




($R_a$,*S*)-**4e**

**$^1$H-NMR** ($CD_3OD$, 600 MHz): $\delta$ 8.97 (s, 1H), 8.53 (s, 1H), 8.32 (s, 2H), 8.10 (s, 1H), 8.08 (m, 1H), 8.02 (d, *J* = 8.19 Hz, 1H), 7.96 (s, 1H), 7.42 (m, 3H), 7.29 (m, 3H), 4.28 (s, 1H), 1.28 (s, 9H).

**$^{13}$C-NMR** ($CD_3OD$, 125 MHz): $\delta$ 168.5, 156.4, 143.0, 139.1, 139.0, 135.4, 132.8, 132.6, 132.3, 132.0, 131.6, 131.2, 131.0, 130.5, 129.9, 129.8, 129.6, 128.2, 126.3,126.1, 125.4, 125.0, 124.3, 123.9, 118.8, 116.9, 84.3, 34.5, 28.2.

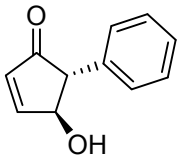
**$^{19}$F-NMR** ($CD_3OD$, 565 MHz): $\delta$ -64.11.

**HRMS** (ESI, negative ions): calculated for $C_{35}H_{25}NO_6F_6V$: *m*/z 720.1031 [M-H]$^-$, found 720.1032

**3-3. Vanadium-catalyzed catalytic and enantioselective *oxa*-Piancatelli reaction**

Furfuryl alcohol **1** (0.10 mmol, 17.42 mg) was weighed in a dry test tube and the test tube was equipped with a stir bar, evacuated and backfilled with $N_2$. To the test tube, *i*-$Pr_2O$ (0.2 mL), BHT (1.0 eq., 22.04 mg), and $H_2O$ (5 μL) were added. Then vanadium catalyst ($R_a$,*S*)-**4e** (10 mol %, 3.61 mg) was added at 20 °C. After stirring at 20 °C for 48 hours, the reaction mixture was applied to a short-pad silica column, flushed with Hexane/EtOAc (1/1) mixed solvent, and the solvent was evaporated. The crude mixture was subjected to silica gel flash chromatography to give the pure product.

*trans*-4-hydroxy-5-phenylcyclopent-2-en-1-one (**2a**)




15.0 mg. 84% yield. yellow oil.

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.64 (dd, $J$ = 6.0, 2.3 Hz, 1H), 7.28-7.38 (m, 3H), 7.13-7.15 (m, 2H), 6.36 (dd, $J$ = 6.0, 1.4 Hz, 1H), 5.00 (s, 1H), 3.49 (d, $J$ = 2.8 Hz, 1H), 2.36 (bs, 1H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 205.5, 161.8, 136.7, 134.4, 128.9, 128.3, 127.4, 78.9, 62.1.

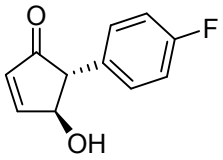
**HRMS** (ESI): calcd for $C_{11}H_{10}O_2Na$: *m/z* 197.0578 $[M + Na]^+$, found 197.0568.

**IR** (KBr): 3400, 3062, 1702, 1496, 1336, 1163, 1036, 917, 750 $cm^{-1}$.

**Enantiomeric ratio**: 93:7 er, determined by HPLC (CHIRALPAK IE, hexane/2-popanol = 7/1; flow rate 1.0 mL/min; 25 °C; 210 nm) first peak: $t_R$ = 15.5 min, second peak: $t_R$ = 18.2 min.

$[\alpha]_D^{21}$ = +107.7 (*c* 0.75, $CHCl_3$) for 93:7 er.

*trans*-5-(4-fluorophenyl)-4-hydroxycyclopent-2-en-1-one (**2b**)




16.3 mg. 87% yield. yellow oil.

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.62 (dd, $J$ = 5.8, 2.3 Hz, 1H), 7.01-7.14 (m, 4H), 6.34 (dd, $J$ = 5.8, 1.4 Hz, 1H), 4.94-4.96 (m, 1H), 3.44 (d, $J$ = 3.2 Hz, 1H), 2.52 (bs, 1H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 205.1, 162.1 (d, $^1J_{C\text{-}F}$ = 246.3 Hz), 161.7, 134.3, 132.4 (d, $^4J_{C\text{-}F}$ = 2.9 Hz), 129.9 (d, $^3J_{C\text{-}F}$ = 7.7 Hz), 115.8 (d, $^2J_{C\text{-}F}$ = 22.0 Hz), 78.9, 61.3.

**$^{19}$F NMR** (565 MHz, $CDCl_3$): $\delta$ -115.8.

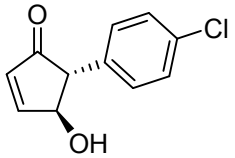
**HRMS** (ESI): calcd for $C_{11}H_9FO_2Na$: *m/z* 215.0484 $[M + Na]^+$, found 215.0473.

**IR** (KBr): 3388, 3070, 2907, 1702, 1512, 1340, 1227, 1037, 825 $cm^{-1}$.

**Enantiomeric ratio**: 90:10 er, determined by HPLC (CHIRALPAK IF, hexane/2-popanol = 15/1; flow rate 1.0 mL/min; 25 °C; 210 nm) first peak: $t_R$ = 12.0 min, second peak: $t_R$ = 13.4 min.

$[\alpha]_D^{22}$ = +84.4 (*c* 0.82, $CHCl_3$) for 90:10 er.

*trans*-5-(4-chlorophenyl)-4-hydroxycyclopent-2-en-1-one (**2c**)




15.2 mg. 73% yield. yellow oil.

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.64 (dd, $J$ = 5.6, 2.3 Hz, 1H), 7.32-7.35 (m, 2H), 7.08-7.11 (m, 2H), 6.36 (dd, $J$ = 5.6, 1.4 Hz, 1H), 4.96-4.98 (m, 1H), 3.45 (d, $J$ = 3.2 Hz, 1H), 2.38 (bs, 1H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 204.8, 161.8, 135.1, 134.3, 133.4, 129.6, 129.1, 78.7, 61.3.

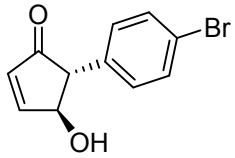
**HRMS** (ESI): calcd for $C_{11}H_9ClO_2Na$: *m/z* 231.0189 $[M + Na]^+$, found 231.0179.

**IR** (KBr): 3494, 3021, 2890, 1692, 1493, 1335, 1160, 1039, 813 $cm^{-1}$.

**Enantiomeric ratio**: 90:10 er, determined by HPLC (CHIRALPAK IE, hexane/2-popanol = 20/1; flow rate 1.0 mL/min; 25 °C; 210 nm) first peak: $t_R$ = 31.5 min, second peak: $t_R$ = 33.9 min.

$[\alpha]_D^{22}$ = +83.6 ($c$ 0.76, $CHCl_3$) for 90:10 er.

*trans*-5-(4-bromophenyl)-4-hydroxycyclopent-2-en-1-one (**2d**)




19.1 mg. 75% yield. yellow oil.

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.63 (dd, $J$ = 5.7, 2.3 Hz, 1H), 7.47-7.50 (m, 2H), 7.02-7.04 (m, 2H), 6.35 (dd, $J$ = 5.7, 1.4 Hz, 1H), 4.97 (s, 1H), 3.43 (d, $J$ = 3.2 Hz, 1H), 2.35 (bs, 1H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 204.7, 161.8, 135.6, 134.3, 132.0, 123.00, 121.5, 78.7, 61.4.

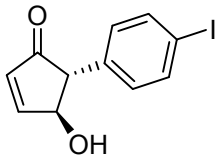
**HRMS** (ESI): calcd for $C_{11}H_9BrO_2Na$: *m/z* 274.9684 $[M + Na]^+$, found 274.9673.

**IR** (KBr): 3401, 3064, 2903, 1702, 1488, 1337, 1104, 1034, 812 $cm^{-1}$.

**Enantiomeric ratio**: 90:10 er, determined by HPLC (CHIRALPAK IE, hexane/2-popanol = 15/1; flow rate 1.0 mL/min; 25 °C; 210 nm) first peak: $t_R$ = 14.1 min, second peak: $t_R$ = 15.5 min.

$[\alpha]_D^{22}$ = +82.6 ($c$ 0.96, $CHCl_3$) for 90:10 er.

*trans*-4-hydroxy-5-(4-iodophenyl)cyclopent-2-en-1-one (**2e**)




19.3 mg. 65% yield. yellow oil.

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.67-7.70 (m, 2H), 7.64 (dd, $J$ = 6.0, 2.3 Hz, 1H), 6.89-6.92 (m, 2H), 6.36 (dd, $J$ = 5.5, 1.4 Hz, 1H), 4.98 (s, 1H), 3.42 (d, $J$ = 2.8 Hz, 1H), 2.31 (bs, 1H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 204.7, 161.8, 138.0, 136.3, 134.3, 130.3, 93.0, 78.6, 61.5.

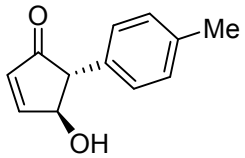
**HRMS** (ESI): calcd for $C_{11}H_9IO_2Na$: *m/z* 322.9545 $[M + Na]^+$, found 322.9537.

**IR** (KBr): 3470, 3076, 2910, 1685, 1588, 1487, 1326, 1041, 809 $cm^{-1}$.

**Enantiomeric ratio**: 91:9 er, determined by HPLC (CHIRALPAK IF-3, hexane/2-popanol = 9/1; flow rate 1.0 mL/min; 25 °C; 210 nm) first peak: $t_R$ = 7.7 min, second peak: $t_R$ = 8.8 min.

$[\alpha]_D^{25}$ = +84.97 ($c$ 0.97, $CHCl_3$) for 91:9 er.

*trans*-4-hydroxy-5-(*p*-tolyl)cyclopent-2-en-1-one (**2f**)




10.9 mg. 57% yield. yellow oil.

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.61 (dd, $J$ = 6.0, 2.3 Hz, 1H), 7.16 (d, $J$ = 8.0 Hz, 2H), 7.02 (d, $J$ = 8.0 Hz, 2H), 6.33 (dd, $J$ = 6.0, 1.4 Hz, 1H), 4.97 (s, 1H), 3.40 (d, $J$ = 2.8 Hz, 1H), 2.41 (bs, 1H), 2.34 (s, 3H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 205.6, 161.6, 137.1, 134.4, 133.6, 129.6, 128.2, 79.0, 61.8, 21.1.

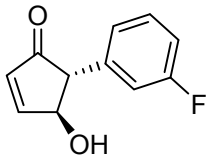
**HRMS** (ESI): calcd for $C_{12}H_{12}O_2Na$: *m/z* 211.0735 $[M + Na]^+$, found 211.0725.

**IR** (KBr): 3398, 3019, 2920, 1702, 1514, 1337, 1034, 804 $cm^{-1}$.

**Enantiomeric ratio**: 93:7 er, determined by HPLC (CHIRALPAK IE, hexane/2-popanol = 15/1; flow rate 1.0 mL/min; 25 °C; 210 nm) first peak: $t_R$ = 37.3 min, second peak: $t_R$ = 44.5 min.

$[\alpha]_D^{22}$ = +118.5 ($c$ 0.55, $CHCl_3$) for 93:7 er.

*trans*-5-(3-fluorophenyl)-4-hydroxycyclopent-2-en-1-one (**2g**)




11.9 mg. 62% yield. yellow oil.

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.62 (dd, $J$ = 6.0, 2.3 Hz, 1H), 7.29-7.34 (m, 1H), 6.97-7.02 (m, 1H), 6.93 (d, $J$ = 7.8 Hz, 1H), 6.83-6.87 (m, 1H), 6.34 (dd, $J$ = 6.0, 1.4 Hz, 1H), 4.97 (s, 1H), 3.45 (d, $J$ = 3.2 Hz, 1H), 2.58 (bs, 1H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 204.5, 162.9 (d, $^1J_{C\text{-}F}$ = 250.1 Hz), 161.7, 139.0 (d, $^3J_{C\text{-}F}$ = 7.7 Hz), 134.4, 130.4 (d, $^3J_{C\text{-}F}$ = 8.6 Hz), 124.0 (d, $^4J_{C\text{-}F}$ = 2.9 Hz), 115.3 (d, $^2J_{C\text{-}F}$ = 21.1 Hz), 114.5 (d, $^2J_{C\text{-}F}$ = 21.1 Hz), 78.7, 61.6.

**$^{19}$F NMR** (565 MHz, $CDCl_3$): $\delta$ -113.3.

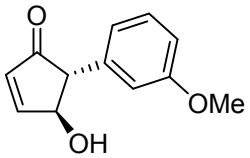
**HRMS** (ESI): calcd for $C_{11}H_9FO_2Na$: *m/z* 215.0484 $[M + Na]^+$, found 215.0474.

**IR** (KBr): 3416, 3080, 2909, 1704, 1514, 1252, 1034, 821 $cm^{-1}$.

**Enantiomeric ratio**: 91:9 er, determined by HPLC (CHIRALPAK IE, hexane/2-popanol = 9/1; flow rate 1.0 mL/min; 25 °C; 210 nm) first peak: $t_R$ = 13.9 min, second peak: $t_R$ = 17.1 min.

$[\alpha]_D^{22}$ = +63.4 ($c$ 0.60, $CHCl_3$) for 91:9 er.

*trans*-4-hydroxy-5-(3-methoxyphenyl)cyclopent-2-en-1-one (**2h**)




18.2 mg. 90% yield. yellow oil.

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.63 (dd, $J$ = 5.7, 2.3 Hz, 1H), 7.27 (t, $J$ = 7.8 Hz, 1H), 6.84 (dd, $J$ = 7.8, 2.3 Hz, 1H), 6.71 (d, $J$ = 7.8 Hz, 1H), 6.68 (d, $J$ = 2.3 Hz, 1H), 6.34 (dd, $J$ = 5.7, 1.4 Hz, 1H), 5.00 (s, 1H), 3.79 (s, 3H), 3.42 (d, $J$ = 2.8 Hz, 1H), 2.43 (bs, 1H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 205.3, 161.8, 159.9, 138.2, 134.4, 130.0, 120.5, 114.2, 112.7, 78.9, 62.0, 55.2.

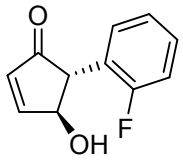
**HRMS** (ESI): calcd for $C_{12}H_{12}O_3Na$: *m/z* 227.0684 $[M + Na]^+$, found 227.0675.

**IR** (KBr): 3399, 2940, 2838, 1704, 1588, 1492, 1260, 1041, 781 $cm^{-1}$.

**Enantiomeric ratio**: 89:11 er, determined by HPLC (CHIRALPAK IF-3, hexane/2-popanol = 9/1; flow rate 1.0 mL/min; 25 °C; 210 nm) first peak: $t_R$ = 25.4 min, second peak: $t_R$ = 44.2 min.

$[\alpha]_D^{22}$ = +78.2 ($c$ 0.91, $CHCl_3$) for 89:11 er.

*trans*-5-(2-fluorophenyl)-4-hydroxycyclopent-2-en-1-one (**2i**)




8.4 mg. 45% yield. yellow oil.

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.60 (dd, $J$ = 5.6, 2.3 Hz, 1H), 7.26-7.32 (m, 1H), 7.05-7.19 (m, 3H), 6.37 (d, $J$ = 5.6 Hz, 1H), 5.04 (s, 1H), 3.56 (d, $J$ = 2.8 Hz, 1H), 2.42 (bd, $J$ = 6.0 Hz, 1H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 204.2, 161.2, 161.0 (d, $^1J_{C\text{-}F}$ = 245.4 Hz), 134.0, 131.2 (d, $^3J_{C\text{-}F}$ = 3.8 Hz), 129.4 (d, $^3J_{C\text{-}F}$ = 8.6 Hz), 124.5 (d, $^4J_{C\text{-}F}$ = 2.9 Hz), 124.1 (d, $^2J_{C\text{-}F}$ = 15.3 Hz), 115.7 (d, $^2J_{C\text{-}F}$ = 21.1 Hz), 78.0, 57.9.

**$^{19}$F NMR** (565 MHz, $CDCl_3$): $\delta$ -117.0.

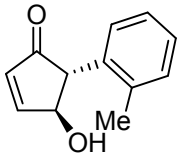
**HRMS** (ESI): calcd for $C_{11}H_9FO_2Na$: *m/z* 215.0484 $[M + Na]^+$, found 215.0474.

**IR** (KBr): 3399, 3068, 2910, 1705, 1494, 1233, 1103, 759 $cm^{-1}$.

**Enantiomeric ratio**: 75:25 er, determined by HPLC (CHIRALPAK IE, hexane/2-popanol = 9/1; flow rate 1.0 mL/min; 25 °C; 210 nm) first peak: $t_R$ = 13.9 min, second peak: $t_R$ = 17.1 min.

$[\alpha]_D^{22}$ = +63.4 (*c* 0.60, $CHCl_3$) for 75:25 er.

*trans*-4-hydroxy-5-(*o*-tolyl)cyclopent-2-en-1-one (**2j**)




10.0 mg. 52% yield. yellow oil.

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.57 (dd, $J$ = 6.0, 1.8 Hz, 1H), 7.12-7.20 (m, 3H), 6.86 (d, $J$ = 6.9 Hz, 1H), 6.32 (d, $J$ = 5.5 Hz, 1H), 4.92 (s, 1H), 3.65 (d, $J$ = 2.8 Hz, 1H), 2.68 (bs, 1H), 2.29 (s, 3H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 206.0, 161.7, 137.0, 135.5, 134.5, 130.8, 128.2, 127.5, 126.4, 78.9, 59.7, 20.0.

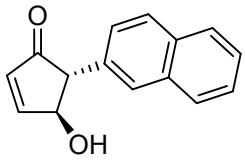
**HRMS** (ESI): calcd for $C_{12}H_{12}O_2Na$: *m/z* 211.0735 $[M + Na]^+$, found 211.0726.

**IR** (KBr): 3398, 3063, 2924, 1702, 1494, 1339, 1164, 1032, 753 $cm^{-1}$.

**Enantiomeric ratio**: 72:28 er, determined by HPLC (CHIRALPAK IF-3, hexane/2-popanol = 9/1; flow rate 1.0 mL/min; 25 °C; 210 nm) first peak: $t_R$ = 10.2 min, second peak: $t_R$ = 11.8 min.

$[\alpha]_D^{19}$ = +40.6 (*c* 0.50, $CHCl_3$) for 72:28 er.

*trans*-4-hydroxy-5-(naphthalen-2-yl)cyclopent-2-en-1-one (**2k**)



14.3 mg. 62% yield. yellow oil.

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.79-7.85 (m, 3H), 7.67-7.69 (m, 2H), 7.45-7.50 (m, 2H), 7.19 (dd, $J$ = 8.7, 1.8 Hz, 1H), 6.41 (dd, $J$ = 5.5, 1.4 Hz, 1H), 5.10 (s, 1H), 3.63 (d, $J$ = 2.8 Hz, 1H), 2.37 (bs, 1H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 205.4, 161.8, 134.5, 134.0, 133.4, 132.6, 128.8, 127.7, 126.36, 126.0, 125.7, 78.9, 62.2. (Two carbons are overlapping.)

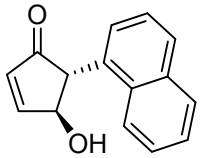
**HRMS** (ESI): calcd for $C_{15}H_{12}O_2Na$: *m/z* 247.0735 $[M + Na]^+$, found 247.0726.

**IR** (KBr): 3402, 3055, 2917, 1704, 1508, 1337, 1034, 816, 750 $cm^{-1}$.

**Enantiomeric ratio**: 89:11 er, determined by HPLC (CHIRALPAK IF-3, hexane/2-popanol = 9/1; flow rate 1.0 mL/min; 25 °C; 210 nm) first peak: $t_R$ = 17.1 min, second peak: $t_R$ = 20.8 min.

$[\alpha]_D^{18}$ = +113.4 (*c* 0.72, $CHCl_3$) for 89:11 er.

*trans*-4-hydroxy-5-(naphthalen-1-yl)cyclopent-2-en-1-one (**2l**)



13.2 mg. 55% yield. yellow oil.

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.87-7.91 (m, 1H), 7.81 (d, $J$ = 8.2 Hz, 1H), 7.66-7.69 (m, 2H), 7.46-7.52 (m, 2H), 7.43 (dd, $J$ = 8.2, 7.3 Hz,1H), 7.23 (d, $J$ = 6.9 Hz, 1H), 6.49 (dd, $J$ = 6.0, 1.4 Hz, 1H), 5.14 (s, 1H), 4.07 (d, $J$ = 2.3 Hz, 1H), 2.41 (d, $J$ = 2.3 Hz, 1H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 205.9, 161.2, 134.7, 134.2, 133.2, 131.8, 129.1, 128.3, 127.2, 126.5, 125.9, 125.5, 123.4, 78.7, 60.4.

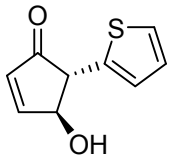
**HRMS** (ESI): calcd for $C_{15}H_{12}O_2Na$: *m/z* 247.0735 $[M + Na]^+$, found 247.0728.

**IR** (KBr): 3410, 3059, 2916, 1706, 1594, 1336, 1161, 1028, 776 $cm^{-1}$.

**Enantiomeric ratio**: 71:29 er, determined by HPLC (CHIRALPAK IH, hexane/2-popanol = 7/1; flow rate 1.0 mL/min; 25 °C; 210 nm) first peak: $t_R$ = 24.9 min, second peak: $t_R$ = 36.3 min.

$[\alpha]_D^{23}$ = –19.4 (*c* 0.66, $CHCl_3$) for 71:29 er.

*trans*-4-hydroxy-5-(thiophen-2-yl)cyclopent-2-en-1-one (**2m**)




14.4 mg. 71% yield. yellow oil.

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.58 (dd, $J$ = 6.0, 2.3 Hz, 1H), 7.25 (dd, $J$ = 5.5, 1.4 Hz, 1H), 6.98-7.02 (m, 2H), 6.31 (dd, $J$ = 6.0, 1.4 Hz, 1H), 5.05 (s, 1H), 3.74 (d, $J$ = 2.8 Hz, 1H), 2.75 (bs, 1H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 203.0, 161.2, 137.5, 133.6, 127.1, 126.0, 124.9, 78.8, 56.9.

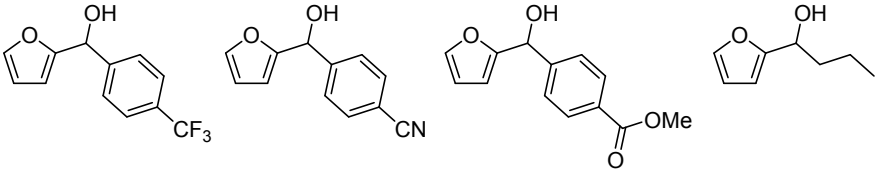
**HRMS** (ESI): calcd for $C_9H_8O_2SNa$: *m/z* 203.0143 $[M + Na]^+$, found 203.0135.

**IR** (KBr): 3410, 3108, 2880, 1707, 1436, 1338, 1190, 1113, 704 $cm^{-1}$.

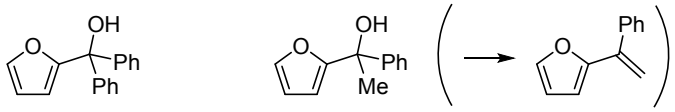
**Enantiomeric ratio**: 88:12 er, determined by HPLC (CHIRALPAK IF-3, hexane/2-popanol = 4/1; flow rate 1.0 mL/min; 25 °C; 210 nm) first peak: $t_R$ = 7.7 min, second peak: $t_R$ = 9.1 min.

$[\alpha]_D^{24}$ = +93.3 (*c* 0.72, $CHCl_3$) for 88:12 er.

*Unsuccessful examples of starting materials*

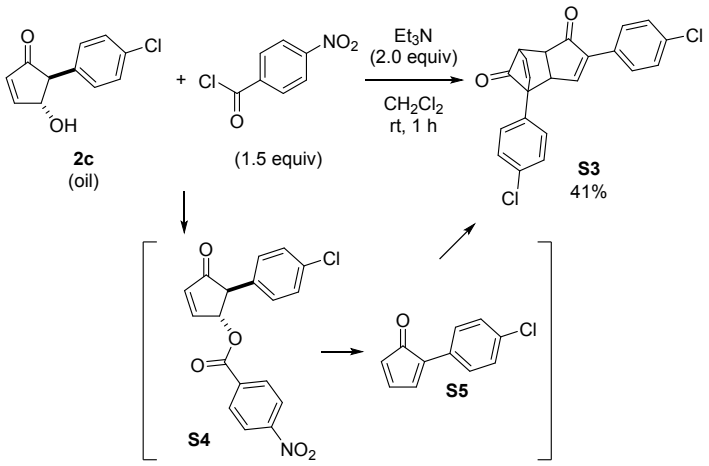



No desired product and decomposition of starting materials were observed.




complex mixture dehydration occurred

**3-4. Derivatization**




Compound **S3**.

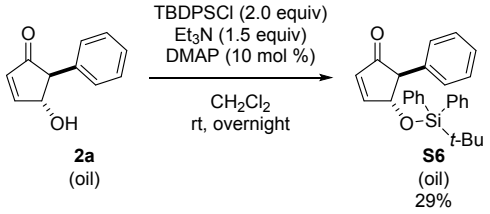
To a solution of **2c** (37.5 mg, 0.18 mmol) and $Et_3N$ (50 μL mg, 0.36 mmol) in $CH_2Cl_2$ (0.9 mL), 4-Nitrobenzoyl Chloride (50 mg, 0.27 mmol) is added at 0 °C. The reaction mixture is then stirred for 1 hour at rt. This mixture was treated with sat. $NH_4Cl$ aq. and extracted with $CH_2Cl_2$. The combined organic phases are washed with sat. NaCl aq. and dried over $Na_2SO_4$, and the solvent was evaporated off. The residue obtained was purified by silica-gel column chromatography to afford **S3** (13.9 mg, 41%, white solid).

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.59-7.63 (m, 2H), 7.56 (d, $J$ = 2.8 Hz, 1H), 7.43-7.46 (m, 2H), 7.32-7.39 (m, 4H), 6.48 (dd, $J$ = 6.9, 3.2 Hz, 1H), 6.34 (d, $J$ = 6.9 Hz, 1H), 3.73-3.78 (m, 2H), 3.33 (t, $J$ = 5.7 Hz, 1H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 203.5, 198.3, 154.2, 149.9, 135.2, 133.9, 132.7, 132.5, 129.4, 129.2, 129.0, 128.7, 128.3, 61.0, 51.6, 46.2, 43.7 (One carbon is overlapping).

**HRMS (ESI)**: calcd for $C_{22}H_{14}Cl_2O_2Na$: $m/z$ 403.0269 $[M + Na]^+$, found 403.0258.

**IR** (KBr): 3085, 2925, 1782, 1702, 1492, 1321, 1090, 823, 742 $cm^{-1}$.




Compound **S6**.

To a solution of **2a** (52.3 mg, 0.30 mmol), $Et_3N$ (62.7 μL mg, 0.45 mmol), and DMAP (3.7 mg, 0.03 mmol) in $CH_2Cl_2$ (1 mL), TBDPCl (156 μL, 0.6 mmol) is added at 0 °C. The reaction mixture is then stirred overnight at rt. This mixture was treated with sat. $NH_4Cl$ aq. and extracted with $CH_2Cl_2$. The combined organic phases are washed with sat. NaCl aq. and dried over $Na_2SO_4$, and the solvent was evaporated off. The residue obtained was purified by silica-gel column chromatography and GPC to afford **S6** (35.5 mg, 29%, colorless oil).

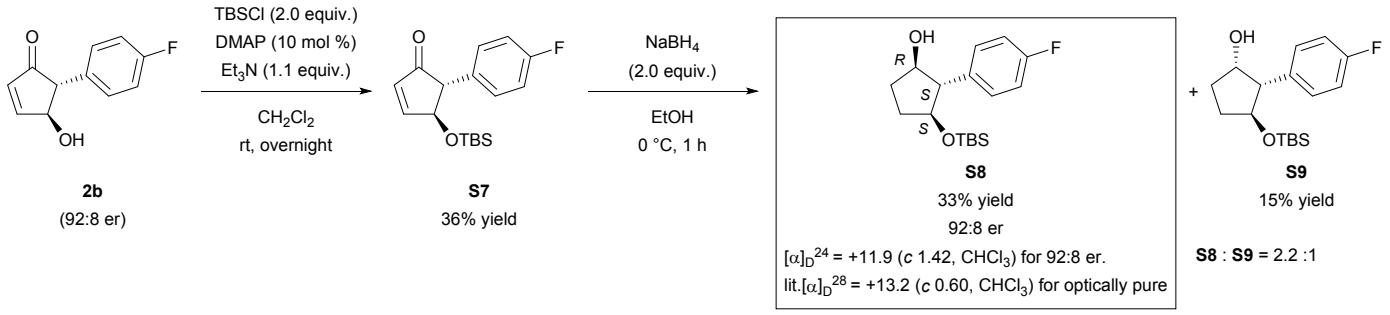
**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.59-7.61 (m, 2H), 7.32-7.45 (m, 6H), 7.29 (dd, $J$ = 6.0, 2.3 Hz, 1H), 7.24-7.26 (m, 3H), 7.18 (t, $J$ = 7.8 Hz, 2H), 6.91-6.93 (m, 2H), 6.21 (dd, $J$ = 6.0, 1.4 Hz, 1H), 4.95-4.96 (m, 1H), 3.59 (d, $J$ = 2.8 Hz, 1H), 1.03 (s, 9H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 205.6, 162.5, 136.9, 135.7, 133.7, 133.3, 132.3, 123.0, 129.8, 128.7, 128.5, 127.8, 127.6, 127.1, 80.1, 62.6, 26.7, 19.0. (One carbon is overlapping.)

**HRMS (ESI)**: calcd for $C_{27}H_{28}O_2SiNa$: *m/z* 435.1756 $[M + Na]^+$, found 435.1747.

**IR** (KBr): 3067, 3031, 2933, 2858, 1720, 1427, 1360, 1199, 1080, 820, 701 $cm^{-1}$.

The absolute configuration of synthesized compound **2b** was determined by comparison of the specific rotation with that of the reported compounds **S8** after TBS protection and reduction.




Compound **S7**.

To a solution of **2b** (153.8 mg, 0.80 mmol), $Et_3N$ (0.12 mL, 0.88 mmol), and DMAP (9.8 mg, 0.08 mmol) in $CH_2Cl_2$ (4 mL), TBSCl (241.2 mg, 1.6 mmol) was added at 0 °C. The reaction mixture was then stirred overnight at rt. This mixture was treated with sat. $NH_4Cl$ aq. and extracted with $CH_2Cl_2$. The combined organic phases are washed with sat. NaCl aq. and dried over $Na_2SO_4$, and the solvent was evaporated off. The residue obtained was purified by silica-gel column chromatography and GPC to afford **S6** (89.5 mg, 36%, colorless oil).

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.51 (dd, $J$ = 6.0, 2.3 Hz, 1H), 7.01-7.08 (m, 4H), 6.29 (dd, $J$ = 6.0, 2.3 Hz, 1H), 4.86-4.87 (m, 1H), 3.41 (d, J = 2.8 Hz, 1H), 0.86 (s, 9H), -0.03 (s, 3H), -0.09 (s, 3H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 205.1, 162.4, 162.0 (d, $^1J_{C\text{-}F}$ = 245.4 Hz), 133.5, 132.8 (d, $^4J_{C\text{-}F}$ = 2.9 Hz), 130.0 (d, $^3J_{C\text{-}F}$ = 7.7 Hz), 115.7 (d, $^2J_{C\text{-}F}$ = 21.1 Hz), 79.7, 62.1, 25.6, 17.9, -4.8, -5.0.

**HRMS** (ESI): calcd for $C_{17}H_{23}FO_2SiNa$: *m/z* 329.1349 $[M + Na]^+$, found 329.1340.

**IR** (KBr): 3055, 2932, 2858, 1721, 1511, 1227, 1076, 845, 778 $cm^{-1}$.

Preparation of compound **S8** and **S9**.

To a solution of **S6** (85.8 mg, 0.28 mmol) in EtOH (4.7 mL), $NaBH_4$ (22.2 mg, 0.56 mmol) was added at 0 °C. The reaction mixture was then stirred for 1 h at 0 °C. This mixture was treated with sat. $NH_4Cl$ aq. and extracted with $CH_2Cl_2$. The combined organic phases are washed with sat. NaCl aq. and dried over $Na_2SO_4$, and the solvent was evaporated off. The residue obtained was purified by PTLC to afford **S8** (28.4 mg, 33%, colorless oil) and **S9** (13 mg, 15%, colorless oil).

Compound **S8**

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.14-7.18 (m, 2H), 6.99-7.03 (m, 2H), 4.21-4.24 (m, 1H), 4.04-4.09 (m, 1H), 2.90 (t, $J$ = 7.2 Hz, 1H), 1.83-2.14 (m, 5H), 0.79 (s, 9H), -0.13 (s, 3H), -0.20 (s, 3H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 161.7 (d, $^1J_{C\text{-}F}$ = 244.4 Hz), 136.4 (d, $^4J_{C\text{-}F}$ = 2.9 Hz), 129.1 (d, $^3J_{C\text{-}F}$ = 8.6 Hz), 115.3 (d, $^2J_{C\text{-}F}$ = 21.1 Hz), 79.0, 77.2, 62.3, 32.3, 31.8, 25.7, 17.9, -4.99, -5.07.

