[Cp*RhCl$_2$]$_2$ Catalyzed Three-Component Coupling Cyclization of 2,3-Allenoic Acids with 2,3-Allenols in the Presence of Cu(OAc)$_2$·H$_2$O

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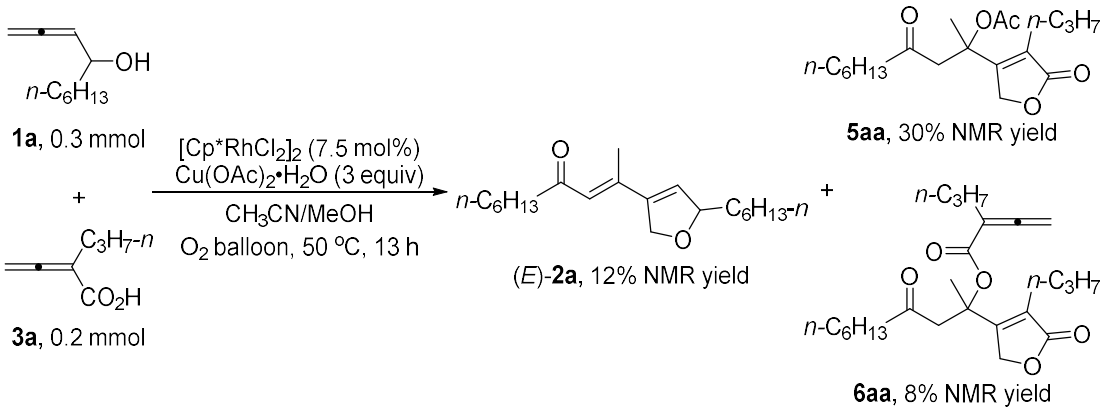
General Information

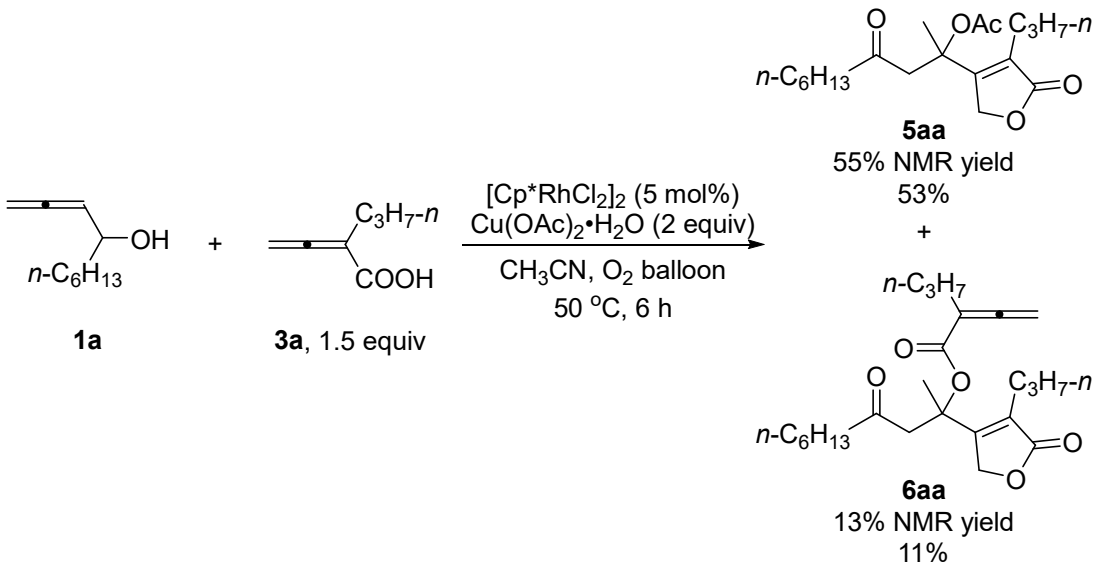
NMR spectra were taken with the 600 MHz, 400 MHz, or 300 MHz Bruker Advance spectrometer (600 MHz, 400 MHz, or 300 MHz for ^{1}H NMR, 100 MHz or 75 MHz for ^{13}C NMR, and 282 MHz for ^{19}F NMR) in $CDCl_3$. All ^{1}H NMR experiments were measured with tetramethylsilane (0 ppm) in $CDCl_3$ as the internal reference; ^{13}C NMR experiments were measured in relative to the signal of $CDCl_3$ (77.0 ppm). ^{19}F NMR experiments were measured in relative to the signal of $CFCl_3$ (0 ppm) in $CDCl_3$ as the internal reference. IR spectra were recorded with a Perkin–Elmer 983G instrument. Elemental analyses were conducted with a Carlo-Erba EA1110 elementary analysis instrument. Mass spectrometry was performed with an HP 5989A system. High-resolution mass spectrometry was determined with a Finnigan MAT 8430 or Bruker APEXIII instrument. $[Cp^*RhCl_2]_2$ was purchased from *HWRK CHEM*. The range of boiling point of petroleum ether used for chromatography was 60~90 ºC unless noted otherwise. Flash column chromatography was performed on silica gel H unless noted otherwise. 2,3-Allenols,[1] 2,3-allenoic acids,[2] and (*E*)-**4aa**[3] were prepared according to the literature procedures. Other commercially available reagents were purchased and used as received.

1. The Reaction of 1a with 3a in the Presence of $[Cp^*RhCl_2]_2$ (5 mol%) and $Cu(OAc)_2 \cdot H_2O$ (2.0 equiv) (fjj-1-028)



To a dry Schlenk tube were added $[Cp^*RhCl_2]_2$ (9.5 mg, 0.015 mmol), $Cu(OAc)_2 \cdot H_2O$ (120.4 mg, 0.6 mmol), and **3a** (25.6 mg, 0.2 mmol) sequentially. The Schlenk tube was degassed under vacuum and backfilled with O_2 with a balloon of O_2 for three times. Then **1a** (46.8 mg, 0.3 mmol), CH_3CN (0.6 mL), and MeOH (30 μL) were added under oxygen atmosphere sequentially. After being stirred for 13 h at 50 °C, the resulting mixture was filtered through a short column of silica gel eluted with ethyl acetate (20 mL × 3). After evaporation of the solvent, the crude product was analyzed by ^1H NMR spectrum with 13.8 μL of mesitylene as the internal standard: The reaction afforded (*E*)-**2a** in 12% NMR yield (based on **1a**) together with **5aa** in 30% NMR yield and **6aa** in 8% NMR yield.

Synthesis of **6aa**. (fjj-2-103)



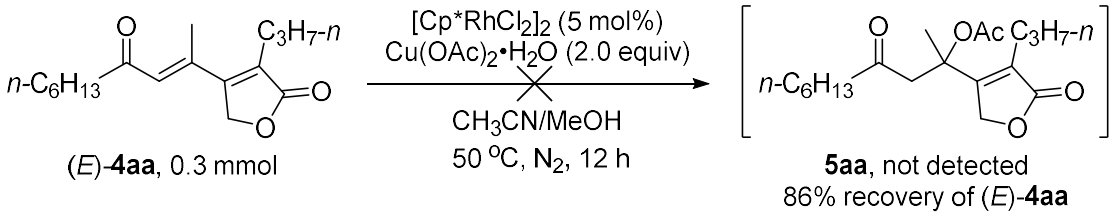
To a dry Schlenk tube were added $[Cp^*RhCl_2]_2$ (15.7 mg, 0.025 mmol), $Cu(OAc)_2 \cdot H_2O$ (199.5 mg, 1.0 mmol), and **3a** (94.8 mg, 0.75 mmol) sequentially. The Schlenk tube was degassed under vacuum and backfilled with O_2 with a balloon of O_2 for three times. Then **1a** (76.5 mg, 0.5 mmol), CH_3CN (2 mL), and MeOH (100 μL) were added under oxygen atmosphere sequentially. After being continuously stirred at 50 °C for 6 h, the reaction was complete as monitored by thin layer chromatography (TLC). After filtration through a short column of silica gel eluted with ethyl acetate (20 mL × 3), the combined filtrate was concentrated in vacuo. The reaction afforded **5aa** in 53% NMR yield together with 13% NMR yield of **6aa**, which was analyzed by ^1H NMR using 23 μL of mesitylene as the internal standard. The crude residual was purified by chromatography on silica gel [eluent: petroleum ether/ethyl acetate = 15/1 (1000 mL) to 10/1 (500 mL), then 5/1 (500 mL)] to afford **6aa** (21.0 mg, 11%) and **5aa** (89.4 mg, 53%).

6aa: Oil; ^1H NMR (300 MHz, $CDCl_3$) δ 5.15 (t, J = 3.0 Hz, 2 H, $=CH_2$), 5.07 (d, J

= 14.7 Hz, 1 H, one proton of OCH_2), 4.99 (d, J = 14.4 Hz, 1 H, one proton of OCH_2), 3.06 (d, J = 14.7 Hz, 1 H, one proton of CH_2), 2.75 (d, J = 14.7 Hz, 1 H, one proton of CH_2), 2.43 (t, J = 7.2 Hz, 2 H, CH_2), 2.34-2.14 (m, 4 H, CH_2 × 2), 1.62-1.41 (m, 9 H, CH_2 × 3 and CH_3), 1.34-1.16 (m, 6 H, CH_2 × 3), 0.98-0.82 (m, 9 H, CH_3 × 3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 214.4, 206.7, 171.8, 166.7, 158.2, 130.6, 99.5, 85.2, 79.3, 58.4, 48.7, 44.8, 31.5, 29.9, 28.6, 25.6, 25.2, 23.1, 22.4, 21.3, 21.1, 14.0, 13.7, 13.5; IR (neat) ν (cm^{-1}) 2960, 2933, 2873, 1964, 1940, 1760, 1716, 1457, 1378, 1246, 1119, 1051; MS (EI): *m/z* (%) 404 (M^+, 0.38), 279 (100); HRMS Calcd for $C_{24}H_{36}O_5$ (M^+): 404.2563; Found: 404.2565.

5aa: Oil; ^1H NMR (300 MHz, $CDCl_3$) δ 4.95 (s, 2 H, CH_2), 3.07 (d, J = 14.7 Hz, 1 H, one proton from CH_2), 2.75 (d, J = 14.7 Hz, 1 H, one proton from CH_2), 2.44 (t, J = 7.2 Hz, 2 H, CH_2), 2.30 (t, J = 7.4 Hz, 2 H, CH_2), 2.10 (s, 3 H, CH_3), 1.62-1.32 (m, 7 H, CH_2 × 2 and CH_3), 1.36-1.13 (m, 6 H, CH_2× 3), 0.94 (t, J = 7.4 Hz, 3 H, CH_3), 0.87 (t, J = 6.2 Hz, 3 H, CH_3);

2. The Reaction of (*E*)-4aa in the Presence of [Cp*RhCl$_2$]$_2$ (5 mol%) and Cu(OAc)$_2$·H$_2$O (2.0 equiv) (fjj-2-042)



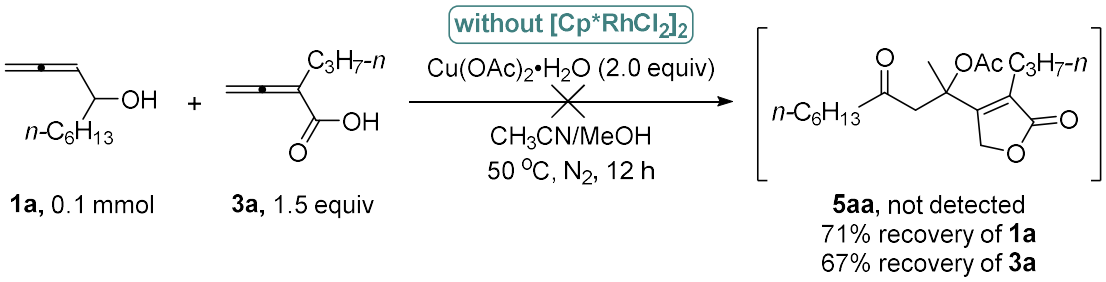
(*E*)-**4aa**, 0.3 mmol

5aa, not detected
86% recovery of (*E*)-**4aa**

To a dry Schlenk tube were added [Cp*RhCl$_2$]$_2$ (9.2 mg, 0.015 mmol) and Cu(OAc)$_2$·H$_2$O (119.0 mg, 0.6 mmol) sequentially. The Schlenk tube was degassed under vacuum and backfilled with nitrogen for three times. Then (*E*)-**4aa** (83.0 mg, 0.3 mmol), CH$_3$CN (0.8 mL), and MeOH (40 μL) were added under nitrogen atmosphere sequentially. After being stirred for 12 h at 50 °C, the resulting mixture was filtered through a short column of silica gel eluted with ethyl acetate (20 mL × 3). After evaporation of the solvent, the crude product was analyzed by ^{1}H NMR spectrum with 13.8 μL of mesitylene as the internal standard. No signal of the corresponding product was found with the recovery of (*E*)-**4aa** in 86%.

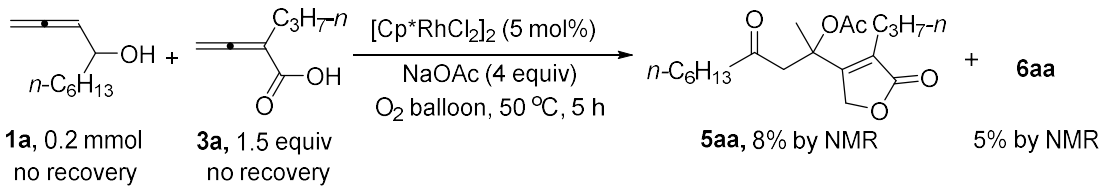
3. Control Experiments and Isotopic Labeling Experiments

3.1 The reaction of **1a** with **3a** without $[Cp^*RhCl_2]_2$. (fjj-2-026)



To a dry Schlenk tube were added $Cu(OAc)_2 \cdot H_2O$ (39.9 mg, 0.2 mmol) and **3a** (19.4 mg, 0.15 mmol) sequentially. The Schlenk tube was degassed under vacuum and backfilled with nitrogen for three times. Then **1a** (15.0 mg, 0.1 mmol), CH_3CN (0.28 mL), and MeOH (13 μL) were added under nitrogen atmosphere sequentially. After being stirred for 12 h at 50 °C, the resulting mixture was filtered through a short column of silica gel eluted with ethyl acetate (20 mL × 3). After evaporation of the solvent, the crude product was analyzed by ^1H NMR spectrum with 4.6 μL of mesitylene as the internal standard. No signal of the corresponding product **5aa** was found with the recovery of **1a** at 71% and **3a** at 67%.

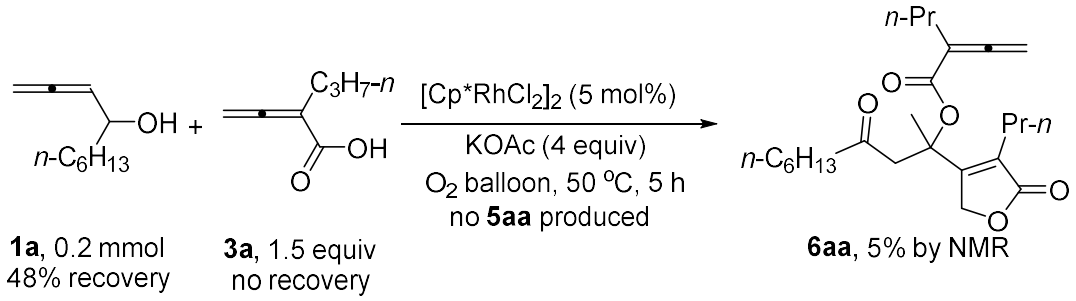
3.2 The reaction of **1a** with **3a** using O_2 as oxidant and NaOAc as the acetoxylation agent. (fjj-7-194)



To a dry Schlenk tube were added $[Cp^*RhCl_2]_2$ (6.2 mg, 0.01 mmol), NaOAc (65.8

mg, 0.8 mmol), and **3a** (38.0 mg, 0.3 mmol) sequentially. The Schlenk tube was degassed under vacuum and backfilled with O_2 for three times. Then **1a** (30.9 mg, 0.2 mmol), CH_3CN (0.54 mL), and MeOH (26 μL) were added under O_2 atmosphere sequentially. After being stirred for 5 h at 50 °C, the resulting mixture was filtered through a short column of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent, the crude product was analyzed by ^1H NMR spectrum with 9.2 μL of mesitylene as the internal standard: 8% NMR yield of **5aa** and 5% NMR yield of **6aa** was detected.

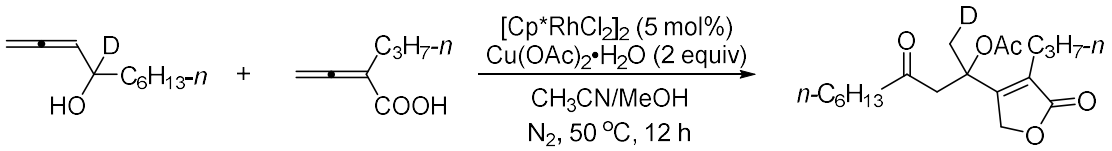
3.3 The reaction of **1a** with **3a** using O_2 as oxidant and KOAc as the acetoxylation agent. (fjj-7-195)



To a dry Schlenk tube were added $[Cp^*RhCl_2]_2$ (6.2 mg, 0.01 mmol), KOAc (78.5 mg, 0.8 mmol), and **3a** (37.8 mg, 0.3 mmol) sequentially. The Schlenk tube was degassed under vacuum and backfilled with O_2 for three times. Then **1a** (30.9 mg, 0.2 mmol), CH_3CN (0.54 mL), and MeOH (26 μL) were added under O_2 atmosphere sequentially. After being stirred for 5 h at 50 °C, the resulting mixture was filtered through a short column of silica gel eluted with ethyl acetate (10 mL × 3). After

evaporation of the solvent, the crude product was analyzed by ^{1}H NMR spectrum with 9.2 μL of mesitylene as the internal standard:No signal of **5aa** and 5% NMR yield of **6aa** was detected with the recovery of **1a** at 48%.

3.4 Synthesis of [D]-**5aa**. (fjj-1-085)



[D]-**1a**
1 mmol

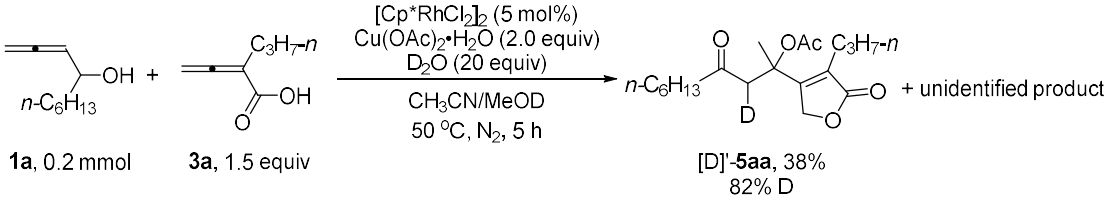
3a, 1.5 equiv

[D]-**5aa**, 55%
(> 99% D)

Typical Procedure I: To a dry Schlenk tube were added $[Cp^*RhCl_2]_2$ (30.9 mg, 0.05 mmol), $Cu(OAc)_2 \cdot H_2O$ (399.4 mg, 2.0 mmol), and **3a** (189.8 mg, 1.5 mmol). The reaction vessel was degassed under vacuum and backfilled with nitrogen for three times. Then [D]-**1a** (155.2 mg, 1.0 mmol), CH_3CN (2.7 mL), and MeOH (0.13 mL) were added under nitrogen atmosphere sequentially. After being continuously stirred at 50 °C for 12 h, the reaction was complete as monitored by thin layer chromatography (TLC). After filtration through a short column of silica gel eluted with ethyl acetate (20 mL × 3), the combined filtrate was concentrated in vacuo and the crude residual was purified by chromatography on silica gel [eluent: petroleum ether/ethyl acetate = 15/1 (500 mL) to 10/1 (500 mL), then 5/1 (500 mL)] to afford [D]-**5aa** (187.0 mg, 55%) as an oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.95 (dd, J_1= 14.6 Hz, J_2= 14.4 Hz, 2 H, OCH_2), 3.07 (d, J = 14.7 Hz, 1 H, one proton of CH_2), 2.74 (d, J = 14.7 Hz, 1 H, one proton of CH_2), 2.44 (t, J = 7.4 Hz, 2 H, CH_2), 2.31 (t, J = 7.7 Hz, 2 H, CH_2), 2.11 (s, 3 H, OAc),

1.63-1.36 (m, 6 H, CH_2 × 2 and CH_2D), 1.36-1.12 (m, 6 H, CH_2 × 3), 0.94 (t, J = 7.4 Hz, 3 H, CH_3), 0.87 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.5, 171.7, 169.9, 157.7, 131.1, 84.9, 57.4, 48.5, 44.6, 31.4, 28.5, 25.6, 24.9 (t, J = 19.3 Hz), 23.0, 22.3, 21.3, 20.5, 13.9, 13.6; IR (neat) ν (cm^{-1}) 2959, 2932, 2873, 1762, 1747, 1710, 1682, 1452, 1368, 1218, 1160, 1128, 1023; MS (EI): *m/z* (%) 339 (M^+, 2.83), 43 (100); HRMS Calcd for $C_{19}H_{29}DO_5$ $(M)^+$: 339.2156; Found: 339.2159.

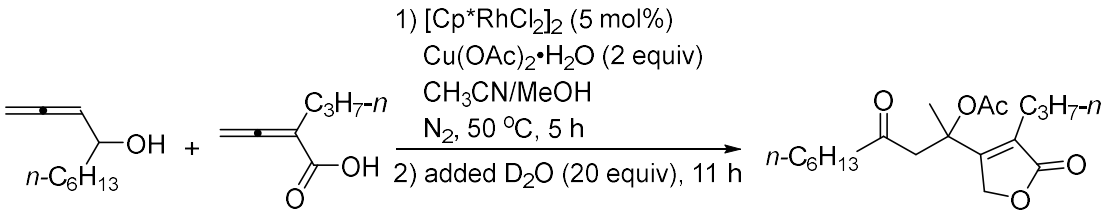
3.5 Synthesis of [D]'-**5aa**. (fjj-5-071)



To a dry Schlenk tube were added $[Cp^*RhCl_2]_2$ (6.2 mg, 0.01 mmol), $Cu(OAc)_2{\cdot}H_2O$ (80.2 mg, 0.4 mmol), and **3a** (38.0 mg, 0.3 mmol) sequentially. The reaction vessel was degassed under vacuum and backfilled with nitrogen for three times. Then **1a** (30.9 mg, 0.2 mmol), CH_3CN (0.54 mL), MeOD (27 μL), and D_2O (80 mg, 4 mmol) were added under nitrogen atmosphere sequentially. After being continuously stirred at 50 °C for 5 h, the reaction was complete as monitored by thin layer chromatography (TLC). After filtration through a short column of silica gel eluted with ethyl acetate (20 mL × 3), the combined filtrate was concentrated in vacuo and the crude residual was purified by chromatography on silica gel (300~400 mesh) [eluent: petroleum ether/ethyl acetate = 9/1 (600 mL)] to afford [D]'-**5aa** (26.0 mg, 38%, 82% D) and some unidentified products.

[D]'-**5aa**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.00-4.89 (m, 2 H, OCH_2), [3.09-3.00 (m, 0.62 H), 2.77-2.68 (m, 0.56 H), one proton of CHD], 2.44 (t, *J* = 7.4 Hz, 2 H, CH_2), 2.30 (t, *J* = 7.6 Hz, 2 H, CH_2), 2.10 (s, 3 H, OAc), 1.63-1.42 (m, 7 H, CH_3, and CH_2 × 2), 1.37-1.13 (m, 6 H, CH_2 × 3), 0.94 (t, *J* = 7.4 Hz, 3 H, CH_3), 0.87 (t, *J* = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 48.4 (t, *J* = 19.4 Hz); IR (neat) *ν* (cm^{-1}) 2960, 2933, 2873, 1755, 1716, 1456, 1368, 1221, 1029; MS (EI): *m/z* (%) 339 (M^+, 4.96), 43 (100); HRMS Calcd for $C_{19}H_{29}DNaO_5$ $(M + Na)^+$: 362.2048; Found: 362.2046. The following signals are discernible for **5aa**: ^{13}C NMR (100 MHz, $CDCl_3$) δ 48.7.

3.6 The reaction of **1a** and **3a** under standard conditions quenched with D_2O (fjj-5-073)



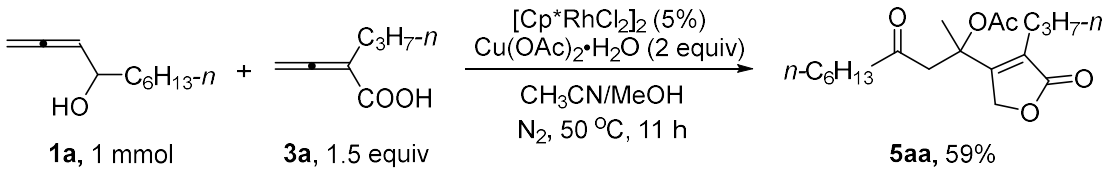
1a, 0.2 mmol **3a**, 1.5 equiv **5aa**, 45% (0% D)

To a dry Schlenk tube were added $[Cp^*RhCl_2]_2$ (6.2 mg, 0.01 mmol), $Cu(OAc)_2 \cdot H_2O$ (79.8 mg, 0.4 mmol), and **3a** (37.8 mg, 0.3 mmol). The reaction vessel was degassed under vacuum and backfilled with nitrogen for three times. Then **1a** (30.9 mg, 0.2 mmol), CH_3CN (0.54 mL), and MeOH (28 μL) were added under nitrogen atmosphere sequentially. After being continuously stirred at 50 °C for 5 h, the reaction was complete as monitored by thin layer chromatography (TLC). Then D_2O (80 mg, 4 mmol) was added. The resulting mixture was continuously stirred at 50 °C for 11 h.

After filtration through a short column of silica gel eluted with ethyl ether (20 mL × 3), the combined filtrate was concentrated in vacuo and the crude residual was purified by chromatography on silica gel [eluent: petroleum ether/ethyl acetate = 9/1 (500 mL)] to afford **5aa** (30.6 mg, 45%) as an oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.94 (s, 2 H, OCH_2), 3.05 (d, *J* = 14.7 Hz, 1 H, one proton of CH_2), 2.73 (d, *J* = 14.7 Hz, 1 H, one proton of CH_2), 2.44 (t, *J* = 7.2 Hz, 2 H, CH_2), 2.30 (t, *J* = 7.5 Hz, 2 H, CH_2), 2.10 (s, 3 H, OAc), 1.63-1.40 (m, 7 H, CH_2 × 2 and CH_3), 1.38-1.13 (m, 6 H, CH_2 × 3), 0.94 (t, *J* = 7.4 Hz, 3 H, CH_3), 0.87 (t, *J* = 6.6 Hz, 3 H, CH_3).

4. Rh(III)-Catalyzed Oxidative Cross-coupling Cyclization of 2,3- Allenoic Acids and 2,3-Allenols

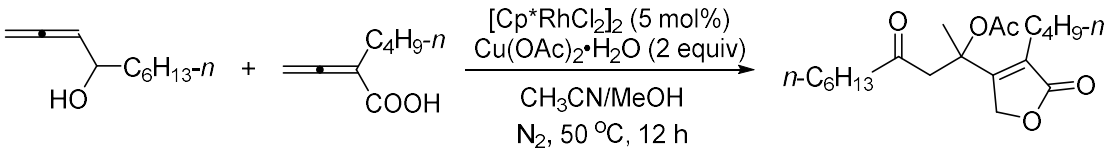
4.1 Synthesis of 4-(2-acetoxy-4-oxodecan-2-yl)-3-propyl-2(*5H*)-furanone **5aa** (fjj-1-052)



1a, 1 mmol **3a**, 1.5 equiv **5aa**, 59%

Following **Typical Procedure I**, the reaction of **1a** (152.8 mg, 1.0 mmol), **3a** (189.2 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (399.9 mg, 2.0 mmol) in CH_3CN (2.7 mL)/MeOH (0.13 mL) afforded **5aa** (197.1 mg, 59%) as an oil [eluent: petroleum ether/ethyl acetate = 50/1 (300 mL) →15/1 (800 mL) → 10/1 (500 mL) → 5/1 (500 mL)]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.95 (dd, J_1 = 14.7 Hz, J_2 = 14.4 Hz, 2 H, OCH_2), 3.06 (d, J = 15.0 Hz, 1 H, one proton from CH_2), 2.74 (d, J = 14.7 Hz, 1 H, one proton from CH_2), 2.44 (t, J = 7.2 Hz, 2 H, CH_2), 2.31 (t, J = 7.5 Hz, 2 H, CH_2), 2.10 (s, 3 H, CH_3), 1.61-1.43 (m, 7 H, CH_2 × 2 and CH_3), 1.35-1.18 (m, 6 H, CH_2 × 3), 0.94 (t, J = 7.4 Hz, 3 H, CH_3), 0.87 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.5, 171.7, 169.9, 157.7, 131.2, 85.0, 57.4, 48.6, 44.6, 31.4, 28.6, 25.7, 25.2, 23.1, 22.3, 21.3, 20.6, 13.9, 13.7; IR (neat) ν (cm^{-1}) 2960, 2933, 2873, 1748, 1712, 1678, 1456, 1406, 1368, 1221, 1135, 1030; MS (EI): *m/z* (%) 338 (M^+, 1.86), 43 (100); HRMS Calcd for $C_{19}H_{30}O_5Na$ $(M+Na)^+$: 361.1985; Found: 361.1985.

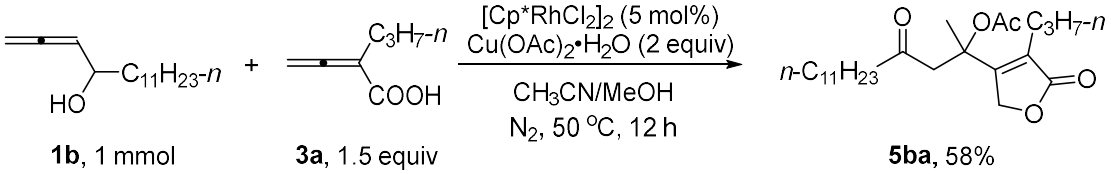
4.2 Synthesis of 4-(2-acetoxy-4-oxodecan-2-yl)-3-butyl-2(*5H*)-furanone **5ab** (fjj-1-104)



1a, 1 mmol **3b**, 1.5 equiv **5ab**, 57%

Following **Typical Procedure I**, the reaction of **1a** (154.2 mg, 1.0 mmol), **3b** (210.4 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (30.9 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (399.4 mg, 2.0 mmol) in CH_3CN (2.7 mL)/MeOH (0.13 mL) afforded **5ab** (201.2 mg, 57%) as an oil [eluent: petroleum ether/ethyl acetate = 10/1 (500 mL) to 5/1 (500 mL)]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.94 (dd, J_1 = 14.9 Hz, J_2 = 14.7 Hz, 2 H, OCH_2), 3.05 (d, J = 14.7 Hz, 1 H, one proton of CH_2), 2.73 (d, J = 14.7 Hz, 1 H, one proton of CH_2), 2.44 (t, J = 7.2 Hz, 2 H, CH_2), 2.32 (t, J = 7.7 Hz, 2 H, CH_2), 2.11 (s, 3 H, OAc), 1.57-1.41 (m, 7 H, CH_3 and CH_2 × 2), 1.41-1.16 (m, 8 H, CH_2 × 4), 0.92 (t, J = 7.2 Hz, 3 H, CH_3), 0.87 (t, J = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.6, 171.8, 170.0, 157.4, 131.4, 85.0, 57.5, 48.7, 44.7, 31.5, 30.1, 28.6, 25.2, 23.6, 23.1, 22.5, 22.4, 20.6; 13.9, 13.8; IR (neat) ν (cm^{-1}) 2959, 2932, 2861, 1747, 1710, 1455, 1403, 1368, 1283, 1220, 1127, 1038; MS (EI): *m/z* (%) 352 (M^+, 2.53), 43 (100); HRMS Calcd for $C_{20}H_{32}O_5$ (M^+): 352.2250; Found: 352.2249.

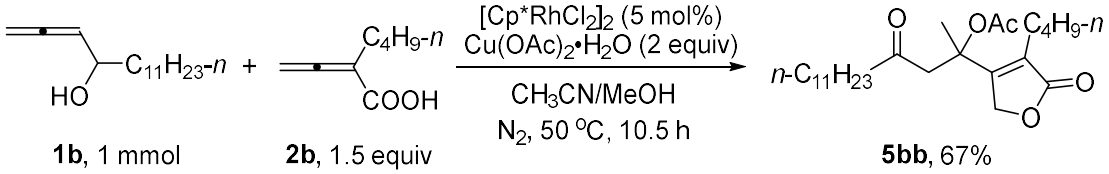
4.3 Synthesis of 4-(2-acetoxy-4-oxopentadecan-2-yl)-3-propyl-2(*5H*)-furanone **5ba** (fjj-1-073)



1b, 1 mmol **3a**, 1.5 equiv **5ba**, 58%

Following **Typical Procedure I**, the reaction of **1b** (224.3 mg, 1.0 mmol), **3a** (189.4 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (31.2 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (400.1 mg, 2.0 mmol) in CH_3CN (2.7 mL)/MeOH (0.13 mL) afforded **5ba** (237.1 mg, 58%) as an oil [eluent: petroleum ether/ethyl acetate = 10/1 (1000 mL) to 5/1 (1000 mL)]: ^1H NMR (300 MHz, $CDCl_3$) δ 4.95 (dd, J_1 = 14.6 Hz, J_2 = 14.1 Hz, 2 H, OCH_2), 3.06 (d, J = 14.7 Hz, 1 H, one proton of CH_2), 2.74 (d, J = 15.0 Hz, 1 H, one proton of CH_2), 2.44 (t, J = 7.4 Hz, 2 H, CH_2), 2.31 (t, J = 7.7 Hz, 2 H, CH_2), 2.10 (s, 3 H, OAc), 1.62-1.38 (m, 7 H, $CH_2 \times 2$ and CH_3), 1.37-1.14 (m, 16 H, $CH_2 \times 8$), 1.00-0.82 (m, 6 H, $CH_3 \times 2$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.5, 171.7, 169.9, 157.7, 131.2, 85.0, 57.4, 48.6, 44.6, 31.8, 29.5, 29.32, 29.29, 29.2, 28.9, 25.7, 25.2, 23.1, 22.6, 21.3, 20.5, 14.0, 13.7; IR (neat) v (cm^{-1}) 2959, 2925, 2854, 1767, 1747, 1715, 1682, 1465, 1455, 1401, 1367, 1200, 1135, 1029; MS (EI): *m/z* (%) 408 (M^+, 1.79), 43 (100); HRMS Calcd for $C_{24}H_{40}O_5$ (M^+): 408.2876; Found: 408.2875.

4.4 Synthesis of 4-(2-acetoxy-4-oxopentadecan-2-yl)-3-butyl-2(*5H*)-furanone **5bb** (fjj-1-075)

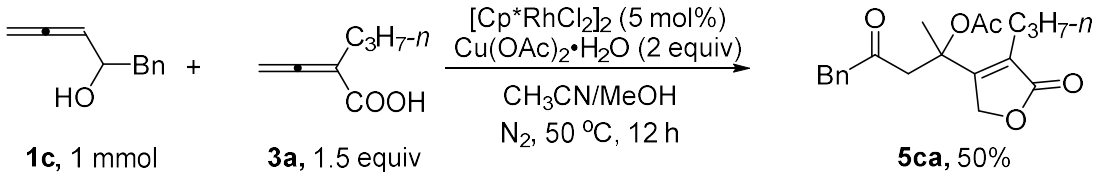


1b, 1 mmol **2b**, 1.5 equiv **5bb**, 67%

Following **Typical Procedure I**, the reaction of **1b** (226.5 mg, 1.0 mmol), **2b** (210.1 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (31.3 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (400.1 mg, 2.0 mmol) in CH_3CN (2.7 mL)/MeOH (0.13 mL) afforded **5bb** (286.2 mg, 67%)

as an oil [eluent: petroleum ether/ethyl acetate = 10/1 (500 mL) to 5/1 (500 mL)]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.94 (dd, J_1 = 14.9 Hz, J_2 = 14.7 Hz, 2 H, OCH_2), 3.05 (d, J = 14.7 Hz, 1 H, one proton of CH_2), 2.72 (d, J = 14.7 Hz, 1 H, one proton of CH_2), 2.43 (t, J = 7.2 Hz, 2 H, CH_2), 2.32 (t, J = 7.7 Hz, 2 H, CH_2), 2.10 (s, 3 H, OAc), 1.56-1.40 (m, 7 H, $CH_2 \times 2$ and CH_3), 1.40-1.16 (m, 18 H, $CH_2 \times 9$), 0.98-0.80 (m, 6 H, $CH_3 \times 2$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.6, 171.7, 170.0, 157.4, 131.5, 85.0, 57.5, 48.7, 44.7, 31.8, 30.1, 29.5, 29.4, 29.3, 29.2, 29.0, 25.2, 23.6, 23.2, 22.6, 22.5, 20.6, 14.0, 13.7; IR (neat) ν (cm^{-1}) 2955, 2924, 2852, 1768, 1747, 1715, 1682, 1461, 1455, 1406, 1368, 1225, 1129, 1035; MS (EI): *m/z* (%) 422 (M^+, 4.92), 43 (100); HRMS Calcd for $C_{25}H_{42}O_5$ (M^+): 422.3032; Found: 422.3032.

4.5 Synthesis of 4-(2-acetoxy-4-oxo-5-phenylpentan-2-yl)-3-propyl-2(*5H*)-furanone **5ca** (fjj-1-076)

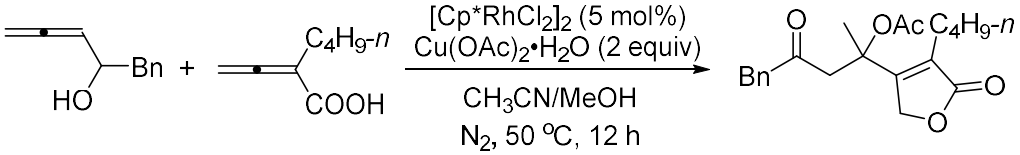


1c, 1 mmol **3a**, 1.5 equiv **5ca**, 50%

Following **Typical Procedure I**, the reaction of **1c** (160.2 mg, 1.0 mmol), **3a** (189.2 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (31.2 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (399.8 mg, 2.0 mmol) in CH_3CN (2.7 mL)/MeOH (0.13 mL) afforded **5ca** (173.0 mg, 50%) as an oil [eluent: petroleum ether/ethyl acetate = 10/1 (500 mL) to 5/1 (1000 mL)]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37-7.21 (m, 3 H, ArH), 7.14 (d, J = 6.6 Hz, 2 H, ArH), 4.91 (d, J = 14.7 Hz, 1 H, one proton of OCH_2), 4.86 (d, J = 14.4 Hz, 1 H, one proton of OCH_2),

3.72 (s, 2 H, CH_2), 3.10 (d, J = 15.3 Hz, 1 H, one proton of CH_2), 2.77 (d, J = 15.3 Hz, 1 H, one proton of CH_2), 2.29 (t, J = 7.5 Hz, 2 H, CH_2), 2.04 (s, 3 H, OAc), 1.62-1.43 (m, 5 H, CH_2, CH_3), 0.91 (t, J = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.6, 171.6, 169.9, 157.5, 133.2, 131.2, 129.4, 128.7, 127.1, 84.9, 57.3, 51.4, 47.6, 25.6, 25.1, 21.2, 20.5, 13.6; IR (neat) ν (cm^{-1}) 3089, 3063, 3030, 2962, 2935, 2873, 1748, 1715, 1681, 1603, 1497, 1455, 1367, 1309, 1222, 1131, 1048, 1026; MS (EI): *m/z* (%) 344 (M^+, 7.29), 211 (100); HRMS Calcd for $C_{20}H_{24}O_5$ (M^+): 344.1624; Found: 344.1625.

4.6 Synthesis of 4-(2-acetoxy-4-oxo-5-phenylpentan-2-yl)-3-butyl-2(*5H*)-furanone **5cb** (fjj-1-087)

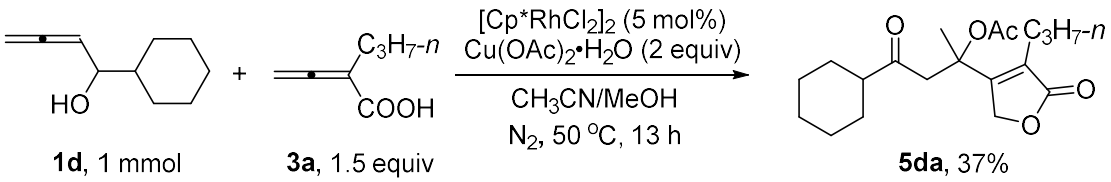


1c, 1 mmol **3b,** 1.5 equiv **5cb,** 50%

Following **Typical Procedure I**, the reaction of **1c** (159.2 mg, 1.0 mmol), **3b** (210.6 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (399.7 mg, 2.0 mmol) in CH_3CN (2.7 mL)/MeOH (0.13 mL) afforded **5cb** (179.7 mg, 50%) as an oil [eluent: petroleum ether/ethyl acetate = 15/1 (500 mL) to 8/1 (500 mL), then 5/1 (1000 mL)]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38-7.23 (m, 3 H, ArH), 7.17-7.10 (m, 2 H, ArH), 4.91 (d, J = 14.4 Hz, 1 H, one proton of OCH_2), 4.86 (d, J = 14.4 Hz, 1 H, one proton of OCH_2), 3.72 (s, 2 H, CH_2), 3.10 (d, J = 15.0 Hz, 1 H, one proton of CH_2), 2.76 (d, J = 15.3 Hz, 1 H, one proton of CH_2), 2.31 (t, J = 7.5 Hz, 2 H, CH_2), 2.05 (s, 3 H, OAc), 1.56-1.39 (m, 5 H, CH_2 and CH_3), 1.39-1.22 (m, 2 H, CH_2), 0.91 (t, J = 7.1

Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.7, 171.7, 169.9, 157.3, 133.2, 131.5, 129.5, 128.7, 127.2, 85.0, 57.4, 51.5, 47.7, 30.1, 25.1, 23.6, 22.4, 20.5, 13.7; IR (neat) *v* (cm^{-1}) 3092, 3062, 3030, 2958, 2929, 2865, 1748, 1714, 1603, 1497, 1455, 1362, 1308, 1220, 1124, 1034; MS (EI): *m/z* (%) 358 (M^+, 5.66), 225 (100); HRMS Calcd for $C_{21}H_{26}O_5$ (M^+): 358.1780; Found: 358.1779.

4.7 Synthesis of 4-(2-acetoxy-4-cyclohexyl-4-oxobutan-2-yl)-3-propyl-2(*5H*)-furanone **5da** (fjj-1-066)

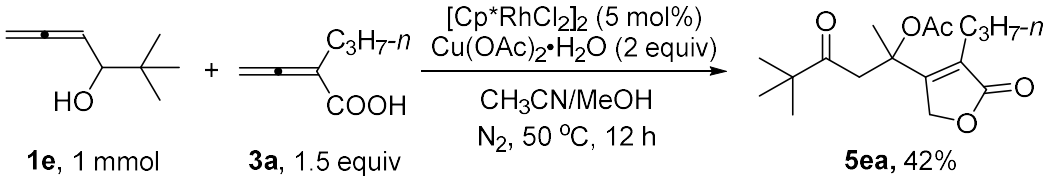


1d, 1 mmol **3a**, 1.5 equiv **5da**, 37%

Following **Typical Procedure I**, the reaction of **1d** (151.6 mg, 1.0 mmol), **3a** (189.3 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (399.6 mg, 2.0 mmol) in CH_3CN (2.7 mL)/MeOH (0.13 mL) afforded **5da** (124.7 mg, 37%) as an oil [eluent: petroleum ether/ethyl acetate = 10/1 (500 mL) to 5/1 (500 mL)]: ^1H NMR (300 MHz, $CDCl_3$) δ 4.98 (d, *J* = 14.4 Hz, 1 H, one proton of OCH_2), 4.92 (d, *J* = 14.4 Hz, 1 H, one proton of OCH_2), 3.16 (d, *J* = 15.3 Hz, 1 H, one proton of CH_2), 2.75 (d, *J* = 15.3 Hz, 1 H, one proton of CH_2), 2.42-2.25 (m, 3 H, CH and CH_2), 2.10 (s, 3 H, OAc), 1.89-1.60 (m, 5 H, CH_2 × 2 and one proton of CH_2), 1.60-1.42 (m, 5 H, CH_2 and CH_3), 1.36-1.10 (m, 5 H, CH_2 × 2 and one proton of CH_2), 0.94 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 209.3, 171.8, 170.0, 157.8, 131.1, 85.1, 57.6, 51.8, 46.6, 28.1, 27.6, 25.7, 25.6, 25.5, 25.3, 21.3, 20.6, 13.7; IR (neat) *v* (cm^{-1}) 2959,

2930, 2855, 1748, 1706, 1451, 1372, 1291, 1221, 1136, 1028; MS (EI): *m/z* (%) 336 (M^+, 4.52), 83 (100); HRMS Calcd for $C_{19}H_{28}O_5$ (M^+): 336.1937; Found: 336.1934.

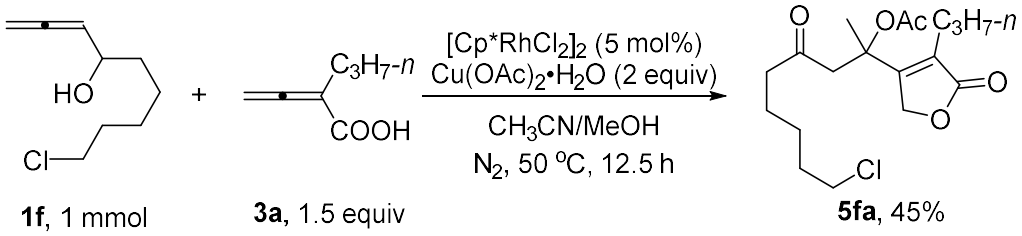
4.8 Synthesis of 4-(2-acetoxy-5,5-dimethyl-4-oxohexan-2-yl)-3-propyl-2(*5H*)-furanone **5ea** (fjj-1-068)



1e, 1 mmol **3a**, 1.5 equiv **5ea**, 42%

Following **Typical Procedure I**, the reaction of **1e** (127.8 mg, 1.0 mmol), **3a** (189.3 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (31.3 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (398.3 mg, 2.0 mmol) in CH_3CN (2.7 mL)/MeOH (0.13 mL) afforded **5ea** (130.8 mg, 42%) as an oil [eluent: petroleum ether/ethyl acetate = 10/1 (500 mL) to 5/1 (600 mL)]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.00 (d, *J* = 14.1 Hz, 1 H, one proton of OCH_2), 4.94 (d, *J* = 14.4 Hz, 1 H, one proton of OCH_2), 3.31 (d, *J* = 16.5 Hz, 1 H, one proton of CH_2), 2.76 (d, *J* = 16.2 Hz, 1 H, one proton of CH_2), 2.32 (t, *J* = 7.5 Hz, 2 H, CH_2), 2.10 (s, 3 H, OAc), 1.64-1.48 (m, 5 H, CH_2 and CH_3), 1.11 (s, 9 H, $CH_3 \times 3$), 0.95 (t, *J* = 7.5 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 210.6, 172.0, 170.0, 158.1, 130.7, 84.9, 57.7, 44.6, 42.4, 25.9, 25.72, 25.67, 21.3, 20.6, 13.7; IR (neat) *v* (cm^{-1}) 2963, 2933, 2870, 1748, 1713, 1507, 1479, 1463, 1452, 1394, 1367, 1261, 1222, 1170, 1123, 1051, 1026; MS (EI): *m/z* (%) 310 (M^+, 3.70), 43 (100); HRMS Calcd for $C_{17}H_{26}O_5$ (M^+): 310.1780; Found: 310.1780.

4.9 Synthesis of 4-(2-acetoxy-9-chloro-4-oxononan-2-yl)-3-propyl-2(*5H*)-furanone **5fa** (fjj-1-072)

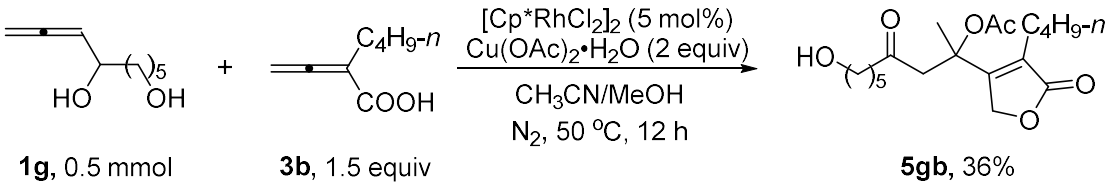


1f, 1 mmol **3a**, 1.5 equiv **5fa**, 45%

Following **Typical Procedure I**, the reaction of **1f** (173.4 mg, 1.0 mmol), **3a** (189.5 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (31.2 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (400.8 mg, 2.0 mmol) in CH_3CN (2.7 mL)/MeOH (0.13 mL) afforded **5fa** (161.2 mg, 45%) as an oil [eluent: petroleum ether/ethyl acetate = 10/1 (500 mL) to 5/1 (1500 mL)]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.95 (dd, J_1 = 14.4 Hz, J_2 = 14.1 Hz, 2 H, OCH_2), 3.52 (t, J = 6.8 Hz, 2 H, CH_2Cl), 3.07 (d, J = 15.0 Hz, 1 H, one proton of CH_2), 2.76 (d, J = 15.0 Hz, 1 H, one proton of CH_2), 2.48 (t, J = 7.1 Hz, 2 H, CH_2), 2.31 (t, J = 7.5 Hz, 2 H, CH_2), 2.11 (s, 3 H, OAc), 1.81-1.68 (m, 2 H, CH_2), 1.62-1.45 (m, 7 H, $CH_2 \times 2$ and CH_3), 1.45-1.30 (m, 2 H, CH_2), 0.94 (t, J = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.9, 171.6, 169.8, 157.6, 130.9, 84.8, 57.3, 48.4, 44.6, 44.1, 32.1, 25.9, 25.5, 25.0, 22.1, 21.2, 20.4, 13.5; IR (neat) ν (cm^{-1}) 2955, 2928, 2873, 1747, 1712, 1682, 1455, 1406, 1373, 1257, 1219, 1134, 1100, 1066, 1026; MS (EI): *m/z* (%) 360 [M(^{37}Cl)$^+$, 1.01], 358 [M(^{35}Cl)$^+$, 2.08], 43 (100); HRMS Calcd for $C_{18}H_{27}{}^{35}ClO_5$ (M^+): 358.1547; Found: 358.1548.

