

Solid-State Mechanochemistry for the Rapid and Efficient Synthesis of Tris-Cyclometalated Iridium(III) Complexes

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1. Chemicals and Instrumentation

The starting materials were obtained from commercial suppliers and used as received. 2-Aryl pyridine ligands (**2b–2i**) were prepared according to the reported procedures.¹ Solvents were purchased from commercial suppliers and further dried over molecular sieve (MS 4Å). All mechanochemical reactions were carried out using grinding vessels in a Retsch MM400 mill (Figure S1). Both jars (1.5 mL, 5.0 mL) and balls (5 mm, 10 mm) are made of stainless-steel (SUS400B and SUS420J2, respectively) (Figure S2). The heat gun Takagi HG-1450B with temperature control function was used for high-temperature ball-milling reactions (Figure S3). NMR spectra were recorded on JEOL JNM-EC X400P and JNM-ECS400 spectrometers (¹H: 392 or 396 or 399 or 401 MHz, ¹³C: 99 or 100 MHz). Tetramethylsilane (¹H), CDCl₃ (¹³C) was employed as external standards, respectively. Multiplicity was recorded as follows: s = singlet, brs = broad singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sept = septet, o = octet, m = multiplet. Thermography was recorded with an NEC Avio Thermo GEAR G120. High-resolution mass spectra were recorded at the Global Facility Center, Hokkaido University.



Figure S1. Retsch MM400 used in this study.

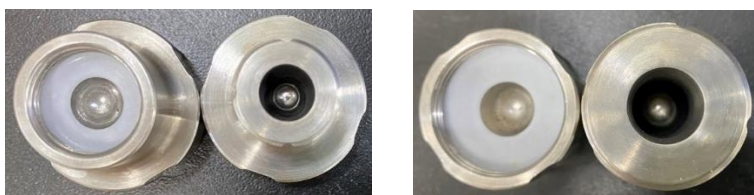


Figure S2. Stainless jar and ball used in this study. (Left : 1.5 ml jar and 5 mm ball, Right : 5.0 ml jar and 10 mm ball)

2. Details of High-Temperature Ball-Milling Method

The heat gun was fixed with clamps and placed directly above the ball milling jar (distance between the heat gun and ball milling jar: ca. 1 cm) (Figure S3).² The mechanochemical reactions were conducted while applying heated air to the outside of the milling jar (the preset temperature at 300 °C). The temperature inside the milling jar after the mechanochemical reactions was confirmed by observation with a thermography camera immediately after the milling jar (approximately 135 °C) (Figure S4).

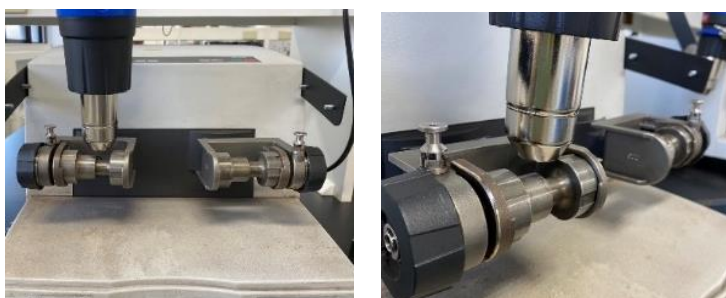


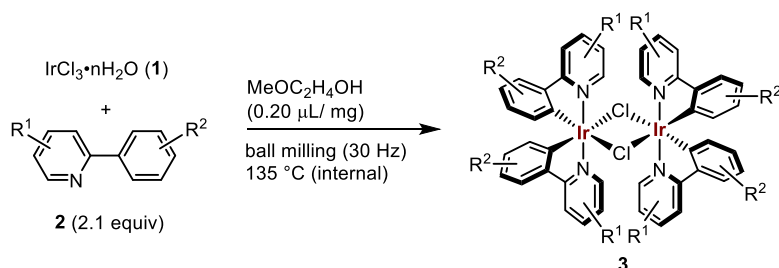
Figure S3. The set-up procedure for a heat gun on MM400.



Figure S4. Thermography images.

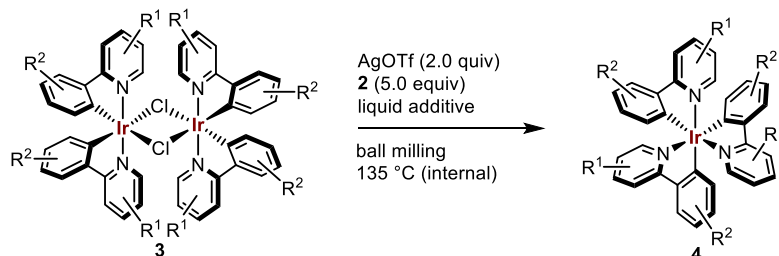
3. General Procedure for Solid-State Synthesis of Iridium(III) Complexes

1) Procedure A: solid-state synthesis of chloride-bridged dimer **3**



Iridium(III) chloride hydrate **1** (0.20 mmol), 2-aryl pyridine ligand **2** (0.42 mmol, 2.1 equiv), and 2-methoxyethanol (0.20 $\mu\text{L/mg}$) were placed in a ball milling vessel (stainless-steel, 1.5 mL) loaded with one grinding ball (stainless-steel, diameter: 5 mm). After being closed, the vessel placed in the ball mill machine (Retsch MM400, 10 or 30 min at 30 Hz) with a heat gun (the preset temperature at 300 $^\circ\text{C}$). After ball milling, the jar was then cooled rapidly with cold water and opened. The mixture was filtered by H_2O and CH_2Cl_2 to give the corresponding chloride-bridged dimer **3**. Because the low solubility of **3** in organic solvent, the crude product **3** was used in the next step without further purification.

2) Procedure B: solid-state synthesis of tris-cyclometalated iridium(III) complexes **4**



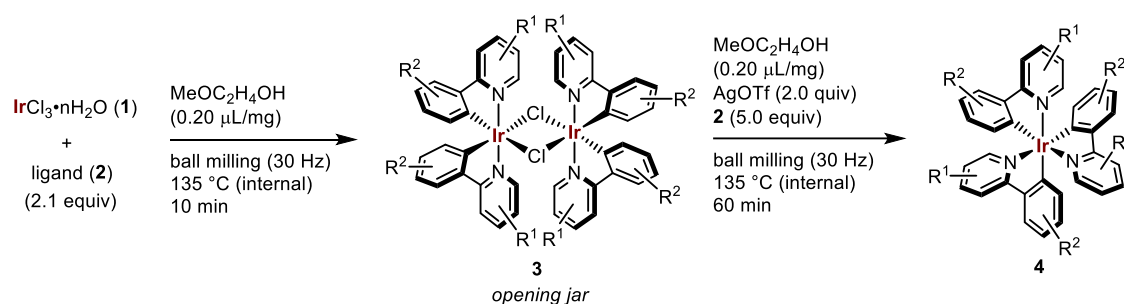
Method 1 (2-methoxyethanol was used as a liquid additive)

3 (1.0 equiv), **2** (5.0 equiv), AgOTf (2.0 equiv) and 2-methoxyethanol (0.20 $\mu\text{L/mg}$) were placed in a ball milling vessel (stainless-steel, 1.5 mL) loaded with one grinding ball (stainless-steel, diameter: 5 mm). After being closed, the vessel placed in the ball mill machine (Retsch MM400, 60 or 90 min at 30 Hz) with a heat gun (the preset temperature at 300 $^\circ\text{C}$). After ball milling, the jar was then cooled rapidly with cold water and opened. The mixture was filtered by CH_2Cl_2 to remove silver salts. The crude mixture was then purified by flash column chromatography (SiO_2 , typically CH_2Cl_2 /hexane, typically 0:100-100:0) or reprecipitation from CH_2Cl_2 /hexane to give the corresponding tris-cyclometalated iridium(III) complexes **4**.

Method 2 (DMF was used as a liquid addvie)

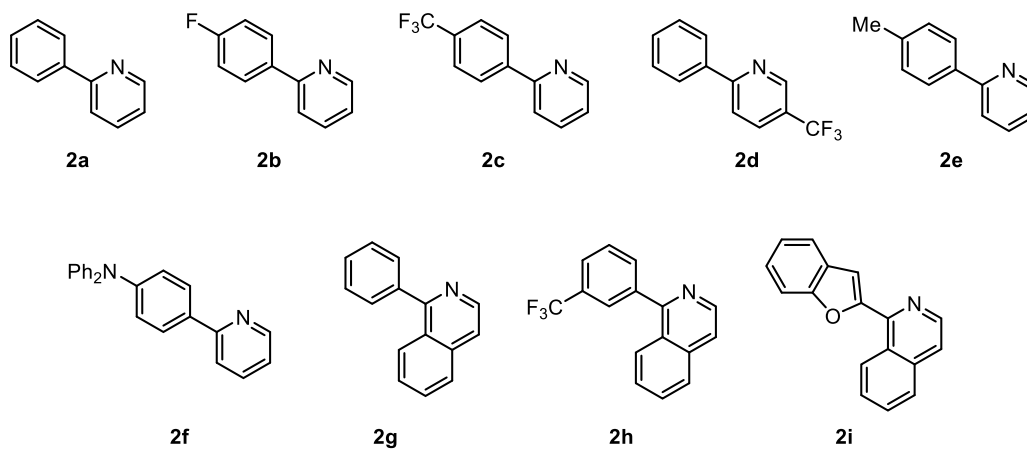
3 (1.0 equiv), **2** (5.0 equiv), AgOTf (2.0 equiv) and *N,N*-dimethylformamide (DMF) (0.50 $\mu\text{L}/\text{mg}$) were placed in a ball milling vessel (stainless, 5.0 mL) loaded with one grinding ball (stainless, diameter: 10 mm). After being closed, the vessel placed in the ball mill machine (Retsch MM400, 60 or 90 min at 20 Hz) with a heat gun (the preset temperature at 300 $^{\circ}\text{C}$). After ball milling, the jar was then cooled rapidly with cold water and opened. The mixture was filtered by CH_2Cl_2 to remove Ag salts. The crude mixture was then purified by flash column chromatography (SiO_2 , typically $\text{CH}_2\text{Cl}_2/\text{hexane}$, typically 0:100-100:0) or reprecipitation from $\text{CH}_2\text{Cl}_2/\text{hexane}$ to give the corresponding tris-cyclometalated iridium(III) complexes **4**.

3) Procedure C: solid-state one-pot synthesis of tris-cyclometalated iridium(III) complexes **4**

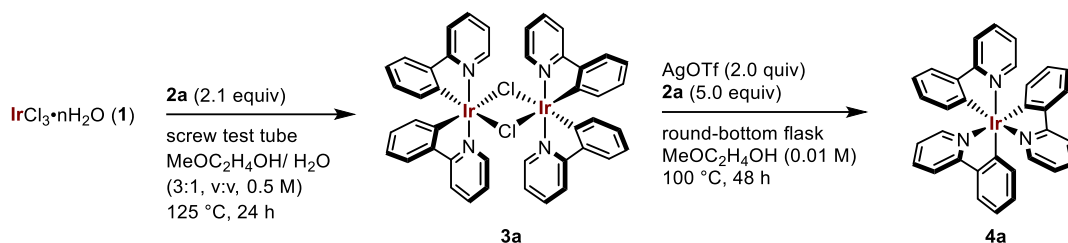


Iridium(III) chloride hydrate **1** (0.20 mmol), **2** (0.42 mmol, 2.1 equiv), and 2-methoxyethanol (0.20 $\mu\text{L}/\text{mg}$) were placed in a ball milling vessel (stainless, 1.5 mL) loaded with one grinding ball (stainless, diameter: 5 mm). After being closed, the vessel placed in the ball mill machine (Retsch MM400, 10 or 30 min at 30 Hz) with a heat gun (the preset temperature at 300 $^{\circ}\text{C}$). After ball milling, the jar was rapidly opened. Then **2** (5.0 equiv to **3**), AgOTf (2.0 equiv to **3**) and 2-methoxyethanol (0.20 $\mu\text{L}/\text{mg}$) were placed in a the vessel. After being closed, the vessel placed in the ball mill machine (Retsch MM400, 60 min at 30 Hz) with a heat gun (the preset temperature at 300 $^{\circ}\text{C}$). After ball milling, the jar was then cooled rapidly with cold water and opened. The mixture was filtered by CH_2Cl_2 to remove Ag salts. The crude mixture was then purified by flash column chromatography (SiO_2 , typically $\text{CH}_2\text{Cl}_2/\text{hexane}$, typically 0:100-100:0) or reprecipitation from $\text{CH}_2\text{Cl}_2/\text{hexane}$ to give the corresponding tris-cyclometalated iridium(III) complexes **4**.

4. List of Ligands Used in This Study.



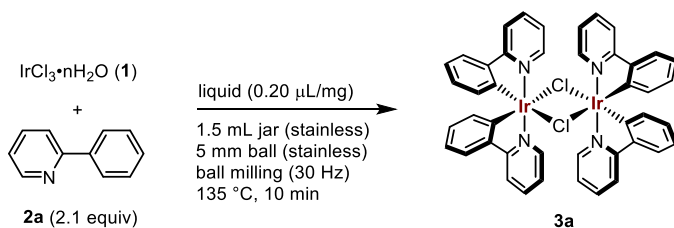
5. Attempts for One-Pot Synthesis in Solution



An oven-dried reaction vial was charged with iridium(III) chloride hydrate **1** (59.6 mg, 0.20 mmol), **2a** (68.5 mg, 0.44 mmol, 2.2 equiv) and then the vial was capped with a rubber septum. The vial with reagents was refilled with a nitrogen atmosphere and the solids were suspended in 2-methoxyethanol/H₂O (300 μL/100 μL), then the suspension was allowed to stir at 125 °C for 24 h. After completing the reaction, the mixture was moved an oven-dried round-bottom flask and added **2** (77.6 mg, 0.5 mmol, 5.0 equiv to **3a**), AgOTf (52.5 mg, 0.20 mmol, 2.0 equiv to **3a**). The flask was refilled with a nitrogen atmosphere and the solids were suspended in 2-methoxyethanol (20 mL), then the suspension was allowed to stir at 100 °C for 48 h. After completing the reaction, the mixture was cooled and opened. The mixture was filtered by MeOH/CH₂Cl₂ to give the crude mixture. The desired product **4a** was not detected by ¹H NMR analysis of the crude mixture.

6. Effects of Liquid Additives

We have investigated the effect of other liquid additives. We have tested polar solvents such as EtOH, glycerol, DMF, and DMSO, which furnished **3a** in comparable yields (55-75%) that are lower than that of the conditions using 2-methoxyethanol. The use of PEG-400 did not improve the yield of **3a** (78%). We also checked a non-polar solvent (toluene), which provided inferior results (58%).

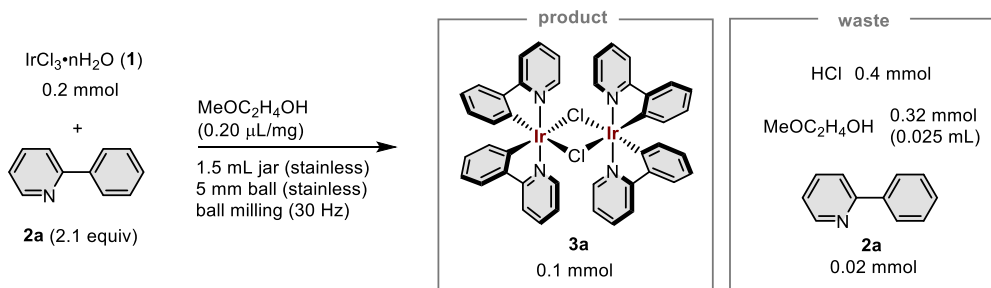


liquid	yield of 3a (%)
MeOC ₂ H ₄ OH	80%
EtOH	75%
Glycerol	71%
DMF	70%
DMSO	55%
PEG-400	78%
toluene	58%

Table S1. Effects of liquid additives for the first ligand exchange.

7. Calculations of E-Factors

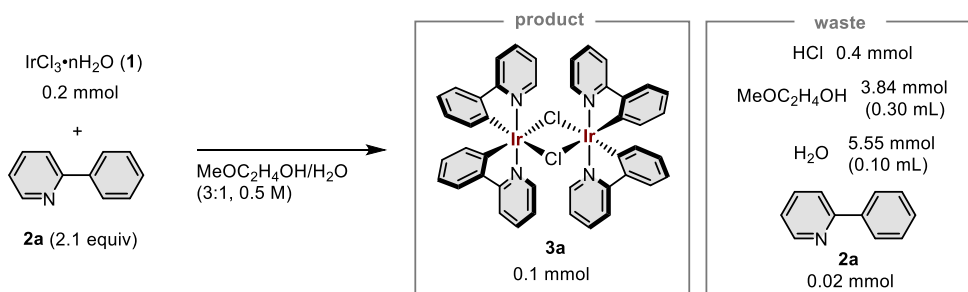
For the first ligand exchange



E-factor for the first ligand exchange in the solid-state.

this work	M _w	mmol	mg
product (3a)	1072.10	0.10	107.21
waste	M _w	mmol	mg
HCl	36.46	0.40	14.58
MeOC ₂ H ₄ OH	76.10	0.32	24.35
unreacted 2a	155.20	0.020	3.10
total			149.24

$$E_{\text{mechanochemical}} = 149.24 / 107.21 = 1.39$$

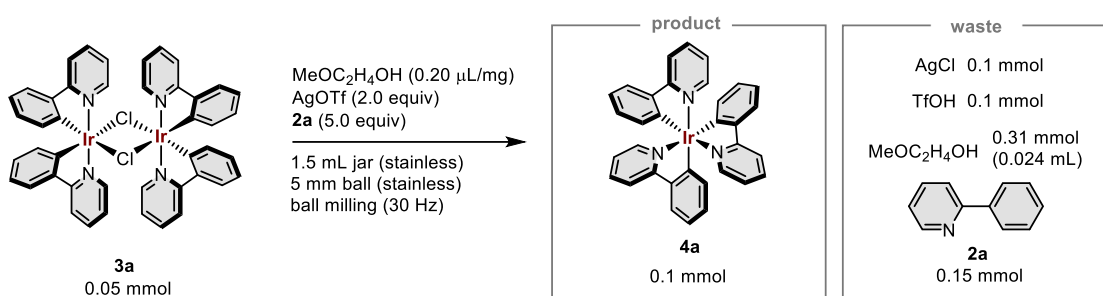


E-factor for the first ligand exchange in solution.

this work	M _w	mmol	mg
product (3a)	1072.10	0.10	107.21
waste	M _w	mmol	mg
HCl	36.46	0.40	14.58
MeOC ₂ H ₄ OH	76.10	3.84	292.22
H ₂ O	18.02	5.55	100.01
unreacted 2a	155.20	0.020	3.10
total			517.12

$$E_{\text{solution}} = 517.12 / 107.21 = 4.82$$

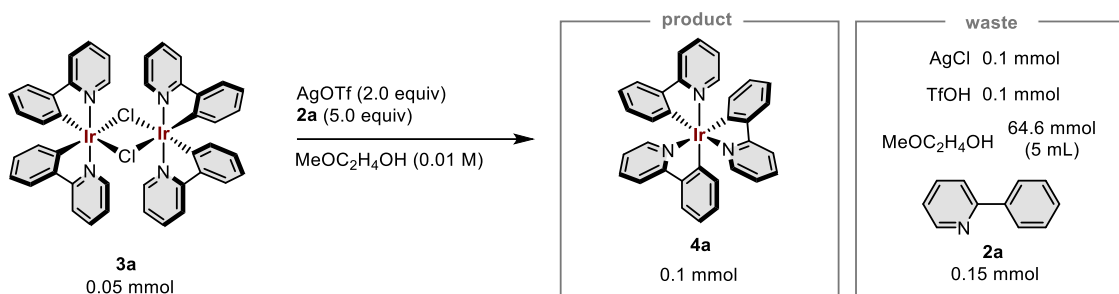
For the second ligand exchange



E-factor for the second ligand exchange in the solid-state.

this work	M_w	mmol	mg
product (4a)	654.79	0.10	65.48
waste	M_w	mmol	mg
AgCl	143.32	0.10	14.33
TfOH	150.07	0.10	15.01
$\text{MeOC}_2\text{H}_4\text{OH}$	76.10	0.31	23.56
unreacted 2a	155.20	0.15	23.28
total			141.66

$$E_{\text{mechanochemical}} = 141.66 / 65.48 = \mathbf{2.16}$$



E-factor for the second ligand exchange in solution.