**Enantiomeric ratio**: 92:8 er, determined by HPLC (CHIRALPAK IF-3, hexane/2-popanol = 99/1; flow rate 1.0 mL/min; 25 °C; 265 nm) first peak: $t_R$ = 16.6 min, second peak: $t_R$ = 17.9 min.

$[\alpha]_D^{24}$ = +11.9 ($c$ 1.42, $CHCl_3$) for 92:8 er.

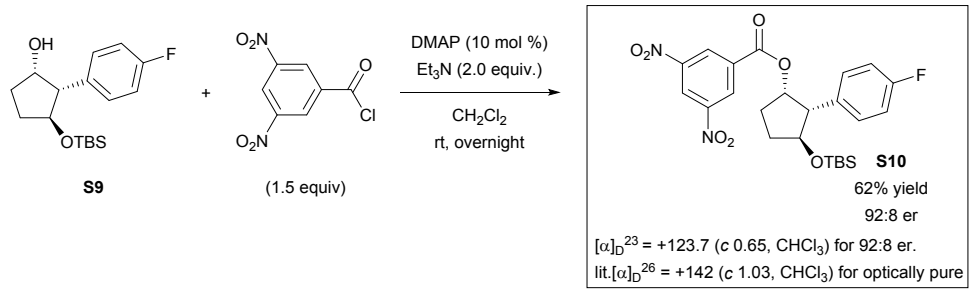
Reported data for (1*R*,2*S*,3*S*)-3-(*tert*-Butyldimethylsilyloxy)-2-(4-fluorophenyl)cyclopentan-1-ol (**S8**).[8)]
$[\alpha]_D^{28}$ = +13.2 (*c* 0.60, $CHCl_3$) (optically pure).

Compound **S9**

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 7.26-7.29 (m, 2H), 7.01-7.05 (m, 2H), 4.53-4.59 (m, 1H), 4.23-4.26 (m, 1H), 2.95 (dd, J = 8.7, 5.0 Hz, 1H), 2.21-2.29 (m, 2H), 1.73, 1.80 (m, 1H), 1.62-1.68 (m, 1H), 1.26 (s, 1H), 0.78 (s, 9H), -0.07 (s, 3H), -0.18 (m, 3H).

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 161.9 (d, $^1J_{C\text{-}F}$ = 244.4 Hz), 134.1 (d, $^4J_{C\text{-}F}$ = 2.9 Hz), 130.8 (d, $^3J_{C\text{-}F}$ = 8.6 Hz), 115.1 (d, $^2J_{C\text{-}F}$ = 21.1 Hz), 76.9, 74.2, 59.7, 32.6, 31.8, 25.7, 17.9, -4.6, -5.0.

The absolute configuration of synthesized compound (*S*,*S*)-**2b** was reconfirmed by comparison of the specific rotation with that of the other reported compound **S10** after acylation.




Compound **S10**.

To a solution of **S9** (13 mg, 0.042 mmol) and DMAP (0.5 mg, 0.0042 mmol) in $CH_2Cl_2$ (0.42 mL), 3,5-dinitrobenzoyl chloride (14.5 mg, 0.063 mmol) and $Et_3N$ (12 µL, 0.88 mmol) were added at 0 °C. The reaction mixture is then stirred overnight at rt. This mixture was treated with sat. $NH_4Cl$ aq. and extracted with $CH_2Cl_2$. The combined organic phases are washed with sat. NaCl aq. and dried over $Na_2SO_4$, and the solvent was evaporated off. The residue obtained was purified by PTLC to afford **S10** (13 mg, 62%, colorless oil).

**$^1$H NMR** (400 MHz, $CDCl_3$): $\delta$ 9.17 (t, *J* = 1.9 Hz, 1H), 8.89 (d, *J* = 1.9 Hz, 2H), 7.23-7.28 (m, 2H), 6.96-7.02 (m, 2H), 5.61 (td, *J* = 6.9, 3.7 Hz, 1H), 4.62-4.67 (m, 1H), 3.31 (t, *J* = 7.3 Hz, 1H), 2.49-2.58 (m, 1H), 2.24-2.32 (m, 1H), 1.94-2.05 (m, 1H), 1.73-1.82 (m, 1H), 0.79 (s, 9H), -0.02 (s, 3H), -0.12 (s, 3H)

**$^{13}$C NMR** (100 MHz, $CDCl_3$): $\delta$ 161.8 (d, $^1J_{C\text{-}F}$ = 246.3 Hz), 161.6, 148.5, 133.9, 133.1 (d, $^4J_{C\text{-}F}$ = 2.9 Hz), 130.7 (d, $^3J_{C\text{-}F}$ = 8.6 Hz), 129.1, 122.2, 115.1 (d, $^2J_{C\text{-}F}$ = 21.1 Hz), 78.7, 77.5, 57.6, 32.8, 30.2, 25.6, 17.9, -4.6, -4.9.

**Enantiomeric ratio**: 92:8 er, determined by HPLC (CHIRALPAK IF-3, hexane/2-popanol = 99/1; flow rate 1.0 mL/min; 25 °C; 220 nm) first peak: $t_R$ = 19.6 min, second peak: $t_R$ = 20.8 min.
$[\alpha]_D^{23}$ = + 123.7 (*c* 0.65, $CHCl_3$) for 92:8 er.

Reported data for (1*S*,2*S*,3*S*)-3-(*tert*-Butyldimethylsilyloxy)-2-(4-fluorophenyl)cyclopentyl 3,5-Dinitrobenzoate (S**10**).[8)]
$[\alpha]_D^{26}$ = +142 (*c* 1.03, $CHCl_3$) (optically pure).

**4. References**


1) Y. Chai, Y. Tang, I. Atodiresei, and M. Rueping, *Angew. Chem. Int. Ed.* **2016**, *55*, 14126.
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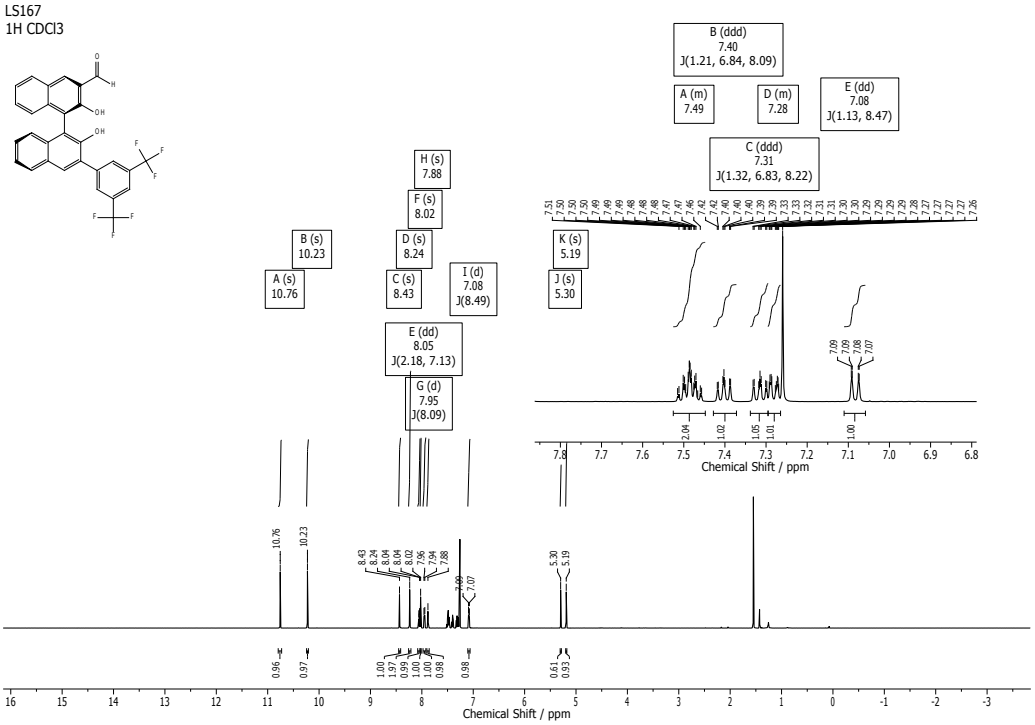
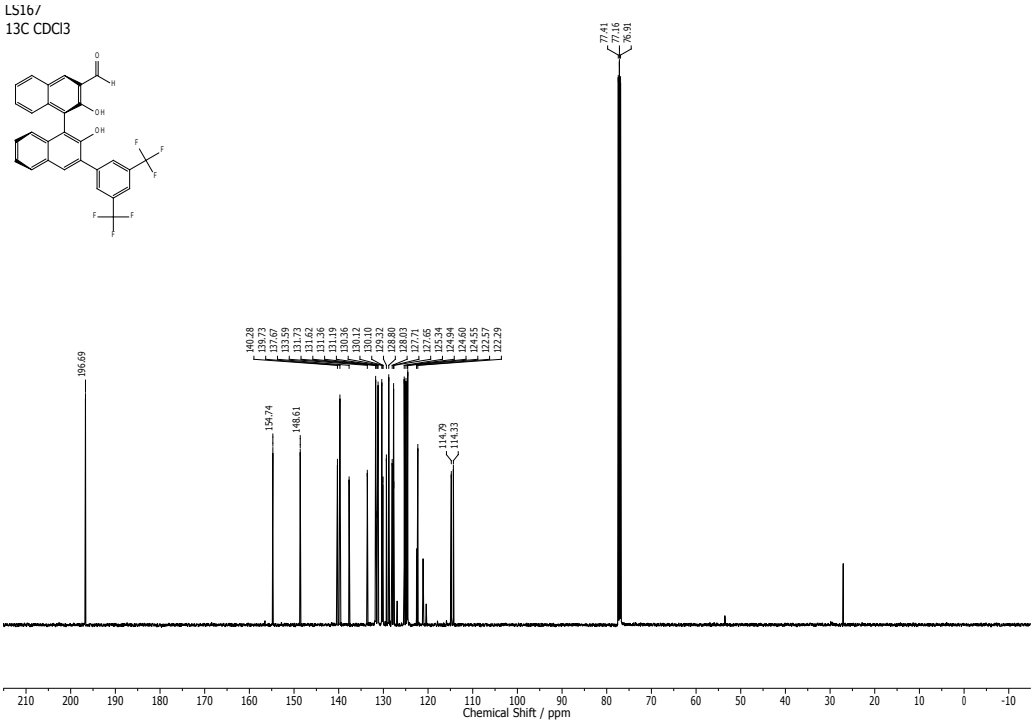
8) K. Nakata, Y. Kiyotsuka, T. Kitazume, Y. Kobayashi, *Synlett* **2011**, 2872.

## 5. NMR spectra



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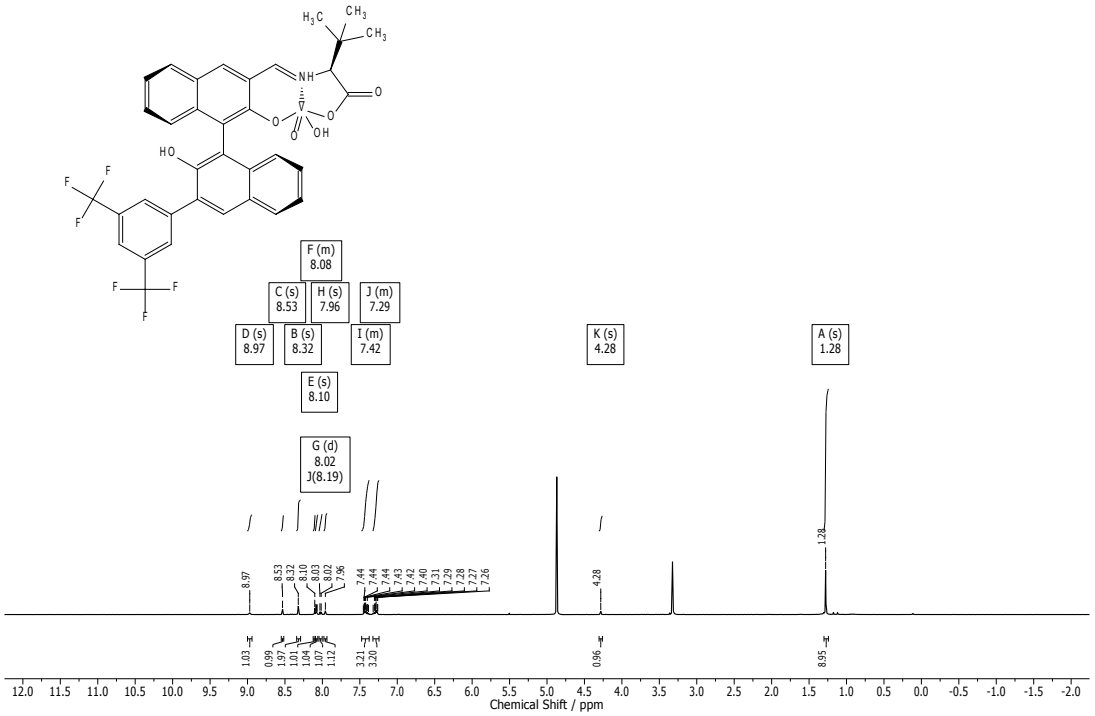


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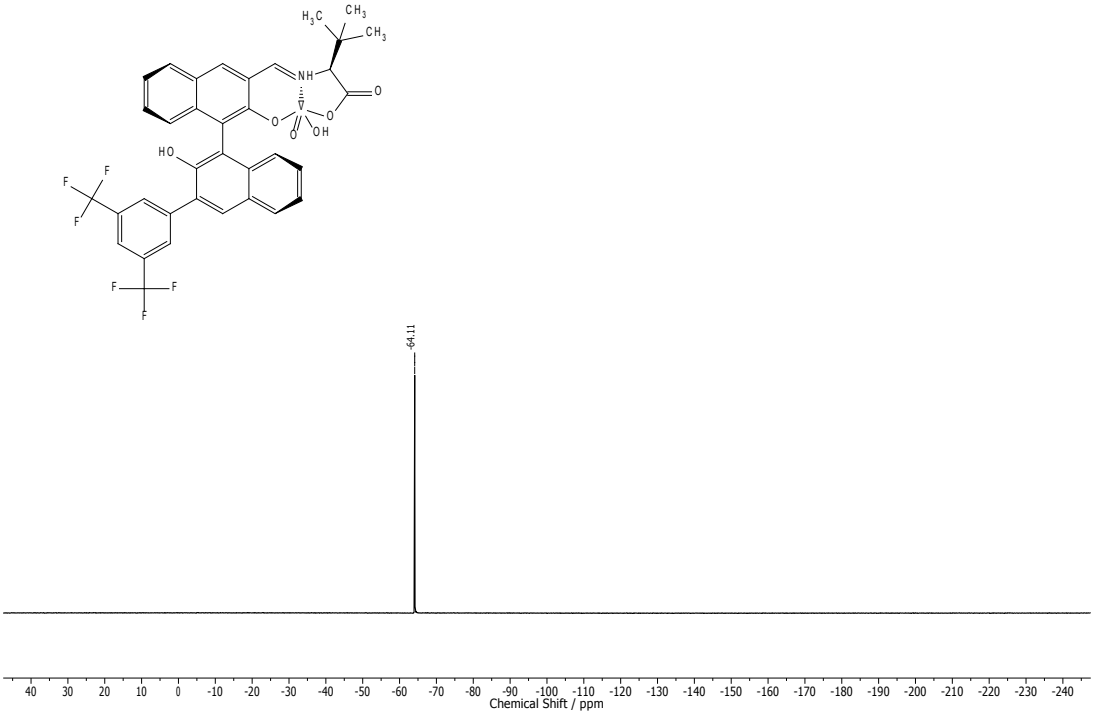

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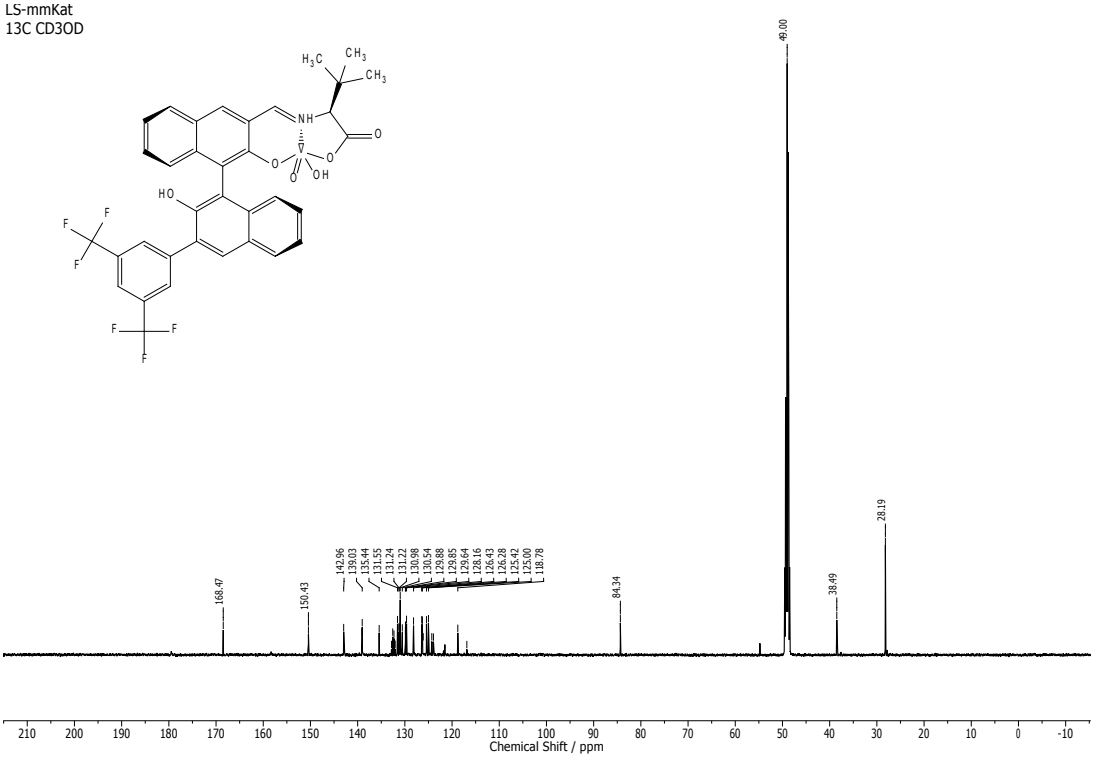

