

## Supporting Information

### Diaryliodonium Salt-Mediated Intramolecular C–N Bond Formation Using

### Boron-Masking *N*-Hydroxyamides

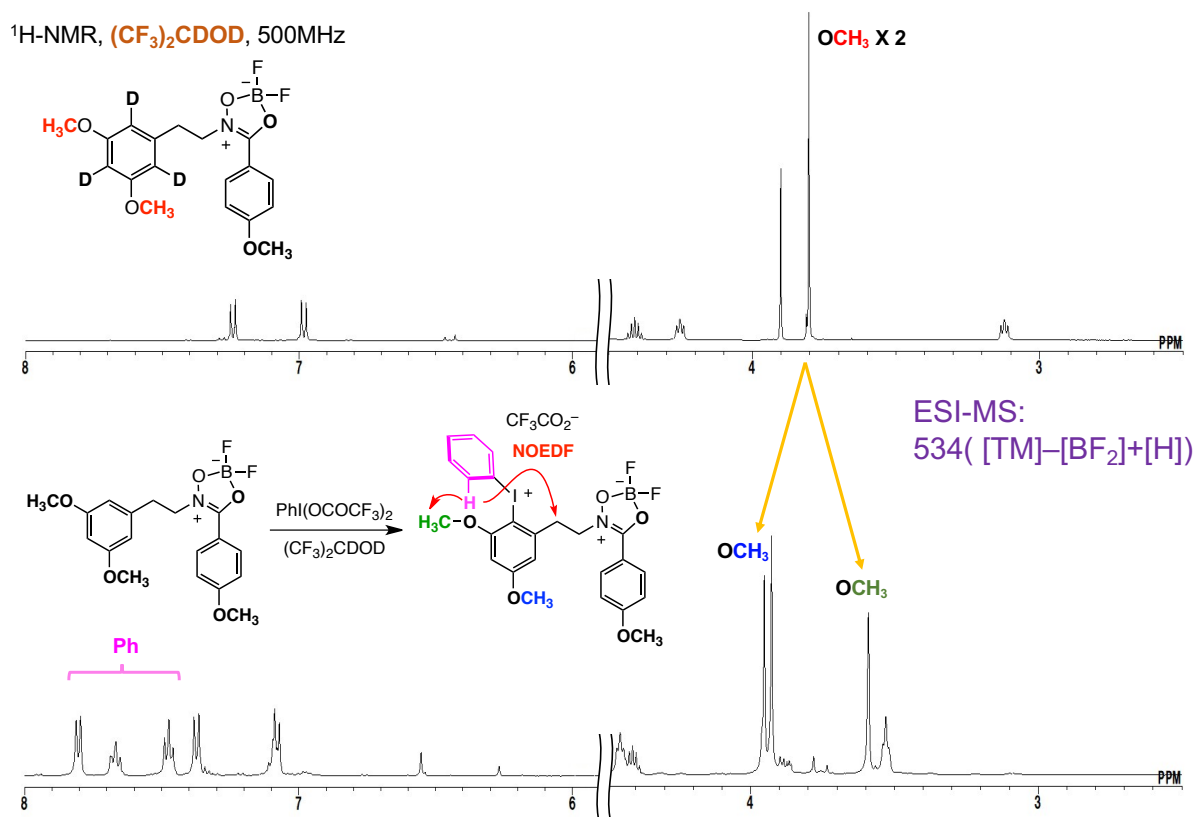
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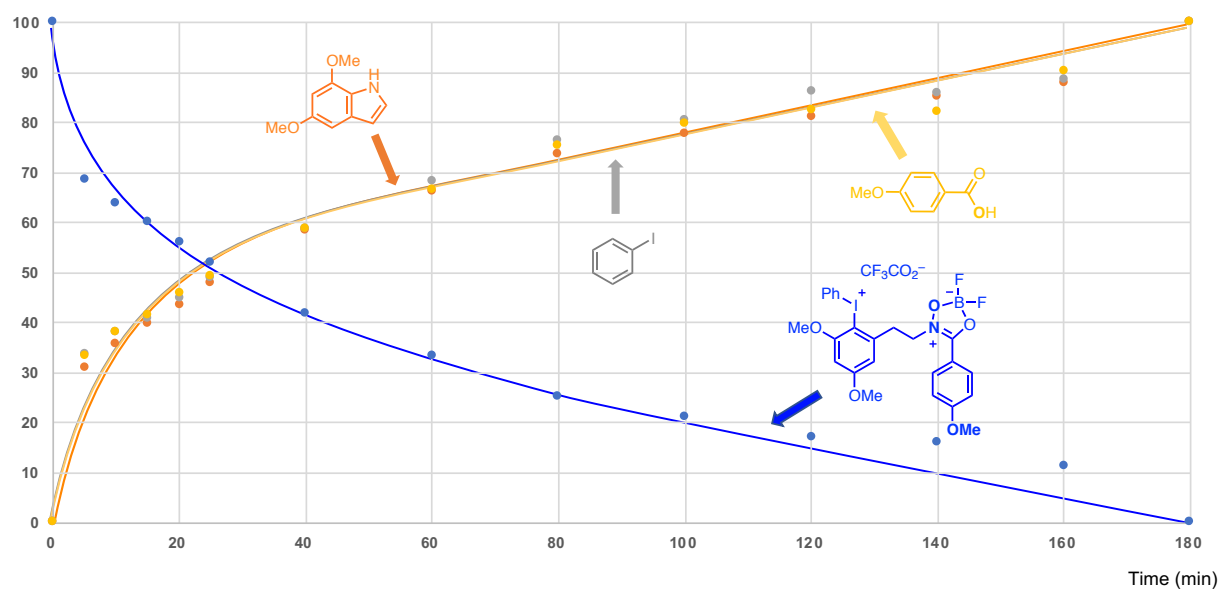
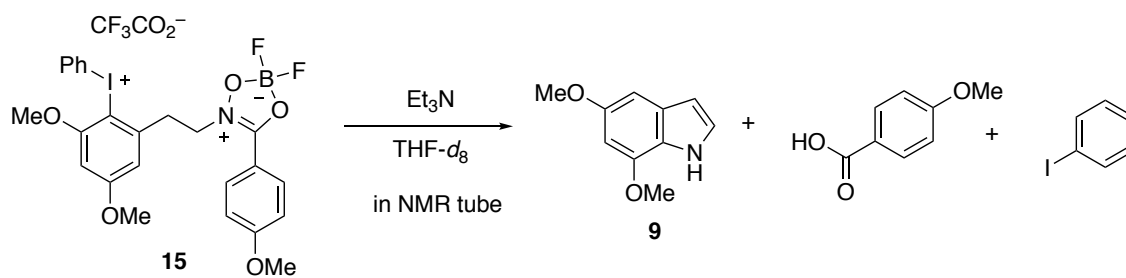
**General Remarks:** All reactions were monitored by thin-layer chromatography using Merck 60 F254 precoated silica gel plates (0.25 mm thickness). Specific optical rotations were measured using a JASCO P-1020 polarimeter. FT-IR spectra were recorded on a SHIMADZU IR Affinity-IS. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a JEOL ECX 500 FT-NMR spectrometer (500 MHz for <sup>1</sup>H NMR, 125 MHz for <sup>13</sup>C NMR) instrument. Data for <sup>1</sup>H NMR are reported as chemical shift ( $\delta$  ppm), multiplicity (s = singlet, d = doublet, t = triplet, dd = doubledoublet, tt = tripletriplet, ddd = doubledoubledoublet, dt = doubletriplet, q = quartet, quint = quintet, sext = sextet, m = multiplet, br = broad), coupling constant (Hz), integration, and assignment. Data for <sup>13</sup>C NMR are reported as chemical shift. The high-resolution mass spectra were recorded on a BRUKER impact II. Preparative thin layer chromatography was performed using Merck 60 F254 precoated silica gel plates (0.25 mm thickness). Flash chromatography was performed using silica gel 60N of Kanto Chemical Co. Int., Tokyo, Japan. Oil baths were used for reactions performed at temperatures in excess of room temperature.

Step 1 (PIFA treatment) was carried out in  $(\text{CF}_3)_2\text{CDOD}$ .



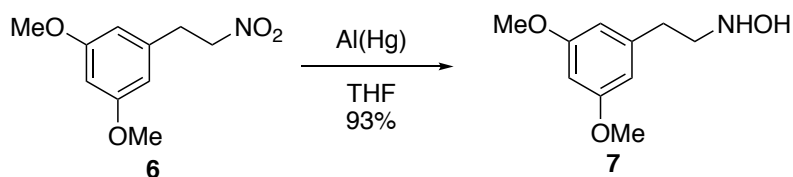
**Figure S1.**  $^1\text{H-NMR}$  of **12** and iodonium salt **15** in  $(\text{CF}_3)_2\text{CDOD}$ .

The structure elucidation of **15** by 2D-NMR and NOE was carried out before deuteration on aromatic protons (see page S 91–S99).



**Figure S2.** Kinetic study (NMR monitoring from intermediate **15** to indole **9**).

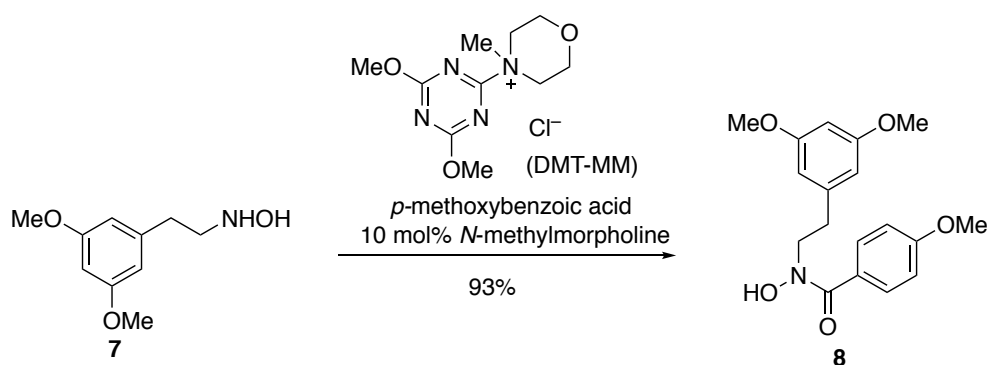
### Synthesis of hydroxylamine 7.



To a solution of known nitro compound **6** (1.42 g, 6.72 mmol)<sup>S1</sup> in THF (269 mL) and H<sub>2</sub>O (26.9 mL), freshly prepared Al(Hg) (from 2.8 g of Al) was added at room temperature under Ar atmosphere. The reaction mixture was vigorously stirred for 2 h at room temperature before being quenched with saturated aqueous NaHCO<sub>3</sub>. The resulting mixture was filtered by Celite pad. The filtrate was extracted three times with 10% MeOH/CHCl<sub>3</sub>. The combined organic layer was washed with brine, dried over MgSO<sub>4</sub> and concentrated under reduced pressure. Flash chromatography (50% EtOAc/*n*-hexans) provided hydroxylamine **7** (1.22 g, 93%) as a pale yellow solid.

N-(3,5-Dimethoxyphenethyl)hydroxylamine (7); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 6.38 (d, *J* = 1.5 Hz, 2H), 6.33 (t, *J* = 2.0 Hz, 1H), 3.78 (s, 6H), 3.17 (t, *J* = 7.3 Hz, 2H), 2.82 (t, *J* = 7.0 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 161.0, 141.7, 106.9, 98.4, 55.4, 54.8, 33.7; IR (neat)  $\nu_{\max}$  3289, 2909, 2835, 1593, 1460, 1425, 1341, 1202, 1144, 1053, 980, 918, 820 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>10</sub>H<sub>16</sub>NO<sub>3</sub> [M+H]<sup>+</sup>: 198.1125, found: 198.1123; mp: 43–45 °C.

### Synthesis of *N*-hydroxyamide **8**.

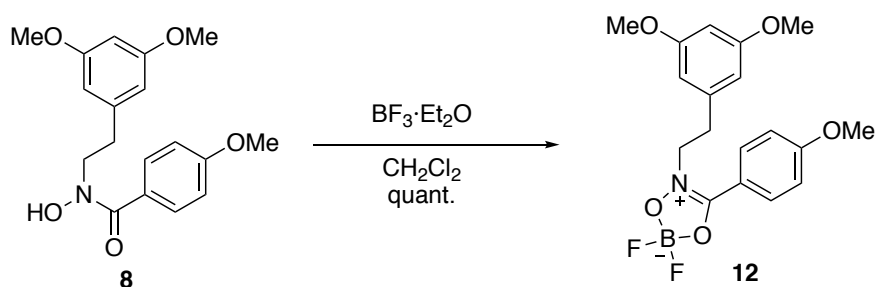


To a solution of *p*-methoxybenzoic acid (771 mg, 5.07 mmol) and *N*-methylmorpholine (NMM, 56  $\mu$ L, 0.507 mmol) in CH<sub>3</sub>CN (15 mL), 4-(4,6-dimethoxy-1,3,5-triazin-2-yl)-4-methylmorpholinium chloride (DMT-MM, 1.68 g, 6.08 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 3 h at room temperature. The resulting mixture was cooled to -20 °C before an addition of the solution of hydroxylamine **7** (1.0 g, 5.07 mmol) in CH<sub>3</sub>CN (10 mL). The

reaction mixture was stirred for 3 h at  $-20\text{ }^{\circ}\text{C}$  under Ar atmosphere. The resulting mixture was quenched with excess amount of 1M HCl/H<sub>2</sub>O at  $-20\text{ }^{\circ}\text{C}$ . The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 2% CH<sub>3</sub>CN/CHCl<sub>3</sub>) provided *N*-hydroxyamide **8** (1.57 g, 93%) as a white solid.

*N*-(3,5-Dimethoxyphenethyl)-*N*-hydroxy-4-methoxybenzamide (**8**): <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.60 (br. s, 1H), 7.10 (d,  $J = 8.5$  Hz, 2H), 6.79 (d,  $J = 8.5$  Hz, 2H), 6.33 (t,  $J = 2.3$  Hz, 1H), 6.22 (d,  $J = 2.5$  Hz, 2H), 3.88 (t,  $J = 6.8$  Hz, 2H), 3.81 (s, 3H), 3.73 (s, 6H), 2.94 (t,  $J = 6.8$  Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  167.9, 161.6, 161.0, 140.2, 129.6, 124.2, 113.7, 107.1, 99.1, 55.5, 55.4, 52.6, 33.8; IR (neat)  $\nu_{\text{max}}$  2841, 1591, 1499, 1431, 1429, 1356, 1308, 1267, 1256, 1204, 1150, 1069, 1024, 974, 918, 868, 837, 797, 756, 725 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>18</sub>H<sub>22</sub>NO<sub>5</sub> [M+H]<sup>+</sup>: 332.1492, found: 332.1484.; mp: 95–96 °C.

#### Synthesis of boron complex **12**.

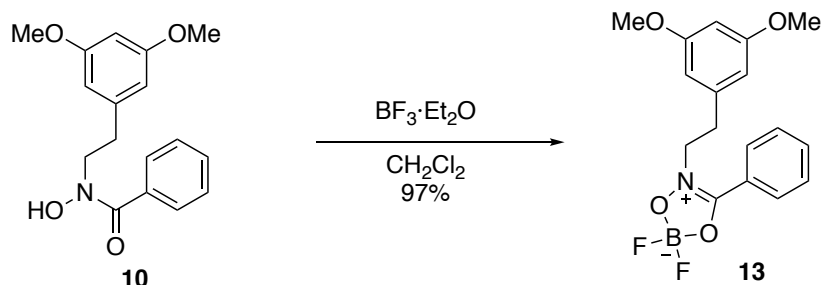


To a solution of *N*-hydroxyamide **8** (1.5 g, 4.53 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (20 mL), BF<sub>3</sub>·Et<sub>2</sub>O (1.1 mL, 9.06 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 1 h at room temperature. Excess BF<sub>3</sub>·Et<sub>2</sub>O and the solvent were removed from the reaction vessel under reduced pressure. Obtained white solids were collected by filtration and washed with cold Et<sub>2</sub>O. Desired pure boron complex **12** (1.72 g) was obtained in quantitative yield as colorless crystals. Recrystallization to measure X-ray diffraction was performed with hot Et<sub>2</sub>O. The relevant crystallographic data for the **12** have been deposited at Cambridge Crystallographic Data Centre (CCDC) with CCDC No. 1963790.

**Boron complex 12**: <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.11 (d,  $J = 9.0$  Hz, 2H), 6.84 (d,  $J = 8.5$  Hz, 2H), 6.28 (s, 3H), 4.13 (t,  $J = 6.3$  Hz, 2H), 3.82 (s, 3H), 3.70 (s, 6H), 3.08 (t,  $J = 6.0$  Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  163.5, 162.8, 161.3, 138.4, 130.4, 114.5, 113.9, 106.6, 100.2, 55.7, 55.4, 52.4, 32.9; IR (neat)  $\nu_{\text{max}}$  2968, 2839, 1595, 1528, 1456, 1360, 1308, 1261, 1165, 1152, 1124, 1096, 1038, 1018, 988, 934, 880, 841, 826, 762, 689 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>18</sub>H<sub>22</sub>NO<sub>5</sub> [M-BF<sub>2</sub>+H<sub>2</sub>]<sup>+</sup>: 332.1492,

found: 332.1484; mp: 125–127 °C.

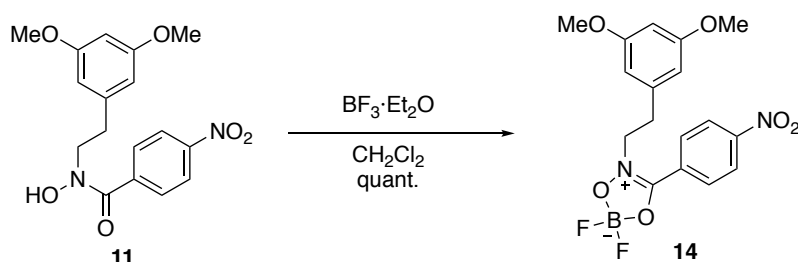
#### Synthesis of boron complex **13**.



To a solution of *N*-hydroxyamide **10** (794.0 mg, 2.64 mmol) in  $\text{CH}_2\text{Cl}_2$  (8.8 mL),  $\text{BF}_3 \cdot \text{Et}_2\text{O}$  (651.6  $\mu\text{L}$ , 15.28 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 1 h at room temperature. Excess  $\text{BF}_3 \cdot \text{Et}_2\text{O}$  and the solvent were removed from the reaction vessel under reduced pressure to provide boron complex **13** (891.8 mg, 97%). Obtained materials was enough pure, then it was employed to the next reaction without further purification.

**Boron complex **13****;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.53 (t,  $J = 7.5$  Hz, 1H), 7.36 (t,  $J = 8.0$  Hz, 2H), 7.10 (d,  $J = 8.0$  Hz, 2H), 6.31 (t,  $J = 2.3$  Hz, 1H), 6.27 (d,  $J = 2.0$  Hz, 2H), 4.13 (t,  $J = 6.0$  Hz, 2H), 3.71 (s, 6H), 3.08 (t,  $J = 6.0$  Hz, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  163.1, 161.4, 138.3, 133.3, 129.1, 128.3, 122.2, 106.6, 100.3, 55.5, 52.4, 32.9; IR (neat)  $\nu_{\text{max}}$  3022, 2940, 2851, 1595, 1470, 1462, 1431, 1356, 1298, 1261, 1204, 1150, 1067, 1040, 988, 939, 826, 785, 692  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{17}\text{H}_{20}\text{NO}_4$   $[\text{M}-\text{BF}_2+\text{H}_2]^+$ : 302.1387, found: 302.1367; mp: 92–94 °C.

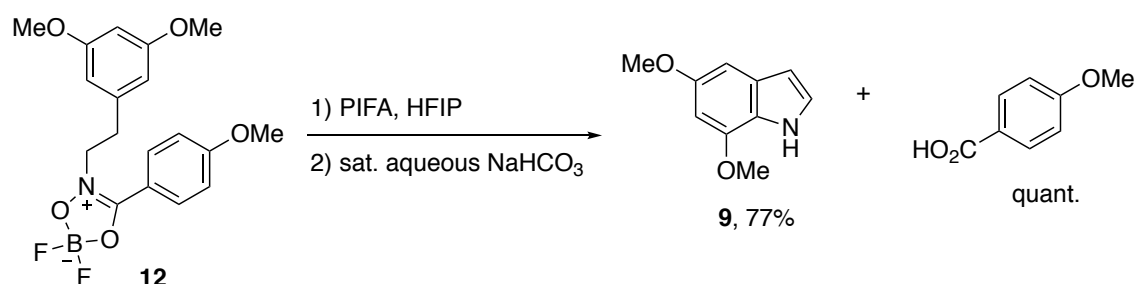
#### Synthesis of boron complex **14**.



To a solution of *N*-hydroxyamide **11** (46.9 mg, 0.136 mmol) in  $\text{CH}_2\text{Cl}_2$  (4.5 mL),  $\text{BF}_3 \cdot \text{Et}_2\text{O}$  (33.4  $\mu\text{L}$ , 0.271 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 1 h at room temperature. Excess  $\text{BF}_3 \cdot \text{Et}_2\text{O}$  and the solvent were removed from the reaction vessel under reduced pressure to provide boron complex **14** (53.9 mg, quant.). Obtained materials was enough pure, then it was employed to the next reaction without further purification.

**Boron complex 14**;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.19 (d,  $J = 8.5$  Hz, 2H), 7.22 (d,  $J = 8.5$  Hz, 2H), 6.33 (t,  $J = 2.3$  Hz, 1H), 6.22 (d,  $J = 2.0$  Hz, 2H), 4.12 (t,  $J = 5.5$  Hz, 2H), 3.72 (s, 6H), 3.10 (t,  $J = 5.8$  Hz, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  161.6, 161.3, 150.3, 138.0, 129.7, 127.9, 124.1, 106.7, 100.2, 55.5, 52.9, 32.9; IR (neat)  $\nu_{\text{max}}$  2361, 1589, 1522, 1456, 1433, 1356, 1314, 1209, 1163, 1123, 1067, 1034, 995, 937, 856, 835, 754, 704  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{17}\text{H}_{19}\text{N}_2\text{O}_6$   $[\text{M}-\text{BF}_2+\text{H}_2]^+$ : 347.1238, found: 347.1233; mp: 176–179  $^\circ\text{C}$ .

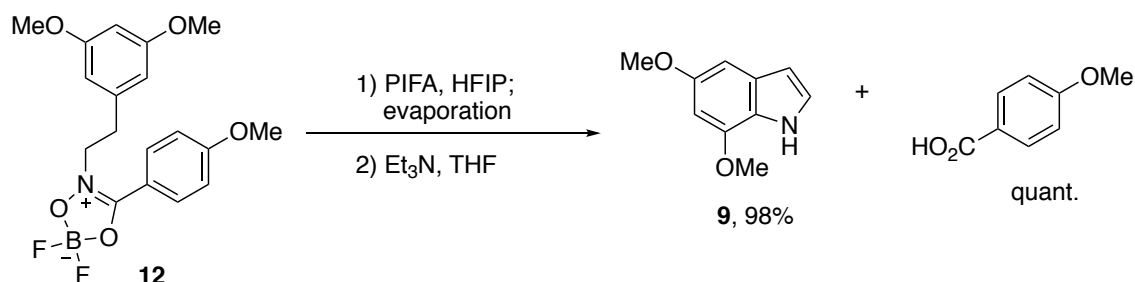
**General protocol of our discovered indole synthesis; Method A (Table 1, entry 1)**



To a solution of boron complex **12** (20.0 mg, 0.0528 mmol) in 1,1,1,3,3,3-hexafluoroisopropanol (HFIP, 3.6 mL), [bis(trifluoroacetoxy)iodo]benzene (PIFA, 22.6 mg, 0.0528 mmol) was added at 0  $^\circ\text{C}$  under Ar atmosphere. The reaction mixture was stirred for 5 min (Time A) before an addition of saturated aqueous  $\text{NaHCO}_3$  (5 mL). The resulting mixture was stirred for 15 min (Time B) at 0  $^\circ\text{C}$ . The aqueous layer was extracted three times with  $\text{CH}_2\text{Cl}_2$ . The combined organic layer was washed with saturated aqueous  $\text{NaCl}$ , dried over  $\text{MgSO}_4$ , and concentrated under reduced pressure. PTLC ( $\text{SiO}_2$ , 5%  $\text{MeOH}/\text{CHCl}_3$ ) provided 5,7-dimethoxyindole (**9**, 7.2 mg, 77%) as a pale yellow oil. The aqueous layer was acidified with aqueous 1M  $\text{HCl}$ , then extracted three times with  $\text{CH}_2\text{Cl}_2$ . The combined organic layer was washed with saturated aqueous  $\text{NaCl}$ , dried over  $\text{MgSO}_4$ , and concentrated under reduced pressure to provide *p*-methoxybenzoic acid (8.2 mg, quant.).

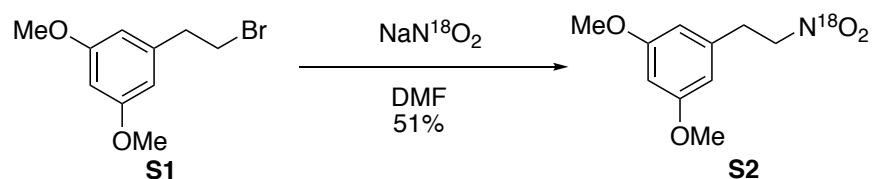
3,5-Dimethoxyindole (**9**) (identified with reported data<sup>S2</sup>);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.33 (br s, 1H), 7.14 (t,  $J = 2.8$  Hz, 1H), 6.73 (d,  $J = 2.0$  Hz, 1H), 6.48 (t,  $J = 2.5$  Hz, 1H), 6.39 (d,  $J = 1.5$  Hz, 1H), 3.94 (s, 1H), 3.88 (s, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  155.1, 146.6, 128.6, 124.1, 121.9, 102.8, 94.2, 93.8, 55.9, 55.5.

### Method B (Table 1, entry 2)



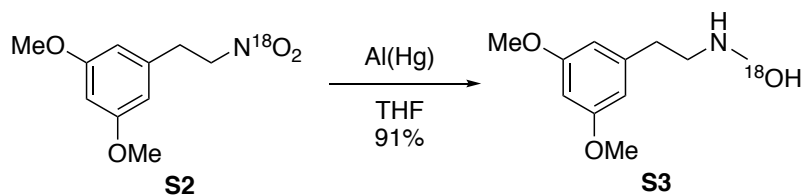
To a solution of boron complex **12** (20.0 mg, 0.0528 mmol) in 1,1,1,3,3,3-hexafluoroisopropanol (HFIP, 3.6 mL), [bis(trifluoroacetoxy)iodo]benzene (PIFA, 22.6 mg, 0.0528 mmol) was added at 0 °C under Ar atmosphere. The reaction mixture was stirred for 5 min (Time A) before removal of solvent. The residue was dissolved to THF (3.6 mL) and the resulting mixture was cooled at 0 °C. Et<sub>3</sub>N (73.2 μL, 0.528 mmol) was added to the resulting mixture and the reaction mixture was stirred for 2 h (Time B) at 0 °C under Ar atmosphere. The resulting mixture was quenched with saturated aqueous NaHCO<sub>3</sub>. The aqueous layer was extracted three times with CH<sub>2</sub>Cl<sub>2</sub>. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 10% *n*-hexanes/CHCl<sub>3</sub>) provided 5,7-dimethoxyindole (**9**, 9.2 mg, 98%) as a pale yellow oil. The aqueous layer was acidified with aqueous 1M HCl, then extracted three times with CH<sub>2</sub>Cl<sub>2</sub>. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure to provide *p*-methoxybenzoic acid (8.2 mg, quant.).

### Synthesis of <sup>18</sup>O-enrich nitro compound **S2**.

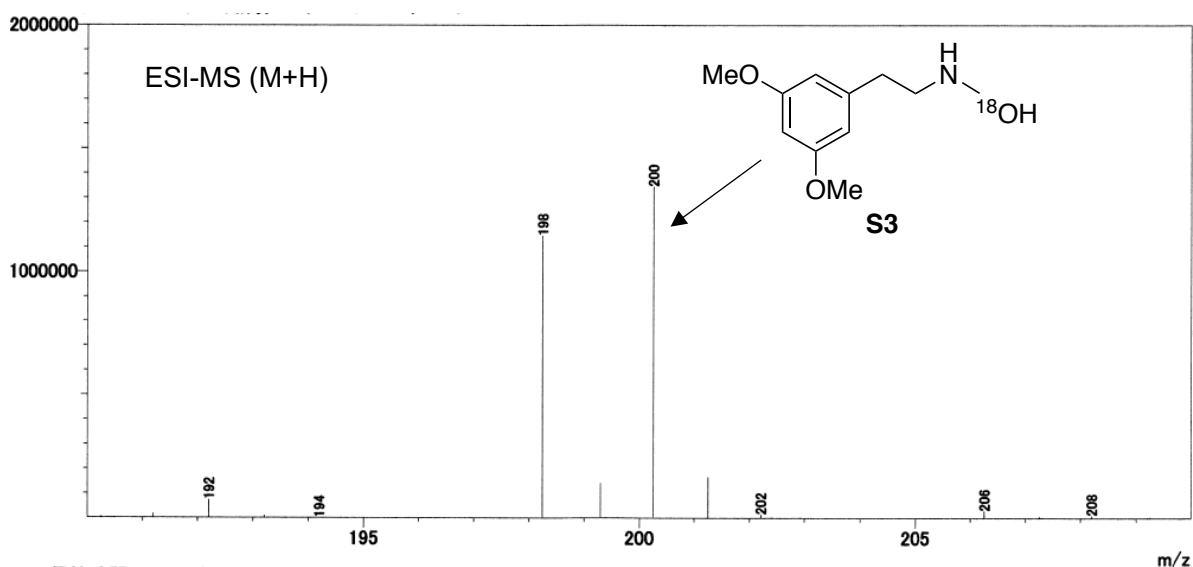


To a solution of known compound **S1** (739.0 mg, 3.02 mmol)<sup>S3)</sup> in DMF (10 mL), freshly prepared <sup>18</sup>O-enrich NaN<sup>18</sup>O<sub>2</sub> (208.0 mg, 3.02 mmol)<sup>S4)</sup> in DMF (8 mL) was slowly added at -78 °C under Ar atmosphere. The reaction mixture was slowly warmed up to room temperature, and stirred for 14 h at room temperature before an addition of H<sub>2</sub>O (50 mL). The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 10% EtOAc/*n*-hexanes) provided <sup>18</sup>O-enrich nitro compound **S2** (325.3 mg, 51%) as a pale yellow oil.

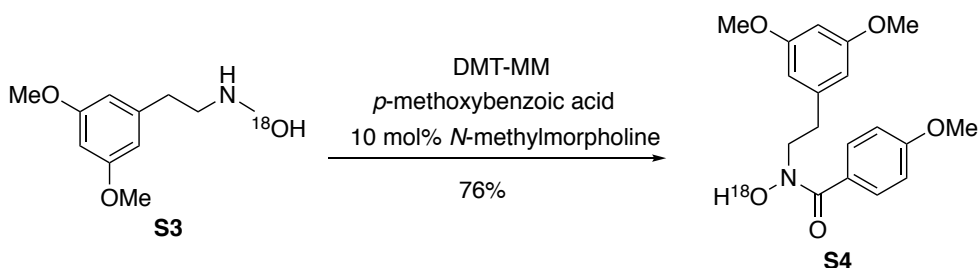
### Synthesis of $^{18}\text{O}$ -enrich hydroxylamine **S3**.



To a solution of nitro compound **S2** (150.0 mg, 0.71 mmol) in THF (28 mL) and  $\text{H}_2\text{O}$  (2.8 mL), freshly prepared  $\text{Al(Hg)}$  (from 300.0 mg of Al) was added at room temperature under Ar atmosphere. The reaction mixture was vigorously stirred for 8 h at room temperature before being quenched with saturated aqueous  $\text{NaHCO}_3$ . The resulting mixture was filtrated by Celite pad. The filtrate was extracted three times with 10% MeOH/ $\text{CHCl}_3$ . The combined organic layer was washed with brine, dried over  $\text{MgSO}_4$  and concentrated. Flash chromatography (50% EtOAc/*n*-hexans) provided  $^{18}\text{O}$ -enrich hydroxylamine **S3** (128.8 mg, 91%) as colorless oil.

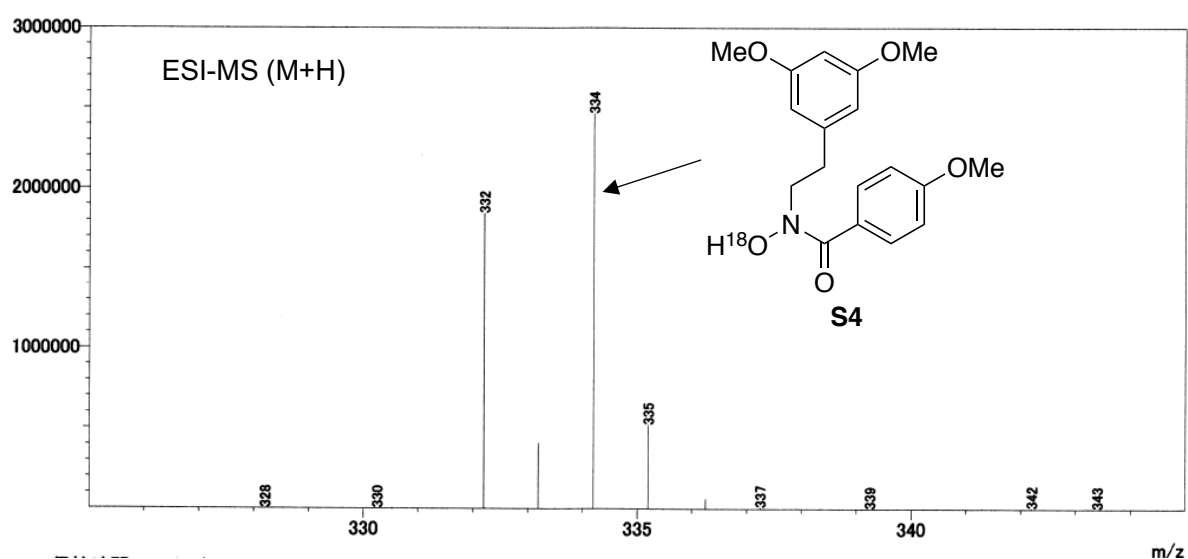


### Synthesis of $^{18}\text{O}$ -enrich *N*-hydroxyamide **S4**.

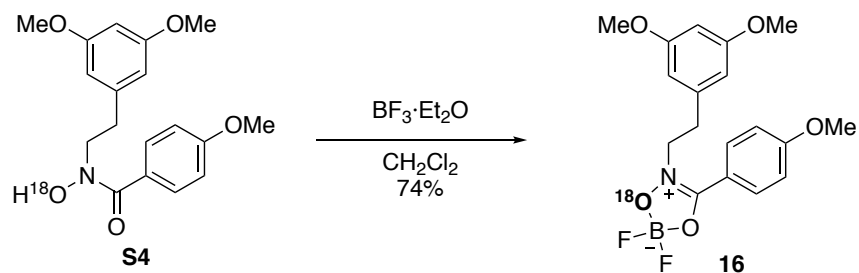


To a solution of *p*-methoxybenzoic acid (97.9 mg, 0.644 mmol) and *N*-methylmorpholine (NMM, 7.1

$\mu\text{L}$ , 0.0644 mmol) in  $\text{CH}_3\text{CN}$  (5 mL), 4-(4,6-dimethoxy-1,3,5-triazin-2-yl)-4-methylmorpholium chloride (DMT-MM, 213.8 mg, 0.773 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 3 h at room temperature. The resulting mixture was cooled to  $-20\text{ }^\circ\text{C}$  before an addition of the solution of hydroxylamine **S3** (127.0 mg, 0.644 mmol) in  $\text{CH}_3\text{CN}$  (3 mL). The reaction mixture was stirred for 1 h at  $-20\text{ }^\circ\text{C}$  under Ar atmosphere. The resulting mixture was quenched with excess amount of aqueous 1M HCl at  $-20\text{ }^\circ\text{C}$ . The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over  $\text{MgSO}_4$ , and concentrated under reduced pressure. Flash chromatography ( $\text{SiO}_2$ , 2%  $\text{CH}_3\text{CN}/\text{CHCl}_3$ ) provided *N*-hydroxyamide **S4** (163.0 mg, 76%) as a pale yellow solid.

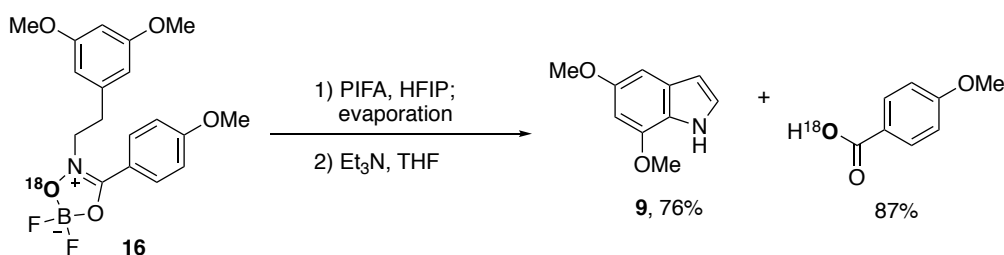


#### Synthesis $^{18}\text{O}$ -enrich boron complex **16**.

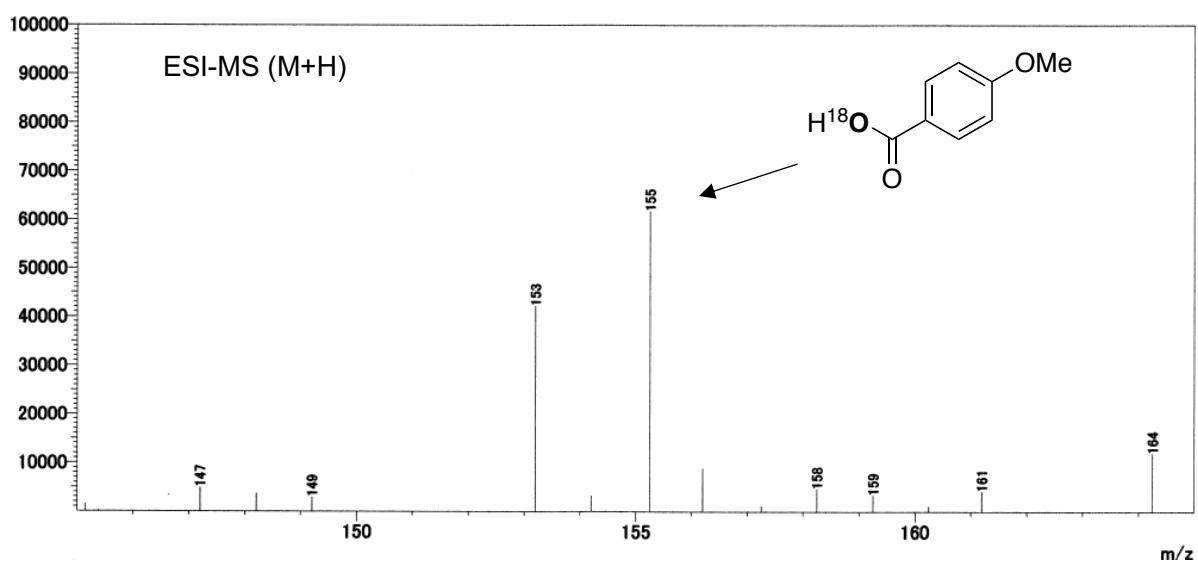


To a solution of *N*-hydroxyamide **S4** (160.0 mg, 0.483 mmol) in  $\text{CH}_2\text{Cl}_2$  (10 mL),  $\text{BF}_3 \cdot \text{Et}_2\text{O}$  (120.0  $\mu\text{L}$ , 0.966 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 1 h at room temperature. Excess  $\text{BF}_3 \cdot \text{Et}_2\text{O}$  and the solvent were removed from the reaction vessel under reduced pressure. Obtained white solids were collected by filtration and washed with cold  $\text{Et}_2\text{O}$ . Desired pure boron complex **16** (136.1 mg, 74%) was obtained as colorless crystals.

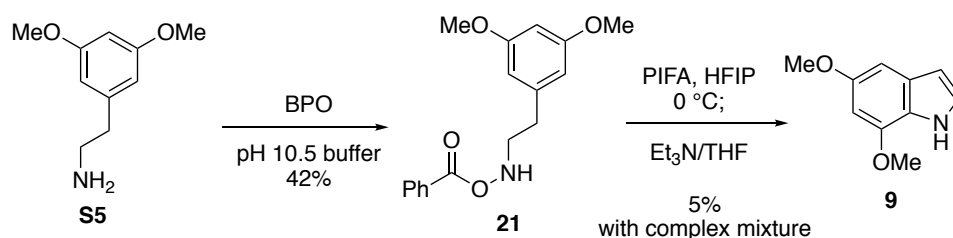
The key indole synthesis using  $^{18}\text{O}$ -enrich boron complex **16** to confirm origin of oxygen in recovered *p*-methoxybenzoic acid.



To a solution of boron complex **16** (20.0 mg, 0.0525 mmol) in 1,1,1,3,3,3-hexafluoroisopropanol (HFIP, 3.5 mL), [bis(trifluoroacetoxy)iodo]benzene (PIFA, 22.6 mg, 0.0525 mmol) was added at 0 °C under Ar atmosphere. The reaction mixture was stirred for 5 min before removal of solvent. The residue was dissolved to THF (3.5 mL) and the resulting mixture was cooled at 0 °C.  $\text{Et}_3\text{N}$  (73.1  $\mu\text{L}$ , 0.525 mmol) was added to the resulting mixture and the reaction mixture was stirred for 2 h at 0 °C under Ar atmosphere. The resulting mixture was quenched with saturated aqueous  $\text{NaHCO}_3$ . The aqueous layer was extracted three times with  $\text{CH}_2\text{Cl}_2$ . The combined organic layer was washed with saturated aqueous  $\text{NaCl}$ , dried over  $\text{MgSO}_4$ , and concentrated under reduced pressure. Flash chromatography ( $\text{SiO}_2$ , 10% *n*-hexanes/ $\text{CHCl}_3$ ) provided 5,7-dimethoxyindole (**9**, 7.1 mg, 76%) as a pale yellow oil. The aqueous layer was acidified with aqueous 1M  $\text{HCl}$ , then extracted three times with  $\text{CH}_2\text{Cl}_2$ . The combined organic layer was washed with saturated aqueous  $\text{NaCl}$ , dried over  $\text{MgSO}_4$ , and concentrated under reduced pressure to provide  $^{18}\text{O}$ -enrich *p*-methoxybenzoic acid (7.0 mg, 87%).



Synthesis of *O*-benzoylhydroxylamine **21** followed by PIFA oxidation.



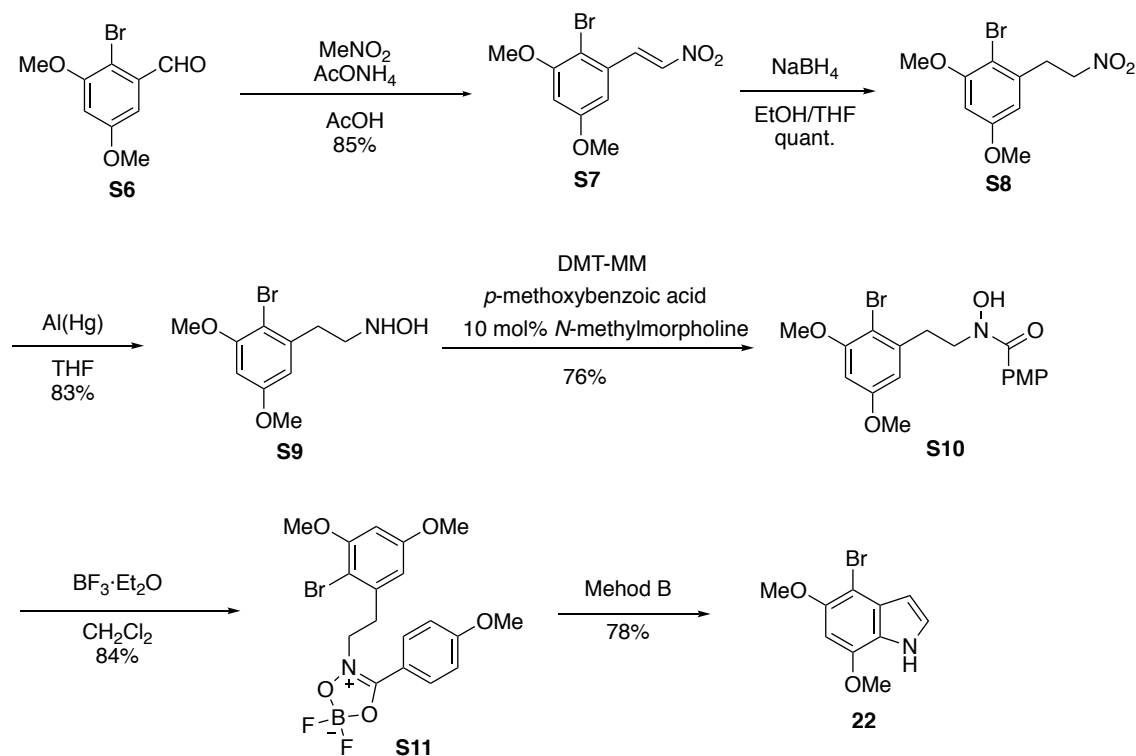
A solution of benzoyl peroxide (BPO, 267.3 mg, 1.10 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (5.5 mL) was added quickly to a mixture of 3,5-dimethoxyphenethylamine (200.0 mg, 1.10 mmol) and a pH 10.5 buffer solution (5.5 mL) at room temperature. TLC was used to monitor the consumption of starting material. After the reaction was complete, the aqueous layer was extracted twice with CH<sub>2</sub>Cl<sub>2</sub>. The combined organic layer was washed with brine, dried over MgSO<sub>4</sub> and concentrated under reduced pressure. Flash chromatography (CHCl<sub>3</sub>) provided *O*-benzoylhydroxylamine **21** (140.0 mg, 42%) as a pale yellow oil.

*O*-Benzoyl-*N*-(3,5-dimethoxyphenethyl)hydroxylamine (**21**); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.00 (d, *J* = 7.0 Hz, 2H), 7.81 (br s, 1H), 7.57 (t, *J* = 7.5 Hz, 1H), 7.46 (t, *J* = 7.8 Hz, 2H), 6.39 (d, *J* = 2.5 Hz, 2H), 6.33 (t, *J* = 2.0 Hz, 1H), 3.77 (s, 6H), 3.42 (t, *J* = 6.8 Hz, 2H), 2.90 (t, *J* = 7.0 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 166.8, 161.1, 141.1, 133.5, 129.5, 128.7, 107.0, 106.9, 98.6, 55.4, 53.5, 34.2; IR (neat) ν<sub>max</sub> 1717, 1593, 1452, 1429, 1346, 1285, 1267, 1204, 1148, 1065, 1024, 924, 829, 754, 708 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>17</sub>H<sub>20</sub>NO<sub>4</sub> [M+H]<sup>+</sup>: 302.1387, found: 302.1383.

To a solution of *O*-benzoylhydroxylamine **21** (40.0 mg, 0.133 mmol) in 1,1,1,3,3,3-hexafluoroisopropanol (HFIP, 9.0 mL), [bis(trifluoroacetoxy)iodo]benzene (PIFA, 57.1 mg, 0.133 mmol) was added at 0 °C under Ar atmosphere. The reaction mixture was stirred for 5 min before removal of solvent. The residue was dissolved to THF (9.0 mL) and the resulting mixture was cooled at 0 °C. Et<sub>3</sub>N (185.0 μL, 1.33 mmol) was added to the resulting mixture and the reaction mixture was stirred for 2 h at 0 °C under Ar atmosphere. The resulting mixture was quenched with saturated aqueous NaHCO<sub>3</sub>. The aqueous layer was extracted three times with CH<sub>2</sub>Cl<sub>2</sub>. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 5% EtOAc/*n*-hexanes) provided 5,7-dimethoxyindole (**9**, 1.2 mg, 5%) as a pale yellow oil.

## Substrate scope:

### Synthesis of 4-bromo-5,7-dimethoxy indole (22).



To a solution of 2-bromo-3,5-dimethoxybenzaldehyde (**S6**, 1.0 g, 4.31 mmol) and MS 3Å in acetic acid (3.5 mL), ammonium acetate (332.0 mg, 4.31 mmol) and nitromethane (11.6 mL, 21.6 mmol) were added at room temperature under Ar atmosphere. The reaction mixture was stirred for 2 h at 100 °C in before removal of MS 3Å by Celite filtration. The resulting mixture was quenched with saturated aqueous  $\text{NaHCO}_3$ . The aqueous layer was extracted three times with  $\text{CH}_2\text{Cl}_2$ . The combined organic layer was washed with saturated aqueous  $\text{NaCl}$ , dried over  $\text{MgSO}_4$ , and concentrated under reduced pressure. Recrystallization with  $\text{MeOH}$  provided pure nitrostyrene derivative **S7** (1.06 g, 85%) as yellow crystals.

(*E*)-2-Bromo-1,5-dimethoxy-3-(2-nitrovinyl)benzene (**S7**):  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.44 (d,  $J = 14.0$  Hz, 1H), 7.50 (d,  $J = 14.0$  Hz, 1H), 6.65 (d,  $J = 3.0$  Hz, 1H), 6.59 (d,  $J = 3.0$  Hz, 1H), 3.91 (s, 3H), 3.85 (s, 3H);  $^{13}\text{C NMR}$  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  160.0, 157.7, 139.3, 138.4, 131.8, 107.6, 103.9, 102.7, 56.7, 55.9; IR (neat)  $\nu_{\text{max}}$  3098, 1632, 1574, 1470, 1450, 1325, 1204, 1165, 1080, 970, 957, 829, 768, 658  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{10}\text{H}_{11}\text{BrNO}_4$   $[\text{M}+\text{H}]^+$ : 287.9866, found: 287.9849; mp: 176–178 °C.

To a solution of NaBH<sub>4</sub> (289.0 mg, 7.64 mmol) in EtOH (19 mL), a solution of **S7** (1.05g, 3.82 mmol) in THF (19 mL) was slowly added at 0 °C under Ar atmosphere. The reaction mixture was stirred for 1 h at 0 °C. The resulting mixture was quenched with excess amount of 1M HCl/H<sub>2</sub>O at 0 °C followed by an addition of EtOAc. The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 15% EtOAc/*n*-hexanes) provided nitroethane **S8** (1.13 g, quant.) as a white solid.

2-Bromo-1,5-dimethoxy-3-(2-nitroethyl)benzene (S8); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 6.42 (s, 2H), 4.64 (t, *J* = 7.0 Hz, 2H), 3.87 (s, 3H), 3.79 (s, 3H), 3.44 (t, *J* = 7.5 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 160.0, 157.2, 136.9, 107.4, 104.6, 99.0, 74.5, 56.5, 55.7, 34.5; IR (neat) ν<sub>max</sub> 3098, 2955, 2843, 1543, 1420, 1333, 1325, 1236, 1207, 1161, 1090, 1030, 949, 827, 723 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>10</sub>H<sub>13</sub>BrNO<sub>4</sub> [M+H]<sup>+</sup>: 290.0022, found: 290.0013; mp : 72–73 °C.

To a solution of **S8** (20.0 mg, 0.069 mmol) in THF (2.8 mL) and H<sub>2</sub>O (0.28 mL), freshly prepared Al(Hg) (from 40.0 mg of Al) was added at room temperature under Ar atmosphere. The reaction mixture was vigorously stirred for 8 h at room temperature before being quenched with saturated aqueous NaHCO<sub>3</sub>. The resulting mixture was filtrated by Celite pad. The filtrate was extracted three times with 10% MeOH/CHCl<sub>3</sub>. The combined organic layer was washed with brine, dried over MgSO<sub>4</sub> and concentrated. Flash chromatography (5% CH<sub>3</sub>CN/CHCl<sub>3</sub>) provided hydroxylamine **S9** (15.8 mg, 83%) as a pale yellow solid.

*N*-(2-Bromo-3,5-dimethoxyphenethyl)hydroxylamine (S9); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 6.46 (d, *J* = 2.5 Hz, 1H), 6.37 (d, *J* = 2.5 Hz, 1H), 3.86 (s, 3H), 3.79 (s, 3H), 3.19 (t, *J* = 7.0 Hz, 2H), 3.03 (t, *J* = 7.0 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 159.7, 157.0, 140.7, 107.3, 105.0, 98.0, 56.4, 55.7, 53.2, 34.3; IR (neat) ν<sub>max</sub> 3260, 2841, 1585, 1456, 1416, 1341, 1273, 1206, 1163, 1097, 1020, 824, 743 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>10</sub>H<sub>15</sub>BrNO<sub>3</sub> [M+H]<sup>+</sup>: 276.0230, found: 276.0221; mp: 74–75 °C.

To a solution of *p*-methoxybenzoic acid (56.0 mg, 0.36 mmol) and *N*-methylmorpholine (NMM, 4.0 μL, 0.036 mmol) in CH<sub>3</sub>CN (7 mL), 4-(4,6-dimethoxy-1,3,5-triazin-2-yl)-4-methylmorpholinium chloride (DMT-MM, 120.0 mg, 0.43 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 3 h at room temperature. The resulting mixture was cooled to –20 °C before an addition of the solution of hydroxylamine **S9** (100.0 mg, 0.36 mmol) in CH<sub>3</sub>CN (7 mL). The reaction mixture was stirred for 1 h at –20 °C under Ar atmosphere. The resulting mixture was quenched with excess amount of 1M HCl/H<sub>2</sub>O at –20 °C. The aqueous layer was extracted three times with EtOAc.

The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 2% CH<sub>3</sub>CN/CHCl<sub>3</sub>) provided *N*-hydroxyamide **S10** (156.0 mg, quant.) as a white solid.

*N*-(2-Bromo-3,5-dimethoxyphenethyl)-*N*-hydroxy-4-methoxybenzamide (**S10**); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.12 (d, *J* = 8.5 Hz, 2H), 6.78 (d, *J* = 7.0 Hz, 2H), 6.40 (d, *J* = 3.0 Hz, 1H), 3.37 (d, *J* = 3.0 Hz, 1H), 3.95 (t, *J* = 7.0 Hz, 2H), 3.83 (s, 3H), 3.81 (s, 3H), 3.78 (s, 3H), 3.14 (t, *J* = 6.8 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 168.2, 161.7, 159.7, 156.9, 138.9, 129.6, 123.9, 113.7, 107.7, 105.0, 98.8, 56.4, 55.7, 55.5, 50.6, 34.4; IR (neat) ν<sub>max</sub> 3009, 2934, 2839, 1585, 1514, 1456, 1418, 1319, 1254, 1171, 1163, 1086, 1022, 930, 837, 750 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>18</sub>H<sub>21</sub>BrNO<sub>5</sub> [M+H]<sup>+</sup>: 410.0598, found: 410.0584; mp: 119–121 °C.

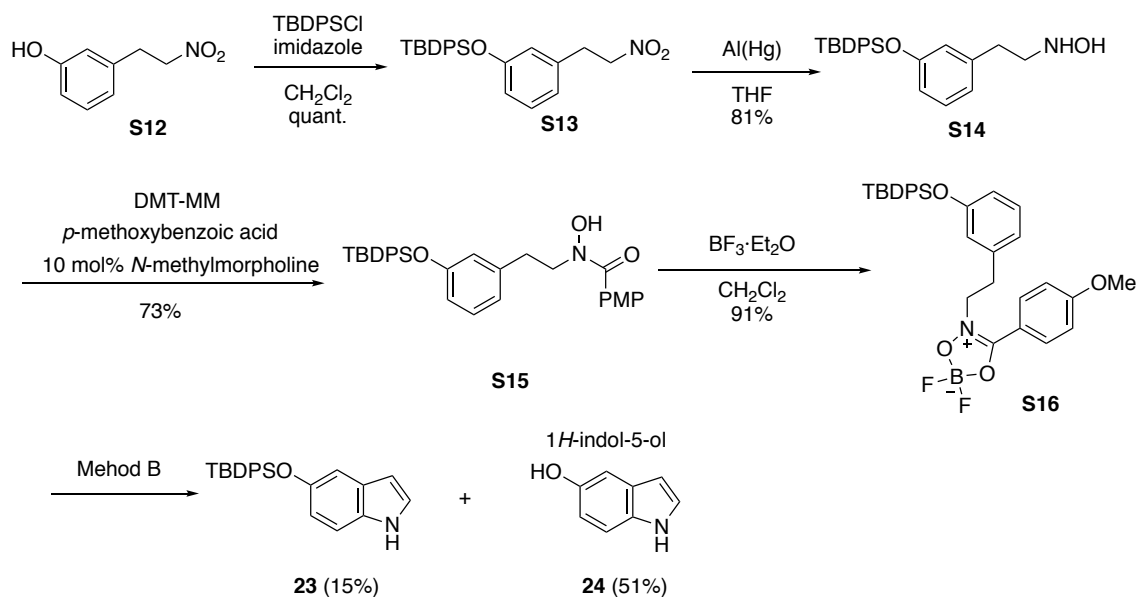
To a solution of *N*-hydroxyamide **S10** (100.0 mg, 0.246 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (8 mL), BF<sub>3</sub>·Et<sub>2</sub>O (70.0 μL, 0.492 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 1 h at room temperature. Excess BF<sub>3</sub>·Et<sub>2</sub>O and the solvent were removed from the reaction vessel under reduced pressure. Obtained white solids were collected by filtration and washed with cold Et<sub>2</sub>O. Desired pure boron complex **S11** (95.0 mg, 84%) was obtained as colorless crystals.

Boron complex **S11**; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.18 (d, *J* = 8.5 Hz, 2H), 6.84 (d, *J* = 8.5 Hz, 2H), 6.51 (d, *J* = 3.0 Hz, 1H), 6.32 (d, *J* = 3.0 Hz, 1H), 4.27 (t, *J* = 6.0 Hz, 2H), 3.84 (s, 3H), 3.82 (s, 3H), 3.76 (s, 3H), 3.29 (t, *J* = 6.0 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 163.6, 163.0, 160.2, 156.9, 137.0, 130.3, 114.5, 113.8, 107.0, 104.3, 100.1, 56.3, 55.9, 55.8, 50.1, 33.8; IR (neat) ν<sub>max</sub> 2951, 2847, 2363, 1670, 1607, 1526, 1452, 1354, 1323, 1265, 1211, 1165, 1072, 1020, 988, 926, 847, 762, 731, 654 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>18</sub>H<sub>21</sub>BrNO<sub>5</sub> [M-BF<sub>2</sub>+H<sub>2</sub>]<sup>+</sup>: 410.0598, found: 410.0581; mp : 136–138 °C.

Indole formation using **S11** was carried out with general protocol (method B; Time A: 15 min; Time B: 3 h), and desired indole **22** was obtained in 78% yield as a white solid.

4-Bromo-5,7-dimethoxy-1*H*-indole (**22**); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.34 (br s, 1H), 7.21 (t, *J* = 2.5 Hz, 1H), 6.54 (t, *J* = 2.5 Hz, 1H), 6.47 (s, 1H), 3.96 (s, 3H), 3.93 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 150.6, 145.8, 129.8, 125.0, 122.4, 103.3, 94.2, 93.2, 58.8, 55.8; IR (neat) ν<sub>max</sub> 3393, 3005, 2920, 2853, 1638, 1578, 1481, 1420, 1315, 1207, 1128, 1082, 972, 899, 843, 716, 719 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>10</sub>H<sub>11</sub>BrNO<sub>2</sub> [M+H]<sup>+</sup> : 255.9968, found: 255.9964; mp: 90–93 °C.

Synthesis of 5-*tert*-butyldiphenylsilyloxyindole (**23**) and 5-hydroxyindole (**24**).



To a solution of known 3-(2-nitroethyl)phenol (**S12**, 700.0 mg, 4.19 mmol)<sup>S5</sup> and imidazole (570.0 mg, 8.38 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (40 mL), *tert*-butyldiphenylchlorosilane (2.4 mL, 6.28 mmol) at room temperature under Ar atmosphere. The reaction mixture was stirred for 30 min before being quenched with 1M HCl/H<sub>2</sub>O. The aqueous layer was extracted three times with CH<sub>2</sub>Cl<sub>2</sub>. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 40% CH<sub>2</sub>Cl<sub>2</sub>/*n*-hexanes) provided compound **S13** (156.0 mg, quant.) as a colorless oil.

**3-(2-Nitroethyl)phenol (S13)**: <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.73 (d, *J* = 1.0 Hz, 4H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.40 (t, *J* = 7.3 Hz, 4H), 7.05 (t, *J* = 8.0 Hz, 1H), 6.71 (t, *J* = 1.0, 8.0 Hz, 2H), 6.58 (s, 1H), 4.37 (t, *J* = 7.3 Hz, 2H), 3.12 (t, *J* = 7.5 Hz, 1H), 1.13 (s, 9H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 156.1, 136.8, 135.6, 132.8, 130.1, 129.8, 127.9, 121.4, 120.1, 118.9, 76.1, 33.2, 26.6, 19.6; IR (neat) ν<sub>max</sub> 2930, 2859, 1553, 1487, 1427, 1377, 1271, 1159, 1113, 974, 972, 937, 822, 785 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>24</sub>H<sub>27</sub>NNaO<sub>3</sub>Si [M+Na]<sup>+</sup>: 428.1652, found: 428.1659.

