Iron-catalyzed Aerobic Oxidation of Silyl Ethers to Carboxylic Acids

Zuizhi Lin[a,b] and Shengming Ma*[,a,c,d]

[a] State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 345 Lingling Lu, Shanghai 200032, China
E-mail: masm@sioc.ac.cn

[b] University of Chinese Academy of Sciences, Beijing 100049, China

[c] Research Center for Molecular Recognition and Synthesis, Department of Chemistry, Fudan University, 220 Handan Lu, Shanghai 200433, China

[d] Laboratory of Molecular Recognition and Synthesis, Department of Chemistry Zhejiang University Hangzhou, Zhejiang 310027, P. R. China.

General Information

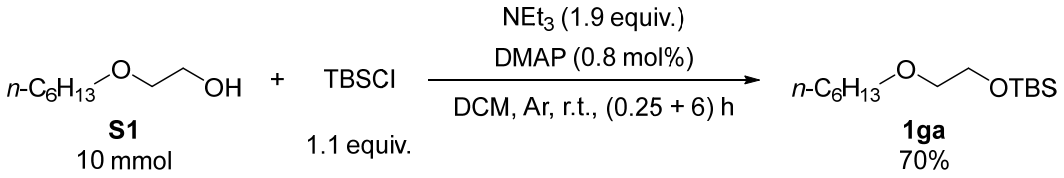
NMR spectra were taken with Agilent, Varian Mercury, Bruker 400 MHz NMR spectrometer (400 MHz for ^{1}H NMR; 100 MHz for ^{13}C NMR) or Bruker 300 MHz NMR spectrometer (300 MHz for ^{1}H NMR; 75 MHz for ^{13}C NMR). $Fe(NO_3)_3 \cdot 9H_2O$ and KCl were purchased from Sinopharm Chemical Reagent Co., Ltd. or Shanghai Macklin Biochemical Co., Ltd.; TEMPO was purchased from Anhui Zesheng Technology Co., Ltd.; 4-OH-TEMPO and alcohol **S1** were purchased from Shanghai Adamas Reagent Co., Ltd. or Shanghai Darui Fine Chemical Co., Ltd.; Petroleum ether (b.p. 60-90 ^{o}C) and ethyl acetate were purchased from Shanghai Titan Scientific Co. Ltd. Dichloromethane was dried over CaH_2 and freshly distilled before use; THF was used directly without further treatment. Other reagents were all commercially available and used as received without further purification. All the temperatures were referred to the oil baths used. TBS: *tert*-butyldimethylsilyl; TMS: trimethylsilyl; DMPS: dimethylphenylsilyl; TPS: triphenylsilyl; TBDPS: *tert*-butyldiphenylsilyl; TIPS: triisopropylsilyl.

Experimental details and analytical data

1. Synthesis of silyl ethers

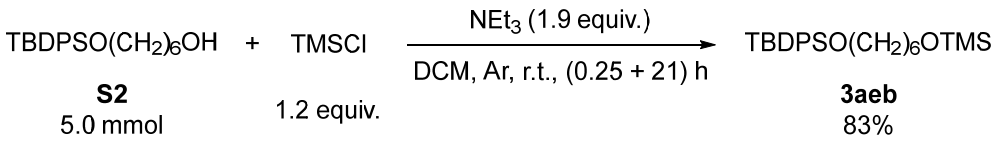
Silyl ethers **1aa**,[1,2] **1ab**,[1,3] **1ac**,[1,3] **1ad**,[1,4] **1ae**,[1,5] **1af**,[1,4] **1ba**,[1,6] **1ca**,[1,7] **1da**,[1,8] **1ea**,[1,9] **1fa**,[1,10] **1ha**,[1,11] **1ia**,[1,7] **1ja**,[1,12] **1ka**,[1,13] **1la**,[1,7] **3aea**,[1,7] **S2**,[1,14] **S3**,[1,7] and **S4**[1,15] were prepared according to the reported procedures.