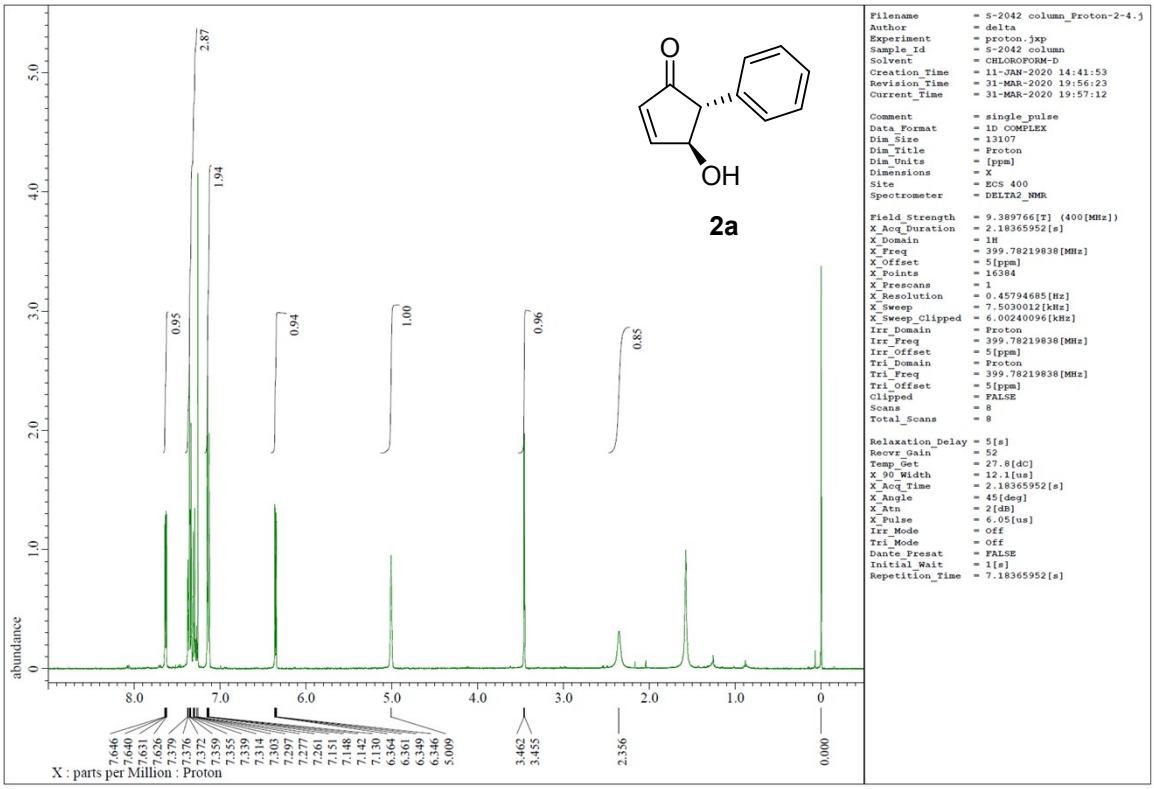
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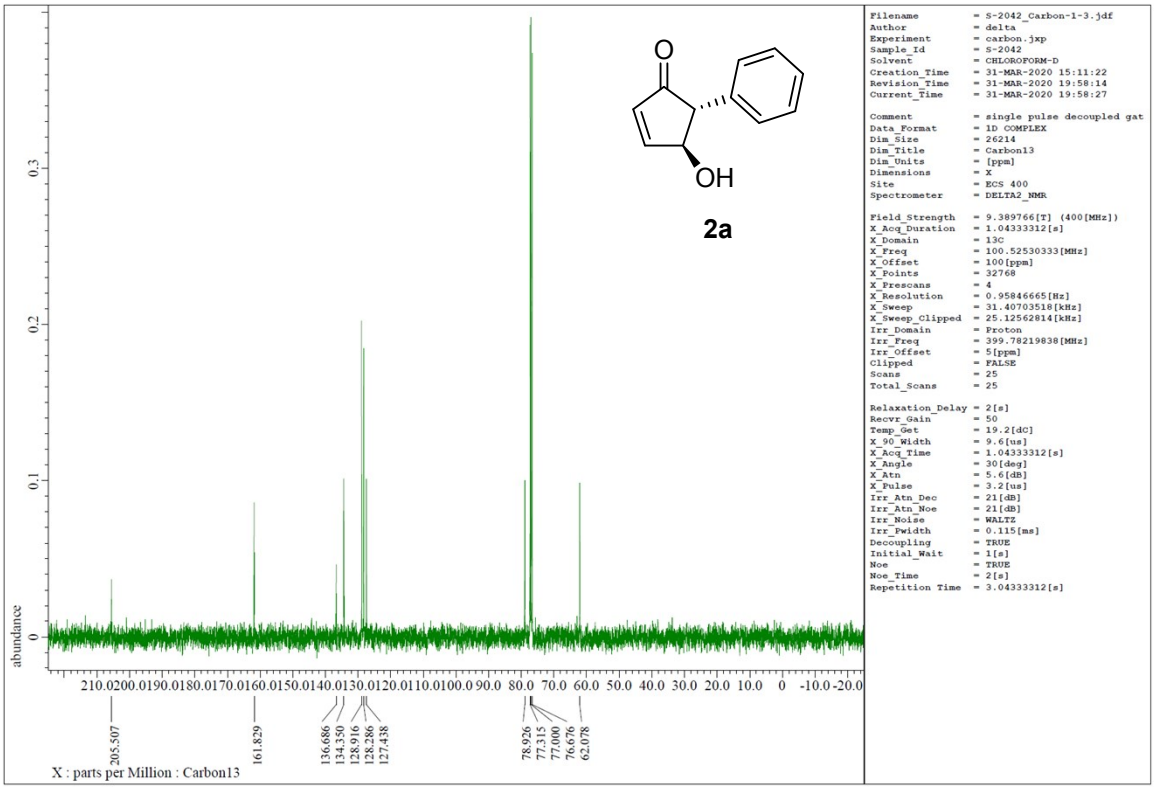
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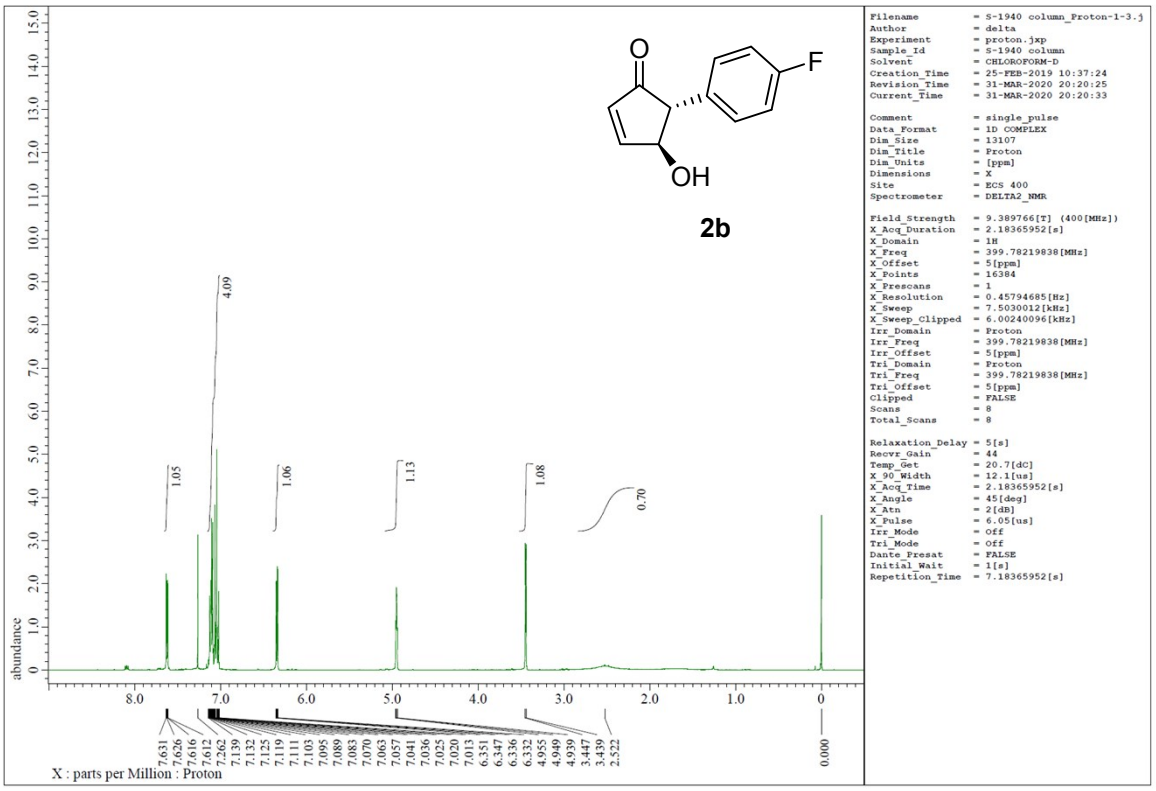
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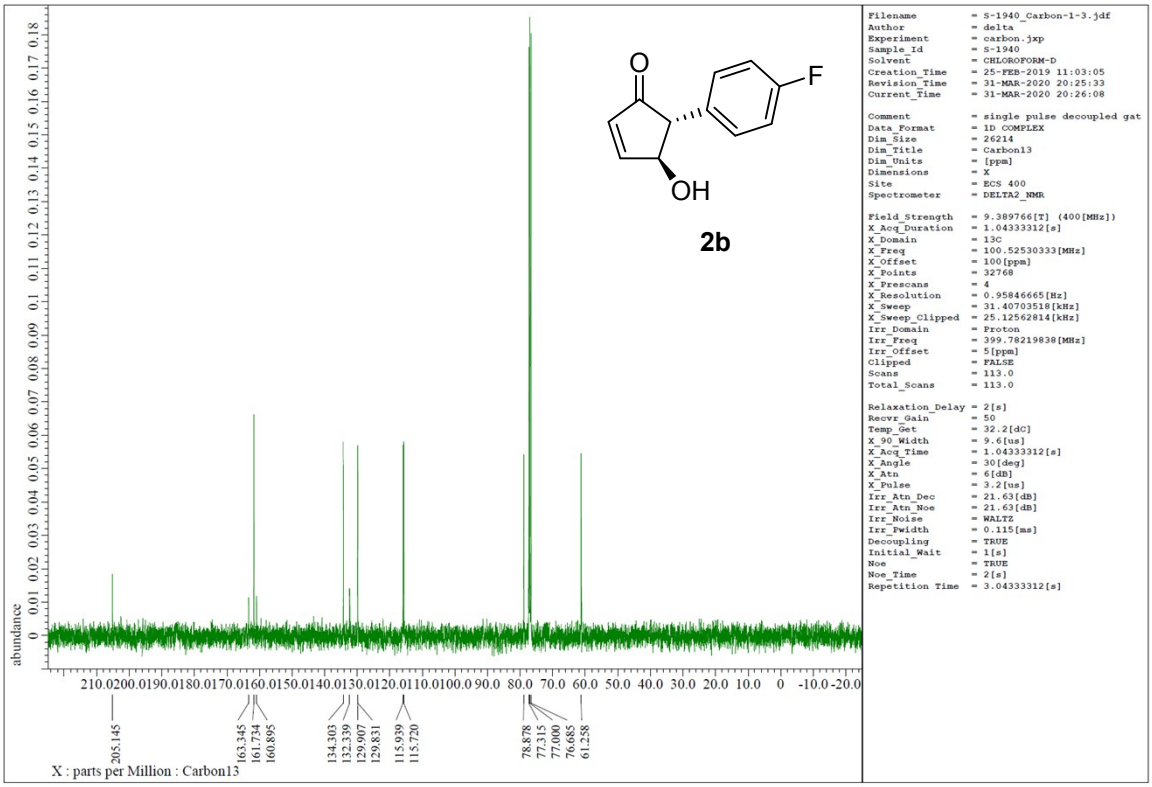
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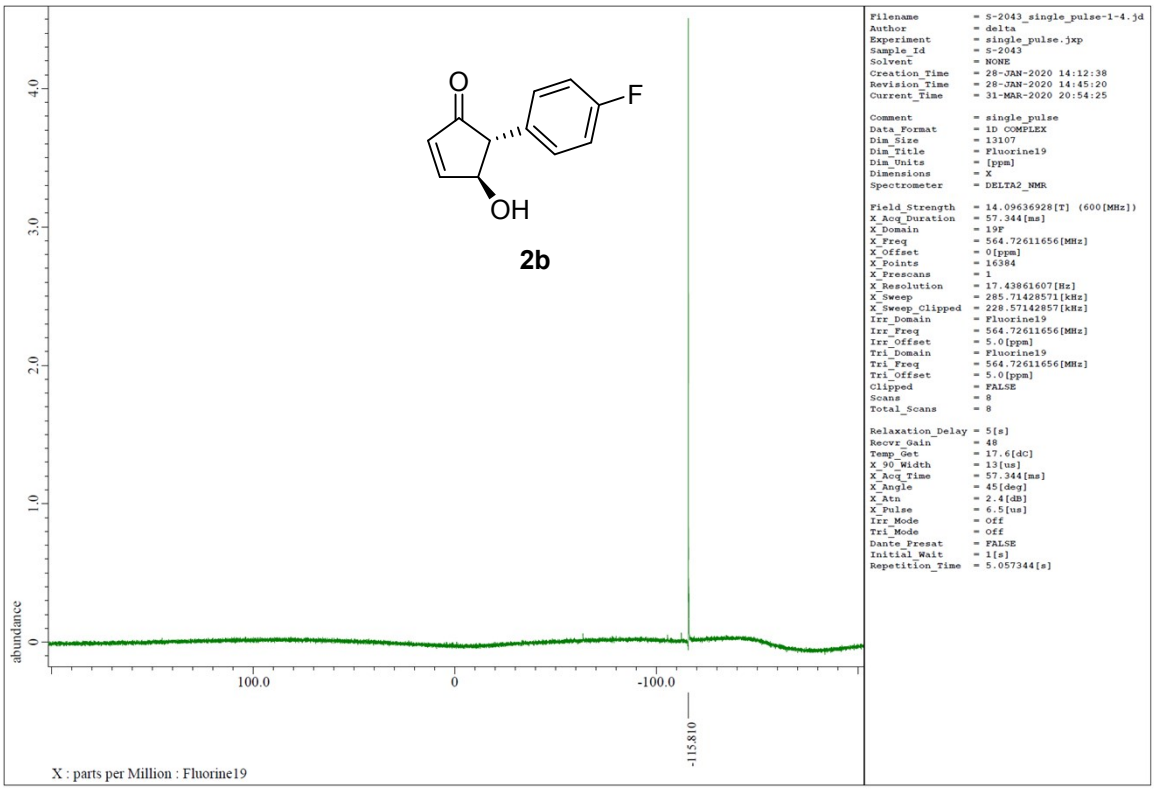
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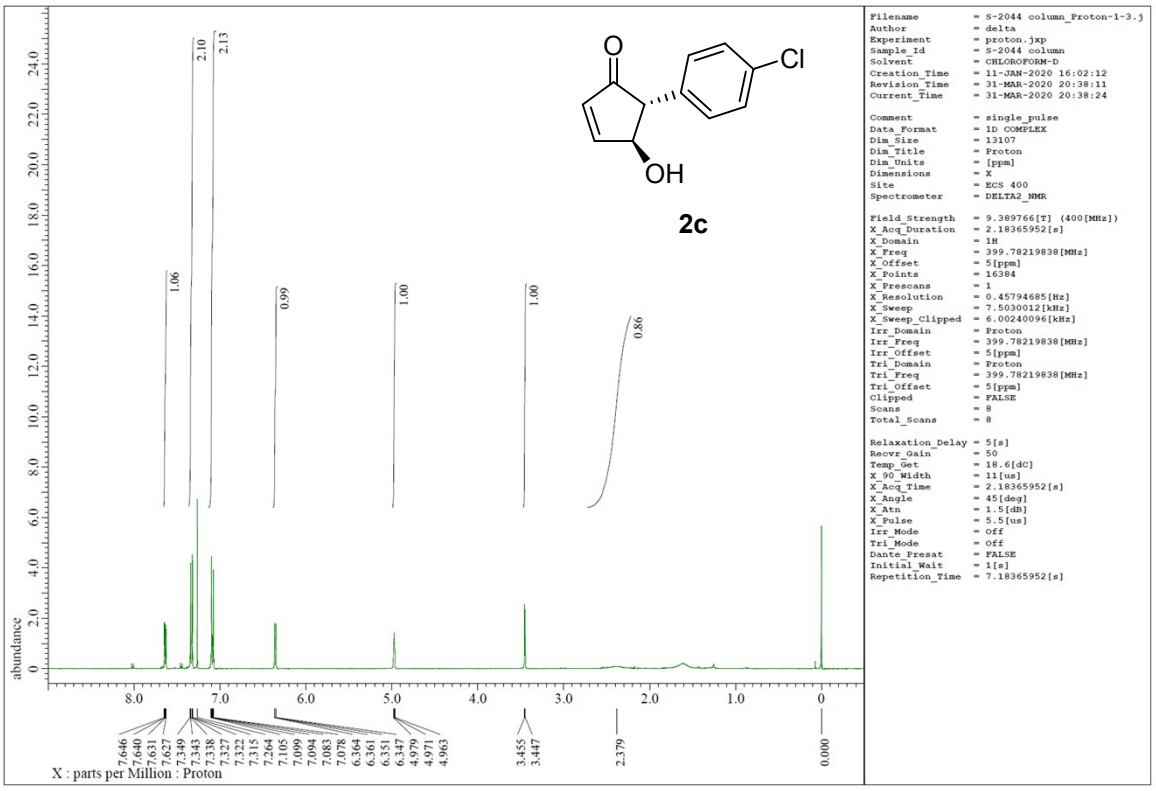
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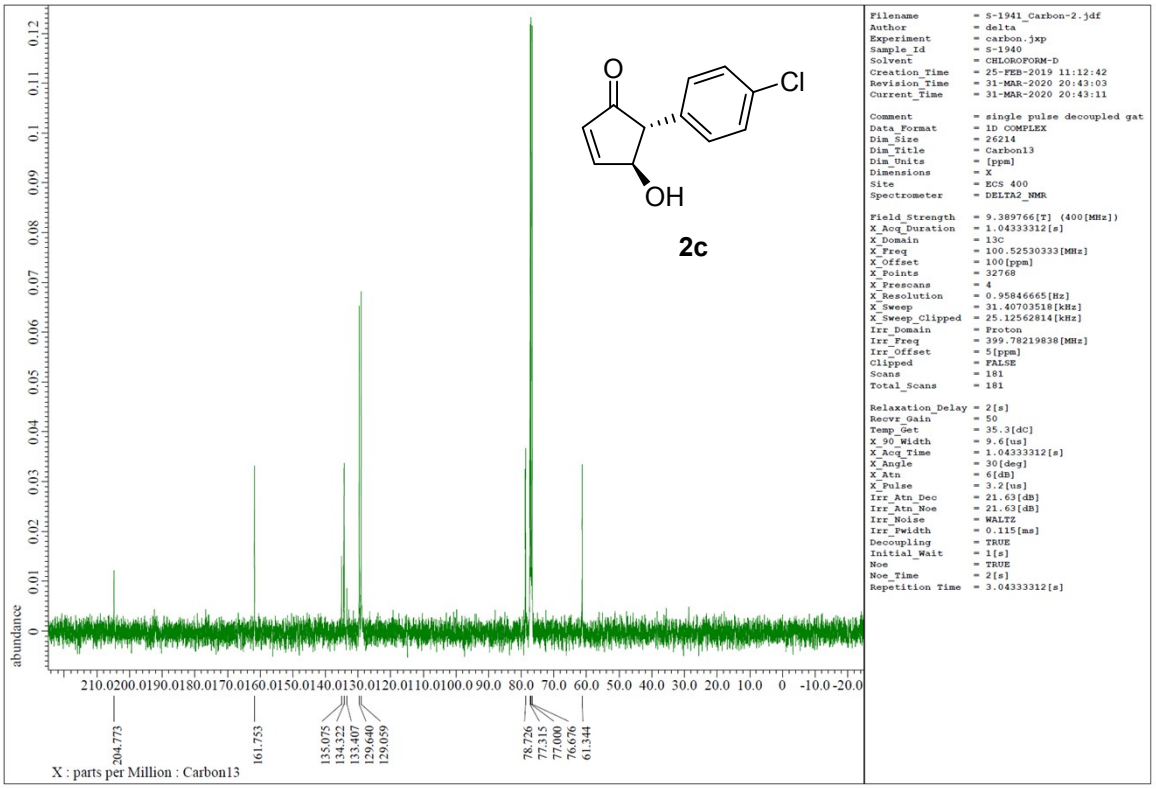
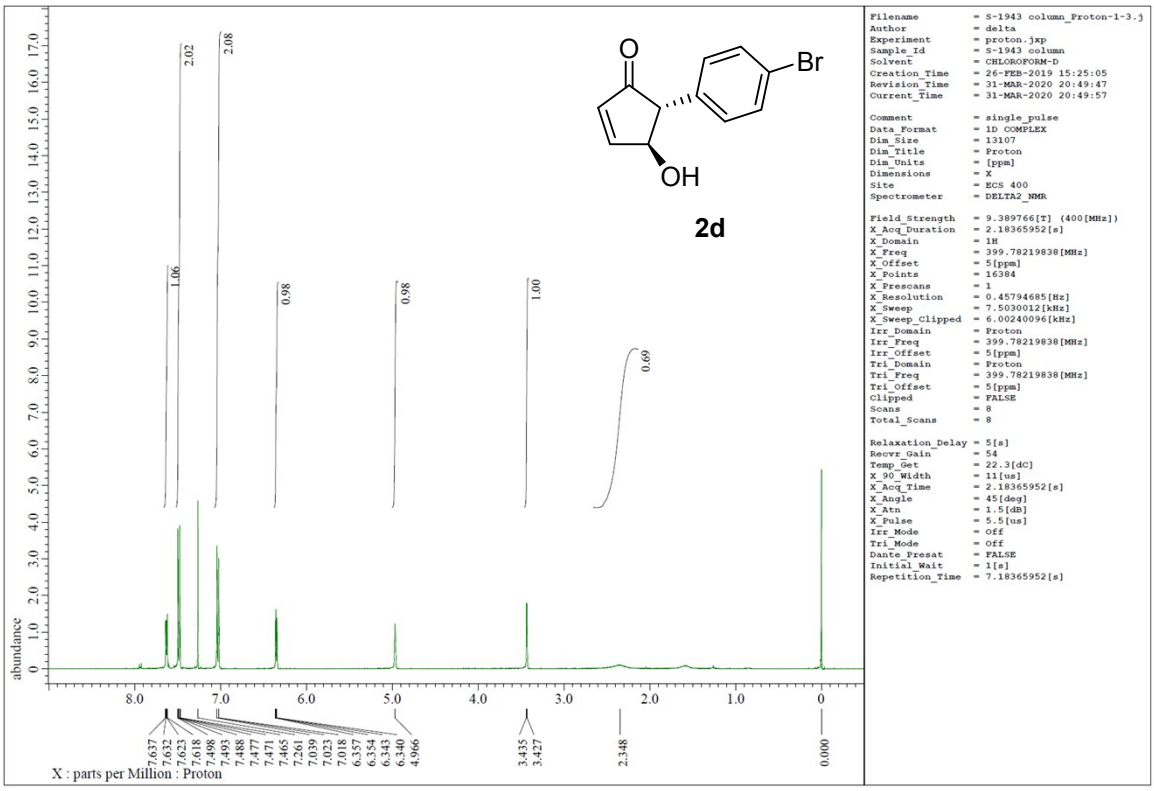
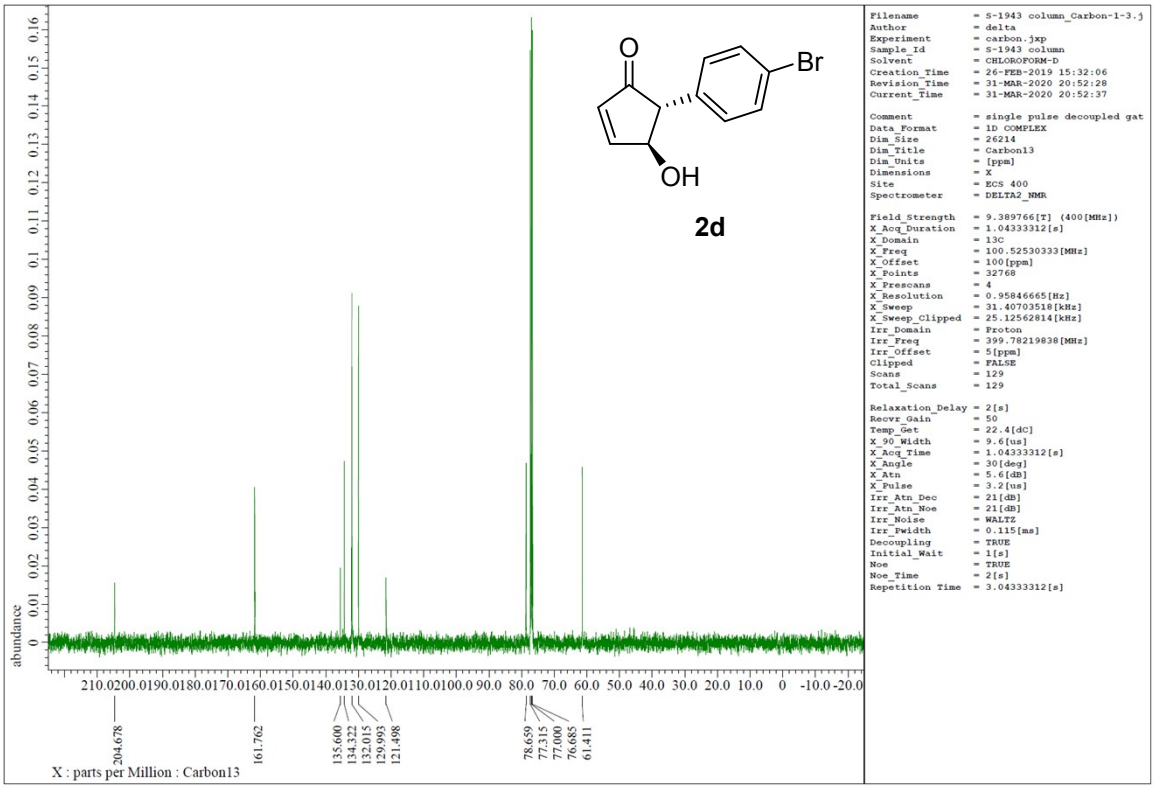
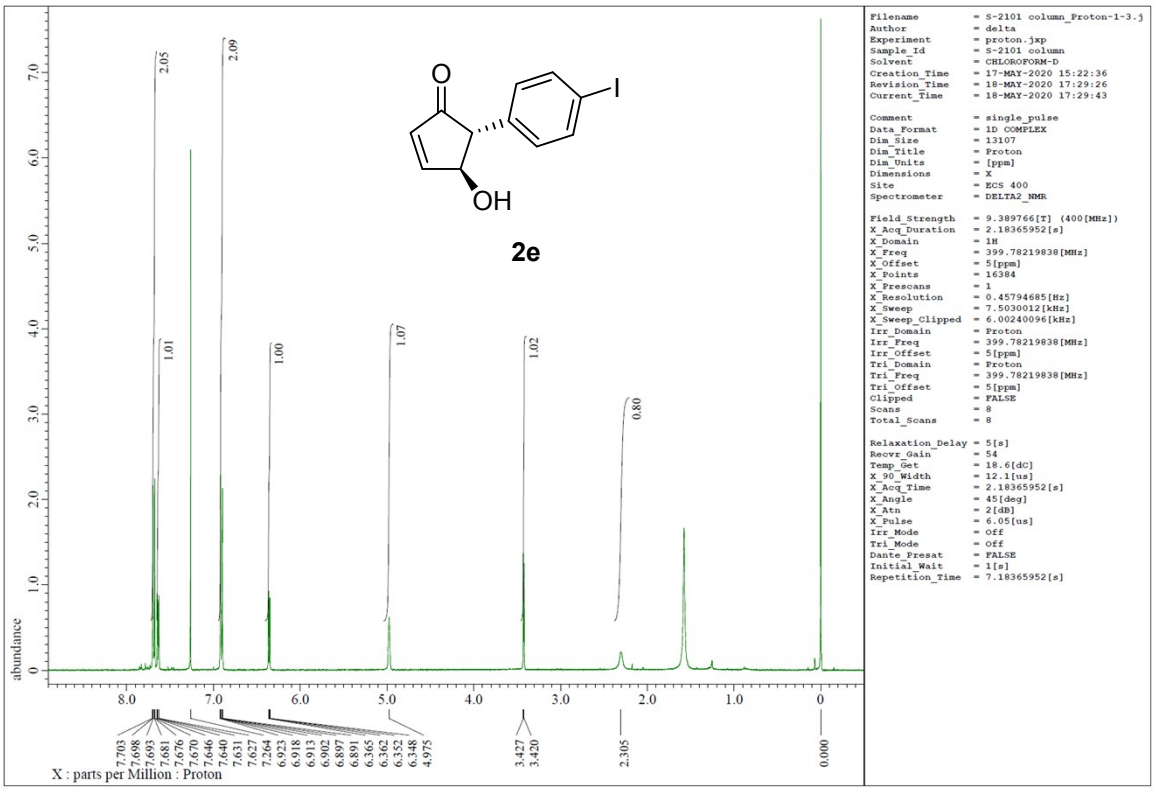
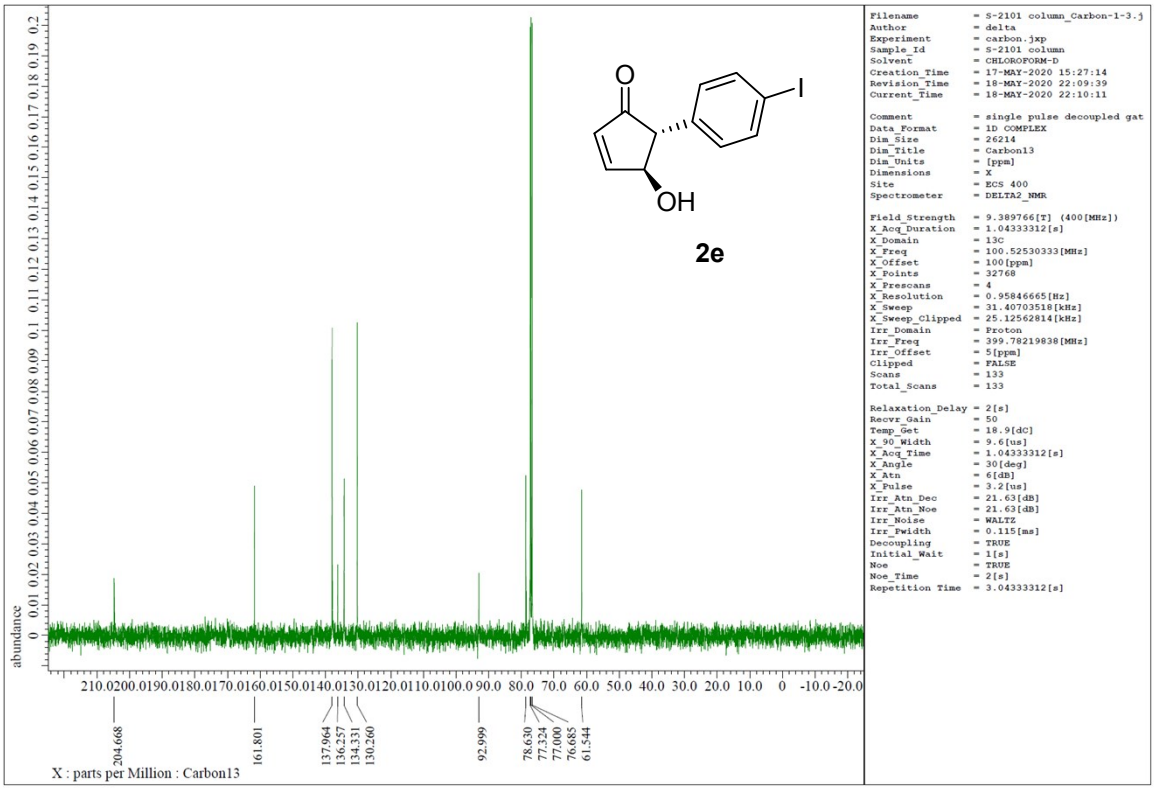
Temp_Get = 18.6[dC]
X_90_Width = 11[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 1.5[dB]
X_Pulse = 5.5[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]

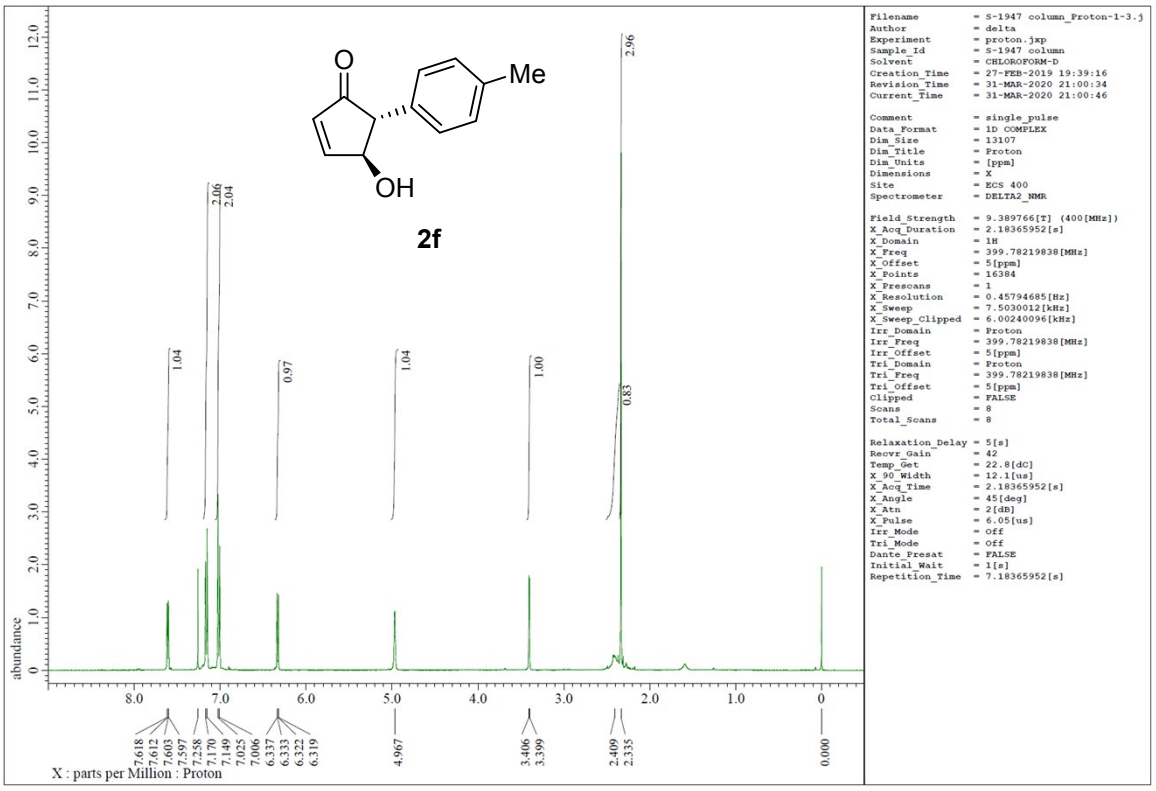

O
Cl
OH
2c
abundance
X : parts per Million : Carbon13
Filename = S-1941_Carbon-2.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = S-1940
Solvent = CHLOROFORM-D
Creation_Time = 25-FEB-2019 11:12:42
Revision_Time = 31-MAR-2020 20:43:03
Current_Time = 31-MAR-2020 20:43:11
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703510[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 181
Total_Scans = 181
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 35.3[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.63[dB]
Irr_Atn_Noe = 21.63[dB]
Irr_Noise = WALTZ
Irr_Fwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

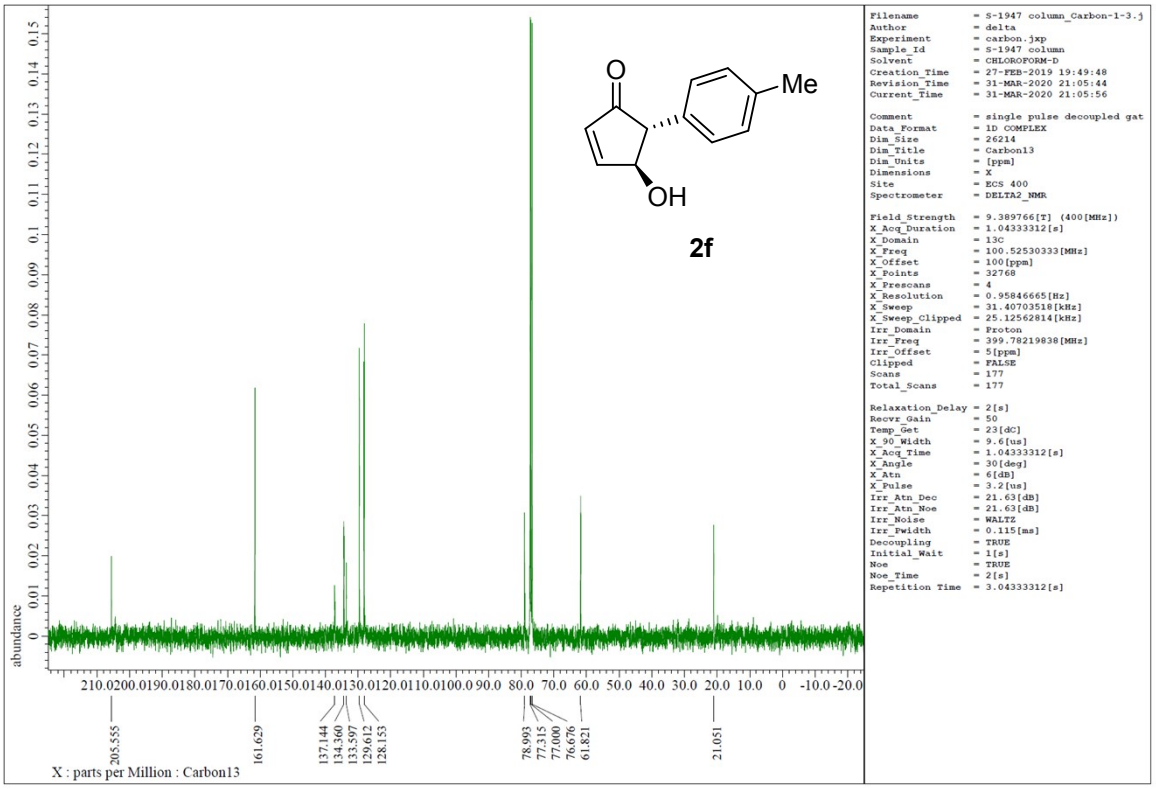

O
Br
OH
2d
2.02
2.08
1.06
0.98
0.98
1.00
0.69
abundance
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.637
7.632
7.623
7.618
7.498
7.493
7.488
7.477
7.471
7.465
7.261
7.039
7.023
7.018
6.357
6.354
6.343
6.340
4.966
3.435
3.427
2.348
0.000
X : parts per Million : Proton
Filename = S-1943 column_Proton-1-3.j
Author = delta
Experiment = proton.jxp
Sample_Id = S-1943 column
Solvent = CHLOROFORM-D
Creation_Time = 26-FEB-2019 15:25:05
Revision_Time = 31-MAR-2020 20:49:47
Current_Time = 31-MAR-2020 20:49:57
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 54
Temp_Get = 22.3[dC]
X_90_Width = 11[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 1.5[dB]
X_Pulse = 5.5[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]

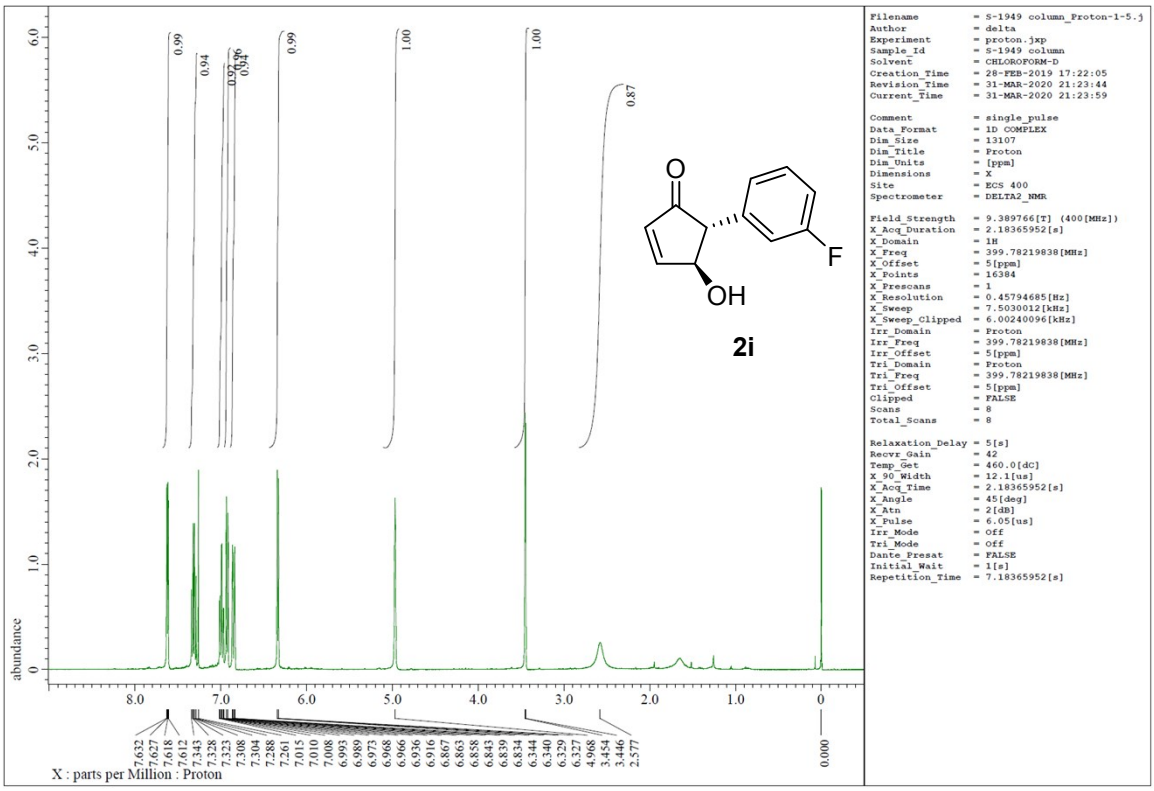

O
Br
OH
2d
abundance
0.16
0.15
0.14
0.13
0.12
0.11
0.1
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
204.678
161.762
135.600
134.322
132.015
129.993
121.498
78.659
77.315
77.000
76.685
61.411
X : parts per Million : Carbon13
Filename = S-1943 column_Carbon-1-3.j
Author = delta
Experiment = carbon.jxp
Sample_Id = S-1943 column
Solvent = CHLOROFORM-D
Creation_Time = 26-FEB-2019 15:32:06
Revision_Time = 31-MAR-2020 20:52:28
Current_Time = 31-MAR-2020 20:52:37
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 129
Total_Scans = 129
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 22.4[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 5.6[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21[dB]
Irr_Atn_Noe = 21[dB]
Irr_Noise = WALTZ
Irr_Fwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

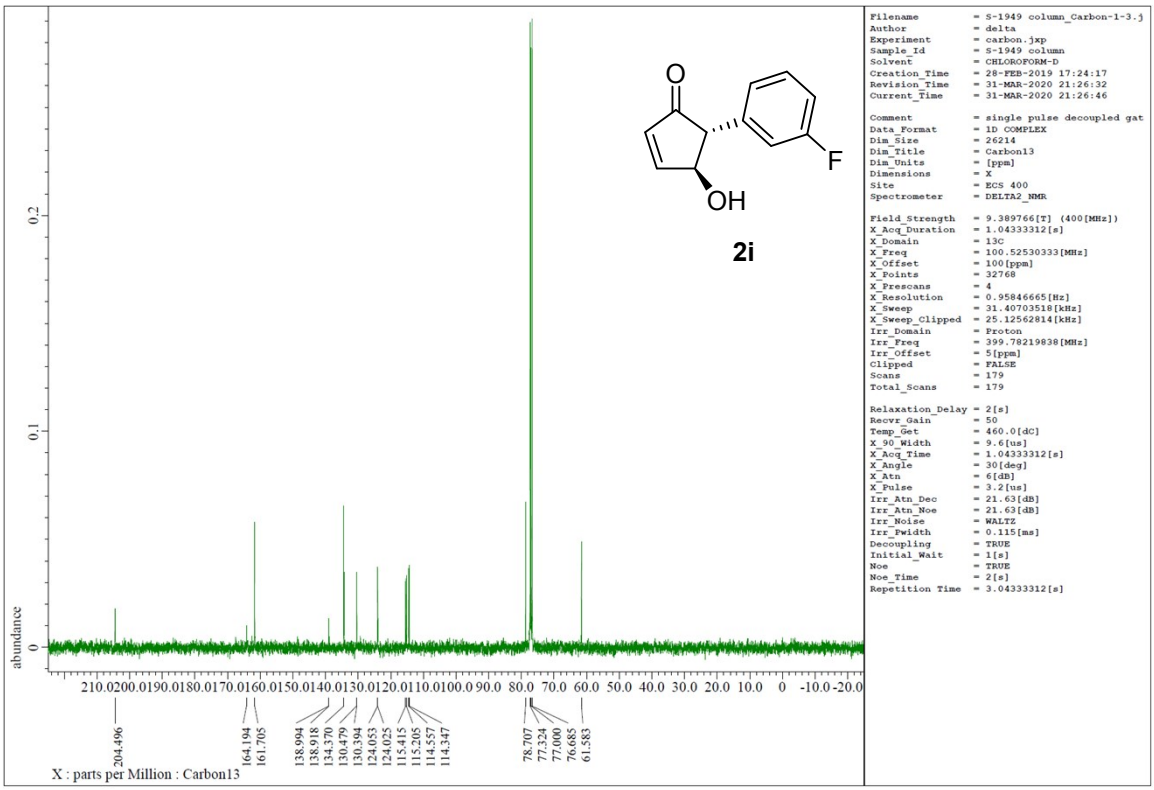

O
I
OH
2e
abundance
X : parts per Million : Proton
Filename = S-2101 column_Proton-1-3.j
Author = delta
Experiment = proton.jxp
Sample_Id = S-2101 column
Solvent = CHLOROFORM-D
Creation_Time = 17-MAY-2020 15:22:36
Revision_Time = 18-MAY-2020 17:29:26
Current_Time = 18-MAY-2020 17:29:43
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 54
Temp_Get = 18.6[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]

