Supplementary Information

Rhodium-catalyzed asymmetric addition of arylboronic acids to 2*H*-chromenes leading to 3-arylchromane derivatives

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Contents of Supplementary Information:

1. General

All anaerobic and moisture-sensitive manipulation were carried out with standard Schlenk techniques under predried nitrogen. NMR spectra were recorded on a JEOL JNM ECA-400 spectrometer (400 MHz for ^{1}H NMR, 100 MHz for ^{13}C NMR). Chemical shifts are reported in δ (ppm) referenced to the residual peaks of $CDCl_3$ (δ 7.26) for ^{1}H NMR and $CDCl_3$ (δ 77.00) for ^{13}C NMR. The following abbreviations are used; s, singlet: d, doublet: t, triplet: q, quartet: quint, quintet: m, multiplet. High-resolution mass spectra were obtained with a JEOL AccuTOF LC-plus JMS-T100LP spectrometer. Optical rotations were measured on JASCO P-2200 polarimeter. Preparative thin-layer chromatography was performed with Silica Gel 70 PF_{254} (Wako). Alumina (active 200) for column chromatography was purchased from Nacalai Tesque.

2. Materials

Dehydrated solvents were purchased and used after deoxygenated by bubbling N_2. Rhodium complexes [Rh(OH)((*S*,*S*)-Ph-tfb*)]$_2$, [Rh(OH)((*S*,*S*)-Bn-tfb*)]$_2$, and [Rh(OH)((*S*,*S*)-Fc-tfb*)]$_2$ were prepared according to the reported procedures.[1] Compounds **2a**–**j** were purchased from commercial suppliers and used as received.

3. Preparation of alkenes 1

Compound **1a** (CAS: 254-04-6),[2] **1b** (CAS: 457628-47-6),[3] **1c** (CAS: 16336-27-9),[3] **1d** (CAS: 18385-84-7),[3] **1e** (CAS: 1015938-77-8),[3] **1f** (CAS: 42969-79-9),[3] **1g** (CAS: 90448-25-2),[3] **1i** (CAS: 10174-55-7),[4] **1j** (CAS: 254-37-5),[5] **1k** (CAS: 2733-79-1), and **1n** (CAS: 18385-89-2)[6] were prepared according to the reported procedures. Compound **1h** was prepared according to the procedure for **1b**. Compounds **1l** and **1m** was purchased and used as received.

4. Procedure for rhodium-catalyzed addition of *p*-tolylboronic acid (2a) to 2*H*-chromene (1a) (Table 1)

A rhodium complex (5 mol% of Rh) and *p*-tolylboronic acid (**2a**) (271.9 mg, 2.0 mmol) were placed in a Schlenk tube under N_2. Then, 1,4-dioxane (0.40 mL) and 2*H*-chromene (**1a**) (26.4 mg, 0.20 mmol) were added to the tube successively, and the mixture was stirred at 60 ºC for 20 h. The mixture was passed through a short column of alumina with CH_2Cl_2 as an eluent, and the solvent was removed on a rotary evaporator. The ee was measured by chiral HPLC analysis after isolation of the product by preparative TLC on silica gel eluted with EtOAc/hexane (1:50).

5. Procedure for rhodium-catalyzed asymmetric addition of arylboronic acids 2 to chromene derivatives 1 (Table 2 and Scheme 3)

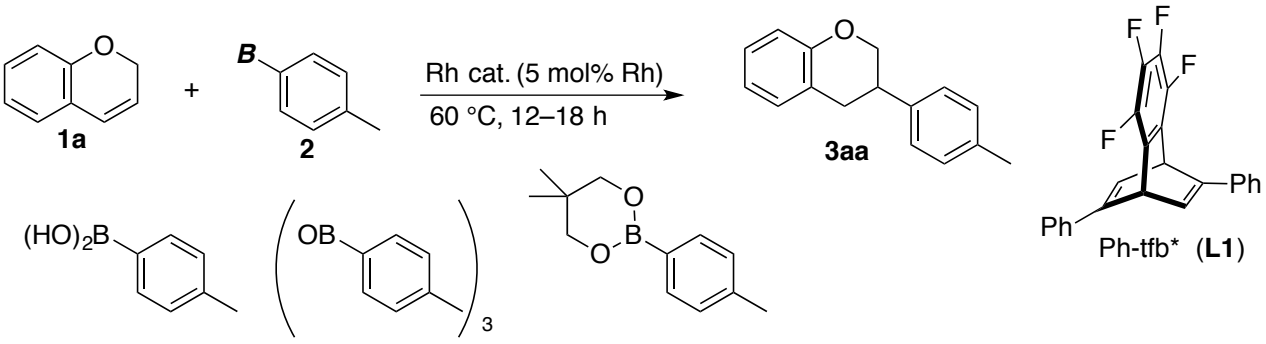
[Rh(OH)((*S*,*S*)-**L1**)]$_2$ (5.0 mg, 0.010 mmol, 5 mol% of Rh) and arylboronic acid **2** (2.0 mmol) were placed in a Schlenk tube under N_2. Then, 1,4-dioxane (0.40 mL) and chromene **1** (0.20 mmol) were added to the tube successively, and the mixture was stirred at 60 °C for 20 h. The mixture was passed through a short column of alumina with CH_2Cl_2 as an eluent, and the

solvent was removed on a rotary evaporator. The residue was subjected to preparative TLC on silica gel eluted with EtOAc/hexane (1:10–1:50). The ee was measured by chiral HPLC analysis after isolation of the products.

6. Results of the reactions under several reaction conditions

Effects of bases, solvents, ligands, and proton sources are shown in Table S1.

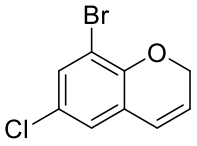
Table S1. Rh-catalyzed addition of **2** to **1a**



entry	catalyst	solvent	boron reagent	note	yield (%)[b]
1	$[Rh(OH)(cod)]_2$	1,4-dioxane	**2a**	at 80 °C	17
2	$[Rh(OH)(cod)]_2$	1,4-dioxane/H_2O (9:1)	**2a**	at 80 °C	17
3	$[Rh(OH)(cod)]_2$	1,4-dioxane/H_2O (9:1)	**2a"**	at 80 °C	17
4	$[Rh(OH)(cod)]_2$	toluene/*t*-amyl alcohol (1:1)	**2a**	at 80 °C	20
5	$[Rh(OH)(cod)]_2$	toluene/*t*-amyl alcohol (1:1)	**2a"**	at 80 °C	48
6	$[Rh(OH)(cod)]_2$	toluene/methanol (1:1)	**2a"**	at 80 °C	22
7	$[Rh(OH)(cod)]_2$	toluene/*t*-amyl alcohol (1:1)	**2a"**		33
8	$[Rh(OH)(cod)]_2$	toluene/H_2O (20:1)	**2a"**	$NaHCO_3$ (1 equiv.)	33
9	$[Rh(OH)(cod)]_2$	toluene/*t*-amyl alcohol (1:1)	**2a'**		42
10	$[Rh(OH)(cod)]_2$	toluene/methanol (1:1)	**2a'**		16
11	$[Rh(OH)((R,R)\text{-}\mathbf{L1})]_2$	1,4-dioxane	**2a**		47 (96% ee)[c]
12	$[Rh(OH)((R,R)\text{-}\mathbf{L1})]_2$	1,4-dioxane	**2a**	Et_3N (1 equiv.)	45
13	$[Rh(OH)((R,R)\text{-}\mathbf{L1})]_2$	toluene/*t*-amyl alcohol (1:1)	**2a'**		24
14	$[Rh(OH)((R,R)\text{-}\mathbf{L1})]_2$	1,4-dioxane/H_2O (7.5 equiv.)	**2a"**		49
15	$[Rh(OH)((R,R)\text{-}\mathbf{L1})]_2$	1,4-dioxane/H_2O (7.5 equiv.)	**2a'**		42
16	$[Rh(OH)((R,R)\text{-}\mathbf{L1})]_2$	1,4-dioxane/H_2O (1.5 equiv.)	**2a'**		46
17[d]	$[Rh(OH)((S,S)\text{-}\mathbf{L1})]_2$	1,4-dioxane	**2a**		45[e] (97% ee)[c]
18[e]	$[Rh(OH)((S,S)\text{-}\mathbf{L1})]_2$	1,4-dioxane	**2a**		67[e] (97% ee)[c]

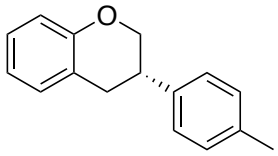
[a]Reaction conditions: **1a** (0.10 mmol), **2** (2.5 equiv. of B), and Rh catalyst (5 mol% of Rh) in solvent (0.4 mL) at 60 ºC for 12–18 h. [b]Determined by ^1H NMR. [c]Determined by HPLC analysis with a chiral stationary phase column: Chiralcel OB-H. [d]The reaction of **1a** (0.20 mmol) with **2a** (0.50 mmol) for 20 h. [e]Isolated yield. [f]The reaction of **1a** (0.20 mmol) with **2a** (2.0 mmol) for 20 h.

7. Characterization of the substrate and products



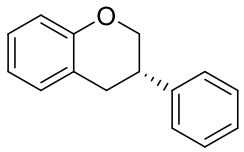
1h

Compound 1h (Scheme 3: colorless solid). ^{1}H NMR ($CDCl_3$) δ 4.95 (dd, *J* = 3.5, 2.1 Hz, 2H), 5.83 (dt, *J* = 10.0, 3.5 Hz, 1H), 6.31 (dd, *J* = 10.0, 2.1 Hz, 1H), 6.87 (d, *J* = 2.4 Hz, 1H), 7.29 (d, *J* = 2.4 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 66.5, 109.9, 123.2, 123.7, 124.2, 125.4, 126.1, 131.6, 149.4. HRMS (DART) calcd for $C_9H_7{}^{79}Br^{35}ClO$ $(M+H)^+$ 244.9369, found 244.9372.



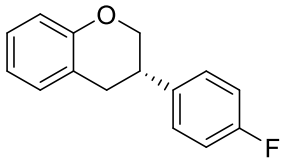
3aa

Compound 3aa (Table 1, entry 2: colorless solid, 29.9 mg, 67% yield, 97% ee). The ee was measured by HPLC (Chiralcel OB-H, hexane/2-propanol = 98:2, flow 0.5 mL/min, 254 nm, t_1 = 13.7 min (major), t_2 = 20.2 min (minor)): $[\alpha]^{25}_D$ –3 (*c* 0.71, $CHCl_3$) for 97% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.35 (s, 3H), 3.00 (ddd, *J* = 16.0, 6.2, 1.9 Hz, 1H), 3.07 (dd, *J* = 16.0, 10.5 Hz, 1H), 3.23 (tdd, *J* = 10.5, 6.2, 3.7 Hz, 1H), 4.02 (t, *J* = 10.5 Hz, 1H), 4.35 (ddd, *J* =10.5, 3.7, 1.9 Hz, 1H), 6.81–6.94 (m, 2H), 7.04–7.24 (m, 6H); ^{13}C NMR ($CDCl_3$) δ 21.0, 32.4, 38.1, 71.0, 116.5, 120.3, 122.0, 127.2, 127.4, 129.4, 129.8, 136.7, 138.2, 154.3. HRMS (DART) calcd for $C_{16}H_{17}O$ $(M+H)^+$ 225.1279, found 225.1270.



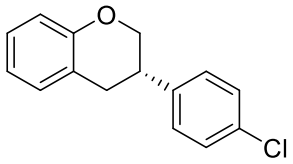
3ab

Compound 3ab (Table 2, entry 1: colorless solid, 29.4 mg, 70% yield, 96% ee, CAS: 20879-06-5). The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 98:2, flow 0.5 mL/min, 254 nm, t_1 = 24.8 min (major), t_2 = 31.3 min (minor)): $[\alpha]^{25}_D$ –6 (*c* 1.20, $CHCl_3$) for 96% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.98–3.14 (m, 2H), 3.26 (tdd, *J* = 10.5, 6.2, 3.7 Hz, 1H), 4.05 (t, *J* = 10.5 Hz, 1H), 4.37 (ddd, *J* =10.5, 3.7, 2.1 Hz, 1H), 6.88 (t, *J* = 7.5 Hz, 2H), 7.08–7.17 (m, 2H), 7.24–7.31 (m, 3H), 7.37 (t, *J* = 7.5 Hz, 2H).



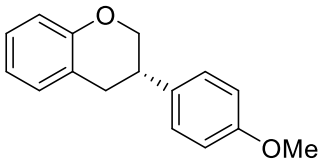
3ac

Compound 3ac (Table 2, entry 2: colorless solid, 25.1 mg, 55% yield, 97% ee, CAS: 2128324-29-6). The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 98:2, flow 0.5 mL/min, 254 nm, t_1 = 27.4 min (major), t_2 = 32.0 min (minor)): $[\alpha]^{25}_D$ +0.7 (*c* 0.69, $CHCl_3$) for 97% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.99–3.09 (m, 2H), 3.21–3.31 (m, 1H), 4.10 (t, *J* = 10.5 Hz, 1H), 4.34 (dd, *J* =10.5, 4.2 Hz, 1H), 6.85–6.93 (m, 2H), 7.01–7.18 (m, 4H), 7.19–7.25 (m, 2H).



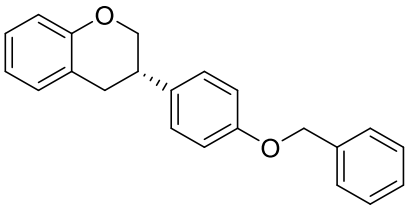
3ad

Compound 3ad (Table 2, entry 3: colorless solid, 28.6 mg, 59% yield, 94% ee). The ee was measured by HPLC (Chiralcel OB-H, hexane/2-propanol = 98:2, flow 0.5 mL/min, 254 nm, t_1 = 19.5 min (major), t_2 = 26.9 min (minor)): $[\alpha]^{25}_D$ +3 (*c* 0.78, $CHCl_3$) for 94% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.98–3.09 (m, 2H), 3.18–3.30 (m, 1H), 4.04 (t, *J* = 10.5 Hz, 1H), 4.33 (dd, *J* = 10.5, 3.2 Hz, 1H), 6.84–6.92 (m, 2H), 7.06–7.22 (m, 4H), 7.33 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 32.2, 38.0, 70.5, 116.6, 120.5, 121.5, 127.5, 128.7, 128.9, 129.7, 132.8, 139.8, 154.2. HRMS (DART) calcd for $C_{15}H_{14}{}^{35}ClO$ $(M+H)^+$ 245.0733, found 245.0736.



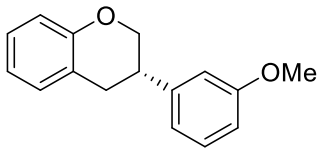
3ae

Compound 3ae (Table 2, entry 4: colorless solid, 34.3 mg, 72% yield, 95% ee, CAS: 169125-23-9). The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 10:1, flow 0.5 mL/min, 254 nm, t_1 = 23.4 min (major), t_2 = 27.7 min (minor)): $[\alpha]^{25}_D$ –2 (*c* 0.43, $CHCl_3$) for 95% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.99 (dd, *J* = 16.0, 6.5 Hz, 1H), 3.05 (dd, *J* = 16.0, 10.5 Hz, 1H), 3.22 (tdd, *J* = 10.5, 6.5, 3.6 Hz, 1H), 3.81 (s, 3H), 3.99 (t, *J* = 10.5 Hz, 1H), 4.33 (ddd, *J* =10.5, 3.6, 1.8 Hz, 1H), 6.84–6.93 (m, 4H), 7.08–7.16 (m, 2H), 7.18 (d, *J* = 8.7 Hz, 2H).



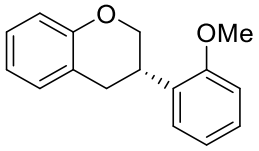
3af

Compound 3af (Table 2, entry 5: colorless solid, 34.6 mg, 55% yield, 97% ee). The ee was measured by HPLC (Chiralpak IA, hexane/$CHCl_3$ = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 24.5 min (major), t_2 = 28.3 min (minor)): $[\alpha]^{25}_D$ –3 (*c* 0.73, $CHCl_3$) for 97% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.95–3.09 (m, 2H), 3.21 (tdd, *J* = 10.5, 6.5, 3.4 Hz, 1H), 4.00 (t, *J* = 10.5 Hz, 1H), 4.33 (ddd, *J* = 10.5, 3.4, 1.6 Hz, 1H), 5.07 (s, 2H), 6.88 (td, *J* = 8.0, 1.6 Hz, 2H), 6.97 (d, *J* = 8.4 Hz, 2H), 7.07–7.21 (m, 4H), 7.30–7.48 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 32.5, 37.7, 70.0, 71.0, 115.1, 116.5, 120.3, 122.0, 127.39, 127.44, 128.0, 128.3, 128.6, 129.8, 133.6, 137.0, 154.3, 157.8. HRMS (DART) calcd for $C_{22}H_{21}O_2$ $(M+H)^+$ 317.1542, found 317.1538.



3ag

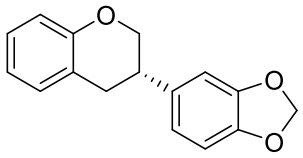
Compound 3ag (Table 2, entry 6: pale yellow solid, 21.6 mg, 45% yield, 95% ee). The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 19:1, flow 0.5 mL/min, 254 nm, t_1 = 24.8 min (major), t_2 = 28.5 min (minor)): $[\alpha]^{25}_D$ –2 (*c* 0.68, $CHCl_3$) for 95% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.97–3.13 (m, 2H), 3.24 (tdd, *J* = 10.8, 6.5, 3.6 Hz, 1H), 3.82 (s, 3H), 4.04 (t, *J* = 10.8 Hz, 1H), 4.37 (ddd, *J* = 10.8, 3.6, 1.9 Hz, 1H), 6.78–6.92 (m, 5H), 7.08–7.15 (m, 2H), 7.29 (t, *J* = 7.8 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 32.3, 38.6, 55.2, 70.8, 112.0, 113.5, 116.5, 119.6, 120.4, 121.9, 127.4, 129.8, 142.9, 154.2, 159.8. HRMS (DART) calcd for $C_{16}H_{17}O_2$ $(M+H)^+$ 241.1229, found 241.1219.



3ah

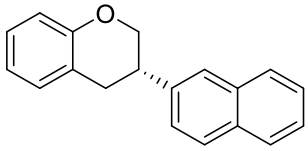
Compound 3ah (Table 2, entry 7: pale yellow solid, 21.7 mg, 45% yield, 95% ee). The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 10:1, flow 0.5 mL/min, 254 nm, t_1 = 19.1 min (major), t_2 = 21.7 min (minor)): $[\alpha]^{25}_D$ +7 (*c* 0.57, $CHCl_3$) for 95% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.98 (ddd, *J* = 16.0, 5.5, 2.0 Hz, 1H), 3.11 (dd, *J* = 16.0, 10.6 Hz, 1H), 3.71 (tdd, *J* = 10.6, 5.5, 3.6 Hz, 1H), 3.85 (s, 3H), 4.08 (t, *J* = 10.6 Hz, 1H), 4.38 (ddd, *J* = 10.6, 3.6, 2.0 Hz, 1H), 6.85–

6.99 (m, 4H), 7.08–7.18 (m, 3H), 7.23–7.30 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 30.9, 31.9, 55.3, 69.9, 110.5, 116.5, 120.2, 120.7, 122.4, 127.1, 127.3, 127.9, 129.4, 129.8, 154.4, 157.3. HRMS (DART) calcd for $C_{16}H_{17}O_2$ $(M+H)^+$ 241.1229, found 241.1227.



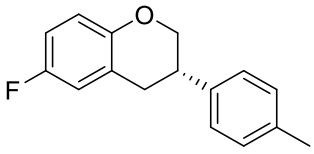
3ai

Compound 3ai (Table 2, entry 8: colorless solid, 25.9 mg, 53% yield, 96% ee). The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 10:1, flow 0.5 mL/min, 254 nm, t_1 = 27.8 min (major), t_2 = 30.8 min (minor)): $[\alpha]^{25}_D$ +2 (*c* 0.54, $CHCl_3$) for 96% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.94–3.06 (m, 2H), 3.14–3.23 (m, 1H), 3.97 (t, *J* = 10.5 Hz, 1H), 4.32 (dd, *J* = 10.5, 3.8 Hz, 1H), 5.96 (s, 2H), 6.69–6.74 (m, 2H), 6.80 (d, *J* = 7.6 Hz, 1H), 6.83–6.91 (m, 2H), 7.09 (d, *J* = 7.6 Hz, 1H), 7.13 (t, *J* = 7.6 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 32.6, 38.3, 71.0, 101.0, 107.7, 108.5, 116.5, 120.35, 120.38, 121.8, 127.4, 129.7, 135.2, 146.5, 147.9, 154.2. HRMS (DART) calcd for $C_{16}H_{15}O_3$ $(M+H)^+$ 255.1021, found 255.1021.



3aj

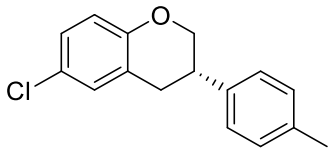
Compound 3aj (Table 2, entry 9: colorless solid, 27.9 mg, 54% yield, 92% ee, CAS: 2128324-32-1). The ee was measured by HPLC (Chiralcel OB-H, hexane/2-propanol = 98:2, flow 0.5 mL/min, 254 nm, t_1 = 28.3 min (major), t_2 = 40.3 min (minor)): $[\alpha]^{25}_D$ +19 (*c* 1.01, $CHCl_3$) for 92% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 3.11 (dd, *J* = 16.0, 3.6 Hz, 1H), 3.21 (dd, *J* = 16.0, 10.7 Hz, 1H), 3.38–3.48 (m, 1H), 4.15 (t, *J* = 10.7 Hz, 1H), 4.46 (dt, *J* =10.7, 2.4 Hz, 1H), 6.87–6.93 (m, 2H), 7.09–7.17 (m, 2H), 7.40 (d, *J* = 7.5 Hz, 1H), 7.39–7.51 (m, 2H), 7.70 (s, 1H), 7.81–7.86 (m, 3H).



3ba

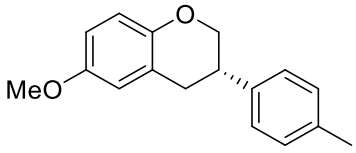
Compound 3ba (Scheme 3: pale yellow solid, 37.1 mg, 77% yield, 92% ee). The ee was measured by HPLC (Chiralcel OB-H, hexane/2-propanol = 98:2, flow 0.5 mL/min, 254 nm, t_1 = 10.9 min (major), t_2 = 13.4 min (minor)): $[\alpha]^{25}_D$ –9 (*c* 1.09, $CHCl_3$) for 92% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.36 (s, 3H), 2.93−3.09 (m, 2H), 3.21 (tdd, *J* = 10.5, 6.7, 3.7 Hz, 1H), 3.99 (t, *J* = 10.5

Hz, 1H), 4.33 (ddd, J = 10.5, 3.7, 2.0 Hz, 1H), 6.77−6.87 (m, 3H), 7.14 (d, J = 8.2 Hz, 2H), 7.19 (d, J = 8.2 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 21.0, 32.5, 37.8, 71.0, 114.1 (d, $J_{C\text{-}F}$ = 23 Hz), 115.5 (d, $J_{C\text{-}F}$ = 23 Hz), 117.3 (d, $J_{C\text{-}F}$ = 8 Hz), 123.1 (d, $J_{C\text{-}F}$ = 8 Hz), 127.2, 129.5, 136.8, 137.9, 150.3, 156.7 (d, $J_{C\text{-}F}$ = 237 Hz). HRMS (DART) calcd for $C_{16}H_{16}FO$ $(M+H)^+$ 243.1185, found 243.1180.



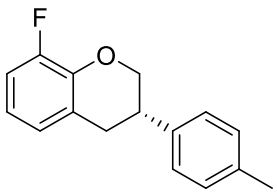
3ca

Compound 3ca (Scheme 3: pale yellow solid, 39.1 mg, 76% yield, 97% ee). The ee was measured by HPLC (Chiralcel OB-H, hexane/2-propanol = 98:2, flow 0.5 mL/min, 254 nm, t_1 = 11.9 min (major), t_2 = 13.4 min (minor)): $[\alpha]^{25}_D$ –0.8 (*c* 0.82, $CHCl_3$) for 97% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.37 (s, 3H), 2.93–3.08 (m, 2H), 3.20 (tdd, J = 10.8, 6.6, 3.5 Hz, 1H), 4.01 (t, J = 10.8 Hz, 1H), 4.35 (ddd, J = 10.8, 3.5, 2.2 Hz, 1H), 6.81 (d, J = 9.2 Hz, 1H), 7.06–7.14 (m, 2H), 7.15 (d, J = 8.0 Hz, 2H), 7.20 (d, J = 8.0 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 21.0, 32.2, 37.7, 71.0, 117.8, 123.6, 125.0, 127.2, 127.3, 129.2, 129.5, 136.9, 137.7, 152.9. HRMS (DART) calcd for $C_{16}H_{16}{}^{35}ClO$ $(M+H)^+$ 259.0890, found 259.0896.



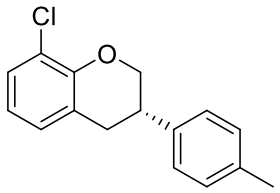
3da

Compound 3da (Scheme 3: colorless solid, 26.3 mg, 52% yield, 96% ee). The ee was measured by HPLC (Chiralcel OB-H, hexane/2-propanol = 98:2, flow 0.5 mL/min, 254 nm, t_1 = 19.1 min (major), t_2 = 23.9 min (minor)): $[\alpha]^{25}_D$ –3 (*c* 0.86, $CHCl_3$) for 96% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.36 (s, 3H), 2.98 (ddd, J = 16.0, 5.8, 2.0 Hz, 1H), 3.06 (dd, J = 16.0, 10.5 Hz, 1H), 3.22 (tdd, J = 10.5, 5.8, 3.7 Hz, 1H), 3.78 (s, 3H), 3.98 (t, J = 10.5 Hz, 1H), 4.31 (ddd, J = 10.5, 3.7, 2.0 Hz, 1H), 6.65 (d, J = 2.8 Hz, 1H), 6.73 (dd, J = 9.0, 2.8 Hz, 1H), 6.81 (d, J = 8.8 Hz, 1H), 7.15 (d, J = 8.4 Hz, 2H), 7.18 (d, J = 8.4 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 21.0, 32.7, 38.2, 55.7, 70.9, 113.4, 114.2, 117.1, 122.6, 127.2, 129.4, 136.7, 138.3, 148.3, 153.3. HRMS (DART) calcd for $C_{17}H_{19}O_2$ $(M+H)^+$ 255.1385, found 255.1383.



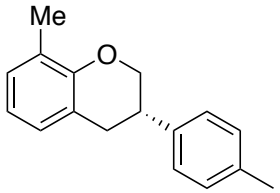
3ea

Compound 3ea (Scheme 3: pale yellow solid, 29.4 mg, 61% yield, 96% ee). The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 98:2, flow 0.5 mL/min, 254 nm, t_1 = 27.7 min (major), t_2 = 34.4 min (minor)): $[\alpha]^{25}_D$ –5 (*c* 1.30, $CHCl_3$) for 96% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.36 (s, 3H), 2.97−3.12 (m, 2H), 3.24 (tdd, *J* = 10.5, 5.5, 3.7 Hz, 1H), 4.05 (t, *J* = 10.5 Hz, 1H), 4.45 (ddd, *J* = 10.5, 3.7, 1.9 Hz, 1H), 6.76−6.82 (m, 1H), 6.87 (d, *J* = 7.2 Hz, 1H), 6.90−6.98 (m, 1H), 7.15 (d, *J* = 8.2 Hz, 2H), 7.19 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 21.0, 32.1, 37.8, 71.1, 113.7 (d, $J_{C\text{-}F}$ = 18 Hz), 113.9 (d, $J_{C\text{-}F}$ = 8 Hz), 124.6 (d, $J_{C\text{-}F}$ = 10 Hz), 124.7, 127.2, 129.5, 136.9, 137.6, 142.5 (d, $J_{C\text{-}F}$ =11 Hz), 151.6 (d, $J_{C\text{-}F}$ = 243 Hz). HRMS (DART) calcd for $C_{16}H_{16}FO$ $(M+H)^+$ 243.1185, found 243.1194.



3fa

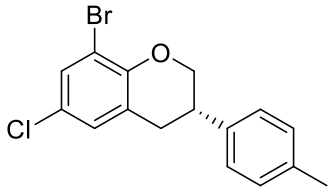
Compound 3fa (Scheme 3: pale yellow solid, 30.0 mg, 58% yield, 95% ee). The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 98:2, flow 0.5 mL/min, 254 nm, t_1 = 29.4 min (major), t_2 = 35.4 min (minor)): $[\alpha]^{25}_D$ –31 (*c* 1.14, $CHCl_3$) for 95% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.37 (s, 3H), 3.01 (ddd, *J* = 16.0, 5.6, 2.0 Hz, 1H), 3.08 (dd, *J* = 16.0, 10.7 Hz, 1H), 3.23 (tdd, *J* = 10.7, 5.6, 3.8 Hz, 1H), 4.08 (t, *J* = 10.7 Hz, 1H), 4.51 (ddd, *J* = 10.7, 3.8, 2.0 Hz, 1H), 6.82 (t, *J* = 7.5 Hz. 1H), 7.01 (d, *J* = 7.5 Hz, 1H), 7.15 (d, *J* = 8.2 Hz, 2H), 7.20 (d, *J* = 8.2 Hz, 2H), 7.23 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 21.0, 32.5, 37.8, 71.6, 120.4, 121.4, 123.8, 127.2, 128.0, 128.1, 129.5, 136.9, 137.5, 149.9. HRMS (DART) calcd for $C_{16}H_{16}{}^{35}ClO$ $(M+H)^+$ 259.0890, found 259.0890.



3ga

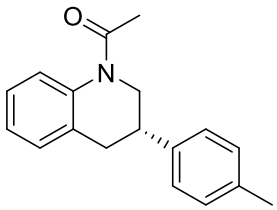
Compound 3ga (Scheme 3: colorless solid, 24.7 mg, 52% yield, 97% ee). The ee was measured by HPLC (Chiralcel OB-H, hexane/2-propanol = 98:2, flow 0.5 mL/min, 254 nm, t_1 = 18.1 min (major), t_2 = 22.5 min (minor)): $[\alpha]^{25}_D$ –14 (*c* 0.77, $CHCl_3$) for 97% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.24 (s, 3H), 2.37 (s, 3H), 3.00 (ddd, *J* = 16.0, 6.3, 2.2 Hz, 1H), 3.08 (dd, *J* = 16.0, 10.8 Hz, 1H), 3.22 (tdd, *J* = 10.8, 6.3, 3.9 Hz, 1H), 4.02 (t, *J* = 10.8 Hz, 1H), 4.42 (ddd, *J* = 10.8, 3.9, 2.2 Hz, 1H), 6.80 (t, *J* = 7.4 Hz, 1H), 6.97 (d, *J* = 7.0 Hz, 1H), 7.02 (d, *J* = 7.0 Hz, 1H), 7.17 (d, *J* = 8.2

Hz, 2H), 7.20 (d, J = 8.2 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 16.1, 21.0, 32.6, 38.2, 71.1, 119.7, 121.5, 125.7, 127.2, 127.3, 128.5, 129.4, 136.6, 138.4, 152.4. HRMS (DART) calcd for $C_{17}H_{19}O$ $(M+H)^+$ 239.1436, found 239.1436.



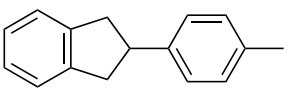
3ha

Compound 3ha (Scheme 3: pale yellow solid, 49.6 mg, 73% yield, 92% ee). The ee was measured by HPLC (Chiralcel OB-H, hexane/2-propanol = 98:2, flow 0.5 mL/min, 254 nm, t_1 = 23.0 min (major), t_2 = 28.8 min (minor)): $[\alpha]^{25}_D$ –19 (*c* 1.02, $CHCl_3$) for 92% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.36 (s, 3H), 2.97 (ddd, J = 16.4, 5.6, 2.1 Hz, 1H), 3.05 (dd, J = 16.4, 10.8 Hz, 1H), 3.20 (tdd, J = 10.8, 5.6, 3.7 Hz, 1H), 4.06 (t, J = 10.8 Hz, 1H), 4.48 (ddd, J = 10.8, 3.7, 2.1 Hz, 1H), 7.04 (d, J = 2.8 Hz, 1H), 7.12 (d, J = 7.6 Hz, 2H), 7.19 (d, J = 7.6 Hz, 2H), 7.38 (d, J = 2.8 Hz 1H); ^{13}C NMR ($CDCl_3$) δ 21.0, 32.4, 37.5, 71.8, 111.0, 124.8, 125.1, 127.1, 128.5, 129.6, 130.5, 136.9, 137.1, 149.7. HRMS (DART) calcd for $C_{16}H_{15}{}^{79}Br^{35}ClO$ $(M+H)^+$ 336.9995, found 336.9990.



3ia

Compound 3ia (Scheme 3: colorless solid, 40.6 mg, 76% yield, 97% ee). The ee was measured by HPLC (Chiralpak IB, hexane/$CHCl_3$ = 4:1, flow 0.5 mL/min, 254 nm, t_1 = 33.4 min (minor), t_2 = 34.2 min (major)): $[\alpha]^{25}_D$ +26 (*c* 0.98, $CHCl_3$) for 97% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.21 (s, 3H), 2.34 (s, 3H), 2.86−3.01 (m, 1H), 3.12−3.23 (m, 2H), 3.65 (dd, J = 12.6, 9.8 Hz, 1H), 4.21 (br s, 1H), 7.09–7.26 (m, 8H); ^{13}C NMR ($CDCl_3$) δ 21.0, 23.2, 34.8, 41.2, 49.2 (br), 124.5, 125.1, 126.1, 127.0, 128.7, 129.4, 132.2 (br), 136.6, 139.0 (br), 139.7, 170.1. HRMS (ESI) calcd for $C_{18}H_{19}NNaO$ $(M+Na)^+$ 288.1364, found 288.1362.



3ma

Compound 3ma (CAS: 80393-27-7, colorless solid, 9.2 mg, 22% yield). ^{1}H NMR ($CDCl_3$) δ 2.34 (s, 3H), 3.06 (dd, J = 15.4, 8.6 Hz, 2H), 3.33 (dd, J = 15.4, 8.6 Hz, 2H), 3.66 (quint, J = 8.6 Hz, 1H), 7.12 (d, J = 7.6 Hz, 2H), 7.16–7.26 (m, 6H).

8. Synthesis of (–)-(*S*)-equol (4)

A mixture of [Rh(OH)((*S*,*S*)-**L1**)]$_2$ (5.0 mg, 0.010 mmol, 5 mol% of Rh) and *p*-methoxyphenylboronic acid (**2e**) (101 mg, 0.66 mmol), and chromene **1n** (32.4 mg, 0.20 mmol) in 1,4-dioxane (0.40 mL) was stirred at 60 ºC for 1 h. Then, **2e** (101 mg, 0.66 mmol) was added to the mixture every 1 h twice, and the mixture was stirred at 60 °C for 18 h. The mixture was passed through a short column of alumina with CH_2Cl_2 as an eluent, and the solvent was removed on a rotary evaporator. The residue was subjected to preparative TLC on silica gel eluted with EtOAc/hexane (1:20) to give **3ne** (23.0 mg, 43% yield). **Compound 3ne**[7] (Scheme 4: colorless solid, 92% ee). The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 44.0 min (major), t_2 = 54.9 min (minor)): $[\alpha]^{25}_D$ –14 (*c* 0.94, $CHCl_3$) for 92% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.89–3.01 (m, 2H), 3.18 (tdd, *J* = 9.7, 6.8, 3.4 Hz, 1H), 3.78 (s, 3H), 3.81 (s, 3H), 3.98 (t, *J* = 10.5 Hz, 1H), 4.31 (dd, *J* = 10.5, 4.0 Hz, 1H), 4.73 (d, *J* = 2.4 Hz, 1H), 6.48 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.97 (d, *J* = 8.8 Hz, 2H), 6.99 (d, *J* = 8.8 Hz, 1H), 7.17 (d, *J* = 8.8 Hz, 2H).

