Supplementary Information

Enantioselective 1,4-addition of cyclopropylboronic acid catalyzed by rhodium/chiral diene complexes
Ryosuke Takechi and Takahiro Nishimura*

Department of Chemistry, Graduate School of Science, Kyoto University
Sakyo, Kyoto 606-8502, Japan
E-mail: tnishi@kuchem.kyoto-u.ac.jp

Contents of Supplementary Information:

1. General S-2
2. Materials S-2
3. Preparation of (S,S)-L3, [RhCl((S,S)-L3)]2, and [Rh(OH)((S,S)-L3)]2 S-2 — S-4
4. Preparation of electron-deficient alkenes 1 and 4 S-4
5. Preparation of compounds 10 and 10-d2 S-4 — S-5
6. General procedure for Table 1 S-5
7. General procedure for Scheme 2 S-5
8. General procedure for Scheme 3 S-5
9. General procedure for eq 3 S-6
10. Characterization of the products S-6 — S-14
11. X-Ray data of 3a S-15 — S-16
12. 1H and 13C NMR spectra and HPLC charts S-17 — S-62
1. General

All anaerobic and moisture-sensitive manipulations were carried out with standard Schlenk techniques under predried nitrogen. NMR spectra were recorded on a JEOL JNM ECA-600 spectrometer (600 MHz for $^1$H, 150 MHz for $^{13}$C). Chemical shifts are reported in $\delta$ (ppm) referenced to the residual peaks of CDCl$_3$ ($\delta$ 7.26) and CD$_2$Cl$_2$-d$_2$ (CDHCl$_2$, $\delta$ 5.30) for $^1$H NMR, and CDCl$_3$ ($\delta$ 77.00) for $^{13}$C NMR. The following abbreviations are used; s, singlet; d, doublet; t, triplet; q, quartet; sept, septet; m, multiplet; br, broad. High-resolution mass spectra (TOF-MS) were obtained with a Bruker micrOTOF spectrometer. Flash column chromatography was performed with Silica Gel 60 N (spherical, neutral) (Cica-Reagent). Preparative thin-layer chromatography was performed with Silica Gel 60 PF$_{254}$ (Merck). Alumina (activated 200) for column chromatography was purchased from Nacalai Tesque.

2. Materials

Toluene and 1,4-dioxane were purified by passing through a neutral alumina column under N$_2$. Rhodium complexes [RhCl((S,S)-Fc-tfb*)]$_2$, [RhCl((R,R)-Ph-tfb*)]$_2$, [RhCl((S,S)-Bn-tfb*)]$_2$, [Rh(OH)((S,S)-Bn-tfb*)]$_2$, [RhCl((R)-L1)]$_2$, [RhCl((R)-L2)]$_2$, and [RhCl((R)-binap)]$_2$ were prepared according to the reported procedures. Ligand (S,S)-L3 [RhCl((S,S)-L3)]$_2$, and [Rh(OH)((S,S)-L3)]$_2$ were prepared as shown below.

3. Preparation of (S,S)-L3, [RhCl((S,S)-L3)]$_2$, and [Rh(OH)((S,S)-L3)]$_2$

Ligand L3: To a solution of (R,R)-s1$^1$ (527 mg, 1.0 mmol) and PdCl$_2$(IPr)(3-chloropyridine) $^6$ (34.0 mg, 0.050 mmol) in THF (4 mL) was added

---


---
neopentylmagnesium bromide (0.91 M in THF, 5.5 mL, 5.0 mmol), prepared from neopentyl bromide with Mg, at room temperature and the mixture was stirred for 2 h. Saturated NH₄Cl (aq.) was added to the mixture and it was extracted with diethyl ether. The organic layer was passed through a short column of alumina with Et₂O, and the solvent was removed on a rotary evaporator.

The residue was subjected to flash column chromatography on silica gel with hexane to give (S,S)-L3 (311 mg, 0.81 mmol, 81% yield). ¹H NMR (CDCl₃) δ 0.87 (s, 18H), 2.09 (d, J = 13.6 Hz, 2H), 2.14 (d, J = 13.6 Hz, 2H), 4.91 (d, J = 5.5 Hz, 2H), 6.39 (d, J = 5.5 Hz, 2H); ¹³C NMR (CDCl₃) δ 29.8 (6C), 32.3 (2C), 47.5 (2C), 47.7 (2C), 130.6–130.8 (m, 2C), 135.0 (2C), 136.1–142.2 (m, 4C), 152.8 (2C). HRMS (APCI) calcd for C₂₂H₂₈F₄ (M)⁺ 366.1965, found 366.1968. [α]²⁰ D +32 (c 0.99, CHCl₃) for (S,S)-L3.

![Reaction Diagram](image-url)

[RhCl((S,S)-L3)]₂: To a suspension of [RhCl(C₂H₄)₂]₂ ⁷ (236 mg, 0.61 mmol) in dichloromethane (4 mL) was added (S,S)-L3 (311 mg, 0.81 mmol) in dichloromethane (1 mL) at room temperature and the mixture was stirred for 3 h. The mixture was concentrated under vacuum. The residue was subjected to flash column chromatography on silica gel with hexane, and then, dichloromethane under air to give [RhCl((S,S)-L3)]₂ (309 mg, 0.31 mmol, 76% yield). ¹H NMR (CDCl₃) δ 0.66 (s, 36H), 1.58 (d, J = 14.0 Hz, 4H), 2.14 (d, J = 14.0 Hz, 4H), 3.39 (d, J = 5.8 Hz, 4H), 5.39 (d, J = 5.8 Hz, 4H). HRMS (ESI) calcd for C₂₂H₂₈Cl₂F₄Rh (M/2+Cl)⁺ 539.0408, found 539.0405. [α]²⁰ D +11 (c 0.26, CHCl₃) for [RhCl((S,S)-L3)]₂.

[RhCl((S,S)-L3)]₂ + 1 M KOHaq [Rh(OH)((S,S)-L3)]₂

[Rh(OH)((S,S)-L3)]₂: To a solution of [RhCl((S,S)-L3)]₂ (236 mg, 0.31 mmol) in THF (2 mL) was added KOHaq (1 M, 6.1 mL, 6.1 mmol) at room temperature and the mixture was stirred for 0.5 h. Most of THF was removed under vacuum, and dichloromethane (1 mL) was added. The mixture was washed with 1 M KOHaq (1 mL) and H₂O (1 mL x 3) under nitrogen atmosphere. The organic layer was passed through a short column of Na₂SO₄ with dichloromethane and concentrated on a rotary evaporator. The residue was dried under vacuum to give [Rh(OH)((S,S)-L3)]₂ (268 mg, 0.28 mmol, 90% yield). ¹H NMR (CD₂Cl₂) δ 0.61 (s, 36H), 1.02 (d,

---

J = 13.6 Hz, 4H), 2.66 (d, J = 13.6 Hz, 4H), 2.95 (d, J = 5.5 Hz, 4H), 5.31 (d, J = 5.5 Hz, 4H). HRMS (ESI) calc'd for C_{12}H_{26}F_{4}Rh (M/2–OH)^+ 469.1020, found 469.10188. [α]_{D}^{20} −12 (c 0.25, CH_{2}Cl_{2}) for [RhCl((S,S)-L3)]_{2}.

4. Preparation of electron-deficient alkenes 1 and 4

Compounds 1a (CAS: 16212-08-1), 8 1b (CAS: 876293-46-8), 9 1c (CAS: 35836-45-4), 9 1d (CAS: 107772-61-2), 9 1e (CAS: 1380035-31-3), 9 1f (CAS: 71964-05-1), 9 1g (CAS: 121033-92-9), 10 1h (CAS: 1380035-23-3), 8 1i (CAS: 14554-08-6), 11 and 1j (CAS: 17299-32-0) 12 were prepared according to the reported procedures. Compounds 4e (CAS: 710969-63-4), 13 4f (CAS: 710969-63-4), 14 4g (CAS: 28638-59-7), 15 4h (CAS: 5153-70-8), 15 and 4i (CAS: 5576-97-6) 15 were prepared according to the reported procedures. Cyclopropylboronic acid (2), compounds 4a–d, and 4j were purchased from commercial suppliers and used as received. Cyclopropylboronate 10 was prepared as shown below. 16

5. Preparation of compounds 10 (CAS: 1612136-76-1) and 10-d_{2}

To a solution of diiodomethane (1.21 mL, 15 mmol) in dichloromethane (25 mL) at 0 °C was added diethylzinc (1 M in toluene, 7.5 mL, 7.5 mmol), and the mixture was stirred at the same temperature for 2 h. Compound s2 (648 mg, 3.00 mmol) in dichloromethane (5 mL) was added to the mixture at 0 °C, and the mixture was stirred at room temperature for 36 h. Saturated NH_{4}Cl (aq.) was added to the mixture and it was extracted with dichloromethane. The organic layer was dried over Na_{2}SO_{4}, filtered, and concentrated on a rotary evaporator. The residue was subjected to flash column chromatography on silica gel with hexane/ethyl acetate (2:1) to give 10 (581 mg, 2.52 mmol, 84% yield). ^{1}H NMR (CDCl_{3}) δ 0.16–0.25 (m, 1H), 0.92–0.98 (m, 1H), 0.97 (s, 6H), 1.08–

---

1.14 (m, 1H), 2.00–2.06 (m, 1H), 3.59 (s, 4H), 7.08 (d, \( J = 7.5 \text{ Hz} \), 2H), 7.12 (t, \( J = 7.5 \text{ Hz} \), 1H), 7.24 (t, \( J = 7.5 \text{ Hz} \), 2H).

