

## **Solid-state cross-coupling reactions of insoluble aryl halides under polymer-assisted grinding conditions**

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## 1. Chemicals and Instrumentation

The starting materials and the polymers were obtained from commercial suppliers and used as received. Solvents were purchased from commercial suppliers, and further dried over molecular sieves (MS 4Å). All mechanochemical reactions were carried out using grinding vessels in a Retsch MM400 mill (Figure S1). Both jars (1.5 mL) and balls (5 mm) are made of stainless (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 ( $^1\text{H}$ : 392 or 396 or 399 or 401 MHz,  $^{13}\text{C}$ : 99 or 100 MHz). Tetramethylsilane ( $^1\text{H}$ ) and  $\text{CDCl}_3$  ( $^{13}\text{C}$ ) were 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. 1,1,2,2-Tetrachloroethane was used as an internal standard to determine NMR yields. Powder diffraction data were recorded on a Rigaku SmartLab diffractometer with  $\text{Cu-K}_\alpha$  radiation and D/teX Ultra detector covering 5-60° (2 $\theta$ ). Scanning electron microscopy (SEM) analysis was carried out with JEOL JSM-6510LV. High-resolution mass spectra were recorded at the Global Facility Center, Hokkaido University.



**Figure S1.** Retsch MM400 used in this study.



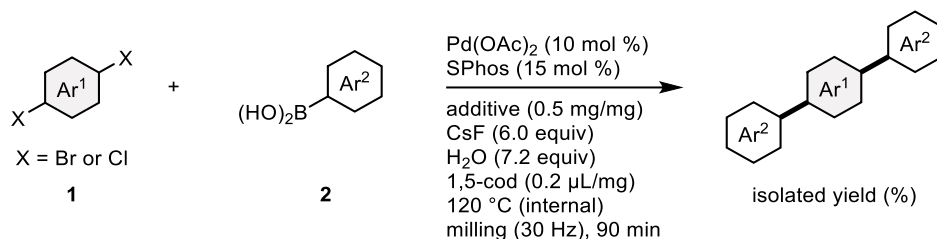
**Figure S2.** Stainless jar and ball used in this study.



**Figure S3.** The temperature controllable heat gun Takagi HG-1450B used in this study.

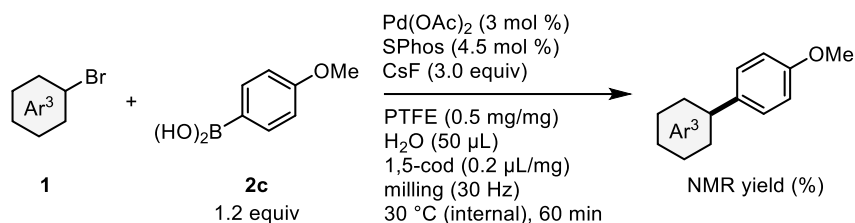
## 2. General Procedure for Mechanochemical Cross-Coupling.

### For poorly soluble aryl halides (1a–1d)



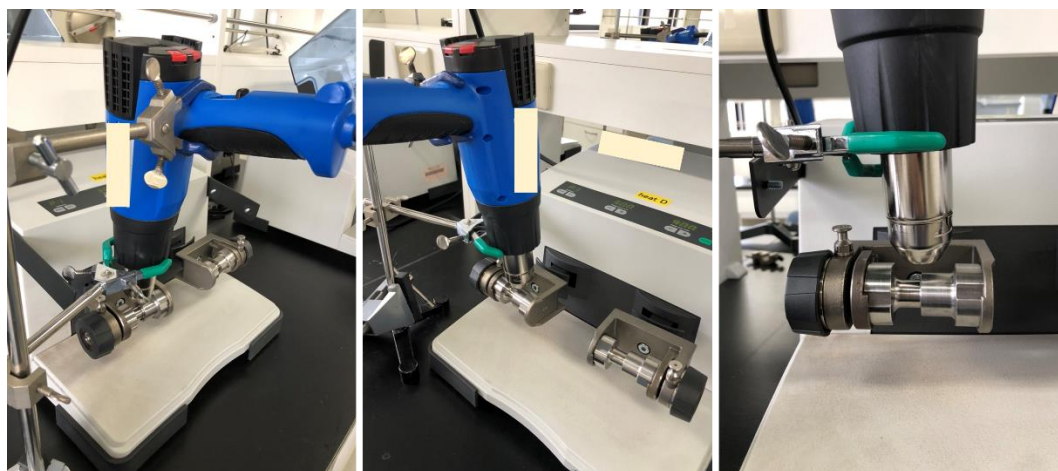
Aryl halide **1** (0.1 mmol), aryl boronic acid **2** (0.24 mmol, 2.4 equiv), Pd(OAc)<sub>2</sub> (0.01 mmol, 10 mol %) and SPhos (0.015 mmol, 15 mol %), CsF (0.6 mmol, 6.0 equiv), additive (0.5 mg/mg) were placed in a ball milling vessel (stainless, 1.5 mL) loaded with one grinding ball (stainless, diameter: 5 mm). Then H<sub>2</sub>O (50 μL) and 1,5-cod (0.2 μL/mg) were added via syringe. After the vessel was closed in air without purging with inert gas, the vessel was placed in the ball mill (Retsch MM400, 90 min at 30 Hz) and a heat gun (the preset temperature at 250 °C). After 90 min, the jar was then cooled rapidly with cold water and opened. The mixture was filtration with CH<sub>2</sub>Cl<sub>2</sub> to remove the starting material and inorganic salts. The crude mixture was then purified by flash column chromatography (SiO<sub>2</sub>, typically CH<sub>2</sub>Cl<sub>2</sub>/hexane, typically 0:100–100:0) to give the corresponding coupling product **3**.

