Electronic Supplementary Information

Ruthenium-catalyzed regio- and site-selective ortho C–H borylation of phenol derivatives

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Table of Contents
S2 General
S3-S7 Preparation of phosphinites
Spectral data
S8-S16 Procedure for C–H borylation of aryl phosphinites
Spectral data
S17-S18 Procedure for C–H borylation of aryl phosphites
Procedure for one-pot C–H borylation of aryl phosphites
S19 Deuterium labeling experiments
S20 References
S21–97 NMR spectra
**General:** All operations were performed under an argon atmosphere. $^1$H, $^{13}$C and $^{31}$P spectra were recorded on an ECS-400 (400 MHz for $^1$H, 128 MHz for $^{11}$B, 100 MHz for $^{13}$C, and 160 MHz for $^{31}$P), an ECX-400 (400 MHz for $^1$H and 160 MHz for $^{31}$P), or an ECZ-500 (500 MHz for $^1$H, 125 MHz for $^{13}$C, and 200 MHz for $^{31}$P) spectrometer in CDCl$_3$, C$_6$D$_6$, acetone-$d_6$, or dmso-$d_6$. Chemical shifts are expressed in parts per million (ppm) downfield from tetramethylsilane ($\delta_H$ 0.00, $\delta_C$ 0.00), BF$_3$•OEt$_2$ ($\delta_B$ 0.00), 85% H$_3$PO$_4$ aq. ($\delta_P$ 0.00), or C$_6$F$_6$ ($\delta_F$ -163.00) and are referenced to residual solvents ($\delta_H$ 7.26 and $\delta_C$ 77.0 for chloroform, $\delta_H$ 7.16 and $\delta_C$ 128.06 for C$_6$H$_6$, $\delta_H$ 7.05 and $\delta_C$ 206.26 for acetone-$d_6$, and $\delta_H$ 2.50 for dmso-$d_6$). IR spectra were recorded on an FT/IR-460 plus (JASCO Co., Ltd.) with an ATR PRO450-S accessory (JASCO Co., Ltd.). High resolution mass spectra (HRMS) were recorded on a BRUKER micrOTOF II and a JEOL JMS-T100 spectrometer. Silica Gel 60 (Kanto Chemical Co., Inc.) was used for flash column chromatography. A Merck Kieselgel 60 F$_{254}$ (0.25 mm thickness, coated on glass 20 x 20 cm$^2$) plate was used for analytical thin layer chromatography (TLC). THF, Et$_2$O, hexanes and toluene were purified by solvent purification system of Glass-Contour. $n$-Octane was purchased from Tokyo Chemical Industry and degassed by freeze-pump-thaw technique and dried over 4A molecular sieves. Benzene-$d_6$ was purchased from ACROS chemicals and dried and degassed by benzophenone ketyl. Chloroform-$d$ was purchased from Kanto Chemicals. Unless otherwise noted, materials were purchased from Sigma-Aldrich Corporation, FUJIFILM Wako Pure Chemical Corporation, Tokyo Chemical Industry Corporation, and Kanto Chemical Corporation.
**General procedure for the synthesis of aryl diphenylphosphinites**

In a 100 mL two-neck flask, a phenol derivative (10.0 mmol) was dissolved in Et₂O (30.0 mL) under an argon atmosphere. The flask was cooled to 0 °C in an ice-bath, and triethylamine (10.0 mmol) and chlorodiphenylphosphine (10.0 mmol) were successively added. The flask was allowed to stand at room temperature and stirred overnight. The solution was filtered through a short pad of alumina and Celite®, and the filtrate was concentrated under reduced pressure to give the product.

1b (colorless oil, 2.0 g, 7.0 mmol, 70%)
The spectral data were in good agreement with the literature values.¹
¹H NMR (CDCl₃, 400 MHz) δ = 7.61-7.56 (m, 4H), 7.42-7.32 (m, 6H), 7.10-6.98 (m, 4H), 2.28 (s, 3H).

1c (white solid, 1.6 g, 5.2 mmol, 51%)
The spectral data were in good agreement with the literature values.²
¹H NMR (CDCl₃, 400 MHz) δ = 7.61-7.55 (m, 4H), 7.43-7.34 (m, 6H), 7.05-6.90 (m, 2H), 6.82-6.76 (m, 2H), 3.75 (s, 3H).

1d (colorless oil, 1.6 g, 4.5 mmol, 45%)
¹H NMR (CDCl₃, 400 MHz) δ = 7.58-7.52 (m, 4H), 7.20 (d, J = 8.0 Hz, 2H), 7.13-7.40 (m, 6H), 7.97 (d, J = 8.0 Hz, 2H); ³¹P NMR (CDCl₃, 200 MHz) δ = 113.3; ¹⁹F NMR (CDCl₃, 465 MHz) δ = -62.5; ¹³C NMR (CDCl₃, 125 MHz) δ = 160.2 (d, J = 10.7 Hz), 140.6 (d, J = 18.3 Hz), 131.0 (d, J = 22.8 Hz).
Hz), 130.3, 128.9 (d, $J = 7.5$ Hz), 127.2 (d, $J = 3.8$ Hz), 125.0 (q, $J = 272$ Hz), 124.7 (q, $J = 32.5$ Hz), 119.1 (d, $J = 11.3$ Hz); IR (ATR) 3053.6, 1610.3, 1510.1, 1434.8, 1322.0, 1228.4, 1106.0 cm$^{-1}$; HRMS (ESI): Calcd for C$_{19}$H$_{15}$F$_{3}$OP $[M+H]^+$:347.0807; Found: 347.0790.

1e (colorless oil, 2.1 g, 7.1 mmol, 71%)
$^1$H NMR (C$_6$D$_6$, 500 MHz) $\delta$ = 7.62-7.54 (m, 4H), 7.14-7.02 (m, 6H), 6.92-6.86 (m, 2H), 6.68-6.60 (m, 2H); $^{31}$P NMR (C$_6$D$_6$, 200 MHz) $\delta$ = 114.4; $^{19}$F NMR (C$_6$D$_6$, 465 MHz) $\delta$ = −112.3; $^{13}$C NMR (C$_6$D$_6$, 125 MHz) $\delta$ = 158.8 (d, $J = 241.8$ Hz), 153.9 (d, $J = 2.4$ Hz), 141.4 (d, $J = 18.0$ Hz), 130.9 (d, $J = 22.8$ Hz), 130.0, 128.8 (d, $J = 7.1$ Hz), 120.4 (dd, $J = 10.8, 8.4$ Hz), 116.2 (d, $J = 22.8$ Hz); IR (ATR) 3053.7, 1497.5, 1433.8, 1194.7, 1090.6 cm$^{-1}$; HRMS (ESI): Calcd for C$_{18}$H$_{15}$FOP $[M+H]^+$:297.0839; Found: 297.0831.

1f (white solid, 2.0 g, 6.5 mmol, 65%)
The spectral data were in good agreement with the literature values.$^3$
$^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ = 7.61-7.54 (m, 4H), 7.44-7.37 (m, 6H), 7.23-7.19 (m, 2H), 7.08-7.03 (m, 2H).

1g (white solid, 2.7 g, 5.5 mmol, 75%)
$^1$H NMR (C$_6$D$_6$, 500 MHz) $\delta$ = 7.58-7.51 (m, 4H), 7.11-7.01 (m, 8H), 6.84-6.78 (m, 2H); $^{31}$P NMR (C$_6$D$_6$, 200 MHz) $\delta$ = 113.6; $^{13}$C NMR (C$_6$D$_6$, 125 MHz) $\delta$ = 156.9 (d, $J = 9.6$ Hz), 141.1 (d, $J = 18.0$ Hz), 132.8, 131.0 (d, $J = 22.8$ Hz), 130.1, 128.8 (d, $J = 7.1$ Hz), 121.0 (d, $J = 10.8$ Hz), 115.4; IR (ATR) 3065.3, 1582.3, 1573.6, 1478.2, 1433.8, 1220.7, 1097.3 cm$^{-1}$; HRMS (ESI): Calcd for C$_{18}$H$_{15}$BrOP $[M+H]^+$:357.0038; Found: 357.0040.
1h (white solid, 2.0 g, 4.9 mmol, 49%)  
$^1$H NMR (C$_6$D$_6$, 500 MHz) $\delta$ = 7.55 (t, $J = 7.5$ Hz, 4H), 7.25 (d, $J = 8.3$ Hz, 2H), 7.14-7.01 (m, 6H), 6.72 (d, $J = 8.3$ Hz, 2H); $^{31}$P NMR (C$_6$D$_6$, 200 MHz) $\delta$ = 113.1; $^{13}$C NMR (C$_6$D$_6$, 125 MHz) $\delta$ = 157.6 (d, $J = 10.8$ Hz), 141.0 (d, $J = 17.9$ Hz), 138.8, 130.9 (d, $J = 22.8$ Hz), 130.1, 128.8 (d, $J = 7.1$ Hz), 121.4 (d, $J = 10.8$ Hz), 85.7; IR (ATR) 3073.9, 1577.5, 1475.3, 1211.1, 1164.8, 1092.5 cm$^{-1}$; HRMS (ESI): Calcd for C$_{18}$H$_{15}$IOP [M+H]$^+$: 404.9900; Found: 404.9886.

1i (white solid, 1.1 g, 3.3 mmol, 34%)  
$^1$H NMR (C$_6$D$_6$, 500 MHz) $\delta$ = 8.03 (d, $J = 8.5$, 2H), 7.55 (t, $J = 7.5$ Hz, 4H), 7.20-7.02 (m, 8H), 3.45 (s, 3H); $^{31}$P NMR (C$_6$D$_6$, 200 MHz) $\delta$ = 112.5; $^{13}$C NMR (C$_6$D$_6$, 125 MHz) $\delta$ = 165.9, 161.1 (d, $J = 10.9$ Hz), 140.4 (d, $J = 16.9$ Hz), 131.7, 130.7 (d, $J = 22.8$ Hz), 129.9, 128.5 (d, $J = 6.0$ Hz), 124.9, 118.5 (d, $J = 12.0$ Hz), 51.2; IR (ATR) 1718.3, 1598.7, 1503.2, 1431.9, 1309.4, 1271.8, 1235.2, 1167.7, 1101.2, 1009.6 cm$^{-1}$; HRMS (ESI): Calcd for C$_{20}$H$_{18}$O$_3$P [M+H]$^+$: 337.0988; Found: 337.0985.

1j (white solid, 2.3 g, 7.0 mmol, 70%)  
The spectral data were in good agreement with the literature values.$^4$  
$^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ = 7.79-7.74 (m, 2H), 7.70 (d, $J = 10.0$ Hz, 1H), 7.64 (td, $J = 10.0$, 3.0 Hz, 4H), 7.51 (t, $J = 3.0$ Hz 1H), 7.45-7.38 (m, 7H), 7.38-7.32 (m, 2H).

1k (white solid, 1.2 g, 4.1 mmol, 41%)  
$^1$H NMR (C$_6$D$_6$, 500 MHz) $\delta$ = 7.68-7.62 (m, 4H), 7.12-7.02 (m, 7H), 6.99 (t, $J = 7.8$ Hz, 1H), 6.67 (d, $J = 7.8$ Hz, 2H), 2.00 (s, 3H); $^{31}$P NMR (C$_6$D$_6$, 200 MHz) $\delta$ = 110.9; $^{13}$C NMR (C$_6$D$_6$, 125 MHz) $\delta$ = 158.0 (d, $J = 9.5$ Hz), 141.8 (d, $J = 17.9$ Hz), 139.9, 130.9 (d, $J = 22.8$ Hz), 129.9, 129.7, 128.8
(d, $J = 7.1$ Hz), 123.8, 120.1 (d, $J = 10.8$ Hz), 116.3 (d, $J = 12.0$ Hz), 21.3; IR (ATR) 3052.8, 1603.5, 1582.3, 1484.0, 1433.8, 1250.6, 1145.5, 1094.4 cm$^{-1}$; HRMS (ESI): Calcd for C$_{19}$H$_{18}$OP [M+H]$^+$: 293.1090; Found: 293.1088.