Compound 10–d was prepared in 81% yield according to the same procedures for 10 using diiodomethane-\( \text{d}_2 \). \(^1\)H NMR (CDCl\(_3\)) \( \delta \) 0.17 (d, \( J = 5.5 \text{ Hz} \), 1H), 0.96 (s, 6H), 2.02 (d, \( J = 5.5 \text{ Hz} \), 1H), 3.58 (s, 4H), 7.07 (d, \( J = 7.5 \text{ Hz} \), 2H), 7.11 (t, \( J = 7.5 \text{ Hz} \), 1H), 7.23 (t, \( J = 7.5 \text{ Hz} \), 2H).

HRMS (ESI) calcd for C\(_{14}\)H\(_{17}\)BD\(_2\)NaO\(_2\) (M+Na)\(^+\) 255.1498, found 255.1495.

6. General procedure for Table 1

A rhodium catalyst (0.0030 mmol of Rh), 1a (25.8 mg, 0.10 mmol), K\(_3\)PO\(_4\) (21.2 mg, 0.10 mmol) and cyclopropylboronic acid (2) (21.5 mg, 0.25 mmol) were placed in a Schlenk tube under nitrogen. Toluene (0.4 mL) was added and the mixture was stirred at 60 °C for 12 h. The mixture was passed through a short column of silica gel with ethyl acetate as eluent. The solvent was removed on a rotary evaporator and the yield of the product was determined by \(^1\)H NMR using 1,4-dimethoxybenzene as an internal standard. For isolation of the product, the crude mixture was subjected to preparative TLC on silica gel with toluene to give 3a. The ee of 3a was determined by chiral HPLC (Daicel chiralpak AD-H).

7. General procedure for Scheme 2

[Rh(OH)((S,S)-Fc-tfb*)]\(_2\) (4.4 mg, 0.0060 mmol of Rh), 1 (0.20 mmol), K\(_3\)PO\(_4\) (42.4 mg, 0.20 mmol), and cyclopropylboronic acid (2) (43.0 mg, 0.50 mmol) were placed in a Schlenk tube under nitrogen. 1,4-Dioxane or toluene (0.8 mL) was added and the mixture was stirred at 60 °C for 24 h. The mixture was passed through a short column of silica gel with ethyl acetate as eluent. The solvent was removed on a rotary evaporator and the residue was subjected to preparative TLC on silica gel with hexane/ethyl acetate to give 3.

8. General procedure for Scheme 3

[Rh(OH)((S,S)-Bn-tfb*)]\(_2\) (3.2 mg, 0.0060 mmol of Rh), 4 (0.20 mmol), K\(_3\)PO\(_4\) (42.4 mg, 0.20 mmol), and cyclopropylboronic acid (2) (60.1 mg, 0.70 mmol) were placed in a Schlenk tube under nitrogen. 1,4-Dioxane (0.8 mL) was added and the mixture was stirred at 60 °C for 12 h. The mixture was passed through a short column of silica gel with ethyl acetate as eluent. The solvent was removed on a rotary evaporator and the residue was subjected to preparative TLC on silica gel with hexane/ethyl acetate to give 5. For nitroalkenes 4g–i, the reaction was conducted in the presence of [Rh(OH)((S,S)-L3)]\(_2\) (2.8 mg, 0.0060 mmol of Rh) and KHF\(_2\) (15.6 mg, 0.20 mmol) in toluene (0.8 mL) instead of [Rh(OH)((S,S)-Bn-tfb*)]\(_2\) and K\(_3\)PO\(_4\), respectively.

9. General procedure for eq 3

[Rh(OH)((S,S)-Bn-tfb*)]\(_2\) (1.6 mg, 0.0030 mmol of Rh), 4j (7.0 mg, 0.10 mmol), K\(_3\)PO\(_4\)
(21.2 mg, 0.10 mmol), and cyclopropylboronate 10 (69.0 mg, 0.30 mmol) were placed in a Schlenk tube under nitrogen. 1,4-Dioxane (0.8 mL) and methanol (12 µL, 0.30 mmol) were added and the mixture was stirred at 80 °C for 24 h. The mixture was passed through a short column of silica gel with ethyl acetate as eluent. The solvent was removed on a rotary evaporator and the residue was subjected to preparative TLC on silica gel with hexane/ethyl acetate to give 11.

10. Characterization of the products

The absolute configuration of the product 3a was determined by X-ray crystallographic analysis. For others, they were assigned by consideration of the stereochemical pathway.

**Compound 3a** (Table 1, entry 2, 96% yield, 97% ee). The ee was measured by HPLC (Chiralcel OD-H column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, t₁ = 22.6 min (R), t₂ = 25.2 min (S)); [α]$_{D}^{20}$ +31 (c 1.08, CHCl₃) for 97% ee (S). $^1$H NMR (CDCl₃) δ 0.14–0.20 (m, 1H), 0.29–0.35 (m, 1H), 0.39–0.45 (m, 1H), 0.57–0.64 (m, 1H), 1.01–1.08 (m, 1H), 2.37 (s, 3H), 2.51 (t, d, $J$ = 9.5, 8.0, 5.5 Hz, 1H), 3.60 (dd, $J$ = 14.3, 5.5 Hz, 1H), 3.63 (dd, $J$ = 14.3, 8.0 Hz, 1H), 7.03 (d, $J$ = 8.1 Hz, 2H), 7.12–7.19 (m, 5H), 7.55 (d, $J$ = 8.1 Hz, 2H); $^{13}$C NMR (CDCl₃) δ 4.8, 5.8, 18.1, 21.5, 45.7, 62.3, 126.6, 127.5, 127.8, 128.4, 129.5, 137.1, 141.7, 144.0. HRMS (ESI) calcd for C₁₈H₂₀NaO₂S (M+Na)$^+$ 323.1076, found 323.1067.

**Compound 3b** (Scheme 2, 97% yield, 93% ee). The ee was measured by HPLC (Chiralpak AD-H column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, t₁ = 22.7 min (R), t₂ = 24.9 min (S)); [α]$_{D}^{20}$ +18 (c 1.00, CHCl₃) for 93% ee (S). $^1$H NMR (CDCl₃) δ 0.20–0.28 (m, 2H), 0.45–0.49 (m, 2H), 1.03–1.10 (m, 1H), 2.40 (s, 3H), 2.71 (td, $J$ = 8.8, 4.8 Hz, 1H), 3.50 (dd, $J$ = 14.3, 4.8 Hz, 1H), 3.72 (dd, $J$ = 14.3, 8.8 Hz, 1H), 5.96 (d, $J$ = 2.7 Hz, 1H), 6.15 (dd, $J$ = 2.7, 1.4 Hz, 1H), 7.11 (d, $J$ = 1.4 Hz, 1H), 7.24 (d, $J$ = 8.1 Hz, 2H), 7.64 (d, $J$ = 8.1 Hz, 2H); $^{13}$C NMR (CDCl₃) δ 4.6, 4.9, 16.0, 21.5, 38.9, 59.9, 106.3, 110.0, 127.8, 129.6, 136.9, 141.4, 144.1, 153.7. HRMS (ESI) calcd for C₁₆H₁₈NaO₂S (M+Na)$^+$ 313.0869, found 313.0873.
Compound 3c (Scheme 2, 97% yield, 93% ee). The ee was measured by HPLC (Chiralpak AD-H column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, $t_1 = 25.7$ min ($S$), $t_2 = 27.5$ min ($R$)); $[\alpha]_D^{30} +21$ (c 0.99, CHCl$_3$) for 93% ee ($R$). $^1$H NMR (CDCl$_3$) $\delta$ 0.27–0.38 (m, 2H), 0.51–0.57 (m, 1H), 0.58–0.63 (m, 1H), 1.04–1.11 (m, 1H), 2.40 (s, 3H), 2.88 (dt, $J = 9.5$, 6.1 Hz, 1H), 3.61 (d, $J = 6.1$ Hz, 2H), 6.62 (d, $J = 3.4$ Hz, 1H), 6.81 (dd, $J = 5.5$, 3.4 Hz, 1H), 7.07 (d, $J = 5.5$ Hz, 1H), 7.23 (d, $J = 8.1$ Hz, 2H), 7.64 (d, $J = 8.1$ Hz, 2H); $^{13}$C NMR (CDCl$_3$) $\delta$ 5.3, 5.8, 19.1, 21.5, 40.9, 63.2, 123.8, 123.5, 126.5, 127.9, 129.6, 137.1, 144.3, 145.3. HRMS (ESI) calcd for C$_{16}$H$_{18}$NaO$_2$S (M+Na)$^+$ 329.0640, found 329.0642.