To a solution of **S13** (1.0 g, 2.47 mmol) in THF (100 mL) and H<sub>2</sub>O (10 mL), freshly prepared Al(Hg) (from 2.0 g of Al) was added at room temperature under Ar atmosphere. The reaction mixture was vigorously stirred for 8 h at room temperature before being quenched with saturated aqueous NaHCO<sub>3</sub>. The resulting mixture was filtrated by Celite pad. The filtrate was extracted three times with 10% MeOH/CHCl<sub>3</sub>. The combined organic layer was washed with brine, dried over MgSO<sub>4</sub> and concentrated.

Flash chromatography (2% CH<sub>3</sub>CN/CHCl<sub>3</sub>) provided hydroxylamine **S14** (776.0 mg, 81%) as a colorless oil.

*tert*-Butyl(3-(2-nitroethyl)phenoxy)diphenylsilane (**S14**); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.71 (d, *J* = 7.5 Hz, 4H), 7.42 (t, *J* = 7.3 Hz, 2H), 7.37 (t, *J* = 7.5 Hz, 4H), 7.02 (t, *J* = 7.8 Hz, 1H), 6.72 (d, *J* = 7.5 Hz, 1H), 6.60-6.63 (m, 2H), 2.98 (t, *J* = 6.8 Hz, 2H), 2.68 (t, *J* = 6.5 Hz, 2H), 1.11 (s, 9H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 155.8, 140.6, 135.7, 133.0, 130.0, 129.4, 127.9, 121.8, 120.4, 117.8, 54.8, 33.1, 26.7, 19.6; IR (neat) ν<sub>max</sub> 2932, 2857, 1584, 1483, 1427, 1271, 1157, 1113, 974, 822, 743 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>24</sub>H<sub>30</sub>NO<sub>2</sub>Si [M+H]<sup>+</sup>: 392.2040, found: 392.2037.

To a solution of *p*-methoxybenzoic acid (155.0 mg, 1.02 mmol) and *N*-methylmorpholine (NMM, 11.0 μL, 0.102 mmol) in CH<sub>3</sub>CN (20 mL), 4-(4,6-dimethoxy-1,3,5-triazin-2-yl)-4-methylmorpholinium chloride (DMT-MM, 339.0 mg, 1.02 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 3 h at room temperature. The resulting mixture was cooled to -20 °C before an addition of the solution of hydroxylamine **S14** (400.0 mg, 1.02 mmol) in CH<sub>3</sub>CN (20 mL). The reaction mixture was stirred for 1 h at -20 °C under Ar atmosphere. The resulting mixture was quenched with excess amount of 1M HCl/H<sub>2</sub>O at -20 °C. The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 2% CH<sub>3</sub>CN/benzene) provided *N*-hydroxyamide **S15** (391.9 mg, 73%) as a brown oil.

*N*-(3-((*tert*-Butyldiphenylsilyl)oxy)phenethyl)-*N*-hydroxy-4-methoxybenzamide (**S15**); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.38 (br s, 1H), 7.55 (dd, *J* = 1.0, 8.0 Hz, 4H), 7.40 (tt, *J* = 1.8, 7.5 Hz, 2H), 7.33 (t, *J* = 7.5 Hz, 4H), 7.08 (d, *J* = 8.5 Hz, 2H), 6.97 (t, *J* = 7.8 Hz, 1H), 6.78 (dt, *J* = 2.3, 9.0 Hz, 2H), 6.60 (dd, *J* = 1.5, 8.0 Hz, 2H), 6.55 (t, *J* = 1.8 Hz, 1H), 3.80 (s, 3H), 3.72 (t, *J* = 6.5 Hz, 2H), 2.84 (t, *J* = 7.0 Hz, 2H), 1.09 (s, 9H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 167.8, 161.6, 155.9, 139.2, 135.6, 133.5, 133.0, 130.0, 129.7, 129.4, 127.9, 121.9, 120.7, 118.2, 113.8, 55.5, 52.6, 33.5, 26.7, 19.6; IR (neat) ν<sub>max</sub> 2932, 2857, 2359, 1605, 1485, 1427, 1252, 1159, 1113, 1028, 972, 932, 837, 714 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>32</sub>H<sub>36</sub>NO<sub>4</sub>Si [M+H]<sup>+</sup>: 526.2408, found: 526.2407.

To a solution of *N*-hydroxyamide **S15** (100.0 mg, 0.190 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (6 mL), BF<sub>3</sub>·Et<sub>2</sub>O (94.0 μL, 0.761 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 1 h at room temperature. Excess BF<sub>3</sub>·Et<sub>2</sub>O and the solvent were removed from the reaction vessel under reduced pressure. Obtained white solids were collected by filtration and washed with cold Et<sub>2</sub>O. Desired pure boron complex **S16** (99.0 mg, 91%) was obtained as colorless crystals.

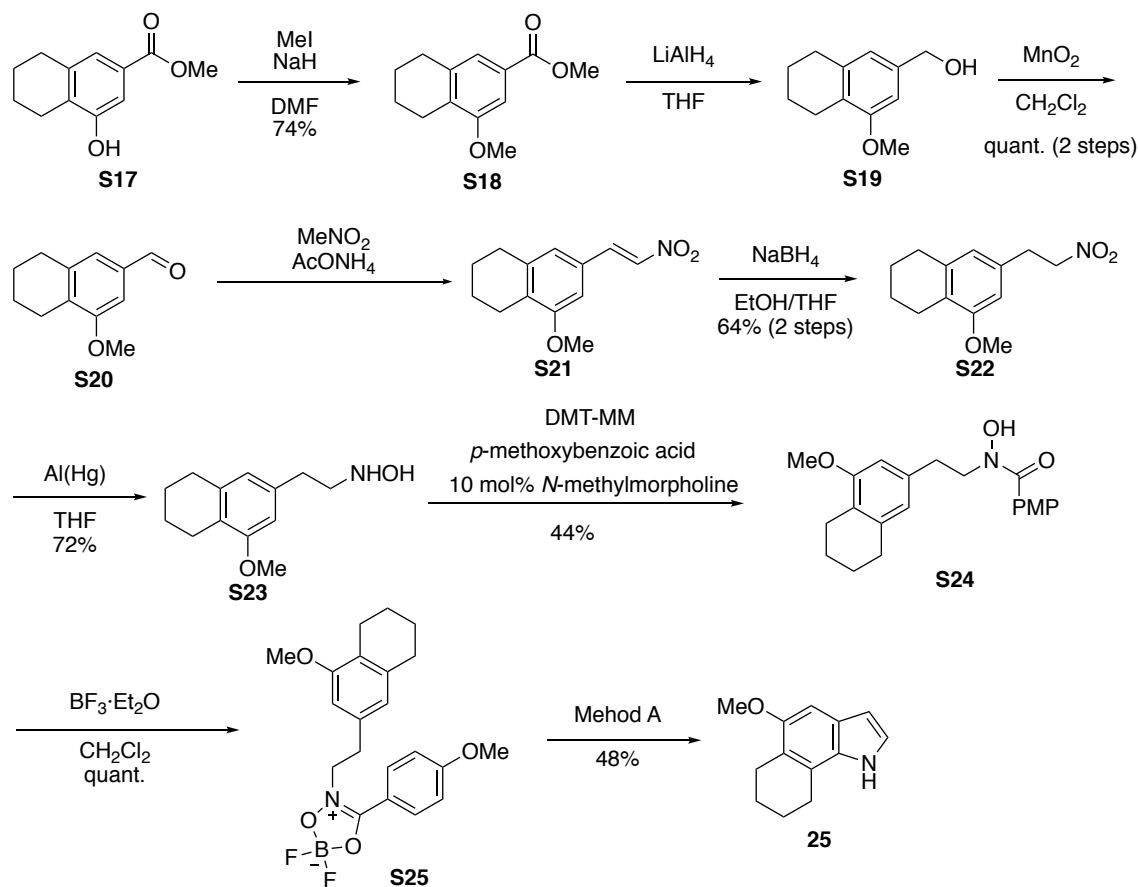
**Boron complex S16**;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.66–7.68 (m, 4H), 7.40 (tt,  $J = 7.5, 2.0$  Hz, 2H), 7.31–7.34 (m, 4H), 7.13 (dt,  $J = 2.3, 7.5$  Hz, 2H), 6.98 (t,  $J = 8.0$  Hz, 1H), 6.83 (dt,  $J = 2.3, 8.5$  Hz, 2H), 6.69 (d,  $J = 7.5$  Hz, 1H), 6.65 (t,  $J = 2.0$  Hz, 1H), 6.59 (dd,  $J = 7.8, 1.8$  Hz, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  163.7, 162.4, 156.2, 137.6, 135.6, 132.8, 130.6, 130.1, 129.9, 127.9, 121.7, 120.5, 118.8, 114.7, 114.2, 55.7, 52.4, 32.6, 26.6, 19.6; IR (neat)  $\nu_{\text{max}}$  3075, 2028, 2934, 2859, 1605, 1485, 1443, 1314, 1269, 1165, 1115, 1086, 1042, 982, 941, 841, 787, 746, 700  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{32}\text{H}_{36}\text{NO}_4$  [ $\text{M}-\text{BF}_2+\text{H}_2$ ] $^+$ : 526.2408, found: 526.2385; mp :126–128  $^\circ\text{C}$ .

Indole formation using **S19** was carried out with general protocol (method B; Time A: 10 min; Time B: 3 h), then 5-*tert*-butyldiphenylsilyloxyindole (**23**) and 5-hydroxyindole (**24**) were obtained in 15% and 51% yield respectively.

5-((*tert*-Butyldiphenylsilyl)oxy) indole (23);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.83–7.85 (m, 5H), 7.56 (tt,  $J = 1.9, 7.5$  Hz, 2H), 7.39–7.42 (m, 4H), 7.07–7.10 (m, 2H), 7.06 (t,  $J = 2.5$  Hz, 1H), 6.79 (dd,  $J = 2.5, 9.0$  Hz, 1H), 6.34–6.35 (m, 1H), 1.19 (s, 9H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  149.6, 135.7, 133.7, 131.2, 129.8, 128.4, 127.8, 124.9, 115.9, 111.2, 109.7, 102.3, 26.8, 19.7; IR (neat)  $\nu_{\text{max}}$  3420, 2930, 2859, 2363, 1622, 1576, 1472, 1427, 1285, 1219, 1155, 1113, 964, 874, 822, 723  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{24}\text{H}_{26}\text{NOSi}$  [ $\text{M}+\text{H}$ ] $^+$ : 372.1778, found: 372.1778.

5-Hydroxyindole (24) (identified with authentic sample);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.05 (br s, 1H), 7.25–7.27 (m, 1H), 7.19 (t,  $J = 3.0$  Hz, 1H), 7.05 (d,  $J = 2.5$  Hz, 1H), 6.78 (dd,  $J = 2.5, 9.0$  Hz, 1H), 6.44 (d,  $J = 2.5$  Hz, 1H), 4.54 (s, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  149.7, 131.3, 128.7, 125.4, 111.9, 111.7, 105.2, 102.2.

Synthesis of 5-methoxy-6,7,8,9-tetrahydro-1*H*-benzo[*g*]indole (**25**).



To a solution of methyl 4-hydroxy-5,6,7,8-tetrahydronaphthalene-2-carboxylate (**S17**, 3.80 g, 18.4 mmol) in DMF (124 mL), NaH (663.0 mg, 27.64 mmol) was added at room temperature under Ar atmosphere. After 20 min stirring, MeI (1.72 mL, 27.64 mmol) was added to the reaction mixture. The reaction mixture was stirred for 2 h at room temperature before being quenched with saturated aqueous NH<sub>4</sub>Cl. The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 4% EtOAc/*n*-hexanes) provided **S18** (2.99 g, 74%) as a colorless oil.

Methyl 4-methoxy-5,6,7,8-tetrahydronaphthalene-2-carboxylate (**S18**): <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.41 (s, 1H), 7.28 (s, 1H), 3.90 (s, 3H), 3.86 (s, 3H), 2.77 (t, *J* = 5.8 Hz, 2H), 2.66 (t, *J* = 6.0 Hz, 2H), 1.73–1.81 (m, 4H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 167.6, 157.4, 138.5, 131.9, 127.6, 123.1, 107.3, 55.5, 52.1, 29.7, 23.6, 22.7, 22.6; IR (neat)  $\nu_{\max}$  2936, 2839, 1717, 1584, 1435, 1346, 1287, 1231, 1177, 1101, 1065, 993, 891, 766 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>13</sub>H<sub>17</sub>O<sub>3</sub> [M+H]<sup>+</sup>: 221.1172, found: 221.1161.

To a solution of **S18** (2.75 g, 12.5 mmol) in THF (25 mL), LiAlH<sub>4</sub> (710.7 mg, 18.7 mmol) was slowly added at 0 °C under Ar atmosphere. The reaction mixture was stirred for 5 min before being quenched with saturated aqueous NH<sub>4</sub>Cl. The resulting mixture was filtrated with Celite pad. The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. The crude materials of **S19** was directly employed to the next reaction without further purification.

To a solution of the crude mixture of **S19** in CH<sub>2</sub>Cl<sub>2</sub> (125 mL), MnO<sub>2</sub> (10.87 g, 0.125mol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 8 h at room temperature before filtration with Celite pad. The filtrate was concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 4% EtOAc/*n*-hexanes) provided aldehyde **S20** [2.46 g, quant. (2 steps)] as a colorless oil.

4-Methoxy-5,6,7,8-tetrahydronaphthalene-2-carbaldehyde (**S20**); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 9.86 (s, 1H), 7.16 (s, 1H), 7.13 (s, 1H), 3.85 (s, 3H), 2.79 (t, *J* = 5.8 Hz, 2H), 2.67 (t, *J* = 5.8 Hz, 2H), 1.74-1.81 (m, 4H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 192.5, 158.0, 139.1, 134.6, 134.1, 126.0, 104.7, 55.5, 29.6, 23.8, 22.5, 22.4; IR (neat) ν<sub>max</sub> 2930, 2837, 1688, 1582, 1450, 1385, 1344, 1275, 1215, 1132, 1101, 1007, 953, 853, 752, 712 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>12</sub>H<sub>15</sub>O<sub>2</sub> [M+H]<sup>+</sup>: 191.1067, found: 191.1059.

To a solution of **S20** (2.3 g, 12.1 mmol) in nitromethane (66.7 mL), ammonium acetate (1.3 g, 16.9 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 1 h at 130 °C before being quenched with H<sub>2</sub>O at room temperature. The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. The crude materials of **S21** were employed to next reaction without further purification.

To a solution of NaBH<sub>4</sub> (810.9 mg, 21.4 mmol) in EtOH (54.0 mL), a solution of the crude mixture of **S21** in THF (10 mL) was slowly added at 0 °C under Ar atmosphere. The reaction mixture was stirred for 1.5 h at 0 °C. The resulting mixture was quenched with excess amount of 1M HCl/H<sub>2</sub>O at 0 °C followed by an addition of EtOAc. The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 4% EtOAc/*n*-hexanes) provided **S22** [1.81 g, (64%, 2 steps)] as a pale yellow oil.

5-Methoxy-7-(2-nitroethyl)-1,2,3,4-tetrahydronaphthalene (**S22**); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 6.54 (s, 1H), 6.47 (s, 1H), 4.60 (t, *J* = 7.8 Hz, 2H), 3.80 (s, 3H), 3.25 (t, *J* = 7.5 Hz, 2H), 2.71 (t, *J* = 5.8 Hz, 2H),

2.60 (t,  $J = 6.0$  Hz, 2H), 1.71–1.79 (m, 4H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  157.8, 139.1, 133.2, 125.3, 121.5, 107.2, 76.6, 55.4, 33.7, 29.7, 23.0, 22.9, 22.8; IR (neat)  $\nu_{\text{max}}$  2930, 2837, 1584, 1549, 1429, 1377, 1350, 1271, 1200, 1167, 1103, 1067, 1005, 839, 710  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{13}\text{H}_{18}\text{NO}_3$   $[\text{M}+\text{H}]^+$ : 236.1281, found: 236.1269.

To a solution of **S22** (860.0 mg, 3.66 mmol) in THF (136 mL) and  $\text{H}_2\text{O}$  (13.6 mL), freshly prepared  $\text{Al}(\text{Hg})$  (from 1.7 g of Al) was added at room temperature under Ar atmosphere. The reaction mixture was vigorously stirred for 4.5 h at room temperature before being quenched with saturated aqueous  $\text{NaHCO}_3$ . The resulting mixture was filtrated by Celite pad. The filtrate was extracted three times with  $\text{CHCl}_3$ . The combined organic layer was washed with brine, dried over  $\text{MgSO}_4$  and concentrated under reduced pressure. Flash chromatography ( $\text{SiO}_2$ , 25%  $\text{CH}_3\text{CN}/\text{CHCl}_3$ ) provided **S23** (746.0 mg, 72%) as a white solid.

*N*-(2-(4-Methoxy-5,6,7,8-tetrahydronaphthalen-2-yl)ethyl)hydroxylamine (S23);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.56 (s, 1H), 6.51 (s, 1H), 3.80 (s, 3H), 3.19 (t,  $J = 6.8$  Hz, 2H), 2.81 (t,  $J = 7.0$  Hz, 2H), 2.71 (t,  $J = 5.8$  Hz, 2H), 2.60 (t,  $J = 6.0$  Hz, 2H), 1.71–1.79 (m, 4H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  157.6, 138.7, 136.9, 124.0, 121.7, 107.6, 55.4, 55.2, 33.4, 29.8, 23.0 (2C); IR (neat)  $\nu_{\text{max}}$  3252, 2932, 2837, 1614, 1582, 1456, 1423, 1344, 1265, 1200, 1167, 1101, 1065, 1032, 833, 758  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{13}\text{H}_{20}\text{NO}_2$   $[\text{M}+\text{H}]^+$ : 222.1489, found: 222.1481; mp : 58–63  $^\circ\text{C}$ .

To a solution of *p*-methoxybenzoic acid (481.0 mg, 3.16 mmol) and *N*-methylmorpholine (NMM, 34.9  $\mu\text{L}$ , 0.316 mmol) in  $\text{CH}_3\text{CN}$  (40 mL), 4-(4,6-dimethoxy-1,3,5-triazin-2-yl)-4-methylmorpholinium chloride (DMT-MM, 1.05 g, 3.99 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 3 h at room temperature. The resulting mixture was cooled to  $-20$   $^\circ\text{C}$  before an addition of the solution of hydroxylamine **S23** (700.0 mg, 3.16 mmol) in  $\text{CH}_3\text{CN}$  (20 mL). The reaction mixture was stirred for 30 min at  $-20$   $^\circ\text{C}$  under Ar atmosphere. The resulting mixture was quenched with excess amount of 1M  $\text{HCl}/\text{H}_2\text{O}$  at  $-20$   $^\circ\text{C}$ . The aqueous layer was extracted three times with  $\text{EtOAc}$ . The combined organic layer was washed with saturated aqueous  $\text{NaCl}$ , dried over  $\text{MgSO}_4$ , and concentrated under reduced pressure. Flash chromatography ( $\text{SiO}_2$ , 10%  $\text{CH}_3\text{CN}/\text{CHCl}_3$ ) provided *N*-hydroxyamide **S24** (493.6 mg, 44%) as a pale brown solid.

*N*-Hydroxy-4-methoxy-*N*-(2-(4-methoxy-5,6,7,8-tetrahydronaphthalen-2-yl)ethyl)benzamide (S24);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.57 (br s, 1H), 7.05 (d,  $J = 8.5$  Hz, 2H), 6.77 (dt,  $J = 2.5, 9.0$  Hz, 2H), 6.38 (s, 1H), 6.31 (s, 1H), 3.88 (t,  $J = 6.8$  Hz, 2H), 3.81 (s, 3H), 3.71 (s, 3H), 2.91 (t,  $J = 6.8$  Hz, 2H), 2.64 (t,  $J = 6.0$  Hz, 2H), 2.60 (t,  $J = 6.3$  Hz, 2H), 1.71–1.80 (m, 4H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  167.9,

161.5, 138.6, 135.4, 129.6, 124.4, 124.2, 122.0, 113.5, 107.8, 55.5, 55.3, 53.0, 33.6, 29.7, 23.0 (2C); IR (neat)  $\nu_{\max}$  3210, 3067, 2833, 1694, 1456, 1398, 1319, 1063, 814, 781, 760, 741  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{21}\text{H}_{26}\text{NO}_4$   $[\text{M}+\text{H}]^+$ : 356.1856, found: 356.1843; mp: 125–126 °C.

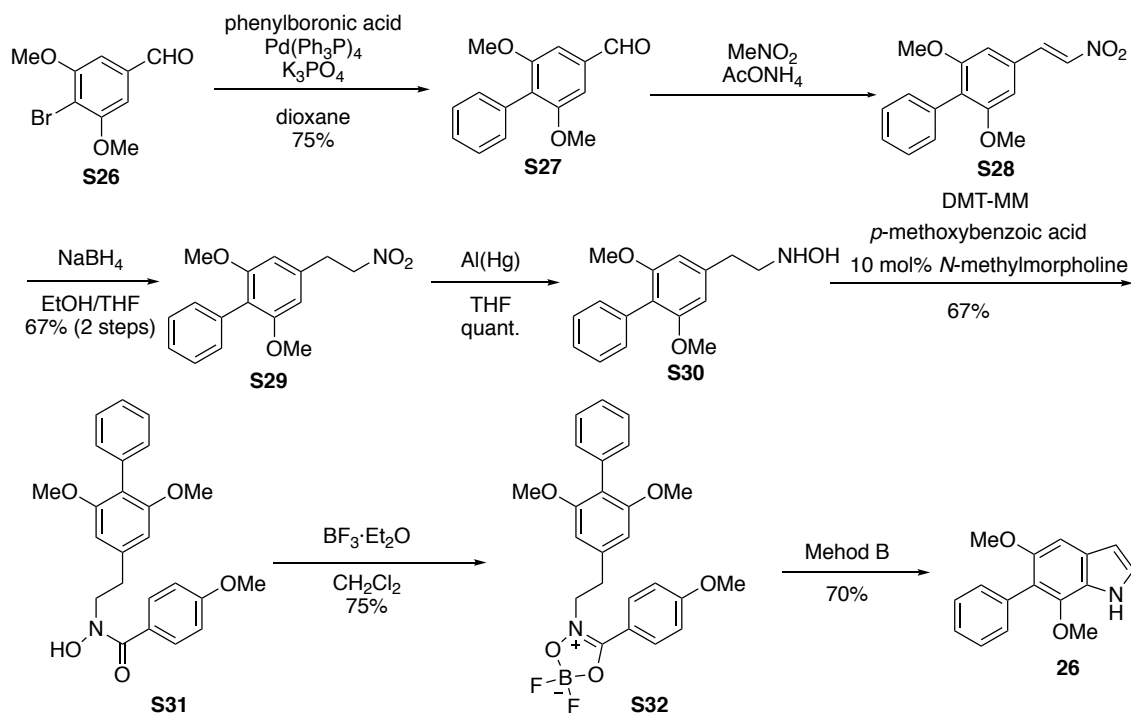
To a solution of *N*-hydroxyamide **S24** (284.1 mg, 0.80 mmol) in  $\text{CH}_2\text{Cl}_2$  (26.7 mL),  $\text{BF}_3 \cdot \text{Et}_2\text{O}$  (202.4  $\mu\text{L}$ , 1.60 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 1 h at room temperature. Excess  $\text{BF}_3 \cdot \text{Et}_2\text{O}$  and the solvent were removed from the reaction vessel under reduced pressure. Obtained white solids were collected by filtration and washed with cold  $\text{Et}_2\text{O}$ . Desired pure boron complex **S25** (357.8 mg, quant.) was obtained as a pale brown oil.

**Boron complex S25**:  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.04 (d,  $J = 8.5$  Hz, 2H), 6.81 (d,  $J = 8.5$  Hz, 2H), 6.41 (d,  $J = 4.5$  Hz, 2H), 4.12 (t,  $J = 6.0$  Hz, 2H), 3.84 (s, 3H), 3.74 (s, 3H), 3.06 (t,  $J = 6.3$  Hz, 2H), 2.59 (t,  $J = 6.0$  Hz, 2H), 2.55 (t,  $J = 6.5$  Hz, 2H), 1.69–1.74 (m, 4H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  163.4, 162.9, 157.9, 139.0, 133.7, 130.4, 125.1, 121.6, 114.3, 107.8, 55.7, 55.4, 53.1, 32.8, 29.6, 23.0 (2C), 22.9; IR (neat)  $\nu_{\max}$  2936, 2839, 1686, 1605, 1522, 1454, 1344, 1310, 1261, 1167, 1074, 982, 932, 837, 733  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{21}\text{H}_{26}\text{NO}_4$   $[\text{M}-\text{BF}_2+\text{H}_2]^+$ : 356.1856, found: 356.1836.

Indole formation using **S25** was carried out with general protocol (method A; Time A: 3 min; Time B: 1 h), and desired indole **25** was obtained in 48% yield.

5-Methoxy-6,7,8,9-tetrahydro-1*H*-benzo[*g*]indole (**25**):  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.92 (br s, 1H), 7.12 (br s, 1H), 6.93 (s, 1H), 6.47 (t,  $J = 2.3$  Hz, 1H), 3.86 (s, 3H), 2.84 (t,  $J = 5.8$  Hz, 2H), 2.75 (t,  $J = 6.0$  Hz, 2H), 1.87–1.89 (m, 4H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  152.8, 130.0, 124.5, 123.2, 121.7, 120.6, 102.8, 98.1, 55.8, 24.6, 23.7, 23.0, 22.4; IR (neat)  $\nu_{\max}$  3395, 2930, 2363, 1456, 1431, 1292, 1238, 1196, 1171, 1130, 1099, 1069, 991, 949, 887, 827, 756, 721  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{13}\text{H}_{16}\text{NO}$   $[\text{M}+\text{H}]^+$ : 202.1226, found: 202.1217; mp: 85–88 °C.

## Synthesis of 5,7-dimethoxy-6-phenylindole (**26**).



To a solution of methyl 4-bromo-3,5-dimethoxybenzaldehyde (**S26**, 500.0 mg, 2.04 mmol), phenylboronic acid (273.6 mg, 2.24 mmol) and  $\text{K}_3\text{PO}_4$  (649.6 mg, 3.06 mmol) in dioxane (10.2 mL),  $\text{Pd}(\text{PPh}_3)_4$  (58.9 mg, 0.05 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 3 days at 85 °C under Ar atmosphere before filtration with  $\text{SiO}_2$  pad (eluted with 50% EtOAc/*n*-hexanes). Excess amount of  $\text{H}_2\text{O}$  was added to the filtrate. The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over  $\text{MgSO}_4$ , and concentrated under reduced pressure. Flash chromatography ( $\text{SiO}_2$ , 10% EtOAc/*n*-hexanes) provided biphenyl compound **S27** (367.7mg, 75%) as a white solid.

**2,6-Dimethoxy-[1,1'-biphenyl]-4-carbaldehyde (S27)**;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  9.99 (s, 1H), 7.42–7.45 (m, 2H), 7.33–7.38 (m, 3H), 7.18 (s, 2H), 3.82 (s, 6H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  191.9, 158.3, 136.9, 133.1, 130.6, 128.0, 127.6, 125.9, 105.5, 56.3; IR (neat)  $\nu_{\text{max}}$  1684, 1570, 1458, 1418, 1385, 1310, 1225, 1121, 1005, 847, 737  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{15}\text{H}_{15}\text{O}_3$   $[\text{M}+\text{H}]^+$ : 243.1016, found: 243.1010; mp: 143–145 °C.

To a solution of **S27** (200.0 mg, 0.825 mmol) in nitromethane (4.6 mL), ammonium acetate (89.1 mg, 1.16 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 2 h at 130 °C before being quenched with 1M HCl/ $\text{H}_2\text{O}$  at room temperature. The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl,

dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. The crude materials of **S28** were employed to next reaction without further purification.

To a solution of NaBH<sub>4</sub> (62.5 mg, 1.65 mmol) in EtOH (4.0 mL), a solution of the crude mixture of **S28** in THF (3 mL) was slowly added at 0 °C under Ar atmosphere. The reaction mixture was stirred for 40 min at 0 °C. The resulting mixture was quenched with excess amount of 1M HCl/H<sub>2</sub>O at 0 °C followed by an addition of EtOAc. The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 70% CHCl<sub>3</sub>/*n*-hexanes) provided **S29** [132.4 mg, 67% (2 steps)] as a pale yellow oil.

2,6-Dimethoxy-4-(2-nitroethyl)-1,1'-biphenyl (S29); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.41 (t, *J* = 7.5 Hz, 2H), 7.31–7.34 (m, 3H), 6.49 (s, 2H), 4.68 (t, *J* = 7.5 Hz, 2H), 3.73 (s, 6H), 3.36 (t, *J* = 7.5 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 158.0, 136.6, 133.8, 131.0, 127.8, 127.0, 118.7, 104.6 (2C), 76.4, 56.1 (2C), 34.2; IR (neat) ν<sub>max</sub> 3019, 2938, 2839, 1582, 1537, 1458, 1416, 1379, 1215, 1124, 1009, 910, 870, 791, 745 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>16</sub>H<sub>18</sub>NO<sub>4</sub> [M+H]<sup>+</sup>: 288.1230, found: 288.1223; mp : 139–140 °C.

To a solution of **S29** (132.0 mg, 0.46 mmol) in THF (18.4 mL) and H<sub>2</sub>O (1.84 mL), freshly prepared Al(Hg) (from 264.0 mg of Al) was added at room temperature under Ar atmosphere. The reaction mixture was vigorously stirred for 8 h at room temperature before being quenched with saturated aqueous NaHCO<sub>3</sub>. The resulting mixture was filtrated by Celite pad. The filtrate was extracted three times with CHCl<sub>3</sub>. The combined organic layer was washed with brine, dried over MgSO<sub>4</sub> and concentrated to provide hydroxylamine **S30** (126.0 mg, quant.) as a white solid.

*N*-(2-(2,6-Dimethoxy-[1,1'-biphenyl]-4-yl)ethyl)hydroxylamine (S30); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.40 (t, *J* = 7.3 Hz, 2H), 7.29–7.34 (m, 3H), 6.52 (s, 2H), 3.73 (s, 6H), 3.27 (t, *J* = 7.0 Hz, 2H), 2.93 (t, *J* = 6.8 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 157.8, 140.3, 134.2, 131.1, 127.8, 126.9, 117.7, 104.9, 56.1, 55.1, 34.2; IR (neat) ν<sub>max</sub> 2932, 2837, 2361, 1609, 1580, 1462, 1412, 1319, 1231, 1123, 1009, 901, 849, 812, 766, 694 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>16</sub>H<sub>20</sub>NO<sub>3</sub> [M+H]<sup>+</sup>: 274.1438, found: 274.1428; mp: 122–129 °C.

To a solution of *p*-methoxybenzoic acid (69.2 mg, 0.46 mmol) and *N*-methylmorpholine (NMM, 5.0 μL, 0.045 mmol) in CH<sub>3</sub>CN (9 mL), 4-(4,6-dimethoxy-1,3,5-triazin-2-yl)-4-methylmorpholinium chloride (DMT-MM, 69.2 mg, 0.46 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 3 h at room temperature. The resulting mixture was cooled to –20 °C

before an addition of the solution of hydroxylamine **S30** (124.3 mg, 0.46 mmol) in CH<sub>3</sub>CN (7 mL). The reaction mixture was stirred for 1 h at –20 °C under Ar atmosphere. The resulting mixture was quenched with excess amount of 1M HCl/H<sub>2</sub>O at –20 °C. The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 5% CH<sub>3</sub>CN/CHCl<sub>3</sub>) provided *N*-hydroxyamide **S31** (138.1 mg, 74%) as a pale yellow solid.

*N*-(2-(2,6-Dimethoxy-[1,1'-biphenyl]-4-yl)ethyl)-*N*-hydroxy-4-methoxybenzamide (**S31**); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.75 (br s, 1H), 7.41 (t, *J* = 7.8 Hz, 2H), 7.33 (d, *J* = 7.5 Hz, 3H), 7.09 (d, *J* = 8.5 Hz, 2H), 6.80 (d, *J* = 8.5 Hz, 2H), 6.33 (s, 2H), 3.97 (t, *J* = 6.0 Hz, 2H), 3.78 (s, 3H), 3.63 (s, 6H), 3.01 (t, *J* = 6.0 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 168.0, 161.5, 157.7, 138.8, 134.1, 131.0, 129.6, 127.8, 126.9, 124.2, 118.0, 113.6, 105.2, 56.0, 55.5, 52.7, 34.0; IR (neat)  $\nu_{\max}$  1599, 1570, 1516, 1420, 1416, 1304, 1240, 1179, 1119, 1024, 837, 745 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>24</sub>H<sub>26</sub>NO<sub>5</sub> [M+H]<sup>+</sup>: 408.1805, found: 408.1790; mp: 158–159 °C.

To a solution of *N*-hydroxyamide **S31** (121.5 mg, 0.30 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (10 mL), BF<sub>3</sub>·Et<sub>2</sub>O (70.0 μL, 0.492 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 1 h at room temperature. Excess BF<sub>3</sub>·Et<sub>2</sub>O and the solvent were removed from the reaction vessel under reduced pressure. Obtained white solids were collected by filtration and washed with cold 50% CH<sub>2</sub>Cl<sub>2</sub>/*n*-hexanes. Recrystallization with 50% CH<sub>2</sub>Cl<sub>2</sub>/*n*-hexanes provided pure boron complex **S32** (103.0 mg, 75%) was obtained as colorless crystals.

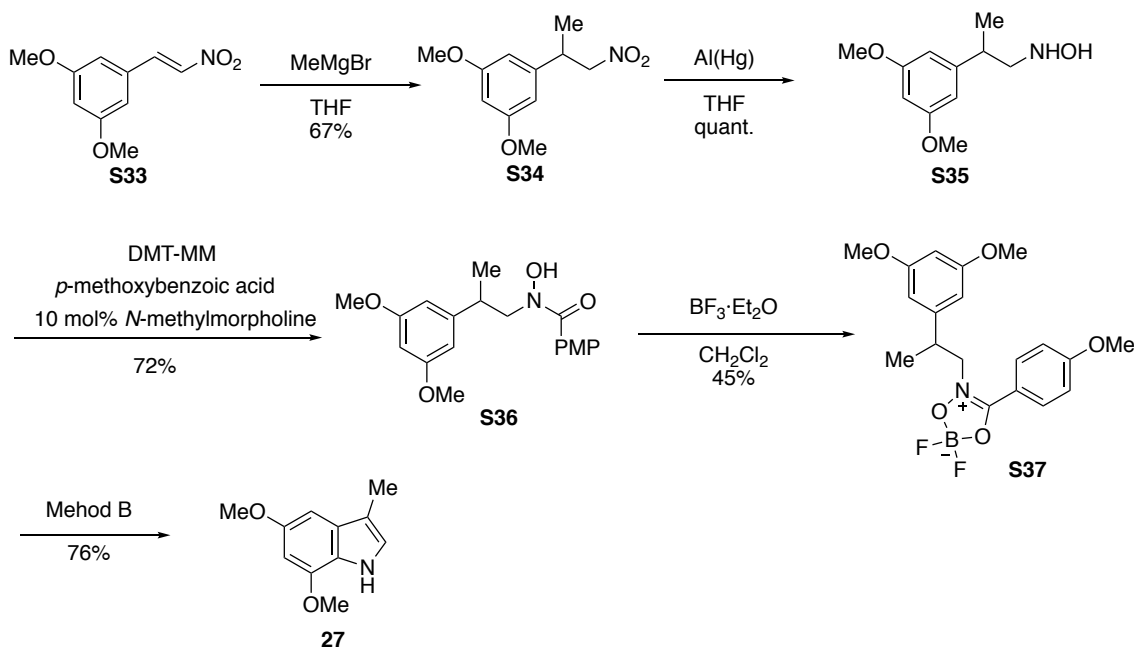
Boron complex **S32**; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.40 (t, *J* = 7.3 Hz, 2H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.26–7.28 (m, 2H), 7.12 (dt, *J* = 2.3, 8.5 Hz, 2H), 6.85 (dt, *J* = 2.6, 9.0 Hz, 2H), 6.40 (s, 2H), 4.20 (t, *J* = 6.0 Hz, 2H), 3.81 (s, 3H), 3.64 (s, 6H), 3.17 (t, *J* = 6.3 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 163.5, 163.2, 158.0, 137.0, 133.8, 130.9, 130.4, 127.8, 127.0, 118.5, 114.5, 114.2, 104.9, 56.0, 55.7, 52.7, 33.4; IR (neat)  $\nu_{\max}$  2940, 2938, 2839, 1605, 1524, 1458, 1416, 1312, 1263, 1146, 1119, 1069, 984, 935, 827, 768, 735, 702 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>24</sub>H<sub>26</sub>NO<sub>5</sub> [M–BF<sub>2</sub>+H<sub>2</sub>]<sup>+</sup>: 408.1805, found: 408.1790; mp: 202–205 °C.

Indole formation using **S32** was carried out with general protocol (method B; Time A: 15 min; Time B: 23 h), and desired indole **26** was obtained in 70% yield.

5,7-Dimethoxy-6-phenyl-1*H*-indole (**26**); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.27 (br s, 1H), 7.42–7.46 (m, 4H), 7.34 (s, 1H), 7.20 (d, *J* = 1.5 Hz, 1H), 6.96 (s, 1H), 6.53 (s, 1H), 3.75 (s, 3H), 3.47 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 152.5, 143.7, 134.9, 131.3, 128.2, 128.0, 126.9, 124.8, 124.7, 119.1, 103.1,

97.4, 60.7, 56.5; IR (neat)  $\nu_{\max}$  3431, 2997, 2924, 1628, 1564, 1477, 1435, 1396, 1308, 1265, 1227, 1188, 1146, 1080, 988, 934, 829, 822, 745, 704  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{16}\text{H}_{16}\text{NO}_2$   $[\text{M}+\text{H}]^+$ : 254.1176, found: 254.1167; mp: 165–168 °C.

### Synthesis of 5,7-dimethoxy-3-methylindole (27).



To a solution of nitrostyrene **S33**<sup>S2)</sup> (1.0 g, 4.78 mmol) in THF (96 mL),  $\text{MeMgBr}$  (1.9 mL, 5.74 mmol, 3M THF solution) was slowly added at  $-40$  °C under Ar atmosphere. The reaction mixture was stirred for 1 h at  $-40$  °C before being quenched with excess amount of 1M  $\text{HCl}/\text{H}_2\text{O}$  at  $-40$  °C. The aqueous layer was extracted three times with  $\text{EtOAc}$ . The combined organic layer was washed with saturated aqueous  $\text{NaCl}$ , dried over  $\text{MgSO}_4$ , and concentrated under reduced pressure. Flash chromatography ( $\text{SiO}_2$ , 5%  $\text{EtOAc}/n$ -hexanes) provided **S34** (720.6 mg, 67%) as a pale brown solid.

1,3-Dimethoxy-5-(1-nitropropan-2-yl)benzene (S34);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.36 (s, 3H), 4.53 (dd,  $J = 12.0, 7.5$  Hz, 1H), 4.45 (dd,  $J = 12.5, 8.5$  Hz, 1H), 3.77 (s, 6H), 3.55 (sext,  $J = 7.5$  Hz, 1H), 1.35 (d,  $J = 7.0$  Hz, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  161.2, 143.4, 105.2, 99.0, 81.7, 55.4, 38.9, 18.7; IR (neat)  $\nu_{\max}$  3011, 2940, 2841, 1690, 1597, 1545, 1458, 1385, 1354, 1306, 1196, 1153, 1063, 957, 881, 849, 704  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{11}\text{H}_{16}\text{NO}_4$   $[\text{M}+\text{H}]^+$ : 226.1074, found: 226.1063; mp: 32–34 °C.

To a solution of **S34** (331.0 mg, 1.47 mmol) in THF (60 mL) and H<sub>2</sub>O (6.0 mL), freshly prepared Al(Hg) (from 660.0 mg of Al) was added at room temperature under Ar atmosphere. The reaction mixture was vigorously stirred for 8 h at room temperature before being quenched with saturated aqueous NaHCO<sub>3</sub>. The resulting mixture was filtrated by Celite pad. The filtrate was extracted three times with CHCl<sub>3</sub>. The combined organic layer was washed with brine, dried over MgSO<sub>4</sub> and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 5% MeOH/ CHCl<sub>3</sub>) provided **S35** (309.1 mg, quant.) as a colorless oil.

*N*-(2-(3,5-Dimethoxyphenyl)propyl)hydroxylamine (**S35**); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 6.37 (d, *J* = 2.5 Hz, 2H), 6.34 (t, *J* = 2.3 Hz, 1H), 3.77 (s, 6H), 3.00–3.06 (m, 3H), 1.24 (d, *J* = 7.0 Hz, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 161.0, 147.0, 105.4, 98.3, 60.6, 55.3, 37.2, 19.7; IR (neat)  $\nu_{\max}$  2959, 2837, 1593, 1456, 1348, 1290, 1204, 1152, 1057, 1026, 926, 831, 754 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>11</sub>H<sub>18</sub>NO<sub>3</sub> [M+H]<sup>+</sup>: 212.1281, found: 212.1281.

To a solution of *p*-methoxybenzoic acid (203.0 mg, 1.34 mmol) and *N*-methylmorpholine (NMM, 14.7 μL, 0.134 mmol) in CH<sub>3</sub>CN (20 mL), 4-(4,6-dimethoxy-1,3,5-triazin-2-yl)-4-methylmorpholium chloride (DMT-MM, 443.3 mg, 1.60 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 3 h at room temperature. The resulting mixture was cooled to –20 °C before an addition of the solution of hydroxylamine **S35** (272.0 mg, 1.29 mmol) in CH<sub>3</sub>CN (7 mL). The reaction mixture was stirred for 30 min at –20 °C under Ar atmosphere. The resulting mixture was quenched with excess amount of 1M HCl/H<sub>2</sub>O at –20 °C. The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 35% EtOAc/*n*-hexanes) provided *N*-hydroxyamide **S36** (319.5 mg, 72%) as a pale yellow oil.

*N*-(2-(3,5-Dimethoxyphenyl)propyl)-*N*-hydroxy-4-methoxybenzamide (**S36**); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.09 (d, *J* = 8.0 Hz, 2H), 6.80 (d, *J* = 8.5 Hz, 2H), 6.32 (t, *J* = 2.0 Hz, 1H), 6.21 (d, *J* = 2.5 Hz, 2H), 3.82 (s, 3H), 3.73–3.80 (m, 2H), 3.72 (s, 6H), 3.36 (sext, *J* = 7.2 Hz, 1H), 1.20 (d, *J* = 7.5 Hz, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 167.8, 161.5, 160.9, 145.4, 129.7, 124.3, 113.7, 105.5, 98.9, 58.1, 55.5, 55.4, 37.5, 18.1; IR (neat)  $\nu_{\max}$  3144, 2936, 2841, 1587, 1501, 1454, 1429, 1356, 1331, 1294, 1256, 1204, 1165, 1069, 1024, 986, 918, 835, 797, 756 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>19</sub>H<sub>24</sub>NO<sub>5</sub> [M+H]<sup>+</sup>: 346.1649, found: 346.1648.

To a solution of *N*-hydroxyamide **S36** (286.0 mg, 0.828 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (28 mL), BF<sub>3</sub>·Et<sub>2</sub>O (204.0 μL, 1.66 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred

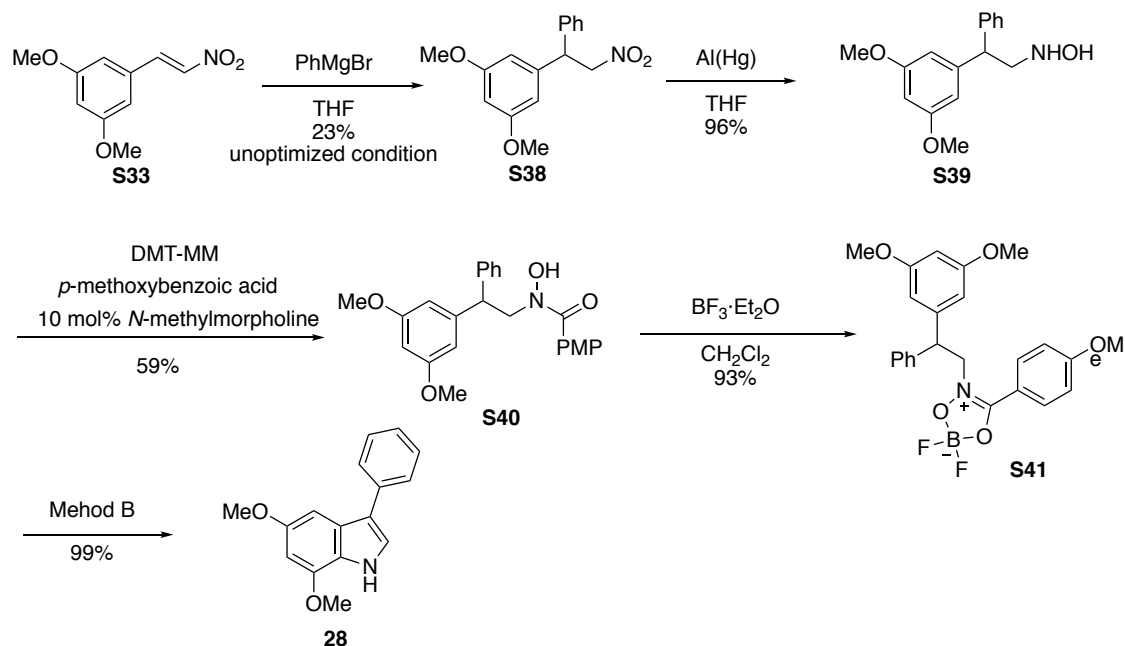
for 1 h at room temperature. Excess  $\text{BF}_3 \cdot \text{Et}_2\text{O}$  and the solvent were removed from the reaction vessel under reduced pressure. Obtained white solids were collected by filtration and washed with cold  $\text{Et}_2\text{O}$ . Desired pure boron complex **S37** (146.0 mg, 45%) was obtained as a white solid.

**Boron complex S37**:  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.12-7.14 (m, 2H), 6.84–6.86 (m, 2H), 6.28 (d,  $J$  = 2.0 Hz, 1H), 6.26 (d,  $J$  = 1.5 Hz, 2H), 3.97 (d,  $J$  = 6.0 Hz, 2H), 3.85 (s, 3H), 3.72 (s, 6H), 3.51 (m, 1H), 1.33 (d,  $J$  = 7.5 Hz, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  163.5, 162.9, 161.2, 143.4, 130.4, 114.5, 114.2, 105.0, 100.1, 58.0, 55.7, 55.4, 37.3, 17.4; IR (neat)  $\nu_{\text{max}}$  1589, 1520, 1456, 1360, 1275, 1206, 1144, 1074, 1042, 928, 839, 702  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{19}\text{H}_{24}\text{NO}_5$   $[\text{M}-\text{BF}_2+\text{H}_2]^+$ : 346.1649, found: 346.1626; mp: 133–135  $^\circ\text{C}$ .

Indole formation using **S37** was carried out with general protocol (method B; Time A: 10 min; Time B: 6 h), and desired indole **27** was obtained in 76% yield.

**5,7-Dimethoxy-3-methyl-1H-indole (27)**:  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.97 (br s, 1H), 6.91 (s, 1H), 6.59 (d,  $J$  = 2.5 Hz, 1H), 6.35 (d,  $J$  = 2.5 Hz, 1H), 3.91 (s, 3H), 3.87 (s, 3H), 2.29 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  154.8, 146.5, 128.9, 122.1, 121.7, 112.0, 94.2, 92.2, 56.0, 55.5, 10.0; IR (neat)  $\nu_{\text{max}}$  3387, 3009, 2959, 2920, 2363, 1636, 1584, 1493, 1443, 1371, 1323, 1256, 1209, 1128, 1059, 1047, 984, 928, 833, 791, 731  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{11}\text{H}_{14}\text{NO}_2$   $[\text{M}+\text{H}]^+$ : 192.1019, found: 192.1009; mp: 77–78  $^\circ\text{C}$ .

### Synthesis of 5,7-dimethoxy-3-phenylindole (**28**).



To a solution of nitrostyrene **S33** (1.0 g, 4.78 mmol) in THF (100 mL), PhMgBr (7.2 mL, 7.18 mmol, 1M THF solution) was slowly added at  $-40\text{ }^{\circ}\text{C}$  under Ar atmosphere. The reaction mixture was stirred for 30 min at  $-40\text{ }^{\circ}\text{C}$  before being quenched with excess amount of 1M HCl/H<sub>2</sub>O at  $-40\text{ }^{\circ}\text{C}$ . The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 5% EtOAc/*n*-hexanes) provided **S38** (317.5 mg, 23%) as a pale yellow solid.

**1,3-Dimethoxy-5-(2-nitro-1-phenylethyl)benzene (S38)**; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.33 (t,  $J = 7.0$  Hz, 2H), 7.24–7.27 (m, 3H), 6.38 (d,  $J = 1.5$  Hz, 2H), 6.35 (t,  $J = 2.0$  Hz, 1H), 4.94–4.96 (m, 2H), 4.83 (t,  $J = 8.3$  Hz, 1H), 3.76 (s, 6H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  161.3, 141.6, 139.1, 129.2, 127.8, 127.7, 106.2, 99.0, 79.2, 55.5, 49.2; IR (neat)  $\nu_{\text{max}}$  2839, 2361, 1593, 1549, 1431, 1429, 1377, 1344, 1317, 1292, 1204, 1153, 1067, 1003, 926, 833, 766 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>16</sub>H<sub>18</sub>NO<sub>4</sub> [M+H]<sup>+</sup>: 288.1230, found: 288.1234; mp : 141–143 °C.

To a solution of **S38** (283.0 mg, 0.990 mmol) in THF (40 mL) and H<sub>2</sub>O (4.0 mL), freshly prepared Al(Hg) (from 566.0 mg of Al) was added at room temperature under Ar atmosphere. The reaction mixture was vigorously stirred for 4 h at room temperature before being quenched with saturated aqueous NaHCO<sub>3</sub>. The resulting mixture was filtrated by Celite pad. The filtrate was extracted three times with CHCl<sub>3</sub>. The combined organic layer was washed saturated aqueous NaCl, dried over MgSO<sub>4</sub> and

concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 5% MeOH/ CHCl<sub>3</sub>) provided **S39** (216.6 mg, 96%) as a white solid.

*N*-(2-(3,5-Dimethoxyphenyl)-2-phenylethyl)hydroxylamine (**S39**); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.30 (t, *J* = 7.5 Hz, 2H), 7.25 (d, *J* = 6.5 Hz, 2H), 7.21 (t, *J* = 7.0 Hz, 1H), 6.41 (d, *J* = 2.5 Hz, 2H), 6.32 (t, *J* = 2.0 Hz, 1H), 4.32 (t, *J* = 7.8 Hz, 1H), 3.75 (s, 6H), 3.50 (d, *J* = 7.5 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 161.1, 144.7, 142.1, 128.8, 128.1, 126.9, 106.5, 98.4, 58.2, 55.4, 48.0; IR (neat)  $\nu_{\max}$  2930, 2837, 1593, 1452, 1427, 1342, 1290, 1204, 1150, 1063, 926, 831, 752 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>16</sub>H<sub>20</sub>NO<sub>3</sub> [M+H]<sup>+</sup>: 274.1438, found: 274.1439; mp: 142–143 °C.

To a solution of *p*-methoxybenzoic acid (120.2 mg, 0.79 mmol) and *N*-methylmorpholine (NMM, 8.7 μL, 0.079 mmol) in CH<sub>3</sub>CN (10 mL), 4-(4,6-dimethoxy-1,3,5-triazin-2-yl)-4-methylmorpholinium chloride (DMT-MM, 262.4 mg, 0.948 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 3 h at room temperature. The resulting mixture was cooled to –20 °C before an addition of the solution of hydroxylamine **S39** (216.0 mg, 0.79 mmol) in CH<sub>3</sub>CN (6 mL). The reaction mixture was stirred for 3 h at –20 °C under Ar atmosphere. The resulting mixture was quenched with excess amount of 1M HCl/H<sub>2</sub>O at –20 °C. The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 35% EtOAc/*n*-hexanes) provided *N*-hydroxyamide **S40** (189.4 mg, 59%) as a pale brown solid.

*N*-(2-(3,5-Dimethoxyphenyl)-2-phenylethyl)-*N*-hydroxy-4-methoxybenzamide (**S40**); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.24–7.285 (m, 3H), 7.14 (d, *J* = 7.0 Hz, 2H), 6.98 (d, *J* = 8.0 Hz, 2H), 6.79 (d, *J* = 9.5 Hz, 2H), 6.34 (t, *J* = 7.3 Hz, 1H), 6.28 (d, *J* = 2.5 Hz, 2H), 4.65 (t, *J* = 7.3 Hz, 1H), 4.26–4.28 (m, 2H), 3.84 (s, 3H), 3.73 (s, 6H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 167.9, 161.5, 160.9, 143.3, 140.8, 129.8, 128.7, 128.3, 127.1, 124.1, 113.6, 106.7, 98.7, 55.8, 55.5, 55.4, 47.7; IR (neat)  $\nu_{\max}$  2938, 2837, 1591, 1508, 1429, 1302, 1260, 1161, 1157, 1069, 1024, 943, 837, 797, 704 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>24</sub>H<sub>26</sub>NO<sub>5</sub> [M+H]<sup>+</sup>: 408.1805, found: 408.1785; mp: 139–140 °C.

To a solution of *N*-hydroxyamide **S40** (100.0 mg, 0.246 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (28 mL), BF<sub>3</sub>·Et<sub>2</sub>O (60.6 μL, 0.491 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 1 h at room temperature. Excess BF<sub>3</sub>·Et<sub>2</sub>O and the solvent were removed from the reaction vessel under reduced pressure. Obtained white solids were collected by filtration and washed with cold Et<sub>2</sub>O. Desired pure boron complex **S41** (103.9 mg, 93%) was obtained as a white solid.

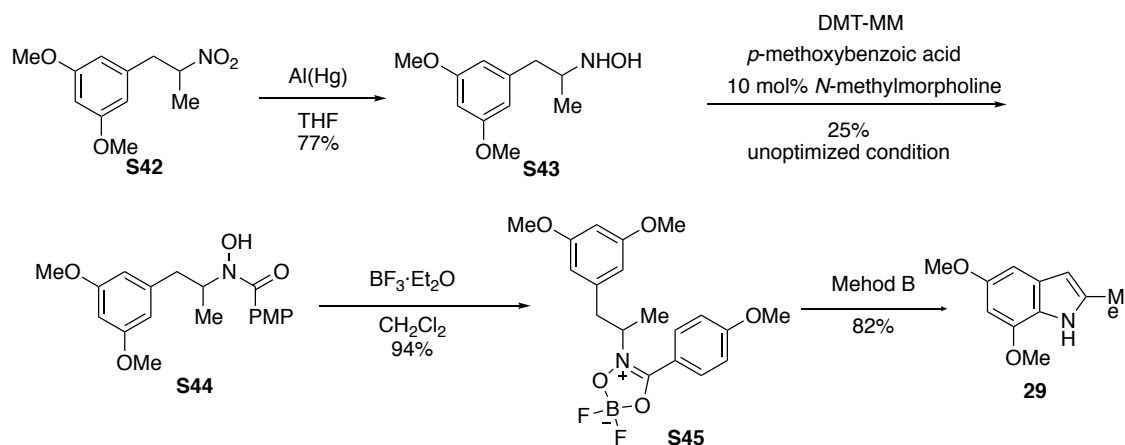
Boron complex **S41**; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.22–7.29 (m, 4H), 7.16 (d, *J* = 7.0 Hz, 2H), 7.05

(d,  $J = 9.0$  Hz, 2H), 6.84 (d,  $J = 9.0$  Hz, 2H), 6.30 (t,  $J = 2.3$  Hz, 1H), 6.26 (d,  $J = 2.0$  Hz, 2H), 4.76 (t,  $J = 7.8$  Hz, 1H), 4.47 (dd,  $J = 8.5, 13.5$  Hz, 1H), 4.41 (dd,  $J = 7.0, 13.0$  Hz, 1H), 3.85 (s, 3H), 3.70 (s, 6H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  163.5, 163.3, 161.2, 141.9, 139.2, 130.4, 129.0, 128.0, 127.7, 114.6, 114.2, 106.3, 99.7, 55.7 (2C), 55.4, 47.2; IR (neat)  $\nu_{\text{max}}$  2940, 2843, 1595, 1526, 1458, 1425, 1312, 1265, 1202, 1144, 1072, 1016, 976, 841, 702  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{24}\text{H}_{26}\text{NO}_5$   $[\text{M}-\text{BF}_2+\text{H}_2]^+$ : 408.1805, found: 408.1780; mp: 139–141  $^\circ\text{C}$ .

Indole formation using **S41** was carried out with general protocol (method B; Time A: 15 min; Time B: 6 h), and desired indole **28** was obtained in 99% yield.

5,7-Dimethoxy-3-phenyl-1*H*-indole (**28**);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.37 (br s, 1H), 7.65 (d,  $J = 6.5$  Hz, 2H), 4.46 (t,  $J = 7.8$  Hz, 2H), 7.30 (t,  $J = 2.8$  Hz, 2H), 6.96 (d,  $J = 1.5$  Hz, 1H), 6.41 (d,  $J = 2.5$  Hz, 1H), 4.00 (s, 3H), 3.87 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  155.7, 146.7, 136.0, 128.9, 127.5, 126.4, 126.0, 122.7, 121.8, 118.7, 94.7, 93.0, 56.1, 55.6; IR (neat)  $\nu_{\text{max}}$  3308, 3003, 2961, 2835, 1634, 1595, 1487, 1447, 1331, 1298, 1273, 1211, 1207, 1148, 1086, 1051, 1003, 935, 797, 743, 694, 675  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{16}\text{H}_{16}\text{NO}_2$   $[\text{M}+\text{H}]^+$ : 254.1176, found: 254.1161; mp: 91–94  $^\circ\text{C}$ .

#### Synthesis of 5,7-dimethoxy-2-methylindole (**29**)



To a solution of known nitro compound **S42** (274.0 mg, 1.216 mmol)<sup>S6)</sup> in THF (50 mL) and  $\text{H}_2\text{O}$  (5.0 mL), freshly prepared Al(Hg) (from 550.0 mg of Al) was added at room temperature under Ar atmosphere. The reaction mixture was vigorously stirred for 8 h at room temperature before being quenched with saturated aqueous  $\text{NaHCO}_3$ . The resulting mixture was filtrated by Celite pad. The filtrate was extracted three times with  $\text{CHCl}_3$ . The combined organic layer was washed with saturated aqueous NaCl, dried over  $\text{MgSO}_4$  and concentrated under reduced pressure. Flash chromatography ( $\text{SiO}_2$ , 2%MeOH/  $\text{CHCl}_3$ ) provided **S43** (198.6 mg, 77%) as a white solid.

*N*-(1-(3,5-Dimethoxyphenyl)propan-2-yl)hydroxylamine (S43);  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.36 (s, 2H), 6.33 (d,  $J = 2.0$  Hz, 1H), 3.78 (s, 6H), 3.21 (sext,  $J = 6.0$  Hz, 1H), 2.79 (dd,  $J = 7.0, 13.0$  Hz, 1H), 2.56 (dd,  $J = 6.8, 13.3$  Hz, 1H), 1.11 (dd,  $J = 1.8, 6.3$  Hz, 3H);  $^{13}\text{C NMR}$  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  161.0, 141.1, 107.4, 98.4, 58.4, 55.4, 40.4, 17.8; IR (neat)  $\nu_{\text{max}}$  3250, 2972, 2845, 1591, 1454, 1348, 1296, 1206, 1148, 1053, 966, 930, 895, 831  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{11}\text{H}_{18}\text{NO}_3$   $[\text{M}+\text{H}]^+$ : 212.1281, found: 212.1281; mp: 88–90 °C.

To a solution of *p*-methoxybenzoic acid (108.0 mg, 0.710 mmol) and *N*-methylmorpholine (NMM, 7.8  $\mu\text{L}$ , 0.071 mmol) in  $\text{CH}_3\text{CN}$  (10 mL), 4-(4,6-dimethoxy-1,3,5-triazin-2-yl)-4-methylmorpholium chloride (DMT-MM, 235.8 mg, 0.852 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 3 h at room temperature. The resulting mixture was cooled to  $-20$  °C before an addition of the solution of hydroxylamine S43 (150.0 mg, 0.71 mmol) in  $\text{CH}_3\text{CN}$  (4 mL). The reaction mixture was stirred for 1 h at  $-20$  °C under Ar atmosphere. The resulting mixture was quenched with excess amount of 1M HCl/ $\text{H}_2\text{O}$  at  $-20$  °C. The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over  $\text{MgSO}_4$ , and concentrated under reduced pressure. Flash chromatography ( $\text{SiO}_2$ , 35% EtOAc/*n*-hexanes) provided *N*-hydroxyamide S44 (62.2 mg, 25%) as a brown oil.

*N*-(1-(3,5-Dimethoxyphenyl)propan-2-yl)-*N*-hydroxy-4-methoxybenzamide (S44);  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.94 (d,  $J = 8.0$  Hz, 2H), 6.76 (dt,  $J = 2.3, 9.0$  Hz, 2H), 6.34 (t,  $J = 2.3$  Hz, 1H), 6.18 (d,  $J = 2.0$  Hz, 2H), 3.80 (s, 3H), 3.71 (s, 6H), 3.09 (dd,  $J = 10.3, 13.8$  Hz, 1H), 2.57 (dd,  $J = 4.3, 13.8$  Hz, 1H), 1.41 (d,  $J = 7.0$  Hz, 3H);  $^{13}\text{C NMR}$  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  167.6, 161.4, 160.9, 140.4, 129.3, 124.6, 113.7, 107.2, 99.2, 59.0, 55.5, 55.4, 40.1, 18.8; IR (neat)  $\nu_{\text{max}}$  2934, 2837, 1593, 1514, 1429, 1427, 1300, 1252, 1204, 1148, 1057, 1028, 930, 835. 752  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{19}\text{H}_{24}\text{NO}_5$   $[\text{M}+\text{H}]^+$ : 346.1649, found: 346.1650.