![OPPh$_2$](image)

**11** (colorless oil, 1.9 g, 6.2 mmol, 62%)

$^1$H NMR (C$_6$D$_6$, 500 MHz) $\delta = 7.56$-7.51 (m, 4H), 7.30 (dd, $J = 4.0$, 2.3 Hz 1H), 7.10-7.00 (m, 6H), 6.93 (br d, $J = 8.0$ Hz, 1H), 6.79 (dd, $J = 8.0$, 2.3 Hz, 1H), 6.68 (t, $J = 8.0$ Hz, 1H); $^{31}$P NMR (C$_6$D$_6$, 200 MHz) $\delta = 113.6$; $^{13}$C NMR (C$_6$D$_6$, 125 MHz) $\delta = 158.5$ (d, $J = 10.8$ Hz), 140.8 (d, $J = 18.0$ Hz), 135.2, 130.9 (d, $J = 22.8$ Hz), 130.7, 130.1, 128.8 (d, $J = 7.3$ Hz), 123.0, 119.8 (d, $J = 10.8$ Hz), 117.3 (d, $J = 11.9$ Hz); IR (ATR) 3055.7, 1586.2, 1472.4, 1433.8, 1215.9, 1088.6 cm$^{-1}$; HRMS (ESI): Calcd for C$_{18}$H$_{15}$ClOP [M+H]$^+$: 313.0544; Found: 313.0534.

![OPPh$_2$](image)

**1m** (colorless oil, 1.9 g, 6.3 mmol, 63%)

$^1$H NMR (C$_6$D$_6$, 500 MHz) $\delta = 7.67$-7.61 (m, 4H), 7.11-7.01 (m, 6H), 6.98-6.89 (m, 3H), 6.51 (dd, $J = 8.5$, 2.0 Hz, 1H), 3.20 (s, 3H); $^{31}$P NMR (C$_6$D$_6$, 200 MHz) $\delta = 111.3$; $^{13}$C NMR (C$_6$D$_6$, 125 MHz) $\delta = 161.5$, 159.4 (d, $J = 10.8$ Hz), 141.5 (d, $J = 21.6$ Hz), 131.0 (d, $J = 22.8$ Hz), 130.4, 129.9, 128.8 (d, $J = 7.3$ Hz), 111.3 (d, $J = 12.0$ Hz), 109.0, 105.4 (d, $J = 10.8$ Hz), 54.8; IR (ATR) 3071.1, 3053.7, 3002.6, 2957.3, 2938.0, 2832.9, 1598.7, 1588.1, 1487.8, 1433.8, 1261.2, 1191.8, 1138.8 cm$^{-1}$; HRMS (ESI): Calcd for C$_{19}$H$_{18}$O$_2$P [M+H]$^+$: 309.1034; Found: 309.1034.

![OPPh$_2$](image)

**1n** (white solid, 2.3 g, 7.0 mmol, 70%)

$^1$H NMR (C$_6$D$_6$, 500 MHz) $\delta = 7.64$-7.56 (m, 4H), 7.12-7.01 (m, 6H), 6.81 (s, 1H), 6.59 (dt, $J = 8.5$, 2.5 Hz, 1H), 6.46 (d, $J = 8.5$ Hz, 1H), 5.22 (s, 2H); $^{31}$P NMR (C$_6$D$_6$, 200 MHz) $\delta = 114.4$; $^{13}$C NMR (C$_6$D$_6$, 125 MHz) $\delta = 153.0$ (d, $J = 9.6$ Hz), 148.8, 143.6, 141.7 (d, $J = 18.0$ Hz), 131.0 (d, $J = 21.6$ Hz), 129.9, 128.7 (d, $J = 6.0$ Hz), 111.4 (d, $J = 12.0$ Hz), 108.3, 102.1 (d, $J = 9.5$ Hz), 101.3; IR (ATR) 2909.1, 1624.7, 1500.4, 1483.0, 1240.0, 1183.1, 1122.4, 1094.4 cm$^{-1}$; HRMS (ESI): Calcd for C$_{19}$H$_{16}$O$_3$P [M+H]$^+$: 323.0832; Found: 323.0819.

S6
10 was synthesized according to the slightly modified procedure as follows.

In a 100 mL two-neck flask, 6-hydroxyquinoline (1.45 g, 10.0 mmol) was dissolved in THF (30.0 mL) under an argon atmosphere. The flask was cooled to 0 °C in an ice-bath, and triethylamine (2.77 mL, 20.0 mmol) and chlorodiphenylphosphine (1.84 mL, 10.0 mmol) were successively added. The flask was allowed to stand at room temperature and stirred for 2 h. The solution was filtered through a short pad of Celite®. After the filtrate was concentrated under reduced pressure, the resulting solid was precipitated from hexane/ dichloromethane to give 10.

$^1$H NMR (CDCl$_3$, 500 MHz) $\delta = 8.79$ (dd, $J = 4.0$, 1.5 Hz, 1H), 8.04-8.00 (m, 2H), 7.68-7.60 (m, 4H), 7.57 (dd, $J = 9.0$, 2.5 Hz 1H), 7.47 (t, $J = 2.5$ Hz, 1H) 7.45-7.38(m, 6H), 7.32 (dd, $J = 8.5$, 4.0 Hz 1H); $^{31}$P NMR (CDCl$_3$ 200 MHz) $\delta = 112.4$; $^{13}$C NMR (CDCl$_3$ 125 MHz) $\delta = 155.4$ (d, $J = 9.5$ Hz), 148.9, 145.0, 140.4 (d, $J = 16.8$ Hz), 135.3, 131.1, 130.7 (d, $J = 22.8$ Hz), 130.1, 129.2, 128.7 (d, $J = 7.3$ Hz), 124.2 (d, $J = 7.1$ Hz), 121.5, 113.1 (d, $J = 13.0$ Hz); IR (ATR) 3067.2, 2351.8, 2320.0, 1619.9, 1592.9, 1497.5, 1376.0, 1319.1, 1220.7, 1160.0 cm$^{-1}$; HRMS (ESI+) Calcd for C$_{21}$H$_{17}$NOP [M+H]$^+$: 330.1048; Found: 330.1057.

4 (white solid, 1.3 g, 4.7 mmol, 47%)

The spectral data were in good agreement with the literature values

$^1$H NMR (C$_6$D$_6$, 500 MHz) $\delta = 7.48$-7.42 (m, 4H), 7.40-7.33 (m, 6H), 7.19 (t, $J = 7.9$ Hz, 2H), 7.04-6.98 (m, 2H), 6.81 (t, $J = 7.9$ Hz, 1H), 4.37 (d, $J = 7.4$ Hz, 1H).
General procedure for C–H borylation of aryl diphenylphosphinites

Procedure A

A solution of $[\text{RuCl}_2(p$-cymene)$]_2$ (0.9 mg, 1.5 µmol) and aryl diphenylphosphinite 1 (0.30 mmol) in n-octane (1.5 mL) was placed in a glass tube (inside diameter = 1.7 cm, length = 18 cm) equipped with a three-way stop cock under Ar. HBpin (47.4 µL, 0.33 mmol) was added to the solution at room temperature, and the glass tube was closed. Then the mixture was stirred at 150 ºC in the closed system for 6 h. 1N HCl aq. was added to the solution at room temperature, and the mixture was stirred for 24 h. The mixture was extracted with AcOEt three times, and the combined organic layers were washed with brine and dried over MgSO$_4$. After filtration to remove the drying agent, the filtrate was evaporated to give a crude product. To a MeOH solution (1.5 mL) of the crude product was added KHF$_2$ (93.7 mg, 1.2 mmol) at room temperature, and the mixture was stirred for 24 h. The suspension was evaporated at 40˚C for 20 min, and residual white solids were dissolved in acetone (5.0 mL). After insoluble salts were filtered off, the filtrate was concentrated to ca. 1 mL volume. Then, toluene (ca. 1 mL) was added, and the remaining acetone was removed under reduced pressure to give the suspension in toluene. The resulting beige solids were collected by filtration and rinsed with excess n-hexane and a small amount of cold MeOH (ca. 0˚C) to give aryl trifluoroborate 2.

Procedure B

A solution of $[\text{RuCl}_2(p$-cymene)$]_2$ (0.9 mg, 1.5 µmol) and aryl diphenylphosphinite 1 (0.30 mmol) in n-octane (1.5 mL) was placed in a glass tube (inside diameter = 1.7 cm, length = 18 cm) equipped with a three-way stop cock under Ar. HBpin (47.4 µL, 0.33 mmol) was added to the solution at room temperature, and the glass tube was closed. Then the mixture was stirred at 150 ºC in the closed system for 6 h. 1N HCl aq. was added to the solution at room temperature, and the mixture was stirred for 24 h. The mixture was extracted with AcOEt three times, and the combined organic layers were washed with brine and dried over MgSO$_4$. After filtration to remove the drying agent, the filtrate was evaporated to give a crude product. To a MeOH solution (1.5 mL) of the crude product was added KHF$_2$ (93.7 mg, 1.2 mmol) at room temperature, and the mixture was stirred for 24 h. After the suspension was evaporated at 40˚C for 20 min, the resulting beige solid
was collected by filtration and rinsed with a small amount of cold MeOH (ca. 0°C) to give aryl trifluoroborate 2.

This reaction should be conducted inside an explosion proof wall. This reaction is performed in a sealed glass tube at 150 °C which is over the boiling point of n-octane. Furthermore, H₂ is generated during the reaction. The volume of the dead space inside the glass ware is ca. 34.5 cm³.

Synthesized according to the procedure A. 
2a (white solid, 46 mg, 0.23 mmol, 77%)
The spectral data were in good agreement with the literature values.⁶
¹H NMR (acetone-d₆, 500 MHz) δ = 7.41 (q, J = 10.5 Hz, 1H), 7.27 (br d, J = 7.0 Hz, 1H), 6.93-6.88 (m, 1H), 6.63-6.57 (m, 1H), 6.52 (d, J = 8.5 Hz, 1H).

Synthesized according to the procedure A. 
2b (white solid, 49 mg, 0.23 mmol, 76%)
The spectral data were in good agreement with the literature values.⁶
¹H NMR (acetone-d₆, 500 MHz) δ = 7.23 (q, J = 10.5 Hz, 1H), 7.08 (br s, 1H), 6.71 (dd, J = 8.0, 2.5 Hz, 1H), 6.41 (d, J = 8.0 Hz, 1H), 2.13 (s, 3H).

Synthesized according to the procedure B. 
2c (white solid, 68 mg, 0.29 mmol, 96%)
The spectral data were in good agreement with the literature values.⁷
¹H NMR (acetone-d₆, 500 MHz) δ = 7.07 (q, J = 11.0 Hz, 1H), 6.84 (br s, 1H), 6.45 (dd, J = 8.5, 3.5 Hz, 1H), 6.40 (d, J = 8.5 Hz, 1H), 3.61 (s, 3H).
Synthesized according to the procedure A.

2d (white solid, 46 mg, 0.17 mmol, 57%)

$^1$H NMR (acetone-$d_6$, 500 MHz) $\delta = 8.02$ (q, $J = 11.5$ Hz, 1H), 7.59 (br s, 1H), 7.24 (dd, $J = 8.5$, 2.0 Hz, 1H), 6.66 (d, $J = 8.5$ Hz, 1H); $^{13}$C NMR (acetone-$d_6$, 125 MHz) $\delta = 164.2$, 131.2, 126.8 (q, $J = 271.6$ Hz), 124.9 (d, $J = 3.6$ Hz), 120.6 (q, $J = 31.1$ Hz), 114.3, 1C(C–B) is missing.; $^{19}$F NMR (acetone-$d_6$, 465 MHz), $\delta = -62.3$ (s), -139.2 (m); $^{11}$B NMR (acetone-$d_6$, 158 MHz) $\delta = 3.06$ (q, $J = 52.8$ Hz); IR (ATR) 1329.7, 1239.0, 1181.2, 1113.7, 1079.0 cm$^{-1}$; HRMS (ESI): Calcd for C$_7$H$_4$BF$_6$O [M–K$^-$]: 229.0265; Found: 229.0255.

Synthesized according to the procedure A.

2e (white solid, 52 mg, 0.24 mmol, 80%)

$^1$H NMR (acetone-$d_6$, 500 MHz) $\delta = 7.28$ (q, $J = 10.5$ Hz 1H), 6.93 (br d, $J = 7.5$ Hz, 1H), 6.62 (td, $J = 8.5$, 3.5 Hz, 1H), 6.49 (dd, $J = 8.5$, 4.0 Hz 1H); $^{13}$C NMR (acetone-$d_6$, 125 MHz) $\delta = 157.4$ (d, $J = 234.6$ Hz), 156.9, 119.1(d, $J = 19.1$ Hz), 114.6, 113.2 (d, $J = 24.0$ Hz), 1C(C–B) is missing.; $^{19}$F NMR (acetone-$d_6$, 465 MHz), $\delta = -131.0$ (s), -139.7 (m); $^{11}$B NMR (acetone-$d_6$, 158 MHz) $\delta = 2.99$ (q, $J = 54.2$ Hz); IR (ATR) 3565.7, 1487.8, 1417.4, 1265.1, 1187.0, 1144.6 cm$^{-1}$; HRMS (ESI): Calcd for C$_6$H$_4$BF$_4$O [M–K$^-$]: 179.0297; Found: 179.0294.