Compound 3d (Scheme 2, 51% yield, 96% ee). The ee was measured by HPLC (Chiralpak AD-H column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, $t_1 = 25.7$ min ($R$), $t_2 = 32.6$ min ($S$)); $[\alpha]_D^{30} +30$ (c 1.01, CHCl$_3$) for 96% ee ($S$). $^1$H NMR (CDCl$_3$) $\delta$ 0.14–0.20 (m, 1H), 0.31–0.37 (m, 1H), 0.43–0.49 (m, 1H), 0.61–0.67 (m, 1H), 0.99–1.06 (m, 1H), 2.38 (s, 3H), 2.48 (dt, $J = 9.5$, 6.8 Hz, 1H), 3.62 (d, $J = 6.8$ Hz, 2H), 7.09 (dd, $J = 8.1$, 4.1 Hz, 1H), 7.19 (d, $J = 8.1$ Hz, 2H), 7.35–7.39 (m, 1H), 7.55 (d, $J = 8.1$ Hz, 2H), 8.32 (s, 1H), 8.40 (d, $J = 4.1$ Hz, 1H); $^{13}$C NMR (CDCl$_3$) $\delta$ 4.9, 5.9, 18.1, 21.5, 43.5, 61.6, 123.3, 127.8, 129.7, 134.6, 136.7, 137.1, 144.5, 148.1, 149.2. HRMS (ESI) calcd for C$_{17}$H$_{19}$NNaO$_2$S (M+Na)$^+$ 324.1029, found 324.1020.

Compound 3e (Scheme 2, 78% yield, 83% ee). The ee was measured by HPLC (Chiralpak AS-H column, hexane/2-propanol = 80:20, flow 0.5 mL/min, 254 nm, $t_1 = 33.9$ min ($S$), $t_2 = 48.9$ min ($R$)); $[\alpha]_D^{30} +37$ (c 1.03, CHCl$_3$) for 83% ee ($S$). $^1$H NMR (CDCl$_3$) $\delta$ 0.09–0.18 (m, 2H), 0.33–0.44 (m, 2H), 0.71–0.78 (m, 1H), 1.52 (s, 3H), 1.53 (s, 3H), 2.36–2.46 (m, 1H), 2.43 (s, 3H), 3.18 (dd, $J = 14.3$, 8.2 Hz, 1H), 3.31 (dd, $J = 14.3$, 4.1 Hz, 1H), 4.63 (d, $J = 10.2$ Hz, 1H), 7.31
\[(d, J = 8.1 \text{ Hz}, 2H), 7.71 (d, J = 8.1 \text{ Hz}, 2H); ^{13}\text{C NMR (CDCl}_3) \delta 3.0, 3.7, 16.6, 18.2, 21.5, 25.7, 37.3, 61.8, 124.2, 128.0, 129.5, 133.7, 137.6, 144.1. \text{ HRMS (ESI) calcd for C}_{16}\text{H}_{22}\text{NaO}_2\text{S (M+Na)}^+ 301.1233, \text{ found 301.1231.} \]

![Diagram 3f](image)

**Compound 3f** (Scheme 2, 94% yield, 96% ee). The ee was measured by HPLC (Chiralpak IC column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, \(t_1 = 44.1 \text{ min (R)}, t_2 = 46.3 \text{ min (S)}\); \([\alpha]_{D}^{20} = -27 (c 1.00, \text{CHCl}_3)\) for 96% ee \((R)\). \(^1\text{H NMR (CDCl}_3) \delta -0.02–0.03 (m, 1H), 0.17–0.22 (m, 1H), 0.33–0.39 (m, 1H), 0.50–0.56 (m, 1H), 0.58–0.65 (m, 1H), 0.82–0.90 (m, 3H), 1.20–1.30 (m, 4H), 1.32–1.41 (m, 1H), 1.43–1.51 (m, 1H), 1.59–1.67 (m, 1H), 2.45 (s, 3H), 3.14 (dd, \(J = 14.3, 7.5 \text{ Hz}, 1H\)), 3.21 (dd, \(J = 14.3, 4.1 \text{ Hz}, 1H\)), 7.34 (d, \(J = 8.1 \text{ Hz}, 2H\)), 7.78 (d, \(J = 8.1 \text{ Hz}, 2H\)); \(^{13}\text{C NMR (CDCl}_3) \delta 3.7, 6.0, 14.0, 16.5, 21.6, 22.9, 28.5, 34.4, 39.2, 61.5, 127.8, 129.8, 137.6, 144.3. \text{ HRMS (ESI) calcd for C}_{16}\text{H}_{24}\text{NaO}_2\text{S (M+Na)}^+ 303.1389, \text{ found 303.1387.} \]

![Diagram 3g](image)

**Compound 3g** (Scheme 2, 92% yield, 97% ee). The ee was measured by HPLC (Chiralpak IC column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, \(t_1 = 55.3 \text{ min (R)}, t_2 = 66.8 \text{ min (S)}\); \([\alpha]_{D}^{20} = -15 (c 1.01, \text{CHCl}_3)\) for 97% ee \((R)\). \(^1\text{H NMR (CDCl}_3) \delta -0.02–0.09 (m, 2H), 0.35–0.40 (m, 1H), 0.42–0.49 (m, 1H), 0.60–0.69 (m, 1H), 1.47–1.53 (m, 1H), 2.44 (s, 3H), 2.89 (dd, \(J = 13.6, 6.8 \text{ Hz}, 1H\)), 2.91 (dd, \(J = 13.6, 5.5 \text{ Hz}, 1H\)), 3.12 (dd, \(J = 14.3, 4.1 \text{ Hz}, 1H\)), 3.20 (dd, \(J = 14.3, 8.2 \text{ Hz}, 1H\)), 7.17–7.22 (m, 3H), 7.24–7.27 (m, 2H), 7.33 (d, \(J = 8.1 \text{ Hz}, 2H\)), 7.76 (d, \(J = 8.1 \text{ Hz}, 2H\)); \(^{13}\text{C NMR (CDCl}_3) \delta 4.5, 5.7, 16.0, 21.6, 39.7, 40.9, 59.7, 126.1, 127.7, 128.1, 129.8, 132.3, 138.6, 144.4. \text{ HRMS (ESI) calcd for C}_{16}\text{H}_{22}\text{NaO}_2\text{S (M+Na)}^+ 337.1233, \text{ found 337.1232.} \]
**Compound 3h** (Scheme 2, 80% yield, 98% ee). The ee was measured by HPLC (Chiralpak AS-H column, hexane/2-propanol = 90:10, flow 1.0 mL/min, 254 nm, \( t_1 = 5.6 \) min (S), \( t_2 = 6.7 \) min (R); \([\alpha]_D^{20} +8 \) (c 1.36, CHCl\(_3\)) for 98% ee (S). \(^1\)H NMR (CDCl\(_3\)) \( \delta \) 0.21–0.26 (m, 1H), 0.49–0.56 (m, 2H), 0.72–0.78 (m, 1H), 1.20–1.27 (m, 1H), 2.32 (s, 6H), 2.73 (dt, \( J = 9.6, 6.8 \) Hz, 1H), 3.84–3.92 (m, 2H), 7.02–7.07 (m, 3H), 7.25–7.31 (m, 3H), 7.33–7.78 (m, 2H); \(^{13}\)C NMR (CDCl\(_3\)) \( \delta \) 4.6, 6.2, 17.2, 17.6, 46.4, 58.7, 126.7, 127.2, 127.5, 128.7, 129.2, 132.1, 141.8, 146.8. HRMS (ESI) calcd for C\(_{19}\)H\(_{22}\)NaO\(_3\)S (M+Na)\(^+\) 353.1182, found 353.1174.

**Compound 3i** (Scheme 2, 86% yield, 92% ee). The ee was measured by HPLC (Chiralpak AS-H column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, \( t_1 = 30.4 \) min (S), \( t_2 = 36.8 \) min (R); \([\alpha]_D^{20} +27 \) (c 1.00, CHCl\(_3\)) for 92% ee (S). \(^1\)H NMR (CDCl\(_3\)) \( \delta \) 0.19–0.23 (m, 1H), 0.40–0.45 (m, 1H), 0.47–0.53 (m, 1H), 0.67–0.73 (m, 1H), 1.12–1.18 (m, 1H), 1.14 (t, \( J = 7.0 \) Hz, 3H), 2.50 (dt, \( J = 9.5, 7.8 \) Hz, 1H), 3.58 (d, \( J = 7.8 \) Hz, 2H), 3.95 (dq, \( J = 9.5, 7.0 \) Hz, 1H), 4.03 (dq, \( J = 9.5, 7.0 \) Hz, 2H), 7.25 (d, \( J = 7.5 \) Hz, 2H), 7.28 (t, \( J = 7.5 \) Hz, 1H), 7.35 (t, \( J = 7.5 \) Hz, 2H); \(^{13}\)C NMR (CDCl\(_3\)) \( \delta \) 4.7, 5.9, 14.7, 17.5, 46.5, 56.5, 65.8, 127.1, 127.5, 128.6, 142.0. HRMS (ESI) calcd for C\(_{13}\)H\(_{18}\)NaO\(_3\)S (M+Na)\(^+\) 277.0869, found 277.0866.

**Compound 3j** (Scheme 2, 96% yield, 98% ee). The ee was measured by HPLC (Chiralpak AD-H column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, \( t_1 = 24.2 \) min (R), \( t_2 = 27.6 \) min (S)); \([\alpha]_D^{20} +35 \) (c 0.95, CHCl\(_3\)) for 98% ee (S). \(^1\)H NMR (CDCl\(_3\)) \( \delta \) 0.37–0.49 (m, 2H), 0.63–0.70 (m, 1H), 1.05–1.12 (m, 1H), 2.47 (dt, \( J = 8.9, 5.5 \) Hz, 1H), 2.92–3.04 (m, 4H), 3.39 (dd, \( J = 14.3, 5.5 \) Hz, 1H), 3.44 (dd, \( J = 14.3, 8.9 \) Hz, 1H), 3.49–3.56 (m, 4H), 7.22 (d, \( J = 7.5 \) Hz, 2H), 7.27 (t, \( J = 7.5 \) Hz, 1H), 7.35 (t, \( J = 7.5 \) Hz, 2H); \(^{13}\)C NMR (CDCl\(_3\)) \( \delta \)
4.7, 5.9, 18.0, 45.3, 46.2, 55.4, 66.4, 127.1, 127.4, 128.7, 142.5. HRMS (ESI) calcd for 
C_{12}H_{27}NNaO_5S (M+Na)^+ 318.1134, found 318.1136.