To a solution of *N*-hydroxyamide S44 (58.0 mg, 0.168 mmol) in  $\text{CH}_2\text{Cl}_2$  (5.6 mL),  $\text{BF}_3 \cdot \text{Et}_2\text{O}$  (41.4  $\mu\text{L}$ , 0.336 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 1 h at room temperature. Excess  $\text{BF}_3 \cdot \text{Et}_2\text{O}$  and the solvent were removed from the reaction vessel under reduced pressure. Obtained white solids were collected by filtration and washed with cold  $\text{Et}_2\text{O}$ . Desired pure boron complex S45 (62.2 mg, 94%) was obtained as a white solid.

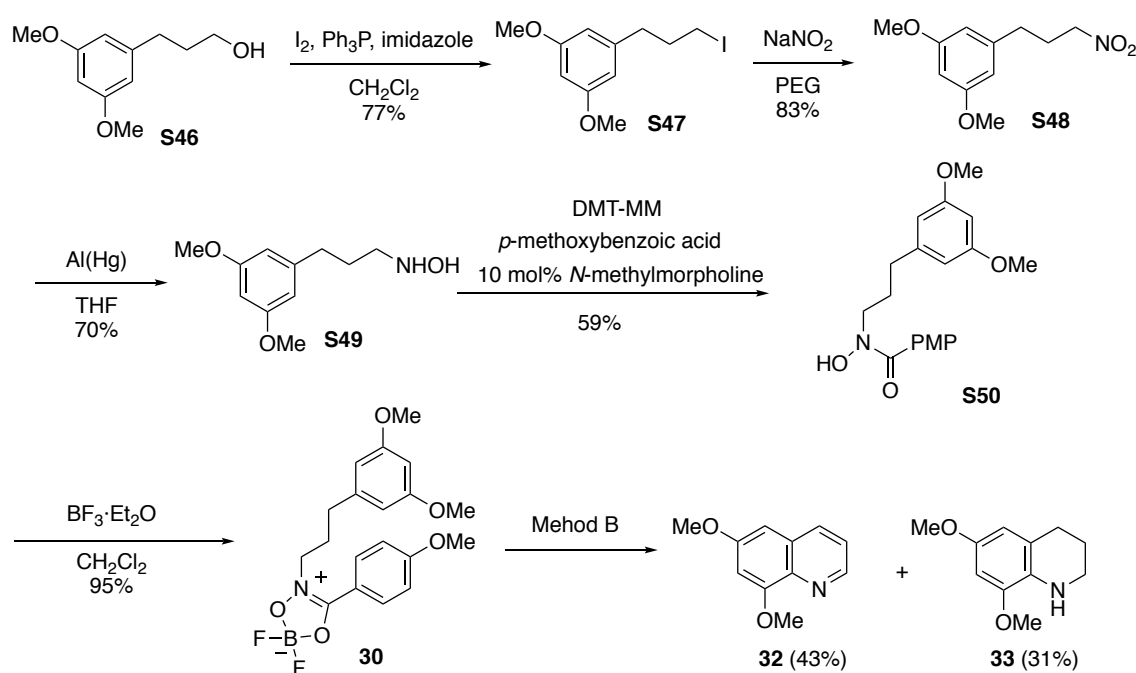
Boron complex S45;  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.01 (dt,  $J = 2.5, 9.0$  Hz, 2H), 6.82 (dt,  $J = 2.3, 8.5$  Hz, 2H), 6.30 (t,  $J = 2.3$  Hz, 1H), 6.26 (d,  $J = 2.5$  Hz, 2H), 4.37–4.44 (m, 1H), 3.83 (s, 3H), 3.71 (s, 6H), 3.21 (dd,  $J = 10.8, 13.8$  Hz, 1H), 2.71 (dd,  $J = 3.3, 13.8$  Hz, 1H), 1.60 (d,  $J = 6.0$  Hz, 3H);  $^{13}\text{C NMR}$  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  163.3, 162.3, 161.2, 138.8, 130.2, 114.5, 114.4, 106.6, 100.5, 60.2, 55.7, 55.5, 40.1,

18.4; IR (neat)  $\nu_{\max}$  3009, 2940, 2843, 1591, 1516, 1449, 1344, 1269, 1263, 1204, 1150, 1061, 991, 930, 843, 735, 706  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{19}\text{H}_{24}\text{NO}_5$   $[\text{M}-\text{BF}_2+\text{H}_2]^+$ : 346.1649, found: 346.1625; mp: 133–135 °C.

Indole formation using **S47** was carried out with general protocol (method B; Time A: 10 min; Time B: 24 h), and desired indole **29** was obtained in 82% yield.

**5,7-Dimethoxy-2-methyl-1*H*-indole (29)**;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.97 (br s, 1H), 6.59 (d,  $J = 2.0$  Hz, 1H), 6.29 (d,  $J = 2.0$  Hz, 1H), 6.12 (s, 1H), 3.91 (s, 3H), 3.83 (s, 3H), 2.41 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  155.0, 145.9, 135.1, 129.7, 121.5, 100.8, 93.5, 93.3, 56.0, 55.5, 13.8; IR (neat)  $\nu_{\max}$  3323, 3003, 2934, 2835, 1630, 1587, 1582, 1499, 1454, 1402, 1321, 1246, 1204, 1140, 1109, 1043, 991, 930, 812, 785, 743, 718  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{11}\text{H}_{14}\text{NO}_2$   $[\text{M}+\text{H}]^+$ : 192.1019, found: 192.1009; mp: 96–97.8 °C.

#### Synthesis of 6,8-dimethoxyquinoline (**32**) and 6,8-dimethoxy-1,2,3,4-tetrahydroquinoline (**33**).



To a solution of iodine (25.0 g, 9.94 mmol) in  $\text{CH}_2\text{Cl}_2$  (40 mL),  $\text{Ph}_3\text{P}$  (26.0 g, 9.94 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 10 min. To the resulting mixture, imidazole (1.1 g, 16.6 mmol) and a solution of 3-(3,5-dimethoxyphenyl)propan-1-ol (**S46**, 1.3 g, 6.63 mmol) in  $\text{CH}_2\text{Cl}_2$  (40 mL) were slowly added. The reaction mixture was stirred for 1 h at room temperature. The resulting mixture was quenched with saturated aqueous  $\text{Na}_2\text{S}_2\text{O}_5$ . The aqueous layer was extracted three times with  $\text{CHCl}_3$ . The combined organic layer was washed with saturated aqueous

NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 10% EtOAc/*n*-hexanes) provided **S47** (5.57 g, 77%) as a pale yellow oil.

1-(3-Iodopropyl)-3,5-dimethoxybenzene (S47); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 6.37 (d, *J* = 2.5 Hz, 2H), 6.33 (t, *J* = 2.0 Hz, 1H), 3.79 (s, 6H), 3.17 (t, *J* = 7.0 Hz, 2H), 2.67 (t, *J* = 7.0 Hz, 2H), 2.12 (quint, *J* = 7.0 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 160.9, 142.8, 106.7, 98.1, 55.4, 36.5, 34.7, 6.6; IR (neat)  $\nu_{\max}$  2936, 2835, 1593, 1458, 1427, 1350, 1292, 1204, 1148, 1059, 922, 829, 694 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>11</sub>H<sub>16</sub>IO<sub>2</sub> [M+H]<sup>+</sup>: 307.0190, found: 307.0172.

To a solution of known compound **S47** (1.50 g, 4.90 mmol) in polyethylene glycol (10 mL), NaNO<sub>2</sub> (1.0 g, 14.7 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 3 h at room temperature before an addition of H<sub>2</sub>O (50 mL). The aqueous layer was extracted three times with Et<sub>2</sub>O. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 10% EtOAc/*n*-hexanes) provided nitro compound **S48** (916.0 mg, 83%) as a pale yellow oil.

1,3-Dimethoxy-5-(3-nitropropyl)benzene (S48); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 6.32–6.34 (m, 3H), 4.37 (t, *J* = 6.8 Hz, 2H), 3.78 (s, 6H), 2.66 (t, *J* = 7.5 Hz, 2H), 2.31 (quint, *J* = 7.3 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 161.1, 141.9, 106.6, 98.5, 74.7, 55.4, 32.6, 28.7; IR (neat)  $\nu_{\max}$  2938, 2839, 1595, 1547, 1460, 1429, 1381, 1294, 1204, 1148, 1055, 924, 833, 696 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>11</sub>H<sub>16</sub>NO<sub>4</sub> [M+H]<sup>+</sup>: 226.1074, found: 226.1061.

To a solution of **S48** (900.0 mg, 4.0 mmol) in THF (160 mL) and H<sub>2</sub>O (16 mL), freshly prepared Al(Hg) (from 1.8 g of Al) was added at room temperature under Ar atmosphere. The reaction mixture was vigorously stirred for 8 h at room temperature before being quenched with saturated aqueous NaHCO<sub>3</sub>. The resulting mixture was filtrated by Celite pad. The filtrate was extracted three times with CHCl<sub>3</sub>. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub> and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 20–50% CH<sub>3</sub>CN/toluene gradient) provided **S49** (589.0 mg, 70%) as a colorless oil.

*N*-(3-(3,5-Dimethoxyphenyl)propyl)hydroxylamine (S49); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 6.35 (d, *J* = 2.5 Hz, 2H), 6.30 (t, *J* = 2.0 Hz, 1H), 3.77 (s, 6H), 2.96 (t, *J* = 7.0 Hz, 2H), 2.61 (t, *J* = 7.8 Hz, 2H), 1.87 (quint, *J* = 7.5 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 160.9, 144.4, 106.6, 97.9, 55.4, 53.4, 33.8, 28.6; IR (neat)  $\nu_{\max}$  3260, 2932, 2883, 1593, 1462, 1427, 1352, 1294, 1206, 1148, 1055, 993, 922, 831 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>11</sub>H<sub>18</sub>NO<sub>3</sub> [M+H]<sup>+</sup>: 212.1281, found: 212.1270.

To a solution of *p*-methoxybenzoic acid (418.0 mg, 2.75 mmol) and *N*-methylmorpholine (NMM, 30.0  $\mu$ L, 0.275 mmol) in CH<sub>3</sub>CN (55 mL), 4-(4,6-dimethoxy-1,3,5-triazin-2-yl)-4-methylmorpholium chloride (DMT-MM, 798.0 mg, 2.89 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 3 h at room temperature. The resulting mixture was cooled to  $-20$  °C before an addition of the solution of hydroxylamine **S49** (580.0 mg, 1.02 mmol) in CH<sub>3</sub>CN (55 mL). The reaction mixture was stirred for 1 h at  $-20$  °C under Ar atmosphere. The resulting mixture was quenched with excess amount of 1M HCl/H<sub>2</sub>O at  $-20$  °C. The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. Flash chromatography (SiO<sub>2</sub>, 2% CH<sub>3</sub>CN/CHCl<sub>3</sub>) provided *N*-hydroxyamide **S50** (557 mg, 59%) as a white solid.

*N*-(3-(3,5-Dimethoxyphenyl)propyl)-*N*-hydroxy-4-methoxybenzamide (**S50**); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.56 (br s, 1H), 7.43 (d,  $J$  = 9.0 Hz, 1H), 6.88 (dt,  $J$  = 2.4, 9.5 Hz, 2H), 6.29 (t,  $J$  = 2.0 Hz, 1H), 6.27 (d,  $J$  = 2.5 Hz, 2H), 3.84 (s, 3H), 3.75 (s, 6H), 3.69 (t,  $J$  = 7.3 Hz, 2H), 2.55 (t,  $J$  = 7.5 Hz, 2H), 2.07 (quint,  $J$  = 7.4 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  167.6, 161.8, 161.0, 143.3, 129.8, 124.2, 114.0, 106.5, 98.1, 55.5, 55.4, 50.8, 33.0, 28.9; IR (neat)  $\nu_{\max}$  3190, 2936, 2841, 1589, 1518, 1454, 1412, 1310, 1252, 1159, 1069, 1024, 939, 827, 760, 700, 667 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>19</sub>H<sub>24</sub>NO<sub>5</sub> [M+H]<sup>+</sup>: 346.1649, found: 346.1626; mp: 123–126 °C.

To a solution of *N*-hydroxyamide **S50** (550.0 mg, 0.159 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (53 mL), BF<sub>3</sub>·Et<sub>2</sub>O (393.0  $\mu$ L, 3.18 mmol) was added at room temperature under Ar atmosphere. The reaction mixture was stirred for 1 h at room temperature. Excess BF<sub>3</sub>·Et<sub>2</sub>O and the solvent were removed from the reaction vessel under reduced pressure. Obtained white solids were collected by filtration and washed with cold Et<sub>2</sub>O. Desired pure boron complex **30** (592.0 mg, 95%) was obtained as white crystals.

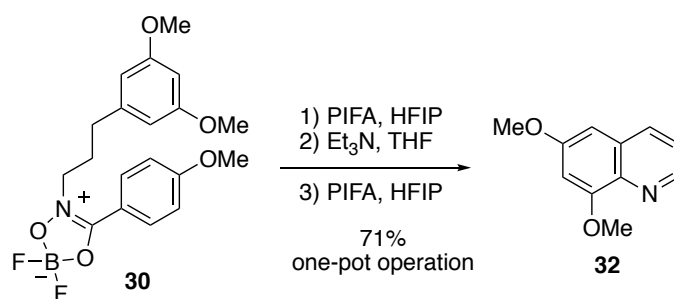
Boron complex **30**; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.50 (dt,  $J$  = 2.3, 8.5 Hz, 2H), 6.95 (dt,  $J$  = 2.8, 9.5 Hz, 2H), 6.30 (t,  $J$  = 2.3 Hz, 1H), 6.27 (d,  $J$  = 2.5 Hz, 2H), 3.94 (t,  $J$  = 7.0 Hz, 2H), 3.89 (s, 3H), 3.75 (s, 6H), 2.67 (t,  $J$  = 7.3 Hz, 2H), 2.29 (quint,  $J$  = 7.1 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  164.0, 161.5, 161.2, 142.0, 130.9, 114.9, 114.0, 106.6, 98.4, 55.8, 55.4, 50.0, 32.3, 28.1; IR (neat)  $\nu_{\max}$  2967, 2934, 2843, 1605, 1456, 1302, 1263, 1157, 1121, 1061, 982, 937, 881, 837, 768, 737 cm<sup>-1</sup>; HRMS (ESI) calculated for C<sub>19</sub>H<sub>24</sub>NO<sub>5</sub> [M–BF<sub>2</sub>+H<sub>2</sub>]<sup>+</sup>: 346.1649, found: 346.1626; mp: 112–113 °C.

Indole formation using **30** was carried out with general protocol (method B; Time A: 10 min; Time B: 3.5 h), and desired 6,8-dimethoxyquinoline (**32**) and 6,8-dimethoxy-1,2,3,4-tetrahydroquinoline (**33**) were obtained in 43% and 31% yield respectively.

6,8-Dimethoxyquinoline (32);  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.75 (dd,  $J = 2.0, 4.0$  Hz, 1H), 8.00 (dd,  $J = 1.8, 8.8$  Hz, 1H), 7.36 (dd,  $J = 4.3, 8.3$  Hz, 1H), 6.71 (d,  $J = 3.0$  Hz, 1H), 6.65 (d,  $J = 2.5$  Hz, 1H), 4.05 (s, 3H), 3.92 (s, 3H);  $^{13}\text{C NMR}$  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  158.4, 156.5, 146.9, 137.1, 134.8, 130.1, 122.3, 101.3, 97.0, 56.2, 55.7; IR (neat)  $\nu_{\text{max}}$  3013, 2932, 2849, 1618, 1580, 1504, 1452, 1422, 1381, 1335, 1261, 1217, 1123, 1121, 1051, 997, 943, 891, 831, 791  $\text{cm}^{-1}$ ; HRMS (ESI) calculated for  $\text{C}_{11}\text{H}_{12}\text{NO}_2$   $[\text{M}+\text{H}]^+$ : 190.0863, found: 190.0852.

6,8-Dimethoxy-1,2,3,4-tetrahydroquinoline (33);  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.29 (d,  $J = 2.0$  Hz, 1H), 6.18 (d,  $J = 2.5$  Hz, 1H), 3.80 (s, 3H), 3.74 (s, 3H), 3.28 (t,  $J = 5.3$  Hz, 2H), 2.75 (t,  $J = 6.5$  Hz, 2H), 1.95 (quint,  $J = 6.0$  Hz, 2H);  $^{13}\text{C NMR}$  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  151.6, 147.7, 128.8, 122.0, 104.9, 97.0, 55.9, 55.5, 41.9, 27.1, 22.7; HRMS (ESI) calculated for  $\text{C}_{11}\text{H}_{12}\text{NO}_2$   $[\text{M}+\text{H}]^+$ : 194.1176, found: 194.1176.

#### One-Pot synthesis of 6,8-dimethoxyquinoline (32).

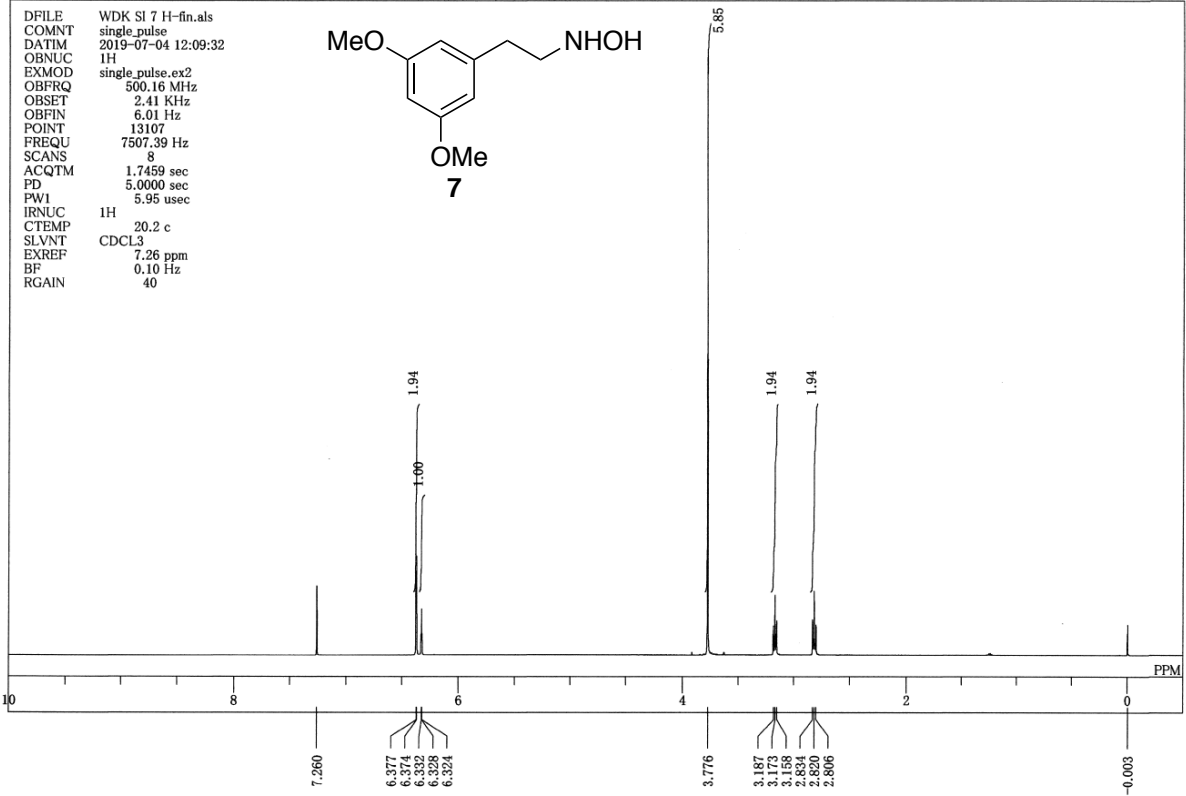


To a solution of boron complex **30** (20.0 mg, 0.0509 mmol) in 1,1,1,3,3,3-hexafluoroisopropanol (HFIP, 3.4 mL), [bis(trifluoroacetoxy)iodo]benzene (PIFA, 21.9 mg, 0.0509 mmol) was added at 0 °C under Ar atmosphere. The reaction mixture was stirred for 15 min before removal of solvent. The residue was dissolved to dry THF (3.4 mL) and the resulting mixture was cooled at 0 °C.  $\text{Et}_3\text{N}$  (21.3  $\mu\text{L}$ , 0.153 mmol) was added to the resulting mixture and the reaction mixture was stirred for 9 h at 0 °C under Ar atmosphere. The  $\text{Et}_3\text{N}$  and solvent was removed from reaction vessel under reduced pressure. the crude mixture of **32** and **33** were dissolved in 1,1,1,3,3,3-hexafluoroisopropanol (HFIP, 3.4 mL), and the resulting mixture was cooled at 0 °C. [Bis(trifluoroacetoxy)iodo]benzene (PIFA, 21.9 mg, 0.0509 mmol) was added to the resulting mixture at 0 °C. The reaction mixture was stirred for 6 h at 0 °C under Ar atmosphere. The resulting mixture was quenched with 15%  $\text{NaOH}/\text{H}_2\text{O}$ . The aqueous layer was extracted three times with EtOAc. The combined organic layer was washed with saturated aqueous NaCl, dried over  $\text{MgSO}_4$ , and concentrated under reduced pressure. Flash chromatography ( $\text{SiO}_2$ , 66% EtOAc/*n*-hexanes) provided 6,8-dimethoxyquinoline (**32**, 6.8 mg, 71%) as a pale yellow oil.