Synthesized according to the procedure A.

2f (white solid, 69 mg, 0.29 mmol, 98%)

The spectral data were in good agreement with literature values.$^6$

$^1$H NMR (acetone-$d_6$, 500 MHz) $\delta = 7.54$ (q, $J = 11.0$ Hz, 1H), 7.19 (br s, 1H), 6.85 (dd, $J = 8.8$, 3.0 Hz, 1H), 6.48 (d, $J = 8.8$ Hz 1H).
Synthesized according to the procedure A.

**2g** (white solid, 74 mg, 0.26 mmol, 88%)

$^1$H NMR (acetone-$d_6$, 500 MHz) $\delta$ = 7.55 (q, $J = 11.0$ Hz 1H), 7.36 (br s, 1H), 7.02 (dd, $J = 8.5$, 3.0 Hz, 1H), 6.47 (d, $J = 8.5$ Hz 1H); $^{13}$C NMR (acetone-$d_6$, 125 MHz) $\delta$ =160.4, 136.6, 130.1, 116.5, 111.6, 1C(C–B) is missing.; $^{19}$F NMR (acetone-$d_6$, 465 MHz), $\delta$ = −139.3 (m); $^{11}$B NMR (acetone-$d_6$, 158 MHz) $\delta$ = 2.85 (q, $J = 52.8$ Hz); IR (ATR) 3523.3, 1600.6, 1482.0, 1390.4, 1241.0, 1121.4 cm$^{-1}$; HRMS (ESI): Calcd for C$_6$H$_4$BBrF$_3$O [M–K$^-$]: 238.9496; Found: 238.9502.

Synthesized according to the procedure B.

**2h** (white solid, 95 mg, 0.29 mmol, 97%)

$^1$H NMR (acetone-$d_6$, 500 MHz) $\delta$ = 7.60-7.51 (m, 2H), 7.21(dd, $J = 8.5$, 2.5 Hz, 1H), 6.39 (d, $J = 8.5$ Hz, 1H); $^{13}$C NMR (acetone-$d_6$, 125 MHz) $\delta$ =160.1, 142.8, 136.2, 117.3, 82.1, 1C(C–B) is missing.; $^{19}$F NMR (acetone-$d_6$, 465 MHz), $\delta$ = −137.9 (m); $^{11}$B NMR (acetone-$d_6$, 158 MHz) $\delta$ = 2.71 (q, $J = 54.2$ Hz); IR (ATR) 3505.0, 1595.8, 1478.2, 1381.8, 1245.8, 1177.3, 1120.4 cm$^{-1}$; HRMS (ESI): Calcd for C$_6$H$_4$BF$_3$IO [M–K$^-$]: 286.9358; Found: 286.9361.

Synthesized according to the procedure B.

**2i** (white solid, 58 mg, 0.22 mmol, 75%)

$^1$H NMR (acetone-$d_6$, 500 MHz) $\delta$ = 8.12-8.02 (m, 2H), 7.65 (dd, $J = 8.5$, 2.5 Hz, 1H), 6.59 (d, $J = 8.5$ Hz 1H), 3.76 (s, 3H); $^{13}$C NMR (acetone-$d_6$, 125 MHz) $\delta$ =168.2, 165.8, 136.7, 130.1, 121.1, 114.2, 51.4, 1C(C–B) is missing.; $^{19}$F NMR (acetone-$d_6$, 465 MHz), $\delta$ = −138.7 (m); $^{11}$B NMR (acetone-$d_6$, 158 MHz) $\delta$ = 3.22 (q, $J = 52.8$ Hz); IR (ATR) 3512.7, 1700.0, 1613.2, 1438.6, 1313.3, 1229.4, 1163.8 cm$^{-1}$; HRMS (ESI): Calcd for C$_8$H$_7$BF$_3$O$_3$ [M–K$^-$]: 219.0446; Found: 219.0445.
Synthesized according to the procedure B.

**2j** (white solid, 65 mg, 0.26 mmol, 86%)

The spectral data were in good agreement with literature values.\(^8\)

\(^1\)H NMR (dms-o-d\(_6\), 500 MHz) \(\delta = 7.68-7.52\) (m, 4H), 7.24 (t, \(J = 7.5\) Hz, 1H), 7.14 (d, \(J = 7.5\) Hz 1H), 6.88 (s, 1H)

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Synthesized according to the procedure A.

**2k** (white solid, 55 mg, 0.26 mmol, 85%)

The spectral data were in good agreement with literature values.\(^6\)

\(^1\)H NMR (acetone-d\(_6\), 500 MHz) \(\delta = 7.40\) (q, \(J = 10.5\) Hz, 1H), 7.14 (d, \(J = 6.5\) Hz, 1H), 6.43 (d, \(J = 6.5\) Hz 1H), 6.35 (s, 1H), 2.15 (s, 3H).

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**Borylation of 1l:** A solution of [RuCl\(_2\)(p-cymene)]\(_2\) (0.9 mg, 1.5 \(\mu\)mol) and 1l (93.8 mg, 0.30 mmol) in \(n\)-octane (1.5 mL) was placed in a glass tube (inside diameter = 1.7 cm, length = 18 cm) equipped with a three-way stop cock under Ar. HBpin (47.4 \(\mu\)L, 0.33 mmol) was added to the solution at room temperature, and the glass tube was closed. Then the mixture was stirred at 150 \(^\circ\)C in the closed system for 6 h. 1N HCl aq. was added to the solution at room temperature, and the mixture was stirred for 24 h. The mixture was extracted with AcOEt three times, and the combined organic layers were washed with brine and dried over MgSO\(_4\). After filtration to remove the drying agent, the filtrate was evaporated to give a crude product. The crude mixture was passed through a short pad of silica gel using CH\(_2\)Cl\(_2\) as an eluent to afford a mixture of the borylation products 2l and 3l as colorless oil (53 mg, 0.21 mmol, 69%, 2l:3l = 89:11). These regioisomers were inseparable by column chromatography, and the ratio was determined by \(^1\)H NMR.

The spectral data were in good agreement with literature values.\(^9\)
2l: $^1$H NMR (CDCl$_3$, 500 MHz) $\delta = 7.91$ (s, 1H), 7.51 (d, $J = 10.0$ Hz, 1H), 6.91-6.82 (m, 2H), 1.36 (s, 12H).

3l: $^1$H NMR (CDCl$_3$, 500 MHz) $\delta = 8.63$ (s, 1H), 7.23 (t, $J = 10.8$ Hz, 1H), 6.77(d, $J = 10.8$ Hz, 1H), 1.40 (s, 12H), a phenolic OH is missing.

Borylation of 1m: A solution of [RuCl$_2$(p-cymene)]$_2$ (0.9 mg, 1.5 µmol) and 1m (92.5 mg, 0.30 mmol) in n-octane (1.5 mL) was placed in a glass tube (inside diameter = 1.7 cm, length = 18 cm) equipped with a three-way stop cock under Ar. HBpin (47.4 µL, 0.33 mmol) was added to the solution at room temperature, and the glass tube was closed. Then the mixture was stirred at 150 °C in the closed system. After 6 h, the solvent was removed under reduced pressure, and the obtained crude material was suspended in THF (1.0 mL). BH$_3$•thf (0.9 M solution in THF, 1.5 mL, 1.4 mmol) was added to the solution at room temperature, and then the mixture was stirred for 3 h. The reaction was quenched by adding H$_2$O at 0 °C, and the obtained crude material was purified by silica gel chromatography (hexane: ethyl acetate = 5:1) to give the borylation product 2m containing a small amount of 1m. These compounds were inseparable, and the yield of 2m was calculated to be 68% (0.21 mmol) by $^1$H NMR.

$^1$H NMR (CDCl$_3$, 500 MHz) $\delta = 8.06-7.98$ (m, 4H), 7.65 (d, $J = 8.8$ Hz 1H), 7.58-7.42 (m, 6H), 6.71(s, 1H), 6.63 (d, $J = 8.8$ Hz, 1H), 3.67 (s, 1H), 1.27 (s, 12H), BH$_3$ are missing.; $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta = 163.0$, 158.8 (d $J = 4.7$ Hz), 138.2, 132.6, 132.3, 132.1, 132.0, 128.5 (d, $J = 10.4$ Hz) 110.4, 106.0 (d, $J = 4.8$ Hz), 83.5, 55.4, 25.0; $^{31}$P NMR (CDCl$_3$, 200 MHz) $\delta = 107.6$ (br), $^{11}$B NMR (CDCl$_3$, 158 MHz) $\delta = 29.9$ (br), $-39.3$ (br); IR (ATR) 2975.6, 2929.3, 2384.6, 1605.5, 1416.5, 1349.9, 1238.1, 1131.1, 1074.2 cm$^{-1}$; HRMS (FD): Calcd for C$_{25}$H$_{31}$B$_2$O$_4$P [M]$^+$:448.2146; Found: 448.2138.

Borylation of 1n: A solution of [RuCl$_2$(p-cymene)]$_2$ (0.9 mg, 1.5 µmol) and 1n (96.7 mg, 0.30 mmol) in n-octane (1.5 mL) was placed in a glass tube (inside diameter = 1.7 cm, length = 18 cm)
equipped with a three-way stop cock under Ar. HBpin (47.4 μL, 0.33 mmol) was added to the solution at room temperature, and the glass tube was closed. Then the mixture was stirred at 150 °C in the closed system. After 6 h, the solvent was removed under reduced pressure, and the obtained crude material was suspended in THF (1.0 mL). BH₃•thf (0.9 M solution in THF, 1.5 mL, 1.4 mmol) was added to the solution at room temperature, and then the mixture was stirred for 3 h. The reaction was quenched by adding H₂O at 0 °C, and the obtained crude material was purified by silica gel chromatography using toluene as an eluent to give a fraction of 2n containing a small amount of 3n (2n:3n = 93:7) and a fraction of 3n containing a small amount of 1n and 2n (1n:2n:3n = 14:3:83). The yields were calculated to be 55% for 2n (0.16 mmol) and 34% for 3n (0.10 mmol) by ¹H NMR.

2n (white solid, 0.16 mmol, 55%)
¹H NMR (CDCl₃, 500 MHz) δ = 8.00-7.94 (m, 4H), 7.54-7.43 (m, 2H), 7.48-7.42 (m, 4H), 7.13 (s, 1H), 6.48 (s, 1H), 5.92 (s, 2H), 1.21 (s, 12H). BH₃ are missing.; ¹³C NMR (CDCl₃, 100 MHz) δ = 152.9 (d, J = 4.8 Hz), 150.6, 144.3, 132.7, 132.2, 132.1, 132.0, 128.6 (d, J = 10.8 Hz), 114.4, 102.9, 101.9, 83.7, 24.9; ³¹P NMR (CDCl₃, 200 MHz) δ = 109.2 (br); ¹¹B NMR (CDCl₃, 158 MHz) δ = 30.1 (br), -39.8 (br); IR (ATR) 3059.5, 2975.6, 2385.5, 1725.0, 1619.9, 1485.9, 1424.2, 1370.2, 1315.2, 1241.9, 1110.8, 1037.5 cm⁻¹; HRMS (FD): Calcd for C₂₅H₂₉B₂O₅P [M⁺]: 462.1939; Found: 462.1928.

