Copper-catalyzed Remote Double Functionalization of Allenynes

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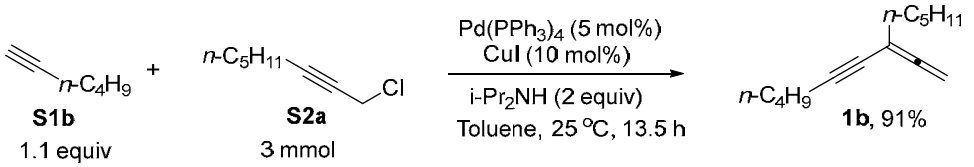
Supporting Information

General Information

^{1}H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ with a Bruker AM 300 MHz NMR spectrometer (^{1}H at 300 MHz, ^{13}C at 75 MHz, ^{19}F at 282 MHz) using TMS (^{1}H, $\delta = 0$), residual $CHCl_3$ (^{13}C, $\delta = 77.0$), and $CFCl_3$ (^{19}F, $\delta = 0$) as the internal standards.# IR spectra were recorded with a Perkin–Elmer 983G instrument. Elemental analyses were measured 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. $Pd(PPh_3)_4$ was purchased from *Dibai Chemical*. *i*-Pr_2NH and CuI were purchased from *Adamas*. $Cu(CH_3CN)_4PF_6$ was purchased from *Strem*. Binap was purchased from *Bidepharm*. B_2Pin_2 was purchased from *Chembee*. NaO^tBu and $PhMe_2SiBPin$ were purchased from *Energy Chemicals*. MeOH and ^{t}BuOH were dried over magnesium with iodine as the indicator and distilled freshly before use. THF and toluene were distilled over Na wire using benzophenone as the indicator under N_2 right before use. All the temperatures are referred to the oil baths used. Compounds **1a**,[1] **1r**,[2] **1t**[2], and **1z'**[1] were prepared as reported.

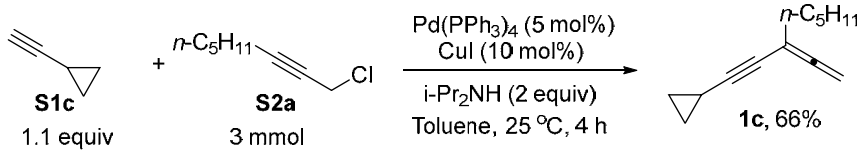
1. Synthesis of starting materials.

1.1 Preparation of 3-pentylnona-1,2-dien-4-yne **1b** (syl-8-1).[1]



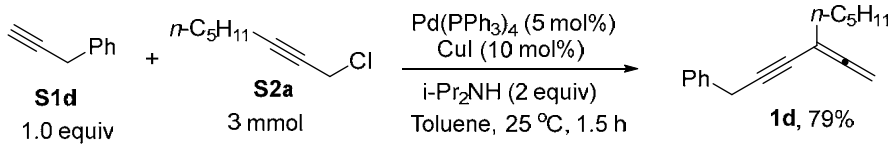
Typical Procedure I: To a Schlenk flask were added **S2a** (0.46 mL, d = 0.931 g/mL, 0.428 g, 3.0 mmol), $Pd(PPh_3)_4$ (0.1770 g, 0.15 mmol), and toluene (3 mL) under N_2 atmosphere. The resulting mixture was stirred at 25 °C for 5 minutes followed by the addition of **S1b** (0.38 mL, d = 0.715 g/mL, 0.272 g, 3.3 mmol), toluene (3 mL), CuI (0.0569 g, 0.3 mmol), and *i*-Pr_2NH (0.84 mL, d = 0.722 g/mL, 0.606 g, 6 mmol) sequentially under N_2. The resulting mixture was stirred at 25 °C for 13.5 hours as monitored by TLC and then filtrated through a pad of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **1b** (0.5121 g, 91%) (eluent: petroleum ether (60~90 °C) (300 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.95-4.80 (m, 2 H, $=CH_2$), 2.32 (t, *J* = 6.9 Hz, 2 H, CH_2), 2.18-2.00 (m, 2 H, CH_2), 1.65-1.24 (m, 10 H, CH_2 × 5), 1.02-0.83 (m, 6 H, CH_3 × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.4, 92.6, 89.9, 76.1, 75.2, 33.6, 31.1, 30.9, 27.3, 22.4, 21.9, 19.2, 14.0, 13.5; IR (neat) ν (cm^{-1}) 2958, 2931, 2873, 2860, 2224, 1942, 1466, 1433, 1379, 1330; MS (EI) *m/z*: 190 (M^+, 0.65), 161 ($(M-Et)^+$, 40.00), 91 (100); HRMS calcd. for $C_{14}H_{22}$ (M^+): 190.1722; Found: 190.1723.

1.2. Preparation of 5-cyclopropyl-3-pentylpenta-1,2-dien-4-yne **1c** (syl-8-17).



Following **Typical Procedure I**, the reaction of **S2a** (0.46 mL, d = 0.931 g/mL, 0.428 g, 3.0 mmol), $Pd(PPh_3)_4$ (0.1769 g, 0.15 mmol), toluene (4.5 mL), **S1c** (0.28 mL, d = 0.78 g/mL, 0.218 g, 3.3 mmol), toluene (4.5 mL), CuI (0.0581 g, 0.3 mmol), and *i*-Pr_2NH (0.84 mL, d = 0.722 g/mL, 0.606 g, 6 mmol) afforded **1c** (0.3426 g, 66%) (eluent: petroleum ether (60~90 °C) (450 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 4.88 (t, *J* = 2.9 Hz, 2 H, $=CH_2$), 2.15-1.95 (m, 2 H, CH_2), 1.57-1.42 (m, 2 H, CH_2), 1.42-1.15 (m, 5 H, $CH_2 \times 2 + CH$), 0.89 (t, *J* = 6.9 Hz, 3 H, CH_3), 0.83-0.74 (m, 2 H, CH_2), 0.74-0.60 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.6, 95.5, 89.8, 76.3, 70.5, 33.5, 31.1, 27.3, 22.4, 14.0, 8.5, 0.27; IR (neat) ν (cm^{-1}) 3093, 3012, 3956, 2929, 2858, 2223, 1941, 1466, 1431, 1379, 1363, 1052, 1028; MS (EI) *m/z*: 174 (M^+, 1.03), 117 (100); HRMS calcd. for $C_{13}H_{18}$ (M^+): 174.1409; Found: 174.1411.

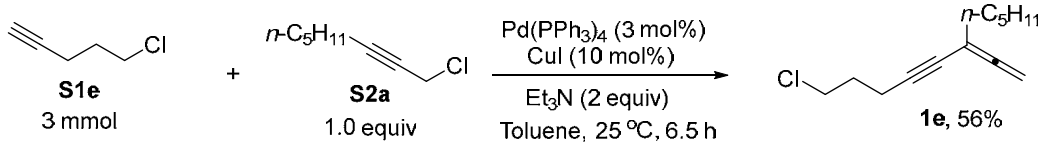
1.3. Preparation of 6-phenyl-3-pentylhexa-1,2-dien-4-yne **1d** (syl-8-14).



Following **Typical Procedure I**, the reaction of **S2a** (0.46 mL, d = 0.931 g/mL, 0.428 g, 3.0 mmol), $Pd(PPh_3)_4$ (0.1771 g, 0.15 mmol), toluene (4.5 mL), **S1d** (0.37 mL, d = 0.934 g/mL, 0.346 g, 3.0 mmol), toluene (4.5 mL), CuI (0.0580 g, 0.3 mmol), and *i*-Pr_2NH (0.84 mL, d = 0.722 g/mL, 0.606 g, 6 mmol) afforded **1d** (0.5270 g, 79%) (eluent: petroleum ether (60~90 °C) (600 mL) to petroleum ether (60~90 °C)/ethyl

acetate = 50/1 (204 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47-7.15 (m, 5 H, ArH), 5.00-4.84 (m, 2 H, $=CH_2$), 3.74 (s, 2 H, CH_2), 2.23-2.05 (m, 2 H, CH_2), 1.62-1.43 (m, 2 H, CH_2), 1.40-1.20 (m, 4 H, $CH_2 \times 2$), 0.89 (t, J = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.5, 136.8, 128.4, 127.8, 126.5, 89.7, 89.6, 77.6, 76.4, 33.4, 31.1, 27.4, 25.8, 22.4, 14.0; IR (neat) ν (cm^{-1}) 3086, 3064, 3030, 2956, 2928, 2858, 2228, 1941, 1603, 1495, 1453, 1420, 1181, 1074, 1030; MS (EI) *m/z*: 224 (M^+, 1.30), 167 (100), 153 (100); HRMS calcd. for $C_{17}H_{20}$ (M^+): 224.1565; Found: 224.1564.

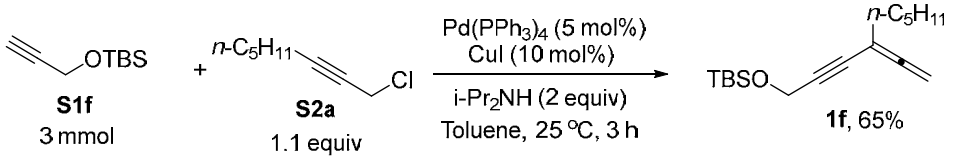
1.4. Preparation of 8-chloro-3-pentylocta-1,2-dien-4-yne **1e** (syl-7-172).



To a Schlenk flask were added **S2a** (0.46 mL, d = 0.931 g/mL, 0.428 g, 3.0 mmol), $Pd(PPh_3)_4$ (0.1051 g, 0.09 mmol), and toluene (3 mL) under N_2 atmosphere. The resulting mixture was stirred at 25 °C for 5 minutes followed by the addition of **S1e** (0.32 mL, d = 0.968 g/mL, 0.310 g, 3 mmol), toluene (3 mL), CuI (0.0571 g, 0.3 mmol), and Et_3N (0.83 mL, d = 0.728 g/mL, 0.604 g, 6 mmol) sequentially under N_2. The resulting mixture was stirred at 25 °C for 6.5 hours as monitored by TLC and then filtrated through a pad of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **1e** (0.3561 g, 56%) (eluent: petroleum ether (60~90 °C) (300 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.93-4.80 (m, 2 H, $=CH_2$), 3.66 (t, J = 6.5 Hz, 2 H, CH_2), 2.52 (t, J = 6.6 Hz, 2 H, CH_2), 2.15-1.87 (m, 4 H, $CH_2 \times 2$), 1.56-1.41 (m, 2 H, CH_2),

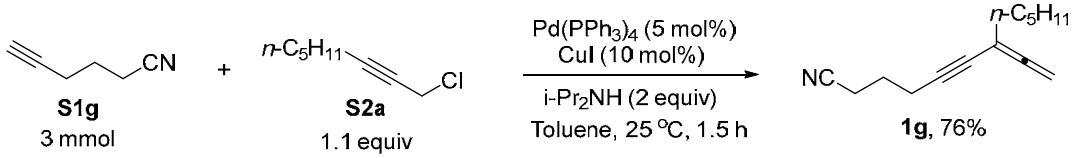
1.40-1.20 (m, 4 H, $CH_2 \times 2$), 0.90 (t, $J = 6.5$ Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.4, 90.2, 89.6, 76.4, 43.7, 33.4, 31.4, 31.0, 27.3, 22.4, 17.0, 14.0; IR (neat) ν (cm^{-1}) 2957, 2928, 2858, 2215, 1942, 1457, 1433, 1291; MS (EI) *m/z*: 212 (M^+(^{37}Cl), 0.20), 210 (M^+(^{35}Cl), 0.57), 183 ((M^+(^{37}Cl)-Et), 16.64), 181 ((M^+(^{35}Cl)-Et), 45.26), 91 (100); HRMS calcd. for $C_{13}H_{19}{}^{35}Cl$ (M^+): 210.1175; Found: 210.1176.

1.5. Preparation of 6-((*tert*-butyldimethylsilyl)oxy)-3-pentylhexa-1,2-dien-4-yne **1f** (syl-7-199, syl-8-75).



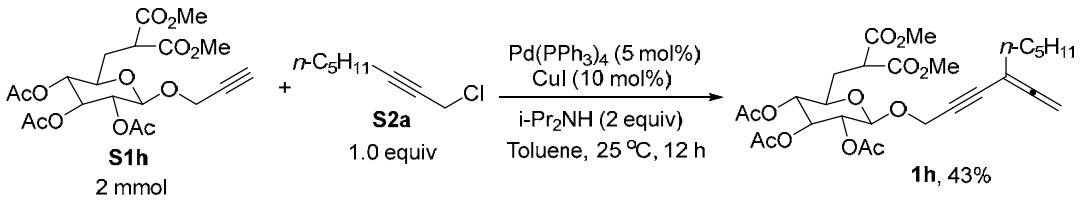
Following **Typical Procedure I**, the reaction of **S2a** (0.51 mL, d = 0.931 g/mL, 0.475 g, 3.3 mmol), $Pd(PPh_3)_4$ (0.1734 g, 0.15 mmol), toluene (4.5 mL), **S1f** (0.5101 g, 3 mmol), toluene (4.5 mL), CuI (0.0572 g, 0.3 mmol), and *i*-Pr_2NH (0.84 mL, d = 0.722 g/mL, 0.606 g, 6 mmol) afforded **1f** (0.5446 g, 65%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 300/1 (903 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 4.95-4.85 (m, 2 H, $=CH_2$), 4.43 (s, 2 H, OCH_2), 2.13-2.03 (m, 2 H, CH_2), 1.56-1.40 (m, 2 H, CH_2), 1.37-1.22 (m, 4 H, $CH_2 \times 2$), 0.94-0.82 (m, 12 H, $CH_3 \times 4$), 0.12 (s, 6 H, $CH_3 \times 2$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.6, 90.0, 89.3, 80.0, 52.3, 33.1, 31.1, 27.4, 25.8, 22.4, 18.3, 14.0, -5.1; IR (neat) ν (cm^{-1}) 2956, 2929, 2858, 2219, 1942, 1474, 1463, 1363, 1255, 1085; MS (EI) *m/z*: 278 (M^+, 0.66), 221 ((M-^{t}Bu)$^+$, 23.89), 191 (100); HRMS calcd. for $C_{17}H_{31}OSi$ $(M + H)^+$: 279.2139; Found: 279.2141.

1.6. Preparation of 8-cyano-3-pentylocta-1,2-dien-4-yne **1g** (syl-7-197).



Following **Typical Procedure I**, the reaction of **S2a** (0.51 mL, d = 0.931 g/mL, 0.475 g, 3.3 mmol), $Pd(PPh_3)_4$ (0.1729 g, 0.15 mmol), toluene (3 mL), **S1g** (0.31 mL, d = 0.889 g/mL, 0.276 g, 3 mmol), toluene (3 mL), CuI (0.0575 g, 0.3 mmol), and *i*-Pr_2NH (0.84 mL, d = 0.722 g/mL, 0.606 g, 6 mmol) afforded **1g** (0.4526 g, 76%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 15/1 (320 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 4.98-4.78 (m, 2 H, $=CH_2$), 2.60-2.37 (m, 4 H, CH_2 × 2), 2.12-1.96 (m, 2 H, CH_2), 1.95-1.77 (m, 2 H, CH_2), 1.58-1.41 (m, 2 H, CH_2), 1.38-1.17 (m, 4 H, CH_2 × 2), 0.90 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.5, 119.1, 89.4, 89.0, 76.5, 33.3, 31.0, 27.3, 24.7, 22.4, 18.7, 16.1, 14.0; IR (neat) ν (cm^{-1}) 2955, 2931, 2859, 2248, 1941, 1715, 1456, 1432, 1379, 1347, 1313; MS (EI) *m/z*: 201 (M^+, 4.05), 144 (100); HRMS calcd. for $C_{14}H_{20}N$ $(M + H)^+$: 202.1590; Found: 202.1590.

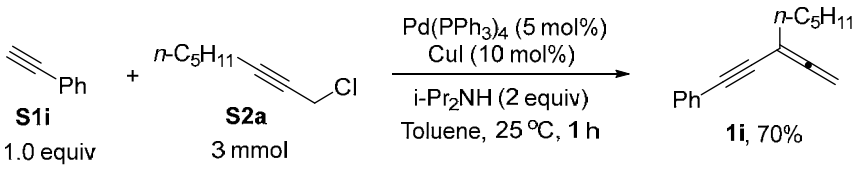
1.7. Preparation of **1h** (syl-8-73).



Following **Typical Procedure I**, the reaction of **S2a** (0.31 mL, d = 0.931 g/mL, 0.289 g, 2.0 mmol), $Pd(PPh_3)_4$ (0.1157 g, 0.1 mmol), toluene (3 mL), **S1h** (0.9171 g, 2 mmol), toluene (3 mL), CuI (0.0390 g, 0.2 mmol), and *i*-Pr_2NH (0.56 mL, d = 0.722 g/mL, 0.404 g, 4 mmol) afforded **1h** (0.4860 g, 43%) (eluent: petroleum ether (60~90

°C)/ethyl acetate/DCM = 5/1/1 (840 mL)): liquid; $[\alpha]_D^{20}$ = -50.708 (c = 0.65, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 5.19 (t, *J* = 9.5 Hz, 1 H, OCH), 5.04-4.82 (m, 4 H, $=CH_2$ + OCH_2), 4.64 (d, *J* = 8.1 Hz, 1 H, OCH), 4.45 (s, 2 H, OCH_2), 3.77 (s, 3 H, OCH_3), 3.73 (s, 3 H, OCH_3), 3.70-3.62 (m, 1 H, OCH), 3.60-3.45 (m, 1 H, OCH), 2.33-2.18 (m, 1 H, one proton of CH_2), 2.17-1.94 (m, 12 H, CH_3 × 3 + CH_2 + one proton of CH_2), 1.57-1.42 (m, 2 H, CH_2), 1.40-1.27 (m, 4 H, CH_2 × 2), 0.91 (t, *J* = 6.5 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.7, 170.2, 169.6, 169.4, 169.2, 98.2, 88.8, 85.4, 82.3, 76.9, 72.7, 71.8, 71.1, 71.0, 56.8, 52.7, 52.6, 47.5, 32.9, 30.9, 30.3, 27.2, 22.3, 20.6, 20.5, 13.9; IR (neat) ν (cm^{-1}) 2956, 2860, 2222, 1940, 1758, 1437, 1367, 1246, 1217, 1159, 1058; MS (EI) *m/z*: 360 ((M - OAc × 3 - Et)$^+$, 19.91), 126 (100); HRMS calcd. for $C_{28}H_{38}NaO_{12}^+$ (M^+ + Na): 589.2255; Found: 589.2256.

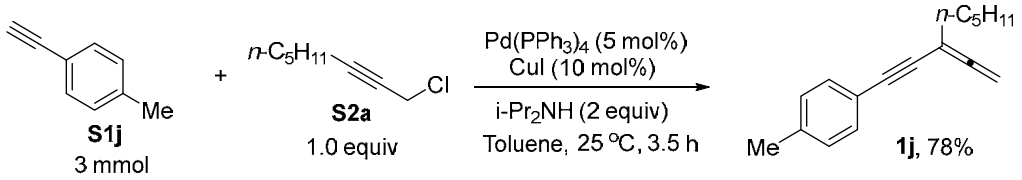
1.8. Preparation of 5-phenyl-3-pentylpenta-1,2-dien-4-yne **1i** (syl-8-6).



Following **Typical Procedure I**, the reaction of **S2a** (0.46 mL, d = 0.931 g/mL, 0.428 g, 3.0 mmol), $Pd(PPh_3)_4$ (0.1775 g, 0.15 mmol), toluene (4.5 mL), **S1i** (0.33 mL, d = 0.93 g/mL, 0.307 g, 3.0 mmol), toluene (4.5 mL), CuI (0.0580 g, 0.3 mmol), and *i*-Pr_2NH (0.84 mL, d = 0.722 g/mL, 0.606 g, 6 mmol) afforded **1i** (0.4381 g, 70%) (eluent: petroleum ether (60~90 °C) (360 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.50-7.38 (m, 2 H, ArH), 7.34-7.24 (m, 3 H, ArH), 4.98 (t, *J* = 3.0 Hz, 2 H, $=CH_2$), 2.27-2.14 (m, 2 H, CH_2), 1.67-1.50 (m, 2 H, CH_2), 1.42-1.27 (m, 4 H, CH_2 × 2), 0.91 (t, *J* = 6.9 Hz, 3

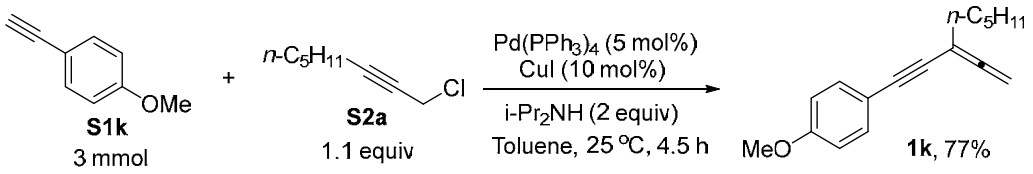
H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.6, 131.4, 128.2, 128.0, 123.5, 91.5, 89.9, 84.6, 76.7, 33.3, 31.1, 27.4, 22.4, 14.0; IR (neat) ν (cm^{-1}) 2956, 2928, 2858, 2209, 1938, 1597, 1490, 1466, 1443, 1378, 1324, 1069, 1026; MS (EI) *m/z*: 210 (M^+, 10.28), 154 (100), 153 (100); HRMS calcd. for $C_{16}H_{18}$ (M^+): 210.1409; Found: 210.1408.

1.9. Preparation of 3-pentyl-5-(*p*-tolyl)penta-1,2-dien-4-yne **1j** (syl-8-30).



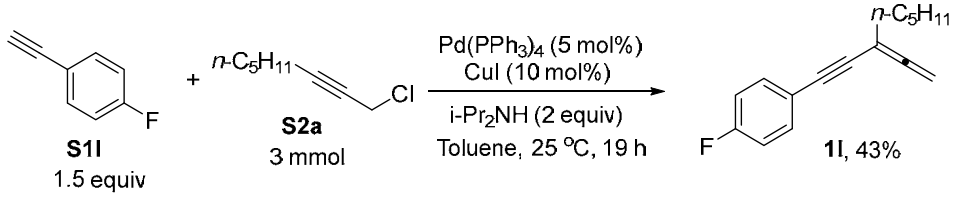
Following **Typical Procedure I**, the reaction of **S2a** (0.46 mL, d = 0.931 g/mL, 0.428 g, 3.0 mmol), $Pd(PPh_3)_4$ (0.1780 g, 0.15 mmol), toluene (4.5 mL), **S2a** (0.4088 g, 3.0 mmol), toluene (4.5 mL), CuI (0.0575 g, 0.3 mmol), and *i*-Pr_2NH (0.84 mL, d = 0.722 g/mL, 0.606 g, 6 mmol) afforded **1j** (0.5265 g, 78%) (eluent: petroleum ether (60~90 °C) (500 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.32 (d, *J* = 8.1 Hz, 2 H, ArH), 7.09 (d, *J* = 8.1 Hz, 2 H, ArH), 4.96 (t, *J* = 2.7 Hz, 2 H, $=CH_2$), 2.32 (s, 3 H, CH_3), 2.25-2.12 (m, 2 H, CH_2), 1.67-1.46 (m, 2 H, CH_2), 1.42-1.22 (m, 4 H, $CH_2 \times 2$), 0.90 (t, *J* = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.6, 138.0, 131.3, 129.0, 120.5, 91.7, 90.0, 83.8, 76.6, 33.4, 31.1, 27.4, 22.4, 21.4, 14.0; IR (neat) ν (cm^{-1}) 2956, 2927, 2858, 2211, 1937, 1510, 1465, 1378, 1321; MS (EI) *m/z*: 224 (M^+, 21.83), 168 (100); HRMS calcd. for $C_{17}H_{20}$ (M^+): 224.1565; Found: 224.1563.

1.10. Preparation of 5-(4-methoxyphenyl)-3-pentylpenta-1,2-dien-4-yne **1k** (syl-8-24).



Following **Typical Procedure I**, the reaction of **S2a** (0.51 mL, d = 0.931 g/mL, 0.474 g, 3.3 mmol), $Pd(PPh_3)_4$ (0.1760 g, 0.15 mmol), toluene (4.5 mL), **S1k** (0.3970 g, 3.0 mmol), toluene (4.5 mL), CuI (0.0575 g, 0.3 mmol), and *i*-Pr_2NH (0.84 mL, d = 0.722 g/mL, 0.606 g, 6 mmol) afforded **1k** (0.5540 g, 77%) (eluent: petroleum ether (60~90 °C)/DCM = (8/1) (450 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.7 Hz, 2 H, ArH), 6.82 (d, *J* = 9.0 Hz, 2 H, ArH), 4.96 (t, *J* = 2.7 Hz, 2 H, $=CH_2$), 3.78 (s, 3 H, OCH_3), 2.27-2.13 (m, 2 H, CH_2), 1.67-1.49 (m, 2 H, CH_2), 1.42-1.25 (m, 4 H, $CH_2 \times 2$), 0.91 (t, *J* = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.5, 159.4, 132.8, 115.6, 113.8, 91.4, 90.0, 83.0, 76.6, 55.1, 33.4, 31.1, 27.4, 22.4, 14.0; IR (neat) ν (cm^{-1}) 2956, 2931, 2858, 2194, 1937, 1754, 1716, 1663, 1605, 1569, 1510, 1465, 1442, 1378, 1288, 1249, 1172, 1106, 1033; MS (EI) *m/z*: 240 (M^+, 53.03), 184 (100); HRMS calcd. for $C_{17}H_{20}O$ (M^+): 240.1514; Found: 240.1514.

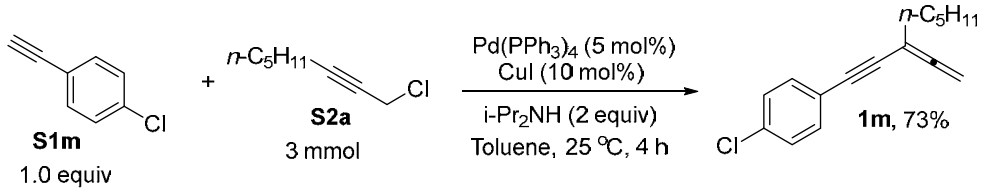
1.11. Preparation of 5-(4-fluorophenyl)-3-pentylpenta-1,2-dien-4-yne **1l** (syl-8-47).



Following **Typical Procedure I**, the reaction of **S2a** (0.46 mL, d = 0.931 g/mL, 0.428 g, 3.0 mmol), $Pd(PPh_3)_4$ (0.1767 g, 0.15 mmol), toluene (4.5 mL), **S1l** (0.5398 g, 3 mmol), toluene (4.5 mL), CuI (0.0572 g, 0.3 mmol), and *i*-Pr_2NH (0.84 mL, d = 0.722

g/mL, 0.606 g, 6 mmol) afforded **1l** (0.2933 g, 43%) (eluent: petroleum ether (60~90 °C) (500 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50-7.33 (m, 2 H, ArH), 6.99 (t, *J* = 8.7 Hz, 2 H, ArH), 5.08-4.92 (m, 2 H, =CH_2), 2.26-2.12 (m, 2 H, CH_2), 1.68-1.48 (m, 2 H, CH_2), 1.45-1.25 (m, 4 H, CH_2 × 2), 0.91 (t, *J* = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.6, 162.3 (d, *J* = 247.5 Hz), 133.2 (d, *J* = 8.3 Hz), 119.6 (d, *J* = 3.5 Hz), 115.5 (d, *J* = 21.4 Hz), 90.3, 89.7, 84.2, 76.8, 33.3, 31.1, 27.4, 22.4, 14.0; ^{19}F NMR (282 MHz, $CDCl_3$) δ -111.7; IR (neat) ν (cm^{-1}) 2957, 2930, 2859, 1937, 1601, 1507, 1466, 1232, 1155, 1092; MS (EI) *m/z*: 228 (M^+, 1.79), 172 (100); HRMS calcd. for $C_{16}H_{17}F$ (M^+): 228.1314; Found: 228.1313.

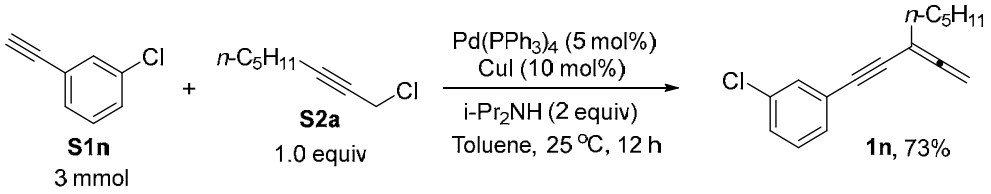
1.12. Preparation of 5-(4-chlorophenyl)-3-pentylpenta-1,2-dien-4-yne **1m** (syl-8-19).



Following **Typical Procedure I**, the reaction of **S2a** (0.46 mL, d = 0.931 g/mL, 0.428 g, 3.0 mmol), $Pd(PPh_3)_4$ (0.1710 g, 0.15 mmol), toluene (4.5 mL), **S1m** (0.4091 g, 3.0 mmol), toluene (4.5 mL), CuI (0.0580 g, 0.3 mmol), and *i*-Pr_2NH (0.84 mL, d = 0.722 g/mL, 0.606 g, 6 mmol) afforded **1m** (0.5360 g, 73%) (eluent: petroleum ether (60~90 °C) (360 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 (d, *J* = 8.1 Hz, 2 H, ArH), 7.27 (d, *J* = 8.7 Hz, 2 H, ArH), 4.99 (t, *J* = 2.9 Hz, 2 H, =CH_2), 2.27-2.13 (m, 2 H, CH_2), 1.67-1.47 (m, 2 H, CH_2), 1.45-1.25 (m, 4 H, CH_2 × 2), 0.91 (t, *J* = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.7, 133.9, 132.6, 128.5, 122.0, 90.3, 89.7, 85.6, 76.8, 33.2, 31.1, 27.4, 22.4, 14.0; IR (neat) ν (cm^{-1}) 2956, 2929, 2858, 2210, 1937,

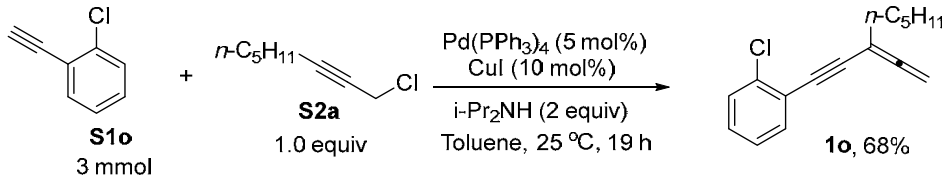
1591, 1489, 1466, 1397, 1379, 1091, 1014; MS (EI) *m/z*: 246 (M^+(^{37}Cl), 3.03), 244 (M^+(^{35}Cl), 8.67), 188 (100); HRMS calcd. for $C_{16}H_{17}{}^{35}Cl$ (M^+): 244.1019; Found: 244.1019.

1.13. Preparation of 5-(3-chlorophenyl)-3-pentylpenta-1,2-dien-4-yne **1n** (syl-8-33).



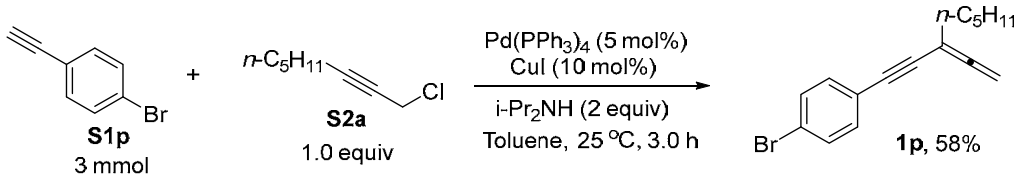
Following **Typical Procedure I**, the reaction of **S2a** (0.46 mL, d = 0.931 g/mL, 0.428 g, 3.0 mmol), $Pd(PPh_3)_4$ (0.1771 g, 0.15 mmol), toluene (4.5 mL), **S1n** (0.37 mL, d = 1.109 g/mL, 0.410 g, 3 mmol), toluene (4.5 mL), CuI (0.0572 g, 0.3 mmol), and *i*-Pr_2NH (0.84 mL, d = 0.722 g/mL, 0.606 g, 6 mmol) afforded **1n** (0.5365 g, 73%) (eluent: petroleum ether (60~90 °C) (400 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42 (s, 1 H, ArH), 7.35-7.15 (m, 3 H, ArH), 5.00 (t, *J* = 2.6 Hz, 2 H, =CH_2), 2.27-2.10 (m, 2 H, CH_2), 1.67-1.47 (m, 2 H, CH_2), 1.42-1.26 (m, 4 H, CH_2 × 2), 0.91 (t, *J* = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.8, 134.0, 131.2, 129.5, 129.4, 128.2, 125.2, 90.0, 89.6, 85.9, 76.9, 33.2, 31.1, 27.4, 22.4, 14.0; IR (neat) ν (cm^{-1}) 2956, 2928, 2858, 2212, 1937, 1591, 1560, 1475, 1093, 1078; MS (EI) *m/z*: 246 (M^+(^{37}Cl), 1.20), 244 (M^+(^{35}Cl), 3.45), 188 (100); HRMS calcd. for $C_{16}H_{17}{}^{35}Cl$ (M^+): 244.1019; Found: 244.1020.

1.14. Preparation of 5-(2-chlorophenyl)-3-pentylpenta-1,2-dien-4-yne **1o** (syl-8-48).



Following **Typical Procedure I**, the reaction of **S2a** (0.46 mL, d = 0.931 g/mL, 0.428 g, 3.0 mmol), $Pd(PPh_3)_4$ (0.1770 g, 0.15 mmol), toluene (4.5 mL), **S1o** (0.36 mL, d = 1.125 g/mL, 0.405 g, 3 mmol), toluene (4.5 mL), CuI (0.0570 g, 0.3 mmol), and *i*-Pr_2NH (0.84 mL, d = 0.722 g/mL, 0.606 g, 6 mmol) afforded **1o** (0.4947 g, 68%) (eluent: petroleum ether (60~90 °C) (500 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.52-7.41 (m, 1 H, ArH), 7.41-7.30 (m, 1 H, ArH), 7.26-7.10 (m, 2 H, ArH), 5.00 (t, *J* = 2.9 Hz, 2 H, $=CH_2$), 2.32-2.13 (m, 2 H, CH_2), 1.71-1.55 (m, 2 H, CH_2), 1.43-1.23 (m, 4 H, CH_2 × 2), 0.91 (t, *J* = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.7, 135.7, 132.9, 129.2, 128.9, 126.3, 123.4, 89.9, 89.8, 88.3, 76.8, 33.3, 31.1, 27.4, 22.4, 14.0; IR (neat) ν (cm^{-1}) 2956, 2929, 2858, 2211, 1936, 1474, 1437, 1326, 1127, 1052, 1033; MS (EI) *m/z*: 246 (M^+(^{37}Cl), 0.88), 244 (M^+(^{35}Cl), 2.64), 188 (100); HRMS calcd. for $C_{16}H_{17}{}^{35}Cl$ (M^+): 244.1019; Found: 244.1021.

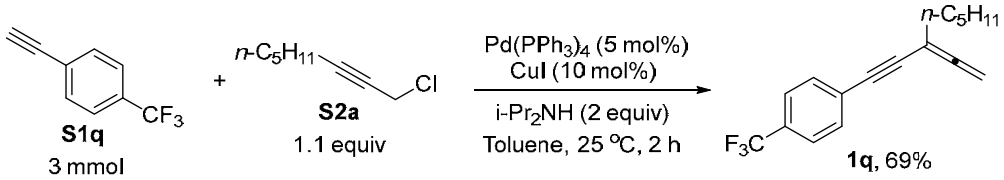
1.15. Preparation of 5-(4-bromophenyl)-3-pentylpenta-1,2-dien-4-yne **1p** (syl-8-28).



Following **Typical Procedure I**, the reaction of **S2a** (0.46 mL, d = 0.931 g/mL, 0.428 g, 3.0 mmol), $Pd(PPh_3)_4$ (0.1775 g, 0.15 mmol), toluene (4.5 mL), **S1p** (0.5417 g, 3 mmol), toluene (4.5 mL), CuI (0.0580 g, 0.3 mmol), and *i*-Pr_2NH (0.84 mL, d =

0.722 g/mL, 0.606 g, 6 mmol) afforded **1p** (0.5059 g, 58%) (eluent: petroleum ether (60~90 °C) (400 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48-7.38 (m, 2 H, ArH), 7.33-7.22 (m, 2 H, ArH), 4.99 (t, *J* = 2.7 Hz, 2 H, =CH_2), 2.28-2.12 (m, 2 H, CH_2), 1.67-1.48 (m, 2 H, CH_2), 1.41-1.25 (m, 4 H, CH_2 × 2), 0.91 (t, *J* = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.7, 132.8, 131.5, 122.5, 122.2, 90.3, 89.7, 85.8, 76.9, 33.2, 31.1, 27.4, 22.4, 14.0; IR (neat) ν (cm^{-1}) 2955, 2928, 2857, 2210, 1937, 1586, 1486, 1466, 1393, 1070, 1011; MS (EI) *m/z*: 290 (M^+(^{81}Br), 4.68), 288 (M^+(^{79}Br), 4.90), 234 (100), 232 (100); HRMS calcd. for $C_{16}H_{17}{}^{79}Br$ (M^+): 288.0514; Found: 288.0516.

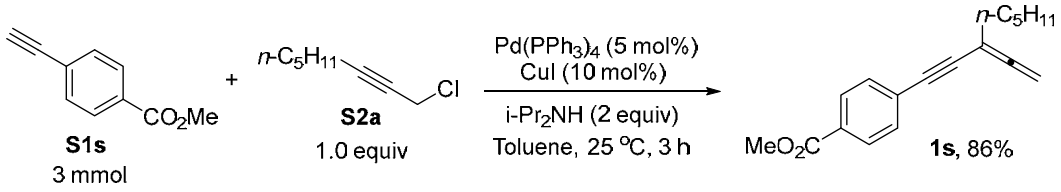
1.16. Preparation of 5-(4-trifluoromethylphenyl)-3-pentylpenta-1,2-dien-4-yne **1q** (syl-8-22).



Following **Typical Procedure I**, the reaction of **S2a** (0.51 mL, d = 0.931 g/mL, 0.475 g, 3.3 mmol), $Pd(PPh_3)_4$ (0.1760 g, 0.15 mmol), toluene (4.5 mL), **S1q** (0.5092 g, 3.0 mmol), toluene (4.5 mL), CuI (0.0580 g, 0.3 mmol), and *i*-Pr_2NH (0.84 mL, d = 0.722 g/mL, 0.606 g, 6 mmol) afforded **1q** (0.5747 g, 69%) (eluent: petroleum ether (60~90 °C) (360 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.4 Hz, 2 H, ArH), 7.52 (d, *J* = 8.7 Hz, 2 H, ArH), 5.02 (t, *J* = 2.9 Hz, 2 H, =CH_2), 2.28-2.14 (m, 2 H, CH_2), 1.66-1.48 (m, 2 H, CH_2), 1.44-1.25 (m, 4 H, CH_2 × 2), 0.91 (t, *J* = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.9, 131.6, 129.7 (q, *J* = 32.4 Hz), 127.43, 127.41, 125.2 (q, *J* = 3.7 Hz), 124.0 (q, *J* = 270.5 Hz), 90.1, 89.6, 87.3, 33.2, 31.1, 27.5,

22.5, 14.0; ^{19}F NMR (282 MHz, $CDCl_3$) δ -63.2; IR (neat) ν (cm^{-1}) 2958, 2931, 2860, 2211, 1937, 1615, 1324, 1168, 1130, 1105, 1066, 1017; MS (EI) *m/z*: 278 (M^+, 3.00), 222 (100); HRMS calcd. for $C_{17}H_{17}F_3$ (M^+): 278.1282; Found: 278.1281.

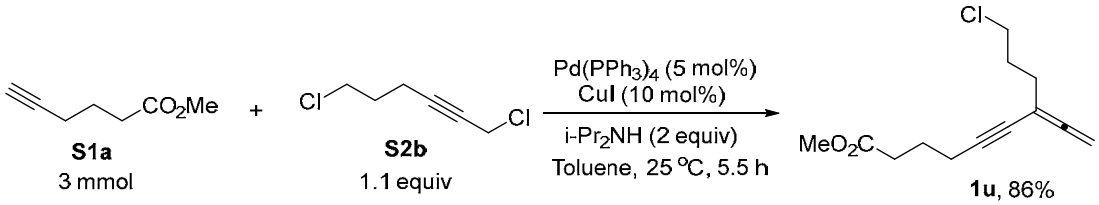
1.17. Preparation of 5-(4-(methoxycarbonyl)phenyl)-3-pentylpenta-1,2-dien-4-yne **1s** (syl-8-42).



Following **Typical Procedure I**, the reaction of **S2a** (0.46 mL, d = 0.931 g/mL, 0.428 g, 3.0 mmol), $Pd(PPh_3)_4$ (0.1770 g, 0.15 mmol), toluene (4.5 mL), **S1s** (0.4802 g, 3 mmol), toluene (4.5 mL), CuI (0.0568 g, 0.3 mmol), and *i*-Pr_2NH (0.84 mL, d = 0.722 g/mL, 0.606 g, 6 mmol) afforded **1s** (0.6893 g, 86%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 30/1 (465 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.1 Hz, 2 H, ArH), 7.48 (d, *J* = 8.1 Hz, 2 H, ArH), 5.12-4.90 (m, 2 H, $=CH_2$), 3.91 (s, 3 H, OCH_3), 2.30-2.13 (m, 2 H, CH_2), 1.64-1.50 (m, 2 H, CH_2), 1.43-1.22 (m, 4 H, CH_2 × 2), 0.91 (t, *J* = 6.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.8, 166.5, 131.2, 129.4, 129.1, 128.2, 90.6, 89.6, 87.8, 76.9, 52.1, 33.1, 31.0, 27.4, 22.4, 14.0; IR (neat) ν (cm^{-1}) 2954, 2930, 2859, 2208, 1936, 1727, 1605, 1435, 1406, 1307, 1275, 1192, 1175, 1107, 1018; MS (EI) *m/z*: 268 (M^+, 2.91), 212 (100); HRMS calcd. for $C_{18}H_{20}O_2$ (M^+): 268.1463; Found: 268.1463.

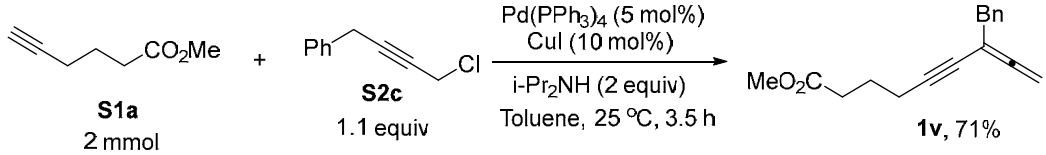
1.18. Preparation of 3-(3-chloropropyl)-8-(methoxycarbonyl)octa-1,2-dien-4-yne **1u**

(syl-8-88).



Following **Typical Procedure I**, the reaction of **S2b** (0.4957 g, 3.3 mmol), $Pd(PPh_3)_4$ (0.1730 g, 0.15 mmol), toluene (4.5 mL), **S1a** (0.39 mL, d = 0.96 g/mL, 0.374 g, 3 mmol), toluene (4.5 mL), CuI (0.0572 g, 0.3 mmol), and *i*-Pr_2NH (0.84 mL, d = 0.722 g/mL, 0.606 g, 6 mmol) afforded **1u** (0.6139 g, 86%) (eluent: petroleum ether (60~90 °C)/ ethyl acetate = (20/1) (420 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 4.98-4.85 (m, 2 H, $=CH_2$), 3.68 (s, 3 H, OCH_3), 3.58 (t, *J* = 6.5 Hz, 2 H, CH_2), 2.45 (t, *J* = 7.5 Hz, 2 H, CH_2), 2.40 (t, *J* = 7.2 Hz, 2 H, CH_2), 2.31-2.18 (m, 2 H, CH_2), 2.04-1.92 (m, 2 H, CH_2), 1.92-1.79 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.4, 173.5, 91.6, 88.3, 77.0, 75.6, 51.5, 44.0, 32.8, 30.7, 30.5, 23.8, 18.9; IR (neat) ν (cm^{-1}) 2953, 2219, 1941, 1737, 1437, 1369, 1314, 1221, 1161; MS (EI) *m/z*: 242 (M^+(^{37}Cl), 1.23), 240 (M^+(^{35}Cl), 3.80), 91 (100); HRMS calcd. for $C_{13}H_{17}O_2{}^{35}Cl$ (M^+): 240.0917; Found: 240.0918.

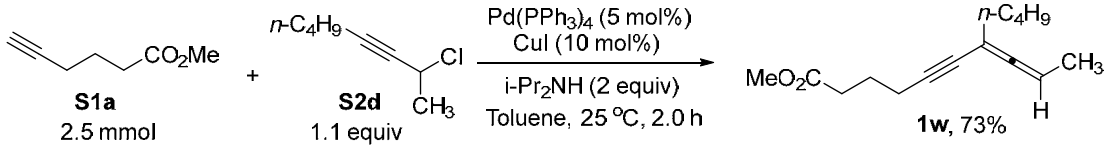
1.19. Preparation of 3-benzyl-8-(methoxycarbonyl)octa-1,2-dien-4-yne **1v** (syl-8-110).



Following **Typical Procedure I**, the reaction of **S2c** (0.3602 g, 2.2 mmol), $Pd(PPh_3)_4$ (0.1151 g, 0.1 mmol), toluene (3 mL), **S1a** (0.26 mL, d = 0.96 g/mL, 0.250 g, 2 mmol), toluene (3 mL), CuI (0.0380 g, 0.2 mmol), and *i*-Pr_2NH (0.56 mL, d = 0.722

g/mL, 0.404 g, 4 mmol) afforded **1v** (0.3560 g, 71%) (eluent: petroleum ether (60~90 °C)/ ethyl acetate = (30/1) (413 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.13 (m, 5 H, ArH), 4.98-4.85 (m, 2 H, $=CH_2$), 3.66 (s, 3 H, OCH_3), 3.45-3.34 (m, 2 H, CH_2), 2.32 (t, *J* = 7.5 Hz, 4 H, CH_2), 1.85-1.71 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.7, 173.5, 138.5, 128.9, 128.1, 126.4, 91.9, 89.6, 76.8, 75.9, 51.4, 40.2, 32.6, 23.7, 18.9; IR (neat) ν (cm^{-1}) 3028, 2950, 2224, 1942, 1737, 1496, 1454, 1435, 1369, 1314, 1220, 1159; MS (EI) *m/z*: 254 (M^+, 1.23), 91 (100); HRMS calcd. for $C_{17}H_{18}O_2$ (M^+): 254.1307; Found: 254.1306.

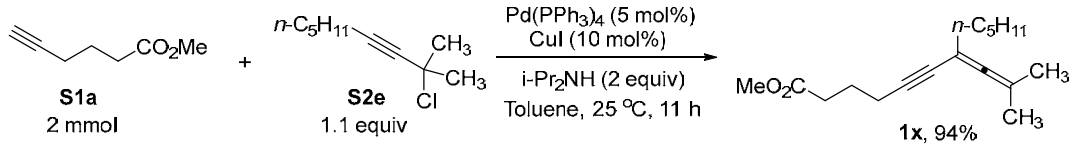
1.20. Preparation of 9-methoxycarbonyl-4-pentylnona-2,3-dien-5-yne **1w** (syl-8-138).



Following **Typical Procedure I**, the reaction of **S2d** (0.3965 g, 2.75 mmol), $Pd(PPh_3)_4$ (0.1446 g, 0.125 mmol), toluene (3.75 mL), **S1a** (0.33 mL, d = 0.96 g/mL, 0.317 g, 2.5 mmol), toluene (3.75 mL), CuI (0.0470 g, 0.25 mmol), and *i*-Pr_2NH (0.70 mL, d = 0.722 g/mL, 0.505 g, 5 mmol) afforded **1w** (0.4321 g, 73%) (eluent: petroleum ether (60~90 °C)/ ethyl acetate = (30/1) (465 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.30-5.18 (m, 1 H, =CH), 3.66 (s, 3 H, OCH_3), 2.43 (t, *J* = 7.5 Hz, 2 H, CH_2), 2.37 (t, *J* = 6.9 Hz, 2 H, CH_2), 2.10-1.97 (m, 2 H, CH_2), 1.91-1.77 (m, 2 H, CH_2), 1.66 (d, *J* = 7.2 Hz, 3 H, CH_3), 1.50-1.23 (m, 4 H, CH_2 × 2), 0.89 (t, *J* = 7.1 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 209.2, 173.6, 89.6, 89.3, 87.1, 77.3, 51.5, 33.8, 32.8, 29.8, 23.9, 21.9, 19.0, 14.2, 13.8; IR (neat) ν (cm^{-1}) 2955, 2929, 2872, 2215, 1950, 1740,

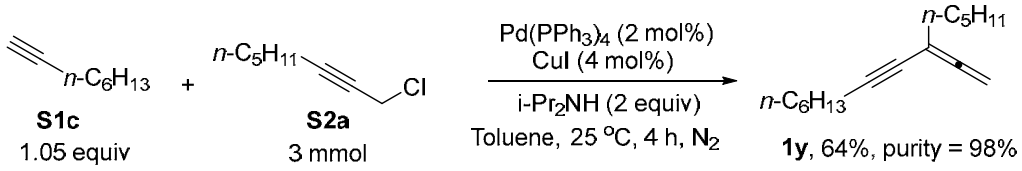
1452, 1436, 1370, 1318, 1250, 1220, 1161; MS (EI) *m/z*: 234 (M^+, 2.54), 117 (100); HRMS calcd. for $C_{15}H_{22}O_2$ (M^+): 234.1620; Found: 234.1619.

1.21. Preparation of 2-methyl-9-(methoxycarbonyl)-4-pentylnona-2,3-dien-5-yne **1x** (syl-8-171).



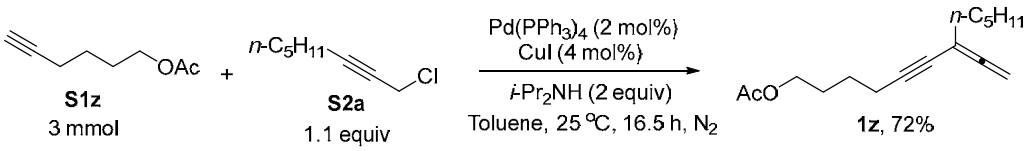
Following **Typical Procedure I**, the reaction of **S2e** (0.3788 g, 2.2 mmol), $Pd(PPh_3)_4$ (0.1160 g, 0.1 mmol), toluene (3 mL), **S1a** (0.26 mL, d = 0.96 g/mL, 0.250 g, 2.0 mmol), toluene (3 mL), CuI (0.0381 g, 0.2 mmol), and *i*-Pr_2NH (0.56 mL, d = 0.722 g/mL, 0.404 g, 4 mmol) afforded **1x** (0.4885 g, 94%) (eluent: petroleum ether (60~90 °C)/ ethyl acetate = (30/1) (372 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 3.68 (s, 3 H, OCH_3), 2.45 (t, *J* = 7.5 Hz, 2 H, CH_2), 2.38 (t, *J* = 7.1 Hz, 2 H, CH_2), 2.04 (t, *J* = 7.2 Hz, 2 H, CH_2), 1.93-1.80 (m, 2 H, CH_2), 1.71 (s, 6 H, CH_3 × 2), 1.51-1.39 (m, 2 H, CH_2), 1.37-1.23 (m, 4 H, CH_2 × 2), 0.89 (t, *J* = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.0, 173.7, 96.7, 88.4, 87.5, 78.0, 51.5, 34.4, 32.9, 30.9, 27.4, 24.0, 22.4, 20.4, 19.0, 14.0; IR (neat) ν (cm^{-1}) 2932, 2858, 2219, 1955, 1742, 1437, 1363, 1319, 1220, 1160, 1106, 1058; MS (EI) *m/z*: 262 (M^+, 1.01), 133 (100); HRMS calcd. for $C_{17}H_{26}O_2$ (M^+): 262.1933; Found: 262.1934.

1.22. Preparation of 3-pentylundeca-1,2-dien-4-yne **1y** (zj-5-23, syl-10-22).



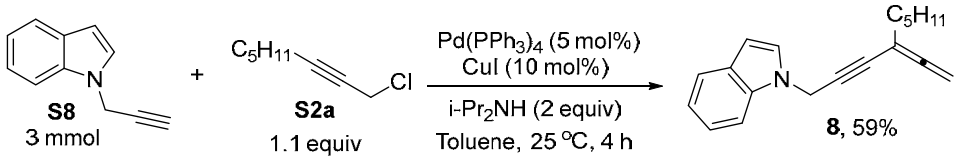
Typical Procedure II: To a Schlenk flask were added **S2a** (0.4321 g, 3.0 mmol), $Pd(PPh_3)_4$ (0.0699 g, 0.06 mmol), and toluene (4.5 mL) under N_2 atmosphere. The resulting mixture was stirred at 25 ºC for 5 minutes followed by the addition of **S1c** (0.3415 g, 3.15 mmol), toluene (4.5 mL), CuI (0.0228 g, 0.12 mmol), and *i*-Pr_2NH (0.6075 g, 6 mmol) sequentially under N_2. The resulting mixture was stirred at 25 ºC for 4 hours as monitored by TLC and then filtrated through a pad of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford impure **1y** (525.6 mg) (eluent: petroleum ether (60~90 ºC) (350 mL)), which was further purified by chromatography on silica gel to afford **1y** (429.5 mg, 98% purity, 64%) (eluent: petroleum ether (60~90 ºC) (300 mL)): liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.90-4.84 (m, 2 H, $=CH_2$), 2.31 (tt, J_1 = 7.1 Hz, J_2 = 1.2 Hz, 2 H, CH_2), 2.13-2.01 (m, 2 H, CH_2), 1.58-1.45 (m, 4 H, CH_2 × 2), 1.42-1.22 (m, 10 H, CH_2 × 5), 0.93-0.85 (m, 6 H, CH_3 × 2); ^{13}C NMR (125 MHz, $CDCl_3$) δ 213.4, 92.8, 90.0, 76.2, 75.3, 33.6, 31.3, 31.1, 28.8, 28.5, 27.4, 22.5, 22.4, 19.6, 14.0; IR (neat) ν (cm^{-1}) 2956, 2930, 2858, 2215, 1942; MS (EI) *m/z*: 218 (M^+, 0.31), 189 (M-Et)$^+$, 10.10), 91 (100); HRMS calcd. for $C_{16}H_{26}$ (M^+): 218.2035; Found: 218.2035.

1.23. Preparation of 9-acetoxy-3-pentylnona-1,2-dien-4-yne **1z** (syl-10-34).



Following **Typical Procedure II**, the reaction of **S2a** (0.51 mL, d = 0.931 g/mL, 0.475 g, 3.3 mmol), $Pd(PPh_3)_4$ (0.0690 g, 0.06 mmol), toluene (4.5 mL), **S1z** (0.4215 g, 3 mmol), toluene (4.5 mL), CuI (0.0235 g, 0.12 mmol), and *i*-Pr_2NH (0.84 mL, d = 0.722 g/mL, 0.606 g, 6 mmol) afforded **1z** (0.5383 g, 72%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 30/1 (372 mL)): liquid; ^1H NMR (500 MHz, $CDCl_3$) δ 4.93-4.87 (m, 2 H, $=CH_2$), 4.09 (t, *J* = 6.5 Hz, 2 H, OCH_2), 2.37 (t, *J* = 7.3 Hz, 2 H, CH_2), 2.12-2.02 (m, 5 H, CH_2 + CH_3), 1.78-1.72 (m, 2 H, CH_2), 1.66-1.57 (m, 2 H, CH_2), 1.54-1.45 (m, 2 H, CH_2), 1.37-1.25 (m, 4 H, CH_2 × 2), 0.89 (t, *J* = 7.0 Hz, 3 H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 213.4, 171.1, 91.7, 89.8, 76.3, 75.9, 64.0, 33.5, 31.1, 27.8, 27.3, 25.2, 22.4, 20.9, 19.2, 14.0; IR (neat) ν (cm^{-1}) 2956, 2925, 2860, 2219, 1942, 1742, 1241, 1047; MS (EI) *m/z*: 248 (M^+, 1.54), 117 (100); HRMS calcd. for $C_{16}H_{24}NaO_2$ (M^+ + Na): 271.1669; Found: 271.1670.

1.24. Preparation of 6-(1*H*-indol-1-yl)-3-pentylhexa-1,2-dien-4-yne **8** (syl-8-39).

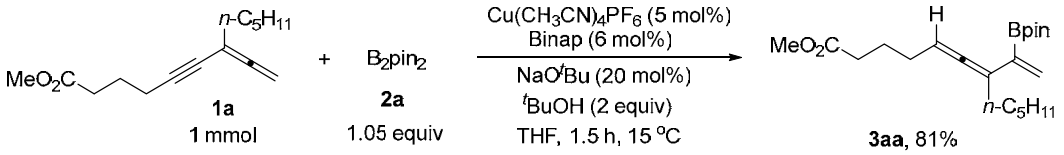


Following **Typical Procedure I**, the reaction of **S2a** (0.51 mL, d = 0.931 g/mL, 0.475 g, 3.3 mmol), $Pd(PPh_3)_4$ (0.1770 g, 0.15 mmol), toluene (4.5 mL), **S8** (0.4651 g, 3 mmol), toluene (4.5 mL), CuI (0.0565 g, 0.3 mmol), and *i*-Pr_2NH (0.84 mL, d = 0.722 g/mL, 0.606 g, 6 mmol) afforded pure **8** (0.2330 g) and impure product (eluent:

petroleum ether (60~90 °C)/ethyl ether = 100/1/ (606 mL)). The impure product was further purified by chromatography on silica gel (eluent: petroleum ether (60~90 °C)/DCM = 10/1 (440 mL)) to afford pure **8** (0.2318 g). The two parts of the product were combined to produce pure **8** (0.4648 g, 59%): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.61 (d, *J* = 7.8 Hz, 1 H, ArH), 7.36 (d, *J* = 8.1 Hz, 1 H, ArH), 7.26-7.02 (m, 3 H, ArH), 6.49 (d, *J* = 3.0 Hz, 1 H, ArH), 4.95-4.80 (m, 4 H, $=CH_2$ + NCH_2), 2.13-1.97 (m, 2 H, CH_2), 1.55-1.38 (m, 2 H, CH_2), 1.35-1.18 (m, 4 H, CH_2 × 2), 0.86 (t, *J* = 6.3 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 213.6, 135.7, 128.8, 127.2, 121.6, 120.9, 119.6, 109.4, 101.6, 88.9, 85.1, 80.6, 76.9, 36.6, 33.0, 31.0, 27.3, 22.3, 14.0; IR (neat) ν (cm^{-1}) 3055, 2955, 2928, 2857, 2226, 1940, 1614, 1513, 1483, 1463, 1431, 1399, 1362, 1337, 1314, 1257, 1187; MS (EI) *m/z*: 263 (M^+, 100); HRMS calcd. for $C_{19}H_{21}N$ (M^+): 263.1674; Found: 263.1673.

2. Synthesis of 1,3,4-trienyl boronates.

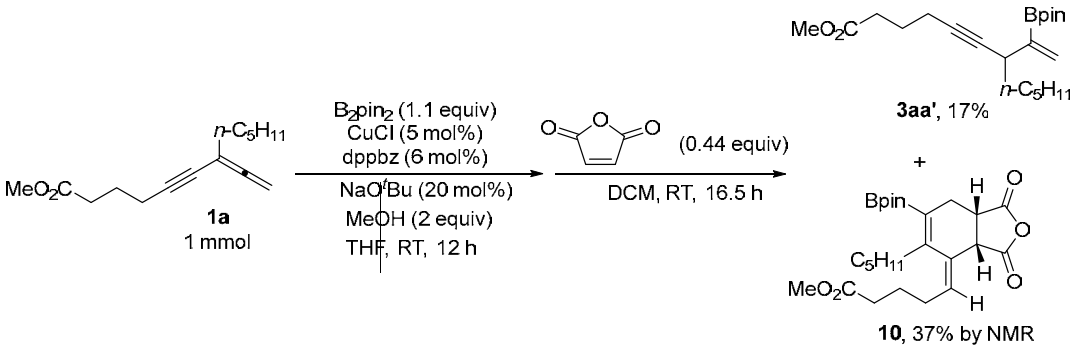
2.1. Preparation of 4,4,5,5-tetramethyl-2-(8-(methoxycarbonyl-)3-pentylocta-1,3,4-trien-2-yl)-1,3,2-dioxaborolane **3aa** (syl-7-192).



Typical Procedure III: To a flame-dried Schlenk tube were added B_2Pin_2 (0.2669 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0189 g, 0.05 mmol), BINAP (0.0372 g, 0.06 mmol), NaO^tBu (0.0190 g, 0.2 mmol), ^{t}BuOH (0.19 mL, d = 0.775 g/mL, 0.147 g, 2 mmol), **1a** (0.2339 g, 1 mmol), and THF (5 mL) under N_2 atmosphere. The resulting mixture was stirred at 15 °C for 1.5 hours as monitored by TLC. The resulting mixture was filtrated

through a pad of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent under reduced pressure, the yield of the crude product was determined by ^{1}H NMR analysis using mesitylene (46 μL) as the internal standard (87% by NMR). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **3aa** (0.2932 g, 81%) (eluent: petroleum ether (60~90 ºC)/ethyl acetate = 20/1 (315 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.64 (s, 1 H, one proton of $=CH_2$), 5.59 (s, 1 H, one proton of $=CH_2$), 5.27-5.18 (m, 1 H, =CH), 3.67 (s, 3 H, OCH_3), 2.37 (t, *J* = 7.5 Hz, 2 H, CH_2), 2.20-2.00 (m, 4 H, CH_2 × 2), 1.84-1.70 (m, 2 H, CH_2), 1.50-1.37 (m, 2 H, CH_2), 1.35-1.20 (m, 16 H, CH_2 × 2 + CH_3 × 4), 0.88 (t, *J* = 6.5 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.9, 174.0, 124.2, 106.8, 90.9, 83.4, 51.4, 33.6, 31.7, 29.3, 28.5, 27.3, 24.7, 24.6, 24.3, 22.5, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2976, 2955, 2930, 2859, 1943, 1741, 1585, 1371, 1312, 1214, 1145, 1112; MS (EI) *m/z*: 362 (M^+(^{11}B), 37.44), 361 (M^+(^{10}B), 9.42), 83 (100); HRMS calcd. for $C_{21}H_{35}O_4{}^{11}B$ (M^+): 362.2628; Found: 362.2628.

Preparation of 4,4,5,5-tetramethyl-2-(8-(methoxycarbonyl)-3-pentylocta-1-en-4-yn-2-yl)-1,3,2-dioxaborolane **3aa'** (syl-9-90, syl-9-92)



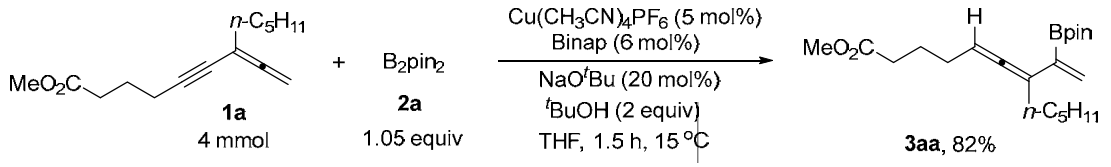
To a flame-dried Schlenk tube were added B_2Pin_2 (0.2795 g, 1.1 mmol), CuCl

(0.0053 g, 0.05 mmol), dppbz (0.0267 g, 0.06 mmol), NaO^{t}Bu (0.0194 g, 0.2 mmol), MeOH (81 μL, d = 0.793 g/mL, 0.0642 g, 2 mmol), **1a** (0.2345 g, 1 mmol), and THF (5 mL) under N_2 atmosphere. The resulting mixture was stirred at room temperature for 12 hours as monitored by TLC. The resulting mixture was filtrated through a pad of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford mixture (eluent: petroleum ether (60~90 ºC)/ethyl acetate = 20/1 (315 mL)).

To a flame-dried Schlenk tube were added the above-prepared mixture, DCM (4 mL), and maleic anhydride (0.0428 g, 0.44 mmol) under N_2 atmosphere. The resulting mixture was stirred at room temperature for 16.5 h as monitored by TLC. After evaporation of the solvent under reduced pressure, the yield of the crude product was determined by ^{1}H NMR analysis using mesitylene (18.4 μL) as the internal standard (37% yield for **10** by NMR and 17% yield for **3aa'** by NMR). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford pure **3aa'** (0.0620 g, 17% yield from **1a**) (eluent: petroleum ether/ethyl acetate = 20/1 (315 mL)): liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.95 (s, 1 H, one proton of $=CH_2$), 5.87 (d, J = 2.8 Hz, 1 H, one proton of $=CH_2$), 3.67 (s, 3 H, OCH_3), 3.28-3.21 (m, 1 H, CH), 2.46 (t, J = 7.4 Hz, 2 H, CH_2), 2.28 (td, J_1 = 6.8 Hz, J_2 = 2.0 Hz, 2 H, CH_2), 1.88-1.78 (m, 2 H, CH_2), 1.69-1.55 (m, 1 H, one proton of CH_2), 1.47-1.20 (m, 19 H, one proton of CH_2 + CH_2 × 3 + CH_3 × 4), 0.88 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.8, 129.0, 83.4, 82.5, 82.1, 51.5, 36.3, 35.6, 32.8, 31.5, 26.8, 24.9, 24.4, 24.3, 22.5, 18.3, 14.0; The carbon atom directly attached to the boron atom was not detected; Ram

(neat) ν (cm^{-1}) 2932, 2874, 2229, 1743, 1615, 1438; MS (EI) *m/z*: 362 (M^+(^{11}B), 3.71), 361 (M^+(^{10}B), 1.46), 331 (100); HRMS calcd. for $C_{21}H_{35}O_4{}^{11}B$ (M^+): 362.2628; Found: 362.2629.

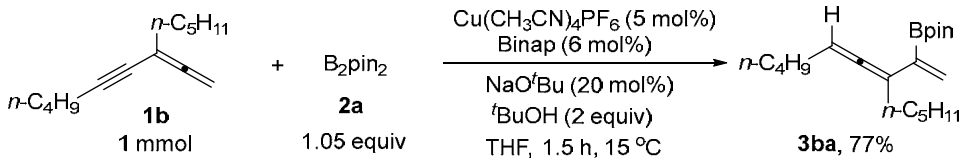
Gram-scale reaction (syl-8-99)**:**



To a flame-dried Schlenk tube were added B_2Pin_2 (1.0670 g, 4.2 mmol), $Cu(CH_3CN)_4PF_6$ (0.0747 g, 0.2 mmol), BINAP (0.1491 g, 0.24 mmol), NaO^tBu (0.0769 g, 0.8 mmol), ^{t}BuOH (0.73 mL, d = 0.81 g/mL, 0.591 g, 8 mmol), **1a** (0.9365 g, 4 mmol), and THF (20 mL) under N_2 atmosphere. The resulting mixture was stirred at 15 °C for 1.5 hours as monitored by TLC and filtrated through a pad of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **3aa** (1.1921 g, 82%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 20/1 (420 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.64 (s, 1 H, one proton of $=CH_2$), 5.59 (s, 1 H, one proton of $=CH_2$), 5.26-5.18 (m, 1 H, =CH), 3.67 (s, 3 H, OCH_3), 2.37 (t, *J* = 7.4 Hz, 2 H, CH_2), 2.18-2.00 (m, 4 H, CH_2 × 2), 1.85-1.72 (m, 2 H, CH_2), 1.50-1.20 (m, 18 H, CH_2 × 3 + CH_3 × 4), 0.88 (t, *J* = 6.5 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.9, 174.0, 124.3, 106.8, 91.0, 83.4, 51.4, 33.6, 31.7, 29.4, 28.5, 27.3, 24.7, 24.6, 24.3, 22.5, 14.1.

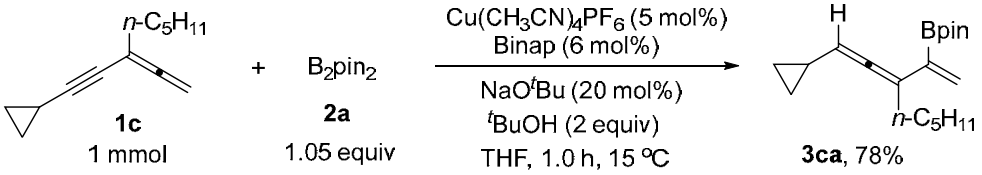
2.2. Preparation of 4,4,5,5-tetramethyl-2-(3-pentylnona-1,3,4-trien-2-yl)-1,3,2-

dioxaborolane **3ba** (syl-8-2).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2666 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0187 g, 0.05 mmol), BINAP (0.0372 g, 0.06 mmol), NaO^tBu (0.0193 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1b** (0.1901 g, 1 mmol), and THF (5 mL) afforded **3ba** (0.2457 g, 77%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 100/1 (505 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.61 (s, 1 H, one proton of $=CH_2$), 5.55 (s, 1 H, one proton of $=CH_2$), 5.28-5.19 (m, 1 H, =CH), 2.17-2.10 (m, 2 H, CH_2), 2.02 (q, *J* = 6.9 Hz, 2 H, CH_2), 1.52-1.18 (m, 22 H, $CH_2 \times 5 + CH_3 \times 4$), 0.95-0.82 (m, 6 H, $CH_3 \times 2$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.8, 123.4, 106.3, 92.0, 83.4, 31.7, 31.3, 29.2, 28.8, 27.3, 24.7, 24.6, 22.6, 22.3, 14.0, 13.9; The carbon directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2958, 2929, 2873, 2859, 1943, 1585, 1371, 1310, 1146, 1111; MS (EI) *m/z*: 319 (($M(^{11}B) + H)^+$, 12.48), 318 ($M^+(^{11}B)$, 51.57), 317 ($M^+(^{10}B)$, 12.64), 83 (100); HRMS calcd. for $C_{20}H_{35}{}^{11}BO_2$ (M^+): 318.2730; Found: 318.2730.

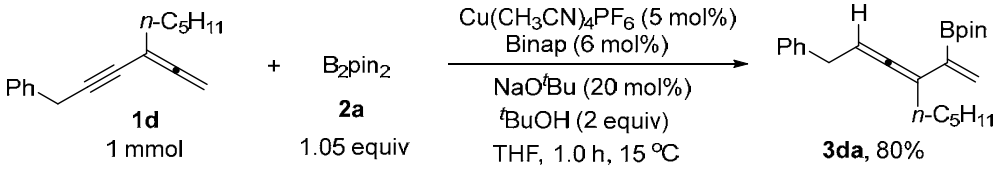
2.3. Preparation of 2-(5-cyclopropyl-3-pentylpenta-1,3,4-trien-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **3ca** (syl-8-18).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2670 g, 1.05 mmol),

$Cu(CH_3CN)_4PF_6$ (0.0190 g, 0.05 mmol), BINAP (0.0377 g, 0.06 mmol), NaO^tBu (0.0192 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1c** (0.1743 g, 1 mmol), and THF (5 mL) afforded **3ca** (0.2371 g, 78%) (eluent: petroleum ether (60~90 ºC)/DCM = 8/1 (500 mL) to 5/1 (280 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.62 (s, 1 H, one proton of $=CH_2$), 5.55 (s, 1 H, one proton of $=CH_2$), 5.08 (d, *J* = 7.5 Hz, 1 H, =CH), 2.23-2.08 (m, 2 H, CH_2), 1.53-1.36 (m, 2 H, CH_2), 1.36-1.15 (m, 17 H, $CH_3 \times 4 + CH_2 \times 2 + CH$), 0.88 (t, *J* = 6.8 Hz, 3 H, CH_3), 0.72-0.60 (m, 2 H, CH_2), 0.45-0.28 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.4, 123.7, 108.1, 96.2, 83.4, 31.7, 29.4, 27.3, 24.7, 24.6, 22.5, 14.0, 9.7, 6.5, 6.3; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 3080, 2978, 2958, 2930, 2859, 1942, 1585, 1371, 1312, 1214, 1145, 1112; MS (EI) *m/z*: 302 (M^+(^{11}B), 35.00), 301 (M^+(^{10}B), 9.04), 80 (100); HRMS calcd. for $C_{19}H_{31}{}^{11}BO_2$ (M^+): 302.2417; Found: 302.2417.

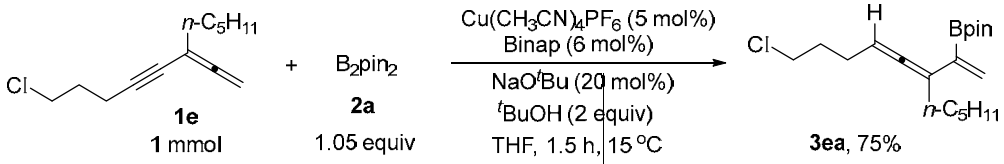
2.4. Preparation of 4,4,5,5-tetramethyl-2-(3-pentyl-6-phenylhexa-1,3,4-trien-2-yl)-1,3,2-dioxaborolane **3da** (syl-8-15).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2670 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0188 g, 0.05 mmol), BINAP (0.0376 g, 0.06 mmol), NaO^tBu (0.0190 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1d** (0.2248 g, 1 mmol), and THF (5 mL) afforded **3da** (0.2832 g, 80%) (eluent: petroleum ether

(60~90 °C)/DCM = 8/1 (450 mL) to 5/1 (480 mL) to 4/1 (500 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32-7.13 (m, ArH, 5 H), 5.65 (s, 1 H, one proton of $=CH_2$), 5.62 (s, 1 H, one proton of $=CH_2$), 5.45-5.35 (m, 1 H, =CH), 3.48-3.27 (m, 2 H, CH_2), 2.22-2.08 (m, 2 H, CH_2), 1.50-1.20 (m, 18 H, $CH_2 \times 3 + CH_3 \times 4$), 0.87 (t, J = 5.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.1, 140.7, 128.5, 128.2, 125.9, 124.8, 106.8, 91.3, 83.4, 35.8, 31.7, 29.5, 27.3, 24.7, 24.6, 22.5, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 3063, 3027, 2977, 2957, 2929, 2858, 1943, 1603, 1585, 1495, 1371, 1311, 1145, 1112; MS (EI) *m/z*: 352 (M^+(^{11}B), 39.35), 351 (M^+(^{10}B), 10.39), 91 (100); HRMS calcd. for $C_{23}H_{33}{}^{11}BO_2$ (M^+): 352.2574; Found: 352.2573.

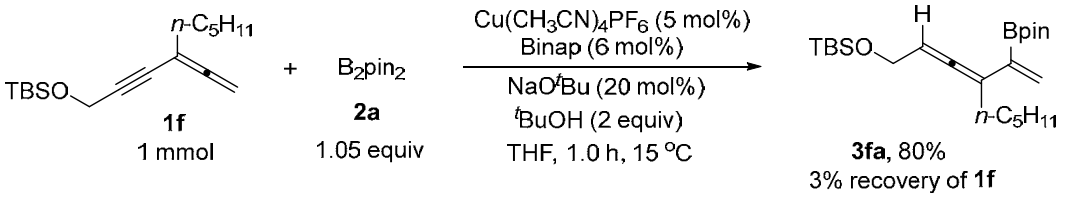
2.5. Preparation of 2-(8-chloro-3-pentylocta-1,3,4-trien-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **3ea** (syl-7-193).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2670 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0186 g, 0.05 mmol), BINAP (0.0372 g, 0.06 mmol), NaO^tBu (0.0192 g, 0.2 mmol), ^{t}BuOH (0.19 mL, d = 0.775 g/mL, 0.147 g, 2 mmol), **1e** (0.2106 g, 1 mmol), and THF (5 mL) afforded **3ea** (0.2528 g, 75%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 50/1 (306 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.64 (s, 1 H, one proton of $=CH_2$), 5.61 (s, 1 H, one proton of $=CH_2$), 5.32-5.22 (m, 1 H, =CH), 3.59 (t, J = 6.5 Hz, 2 H, CH_2), 2.25-2.08 (m, 4 H, $CH_2 \times 2$), 1.98-1.85 (m, 2 H,

CH_2), 1.50-1.22 (m, 18 H, $CH_2 \times 3 + CH_3 \times 4$), 0.89 (t, J = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.8, 124.3, 107.1, 90.4, 83.3, 44.6, 31.6, 31.5, 29.2, 27.2, 26.0, 24.62, 24.56, 22.5, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2977, 2958, 2930, 2859, 1943, 1585, 1371, 1312, 1213, 1144, 1112; MS (EI) *m/z*: 340 (M^+(^{37}Cl)(^{11}B), 13.47), 339 (M^+(^{37}Cl)(^{10}B), 11.19), 338 (M^+(^{35}Cl)(^{11}B), 38.08), 337 (M^+(^{35}Cl)(^{10}B), 9.06), 83 (100); HRMS calcd. for $C_{19}H_{32}{}^{11}BO_2{}^{35}Cl$ (M^+): 338.2184; Found: 338.2185.

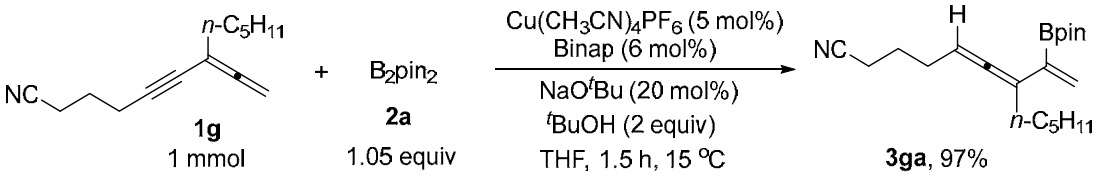
2.6. Preparation of 2-(6-(tert-butyldimethylsilyl)oxy-3-pentylhexa-1,3,4-trien-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **3fa** (syl-7-200, syl-8-76).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2670 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0187 g, 0.05 mmol), BINAP (0.0375 g, 0.06 mmol), NaO^tBu (0.0190 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1f** (0.2782 g, 1 mmol), and THF (5 mL) afforded **3fa** (0.3242 g, 80%) (eluent: petroleum ether (60~90 °C)/DCM = 7/1 (400 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 5.66 (s, 1 H, one proton of $=CH_2$), 5.61 (s, 1 H, one proton of $=CH_2$), 5.38-5.30 (m, 1 H, =CH), 4.28-4.12 (m, 2 H, OCH_2), 2.23-2.08 (m, 2 H, CH_2), 1.50-1.37 (m, 2 H, CH_2), 1.36-1.20 (m, 16 H, $CH_2 \times 2 + CH_3 \times 4$), 0.93-0.80 (m, 12 H, $CH_3 \times 4$), 0.06 (s, 6 H, $CH_3 \times 2$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.6, 125.0, 107.7, 92.8, 83.5, 62.0, 31.7, 29.4, 27.4, 25.9, 24.7, 24.6, 22.5, 18.3, 14.1, -5.1, -5.2; The carbon atom directly attached to the boron

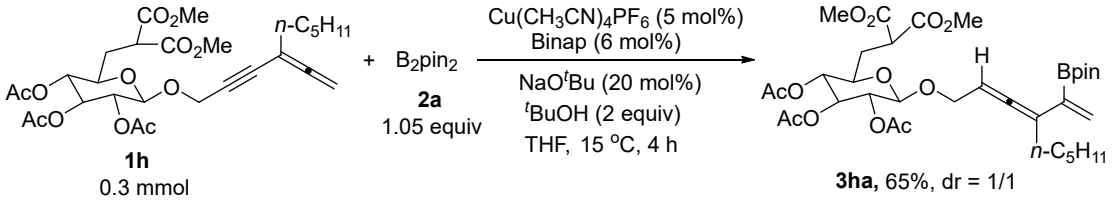
atom was not detected; IR (neat) ν (cm^{-1}) 2956, 2929, 2857, 1946, 1587, 1471, 1459, 1371, 1312, 1255, 1146, 1090, 1004; MS (EI) *m/z*: 406 (M^+(^{11}B), 17.46), 405 (M^+(^{10}B), 4.93), 83 (100); HRMS calcd. for $C_{23}H_{43}{}^{11}BO_3Si$ (M^+): 406.3075; Found: 406.3077.

2.7. Preparation of 2-(8-cyano-3-pentylocta-1,3,4-trien-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **3ga** (syl-7-198).



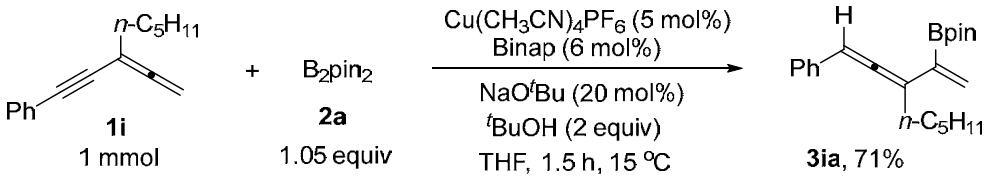
Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2668 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0188 g, 0.05 mmol), BINAP (0.0375 g, 0.06 mmol), NaO^tBu (0.0190 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1g** (0.2012 g, 1 mmol), and THF (5 mL) afforded **3ga** (0.3192 g, 97%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 15/1 (320 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.65 (d, *J* = 5.7 Hz, 2 H, $=CH_2$), 5.29-5.19 (m, 1 H, =CH), 2.41 (t, *J* = 6.9 Hz, 2 H, CH_2), 2.23-2.08 (m, 4 H, CH_2 × 2), 1.90-1.74 (m, 2 H, CH_2), 1.50-1.20 (m, 18 H, CH_2 × 3 + CH_3 × 4), 0.89 (t, *J* = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.8, 125.0, 119.7, 107.6, 89.8, 83.5, 31.7, 29.4, 27.6, 27.4, 24.7, 24.6, 24.4, 22.5, 16.5, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2958, 2931, 2860, 2247, 1943, 1585, 1372, 1313, 1214, 1144; MS (EI) *m/z*: 329 (M^+(^{11}B), 34.75), 328 (M^+(^{10}B), 14.68), 272 (100); HRMS calcd. for $C_{20}H_{32}{}^{11}BO_2N$ (M^+): 329.2526; Found: 329.2528.

2.8. Preparation of **3ha** (syl-8-74).



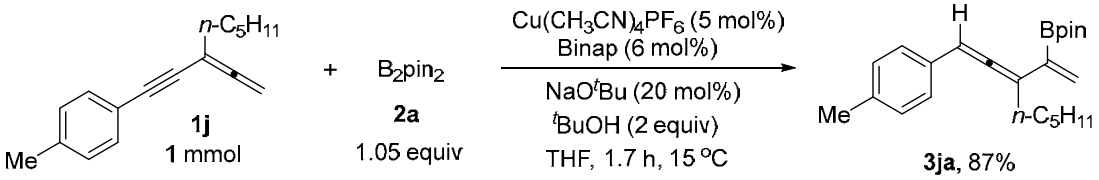
Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.0803 g, 0.315 mmol), $Cu(CH_3CN)_4PF_6$ (0.0058 g, 0.015 mmol), BINAP (0.0114 g, 0.018 mmol), NaO^tBu (0.0061 g, 0.06 mmol), ^{t}BuOH (0.055 mL, d = 0.81 g/mL, 0.0446 g, 0.6 mmol), **1h** (0.1705 g, 0.3 mmol), and THF (1.5 mL) afforded **3ha** (0.1367 g, 65%) (eluent: petroleum ether (60~90 °C)/ethyl acetate/DCM = 7/1/1 (630 mL) to 5/1/1 (560 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.71 (s, 1 H, one proton of $=CH_2$), 5.68 (s, 1 H, one proton of $=CH_2$), 5.37-5.22 (m, 1 H, =CH), 5.15 (t, *J* = 9.3 Hz, 1 H, OCH), 4.96 (t, *J* = 9.0 Hz, 1 H, one proton of OCH_2), 4.88 (t, *J* = 9.6 Hz, 1 H, one proton of OCH_2), 4.52 (d, *J* = 7.8 Hz, 1 H, OCH), 4.40-4.23 (m, 1 H, OCH), 4.18-4.00 (m, 1 H, OCH), 3.82-3.62 (m, 7 H, OCH_3 × 2 + OCH), 3.51 (t, *J* = 9.6 Hz, 1 H, CH), 2.31-1.94 (m, 13 H, CH_3 × 3 + CH_2 × 2), 1.54-1.20 (m, 18 H, CH_3 × 4 + CH_2 × 3), 0.98-0.82 (m, 3 H, CH_3); IR (neat) ν (cm^{-1}) 2956, 2929, 2860, 1946, 1758, 1436, 1372, 1315, 1244, 1217, 1162, 1145, 1068, 1044; The carbon atom directly attached to the boron atom was not detected; MS (EI) *m/z*: 360 ((M - OAc × 3 - Me × 2 - Bpin)$^+$, 9.91), 126 (100); HRMS calcd. for $C_{34}H_{51}{}^{11}BNaO_{14}^+$ (M^+ + Na): 717.3264; Found: 717.3265.

2.9. Preparation of 4,4,5,5-tetramethyl-2-(3-pentyl-5-phenylpenta-1,3,4-trien-2-yl)-1,3,2-dioxaborolane **3ia** (syl-8-7).



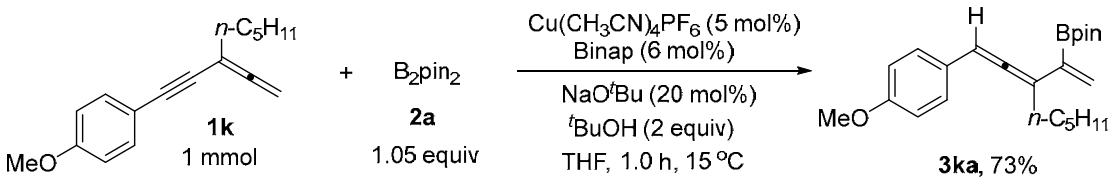
Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2665 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0190 g, 0.05 mmol), BINAP (0.0376 g, 0.06 mmol), NaO^tBu (0.0190 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1i** (0.2103 g, 1 mmol), and THF (5 mL) afforded **3ia** (0.2418 g, 71%) (eluent: petroleum ether (60~90 °C)/DCM = 6/1 (560 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37-7.22 (m, 4 H, ArH), 7.19-7.11 (m, 1 H, ArH), 6.32-6.24 (m, 1 H, =CH), 5.76 (s, 1 H, one proton of $=CH_2$), 5.68 (s, 1 H, one proton of $=CH_2$), 2.36-2.23 (m, 2 H, CH_2), 1.58-1.44 (m, 2 H, CH_2), 1.39-1.25 (m, 4 H, $CH_2 \times 2$), 1.17 (s, 6 H, $CH_3 \times 2$), 1.13 (s, 6 H, $CH_3 \times 2$), 0.84 (t, J = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.1, 135.3, 128.3, 127.0, 126.4, 125.2, 110.4, 95.8, 83.6, 31.7, 29.6, 27.2, 24.6, 24.5, 22.5, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2977, 2958, 2929, 2859, 1928, 1598, 1586, 1414, 1389, 1315, 1213, 1197, 1144, 1112; MS (EI) *m/z*: 338 (M^+(^{11}B), 100), 337 (M^+(^{10}B), 30.14); HRMS calcd. for $C_{22}H_{31}{}^{11}BO_2$ (M^+): 338.2417; Found: 338.2415.

2.10. Preparation of 4,4,5,5-tetramethyl-2-(3-pentyl-5-(p-tolyl)penta-1,3,4-trien-2-yl)-1,3,2-dioxaborolane **3ja** (syl-8-32).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2663 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0188 g, 0.05 mmol), BINAP (0.0375 g, 0.06 mmol), NaO^tBu (0.0195 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1j** (0.2242 g, 1 mmol), and THF (5 mL) afforded **3ja** (0.3052 g, 87%) (eluent: petroleum ether (60~90 °C)/DCM = 5/1 (600 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.21 (d, *J* = 7.8 Hz, 2 H, ArH), 7.08 (d, *J* = 7.8 Hz, 2 H, ArH), 6.30-6.20 (m, 1 H, =CH), 5.74 (s, 1 H, one proton of $=CH_2$), 5.66 (s, 1 H, one proton of $=CH_2$), 2.37-2.20 (m, 5 H, CH_3 + CH_2), 1.57-1.40 (m, 2 H, CH_2), 1.37-1.25 (m, 4 H, CH_2 × 2), 1.17 (s, 6 H, CH_3 × 2), 1.14 (s, 6 H, CH_3 × 2), 0.84 (t, *J* = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.8, 136.1, 132.2, 129.0, 126.8, 124.9, 110.2, 95.6, 83.5, 31.7, 29.5, 27.2, 24.6, 24.5, 22.5, 21.1, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2977, 2951, 2928, 2859, 1929, 1588, 1513, 1410, 1315, 1214, 1196, 1145, 1111; MS (EI) *m/z*: 352 (M^+(^{11}B), 100), 351 (M^+(^{10}B), 26.46); HRMS calcd. for $C_{23}H_{33}{}^{11}BO_2$ (M^+): 352.2574; Found: 352.2575.

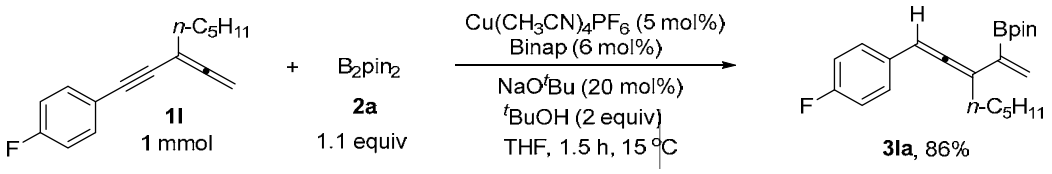
2.11. Preparation of 4,4,5,5-tetramethyl-2-(5-(4-methoxyphenyl)-3-pentylpenta-1,3,4-trien-2-yl)-1,3,2-dioxaborolane **3ka** (syl-8-25).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2670 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0188 g, 0.05 mmol), BINAP (0.0376 g, 0.06 mmol), NaO^tBu (0.0193 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1k** (0.2401

g, 1 mmol), and THF (5 mL) afforded **3ka** (0.2688 g, 73%) (eluent: petroleum ether (60~90 ºC)/DCM = 5/1 (840 mL) to 3/1 (240 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.24 (d, *J* = 8.4 Hz, 2 H, ArH), 6.83 (d, *J* = 8.7 Hz, 2 H, ArH), 6.28-6.18 (m, 1 H, =CH), 5.73 (s, 1 H, one proton of $=CH_2$), 5.64 (s, 1 H, one proton of $=CH_2$), 3.79 (s, 3 H, OCH_3), 2.35-2.20 (m, 2 H, CH_2), 1.57-1.42 (m, 2 H, CH_2), 1.37-1.25 (m, 4 H, CH_2 × 2), 1.17 (s, 6 H, CH_3 × 2), 1.14 (s, 6 H, CH_3 × 2), 0.84 (t, *J* = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.6, 158.4, 128.0, 127.6, 124.6, 113.7, 110.3, 95.2, 83.5, 55.2, 31.7, 29.5, 27.2, 24.6, 24.5, 22.5, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2977, 2956, 2930, 2858, 1928, 1608, 1587, 1511, 1466, 1410, 1358, 1315, 1248, 1170, 1144, 1108, 1036; MS (EI) *m/z*: 368 (M^+(^{11}B), 100), 367 (M^+(^{10}B), 32.77); HRMS calcd. for $C_{23}H_{33}{}^{11}BO_3$ (M^+): 368.2523; Found: 368.2523.

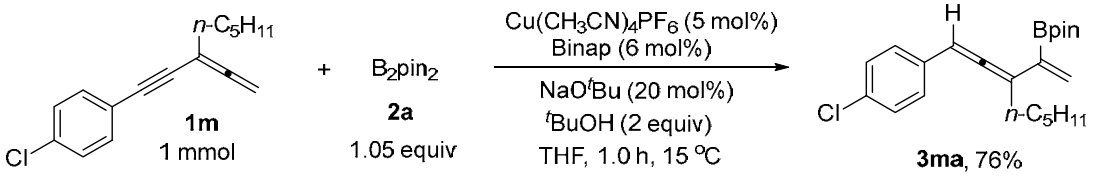
2.12. Preparation of 2-(5-(4-fluorophenyl)-3-pentylpenta-1,3,4-trien-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **3la** (syl-8-49).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2798 g, 1.1 mmol), $Cu(CH_3CN)_4PF_6$ (0.0188 g, 0.05 mmol), BINAP (0.0377 g, 0.06 mmol), NaO^tBu (0.0192 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1l** (0.2275 g, 1 mmol), and THF (5 mL) afforded **3la** (0.3047 g, 86%) (eluent: petroleum ether (60~90 ºC)/DCM = 5/1 (600 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33-7.22 (m,

2 H, ArH), 6.97 (t, J = 8.7 Hz, 2 H, ArH), 6.27-6.18 (m, 1 H, =CH), 5.76 (s, 1 H, one proton of $=CH_2$), 5.69 (s, 1 H, one proton of $=CH_2$), 2.35-2.23 (m, 2 H, CH_2), 1.56-1.40 (m, 2 H, CH_2), 1.35-1.25 (m, 4 H, $CH_2 \times 2$), 1.17 (s, 6 H, $CH_3 \times 2$), 1.13 (s, 6 H, $CH_3 \times 2$), 0.84 (t, J = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.0 (d, J = 2.0 Hz), 161.7 (d, J = 243.4 Hz), 131.2 (d, J = 2.8 Hz), 128.3 (d, J = 8.3 Hz), 125.3, 115.1 (d, J = 21.4 Hz), 110.5, 94.7, 83.6, 31.7, 29.5, 27.2, 24.54, 24.51, 22.5, 14.0; The carbon atom directly attached to the boron atom was not detected; ^{19}F NMR (282 MHz, $CDCl_3$) δ -116.7; IR (neat) ν (cm^{-1}) 2978, 2929, 2859, 1928, 1602, 1508, 1412, 1316, 1274, 1225, 1196, 1144, 1112; MS (EI) *m/z*: 356 (M^+(^{11}B), 100), 355 (M^+(^{10}B), 27.13); HRMS calcd. for $C_{22}H_{30}{}^{11}BFO_2$ (M^+): 356.2323; Found: 356.2323.

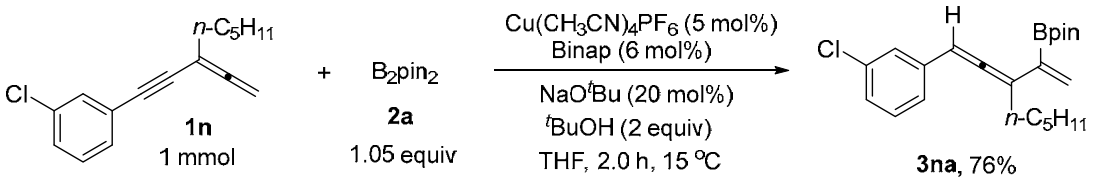
2.13. Preparation of 2-(5-(4-chlorophenyl)-3-pentylpenta-1,3,4-trien-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **3ma** (syl-8-21).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2670 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0184 g, 0.05 mmol), BINAP (0.0377 g, 0.06 mmol), NaO^tBu (0.0195 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1m** (0.2440 g, 1 mmol), and THF (5 mL) afforded **3ma** (0.2833 g, 76%) (eluent: petroleum ether (60~90 °C)/DCM = 7/1 (480 mL) to 5/1 (240 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.28-7.20 (m, 4 H, ArH), 6.26-6.18 (m, 1 H, =CH), 5.76 (s, 1 H, one proton of $=CH_2$), 5.71 (s, 1 H, one proton of $=CH_2$), 2.29 (td, J_1 = 7.4 Hz, J_2 = 2.7 Hz, 2 H, CH_2), 1.58-

1.38 (m, 2 H, CH_2), 1.37-1.22 (m, 4 H, CH_2 × 2), 1.17 (s, 6 H, CH_3 × 2), 1.13 (s, 6 H, CH_3 × 2), 0.84 (t, J = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.3, 133.8, 131.9, 128.4, 128.1, 125.6, 110.6, 94.8, 83.5, 31.6, 29.4, 27.1, 24.5, 22.4, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2977, 2958, 2929, 2859, 1928, 1589, 1490, 1409, 1316, 1273, 1214, 1197, 1166, 1144, 1091, 1013; MS (EI) *m/z*: 374 (M^+(^{37}Cl)(^{11}B), 41.81), 373 (M^+(^{37}Cl)(^{10}B), 37.30), 372 (M^+(^{35}Cl)(^{11}B), 100), 371 (M^+(^{35}Cl)(^{10}B), 28.27); HRMS calcd. for $C_{22}H_{30}{}^{11}B^{35}ClO_2$ (M^+): 372.2027; Found: 372.2029.

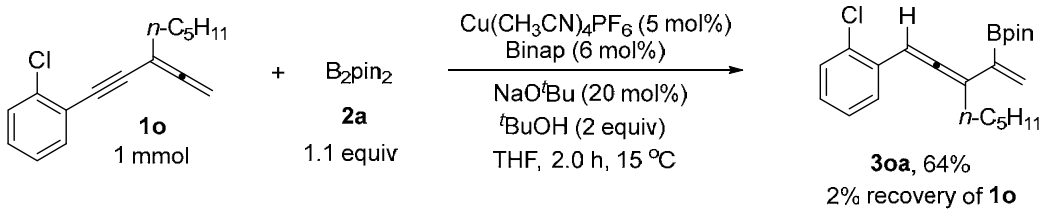
2.14. Preparation of 2-(5-(3-chlorophenyl)-3-pentylpenta-1,3,4-trien-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **3na** (syl-8-34).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2670 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0185 g, 0.05 mmol), BINAP (0.0376 g, 0.06 mmol), NaO^tBu (0.0196 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1n** (0.2433 g, 1 mmol), and THF (5 mL) afforded **3na** (0.2816 g, 76%) (eluent: petroleum ether (60~90 °C)/DCM = 5/1 (480 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.38-7.32 (m, 1 H, ArH), 7.23-7.07 (m, 3 H, ArH), 6.26-6.15 (m, 1 H, =CH), 5.78 (s, 1 H, one proton of $=CH_2$), 5.74 (s, 1 H, one proton of $=CH_2$), 2.30 (td, J_1 = 7.4 Hz, J_2 = 2.7 Hz, 2 H, CH_2), 1.58-1.40 (m, 2 H, CH_2), 1.37-1.22 (m, 4 H, CH_2 × 2), 1.18 (s, 6 H, CH_3 × 2), 1.14 (s, 6 H, CH_3 × 2), 0.84 (t, J = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ

208.4, 137.4, 134.2, 129.4, 126.9, 126.4, 126.1, 125.1, 110.6, 94.7, 83.6, 31.7, 29.6, 27.1, 24.6, 24.5, 22.4, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2978, 2957, 2929, 2858, 1929, 1593, 1569, 1477, 1412, 1316, 1273, 1214, 1196, 1165, 1144, 1112, 1077; MS (EI) *m/z*: 374 ($M^+(^{37}Cl)(^{11}B)$, 29.31), 373 ($M^+(^{37}Cl)(^{10}B)$, 25.78), 372 ($M^+(^{35}Cl)(^{11}B)$, 79.01), 371 ($M^+(^{35}Cl)(^{10}B)$, 19.55), 83 (100); HRMS calcd. for $C_{22}H_{30}{}^{11}BO_2{}^{35}Cl$ (M^+): 372.2027; Found: 372.2029.

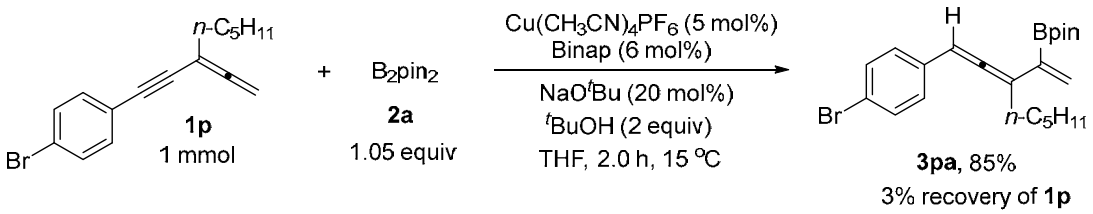
2.15. Preparation of 2-(5-(2-chlorophenyl)-3-pentylpenta-1,3,4-trien-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **3oa** (syl-8-50).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2796 g, 1.1 mmol), $Cu(CH_3CN)_4PF_6$ (0.0187 g, 0.05 mmol), BINAP (0.0374 g, 0.06 mmol), NaO^tBu (0.0195 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1o** (0.2445 g, 1 mmol), and THF (5 mL) afforded **3oa** (0.2384 g, 64%) (eluent: petroleum ether (60~90 °C)/DCM = 7/1 (560 mL) to 5/1 (240 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.54 (d, *J* = 7.2 Hz, 1 H, ArH), 7.31 (d, *J* = 7.5 Hz, 1 H, ArH), 7.17 (t, *J* = 7.5 Hz, 1 H, ArH), 7.08 (t, *J* = 7.4 Hz, 1 H, ArH), 6.78-6.70 (m, 1 H, =CH), 5.77 (s, 1 H, one proton of $=CH_2$), 5.71 (s, 1 H, one proton of $=CH_2$), 2.30 (td, J_1 = 7.5 Hz, J_2 = 2.7 Hz, 2 H, CH_2), 1.58-1.44 (m, 2 H, CH_2), 1.37-1.25 (m, 4 H, CH_2 × 2), 1.17 (s, 6 H, CH_3 × 2), 1.14 (s, 6 H, CH_3 × 2), 0.84 (t, *J* = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$)

δ 209.2, 132.9, 131.7, 129.3, 128.9, 127.4, 126.4, 125.6, 110.4, 92.1, 83.6, 31.7, 29.4, 27.2, 24.54, 24.48, 22.4, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 3069, 2978, 2957, 2929, 2859, 1928, 1589, 1567, 1477, 1442, 1413, 1358, 1316, 1273, 1218, 1198, 1144, 1111, 1049, 1032; MS (EI) *m/z*: 374 (M^+(^{37}Cl)(^{11}B), 32.41), 373 (M^+(^{37}Cl)(^{10}B), 28.71), 372 (M^+(^{35}Cl)(^{11}B), 86.91), 371 (M^+(^{35}Cl)(^{10}B), 21.90), 83 (100); HRMS calcd. for $C_{22}H_{30}{}^{11}BO_2{}^{35}Cl$ (M^+): 372.2027; Found: 372.2026.

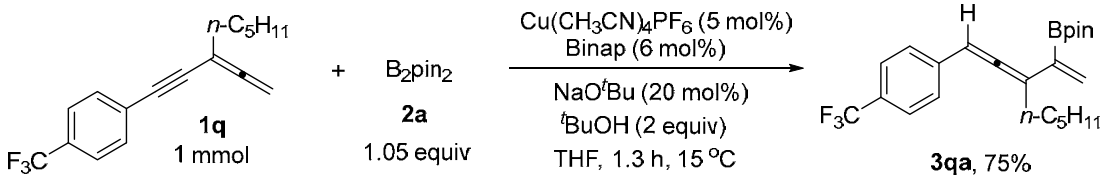
2.16. Preparation of 2-(5-(4-bromophenyl)-3-pentylpenta-1,3,4-trien-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **3pa** (syl-8-29).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2670 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0189 g, 0.05 mmol), BINAP (0.0376 g, 0.06 mmol), NaO^tBu (0.0191 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1p** (0.2873 g, 1 mmol), and THF (5 mL) afforded **3pa** (0.3518 g, 85%) (eluent: petroleum ether (60~90 °C)/DCM = 5/1 (480 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.1 Hz, 2 H, ArH), 7.19 (d, *J* = 8.4 Hz, 2 H, ArH), 6.25-6.17 (m, 1 H, =CH), 5.76 (s, 1 H, one proton of $=CH_2$), 5.70 (s, 1 H, one proton of $=CH_2$), 2.28 (td, J_1 = 7.4 Hz, J_2 = 2.7 Hz, 2 H, CH_2), 1.55-1.42 (m, 2 H, CH_2), 1.36-1.25 (m, 4 H, CH_2 × 2), 1.17 (s, 6 H, CH_3 × 2), 1.13 (s, 6 H, CH_3 × 2), 0.84 (t, *J* = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz,

$CDCl_3$) δ 208.4, 134.3, 131.3, 128.5, 125.7, 120.0, 110.7, 94.9, 83.6, 31.7, 29.4, 27.1, 24.54, 24.52, 22.4, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2977, 2957, 2928, 2858, 1929, 1589, 1487, 1412, 1379, 1371, 1316, 1273, 1214, 1197, 1166, 1144, 1112, 1070, 1010; MS (EI) *m/z*: 418 (M^+(^{81}Br)(^{11}B), 82.41), 417 (M^+(^{81}Br)(^{10}B), 40.39), 416 (M^+(^{79}Br)(^{11}B), 82.70), 415 (M^+(^{79}Br)(^{10}B), 21.02), 101 (100); HRMS calcd. for $C_{22}H_{30}{}^{11}BO_2{}^{79}Br$ (M^+): 416.1522; Found: 416.1522.

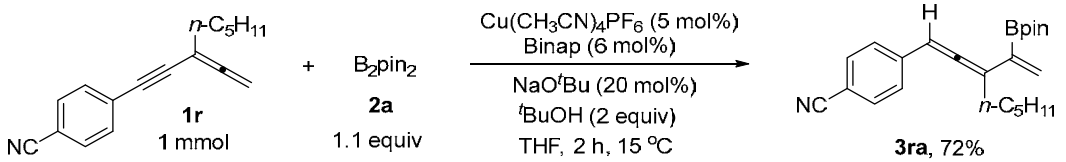
2.17. Preparation of 2-(5-(4-trifluoromethylphenyl)-3-pentylpenta-1,3,4-trien-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **3qa** (syl-8-23).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2671 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0186 g, 0.05 mmol), BINAP (0.0376 g, 0.06 mmol), NaO^tBu (0.0191 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1q** (0.2784 g, 1 mmol), and THF (5 mL) afforded **3qa** (0.3045 g, 75%) (eluent: petroleum ether (60~90 °C)/DCM = 7/1 (480 mL) to 5/1 (240 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.1 Hz, 2 H, ArH), 7.42 (d, *J* = 8.1 Hz, 2 H, ArH), 6.33-6.25 (m, 1 H, =CH), 5.80 (s, 1 H, one proton of $=CH_2$), 5.75 (s, 1 H, one proton of $=CH_2$), 2.31 (td, J_1 = 7.5 Hz, J_2 = 2.7 Hz, 2 H, CH_2), 1.56-1.41 (m, 2 H, CH_2), 1.37-1.25 (m, 4 H, CH_2 × 2), 1.16 (s, 6 H, CH_3 × 2), 1.13 (s, 6 H, CH_3 × 2), 0.84 (t, *J* = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 209.2, 139.3, 128.3 (q, *J* = 32.0 Hz), 127.0, 126.3, 125.2 (q,

J = 3.7 Hz), 124.4 (q, J = 270.2 Hz), 110.8, 94.9, 83.6, 31.7, 29.5, 27.2, 24.53, 24.51, 22.4, 14.0; The carbon atom directly attached to the boron atom was not detected; ^{19}F NMR (282 MHz, $CDCl_3$) δ -62.8; IR (neat) ν (cm^{-1}) 2978, 2931, 2860, 1928, 1615, 1590, 1324, 1165, 1144, 1124, 1066, 1017; MS (EI) *m/z*: 406 (M^+(^{11}B), 67.96), 405 (M^+(^{10}B), 17.98), 83 (100); HRMS calcd. for $C_{23}H_{30}{}^{11}BO_2F_3$ (M^+): 406.2291; Found: 406.2290.

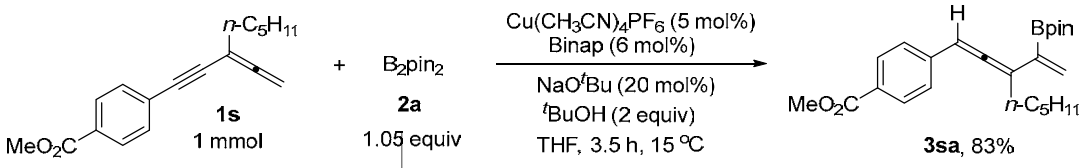
2.18. Preparation of 2-(5-(4-cyanophenyl)-3-pentylpenta-1,3,4-trien-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **3ra** (syl-8-52).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2797 g, 1.1 mmol), $Cu(CH_3CN)_4PF_6$ (0.0187 g, 0.05 mmol), BINAP (0.0377 g, 0.06 mmol), NaO^tBu (0.0193 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1r** (0.2351 g, 1 mmol), and THF (5 mL) afforded **3ra** (0.2616 g, 72%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 100/1 (505 mL) to 50/1 (510 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.57 (d, J = 8.1 Hz, 2 H, ArH), 7.41 (d, J = 8.4 Hz, 2 H, ArH), 6.32-6.22 (m, 1 H, =CH), 5.81 (s, 1 H, one proton of $=CH_2$), 5.78 (s, 1 H, one proton of $=CH_2$), 2.31 (td, J_1 = 7.4 Hz, J_2 = 2.7 Hz, 2 H, CH_2), 1.57-1.42 (m, 2 H, CH_2), 1.36-1.23 (m, 4 H, CH_2 × 2), 1.16 (s, 6 H, CH_3 × 2), 1.12 (s, 6 H, CH_3 × 2), 0.84 (t, J = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 209.8, 140.7, 132.1, 127.4, 126.8, 119.3, 111.0, 109.5, 95.0, 83.6, 31.6, 29.4, 27.1, 24.51, 24.49, 22.4, 14.0; The carbon atom

directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2977, 2959, 2930, 2859, 2226, 1928, 1604, 1502, 1411, 1318, 1202, 1168, 1144, 1111; MS (EI) *m/z*: 363 (M^+(^{11}B), 43.52), 362 (M^+(^{10}B), 11.17), 83 (100); HRMS calcd. for $C_{23}H_{30}{}^{11}BNO_2$ (M^+): 363.2370; Found: 363.2369.

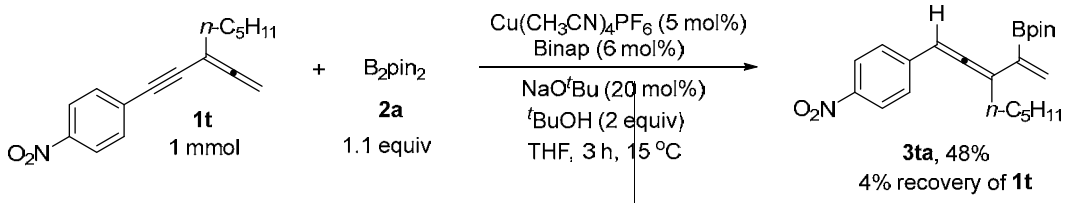
2.19. Preparation of 4,4,5,5-tetramethyl-2-(5-(4-(methoxycarbonyl)phenyl)-3-pentylpenta-1,3,4-trien-2-yl)-1,3,2-dioxaborolane **3sa** (syl-8-43).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2670 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0186 g, 0.05 mmol), BINAP (0.0374 g, 0.06 mmol), NaO^tBu (0.0193 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1s** (0.2677 g, 1 mmol), and THF (5 mL) afforded **3sa** (0.3269 g, 83%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 40/1 (512.5 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.1 Hz, 2 H, ArH), 7.38 (d, *J* = 8.4 Hz, 2 H, ArH), 6.37-6.25 (m, 1 H, =CH), 5.79 (s, 1 H, one proton of $=CH_2$), 5.74 (s, 1 H, one proton of $=CH_2$), 3.90 (s, 3 H, OCH_3), 2.31 (td, J_1 = 7.4 Hz, J_2 = 2.7 Hz, 2 H, CH_2), 1.60-1.41 (m, 2 H, CH_2), 1.38-1.23 (m, 4 H, CH_2 × 2), 1.16 (s, 6 H, CH_3 × 2), 1.12 (s, 6 H, CH_3 × 2), 0.83 (t, *J* = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 209.5, 167.1, 140.5, 129.6, 127.9, 126.8, 126.1, 110.6, 95.3, 83.6, 51.9, 31.6, 29.4, 27.1, 24.52, 24.50, 22.4, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2977, 2954, 2930, 2859, 1927, 1723, 1606, 1508, 1435, 1316, 1276, 1193, 1174, 1144, 1109,

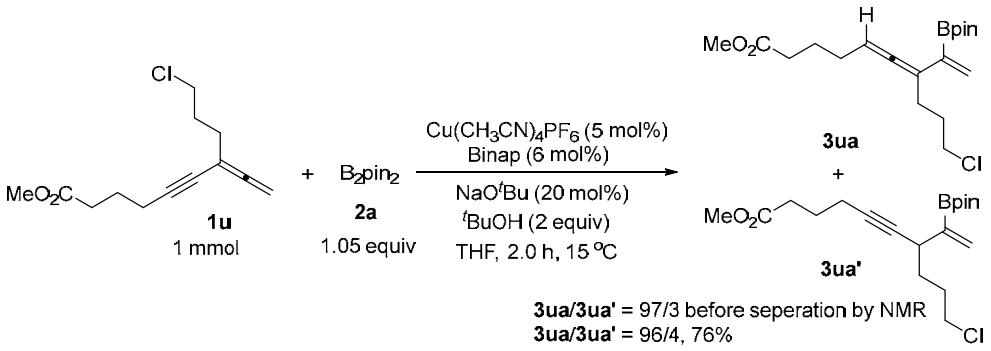
1018; MS (EI) *m/z*: 396 (M^+(^{11}B), 100), 395 (M^+(^{10}B), 23.98); HRMS calcd. for $C_{24}H_{33}{}^{11}BO_4$ (M^+): 396.2472; Found: 396.2469.

2.20. Preparation of 4,4,5,5-tetramethyl-2-(5-(4-nitrophenyl)-3-pentylpenta-1,3,4-trien-2-yl)-1,3,2-dioxaborolane **3ta** (syl-8-46).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2798 g, 1.1 mmol), $Cu(CH_3CN)_4PF_6$ (0.0187 g, 0.05 mmol), BINAP (0.0376 g, 0.06 mmol), NaO^tBu (0.0190 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1t** (0.2542 g, 1 mmol), and THF (5 mL) afforded **3ta** (0.1851 g, 48%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 100/1 (505 mL) to 20/1 (105 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16 (d, *J* = 8.4 Hz, 2 H, ArH), 7.46 (d, *J* = 8.7 Hz, 2 H, ArH), 6.33 (s, 1 H, =CH), 5.81 (d, *J* = 6.6 Hz, 2 H, $=CH_2$), 2.33 (td, J_1 = 7.2 Hz, J_2 = 2.7 Hz, 2 H, CH_2), 1.58-1.42 (m, 2 H, CH_2), 1.36-1.23 (m, 4 H, CH_2 × 2), 1.16 (s, 6 H, CH_3 × 2), 1.13 (s, 6 H, CH_3 × 2), 0.84 (t, *J* = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 210.5, 146.2, 142.9, 127.3, 127.1, 123.7, 111.0, 94.7, 83.7, 31.6, 29.5, 27.1, 24.54, 24.51, 22.4, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2978, 2930, 2859, 1928, 1602, 1508, 1411, 1315, 1225, 1144, 1112; MS (EI) *m/z*: 383 (M^+(^{11}B), 37.96), 382 (M^+(^{10}B), 9.03), 83 (100); HRMS calcd. for $C_{22}H_{30}{}^{11}BNO_4$ (M^+): 383.2268; Found: 383.2270.

2.21. Preparation of 2-(3-(3-chloropropyl)-8-(methoxycarbonyl)octa-1,3,4-trien-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **3ua** (syl-8-89).



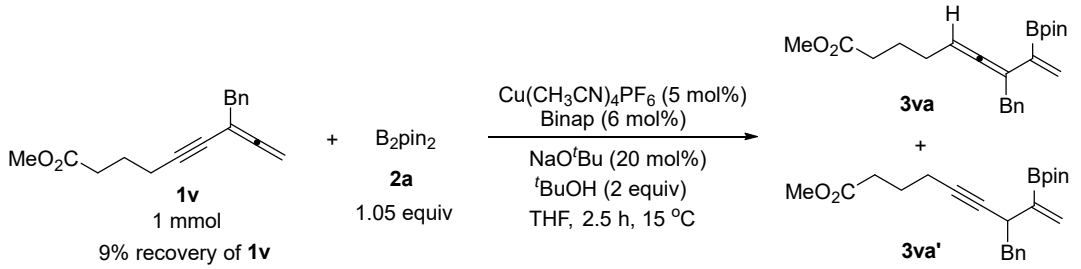
Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2669 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0188 g, 0.05 mmol), BINAP (0.0378 g, 0.06 mmol), NaO^tBu (0.0190 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1u** (0.2403 g, 1 mmol), and THF (5 mL) afforded **3ua** and **3ua'** (0.2902 g, **3ua**/**3ua'** = 25/1, 76%) (**3ua**/**3ua'** = 97/3 determined by ^{1}H NMR of crude product) (eluent: petroleum ether (60~90 °C)/ ethyl acetate = 20/1 (525 mL)).

3ua: liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.65 (d, *J* = 5.1 Hz, 2 H, $=CH_2$), 5.33-5.23 (m, 1 H, =CH), 3.67 (s, 3 H, OCH_3), 3.57 (t, *J* = 6.6 Hz, 2 H, CH_2), 2.42-2.27 (m, 4 H, $CH_2 \times 2$), 2.13-2.02 (m, 2 H, CH_2), 1.98-1.86 (m, 2 H, CH_2), 1.84-1.72 (m, 2 H, CH_2), 1.27 (s, 12 H, $CH_3 \times 4$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.5, 173.8, 124.9, 105.3, 91.7, 83.5, 51.4, 44.7, 33.5, 30.6, 28.4, 26.6, 24.64, 24.55, 24.2; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2978, 2953, 1943, 1740, 1585, 1436, 1371, 1313, 1213, 1144; MS (EI) *m/z*: 370 (M^+(^{37}Cl)(^{11}B), 2.26), 369 (M^+(^{37}Cl)(^{10}B), 4.50), 368 (M^+(^{35}Cl)(^{11}B), 5.09), 367 (M^+(^{35}Cl)(^{10}B), 2.02), 283 (100); HRMS calcd. for $C_{19}H_{30}{}^{11}BO_4{}^{35}Cl$ (M^+): 368.1926; Found: 368.1924.

The following signals are discernible for **3ua'**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.98

(s, 1 H, one proton of =CH_2), 5.91 (s, 1 H, one proton of =CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 35.6, 32.8, 32.6, 30.2, 26.8, 24.8, 24.4, 18.2.

2.22. Preparation of 2-(3-benzyl-8-(methoxycarbonyl)octa-1,3,4-trien-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **3va** (syl-8-113).