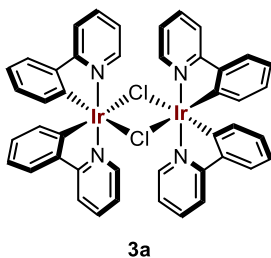
this work	M_w	mmol	mg
product (4a)	654.79	0.10	65.48
waste	M_w	mmol	mg
AgCl	143.32	0.10	14.33
TfOH	150.07	0.10	15.01
$\text{MeOC}_2\text{H}_4\text{OH}$	76.10	64.6	4915.74
unreacted 2a	155.20	0.15	23.28
total			5033.84

$$E_{\text{solution}} = 5033.84 / 65.48 = \mathbf{76.88}$$

8. Characterization of Products.

In this study, the generation of meridional (*mer*) tris-cyclometalated iridium (III) complexes were not confirmed in the crude reaction mixtures, and the facial (*fac*) complexes were obtained exclusively under mechanochemical conditions.³

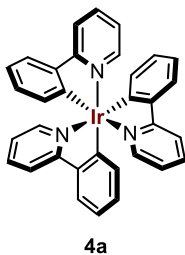
Dichlorotetrakis[2-(2-pyridinyl)phenyl]diiridium(III) (**3a**).



The reaction was performed according to the general procedure A (ball-milling time: 10 min). The reaction was carried out with 59.7 mg (0.20 mmol) of **1**. The product **3a** was obtained as a yellow powder (87.1 mg, 0.081 mmol, 81% yield) after reprecipitation from CH₂Cl₂/hexane.

¹H NMR (400 MHz, CDCl₃, δ): 5.93 (d, *J* = 8.0 Hz, 4H), 6.56 (td, *J* = 0.8, 7.2 Hz, 4H), 6.72–6.80 (m, 8H), 7.49 (dd, *J* = 1.2, 8.0 Hz, 4H), 7.74 (td, *J* = 1.2, 8.0 Hz, 4H), 7.87 (d, *J* = 8.0 Hz, 4H), 9.24 (dd, *J* = 0.8, 5.6 Hz, 4H). ¹³C NMR (99 MHz, CDCl₃, δ): 118.4 (CH), 121.3 (CH), 122.1 (CH), 123.6 (CH), 126.3 (CH), 129.1 (CH), 130.6 (CH), 136.1 (CH), 143.7 (CH), 145.3 (C), 147.5 (C), 151.7 (CH), 168.5 (C). The monomer of **3a** was only detected by ESI high-resolution mass spectrometry. HRMS-ESI (*m/z*): [M–Cl]⁺ calcd for C₂₂H₁₆IrN₂, 499.0914; found, 499.0912.

fac-Tris(2-phenylpyridinato)iridium(III) (**4a**).

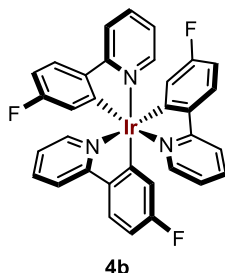


The reaction was performed according to the general procedure B, Method 1 (ball-milling time: 60 min). The reaction was carried out with 54.4 mg (0.051 mmol) of **3a**. The product **4a** was obtained as a yellow powder (47.9 mg, 0.073 mmol, 73% yield) after purification by silica-gel column chromatography (SiO₂, CH₂Cl₂/hexane, 0:100–50:50).

¹H NMR (392 MHz, CDCl₃, δ): 6.80–6.92 (m, 12H), 7.53 (d, *J* = 4.8 Hz, 3H), 7.59 (td, *J* = 1.2, 8.0 Hz, 3H), 7.66 (d, *J* = 7.2 Hz, 3H), 7.88 (d, *J* = 8.4 Hz, 3H). ¹³C NMR signals were barely detected

because of the low solubility of **4a**. HRMS-ESI (m/z): $[M+Na]^+$ calcd for $C_{33}H_{24}IrN_3Na$, 676.1468; found, 676.1465.

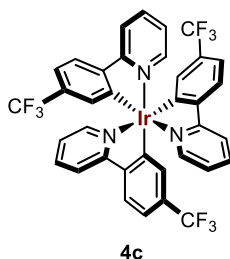
***fac*-Tris[2-(4-fluorophenyl)pyridinato]iridium(III) (**4b**).**



The reactions were performed according to the general procedure A (ball-milling time: 10 min) for the first ligand exchange and the general procedure B, Method 1 (ball-milling time: 60 min) for the second ligand exchange. The first ligand exchange reaction was carried out with 59.7 mg (0.20 mmol) of **1** to give the crude dimer product **3b** (93.2 mg, 0.09781 mmol, 81% yield), which was used directly for the next step without further purification. The second ligand exchange reaction was carried out with 85.4 mg of **3b** (0.075 mmol). The product **4b** was obtained as a yellow-green powder (85.2 mg, 0.120 mmol, 81% yield) (66% yield in 2 steps) after purification by silica-gel column chromatography (SiO_2 , CH_2Cl_2 /hexane, 0:100–50:50).

1H NMR (392 MHz, $CDCl_3$, δ): 6.45 (dd, $J = 2.7, 10.2$ Hz, 3H), 6.61 (td, $J = 2.7, 8.8$ Hz, 3H), 6.88 (t, $J = 6.3$ Hz, 3H), 7.47 (d, $J = 5.5$ Hz, 3H), 7.62 (dd, $J = 5.9, 8.6$ Hz, 6H), 7.82 (d, $J = 8.6$ Hz, 3H). ^{13}C NMR (99 MHz, $CDCl_3$, δ): 107.6 (d, $J = 23.4$ Hz, CH), 118.8 (CH), 121.7 (CH), 122.2 (d, $J = 16.9$ Hz, CH), 125.7 (d, $J = 9.5$ Hz, CH), 136.3 (CH), 139.8 (C), 147.0 (CH), 163.1 (d, $J = 52.6$ Hz, C), 164.4 (d, $J = 193.5$ Hz, C), 165.5 (C). HRMS-ESI (m/z): $[M]^+$ calcd for $C_{33}H_{21}F_3IrN_3$, 707.1294; found, 707.1286.

***fac*-Tris{2-[4-(trifluoromethyl)phenyl]pyridinato}iridium(III) (**4c**).**

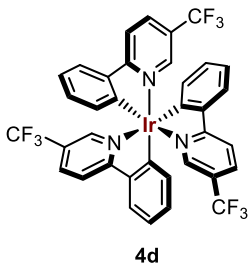


The reactions were performed according to the general procedure A (ball milling time: 10 min) for the first ligand exchange and the general procedure B, Method 1 (ball-milling time: 60 min) for the second ligand exchange. The first ligand exchange reaction was carried out with 60.3 mg (0.20 mmol) of **1** to give the crude dimer product **3c** (130.4 mg, 0.097 mmol, 96% yield), which was used directly for the

next step without further purification. The second ligand exchange reaction was carried out with 121.2 mg of **3c** (0.090 mmol). The product **4c** was obtained as a yellow powder (91.7 mg, 0.107 mmol, 59% yield) (57% yield in 2 steps) after purification by silica-gel column chromatography (SiO₂, CH₂Cl₂/hexane, 0:100–50:50).

¹H NMR (392 MHz, CDCl₃, δ): 6.94 (d, *J* = 1.2 Hz, 3H), 6.97–7.02 (m, 3H), 7.15 (dd, *J* = 2.0, 8.0 Hz, 3H), 7.50–7.55 (m, 3H), 7.68–7.74 (m, 6H), 7.97 (d, *J* = 8.0 Hz, 3H). ¹³C NMR signals were barely detected because of the low solubility of **4c**. HRMS-ESI (*m/z*): [M]⁺ calcd for C₃₆H₂₁F₉IrN₃, 857.1198; found, 857.1192.

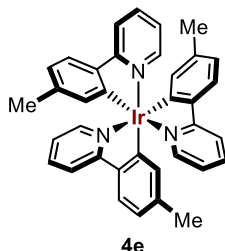
***fac*-Tris[2-phenyl-5-(trifluoromethyl)pyridinato]iridium(III) (**4d**).**



The reactions were performed according to the general procedure A (ball-milling time: 10 min) for the first ligand exchange and the general procedure B, Method 1 (ball-milling time: 60 min) for the second ligand exchange. The first ligand exchange reaction was carried out with 59.7 mg (0.20 mmol) of **1** to give the crude dimer product **3d** (71.5 mg, 0.054 mmol, 53% yield), which was used directly for the next step without further purification. The second ligand exchange reaction was carried out with 64.8 mg of **3d** (0.048 mmol). The product **4d** was obtained as a yellow powder (55.2 mg, 0.064 mmol, 67% yield) (36% yield in 2 steps) after purification by silica-gel column chromatography (SiO₂, CH₂Cl₂/hexane, 0:100–50:50).

¹H NMR (400 MHz, CDCl₃, δ): 6.81 (dd, *J* = 1.2, 7.6 Hz, 3H), 6.92 (td, *J* = 1.2, 7.6 Hz, 3H), 6.99 (td, *J* = 1.2, 7.2 Hz, 3H), 7.67 (s, 3H), 7.73 (dd, *J* = 1.6, 8.0 Hz, 3H), 7.85 (dd, *J* = 2.0, 8.8 Hz, 3H), 8.02 (d, *J* = 8.4 Hz, 3H). ¹³C NMR (99 MHz, CDCl₃, δ): 118.9 (CH), 120.9 (CH), 121.3 (C), 124.1 (C), 124.8 (C), 125.1 (C), 125.6 (CH), 131.5 (CH), 133.7 (CH), 137.2 (CH), 141.8 (C), 143.79 (CH), 143.84 (CH), 161.0 (C), 170.0 (C). HRMS-ESI (*m/z*): [M]⁺ calcd for C₃₆H₂₁F₉IrN₃, 857.1198; found, 857.1180

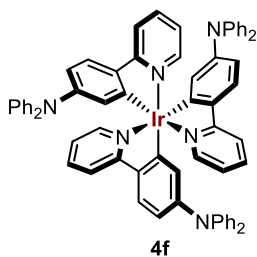
***fac*-Tris[2-(*p*-tolyl)pyridinato]iridium(III) (**4e**).**



The reactions were performed according to the general procedure A (ball-milling time: 10 min) for the first ligand exchange and the general procedure B, Method 1 (ball-milling time: 60 min) for the second ligand exchange. The first ligand exchange reaction was carried out with 60.4 mg (0.20 mmol) of **1** to give the crude dimer product **3e** (97.8 mg, 0.087 mmol, 86% yield), which was used directly for the next step without further purification. The second ligand exchange reaction was carried out with 92.1 mg of **3e** (0.082 mmol). The product **4e** was obtained as a white powder (35.5 mg, 0.051 mmol, 31% yield) (27% yield in 2 steps) after purification by silica-gel column chromatography (SiO₂, CH₂Cl₂/hexane, 0:100–50:50).

¹H NMR (392 MHz, CDCl₃, δ): 2.12 (s, 9H), 6.57 (s, 3H), 6.66 (d, *J* = 7.4 Hz, 3H), 6.77 (s, 3H), 7.43 (s, 3H), 7.51–7.59 (m, 6H), 7.81 (d, *J* = 8.2 Hz, 3H). ¹³C NMR (99 MHz, CDCl₃, δ): 21.9 (CH₃), 118.3 (CH), 121.0 (CH), 121.3 (CH), 123.6 (CH), 135.6 (CH), 137.7 (CH), 139.4 (C), 141.1 (C), 146.9 (CH), 161.4 (C), 166.7 (C). HRMS-ESI (*m/z*): [M]⁺ calcd for C₃₆H₃₀IrN₃, 695.2046; found, 695.2042.

***fac*-Tris[*N,N*-diphenyl-4-(pyridin-2-yl)anilinato]iridium(III) (**4f**).**

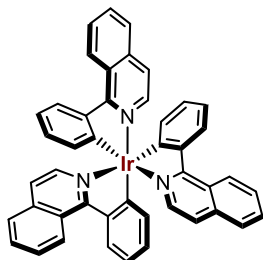


The reactions were performed according to the general procedure A (ball-milling time: 10 min) for the first ligand exchange and the general procedure B, Method 1 (ball-milling time: 60 min) for the second ligand exchange. The first ligand exchange reaction was carried out with 60.2 mg (0.20 mmol) of **1** to give the crude dimer product **3f** (190.3 mg, 0.109 mmol, >99% yield), which was used directly for the next step without further purification. The second ligand exchange reaction was carried out with 178.1 mg of **3f** (0.102 mmol). The product **4f** was obtained as a red powder (49.4 mg, 0.043 mmol, 21% yield) (21% yield in 2 steps) after purification by silica-gel column chromatography (SiO₂, CH₂Cl₂/hexane, 0:100–50:50).

¹H NMR (392 MHz, CDCl₃, δ): 6.25 (dd, *J* = 2.4, 8.6 Hz, 3H), 6.58 (d, *J* = 2.7 Hz, 3H), 6.76–6.90 (m,

21H), 7.09 (t, $J = 7.8$ Hz, 12H), 7.27 (d, $J = 7.4$ Hz, 3H), 7.51–7.57 (m, 6H), 7.69 (d, $J = 7.8$ Hz, 3H). ^{13}C NMR (99 MHz, CDCl_3 , δ): 114.5 (CH), 117.7 (CH), 120.5 (CH), 122.1 (CH), 124.5 (CH), 124.7 (CH), 128.6 (CH), 130.1 (CH), 135.4 (CH), 137.5 (C), 147.2 (C), 147.6 (CH), 148.2 (C), 161.8 (C), 166.2 (C). HRMS-ESI (m/z): $[\text{M}]^+$ calcd for $\text{C}_{69}\text{H}_{51}\text{IrN}_6$, 1154.3781; found, 1154.3813.

***fac*-Tris(1-phenylisoquinolino)iridium(III) (4g).**

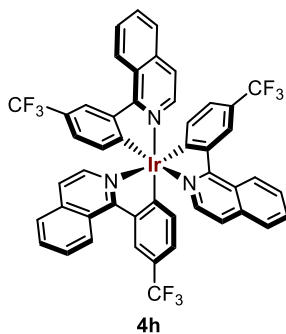


4g

The reactions were performed according to the general procedure A (ball-milling time: 30 min) for the first ligand exchange and the general procedure B, Method 2 (ball-milling time: 90 min) for the second ligand exchange. The first ligand exchange reaction was carried out with 59.7 mg (0.20 mmol) of **1** to give the crude dimer product **3g** (77.0 mg, 0.061 mmol, 61% yield), which was used directly for the next step without further purification. The second ligand exchange reaction was carried out with 62.2 mg of **3g** (0.049 mmol). The product **4g** was obtained as a red powder (71.5 mg, 0.089 mmol, 91% yield) (56% yield in 2 steps) after purification by silica-gel column chromatography (SiO_2 , CH_2Cl_2 /hexane, 0:100–80:20).

^1H NMR (392 MHz, CDCl_3 , δ): 6.87 (t, $J = 7.3$ Hz, 3H), 6.95–7.01 (m, 6H), 7.12 (d, $J = 6.3$ Hz, 3H), 7.31 (d, $J = 6.3$ Hz, 3H), 7.61–7.68 (m, 6H), 7.70–7.75 (m, 3H), 8.20 (d, $J = 7.8$ Hz, 3H), 8.94–9.00 (m, 3H). ^{13}C NMR signals were barely detected because of the low solubility of **4g**. HRMS-ESI (m/z): $[\text{M}]^+$ calcd for $\text{C}_{45}\text{H}_{30}\text{IrN}_3$, 803.2046; found, 803.2038.

***fac*-Tris{1-[3-(trifluoromethyl)phenyl]isoquinolino}iridium(III) (4h).**



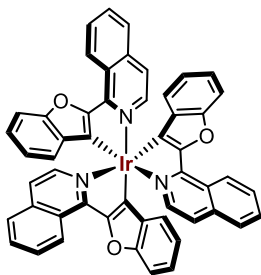
4h

The reactions were performed according to the general procedure A (ball-milling time: 30 min) for the

first ligand exchange and the general procedure B, Method 1 (ballmilling time: 90 min) for the second ligand exchange. The first ligand exchange reaction was carried out with 60.3 mg (0.20 mmol) of **1** to give the crude dimer product **3h** (157.6 mg, 0.102 mmol, >99% yield), which was used directly for the next step without further purification. The second ligand exchange reaction was carried out with 132.5 mg of **3h** (0.086 mmol). The product **4h** was obtained as a red powder (106.2 mg, 0.105 mmol, 61% yield) (61% yield in 2 steps) after purification by silica-gel column chromatography (SiO₂, CH₂Cl₂/hexane, 0:100–80:20).

¹H NMR (392 MHz, CDCl₃, δ): 7.00 (d, *J* = 7.8 Hz, 3H), 7.06 (d, *J* = 8.2 Hz, 3H), 7.25 (d, *J* = 6.3 Hz, 3H), 7.33 (d, *J* = 5.9 Hz, 3H), 7.70–7.83 (m, 9H), 8.40 (s, 3H), 8.88 (d, *J* = 7.8 Hz, 3H). ¹³C NMR (99 MHz, CDCl₃, δ): 121.5 (CH), 126.0 (CH), 126.1 (CH), 126.5 (C), 126.9 (CH), 127.3 (CH), 128.6 (CH), 130.8 (CH), 136.8 (C), 137.2 (CH), 139.4 (CH), 145.4 (C), 166.1 (C), 168.8 (C). HRMS-ESI (*m/z*): [M]⁺ calcd for C₄₈H₂₇F₉IrN₃, 1007.1667; found, 1007.1656.

***fac*-Tris[1-(benzofuran-2-yl)isoquinolinato]iridium(III) (**4i**).**

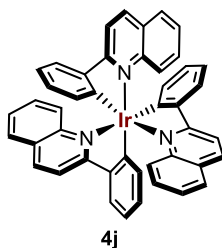


4i

The reactions were performed according to the general procedure A (ball-milling time: 30 min) for the first ligand exchange and the general conditions B, Method 1 (ball-milling time: 90 min) for the second ligand exchange. The first ligand exchange reaction was carried out with 61.5 mg (0.21 mmol) of **1** to give the crude dimer product **3i** (134.2 mg, 0.091 mmol, 91% yield) which was used directly for the next step without further purification. The second-step reaction was carried out with 128.2 mg of **3i** (0.089 mmol). The product **4i** was obtained as a red powder (116.0 mg, 0.125 mmol, 70% yield) (64% yield in 2 steps) after purification by silica-gel column chromatography (SiO₂, CH₂Cl₂/hexane, 0:100–70:30).

¹H NMR (392 MHz, CDCl₃, δ): 6.66 (d, *J* = 7.8 Hz, 3H), 6.78 (t, *J* = 7.6 Hz, 3H), 7.03 (d, *J* = 6.2 Hz, 3H), 7.19 (t, *J* = 7.8 Hz, 3H), 7.38 (d, *J* = 6.3 Hz, 3H), 7.52 (d, *J* = 8.2 Hz, 3H), 7.64–7.71 (m, 9H), 9.41 (d, *J* = 8.2 Hz, 3H). ¹³C NMR (99 MHz, CDCl₃, δ): 110.9 (CH), 117.8 (CH), 122.2 (CH), 124.6 (C), 125.5 (CH), 125.8 (CH), 126.1 (CH), 127.1 (CH), 127.5 (CH), 131.2 (CH), 136.6 (C), 136.7 (C), 137.3 (C), 141.6 (CH), 155.3 (C), 157.3 (C), 159.2 (C). HRMS-EI (*m/z*): [M]⁺ calcd for C₅₁H₃₀IrN₃O₃, 923.1893; found, 923.1893.