3n (white solid, 0.1 mmol, 34%)
¹H NMR (CDCl₃, 500 MHz) δ = 7.98-7.90 (m, 4H), 7.54-7.43 (m, 6H), 6.60 (d, J = 8.5 Hz, 1H), 6.33 (d, J = 8.5, 1.0 Hz, 1H), 5.99 (s, 2H), 1.21 (s, 12H); ¹³C NMR (CDCl₃, 100 MHz) δ = 153.7, 151.1 (d,J = 4.8 Hz), 144.0, 132.8, 132.3, 132.0, 131.9, 128.5 (d, J = 10.8 Hz) 112.7 (d, J = 4.8 Hz), 109.9, 101.7, 83.8, 24.9; ³¹P NMR (CDCl₃, 200 MHz) δ = 108.6 (br); ¹¹B NMR (CDCl₃, 158 MHz) δ = 29.3 (br), -40.2 (br); IR (ATR) 2974.7, 2917.8, 2397.1, 1638.2, 1437.7, 1329.7, 1209.2, 1135.9, 1050.1 cm⁻¹; HRMS (FD): Calcd for C₂₅H₂₉B₂O₅P [M⁺]:462.1939; Found: 462.1956.
Borylation of 1o: A solution of [RuCl$_2$(p-cymene)]$_2$ (1.8 mg, 3.0 µmol) and 1o (98.8 mg, 0.30 mmol) in n-octane (1.5 mL) was placed in a glass tube (inside diameter = 1.7 cm, length = 18 cm) equipped with a three-way stop cock under Ar. HBpin (47.4 µL, 0.33 mmol) was added to the solution at room temperature, and the glass tube was closed. Then the mixture was stirred at 150 ºC in the closed system for 90 min. After removal of the solvent under reduced pressure, H$_2$O (1.0 mL), KHF$_2$ (93.7 mg, 1.2 mmol), and MeOH (1.0 mL) were successively added and then stirred at room temperature. After 24 h, K$_2$CO$_3$ (80.5 mg, 0.6 mmol) was added to the solution, and the mixture was stirred for 30 min. The suspension was evaporated at 40 ºC for 20 min, and the resulting beige solids were collected by filtration, which were rinsed with a small amount of cold MeOH to give 2o containing small amounts of KBF$_4$ and K[HOBF$_3$] (ca. 2o:KBF$_4$:K[HOBF$_3$] = 100:2.5 in $^{19}$F NMR) (60.3 mg). The yield of 2o was calculated to be 78%.  

$^1$H NMR (dmsod$_6$, 500 MHz) $\delta = 8.55$ (dd, $J = 4.5$, 1.5 Hz, 1H), 7.99 (dd, $J = 8.0$, 0.8 Hz, 1H), 7.88-7.78 (m, 2H), 7.25 (dd, $J = 8.0$, 4.0 Hz, 1H), 6.92 (s, 1H; $^{13}$C NMR (dmsod$_6$, 100 MHz) $\delta$ =158.7, 146.1, 143.7, 133.4, 133.1, 128.3, 120.1, 106.2, 115.3, 114.5 cm$^{-1}$; $^{19}$F NMR (dmsod$_6$, 465 MHz), $\delta = -136.1$; $^{11}$B NMR (dmsod$_6$, 158 MHz) $\delta = 2.26$ (br); IR (ATR) 3045.2, 2926.0, 1625.1, 1440.6, 1416.4, 1349.3, 1330.7, 1222.6, 1157.3, 1114.5 cm$^{-1}$; HRMS (ESI–) Calcd for C$_9$H$_6$BF$_3$NO [M–K]–:212.0495; Found: 212.0504.

Borylation of 4: A solution of [RuCl$_2$(p-cymene)]$_2$ (0.9 mg, 1.5 µmol) and 4 (83.2 mg, 0.30 mmol) in n-octane (1.5 mL) was placed in a glass tube (inside diameter = 1.7 cm, length = 18 cm) equipped with a three-way stop cock under Ar. HBpin (47.4 µL, 0.33 mmol) was added to the solution at room temperature, and the glass tube was closed. Then the mixture was stirred at 150 ºC in the closed system for 6 h. 1N HCl aq. was added to the solution at room temperature, and the mixture was stirred for 24 h. The mixture was extracted with AcOEt three times, and the combined organic layers were washed with brine and dried over MgSO$_4$. After the filtration of the drying
agent, the filtrate was evaporated to give a crude product. The crude product was purified by silica gel chromatography (hexane: ethyl acetate = 20:1) to give the borylation products 5 as a colorless oil (30 mg, 0.14 mmol, 46%).

The spectral data were in good agreement with the literature values.\textsuperscript{10}

\textsuperscript{1}H NMR (CDCl\textsubscript{3}, 400 MHz) \( \delta = 7.61 \text{ (dd, } J = 7.6, 1.6 \text{ Hz, } 1\text{H}), 7.24-7.18 \text{ (m, } 1\text{H}), 6.67 \text{ (t, } J = 7.6 \text{ Hz, } 1\text{H}), 6.59 \text{ (d, } J = 7.6 \text{ Hz, } 1\text{H}), 4.73 \text{ (br s, } 2\text{H}), 1.34 \text{ (s, } 12\text{H}).
**Procedure for C–H borylation of aryl phosphites (Scheme 3)**

A solution of $[\text{RuCl}_2(p$-cymene)]$_2$ (1.8 mg, 3.0 µmol) and aryl phosphate 6 (0.30 mmol) in $n$-octane (1.5 mL) was placed in a glass tube (inside diameter = 1.7 cm, length = 18 cm) equipped with a three-way stop cock under Ar. HBpin (133.7 µL, 0.93 mmol) was added to the solution at room temperature, and the glass tube was closed. Then the mixture was stirred at 150 °C in the closed system for 6 h. 4N HCl aq. was added to the solution at room temperature, and the mixture was stirred for 48 h. The mixture was extracted with AcOEt three times, and the combined organic layers were washed with brine and dried over MgSO$_4$. After filtration to remove the drying agent, the filtrate was evaporated to give a crude product. To a MeOH solution (1.5 mL) of the crude product was added KHF$_2$ (281 mg, 3.6 mmol) at room temperature, and the mixture was stirred for 24 h. The suspension was evaporated at 40°C for 20 min, and residual white solids were dissolved in acetone (5.0 mL). After insoluble salts were filtered off, the filtrate was concentrated to ca. 1 mL volume. Then, toluene (ca. 1 mL) was added, and the remaining acetone was removed under reduced pressure to give suspension in toluene. The resulting beige solids were collected by filtration and rinsed with excess $n$-hexane and a small amount of cold MeOH (ca. 0°C) to give aryl trifluoroborate 2.

**Procedure for one-pot borylation of phenol (Scheme 4)**

A solution of phenol (282 mg, 3.0 mmol) and P(NMe$_2$)$_3$ (181.3 µL, 1.0 mmol) in toluene (1.0 mL) was placed in a 50 mL Schlenk flask under Ar, and the glass tube was closed. Then the mixture was stirred at 130 °C in the closed system. After 8 h, the solvent was removed under reduced pressure at 100 °C for 1 h to give triphenyl phosphate 6a. To the same flask was added $[\text{RuCl}_2(p$-cymene)]$_2$ (30.6 mg, 50 µmol), $n$-octane (5.0 mL) and HBpin (575.2 µL, 4.0 mmol) under Ar, and then the mixture was stirred at 150 °C in the closed system. After 6 h, KHF$_2$ (937 mg, 12 mmol), MeOH (3.0 mL) and H$_2$O (2.0 mL) were added at room temperature, and the solution was stirred for 24 h. After removal of volatile materials, the residual white solids were dissolved in acetone (10~15 mL). After insoluble salts were filtered off, the filtrate was concentrated to ca. 1 mL volume. Then, toluene (ca.
3 mL) was added, and the remaining acetone was removed under reduced pressure to give suspension in toluene. The resulting beige solids were collected by filtration and rinsed with excess n-hexane and a small amount of cold MeOH (ca. 0°C) to give aryl trifluoroborate 2a as white solids (342 mg, 1.7 mmol, 57% based on phenol).
Deuterium labeling experiments

1. Intermolecular competition experiment (Scheme 5a)

A solution of [RuCl₂(p-cymene)]₂ (0.9 mg, 1.5 µmol), 1a (41.7 mg, 0.15 mmol) and 1a-d (42.5 mg, 0.15 mmol) in n-octane (1.5 mL) was placed in a glass tube (inside diameter = 1.7 cm, length = 18 cm) equipped with a three-way stop cock under Ar. HBpin (21.6 µL, 0.15 mmol) was added to the solution at room temperature, and the glass tube was closed. Then the mixture was stirred at 150 °C in the closed system for 6 h. 1N HCl aq. was added to the solution at room temperature, and the mixture was stirred for 24 h. The mixture was extracted with AcOEt three times, and the combined organic layers were washed with brine and dried over MgSO₄. After the filtration of the drying agent, the filtrate was evaporated to give a pale-yellow oil. The yields of borylation products were determined to be 2a = 37% and 2a-d = 29% by ¹H NMR using tetrachloroethane (10 µL, 0.095 mmol) as an internal standard. Therefore, the intermolecular KIE was calculated to be KIE = 1.3.

2. Reaction of 1a with DBpin (Scheme 5b)

A solution of [RuCl₂(p-cymene)]₂ (0.9 mg, 1.5 µmol) and 1a (83.5 mg, 0.3 mmol) in n-octane (1.5 mL) was placed in a glass tube (inside diameter = 1.7 cm, length = 18 cm) equipped with a three-way stop cock under Ar. DBpin (47.8 µL, 0.33 mmol) was added to the solution at room temperature, and the glass tube was closed. Then the mixture was stirred at 150 °C in the closed system for 6 h. 1N HCl aq. was added to the solution at room temperature, and the mixture was stirred for 24 h. The mixture was extracted with AcOEt three times, and the combined organic layers were washed with brine and dried over MgSO₄. After the filtration of the drying agent, the filtrate was evaporated to give a pale-yellow oil. The yields of 2a was determined to be 73% with 36% deuterium incorporation by ¹H NMR using tetrachloroethane (10 µL, 0.095 mmol) as an internal standard.
References
23

abundance

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## Spectral Parameters

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- **Author:** delta
- **Experiment:** proton.jxp
- **Sample_Id:** YH(S-p-F)
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### Spectrometer Parameters

- **Spectrometer:** JNM-EC500/M/S1
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- **Filename:** YH(S-p-F)_Proton-1-1.jdf

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### Graphical Representation

- **Abundance**
- **X : parts per Million : Proton**

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**Supplementary Information**

- **Filename:** YH(S-p-F)_Proton-1-2.jdf
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\text{machinephase}
\]

\[
\text{ppm}
\]

\[
\text{phase}(27, 0, 50\,[\%])
\]

\[
\text{ibase_correct}(:, \text{FALSE, FALSE, FALSE})
\]

\[
\text{base_correct}(\text{Akima, 5, 0, FALSE, 3, None, FA})
\]

\[
\text{Derived from: \textit{YH(S-p-F)}$_{31P}$1Hdec-1-1.jdf}
\]

---

**Filename**                 = \textit{YH(S-p-F)}$_{31P}$1Hdec-1-1.jdf  
**Author**                   = delta  
**Experiment**               = single_pulse_dec.j  
**Sample_Id**                = YH(S-p-F)  
**Solvent**                  = BENZENE-D6  
**Actual_Start_Time**        = 2-APR-2020 11:55:  
**Revision_Time**            = 7-APR-2020 12:34:  
**Comment**                  = single pulse decou  
**Data_Format**              = 1D REAL  
**Dim_Size**                 = 26214  
**X_Domain**                 = Phosphorus31  
**Dim_Title**                = Phosphorus31  
**Dim_Units**                = [ppm]  
**Dimensions**               = X  
**Spectrometer**             = JNM-ECZ500R/S1  
**Field_Strength**           = 11.62926421[T] (50  
**X_Acq_Duration**           = 0.18874368[s]  
**X_Offset**                 = 0[ppm]  
**X_Points**                 = 32768  
**X_Prescans**               = 4  
**X_Resolution**             = 5.29819065[Hz]  
**X_Sweep**                  = 173.61111111[kHz]  
**X_Sweep_Clipped**          = 138.8888889[kHz]  
**Irr_Domain**               = Proton  
**Irr_Freq**                 = 495.13191398[MHz]  
**Irr_Offset**               = 5[ppm]  
**Irr_Decoupling**           = TRUE  
**Irr_Noise**                = WALTZ  
**Scans**                    = 64  
**Total_Scans**              = 64  
**Relaxation_Delay**         = 1[s]  
**Recvr_Gain**               = 66  
**Tamp_Get**                 = 20.2[dc]  
**X_BO_Width**               = 19.78[us]  
**X_Acq_Time**               = 0.18874368[s]  
**X_Angle**                  = 30[deg]  
**X_Atn**                    = 4(4[DB])  
**Irr_Atn_Dec**              = 23.948[DB]  
**Irr_Atn_Dec_Calc**         = 23.948[DB]  
**Irr_Atn_How**              = 23.948[DB]  
**Irr_Dec_Bandwidth_Hz**     = 5.97265078[kHz]  
**Irr_Dec_Bandwidth_Ppm**    = 12.07407703[ppm]  
**Irr_Dec_Freq**             = 495.13191398[MHz]  
**Irr_Dec_Merit_Factor**     = 2.2  
**Irr_Decoupling**           = TRUE  
**Irr_Noise**                = WALTZ  