### For soluble aryl halides (1e–1g)

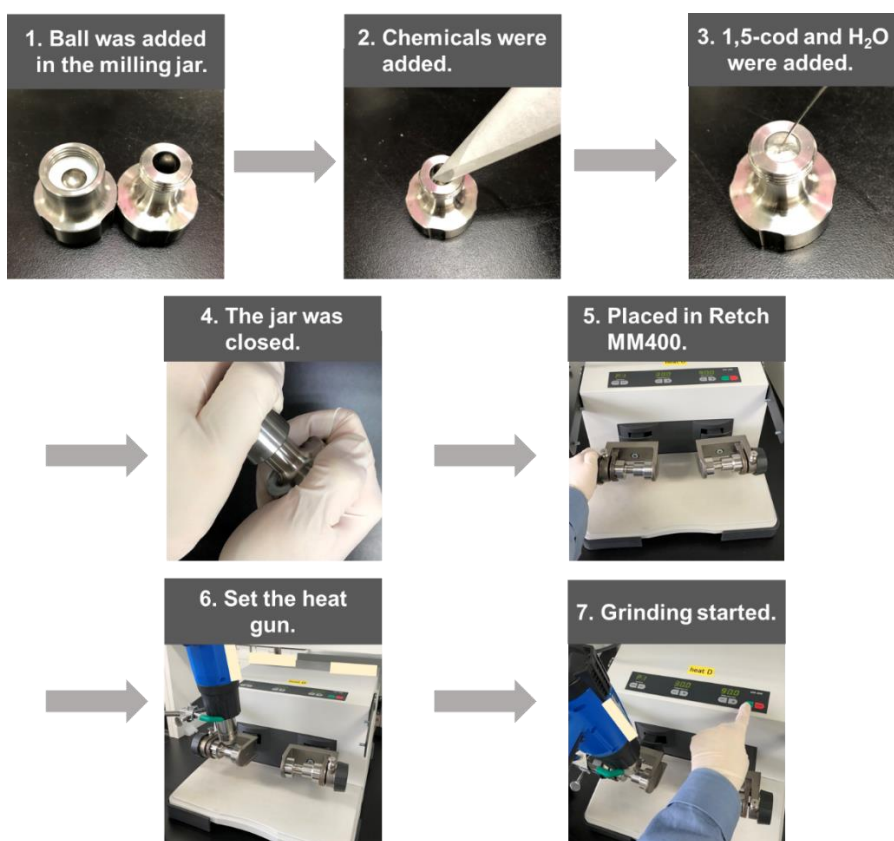


Aryl halide **1** (0.2 mmol), aryl boronic acid **2c** (0.24 mmol, 1.2 equiv), Pd(OAc)<sub>2</sub> (0.006 mmol, 3 mol %) and SPhos (0.009 mmol, 4.5 mol %), CsF (0.6 mmol, 3.0 equiv), PTFE (0.5 mg/mg) were placed in a ball milling vessel (stainless, 1.5 mL) loaded with one grinding ball (stainless, diameter: 5 mm). Then H<sub>2</sub>O (50 μL) and 1,5-cod (0.2 μL/mg) were added via syringe. After the vessel was closed in air without purging with inert gas, the vessel was placed in the ball mill (Retch MM400, 60 min at 30 Hz). After 60 min, the mixture was passed through a short silica gel column eluting with CH<sub>2</sub>Cl<sub>2</sub> to remove inorganic salts.

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 S4). The set-up procedure for high-temperature ball-milling reactions was shown in Figure S5. First, one grinding ball (stainless, diameter: 5 mm) was loaded in a ball milling jar (stainless, 1.5 mL). Then solid and liquid materials were added to the jar. After the ball milling jar was closed, the jar was placed in the ball mill (Retsch MM400), and a heat gun was placed directly above the ball-milling jar. The mechanochemical cross-coupling reactions were conducted while applying heated air to the outside of the milling jar (the preset temperature at 250 °C). The internal temperature was confirmed by thermography immediately after opening the jar.



**Figure S4.** The set-up procedure for a heat gun on MM400.

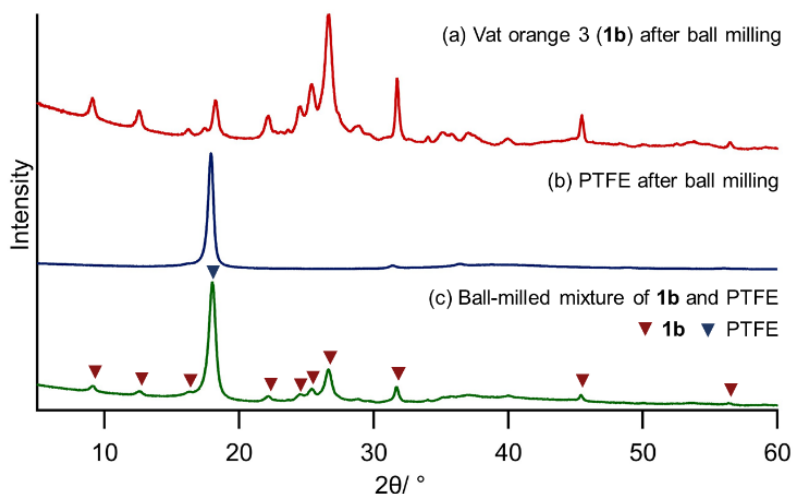


**Figure S5.** The set-up procedure of mechanochemical cross-coupling reactions using a heat gun.

### 3. Details of Powder X-Ray Diffraction Analysis

The sample was prepared under the following conditions:

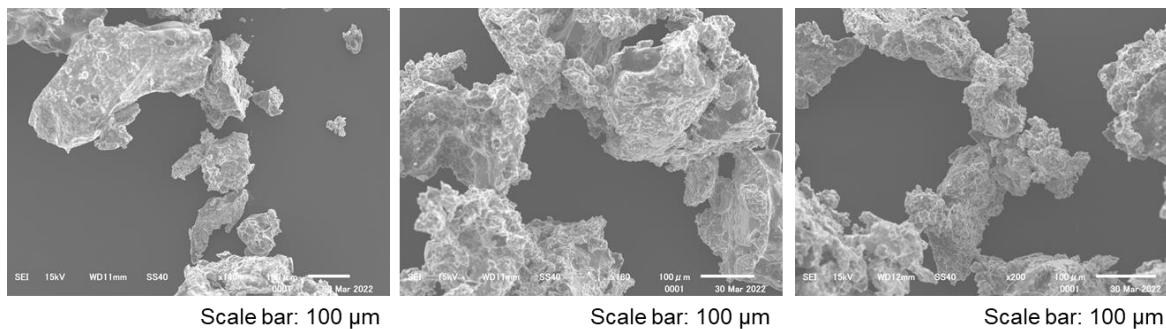
- (a) **1b** (100 mg) was placed in a ball milling vessel (stainless, 1.5 mL) loaded with one grinding ball (stainless, diameter: 5 mm). After the vessel was closed, the vessel was placed in the ball mill (90 min at 30 Hz) and a heat gun (the preset temperature at 250 °C).
- (b) PTFE (100 mg) was placed in a ball milling vessel (stainless, 1.5 mL) loaded with one grinding ball (stainless, diameter: 5 mm). After the vessel was closed, the vessel was placed in the ball mill (90 min at 30 Hz) and a heat gun (the preset temperature at 250 °C).
- (c) **1b** (46.4 mg) and PTFE (100 mg) were placed in a ball milling vessel (stainless, 1.5 mL) loaded with one grinding ball (stainless, diameter: 5 mm). After the vessel was closed, the vessel was placed in the ball mill (90 min at 30 Hz) and a heat gun (the preset temperature at 250 °C).



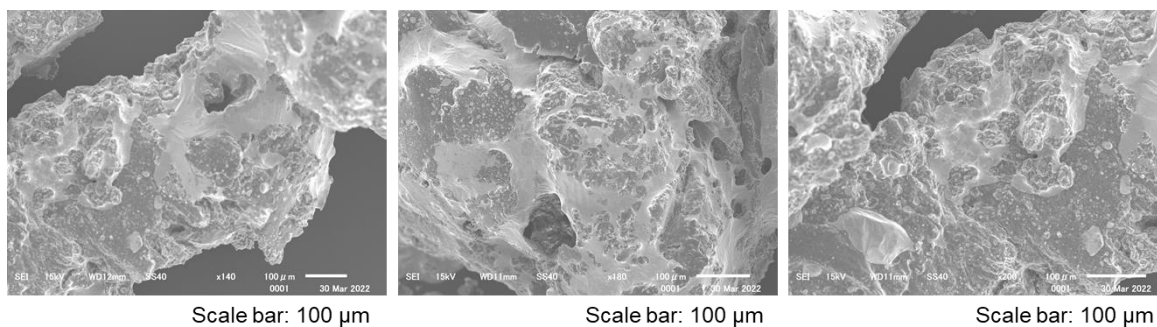
**Figure S6.** PXRD analysis of the ball-milled mixture of **1b** and PTFE.

#### 4. Details of Scanning Electron Microscopy

The sample was prepared by the following conditions: 0.1 mmol of **1a**; 0.24 mmol of **2a**; 0.01 mmol of Pd(OAc)<sub>2</sub>; 0.015 mmol of SPhos; 0.6 mmol of CsF; H<sub>2</sub>O (50 μL), 1,5-cod (0.2 μL/mg) and PTFE (0.5 mg/mg) in a stainless-steel ball-milling jar (1.5 mL) with a stainless-steel ball (5 mm) ; heat gun (set temp.; 250 °C); 30 Hz; 90 min. The samples for the characterization by scanning electron microscopy (SEM) were prepared by platinum coating with thin carbon film.



**Figure S7.** SEM analysis of the crude mixture of **1a** and **2a** under ball milling conditions without PTFE.



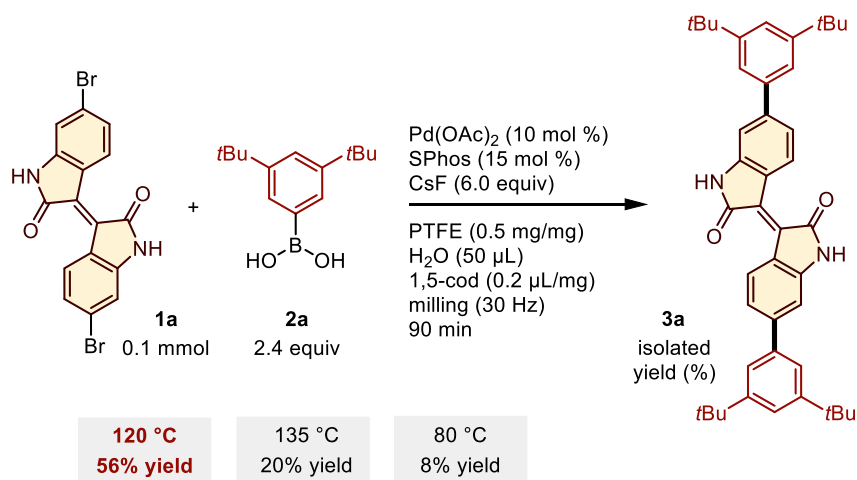
**Figure S8.** SEM analysis of the crude mixture of **1a** and **2a** under ball milling conditions with PTFE.