4.10 Synthesis of 4-(2-acetoxy-9-hydroxy-4-oxononan-2-yl)-3-butyl-2(*5H*)-furanone

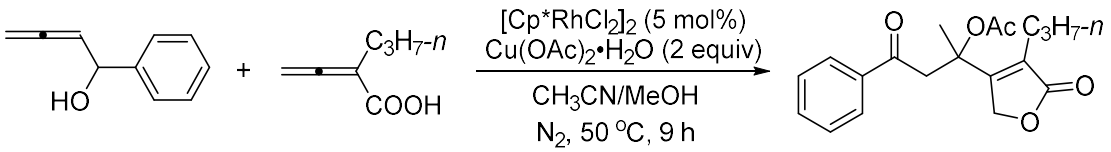
5gb (fjj-1-111)



1g, 0.5 mmol **3b**, 1.5 equiv **5gb**, 36%

Following **Typical Procedure I**, the reaction of **1g** (77.5 mg, 0.5 mmol), **3b** (105.5 mg, 0.75 mmol), $[Cp^*RhCl_2]_2$ (15.9 mg, 0.025 mmol), and $Cu(OAc)_2 \cdot H_2O$ (199.8 mg, 2.0 mmol) in CH_3CN (1.35 mL)/MeOH (67.5 μL) afforded **5gb** (65.9 mg, 36%, 95% purity) as an oil [eluent: petroleum ether/ethyl acetate = 5/1 (500 mL) to 1/1 (500 mL), then 1/2 (500 mL)]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.94 (dd, J_1 = 14.7 Hz, J_2 = 14.7 Hz, 2 H, OCH_2), 3.71 (s, 2 H, OCH_2), 3.07 (d, J = 14.4 Hz, 1 H, one proton of CH_2), 2.95-2.60 (m, 2 H, one proton of CH_2 and OH), 2.48 (t, J = 6.7 Hz, 2 H, CH_2), 2.32 (t, J = 8.1 Hz, 2 H, CH_2), 2.11 (s, 3 H, OAc), 1.64-1.42 (m, 9 H, CH_3 and CH_2 × 3), 1.42-1.22 (m, 4 H, CH_2 × 2), 0.93 (t, J = 7.1 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.5, 171.9, 170.0, 157.6, 131.3, 85.1, 62.4, 57.4, 48.6, 44.4, 32.3, 30.1, 25.1, 25.0, 23.5, 22.7, 22.4, 20.6, 13.7; IR (neat) ν (cm^{-1}) 3479, 2935, 2865, 1747, 1454, 1369, 1227, 1132, 1039; MS (EI): *m/z* (%) 354 (M^+, 0.40), 43 (100); HRMS Calcd for $C_{19}H_{30}O_6$ (M^+): 354.2042; Found: 354.2039.

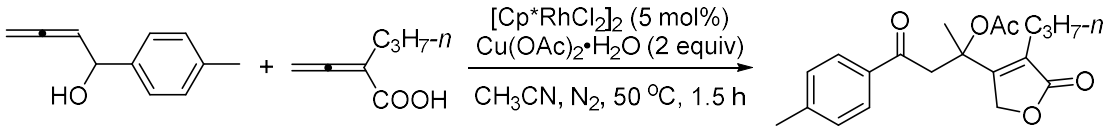
4.11 Synthesis of 4-(2-acetoxy-4-oxo-4-phenylbutan-2-yl)-3-propyl-2(*5H*)-furanone **5ha** (fjj-1-064)



1h, 1 mmol **3a**, 1.5 equiv **5ha**, 50%

Following **Typical Procedure I**, the reaction of **1h** (146.3 mg, 1.0 mmol), **3a** (189.0 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (30.9 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (399.2 mg, 2.0 mmol) in CH_3CN (2.7 mL)/MeOH (0.13 mL) afforded **5ha** (163.3 mg, 50%) as an oil [eluent: petroleum ether/ethyl acetate = 10/1 (500 mL) to 5/1 (1500 mL)]: ^1H NMR (300 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.1 Hz, 2 H, ArH), 7.60-7.50 (m, 1 H, ArH), 7.44 (t, *J* = 7.1 Hz, 2 H, ArH), 5.03 (d, *J* = 14.1 Hz, 1 H, one proton of OCH_2), 4.96 (d, *J* = 14.4 Hz, 1 H, one proton of OCH_2), 3.72 (d, *J* = 15.6 Hz, 1 H, one proton of CH_2), 3.29 (d, *J* = 15.6 Hz, 1 H, one proton of CH_2), 2.31 (t, *J* = 7.7 Hz, 2 H, CH_2), 1.97 (s, 3 H, OAc), 1.63 (s, 3 H, CH_3), 1.60-1.42 (m, 2 H, CH_2), 0.92 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.4, 171.6, 169.8, 157.1, 136.6, 133.3, 131.4, 128.4, 128.1, 84.9, 57.3, 44.1, 25.49, 25.46, 21.1, 20.3, 13.5; IR (neat) *v* (cm^{-1}) 3061, 2962, 2933, 2869, 1748, 1694, 1682, 1597, 1581, 1448, 1364, 1223, 1123, 1057, 1023; MS (EI): *m/z* (%) 330 (M^+, 3.97), 105 (100); HRMS Calcd for $C_{19}H_{22}O_5$ (M^+): 330.1467; Found: 330.1468.

4.12 Synthesis of 4-(2-acetoxy-4-oxo-4-(*p*-tolyl)butan-2-yl)-3-propyl-2(*5H*)-furanone **5ia** (fjj-2-022)

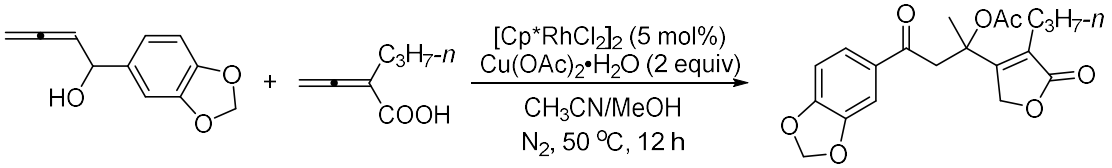


1i, 1 mmol **3a**, 1.5 equiv **5ia**, 46%

Typical Procedure II: To a dry Schlenk tube were added $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), $Cu(OAc)_2 \cdot H_2O$ (399.3 mg, 2.0 mmol), and **3a** (189.2 mg, 1.5 mmol). The reaction vessel was degassed under vacuum and backfilled with nitrogen for three times. Then **1i** (160.3 mg, 1.0 mmol) and CH_3CN (2.7 mL) were added under nitrogen atmosphere sequentially. After being continuously stirred at 50 °C for 1.5 h, the reaction was complete as monitored by thin layer chromatography (TLC). After filtration through a short column of silica gel eluted with ethyl acetate (20 mL × 3), the combined filtrate was concentrated in vacuo and the crude residual was purified by chromatography on silica gel [eluent: petroleum ether/ethyl ether = 3/1 (1000 mL) to petroleum ether/ethyl acetate = 2/1 (300 mL)] to afford **5ia** (160.2 mg, 46%) as an oil: ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.79 (d, *J* = 8.4 Hz, 2 H, ArH), 7.24 (d, *J* = 8.1 Hz, 2 H, ArH), 5.03 (d, *J* = 14.1 Hz, 1 H, one proton of OCH_2), 4.97 (d, *J* = 14.1 Hz, 1 H, one proton of OCH_2), 3.70 (d, *J* = 15.3 Hz, 1 H, one proton of CH_2), 3.21 (d, *J* = 15.3 Hz, 1 H, one proton of CH_2), 2.40 (s, 3 H, CH_3), 2.30 (t, *J* = 7.5 Hz, 2 H, CH_2), 2.00 (s, 3 H, OAc), 1.63 (s, 3 H, CH_3), 1.60-1.42 (m, 2 H, CH_2), 0.92 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.1, 171.6, 169.9, 157.3, 144.3, 134.3, 131.4, 129.2, 128.4, 85.0, 57.4, 44.3, 25.6, 25.5, 21.5, 21.2, 20.4, 13.6; IR (neat) *v* (cm^{-1}) 3032, 2962, 2934, 2873, 1747, 1690, 1669, 1607, 1572, 1451, 1407, 1366, 1224, 1183, 1121, 1057, 1028; MS (EI): *m/z* (%) 344 (M^+, 8.82), 119 (100); HRMS Calcd for $C_{20}H_{24}O_5$ (M^+):

344.1624; Found: 344.1623.

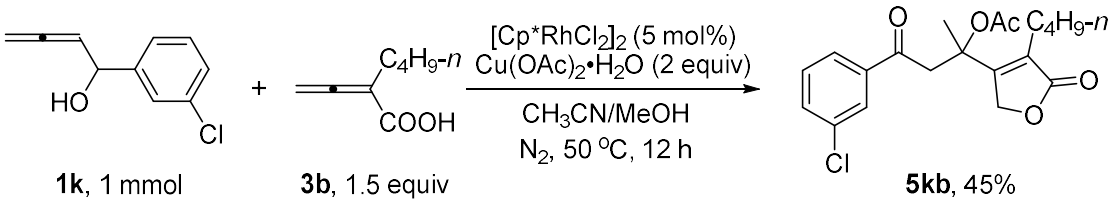
4.13 Synthesis of 4-(2-acetoxy-4-(3,4-(methylenedioxy)phenyl)-4-oxobutan-2-yl)-3-propyl-2(*5H*)-furanone **5ja** (fjj-1-054)



1j, 1 mmol **3a**, 1.5 equiv **5ja**, 49%

Following **Typical Procedure I**, the reaction of **1j** (189.0 mg, 1.0 mmol), **3a** (189.1 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (30.8 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (399.2 mg, 2.0 mmol) in CH_3CN (2.7 mL)/MeOH (0.13 mL) afforded **5ja** (183.7 mg, 49%) as an oil [eluent: petroleum ether/ethyl acetate = 20/1 (1000 mL) to 5/1 (1000 mL), then 2/1 (200 mL)]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.52 (dd, J_1 = 8.1 Hz, J_2 = 1.5 Hz, 1 H, ArH), 7.34 (d, J = 1.5 Hz, 1 H, ArH), 6.84 (d, J = 8.1 Hz, 1 H, ArH), 6.04 (s, 2 H, O_2CH_2), 5.01 (dd, J_1 = 14.7 Hz, J_2 = 14.4 Hz, 2 H, OCH_2), 3.66 (d, J = 15.3 Hz, 1 H, one proton of CH_2), 3.14 (d, J = 15.3 Hz, 1 H, one proton of CH_2), 2.30 (t, J = 7.7 Hz, 2 H, CH_2), 2.04 (s, 3 H, OAc), 1.63 (s, 3 H, CH_3), 1.58-1.43 (m, 2 H, CH_2), 0.91 (t, J = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 193.5, 171.7, 170.0, 157.5, 152.1, 148.2, 131.7, 131.4, 125.2, 107.9, 107.8, 101.9, 85.1, 57.6, 44.5, 25.64, 25.58, 21.3, 20.5, 13.6; IR (neat) v (cm^{-1}) 3080, 2963, 2929, 2874, 1748, 1682, 1604, 1505, 1489, 1446, 1367, 1256, 1220, 1108, 1036; MS (EI): *m/z* (%) 374 (M^+, 8.42), 149 (100); HRMS Calcd for $C_{20}H_{22}O_7Na$ $(M+Na)^+$: 397.1258; Found: 397.1257.

4.14 Synthesis of 4-(2-acetoxy-4-(3-chlorophenyl)-4-oxobutan-2-yl)-3-butyl-2(*5H*)-furanone **5kb** (fjj-1-095)

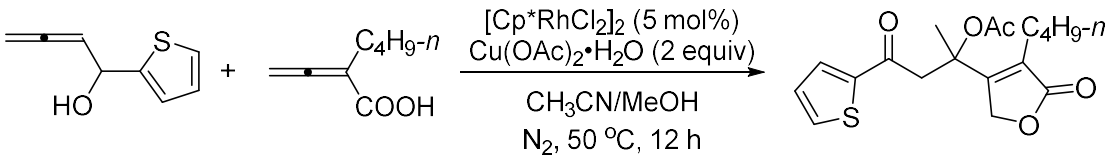


1k, 1 mmol **3b**, 1.5 equiv **5kb**, 45%

Following **Typical Procedure I**, the reaction of **1k** (180.2 mg, 1.0 mmol), **3b** (210.3 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (31.1 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (399.2 mg, 2.0 mmol) in CH_3CN (2.7 mL)/MeOH (0.13 mL) afforded **5kb** (170.3 mg, 45%) as an oil [eluent: petroleum ether/ethyl acetate = 10/1 (500 mL) to 5/1 (1000 mL), then 4/1 (500 mL)]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.87-7.83 (m, 1 H, ArH), 7.78 (d, *J* = 3.9 Hz, 1 H, ArH), 7.54 (d, *J* = 7.8 Hz , 1 H, ArH), 7.41 (t, *J* = 8.0 Hz, 1 H, ArH), 5.02 (d, *J* = 14.1 Hz, 1 H, one proton of OCH_2), 4.96 (d, *J* = 14.1 Hz, 1 H, one proton of OCH_2), 3.70 (d, *J* = 15.9 Hz, 1 H, one proton of CH_2), 3.23 (d, *J* = 15.6 Hz, 1 H, one proton of CH_2), 2.32 (t, *J* = 7.7 Hz, 2 H, CH_2), 2.02 (s, 3 H, OAc), 1.64 (s, 3 H, CH_3), 1.54-1.38 (m, 2 H, CH_2), 1.38-1.22 (m, 2 H, CH_2), 0.92 (t, *J* = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 194.4, 171.6, 170.0, 156.7, 138.3, 135.0, 133.4, 132.1, 130.0, 128.3, 126.6, 84.9, 57.4, 44.6, 30.1, 25.6, 23.6, 22.4, 20.5; 13.8; IR (neat) *v* (cm^{-1}) 3062, 2958, 2929, 2872, 1755, 1748, 1698, 1682, 1570, 1424, 1366, 1224, 1123, 1031; MS (EI): *m/z* (%) 380 [M(^{37}Cl)$^+$, 2.47], 378 [M(^{35}Cl)$^+$, 7.20], 139 (100); HRMS Calcd for $C_{20}H_{23}O_5{}^{35}Cl$ (M^+): 378.1234; Found: 378.1234.

4.15 Synthesis of 4-(2-acetoxy-4-oxo-4-(thiophen-2-yl)butan-2-yl)-3-butyl-2(*5H*)-

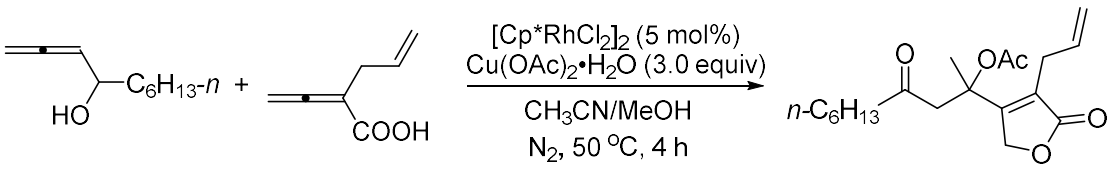
furanone **5lb** (fjj-1-090)



1l, 1 mmol **3b**, 1.5 equiv **5lb**, 46%

Following **Typical Procedure I**, the reaction of **1l** (152.4 mg, 1.0 mmol), **3b** (210.3 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (31.1 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (399.2 mg, 2.0 mmol) in CH_3CN (2.7 mL)/MeOH (0.13 mL) afforded **5lb** (163.0 mg, 46%) as an oil [eluent: petroleum ether/ethyl acetate = 5/1 (1500 mL)]: ^1H NMR (300 MHz, $CDCl_3$) δ 7.74 (d, *J* = 3.9 Hz, 1 H, ArH), 7.67 (d, *J* = 4.8 Hz, 1 H, ArH), 7.13 (t, *J* = 4.5 Hz, 1 H, ArH), 5.03 (d, *J* = 14.4 Hz, 1 H, one proton of OCH_2), 4.98 (d, *J* = 14.4 Hz, 1 H, one proton of OCH_2), 3.63 (d, *J* = 15.0 Hz, 1 H, one proton of CH_2), 3.19 (d, *J* = 15.0 Hz, 1 H, one proton of CH_2), 2.31 (t, *J* = 7.5 Hz, 2 H, CH_2), 2.04 (s, 3 H, OAc), 1.63 (s, 3 H, CH_3), 1.52-1.37 (m, 2 H, CH_2), 1.37-1.22 (m, 2 H, CH_2), 0.90 (t, *J* = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 188.1, 171.5, 169.9, 157.0, 144.1, 134.8, 133.4, 131.7, 128.3, 84.9, 57.4, 45.7, 30.0, 25.3, 23.4, 22.3, 20.4; 13.7; IR (neat) *v* (cm^{-1}) 3094, 2958, 2929, 2872, 1747, 1667, 1651, 1519, 1455, 1417, 1362, 1227, 1109, 1051; MS (EI): *m/z* (%) 350 (M^+, 7.34), 111 (100); HRMS Calcd for $C_{18}H_{22}O_5S$ (M^+): 350.1188; Found: 350.1189.

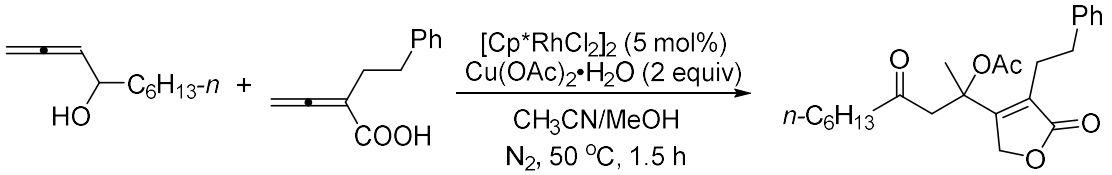
4.16 Synthesis of 4-(2-acetoxy-4-oxodecan-2-yl)-3-allyl-2(*5H*)-furanone **5ad** (fjj-1-135, fjj-2-008)



1a, 1.2 equiv **3d**, 1 mmol **5ad**, 37%

Following **Typical Procedure I**, the reaction of **1a** (185.0 mg, 1.2 mmol), **3d** (124.2 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.1 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (599.1 mg, 3.0 mmol) in CH_3CN (2.7 mL)/MeOH (0.13 mL) afforded **5ad** (126.0 mg, 37%) as an oil [eluent: petroleum ether/ethyl acetate = 5/1 (1000 mL)]: ^1H NMR (300 MHz, $CDCl_3$) δ 5.91-5.72 (m, 1 H, =CH), 5.17-5.03 (m, 2 H, $=CH_2$), 4.95 (s, 2 H, OCH_2), 3.13-3.01 (m, 3 H, CH_2 and one proton of CH_2), 2.77 (d, J = 15.0 Hz, 1 H, one proton of CH_2), 2.44 (t, J = 7.4 Hz, 2 H, CH_2), 2.10 (s, 3 H, OAc), 1.60-14.1 (m, 5 H, CH_3 and CH_2), 1.37-1.15 (m, 6 H, $CH_2 \times 3$), 0.87 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.4, 171.2, 169.8, 159.0, 132.7, 128.3, 116.6, 85.0, 57.4, 48.4, 44.5, 31.3, 28.4, 27.6, 25.1, 23.0, 22.2, 20.4, 13.8; IR (neat) ν (cm^{-1}) 3077, 2955, 2925, 2858, 1755, 1716, 1640, 1368, 1218, 1122, 1047; MS (EI): *m/z* (%) 336 (M^+, 1.10), 164 (100); HRMS Calcd for $C_{19}H_{28}O_5$ (M^+): 336.1937; Found: 336.1934.

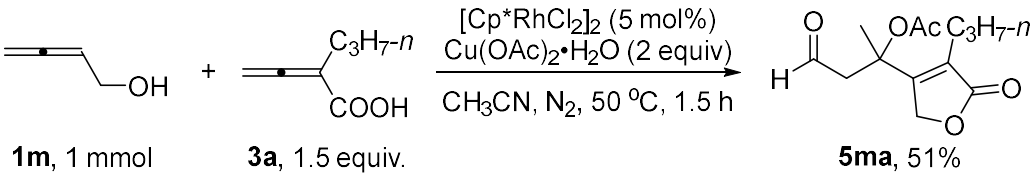
4.17 Synthesis of 4-(2-acetoxy-4-oxodecan-2-yl)-3-phenethyl-2(*5H*)-furanone **5ae** (fjj-2-018)



1a **3e**, 1.5 equiv **5ae**, 51%, 97% purity

Following **Typical Procedure I**, the reaction of **1a** (154.0 mg, 1.0 mmol), **3e** (282.4 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (30.9 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (399.8 mg, 2.0 mmol) in CH_3CN (2.7 mL)/MeOH (0.13 mL) afforded **5ae** (210.2 mg, 51%, 97% purity) as an oil [eluent: petroleum ether/ethyl ether = 3/1 (1000 mL)]: ^1H NMR (300 MHz, $CDCl_3$) δ 7.37-7.03 (m, 5 H, ArH), 4.57 (d, J = 14.4 Hz, 1 H, one proton of OCH_2), 4.46 (d, J = 14.1 Hz, 1 H, one proton of OCH_2), 2.93 (d, J = 15.0 Hz, 1 H, one proton of CH_2), 2.88-2.78 (m, 2 H, CH_2), 2.74-2.57 (m, 3 H, one proton of CH_2 and CH_2), 2.42 (td, J_1 = 7.3 Hz, J_2 = 2.1 Hz, 2 H, CH_2), 2.02 (s, 3 H, OAc), 1.57-1.37 (m, 5 H, CH_3 and CH_2), 1.36-1.16 (m, 6 H, CH_2 × 3), 0.87 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.4, 171.5, 169.7, 158.6, 140.5, 129.9, 128.6, 128.3, 126.2, 85.0, 57.0, 48.6, 44.5, 33.6, 31.5, 28.6, 25.8, 24.7, 23.1, 22.3, 20.5, 13.9; IR (neat) v (cm^{-1}) 3063, 3027, 2931, 2859, 1748, 1712, 1601, 1496, 1455, 1368, 1220, 1047; MS (EI): m/z (%) 400 (M^+, 6.12), 91 (100); HRMS Calcd for $C_{24}H_{32}O_5$ (M^+): 400.2250; Found: 400.2248.

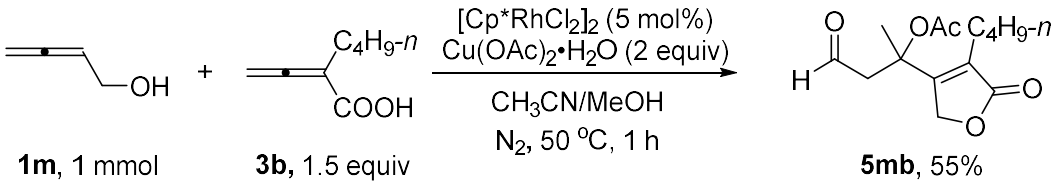
4.18 Synthesis of 4-(2-acetoxy-4-oxobutan-2-yl)-3-propyl-2(*5H*)-furanone **5ma** (fjj-2-023)



Following **Typical Procedure II**, the reaction of **1m** (70.3 mg, 1.0 mmol), **3a** (189.4 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (399.5 mg, 2.0 mmol) in CH_3CN (2.7 mL) afforded **5ma** (129.8 mg, 51%) as an oil [eluent:

petroleum ether/ethyl ether = 2/1 (1000 mL) to 1/1 (500 mL)]: ^{1}H NMR (300 MHz, $CDCl_3$) 9.63 (t, J = 2.4 Hz, 1 H, CHO), 4.97 (d, J = 14.1 Hz, 1 H, one proton of OCH_2), 4.87 (d, J = 14.1 Hz, 1 H, one proton of OCH_2), 2.98 (dd, J_1 = 16.1 Hz, J_2 = 2.3 Hz, 1 H, one proton of CH_2), 2.74 (dd, J_1 = 15.9 Hz, J_2 = 2.4 Hz, 1 H, one proton of CH_2), 2.33 (t, 2 H, J = 7.5 Hz, CH_2), 2.11 (s, 3 H, OAc), 1.66-1.41 (m, 5 H, CH_2 and CH_3), 0.93 (t, J = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.0, 171.1, 169.8, 156.9, 132.1, 84.4, 56.7, 49.2, 25.5, 24.7, 21.2, 20.3, 13.5; IR (neat) v (cm^{-1}) 2964, 2929, 2875, 2744, 1747, 1676, 1455, 1368, 1222, 1136, 1064, 1030; MS (EI): *m/z* (%) 254 (M^+, 2.49), 43 (100); HRMS Calcd for $C_{13}H_{18}O_5$ (M^+): 254.1154; Found: 254.1156.

4.19 Synthesis of 4-(2-acetoxy-4-oxobutan-2-yl)-3-butyl-2(*5H*)-furanone **5mb** (fjj-2-001)

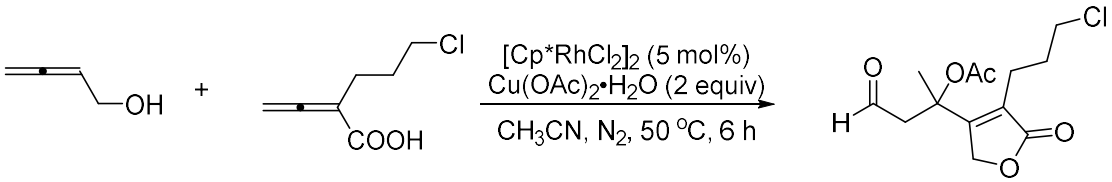


1m, 1 mmol **3b,** 1.5 equiv **5mb**, 55%

Following **Typical Procedure I**, the reaction of **1m** (70.2 mg, 1.0 mmol), **3b** (210.3 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (30.9 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (399.1 mg, 2.0 mmol) in CH_3CN (2.7 mL)/MeOH (0.13 mL) afforded **5mb** (149.0 mg, 55%) as an oil [eluent: petroleum ether/ethyl ether = 4/1 (500 mL) to 2/1 (200 mL)]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.67-9.56 (m, 1 H, CHO), 5.02-4.80 (m, 2 H, OCH_2), 3.03-2.90 (m, 1 H, one proton of CH_2), 2.72 (dd, J_1 = 15.9 Hz, J_2 = 2.1 Hz, 1 H, one proton of CH_2), 2.40-2.27 (m, 2 H, CH_2), 2.14-2.03 (m, 3 H, OAc), 1.61-1.54 (m, 3 H, CH_3), 1.54-1.42

(m, 2 H, CH_2), 1.41-1.23 (m, 2 H, CH_2), 0.97-0.86 (m, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.1, 171.2, 169.9, 156.6, 132.5, 84.4, 56.8, 49.3, 30.1, 24.7, 23.5, 22.4, 20.4, 13.6; IR (neat) *v* (cm^{-1}) 3480, 2959, 2925, 2873, 2745, 1748, 1674, 1455, 1380, 1227, 1137, 1032; MS (EI): *m/z* (%) 268 (M^+, 4.24), 43 (100); HRMS Calcd for $C_{14}H_{20}O_5$ (M^+): 268.1311; Found: 268.1311.

4.20 Synthesis of 4-(2-acetoxy-4-oxobutan-2-yl)-3-(3-chloropropyl)-2(*5H*)-furanone. **5mc** (fjj-2-094)

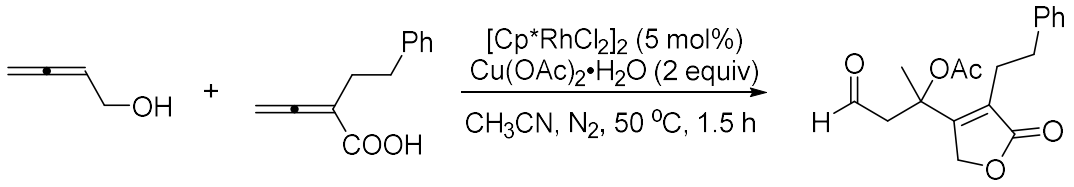


1m, 1 mmol **1c**, 1.5 equiv. **5mc**, 38%, 95% purity

Following **Typical Procedure II**, the reaction of **1m** (69.3 mg, 1.0 mmol), **3c** (240.4 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (399.3 mg, 2.0 mmol) in CH_3CN (2.7 mL) afforded **5mc** (115.0 mg, 38%, 95% purity) as an oil [200~300 mesh silica gel, eluent: petroleum ether/ethyl ether = 5/1 (1000 mL) to 3/1 (500 mL), then 1/1 (1000 mL)]: ^1H NMR (300 MHz, $CDCl_3$) δ 9.64 (d, *J* = 1.2 Hz, 1 H, CHO), 4.99 (d, *J* = 14.4 Hz, 1 H, one proton of OCH_2), 4.89 (d, *J* = 14.1 Hz, 1 H, one proton of OCH_2), 3.57 (t, *J* = 6.0 Hz, 2 H, CH_2Cl), 3.02 (dd, J_1 = 16.2 Hz, J_2 = 1.5 Hz, 1 H, one proton of CH_2), 2.77 (dd, J_1 = 16.2 Hz, J_2 = 2.1 Hz, 1 H, one proton of CH_2), 2.62-2.46 (m, 2 H, CH_2), 2.17-1.97 (m, 5 H, OAc and CH_2), 1.57 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.8, 171.0, 169.9, 158.0, 130.5, 84.6, 56.8, 49.0, 44.0,

29.9, 24.7, 21.1, 20.4; IR (neat) v (cm^{-1}) 2963, 2933, 2852, 2746, 1747, 1680, 1446, 1369, 1306, 1223, 1134, 1064; MS (EI): *m/z* (%) 290 [M(^{37}Cl)$^+$, 0.2], 288 [M(^{35}Cl)$^+$, 0.6], 43 (100); HRMS Calcd for $C_{13}H_{17}O_5{}^{35}Cl$ (M^+): 288.0765; Found: 288.0766.

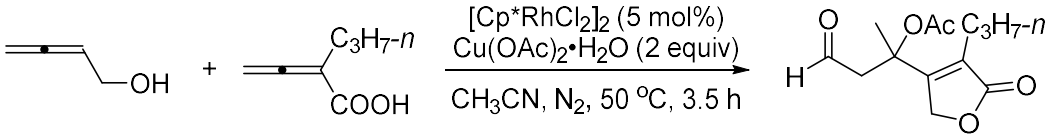
4.21 Synthesis of 4-(2-acetoxy-4-oxobutan-2-yl)-3-phenethyl-2(*5H*)-furanone **5me** (fjj-2-020)



1m, 1 mmol **3e**, 1.5 equiv **5me**, 46%

Following **Typical Procedure II**, the reaction of **1m** (69.7 mg, 1.0 mmol), **3e** (282.5 mg, 1.5 mmol), $[Cp^*RhCl_2]_2$ (31.1 mg, 0.05 mmol), and $Cu(OAc)_2 \cdot H_2O$ (399.3 mg, 2.0 mmol) in CH_3CN (2.7 mL) afforded **5me** (145.5 mg, 46%) as an oil [eluent: petroleum ether/ethyl ether = 2/1 (1000 mL) to petroleum ether/ethyl acetate = 1/1 (500 mL)]: ^1H NMR (300 MHz, $CDCl_3$) δ 9.53 (t, J = 2.4 Hz, 1 H, CHO), 7.33-7.06 (m, 5 H, ArH), 4.47 (d, J = 14.4 Hz, 1 H, one proton of OCH_2), 4.30 (d, J = 14.1 Hz, 1 H, one proton of OCH_2), 2.96-2.78 (m, 3 H, one proton of CH_2 and CH_2), 2.76-2.52 (m, 3 H, one proton of CH_2 and CH_2), 2.02 (s, 3 H, OAc), 1.46 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.1, 171.0, 169.7, 158.0, 140.1, 130.7, 128.7, 128.5, 126.4, 84.6, 56.4, 49.4, 33.4, 25.8, 24.6, 20.4; IR (neat) v (cm^{-1}) 3062, 3027, 2934, 2864, 2747, 1747, 1721, 1603, 1496, 1455, 1368, 1223, 1047; MS (EI): *m/z* (%) 316 (M^+, 3.58), 91 (100); HRMS Calcd for $C_{18}H_{20}O_5$ (M^+): 316.1311; Found: 316.1312.

5. A Gram Scale Reaction

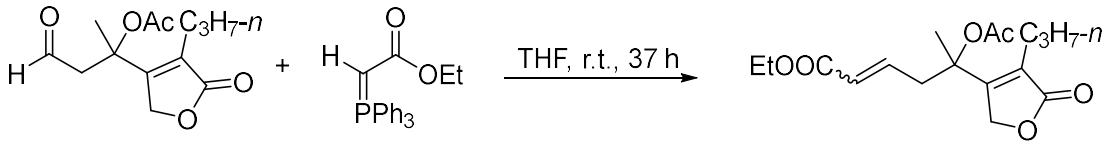


1m, 8 mmol **3a**, 1.5 equiv. **5ma**, 1.0113 g, 50%

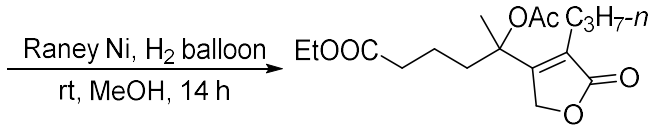
Following **Typical Procedure II**, the reaction of **1m** (559.7 mg, 8 mmol), **3a** (1514.2 mg, 12 mmol), $[Cp^*RhCl_2]_2$ (0.2473 g, 0.4 mmol), and $Cu(OAc)_2 \cdot H_2O$ (3195.3 mg, 16 mmol) in CH_3CN (21.6 mL) afforded **5ma** (1.0113 g, 50%) as an oil [eluent: petroleum ether/ethyl acetate = 5/1 (2000 mL)]: ^1H NMR (300 MHz, $CDCl_3$) 9.63 (s, 1 H, CHO), 4.96 (d, J = 14.1 Hz, 1 H, one proton of OCH_2), 4.85 (d, J = 14.1 Hz, 1 H, one proton of OCH_2), 2.96 (d, J = 15.9 Hz, 1 H, one proton of CH_2), 2.71 (d, J = 15.9 Hz, 1 H, one proton of CH_2), 2.33 (t, 2 H, J = 7.5 Hz, CH_2), 2.11 (s, 3 H, OAc), 1.65-1.46 (m, 5 H, CH_2 and CH_3), 0.93 (t, J = 7.4 Hz, 3 H, CH_3).

6. Synthetic Applications

6.1 Preparation of 4-(2-acetoxy-6-ethoxy-6-oxohexan-2-yl)-3-propyl-2(*5H*)-furanone **7** (fjj-4-017, 018)



5ma, 0.2 mmol 1.2 equiv. **S7**, 83%, *E*/*Z* = 84/16



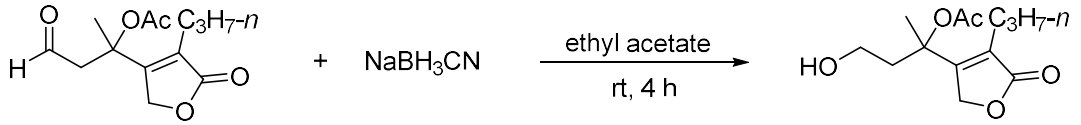
7, 63% from **5ma** to **7**

To a Schlenk tube was added ethyl (triphenylphosphoranylidene)acetate (83.9 mg, 0.24 mmol). After degassed under vacuum and backfilled with nitrogen for three times at room temperature, THF (1 mL) and **5ma** (50.1 mg, 0.2 mmol)/THF (0.5 mL) were added under nitrogen atmosphere. After being continuously stirred at room temperature for 37 h, the reaction was complete as monitored by thin layer chromatography (TLC). The reaction mixture was concentrated in vacuo and the crude residual was purified by chromatography on silica gel [eluent: petroleum ether/ethyl acetate = 5/1 (1000 mL)] to afford the corresponding alkenoate **S7** (53.3 mg, *E*/*Z* = 84/16, 83%).

A Schlenk tube was degassed under vacuum and backfilled with nitrogen for three times at room temperature. Then Raney Ni (5 mg, *Note*: commercial product nickel from *Aladdin* is stabilized with water) was added and washed with MeOH for three times to remove water. Then the Schlenk tube was degassed under vacuum and backfilled with hydrogen for three times, which was followed by the addition of the

above prepared alkenoate **S7** /MeOH (2 mL) under hydrogen atmosphere. After being continuously stirred at room temperature for 14 h, the reaction was complete as monitored by thin layer chromatography (TLC). After filtration through a short column of Celite® pad eluted with ethyl acetate (20 mL × 3), the combined filtrate was concentrated in vacuo and the crude residual was purified by chromatography on silica gel (200~300 mesh) [eluent: petroleum ether/ethyl acetate = 8/1 (600 mL)] to afford **7** (40.7 mg, 63% from two steps) as an oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.91 (d, J = 14.1 Hz, 1 H, one proton of OCH_2), 4.85 (d, J = 14.4 Hz, 1 H, one proton of OCH_2), 4.12 (q, J = 7.2 Hz, 2 H, OCH_2), 2.41-2.20 (m, 4 H, CH_2 × 2), 2.13 (s, 3 H, CH_3), 1.98-1.70 (m, 2 H, CH_2), 1.65-1.35 (m, 7 H, CH_2 × 2 and CH_3), 1.25 (t, J = 7.1 Hz, 3 H, CH_3), 0.93 (t, J = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.8, 172.1, 170.1, 157.4, 131.7, 87.0, 60.3, 57.1, 36.7, 33.6, 25.6, 24.4, 21.5, 20.5, 18.5, 14.1, 13.7; IR (neat) ν (cm^{-1}) 2963, 2935, 2874, 1748, 1456, 1372, 1222, 1028; MS (EI): *m/z* (%) 326 (M^+, 23.9), 151 (100); HRMS Calcd for $C_{17}H_{26}O_6$ $(M)^+$: 326.1729; Found: 326.1730.

6.2 Preparation of 4-(2-acetoxy-4-hydroxybutan-2-yl)-3-propyl-2(*5H*)-furanone **8**[4] (fjj-2-090)

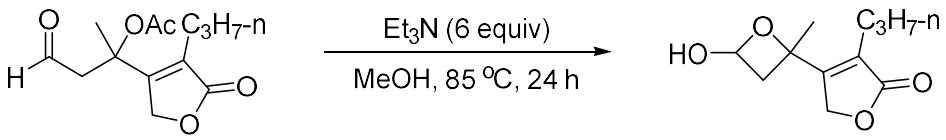


5ma, 0.2 mmol 4.5 equiv. **8**, 79%

To a dry Schlenk tube were added **5ma** (49.6 mg, 0.2 mmol) and ethyl acetate (2 mL). Sodium cyanoborohydride (57.4 mg, 4.5 mmol) was added in three portions (13.2

mg, 14.2 mg, and 30.0 mg) every hour. After being continuously stirred at room temperature for 4 h, the reaction was complete as monitored by thin layer chromatography (TLC). After filtration through a short column of silica gel eluted with ethyl acetate (20 mL × 3), the combined filtrate was concentrated in vacuo and the crude residual was purified by chromatography on silica gel (200~300 mesh) [eluent: petroleum ether/ethyl acetate = 2/1 (1500 mL)] to afford **8** (39.5 mg, 79%) as an oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.92 (s, 2 H, OCH_2), 3.73-3.48 (m, 2 H, OCH_2), 2.31 (t, *J* = 7.4 Hz, 2 H, CH_2), 2.23-1.93 (m, 6 H, CH_3, CH_2, and OH), 1.64-1.42 (m, 5 H, CH_2 and CH_3), 0.93 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.3, 170.2, 158.6, 130.9, 86.3, 57.6, 57.5, 39.8, 25.6, 24.7, 21.4, 20.6, 13.7; IR (neat) *v* (cm^{-1}) 3478, 2963, 2934, 2875, 1747, 1674, 1455, 1379, 1367, 1122, 1138, 1053, 1031; MS (EI): *m/z* (%) 256 (M^+, 4.51), 43 (100); HRMS Calcd for $C_{13}H_{20}O_5$ (M^+): 256.1311; Found: 256.1312.

6.3 Preparation of 4-(4-hydroxy-2-methyloxetan-2-yl)-3-propyl-2(*5H*)-furanone **9**. (fjj-2-098, fjj-2-162)



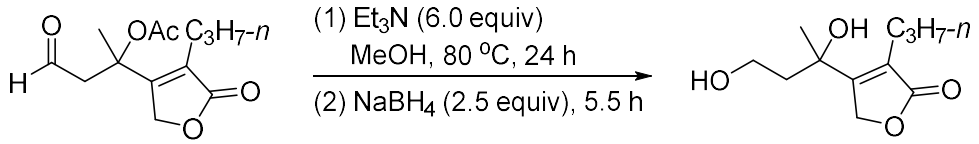
5ma, 0.2 mmol

9, 84%
dr = 2.6/1

To a dry Schlenk tube were added **5ma** (51.1 mg, 0.2 mmol), MeOH (4 mL), and Et_3N (166 μL, 1.2 mmol) sequentially. After being continuously stirred at 85 °C for 24

h, the reaction was complete as monitored by thin layer chromatography (TLC). After filtration through a short column of silica gel eluted with ethyl acetate (20 mL × 3), the combined filtrate was then concentrated in vacuo and the crude residual was purified by chromatography on silica gel (200~300 mesh) [eluent: petroleum ether/ethyl acetate = 5/1 (300 mL) to 3/1 (500 mL)] to afford **9** (35.7 mg, 84%, dr = 2.6:1) as an oil: ^{1}H NMR (600 MHz, $CDCl_3$) δ [5.43 (t, *J* = 4.2 Hz, 0.67 H), 5.12-5.05 (m, 0.27 H), OCHO], [4.73 (d, *J* = 13.8 Hz, 0.27 H), 4.64 (d, *J* = 12.6 Hz, 0.72 H), one proton of OCH_2], [4.44 (d, *J* = 12.0 Hz, 0.72 H), 4.28 (d, *J* = 14.4 Hz, 0.28 H), one proton of OCH_2], [3.00 (d, *J* = 6.0 Hz, 0.22 H), 2.74-2.65 (m, 0.59 H), OH], [2.54 (dd, J_1 = 15.0 Hz, J_2 = 3.0 Hz, 0.28 H), 2.46 (d, *J* = 13.2 Hz, 0.70 H), one proton of CH_2], 2.33-2.19 (m, 2 H, CH_2), [1.94-1.84 (m, 0.7 H, one proton of CH_2), 1.74-1.66 (m, 2.38 H, CH_3 and one proton of CH_2), 1.64-1.47 (m, 3.94 H, CH_3 and CH_2)], 0.91 (t, *J* = 7.5 Hz, 3 H, CH_3); IR (neat) *v* (cm^{-1}) 3419, 2962, 2929, 2873, 1751, 1732, 1457, 1337, 1212, 1188, 1062, 1004; MS (EI): *m/z* (%) 212 (M^+, 3.1), 43 (100); HRMS Calcd for $C_{11}H_{16}O_4$ (M^+): 212.1049; Found: 212.1050.

6.5 Preparation of 4-(2,4-dihydroxybutan-2-yl)-3-propyl-2(*5H*)-furanone **10**. (fjj-2-180)

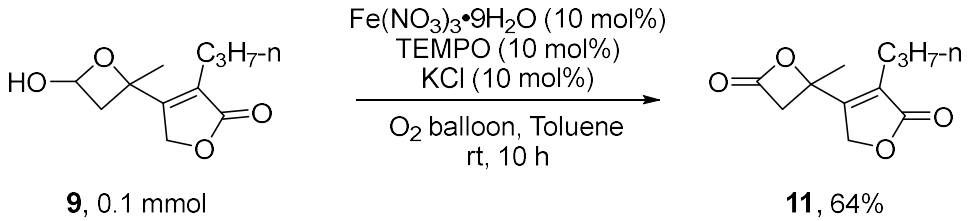


5ma, 0.2 mmol **10**, 56%

To a dry Schlenk tube were added **5ma** (51.0 mg, 0.2 mmol), MeOH (4 mL), and

Et_3N (166 μL, 1.2 mmol) sequentially. After being continuously stirred at 80 °C for 24 h, the resulting mixture was cooled to room temperature and treated with $NaBH_4$ (19.0 mg, 0.5 mmol). After being continuously stirred at room temperature for 5.5 h, the reaction was complete as monitored by thin layer chromatography (TLC). After filtration through a short column of silica gel eluted with ethyl acetate (10 mL × 3), the combined filtrate was then concentrated in vacuo and the crude residual was purified by chromatography on silica gel (200~300 mesh) [eluent: dichloromethane/ methanol = 50/1 (800 mL)] to afford **10** (24.2 mg, 56%) as an oil: ^{1}H NMR (300 MHz, $CDCl_3$) 4.62 (d, *J* = 14.1 Hz, 1 H, one proton of OCH_2), 4.29 (d, *J* = 14.1 Hz, 2 H, one proton of OCH_2 and OH), 3.76-3.58 (m, 1 H, one proton of CH_2), 3.47 (t, *J* = 10.5 Hz, 1 H, one proton of CH_2), 3.18 (bs, 1 H, OH), 2.39-2.08 (m, 4 H, CH_2 × 2), 1.70-1.36 (m, 5 H, CH_2 and CH_3), 0.91 (t, *J* = 7.5 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.4, 164.6, 128.2, 86.3, 56.8, 56.5, 39.7, 25.4, 25.0, 21.5, 13.7; IR (neat) *v* (cm^{-1}) 3428, 2962, 2929, 2874, 1729, 1668, 1456, 1379, 1315, 1181, 1136, 1051; MS (EI): *m/z* (%) 214 (M^+, 5.1), 43 (100); HRMS Calcd for $C_{11}H_{18}O_4$ (M^+): 214.1205; Found: 214.1207.

6.4 Preparation of 4-(β-methyl-β-lactonyl)-3-propyl-2(*5H*)-furanone **11**[5] (fjj-4-014)



9, 0.1 mmol **11**, 64%

A dry Schlenk tube containing $Fe(NO_3)_3·9H_2O$ (3.9 mg, 0.01 mmol), TEMPO (1.5

mg, 0.01 mmol), and KCl (0.9 mg, 0.01 mmol) was degassed under vacuum and backfilled with oxygen for three times. Then **9** (22.1 mg, 0.1 mmol) and toluene (0.5 mL) were added under oxygen atmosphere sequentially. After being continuously stirred at room temperature for 10 h, the reaction was complete as monitored by thin layer chromatography (TLC). After filtration through a short column of silica gel eluted with ethyl acetate (20 mL × 3), the combined filtrate was concentrated in vacuo and the crude residual was purified by chromatography on silica gel (200~300 mesh) [eluent: petroleum ether/ethyl acetate = 5/1 (600 mL)] to afford **11** (14.1 mg, 64%) as an oil: ^{1}H NMR (300 MHz, $CDCl_3$) 5.31 (d, J = 15.6 Hz, 1 H, one proton of OCH_2), 5.18 (d, J = 15.3 Hz, 1 H, one proton of OCH_2), 3.26 (d, J = 16.8 Hz, 1 H, one proton of CH_2), 2.65 (d, J = 17.1 Hz, 1 H, one proton of CH_2), 2.45-2.15 (m, 2 H, CH_2), 1.73-1.47 (m, 5 H, CH_2 and CH_3), 0.93 (t, J = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.0, 165.7, 153.6, 127.9, 79.6, 63.5, 44.2, 25.7, 23.4, 21.0, 13.6; IR (neat) ν (cm^{-1}) 2963, 2934, 2874, 1747, 1457, 1384, 1310, 1238, 1182, 1115, 1061, 1035; MS (EI): *m/z* (%) 210 (M^+, 15.0), 167 (100); HRMS Calcd for $C_{11}H_{14}O_4$ (M^+): 210.0892; Found: 210.0893.