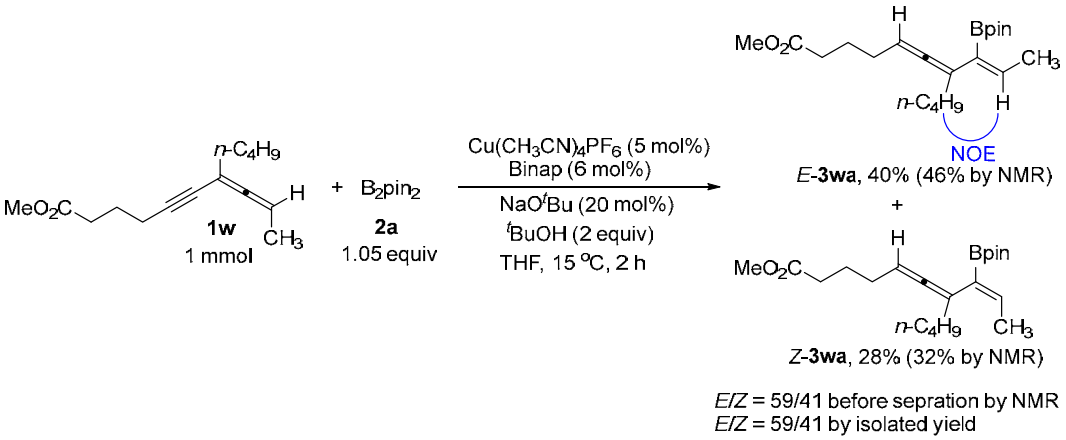
Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2669 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0188 g, 0.05 mmol), BINAP (0.0372 g, 0.06 mmol), NaO^tBu (0.0192 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1v** (0.2541 g, 1 mmol), and THF (5 mL) afforded **3va** and **3va'** (0.2139 g, **3va**/**3va'** = 95/5, 53%) (eluent: petroleum ether (60~90 °C)/ ethyl acetate = 30/1 (465 mL)).

3va: liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30-7.10 (m, 5 H, ArH), 5.67 (s, 1 H, one proton of =CH_2), 5.62 (s, 1 H, one proton of =CH_2), 5.16-5.07 (m, 1 H, =CH), 3.65 (s, 3 H, OCH_3), 3.53 (s, 2 H, CH_2), 2.20 (t, *J* = 7.5 Hz, 2 H, CH_2), 2.00-1.90 (m, 2 H, CH_2), 1.68-1.56 (m, 2 H, CH_2), 1.26 (s, 12 H, CH_3 × 4); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.3, 174.0, 139.7, 129.0, 127.9, 125.8, 125.2, 106.2, 91.2, 83.5, 51.3, 36.8, 33.3, 28.2, 24.7, 24.6, 24.1; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2978, 2950, 1944, 1739, 1371, 1313, 1212, 1143, 1075; MS (EI) *m/z*: 382 (M^+(^{11}B), 36.79), 381 (M^+(^{10}B), 9.11), 91 (100); HRMS calcd. for $C_{23}H_{31}{}^{11}BO_4$

(M^+): 382.2315; Found: 382.2314.

The following signals are discernible for **3va'**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.93 (s, 1 H, one proton of $=CH_2$), 5.91 (s, 1 H, one proton of $=CH_2$), 3.67 (s, 3 H, OCH_3), 2.35 (t, *J* = 7.5 Hz, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 129.4, 127.8, 83.5, 32.7, 26.8, 24.9.

2.23. Preparation of (***E***)-4,4,5,5-tetramethyl-2-(9-methoxycarbonyl-4-pentylnona-2,4,5-trien-3-yl)-1,3,2-dioxaborolane *E*-**3wa** and *Z*-4,4,5,5-tetramethyl-2-(9-methoxycarbonyl-4-pentylnona-2,4,5-trien-3-yl)-1,3,2-dioxaborolane *Z*-**3wa** (syl-8-140).



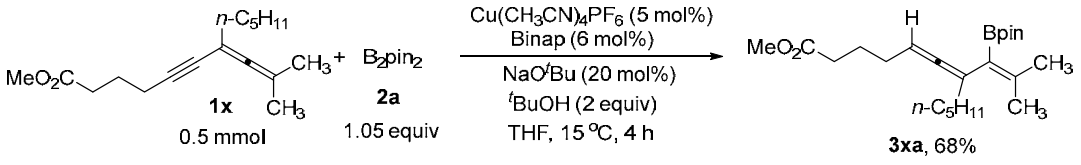
Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2670 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0183 g, 0.05 mmol), BINAP (0.0372 g, 0.06 mmol), NaO^tBu (0.0191 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1w** (0.2345 g, 1 mmol), and THF (5 mL) afforded *E*-**3wa** (0.1436 g, 40%) and *Z*-**3wa** (0.1011 g, 28%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 30/1 (620 mL)) (*E*/*Z* = 59/41 determined by ^{1}H NMR of crude product).

E-**3wa**: liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.50 (q, *J* = 6.6 Hz, 1 H, CH), 5.05-4.95

(m, 1 H, CH), 3.66 (s, 3 H, OCH_3), 2.35 (t, J = 7.5 Hz, 2 H, CH_2), 2.17-2.00 (m, 4 H, CH_2 × 2), 1.83-1.69 (m, 5 H, CH_3 + CH_2), 1.42-1.18 (m, 16 H, CH_2 × 2 + CH_3 × 4), 0.88 (t, J = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.4, 174.1, 142.1, 103.2, 88.7, 83.1, 51.4, 33.4, 32.9, 30.0, 28.5, 24.7, 24.5, 22.3, 15.9, 13.9; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2977, 2955, 2932, 2859, 1957, 1742, 1617, 1436, 1373, 1354, 1332, 1307, 1214, 1145, 1008; MS (EI) *m/z*: 362 (M^+(^{11}B), 14.91), 361 (M^+(^{10}B), 4.33), 101 (100); HRMS calcd. for $C_{21}H_{35}O_4{}^{11}B$ (M^+): 362.2628; Found: 362.2630.

Z-**3wa**: liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.03 (q, J = 6.8 Hz, 1 H, CH), 5.22 (s, 1 H, CH), 3.66 (s, 3 H, OCH_3), 2.36 (t, J = 7.4 Hz, 2 H, CH_2), 2.16-1.98 (m, 4 H, CH_2 × 2), 1.90-1.70 (m, 5 H, CH_3 + CH_2), 1.47-1.27 (m, 16 H, CH_2 × 2 + CH_3 × 4), 0.89 (t, J = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.0, 174.0, 132.5, 107.7, 91.2, 83.4, 51.4, 33.6, 29.9, 29.2, 28.7, 25.1, 24.7, 24.2, 22.6, 17.6, 13.9; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2978, 2956, 2931, 2860, 1941, 1742, 1611, 1436, 1339, 1304, 1266, 1145; MS (EI) *m/z*: 362 (M^+(^{11}B), 26.65), 361 (M^+(^{10}B), 6.36), 83 (100); HRMS calcd. for $C_{21}H_{35}O_4{}^{11}B$ (M^+): 362.2628; Found: 362.2629 .

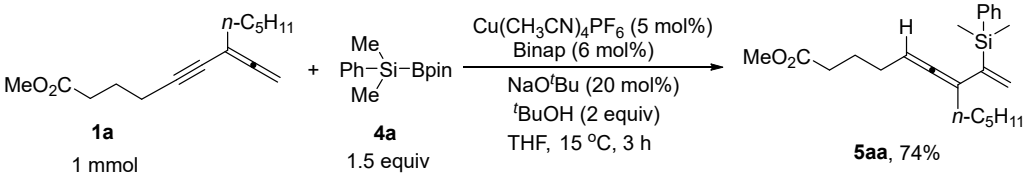
2.24. Preparation of 4,4,5,5-tetramethyl-2-(2-methyl-9-methoxycarbonyl-4-pentylnona-2,4,5-trien-3-yl)-1,3,2-dioxaborolane **3xa** (syl-8-172, syl-8-184).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.1337 g, 0.525 mmol), $Cu(CH_3CN)_4PF_6$ (0.0095 g, 0.025 mmol), BINAP (0.0186 g, 0.03 mmol), NaO^tBu (0.0095 g, 0.1 mmol), ^{t}BuOH (91 μL, d = 0.81 g/mL, 0.0737 g, 1 mmol), **1x** (0.1312 g, 0.5 mmol), and THF (2.5 mL) afforded **3xa** (0.1320 g, 68%) (eluent: petroleum ether (60~90 ºC)/ethyl acetate = 40/1 (512.5 mL)): liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.05-4.97 (m, 1 H, =CH), 3.66 (s, 3 H, OCH_3), 2.35 (t, *J* = 7.6 Hz, 2 H, CH_2), 2.08-1.94 (m, 7 H, $CH_2 \times 2 + CH_3$), 1.81-1.71 (m, 5 H, $CH_3 + CH_2$), 1.43-1.33 (m, 2 H, CH_2), 1.33-1.22 (16 H, $CH_2 \times 2 + CH_3 \times 4$), 0.88 (t, *J* = 7.0 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.2, 174.2, 148.4, 105.9, 88.7, 82.9, 51.4, 33.7, 33.5, 31.6, 28.4, 27.4, 24.73, 24.67, 24.6, 24.3, 22.6, 22.5, 14.1; The carbon directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2977, 2955, 2930, 2858, 1957, 1742, 1618, 1437, 1370, 1341, 1297, 1214, 1146, 1071; MS (EI) *m/z*: 390 (M^+(^{11}B), 7.47), 389 (M^+(^{10}B), 2.09), 101 (100); HRMS calcd. for $C_{23}H_{39}O_4{}^{11}B$ (M^+): 390.2941; Found: 390.2942.

3. Synthesis of 1,3,4-trienyl silanes.

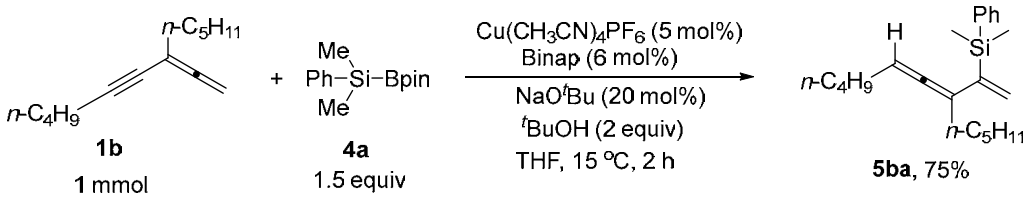
3.1. Preparation of dimethyl(8-methoxycarbonyl-3-pentylocta-1,3,4-trien-2-yl)(phenyl)silane **5aa** (syl-8-117).



Typical Procedure IV: To a flame-dried Schlenk tube were added $Cu(CH_3CN)_4PF_6$ (0.0189 g, 0.05 mmol), BINAP (0.0374 g, 0.06 mmol), NaO^tBu

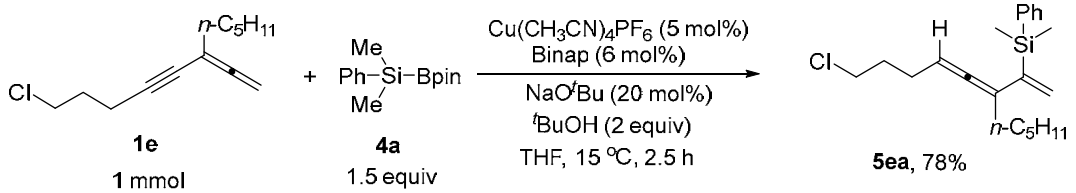
(0.0191 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **4a** (0.41 mL, d = 0.962 g/mL, 0.394 g, 1.5 mmol), **1a** (0.2343 g, 1 mmol), and THF (5 mL) under N_2 atmosphere. The resulting mixture was stirred at 15 °C for 3 hours as monitored by TLC and filtrated through a pad of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent under reduced pressure, the yield of the crude product was determined by ^{1}H NMR analysis using mesitylene (46 μL) as the internal standard (75% by NMR). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **5aa** (0.2742 g, 74%) (eluent: petroleum ether (60~90 °C)/DCM = 7/1 (1040 mL) to 5/1 (240 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.52-7.40 (m, 2 H, ArH), 7.38-7.27 (m, 3 H, ArH), 5.82 (s, 1 H, one proton of $=CH_2$), 5.47-5.35 (m, 1 H, one proton of $=CH_2$), 5.02-4.90 (m, 1 H, =CH), 3.65 (s, 3 H, OCH_3), 2.25-2.05 (m, 4 H, CH_2 × 2), 1.75-1.60 (m, 2 H, CH_2), 1.54-1.18 (m, 8 H, CH_2 × 4), 0.88 (t, *J* = 6.8 Hz, 3 H, CH_3), 0.40 (s, 3 H, CH_3), 0.35 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.1, 173.9, 145.3, 139.1, 133.9, 128.5, 127.5, 124.4, 107.2, 92.2, 51.4, 33.6, 31.7, 29.7, 28.3, 27.3, 24.1, 22.5, 14.1, -1.6, -2.1; IR (neat) ν (cm^{-1}) 3068, 2954, 2930, 2858, 1940, 1742, 1461, 1436, 1428, 1247, 1112; MS (EI) *m/z*: 370 (M^+, 6.41), 135 (100); HRMS calcd. for $C_{23}H_{34}O_2Si$ (M^+): 370.2328; Found: 370.2330.

3.2. Preparation of dimethyl(3-pentylnona-1,3,4-trien-2-yl)(phenyl)silane **5ba** (syl-8-119).



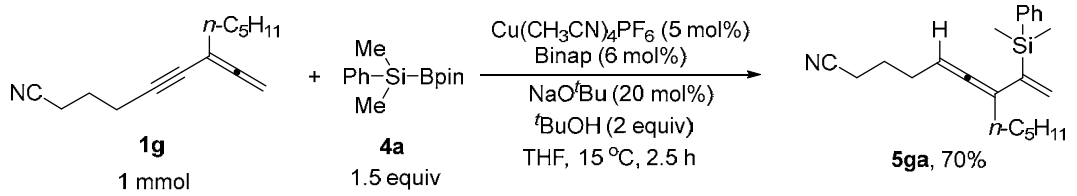
Following **Typical Procedure IV**, the reaction of $Cu(CH_3CN)_4PF_6$ (0.0190 g, 0.05 mmol), BINAP (0.0374 g, 0.06 mmol), NaO^{t}Bu (0.0194 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **4a** (0.41 mL, d = 0.962 g/mL, 0.394 g, 1.5 mmol), **1b** (0.1901 g, 1 mmol), and THF (5 mL) afforded **5ba** (0.2438 g, 75%) (eluent: petroleum ether (60~90 °C) (300 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.55-7.40 (m, 2 H, ArH), 7.38-7.24 (m, 3 H, ArH), 5.79 (s, 1 H, one proton of $=CH_2$), 5.37 (s, 1 H, one proton of $=CH_2$), 5.05-4.92 (m, 1 H, =CH), 2.26-2.03 (m, 2 H, CH_2), 1.67 (q, *J* = 7.0 Hz, 2 H, CH_2), 1.48-1.00 (m, 10 H, CH_2 × 5), 0.95-0.72 (m, 6 H, CH_3 × 2), 0.82 (t, *J* = 7.1 Hz, 3 H, CH_3), 0.40 (s, 3 H, CH_3), 0.36 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.0, 145.6, 139.2, 133.9, 128.5, 127.4, 124.0, 106.7, 93.3, 31.7, 31.1, 29.7, 28.7, 27.2, 22.6, 22.4, 14.1, 13.9, -1.6, -2.0; IR (neat) ν (cm^{-1}) 3068, 2957, 2929, 2872, 2858, 1940, 1571, 1466, 1428, 1378, 1246, 1112; MS (EI) *m/z*: 327 ($(M + H)^+$, 6.38), 326 (M^+, 21.27), 135 (100); HRMS calcd. for $C_{22}H_{34}Si$ (M^+): 326.2430; Found: 326.2432.

3.3. Preparation of dimethyl(8-chloro-3-pentylocta-1,3,4-trien-2-yl)(phenyl)silane **5ea** (syl-8-123).



Following **Typical Procedure IV**, the reaction of $Cu(CH_3CN)_4PF_6$ (0.0186 g, 0.05 mmol), BINAP (0.0375 g, 0.06 mmol), NaO^{t}Bu (0.0194 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **4a** (0.41 mL, d = 0.962 g/mL, 0.394 g, 1.5 mmol), **1e** (0.2105 g, 1 mmol), and THF (5 mL) afforded **5ea** (0.2702 g, 78%) (eluent: petroleum ether (400 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.55-7.38 (m, 2 H, ArH), 7.37-7.25 (m, 3 H, ArH), 5.83 (s, 1 H, one proton of $=CH_2$), 5.44 (s, 1 H, one proton of $=CH_2$), 5.05-4.92 (m, 1 H, =CH), 3.42-3.27 (m, 2 H, CH_2), 2.26-2.05 (m, 2 H, CH_2), 1.85-1.65 (m, 2 H, CH_2), 1.63-1.18 (m, 8 H, CH_2 × 4), 0.88 (t, *J* = 6.6 Hz, 3 H, CH_3), 0.40 (s, 3 H, CH_3), 0.35 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.1, 145.1, 139.1, 133.8, 128.6, 127.5, 124.5, 107.6, 91.7, 44.5, 31.7, 31.5, 29.7, 27.3, 26.1, 22.6, 14.1, -1.6, -2.2; IR (neat) ν (cm^{-1}) 3068, 2956, 2929, 2858, 1939, 1428, 1247, 1112; MS (EI) *m/z*: 348 (M^+(^{37}Cl), 1.78), 346 (M^+(^{35}Cl), 4.59), 135 (100); HRMS calcd. for $C_{21}H_{31}{}^{35}ClSi$ (M^+): 346.1884; Found: 346.1884.

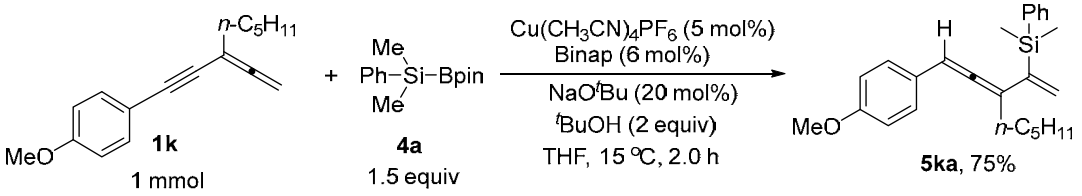
3.4. Preparation of dimethyl(8-cyano-3-pentylocta-1,3,4-trien-2-yl)(phenyl)silane **5ga** (syl-8-121).



Following **Typical Procedure IV**, the reaction of $Cu(CH_3CN)_4PF_6$ (0.0188 g, 0.05 mmol), BINAP (0.0375 g, 0.06 mmol), NaO^{t}Bu (0.0191 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **4a** (0.41 mL, d = 0.962 g/mL, 0.394 g, 1.5 mmol), **1g** (0.2015 g, 1 mmol), and THF (5 mL) afforded **5ga** (0.2369 g, 70%) (eluent: petroleum

ether (60~90 °C)/DCM = 5/1 (960 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.55-7.40 (m, 2 H, ArH), 7.38-7.26 (m, 3 H, ArH), 5.86 (s, 1 H, one proton of $=CH_2$), 5.48 (s, 1 H, one proton of $=CH_2$), 5.05-4.92 (m, 1 H, =CH), 2.23-2.03 (m, 4 H, $CH_2 \times 2$), 1.82-1.60 (m, 2 H, CH_2), 1.47-1.15 (m, 8 H, $CH_2 \times 4$), 0.88 (t, J = 6.6 Hz, 3 H, CH_3), 0.41 (s, 3 H, CH_3), 0.34 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.1, 144.8, 139.1, 133.8, 128.6, 127.5, 124.9, 119.5, 108.0, 91.1, 31.7, 29.7, 27.7, 27.3, 24.2, 22.5, 16.5, 14.1, -1.5, -2.3; IR (neat) ν (cm^{-1}) 3068, 2956, 2930, 2858, 2247, 1940, 1455, 1428, 1247, 1112; MS (EI) *m/z*: 337 (M^+, 15.57), 135 (100); HRMS calcd. for $C_{22}H_{31}NSi$ (M^+): 337.2226; Found: 337.2225.

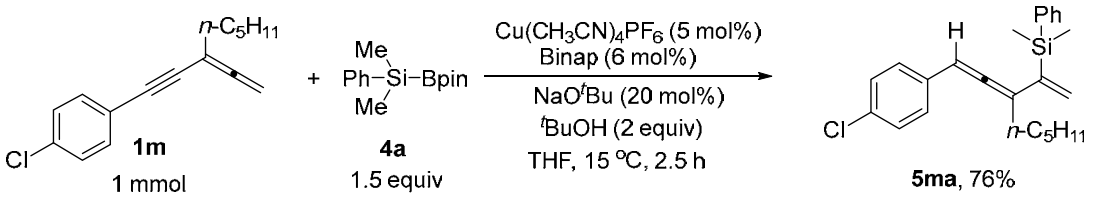
3.5. Preparation of dimethyl(5-(4-methoxyphenyl)-3-pentylpenta-1,3,4-trien-2-yl)(phenyl)silane **5ka** (syl-8-133).



Following **Typical Procedure IV**, the reaction of $Cu(CH_3CN)_4PF_6$ (0.0184 g, 0.05 mmol), BINAP (0.0372 g, 0.06 mmol), NaO^{t}Bu (0.0189 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **4a** (0.41 mL, d = 0.962 g/mL, 0.394 g, 1.5 mmol), **1k** (0.2401 g, 1 mmol), and THF (5 mL) afforded impure **5ka** (0.3090 g) (eluent: petroleum ether/DCM = 8/1 (450 mL)), which was further purified by recrystallization (acetone/MeOH) to afford pure **5ka** (0.2822 g, 75%): solid; m.p. 61.3-63.5 °C (acetone/MeOH); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41 (d, J = 6.9 Hz, 2 H, ArH), 7.33-7.18 (m, 3 H, ArH), 6.97 (d, J = 8.4 Hz, 2 H, ArH), 6.75 (d, J = 8.7 Hz, 2 H, ArH), 5.99

(s, 1 H, one proton of $=CH_2$), 5.89 (s, 1 H, one proton of $=CH_2$), 5.45 (s, 1 H, =CH), 3.79 (s, 3 H, OMe), 2.38-2.17 (m, 2 H, CH_2), 1.51-1.35 (m, 2 H, CH_2), 1.35-1.17 (m, 4 H, $CH_2 \times 2$), 0.83 (t, J = 6.6 Hz, 3 H, CH_3), 0.35 (s, 3 H, CH_3), 0.32 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.4, 158.5, 145.3, 138.5, 133.9, 128.5, 127.9, 127.4, 127.0, 125.0, 113.9, 110.9, 96.3, 55.2, 31.8, 30.0, 27.2, 22.5, 14.1, -2.1, -2.2; IR (neat) ν (cm^{-1}) 3068, 2956, 2928, 2858, 1924, 1487, 1466, 1428, 1247, 1112, 1070, 1010; MS (EI) *m/z*: 376 (M^+, 33.43), 135 (100); Anal. Calcd. for $C_{25}H_{32}OSi$ (%): C 79.73, H 8.56; Found: C 79.26, H 8.44.

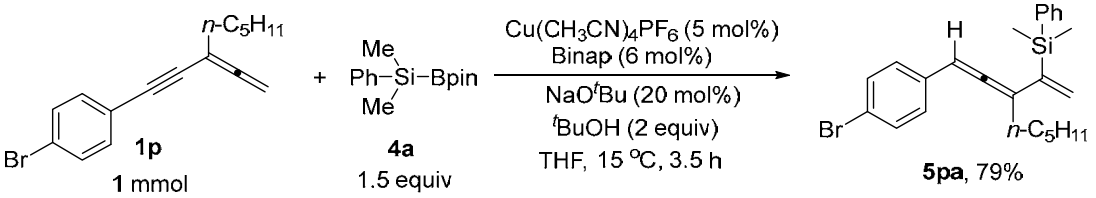
3.6. Preparation of dimethyl(5-(4-chlorophenyl)-3-pentylpenta-1,3,4-trien-2-yl)(phenyl)silane **5ma** (syl-8-125).



Following **Typical Procedure IV**, the reaction of $Cu(CH_3CN)_4PF_6$ (0.0190 g, 0.05 mmol), BINAP (0.0376 g, 0.06 mmol), NaO^tBu (0.0190 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **4a** (0.41 mL, d = 0.962 g/mL, 0.394 g, 1.5 mmol), **1m** (0.2443 g, 1 mmol), and THF (5 mL) afforded **5ma** (0.2910 g, 76%) (eluent: petroleum ether (360 mL)): solid; m.p. 49.7-50.7 °C (acetone/MeOH); ^1H NMR (300 MHz, $CDCl_3$) δ 7.38 (d, J = 7.5 Hz, 2 H, ArH), 7.29-7.07 (m, 5 H, ArH), 6.90 (d, J = 8.4 Hz, 2 H, ArH), 5.97 (s, 1 H, one proton of $=CH_2$), 5.93 (s, 1 H, one proton of $=CH_2$), 5.50 (s, 1 H, =CH), 2.38-2.17 (m, 2 H, CH_2), 1.50-1.34 (m, 2 H, CH_2), 1.33-1.15 (m, 4 H, $CH_2 \times 2$), 0.83 (t, J = 6.6 Hz, 3 H, CH_3), 0.36 (s, 3 H, CH_3), 0.31 (s, 3 H, CH_3); ^{13}C

NMR (75 MHz, $CDCl_3$) δ 208.1, 144.7, 138.2, 133.8, 133.2, 132.0, 128.6, 128.5, 127.9, 127.5, 125.7, 111.4, 96.0, 31.7, 29.9, 27.2, 22.5, 14.1, -2.1, -2.2; IR (neat) ν (cm^{-1}) 3068, 2956, 2929, 2858, 1925, 1490, 1466, 1428, 1247, 1112, 1090, 1013; MS (EI) *m/z*: 382 (M^+(^{37}Cl), 6.00), 380 (M^+(^{35}Cl), 14.88), 135 (100); Anal. Calcd. for $C_{24}H_{29}SiCl$ (%): C 75.65, H 7.67; Found: C 75.51, H 7.60.

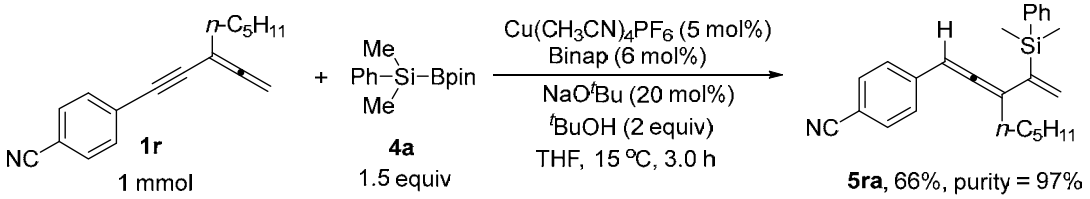
3.7. Preparation of dimethyl(5-(4-bromophenyl)-3-pentylpenta-1,3,4-trien-2-yl)(phenyl)silane **5pa** (syl-8-134).



Following **Typical Procedure IV**, the reaction of $Cu(CH_3CN)_4PF_6$ (0.0188 g, 0.05 mmol), BINAP (0.0378 g, 0.06 mmol), NaO^{t}Bu (0.0192 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **4a** (0.41 mL, d = 0.962 g/mL, 0.394 g, 1.5 mmol), **1p** (0.2882 g, 1 mmol), and THF (5 mL) afforded **5pa** (0.3346 g, 79%) (eluent: petroleum ether (360 mL)): solid; m.p. 53.1-55.0 °C (DCM/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 (d, *J* = 7.2 Hz, 2 H, ArH), 7.31-7.14 (m, 5 H, ArH), 6.84 (d, *J* = 8.4 Hz, 2 H, ArH), 5.95 (s, 1 H, one proton of $=CH_2$), 5.93 (s, 1 H, one proton of $=CH_2$), 5.51 (s, 1 H, =CH), 2.38-2.17 (m, 2 H, CH_2), 1.50-1.36 (m, 2 H, CH_2), 1.33-1.15 (m, 4 H, CH_2 × 2), 0.83 (t, *J* = 6.6 Hz, 3 H, CH_3), 0.36 (s, 3 H, CH_3), 0.31 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.1, 144.6, 138.2, 133.8, 133.7, 131.4, 128.6, 128.2, 127.5, 125.7, 120.1, 111.4, 96.1, 31.7, 29.9, 27.1, 22.5, 14.1, -2.1, -2.2; IR (neat) ν (cm^{-1}) 3068, 2956, 2928, 2858, 1924, 1487, 1466, 1428, 1247, 1112, 1070, 1010; MS (EI) *m/z*: 426

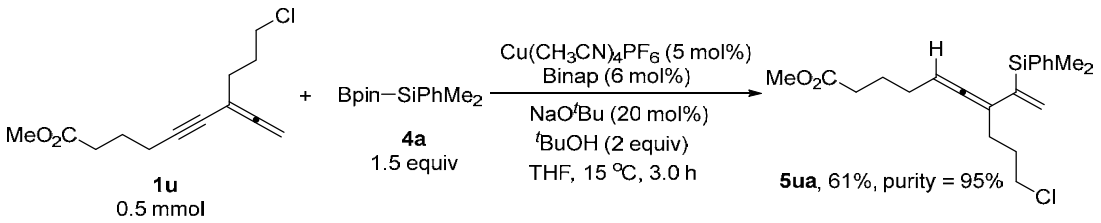
(M (^{81}Br)$^+$, 4.64), 424 (M (^{79}Br)$^+$, 4.39), 135 (100); Anal. Calcd. for $C_{24}H_{29}BrSi$ (%): C 67.75, H 6.87; Found: C 67.75, H 6.84.

3.8. Preparation of dimethyl(5-(4-cyanophenyl)-3-pentylpenta-1,3,4-trien-2-yl)(phenyl)silane **5ra** (syl-8-135).



Following **Typical Procedure IV**, the reaction of $Cu(CH_3CN)_4PF_6$ (0.0186 g, 0.05 mmol), BINAP (0.0374 g, 0.06 mmol), NaO^{t}Bu (0.0190 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **4a** (0.41 mL, d = 0.962 g/mL, 0.394 g, 1.5 mmol), **1r** (0.2350 g, 1 mmol), and THF (5 mL) afforded impure **5ra** (0.2705 g) (eluent: petroleum ether/DCM = 3/1 (600 mL)). The impure product was recrystallized to afford impure **5ra** (0.2506 g, purity = 97%, 66%): solid; m.p. 77.8-79.4 °C (DCM/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43-7.30 (m, 4 H, ArH), 7.26-7.09 (m, 3 H, ArH), 6.97 (d, *J* = 8.4 Hz, 2 H, ArH), 6.03 (s, 1 H, one proton of $=CH_2$), 5.98 (s, 1 H, one proton of $=CH_2$), 5.58 (s, 1 H, =CH), 2.41-2.21 (m, 2 H, CH_2), 1.53-1.35 (m, 2 H, CH_2), 1.35-1.15 (m, 4 H, CH_2 × 2), 0.83 (t, *J* = 6.6 Hz, 3 H, CH_3), 0.38 (s, 3 H, CH_3), 0.31 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 209.4, 143.9, 139.8, 137.8, 133.7, 132.0, 128.6, 127.5, 126.9, 126.3, 119.2, 111.8, 109.4, 96.0, 31.6, 29.8, 27.1, 22.4, 14.0, -2.0, -2.5; IR (neat) ν (cm^{-1}) 3068, 2956, 2929, 2858, 2226, 1923, 1604, 1501, 1428, 1248, 1111; MS (EI) *m/z*: 371 (M^+, 22.43), 135 (100); HRMS calcd. for $C_{25}H_{29}NSi$ (M^+): 371.2069; Found: 371.2068.

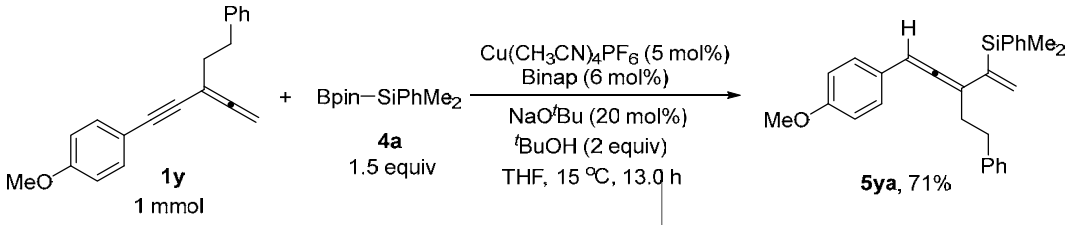
3.9. Preparation of dimethyl(8-methoxycarbonyl-3-(3-chloropropyl)octa-1,3,4-trien-2-yl)(phenyl)silane **5ua** (syl-10-74).



Following **Typical Procedure IV**, the reaction of $Cu(CH_3CN)_4PF_6$ (0.0091 g, 0.025 mmol), Binap (0.0185 g, 0.03 mmol), NaO^tBu (0.0096 g, 0.1 mmol), ^{t}BuOH (91 μL, d = 0.81 g/mL, 0.074 g, 1 mmol), **4a** (0.1965 g, 0.75 mmol), **1u** (0.1202 g, 0.5 mmol), and THF (2.5 mL) afforded **5ua** (0.1216 g, 61%, purity = 95%) (eluent: petroleum ether (60~90 °C)/DCM = 4/1 (600 mL) to 3/1 (200 mL)): liquid; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.50-7.42 (m, 2 H, ArH), 7.34-7.28 (m, 3 H, ArH), 5.83 (s, 1 H, one proton of $=CH_2$), 5.45 (s, 1 H, one proton of $=CH_2$), 5.04-4.98 (m, 1 H, =CH), 3.65 (s, 3 H, OCH_3), `3.52 (t, *J* = 6.6 Hz, 2 H, CH_2), 2.38-2.25 (m, 2 H, CH_2), 2.19-2.15 (m, 2 H, CH_2), 1.94-1.84 (m, 2 H, CH_2), 1.73-1.60 (m, 2 H, CH_2), 1.52-1.37 (m, 2 H, CH_2), 0.40 (s, 3 H, CH_3), 0.35 (s, 3 H, CH_3); ^{13}C NMR (150 MHz, $CDCl_3$) δ 205.8, 173.8, 144.9, 138.8, 133.8, 128.7, 127.5, 124.8, 105.9, 93.0, 51.5, 44.7, 33.5, 30.5, 28.3, 27.0, 24.0, -1.7, -2.2; IR (neat) ν (cm^{-1}) 3068, 2953, 1940, 1739, 1428, 1247, 1112; MS (EI) *m/z*: 378 (M^+(^{37}Cl), 1.06), 376 (M^+(^{35}Cl), 2.58), 135 (100); HRMS calcd. for $C_{21}H_{29}{}^{35}ClNaO_2Si$ (M^+ + Na): 399.1518; Found: 399.1519.

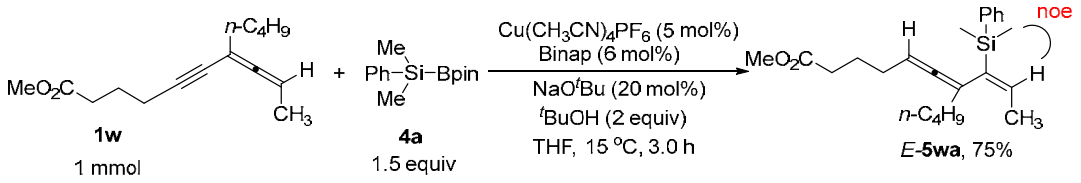
3.10. Preparation of (5-(4-methoxyphenyl)-3-phenethylpenta-1,3,4-trien-2-

yl)dimethyl(phenyl)silane **5ya** (syl-10-77).



Following **Typical Procedure IV**, the reaction of $Cu(CH_3CN)_4PF_6$ (0.0186 g, 0.05 mmol), Binap (0.0376 g, 0.06 mmol), NaO^tBu (0.0192 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **4a** (0.3931 g, 1.5 mmol), **1y** (0.2742 g, 1 mmol), and THF (5 mL) afforded **5ya** (0.2929 g, 71%) (eluent: petroleum ether (60~90 °C)/DCM = 8/1 (360 mL)): liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46-7.40 (m, 2 H, ArH), 7.32-7.20 (m, 5 H, ArH), 7.19-7.12 (m, 3 H, ArH), 6.88 (d, *J* = 9.0 Hz, 2 H, ArH), 6.73 (d, *J* = 9.0 Hz, 2 H, ArH), 5.97 (s, 1 H, CH), 5.92 (s, 1 H, CH), 5.48 (s, 1 H, CH), 3.78 (s, 3 H, OCH_3), 2.83-2.52 (m, 4 H, CH_2 × 2), 0.35 (s, 3 H, CH_3), 0.32 (s, 3 H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 207.5, 158.6, 145.0, 142.1, 138.4, 133.9, 128.6, 128.5, 128.3, 128.0, 127.5, 126.7, 125.7, 125.2, 113.9, 110.2, 96.8, 55.3, 33.8, 32.0, -2.1, -2.2; IR (neat) ν (cm^{-1}) 3066, 3025, 2999, 2953, 2835, 1924, 1607, 1510, 1296, 1248, 1170, 1110, 1035; MS (EI) *m/z*: 410 (M^+, 100); HRMS calcd. for $C_{28}H_{31}OSi$ (M^+ + H): 411.2139; Found: 411.2141.

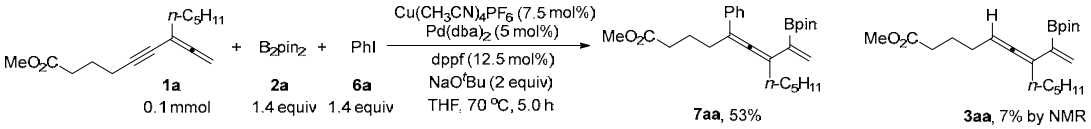
3.11. Preparation of (*E*)-dimethyl(9-methoxycarbonyl-4-butylnona-2,4,5-trien-3-yl)(phenyl)silane (*E*)-**5wa** (syl-10-68).



Following **Typical Procedure IV**, the reaction of $Cu(CH_3CN)_4PF_6$ (0.0188 g, 0.05 mmol), Binap (0.0374 g, 0.06 mmol), NaO^{t}Bu (0.0195 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **4a** (0.41 mL, d = 0.962 g/mL, 0.394 g, 1.5 mmol), **1w** (0.2341 g, 1 mmol), and THF (5 mL) afforded *E*-**5wa** (0.2925 g, 75%) (eluent: petroleum ether (60~90 °C)/DCM = 5/1 (600 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.58-7.45 (m, 2 H, ArH), 7.40-7.27 (m, 3 H, ArH), 5.90 (q, *J* = 6.5 Hz, 1 H, =CH), 5.00-4.88 (m, 1 H, =CH), 3.66 (s, 3 H, OCH_3), 2.30 (t, *J* = 7.7 Hz, 2 H, CH_2), 1.94 (q, *J* = 7.0 Hz, 2 H, CH_2), 1.85-1.57 (m, 7 H, $CH_2 \times 2 + CH_3$), 1.40-1.14 (m, 4 H, $CH_2 \times 2$), 0.81 (t, *J* = 6.8 Hz, 3 H, CH_3), 0.36 (s, 6 H, $CH_3 \times 2$); ^{13}C NMR (125 MHz, $CDCl_3$) δ 200.3, 174.1, 139.5, 138.8, 137.1, 134.1, 128.7, 127.5, 104.5, 89.5, 51.4, 33.4, 33.1, 29.7, 28.8, 24.7, 22.4, 16.1, 13.9, -2.5, -2.6; IR (neat) ν (cm^{-1}) 2955, 2931, 2858, 1958, 1742, 1605, 1435, 1247, 1109; MS (EI) *m/z*: 370 (M^+, 11.27), 135 (100); HRMS calcd. for $C_{23}H_{34}NaO_2Si$ (M^+ + Na): 393.2220; Found: 393.2218.

4. Synthesis of 5-arylalka-1,3,4-trien-2-yl boronates.

4.1. Preparation of 4,4,5,5-tetramethyl-2-(8-methoxycarbonyl-3-pentyl-5-phenylocta-1,3,4-trien-2-yl)-1,3,2-dioxaborolane **7aa** (zj-4-141).

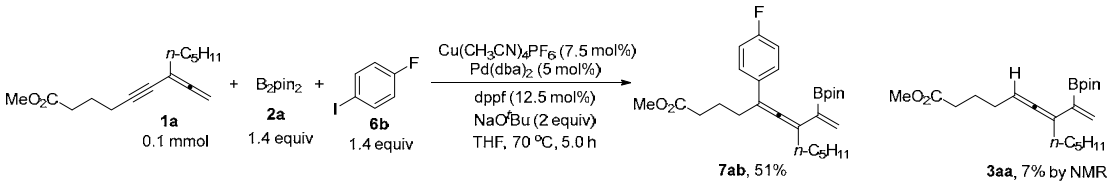


Typical Procedure V: To a flame-dried Schlenk tube were added B_2Pin_2 (0.0357

g, 0.14 mmol), $Cu(CH_3CN)_4PF_6$ (0.0028 g, 0.0075 mmol), dppf (0.0069 g, 0.0125 mmol), NaO^tBu (0.0193 g, 0.2 mmol), $Pd(dba)_2$ (0.0029 g, 0.005 mmol), THF (0.5 mL), **1a** (0.0234 g, 0.1 mmol), **6a** (0.0287 g, 0.14 mmol), and THF (0.5 mL) under N_2 atmosphere. The resulting mixture was stirred at 70 °C for 5 hours as monitored by TLC. The resulting mixture was filtrated through a pad of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **7aa** (0.0231 g, 53%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 40/1 (410 mL)): liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43-7.38 (m, 2 H, ArH), 7.31-7.26 (m, 2 H, ArH), 7.20-7.13 (m, 1 H, ArH), 5.73 (d, *J* = 2.4 Hz, 1 H, one proton of $=CH_2$), 5.67 (d, *J* = 2.4 Hz, 1 H, one proton of $=CH_2$), 3.66 (s, 3 H, OCH_3), 2.55-2.39 (m, 4 H, CH_2 × 2), 2.28 (t, *J* = 7.8 Hz, 2 H, CH_2), 1.96-1.85 (m, 2 H, CH_2), 1.52-1.41 (m, 2 H, CH_2), 1.31-1.25 (m, 4 H, CH_2 × 2), 1.16 (s, 6 H, CH_3 × 2), 1.14 (s, 6 H, CH_3 × 2), 0.83 (t, *J* = 7.0 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.0, 174.1, 137.3, 128.1, 126.3, 126.1, 124.8, 109.9, 105.8, 83.4, 51.4, 33.9, 31.9, 29.84, 29.75, 27.4, 24.6, 24.5, 23.2, 22.5, 14.0; The carbon directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2977, 2954, 2930, 2858, 1926, 1741, 1596, 1494, 1371, 1314, 1145; MS (EI) *m/z*: 438 (M^+(^{11}B), 81.50), 437 (M^+(^{10}B), 20.03), 237 (100); HRMS calcd. for $C_{27}H_{39}{}^{11}BNaO_4$ $(M + Na)^+$: 461.2834; Found: 461.2835.

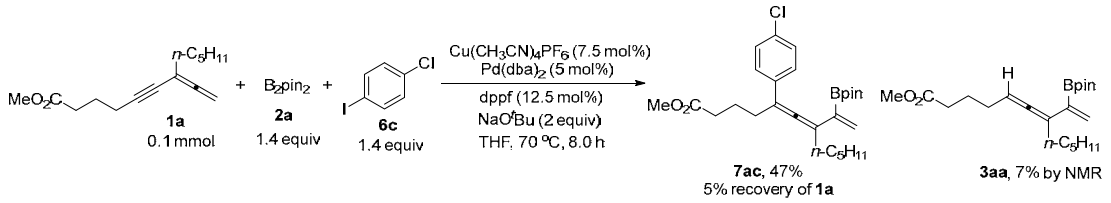
Note：The reaction is very sensitive to water. Hydroboronated product would be formed if water is not removed strictly from the system.

4.2. Preparation of 4,4,5,5-tetramethyl-2-(8-methoxycarbonyl-3-pentyl-5-(4-fluorophenyl)octa-1,3,4-trien-2-yl)-1,3,2-dioxaborolane **7ab** (zj-4-148, syl-10-128).



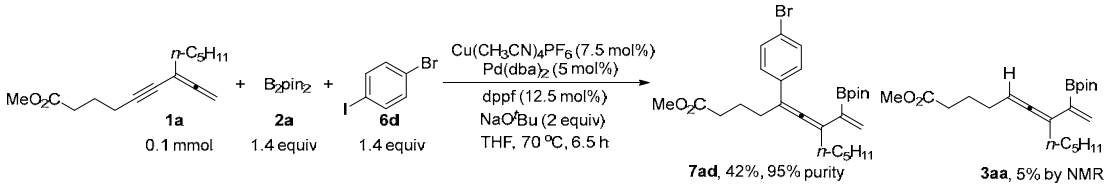
Following **Typical Procedure V**, the reaction of B_2Pin_2 (0.0357 g, 0.14 mmol), $Cu(CH_3CN)_4PF_6$ (0.0028 g, 0.0075 mmol), dppf (0.0069 g, 0.0125 mmol), NaO^tBu (0.0192 g, 0.2 mmol), $Pd(dba)_2$ (0.0028 g, 0.005 mmol), **1a** (0.0234 g, 0.1 mmol), **6b** (0.0312 g, 0.14 mmol), and THF (1 mL) afforded **7ab** (0.0235 g, 51%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 30/1 (310 mL)): liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.33 (m, 2 H, ArH), 7.01-6.93 (m, 2 H, ArH), 5.73 (d, *J* = 2.4 Hz, 1 H, one proton of $=CH_2$), 5.68 (d, *J* = 2.0 Hz, 1 H, one proton of $=CH_2$), 3.67 (s, 3 H, OCH_3), 2.52-2.36 (m, 4 H, $CH_2 \times 2$), 2.27 (t, *J* = 7.6 Hz, 2 H, CH_2), 1.93-1.83 (m, 2 H, CH_2), 1.51-1.38 (m, 2 H, CH_2), 1.31-1.24 (m, 4 H, $CH_2 \times 2$), 1.16 (s, 6 H, $CH_3 \times 2$), 1.14 (s, 6 H, $CH_3 \times 2$), 0.83 (t, *J* = 7.0 Hz, 3 H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 206.7 (d, *J* = 2.3 Hz), 174.0, 161.6 (d, *J* = 243.6 Hz), 133.2 (d, *J* = 3.5 Hz), 127.6 (d, *J* = 8.0 Hz), 125.0, 114.9 (d, *J* = 20.8 Hz), 110.1, 104.9, 83.4, 51.4, 33.8, 31.9, 30.0, 29.7, 27.4, 24.6, 24.5, 23.1, 22.5, 14.0; The carbon directly attached to the boron atom was not detected; ^{19}F NMR (376 MHz, $CDCl_3$) δ -117.4; IR (neat) ν (cm^{-1}) 2977, 2955, 2930, 2859, 1927, 1740, 1601, 1508, 1314, 1230, 1159, 1145; MS (EI) *m/z*: 456 (M^+(^{11}B), 85.14), 455 (M^+(^{10}B), 20.82), 267 (100); HRMS calcd. for $C_{27}H_{38}{}^{11}BFNaO_4$ $(M + Na)^+$: 479.2739; Found: 479.2743.

4.3. Preparation of 4,4,5,5-tetramethyl-2-(8-methoxycarbonyl-3-pentyl-5-(4-chlorophenyl)octa-1,3,4-trien-2-yl)-1,3,2-dioxaborolane **7ac** (zj-4-146, syl-10-124).



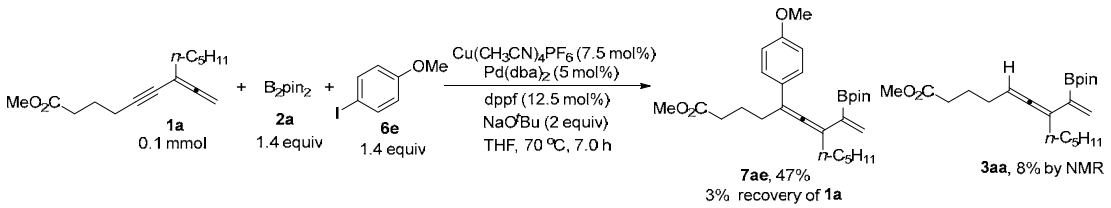
Following **Typical Procedure V**, the reaction of B_2Pin_2 (0.0357 g, 0.14 mmol), $Cu(CH_3CN)_4PF_6$ (0.0028 g, 0.0075 mmol), dppf (0.0069 g, 0.0125 mmol), NaO^tBu (0.0192 g, 0.2 mmol), $Pd(dba)_2$ (0.0029 g, 0.005 mmol), **1a** (0.0234 g, 0.1 mmol), **6c** (0.0335 g, 0.14 mmol), and THF (1 mL) afforded **7ac** (0.0223 g, 47%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 30/1 (310 mL)): liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (d, *J* = 8.4 Hz, 2 H, ArH), 7.24 (d, *J* = 8.4 Hz, 2 H, ArH), 5.74 (d, 1 H, *J* = 2.4 Hz, one proton of $=CH_2$), 5.70 (d, *J* = 2.4 Hz, 1 H, one proton of $=CH_2$), 3.67 (s, 3 H, OCH_3), 2.53-2.35 (m, 4 H, $CH_2 \times 2$), 2.27 (t, *J* = 7.6 Hz, 2 H, CH_2), 1.94-1.82 (m, 2 H, CH_2), 1.51-1.38 (m, 2 H, CH_2), 1.32-1.25 (m, 4 H, $CH_2 \times 2$), 1.16 (s, 6 H, $CH_3 \times 2$), 1.14 (s, 6 H, $CH_3 \times 2$), 0.83 (t, *J* = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 207.0, 174.0, 135.9, 131.9, 128.2, 127.4, 125.3, 110.3, 105.0, 83.5, 51.5, 33.8, 31.8, 29.75, 29.68, 27.4, 24.6, 24.5, 23.1, 22.5, 14.0; The carbon directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2977, 2954, 2930, 2858, 1926, 1741, 1586, 1491, 1314, 1144; MS (EI) *m/z*: 474 (M^+(^{11}B)(^{37}Cl), 28.72), 473 (M^+(^{10}B)(^{37}Cl), 29.56), 472 (M^+(^{11}B)(^{35}Cl), 77.97), 471 (M^+(^{10}B)(^{35}Cl), 19.02), 101 (100); HRMS calcd. for $C_{27}H_{38}{}^{11}B^{35}ClNaO_4$ $(M + Na)^+$: 495.2444; Found: 495.2444.

4.4. Preparation of 4,4,5,5-tetramethyl-2-(8-methoxycarbonyl-3-pentyl-5-(4-bromophenyl)octa-1,3,4-trien-2-yl)-1,3,2-dioxaborolane **7ad** (zj-4-158-A).



Following **Typical Procedure V**, the reaction of B_2Pin_2 (0.0358 g, 0.14 mmol), $Cu(CH_3CN)_4PF_6$ (0.0028 g, 0.0075 mmol), dppf (0.0069 g, 0.0125 mmol), NaO^tBu (0.0199 g, 0.2 mmol), $Pd(dba)_2$ (0.0029 g, 0.005 mmol), **1a** (0.0234 g, 0.1 mmol), **6d** (0.0399 g, 0.14 mmol), and THF (1 mL) afforded **7ad** (0.0228 g, 42%, 95% purity) (eluent: petroleum ether (60~90 ºC)/ethyl acetate = 37.5/1 (308 mL)): liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.4 Hz, 2 H, ArH), 7.28 (d, *J* = 8.8 Hz, 2 H, ArH), 5.74 (d, 1 H, *J* = 2.0 Hz, one proton of $=CH_2$), 5.70 (d, *J* = 2.4 Hz, 1 H, one proton of $=CH_2$), 3.67 (s, 3 H, OCH_3), 2.51-2.34 (m, 4 H, $CH_2 \times 2$), 2.27 (t, *J* = 7.6 Hz, 2 H, CH_2), 1.94-1.82 (m, 2 H, CH_2), 1.50-1.37 (m, 2 H, CH_2), 1.29-1.24 (m, 4 H, $CH_2 \times 2$), 1.16 (s, 6 H, $CH_3 \times 2$), 1.14 (s, 6 H, $CH_3 \times 2$), 0.83 (t, *J* = 7.0 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.0, 174.0, 136.4, 131.1, 127.8, 125.4, 120.0, 110.3, 105.0, 83.5, 51.5, 33.8, 31.8, 29.69, 29.66, 27.4, 24.6, 24.5, 23.1, 22.5, 14.0; The carbon directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2977, 2929, 2858, 1925, 1740, 1586, 1487, 1314, 1144; MS (EI) *m/z*: 518 (M^+($^{11}B^{81}Br$), 7.48), 517 (M^+($^{10}B^{81}Br$), 3.69), 516 (M^+($^{11}B^{79}Br$), 7.24), 515 (M^+($^{10}B^{79}Br$), 1.84), 101 (100); HRMS calcd. for $C_{27}H_{38}{}^{10}B^{79}BrO_4Na$ $(M + Na)^+$: 538.1975; Found: 538.1973.

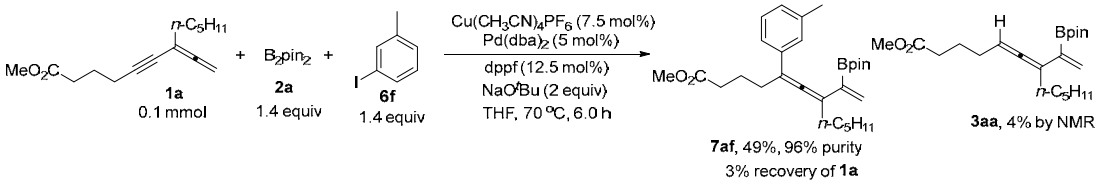
4.5. Preparation of 4,4,5,5-tetramethyl-2-(8-methoxycarbonyl-3-pentyl-5-(4-methoxyphenyl)octa-1,3,4-trien-2-yl)-1,3,2-dioxaborolane **7ae** (zj-4-155, zj-4-135).



Following **Typical Procedure V**, the reaction of B_2Pin_2 (0.0357 g, 0.14 mmol), $Cu(CH_3CN)_4PF_6$ (0.0028 g, 0.0075 mmol), dppf (0.0070 g, 0.0125 mmol), NaO^tBu (0.0195 g, 0.2 mmol), $Pd(dba)_2$ (0.0028 g, 0.005 mmol), **1a** (0.0234 g, 0.1 mmol), **6e**

(0.0330 g, 0.14 mmol), and THF (1 mL) afforded **7ae** (0.0220 g, 47%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 20/1 (315 mL)): liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (d, *J* = 9.2 Hz, 2 H, ArH), 6.83 (d, *J* = 9.2 Hz, 2 H, ArH), 5.71 (d, *J* = 2.4 Hz, 1 H, one proton of $=CH_2$), 5.64 (d, *J* = 2.4 Hz, 1 H, one proton of $=CH_2$), 3.80 (s, 3 H, OCH_3), 3.66 (s, 3 H, OCH_3), 2.54-2.36 (m, 4 H, CH_2 × 2), 2.26 (t, *J* = 7.6 Hz, 2 H, CH_2), 1.95-1.83 (m, 2 H, CH_2), 1.52-1.39 (m, 2 H, CH_2), 1.31-1.25 (m, 4 H, CH_2 × 2), 1.16 (s, 6 H, CH_3 × 2), 1.14 (s, 6 H, CH_3 × 2), 0.83 (t, *J* = 7.0 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.5, 174.1, 158.2, 129.6, 127.2, 124.3, 113.5, 109.9, 105.2, 83.4, 55.2, 51.4, 33.9, 31.9, 30.0, 29.8, 27.4, 24.6, 24.5, 23.2, 22.5, 14.0; The carbon directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2954, 2930, 2858, 1923, 1739, 1607, 1510, 1248, 1145; MS (EI) *m/z*: 468 (M^+(^{11}B), 69.10), 467 (M^+(^{10}B), 16.61), 267 (100); HRMS calcd. for $C_{28}H_{41}{}^{11}BO_5Na$ $(M + Na)^+$: 491.2939; Found: 491.2940.

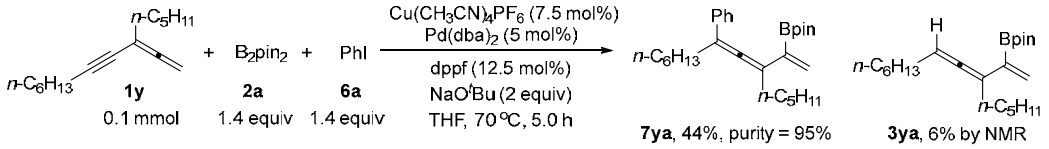
4.6. Preparation of 4,4,5,5-tetramethyl-2-(8-methoxycarbonyl-3-pentyl-5-(3-methylphenyl)octa-1,3,4-trien-2-yl)-1,3,2-dioxaborolane **7af** (zj-4-156).



Following **Typical Procedure V**, the reaction of B_2Pin_2 (0.0357 g, 0.14 mmol), $Cu(CH_3CN)_4PF_6$ (0.0028 g, 0.0075 mmol), dppf (0.0069 g, 0.0125 mmol), NaO^tBu (0.0194 g, 0.2 mmol), $Pd(dba)_2$ (0.0029 g, 0.005 mmol), **1a** (0.0234 g, 0.1 mmol), **6f** (0.0305 g, 0.14 mmol), and THF (1 mL) afforded **7af** (0.0233 g, 49%, 96% purity) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 30/1 (310 mL)): liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23-7.14 (m, 3 H, ArH), 6.98 (d, *J* = 7.2 Hz, 1 H, ArH), 5.73 (d,

J = 2.4 Hz, 1 H, one proton of =CH_2), 5.67 (d, J = 2.0 Hz, 1 H, one proton of =CH_2), 3.66 (s, 3 H, OCH_3), 2.54-2.38 (m, 4 H, CH_2 × 2), 2.33 (s, 3 H, CH_3), 2.27 (t, J = 7.6 Hz, 2 H, CH_2), 1.96-1.84 (m, 2 H, CH_2), 1.53-1.39 (m, 2 H, CH_2), 1.33-1.27 (m, 4 H, CH_2 × 2), 1.17 (s, 6 H, CH_3 × 2), 1.15 (s, 6 H, CH_3 × 2), 0.83 (t, J = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.9, 174.1, 137.5, 137.3, 127.9, 127.1, 126.9, 124.7, 123.3, 109.7, 105.7, 83.4, 51.4, 33.9, 31.9, 29.9, 29.8, 27.4, 24.7, 24.5, 23.2, 22.5, 21.5, 14.0; The carbon directly attached to the boron atom was not detected; IR (neat) ν (cm $^{-1}$) 2976, 2954, 2929, 1927, 1741, 1603, 1436, 1371, 1314, 1145; MS (EI) *m/z*: 452 (M^+(^{11}B), 72.92), 451 (M^+(^{11}B), 21.37), 251 (100); HRMS calcd. for $C_{28}H_{41}{}^{11}BO_4Na$ $(M + Na)^+$: 475.2990; Found: 475.2992.

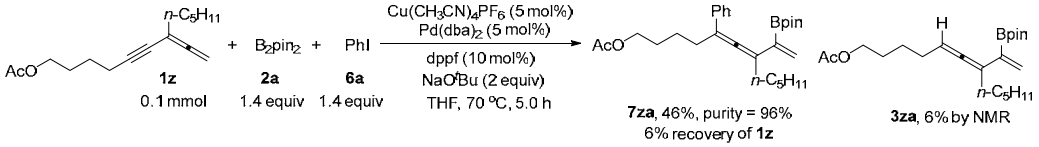
4.7. Preparation of 4,4,5,5-tetramethyl-2-(3-pentyl-5-phenylundeca-1,3,4-trien-2-yl)-1,3,2-dioxaborolane **7ya** (zj-4-164-A, syl-10-133).



Following **Typical Procedure V**: the reaction of B_2Pin_2 (0.0357 g, 0.14 mmol), $Cu(CH_3CN)_4PF_6$ (0.0028 g, 0.0075 mmol), dppf (0.0069 g, 0.0125 mmol), NaO^tBu (0.0195 g, 0.2 mmol), $Pd(dba)_2$ (0.0028 g, 0.005 mmol), **1y** (0.0218 g, 0.1 mmol), **6a** (0.0287 g, 0.14 mmol), and THF (1 mL) afforded **7ya** (0.0201 g, 44%, purity = 95%) (eluent: petroleum ether (60~90 °C)/DCM = 10/1 (330 mL)): liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44-7.38 (m, 2 H, ArH), 7.31-7.27 (m, 2 H, ArH), 7.18-7.12 (m, 1 H, ArH), 5.71 (d, J = 2.4 Hz, 1 H, one proton of =CH_2), 5.62 (d, J = 2.4 Hz, 1 H, one proton of =CH_2), 2.50-2.33 (m, 2 H, CH_2), 2.27 (t, J = 7.6 Hz, 2 H, CH_2), 1.55-1.34 (m, 6 H, CH_2 × 3), 1.33-1.27 (m, 8 H, CH_2 × 4), 1.15 (s, 6 H, CH_3 × 2), 1.13 (s, 6 H, CH_3 × 2), 0.88 (t, J = 7.0 Hz, 3 H, CH_3), 0.83 (t, J = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz,

$CDCl_3$) δ 207.2, 137.7, 128.0, 126.2, 126.1, 124.0, 109.5, 106.6, 83.4, 31.9, 31.8, 30.5, 29.7, 29.5, 27.9, 27.4, 24.61, 24.55, 22.7, 22.5, 14.1, 14.0; The carbon directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2956, 2927, 2857, 1926, 1597, 1313, 1145; MS (EI) *m/z*: 422 (M^+(^{11}B), 94.56), 421 (M^+(^{10}B), 23.65), 237 (100); HRMS calcd. for $C_{28}H_{43}{}^{11}BNaO_2$ $(M + Na)^+$: 445.3248; Found: 445.3243.

4.8. Preparation of 4,4,5,5-tetramethyl-2-(9-acetoxy-3-pentyl-5-phenylnona-1,3,4-trien-2-yl)-1,3,2-dioxaborolane **7za** (zj-4-161-A, syl-10-148).

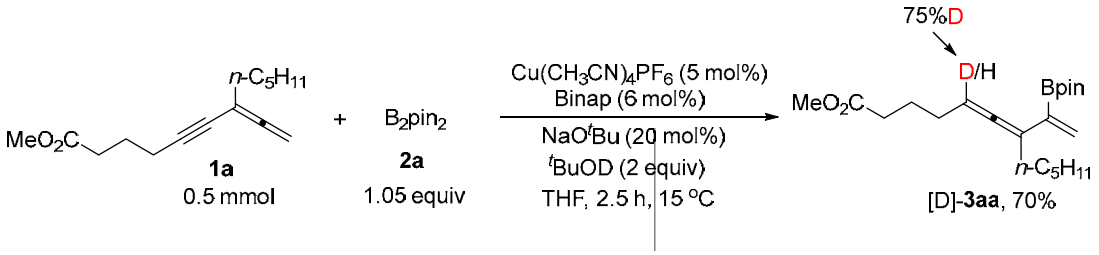


Following **Typical Procedure V**: the reaction of B_2Pin_2 (0.0357 g, 0.14 mmol), $Cu(CH_3CN)_4PF_6$ (0.0028 g, 0.0075 mmol), dppf (0.0069 g, 0.0125 mmol), NaO^tBu (0.0193 g, 0.2 mmol), $Pd(dba)_2$ (0.0029 g, 0.005 mmol), **1z** (0.0248 g, 0.1 mmol), **6a** (0.0287 g, 0.14 mmol), and THF (1 mL) afforded **7za** (0.0217 g, 46%, purity = 96%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 30/1 (310 mL)): liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42-7.37 (m, 2 H, ArH), 7.32-7.26 (m, 2 H, ArH), 7.19-7.14 (m, 1 H, ArH), 5.72 (d, *J* = 2.0 Hz, 1 H, one proton of $=CH_2$), 5.65 (d, *J* = 2.4 Hz, 1 H, one proton of $=CH_2$), 4.08 (t, *J* = 7.0 Hz, 2 H, OCH_2), 2.55-2.38 (m, 2 H, CH_2), 2.28 (t, *J* = 7.8 Hz, 2 H, CH_2), 2.03 (s, 3 H, CH_3), 1.79-1.69 (m, 2 H, CH_2), 1.68-1.60 (m, 2 H, CH_2), 1.52-1.42 (m, 2 H, CH_2), 1.33-1.27 (m, 4 H, CH_2 × 2), 1.15 (s, 6 H, CH_3 × 2), 1.13 (s, 6 H, CH_3 × 2), 0.83 (t, *J* = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.0, 171.2, 137.5, 128.0, 126.2, 126.1, 124.6, 109.8, 106.1, 83.4, 64.5, 31.9, 30.0, 29.7, 28.5, 27.4, 24.6, 24.5, 24.2, 22.5, 21.0, 14.0; The carbon directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2977, 2956, 2930, 2859, 1926, 1742, 1370, 1313, 1240, 1144; MS (EI) *m/z*: 452 (M^+(^{11}B), 70.46), 451 (M^+(^{10}B), 16.99), 236

(100); HRMS calcd. for $C_{28}H_{41}{}^{11}BNaO_4$ $(M + Na)^+$: 475.2990; Found: 475.2993.

5. Mechanistic studies.

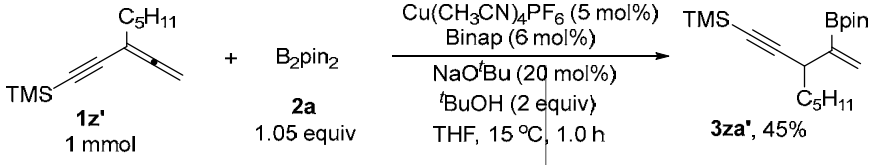
5.1. Preparation of [D]-**3aa** (syl-8-149).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.1337 g, 0.525 mmol), $Cu(CH_3CN)_4PF_6$ (0.0092 g, 0.025 mmol), BINAP (0.0187 g, 0.03 mmol), NaO^tBu (0.0097 g, 0.1 mmol), ^{t}BuOD (93 μL, d = 0.81 g/mL, 0.0753 g, 1 mmol), **1a** (0.1171 g, 0.5 mmol), and THF (2.5 mL) afforded [D]-**3aa** (0.1267 g, 70%) with about 75% deuterated ratio (eluent: petroleum ether (60~90 °C)/ethyl acetate = 25/1 (416 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.64 (d, *J* = 2.4 Hz, 1 H, one proton of $=CH_2$), 5.59 (d, *J* = 2.4 Hz, 1 H, one proton of $=CH_2$), 3.66 (s, 3 H, OCH_3), 2.37 (t, *J* = 7.4 Hz, 2 H, CH_2), 2.15 (t, *J* = 7.8 Hz, 2 H, CH_2), 2.06 (t, *J* = 7.2 Hz, 2 H, CH_2), 1.84-1.72 (m, 2 H, CH_2), 1.50-1.37 (m, 2 H, CH_2), 1.35-1.24 (m, 16 H, $CH_2 \times 2 + CH_3 \times 4$), 0.88 (t, *J* = 6.9 Hz, 3 H, CH_3), the following signal is disscernible for **3aa**: δ 5.23 (s, 0.25 H, =CH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.9, 174.0, 124.2, 106.8, 90.7 (t, *J* = 24.5 Hz), 83.4, 51.3, 33.6, 31.7, 29.3, 28.4, 27.3, 24.7, 24.6, 24.2, 22.5, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2977, 2954, 2930, 2858, 1936, 1741, 1585, 1379, 1372, 1311, 1146, 1118; MS (EI) *m/z*: 363 (M^+(^{11}B), 23.85), 362 (M^+(^{10}B), 13.48), 83 (100); HRMS calcd. for $C_{21}H_{34}DO_4{}^{11}B$ (M^+):

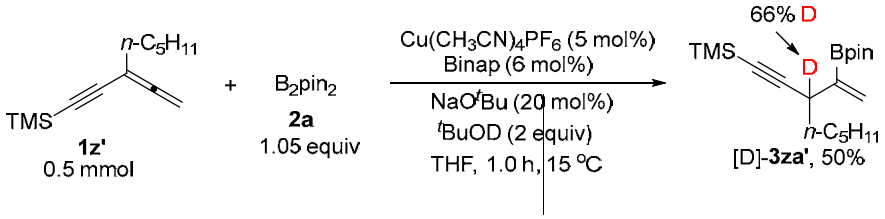
363.2691; Found: 363.2690.

5.2 Preparation of 4,4,5,5-tetramethyl-2-(5-trimethylsilyl-3-pentylpenta-1-en-4-yn-2-yl)-1,3,2-dioxaborolane **3za'** (syl-8-78).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.2672 g, 1.05 mmol), $Cu(CH_3CN)_4PF_6$ (0.0188 g, 0.05 mmol), BINAP (0.0372 g, 0.06 mmol), NaO^tBu (0.0195 g, 0.2 mmol), ^{t}BuOH (0.18 mL, d = 0.81 g/mL, 0.146 g, 2 mmol), **1z'** (0.2058 g, 1 mmol), and THF (5 mL) afforded **3za'** (0.1487 g, 45%) (eluent: petroleum ether (60~90 °C)/DCM = 10/1 (660 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.97 (s, 1 H, one proton of =CH), 5.89 (s, 1 H, one proton of $=CH_2$), 3.29 (t, J_1 = 7.2 Hz, 1 H, CH), 1.67-1.55 (m, 1 H, one proton of CH_2), 1.50-1.16 (m, 19 H, $CH_3 \times 4 + CH_2 \times 3$ + one proton of CH_2), 0.87 (t, J = 6.3 Hz, 3 H, CH_3), 0.14 (s, 9 H, $CH_3 \times 3$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 129.5, 108.6, 87.4, 83.4, 37.2, 35.2, 31.5, 26.6, 24.9, 24.4, 22.4, 14.0, 0.2; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2978, 2959, 2931, 2860, 2169, 1617, 1420, 1410, 1363, 1308, 1249, 1143, 1120; MS (EI) *m/z*: 334 (M^+(^{11}B), 9.61), 333 (M^+(^{10}B), 2.30), 73 (100); HRMS calcd. for $C_{19}H_{35}{}^{11}BSiO_2$ (M^+): 334.2499; Found: 334.2498.

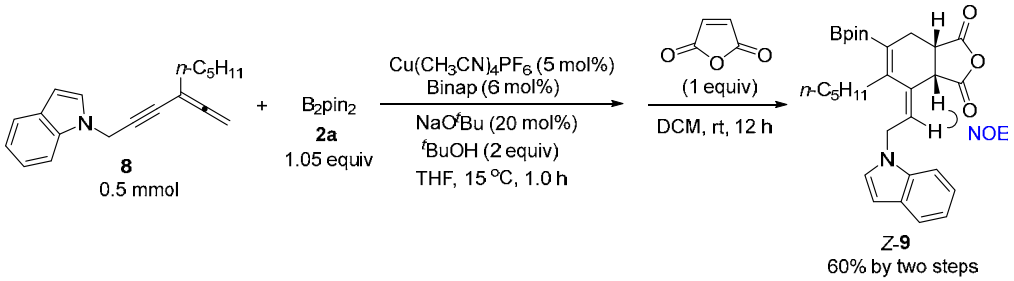
5.3. Preparation of [D]-**3za'** (syl-9-81).



Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.1335 g, 0.525 mmol), $Cu(CH_3CN)_4PF_6$ (0.0094 g, 0.025 mmol), BINAP (0.0188 g, 0.03 mmol), NaO^tBu (0.0096 g, 0.1 mmol), ^{t}BuOD (93 μL, d = 0.81 g/mL, 0.075 g, 1 mmol), **1z'** (0.1032 g, 0.5 mmol), and THF (2.5 mL) afforded [D]-**3za'** (0.0847 g, 50%) (eluent: petroleum ether (60~90 °C)/DCM = 10/1 (880 mL)): liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.99 (s, 1 H, =CH), 5.90 (s, 1 H, one proton of $=CH_2$), 1.67-1.55 (m, 1 H, one proton of CH_2), 1.50-1.15 (m, 19 H, $CH_3 \times 4 + CH_2 \times 3$ + one proton of CH_2), 0.89 (t, J = 8.2 Hz, 3 H, CH_3), 0.15 (s, 9 H, $CH_3 \times 3$), the following signal is disscernible for **7**: δ 3.30 (t, J = 9.2 Hz, 0.34 H, CH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 129.5, 108.6, 87.4, 83.5, 36.8 (t, J = 19.4 Hz), 35.1, 31.5, 26.6, 24.9, 24.4, 22.4, 14.0, 0.2; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2959, 2931, 2860, 2168, 1615, 1418, 1361, 1311, 1250, 1215, 1144; MS (EI) *m/z*: 335 (M^+(^{11}B), 3.56), 334 (M^+(^{10}B), 2.94), 73 (100); HRMS calcd. for $C_{19}H_{34}D^{11}BSiO_2$ (M^+): 335.2562; Found: 335.2564.

6. Synthetic transformations

6.1. Preparation of (3a*S*,7a*S*,*Z*)-4-(2-(1*H*-indol-1-yl)ethylidene)-5-pentyl-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-3a,4,7,7a-tetrahydroisobenzofuran-1,3-dione (3a*S*,7a*S*,*Z*)-**9** (syl-8-40, syl-8-41).[3]

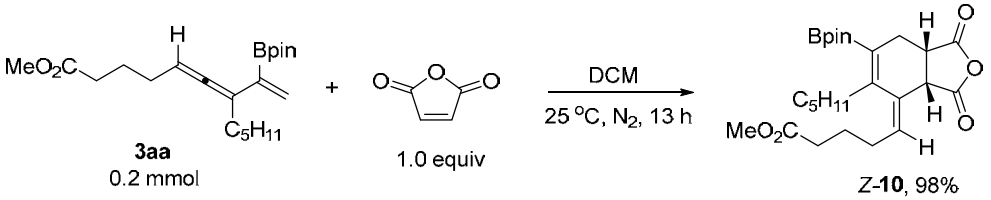


Following **Typical Procedure III**, the reaction of B_2Pin_2 (0.1337 g, 0.525 mmol), $Cu(CH_3CN)_4PF_6$ (0.0091 g, 0.025 mmol), BINAP (0.0187 g, 0.03 mmol), NaO^tBu (0.0095 g, 0.1 mmol), ^{t}BuOH (0.091 mL, d = 0.81 g/mL, 0.074 g, 1 mmol), **8** (0.1322 g, 0.5 mmol), and THF (2.5 mL) afforded the crude product, which was submitted to the next step without further purification.

To a flame-dried Schlenk tube were added the above-prepared crude product, DCM (2 mL), and maleic anhydride (0.0492 g, 0.5 mmol) under N_2 atmosphere. The resulting mixture was stirred at 25 °C for 12 h as monitored by TLC and filtrated through a short column of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford *Z*-**9** (0.1462 g, 60% yield from **8**) (eluent: petroleum ether/ethyl acetate = 10/1 (440 mL) to 8/1 (270 mL)): white solid; m.p. 116.8-117.9 °C (DCM/ petroleum ether); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.8 Hz, 1 H, ArH), 7.23-7.03 (m, 4 H, ArH), 7.51 (d, *J* = 3.0 Hz, 1 H, ArH), 5.60 (t, *J* = 6.5 Hz, 1 H, =CH), 4.88-4.78 (m, 2 H, NCH_2), 3.72 (d, *J* = 9.6 Hz, 1 H, CH), 3.44-3.35 (m, 1 H, CH), 3.20-3.00 (m, 2 H, CH_2), 2.28-2.13 (m, 1 H, one proton of CH_2), 2.02 (dd, J_1 = 14.4 Hz, J_2 = 6.0 Hz, 1 H, one proton of CH_2), 1.52-1.40 (m, 1 H, one proton of CH_2), 1.38-1.10 (m, 17 H, CH_3 × 4 + CH_2 × 2 + one proton of CH_2), 0.89 (t, *J* = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.2, 171.4, 155.4, 135.7, 133.1, 128.8, 128.4, 127.3, 121.7, 121.1, 119.6, 109.1, 101.8,

83.7, 50.9, 45.2, 14.0, 35.1, 31.7, 29.6, 26.8, 24.8, 24.6, 22.1, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (KBr) ν (cm^{-1}) 2977, 2959, 2931, 2861, 1854, 1783, 1591, 1512, 1463, 1371, 1324, 1247, 1216, 1144, 1054, 1009; MS (EI) *m/z*: 489 (M^+(^{11}B), 0.16), 488 (M^+(^{10}B), 0.08), 130 (100); Anal. Calcd. for $C_{29}H_{36}BNO_5$ (%): C 71.17, H 7.41, N 2.86; Found: C 71.16, H 7.43, N 2.64.

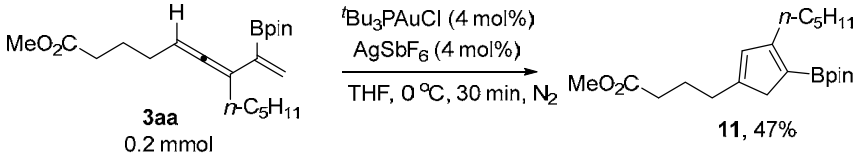
6.2. Preparation of methyl 5-((3a*S*,7a*S*,*Z*)-1,3-dioxo-5-pentyl-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-3,3a,7,7a-tetrahydroisobenzofuran-4(1*H*)-ylidene)pentanoate (3a*S*,7a*S*,*Z*)-**10** (syl-8-100).[3]



To a flame-dried Schlenk tube were added **3aa** (0.0716 g, 0.2 mmol), DCM (1 mL), and maleic anhydride (0.0207 g, 0.2 mmol) under N_2 atmosphere. The resulting mixture was stirred at 25 °C for 13 h as monitored by TLC. After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford *Z*-**10** (0.0896 g, 98%) (eluent: petroleum ether/ethyl acetate = 8/1 (360 mL) to 5/1 (240 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.52 (dd, J_1 = 8.3 Hz, J_2 = 6.2 Hz, 1 H, =CH), 3.76 (d, J = 9.6 Hz, 1 H, CH), 3.67 (s, 3 H, CH_3), 3.47-3.39 (m, 1 H, CH), 3.00-2.85 (m, 2 H, CH_2), 2.37-2.04 (m, 5 H, CH_2 × 2 + one proton of CH_2), 2.02-1.90 (m, 1 H, one proton of CH_2), 1.85-1.64 (m, 2 H, CH_2), 1.43-1.02 (m, 18 H, CH_3 × 4 + CH_2 × 3), 0.87 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.7, 173.5, 172.0, 156.9, 132.7, 130.9, 83.4, 51.4, 51.3, 41.6, 34.1, 33.2, 31.7, 29.5, 28.8, 26.6, 24.7, 24.53, 24.50, 22.1, 13.9; The

carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2976, 2955, 2932, 2861, 1854, 1782, 1738, 1591, 1367, 1326, 1304, 1216, 1144, 1054, 1011; MS (EI) *m/z*: 460 (M^+(^{11}B), 21.77), 459 (M^+(^{10}B), 4.85), 101 (100); HRMS calcd. for $C_{25}H_{37}{}^{11}BO_7$ (M^+): 460.2632; Found: 460.2633.

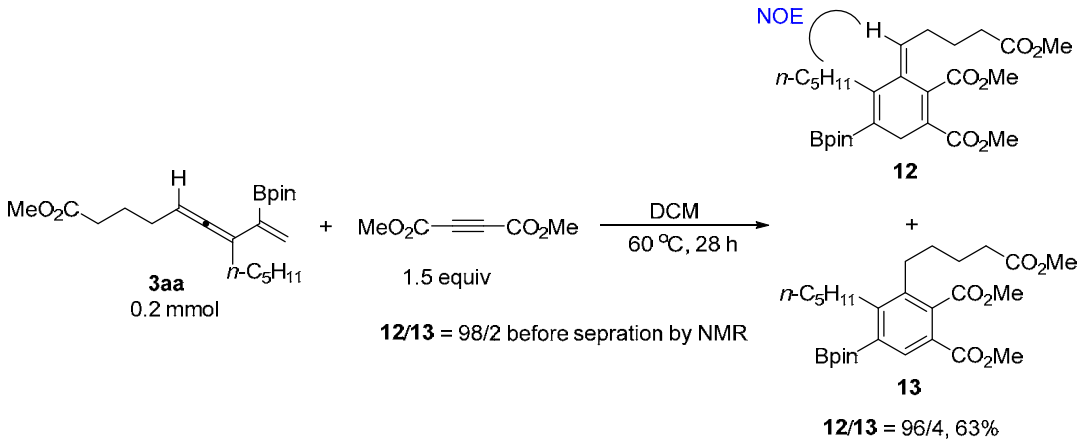
6.3. Preparation of methyl 4-(3-pentyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)cyclopenta-1,3-dien-1-yl)butanoate **11** (syl-8-155).[4]



To a flame-dried Schlenk tube were added ^{t}Bu$_3$PAuCl (0.0036 g, 0.008 mmol), $AgSbF_6$ (0.0036, 0.008 mmol), and THF (1 mL) under N_2 atmosphere. The resulting mixture was stirred with an ice-water bath for 5 minutes followed by the addition of **3aa** (0.0720 g, 0.2 mmol)/THF (3 mL) and stirring 0 °C for 30 minutes as monitored by TLC. The resulting mixture was filtrated through a short pad of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent under reduced pressure, the yield of the crude product was determined by ^{1}H NMR analysis using mesitylene (18.4 μL) as the internal standard (51% by NMR for product **11**). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **11** (0.0338 g, 47%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 20/1 (420 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.18 (s, 1 H, =CH), 3.66 (s, 3 H, OCH_3), 3.06 (s, 2 H, CH_2), 2.62 (t, *J* = 7.4 Hz, 2 H, CH_2), 2.44 (t, *J* = 7.5 Hz, 2 H, CH_2), 2.32 (t, *J* = 7.5 Hz, 2 H, CH_2), 1.93-1.82 (m, 2 H, CH_2), 1.55-1.43 (m, 2 H, CH_2), 1.38-1.23 (m, 16 H, CH_3 × 4

+ CH_2 × 2), 0.88 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 174.0, 163.8, 155.1, 131.2, 82.3, 51.4, 46.5, 33.6, 31.5, 30.2, 29.8, 29.4, 24.8, 24.6, 22.4, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2972, 2955, 2928, 2858, 1742, 1614, 1552, 1388, 1291, 1146, 1069; MS (EI) *m/z*: 362 (M^+(^{11}B), 10.54), 361 (M^+(^{10}B), 2.79), 162 (100); HRMS calcd. for $C_{21}H_{35}O_4{}^{11}B$ (M^+): 362.2628; Found: 362.2628.

6.4. Preparation of (*Z*)-1,2-dimethoxycarbonyl-3-(4-(methoxycarbonyl)butylidene)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-4-pentylcyclohexa-1,4-diene **12** (syl-8-158).[3]

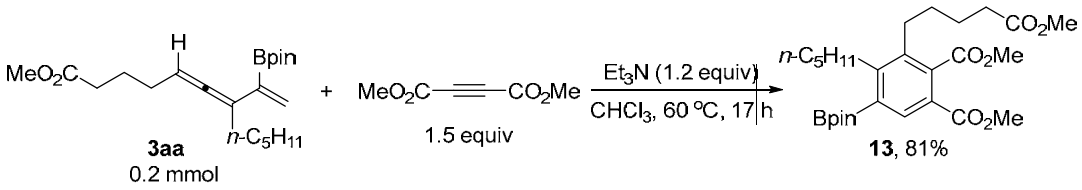


To a flame-dried Schlenk tube were added **3aa** (0.0721 g, 0.2 mmol)/DCM (0.5 mL), and dimethyl but-2-ynedioate (0.0435 g, 0.3 mmol)/DCM (0.5 mL) under N_2 atmosphere. The resulting mixture was stirred at 60 °C for 28 hours as monitored by TLC. After evaporation of the solvent under reduced pressure, the yield of the crude product was determined by ^1H NMR analysis using mesitylene (18.4 μL) as the internal standard (62% yield for **12** by NMR, 1% yield for **13** by NMR, **12**/**13** = 98/2 determined by ^1H NMR of crude product). After evaporation of the solvent, the residue was purified

by chromatography on silica gel to afford **12** and **13** (0.0659 g, **12**/**13** = 96/4, 63%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 7/1 (560 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.49 (t, *J* = 7.1 Hz, 1 H, =CH), 3.86 (s, 3 H, OCH_3), 3.75 (s, 3 H, OCH_3), 3.67 (s, 3 H, OCH_3), 3.15 (s, 2 H, CH_2), 2.74 (t, *J* = 7.4 Hz, 2 H, CH_2), 2.42-2.28 (m, 4 H, CH_2 × 2), 1.83-1.69 (m, 2 H, CH_2), 1.45-1.17 (m, 18 H, CH_3 × 4 + CH_2 × 3), 0.87 (t, *J* = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.6, 169.2, 166.2, 150.1, 144.1, 132.5, 131.6, 126.0, 83.3, 52.4, 52.0, 51.5, 33.4, 33.3, 31.5, 30.52, 30.47, 29.4, 25.0, 24.7, 22.4, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2976, 2953, 2925, 2859, 1738, 1639, 1586, 1435, 1372, 1337, 1270, 1202, 1144; MS (EI) *m/z*: 489 ((M – Me)$^+$ (^{11}B), 9.40), 488 ((M – Me)$^+$ (^{10}B), 33.49), 456 (100); HRMS calcd. for $C_{27}H_{41}{}^{11}BNaO_8$ (M + Na)$^+$: 527.2787; Found: 527.2789.

The following signals are discernible for **13**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.24 (s, 1 H, ArH), 3.94 (s, 3 H, OCH_3), 2.92 (s, 2 H, CH_2), 2.57 (s, 2 H, CH_2).

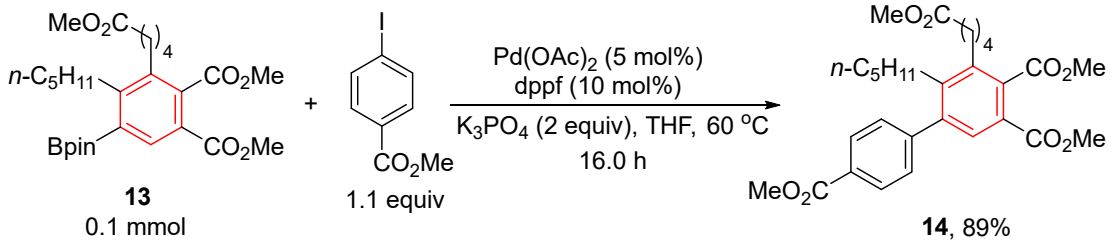
6.5. Preparation of dimethyl 3-(4-methoxycarbonylbutyl)-4-pentyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phthalate **13** (syl-8-170).[3]



To a flame-dried Schlenk tube were added **3aa** (0.0726 g, 0.2 mmol)/$CHCl_3$ (1 mL), dimethyl but-2-ynedioate (0.0430 g, 0.3 mmol)/DCM (1 mL), and Et_3N (33 μL, d = 0.728 g/mL, 0.0240 g, 0.24 mmol) under N_2 atmosphere. The resulting mixture was

stirred at 60 °C for 17 hours as monitored by TLC. After evaporation of the solvent under reduced pressure, the yield of the crude product was determined by ^{1}H NMR analysis using mesitylene (18.4 μL) as the internal standard (86% by NMR). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **13** (0.0823 g, 81%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 6/1 (420 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.24 (s, 1 H, ArH), 3.94 (s, 3 H, OCH_3), 3.87 (s, 3 H, OCH_3), 3.68 (s, 3 H, OCH_3), 2.93 (t, *J* = 7.2 Hz, 2 H, CH_2), 2.58 (t, *J* = 8.1 Hz, 2 H, CH_2), 2.35 (t, *J* = 7.4 Hz, 2 H, CH_2), 1.79-1.67 (m, 2 H, CH_2), 1.65-1.53 (m, 2 H, CH_2), 1.50-1.32 (m, 18 H, CH_3 × 4 + CH_2 × 3), 0.92 (t, *J* = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.8, 170.3, 166.3, 153.8, 137.8, 136.9, 135.5, 124.1, 83.8, 52.3, 52.1, 51.4, 33.6, 32.9, 32.4, 31.7, 30.8, 30.1, 25.3, 24.7, 22.4, 14.0; The carbon atom directly attached to the boron atom was not detected; IR (neat) ν (cm^{-1}) 2976, 2953, 2920, 2852, 1738, 1461, 1436, 1363, 1267, 1196, 1095; MS (EI) *m/z*: 504 (M^+(^{11}B), 0.29), 503 (M^+(^{10}B), 0.09), 474 ((M– Me × 2)$^+$, 18.11), 472 (100); HRMS calcd. for $C_{27}H_{41}O_8{}^{11}B$ (M^+): 504.2894; Found: 504.2897.

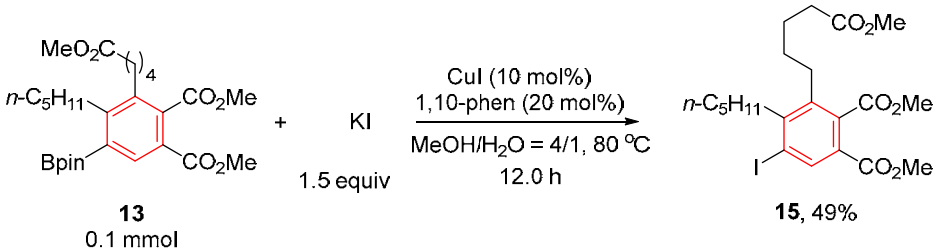
6.6. Preparation of trimethyl 5-(5-methoxy-5-oxopentyl)-6-pentyl-[1,1'-biphenyl]-3,4,4'-tricarboxylate **14** (zj-4-195-A).[5]



To a Schlenk tube were added methyl 4-iodobenzoate (0.0289 g, 0.11 mmol),

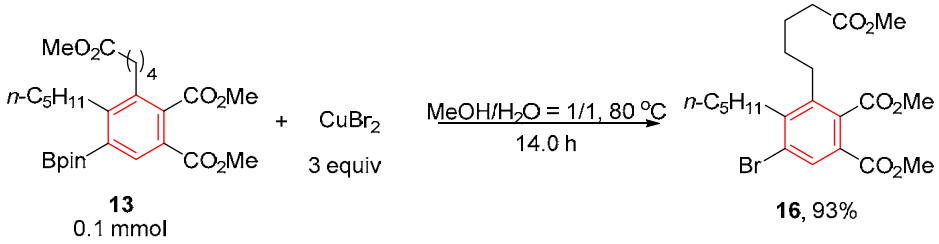
$Pd(OAc)_2$ (0.0011 g, 0.005 mmol), dppf (0.0055 g, 0.01 mmol), K_3PO_4 (0.0427 g, 0.2 mmol), **13** (0.0504 g, 0.1 mmol), and THF (1 mL) under N_2 atmosphere. The resulting mixture was stirred at 60 °C for 16 hours as monitored by TLC. The resulting mixture was filtrated through a pad of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **14** (0.0457 g, 89%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 6/1 (350 mL) to 5/1 (120 mL)): liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 8.0 Hz, 2 H, ArH), 7.60 (s, 1 H, ArH), 7.27 (d, *J* = 8.4 Hz, 2 H, ArH), 3.91 (s, 3 H, CO_2CH_3), 3.89 (s, 3 H, CO_2CH_3), 3.78 (s, 3 H, CO_2CH_3), 3.61 (s, 3 H, CO_2CH_3), 2.61-2.52 (m, 2 H, CH_2), 2.51-2.43 (m, 2 H, CH_2), 2.30 (t, *J* = 7.2 Hz, 2 H, CH_2), 1.74-1.63 (m, 2 H, CH_2), 1.61-1.50 (m, 2 H, CH_2), 1.27-1.13 (m, 2 H, CH_2), 1.08-0.97 (m, 4 H, CH_2 × 2), 0.67 (t, *J* = 6.4 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.8, 170.2, 166.8, 165.9, 145.9, 144.5, 142.7, 138.3, 135.4, 129.43, 129.36, 129.14, 129.12, 124.8, 52.6, 52.4, 52.2, 51.5, 33.6, 31.9, 31.0, 30.5, 30.4, 29.7, 25.4, 21.9, 13.7; IR (neat) ν (cm^{-1}) 2953, 2925, 1725, 1459, 1435, 1274, 1159; MS (ESI) *m/z*: 530 $(M + NH_4)^+$, 513 $(M + H)^+$; HRMS calcd. for $C_{29}H_{36}NaO_8$ $(M + Na)^+$: 535.2302; Found: 535.2306.

6.7. Preparation of dimethyl 5-iodo-3-(5-methoxy-5-oxopentyl)-4-pentylphthalate **15** (zj-5-5).[6]



To a sealed tube (10 mL) were added CuI (0.0018 g, 0.01 mmol), 1,10-phen (0.0036 g, 0.02 mmol), KI (0.0248 g, 0.15 mmol), **13** (0.0505 g, 0.1 mmol), and MeOH (1 mL)/H_2O (0.25 mL) under air atmosphere. The resulting mixture was stirred at 80 °C for 12 hours as monitored by TLC. The resulting mixture was filtrated through a pad of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **15** (0.0249 g, 49%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 20/3 (345 mL)): liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33 (s, 1 H, ArH), 3.93 (s, 3 H, CO_2CH_3), 3.86 (s, 3 H, CO_2CH_3), 3.67 (s, 3 H, CO_2CH_3), 2.83-2.76 (m, 2 H, CH_2), 2.64-2.57 (m, 2 H, CH_2), 2.34 (t, J = 7.5 Hz, 2 H, CH_2), 1.76-1.67 (m, 2 H, CH_2), 1.59-1.53 (m, 2 H, CH_2), 1.52-1.34 (m, 6 H, CH_2 × 3), 0.93 (t, J = 7.3 Hz, 3 H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 173.8, 169.6, 164.7, 149.1, 139.3 (two carbons), 138.1, 136.0, 126.4, 102.5, 52.6, 51.5, 37.9, 33.6, 32.1, 31.6, 30.8, 29.1, 25.3, 22.3, 14.0; IR (neat) ν (cm^{-1}) 2953, 2924, 1741, 1459, 1337, 1276; MS (EI) *m/z*: 504 (M^+, 0.6), 472 (100); HRMS calcd. for $C_{21}H_{29}INaO_6$ $(M + Na)^+$: 527.0901; Found: 527.0903.

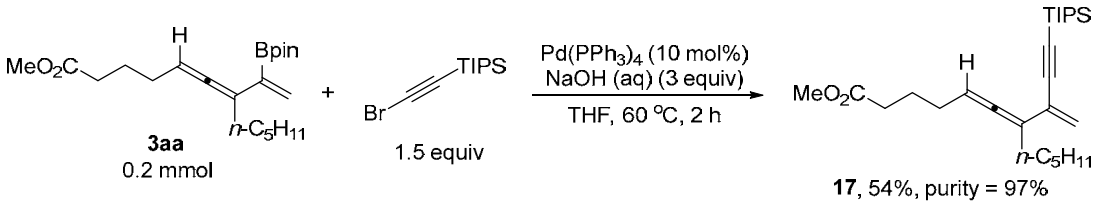
6.8. Preparation of dimethyl 5-bromo-3-(5-methoxy-5-oxopentyl)-4-pentylphthalate **16** (zj-5-6).[6]



To a sealed tube (10 mL) were added $CuBr_2$ (0.0670 g, 0.3 mmol), **13** (0.0504 g,

0.1 mmol), and MeOH (1 mL)/H_2O (1 mL) under air atmosphere. The resulting mixture was stirred at 80 °C for 14 hours as monitored by TLC. The resulting mixture was filtrated through a pad of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **16** (0.0425 g, 93%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 5/1 (420 mL)): liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.05 (s, 1 H, ArH), 3.93 (s, 3 H, CO_2CH_3), 3.86 (s, 3 H, CO_2CH_3), 3.67 (s, 3 H, CO_2CH_3), 2.81-2.74 (m, 2 H, CH_2), 2.63-2.55 (m, 2 H, CH_2), 2.34 (t, *J* = 7.5 Hz, 2 H, CH_2), 1.76-1.67 (m, 2 H, CH_2), 1.60-1.47 (m, 4 H, CH_2 × 2), 1.46-1.33 (m, 4 H, CH_2 × 2), 0.92 (t, *J* = 7.0 Hz, 3 H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 173.8, 169.5, 164.9, 146.2, 139.4, 135.1, 132.5 (two carbons), 126.4, 126.3, 52.6, 51.5, 33.6, 33.0, 32.1, 31.2, 30.8, 29.0, 25.3, 22.3, 14.0; IR (neat) ν (cm^{-1}) 2953, 2925, 1740, 1457, 1278, 1165; MS (EI) *m/z*: 458 (M^+(^{81}Br), 0.26), 456 (M^+(^{79}Br), 0.23), 426 (100); HRMS calcd. for $C_{21}H_{29}BrNaO_6$ $(M + Na)^+$: 479.1040; Found: 479.1039.

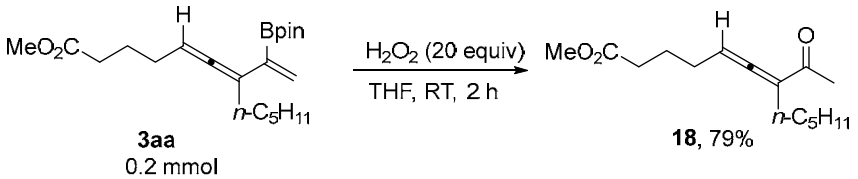
6.9. Preparation of methyl 8-((triisopropylsilyl)ethynyl)-7-pentylnona-5,6,8-trienoate **17** (syl-8-150).[7]



To a Schlenk tube were added $Pd(PPh_3)_4$ (0.0227 g, 0.02 mmol), **3aa** (0.0726 g, 0.2 mmol)/THF (1 mL), (bromoethynyl)triisopropylsilane (0.0785 g, 0.3 mmol)/THF (1 mL), and NaOH aqueous solution (3 M, 0.2 mL, 0.6 mmol) under N_2 atmosphere.

The resulting mixture was stirred at 60 ºC for 2 hours as monitored by TLC and filtrated through a short column of silica gel and eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent under reduced pressure, the yield of the crude product was determined by ^{1}H NMR analysis using mesitylene (18.4 μL) as the internal standard (59% by NMR). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **17** (0.0465 g, Purity = 97%, 54%) (eluent: petroleum ether (60~90 ºC)/DCM = 5/1 (480 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.91 (t, *J* = 7.5 Hz, 1 H, =CH), 4.98-4.90 (m, 2 H, =CH_2), 3.66 (s, 3 H, OCH_3), 2.46 (q, *J* = 7.4 Hz, 2 H, CH_2), 2.33 (t, *J* = 7.8 Hz, 2 H, CH_2), 2.20-2.08 (m, 2 H, CH_2), 1.85-1.73 (m, 2 H, CH_2), 1.53-1.40 (m, 2 H, CH_2), 1.37-1.27 (m, 4 H, CH_2 × 2), 1.12-0.94 (m, 21 H, CH_3 × 6 + CH × 3), 0.89 (t, *J* = 6.3 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 209.1, 174.0, 135.7, 122.0, 104.9, 103.1, 95.7, 78.0, 51.5, 33.6, 31.6, 30.5, 28.9, 27.4, 24.4, 22.5, 18.6, 14.0, 11.3; IR (neat) ν (cm^{-1}) 2942, 2865, 2148, 1941, 1743, 1463, 1436, 1365, 1245, 1199; MS (EI) *m/z*: 416 (M^+, 2.94), 373 (100); HRMS calcd. for $C_{26}H_{44}O_2Si$ (M^+): 416.3111; Found: 416.3109.

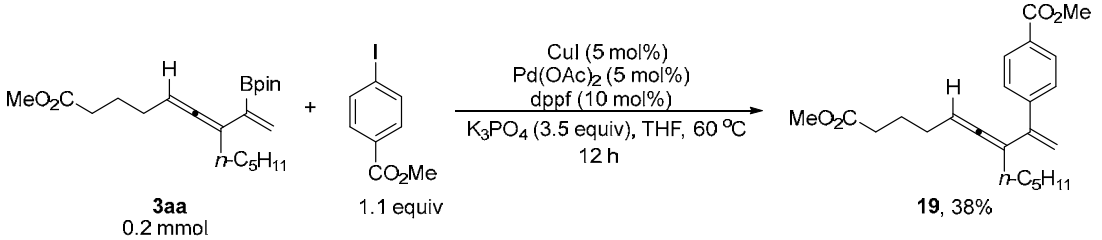
6.10. Preparation of methyl 7-acetyldodeca-5,6-dienoate **18** (syl-8-151).[8]



To a Schlenk tube were added **3aa** (0.0731 g, 0.2 mmol) and THF (2 mL). After cooling to 0 ºC, H_2O_2 (wt.30% in H_2O, 0.4 mL, d = 1.11 g/mL, 4 mmol) was added. Then the cooling bath was removed and THF (2 mL) was added. After the resulting

mixture was stirred at room temperature for 2 hours as monitored by TLC, it was quenched with saturated $Na_2S_2O_3$ solution (5 mL), extracted with ethyl ether (10 mL × 3), combined the organic phase, and dried over anhydrous Na_2SO_4. After filtration and evaporation of the solvent, the yield of the crude product was determined by ^1H NMR analysis using mesitylene (18.4 μL) as the internal standard (84% by NMR). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **18** (0.0401 g, 79%) (eluent: petroleum ether (60~90 ºC)/ ethyl acetate = 15/1 (384 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 5.61-5.53 (m, 1 H, =CH), 3.69 (s, 3 H, OCH_3), 2.40 (t, *J* = 7.4 Hz, 2 H, CH_2), 2.27 (s, 3 H, CH_3), 2.26-2.12 (m, 4 H, CH_2 × 2), 1.88-1.77 (m, 2 H, CH_2), 1.43-1.22 (m, 6 H, CH_2 × 3), 0.88 (t, *J* = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 212.3, 199.1, 173.5, 110.1, 94.5, 51.5, 33.3, 31.4, 27.7, 27.6, 26.9, 26.4, 24.2, 22.4, 13.9; IR (neat) ν (cm^{-1}) 2955, 2929, 2859, 1944, 1741, 1678, 1437, 1358, 1251; MS (EI) *m/z*: 252 (M^+, 30.57), 165 (100); HRMS calcd. for $C_{15}H_{24}O_3$ (M^+): 252.1725; Found: 252.1724.

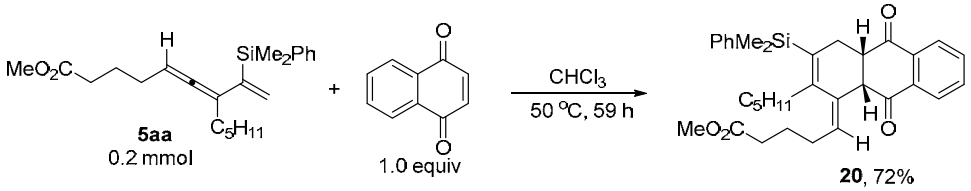
6.11. Preparation of methyl 4-(9-methoxy-9-oxo-3-pentyl-4l5-nona-1,3,4-trien-2-yl)benzoate **19** (zj-4-191, zj-4-188).



To a Schlenk tube were added methyl 4-iodobenzoate (0.0579 g, 0.22 mmol), $Pd(OAc)_2$ (0.0023 g, 0.01 mmol), dppf (0.0114 g, 0.02 mmol), K_3PO_4 (0.1479 g, 0.7

mmol), CuI (0.0020 g, 0.01 mmol), **3aa** (0.0725 g, 0.2 mmol), and THF (2 mL) under N_2 atmosphere. The resulting mixture was stirred at 60 °C for 12 hours as monitored by TLC. The resulting mixture was filtrated through a pad of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **19** (0.0281 g, 38%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 70/1 (710 mL)): liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.5 Hz, 2 H, ArH), 7.34 (d, *J* = 8.5 Hz, 2 H, ArH), 5.27 (s, 1 H, one proton of $=CH_2$), 5.14 (s, 1 H, one proton of $=CH_2$), 5.09-5.03 (m, 1 H, =CH), 3.91 (s, 3 H, CO_2CH_3), 3.63 (s, 3 H, CO_2CH_3), 2.27-2.20 (m, 2 H, CH_2), 2.14-2.06 (m, 2 H, CH_2), 1.99-1.86 (m, 2 H, CH_2), 1.62-1.54 (m, 2 H, CH_2), 1.52-1.46 (m, 2 H, CH_2), 1.37-1.29 (m, 4 H, CH_2 × 2), 0.90 (t, *J* = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 206.1, 173.9, 167.0, 146.9, 146.5, 129.0, 128.8, 128.3, 113.4, 106.7, 92.5, 52.0, 51.4, 33.0, 31.6, 30.0, 28.2, 27.5, 24.2, 22.5, 14.1; IR (neat) ν (cm^{-1}) 2953, 2930, 1942, 1724, 1608, 1436, 1277, 1177, 1019; MS (EI) *m/z*: 370 (M^+, 100); HRMS calcd. for $C_{23}H_{30}NaO_4$ $(M + Na)^+$: 393.2036; Found: 393.2036.

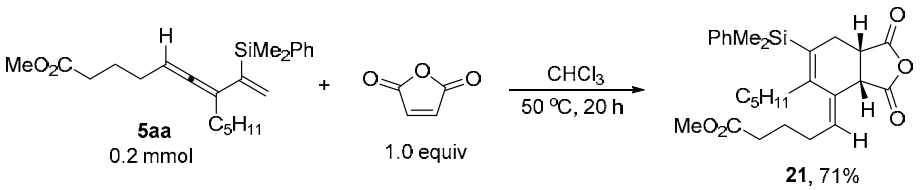
6.12. Preparation of methyl 5-((4a*S*,9a*S*,*Z*)-3-(dimethyl(phenyl)silyl)-9,10-dioxo-2-pentyl-4a,9,9a,10-tetrahydroanthracen-1(4*H*)-ylidene)pentanoate **20** (syl-10-71).[9]



To a flame-dried Schlenk tube were added **5aa** (0.0742 g, 0.2 mmol), $CHCl_3$ (2 mL), and naphthalene-1,4-dione (0.0316 g, 0.2 mmol) under N_2 atmosphere. The

resulting mixture was stirred at 50 °C for 59 h as monitored by TLC. After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **20** (0.0757 g, 72%) (eluent: petroleum ether/ethyl acetate = 10/1 (440 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.12-7.93 (m, 2 H, ArH), 7.80-7.66 (m, 2 H, ArH), 7.45-7.35 (m, 2 H, ArH), 7.35-7.28 (m, 3 H, ArH), 5.26 (t, *J* = 6.8 Hz, 1 H, =CH), 3.89 (d, *J* = 6.0 Hz, 1 H, CH), 3.65 (s, 3 H, OCH_3), 3.41 (q, *J* = 6.3 Hz, 1 H, CH), 2.71 (dd, J_1 = 17.1 Hz, J_2 = 7.2 Hz, 1 H, one proton of CH_2), 2.40-2.06 (m, 7 H, CH_2 × 3 + one proton of CH_2), 1.75-1.60 (m, 2 H, CH_2), 1.20-0.60 (m, 9 H, CH_3 + CH_2 × 3), 0.38 (s, 3 H, CH_3), 0.35 (s, 3 H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 198.0, 197.0, 173.7, 149.1, 139.3, 135.4, 134.3, 134.2, 134.1, 133.9, 133.6, 132.0, 130.9, 128.8, 127.7, 126.9, 126.7, 57.8, 51.5, 48.4, 36.5, 33.4, 31.9, 31.4, 28.9, 28.8, 25.1, 22.4, 14.0, -1.0, -1.1; IR (neat) ν (cm^{-1}) 2953, 2870, 1738, 1689, 1595, 1428, 1283, 1249, 1109; IR (neat) ν (cm^{-1}) 2953, 2870, 1738, 1689, 1595, 1428, 1283, 1249, 1109; MS (EI) *m/z*: 528 (M^+, 5.53), 135 (100); HRMS calcd. for $C_{33}H_{40}NaO_4Si$ $(M + Na)^+$: 551.2588; Found: 551.2591.

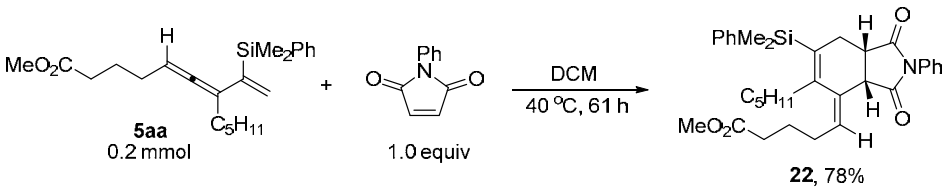
6.13. Preparation of methyl 5-((3a*S*,7a*S*,*Z*)-6-(dimethyl(phenyl)silyl)-1,3-dioxo-5-pentyl-3,3a,7,7a-tetrahydroisobenzofuran-4(1*H*)-ylidene)pentanoate **21** (syl-10-81).[9]



To a flame-dried Schlenk tube were added **5aa** (0.0743 g, 0.2 mmol), $CHCl_3$ (2 mL), and maleic anhydride (0.0199 g, 0.2 mmol) under N_2 atmosphere. The resulting mixture was stirred at 50 °C for 20 h as monitored by TLC. After evaporation of the

solvent, the residue was purified by chromatography on silica gel to afford **21** (0.0671 g, 71%) (eluent: petroleum ether/ethyl acetate = 5/1 (360 mL)): liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53-7.44 (m, 2 H, ArH), 7.40-7.30 (m, 3 H, ArH), 5.47 (dd, J_1 = 8.0 Hz, J_2 = 6.0 Hz, 1 H, =CH), 3.76 (d, J = 9.5 Hz, 1 H, CH), 3.66 (s, 3 H, OCH_3), 3.47-3.40 (m, 1 H, CH), 2.80 (dd, J_1 = 14.5 Hz, J_2 = 2.5 Hz, 1 H, one proton of CH_2), 2.35-2.28 (m, 2 H, CH_2), 2.24-2.04 (m, 4 H, CH_2 × 2), 2.00-1.92 (m, 1 H, one proton of CH_2), 1.82-1.65 (m, 2 H, CH_2), 1.32-1.22 (m, 1 H, one proton of CH_2), 1.15-1.05 (m, 2 H, CH_2), 1.03-0.88 (m, 2 H, CH_2), 0.81-0.70 (m, 4 H, CH_3 + one proton of CH_2), 0.43 (s, 3 H, CH_3), 0.42 (s, 3 H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ 174.1, 173.5, 171.9, 153.5, 138.5, 135.5, 133.7, 131.6, 130.4, 129.1, 127.9, 51.6, 41.9, 35.6, 33.4, 31.8, 28.7, 28.6, 27.9, 24.6, 22.2, 13.9, -1.2, -1.7; IR (neat) ν (cm^{-1}) 2954, 2860, 1855, 1779, 1738, 1565, 1428, 1250, 1220, 1110, 1087, 1047, 1005; MS (EI) *m/z*: 468 (M^+, 3.49), 135 (100); HRMS calcd. for $C_{27}H_{36}NaO_5Si$ $(M + Na)^+$: 491.2224; Found: 491.2226.

6.14. Preparation of methyl 5-((3a*S*,7a*S*,*Z*)-6-(dimethyl(phenyl)silyl)-1,3-dioxo-5-pentyl-2-phenyl-1,2,3,3a,7,7a-hexahydro-4*H*-isoindol-4-ylidene)pentanoate **22** (syl-10-63).[9]

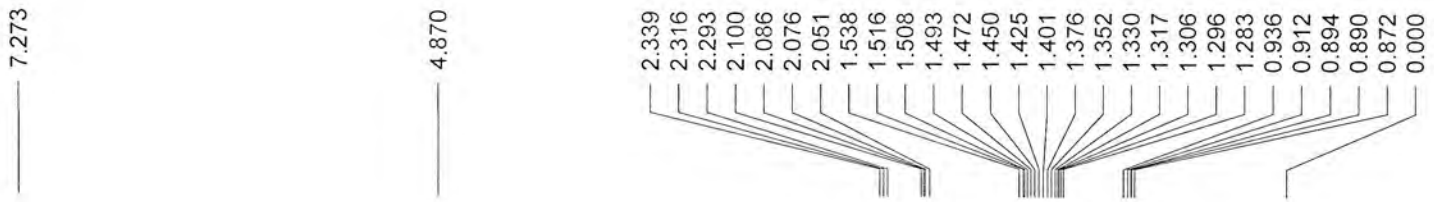


To a flame-dried Schlenk tube were added **5aa** (0.0742 g, 0.2 mmol), DCM (2 mL), and 1-phenyl-1*H*-pyrrole-2,5-dione (0.0350 g, 0.2 mmol) under N_2 atmosphere. The resulting mixture was stirred at 40 °C for 61 h as monitored by TLC. After evaporation

of the solvent, the residue was purified by chromatography on silica gel to afford **22** (0.0847 g, 78%) (eluent: petroleum ether/ethyl acetate = 6/1 (700 mL)): liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50-7.40 (m, 4 H, ArH), 7.39-7.35 (m, 1 H, ArH), 7.35-7.30 (m, 1 H, ArH), 7.28-7.22 (m, 2 H, ArH), 7.20-7.13 (m, 2 H, ArH), 5.48 (dd, J_1 = 8.5 Hz, J_2 = 6.0 Hz, 1 H, =CH), 3.72-3.60 (m, 4 H, CH + CH_3), 3.37-3.27 (m, 1 H, CH), 2.95 (dd, J_1 = 14.5 Hz, J_2 = 2.5 Hz, 1 H, one proton of CH_2), 2.40-2.27 (m, 2 H, CH_2), 2.26-2.06 (m, 4 H, CH_2 × 2), 2.04-1.97 (m, 1 H, one proton of CH_2), 1.84-1.67 (m, 2 H, CH_2), 1.34-1.20 (m, 1 H, one proton of CH_2), 1.10-0.93 (m, 3 H, CH_2 + one proton of CH_2), 0.93-0.80 (m, 1 H, one proton of CH_2), 0.74-0.57 (m, 4 H, CH_3 + one proton of CH_2), 0.44 (s, 3 H, CH_3), 0.42 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 178.8, 176.5, 173.7, 153.2, 138.9, 134.6, 133.7, 132.3, 132.1, 130.6, 129.0, 128.9, 128.4, 127.8, 126.3, 51.5, 51.3, 41.1, 35.8, 33.4, 31.9, 29.1, 28.8, 28.0, 24.7, 22.2, 13.8, -1.0, -1.6; IR (neat) ν (cm^{-1}) 2953, 2858, 1737, 1713, 1499, 1377, 1249, 1181; MS (EI) *m/z*: 543 (M^+, 31.98), 135 (100); HRMS calcd. for $C_{33}H_{41}NNaO_4Si$ $(M + Na)^+$: 566.2697; Found: 566.2698.