A mixture of compound **3ne** (19.0 mg, 0.070 mmol) and pyridinium chloride (203.1 mg, 1.8 mmol) was heated at 160 °C for 48 h. After cooling to room temperature, ethyl acetate and water were added to the mixture. The organic layer was washed with water and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue was subjected to preparative TLC on silica gel eluted with EtOAc/hexane (3:1) to give **4** as a colorless solid (12.2 mg, 72% yield). **Compound 4**: $[\alpha]^{25}_D$ –15 (*c* 0.94, EtOH) for 92% ee (*S*). ^{1}H NMR (acetone-d_6) δ 2.77–2.94 (m, 2H), 3.06 (tdd, *J* = 12.0, 6.0, 3.4 Hz, 1H), 3.92 (t, *J* = 10.6 Hz, 1H), 4.18 (ddd, *J* = 10.6, 3.4, 1.8 Hz, 1H), 6.27 (d, *J* = 2.1 Hz, 1H), 6.35 (dd, *J* = 8.3, 2.1 Hz, 1H), 6.81 (d, *J* = 8.4 Hz, 2H), 6.88 (d, *J* = 8.3 Hz, 1H), 7.15 (d, *J* = 8.4 Hz, 2H), 8.14 (br s, 1H), 8.27 (br s, 1H). The ee of obtained compound **4** was determined by chiral HPLC analysis of compound **3ne**, which was derived from **4** as shown below.

Compound **4** (11.4 mg, 0.047 mmol), potassium carbonate (15.6 mg, 0.11 mmol), iodomethane (7.1 μL, 0.11 mmol), and acetone (0.2 mL) were placed in a Schlenk tube under N_2, and the mixture was stirred at 60 ºC overnight. The mixture was passed through a short silica gel pad with EtOAc as an eluent, and the solvent was removed on a rotary evaporator. The residue was subjected to preparative TLC on silica gel eluted with EtOAc/hexane (1:10) to give **3ne** (12.3 mg, 97% yield, 92% ee). The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 39.9 min (major), t_2 = 50.1 min (minor)).

9. Deuterium-labeling experiments

Procedure for eqn (1)

p-Tolylboroxine (**2a'**) (117.9 mg, 0.33 mmol), D_2O (1 mmol, 18 μL), and 1,4-dioxane (0.20 mL) were placed in a Schlenk tube under N_2, and the mixture was stirred at 60 °C for 30 min. After cooling to room temperature, [Rh(OH)((*S*,*S*)-**L1**)]$_2$ (2.5 mg, 0.0050 mmol, 5 mol% of Rh) and 2*H*-choromene (**1a**) (13.2 mg, 0.10 mmol) were added to the tube successively, and the mixture was

stirred at 60 °C for 20 h. The mixture was passed through a short column of alumina with CH_2Cl_2 as an eluent. The solvent was removed on a rotary evaporator, and the residue was subjected to preparative TLC on silica gel [hexane/EtOAc (50:1)]. The deuterium contents of the product was determined by ^1H NMR (CD_2Cl_2). ^2H NMR (CH_2Cl_2) was also measured to determine the position of the deuterium incorporation.

Procedure for eqn (2)

[Rh(OH)((*S*,*S*)-**L1**)]$_2$ (2.5 mg, 0.0050 mmol, 5 mol% of Rh) and **2b-*d*$_5$** (126.9 mg, 1.0 mmol) were placed in a Schlenk tube under N_2. Then, 1,4-dioxane (0.20 mL) and 2*H*-chromene (**1a**) (13.2 mg, 0.10 mmol) were added to the tube successively, and the mixture was stirred at 60 °C for 20 h. The mixture was passed through a short column of alumina with CH_2Cl_2 as an eluent. The solvent was removed on a rotary evaporator, and the residue was subjected to preparative TLC on silica gel [hexane/EtOAc (20:1)]. The deuterium contents of the product was determined by ^1H NMR (CD_2Cl_2). ^2H NMR (CH_2Cl_2) was also measured to determine the position of the deuterium incorporation.

10. References

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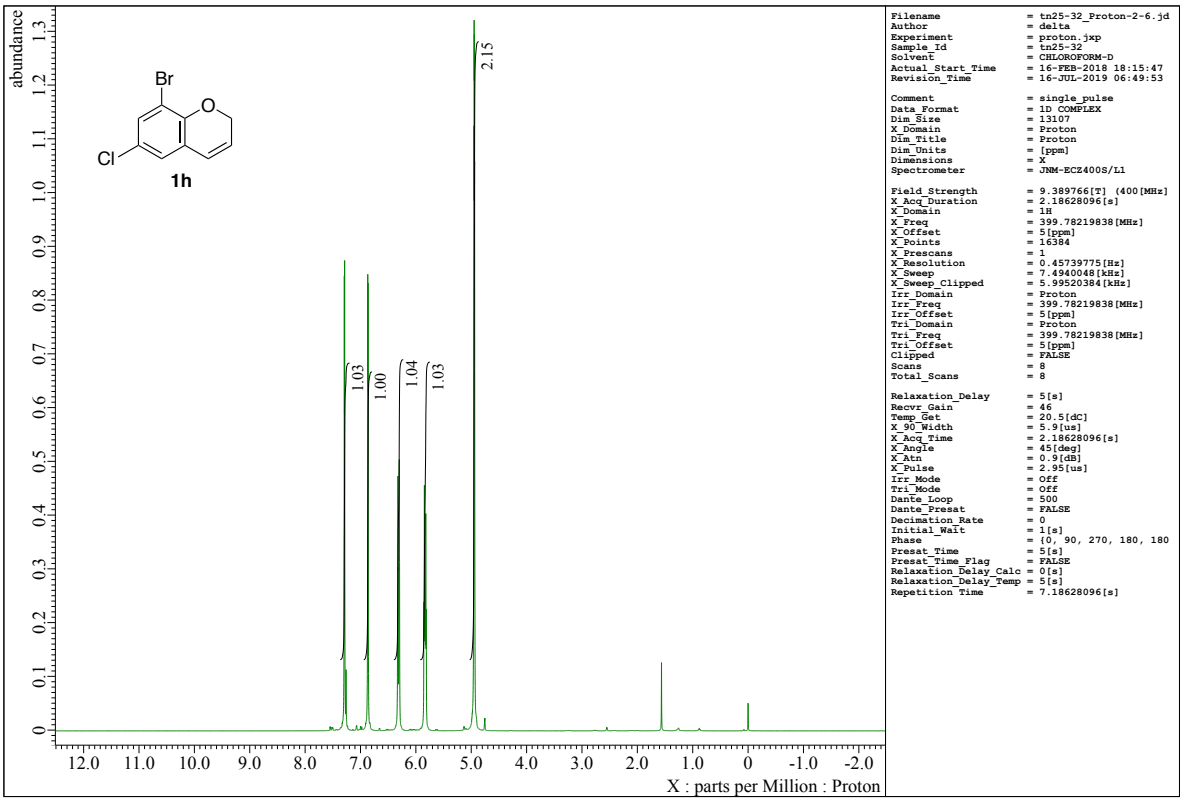
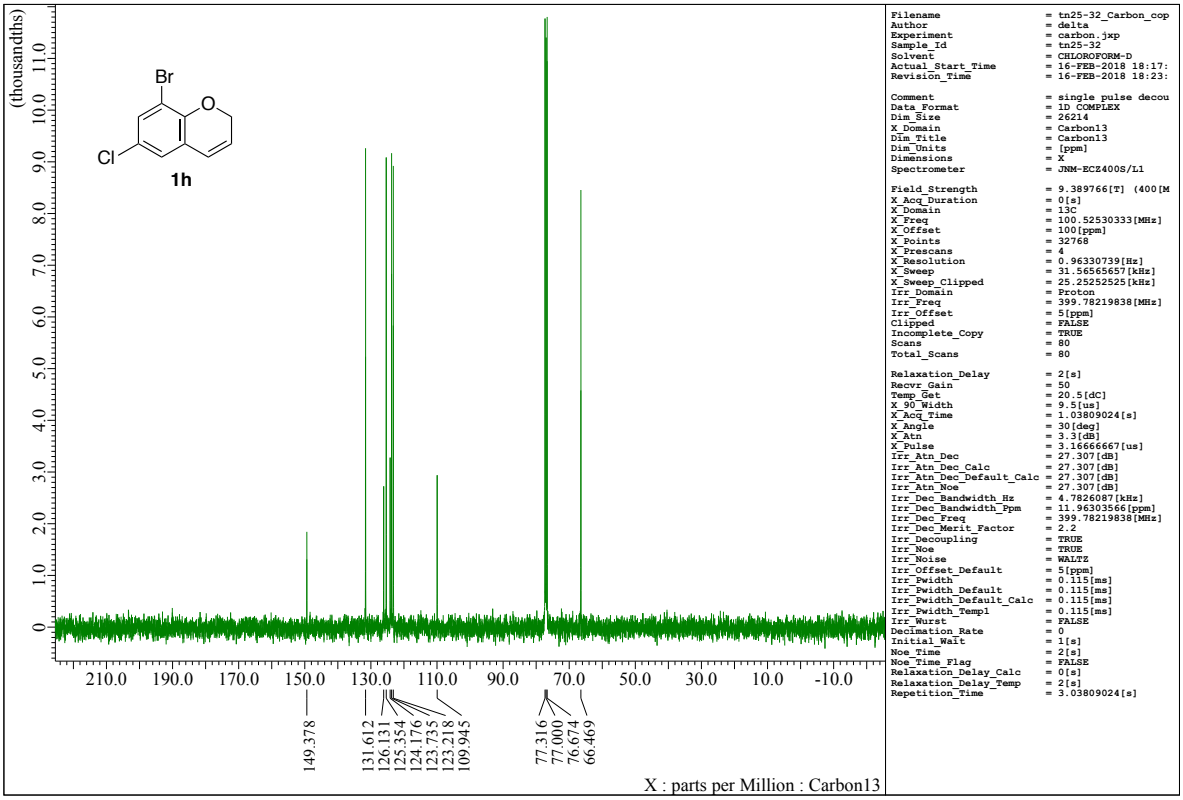
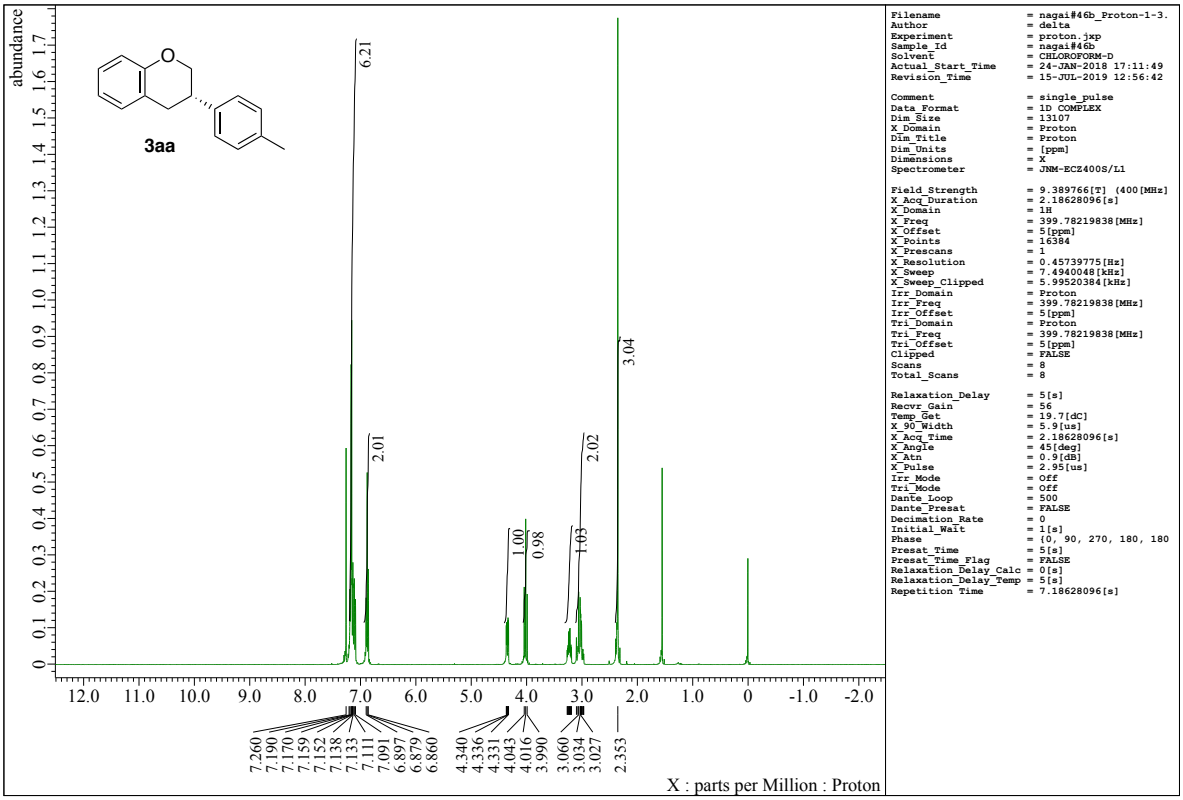
11. NMR spectra and HPLC charts



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7.260 7.190 7.170 7.159 7.152 7.138 7.133 7.111 7.091 6.897 6.879 6.860
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3.060 3.034 3.027
2.353
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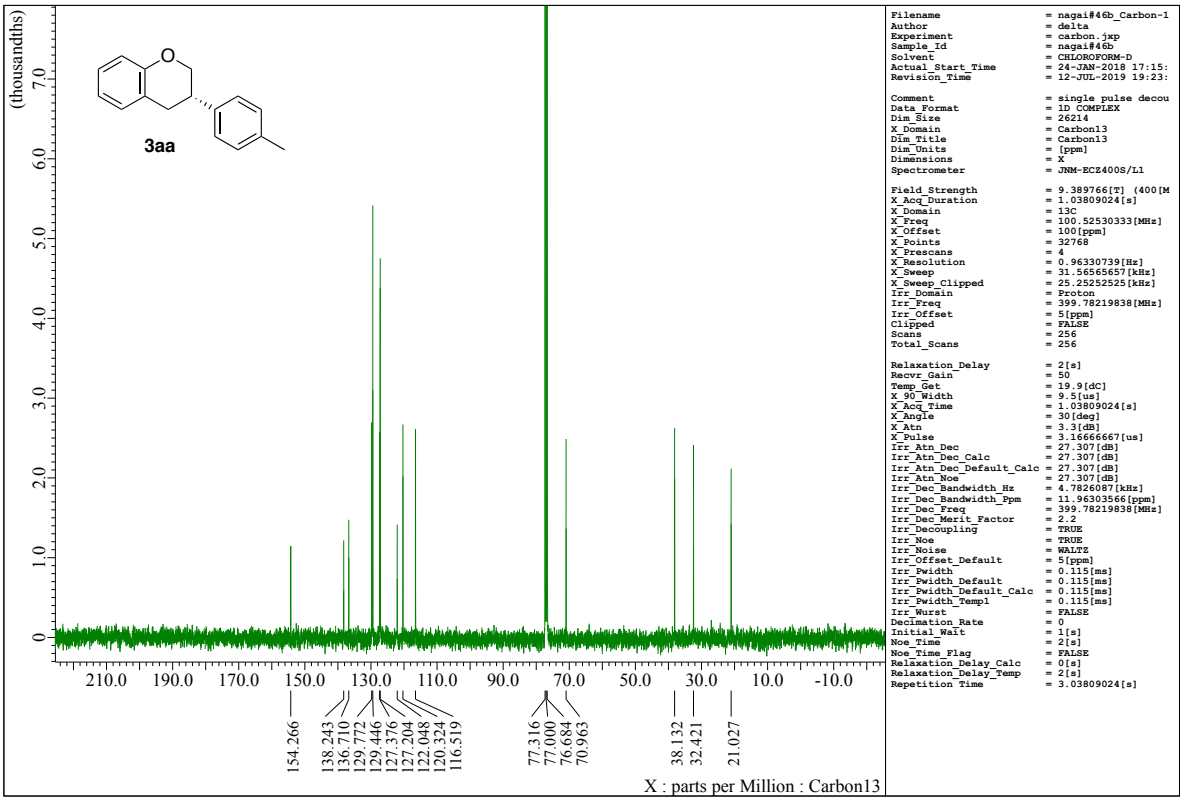
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38.132 32.421 21.027
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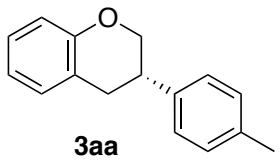
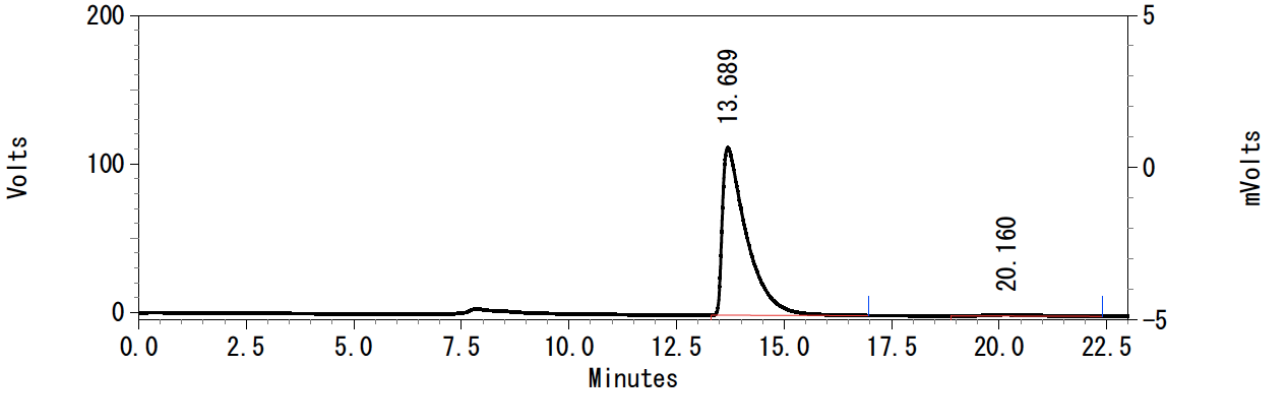
Experiment = carbon.jxp
Sample_Id = nagai#46b
Solvent = CHLOROFORM-D
Actual_Start_Time = 24-JAN-2018 17:15:
Revision_Time = 12-JUL-2019 19:23:
Comment = single_pulse decou
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon13
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[M
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[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 = 19.9[dC]
X_90_Width = 9.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.3[dB]
X_Pulse = 3.16666667[us]
Irr_Atn_Dec = 27.307[dB]
Irr_Atn_Dec_Calc = 27.307[dB]
Irr_Atn_Dec_Default_Calc = 27.307[dB]
Irr_Atn_Noe = 27.307[dB]
Irr_Dec_Bandwidth_Hz = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm = 11.96303566[ppm]
Irr_Dec_Freq = 399.78219838[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noe = TRUE
Irr_Noise = WALTZ
Irr_Offset_Default = 5[ppm]
Irr_Pwidth = 0.115[ms]
Irr_Pwidth_Default = 0.115[ms]
Irr_Pwidth_Default_Calc = 0.115[ms]
Irr_Pwidth_Templ = 0.115[ms]
Irr_Wurst = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Noe_Time = 2[s]
Noe_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 2[s]
Repetition_Time = 3.03809024[s]

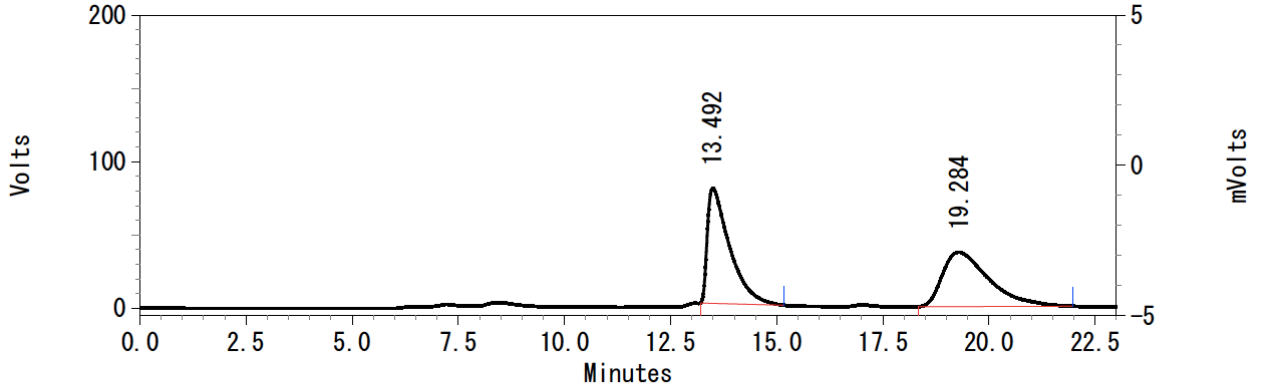


3aa



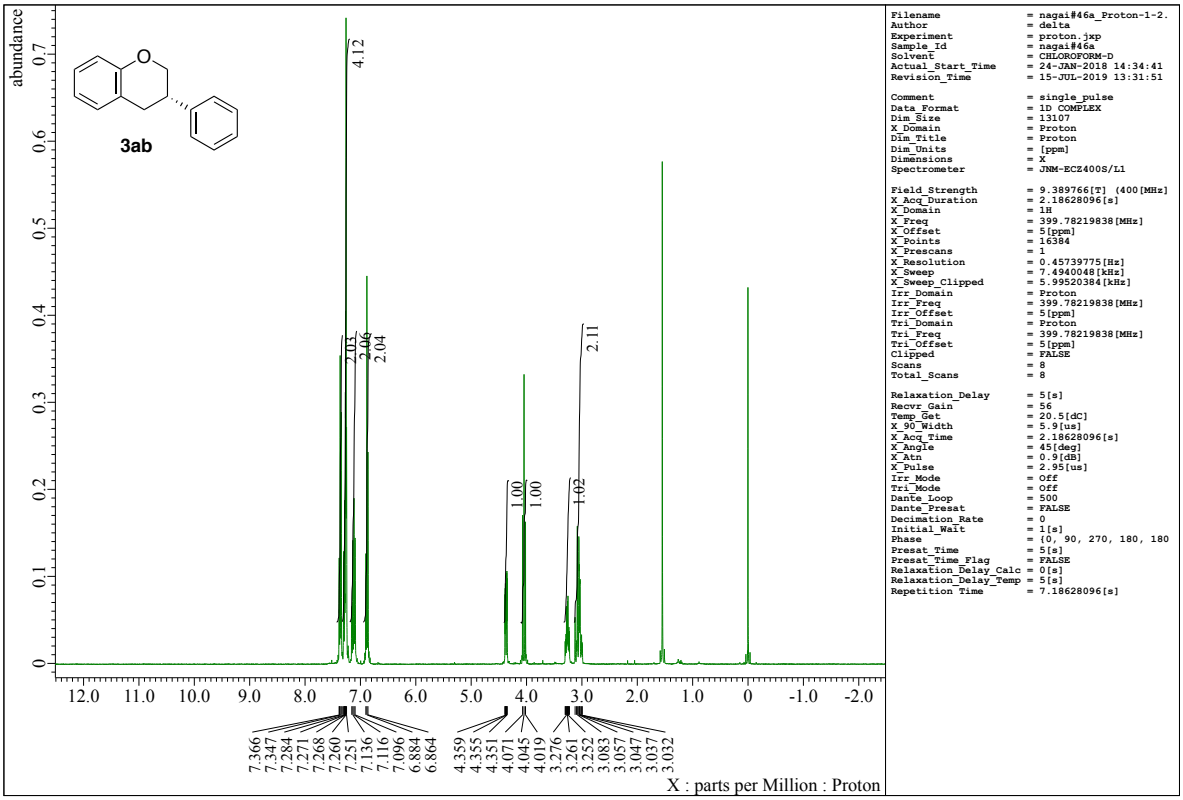
UV Results

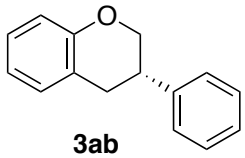
Pk #	Retention Time	Area	Area Percent	Height
1	13.689	4404676	98.523	112785
2	20.160	66033	1.477	863
Totals		4470709	100.000	113648



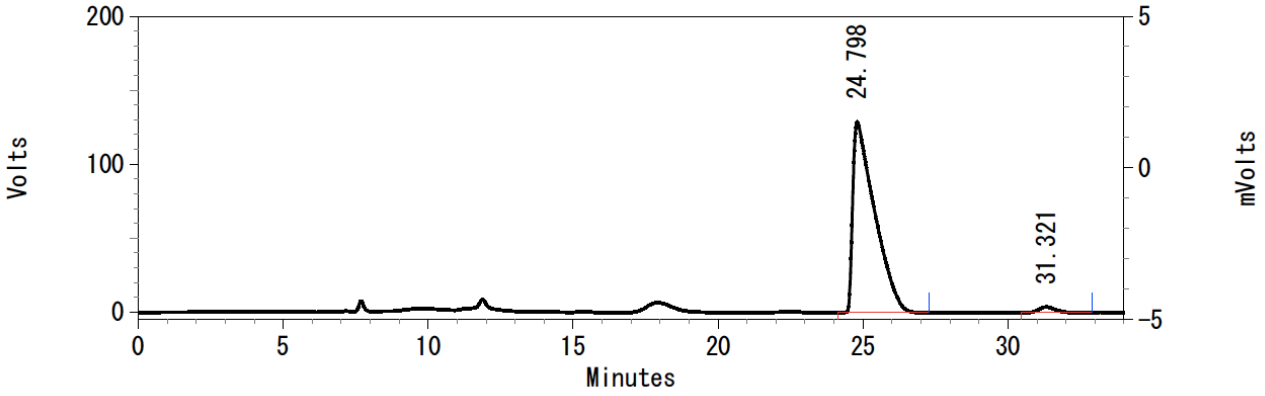
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	13.492	2867768	50.333	78659
2	19.284	2829852	49.667	37126
Totals		5697620	100.000	115785


abundance
3ab
4.12
2.03
2.06
2.04
1.00
1.00
1.02
2.11
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
7.366 7.347 7.284 7.271 7.268 7.260 7.251 7.136 7.116 7.096 6.884 6.864
4.359 4.355 4.351 4.071 4.045 4.019 3.276 3.261 3.252 3.083 3.057 3.047 3.037 3.032
X : parts per Million : Proton
Filename = nagai#46a_Proton-1-2.
Author = delta
Experiment = proton.jxp
Sample_Id = nagai#46a
Solvent = CHLOROFORM-D
Actual_Start_Time = 24-JAN-2018 14:34:41
Revision_Time = 15-JUL-2019 13:31:51
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz]
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[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 = 56
Temp_Get = 20.5[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Loop = 500
Dante_Presat = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Phase = {0, 90, 270, 180, 180
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]

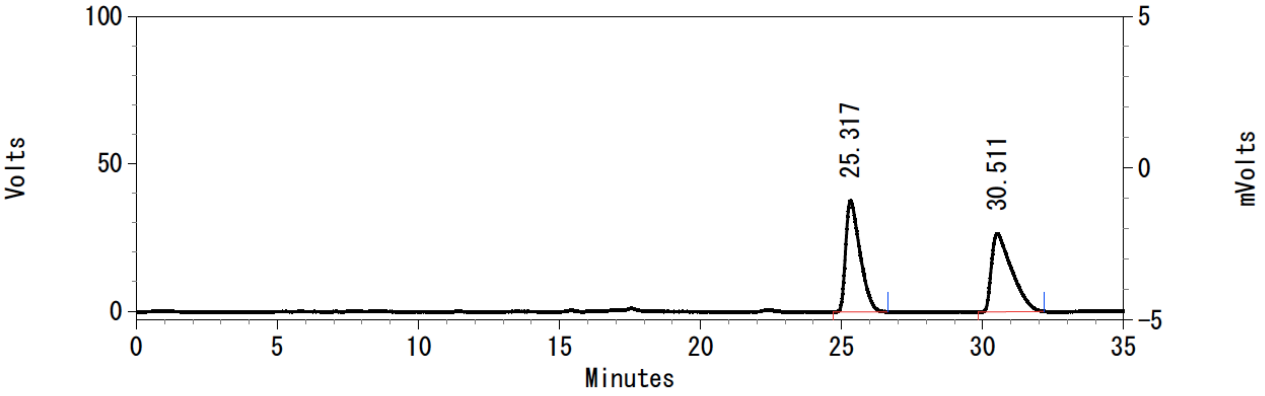


3ab



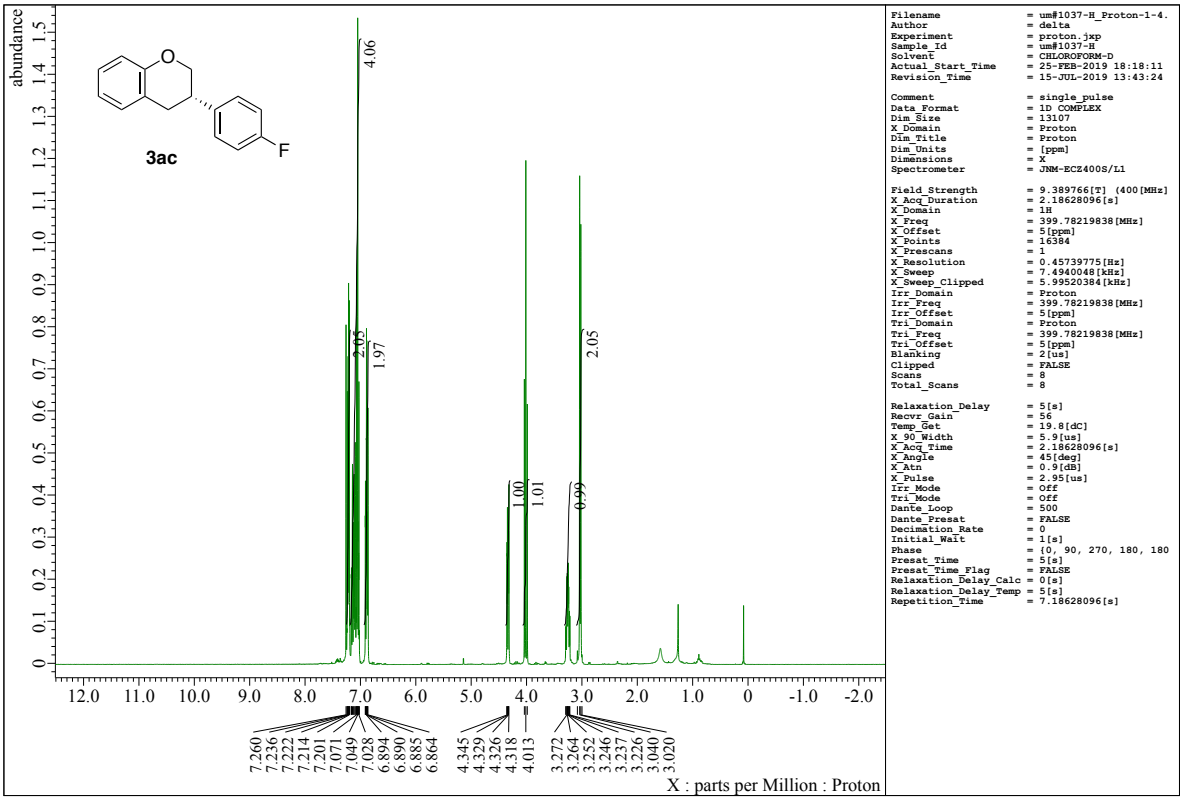
UV-970 Results

Pk #	Retention Time	Area	Area Percent	Height
1	24.798	6737664	97.780	128970
2	31.321	152972	2.220	3811
Totals		6890636	100.000	132781

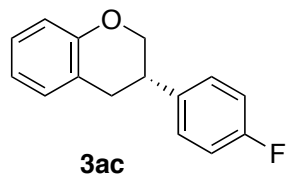


UV-970 Results

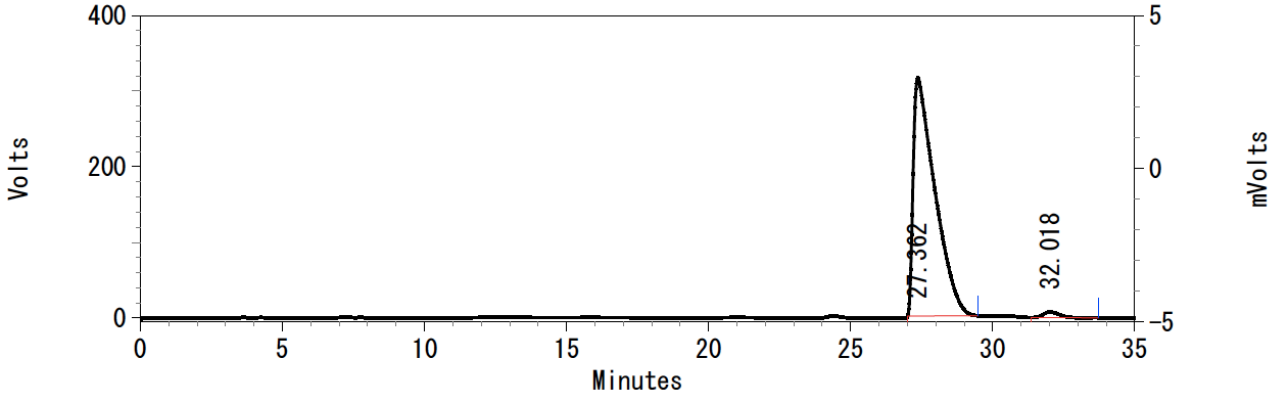
Pk #	Retention Time	Area	Area Percent	Height
1	25.317	1328196	50.029	37752
2	30.511	1326635	49.971	26497
Totals		2654831	100.000	64249




```
Filename                = um#1037-H_Proton-1-4.
Author                  = delta
Experiment              = proton.jxp
Sample_Id               = um#1037-H
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 25-FEB-2019 18:18:11
Revision_Time           = 15-JUL-2019 13:43:24

Comment                 = single_pulse
Data_Format             = 1D COMPLEX
Dim_Size                = 13107
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.389766[T] (400[MHz]
X_Acq_Duration          = 2.18628096[s]
X_Domain                = 1H
X_Freq                  = 399.78219838[MHz]
X_Offset                = 5[ppm]
X_Points                = 16384
X_Prescans              = 1
X_Resolution            = 0.45739775[Hz]
X_Sweep                 = 7.4940048[kHz]
X_Sweep_Clipped         = 5.99520384[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]
Blanking                = 2[us]
Clipped                 = FALSE
Scans                   = 8
Total_Scans             = 8

Relaxation_Delay        = 5[s]
Recvr_Gain              = 56
Temp_Get                = 19.8[dC]
X_90_Width              = 5.9[us]
X_Acq_Time              = 2.18628096[s]
X_Angle                 = 45[deg]
X_Atn                   = 0.9[dB]
X_Pulse                 = 2.95[us]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 500
Dante_Presat            = FALSE
Decimation_Rate         = 0
Initial_Wait            = 1[s]
Phase                   = {0, 90, 270, 180, 180
Presat_Time             = 5[s]
Presat_Time_Flag        = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 5[s]
Repetition_Time         = 7.18628096[s]
```