**Compound 5a** (Scheme 3, 80% yield, 84% ee). The ee was measured by HPLC (Chiralpak IC column, hexane/2-propanol = 95:5, flow 0.5 mL/min, 254 nm, t_1 = 11.5 min (R), t_2 = 12.6 min (S)); [α]_{D}^{20} +37 (c 0.90, CHCl_3) for 84% ee (R). H NMR (CDCl_3) δ 0.16–0.20 (m, 1H), 0.22–0.27 (m, 1H), 0.41–0.46 (m, 1H), 0.52–0.58 (m, 1H), 1.08–1.15 (m, 1H), 2.66 (ddd, J = 9.6, 7.5, 6.8 Hz, 1H), 3.42 (dd, J = 16.3, 6.8 Hz, 1H), 3.46 (dd, J = 16.3, 7.5 Hz, 1H), 7.18–7.22 (m, 1H), 7.28–7.32 (m, 4H), 7.44 (d, J = 7.5 Hz, 2H), 7.54 (t, J = 7.5 Hz, 1H), 7.94 (t, J = 7.5 Hz, 2H); C NMR (CDCl_3) δ 4.3, 5.5, 17.6, 45.7, 46.4, 126.3, 127.4, 128.1, 128.5, 132.8, 137.4, 144.8, 199.2. HRMS (ESI) calcd for C_{18}H_{18}NaO (M+Na)^+ 273.1250, found 273.1248.

**Compound 5b** (Scheme 3, 89% yield, 84% ee). The ee was measured by HPLC (Chiralpak IC column, hexane/2-propanol = 95:5, flow 0.5 mL/min, 254 nm, t_1 = 10.9 min (R), t_2 = 11.6 min (S)); [α]_{D}^{20} +46 (c 0.96, CHCl_3) for 84% ee (R). H NMR (CDCl_3) δ 0.13–0.18 (m, 1H), 0.22–0.28 (m, 1H), 0.41–0.47 (m, 1H), 0.52–0.58 (m, 1H), 1.03–1.10 (m, 1H), 2.62 (ddd, J = 9.5, 7.5, 6.1 Hz, 1H), 3.40 (dd, J = 16.3, 6.1 Hz, 1H), 3.43 (dd, J = 16.3, 7.5 Hz, 1H), 7.21 (d, J = 8.8 Hz, 2H), 7.25 (d, J = 8.8 Hz, 2H), 7.44 (t, J = 7.5 Hz, 2H), 7.55 (t, J = 7.5 Hz, 1H), 8.79 (d, J = 7.5 Hz, 2H); C NMR (CDCl_3) δ 4.4, 5.6, 17.5, 45.4, 45.7, 128.0, 128.5, 128.6, 128.8, 131.9, 133.0, 137.2, 143.3, 198.8. HRMS (ESI) calcd for C_{18}H_{17}ClNaO (M+Na)^+ 307.0860, found 307.0851.

**Compound 5c** (Scheme 3, 95% yield, 86% ee). The ee was measured by HPLC (Chiralpak 1A column x 2, hexane/2-propanol = 95:5, flow 0.5 mL/min, 254 nm, t_1 = 28.3 min (S), t_2 = 30.2 min (R)); [α]_{D}^{20} +40 (c 0.89, CHCl_3) for 86% ee (R). H NMR (CDCl_3) δ 0.16–0.22 (m,
1H), 0.27–0.33 (m, 1H), 0.44–0.49 (m, 1H), 0.55–0.63 (m, 1H), 1.07–1.14 (m, 1H), 2.67–2.73 (m, 1H), 3.45 (dd, J = 16.3, 6.1 Hz, 1H), 3.49 (dd, J = 16.3, 8.2 Hz, 1H), 7.40 (d, J = 8.1 Hz, 2H), 7.45 (t, J = 8.1 Hz, 2H), 7.53–7.57 (m, 3H), 7.92 (d, J = 8.1 Hz, 2H); 13C NMR (CDCl3) δ 4.5, 5.6, 17.4, 45.2, 46.0, 124.3 (q, JF,C = 273 Hz), 125.3 (q, JF,C = 3 Hz), 127.7, 128.0, 128.5 (q, JF,C = 30 Hz), 128.6, 130.1, 137.1, 149.0, 198.5. HRMS (ESI) calcd for C16H17F3NaO (M+Na)+ 341.1124, found 341.1124.

**Compound 5d** (Scheme 3, 63% yield, 81% ee). The ee of 5d was determined by HPLC analysis of 3-cyclopropyl-3-phenylpropan-1-ol (5d'), which was obtained by treatment of 5d with DIBAL-H (2 equiv) in toluene (69% yield). **Compound 5d**:  [α]20D +28 (c 0.72, CHCl3) for 81% ee (R). 1H NMR (CDCl3) δ 0.13–0.18 (m, 1H), 0.25–0.31 (m, 1H), 0.39–0.45 (m, 1H), 0.55–0.61 (m, 1H), 1.00–1.07 (m, 1H), 1.13–1.19 (m, 3H), 2.34–2.41 (m, 1H), 2.71 (dd, J = 15.0, 8.1 Hz, 1H), 2.77 (dd, J = 15.0, 7.5 Hz, 1H), 4.00–4.10 (m, 2H), 7.19–7.26 (m, 3H), 7.30 (t, J = 7.5 Hz, 2H); 13C NMR (CDCl3) δ 4.0, 5.3, 14.1, 17.2, 41.8, 47.2, 60.2, 126.4, 127.3, 128.3, 144.1, 172.3. HRMS (ESI) calcd for C16H16NaO (M+Na)+ 241.1199, found 241.1199. **Compound 5d'**: The ee was measured by HPLC (Chiralcel OJ-H column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, t1 = 16.9 min (S), t2 = 19.7 min (R); [α]20D +27 (c 0.71, CHCl3) for 81% ee (R). 1H NMR (CDCl3) δ 0.06–0.12 (m, 1H), 0.23–0.29 (m, 1H), 0.36–0.42 (m, 1H), 0.58–0.64 (m, 1H), 0.96–1.04 (m, 1H), 1.19 (brs, 1H), 1.92–2.01 (m, 2H), 2.05–2.12 (m, 1H), 3.56 (dt, J = 10.2, 6.8 Hz, 1H), 3.65 (dt, J = 10.2, 6.1 Hz, 1H), 7.19–7.23 (m, 3H), 7.31 (t, J = 8.1 Hz, 2H); 13C NMR (CDCl3) δ 3.8, 5.5, 17.4, 39.5, 47.6, 61.1, 126.2, 127.5, 128.4, 145.3. HRMS (ESI) calcd for C16H16NaO (M+Na)+ 199.1093, found 199.1096.

**Compound 5e** (Scheme 3, 70% yield, 84% ee). The ee was measured by HPLC (Chiralpak AD-H column x 2, hexane/2-propanol = 100:1, flow 0.5 mL/min, 224 nm, t1 = 15.1 min (S), t2 = 15.5 min (R); [α]20D +17 (c 0.85, CHCl3) for 86% ee (R). 1H NMR (CDCl3) δ 0.18–0.23 (m, 1H), 0.26–0.31 (m, 1H), 0.43–0.50 (m, 1H), 0.58–0.65 (m, 1H), 1.03–1.10 (m, 1H), 2.43 (ddd, J = 8.9, 8.2, 7.5 Hz, 1H), 2.98 (ddd, J = 15.0, 8.2 Hz, 1H), 3.01 (dd, J = 15.0, 7.5 Hz, 1H), 5.68 (sept, J
= 6.1 Hz, 1H), 7.20–7.26 (m, 3H), 7.32 (d, J = 7.5 Hz, 2H); $^{13}$C NMR (CDCl$_3$) δ 4.1, 5.3, 17.2, 40.5, 47.0, 66.3 (sept, $J_{F,C} = 34$ Hz), 120.4 (q, $J_{F,C} = 282$ Hz), 126.9, 127.1, 128.5, 142.7, 169.0. HRMS (APCI) calcd for C$_{13}$H$_{14}$F$_6$O$_2$ (M$^+$) 340.0893, found 340.0887.