References:

1. Condon-Gueugnot, S.; Linstrumelle, G. *Tetrahedron* **2000**, *56*, 1851-1857.
2. Girard, D.; Broussous, S.; Provot, O.; Brion, J.; Alami, M. *Tetrahedron Letters* **2007**, *48*, 6022-6026.
3. Wang, H.; Luo, H.; Zhang, Z.; Zheng, W.; Yin, Y.; Qian, H.; Zhang, J.; Ma, S. *J. Am. Chem. Soc*. **2020**, *142*, 9763-9771.
4. Lee, J. H.; Toste, F. D. *Angew. Chem., Int. Ed*. **2007**, *119*, 930-932.
5. Němec, M. Hylsová, L. Maier, J. Flegel, S. Sievers, S. Ziegler, M. Schröder, B.-T. Berger, A. Chaikuad, B. Valčíková, S. Uldrijan, S. Drápela, K. Souček, H. Waldmann, S. Knapp, K. Paruch, *Angew. Chem. Int. Ed.* **2019**, *58*, 1062-1066.
6. G. Wang, L. Liu, H. Wang, Y.-S. Ding, J. Zhou, S. Mao, P. Li, *J. Am. Chem. Soc.* **2017**, *139*, 91-94.
7. Yu, S.; Gong, T.; Fu, Y. *Org. Lett*. **2020**, *22*, 2941-2945.
8. Yang, C.; Liu, Z.; Dai, D.; Li, Q.; Ma, W.; Zhao, M.; Xu. Y. *Org. Lett*. **2020**, *22*, 1360-1367.
9. H. Wang, H. Luo, Z. Zhang, W. Zheng, Y. Yin, H. Qian, J. Zhang, S. Ma, *J. Am. Chem. Soc*. **2020**, *142*, 9763−9771.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1.472
1.450
1.425
1.401
1.376
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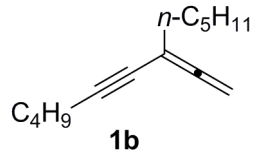
0.000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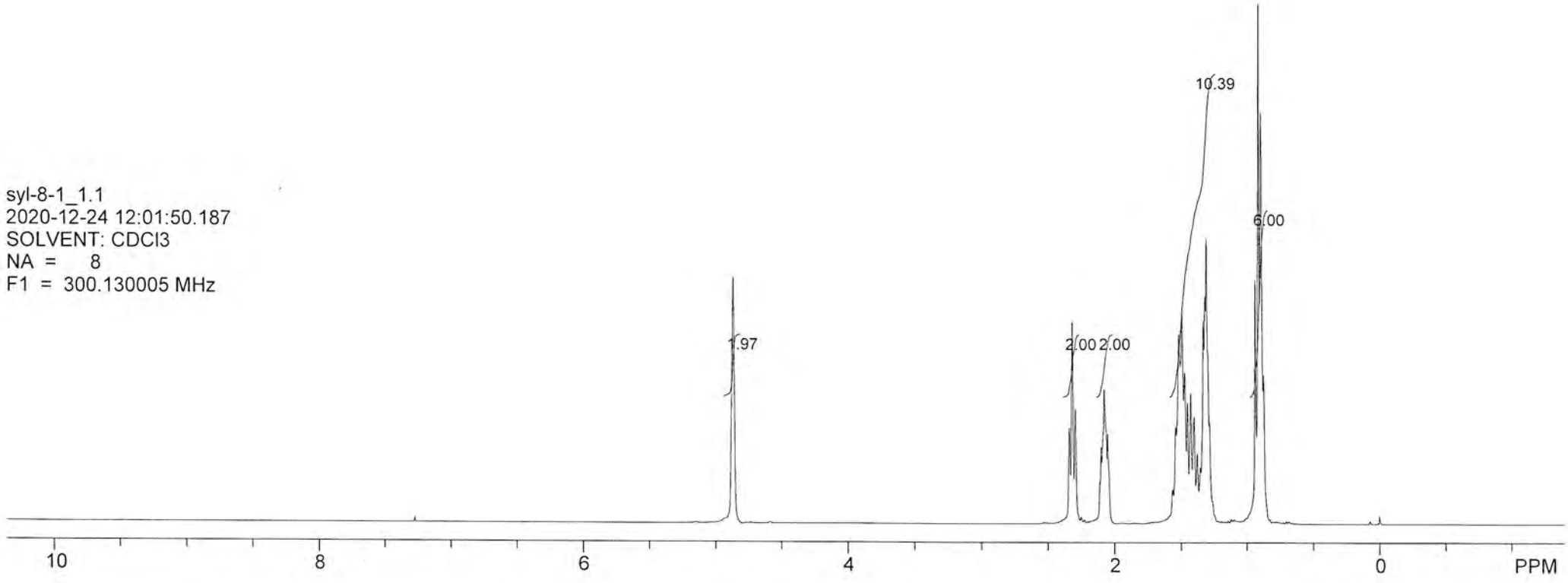
1b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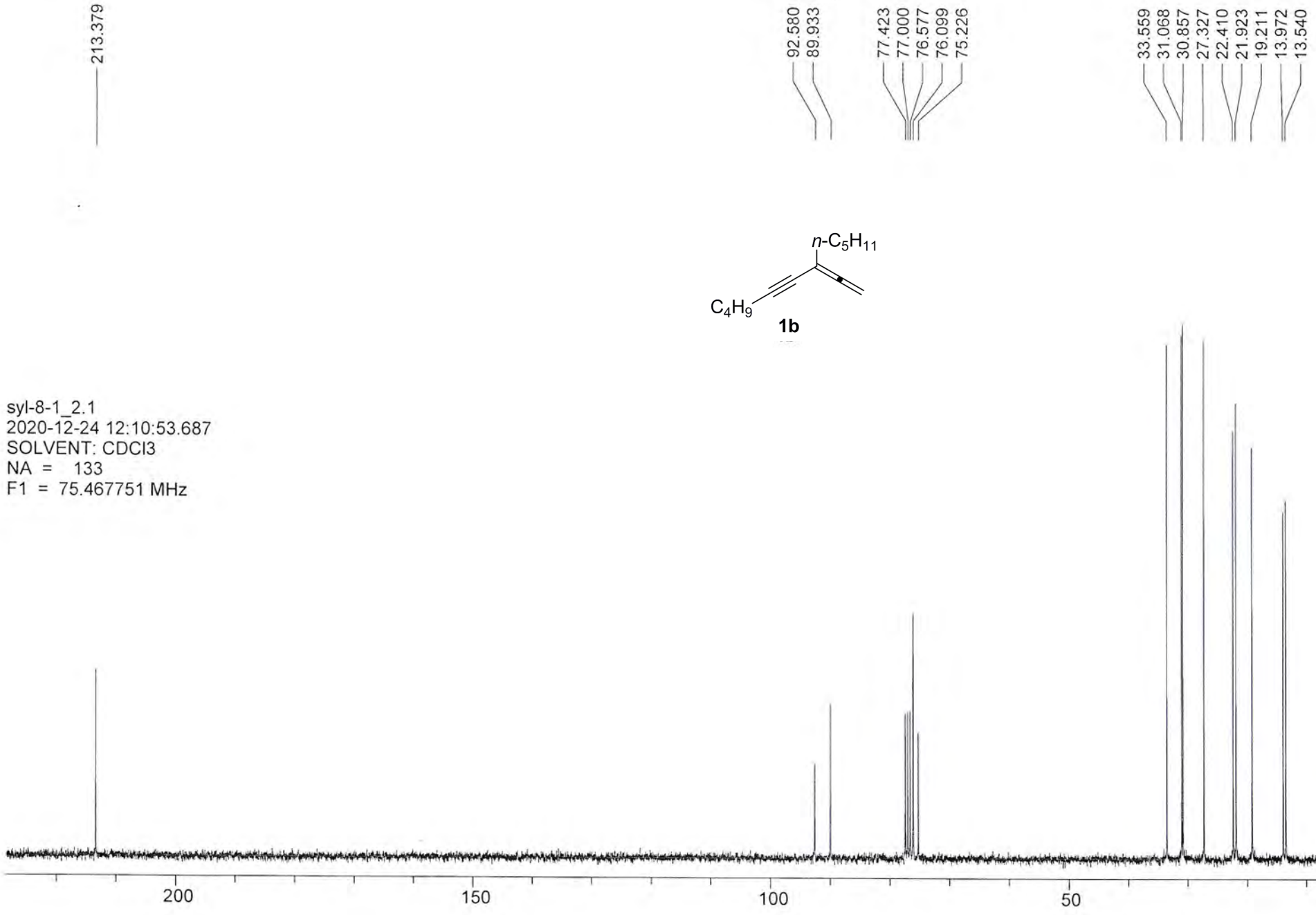
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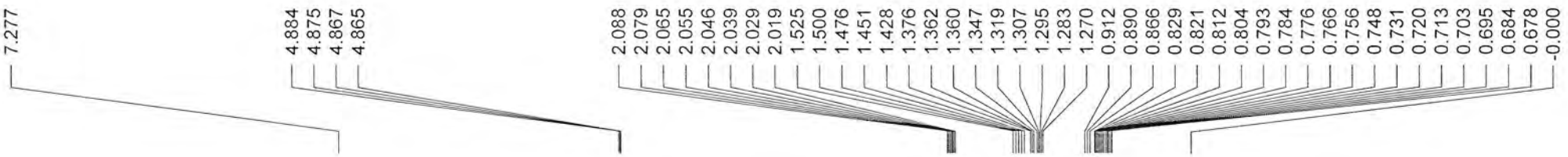
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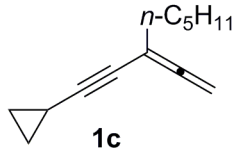
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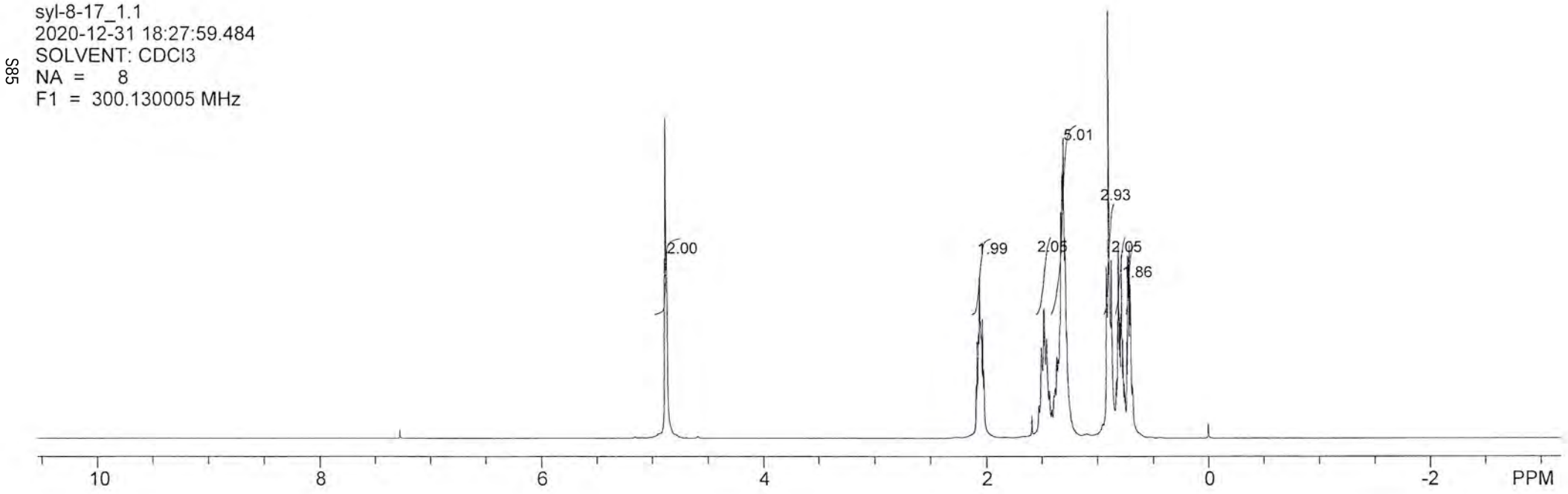
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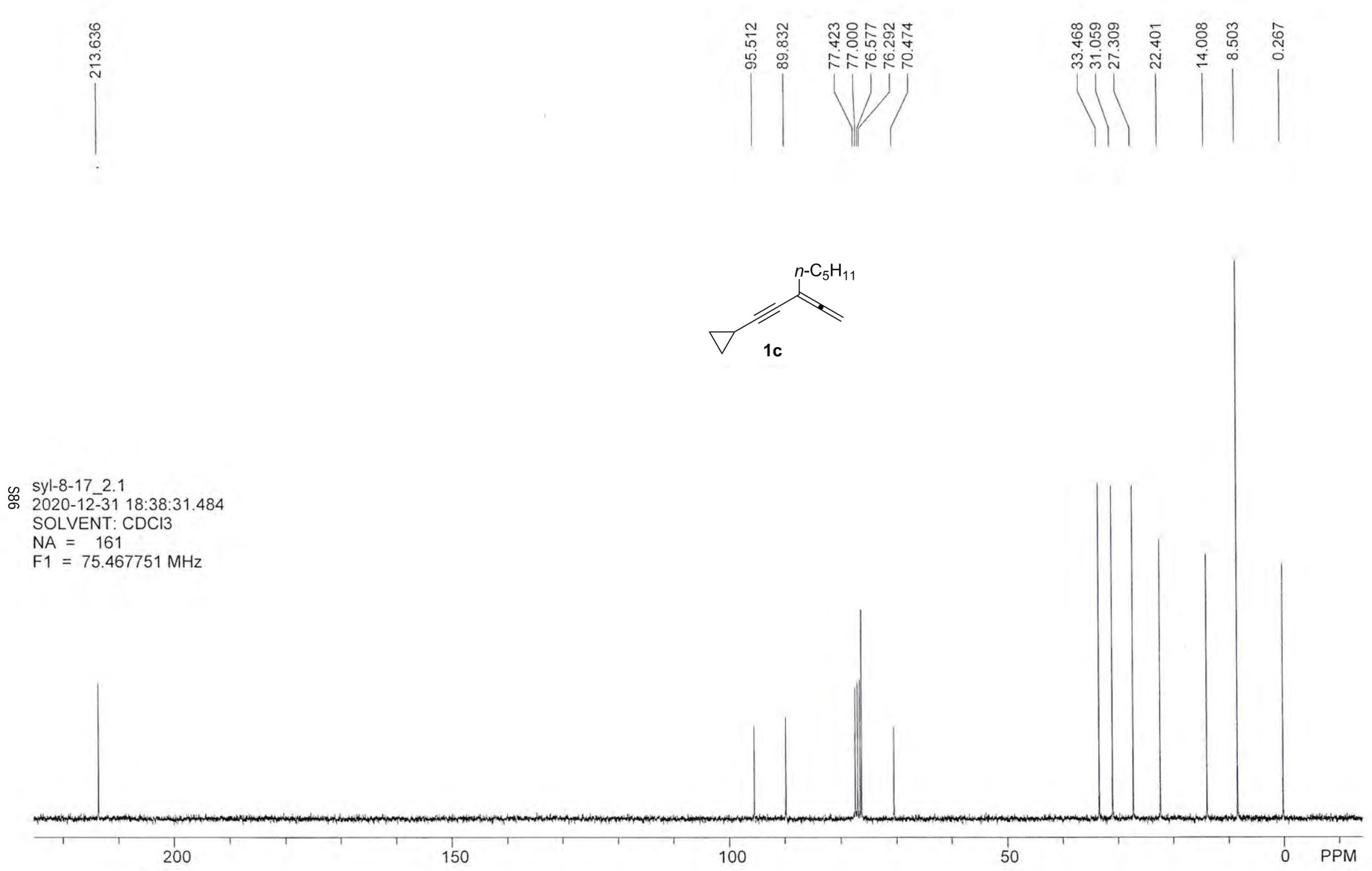
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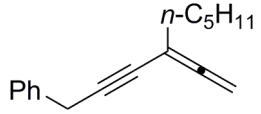
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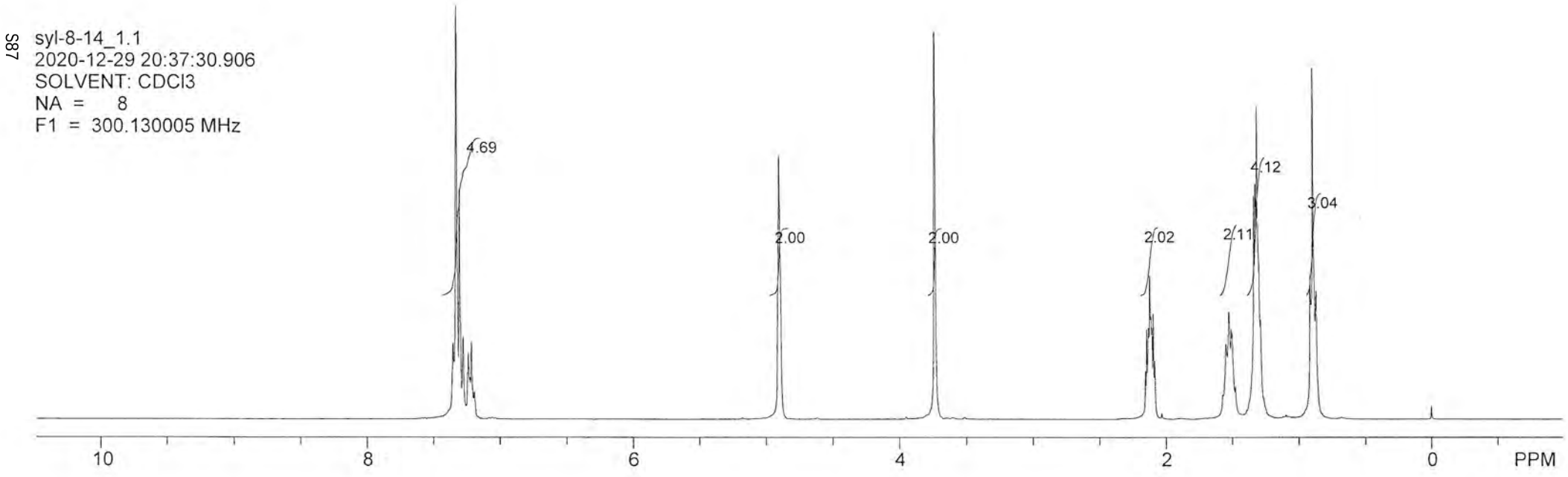
1d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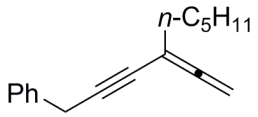
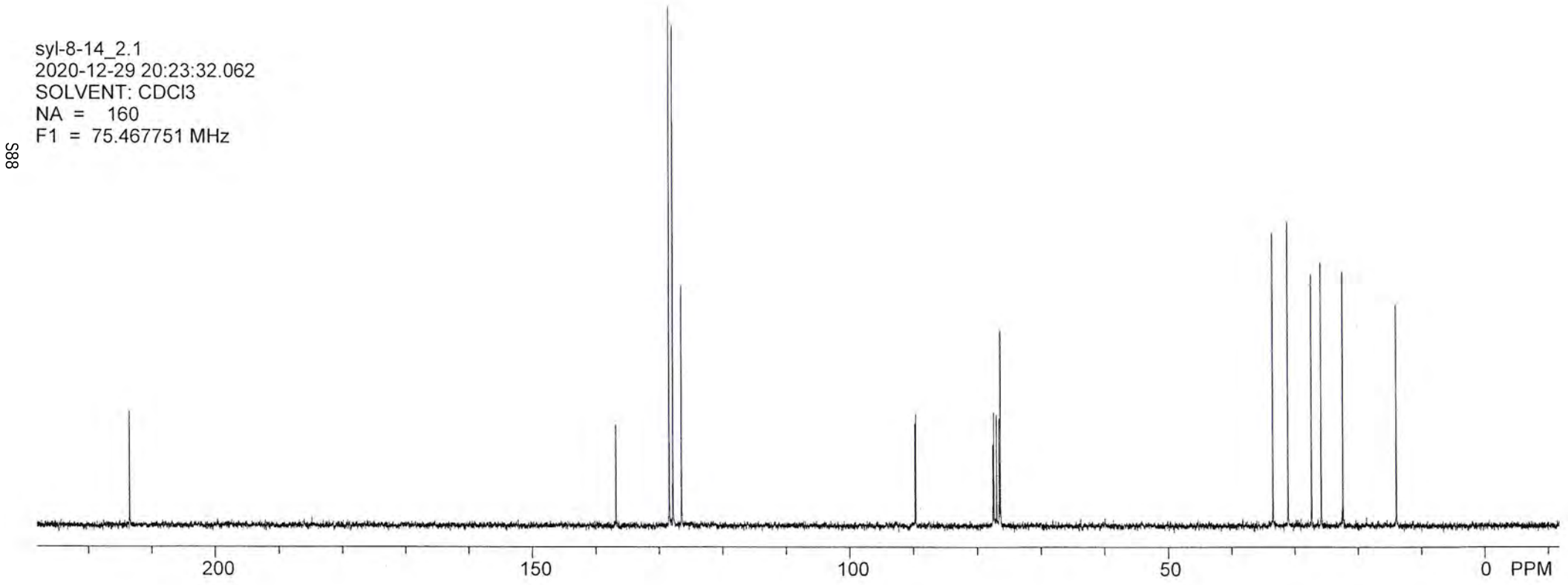
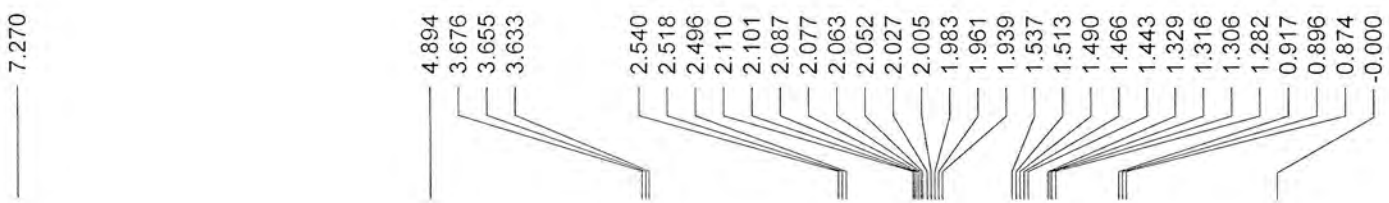
14.008



1d

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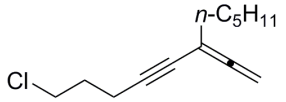
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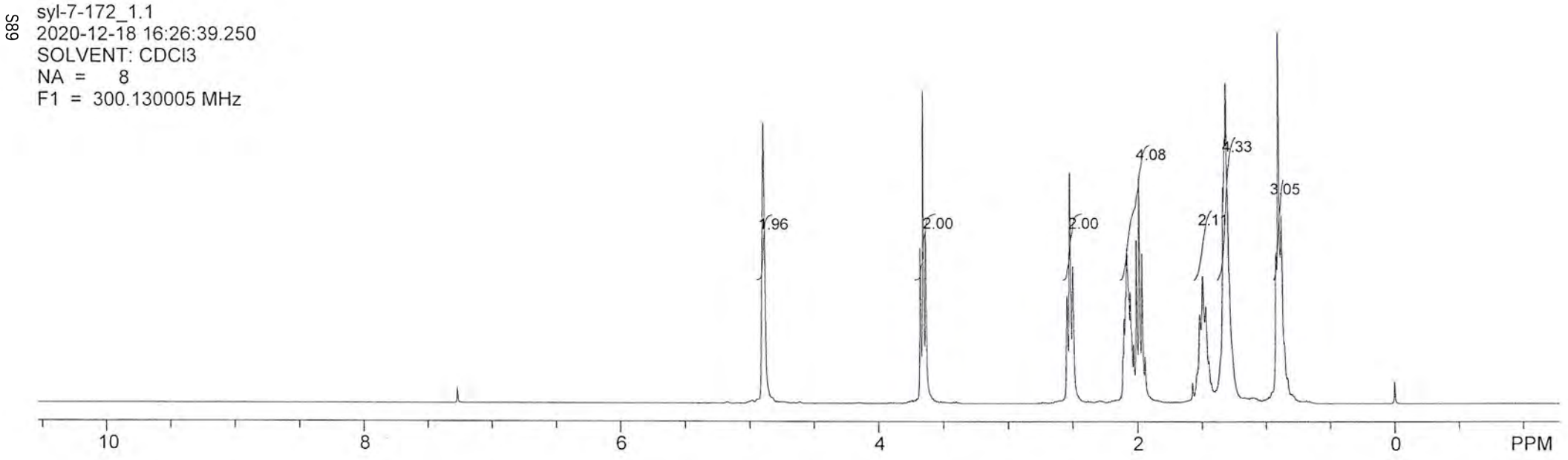
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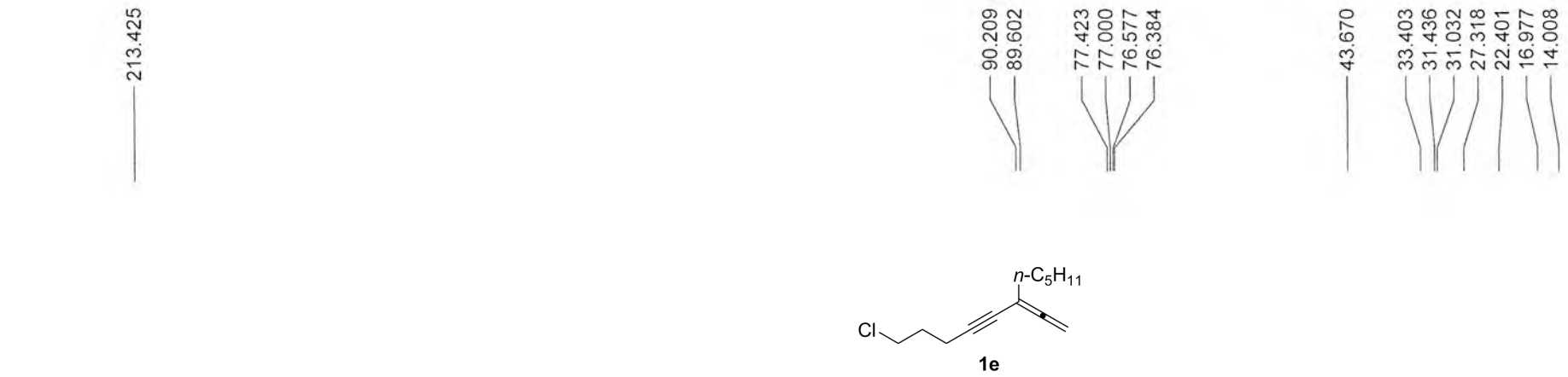
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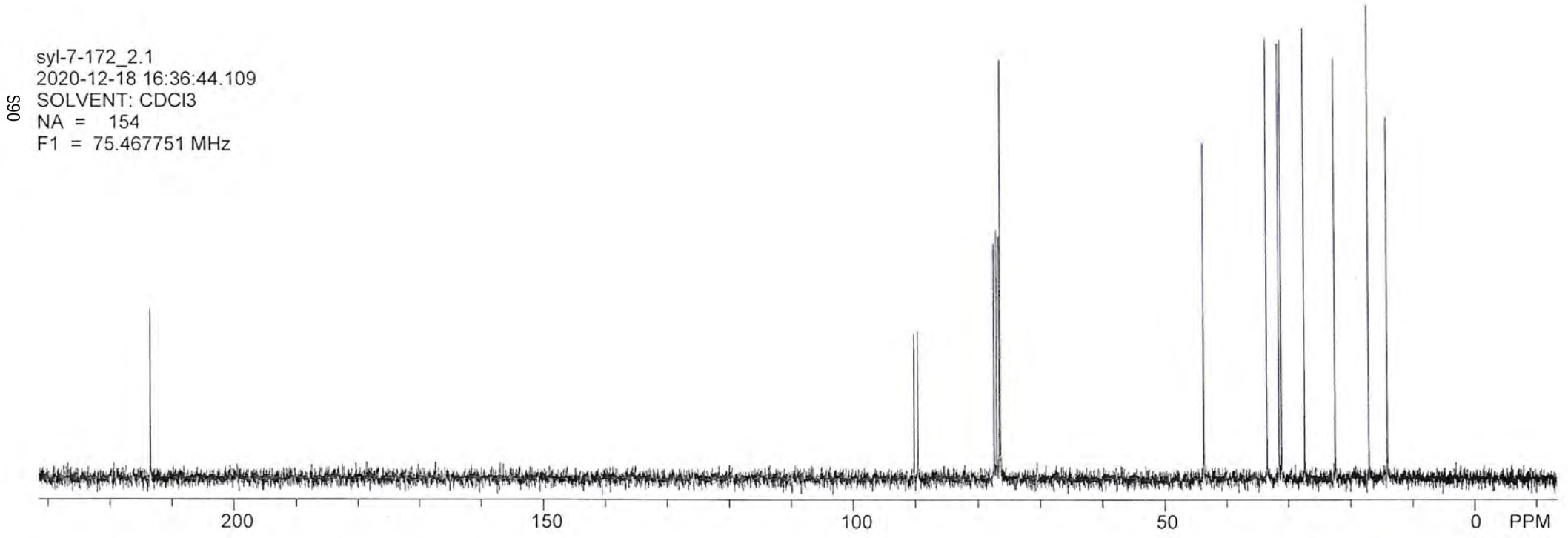
Cl
1e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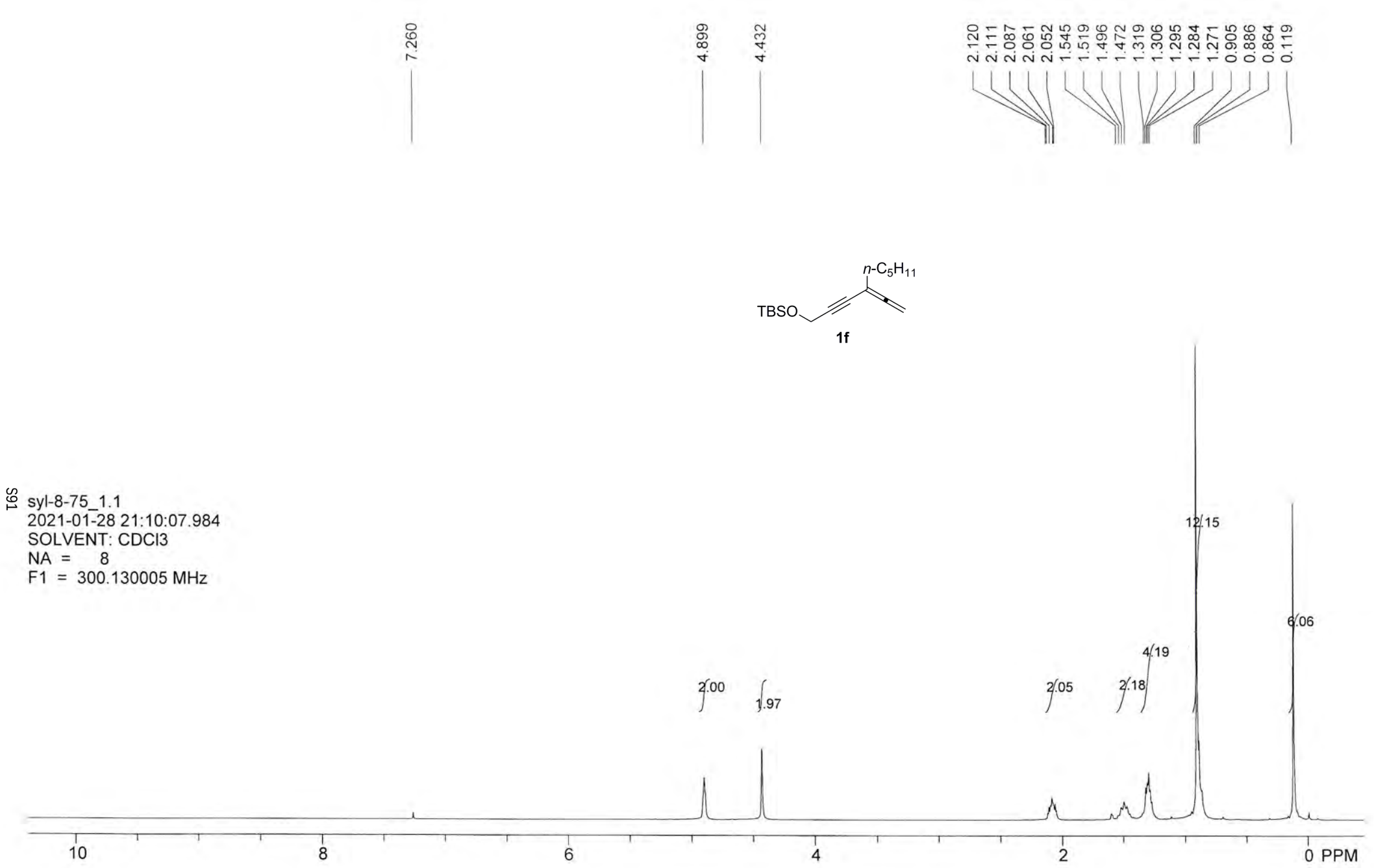
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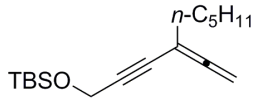

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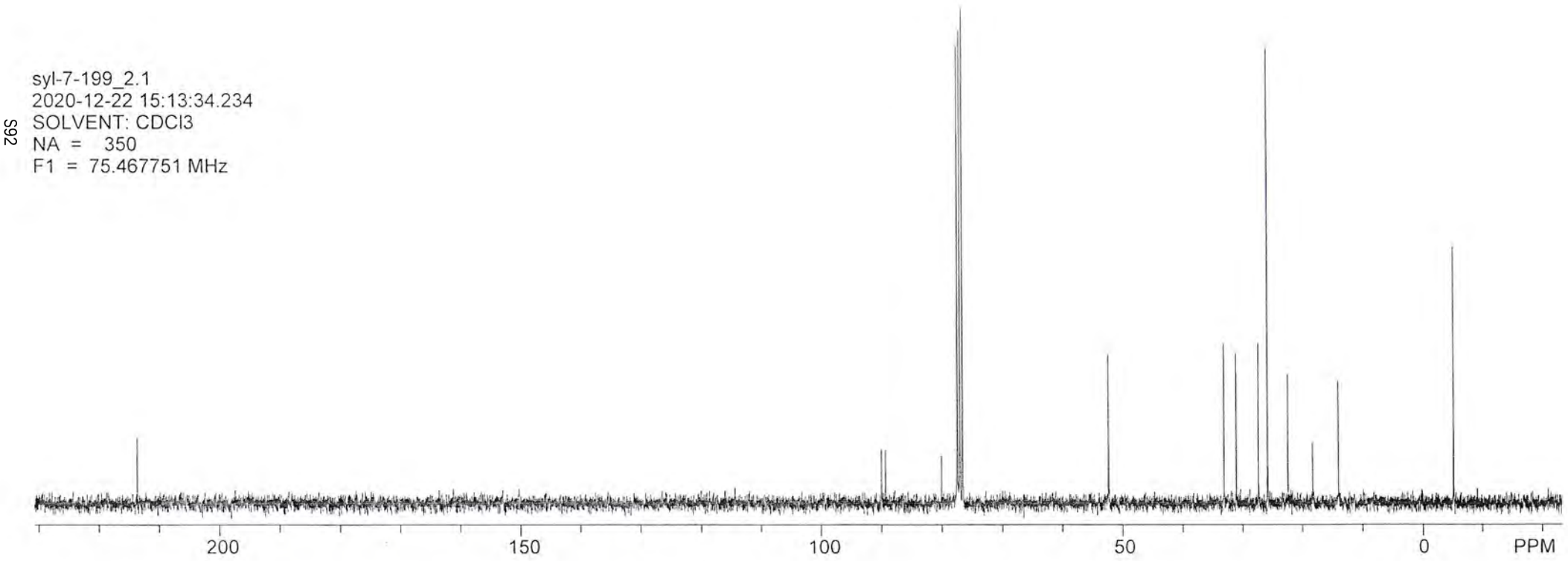

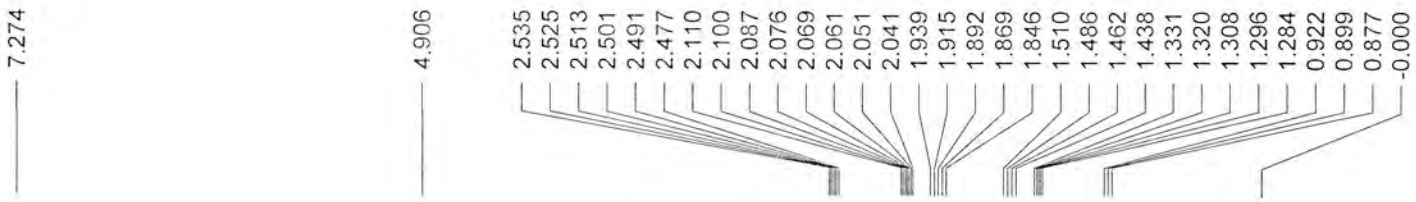
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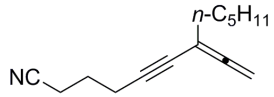
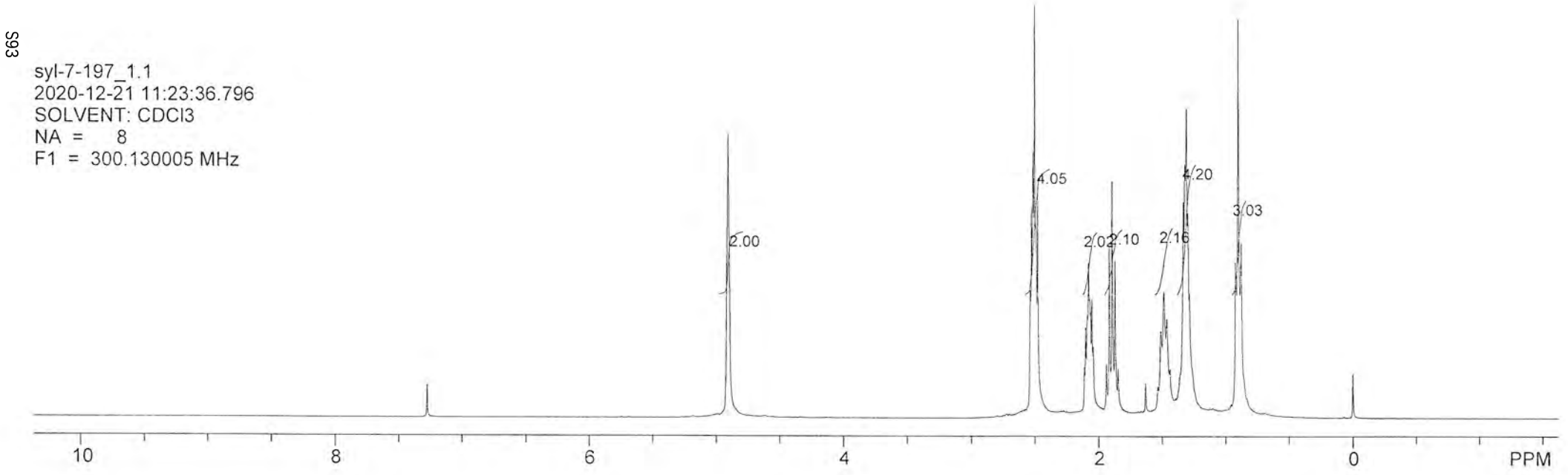
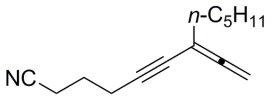
2.513
2.501
2.491
2.477
2.110
2.100
2.087
2.076
2.069
2.061
2.051
2.041
1.939
1.915
1.892
1.869
1.846
1.510
1.486
1.462
1.438
1.331
1.320
1.308
1.296
1.284
0.922
0.899
0.877
-0.000


n-C5H11
NC

1g

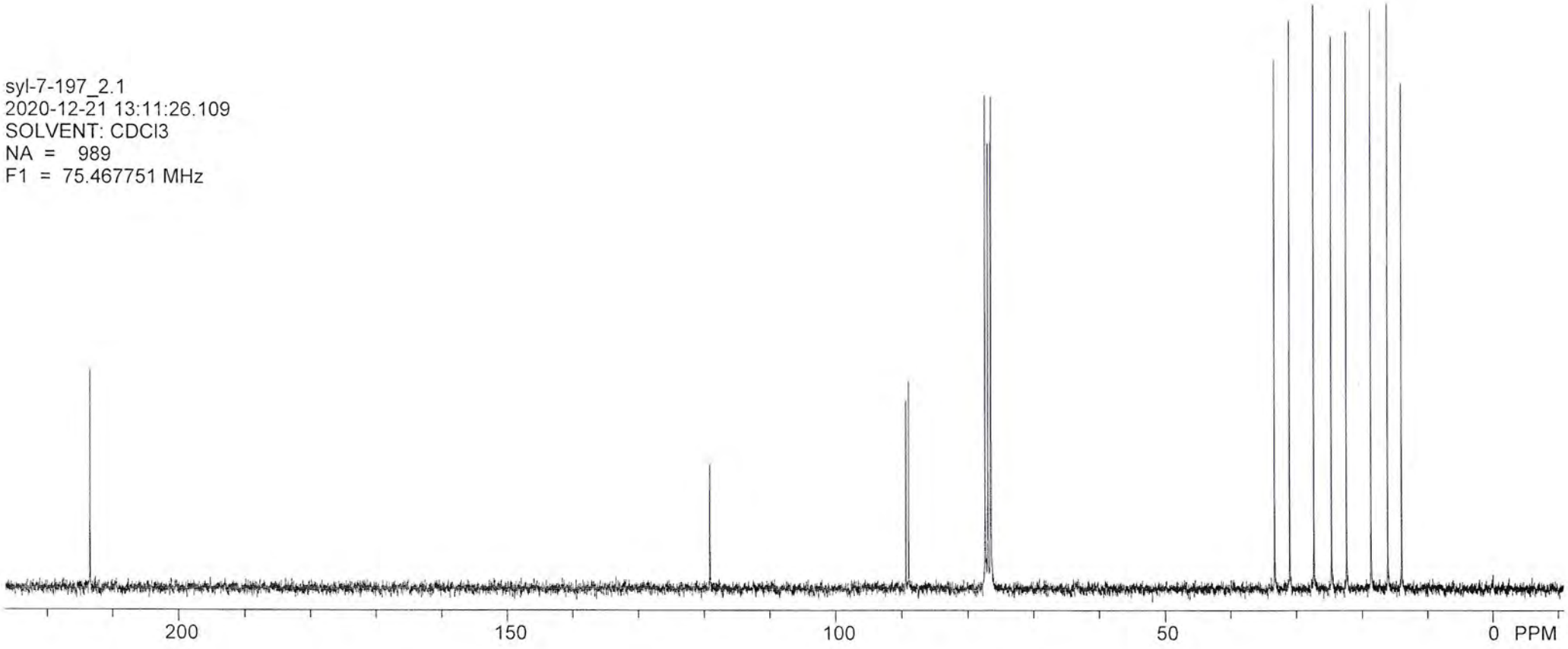

syl-7-197_1.1
2020-12-21 11:23:36.796
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
2.00
4.05
2.02
2.10
2.16
4.20
3.03
10
8
6
4
2
0
PPM

213.489
119.135
89.391
88.968
77.423
77.000
76.577
76.513
33.293
31.004
27.318
24.653
22.364
18.650
16.086
13.972

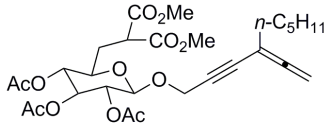


1g

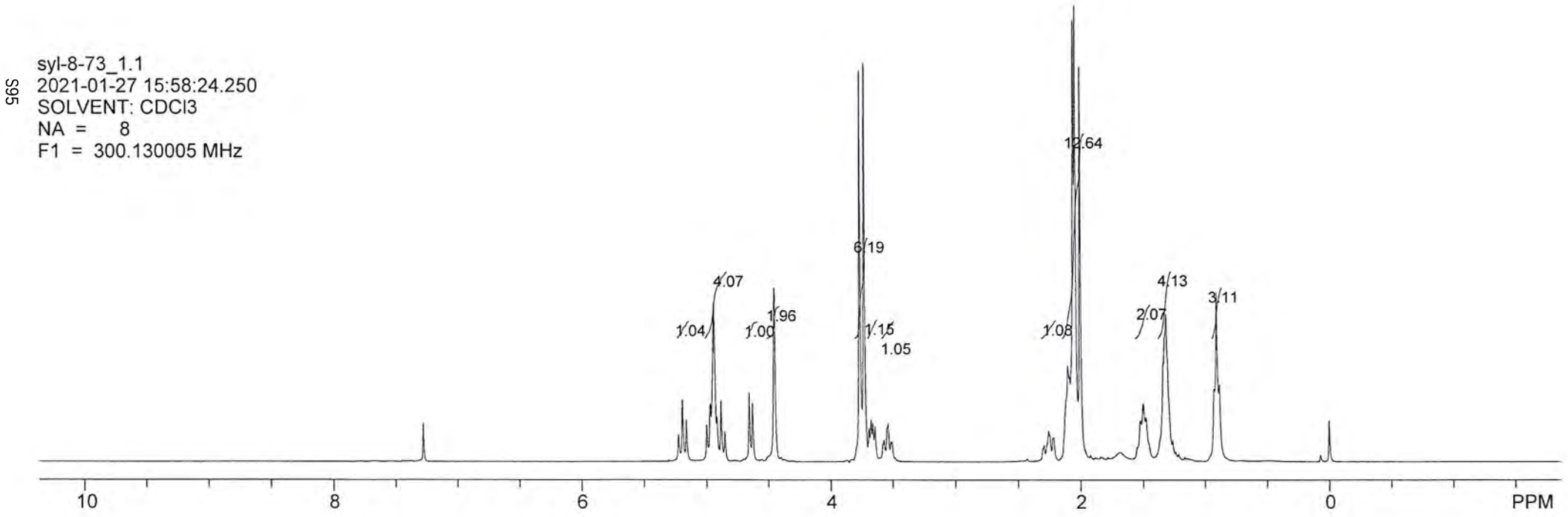
syl-7-197_2.1
2020-12-21 13:11:26.109
SOLVENT: CDCl3
NA = 989
F1 = 75.467751 MHz



5.225 5.193 5.162 4.999 4.972 4.968 4.941 4.916 4.884 4.853 4.656 4.629 4.453 3.770 3.734 3.694 3.676 3.662 3.645 3.573 3.548 3.540 3.516 3.507 2.301 2.292 2.254 2.245 2.221 2.214 2.100 2.086 2.058 2.043 2.003 1.548 1.522 1.499 1.476 1.454 1.338 1.325 1.315 0.926 0.905 0.883 -0.000



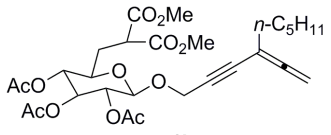
1h



213.682

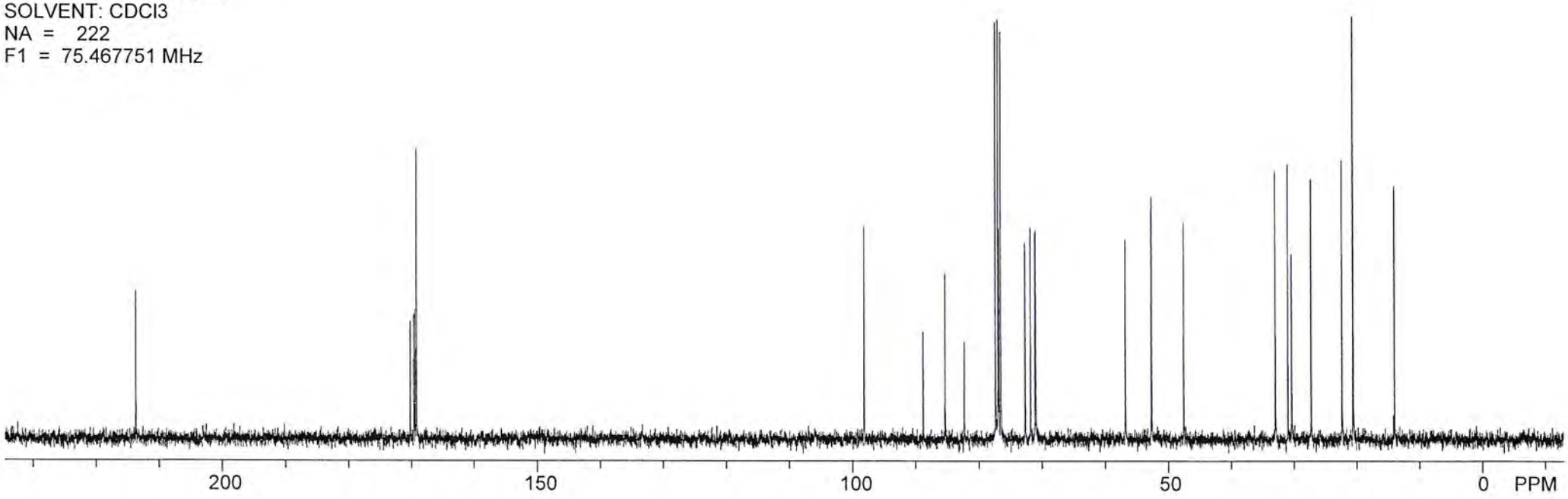
170.168
169.608
169.396
169.176

98.178
88.821
85.374
82.340
77.423
77.000
76.853
76.577
72.698
71.825
71.108
71.016
56.787
52.660
52.623
47.540
32.944
30.940
30.342
27.226
22.309
20.571
20.507
13.944

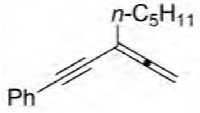


1h

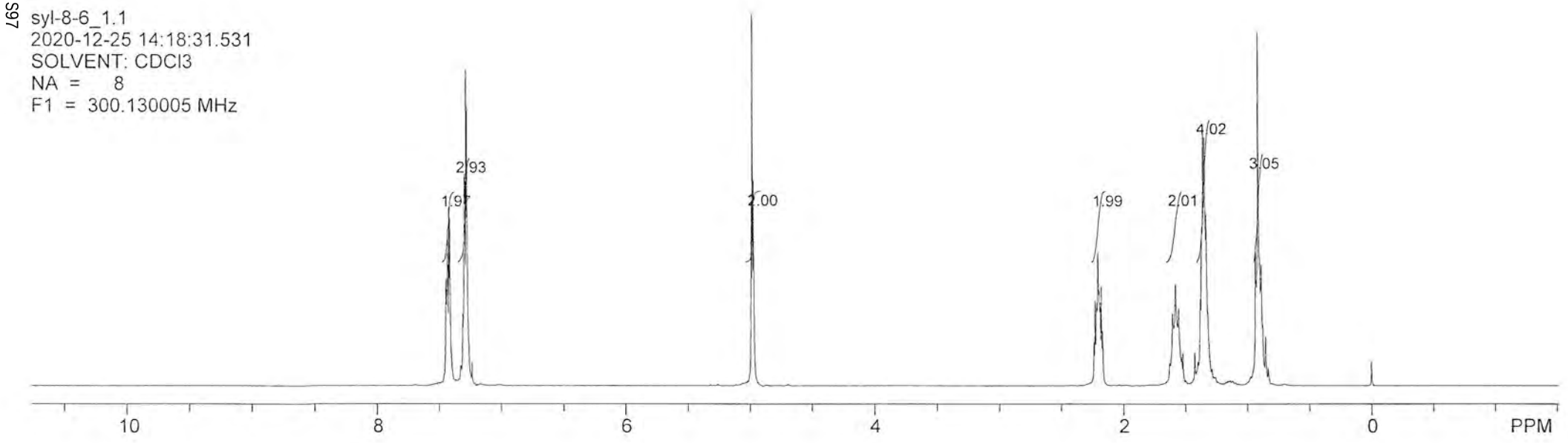
syl-8-73_2.1
2021-01-27 15:15:16.015
SOLVENT: CDCl3
NA = 222
F1 = 75.467751 MHz

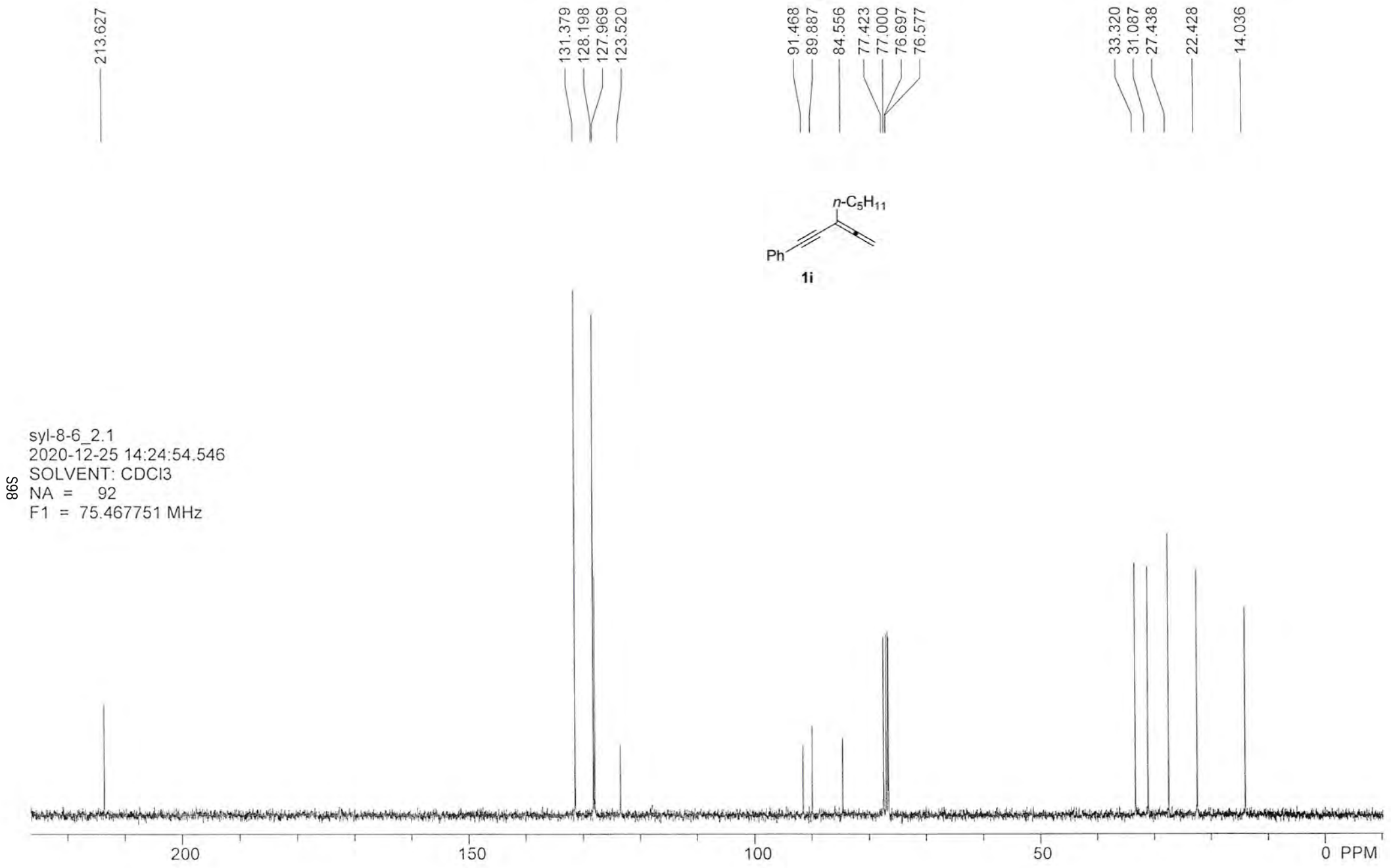


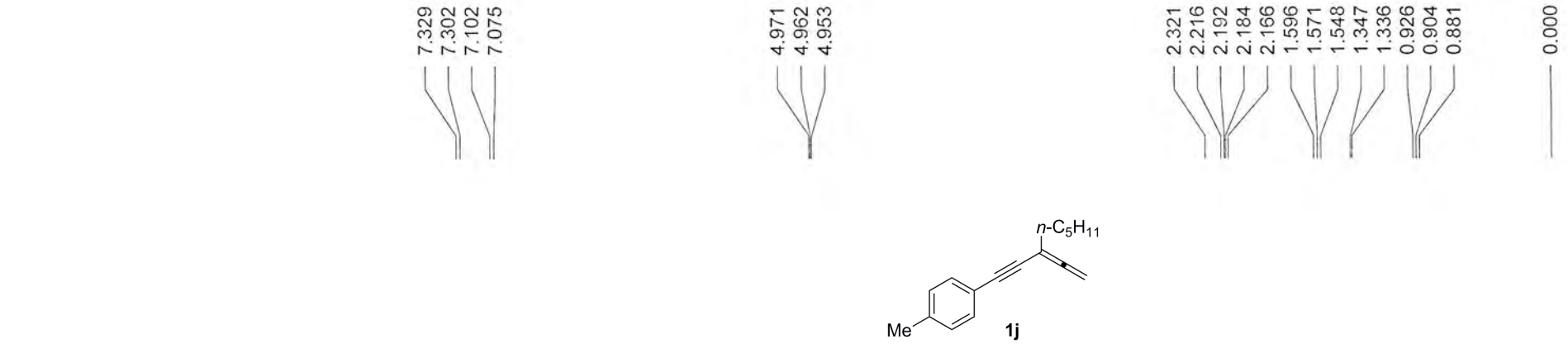

7.444
7.429
7.418
7.411
7.308
7.295
7.290
7.280
7.272
4.988
4.979
4.968
2.235
2.226
2.211
2.202
2.194
2.186
2.176
2.166
1.603
1.594
1.578
1.554
1.377
1.364
1.353
1.341
1.329
1.318
0.931
0.909
0.885
-0.000


n-C5H11
Ph

1i

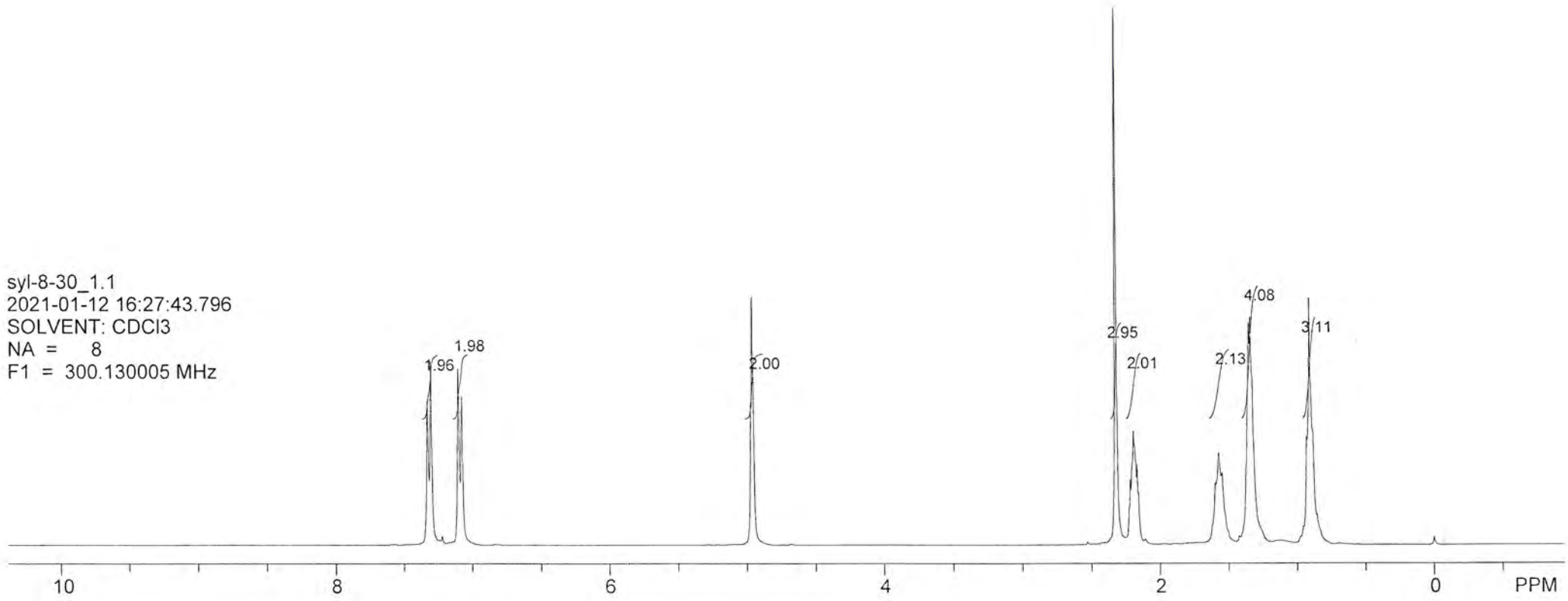

syl-8-6_1.1
2020-12-25 14:18:31.531
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
1.97
2.93
2.00
1.99
2.01
4.02
3.05
10
8
6
4
2
0
PPM

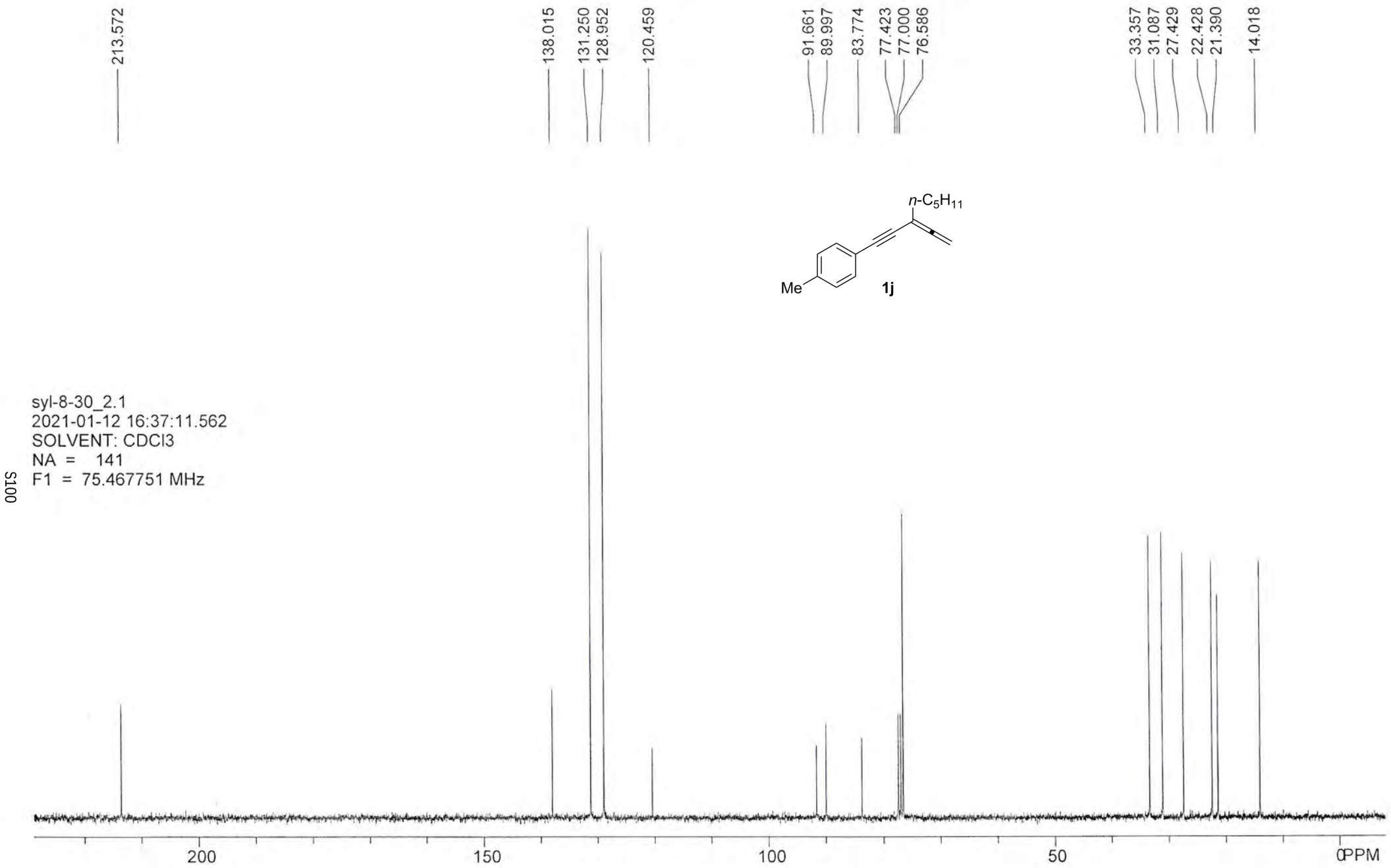

213.627
131.379
128.198
127.969
123.520
91.468
89.887
84.556
77.423
77.000
76.697
76.577
33.320
31.087
27.438
22.428
14.036
n-C5H11
Ph
1i
syl-8-6_2.1
2020-12-25 14:24:54.546
SOLVENT: CDCl3
NA = 92
F1 = 75.467751 MHz
200
150
100
50
0 PPM


7.329
7.302
7.102
7.075
4.971
4.962
4.953
2.321
2.216
2.192
2.184
2.166
1.596
1.571
1.548
1.347
1.336
0.926
0.904
0.881
0.000

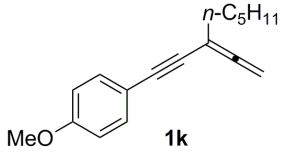
n-C5H11
Me

1j

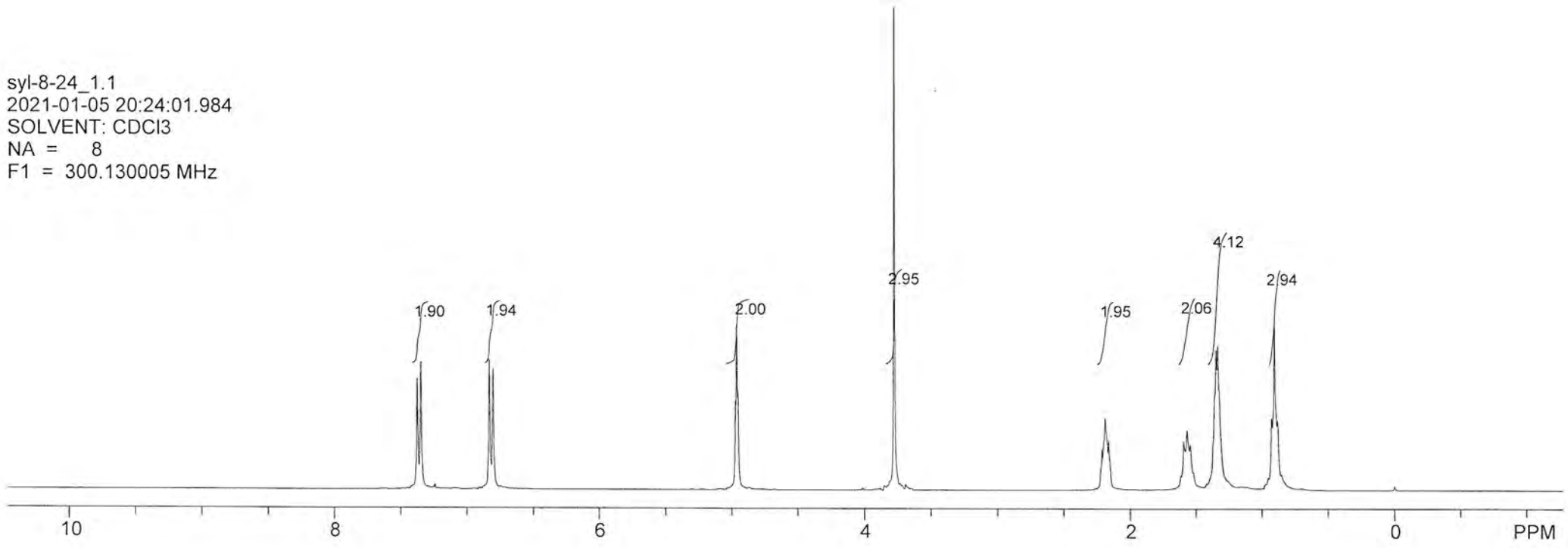

syl-8-30_1.1
2021-01-12 16:27:43.796
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
1.96
1.98
2.00
2.95
2.01
2.13
4.08
3.11
10
8
6
4
2
0
PPM


213.572
138.015
131.250
128.952
120.459
91.661
89.997
83.774
77.423
77.000
76.586
33.357
31.087
27.429
22.428
21.390
14.018
n-C5H11
Me
1j
syl-8-30_2.1
2021-01-12 16:37:11.562
SOLVENT: CDCl3
NA = 141
F1 = 75.467751 MHz
200
150
100
50
0PPM

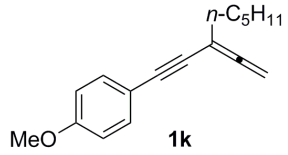

7.376
7.347
7.241
6.831
6.801
4.973
4.963
4.955
3.777
2.198
2.188
2.163
1.595
1.569
1.545
1.521
1.348
1.336
1.325
0.927
0.905
0.882
-0.000


n-C5H11
MeO

1k

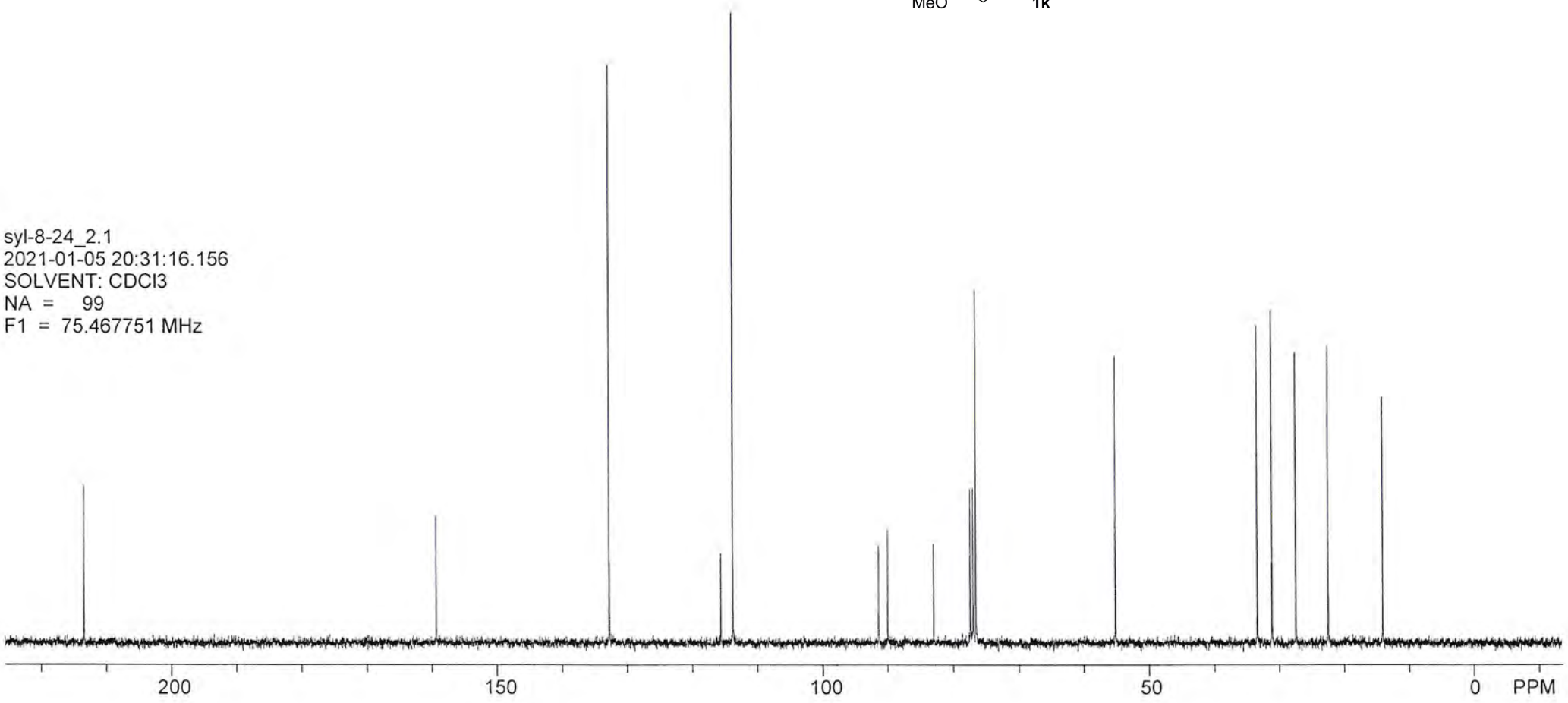

syl-8-24_1.1
2021-01-05 20:24:01.984
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
1.90
1.94
2.00
2.95
1.95
2.06
4.12
2.94
10
8
6
4
2
0
PPM

213.462
159.359
132.767
115.615
113.795
91.413
90.006
82.993
77.423
77.000
76.568
55.142
33.357
31.068
27.410
22.410
14.008

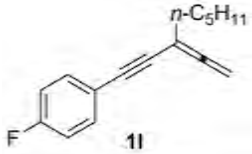


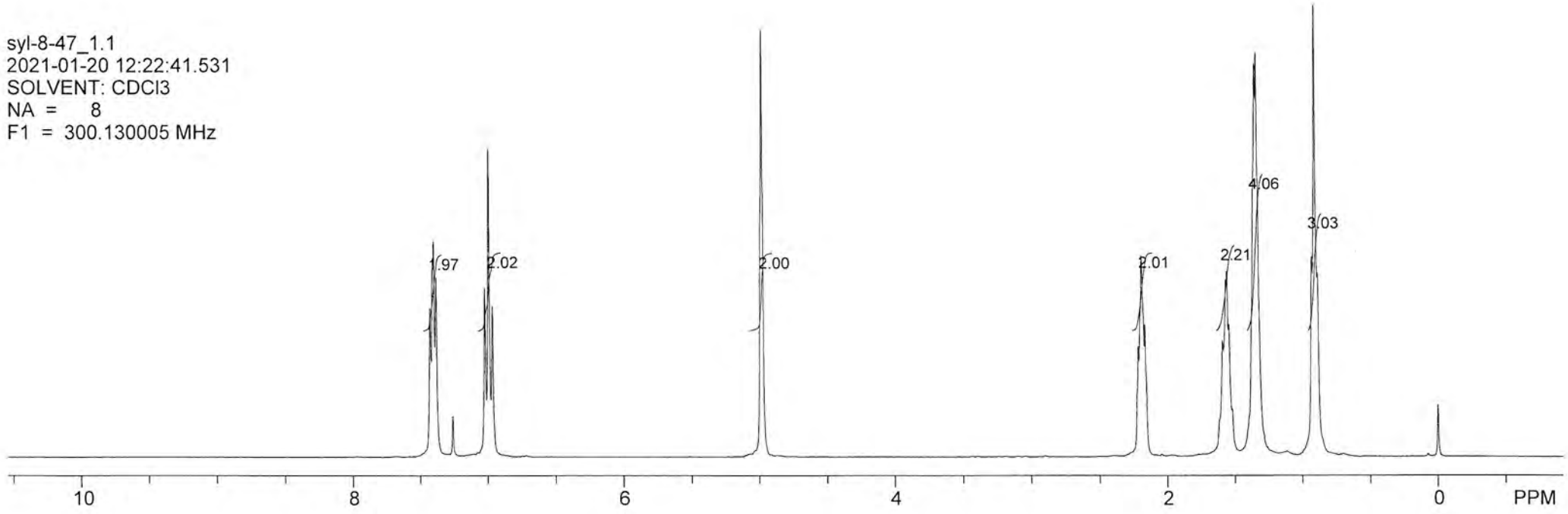
1k

syl-8-24_2.1
2021-01-05 20:31:16.156
SOLVENT: CDCl3
NA = 99
F1 = 75.467751 MHz

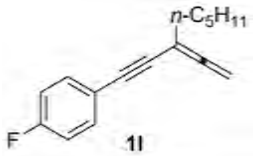



7.428
7.407
7.400
7.381
7.258
7.022
6.993
6.964
4.984
2.215
2.191
2.165
1.590
1.566
1.558
1.543
1.520
1.351
1.339
0.929
0.908
0.885
-0.000

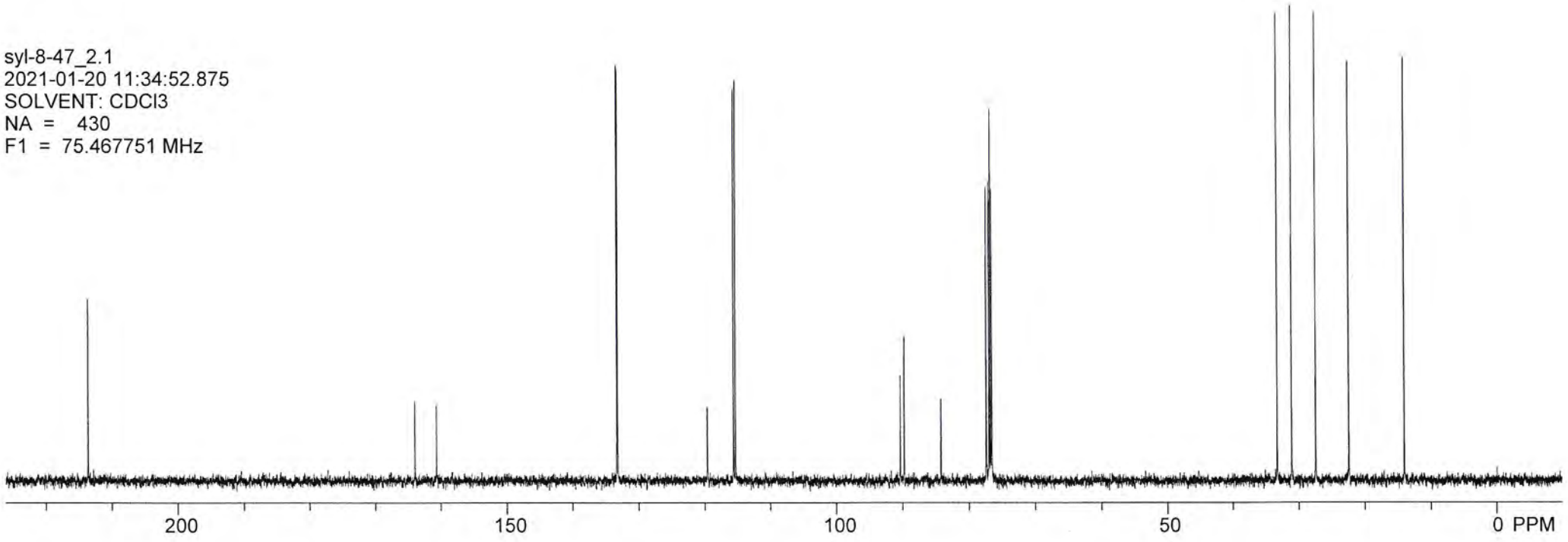

n-C5H11
F
1l


syl-8-47_1.1
2021-01-20 12:22:41.531
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
1.97
2.02
2.00
2.01
2.21
4.06
3.03
10
8
6
4
2
0
PPM

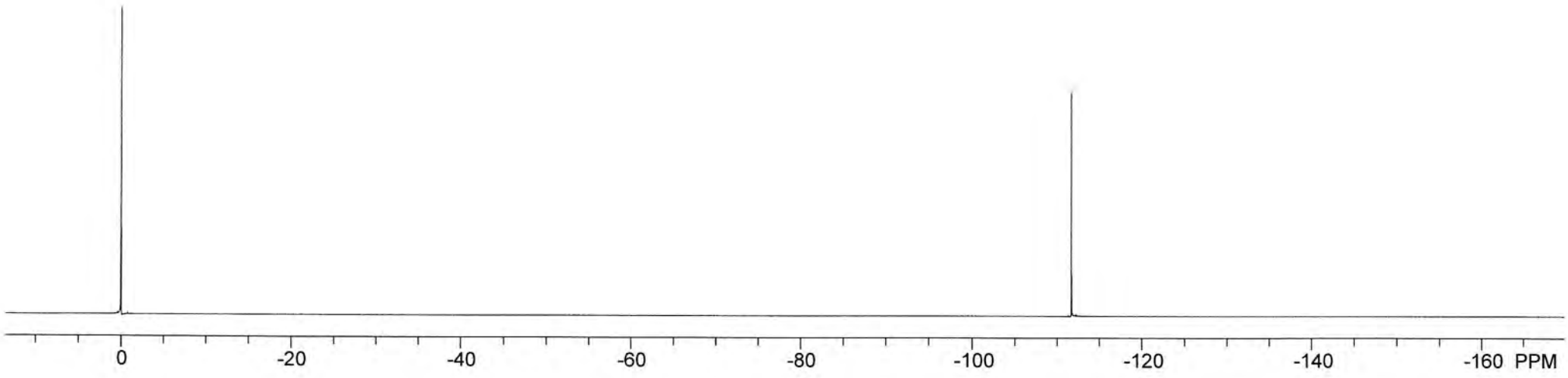
213.609
163.973
160.673
133.281
133.171
119.641
119.595
115.624
115.339
90.337
89.749
84.234
77.423
77.000
76.779
76.577
33.256
31.078
27.438
22.428
14.027



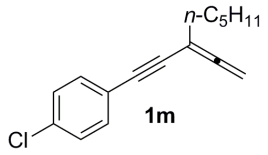
1l

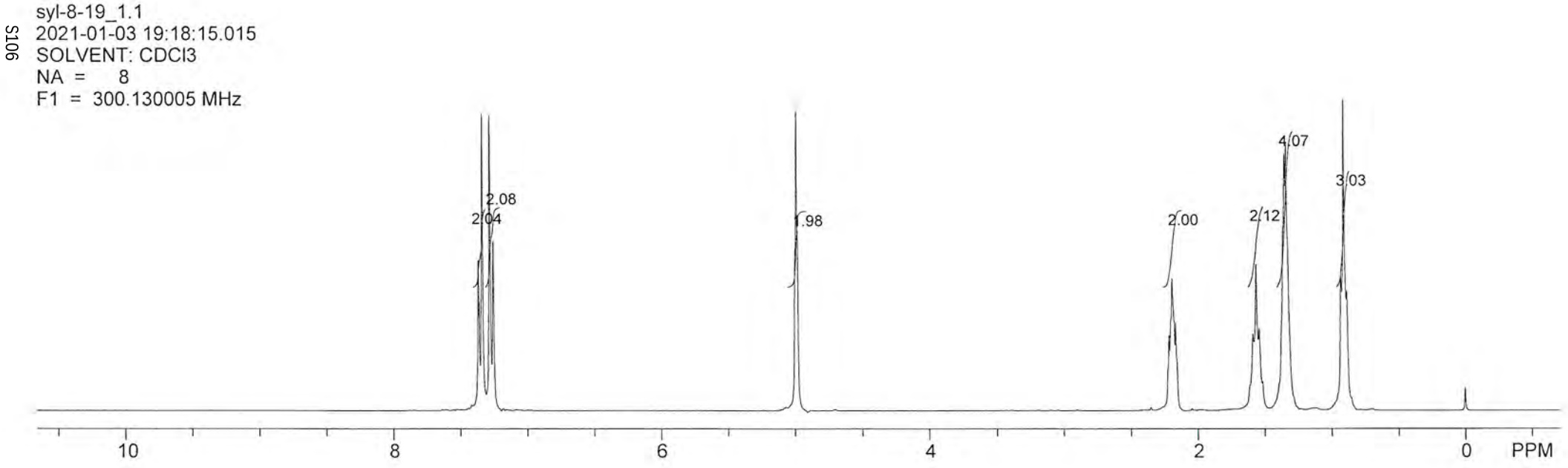


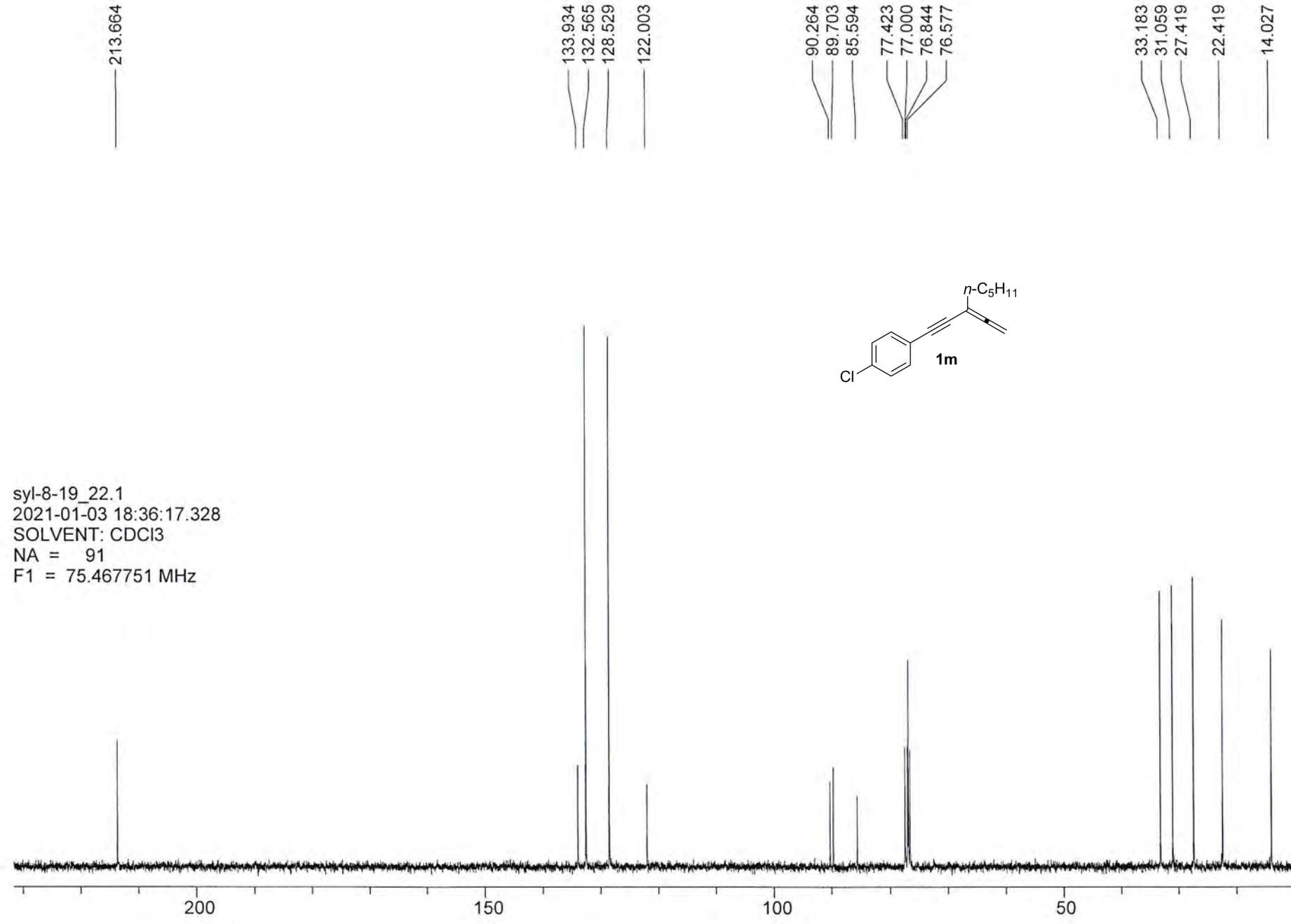
syl-8-47_4.1
2021-01-20 12:39:35.779
SOLVENT: CDCl3
NA = 16
F1 = 282.404358 MHz



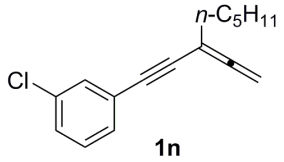

7.365
7.338
7.282
7.253
4.999
4.990
4.980
2.223
2.214
2.200
2.190
2.165
1.608
1.586
1.561
1.538
1.515
1.348
1.337
0.929
0.907
0.883
0.000


n-C5H11
Cl
1m

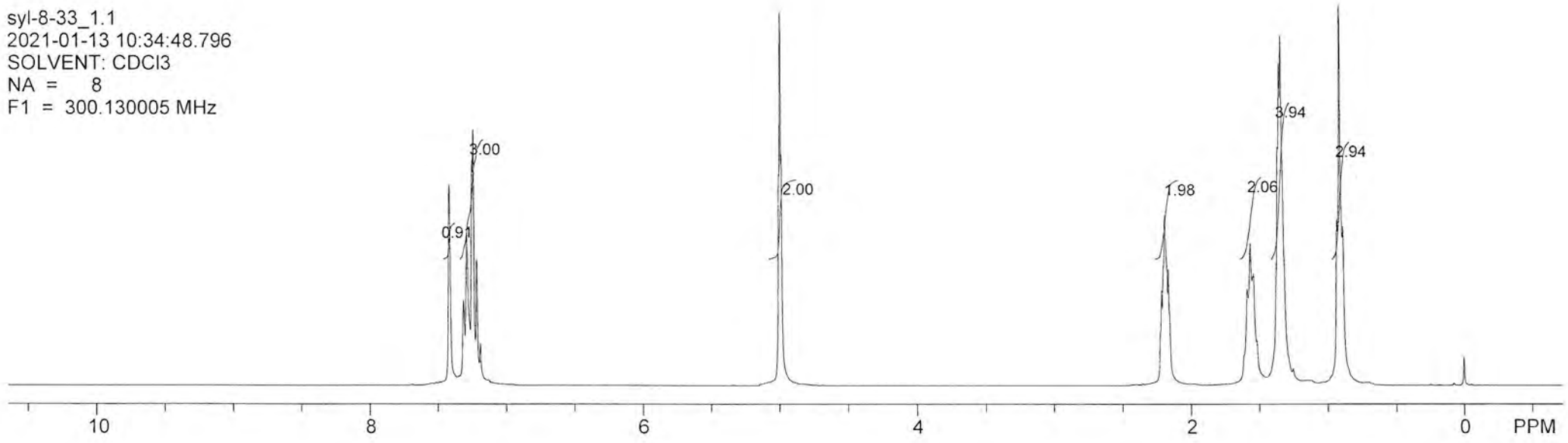

syl-8-19_1.1
2021-01-03 19:18:15.015
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
2.08
2.04
1.98
2.00
2.12
4.07
3.03
10
8
6
4
2
0
PPM


213.664
133.934
132.565
128.529
122.003
90.264
89.703
85.594
77.423
77.000
76.844
76.577
33.183
31.059
27.419
22.419
14.027
n-C5H11
Cl
1m
syl-8-19_22.1
2021-01-03 18:36:17.328
SOLVENT: CDCl3
NA = 91
F1 = 75.467751 MHz
200
150
100
50
0
PPM


7.415
7.312
7.288
7.248
7.239
7.215
7.188
5.004
4.995
4.987
2.214
2.190
2.165
2.156
1.587
1.563
1.547
1.540
1.516
1.360
1.350
1.338
0.931
0.909
0.887
-0.000


n-C5H11
Cl

1n


syl-8-33_1.1
2021-01-13 10:34:48.796
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
0.91
3.00
2.00
1.98
2.06
3.94
2.94
10
8
6
4
2
0
PPM

213.765

134.044
131.223
129.467
129.430
128.208
125.229

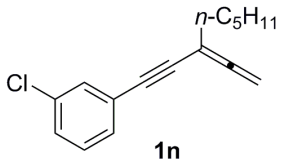
89.951
89.602
85.934

77.423
77.000
76.899
76.577

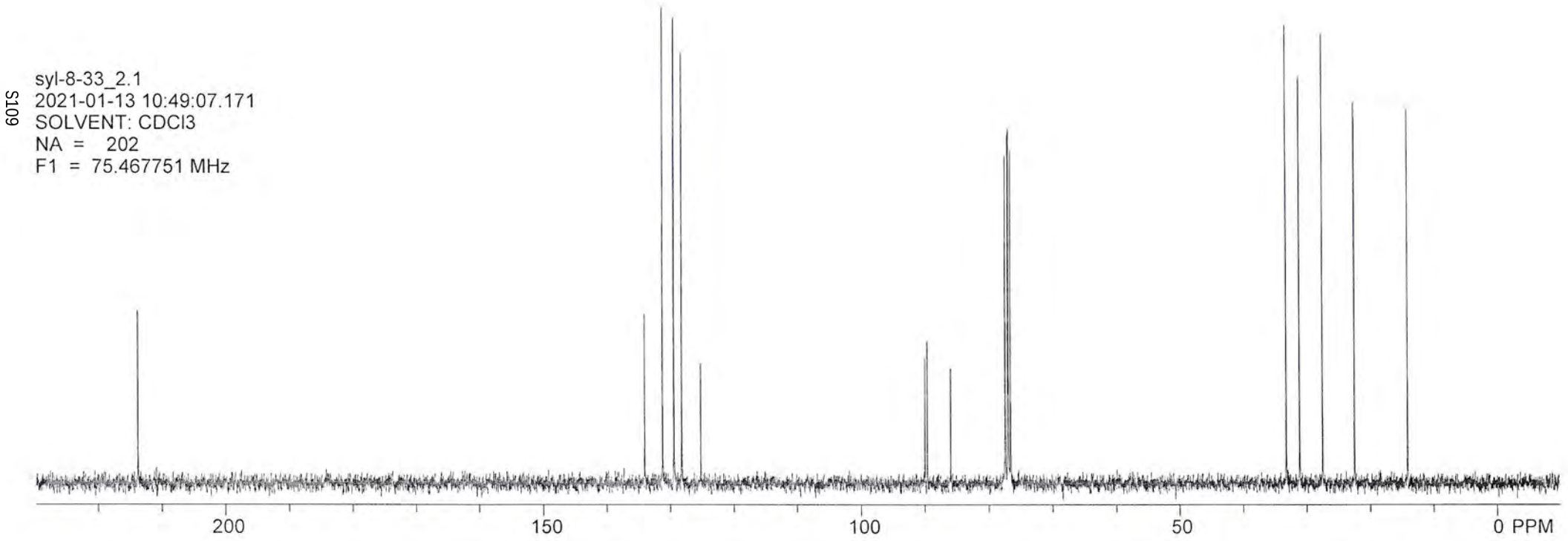
33.164
31.068
27.438

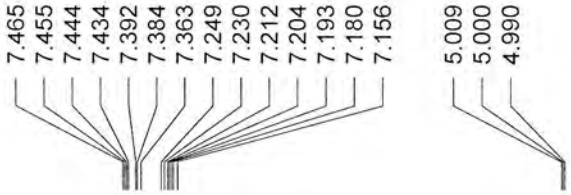
22.428

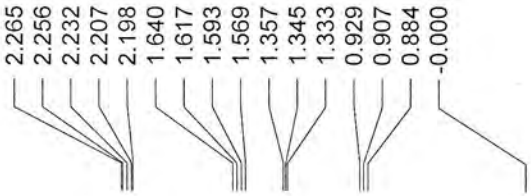
14.045

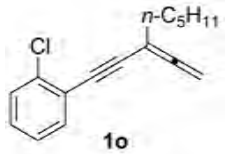


1n

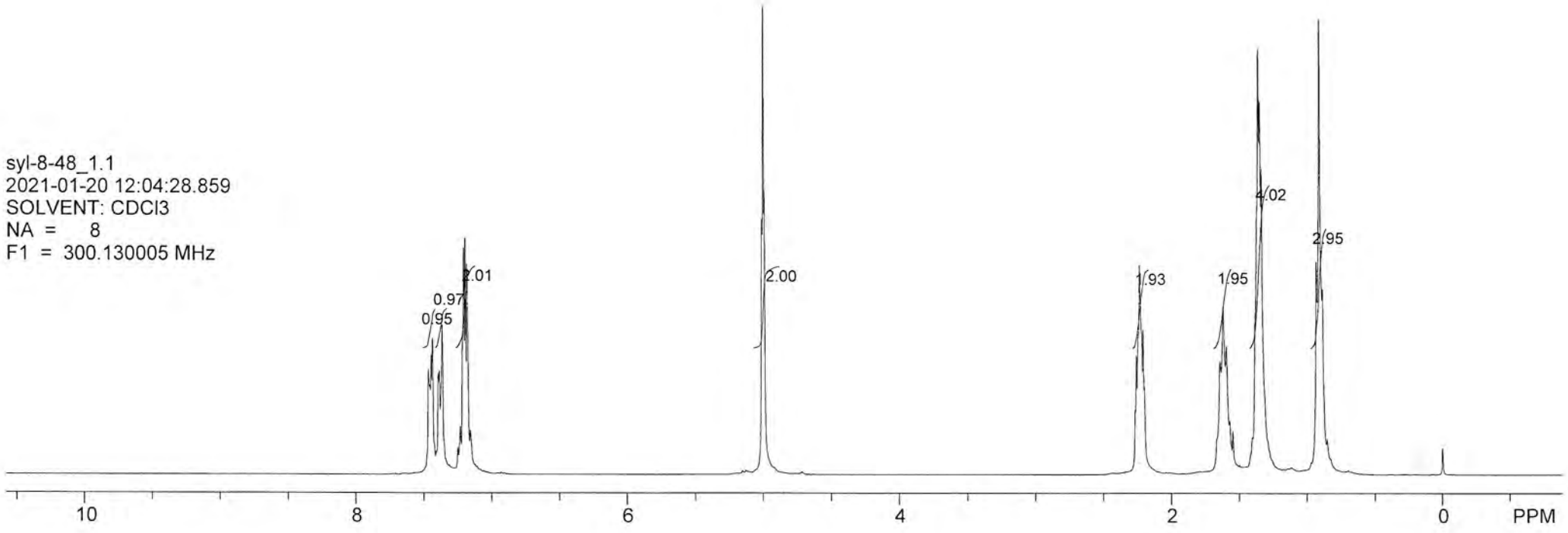


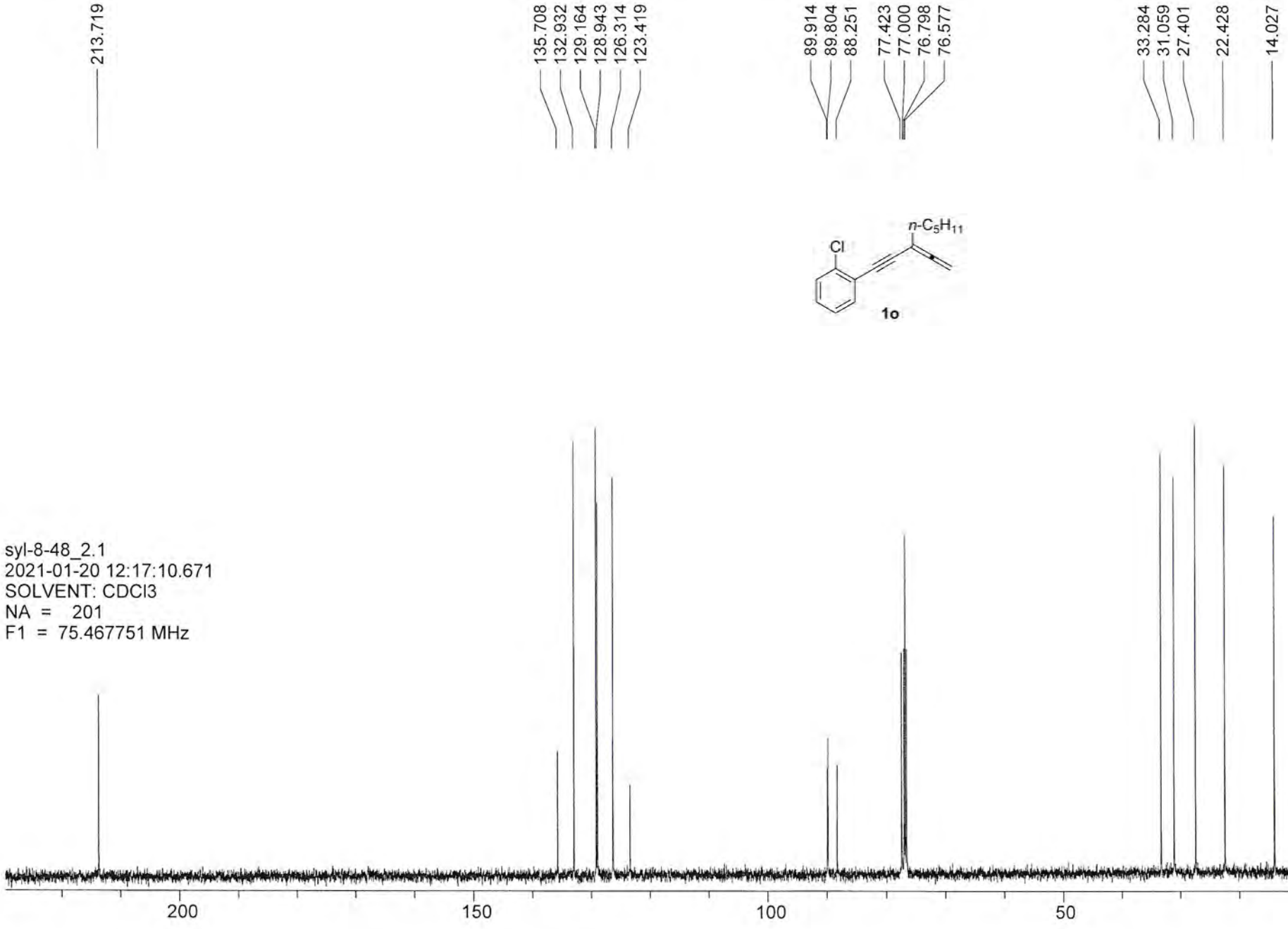

7.465
7.455
7.444
7.434
7.392
7.384
7.363
7.249
7.230
7.212
7.204
7.193
7.180
7.156
5.009
5.000
4.990


2.265
2.256
2.232
2.207
2.198
1.640
1.617
1.593
1.569
1.357
1.345
1.333
0.929
0.907
0.884
-0.000

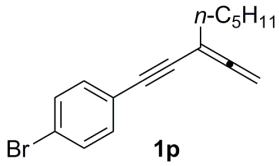

n-C5H11
Cl

1o

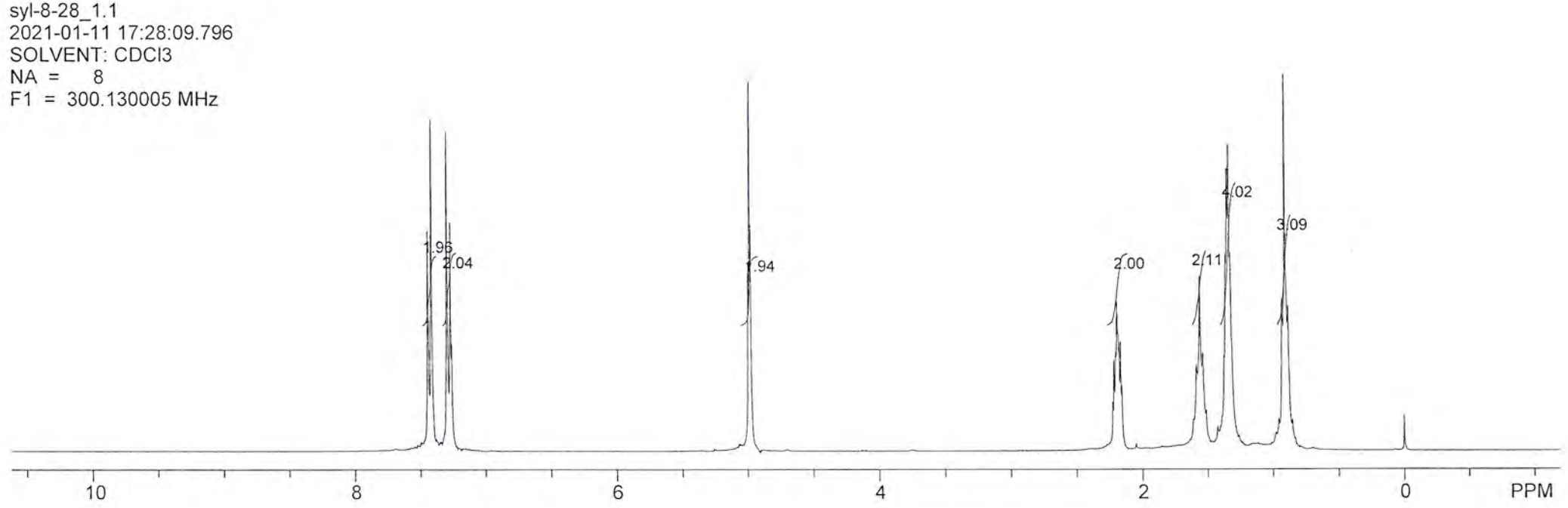

syl-8-48_1.1
2021-01-20 12:04:28.859
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
0.95
0.97
2.01
2.00
1.93
1.95
4.02
2.95
10
8
6
4
2
0
PPM

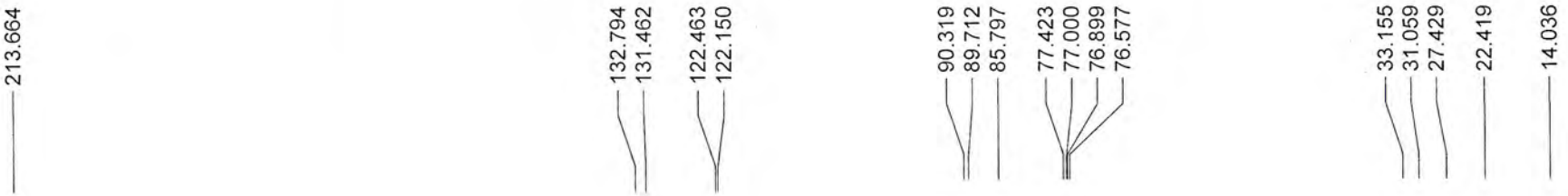

213.719
135.708
132.932
129.164
128.943
126.314
123.419
89.914
89.804
88.251
77.423
77.000
76.798
76.577
33.284
31.059
27.401
22.428
14.027
n-C5H11
Cl
1o
syl-8-48_2.1
2021-01-20 12:17:10.671
SOLVENT: CDCl3
NA = 201
F1 = 75.467751 MHz
200
150
100
50
0
PPM

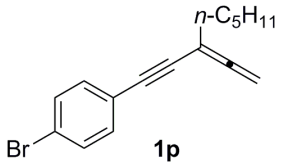

7.445
7.439
7.422
7.416
7.299
7.270
7.260
4.997
4.988
4.979
2.224
2.215
2.200
2.191
2.183
2.175
2.165
2.156
1.586
1.577
1.559
1.537
1.513
1.372
1.360
1.348
1.336
1.325
0.929
0.906
0.883
-0.000


n-C5H11
Br

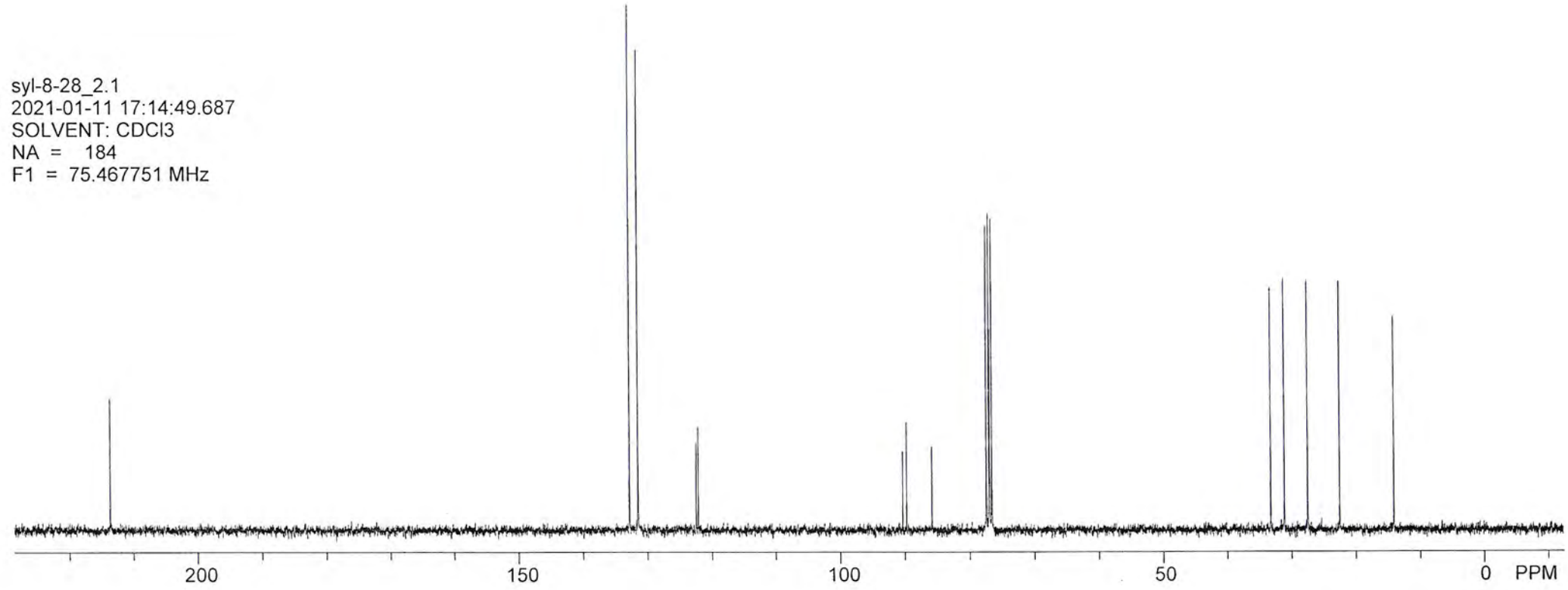
1p


syl-8-28_1.1
2021-01-11 17:28:09.796
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
1.96
2.04
1.94
2.00
2.11
4.02
3.09
10
8
6
4
2
0
PPM

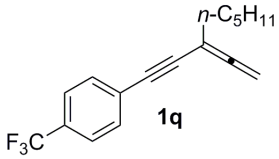

213.664
132.794
131.462
122.463
122.150
90.319
89.712
85.797
77.423
77.000
76.899
76.577
33.155
31.059
27.429
22.419
14.036


n-C5H11
Br

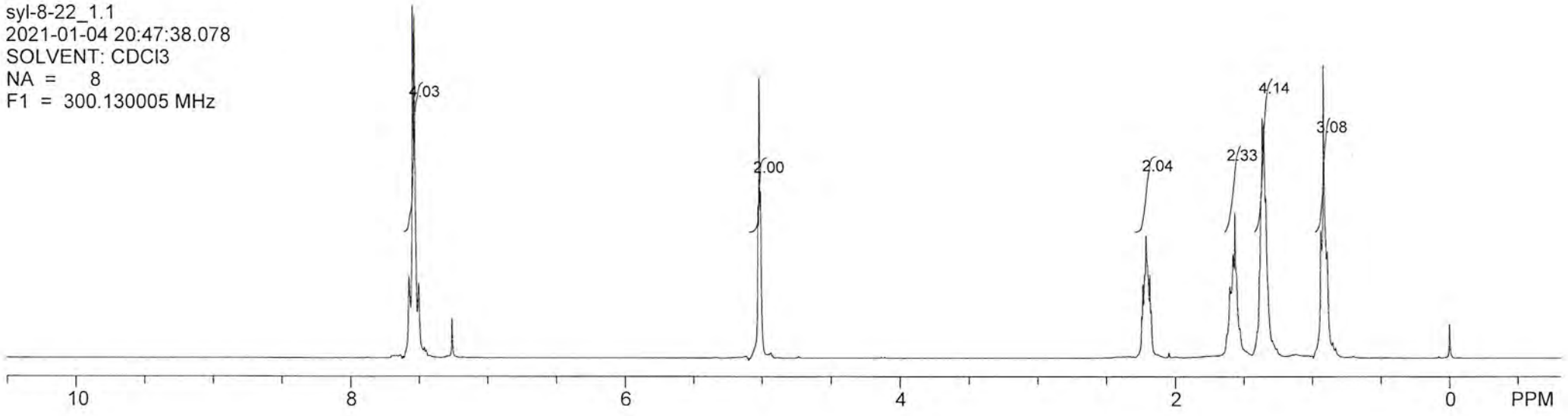
1p

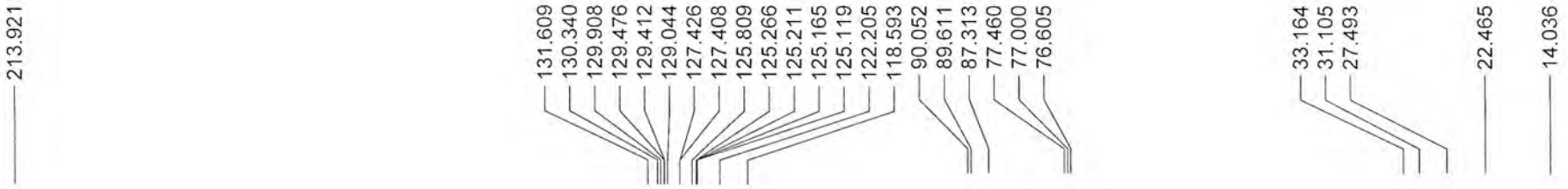

syl-8-28_2.1
2021-01-11 17:14:49.687
SOLVENT: CDCl3
NA = 184
F1 = 75.467751 MHz
200
150
100
50
0
PPM

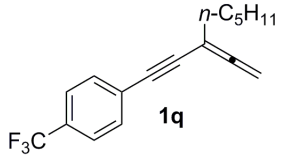

7.573
7.545
7.531
7.502
7.259
5.026
5.016
5.007
2.243
2.234
2.219
2.210
2.184
2.175
1.600
1.575
1.561
1.358
1.346
1.335
0.935
0.912
0.889
-0.000

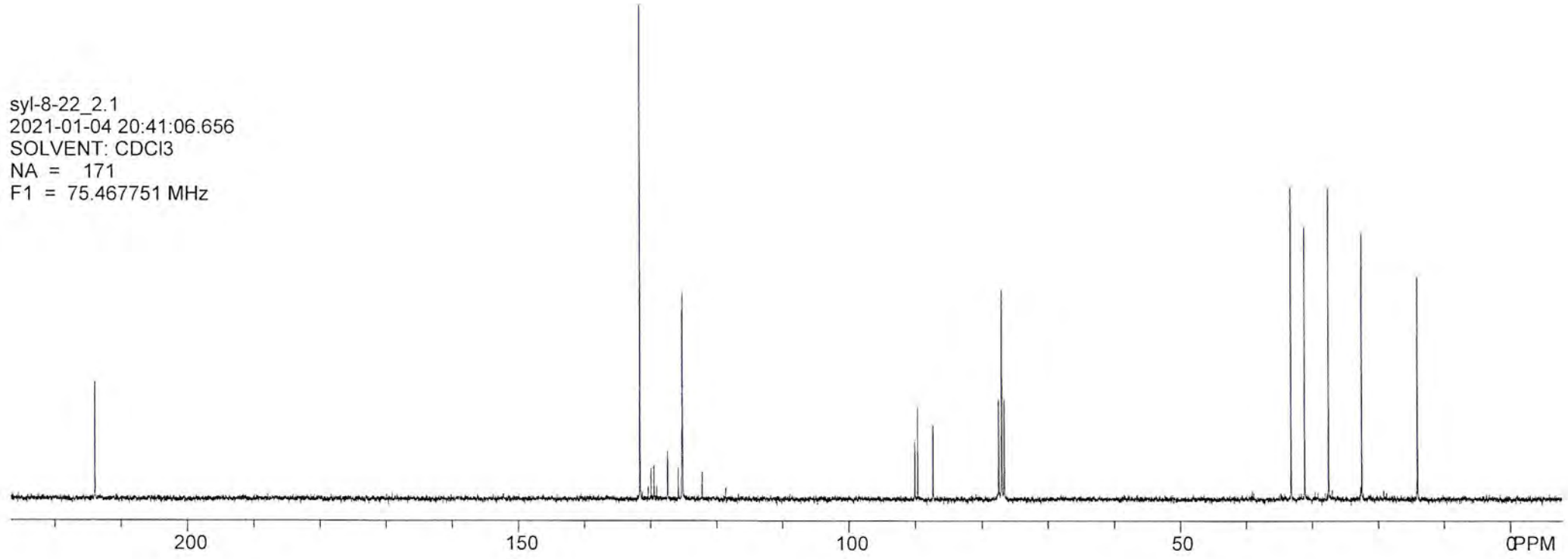

n-C5H11
F3C

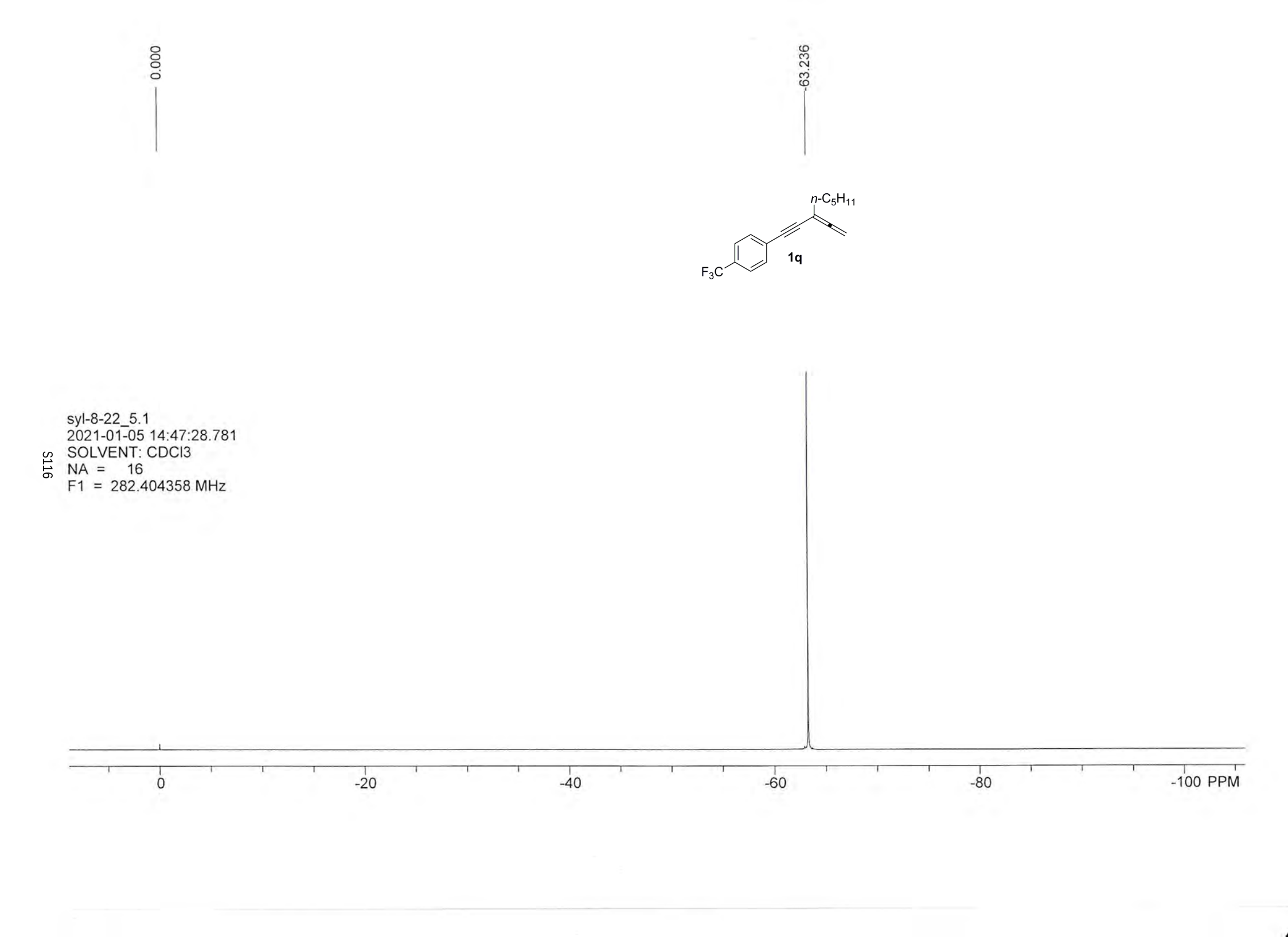
1q

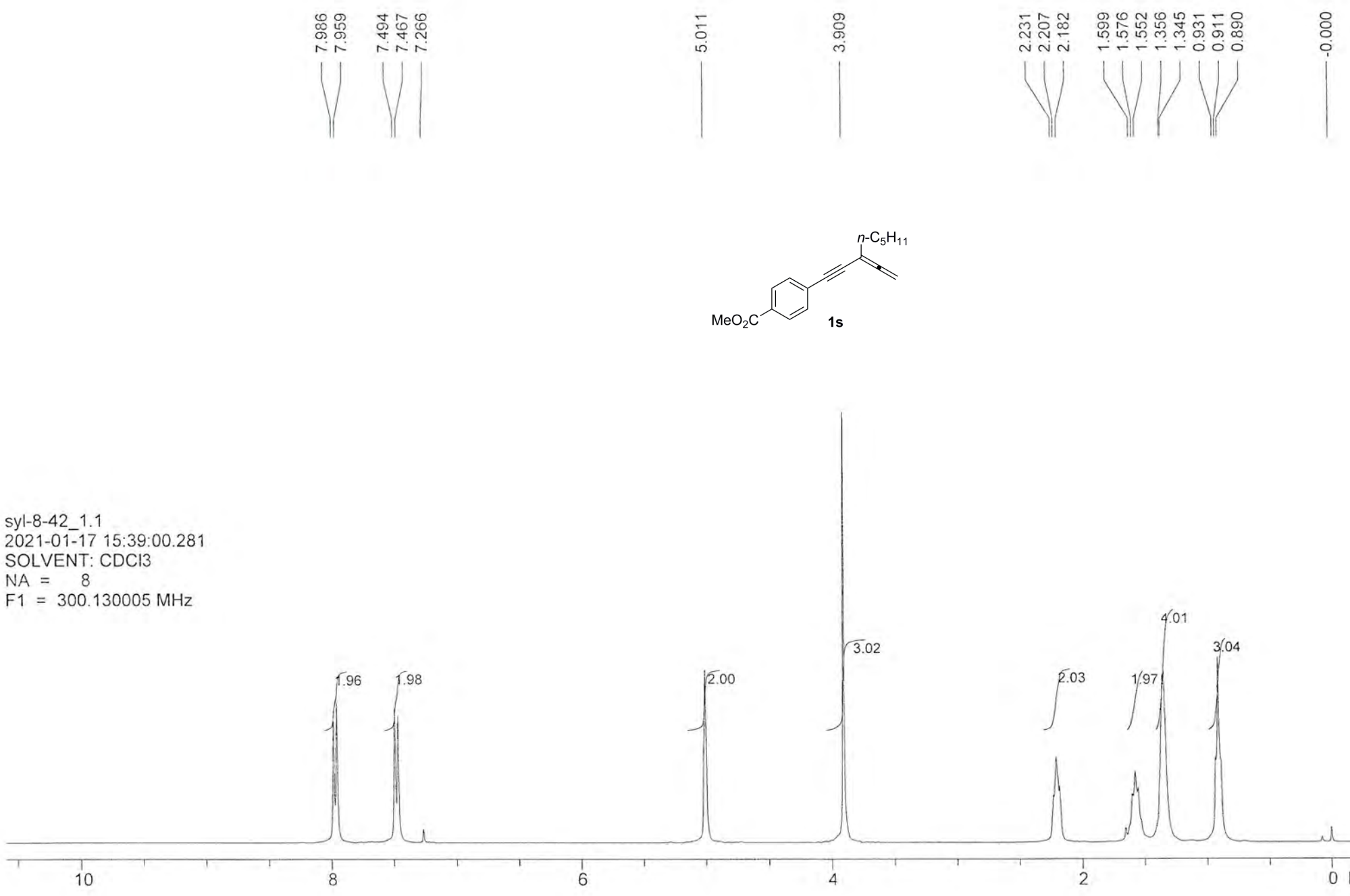

syl-8-22_1.1
2021-01-04 20:47:38.078
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
4.03
2.00
2.04
2.33
4.14
3.08
10
8
6
4
2
0
PPM