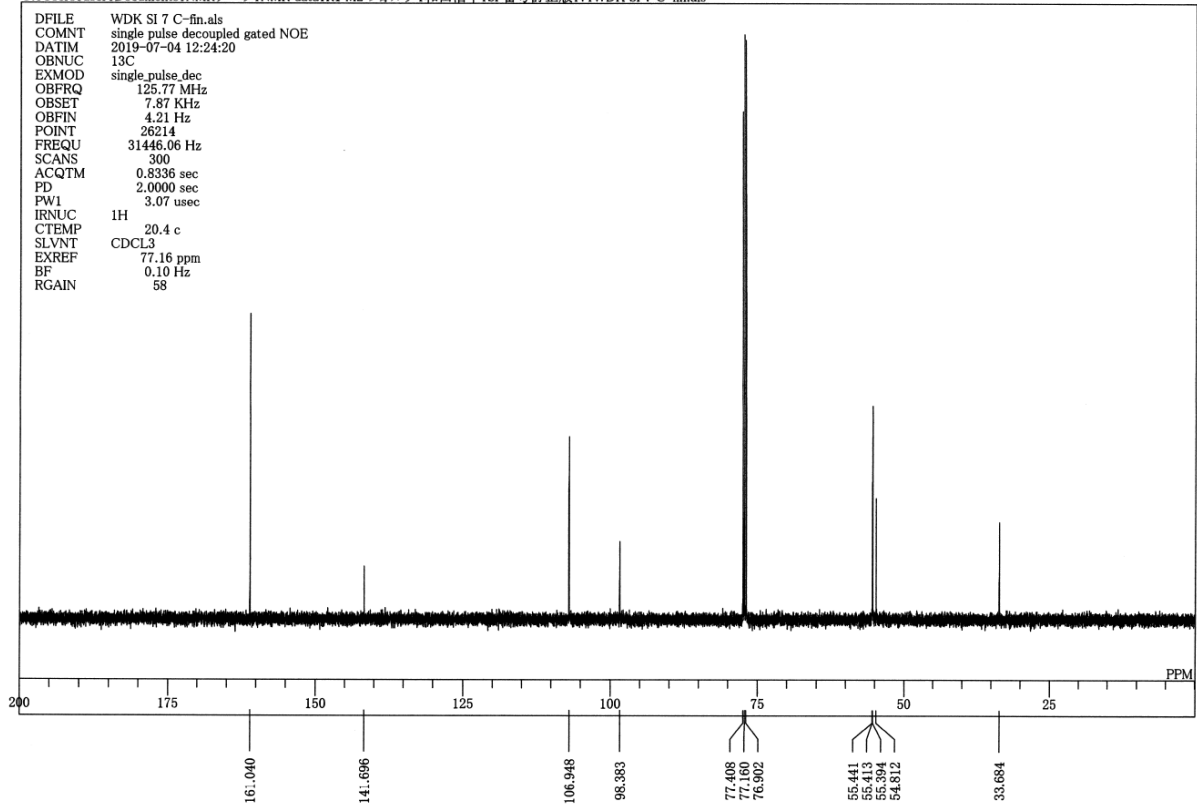
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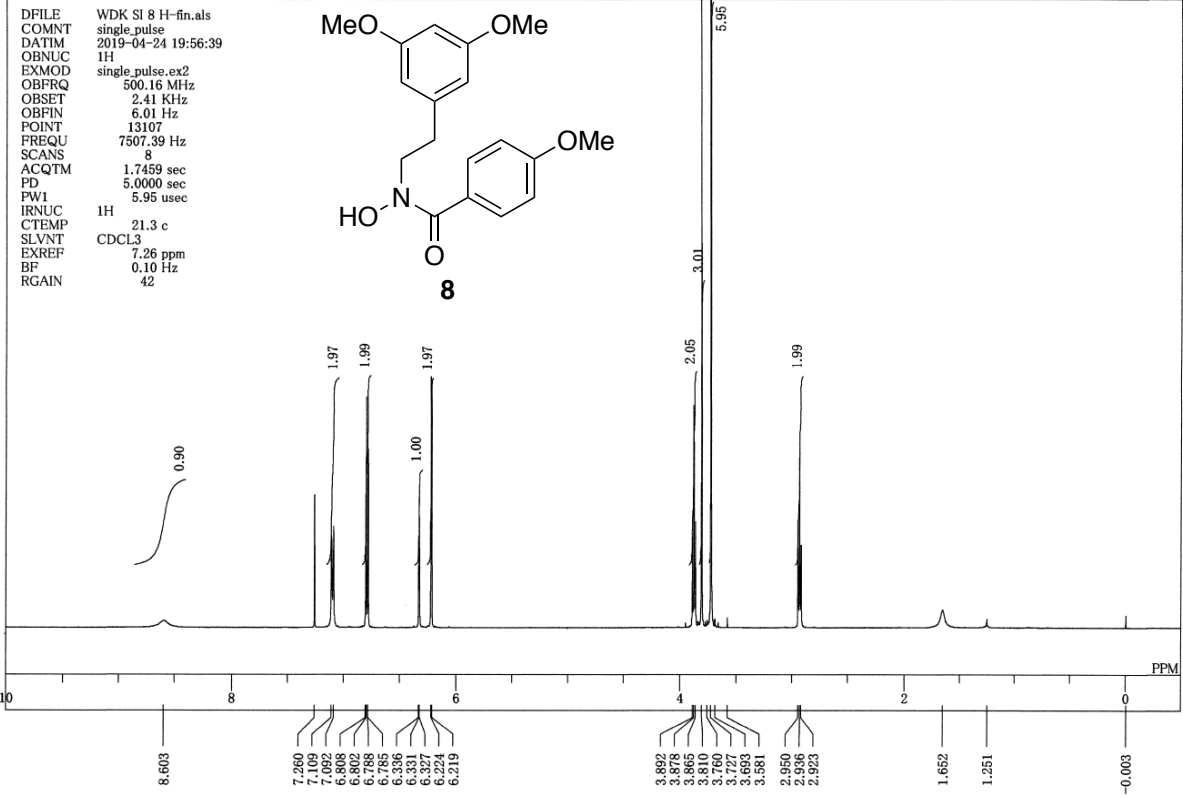
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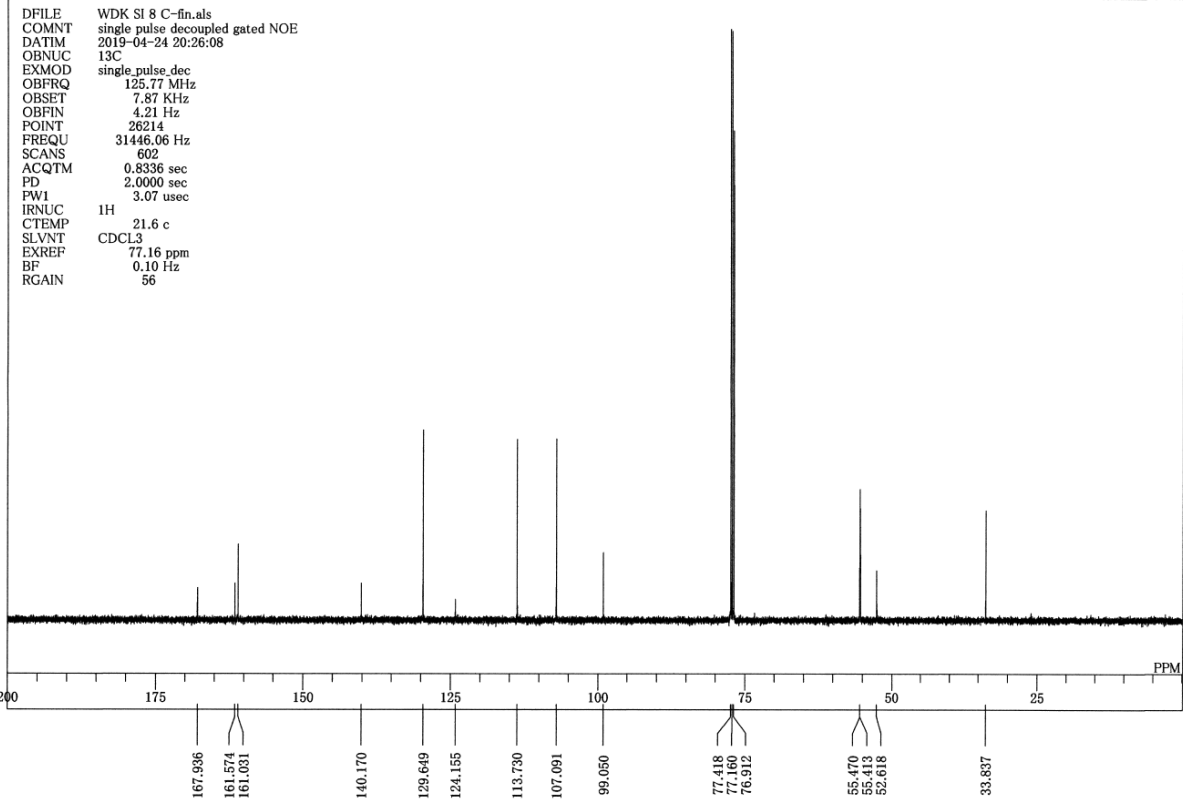
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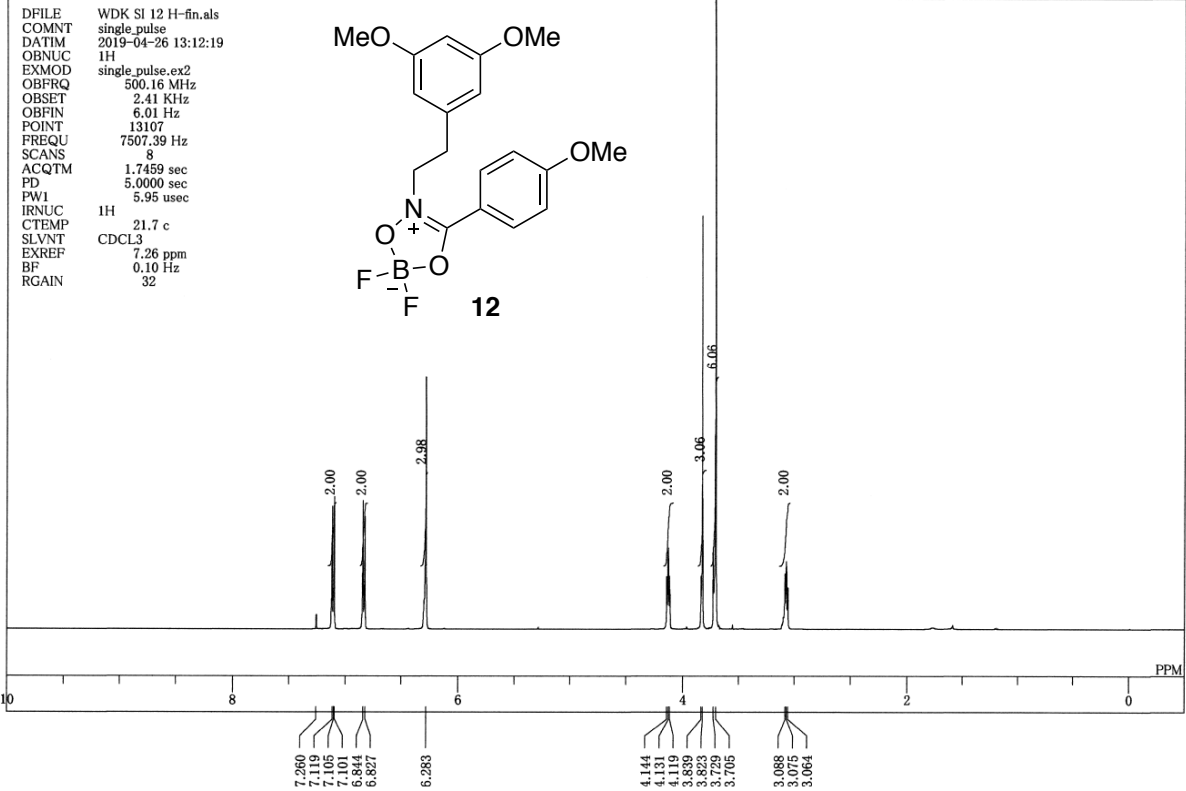
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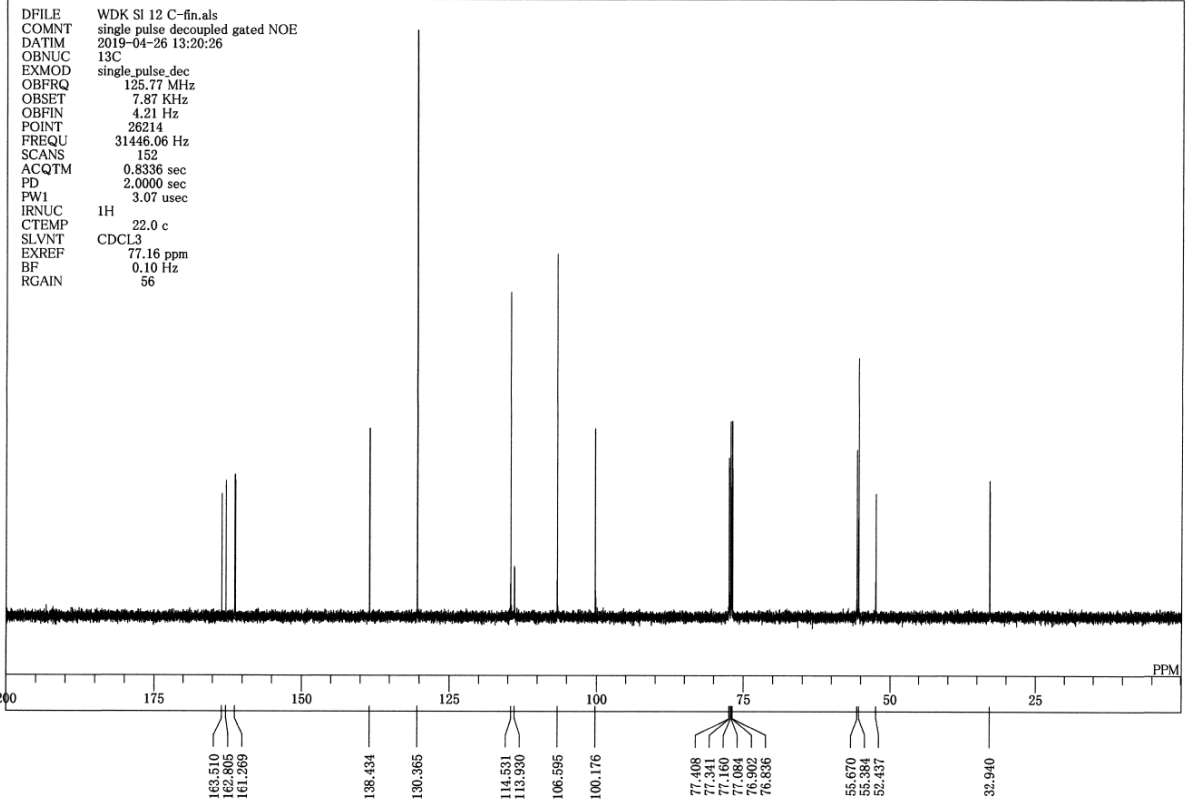
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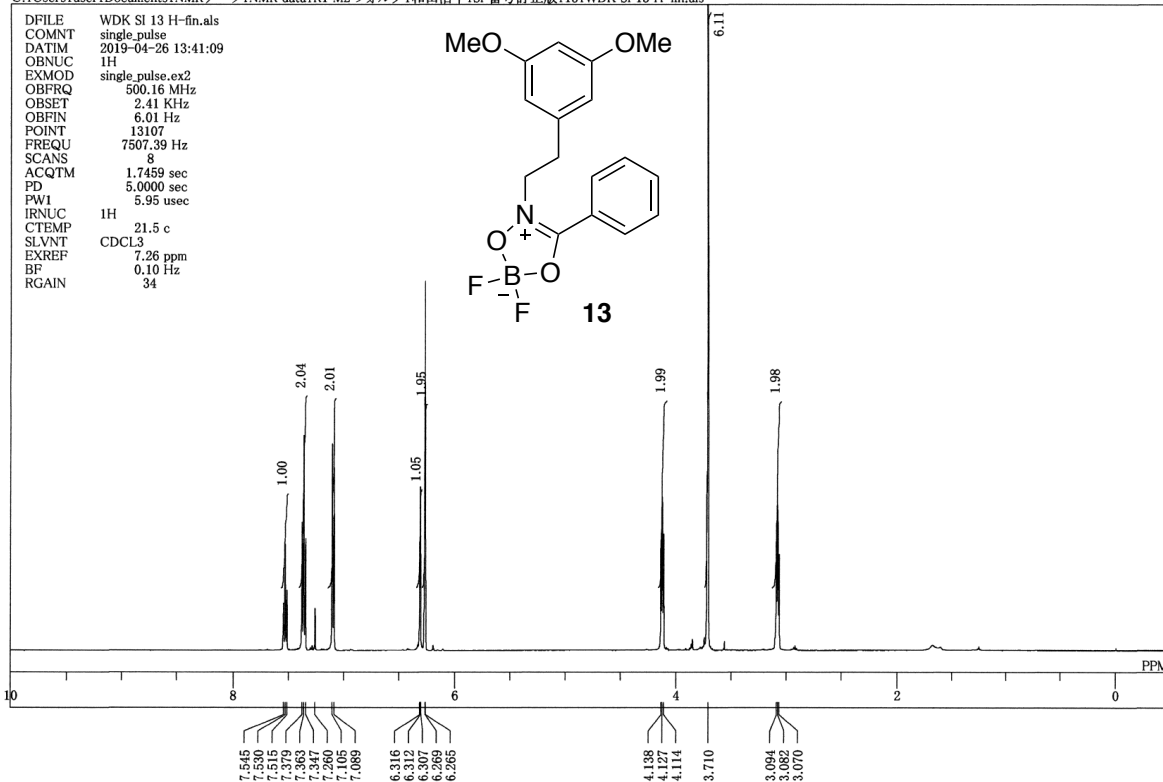
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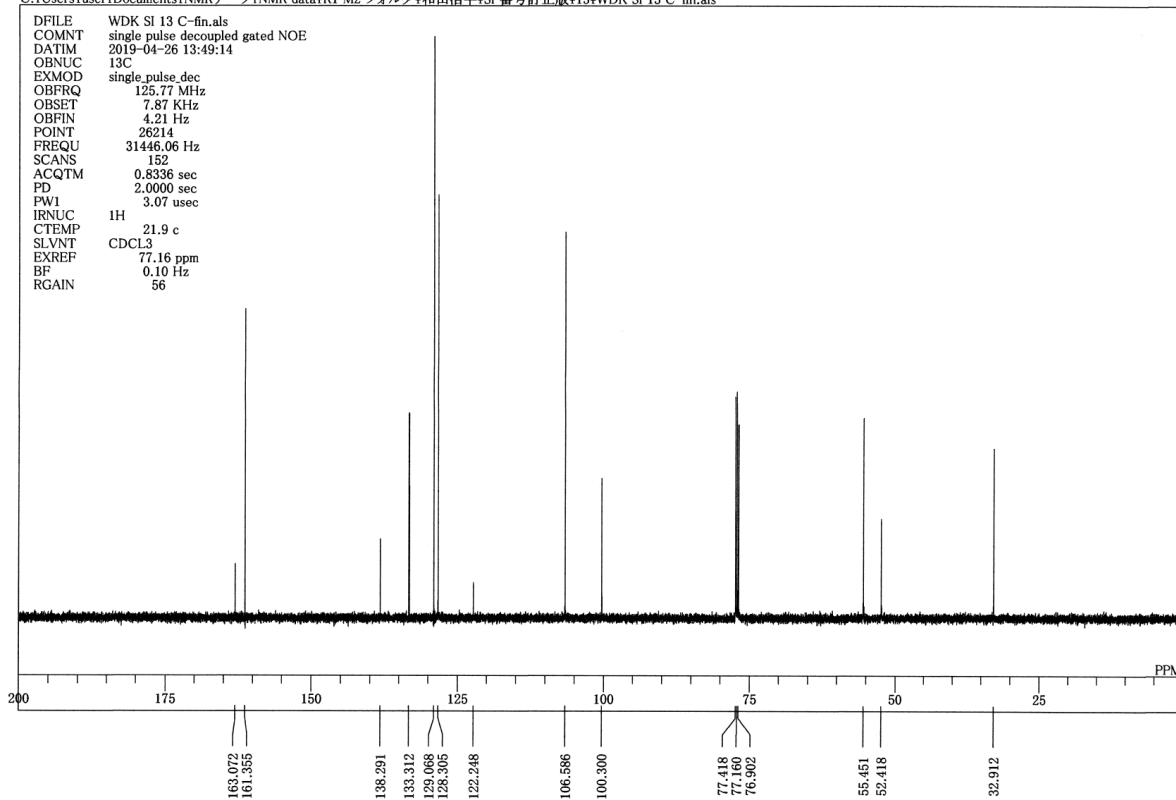
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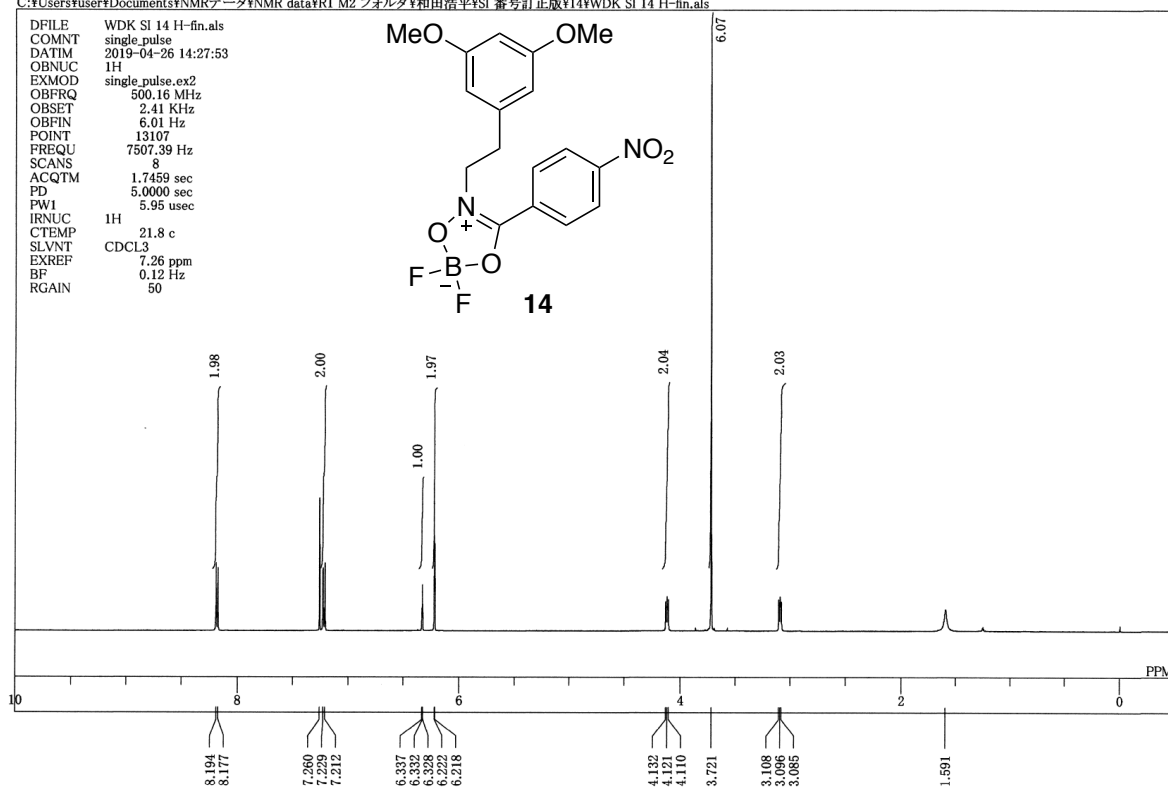
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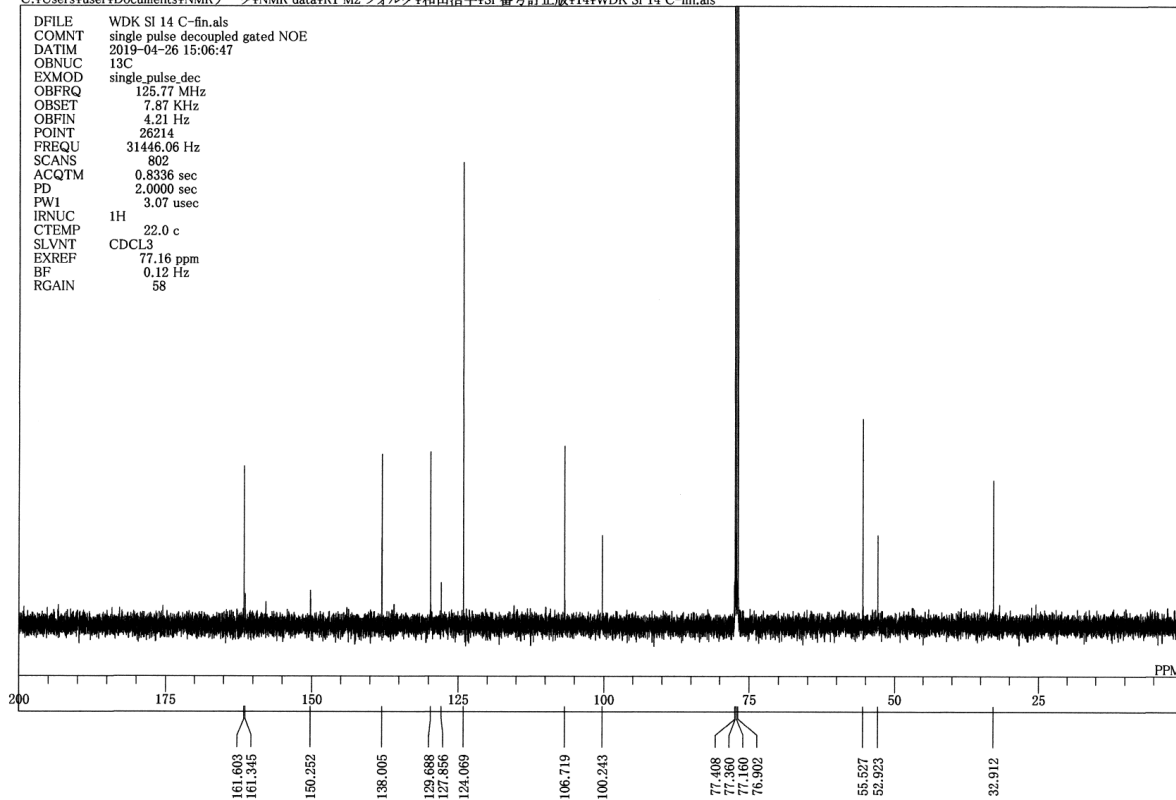
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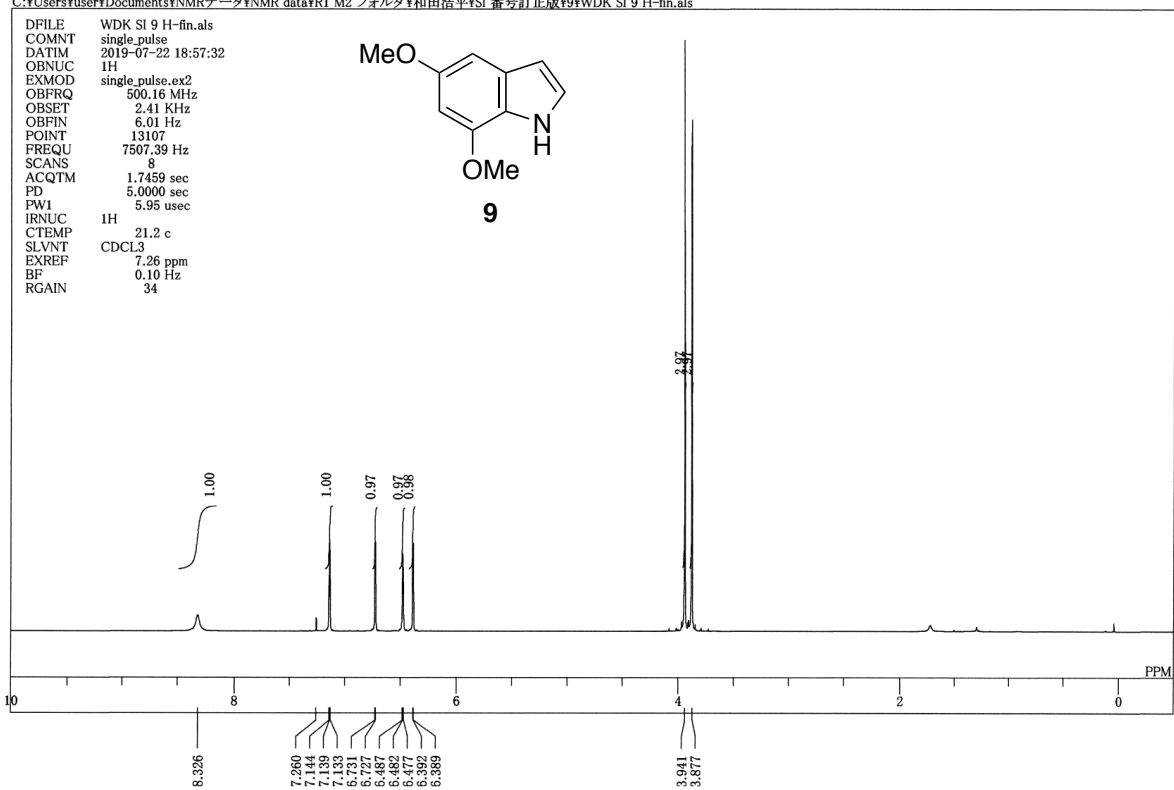
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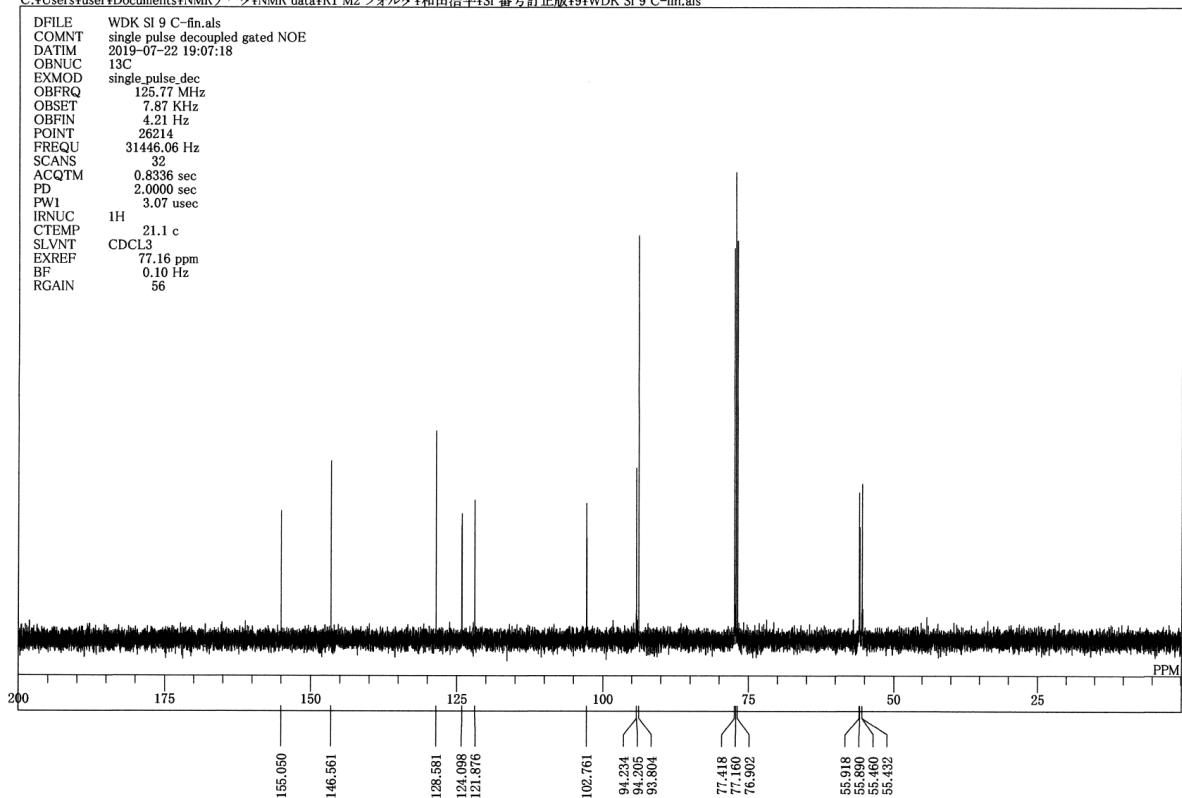
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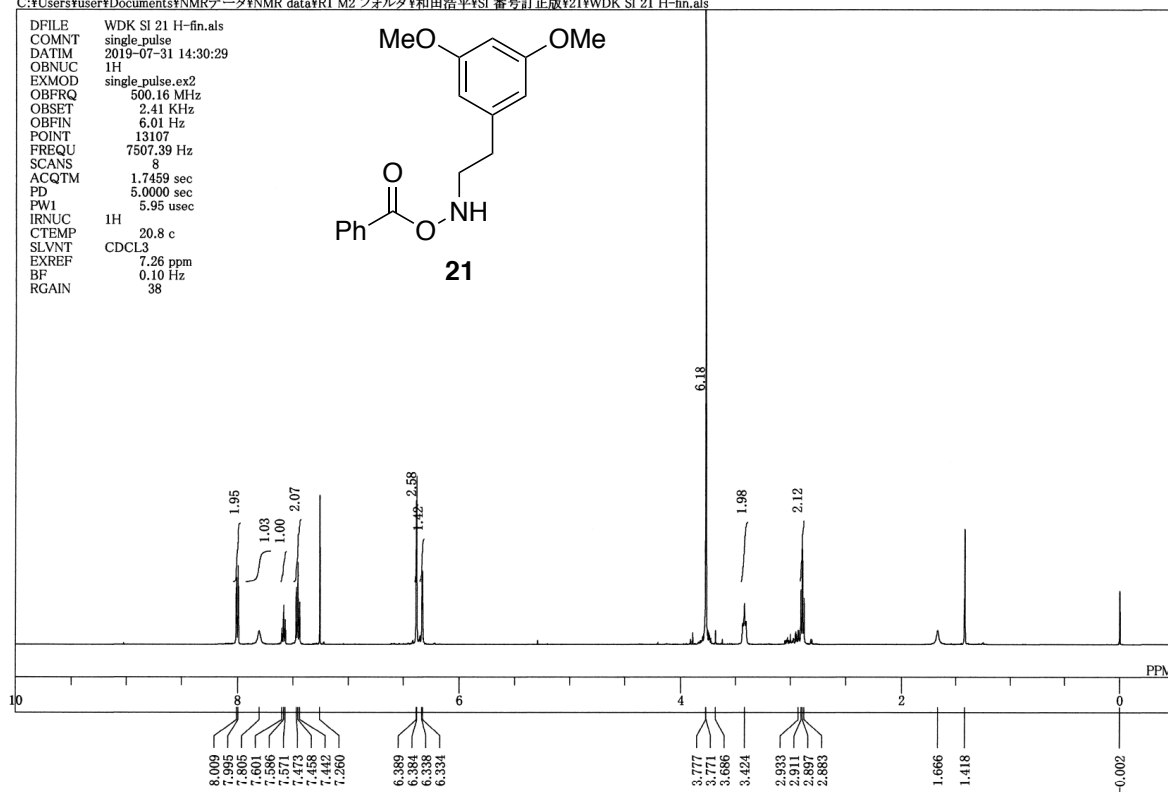
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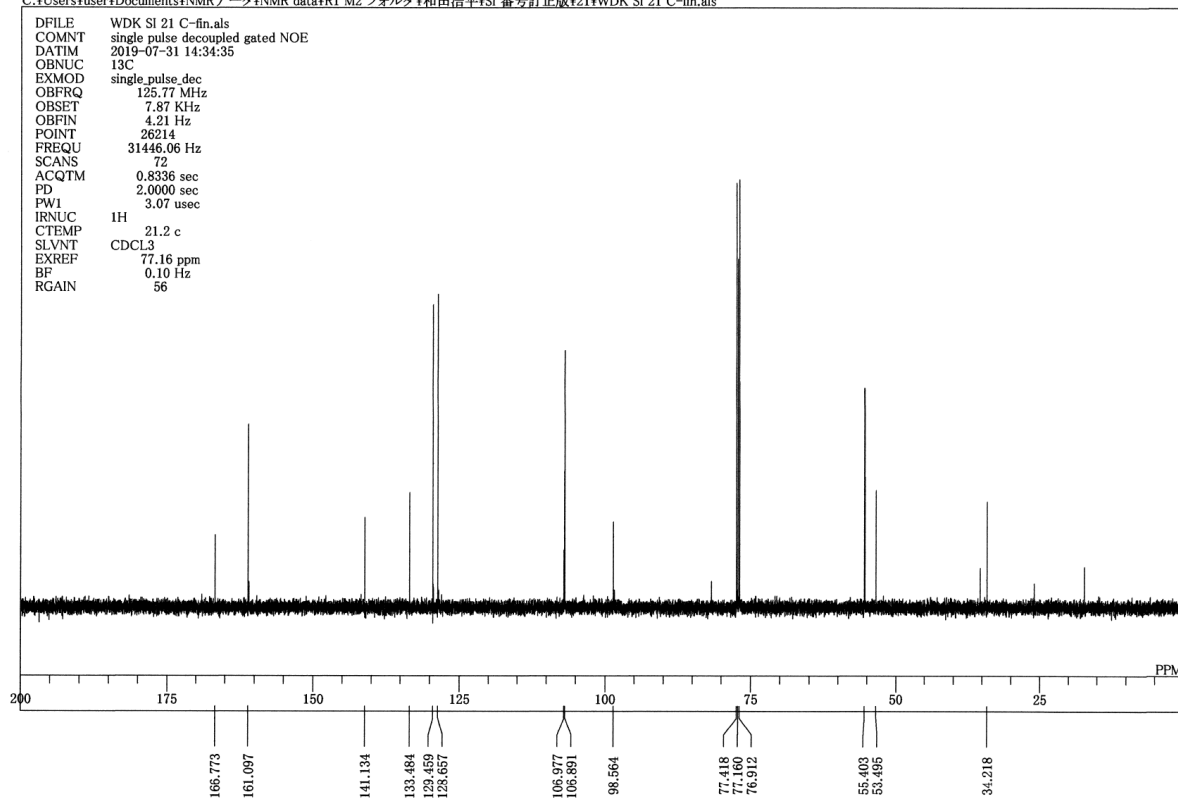
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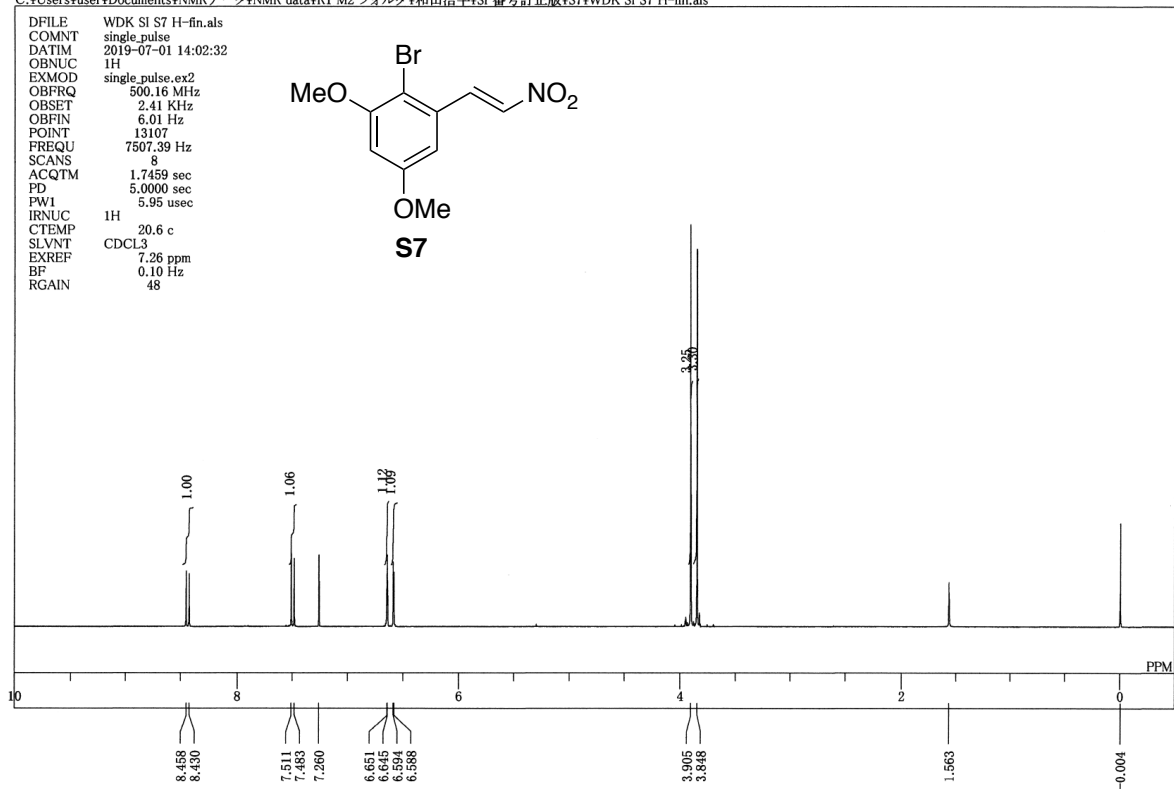
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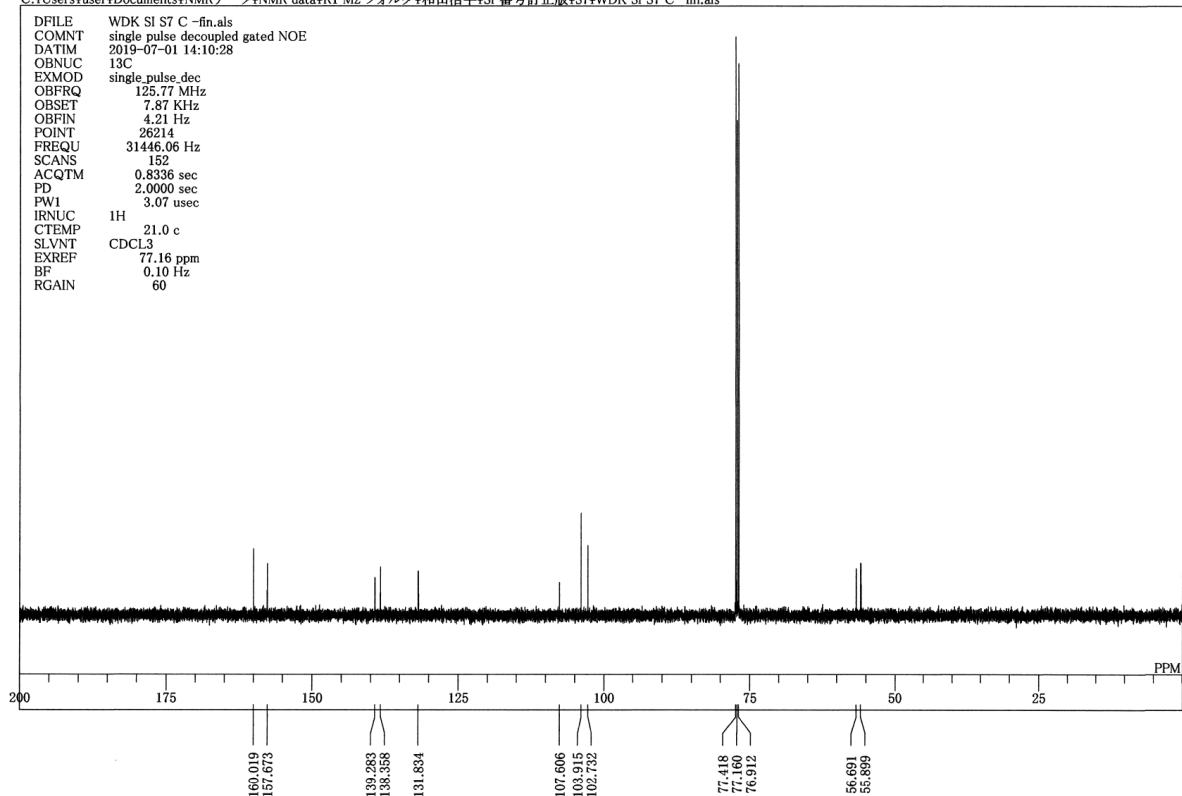
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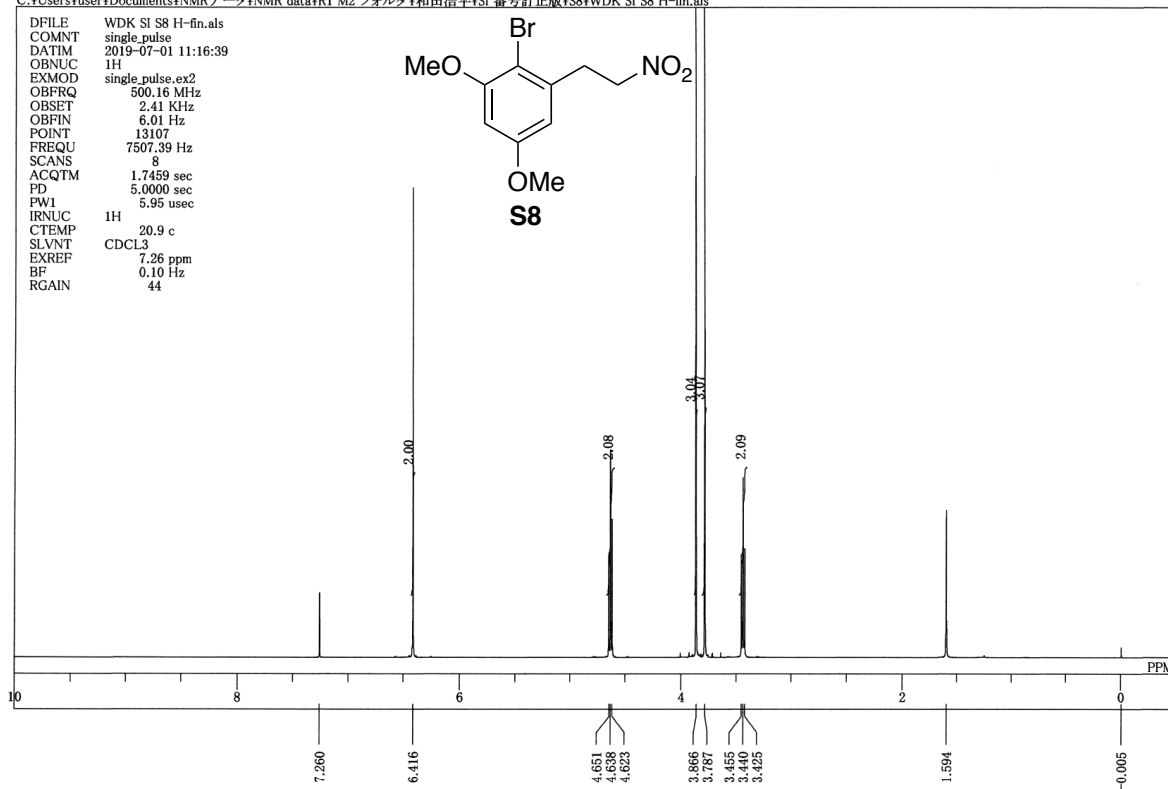
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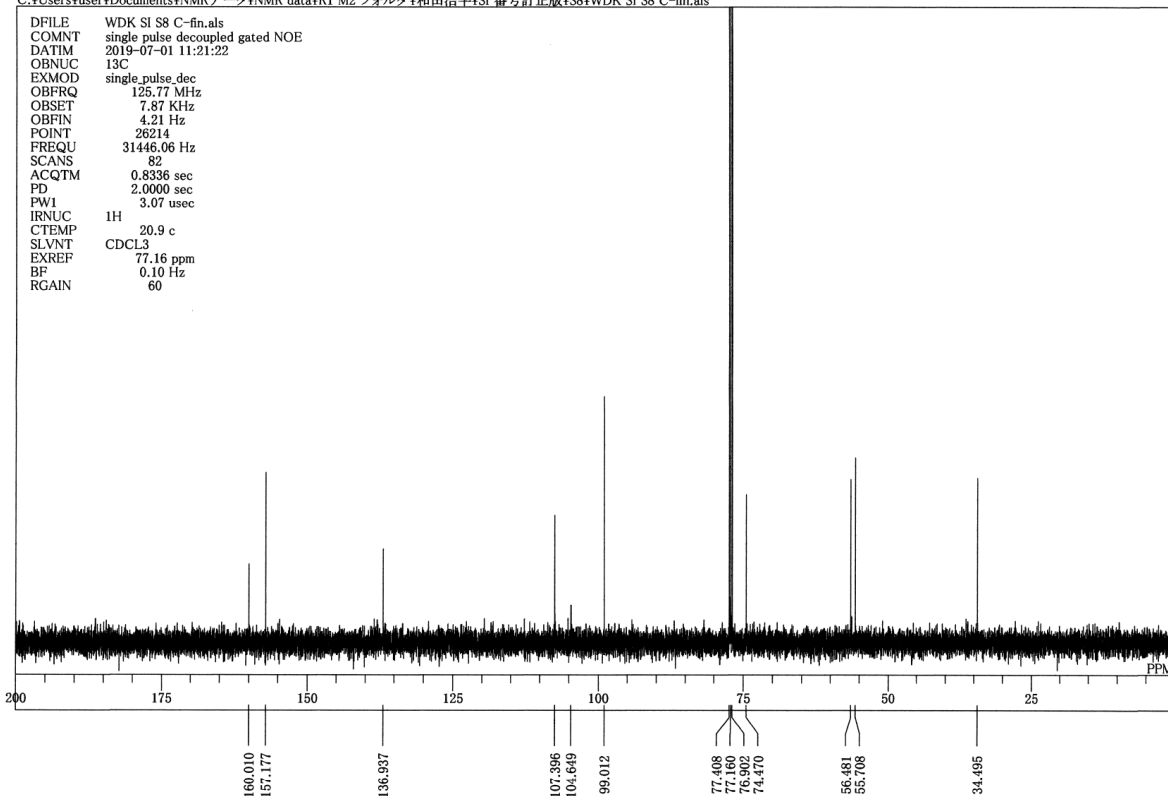
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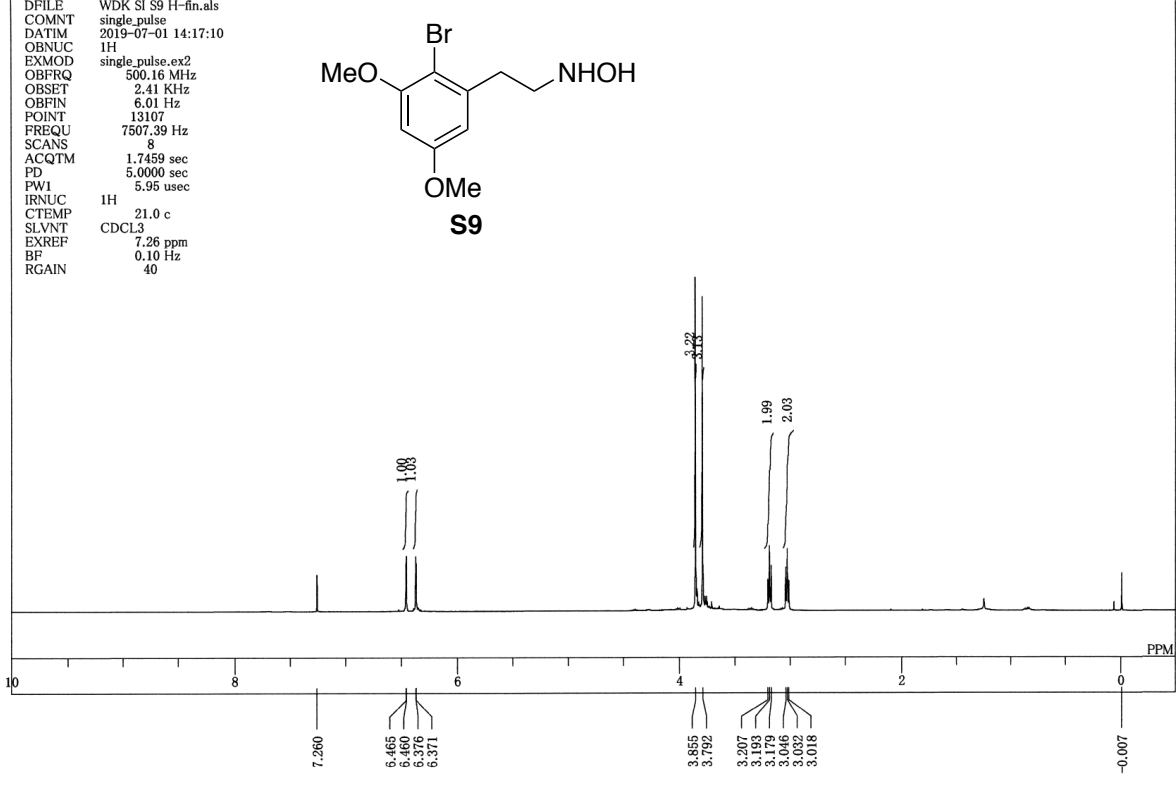
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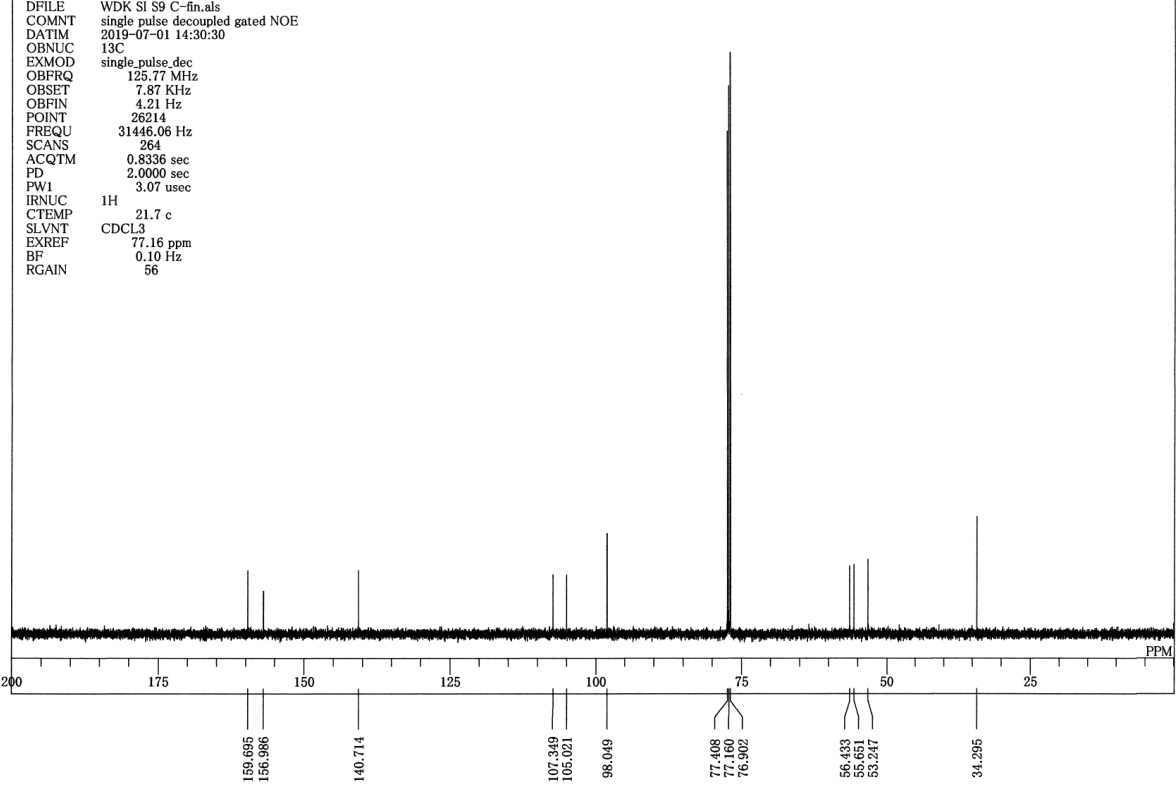
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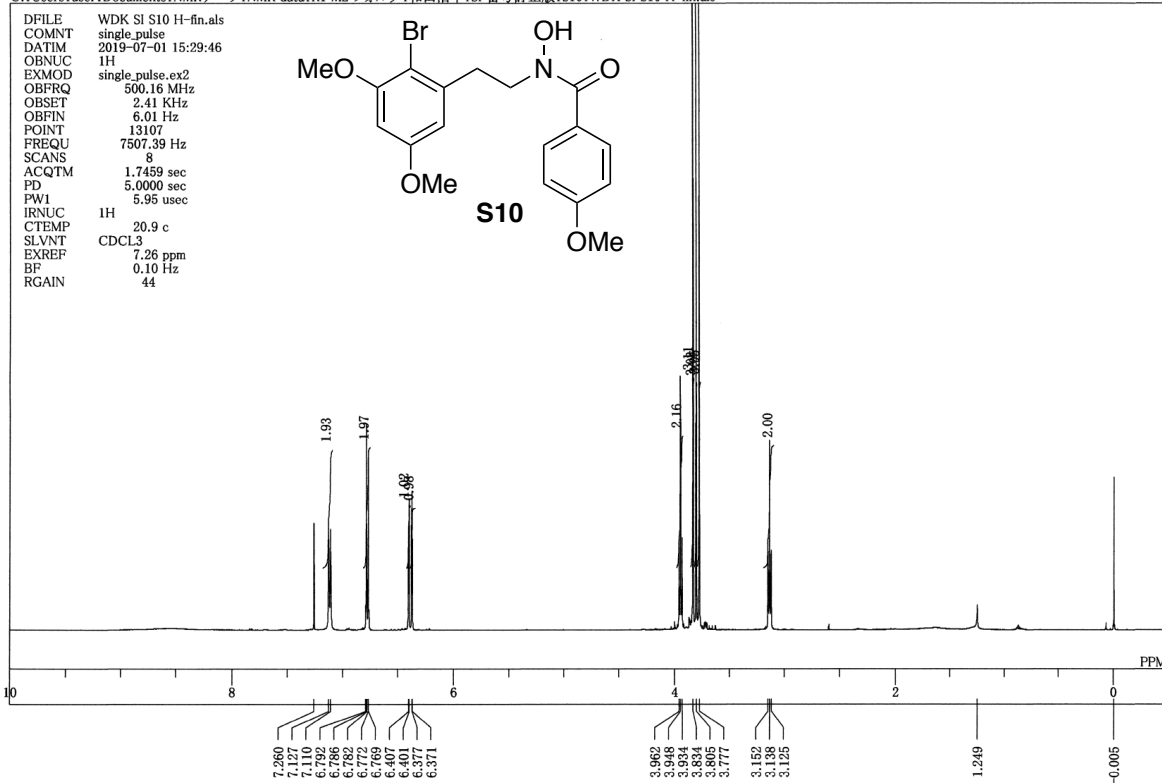
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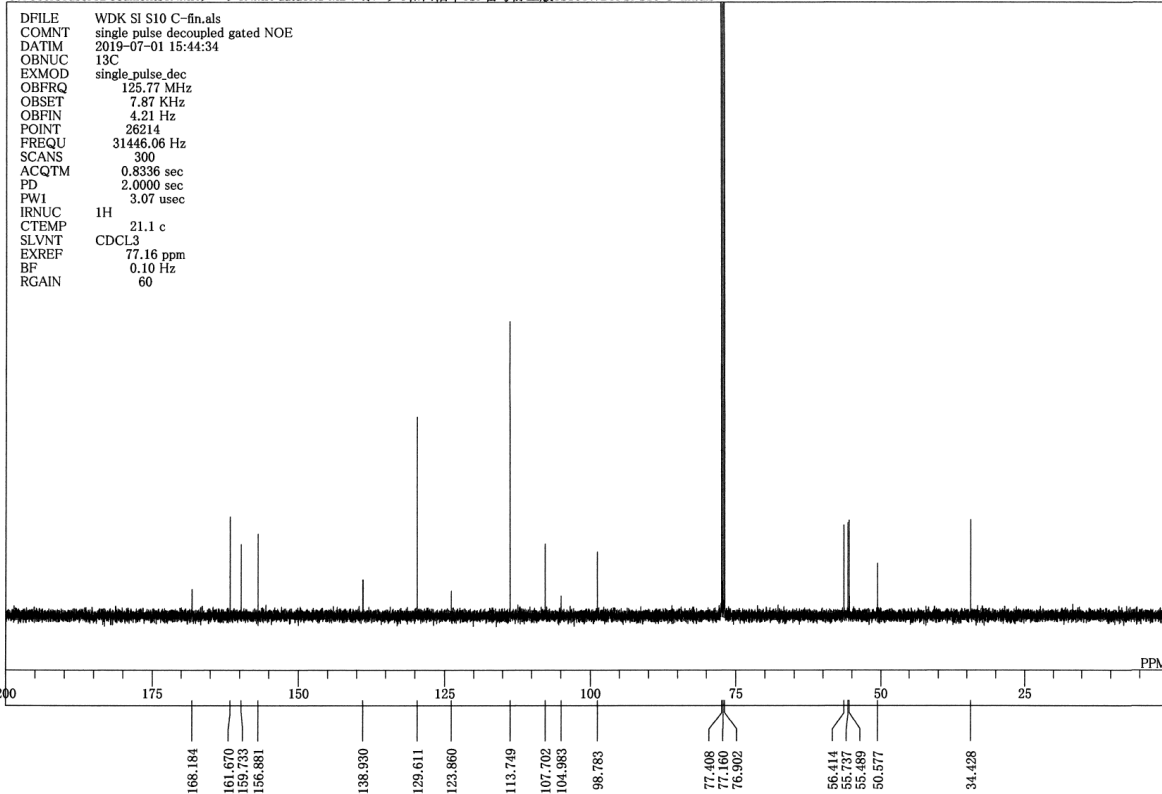
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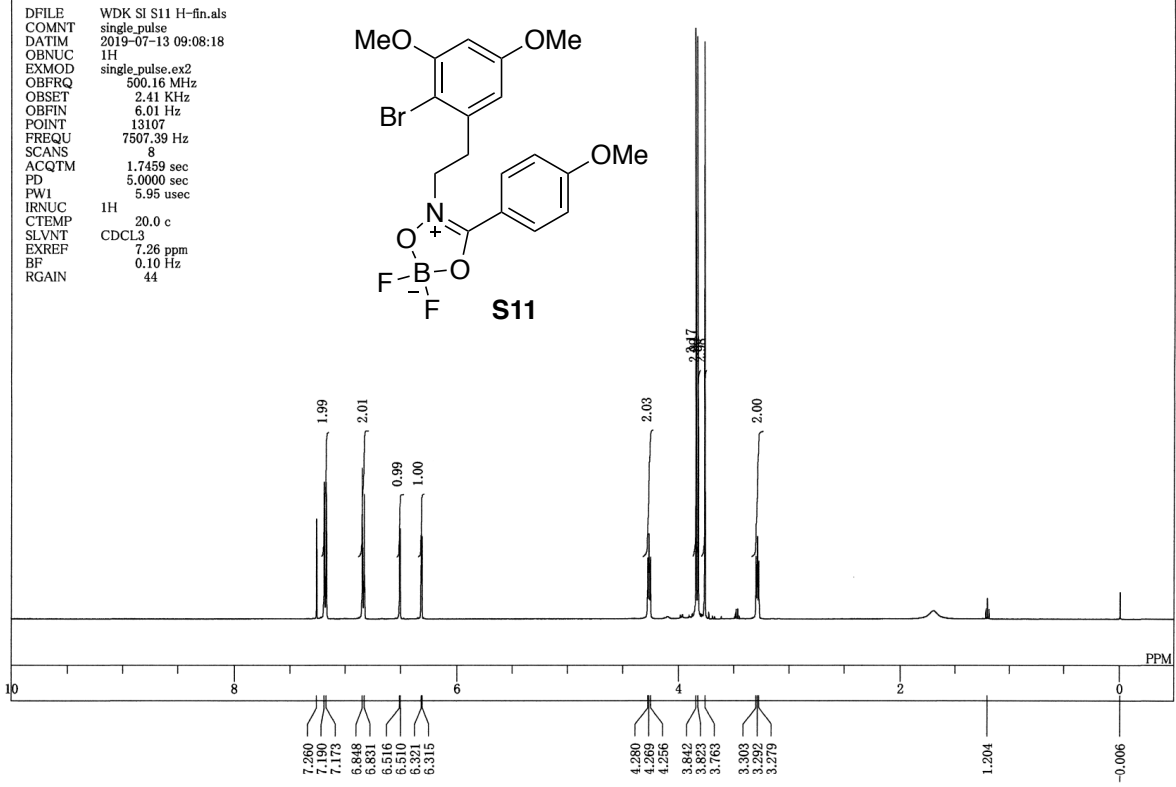
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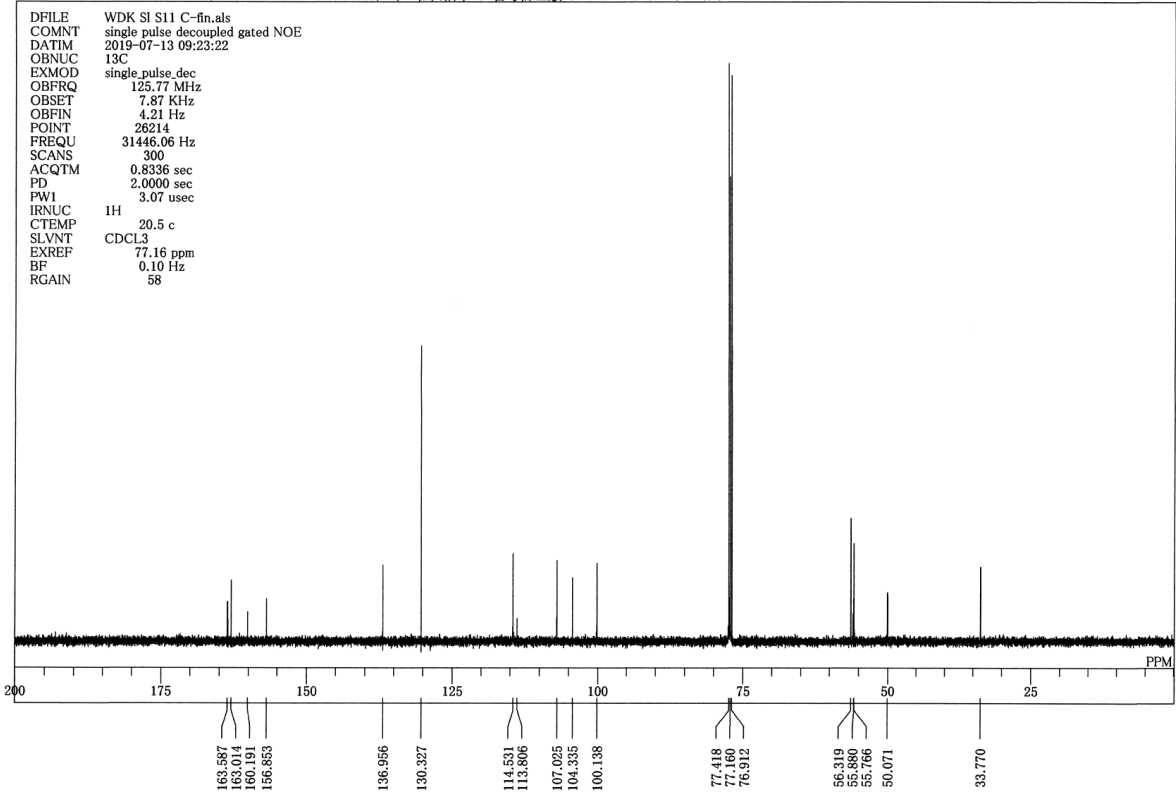
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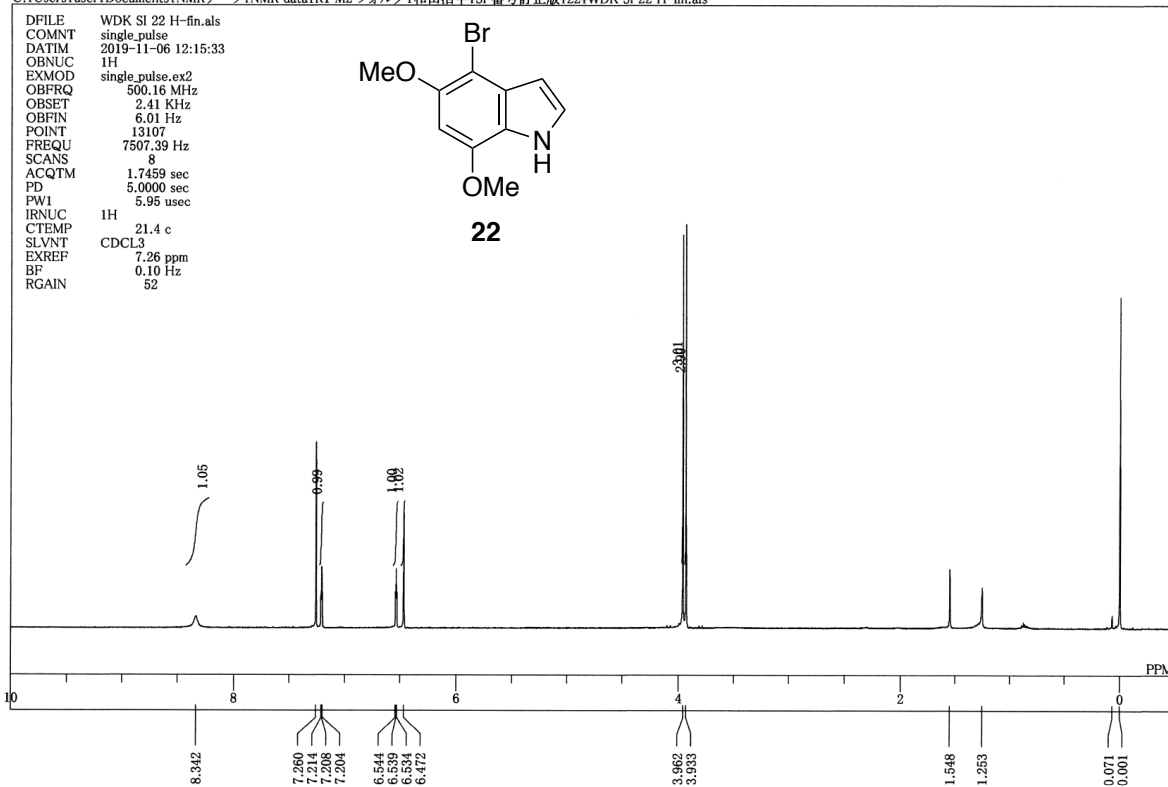
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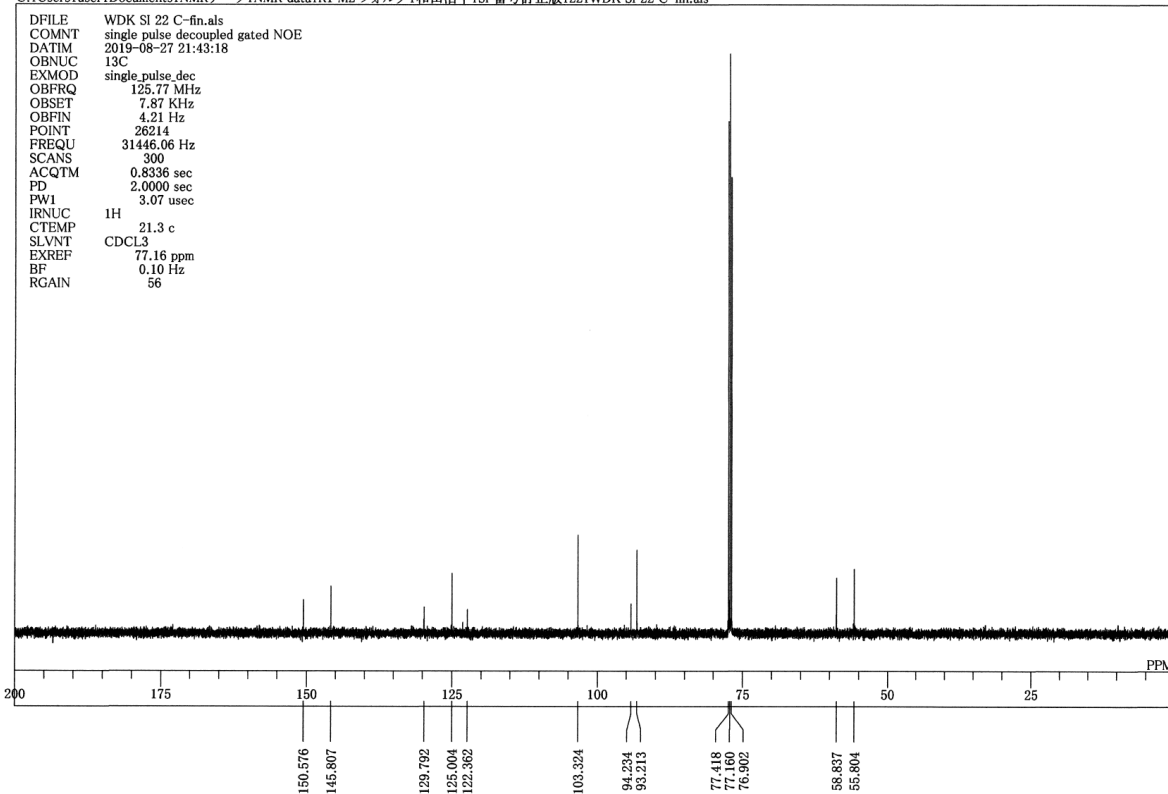
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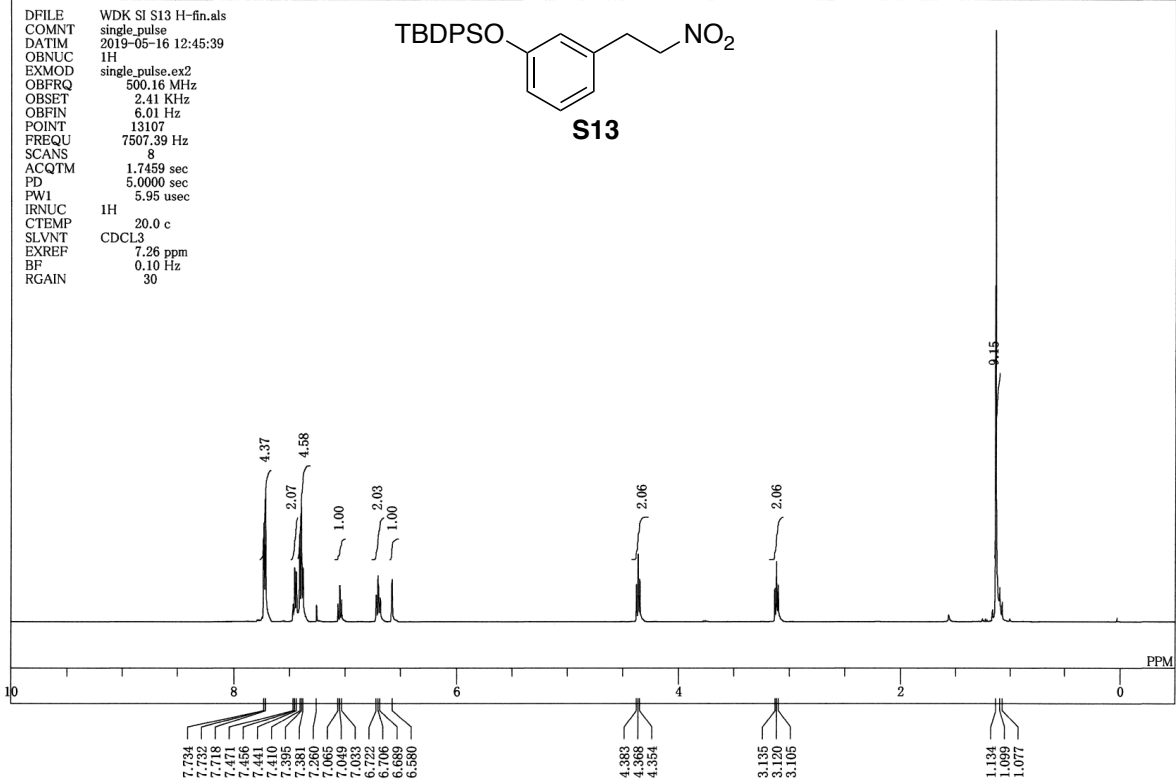
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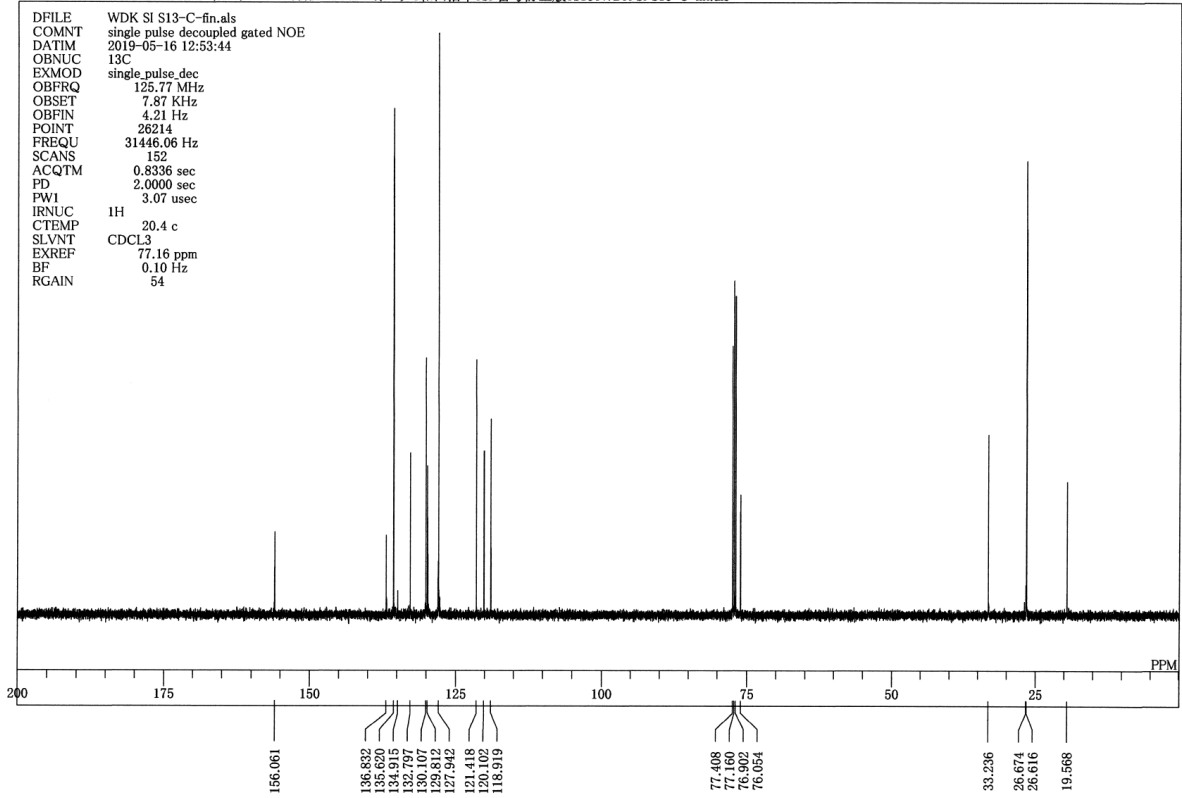
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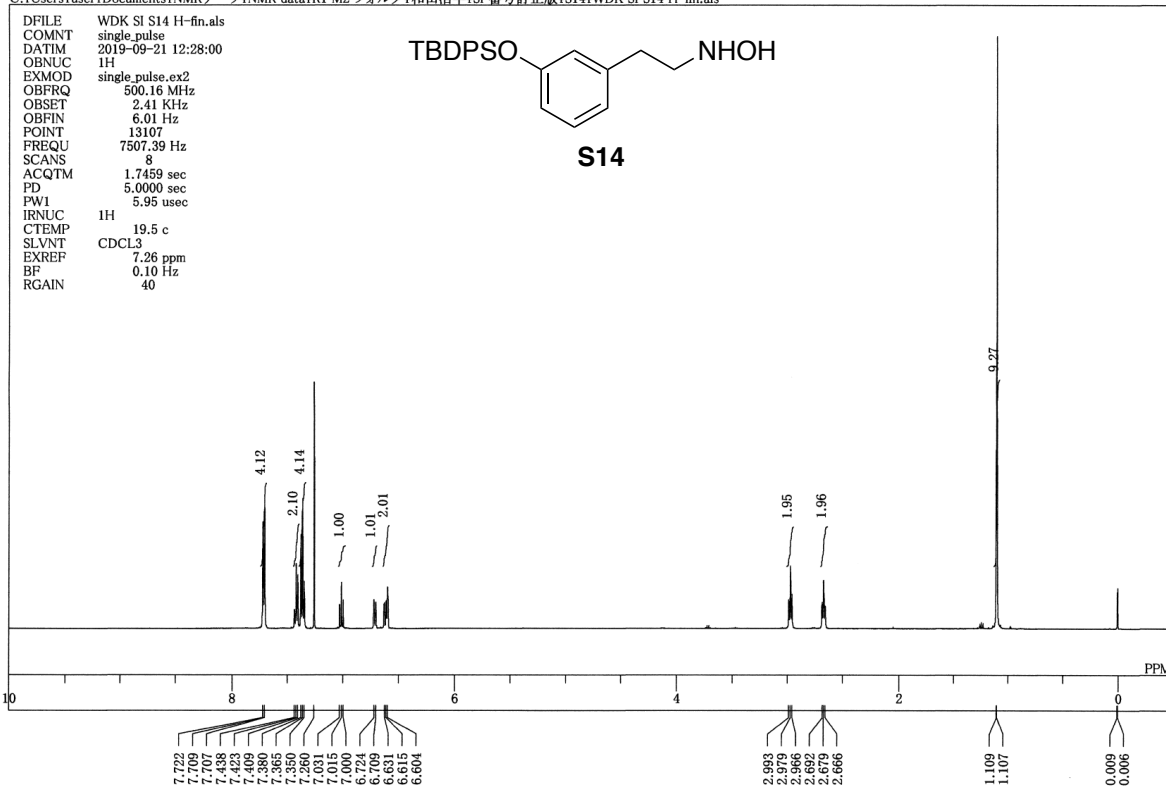
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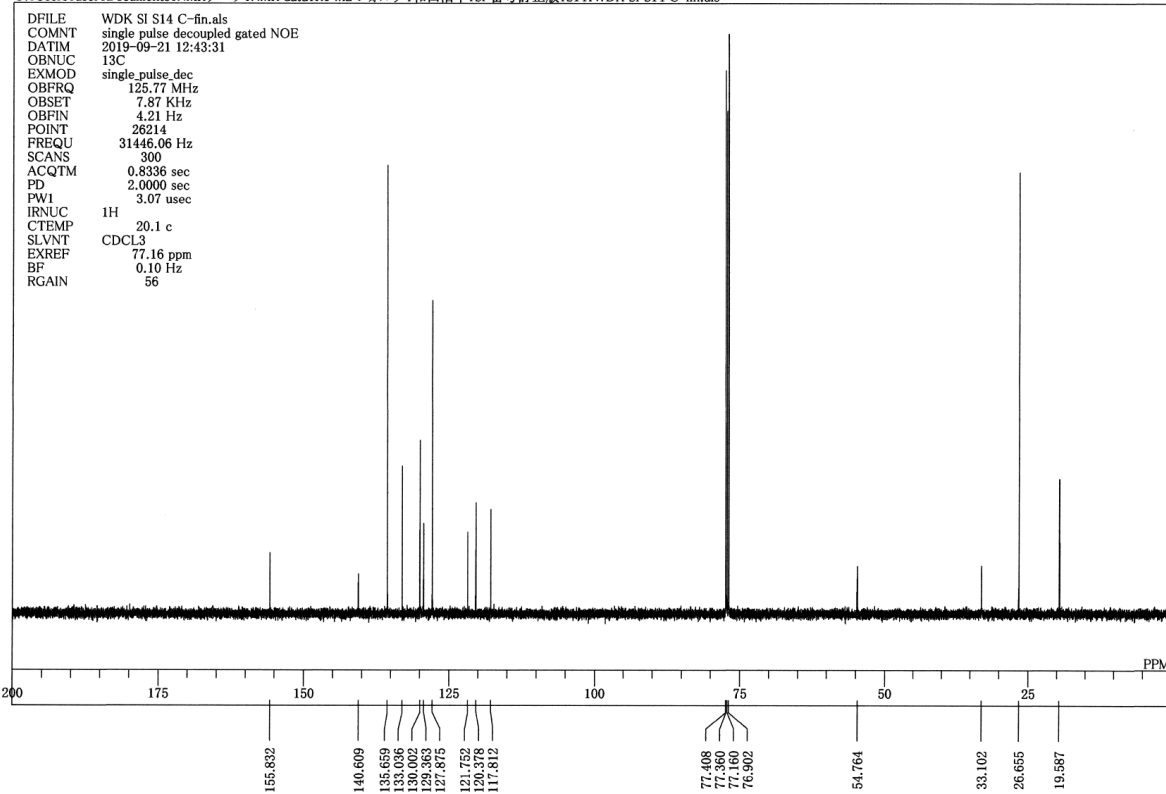
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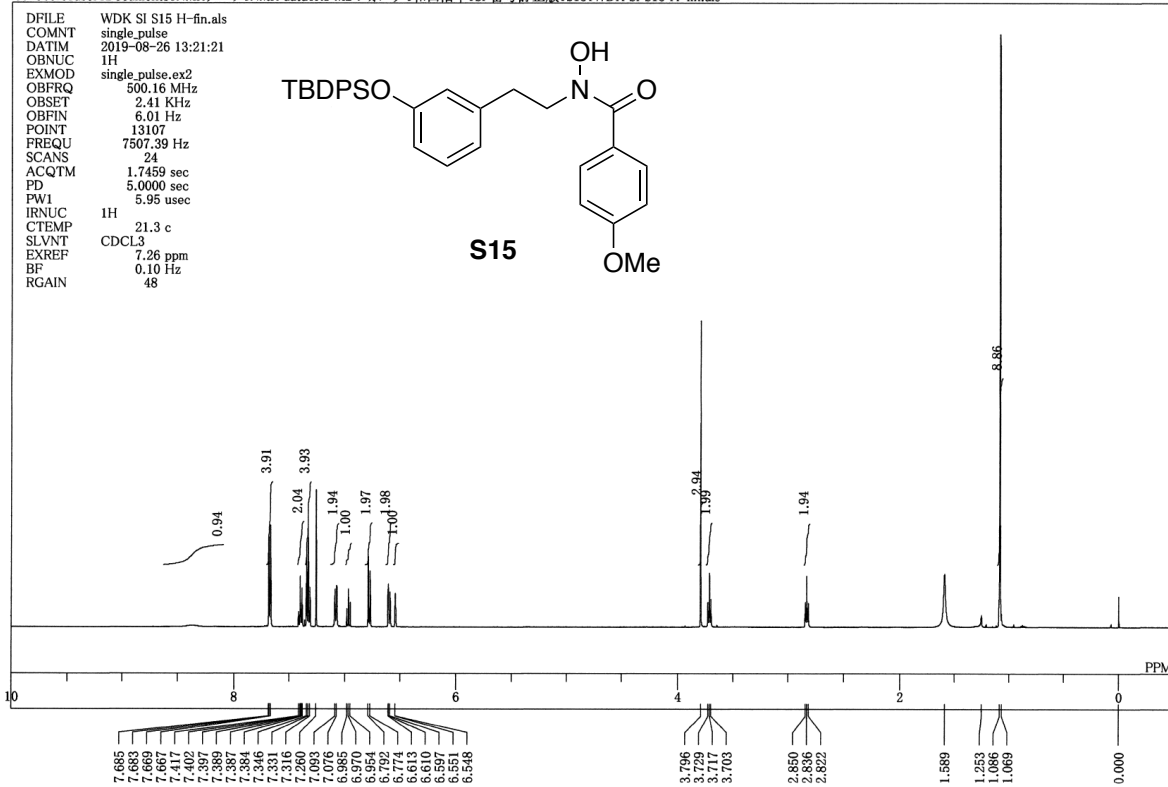
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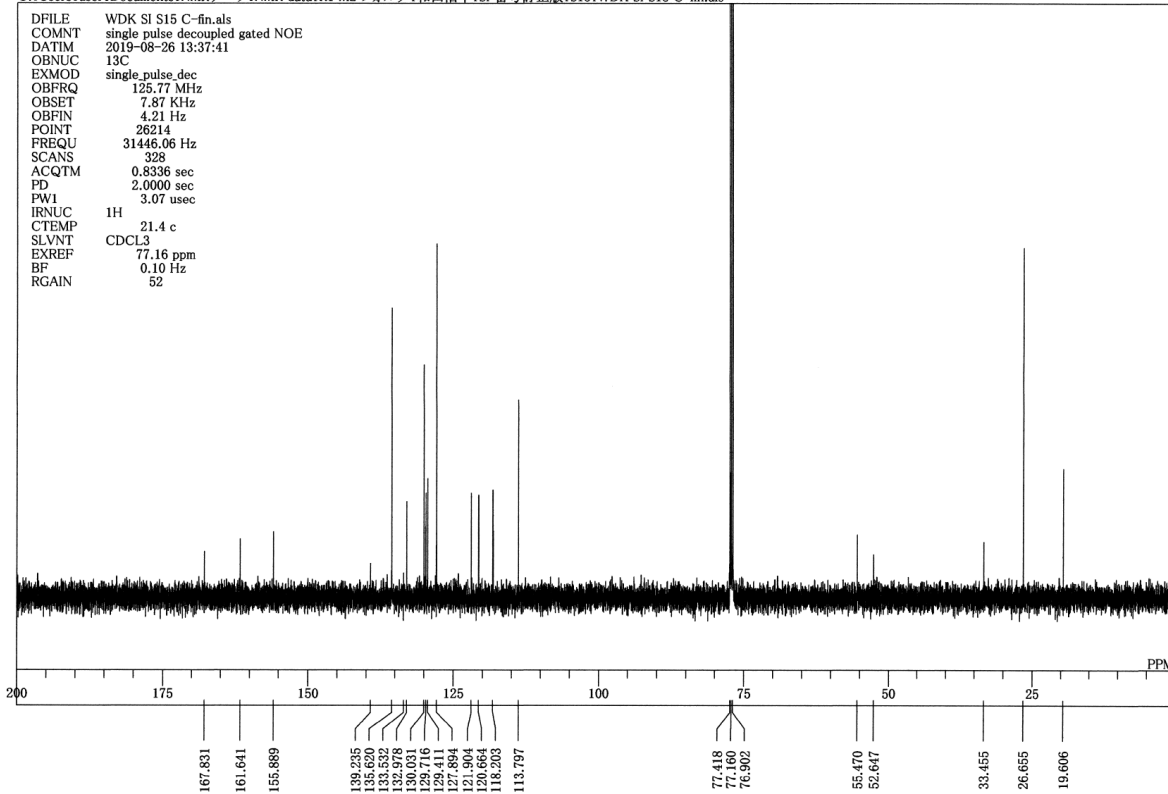
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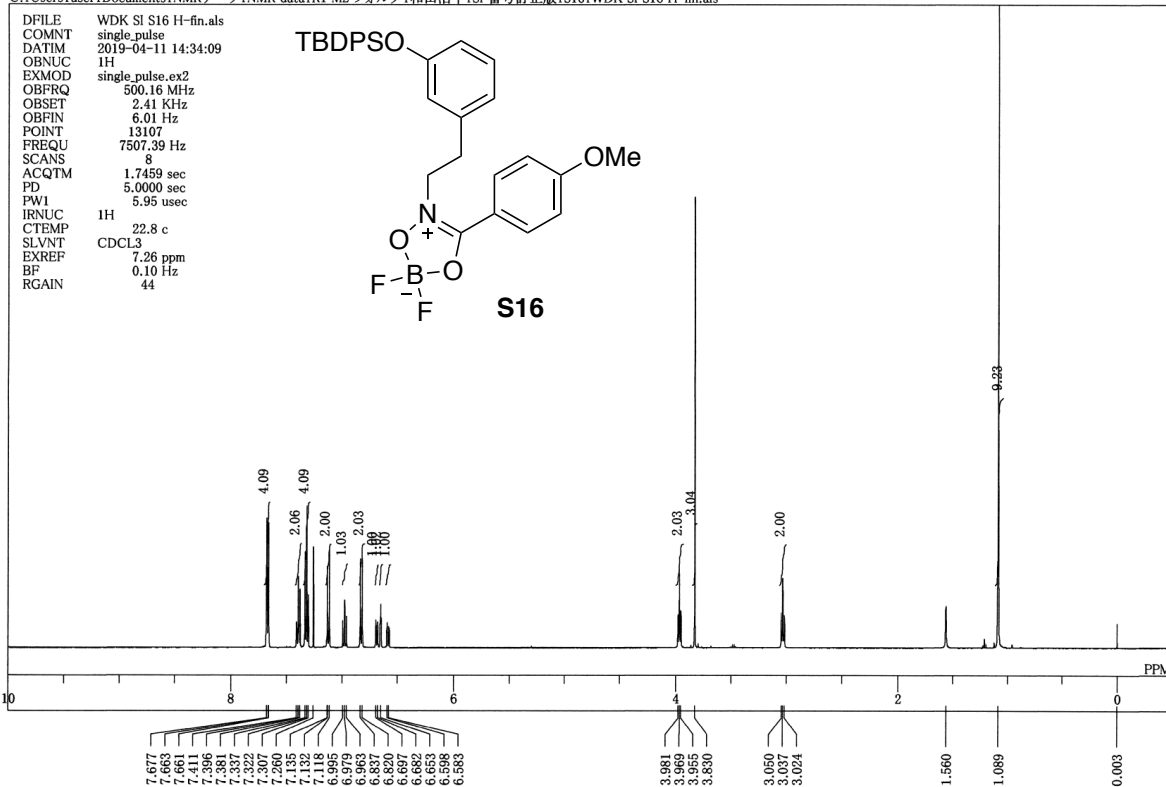
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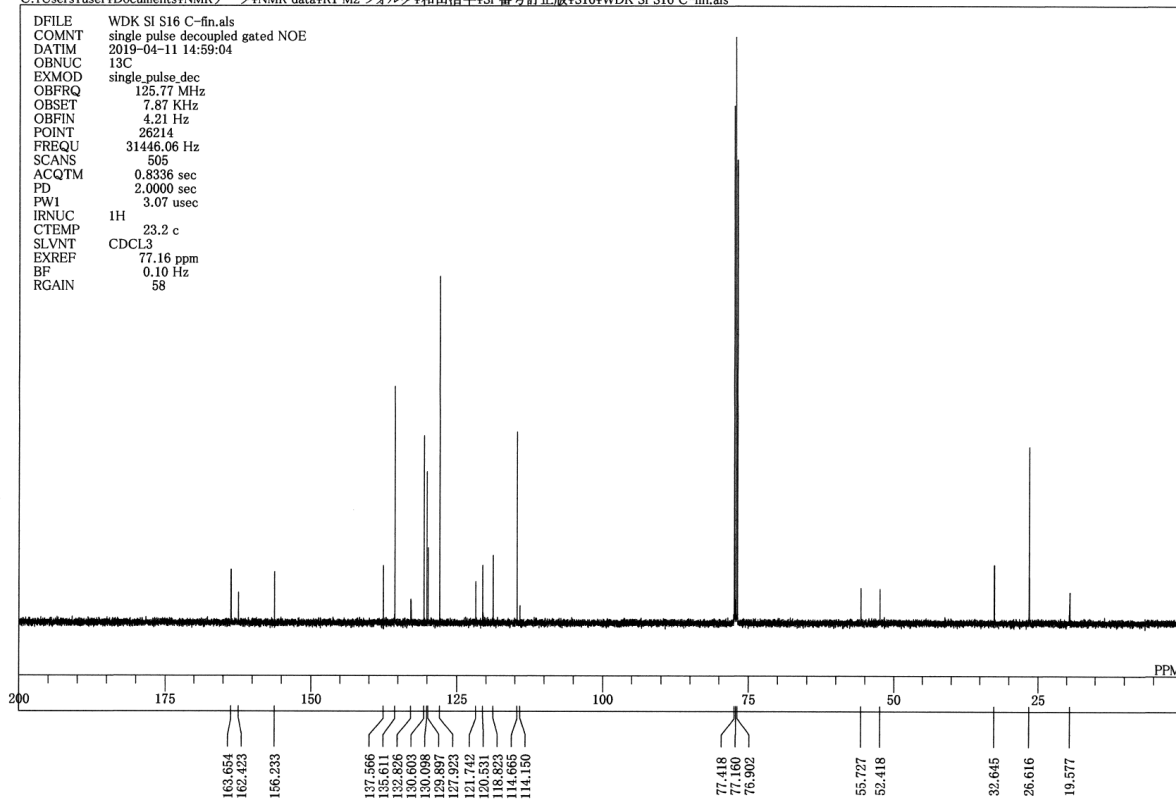
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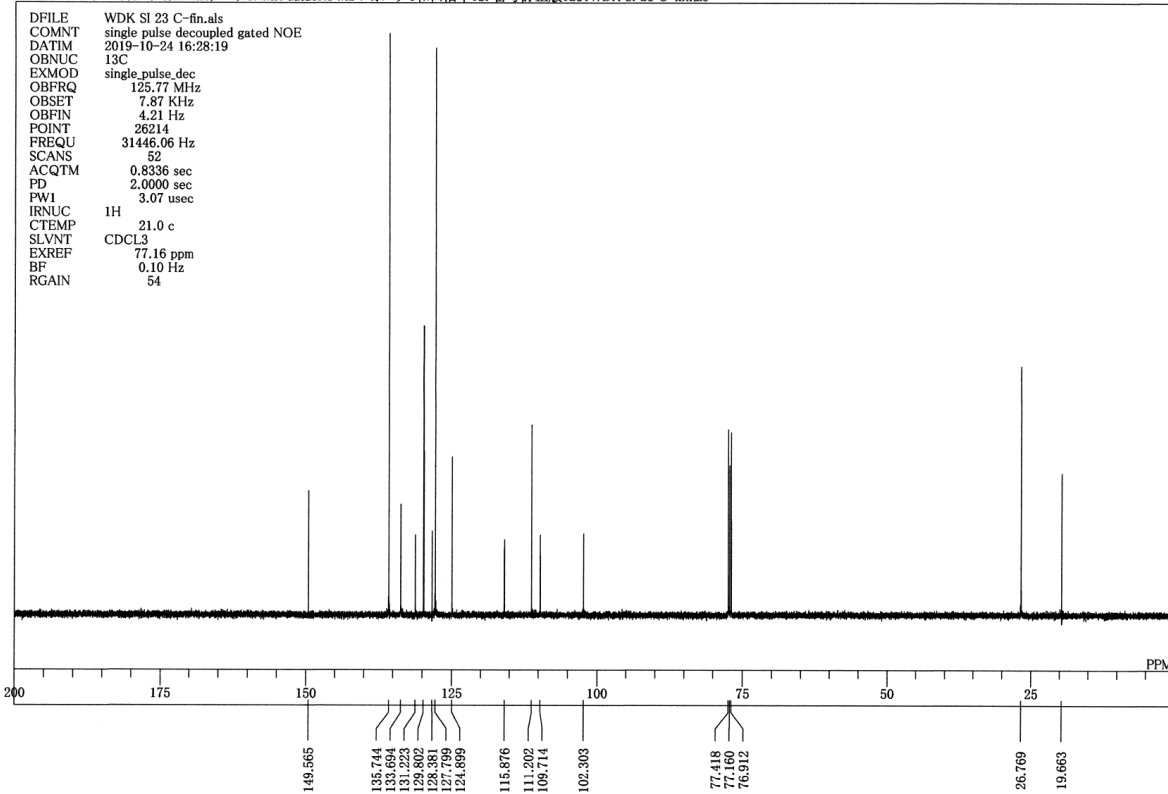
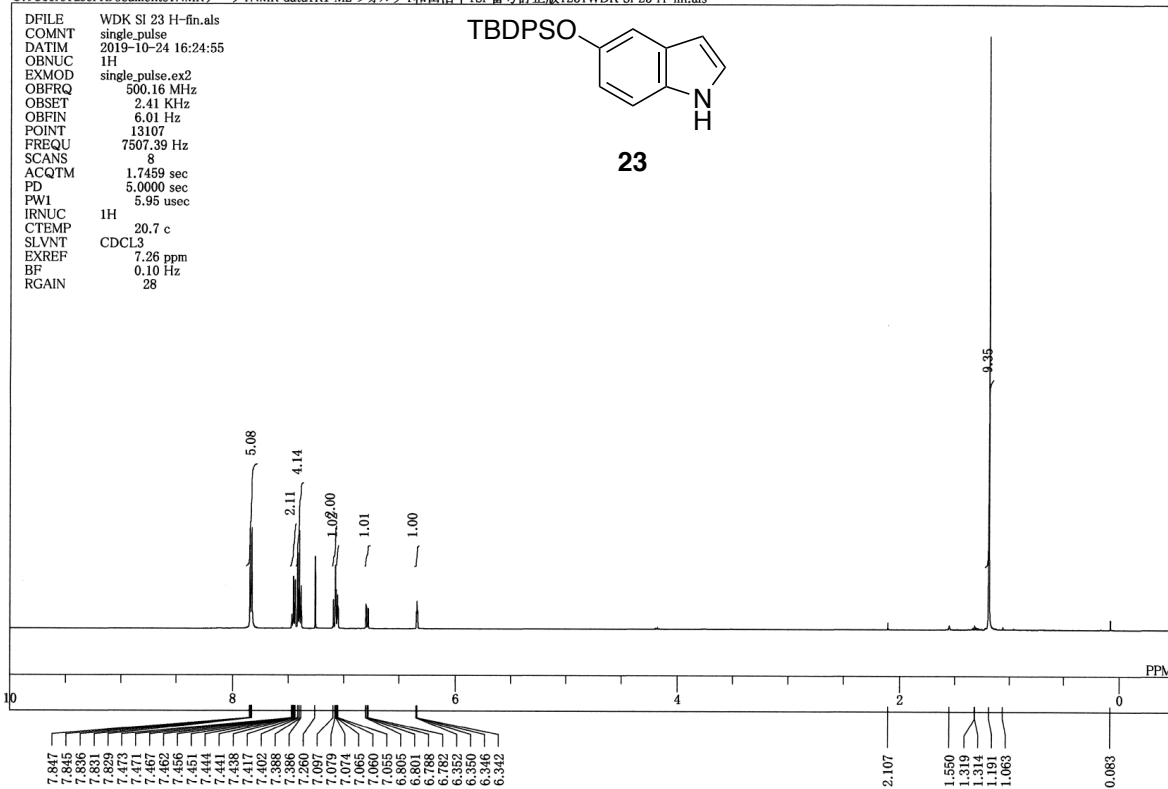