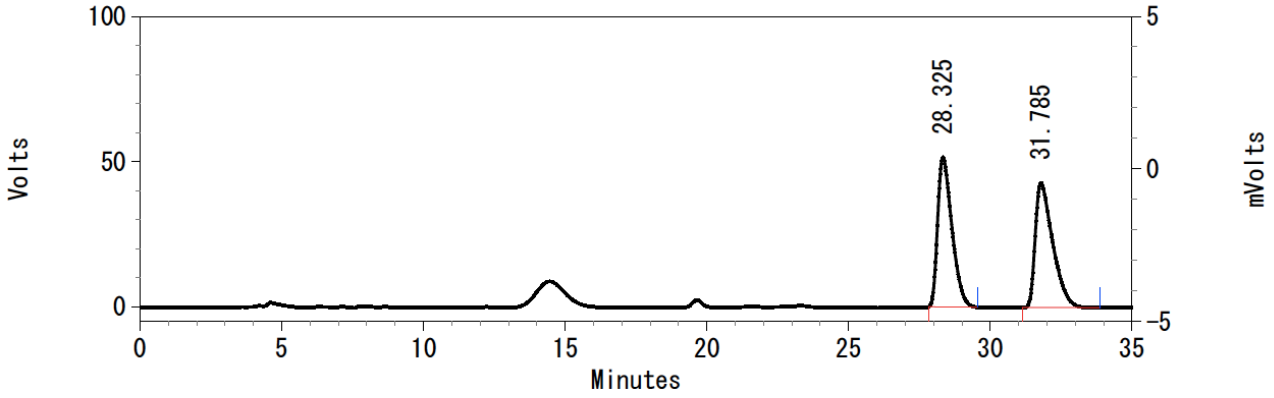


3ac



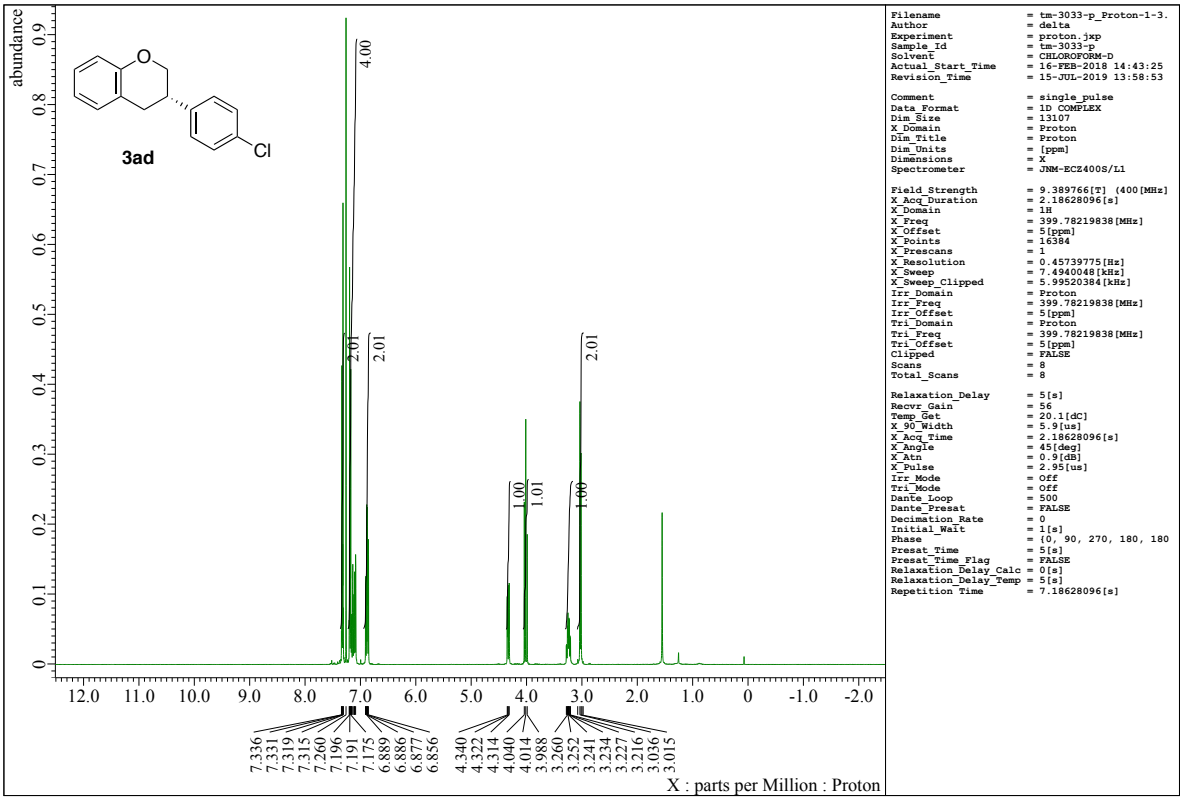
UV-970 Results

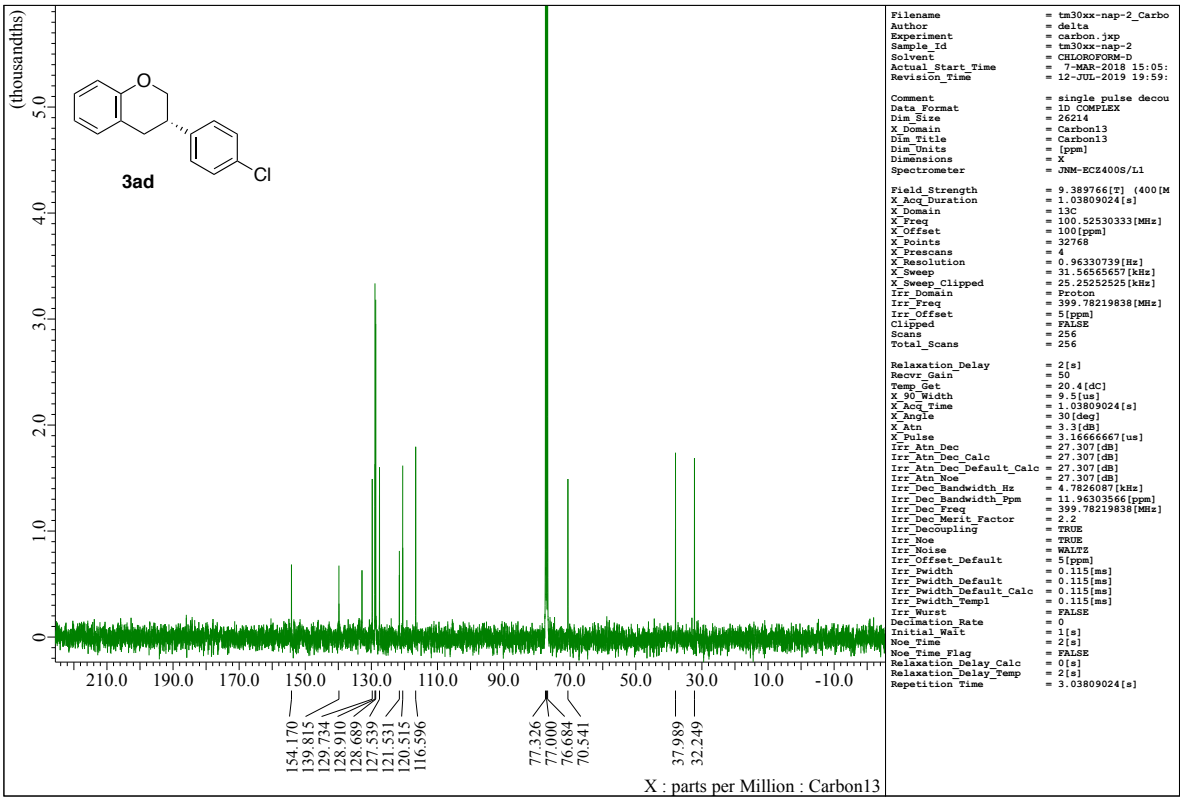
Pk #	Retention Time	Area	Area Percent	Height
1	27.362	16637185	98.358	314753
2	32.018	277797	1.642	7393
Totals		16914982	100.000	322146

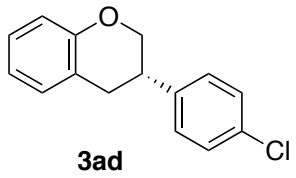


UV-970 Results

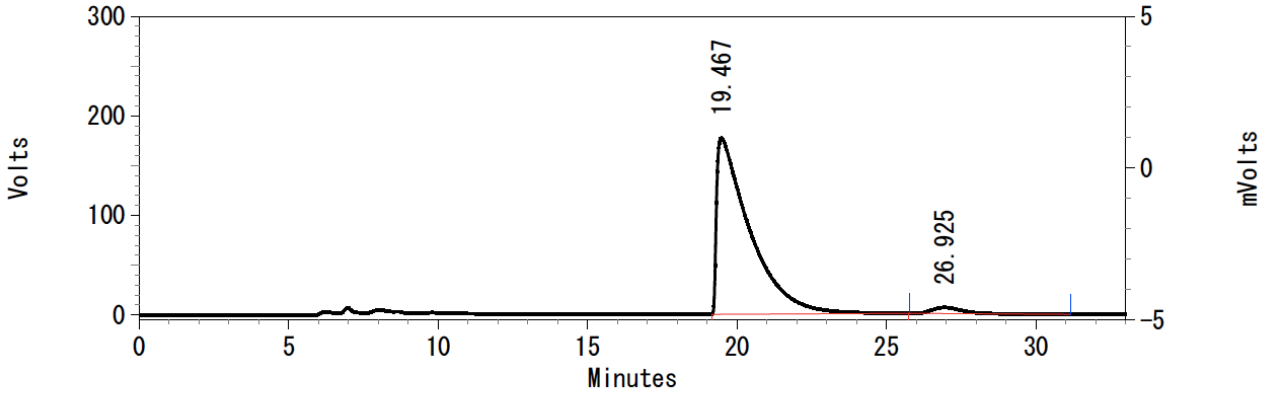
Pk #	Retention Time	Area	Area Percent	Height
1	28.325	1800953	49.550	51527
2	31.785	1833653	50.450	42913
Totals		3634606	100.000	94440


abundance
3ad
O
Cl
4.00
2.01
2.01
2.01
1.00
1.01
1.00
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
7.336 7.331 7.319 7.315 7.260 7.196 7.191 7.175 6.889 6.886 6.877 6.856
4.340 4.322 4.314 4.040 4.014 3.988 3.260 3.252 3.241 3.234 3.227 3.216 3.036 3.015
X : parts per Million : Proton
Filename = tm-3033-p_Proton-1-3.
Author = delta
Experiment = proton.jxp
Sample_Id = tm-3033-p
Solvent = CHLOROFORM-D
Actual_Start_Time = 16-FEB-2018 14:43:25
Revision_Time = 15-JUL-2019 13:58:53
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz]
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.9952038[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 = 56
Temp_Get = 20.1[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Loop = 500
Dante_Preset = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Phase = {0, 90, 270, 180, 180
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]


(thousandths)
3ad
O
Cl
210.0 190.0 170.0 150.0 130.0 110.0 90.0 70.0 50.0 30.0 10.0 -10.0
154.170 139.815 129.734 128.910 128.689 127.539 121.531 120.515 116.596
77.326 77.000 76.684 70.541
37.989 32.249
X : parts per Million : Carbon13
Filename = tm30xx-nap-2_Carbo
Author = delta
Experiment = carbon.jxp
Sample_Id = tm30xx-nap-2
Solvent = CHLOROFORM-D
Actual_Start_Time = 7-MAR-2018 15:05:
Revision_Time = 12-JUL-2019 19:59:
Comment = single pulse decou
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon13
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[M
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[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 = 20.4[dC]
X_90_Width = 9.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.3[dB]
X_Pulse = 3.16666667[us]
Irr_Atn_Dec = 27.307[dB]
Irr_Atn_Dec_Calc = 27.307[dB]
Irr_Atn_Dec_Default_Calc = 27.307[dB]
Irr_Atn_Noe = 27.307[dB]
Irr_Dec_Bandwidth_Hz = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm = 11.96303566[ppm]
Irr_Dec_Freq = 399.78219838[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noe = TRUE
Irr_Noise = WALTZ
Irr_Offset_Default = 5[ppm]
Irr_Pwidth = 0.115[ms]
Irr_Pwidth_Default = 0.115[ms]
Irr_Pwidth_Default_Calc = 0.115[ms]
Irr_Pwidth_Templ = 0.115[ms]
Irr_Wurst = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Noe_Time = 2[s]
Noe_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 2[s]
Repetition_Time = 3.03809024[s]

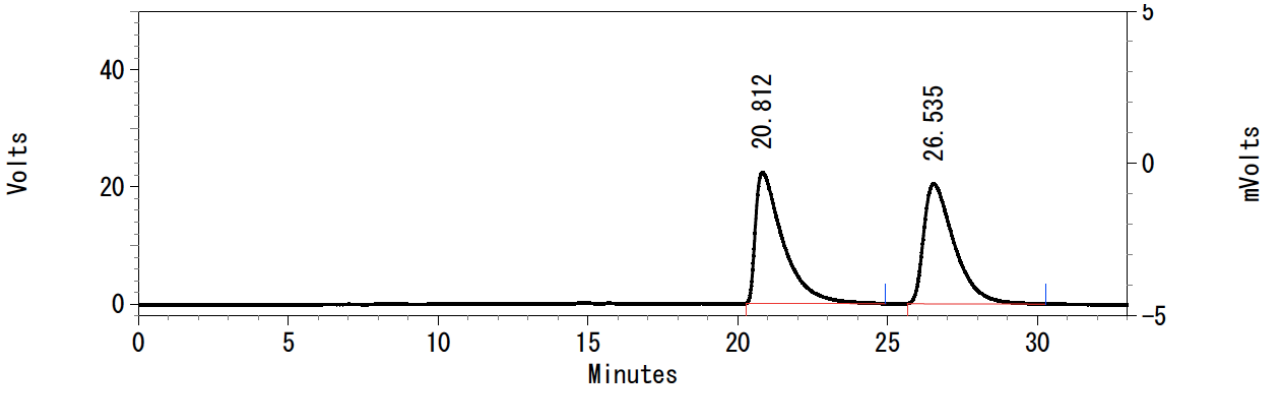


3ad



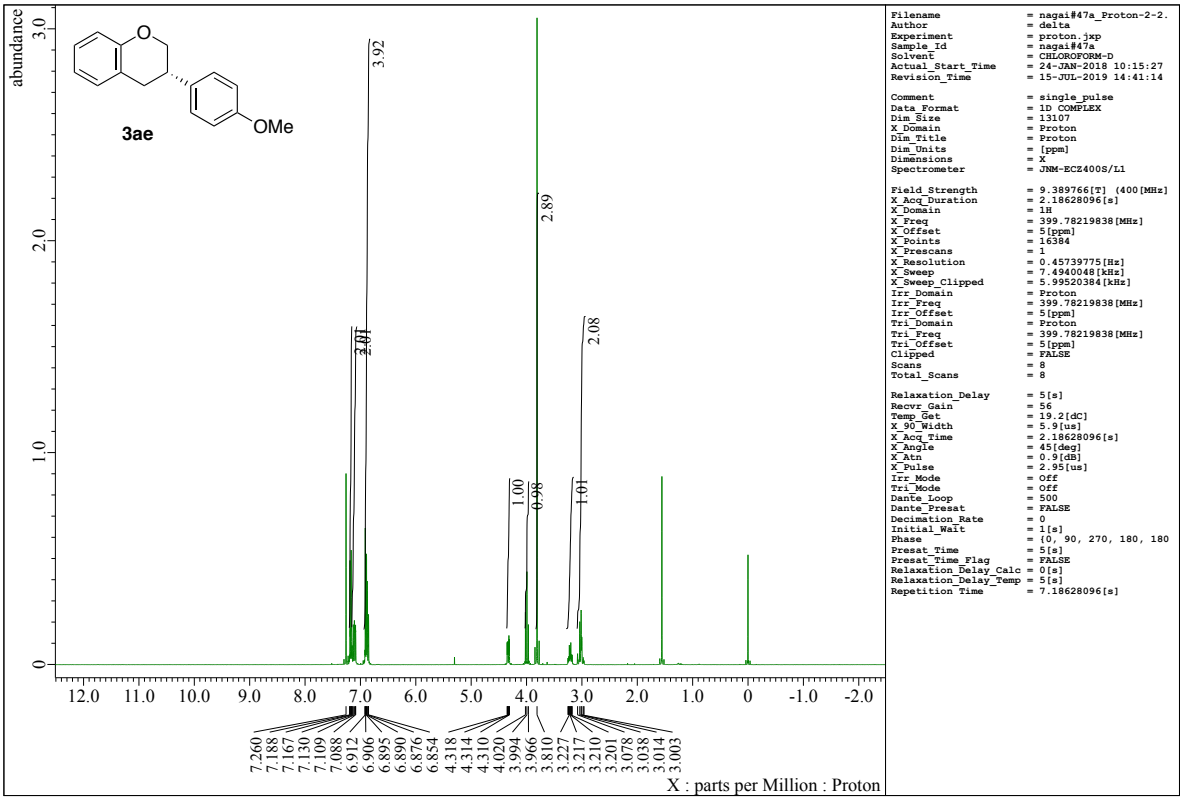
UV-970 Results

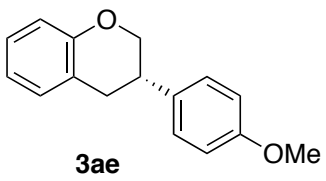
Pk #	Retention Time	Area	Area Percent	Height
1	19.467	13670263	96.984	176900
2	26.925	425082	3.016	6112
Totals		14095345	100.000	183012



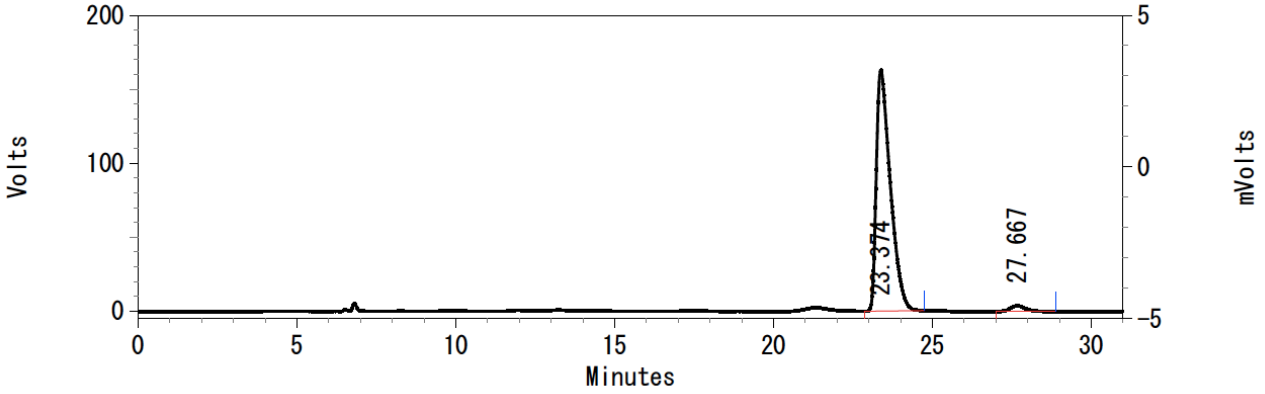
UV-970 Results

Pk #	Retention Time	Area	Area Percent	Height
1	20.812	1434540	49.710	22364
2	26.535	1451271	50.290	20456
Totals		2885811	100.000	42820


abundance
3.0
2.0
1.0
0
O
OMe
3ae
3.92
2.89
2.08
2.01
2.01
1.00
0.98
1.01
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
7.260
7.188
7.167
7.130
7.109
7.088
6.912
6.906
6.895
6.890
6.876
6.854
4.318
4.314
4.310
4.020
3.994
3.966
3.810
3.227
3.217
3.210
3.201
3.078
3.038
3.014
3.003
X : parts per Million : Proton
Filename = nagai#47a_Proton-2-2.
Author = delta
Experiment = proton.jxp
Sample_Id = nagai#47a
Solvent = CHLOROFORM-D
Actual_Start_Time = 24-JAN-2018 10:15:27
Revision_Time = 15-JUL-2019 14:41:14
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz]
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[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 = 56
Temp_Get = 19.2[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Loop = 500
Dante_Presat = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Phase = {0, 90, 270, 180, 180
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]

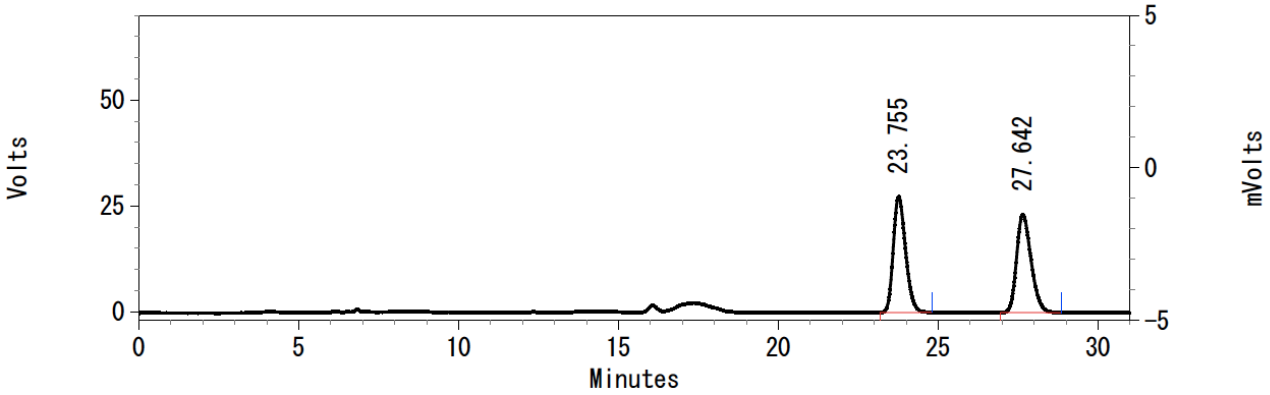


3ae



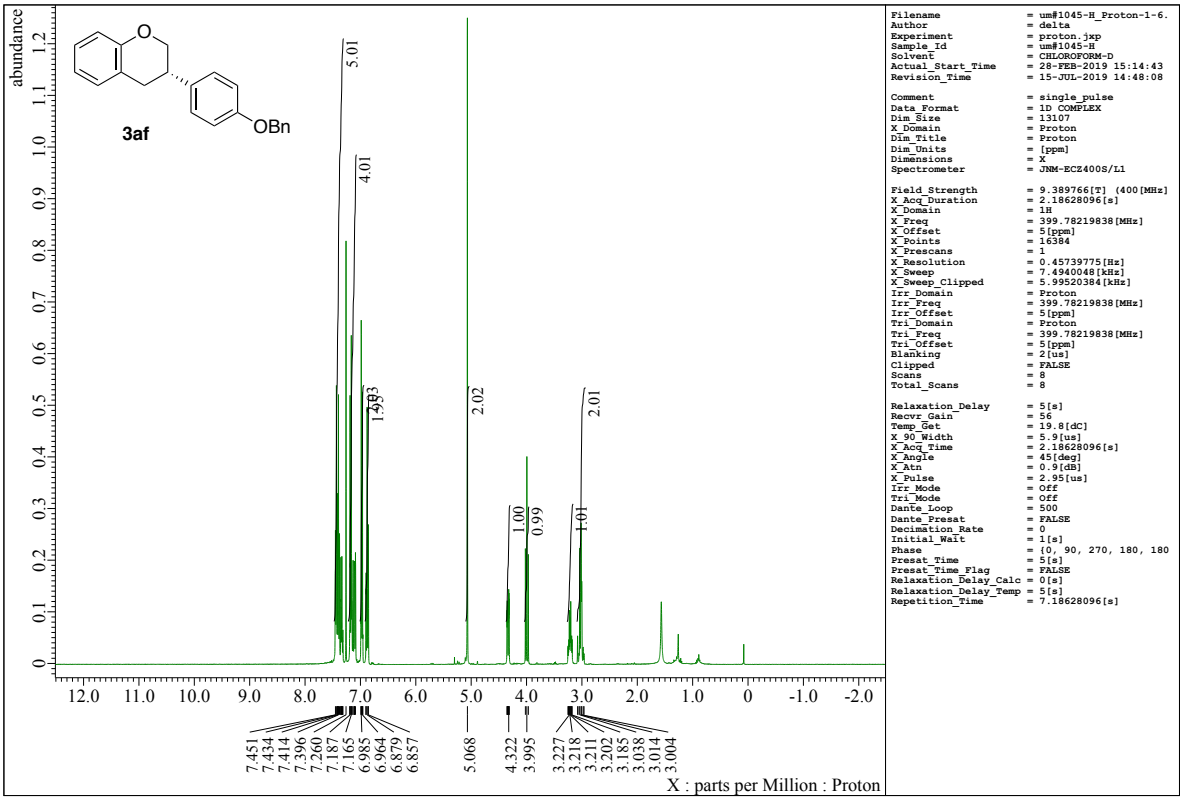
UV-970 Results

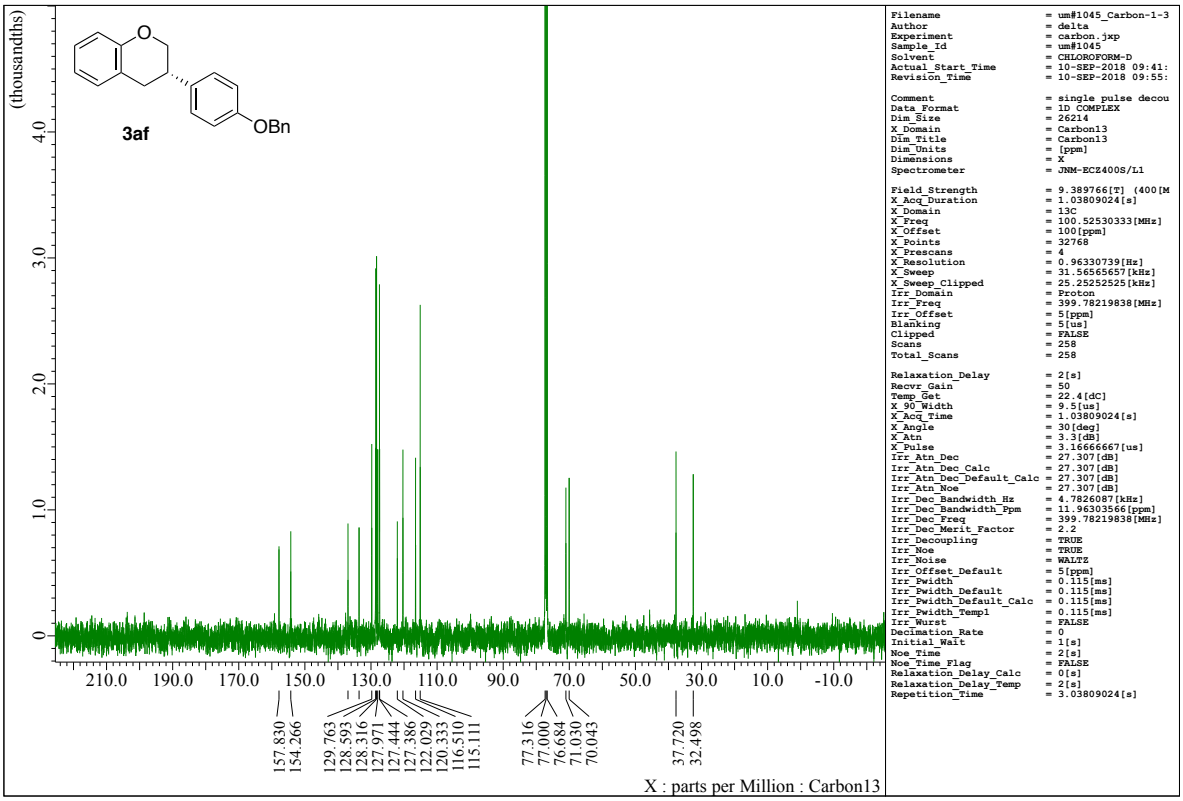
Pk #	Retention Time	Area	Area Percent	Height
1	23.374	4993928	97.550	163030
2	27.667	125405	2.450	3855
Totals		5119333	100.000	166885

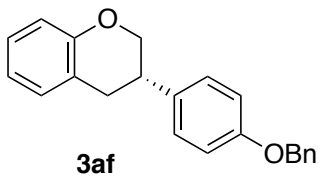


UV-970 Results

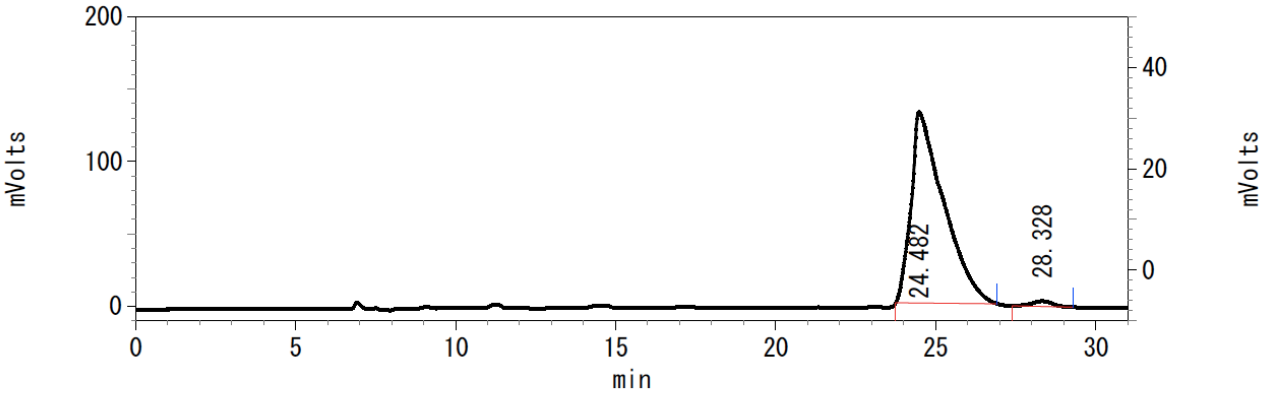
Pk #	Retention Time	Area	Area Percent	Height
1	23.755	730655	50.095	27362
2	27.642	727896	49.905	23158
Totals		1458551	100.000	50520


3af
OBn
abundance
5.01
4.01
1.93
1.93
2.02
1.00
0.99
1.01
2.01
7.451
7.434
7.414
7.396
7.260
7.187
7.165
6.985
6.964
6.879
6.857
5.068
4.322
3.995
3.227
3.218
3.211
3.202
3.185
3.038
3.014
3.004
X : parts per Million : Proton
Filename = um#1045-H_Proton-1-6.
Author = delta
Experiment = proton.jxp
Sample_Id = um#1045-H
Solvent = CHLOROFORM-D
Actual_Start_Time = 28-FEB-2019 15:14:43
Revision_Time = 15-JUL-2019 14:48:08
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz]
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.9952038[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]
Blanking = 2[us]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 19.8[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Loop = 500
Dante_Presat = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Phase = {0, 90, 270, 180, 180
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]

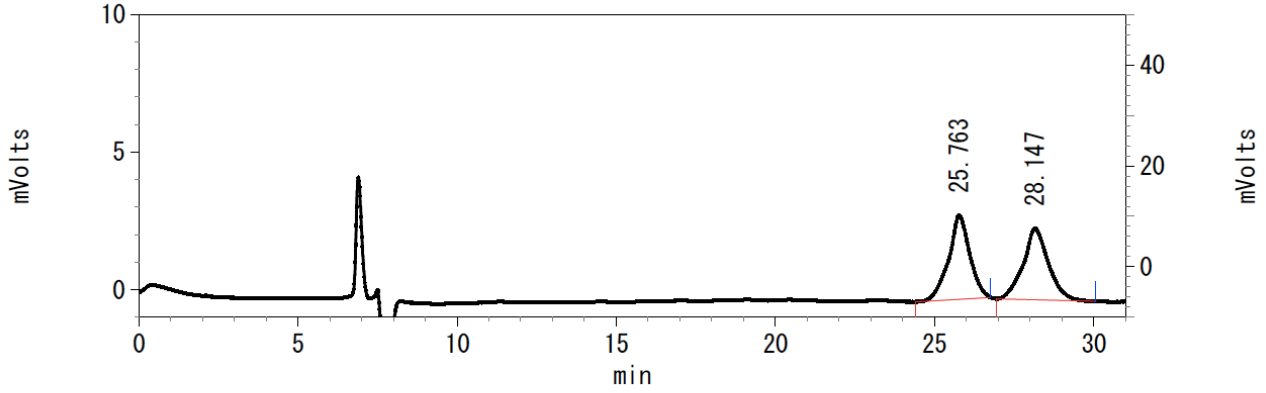

3af
OBn
(thousandths)
157.830
154.266
129.763
128.593
128.316
127.971
127.444
127.386
122.029
120.333
116.510
115.111
77.316
77.000
76.684
71.030
70.043
37.720
32.498
X : parts per Million : Carbon13
Filename = um#1045_Carbon-1-3
Author = delta
Experiment = carbon.jxp
Sample_Id = um#1045
Solvent = CHLOROFORM-D
Actual_Start_Time = 10-SEP-2018 09:41:
Revision_Time = 10-SEP-2018 09:55:
Comment = single pulse decou
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon13
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[M
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Blanking = 5[us]
Clipped = FALSE
Scans = 258
Total_Scans = 258
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 22.4[dC]
X_90_Width = 9.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.3[dB]
X_Pulse = 3.16666667[us]
Irr_Atn_Dec = 27.307[dB]
Irr_Atn_Dec_Calc = 27.307[dB]
Irr_Atn_Dec_Default_Calc = 27.307[dB]
Irr_Atn_Noe = 27.307[dB]
Irr_Dec_Bandwidth_Hz = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm = 11.96303566[ppm]
Irr_Dec_Freq = 399.78219838[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noe = TRUE
Irr_Noise = WALTZ
Irr_Offset_Default = 5[ppm]
Irr_Pwidth = 0.115[ms]
Irr_Pwidth_Default = 0.115[ms]
Irr_Pwidth_Default_Calc = 0.115[ms]
Irr_Pwidth_Templ = 0.115[ms]
Irr_Wurst = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Noe_Time = 2[s]
Noe_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 2[s]
Repetition_Time = 3.03809024[s]