---

![Diagram](S27)
Filename = YH(S-p-F)_19F-1-2.jdf
Author = delta
Experiment = single_pulse.jxp
Sample_Id = YH(S-p-F)
Solvent = BENZENE-D6
Actual_Start_Time =  3-APR-2020 20:06:10
Revision_Time =  7-APR-2020 14:31:23
Comment = single_pulse
Data_Format = 1D REAL
Dim_Size = 26214
X_Domain = Fluorine19
Dim_Title = Fluorine19
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ500R/S1
Field_Strength = 11.62926421[T] (500[M
X_Acq_Duration = 0.28311552[s]
X_Freq = 465.88941346[MHz]
X_Offset = -100[ppm]
X_Points = 32768
X_Sweep = 115.74074074[kHz]
X_Sweep_Clipped = 92.59259259[kHz]
Irr_Freq = 465.8941346[MHz]
Irr_Offset = 5[ppm]
Tri_Freq = 465.8941346[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 20.6[dC]
X_90_Width = 11[us]
X_Acq_Time = 0.28311552[s]
X_Angle = 45[deg]
X_Atn = 3.7[dB]
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]
OPPh₂

1h

--- PROCESSING PARAMETERS ---

Filename = YH(S-p-I)_Proton-1-2.
Author = delta
Experiment = proton.jxp
Sample_Id = YH(S-p-I)
Solvent = BENZENE-D₆
Actual_Start_Time = 3-APR-2020 19:08:47
Revision_Time = 7-APR-2020 09:56:14
Comment = single_pulse
Data_Format = 1D REAL
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JEOL-JECA-ECZ500R/S1
Field_Strength = 11.62926421[T] (500[MG]
X_Acq_Duration = 1.76422912[s]
X_Freq = 495.13191398[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.5668198[Hz]
X_Sweep = 9.28677563[kHz]
X_Sweep_Clipped = 7.42942051[kHz]
Irr_Freq = 495.13191398[MHz]
Irr_Offset = 5[ppm]
Tri_Freq = 495.13191398[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 26
Temp_Get = 19.1[°C]
X_Sweep_Width = 9.58[us]
X_Acq_Time = 1.76422912[s]
X_Angle = 45[°]
X_Pulse = 4.3[μs]
X_Pulse_Width = 4.79[μs]
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]
Preset_Time = 5[s]
### Data Format

<table>
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<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dim_Size</td>
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</tr>
<tr>
<td>X_Domain</td>
<td>31P</td>
</tr>
<tr>
<td>X_Freq</td>
<td>200.43293989 MHz</td>
</tr>
<tr>
<td>X_Offset</td>
<td>0 ppm</td>
</tr>
<tr>
<td>X_Points</td>
<td>32768</td>
</tr>
<tr>
<td>X_Prescans</td>
<td>4</td>
</tr>
<tr>
<td>X_Resolution</td>
<td>5.29819065 Hz</td>
</tr>
<tr>
<td>X_Sweep</td>
<td>173.61111111 kHz</td>
</tr>
<tr>
<td>X_Sweep_Clipped</td>
<td>138.88888889 kHz</td>
</tr>
<tr>
<td>Irr_Domain</td>
<td>Proton</td>
</tr>
<tr>
<td>Irr_Freq</td>
<td>495.13191398 MHz</td>
</tr>
<tr>
<td>Irr_Offset</td>
<td>5 ppm</td>
</tr>
<tr>
<td>Clipped</td>
<td>FALSE</td>
</tr>
<tr>
<td>Scans</td>
<td>64</td>
</tr>
</tbody>
</table>

### Processing Parameters

- sexp(2.0 [Hz], 0.0 [s])
- trapezoid(0[%), 0[%, 80[%, 100[%])
- zerofill(1, TRUE)
- fft(1, TRUE, TRUE)
- ppm
- phase(6.75, 0, 33.97551[%])
- base_correct(Akima, 5.0, FALSE, 3.0, None, Ya)

Derived from: YH(S-p-I)_31P_1Hdec-1-1.jdf
Filename = YH(CO2Me)_31P_1Hdec
Author = delta
Experiment = single_pulse_dec.j
Sample_Id = YH(CO2Me)
Solvent = BENZENE-D6
Actual_Start_Time = 2-APR-2020 19:16:
Revision_Time = 7-APR-2020 12:50:
Comment = single pulse decou
Data_Format = 1D REAL
Dim_Size = 26214
X_Domain = Phosphorus31
Dim_Title = Phosphorus31
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ500R/S1
Field_Strength = 11.62926421[T] (50
X_Acq_Duration = 0.18874368[s]
X_Domain = 31P
X_Freq = 200.43293989[MHz]
X_Offset = 0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 5.29819065[Hz]
X_Sweep = 173.61111111[kHz]
X_Sweep_Clipped = 138.88888889[kHz]
Irr_Domain = Proton
Irr_Freq = 495.13191398[MHz]
Irr_Offset = 5[ppm]
Irr_Offset_Clipped = FALSE
Scans = 64
Total_Scans = 64
Relaxation_Delay = 1[s]
Recvr_Gain = 66
Temp_Set = 20[°C]
X_BW_Width = 19.78[us]
X_Acq_Time = 0.18874368[s]
X_Angle = 30[deg]
X_Atn = 4[DB]
X_Pulse = 6.59333333[us]
Irr_Atn_Dec = 23.948[DB]
Irr_Atn_Dec_Calc = 23.948[DB]
Irr_Atn_Dec_Default_Calc = 23.948[DB]
Irr_Atn_How = 23.948[DB]
Irr_Dec_Bandwidth_Hz = 5.97620587[kHz]
Irr_Dec_Bandwidth_Ppm = 12.07407703[ppm]
Irr_Dec_Freq = 495.13191398[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Hue = TRUE
Irr_Noise = WALTZ
--- PROCESSING PARAMETERS ---
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%,] 0[%,] 80[%,] 100[%] )
sendfill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -30, 0, 50[°] )
base_correct( Allmax, 5, 0, FALSE, 3, None, Y)
Derived from: YH(CO2Me)_31P_1Hdec-1-1.jdf

X : parts per Million : Phosphorus31
Filename = YH(S-m-Me)_Carbon-
Author = delta
Experiment = carbon.jxp
Sample_Id = YH(S-m-Me)
Solvent = BENZENE-D6
Actual_Start_Time = 3-APR-2020 07:33:
Revision_Time = 9-APR-2020 13:49:
Comment = single pulse decou
Data_Format = 1D REAL
Dim_Size = 26214
X_Domain = Carbon13
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ500R/S1
Field_Strength = 11.62926421[T] (50
X_Acq_Duration = 0.8388608[s]
X_Domain = 13C
X_Freq = 124.5010059[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.1920929[Hz]
X_Sweep = 39.0625[kHz]
X_Sweep_Clipped = 31.25[kHz]
Irr_Domain = Proton
Irr_Freq = 495.13191398[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 5000
Total_Scans = 5000
Relaxation_Delay = 2[s]
Recvr_Gain = 56
Temp_Get = 20.4[°C]
X_Width = 9.61[us]
X_Angle = 30[°deg]
X_Atn = 0[°]
X_Pulse = 3.20333333[us]
Irr_Atn_Dec = 23.948[db]
Irr_Atn_Dec_Calc = 23.948[db]
Irr_Atn_Dec_Default_Calc = 23.948[db]
Irr_Atn_Hoe = 23.948[db]
Irr_Dec_Bandwidth_Bps = 5.9726287[kHz]
Irr_Dec_Bandwidth_Bps = 12.07407703[ppm]
Irr_Dec_Freq = 495.13191398[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noise = WALTZ

--- PROCESSING PARAMETERS ---
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
serrfill( 1, TRUE )
eff( 1, TRUE, TRUE )
machinephase
ppp
base_correct( Akima, 5, 0, FALSE, 5, None, FA
reference( 127.75835[ppm], 128.04[ppm] )
Derived from: YH(S-m-Me)_Carbon-1-1.jdf

Filename = YH(S-m-Me)_Carbon-
Author = delta
Experiment = carbon.jxp
Sample_Id = YH(S-m-Me)
Solvent = BENZENE-D6
Actual_Start_Time = 3-APR-2020 07:33:
Revision_Time = 9-APR-2020 13:49:
Comment = single pulse decou
Data_Format = 1D REAL
Dim_Size = 26214
X_Domain = Carbon13
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ500R/S1
Field_Strength = 11.62926421[T] (50
X_Acq_Duration = 0.8388608[s]
X_Domain = 13C
X_Freq = 124.5010059[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.1920929[Hz]
X_Sweep = 39.0625[kHz]
X_Sweep_Clipped = 31.25[kHz]
Irr_Domain = Proton
Irr_Freq = 495.13191398[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 5000
Total_Scans = 5000
Relaxation_Delay = 2[s]
Recvr_Gain = 56
Temp_Get = 20.4[°C]
X_Width = 9.61[us]
X_Angle = 30[°deg]
X_Atn = 0[°]
X_Pulse = 3.20333333[us]
Irr_Atn_Dec = 23.948[db]
Irr_Atn_Dec_Calc = 23.948[db]
Irr_Atn_Dec_Default_Calc = 23.948[db]
Irr_Atn_Hoe = 23.948[db]
Irr_Dec_Bandwidth_Bps = 5.9726287[kHz]
Irr_Dec_Bandwidth_Bps = 12.07407703[ppm]
Irr_Dec_Freq = 495.13191398[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noise = WALTZ

--- PROCESSING PARAMETERS ---
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
serrfill( 1, TRUE )
eff( 1, TRUE, TRUE )
machinephase
ppp
base_correct( Akima, 5, 0, FALSE, 5, None, FA
reference( 127.75835[ppm], 128.04[ppm] )
Derived from: YH(S-m-Me)_Carbon-1-1.jdf
<table>
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<th>X Domain</th>
<th>Phosphorus31</th>
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<tbody>
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<td>X Units</td>
<td>[ppm]</td>
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<tr>
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<td>11.62926421[T]</td>
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<tr>
<td>X Acq_Duration</td>
<td>0.18874368[s]</td>
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<tr>
<td>X Freq</td>
<td>200.43293989[MHz]</td>
</tr>
<tr>
<td>X Offset</td>
<td>0[ppm]</td>
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<tr>
<td>X Points</td>
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</tr>
<tr>
<td>X Resolution</td>
<td>5.29819065[Hz]</td>
</tr>
<tr>
<td>X Sweep</td>
<td>173.61111111[kHz]</td>
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<tr>
<td>X Sweep_Clipped</td>
<td>138.8888889[kHz]</td>
</tr>
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<td>Proton</td>
</tr>
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</tr>
<tr>
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<tr>
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<td>Total Scans</td>
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<tr>
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<td>Reconv_Gain</td>
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<tr>
<td>Temp_Gain</td>
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</tr>
<tr>
<td>X 90_Width</td>
<td>19.78[us]</td>
</tr>
<tr>
<td>X Acq_Time</td>
<td>0.18874368[s]</td>
</tr>
<tr>
<td>X Angle</td>
<td>30[°deg]</td>
</tr>
<tr>
<td>X Attn</td>
<td>4(4)[dB]</td>
</tr>
<tr>
<td>X Pulse</td>
<td>6.5933333333[us]</td>
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<tr>
<td>Irr Attn_Dec</td>
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<td>Irr Noise</td>
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<td>Irr Noise</td>
<td>WALTZ</td>
</tr>
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</table>

Filename = YH(S-m-Cl)_31P_1Hdec-1-1.jdf

OPPh₂

OPPh₂

Cl

11
Filename              = YH(OMO)_Proton-1-2.jd
Author                = delta
Experiment            = proton.jxp
Sample_Id             = YH(OMO)
Solvent               = BENZENE-D6
Actual_Start_Time     = 6-APR-2020 22:17:15
Revision_Time         = 7-APR-2020 10:19:25
Comment               = single_pulse
Data_Format           = 1D COMPLEX
Dim_Size              = 13107
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-EC500R/S1
Field_Strength        = 11.62926421[T] (500[M
X_Acq_Duration        = 1.76422912[s]
X_Domain              = 1H
X_Freq                = 495.13191398[MHz]
X_Offset              = 5[ppm]
X_Points              = 16384
X_Resolution          = 0.5668198[Hz]
X_Sweep               = 9.28677563[kHz]
X_Sweep_Clipped       = 7.42942051[kHz]
Irr_Domain            = Proton
Irr_Freq              = 495.13191398[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 495.13191398[MHz]
Tri_Offset            = 5[ppm]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8
Relaxation_Delay      = 5[s]
Recvr_Gain            = 36
Temp_Get              = 21.4[dc]
X_90_Width            = 9.58[us]
X_Acq_Time            = 1.76422912[s]
X_Angle               = 45[deg]
X_Pulse               = 4.79[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 500
Dante_Preset          = FALSE
Decimation_Rate       = 0
Initial_Wait          = 1[s]
Phase                 = 10, 90, 270, 180, 180
Preset_Time           = 5[s]
<table>
<thead>
<tr>
<th>X : parts per Million : Phosphorus31</th>
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</thead>
<tbody>
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<td>200.0</td>
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<tr>
<td>100.0</td>
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<td>114.352</td>
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</table>