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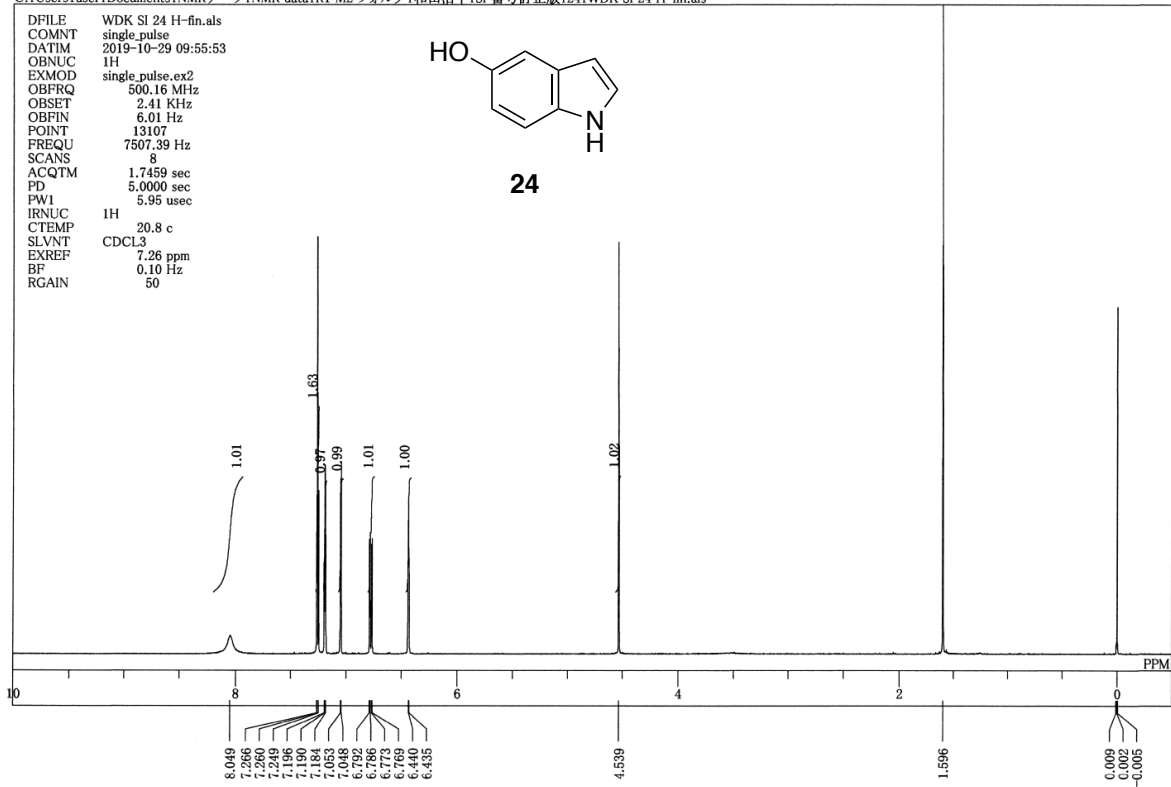


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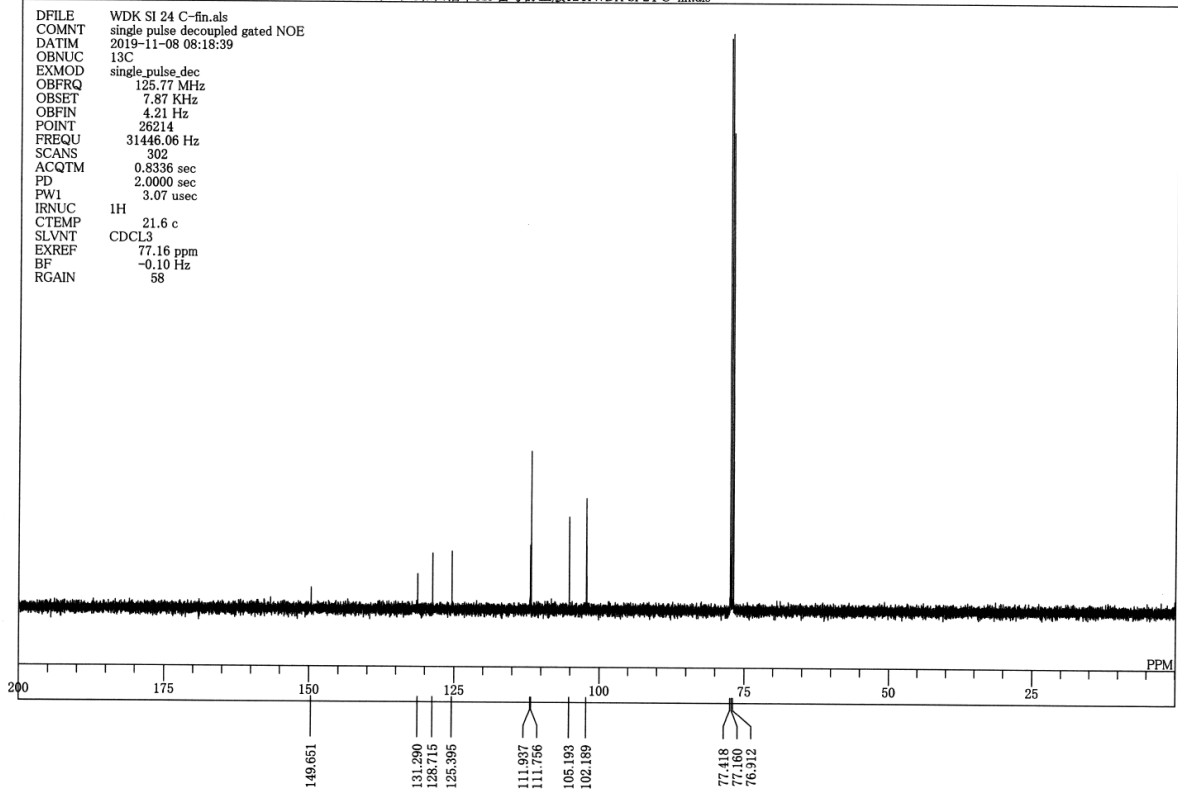