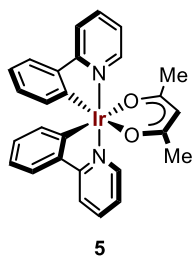
***fac*-Tris(2-phenylquinolino)iridium(III) (4j).**



The reactions were performed according to the general procedure A (ball-milling time: 30 min) for the first ligand exchange and the general conditions B, Method 1 (ball-milling time: 90 min) for the second ligand exchange. The first ligand exchange reaction was carried out with 60.2 mg (0.20 mmol) of **1** to give the crude dimer product **3j** (123.3 mg, 0.097 mmol, 96% yield), which was used directly for the next step without further purification. The second ligand exchange reaction was carried out with 110.0 mg of **3j** (0.086 mmol). The product **4j** was obtained as a red powder (68.3 mg, 0.085 mmol, 49% yield) (47% yield in 2 steps) after purification by silica-gel column chromatography (SiO₂, CH₂Cl₂/hexane, 0:100–100:0).

¹H NMR (392 MHz, CDCl₃, δ): 6.47 (d, *J* = 7.8 Hz, 3H), 6.66–6.71 (m, 6H), 6.85 (t, *J* = 7.4 Hz, 3H), 7.18 (t, *J* = 7.4 Hz, 3H), 7.66 (d, *J* = 7.4 Hz, 3H), 7.74 (d, *J* = 7.8 Hz, 3H), 8.00 (d, *J* = 9.0 Hz, 3H), 8.03–8.10 (m, 6H). ¹³C NMR signals were barely detected because of the low solubility of **4j**. HRMS-EI (*m/z*): [M]⁺ calcd for C₄₅H₃₀IrN₃, 803.2046; found, 803.2051.

Bis[2-(2-pyridinyl-*N*)phenyl-C](2,4-pentanedionato-*O2,O4*)iridium(III) (5).



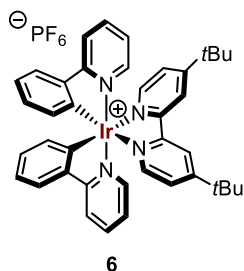
Chloride-bridged dimer **3a** (54.6 mg, 0.051 mmol), acetylacetone (50.6 mg, 0.51 mmol, 10 equiv), K₂CO₃ (140.0 mg, 1.01 mmol, 20 equiv) and 2-methoxyethanol (0.20 μL/mg) were placed in a ball milling vessel (stainless, 1.5 mL) loaded with one grinding ball (stainless, diameter: 5 mm). After being closed, the vessel placed in the ball mill machine (Retsch MM400, 60 min at 30 Hz) with a heat gun (the preset temperature at 300 °C). After ball milling, the jar was then cooled rapidly with cold water and opened. The mixture was filtered by H₂O and Et₂O to give the desired product **5** as a orange powder (49.9 mg, 0.083 mmol, 83% yield).

¹H NMR (400 MHz, CDCl₃, δ): 1.78 (s, 6H), 5.21 (s, 1H), 6.26 (d, *J* = 6.8 Hz, 2H), 6.68 (td, *J* = 1.6, 7.2 Hz, 2H), 6.80 (t, *J* = 8.0 Hz, 2H), 7.10–7.18 (m, 2H), 7.54 (d, *J* = 7.6 Hz, 2H), 7.73 (td, *J* = 1.2,

7.6 Hz, 2H), 7.85 (d, $J = 8.4$ Hz, 2H), 8.51 (d, $J = 4.8$ Hz, 2H). ^{13}C NMR (99 MHz, CDCl_3 , δ): 28.8 (CH₃), 100.3 (CH), 118.3 (CH), 120.6 (CH), 121.4 (CH), 123.8 (CH), 129.1 (CH), 133.0 (CH), 136.7 (CH), 144.6 (CH), 147.7 (C), 148.1 (CH), 168.6 (C), 184.5 (C). HRMS-EI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{27}\text{H}_{23}\text{IrN}_2\text{O}_2\text{Na}$, 621.1258; found, 621.1254.

(4,4'-Di-*tert*-butyl-2,2'-bipyridine)bis[(2-pyridinyl)phenyl]iridium(III) Hexafluorophosphate

(6).

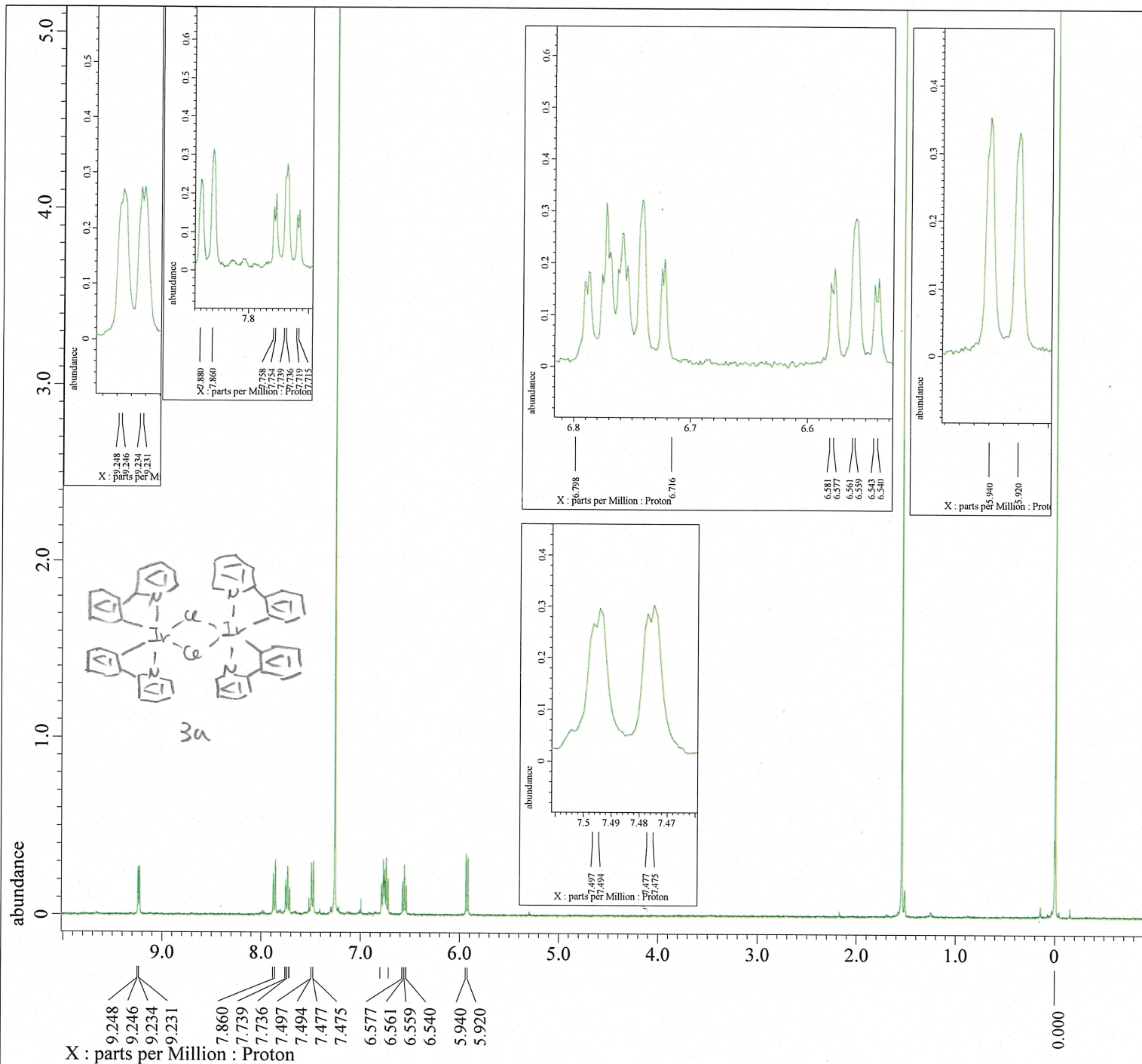


Chloride-bridged dimer **3a** (81.3 mg, 0.076 mmol), 4,4'-di-*tert*-butyl-2,2'-bipyridyl (85.0 mg, 0.32 mmol, 4.2 equiv), ethylene glycol (0.20 $\mu\text{L}/\text{mg}$) were placed in a ball milling vessel (stainless, 1.5 mL) loaded with one grinding ball (stainless, diameter: 5 mm). After being closed, the vessel placed in the ball mill machine (Retsch MM400, 60 min at 30 Hz) with a heat gun (the preset temperature at 300 °C). After ball milling, the jar was then cooled rapidly with cold water and opened. The mixture was extracted with Et_2O three times and the mixture was evaporated under vacuum. Then the mixture was dissolved in saturated KPF_6 aqueous solution and stirred for 1 hour at room temperature. The surry solution was filtered to give the desired product **6** as a red powder (119.9 mg, 0.131 mmol, 88% yield).

^1H NMR (401 MHz, CDCl_3 , δ): 1.45 (s, 18H), 6.29 (d, $J = 7.2$ Hz, 2H), 6.89 (t, $J = 7.0$ Hz, 2H), 7.01 (t, $J = 7.2$ Hz, 2H), 7.11 (s, 2H), 7.38 (d, $J = 4.4$ Hz, 2H), 7.59 (s, 2H), 7.67 (d, $J = 7.2$ Hz, 2H), 7.73–7.86 (m, 4H), 7.90 (d, $J = 6.8$ Hz, 2H), 8.58 (s, 2H). ^{13}C NMR (100 MHz, CDCl_3 , δ): 30.5 (CH₃), 35.7 (C), 119.4 (CH), 122.4 (CH), 123.6 (CH), 124.6 (CH), 125.3 (CH), 130.6 (CH), 131.7 (CH), 138.0 (CH), 143.6 (C), 149.4 (CH), 149.7 (CH), 150.8 (C), 155.7 (C), 164.0 (C), 167.6 (C). HRMS-EI (m/z): $[\text{M}-\text{PF}_6]^+$ calcd for $\text{C}_{40}\text{H}_{40}\text{N}_4\text{Ir}$, 767.2853; found, 767.2844.

9. References.

1. Anuradha, S.; Kip, T.; Megan, K.; Kariate, S. P.; Sadagopan, K.; Jimmie, D. W. *J. Org. Chem.* **776**, 51–59 (2015).
2. Seo, T.; Toyoshima, N; Kubota, K.; Ito, H. *J. Am. Chem. Soc.* **143**, 6165–6175 (2021).
3. Tamayo, A. B.; Alleyne, B. D.; Djurovich, P. I.; Lamansky, S.; Tsyba, I.; Ho, N. N.; Bau, R.; Thompson, M. E. *J. Am. Chem. Soc.* **125**, 7377–7387 (2003).



```

---- PROCESSING PARAMETERS ----
dc balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: TBR711-paper_Proton-1-1.jdf

```

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Filename      = TBR711-paper_Proton-1-2.j
Author       = element
Experiment   = proton.jxp
Sample_Id    = TBR711-paper
Solvent      = CHLOROFORM-D
Actual_Start_Time = 14-MAR-2023 14:07:47
Revision_Time   = 14-MAR-2023 14:18:09

Comment      = single_pulse
Data Format   = 1D COMPLEX
Dim_Size     = 13107
X_Domain     = Proton
Dim_Title    = Proton
Dim_Units    = [ppm]
Dimensions   = X
Site         = JNM-ECS400
Spectrometer = DELTA2_NMR

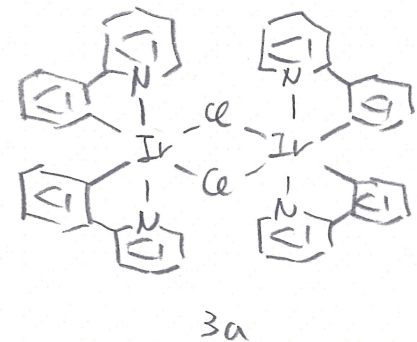
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```

Field Strength = 9.37221[T] (400[MHz])
X_Acq_Duration = 2.1889024[s]
X_Domain       = 1H
X_Freq         = 399.03472754[MHz]
X_Offset       = 5.0[ppm]
X_Points       = 16384
X_Prescans     = 1
X_Resolution   = 0.45684997[Hz]
X_Sweep        = 7.48502994[kHz]
X_Sweep_Clipped = 5.98802395[kHz]
Irr_Domain     = Proton
Irr_Freq       = 399.03472754[MHz]
Irr_Offset     = 5.0[ppm]
Tri_Domain     = Proton
Tri_Freq       = 399.03472754[MHz]
Tri_Offset     = 5.0[ppm]
Clipped        = FALSE
Scans          = 8
Total_Scans    = 8

Relaxation_Delay = 5[s]
Recvr_Gain       = 54
Temp_Get         = 19.4[dC]
X_90_Width      = 6.6[us]
X_Acq_Time      = 2.1889024[s]
X_Angle         = 45[deg]
X_Atn           = 1[dB]
X_Pulse         = 3.3[us]
Irr_Mode        = Off
Tri_Mode        = Off
Dante_Presat    = FALSE
Initial_Wait    = 1[s]
Repetition_Time = 7.1889024[s]

```



```

---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: TBR711-C-1.jdf
  
```

```

Filename      = TBR711-C-2.jdf
Author       = element
Experiment    = single_pulse_dec
Sample_Id     = 1
Solvent      = CHLOROFORM-D
Actual_Start_Time = 8-MAR-2023 05:50:43
Revision_Time = 14-MAR-2023 16:15:39

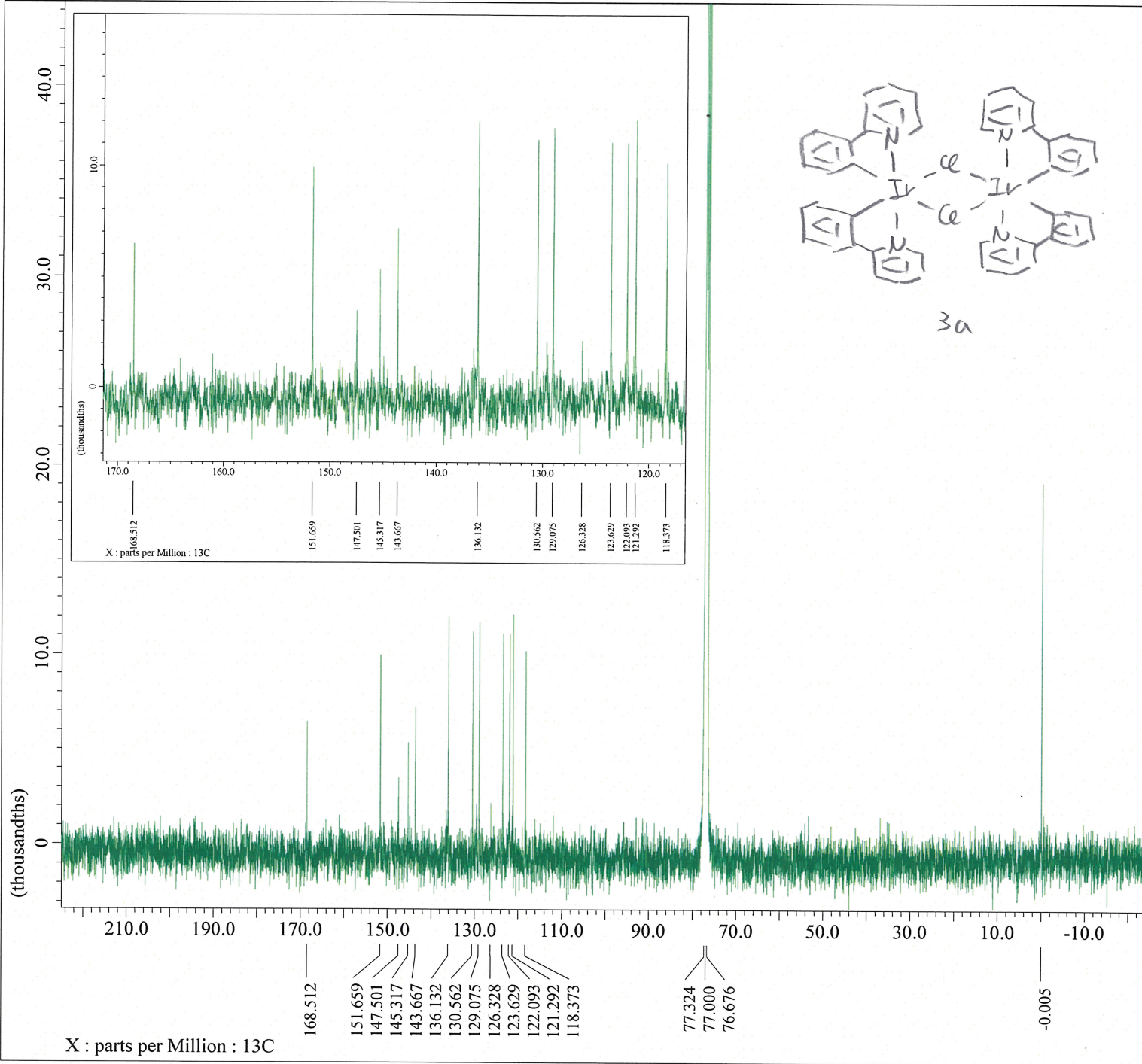
Comment      = single pulse decoupled ga
Data Format   = 1D COMPLEX
Dim_Size     = 26214
X_Domain     = 13C
Dim_Title    = 13C
Dim_Units    = [ppm]
Dimensions   = X
Site         = ECS 400
Spectrometer = JNM-ECS400
  