## Experiment Details

- **Filename**: YH(S-OMO)_31P_1Hdec-1-1.jdf
- **Author**: delta
- **Experiment**: single_pulse_dec.j
- **Sample_Id**: YH(S-OMO)
- **Solvent**: BENZENE-D6
- **Actual_Start_Time**: 7-APR-2020 22:34:
- **Revision_Time**: 8-APR-2020 09:38:
- **Comment**: single pulse decou
- **Data_Format**: 1D REAL
- **Dim_Size**: 26214
- **X Domain**: Phosphorus31
- **Dim_Title**: Phosphorus31
- **Dim_Units**: [ppm]
- **Dimensions**: X
- **Spectrometer**: JNM-ECZ500R/S1
- **Field_Strength**: 11.62926421[T] (50)
- **X_Acq_Duration**: 0.18874368[s]
- **X_Freq**: 200.43293989[MHz]
- **X_Offset**: 0[ppm]
- **X_Points**: 32768
- **X_Prescans**: 4
- **X_Resolution**: 5.29819065[Hz]
- **X_Sweep**: 173.61111111[kHz]
- **X_Sweep_Clipped**: 138.88888889[kHz]
- **Irr_Freq**: 495.13191398[MHz]
- **Irr_Offset**: 5[ppm]
- **Irr_Points**: 5
- **Irr_Clipped**: FALSE
- **Scans**: 64
- **Total_Scans**: 64
- **Relaxation_Delay**: 1[s]
- **Recvr_Gain**: 66
- **Temp_Get**: 20.4[°C]
- **X_Width**: 19.78[us]
- **X_Acq_Time**: 0.18874368[s]
- **X_Angle**: 30[deg]
- **X_ATN**: 6.59333333[us]
- **Irr_ATN_Dec**: 23.948[db]
- **Irr_ATN_Dec_Calc**: 23.948[db]
- **Irr_Attn_Default_Calc**: 23.948[db]
- **Irr_Attn_Noe**: 23.948[db]
- **Irr_Dec_Bandwidth_Hz**: 5.97826087[kHz]
- **Irr_Dec_Bandwidth_Ppm**: 12.07407703[ppm]
- **Irr_Dec_Freq**: 495.13191398[MHz]
- **Irr_Dec_Merit_Factor**: 2.2
- **Irr_Decoupling**: TRUE
- **Irr_Noise**: WALTZ

--- PROCESSING PARAMETERS ---

- `sexp(2.0[Hz], 0.0[s])`
- `trapezoid(0[%, 0[%, 80[%, 100[%])`
- `zerofill(1, TRUE)
- `fft(1, TRUE, TRUE)`
- `machinephase`
- `phase(-43.60394, 0.0[°])`
- `base_correct(Akima, 5.0, FALSE, 3, None, FA)`

Derived from: YH(S-OMO)_31P_1Hdec-1-1.jdf
Filename                 = YH(6-qu)_Carbon-1-
Author                   = delta
Experiment               = carbon.jxp
Sample_Id                = YH(6-qu)
Solvent                  = CHLOROFORM-D
Actual_Start_Time        = 21-JUL-2020 01:20:
Revision_Time            = 22-JUL-2020 17:38:
Comment                  = single pulse decou
Data_Format              = 1D REAL
Data_Size                = 26214
Domain                 = Carbon13
Dim_Title                = Carbon13
Dim_Units                = [ppm]
Dimensions               = X
Spectrometer             = JNM-ECZ500R/S1
Field_Strength           = 11.62926421[T] (50
X_Acq_Duration           = 0.8388608[s]X_Domain                 = 13C
X_Freq                   = 124.5010059[MHz]
X_Offset                 = 100[ppm]
X_Points                 = 32768
X_Prescans               = 4
X_Resolution             = 1.1920929[Hz]
X_Sweep                  = 39.0625[kHz]X_Sweep_Clipped          = 31.25[kHz]
Irr_Domain               = Proton
Irr_Freq                 = 495.13191398[MHz]
Irr_Offset               = 5[ppm]
Clipped                  = FALSE
Scans                    = 1000
Total_Scans              = 1000
Relaxation_Delay         = 2[s]
Recvr_Gain               = 51
Recvr_Gain_Dc            = 61(A)
X_90_Width               = 9.61[us]
X_Acq_Time               = 0.8388608[s]
X_Angle                  = 30[deg]
X_Atn                    = 10[dB]
X_Pulse                  = 3.20333333[us]

---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%,] 0[%,] 80[%,] 100[%,] )
zerofill( 1, TRUE, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
base_correct( Akima, 5, 0, FALSE, 3, None, FA reference( 77.13498[ppm], 77.16[ppm] )
Derived from: YH(6-qu)_Carbon-1-1.jdf

Filename                 = YH(6-qu)_Carbon-
Author                   = delta
Experiment               = carbon.jxp
Sample_Id                = YH(6-qu)
Solvent                  = CHLOROFORM-D
Actual_Start_Time        = 21-JUL-2020 01:20:
Revision_Time            = 22-JUL-2020 17:38:
Comment                  = single pulse decou
Data_Format              = 1D REAL
Data_Size                = 26214
Domain                 = Carbon13
Dim_Title                = Carbon13
Dim_Units                = [ppm]
Dimensions               = X
Spectrometer             = JNM-ECZ500R/S1
Field_Strength           = 11.62926421[T] (50
X_Acq_Duration           = 0.8388608[s]X_Domain                 = 13C
X_Freq                   = 124.5010059[MHz]
X_Offset                 = 100[ppm]
X_Points                 = 32768
X_Prescans               = 4
X_Resolution             = 1.1920929[Hz]
X_Sweep                  = 39.0625[kHz]X_Sweep_Clipped          = 31.25[kHz]
Irr_Domain               = Proton
Irr_Freq                 = 495.13191398[MHz]
Irr_Offset               = 5[ppm]
Clipped                  = FALSE
Scans                    = 1000
Total_Scans              = 1000
Relaxation_Delay         = 2[s]
Recvr_Gain               = 51
Recvr_Gain_Dc            = 61(A)
X_90_Width               = 9.61[us]
X_Acq_Time               = 0.8388608[s]
X_Angle                  = 30[deg]
X_Atn                    = 10[dB]
X_Pulse                  = 3.20333333[us]

---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%,] 0[%,] 80[%,] 100[%,] )
zerofill( 1, TRUE, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
base_correct( Akima, 5, 0, FALSE, 3, None, FA reference( 77.13498[ppm], 77.16[ppm] )
Derived from: YH(6-qu)_Carbon-1-1.jdf

Filename                 = YH(6-qu)_Carbon-
Author                   = delta
Experiment               = carbon.jxp
Sample_Id                = YH(6-qu)
Solvent                  = CHLOROFORM-D
Actual_Start_Time        = 21-JUL-2020 01:20:
Revision_Time            = 22-JUL-2020 17:38:
Comment                  = single pulse decou
Data_Format              = 1D REAL
Data_Size                = 26214
Domain                 = Carbon13
Dim_Title                = Carbon13
Dim_Units                = [ppm]
Dimensions               = X
Spectrometer             = JNM-ECZ500R/S1
Field_Strength           = 11.62926421[T] (50
X_Acq_Duration           = 0.8388608[s]X_Domain                 = 13C
X_Freq                   = 124.5010059[MHz]
X_Offset                 = 100[ppm]
X_Points                 = 32768
X_Prescans               = 4
X_Resolution             = 1.1920929[Hz]
X_Sweep                  = 39.0625[kHz]X_Sweep_Clipped          = 31.25[kHz]
Irr_Domain               = Proton
Irr_Freq                 = 495.13191398[MHz]
Irr_Offset               = 5[ppm]
Clipped                  = FALSE
Scans                    = 1000
Total_Scans              = 1000
Relaxation_Delay         = 2[s]
Recvr_Gain               = 51
Recvr_Gain_Dc            = 61(A)
X_90_Width               = 9.61[us]
X_Acq_Time               = 0.8388608[s]
X_Angle                  = 30[deg]
X_Atn                    = 10[dB]
X_Pulse                  = 3.20333333[us]

---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%,] 0[%,] 80[%,] 100[%,] )
zerofill( 1, TRUE, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
base_correct( Akima, 5, 0, FALSE, 3, None, FA reference( 77.13498[ppm], 77.16[ppm] )
Derived from: YH(6-qu)_Carbon-1-1.jdf
Filename              = YH-54(p-MeO)_Proton-1-1.jdf
Author                = delta
Experiment            = proton.jxp
Sample_Id             = YH-54(p-MeO)
Solvent               = ACETONE-D6
Creation_Time         = 10-MAR-2020 22:02:19
Revision_Time         = 11-MAR-2020 12:52:33
Current_Time          = 11-MAR-2020 12:53:26
Comment               = single_pulse
Data_Format           = 1D REAL
Dim_Size              = 13107
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ500R/S1
Field_Strength        = 11.62926421[T] (500[MHz])
X_Acq_Duration        = 1.76422912[s]
X_Domain              = 1H
X_Freq                = 495.13191398[MHz]
X_Offset              = 5[ppm]
X_Points              = 16384
X_Prescans            = 1
X_Resolution          = 0.5668198[Hz]
X_Sweep               = 9.28677563[kHz]
X_Sweep_Clipped       = 7.42942051[kHz]
Irr_Domain            = Proton
Irr_Freq              = 495.13191398[MHz]
Irr_Offset            = 5[ppm]
Irr_Prescans          = 1
Irr_Resolution        = 0.5668198[Hz]
Irr_Sweep             = 9.28677563[kHz]
Irr_Sweep_Clipped     = 7.42942051[kHz]
Tri_Domain            = Proton
Tri_Freq              = 495.13191398[MHz]
Tri_Offset            = 5[ppm]
Tri_Prescans          = 1
Tri_Resolution        = 0.5668198[Hz]
Tri_Sweep             = 9.28677563[kHz]
Tri_Sweep_Clipped     = 7.42942051[kHz]
Clipped               = FALSE
Decimation_Reg        = r: 1346(1345), g: 52
Scans                 = 8
Total_Scans           = 8
Relaxation_Delay      = 5[s]
Recvr_Gain            = 56
Temp_Set              = 21.8[°C]
X_90_Width            = 7.12[us]
X_Angle               = 45[deg]
X_Atn                 = 3.3[dB]
X_Pulse               = 3.56[us]
Irr_Mode              = Off
Tri_Mode              = Off
Comment_1             = *** Pulse ***
Comment_11            = *** preat_time ***
Comment_201           = *** obs_date, preaturation
Comment_202           = *** irr, preaturation
Comment_203           = *** tri, preaturation
Filename              = YH-189(p-F)_19F-1-12.
Author                = delta
Experiment            = single_pulse.jxp
Sample_Id             = YH-189(p-F)
Solvent               = ACETONE-D6
Creation_Time         = 22-FEB-2020 16:34:26
Revision_Time         = 7-MAR-2020 16:49:36
Current_Time          = 7-MAR-2020 16:50:05
Comment               = single_pulse
Data_Format           = 1D REAL
Dim_Size              = 26214
Dim_Title             = Fluorine19
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-EC500R/S1
Field_Strength        = 11.62926421[T] (500[M
X_Acq_Duration        = 0.28311552[s]
X_Domain              = 19F
X_Freq                = 465.88941346[MHz]
X_Offset              = -100[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 3.5321721[Hz]
X_Sweep               = 115.74074074[kHz]
X_Sweep_Clipped       = 92.59259259[kHz]
X_Points              = 32768
X_Prescans            = 1
Irr_Domain            = Fluorine19
Irr_Freq              = 465.88941346[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Fluorine19
Tri_Freq              = 465.88941346[MHz]
Tri_Offset            = 5[ppm]
Clipped               = FALSE
Decimation_Reg        = z: 54 (53), g: 29
Scans                 = 8
Total_Scans           = 8
Relaxation_Delay      = 5[s]
Recvr_Gain            = 56
Temp_Set              = 21.9[dc]
X_90_Width            = 7.81[us]
X_Acq_Duration        = 0.28311552[s]
X_Angle               = 45[deg]
X_Atn                 = 3.7[db]
X_Pulse               = 3.905[us]
Irr_Mode              = Off
Tri_Mode              = Off
Comment_1             = *** Pulse ***
Comment_111           = *** preat_time ***
Comment_201           = *** obs_date_presatu
Comment_202           = *** irr_presaturation
Comment_203           = *** irr_presaturation