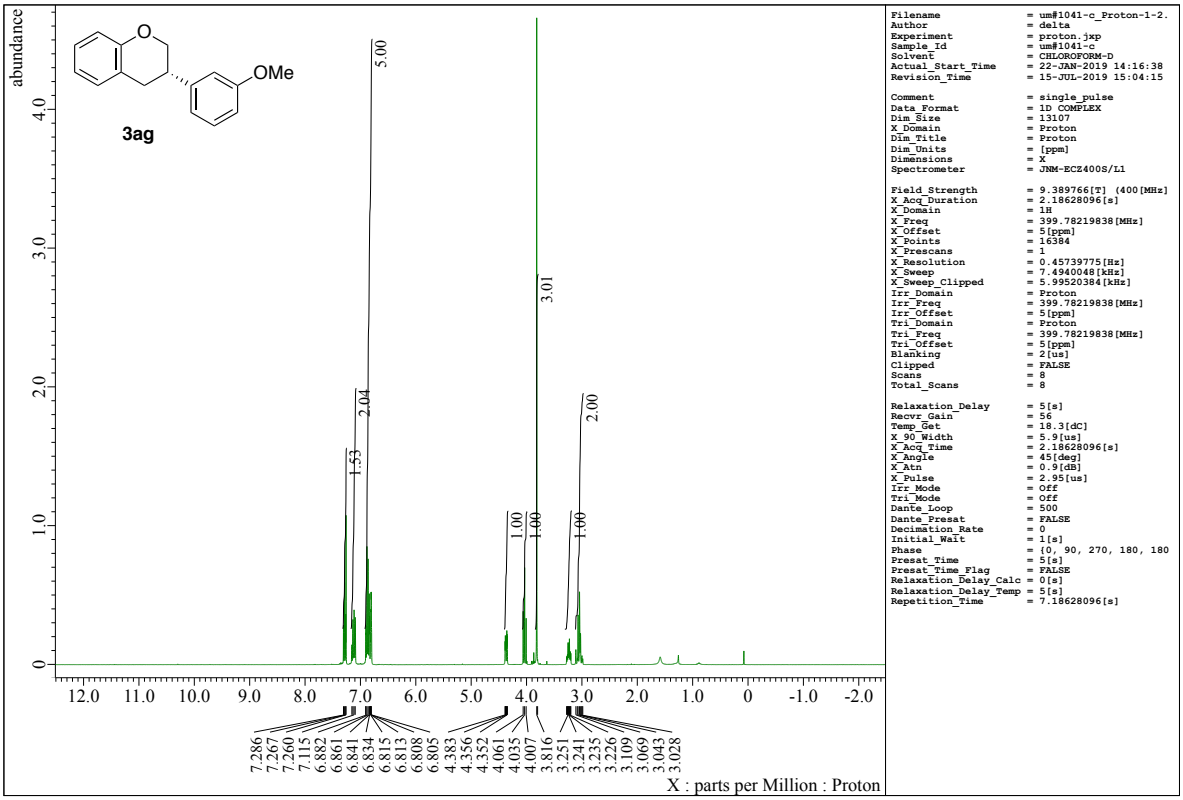
3af



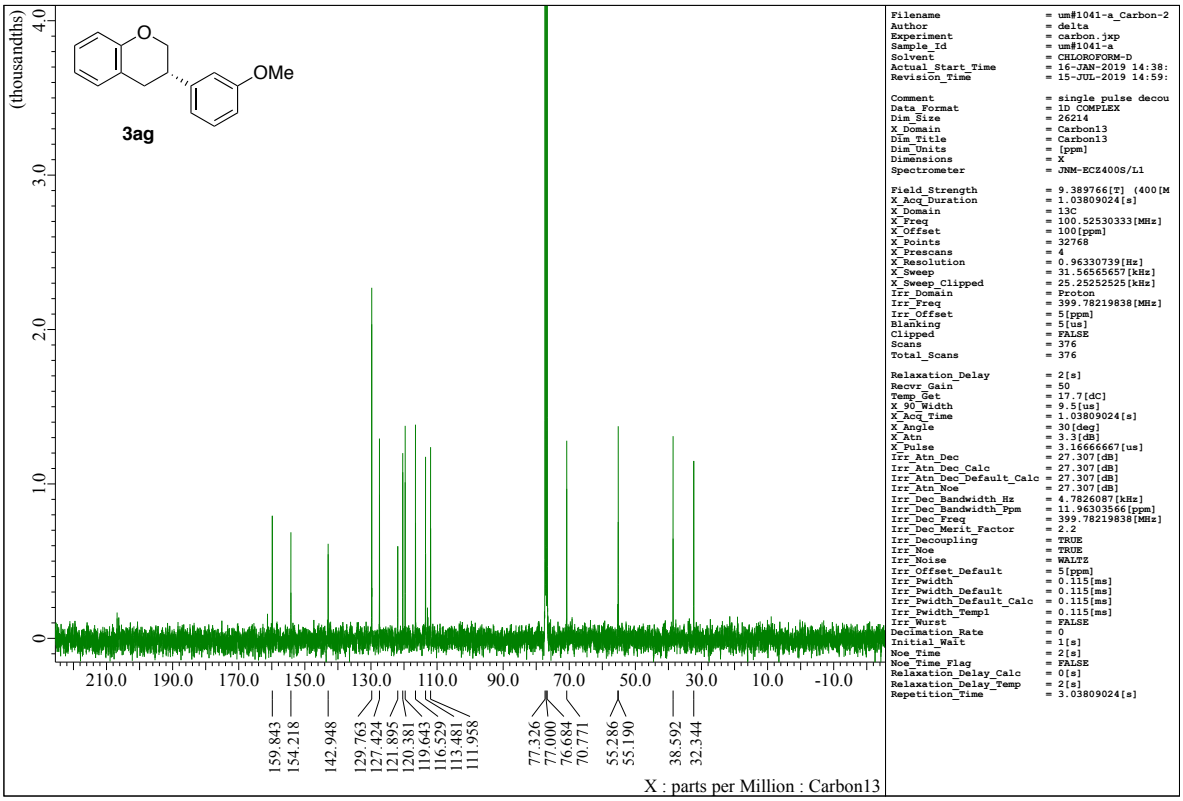
Pk #	Retention Time	Area	Area Percent
1	24.482	9417697	98.272
2	28.328	165570	1.728



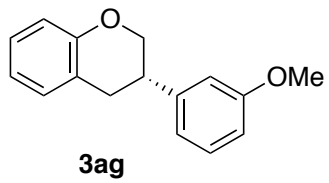
Pk #	Retention Time	Area	Area Percent
1	25.763	141088	49.644
2	28.147	143111	50.356



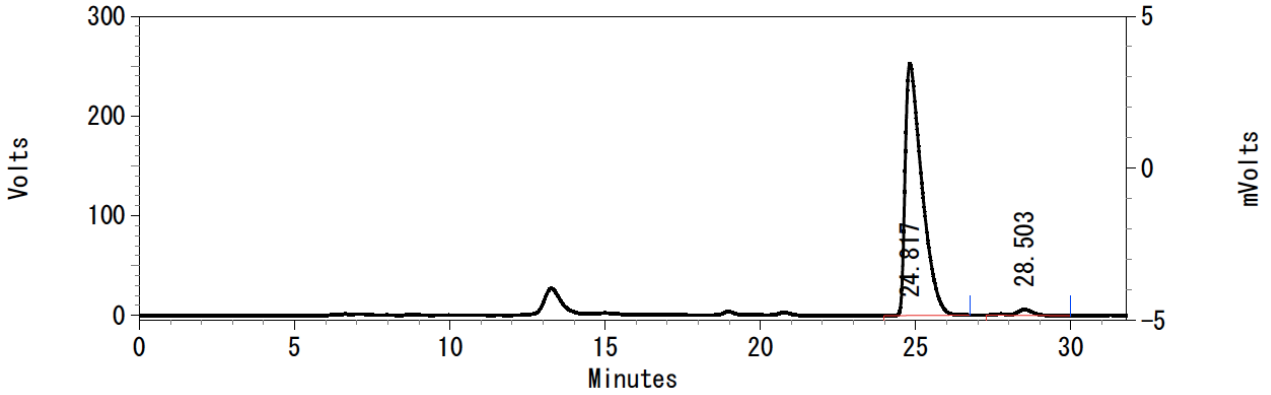

```
Filename                = um#1041-c_Proton-1-2.
Author                  = delta
Experiment              = proton.jxp
Sample_Id               = um#1041-c
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 22-JAN-2019 14:16:38
Revision_Time           = 15-JUL-2019 15:04:15

Comment                 = single_pulse
Data_Format             = 1D COMPLEX
Dim_Size                = 13107
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.389766[T] (400[MHz]
X_Acq_Duration          = 2.18628096[s]
X_Domain                = 1H
X_Freq                  = 399.78219838[MHz]
X_Offset                = 5[ppm]
X_Points                = 16384
X_Prescans              = 1
X_Resolution            = 0.45739775[Hz]
X_Sweep                 = 7.4940048[kHz]
X_Sweep_Clipped         = 5.9952038[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]
Blanking                = 2[us]
Clipped                 = FALSE
Scans                   = 8
Total_Scans             = 8

Relaxation_Delay        = 5[s]
Recvr_Gain              = 56
Temp_Get                = 18.3[dC]
X_90_Width              = 5.9[us]
X_Acq_Time              = 2.18628096[s]
X_Angle                 = 45[deg]
X_Atn                   = 0.9[dB]
X_Pulse                 = 2.95[us]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 500
Dante_Presat            = FALSE
Decimation_Rate         = 0
Initial_Wait            = 1[s]
Phase                   = {0, 90, 270, 180, 180
Presat_Time             = 5[s]
Presat_Time_Flag        = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 5[s]
Repetition_Time         = 7.18628096[s]
```




```
Filename                = um#1041-a_Carbon-2
Author                  = delta
Experiment              = carbon.jxp
Sample_Id               = um#1041-a
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 16-JAN-2019 14:38:
Revision_Time           = 15-JUL-2019 14:59:

Comment                 = single pulse decou
Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Carbon13
Dim_Title               = Carbon13
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.389766[T] (400[M
X_Acq_Duration          = 1.03809024[s]
X_Domain                = 13C
X_Freq                  = 100.52530333[MHz]
X_Offset                = 100[ppm]
X_Points                = 32768
X_Prescans              = 4
X_Resolution            = 0.96330739[Hz]
X_Sweep                 = 31.56565657[kHz]
X_Sweep_Clipped         = 25.25252525[kHz]
Irr_Domain              = Proton
Irr_Freq                = 399.78219838[MHz]
Irr_Offset              = 5[ppm]
Blanking                = 5[us]
Clipped                 = FALSE
Scans                   = 376
Total_Scans             = 376

Relaxation_Delay        = 2[s]
Recvr_Gain              = 50
Temp_Get                = 17.7[dC]
X_90_Width              = 9.5[us]
X_Acq_Time              = 1.03809024[s]
X_Angle                 = 30[deg]
X_Atn                   = 3.3[dB]
X_Pulse                 = 3.16666667[us]
Irr_Atn_Dec             = 27.307[dB]
Irr_Atn_Dec_Calc        = 27.307[dB]
Irr_Atn_Dec_Default_Calc = 27.307[dB]
Irr_Atn_Noe             = 27.307[dB]
Irr_Dec_Bandwidth_Hz    = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm   = 11.96303566[ppm]
Irr_Dec_Freq            = 399.78219838[MHz]
Irr_Dec_Merit_Factor    = 2.2
Irr_Decoupling          = TRUE
Irr_Noe                 = TRUE
Irr_Noise               = WALTZ
Irr_Offset_Default      = 5[ppm]
Irr_Pwidth              = 0.115[ms]
Irr_Pwidth_Default      = 0.115[ms]
Irr_Pwidth_Default_Calc = 0.115[ms]
Irr_Pwidth_Templ        = 0.115[ms]
Irr_Wurst               = FALSE
Decimation_Rate         = 0
Initial_Wait            = 1[s]
Noe_Time                = 2[s]
Noe_Time_Flag           = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 2[s]
Repetition_Time         = 3.03809024[s]
```

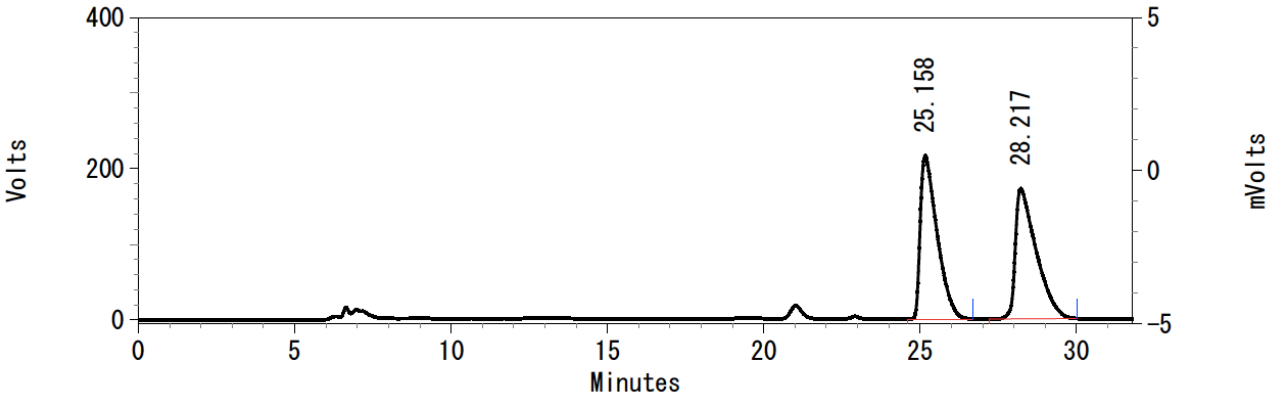


3ag



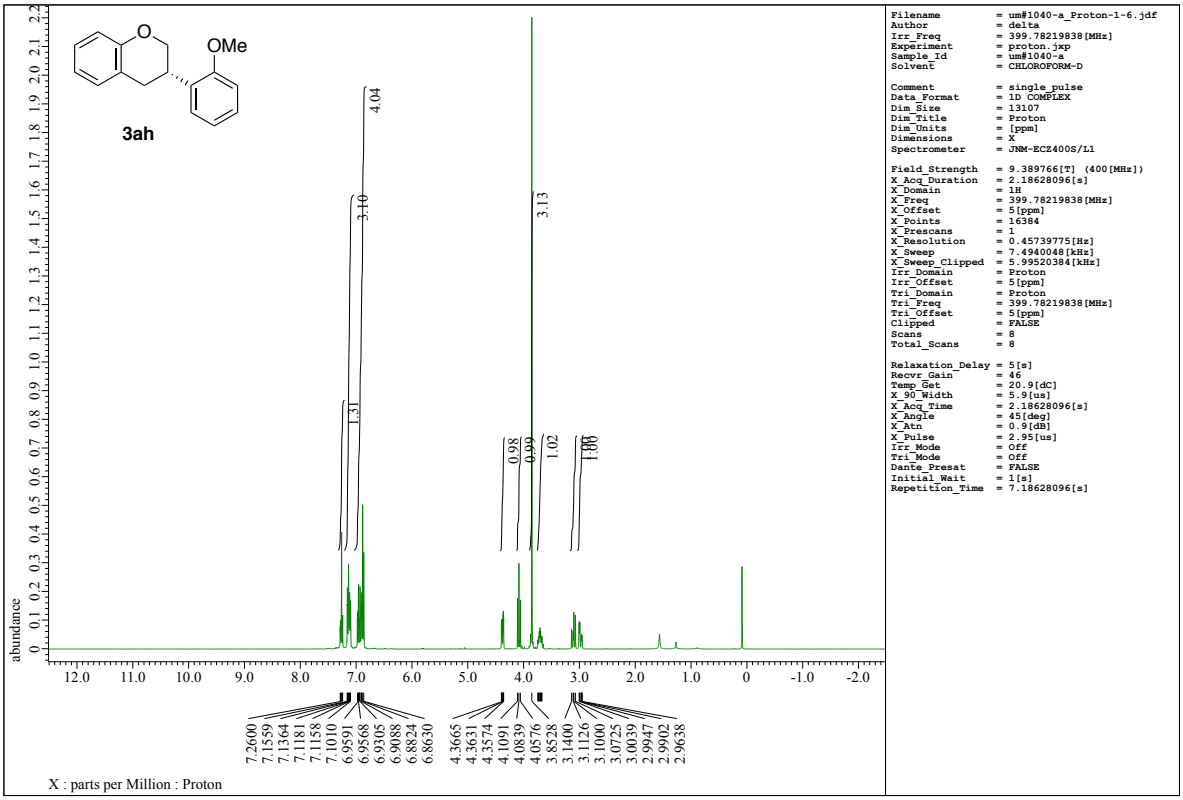
UV Results

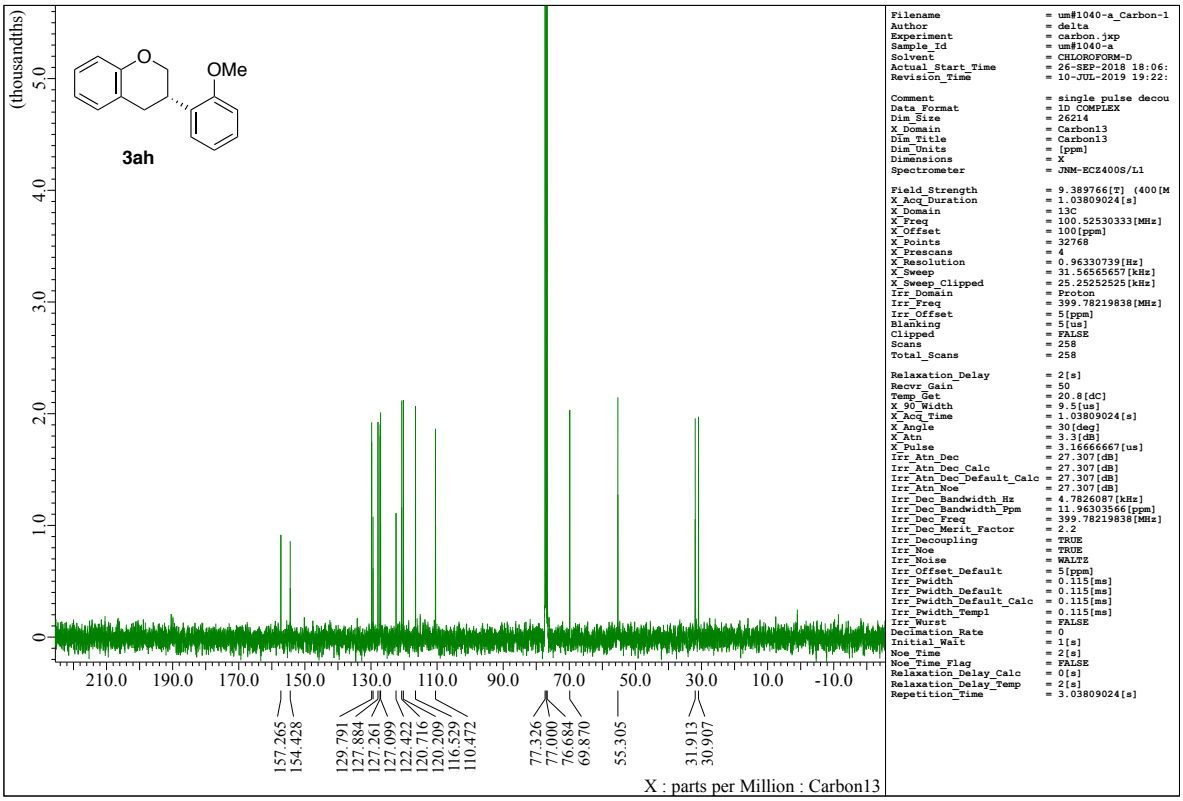
Pk #	Retention Time	Area	Area Percent	Height
1	24.817	9447422	97.661	252872
2	28.503	226295	2.339	6008
Totals		9673717	100.000	258880

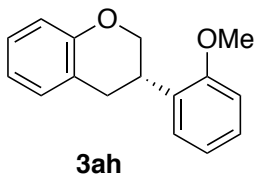


UV Results

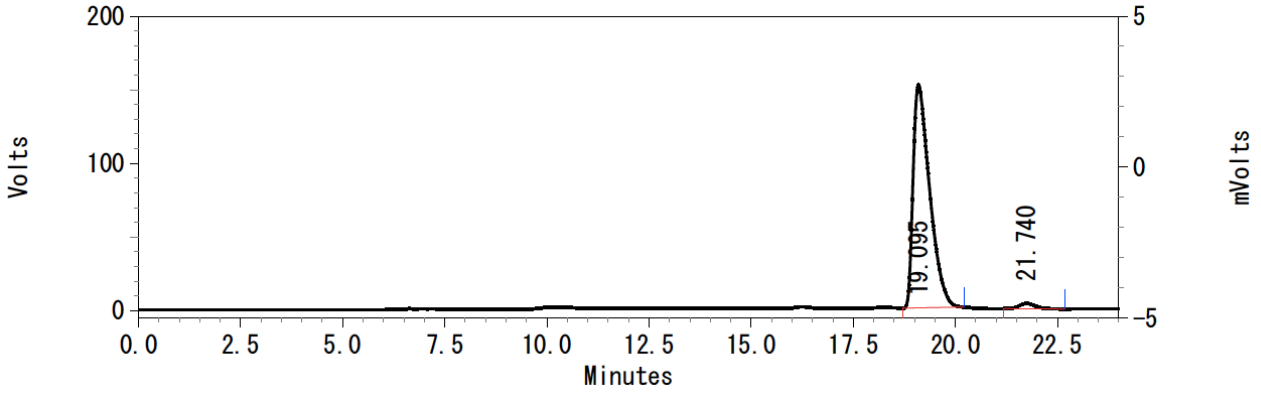
Pk #	Retention Time	Area	Area Percent	Height
1	25.158	8057865	50.246	215767
2	28.217	7979063	49.754	172355
Totals		16036928	100.000	388122


O
OMe
3ah
4.04
3.10
3.13
1.31
0.98
0.99
1.02
1.00
1.00
abundance
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
7.2600 7.1559 7.1364 7.1181 7.1158 7.1010 6.9591 6.9568 6.9305 6.9088 6.8824 6.8630
4.3665 4.3631 4.3574 4.1091 4.0839 4.0576 3.8528 3.1400 3.1126 3.1000 3.0725 3.0039 2.9947 2.9902 2.9638
X : parts per Million : Proton
Filename = um#1040-a_Proton-1-6.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = um#1040-a
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
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 = 46
Temp_Get = 20.9[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]


O
OMe
3ah
(thousandths)
5.0 4.0 3.0 2.0 1.0 0
210.0 190.0 170.0 150.0 130.0 110.0 90.0 70.0 50.0 30.0 10.0 -10.0
157.265 154.428 129.791 127.884 127.261 127.099 122.422 120.716 120.209 116.529 110.472
77.326 77.000 76.684 69.870 55.305 31.913 30.907
X : parts per Million : Carbon13
Filename = um#1040-a_Carbon-1
Author = delta
Experiment = carbon.jxp
Sample_Id = um#1040-a
Solvent = CHLOROFORM-D
Actual_Start_Time = 26-SEP-2018 18:06:
Revision_Time = 10-JUL-2019 19:22:
Comment = single_pulse decou
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon13
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[M
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Blanking = 5[us]
Clipped = FALSE
Scans = 258
Total_Scans = 258
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 20.8[dC]
X_90_Width = 9.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.3[dB]
X_Pulse = 3.16666667[us]
Irr_Atn_Dec = 27.307[dB]
Irr_Atn_Dec_Calc = 27.307[dB]
Irr_Atn_Dec_Default_Calc = 27.307[dB]
Irr_Atn_Noe = 27.307[dB]
Irr_Dec_Bandwidth_Hz = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm = 11.96303566[ppm]
Irr_Dec_Freq = 399.78219838[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noe = TRUE
Irr_Noise = WALTZ
Irr_Offset_Default = 5[ppm]
Irr_Pwidth = 0.115[ms]
Irr_Pwidth_Default = 0.115[ms]
Irr_Pwidth_Default_Calc = 0.115[ms]
Irr_Pwidth_Temp1 = 0.115[ms]
Irr_Wurst = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Noe_Time = 2[s]
Noe_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 2[s]
Repetition_Time = 3.03809024[s]

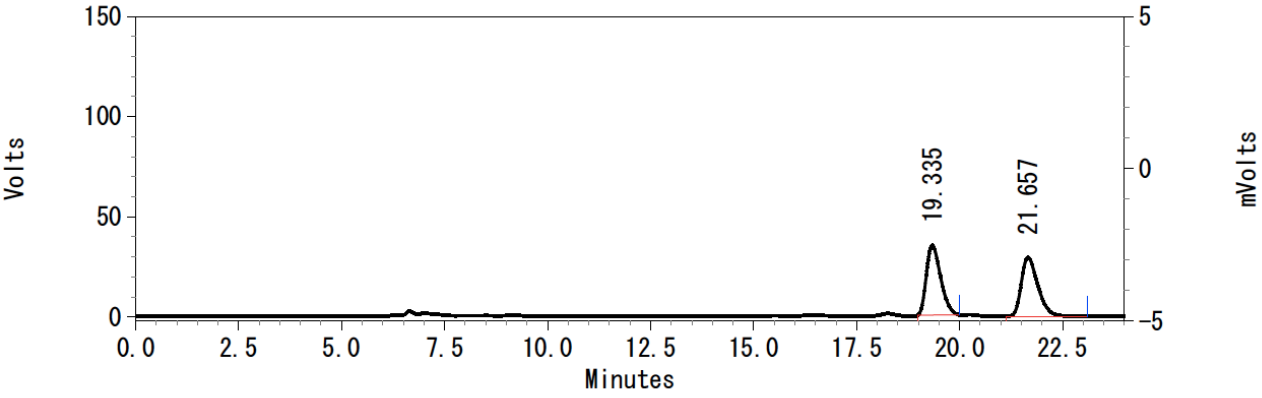


3ah



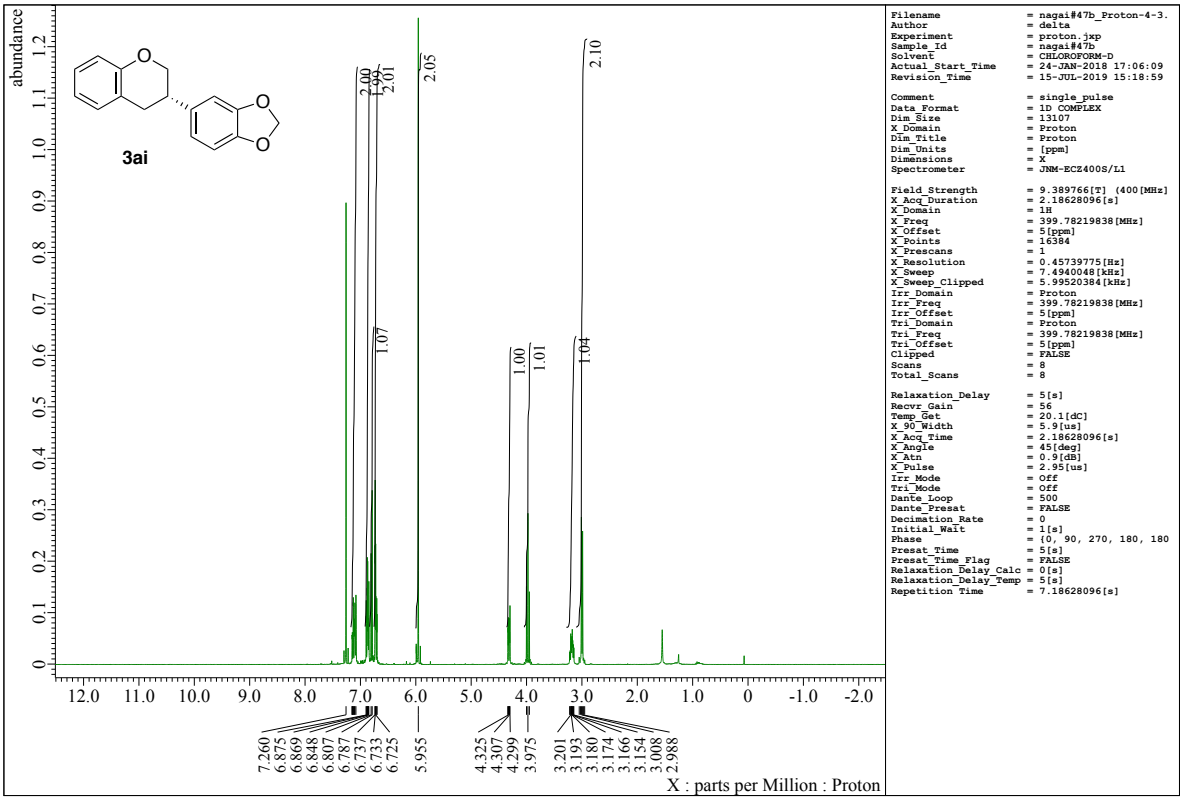
UV Results

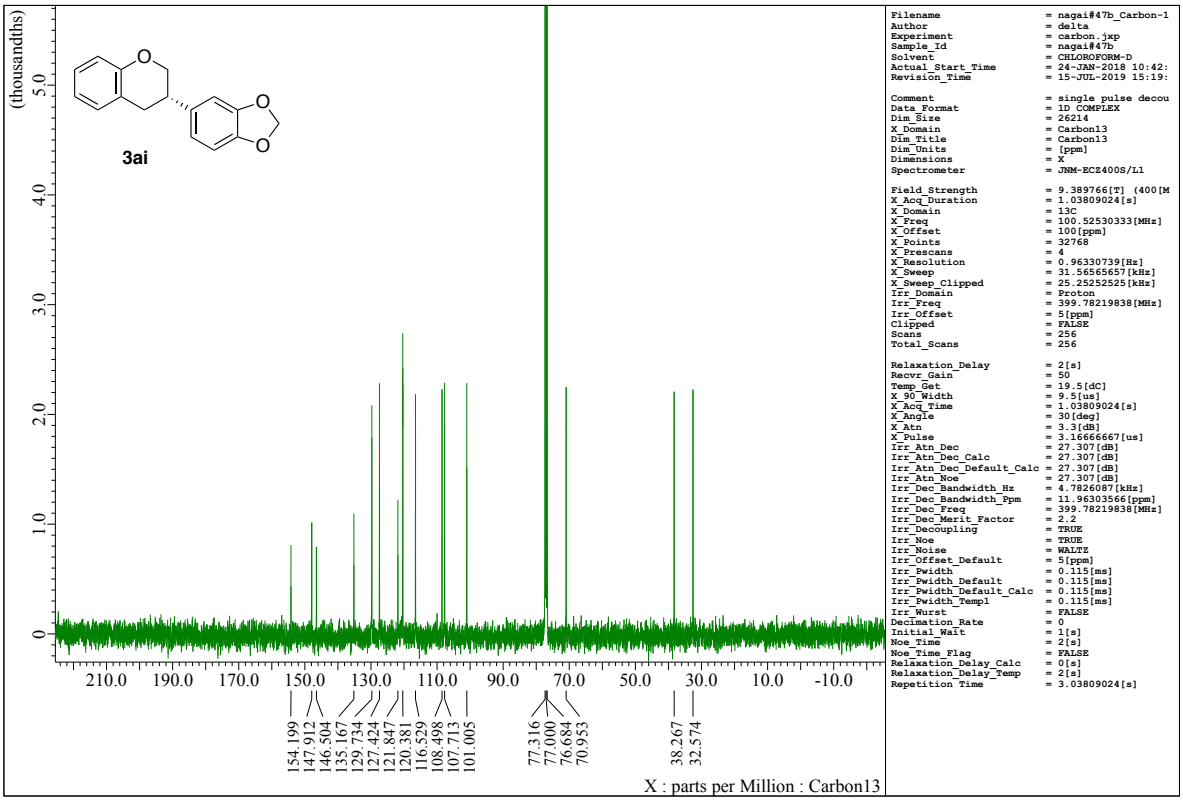
Pk #	Retention Time	Area	Area Percent	Height
1	19.095	4267293	97.700	151298
2	21.740	100465	2.300	3683
Totals		4367758	100.000	154981

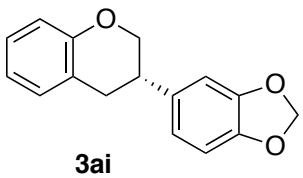


UV Results

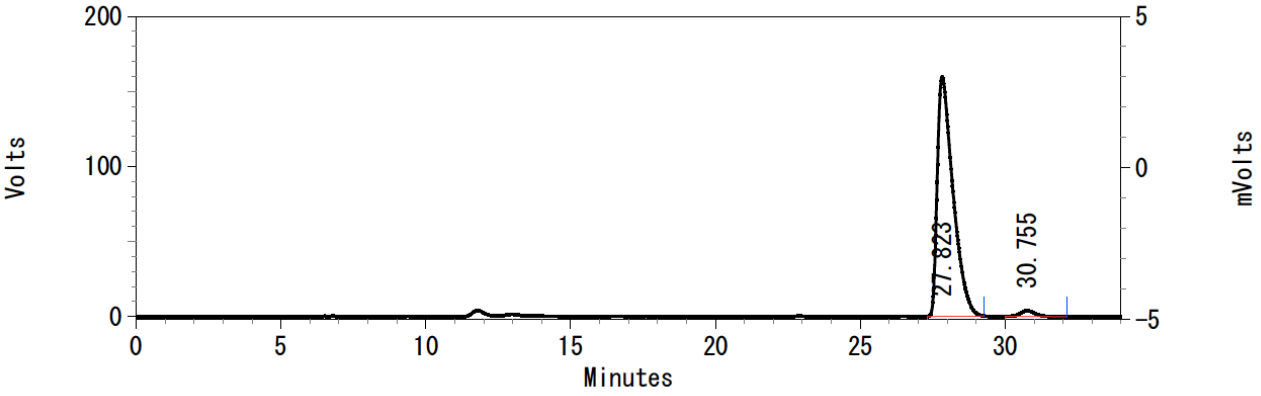
Pk #	Retention Time	Area	Area Percent	Height
1	19.335	835101	50.409	34813
2	21.657	821534	49.591	29431
Totals		1656635	100.000	64244


3ai
abundance
2.00
1.99
2.01
2.05
2.10
1.07
1.00
1.01
1.04
7.260
6.875
6.869
6.848
6.807
6.787
6.737
6.733
6.725
5.955
4.325
4.307
4.299
3.975
3.201
3.193
3.180
3.174
3.166
3.154
3.008
2.988
X : parts per Million : Proton
Filename = nagai#47b_Proton-4-3.
Author = delta
Experiment = proton.jxp
Sample_Id = nagai#47b
Solvent = CHLOROFORM-D
Actual_Start_Time = 24-JAN-2018 17:06:09
Revision_Time = 15-JUL-2019 15:18:59
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz]
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.9952038[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 = 56
Temp_Get = 20.1[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Loop = 500
Dante_Preset = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Phase = {0, 90, 270, 180, 180
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]


3ai
(thousandths)
154.199
147.912
146.504
135.167
129.734
127.424
121.847
120.381
116.529
108.498
107.713
101.005
77.316
77.000
76.684
70.953
38.267
32.574
X : parts per Million : Carbon13
Filename = nagai#47b_Carbon-1
Author = delta
Experiment = carbon.jxp
Sample_Id = nagai#47b
Solvent = CHLOROFORM-D
Actual_Start_Time = 24-JAN-2018 10:42:
Revision_Time = 15-JUL-2019 15:19:
Comment = single pulse decou
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon13
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[M
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[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 = 19.5[dC]
X_90_Width = 9.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.3[dB]
X_Pulse = 3.16666667[us]
Irr_Atn_Dec = 27.307[dB]
Irr_Atn_Dec_Calc = 27.307[dB]
Irr_Atn_Dec_Default_Calc = 27.307[dB]
Irr_Atn_Noe = 27.307[dB]
Irr_Dec_Bandwidth_Hz = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm = 11.96303566[ppm]
Irr_Dec_Freq = 399.78219838[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noe = TRUE
Irr_Noise = WALTZ
Irr_Offset_Default = 5[ppm]
Irr_Pwidth = 0.115[ms]
Irr_Pwidth_Default = 0.115[ms]
Irr_Pwidth_Default_Calc = 0.115[ms]
Irr_Pwidth_Templ = 0.115[ms]
Irr_Wurst = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Noe_Time = 2[s]
Noe_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 2[s]
Repetition_Time = 3.03809024[s]