7. **References**

1. Luo, H.; Ma, S. *Eur. J. Org. Chem.* **2013**, 3041.

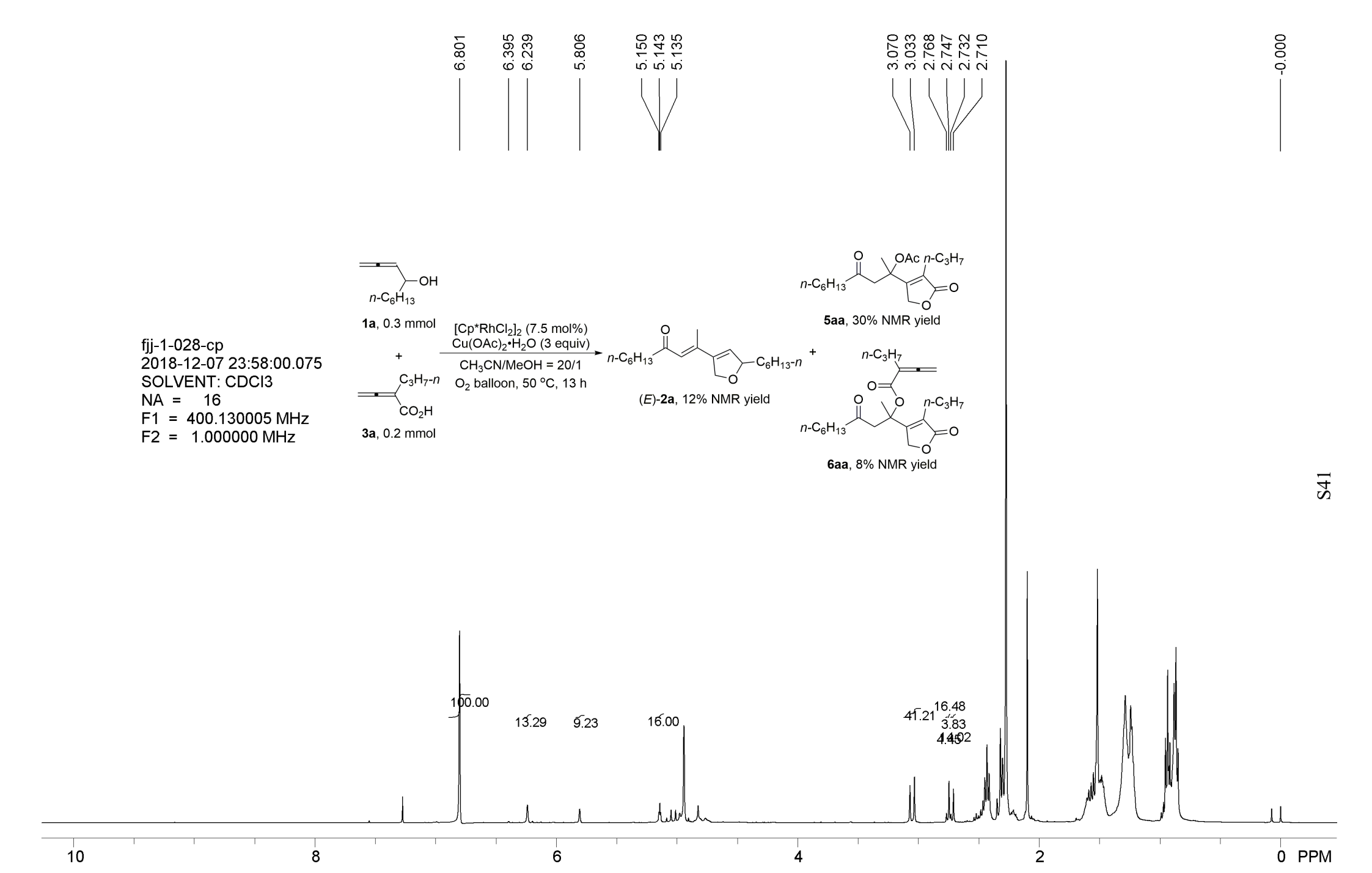
2. (a) Miao, B.; Li, G.; Ma, S. *Chem. Eur.J.* **2015**, *21*, 17224; (b) Castellano, S.; Fiji, H. D. G.; Kinderman, S. S.; Watanabe, M.; de Leon, P.; Tamanoi, F.; Kwon, O. *J. Am. Chem. Soc.* **2007**, *129*, 5843.

3. Fan, J.; Fu, C.; Wu, X.; Ma, S. *Chem. Commun.* **2021**, *57*, 10411.

4. Zeynizadeh, B.; Kouhkan, M. *J. Chin. Chem. Soc.* **2018**, *65*, 1521.

5. Liu, J.; Xie, X.; Ma, S. *Synthesis* **2012**, *44*, 1569.

8. ^{1}H NMR, ^{13}C NMR, and ^{19}F NMR Spectra of the Compounds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3.033
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2.747
2.732
2.710
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F2 = 1.000000 MHz
1a, 0.3 mmol
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[Cp*RhCl2]2 (7.5 mol%)
Cu(OAc)2•H2O (3 equiv)
CH3CN/MeOH = 20/1
O2 balloon, 50 °C, 13 h
(E)-2a, 12% NMR yield
5aa, 30% NMR yield
6aa, 8% NMR yield
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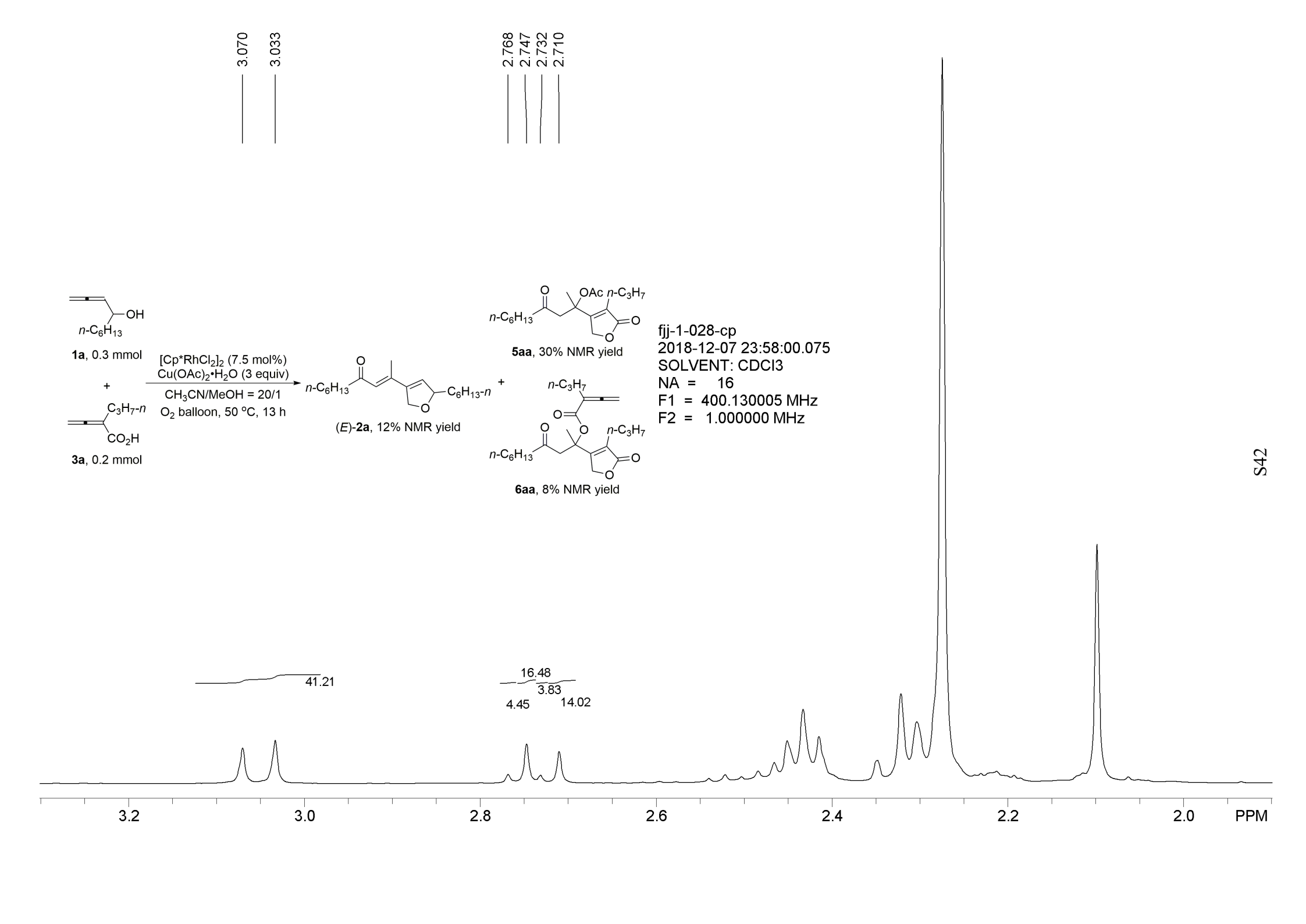
16.00
41.21
16.48
3.83
4.02
10
8
6
4
2
0
PPM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2.747
2.732
2.710
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CO2H
3a, 0.2 mmol
[Cp*RhCl2]2 (7.5 mol%)
Cu(OAc)2•H2O (3 equiv)
CH3CN/MeOH = 20/1
O2 balloon, 50 °C, 13 h
n-C6H13
C6H13-n
(E)-2a, 12% NMR yield
+
OAc n-C3H7
n-C6H13
5aa, 30% NMR yield
n-C3H7
n-C6H13
n-C3H7
6aa, 8% NMR yield
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2018-12-07 23:58:00.075
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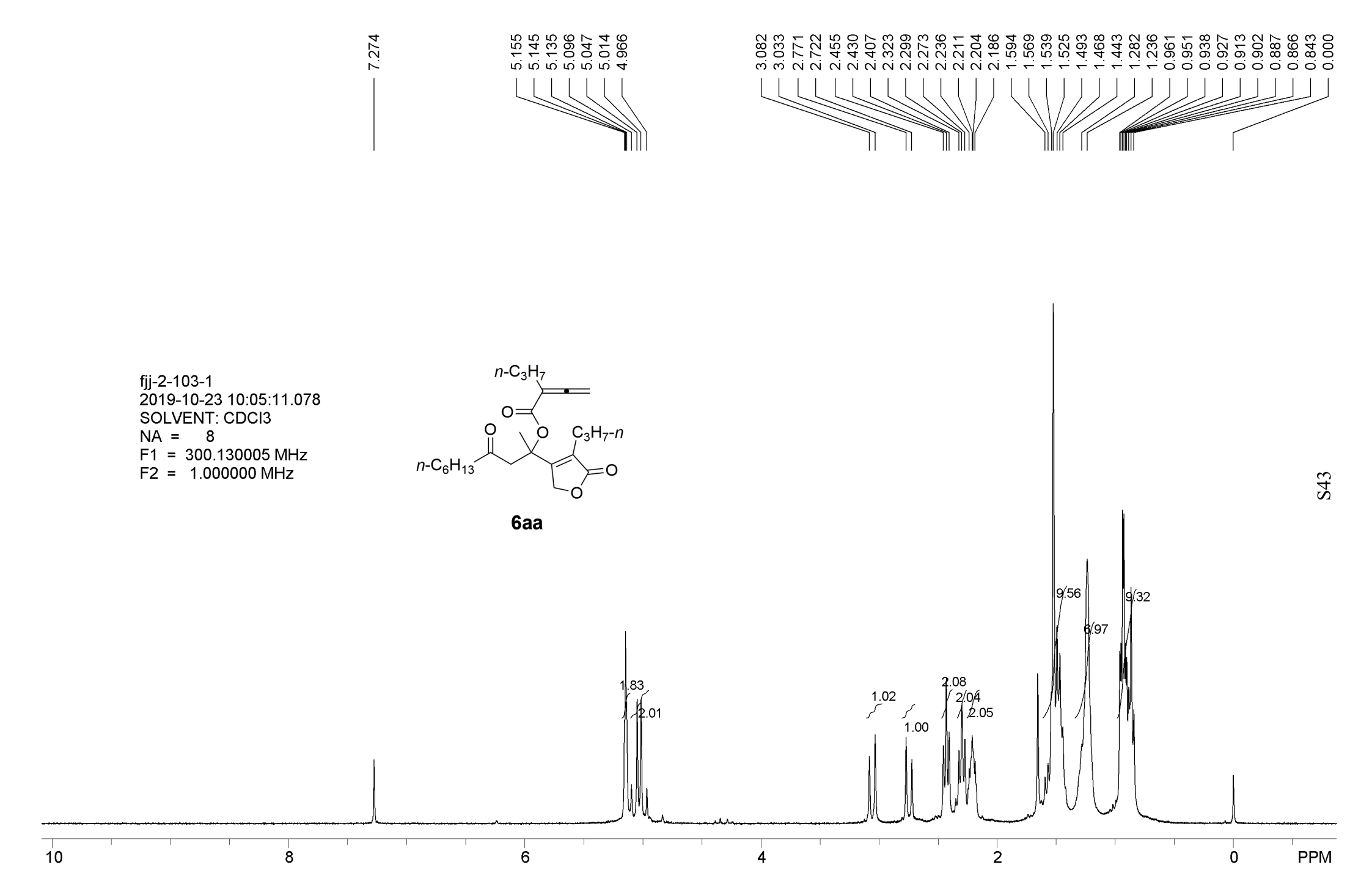
2.0
PPM
S42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2.455
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F2 = 1.000000 MHz
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10
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2
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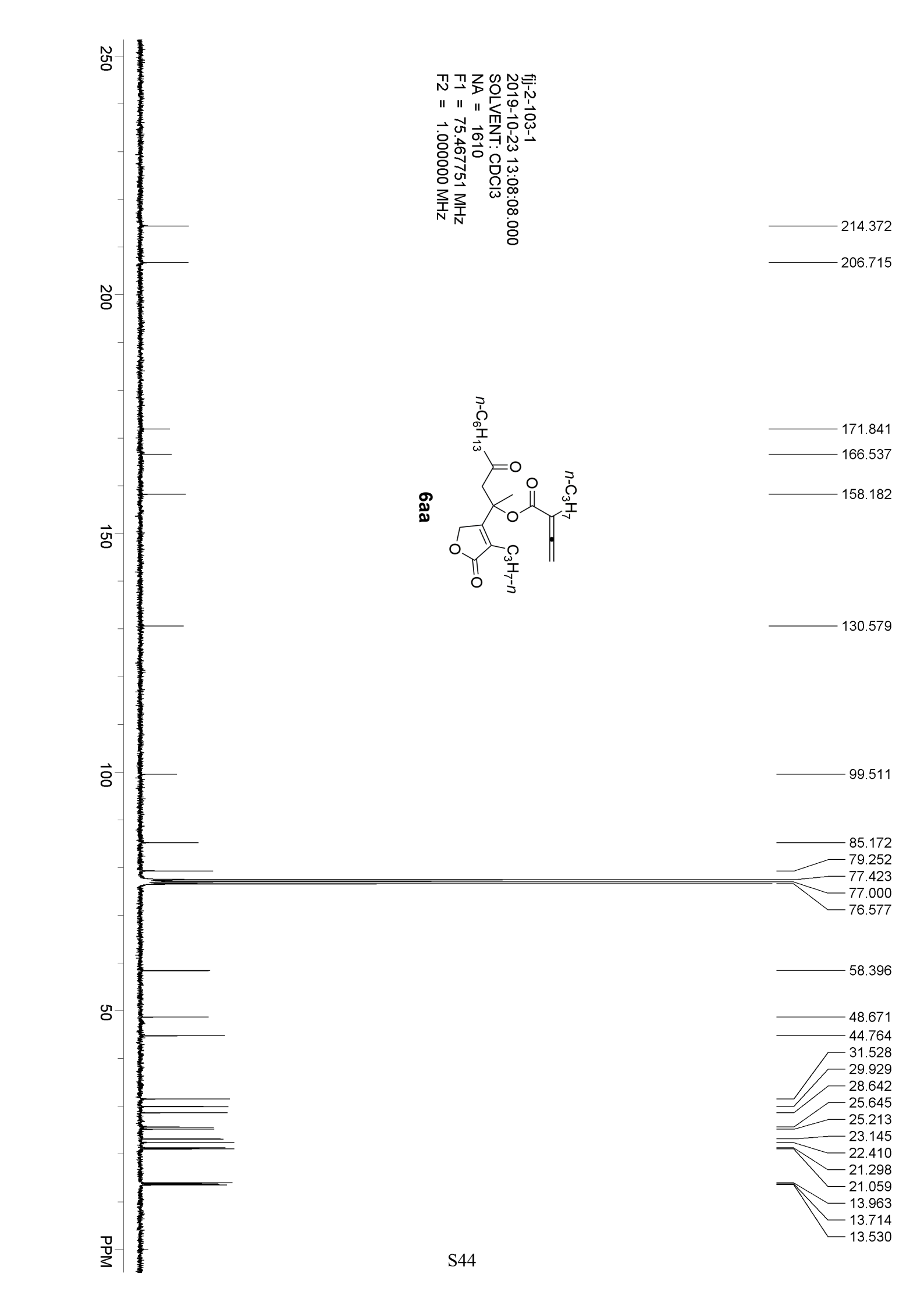
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28.642
25.645
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100
50
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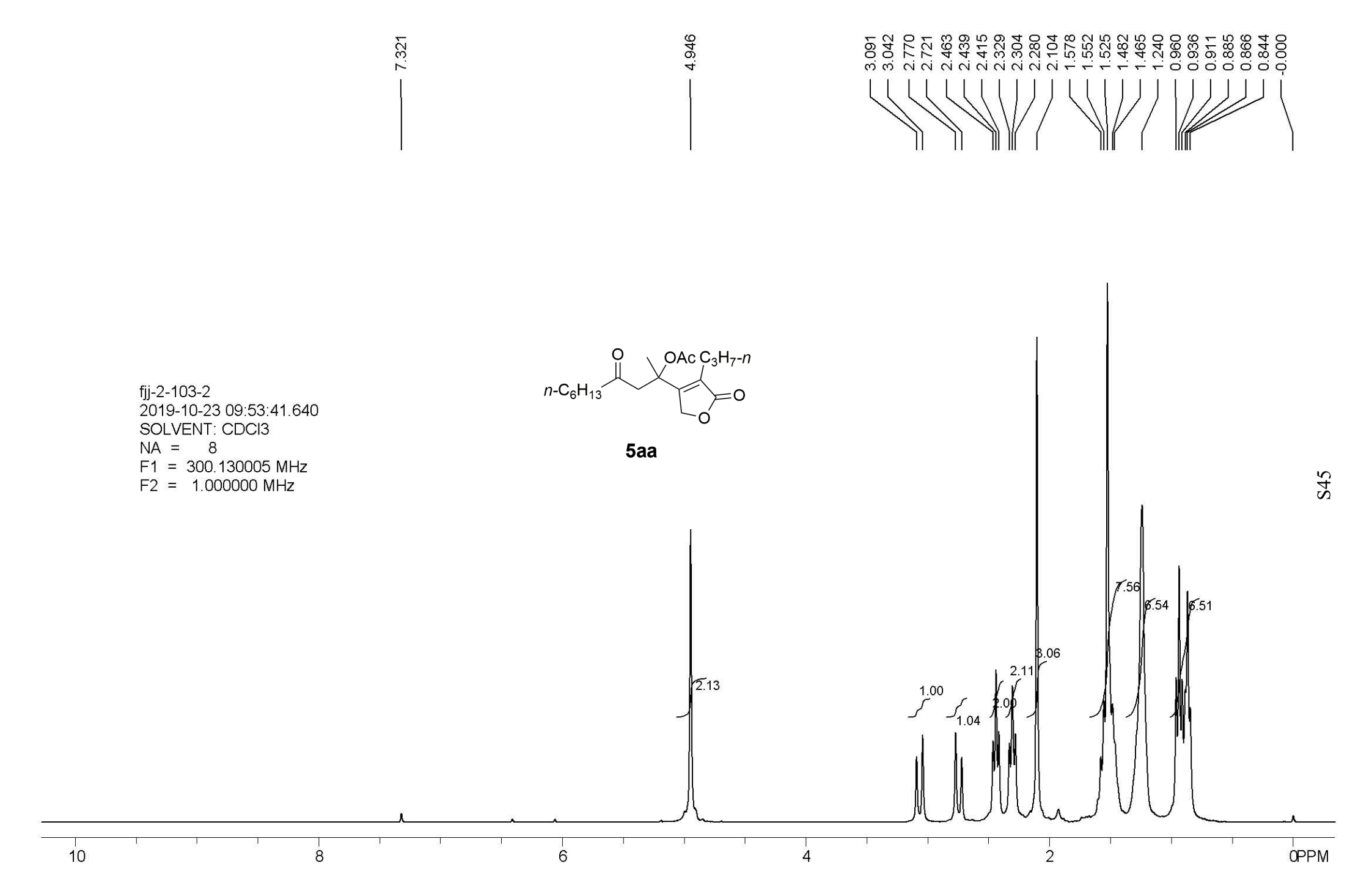
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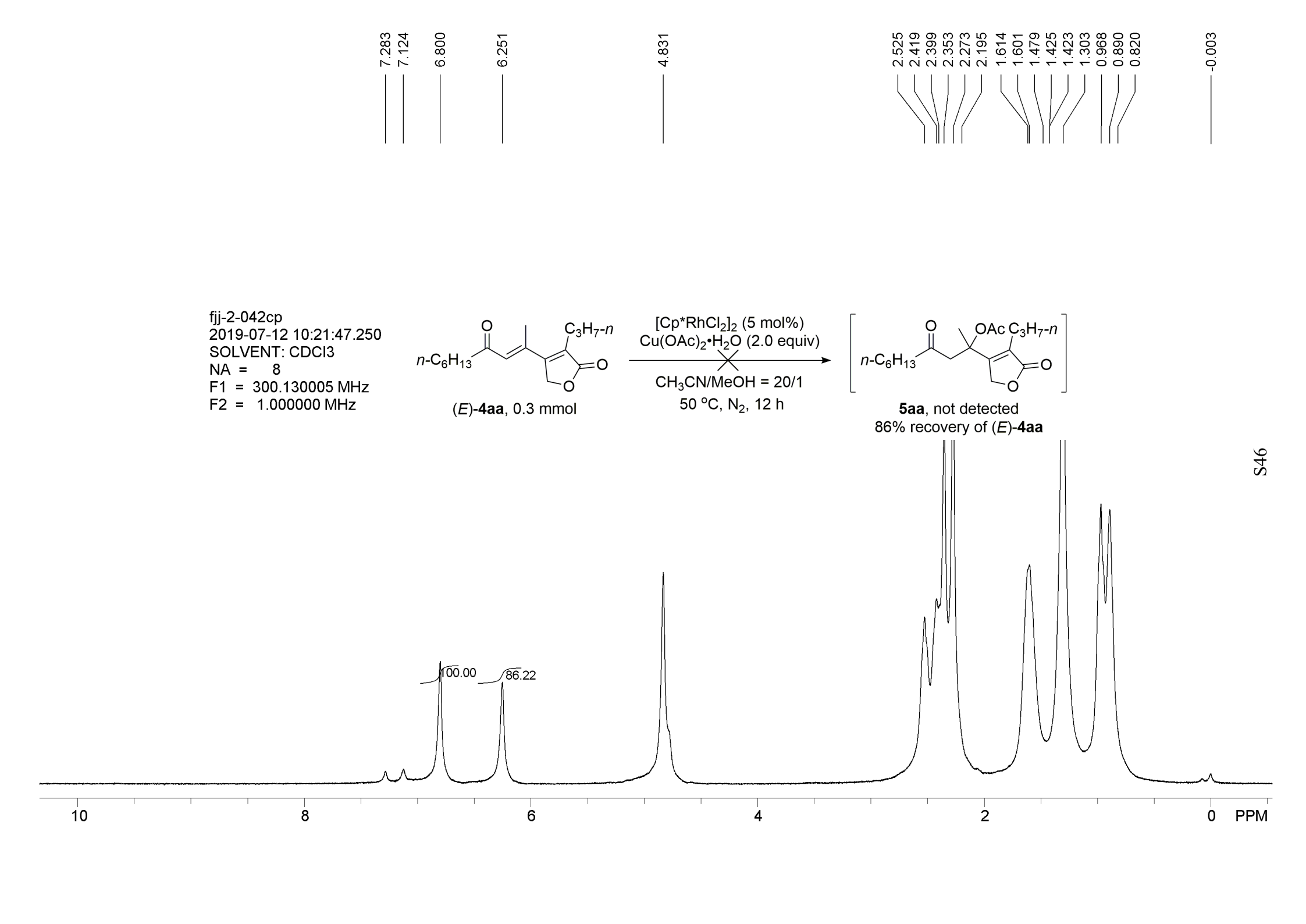
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4
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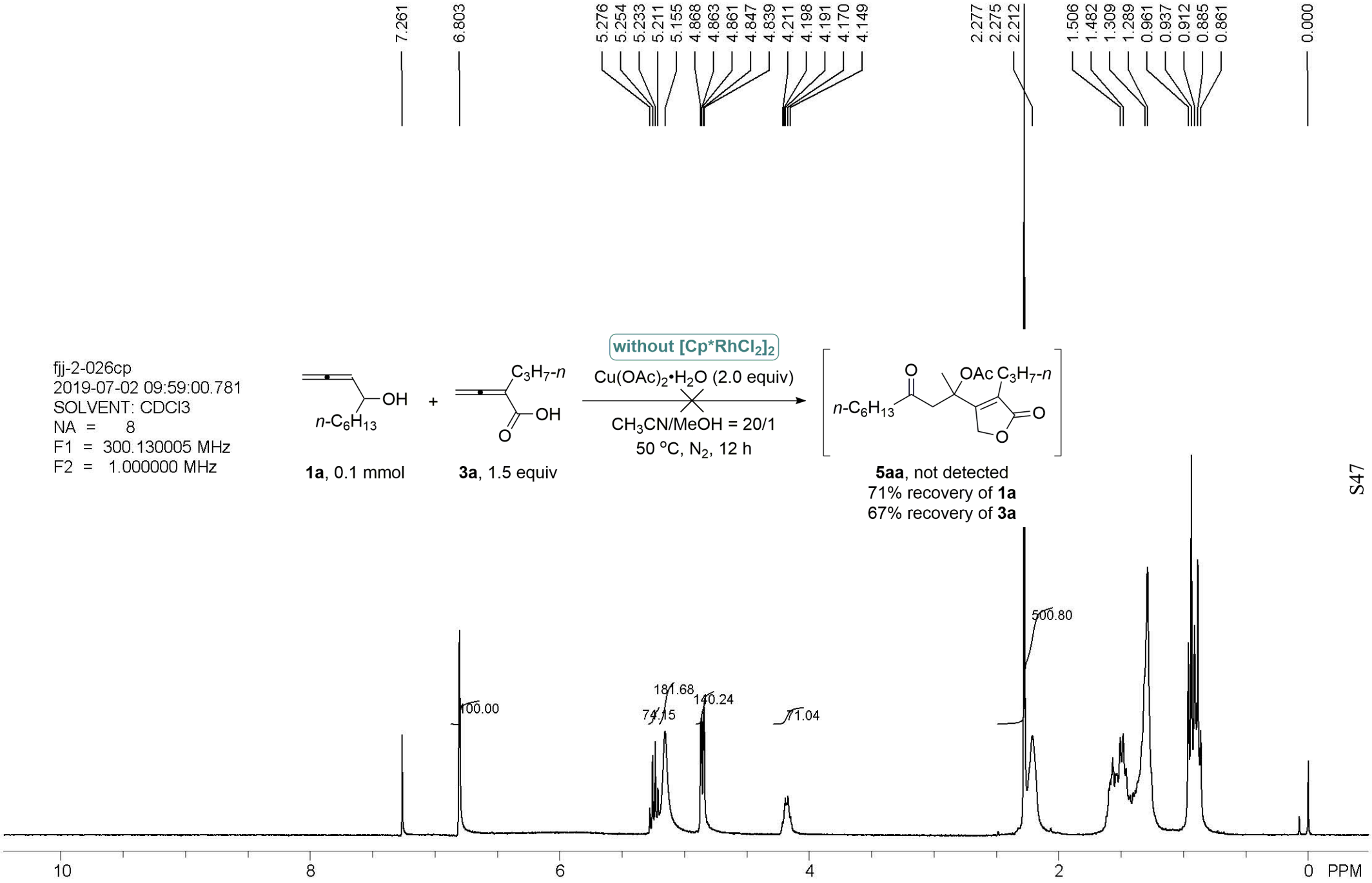
PPM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Cu(OAc)2•H2O (2.0 equiv)
CH3CN/MeOH = 20/1
50 °C, N2, 12 h
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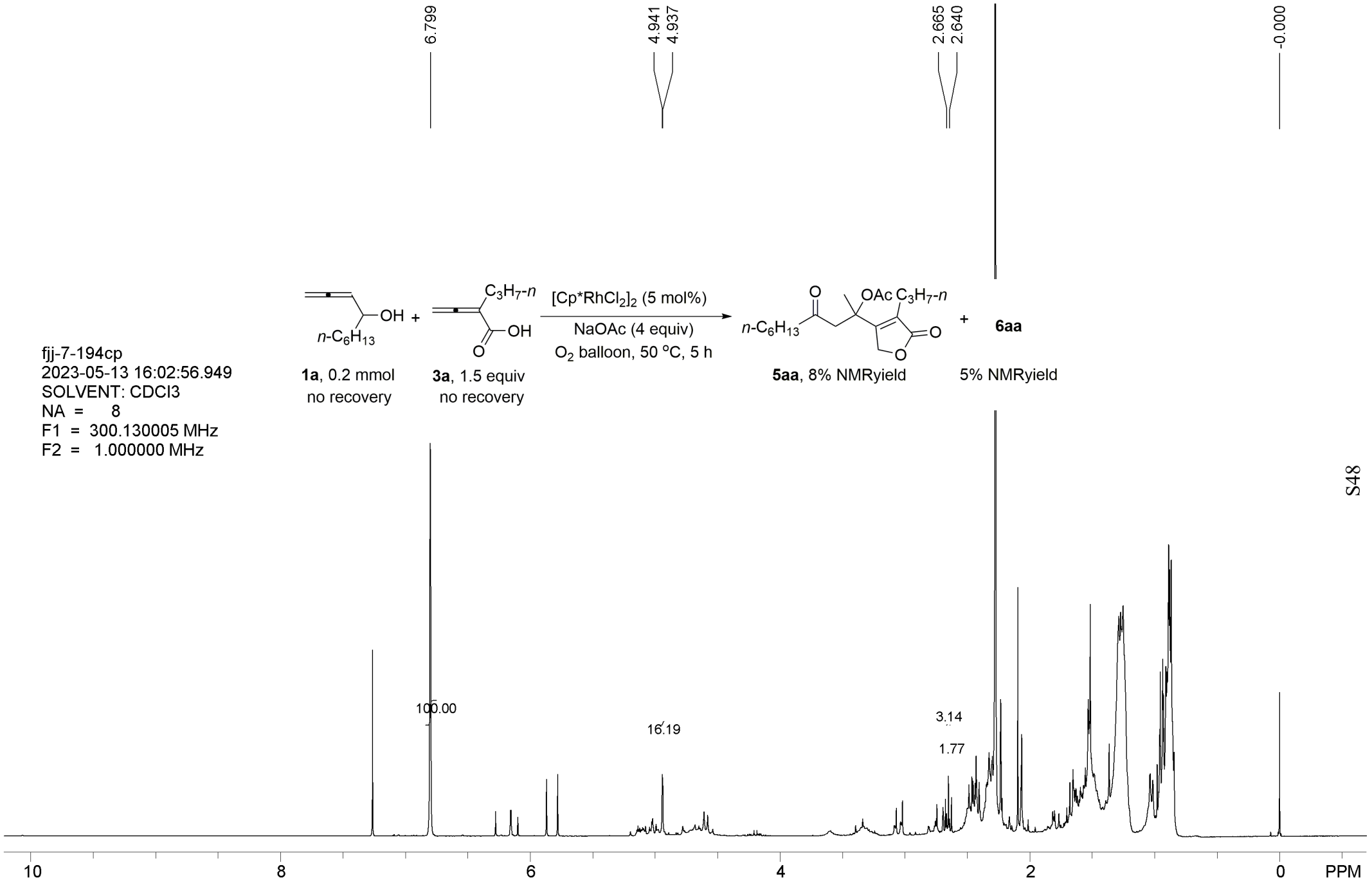
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NaOAc (4 equiv)
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n-C6H13
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O
+
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fjj-7-194cp
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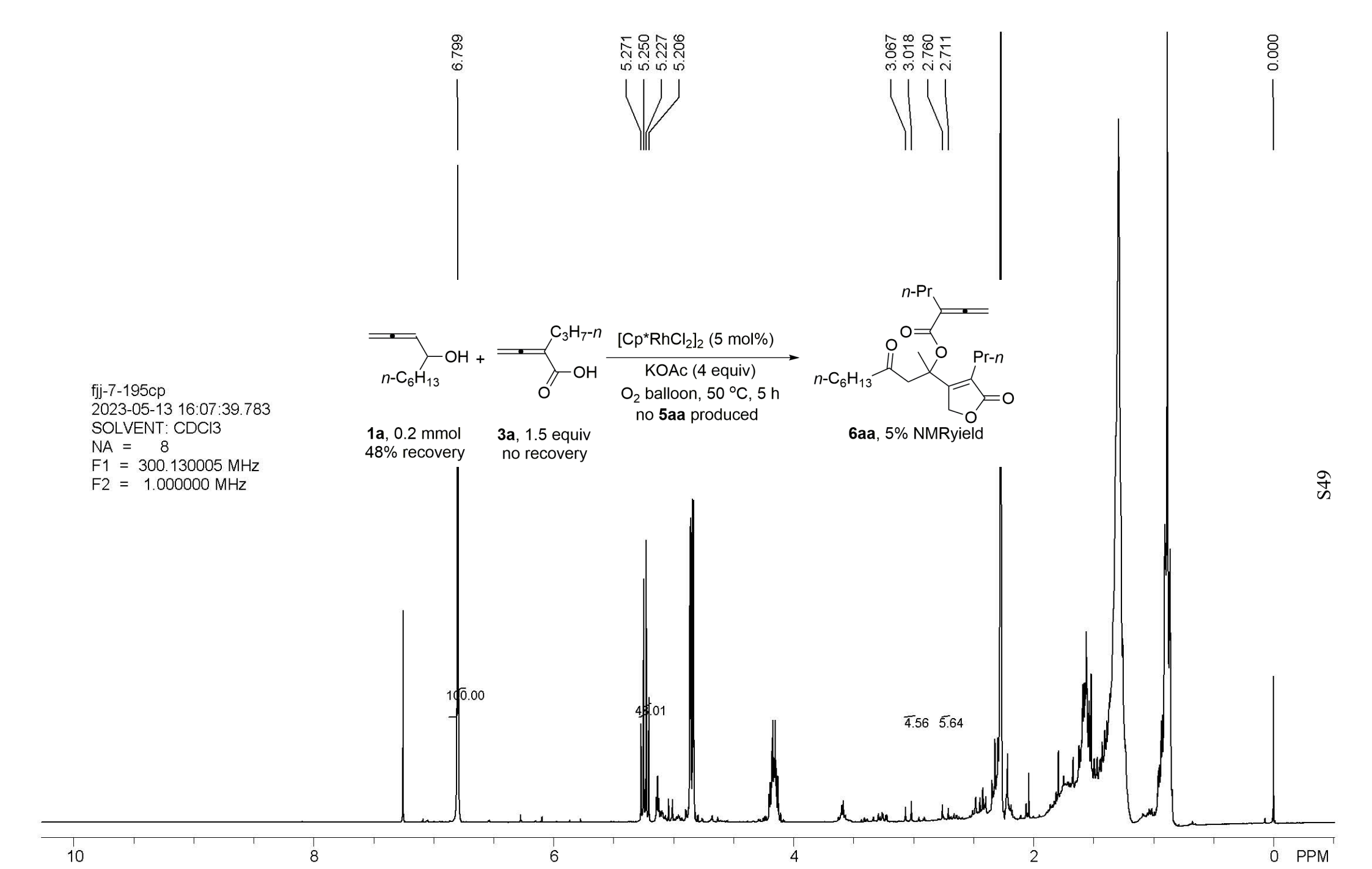
PPM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KOAc (4 equiv)
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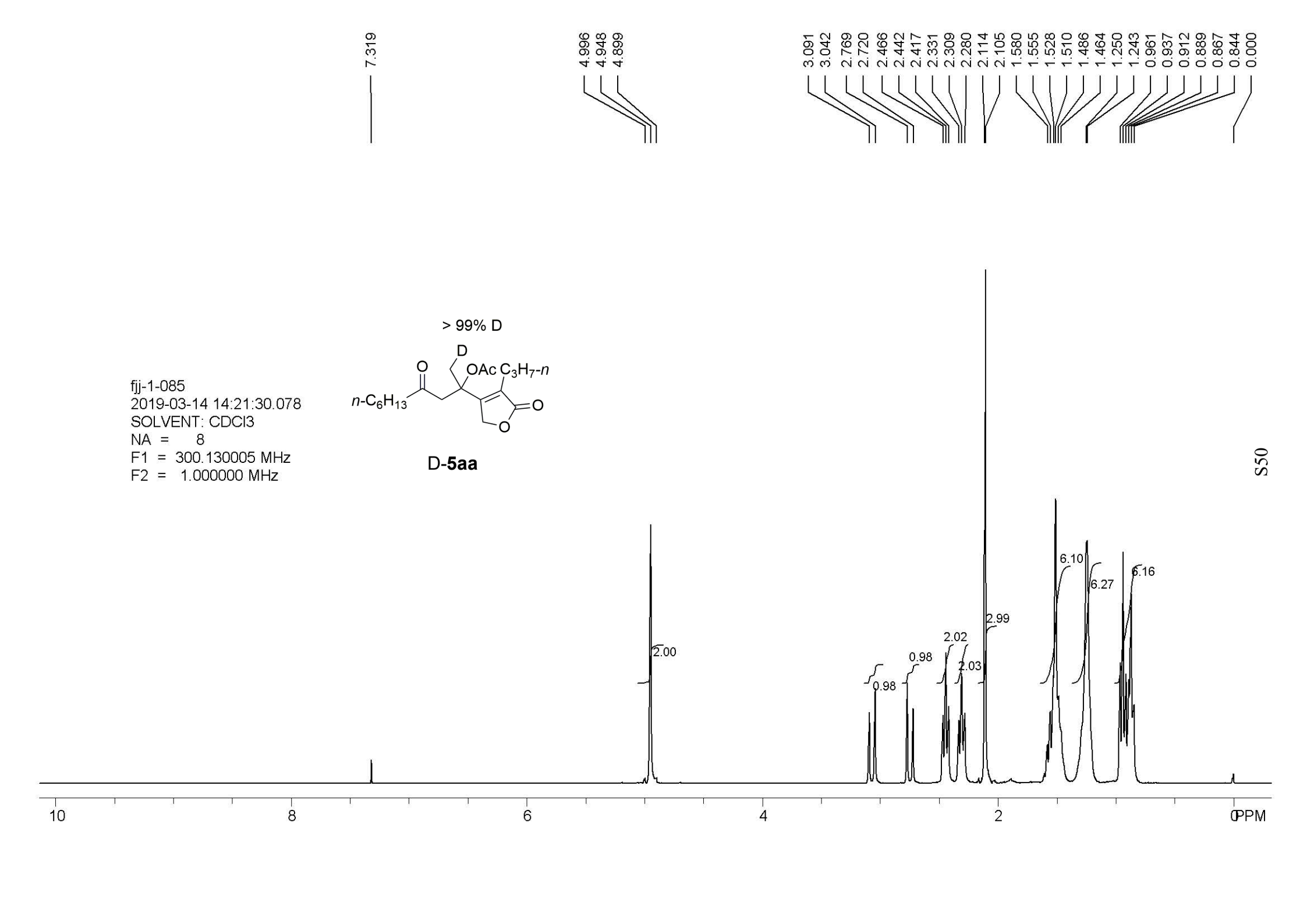
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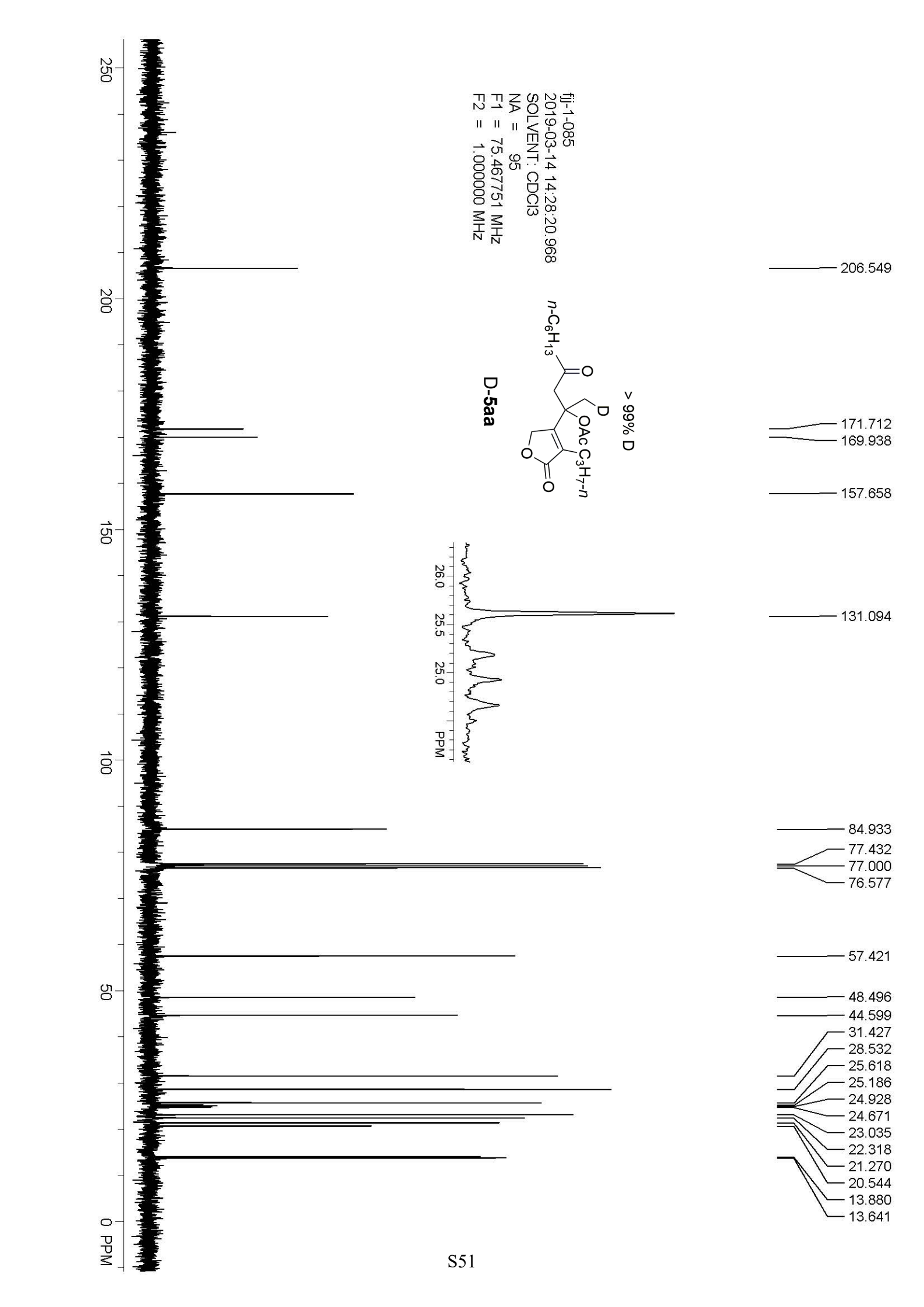
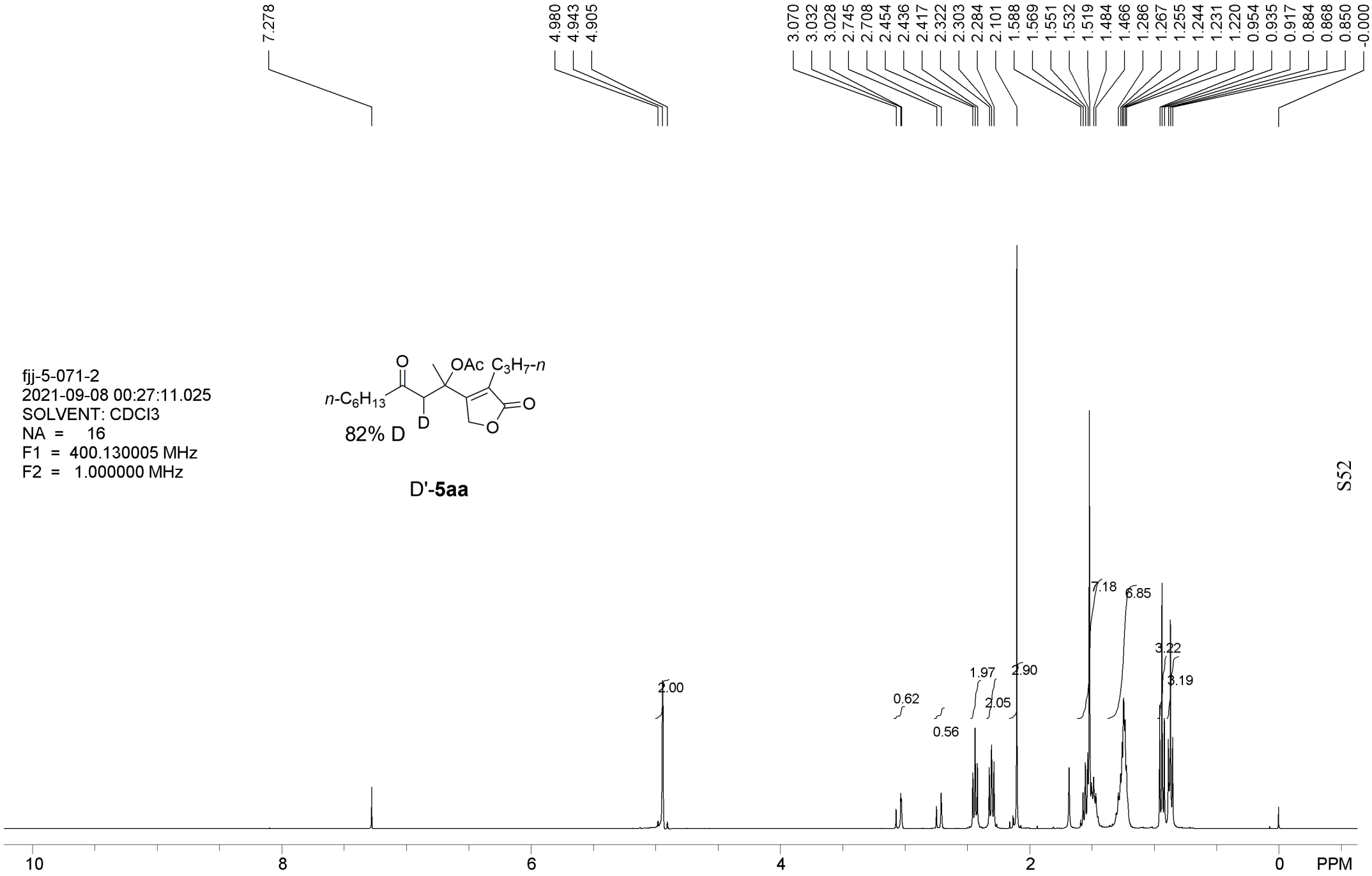
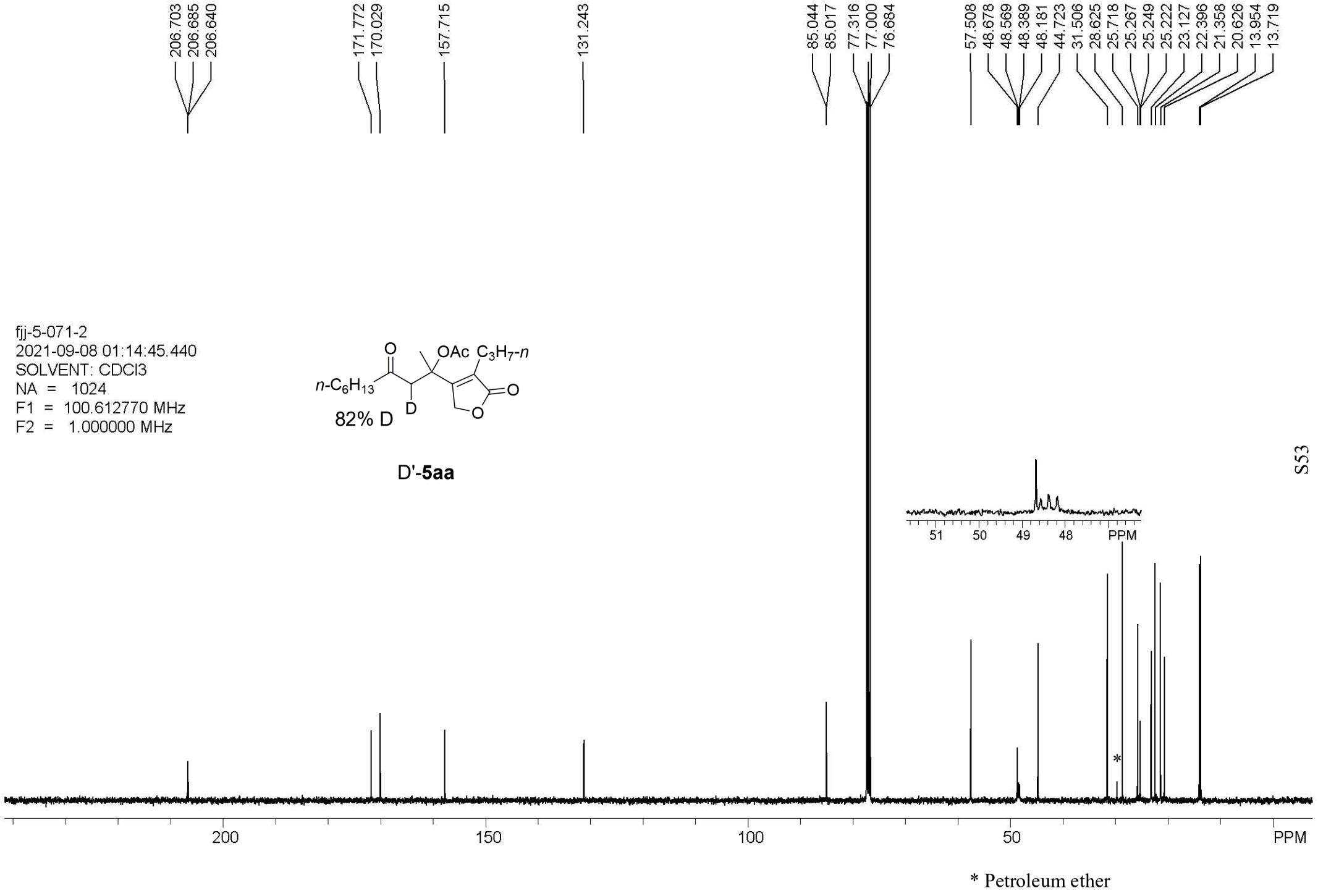
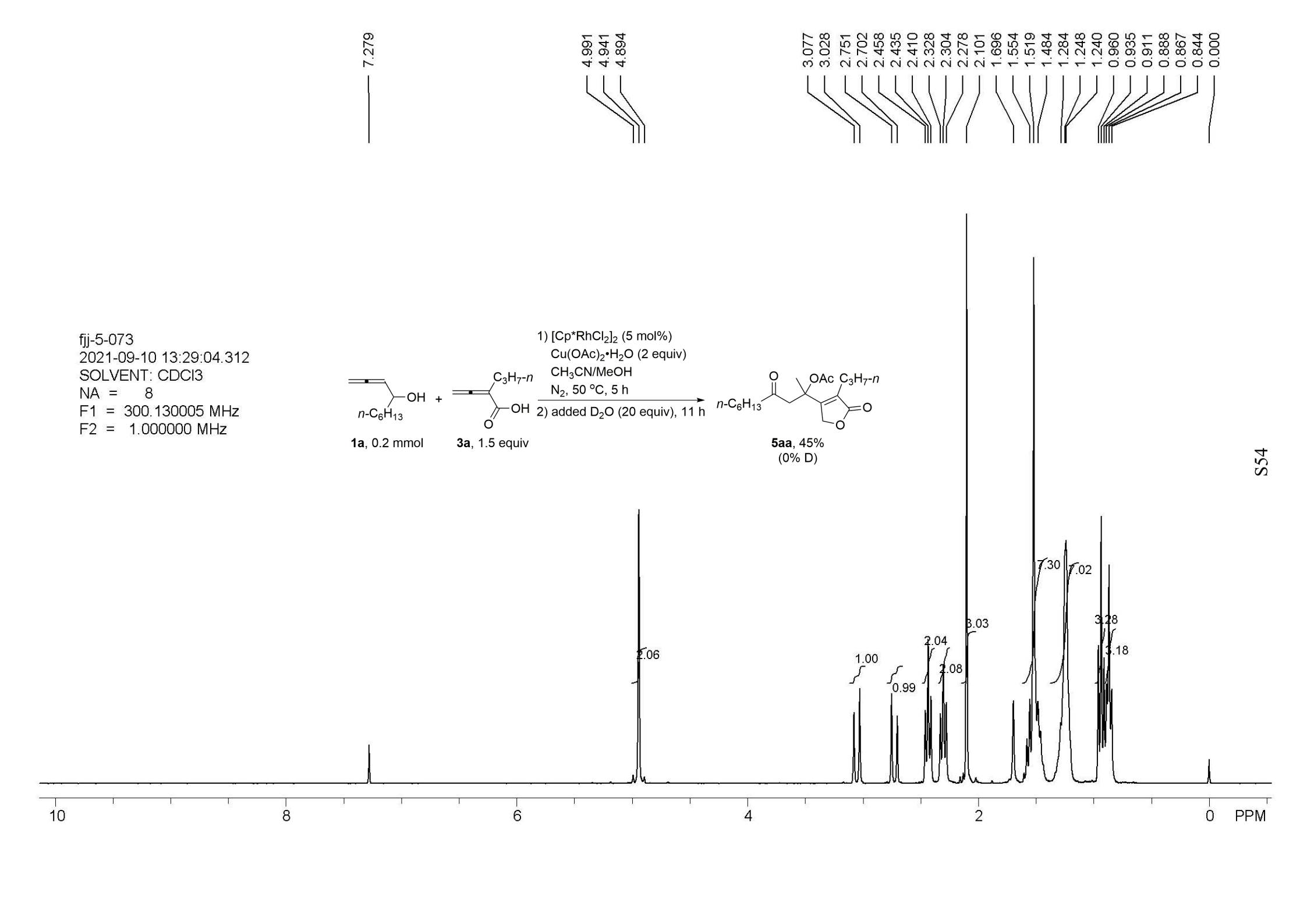
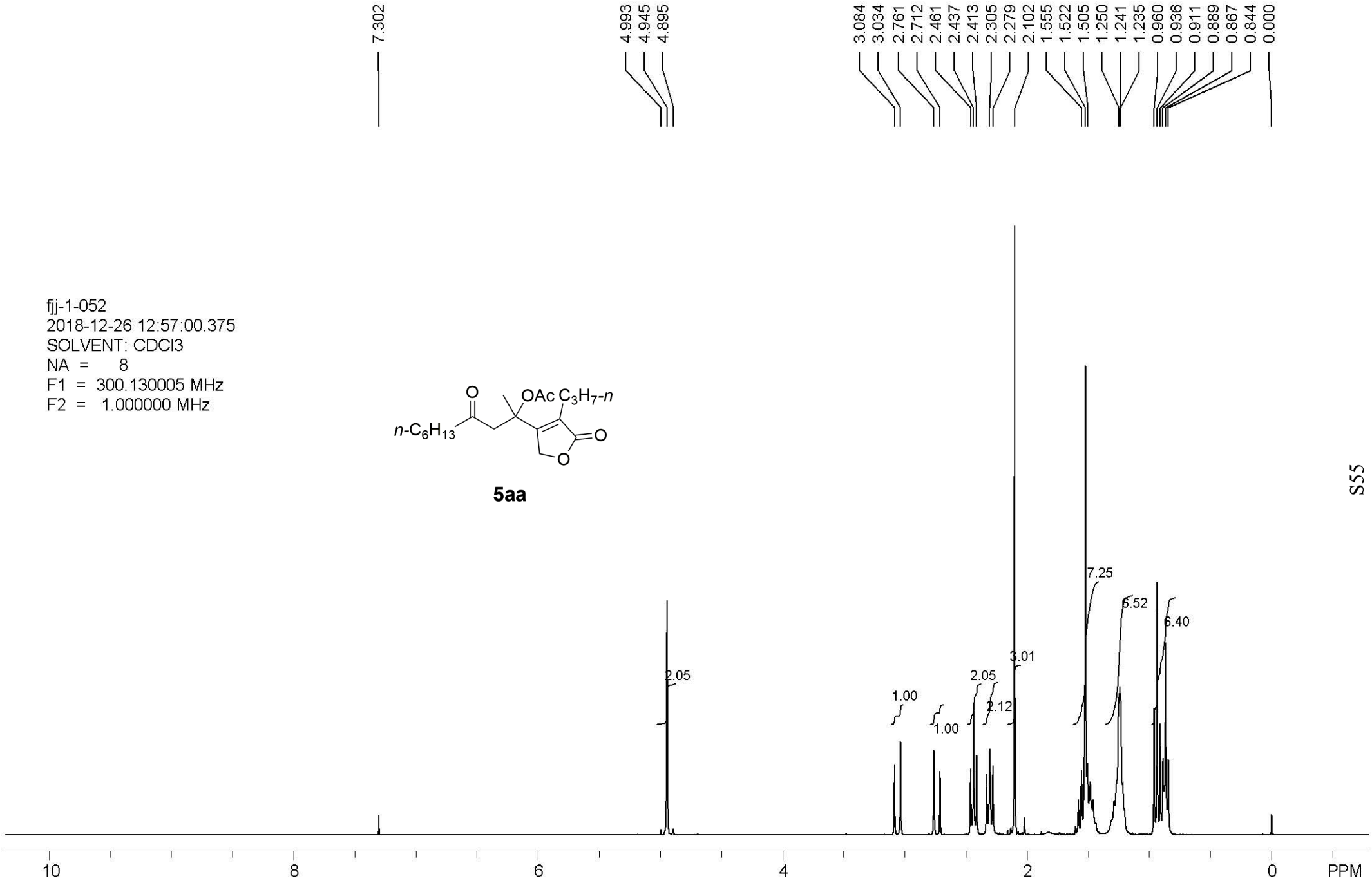
2.309
2.280
2.114
2.105
1.580
1.555
1.528
1.510
1.486
1.464
1.250
1.243
0.961
0.937
0.912
0.889
0.867
0.844
0.000
> 99% D
D
O
OAc
C_3H_7-*n*
n-C_6H_{13}
O
O
D-**5aa**
fjj-1-085
2019-03-14 14:21:30.078
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
S50
2.00
0.98
0.98
2.02
2.03
2.99
6.10
6.27
6.16
10
8
6
4
2
0
PPM