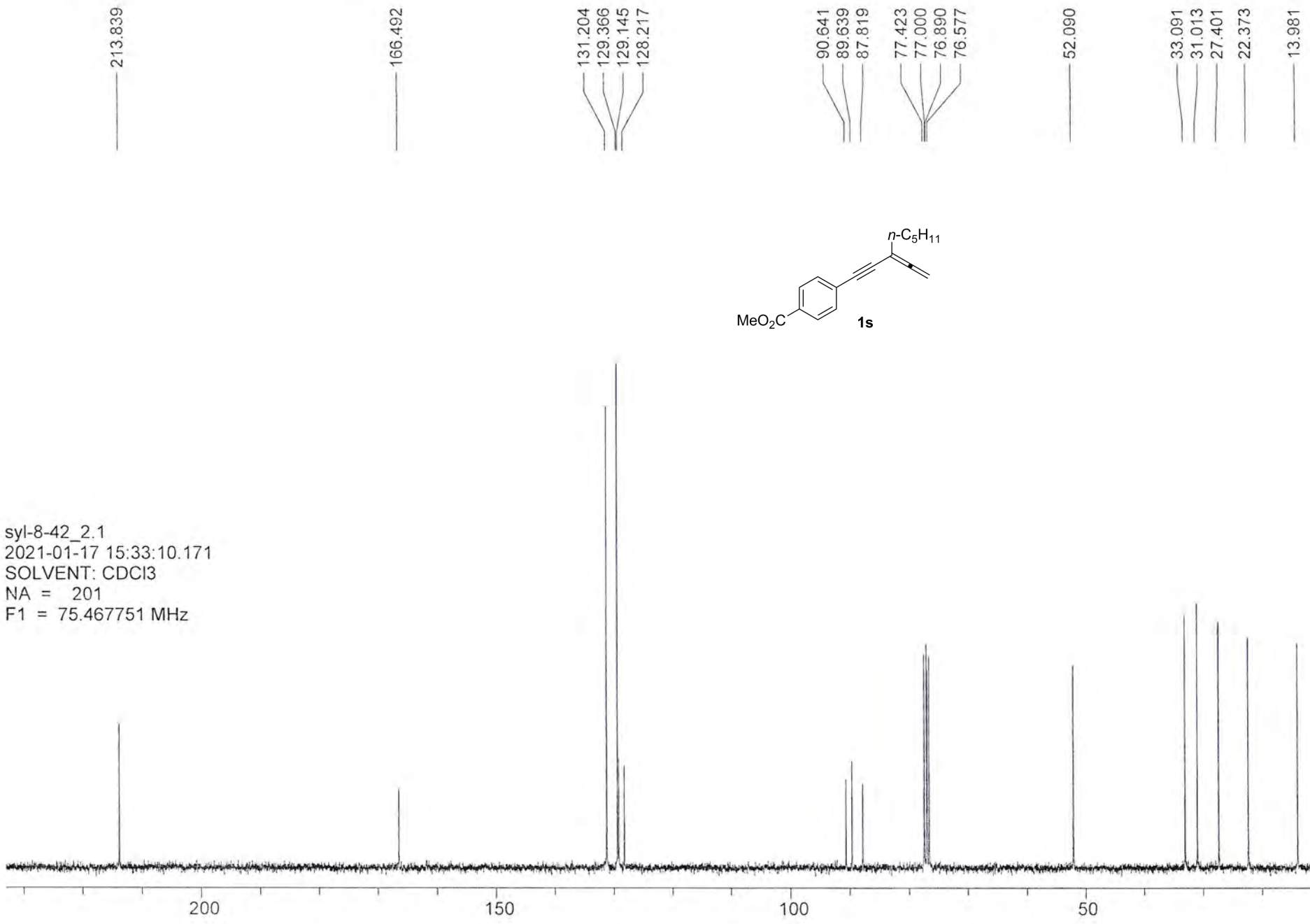

213.921
131.609
130.340
129.908
129.476
129.412
129.044
127.426
127.408
125.809
125.266
125.211
125.165
125.119
122.205
118.593
90.052
89.611
87.313
77.460
77.000
76.605
33.164
31.105
27.493
22.465
14.036

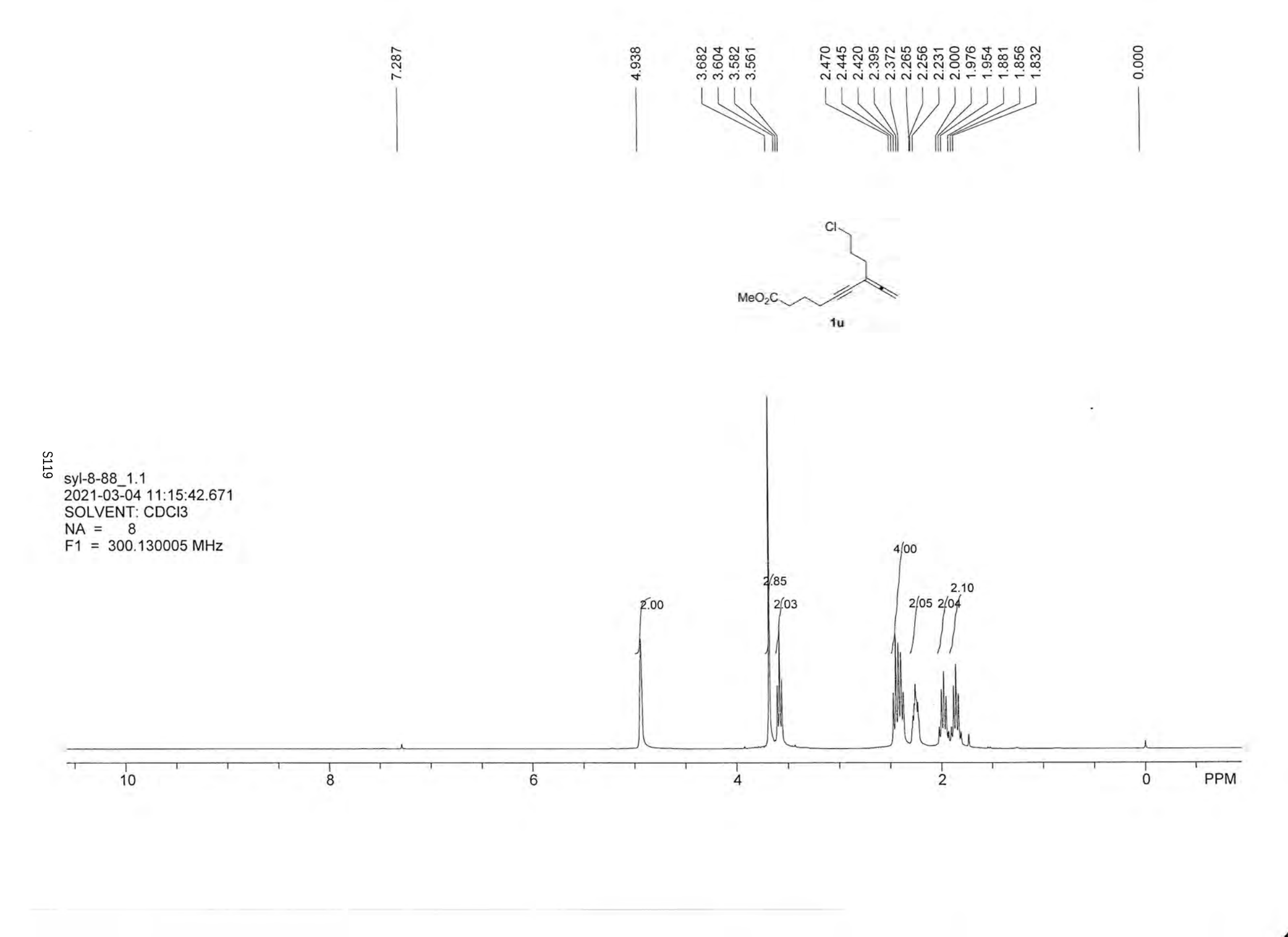

n-C5H11
F3C
1q

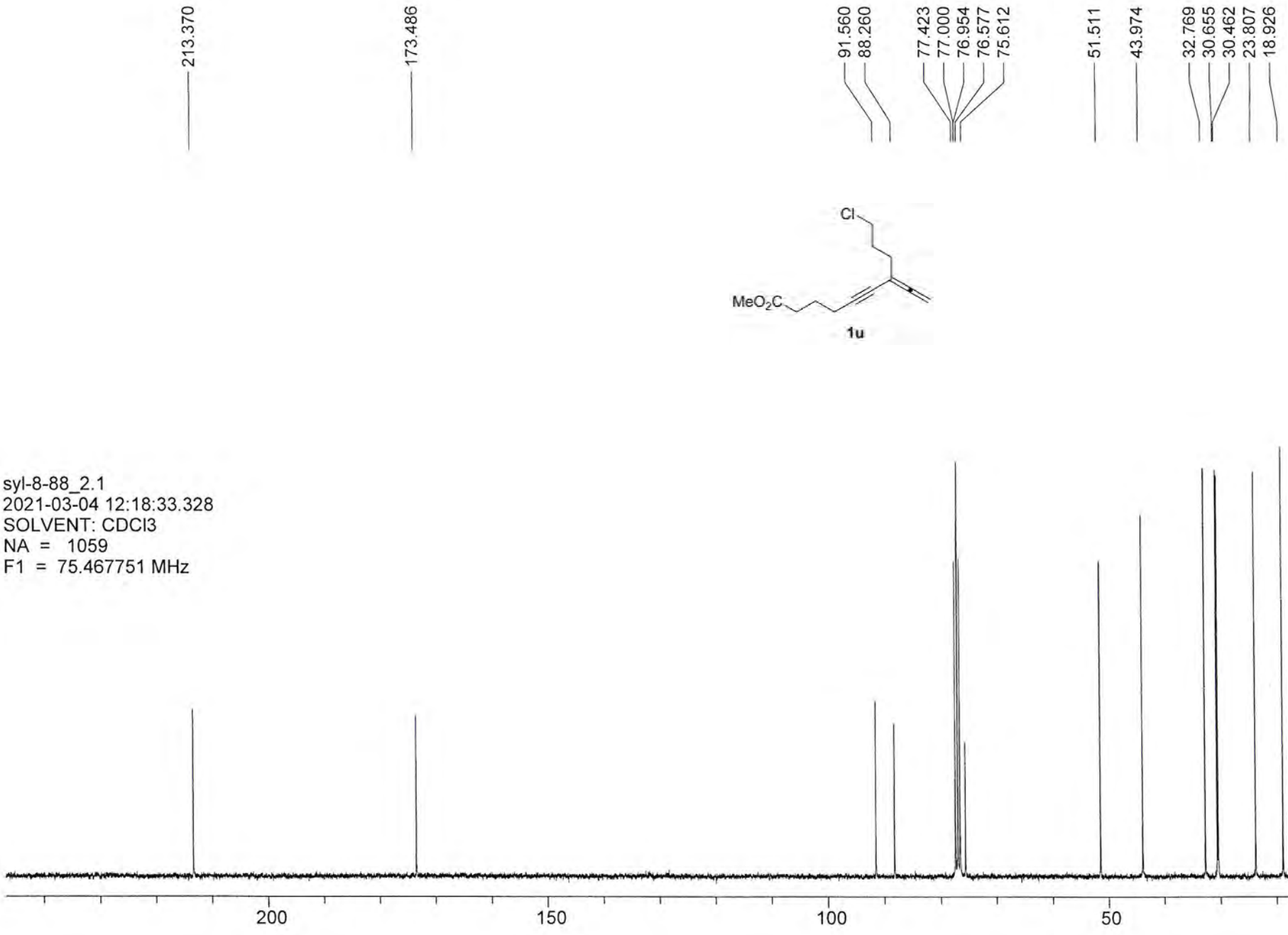

syl-8-22_2.1
2021-01-04 20:41:06.656
SOLVENT: CDCl3
NA = 171
F1 = 75.467751 MHz
200
150
100
50
0
PPM

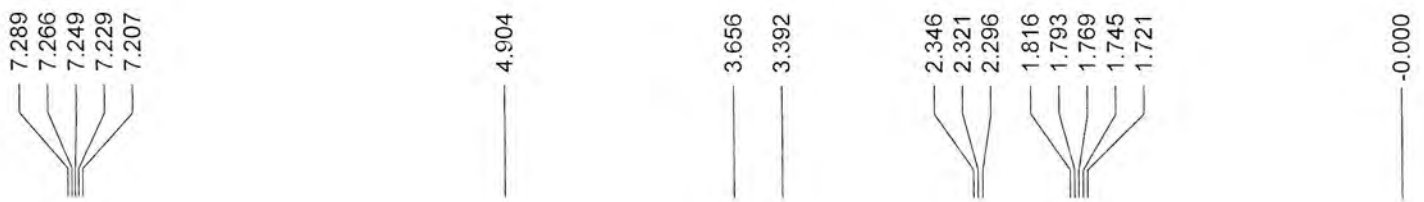

0.000
-63.236
n-C5H11
F3C
1q
syl-8-22_5.1
2021-01-05 14:47:28.781
SOLVENT: CDCl3
NA = 16
F1 = 282.404358 MHz
0
-20
-40
-60
-80
-100 PPM

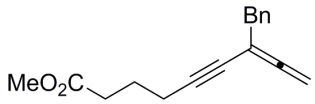

7.986
7.959
7.494
7.467
7.266
5.011
3.909
2.231
2.207
2.182
1.599
1.576
1.552
1.356
1.345
0.931
0.911
0.890
-0.000
n-C5H11
MeO2C
1s
syl-8-42_1.1
2021-01-17 15:39:00.281
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
1.96
1.98
2.00
3.02
2.03
1.97
4.01
3.04
10
8
6
4
2
0 PPM


213.839
166.492
131.204
129.366
129.145
128.217
90.641
89.639
87.819
77.423
77.000
76.890
76.577
52.090
33.091
31.013
27.401
22.373
13.981
n-C5H11
MeO2C
1s
syl-8-42_2.1
2021-01-17 15:33:10.171
SOLVENT: CDCl3
NA = 201
F1 = 75.467751 MHz
200
150
100
50
0 PPM

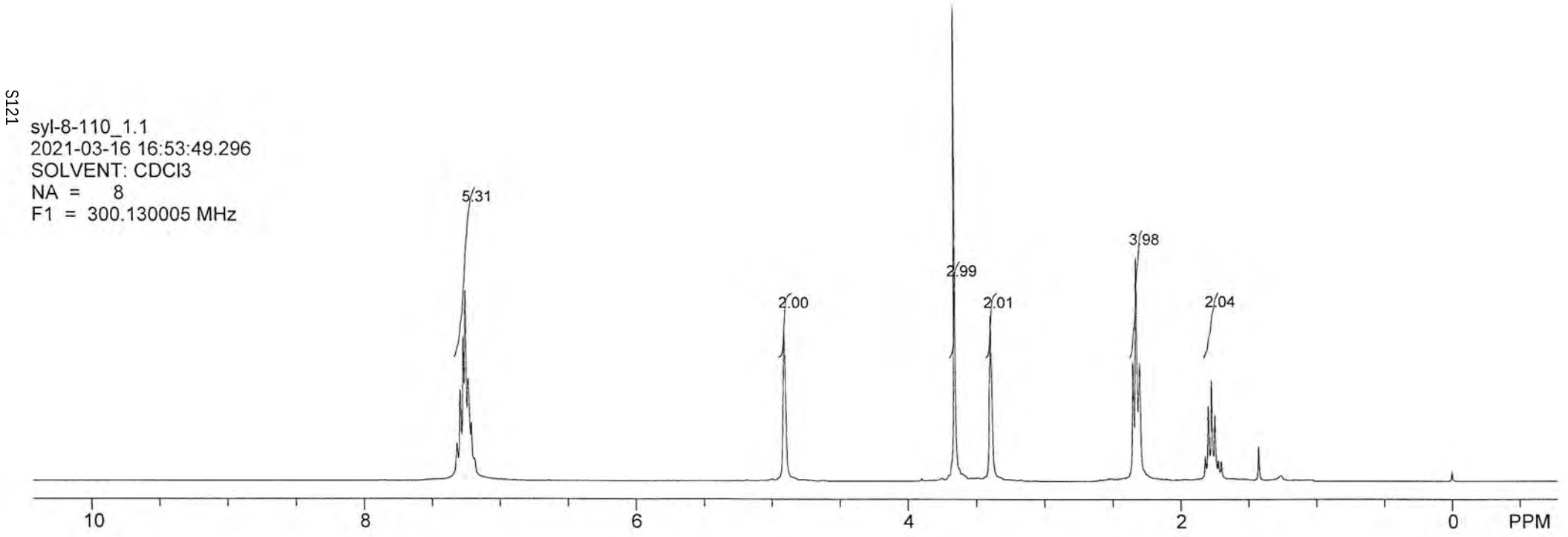

7.287
4.938
3.682
3.604
3.582
3.561
2.470
2.445
2.420
2.395
2.372
2.265
2.256
2.231
2.000
1.976
1.954
1.881
1.856
1.832
0.000
Cl
MeO2C
1u
syl-8-88_1.1
2021-03-04 11:15:42.671
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
2.00
2.85
2.03
4.00
2.05
2.04
2.10
10
8
6
4
2
0
PPM

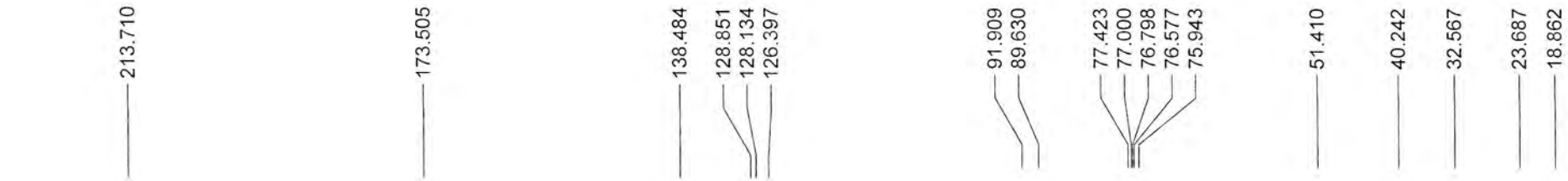

213.370
173.486
91.560
88.260
77.423
77.000
76.954
76.577
75.612
51.511
43.974
32.769
30.655
30.462
23.807
18.926
Cl
MeO2C
1u
syl-8-88_2.1
2021-03-04 12:18:33.328
SOLVENT: CDCl3
NA = 1059
F1 = 75.467751 MHz
200
150
100
50
0
PPM

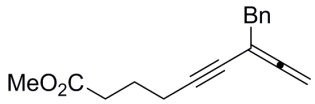

7.289
7.266
7.249
7.229
7.207
4.904
3.656
3.392
2.346
2.321
2.296
1.816
1.793
1.769
1.745
1.721
-0.000


Bn
MeO2C

1v

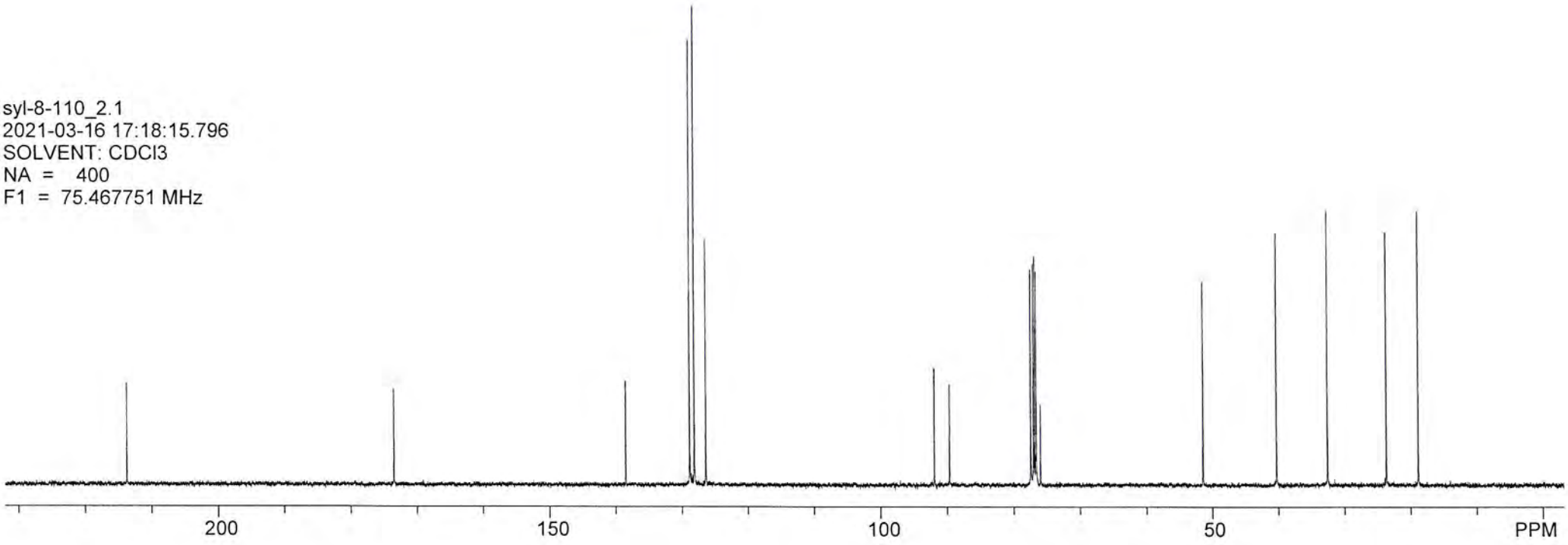

syl-8-110_1.1
2021-03-16 16:53:49.296
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
5.31
2.00
2.99
2.01
3.98
2.04
10
8
6
4
2
0
PPM

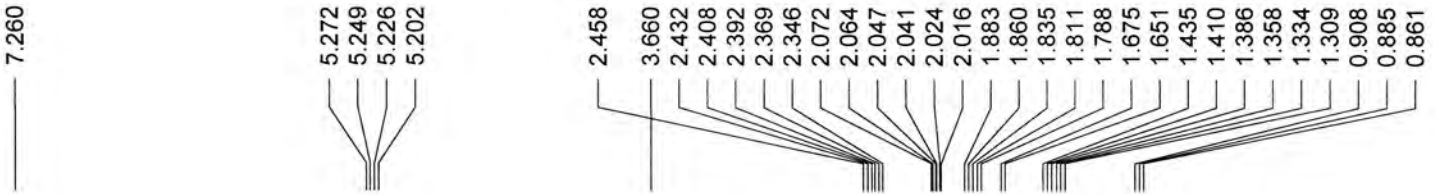


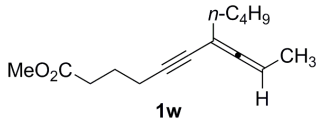


1v

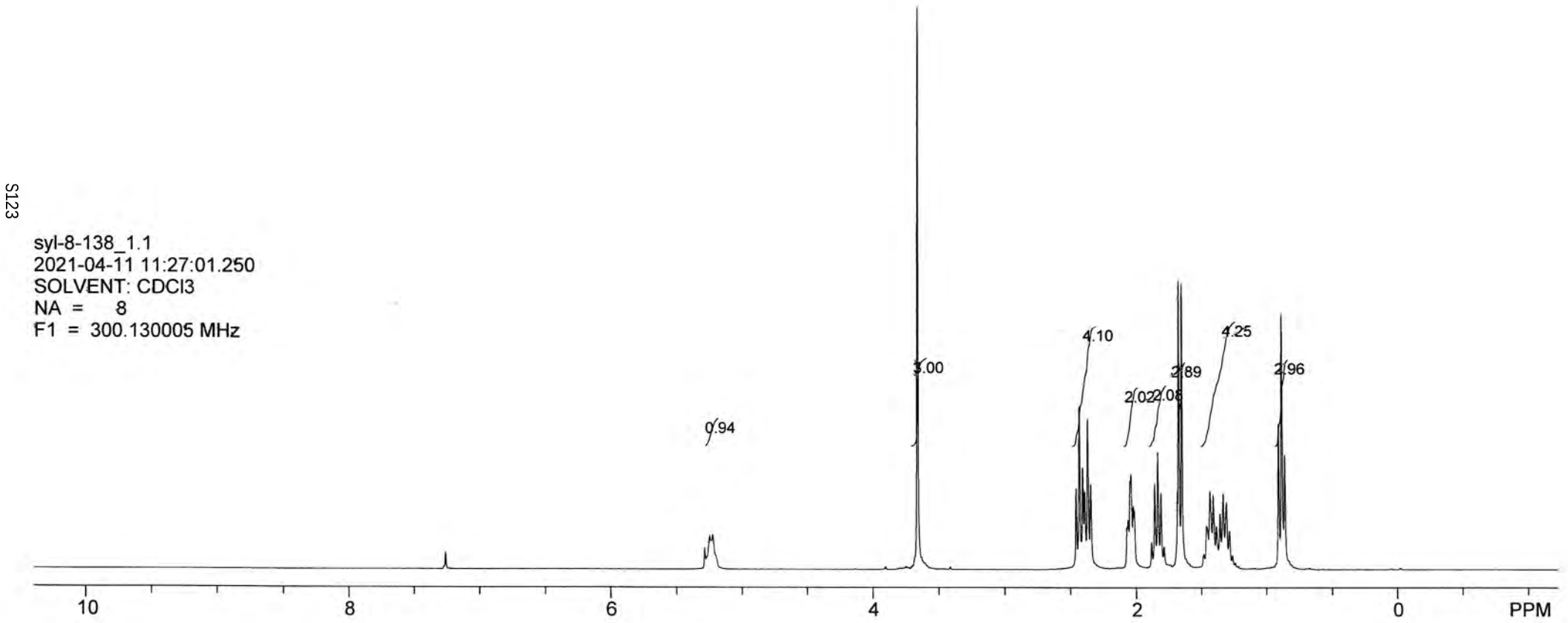
syl-8-110_2.1
2021-03-16 17:18:15.796
SOLVENT: CDCl3
NA = 400
F1 = 75.467751 MHz

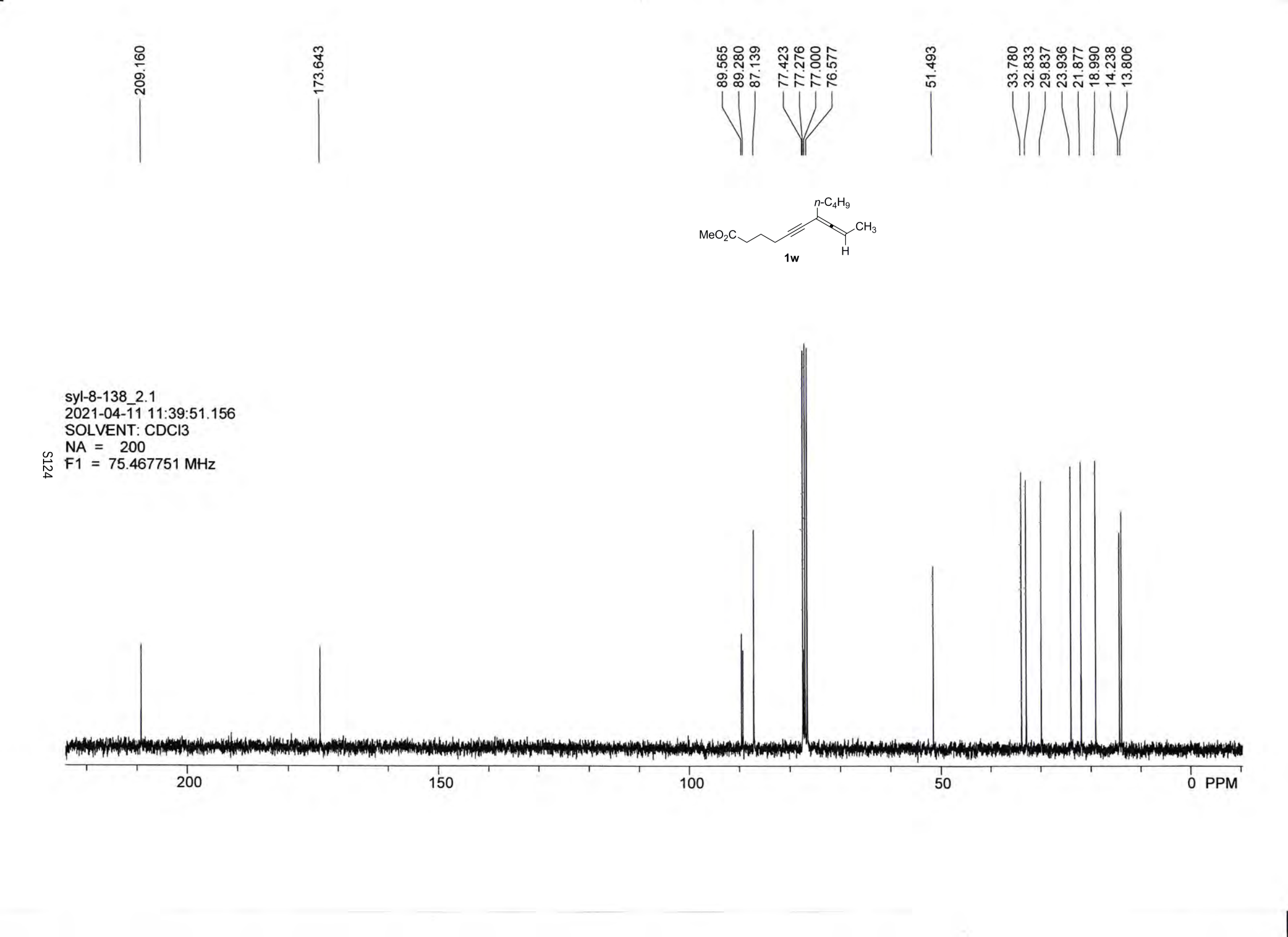


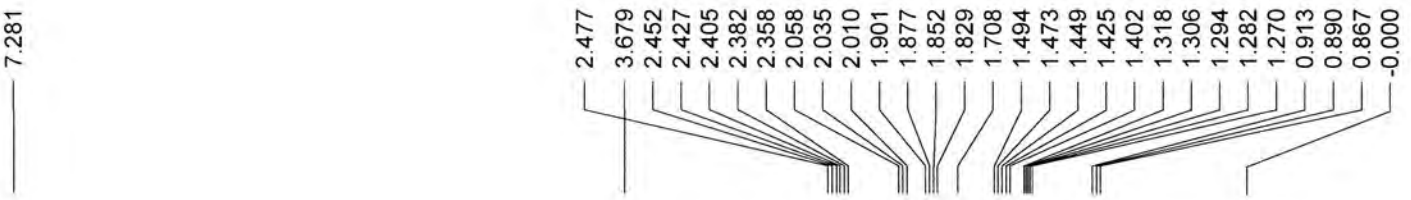

7.260
5.272
5.249
5.226
5.202
2.458
3.660
2.432
2.408
2.392
2.369
2.346
2.072
2.064
2.047
2.041
2.024
2.016
1.883
1.860
1.835
1.811
1.788
1.675
1.651
1.435
1.410
1.386
1.358
1.334
1.309
0.908
0.885
0.861

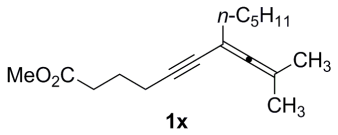

n-C4H9
MeO2C
CH3
H

1w


syl-8-138_1.1
2021-04-11 11:27:01.250
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
0.94
3.00
4.10
2.02
2.08
2.89
4.25
2.96
10
8
6
4
2
0
PPM

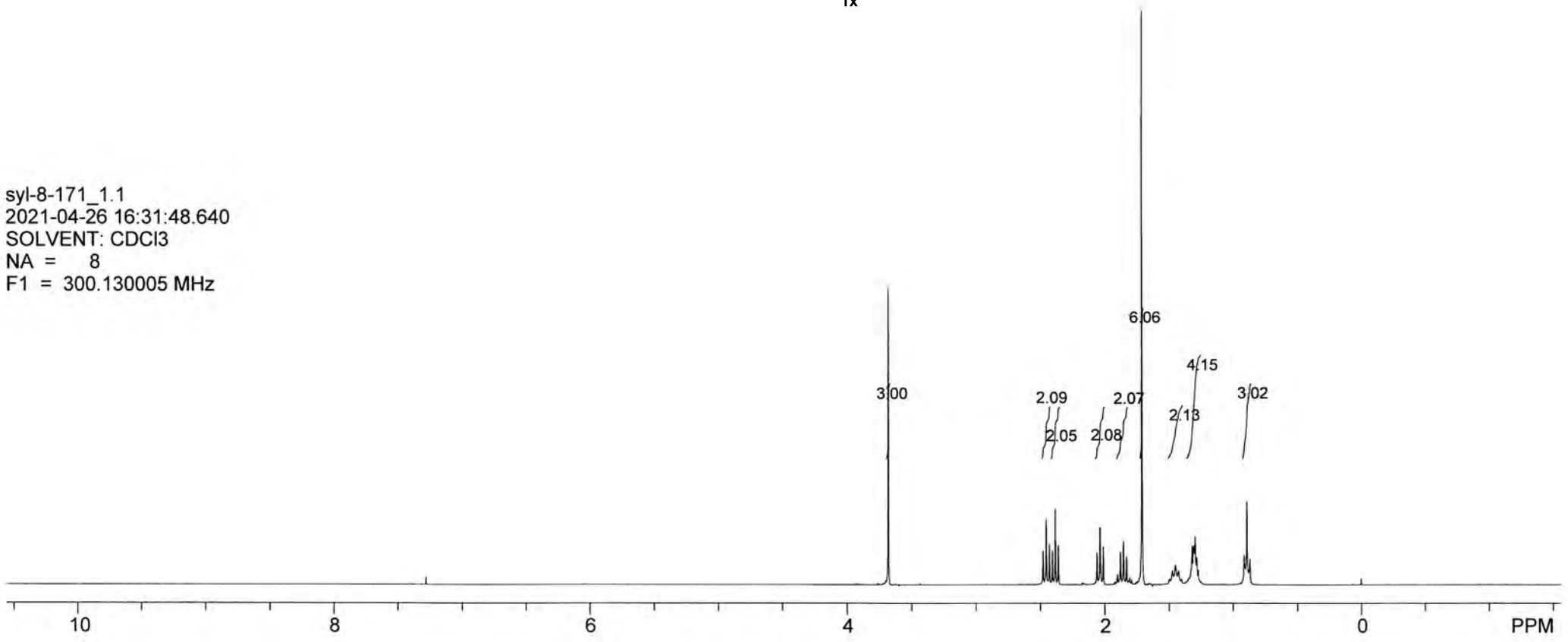

209.160
173.643
89.565
89.280
87.139
77.423
77.276
77.000
76.577
51.493
33.780
32.833
29.837
23.936
21.877
18.990
14.238
13.806
n-C4H9
MeO2C
CH3
H
1w
syl-8-138_2.1
2021-04-11 11:39:51.156
SOLVENT: CDCl3
NA = 200
F1 = 75.467751 MHz
200
150
100
50
0 PPM

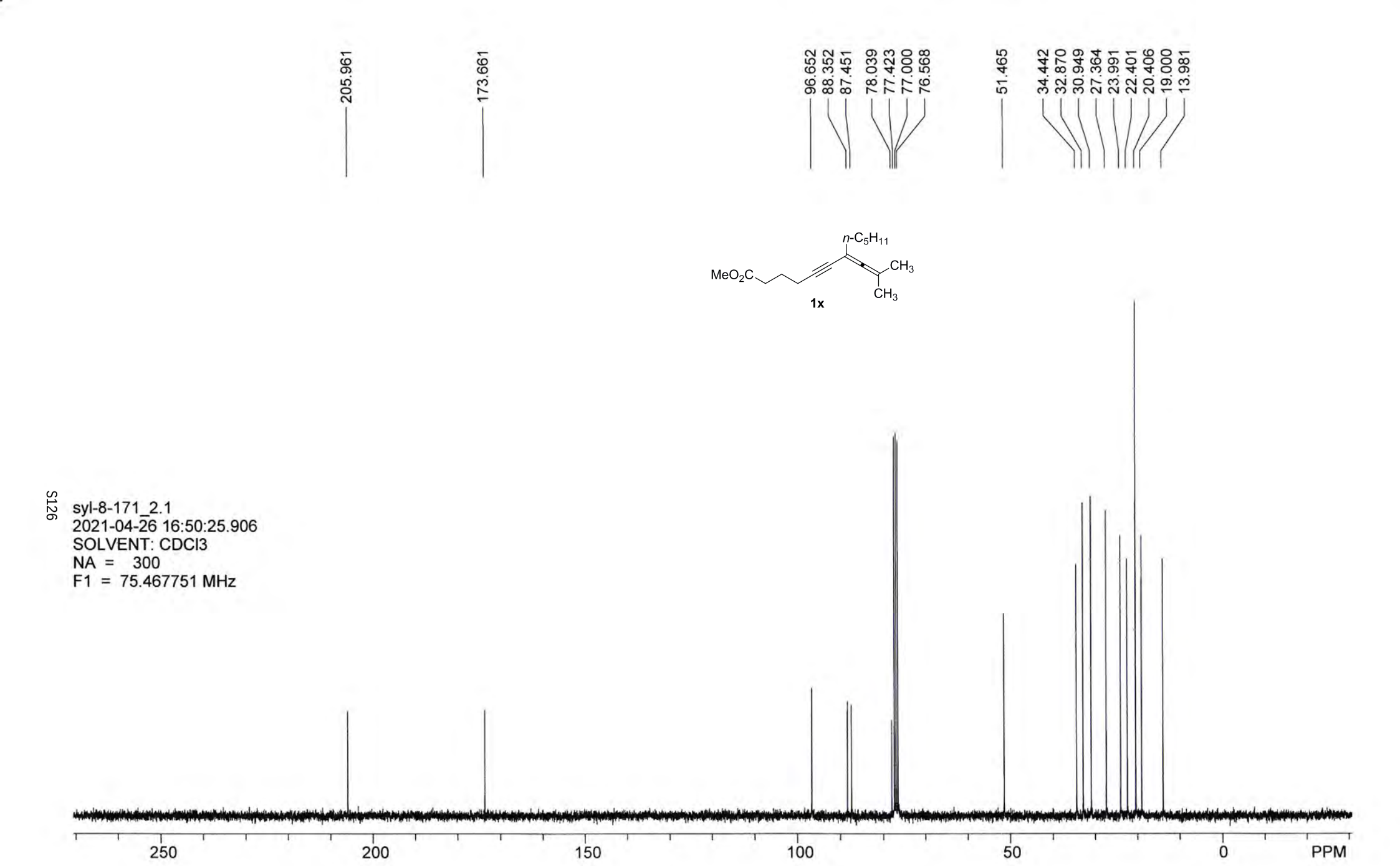


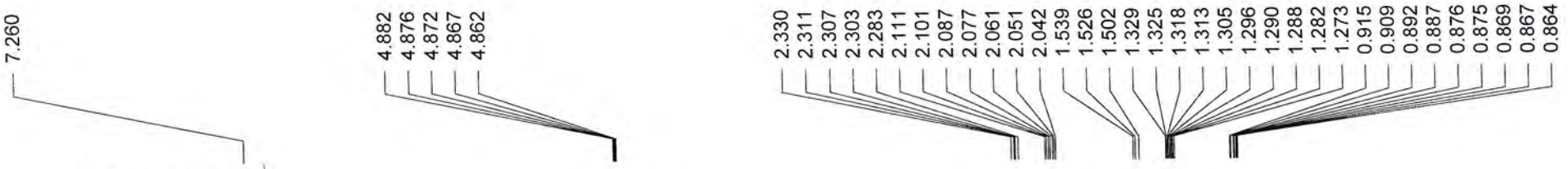


1x

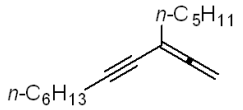
syl-8-171_1.1
2021-04-26 16:31:48.640
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz



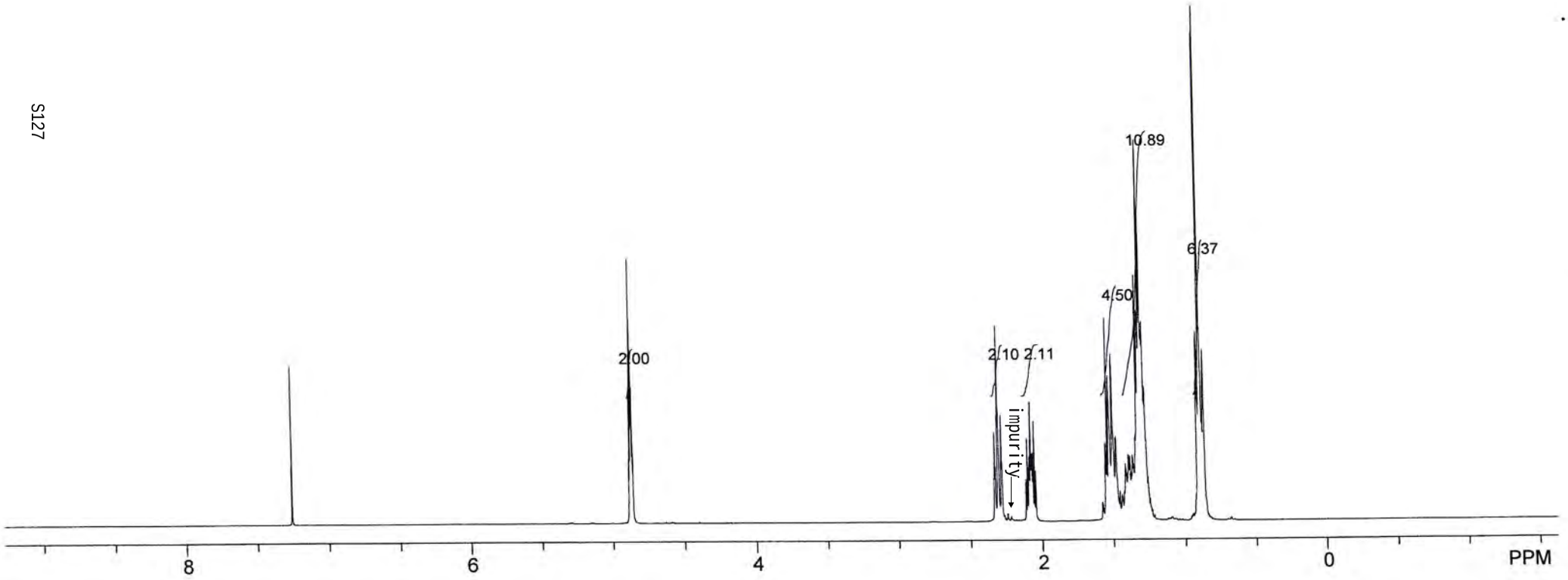

205.961
173.661
96.652
88.352
87.451
78.039
77.423
77.000
76.568
51.465
34.442
32.870
30.949
27.364
23.991
22.401
20.406
19.000
13.981
n-C5H11
MeO2C
CH3
CH3
1x
syl-8-171_2.1
2021-04-26 16:50:25.906
SOLVENT: CDCl3
NA = 300
F1 = 75.467751 MHz
250
200
150
100
50
0
PPM



zj-5-23-pdt-1_2.1
2023-03-12 18:01:16.124
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 300.130005 MHz



1y



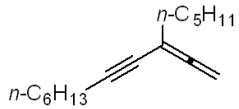
213.431

92.773
90.007

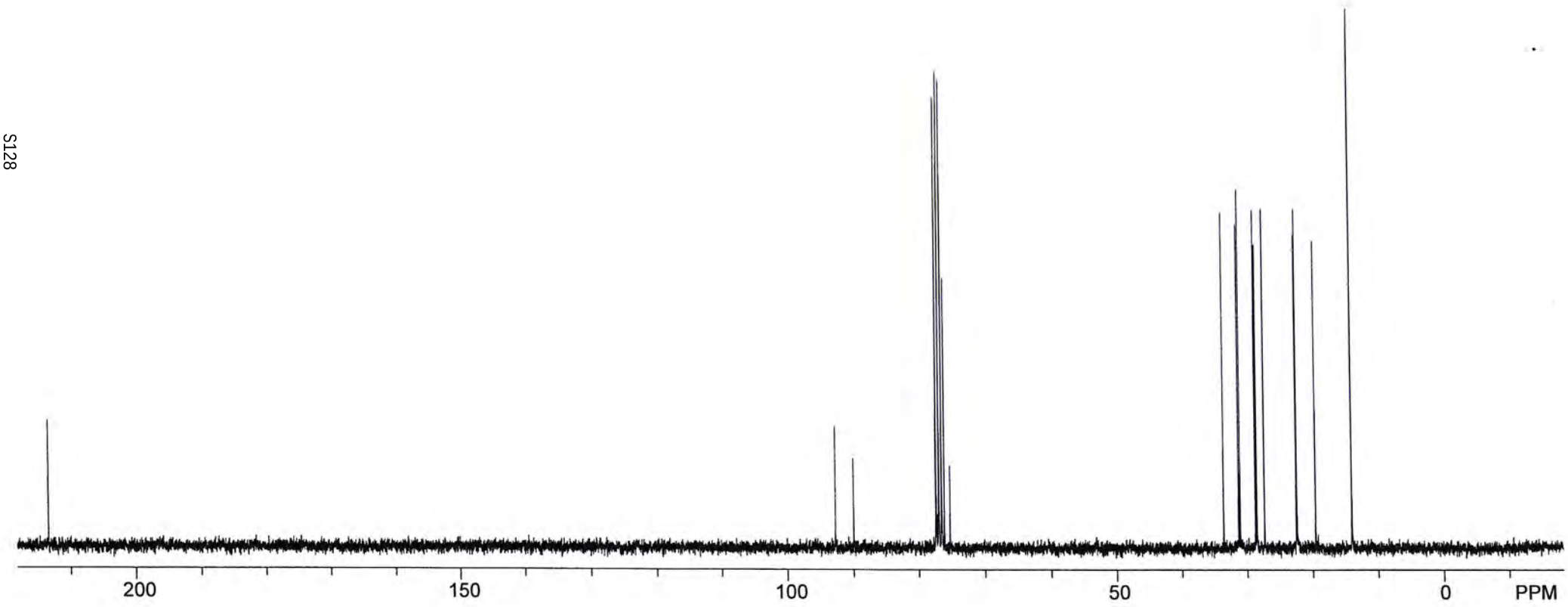
77.419
77.000
76.574
76.169
75.303

33.617
31.328
31.104
28.764
28.548
27.378
22.539
22.438
19.585
14.010

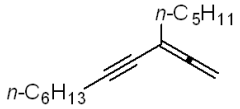
zj-5-23-pdt-C_5.1
2023-03-12 19:04:18.818
NA = 692
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz



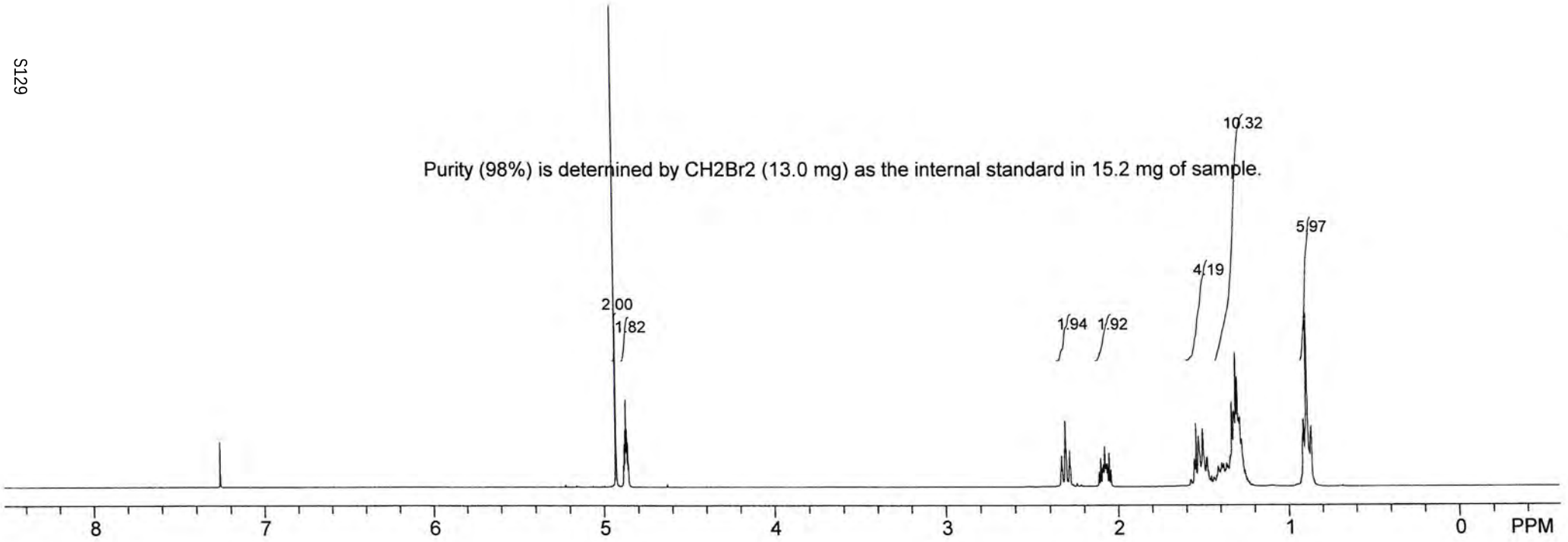
1y



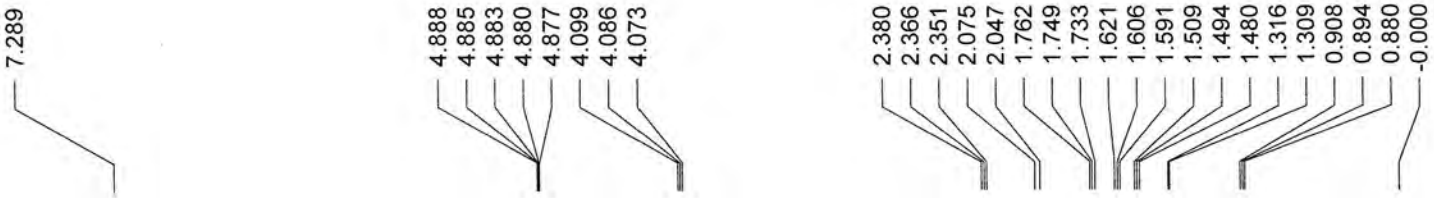
zj-5-23-purity_3.1
2023-03-12 20:54:47.070
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 300.130005 MHz

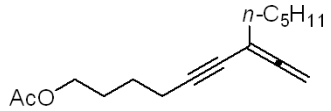


1y



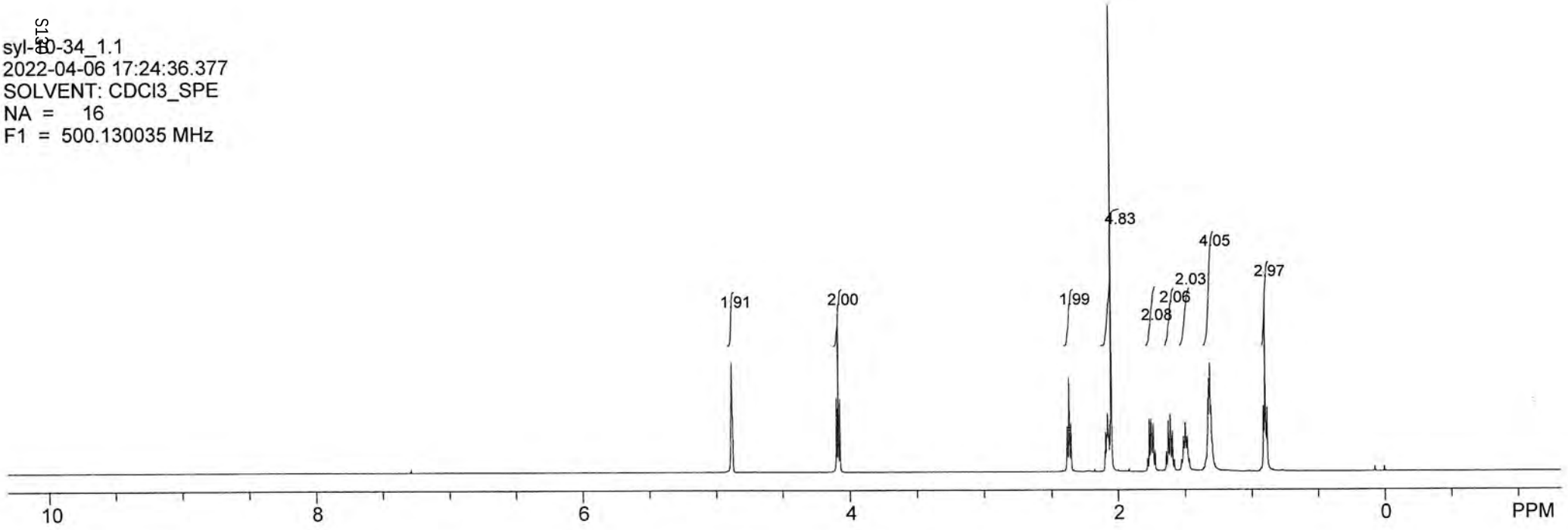
Purity (98%) is determined by CH2Br2 (13.0 mg) as the internal standard in 15.2 mg of sample.

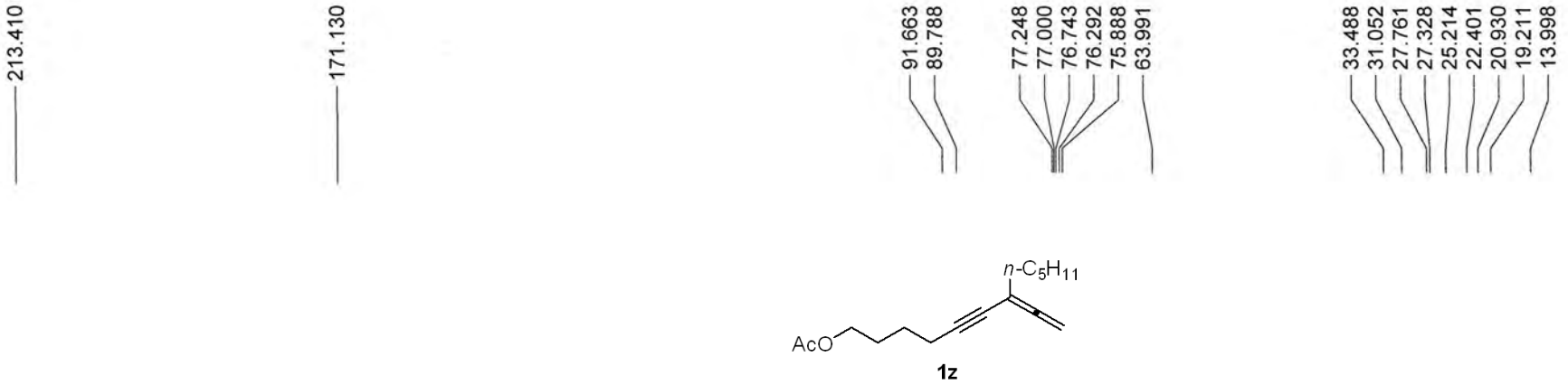




1z

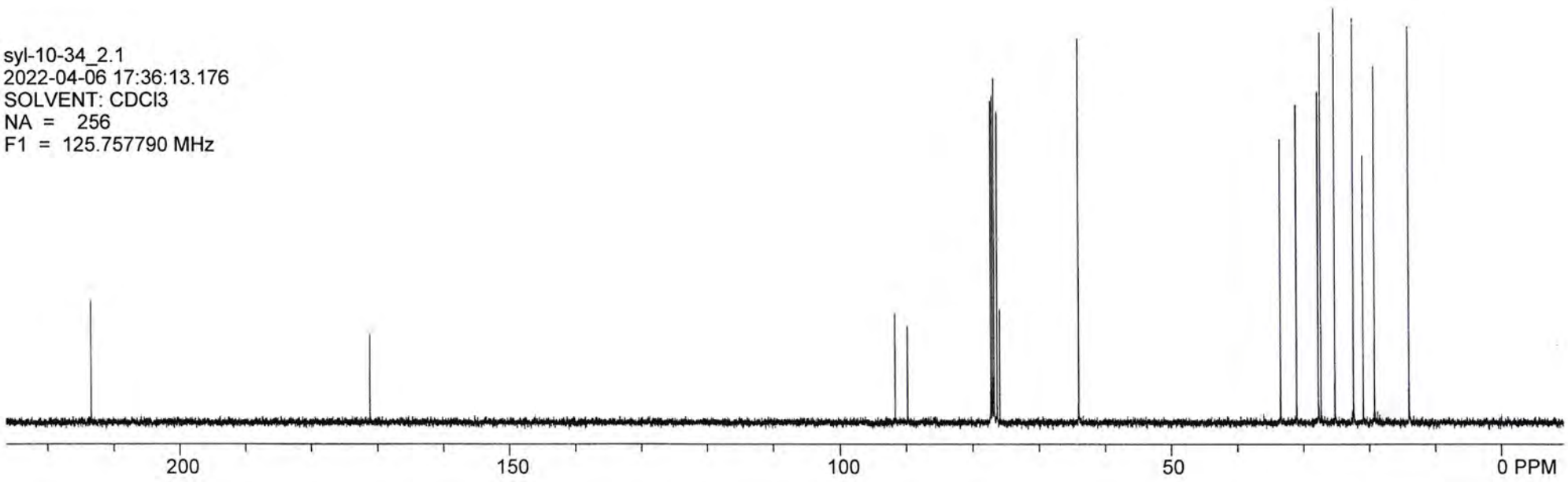
syl-ad-34_1.1
2022-04-06 17:24:36.377
SOLVENT: CDCl3_SPE
NA = 16
F1 = 500.130035 MHz



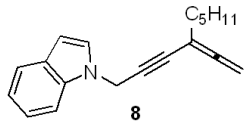

213.410
171.130
91.663
89.788
77.248
77.000
76.743
76.292
75.888
63.991
33.488
31.052
27.761
27.328
25.214
22.401
20.930
19.211
13.998

n-C5H11
AcO

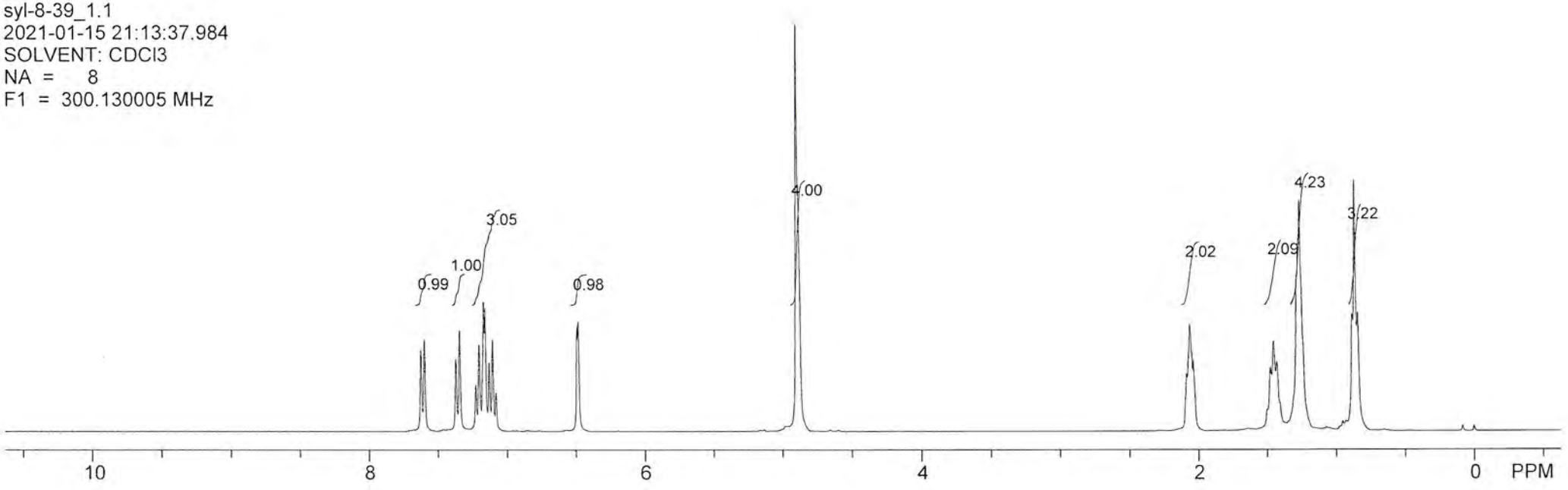
1z

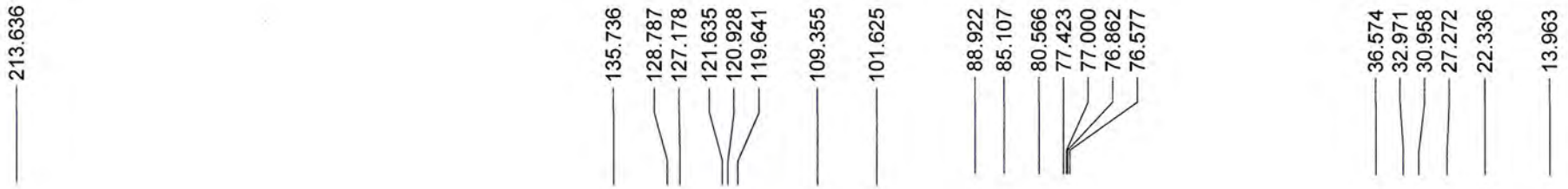

syl-10-34_2.1
2022-04-06 17:36:13.176
SOLVENT: CDCl3
NA = 256
F1 = 125.757790 MHz
200
150
100
50
0 PPM

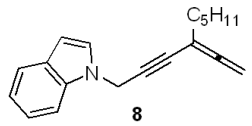

7.623
7.597
7.370
7.343
7.226
7.202
7.169
7.158
7.130
7.105
6.492
6.482
4.898
4.886
2.084
2.060
2.036
1.473
1.451
1.427
1.282
1.268
1.259
0.881
0.862
0.839
-0.000


C_5H_{11}
N

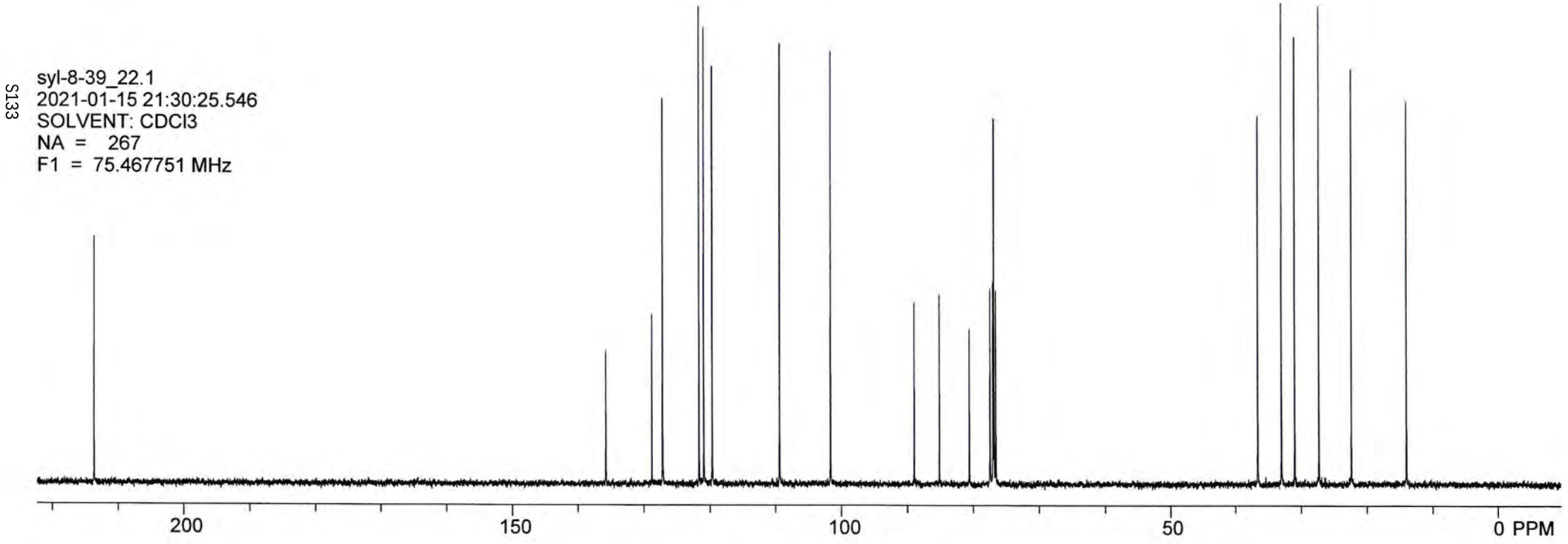
8

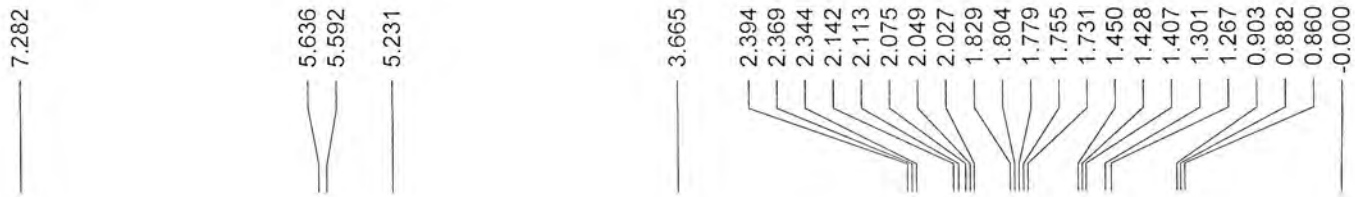

syl-8-39_1.1
2021-01-15 21:13:37.984
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
0.99
1.00
3.05
0.98
4.00
2.02
2.09
4.23
3.22
10
8
6
4
2
0
PPM

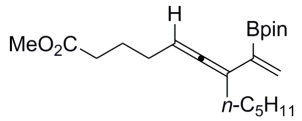

213.636
135.736
128.787
127.178
121.635
120.928
119.641
109.355
101.625
88.922
85.107
80.566
77.423
77.000
76.862
76.577
36.574
32.971
30.958
27.272
22.336
13.963


C_5H_{11}
N

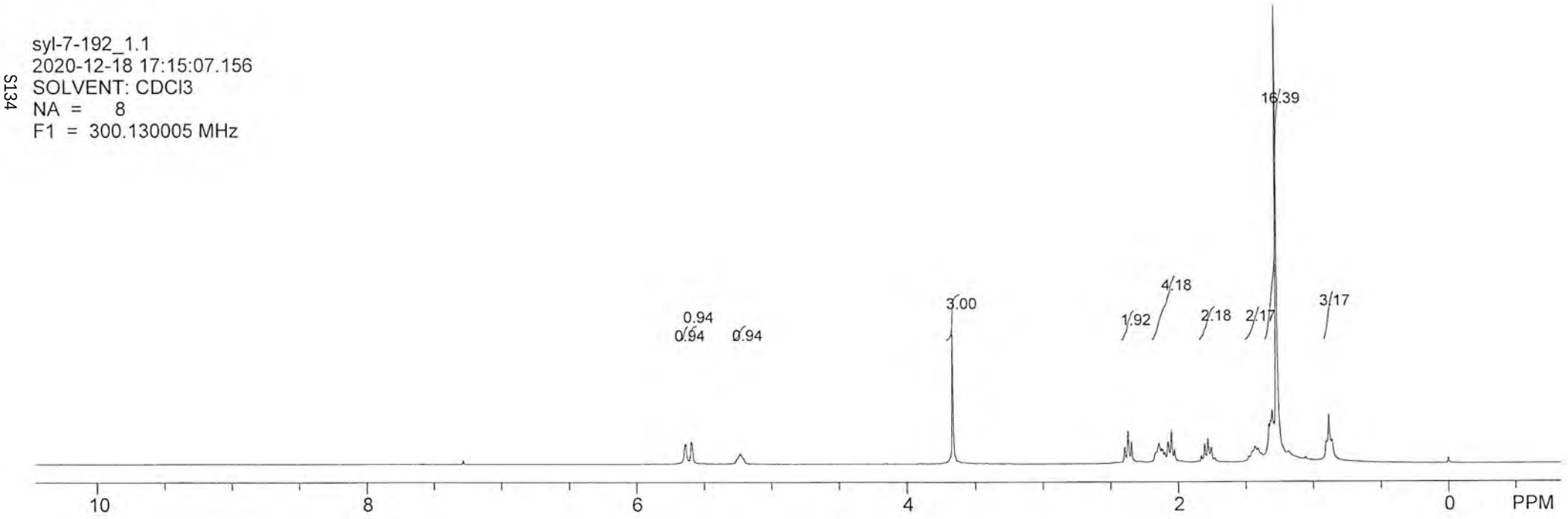
8


syl-8-39_22.1
2021-01-15 21:30:25.546
SOLVENT: CDCl3
NA = 267
F1 = 75.467751 MHz
200
150
100
50
0 PPM

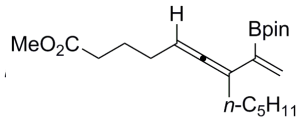

7.282
5.636
5.592
5.231
3.665
2.394
2.369
2.344
2.142
2.113
2.075
2.049
2.027
1.829
1.804
1.779
1.755
1.731
1.450
1.428
1.407
1.301
1.267
0.903
0.882
0.860
-0.000


H
MeO2C
Bpin
n-C5H11

3aa

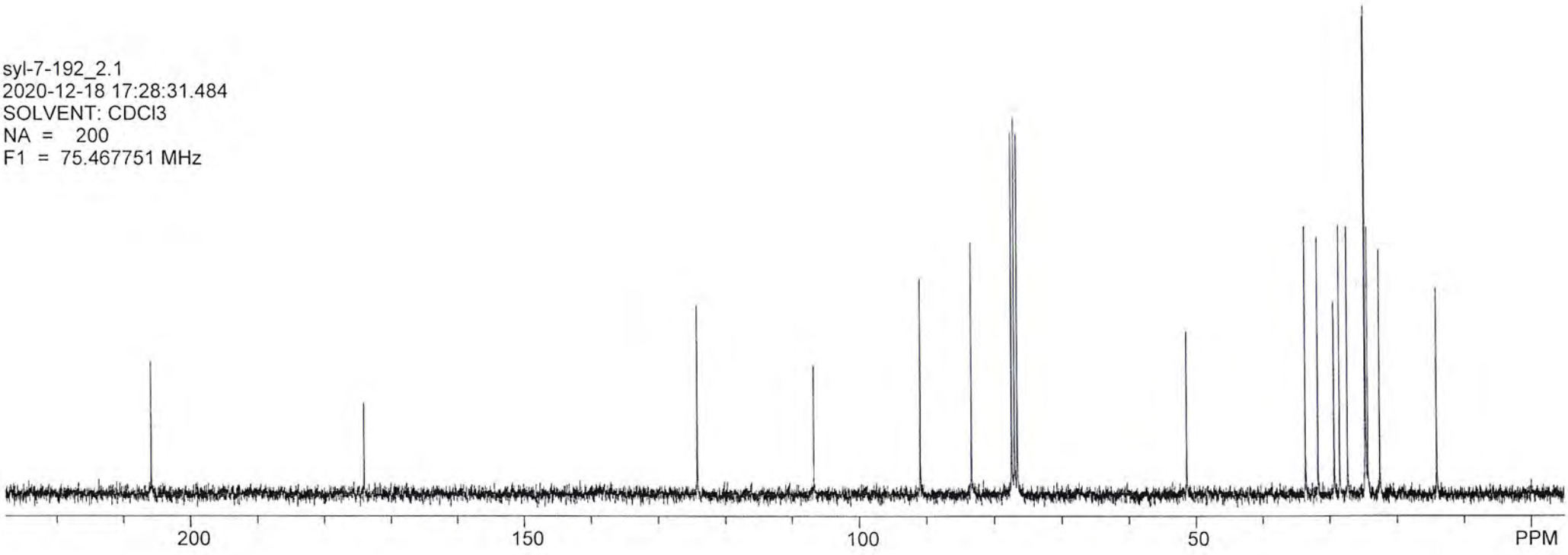

syl-7-192_1.1
2020-12-18 17:15:07.156
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
0.94
0.94
0.94
3.00
1.92
4.18
2.18
2.17
16.39
3.17
10
8
6
4
2
0
PPM

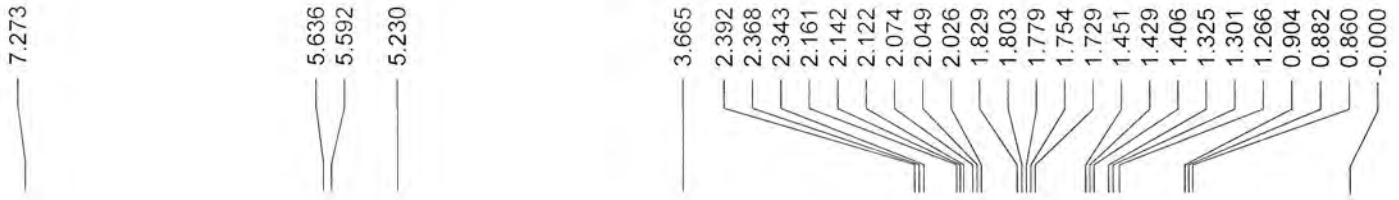
205.869
174.020
124.218
106.781
90.944
83.388
77.423
77.000
76.577
51.382
33.569
31.694
29.304
28.486
27.309
24.680
24.579
24.285
22.511
14.045

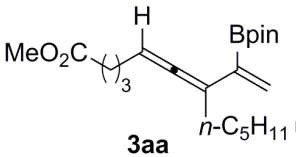


3aa

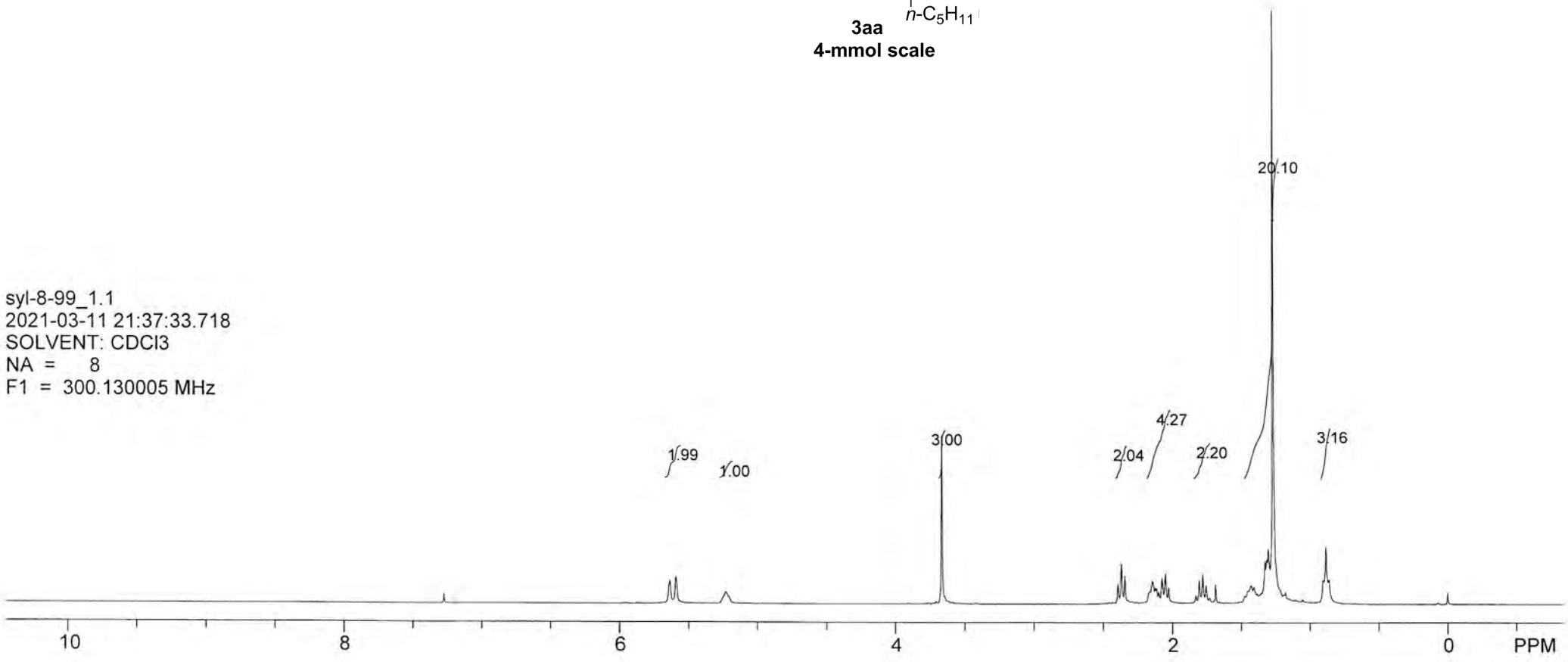
syl-7-192_2.1
2020-12-18 17:28:31.484
SOLVENT: CDCl3
NA = 200
F1 = 75.467751 MHz



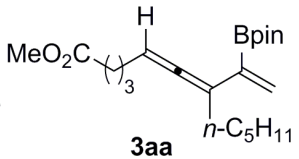




3aa
4-mmol scale

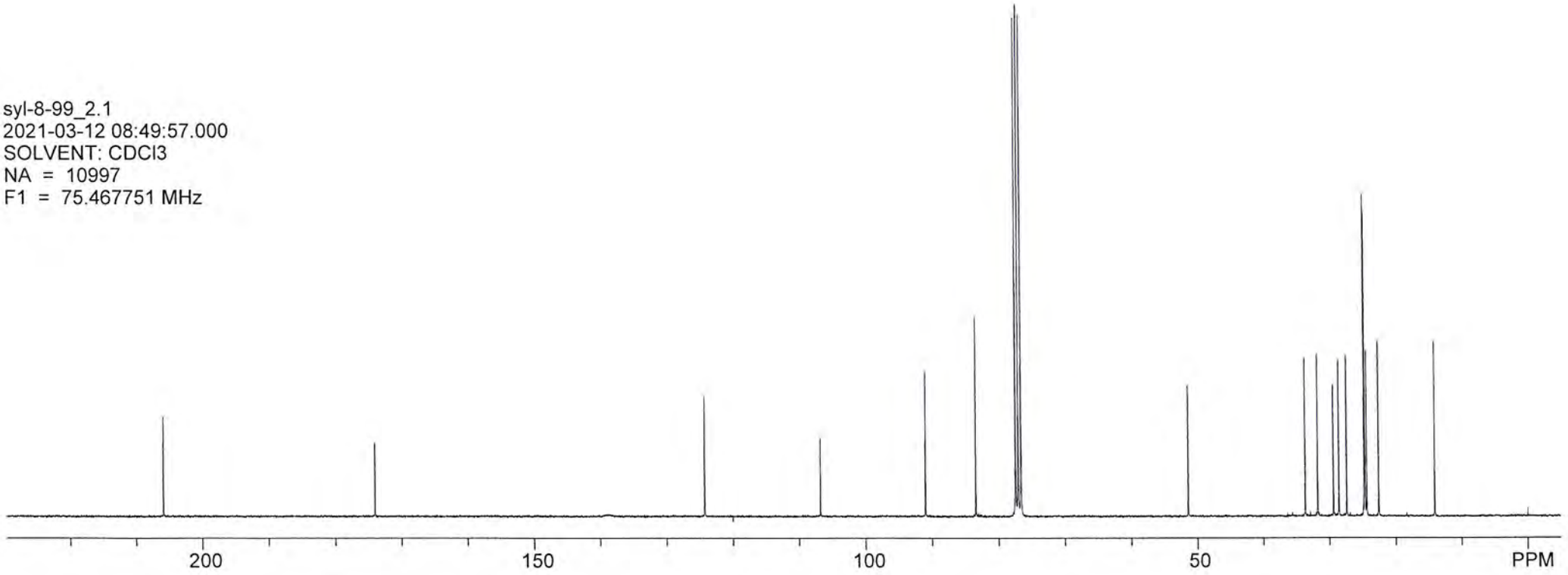


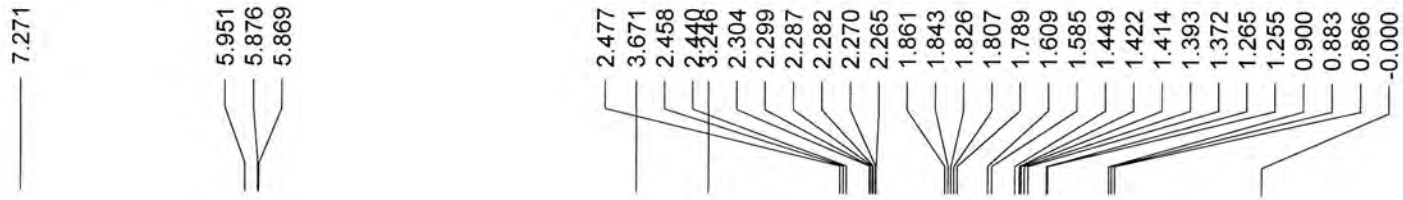
205.878
174.047
124.264
106.818
90.962
83.416
77.423
77.000
76.577
51.401
33.596
31.712
29.350
28.513
27.346
24.708
24.597
24.322
22.529
14.054

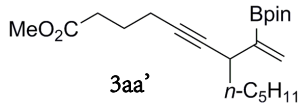


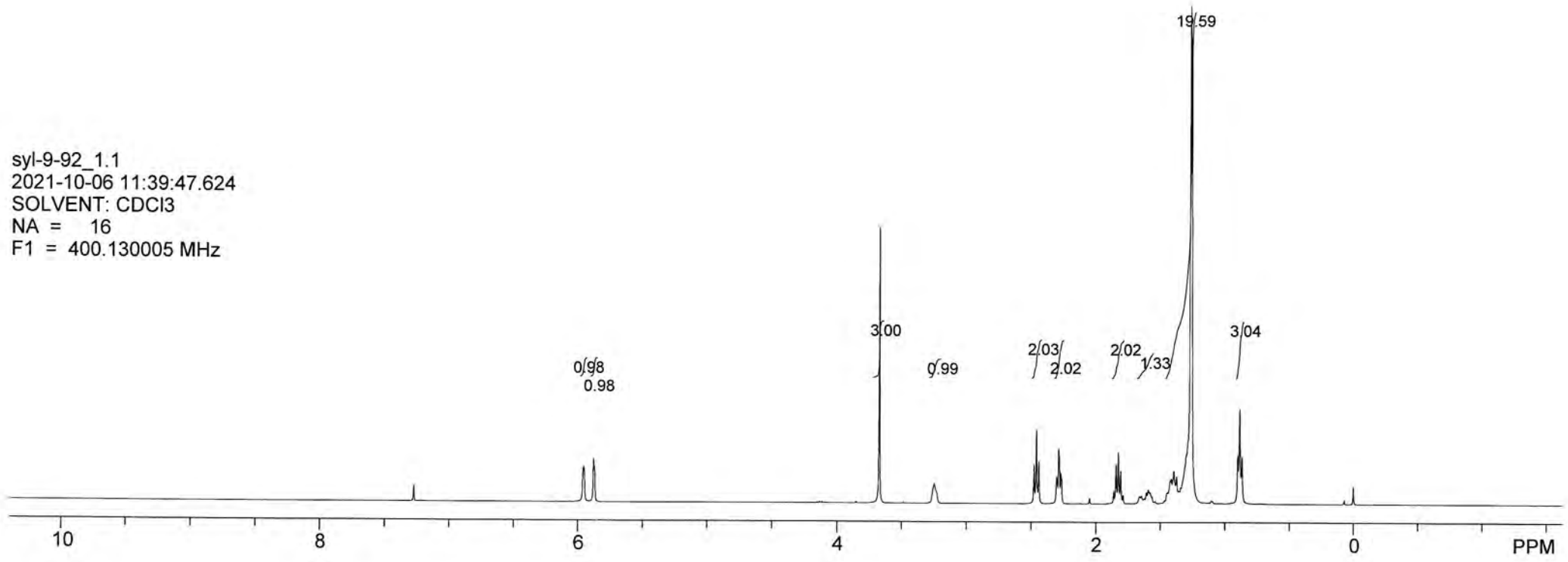
3aa
4-mmol scale

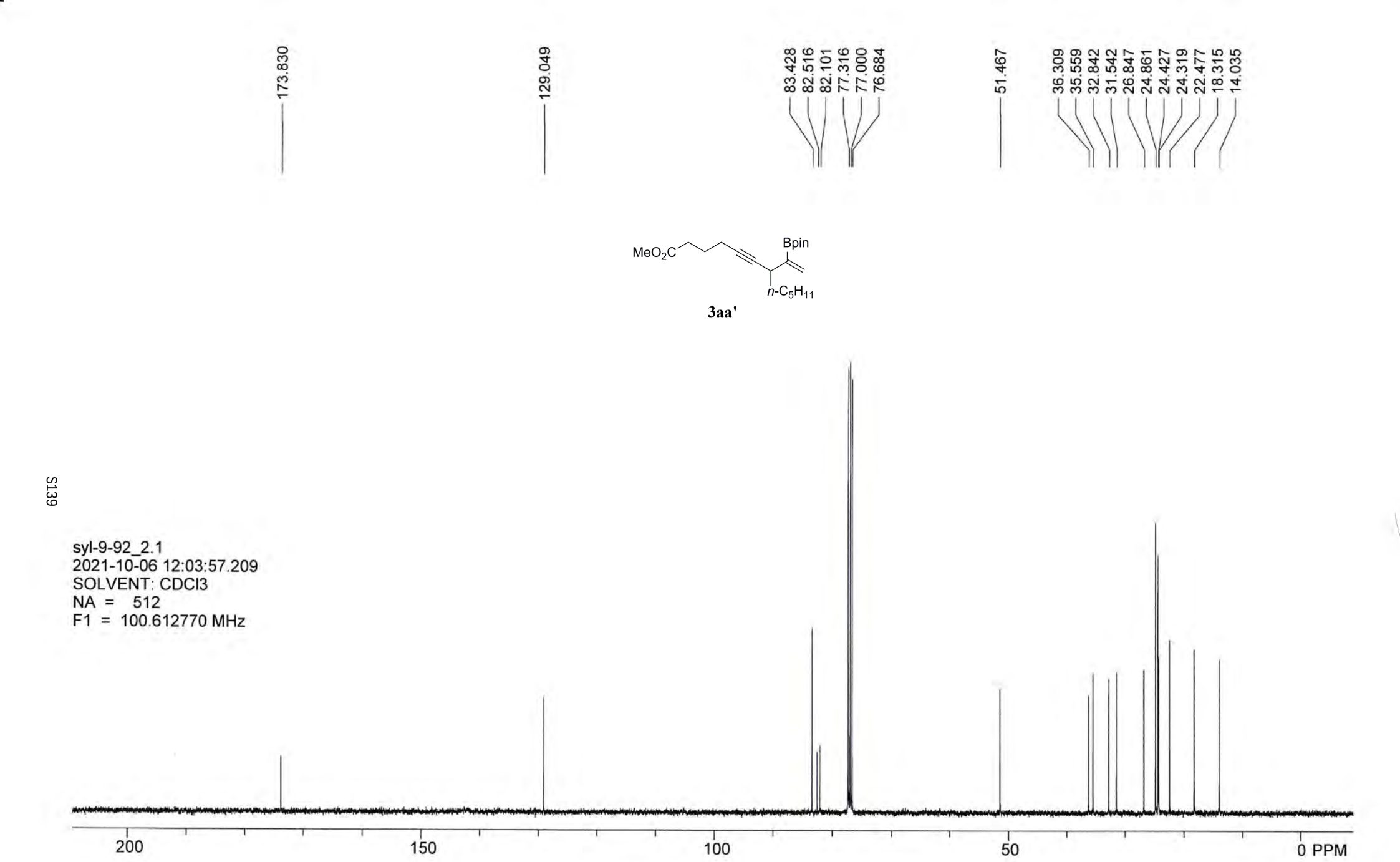
syl-8-99_2.1
2021-03-12 08:49:57.000
SOLVENT: CDCl3
NA = 10997
F1 = 75.467751 MHz

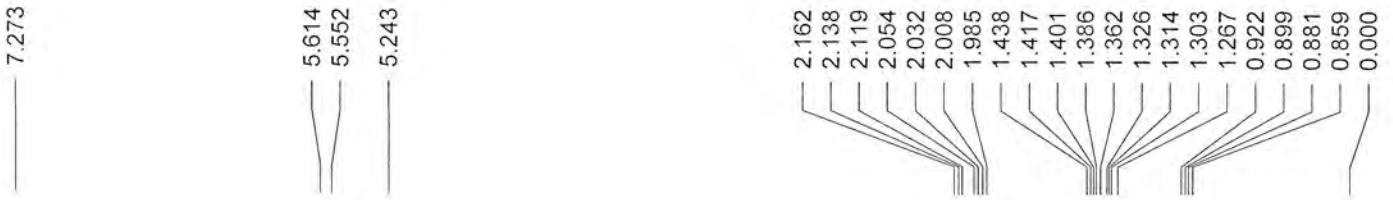


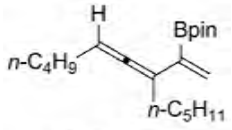

7.271
5.951
5.876
5.869
2.477
3.671
2.458
2.440
3.248
2.304
2.299
2.287
2.282
2.270
2.265
1.861
1.843
1.826
1.807
1.789
1.609
1.585
1.449
1.422
1.414
1.393
1.372
1.265
1.255
0.900
0.883
0.866
-0.000


MeO2C
Bpin
3aa'
n-C5H11

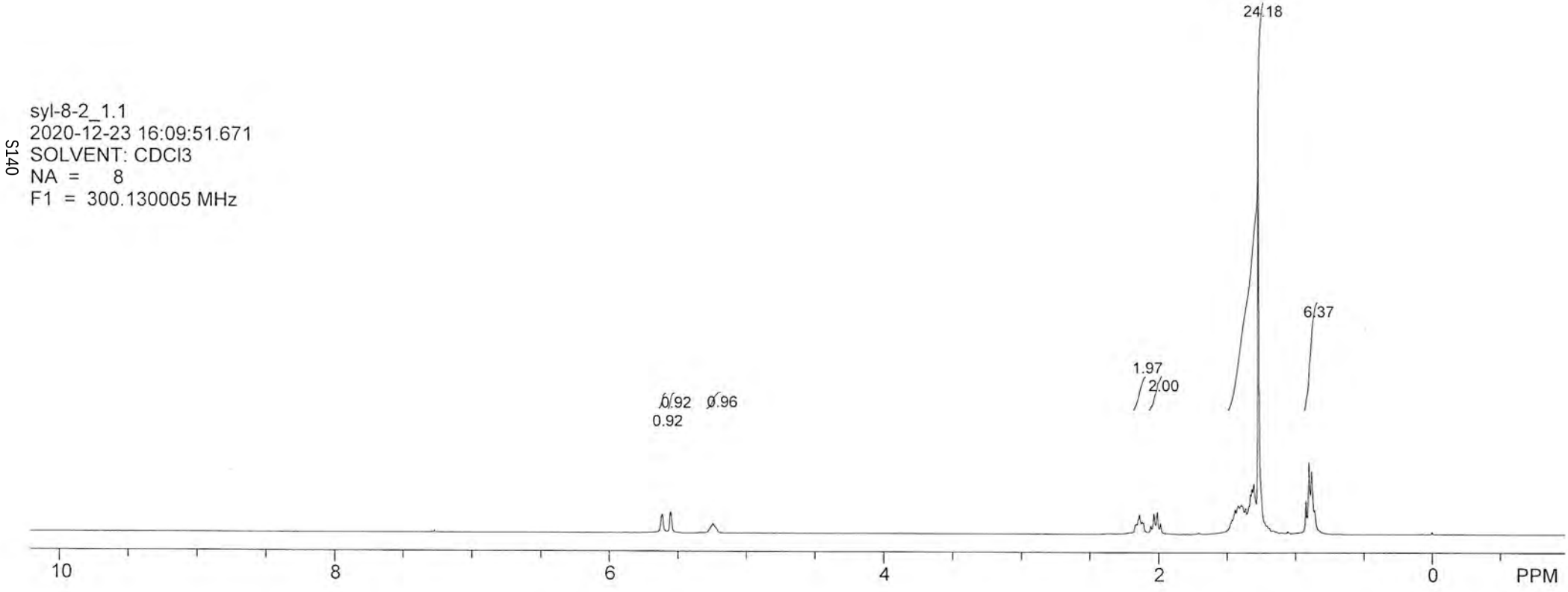

syl-9-92_1.1
2021-10-06 11:39:47.624
SOLVENT: CDCl3
NA = 16
F1 = 400.130005 MHz
19.59
3.00
3.04
2.03
2.02
2.02
1.33
0.99
0.98
0.98
10
8
6
4
2
0
PPM

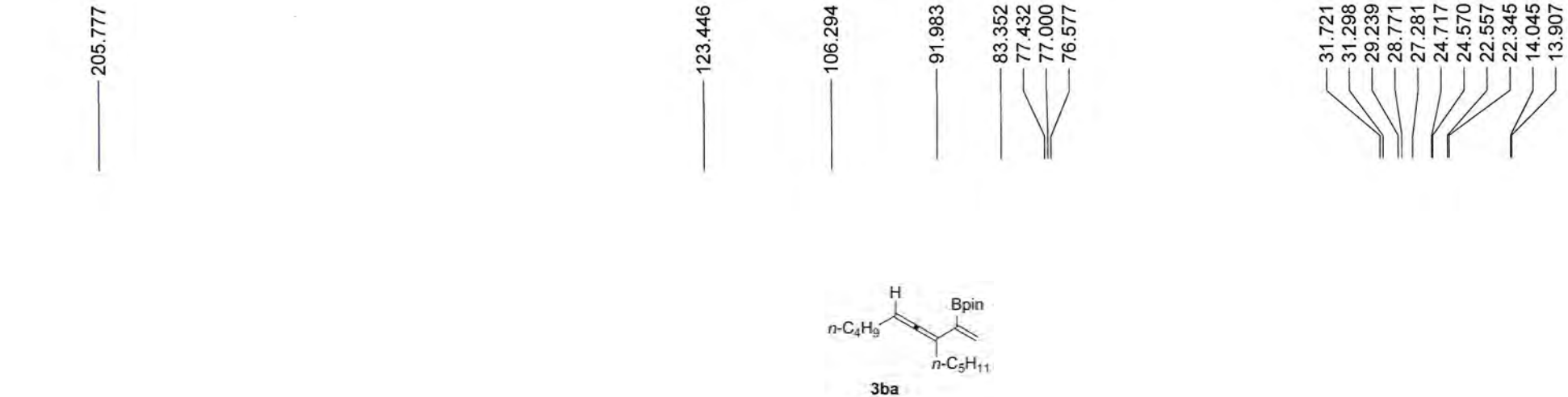

173.830
129.049
83.428
82.516
82.101
77.316
77.000
76.684
51.467
36.309
35.559
32.842
31.542
26.847
24.861
24.427
24.319
22.477
18.315
14.035
MeO2C
Bpin
n-C5H11
3aa'
syl-9-92_2.1
2021-10-06 12:03:57.209
SOLVENT: CDCl3
NA = 512
F1 = 100.612770 MHz
200
150
100
50
0 PPM

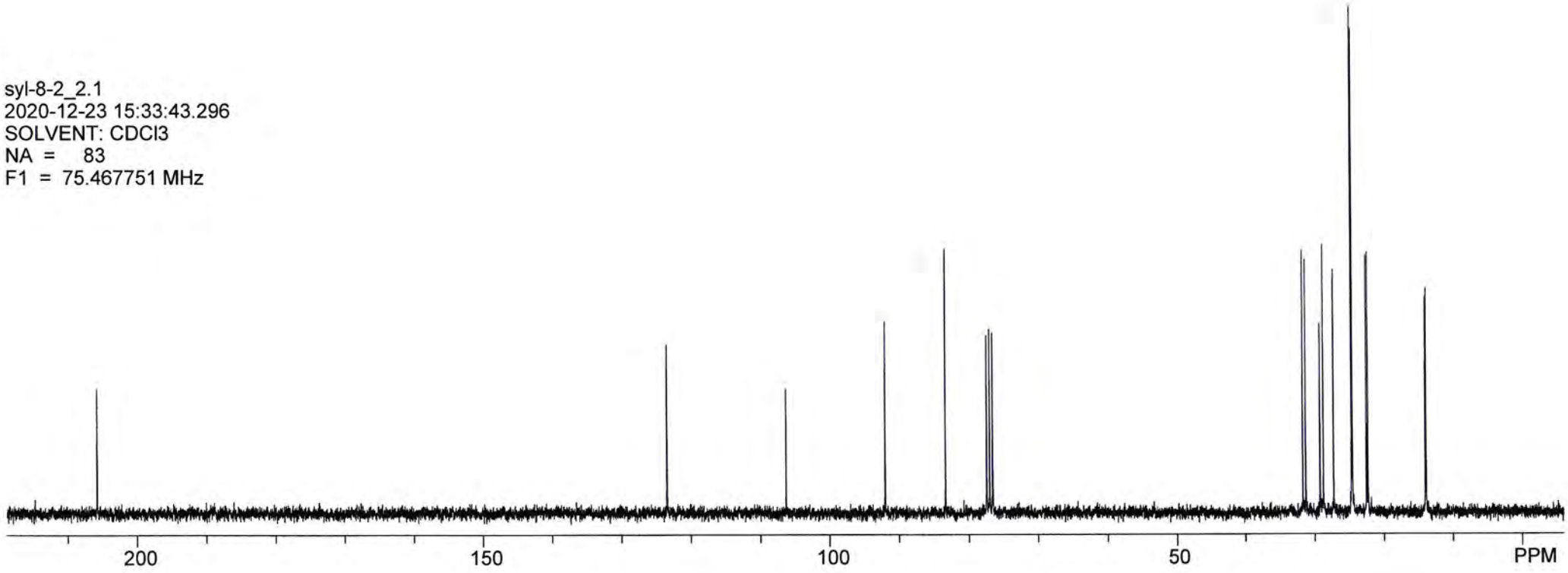

7.273
5.614
5.552
5.243
2.162
2.138
2.119
2.054
2.032
2.008
1.985
1.438
1.417
1.401
1.386
1.362
1.326
1.314
1.303
1.267
0.922
0.899
0.881
0.859
0.000

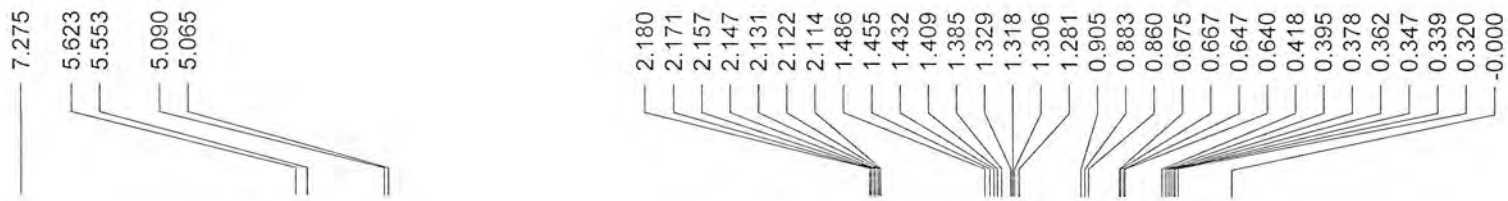

H
Bpin
n-C4H9
n-C5H11

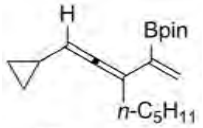
3ba


syl-8-2_1.1
2020-12-23 16:09:51.671
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
24.18
6.37
1.97
2.00
0.92
0.92
0.96
10
8
6
4
2
0
PPM

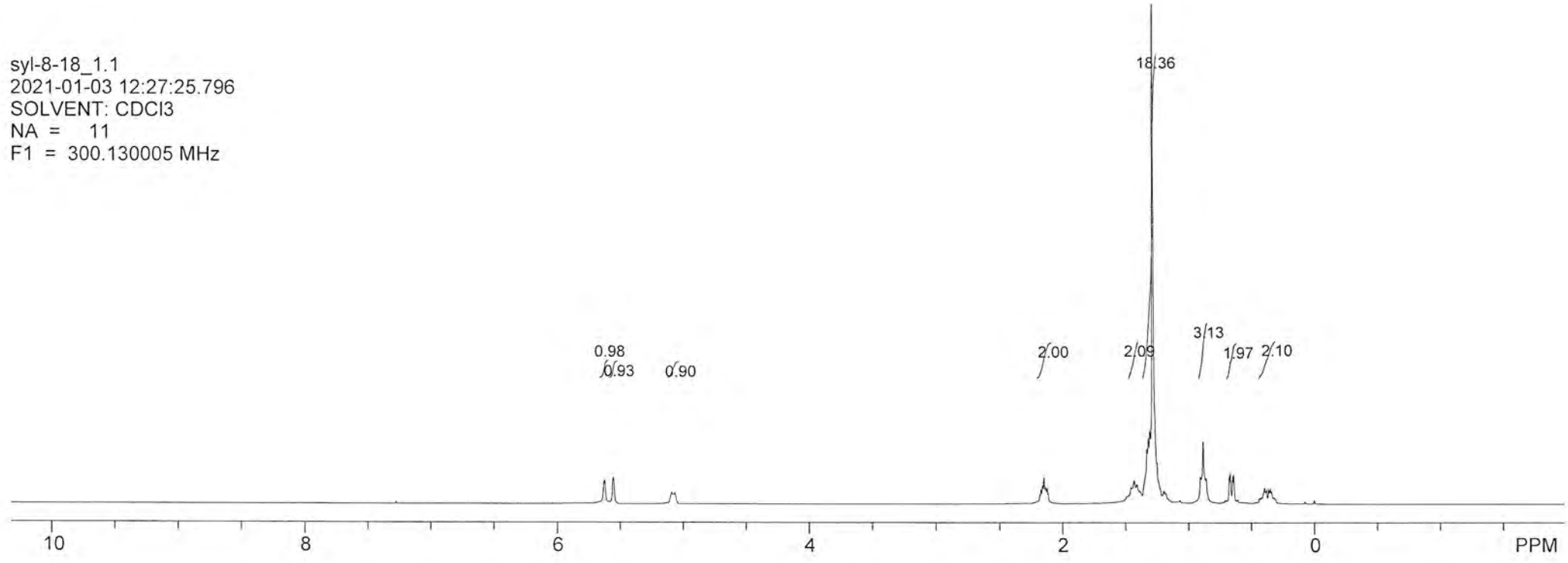

205.777
123.446
106.294
91.983
83.352
77.432
77.000
76.577
31.721
31.298
29.239
28.771
27.281
24.717
24.570
22.557
22.345
14.045
13.907
H
Bpin
n-C4H9
n-C5H11
3ba


syl-8-2_2.1
2020-12-23 15:33:43.296
SOLVENT: CDCl3
NA = 83
F1 = 75.467751 MHz
200
150
100
50
PPM

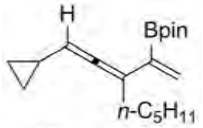

7.275
5.623
5.553
5.090
5.065
2.180
2.171
2.157
2.147
2.131
2.122
2.114
1.486
1.455
1.432
1.409
1.385
1.329
1.318
1.306
1.281
0.905
0.883
0.860
0.675
0.667
0.647
0.640
0.418
0.395
0.378
0.362
0.347
0.339
0.320
-0.000


H
Bpin
n-C5H11

3ca

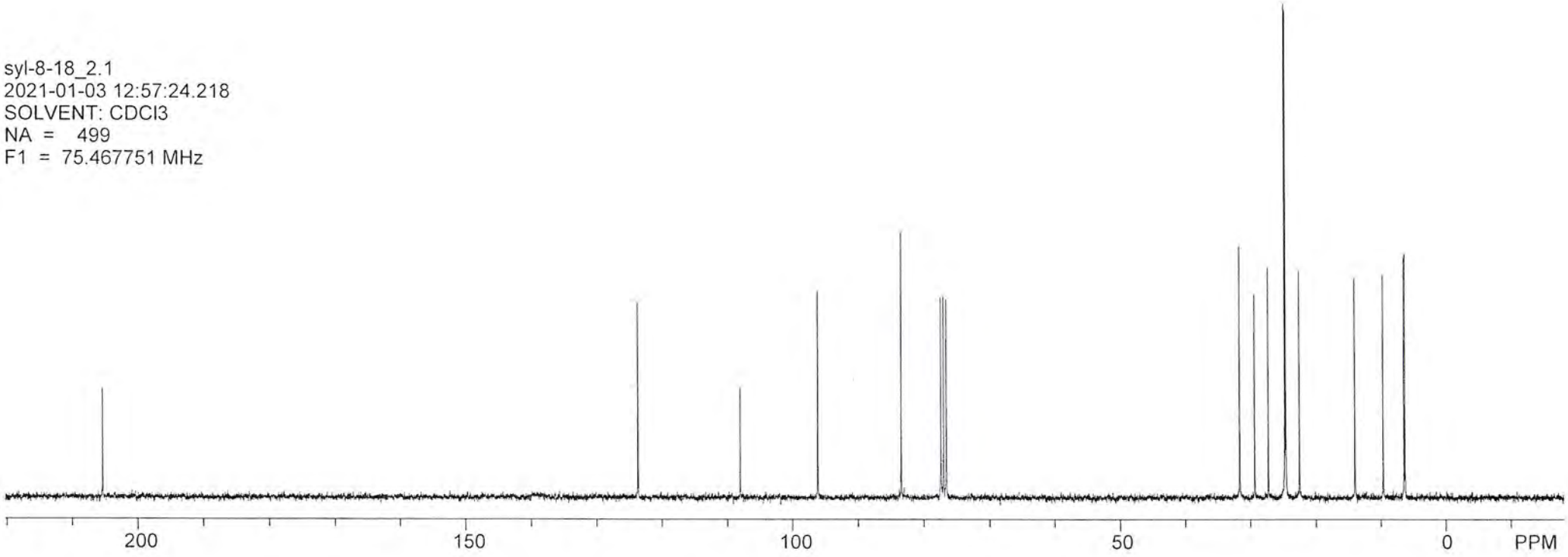

syl-8-18_1.1
2021-01-03 12:27:25.796
SOLVENT: CDCl3
NA = 11
F1 = 300.130005 MHz
18.36
0.98
0.93
0.90
2.00
2.09
3.13
1.97
2.10
10
8
6
4
2
0
PPM

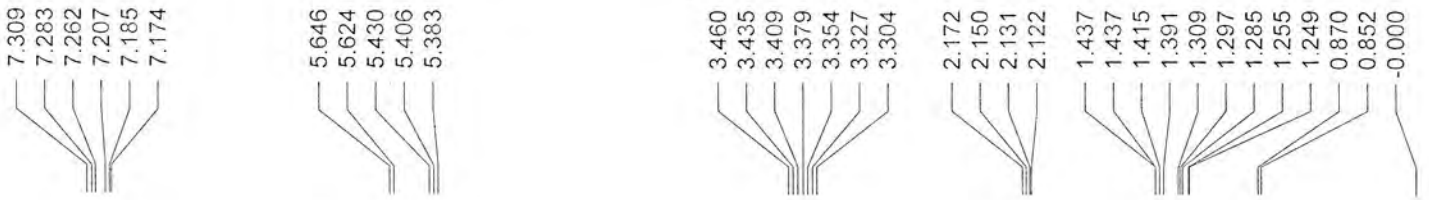
205.373
123.667
108.068
96.165
83.443
77.423
77.000
76.577
31.666
29.368
27.291
24.745
24.597
22.529
14.045
9.698
6.471
6.315

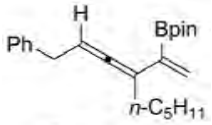


3ca

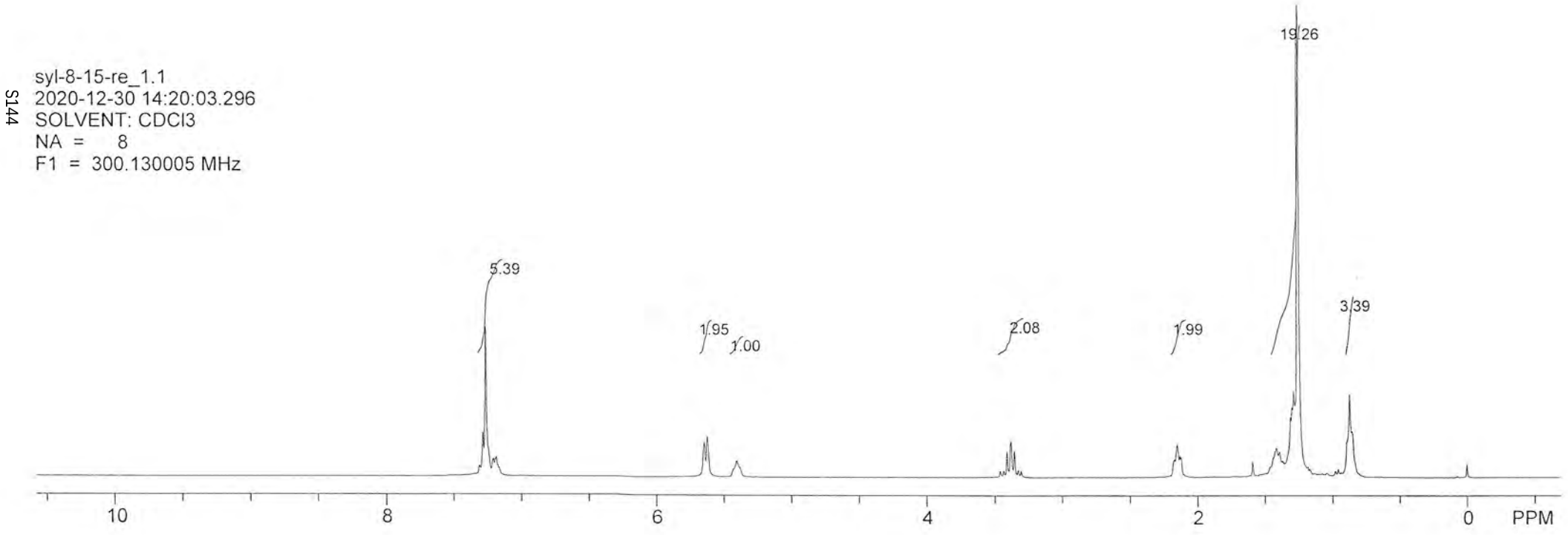
syl-8-18_2.1
2021-01-03 12:57:24.218
SOLVENT: CDCl3
NA = 499
F1 = 75.467751 MHz

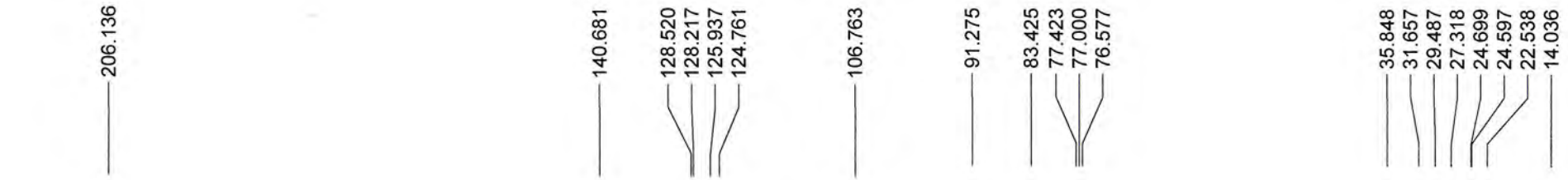


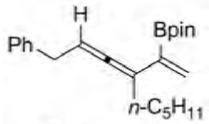

7.309
7.283
7.262
7.207
7.185
7.174
5.646
5.624
5.430
5.406
5.383
3.460
3.435
3.409
3.379
3.354
3.327
3.304
2.172
2.150
2.131
2.122
1.437
1.437
1.415
1.391
1.309
1.297
1.285
1.255
1.249
0.870
0.852
-0.000


H
Ph
Bpin
n-C5H11

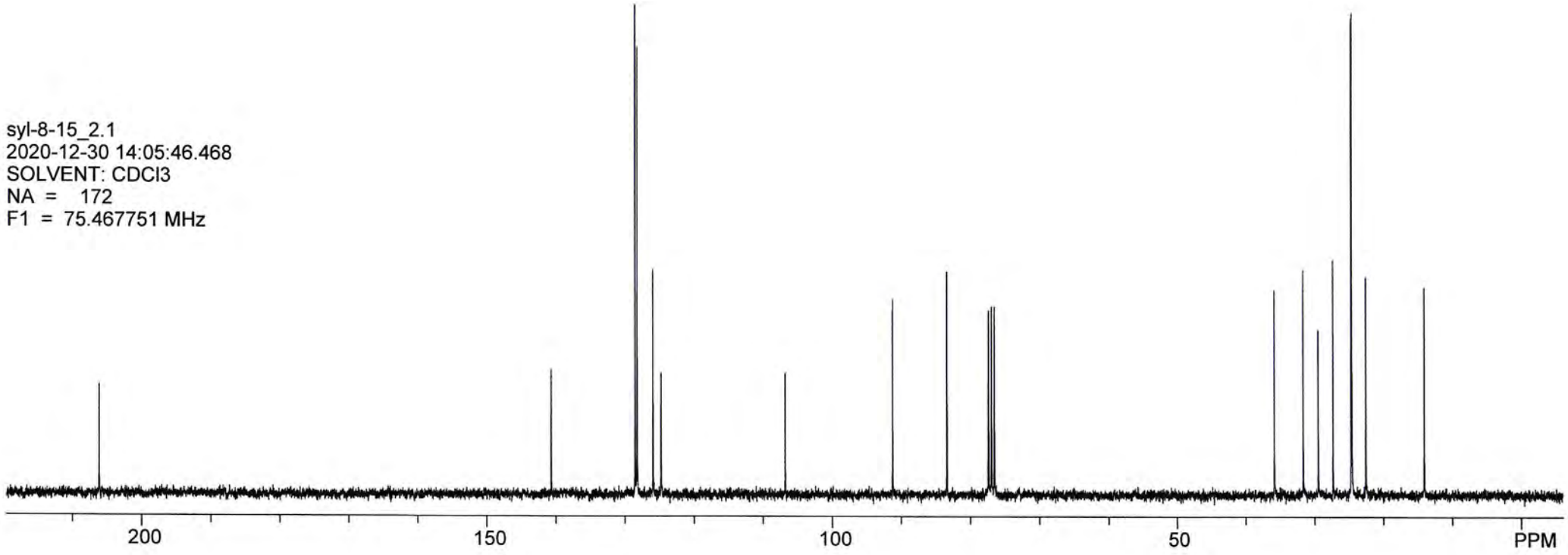
3da

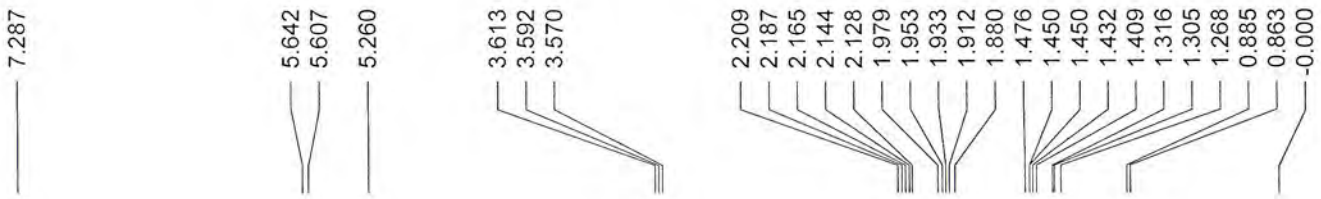

syl-8-15-re_1.1
2020-12-30 14:20:03.296
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
19.26
5.39
1.95
1.00
2.08
1.99
3.39
10
8
6
4
2
0
PPM

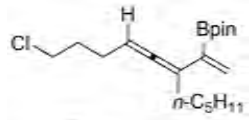

206.136
140.681
128.520
128.217
125.937
124.761
106.763
91.275
83.425
77.423
77.000
76.577
35.848
31.657
29.487
27.318
24.699
24.597
22.538
14.036


H
Ph
Bpin
n-C5H11

3da

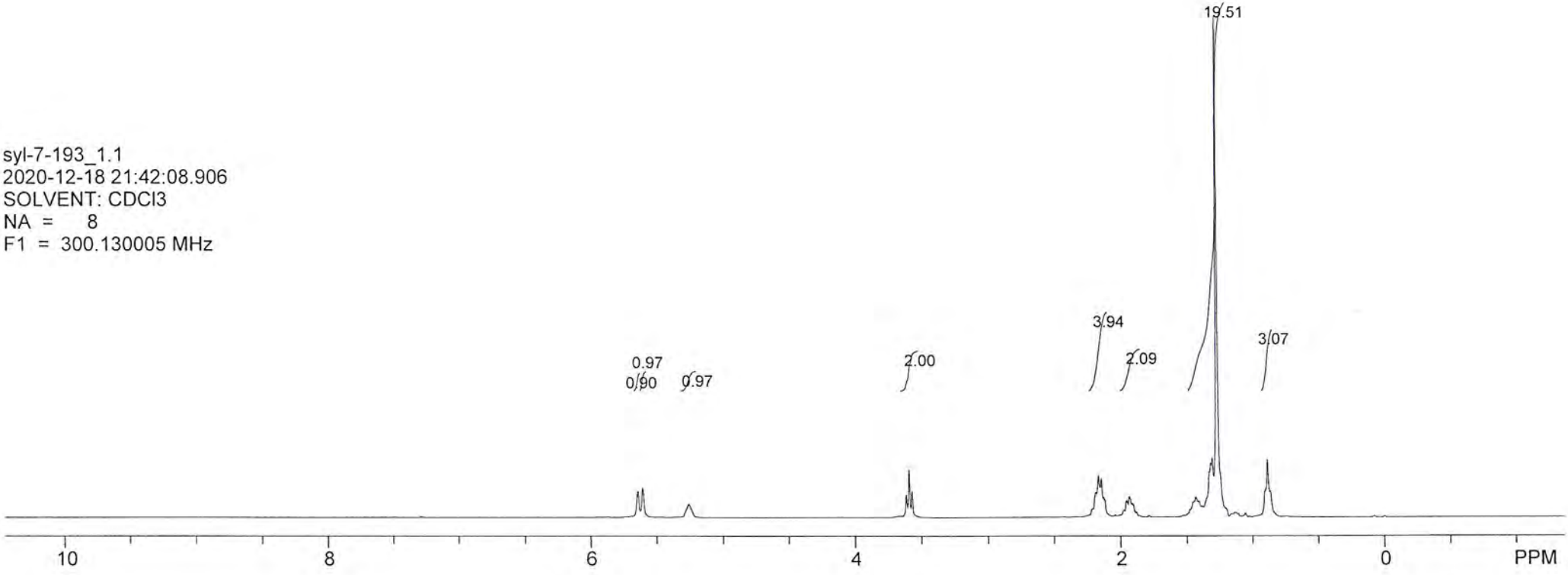

syl-8-15_2.1
2020-12-30 14:05:46.468
SOLVENT: CDCl3
NA = 172
F1 = 75.467751 MHz
200
150
100
50
PPM