1.1 2-(Hexyloxy)-1-(*tert*-butyldimethylsilyloxy)ethane **1ga** (lzz-3-064)



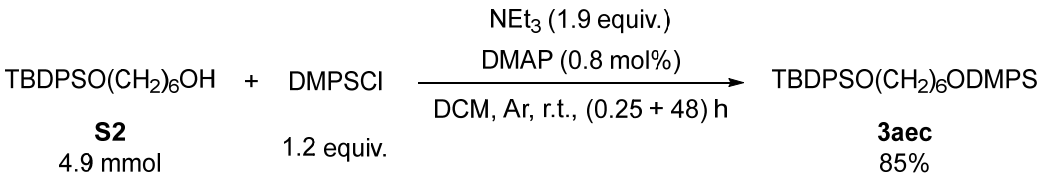
Typical Procedure I: A dried Schlenk tube was degassed and refilled with argon for three times. To this Schlenk tube were added TBSCl (1752.3 mg, 11.4 mmol, 98% purity), DMAP (9.5 mg, 0.08 mmol), anhydrous DCM (20 mL), and NEt_3 (2.6 mL, 0.728 g/mL, 1.8928 g, 18.5 mmol, 99% purity) sequentially. The resulting mixture was stirred at room temperature and alcohol **S1** (1484.8 mg, 10 mmol, 98% purity) was added dropwise over 15 min and then stirred for 6 h as monitored by TLC until the complete consumption of **S1**. After removing the solid by filtration, the residue was eluted with DCM (5 mL). The filtrate was washed with a saturated solution of $NaHCO_3$ (aq., 10 mL) and H_2O (10 mL) sequentially before drying over anhydrous Na_2SO_4. After filtration, the filtrate was concentrated and the residue was purified by column chromatography on silica gel [eluent: petroleum ether (60-90 °C)/ethyl acetate = 50:1 (410 mL)] to afford **1ga** (1845.7 mg, 70%, purity = 98%) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.76 (t, *J* = 5.4 Hz, 2 H, CH_2), 3.53-3.41 (m, 4 H, 2 × CH_2), 1.62-1.50 (m, 2 H, CH_2), 1.39-1.21 (m, 6 H, 3 × CH_2), 0.95-0.83 (m, 12 H, 4 × CH_3), 0.07 (s, 6 H, 2 × $SiCH_3$); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 72.1, 71.5, 62.8, 31.7, 29.7, 25.9, 25.8, 22.6, 18.4, 14.0, -5.3; **MS** (FI) *m/z* 261 $(M+H)^+$, 203 $(M-^tBu)^+$; **IR** (neat): ν = 2954, 2929, 2857, 1465, 1385, 1360, 1294, 1252, 1103 cm^{-1}; **HRMS** (FI) calcd. for $C_{14}H_{33}O_2Si$ $(M+H)^+$: 261.2244, Found: 261.2241.

1.2 1-(Trimethylsilyloxy)-6-(*tert*-butyldiphenylsilyloxy)hexane **3aeb** (lzz-2-049)



A dried Schlenk tube was degassed and refilled with argon for three times. To this Schlenk tube were added TMSCl (696.4 mg, 6.3 mmol, 98% purity), anhydrous DCM (10 mL), and NEt_3 (1.3 mL, 0.728 g/mL, 0.9464 g, 9.3 mmol) sequentially. The resulting solution was stirred at room temperature and alcohol **S2**[4] (1767.7 mg, 5.0 mmol) was added dropwise over 15 min. The resulting mixture was stirred for 21 h at room temperature, quenched with H_2O (20 mL), and extracted with DCM (10 mL × 3). The combined organic layer was dried over anhydrous Na_2SO_4. After filtration, the filtrate was concentrated and the residue was purified by column chromatography on silica gel [eluent: petroleum ether (60-90 °C, 100 mL) to petroleum ether/ethyl acetate = 40:1 (200 mL) to 20:1 (210 mL)] to afford **3aeb** (1762.6 mg, 83%) as a colorless liquid: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.71-7.62 (m, 4 H, ArH), 7.45-7.32 (m, 6 H, ArH), 3.65 (t, *J* = 6.6 Hz, 2 H, OCH_2), 3.55 (t, *J* = 6.8 Hz, 2 H, OCH_2), 1.62-1.47 (m, 4 H, 2 × CH_2), 1.42-1.24 (m, 4 H, 2 × CH_2), 1.04 (s, 9 H, 3 × CH_3), 0.11 (s, 9 H, 3 × $SiCH_3$); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 135.5, 134.1, 129.5, 127.5, 63.9, 62.6, 32.7, 32.5, 26.8, 25.61, 25.57, 19.2, -0.5; **MS** (DART) *m/z* 446 $(M+NH_4)^+$; **IR** (neat): ν = 3071, 2931, 2900, 2858, 1472, 1427, 1389, 1362, 1250, 1188, 1088, 1009 cm^{-1}; **HRMS** (DART) calcd. for $C_{25}H_{41}O_2Si_2$ $(M+H)^+$: 429.2640, Found: 429.2638.