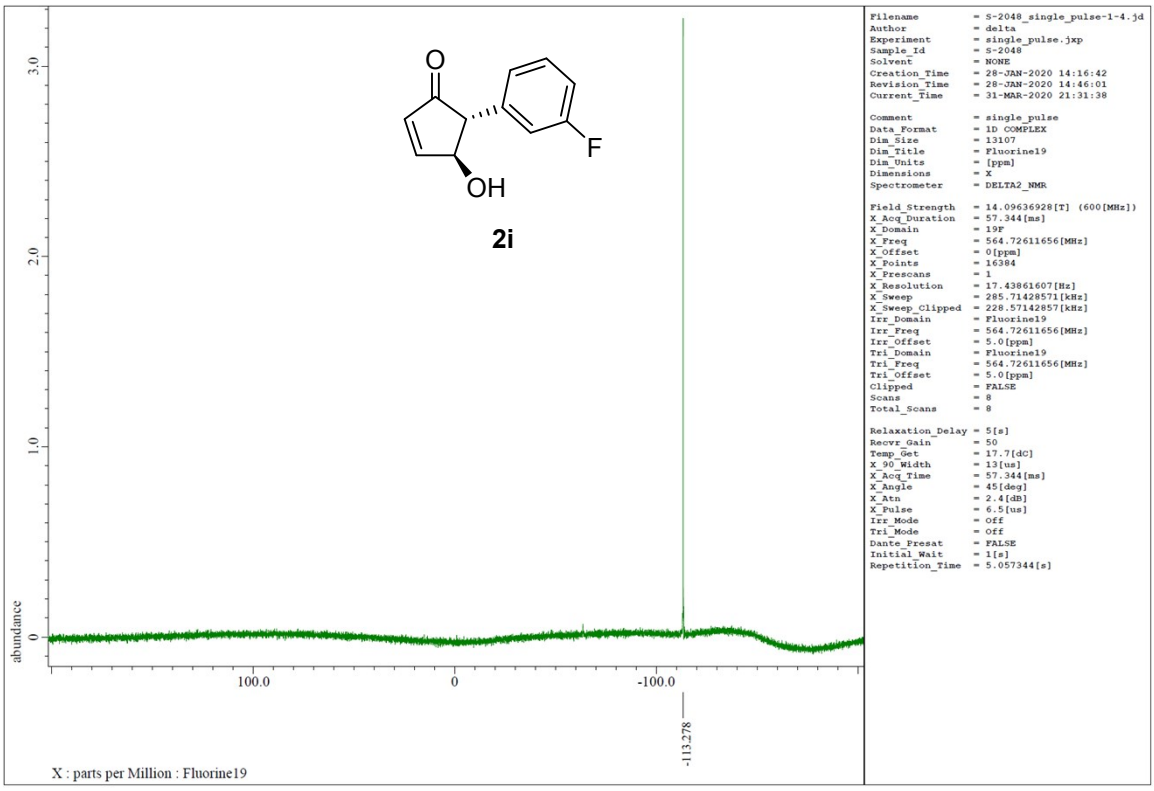

O
I
OH
2e
abundance
X : parts per Million : Carbon13
Filename = S-2101 column_Carbon-1-3.j
Author = delta
Experiment = carbon.jxp
Sample_Id = S-2101 column
Solvent = CHLOROFORM-D
Creation_Time = 17-MAY-2020 15:27:14
Revision_Time = 18-MAY-2020 22:09:39
Current_Time = 18-MAY-2020 22:10:11
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 133
Total_Scans = 133
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 18.9[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.63[dB]
Irr_Atn_Noe = 21.63[dB]
Irr_Noise = WALTZ
Irr_Fwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

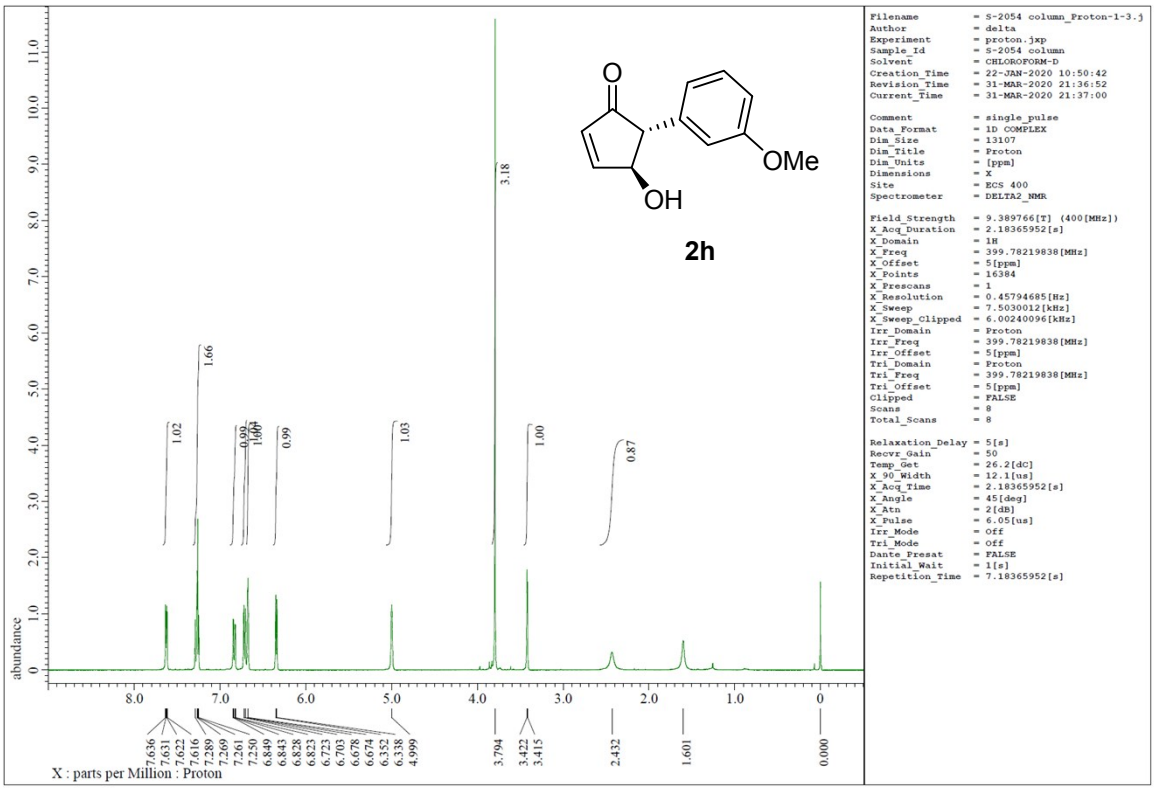

O
Me
OH
2f
2.96
2.06
2.04
1.04
0.97
1.04
1.00
0.83
abundance
X : parts per Million : Proton
7.618
7.612
7.603
7.597
7.258
7.170
7.149
7.025
7.006
6.337
6.333
6.322
6.319
4.967
3.406
3.399
2.409
2.335
0.000
Filename = S-1947 column_Proton-1-3.j
Author = delta
Experiment = proton.jxp
Sample_Id = S-1947 column
Solvent = CHLOROFORM-D
Creation_Time = 27-FEB-2019 19:39:16
Revision_Time = 31-MAR-2020 21:00:34
Current_Time = 31-MAR-2020 21:00:46
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 42
Temp_Get = 22.8[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]

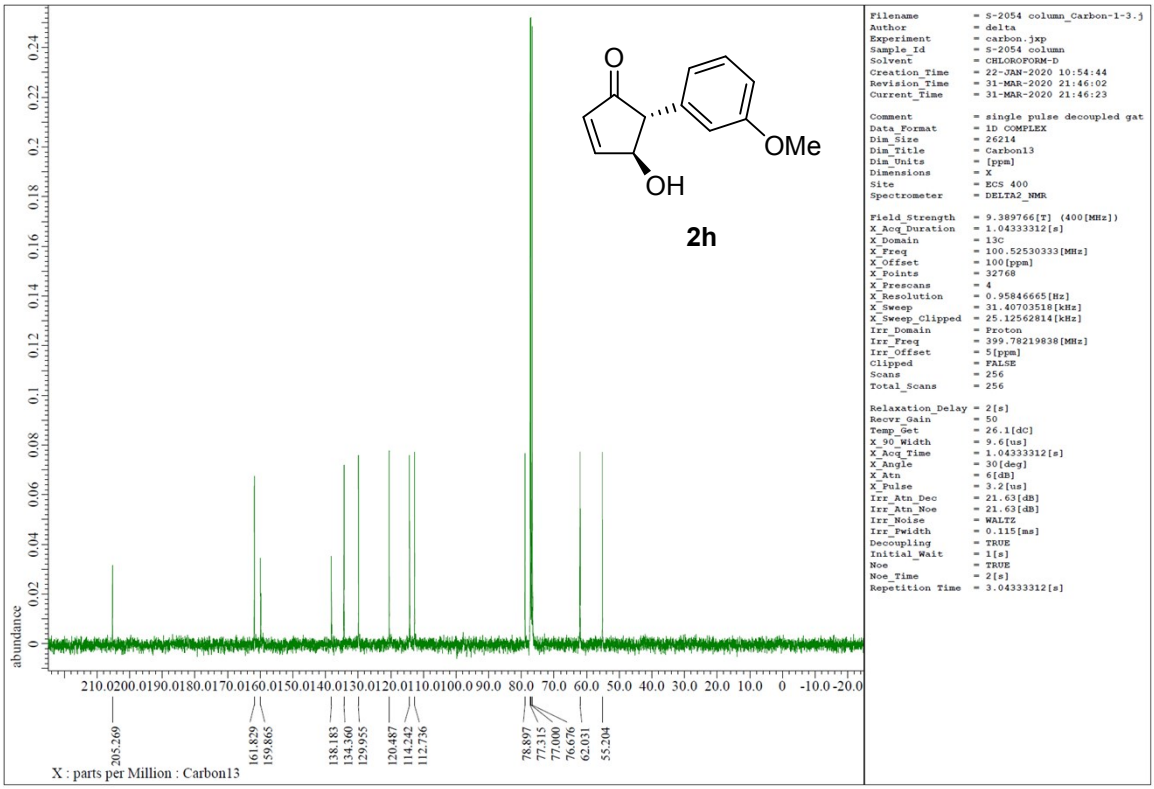

O
Me
OH
2f
abundance
X : parts per Million : Carbon13
205.555
161.629
137.144
134.360
133.597
129.612
128.153
78.993
77.315
77.000
76.676
61.821
21.051
Filename = S-1947 column_Carbon-1-3.j
Author = delta
Experiment = carbon.jxp
Sample_Id = S-1947 column
Solvent = CHLOROFORM-D
Creation_Time = 27-FEB-2019 19:49:48
Revision_Time = 31-MAR-2020 21:05:44
Current_Time = 31-MAR-2020 21:05:56
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 177
Total_Scans = 177
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 23[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.63[dB]
Irr_Atn_Noe = 21.63[dB]
Irr_Noise = WALTZ
Irr_Fwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

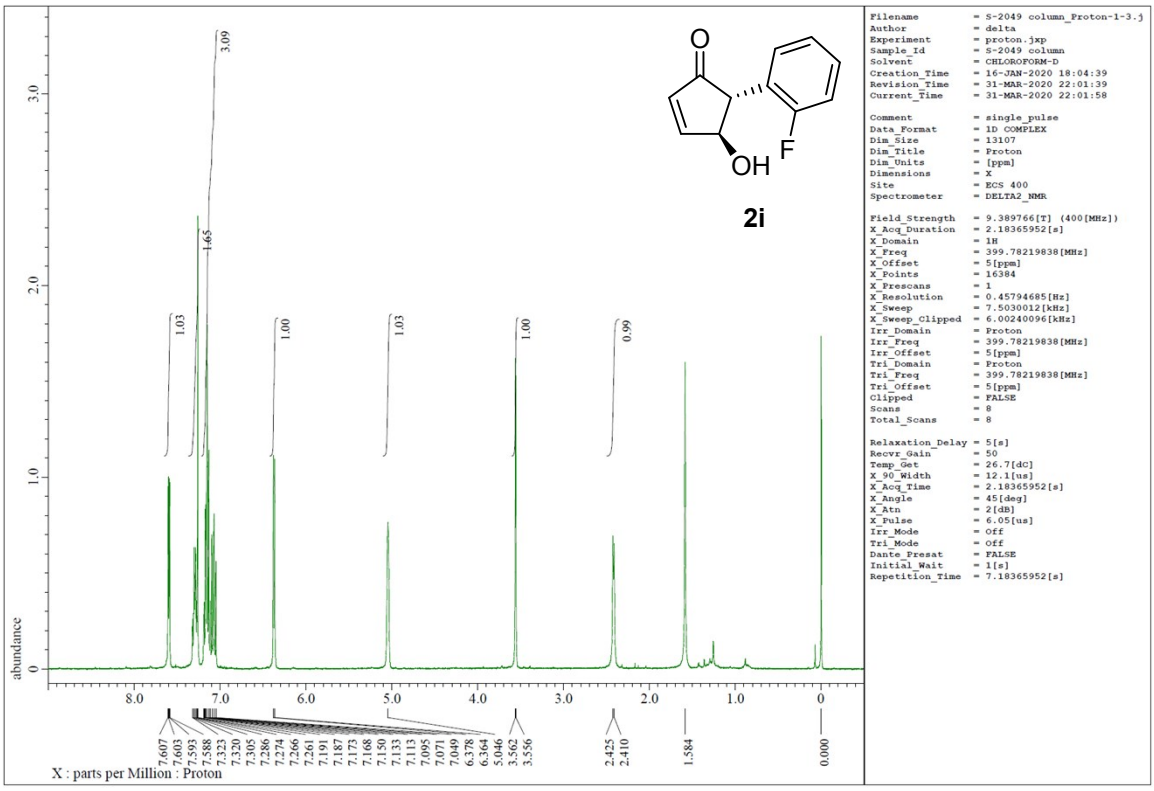

0.99
0.94
0.92
0.96
0.94
0.99
1.00
1.00
0.87
O
F
OH
2i
abundance
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.632
7.627
7.618
7.612
7.343
7.328
7.323
7.308
7.304
7.288
7.261
7.015
7.010
7.008
6.993
6.989
6.973
6.968
6.966
6.936
6.916
6.867
6.865
6.858
6.845
6.839
6.834
6.344
6.340
6.329
6.327
4.968
3.454
3.446
2.577
0.000
X : parts per Million : Proton
Filename = S-1949 column_Proton-1-5.j
Author = delta
Experiment = proton.jxp
Sample_Id = S-1949 column
Solvent = CHLOROFORM-D
Creation_Time = 28-FEB-2019 17:22:05
Revision_Time = 31-MAR-2020 21:23:44
Current_Time = 31-MAR-2020 21:23:59
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 42
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]

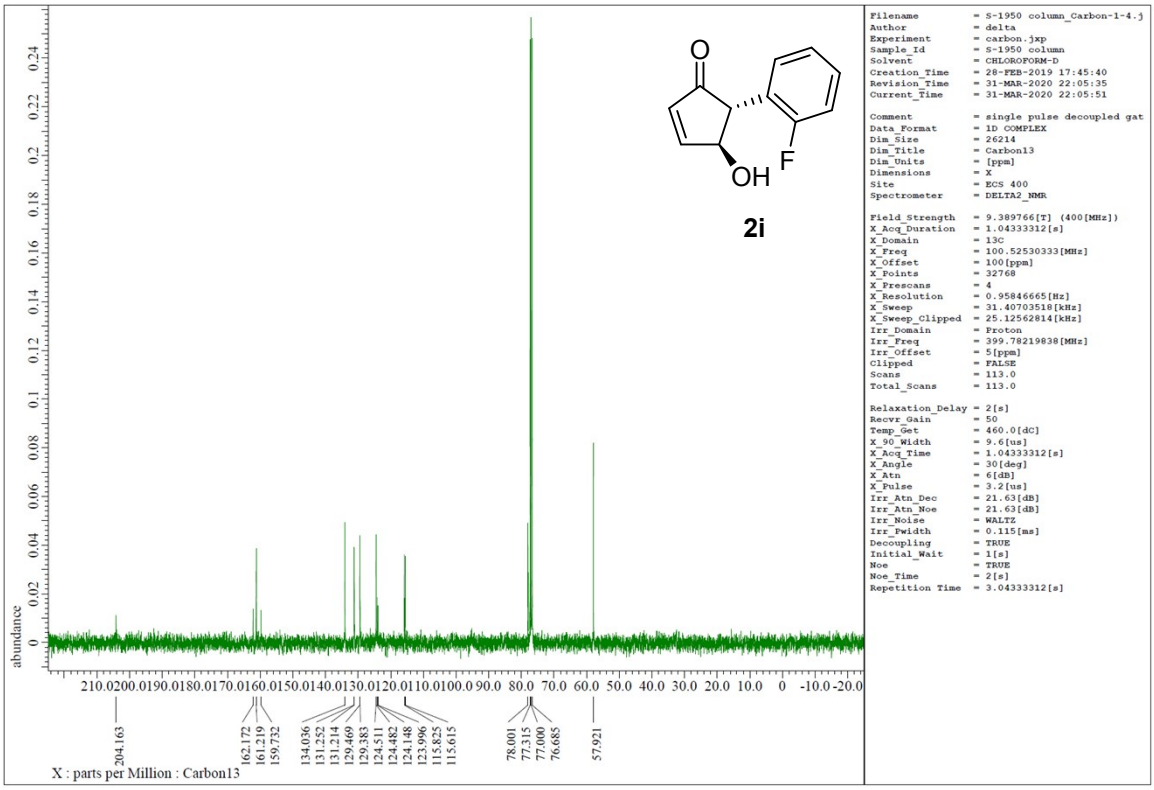

O
F
OH
2i
abundance
0.2
0.1
0
210.0200.0190.0180.0170.0160.0150.0140.0130.0120.0110.0100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
204.496
164.194
161.705
138.994
138.918
134.370
130.479
130.394
124.053
124.025
115.415
115.205
114.557
114.347
78.707
77.324
77.000
76.685
61.583
X : parts per Million : Carbon13
Filename = S-1949 column_Carbon-1-3.j
Author = delta
Experiment = carbon.jxp
Sample_Id = S-1949 column
Solvent = CHLOROFORM-D
Creation_Time = 28-FEB-2019 17:24:17
Revision_Time = 31-MAR-2020 21:26:32
Current_Time = 31-MAR-2020 21:26:46
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 179
Total_Scans = 179
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 460.0[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.63[dB]
Irr_Atn_Noe = 21.63[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

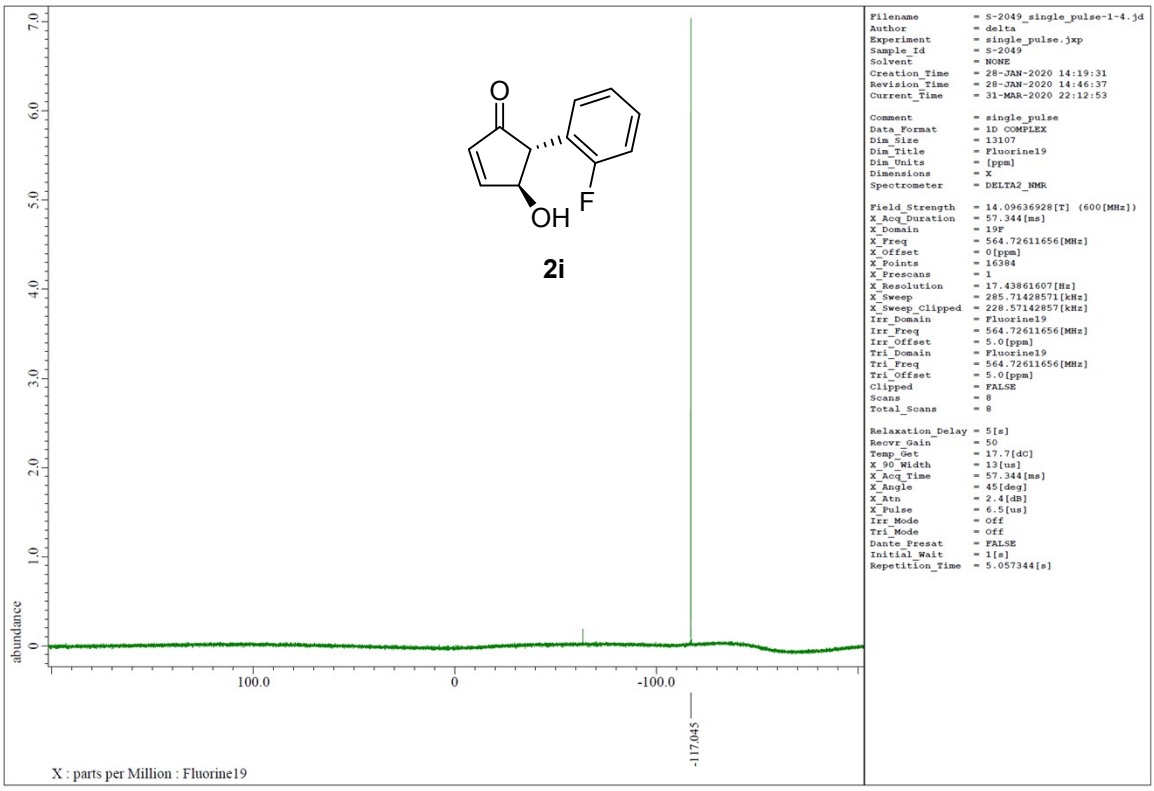

O
F
OH
2i
3.0
2.0
1.0
0
abundance
100.0
0
-100.0
-113.278
X : parts per Million : Fluorine19
Filename = S-2048_single_pulse-1-4.jd
Author = delta
Experiment = single_pulse.jxp
Sample_Id = S-2048
Solvent = NONE
Creation_Time = 28-JAN-2020 14:16:42
Revision_Time = 28-JAN-2020 14:46:01
Current_Time = 31-MAR-2020 21:31:38
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Fluorine19
Dim_Units = [ppm]
Dimensions = X
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 57.344[ms]
X_Domain = 19F
X_Freq = 564.72611656[MHz]
X_Offset = 0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 17.43861607[Hz]
X_Sweep = 285.71428571[kHz]
X_Sweep_Clipped = 228.57142857[kHz]
Irr_Domain = Fluorine19
Irr_Freq = 564.72611656[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Fluorine19
Tri_Freq = 564.72611656[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 17.7[dC]
X_90_Width = 13[us]
X_Acq_Time = 57.344[ms]
X_Angle = 45[deg]
X_Atn = 2.4[dB]
X_Pulse = 6.5[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 5.057344[s]

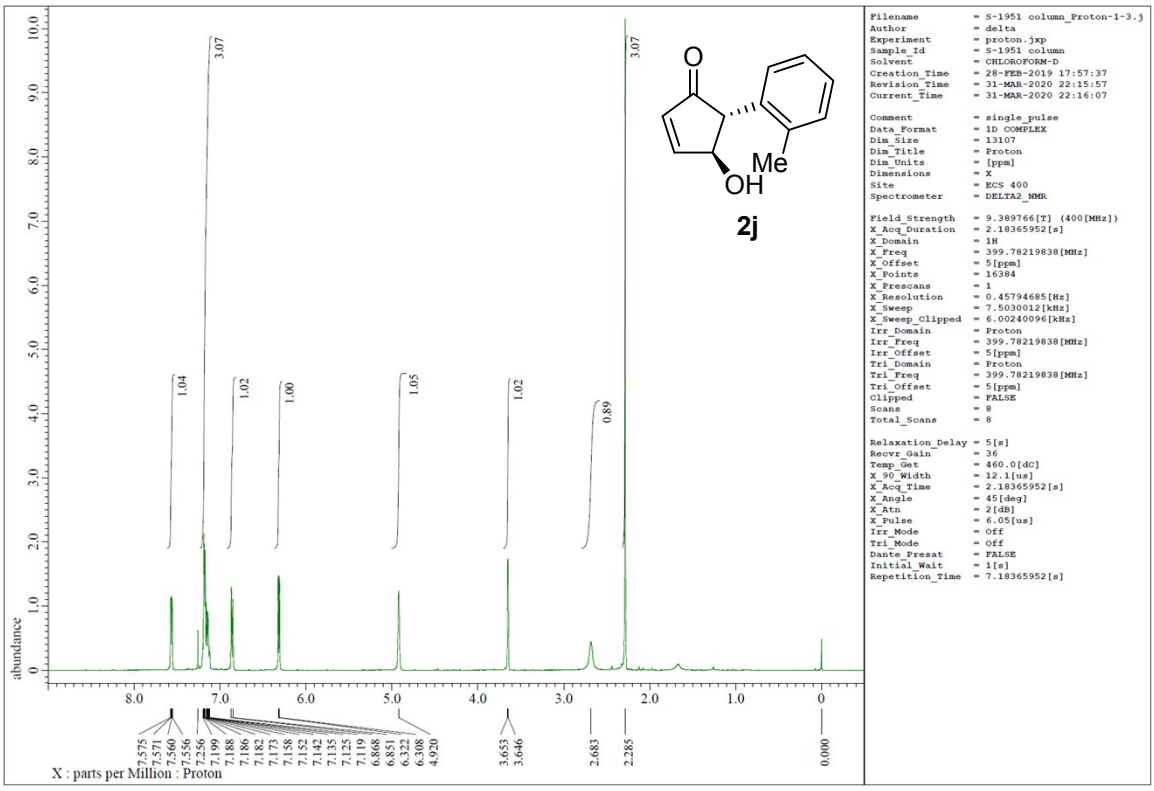

O
OMe
OH
2h
abundance
X : parts per Million : Proton
1.02
1.66
0.99
0.99
1.03
3.18
1.00
0.87
7.636
7.631
7.622
7.616
7.289
7.269
7.261
7.250
6.849
6.843
6.828
6.823
6.723
6.703
6.678
6.674
6.352
6.338
4.999
3.794
3.422
3.415
2.432
1.601
0.000
Filename = S-2054 column_Proton-1-3.j
Author = delta
Experiment = proton.jxp
Sample_Id = S-2054 column
Solvent = CHLOROFORM-D
Creation_Time = 22-JAN-2020 10:50:42
Revision_Time = 31-MAR-2020 21:36:52
Current_Time = 31-MAR-2020 21:37:00
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 26.2[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]

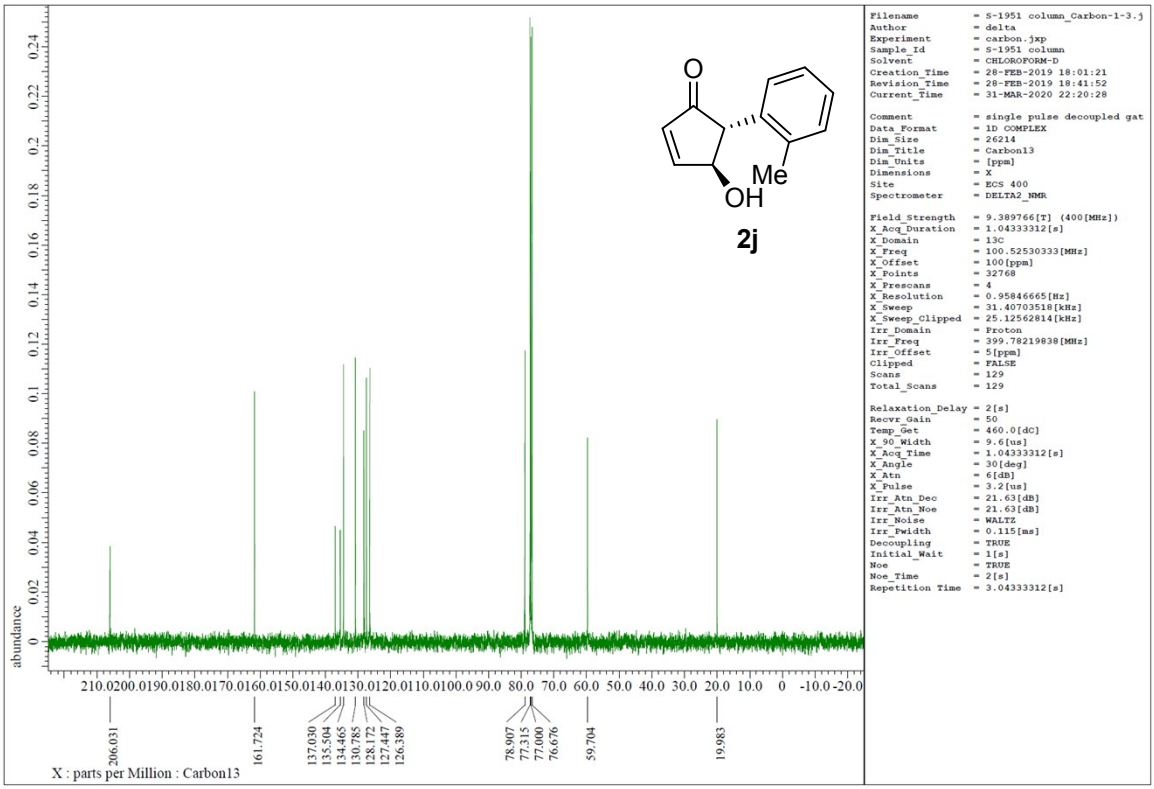

O
OMe
OH
2h
abundance
X : parts per Million : Carbon13
205.269
161.829
159.865
138.183
134.360
129.955
120.487
114.242
112.736
78.897
77.315
77.000
76.676
62.031
55.204
Filename = S-2054 column_Carbon-1-3.j
Author = delta
Experiment = carbon.jxp
Sample_Id = S-2054 column
Solvent = CHLOROFORM-D
Creation_Time = 22-JAN-2020 10:54:44
Revision_Time = 31-MAR-2020 21:46:02
Current_Time = 31-MAR-2020 21:46:23
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 26.1[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.63[dB]
Irr_Atn_Noe = 21.63[dB]
Irr_Noise = WALTZ
Irr_Fwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

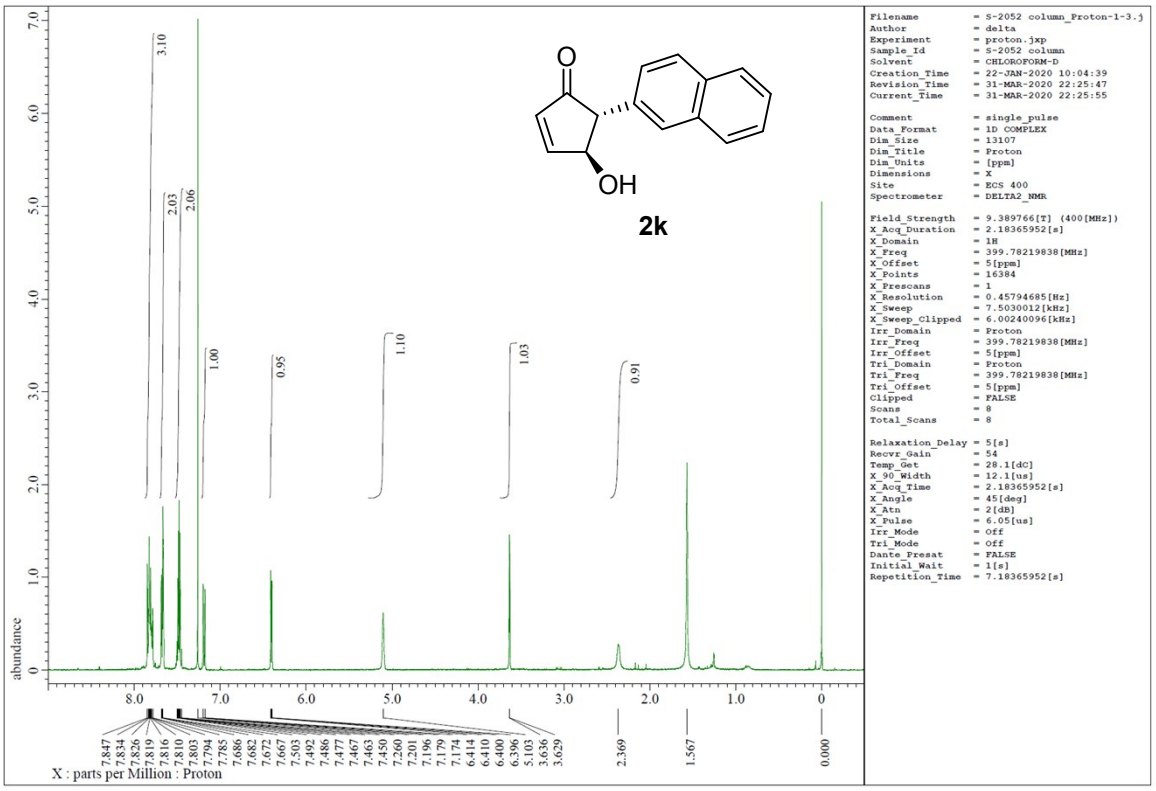

O
OH
F
2i
3.09
1.65
1.03
1.00
1.03
1.00
0.99
abundance
X : parts per Million : Proton
Filename = S-2049 column_Proton-1-3.j
Author = delta
Experiment = proton.jxp
Sample_Id = S-2049 column
Solvent = CHLOROFORM-D
Creation_Time = 16-JAN-2020 18:04:39
Revision_Time = 31-MAR-2020 22:01:39
Current_Time = 31-MAR-2020 22:01:58
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 26.7[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]