## 5. Effect of Reaction Temperature

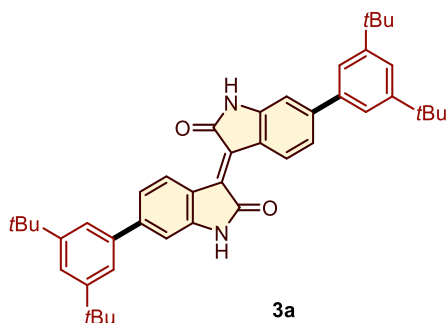
Reactions of 6,6-dibromoisoindigo (**1a**) under POLAG conditions using PTFE at temperatures higher (135 °C) or lower (80 °C) than 120 °C were carried out, but both were found to reduce the yields (20% and 8% yields, respectively). This result suggests that the acceleration effect of PTFE addition is very sensitive to reaction temperature and seems to be more pronounced at temperatures of 120°C.

**Scheme S1.** Solid-state cross-coupling under POLAG conditions at different temperature.



## 6. Characterization of Obtained Coupling Products

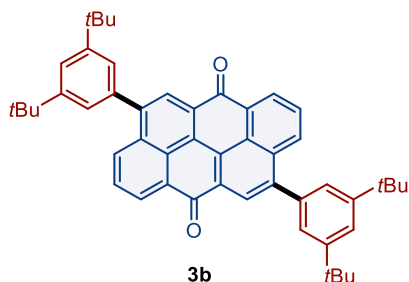
### (*E*)-6,6'-Bis(3,5-di-*tert*-butylphenyl)-[3,3'-biindolinylidene]-2,2'-dione (**3a**).



The reaction was carried out with 42.2 mg (0.10 mmol) of **1a** and 56.6 mg (0.24 mmol) of **2a**. The product **3a** was obtained as a red powder (35.8 mg, 0.056 mmol, 56% yield) after purification by silica-gel column chromatography (SiO<sub>2</sub>, CH<sub>2</sub>Cl<sub>2</sub>/hexane, 60:40–0:100). <sup>1</sup>H NMR were in agreement with the literature.<sup>1</sup>

<sup>1</sup>H NMR (401 MHz, CDCl<sub>3</sub>, δ): 1.39 (s, 36H), 7.06 (d, *J* = 2.0 Hz, 2H), 7.31 (dd, *J* = 1.8, 8.2 Hz, 2H), 7.42–7.52 (m, 6H), 7.69 (s, 2H), 9.19 (d, *J* = 8.4 Hz, 2H).

### 4,10-Bis(3,5-di-*tert*-butylphenyl)naphtho[7,8,1,2,3-nopqr]tetraphene-6,12-dione (**3b**).

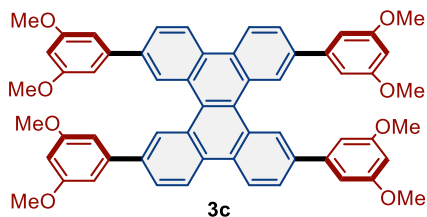


The reaction was carried out with 24.0 mg (0.05 mmol) of **1b** and 29.0 mg (0.12 mmol) of **2a**. The product **3b** was obtained as a red powder (28.0 mg, 0.041 mmol, 79% yield) after purification by silica-gel column chromatography (SiO<sub>2</sub>, CH<sub>2</sub>Cl<sub>2</sub>/hexane, 0:100–40:60). The <sup>1</sup>H NMR spectrum of the product was in agreement with the literature.<sup>1</sup> We carried out the reaction of **1b** several times, and **3b** was obtained in between 79% and 99% yield. This is presumably due to impurities in **1b** or to the low reproducibility of the mixing efficiency of the polymer and the substrate under the present ball-milling conditions.

<sup>1</sup>H NMR (392 MHz, CDCl<sub>3</sub>, δ): 1.43 (s, 36H), 7.44 (d, *J* = 2.0 Hz, 4H), 7.58 (t, *J* = 1.8 Hz, 2H), 7.79 (t, *J* = 7.8 Hz, 2H), 8.36 (d, *J* = 8.6 Hz, 2H), 8.48 (d, *J* = 2.0 Hz, 2H), 8.71 (d, *J* = 7.4 Hz, 2H).

**Note:** The chemical shifts of **3b** in <sup>1</sup>H NMR spectroscopy changed depending on the sample concentration probably because of the aggregation of **3b**. Here we measured the sample of 1.0 × 10<sup>-3</sup> M and the above chemical shifts were obtained.

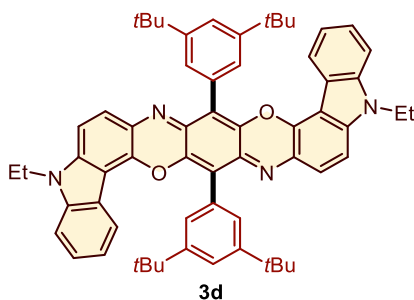
**3,6,11,14-Tetrakis(3,5-dimethoxyphenyl)dibenzo[g,p]chrysene (3c).**



The reaction was carried out with 32.2 mg (0.05 mmol) of **1c** and 43.8 mg (0.48 mmol) of **2b**. The product **3c** was obtained as a white powder (38.5 mg, 0.044 mmol, 88% yield) after purification by silica-gel column chromatography (SiO<sub>2</sub>, CH<sub>2</sub>Cl<sub>2</sub>/hexane, 20:80–100:0).

<sup>1</sup>H NMR (392 MHz, CDCl<sub>3</sub>, δ): 3.70 (s, 24H), 6.45 (t, *J* = 2.0 Hz, 4H), 6.82 (d, *J* = 2.4 Hz, 8H), 7.92 (d, *J* = 8.8 Hz, 4H), 8.78 (d, *J* = 8.4 Hz, 4H), 9.02 (s, 4H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>, δ): 55.3 (CH<sub>3</sub>), 99.9 (CH), 105.2 (CH), 124.2 (CH), 126.0 (CH), 127.6 (CH), 128.5 (C), 129.5 (C), 130.0 (C), 139.3 (C), 143.3 (C), 161.3 (C). HRMS-ESI (*m/z*): [M+H]<sup>+</sup> calcd for C<sub>58</sub>H<sub>49</sub>O<sub>8</sub>, 873.3422; found, 873.3415. mp 147–149 °C.