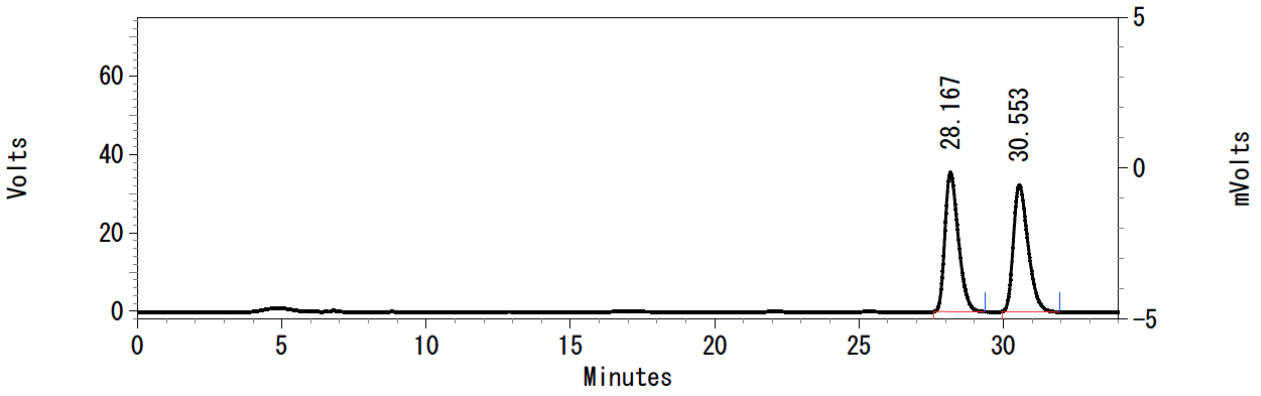


3ai



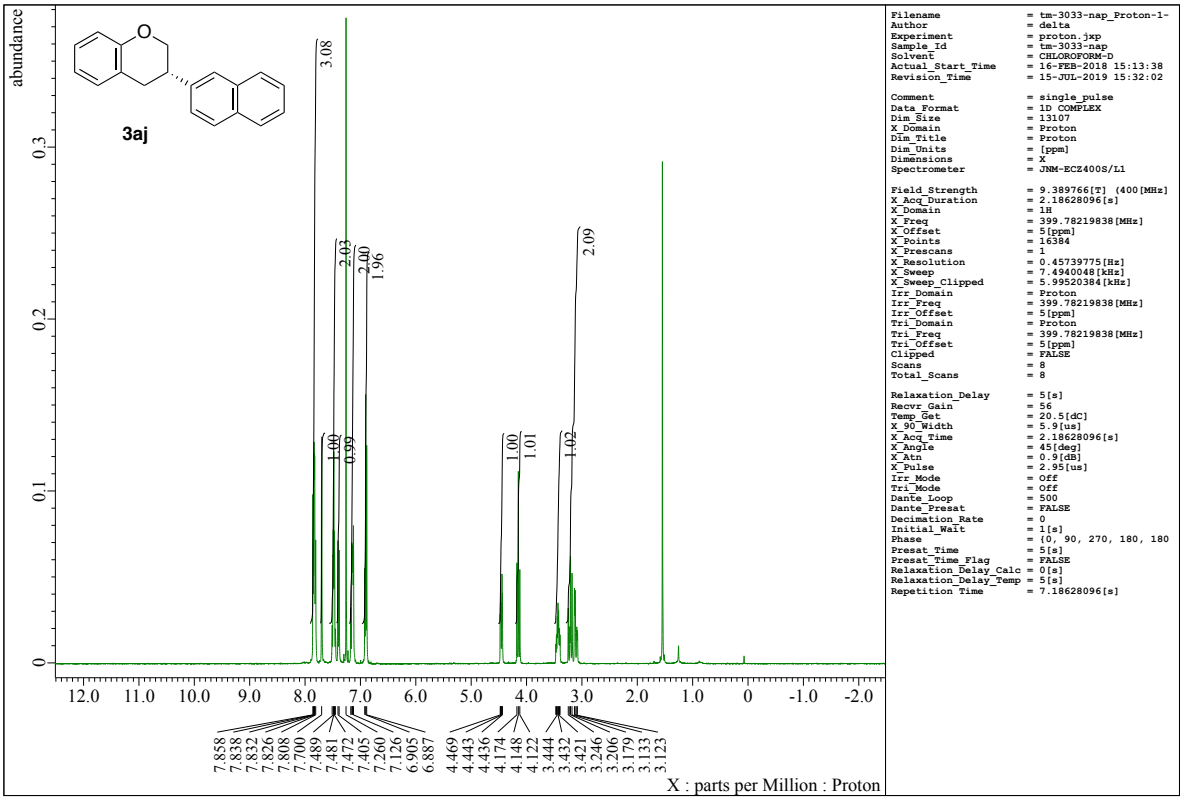
UV-970 Results

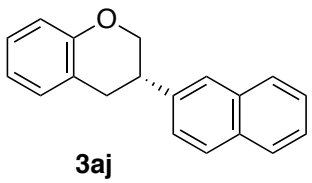
Pk #	Retention Time	Area	Area Percent	Height
1	27.823	6036061	97.807	160003
2	30.755	135314	2.193	3776
Totals		6171375	100.000	163779



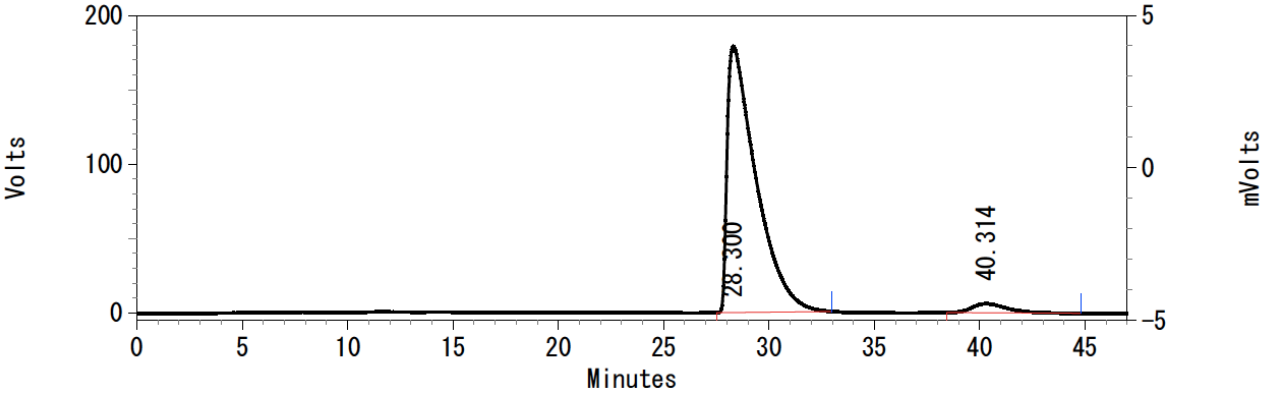
UV-970 Results

Pk #	Retention Time	Area	Area Percent	Height
1	28.167	1145715	50.045	35485
2	30.553	1143657	49.955	32333
Totals		2289372	100.000	67818


abundance
3aj
3.08
2.03
2.00
1.96
2.09
1.00
1.00
0.99
1.00
1.01
1.02
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
7.858 7.838 7.832 7.826 7.808 7.700 7.489 7.481 7.472 7.405 7.260 7.126 6.905 6.887
4.469 4.443 4.436 4.174 4.148 4.122 3.444 3.432 3.421 3.246 3.206 3.179 3.133 3.123
X : parts per Million : Proton
Filename = tm-3033-nap_Proton-1-
Author = delta
Experiment = proton.jxp
Sample_Id = tm-3033-nap
Solvent = CHLOROFORM-D
Actual_Start_Time = 16-FEB-2018 15:13:38
Revision_Time = 15-JUL-2019 15:32:02
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz]
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[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 = 56
Temp_Get = 20.5[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Loop = 500
Dante_Presat = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Phase = {0, 90, 270, 180, 180
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]

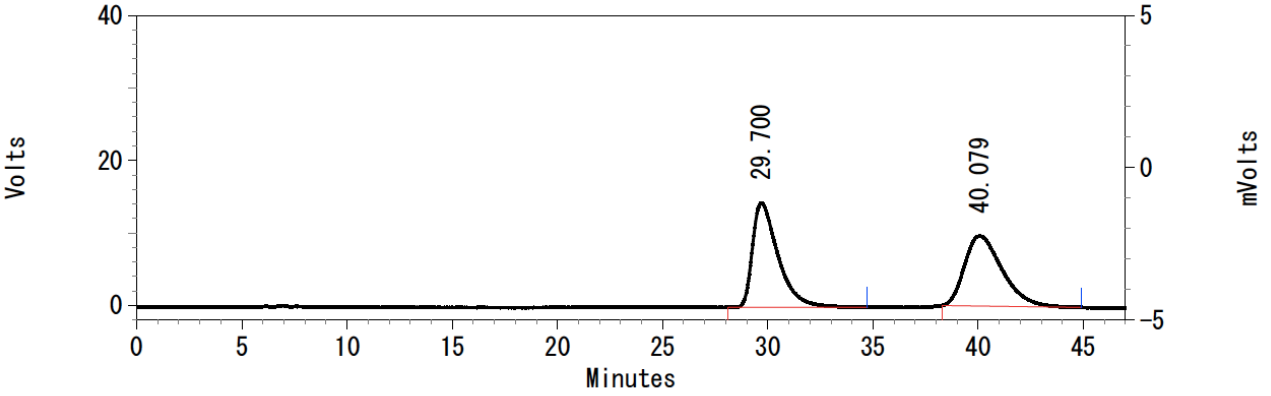


3aj



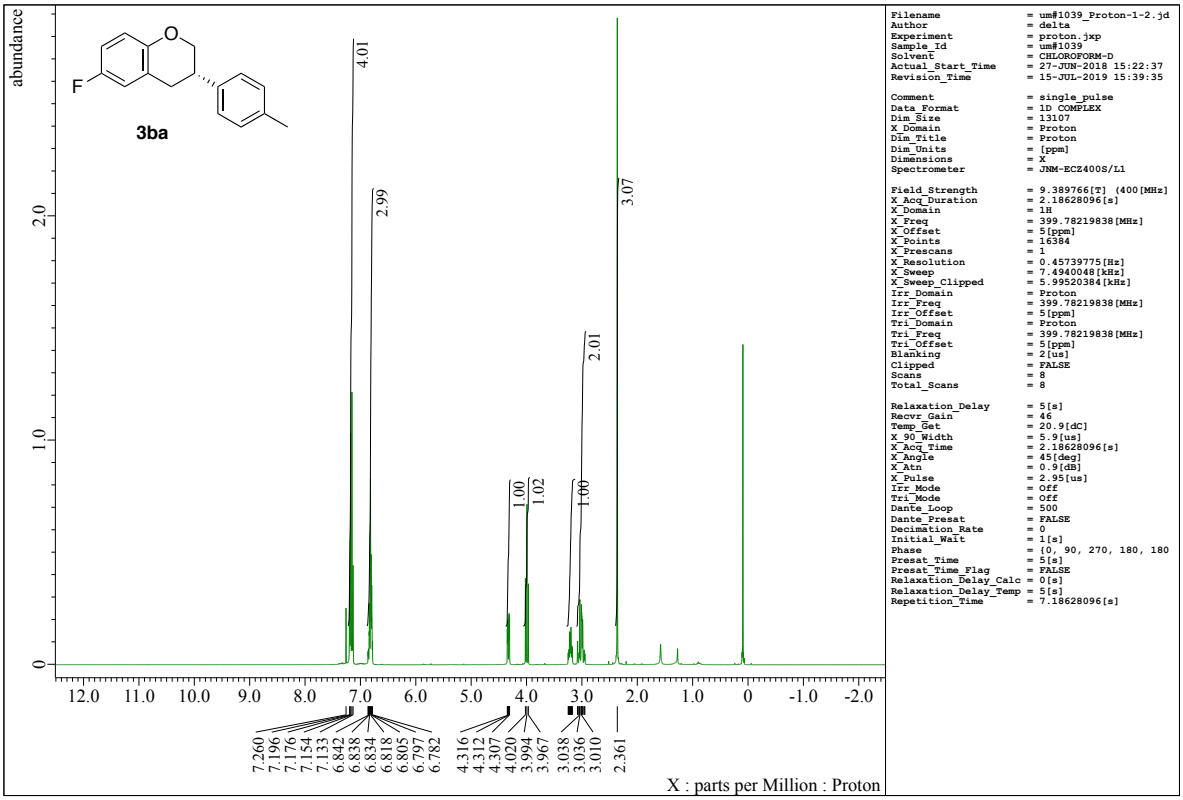
UV-970 Results

Pk #	Retention Time	Area	Area Percent	Height
1	28.300	17032748	96.092	178967
2	40.314	692794	3.908	6083
Totals		17725542	100.000	185050

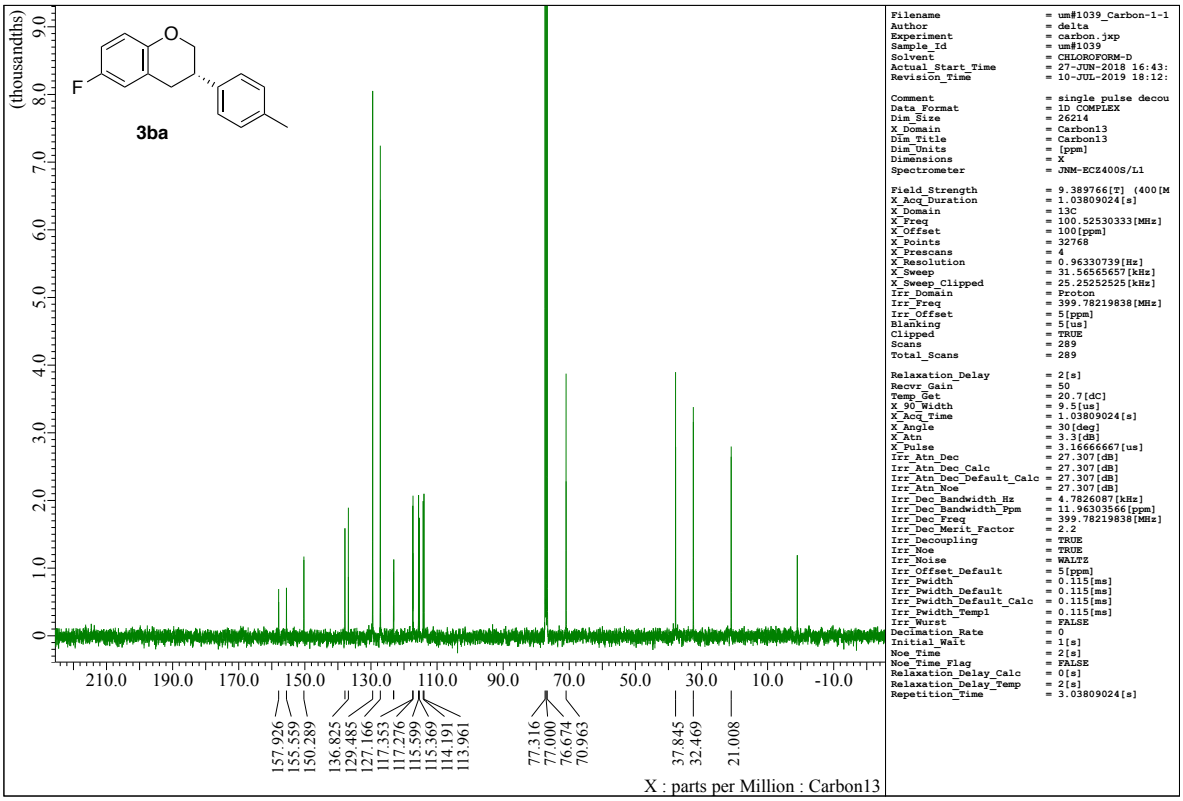


UV-970 Results

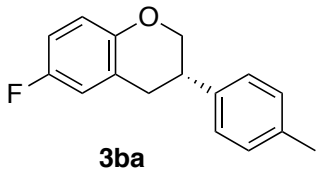
Pk #	Retention Time	Area	Area Percent	Height
1	29.700	1215561	50.259	14389
2	40.079	1203034	49.741	9656
Totals		2418595	100.000	24045



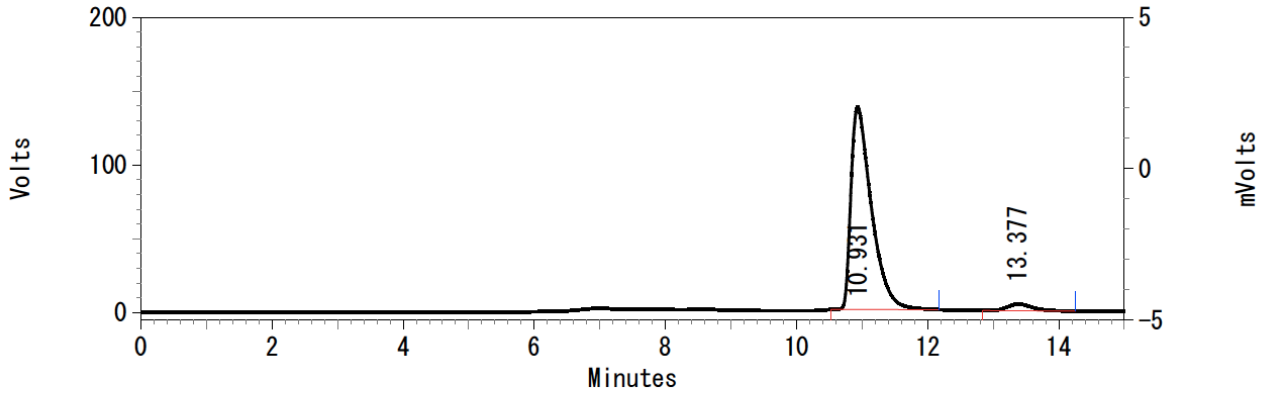

```
Filename                = um#1039_Proton-1-2.jd
Author                  = delta
Experiment              = proton.jxp
Sample_Id               = um#1039
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 27-JUN-2018 15:22:37
Revision_Time           = 15-JUL-2019 15:39:35

Comment                 = single_pulse
Data_Format             = 1D COMPLEX
Dim_Size                = 13107
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.389766[T] (400[MHz]
X_Acq_Duration          = 2.18628096[s]
X_Domain                = 1H
X_Freq                  = 399.78219838[MHz]
X_Offset                = 5[ppm]
X_Points                = 16384
X_Prescans              = 1
X_Resolution            = 0.45739775[Hz]
X_Sweep                 = 7.4940048[kHz]
X_Sweep_Clipped         = 5.99520384[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]
Blanking                = 2[us]
Clipped                 = FALSE
Scans                   = 8
Total_Scans             = 8

Relaxation_Delay        = 5[s]
Recvr_Gain              = 46
Temp_Get                = 20.9[dC]
X_90_Width              = 5.9[us]
X_Acq_Time              = 2.18628096[s]
X_Angle                 = 45[deg]
X_Atn                   = 0.9[dB]
X_Pulse                 = 2.95[us]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 500
Dante_Presat            = FALSE
Decimation_Rate         = 0
Initial_Wait            = 1[s]
Phase                   = {0, 90, 270, 180, 180
Presat_Time             = 5[s]
Presat_Time_Flag        = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 5[s]
Repetition_Time         = 7.18628096[s]
```




```
Filename                = um#1039_Carbon-1-1
Author                  = delta
Experiment              = carbon.jxp
Sample_Id               = um#1039
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 27-JUN-2018 16:43:
Revision_Time           = 10-JUL-2019 18:12:

Comment                 = single pulse decou
Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Carbon13
Dim_Title               = Carbon13
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.389766[T] (400[M
X_Acq_Duration          = 1.03809024[s]
X_Domain                = 13C
X_Freq                  = 100.52530333[MHz]
X_Offset                = 100[ppm]
X_Points                = 32768
X_Prescans              = 4
X_Resolution            = 0.96330739[Hz]
X_Sweep                 = 31.56565657[kHz]
X_Sweep_Clipped         = 25.25252525[kHz]
Irr_Domain              = Proton
Irr_Freq                = 399.78219838[MHz]
Irr_Offset              = 5[ppm]
Blanking                = 5[us]
Clipped                 = TRUE
Scans                   = 289
Total_Scans             = 289

Relaxation_Delay        = 2[s]
Recvr_Gain              = 50
Temp_Get                = 20.7[dC]
X_90_Width              = 9.5[us]
X_Acq_Time              = 1.03809024[s]
X_Angle                 = 30[deg]
X_Atn                   = 3.3[dB]
X_Pulse                 = 3.16666667[us]
Irr_Atn_Dec             = 27.307[dB]
Irr_Atn_Dec_Calc        = 27.307[dB]
Irr_Atn_Dec_Default_Calc = 27.307[dB]
Irr_Atn_Noe             = 27.307[dB]
Irr_Dec_Bandwidth_Hz    = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm   = 11.96303566[ppm]
Irr_Dec_Freq            = 399.78219838[MHz]
Irr_Dec_Merit_Factor    = 2.2
Irr_Decoupling          = TRUE
Irr_Noe                 = TRUE
Irr_Noise               = WALTZ
Irr_Offset_Default      = 5[ppm]
Irr_Pwidth              = 0.115[ms]
Irr_Pwidth_Default      = 0.115[ms]
Irr_Pwidth_Default_Calc = 0.115[ms]
Irr_Pwidth_Templ        = 0.115[ms]
Irr_Wurst               = FALSE
Decimation_Rate         = 0
Initial_Wait            = 1[s]
Noe_Time                = 2[s]
Noe_Time_Flag           = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 2[s]
Repetition_Time         = 3.03809024[s]
```

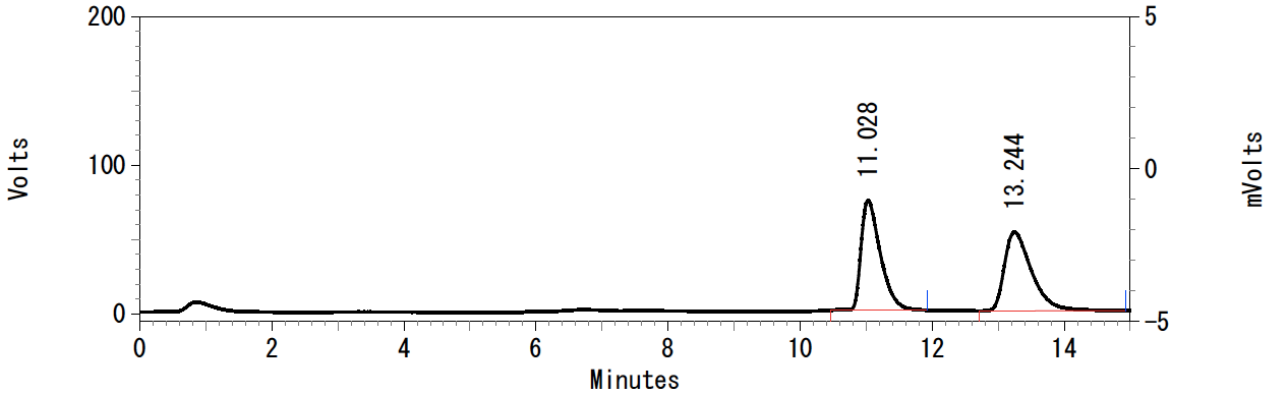


3ba



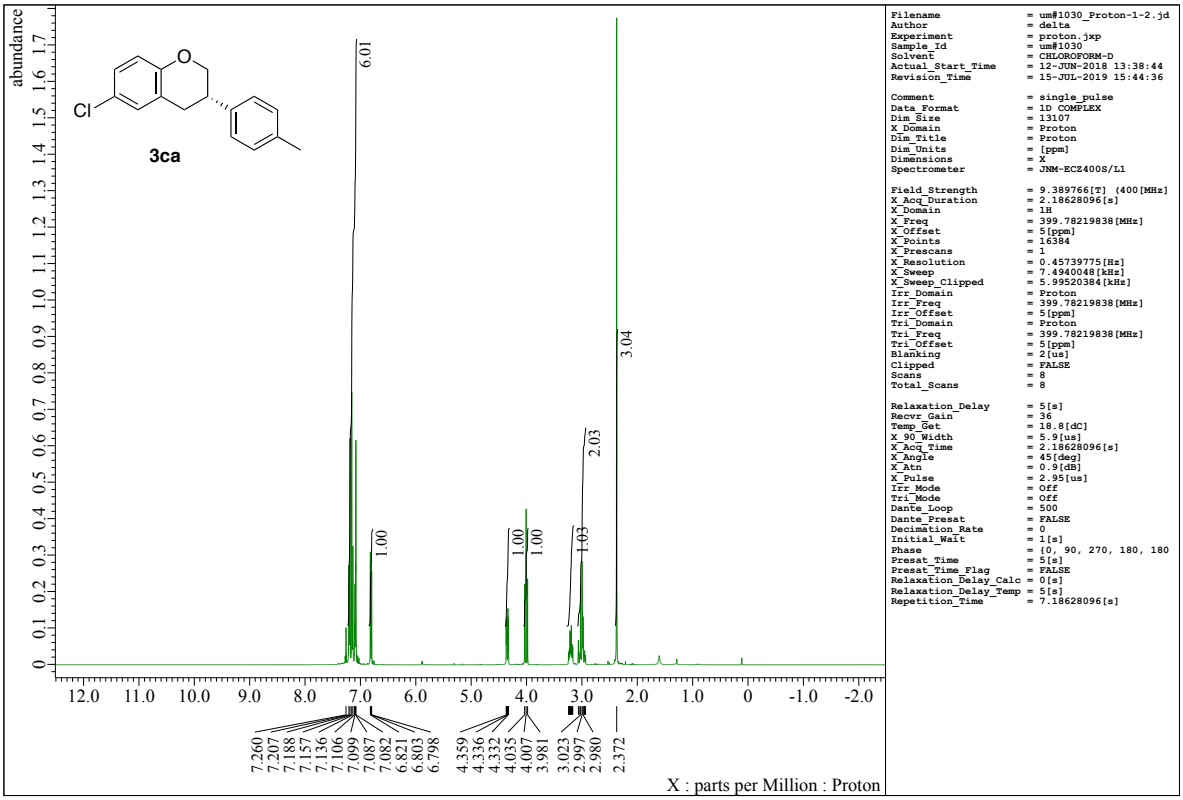
UV Results

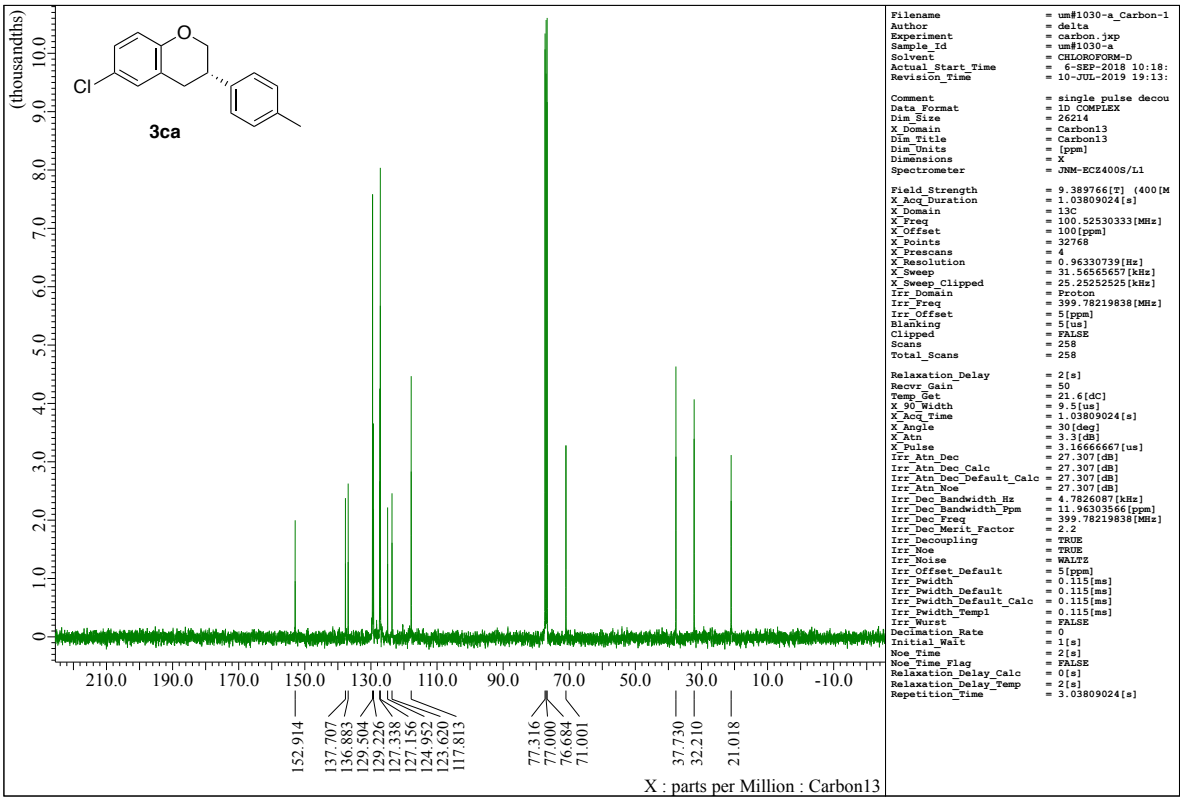
Pk #	Retention Time	Area	Area Percent	Height
1	10.931	2978296	96.185	137043
2	13.377	118144	3.815	4468
Totals		3096440	100.000	141511

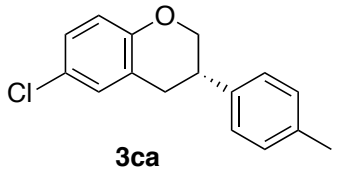


UV Results

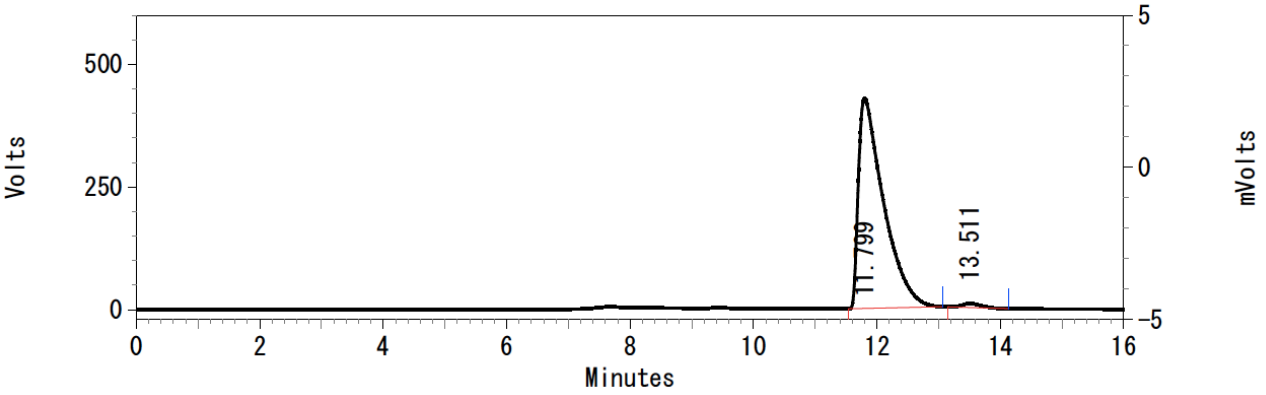
Pk #	Retention Time	Area	Area Percent	Height
1	11.028	1501068	49.704	73791
2	13.244	1518938	50.296	52986
Totals		3020006	100.000	126777


abundance
3ca
6.01
1.00
1.00
1.00
1.03
2.03
3.04
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
7.260 7.207 7.188 7.157 7.136 7.106 7.099 7.087 7.082 6.821 6.803 6.798
4.359 4.336 4.332 4.035 4.007 3.981
3.023 2.997 2.980
2.372
X : parts per Million : Proton
Filename = um#1030_Proton-1-2.jd
Author = delta
Experiment = proton.jxp
Sample_Id = um#1030
Solvent = CHLOROFORM-D
Actual_Start_Time = 12-JUN-2018 13:38:44
Revision_Time = 15-JUL-2019 15:44:36
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz]
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[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]
Blanking = 2[us]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 36
Temp_Get = 18.8[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Loop = 500
Dante_Presat = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Phase = {0, 90, 270, 180, 180
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]


(thousandths)
3ca
210.0 190.0 170.0 150.0 130.0 110.0 90.0 70.0 50.0 30.0 10.0 -10.0
152.914 137.707 136.883 129.504 129.226 127.338 127.156 124.952 123.620 117.813
77.316 77.000 76.684 71.001
37.730 32.210 21.018
X : parts per Million : Carbon13
Filename = um#1030-a_Carbon-1
Author = delta
Experiment = carbon.jxp
Sample_Id = um#1030-a
Solvent = CHLOROFORM-D
Actual_Start_Time = 6-SEP-2018 10:18:
Revision_Time = 10-JUL-2019 19:13:
Comment = single pulse decou
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon13
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[M
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Blanking = 5[us]
Clipped = FALSE
Scans = 258
Total_Scans = 258
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 21.6[dC]
X_90_Width = 9.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.3[dB]
X_Pulse = 3.16666667[us]
Irr_Atn_Dec = 27.307[dB]
Irr_Atn_Dec_Calc = 27.307[dB]
Irr_Atn_Dec_Default_Calc = 27.307[dB]
Irr_Atn_Noe = 27.307[dB]
Irr_Dec_Bandwidth_Hz = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm = 11.96303566[ppm]
Irr_Dec_Freq = 399.78219838[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noe = TRUE
Irr_Noise = WALTZ
Irr_Offset_Default = 5[ppm]
Irr_Pwidth = 0.115[ms]
Irr_Pwidth_Default = 0.115[ms]
Irr_Pwidth_Default_Calc = 0.115[ms]
Irr_Pwidth_Templ = 0.115[ms]
Irr_Wurst = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Noe_Time = 2[s]
Noe_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 2[s]
Repetition_Time = 3.03809024[s]