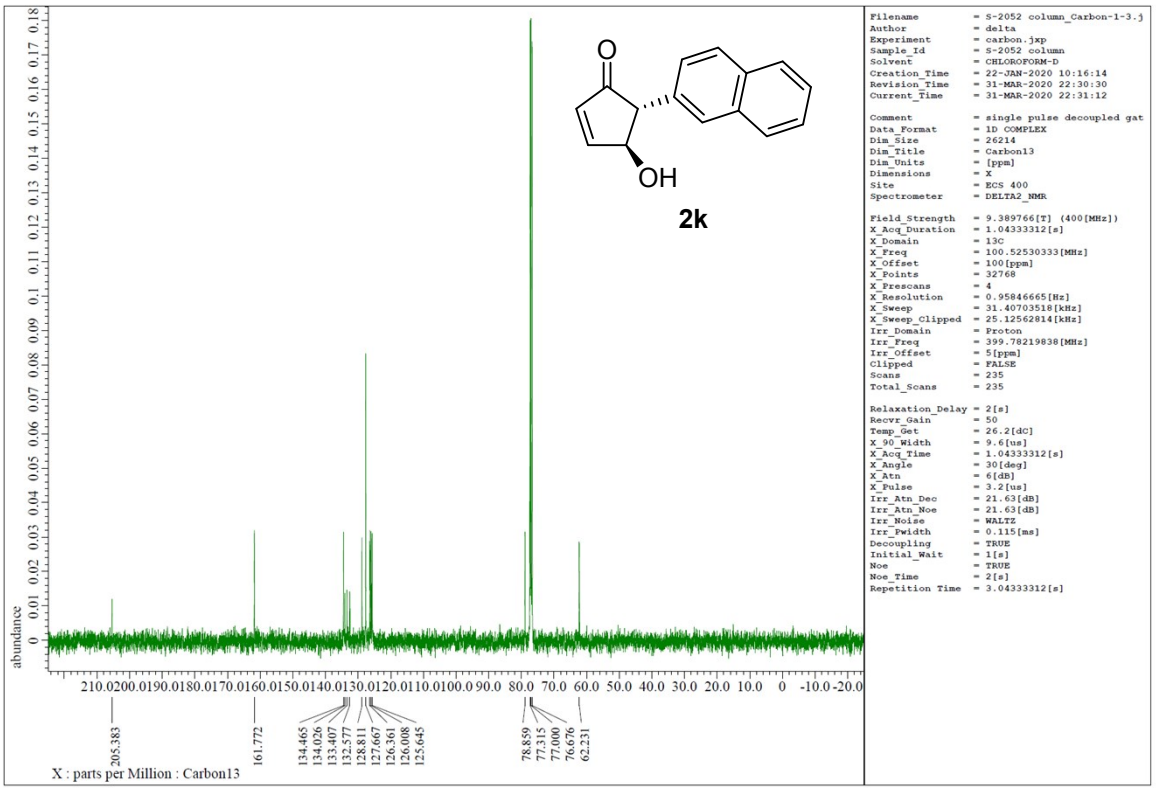

O
OH
F
2i
abundance
X : parts per Million : Carbon13
Filename = S-1950 column_Carbon-1-4.j
Author = delta
Experiment = carbon.jxp
Sample_Id = S-1950 column
Solvent = CHLOROFORM-D
Creation_Time = 28-FEB-2019 17:45:40
Revision_Time = 31-MAR-2020 22:05:35
Current_Time = 31-MAR-2020 22:05:51
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 113.0
Total_Scans = 113.0
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 460.0[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.63[dB]
Irr_Atn_Noe = 21.63[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

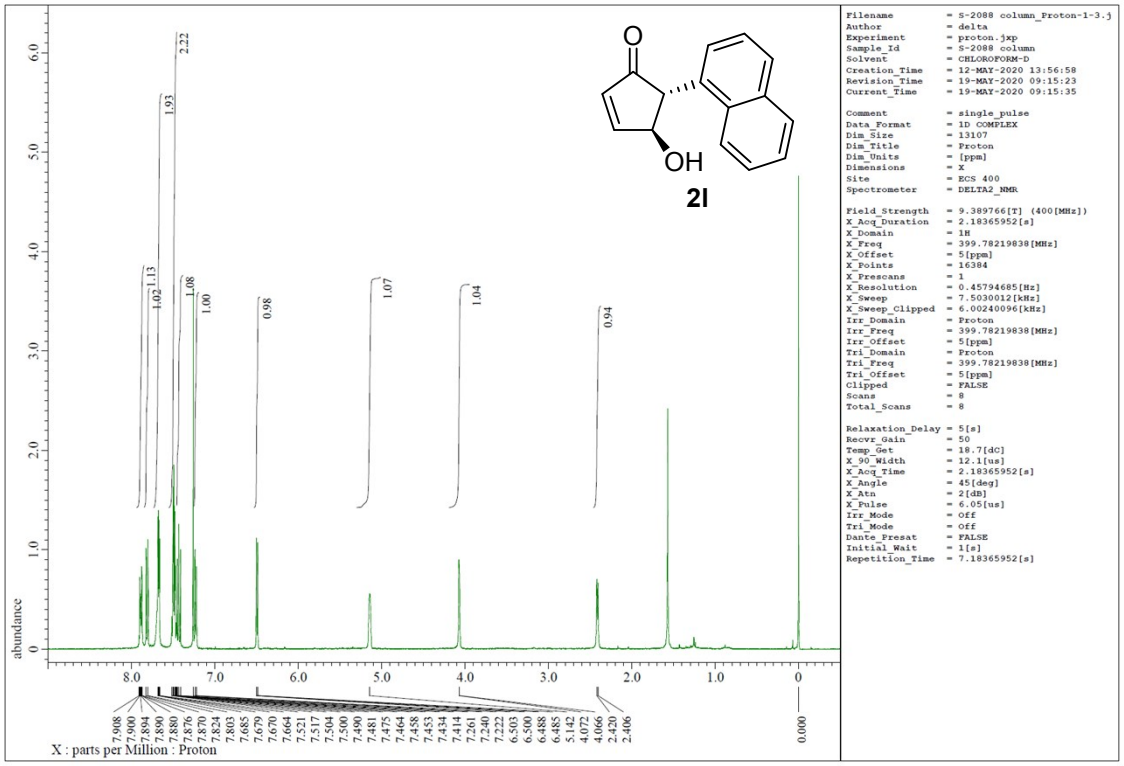

O
OH
F
2i
abundance
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
100.0
0
-100.0
-117.045
X : parts per Million : Fluorine19
Filename = S-2049_single_pulse-1-4.jd
Author = delta
Experiment = single_pulse.jxp
Sample_Id = S-2049
Solvent = NONE
Creation_Time = 28-JAN-2020 14:19:31
Revision_Time = 28-JAN-2020 14:46:37
Current_Time = 31-MAR-2020 22:12:53
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Fluorine19
Dim_Units = [ppm]
Dimensions = X
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 57.344[ms]
X_Domain = 19F
X_Freq = 564.72611656[MHz]
X_Offset = 0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 17.43861607[Hz]
X_Sweep = 285.71428571[kHz]
X_Sweep_Clipped = 228.57142857[kHz]
Irr_Domain = Fluorine19
Irr_Freq = 564.72611656[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Fluorine19
Tri_Freq = 564.72611656[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 17.7[dC]
X_90_Width = 13[us]
X_Acq_Time = 57.344[ms]
X_Angle = 45[deg]
X_Atn = 2.4[dB]
X_Pulse = 6.5[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 5.057344[s]

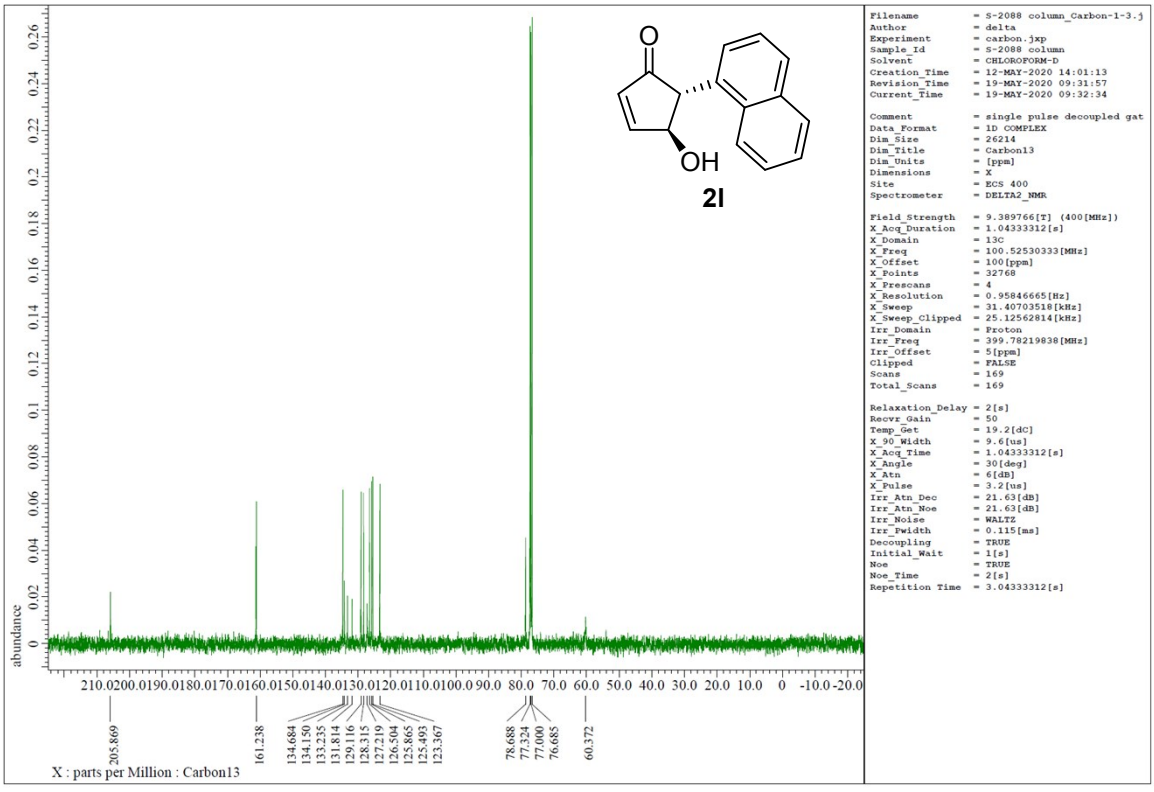

O
Me
OH
2j
3.07
1.04
1.02
1.00
1.05
1.02
0.89
3.07
abundance
X : parts per Million : Proton
Filename = S-1951 column_Proton-1-3.j
Author = delta
Experiment = proton.jxp
Sample_Id = S-1951 column
Solvent = CHLOROFORM-D
Creation_Time = 28-FEB-2019 17:57:37
Revision_Time = 31-MAR-2020 22:15:57
Current_Time = 31-MAR-2020 22:16:07
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 36
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]

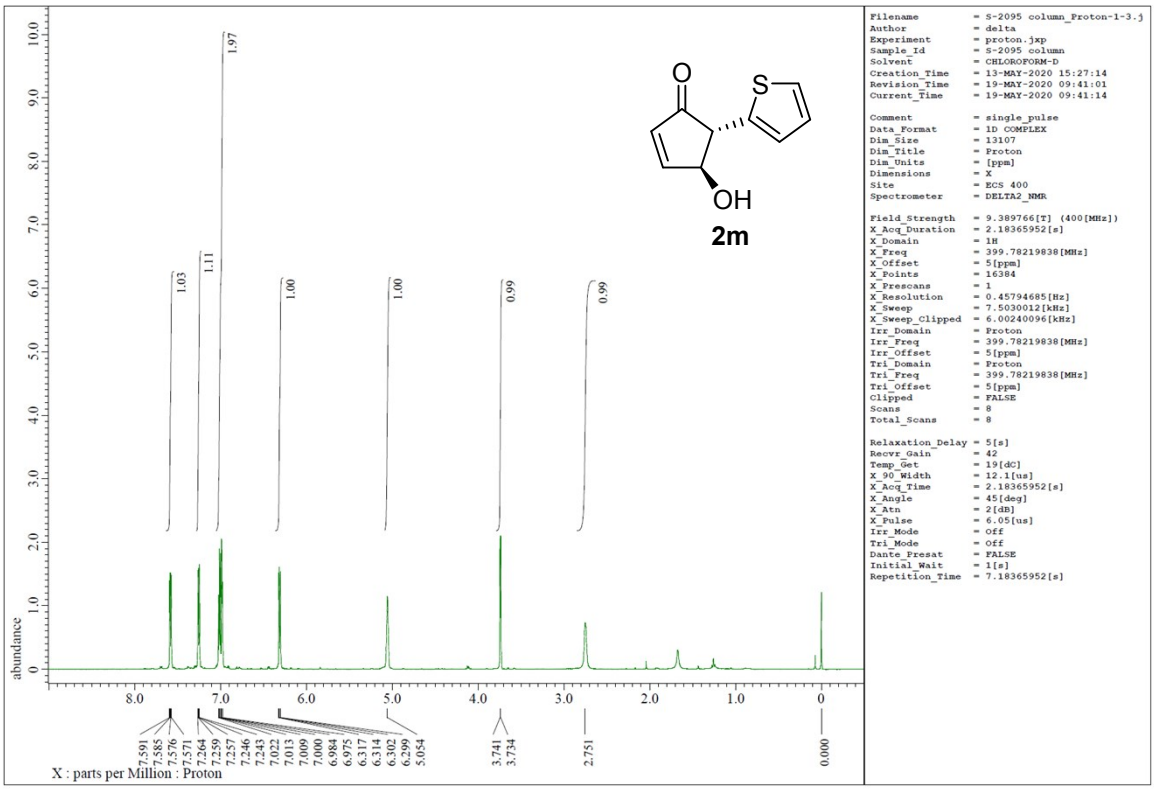

O
Me
OH
2j
abundance
X : parts per Million : Carbon13
Filename = S-1951 column_Carbon-1-3.j
Author = delta
Experiment = carbon.jxp
Sample_Id = S-1951 column
Solvent = CHLOROFORM-D
Creation_Time = 28-FEB-2019 18:01:21
Revision_Time = 28-FEB-2019 18:41:52
Current_Time = 31-MAR-2020 22:20:28
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 129
Total_Scans = 129
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 460.0[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.63[dB]
Irr_Atn_Noe = 21.63[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

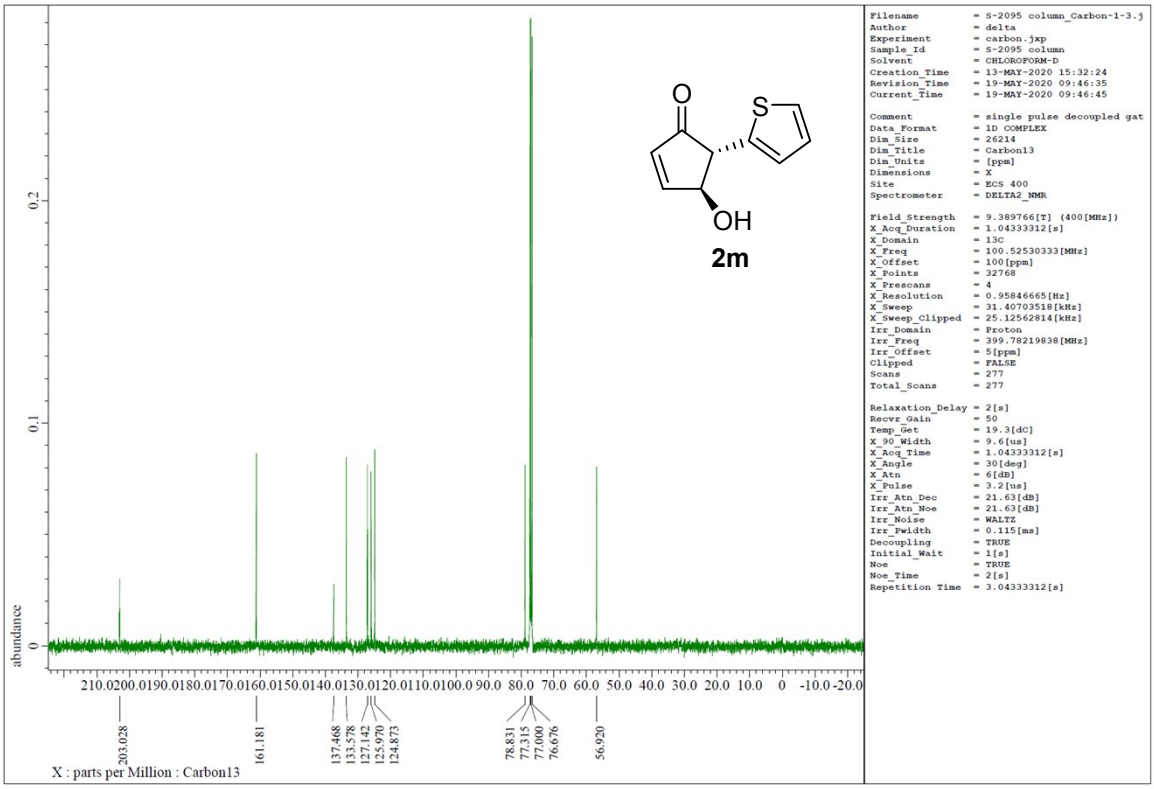

O
OH
2k
3.10
2.03
2.06
1.00
0.95
1.10
1.03
0.91
abundance
X : parts per Million : Proton
Filename = S-2052 column_Proton-1-3.j
Author = delta
Experiment = proton.jxp
Sample_Id = S-2052 column
Solvent = CHLOROFORM-D
Creation_Time = 22-JAN-2020 10:04:39
Revision_Time = 31-MAR-2020 22:25:47
Current_Time = 31-MAR-2020 22:25:55
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 54
Temp_Get = 28.1[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]

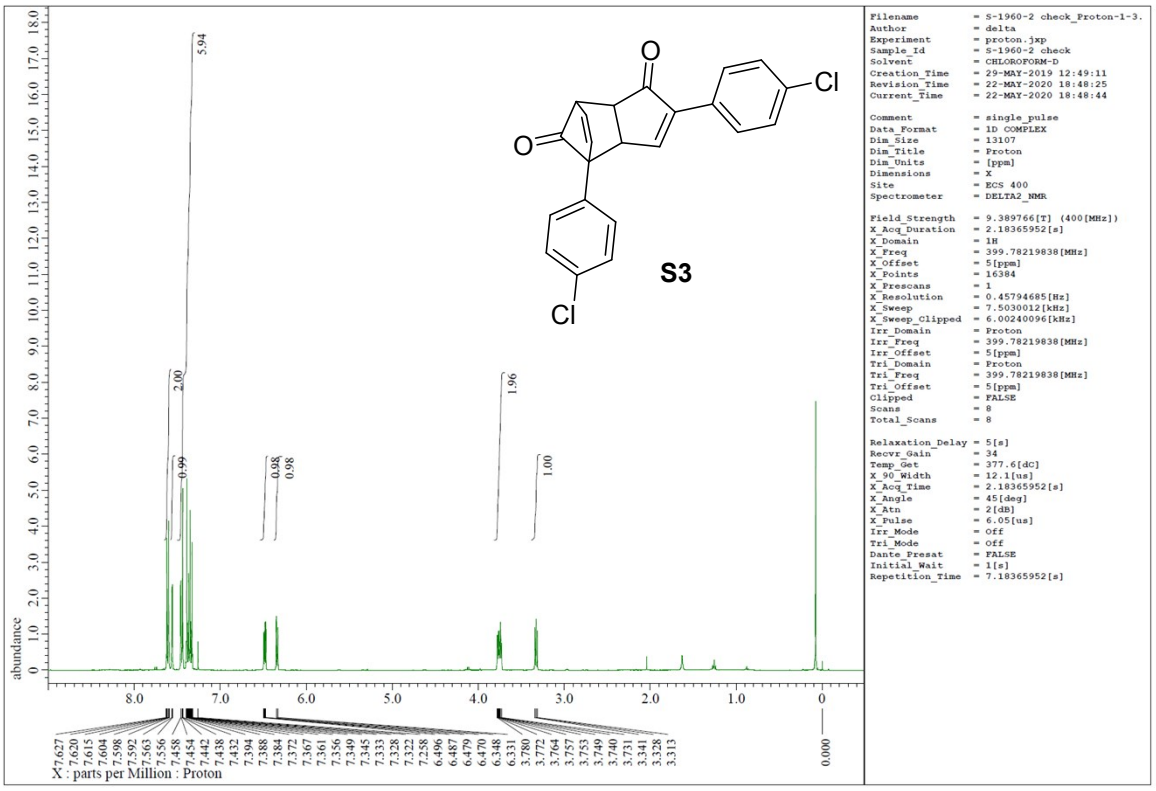

O
OH
2k
abundance
X : parts per Million : Carbon13
Filename = S-2052 column_Carbon-1-3.j
Author = delta
Experiment = carbon.jxp
Sample_Id = S-2052 column
Solvent = CHLOROFORM-D
Creation_Time = 22-JAN-2020 10:16:14
Revision_Time = 31-MAR-2020 22:30:30
Current_Time = 31-MAR-2020 22:31:12
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 235
Total_Scans = 235
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 26.2[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.63[dB]
Irr_Atn_Noe = 21.63[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

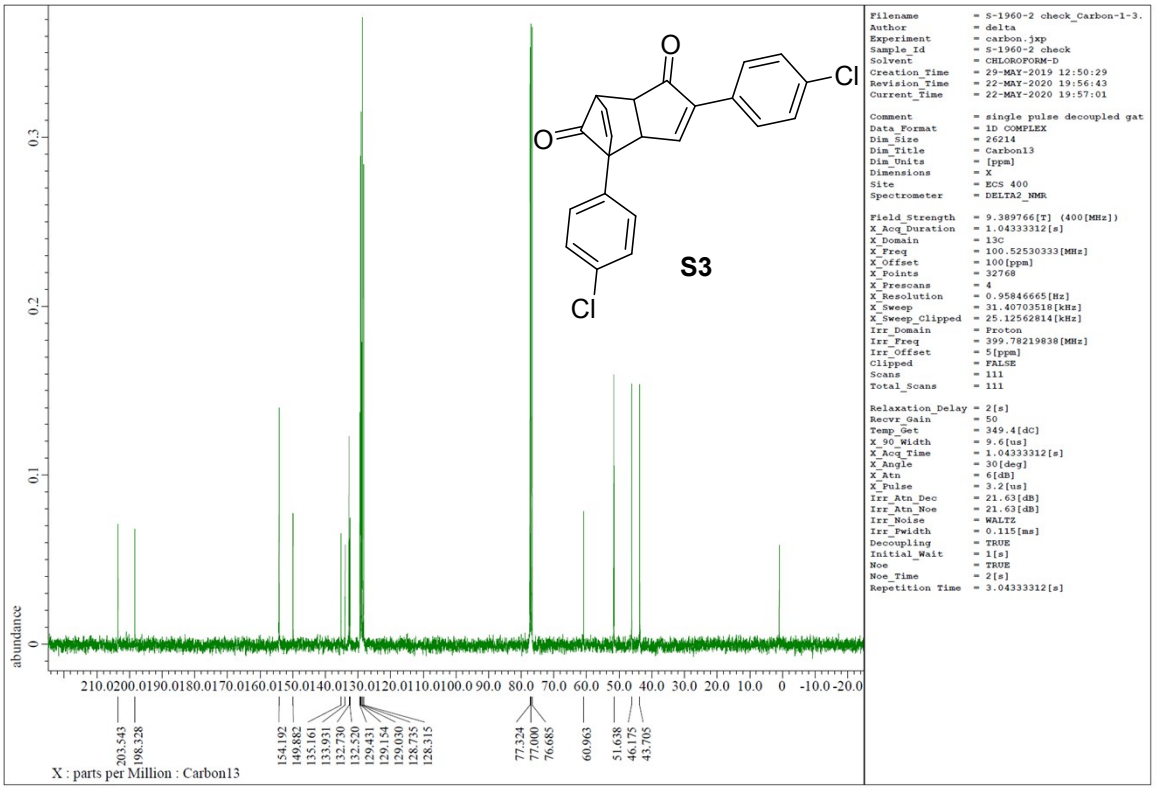

O
OH
2l
abundance
X : parts per Million : Proton
Filename = S-2088 column_Proton-1-3.j
Author = delta
Experiment = proton.jxp
Sample_Id = S-2088 column
Solvent = CHLOROFORM-D
Creation_Time = 12-MAY-2020 13:56:58
Revision_Time = 19-MAY-2020 09:15:23
Current_Time = 19-MAY-2020 09:15:35
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 18.7[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]

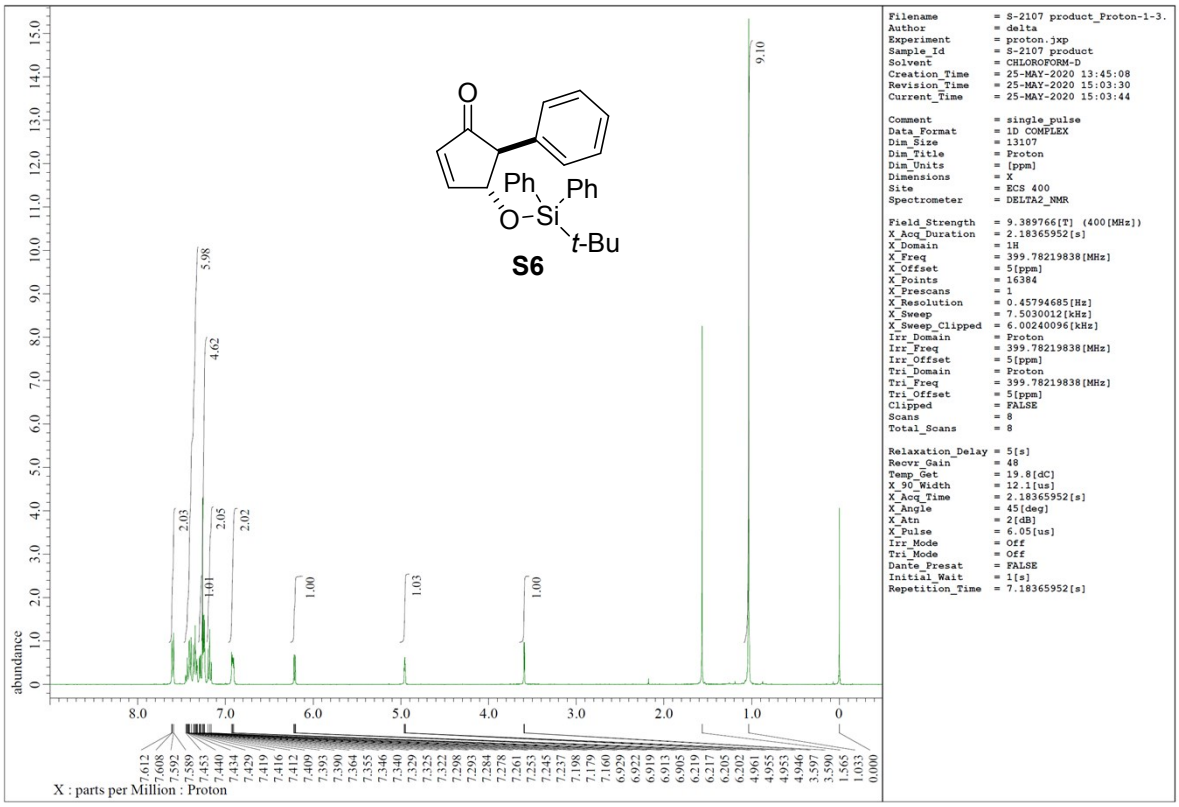

O
OH
2l
abundance
X : parts per Million : Carbon13
Filename = S-2088 column_Carbon-1-3.j
Author = delta
Experiment = carbon.jxp
Sample_Id = S-2088 column
Solvent = CHLOROFORM-D
Creation_Time = 12-MAY-2020 14:01:13
Revision_Time = 19-MAY-2020 09:31:57
Current_Time = 19-MAY-2020 09:32:34
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 169
Total_Scans = 169
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 19.2[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.63[dB]
Irr_Atn_Noe = 21.63[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

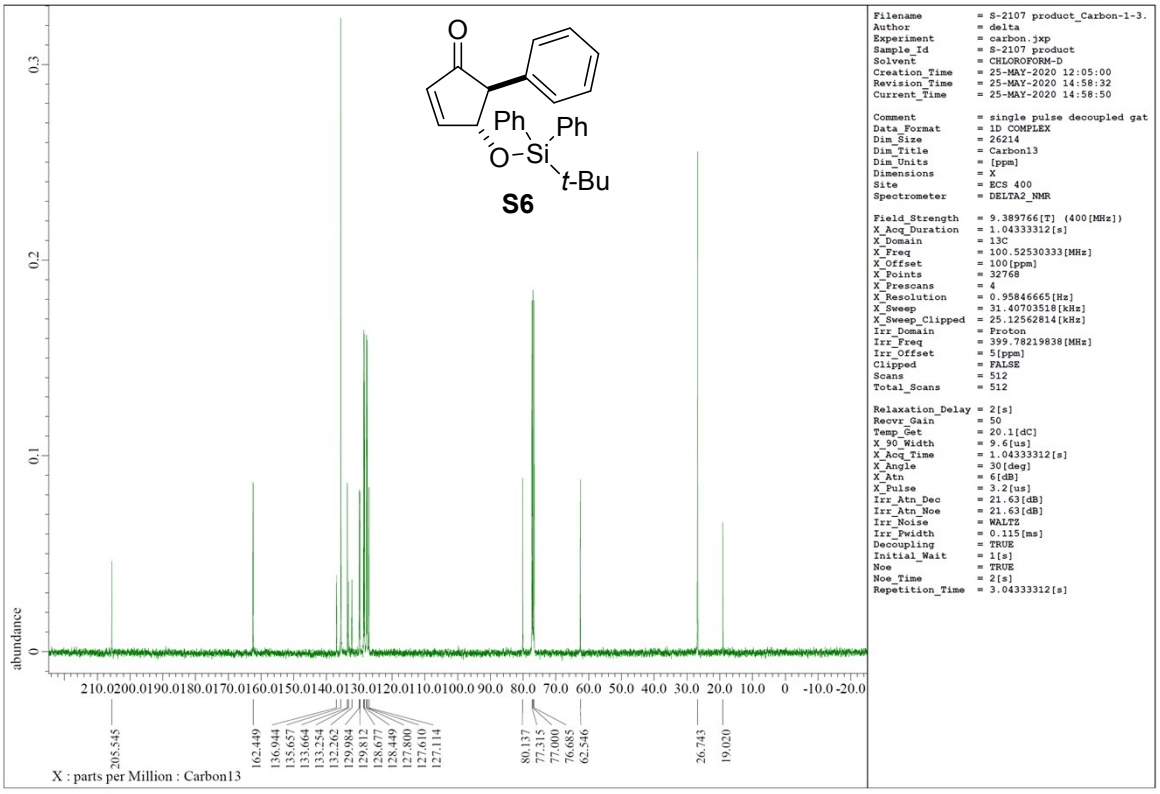

O
S
OH
2m
1.03
1.11
1.97
1.00
1.00
0.99
0.99
abundance
X : parts per Million : Proton
Filename = S-2095 column_Proton-1-3.j
Author = delta
Experiment = proton.jxp
Sample_Id = S-2095 column
Solvent = CHLOROFORM-D
Creation_Time = 13-MAY-2020 15:27:14
Revision_Time = 19-MAY-2020 09:41:01
Current_Time = 19-MAY-2020 09:41:14
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 42
Temp_Get = 19[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]

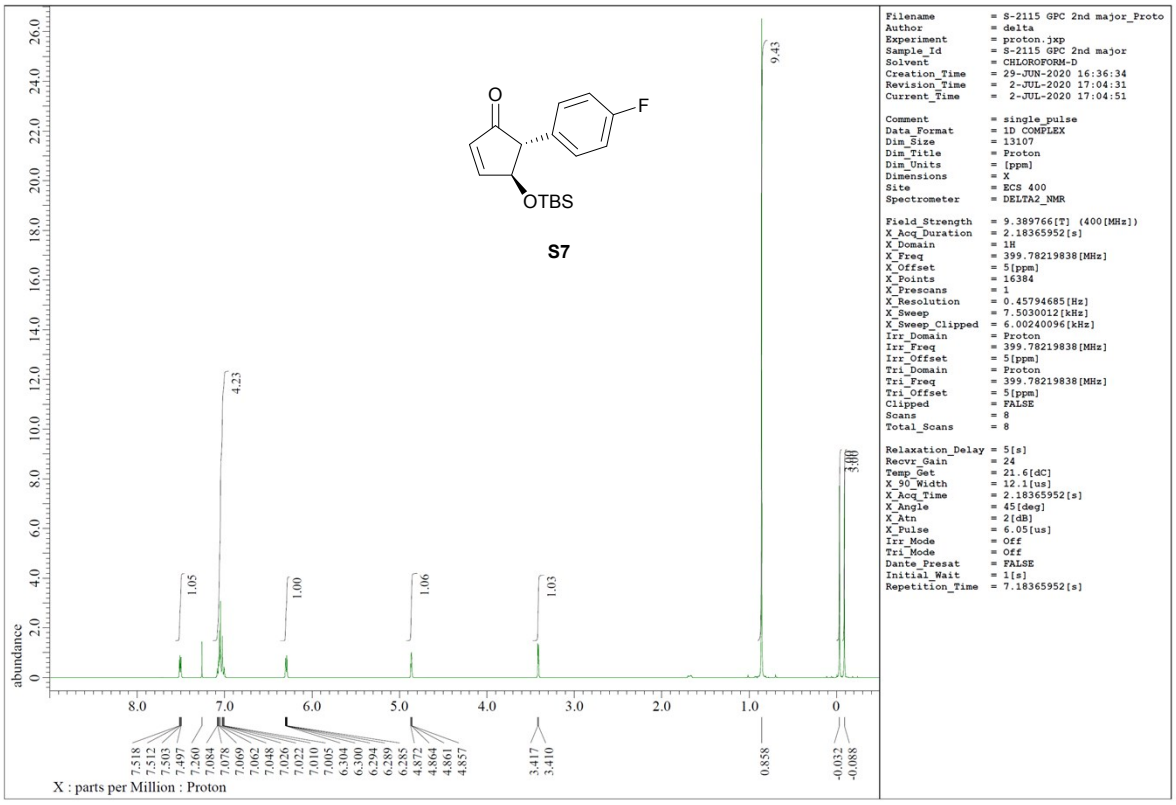

O
S
OH
2m
abundance
X : parts per Million : Carbon13
Filename = S-2095 column_Carbon-1-3.j
Author = delta
Experiment = carbon.jxp
Sample_Id = S-2095 column
Solvent = CHLOROFORM-D
Creation_Time = 13-MAY-2020 15:32:24
Revision_Time = 19-MAY-2020 09:46:35
Current_Time = 19-MAY-2020 09:46:45
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 277
Total_Scans = 277
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 19.3[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.63[dB]
Irr_Atn_Noe = 21.63[dB]
Irr_Noise = WALTZ
Irr_Fwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]