![Diagram](5f)

**Compound 5f** (Scheme 3, 99% yield, 81% ee). The ee of 5f was determined by HPLC analysis of 5f', which was obtained by treatment of 5f with DIBAL-H (4 equiv) in toluene, and then, benzoyl chloride (2.4 equiv) in the presence of triethyl amine (2.4 equiv) and 4-dimethylaminopyridine (10 mol %) in dichloromethane (65% yield). **Compound 5f**: [α]$^0_{D}$ +46 (c 0.89, CHCl$_3$) for 81% ee (R). $^1$H NMR (CDCl$_3$) δ 0.13–0.18 (m, 1H), 0.37–0.43 (m, 1H), 0.45–0.52 (m, 2H), 0.80–0.87 (m, 1H), 1.42 (s, 9H), 1.44 (s, 9H), 1.91 (td, $J = 9.5, 5.4$ Hz, 1H), 2.41 (dd, $J = 16.3, 5.4$ Hz, 1H), 2.67 (dd, $J = 16.3, 9.5$ Hz, 1H); $^{13}$C NMR (CDCl$_3$) δ 3.6, 4.0, 13.6, 28.0, 28.0, 37.9, 47.1, 80.2, 80.4, 171.2, 173.6. HRMS (ESI) calcd for C$_{15}$H$_{26}$NaO$_4$ (M+Na)$^+$ 293.1723, found 293.1719. **Compound 5f'**: The ee was measured by HPLC (Chiralpak IC column, hexane/2-propanol = 95:5, flow 0.5 mL/min, 254 nm, $t_1 = 29.9$ min (R), $t_2 = 36.4$ min (S)); [α]$^0_{D}$ –3 (c 0.88, CHCl$_3$) for 81% ee (R). $^1$H NMR (CDCl$_3$) δ 0.20–0.29 (m, 2H), 0.50–0.56 (m, 1H), 0.58–0.64 (m, 1H), 0.69–0.77 (m, 1H), 1.23–1.32 (m, 1H), 2.01 (dq, $J = 14.0, 6.8$ Hz, 1H), 2.13 (dq, $J = 14.0, 6.9$ Hz, 1H), 4.34 (dd, $J = 10.9, 6.8$ Hz, 1H), 4.49 (dt, $J = 10.9, 5.4$ Hz, 1H), 4.50–4.59 (m, 2H), 7.42 (t, $J = 8.1$ Hz, 2H), 7.44 (t, $J = 8.1$ Hz, 2H), 7.54 (t, $J = 8.1$ Hz, 1H), 7.55 (t, $J = 8.1$ Hz, 1H), 8.02 (d, $J = 8.1$ Hz, 2H), 8.05 (d, $J = 8.1$ Hz, 2H); $^{13}$C NMR (CDCl$_3$) δ 3.4, 4.4, 13.1, 31.6, 40.8, 63.1, 68.1, 128.3, 128.4, 129.48, 129.51, 130.3, 132.86, 132.88, 166.5, 166.6. HRMS (ESI) calcd for C$_{21}$H$_{22}$NaO$_4$ (M+Na)$^+$ 361.1410, found 361.1409.

![Diagram](5g)

**Compound 5g** (Scheme 3, 92% yield, 89% ee). The ee was measured by HPLC (Chiralcel OD-H column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, $t_1 = 12.3$ min (S), $t_2 = 14.6$ min (R)); [α]$^0_{D}$ +34 (c 1.10, CHCl$_3$) for 89% ee (R). $^1$H NMR (CDCl$_3$) δ 0.16–0.21 (m, 1H), 0.30–0.37 (m, 1H), 0.48–0.53 (m, 1H), 0.64–0.70 (m, 1H), 1.04–1.12 (m, 1H), 2.34 (s, 3H), 2.69 (dd, $J = 9.6, 8.1, 7.5$ Hz, 1H), 3.42 (dd, $J = 11.6, 8.1$ Hz, 1H), 3.46 (dd, $J = 11.6, 7.5$ Hz, 1H), 7.13 (d, $J = 8.1$ Hz, 2H), 7.16 (d, $J = 8.1$ Hz, 2H); $^{13}$C NMR (CDCl$_3$) δ 3.4, 5.2, 14.4, 21.0, 48.8, 80.9.
127.2, 129.5, 136.6, 137.2. HRMS (ESI) calcd for C_{12}H_{15}NNaO_2 (M+Na)^+ 228.0995, found 228.0989.

**Compound 5h** (Scheme 3, 80% yield, 89% ee). The ee was measured by HPLC (Chiralcel OD-H column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, \( t_1 = 16.5 \text{ min} \) (S), \( t_2 = 19.2 \text{ min} \) (R)); \([\alpha]^{20}_D +39 \) (c 0.99, CHCl_3) for 89% ee (R). \(^1\)H NMR (CDCl_3) \( \delta 0.15–0.21 \) (m, 1H), 0.31–0.38 (m, 1H), 0.48–0.55 (m, 1H), 0.65–0.71 (m, 1H), 1.01–1.19 (m, 1H), 2.69 (ddd, \( J = 10.2, 8.5, 7.1 \text{ Hz}, 1H \)), 4.65 (dd, \( J = 11.9, 8.5 \text{ Hz}, 1H \)), 4.69 (dd, \( J = 11.9, 7.1 \text{ Hz}, 1H \)), 7.18 (d, \( J = 8.9 \text{ Hz}, 2H \)), 7.32 (d, \( J = 8.9 \text{ Hz}, 2H \)); \(^{13}\)C NMR (CDCl_3) \( \delta 3.6, 5.3, 14.3, 48.6, 80.5, 128.7, 129.0, 133.4, 138.2 \). HRMS (ESI) calcd for C_{12}H_{13}ClNNaO_2 (M+Na)^+ 244.0449, found 244.0451.

**Compound 5i** (Scheme 3, 70% yield, 89% ee). The ee was measured by HPLC (Chiralcel OD-H column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, \( t_1 = 18.8 \text{ min} \) (S), \( t_2 = 24.9 \text{ min} \) (R)); \([\alpha]^{20}_D +26 \) (c 1.08, CHCl_3) for 89% ee (R). \(^1\)H NMR (CDCl_3) \( \delta 2.46 \) (s, 3H), \( 3.01–3.06 \text{ (m, 2H)} \), 3.31–3.36 (m, 2H), 7.11 (d, \( J = 7.5 \text{ Hz}, 2H \)), 7.20 (t, \( J = 7.5 \text{ Hz}, 1H \)), 7.26 (t, \( J = 7.5 \text{ Hz}, 2H \)), 7.34 (d, \( J = 8.1 \text{ Hz}, 2H \)), 7.82 (d, \( J = 8.1 \text{ Hz}, 2H \)). HRMS (ESI) calcd for C_{12}H_{13}ClNNaO_3 (M+Na)^+ 244.0944, found 244.0944.

**Compound 9** [CAS: 19719-87-0] (eq 2, 26% yield). \(^1\)H NMR (CDCl_3) \( \delta 2.46 \) (s, 3H), 3.01–3.06 (m, 2H), 3.31–3.36 (m, 2H), 7.11 (d, \( J = 7.5 \text{ Hz}, 2H \)), 7.20 (t, \( J = 7.5 \text{ Hz}, 1H \)), 7.26 (t, \( J = 7.5 \text{ Hz}, 2H \)), 7.34 (d, \( J = 8.1 \text{ Hz}, 2H \)), 7.82 (d, \( J = 8.1 \text{ Hz}, 2H \)).
Compound 11 (eq 3, 92% yield, 25% ee). The ee was measured by HPLC (Chiralpak IA column, hexane/2-propanol = 100:1, flow 0.5 mL/min, 254 nm, \( t_1 = 14.6 \text{ min (major)} \), \( t_2 = 16.0 \text{ min (minor)} \)). \(^1\)H NMR (CDCl\(_3\)) \( \delta \) 0.76–0.81 (m, 1H), 0.88–0.92 (m, 1H), 1.01–1.09 (m, 1H), 1.62–1.72 (m, 3H), 2.15 (s, 3H), 2.59 (t, \( J = 7.5 \text{ Hz, 2H} \)), 7.02 (d, \( J = 7.8 \text{ Hz, 2H} \)), 7.13 (t, \( J = 7.8 \text{ Hz, 1H} \)), 7.24 (t, \( J = 7.8 \text{ Hz, 2H} \)); \(^{13}\)C NMR (CDCl\(_3\)) \( \delta \) 16.1, 23.0, 23.3, 28.5, 30.1, 43.3, 125.3, 125.6, 128.2, 143.4, 208.7. HRMS (ESI) calcd for C\(_{13}\)H\(_{16}\)NaO (M+Na)\(^+\) 211.1093, found 211.1090.

Compound 11-\(d_2\) (eq 4, 50% yield). \(^1\)H NMR (CDCl\(_3\)) \( \delta \) 1.03 (dd, \( J = 11.6, 6.8 \text{ Hz, 1H} \)), 1.61–1.71 (m, 3H), 2.15 (s, 3H), 2.58 (t, \( J = 7.2 \text{ Hz, 2H} \)), 7.02 (d, \( J = 7.8 \text{ Hz, 2H} \)), 7.13 (t, \( J = 7.8 \text{ Hz, 1H} \)), 7.24 (t, \( J = 7.8 \text{ Hz, 2H} \)). HRMS (ESI) calcd for C\(_{13}\)H\(_{14}\)D\(_2\)NaO (M+Na)\(^+\) 213.1219, found 213.1222.
11. X-Ray data of 3a

A colorless crystal of 3a suitable for X-ray crystallographic analysis was obtained by recrystallization from hexane/2-propanol. The ORTEP drawing of 3a is shown in Figure S1. The crystal structure has been deposited at the Cambridge Crystallographic Centre (deposition number: CCDC 1047801). The data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif. X-Ray data were collected on a Rigaku XtaLAB P200 using a graphite monochromater with Cu-Kα radiation (λ = 1.54187 Å) at 93 K. The structure was solved by direct method (SHELXS-97) and refined with full-matrix least-square technique (SHELXL-97). The data for 3a is summarized in Table S1.

Figure S1. ORTEP illustration of 3a drawn at 50% probability level.