fjj-1-085
2019-03-14 14:28:20.968
SOLVENT: CDCl3
NA = 95
F1 = 75.467751 MHz
F2 = 1.000000 MHz
D-5aa
> 99% D
206.549
171.712
169.938
157.658
131.094
84.933
77.432
77.000
76.577
57.421
48.496
44.599
31.427
28.532
25.618
25.186
24.928
24.671
23.035
22.318
21.270
20.544
13.880
13.641
PPM

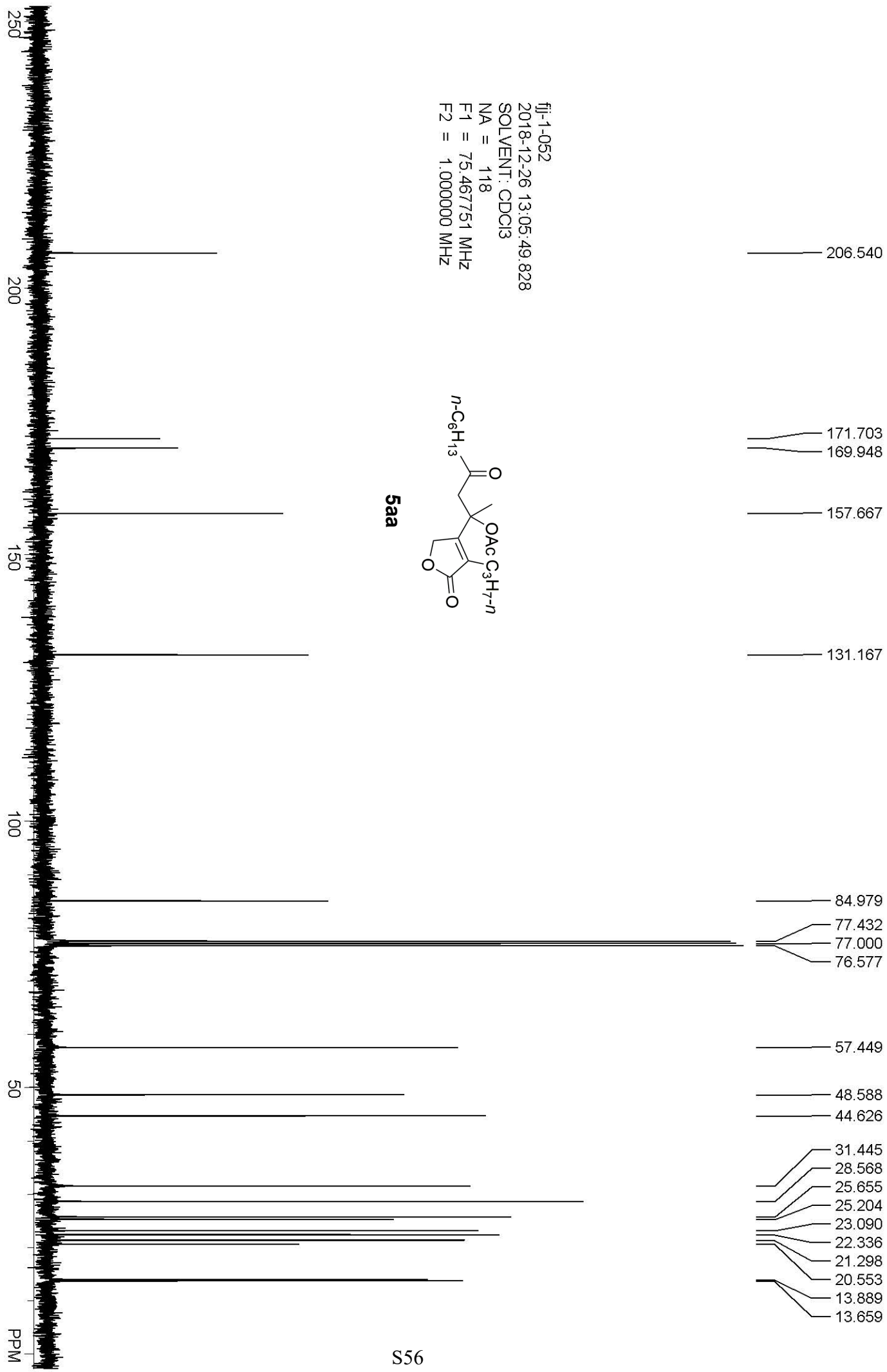

7.278
4.980
4.943
4.905
3.070
3.032
3.028
2.745
2.708
2.454
2.436
2.417
2.322
2.303
2.284
2.101
1.588
1.569
1.551
1.532
1.519
1.484
1.466
1.286
1.267
1.255
1.244
1.231
1.220
0.954
0.935
0.917
0.884
0.868
0.850
-0.000
fjj-5-071-2
2021-09-08 00:27:11.025
SOLVENT: CDCl3
NA = 16
F1 = 400.130005 MHz
F2 = 1.000000 MHz
O
OAc
C3H7-n
n-C6H13
D
O
O
82% D
D'-5aa
S52
2.00
0.62
0.56
1.97
2.05
2.90
7.18
6.85
3.22
3.19
10
8
6
4
2
0
PPM

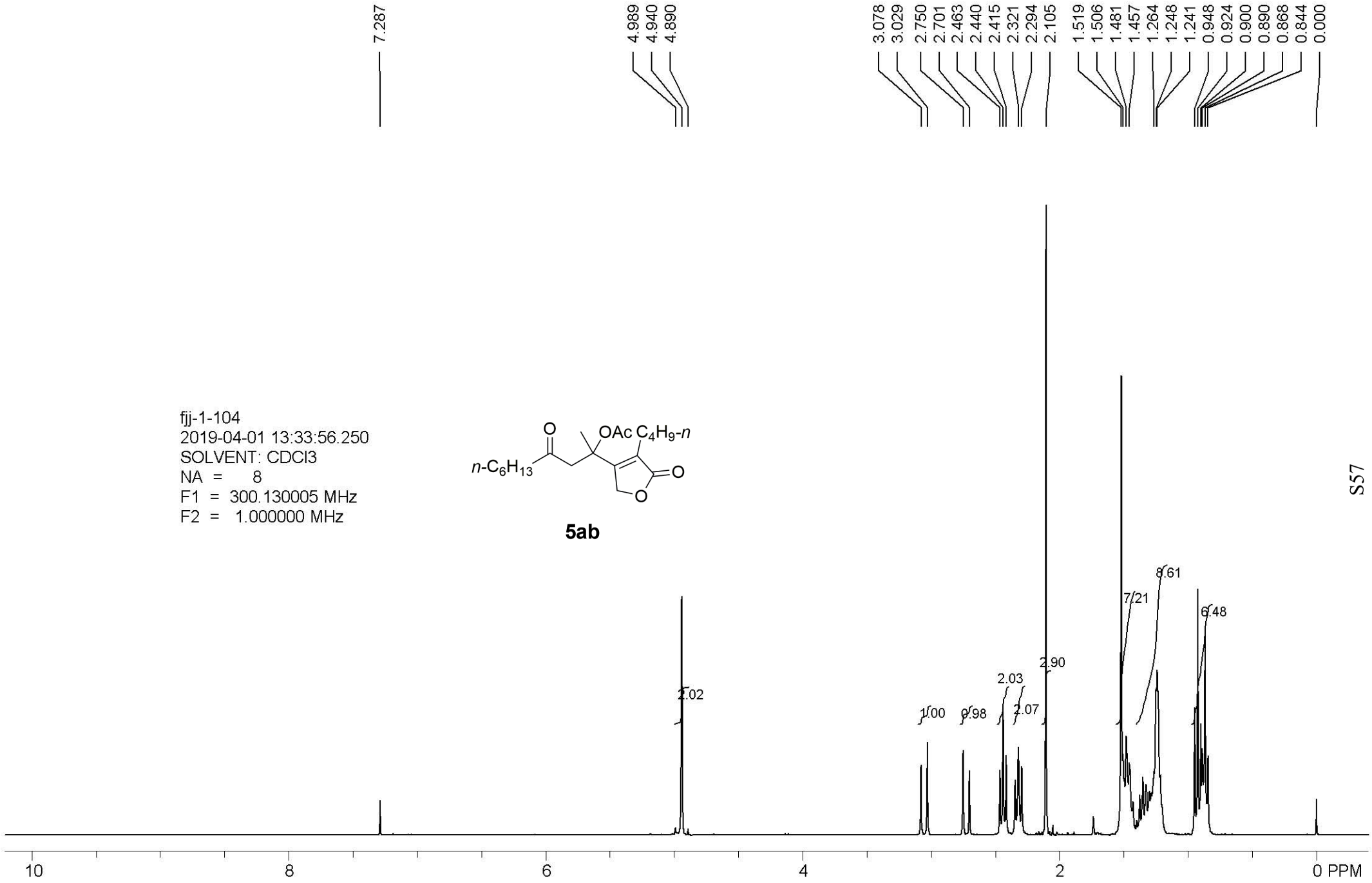

206.703
206.685
206.640
171.772
170.029
157.715
131.243
85.044
85.017
77.316
77.000
76.684
57.508
48.678
48.569
48.389
48.181
44.723
31.506
28.625
25.718
25.267
25.249
25.222
23.127
22.396
21.358
20.626
13.954
13.719
fjj-5-071-2
2021-09-08 01:14:45.440
SOLVENT: CDCl3
NA = 1024
F1 = 100.612770 MHz
F2 = 1.000000 MHz
O
OAc
C_3H_7-*n*
n-C_6H_{13}
D
O
O
82% D
D'-**5aa**
51
50
49
48
PPM
*
200
150
100
50
PPM
* Petroleum ether

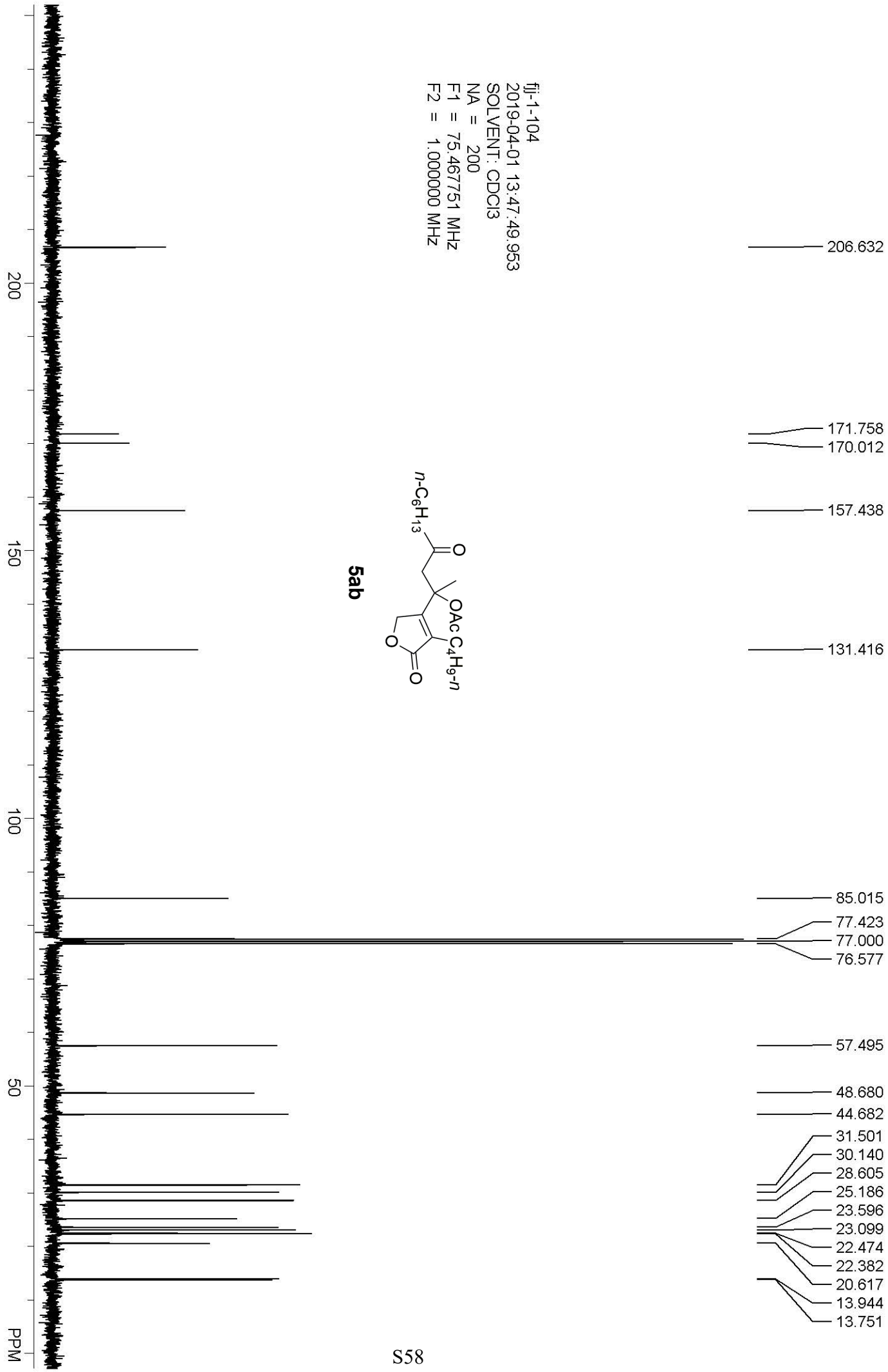

7.279
4.991
4.941
4.894
3.077
3.028
2.751
2.702
2.458
2.435
2.410
2.328
2.304
2.278
2.101
1.696
1.554
1.519
1.484
1.284
1.248
1.240
0.960
0.935
0.911
0.888
0.867
0.844
0.000
fjj-5-073
2021-09-10 13:29:04.312
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
1) [Cp*RhCl2]2 (5 mol%)
Cu(OAc)2•H2O (2 equiv)
CH3CN/MeOH
N2, 50 °C, 5 h
2) added D2O (20 equiv), 11 h
OH
n-C6H13
C3H7-n
OAc
1a, 0.2 mmol
3a, 1.5 equiv
5aa, 45%
(0% D)
S54
2.06
1.00
0.99
2.04
2.08
3.03
7.30
7.02
3.28
3.18
10
8
6
4
2
0
PPM

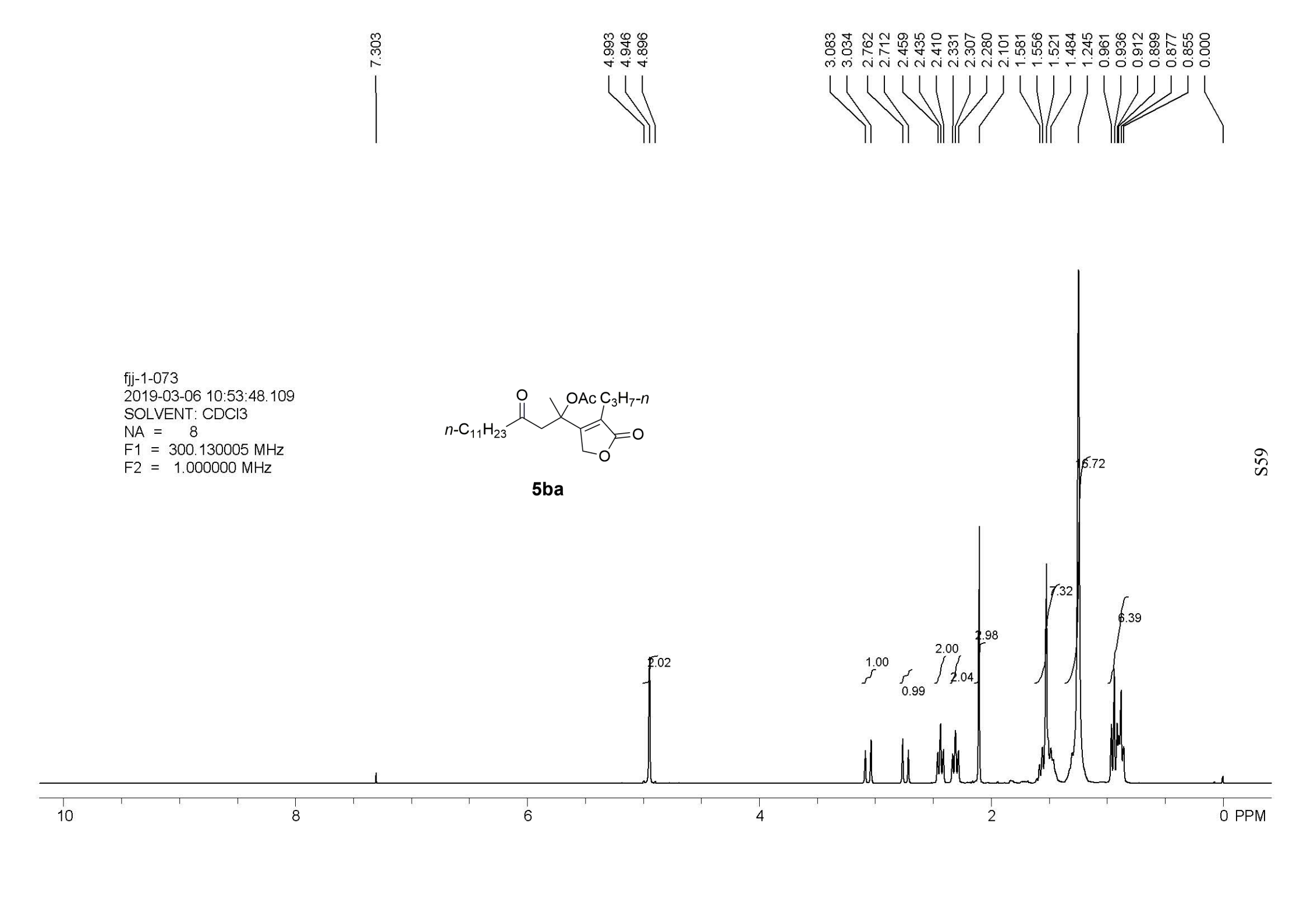

7.302
4.993
4.945
4.895
3.084
3.034
2.761
2.712
2.461
2.437
2.413
2.305
2.279
2.102
1.555
1.522
1.505
1.250
1.241
1.235
0.960
0.936
0.911
0.889
0.867
0.844
0.000
fjj-1-052
2018-12-26 12:57:00.375
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
O
OAc
C_3H_7-*n*
n-C_6H_{13}
O
O
2.05
1.00
1.00
2.05
2.12
3.01
7.25
6.52
6.40
10
8
6
4
2
0
PPM

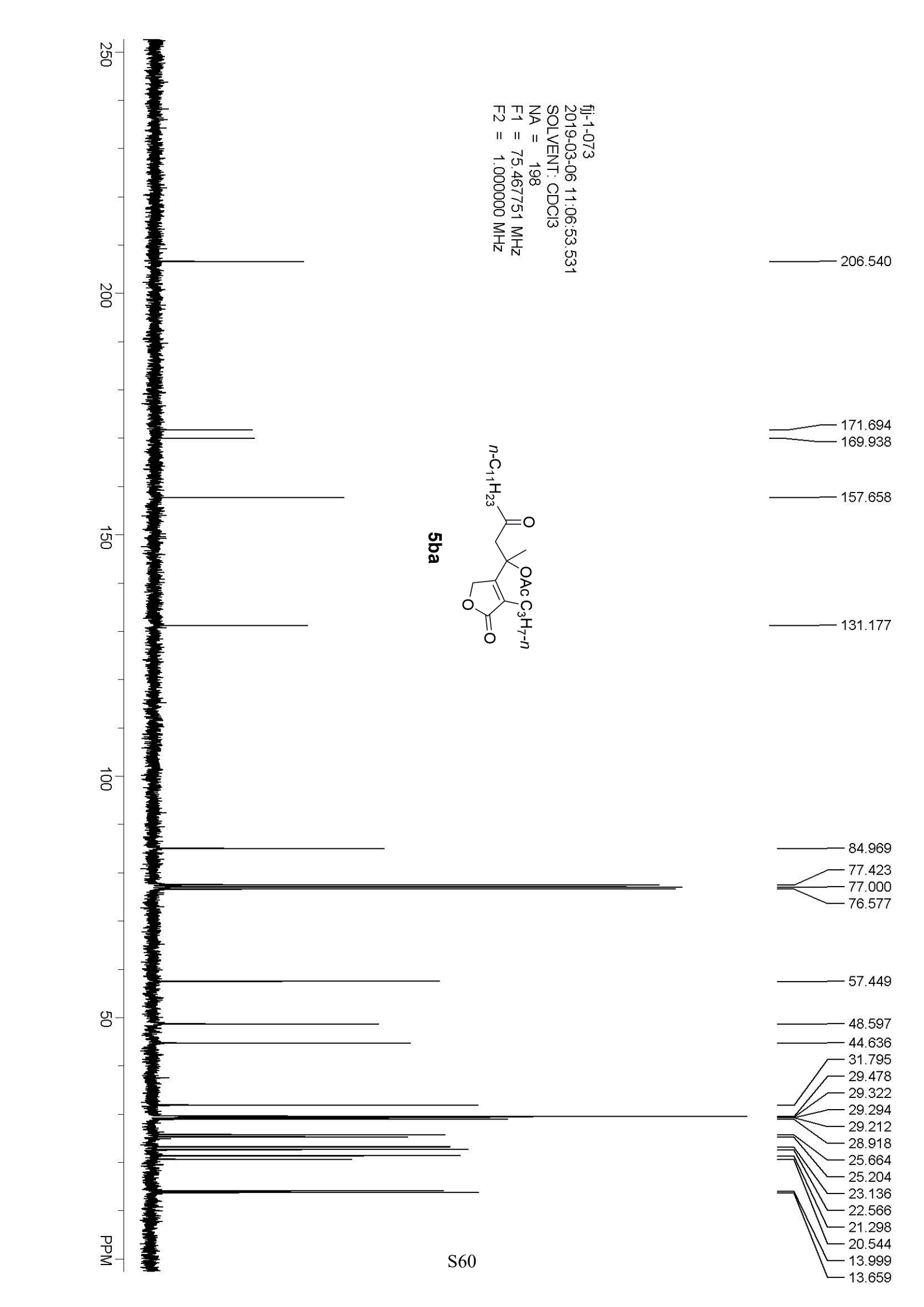
5aa

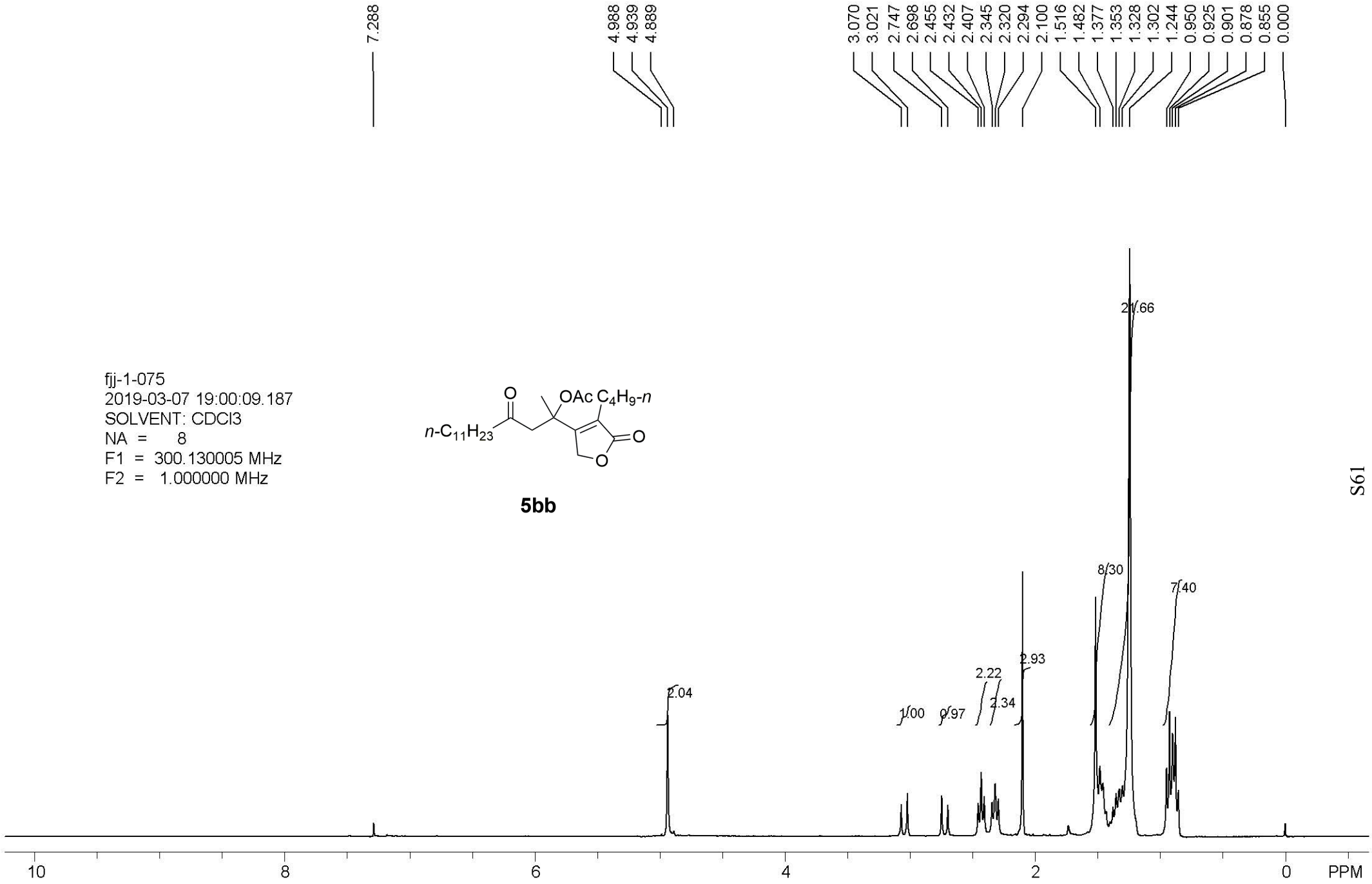

fjj-1-052
2018-12-26 13:05:49.828
SOLVENT: CDCl3
NA = 118
F1 = 75.467751 MHz
F2 = 1.000000 MHz
5aa
206.540
171.703
169.948
157.667
131.167
84.979
77.432
77.000
76.577
57.449
48.588
44.626
31.445
28.568
25.655
25.204
23.090
22.336
21.298
20.553
13.889
13.659
250
200
150
100
50
PPM

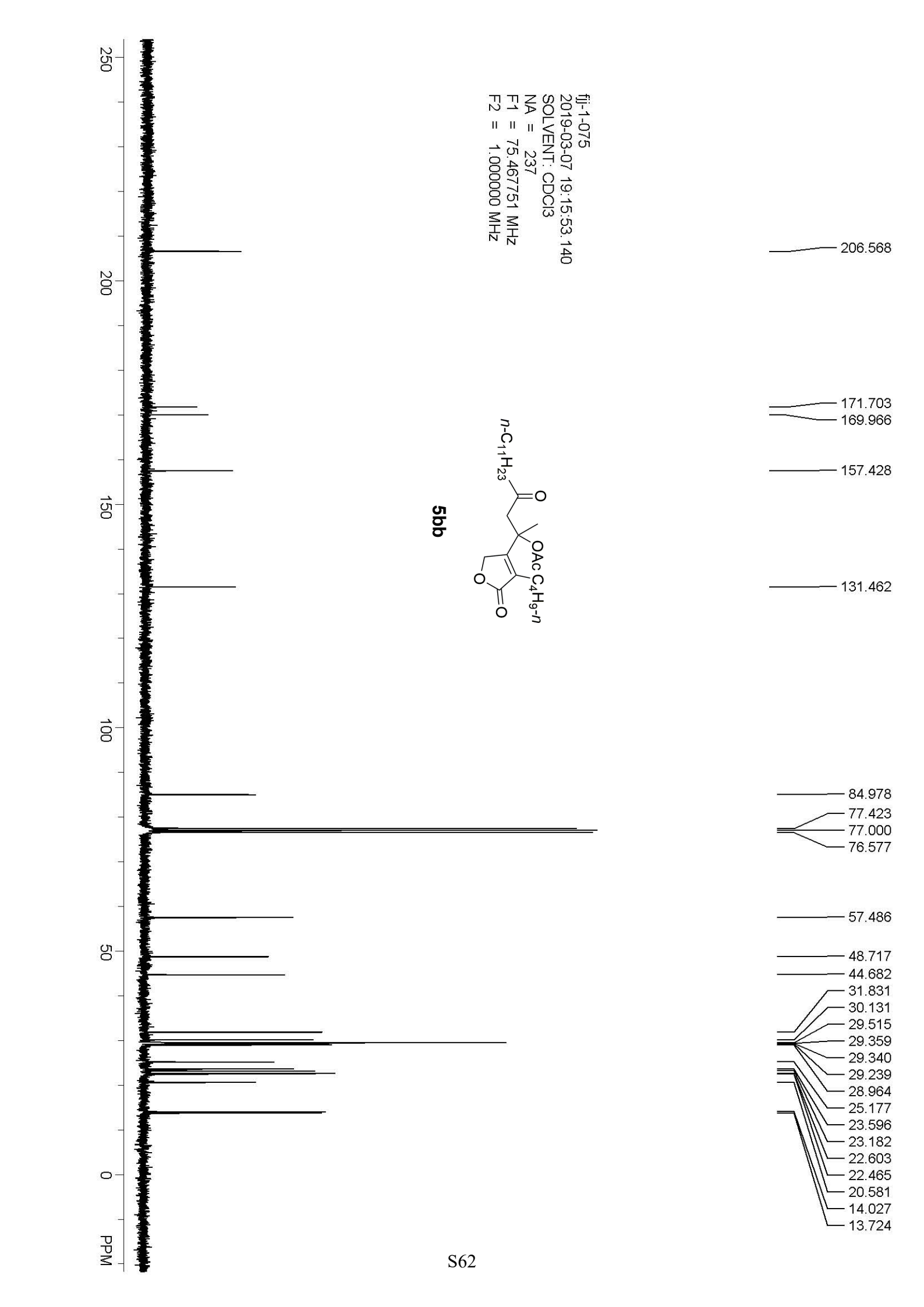

7.287
4.989
4.940
4.890
3.078
3.029
2.750
2.701
2.463
2.440
2.415
2.321
2.294
2.105
1.519
1.506
1.481
1.457
1.264
1.248
1.241
0.948
0.924
0.900
0.890
0.868
0.844
0.000
fjj-1-104
2019-04-01 13:33:56.250
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
O
OAc C4H9-n
n-C6H13
O
O
5ab
S57
2.02
1.00
0.98
2.03
2.07
2.90
7.21
8.61
6.48
10
8
6
4
2
0 PPM

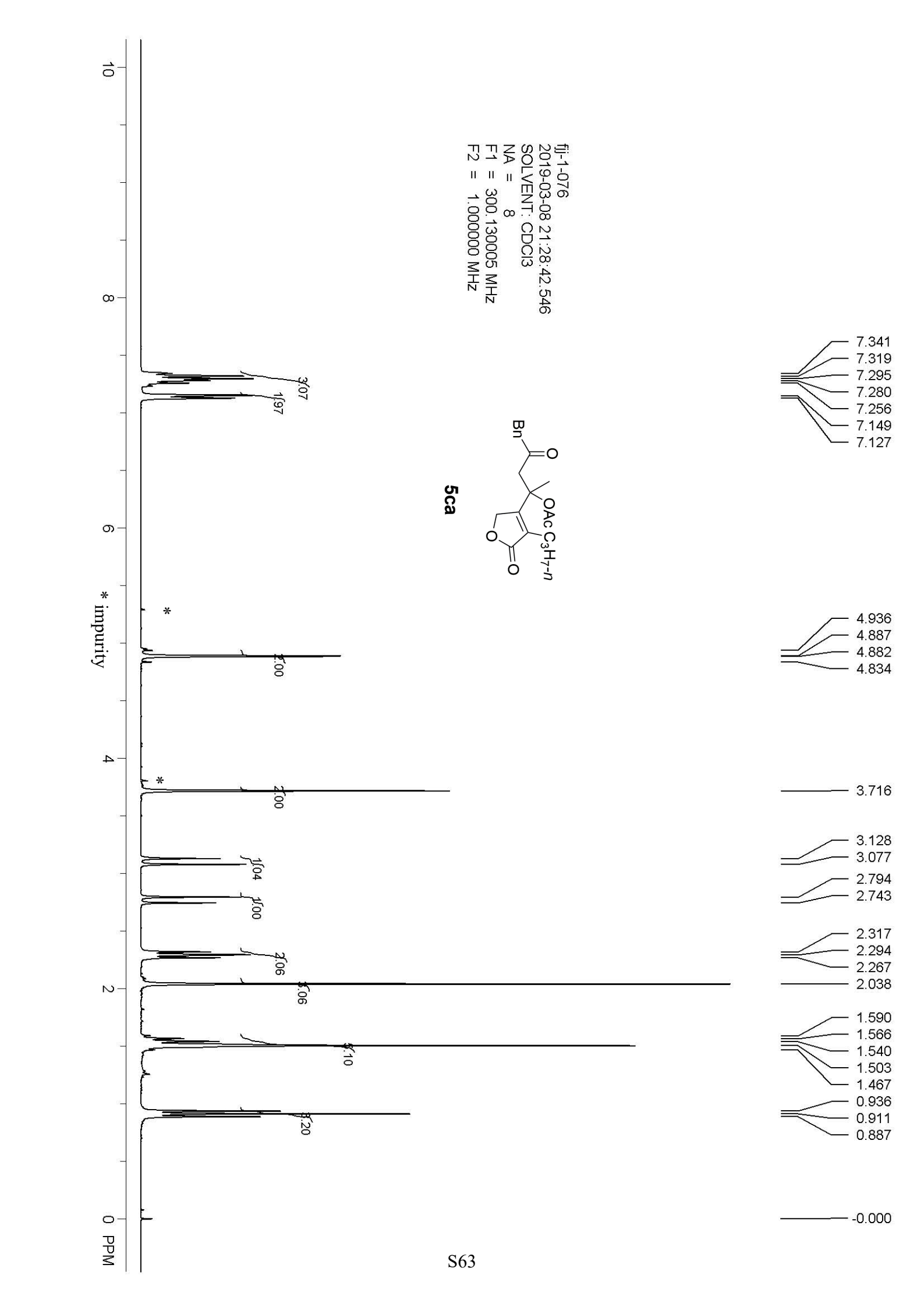

fjj-1-104
2019-04-01 13:47:49.953
SOLVENT: CDCl3
NA = 200
F1 = 75.467751 MHz
F2 = 1.000000 MHz
5ab
206.632
171.758
170.012
157.438
131.416
85.015
77.423
77.000
76.577
57.495
48.680
44.682
31.501
30.140
28.605
25.186
23.596
23.099
22.474
22.382
20.617
13.944
13.751
200
150
100
50
PPM

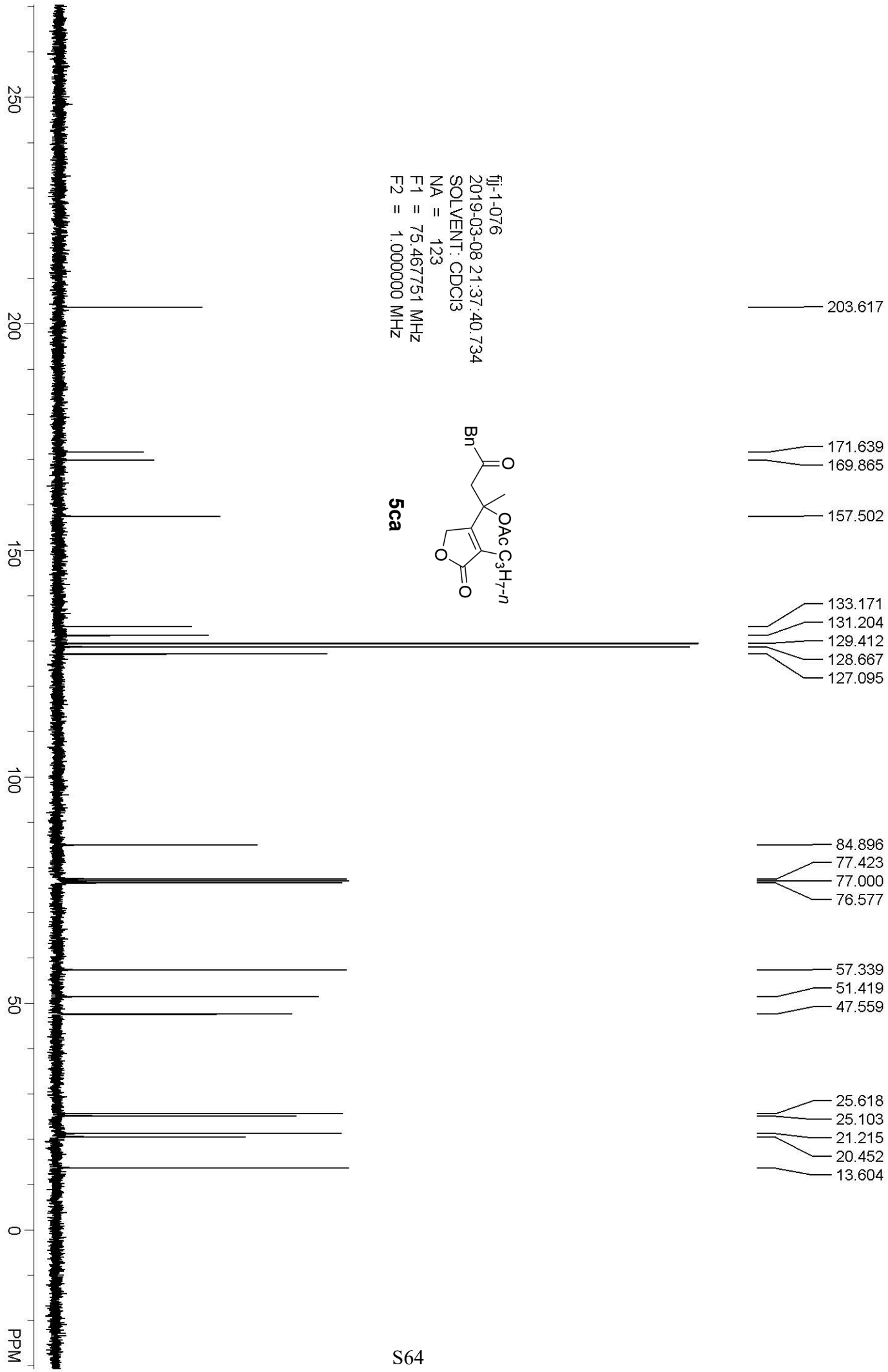

7.303
4.993
4.946
4.896
3.083
3.034
2.762
2.712
2.459
2.435
2.410
2.331
2.307
2.280
2.101
1.581
1.556
1.521
1.484
1.245
0.961
0.936
0.912
0.899
0.877
0.855
0.000
fjj-1-073
2019-03-06 10:53:48.109
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
O
OAc
C3H7-n
n-C11H23
O
O
5ba
S59
2.02
1.00
0.99
2.00
2.04
2.98
7.32
6.72
6.39
10
8
6
4
2
0 PPM

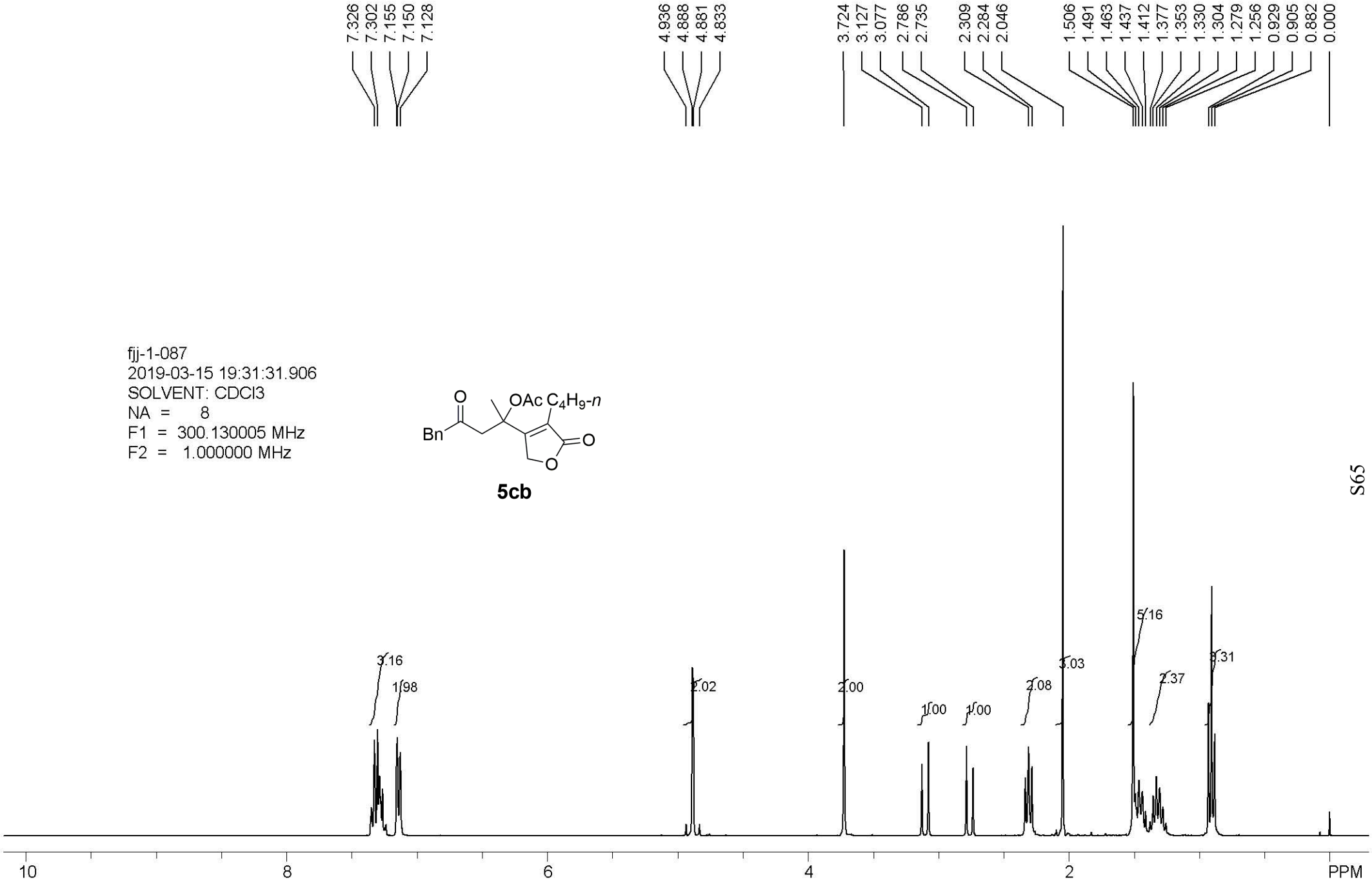

fji-1-073
2019-03-06 11:06:53.531
SOLVENT: CDCl3
NA = 198
F1 = 75.467751 MHz
F2 = 1.000000 MHz
5ba
206.540
171.694
169.938
157.658
131.177
84.969
77.423
77.000
76.577
57.449
48.597
44.636
31.795
29.478
29.322
29.294
29.212
28.918
25.664
25.204
23.136
22.566
21.298
20.544
13.999
13.659
250
200
150
100
50
PPM