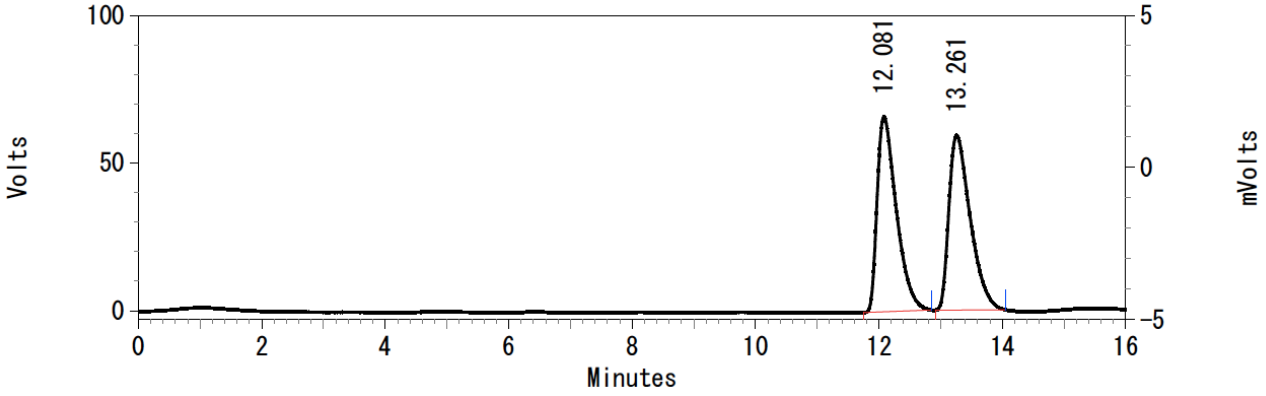


3ca



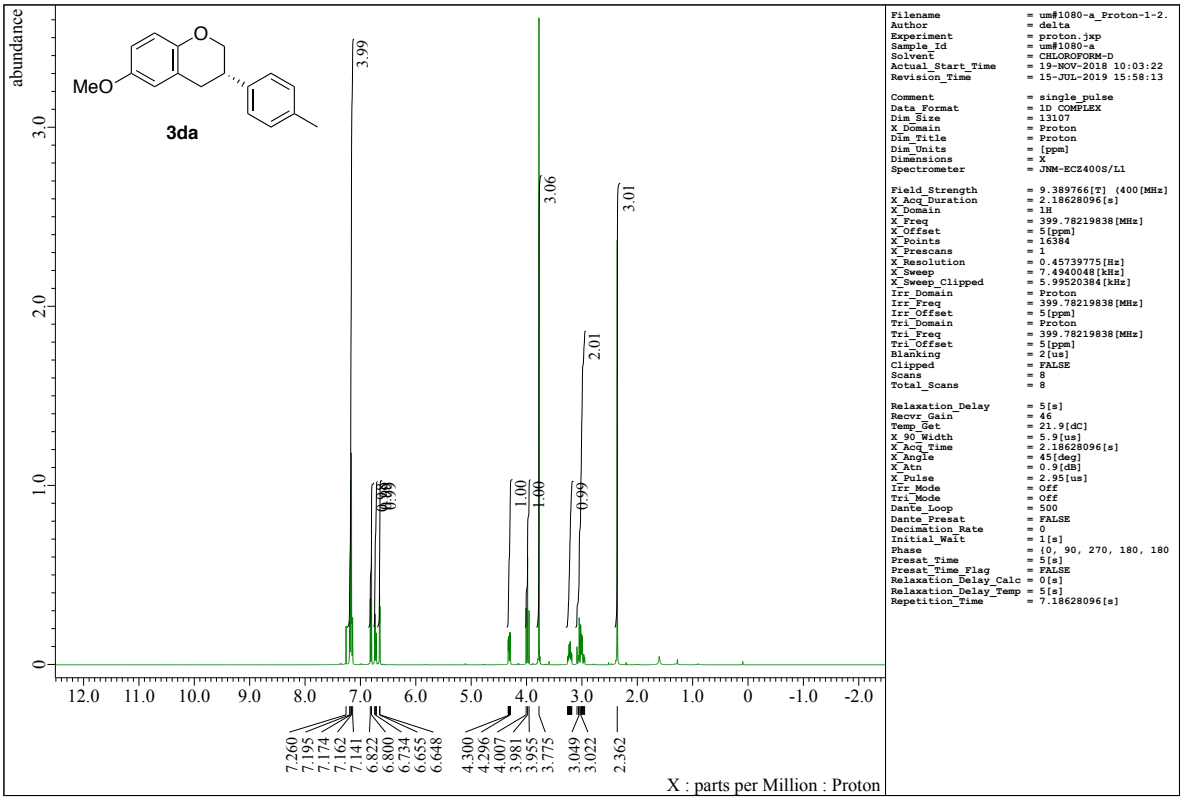
UV-970 Results

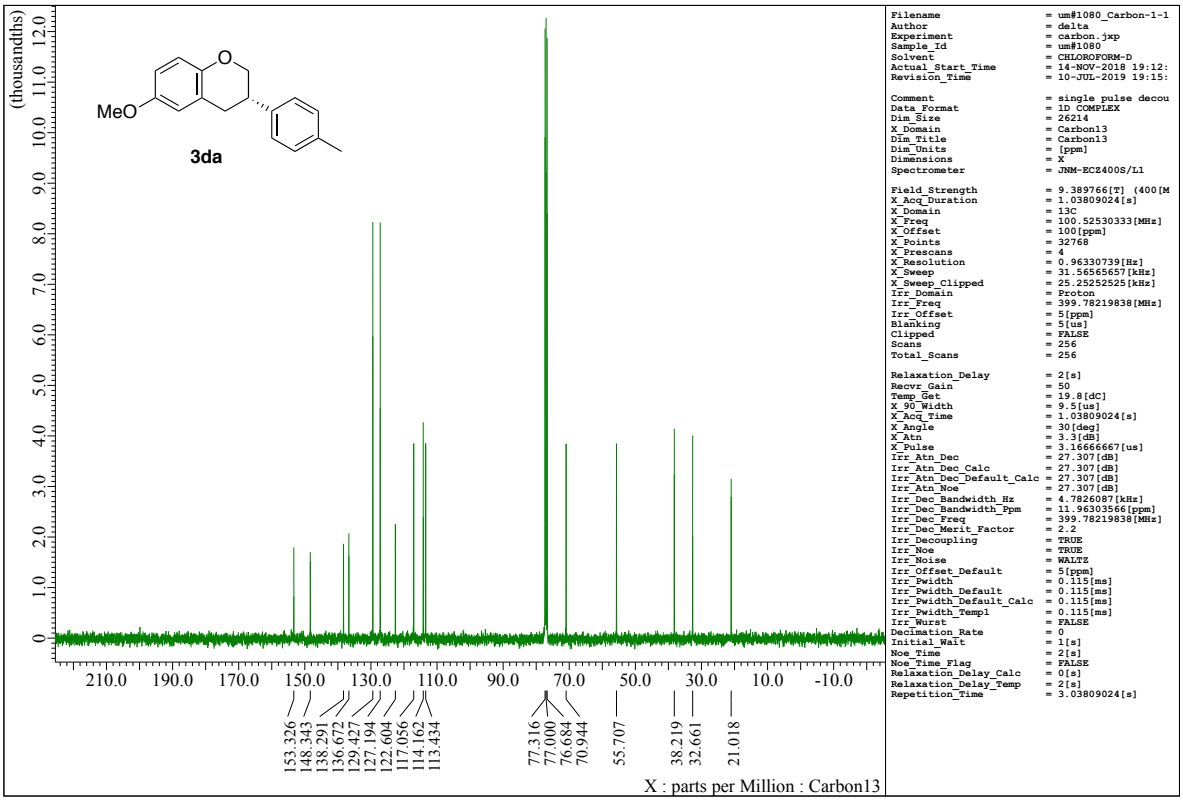
Pk #	Retention Time	Area	Area Percent	Height
1	11.799	12126319	98.612	428686
2	13.511	170682	1.388	8006
Totals		12297001	100.000	436692

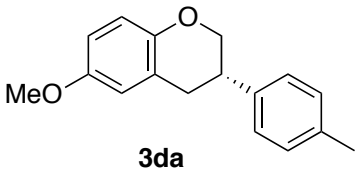


UV-970 Results

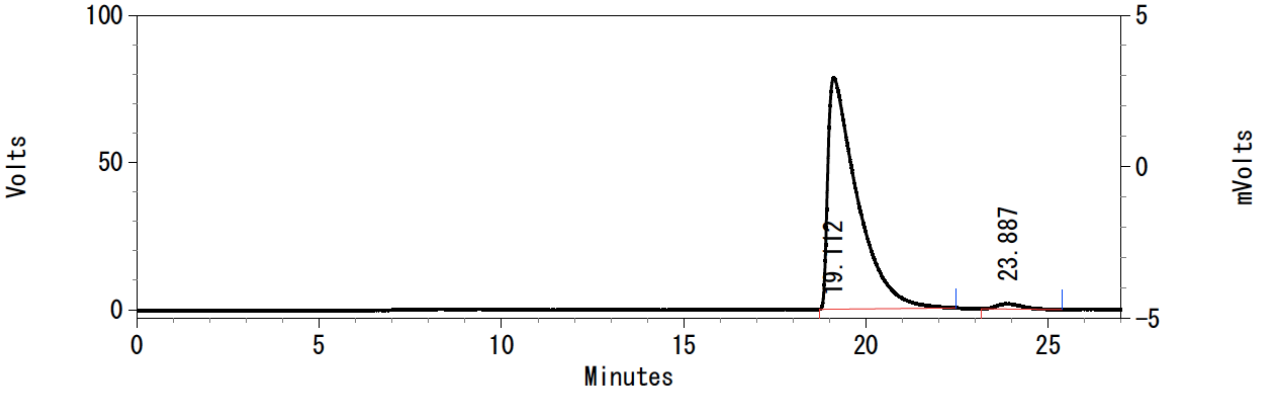
Pk #	Retention Time	Area	Area Percent	Height
1	12.081	1428445	50.002	66026
2	13.261	1428308	49.998	59213
Totals		2856753	100.000	125239


abundance
MeO
3da
3.99
3.06
3.01
2.01
1.00
1.00
0.99
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
7.260
7.195
7.174
7.162
7.141
6.822
6.800
6.734
6.655
6.648
4.300
4.296
4.007
3.981
3.955
3.775
3.049
3.022
2.362
X : parts per Million : Proton
Filename = um#1080-a_Proton-1-2.
Author = delta
Experiment = proton.jxp
Sample_Id = um#1080-a
Solvent = CHLOROFORM-D
Actual_Start_Time = 19-NOV-2018 10:03:22
Revision_Time = 15-JUL-2019 15:58:13
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz]
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[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]
Blanking = 2[us]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 21.9[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Loop = 500
Dante_Presat = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Phase = {0, 90, 270, 180, 180
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]


(thousandths)
MeO
3da
210.0 190.0 170.0 150.0 130.0 110.0 90.0 70.0 50.0 30.0 10.0 -10.0
153.326
148.343
138.291
136.672
129.427
127.194
122.604
117.056
114.162
113.434
77.316
77.000
76.684
70.944
55.707
38.219
32.661
21.018
X : parts per Million : Carbon13
Filename = um#1080_Carbon-1-1
Author = delta
Experiment = carbon.jxp
Sample_Id = um#1080
Solvent = CHLOROFORM-D
Actual_Start_Time = 14-NOV-2018 19:12:
Revision_Time = 10-JUL-2019 19:15:
Comment = single pulse decou
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon13
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[M
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Blanking = 5[us]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 19.8[dC]
X_90_Width = 9.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.3[dB]
X_Pulse = 3.16666667[us]
Irr_Atn_Dec = 27.307[dB]
Irr_Atn_Dec_Calc = 27.307[dB]
Irr_Atn_Dec_Default_Calc = 27.307[dB]
Irr_Atn_Noe = 27.307[dB]
Irr_Dec_Bandwidth_Hz = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm = 11.96303566[ppm]
Irr_Dec_Freq = 399.78219838[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noe = TRUE
Irr_Noise = WALTZ
Irr_Offset_Default = 5[ppm]
Irr_Pwidth = 0.115[ms]
Irr_Pwidth_Default = 0.115[ms]
Irr_Pwidth_Default_Calc = 0.115[ms]
Irr_Pwidth_Temp1 = 0.115[ms]
Irr_Wurst = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Noe_Time = 2[s]
Noe_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 2[s]
Repetition_Time = 3.03809024[s]

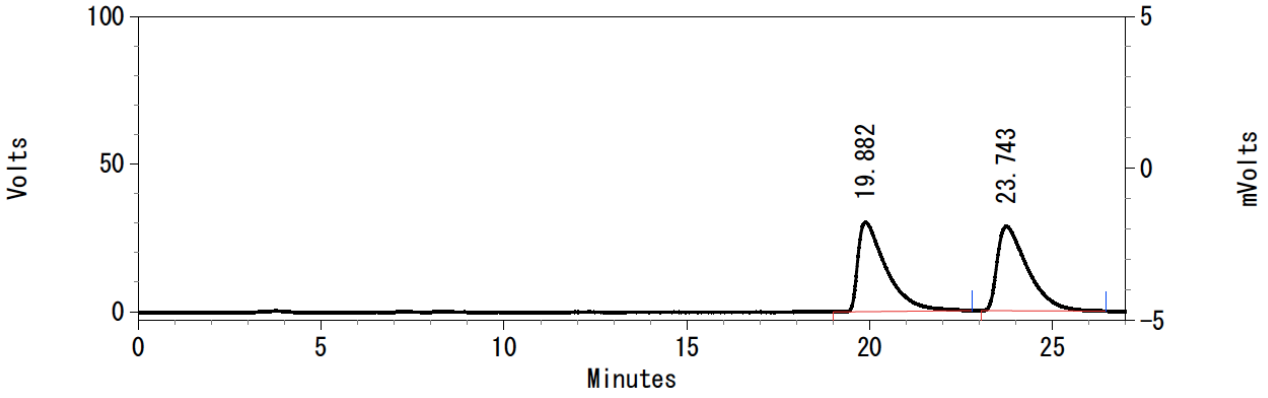


3da



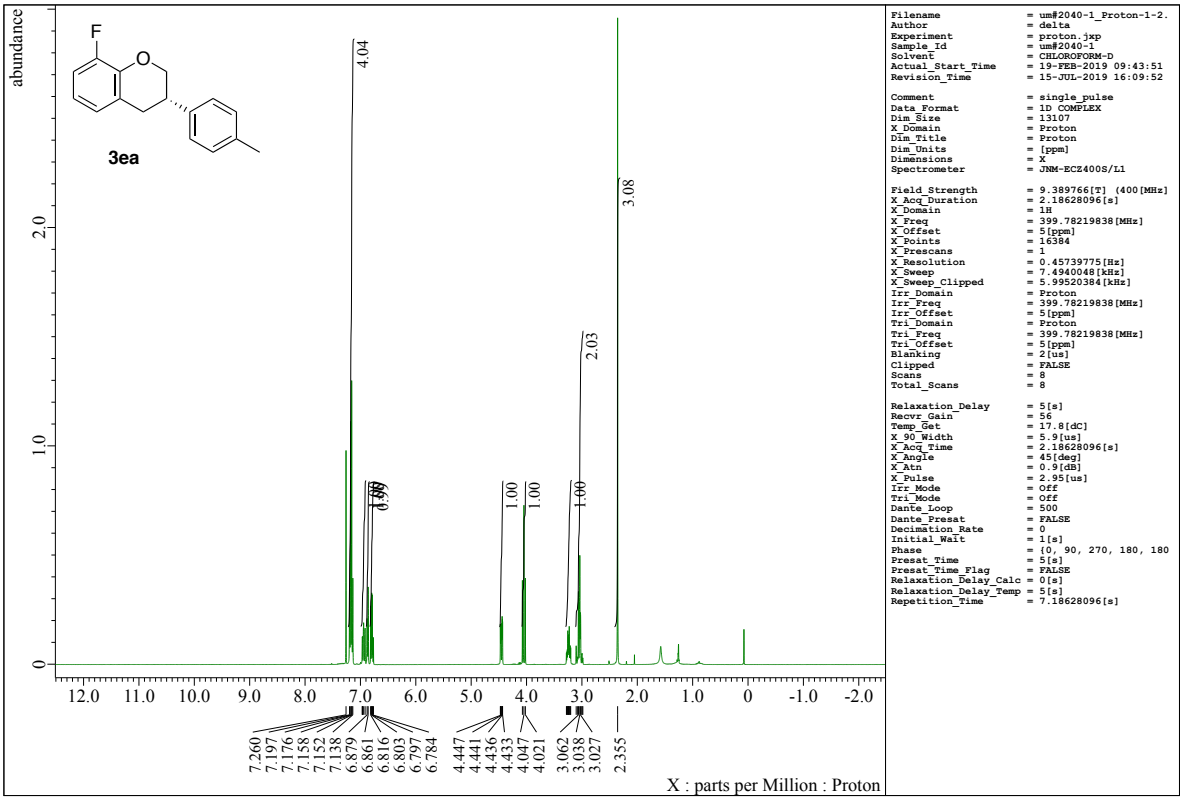
UV-970 Results

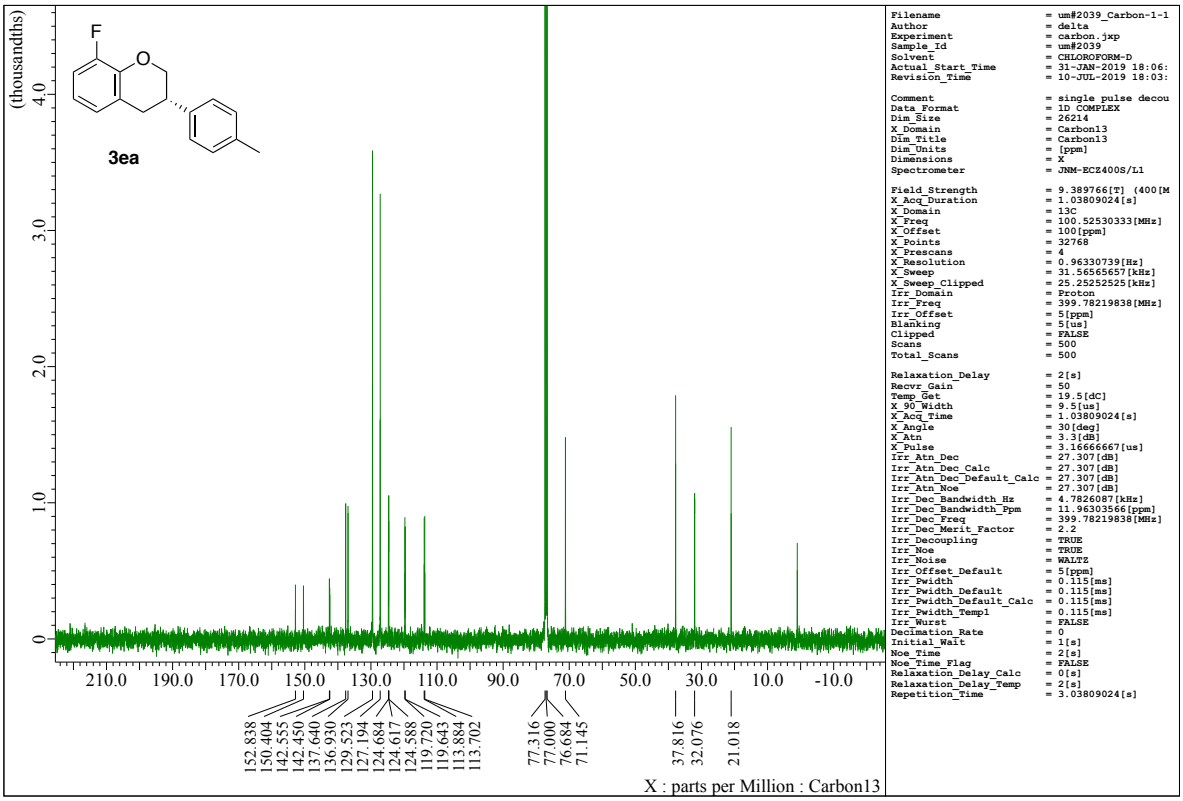
Pk #	Retention Time	Area	Area Percent	Height
1	19.112	4378780	98.179	78608
2	23.887	81208	1.821	1776
Totals		4459988	100.000	80384

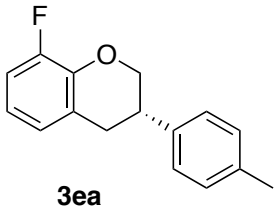


UV-970 Results

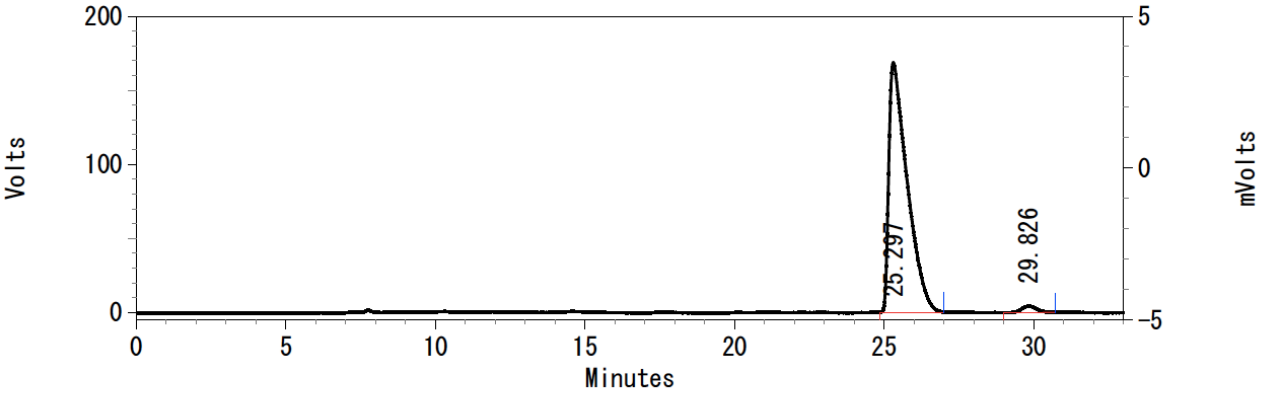
Pk #	Retention Time	Area	Area Percent	Height
1	19.882	1622209	49.673	30240
2	23.743	1643546	50.327	28618
Totals		3265755	100.000	58858


abundance
F
O
3ea
4.04
3.08
2.03
1.00
1.00
1.00
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
7.260
7.197
7.176
7.158
7.152
7.138
6.879
6.861
6.816
6.803
6.797
6.784
4.447
4.441
4.436
4.433
4.047
4.021
3.062
3.038
3.027
2.355
X : parts per Million : Proton
Filename = um#2040-1_Proton-1-2.
Author = delta
Experiment = proton.jxp
Sample_Id = um#2040-1
Solvent = CHLOROFORM-D
Actual_Start_Time = 19-FEB-2019 09:43:51
Revision_Time = 15-JUL-2019 16:09:52
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz]
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[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]
Blanking = 2[us]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 17.8[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Loop = 500
Dante_Presat = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Phase = (0, 90, 270, 180, 180
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]


(thousandths)
F
O
3ea
210.0 190.0 170.0 150.0 130.0 110.0 90.0 70.0 50.0 30.0 10.0 -10.0
152.838
150.404
142.555
142.450
137.640
136.930
129.523
127.194
124.684
124.617
124.588
119.720
119.643
113.884
113.702
77.316
77.000
76.684
71.145
37.816
32.076
21.018
X : parts per Million : Carbon13
Filename = um#2039_Carbon-1-1
Author = delta
Experiment = carbon.jxp
Sample_Id = um#2039
Solvent = CHLOROFORM-D
Actual_Start_Time = 31-JAN-2019 18:06:
Revision_Time = 10-JUL-2019 18:03:
Comment = single pulse decou
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon13
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[M
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Blanking = 5[us]
Clipped = FALSE
Scans = 500
Total_Scans = 500
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 19.5[dC]
X_90_Width = 9.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.3[dB]
X_Pulse = 3.16666667[us]
Irr_Atn_Dec = 27.307[dB]
Irr_Atn_Dec_Calc = 27.307[dB]
Irr_Atn_Dec_Default_Calc = 27.307[dB]
Irr_Atn_Noe = 27.307[dB]
Irr_Dec_Bandwidth_Hz = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm = 11.96303566[ppm]
Irr_Dec_Freq = 399.78219838[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noe = TRUE
Irr_Noise = WALTZ
Irr_Offset_Default = 5[ppm]
Irr_Pwidth = 0.115[ms]
Irr_Pwidth_Default = 0.115[ms]
Irr_Pwidth_Default_Calc = 0.115[ms]
Irr_Pwidth_Temp1 = 0.115[ms]
Irr_Wurst = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Noe_Time = 2[s]
Noe_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 2[s]
Repetition_Time = 3.03809024[s]

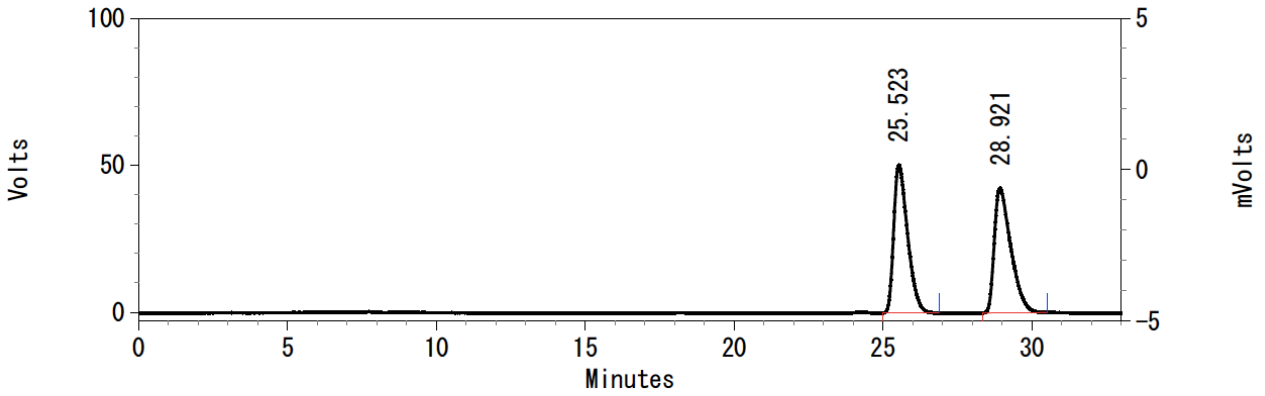


3ea



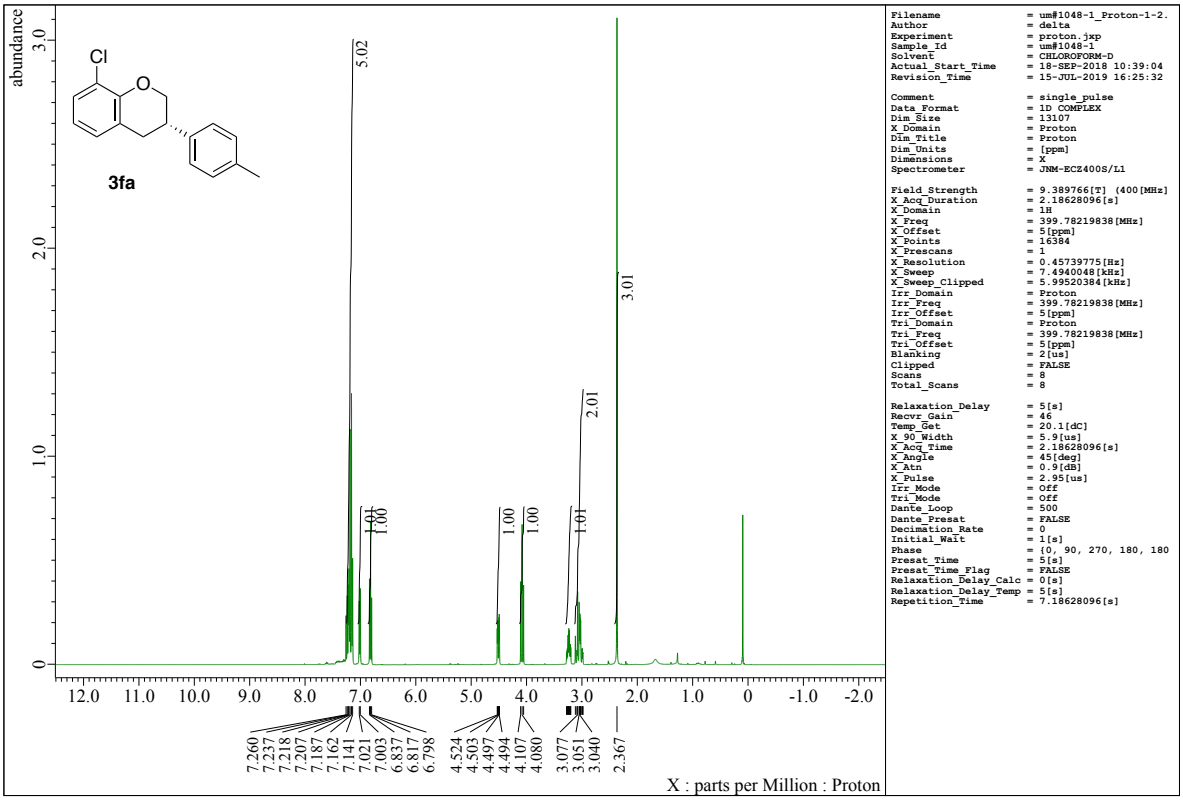
UV-970 Results

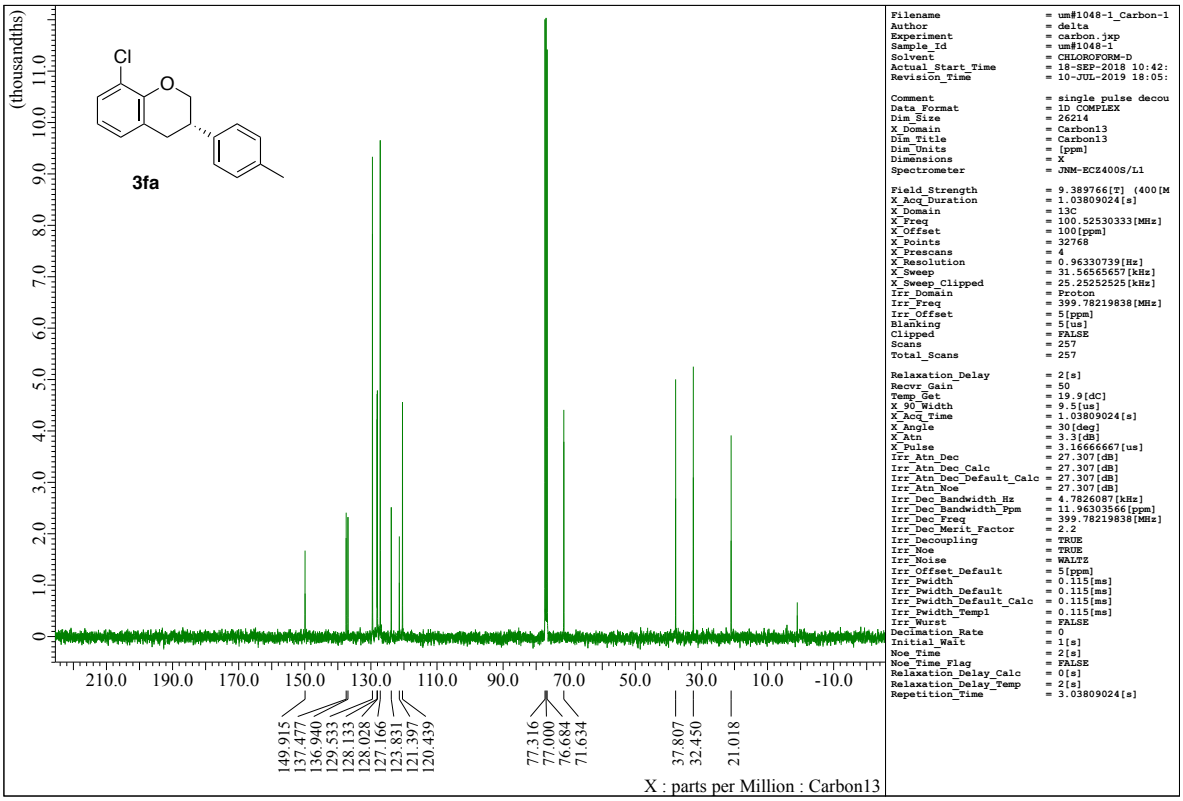
Pk #	Retention Time	Area	Area Percent	Height
1	25.297	7167956	97.937	168424
2	29.826	151027	2.063	4355
Totals		7318983	100.000	172779

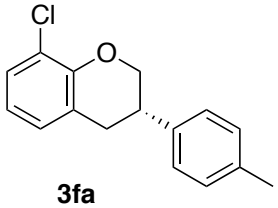


UV-970 Results

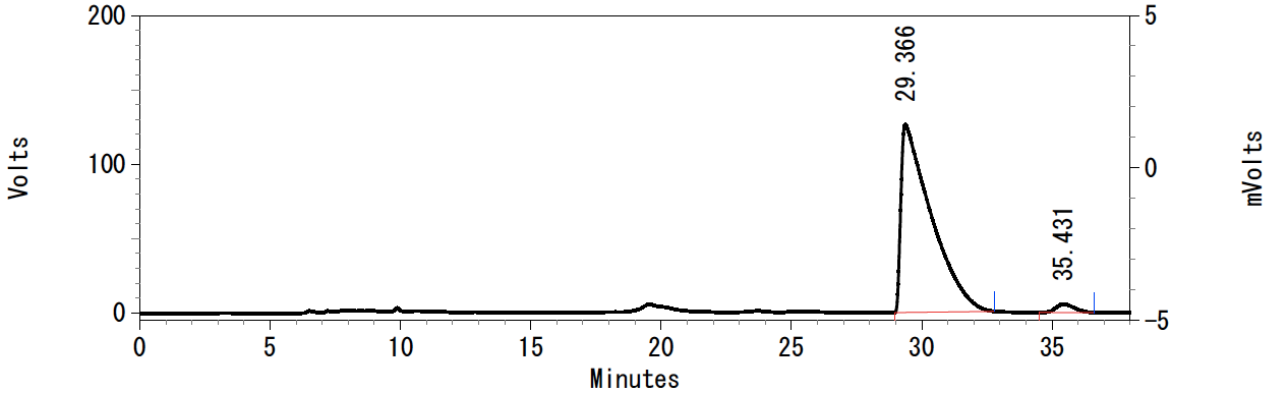
Pk #	Retention Time	Area	Area Percent	Height
1	25.523	1631474	49.943	50341
2	28.921	1635217	50.057	42419
Totals		3266691	100.000	92760


abundance
3fa
5.02
3.01
2.01
1.00
1.00
1.00
1.01
1.01
7.260
7.237
7.218
7.207
7.187
7.162
7.141
7.021
7.003
6.837
6.817
6.798
4.524
4.503
4.497
4.494
4.107
4.080
3.077
3.051
3.040
2.367
X : parts per Million : Proton
Filename = um#1048-1_Proton-1-2.
Author = delta
Experiment = proton.jxp
Sample_Id = um#1048-1
Solvent = CHLOROFORM-D
Actual_Start_Time = 18-SEP-2018 10:39:04
Revision_Time = 15-JUL-2019 16:25:32
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz]
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[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]
Blanking = 2[us]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 20.1[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Loop = 500
Dante_Presat = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Phase = {0, 90, 270, 180, 180
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]


(thousandths)
3fa
149.915
137.477
136.940
129.533
128.133
128.028
127.166
123.831
121.397
120.439
77.316
77.000
76.684
71.634
37.807
32.450
21.018
X : parts per Million : Carbon13
Filename = um#1048-1_Carbon-1
Author = delta
Experiment = carbon.jxp
Sample_Id = um#1048-1
Solvent = CHLOROFORM-D
Actual_Start_Time = 18-SEP-2018 10:42:
Revision_Time = 10-JUL-2019 18:05:
Comment = single pulse decou
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon13
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[M
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Blanking = 5[us]
Clipped = FALSE
Scans = 257
Total_Scans = 257
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 19.9[dC]
X_90_Width = 9.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.3[dB]
X_Pulse = 3.16666667[us]
Irr_Atn_Dec = 27.307[dB]
Irr_Atn_Dec_Calc = 27.307[dB]
Irr_Atn_Dec_Default_Calc = 27.307[dB]
Irr_Atn_Noe = 27.307[dB]
Irr_Dec_Bandwidth_Hz = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm = 11.96303566[ppm]
Irr_Dec_Freq = 399.78219838[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noe = TRUE
Irr_Noise = WALTZ
Irr_Offset_Default = 5[ppm]
Irr_Pwidth = 0.115[ms]
Irr_Pwidth_Default = 0.115[ms]
Irr_Pwidth_Default_Calc = 0.115[ms]
Irr_Pwidth_Templ = 0.115[ms]
Irr_Wurst = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Noe_Time = 2[s]
Noe_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 2[s]
Repetition_Time = 3.03809024[s]