---

17 G. M. Sheldrick, Program for the solution and refinement of crystal structures, University of Göttingen, Göttingen, Germany, 1997.
<table>
<thead>
<tr>
<th><strong>Table S1.</strong> Crystal data and structure refinement for 3a</th>
</tr>
</thead>
<tbody>
<tr>
<td>Empirical formula</td>
</tr>
<tr>
<td>Formula weight</td>
</tr>
<tr>
<td>Temperature</td>
</tr>
<tr>
<td>Crystal system</td>
</tr>
<tr>
<td>Space group</td>
</tr>
<tr>
<td>Unit cell dimensions</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>Volume</td>
</tr>
<tr>
<td>Z</td>
</tr>
<tr>
<td>Density (calculated) [Mg/m³]</td>
</tr>
<tr>
<td>μ (mm⁻¹)</td>
</tr>
<tr>
<td>F(000)</td>
</tr>
<tr>
<td>Reflections collected</td>
</tr>
<tr>
<td>Independent reflections</td>
</tr>
<tr>
<td>Completeness to θ (%)</td>
</tr>
<tr>
<td>Goodness-of-fit</td>
</tr>
<tr>
<td>R₁ [I&gt;2σ (I)]</td>
</tr>
<tr>
<td>wR² (all data)</td>
</tr>
<tr>
<td>Flack parameter</td>
</tr>
<tr>
<td>Largest diff. peak and hole [e⁻/Å³]</td>
</tr>
</tbody>
</table>
12. $^1$H and $^{13}$C NMR spectra and HPLC charts
[RhCl((S,S)-L3)]_2

X: parts per Million: Proton

[Rh(OH)((S,S)-L3)]_2

X: parts per Million: Proton
<table>
<thead>
<tr>
<th>Part Per Million</th>
<th>Proton</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>0.1</td>
<td>0.1</td>
</tr>
<tr>
<td>0.2</td>
<td>0.2</td>
</tr>
<tr>
<td>0.3</td>
<td>0.3</td>
</tr>
<tr>
<td>0.4</td>
<td>0.4</td>
</tr>
<tr>
<td>0.5</td>
<td>0.5</td>
</tr>
<tr>
<td>0.6</td>
<td>0.6</td>
</tr>
<tr>
<td>0.7</td>
<td>0.7</td>
</tr>
<tr>
<td>0.8</td>
<td>0.8</td>
</tr>
<tr>
<td>0.9</td>
<td>0.9</td>
</tr>
<tr>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>1.1</td>
<td>1.1</td>
</tr>
<tr>
<td>1.2</td>
<td>1.2</td>
</tr>
<tr>
<td>1.3</td>
<td>1.3</td>
</tr>
<tr>
<td>1.4</td>
<td>1.4</td>
</tr>
</tbody>
</table>

X: parts per Million
Proton

**Graph:**

- **Sweep:** Carbon13
- **Resolution:** 1.43409672 Hz
- **Points:** 32768
- **Offset:** 100.0 ppm
- **Frequency:** 149.40429612 MHz
- **Acquisition Duration:** 0.69730304 s
- **Sample:** CHLOROFORM-D
- **Experiment:** carbon.jxp
- **Author:** delta
- **Filename:** 4707-D take_Carbon-1-3.jdf
- **Date:** 2-AUG-2014 19:53:42
- **Revision Time:** 2-AUG-2014 19:50:18
- **Creation Time:** 2-AUG-2014 19:35:53

**Graph:**

- **Sweep:** Proton
- **Resolution:** 0.67998169 Hz
- **Points:** 16384
- **Offset:** 5 ppm
- **Frequency:** 594.17058168 MHz
- **Domain:** Proton
- **Acquisition Duration:** 1.47062784 s
- **Sample:** CHLOROFORM-D
- **Experiment:** proton.jxp
- **Author:** delta
- **Filename:** 4707-D take_Proton-1-9.jdf
- **Date:** 2-AUG-2014 19:43:49
- **Revision Time:** 2-AUG-2014 19:42:51
- **Creation Time:** 2-AUG-2014 19:33:08

**Diagram:**

- **Compound:** 3a
- **Spectrometer:** DELTA2_NMR
- **Site:** JNM-ECA600
- **Dimensions:** X
- **Units:** ppm
- **Title:** Carbon13
- **Size:** 26214
- **Format:** 1D COMPLEX
- **Comment:** single_pulse decoupled gated
- **Current Time:** 2-AUG-2014 19:43:49
- **Revision Time:** 2-AUG-2014 19:42:51
- **Creation Time:** 2-AUG-2014 19:33:08
- **Solvent:** CHLOROFORM-D
- **Sample Id:** 4707-D take
- **Experiment:** carbon.jxp
- **Author:** delta
- **Filename:** 4707-D take_Carbon-1-3.jdf

**Diagram:**

- **Compound:** 3a
- **Spectrometer:** DELTA2_NMR
- **Site:** JNM-ECA600
- **Dimensions:** X
- **Units:** ppm
- **Title:** Proton
- **Size:** 13107
- **Format:** 1D COMPLEX
- **Comment:** single_pulse
- **Current Time:** 2-AUG-2014 19:43:49
- **Revision Time:** 2-AUG-2014 19:42:51
- **Creation Time:** 2-AUG-2014 19:33:08
- **Solvent:** CHLOROFORM-D
- **Sample Id:** 4707-D take
- **Experiment:** proton.jxp
- **Author:** delta
- **Filename:** 4707-D take_Proton-1-9.jdf

**Diagram:**

- **Compound:** 3a
- **Spectrometer:** DELTA2_NMR
- **Site:** JNM-ECA600
- **Dimensions:** X
- **Units:** ppm
- **Title:** Proton
- **Size:** 13107
- **Format:** 1D COMPLEX
- **Comment:** single_pulse
- **Current Time:** 2-AUG-2014 19:43:49
- **Revision Time:** 2-AUG-2014 19:42:51
- **Creation Time:** 2-AUG-2014 19:33:08
- **Solvent:** CHLOROFORM-D
- **Sample Id:** 4707-D take
- **Experiment:** proton.jxp
- **Author:** delta
- **Filename:** 4707-D take_Proton-1-9.jdf
<table>
<thead>
<tr>
<th>X</th>
<th>0</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
<th>9</th>
</tr>
</thead>
</table>