S3
Cl
O
O
Cl
5.94
2.00
0.99
0.98
0.98
1.96
1.00
abundance
X : parts per Million : Proton
Filename = S-1960-2 check_Proton-1-3.
Author = delta
Experiment = proton.jxp
Sample_Id = S-1960-2 check
Solvent = CHLOROFORM-D
Creation_Time = 29-MAY-2019 12:49:11
Revision_Time = 22-MAY-2020 18:48:25
Current_Time = 22-MAY-2020 18:48:44
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 34
Temp_Get = 377.6[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]

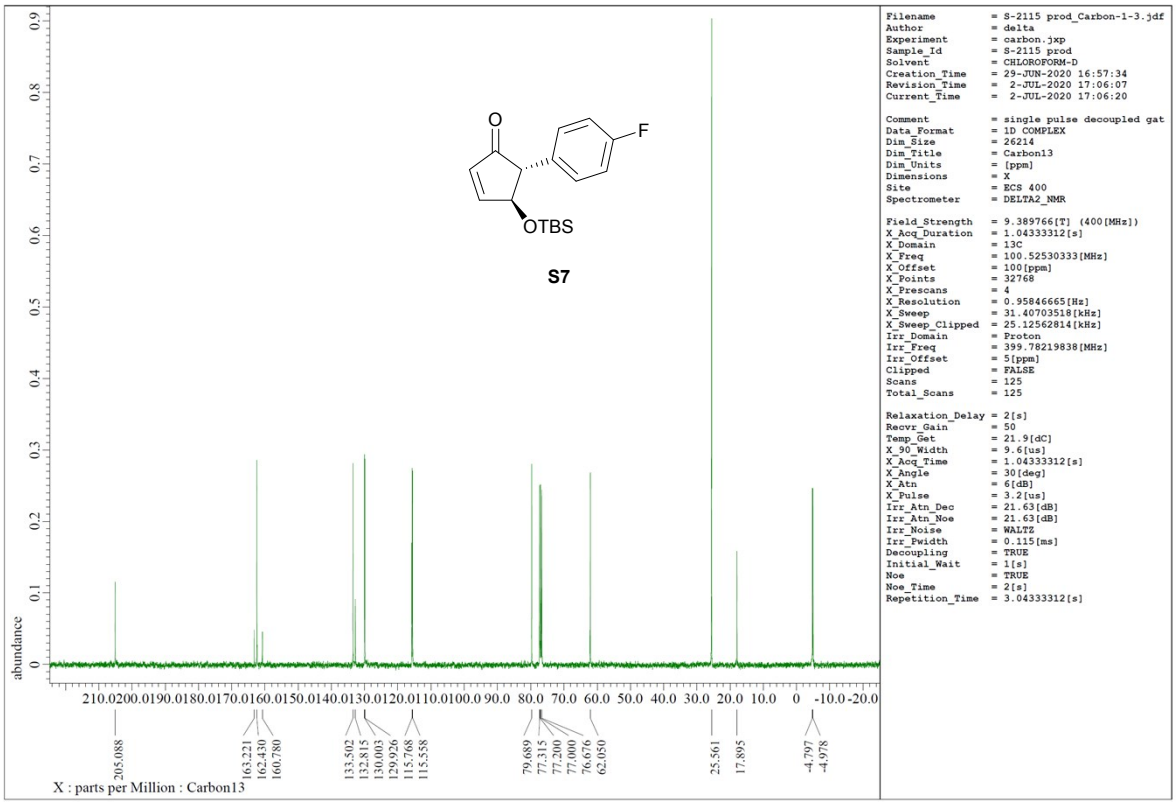

S3
Cl
O
O
Cl
abundance
X : parts per Million : Carbon13
Filename = S-1960-2 check_Carbon-1-3.
Author = delta
Experiment = carbon.jxp
Sample_Id = S-1960-2 check
Solvent = CHLOROFORM-D
Creation_Time = 29-MAY-2019 12:50:29
Revision_Time = 22-MAY-2020 19:56:43
Current_Time = 22-MAY-2020 19:57:01
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 111
Total_Scans = 111
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 349.4[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.63[dB]
Irr_Atn_Noe = 21.63[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]


S6
abundance
X : parts per Million : Proton
Filename = S-2107 product_Proton-1-3.
Author = delta
Experiment = proton.jxp
Sample_Id = S-2107 product
Solvent = CHLOROFORM-D
Creation_Time = 25-MAY-2020 13:45:08
Revision_Time = 25-MAY-2020 15:03:30
Current_Time = 25-MAY-2020 15:03:44
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 48
Temp_Get = 19.8[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]

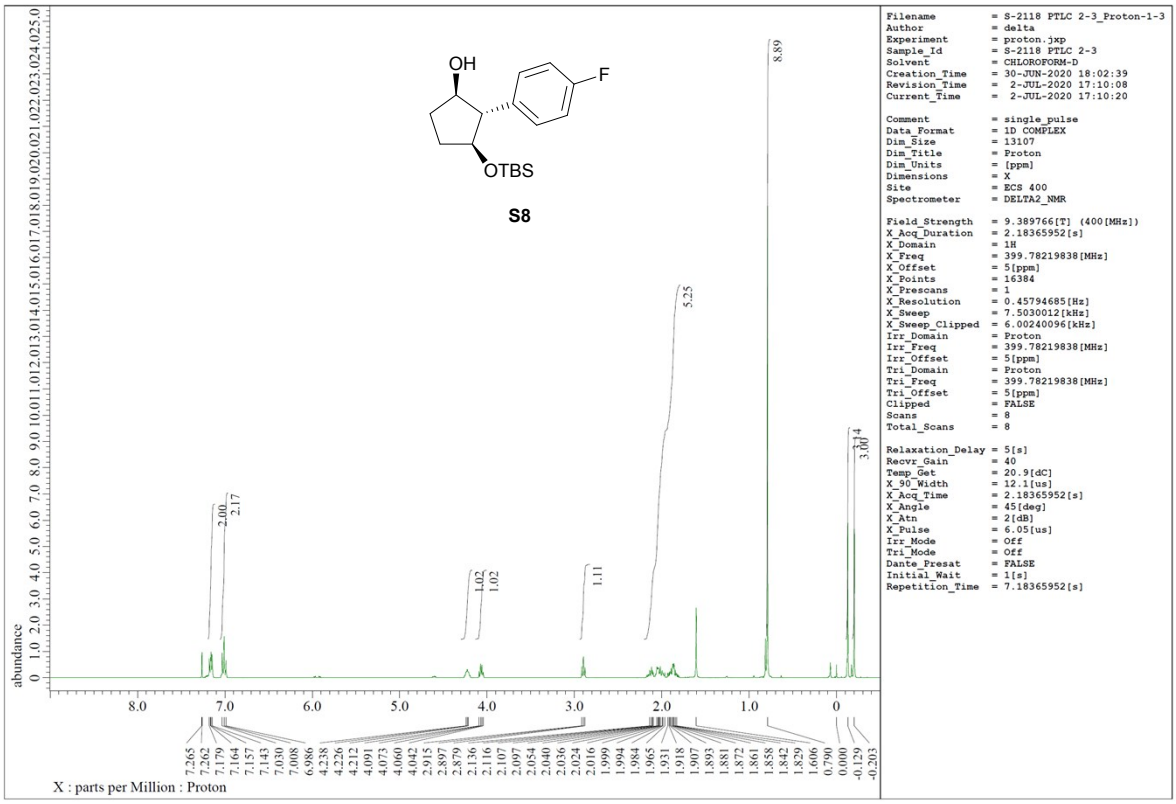

S6
abundance
X : parts per Million : Carbon13
Filename = S-2107 product_Carbon-1-3.
Author = delta
Experiment = carbon.jxp
Sample_Id = S-2107 product
Solvent = CHLOROFORM-D
Creation_Time = 25-MAY-2020 12:05:00
Revision_Time = 25-MAY-2020 14:58:32
Current_Time = 25-MAY-2020 14:58:50
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 512
Total_Scans = 512
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 20.1[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.63[dB]
Irr_Atn_Noe = 21.63[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]



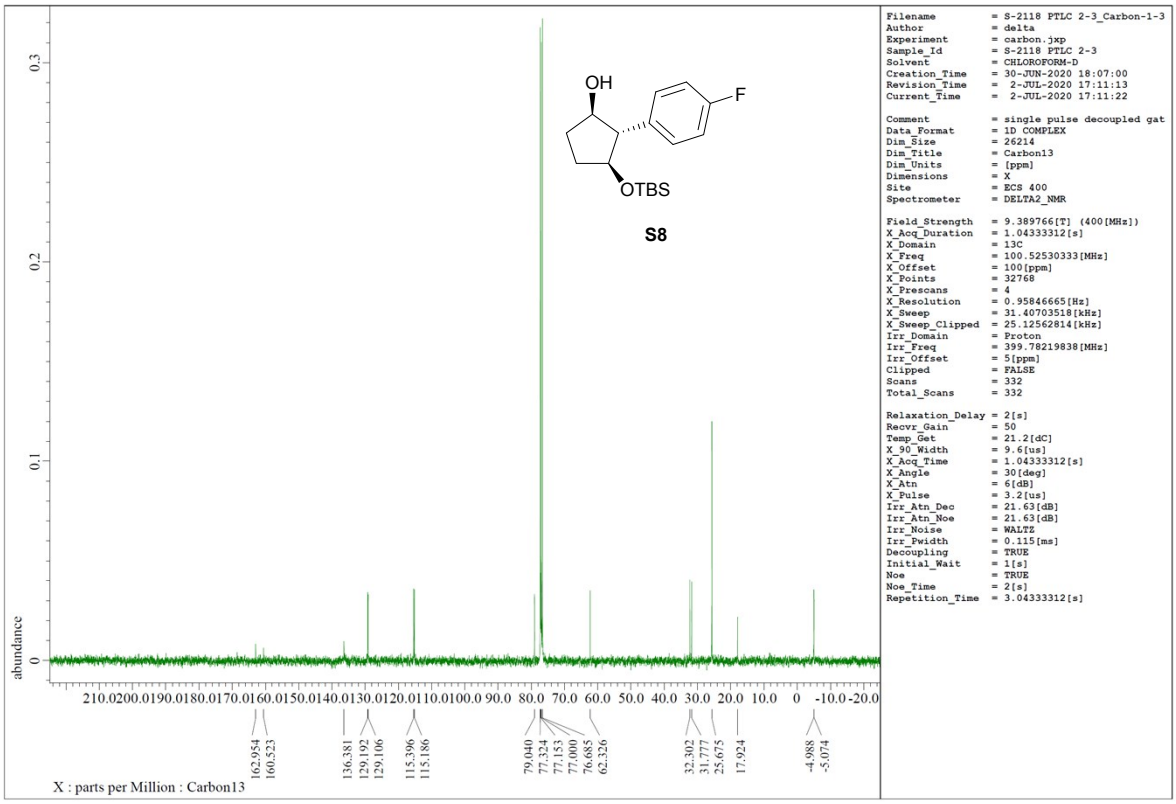

```
Filename          = S-2115 GPC 2nd major_Proto
Author            = delta
Experiment        = proton.jxp
Sample_Id         = S-2115 GPC 2nd major
Solvent           = CHLOROFORM-D
Creation_Time     = 29-JUN-2020 16:36:34
Revision_Time     =  2-JUL-2020 17:04:31
Current_Time      =  2-JUL-2020 17:04:51

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 24
Temp_Get          = 21.6[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



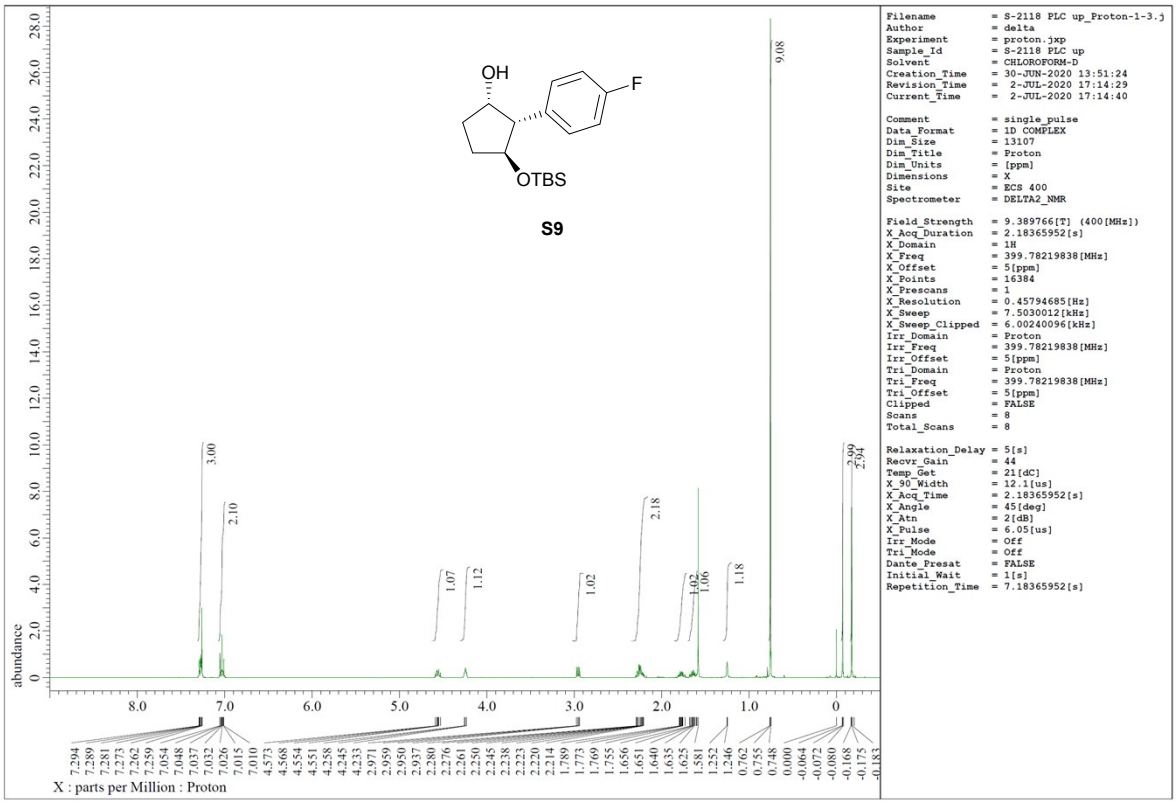

```
Filename          = S-2115 prod_Carbon-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = S-2115 prod
Solvent           = CHLOROFORM-D
Creation_Time     = 29-JUN-2020 16:57:34
Revision_Time     =  2-JUL-2020 17:06:07
Current_Time      =  2-JUL-2020 17:06:20

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 125
Total_Scans       = 125

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 21.9[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```

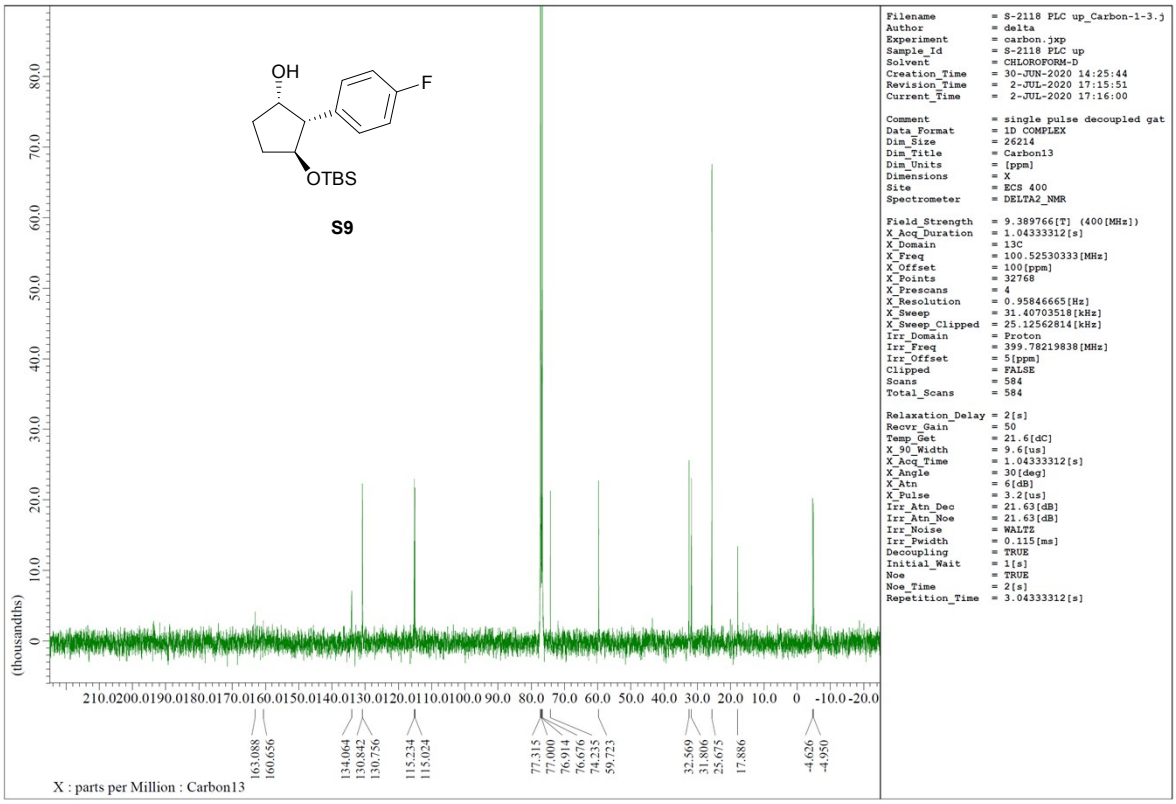


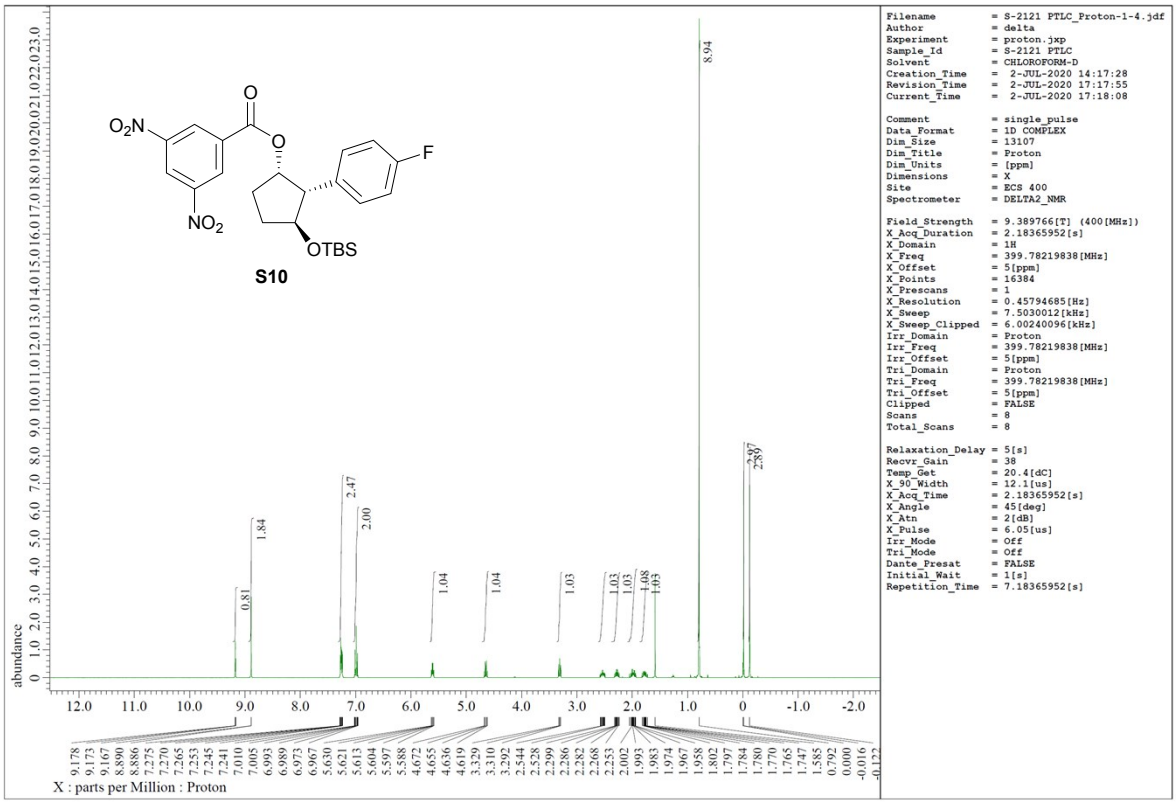

```
Filename          = S-2118 PTLC 2-3_Proton-1-3
Author            = delta
Experiment        = proton.jxp
Sample_Id         = S-2118 PTLC 2-3
Solvent           = CHLOROFORM-D
Creation_Time     = 30-JUN-2020 18:02:39
Revision_Time     =  2-JUL-2020 17:10:08
Current_Time      =  2-JUL-2020 17:10:20

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 20.9[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

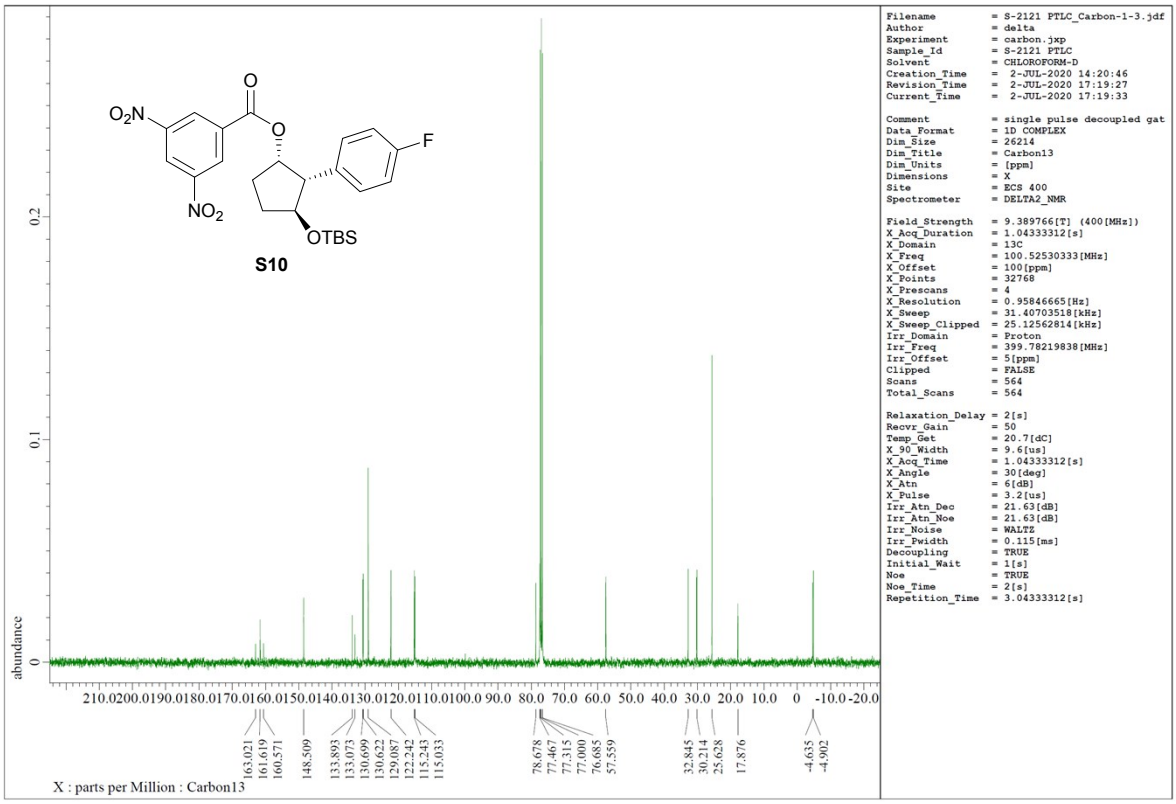



```
Filename          = S-2118 PTLC 2-3_Carbon-1-3
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = S-2118 PTLC 2-3
Solvent           = CHLOROFORM-D
Creation_Time     = 30-JUN-2020 18:07:00
Revision_Time     =  2-JUL-2020 17:11:13
Current_Time      =  2-JUL-2020 17:11:22

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 332
Total_Scans       = 332

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 21.2[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```

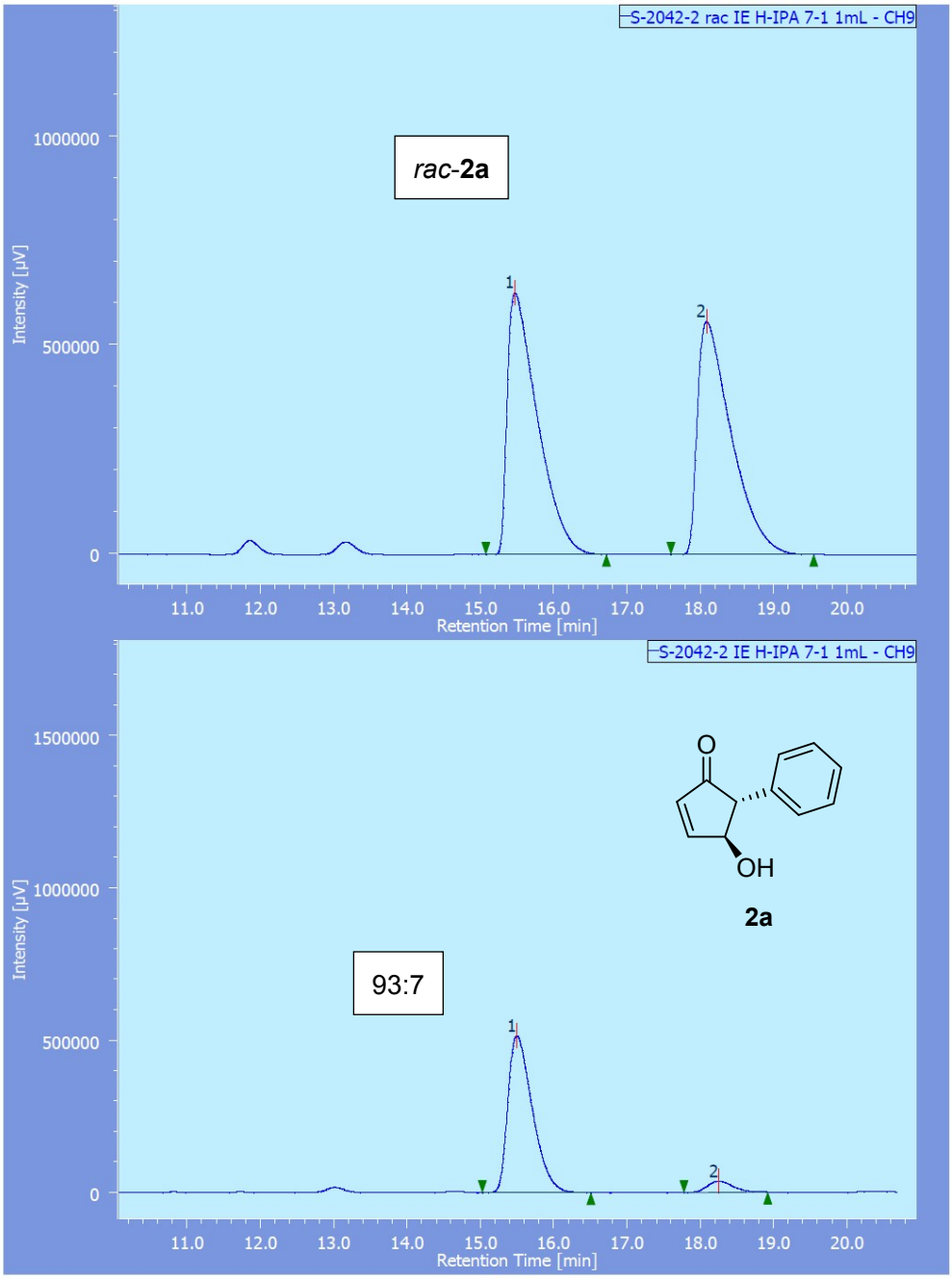

OH
F
OTBS
S9
abundance
X : parts per Million : Proton
Filename = S-2118 PLC up_Proton-1-3.j
Author = delta
Experiment = proton.jxp
Sample_Id = S-2118 PLC up
Solvent = CHLOROFORM-D
Creation_Time = 30-JUN-2020 13:51:24
Revision_Time = 2-JUL-2020 17:14:29
Current_Time = 2-JUL-2020 17:14:40
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 44
Temp_Get = 21[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]


OH
F
OTBS
S9
(thousandths)
X : parts per Million : Carbon13
Filename = S-2118 PLC up_Carbon-1-3.j
Author = delta
Experiment = carbon.jxp
Sample_Id = S-2118 PLC up
Solvent = CHLOROFORM-D
Creation_Time = 30-JUN-2020 14:25:44
Revision_Time = 2-JUL-2020 17:15:51
Current_Time = 2-JUL-2020 17:16:00
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 584
Total_Scans = 584
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 21.6[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.63[dB]
Irr_Atn_Noe = 21.63[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]


S10
abundance
X : parts per Million : Proton
Filename = S-2121 PTLC_Proton-1-4.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = S-2121 PTLC
Solvent = CHLOROFORM-D
Creation_Time = 2-JUL-2020 14:17:28
Revision_Time = 2-JUL-2020 17:17:55
Current_Time = 2-JUL-2020 17:18:08
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 38
Temp_Get = 20.4[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]


S10
abundance
X : parts per Million : Carbon13
Filename = S-2121 PTLC_Carbon-1-3.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = S-2121 PTLC
Solvent = CHLOROFORM-D
Creation_Time = 2-JUL-2020 14:20:46
Revision_Time = 2-JUL-2020 17:19:27
Current_Time = 2-JUL-2020 17:19:33
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 564
Total_Scans = 564
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 20.7[dC]
X_90_Width = 9.6[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3.2[us]
Irr_Atn_Dec = 21.63[dB]
Irr_Atn_Noe = 21.63[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

## 6. HPLC spectra




Channel & Peak Information Table

Chromatogram Name S-2042-2 rac IE H-IPA 7-1 1mL-CH9

Sample Name

Channel Name 230.0nm

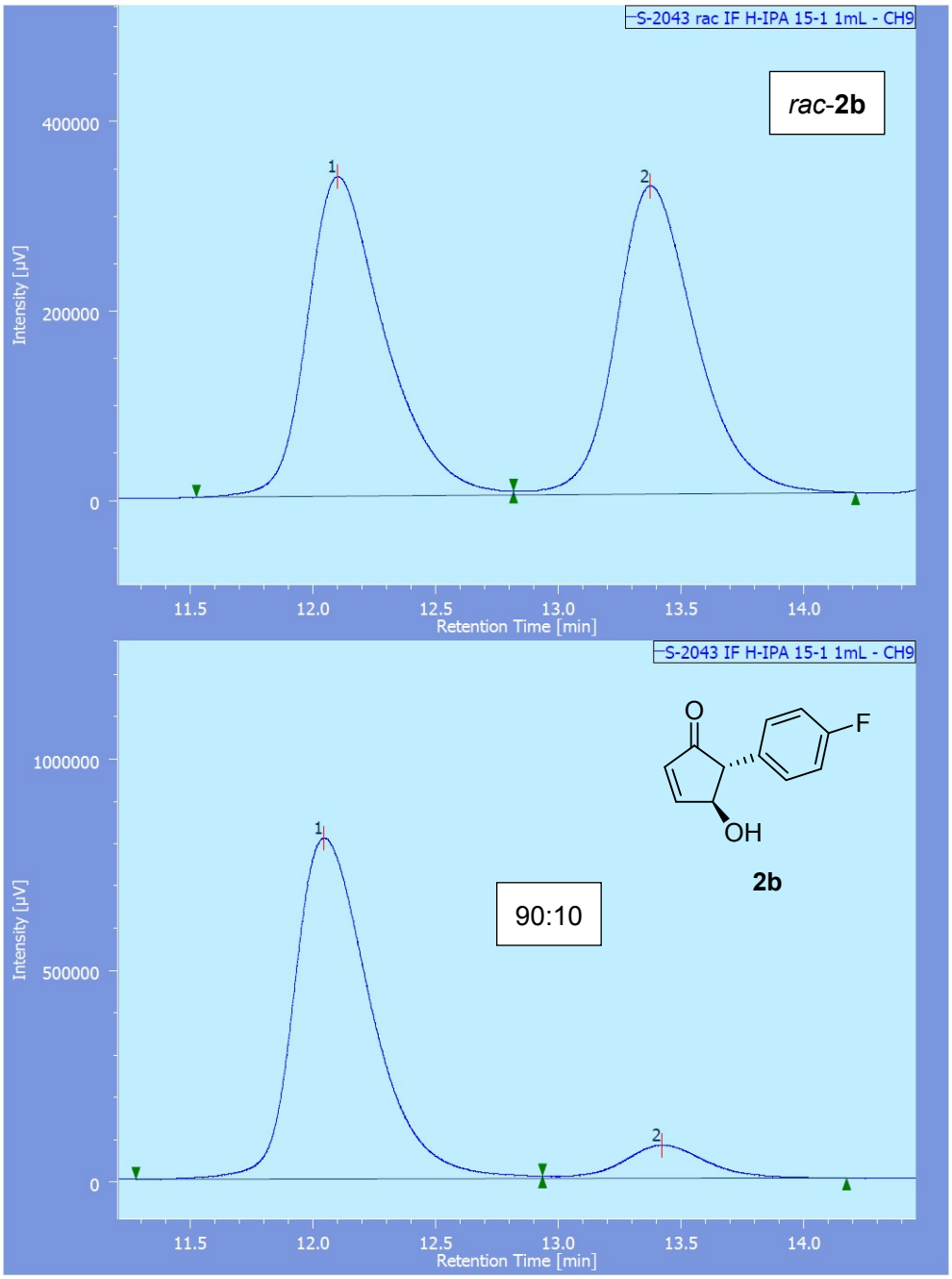
| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 15.468 | 17788014 | 626724 | 49.886 | 52.901 | N/A | 6915 | 3.326 | 2.533 | |
| 2 | Unknown | 9 | 18.082 | 17869287 | 557991 | 50.114 | 47.099 | N/A | 7561 | N/A | 2.371 | |