**9,19-Bis(3,5-di-*tert*-butylphenyl)-5,15-diethyl-5,15-dihydrocarbazolo[3',4':5,6][1,4]oxazino[2,3-b]indolo[3,2-h]phenoxazine (3d).**

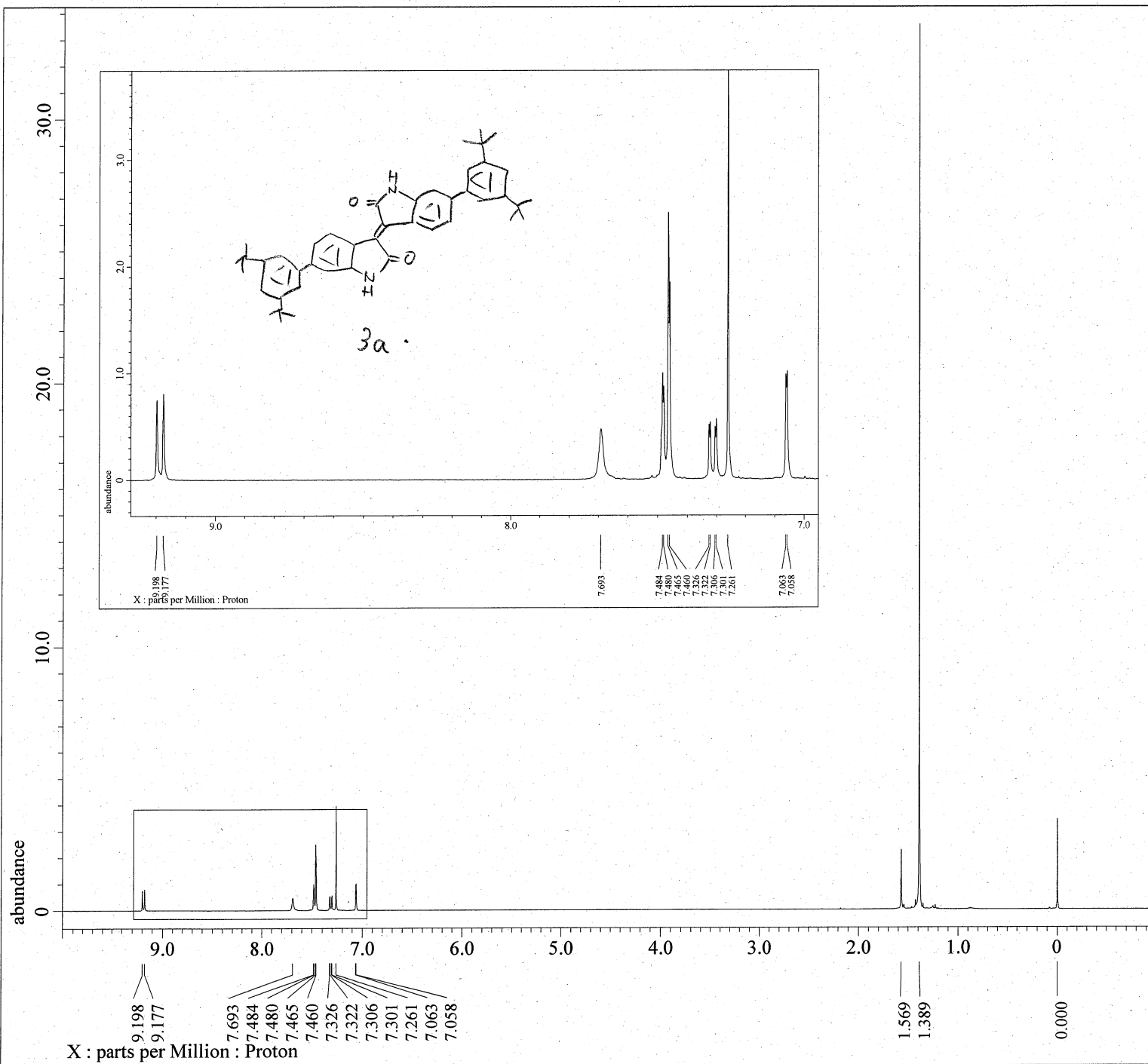


The reaction was carried out with 58.7 mg (0.10 mmol) of **1d** and 56.4 mg (0.24 mmol) of **2a**. The product **3d** was obtained as a purple powder (40.4 mg, 0.045 mmol, 45% yield) after purification by silica-gel column chromatography (SiO<sub>2</sub>, CH<sub>2</sub>Cl<sub>2</sub>/hexane, 0:100–100:0). The <sup>1</sup>H NMR spectrum of the product was in agreement with the literature.<sup>1</sup> The small amounts of impurities were detected by <sup>1</sup>H NMR.

<sup>1</sup>H NMR (401 MHz, CDCl<sub>3</sub>, δ): 1.42 (s, 42H), 4.28 (q, *J* = 7.1 Hz, 4H), 6.90 (t, *J* = 7.4 Hz, 2H), 6.95–7.05 (m, 4H), 7.28–7.31 (m, 2H), 7.31–7.38 (m, 3H), 7.38–7.42 (m, 3H), 7.42–7.50 (m, 2H), 7.62–7.69 (m, 2H).