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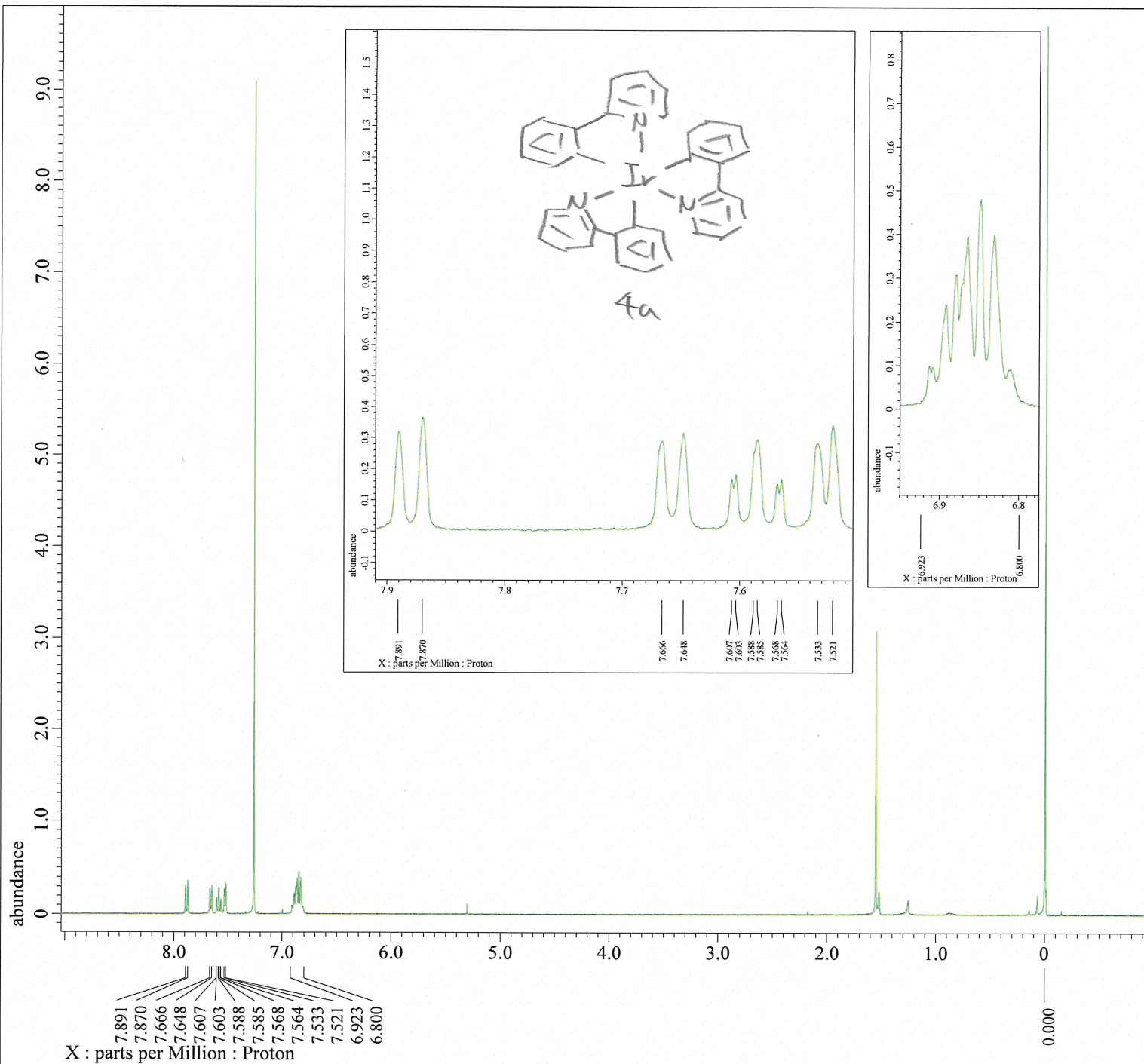
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Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain       = 13C
X_Freq         = 98.51479726[MHz]
X_Offset       = 100[ppm]
X_Points       = 32768
X_Prescans     = 4
X_Resolution   = 0.93958061[Hz]
X_Sweep        = 30.78817734[kHz]
Irr_Domain     = 1H
Irr_Freq       = 391.78655441[MHz]
Irr_Offset     = 5[ppm]
Clipped        = FALSE
Scans          = 10248
Total_Scans    = 10248
  
```

```

Relaxation_Delay = 2[s]
Recvr_Gain       = 60
Temp_Get         = 20[dC]
X_90_Width      = 8.3[us]
X_Acq_Time       = 1.06430464[s]
X_Angle         = 30[deg]
X_Atn           = 4.9[dB]
X_Pulse         = 2.76666667[us]
Irr_Atn_Dec     = 22.45[dB]
Irr_Atn_No     = 22.45[dB]
Irr_Noise       = WALTZ
Decoupling      = TRUE
Initial_Wait    = 1[s]
Noe              = TRUE
Noe_Time        = 2[s]
Repetition_Time = 3.06430464[s]
  
```





```

---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zeroFill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: TBR718_Proton-1-1.jdf

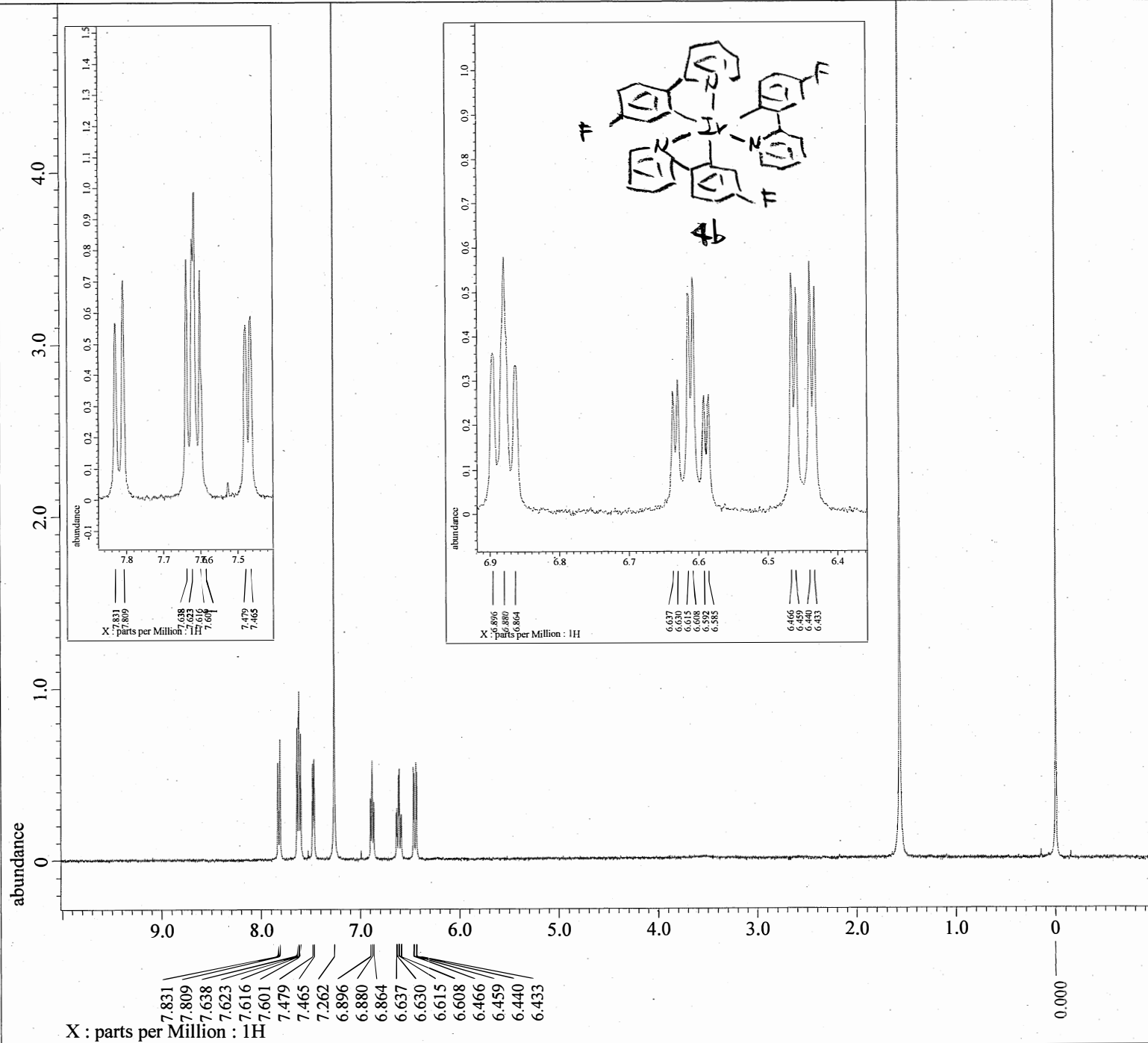
Filename      = TBR718_Proton-1-3.jdf
Author       = element
Experiment   = proton.jxp
Sample Id    = TBR718
Solvent      = CHLOROFORM-D
Actual_Start Time = 10-MAR-2023 10:53:31
Revision_Time = 14-MAR-2023 13:50:44

Comment      = single_pulse
Data Format   = 1D COMPLEX
Dim Size     = 13107
X_Domain     = Proton
Dim Title    = Proton
Dim Units    = [ppm]
Dimensions   = X
Spectrometer = DELTA2_NMR

Field Strength = 9.4073814[T] (400[MHz])
X_Acq_Duration = 2.18103808[s]
X_Domain       = 1H
X_Freq         = 400.53219825[MHz]
X_Offset       = 5[ppm]
X_Points       = 16384
X_Prescans     = 1
X_Resolution  = 0.45849727[Hz]
X_Sweep        = 7.51201923[kHz]
X_Sweep_Clippped = 6.00961538[kHz]
Irr_Domain     = Proton
Irr_Freq       = 400.53219825[MHz]
Irr_Offset     = 5[ppm]
Tri_Domain     = Proton
Tri_Freq       = 400.53219825[MHz]
Tri_Offset     = 5[ppm]
Clipped        = FALSE
Scans          = 8
Total_Scans    = 8

Relaxation_Delay = 5[s]
Recvr_Gain       = 50
Temp_Get         = 19.1[dC]
X_90_Width      = 6.7[us]
X_Acq_Time      = 2.18103808[s]
X_Angle         = 45[deg]
X_Atn           = 0.8[dB]
X_Pulse         = 3.35[us]
Irr_Mode        = Off
Tri_Mode        = Off
Dante_Presat    = FALSE
Initial_Wait    = 1[s]
Repetition_Time = 7.18103808[s]

```



```

---- PROCESSING PARAMETERS ----
dc balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

```

以下に由来: TBR252-retry-1.jdf

```

Filename      = TBR252-retry-3.jdf
Author        = element
Experiment    = single_pulse.ex2
Sample_Id     = 1
Solvent       = CHLOROFORM-D
Actual_Start_Time = 13-OCT-2022 03:45:35
Revision_Time = 14-OCT-2022 21:25:34

```

```

Comment       = single_pulse
Data_Format   = 1D COMPLEX
Dim_Size      = 13107
X_Domain      = 1H
Dim_Title     = 1H
Dim_Units     = [ppm]
Dimensions    = X
Site          = ECS 400
Spectrometer  = JNM-ECS400

```

```

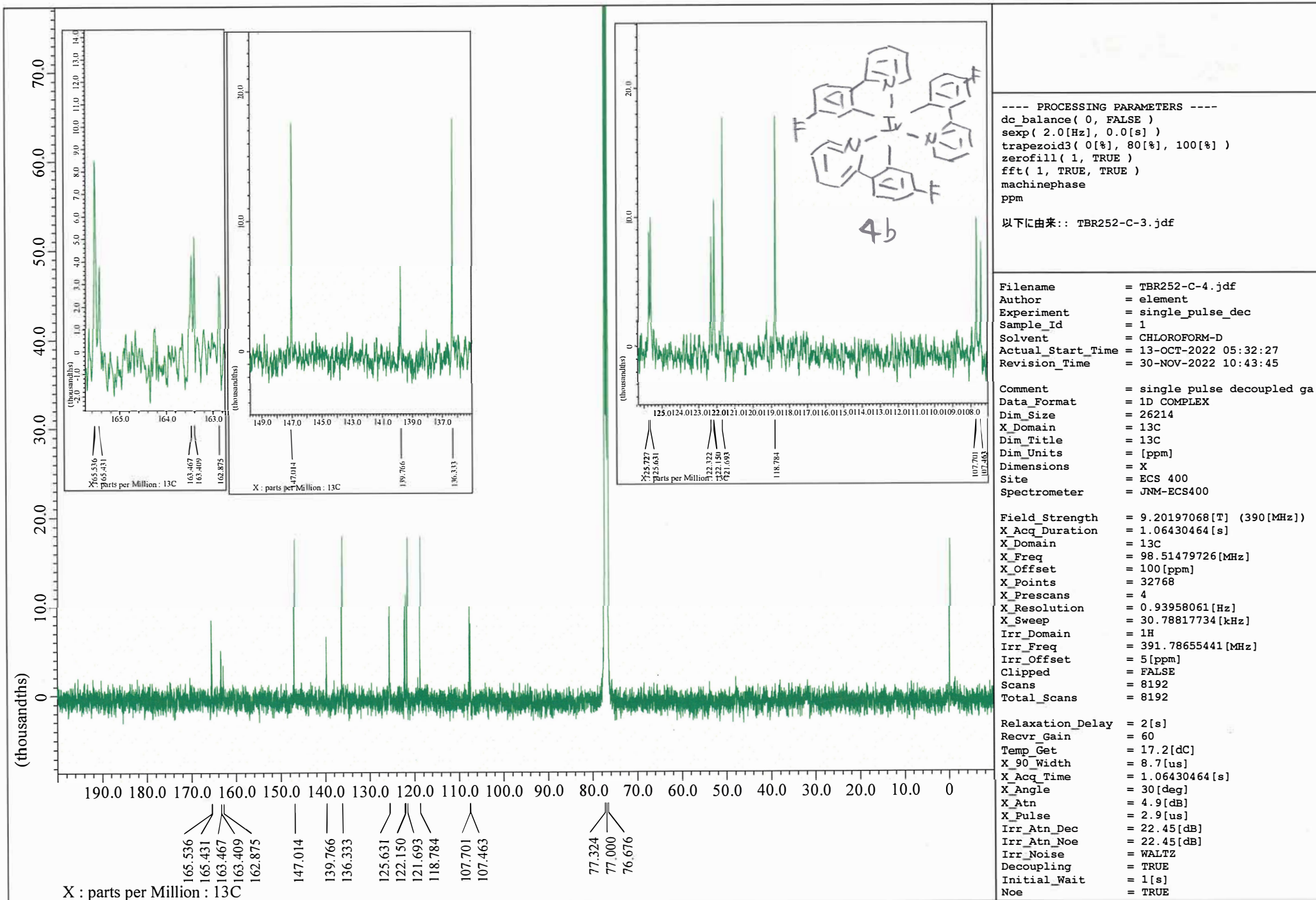
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 2.228224[s]
X_Domain       = 1H
X_Freq         = 391.78655441[MHz]
X_Offset       = 5[ppm]
X_Points       = 16384
X_Prescans     = 1
X_Resolution   = 0.44878791[Hz]
X_Sweep        = 7.35294118[kHz]
Irr_Domain     = 1H
Irr_Freq       = 391.78655441[MHz]
Irr_Offset     = 5[ppm]
Tri_Domain     = 1H
Tri_Freq       = 391.78655441[MHz]
Tri_Offset     = 5[ppm]
Clipped        = FALSE
Scans          = 8
Total_Scans    = 8

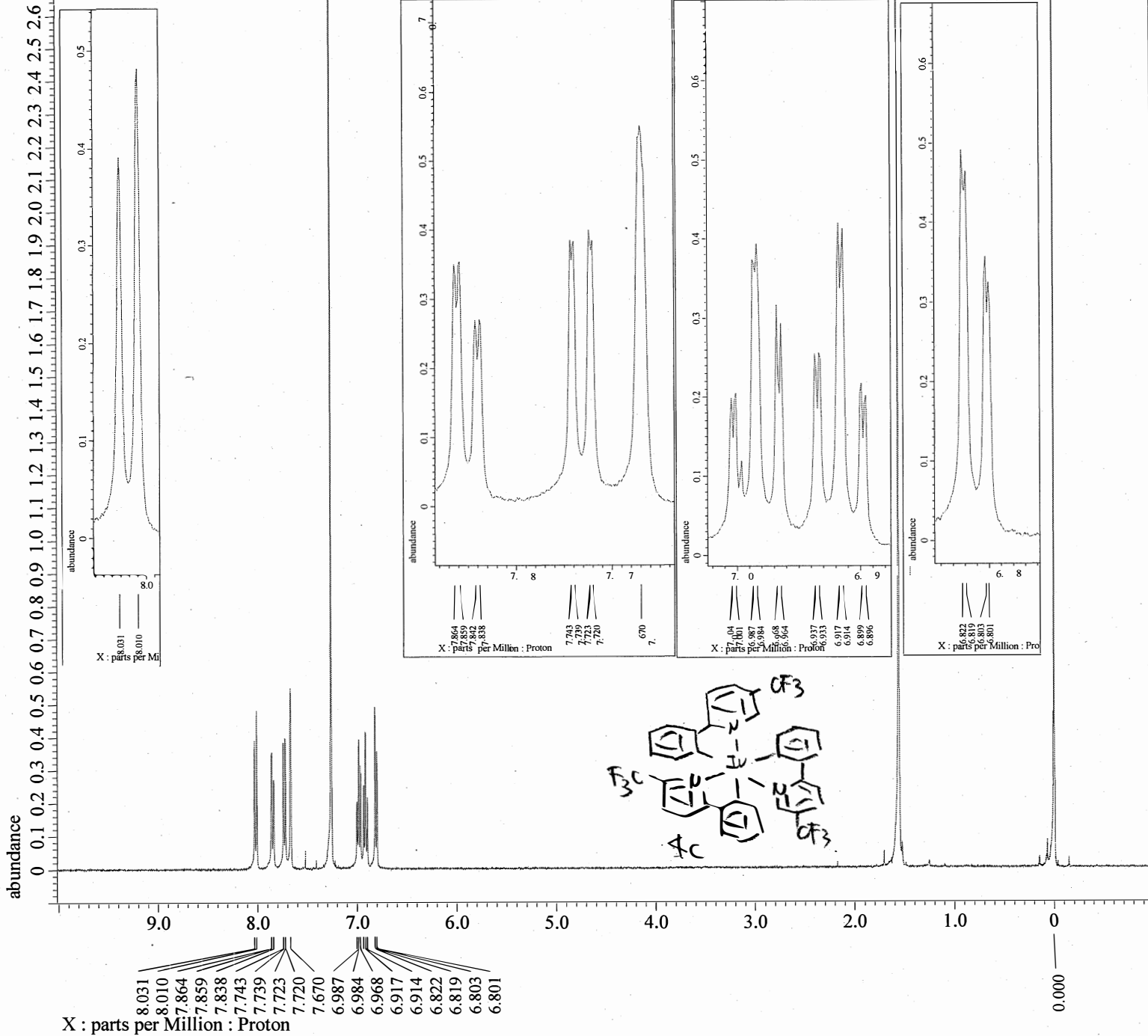
```

```

Relaxation_Delay = 5[s]
Recvr_Gain       = 58
Temp_Get         = 17.7[dc]
X_90_Width       = 10.8[us]
X_Acq_Time       = 2.228224[s]
X_Angle          = 45[deg]
X_Atn            = 1.9[db]
X_Pulse          = 5.4[us]
Irr_Mode         = Off
Tri_Mode         = Off
Dante_Presat     = FALSE

```





```

---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

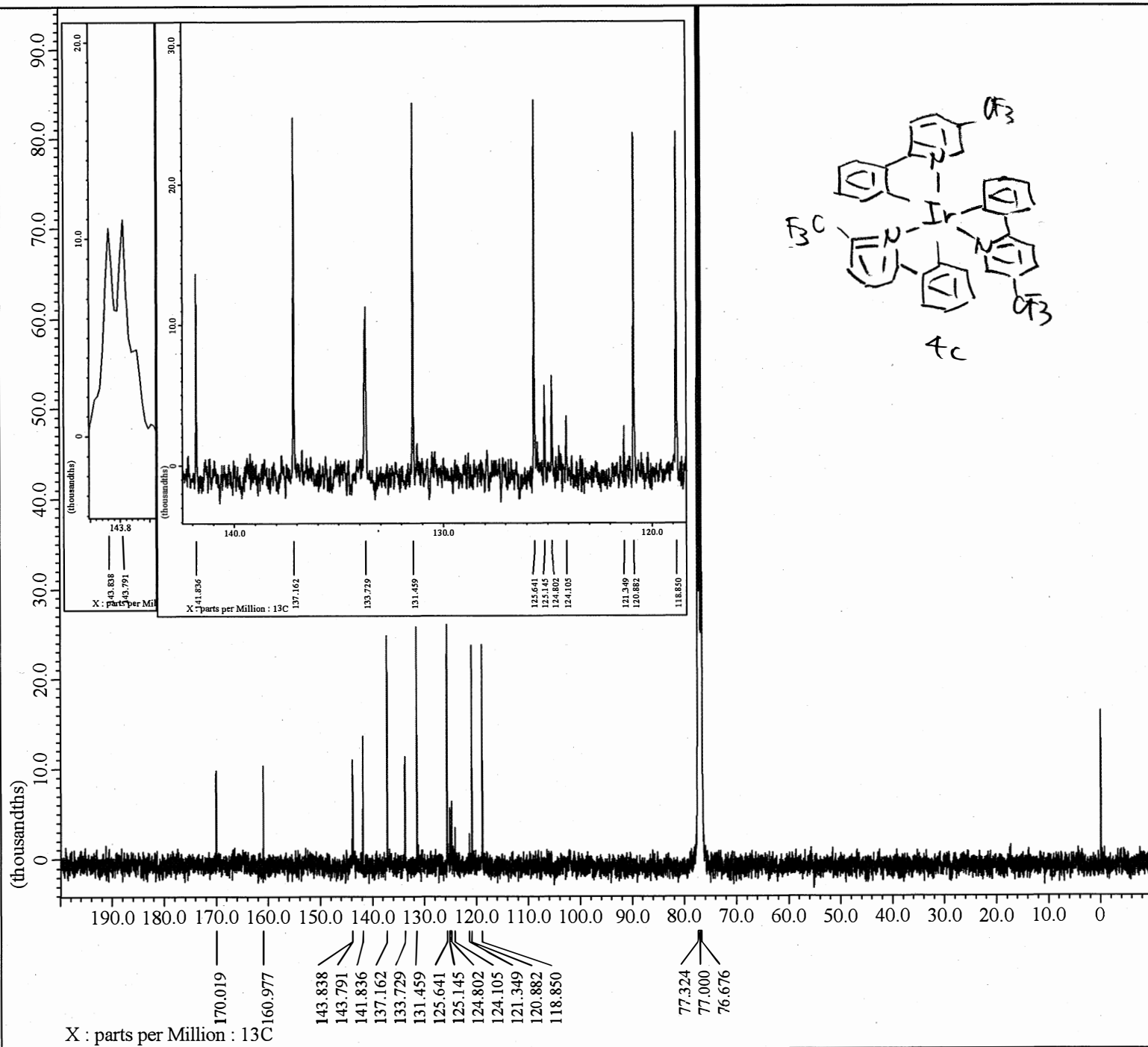
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Author        = element
Experiment    = proton.jxp
Sample Id     = TBR336-re
Solvent       = CHLOROFORM-D
Actual Start Time = 14-OCT-2022 19:08:07
Revision Time  = 17-OCT-2022 17:19:09