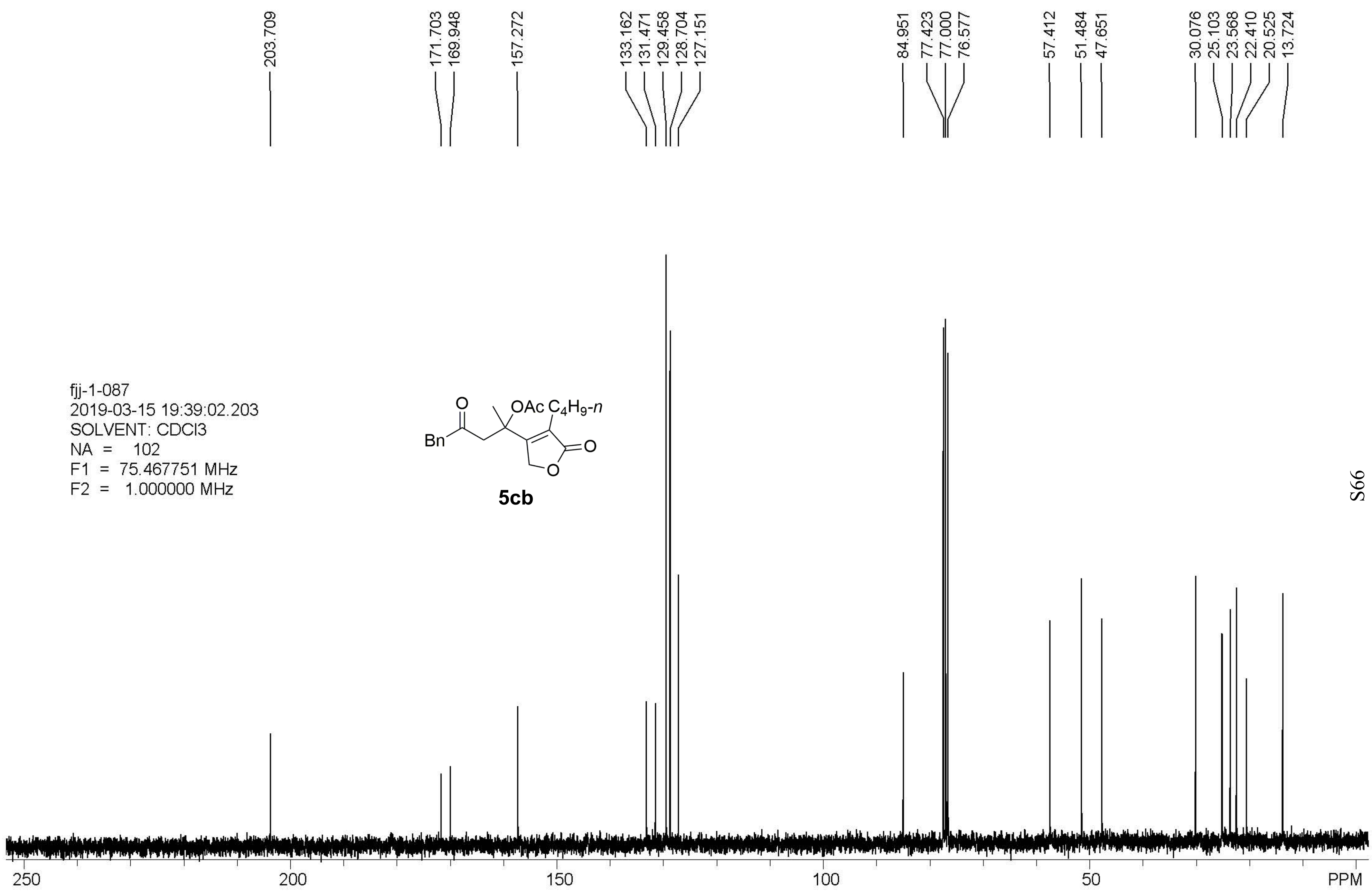

7.288
4.988
4.939
4.889
3.070
3.021
2.747
2.698
2.455
2.432
2.407
2.345
2.320
2.294
2.100
1.516
1.482
1.377
1.353
1.328
1.302
1.244
0.950
0.925
0.901
0.878
0.855
0.000
fjj-1-075
2019-03-07 19:00:09.187
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
n-C11H23
OAc C4H9-n
5bb
21.66
8.30
7.40
2.93
2.22
2.34
2.04
1.00
0.97
10
8
6
4
2
0
PPM


fjj-1-075
2019-03-07 19:15:53.140
SOLVENT: CDCl3
NA = 237
F1 = 75.467751 MHz
F2 = 1.000000 MHz
5bb
206.568
171.703
169.966
157.428
131.462
84.978
77.423
77.000
76.577
57.486
48.717
44.682
31.831
30.131
29.515
29.359
29.340
29.239
28.964
25.177
23.596
23.182
22.603
22.465
20.581
14.027
13.724
250
200
150
100
50
0
PPM

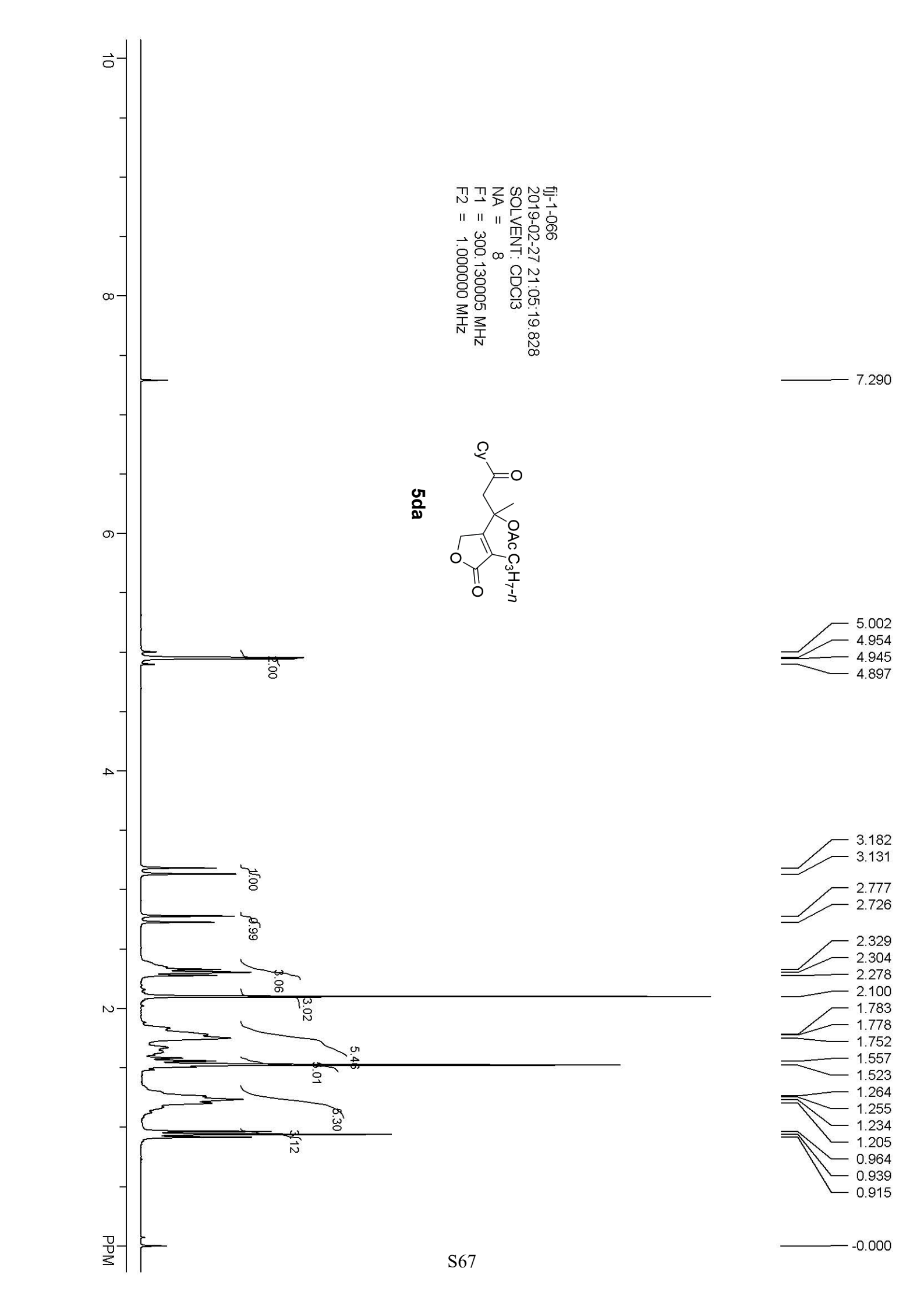

fji-1-076
2019-03-08 21:28:42.546
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
5ca
Bn
OAc
C3H7-n
* impurity
PPM
10
8
6
4
2
0
7.341
7.319
7.295
7.280
7.256
7.149
7.127
4.936
4.887
4.882
4.834
3.716
3.128
3.077
2.794
2.743
2.317
2.294
2.267
2.038
1.590
1.566
1.540
1.503
1.467
0.936
0.911
0.887
-0.000

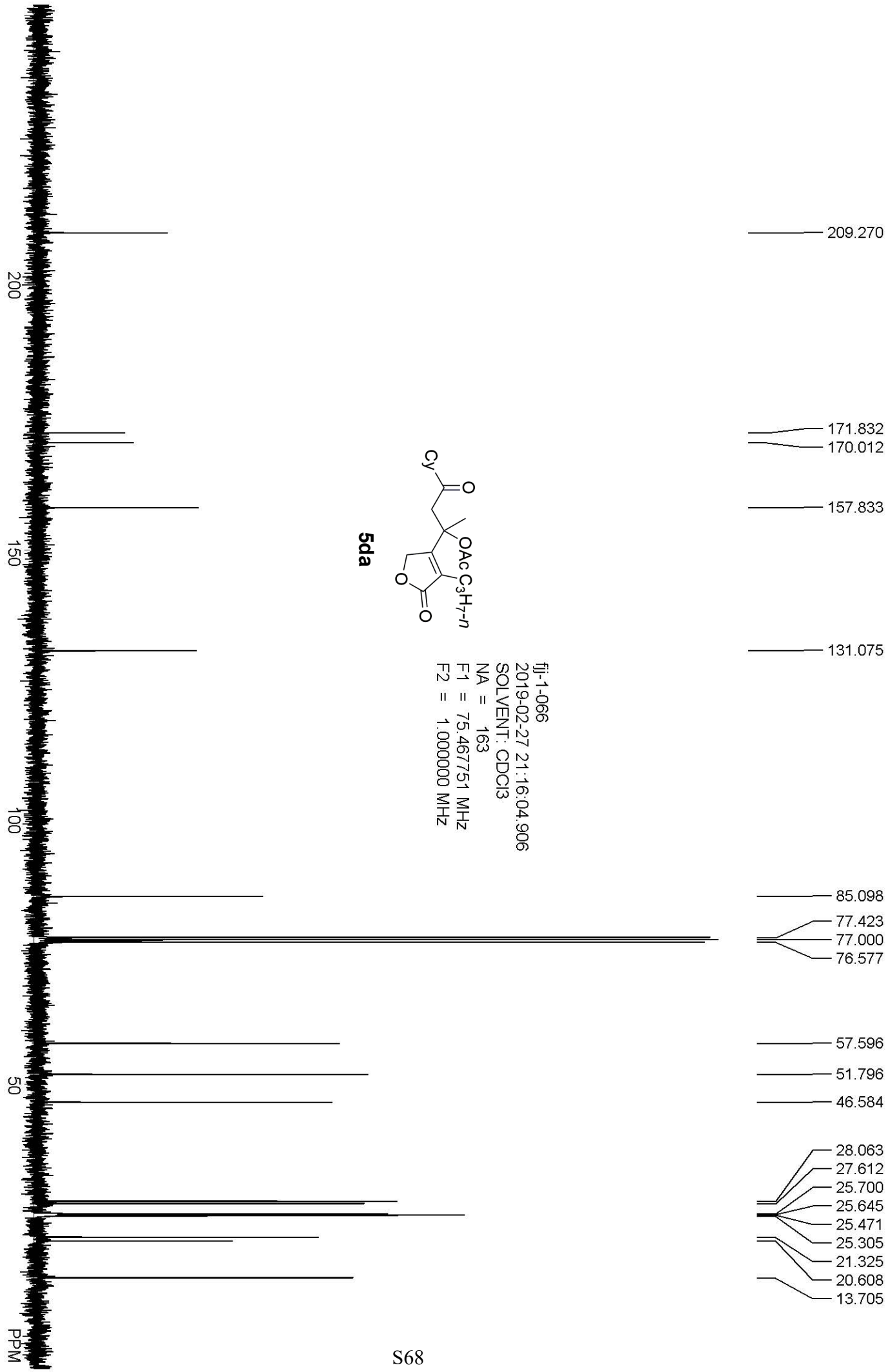

fjj-1-076
2019-03-08 21:37:40.734
SOLVENT: CDCl3
NA = 123
F1 = 75.467751 MHz
F2 = 1.000000 MHz
5ca
Bn
OAc
C3H7-n
203.617
171.639
169.865
157.502
133.171
131.204
129.412
128.667
127.095
84.896
77.423
77.000
76.577
57.339
51.419
47.559
25.618
25.103
21.215
20.452
13.604
250
200
150
100
50
0
PPM

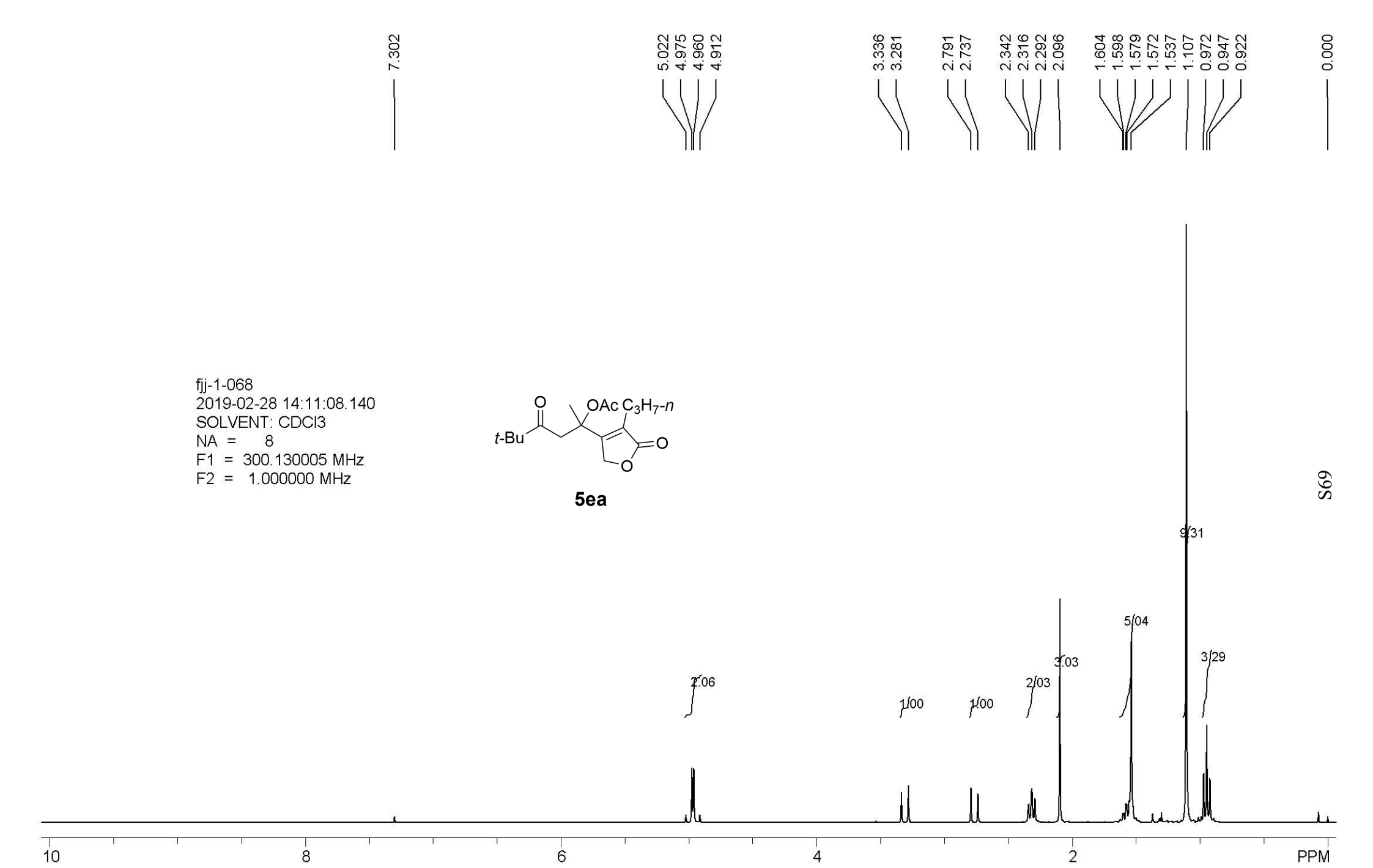

7.326
7.302
7.155
7.150
7.128
4.936
4.888
4.881
4.833
3.724
3.127
3.077
2.786
2.735
2.309
2.284
2.046
1.506
1.491
1.463
1.437
1.412
1.377
1.353
1.330
1.304
1.279
1.256
0.929
0.905
0.882
0.000
fjj-1-087
2019-03-15 19:31:31.906
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
O
OAc C4H9-n
Bn
O
O
5cb
3.16
1.98
2.02
2.00
1.00
1.00
2.08
3.03
5.16
2.37
3.31
10
8
6
4
2
PPM

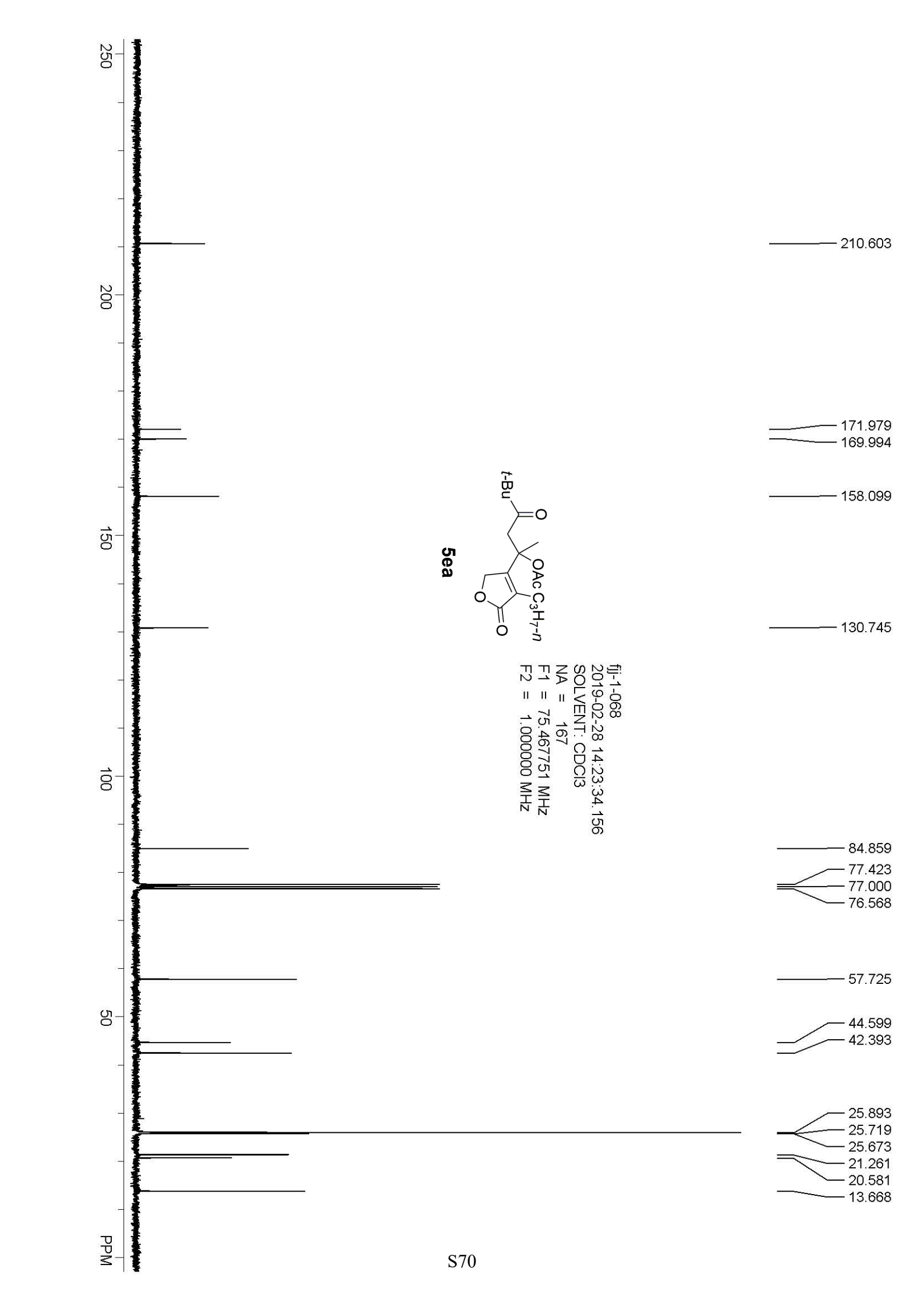


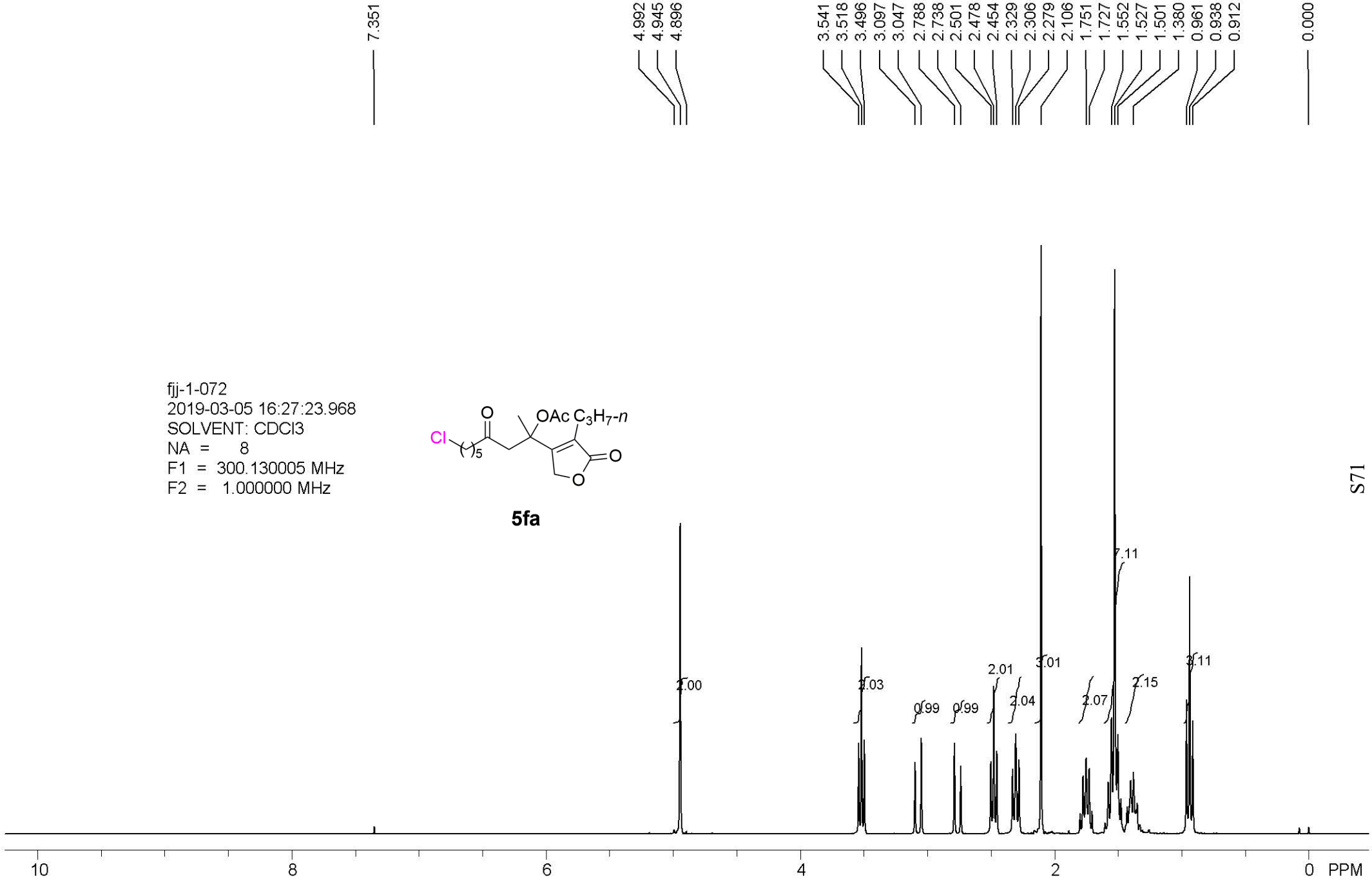
203.709
171.703
169.948
157.272
133.162
131.471
129.458
128.704
127.151
84.951
77.423
77.000
76.577
57.412
51.484
47.651
30.076
25.103
23.568
22.410
20.525
13.724
fjj-1-087
2019-03-15 19:39:02.203
SOLVENT: CDCl3
NA = 102
F1 = 75.467751 MHz
F2 = 1.000000 MHz
O
OAc
C_4H_9-n
Bn
O
O
5cb
250
200
150
100
50
PPM

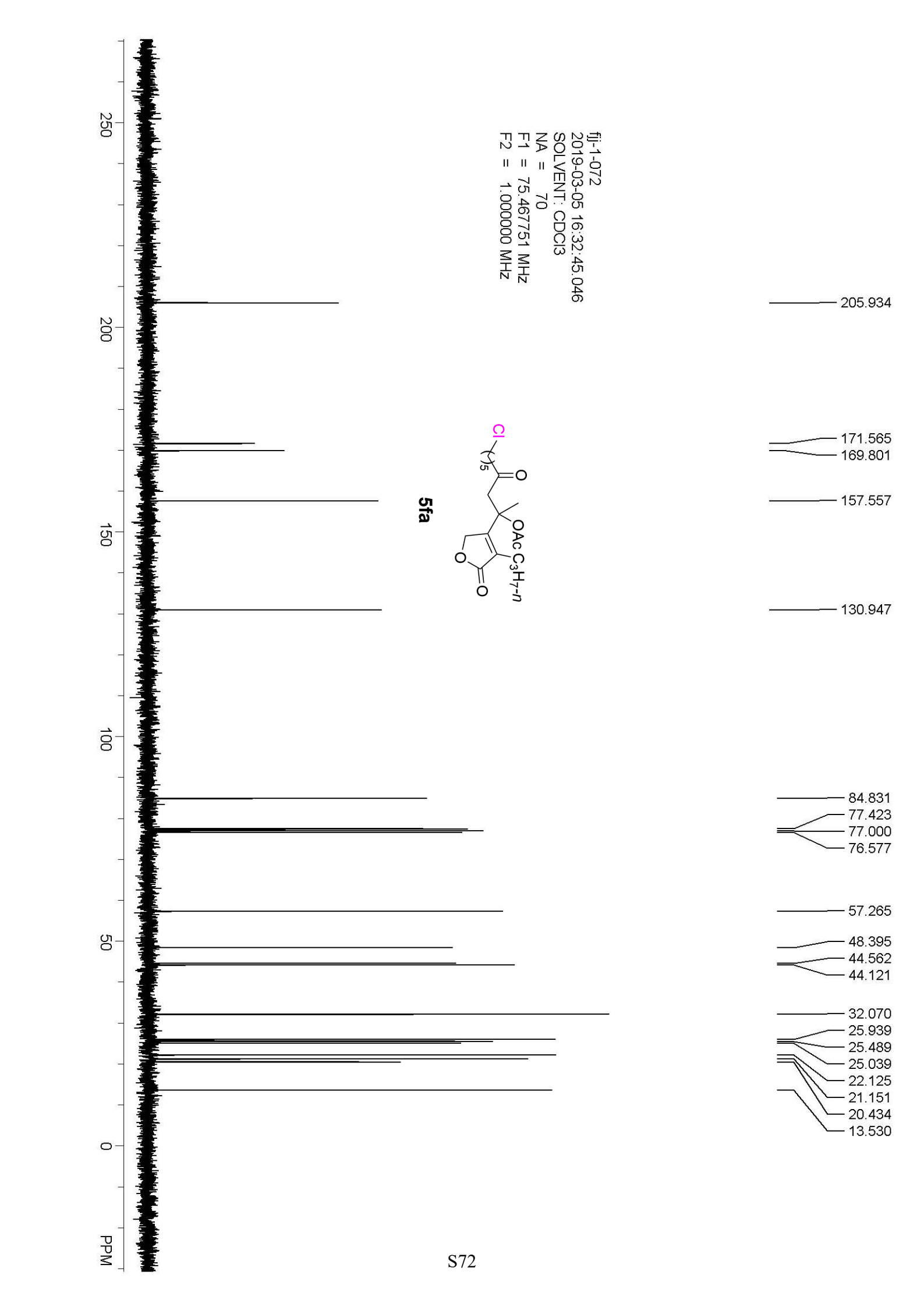

fjj-1-066
2019-02-27 21:05:19.828
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
5da
Cy
O
OAc
C3H7-n
O
O
O
10
8
6
4
2
PPM
2.00
1.00
0.99
3.06
3.02
5.46
5.01
5.30
3.12
7.290
5.002
4.954
4.945
4.897
3.182
3.131
2.777
2.726
2.329
2.304
2.278
2.100
1.783
1.778
1.752
1.557
1.523
1.264
1.255
1.234
1.205
0.964
0.939
0.915
-0.000

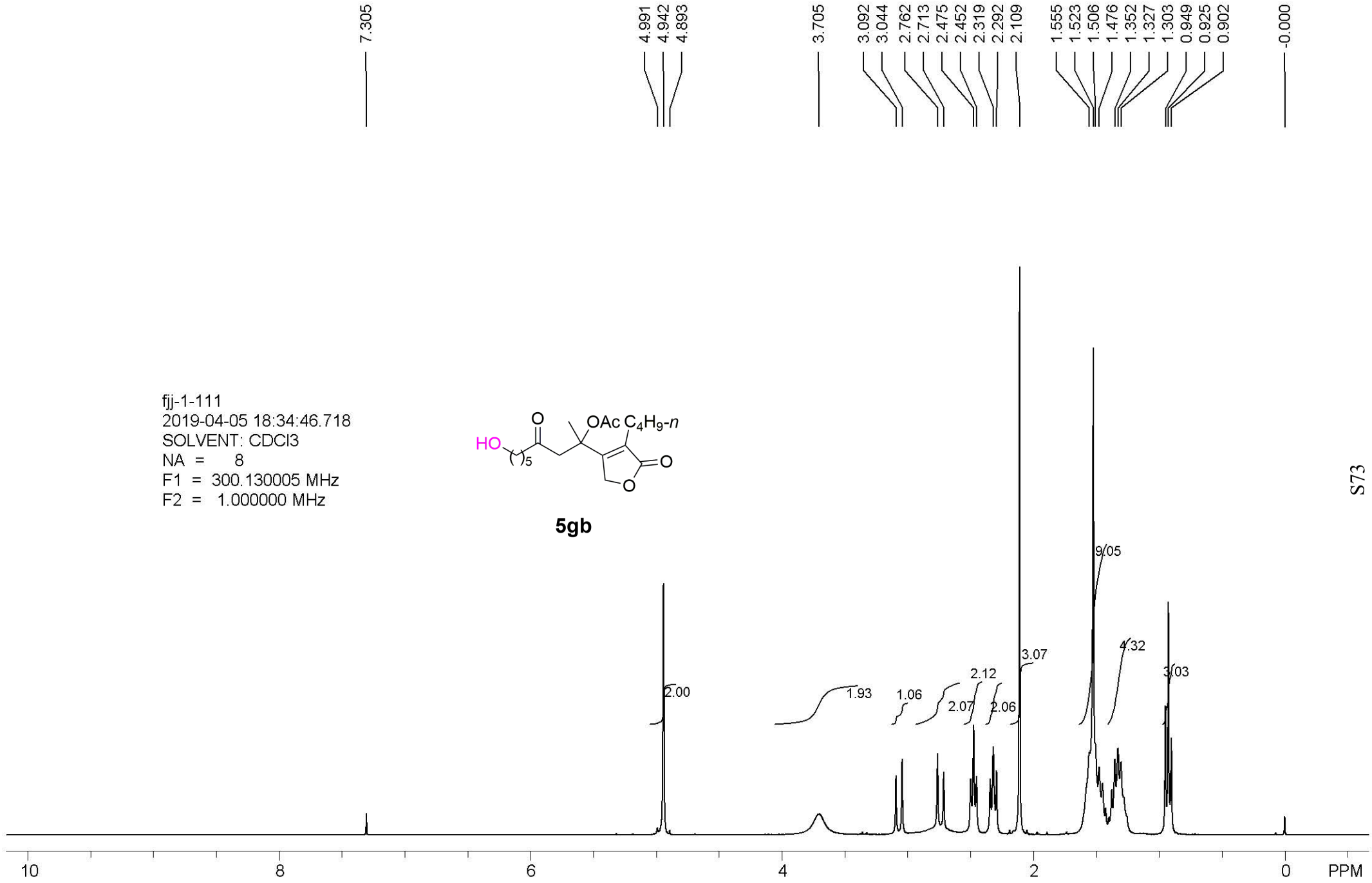

209.270
171.832
170.012
157.833
131.075
85.098
77.423
77.000
76.577
57.596
51.796
46.584
28.063
27.612
25.700
25.645
25.471
25.305
21.325
20.608
13.705
200
150
100
50
PPM
5da
Cy
O
OAc
C3H7-n
O
O
fji-1-066
2019-02-27 21:16:04.906
SOLVENT: CDCl3
NA = 163
F1 = 75.467751 MHz
F2 = 1.000000 MHz

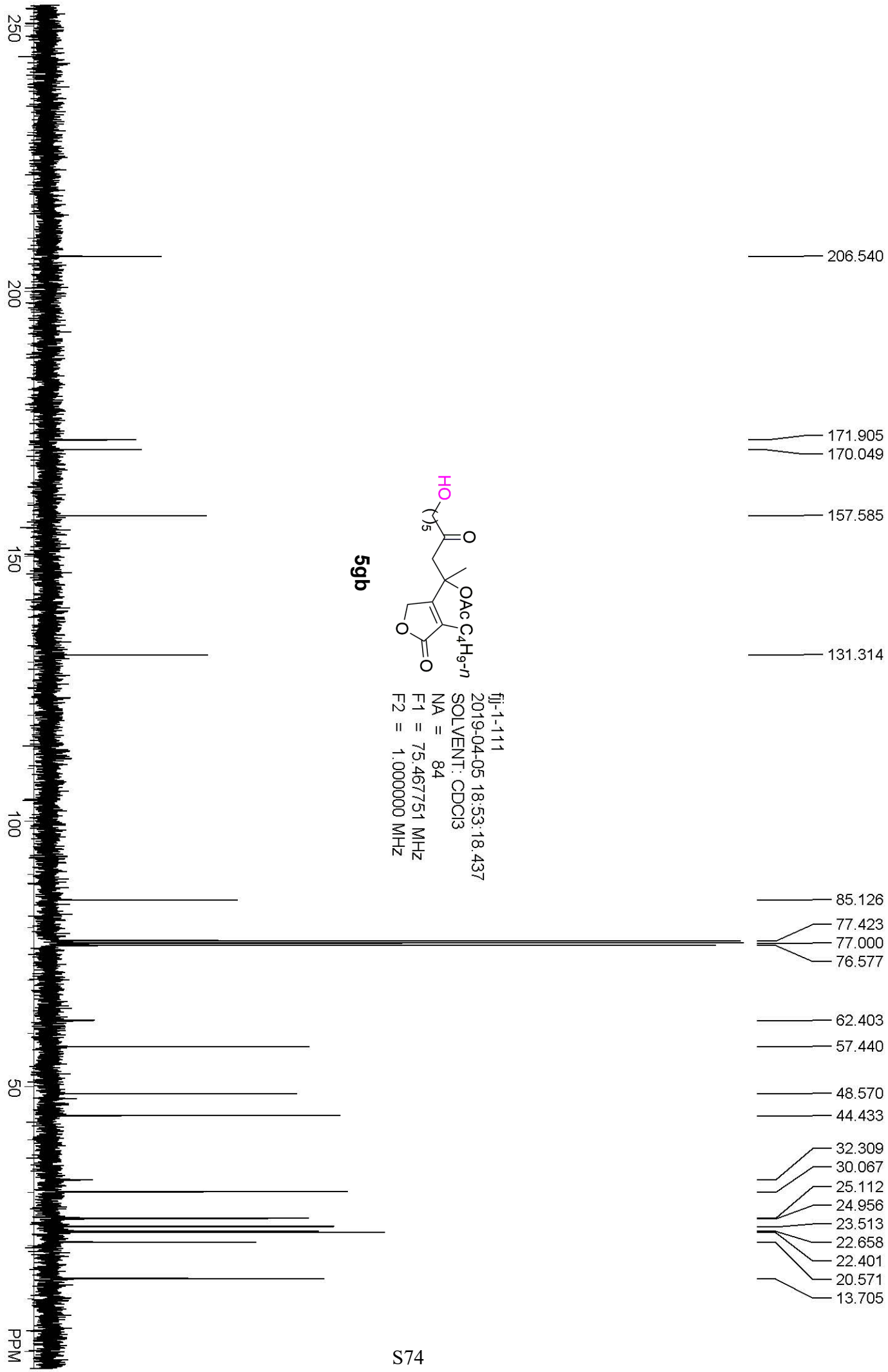

7.302
5.022
4.975
4.960
4.912
3.336
3.281
2.791
2.737
2.342
2.316
2.292
2.096
1.604
1.598
1.579
1.572
1.537
1.107
0.972
0.947
0.922
0.000
fjj-1-068
2019-02-28 14:11:08.140
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
t-Bu
OAc
C3H7-n
5ea
2.06
1.00
1.00
2.03
3.03
5.04
9.31
3.29
10
8
6
4
2
PPM

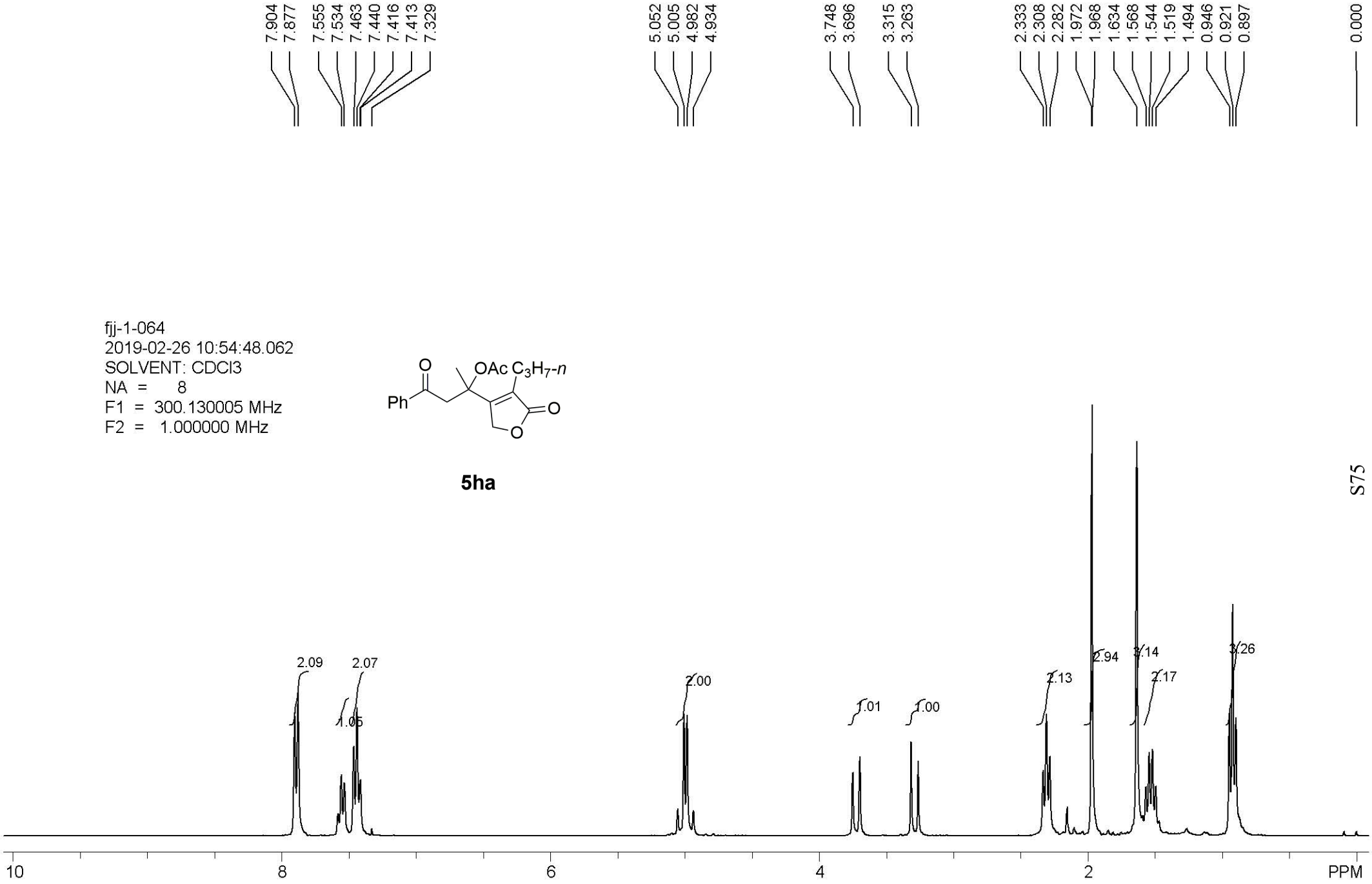

210.603
171.979
169.994
158.099
130.745
84.859
77.423
77.000
76.568
57.725
44.599
42.393
25.893
25.719
25.673
21.261
20.581
13.668
250
200
150
100
50
PPM
5ea
t-Bu
OAc
C3H7-n
fjj-1-068
2019-02-28 14:23:34.156
SOLVENT: CDCl3
NA = 167
F1 = 75.467751 MHz
F2 = 1.000000 MHz

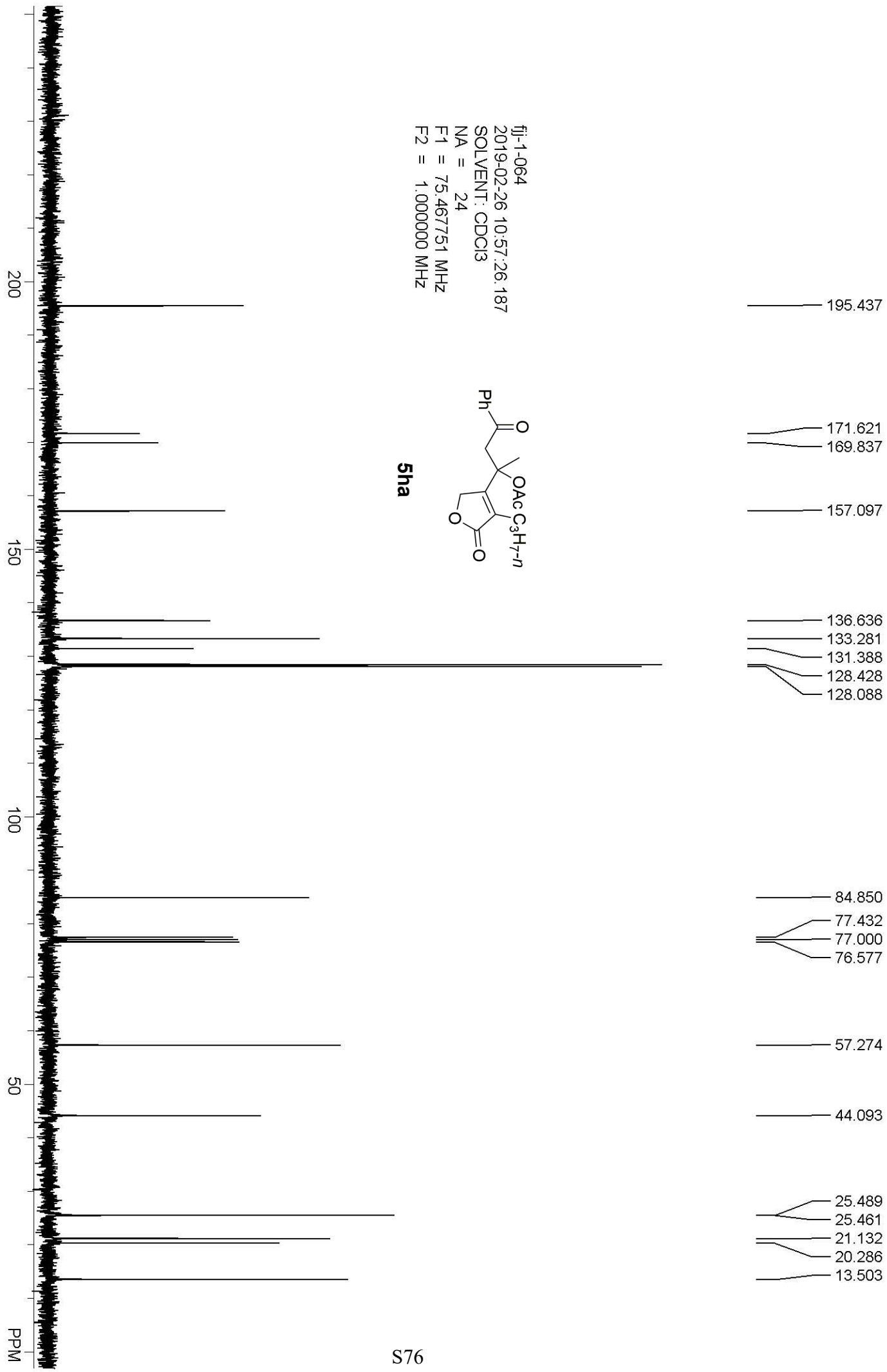

7.351
4.992
4.945
4.896
3.541
3.518
3.496
3.097
3.047
2.788
2.738
2.501
2.478
2.454
2.329
2.306
2.279
2.106
1.751
1.727
1.552
1.527
1.501
1.380
0.961
0.938
0.912
0.000
fjj-1-072
2019-03-05 16:27:23.968
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Cl
O
OAc
C3H7-n
O
O
5fa
2.00
2.03
0.99
0.99
2.01
2.04
3.01
2.07
7.11
2.15
3.11
10
8
6
4
2
0
PPM

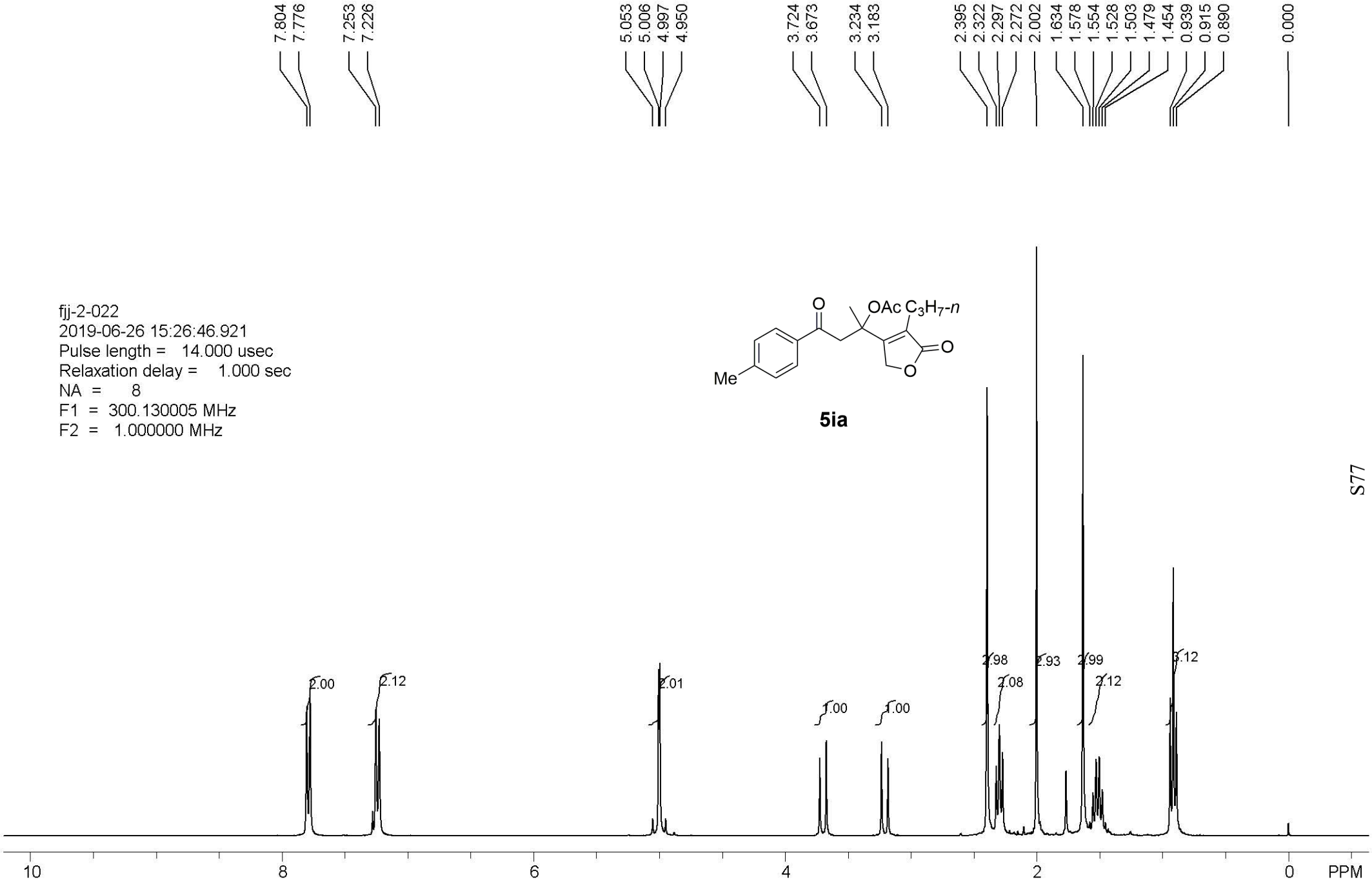

fjj-1-072
2019-03-05 16:32:45.046
SOLVENT: CDCl3
NA = 70
F1 = 75.467751 MHz
F2 = 1.000000 MHz
Cl
O
OAc
C3H7-n
O
O
O
5fa
205.934
171.565
169.801
157.557
130.947
84.831
77.423
77.000
76.577
57.265
48.395
44.562
44.121
32.070
25.939
25.489
25.039
22.125
21.151
20.434
13.530
250
200
150
100
50
0
PPM