## 7. Reference

1. Seo, T.; Toyoshima, N.; Kubota, K.; Ito, H. *J. Am. Chem. Soc.* **2021**, *143*, 6165.



```

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dc_balance( 0, FALSE )
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trapezoid( 0[%], 0[%], 80[%], 100[%] )
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fft( 1, TRUE, TRUE )
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Solvent       = CHLOROFORM-D
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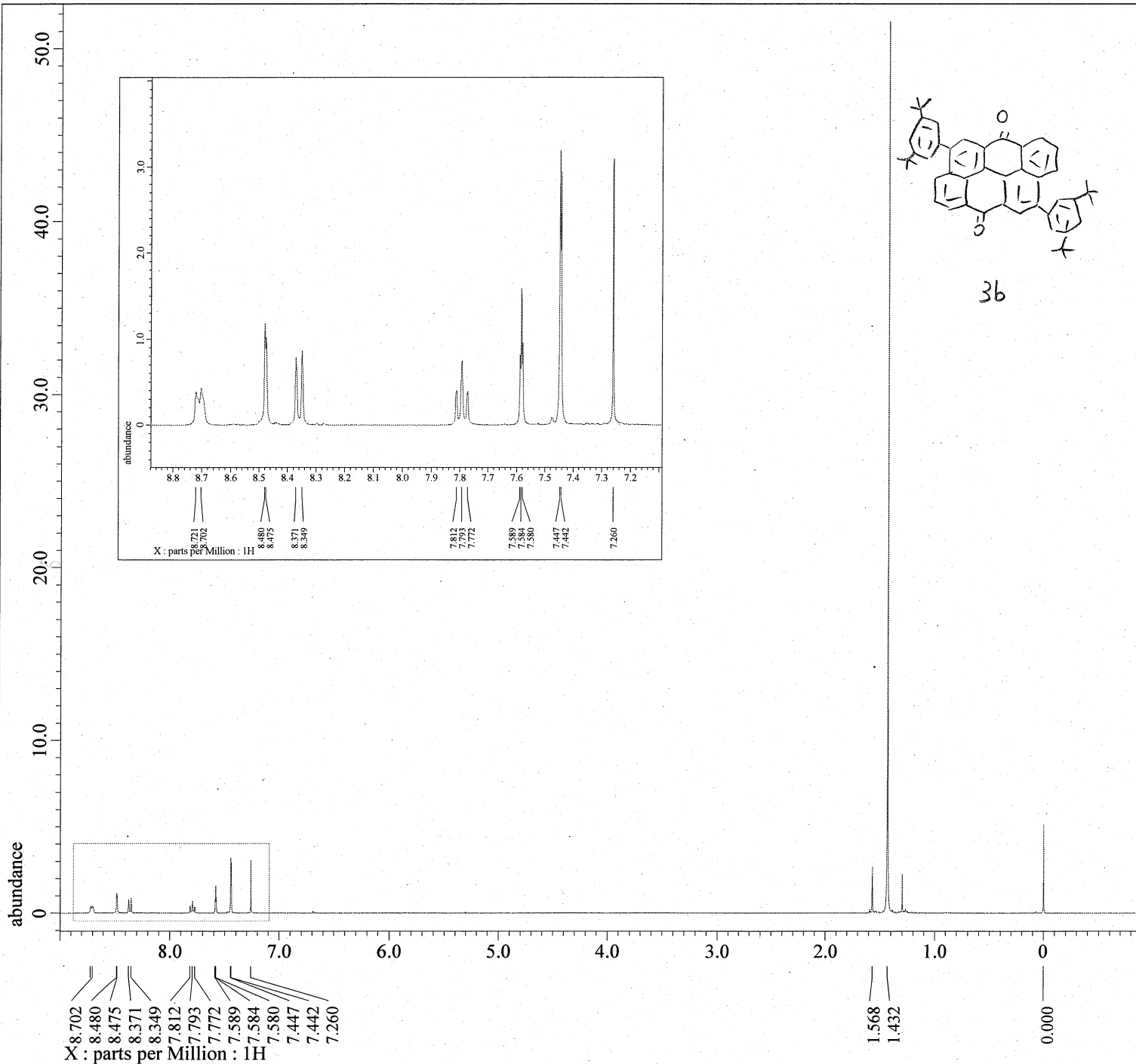
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machinephase
ppm

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Derived from: SEO-2905-1-pure-1.jdf

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Revision_Time  = 26-MAY-2022 18:31:20

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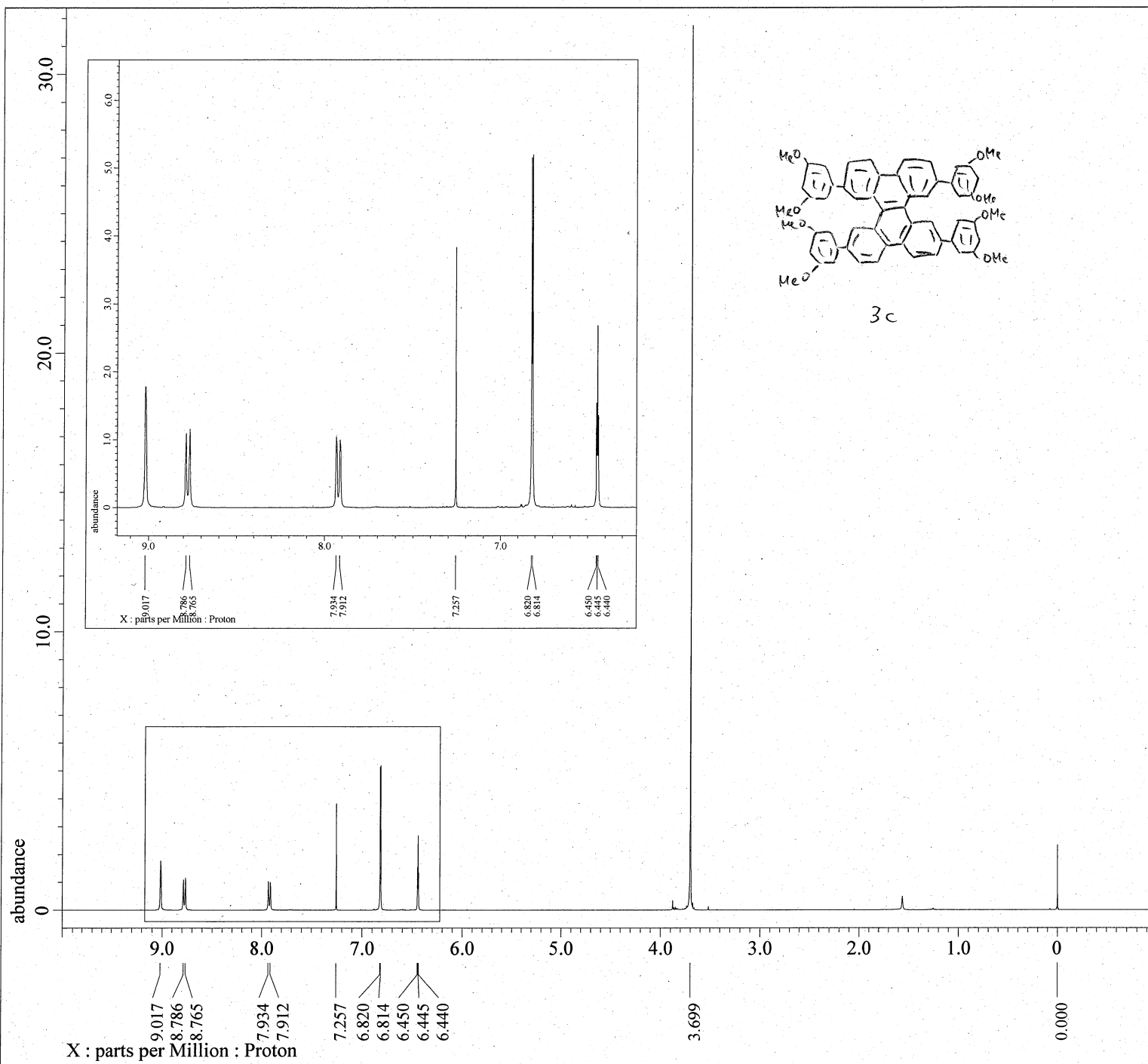
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Total_Scans = 8