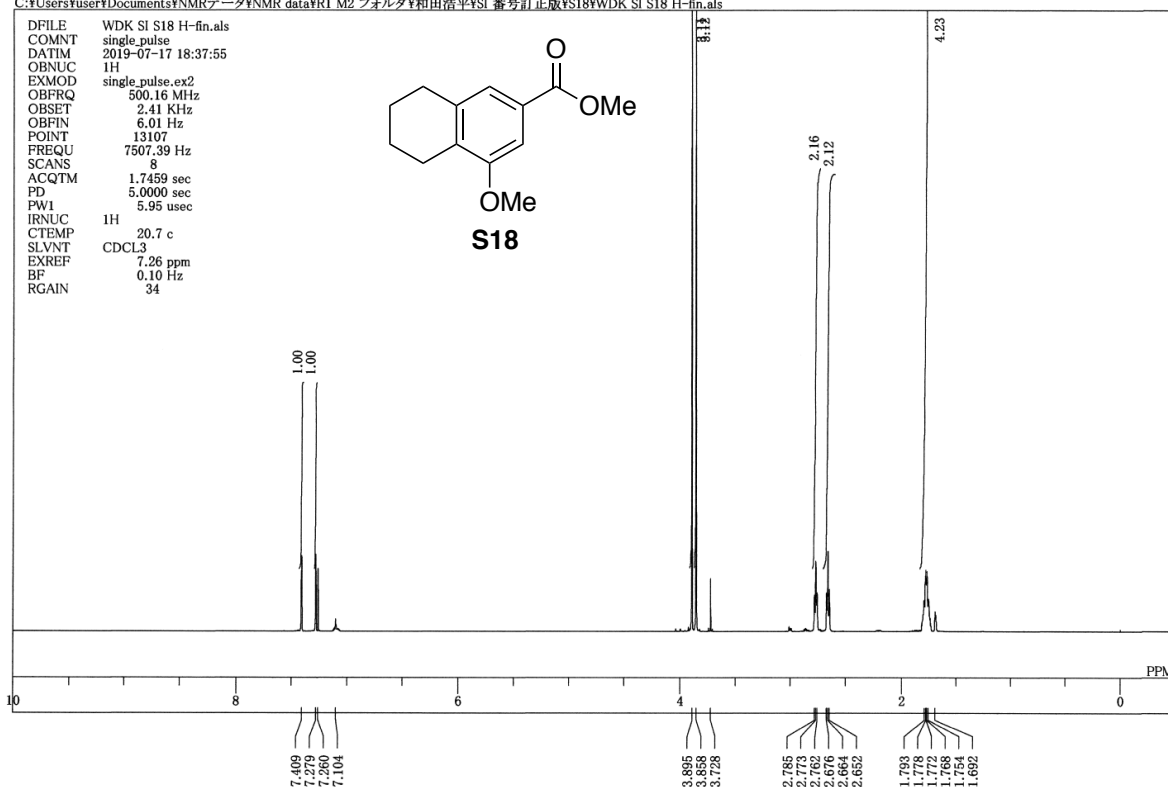
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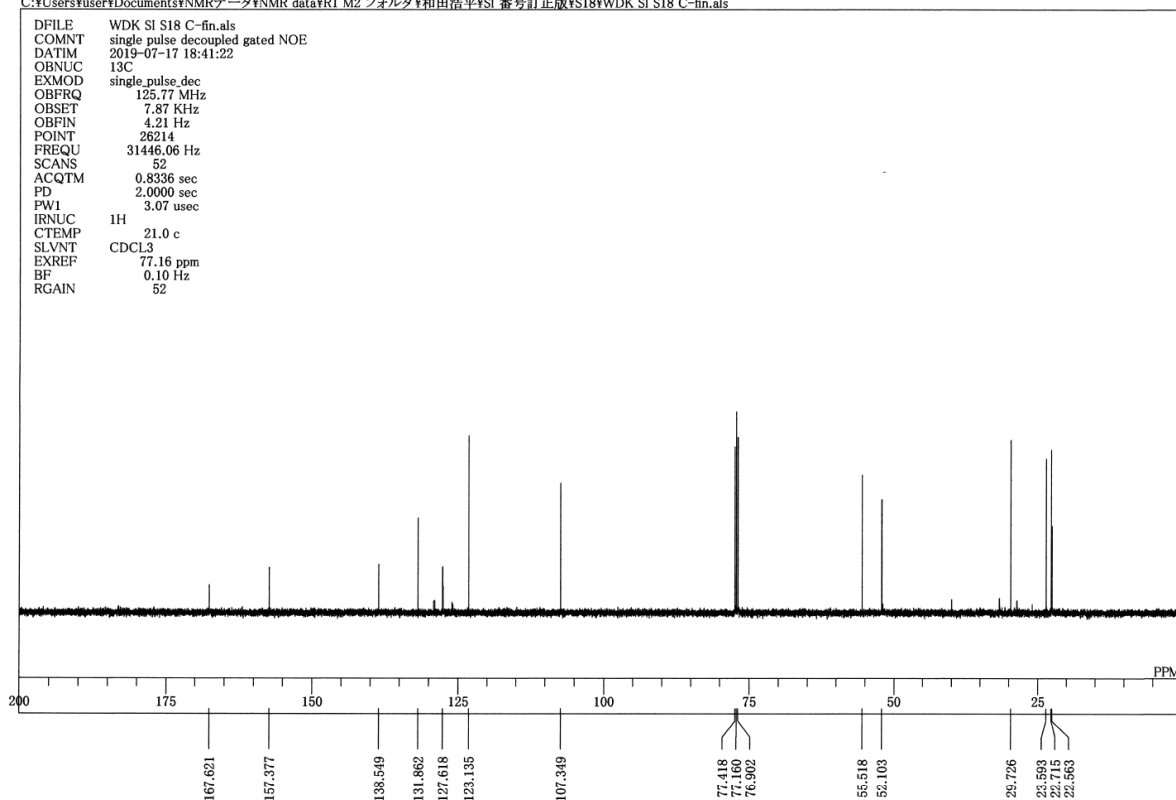
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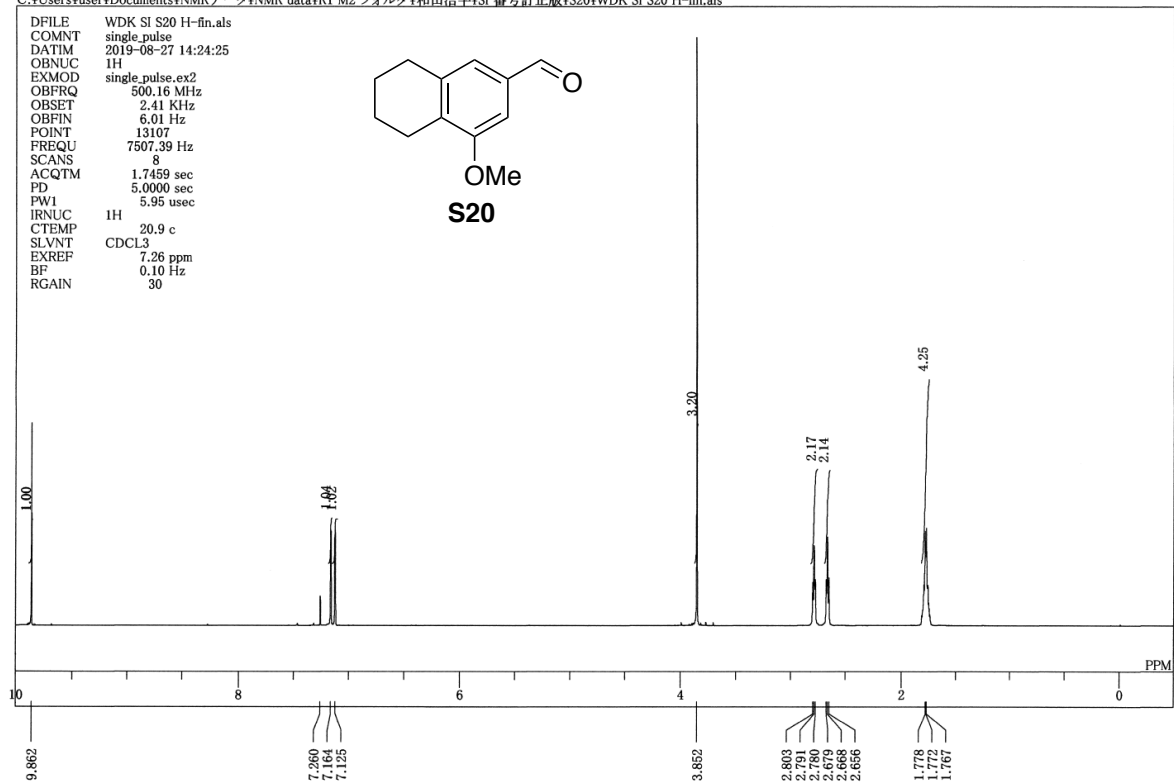
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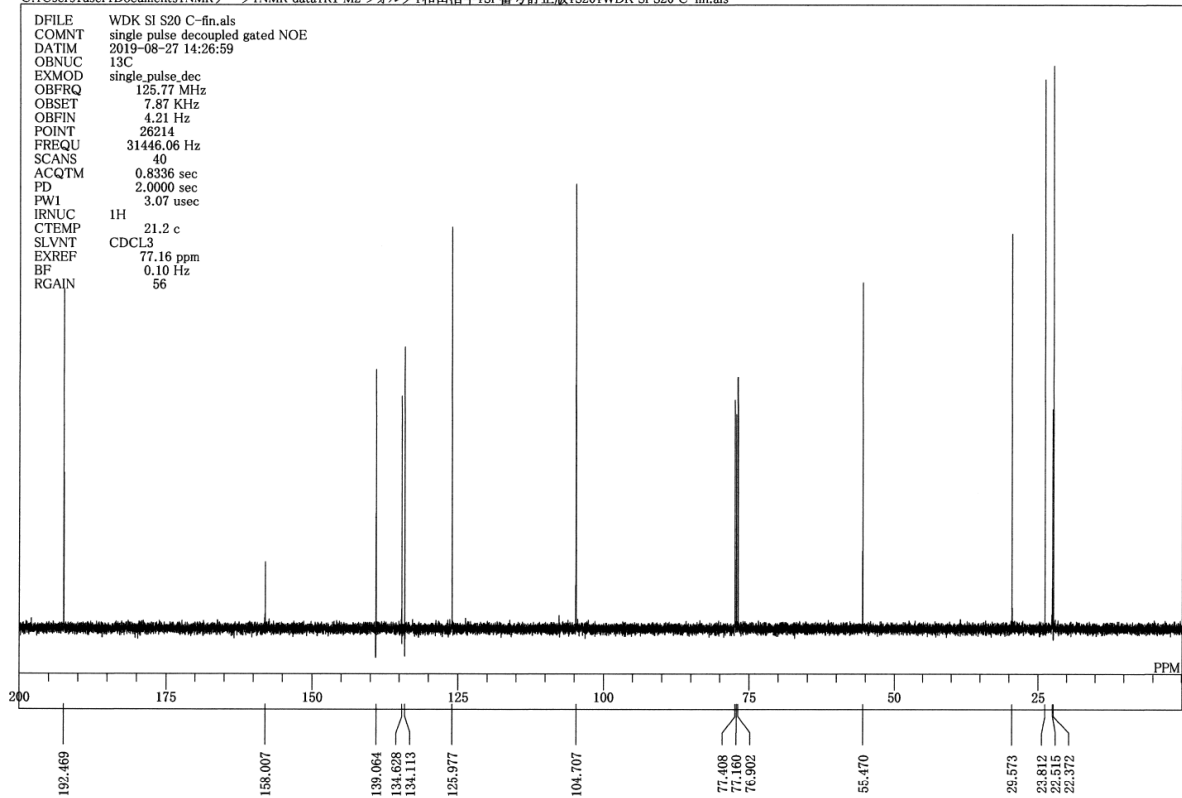
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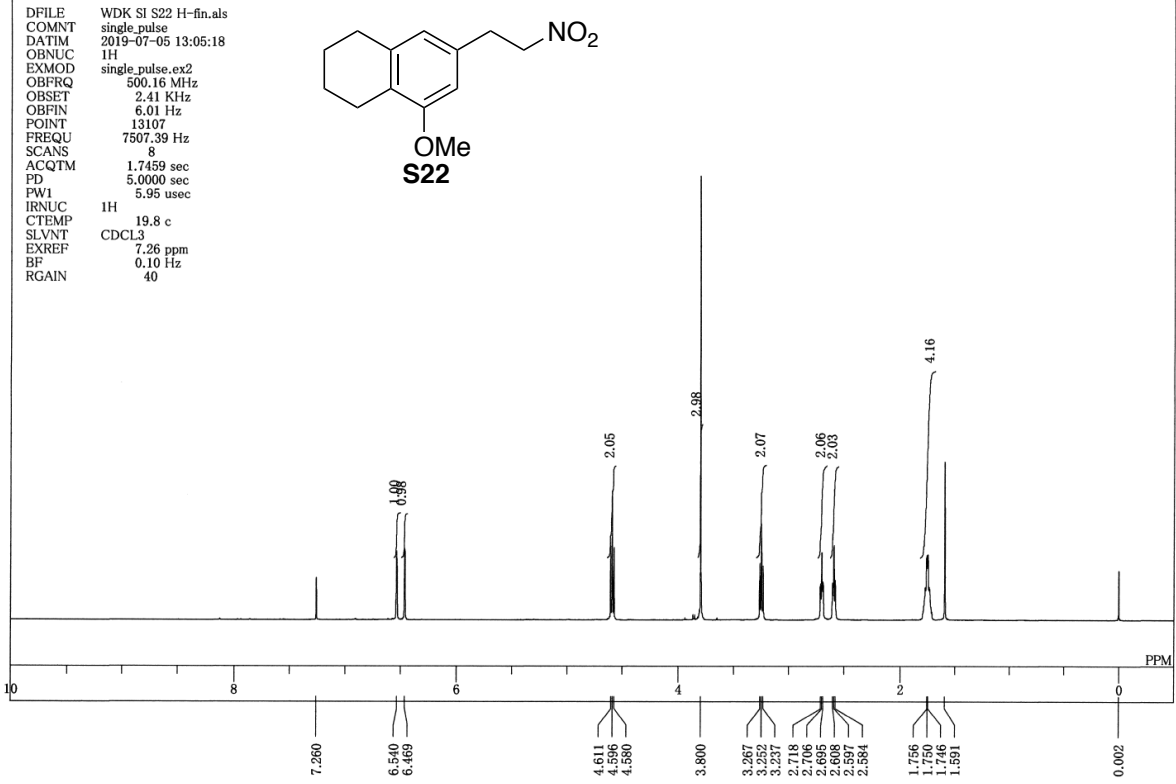
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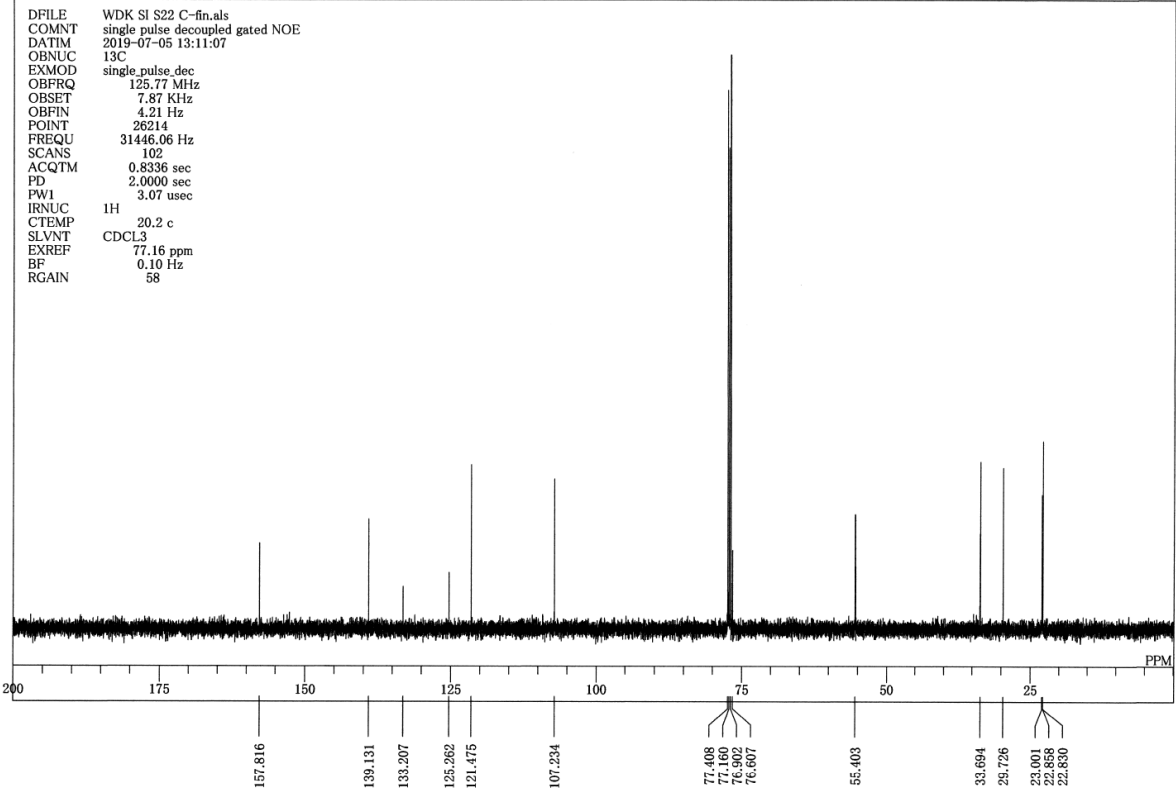
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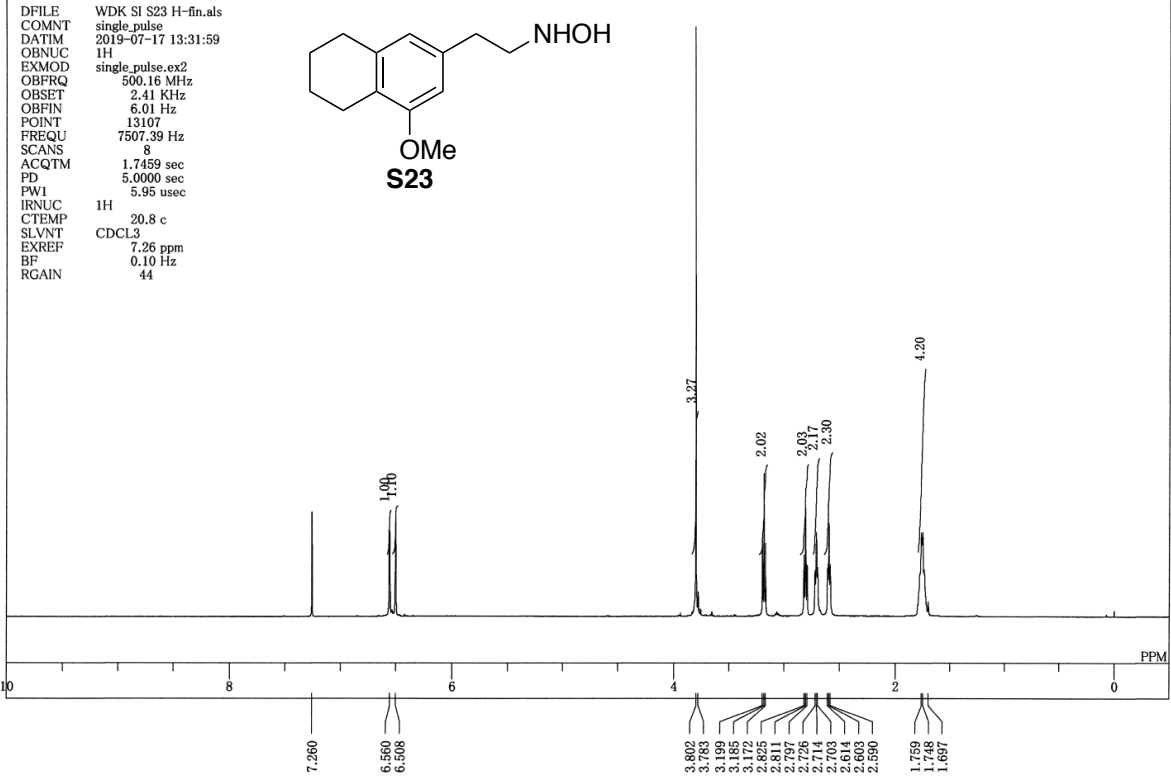
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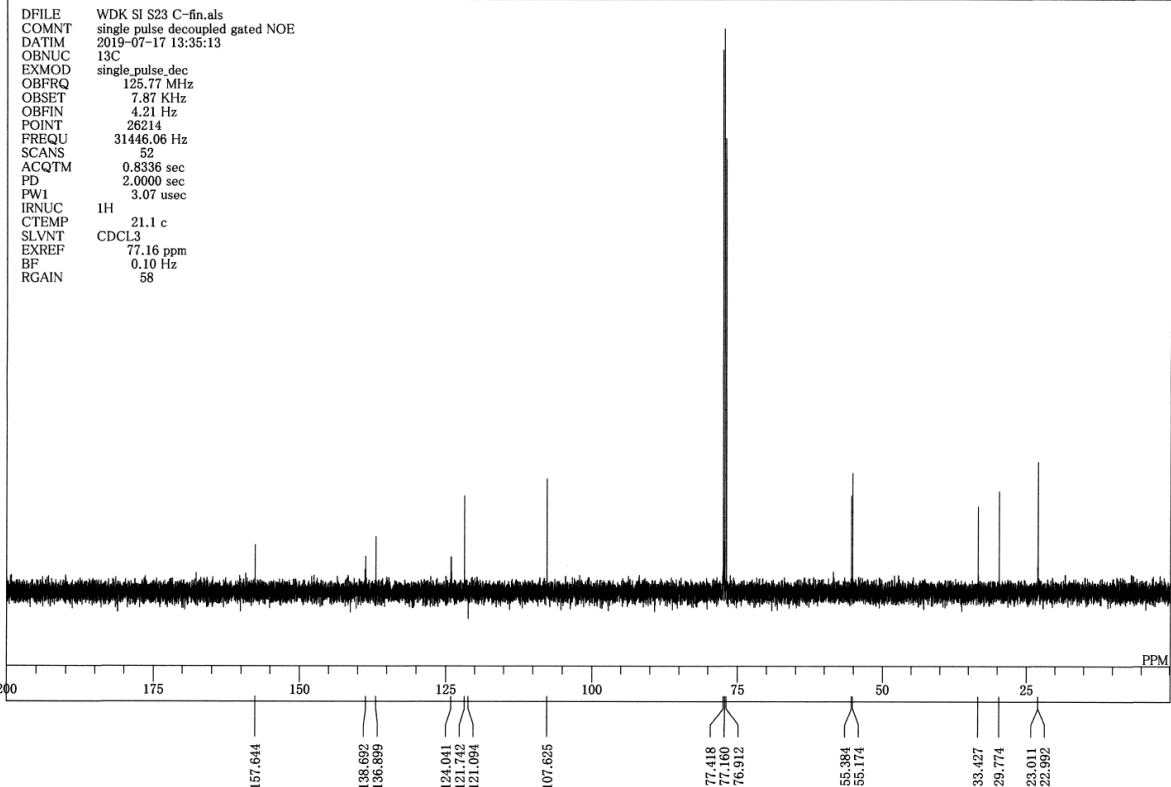
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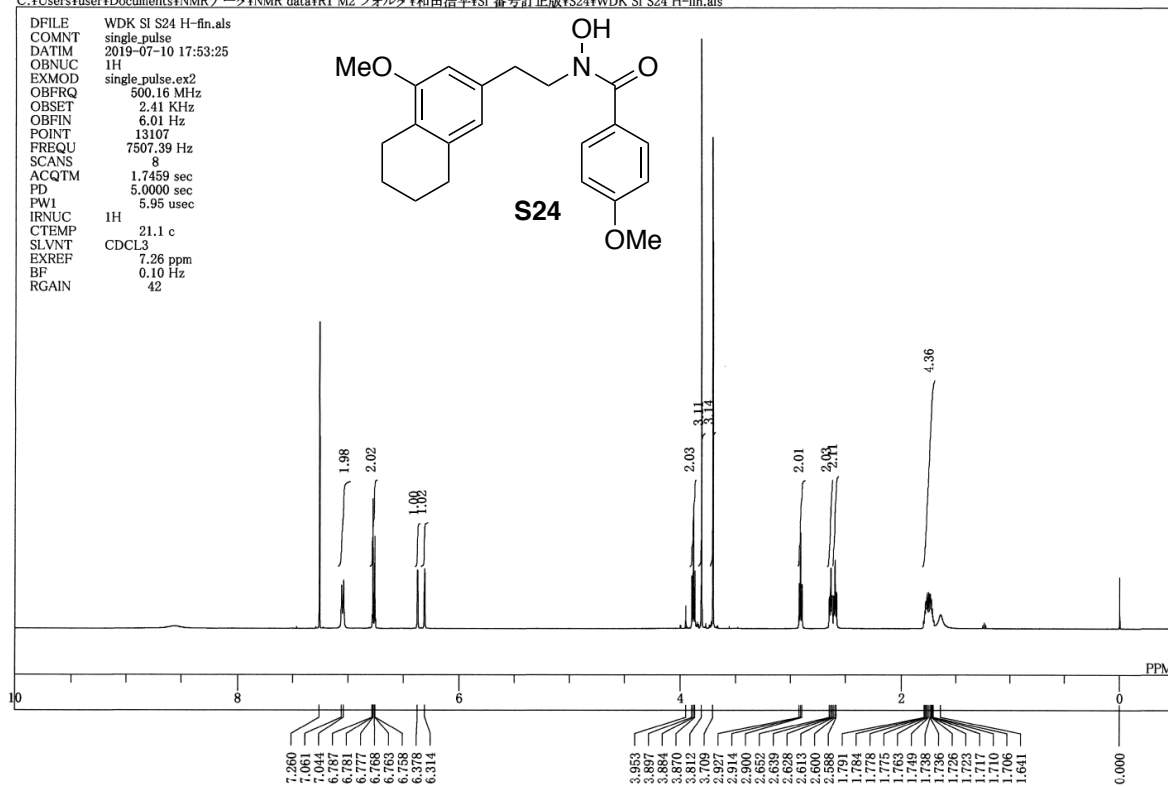
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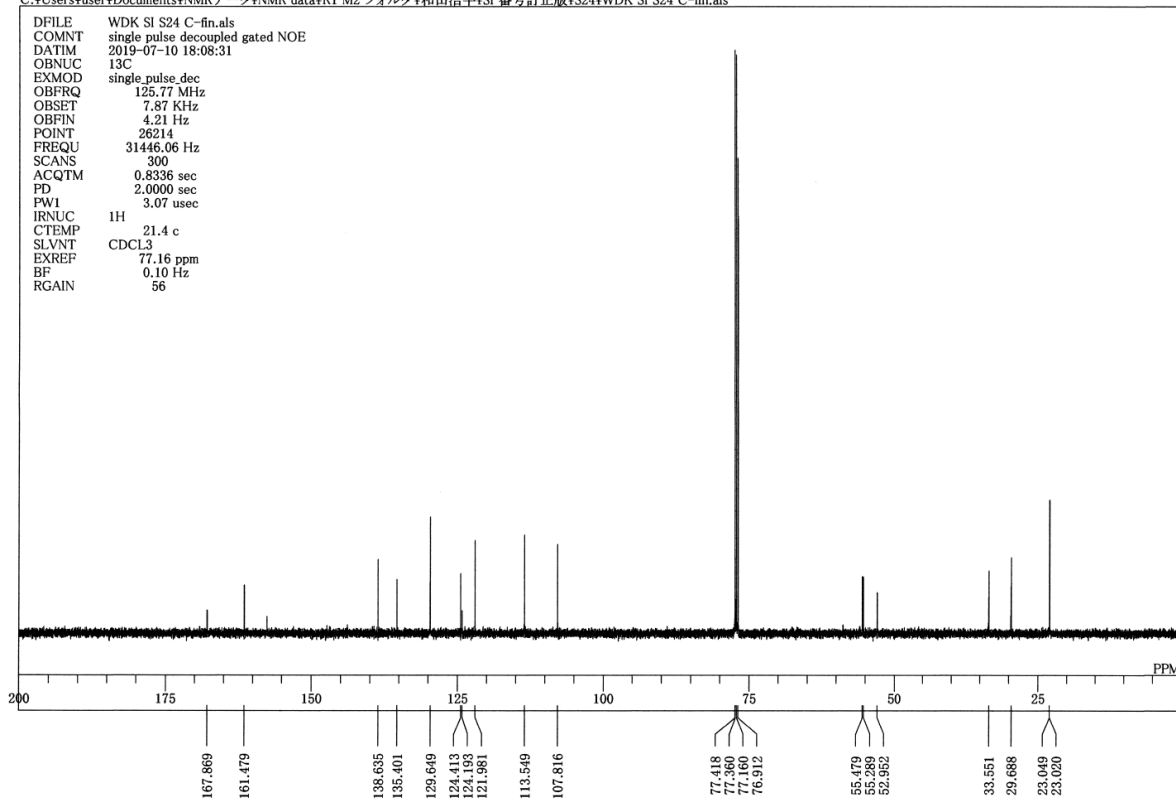
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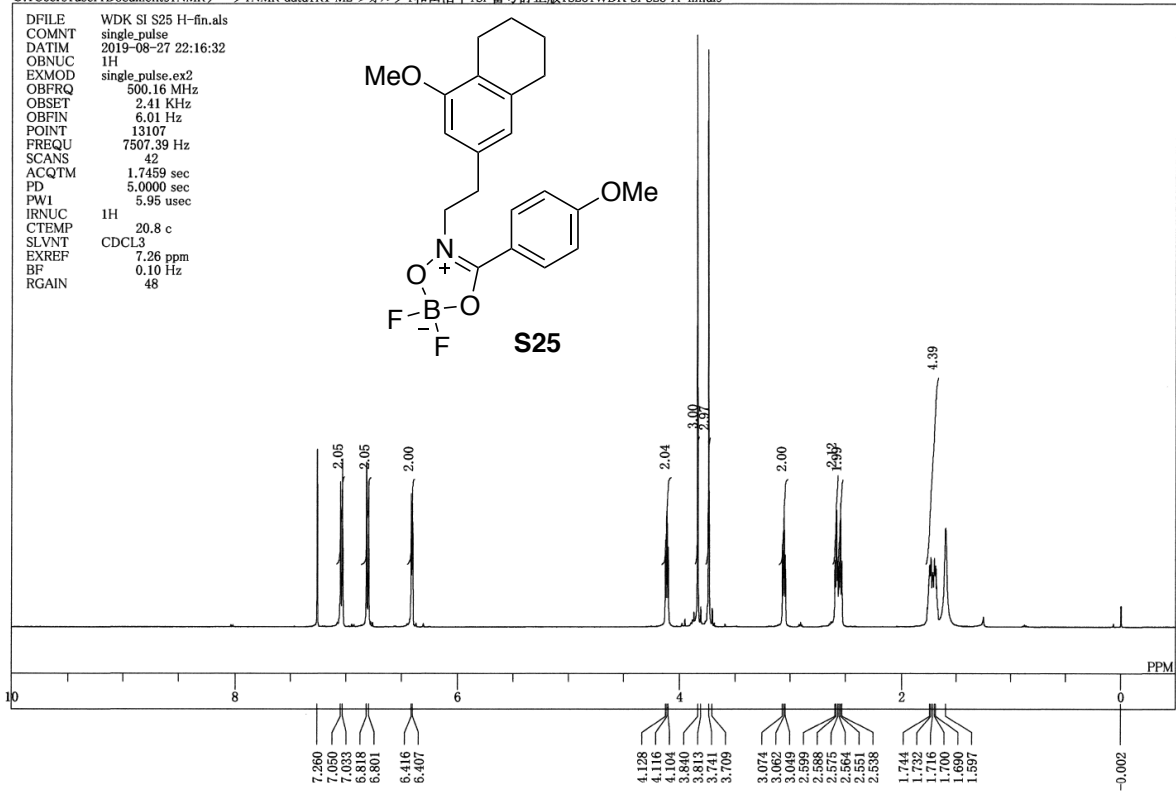
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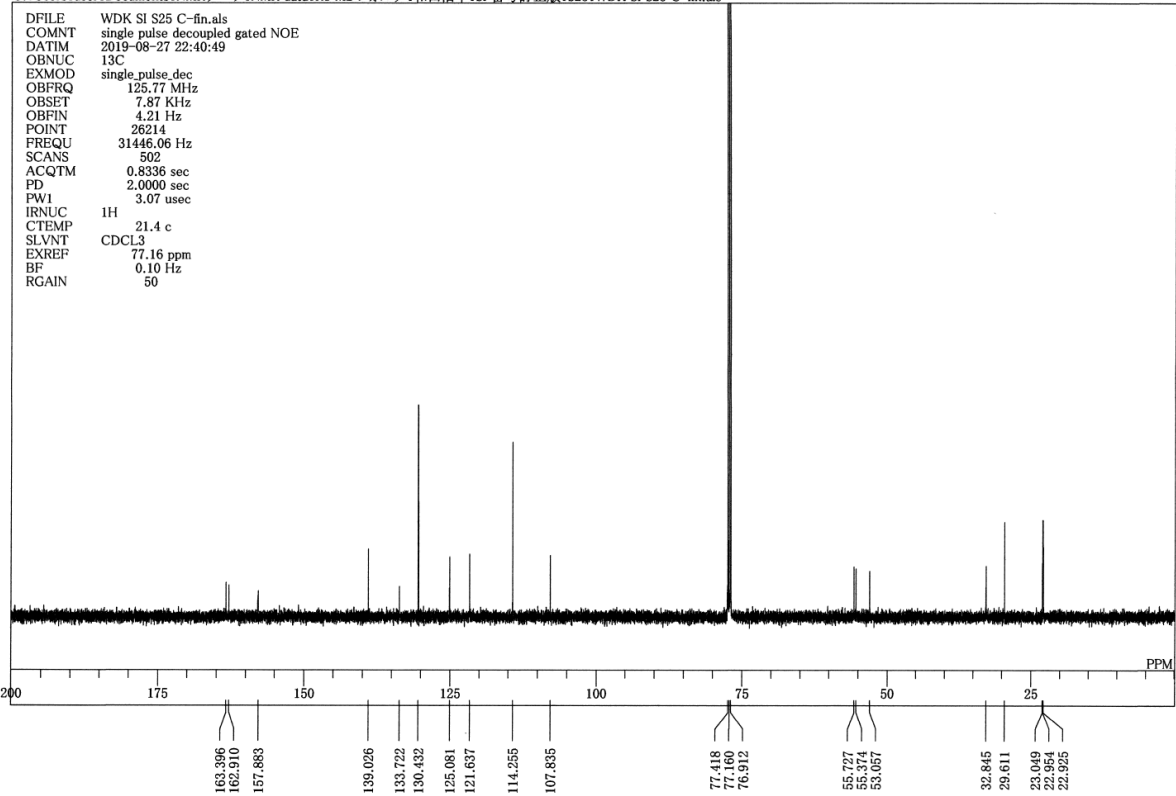
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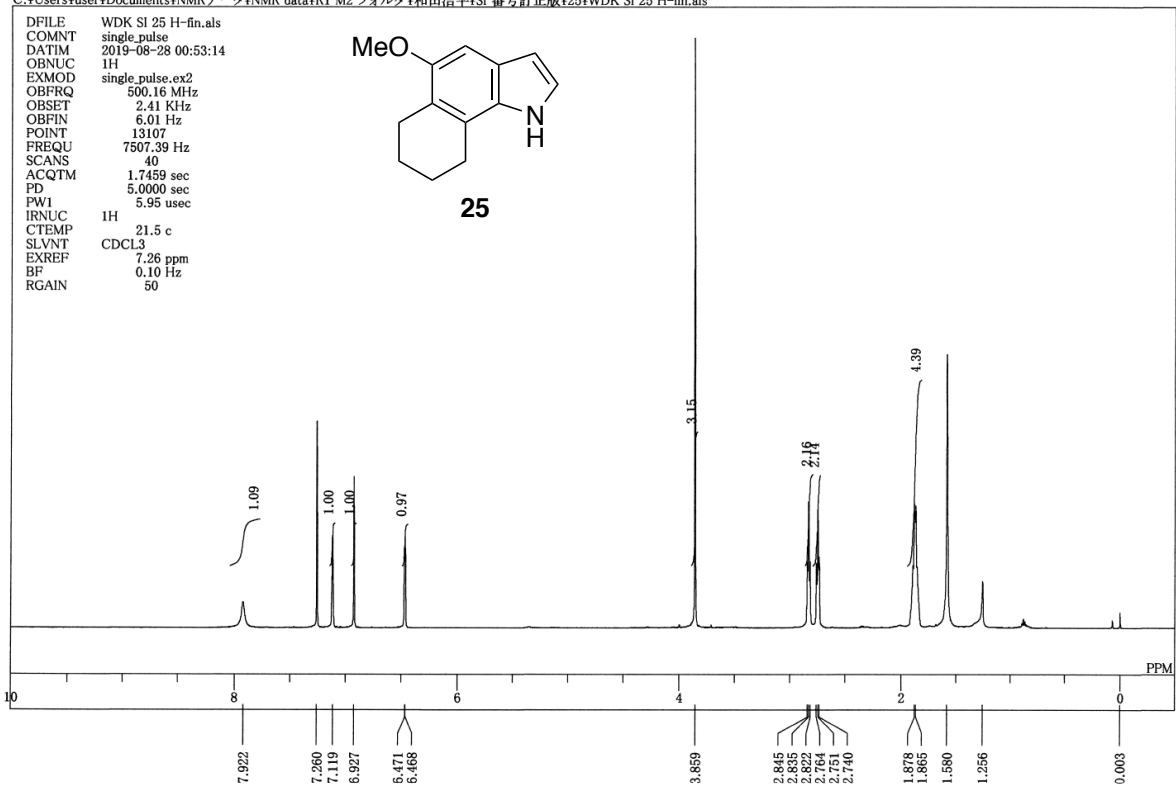
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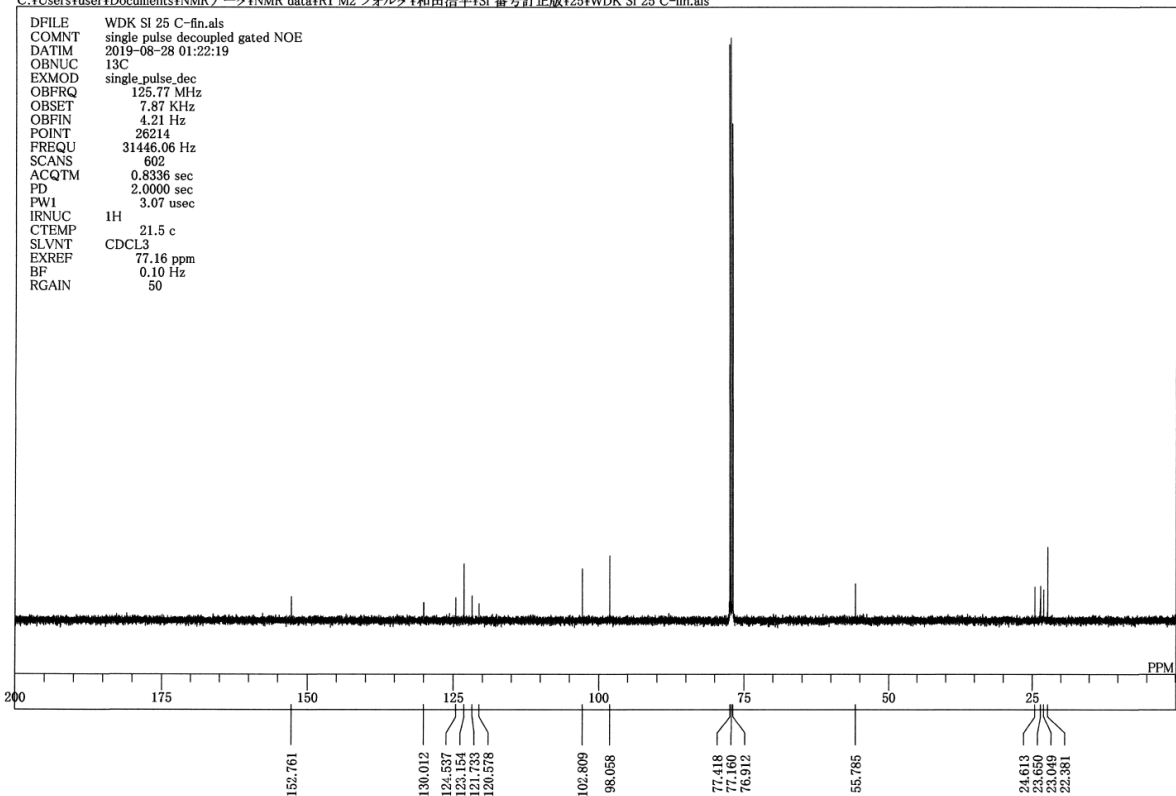
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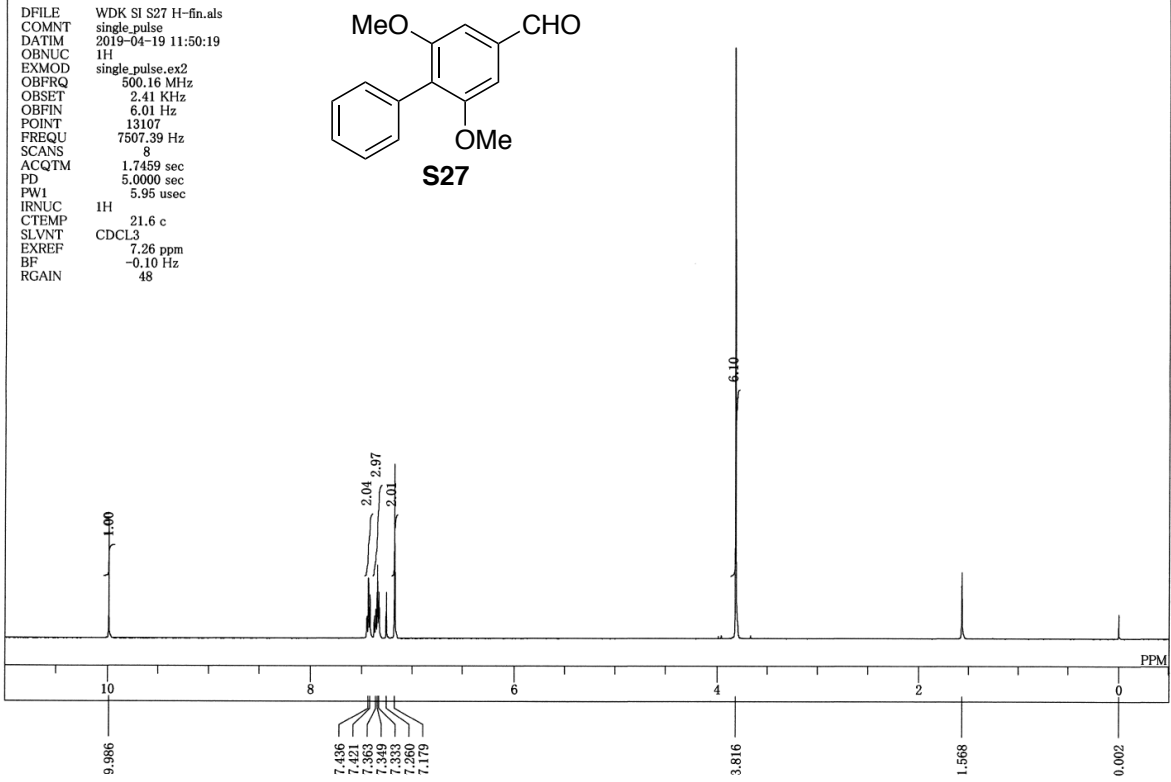
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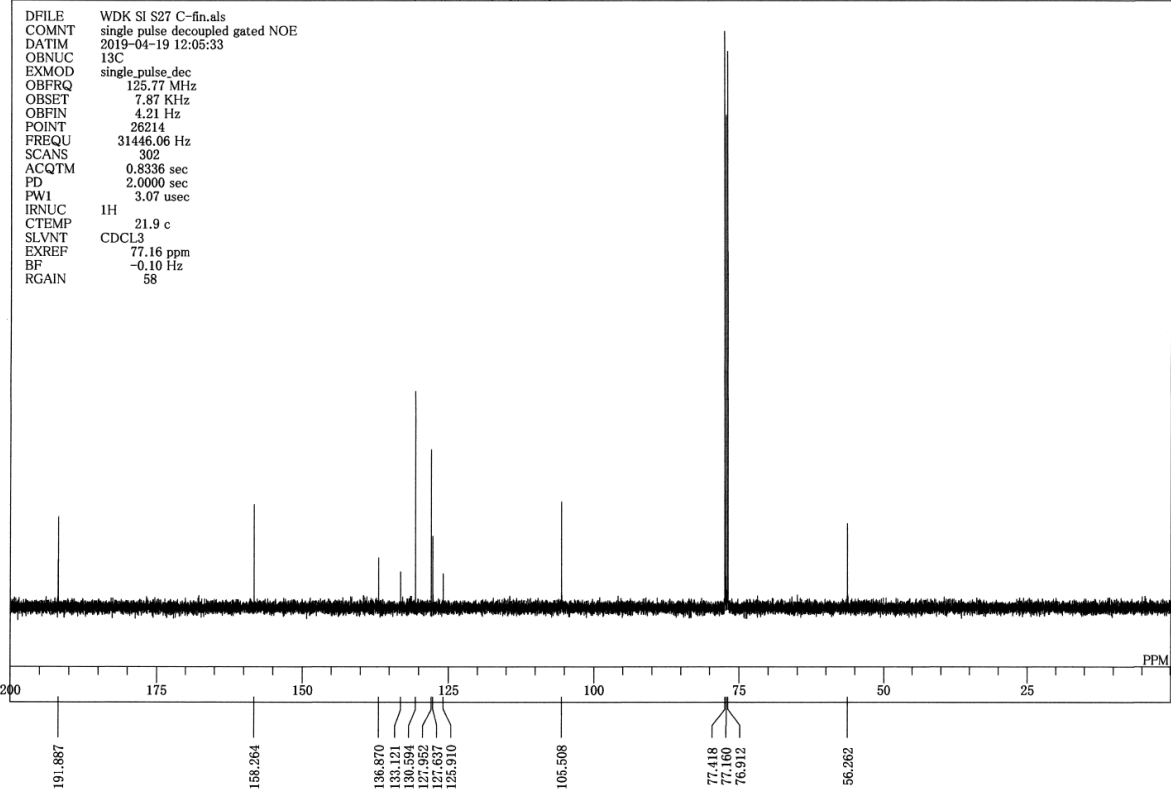
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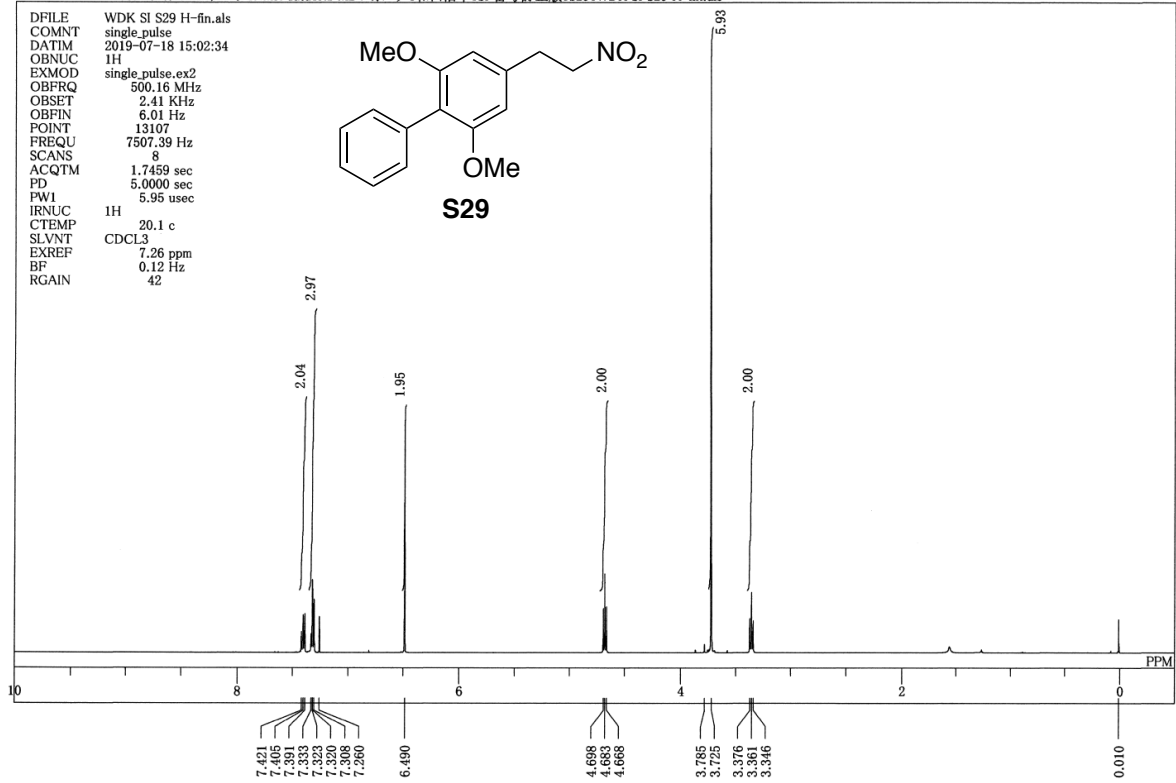
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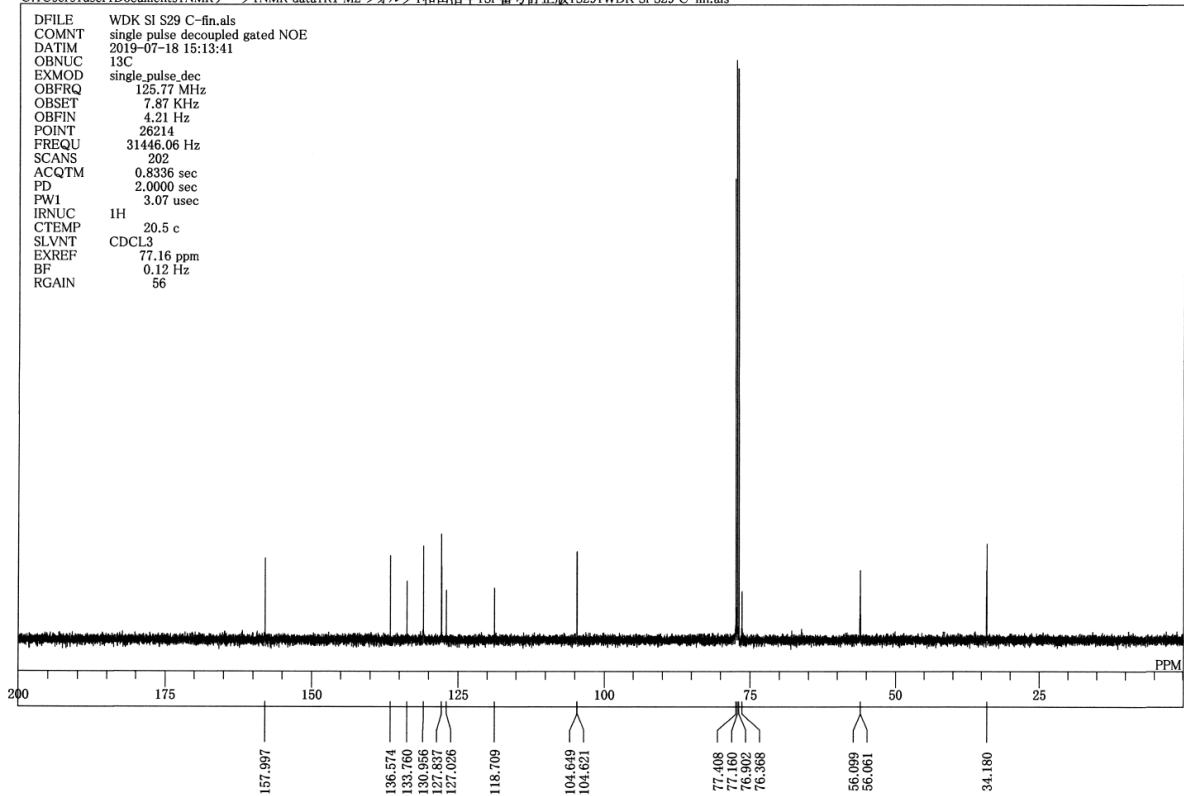
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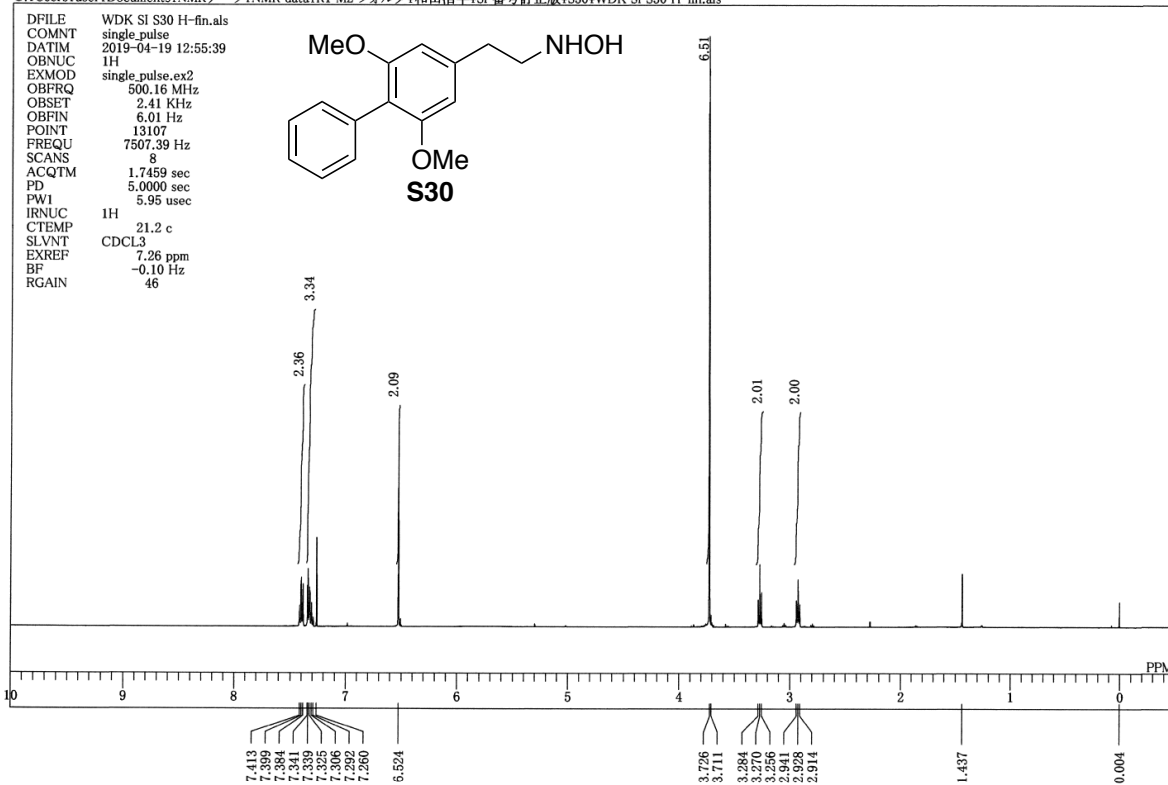
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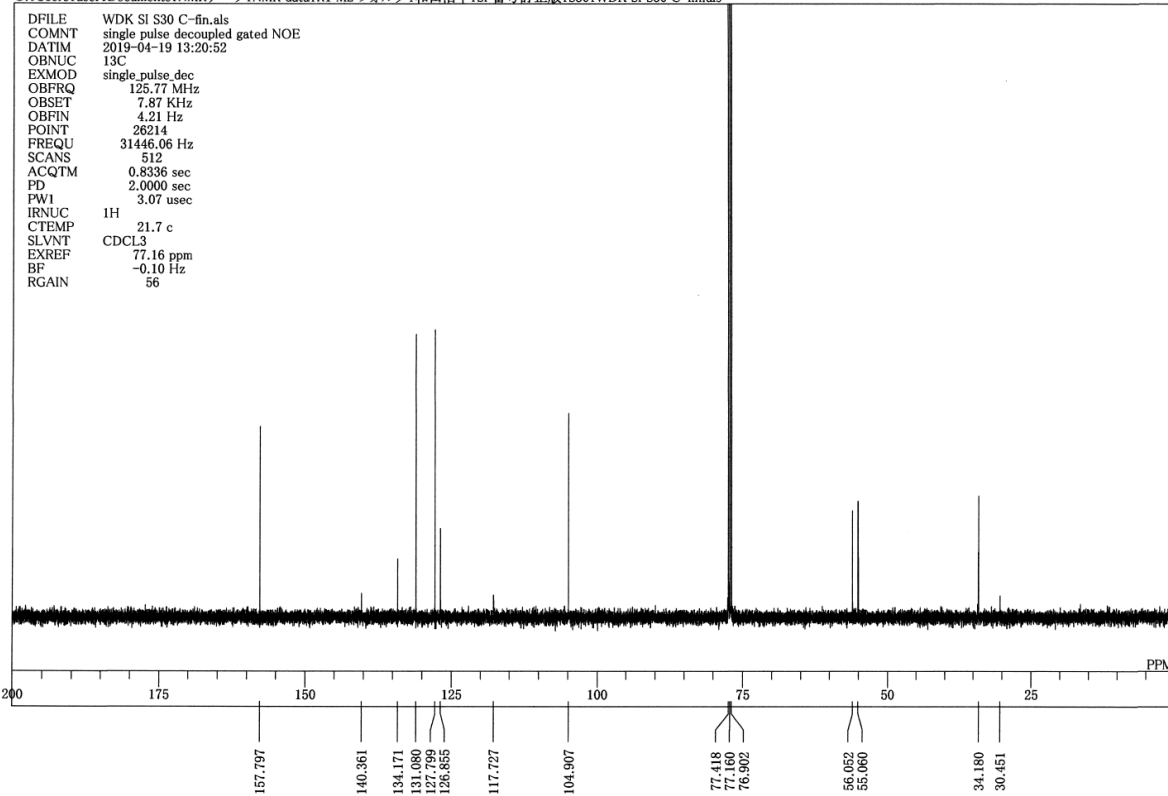
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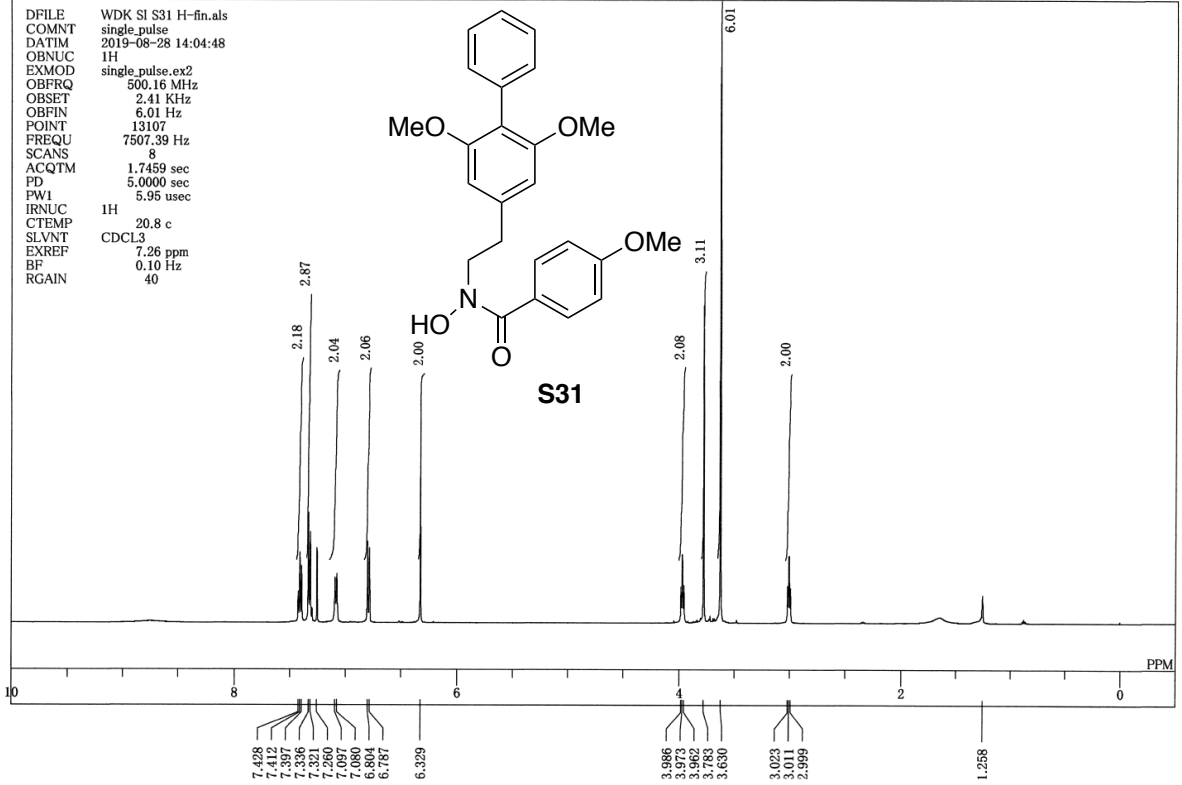
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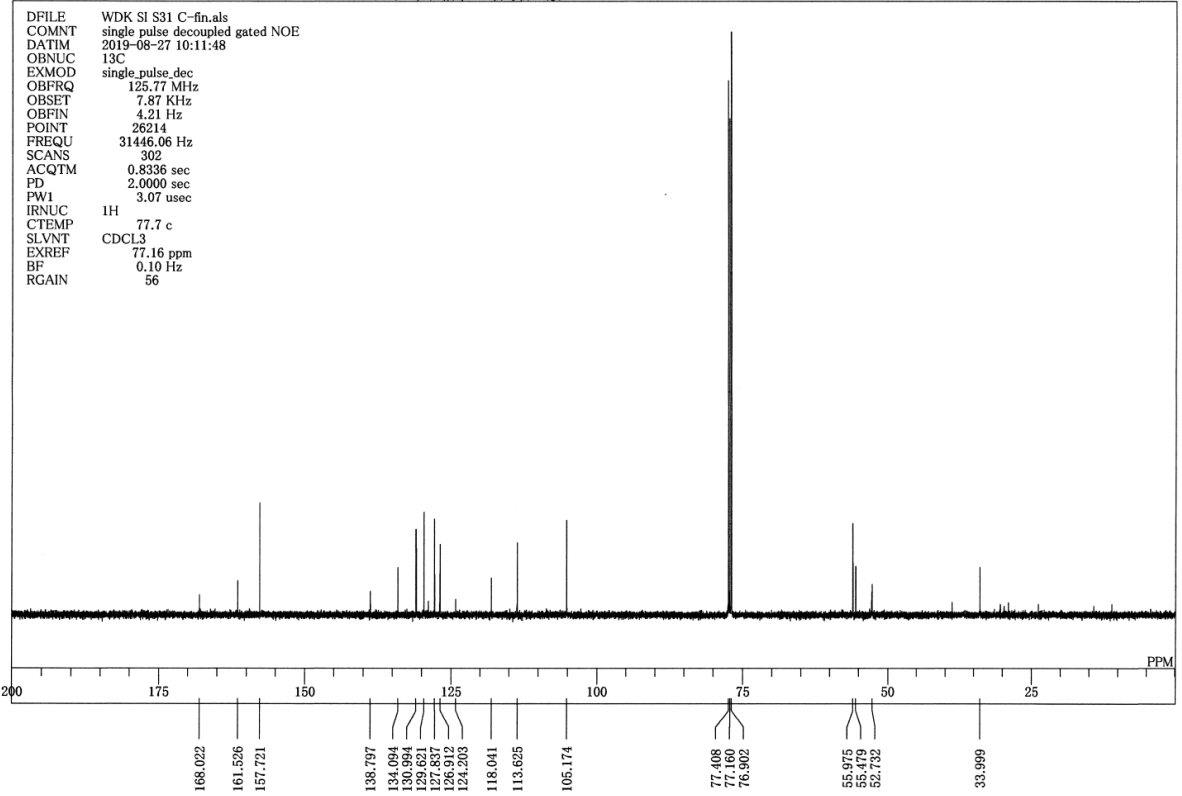
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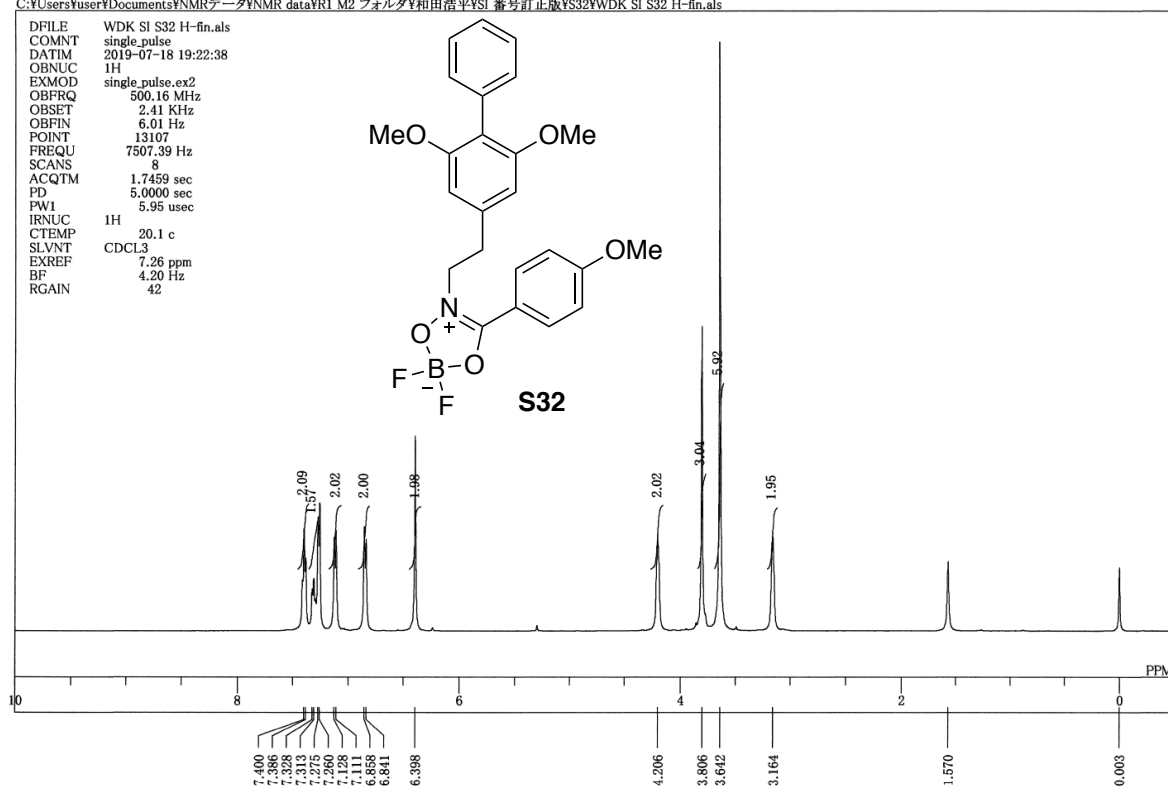
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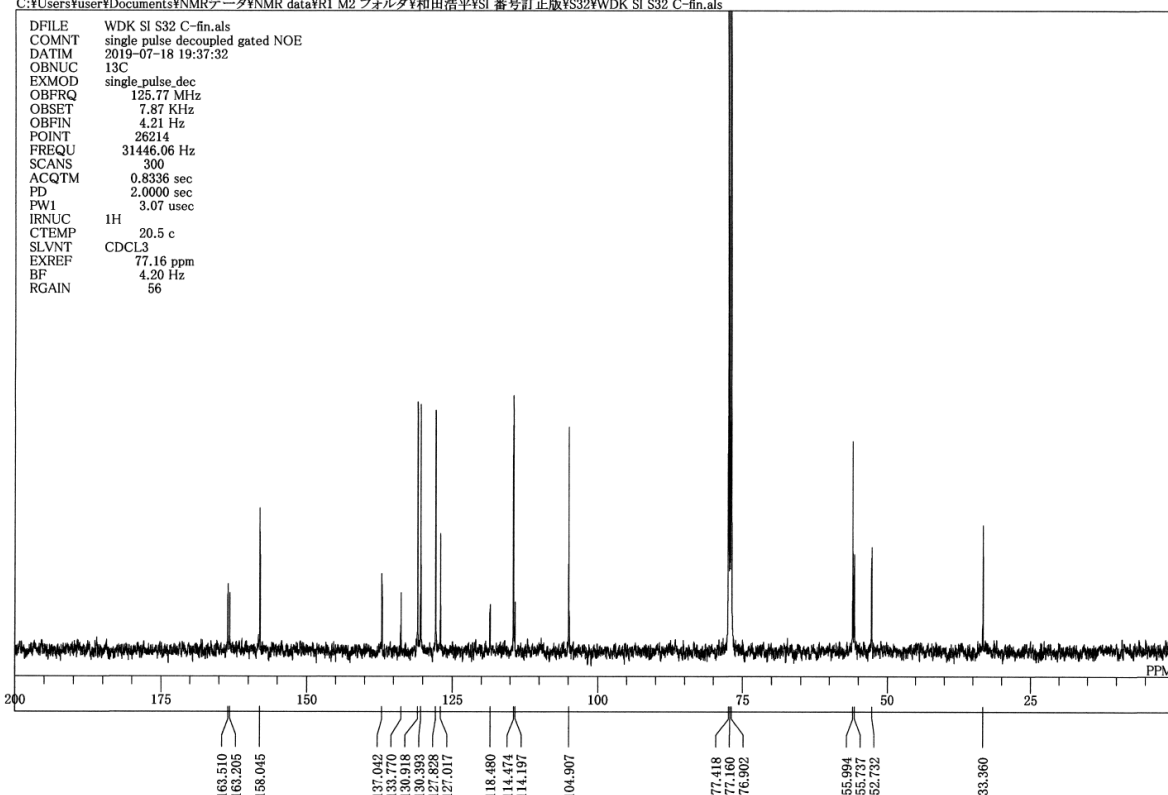
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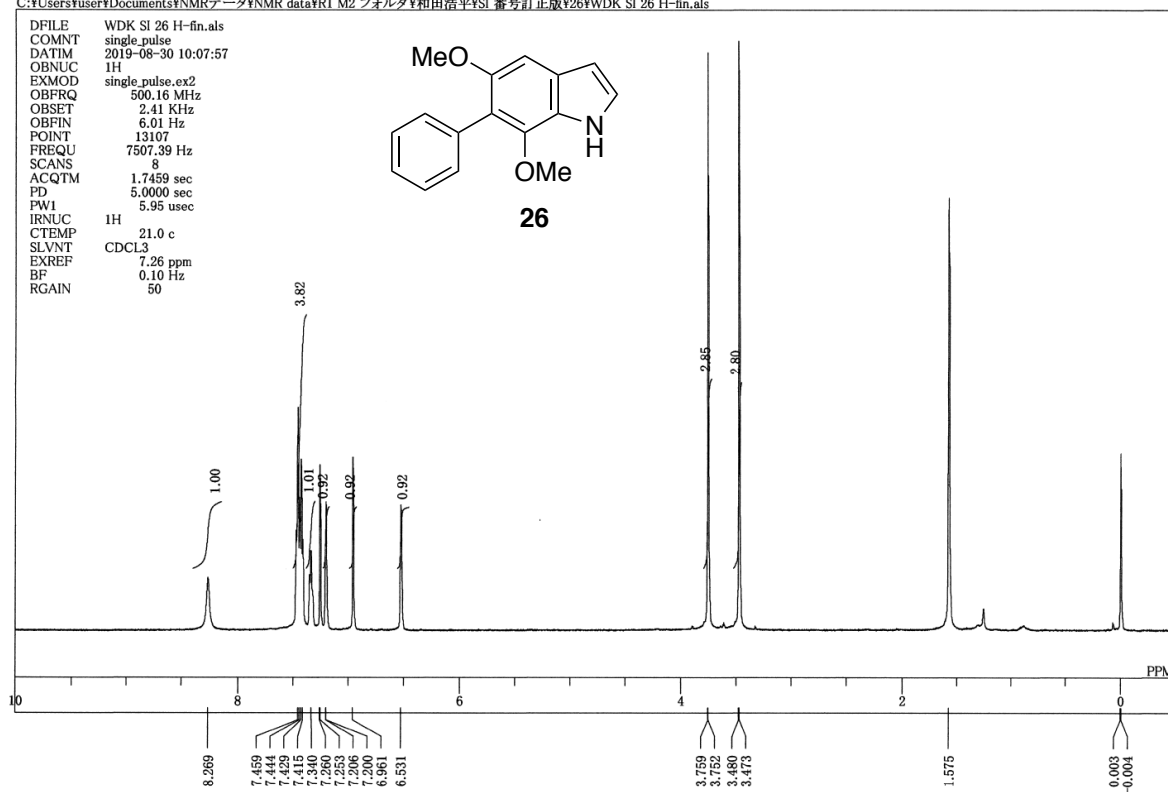
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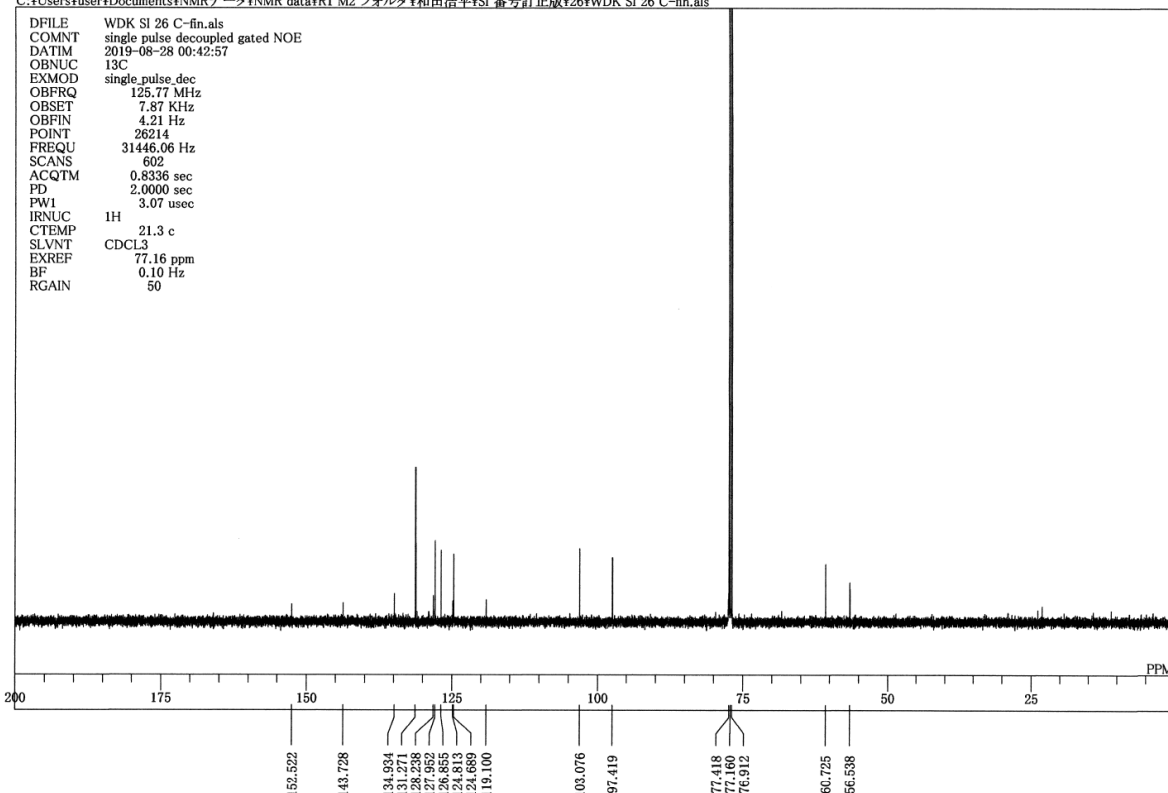
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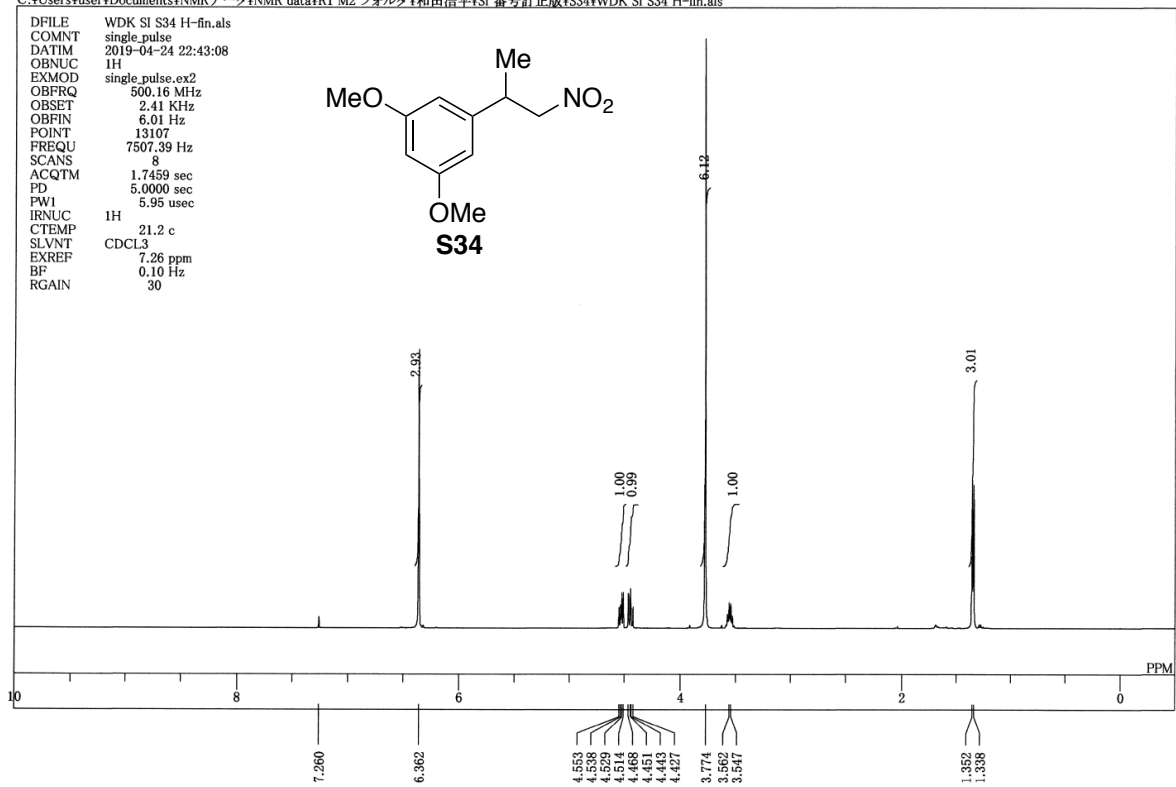
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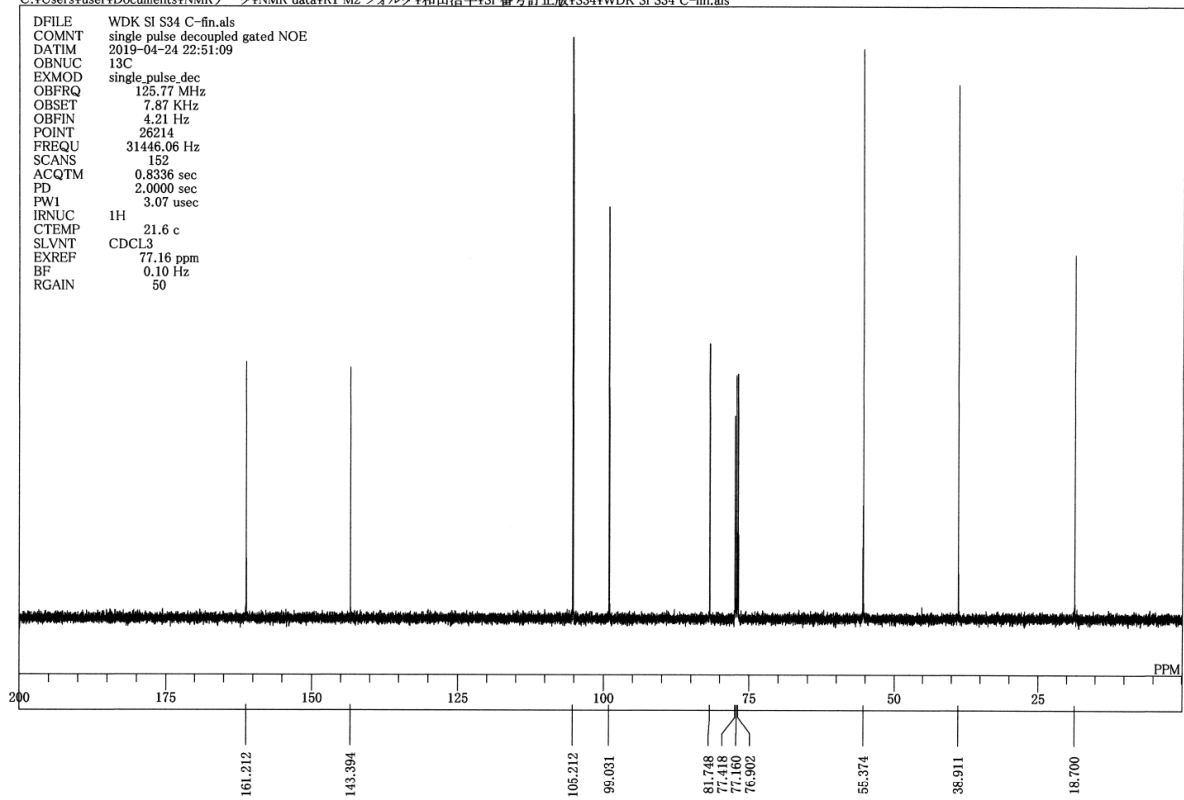
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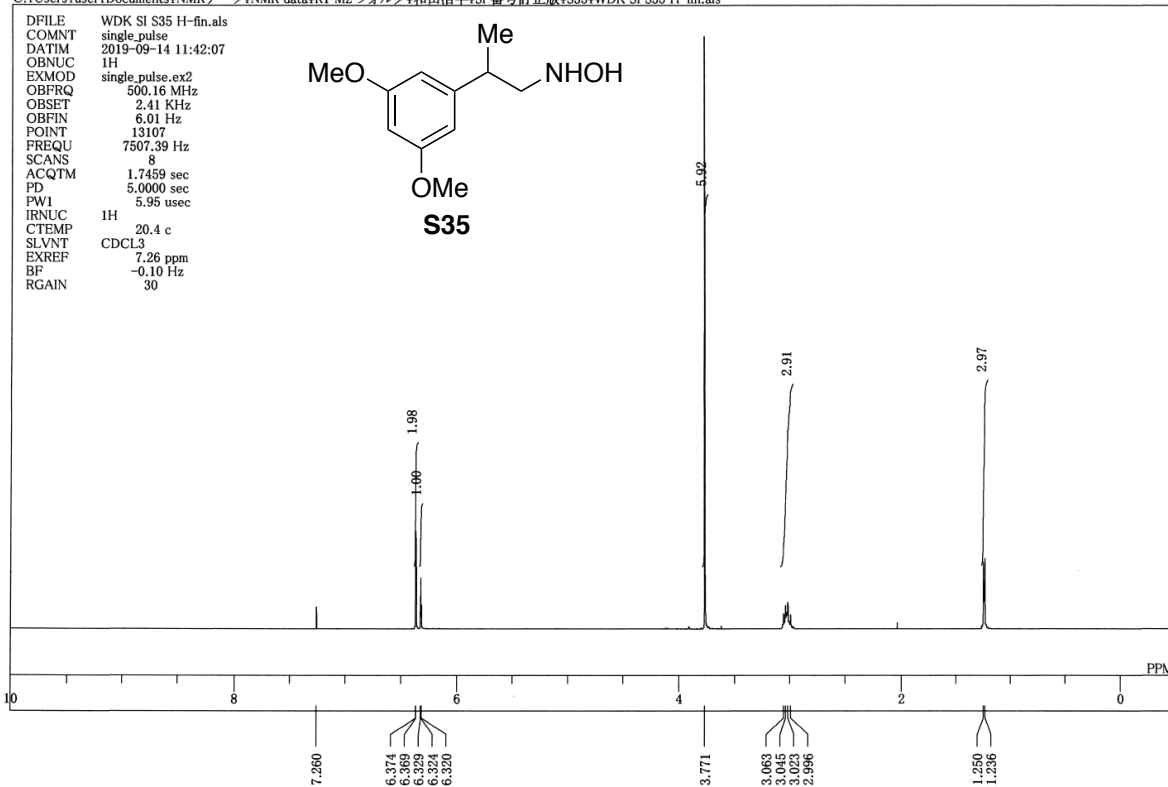
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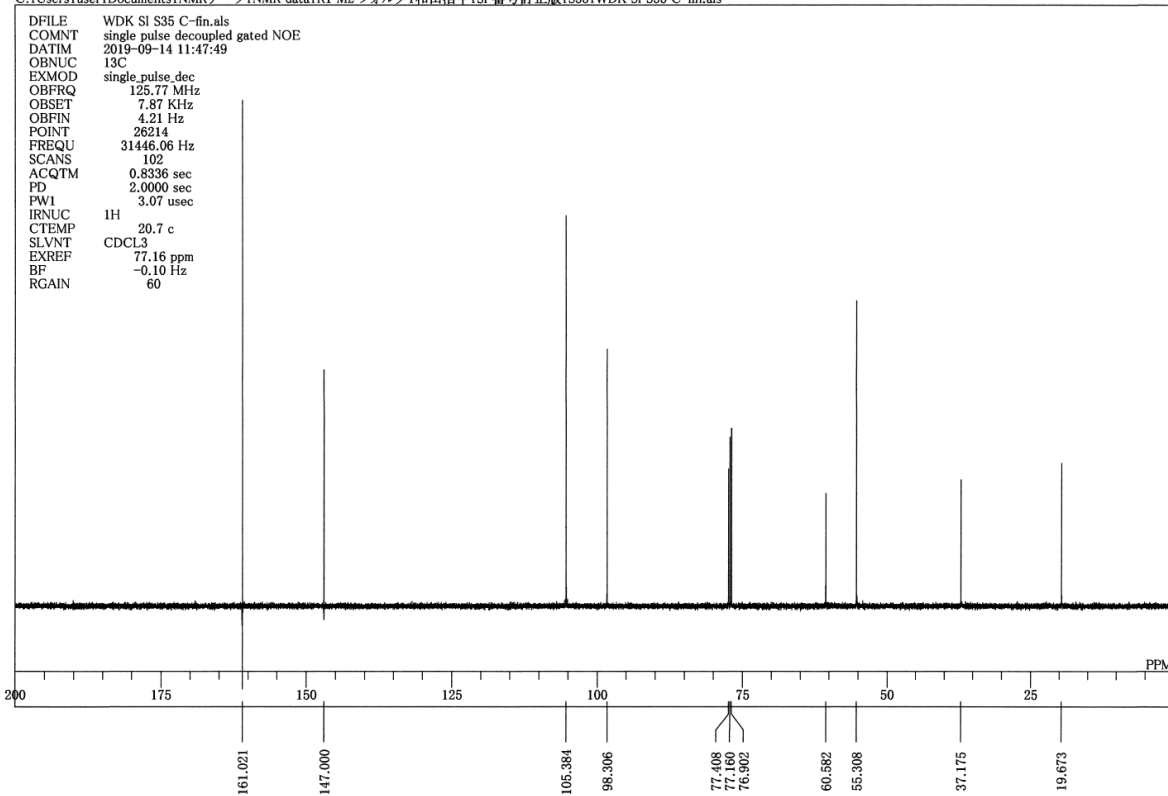
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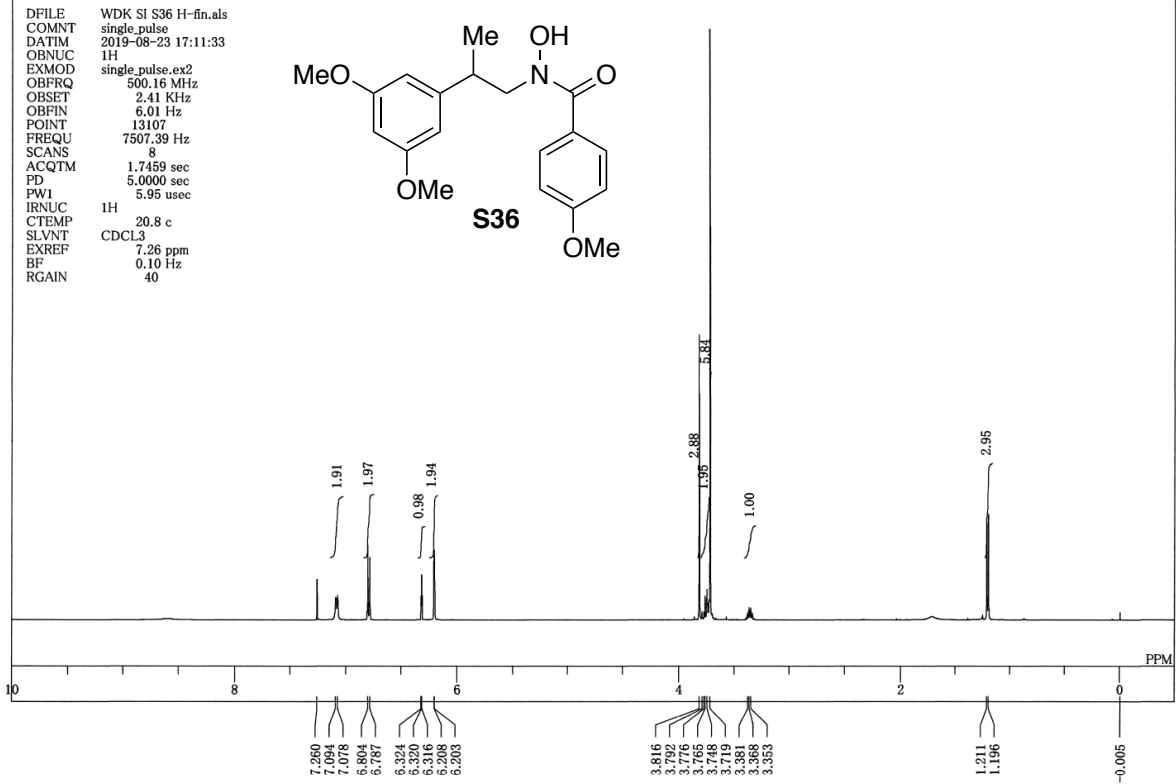
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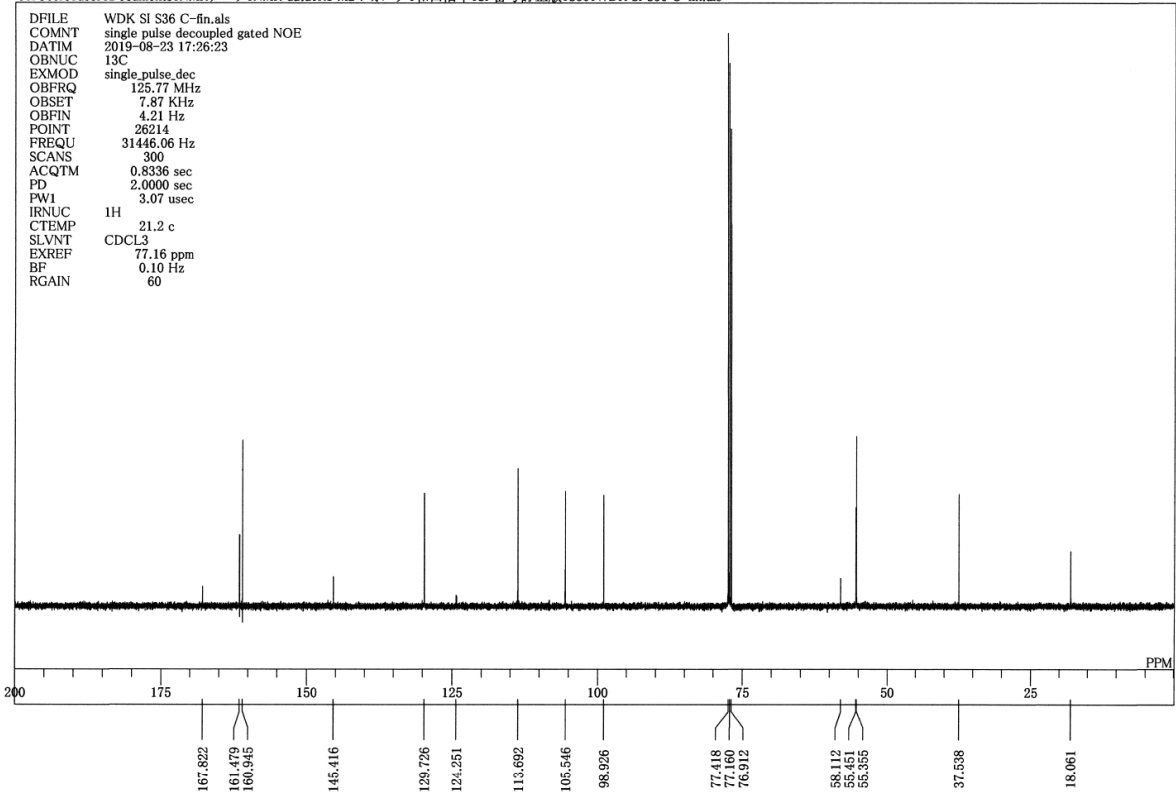
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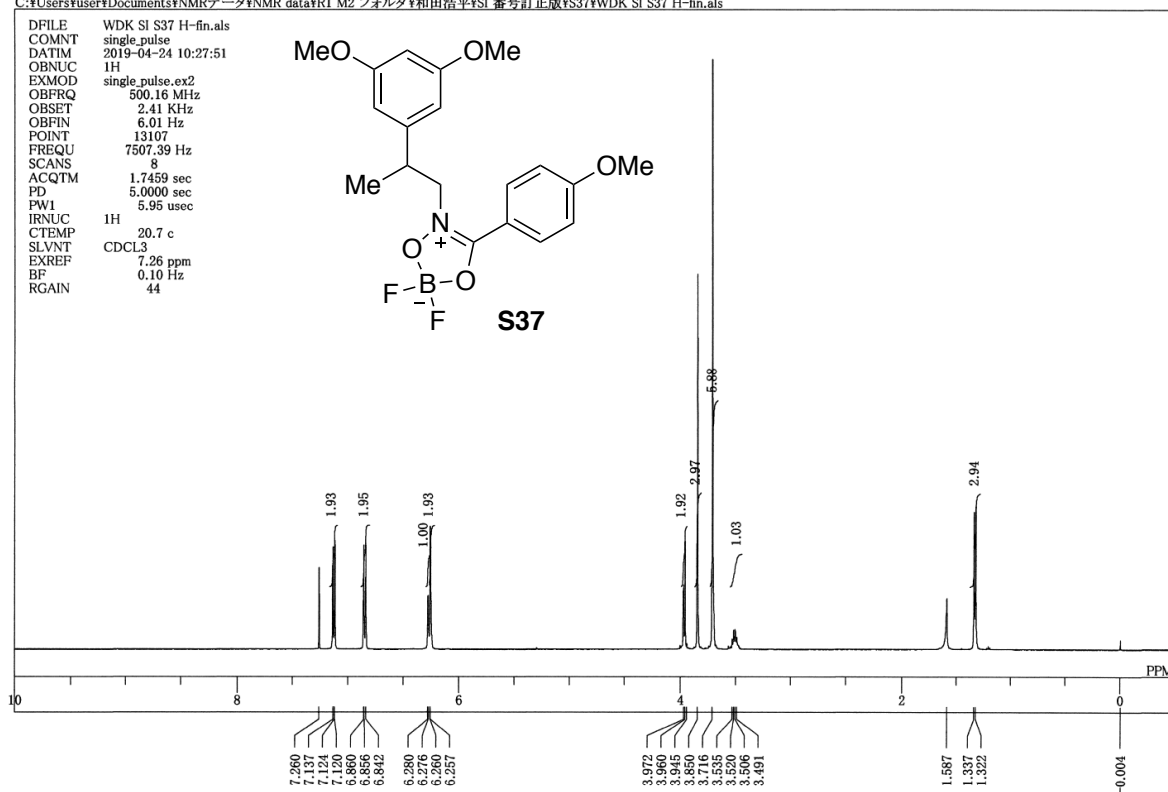
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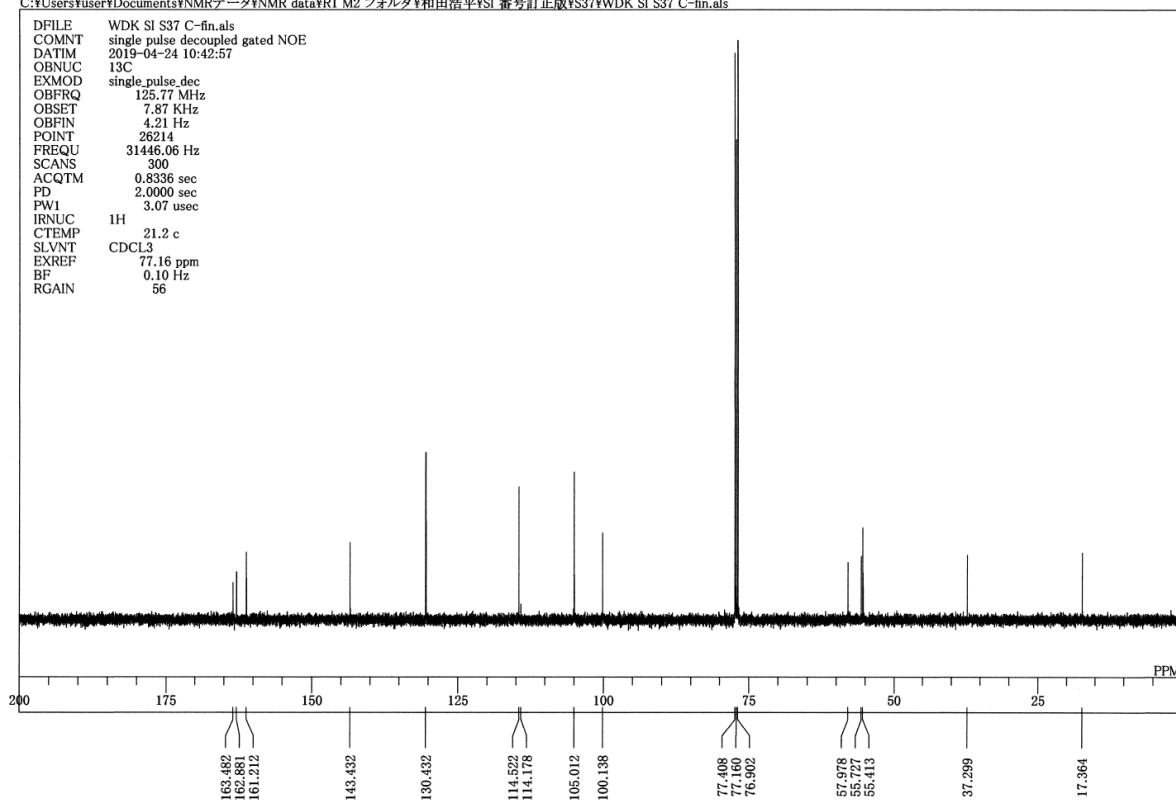
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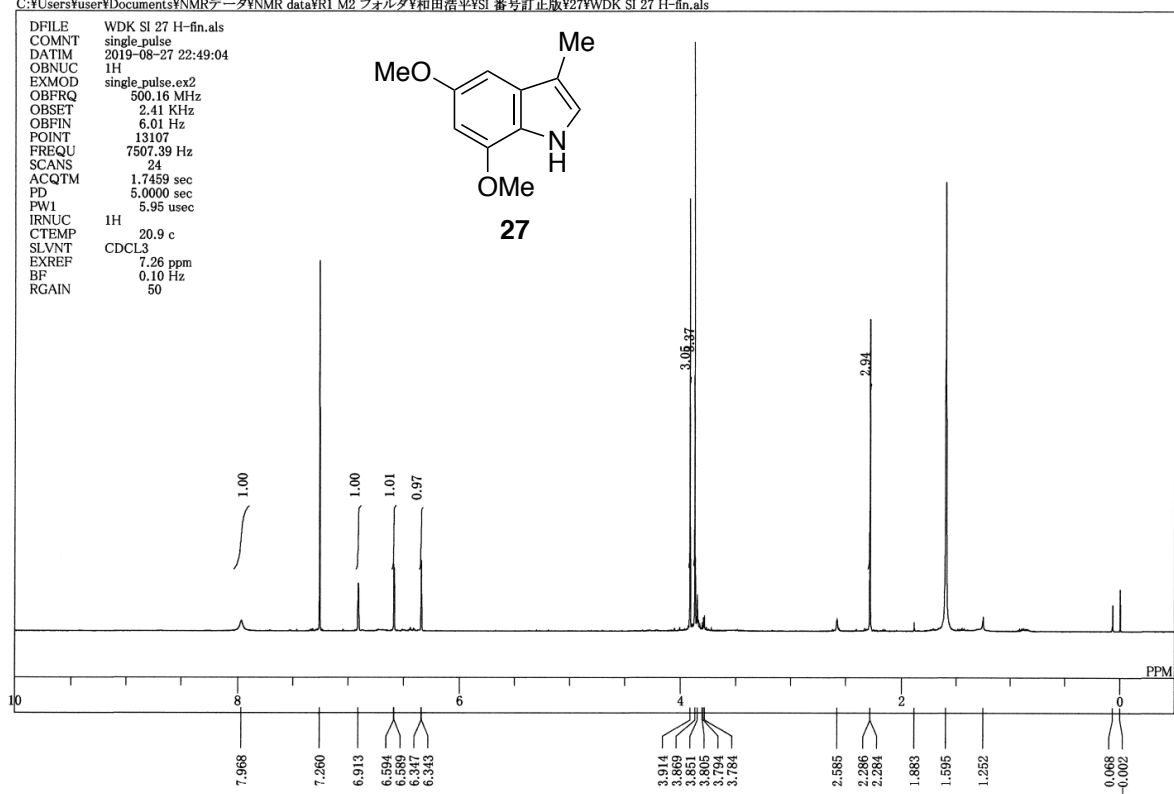
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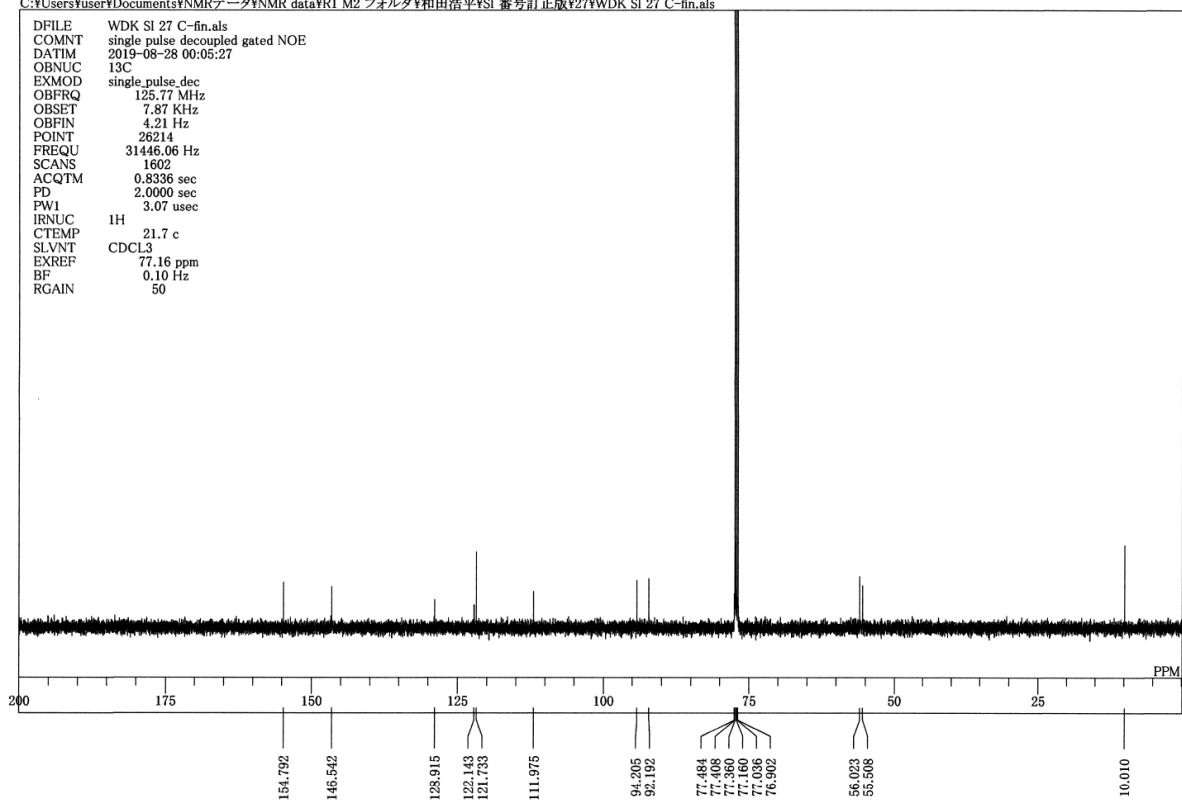
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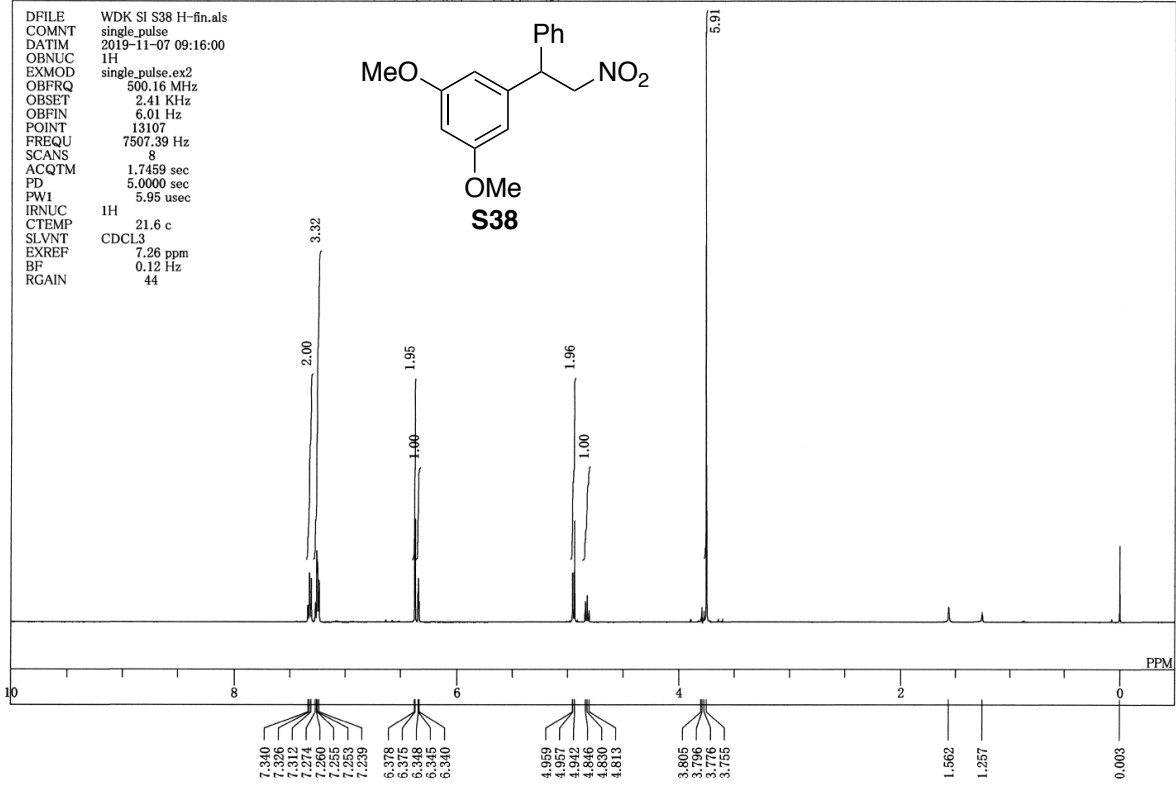
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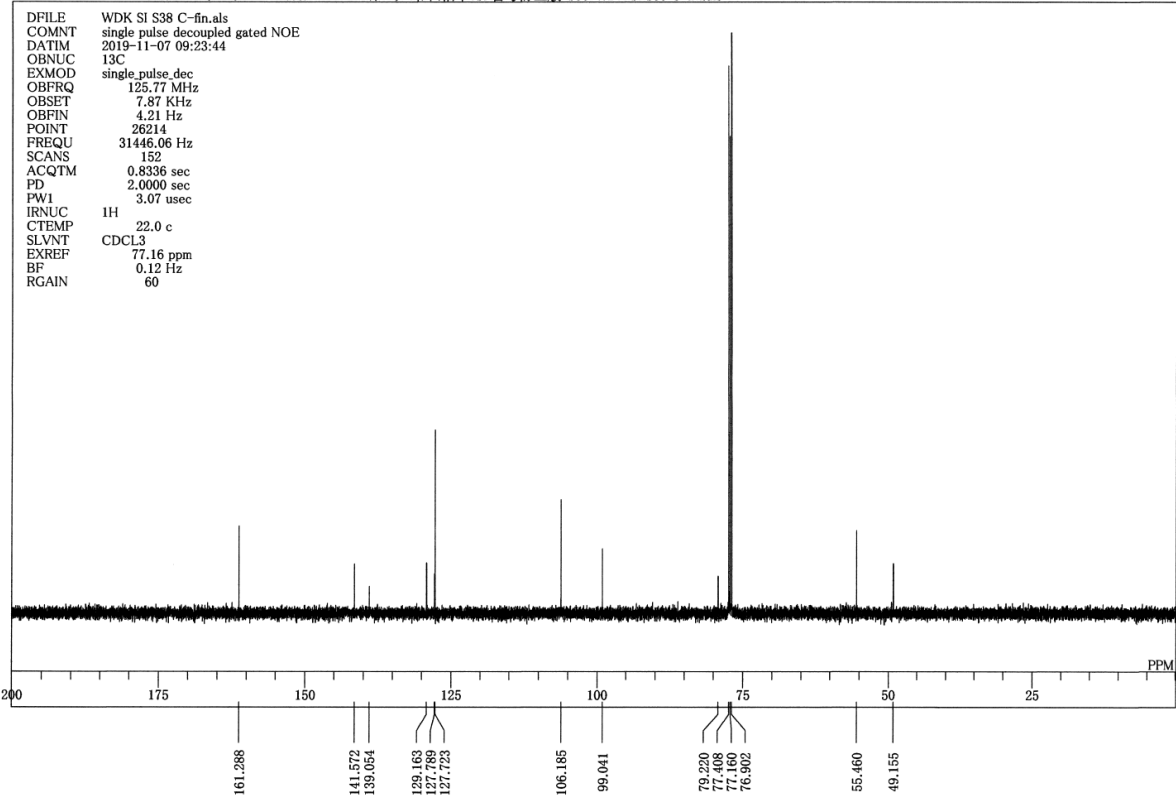
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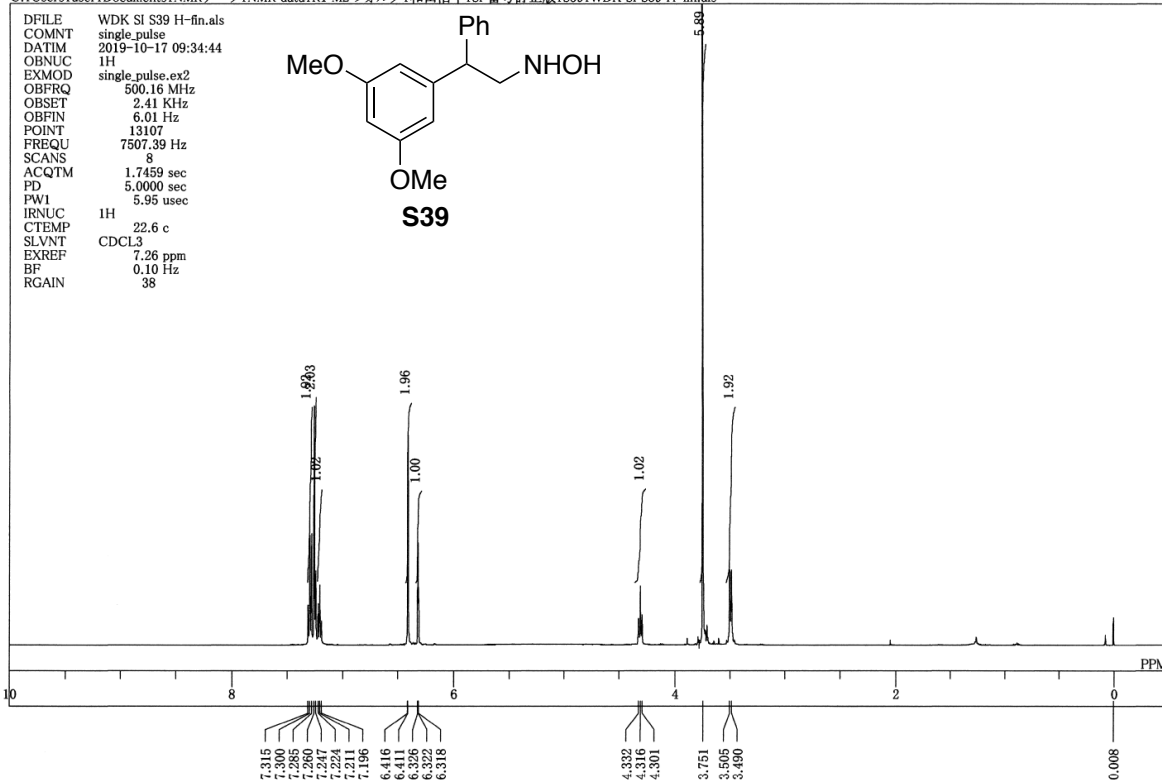
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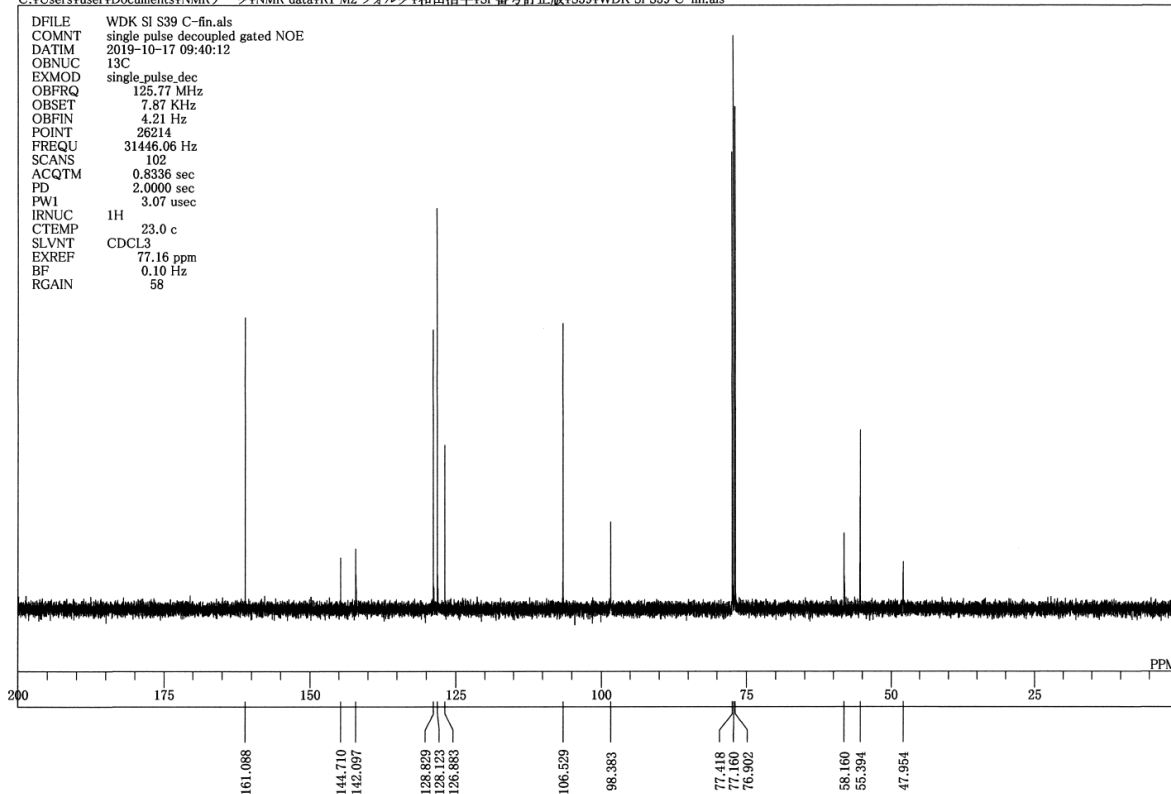
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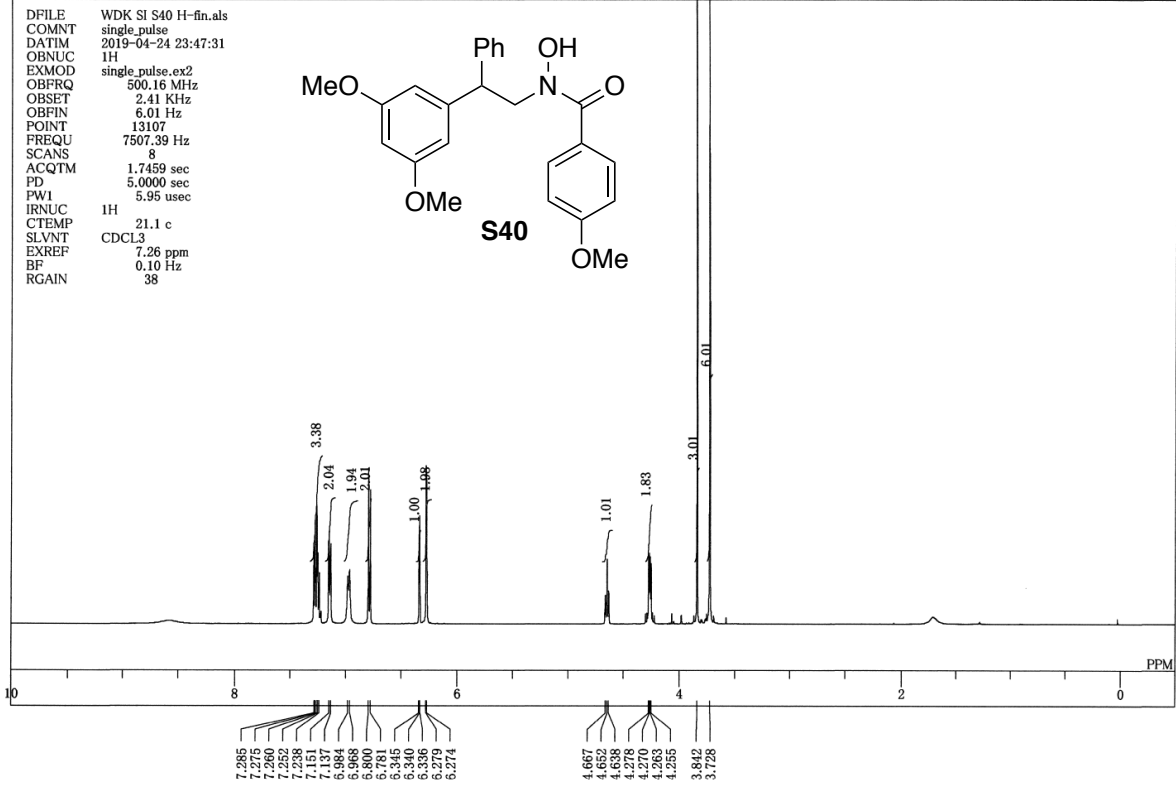
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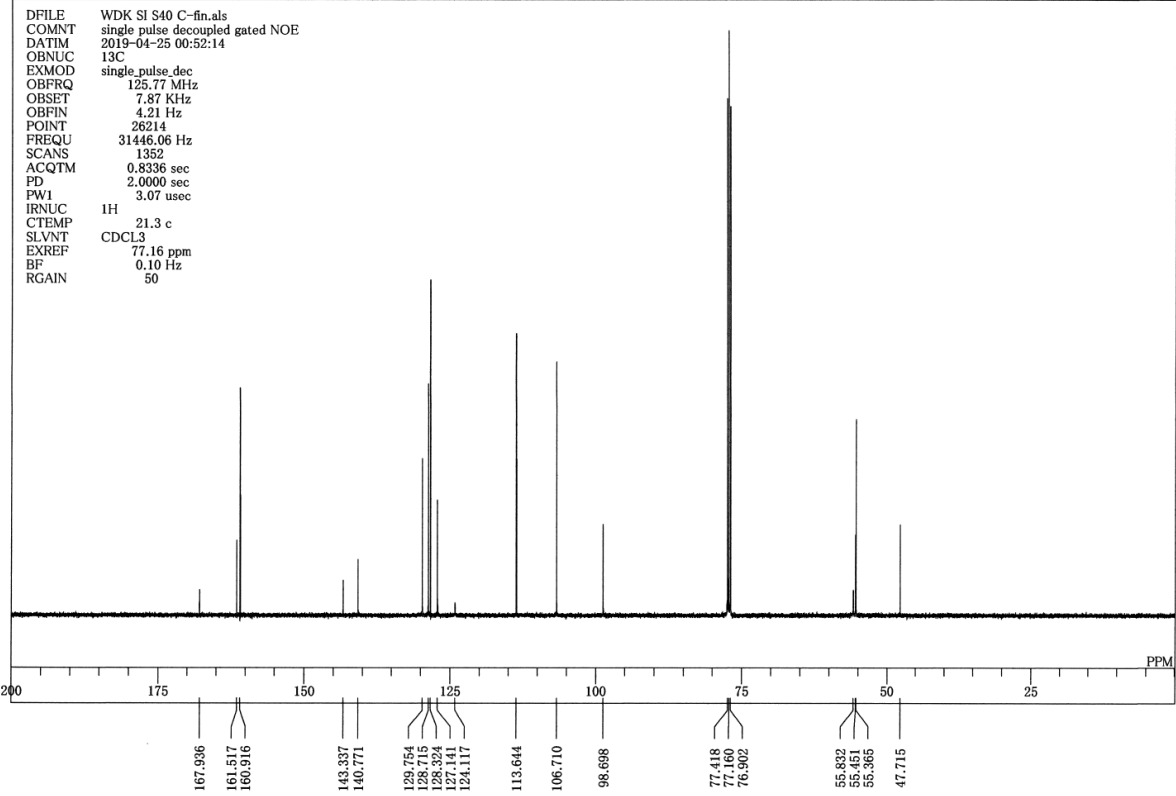
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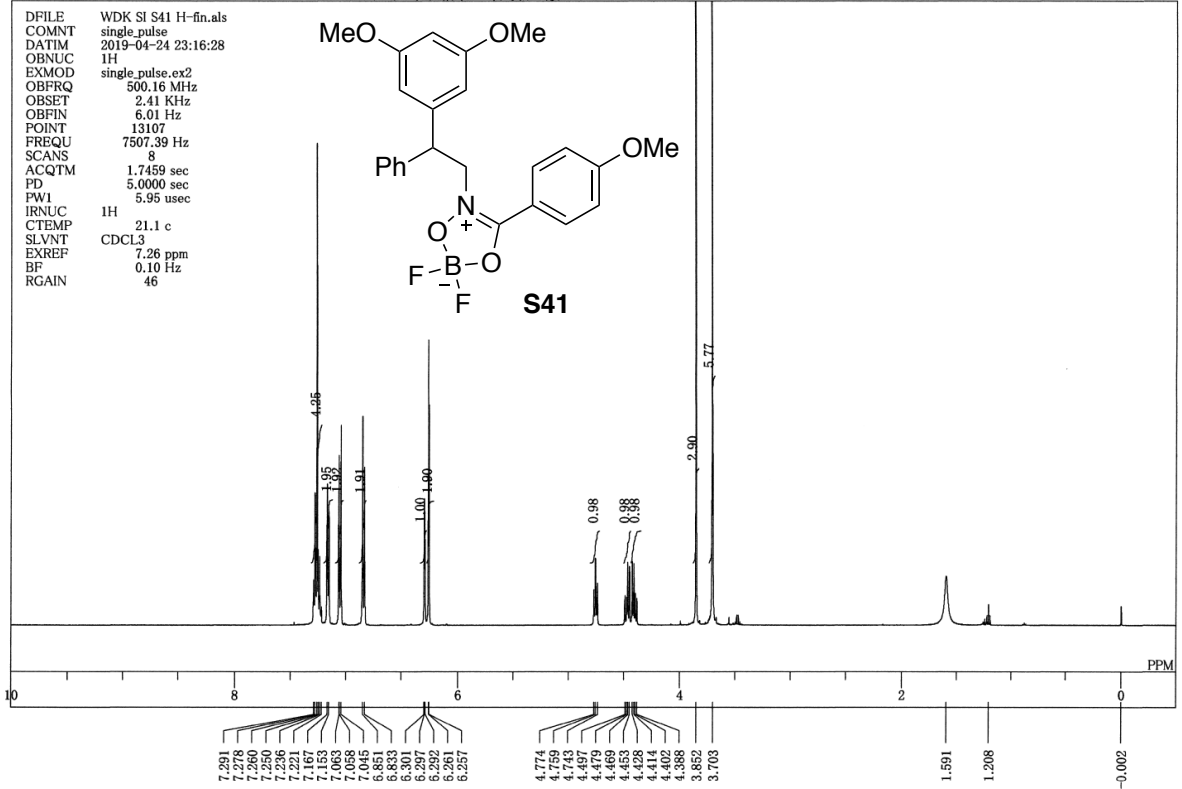
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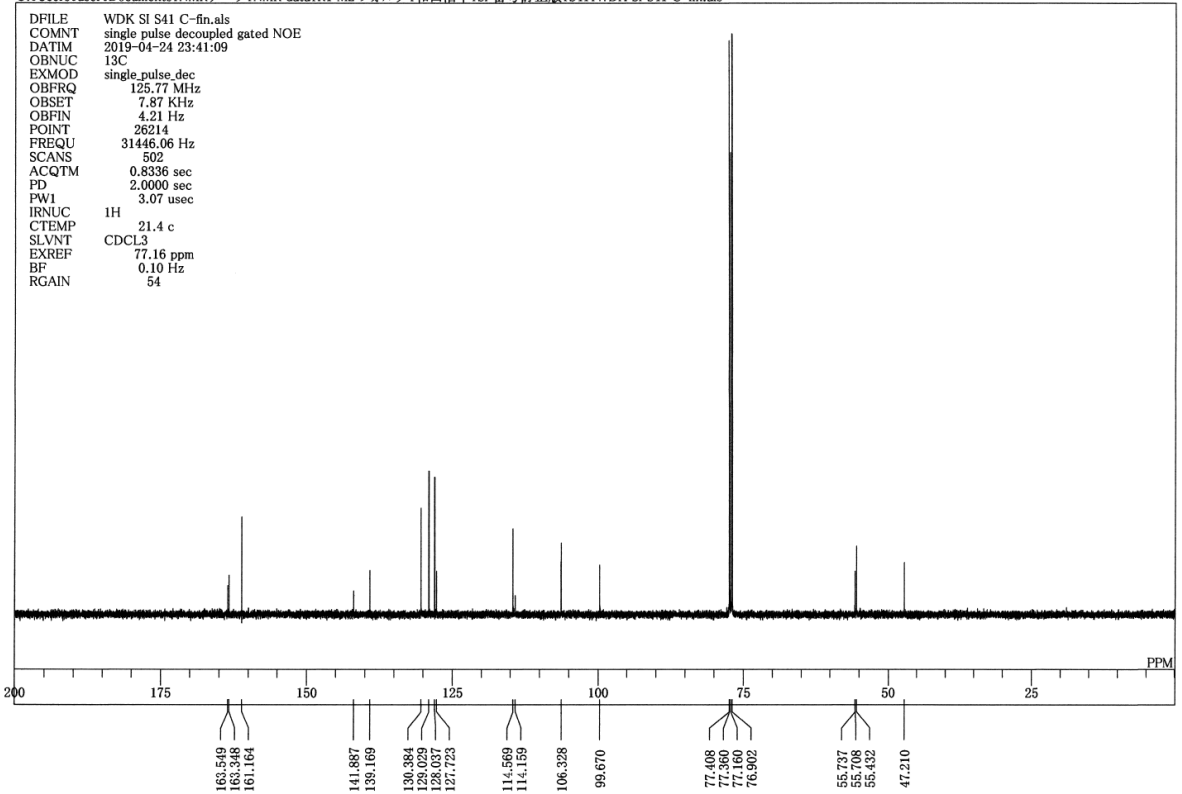
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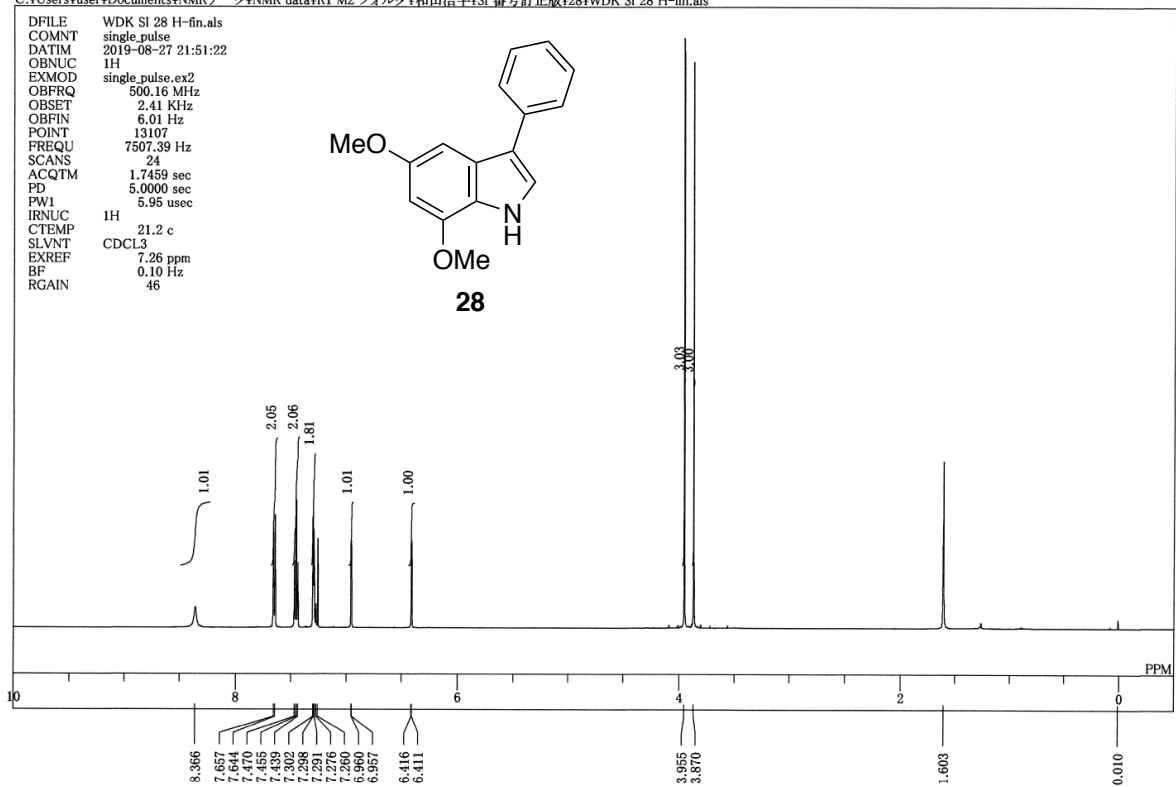
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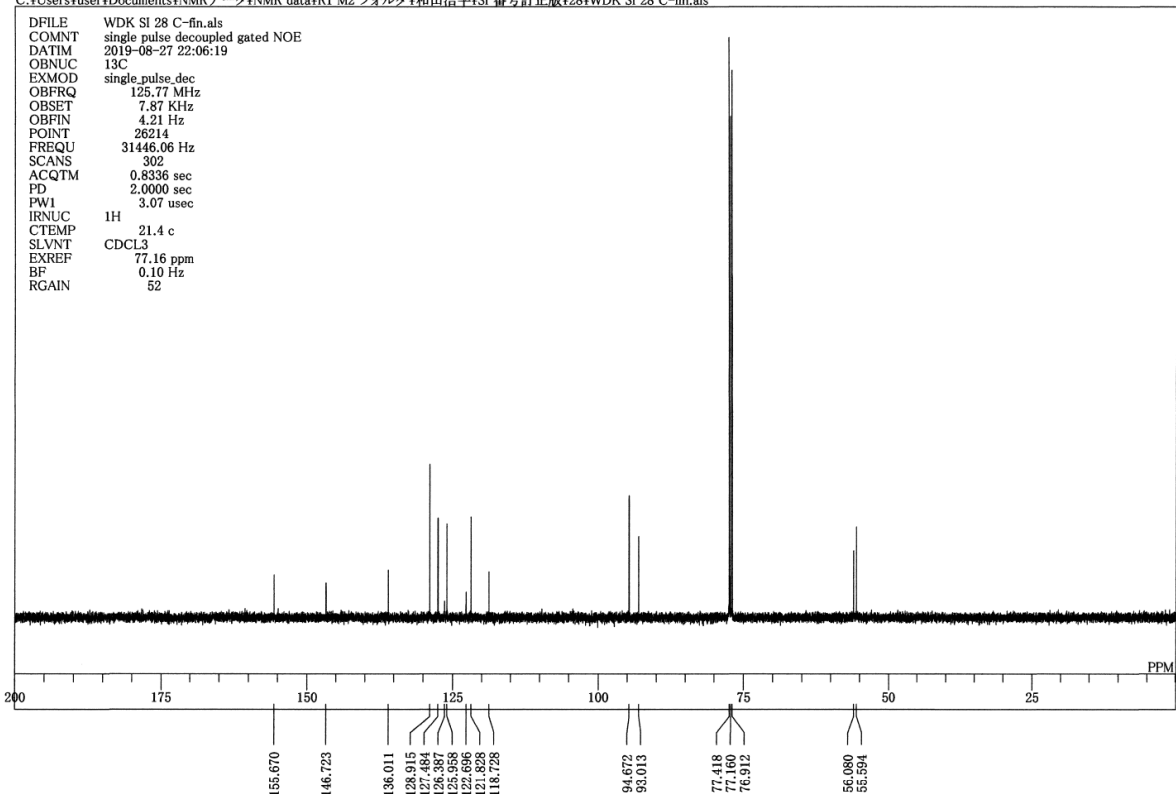
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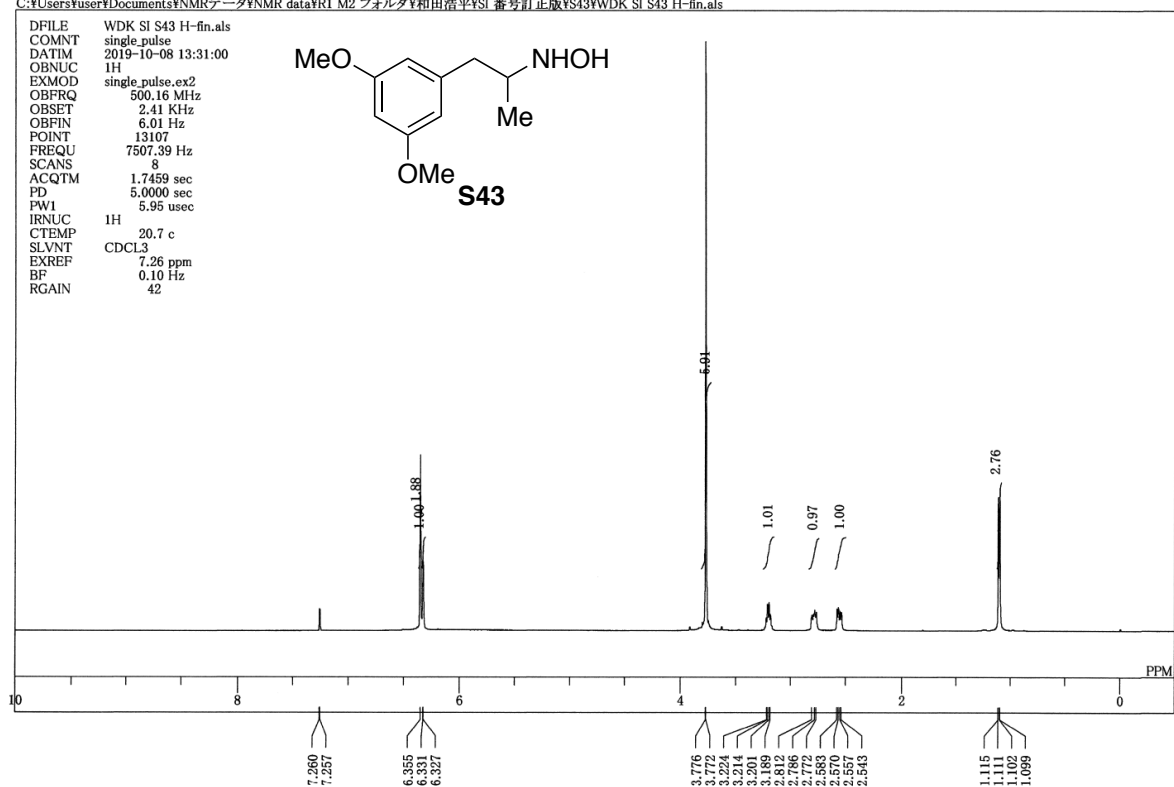
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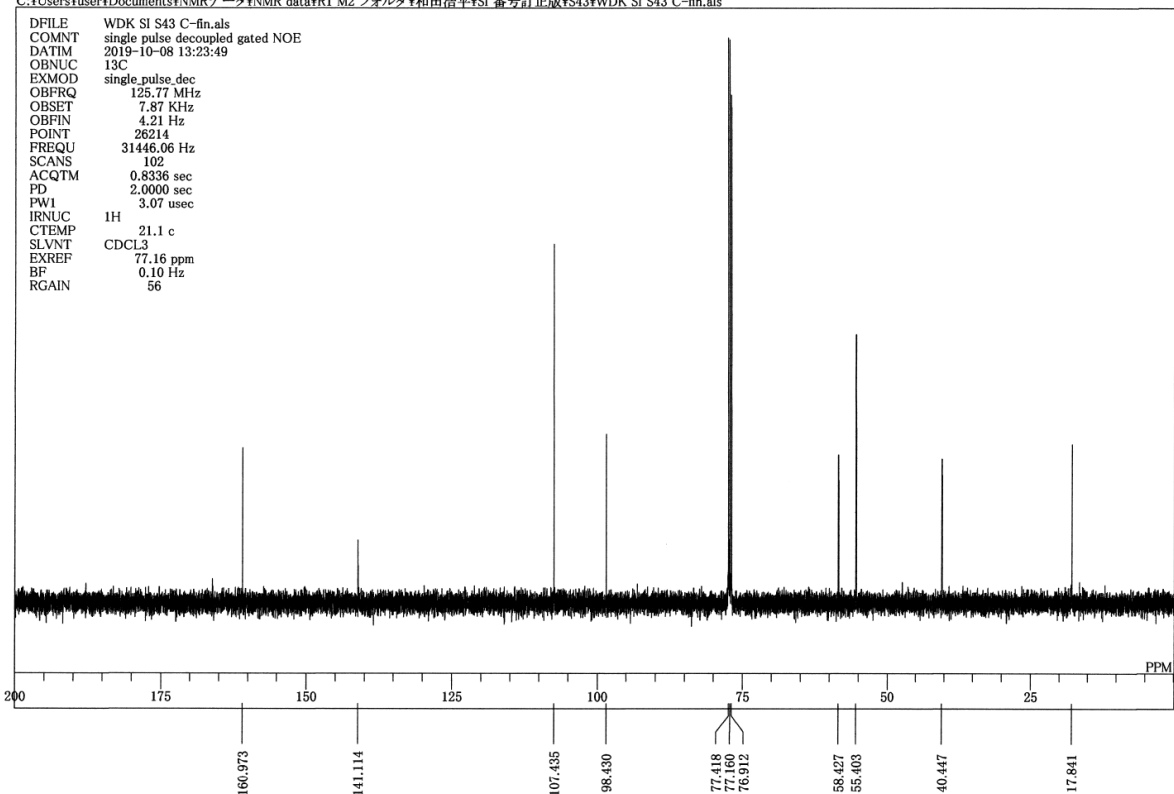
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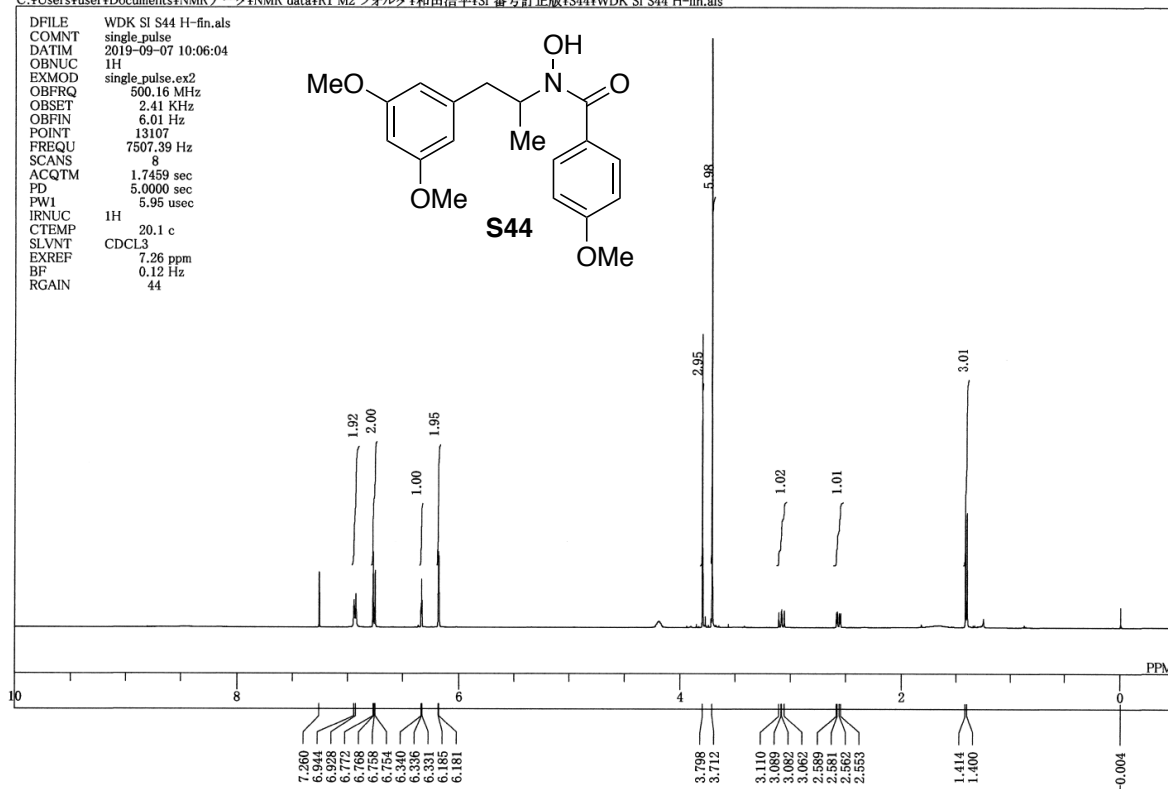
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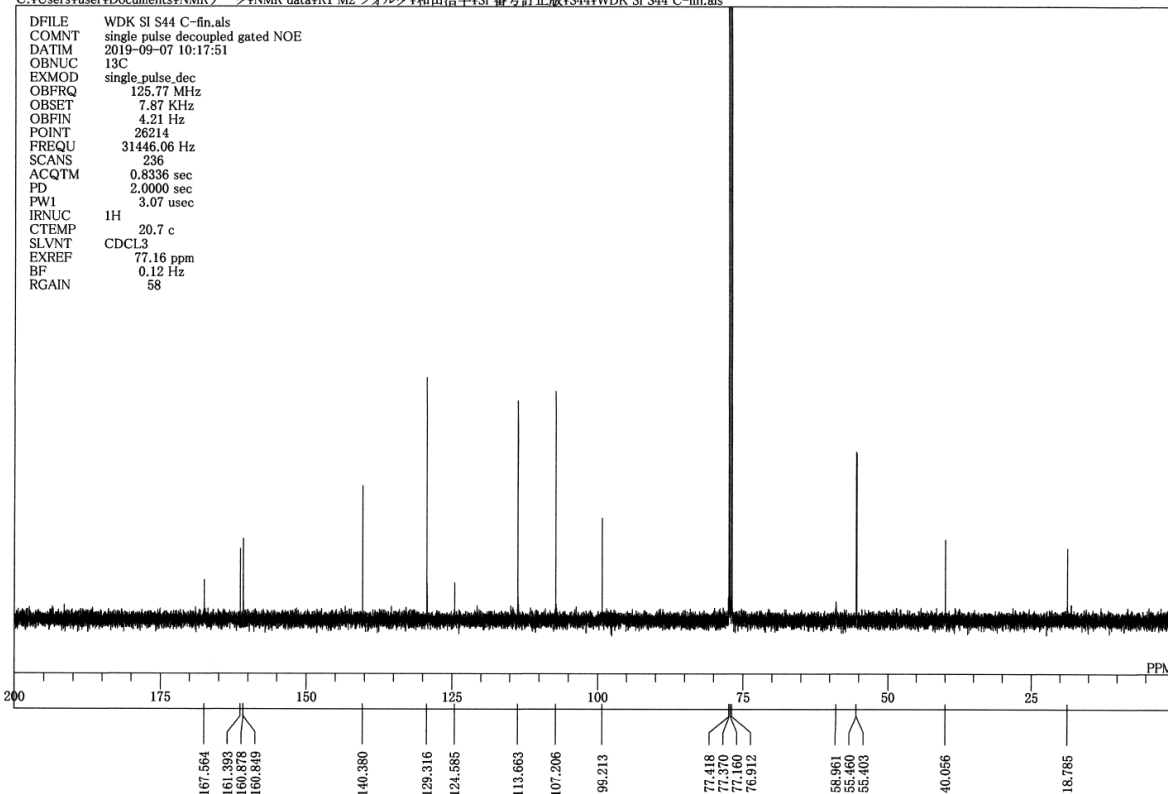
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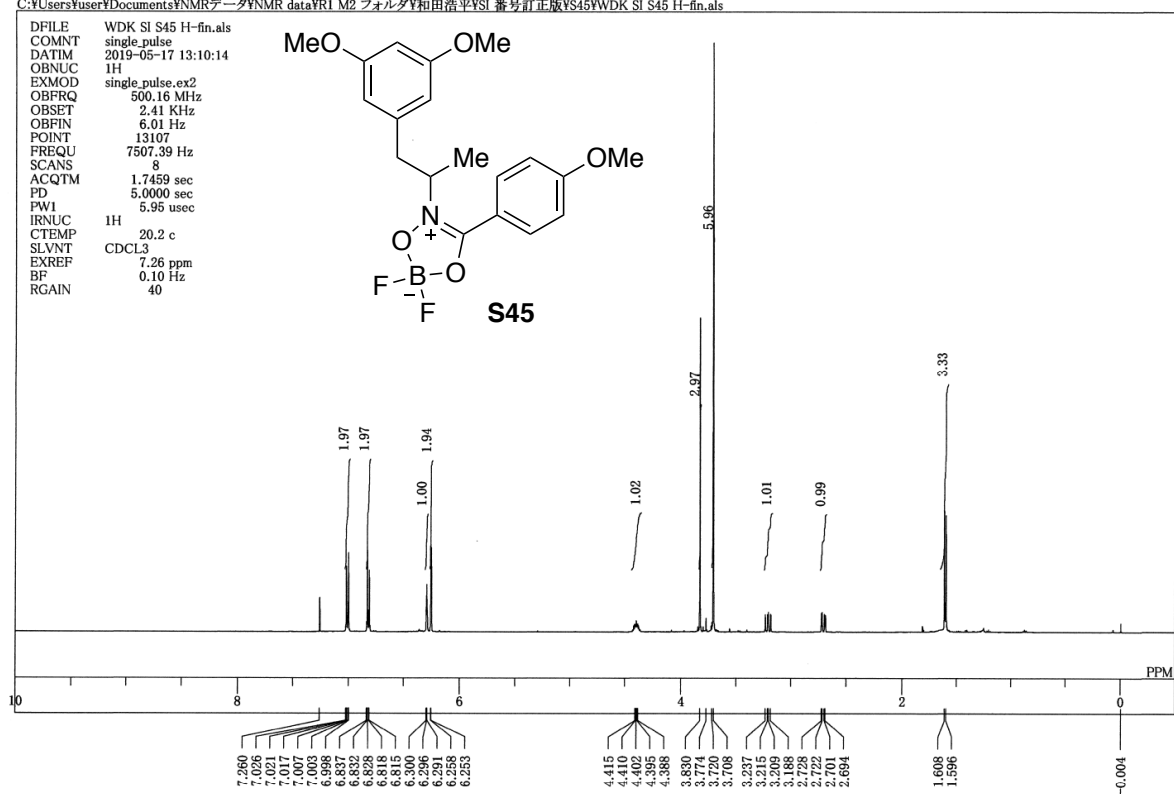
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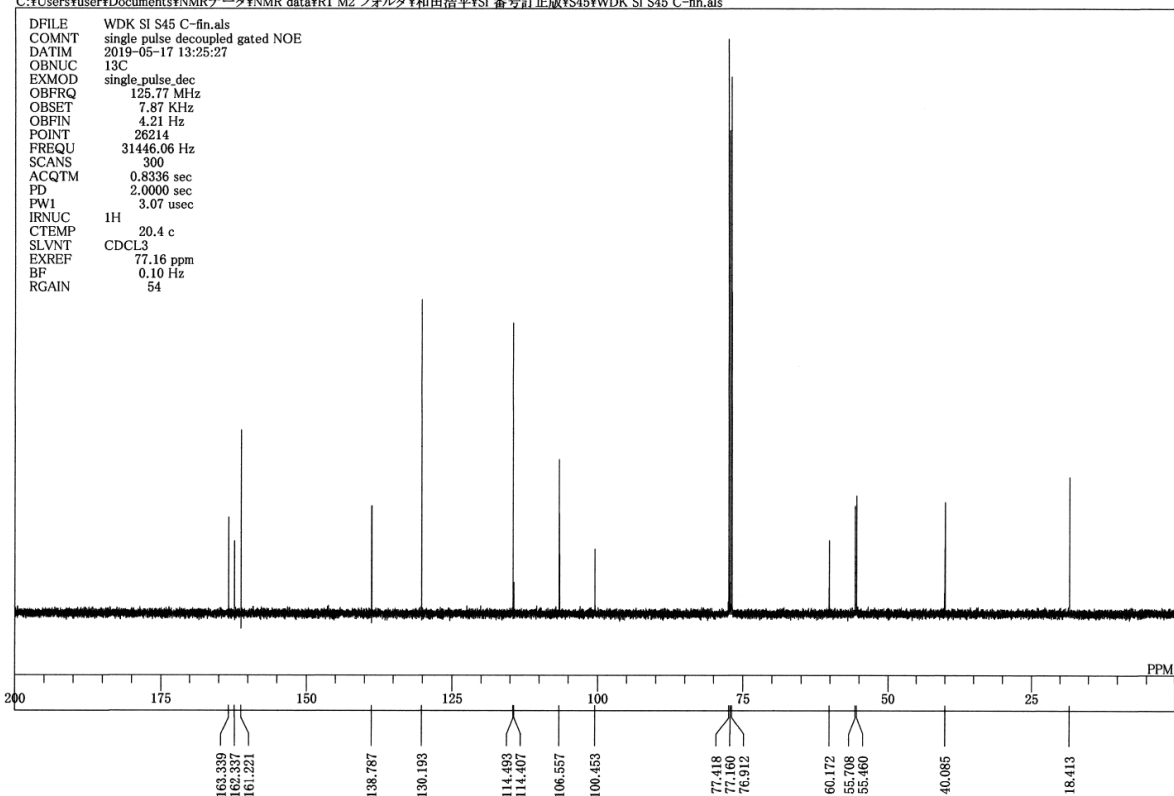
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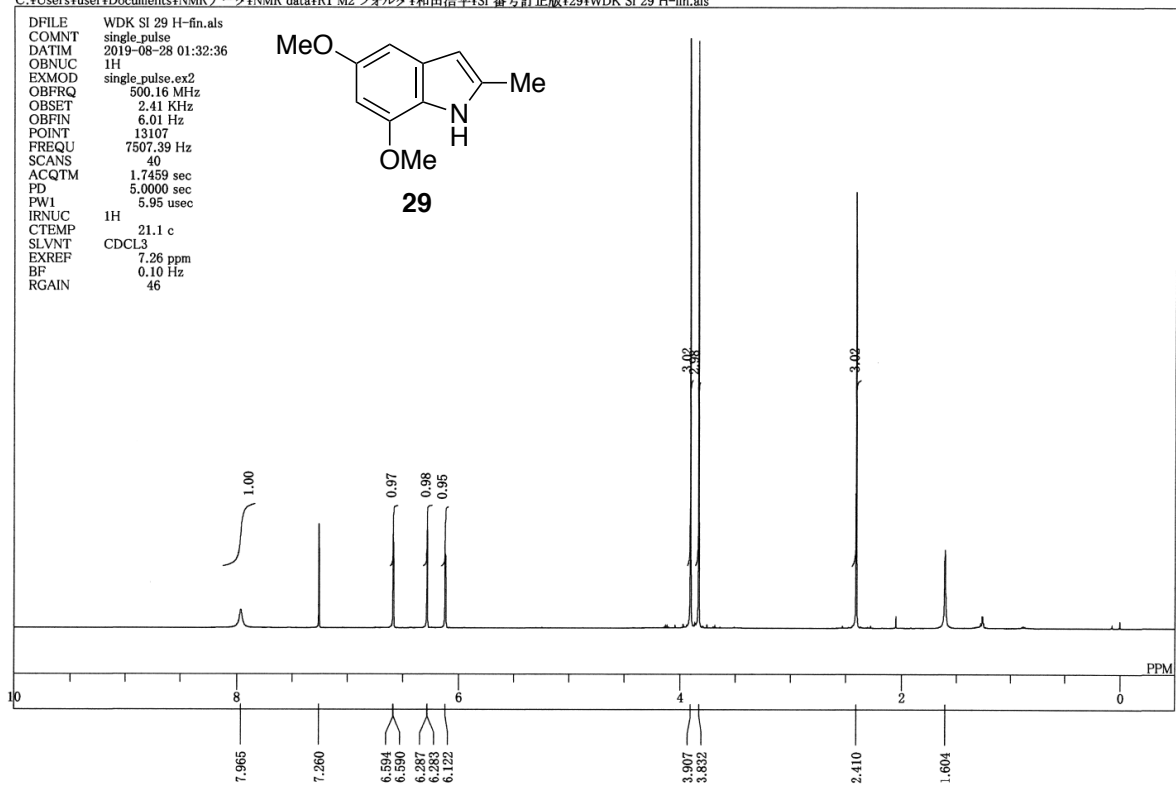
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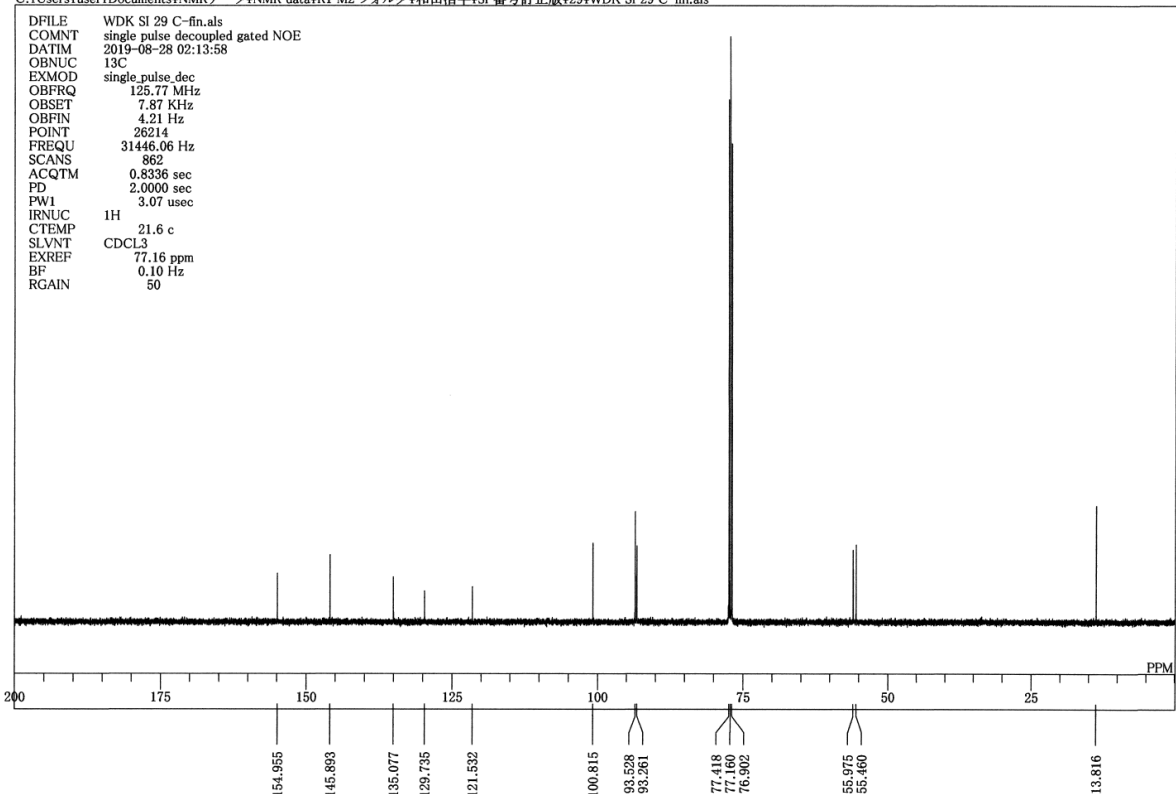
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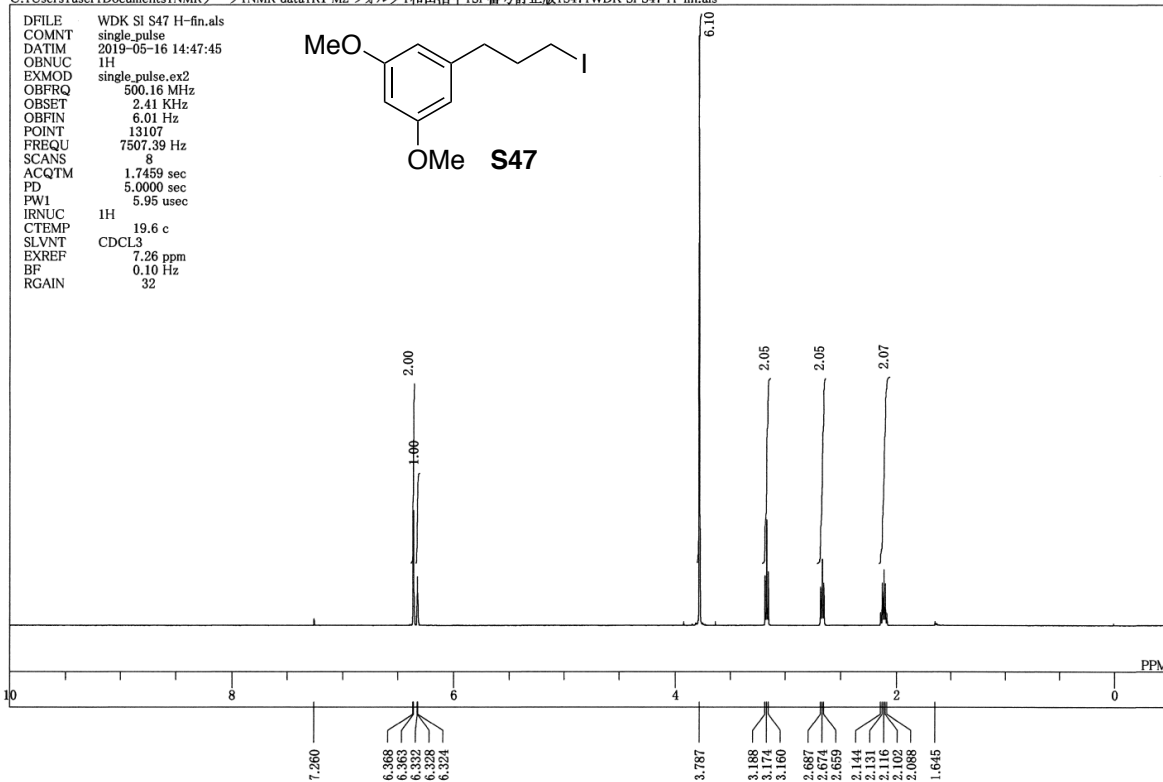
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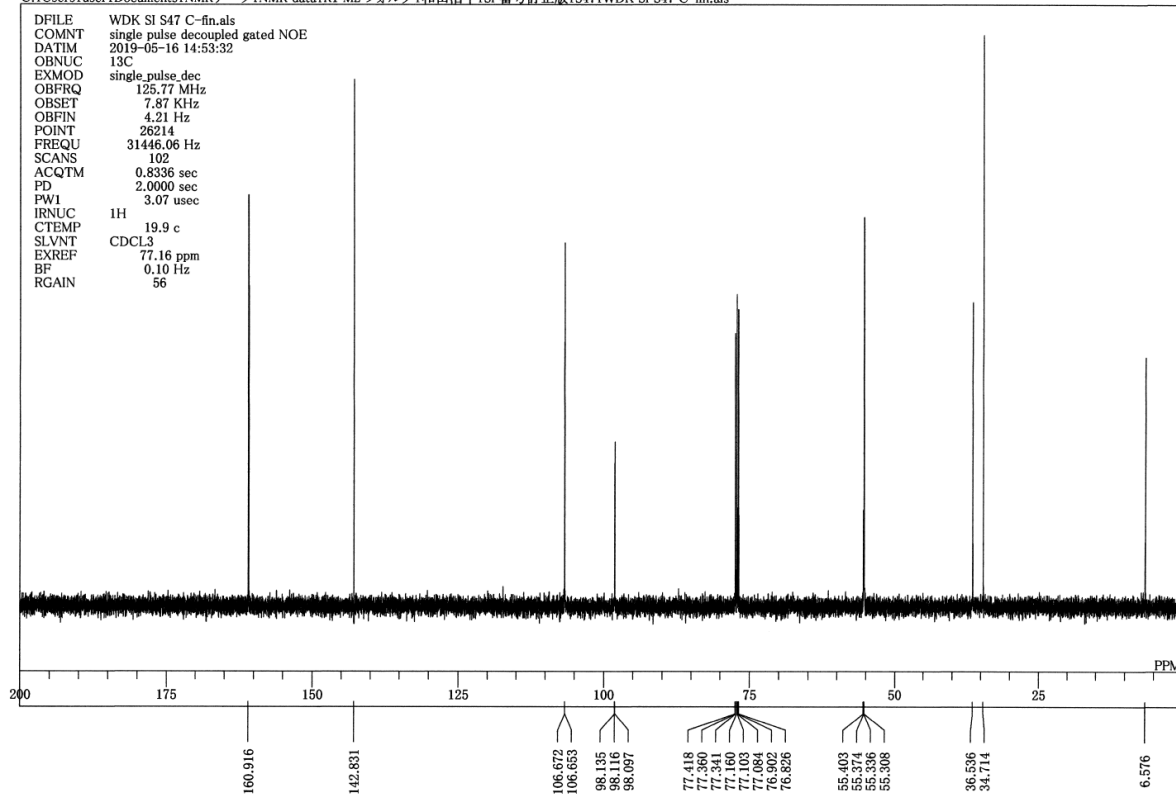
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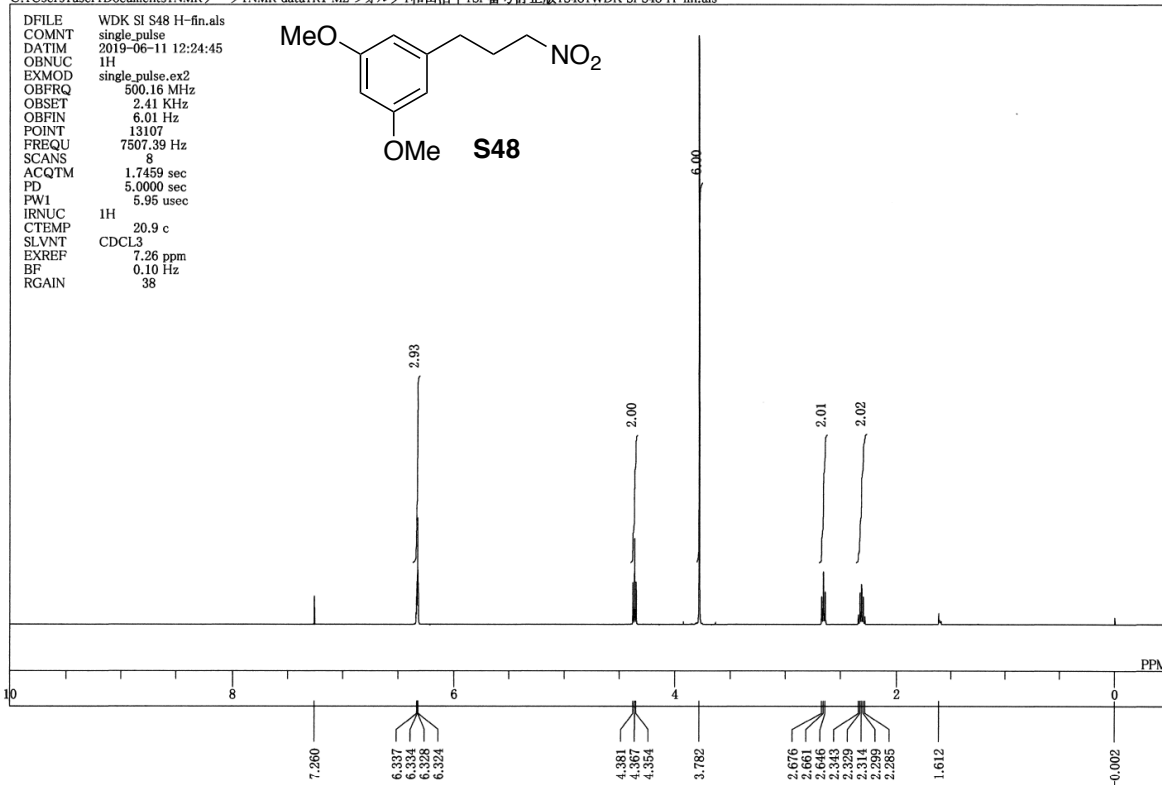
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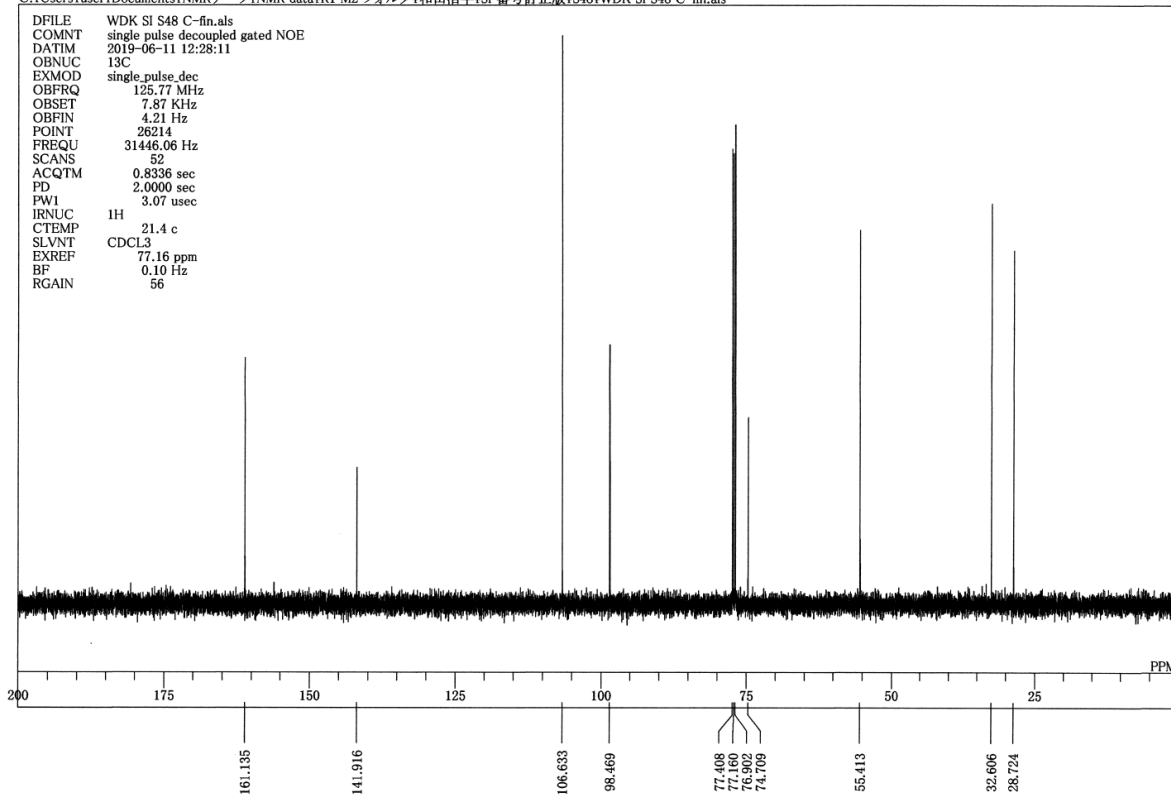
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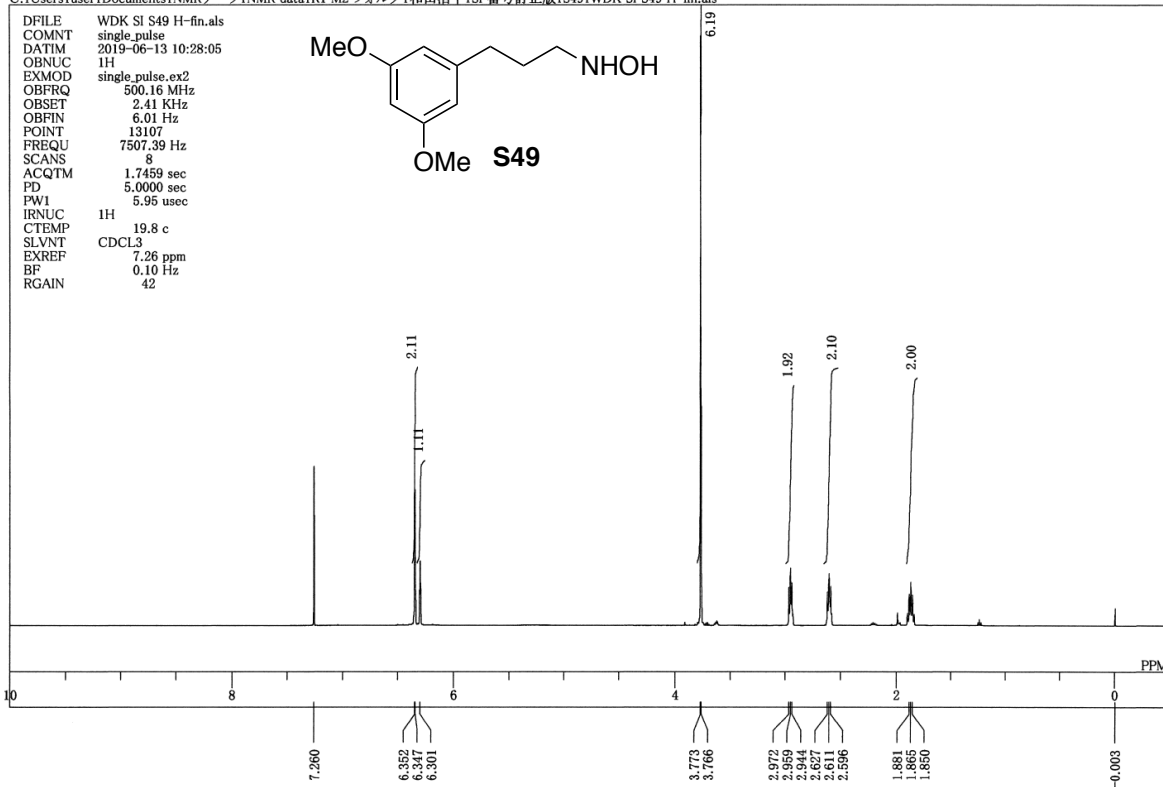
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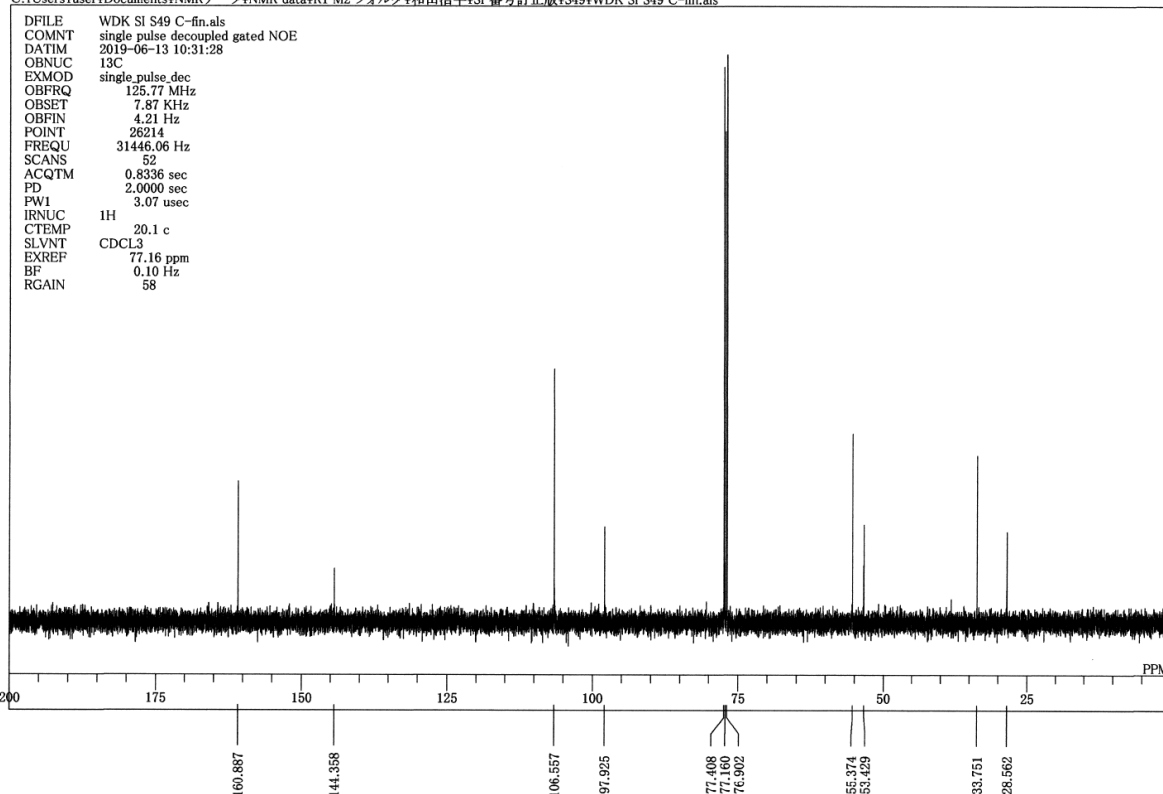
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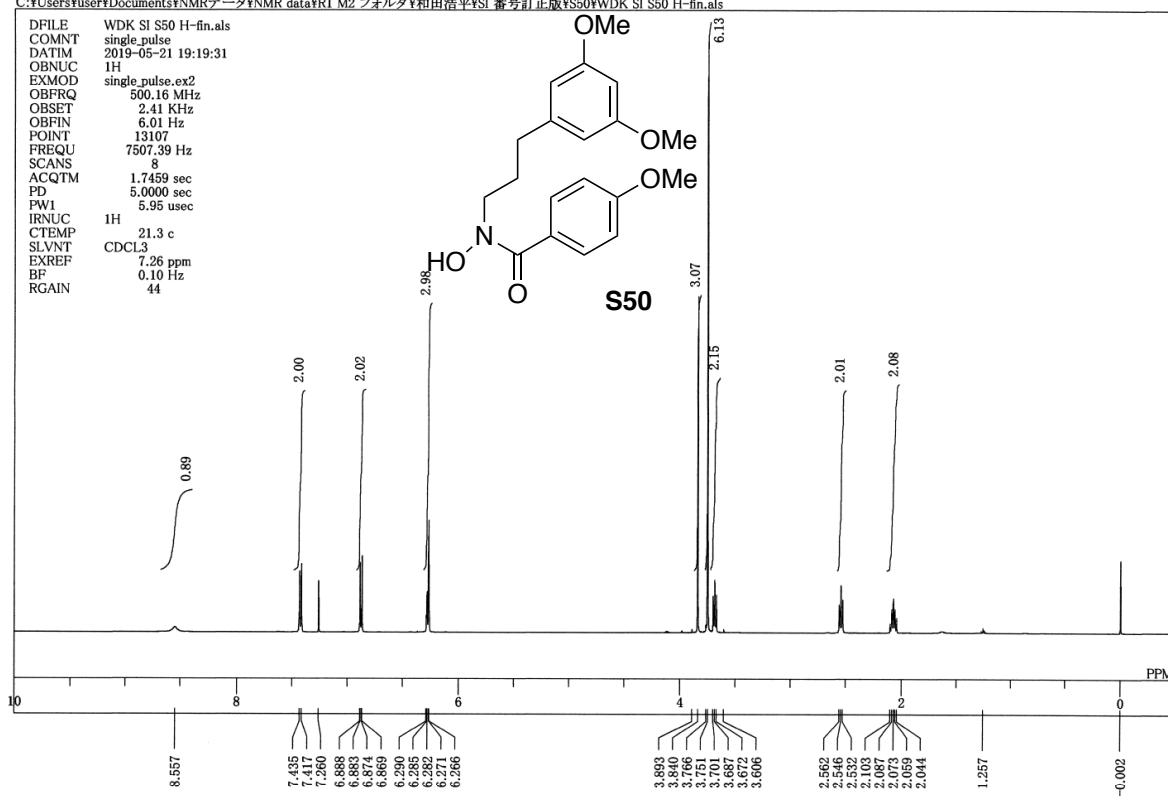
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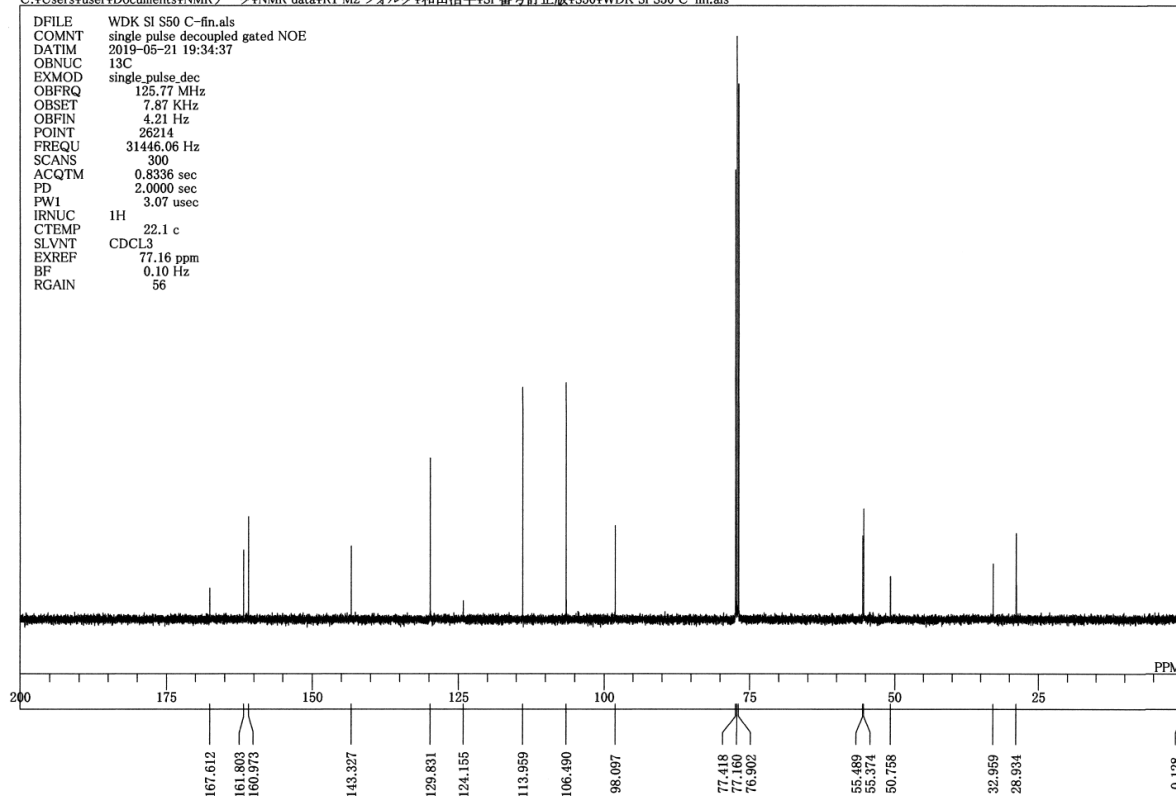
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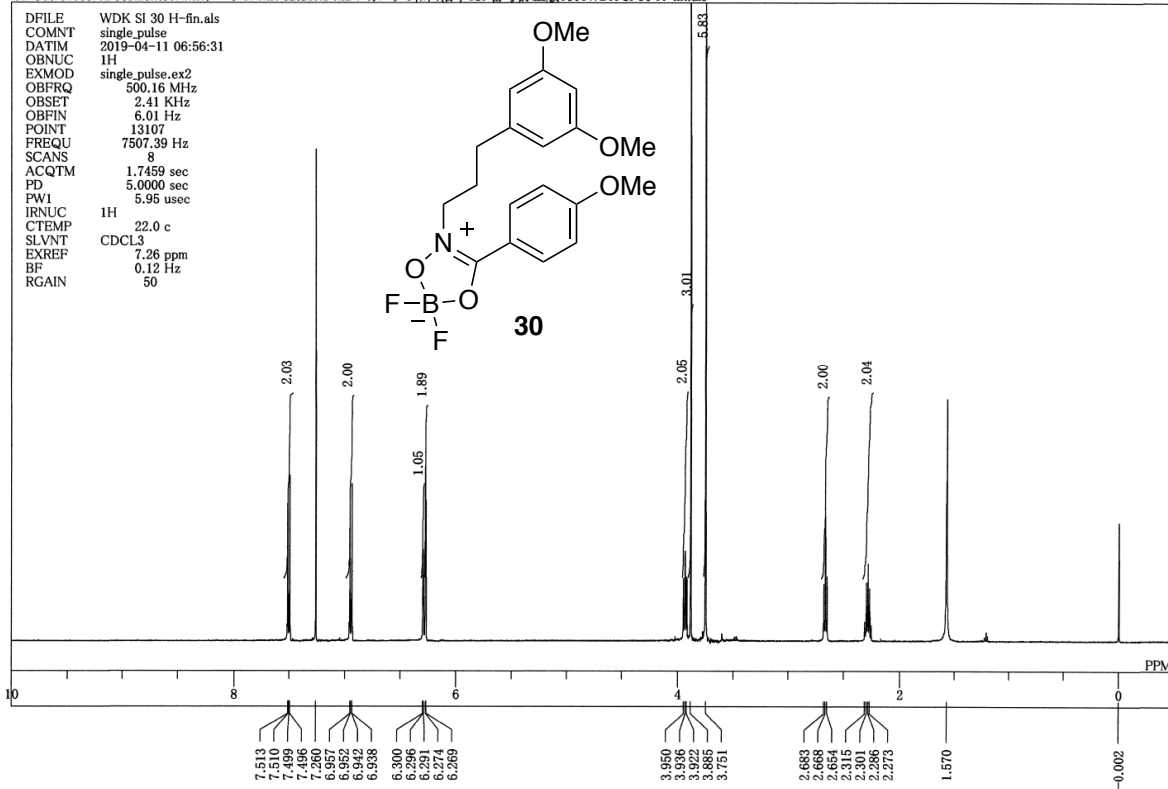
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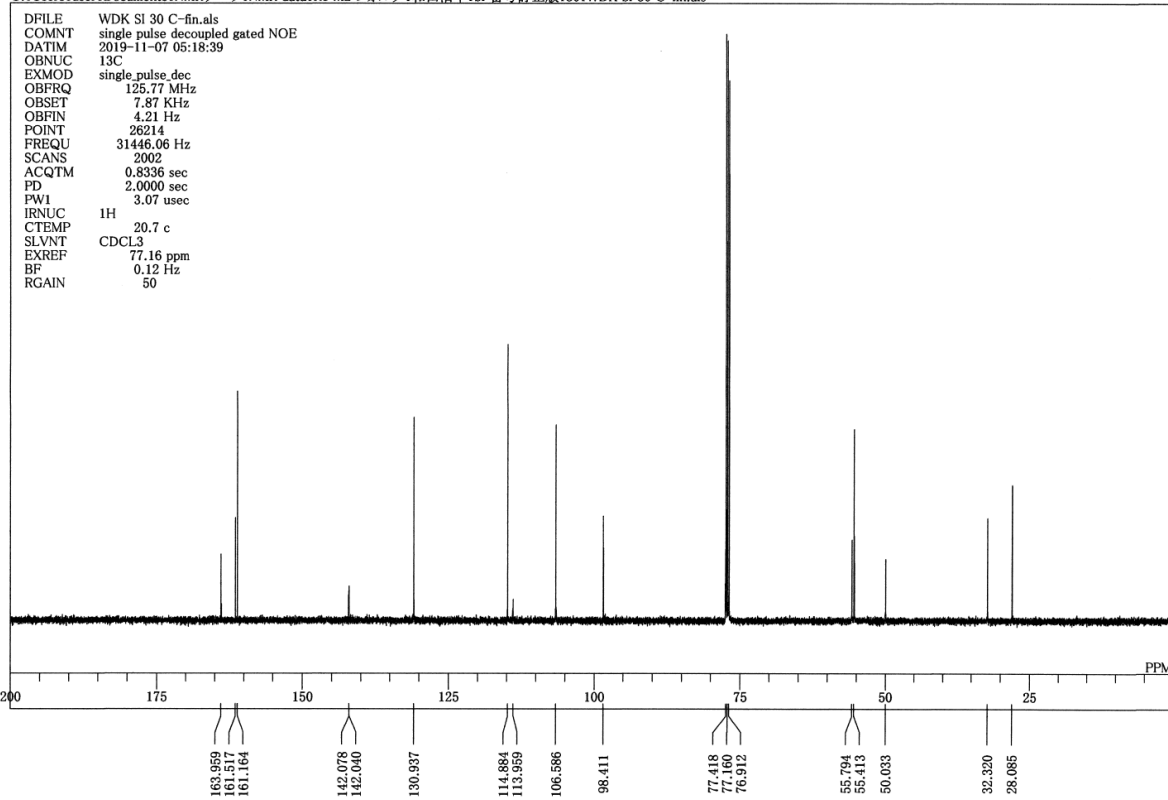
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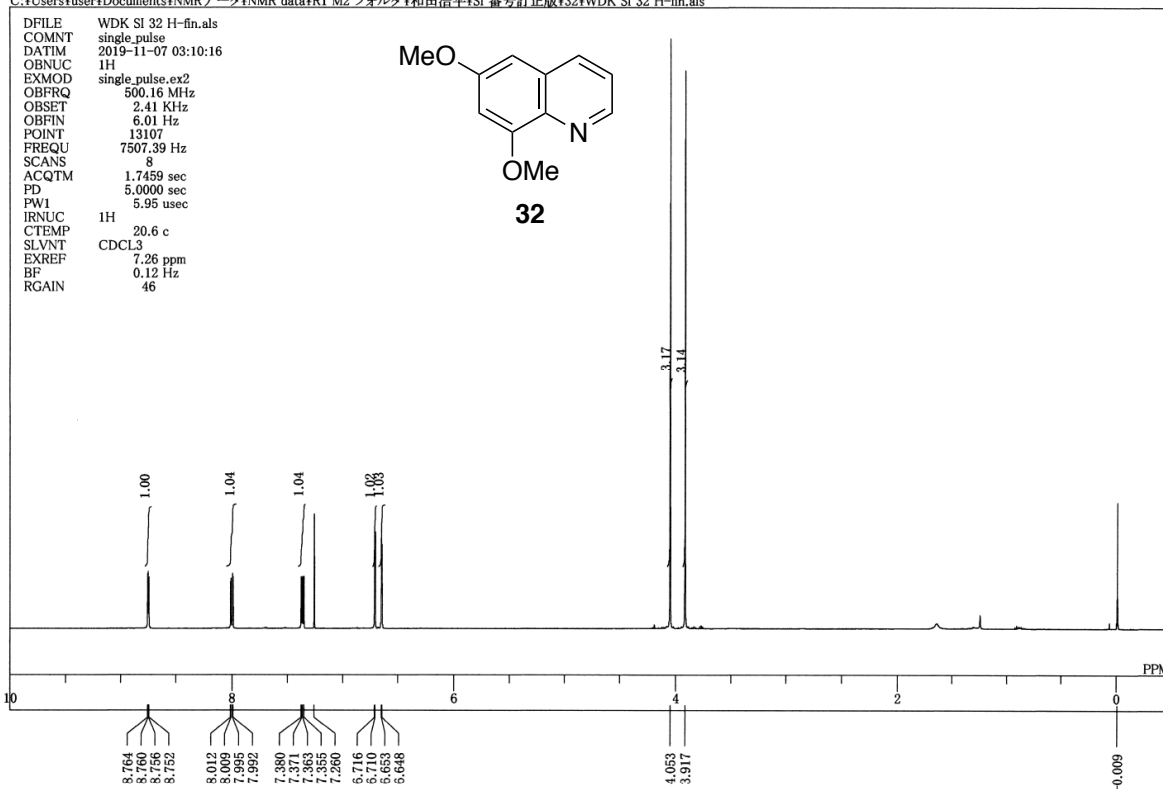
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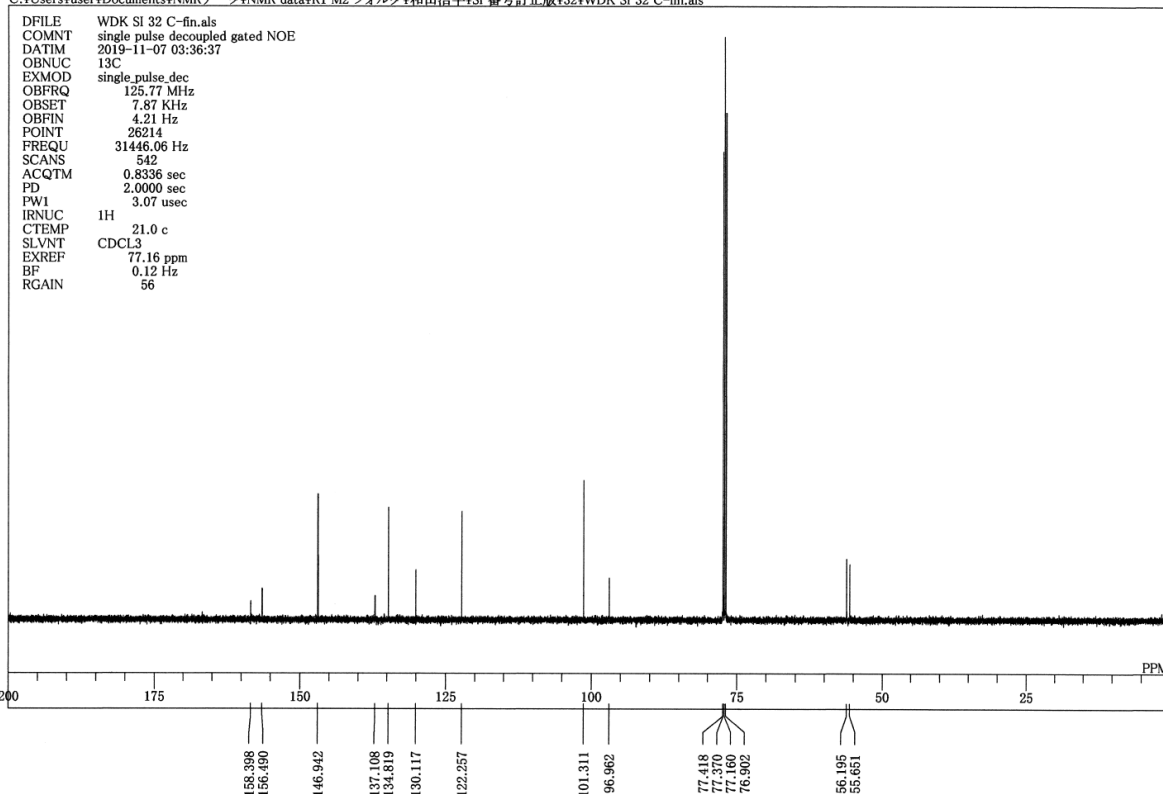
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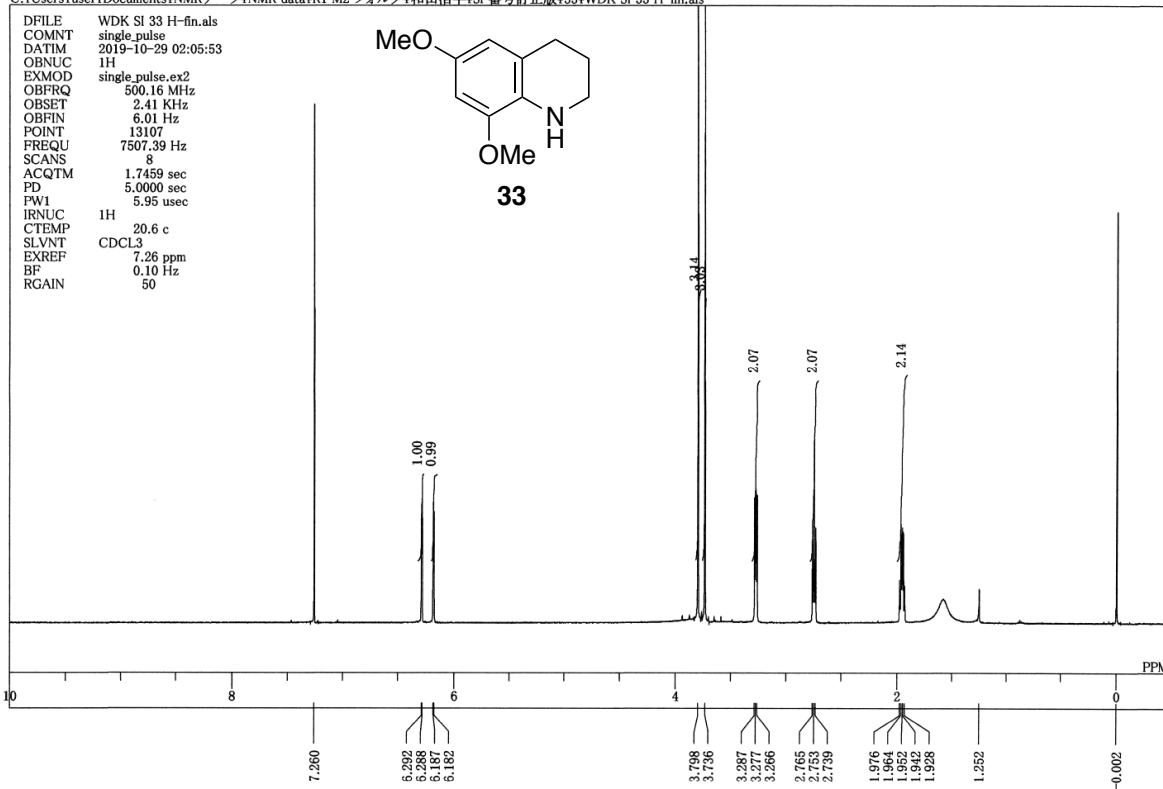
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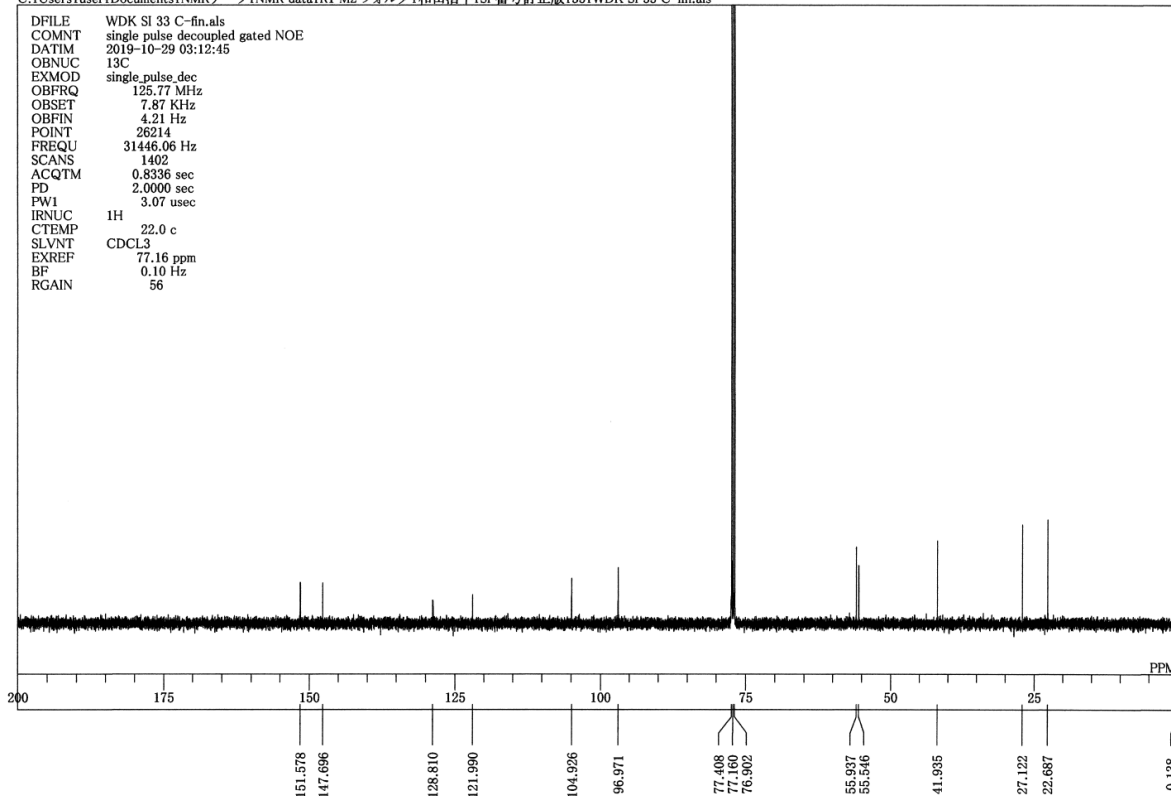
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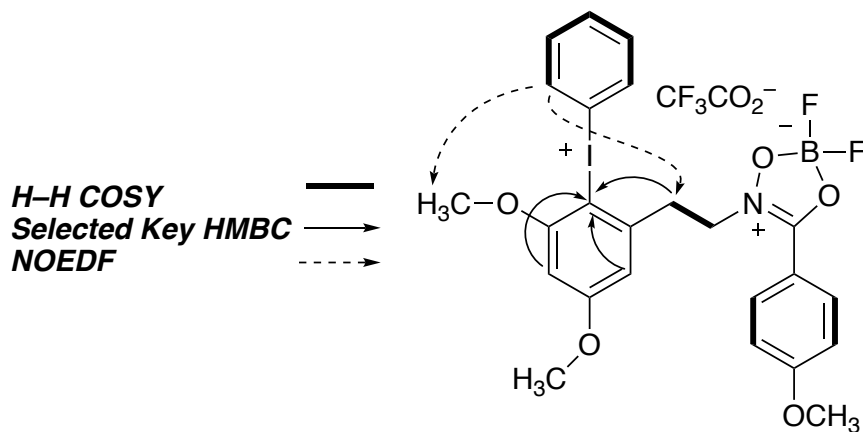
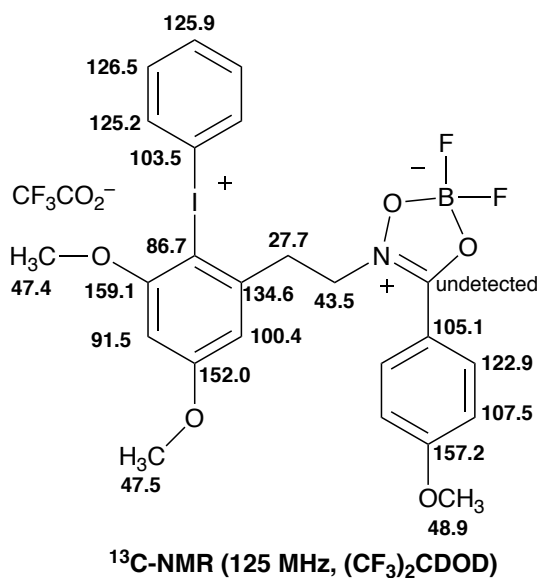
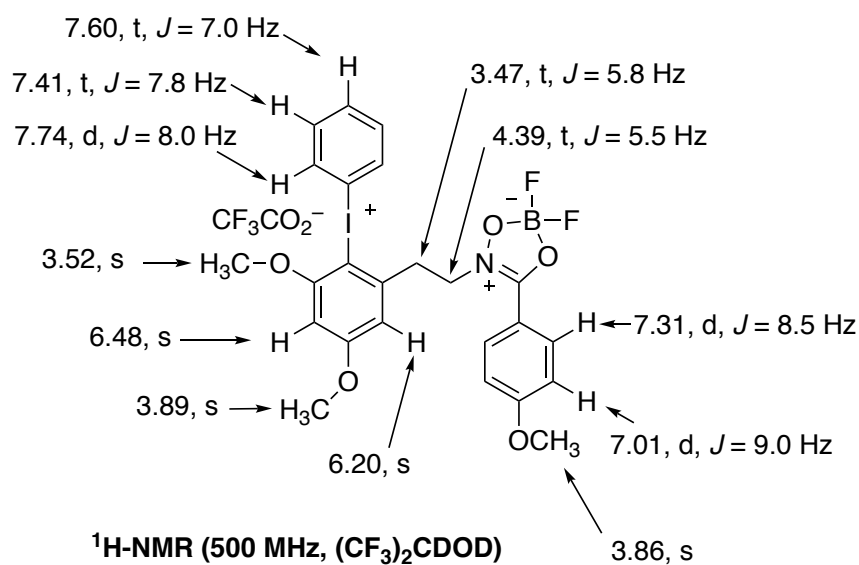
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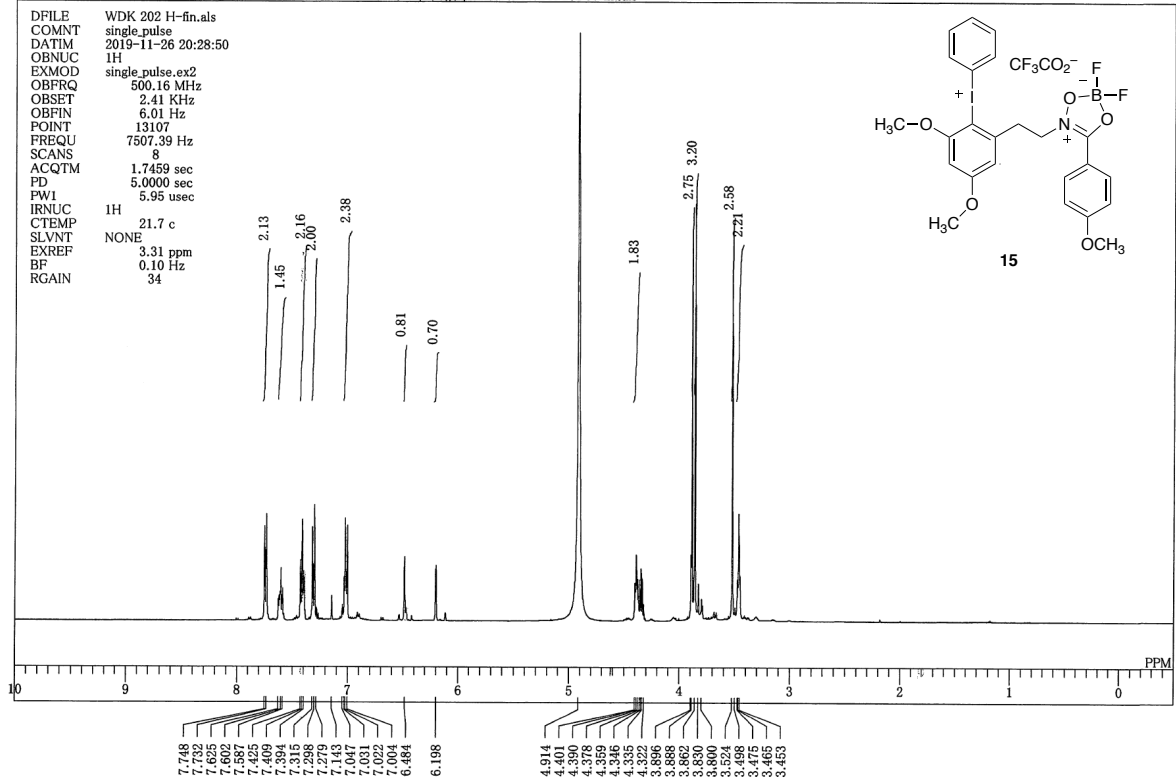
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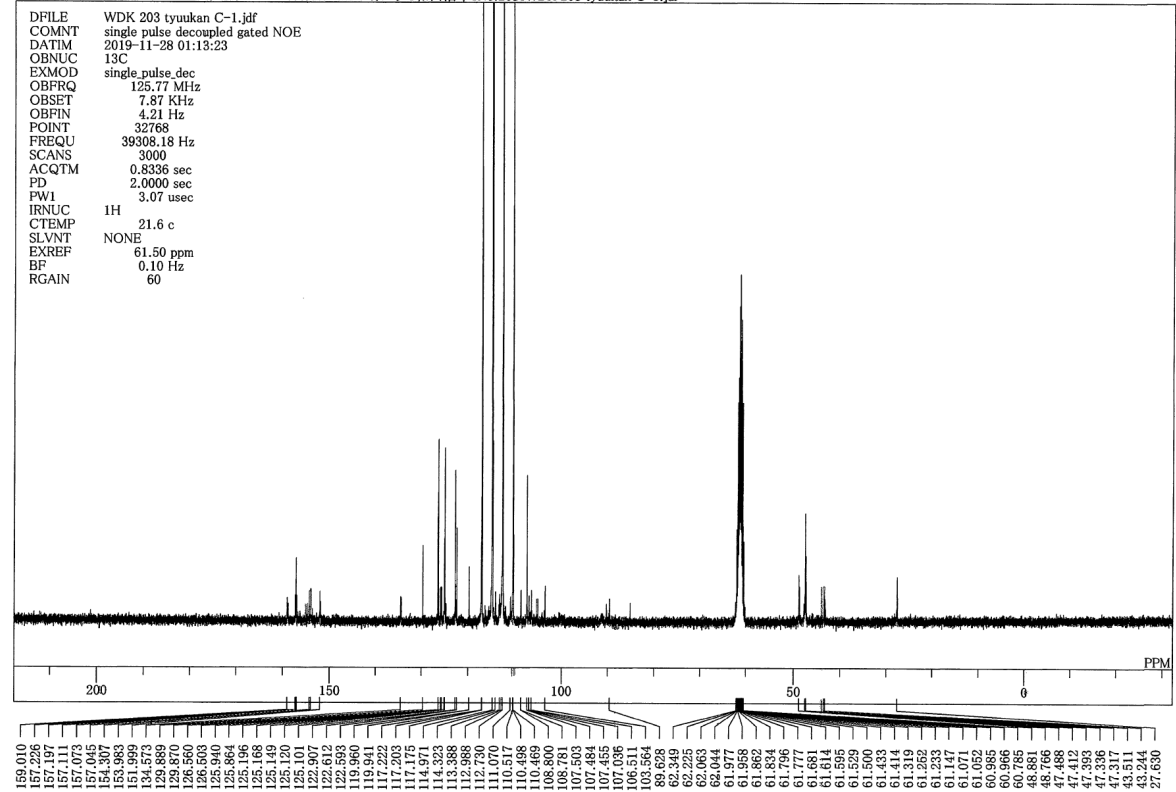
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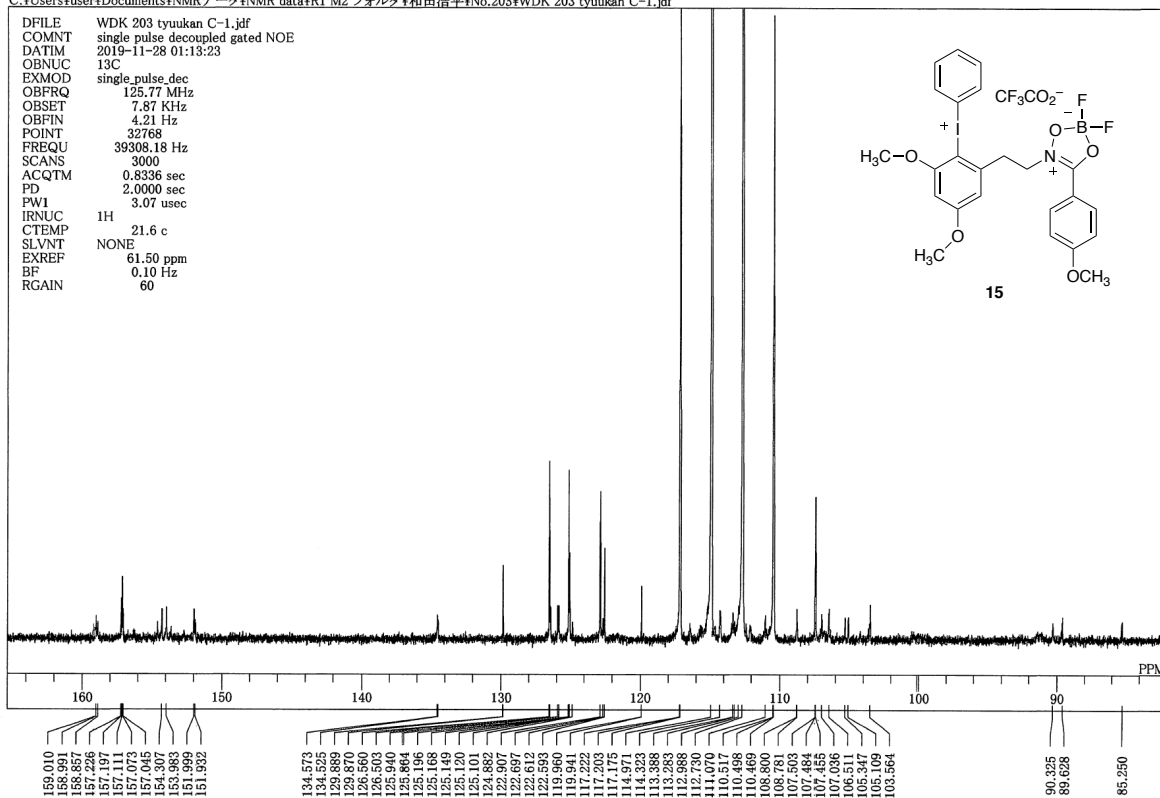
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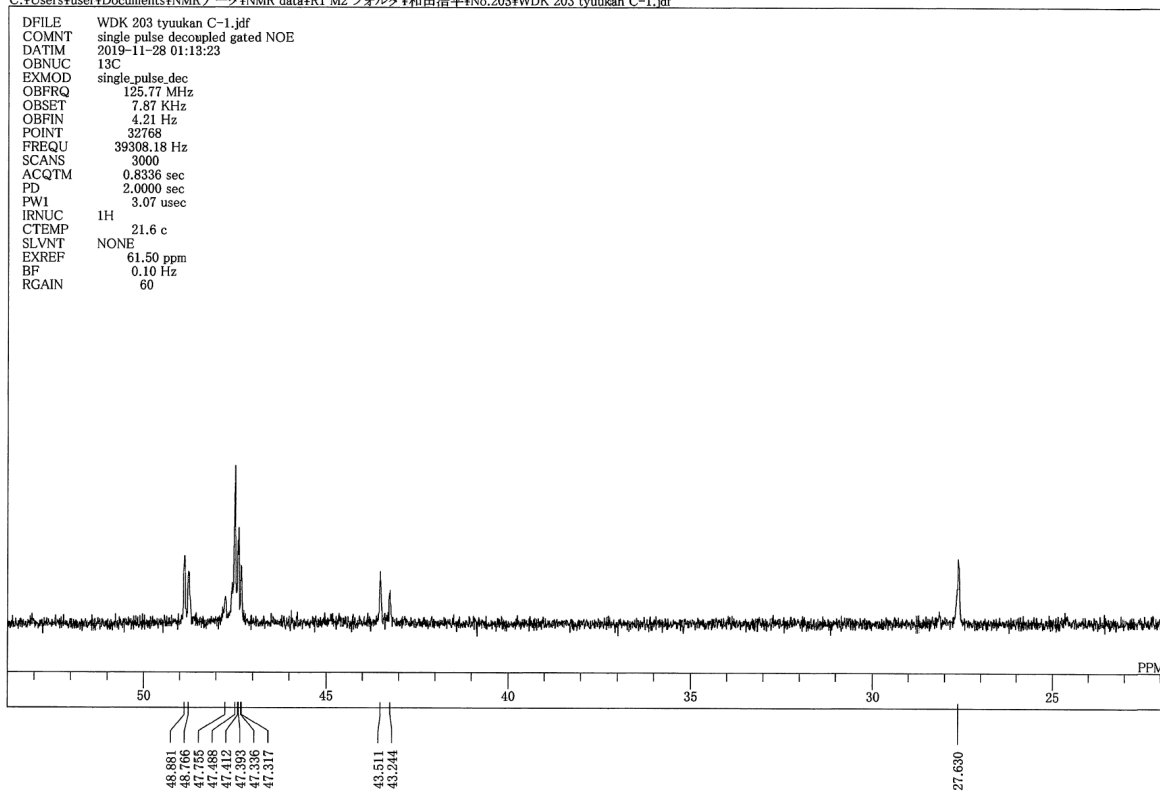
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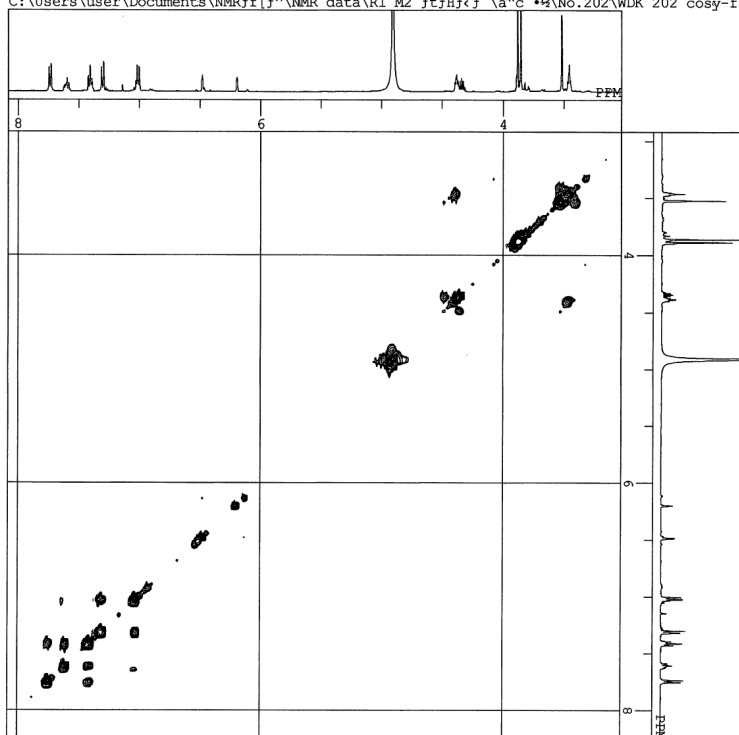
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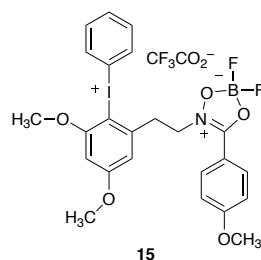
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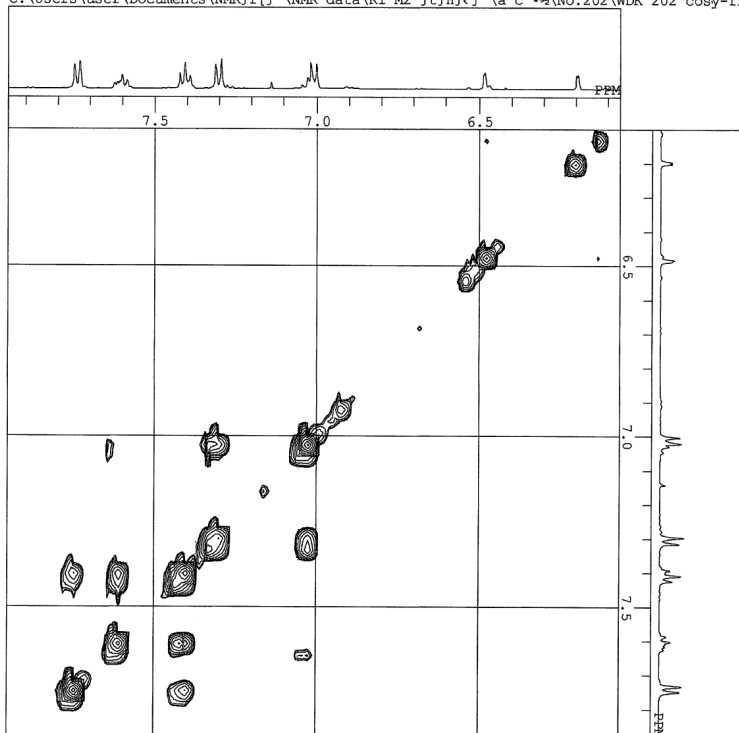