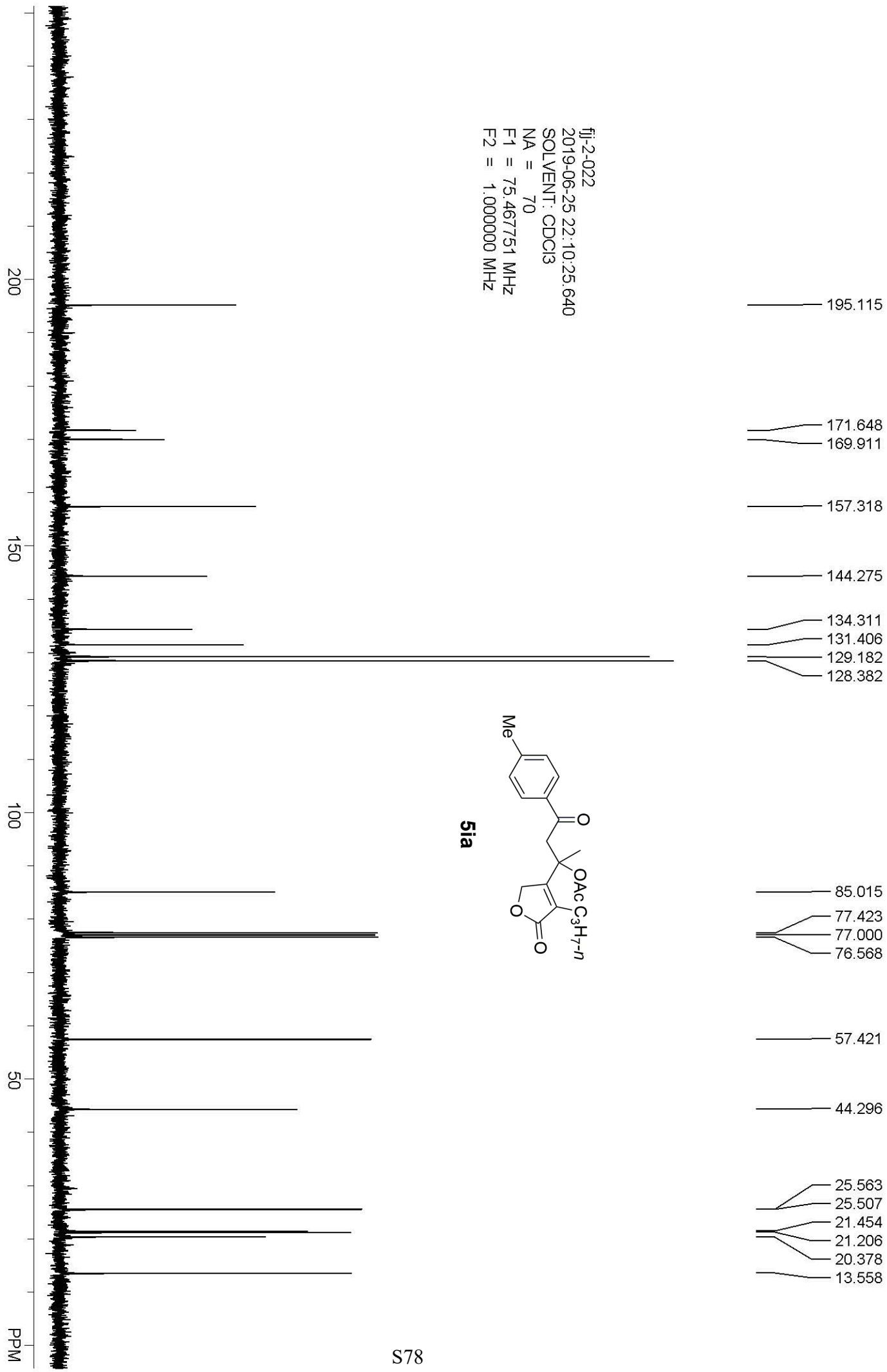

7.305
4.991
4.942
4.893
3.705
3.092
3.044
2.762
2.713
2.475
2.452
2.319
2.292
2.109
1.555
1.523
1.506
1.476
1.352
1.327
1.303
0.949
0.925
0.902
-0.000
fjj-1-111
2019-04-05 18:34:46.718
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
HO
5
O
OAc C4H9-n
O
O
5gb
2.00
1.93
1.06
2.07
2.12
2.06
3.07
9.05
4.32
3.03
10
8
6
4
2
0
PPM

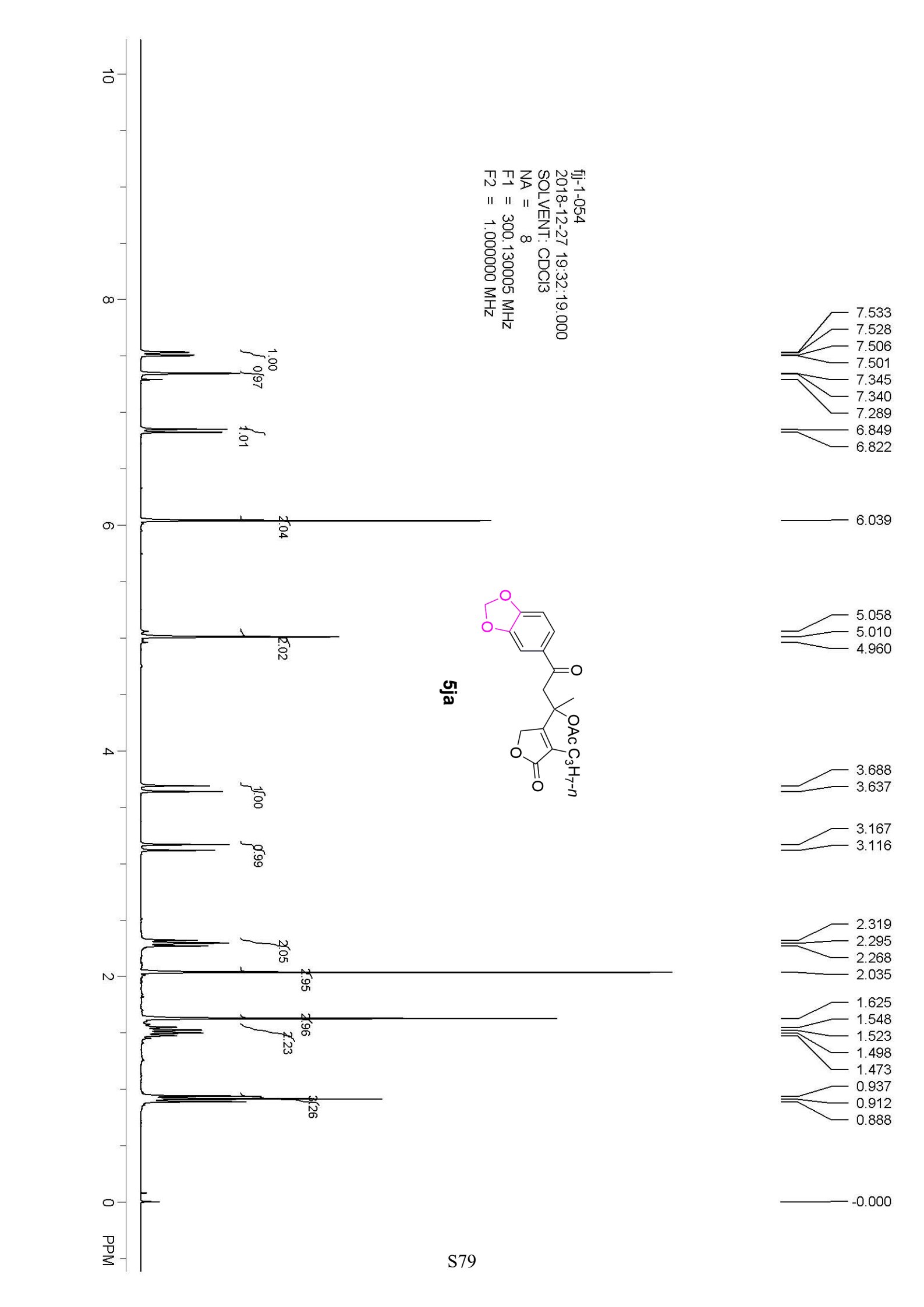

206.540
171.905
170.049
157.585
131.314
85.126
77.423
77.000
76.577
62.403
57.440
48.570
44.433
32.309
30.067
25.112
24.956
23.513
22.658
22.401
20.571
13.705
250
200
150
100
50
PPM
5gb
HO
OAc
C4H9-n
fjj-1-111
2019-04-05 18:53:18.437
SOLVENT: CDCl3
NA = 84
F1 = 75.467751 MHz
F2 = 1.000000 MHz

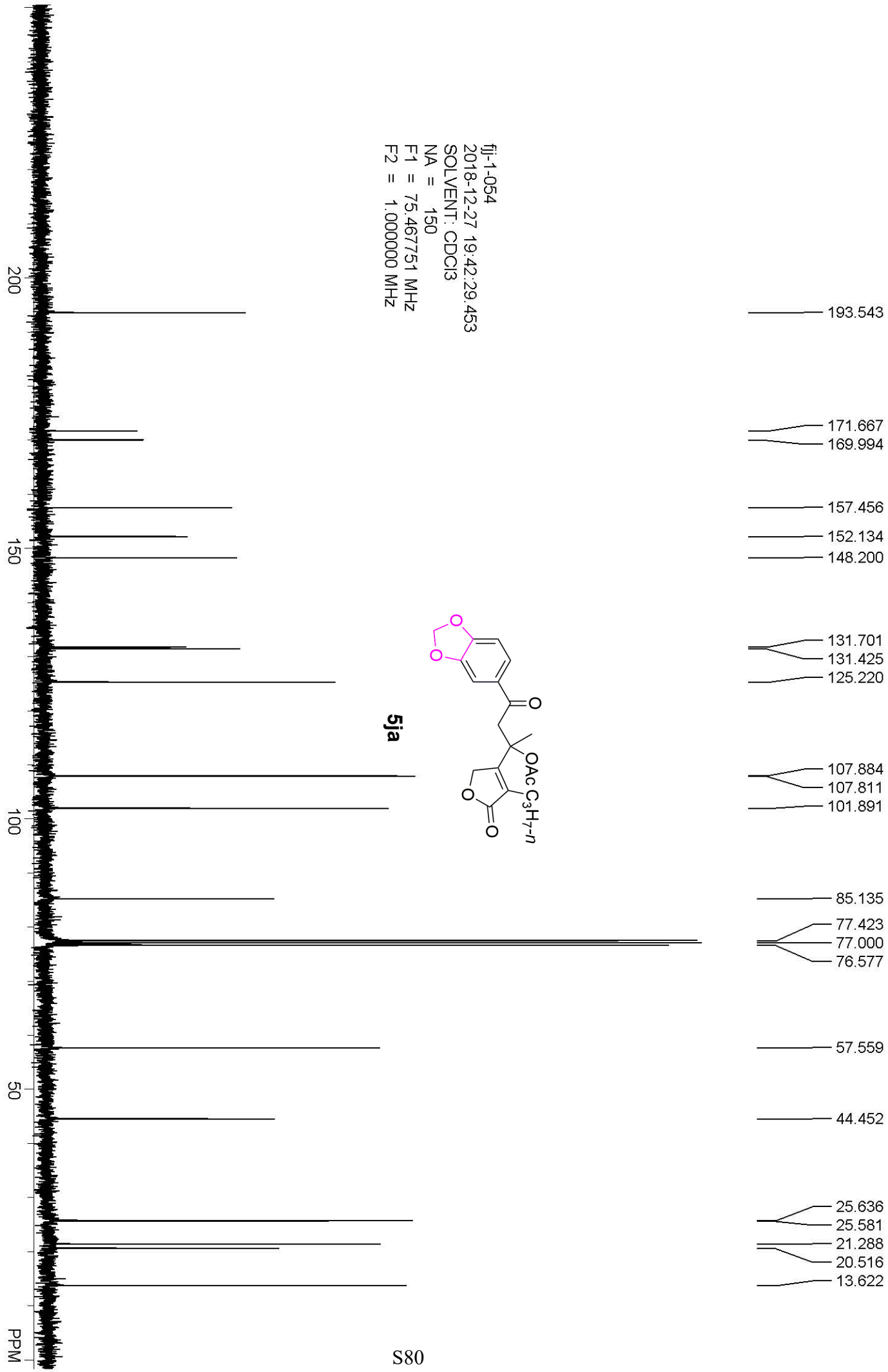

7.904
7.877
7.555
7.534
7.463
7.440
7.416
7.413
7.329
5.052
5.005
4.982
4.934
3.748
3.696
3.315
3.263
2.333
2.308
2.282
1.972
1.968
1.634
1.568
1.544
1.519
1.494
0.946
0.921
0.897
0.000
fjj-1-064
2019-02-26 10:54:48.062
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
O
OAc
C3H7-n
Ph
O
O
5ha
2.09
2.07
1.05
2.00
1.01
1.00
2.13
2.94
3.14
2.17
3.26
10
8
6
4
2
PPM

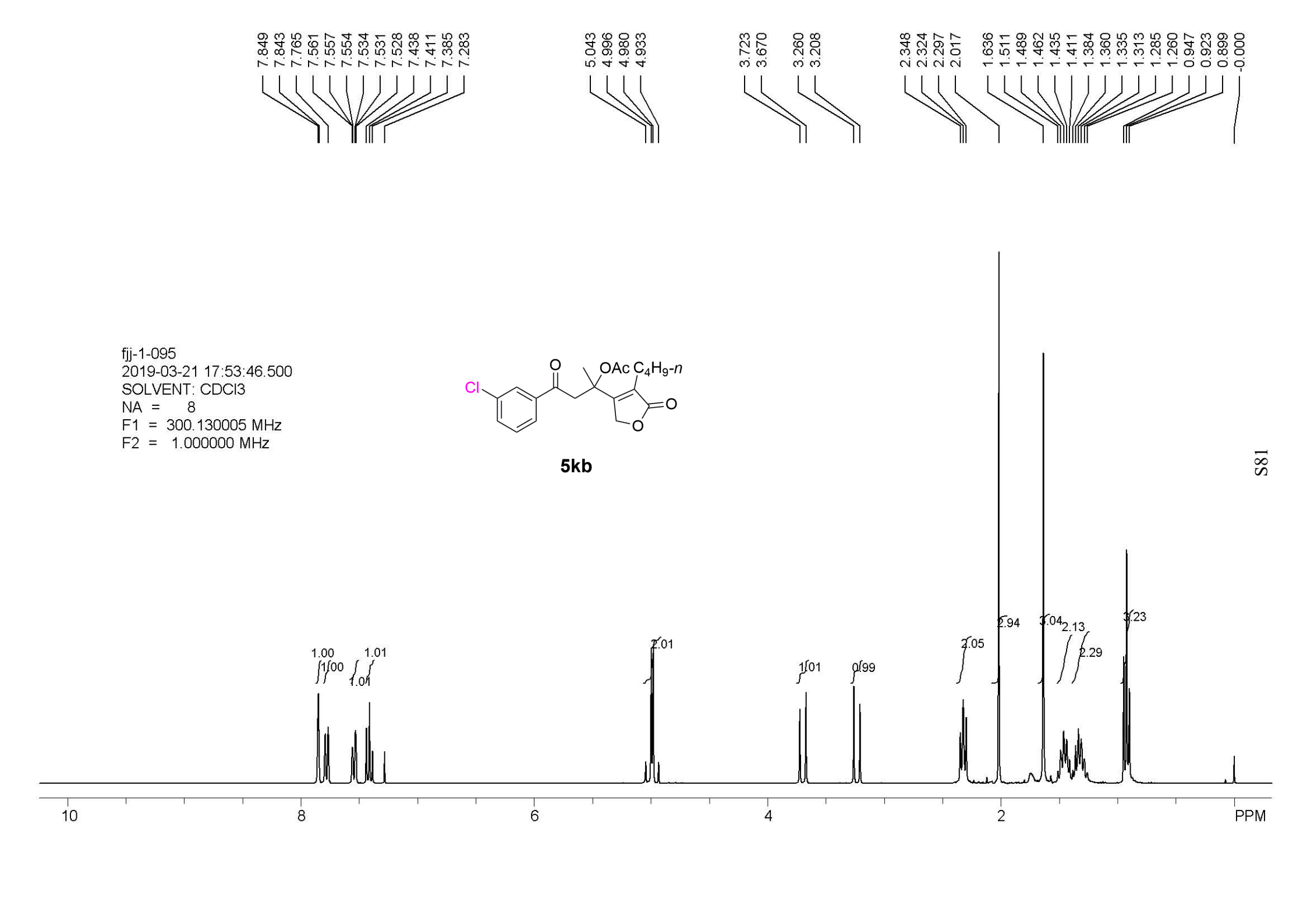

fjj-1-064
2019-02-26 10:57:26.187
SOLVENT: CDCl3
NA = 24
F1 = 75.467751 MHz
F2 = 1.000000 MHz
5ha
Ph
O
OAc
C3H7-n
O
O
195.437
171.621
169.837
157.097
136.636
133.281
131.388
128.428
128.088
84.850
77.432
77.000
76.577
57.274
44.093
25.489
25.461
21.132
20.286
13.503
200
150
100
50
PPM

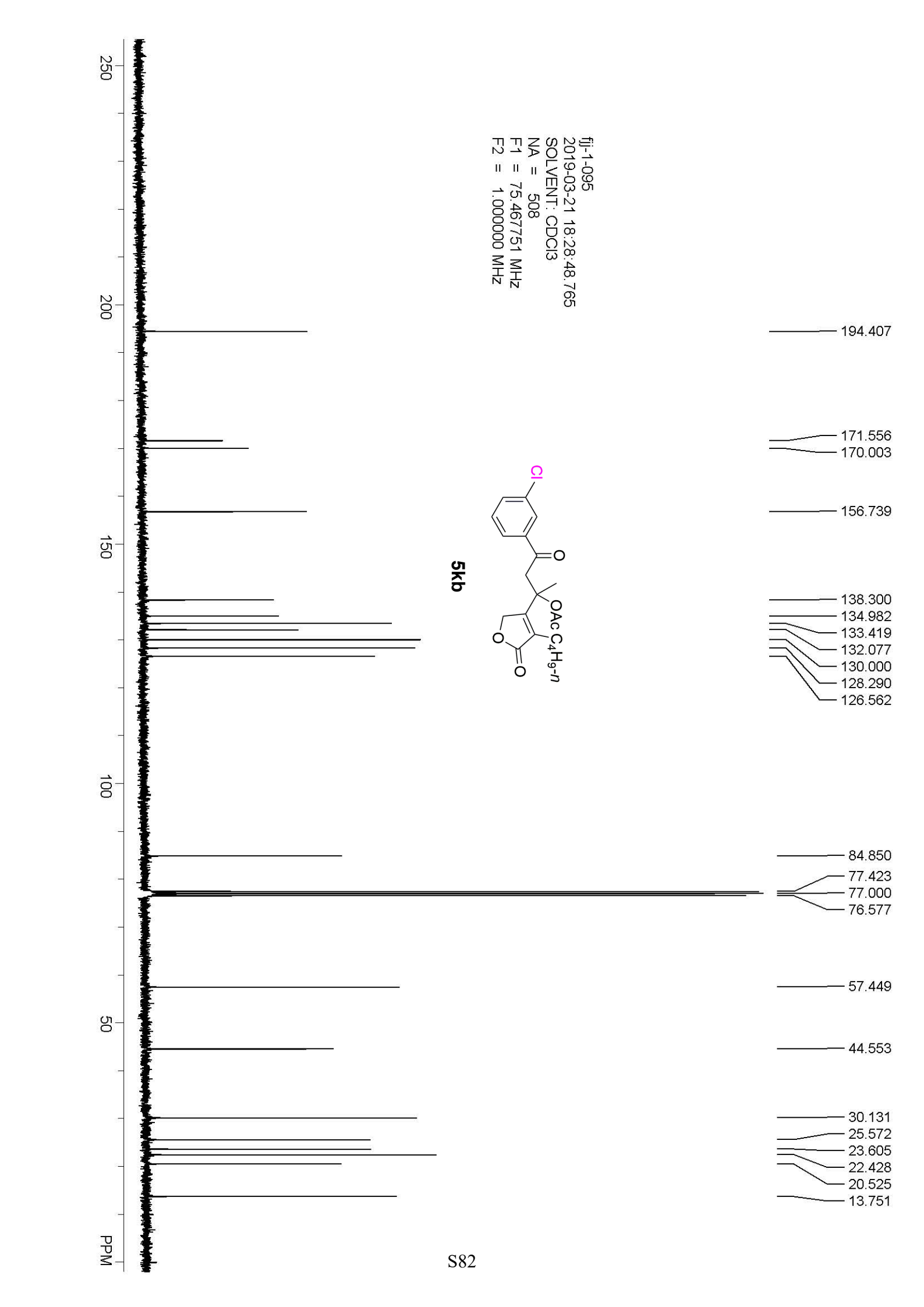

7.804
7.776
7.253
7.226
5.053
5.006
4.997
4.950
3.724
3.673
3.234
3.183
2.395
2.322
2.297
2.272
2.002
1.634
1.578
1.554
1.528
1.503
1.479
1.454
0.939
0.915
0.890
0.000
fjj-2-022
2019-06-26 15:26:46.921
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
O
OAc
C3H7-n
O
Me
O
5ia
2.00
2.12
2.01
1.00
1.00
2.98
2.08
2.93
2.99
2.12
3.12
10
8
6
4
2
0
PPM

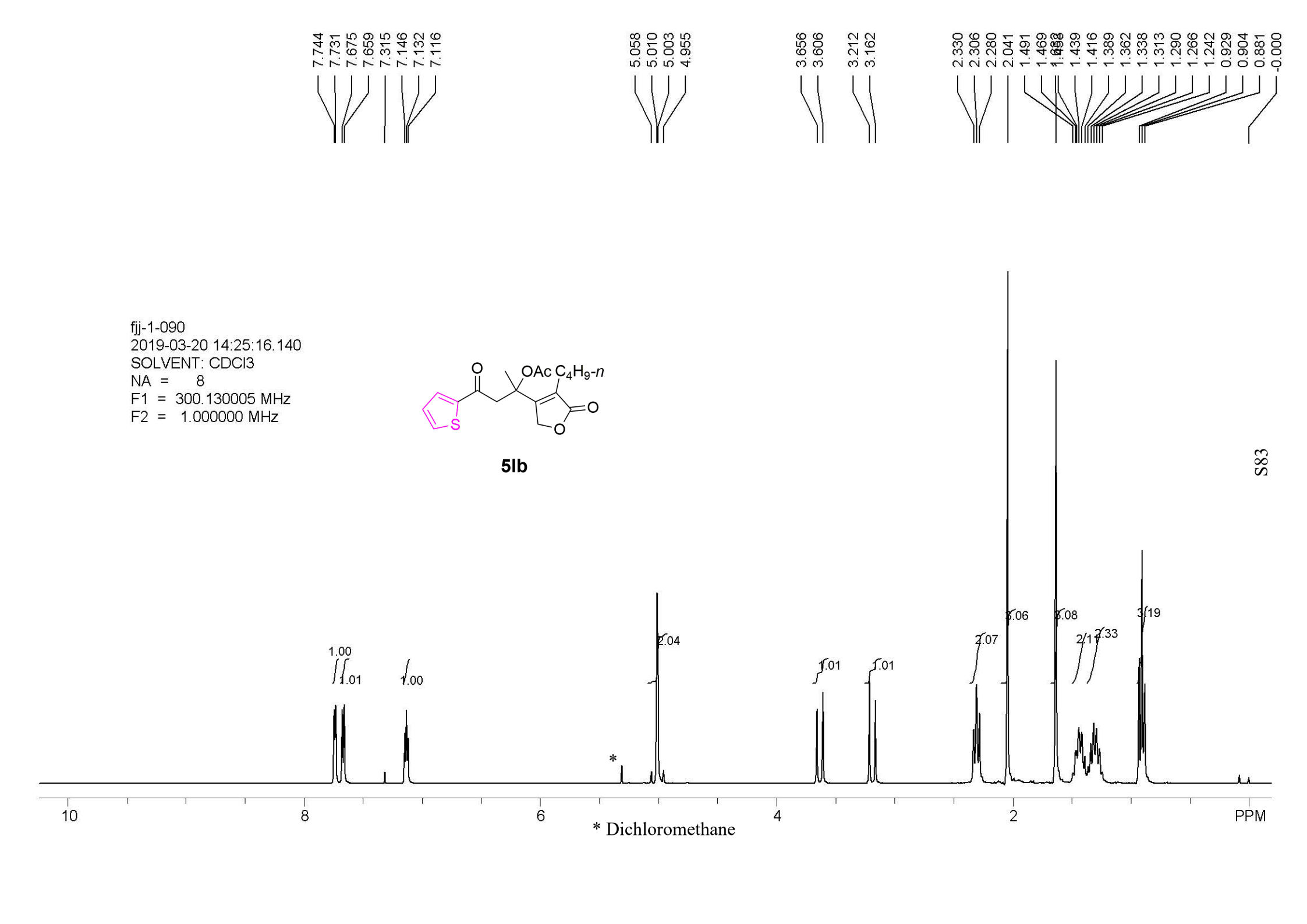

fjj-2-022
2019-06-25 22:10:25.640
SOLVENT: CDCl3
NA = 70
F1 = 75.467751 MHz
F2 = 1.000000 MHz
195.115
171.648
169.911
157.318
144.275
134.311
131.406
129.182
128.382
85.015
77.423
77.000
76.568
57.421
44.296
25.563
25.507
21.454
21.206
20.378
13.558
200
150
100
50
PPM
Me
O
OAc
C3H7-n
O
O
5ia

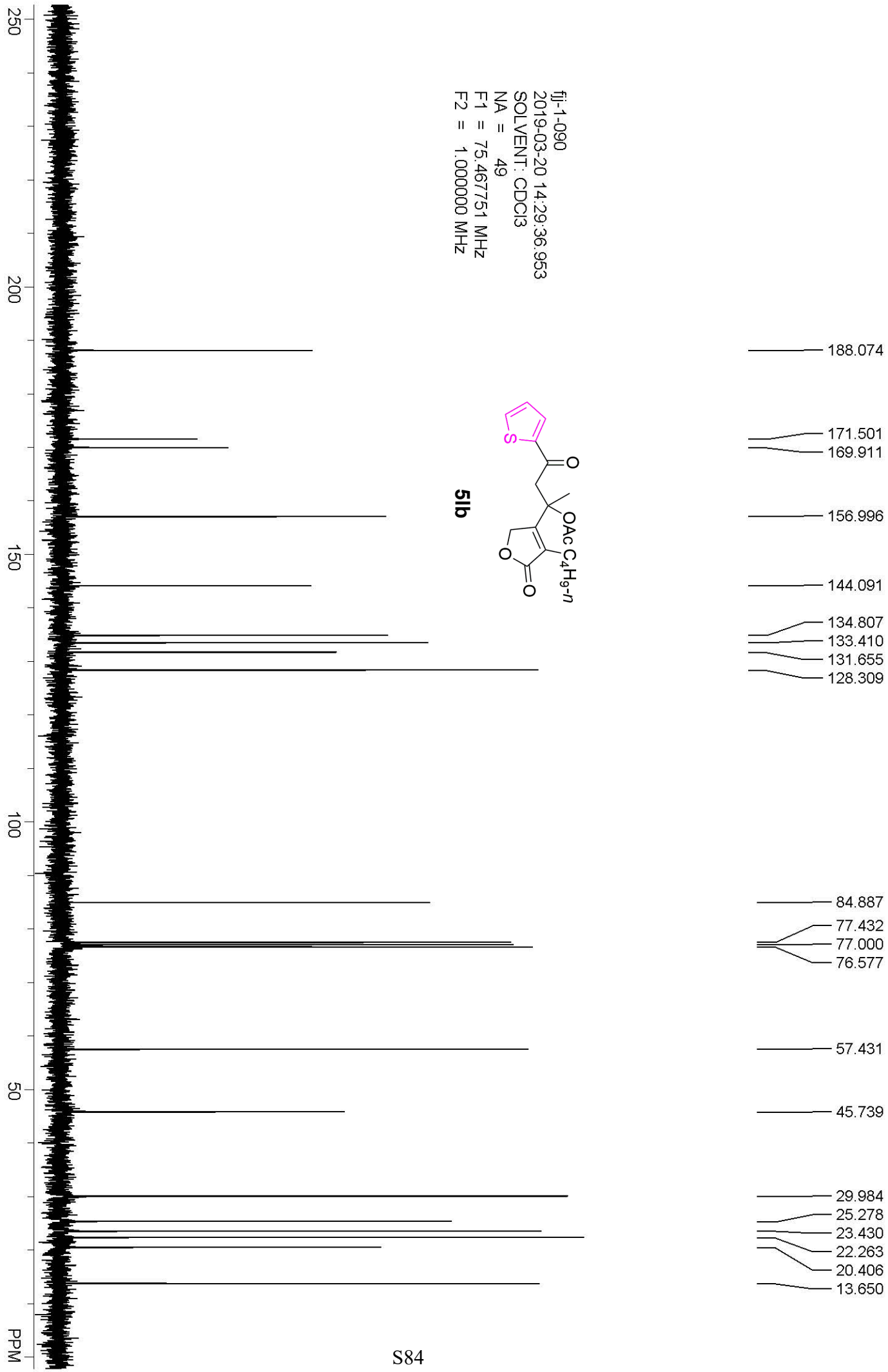

fji-1-054
2018-12-27 19:32:19.000
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
5ja
OAc
C3H7-n
7.533
7.528
7.506
7.501
7.345
7.340
7.289
6.849
6.822
6.039
5.058
5.010
4.960
3.688
3.637
3.167
3.116
2.319
2.295
2.268
2.035
1.625
1.548
1.523
1.498
1.473
0.937
0.912
0.888
-0.000
1.00
0.97
1.01
2.04
2.02
1.00
0.99
2.05
1.95
2.96
7.23
3.26
PPM

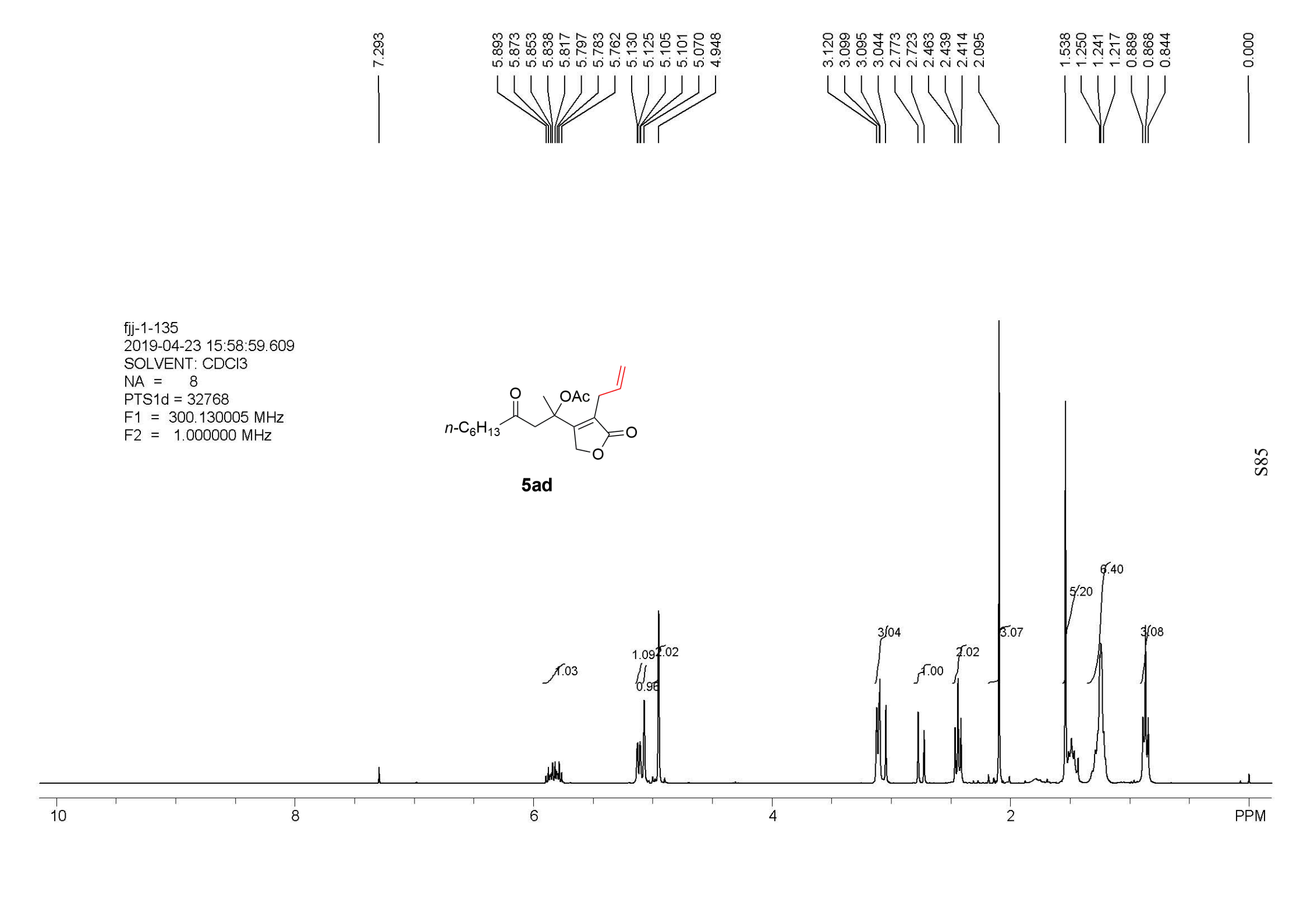

fjj-1-054
2018-12-27 19:42:29.453
SOLVENT: CDCl3
NA = 150
F1 = 75.467751 MHz
F2 = 1.000000 MHz
5ja
OAc
C_3H_{7-n}
193.543
171.667
169.994
157.456
152.134
148.200
131.701
131.425
125.220
107.884
107.811
101.891
85.135
77.423
77.000
76.577
57.559
44.452
25.636
25.581
21.288
20.516
13.622
200
150
100
50
PPM

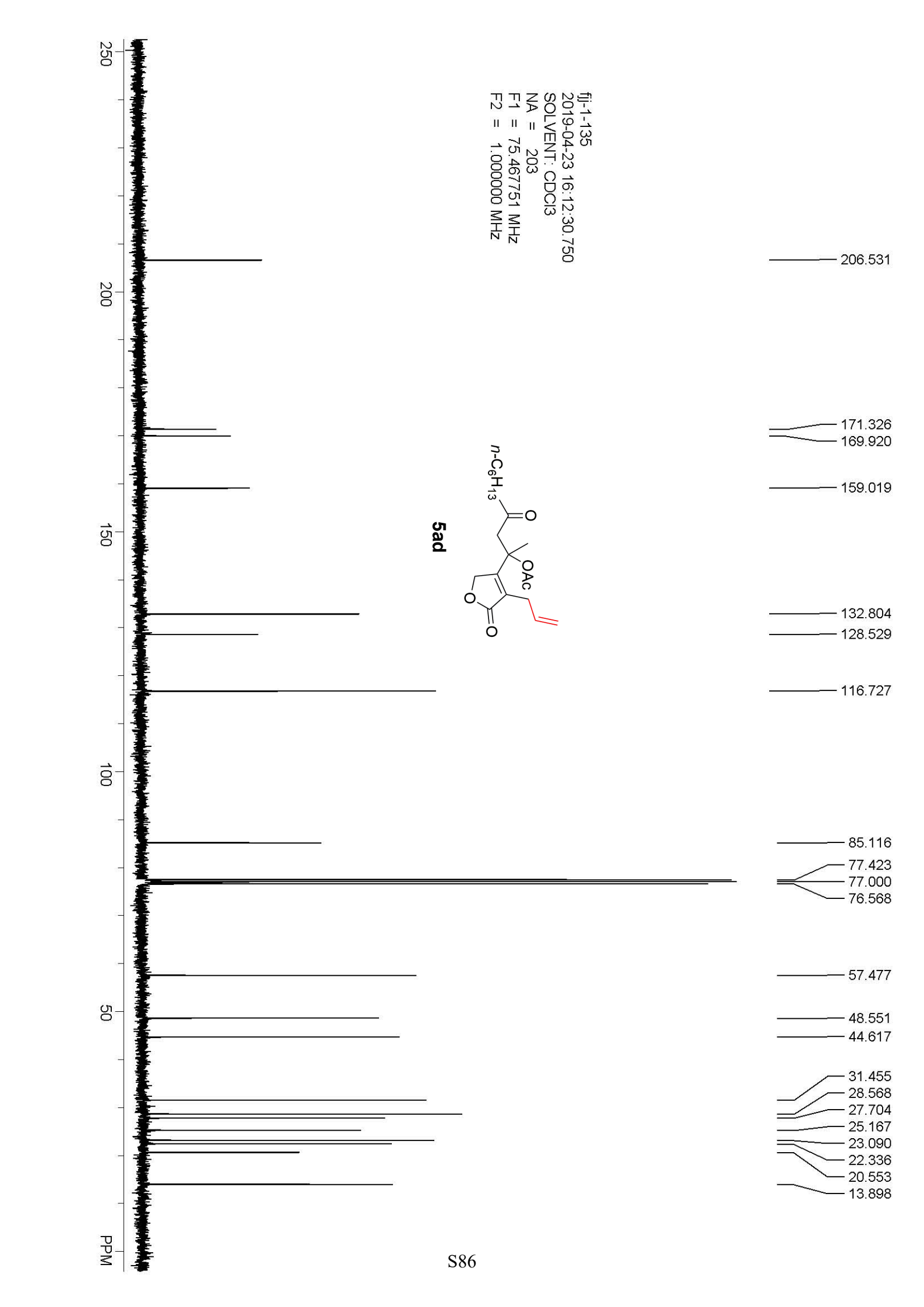

7.849
7.843
7.765
7.561
7.557
7.554
7.534
7.531
7.528
7.438
7.411
7.385
7.283
5.043
4.996
4.980
4.933
3.723
3.670
3.260
3.208
2.348
2.324
2.297
2.017
1.636
1.511
1.489
1.462
1.435
1.411
1.384
1.360
1.335
1.313
1.285
1.260
0.947
0.923
0.899
-0.000
fjj-1-095
2019-03-21 17:53:46.500
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Cl
O
OAc C4H9-n
O
O
5kb
S81
1.00
1.00
1.01
1.01
2.01
1.01
0.99
2.05
2.94
3.04
2.13
2.29
3.23
10
8
6
4
2
PPM

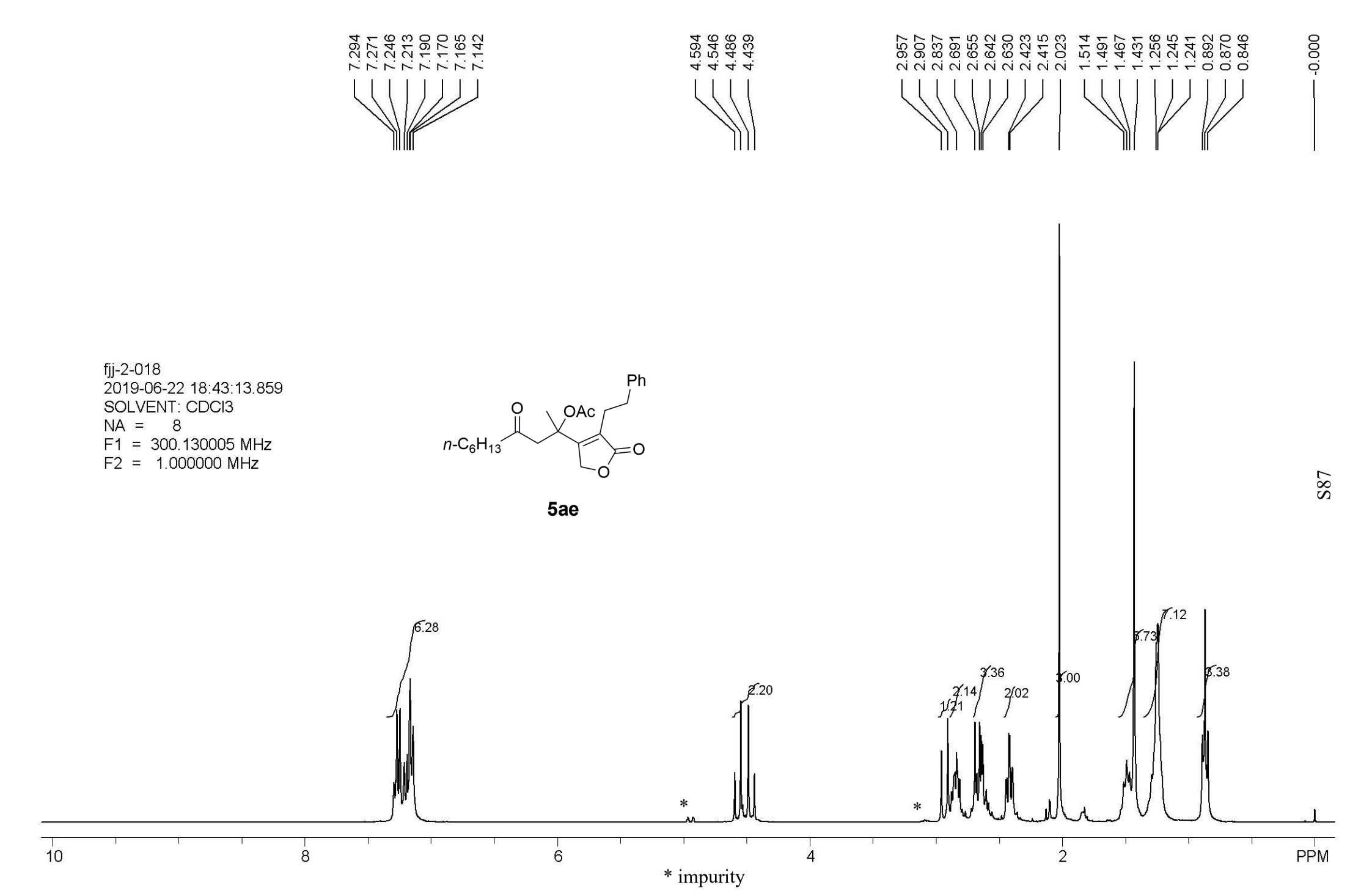

fji-1-095
2019-03-21 18:28:48.765
SOLVENT: CDCl3
NA = 508
F1 = 75.467751 MHz
F2 = 1.000000 MHz
5kb
Cl
O
OAc
C4H9-n
O
O
194.407
171.556
170.003
156.739
138.300
134.982
133.419
132.077
130.000
128.290
126.562
84.850
77.423
77.000
76.577
57.449
44.553
30.131
25.572
23.605
22.428
20.525
13.751
250
200
150
100
50
PPM

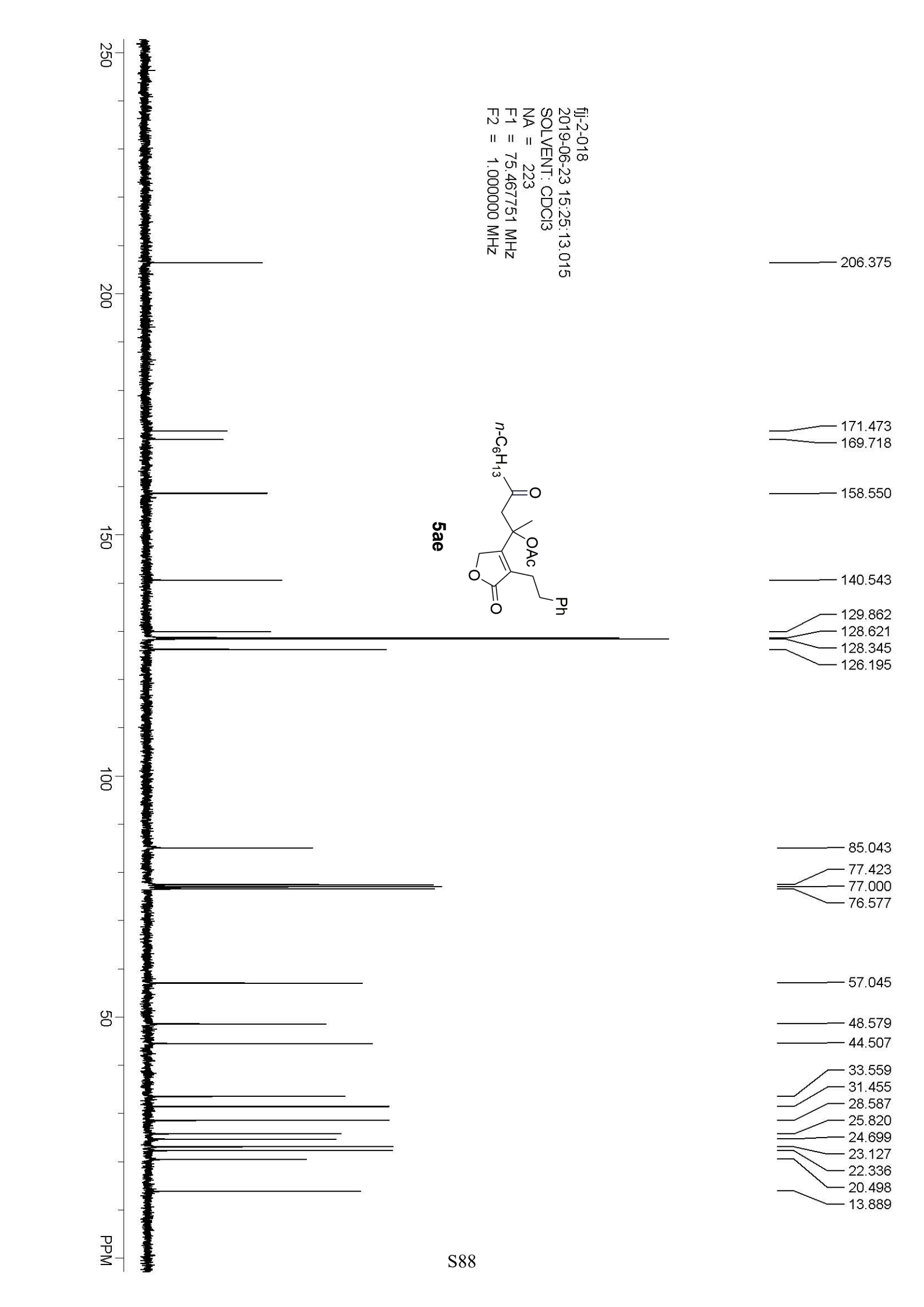

7.744
7.731
7.675
7.659
7.315
7.146
7.132
7.116
5.058
5.010
5.003
4.955
3.656
3.606
3.212
3.162
2.330
2.306
2.280
2.041
1.491
1.469
1.439
1.416
1.389
1.362
1.338
1.313
1.290
1.266
1.242
0.929
0.904
0.881
-0.000
fjj-1-090
2019-03-20 14:25:16.140
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
O
OAc
C4H9-n
O
S
O
5lb
S83
1.00
1.01
1.00
2.04
1.01
1.01
2.07
3.06
3.08
2.11
2.33
3.19
*
10
8
6
4
2
PPM
* Dichloromethane