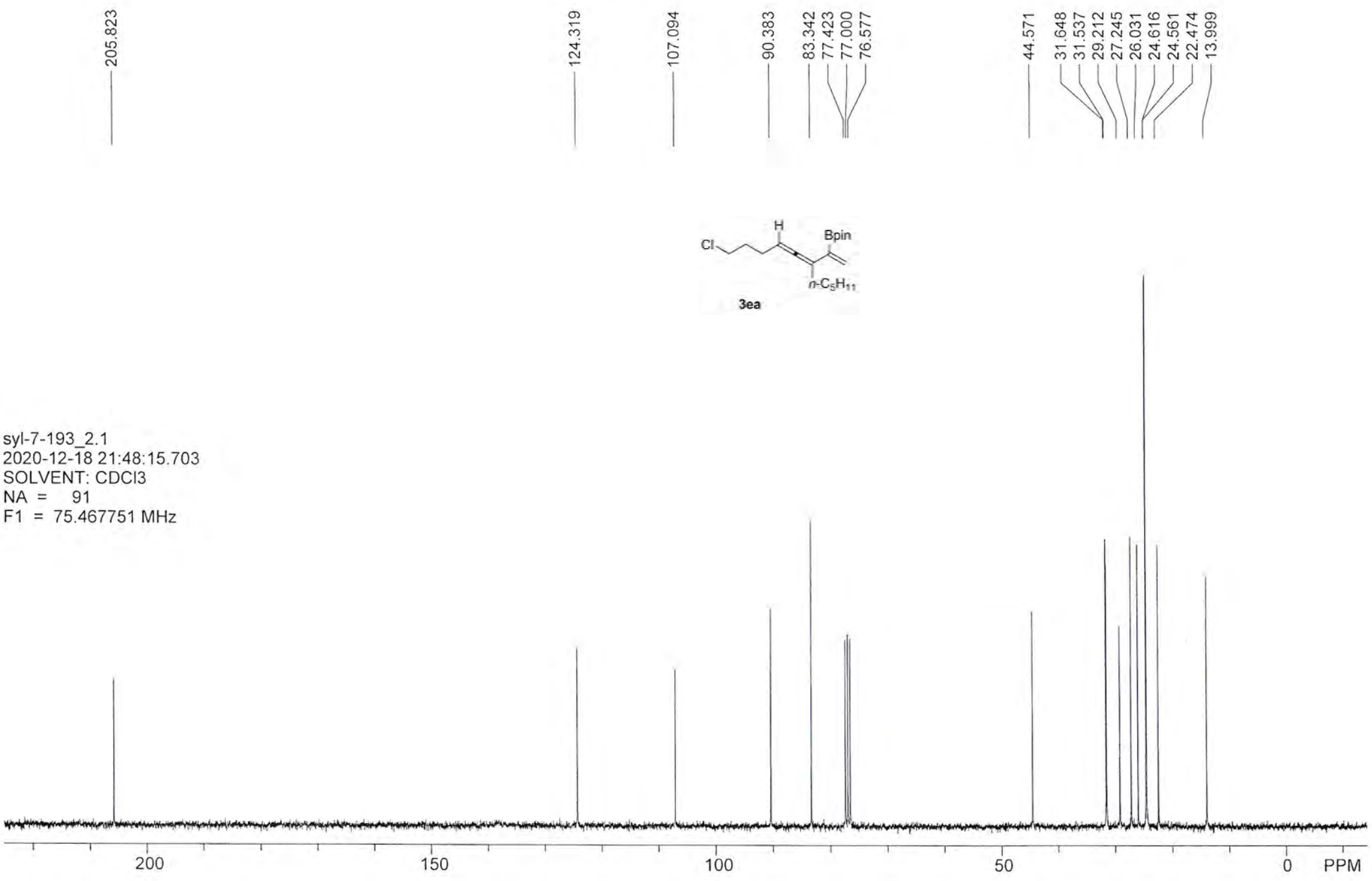


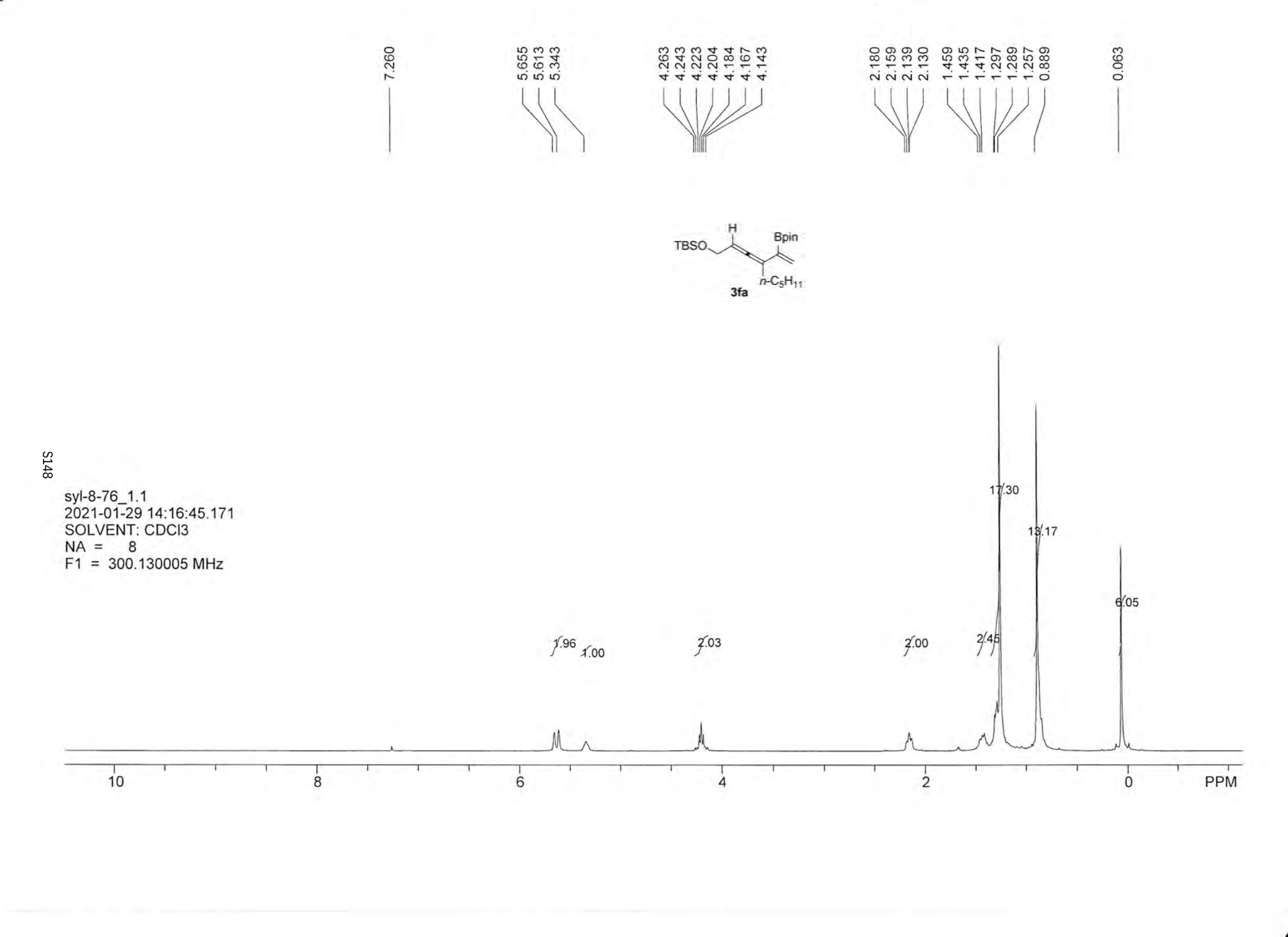


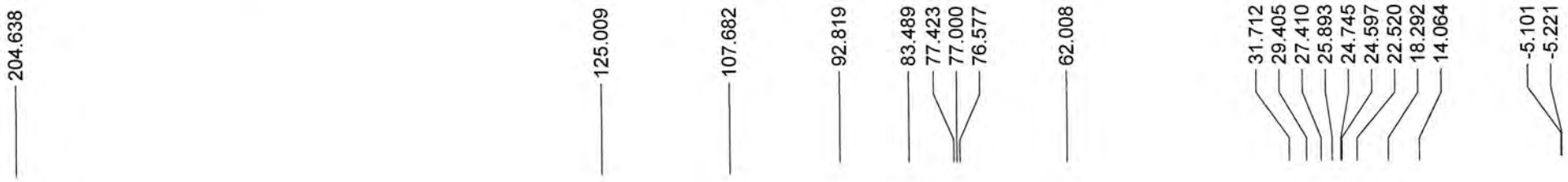
3ea

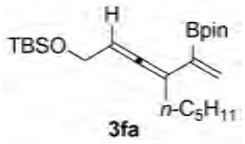
syl-7-193_1.1
2020-12-18 21:42:08.906
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz



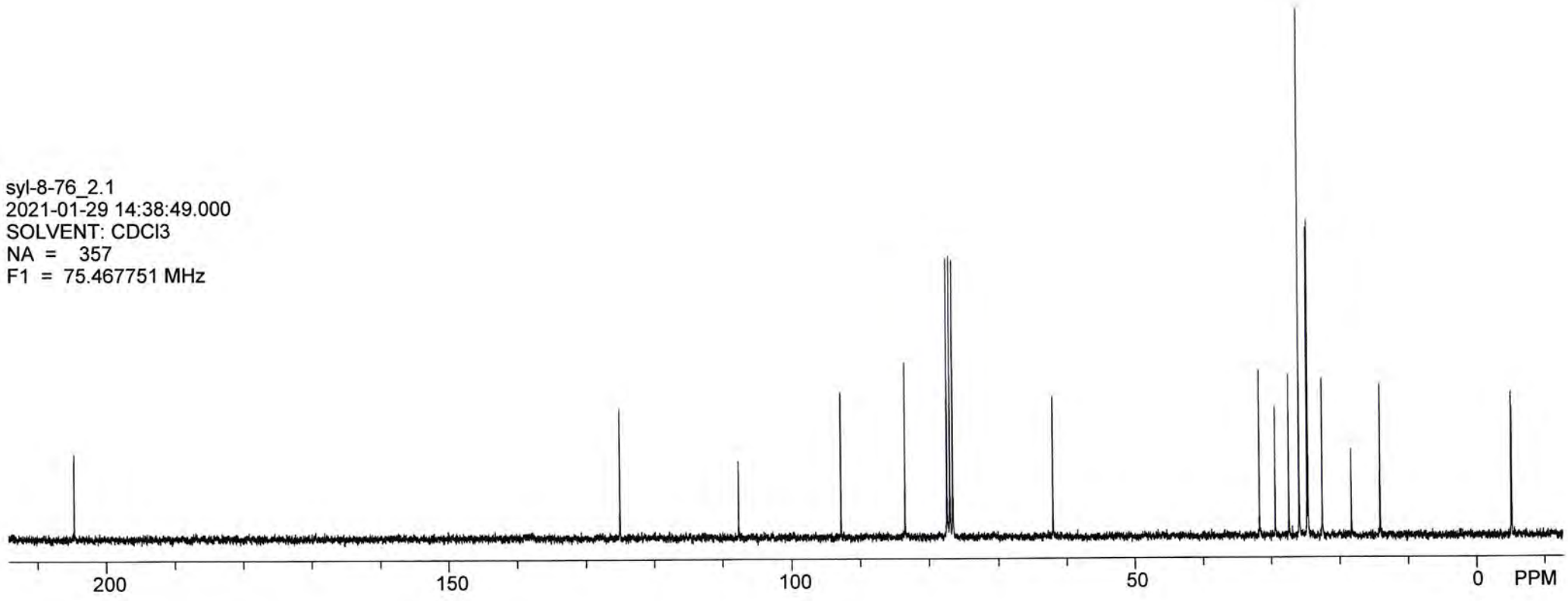

205.823
124.319
107.094
90.383
83.342
77.423
77.000
76.577
44.571
31.648
31.537
29.212
27.245
26.031
24.616
24.561
22.474
13.999
Cl
H
Bpin
n-C5H11
3ea
syl-7-193_2.1
2020-12-18 21:48:15.703
SOLVENT: CDCl3
NA = 91
F1 = 75.467751 MHz
200
150
100
50
0
PPM

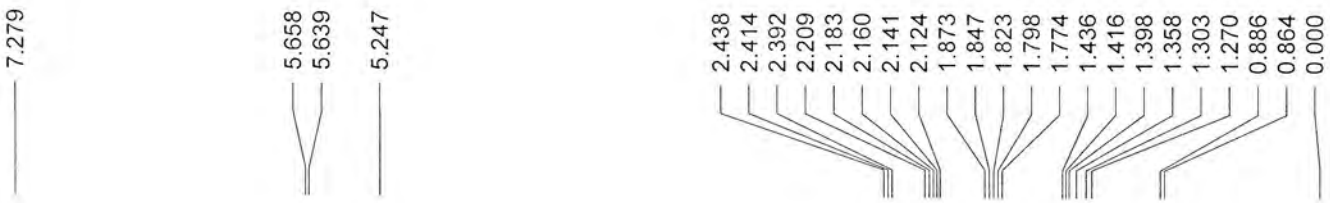

7.260
5.655
5.613
5.343
4.263
4.243
4.223
4.204
4.184
4.167
4.143
2.180
2.159
2.139
2.130
1.459
1.435
1.417
1.297
1.289
1.257
0.889
0.063
TBSO
H
Bpin
n-C5H11
3fa
syl-8-76_1.1
2021-01-29 14:16:45.171
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
1.96
1.00
2.03
2.00
2.45
17.30
13.17
6.05
10
8
6
4
2
0
PPM

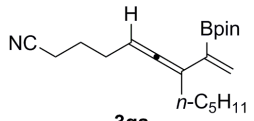

204.638
125.009
107.682
92.819
83.489
77.423
77.000
76.577
62.008
31.712
29.405
27.410
25.893
24.745
24.597
22.520
18.292
14.064
-5.101
-5.221


H
TBSO
Bpin
n-C5H11

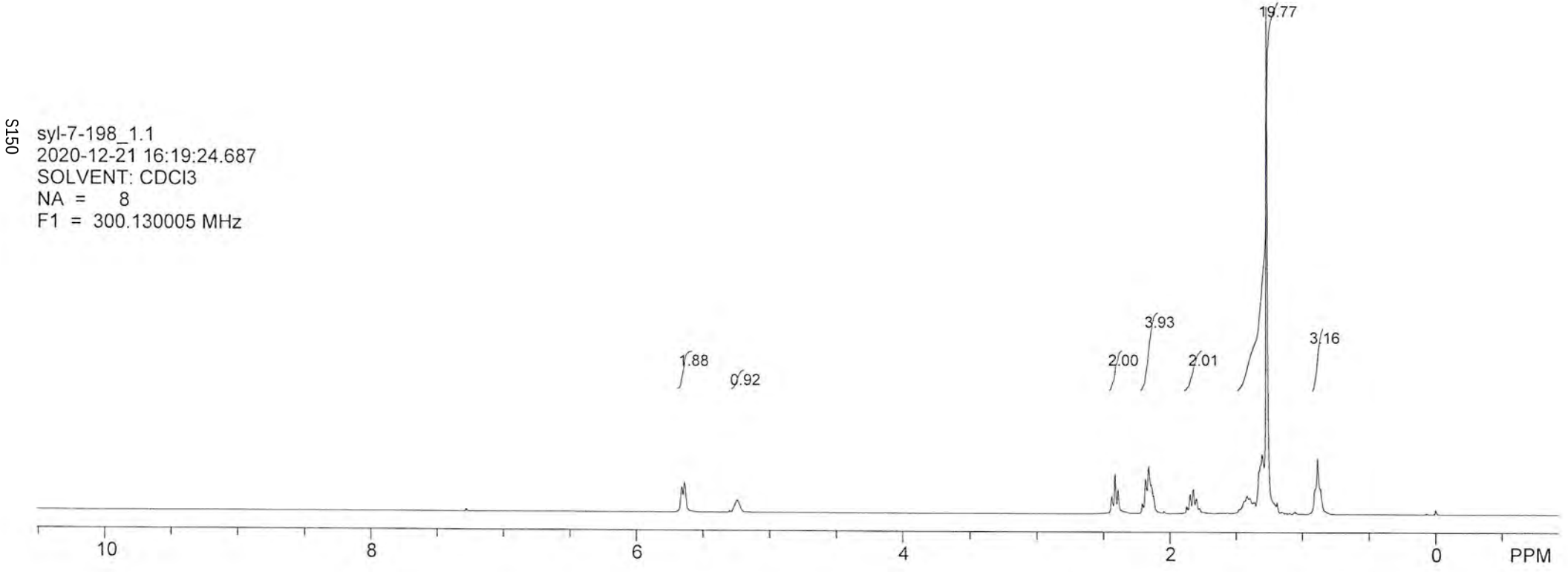
3fa

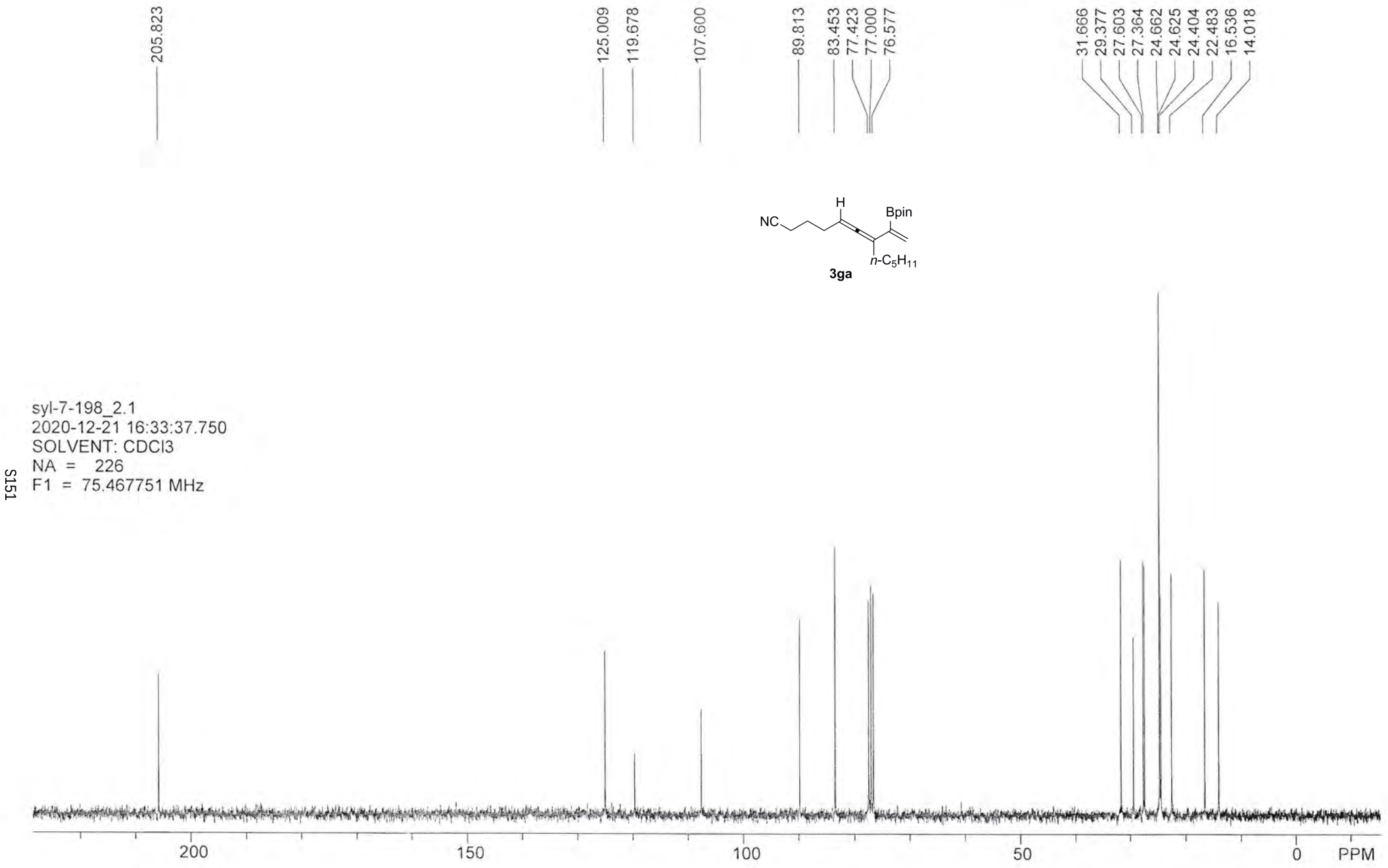

syl-8-76_2.1
2021-01-29 14:38:49.000
SOLVENT: CDCl3
NA = 357
F1 = 75.467751 MHz
200
150
100
50
0
PPM

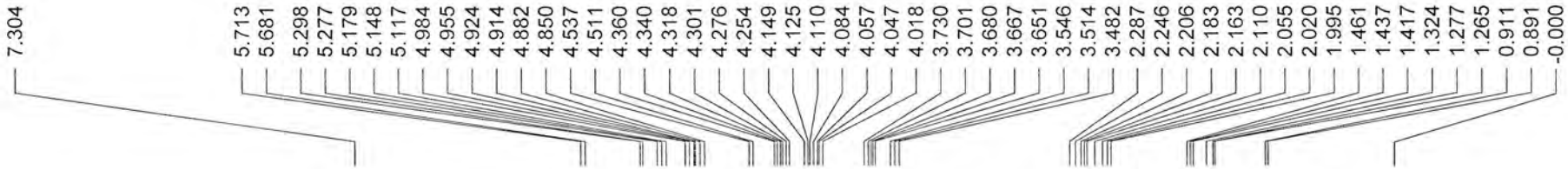

7.279
5.658
5.639
5.247
2.438
2.414
2.392
2.209
2.183
2.160
2.141
2.124
1.873
1.847
1.823
1.798
1.774
1.436
1.416
1.398
1.358
1.303
1.270
0.886
0.864
0.000

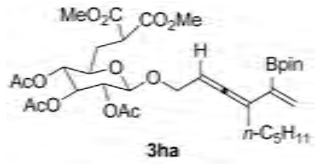

H
NC
Bpin
n-C5H11

3ga

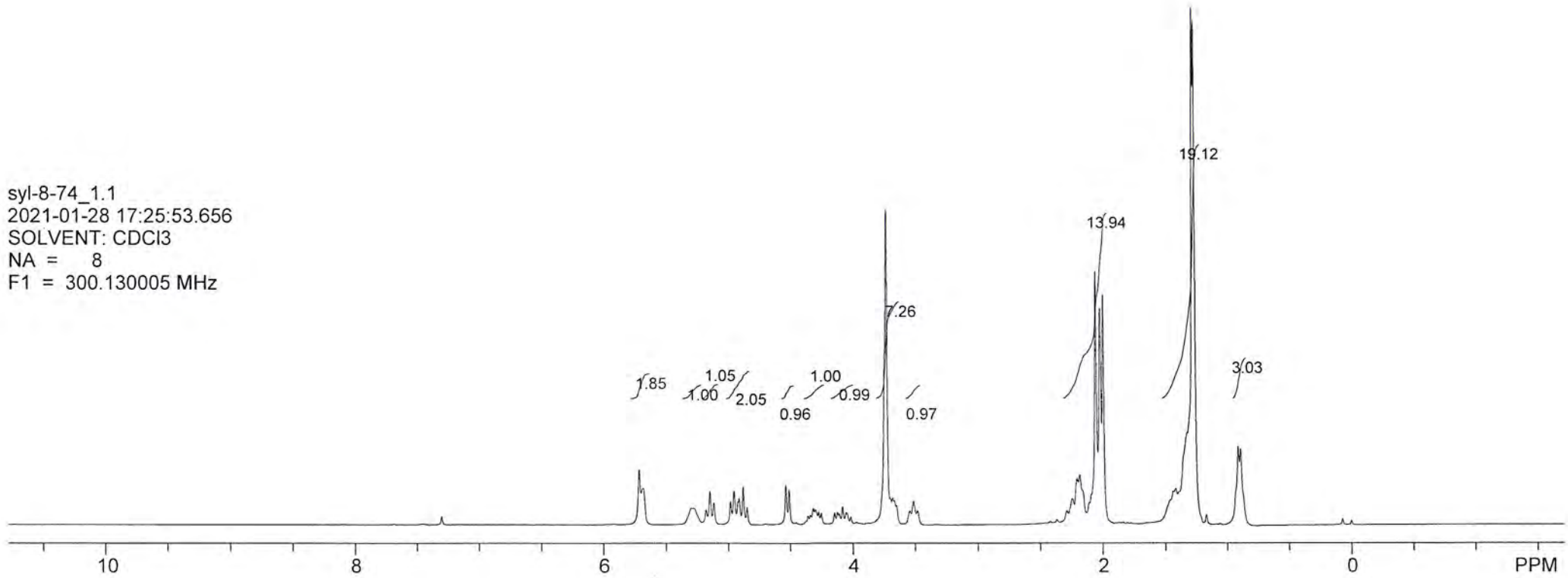

syl-7-198_1.1
2020-12-21 16:19:24.687
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
19.77
3.93
3.16
1.88
0.92
2.00
2.01
10
8
6
4
2
0
PPM


205.823
125.009
119.678
107.600
89.813
83.453
77.423
77.000
76.577
31.666
29.377
27.603
27.364
24.662
24.625
24.404
22.483
16.536
14.018
H
NC
Bpin
n-C5H11
3ga
syl-7-198_2.1
2020-12-21 16:33:37.750
SOLVENT: CDCl3
NA = 226
F1 = 75.467751 MHz
200
150
100
50
0
PPM

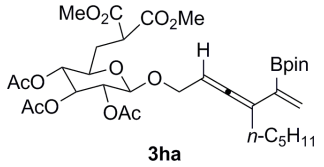

7.304
5.713
5.681
5.298
5.277
5.179
5.148
5.117
4.984
4.955
4.924
4.914
4.882
4.850
4.537
4.511
4.360
4.340
4.318
4.301
4.276
4.254
4.149
4.125
4.110
4.084
4.057
4.047
4.018
3.730
3.701
3.680
3.667
3.651
3.546
3.514
3.482
2.287
2.246
2.206
2.183
2.163
2.110
2.055
2.020
1.995
1.461
1.437
1.417
1.324
1.277
1.265
0.911
0.891
-0.000


MeO2C
CO2Me
AcO
AcO
OAc
O
O
H
Bpin
n-C5H11

3ha

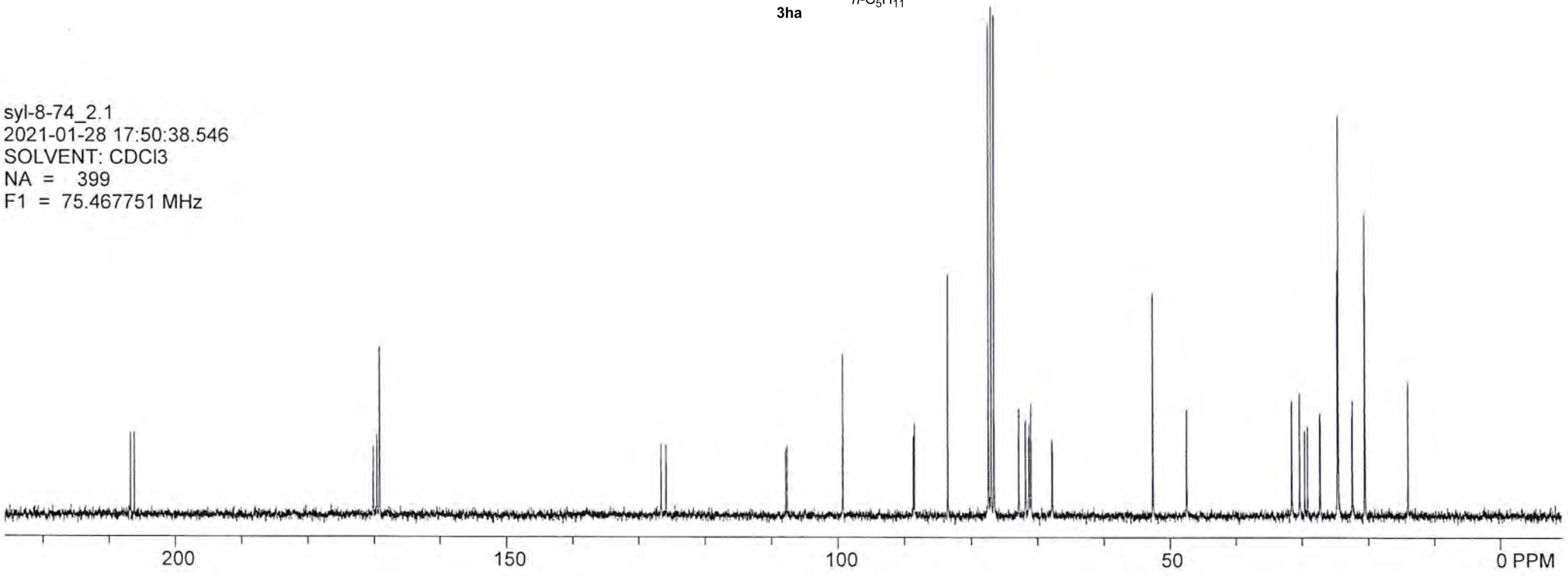

syl-8-74_1.1
2021-01-28 17:25:53.656
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
1.85
1.05
1.00
2.05
0.96
1.00
0.99
7.26
0.97
13.94
19.12
3.03
10
8
6
4
2
0
PPM

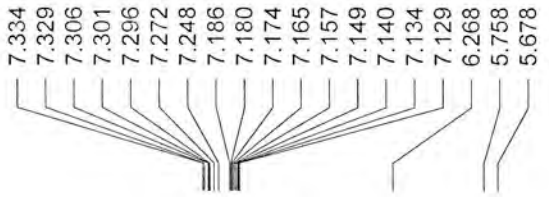
206.761
206.191
170.141
170.104
169.589
169.571
169.194
169.166
169.130
126.608
125.864
107.839
107.646
88.637
88.471
99.263
83.517
77.423
77.000
76.577
72.845
72.818
71.825
71.779
71.319
71.218
70.989
70.970
67.817
67.744
52.596
52.550
47.476
47.439
31.565
31.491
30.333
29.570
29.138
27.318
27.245
24.671
24.634
24.524
22.474
22.391
20.562
20.489
14.008
13.981

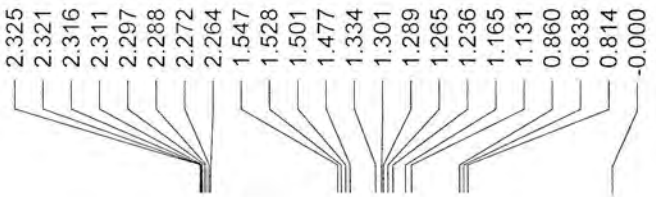


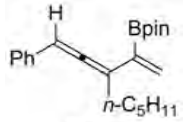
3ha

syl-8-74_2.1
2021-01-28 17:50:38.546
SOLVENT: CDCl3
NA = 399
F1 = 75.467751 MHz



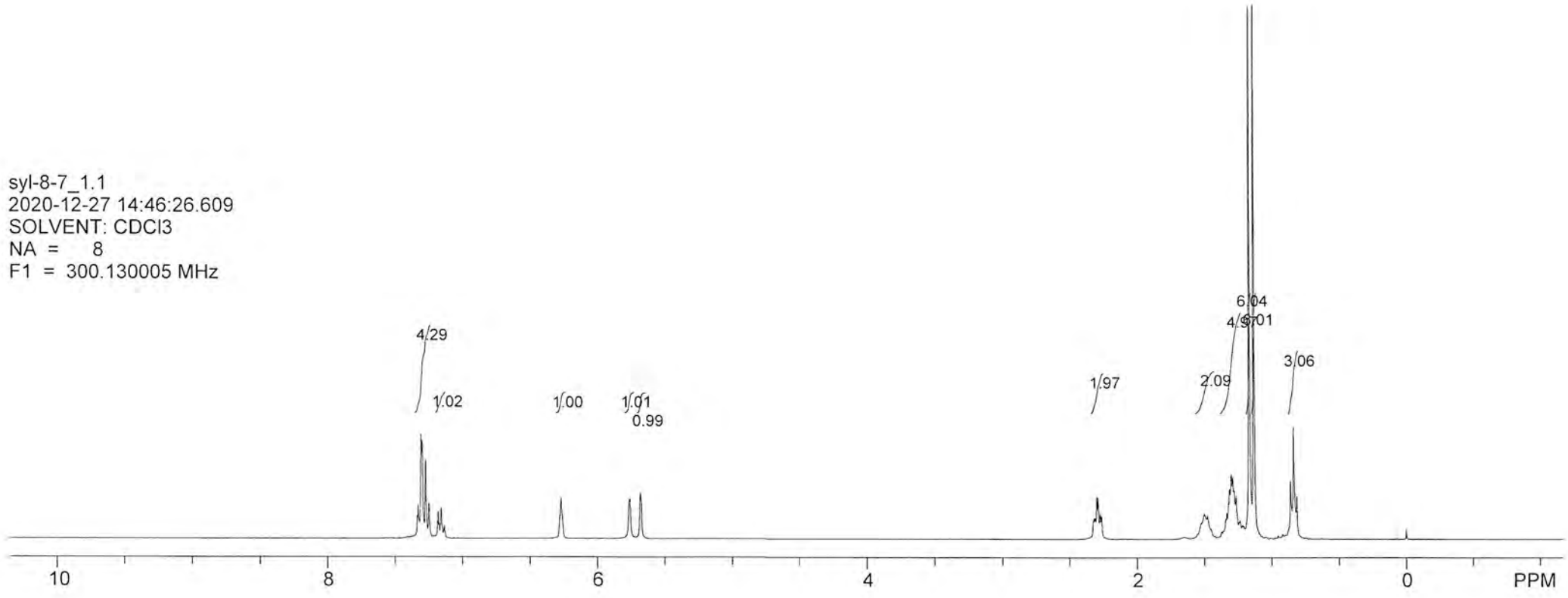


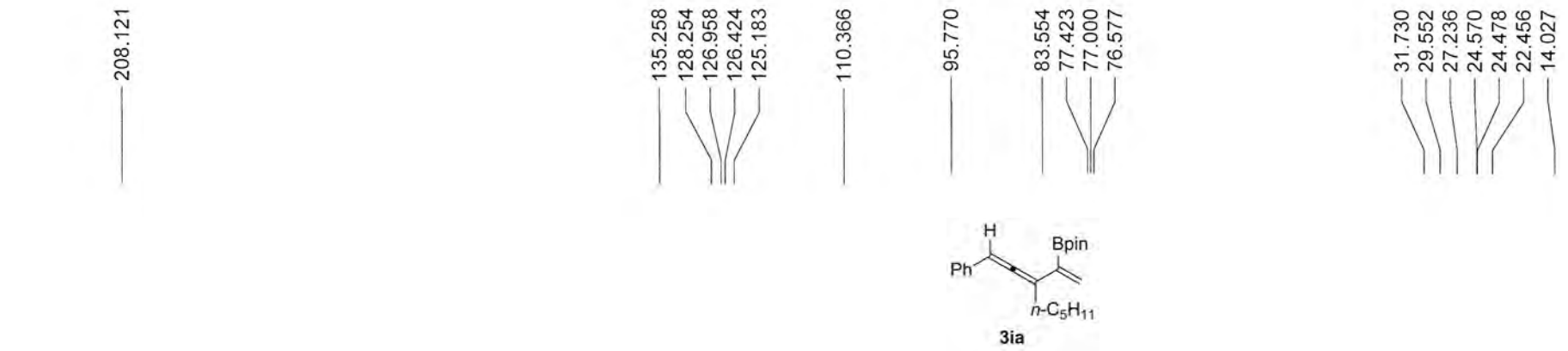




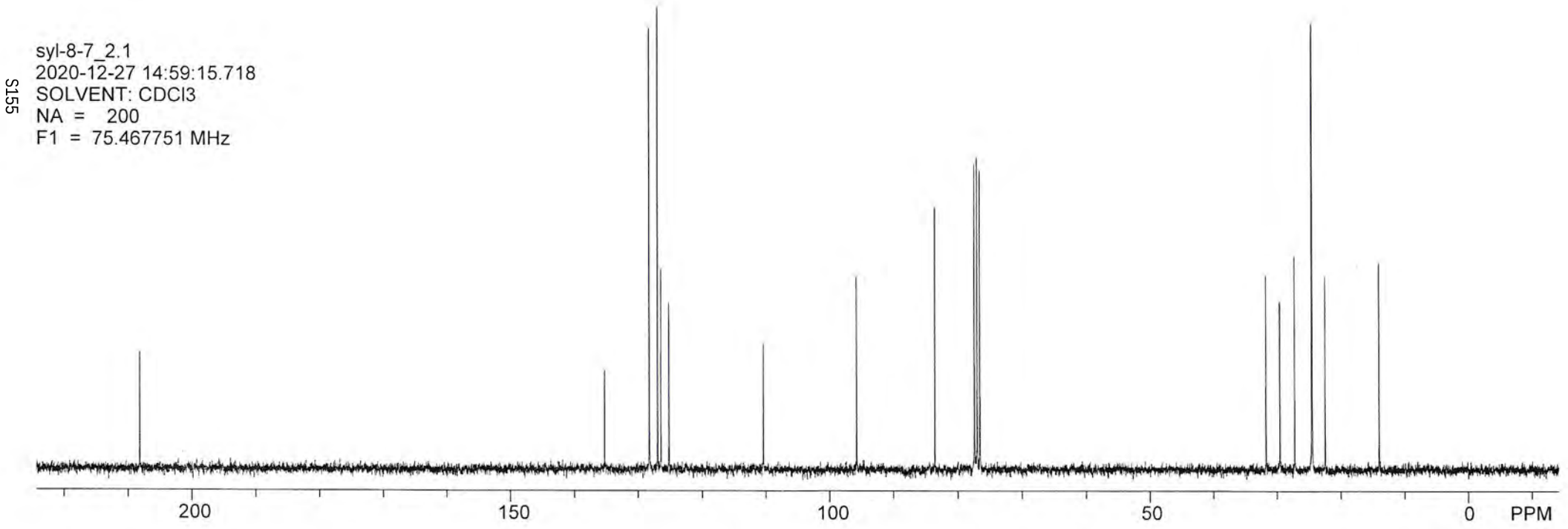
3ia

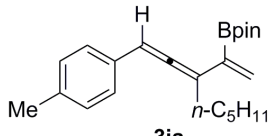
syl-8-7_1.1
2020-12-27 14:46:26.609
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz




208.121
135.258
128.254
126.958
126.424
125.183
110.366
95.770
83.554
77.423
77.000
76.577
31.730
29.552
27.236
24.570
24.478
22.456
14.027
H
Bpin
Ph
n-C5H11

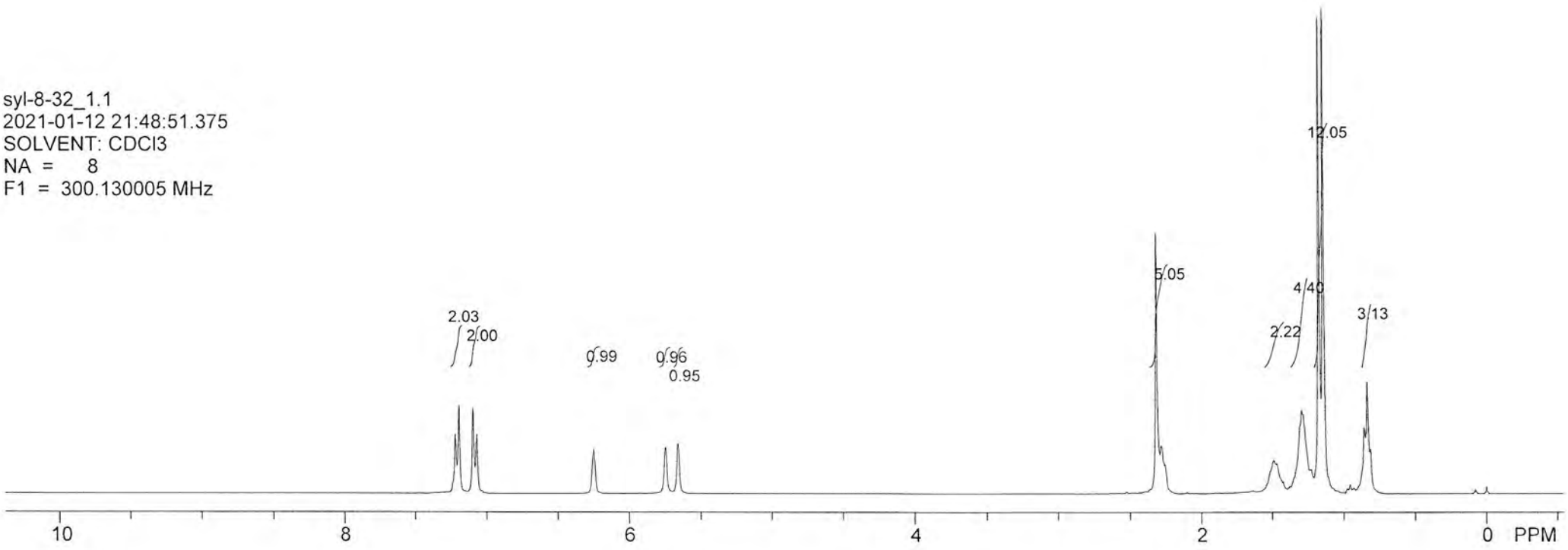
3ia

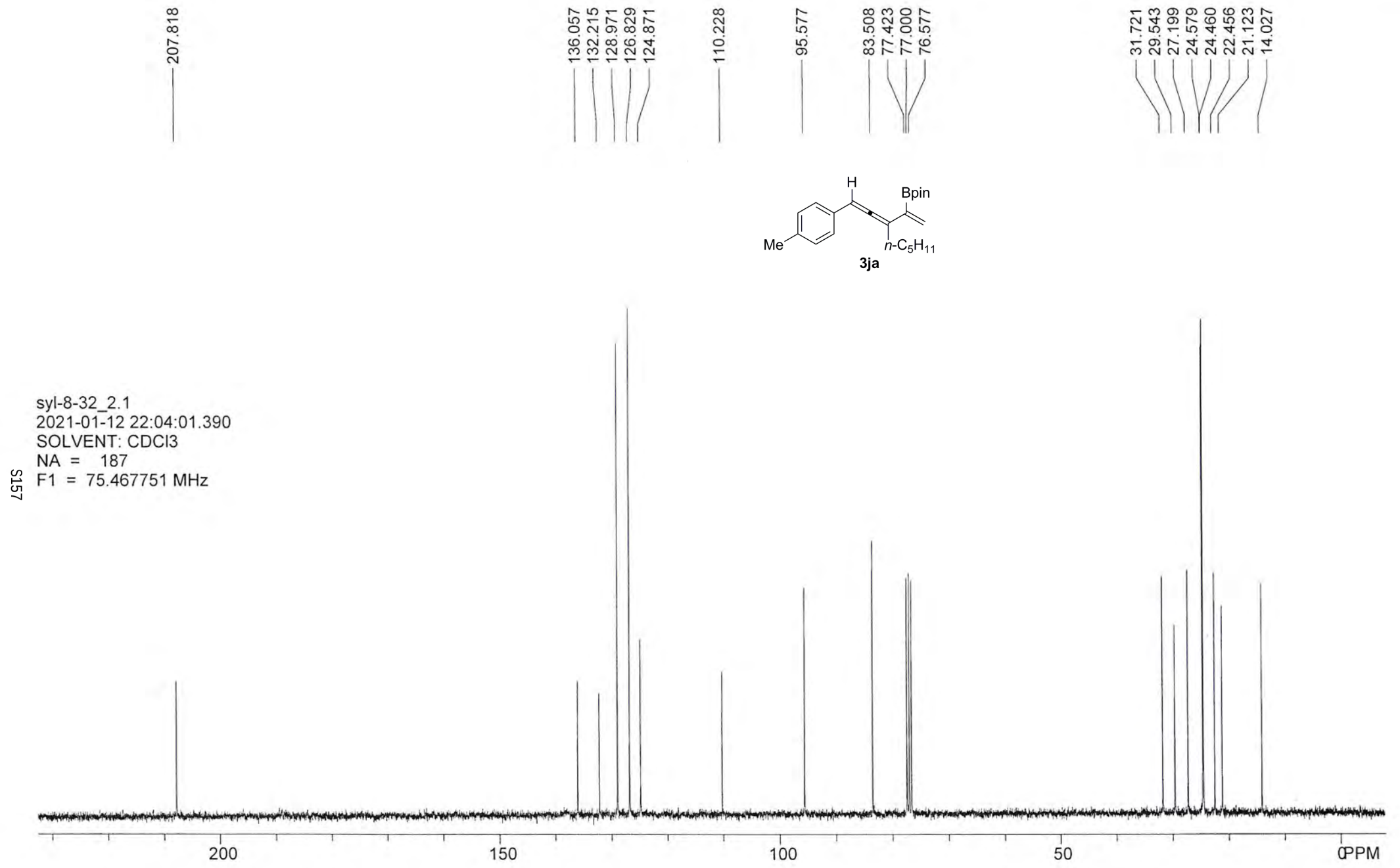

syl-8-7_2.1
2020-12-27 14:59:15.718
SOLVENT: CDCl3
NA = 200
F1 = 75.467751 MHz
200
150
100
50
0
PPM

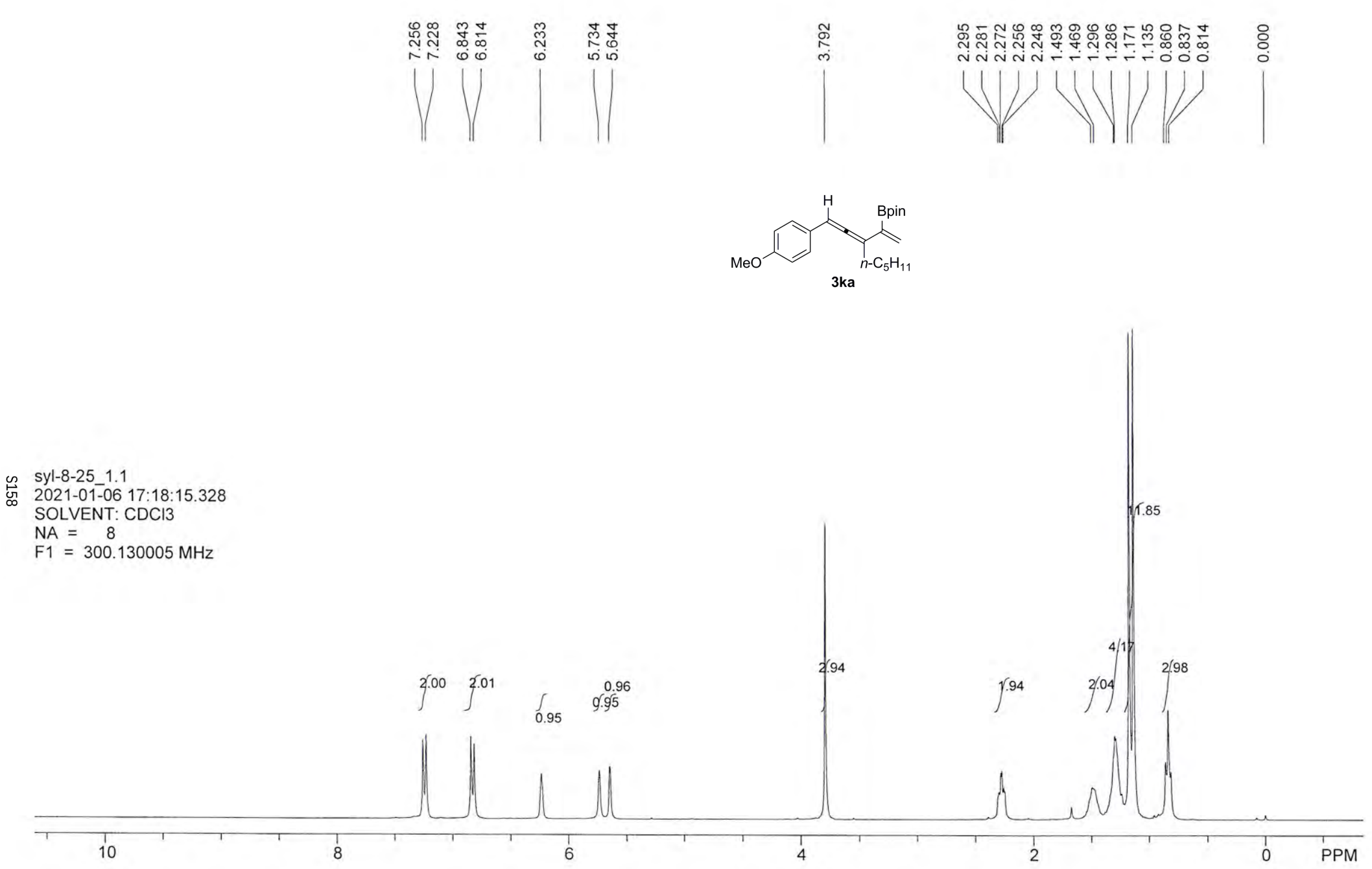


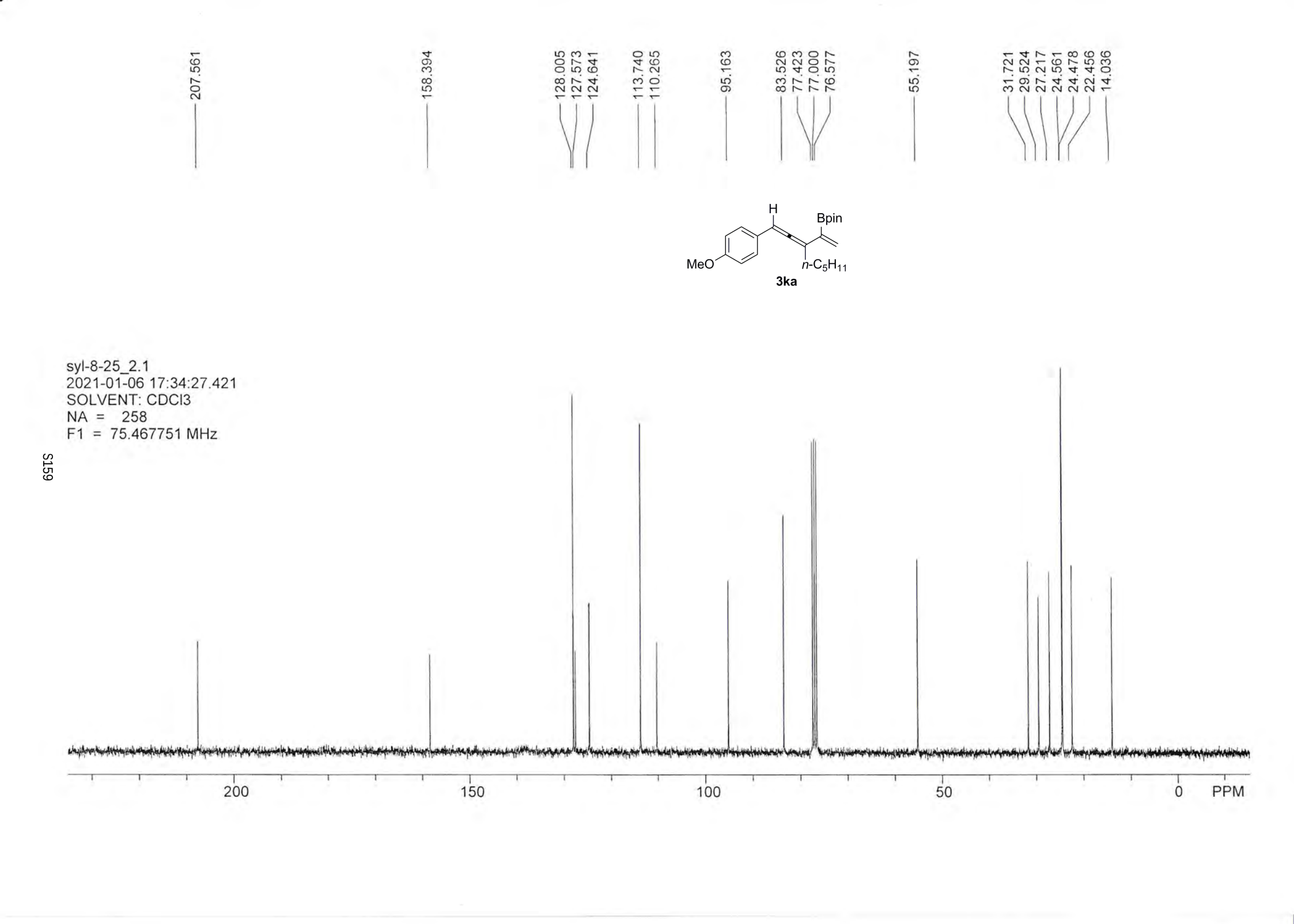
3ja

syl-8-32_1.1
2021-01-12 21:48:51.375
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz

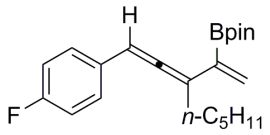



207.818
136.057
132.215
128.971
126.829
124.871
110.228
95.577
83.508
77.423
77.000
76.577
31.721
29.543
27.199
24.579
24.460
22.456
21.123
14.027
H
Bpin
Me
n-C5H11
3ja
syl-8-32_2.1
2021-01-12 22:04:01.390
SOLVENT: CDCl3
NA = 187
F1 = 75.467751 MHz
200
150
100
50
0
PPM

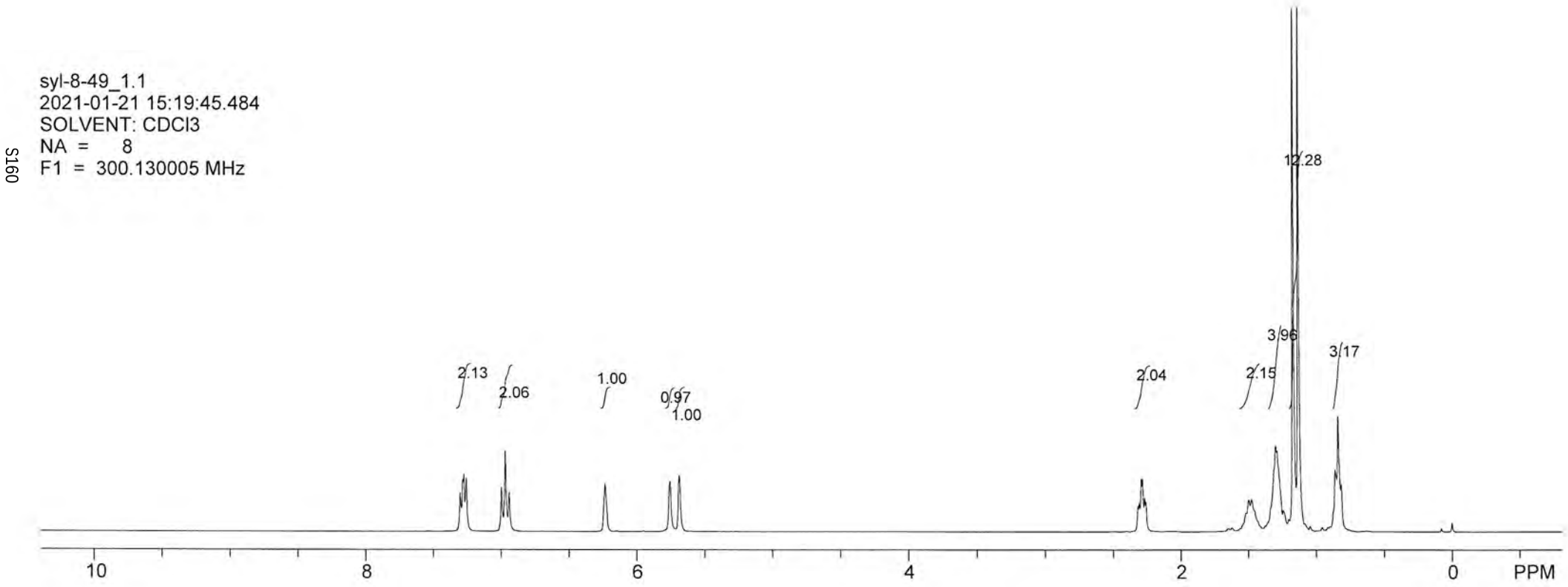

7.256
7.228
6.843
6.814
6.233
5.734
5.644
3.792
2.295
2.281
2.272
2.256
2.248
1.493
1.469
1.296
1.286
1.171
1.135
0.860
0.837
0.814
0.000
H
Bpin
MeO
n-C5H11
3ka
syl-8-25_1.1
2021-01-06 17:18:15.328
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
2.00
2.01
0.95
0.96
0.95
2.94
1.94
2.04
4.17
11.85
2.98
10
8
6
4
2
0
PPM

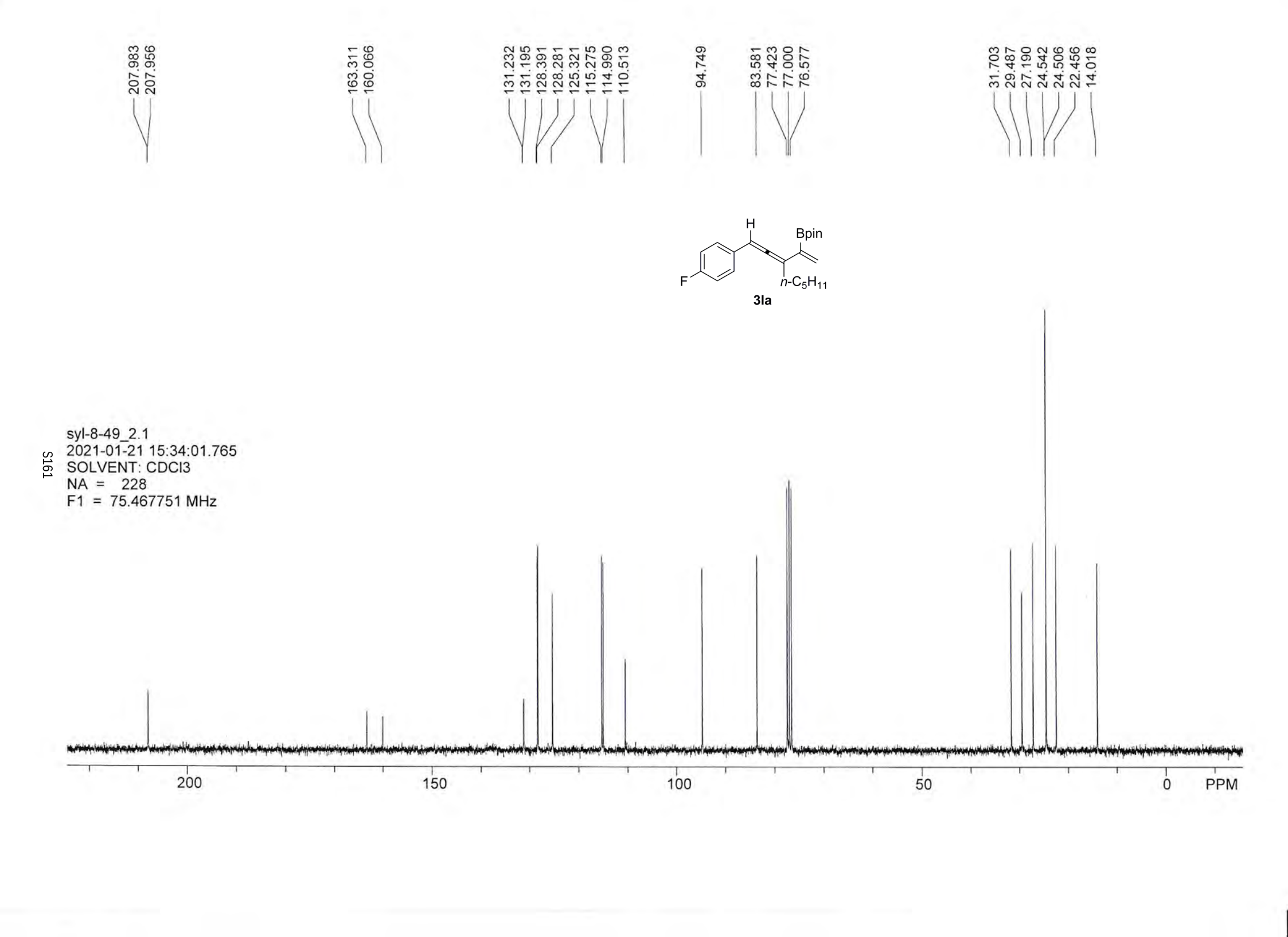

207.561
158.394
128.005
127.573
124.641
113.740
110.265
95.163
83.526
77.423
77.000
76.577
55.197
31.721
29.524
27.217
24.561
24.478
22.456
14.036
H
Bpin
MeO
n-C5H11
3ka
syl-8-25_2.1
2021-01-06 17:34:27.421
SOLVENT: CDCl3
NA = 258
F1 = 75.467751 MHz
200
150
100
50
0
PPM

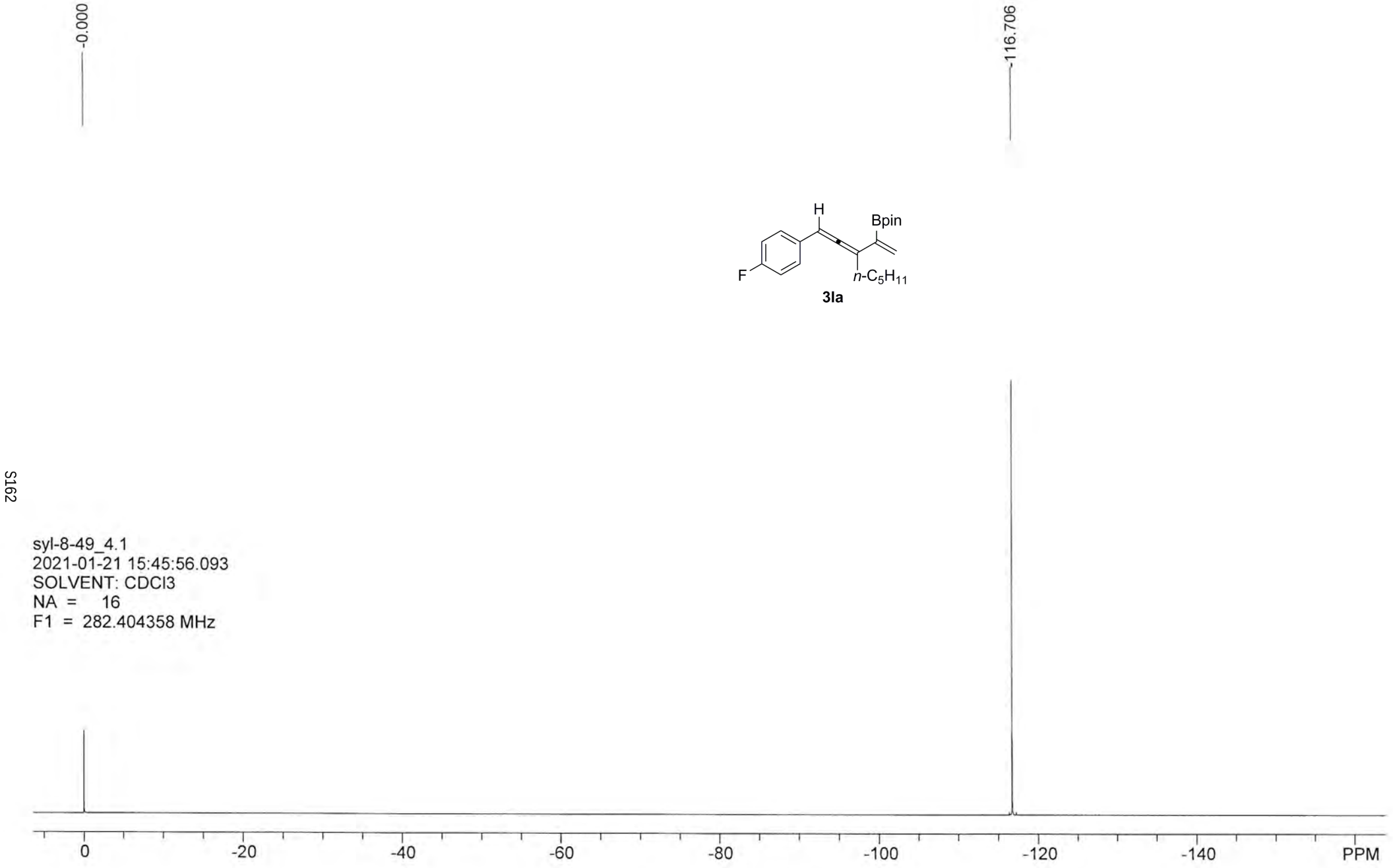

7.302
7.274
7.256
6.997
6.968
6.939
6.233
5.756
5.687
2.316
2.306
2.291
2.282
2.266
2.256
1.518
1.495
1.476
1.297
1.287
1.167
1.128
0.858
0.836
0.814
-0.000

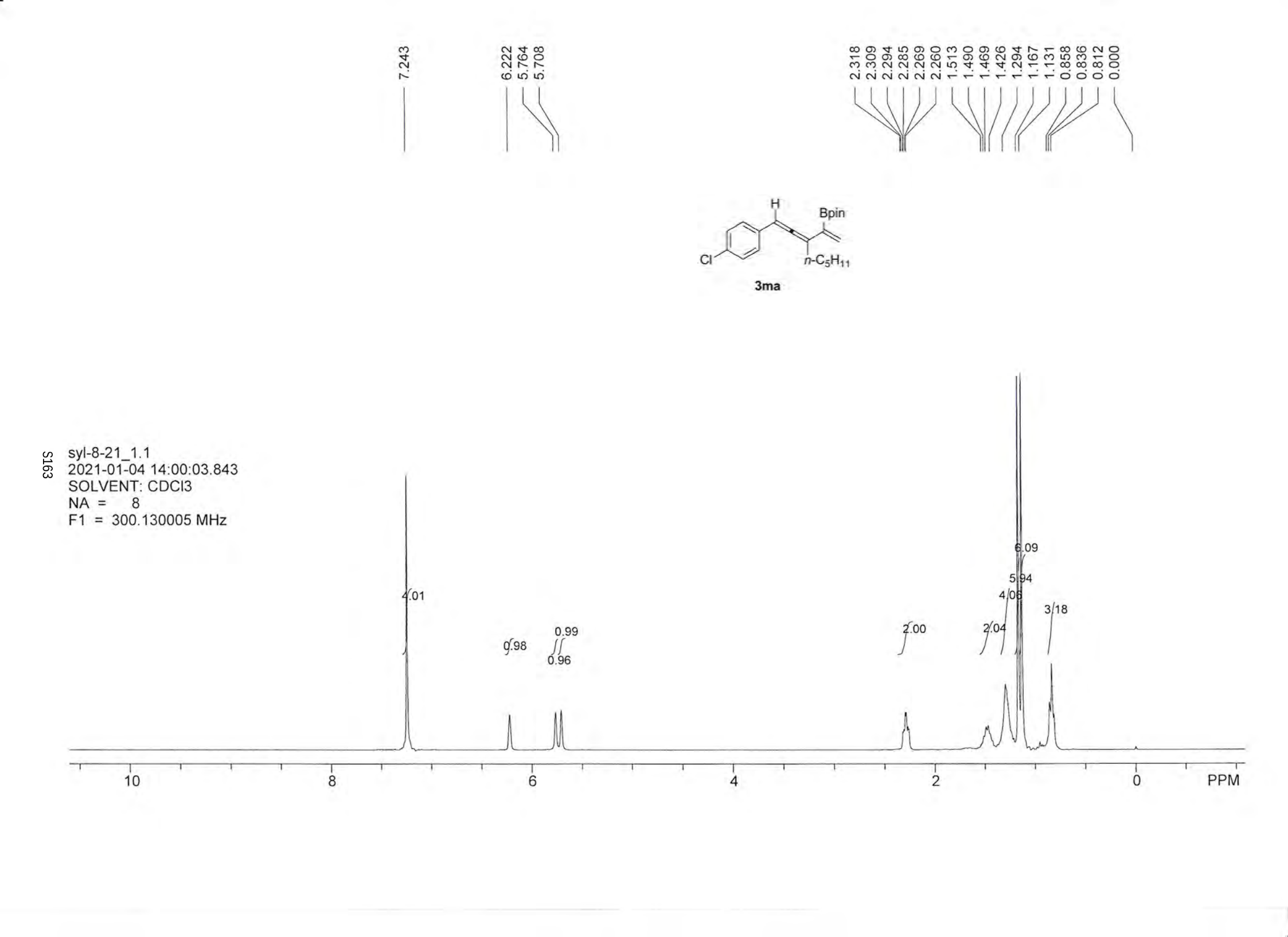

H
Bpin
F
n-C5H11

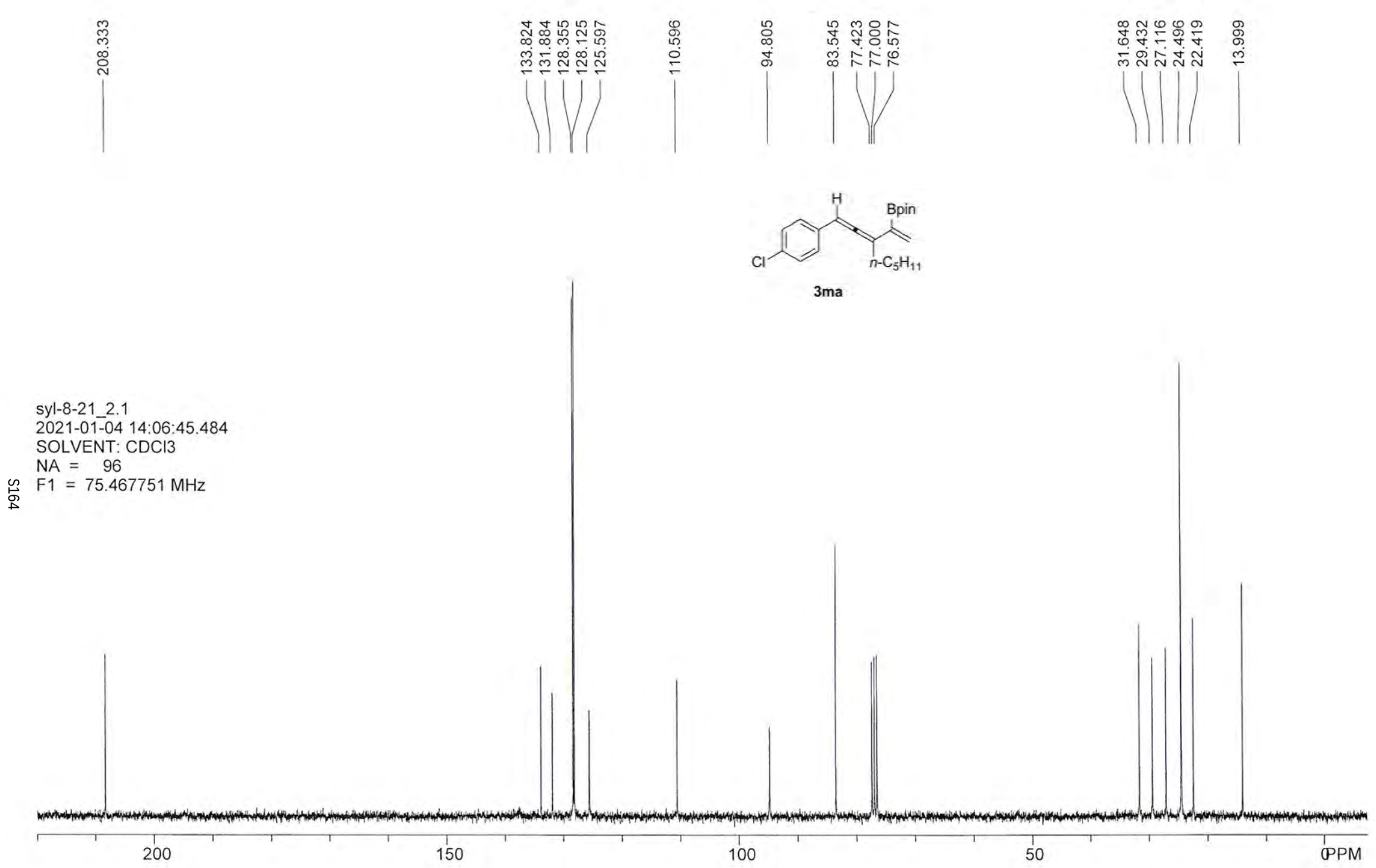
3la

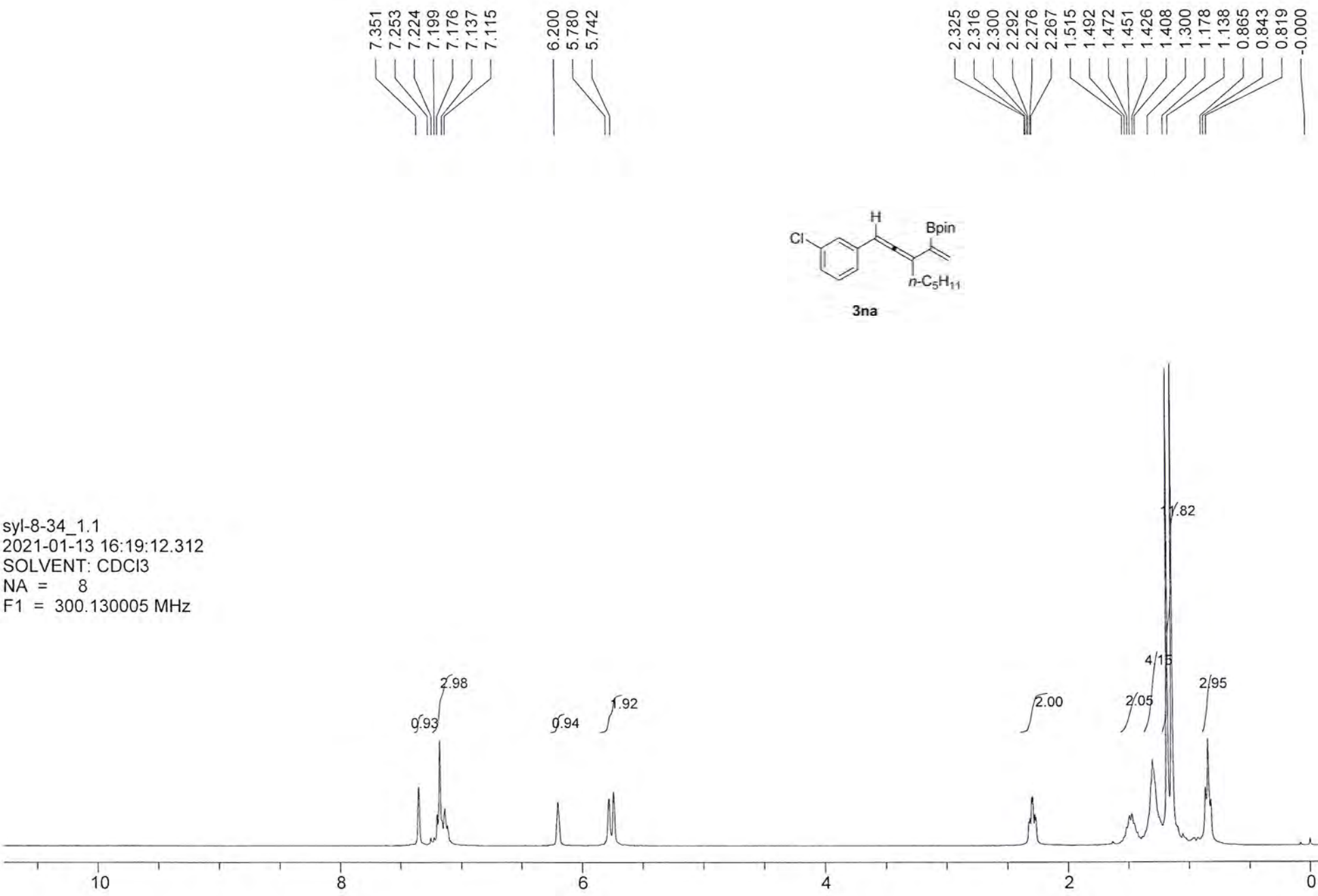

syl-8-49_1.1
2021-01-21 15:19:45.484
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
2.13
2.06
1.00
0.97
1.00
2.04
2.15
3.96
12.28
3.17
10
8
6
4
2
0
PPM

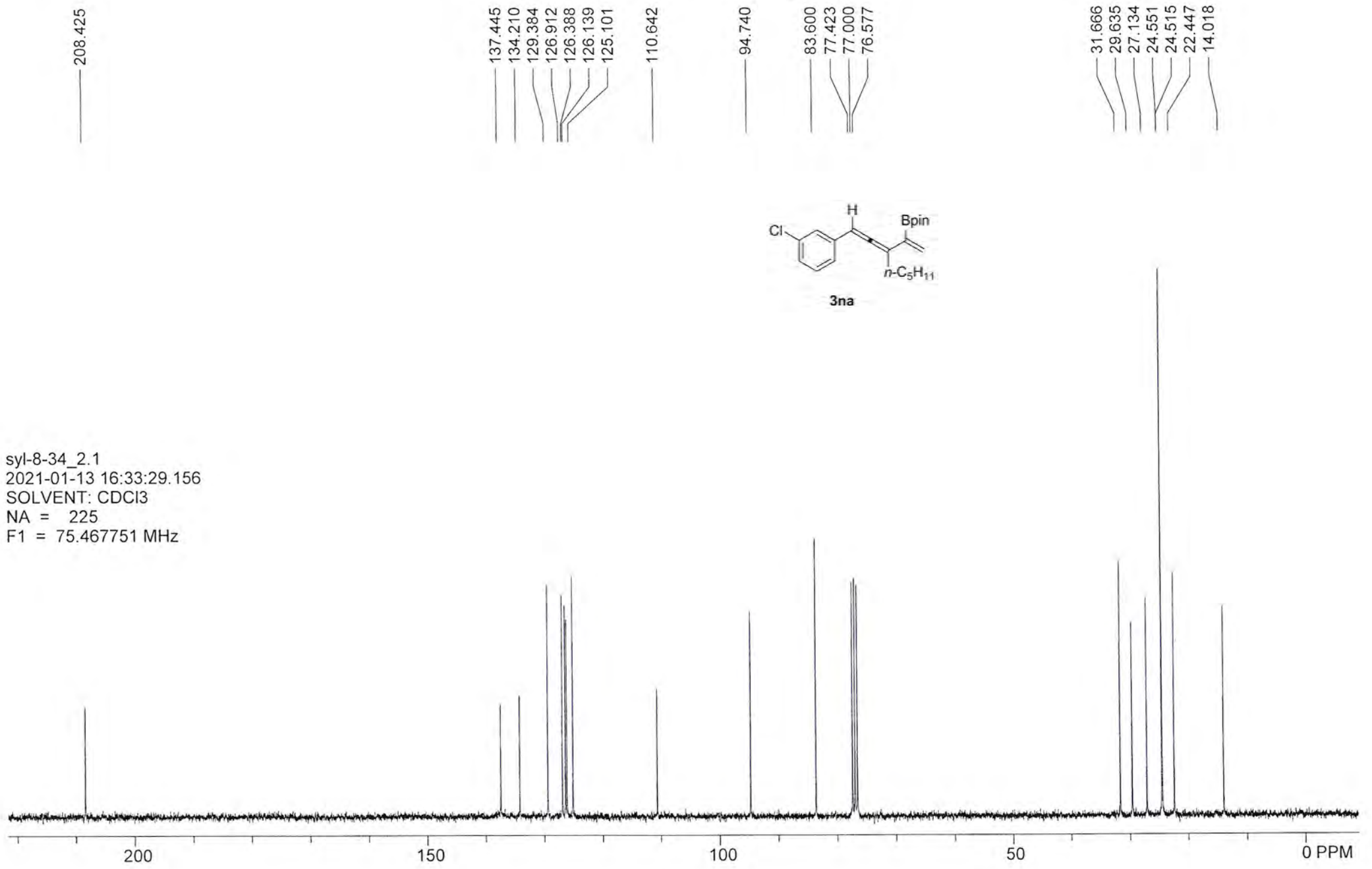

207.983
207.956
163.311
160.066
131.232
131.195
128.391
128.281
125.321
115.275
114.990
110.513
94.749
83.581
77.423
77.000
76.577
31.703
29.487
27.190
24.542
24.506
22.456
14.018
H
Bpin
F
n-C5H11
3la
syl-8-49_2.1
2021-01-21 15:34:01.765
SOLVENT: CDCl3
NA = 228
F1 = 75.467751 MHz
200
150
100
50
0
PPM

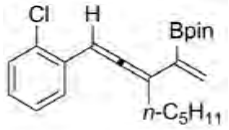

-0.000
-116.706
H
Bpin
F
n-C5H11
3la
syl-8-49_4.1
2021-01-21 15:45:56.093
SOLVENT: CDCl3
NA = 16
F1 = 282.404358 MHz
0
-20
-40
-60
-80
-100
-120
-140
PPM


7.243
6.222
5.764
5.708
2.318
2.309
2.294
2.285
2.269
2.260
1.513
1.490
1.469
1.426
1.294
1.167
1.131
0.858
0.836
0.812
0.000
H
Bpin
Cl
n-C5H11
3ma
syl-8-21_1.1
2021-01-04 14:00:03.843
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
4.01
0.98
0.99
0.96
2.00
2.04
4.06
6.09
5.94
3.18
10
8
6
4
2
0
PPM


208.333
133.824
131.884
128.355
128.125
125.597
110.596
94.805
83.545
77.423
77.000
76.577
31.648
29.432
27.116
24.496
22.419
13.999
H
Bpin
Cl
n-C5H11
3ma
syl-8-21_2.1
2021-01-04 14:06:45.484
SOLVENT: CDCl3
NA = 96
F1 = 75.467751 MHz
200
150
100
50
0
PPM

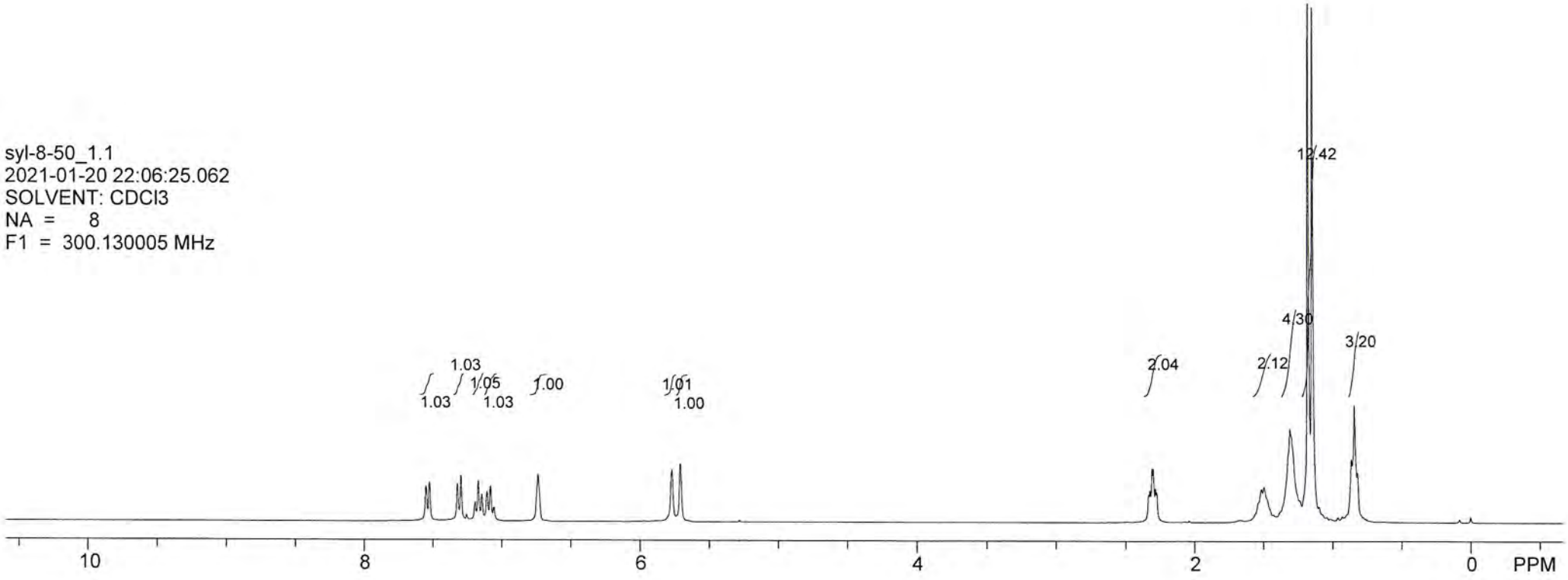

7.351
7.253
7.224
7.199
7.176
7.137
7.115
6.200
5.780
5.742
2.325
2.316
2.300
2.292
2.276
2.267
1.515
1.492
1.472
1.451
1.426
1.408
1.300
1.178
1.138
0.865
0.843
0.819
-0.000
Cl
H
Bpin
n-C5H11
3na
syl-8-34_1.1
2021-01-13 16:19:12.312
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
0.93
2.98
0.94
1.92
2.00
2.05
4.15
11.82
2.95
10
8
6
4
2
0
PPM

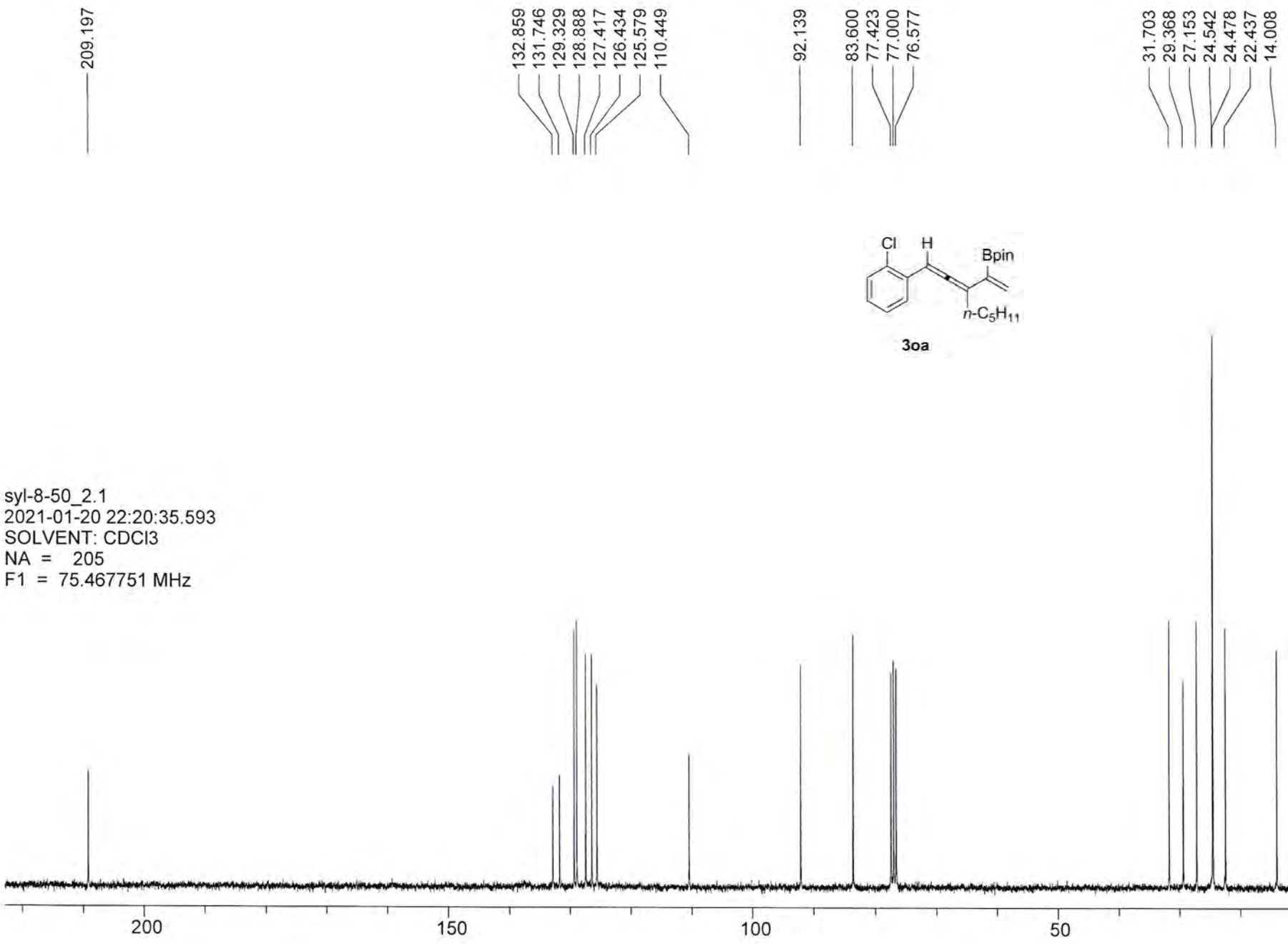

208.425
137.445
134.210
129.384
126.912
126.388
126.139
125.101
110.642
94.740
83.600
77.423
77.000
76.577
31.666
29.635
27.134
24.551
24.515
22.447
14.018
Cl
H
Bpin
n-C5H11
3na
syl-8-34_2.1
2021-01-13 16:33:29.156
SOLVENT: CDCl3
NA = 225
F1 = 75.467751 MHz
200
150
100
50
0 PPM



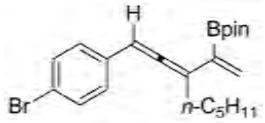
3oa

syl-8-50_1.1
2021-01-20 22:06:25.062
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz

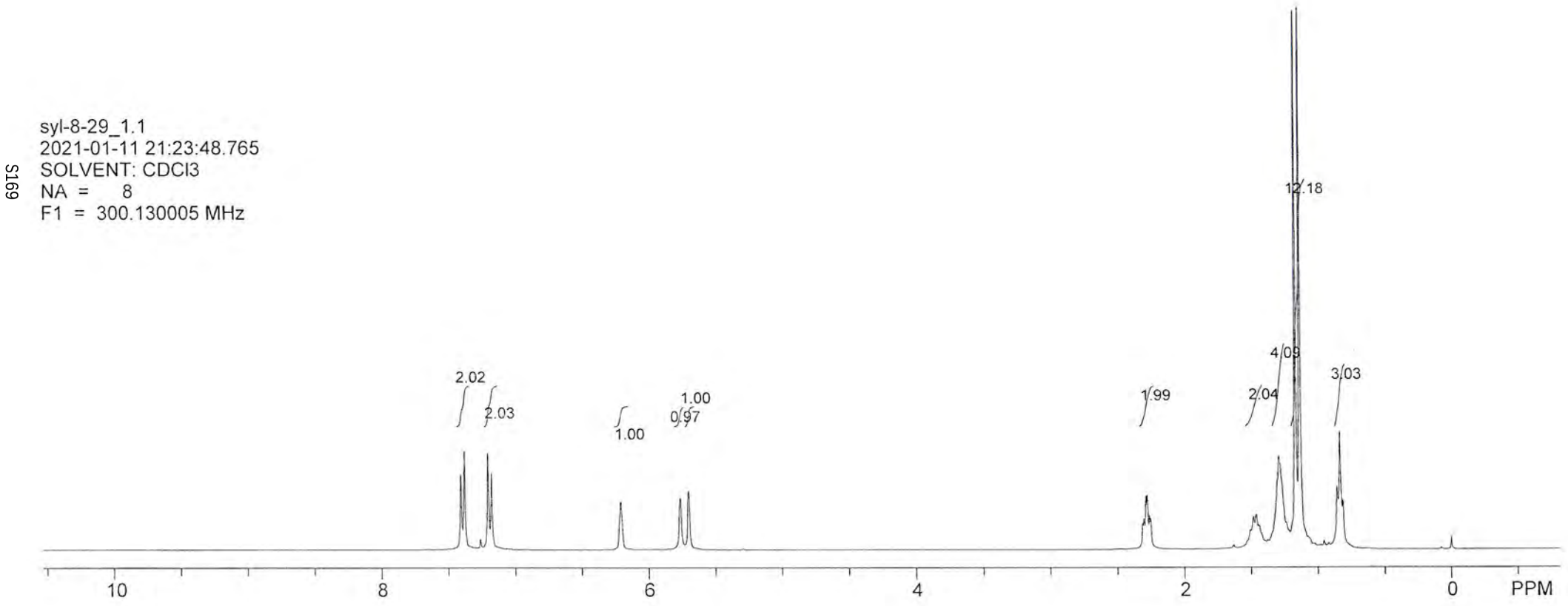



209.197
132.859
131.746
129.329
128.888
127.417
126.434
125.579
110.449
92.139
83.600
77.423
77.000
76.577
31.703
29.368
27.153
24.542
24.478
22.437
14.008
Cl
H
Bpin
n-C5H11
3oa
syl-8-50_2.1
2021-01-20 22:20:35.593
SOLVENT: CDCl3
NA = 205
F1 = 75.467751 MHz
200
150
100
50
0
PPM

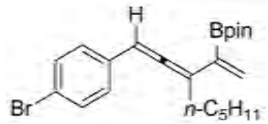

7.407
7.380
7.261
7.205
7.177
6.210
5.764
5.703
2.313
2.304
2.289
2.280
2.264
2.255
1.505
1.482
1.460
1.438
1.291
1.281
1.167
1.133
0.857
0.836
0.812
0.000


H
Bpin
Br
n-C5H11

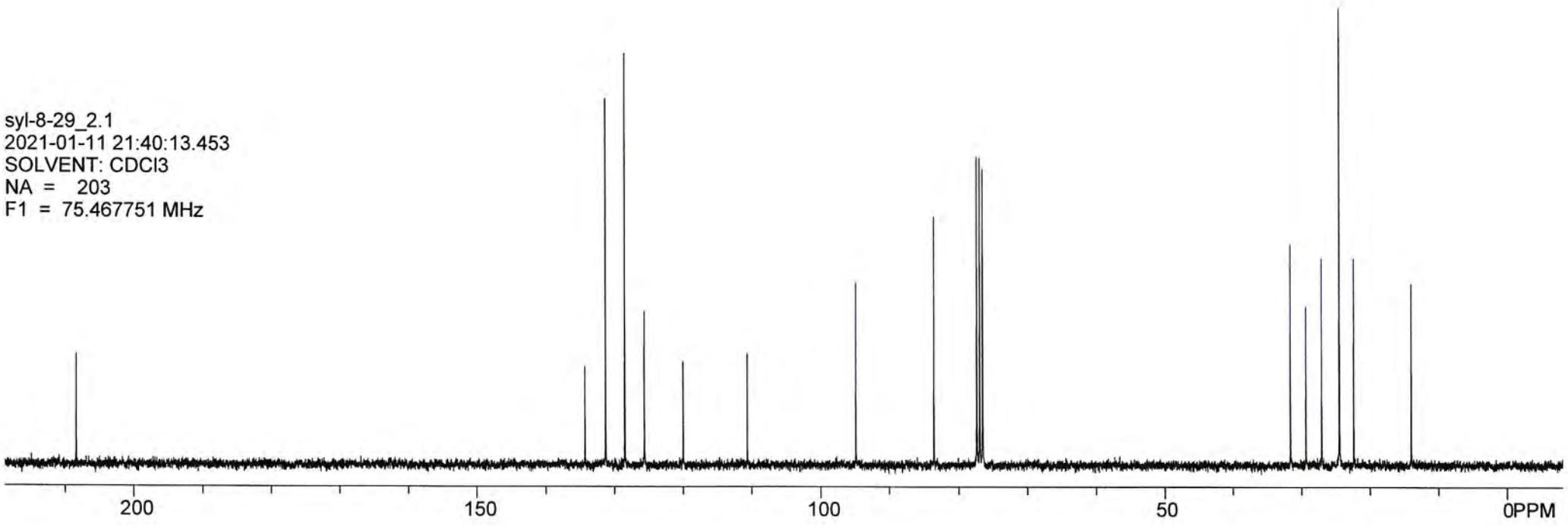
3pa


syl-8-29_1.1
2021-01-11 21:23:48.765
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
2.02
2.03
1.00
1.00
0.97
1.99
2.04
4.09
12.18
3.03
10
8
6
4
2
0
PPM

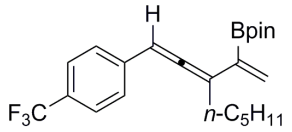
208.379
134.320
131.305
128.502
125.652
119.990
110.679
94.906
83.591
77.423
77.000
76.568
31.666
29.405
27.116
24.542
24.515
22.437
14.027



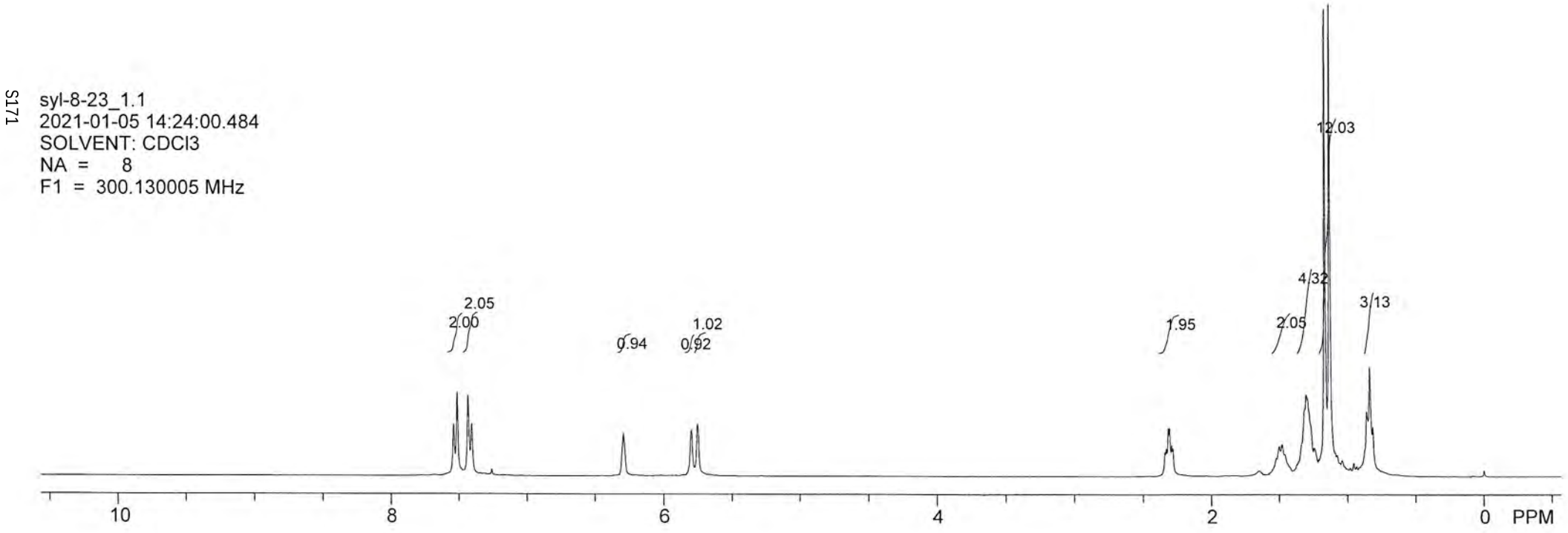
3pa

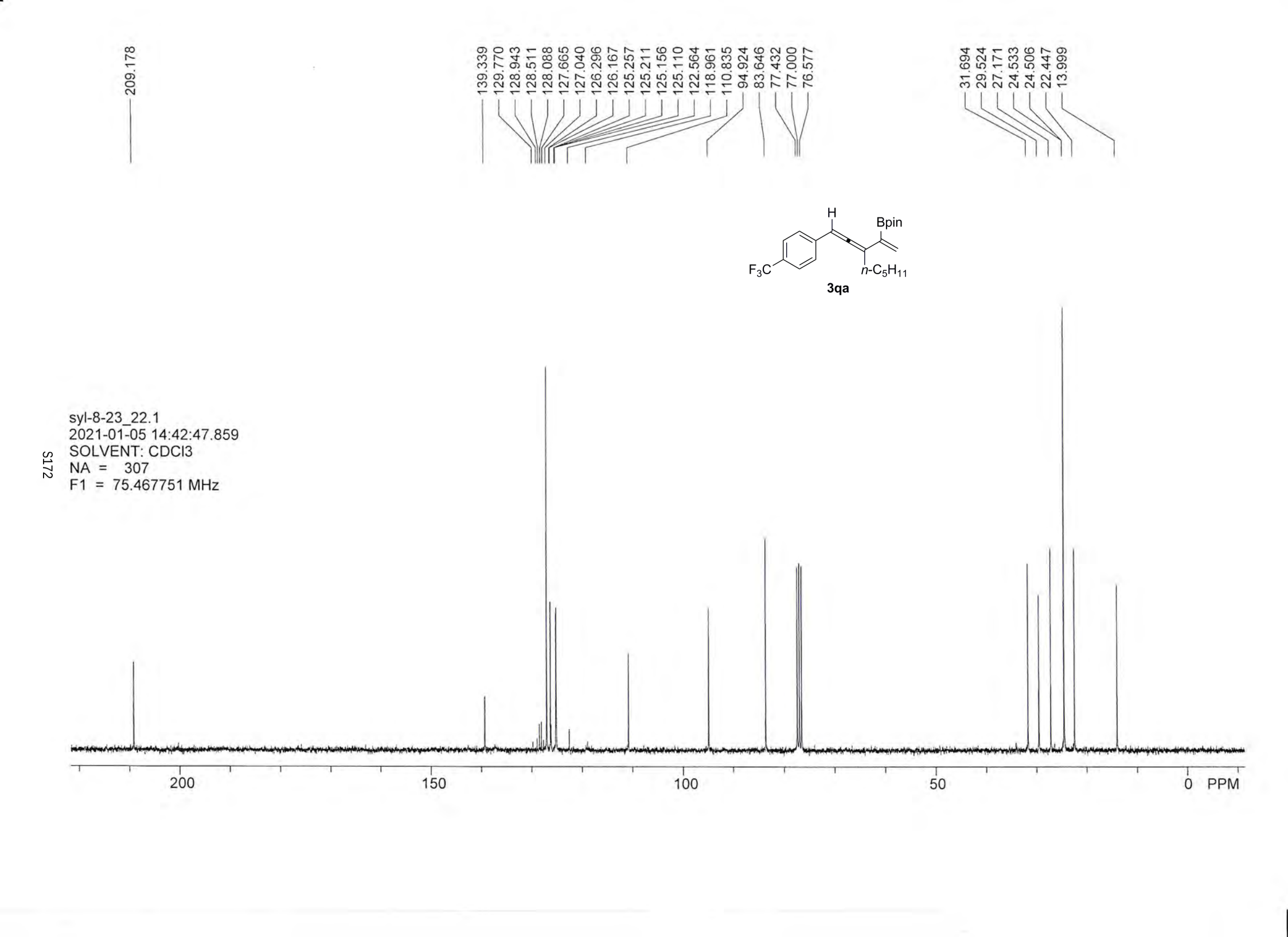


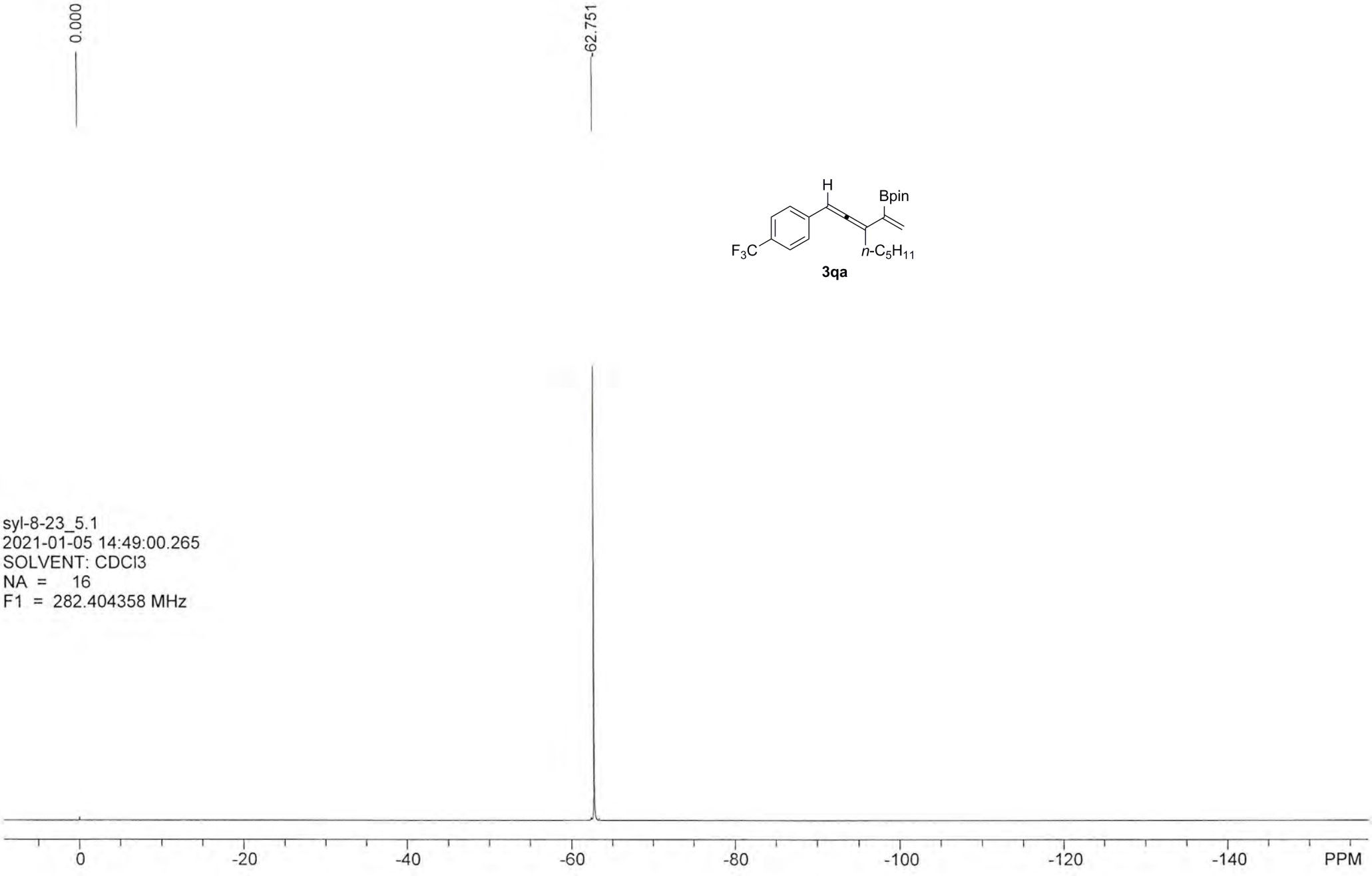

7.539
7.512
7.433
7.406
7.258
6.293
5.798
5.753
2.342
2.333
2.317
2.308
2.292
2.283
1.523
1.500
1.479
1.459
1.313
1.302
1.292
1.164
1.129
0.861
0.838
0.815
-0.000

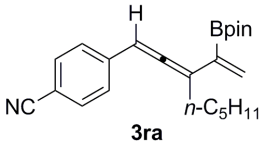

H
Bpin
F3C
n-C5H11

3qa


syl-8-23_1.1
2021-01-05 14:24:00.484
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
2.05
2.00
0.94
1.02
0.92
1.95
2.05
12.03
4.32
3.13
10
8
6
4
2
0
PPM

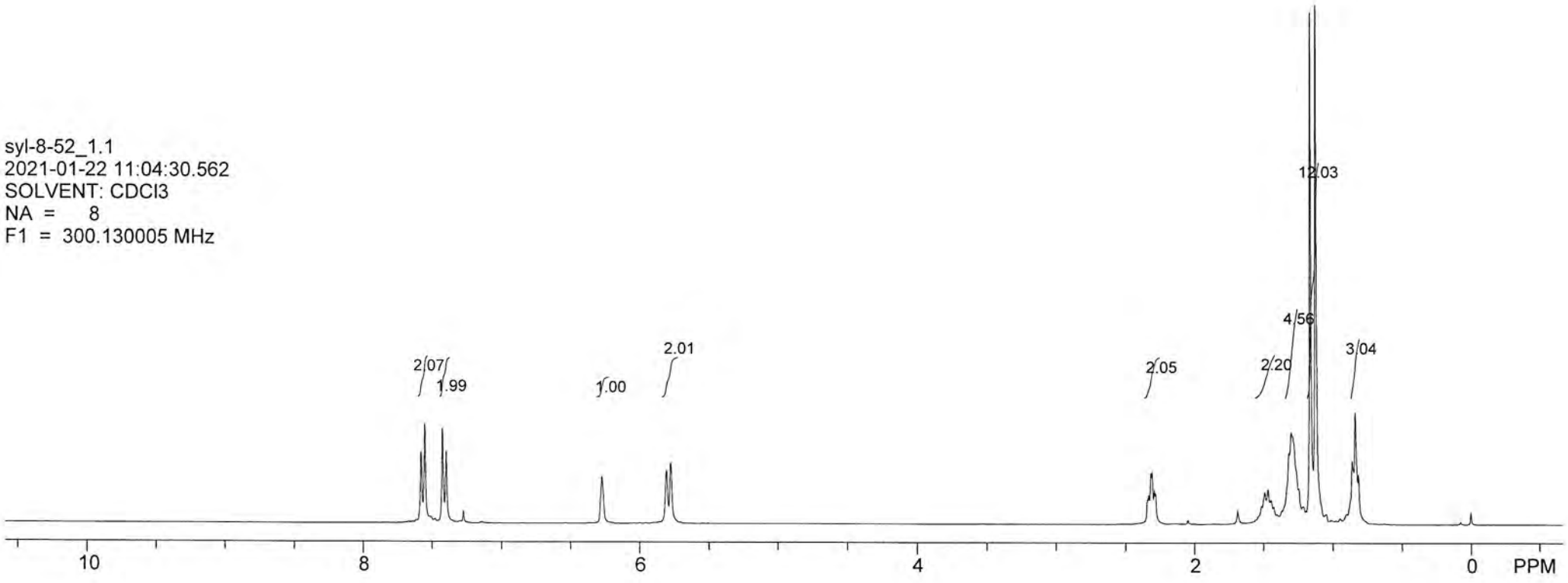

209.178
139.339
129.770
128.943
128.511
128.088
127.665
127.040
126.296
126.167
125.257
125.211
125.156
125.110
122.564
118.961
110.835
94.924
83.646
77.432
77.000
76.577
31.694
29.524
27.171
24.533
24.506
22.447
13.999
H
Bpin
F3C
n-C5H11
3qa
syl-8-23_22.1
2021-01-05 14:42:47.859
SOLVENT: CDCl3
NA = 307
F1 = 75.467751 MHz
200
150
100
50
0 PPM


0.000
-62.751
H
Bpin
F3C
n-C5H11
3qa
syl-8-23_5.1
2021-01-05 14:49:00.265
SOLVENT: CDCl3
NA = 16
F1 = 282.404358 MHz
0
-20
-40
-60
-80
-100
-120
-140
PPM

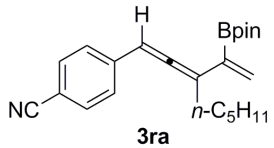


3ra

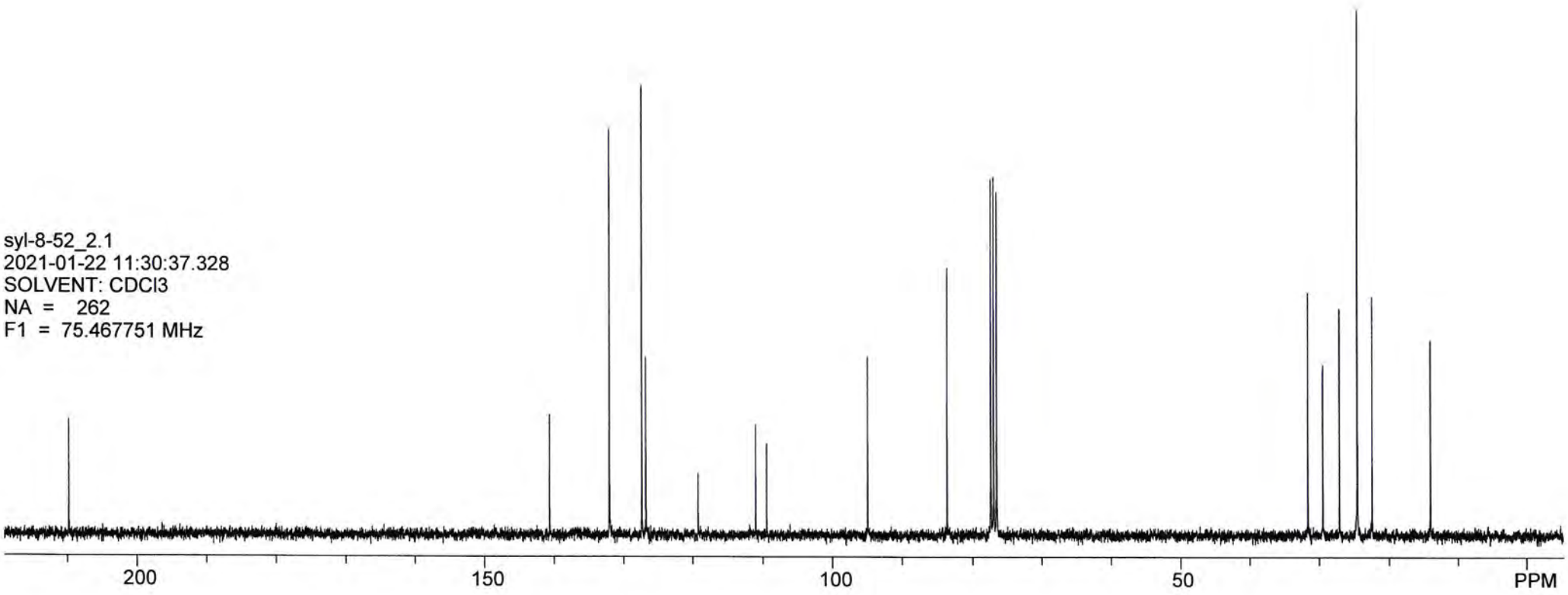
syl-8-52_1.1
2021-01-22 11:04:30.562
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz

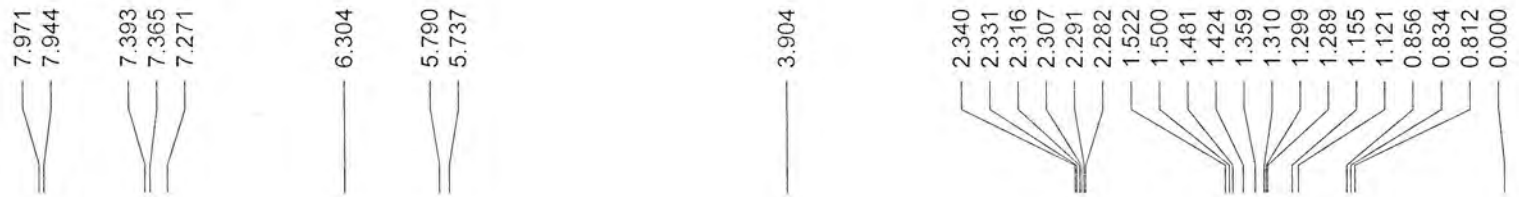


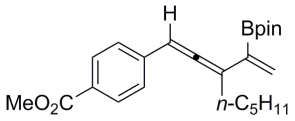
209.803
140.653
132.059
127.371
126.792
119.255
111.046
109.465
94.961
83.636
77.423
77.000
76.577
31.611
29.442
27.079
24.515
24.487
22.382
13.981



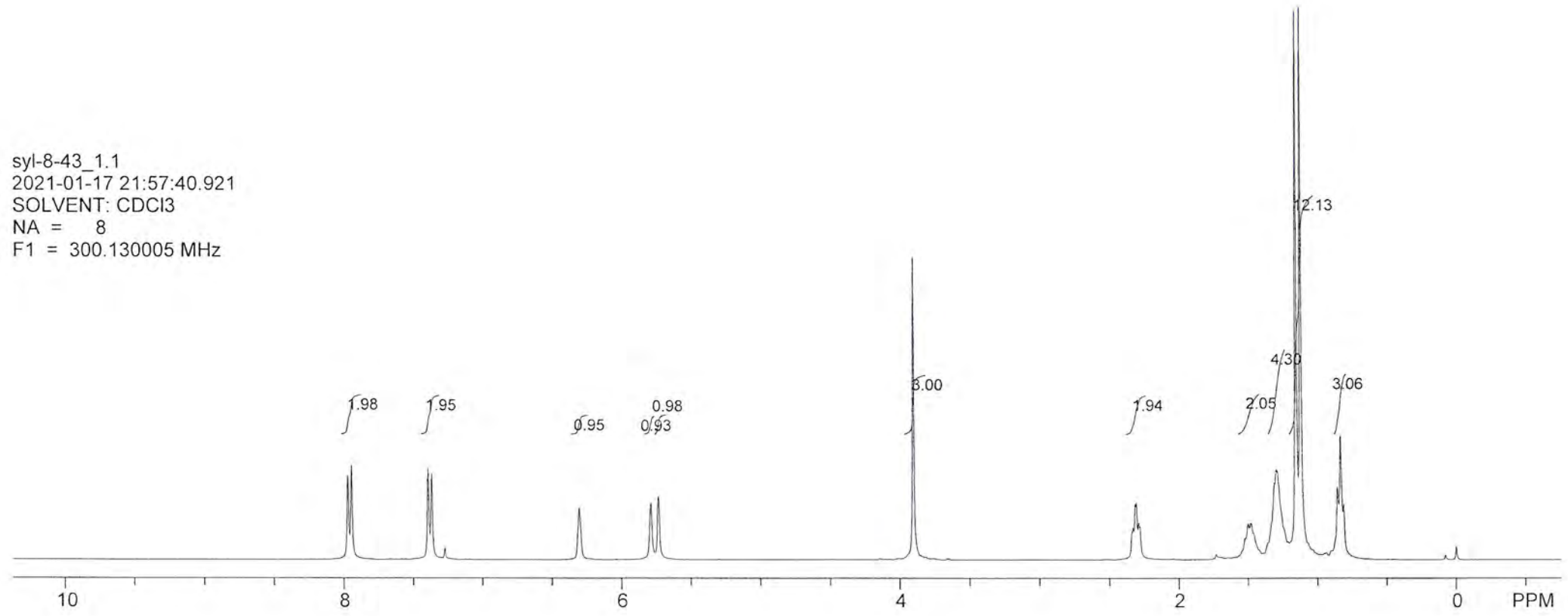
3ra



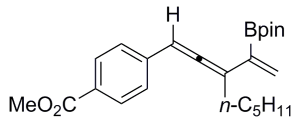

7.971
7.944
7.393
7.365
7.271
6.304
5.790
5.737
3.904
2.340
2.331
2.316
2.307
2.291
2.282
1.522
1.500
1.481
1.424
1.359
1.310
1.299
1.289
1.155
1.121
0.856
0.834
0.812
0.000