--- PROCESSING PARAMETERS ---
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%, 0[%, 80[%, 100[%] )
szerfill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -1.25, -21, 64.94487[%] )
base_correct( None, 0, Smooth )

以下为数据： YH-189(p-F)_19F-1-1.jdf

Filename              = YH-189(p-F)_19F-1-12.
Author                = delta
Experiment            = single_pulse.jxp
Sample_Id             = YH-189(p-F)
Solvent               = ACETONE-D6
Creation_Time         = 22-FEB-2020 16:34:26
Revision_Time         = 7-MAR-2020 16:49:36
Current_Time          = 7-MAR-2020 16:50:05
Comment               = single_pulse
Data_Format           = 1D REAL
Dim_Size              = 26214
Dim_Title             = Fluorine19
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-EC500R/S1
Field_Strength        = 11.62926421[T] (500[M
X_Acq_Duration        = 0.28311552[s]
X_Domain              = 19F
X_Freq                = 465.88941346[MHz]
X_Offset              = -100[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 3.5321721[Hz]
X_Sweep               = 115.74074074[kHz]
X_Sweep_Clipped       = 92.59259259[kHz]
X_Points              = 32768
X_Prescans            = 1
Irr_Domain            = Fluorine19
Irr_Freq              = 465.88941346[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Fluorine19
Tri_Freq              = 465.88941346[MHz]
Tri_Offset            = 5[ppm]
Clipped               = FALSE
Decimation_Reg        = z: 54 (53), g: 29
Scans                 = 8
Total_Scans           = 8
Relaxation_Delay      = 5[s]
Recvr_Gain            = 56
Temp_Set              = 21.9[dc]
X_90_Width            = 7.81[us]
X_Acq_Duration        = 0.28311552[s]
X_Angle               = 45[deg]
X_Atn                 = 3.7[db]
X_Pulse               = 3.905[us]
Irr_Mode              = Off
Tri_Mode              = Off
Comment_1             = *** Pulse ***
Comment_111           = *** preat_time ***
Comment_201           = *** obs_date_presatu
Comment_202           = *** irr_presaturation
Comment_203           = *** irr_presaturation

--- PROCESSING PARAMETERS ---
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%, 0[%, 80[%, 100[%] )
szerfill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -1.25, -21, 64.94487[%] )
base_correct( None, 0, Smooth )

以下为数据： YH-189(p-F)_19F-1-1.jdf
### Processing Parameters

- **Filename**: YH-170-(p-Br)_Proton-1-1.jdf
- **Author**: delta
- **Experiment**: proton.jxp
- **Sample_Id**: YH-170-(p-Br)
- **Solvent**: ACETONE-D6
- **Actual_Start_Time**: 21-FEB-2020 18:30:09
- **Comment**: single_pulse
- **Data_Format**: 1D REAL
- **Dim_Size**: 104858
- **Dim_Title**: Proton
- **Dim_Units**: [ppm]
- **Dimensions**: X
- **Spectrometer**: JNM-ECX500R/S1
- **Field_Strength**: 11.62926421[T] (500[MHz])
- **X_Acq_Duration**: 14.11383296[s]
- **X_Domain**: Proton
- **X_Freq**: 495.13191398[MHz]
- **X_Offset**: 5[ppm]
- **X_Points**: 131072
- **X_Prescans**: 1
- **X_Resolution**: 70.85247522[mHz]
- **X_Sweep**: 9.28677563[kHz]
- **X_Sweep_Clipped**: 7.42942051[kHz]
- **Irr_Domain**: Proton
- **Irr_Freq**: 495.13191398[MHz]
- **Irr_Offset**: 5[ppm]
- **Tri_Domain**: Proton
- **Tri_Freq**: 495.13191398[MHz]
- **Tri_Offset**: 5[ppm]
- **Clipped**: FALSE
- **Scans**: 8
- **Total_Scans**: 8
- **Relaxation_Delay**: 3[s]
- **Recvr_Gain**: 66
- **Temp_Get**: 21.9[°C]
- **X_BC_Width**: 7.12[us]
- **X_Acq_Time**: 14.11383296[s]
- **X_Angle**: 45[°]
- **X_Atn**: 3.3[db]
- **X_Pulse**: 3.56[us]
- **Irr_Mode**: Off
- **Tri_Mode**: Off
- **Dante_Loop**: 299
- **Dante_Preset**: FALSE
- **Decimation_Rate**: 0
- **Initial_Wait**: 1[s]
- **Phase**: 10, 90, 270, 180, 180
- **Preset_Time**: 3[s]

---

**Data Table**

<table>
<thead>
<tr>
<th>X (ppm)</th>
<th>Proton</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.584</td>
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<tr>
<td>7.561</td>
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<tr>
<td>7.539</td>
<td></td>
</tr>
<tr>
<td>7.516</td>
<td></td>
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<tr>
<td>7.357</td>
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<tr>
<td>7.035</td>
<td></td>
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<tr>
<td>7.030</td>
<td></td>
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<tr>
<td>7.018</td>
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<tr>
<td>7.012</td>
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<tr>
<td>6.485</td>
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<td>6.468</td>
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<tr>
<td>2.835</td>
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<td>2.802</td>
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<td>2.059</td>
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<td>2.056</td>
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<td>2.046</td>
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<tr>
<td>2.043</td>
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</tr>
<tr>
<td>2.041</td>
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</tr>
</tbody>
</table>

**Diagram**

[Diagram of chemical structure with peaks identified]
### Processing Parameters

- Filename: YH-170-(p-Br)_Carbon13.jdf
- Author: delta
- Experiment: carbon.jxp
- Sample_Id: YH-170-(p-Br)
- Solvent: ACETONE-D6
- Actual_Start_Time: 22-FEB-2020 00:05:
- Revision_Time: 9-APR-2020 14:25:
- Comment: single pulse decou
- Data_Format: 1D REAL
- Dim_Size: 26214
- Dim_Title: Carbon13
- Dim_Units: [ppm]
- Dimensions: X
- Spectrometer: JNM-ECZ500R/S1
- Field_Strength: 11.62926421[T] (50 T)
- X_Acq_Duration: 0.8388608[s]
- X_Domain: 13C
- X_Freq: 124.5010059[MHz]
- X_Offset: 100[ppm]
- X_Points: 32768
- X_Prescans: 4
- X_Resolution: 1.1920929[Hz]
- X_Sweep: 39.0625[kHz]
- X_Sweep_Clipped: 31.25[kHz]
- Irr_Domain: Proton
- Irr_Freq: 495.13191398[MHz]
- Irr_Offset: 5[ppm]
- Irr_Atn_Dec: 26.4[db]
- Irr_Atn_Dec_Calc: 26.4[db]
- Irr_Atn_Dec_Default_Calc: 26.4[db]
- Irr_Atn_Dec_Noe: 26.4[db]
- Irr_Dec_Bandwidth_Hz: 5.97826087[kHz]
- Irr_Dec_Bandwidth_Ppm: 12.07407703[ppm]
- Irr_Dec_Freq: 495.13191398[MHz]
- Irr_Dec_Merit_Factor: 2.2
- Irr_Decoupling: TRUE
- Irr_Noe: TRUE
- Irr_Noise: WALTZ
- Derived from: YH-170-(p-Br)_Carbon13-1-1.jdf

---

### Table of Values

<table>
<thead>
<tr>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>206.065</td>
</tr>
<tr>
<td>206.307</td>
</tr>
<tr>
<td>206.815</td>
</tr>
<tr>
<td>208.306</td>
</tr>
<tr>
<td>208.991</td>
</tr>
<tr>
<td>210.096</td>
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<td>211.699</td>
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<tr>
<td>213.991</td>
</tr>
<tr>
<td>216.554</td>
</tr>
<tr>
<td>219.096</td>
</tr>
</tbody>
</table>

---

### Diagram

![Diagram](image-url)

- OH
- Br
- BF₃K
- 2g

---

### Carbon-13 Spectrogram

- X: parts per Million: Carbon13
- S65
Filename              = YH-170-(p-Br)_19F-1-1
Author                = delta
Experiment            = single_pulse.jxp
Sample_Id             = YH-170-(p-Br)
Solvent               = ACETONE-D6
Creation_Time         = 21-FEB-2020 18:36:18
Revision_Time         = 22-FEB-2020 19:06:16
Current_Time          = 22-FEB-2020 19:06:53
Comment               = single_pulse
Data_Format           = 1D REAL
Dim_Size              = 26214
Dim_Title             = Fluorine19
Dimensions            = X
Spectrometer          = JNM-ECZ500R/S1
Field_Strength        = 11.62926421[T] (500[M
X_Acq_Duration        = 0.28311552[s]
X_Domain              = 19F
X_Freq                = 465.88941346[MHz]
X_Offset              = -100[ppm]
X_Points              = 32768
X_Sweep_05            = 115.74074074[kHz]
X_Sweep_05_Clipped    = 92.59259259[kHz]
Irr_Domain            = Fluorine19
Irr_Freq              = 465.88941346[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Fluorine19
Tri_Freq              = 465.88941346[MHz]
Tri_Offset            = 5[ppm]
Clipped               = FALSE
Decimation_Reg        = r: 54( 53),g: 29
Scans                 = 8
Total_Scans           = 8
Relaxation_Delay      = 5[s]
Recvr_Gain            = 56
Temp_Set              = 21.1[°C]
X_90_Width            = 7.81[us]
X_Acq_Time            = 0.28311552[s]
X_Circle              = 45[°]
X_Angle               = 3.7[°]
X_Pulse               = 3.905[us]
Irr_Mode              = Off
Tri_Mode              = Off
Comment_1             = *** Pulse ***
Comment_11            = *** presat_time ***
Comment_201           = *** obs_date_presetu
Comment_202           = *** irr_preseturation
Comment_203           = *** irr_preseturation
--- PROCESSING PARAMETERS ---
sexp( 0.2Hz, 0.0[s] )
trapezoid( 0[%, 0[%, 80[%, 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -1.90826, 0, 50[%] )
base_correct( None, 0, Smooth )

以下为来自：YH-170-(p-Br)_19F-1-1.jdf
<table>
<thead>
<tr>
<th>Parts per Million</th>
<th>Fluorine 19</th>
</tr>
</thead>
<tbody>
<tr>
<td>-137.0</td>
<td>-137.650</td>
</tr>
<tr>
<td>-137.650</td>
<td>-137.741</td>
</tr>
<tr>
<td>-137.854</td>
<td>-137.968</td>
</tr>
<tr>
<td>-138.067</td>
<td>-152.016</td>
</tr>
</tbody>
</table>

**Filename:** YH-14(p-I)_19F-1-1.jdf

**Author:** delta

**Experiment:** single_pulse.jxp

**Sample_Id:** YH-14(p-I)

**Solvent:** ACETONE-D6

**Creation_Time:** 22-FEB-2020 13:51:46

**Revision_Time:** 22-FEB-2020 19:23:29

**Current_Time:** 22-FEB-2020 19:23:43

**Comment:** single_pulse

**Data_Format:** 1D REAL

**Dim_Size:** 26214

**Dim_Title:** Fluorine19

**Dim_Units:** [ppm]

**Dimensions:** X

**Spectrometer:** JNM-ECZ500R/S1

**Field_Strength:** 11.62926421 [T] (500 [MHz])

**X_Acq_Duration:** 0.28311552 [s]

**X_Domain:** 19F

**X_Freq:** 465.88941346 [MHz]

**X_Offset:** -100 [ppm]

**X_Points:** 32768

**X_Prescans:** 1

**X_Resolution:** 3.5321271 [MHz]

**X_Sweep:** 115.74074074 [kHz]

**X_Sweep_Clipped:** 92.59259259 [kHz]

**Irr_Domain:** Fluorine19

**Irr_Freq:** 465.88941346 [MHz]

**Irr_Offset:** 5 [ppm]

**Tri_Domain:** Fluorine19

**Tri_Freq:** 465.88941346 [MHz]

**Tri_Offset:** 5 [ppm]

**Clipped:** FALSE

**Decimation_Reg:** r: 54(53), g: 29

**Scans:** 8

**Total_Scans:** 8

**Relaxation_Delay:** 5 [s]

**Recvr_Gain:** 56

**Temp_Get:** 21.9 [dC]

**X_Angle:** 45 [deg]

**X_Atn:** 3.7 [dB]

**X_Pulse:** 3.905 [us]

**Tri_Mode:** Off

**Irr_Mode:** Off

**Comment_1:** *** Pulse ***

**Comment_111:** *** presat_time ***

**Comment_201:** *** obs_date_presetu

**Comment_202:** *** irr_preseturation

**Comment_203:** *** tri_preseturation

--- PROCESSING PARAMETERS ---

sexp(0.2 [Hz], 0.0 [s])
trapezoid(0 [us], 0 [us], 80 [us], 100 [us])
somefill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
phase(-0.73926, 0, 50 [s])
base_correct(None, 0, Smooth)