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X Atn          = 1.9[dB]
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Dante_Presat   = FALSE
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Repetition_Time = 7.228224[s]

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fft( 1, TRUE, TRUE )
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ppm
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Dimensions    = X
Site          = JNM-ECS400
Spectrometer  = DELTA2_NMR

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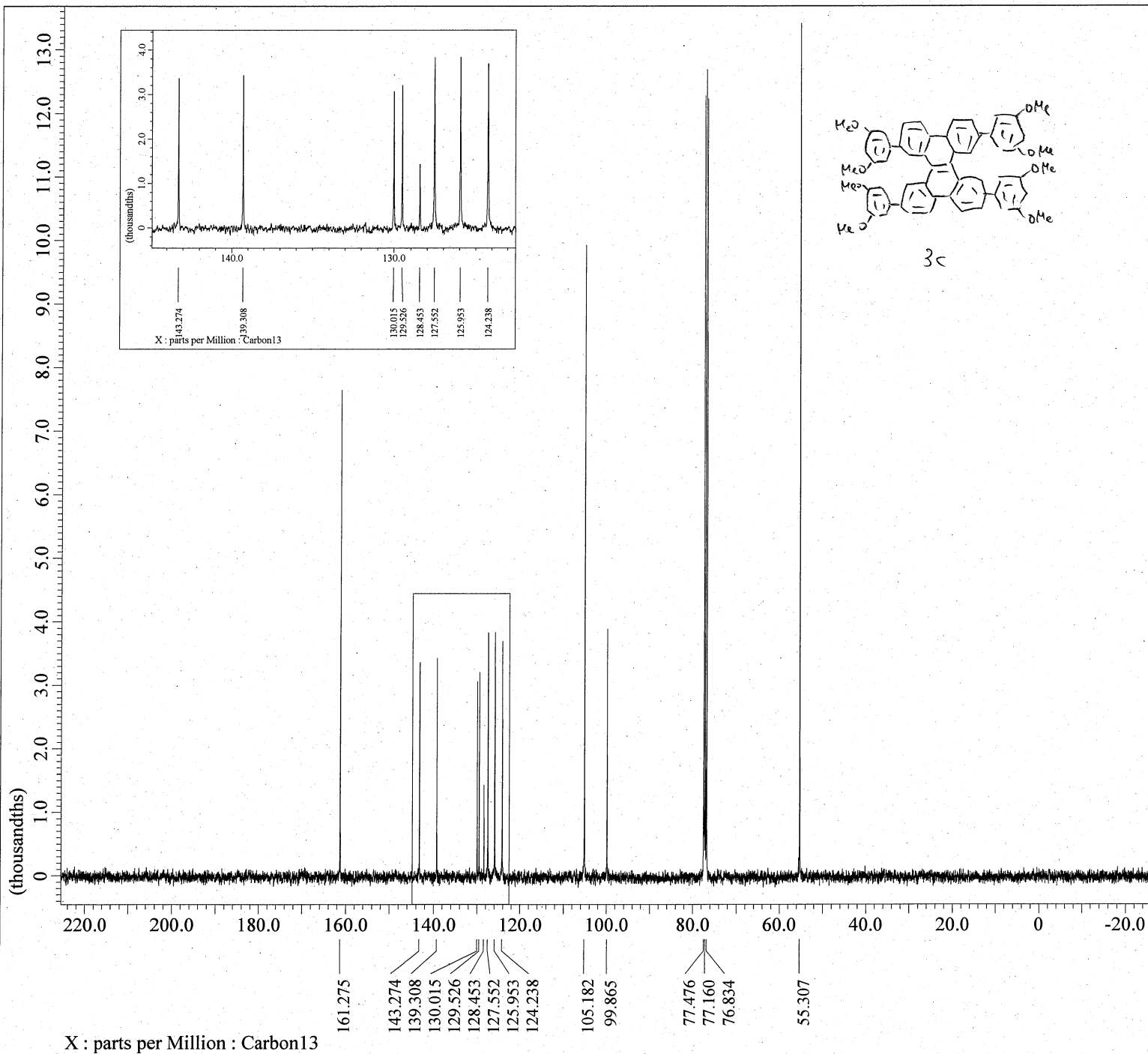
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fft( 1, TRUE, TRUE )
machinephase
ppm

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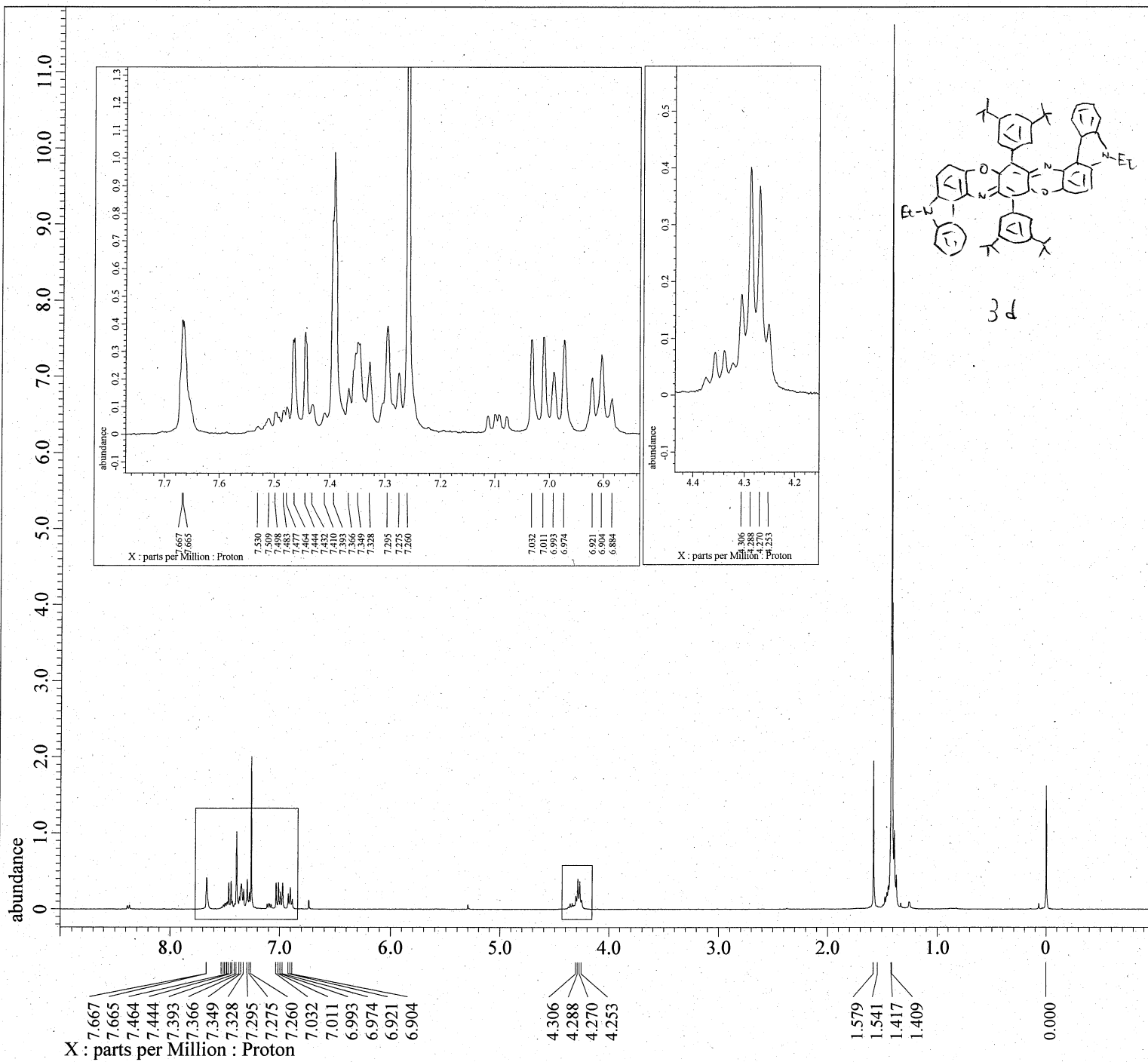
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Irr_Dec_Bandwidth_Hz = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm = 12.08082432[ppm]
Irr_Dec_Freq      = 395.88430144[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling    = TRUE
Irr_Noe           = TRUE
Irr_Noise         = WALTZ
Irr_Offset_Default = 5[ppm]

```





```

---- 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

以下に由来:: SEO-2794-1-pure_Proton-1-1.jdf

Filename      = SEO-2794-1-pure_Proton-1-
Author       = element
Experiment   = proton.jxp
Sample_Id    = SEO-2794-1-pure
Solvent      = CHLOROFORM-D
Actual_Start_Time = 5-APR-2022 10:18:59
Revision_Time  = 5-APR-2022 11:45:12

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 Clipped = 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       = 44
Temp_Get         = 18.9[dC]
X 90_Width      = 6[us]
X Acq Time       = 2.18103808[s]
X Angle         = 45[deg]
X Atn           = 0.8[dB]
X Pulse         = 3[us]
Irr Mode        = Off
Tri Mode        = Off
Dante_Presat    = FALSE
Initial_Wait    = 1[s]
Repetition_Time = 7.18103808[s]
  
```