H
Bpin
MeO2C
n-C5H11

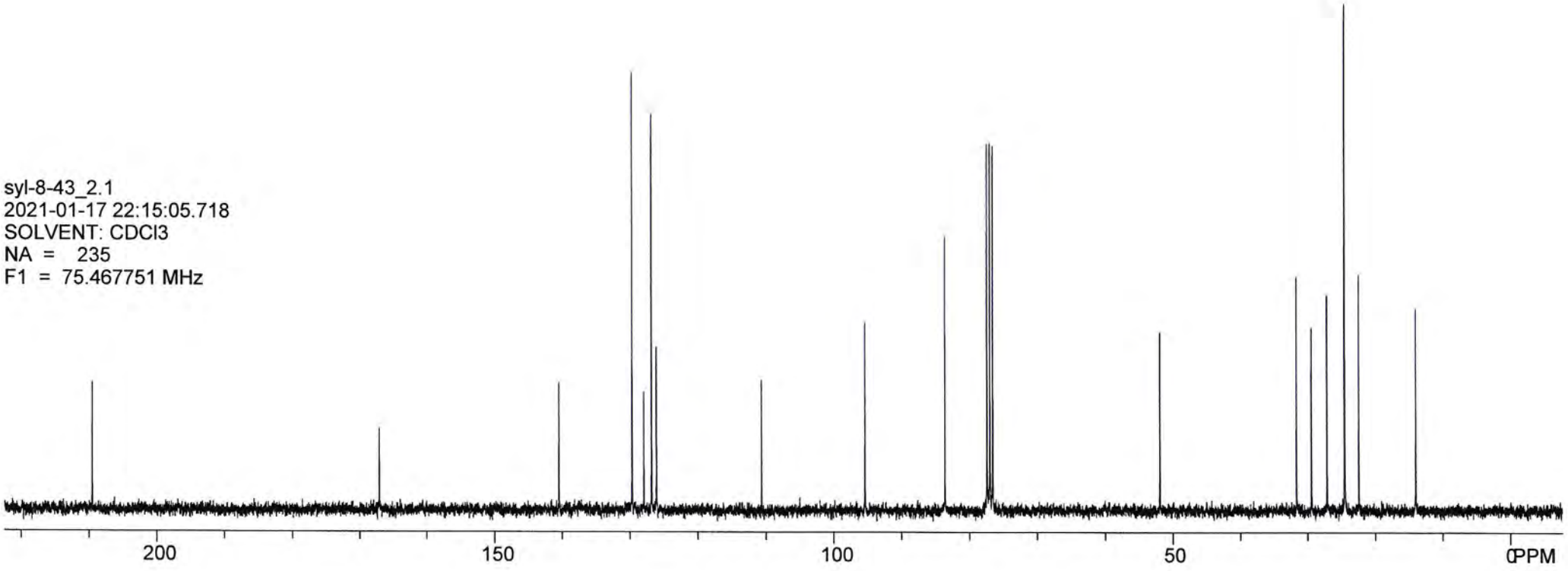
3sa

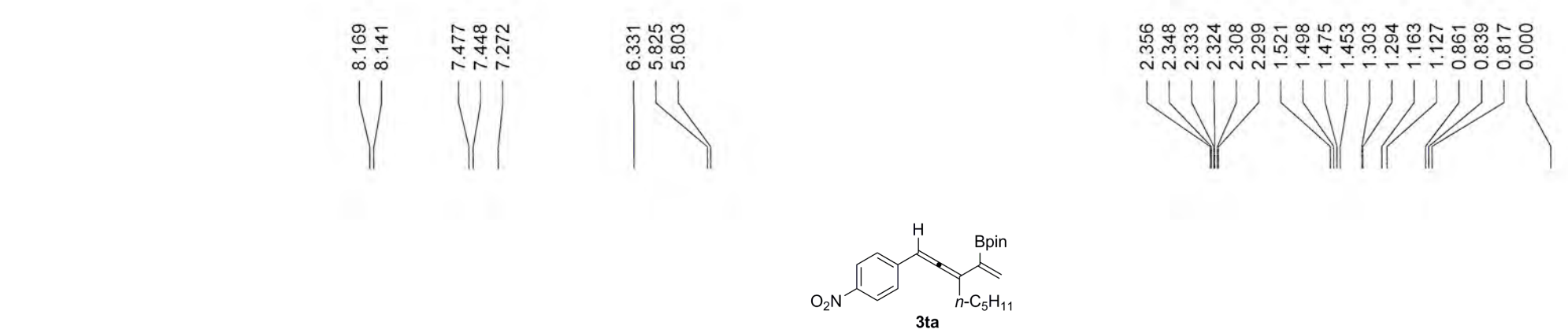

syl-8-43_1.1
2021-01-17 21:57:40.921
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
1.98
1.95
0.95
0.98
0.93
3.00
1.94
2.05
4.30
12.13
3.06
10
8
6
4
2
0
PPM

209.518
167.061
140.451
129.632
127.913
126.774
126.057
110.614
95.347
83.600
77.423
77.000
76.568
51.916
31.638
29.432
27.116
24.524
24.496
22.410
13.999



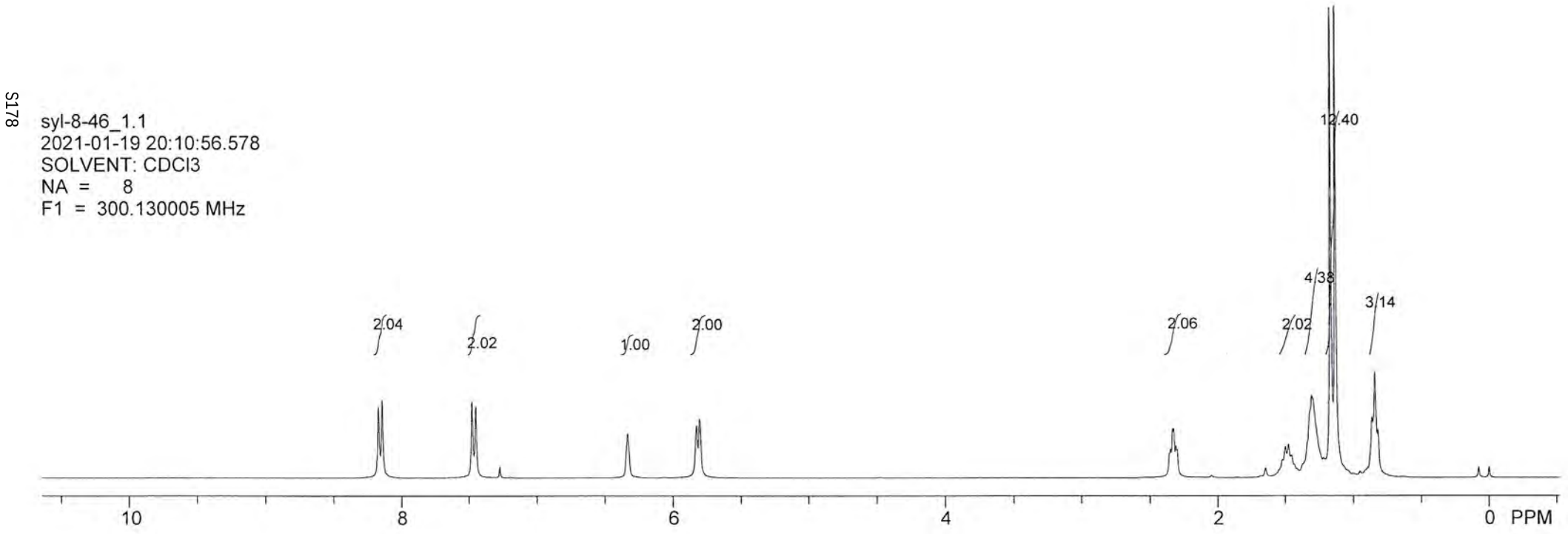
3sa

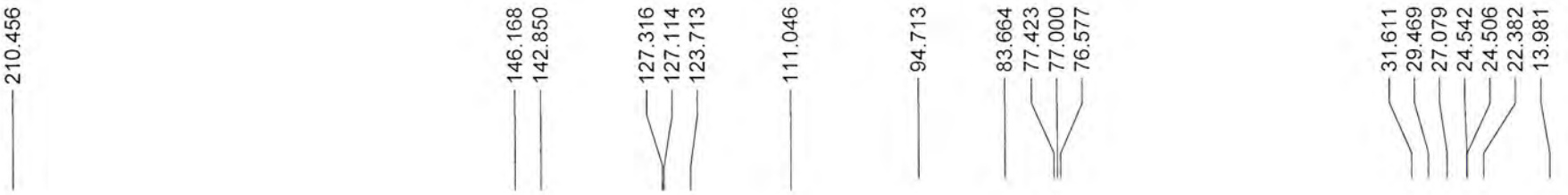


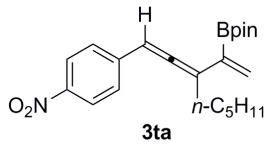

8.169
8.141
7.477
7.448
7.272
6.331
5.825
5.803
2.356
2.348
2.333
2.324
2.308
2.299
1.521
1.498
1.475
1.453
1.303
1.294
1.163
1.127
0.861
0.839
0.817
0.000

H
Bpin
O2N
n-C5H11

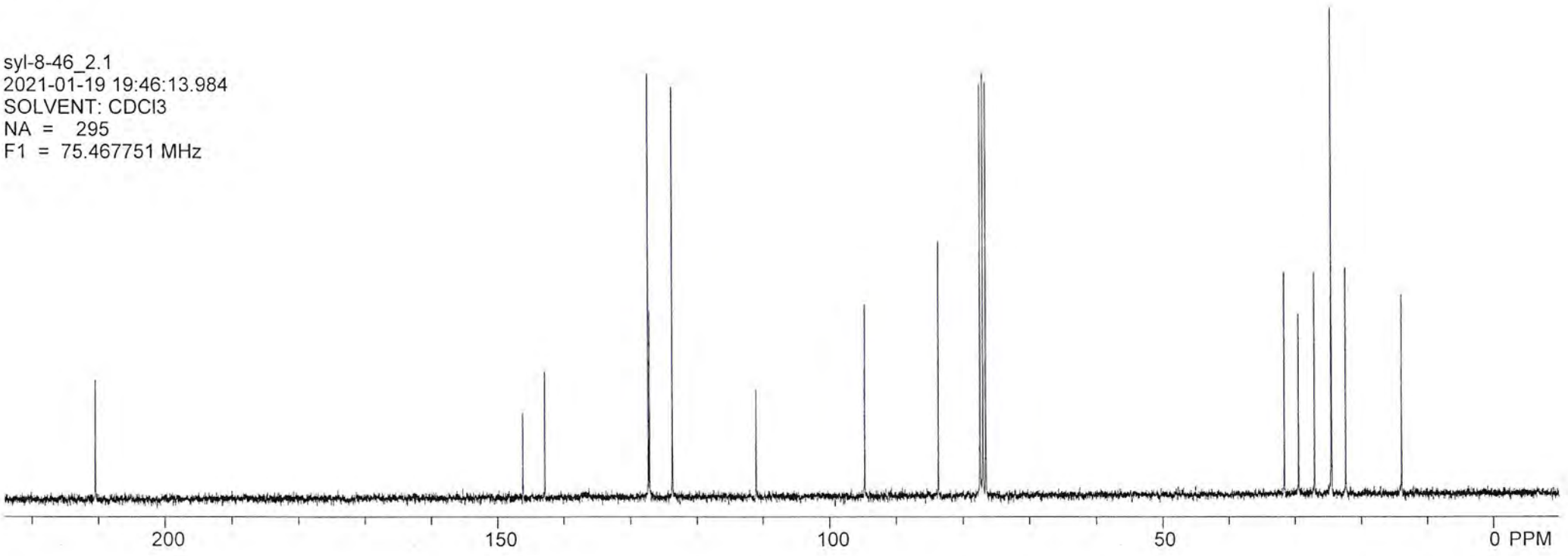
3ta

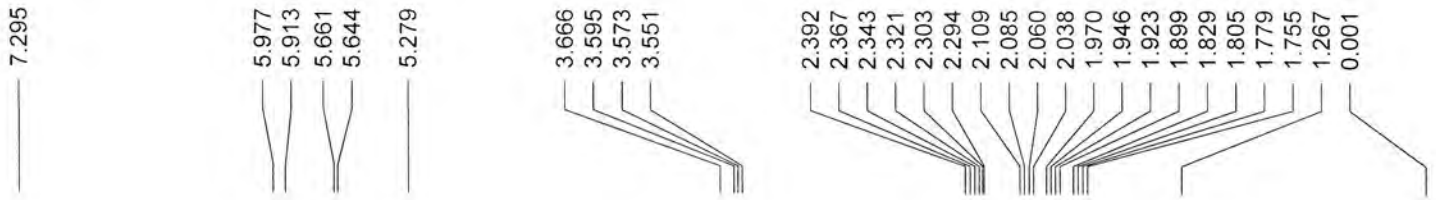

syl-8-46_1.1
2021-01-19 20:10:56.578
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
2.04
2.02
1.00
2.00
2.06
2.02
12.40
4.38
3.14
10
8
6
4
2
0 PPM

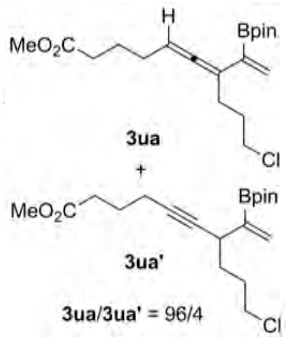

210.456
146.168
142.850
127.316
127.114
123.713
111.046
94.713
83.664
77.423
77.000
76.577
31.611
29.469
27.079
24.542
24.506
22.382
13.981

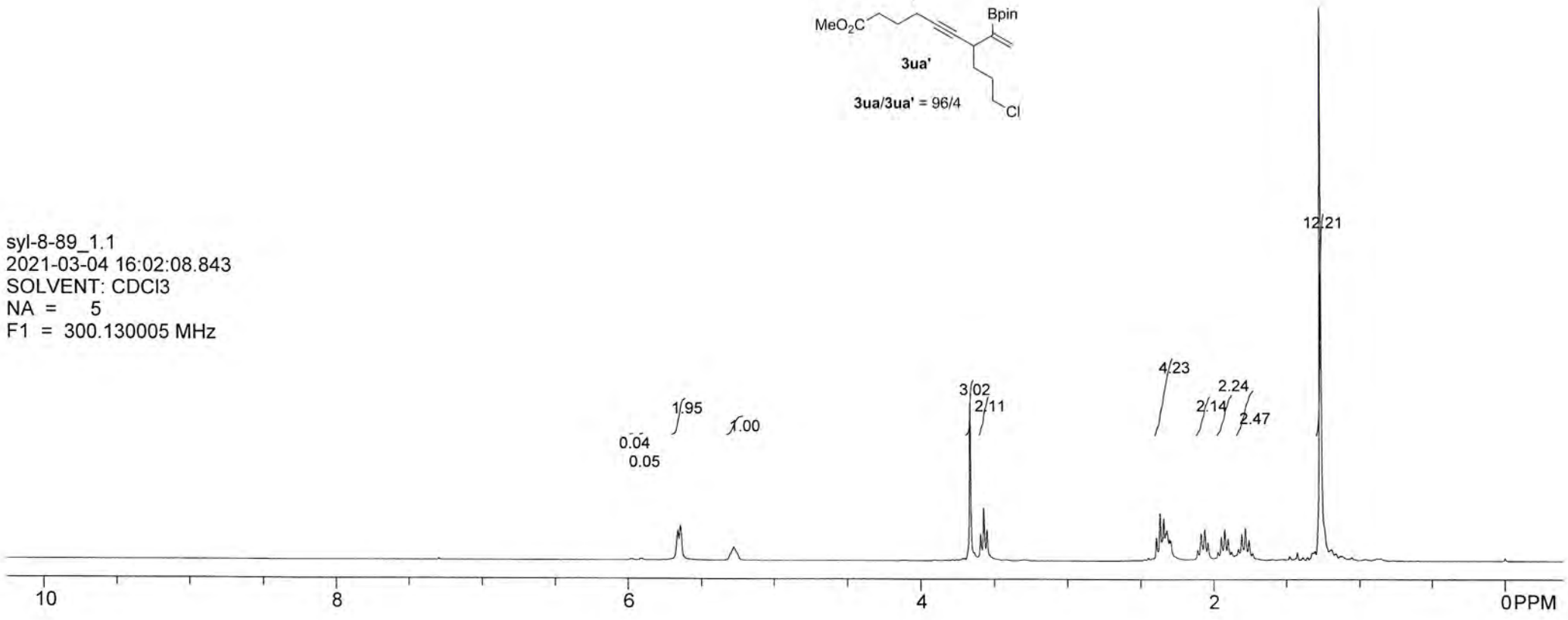

H
Bpin
O_2N
n-C_5H_{11}

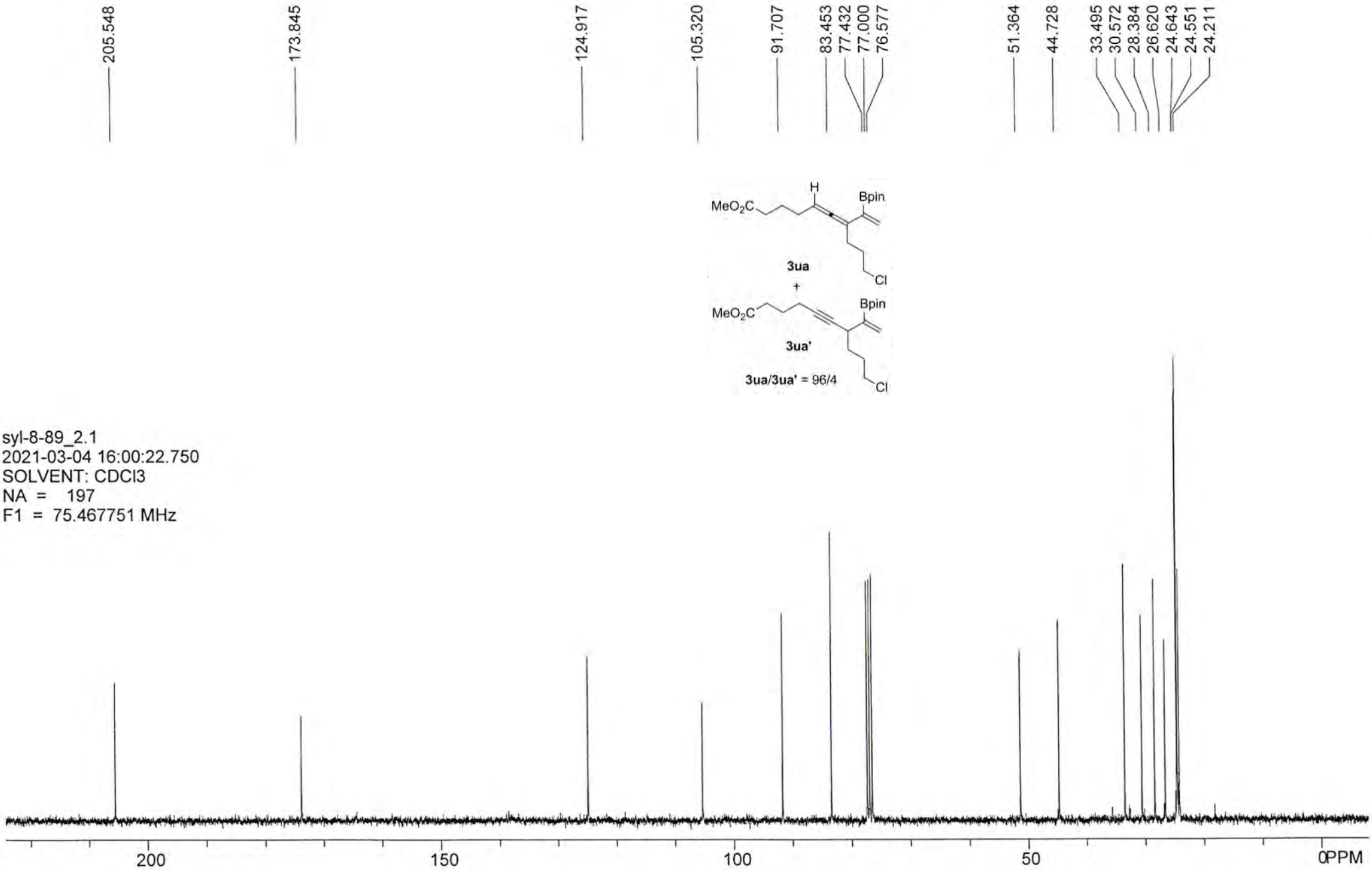
3ta

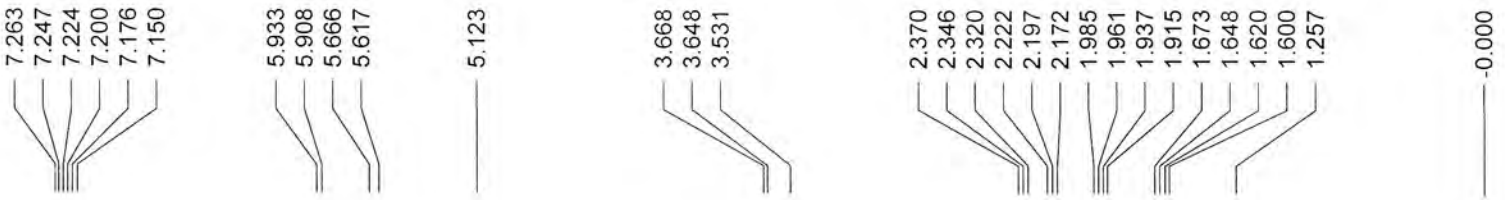

syl-8-46_2.1
2021-01-19 19:46:13.984
SOLVENT: CDCl3
NA = 295
F1 = 75.467751 MHz
200
150
100
50
0 PPM

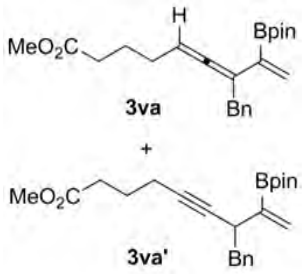

7.295
5.977
5.913
5.661
5.644
5.279
3.666
3.595
3.573
3.551
2.392
2.367
2.343
2.321
2.303
2.294
2.109
2.085
2.060
2.038
1.970
1.946
1.923
1.899
1.829
1.805
1.779
1.755
1.267
0.001


MeO2C
H
Bpin
Cl
3ua
+
MeO2C
Bpin
Cl
3ua'
3ua/3ua' = 96/4

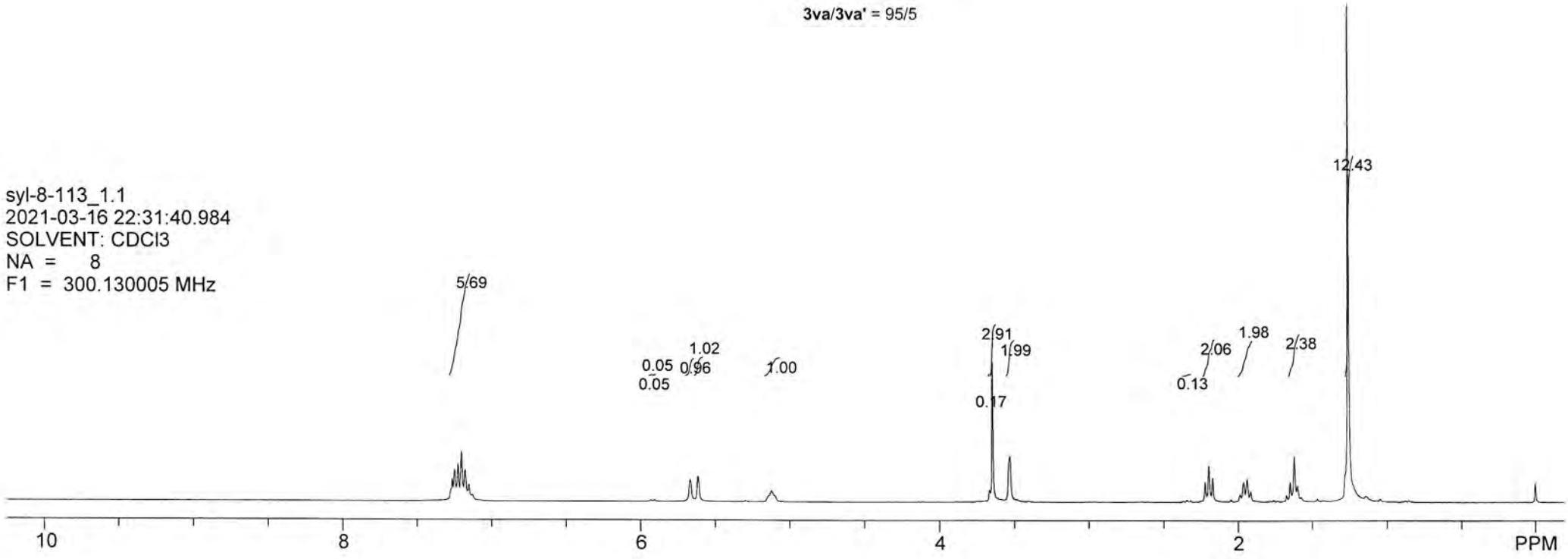

syl-8-89_1.1
2021-03-04 16:02:08.843
SOLVENT: CDCl3
NA = 5
F1 = 300.130005 MHz
0.04
0.05
1.95
1.00
3.02
2.11
4.23
2.14
2.24
2.47
12.21
10
8
6
4
2
0PPM

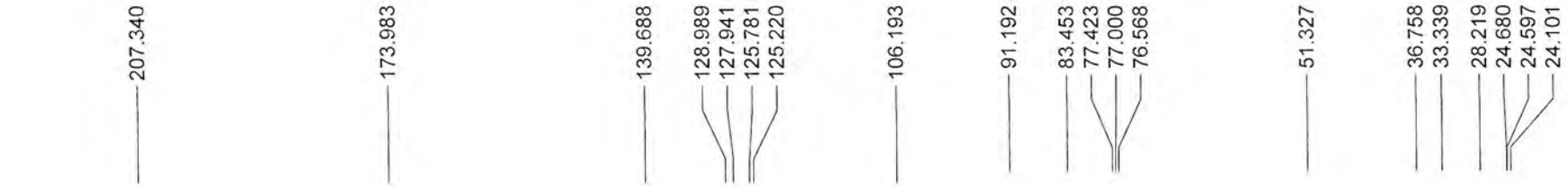

205.548
173.845
124.917
105.320
91.707
83.453
77.432
77.000
76.577
51.364
44.728
33.495
30.572
28.384
26.620
24.643
24.551
24.211
MeO2C
H
Bpin
Cl
3ua
+
MeO2C
Bpin
3ua'
Cl
3ua/3ua' = 96/4
syl-8-89_2.1
2021-03-04 16:00:22.750
SOLVENT: CDCl3
NA = 197
F1 = 75.467751 MHz
200
150
100
50
0
PPM

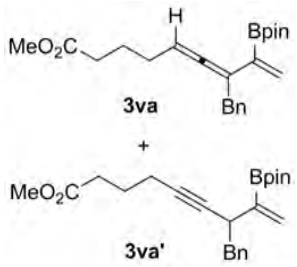

7.263
7.247
7.224
7.200
7.176
7.150
5.933
5.908
5.666
5.617
5.123
3.668
3.648
3.531
2.370
2.346
2.320
2.222
2.197
2.172
1.985
1.961
1.937
1.915
1.673
1.648
1.620
1.600
1.257
-0.000


MeO2C
H
Bpin
Bn
3va
+
MeO2C
Bpin
Bn
3va'

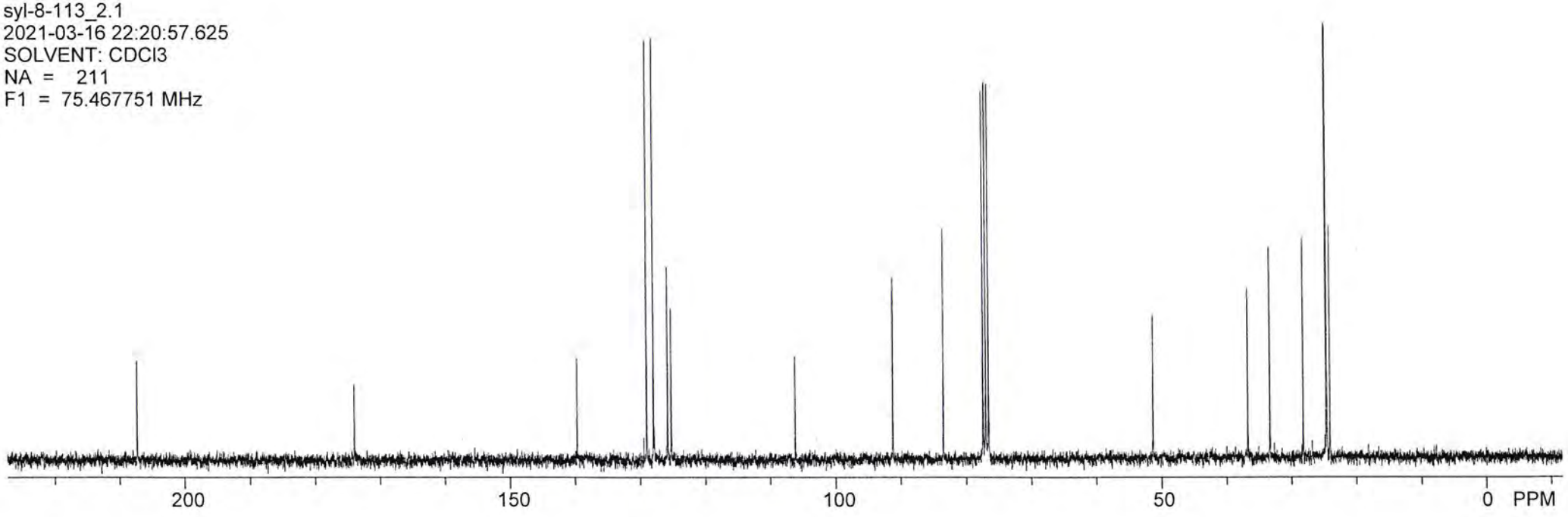
3va/3va' = 95/5

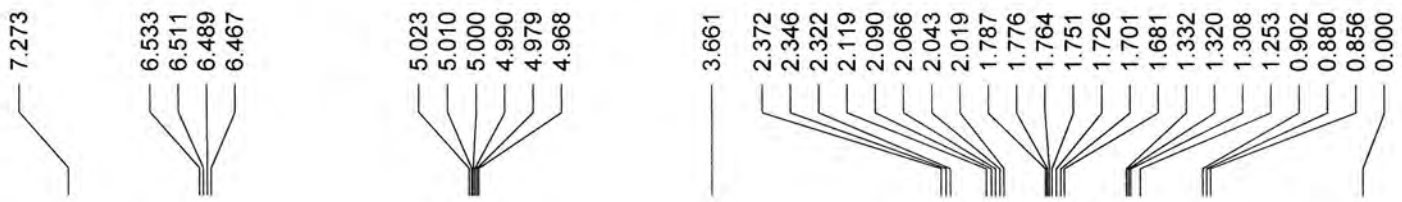

syl-8-113_1.1
2021-03-16 22:31:40.984
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
5.69
0.05
0.05
1.02
0.96
1.00
2.91
1.99
0.17
0.13
2.06
1.98
2.38
12.43
10
8
6
4
2
PPM

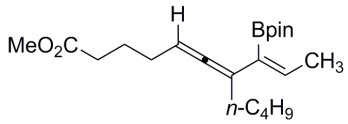

207.340
173.983
139.688
128.989
127.941
125.781
125.220
106.193
91.192
83.453
77.423
77.000
76.568
51.327
36.758
33.339
28.219
24.680
24.597
24.101


MeO2C
H
Bpin
Bn
3va
+
MeO2C
Bpin
Bn
3va'

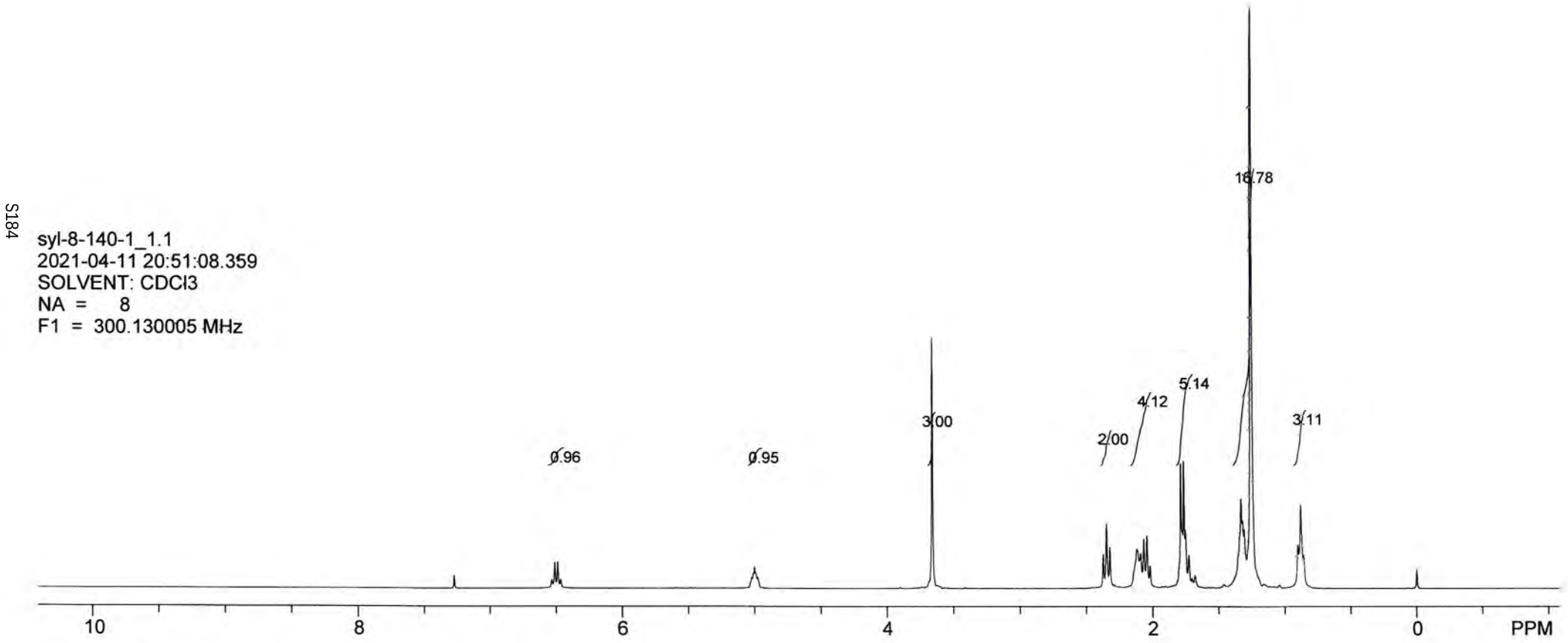
3va/3va' = 95/5


syl-8-113_2.1
2021-03-16 22:20:57.625
SOLVENT: CDCl3
NA = 211
F1 = 75.467751 MHz
200
150
100
50
0 PPM

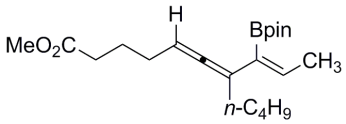

7.273
6.533
6.511
6.489
6.467
5.023
5.010
5.000
4.990
4.979
4.968
3.661
2.372
2.346
2.322
2.119
2.090
2.066
2.043
2.019
1.787
1.776
1.764
1.751
1.726
1.701
1.681
1.332
1.320
1.308
1.253
0.902
0.880
0.856
0.000


H
Bpin
MeO2C
CH3
n-C4H9

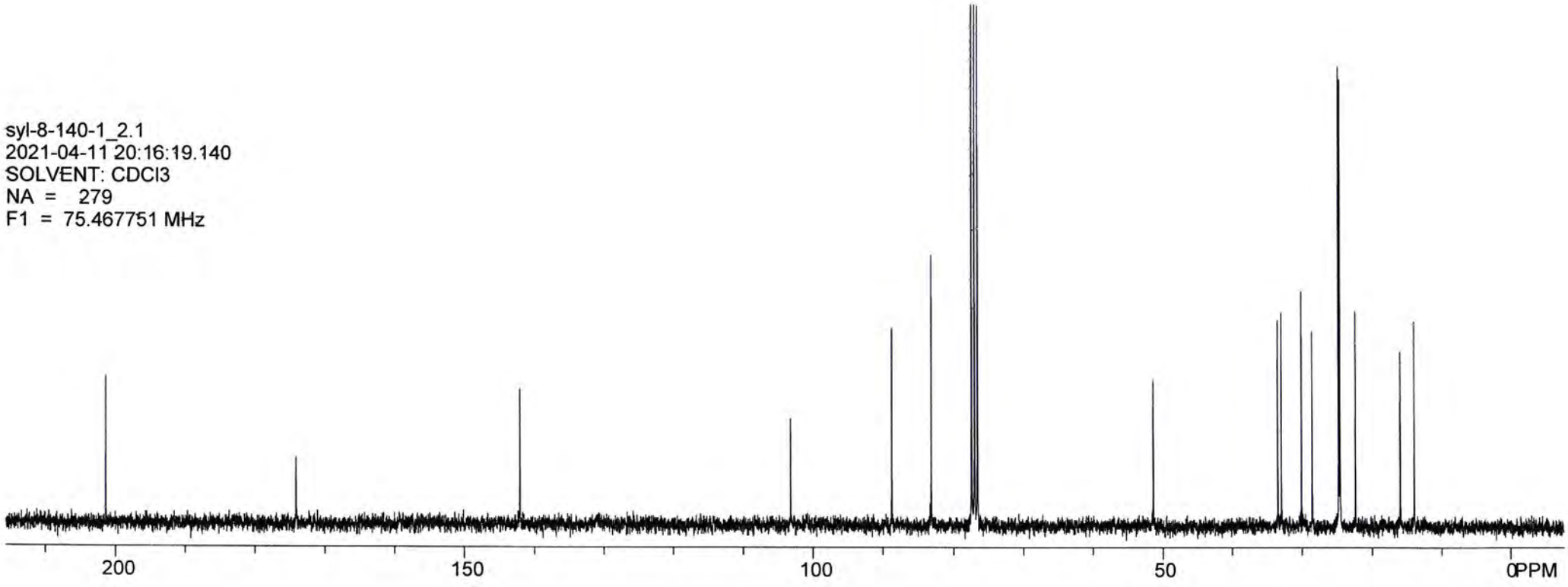
***E*-3wa**

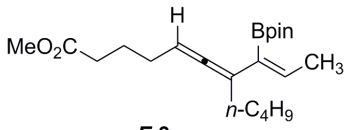

syl-8-140-1_1.1
2021-04-11 20:51:08.359
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
0.96
0.95
3.00
2.00
4.12
5.14
18.78
3.11
10
8
6
4
2
0
PPM


201.411
174.148
142.060
103.169
88.710
83.131
77.423
77.000
76.568
51.364
33.412
32.888
30.030
28.504
24.726
24.533
22.336
15.939
13.917


H
Bpin
MeO2C
CH3
n-C4H9

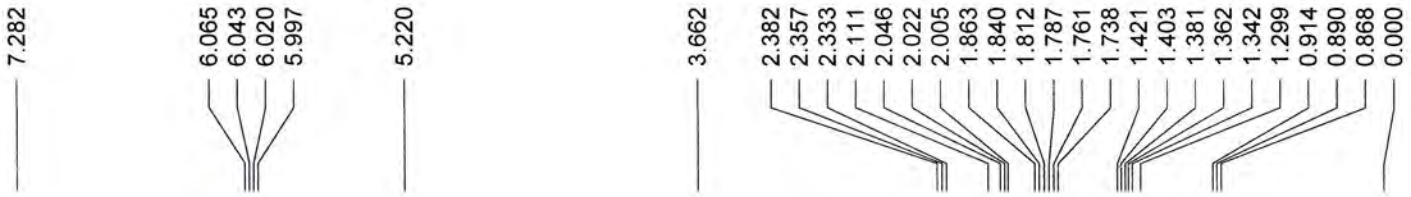
***E*-3wa**

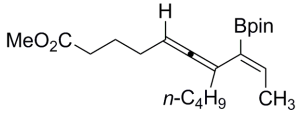

syl-8-140-1_2.1
2021-04-11 20:16:19.140
SOLVENT: CDCl3
NA = 279
F1 = 75.467751 MHz
200
150
100
50
0PPM



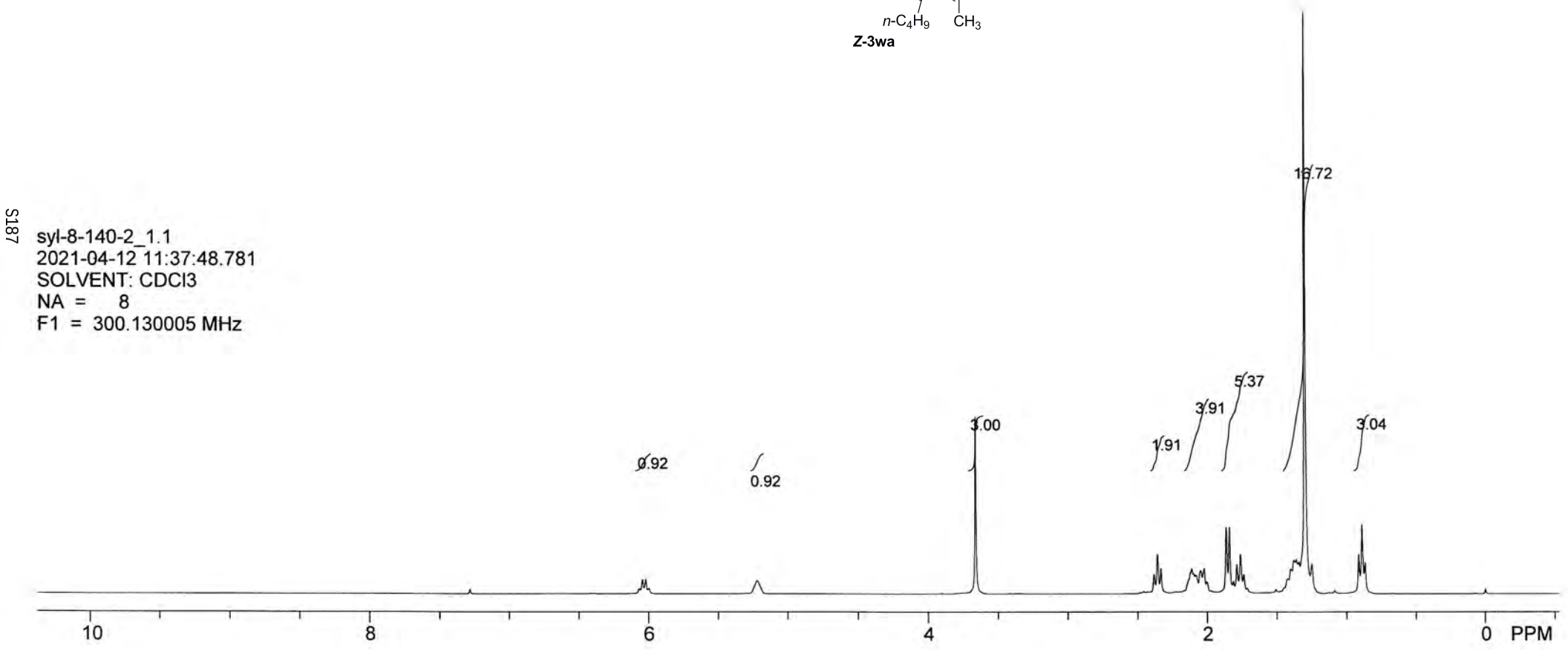
***E*-3wa**

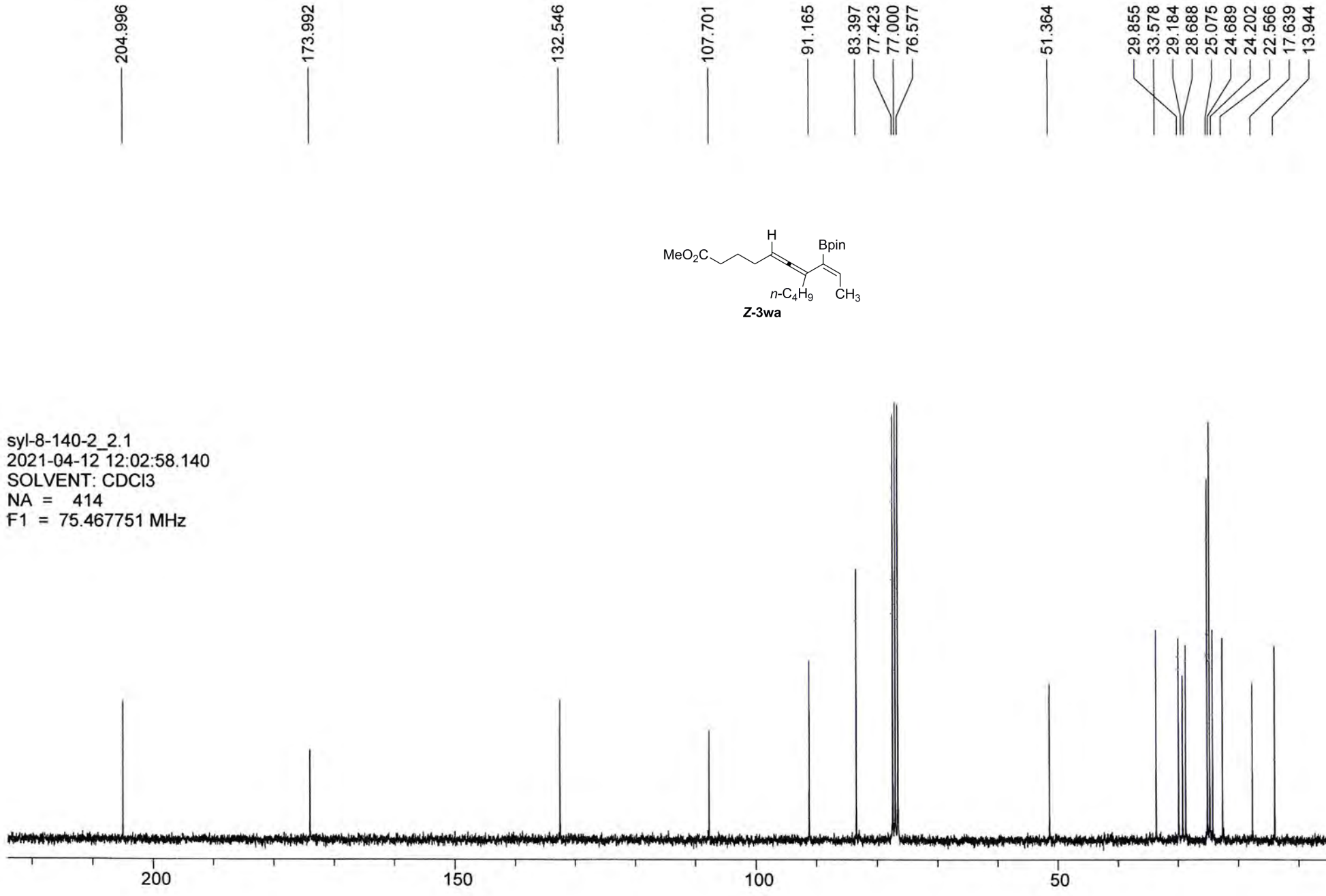
```
Current Data Parameters
NAME      syl-8-140-1-noe
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20210411
Time                22.00
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG         noesygpph
TD                   2048
SOLVENT             CDCl3
NS                     64
DS                      4
SWH              3063.726 Hz
FIDRES           1.495960 Hz
AQ               0.3342836 sec
RG                     203
DW                163.200 usec
DE                  6.50 usec
TE                 300.0 K
d0               0.00014537 sec
D1               2.00000000 sec
D16              0.00010000 sec
D8               0.50000000 sec
IN0              0.00032640 sec
ST1CNT                 128
TAU              0.24840000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.00 usec
p2                  28.00 usec
PL1                  1.00 dB
SFO1          300.1313815 MHz

====== GRADIENT CHANNEL =====
GPNAM1           SINE.100
GPNAM2           SINE.100
GPZ1                80.00 %
GPZ2                40.00 %
P16               1500.00 usec

F1 - Acquisition parameters
ND0                     1
TD                    210
SFO1             300.1314 MHz
FIDRES          14.589169 Hz
SW                10.208 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                   1024
SF            300.1300000 MHz
WDW                 QSINE
SSB                     2
LB                   0.00 Hz
GB                      0
PC                   1.00

F1 - Processing parameters
SI                   1024
MC2           States-TPPI
SF            300.1300000 MHz
WDW                 QSINE
SSB                     2
LB                   0.00 Hz
GB                      0
```

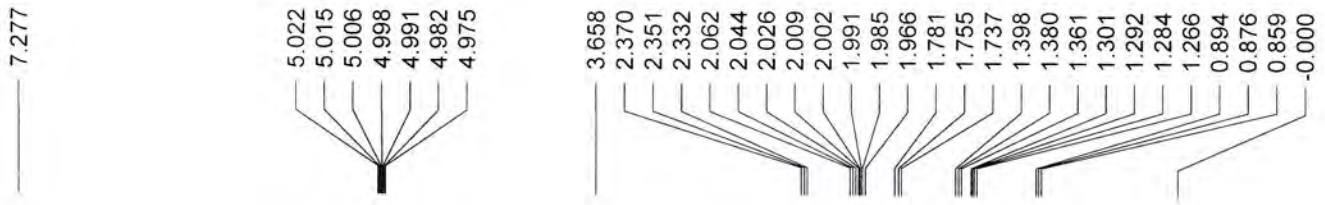

7.282
6.065
6.043
6.020
5.997
5.220
3.662
2.382
2.357
2.333
2.111
2.046
2.022
2.005
1.863
1.840
1.812
1.787
1.761
1.738
1.421
1.403
1.381
1.362
1.342
1.299
0.914
0.890
0.868
0.000

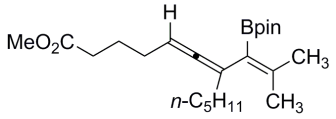

H
Bpin
MeO2C
n-C4H9
CH3

***Z*-3wa**


syl-8-140-2_1.1
2021-04-12 11:37:48.781
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
0.92
0.92
3.00
1.91
3.91
5.37
16.72
3.04
10
8
6
4
2
0
PPM


204.996
173.992
132.546
107.701
91.165
83.397
77.423
77.000
76.577
51.364
29.855
33.578
29.184
28.688
25.075
24.689
24.202
22.566
17.639
13.944
H
MeO2C
Bpin
n-C4H9
CH3
Z-3wa
syl-8-140-2_2.1
2021-04-12 12:02:58.140
SOLVENT: CDCl3
NA = 414
F1 = 75.467751 MHz
200
150
100
50
0
PPM

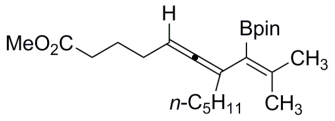

7.277
5.022
5.015
5.006
4.998
4.991
4.982
4.975
3.658
2.370
2.351
2.332
2.062
2.044
2.026
2.009
2.002
1.991
1.985
1.966
1.781
1.755
1.737
1.398
1.380
1.361
1.301
1.292
1.284
1.266
0.894
0.876
0.859
-0.000


H
Bpin
MeO2C
CH3
n-C5H11
CH3

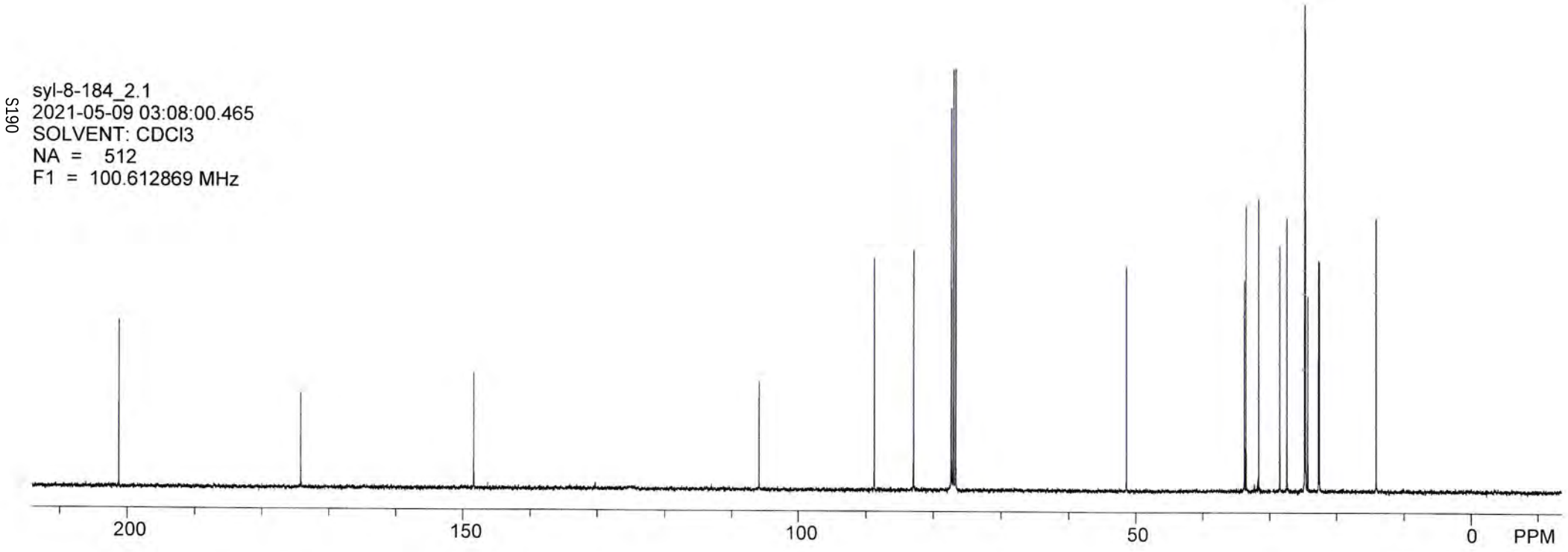
3xa

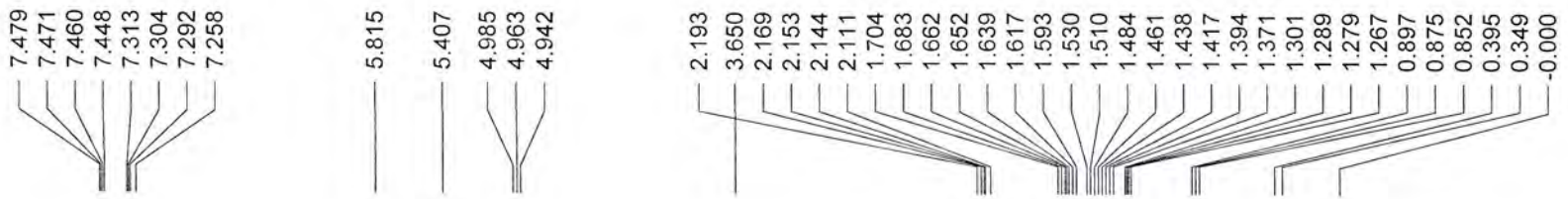
syl-8-184_1.1
2021-05-09 02:44:08.678
SOLVENT: CDCl3
NA = 16
F1 = 400.130035 MHz
0.98
3.00
1.95
6.94
5.19
2.12
16.49
3.02
10
8
6
4
2
0
PPM

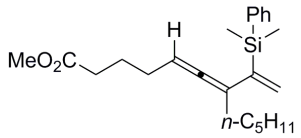

201.205
174.210
148.352
105.855
88.728
82.887
77.316
77.000
76.684
51.350
33.736
33.510
31.623
28.445
27.380
24.734
24.671
24.635
24.274
22.649
22.522
14.081


H
Bpin
MeO2C
CH3
n-C5H11
CH3

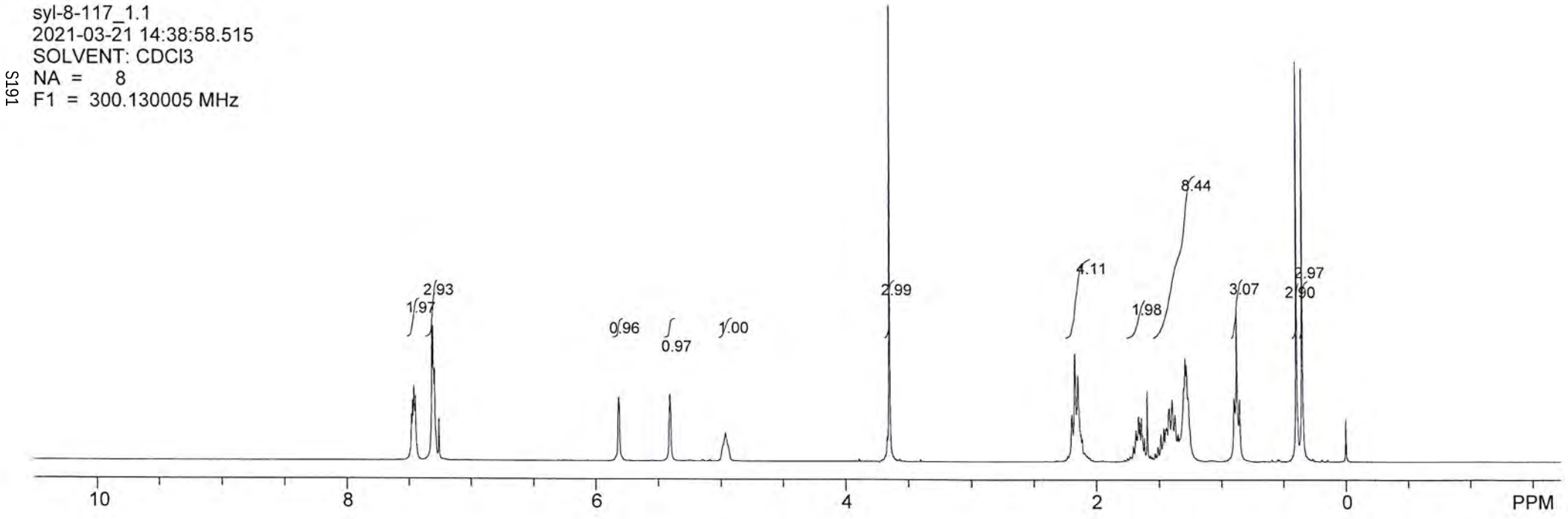
3xa


syl-8-184_2.1
2021-05-09 03:08:00.465
SOLVENT: CDCl3
NA = 512
F1 = 100.612869 MHz
200
150
100
50
0
PPM

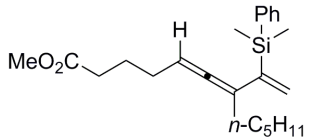

7.479
7.471
7.460
7.448
7.313
7.304
7.292
7.258
5.815
5.407
4.985
4.963
4.942
2.193
3.650
2.169
2.153
2.144
2.111
1.704
1.683
1.662
1.652
1.639
1.617
1.593
1.530
1.510
1.484
1.461
1.438
1.417
1.394
1.371
1.301
1.289
1.279
1.267
0.897
0.875
0.852
0.395
0.349
-0.000


Ph
H
Si
MeO2C
n-C5H11

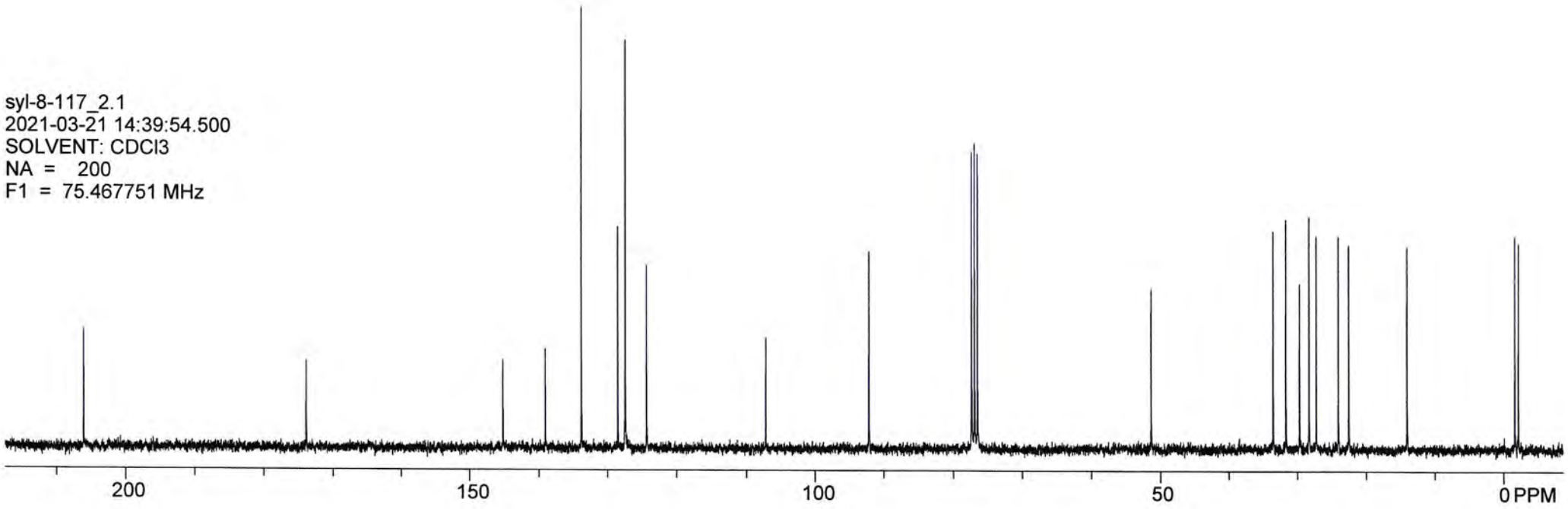
5aa

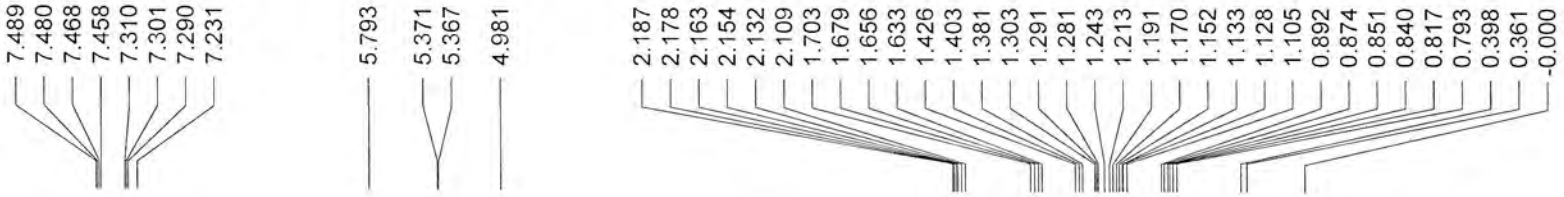

syl-8-117_1.1
2021-03-21 14:38:58.515
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
1.97
2.93
0.96
0.97
1.00
2.99
4.11
8.44
1.98
3.07
2.90
2.97
10
8
6
4
2
0
PPM

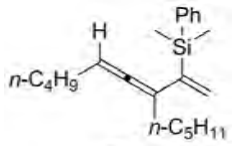
206.108
173.863
145.212
139.072
133.824
128.520
127.436
124.356
107.214
92.185
77.423
77.000
76.577
51.364
33.523
31.657
29.690
28.293
27.236
24.037
22.520
14.064
-1.636
-2.151



5aa

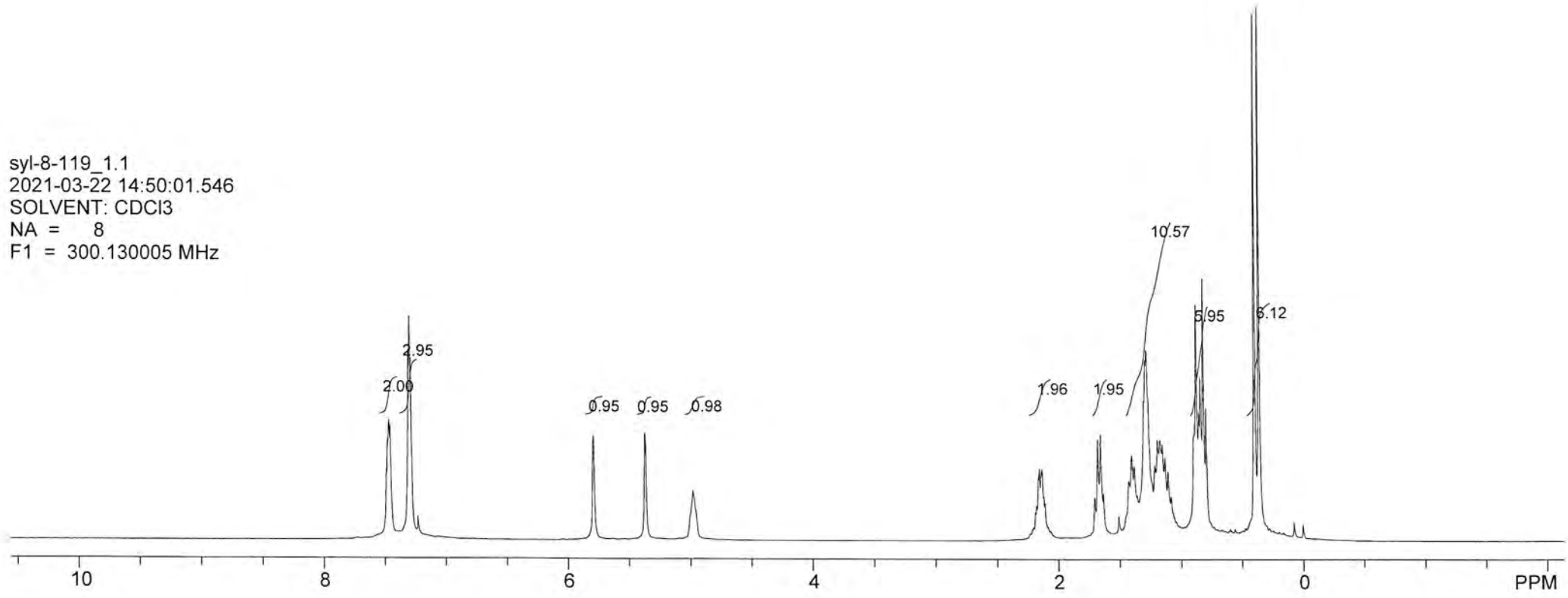




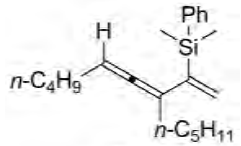


5ba

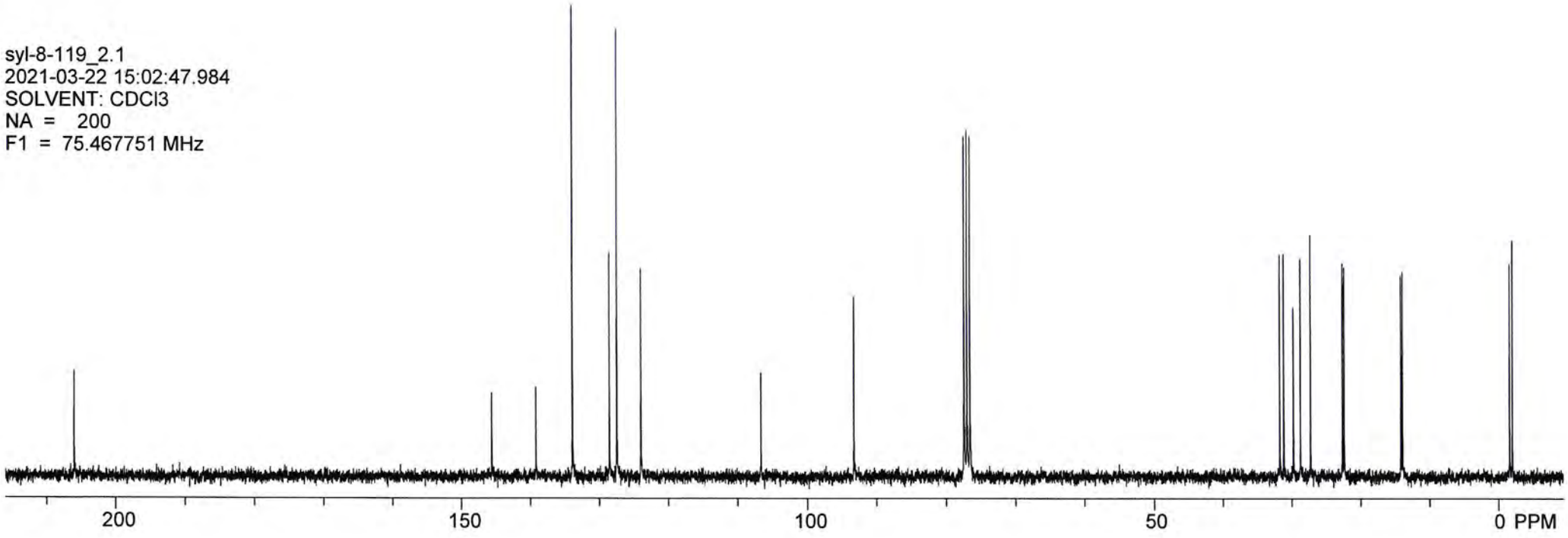
syl-8-119_1.1
2021-03-22 14:50:01.546
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz

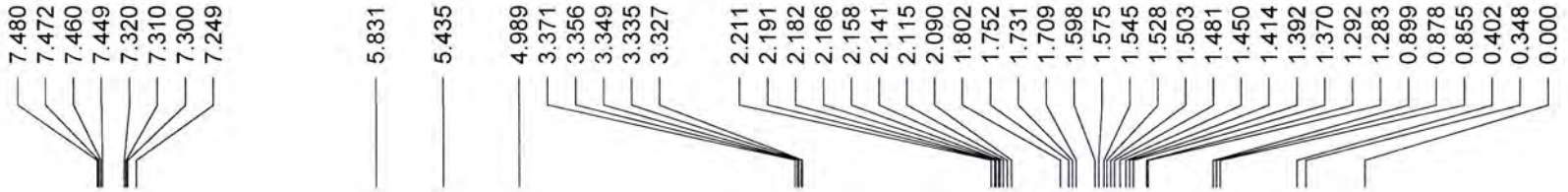


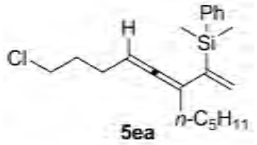
206.026
145.608
139.201
133.907
128.529
127.445
123.970
106.708
93.270
77.432
77.000
76.577
31.712
31.124
29.736
28.706
27.245
22.603
22.355
14.100
13.871
-1.627
-2.031



5ba

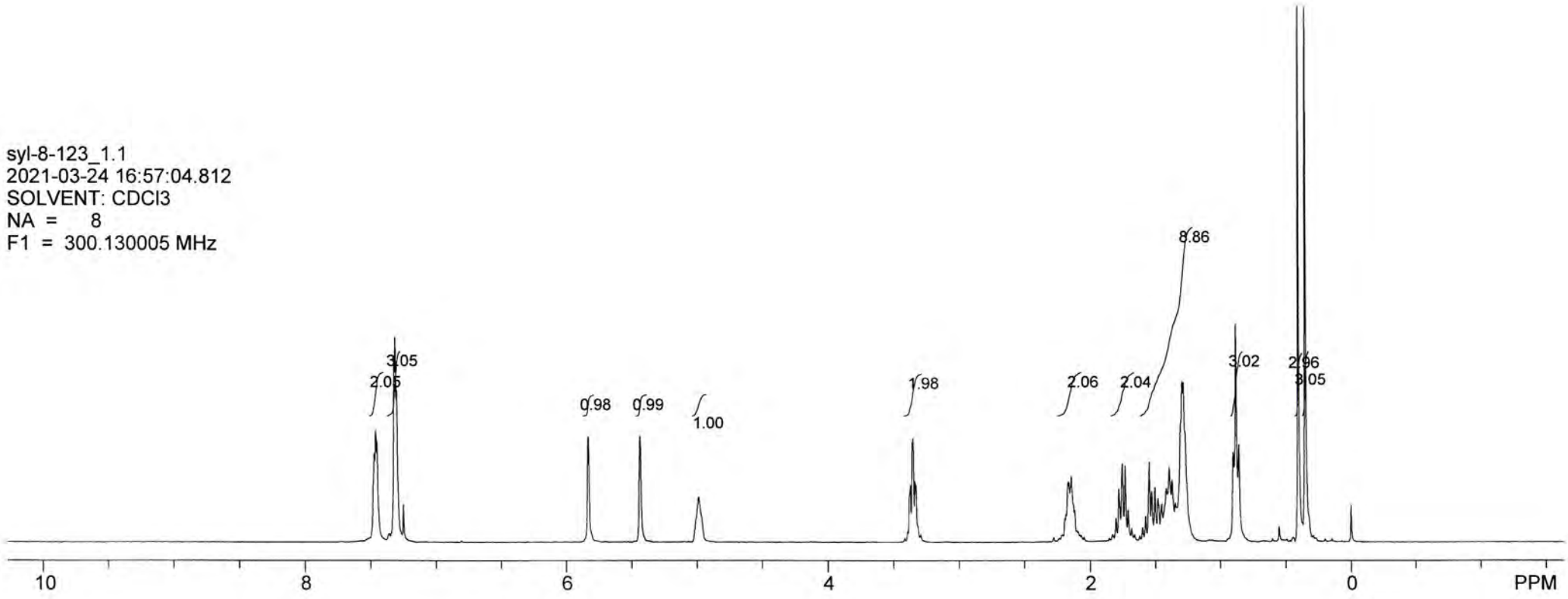




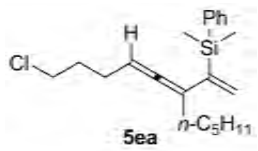


5ea

syl-8-123_1.1
2021-03-24 16:57:04.812
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz

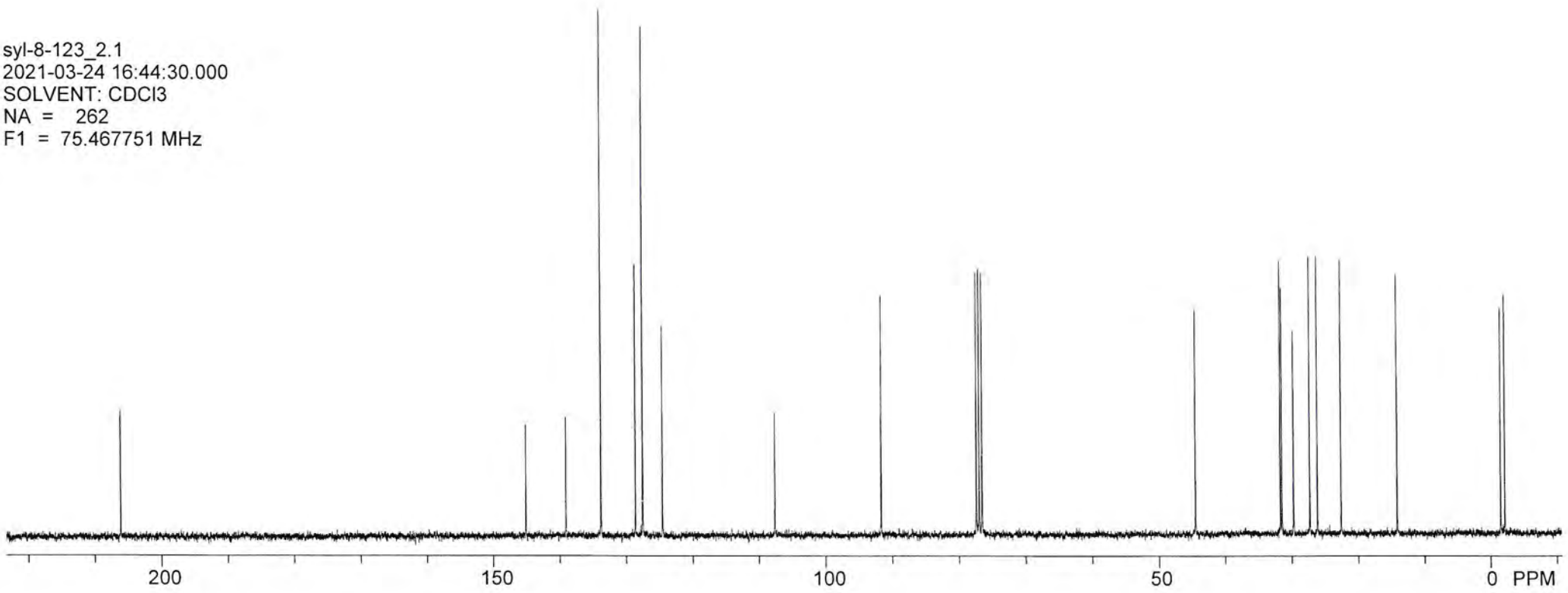


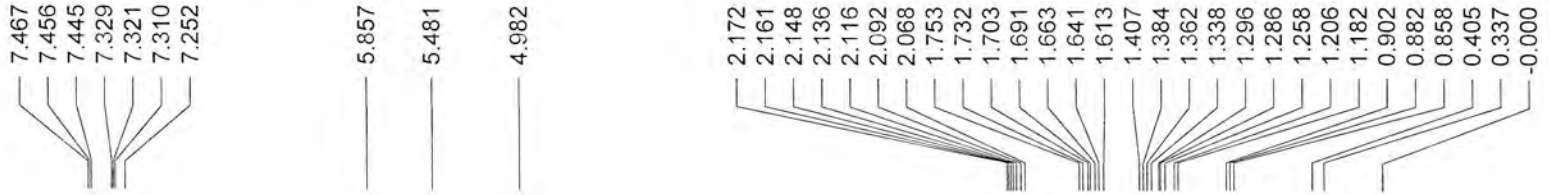
206.117
145.111
139.082
133.815
128.594
127.500
124.531
107.609
91.661
77.423
77.000
76.577
44.479
31.684
31.455
29.708
27.254
26.114
22.557
14.091
-1.553
-2.187

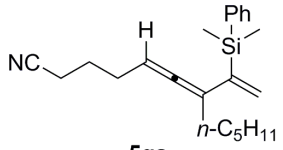


5ea

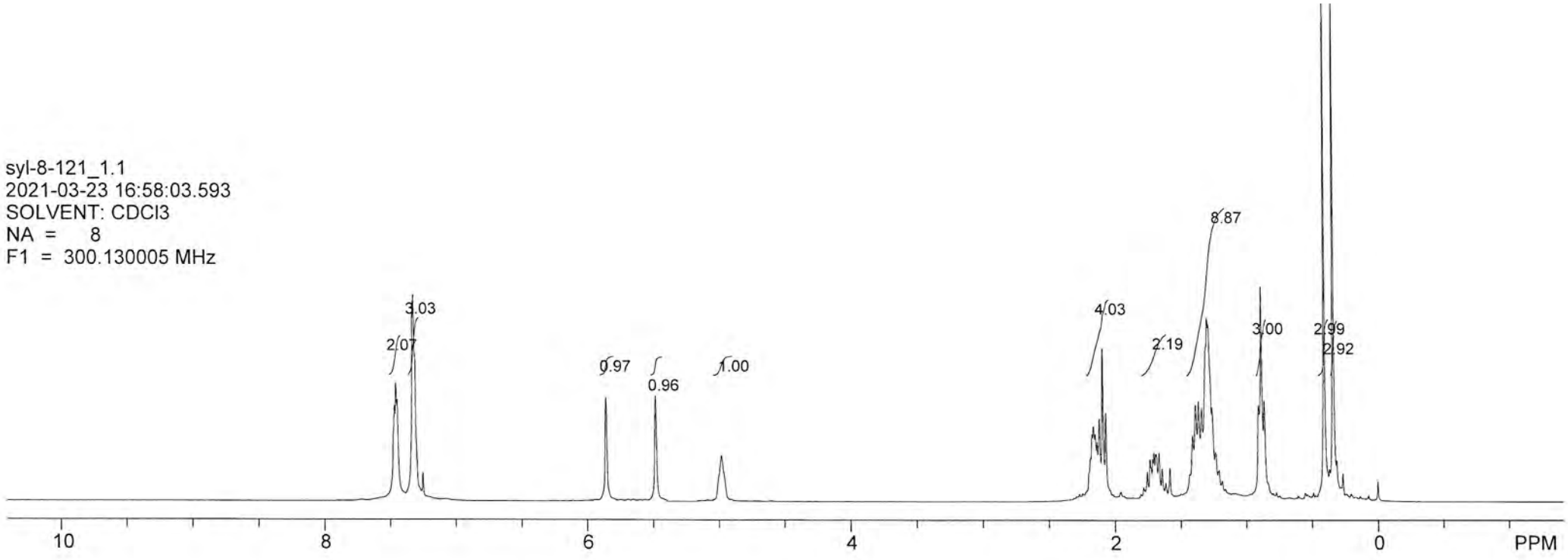
syl-8-123_2.1
2021-03-24 16:44:30.000
SOLVENT: CDCl3
NA = 262
F1 = 75.467751 MHz

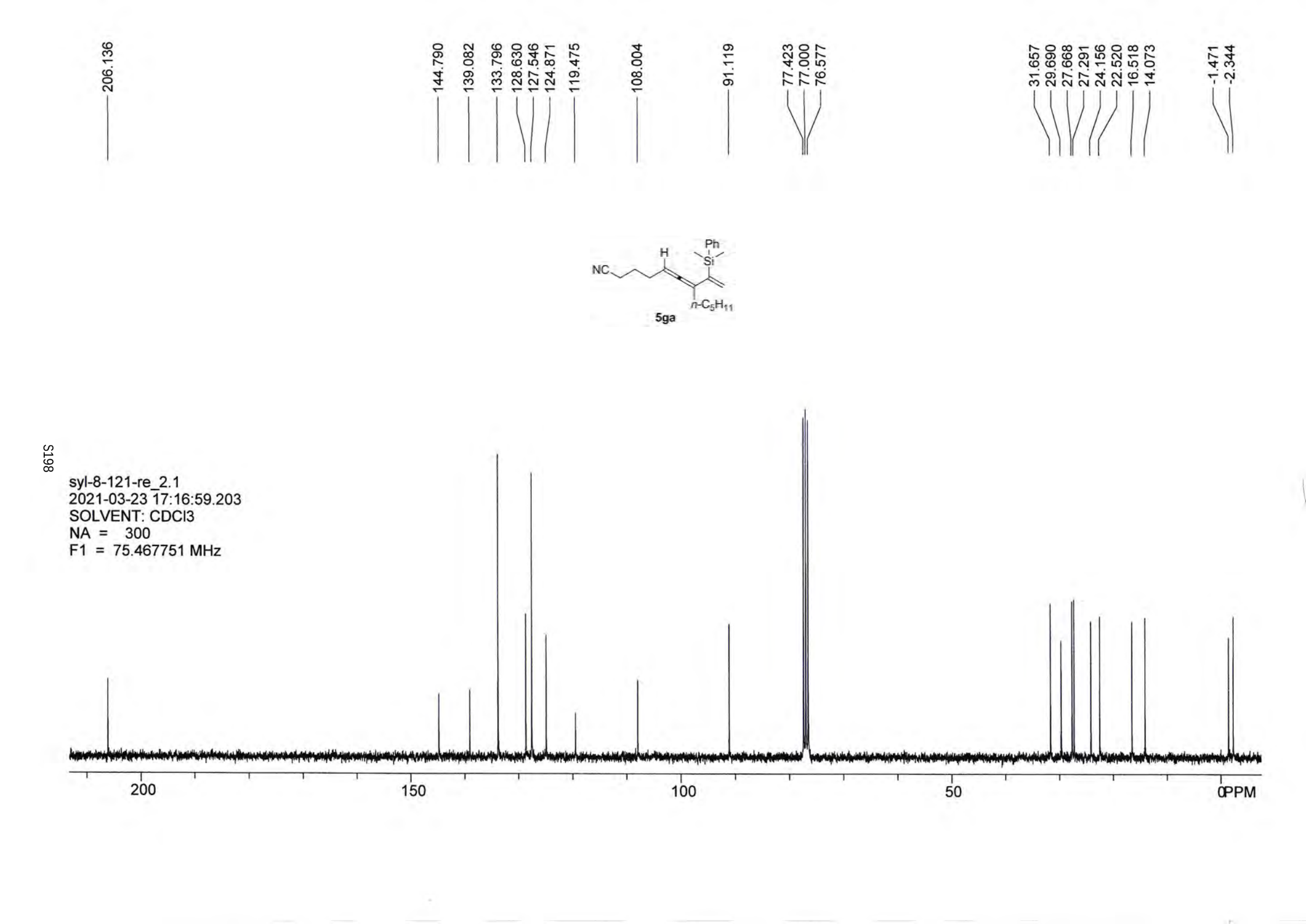



7.467
7.456
7.445
7.329
7.321
7.310
7.252
5.857
5.481
4.982
2.172
2.161
2.148
2.136
2.116
2.092
2.068
1.753
1.732
1.703
1.691
1.663
1.641
1.613
1.407
1.384
1.362
1.338
1.296
1.286
1.258
1.206
1.182
0.902
0.882
0.858
0.405
0.337
-0.000

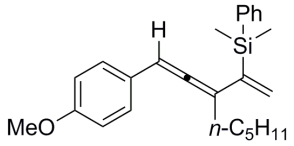

NC
H
Ph
Si
n-C5H11

5ga

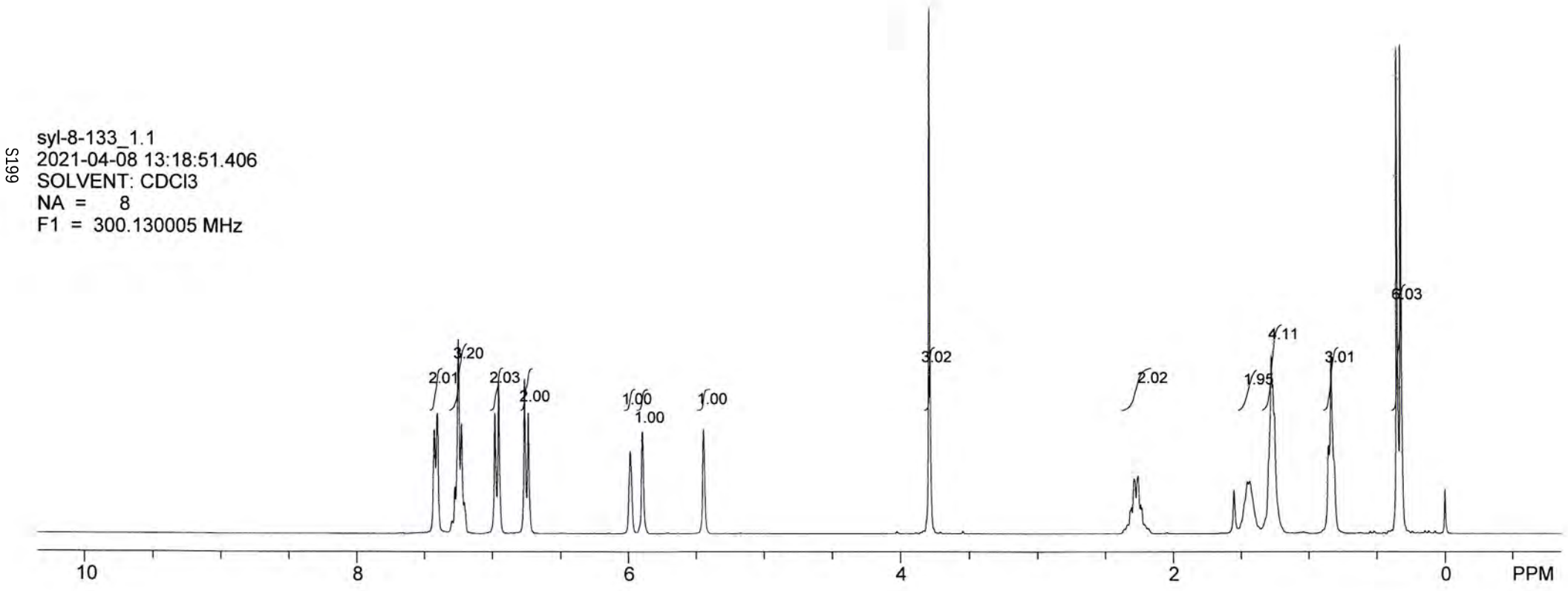

syl-8-121_1.1
2021-03-23 16:58:03.593
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
3.03
2.07
0.97
0.96
1.00
4.03
2.19
8.87
3.00
2.99
2.92
10
8
6
4
2
0
PPM


206.136
144.790
139.082
133.796
128.630
127.546
124.871
119.475
108.004
91.119
77.423
77.000
76.577
31.657
29.690
27.668
27.291
24.156
22.520
16.518
14.073
-1.471
-2.344
NC
H
Ph
Si
n-C5H11
5ga
syl-8-121-re_2.1
2021-03-23 17:16:59.203
SOLVENT: CDCl3
NA = 300
F1 = 75.467751 MHz
200
150
100
50
0PPM

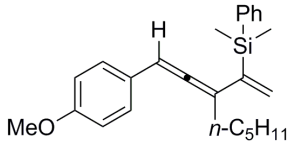

7.426
7.403
7.297
7.275
7.248
7.226
7.205
6.980
6.952
6.764
6.735
5.985
5.894
5.445
3.786
2.334
2.307
2.286
2.257
2.239
2.229
1.450
1.433
1.272
1.261
1.250
0.853
0.831
0.350
0.321
0.000


H
Ph
Si
MeO
n-C5H11

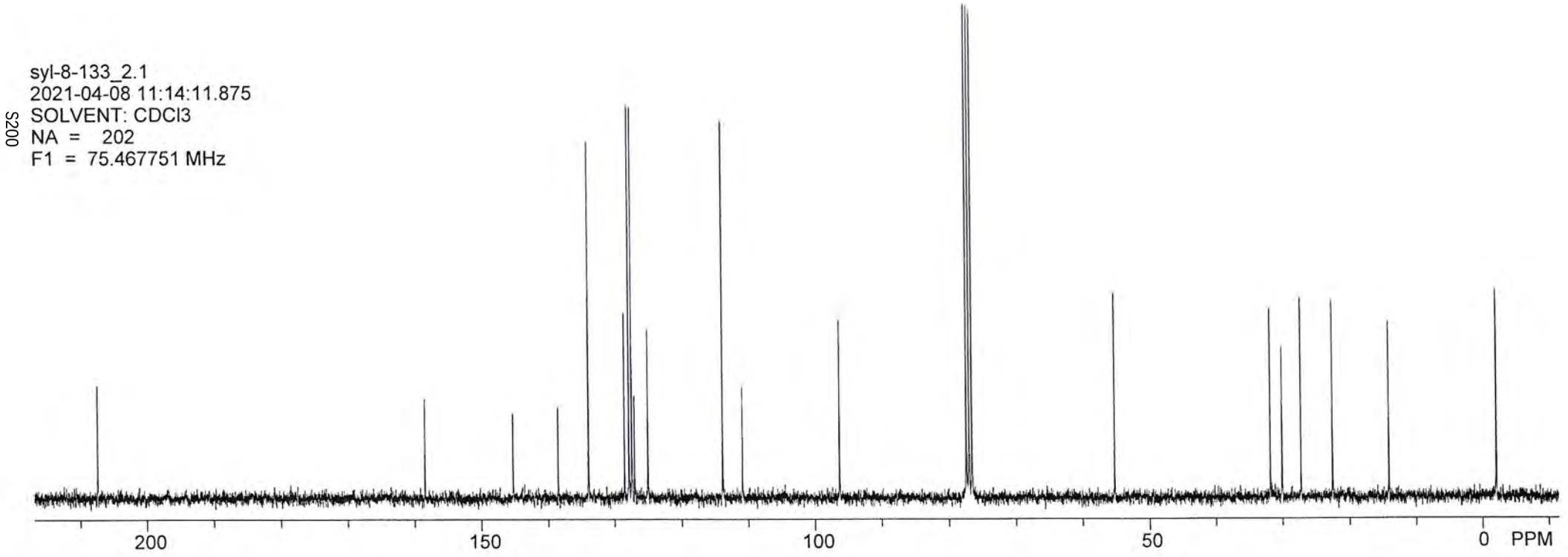
5ka


syl-8-133_1.1
2021-04-08 13:18:51.406
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
2.01
3.20
2.03
2.00
1.00
1.00
1.00
3.02
2.02
1.95
4.11
3.01
6.03
10
8
6
4
2
0
PPM

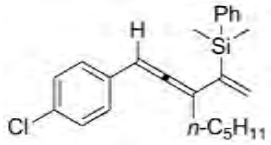
207.413
158.458
145.258
138.512
133.925
128.529
127.868
127.445
127.040
125.036
113.859
110.890
96.330
77.423
77.000
76.577
55.225
31.776
30.021
27.199
22.493
14.073
-2.096
-2.151



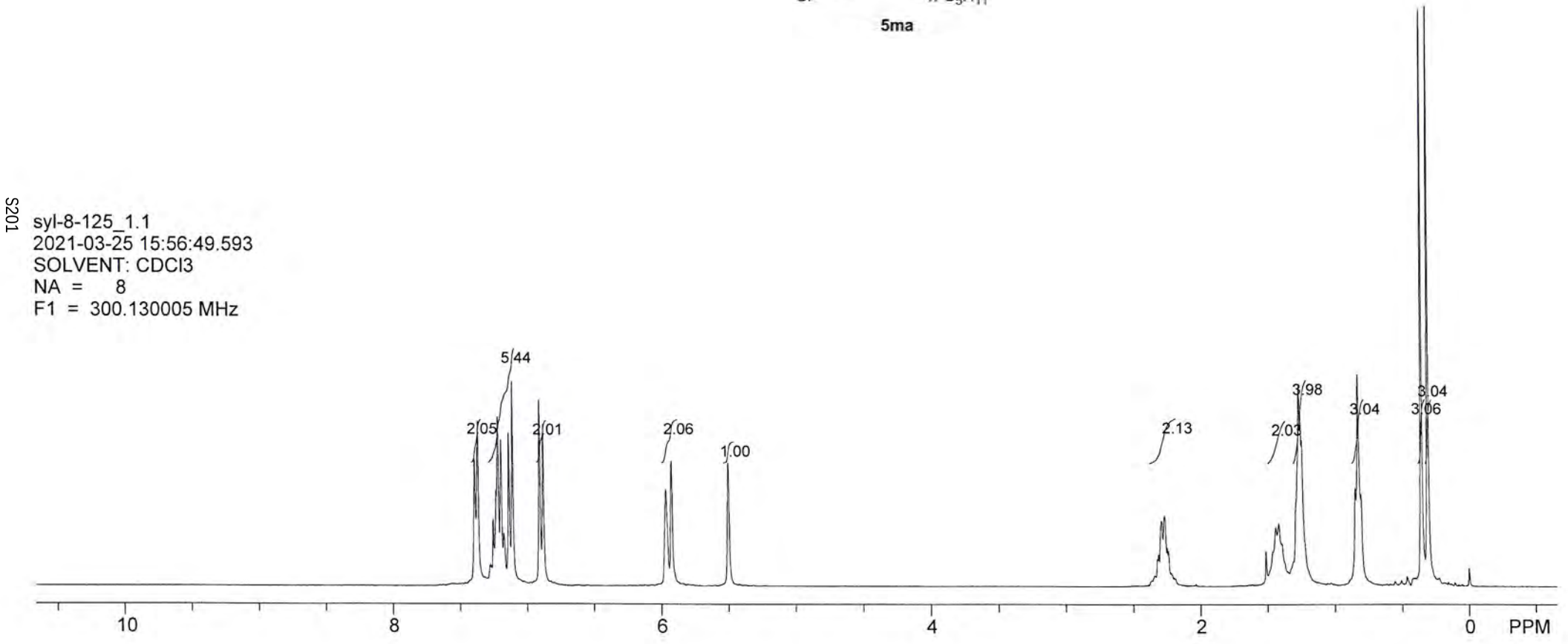
5ka



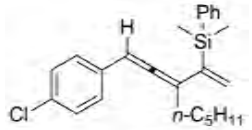

7.393
7.368
7.254
7.231
7.219
7.195
7.139
7.110
6.910
6.882
5.968
5.926
5.504
2.343
2.323
2.313
2.289
2.268
2.248
2.239
2.197
1.460
1.438
1.415
1.392
1.266
1.255
1.243
0.848
0.827
0.804
0.358
0.310
0.000


H
Ph
Si
Cl
n-C5H11

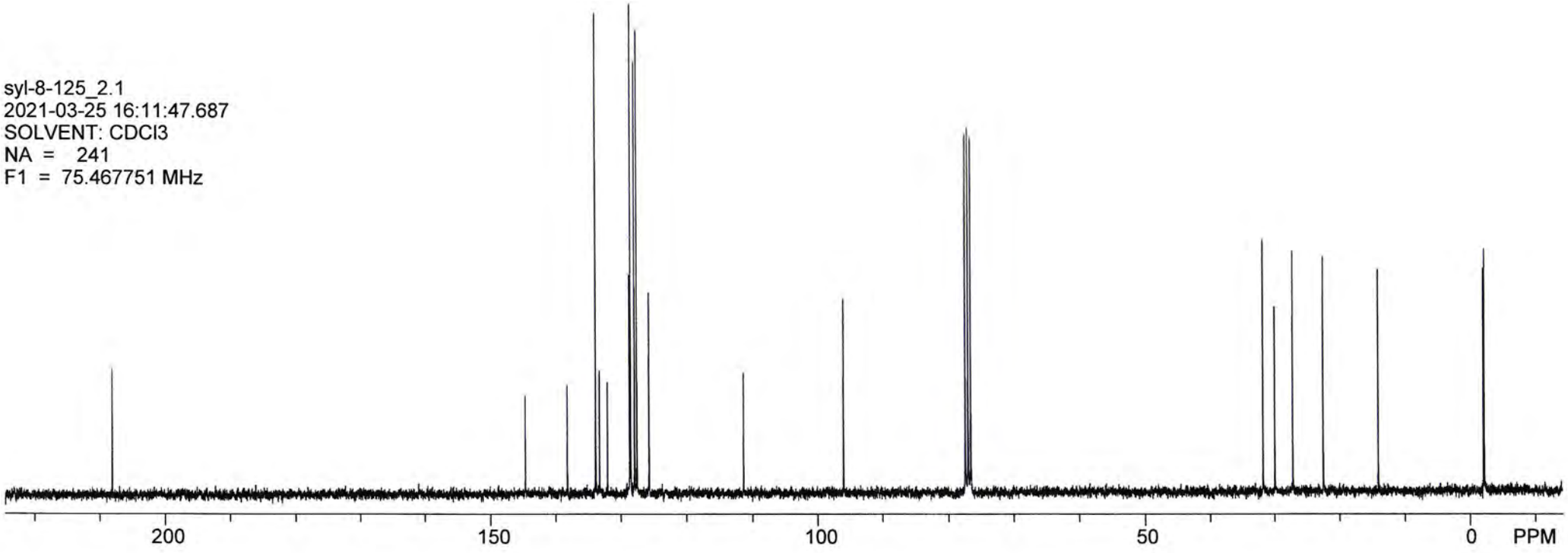
5ma


syl-8-125_1.1
2021-03-25 15:56:49.593
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
5.44
2.05
2.01
2.06
1.00
2.13
2.03
3.98
3.04
3.04
3.06
10
8
6
4
2
0
PPM

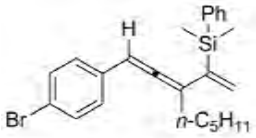
208.075
144.652
138.208
133.842
133.236
132.013
128.630
128.465
127.877
127.500
125.652
111.359
95.990
77.423
77.000
76.577
31.730
29.910
27.153
22.474
14.054
-2.059
-2.224



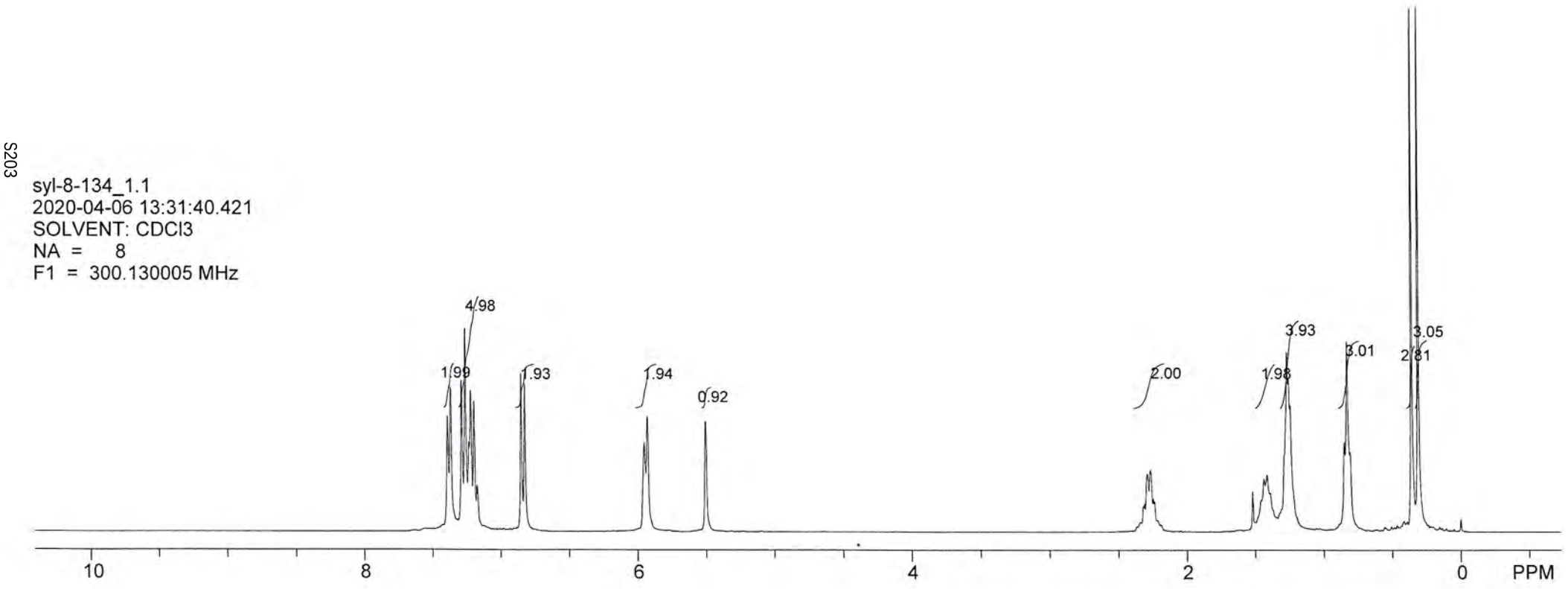
5ma

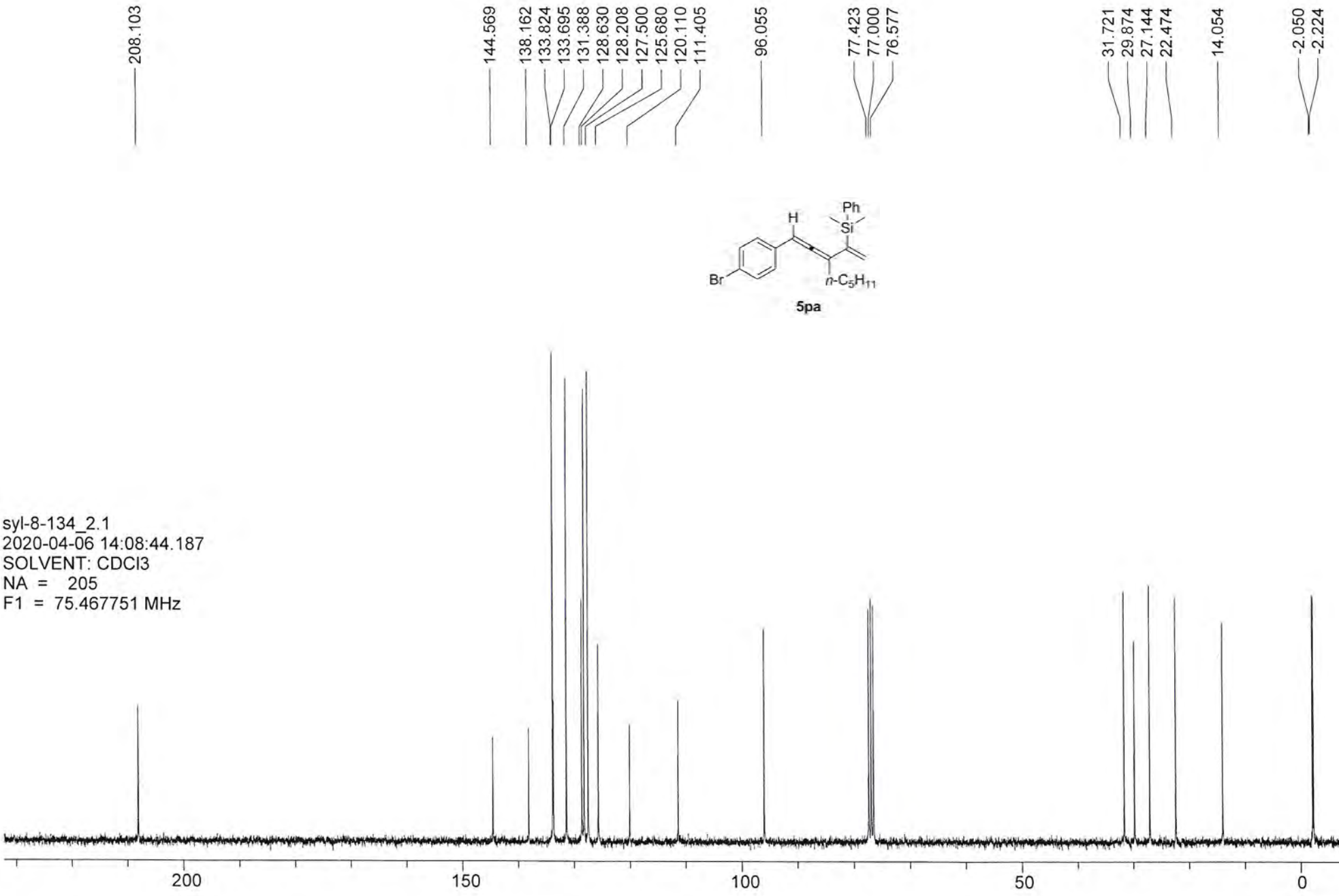



7.390
7.366
7.287
7.259
7.232
7.219
7.195
6.851
6.823
5.952
5.928
5.505
2.321
2.311
2.290
2.268
2.246
2.238
1.435
1.411
1.265
1.254
1.242
0.849
0.827
0.805
0.357
0.308
0.000

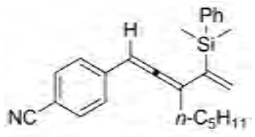

H
Ph
Si
Br
n-C5H11

5pa

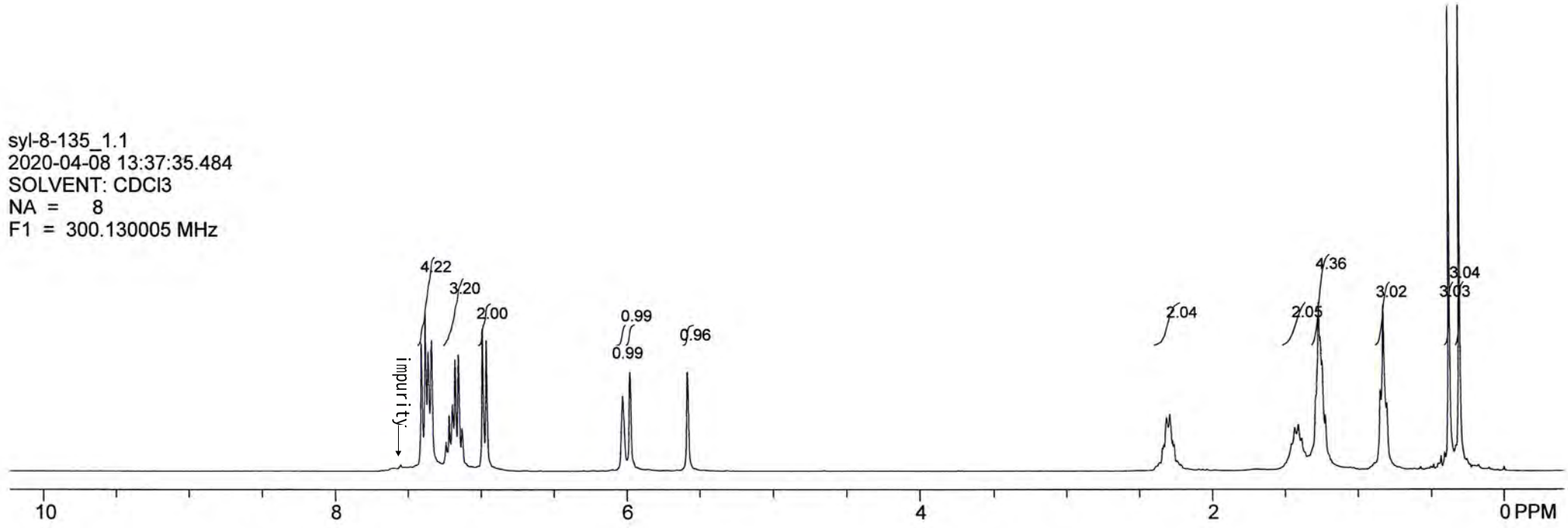

syl-8-134_1.1
2020-04-06 13:31:40.421
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
4.98
1.99
1.93
1.94
0.92
2.00
1.98
3.93
3.01
3.05
2.81
10
8
6
4
2
0
PPM


208.103
144.569
138.162
133.824
133.695
131.388
128.630
128.208
127.500
125.680
120.110
111.405
96.055
77.423
77.000
76.577
31.721
29.874
27.144
22.474
14.054
-2.050
-2.224
H
Ph
Si
Br
n-C5H11
5pa
syl-8-134_2.1
2020-04-06 14:08:44.187
SOLVENT: CDCl3
NA = 205
F1 = 75.467751 MHz
200
150
100
50
0
PPM

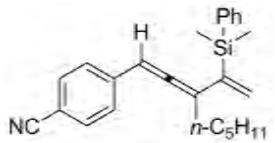

7.406
7.378
7.360
7.334
7.217
7.195
7.175
7.152
6.988
6.960
6.026
5.976
5.581
2.342
2.313
2.291
2.263
1.433
1.409
1.287
1.265
1.255
1.243
1.223
0.846
0.825
0.802
0.376
0.305
-0.000


Ph
H
Si
NC
n-C5H11

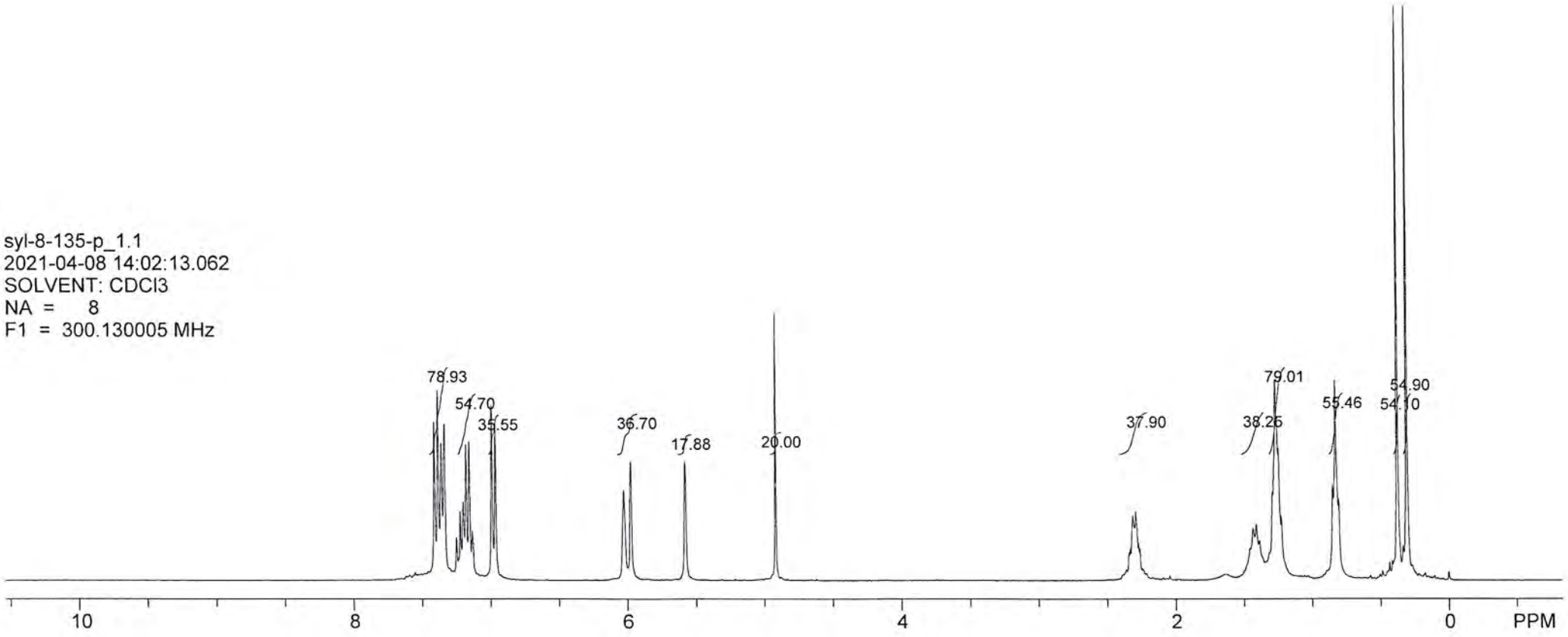
5ra


syl-8-135_1.1
2020-04-08 13:37:35.484
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
4.22
3.20
2.00
0.99
0.99
0.96
2.04
2.05
4.36
3.02
3.04
3.03
impurity
10
8
6
4
2
0 PPM

Purity (97%) is determined by CH_2Br_2 (7 μL, 0.1 mmol) as the internal standard in 68.5 mg of sample.