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Dim Size      = 13107
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Dim Units     = [ppm]
Dimensions    = X
Site          = JNM-ECS400
Spectrometer  = DELTA2_NMR

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X_Freq        = 399.03472754[MHz]
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X_Points      = 16384
X_Prescans    = 1
X_Resolution  = 0.45684997[Hz]
X_Sweep       = 7.48502994[kHz]
X_Sweep_Clippped = 5.98802395[kHz]
Irr_Domain    = Proton
Irr_Freq      = 399.03472754[MHz]
Irr_Offset    = 5.0[ppm]
Tri_Domain    = Proton
Tri_Freq      = 399.03472754[MHz]
Tri_Offset    = 5.0[ppm]
Clipped       = FALSE
Scans         = 8
Total Scans   = 8

Relaxation_Delay = 5[s]
Recvr_Gain       = 48
Temp_Get        = 21.3[dc]
X_90_Width      = 6.6[us]
X_Acq_Time      = 2.1889024[s]
X_Angle         = 45[deg]
X_Atn           = 1[db]
X_Pulse        = 3.3[us]
Irr_Mode        = Off
Tri_Mode        = Off
  
```



```

---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来: TBR336-C-1.jdf

```

```

Filename      = TBR336-C-2.jdf
Author       = element
Experiment    = single_pulse_dec
Sample_Id     = 1
Solvent      = CHLOROFORM-D
Actual_Start_Time = 15-OCT-2022 03:46:46
Revision_Time = 30-NOV-2022 11:01:36

```

```

Comment       = single pulse decoupled ga
Data_Format   = 1D COMPLEX
Dim_Size      = 26214
X_Domain      = 13C
Dim_Title     = 13C
Dim_Units     = [ppm]
Dimensions    = X
Site          = ECS 400
Spectrometer  = JNM-ECS400

```

```

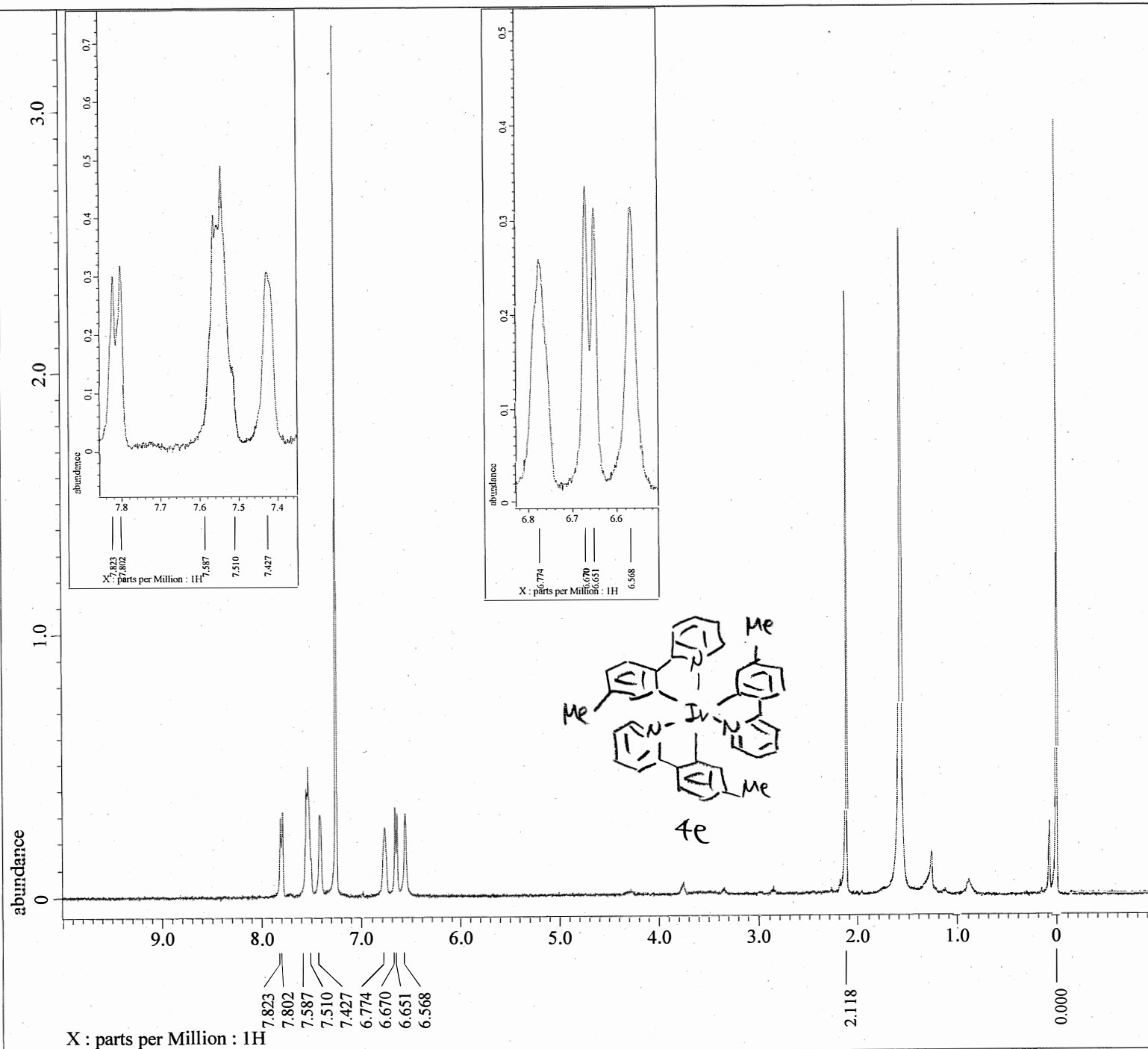
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain       = 13C
X_Freq         = 98.51479726[MHz]
X_Offset       = 100[ppm]
X_Points       = 32768
X_Prescans     = 4
X_Resolution   = 0.93958061[Hz]
X_Sweep        = 30.78817734[kHz]
Irr_Domain     = 1H
Irr_Freq       = 391.78655441[MHz]
Irr_Offset     = 5[ppm]
Clipped        = FALSE
Scans          = 11264
Total_Scans    = 11264

```

```

Relaxation_Delay = 2[s]
Recur_gain       = 60
Temp_Get         = 19.9[dc]
X_90_Width      = 8.7[us]
X_Acq_Time       = 1.06430464[s]
X_Angle         = 30[deg]
X_Atn           = 4.9[db]
X_Pulse         = 2.9[us]
Irr_Atn_Dec     = 22.45[db]
Irr_Atn_No     = 22.45[db]
Irr_Noise       = WALTZ
Decoupling       = TRUE
Initial_Wait    = 1[s]
Noe              = TRUE

```



---- PROCESSING PARAMETERS ----
 dc_balance(0, FALSE)
 sexp(0.2[Hz], 0.0[s])
 trapezoid3(0[%], 80[%], 100[%])
 zerofill(1, TRUE)
 fft(1, TRUE, TRUE)
 machinephase
 ppm

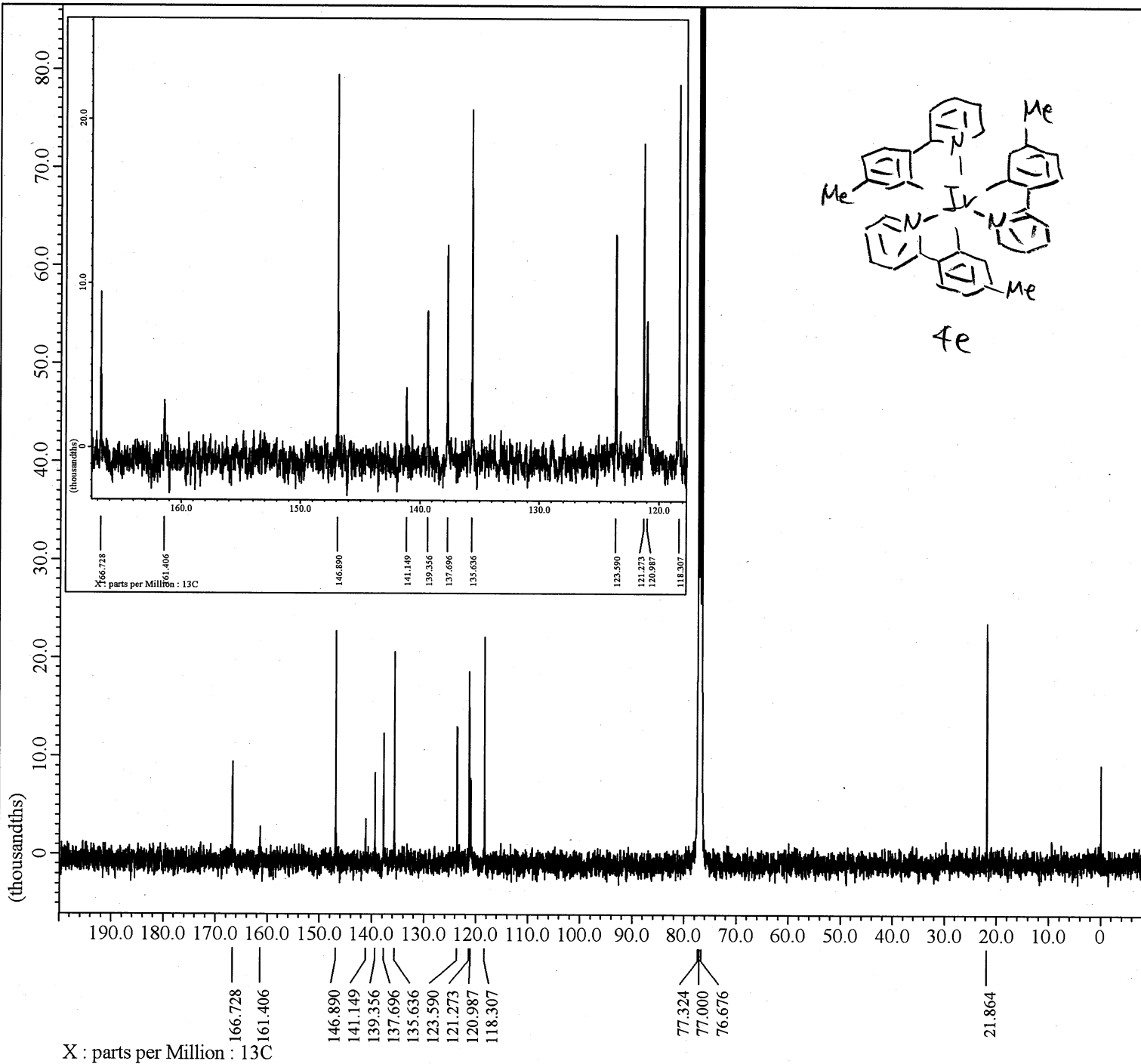
以下に由来: TBR375-redry-1.jdf

Filename = TBR375-redry-4.jdf
 Author = element
 Experiment = single_pulse.ex2
 Sample_Id = S#524701
 Solvent = CHLOROFORM-D
 Actual_Start_Time = 17-OCT-2022 21:13:53
 Revision_Time = 18-OCT-2022 16:35:29

Comment = single_pulse
 Data_Format = 1D_COMPLEX
 Dim_Size = 13107
 X_Domain = 1H
 Dim_Title = 1H
 Dim_Units = [ppm]
 Dimensions = X
 Site = ECS 400
 Spectrometer = JNM-ECS400

Field_Strength = 9.20197068[T] (390[MHz])
 X_Acq_Duration = 2.228224[s]
 X_Domain = 1H
 X_Freq = 391.78655441[MHz]
 X_Offset = 5[ppm]
 X_Points = 16384
 X_Prescans = 1
 X_Resolution = 0.44878791[Hz]
 X_Sweep = 7.35294118[kHz]
 Irr_Domain = 1H
 Irr_Freq = 391.78655441[MHz]
 Irr_Offset = 5[ppm]
 Tri_Domain = 1H
 Tri_Freq = 391.78655441[MHz]
 Tri_Offset = 5[ppm]
 Clipped = FALSE
 Scans = 8
 Total_Scans = 8

Relaxation_Delay = 5[s]
 Recvr_Gain = 56
 Temp_Get = 19.6[dc]
 X_90_Width = 10.8[us]
 X_Acq_Time = 2.228224[s]
 X_Angle = 45[deg]
 X_Atn = 1.9[db]
 X_Pulse = 5.4[us]
 Irr_Mode = Off
 Tri_Mode = Off
 Dante_Presat = FALSE



```

---- PROCESSING PARAMETERS ----
dc balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TBR375-C-2.jdf

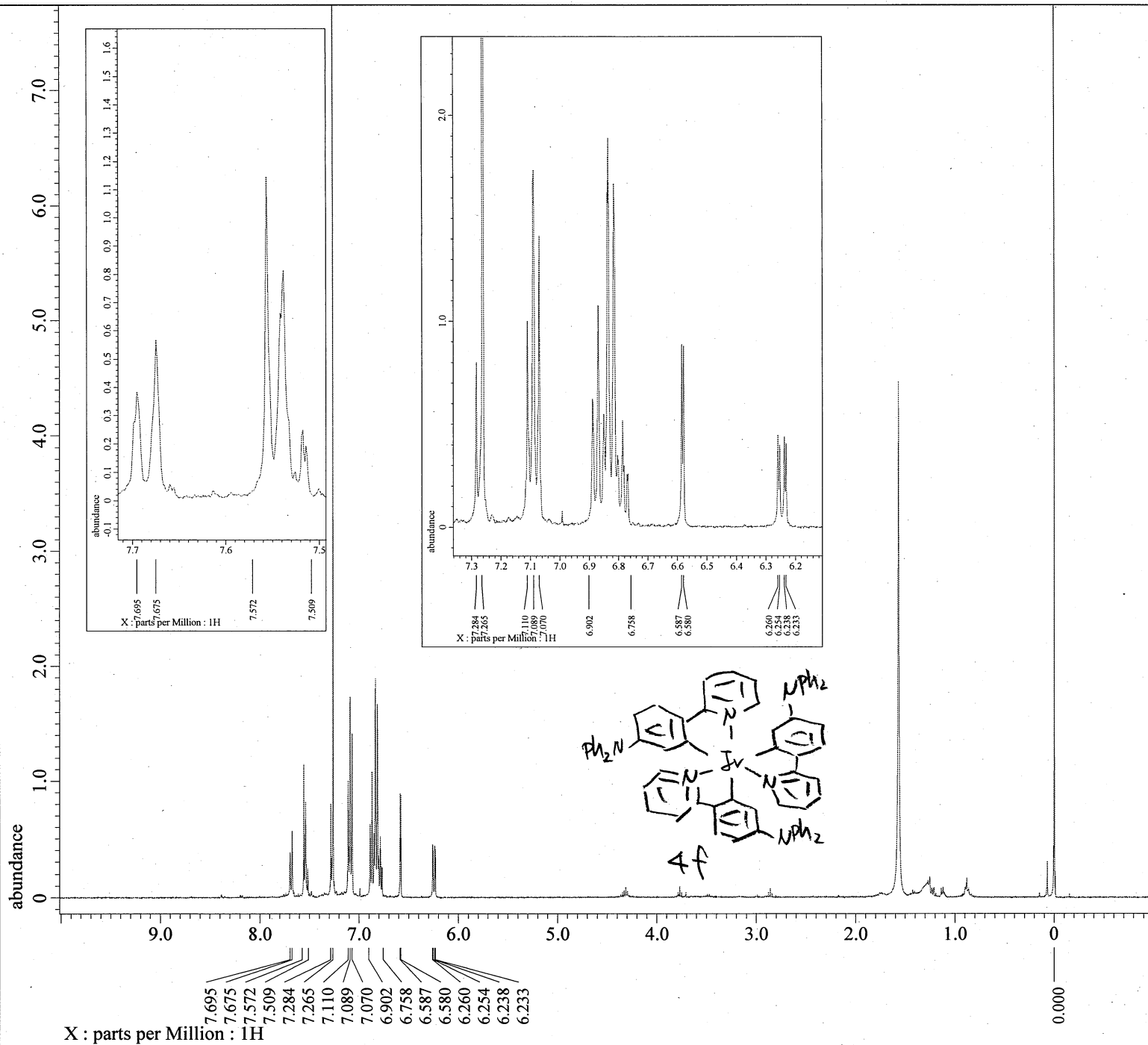
Filename      = TBR375-C-3.jdf
Author       = element
Experiment   = single_pulse_dec
Sample_Id    = 1
Solvent      = CHLOROFORM-D
Actual_Start Time = 24-OCT-2022 03:49:27
Revision_Time = 30-NOV-2022 11:16:50

Comment      = single pulse decoupled ga
Data_Format  = 1D COMPLEX
Dim_Size     = 26214
X_Domain     = 13C
Dim_Title    = 13C
Dim_Units    = [ppm]
Dimensions   = X
Site         = ECS 400
Spectrometer = JNM-ECS400

Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain       = 13C
X_Freq         = 98.51479726[MHz]
X_Offset       = 100[ppm]
X_Points       = 32768
X_Prescans     = 4
X_Resolution  = 0.93958061[Hz]
X_Sweep        = 30.78817734[kHz]
Irr_Domain     = 1H
Irr_Freq       = 391.78655441[MHz]
Irr_Offset     = 5[ppm]
Clipped        = FALSE
Scans          = 11264
Total_Scans    = 11264