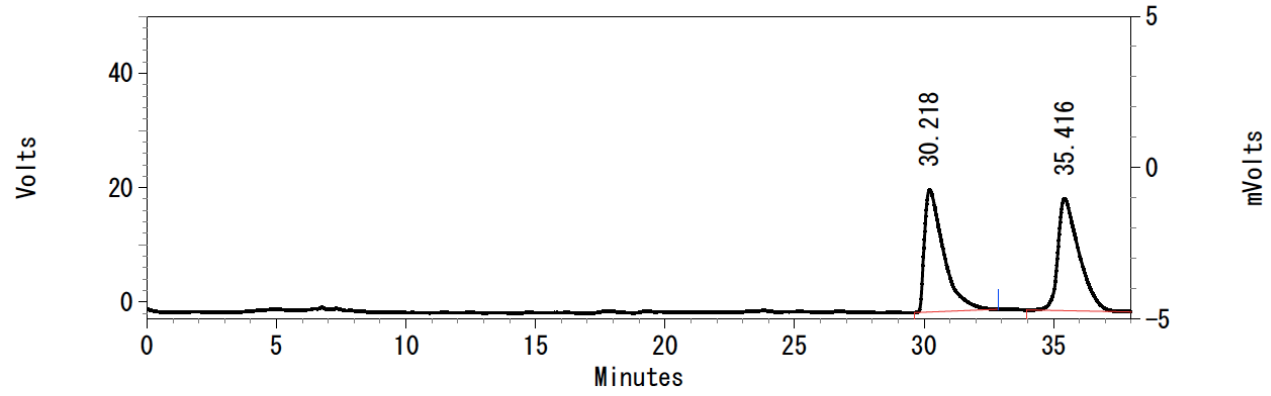


3fa



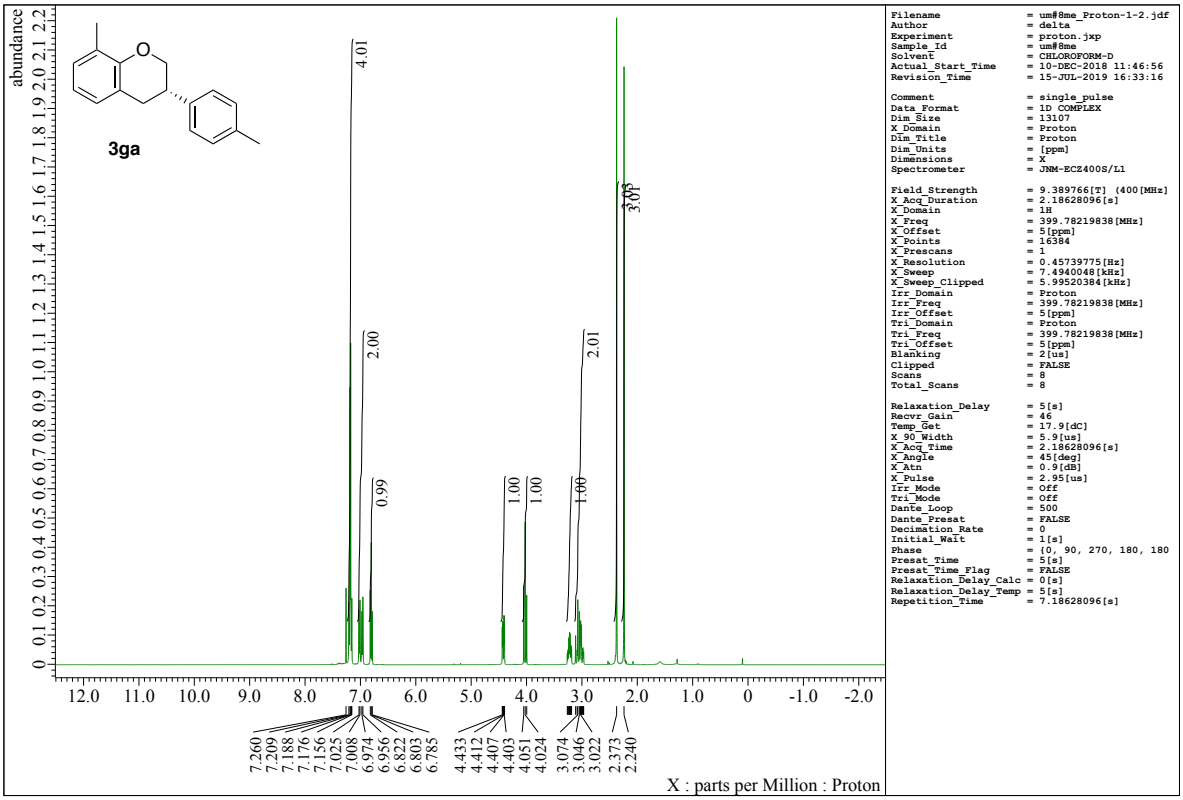
UV Results

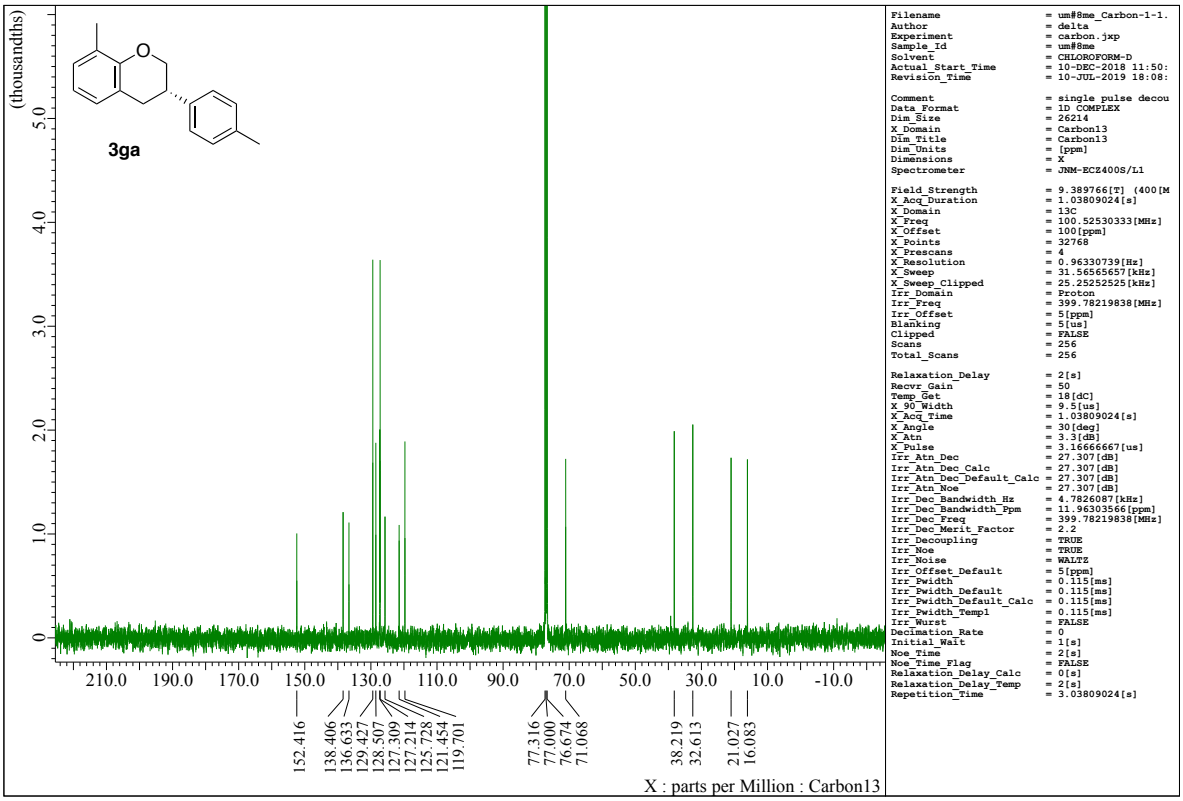
Pk #	Retention Time	Area	Area Percent	Height
1	29.366	10205703	97.339	126301
2	35.431	278967	2.661	5707
Totals		10484670	100.000	132008

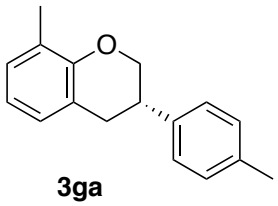


UV Results

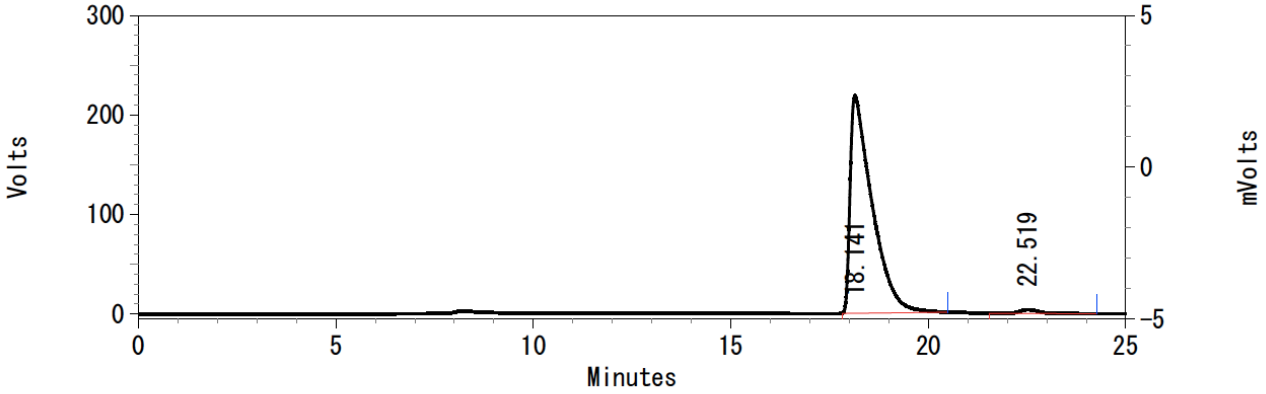
Pk #	Retention Time	Area	Area Percent	Height
1	30.218	1101724	49.772	21460
2	35.416	1111808	50.228	19602
Totals		2213532	100.000	41062


3ga
abundance
4.01
2.00
2.01
3.05
3.01
0.99
1.00
1.00
1.00
7.260
7.209
7.188
7.176
7.156
7.025
7.008
6.974
6.956
6.822
6.803
6.785
4.433
4.412
4.407
4.403
4.051
4.024
3.074
3.046
3.022
2.373
2.240
X : parts per Million : Proton
Filename = um#8me_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = um#8me
Solvent = CHLOROFORM-D
Actual_Start_Time = 10-DEC-2018 11:46:56
Revision_Time = 15-JUL-2019 16:33:16
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz]
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.9952038[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]
Blanking = 2[us]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 17.9[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Loop = 500
Dante_Presat = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Phase = {0, 90, 270, 180, 180
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]


3ga
(thousandths)
152.416
138.406
136.633
129.427
128.507
127.309
127.214
125.728
121.454
119.701
77.316
77.000
76.674
71.068
38.219
32.613
21.027
16.083
X : parts per Million : Carbon13
Filename = um#8me_Carbon-1-1.
Author = delta
Experiment = carbon.jxp
Sample_Id = um#8me
Solvent = CHLOROFORM-D
Actual_Start_Time = 10-DEC-2018 11:50:
Revision_Time = 10-JUL-2019 18:08:
Comment = single pulse decou
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon13
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[M
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Blanking = 5[us]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 18[dC]
X_90_Width = 9.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.3[dB]
X_Pulse = 3.16666667[us]
Irr_Atn_Dec = 27.307[dB]
Irr_Atn_Dec_Calc = 27.307[dB]
Irr_Atn_Dec_Default_Calc = 27.307[dB]
Irr_Atn_Noe = 27.307[dB]
Irr_Dec_Bandwidth_Hz = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm = 11.96303566[ppm]
Irr_Dec_Freq = 399.78219838[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noe = TRUE
Irr_Noise = WALTZ
Irr_Offset_Default = 5[ppm]
Irr_Pwidth = 0.115[ms]
Irr_Pwidth_Default = 0.115[ms]
Irr_Pwidth_Default_Calc = 0.115[ms]
Irr_Pwidth_Temp1 = 0.115[ms]
Irr_Wurst = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Noe_Time = 2[s]
Noe_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 2[s]
Repetition_Time = 3.03809024[s]

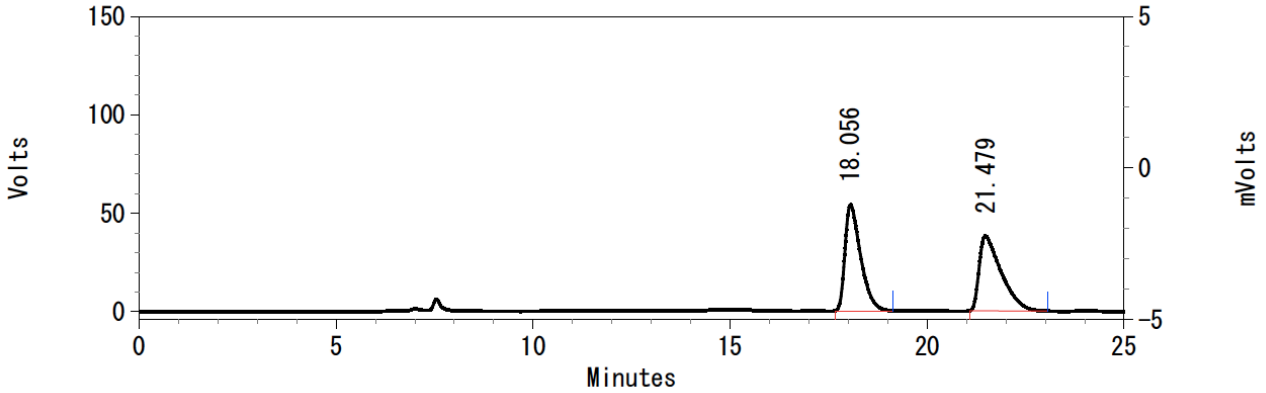


3ga



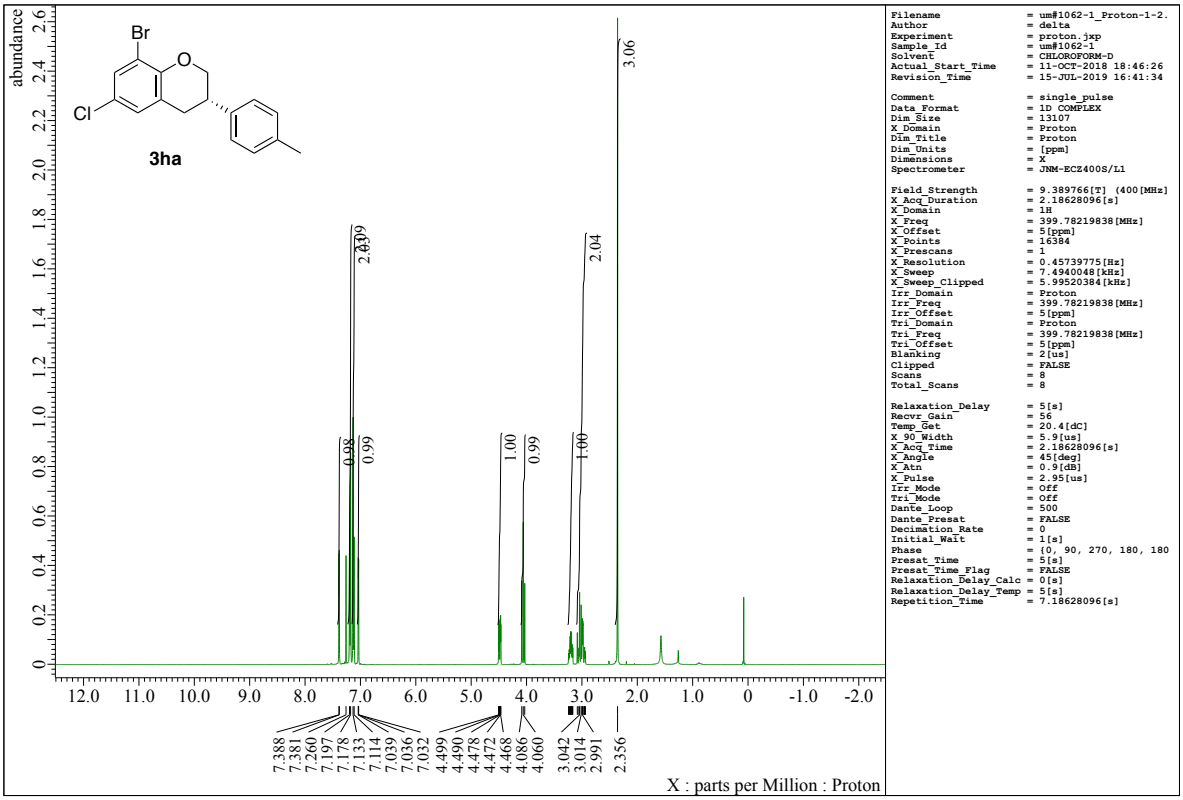
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	18.141	8439272	98.429	218499
2	22.519	134728	1.571	3950
Totals		8574000	100.000	222449

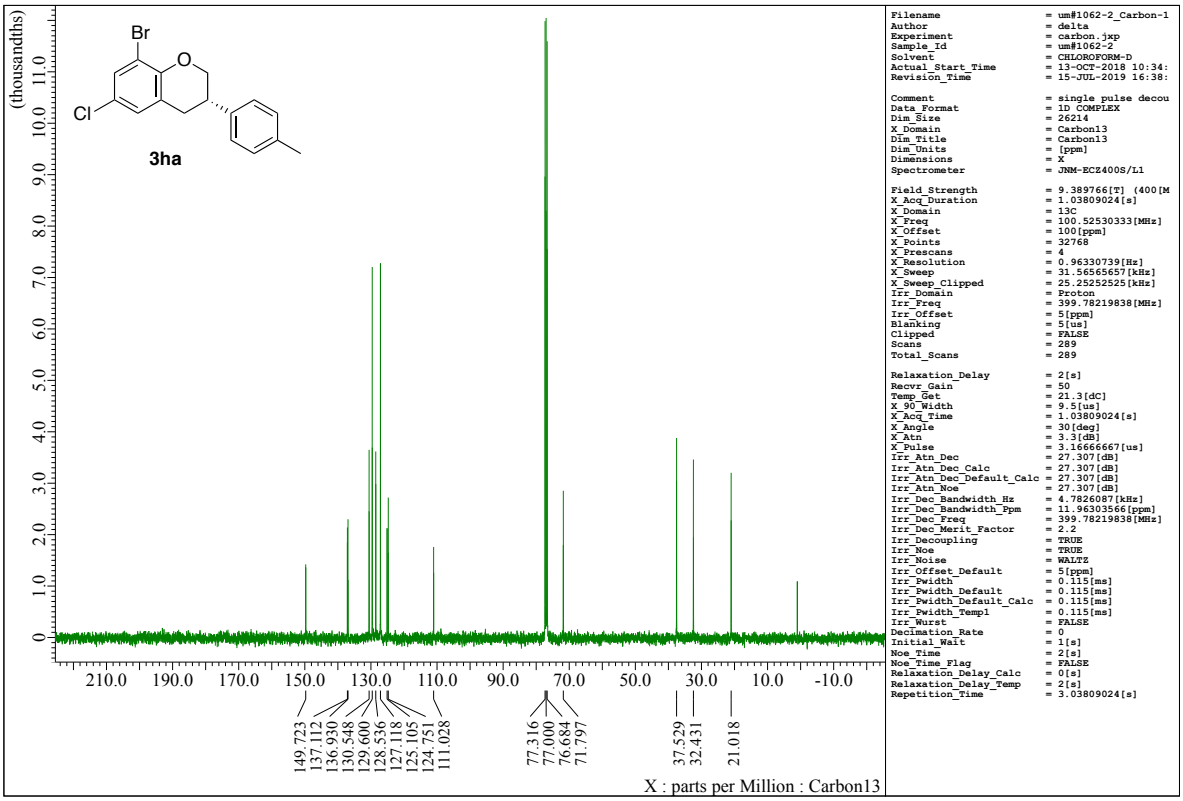


UV Results

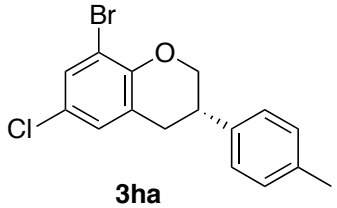
Pk #	Retention Time	Area	Area Percent	Height
1	18.056	1486736	50.198	53904
2	21.479	1475032	49.802	38028
Totals		2961768	100.000	91932



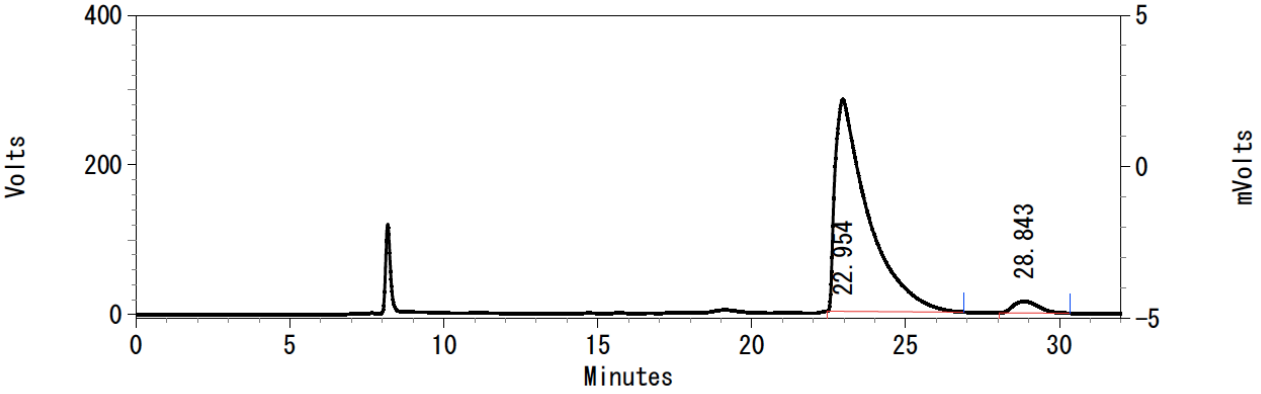

```
Filename                = um#1062-1_Proton-1-2.
Author                  = delta
Experiment              = proton.jxp
Sample_Id               = um#1062-1
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 11-OCT-2018 18:46:26
Revision_Time           = 15-JUL-2019 16:41:34

Comment                 = single_pulse
Data_Format             = 1D COMPLEX
Dim_Size                = 13107
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.389766[T] (400[MHz]
X_Acq_Duration          = 2.18628096[s]
X_Domain                = 1H
X_Freq                  = 399.78219838[MHz]
X_Offset                = 5[ppm]
X_Points                = 16384
X_Prescans              = 1
X_Resolution            = 0.45739775[Hz]
X_Sweep                 = 7.4940048[kHz]
X_Sweep_Clipped         = 5.99520384[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]
Blanking                = 2[us]
Clipped                 = FALSE
Scans                   = 8
Total_Scans             = 8

Relaxation_Delay        = 5[s]
Recvr_Gain              = 56
Temp_Get                = 20.4[dC]
X_90_Width              = 5.9[us]
X_Acq_Time              = 2.18628096[s]
X_Angle                 = 45[deg]
X_Atn                   = 0.9[dB]
X_Pulse                 = 2.95[us]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 500
Dante_Presat            = FALSE
Decimation_Rate         = 0
Initial_Wait            = 1[s]
Phase                   = {0, 90, 270, 180, 180
Presat_Time             = 5[s]
Presat_Time_Flag        = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 5[s]
Repetition_Time         = 7.18628096[s]
```




```
Filename                = um#1062-2_Carbon-1
Author                  = delta
Experiment              = carbon.jxp
Sample_Id               = um#1062-2
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 13-OCT-2018 10:34:
Revision_Time           = 15-JUL-2019 16:38:

Comment                 = single pulse decou
Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Carbon13
Dim_Title               = Carbon13
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.389766[T] (400[M
X_Acq_Duration          = 1.03809024[s]
X_Domain                = 13C
X_Freq                  = 100.52530333[MHz]
X_Offset                = 100[ppm]
X_Points                = 32768
X_Prescans              = 4
X_Resolution            = 0.96330739[Hz]
X_Sweep                 = 31.56565657[kHz]
X_Sweep_Clipped         = 25.25252525[kHz]
Irr_Domain              = Proton
Irr_Freq                = 399.78219838[MHz]
Irr_Offset              = 5[ppm]
Blanking                = 5[us]
Clipped                 = FALSE
Scans                   = 289
Total_Scans             = 289

Relaxation_Delay        = 2[s]
Recvr_Gain              = 50
Temp_Get                = 21.3[dC]
X_90_Width              = 9.5[us]
X_Acq_Time              = 1.03809024[s]
X_Angle                 = 30[deg]
X_Atn                   = 3.3[dB]
X_Pulse                 = 3.16666667[us]
Irr_Atn_Dec             = 27.307[dB]
Irr_Atn_Dec_Calc        = 27.307[dB]
Irr_Atn_Dec_Default_Calc = 27.307[dB]
Irr_Atn_Noe             = 27.307[dB]
Irr_Dec_Bandwidth_Hz    = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm   = 11.96303566[ppm]
Irr_Dec_Freq            = 399.78219838[MHz]
Irr_Dec_Merit_Factor    = 2.2
Irr_Decoupling          = TRUE
Irr_Noe                 = TRUE
Irr_Noise               = WALTZ
Irr_Offset_Default      = 5[ppm]
Irr_Pwidth              = 0.115[ms]
Irr_Pwidth_Default      = 0.115[ms]
Irr_Pwidth_Default_Calc = 0.115[ms]
Irr_Pwidth_Templ        = 0.115[ms]
Irr_Wurst               = FALSE
Decimation_Rate         = 0
Initial_Wait            = 1[s]
Noe_Time                = 2[s]
Noe_Time_Flag           = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 2[s]
Repetition_Time         = 3.03809024[s]
```

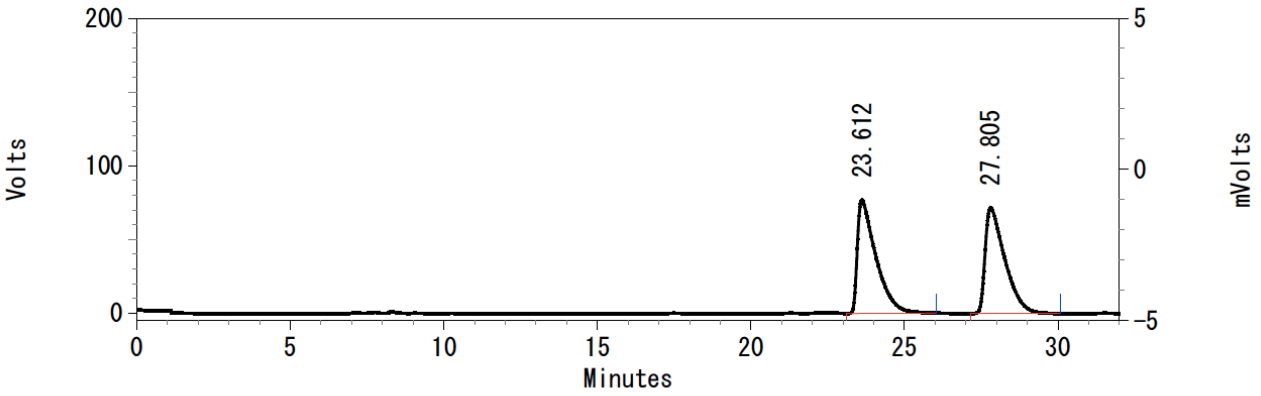


3ha



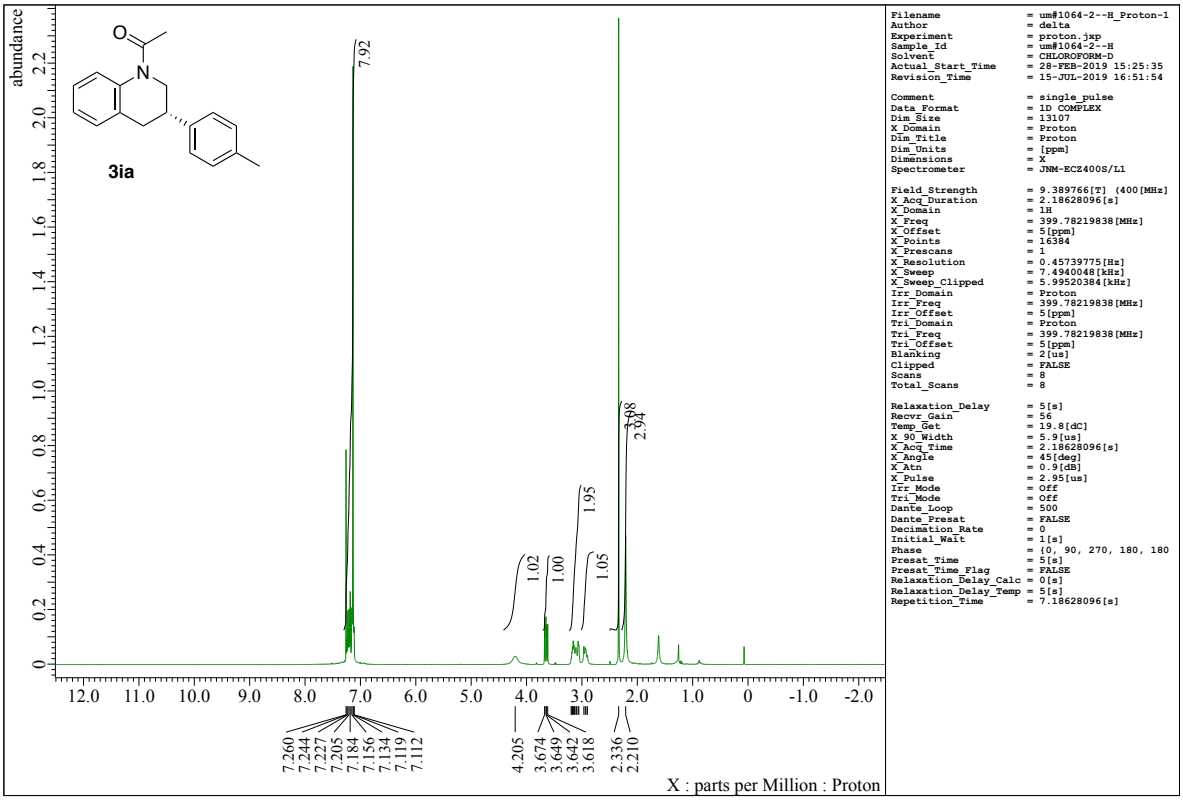
UV Results

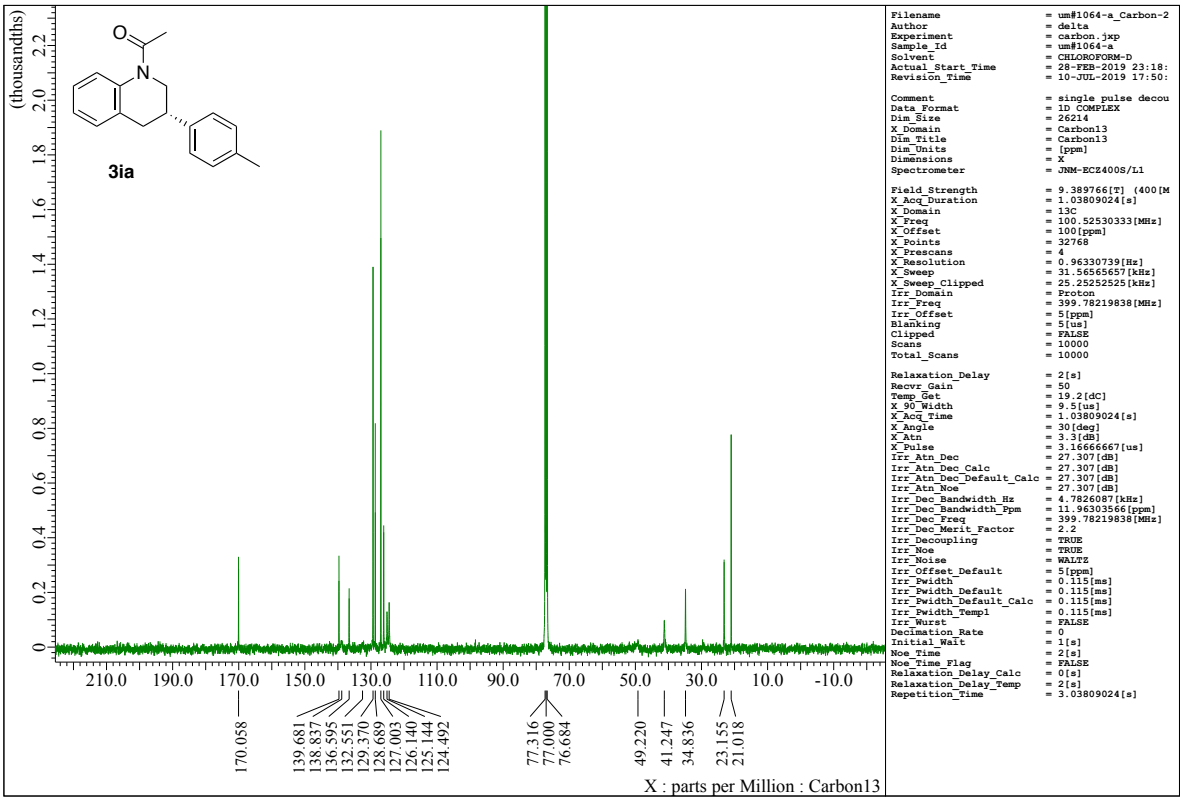
Pk #	Retention Time	Area	Area Percent	Height
1	22.954	21005321	95.964	282738
2	28.843	883500	4.036	15452
Totals		21888821	100.000	298190

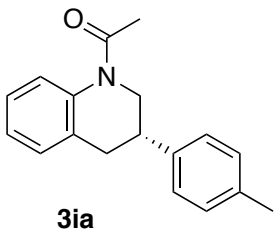


UV Results

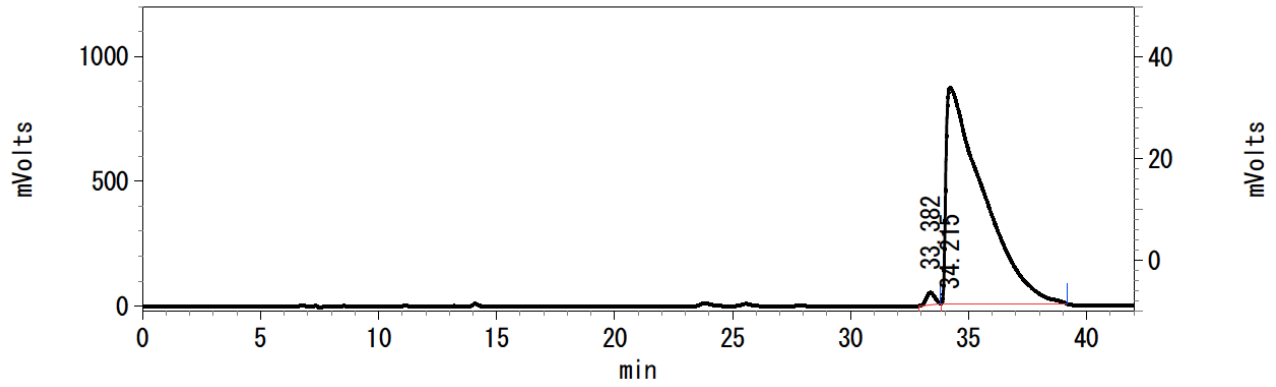
Pk #	Retention Time	Area	Area Percent	Height
1	23.612	3227093	50.358	76838
2	27.805	3181199	49.642	71709
Totals		6408292	100.000	148547


abundance
3ia
7.92
1.02
1.00
1.95
1.05
3.08
2.94
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
7.260 7.244 7.227 7.205 7.184 7.156 7.134 7.119 7.112
4.205 3.674 3.649 3.642 3.618
2.336 2.210
X : parts per Million : Proton
Filename = um#1064-2--H_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = um#1064-2--H
Solvent = CHLOROFORM-D
Actual_Start_Time = 28-FEB-2019 15:25:35
Revision_Time = 15-JUL-2019 16:51:54
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz]
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[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]
Blanking = 2[us]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 19.8[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Loop = 500
Dante_Presat = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Phase = {0, 90, 270, 180, 180
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]

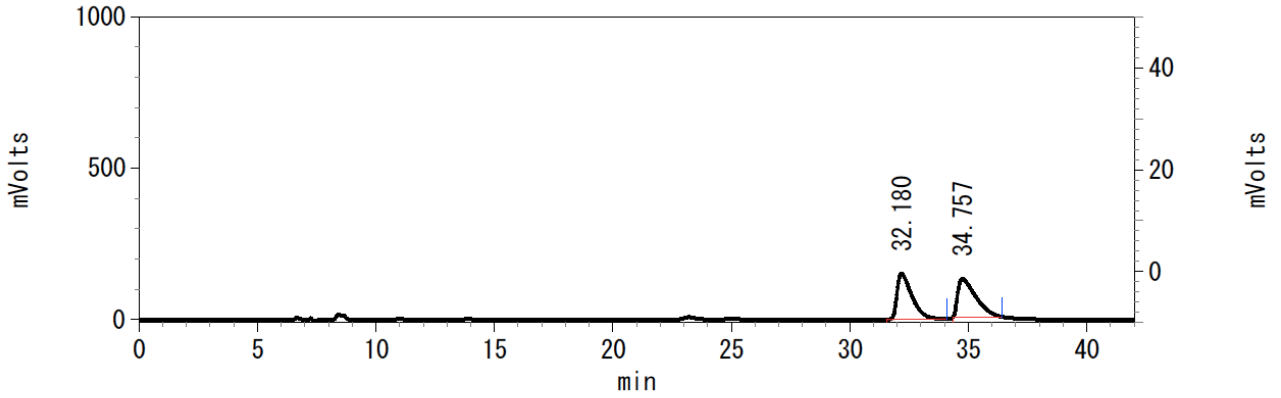

(thousandths)
3ia
210.0 190.0 170.0 150.0 130.0 110.0 90.0 70.0 50.0 30.0 10.0 -10.0
170.058
139.681 138.837 136.595 132.551 129.370 128.689 127.003 126.140 125.144 124.492
77.316 77.000 76.684
49.220 41.247 34.836 23.155 21.018
X : parts per Million : Carbon13
Filename = um#1064-a_Carbon-2
Author = delta
Experiment = carbon.jxp
Sample_Id = um#1064-a
Solvent = CHLOROFORM-D
Actual_Start_Time = 28-FEB-2019 23:18:
Revision_Time = 10-JUL-2019 17:50:
Comment = single pulse decou
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon13
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[M
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Blanking = 5[us]
Clipped = FALSE
Scans = 10000
Total_Scans = 10000
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 19.2[dC]
X_90_Width = 9.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.3[dB]
X_Pulse = 3.16666667[us]
Irr_Atn_Dec = 27.307[dB]
Irr_Atn_Dec_Calc = 27.307[dB]
Irr_Atn_Dec_Default_Calc = 27.307[dB]
Irr_Atn_Noe = 27.307[dB]
Irr_Dec_Bandwidth_Hz = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm = 11.96303566[ppm]
Irr_Dec_Freq = 399.78219838[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noe = TRUE
Irr_Noise = WALTZ
Irr_Offset_Default = 5[ppm]
Irr_Pwidth = 0.115[ms]
Irr_Pwidth_Default = 0.115[ms]
Irr_Pwidth_Default_Calc = 0.115[ms]
Irr_Pwidth_Templ = 0.115[ms]
Irr_Wurst = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Noe_Time = 2[s]
Noe_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 2[s]
Repetition_Time = 3.03809024[s]