**Comment:** single pulse

**Spectrometer:** DELTA2_NMR

**Dimensions:** X

**Dim_Units:** parts per million

**Dim_Title:** Carbon13

**Dim_Size:** 26214

**Data_Format:** 1D COMPLEX

**Current_Time:** 5-AUG-2014 00:56:30

**Revision_Time:** 5-AUG-2014 00:55:46

**Creation_Time:** 5-AUG-2014 00:42:35

**Author:** delta

**Filename:** 4713-B_Carbon-2-3.jdf

**Solvent:** CHLOROFORM-D

**Sample_Id:** 4713-B

**Experiment:** carbon.jxp

---

**Repetition_Time:** 6.47062784 [s]

**Initial_Wait:** 1 [s]

**Dante_Presat:** FALSE

**Irr_Mode:** Off

**X_Pulse:** 6.9 [μs]

**X_Atn:** 3 [dB]

**X_Angle:** 45 [deg]

**X_Acq_Time:** 1.47062784 [s]

**X_90_Width:** 13.8 [μs]

**Temp_Get:** 25.9 [°C]

**Recvr_Gain:** 36

**Relaxation_Delay:** 2 [s]

**Clipped:** FALSE

**Tri_Points:** 16384

**Tri_Offset:** 5 [ppm]

**Tri_Freq:** 594.17058168 [MHz]

**Tri_Domain:** Proton

**Irr_Offset:** 5 [ppm]

**Irr_Freq:** 594.17058168 [MHz]

**Irr_Domain:** Proton

**X_Sweep_Clipped:** 8.91265597 [kHz]

**X_Sweep:** 11.14081996 [kHz]

**X_Points:** 13107

**X_Offset:** 5 [ppm]

**X_Freq:** 594.17058168 [MHz]

**X_Domain:** 1H

**X_Acq_Duration:** 1.47062784 [s]

**Field_Strength:** 13.95540559 [T] (590 [MHz])

**Spectrometer:** DELTA2_NMR

**Dimensions:** X

**Dim_Units:** parts per million

**Dim_Title:** Proton

**Dim_Size:** 13107

**Data_Format:** 1D COMPLEX

**Comment:** single pulse

**Current_Time:** 5-AUG-2014 00:48:02

**Creation_Time:** 5-AUG-2014 00:39:50

**Author:** delta

**Filename:** 4713-B_Proton-2-6.jdf

**Solvent:** CHLOROFORM-D

**Sample_Id:** 4713-B

**Experiment:** proton.jxp

---

**Repetition_Time:** 2.69730304 [s]

**Noe_Time:** 2 [s]

**Noe:** TRUE

**Initial_Wait:** 1 [s]

**Decoupling:** TRUE

**Irr_Pwidth:** 76 [μs]

**Irr_Noise:** WALTZ

**Irr_Atn_No:** 17.8 [dB]

**Irr_Atn_Dec:** 17.8 [dB]

**X_Pulse:** 3 [μs]

**X_Atn:** 6 [dB]

**X_Angle:** 30 [deg]

**X_90_Width:** 9 [μs]

**Temp_Get:** 26.8 [°C]

**Recvr_Gain:** 60

**Relaxation_Delay:** 2 [s]

**Total_Scans:** 256

**Scans:** 256

**Clipped:** TRUE

**Irr_Offset:** 5 [ppm]

**Irr_Freq:** 594.17058168 [MHz]

**Irr_Domain:** Proton

**X_Sweep:** 46.9924812 [kHz]

**X_Resolution:** 1.43409672 [Hz]

**X_Prescans:** 4

**X_Points:** 32768

**X_Offset:** 100.0 [ppm]

**X_Freq:** 149.40429612 [MHz]

**X_Domain:** 13C

**Field_Strength:** 13.95540559 [T] (590 [MHz])

**Spectrometer:** DELTA2_NMR

**Site:** JNM-ECA600

**Dimensions:** X

**Dim_Units:** parts per million

**Dim_Title:** Carbon13

**Dim_Size:** 26214

**Data_Format:** 1D COMPLEX

**Comment:** single pulse decoupled gate

**Current_Time:** 5-AUG-2014 00:56:30

**Revision_Time:** 5-AUG-2014 00:55:46

**Creation_Time:** 5-AUG-2014 00:42:35

**Author:** delta

**Filename:** 4713-B_Carbon-2-3.jdf

**Solvent:** CHLOROFORM-D

**Sample_Id:** 4713-B

**Experiment:** carbon.jxp

---

**Repetition_Time:** 6.47062784 [s]

**Initial_Wait:** 1 [s]

**Dante_Presat:** FALSE

**Irr_Mode:** Off

**X_Pulse:** 6.9 [μs]

**X_Atn:** 3 [dB]

**X_Angle:** 45 [deg]

**X_Acq_Time:** 1.47062784 [s]

**X_90_Width:** 13.8 [μs]

**Temp_Get:** 25.9 [°C]

**Recvr_Gain:** 36

**Relaxation_Delay:** 5 [s]

**Scans:** 8

**Clipped:** FALSE

**Tri_Offset:** 5 [ppm]

**Tri_Freq:** 594.17058168 [MHz]

**Tri_Domain:** Proton

**Irr_Offset:** 5 [ppm]

**Irr_Freq:** 594.17058168 [MHz]

**Irr_Domain:** Proton

**X_Sweep_Clipped:** 8.91265597 [kHz]

**X_Sweep:** 11.14081996 [kHz]

**X_Points:** 16384

**X_Offset:** 5 [ppm]

**X_Freq:** 594.17058168 [MHz]

**X_Domain:** 1H

**X_Acq_Duration:** 1.47062784 [s]

**Field_Strength:** 13.95540559 [T] (590 [MHz])

**Spectrometer:** DELTA2_NMR

**Dimensions:** X

**Dim_Units:** parts per million

**Dim_Title:** Proton

**Dim_Size:** 13107

**Data_Format:** 1D COMPLEX

**Comment:** single pulse

**Current_Time:** 5-AUG-2014 00:48:02

**Creation_Time:** 5-AUG-2014 00:39:50

**Author:** delta

**Filename:** 4713-B_Proton-2-6.jdf

**Solvent:** CHLOROFORM-D

**Sample_Id:** 4713-B

**Experiment:** proton.jxp

---

**Repetition_Time:** 2.69730304 [s]

**Noe_Time:** 2 [s]

**Noe:** TRUE

**Initial_Wait:** 1 [s]

**Decoupling:** TRUE

**Irr_Pwidth:** 76 [μs]

**Irr_Noise:** WALTZ

**Irr_Atn_No:** 17.8 [dB]

**Irr_Atn_Dec:** 17.8 [dB]

**X_Pulse:** 3 [μs]

**X_Atn:** 6 [dB]

**X_Angle:** 30 [deg]

**X_90_Width:** 9 [μs]

**Temp_Get:** 26.8 [°C]

**Recvr_Gain:** 60

**Relaxation_Delay:** 2 [s]

**Total_Scans:** 256

**Scans:** 256

**Clipped:** TRUE

**Irr_Offset:** 5 [ppm]

**Irr_Freq:** 594.17058168 [MHz]

**Irr_Domain:** Proton

**X_Sweep_CLipped:** 8.91265597 [kHz]

**X_Sweep:** 11.14081996 [kHz]

**X_Points:** 16384

**X_Offset:** 5 [ppm]

**X_Freq:** 594.17058168 [MHz]

**X_Domain:** 1H

**X_Acq_Duration:** 1.47062784 [s]

**Field_Strength:** 13.95540559 [T] (590 [MHz])

**Spectrometer:** DELTA2_NMR

**Dimensions:** X

**Dim_Units:** parts per million

**Dim_Title:** Proton

**Dim_Size:** 13107

**Data_Format:** 1D COMPLEX

**Comment:** single pulse

**Current_Time:** 5-AUG-2014 00:48:02

**Creation_Time:** 5-AUG-2014 00:39:50

**Author:** delta

**Filename:** 4713-B_Proton-2-6.jdf

**Solvent:** CHLOROFORM-D

**Sample_Id:** 4713-B

**Experiment:** proton.jxp

---
UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>23.017</td>
<td>8103366</td>
<td>49.900</td>
<td>268107</td>
</tr>
<tr>
<td>2</td>
<td>25.390</td>
<td>8135925</td>
<td>50.100</td>
<td>246945</td>
</tr>
</tbody>
</table>

Totals: 16239291   100.000   515052

UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>22.717</td>
<td>232349</td>
<td>3.333</td>
<td>8949</td>
</tr>
<tr>
<td>2</td>
<td>24.913</td>
<td>6739243</td>
<td>96.667</td>
<td>212014</td>
</tr>
</tbody>
</table>

Totals: 6971592   100.000   220963
### UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>25.602</td>
<td>5495009</td>
<td>49.772</td>
<td>171405</td>
</tr>
<tr>
<td>2</td>
<td>27.502</td>
<td>5545415</td>
<td>50.228</td>
<td>161382</td>
</tr>
</tbody>
</table>

**Totals**

<p>| | | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>11040424</td>
<td>100.000</td>
<td>332787</td>
<td></td>
</tr>
</tbody>
</table>

### UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>25.693</td>
<td>605241</td>
<td>3.658</td>
<td>20100</td>
</tr>
<tr>
<td>2</td>
<td>27.455</td>
<td>15940342</td>
<td>96.342</td>
<td>437905</td>
</tr>
</tbody>
</table>

**Totals**

<p>| | | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>16545583</td>
<td>100.000</td>
<td>458005</td>
<td></td>
</tr>
</tbody>
</table>
### UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>25.128</td>
<td>5957255</td>
<td>49.834</td>
<td>173666</td>
</tr>
<tr>
<td>2</td>
<td>32.241</td>
<td>5996836</td>
<td>50.166</td>
<td>116703</td>
</tr>
</tbody>
</table>

**Totals**

|          | 11954091 | 100.000 | 290369 |

### UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>25.682</td>
<td>172016</td>
<td>2.086</td>
<td>5011</td>
</tr>
<tr>
<td>2</td>
<td>32.601</td>
<td>8074724</td>
<td>97.914</td>
<td>152785</td>
</tr>
</tbody>
</table>

**Totals**

|          | 8246740 | 100.000 | 157796 |

---

S–27
### UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>35.161</td>
<td>1832656</td>
<td>50.345</td>
<td>30123</td>
</tr>
<tr>
<td>2</td>
<td>49.262</td>
<td>1807517</td>
<td>49.655</td>
<td>22182</td>
</tr>
</tbody>
</table>

**Totals**

<p>| | | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>3640173</td>
<td>100.000</td>
<td>52305</td>
<td></td>
</tr>
</tbody>
</table>

### UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>33.869</td>
<td>11337396</td>
<td>91.331</td>
<td>115456</td>
</tr>
<tr>
<td>2</td>
<td>48.945</td>
<td>1076075</td>
<td>8.669</td>
<td>14311</td>
</tr>
</tbody>
</table>

**Totals**

<p>| | | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>12413471</td>
<td>100.000</td>
<td>129767</td>
<td></td>
</tr>
</tbody>
</table>
### UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>44.101</td>
<td>1508893</td>
<td>49.149</td>
<td>31275</td>
</tr>
<tr>
<td>2</td>
<td>45.960</td>
<td>1561116</td>
<td>50.851</td>
<td>27145</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3070009</td>
<td>100.000</td>
<td>58420</td>
</tr>
</tbody>
</table>

### UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>44.054</td>
<td>1902192</td>
<td>98.335</td>
<td>35565</td>
</tr>
<tr>
<td>2</td>
<td>46.265</td>
<td>32209</td>
<td>1.665</td>
<td>604</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1934401</td>
<td>100.000</td>
<td>36169</td>
</tr>
<tr>
<td>Pk #</td>
<td>Retention Time</td>
<td>Area</td>
<td>Area Percent</td>
<td>Height</td>
</tr>
<tr>
<td>------</td>
<td>----------------</td>
<td>----------</td>
<td>--------------</td>
<td>--------</td>
</tr>
<tr>
<td>1</td>
<td>54.836</td>
<td>3136472</td>
<td>49.821</td>
<td>46923</td>
</tr>
<tr>
<td>2</td>
<td>65.619</td>
<td>3158968</td>
<td>50.179</td>
<td>37980</td>
</tr>
<tr>
<td>Totals</td>
<td></td>
<td>6295440</td>
<td>100.000</td>
<td>84903</td>
</tr>
</tbody>
</table>

**UV Results**

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>55.313</td>
<td>969692</td>
<td>98.554</td>
<td>15267</td>
</tr>
<tr>
<td>2</td>
<td>66.787</td>
<td>14227</td>
<td>1.446</td>
<td>200</td>
</tr>
<tr>
<td>Totals</td>
<td></td>
<td>983919</td>
<td>100.000</td>
<td>15467</td>
</tr>
</tbody>
</table>
3h