5ra



209.399

143.861
139.826
137.841
133.695
132.022
128.612
127.472
126.939
126.305
119.154
111.791
109.392

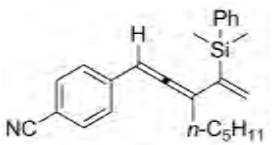
96.027

77.423
77.000
76.577

31.620
29.763
27.052
22.382

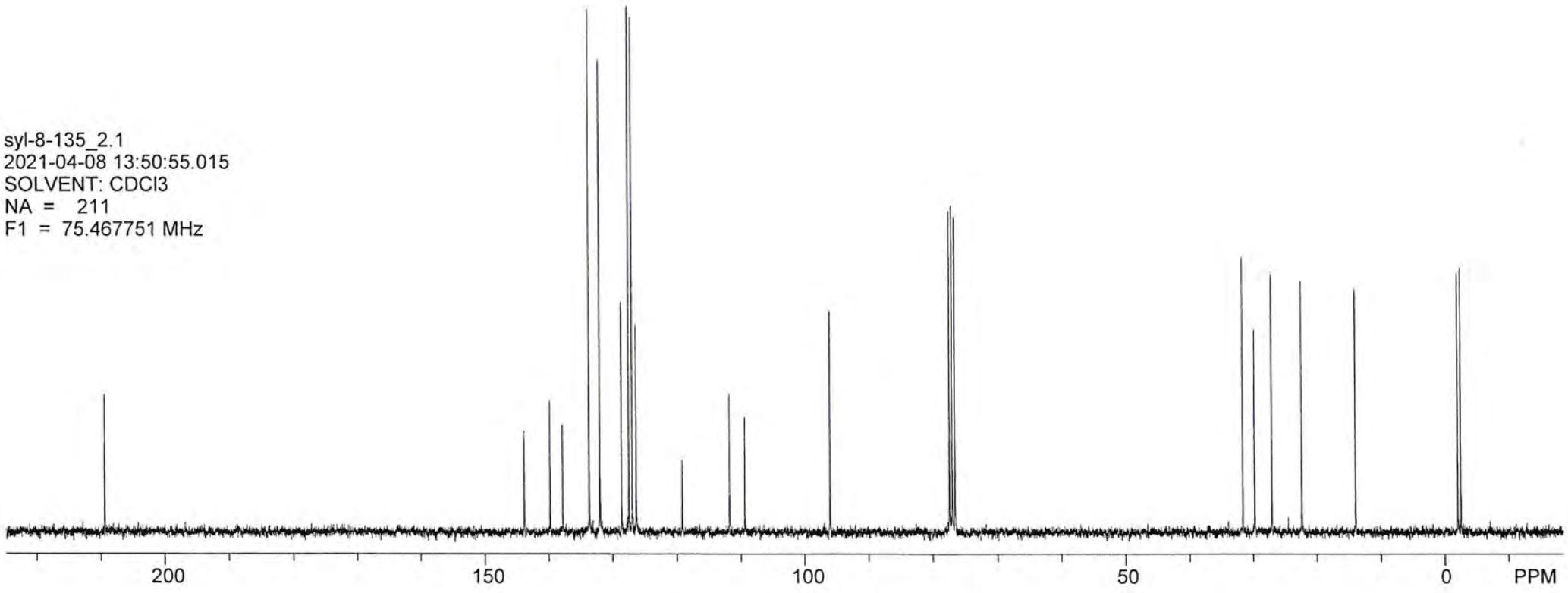
13.972

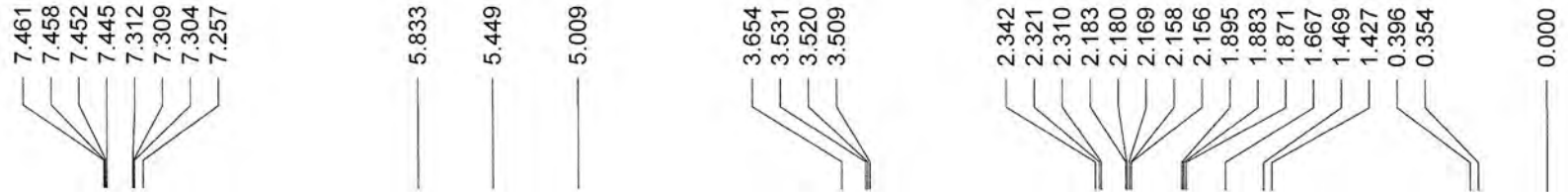
-1.994
-2.482

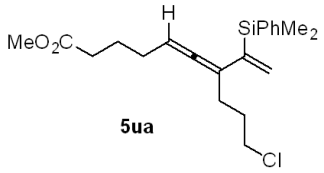


5ra

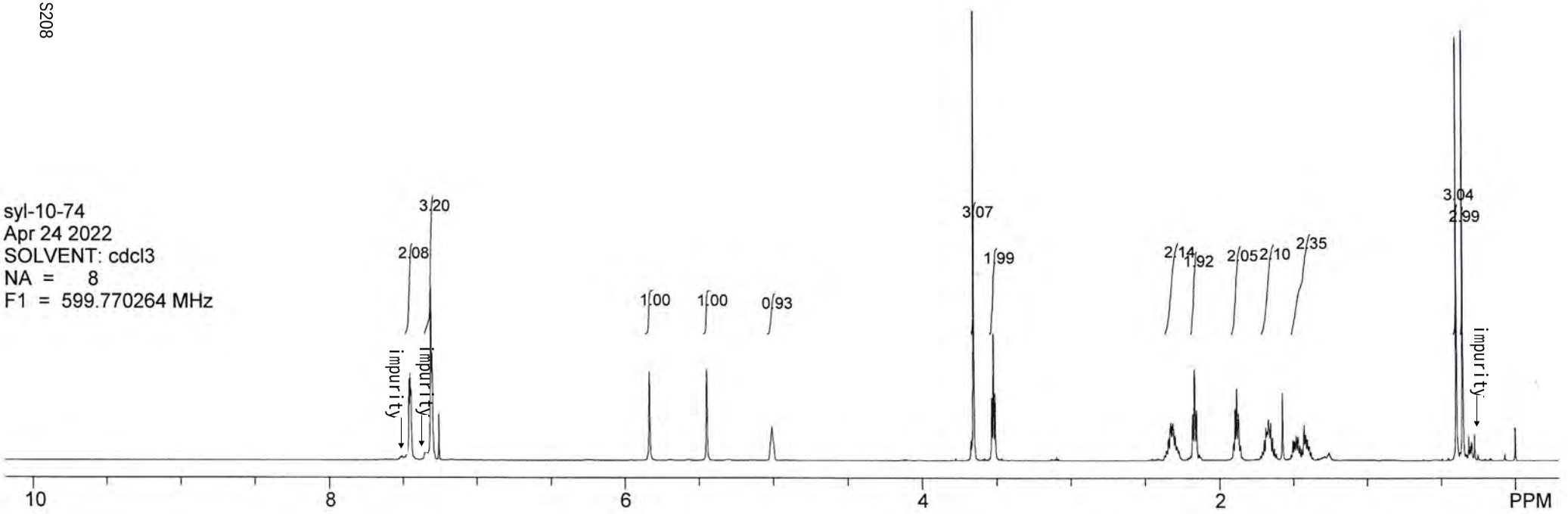
syl-8-135_2.1
2021-04-08 13:50:55.015
SOLVENT: CDCl3
NA = 211
F1 = 75.467751 MHz



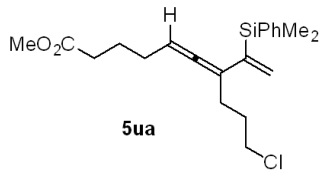

7.461
7.458
7.452
7.445
7.312
7.309
7.304
7.257
5.833
5.449
5.009
3.654
3.531
3.520
3.509
2.342
2.321
2.310
2.183
2.180
2.169
2.158
2.156
1.895
1.883
1.871
1.667
1.469
1.427
0.396
0.354
0.000


H
MeO2C
SiPhMe2
Cl

5ua

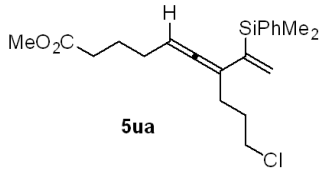

syl-10-74
Apr 24 2022
SOLVENT: cdcl3
NA = 8
F1 = 599.770264 MHz
3.20
2.08
1.00
1.00
0.93
3.07
1.99
2.14
1.92
2.05
2.10
2.35
3.04
2.99
impurity
impurity
impurity
10
8
6
4
2
PPM

205.799
173.789
144.914
138.828
133.807
128.656
127.529
124.777
105.890
93.012
77.207
77.000
76.785
51.452
44.737
33.530
30.541
28.264
26.992
24.041
-1.691
-2.190



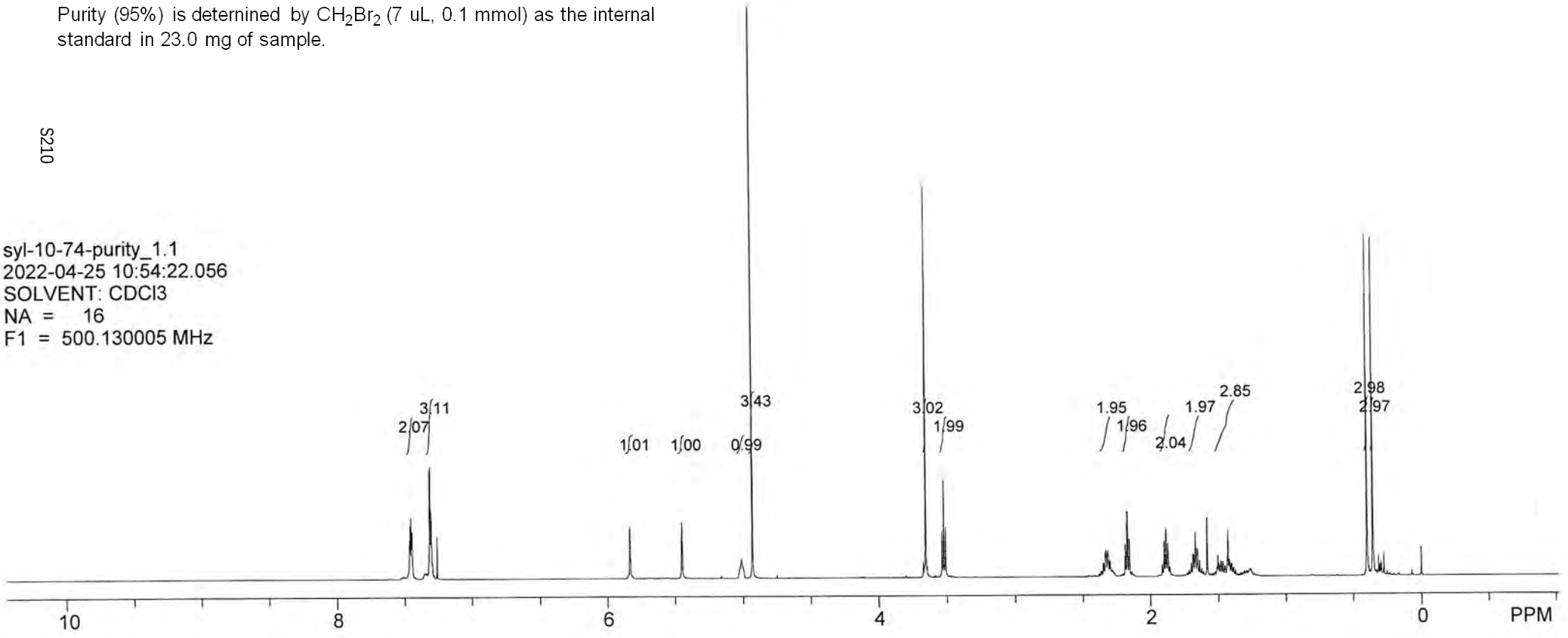
5ua

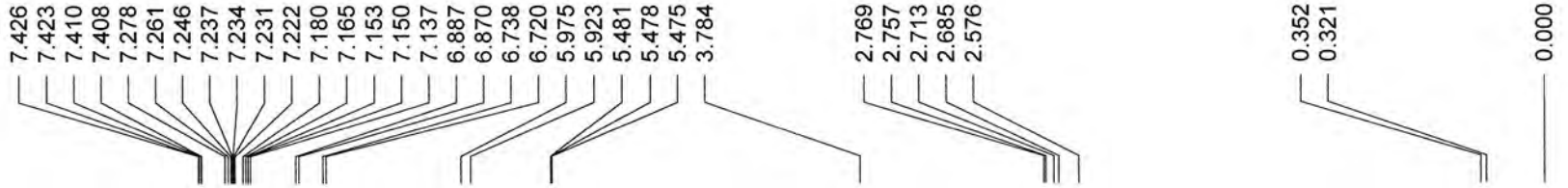
syl-10-74
Apr 24 2022
SOLVENT: cdcl3
NA = 512
F1 = 150.827637 MHz

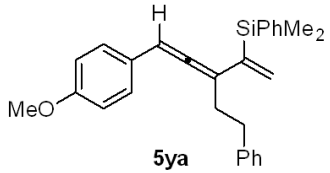


5ua

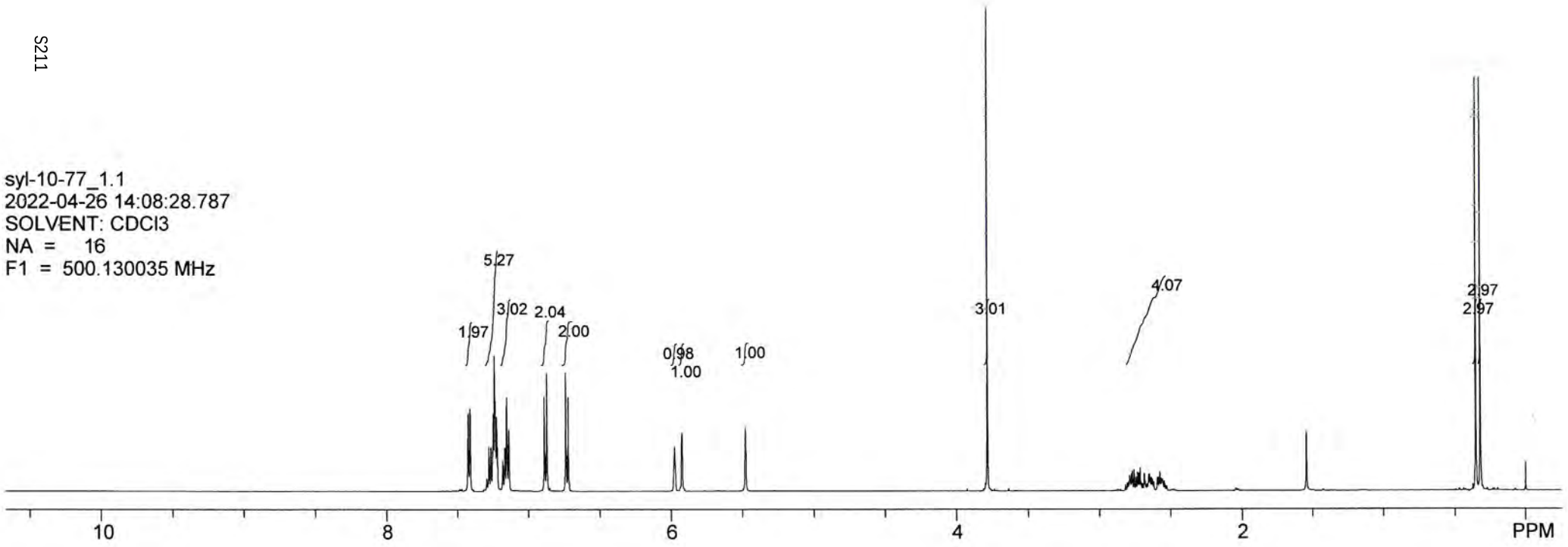
Purity (95%) is deternined by CH_2Br_2 (7 uL, 0.1 mmol) as the internal standard in 23.0 mg of sample.



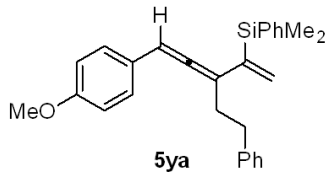

7.426
7.423
7.410
7.408
7.278
7.261
7.246
7.237
7.234
7.231
7.222
7.180
7.165
7.153
7.150
7.137
6.887
6.870
6.738
6.720
5.975
5.923
5.481
5.478
5.475
3.784
2.769
2.757
2.713
2.685
2.576
0.352
0.321
0.000


H
SiPhMe2
MeO
Ph

5ya

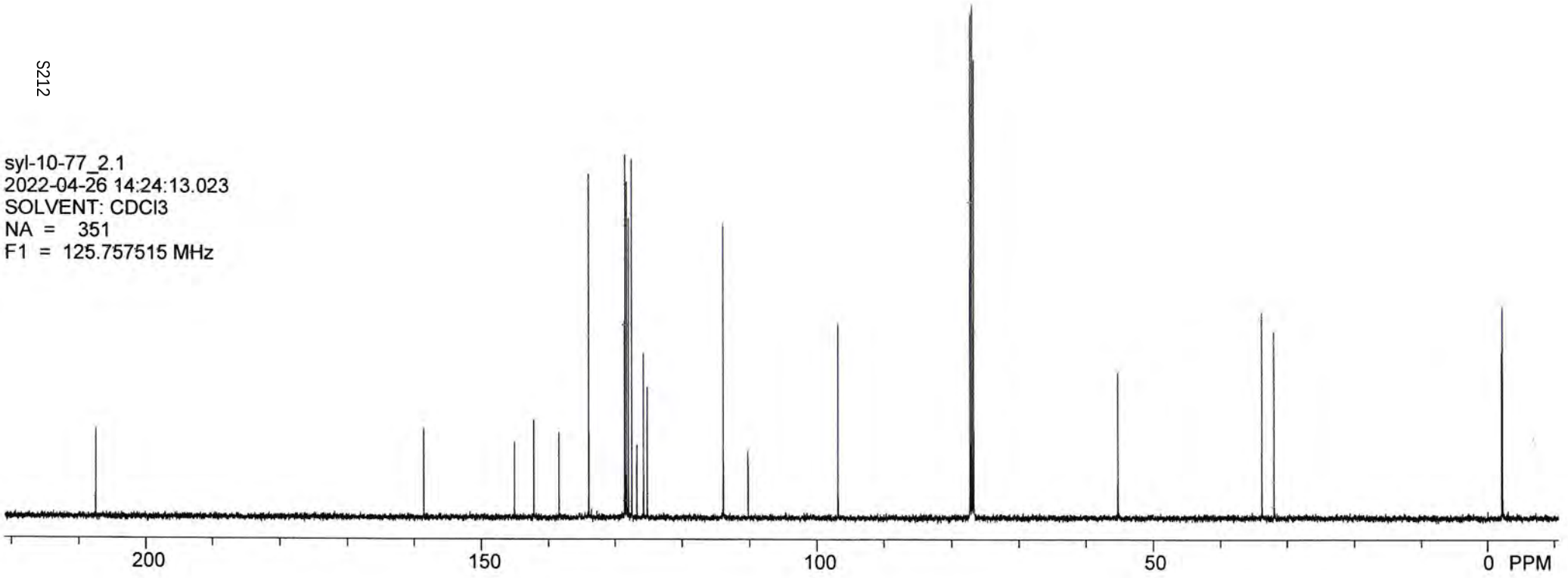

syl-10-77_1.1
2022-04-26 14:08:28.787
SOLVENT: CDCl3
NA = 16
F1 = 500.130035 MHz
5.27
3.02
2.04
2.00
1.97
0.98
1.00
1.00
3.01
4.07
2.97
2.97
10
8
6
4
2
PPM

207.499
158.554
145.031
142.135
138.402
133.934
128.584
128.483
128.262
127.977
127.490
126.717
125.734
125.173
113.893
110.215
96.830
77.257
77.000
76.752
55.258
33.764
31.971
-2.118
-2.238

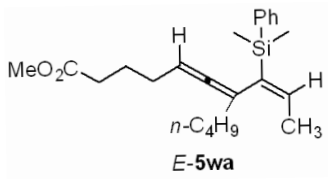


5ya

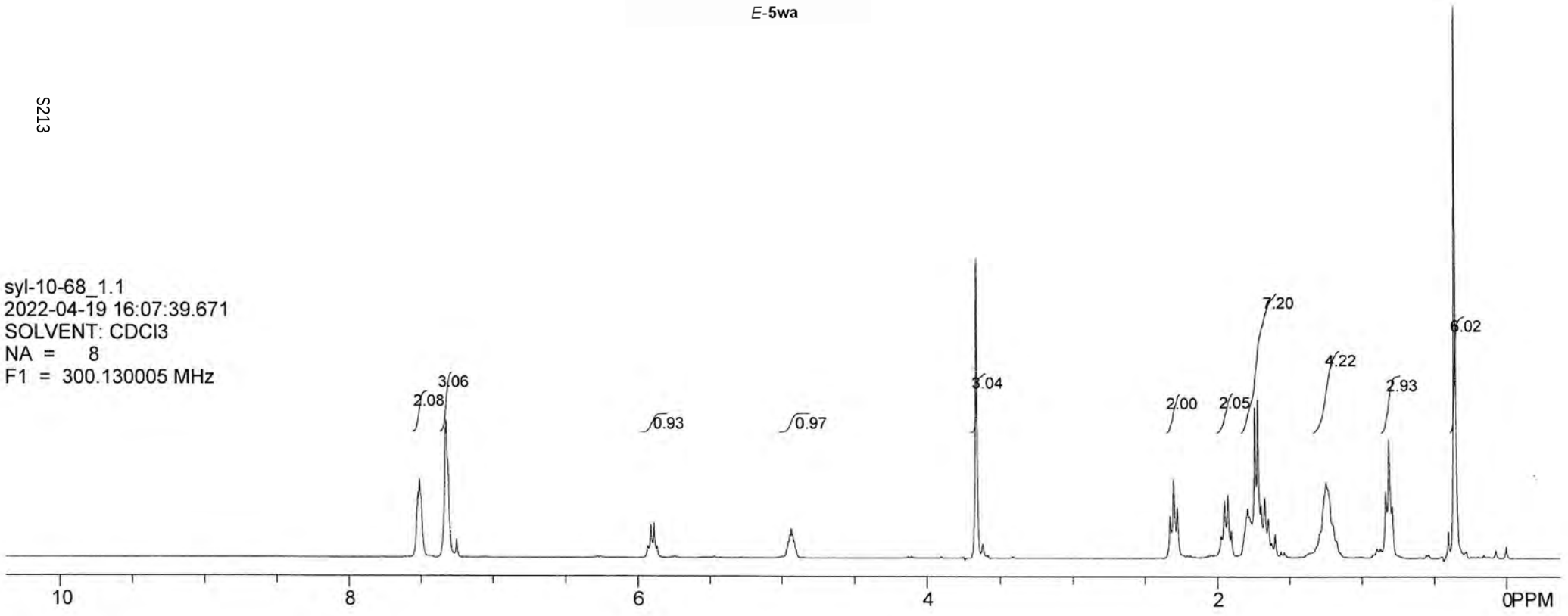
syl-10-77_2.1
2022-04-26 14:24:13.023
SOLVENT: CDCl3
NA = 351
F1 = 125.757515 MHz

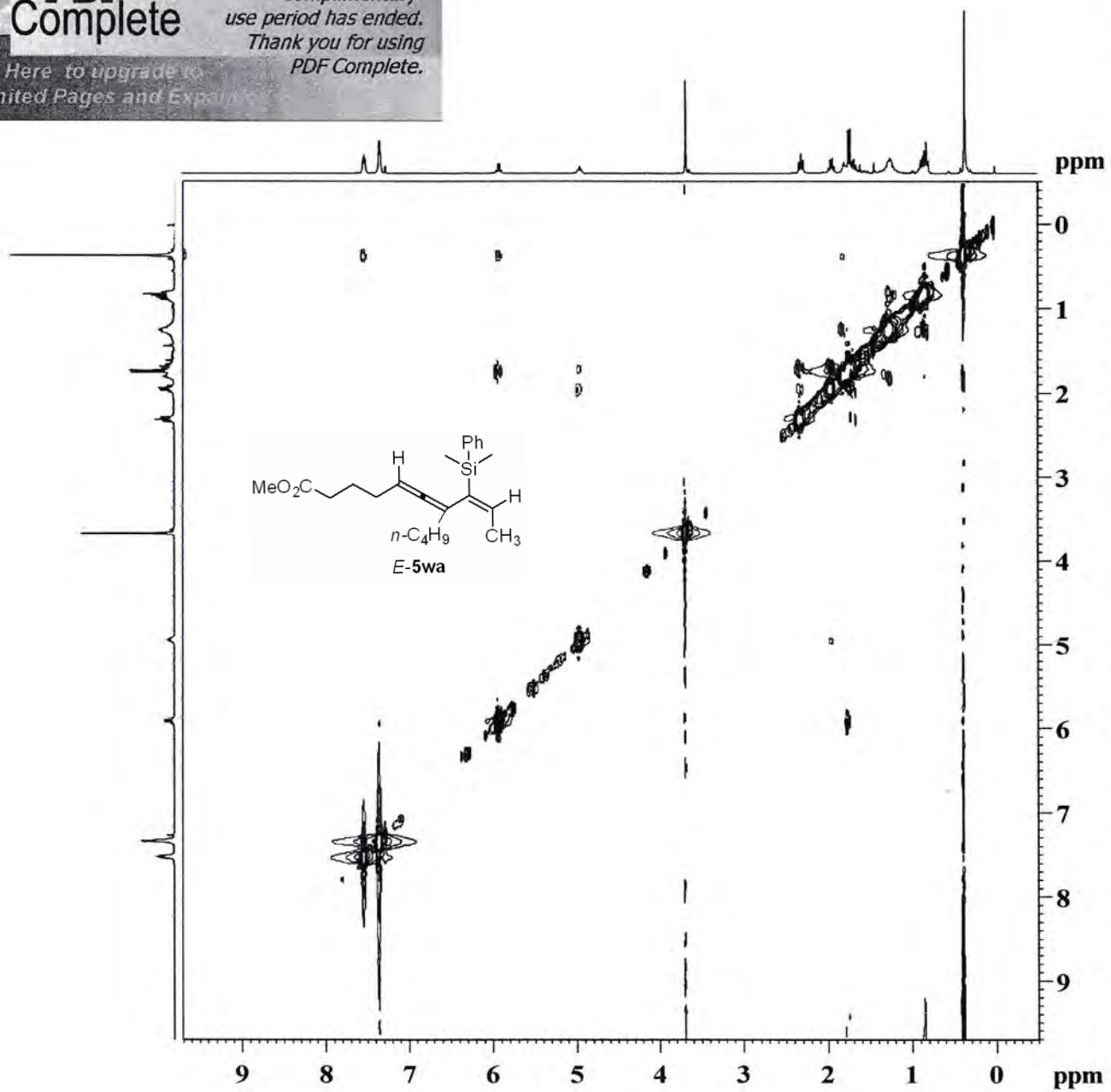



7.521
7.510
7.330
7.324
7.253
5.931
5.910
5.888
5.866
4.943
4.932
4.922
3.660
2.326
2.300
2.275
1.973
1.950
1.926
1.903
1.739
1.717
1.670
1.646
1.243
1.231
0.832
0.810
0.787
0.356
-0.000


Ph
Si
H
MeO2C
H
n-C4H9
CH3

E-**5wa**


syl-10-68_1.1
2022-04-19 16:07:39.671
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
2.08
3.06
0.93
0.97
3.04
2.00
2.05
7.20
4.22
2.93
6.02
10
8
6
4
2
0PPM



BRUKER

Current Data Parameters
NAME syl-10-68-noe
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220421
Time 23.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG noesygpph
TD 2048
SOLVENT CDCl3
NS 64
DS 4
SWH 3063.726 Hz
FIDRES 1.495960 Hz
AQ 0.3342836 sec
RG 203
DW 163.200 usec
DE 6.50 usec
TE 300.0 K
d0 0.00014537 sec
D1 2.00000000 sec
D16 0.00010000 sec
D8 0.50000000 sec
IN0 0.00032640 sec
ST1CNT 128
TAU 0.24840000 sec

CHANNEL f1
NUC1 1H
P1 14.00 usec
p2 28.00 usec
PL1 1.00 dB
SFO1 300.1313815 MHz

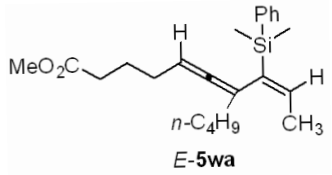
GRADIENT CHANNEL
GPNAM1 SINE.100
GPNAM2 SINE.100
GPZ1 80.00 %
GPZ2 40.00 %
P16 1500.00 usec

F1 - Acquisition parameters
ND0 1
TD 171
SFO1 300.1314 MHz
FIDRES 17.916523 Hz
SW 10.208 ppm
FnMODE States-TPPI

F2 - Processing parameters
SI 1024
SF 300.1300000 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.00

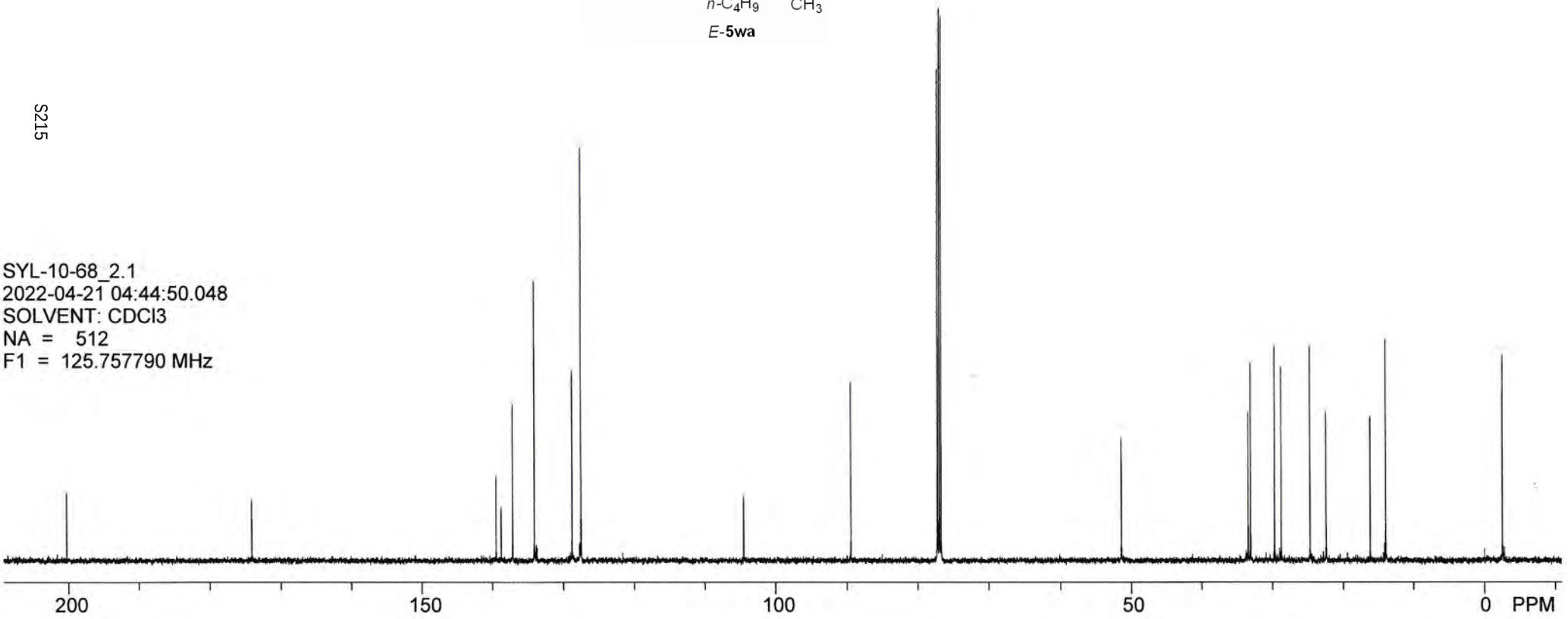
F1 - Processing parameters
SI 1024
MC2 States-TPPI
SF 300.1300000 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

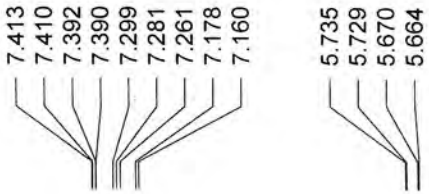
200.255
174.118
139.469
138.770
137.133
134.054
128.749
127.499
104.534
89.457
77.248
77.000
76.743
51.406
33.405
33.074
29.691
28.763
24.672
22.410
16.140
13.934
-2.541
-2.559

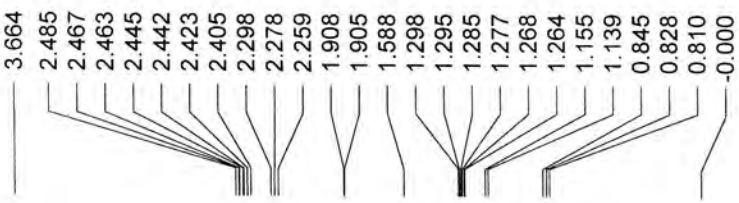


E-**5wa**

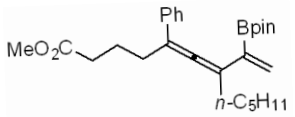
SYL-10-68_2.1
2022-04-21 04:44:50.048
SOLVENT: CDCl3
NA = 512
F1 = 125.757790 MHz



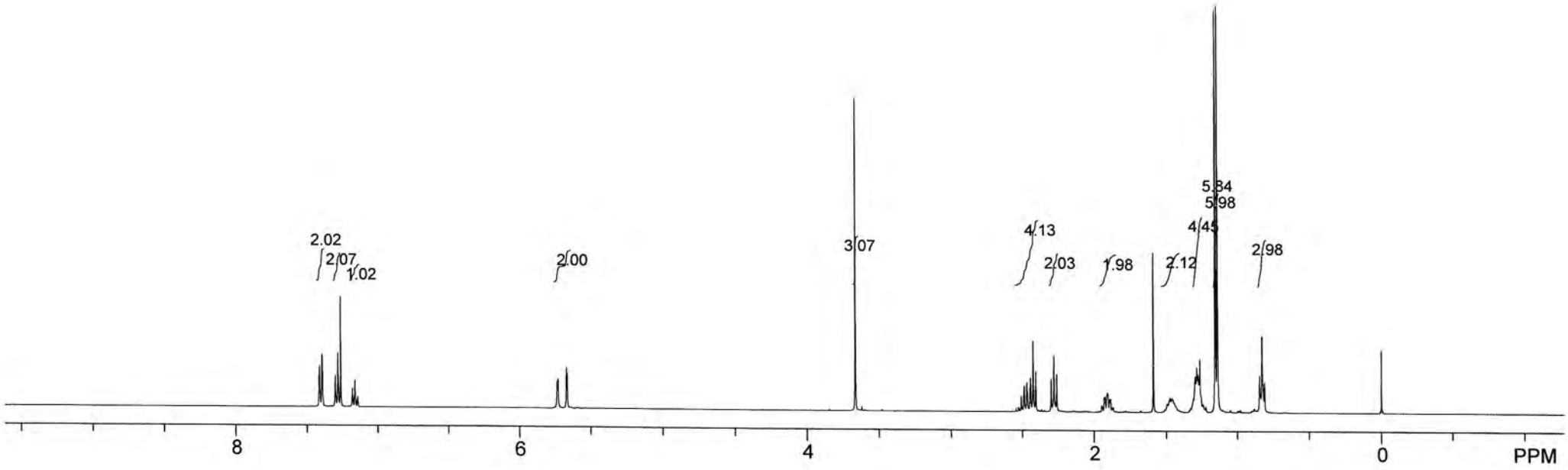


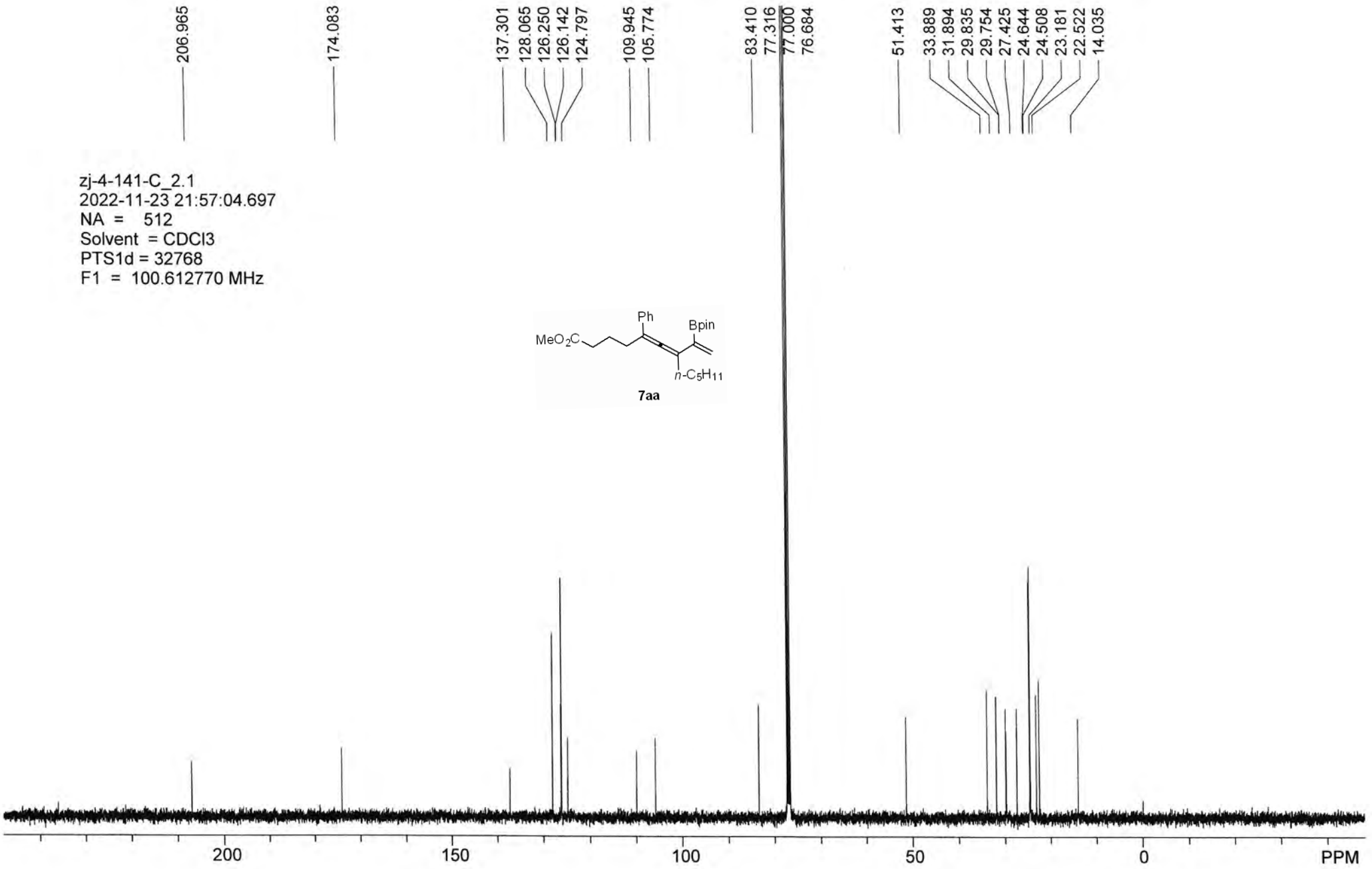


zj-4-141-H_1.1
2022-11-23 21:32:36.532
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz

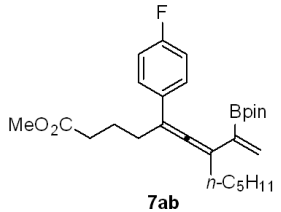


7aa

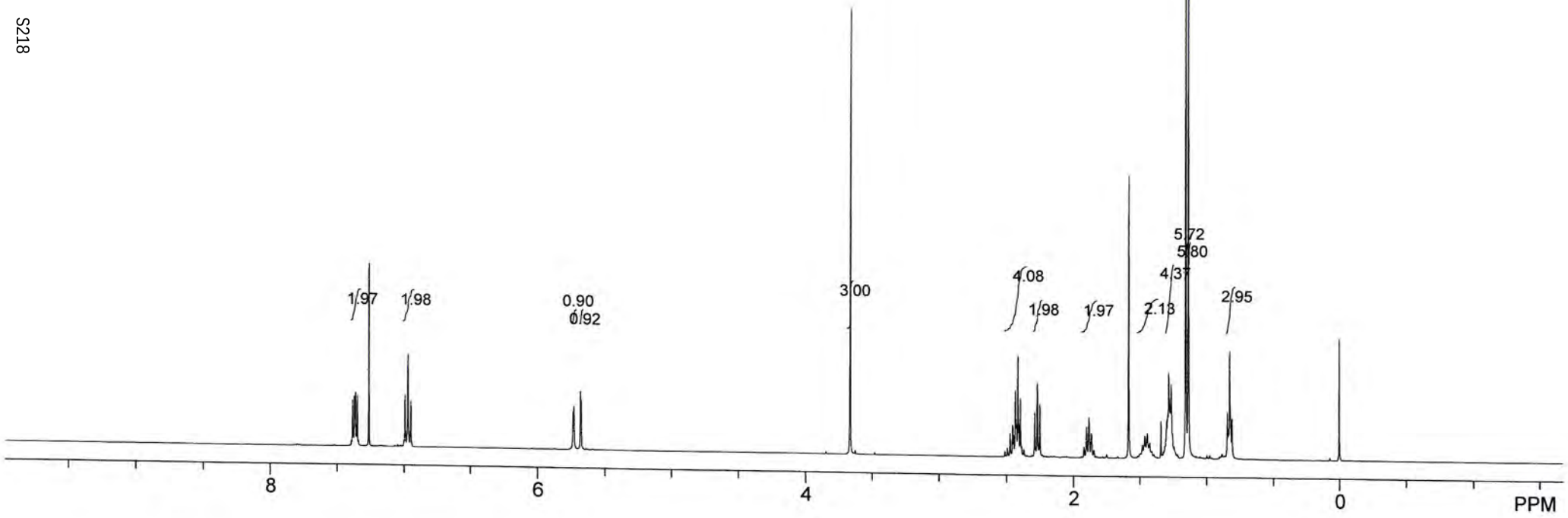



206.965
174.083
137.301
128.065
126.250
126.142
124.797
109.945
105.774
83.410
77.316
77.000
76.684
51.413
33.889
31.894
29.835
29.754
27.425
24.644
24.508
23.181
22.522
14.035
zj-4-141-C_2.1
2022-11-23 21:57:04.697
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
Ph
Bpin
MeO2C
n-C5H11
7aa
200
150
100
50
0
PPM

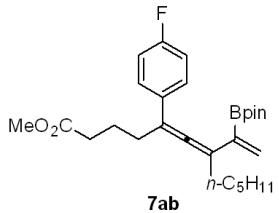
zj-4-148-F-pdt-H_1.1
2022-11-27 11:12:33.395
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz



7ab



206.754
206.736
174.026
162.571
160.622
133.245
133.217
127.646
127.582
125.007
114.941
114.775
110.105
104.938
83.426
77.257
77.000
76.752
51.433
33.782
31.852
29.967
29.700
27.384
24.616
24.534
23.100
22.502
14.017

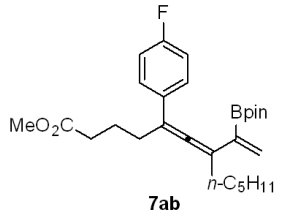


7ab

syl-10-128_2.1
2022-06-04 13:36:52.042
SOLVENT: CDCl3
NA = 512
F1 = 125.757790 MHz

-0.000

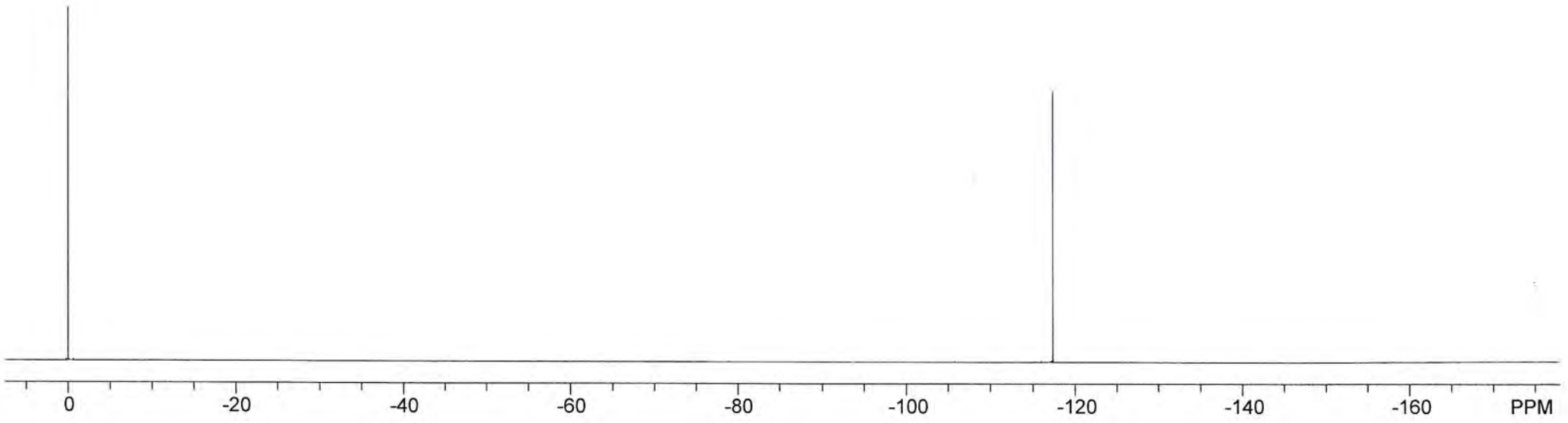
-117.373



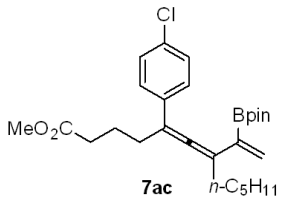
7ab

syl-10-128_1.1
2022-06-08 18:20:25.526
SOLVENT: CDCl3
NA = 64
F1 = 376.498352 MHz

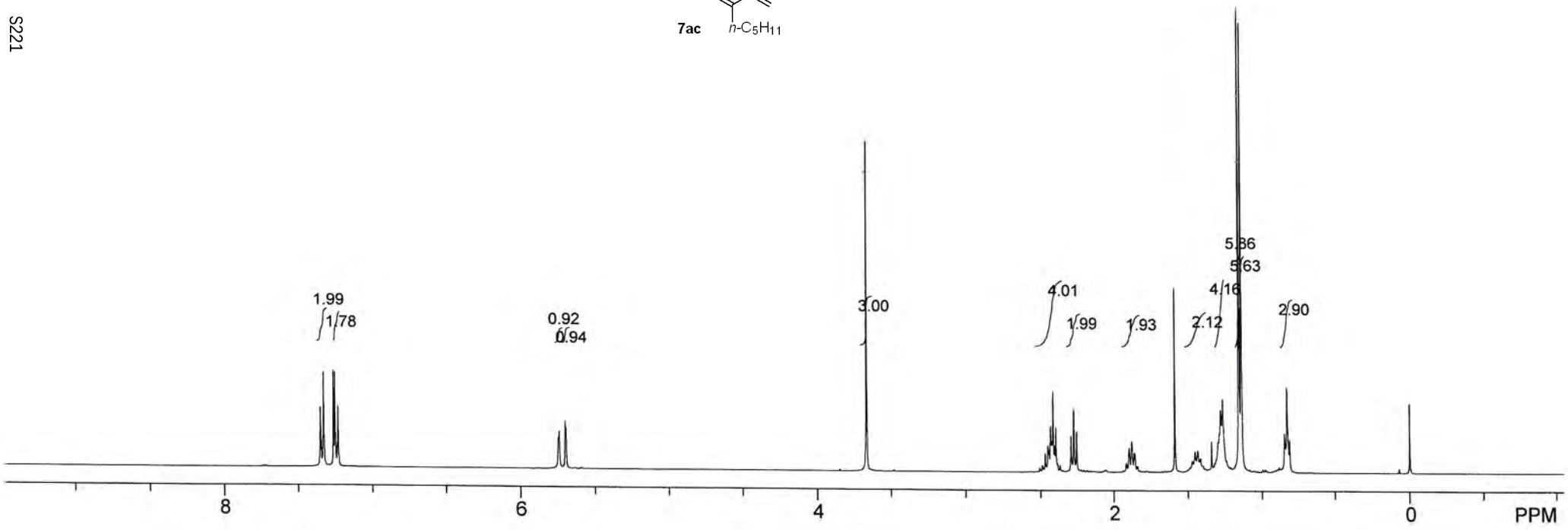
S220

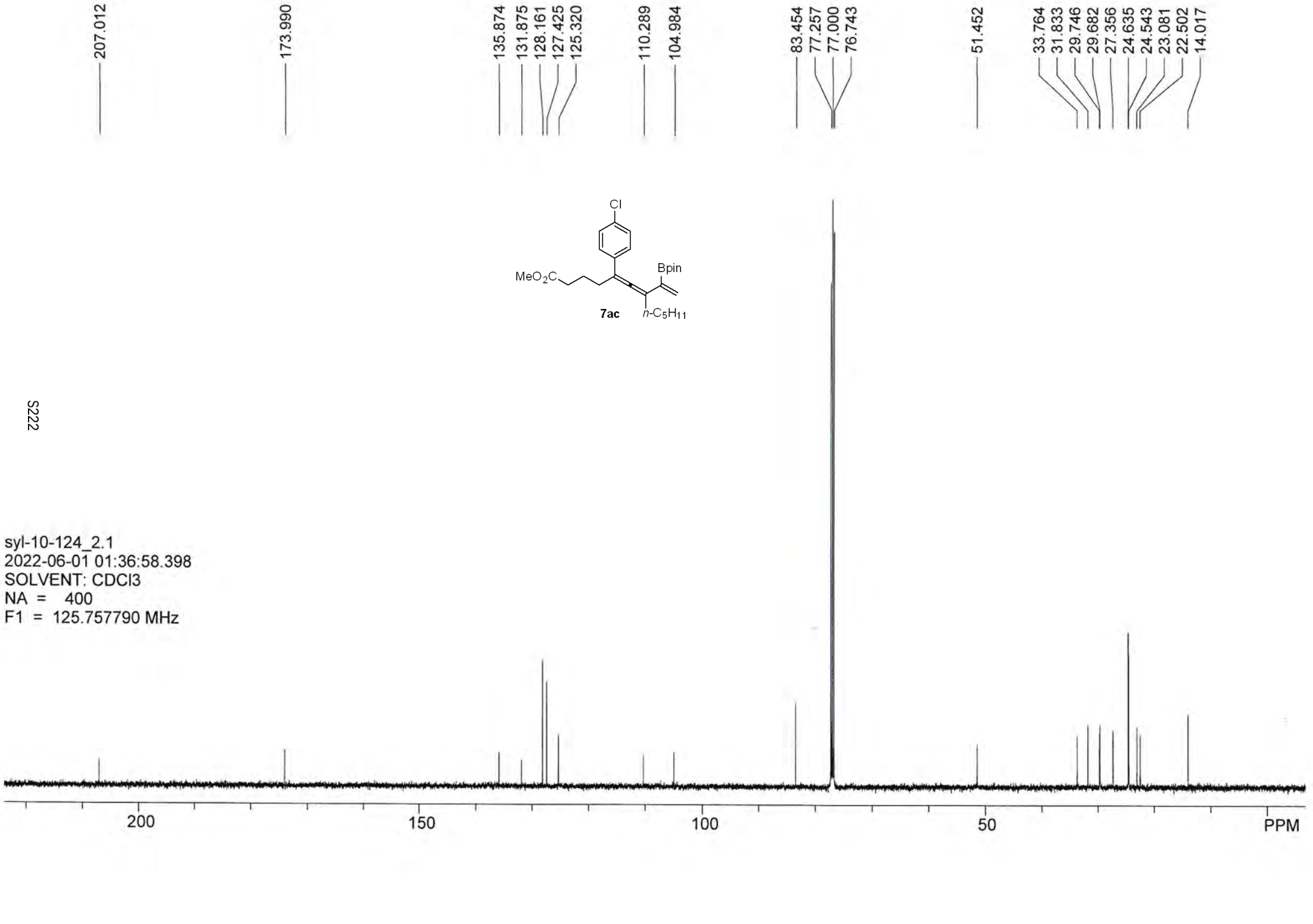


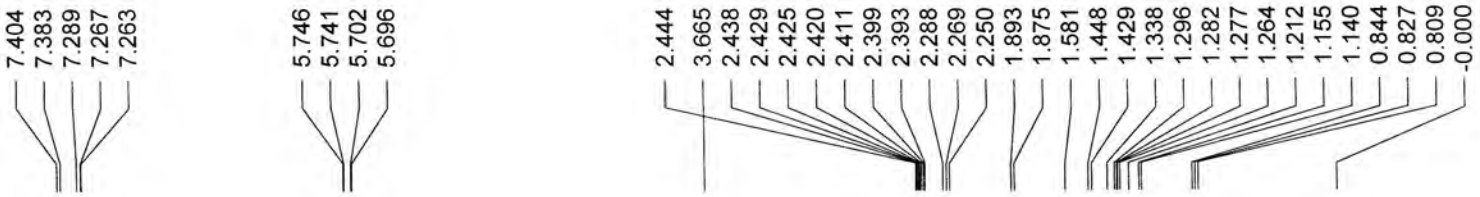
zj-4-146-pdt-H_1.1
2022-11-26 12:00:41.410
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz



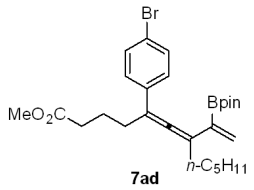
7ac



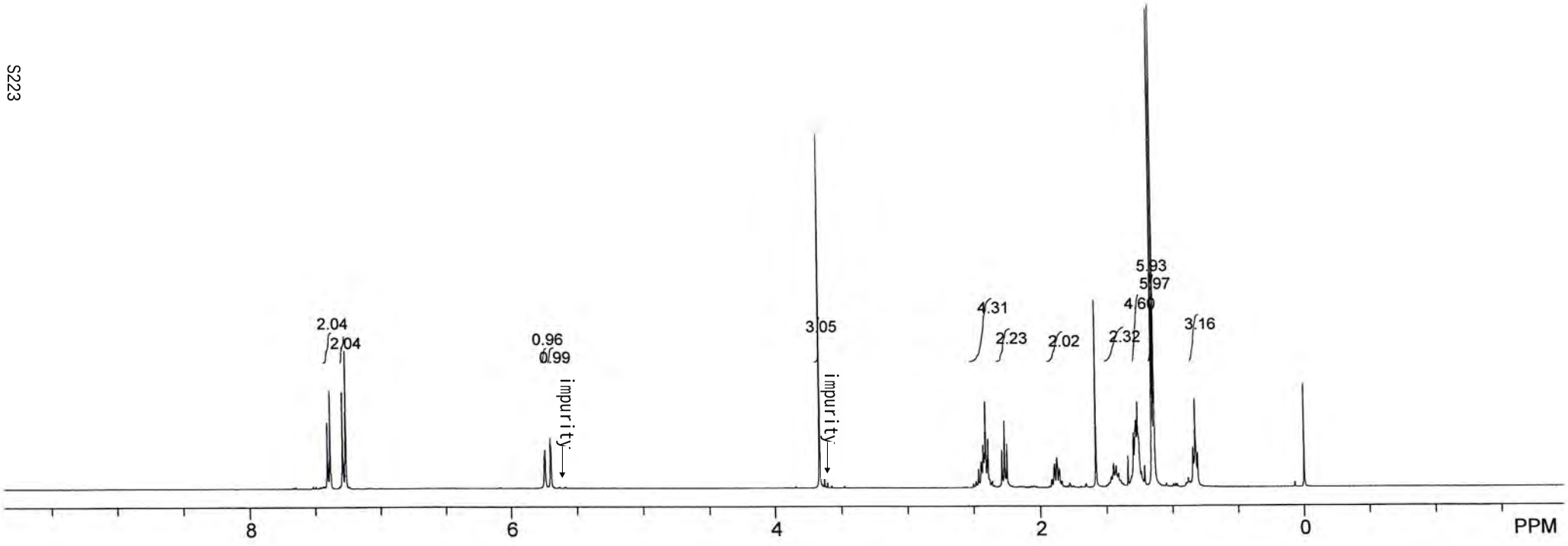

207.012
173.990
135.874
131.875
128.161
127.425
125.320
110.289
104.984
83.454
77.257
77.000
76.743
51.452
33.764
31.833
29.746
29.682
27.356
24.635
24.543
23.081
22.502
14.017
Cl
MeO2C
Bpin
7ac
n-C5H11
syl-10-124_2.1
2022-06-01 01:36:58.398
SOLVENT: CDCl3
NA = 400
F1 = 125.757790 MHz
200
150
100
50
PPM

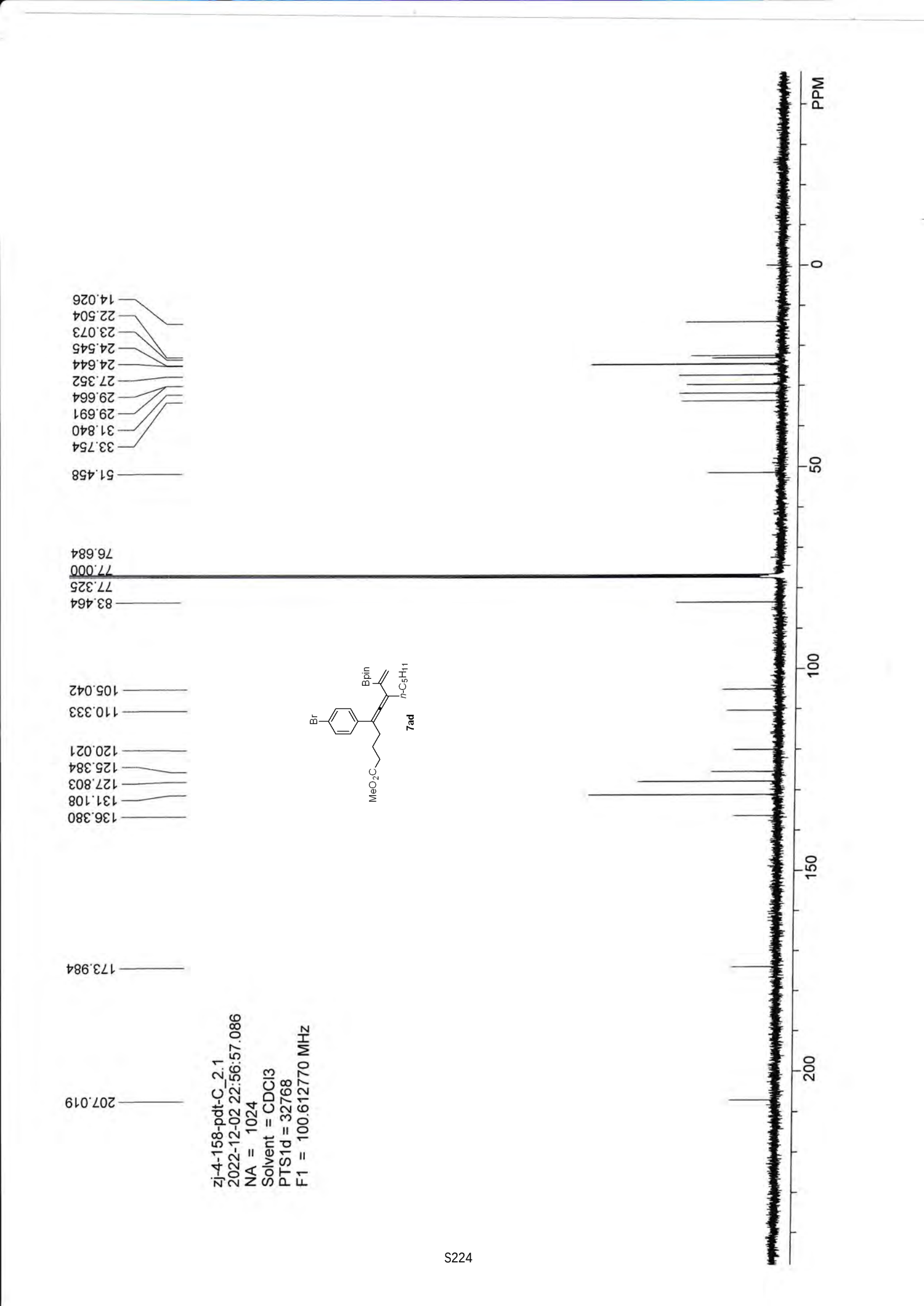


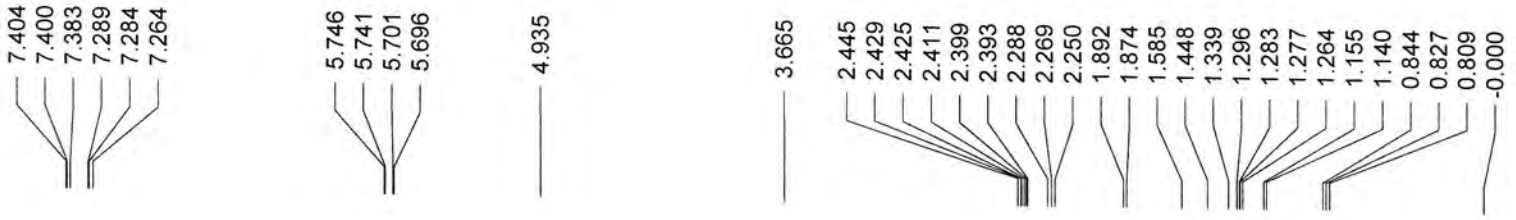
zj-4-158-pdt-H_1.1
2022-12-02 22:10:20.952
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz



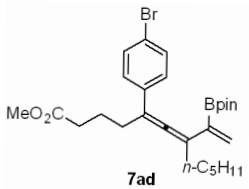
7ad




zj-4-158-pdt-C_2.1
2022-12-02 22:56:57.086
NA = 1024
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
207.019
173.984
136.380
131.108
127.803
125.384
120.021
110.333
105.042
83.464
77.325
77.000
76.684
51.458
33.754
31.840
29.691
29.664
27.352
24.644
24.545
23.073
22.504
14.026
PPM
0
50
100
150
200
Br
Bpin
n-C5H11
MeO2C
7ad

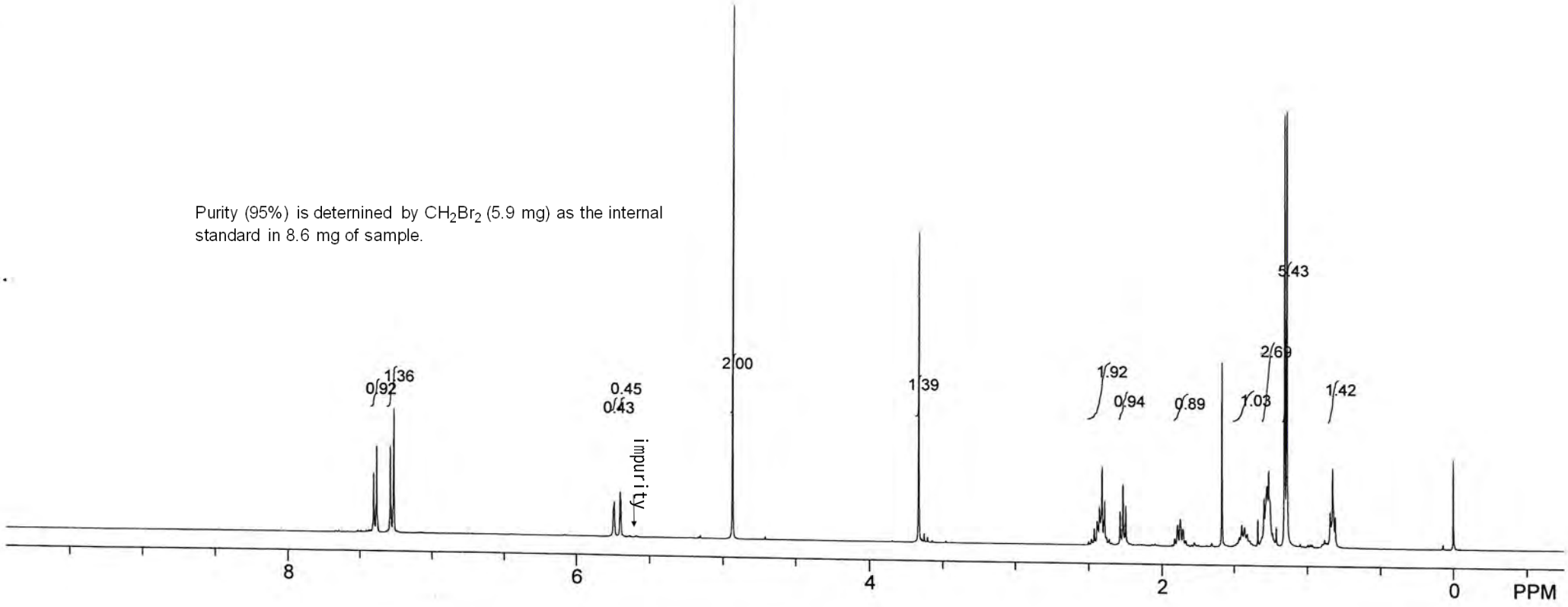


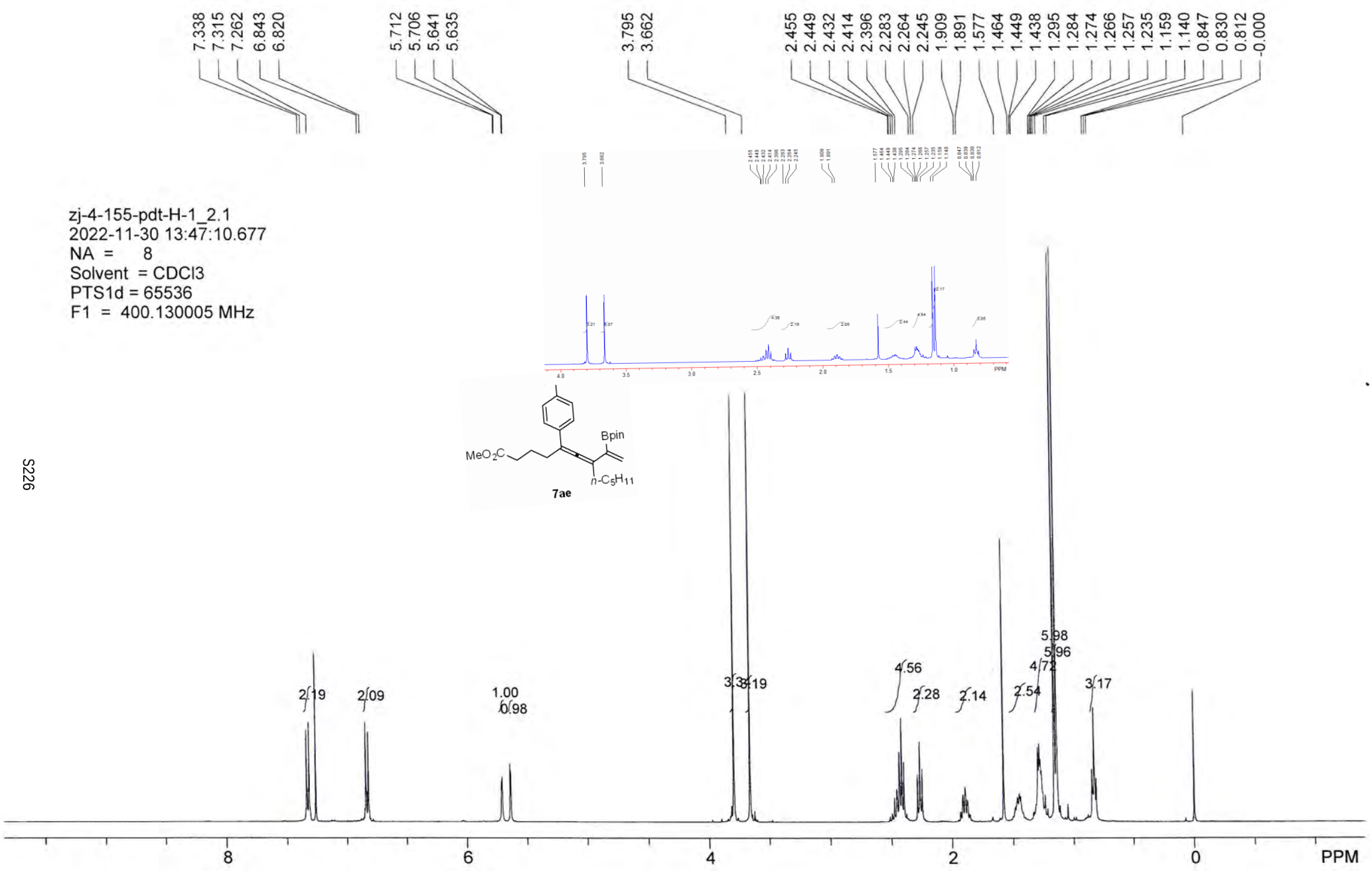
zj-4-158-purity_1.1
2022-12-03 10:08:24.858
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz

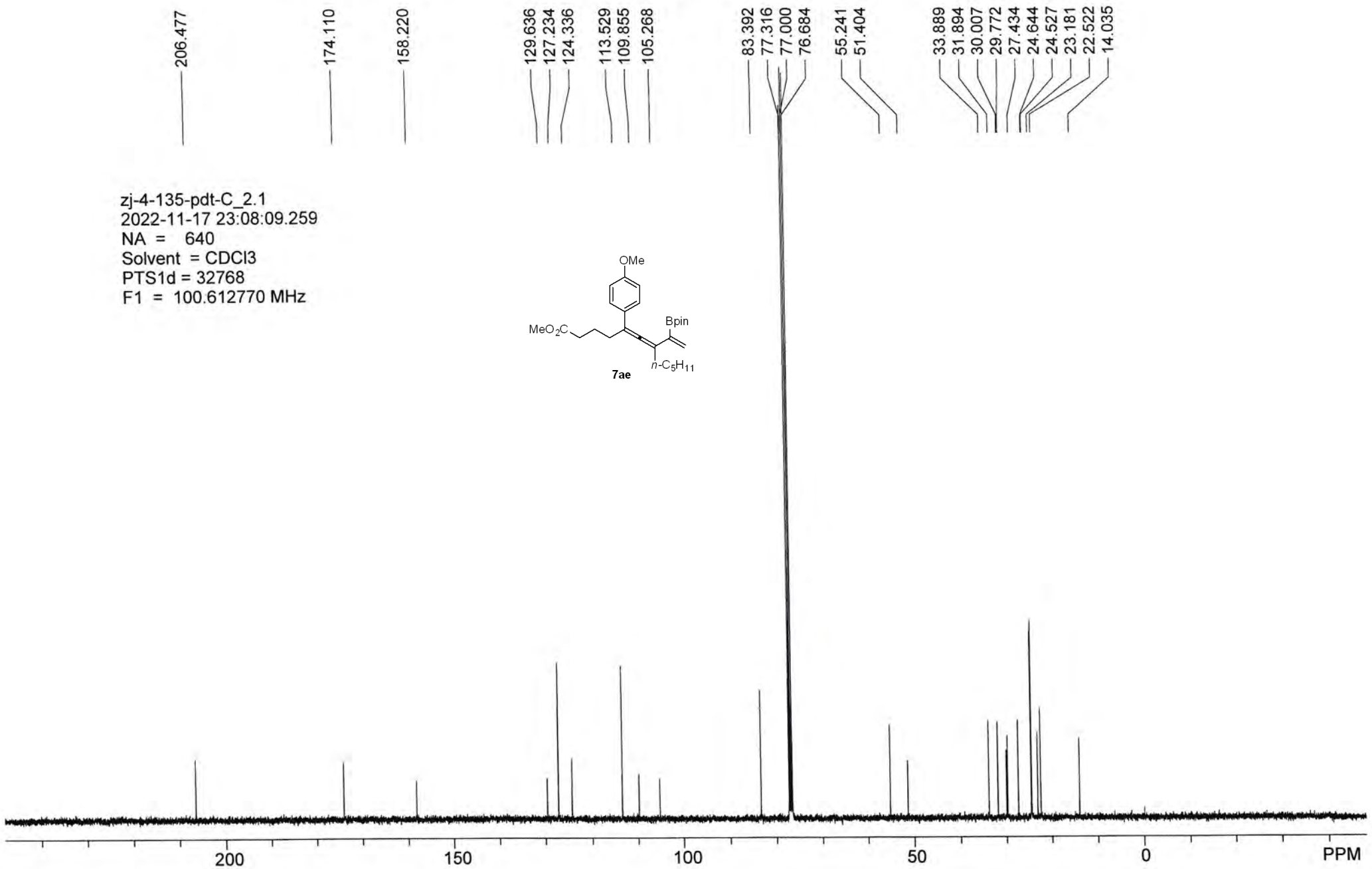


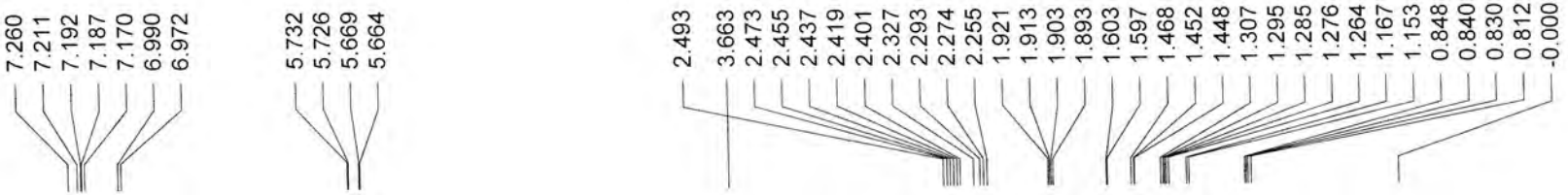
7ad

Purity (95%) is determined by CH_2Br_2 (5.9 mg) as the internal standard in 8.6 mg of sample.

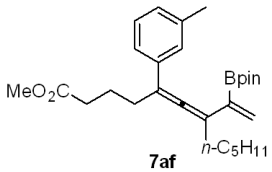



7.338
7.315
7.262
6.843
6.820
5.712
5.706
5.641
5.635
3.795
3.662
2.455
2.449
2.432
2.414
2.396
2.283
2.264
2.245
1.909
1.891
1.577
1.464
1.449
1.438
1.295
1.284
1.274
1.266
1.257
1.235
1.159
1.140
0.847
0.830
0.812
-0.000
zj-4-155-pdt-H-1_2.1
2022-11-30 13:47:10.677
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
MeO2C
Bpin
n-C5H11
7ae
2.19
2.09
1.00
0.98
3.38
3.19
4.56
2.28
2.14
2.54
4.72
5.98
5.96
3.17
8
6
4
2
0
PPM

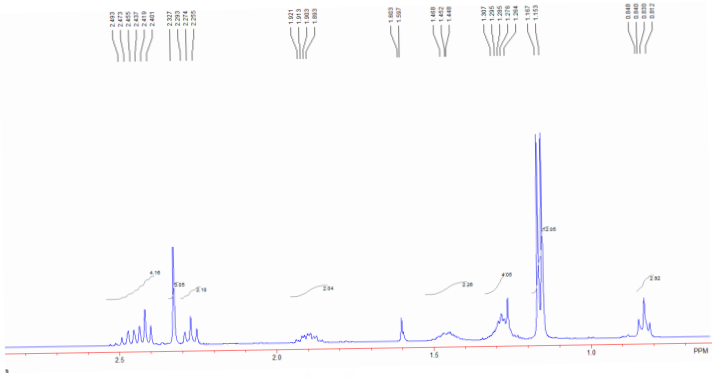

206.477
174.110
158.220
129.636
127.234
124.336
113.529
109.855
105.268
83.392
77.316
77.000
76.684
55.241
51.404
33.889
31.894
30.007
29.772
27.434
24.644
24.527
23.181
22.522
14.035
zj-4-135-pdt-C_2.1
2022-11-17 23:08:09.259
NA = 640
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
OMe
MeO2C
Bpin
n-C5H11
7ae
200
150
100
50
0
PPM

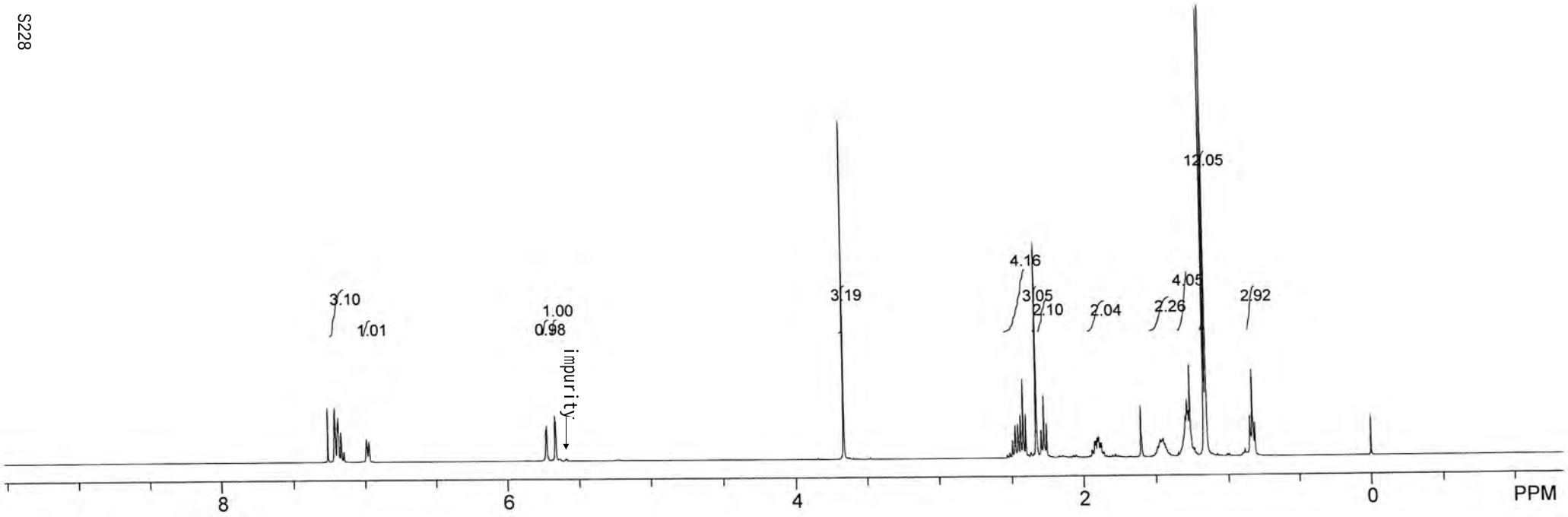


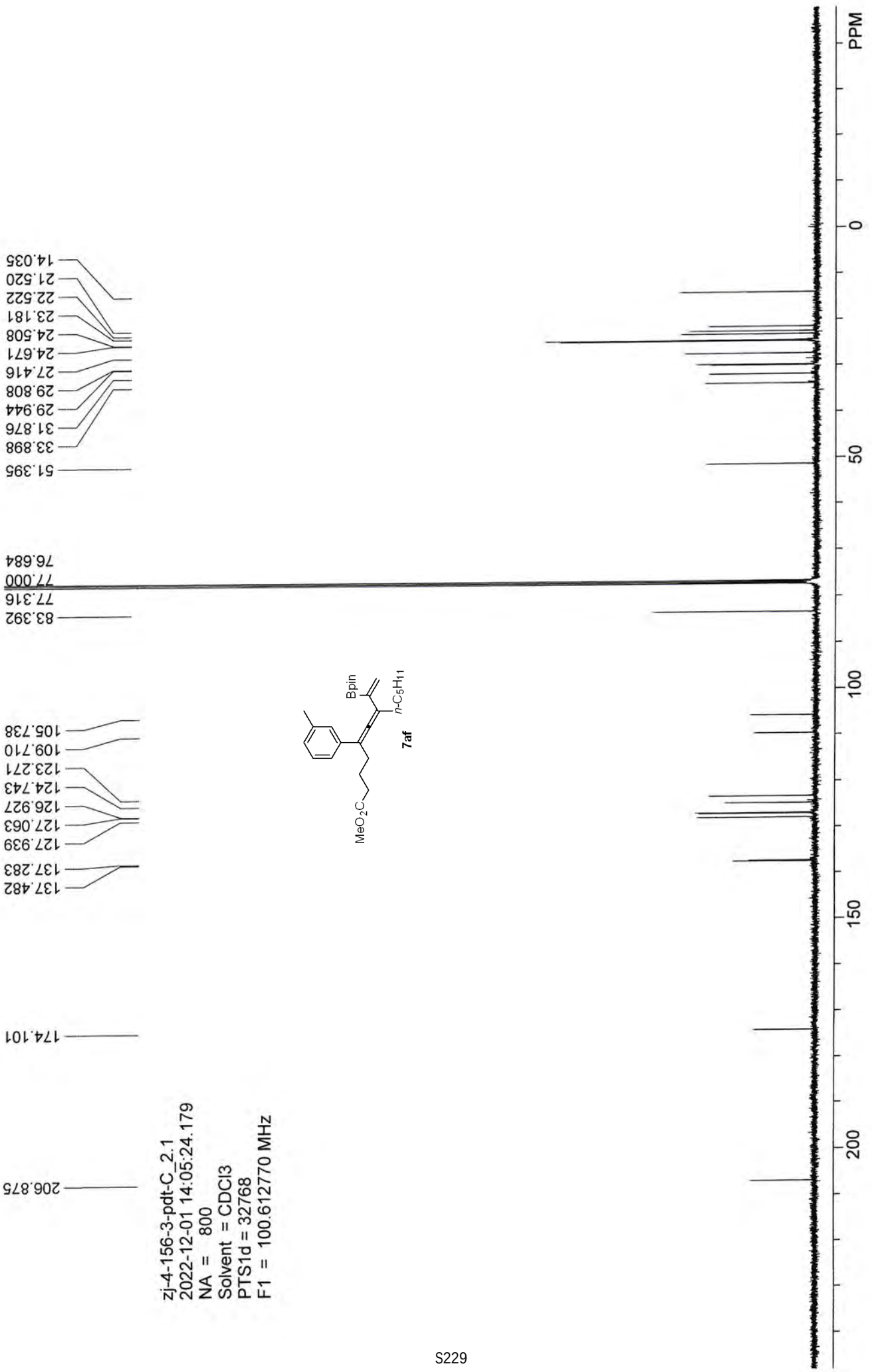
zj-4-156-3-pdt-H_1.1
2022-12-01 13:28:44.810
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz

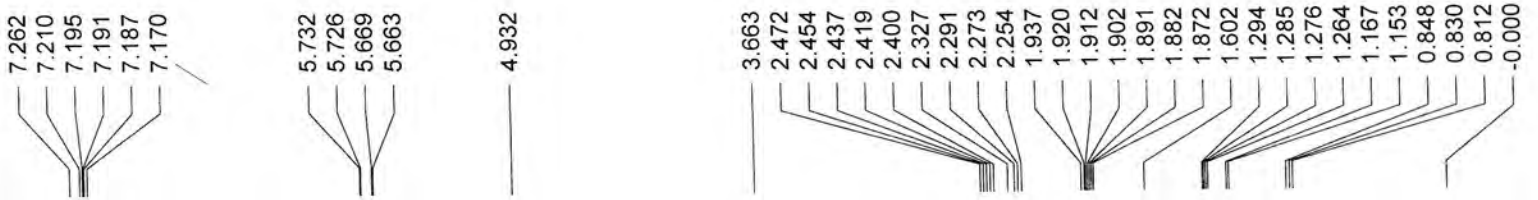


7af

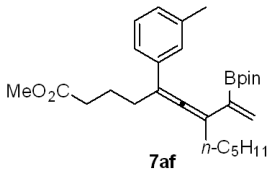





zj-4-156-3-pdt-C_2.1
2022-12-01 14:05:24.179
NA = 800
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
206.875
174.101
137.482
137.283
127.939
127.063
126.927
124.743
123.271
109.710
105.738
83.392
77.316
77.000
76.684
51.395
33.898
31.876
29.944
29.808
27.416
24.671
24.508
23.181
22.522
21.520
14.035
Bpin
n-C_5H_{11}
MeO_2C
7af
PPM
0
50
100
150
200

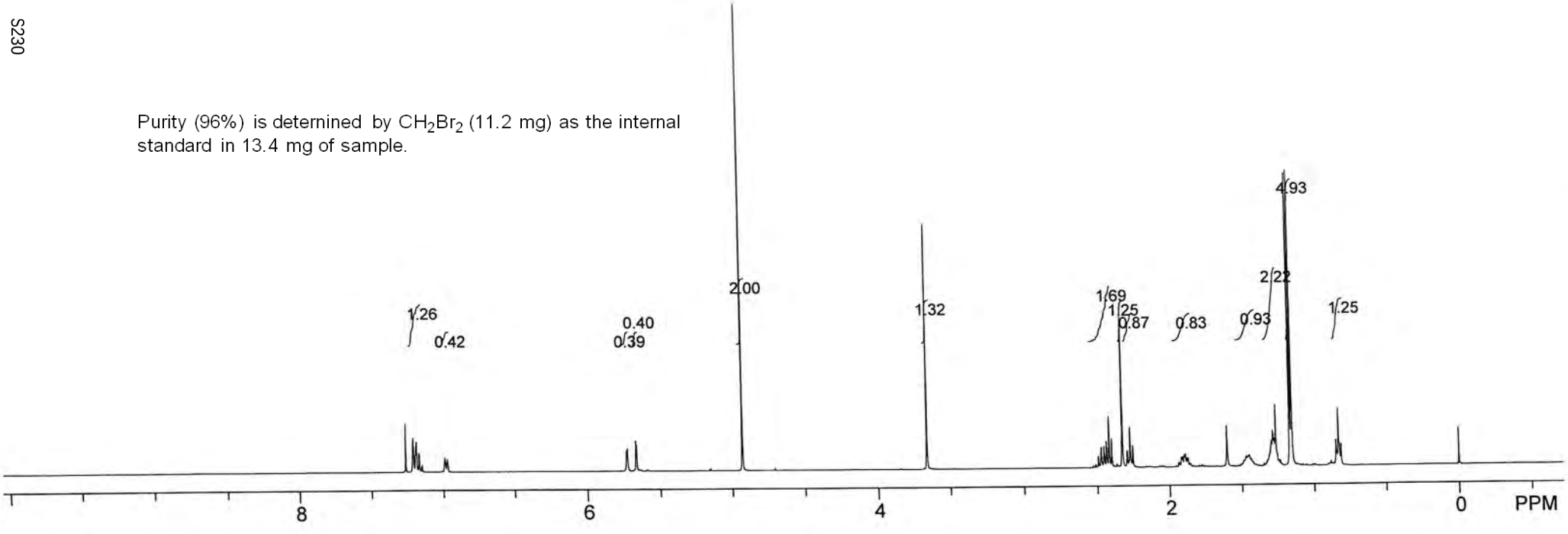


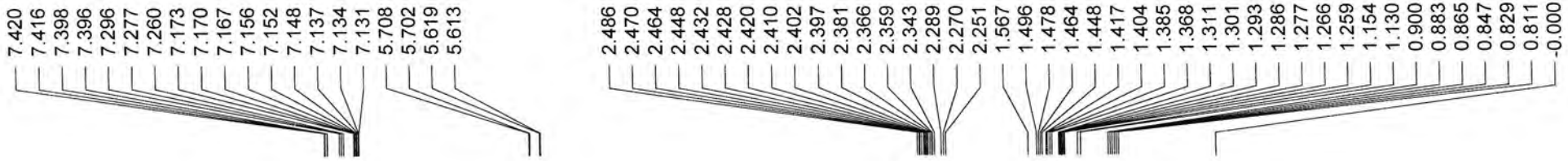
zj-4-156-3-purity_1.1
2022-12-02 09:10:06.113
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz



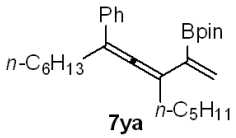
7af

Purity (96%) is determined by CH_2Br_2 (11.2 mg) as the internal standard in 13.4 mg of sample.

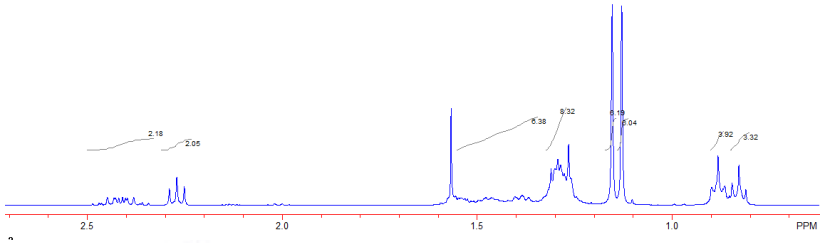


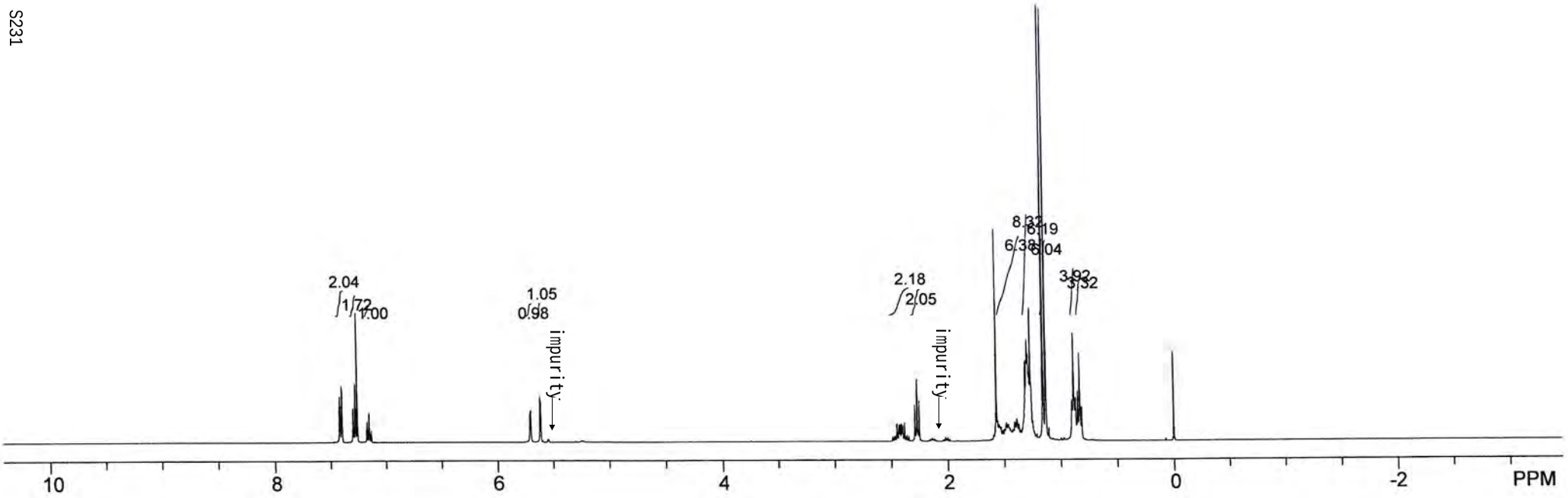


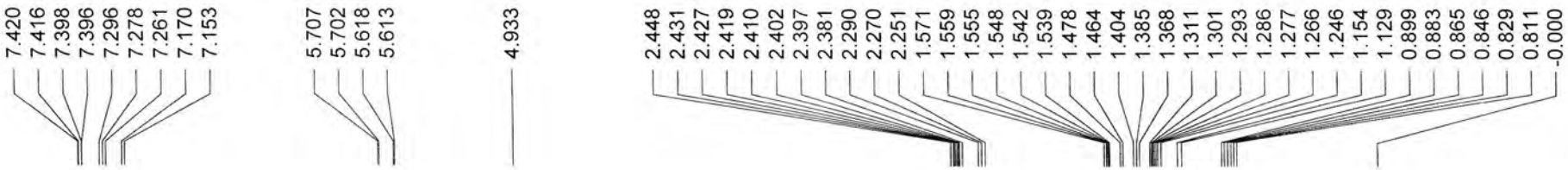
zj-4-164-C6-pdt-H_1.1
2022-12-07 16:35:00.492
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz



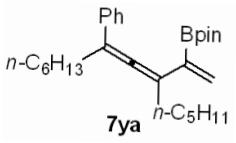
7ya





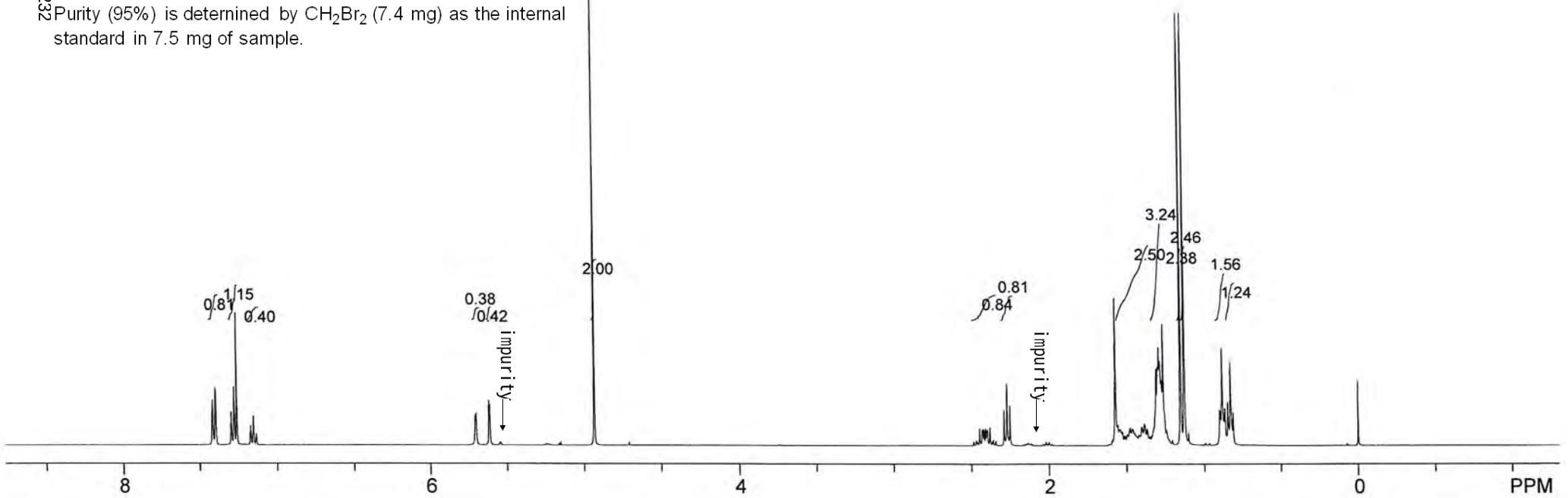


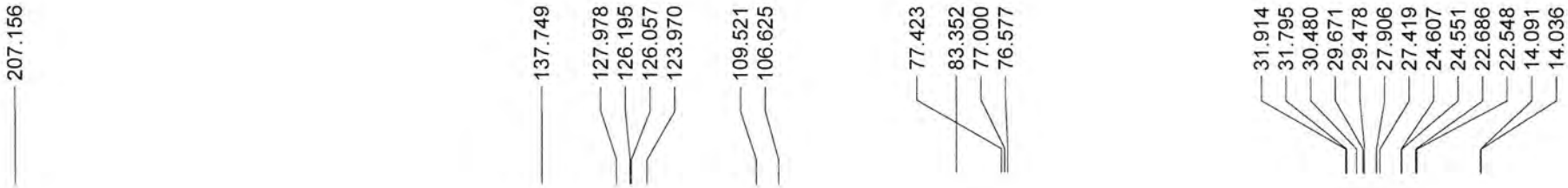
zj-4-164-purity_1.1
2022-12-07 20:24:51.743
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz

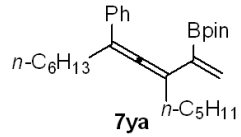


7ya

Purity (95%) is determined by CH_2Br_2 (7.4 mg) as the internal standard in 7.5 mg of sample.

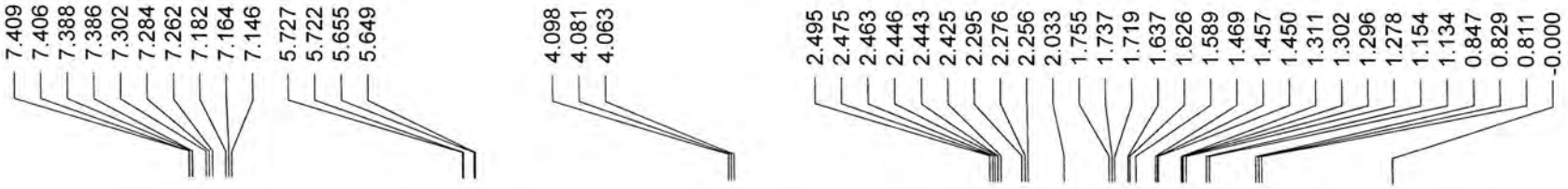




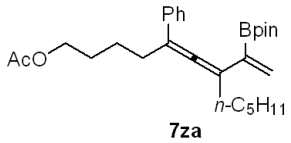


7ya

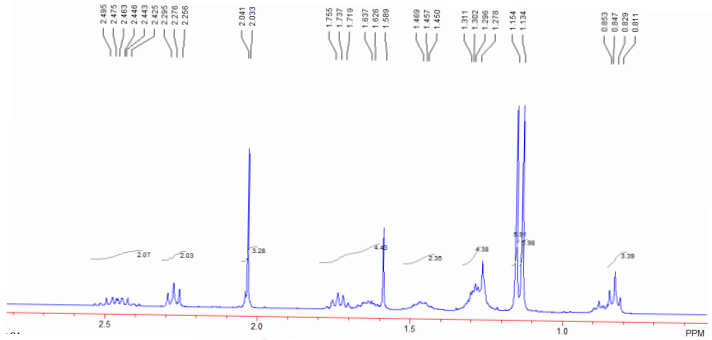
syl-10-133_2.1
2022-06-10 22:58:32.531
SOLVENT: CDCl3
NA = 1000
F1 = 75.467751 MHz

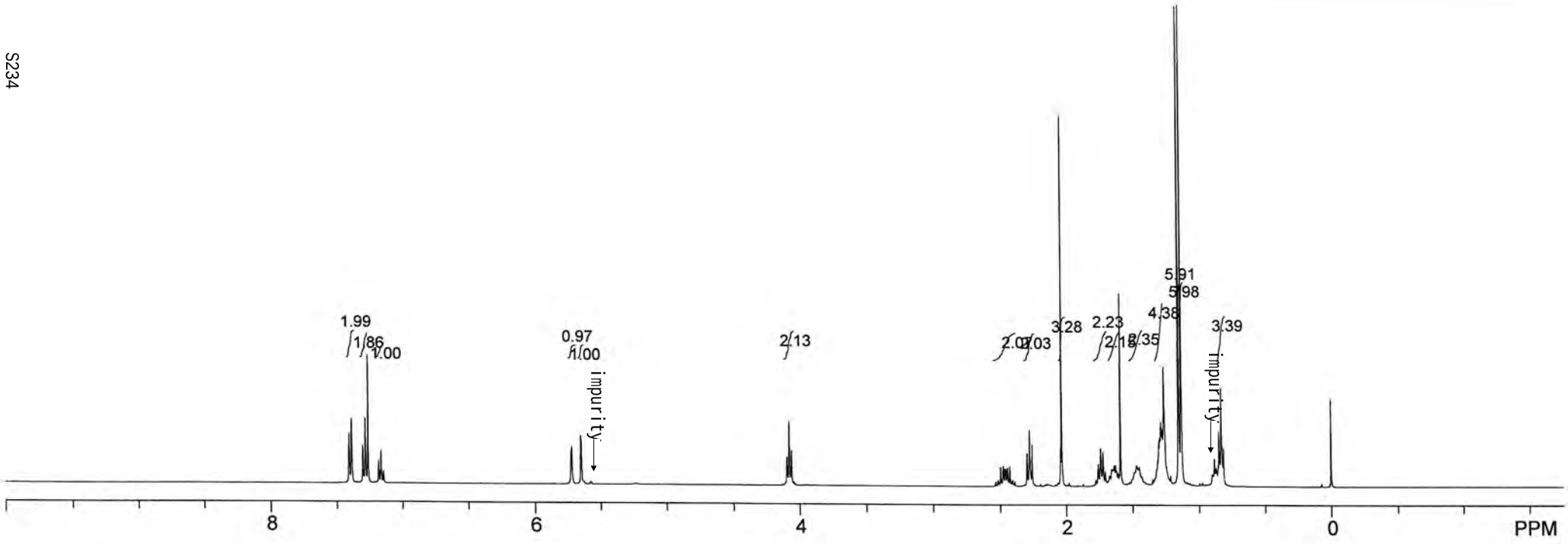


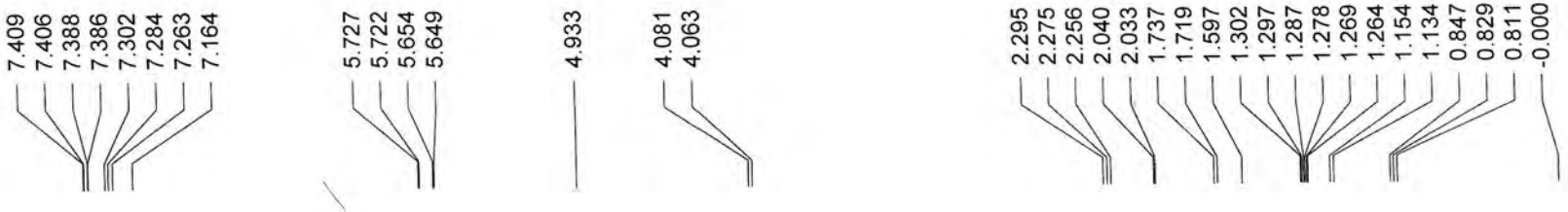
zj-4-161-pdt-H_1.1
2022-12-05 11:27:16.243
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz

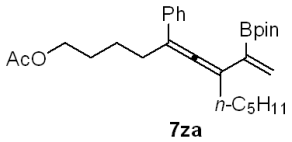


7za

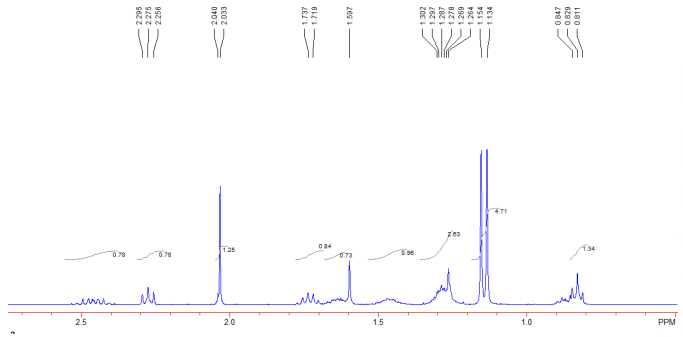








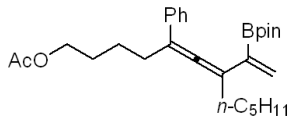
7za



zj-4-161-purity_1.1
2022-12-05 13:50:10.206
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz

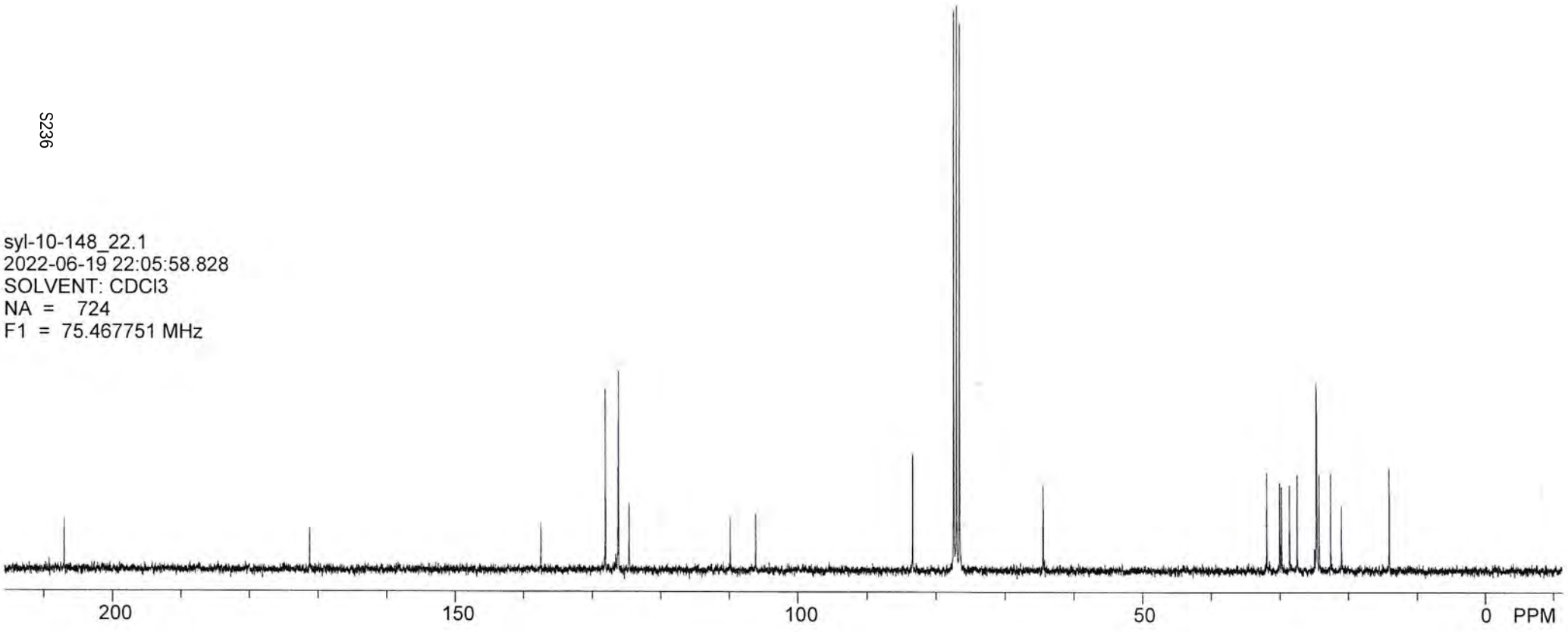
Purity (96%) is determined by CH_2Br_2 (13.2 mg) as the internal standard in 13.3 mg of sample.

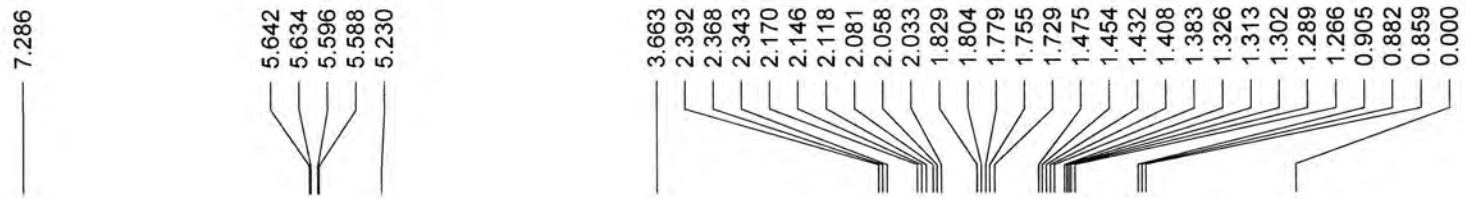
206.963
171.179
137.464
128.042
126.204
126.121
124.586
109.833
106.120
83.361
77.423
77.000
76.577
64.490
31.868
29.984
29.726
28.541
27.419
24.625
24.542
24.202
22.511
20.957
14.018

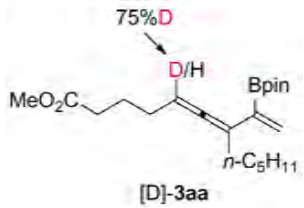


7za

syl-10-148_22.1
2022-06-19 22:05:58.828
SOLVENT: CDCl3
NA = 724
F1 = 75.467751 MHz

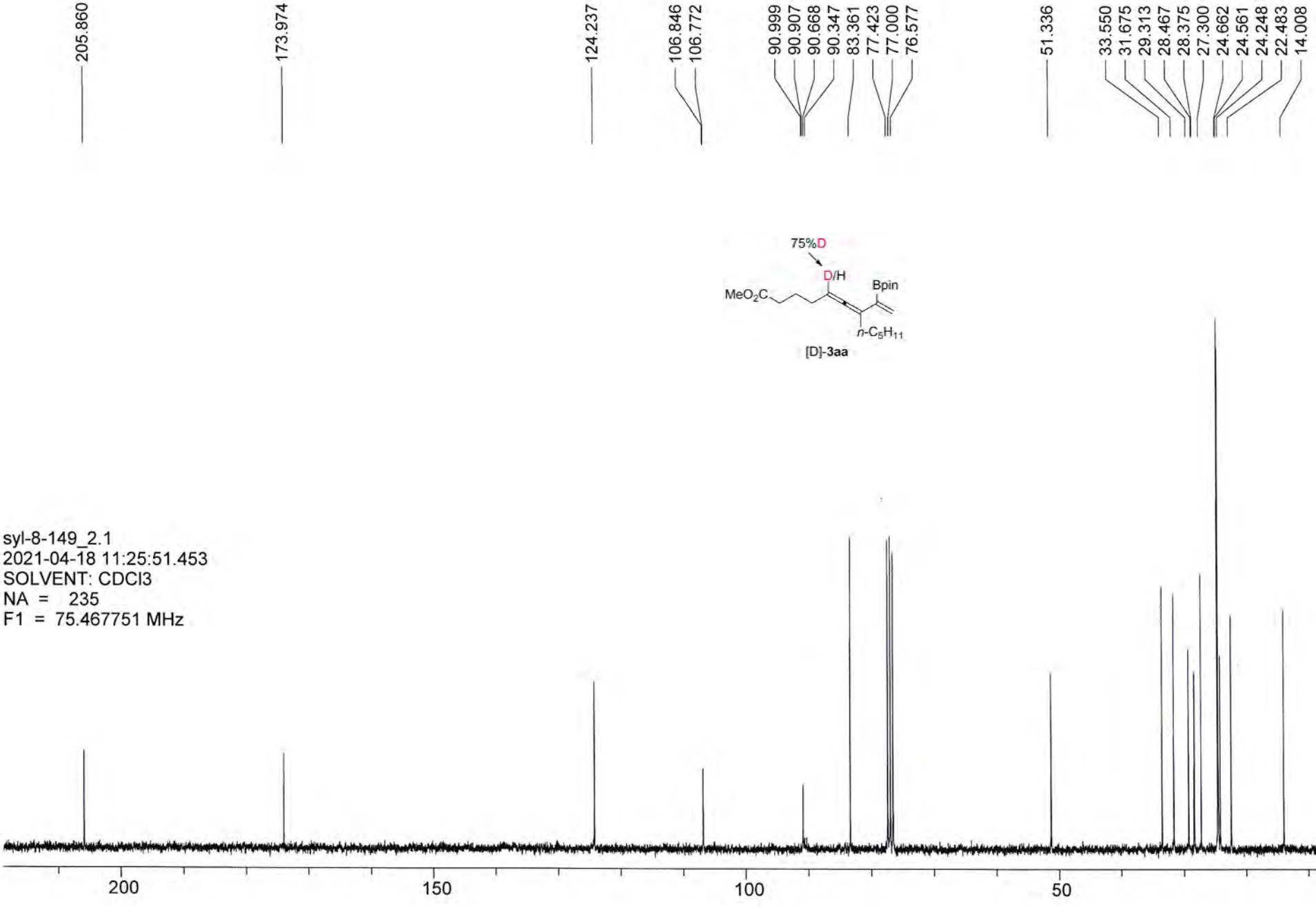


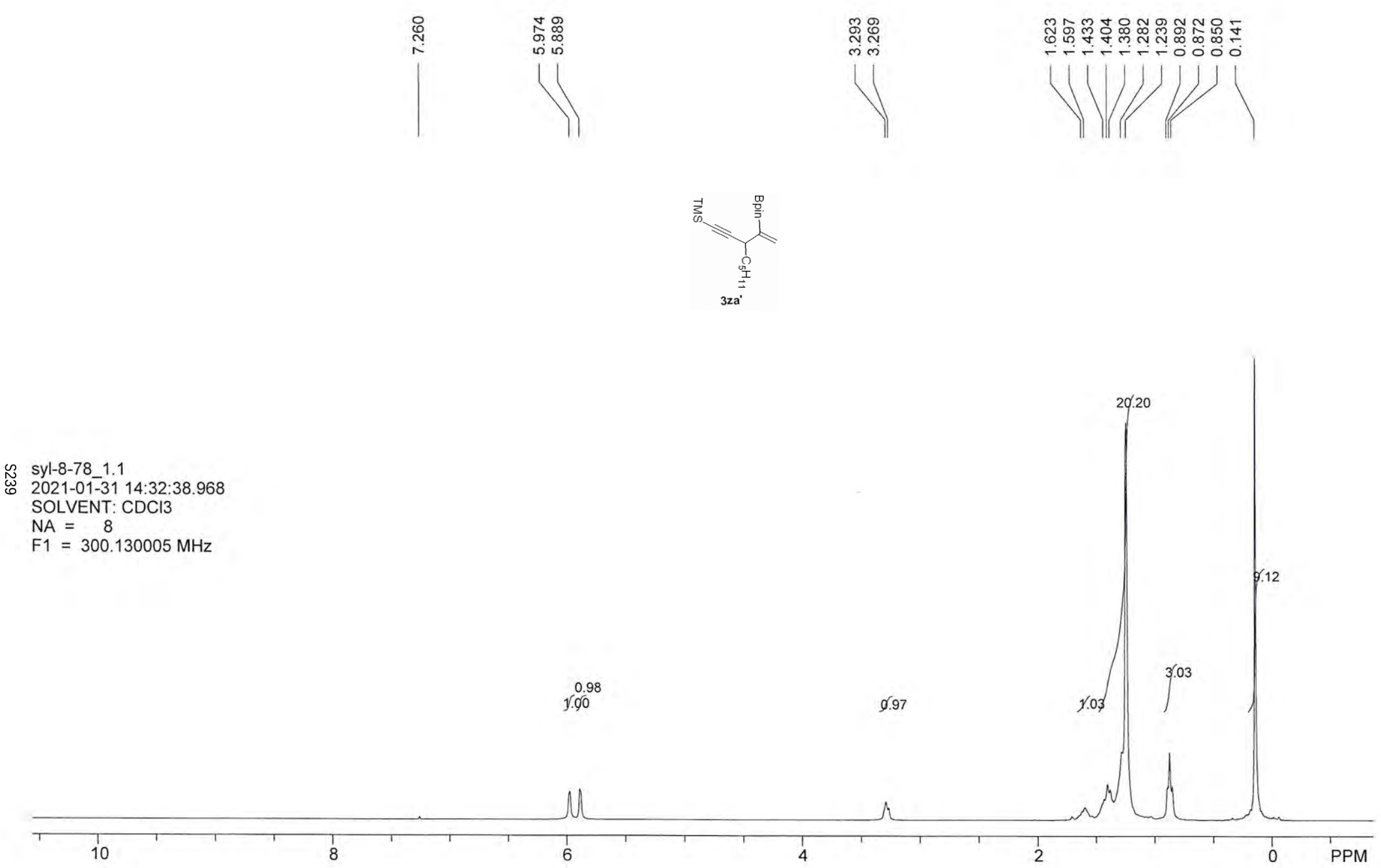


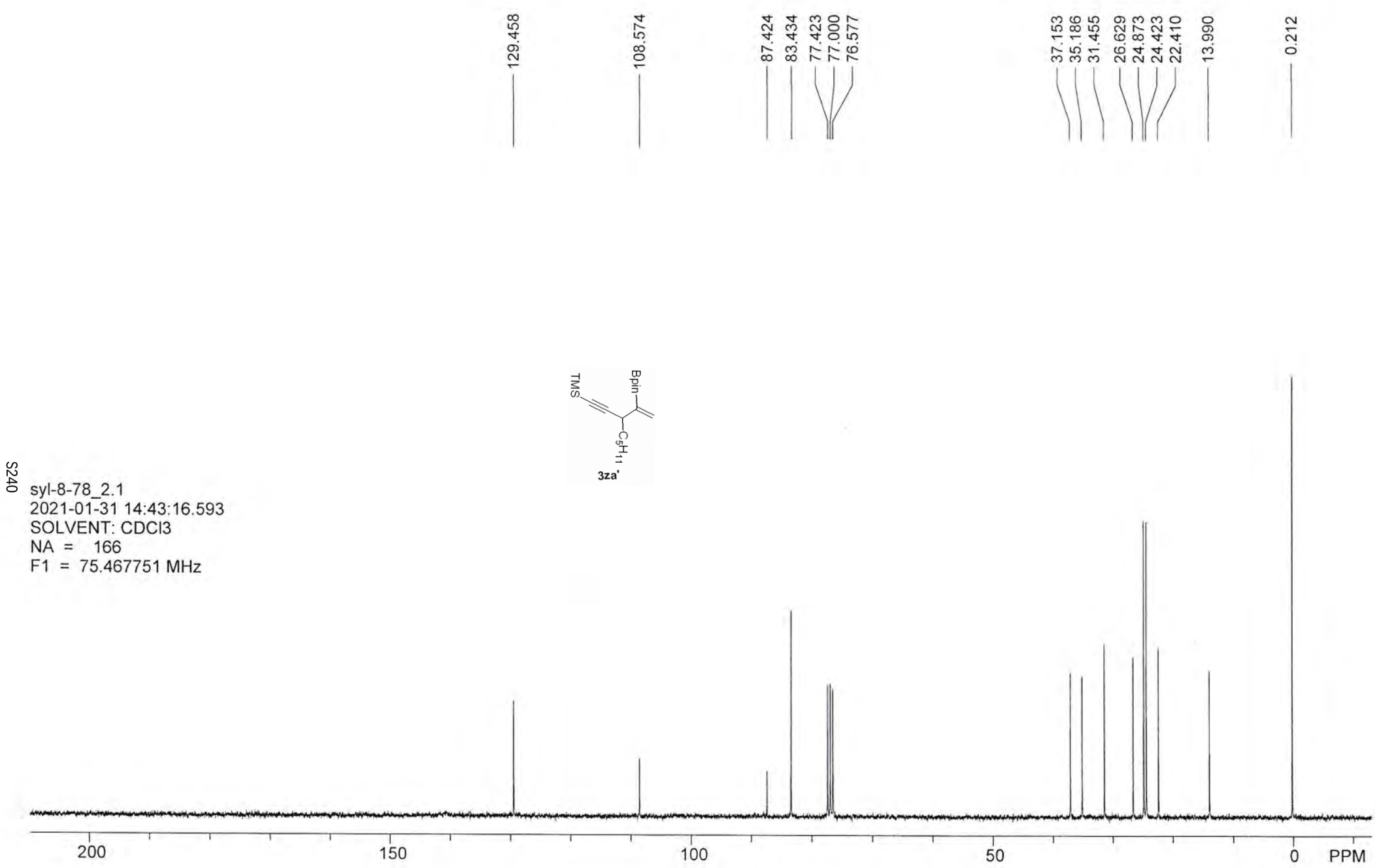


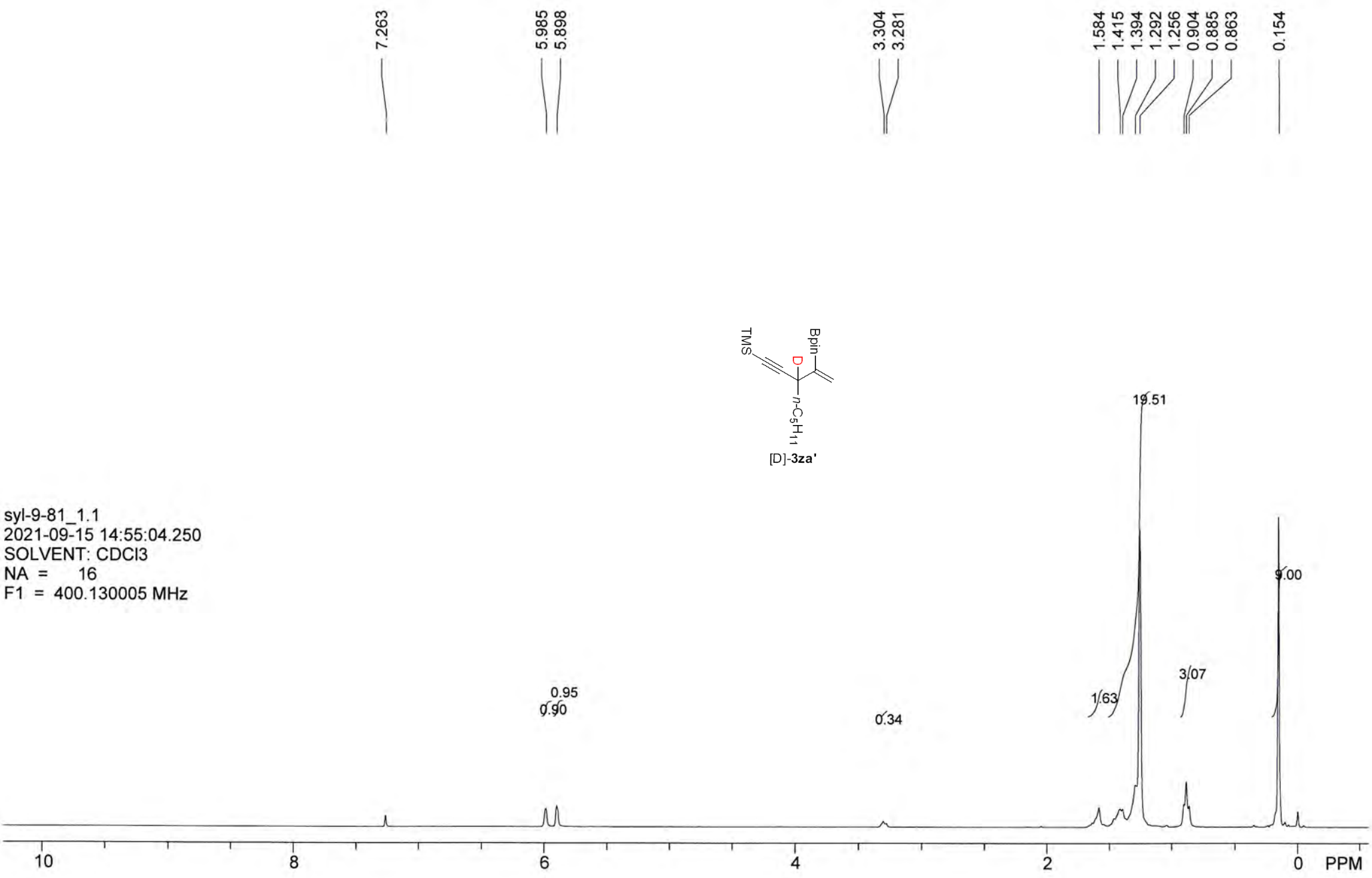
[D]-**3aa**

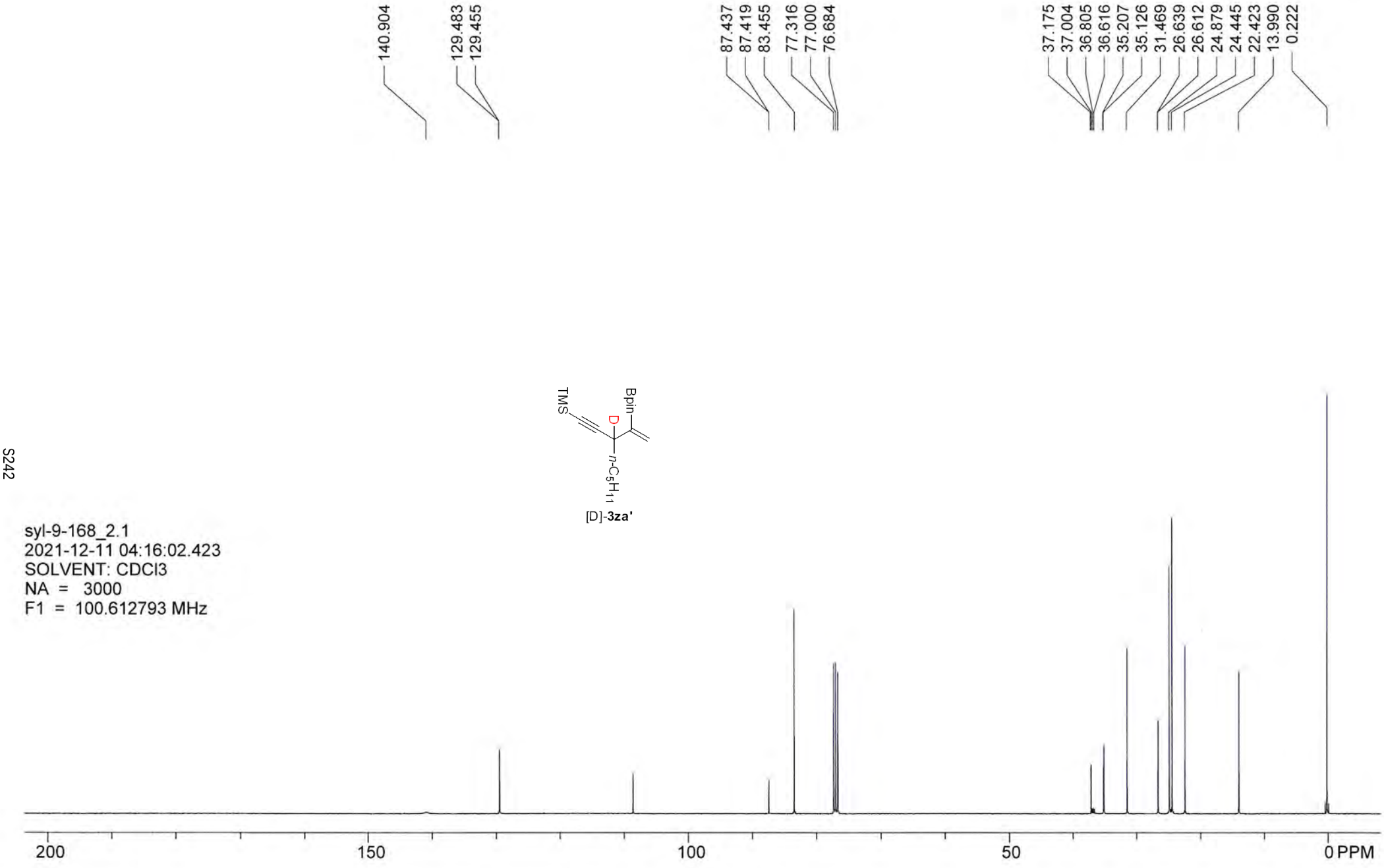
syl-8-149_1.1
2021-04-18 11:10:30.062
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz