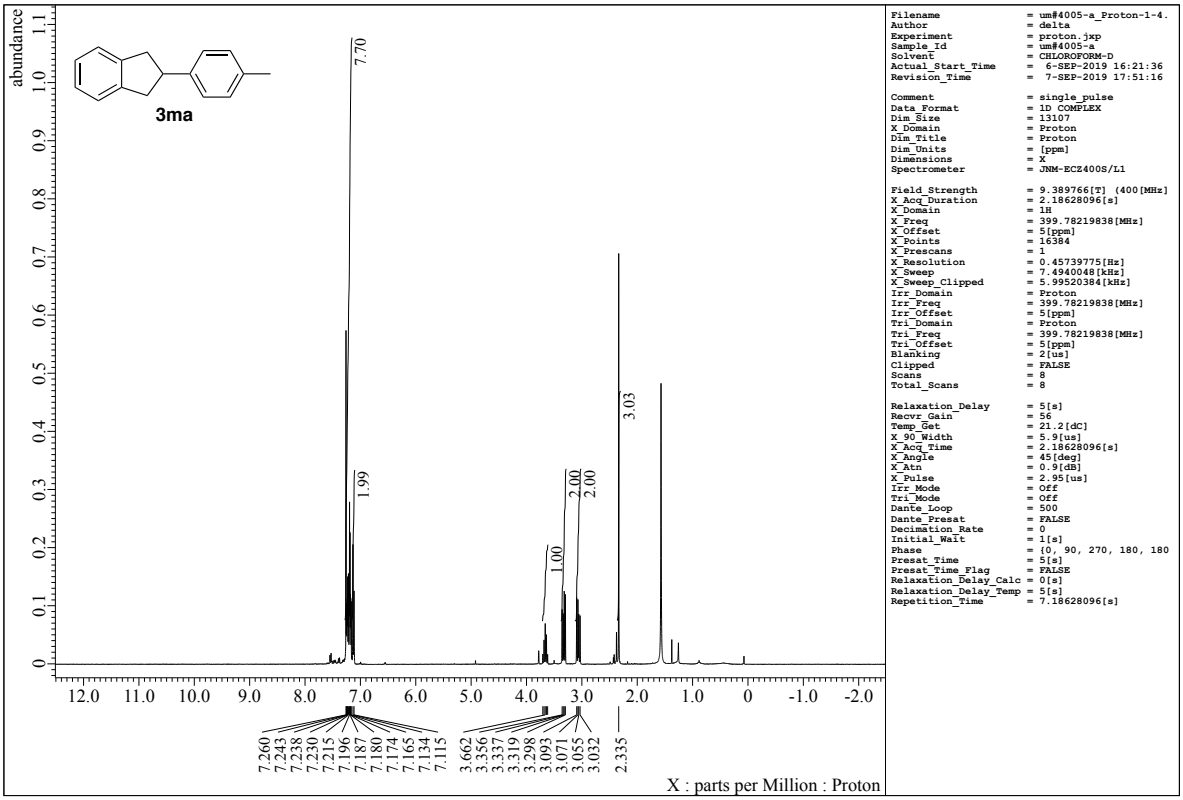
3ia

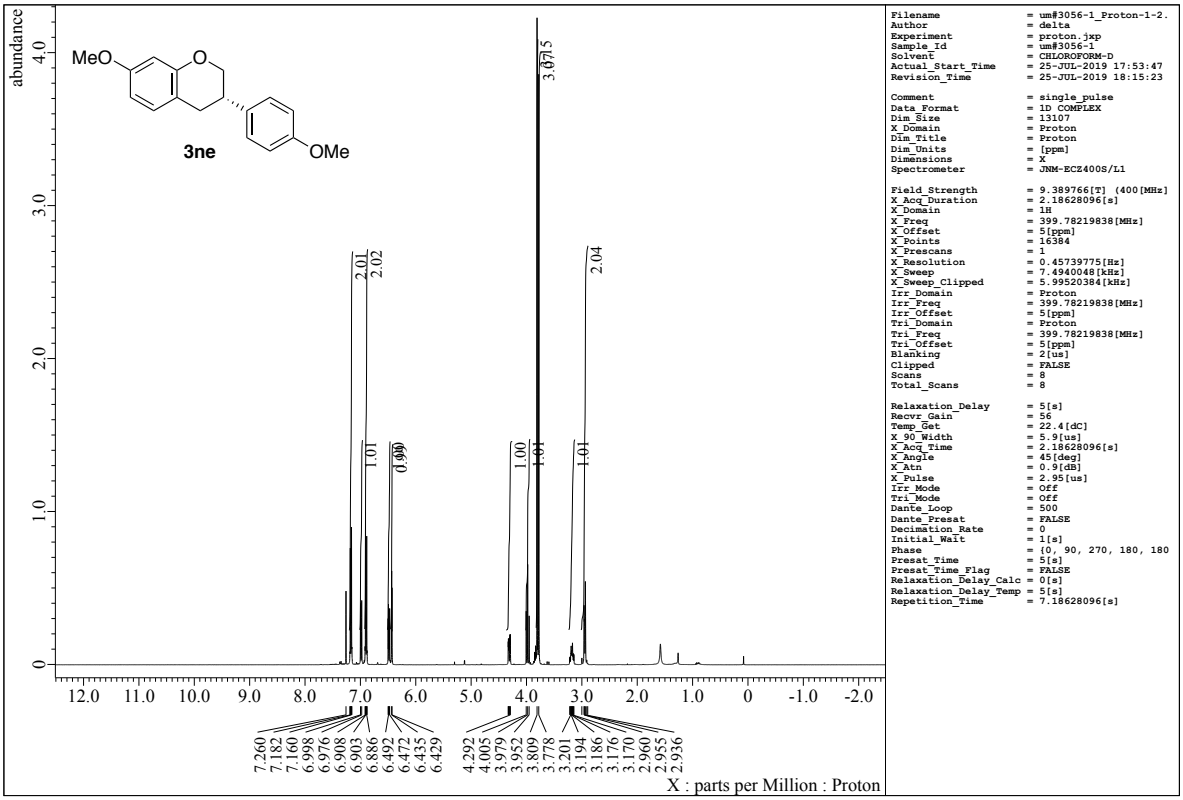


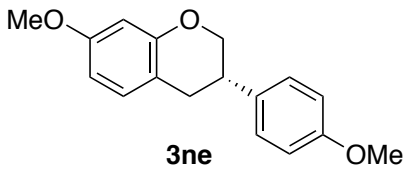
Pk #	Retention Time	Area	Area Percent
1	33.382	1206568	1.257
2	34.215	94791370	98.743



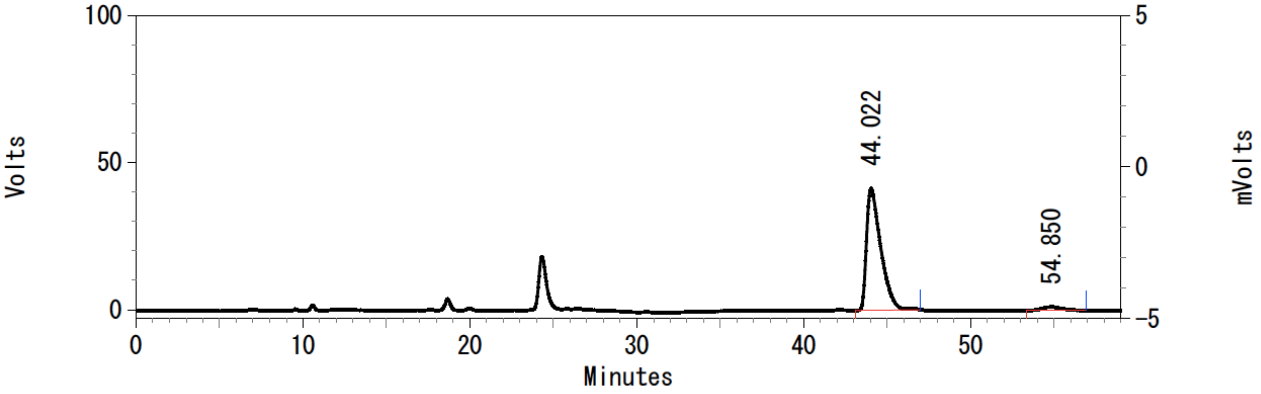
Pk #	Retention Time	Area	Area Percent
1	32.180	6497754	49.518
2	34.757	6624176	50.482


3ma
abundance
7.70
1.99
1.00
2.00
2.00
3.03
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
7.260 7.243 7.238 7.230 7.215 7.196 7.187 7.180 7.174 7.165 7.134 7.115
3.662 3.356 3.337 3.319 3.298 3.093 3.071 3.055 3.032
2.335
X : parts per Million : Proton
Filename = um#4005-a_Proton-1-4.
Author = delta
Experiment = proton.jxp
Sample_Id = um#4005-a
Solvent = CHLOROFORM-D
Actual_Start_Time = 6-SEP-2019 16:21:36
Revision_Time = 7-SEP-2019 17:51:16
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz]
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[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]
Blanking = 2[us]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 21.2[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Loop = 500
Dante_Presat = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Phase = {0, 90, 270, 180, 180
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]


MeO
O
OMe
3ne
abundance
4.0
3.0
2.0
1.0
0
2.01
2.02
2.04
3.07.5
1.01
1.00
1.01
1.01
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
7.260
7.182
7.160
6.998
6.976
6.908
6.903
6.886
6.492
6.472
6.435
6.429
4.292
4.005
3.979
3.952
3.809
3.778
3.201
3.194
3.186
3.176
3.170
2.960
2.955
2.936
X : parts per Million : Proton
Filename = um#3056-1_Proton-1-2.
Author = delta
Experiment = proton.jxp
Sample_Id = um#3056-1
Solvent = CHLOROFORM-D
Actual_Start_Time = 25-JUL-2019 17:53:47
Revision_Time = 25-JUL-2019 18:15:23
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz]
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[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]
Blanking = 2[us]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 22.4[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Loop = 500
Dante_Presat = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Phase = {0, 90, 270, 180, 180
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]

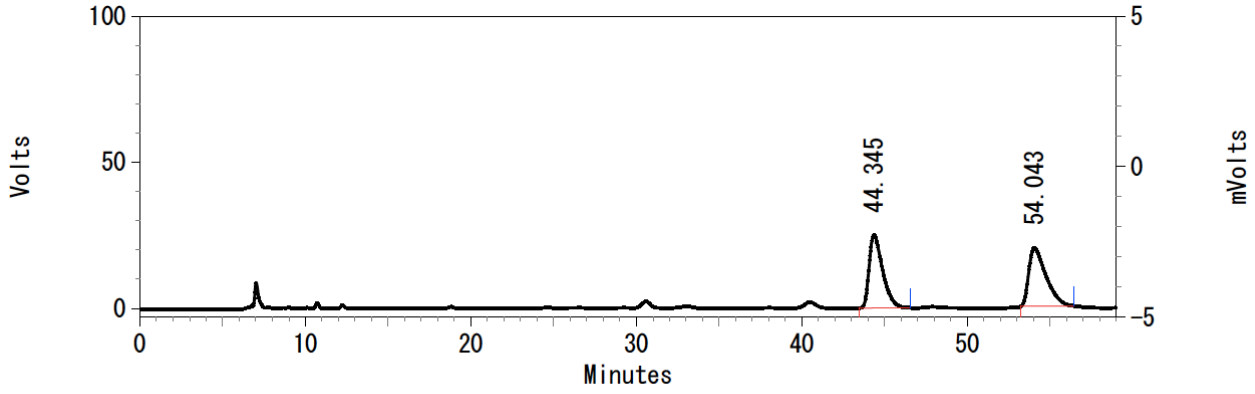


3ne



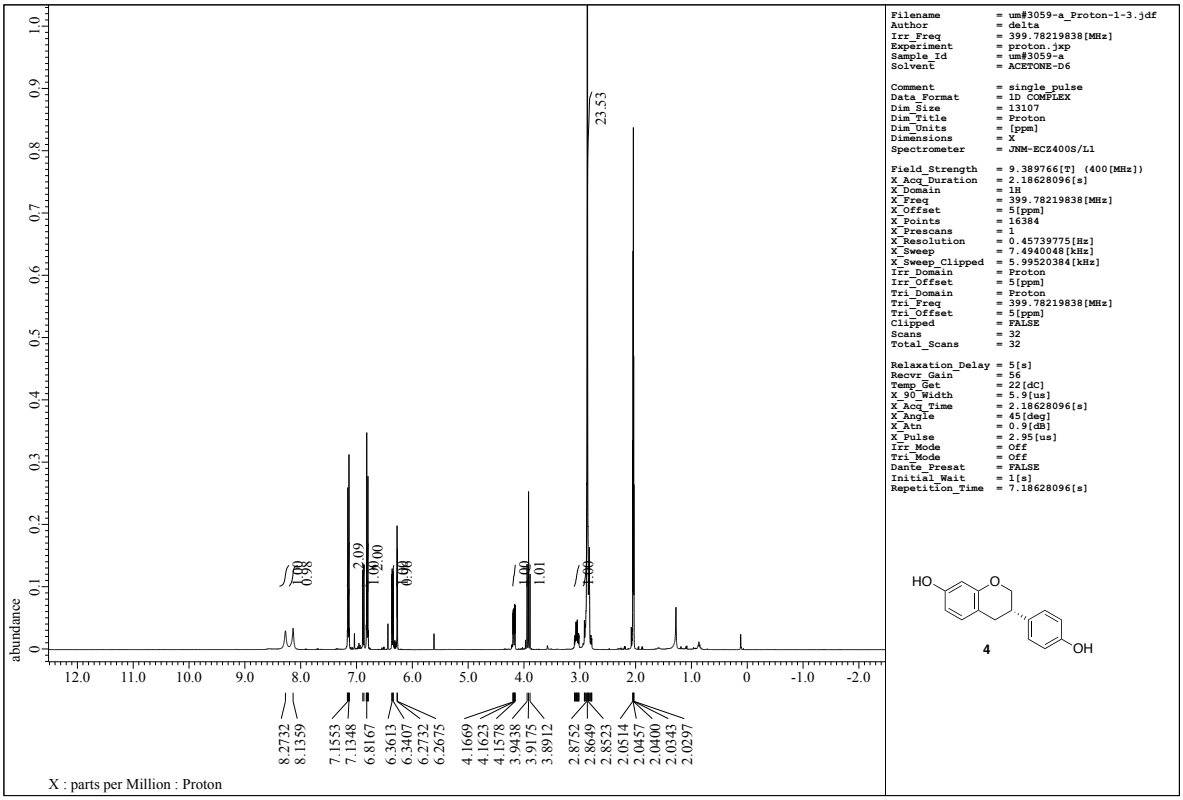
UV-970 Results

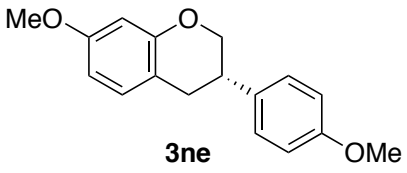
Pk #	Retention Time	Area	Area Percent	Height
1	44.022	2452356	96.105	41385
2	54.850	99401	3.895	1247
Totals		2551757	100.000	42632



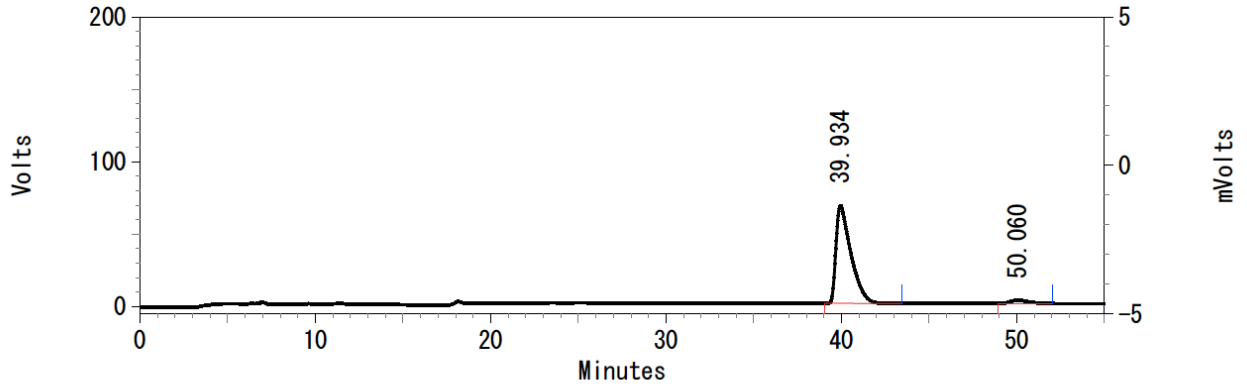
UV-970 Results

Pk #	Retention Time	Area	Area Percent	Height
1	44.345	1418728	49.598	25023
2	54.043	1441708	50.402	19980
Totals		2860436	100.000	45003


abundance
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
0.98 2.09 1.00 0.98 1.00 1.01 3.80 23.53
8.2732 8.1359 7.1553 7.1348 6.8167 6.3613 6.3407 6.2732 6.2675 4.1669 4.1623 4.1578 3.9438 3.9175 3.8912 2.8752 2.8649 2.8523 2.0514 2.0457 2.0400 2.0343 2.0297
X : parts per Million : Proton
Filename = um#3059-a_Proton-1-3.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = um#3059-a
Solvent = ACETONE-D6
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 32
Total_Scans = 32
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 22[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
RepetitTon_Time = 7.18628096[s]
HO
O
OH
4

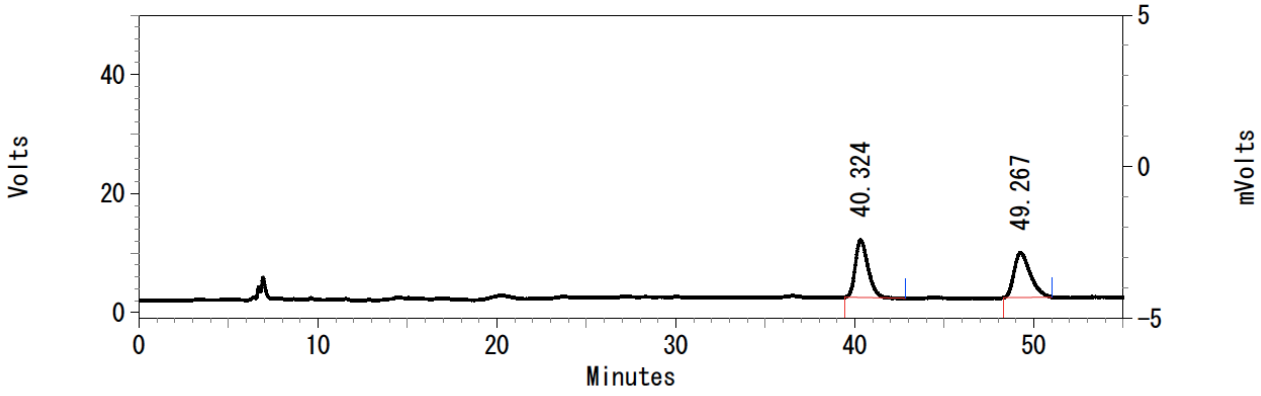


3ne derived from **4**



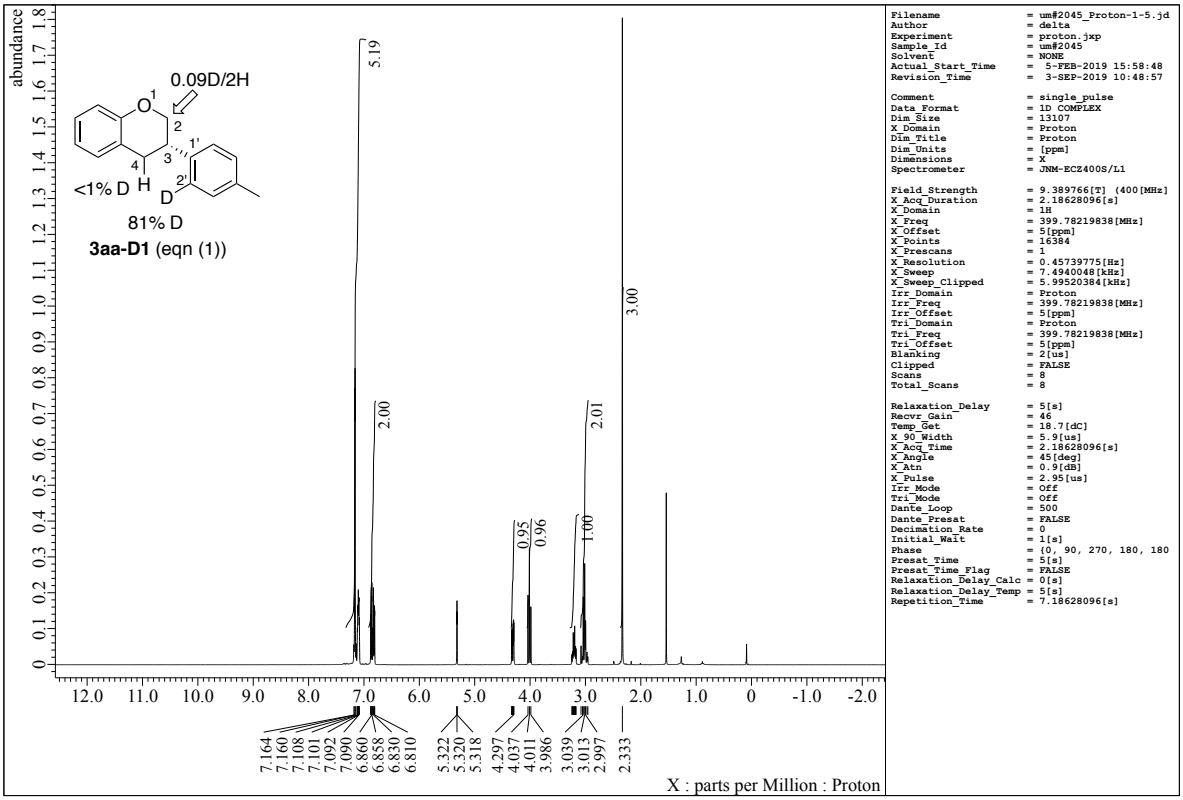
UV-970 Results

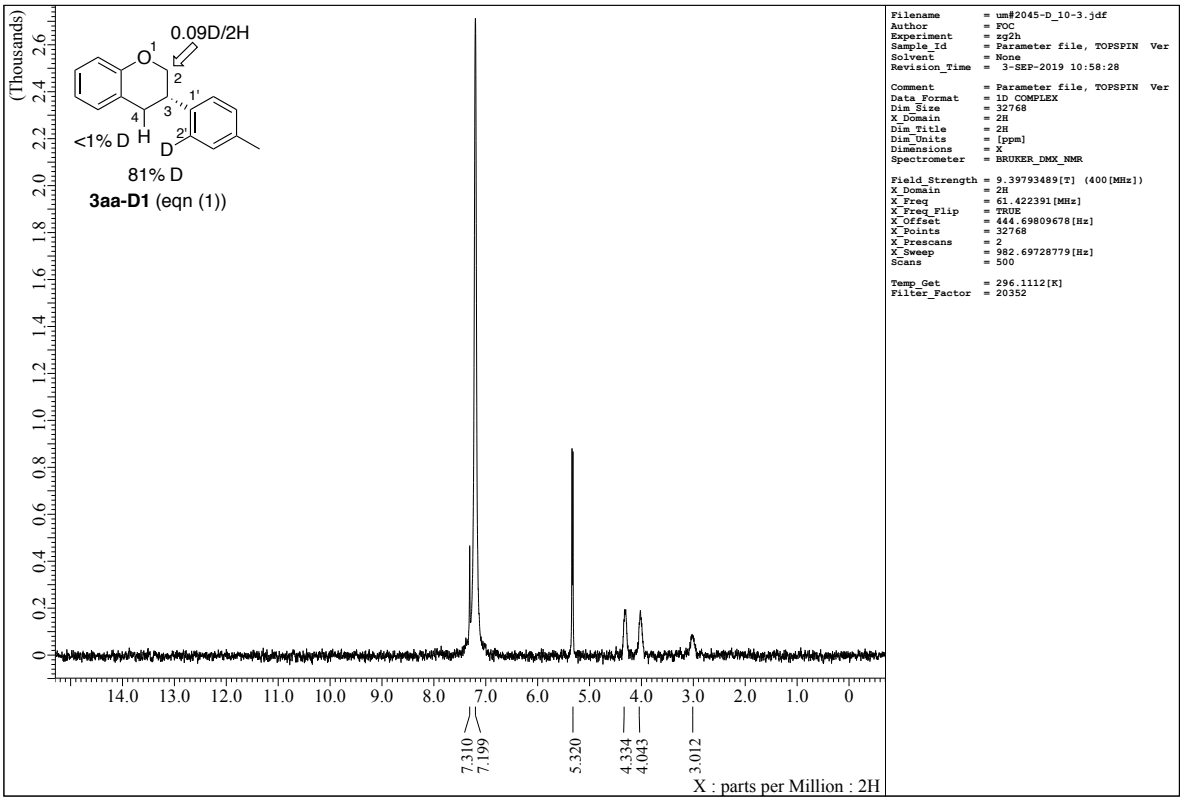
Pk #	Retention Time	Area	Area Percent	Height
1	39.934	3981789	95.842	67596
2	50.060	172734	4.158	2345
Totals		4154523	100.000	69941

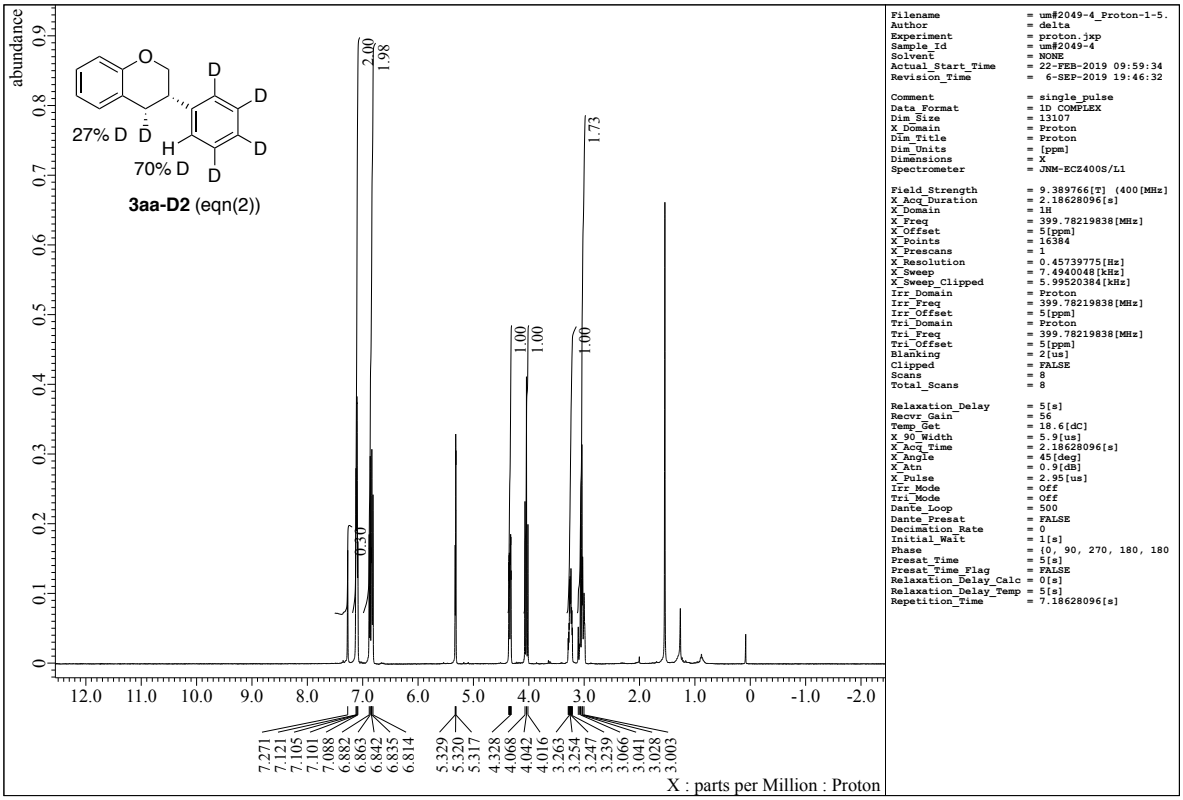


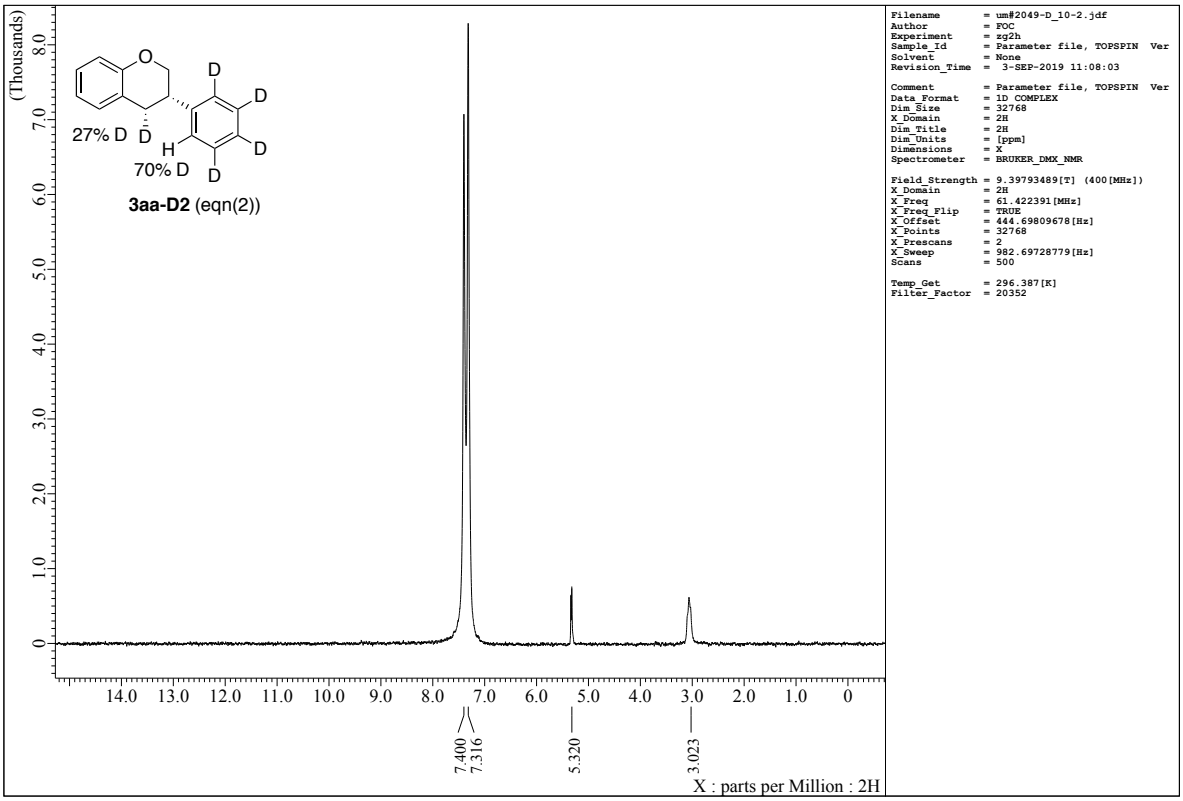
UV-970 Results

Pk #	Retention Time	Area	Area Percent	Height
1	40.324	472941	49.826	9661
2	49.267	476243	50.174	7521
Totals		949184	100.000	17182


abundance
0.09D/2H
<1% D H
81% D
3aa-D1 (eqn (1))
5.19
2.00
2.01
3.00
0.95
0.96
1.00
7.164
7.160
7.108
7.101
7.092
7.090
6.860
6.858
6.830
6.810
5.322
5.320
5.318
4.297
4.037
4.011
3.986
3.039
3.013
2.997
2.333
X : parts per Million : Proton
Filename = um#2045_Proton-1-5.jd
Author = delta
Experiment = proton.jxp
Sample_Id = um#2045
Solvent = NONE
Actual_Start_Time = 5-FEB-2019 15:58:48
Revision_Time = 3-SEP-2019 10:48:57
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz]
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[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]
Blanking = 2[us]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 18.7[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Loop = 500
Dante_Presat = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Phase = {0, 90, 270, 180, 180
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]


(Thousands)
0.09D/2H
<1% D H
81% D
3aa-D1 (eqn (1))
7.310
7.199
5.320
4.334
4.043
3.012
X : parts per Million : 2H
Filename = um#2045-D_10-3.jdf
Author = FOC
Experiment = zg2h
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = None
Revision_Time = 3-SEP-2019 10:58:28
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 2H
Dim_Title = 2H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 2H
X_Freq = 61.422391[MHz]
X_Freq_Flip = TRUE
X_Offset = 444.69809678[Hz]
X_Points = 32768
X_Prescans = 2
X_Sweep = 982.69728779[Hz]
Scans = 500
Temp_Get = 296.1112[K]
Filter_Factor = 20352


abundance
27% D
70% D
3aa-D2 (eqn(2))
2.00
1.98
1.73
1.00
1.00
1.00
0.30
7.271
7.121
7.105
7.101
7.088
6.882
6.863
6.842
6.835
6.814
5.329
5.320
5.317
4.328
4.068
4.042
4.016
3.263
3.254
3.247
3.239
3.066
3.041
3.028
3.003
X : parts per Million : Proton
Filename = um#2049-4_Proton-1-5.
Author = delta
Experiment = proton.jxp
Sample_Id = um#2049-4
Solvent = NONE
Actual_Start_Time = 22-FEB-2019 09:59:34
Revision_Time = 6-SEP-2019 19:46:32
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz]
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[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]
Blanking = 2[us]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 18.6[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Loop = 500
Dante_Presat = FALSE
Decimation_Rate = 0
Initial_Wait = 1[s]
Phase = {0, 90, 270, 180, 180
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]


(Thousands)
27% D
70% D
3aa-D2 (eqn(2))
7.400
7.316
5.320
3.023
X : parts per Million : 2H
Filename = um#2049-D_10-2.jdf
Author = FOC
Experiment = zg2h
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = None
Revision_Time = 3-SEP-2019 11:08:03
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 2H
Dim_Title = 2H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 2H
X_Freq = 61.422391[MHz]
X_Freq_Flip = TRUE
X_Offset = 444.69809678[Hz]
X_Points = 32768
X_Prescans = 2
X_Sweep = 982.69728779[Hz]
Scans = 500
Temp_Get = 296.387[K]
Filter_Factor = 20352