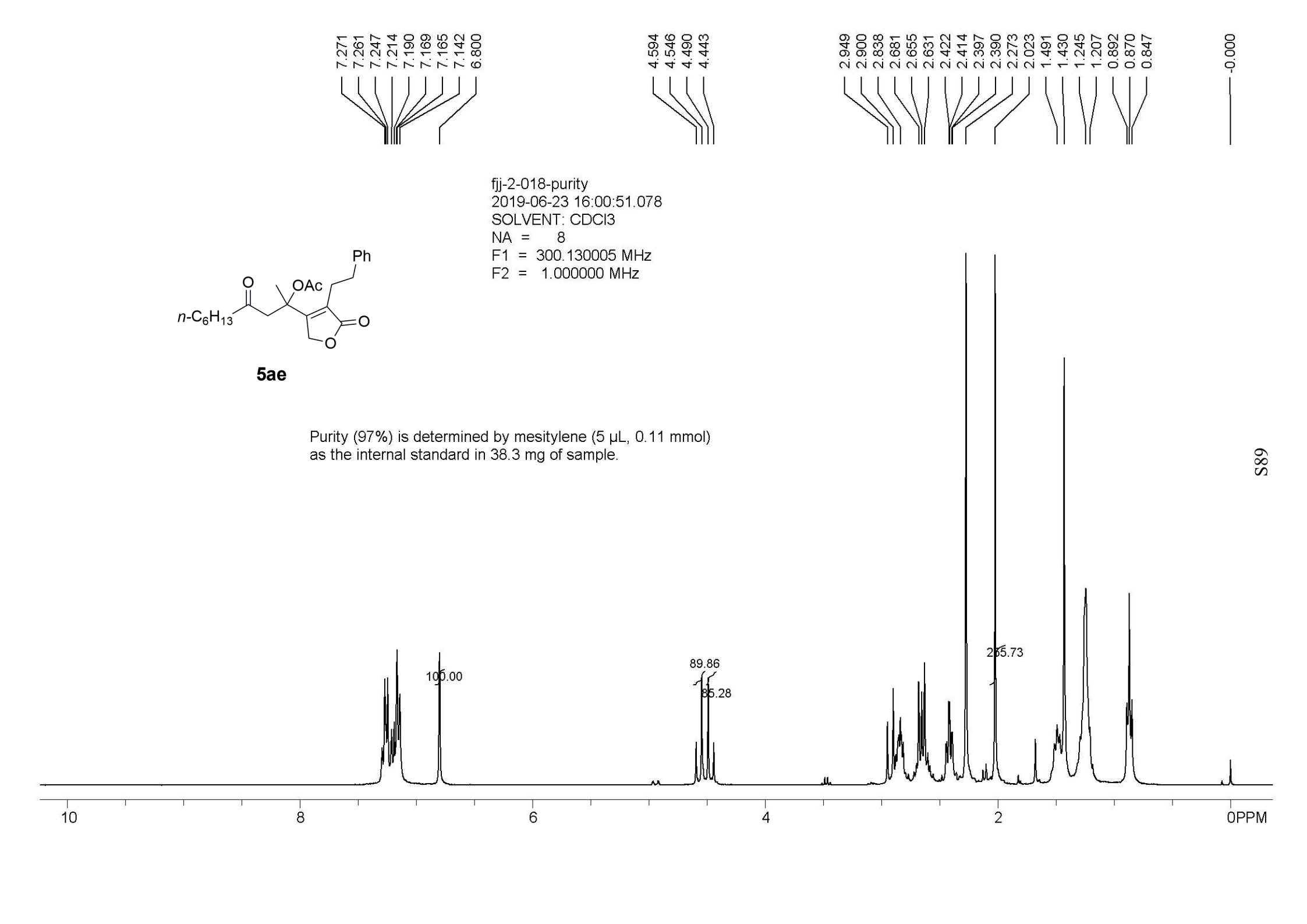

fiji-1-090
2019-03-20 14:29:36.953
SOLVENT: CDCl3
NA = 49
F1 = 75.467751 MHz
F2 = 1.000000 MHz
5lb
OAc
C4H9-n
188.074
171.501
169.911
156.996
144.091
134.807
133.410
131.655
128.309
84.887
77.432
77.000
76.577
57.431
45.739
29.984
25.278
23.430
22.263
20.406
13.650
250
200
150
100
50
PPM

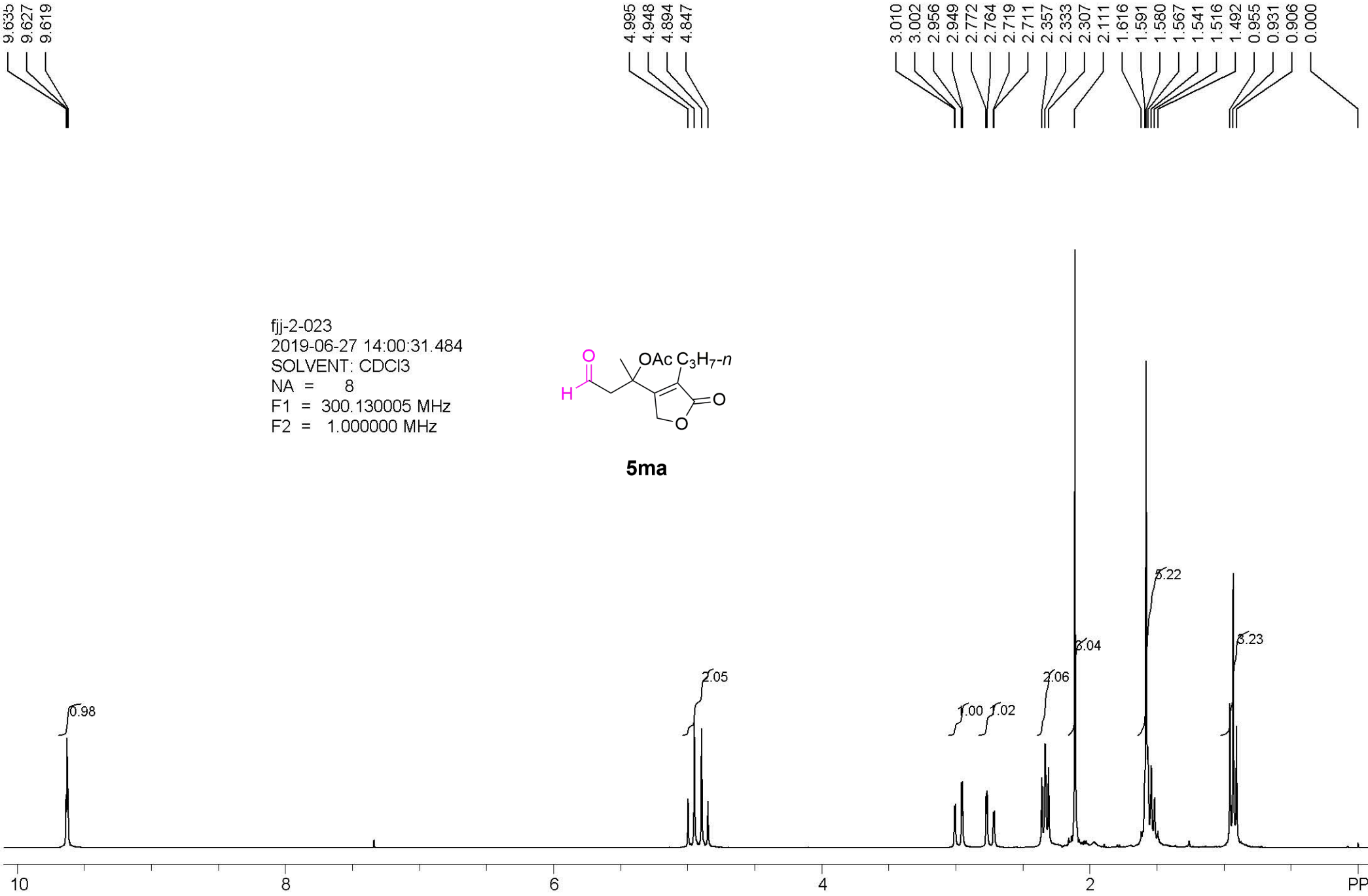

7.293
5.893
5.873
5.853
5.838
5.817
5.797
5.783
5.762
5.130
5.125
5.105
5.101
5.070
4.948
3.120
3.099
3.095
3.044
2.773
2.723
2.463
2.439
2.414
2.095
1.538
1.250
1.241
1.217
0.889
0.868
0.844
0.000
fjj-1-135
2019-04-23 15:58:59.609
SOLVENT: CDCl3
NA = 8
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
n-C6H13
OAc
O
O
O
5ad
S85
1.03
1.09
2.02
0.96
3.04
1.00
2.02
3.07
5.20
6.40
3.08
10
8
6
4
2
PPM

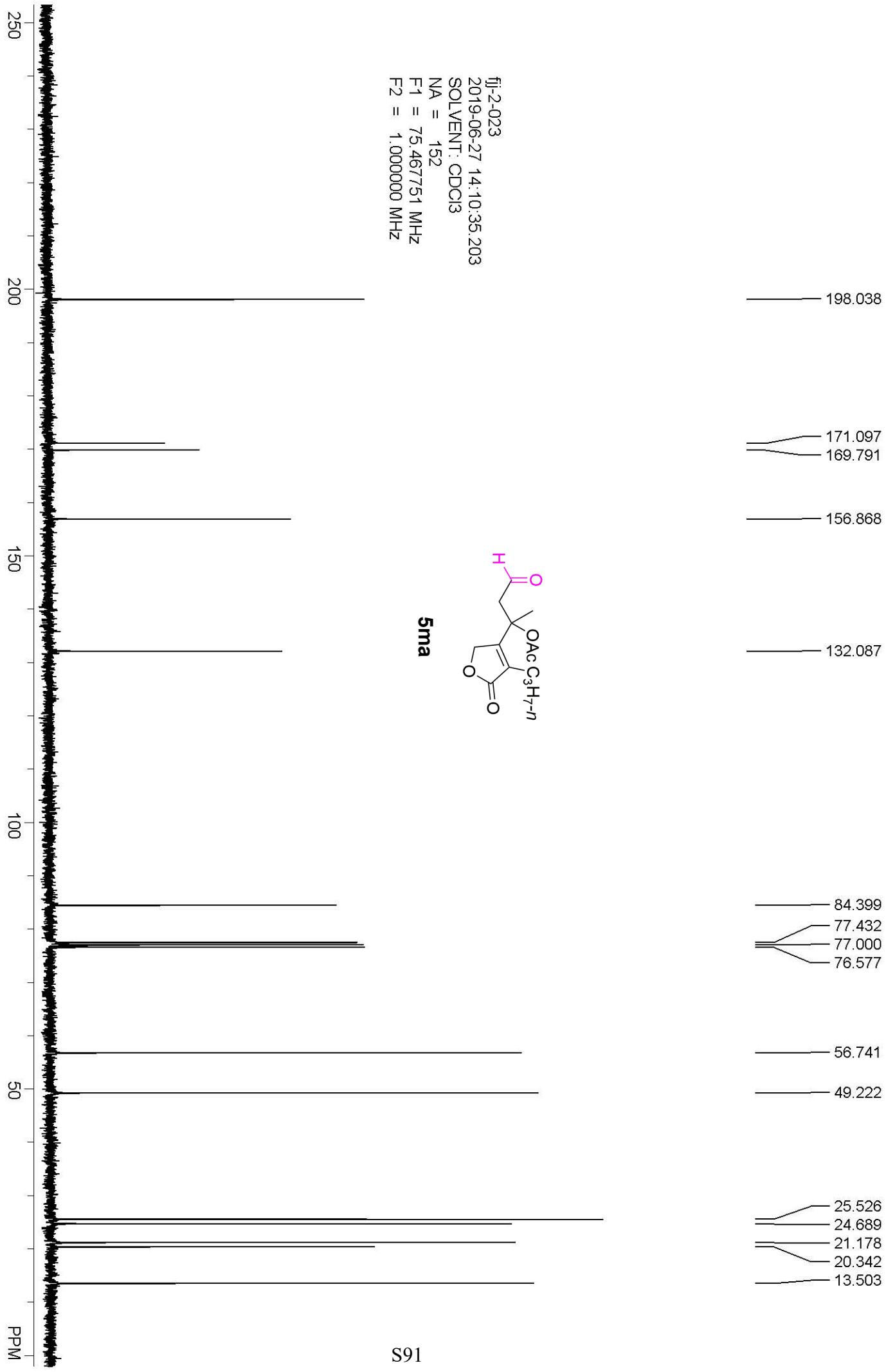

fjj-1-135
2019-04-23 16:12:30.750
SOLVENT: CDCl3
NA = 203
F1 = 75.467751 MHz
F2 = 1.000000 MHz
5ad
n-C6H13
O
OAc
O
O
206.531
171.326
169.920
159.019
132.804
128.529
116.727
85.116
77.423
77.000
76.568
57.477
48.551
44.617
31.455
28.568
27.704
25.167
23.090
22.336
20.553
13.898
250
200
150
100
50
PPM

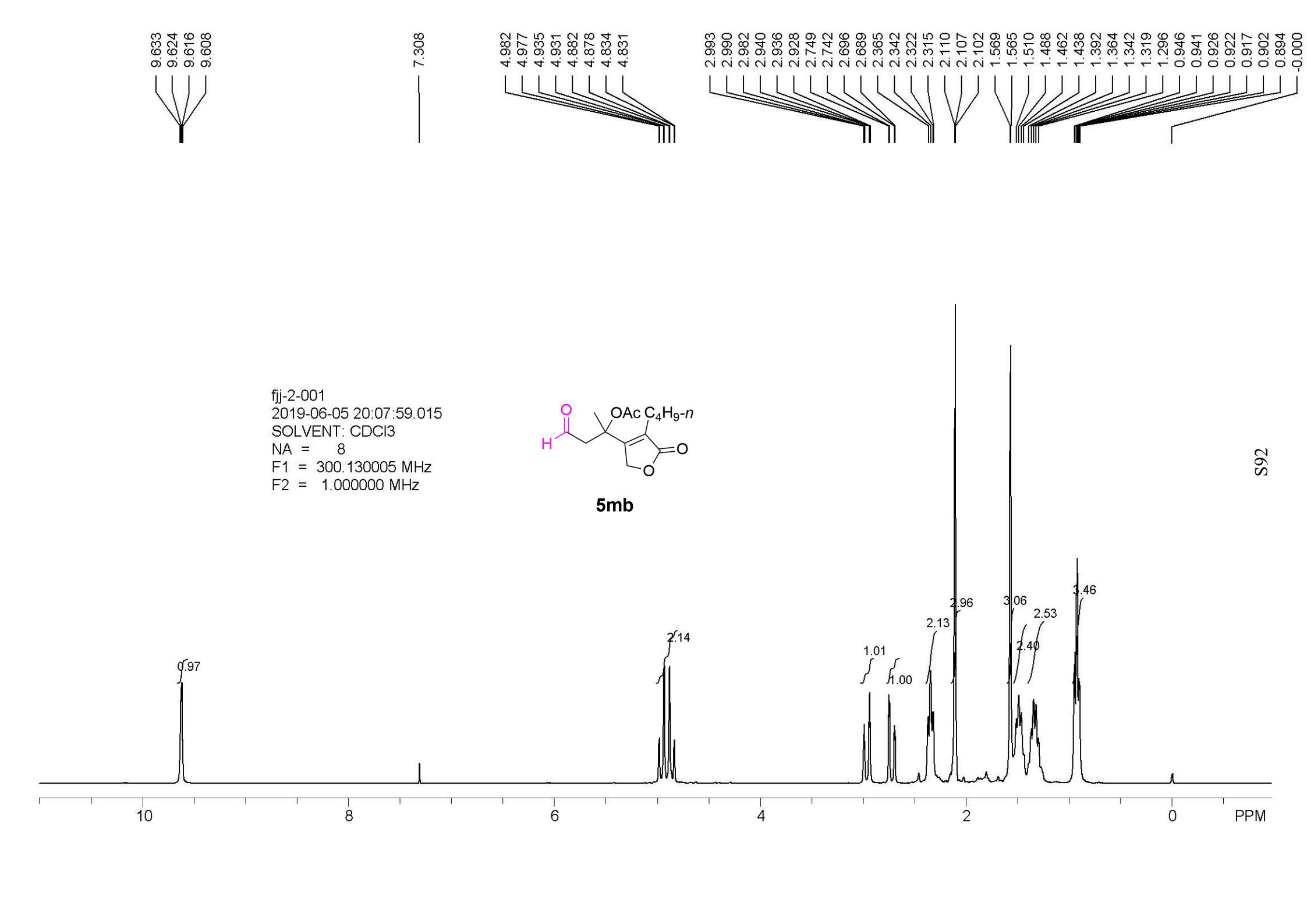

7.294
7.271
7.246
7.213
7.190
7.170
7.165
7.142
4.594
4.546
4.486
4.439
2.957
2.907
2.837
2.691
2.655
2.642
2.630
2.423
2.415
2.023
1.514
1.491
1.467
1.431
1.256
1.245
1.241
0.892
0.870
0.846
-0.000
fjj-2-018
2019-06-22 18:43:13.859
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
OAc
O
O
O
n-C6H13
5ae
S87
6.28
2.20
1.21
2.14
3.36
2.02
3.00
6.73
7.12
3.38
*
*
10
8
6
4
2
PPM
* impurity

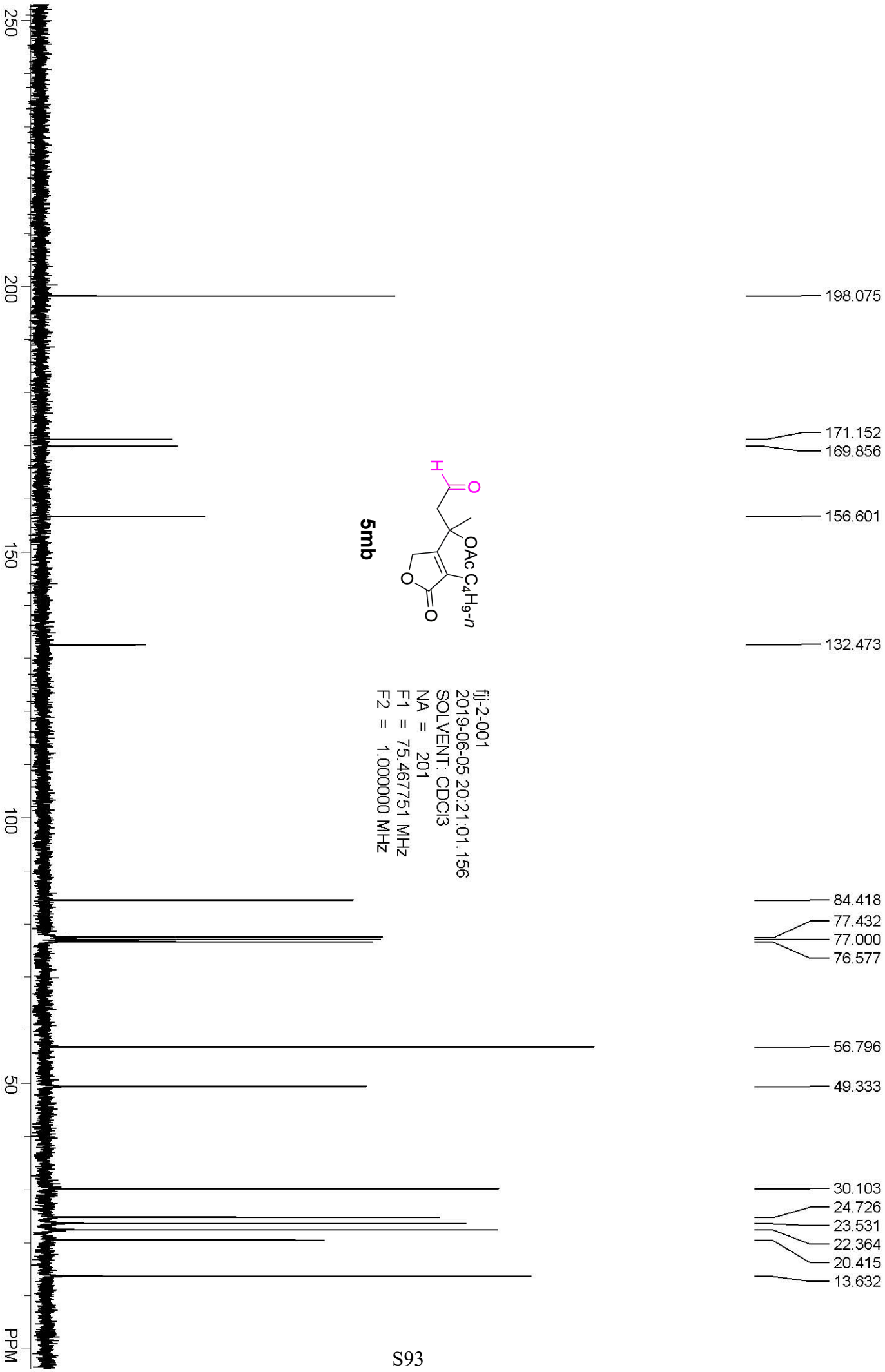

fjj-2-018
2019-06-23 15:25:13.015
SOLVENT: CDCl3
NA = 223
F1 = 75.467751 MHz
F2 = 1.000000 MHz
5ae
206.375
171.473
169.718
158.550
140.543
129.862
128.621
128.345
126.195
85.043
77.423
77.000
76.577
57.045
48.579
44.507
33.559
31.455
28.587
25.820
24.699
23.127
22.336
20.498
13.889
PPM

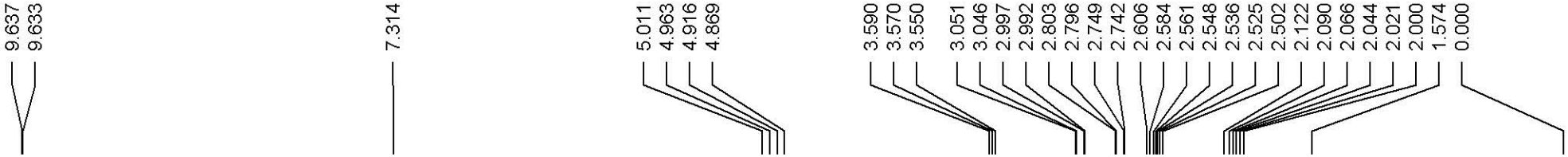

7.271
7.261
7.247
7.214
7.190
7.169
7.165
7.142
6.800
4.594
4.546
4.490
4.443
2.949
2.900
2.838
2.681
2.655
2.631
2.422
2.414
2.397
2.390
2.273
2.023
1.491
1.430
1.245
1.207
0.892
0.870
0.847
-0.000
fjj-2-018-purity
2019-06-23 16:00:51.078
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
O
OAc
n-C6H13
O
O
5ae
Purity (97%) is determined by mesitylene (5 μL, 0.11 mmol)
as the internal standard in 38.3 mg of sample.
100.00
89.86
85.28
265.73
10
8
6
4
2
0PPM


9.635
9.627
9.619
4.995
4.948
4.894
4.847
3.010
3.002
2.956
2.949
2.772
2.764
2.719
2.711
2.357
2.333
2.307
2.111
1.616
1.591
1.580
1.567
1.541
1.516
1.492
0.955
0.931
0.906
0.000
fjj-2-023
2019-06-27 14:00:31.484
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
O
H
OAc C3H7-n
O
O
5ma
0.98
2.05
1.00
1.02
2.06
3.04
3.22
3.23
10
8
6
4
2
PPM

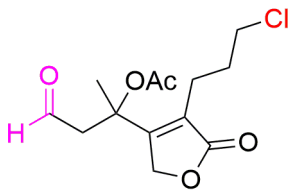

fji-2-023
2019-06-27 14:10:35.203
SOLVENT: CDCl3
NA = 152
F1 = 75.467751 MHz
F2 = 1.000000 MHz
5ma
OAc
C3H7-n
198.038
171.097
169.791
156.868
132.087
84.399
77.432
77.000
76.577
56.741
49.222
25.526
24.689
21.178
20.342
13.503
250
200
150
100
50
PPM


9.633
9.624
9.616
9.608
7.308
4.982
4.977
4.935
4.931
4.882
4.878
4.834
4.831
2.993
2.990
2.982
2.940
2.936
2.928
2.749
2.742
2.696
2.689
2.365
2.342
2.322
2.315
2.110
2.107
2.102
1.569
1.565
1.510
1.488
1.462
1.438
1.392
1.364
1.342
1.319
1.296
0.946
0.941
0.926
0.922
0.917
0.902
0.894
-0.000
fjj-2-001
2019-06-05 20:07:59.015
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
OAc C4H9-n
5mb
S92
0.97
2.14
1.01
1.00
2.13
2.96
3.06
2.53
2.40
3.46
10
8
6
4
2
0
PPM


198.075
171.152
169.856
156.601
132.473
84.418
77.432
77.000
76.577
56.796
49.333
30.103
24.726
23.531
22.364
20.415
13.632
250
200
150
100
50
PPM
5mb
OAc
C_4H_9-n
fij-2-001
2019-06-05 20:21:01.156
SOLVENT: CDCl3
NA = 201
F1 = 75.467751 MHz
F2 = 1.000000 MHz

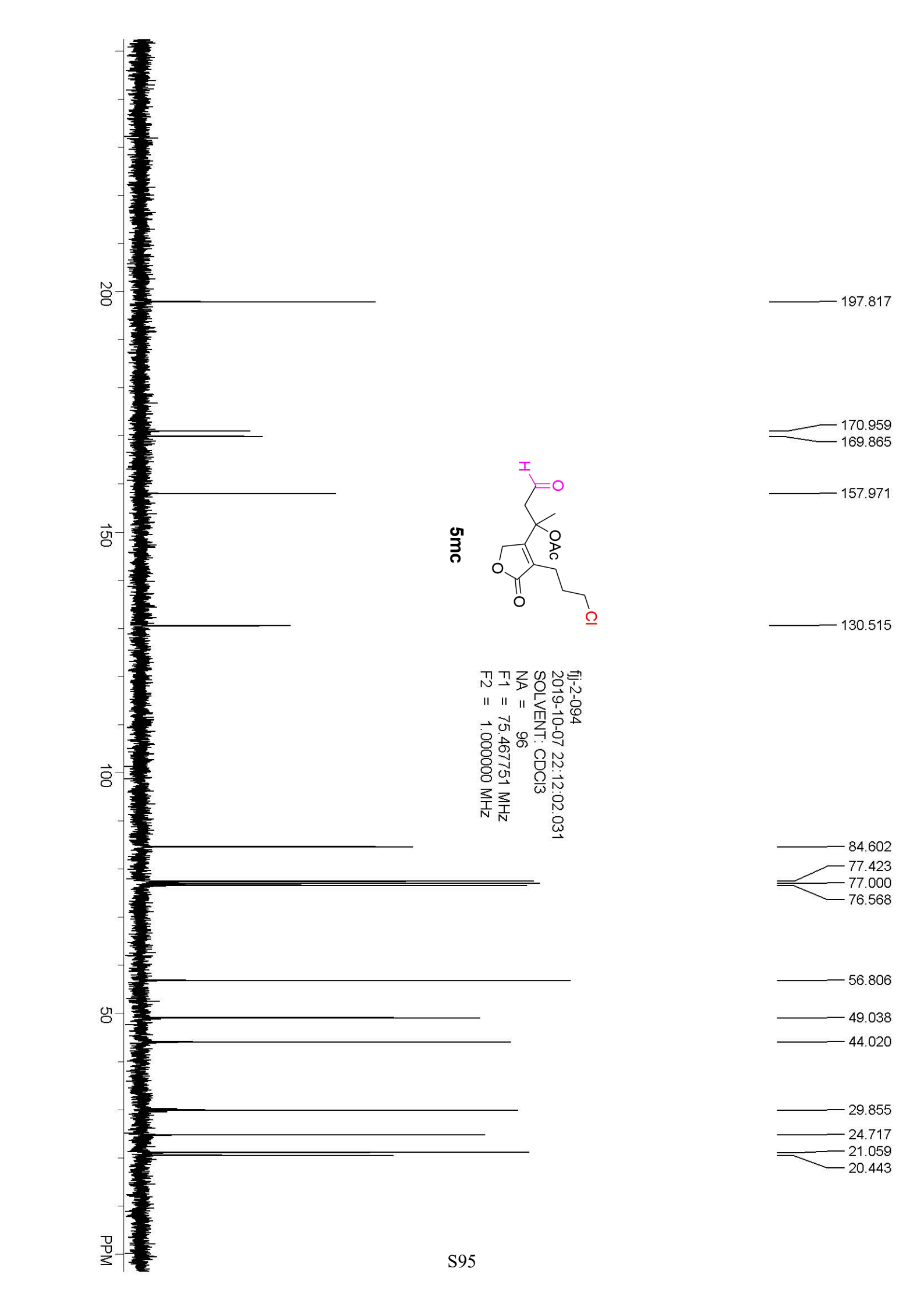


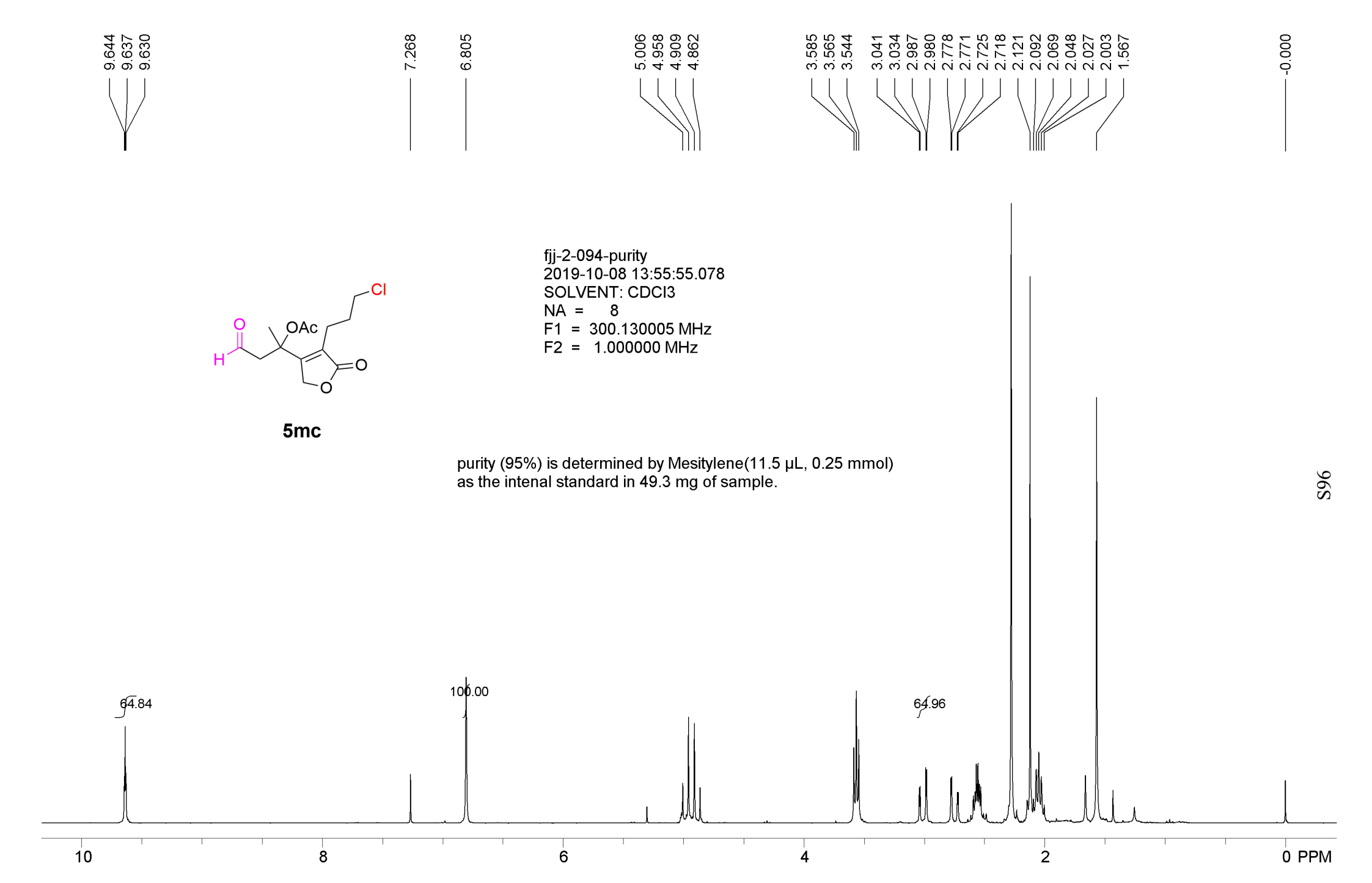
fjj-2-094
2019-10-07 22:04:30.703
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz

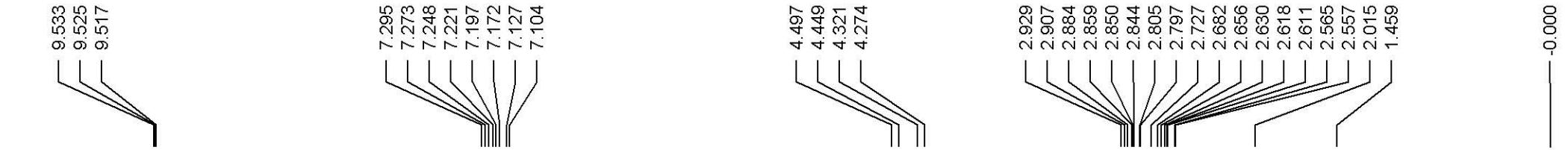


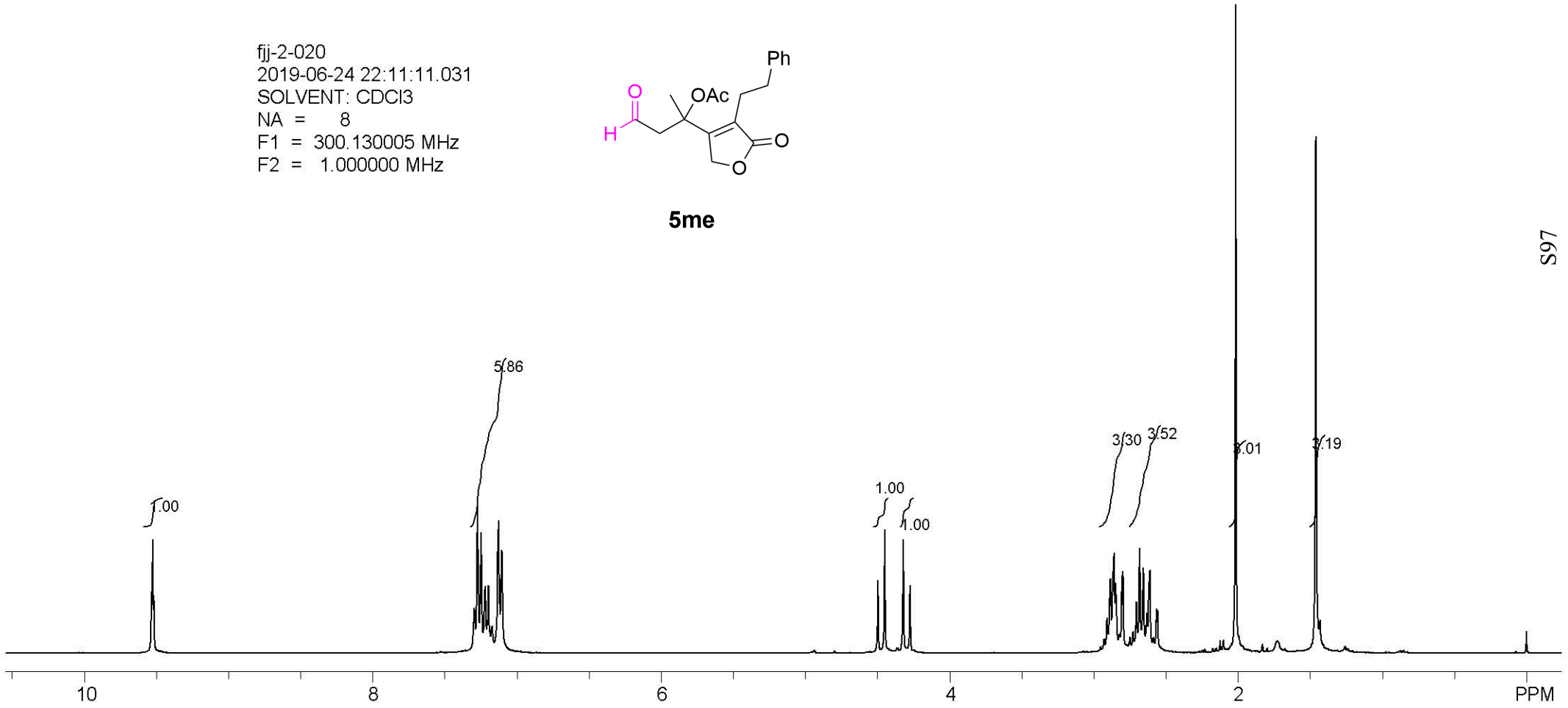
5mc

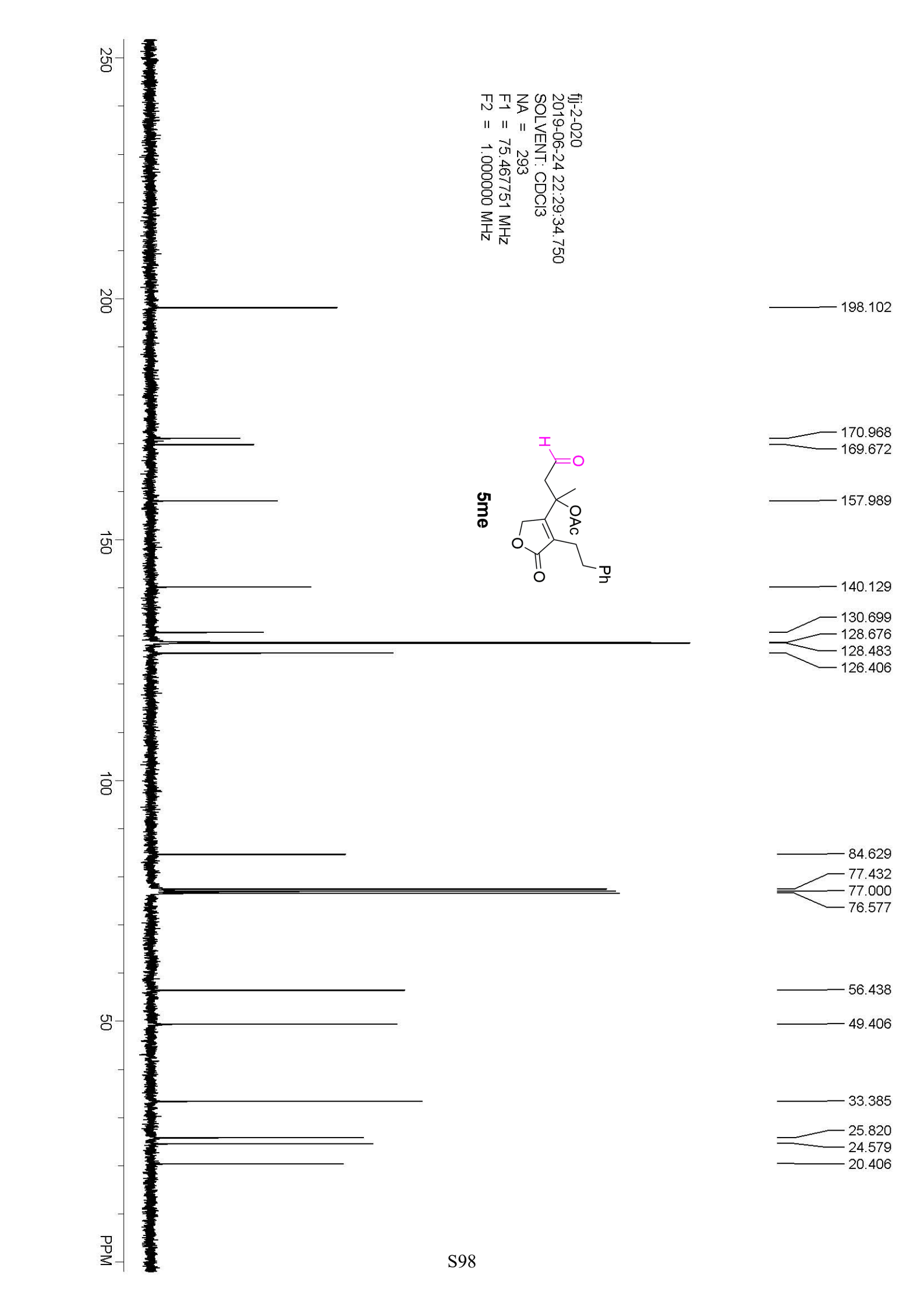
* impurity

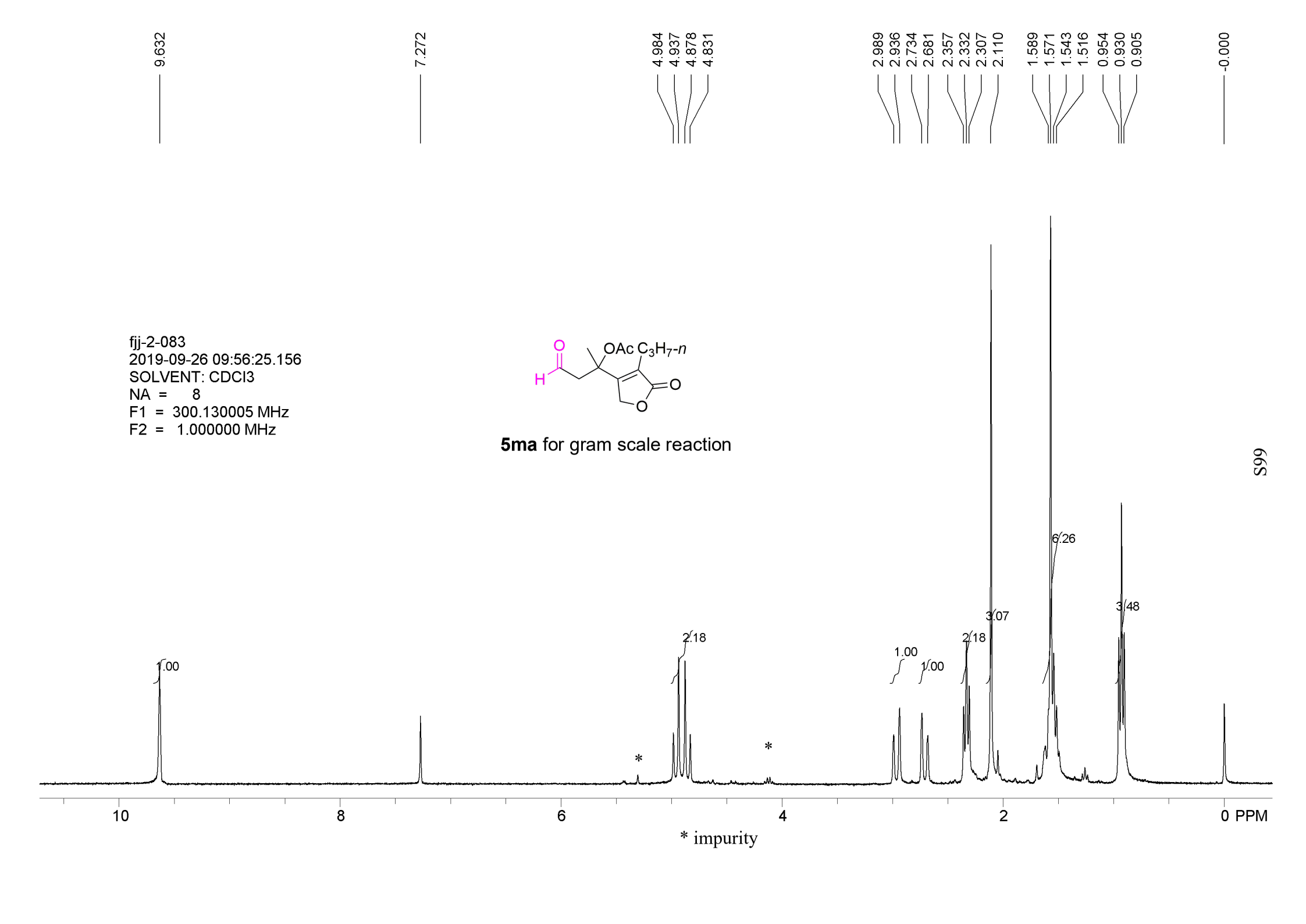

197.817
170.959
169.865
157.971
130.515
84.602
77.423
77.000
76.568
56.806
49.038
44.020
29.855
24.717
21.059
20.443
200
150
100
50
PPM
5mc
O
H
OAc
O
O
Cl
fjj-2-094
2019-10-07 22:12:02.031
SOLVENT: CDCl3
NA = 96
F1 = 75.467751 MHz
F2 = 1.000000 MHz

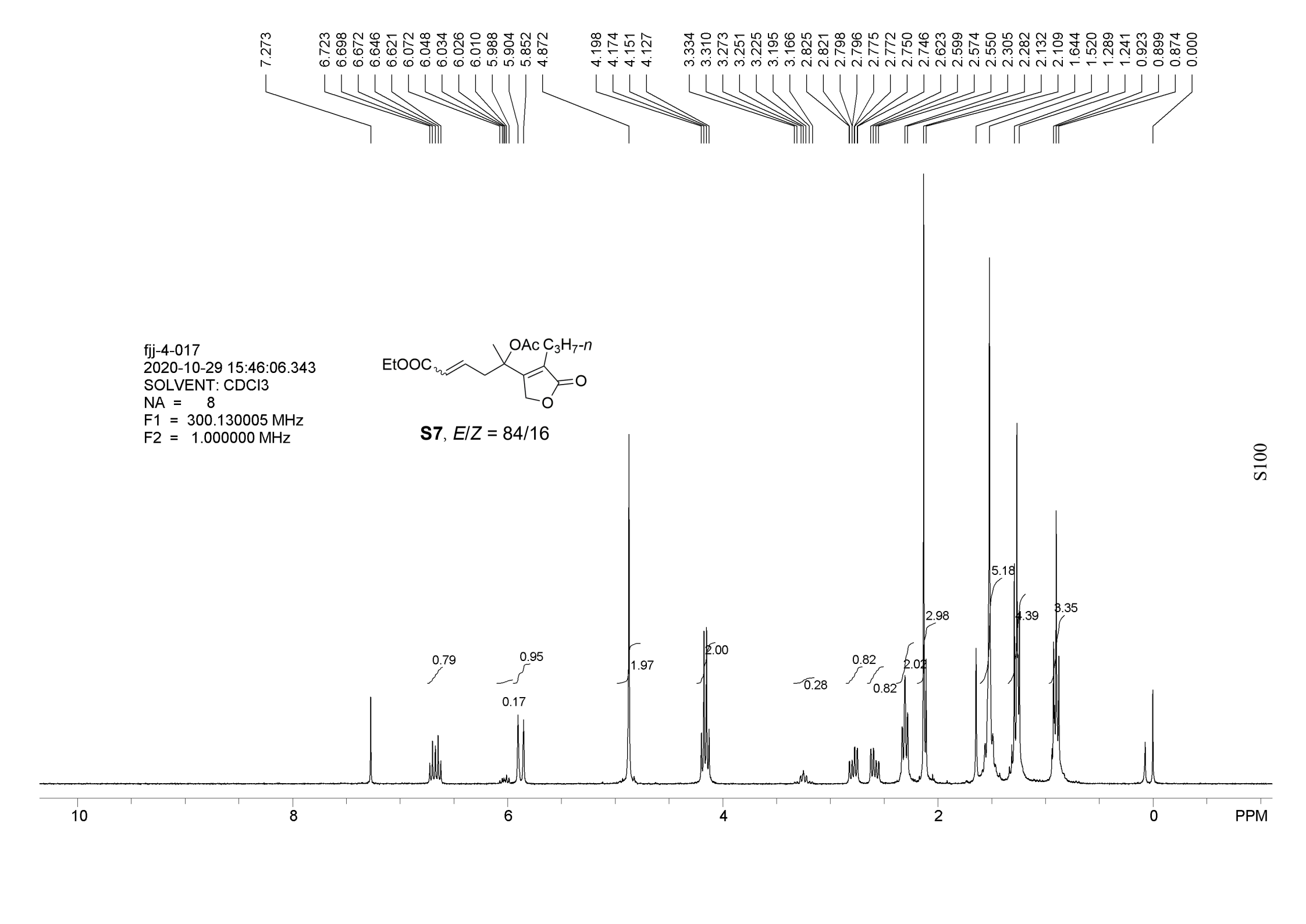

9.644
9.637
9.630
7.268
6.805
5.006
4.958
4.909
4.862
3.585
3.565
3.544
3.041
3.034
2.987
2.980
2.778
2.771
2.725
2.718
2.121
2.092
2.069
2.048
2.027
2.003
1.567
-0.000
Cl
OAc
O
H
O
O
fjj-2-094-purity
2019-10-08 13:55:55.078
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
5mc
purity (95%) is determined by Mesitylene(11.5 μL, 0.25 mmol)
as the intenal standard in 49.3 mg of sample.
64.84
100.00
64.96
10
8
6
4
2
0 PPM