以下の内容：YH-14(p-I)_19F-1-1.jdf

S70
Filename = YH(m-OMe)_11B_1Hdec
Author = delta
Experiment = single_pulse_dec.j
Sample_Id = YH(m-OMe)
Solvent = CHLOROFORM-D
Actual_Start_Time = 8-APR-2020 10:34:
Revision_Time = 8-APR-2020 12:00:
Comment = single pulse decou
Data_Format = 1D REAL
Dim_Size = 26214
Dim_Title = Boron11
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ500R/S1
Field_Strength = 11.62926421[T] (50
X_Acq_Duration = 0.66060288[s]
X_Offset = 0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.51376876[Hz]
X_Sweep = 49.6031746[kHz]
X_Sweep_Clipped = 39.68253968[kHz]
Irr_Domain = Proton
Irr_Freq = 495.13191398[MHz]
Irr_Offset = 5[ppm]
Irr_Sweeps = 300
Irr_Clipped = FALSE
Irr_Noise = WALTZ
Irr_Atn_Dec = 23.948[db]
Irr_Atn_Dec_Default_Calc = 23.948[db]
Irr_Atn_Dec_Calc = 23.948[db]
Relaxation_Delay = 2[s]
Recvr_Gain = 56
Temp_Cel = 20.8[°C]
X_Angle = 30[deg]
X_Atn = 4[db]
X_Pulse = 12.05666667[us]
X_90_Width = 36.17[us]
X_Acn_Time = 0.66060288[s]
X_Prescans = 4
X_Atn_Dec = 23.948[db]
X_Atn_Dec_Default_Calc = 23.948[db]
X_Atn_Dec_Calc = 23.948[db]
Irr_Atn_Dec = 23.948[db]
Irr_Atn_Dec_Default_Calc = 23.948[db]
Irr_Atn_Dec_Calc = 23.948[db]
Irr_Atn_Noise = 2.2
Irr_Decoupling = TRUE
Irr_Noise = WALTZ

--- PROCESSING PARAMETERS ---
samp( 2.0[Hz], 0.0[ms] )
trapezoid( 0[%, 0[%, 80[%, 100[%] )
serrf1( 1, TRUE )
eft( 1, TRUE, TRUE )
machinephase
pp
phase( 17.25, 0, 38.11849[%) )
base_correct( Akima, 5, 0, FALSE, 3, None, YA
Derived from: YH(m-OMe)_11B_1Hdec-1-1.jdf

--- S2 ---

X : parts per Million : Boron11
--- PROCESSING PARAMETERS ---

sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase

ppm

Derived from: YH-295-f1_Proton-1-1.jdf

-- EXPERIMENTAL SETUP --

Filename = YH-295-f1_Proton-1-2.
Author = delta
Experiment = proton.jxp
Sample_Id = YH-295-f1
Solvent = CHLOROFORM-D
Actual_Start_Time = 2-APR-2020 21:18:43
Revision_Time = 7-APR-2020 10:29:22
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-EC500/S1
Field_Strength = 11.62926421[T] (500[MHz])
X_Acq_Duration = 1.76422912[s]
X_Domain = 1H
X_Freq = 495.13191398[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Sweeps = 1
X_Sweep = 9.28677563[kHz]
X_Sweep_Clip = 7.42942051[kHz]
Irr_Freq = 495.13131938[MHz]
Irr_Offset = 5[ppm]
Tri_Freq = 495.13131938[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 36
Temp_Get = 20.8[deg]
X_90_Width = 9.58[us]
X_Angle = 45[deg]
X_Atn = 4.3[db]
X_Pulse = 4.79[us]
Irr_Mode = off
Tri_Mode = off
Dante_Loop = 500
Dante_Preset = FALSE
Decimation_Rate = 0
Initial_Wait = 10, 90, 270, 180
Acq_Sample = 10, 90, 270, 180
Preset_Time = 5[s]
Filename                 = YH-295-f1_11B_1Hdec-1-1.jdf
Author                   = delta
Experiment               = single_pulse_dec.j
Sample_Id                = YH-295-f1
Solvent                  = CHLOROFORM-D
Actual_Start_Time        = 3-APR-2020 13:04:
Revision_Time            = 7-APR-2020 13:01:
Comment                  = single pulse decou
Data_Format              = 1D REAL
Dim_Size                 = 26214
X_Domain                 = Boron11
Dim_Title                = Boron11
Dim_Units                = [ppm]
Dimensions               = X
Spectrometer             = JNM-ECZ500R/S1
Field_Strength           = 11.62926421[T] (50
X_Acq_Duration           = 0.66060288[s]
X_Domain                 = 11B
X_Offset                 = 0[ppm]
X_Points                 = 32768
X_Freq                   = 158.85814241[MHz]
X_Prescans               = 4
X_RESOLUTION             = 1.51376876[Hz]
X_Sweep                  = 49.6031746[kHz]
X_Sweep_Clipped          = 39.68253968[kHz]
Irr_Domain               = Proton
Irr_Freq                 = 495.13191398[MHz]
Irr_Offset               = 5[ppm]
Irr_Sweep_Clipped        = TRUE
Scans                    = 500
Total_Scans              = 500
Relaxation_Delay         = 2[s]
Bcev_Gain                = 50
Temp_Get                 = 20.6[DC]
X_90_Width               = 36.17[us]
X_Acq_Time               = 0.66060288[s]
X_Angle                  = 30[deg]
X_Atn                    = 120[deg]
X_Pulse                  = 12.05666667[us]
Irr_Atn_Dec              = -2.948[db]
Irr_Atn_Dec_Calc         = 23.948[db]
Irr_Atn_Dec_Default_Calc = 23.948[db]
Irr_Atn_Noe              = 23.948[db]
Irr_Dec_Bandwidth_Hz     = 5.9726087[kHz]
Irr_Dec_Bandwidth_Ppm    = 12.07407703[ppm]
Irr_Dec_Freq             = 495.13191398[MHz]
Irr_Dec_Merit_Factor     = 2.2
Irr_Decoupling           = TRUE
Irr_Noise                = WALTZ
Filename                 = YH-295-f2_11B_1Hdec-1-1.jdf
Author                   = delta
Experiment               = single_pulse_dec.j
Sample_Id                = YH-295-f2
Solvent                  = CHLOROFORM-D
Actual_Start_Time        = 3-APR-2020 13:34:
Revision_Time            = 7-APR-2020 12:59:
Comment                  = single pulse decou
Data_Format              = 1D REAL
Dim_Size                 = 26214
X_Domain                 = Boron11
Dim_Title                = Boron11
Dim_Units                = [ppm]
Dimensions               = X
Spectrometer             = JNM-ECZ500R/S1
Field_Strength           = 11.62926421[T] (50
X_Acq_Duration           = 0.66060288[s]
X_Offset                 = 0[ppm]
X_Domain                 = 11B
X_Freq                   = 158.85814241[MHz]
X_Points                 = 32768
X_Prescans               = 4
X_Resolution             = 1.51376876[Hz]
X_Sweep                  = 49.6031746[kHz]
X_Sweep_Clipped          = 39.68253968[kHz]
Irr_Domain               = Protonirr
Irr_Freq                 = 495.13191398[MHz]
Irr_Offset               = 5[ppm]
Irr_Sweep_Clipped        = FALSE
Scans                    = 500
Total_Scans              = 500
Relaxation_Delay         = 2[s]
Recvr_Gain               = 56
Temp_Get                 = 21[°C]
X_Pulse                  = 36.17[us]
X_Angle                  = 27.84577%X
X_Pulse                  = 12.0566667[us]
Irr_Dec_Freq             = 495.13191398[MHz]
Irr_Dec_Noise            = WALTZ
--- PROCESSING PARAMETERS ---
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 90[%,] 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 15, 0, 38.85477[°] )
base_correct( Akima, 5, 0, FALSE, 3, None, FA
Derived from: YH-295-f2_11B_1Hdec-1-1.jdf

--- PROCESSING PARAMETERS ---
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 15, 0, 38.85477[°] )
base_correct( Akima, 5, 0, FALSE, 3, None, FA
Derived from: YH-295-f2_11B_1Hdec-1-1.jdf

X : parts per Million : Boron11

O
O
O
Bpin
PPh2
BH3

3n
<table>
<thead>
<tr>
<th>X (ppm)</th>
<th>Carbon13</th>
</tr>
</thead>
<tbody>
<tr>
<td>158.661</td>
<td>158.661</td>
</tr>
<tr>
<td>146.061</td>
<td>146.061</td>
</tr>
<tr>
<td>143.724</td>
<td>143.724</td>
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<tr>
<td>133.441</td>
<td>133.441</td>
</tr>
<tr>
<td>133.087</td>
<td>133.087</td>
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<tr>
<td>128.347</td>
<td>128.347</td>
</tr>
<tr>
<td>120.093</td>
<td>120.093</td>
</tr>
<tr>
<td>106.229</td>
<td>106.229</td>
</tr>
</tbody>
</table>

**Dimensions:**
- X

**Spectrometer:**
- JNM-ECZ500R/S1

**Field Strength:**
- 11.62926421 [T]

**X_Acq_Duration:**
- 0.8388608 [s]

**X_Freq:**
- 124.5010059 [MHz]

**X_Offset:**
- 100 [ppm]

**X_Points:**
- 32768

**X_Prescans:**
- 4

**X_Resolution:**
- 1.1920929 [Hz]

**X_Sweep:**
- 39.0625 [kHz]

**Irr_Domain:**
- Proton

**Irr_Freq:**
- 495.13191398 [MHz]

**Irr_Offset:**
- 5 [ppm]

**Irr_Decoupling:**
- TRUE

**Irr_Noise:**
- WALTZ

--- PROCESSING PARAMETERS ---

Sweep: 2.0 [Hz], 0.0 [s]
Trapezoid: 0 [%, 0 [%, 80 [%, 100 [%]]
zerofill: 1, TRUE, TRUE
ft: 1, TRUE, TRUE
machinephase
ppm
base_correct: Akima, 5, 0, FALSE, 3, None, FA
Derived from: YH-340_Carbon-2-1.jdf
Filename              = YH-240_19F-1-2.jdf
Author                = delta
Experiment            = single_pulse.jxp
Sample_Id             = YH-240
Solvent               = DMSO-D6
Actual_Start_Time     = 22-JUL-2020 13:18:55
Revision_Time         = 22-JUL-2020 17:44:00
Comment               = single_pulse
Data_Format           = 1D REAL
Dim_Size              = 26214
Domain               = Fluorine19
Dim_Title             = Fluorine19
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ500R/S1
Field_Strength        = 11.62926421[T] (500[M
X_Acq_Duration        = 0.28311552[s]
X_Freq                = 465.88941346[MHz]
X_Offset              = -100[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 3.5321271[Hz]
X_Sweep               = 115.74074074[kHz]
X_Sweep_Clipped       = 92.59259259[kHz]
Irr_Domain            = Fluorine19
Irr_Freq              = 465.88941346[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Fluorine19
Tri_Freq              = 465.88941346[MHz]
Tri_Offset            = 5[ppm]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8
Relaxation_Delay      = 5[s]
Decimation_Rate       = 0
Initial_Wait          = 1[s]
Freq_Time             = 5[s]

--- PROCESSING PARAMETERS ---
sexp( 0.2[Hz], 0.0[ms] )
trapezoid( 0[%, 80[%, 100[%] )
serrfill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
base_correct( Akima, 0, OFF, 3, None, FALSE )
Derived from: YH-240_19F-1-1.jdf

--- PROCESSING PARAMETERS ---
sexp( 0.2[Hz], 0.0[ms] )
trapezoid( 0[%, 80[%, 100[%] )
serrfill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
base_correct( Akima, 0, FALSE, 3, None, FALSE )
Derived from: YH-240_19F-1-1.jdf

X : parts per Million : Fluorine19
In the 1H NMR of the separately prepared Pro-H, these peaks appear in an integration ratio of a:b = 1:12.9.

1H of Pro-H

12H of (Pro-H + Pro-D)
S97