Chromatogram Name S-2042-2 IE H-IPA 7-1 1mL-CH9

Sample Name

Channel Name 210.0nm

| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 15.495 | 11890450 | 514674 | 92.986 | 93.339 | N/A | 10412 | 4.418 | 1.583 | |
| 2 | Unknown | 9 | 18.247 | 896915 | 36730 | 7.014 | 6.661 | N/A | 12941 | N/A | 1.214 | |




Channel & Peak Information Table

Chromatogram Name S-2043 rac IF H-IPA 15-1 1mL-CH9

Sample Name

Channel Name 210.0nm

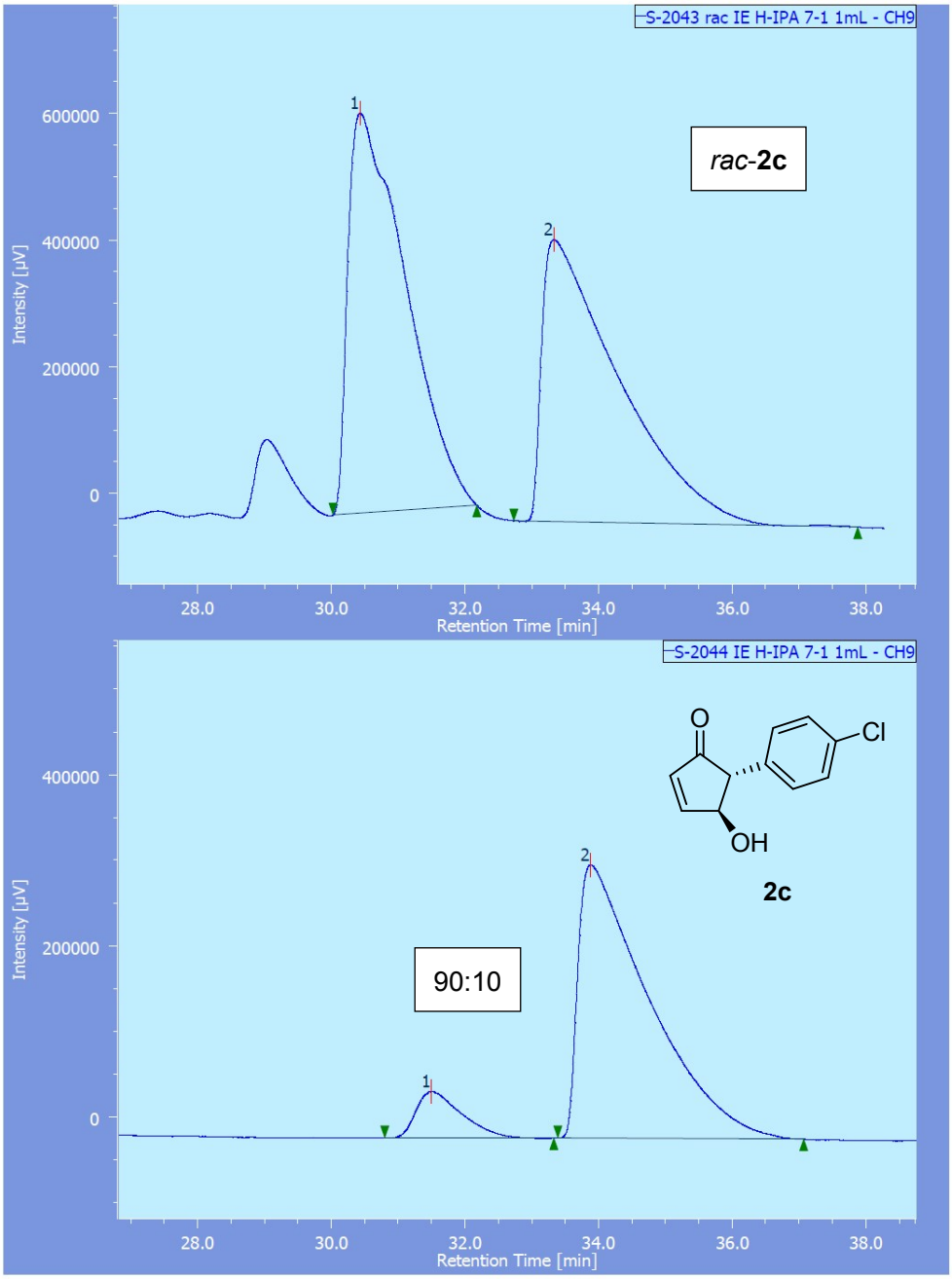
| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 12.100 | 7396853 | 337280 | 50.134 | 50.921 | N/A | 7630 | 2.265 | 1.416 | |
| 2 | Unknown | 9 | 13.373 | 7357273 | 325081 | 49.866 | 49.079 | N/A | 8705 | N/A | 1.229 | |

Chromatogram Name S-2043 IF H-IPA 15-1 1mL-CH9

Sample Name

Channel Name 210.0nm

| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 12.045 | 17541947 | 805905 | 90.337 | 91.071 | N/A | 7648 | 2.422 | 1.434 | |
| 2 | Unknown | 9 | 13.422 | 1876488 | 79019 | 9.663 | 8.929 | N/A | 8310 | N/A | N/A | |




Channel & Peak Information Table

Chromatogram Name S-2043 rac IE H-IPA 7-1 1mL-CH9

Sample Name

Channel Name 210.0nm

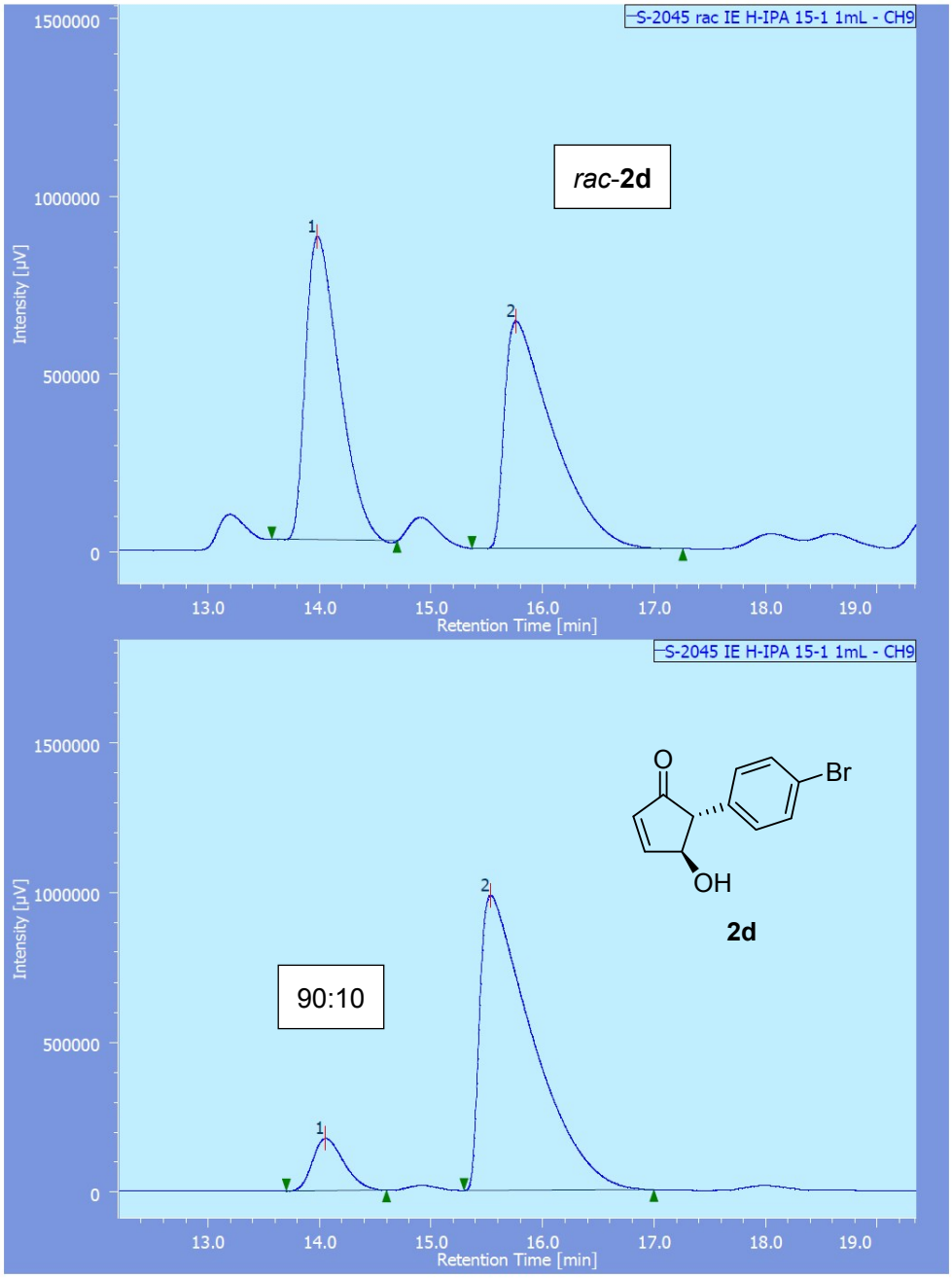
| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 30.425 | 37854021 | 631571 | 51.602 | 58.694 | N/A | 5372 | 1.570 | 2.863 | |
| 2 | Unknown | 9 | 33.330 | 35504341 | 444461 | 48.398 | 41.306 | N/A | 4227 | N/A | 4.474 | |

Chromatogram Name S-2044 IE H-IPA 7-1 1mL-CH9

Sample Name

Channel Name 210.0nm

| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 31.493 | 2558768 | 54230 | 9.818 | 14.528 | N/A | 10339 | 1.530 | 1.693 | |
| 2 | Unknown | 9 | 33.877 | 23503838 | 319039 | 90.182 | 85.472 | N/A | 5164 | N/A | 3.921 | |




Channel & Peak Information Table

Chromatogram Name S-2045 rac IE H-IPA 15-1 1mL-CH9

Sample Name

Channel Name 210.0nm

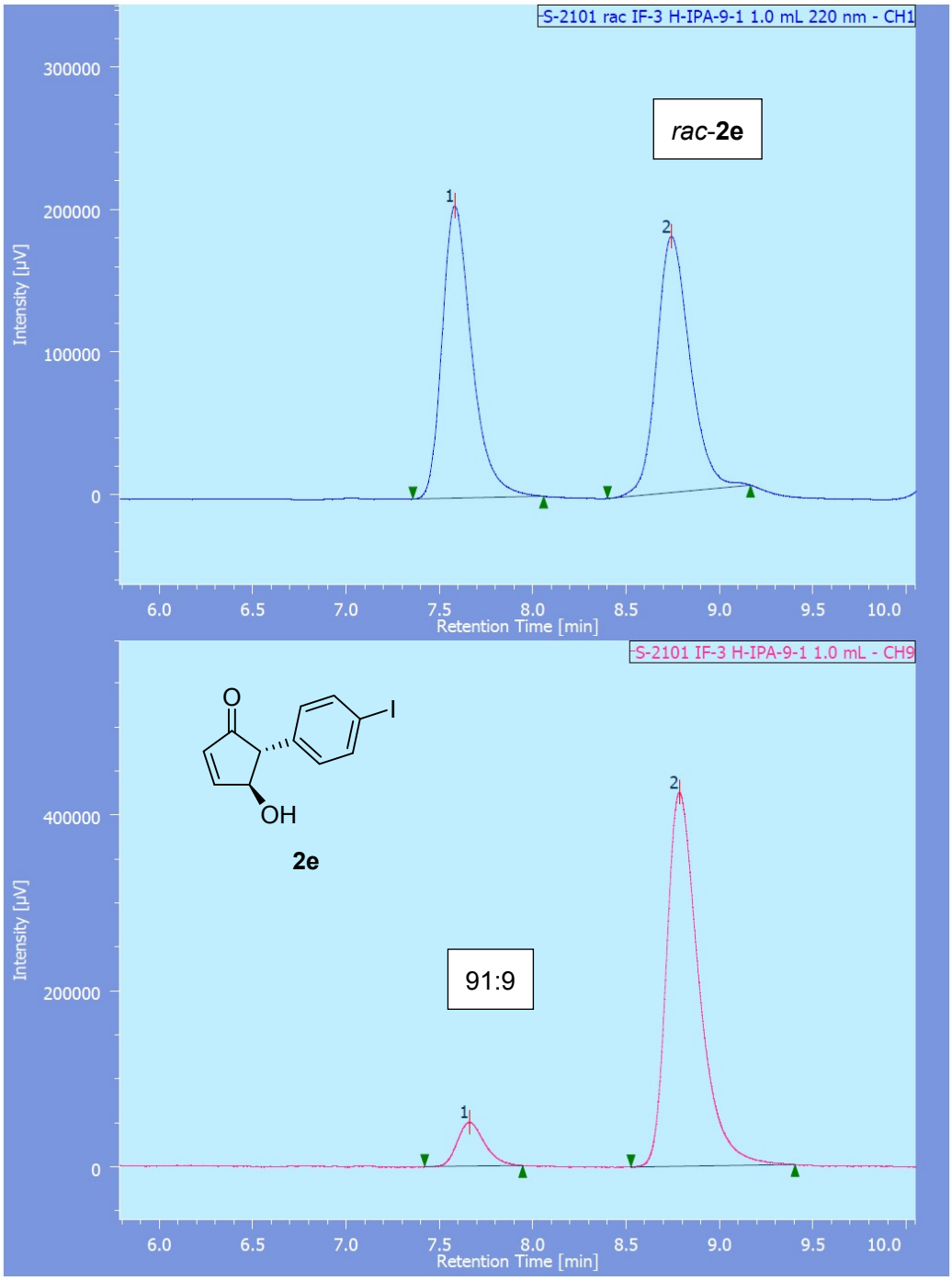
| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 13.978 | 17825152 | 852798 | 47.889 | 57.146 | N/A | 10063 | 2.667 | 1.657 | |
| 2 | Unknown | 9 | 15.758 | 19396859 | 639507 | 52.111 | 42.854 | N/A | 6517 | N/A | 2.835 | |

Chromatogram Name S-2045 IE H-IPA 15-1 1mL-CH9

Sample Name

Channel Name 210.0nm

| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 14.052 | 3415134 | 174750 | 9.521 | 15.079 | N/A | 11851 | 2.171 | 1.323 | |
| 2 | Unknown | 9 | 15.530 | 32453372 | 984115 | 90.479 | 84.921 | N/A | 5349 | N/A | 3.412 | |




Channel & Peak Information Table

Chromatogram Name S-2101 rac IF-3 H-IPA-9-1 1.0 mL 220 nm-CH1

Sample Name

Channel Name UV-2075

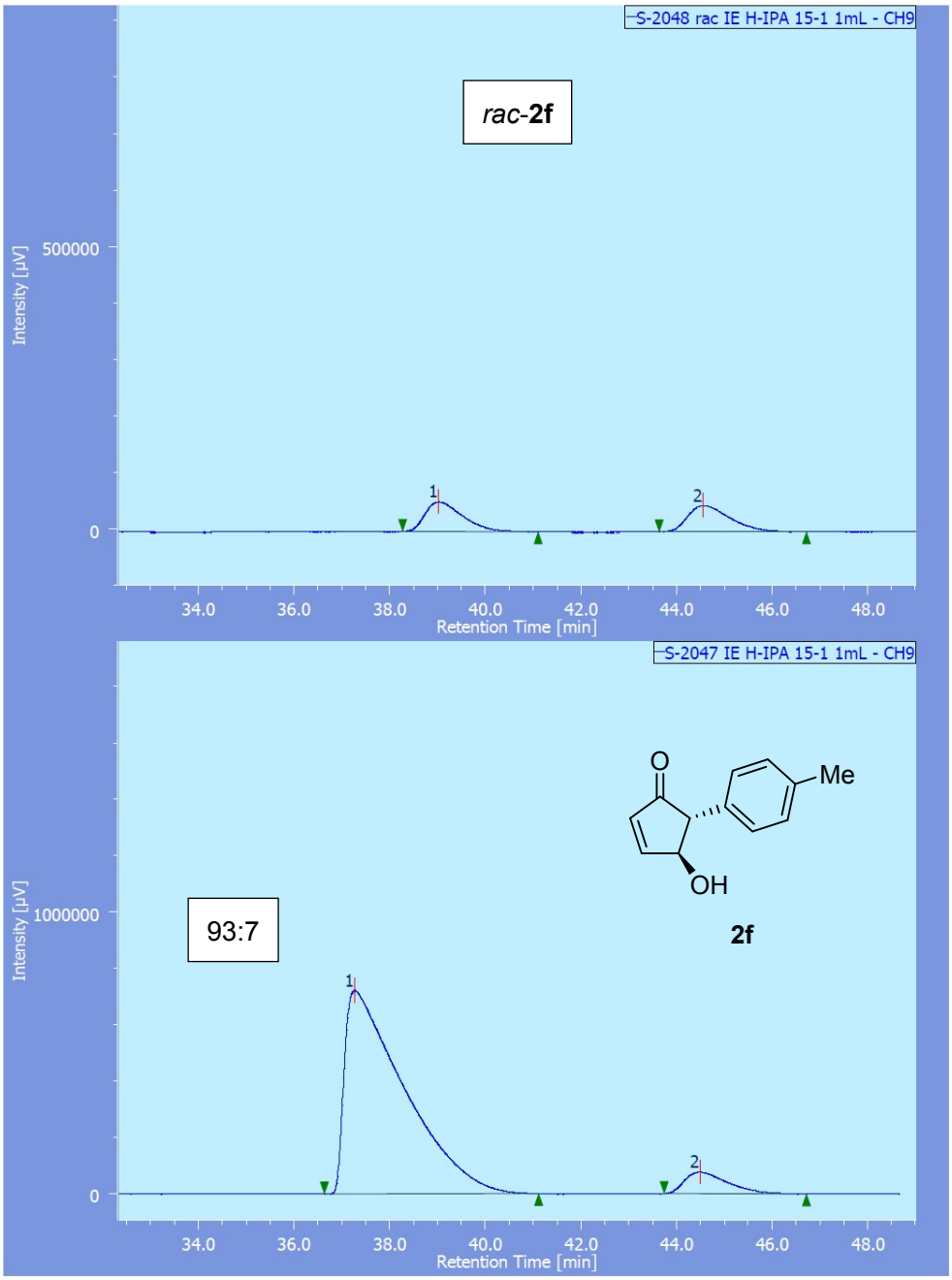
| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 1 | 7.583 | 2264918 | 205115 | 50.536 | 53.281 | N/A | 11776 | 3.905 | 1.363 | |
| 2 | Unknown | 1 | 8.742 | 2216842 | 179856 | 49.464 | 46.719 | N/A | 12298 | N/A | 1.195 | |

Chromatogram Name S-2101 IF-3 H-IPA-9-1 1.0 mL-CH9

Sample Name

Channel Name 210.0nm

| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 7.660 | 507536 | 49940 | 9.024 | 10.508 | N/A | 13450 | 3.972 | 1.287 | |
| 2 | Unknown | 9 | 8.783 | 5117037 | 425339 | 90.976 | 89.492 | N/A | 13446 | N/A | 1.498 | |




Channel & Peak Information Table

Chromatogram Name S-2048 rac IE H-IPA 15-1 1mL-CH9

Sample Name

Channel Name 210.0nm

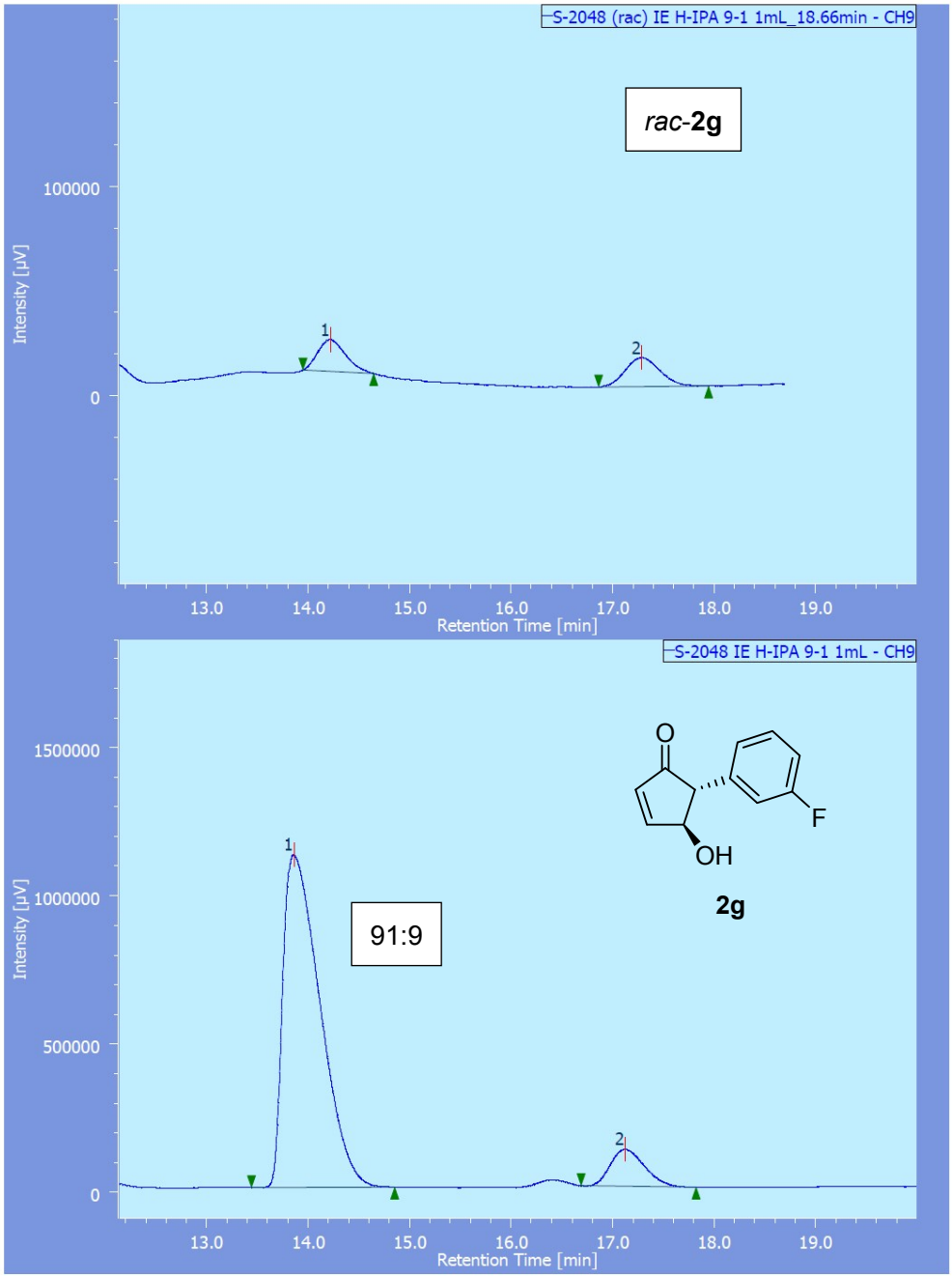
| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 39.020 | 2912879 | 52982 | 50.046 | 52.968 | N/A | 11799 | 3.628 | 1.583 | |
| 2 | Unknown | 9 | 44.550 | 2907554 | 47045 | 49.954 | 47.032 | N/A | 12098 | N/A | 1.535 | |

Chromatogram Name S-2047 IE H-IPA 15-1 1mL-CH9

Sample Name

Channel Name 210.0nm

| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 37.267 | 62774916 | 721947 | 92.864 | 90.434 | N/A | 4353 | 3.690 | 3.966 | |
| 2 | Unknown | 9 | 44.480 | 4823582 | 76369 | 7.136 | 9.566 | N/A | 11479 | N/A | 1.677 | |




Channel & Peak Information Table

Chromatogram Name S-2048 (rac) IE H-IPA 9-1 1mL_18.66min-CH9

Sample Name

Channel Name 210.0nm

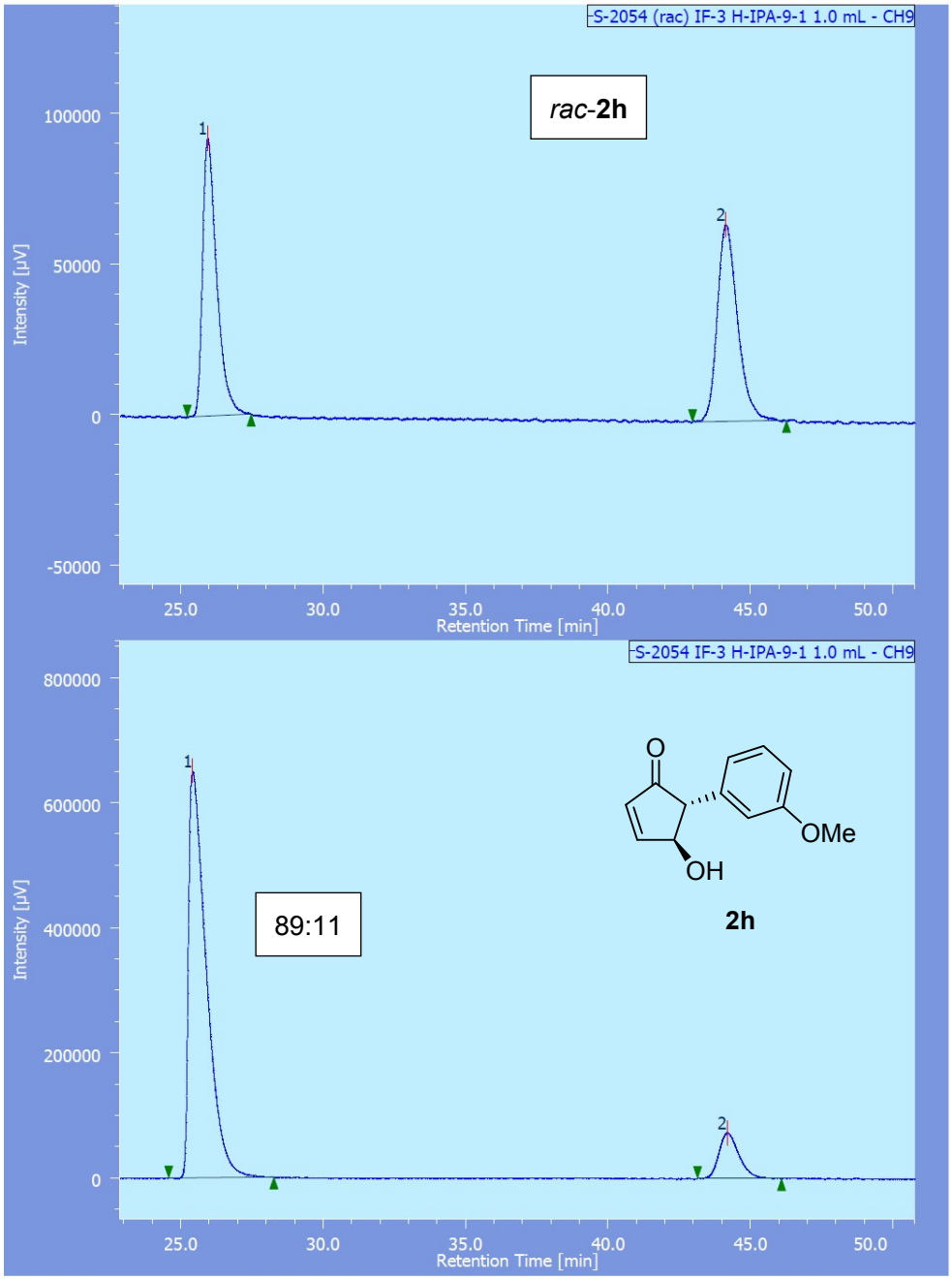
| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 14.217 | 290944 | 15164 | 47.062 | 52.347 | N/A | 12175 | 5.363 | 1.250 | |
| 2 | Unknown | 9 | 17.282 | 327268 | 13804 | 52.938 | 47.653 | N/A | 12013 | N/A | 1.121 | |

Chromatogram Name S-2048 IE H-IPA 9-1 1mL-CH9

Sample Name

Channel Name 210.0nm

| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 13.857 | 28410806 | 1120937 | 90.650 | 89.986 | N/A | 6531 | 4.978 | 2.025 | |
| 2 | Unknown | 9 | 17.120 | 2930269 | 124738 | 9.350 | 10.014 | N/A | 11863 | N/A | 1.266 | |




Channel & Peak Information Table

Chromatogram Name S-2054 (rac) IF-3 H-IPA-9-1 1.0 mL-CH9

Sample Name

Channel Name 210.0nm

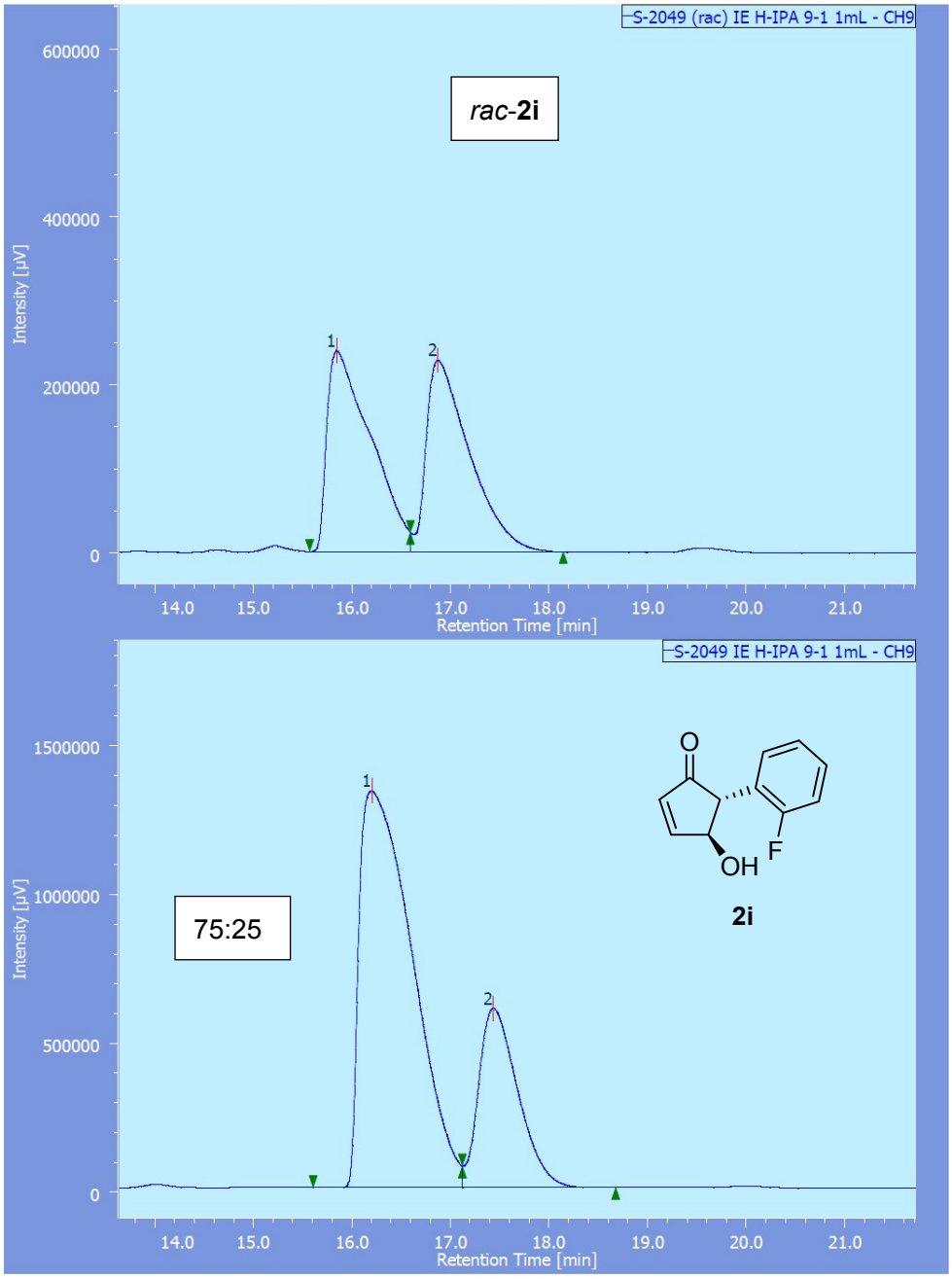
| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 25.952 | 3159488 | 92302 | 49.450 | 58.571 | N/A | 14582 | 17.179 | 1.667 | |
| 2 | Unknown | 9 | 44.133 | 3229722 | 65288 | 50.550 | 41.429 | N/A | 19543 | N/A | 1.313 | |