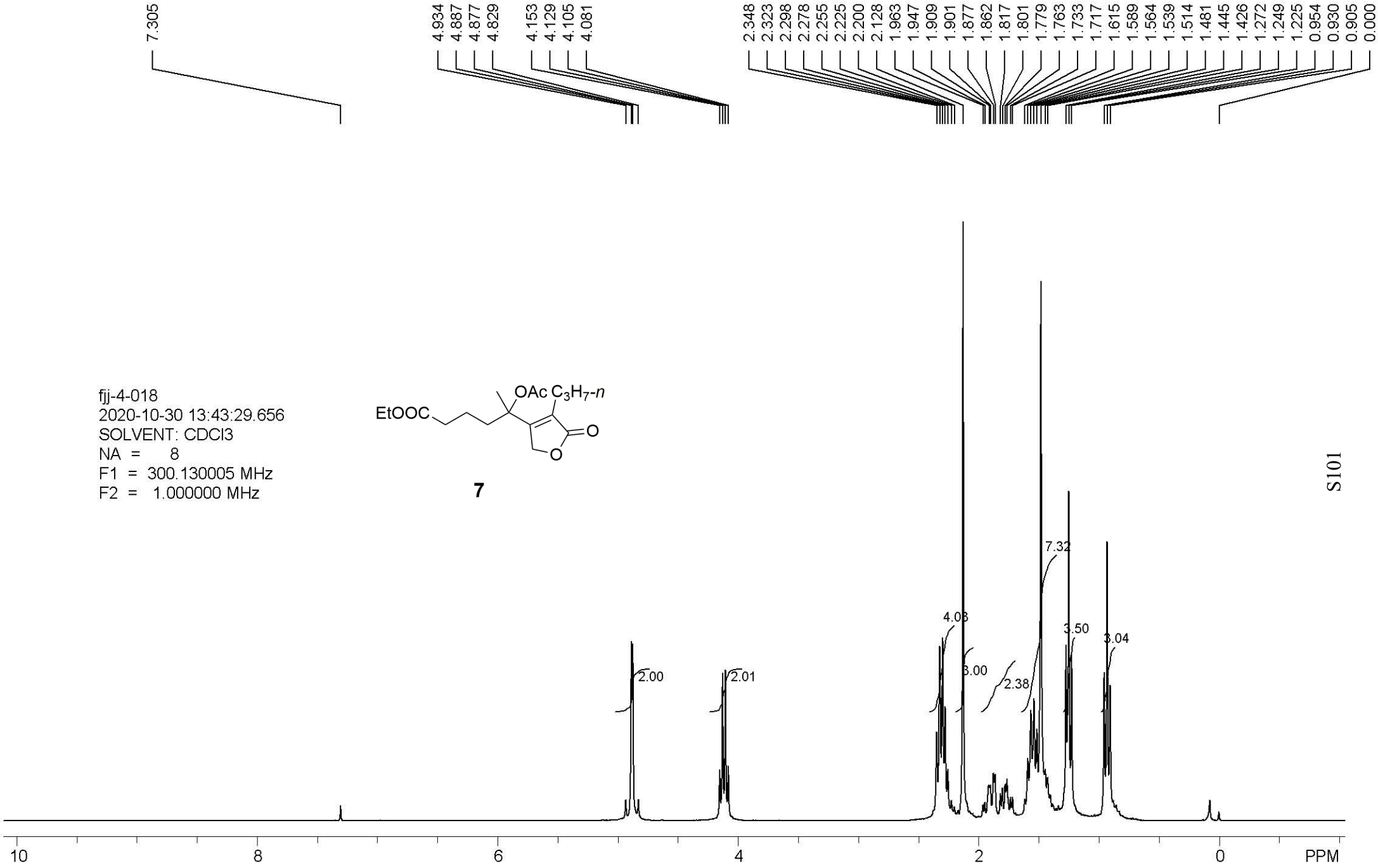

9.533
9.525
9.517
7.295
7.273
7.248
7.221
7.197
7.172
7.127
7.104
4.497
4.449
4.321
4.274
2.929
2.907
2.884
2.859
2.850
2.844
2.805
2.797
2.727
2.682
2.656
2.630
2.618
2.611
2.565
2.557
2.015
1.459
-0.000

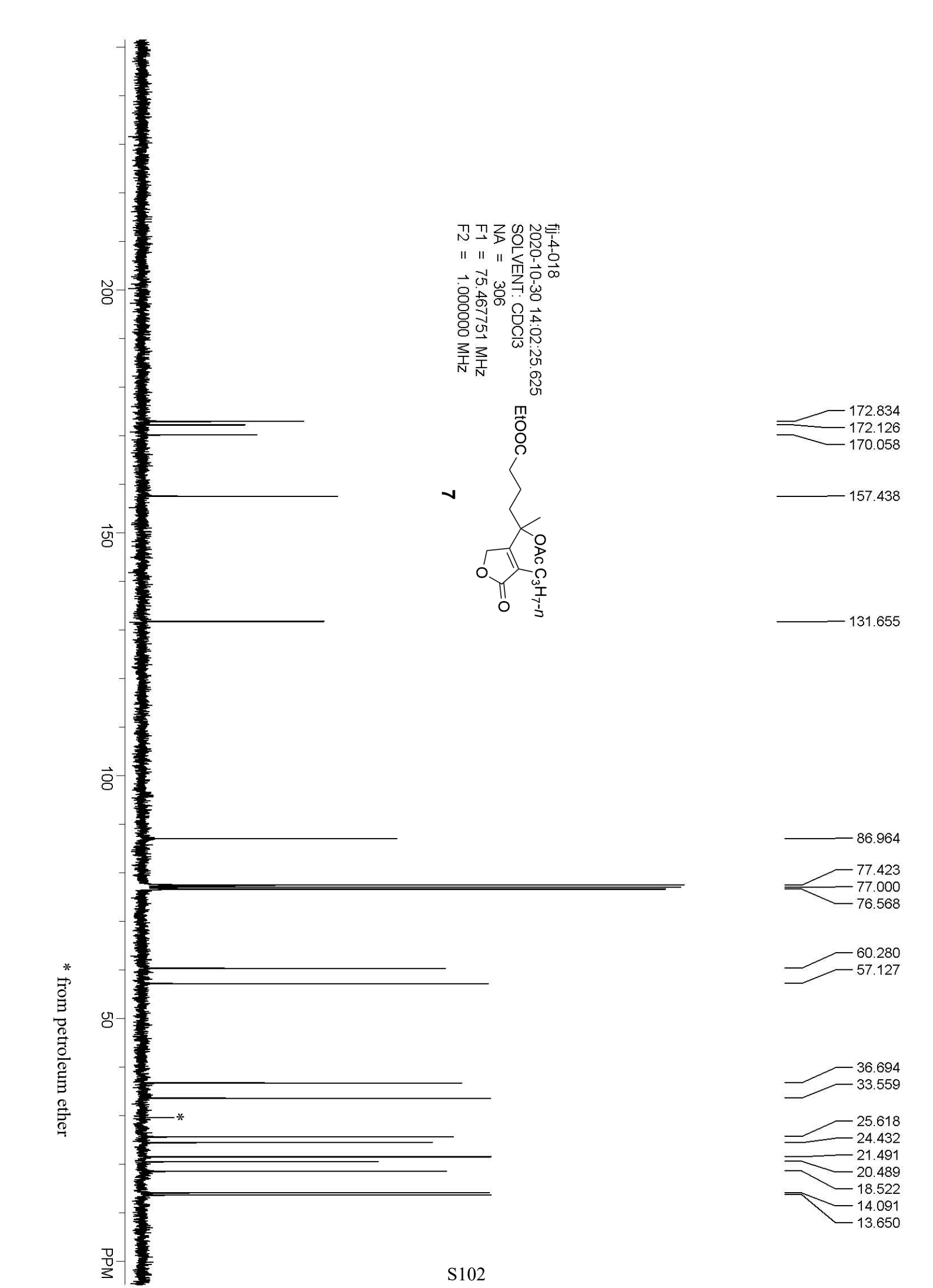

fjj-2-020
2019-06-24 22:11:11.031
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
O
OAc
H
O
O
5me
S97
1.00
5.86
1.00
1.00
3.30
3.52
3.01
3.19
10
8
6
4
2
PPM


fjj-2-020
2019-06-24 22:29:34.750
SOLVENT: CDCl3
NA = 293
F1 = 75.467751 MHz
F2 = 1.000000 MHz
5me
198.102
170.968
169.672
157.989
140.129
130.699
128.676
128.483
126.406
84.629
77.432
77.000
76.577
56.438
49.406
33.385
25.820
24.579
20.406
250
200
150
100
50
PPM

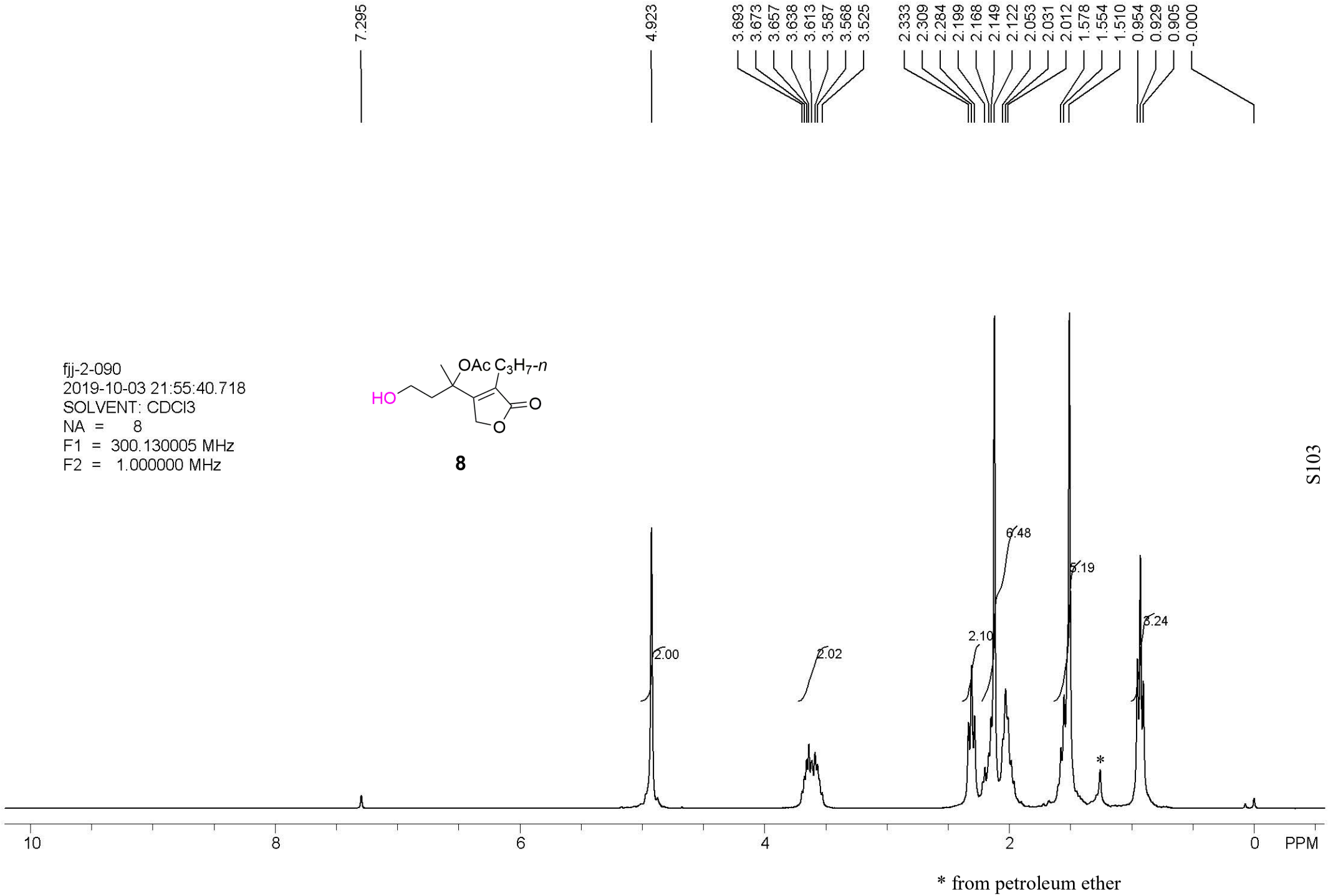

9.632
7.272
4.984
4.937
4.878
4.831
2.989
2.936
2.734
2.681
2.357
2.332
2.307
2.110
1.589
1.571
1.543
1.516
0.954
0.930
0.905
-0.000
fjj-2-083
2019-09-26 09:56:25.156
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
OAc C3H7-n
H
O
O
O
5ma for gram scale reaction
S99
1.00
2.18
1.00
1.00
2.18
3.07
6.26
3.48
*
*
10
8
6
4
2
0 PPM
* impurity

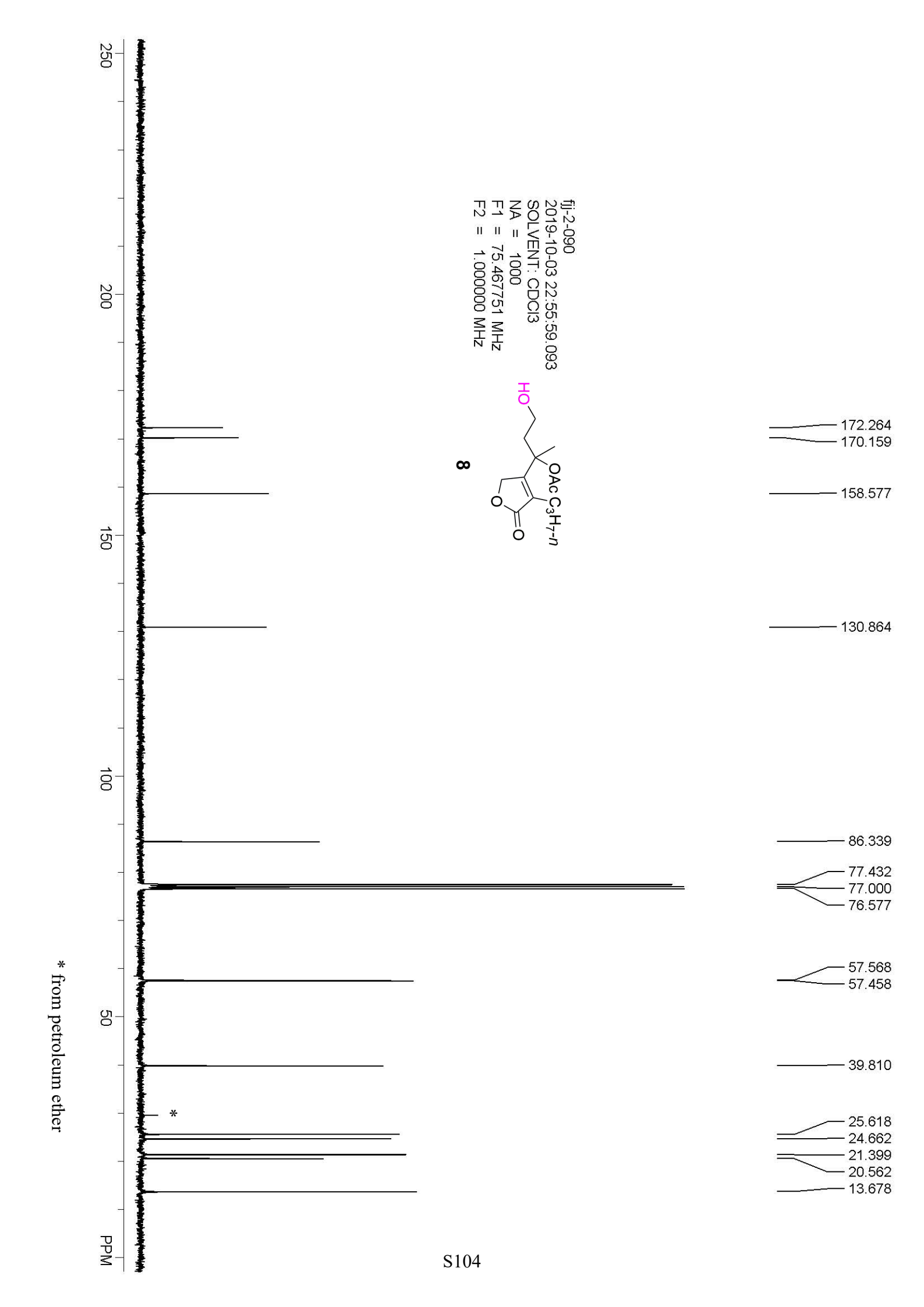

7.273
6.723
6.698
6.672
6.646
6.621
6.072
6.048
6.034
6.026
6.010
5.988
5.904
5.852
4.872
4.198
4.174
4.151
4.127
3.334
3.310
3.273
3.251
3.225
3.195
3.166
2.825
2.821
2.798
2.796
2.775
2.772
2.750
2.746
2.623
2.599
2.574
2.550
2.305
2.282
2.132
2.109
1.644
1.520
1.289
1.241
0.923
0.899
0.874
0.000
fjj-4-017
2020-10-29 15:46:06.343
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
EtOOC
OAc
C3H7-n
O
O
S7, E/Z = 84/16
0.79
0.95
0.17
1.97
2.00
0.28
0.82
0.82
2.02
2.98
5.18
4.39
3.35
10
8
6
4
2
0
PPM
S100


7.305
4.934
4.887
4.877
4.829
4.153
4.129
4.105
4.081
2.348
2.323
2.298
2.278
2.255
2.225
2.200
2.128
1.963
1.947
1.909
1.901
1.877
1.862
1.817
1.801
1.779
1.763
1.733
1.717
1.615
1.589
1.564
1.539
1.514
1.481
1.445
1.426
1.272
1.249
1.225
0.954
0.930
0.905
0.000
fjj-4-018
2020-10-30 13:43:29.656
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
EtOOC
OAc
C_3H_7-*n*
O
O
7
S101
2.00
2.01
4.08
3.00
2.38
7.32
3.50
3.04
10
8
6
4
2
0
PPM

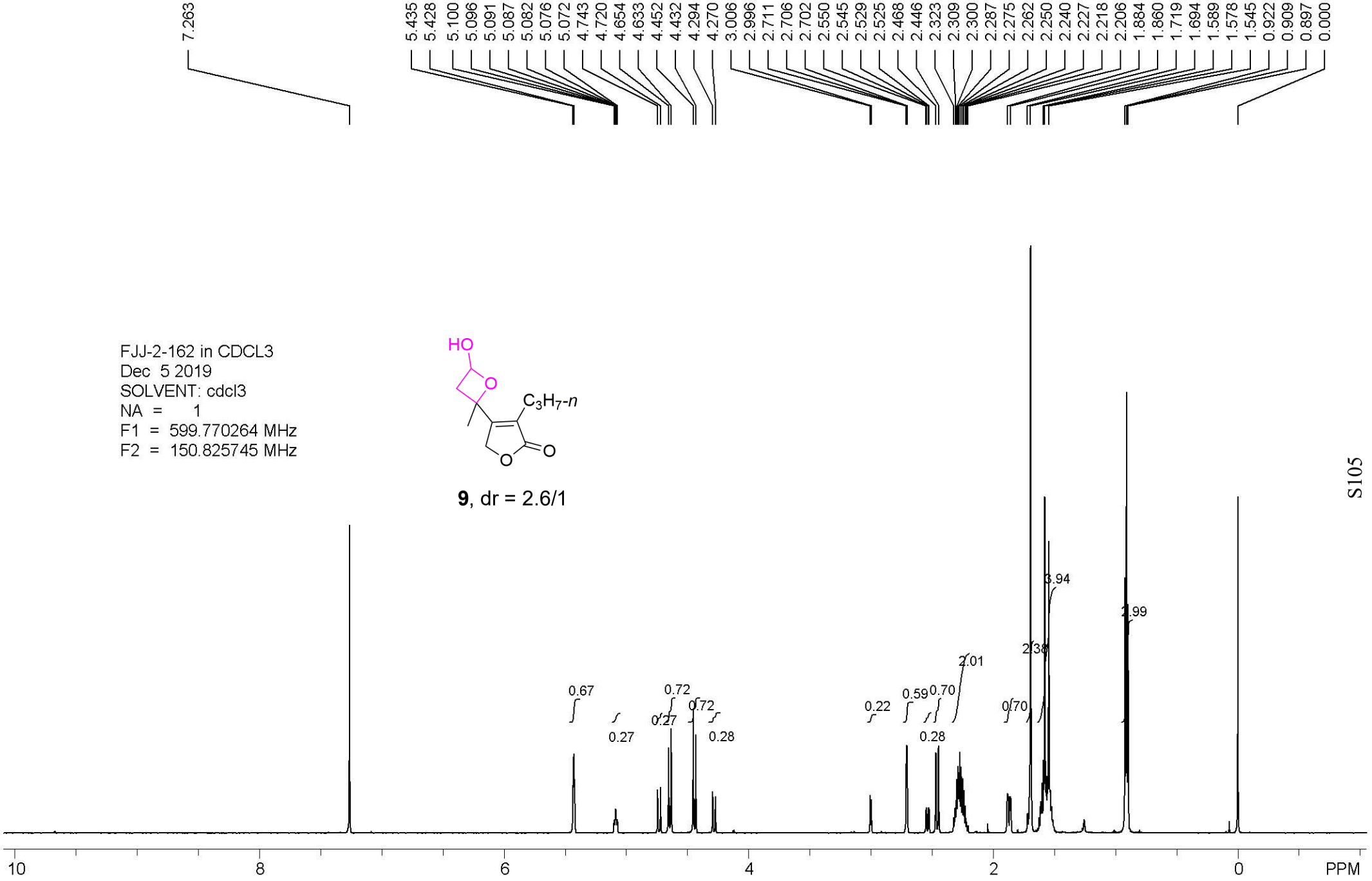


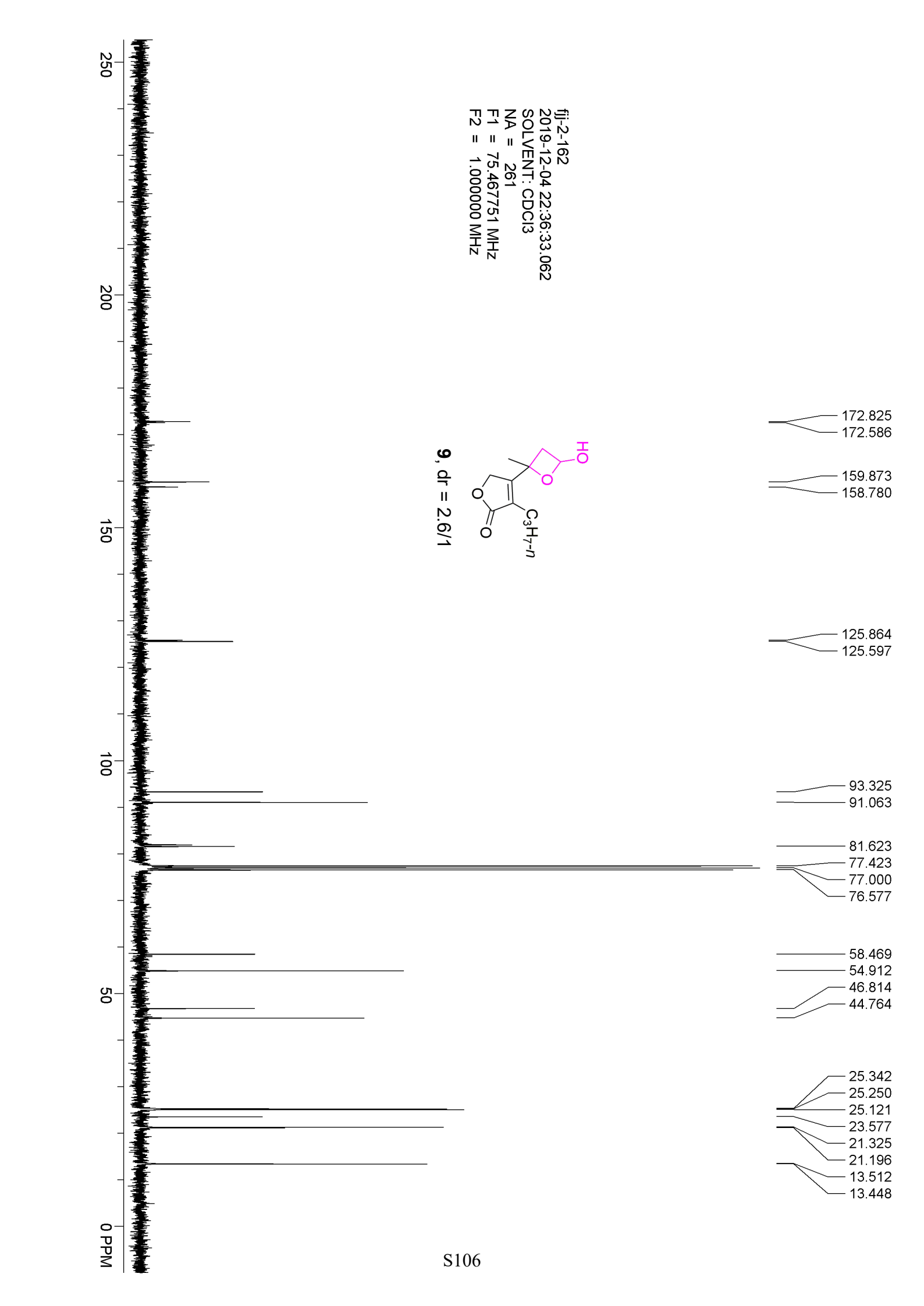
* from petroleum ether

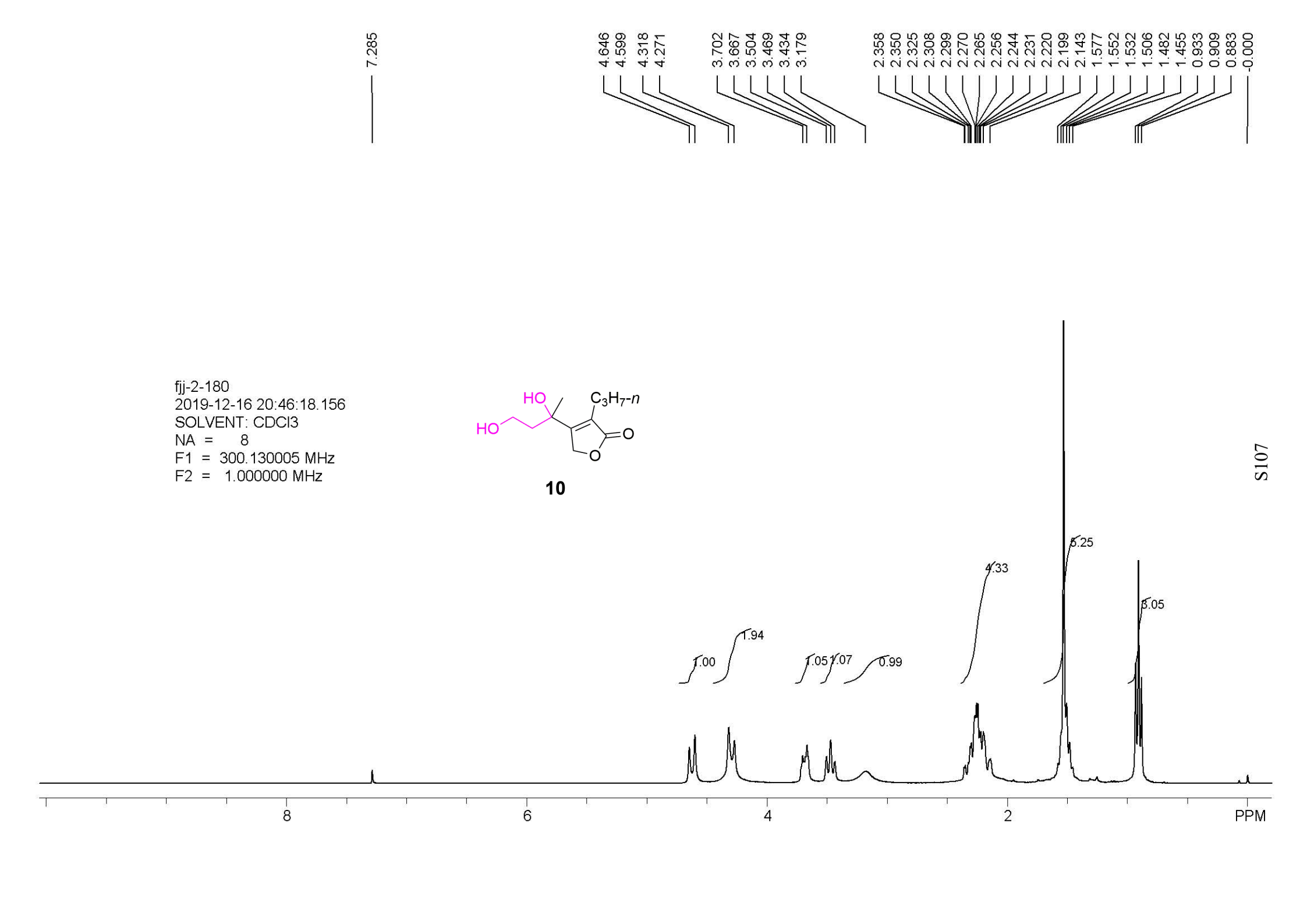

7.295
4.923
3.693
3.673
3.657
3.638
3.613
3.587
3.568
3.525
2.333
2.309
2.284
2.199
2.168
2.149
2.122
2.053
2.031
2.012
1.578
1.554
1.510
0.954
0.929
0.905
-0.000
fjj-2-090
2019-10-03 21:55:40.718
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
HO
OAc
C_3H_7-*n*
O
O
8
2.00
2.02
2.10
6.48
5.19
3.24
*
10
8
6
4
2
0
PPM
* from petroleum ether

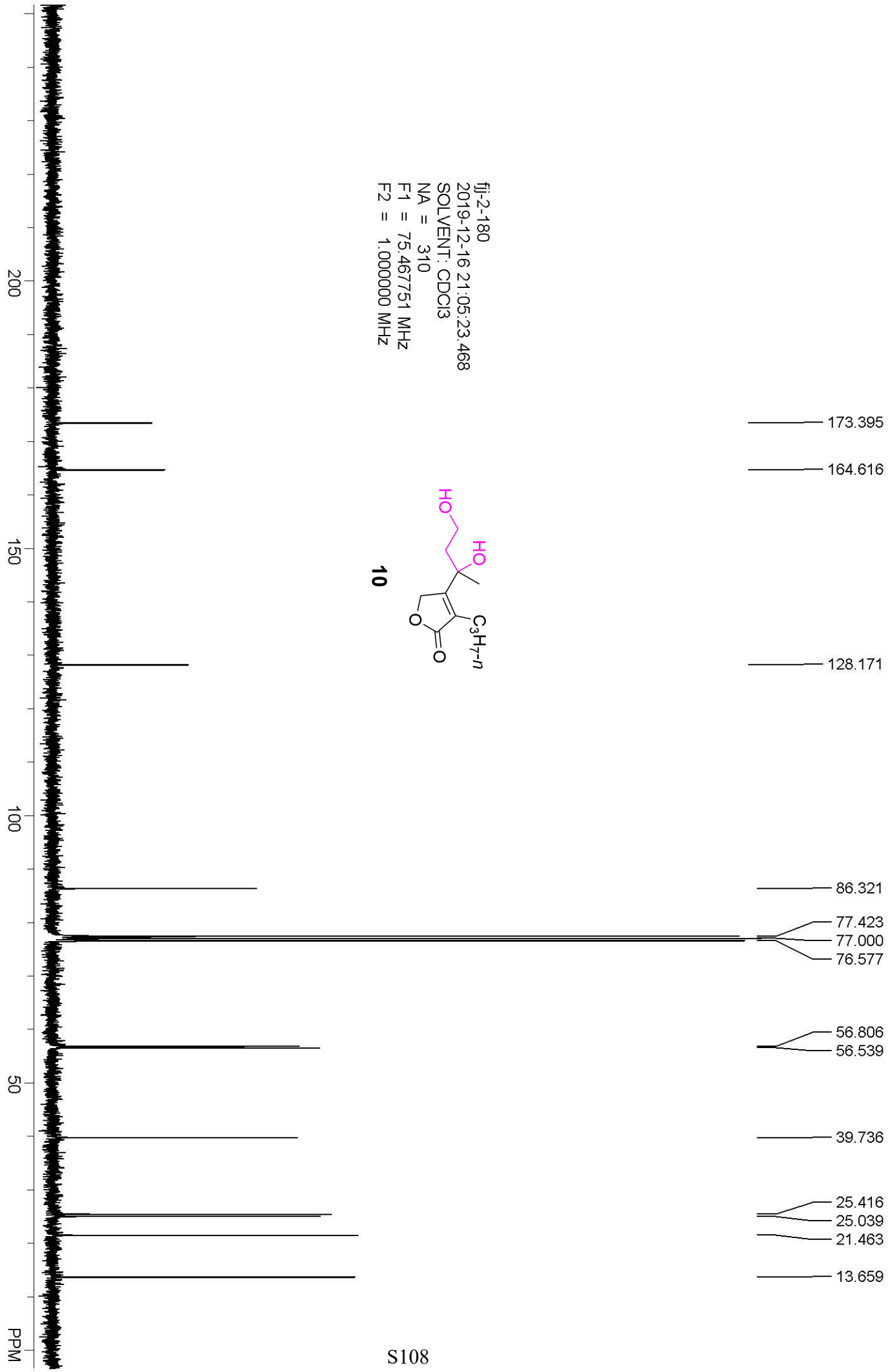


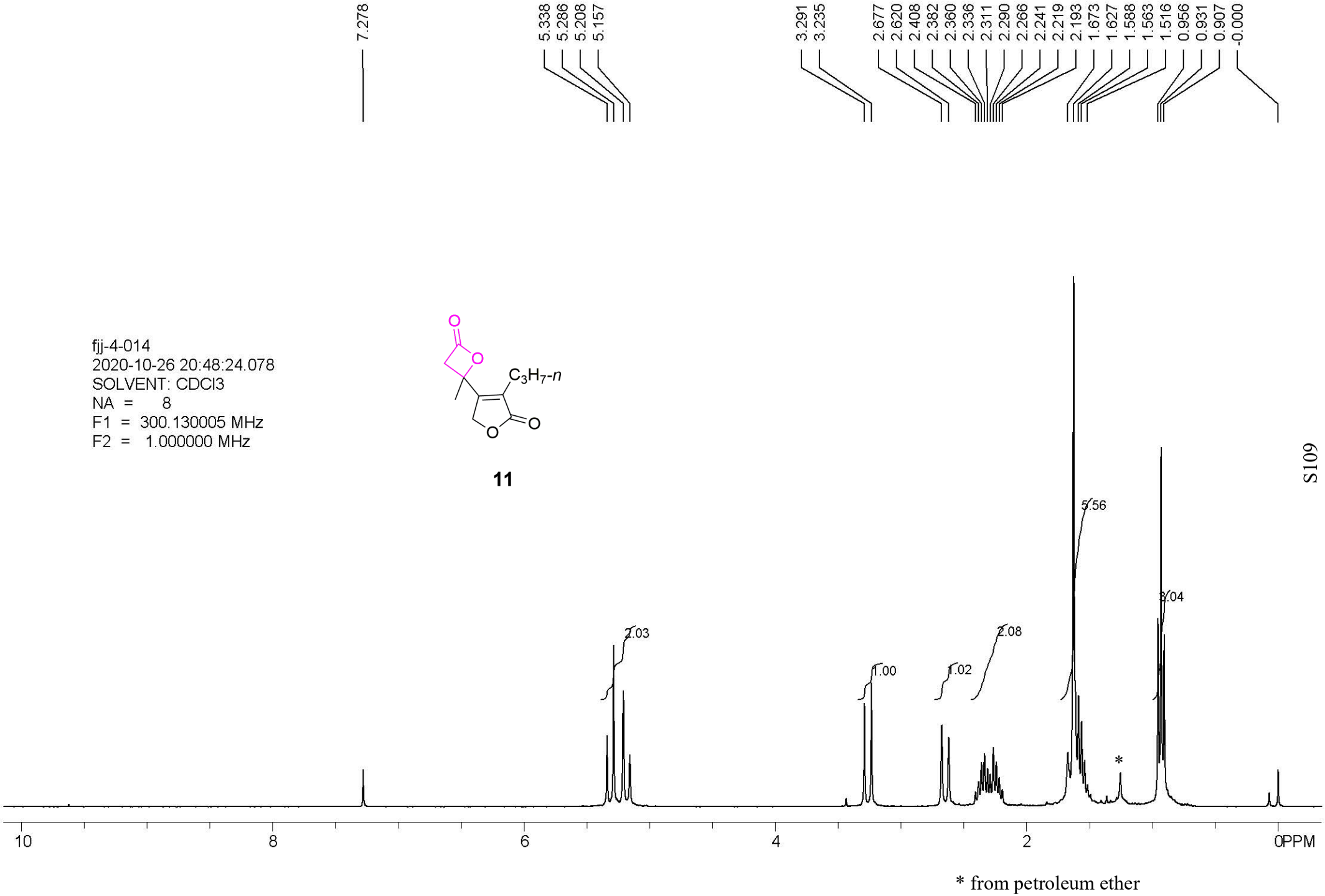
* from petroleum ether

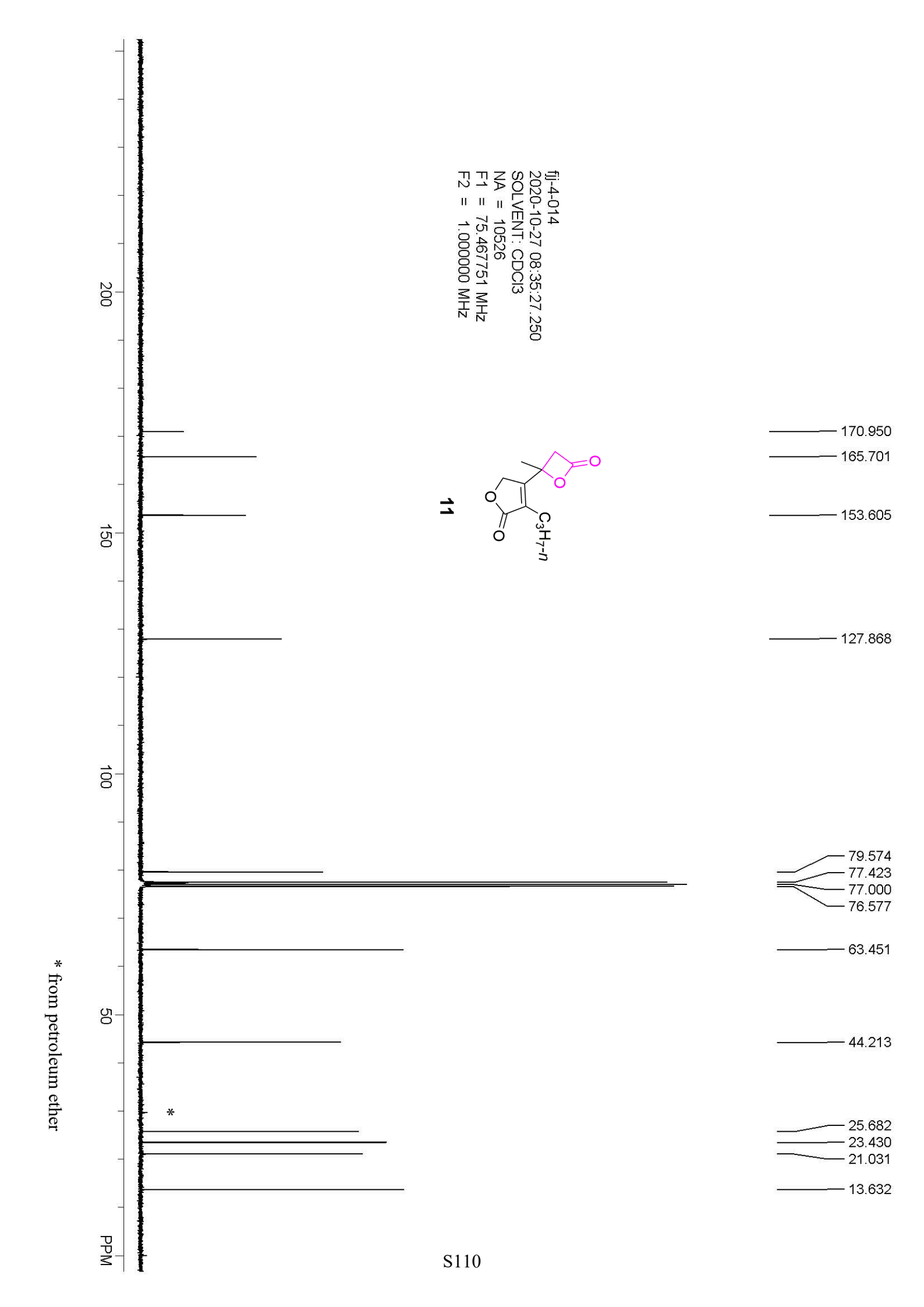

7.263
5.435
5.428
5.100
5.096
5.091
5.087
5.082
5.076
5.072
4.743
4.720
4.654
4.633
4.452
4.432
4.294
4.270
3.006
2.996
2.711
2.706
2.702
2.550
2.545
2.529
2.525
2.468
2.446
2.323
2.309
2.300
2.287
2.275
2.262
2.250
2.240
2.227
2.218
2.206
1.884
1.860
1.719
1.694
1.589
1.578
1.545
0.922
0.909
0.897
0.000
FJJ-2-162 in CDCL3
Dec 5 2019
SOLVENT: cdcl3
NA = 1
F1 = 599.770264 MHz
F2 = 150.825745 MHz
HO
O
C3H7-n
O
O
9, dr = 2.6/1
S105
0.67
0.27
0.72
0.27
0.72
0.28
0.22
0.59
0.70
0.28
2.01
0.70
2.38
3.94
2.99
10
8
6
4
2
0
PPM


fjj-2-162
2019-12-04 22:36:33.062
SOLVENT: CDCl3
NA = 261
F1 = 75.467751 MHz
F2 = 1.000000 MHz
9, dr = 2.6/1
HO
O
O
O
C3H7-n
172.825
172.586
159.873
158.780
125.864
125.597
93.325
91.063
81.623
77.423
77.000
76.577
58.469
54.912
46.814
44.764
25.342
25.250
25.121
23.577
21.325
21.196
13.512
13.448
250
200
150
100
50
0 PPM

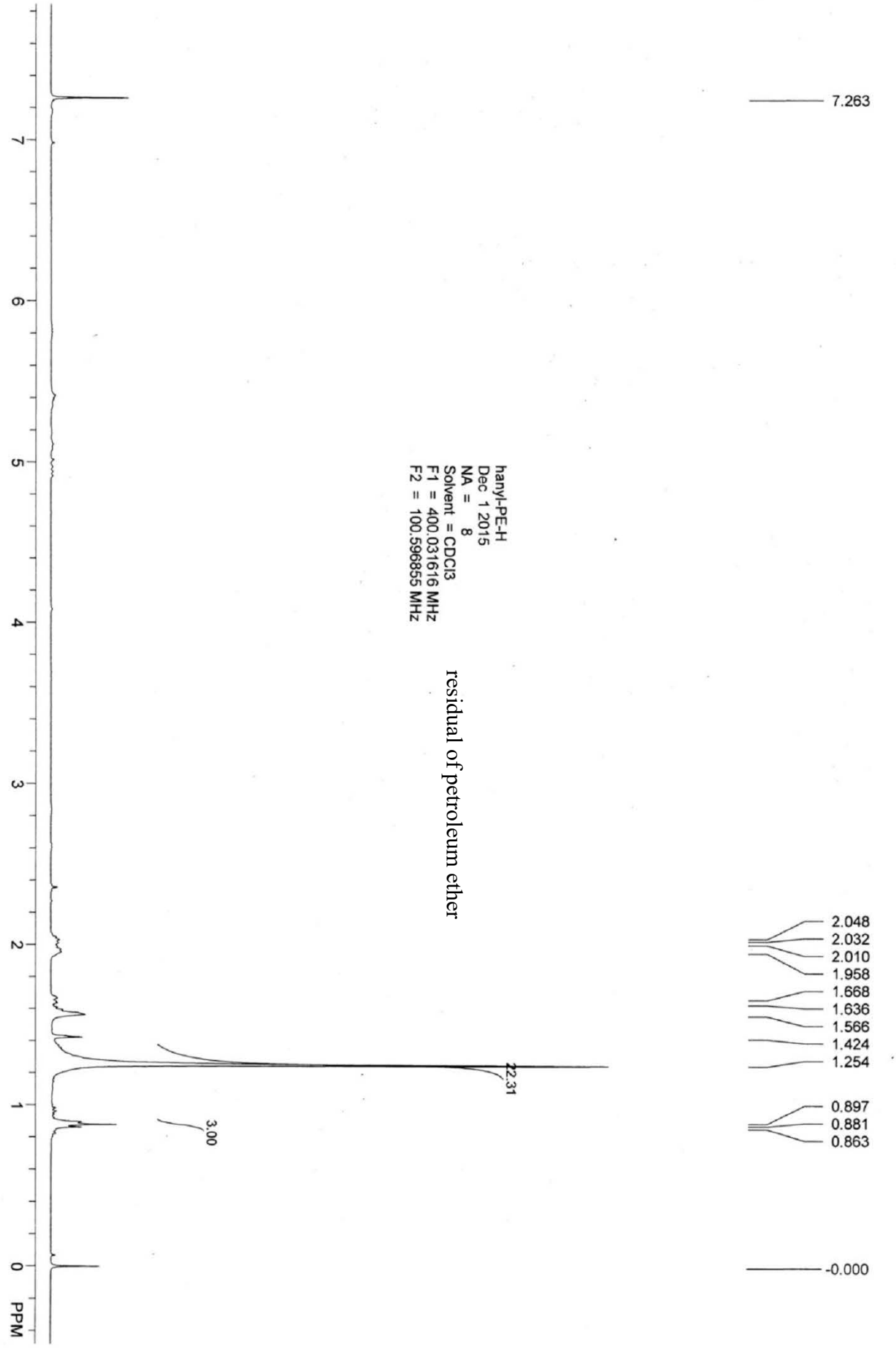

7.285
4.646
4.599
4.318
4.271
3.702
3.667
3.504
3.469
3.434
3.179
2.358
2.350
2.325
2.308
2.299
2.270
2.265
2.256
2.244
2.231
2.220
2.199
2.143
1.577
1.552
1.532
1.506
1.482
1.455
0.933
0.909
0.883
-0.000
fjj-2-180
2019-12-16 20:46:18.156
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
HO
HO
C3H7-n
O
O
10
S107
1.00
1.94
1.05
1.07
0.99
4.33
5.25
3.05
8
6
4
2
PPM

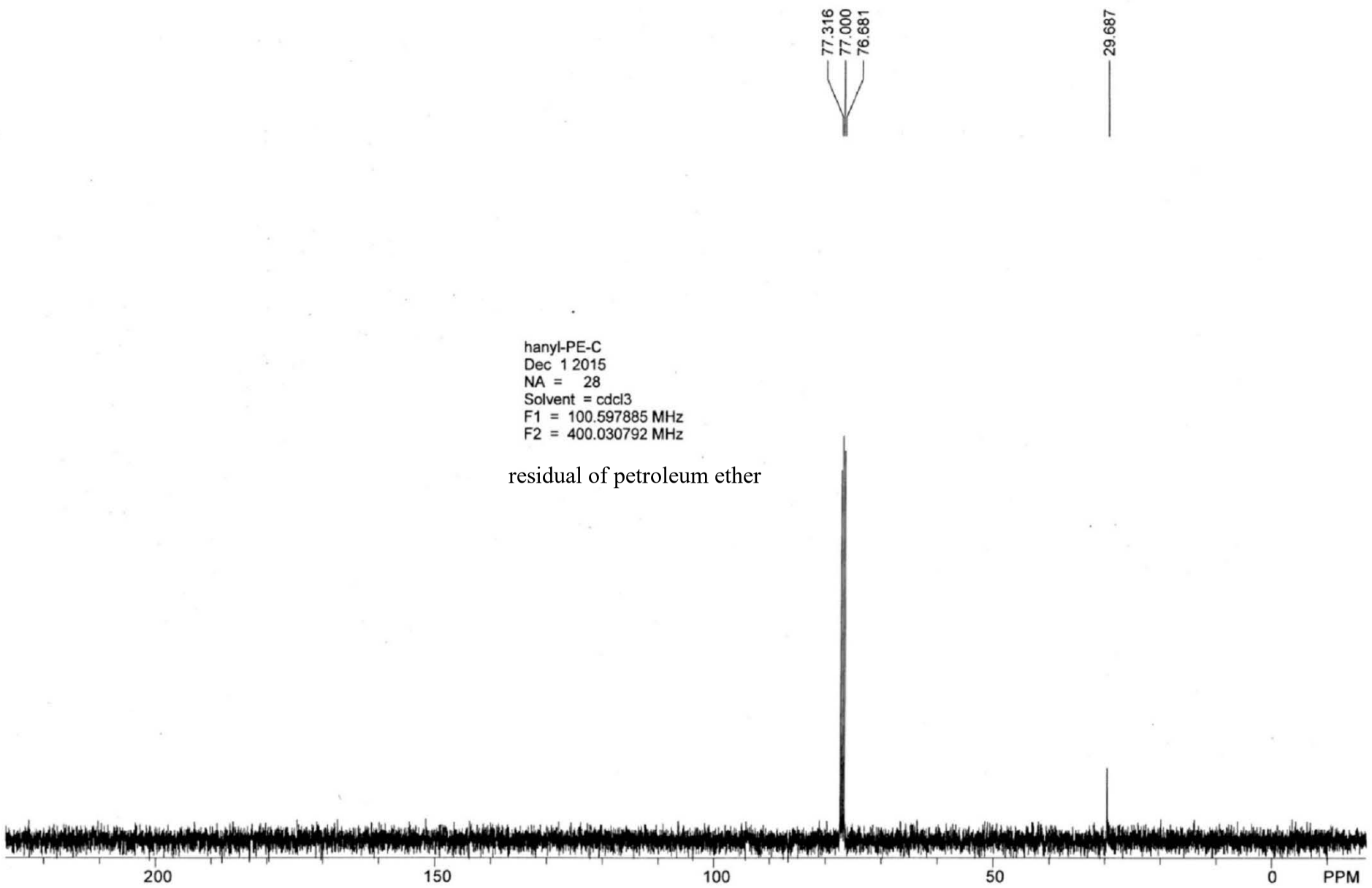

fjl-2-180
2019-12-16 21:05:23.468
SOLVENT: CDCl3
NA = 310
F1 = 75.467751 MHz
F2 = 1.000000 MHz
10
HO
HO
O
O
C3H7-n
200
150
100
50
PPM
173.395
164.616
128.171
86.321
77.423
77.000
76.577
56.806
56.539
39.736
25.416
25.039
21.463
13.659


7.278
5.338
5.286
5.208
5.157
3.291
3.235
2.677
2.620
2.408
2.382
2.360
2.336
2.311
2.290
2.266
2.241
2.219
2.193
1.673
1.627
1.588
1.563
1.516
0.956
0.931
0.907
-0.000
fjj-4-014
2020-10-26 20:48:24.078
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
O
O
O
O
C3H7-n
11
2.03
1.00
1.02
2.08
5.56
3.04
*
10
8
6
4
2
0PPM
* from petroleum ether



* from petroleum ether


hanyl-PE-H
Dec 1 2015
NA = 8
Solvent = CDCl3
F1 = 400.031616 MHz
F2 = 100.596855 MHz
residual of petroleum ether
22.31
3.00
7.263
2.048
2.032
2.010
1.958
1.668
1.636
1.566
1.424
1.254
0.897
0.881
0.863
-0.000
PPM


77.316
77.000
76.681
29.687
hanyl-PE-C
Dec 1 2015
NA = 28
Solvent = cdcl3
F1 = 100.597885 MHz
F2 = 400.030792 MHz
residual of petroleum ether
200
150
100
50
0
PPM