Relaxation_Delay = 2[s]
Recvr_Gain       = 60
Temp_Get         = 17.5[dC]
X_90_Width      = 8.7[us]
X_Acq_Time       = 1.06430464[s]
X_Angle          = 30[deg]
X_Atn            = 4.9[dB]
X_Pulse         = 2.9[us]
Irr_Atn_Dec     = 22.45[dB]
Irr_Atn_No     = 22.45[dB]
Irr_Noise       = WALTZ
Decoupling      = TRUE
Initial_Wait    = 1[s]
Noe              = TRUE
Noe_Time        = 2[s]
Repetition_Time = 3.06430464[s]

```



---- PROCESSING PARAMETERS ----
 dc balance(0, FALSE)
 sexp(0.2[Hz], 0.0[s])
 trapezoid3(0[%], 80[%], 100[%])
 zerofill(1, TRUE)
 fft(1, TRUE, TRUE)
 machinephase
 ppm

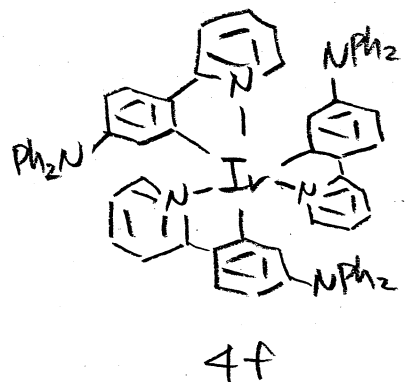
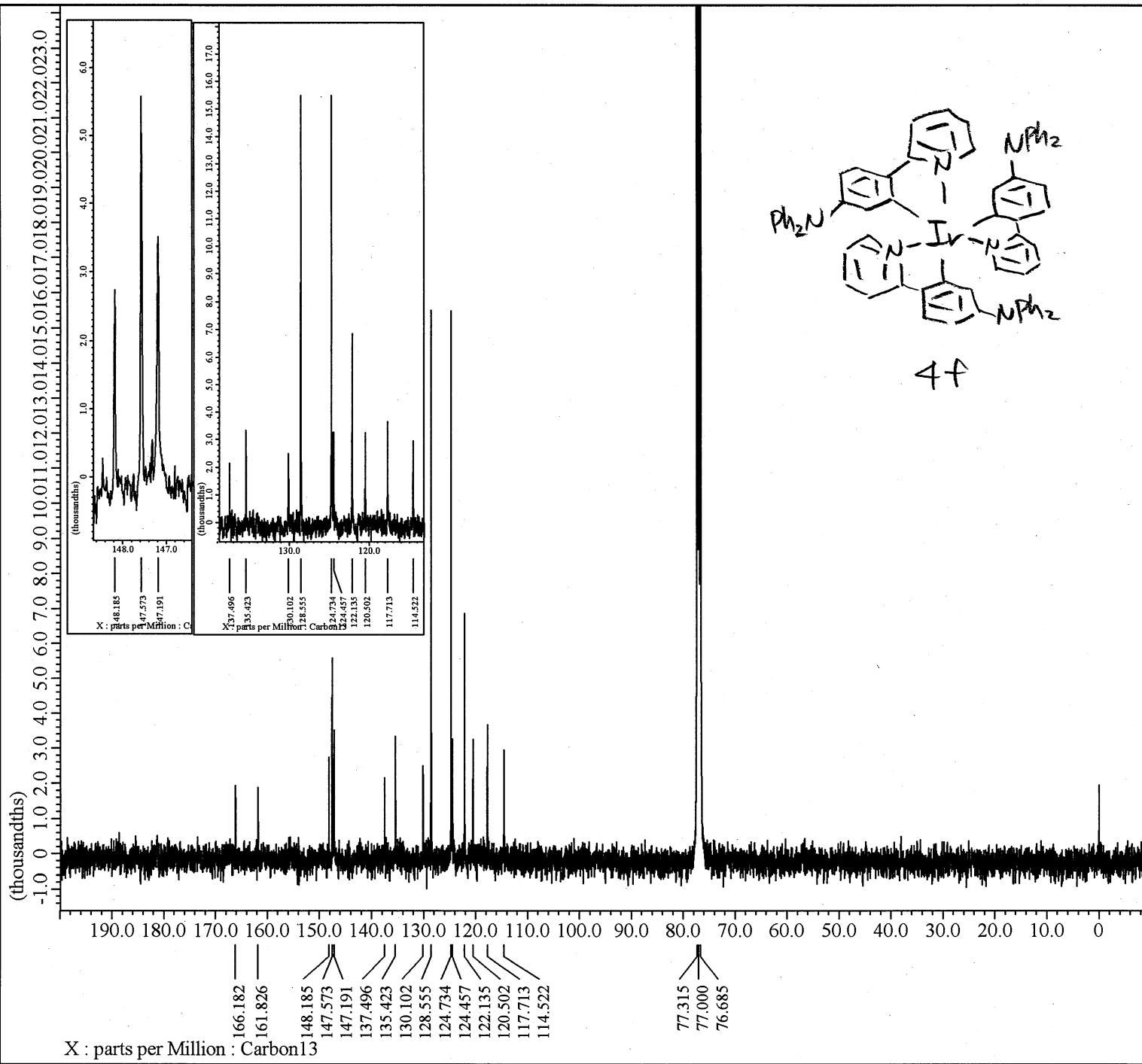
以下に由来: TBR389-redry-1.jdf

Filename = TBR389-redry-3.jdf
 Author = element
 Experiment = single_pulse.ex2
 Sample Id = #529608
 Solvent = CHLOROFORM-D
 Actual_Start Time = 17-OCT-2022 21:22:06
 Revision_Time = 18-OCT-2022 16:50:19

Comment = single_pulse
 Data_Format = 1D_COMPLEX
 Dim Size = 13107
 X_Domain = 1H
 Dim_Title = 1H
 Dim_Units = [ppm]
 Dimensions = X
 Site = ECS 400
 Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390[MHz])
 X_Acq_Duration = 2.228224[s]
 X_Domain = 1H
 X_Freq = 391.78655441[MHz]
 X_Offset = 5[ppm]
 X_Points = 16384
 X_Prescans = 1
 X_Resolution = 0.44878791[Hz]
 X_Sweep = 7.35294118[kHz]
 Irr_Domain = 1H
 Irr_Freq = 391.78655441[MHz]
 Irr_Offset = 5[ppm]
 Tri_Domain = 1H
 Tri_Freq = 391.78655441[MHz]
 Tri_Offset = 5[ppm]
 Clipped = FALSE
 Scans = 8
 Total_Scans = 8

Relaxation_Delay = 5[s]
 Recvr_Gain = 56
 Temp_Get = 19.5[dc]
 X_90_Width = 10.8[us]
 X_Acq_Time = 2.228224[s]
 X_Angle = 45[deg]
 X_Atn = 1.9[dB]
 X_Pulse = 5.4[us]
 Irr_Mode = Off
 Tri_Mode = Off
 Dante_Presat = FALSE



```

---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

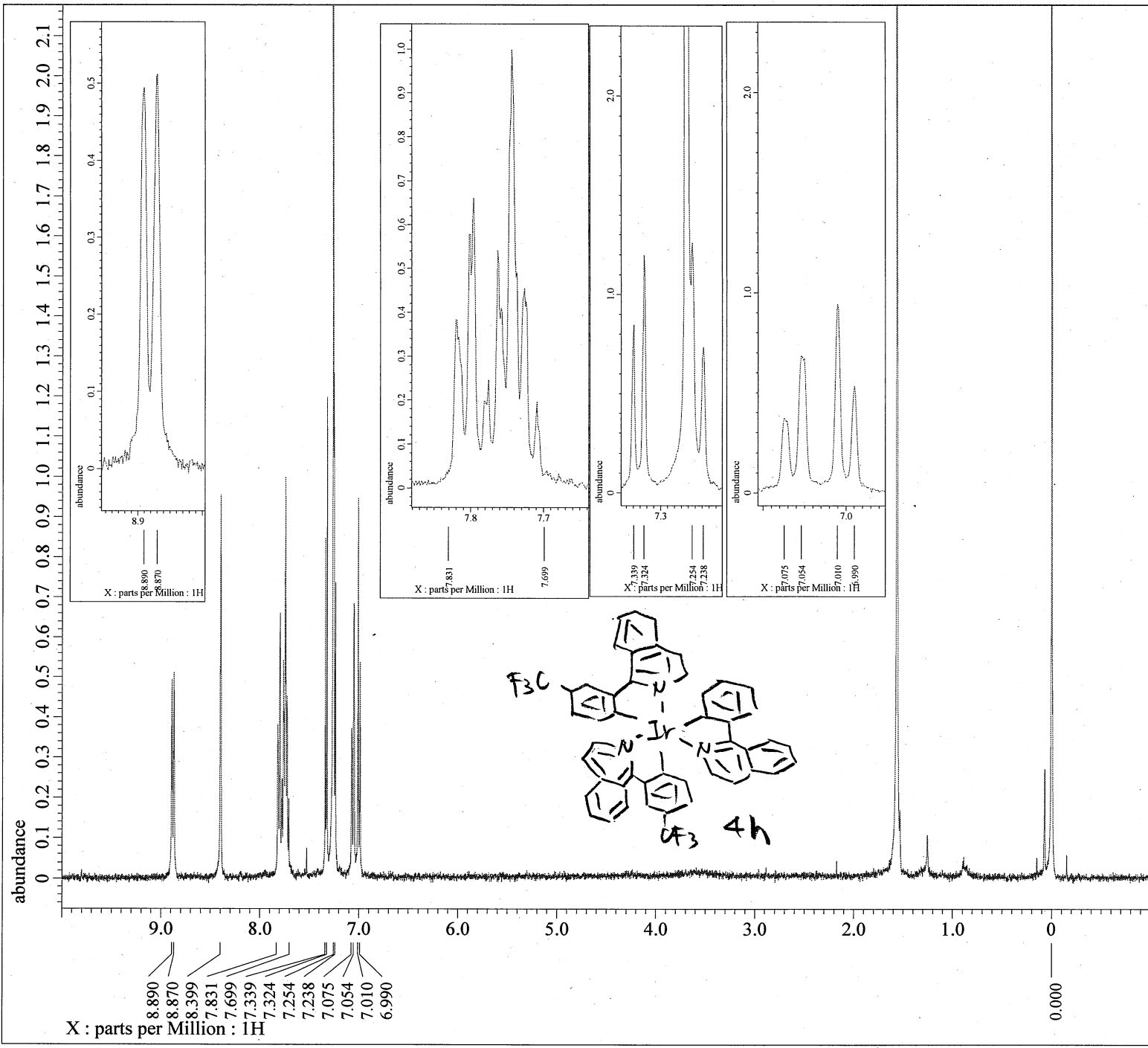
Derived from: TBR389-C_Carbon-1-1.jdf

Filename      = TBR389-C_Carbon-1-2.jdf
Author       = element
Experiment   = carbon.jxp
Sample_Id    = TBR389-C
Solvent      = CHLOROFORM-D
Actual_Start_Time = 25-OCT-2022 22:35:00
Revision_Time  = 30-NOV-2022 11:33:44

Comment      = single pulse decoupled ga
Data_Format  = 1D COMPLEX
Dim_Size     = 26214
X_Domain     = Carbon
Dim_Title    = Carbon13
Dim_Units    = [ppm]
Dimensions   = X
Site         = JNM-ECS400
Spectrometer = DELTA2_NMR

Field_Strength = 9.37221[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain       = 13C
X_Freq         = 100.33735165[MHz]
X_Offset      = 100.0[ppm]
X_Points      = 32768
X_Prescans    = 4
X_Resolution  = 0.95846665[Hz]
X_Sweep       = 31.40703518[kHz]
X_Sweep_Clippped = 25.12562814[kHz]
Irr_Domain    = Proton
Irr_Freq     = 399.03472754[MHz]
Irr_Offset   = 5.0[ppm]
Clipped      = FALSE
Scans        = 11264
Total_Scans   = 11264

Relaxation_Delay = 2[s]
Recvr_Gain       = 50
Temp_Get        = 18.1[dC]
X_90_Width     = 10.9[us]
X_Acq_Time     = 1.04333312[s]
X_Angle        = 30[deg]
X_Atn          = 5.4[dB]
X_Pulse        = 3.63333333[us]
Irr_Atn_Dec    = 25.823[dB]
Irr_Atn_Noe    = 25.823[dB]
Irr_Noise      = WALTZ
Irr_Pwidth     = 0.115[ms]
Decoupling     = TRUE
Initial_Wait   = 1[s]
Noe            = TRUE
Noe_Time       = 2[s]
Repetition_Time = 3.04333312[s]
  
```

```

---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

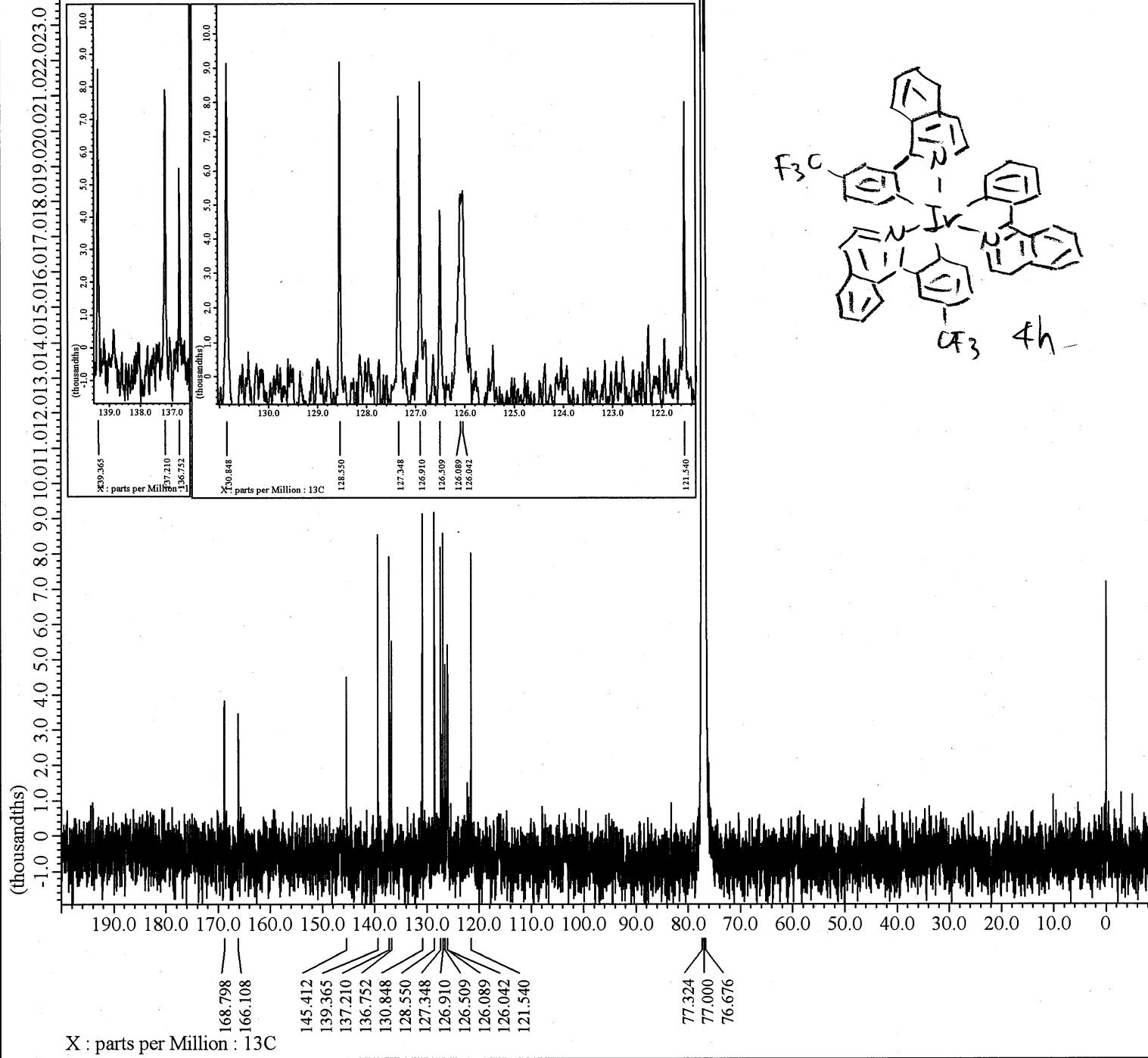
以下に由来: TBR268-re-1.jdf

Filename           = TBR268-re-3.jdf
Author             = element
Experiment         = single_pulse.ex2
Sample Id          = S#692904
Solvent            = CHLOROFORM-D
Actual_Start Time  = 15-OCT-2022 01:54:37
Revision_Time      = 14-OCT-2022 21:39:50

Comment           = single_pulse
Data_Format       = 1D_COMPLEX
Dim_Size          = 13107
X_Domain          = 1H
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 2.228224[s]
X_Domain          = 1H
X_Freq            = 391.78655441[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.44878791[Hz]
X_Sweep           = 7.35294118[kHz]
Irr_Domain        = 1H
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 391.78655441[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 60
Temp_Get          = 20[dC]
X_90_Width        = 10.8[us]
X_Acq_Time        = 2.228224[s]
X_Angle           = 45[deg]
X_Atn             = 1.9[dB]
X_Pulse           = 5.4[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
  
```

```

---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
Derived from: TBR268-C-1.jdf

```

```

Filename      = TBR268-C-2.jdf
Author       = element
Experiment    = single_pulse_dec
Sample_Id    = 1
Solvent      = CHLOROFORM-D
Actual_Start_Time = 25-OCT-2022 03:43:09
Revision_Time  = 30-NOV-2022 14:17:19

```

```

Comment      = single pulse decoupled ga
Data Format   = 1D COMPLEX
Dim Size     = 26214
X Domain     = 13C
Dim Title    = 13C
Dim Units    = [ppm]
Dimensions   = X
Site         = ECS 400
Spectrometer = JNM-ECS400

```

```

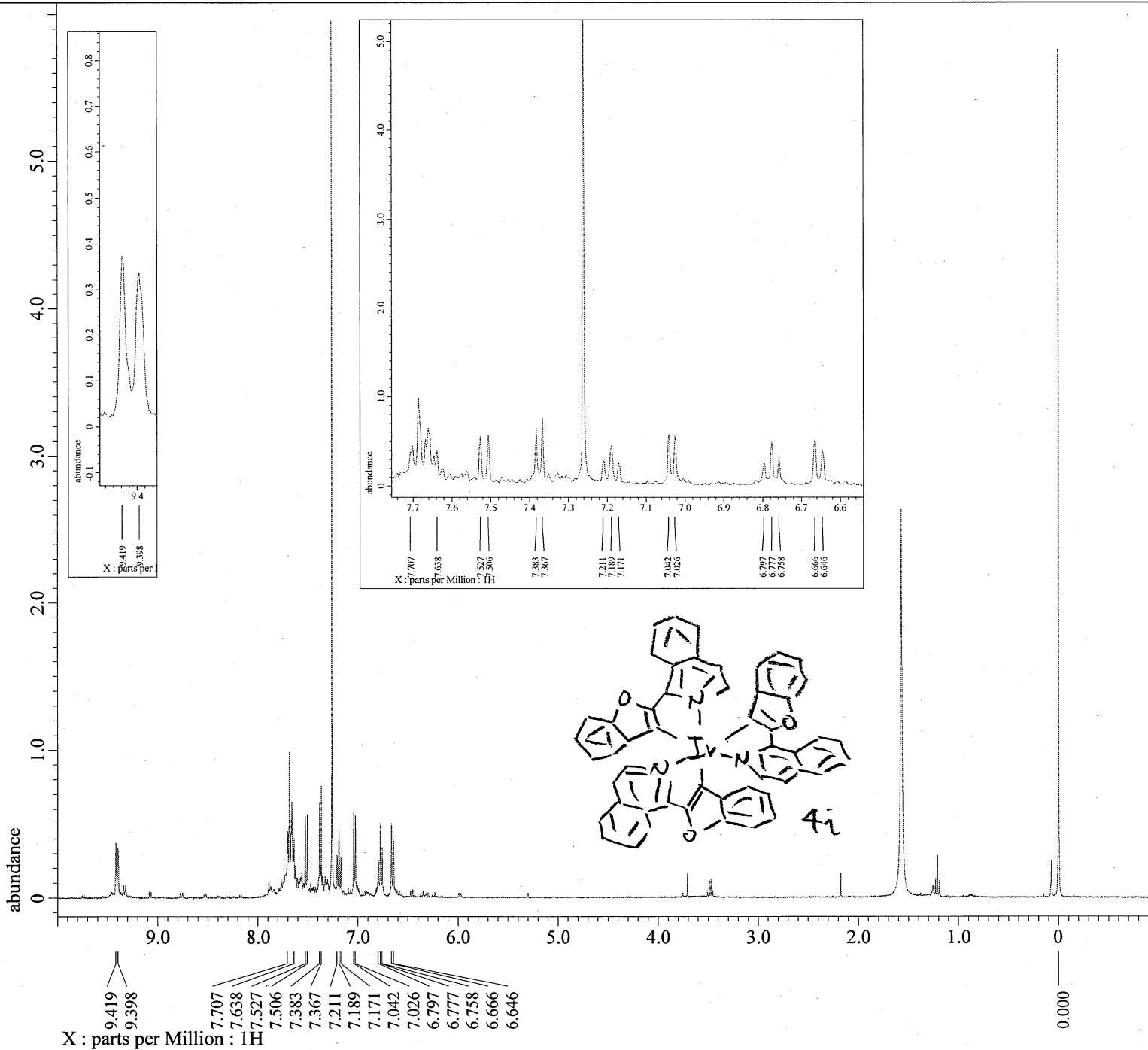
Field Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain       = 13C
X_Freq         = 98.51479726[MHz]
X_Offset       = 100[ppm]
X_Points       = 32768
X_Prescans     = 4
X_Resolution   = 0.93958061[Hz]
X_Sweep        = 30.78817734[kHz]
Irr_Domain     = 1H
Irr_Freq       = 391.78655441[MHz]
Irr_Offset     = 5[ppm]
Clipped        = FALSE
Scans          = 11264
Total_Scans    = 11264

```