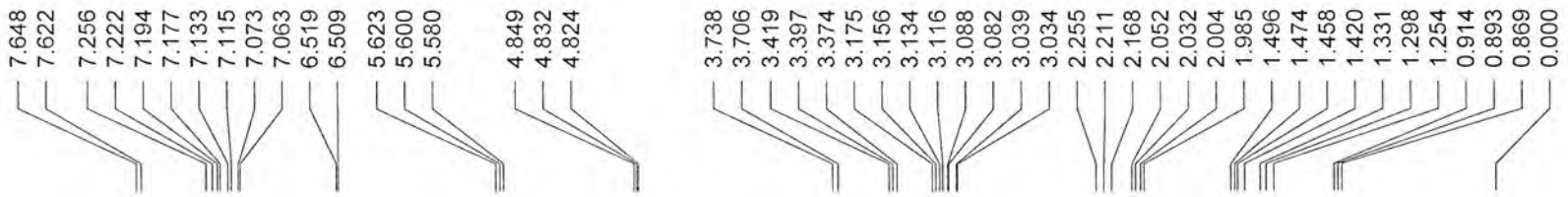

205.860
173.974
124.237
106.846
106.772
90.999
90.907
90.668
90.347
83.361
77.423
77.000
76.577
51.336
33.550
31.675
29.313
28.467
28.375
27.300
24.662
24.561
24.248
22.483
14.008
75%D
D/H
Bpin
MeO2C
n-C5H11
[D]-3aa
syl-8-149_2.1
2021-04-18 11:25:51.453
SOLVENT: CDCl3
NA = 235
F1 = 75.467751 MHz
200
150
100
50
0PPM

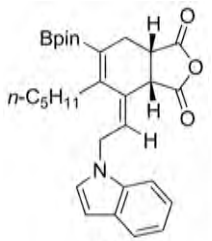

7.260
5.974
5.889
3.293
3.269
1.623
1.597
1.433
1.404
1.380
1.282
1.239
0.892
0.872
0.850
0.141
TMS
Bpin
C_5H_{11}
3za'
syl-8-78_1.1
2021-01-31 14:32:38.968
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
0.98
1.00
0.97
1.03
20.20
3.03
9.12
10
8
6
4
2
0
PPM


129.458
108.574
87.424
83.434
77.423
77.000
76.577
37.153
35.186
31.455
26.629
24.873
24.423
22.410
13.990
0.212
TMS
Bpin
C_5H_{11}
3za'
syl-8-78_2.1
2021-01-31 14:43:16.593
SOLVENT: CDCl3
NA = 166
F1 = 75.467751 MHz
200
150
100
50
0
PPM

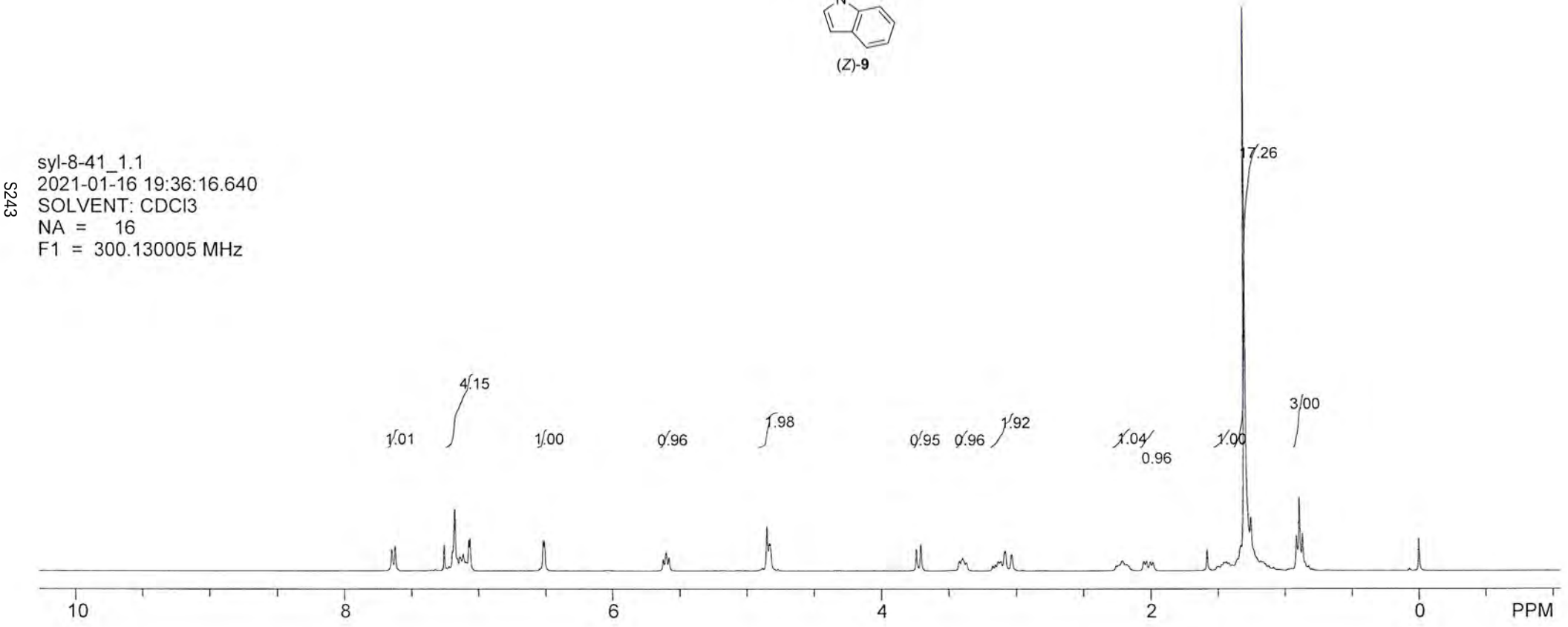

7.263
5.985
5.898
3.304
3.281
1.584
1.415
1.394
1.292
1.256
0.904
0.885
0.863
0.154
TMS
Bpin
D
n-C5H11
[D]-3za'
syl-9-81_1.1
2021-09-15 14:55:04.250
SOLVENT: CDCl3
NA = 16
F1 = 400.130005 MHz
19.51
9.00
3.07
1.63
0.95
0.90
0.34
10
8
6
4
2
0
PPM

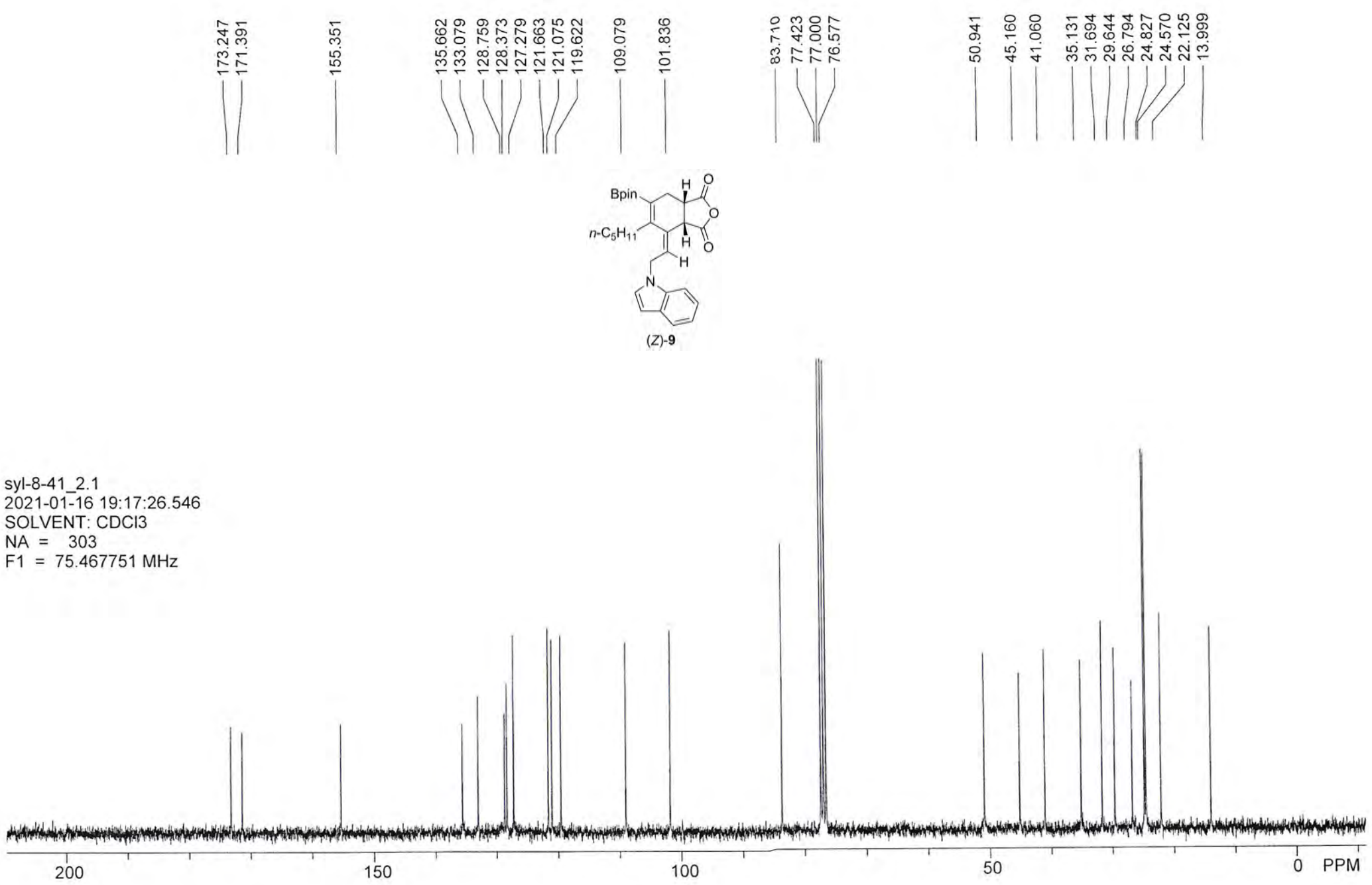

140.904
129.483
129.455
87.437
87.419
83.455
77.316
77.000
76.684
37.175
37.004
36.805
36.616
35.207
35.126
31.469
26.639
26.612
24.879
24.445
22.423
13.990
0.222
TMS
D
Bpin
n-C5H11
[D]-3za'
syl-9-168_2.1
2021-12-11 04:16:02.423
SOLVENT: CDCl3
NA = 3000
F1 = 100.612793 MHz
200
150
100
50
0 PPM

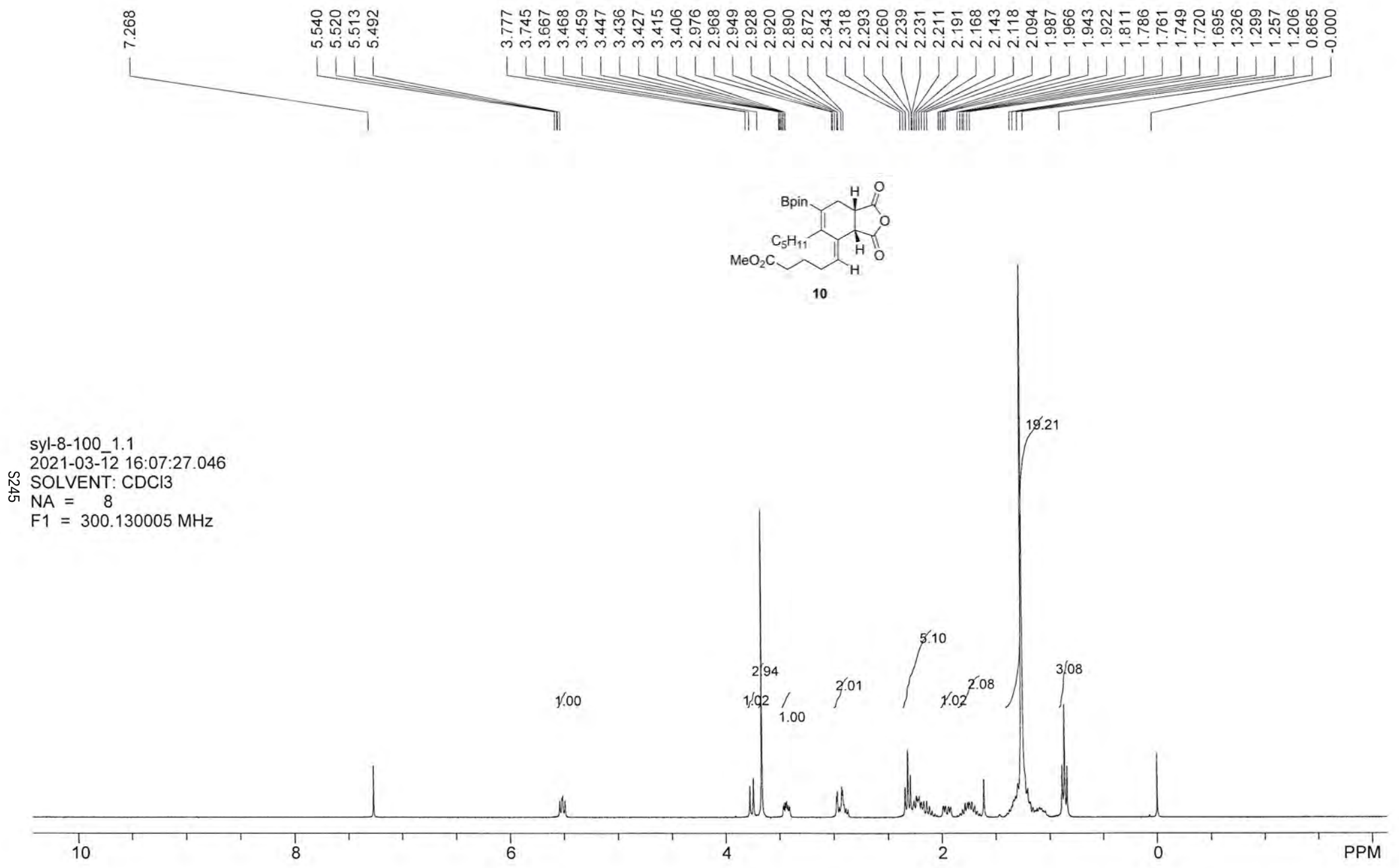

7.648
7.622
7.256
7.222
7.194
7.177
7.133
7.115
7.073
7.063
6.519
6.509
5.623
5.600
5.580
4.849
4.832
4.824
3.738
3.706
3.419
3.397
3.374
3.175
3.156
3.134
3.116
3.088
3.082
3.039
3.034
2.255
2.211
2.168
2.052
2.032
2.004
1.985
1.496
1.474
1.458
1.420
1.331
1.298
1.254
0.914
0.893
0.869
0.000

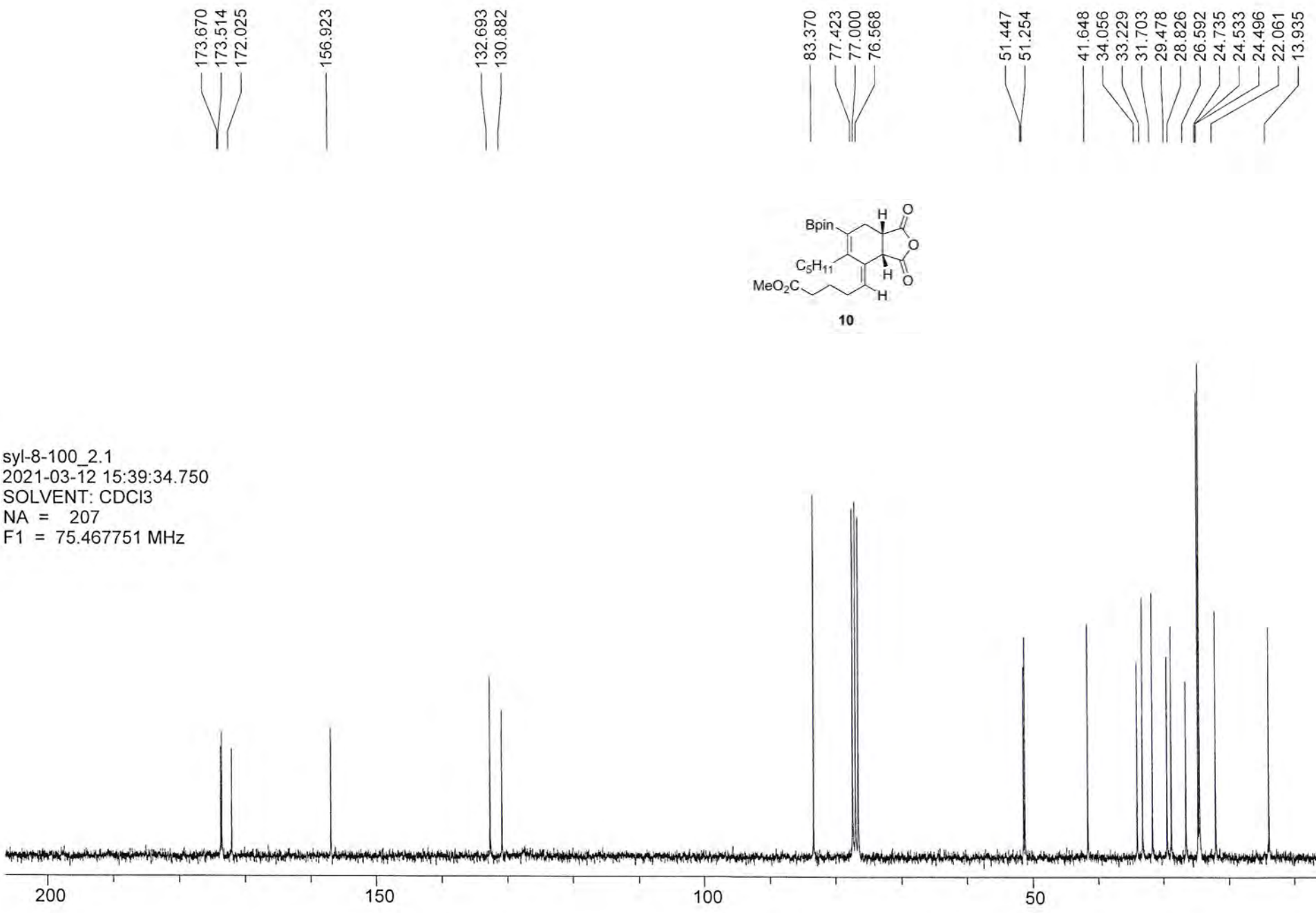

Bpin
H
O
O
O
n-C5H11
H
H
N

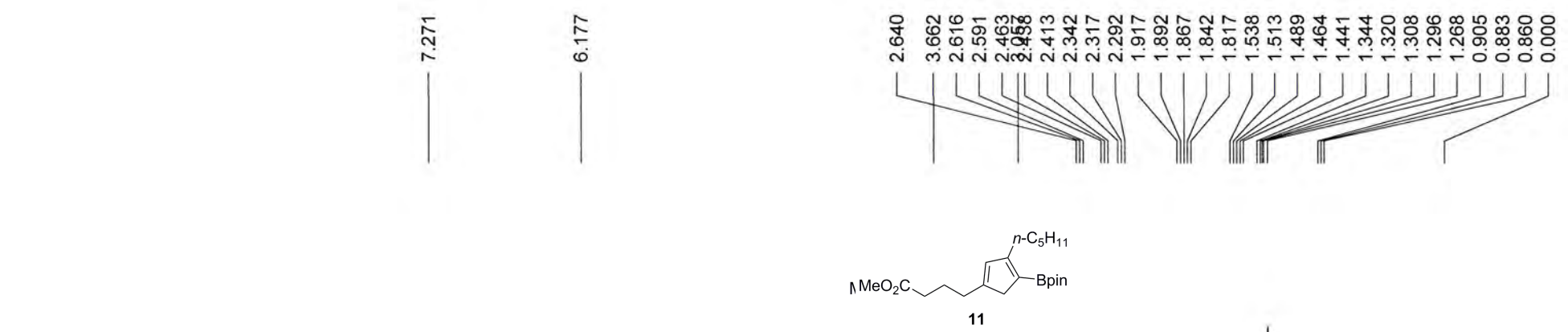
(Z)-**9**


syl-8-41_1.1
2021-01-16 19:36:16.640
SOLVENT: CDCl3
NA = 16
F1 = 300.130005 MHz
17.26
4.15
1.01
1.00
0.96
1.98
0.95
0.96
1.92
1.04
0.96
1.00
3.00
10
8
6
4
2
0
PPM


173.247
171.391
155.351
135.662
133.079
128.759
128.373
127.279
121.663
121.075
119.622
109.079
101.836
83.710
77.423
77.000
76.577
50.941
45.160
41.060
35.131
31.694
29.644
26.794
24.827
24.570
22.125
13.999
Bpin
n-C5H11
(Z)-9
syl-8-41_2.1
2021-01-16 19:17:26.546
SOLVENT: CDCl3
NA = 303
F1 = 75.467751 MHz
200
150
100
50
0
PPM

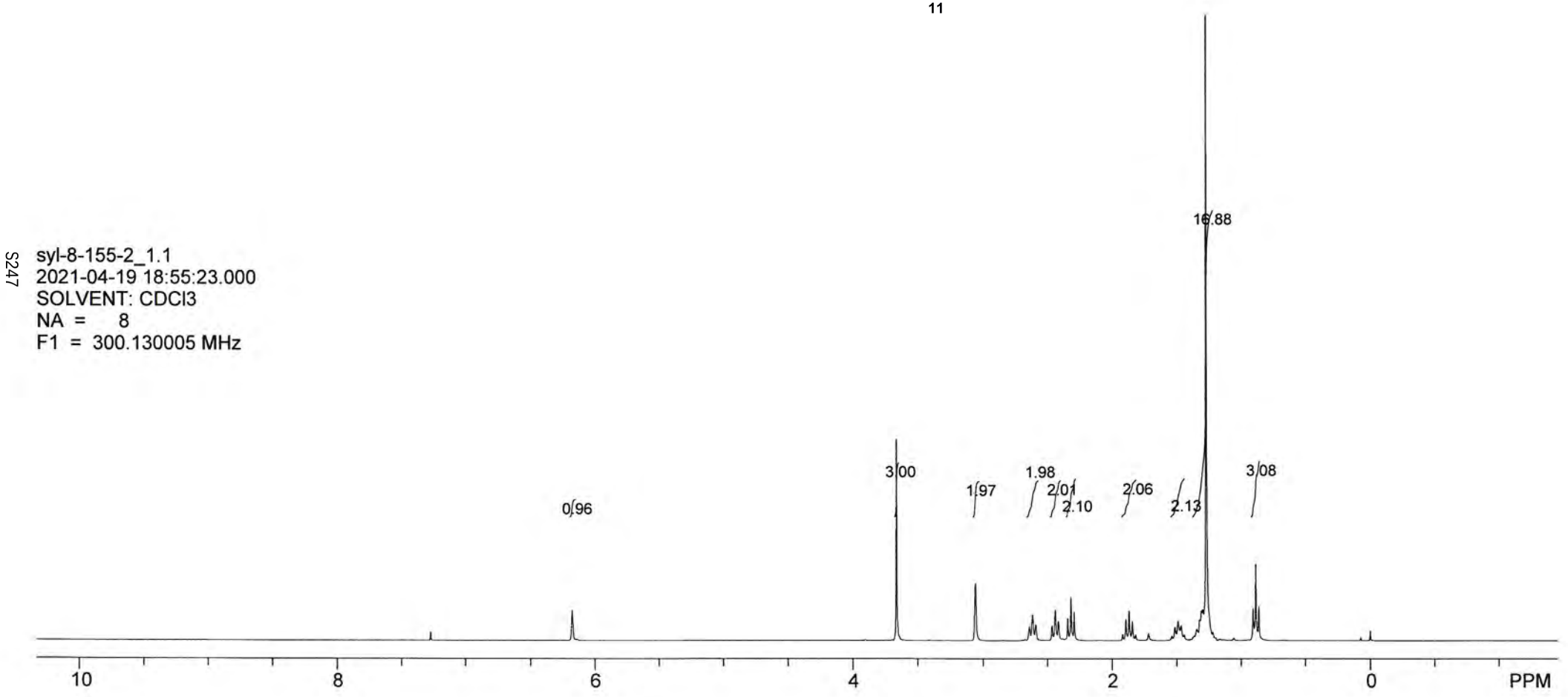

7.268
5.540
5.520
5.513
5.492
3.777
3.745
3.667
3.468
3.459
3.447
3.436
3.427
3.415
3.406
2.976
2.968
2.949
2.928
2.920
2.890
2.872
2.343
2.318
2.293
2.260
2.239
2.231
2.211
2.191
2.168
2.143
2.118
2.094
1.987
1.966
1.943
1.922
1.811
1.786
1.761
1.749
1.720
1.695
1.326
1.299
1.257
1.206
0.865
-0.000
Bpin
H
O
O
O
C_5H_{11}
H
O
MeO_2C
H
10
syl-8-100_1.1
2021-03-12 16:07:27.046
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
19.21
5.10
3.08
2.94
2.08
2.01
1.00
1.02
1.00
1.02
10
8
6
4
2
0
PPM


173.670
173.514
172.025
156.923
132.693
130.882
83.370
77.423
77.000
76.568
51.447
51.254
41.648
34.056
33.229
31.703
29.478
28.826
26.592
24.735
24.533
24.496
22.061
13.935
Bpin
C_5H_{11}
MeO_2C
H
O
O
O
H
H
10
syl-8-100_2.1
2021-03-12 15:39:34.750
SOLVENT: CDCl3
NA = 207
F1 = 75.467751 MHz
200
150
100
50
0 PPM

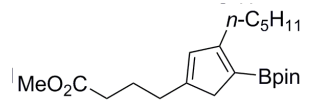

7.271
6.177
2.640
3.662
2.616
2.591
2.463
2.438
2.413
2.342
2.317
2.292
1.917
1.892
1.867
1.842
1.817
1.538
1.513
1.489
1.464
1.441
1.344
1.320
1.308
1.296
1.268
0.905
0.883
0.860
0.000

n-C5H11
MeO2C
Bpin

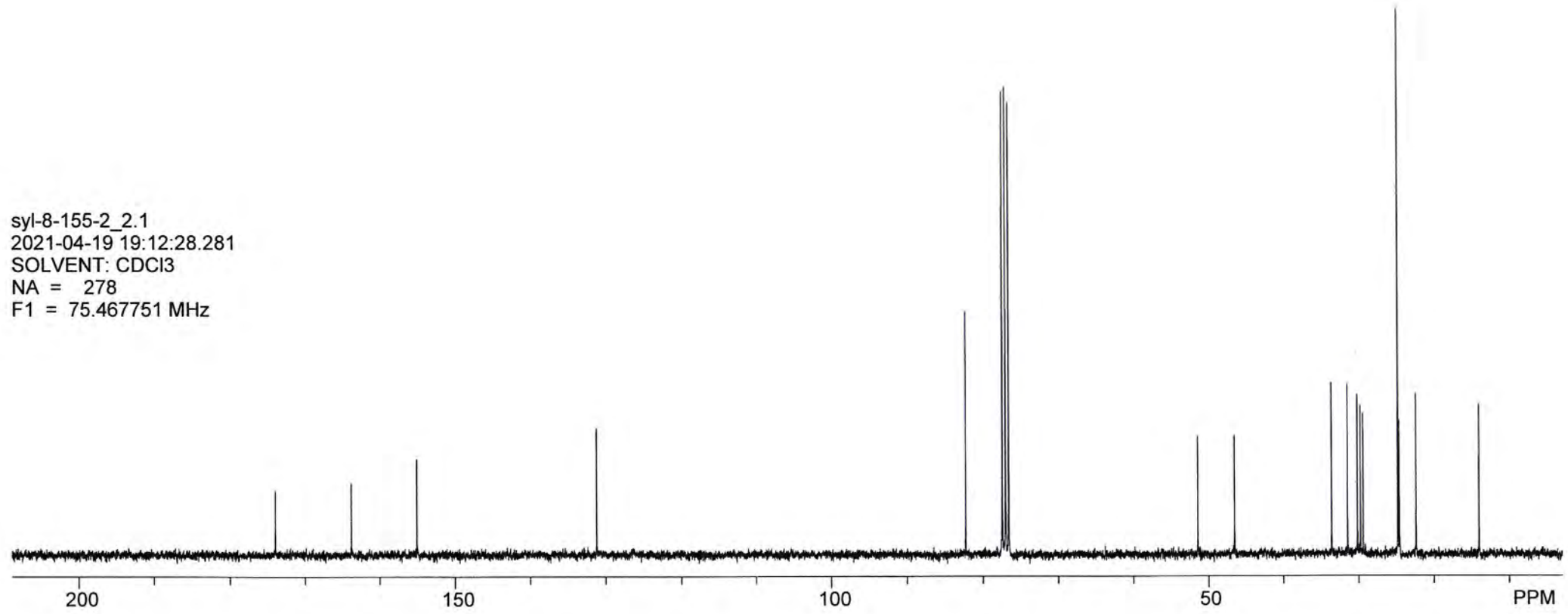
11

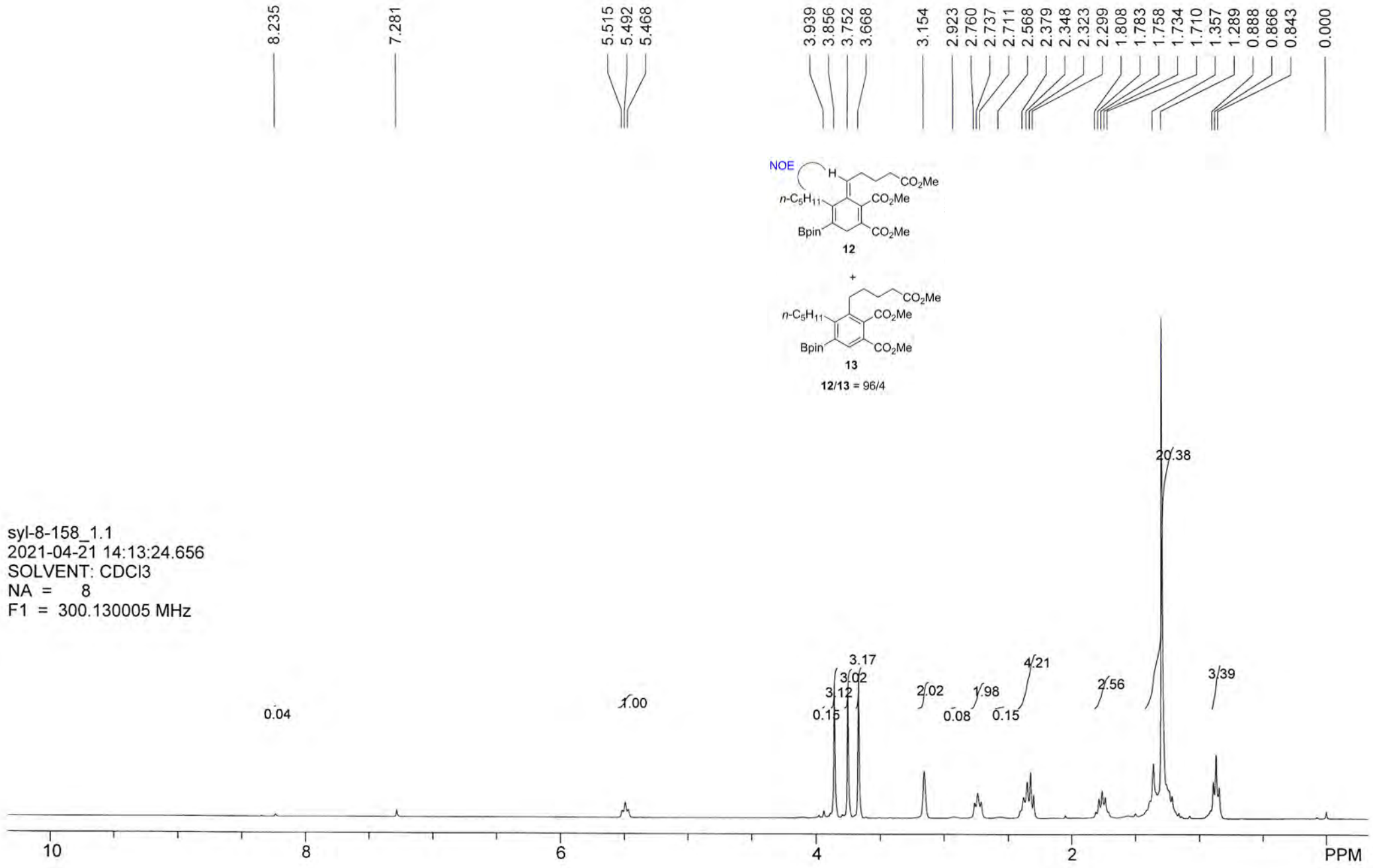

syl-8-155-2_1.1
2021-04-19 18:55:23.000
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
16.88
3.00
1.97
1.98
2.01
2.10
2.06
2.13
3.08
0.96
10
8
6
4
2
0
PPM

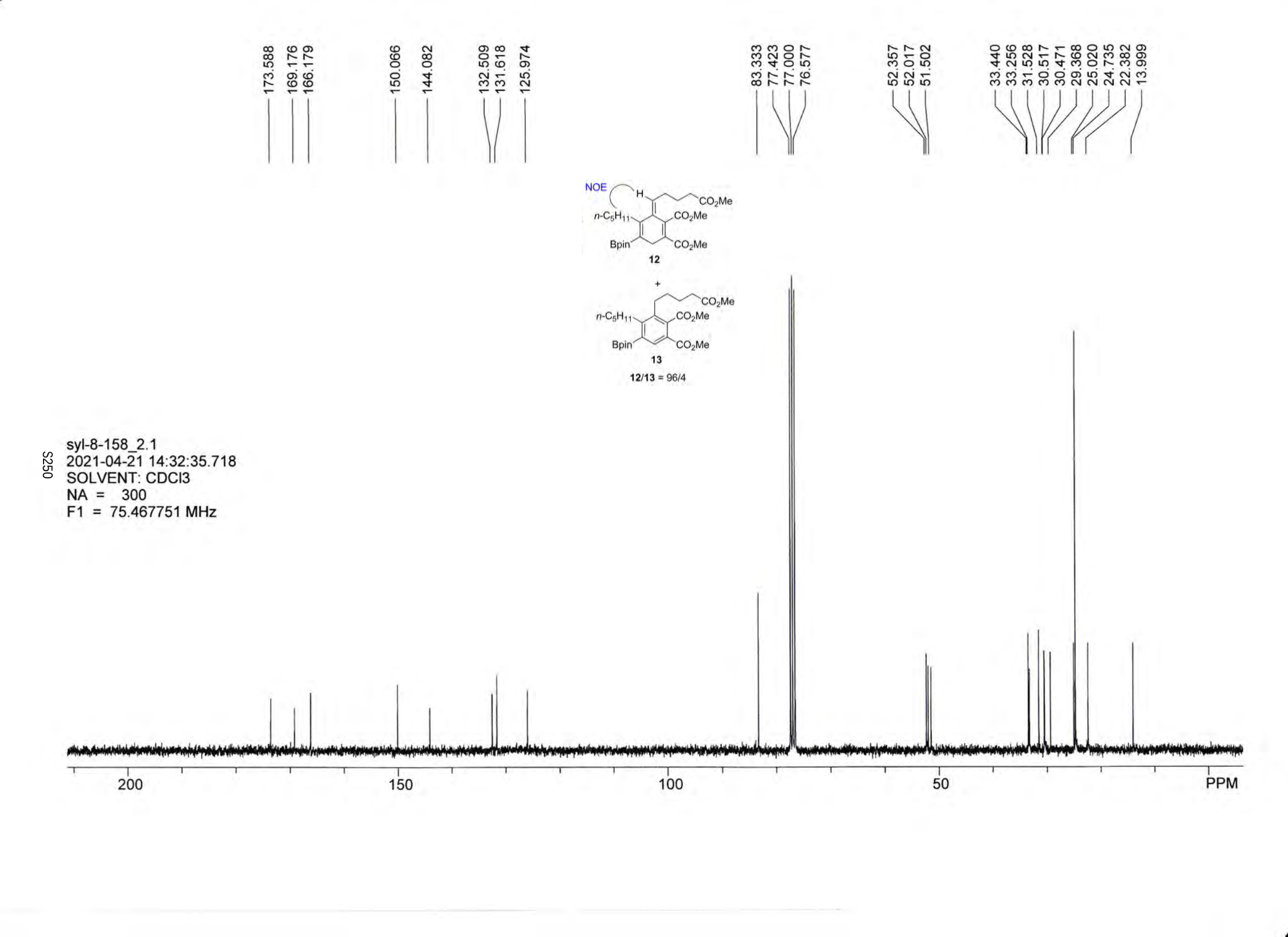

173.974
163.808
155.066
131.186
82.276
77.423
77.000
76.577
51.438
46.538
33.605
31.482
30.177
29.772
29.423
24.781
24.597
22.382
13.999

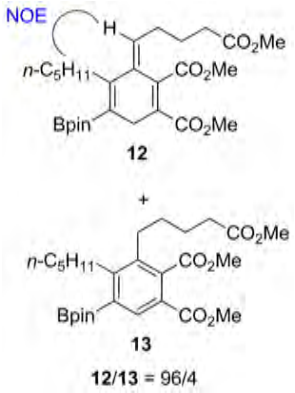

n-C5H11
MeO2C
Bpin

11

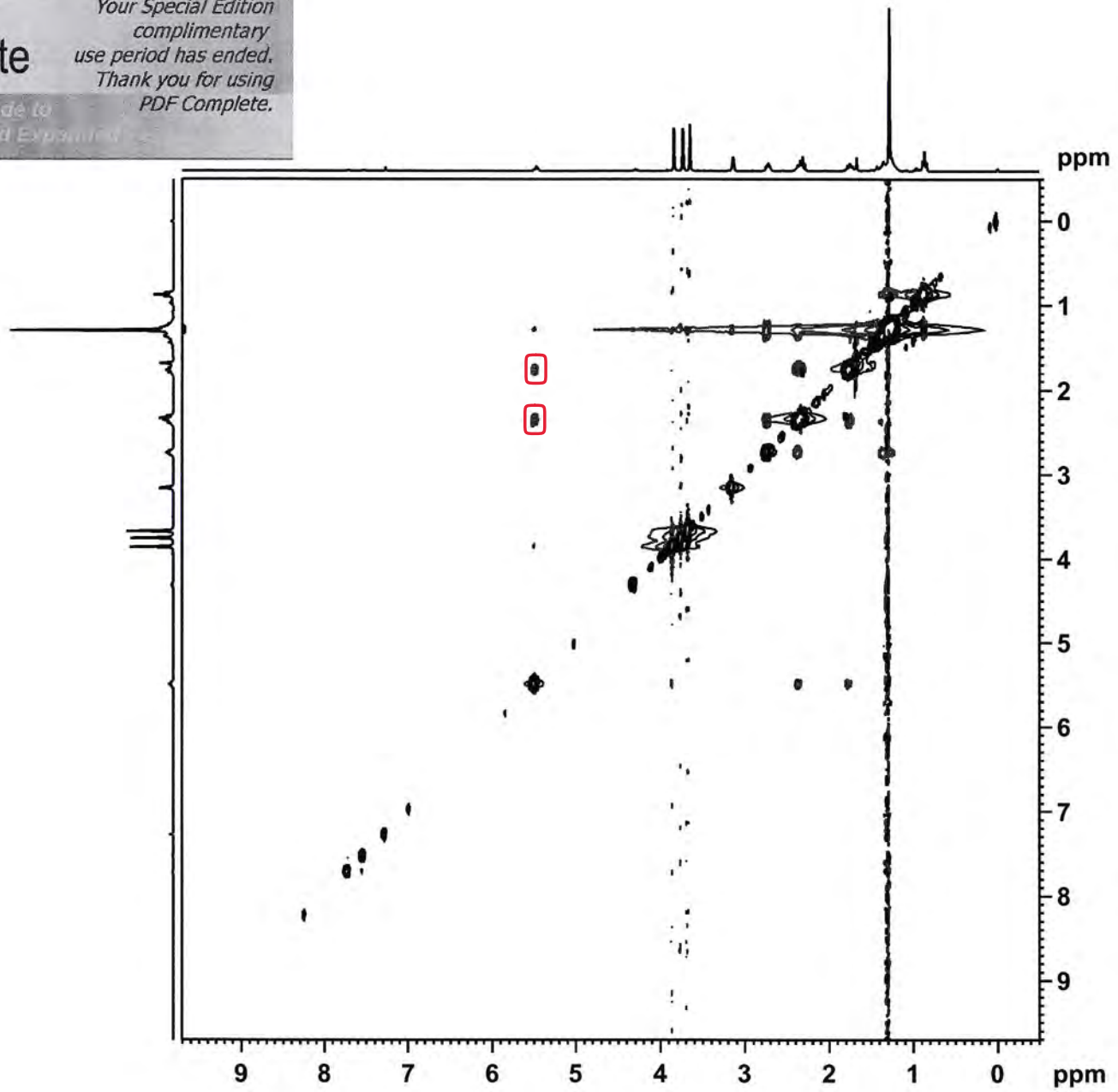

syl-8-155-2_2.1
2021-04-19 19:12:28.281
SOLVENT: CDCl3
NA = 278
F1 = 75.467751 MHz
200
150
100
50
PPM

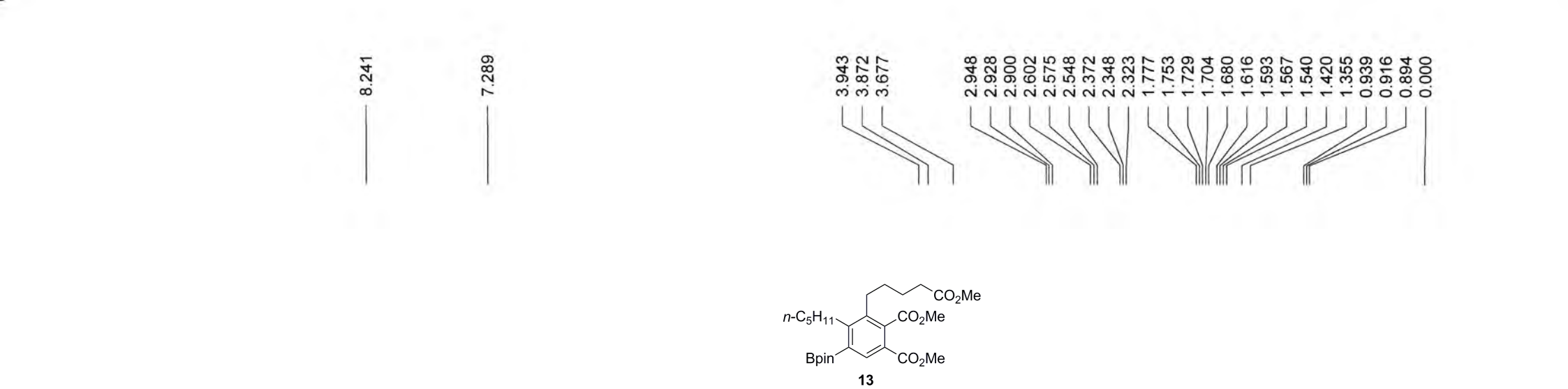

8.235
7.281
5.515
5.492
5.468
3.939
3.856
3.752
3.668
3.154
2.923
2.760
2.737
2.711
2.568
2.379
2.348
2.323
2.299
1.808
1.783
1.758
1.734
1.710
1.357
1.289
0.888
0.866
0.843
0.000
NOE
H
CO2Me
n-C5H11
CO2Me
Bpin
CO2Me
12
+
CO2Me
n-C5H11
CO2Me
Bpin
CO2Me
13
12/13 = 96/4
syl-8-158_1.1
2021-04-21 14:13:24.656
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
0.04
1.00
0.15
3.12
3.17
3.02
2.02
0.08
1.98
0.15
4.21
2.56
20.38
3.39
10
8
6
4
2
PPM


173.588
169.176
166.179
150.066
144.082
132.509
131.618
125.974
83.333
77.423
77.000
76.577
52.357
52.017
51.502
33.440
33.256
31.528
30.517
30.471
29.368
25.020
24.735
22.382
13.999
NOE
H
CO2Me
n-C5H11
CO2Me
Bpin
CO2Me
12
+
CO2Me
n-C5H11
CO2Me
Bpin
CO2Me
13
12/13 = 96/4
syl-8-158_2.1
2021-04-21 14:32:35.718
SOLVENT: CDCl3
NA = 300
F1 = 75.467751 MHz
200
150
100
50
PPM



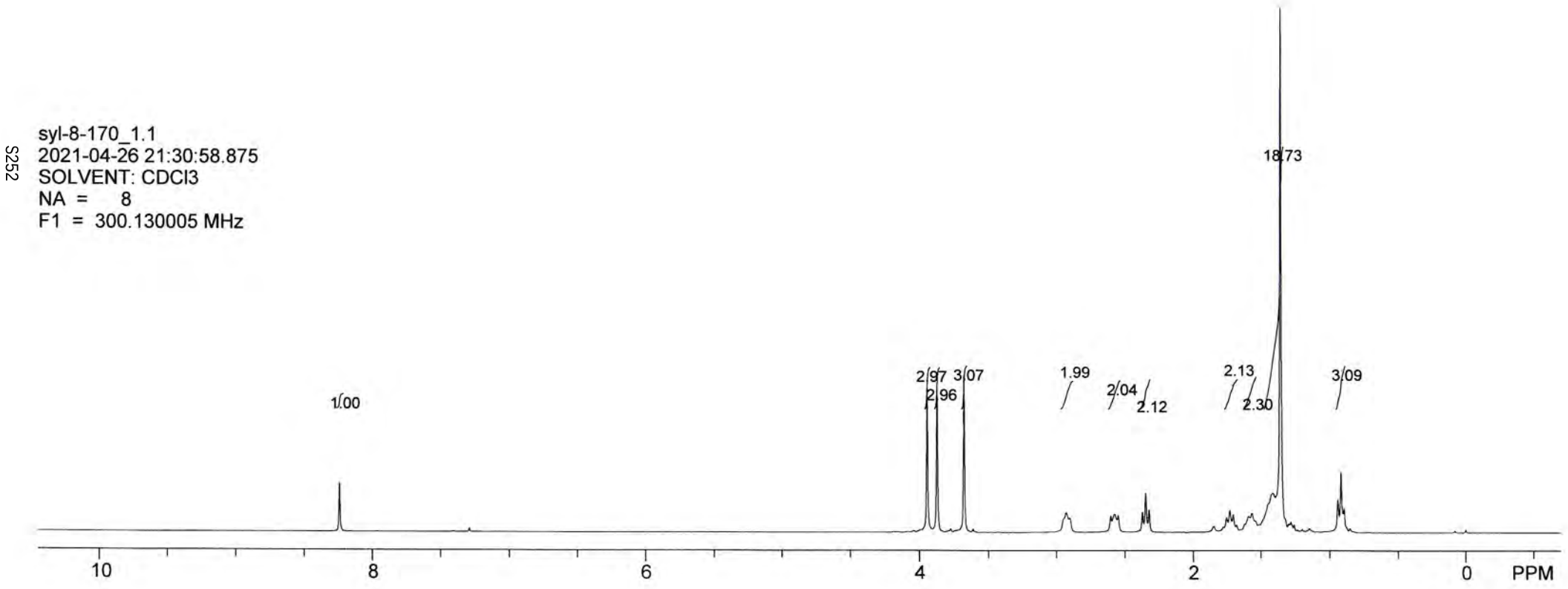
12/13 = 96/4



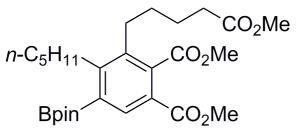

8.241
7.289
3.943
3.872
3.677
2.948
2.928
2.900
2.602
2.575
2.548
2.372
2.348
2.323
1.777
1.753
1.729
1.704
1.680
1.616
1.593
1.567
1.540
1.420
1.355
0.939
0.916
0.894
0.000

CO2Me
n-C5H11
CO2Me
Bpin
CO2Me

13

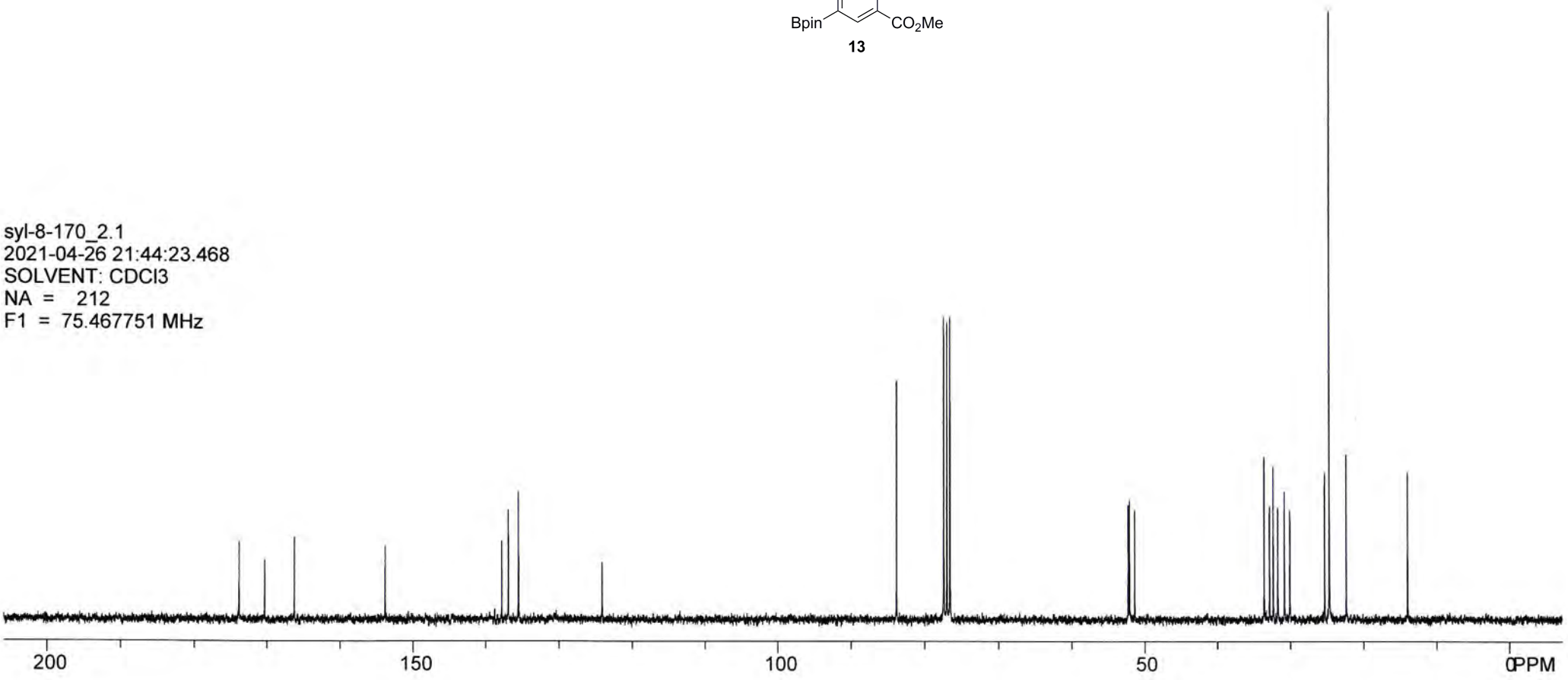

syl-8-170_1.1
2021-04-26 21:30:58.875
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
1.00
2.97
2.96
3.07
1.99
2.04
2.12
2.13
2.30
18.73
3.09
10
8
6
4
2
0
PPM

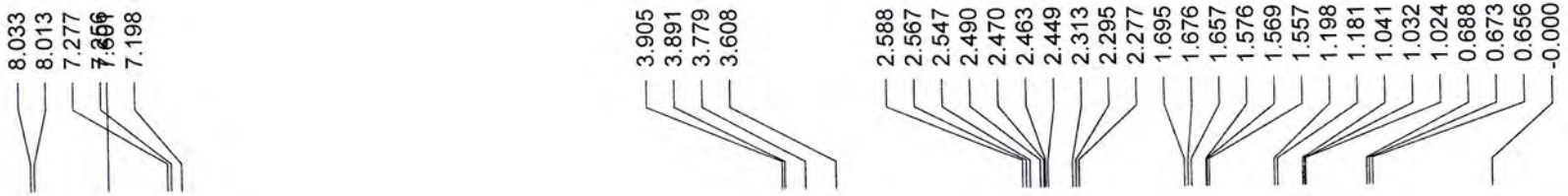
173.827
170.306
166.253
153.807
137.776
136.885
135.497
124.090
83.811
77.423
77.000
76.568
52.292
52.118
51.401
33.605
32.861
32.365
31.749
30.839
30.103
25.342
24.717
22.401
13.999



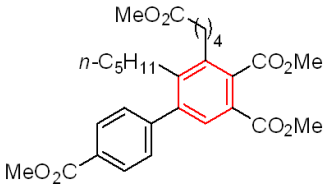
13

syl-8-170_2.1
2021-04-26 21:44:23.468
SOLVENT: CDCl3
NA = 212
F1 = 75.467751 MHz

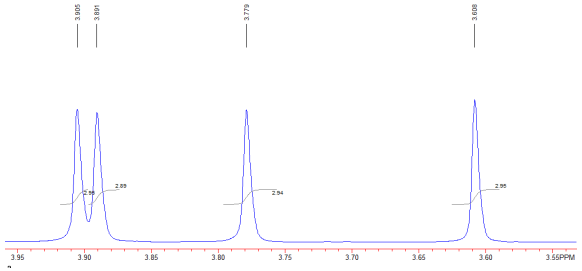


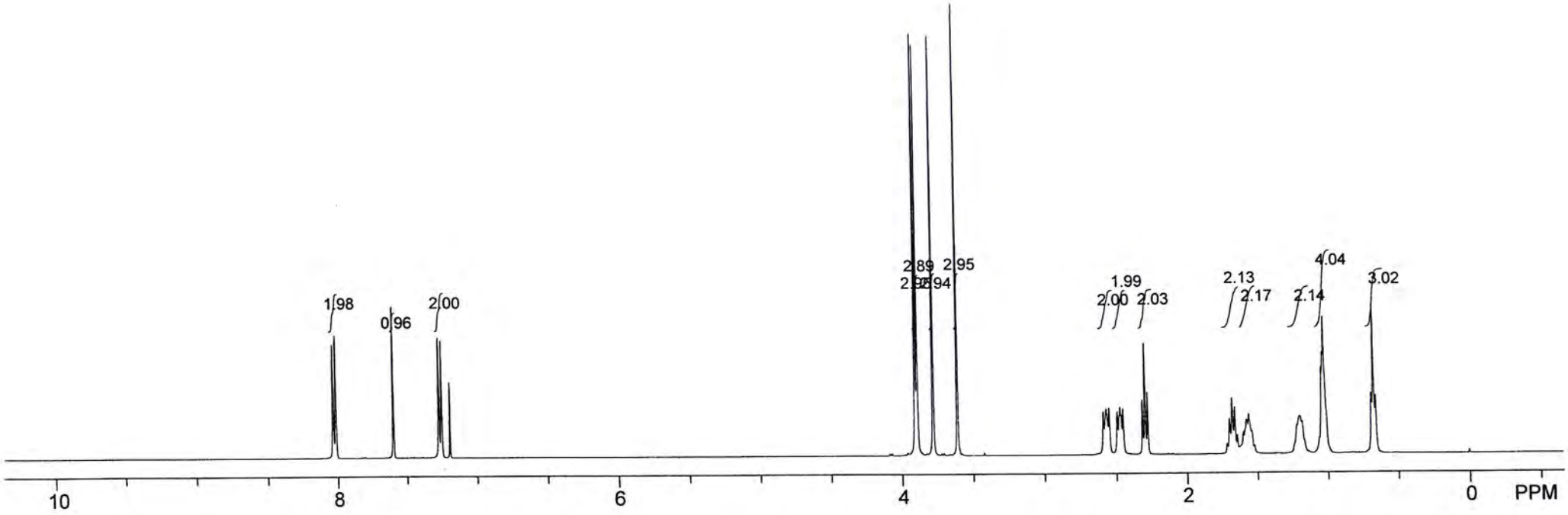


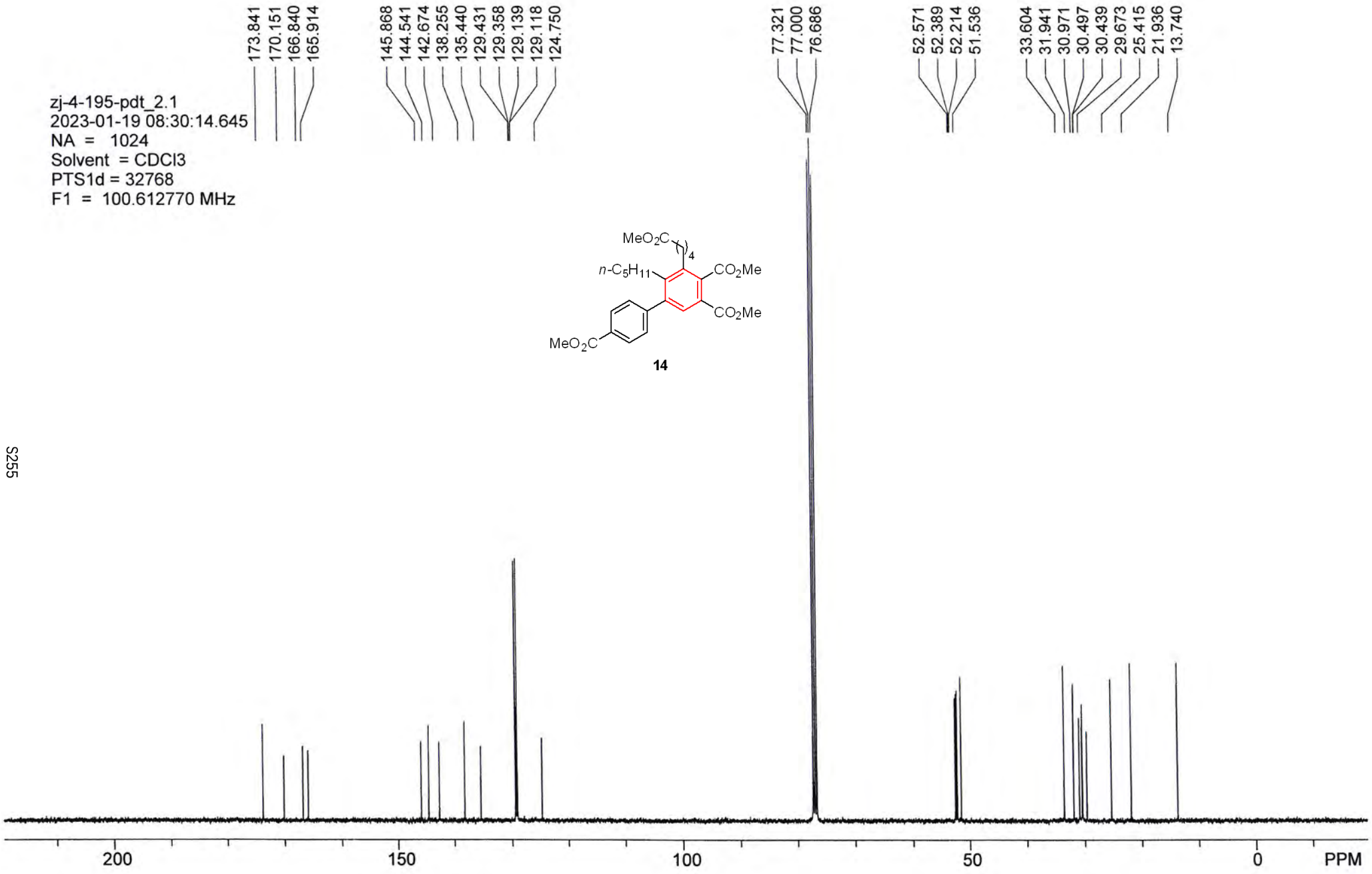
zj-4-195-pdt_1.1
2023-01-19 07:31:17.517
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130035 MHz

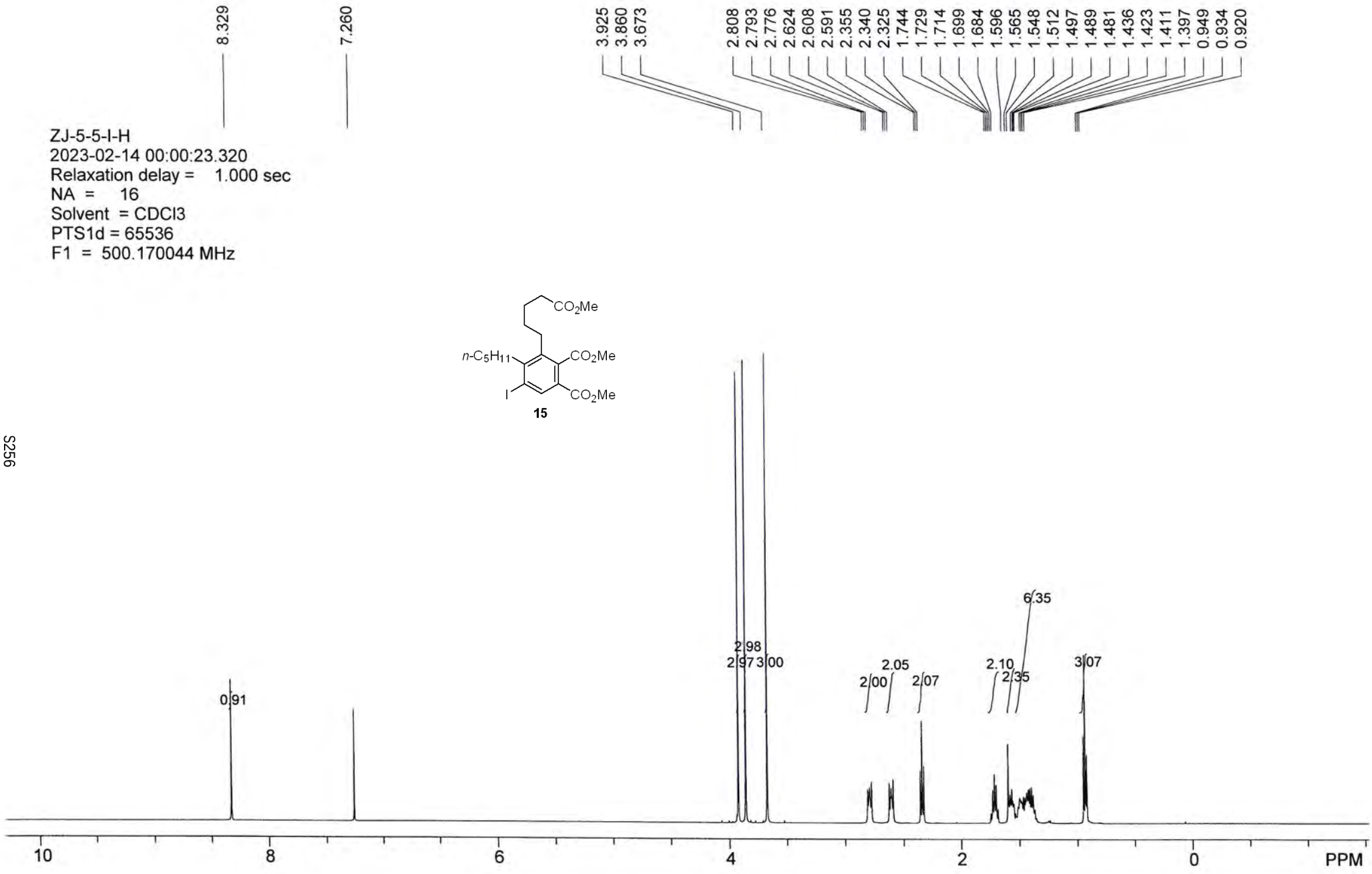


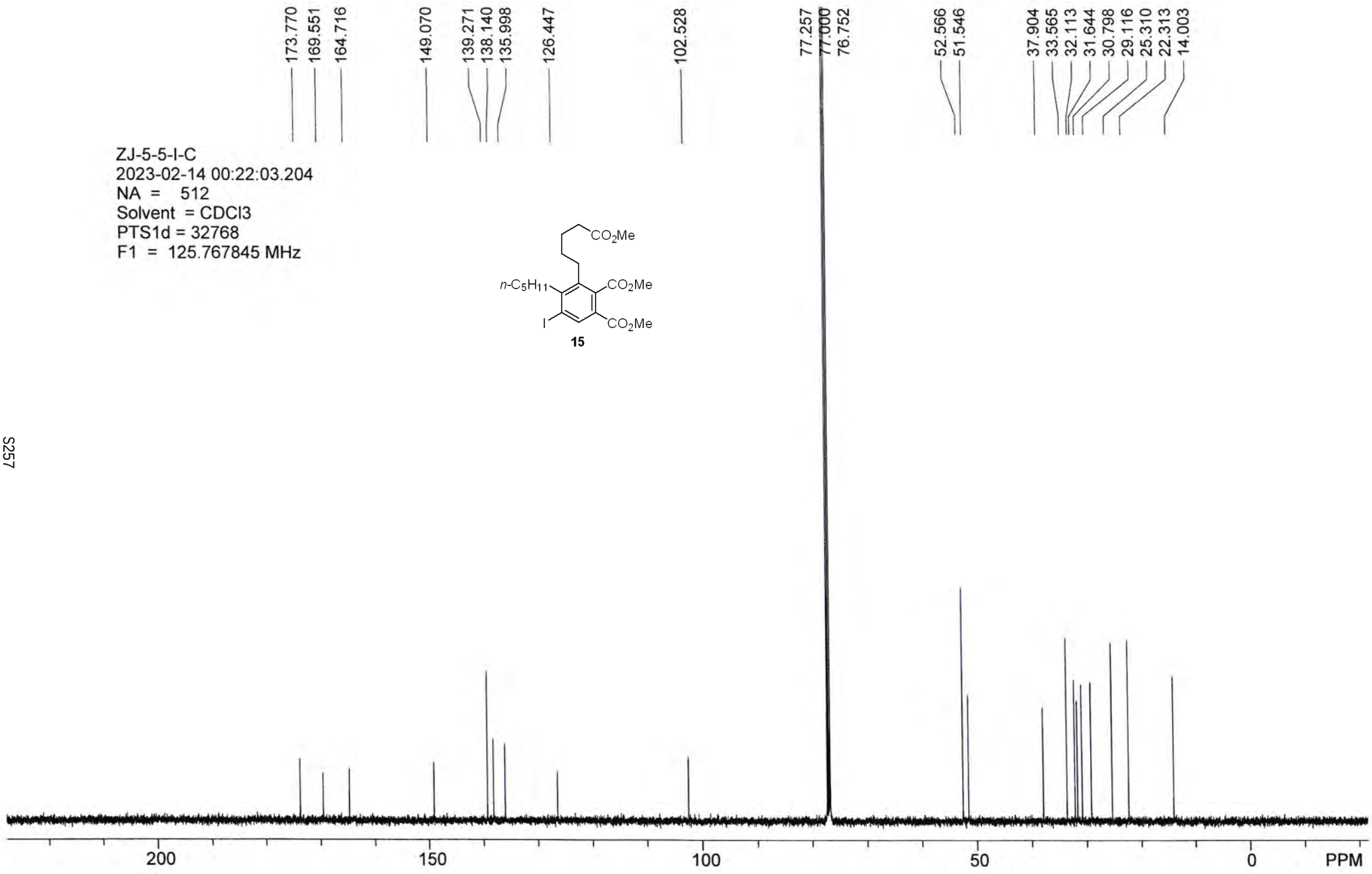
14

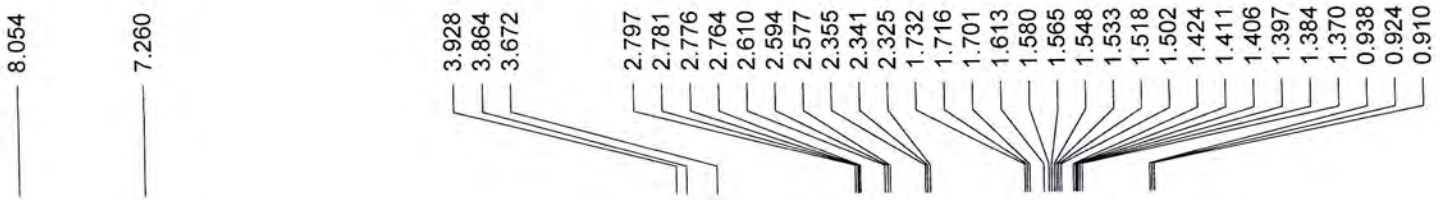




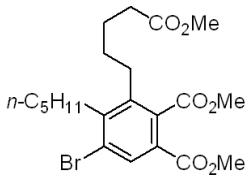

173.841
170.151
166.840
165.914
145.868
144.541
142.674
138.255
135.440
129.431
129.358
129.139
129.118
124.750
77.321
77.000
76.686
52.571
52.389
52.214
51.536
33.604
31.941
30.971
30.497
30.439
29.673
25.415
21.936
13.740
zj-4-195-pdt_2.1
2023-01-19 08:30:14.645
NA = 1024
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
MeO2C
4
n-C5H11
CO2Me
CO2Me
MeO2C
14
200
150
100
50
0
PPM


8.329
7.260
3.925
3.860
3.673
2.808
2.793
2.776
2.624
2.608
2.591
2.355
2.340
2.325
1.744
1.729
1.714
1.699
1.684
1.596
1.565
1.548
1.512
1.497
1.489
1.481
1.436
1.423
1.411
1.397
0.949
0.934
0.920
ZJ-5-5-I-H
2023-02-14 00:00:23.320
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 500.170044 MHz
CO2Me
n-C5H11
CO2Me
I
CO2Me
15
0.91
2.98
2.97
3.00
2.00
2.05
2.07
2.10
2.35
6.35
3.07
10
8
6
4
2
0
PPM

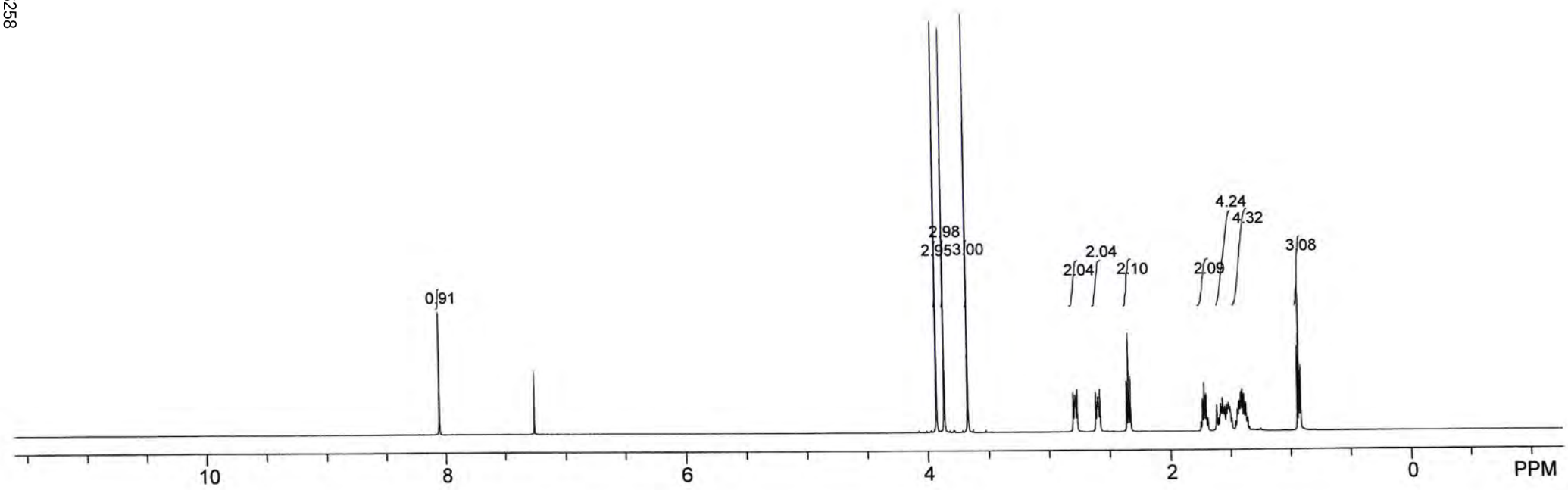

173.770
169.551
164.716
149.070
139.271
138.140
135.998
126.447
102.528
77.257
77.000
76.752
52.566
51.546
37.904
33.565
32.113
31.644
30.798
29.116
25.310
22.313
14.003
ZJ-5-5-I-C
2023-02-14 00:22:03.204
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 125.767845 MHz
CO2Me
n-C5H11
CO2Me
I
CO2Me
15
200
150
100
50
0
PPM

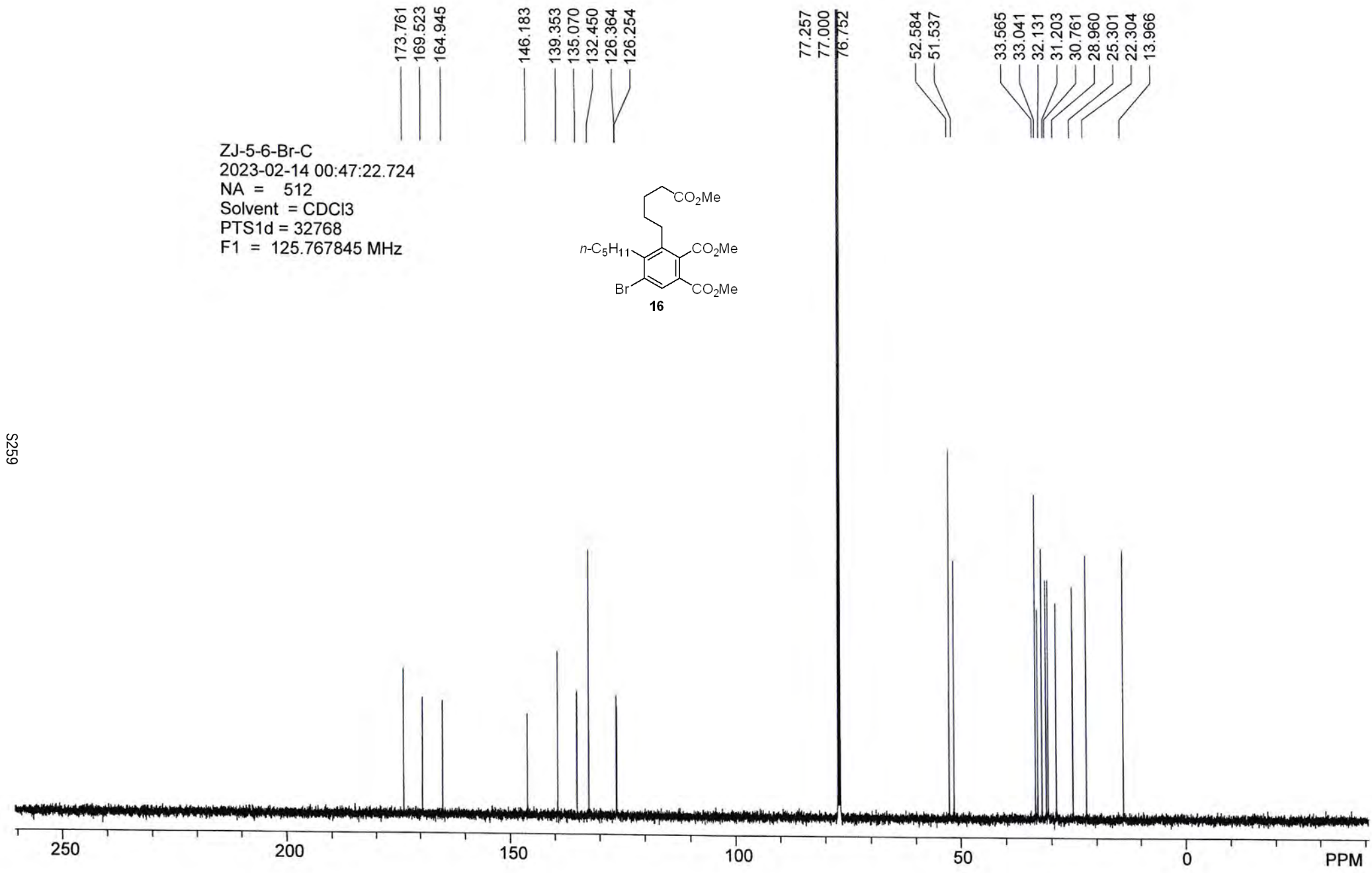


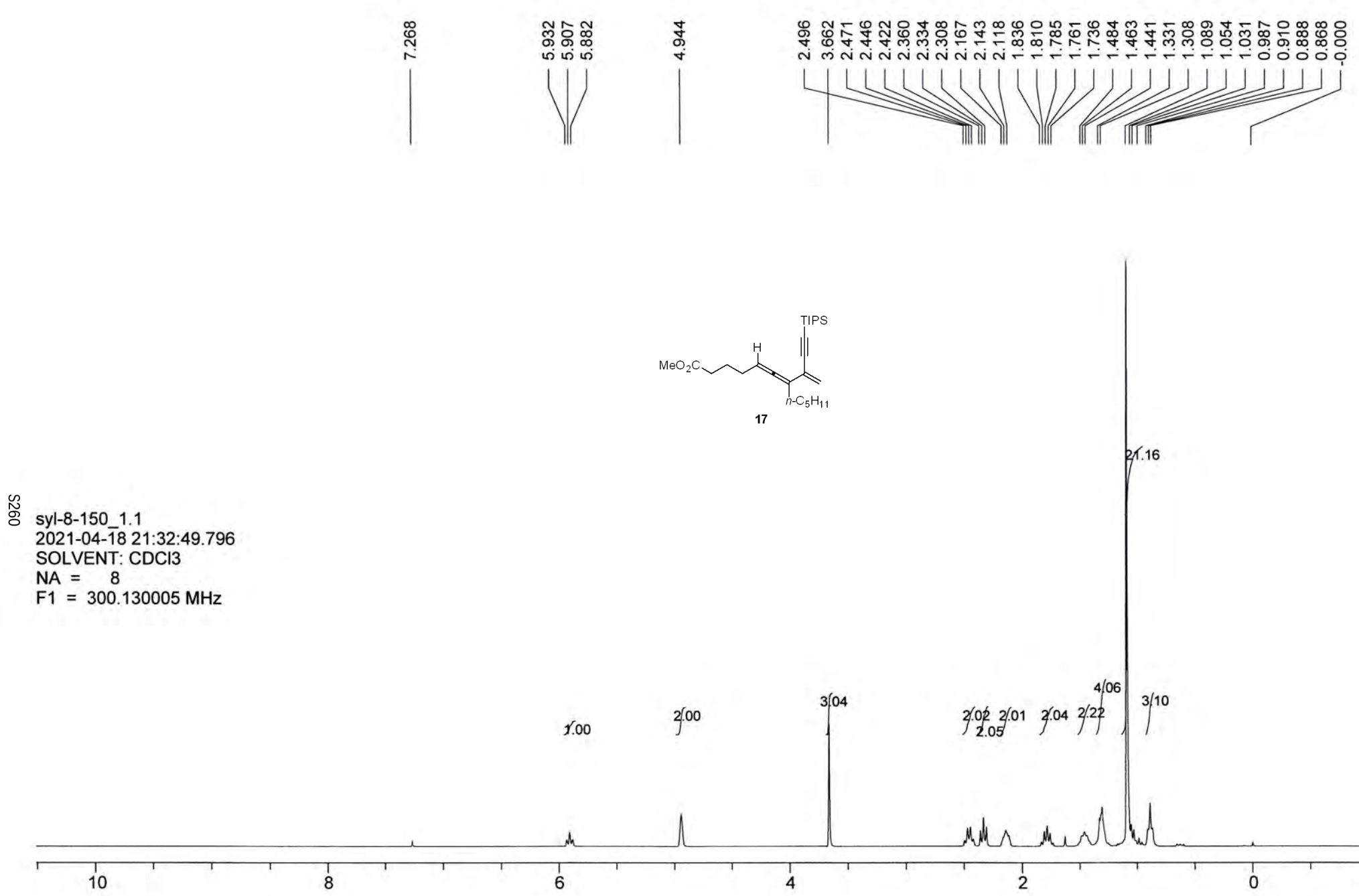
ZJ-5-6-Br-H
2023-02-14 00:26:12.412
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 500.170044 MHz

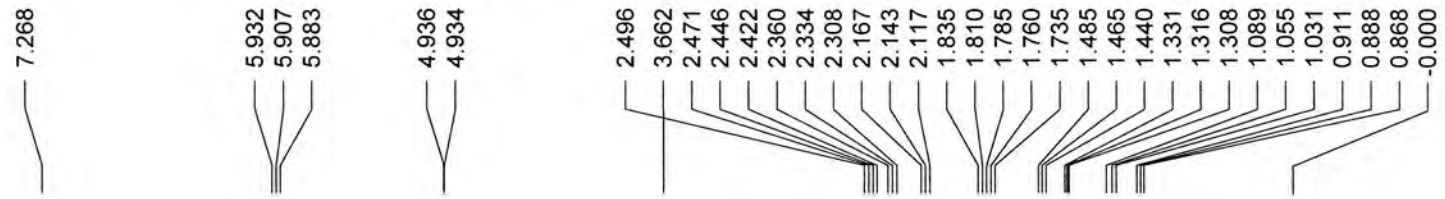


16

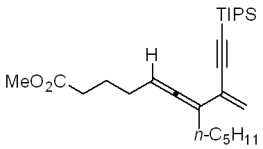



173.761
169.523
164.945
146.183
139.353
135.070
132.450
126.364
126.254
77.257
77.000
76.752
52.584
51.537
33.565
33.041
32.131
31.203
30.761
28.960
25.301
22.304
13.966
ZJ-5-6-Br-C
2023-02-14 00:47:22.724
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 125.767845 MHz
CO2Me
n-C5H11
CO2Me
Br
CO2Me
16
250
200
150
100
50
0
PPM


7.268
5.932
5.907
5.882
4.944
2.496
3.662
2.471
2.446
2.422
2.360
2.334
2.308
2.167
2.143
2.118
1.836
1.810
1.785
1.761
1.736
1.484
1.463
1.441
1.331
1.308
1.089
1.054
1.031
0.987
0.910
0.888
0.868
-0.000
TIPS
H
MeO2C
n-C5H11
17
syl-8-150_1.1
2021-04-18 21:32:49.796
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
1.00
2.00
3.04
2.02
2.05
2.01
2.04
2.22
4.06
21.16
3.10
10
8
6
4
2
0
PPM

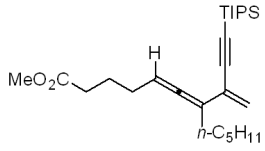


Purity (97%) is determined by CH_2Br_2 (7 μL, 0.1 mmol) as the internal standard in 40.6 mg of sample.



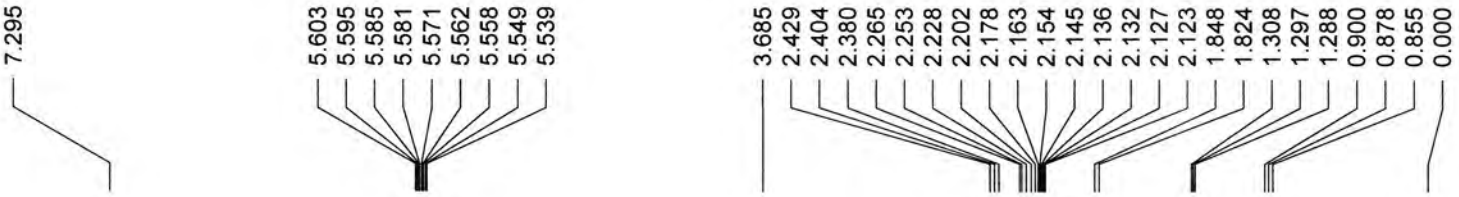
17

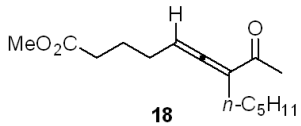
209.114
173.964
135.690
121.957
104.925
103.132
95.659
77.956
77.423
77.000
76.577
51.456
33.578
31.620
30.462
28.936
27.392
24.404
22.493
18.632
14.045
11.278



17

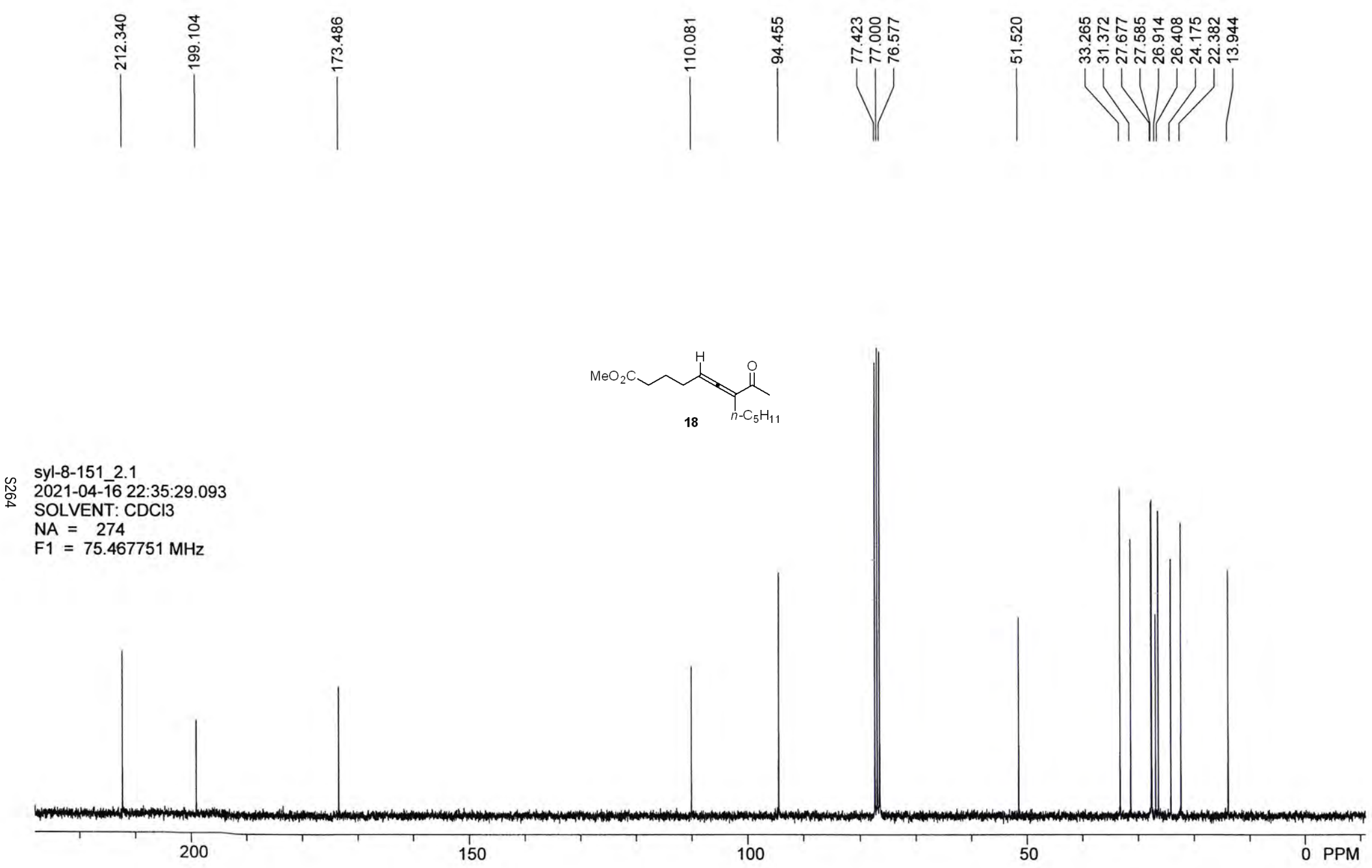
syl-8-150_2.1
2021-04-18 21:49:44.750
SOLVENT: CDCl3
NA = 273
F1 = 75.467751 MHz

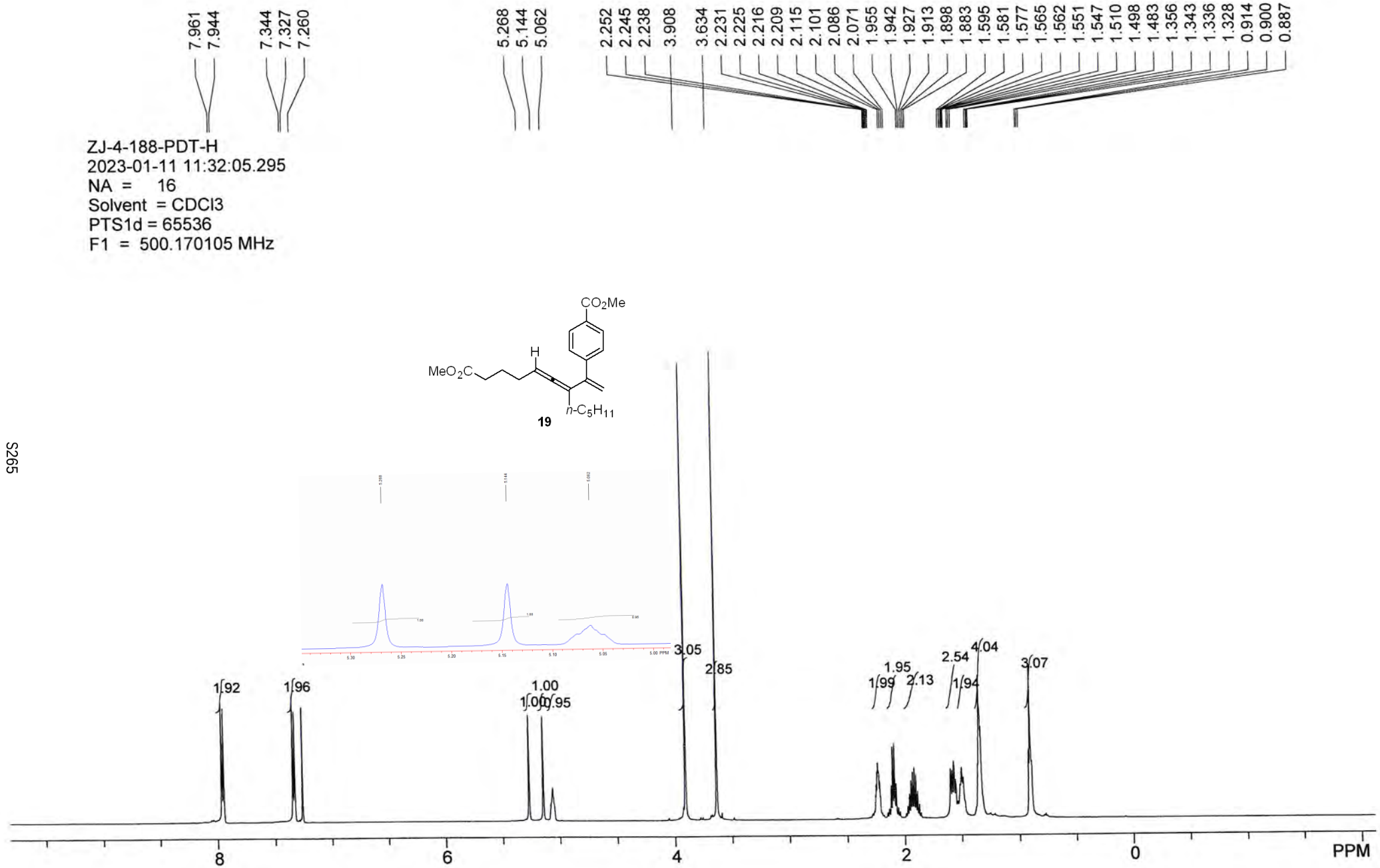

7.295
5.603
5.595
5.585
5.581
5.571
5.562
5.558
5.549
5.539
3.685
2.429
2.404
2.380
2.265
2.253
2.228
2.202
2.178
2.163
2.154
2.145
2.136
2.132
2.127
2.123
1.848
1.824
1.308
1.297
1.288
0.900
0.878
0.855
0.000

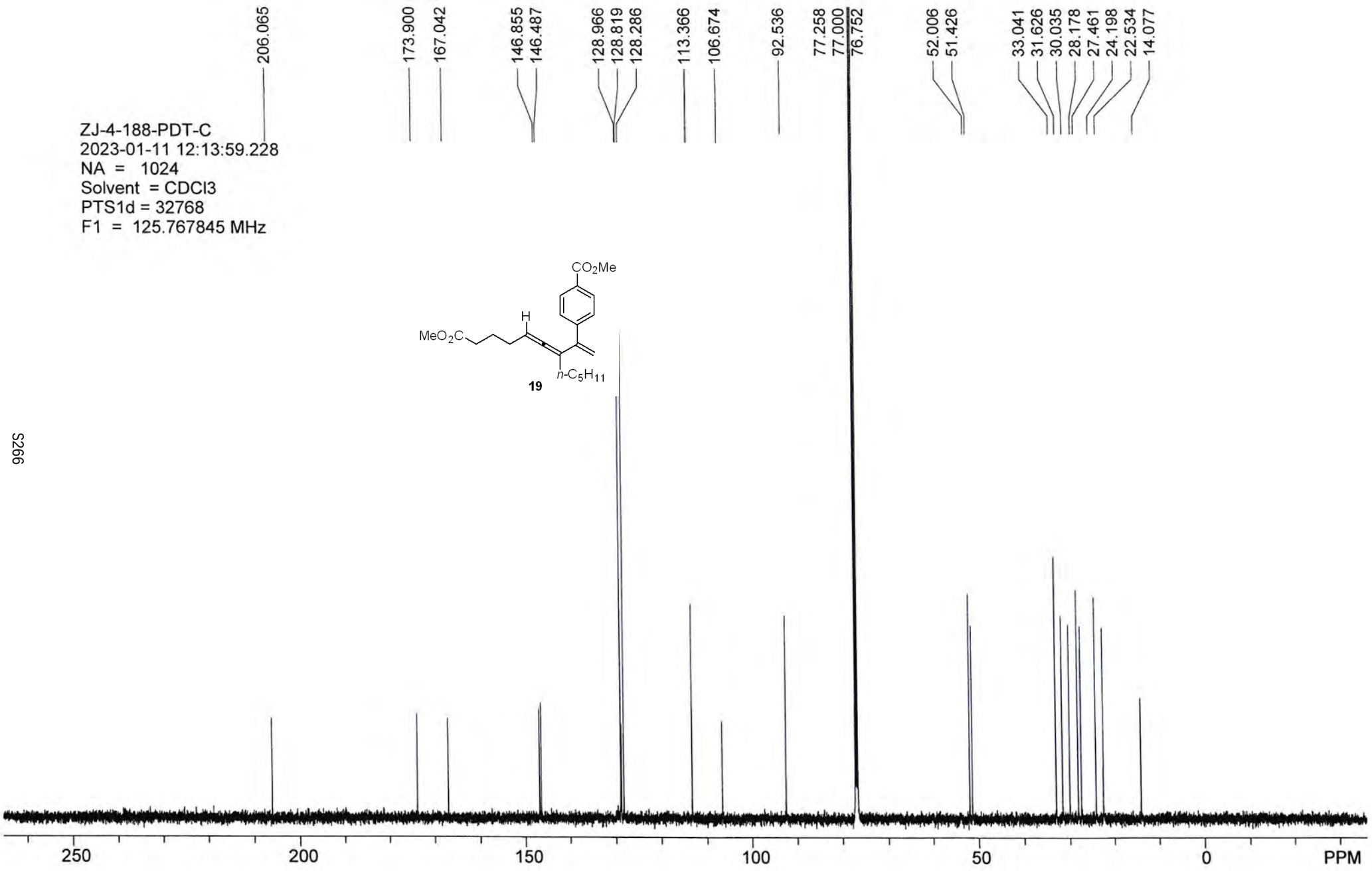

MeO2C
H
O
n-C5H11

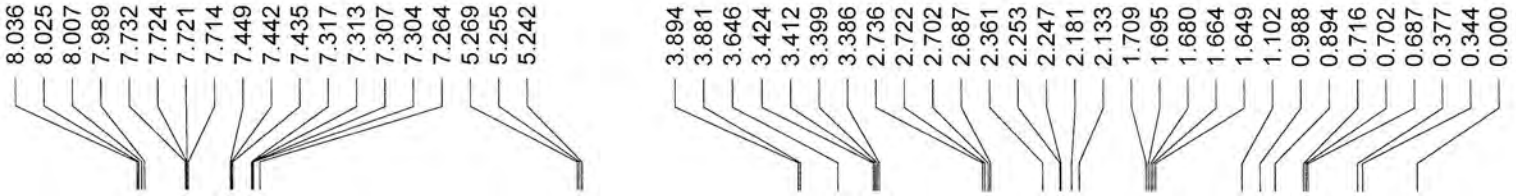
18

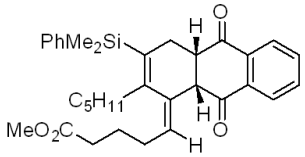
syl-8-151_1.1
2021-04-16 22:18:41.796
SOLVENT: CDCl3
NA = 8
F1 = 300.130005 MHz
1.00
3.00
2.05
2.96
4.13
2.20
6.19
3.00
10
8
6
4
2
0
PPM


212.340
199.104
173.486
110.081
94.455
77.423
77.000
76.577
51.520
33.265
31.372
27.677
27.585
26.914
26.408
24.175
22.382
13.944
MeO2C
H
O
n-C5H11
18
syl-8-151_2.1
2021-04-16 22:35:29.093
SOLVENT: CDCl3
NA = 274
F1 = 75.467751 MHz
200
150
100
50
0 PPM

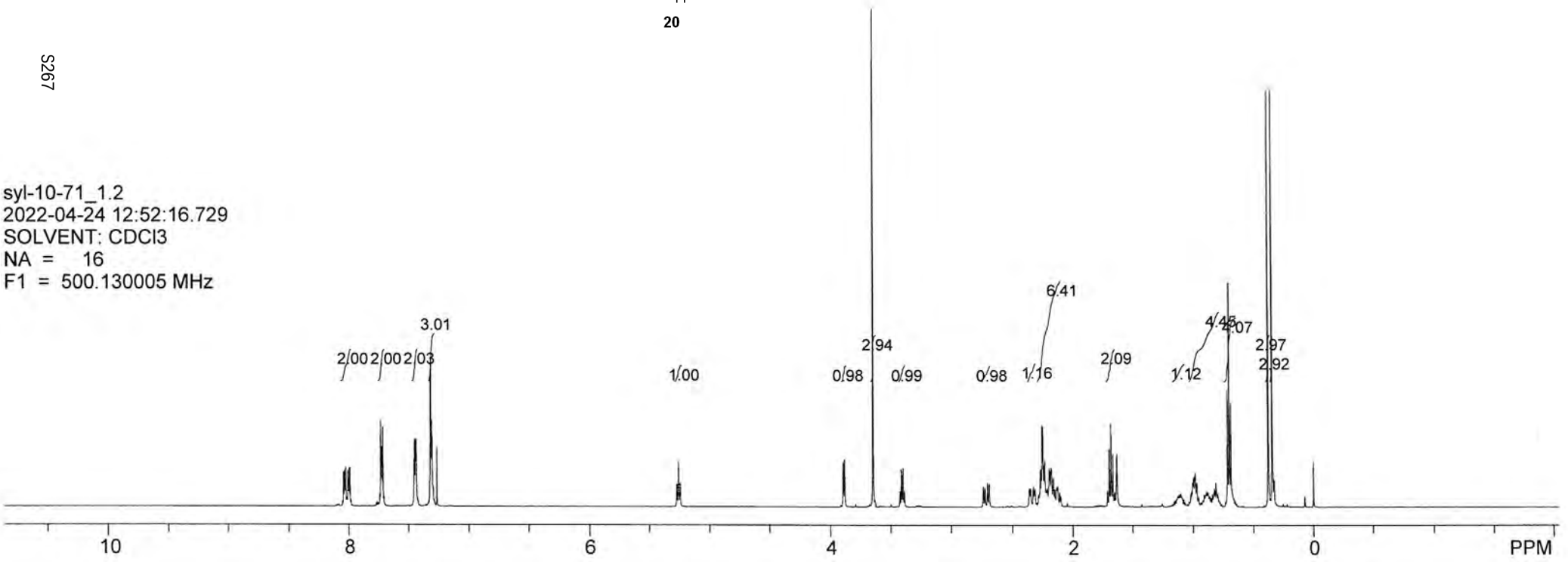

7.961
7.944
7.344
7.327
7.260
5.268
5.144
5.062
2.252
2.245
2.238
3.908
3.634
2.231
2.225
2.216
2.209
2.115
2.101
2.086
2.071
1.955
1.942
1.927
1.913
1.898
1.883
1.595
1.581
1.577
1.565
1.562
1.551
1.547
1.510
1.498
1.483
1.356
1.343
1.336
1.328
0.914
0.900
0.887
ZJ-4-188-PDT-H
2023-01-11 11:32:05.295
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 500.170105 MHz
CO2Me
H
MeO2C
n-C5H11
19
1.92
1.96
1.00
1.00
0.95
3.05
2.85
1.99
1.95
2.13
2.54
1.94
4.04
3.07
8
6
4
2
0
PPM


206.065
173.900
167.042
146.855
146.487
128.966
128.819
128.286
113.366
106.674
92.536
77.258
77.000
76.752
52.006
51.426
33.041
31.626
30.035
28.178
27.461
24.198
22.534
14.077
ZJ-4-188-PDT-C
2023-01-11 12:13:59.228
NA = 1024
Solvent = CDCl3
PTS1d = 32768
F1 = 125.767845 MHz
CO2Me
H
MeO2C
n-C5H11
19
250
200
150
100
50
0
PPM

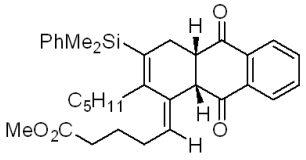

8.036
8.025
8.007
7.989
7.732
7.724
7.721
7.714
7.449
7.442
7.435
7.317
7.313
7.307
7.304
7.264
5.269
5.255
5.242
3.894
3.881
3.646
3.424
3.412
3.399
3.386
2.736
2.722
2.702
2.687
2.361
2.253
2.247
2.181
2.133
1.709
1.695
1.680
1.664
1.649
1.102
0.988
0.894
0.716
0.702
0.687
0.377
0.344
0.000


PhMe2Si
C5H11
MeO2C
H
O
H
O
H

20


syl-10-71_1.2
2022-04-24 12:52:16.729
SOLVENT: CDCl3
NA = 16
F1 = 500.130005 MHz
2.00
2.00
2.03
3.01
1.00
0.98
2.94
0.99
0.98
1.16
6.41
2.09
1.12
4.45
2.07
2.97
2.92
10
8
6
4
2
0
PPM

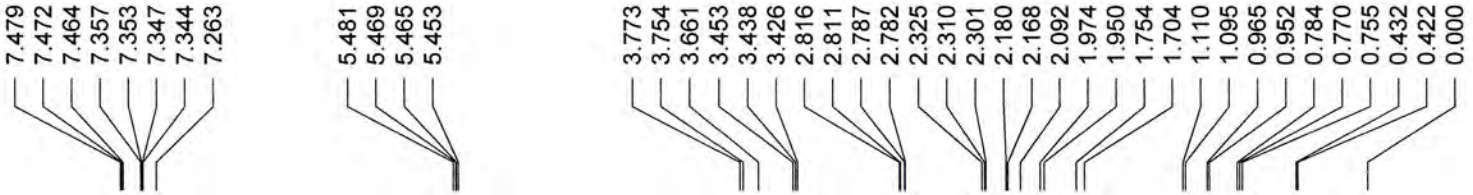
198.030
196.982
173.705
149.057
135.442
134.339
139.285
134.210
134.072
133.943
133.594
131.958
130.910
128.795
127.747
126.874
126.671
77.248
77.000
76.743
57.795
51.479
48.399
36.457
33.414
31.879
31.392
28.891
28.772
25.122
22.401
13.970
-1.042
-1.070

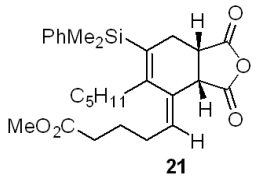


20

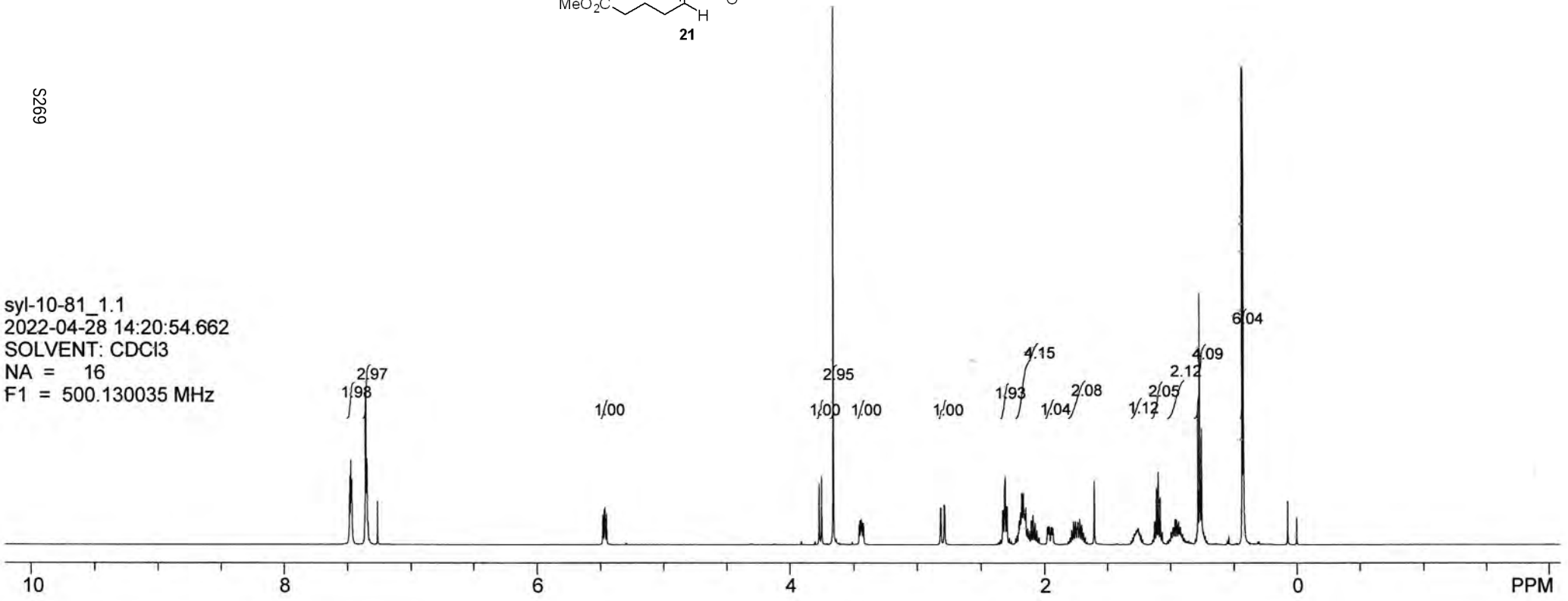
S268

syl-10-71_2.1
2022-04-24 13:09:58.385
SOLVENT: CDCl3
NA = 400
F1 = 125.757660 MHz

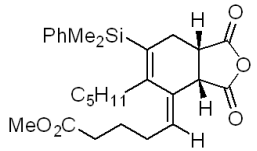

7.479
7.472
7.464
7.357
7.353
7.347
7.344
7.263
5.481
5.469
5.465
5.453
3.773
3.754
3.661
3.453
3.438
3.426
2.816
2.811
2.787
2.782
2.325
2.310
2.301
2.180
2.168
2.092
1.974
1.950
1.754
1.704
1.110
1.095
0.965
0.952
0.784
0.770
0.755
0.432
0.422
0.000


PhMe2Si
C5H11
MeO2C
H
O
O
O
H
H

21

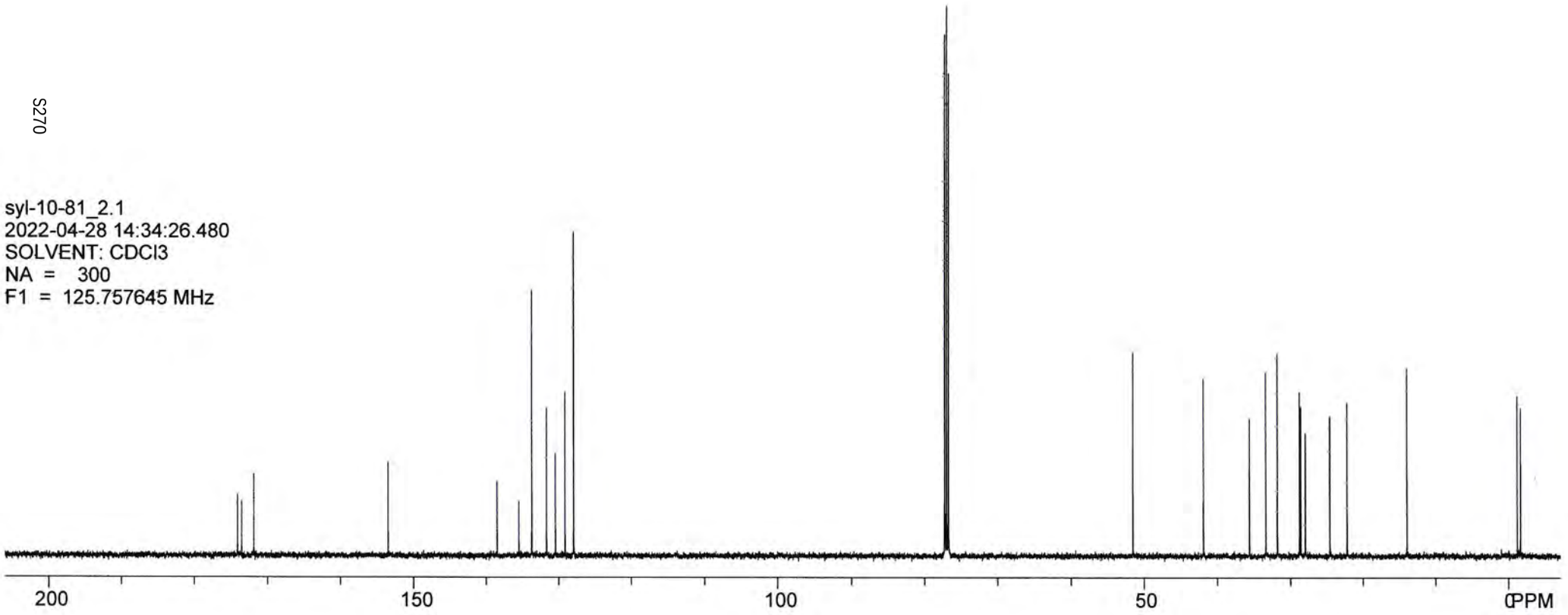

syl-10-81_1.1
2022-04-28 14:20:54.662
SOLVENT: CDCl3
NA = 16
F1 = 500.130035 MHz
1.98
2.97
1.00
2.95
1.00
1.00
1.00
1.93
4.15
1.04
2.08
1.12
2.05
2.12
4.09
6.04
10
8
6
4
2
0
PPM

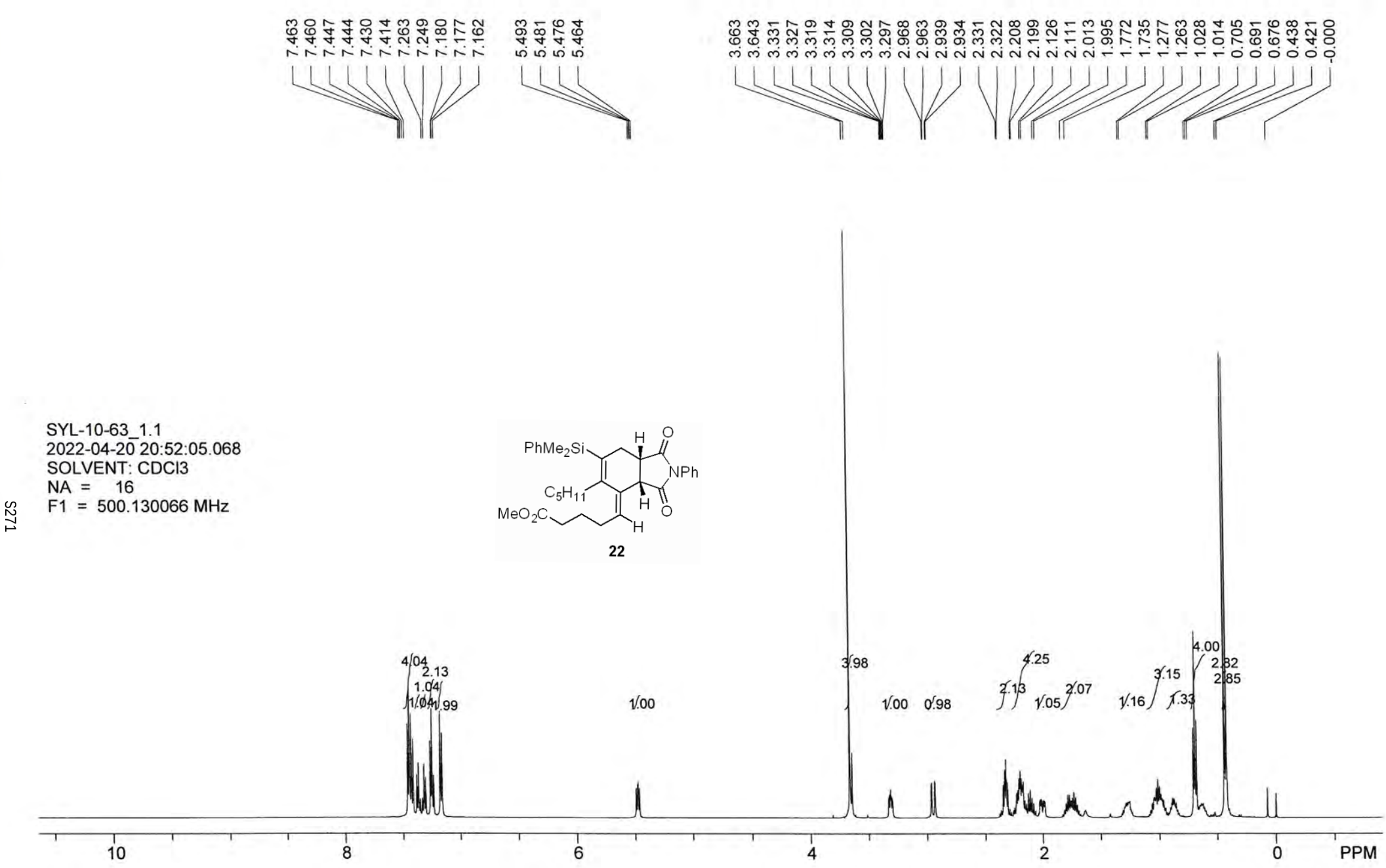
174.081
173.530
171.866
153.470
138.457
135.451
133.658
131.645
130.413
129.098
127.922
77.257
77.000
76.743
51.553
41.909
35.612
33.378
31.796
28.744
28.560
27.935
24.552
22.171
13.915
-1.199
-1.658



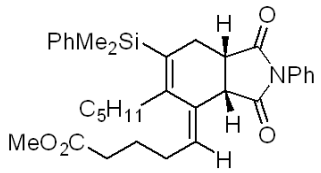
21

syl-10-81_2.1
2022-04-28 14:34:26.480
SOLVENT: CDCl3
NA = 300
F1 = 125.757645 MHz




7.463
7.460
7.447
7.444
7.430
7.414
7.263
7.249
7.180
7.177
7.162
5.493
5.481
5.476
5.464
3.663
3.643
3.331
3.327
3.319
3.314
3.309
3.302
3.297
2.968
2.963
2.939
2.934
2.331
2.322
2.208
2.199
2.126
2.111
2.013
1.995
1.772
1.735
1.277
1.263
1.028
1.014
0.705
0.691
0.676
0.438
0.421
-0.000
SYL-10-63_1.1
2022-04-20 20:52:05.068
SOLVENT: CDCl3
NA = 16
F1 = 500.130066 MHz
PhMe2Si
C5H11
MeO2C
NPh
H
O
22
4.04
2.13
1.04
1.04
1.99
1.00
3.98
1.00
0.98
4.25
2.13
1.05
2.07
1.16
3.15
1.33
4.00
2.82
2.85
10
8
6
4
2
0
PPM

178.779
176.499
173.659
153.249
138.935
134.559
133.732
132.344
132.105
130.588
128.961
128.878
128.381
127.784
126.267
77.257
77.000
76.752
51.498
51.268
41.109
35.832
33.414
31.851
29.139
28.827
27.953
24.727
22.171
13.787
-1.033
-1.603



22

SYL-10-63_2.1
2022-04-20 21:14:04.391
SOLVENT: CDCl3
NA = 512
F1 = 125.757599 MHz