**UV Results**

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.670</td>
<td>951693</td>
<td>50.486</td>
<td>111293</td>
</tr>
<tr>
<td>2</td>
<td>6.800</td>
<td>933379</td>
<td>49.514</td>
<td>85634</td>
</tr>
<tr>
<td><strong>Totals</strong></td>
<td></td>
<td><strong>1885072</strong></td>
<td><strong>100.000</strong></td>
<td><strong>196927</strong></td>
</tr>
</tbody>
</table>

**UV Results**

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.561</td>
<td>2341857</td>
<td>99.115</td>
<td>252591</td>
</tr>
<tr>
<td>2</td>
<td>6.735</td>
<td>20909</td>
<td>0.885</td>
<td>2127</td>
</tr>
<tr>
<td><strong>Totals</strong></td>
<td></td>
<td><strong>2362766</strong></td>
<td><strong>100.000</strong></td>
<td><strong>254718</strong></td>
</tr>
</tbody>
</table>
UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10.843</td>
<td>9056769</td>
<td>49.768</td>
<td>674647</td>
</tr>
<tr>
<td>2</td>
<td>11.703</td>
<td>9141098</td>
<td>50.232</td>
<td>640237</td>
</tr>
</tbody>
</table>

Totals | 18197867 | 100.000 | 1314884 |

UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>11.473</td>
<td>8946687</td>
<td>91.871</td>
<td>616346</td>
</tr>
<tr>
<td>2</td>
<td>12.569</td>
<td>791587</td>
<td>8.129</td>
<td>53970</td>
</tr>
</tbody>
</table>

Totals | 9738274 | 100.000 | 670316 |
\[ \text{UV-970 Results} \]

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>28.295</td>
<td>948816</td>
<td>6.968</td>
<td>37132</td>
</tr>
<tr>
<td>2</td>
<td>30.173</td>
<td>12667533</td>
<td>93.032</td>
<td>434530</td>
</tr>
<tr>
<td><strong>Totals</strong></td>
<td></td>
<td><strong>13616349</strong></td>
<td><strong>100.000</strong></td>
<td><strong>471662</strong></td>
</tr>
</tbody>
</table>

\[ \text{UV-970 Results} \]

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>28.115</td>
<td>9913308</td>
<td>49.701</td>
<td>369947</td>
</tr>
<tr>
<td>2</td>
<td>30.061</td>
<td>10032413</td>
<td>50.299</td>
<td>346556</td>
</tr>
<tr>
<td><strong>Totals</strong></td>
<td></td>
<td><strong>19945721</strong></td>
<td><strong>100.000</strong></td>
<td><strong>716503</strong></td>
</tr>
</tbody>
</table>
UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>30.256</td>
<td>1551683</td>
<td>49.515</td>
<td>93560</td>
</tr>
<tr>
<td>2</td>
<td>36.303</td>
<td>1582063</td>
<td>50.485</td>
<td>26422</td>
</tr>
</tbody>
</table>

Totals: 3133746 100.000 119982

UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>29.853</td>
<td>18016172</td>
<td>90.447</td>
<td>401091</td>
</tr>
<tr>
<td>2</td>
<td>36.449</td>
<td>1902913</td>
<td>9.553</td>
<td>41211</td>
</tr>
</tbody>
</table>

Totals: 19919085 100.000 442302
UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>12.256</td>
<td>1603227</td>
<td>51.247</td>
<td>92454</td>
</tr>
<tr>
<td>2</td>
<td>14.497</td>
<td>1525202</td>
<td>48.753</td>
<td>80250</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3128429</td>
<td>100.000</td>
<td>172704</td>
</tr>
</tbody>
</table>

UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>12.339</td>
<td>177895</td>
<td>5.487</td>
<td>9683</td>
</tr>
<tr>
<td>2</td>
<td>14.567</td>
<td>3064111</td>
<td>94.513</td>
<td>156872</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3242006</td>
<td>100.000</td>
<td>166555</td>
</tr>
</tbody>
</table>
UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>16.379</td>
<td>2416399</td>
<td>49.828</td>
<td>104918</td>
</tr>
<tr>
<td>2</td>
<td>19.100</td>
<td>2433098</td>
<td>50.172</td>
<td>92734</td>
</tr>
<tr>
<td></td>
<td></td>
<td><strong>4849497</strong></td>
<td><strong>100.000</strong></td>
<td><strong>197652</strong></td>
</tr>
</tbody>
</table>

UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>16.451</td>
<td>369533</td>
<td>5.657</td>
<td>15955</td>
</tr>
<tr>
<td>2</td>
<td>19.180</td>
<td>6163015</td>
<td>94.343</td>
<td>225778</td>
</tr>
<tr>
<td></td>
<td></td>
<td><strong>6532548</strong></td>
<td><strong>100.000</strong></td>
<td><strong>241733</strong></td>
</tr>
</tbody>
</table>
O$_2$N\textbullet
\begin{align*}
\text{5i} & \quad \text{NM}e
\end{align*}

### UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>17.821</td>
<td>9223870</td>
<td>49.976</td>
<td>311518</td>
</tr>
<tr>
<td>2</td>
<td>23.467</td>
<td>9232810</td>
<td>50.024</td>
<td>291138</td>
</tr>
<tr>
<td><strong>Totals</strong></td>
<td><strong>18456680</strong></td>
<td>100.000</td>
<td></td>
<td>602656</td>
</tr>
</tbody>
</table>

### UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>18.807</td>
<td>627933</td>
<td>5.411</td>
<td>23929</td>
</tr>
<tr>
<td>2</td>
<td>24.923</td>
<td>10977273</td>
<td>94.589</td>
<td>315078</td>
</tr>
<tr>
<td><strong>Totals</strong></td>
<td><strong>11605206</strong></td>
<td>100.000</td>
<td></td>
<td>339007</td>
</tr>
</tbody>
</table>
### UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>14.609</td>
<td>981738</td>
<td>50.765</td>
<td>37228</td>
</tr>
<tr>
<td>2</td>
<td>15.694</td>
<td>952157</td>
<td>49.235</td>
<td>37005</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Totals</strong></td>
<td></td>
<td><strong>1933895</strong></td>
<td><strong>100.000</strong></td>
<td><strong>74233</strong></td>
</tr>
</tbody>
</table>

### UV Results

<table>
<thead>
<tr>
<th>Pk #</th>
<th>Retention Time</th>
<th>Area</th>
<th>Area Percent</th>
<th>Height</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>14.641</td>
<td>1894804</td>
<td>62.320</td>
<td>61893</td>
</tr>
<tr>
<td>2</td>
<td>16.007</td>
<td>1145624</td>
<td>37.680</td>
<td>19261</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Totals</strong></td>
<td></td>
<td><strong>3040428</strong></td>
<td><strong>100.000</strong></td>
<td><strong>81154</strong></td>
</tr>
<tr>
<td>Parts per Million</td>
<td>Proton</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>------------------</td>
<td>--------</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7.2600</td>
<td>7.2508</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7.2474</td>
<td>7.2383</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7.2280</td>
<td>7.2245</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7.1421</td>
<td>7.1398</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7.1295</td>
<td>7.1169</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7.1147</td>
<td>7.0311</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7.0288</td>
<td>7.0218</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7.0174</td>
<td>7.0103</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.5965</td>
<td>2.5839</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.5724</td>
<td>2.1467</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.6581</td>
<td>1.6329</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.0412</td>
<td>1.0298</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.0218</td>
<td>1.0103</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Filename**: 4Y17-B take_Proton-2-4.jdf

**Author**: delta

**Experiment**: proton.jxp

**Sample_Id**: 4Y17-B take

**Solvent**: CHLOROFORM-D

**Creation_Time**: 10-DEC-2014 17:29:32

**Revision_Time**: 10-DEC-2014 18:05:42

**Current_Time**: 10-DEC-2014 18:06:09

**Comment**: single_pulse

**Data_Format**: 1D COMPLEX

**Dim_Size**: 13107

**Dim_Title**: Proton

**Dim_Units**: [ppm]

**Site**: JNM-ECA600

**Spectrometer**: DELTA2_NMR

**Field_Strength**: 13.95540559[T] (590[MHz])

**X_Acq_Duration**: 1.47062784[s]

**X_Domain**: 1H

**X_Freq**: 594.17058168[MHz]

**X_Offset**: 5[ppm]

**X_Points**: 16384

**X_Prescans**: 1

**X_Resolution**: 0.67998169[Hz]

**X_Sweep**: 11.14081996[kHz]

**X_Sweep_Clipped**: 8.91265597[kHz]

**Irr_Domain**: Proton

**Irr_Freq**: 594.17058168[MHz]

**Irr_Offset**: 5[ppm]

**Tri_Domain**: Proton

**Tri_Freq**: 594.17058168[MHz]

**Tri_Offset**: 5[ppm]

**Clipped**: FALSE

**Scans**: 24

**Total_Scans**: 24

**Relaxation_Delay**: 5[s]

**Temp_Get**: 24.1[dC]

**X_90_Width**: 13.8[us]

**X_Acq_Time**: 1.47062784[s]

**X_Angle**: 45[deg]

**X_Atn**: 3[dB]

**X_Pulse**: 6.9[us]

**Irr_Mode**: Off

**Tri_Mode**: Off

**Dante_Presat**: FALSE

**Initial_Wait**: 1[s]

**Repetition_Time**: 6.47062784[s]