```

Relaxation_Delay = 2[s]
Recvr_Gain       = 58
Temp_Get         = 18.4[dC]
X_90_Width      = 8.7[us]
X_Acq_Time       = 1.06430464[s]
X_Angle         = 30[deg]
X_Atn           = 4.9[dB]
X_Pulse         = 2.9[us]
Irr_Atn_Dec     = 22.45[dB]
Irr_Atn_Noec   = 22.45[dB]
Irr_Noise       = WALTZ
Decoupling      = TRUE
Initial_Wait    = 1[s]
Noe              = TRUE
Noe_Time        = 2[s]
Repetition_Time = 3.06430464[s]

```



```

---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

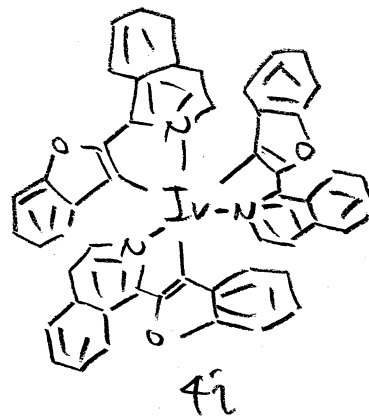
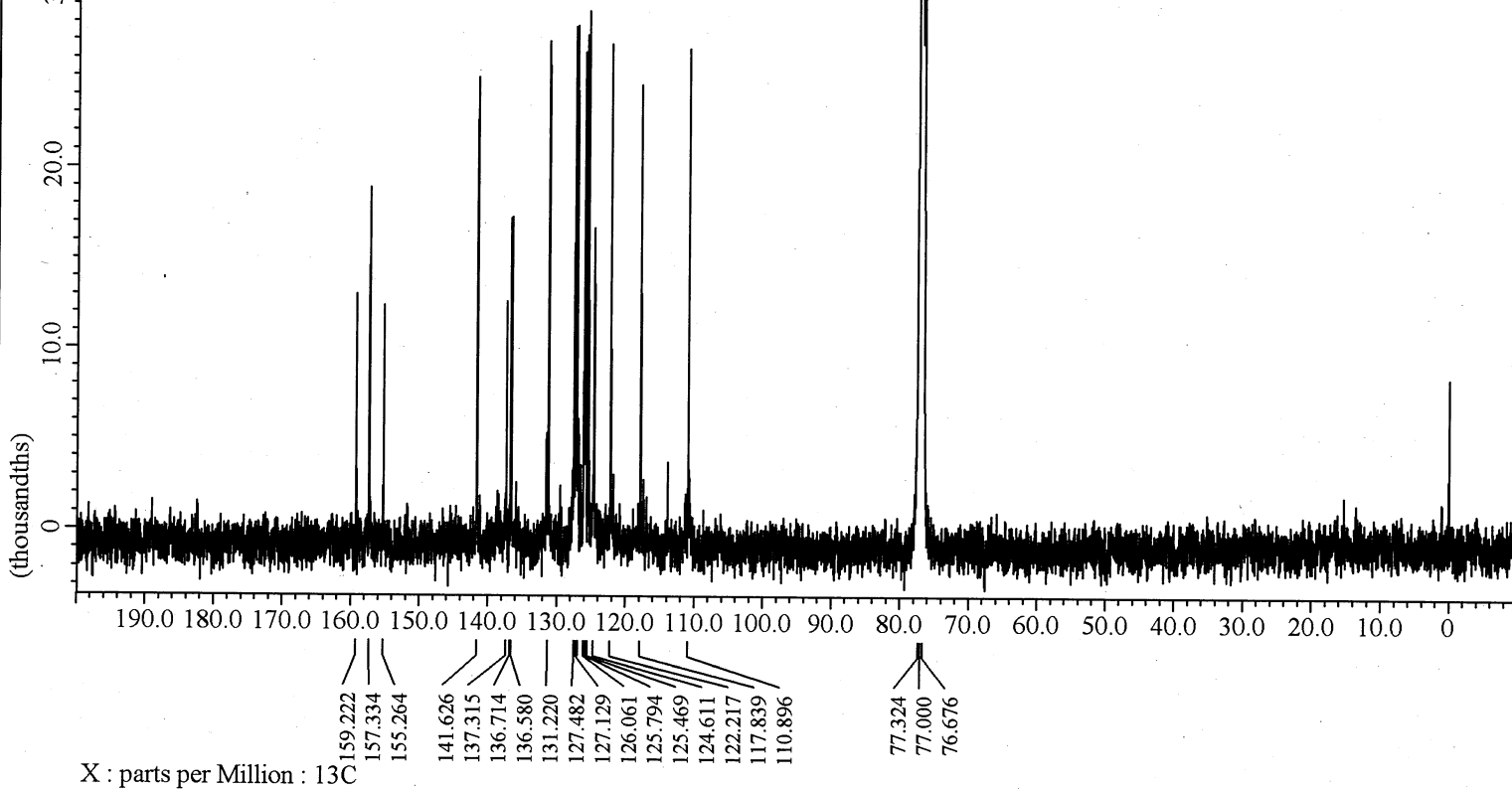
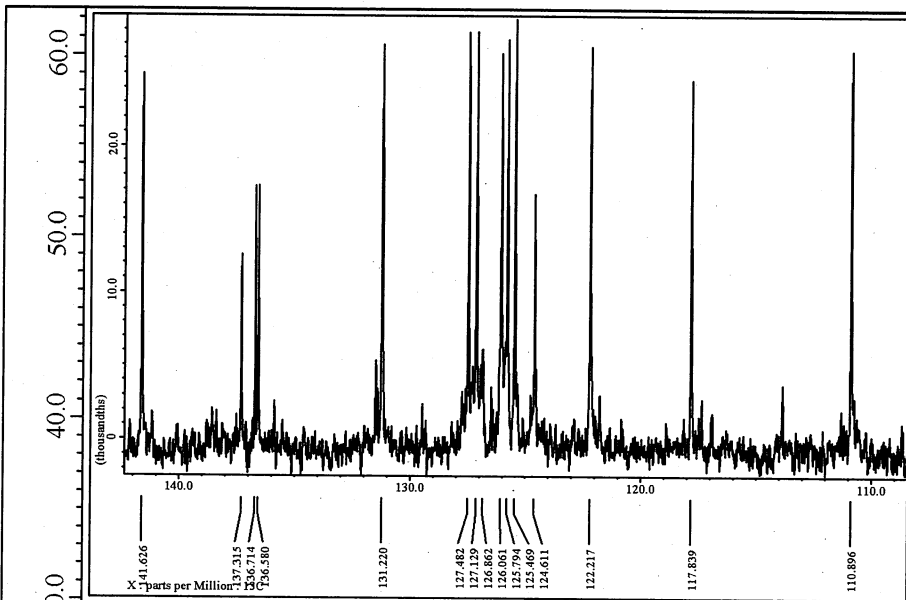
以下に由来: TBR278-reredry-1.jdf

Filename      = TBR278-reredry-3.jdf
Author       = element
Experiment   = single_pulse.ex2
Sample Id    = S#603003
Solvent      = CHLOROFORM-D
Actual_Start_Time = 19-OCT-2022 23:24:07
Revision_Time   = 27-OCT-2022 10:32:08

Comment      = single_pulse
Data Format   = 1D_COMPLEX
Dim Size     = 13107
X_Domain     = 1H
Dim Title    = 1H
Dim Units    = [ppm]
Dimensions   = X
Site         = ECS 400
Spectrometer = JNM-ECS400

Field Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 2.228224[s]
X_Domain       = 1H
X_Freq        = 391.78655441[MHz]
X_Offset      = 5[ppm]
X_Points      = 16384
X_Prescans    = 1
X_Resolution  = 0.44878791[Hz]
X_Sweep       = 7.35294118[kHz]
Irr_Domain    = 1H
Irr_Freq      = 391.78655441[MHz]
Irr_Offset    = 5[ppm]
Tri_Domain    = 1H
Tri_Freq      = 391.78655441[MHz]
Tri_Offset    = 5[ppm]
Clipped       = FALSE
Scans         = 8
Total_Scans   = 8

Relaxation_Delay = 5[s]
Recvr Gain       = 54
Temp_Get        = 19.4[dC]
X_90_Width      = 10.8[us]
X_Acq_Time      = 2.228224[s]
X_Angle         = 45[deg]
X_Atn           = 1.9[dB]
X_Pulse        = 5.4[us]
Irr_Mode        = Off
Tri_Mode        = Off
Dante_Presat    = FALSE
  
```



```

---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

```

Derived from: TBR278-C-1.jdf

```

Filename      = TBR278-C-2.jdf
Author       = element
Experiment    = single_pulse_dec
Sample_Id    = 1
Solvent      = CHLOROFORM-D
Actual_Start_Time = 29-OCT-2022 03:44:40
Revision_Time = 30-NOV-2022 14:19:15

```

```

Comment      = single pulse decoupled ga
Data_Format  = 1D COMPLEX
Dim_Size     = 26214
X_Domain     = 13C
Dim_Title    = 13C
Dim_Units    = [ppm]
Dimensions   = X
Site         = ECS 400
Spectrometer = JNM-ECS400

```

```

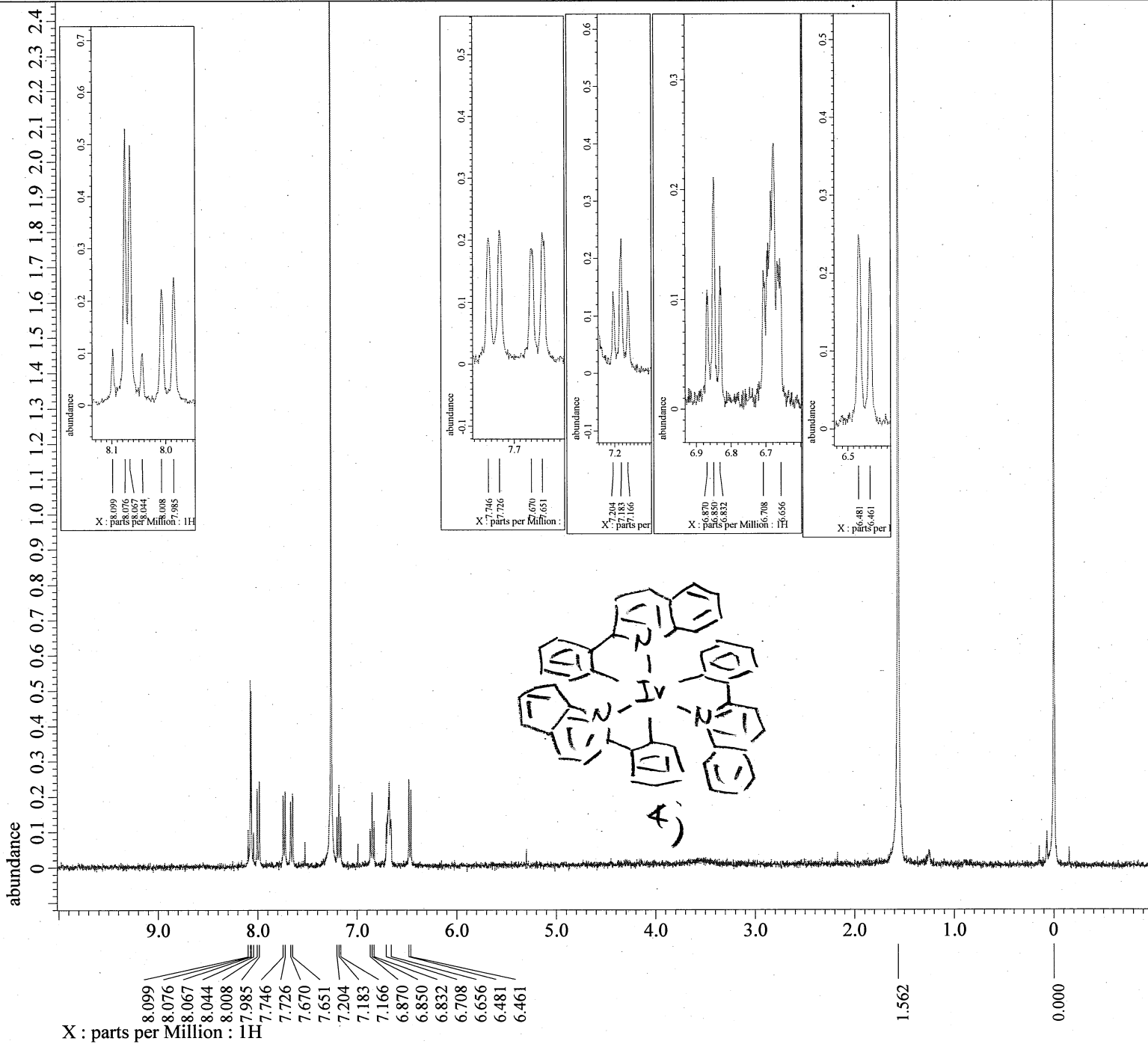
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain       = 13C
X_Freq         = 98.51479726[MHz]
X_Offset       = 100[ppm]
X_Points      = 32768
X_Prescans    = 4
X_Resolution  = 0.93958061[Hz]
X_Sweep       = 30.78817734[kHz]
Irr_Domain     = 1H
Irr_Freq      = 391.78655441[MHz]
Irr_Offset     = 5[ppm]
Clipped       = FALSE
Scans         = 11264
Total_Scans   = 11264

```

```

Relaxation_Delay = 2[s]
Recvr_Gain       = 60
Temp_Get        = 17.9[dC]
X_90_Width      = 8.7[us]
X_Acq_Time      = 1.06430464[s]
X_Angle         = 30[deg]
X_Atn           = 4.9[dB]
X_Pulse         = 2.9[us]
Irr_Atn_Dec     = 22.45[dB]
Irr_Atn_No     = 22.45[dB]
Irr_Noise       = WALTZ
Decoupling      = TRUE
Initial_Wait    = 1[s]
Noe             = TRUE
Noe_Time        = 2[s]
Repetition_Time = 3.06430464[s]

```



```

---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

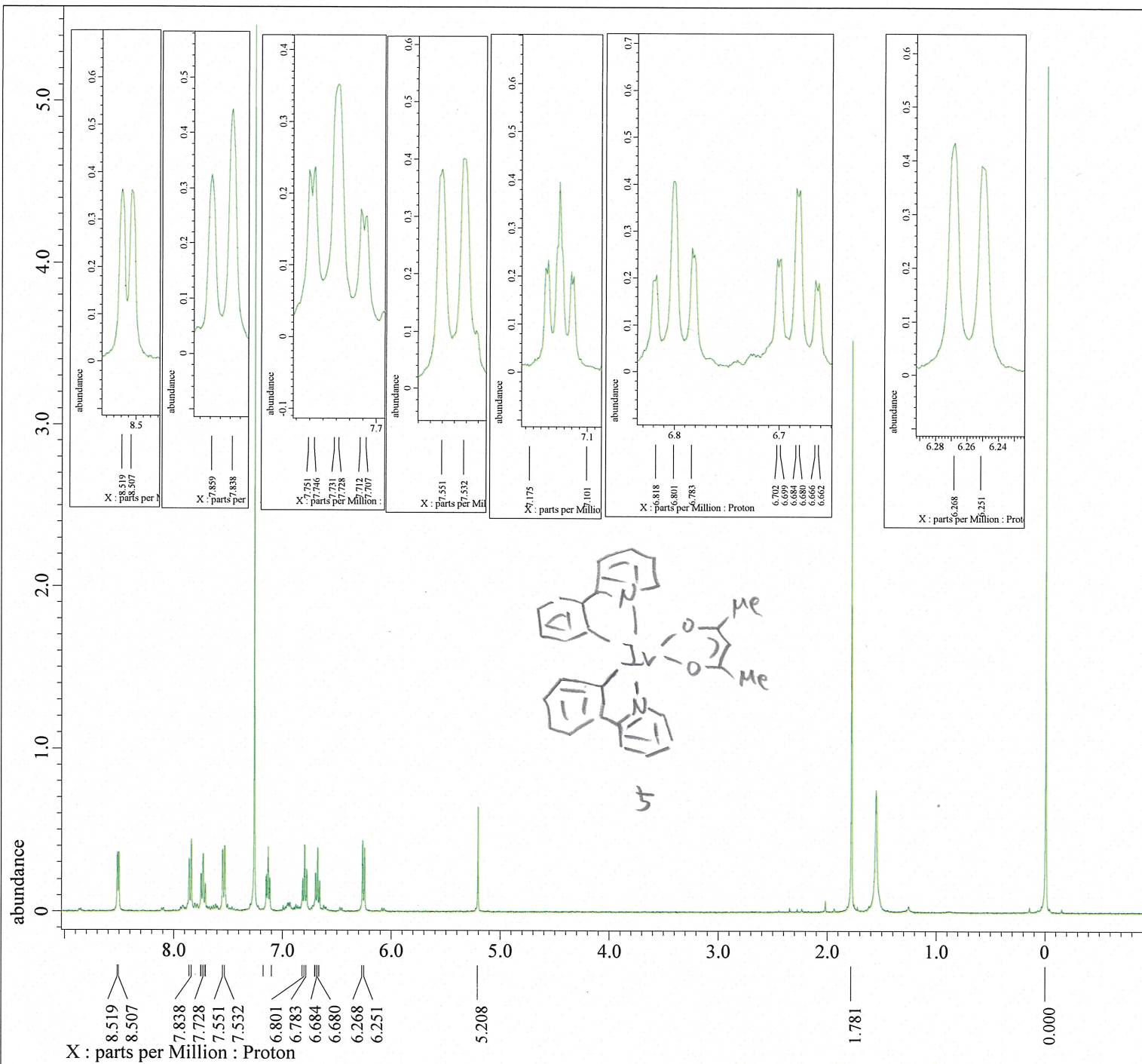
以下に由来: TBR239-re-1.jdf

Filename           = TBR239-re-6.jdf
Author            = element
Experiment        = single_pulse.ex2
Sample Id         = S#688368
Solvent           = CHLOROFORM-D
Actual_Start Time = 15-OCT-2022 01:47:18
Revision Time     = 14-OCT-2022 21:21:13