Chromatogram Name S-2054 IF-3 H-IPA-9-1 1.0 mL-CH9

Sample Name

Channel Name 210.0nm

| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 25.423 | 28110248 | 650365 | 88.641 | 89.906 | N/A | 8409 | 15.814 | 2.757 | |
| 2 | Unknown | 9 | 44.177 | 3602122 | 73017 | 11.359 | 10.094 | N/A | 19389 | N/A | 1.302 | |




Channel & Peak Information Table

Chromatogram Name S-2049 (rac) IE H-IPA 9-1 1mL-CH9

Sample Name

Channel Name 240.0nm

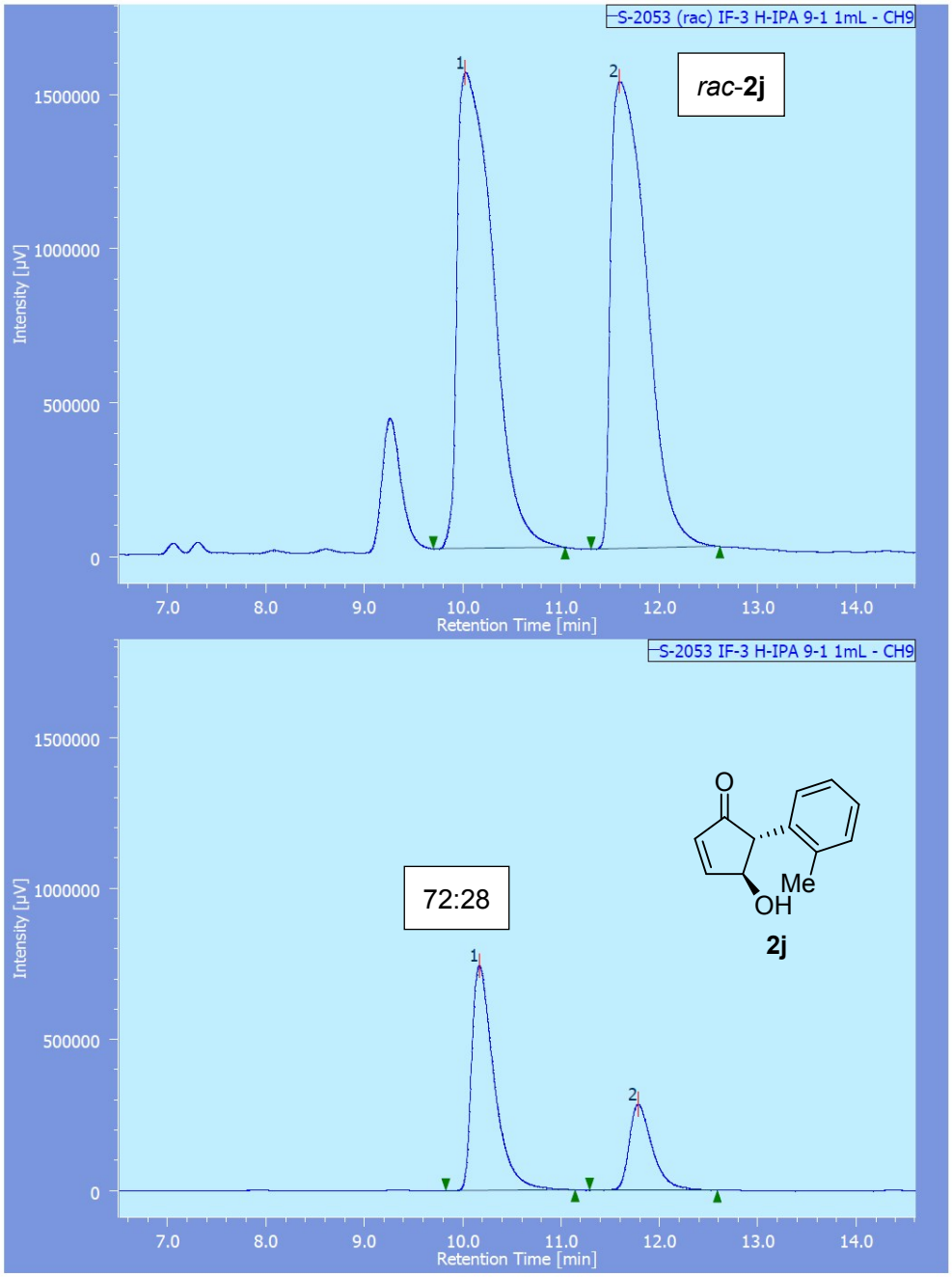
| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 15.845 | 7338133 | 238811 | 51.434 | 51.214 | N/A | 5067 | 1.228 | N/A | |
| 2 | Unknown | 9 | 16.870 | 6928993 | 227489 | 48.566 | 48.786 | N/A | 7426 | N/A | N/A | |

Chromatogram Name S-2049 IE H-IPA 9-1 1mL-CH9

Sample Name

Channel Name 210.0nm

| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 16.203 | 49762201 | 1331870 | 74.934 | 68.893 | N/A | 3868 | 1.394 | N/A | |
| 2 | Unknown | 9 | 17.433 | 16645460 | 601384 | 25.066 | 31.107 | N/A | 9206 | N/A | N/A | |




Channel & Peak Information Table

Chromatogram Name S-2053 (rac) IF-3 H-IPA 9-1 1mL-CH9

Sample Name

Channel Name 210.0nm

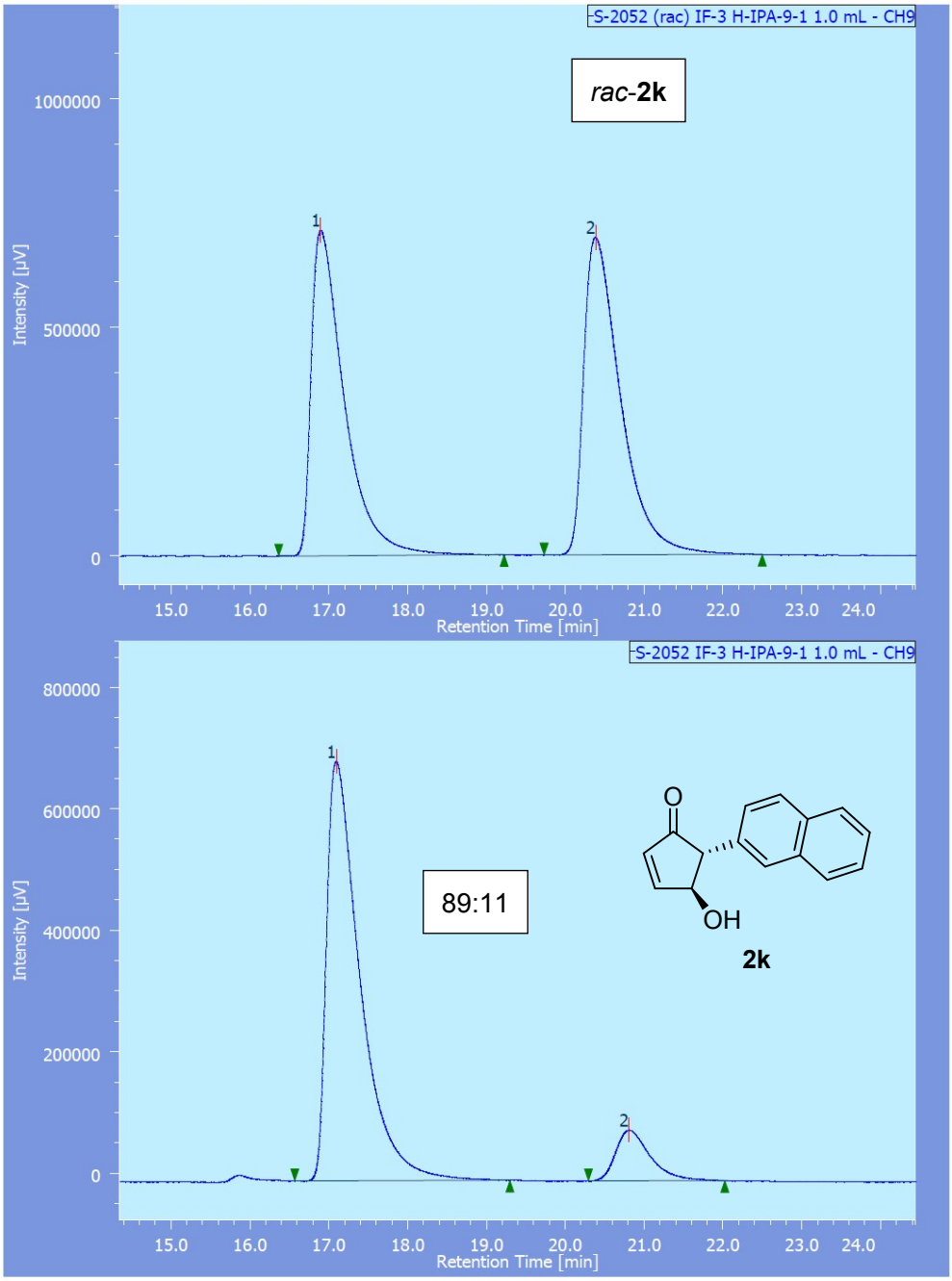
| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 10.025 | 38856018 | 1543821 | 50.623 | 50.485 | N/A | 3432 | 2.291 | 2.367 | |
| 2 | Unknown | 9 | 11.588 | 37899939 | 1514180 | 49.377 | 49.515 | N/A | 4595 | N/A | 2.536 | |

Chromatogram Name S-2053 IF-3 H-IPA 9-1 1mL-CH9

Sample Name

Channel Name 210.0nm

| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 10.167 | 11757808 | 744387 | 71.834 | 72.395 | N/A | 10572 | 4.029 | 1.907 | |
| 2 | Unknown | 9 | 11.780 | 4610330 | 283836 | 28.166 | 27.605 | N/A | 13375 | N/A | 1.541 | |




Channel & Peak Information Table

Chromatogram Name S-2052 (rac) IF-3 H-IPA-9-1 1.0 mL-CH9

Sample Name

Channel Name 210.0nm

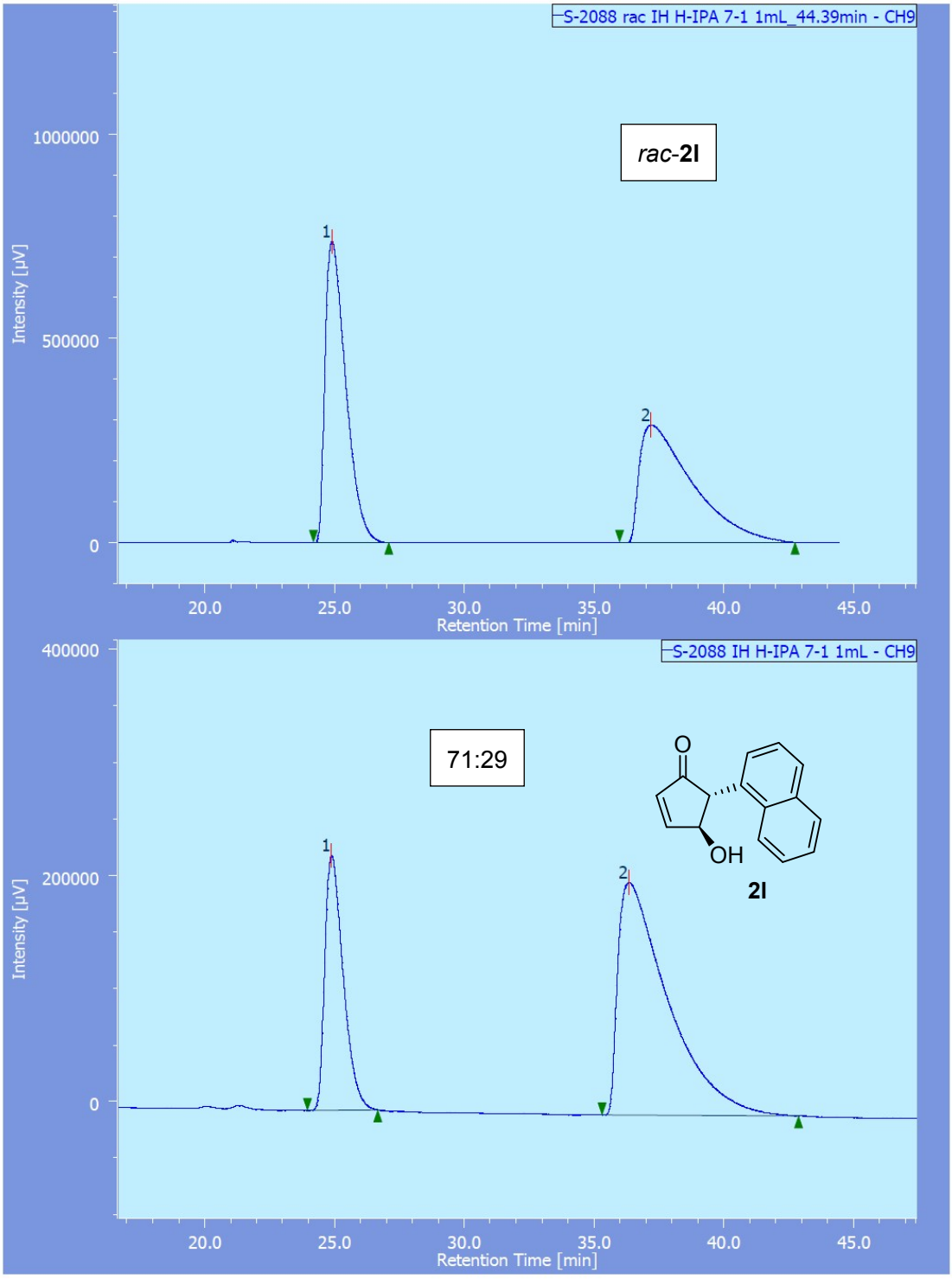
| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 16.902 | 20594138 | 712576 | 47.727 | 50.628 | N/A | 9055 | 4.575 | 2.283 | |
| 2 | Unknown | 9 | 20.383 | 22555756 | 694900 | 52.273 | 49.372 | N/A | 9989 | N/A | 1.962 | |

Chromatogram Name S-2052 IF-3 H-IPA-9-1 1.0 mL-CH9

Sample Name

Channel Name 210.0nm

| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 17.097 | 20385439 | 690446 | 88.954 | 89.225 | N/A | 8899 | 5.014 | 2.315 | |
| 2 | Unknown | 9 | 20.808 | 2531367 | 83381 | 11.046 | 10.775 | N/A | 12004 | N/A | 1.524 | |




Channel & Peak Information Table

Chromatogram Name S-2088 rac IH H-IPA 7-1 1mL_44.39min-CH9

Sample Name

Channel Name MaxABS(220.0-648.0)

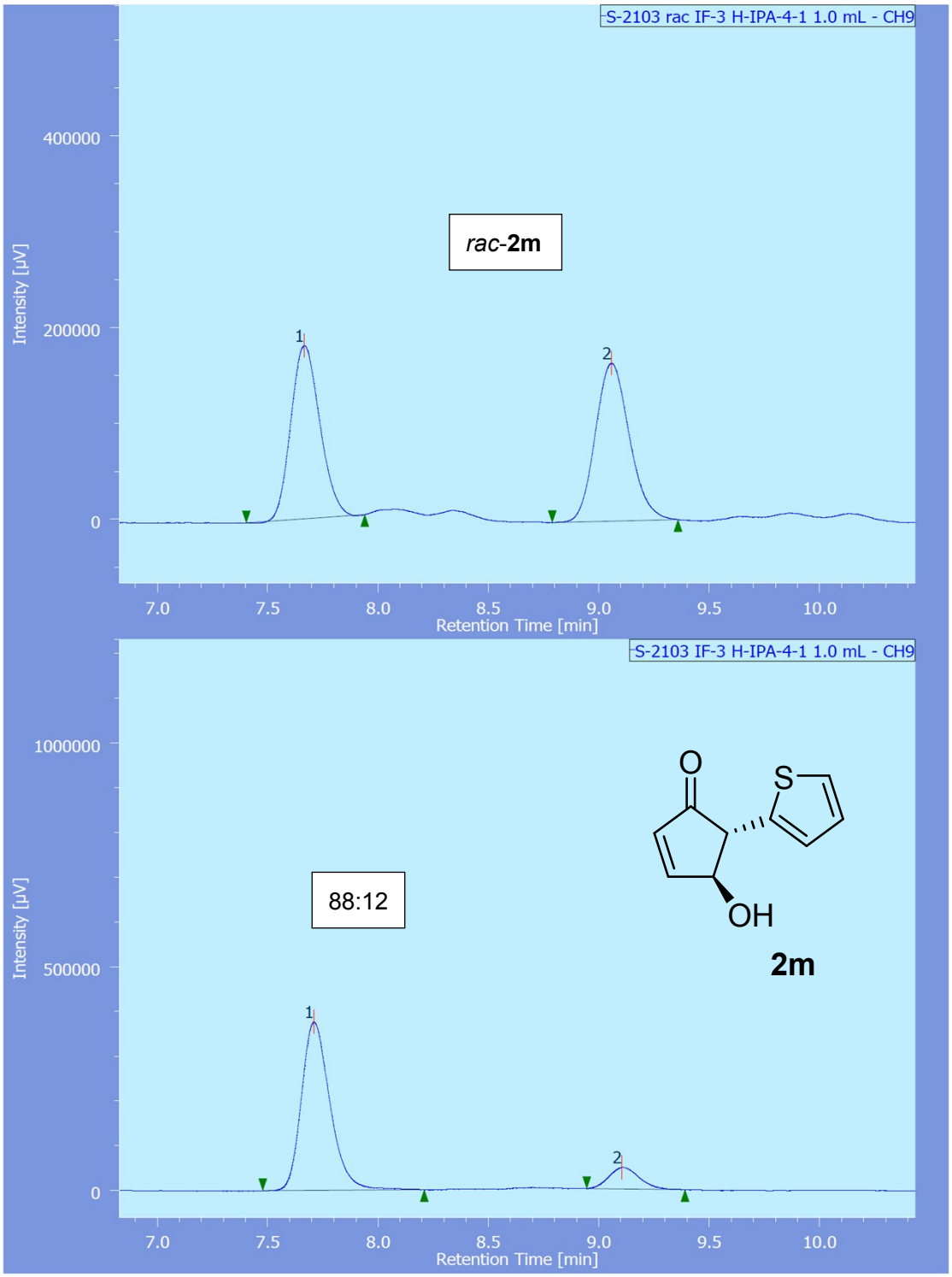
| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 24.897 | 40554003 | 736436 | 49.949 | 71.948 | N/A | 4670 | 4.862 | 1.867 | |
| 2 | Unknown | 9 | 37.192 | 40637157 | 287132 | 50.051 | 28.052 | N/A | 1695 | N/A | 3.301 | |

Chromatogram Name S-2088 IH H-IPA 7-1 1mL-CH9

Sample Name

Channel Name 210.0nm

| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 24.883 | 11140840 | 225473 | 28.740 | 52.268 | N/A | 5907 | 4.938 | 1.607 | |
| 2 | Unknown | 9 | 36.327 | 27624038 | 205902 | 71.260 | 47.732 | N/A | 1879 | N/A | 3.487 | |




Channel & Peak Information Table

Chromatogram Name S-2103 rac IF-3 H-IPA-4-1 1.0 mL-CH9

Sample Name

Channel Name 210.0nm

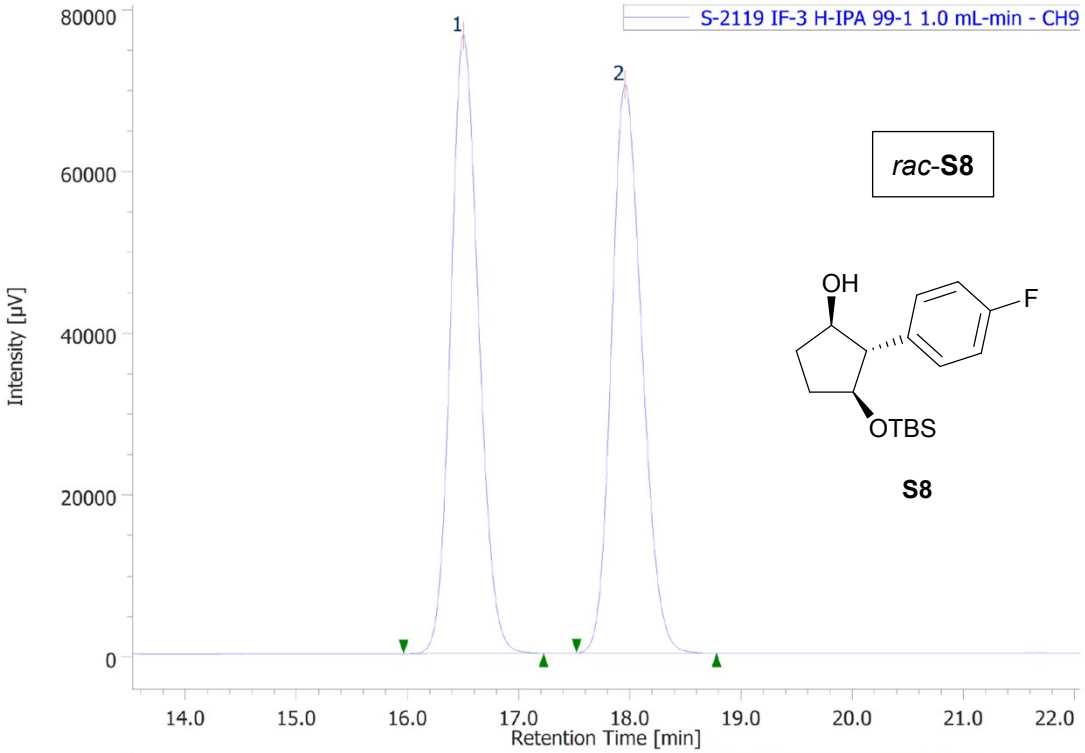
| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 7.667 | 1652903 | 180646 | 48.433 | 52.323 | N/A | 15790 | 5.313 | 1.142 | |
| 2 | Unknown | 9 | 9.058 | 1759871 | 164605 | 51.567 | 47.677 | N/A | 16602 | N/A | 1.132 | |

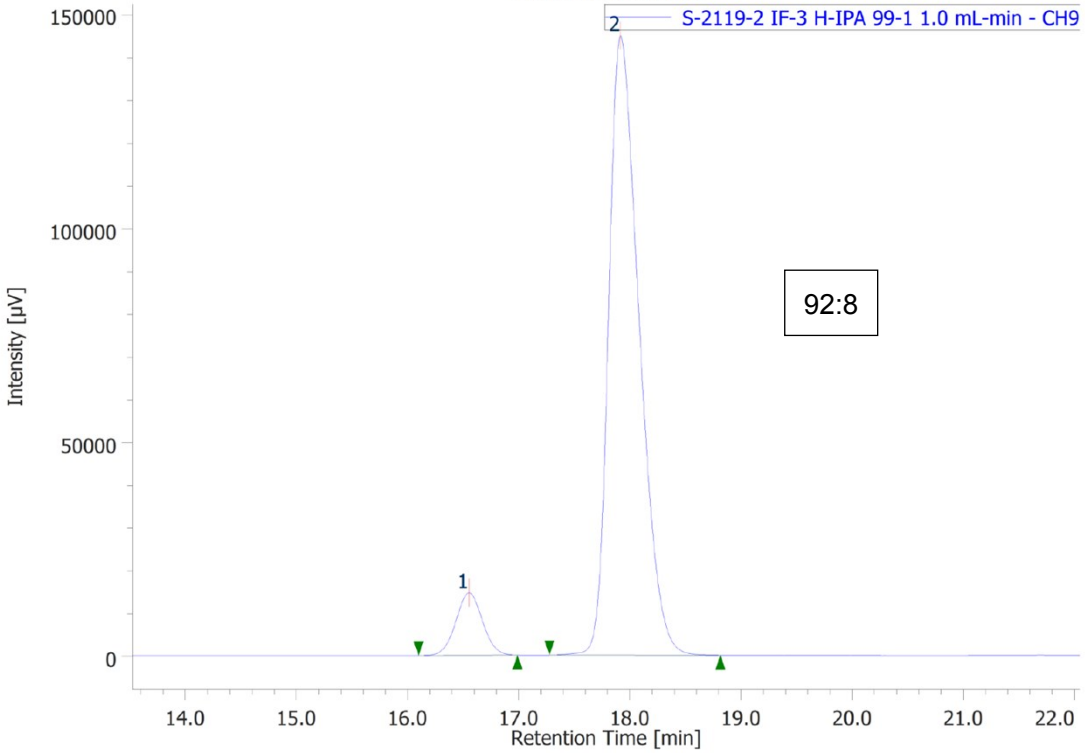
Chromatogram Name S-2103 IF-3 H-IPA-4-1 1.0 mL-CH9

Sample Name

Channel Name 210.0nm

| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 7.710 | 3393492 | 376505 | 87.847 | 88.629 | N/A | 17932 | 5.758 | 1.259 | |
| 2 | Unknown | 9 | 9.105 | 469452 | 48306 | 12.153 | 11.371 | N/A | 20307 | N/A | 1.193 | |



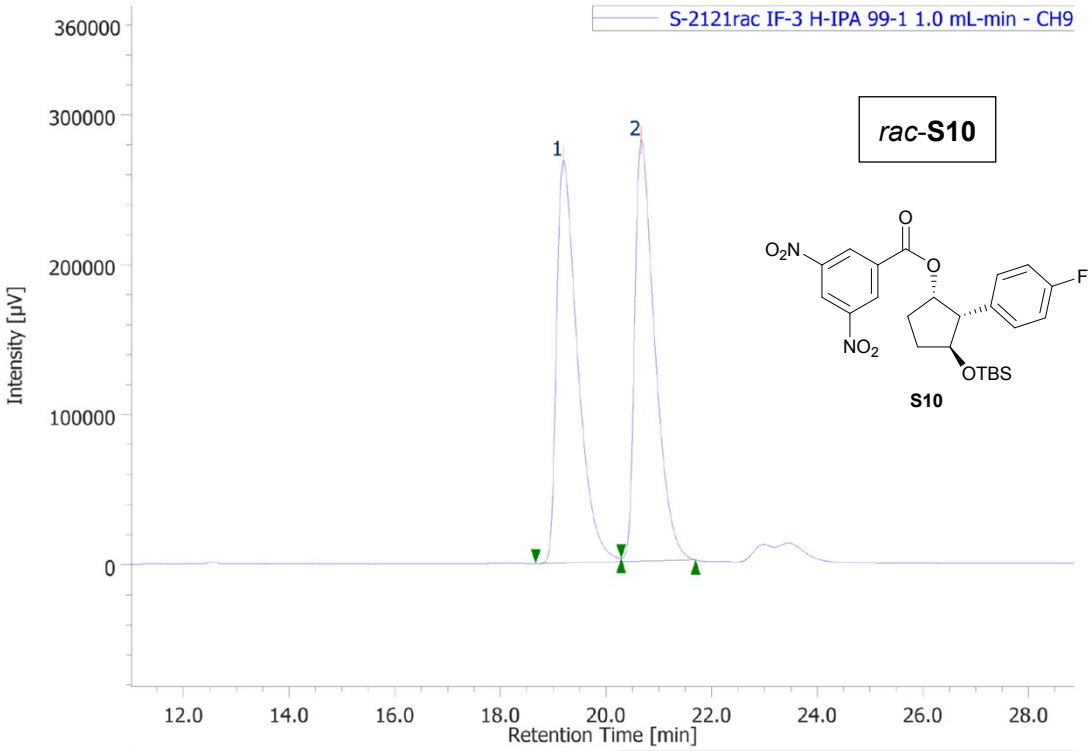

**Channel & Peak Information Table**

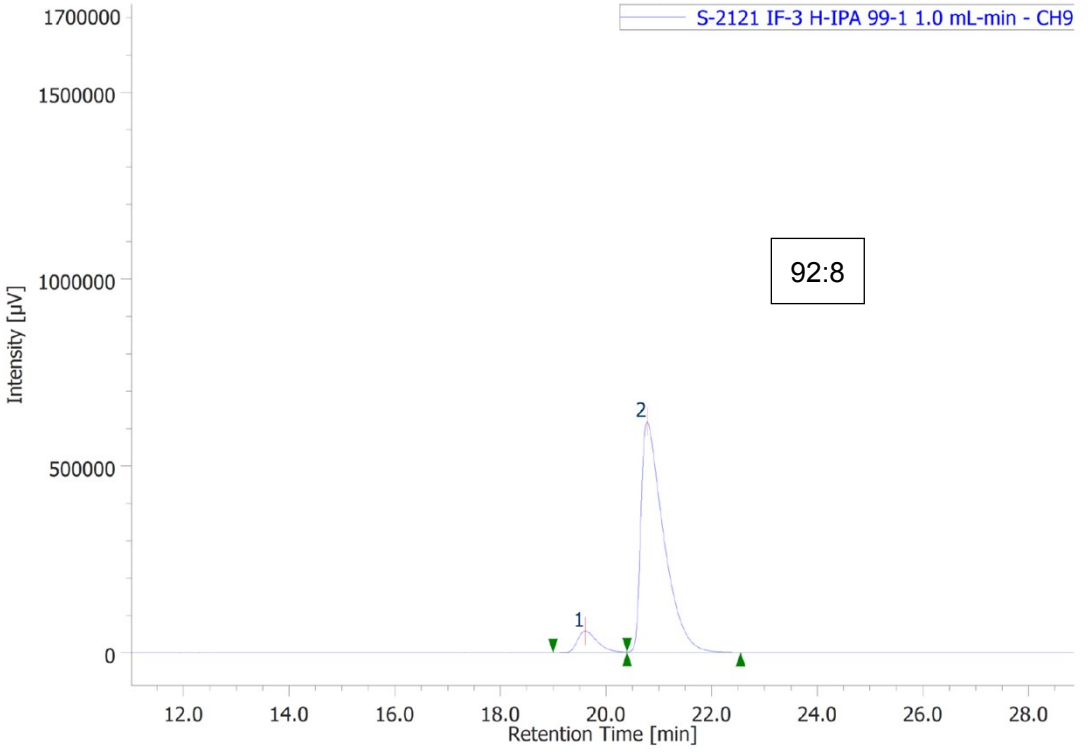
Chromatogram Name: S-2119 IF-3 H-IPA 99-1 1.0 mL-min-CH9
Sample Name:
Channel Name: 265.0nm

| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 16.503 | 1330439 | 76333 | 49.984 | 52.092 | N/A | 20821 | 3.044 | 1.132 | |
| 2 | Unknown | 9 | 17.957 | 1331284 | 70201 | 50.016 | 47.908 | N/A | 20637 | N/A | 1.178 | |

Chromatogram Name: S-2119-2 IF-3 H-IPA 99-1 1.0 mL-min-CH9
Sample Name:
Channel Name: 265.0nm

| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 16.553 | 242755 | 14610 | 8.091 | 9.163 | N/A | 22983 | 2.919 | 1.026 | |
| 2 | Unknown | 9 | 17.917 | 2757410 | 144839 | 91.909 | 90.837 | N/A | 20568 | N/A | 1.380 | |




**Channel & Peak Information Table**

Chromatogram Name S-2121rac IF-3 H-IPA 99-1 1.0 mL-min-CH9
Sample Name
Channel Name 220.0nm

| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 19.193 | 7420921 | 268707 | 49.940 | 48.890 | N/A | 11849 | 2.129 | 1.882 | |
| 2 | Unknown | 9 | 20.667 | 7438779 | 280906 | 50.060 | 51.110 | N/A | 14667 | N/A | 1.810 | |

Chromatogram Name S-2121 IF-3 H-IPA 99-1 1.0 mL-min-CH9
Sample Name
Channel Name 220.0nm

| # | Peak Name | CH | tR [min] | Area [μV·sec] | Height [μV] | Area% | Height% | Quantity | NTP | Resolution | Symmetry Factor | Warning |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Unknown | 9 | 19.603 | 1516231 | 57721 | 7.614 | 8.545 | N/A | 13556 | 1.646 | 1.525 | |
| 2 | Unknown | 9 | 20.777 | 18396519 | 617788 | 92.386 | 91.455 | N/A | 12080 | N/A | 2.299 | |