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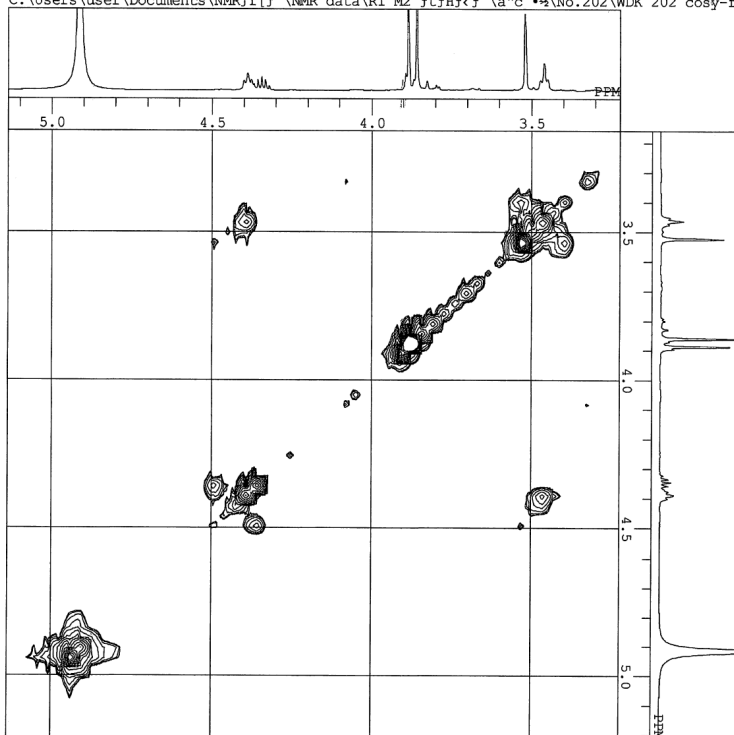
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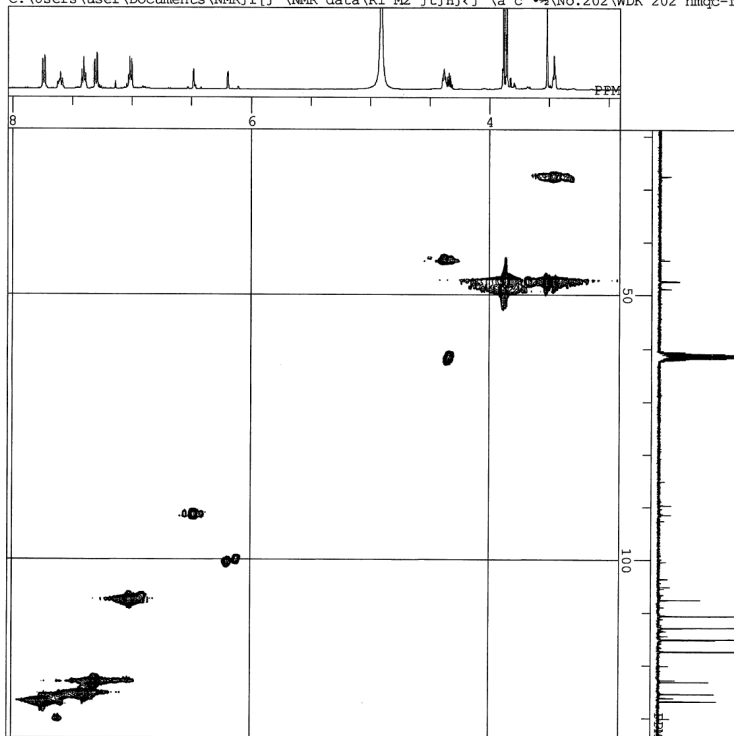
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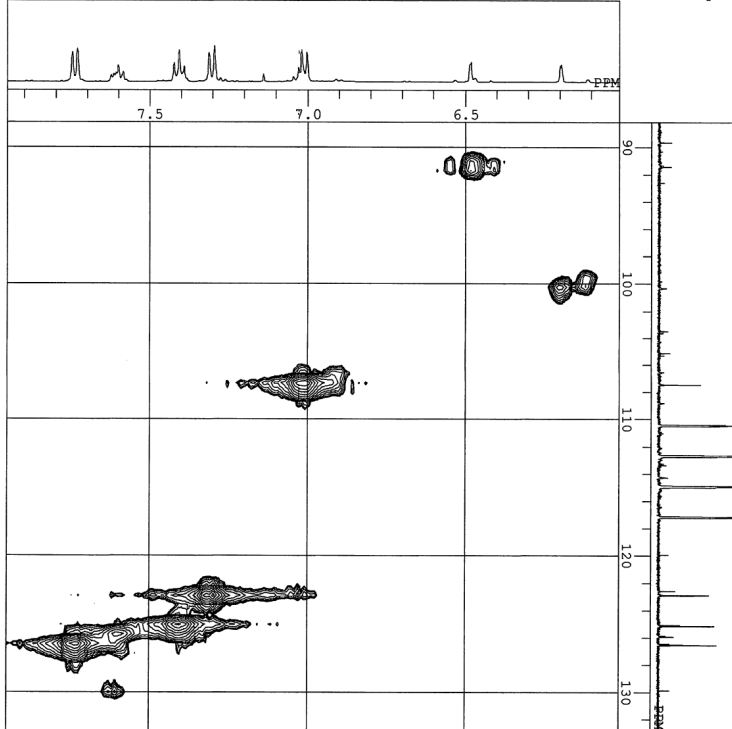
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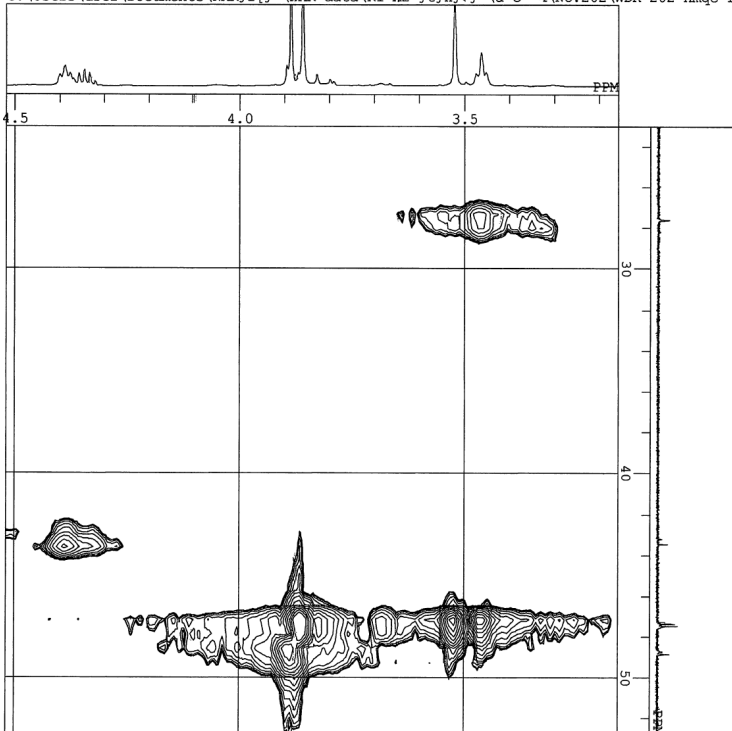
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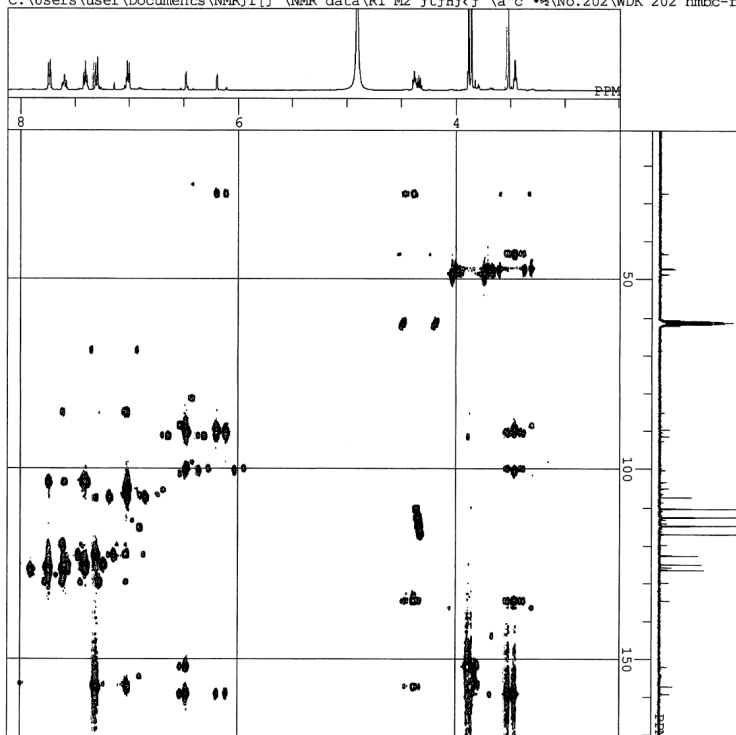
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HMQC