Comment           = single_pulse
Data_Format       = 1D_COMPLEX
Dim Size          = 13107
X_Domain          = 1H
Dim Title         = 1H
Dim Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration   = 2.228224[s]
X_Domain         = 1H
X_Freq           = 391.78655441[MHz]
X_Offset         = 5[ppm]
X_Points         = 16384
X_Prescans       = 1
X_Resolution     = 0.44878791[Hz]
X_Sweep          = 7.35294118[kHz]
Irr_Domain       = 1H
Irr_Freq         = 391.78655441[MHz]
Irr_Offset       = 5[ppm]
Tri_Domain       = 1H
Tri_Freq         = 391.78655441[MHz]
Tri_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 8
Total_Scans      = 8

Relaxation_Delay = 5[s]
Recvr_Gain       = 60
Temp_Get         = 20.1[dc]
X_90_Width       = 10.8[us]
X_Acq_Time       = 2.228224[s]
X_Angle          = 45[deg]
X_Atn            = 1.9[db]
X_Pulse          = 5.4[us]
Irr_Mode         = Off
Tri_Mode         = Off
Dante_Presat     = FALSE
  
```



```

---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zeroFill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: TBR727-paper_Proton-1-1.jdf

```

```

Filename      = TBR727-paper_Proton-1-9.j
Author       = element
Experiment   = proton.jxp
Sample_Id    = TBR727-paper
Solvent      = CHLOROFORM-D
Actual_Start_Time = 14-MAR-2023 14:40:51
Revision_Time   = 14-MAR-2023 13:59:56

Comment      = single_pulse
Data_Format  = 1D COMPLEX
Dim_Size     = 13107
X_Domain     = Proton
Dim_Title    = Proton
Dim_Units    = [ppm]
Dimensions   = X
Site         = JNM-ECS400
Spectrometer = DELTA2_NMR

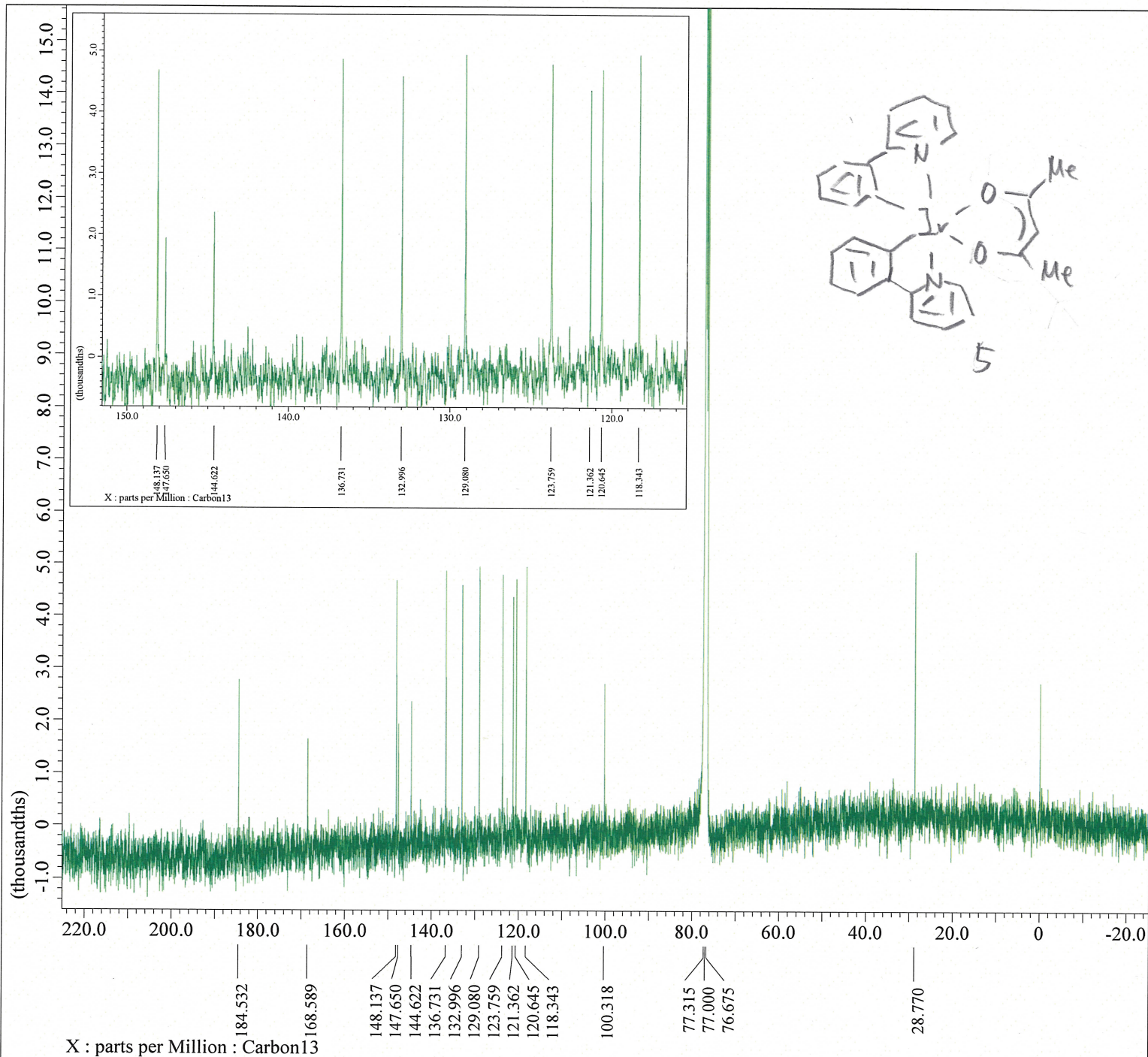
```

```

Field_Strength = 9.37221 [T] (400[MHz])
X_Acq_Duration = 2.1889024 [s]
X_Domain       = 1H
X_Freq         = 399.03472754 [MHz]
X_Offset       = 5.0 [ppm]
X_Points       = 16384
X_Prescans     = 1
X_Resolution   = 0.45684997 [Hz]
X_Sweep        = 7.48502994 [kHz]
X_Sweep_Clippped = 5.98802395 [kHz]
Irr_Domain     = Proton
Irr_Freq       = 399.03472754 [MHz]
Irr_Offset     = 5.0 [ppm]
Tri_Domain     = Proton
Tri_Freq       = 399.03472754 [MHz]
Tri_Offset     = 5.0 [ppm]
Clipped        = FALSE
Scans          = 8
Total_Scans    = 8

Relaxation_Delay = 5 [s]
Recvr_Gain       = 50
Temp_Get         = 19.5 [dC]
X_90_Width      = 6.6 [us]
X_Acq_Time      = 2.1889024 [s]
X_Angle         = 45 [deg]
X_Atn           = 1 [dB]
X_Pulse         = 3.3 [us]
Irr_Mode        = Off
Tri_Mode        = Off
Dante_Presat    = FALSE
Initial_Wait    = 1 [s]
Repetition_Time = 7.1889024 [s]

```



```

---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
Derived from: TBR727_Carbon-1-1.jdf

```

```

Filename      = TBR727_Carbon-1-2.jdf
Author       = element
Experiment   = carbon.jxp
Sample Id    = TBR727
Solvent      = CHLOROFORM-D
Actual Start Time = 9-MAR-2023 22:16:20
Revision Time = 14-MAR-2023 16:17:53

```

```

Comment      = single pulse decoupled ga
Data Format   = 1D COMPLEX
Dim Size     = 26214
X_Domain     = Carbon
Dim Title    = Carbon13
Dim Units    = [ppm]
Dimensions   = X
Site         = JNM-ECS400
Spectrometer = DELTA2_NMR

```

```

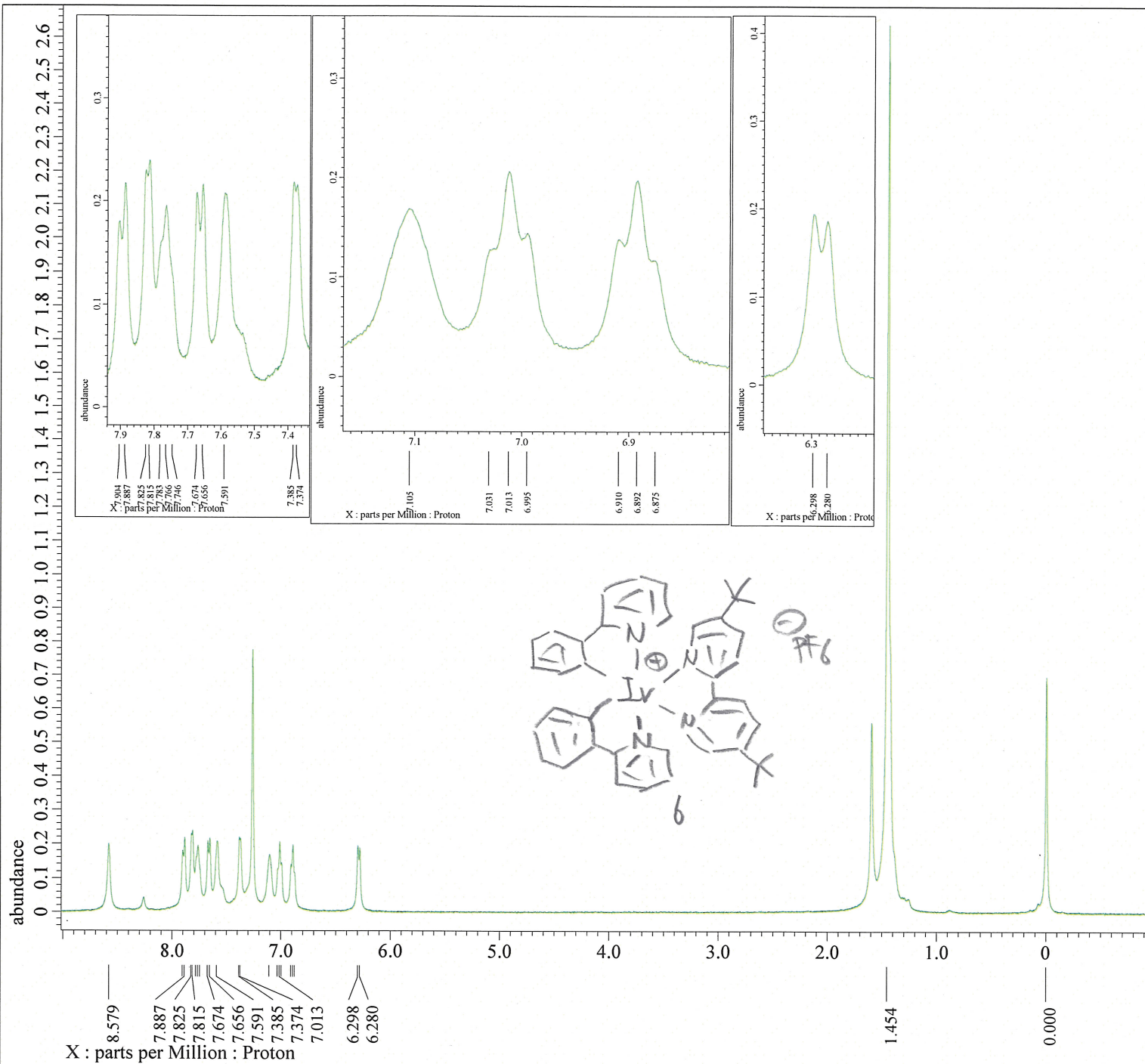
Field Strength = 9.37221[T] (400[MHz])
X_Acq Duration = 1.04333312[s]
X_Domain       = 13C
X_Freq         = 100.33735165[MHz]
X_Offset       = 100.0[ppm]
X_Points       = 32768
X_Prescans     = 4
X_Resolution   = 0.95846665[Hz]
X_Sweep        = 31.40703518[kHz]
X_Sweep_Clip   = 25.12562814[kHz]
Irr_Domain     = Proton
Irr_Freq       = 399.03472754[MHz]
Irr_Offset     = 5.0[ppm]
Clipped        = FALSE
Scans          = 10254
Total_Scans    = 10254

```

```

Relaxation_Delay = 2[s]
Recvr_Gain       = 50
Temp_Get         = 19.4[dC]
X_90_Width       = 10.9[us]
X_Acq_Time       = 1.04333312[s]
X_Angle          = 30[deg]
X_Atn            = 5.4[dB]
X_Pulse          = 3.63333333[us]
Irr_Atn_Dec     = 25.823[dB]
Irr_Atn_No     = 25.823[dB]
Irr_Noise       = WALTZ
Irr_Pwidth      = 0.115[ms]
Decoupling       = TRUE
Initial_Wait     = 1[s]
Noe              = TRUE
Noe_Time        = 2[s]
Repetition_Time = 3.04333312[s]

```



```

---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: TBR724-paper-re_Proton-1-1.jdf

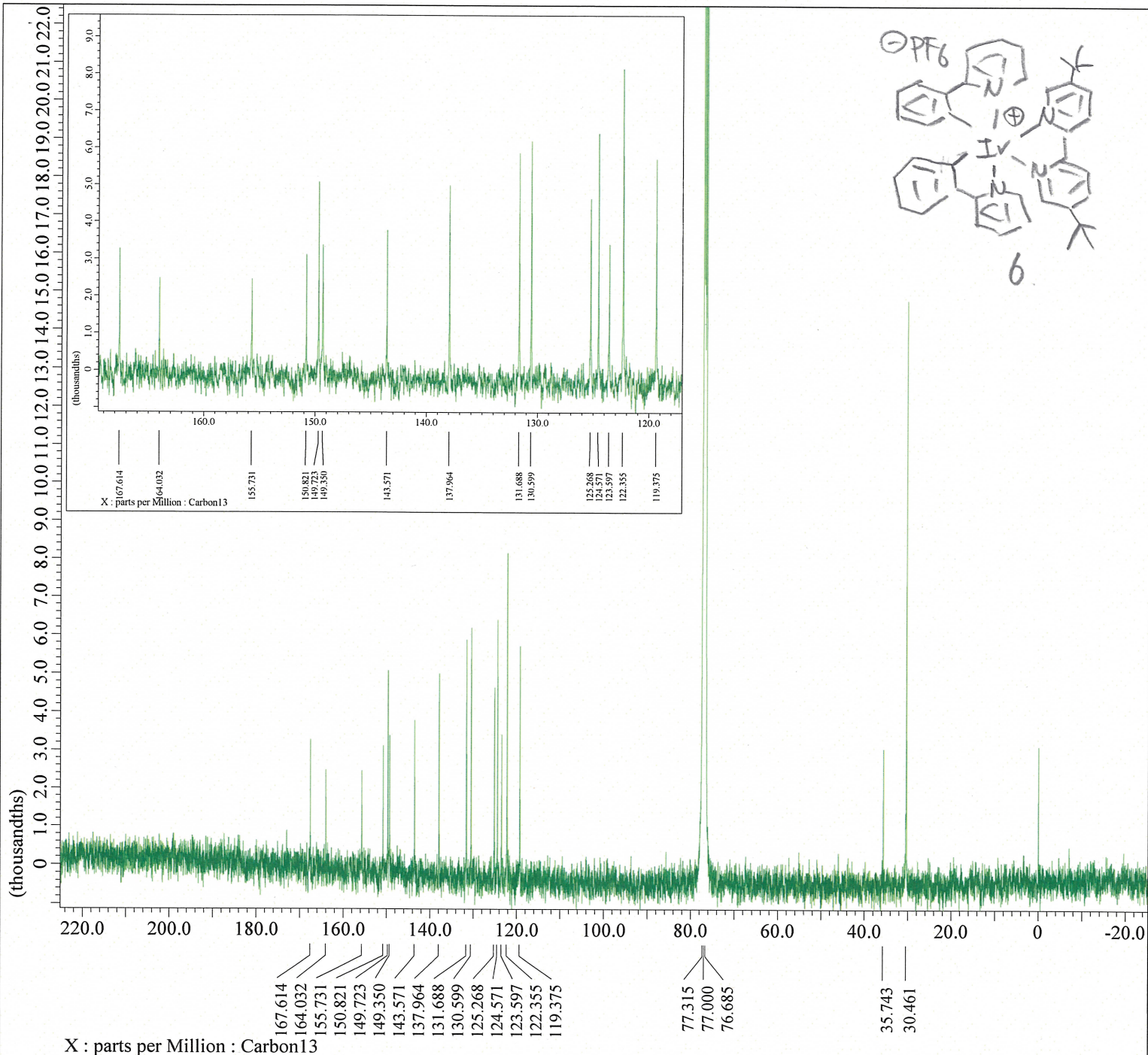
Filename      = TBR724-paper-re_Proton-1-
Author       = element
Experiment   = proton.jxp
Sample Id    = TBR724-paper-re
Solvent      = CHLOROFORM-D
Actual_Start_Time = 16-MAR-2023 10:02:34
Revision_Time   = 16-MAR-2023 10:18:27

Comment      = single_pulse
Data Format   = 1D_COMPLEX
Dim Size     = 13107
X Domain     = Proton
Dim Title    = Proton
Dim Units    = [ppm]
Dimensions   = X
Spectrometer = DELTA2_NMR

Field_Strength = 9.4073814[T] (400[MHz])
X_Acq_Duration = 2.18103808[s]
X_Domain       = 1H
X_Freq        = 400.53219825[MHz]
X_Offset      = 5[ppm]
X_Points      = 16384
X_Prescans    = 1
X_Resolution  = 0.45849727[Hz]
X_Sweep       = 7.51201923[kHz]
X_Sweep_Clippped = 6.00961538[kHz]
Irr_Domain    = Proton
Irr_Freq      = 400.53219825[MHz]
Irr_Offset    = 5[ppm]
Tri_Domain    = Proton
Tri_Freq      = 400.53219825[MHz]
Tri_Offset    = 5[ppm]
Clipped       = FALSE
Scans         = 8
Total_Scans   = 8

Relaxation_Delay = 5[s]
Recvr_Gain       = 46
Temp_Get         = 18.9[dC]
X_90_Width      = 6.7[us]
X_Acq_Time       = 2.18103808[s]
X_Angle         = 45[deg]
X_Atn           = 0.8[dB]
X_Pulse         = 3.35[us]
Irr_Mode        = Off
Tri_Mode        = Off
Dante_Presat    = FALSE
Initial_Wait    = 1[s]
Repetition_Time = 7.18103808[s]

```



```

---- PROCESSING PARAMETERS ----
dc balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zeroFill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: TBR724-C_Carbon-1-1.jdf

```

```

Filename      = TBR724-C_Carbon-1-2.jdf
Author       = element
Experiment   = carbon.jxp
Sample_Id    = TBR724-C
Solvent      = CHLOROFORM-D
Actual_Start_Time = 16-MAR-2023 22:23:38
Revision_Time  = 17-MAR-2023 09:55:33

Comment      = single pulse decoupled ga
Data_Format  = 1D COMPLEX
Dim_Size     = 26214
X_Domain     = Carbon
Dim_Title    = Carbon13
Dim_Units    = [ppm]
Dimensions   = X
Site         = JNM-ECS400
Spectrometer = DELTA2_NMR

```

```

Field_Strength = 9.37221[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain       = 13C
X_Freq         = 100.33735165[MHz]
X_Offset       = 100.0[ppm]
X_Points       = 32768
X_Prescans     = 4
X_Resolution   = 0.95846665[Hz]
X_Sweep        = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain     = Proton
Irr_Freq       = 399.03472754[MHz]
Irr_Offset     = 5.0[ppm]
Clipped        = FALSE
Scans          = 10024
Total_Scans    = 10024

```

```

Relaxation_Delay = 2[s]
Recvr_Gain        = 50
Temp_Get          = 18.5[dC]
X_90_Width        = 10.9[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 5.4[dB]
X_Pulse           = 3.63333333[us]
Irr_Atn_Dec       = 25.823[dB]
Irr_Atn_Noise    = 25.823[dB]
Irr_Noise        = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]

```