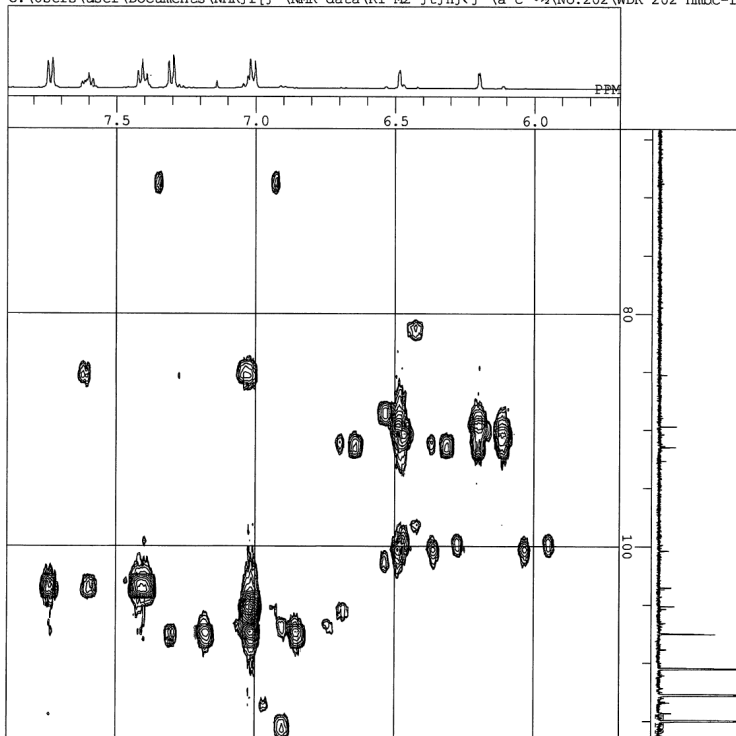
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HMBC

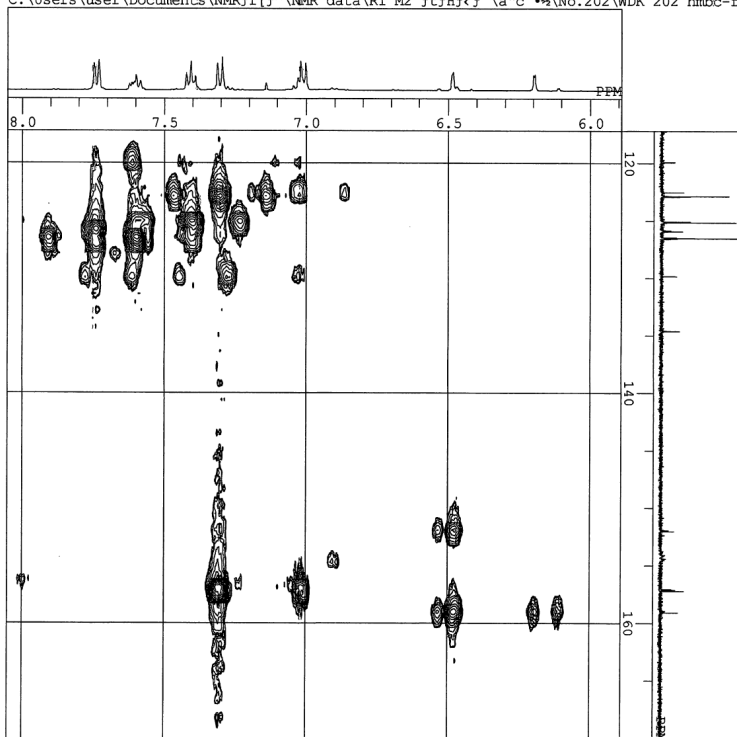
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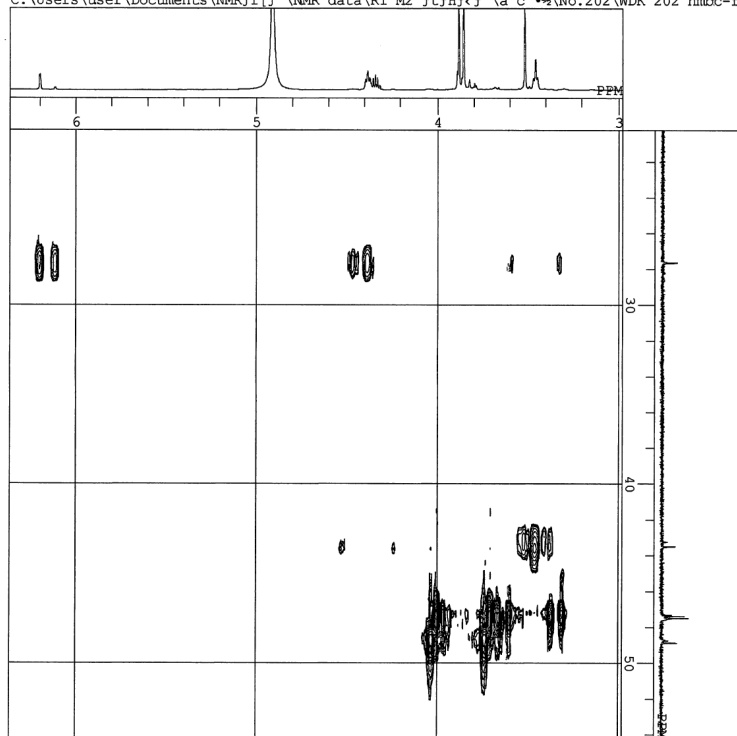
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 SCANS 72  
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HMBC

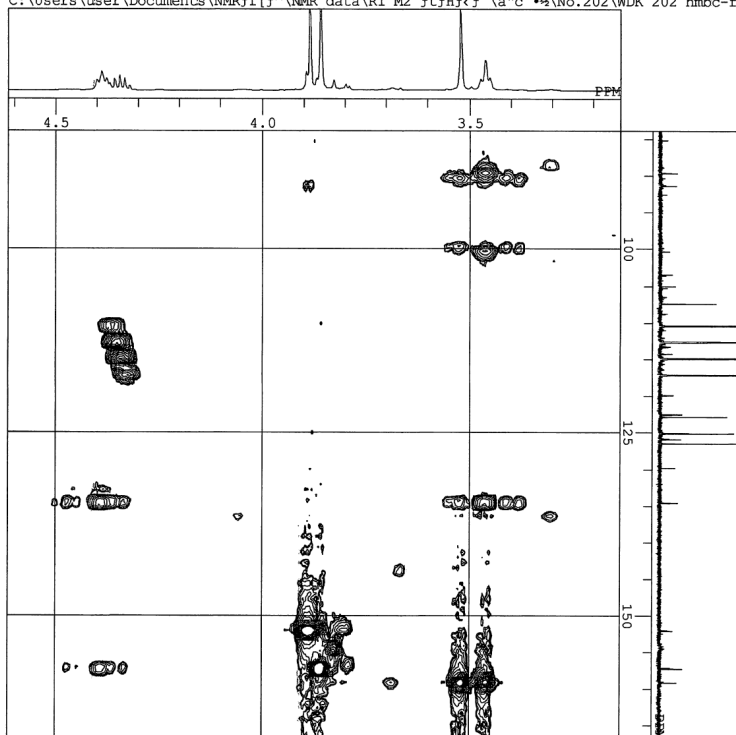
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HMBC

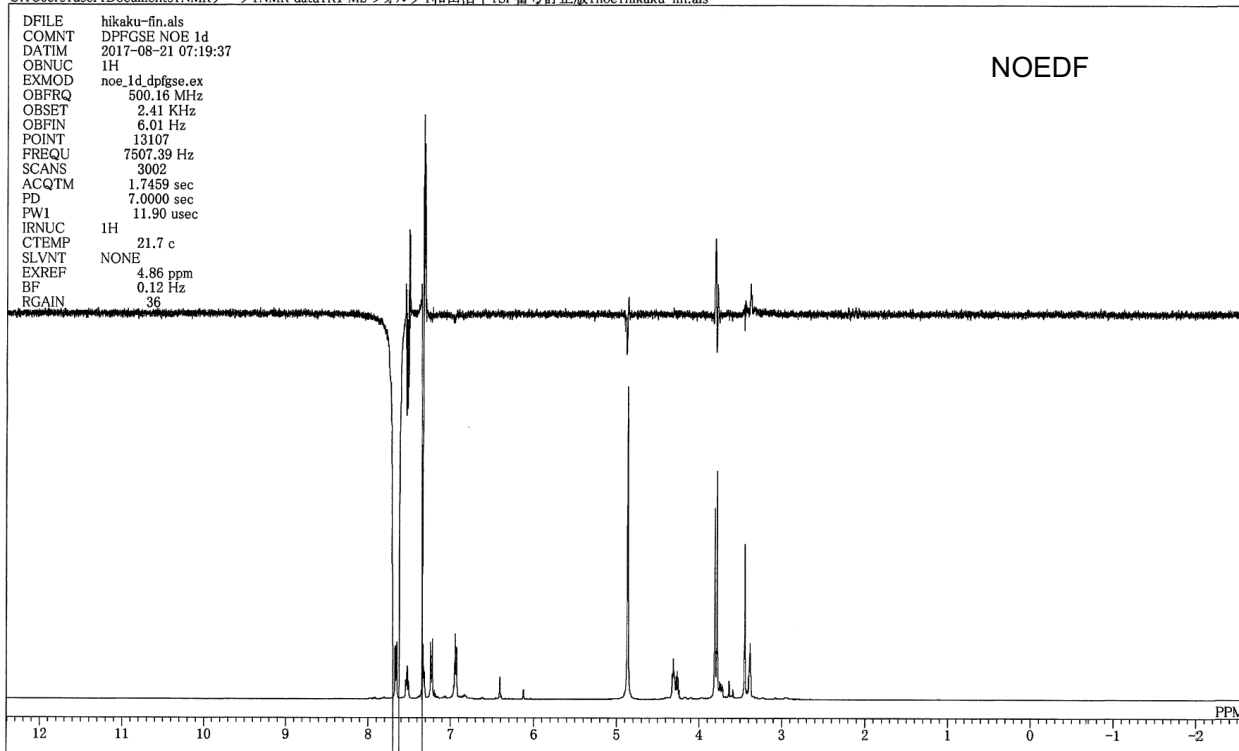
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HMBc

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CTEMP 21.7 c  
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RGAIN 36

NOEDF