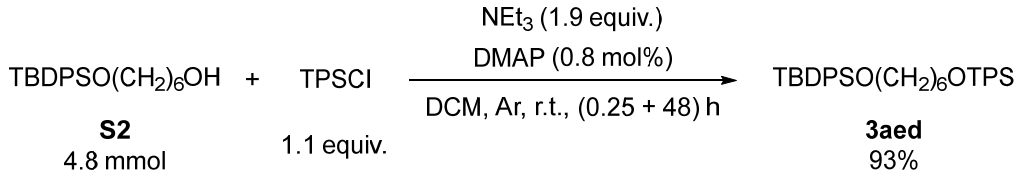
1.3 1-(Dimethylphenylsilyloxy)-6-(*tert*-butyldiphenylsilyloxy)hexane **3aec** (lzz-2-024)



Following **Typical Procedure I**, the reaction of DMPSCl (1036.4 mg, 5.9 mmol, 97% purity), DMAP (5.4 mg, 0.04 mmol), anhydrous DCM (10 mL), NEt_3 (1.3 mL, 0.728 g/mL, 0.9464 g, 9.3 mmol), and alcohol **S2**[4] (1762.4 mg, 4.9 mmol) was stirred

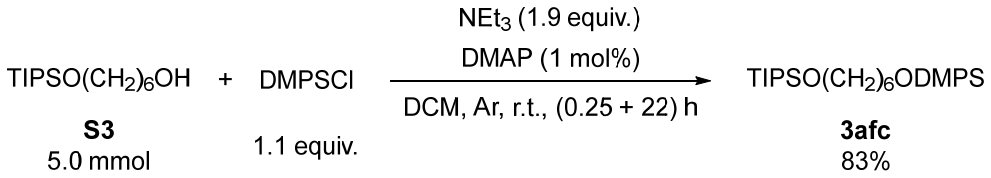
for 48 h and afforded **3aec** (2052.0 mg, 85%) [eluent: petroleum ether (60-90 °C, 100 mL) to petroleum ether/ethyl acetate = 20:1 (210 mL)] as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.70-7.63 (m, 4 H, ArH), 7.60-7.54 (m, 2 H, ArH), 7.43-7.31 (m, 9 H, ArH), 3.63 (t, *J* = 6.4 Hz, 2 H, OCH_2), 3.57 (t, *J* = 6.6 Hz, 2 H, OCH_2), 1.59-1.46 (m, 4 H, 2 × CH_2), 1.37-1.22 (m, 4 H, 2 × CH_2), 1.04 (s, 9 H, 3 × CH_3), 0.37 (s, 6 H, 2 × $SiCH_3$); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 138.0, 135.5, 134.1, 133.4, 129.50, 129.46, 127.8, 127.5, 63.9, 63.0, 32.6, 32.5, 26.8, 25.54, 25.50, 19.2, -1.8; **MS** (DART) *m/z* 508 $(M+NH_4)^+$; **IR** (neat): ν = 3068, 3047, 2933, 2856, 1471, 1427, 1388, 1360, 1303, 1251, 1187, 1109, 1089, 1029 cm^{-1}; **HRMS** (DART) calcd. for $C_{30}H_{43}O_2Si_2$ $(M+H)^+$: 491.2796, Found: 491.2797.

1.4 1-(*tert*-Butyldiphenylsilyloxy)-6-(triphenylsilyloxy)hexane **3aed** (lzz-2-025)



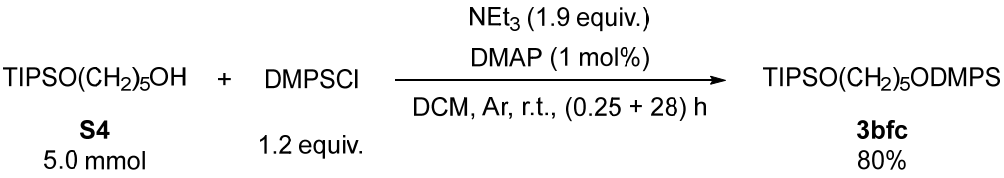
Following **Typical Procedure I**, the reaction of TPSCl (1660.2 mg, 5.5 mmol, 98% purity), DMAP (5.1 mg, 0.04 mmol), anhydrous DCM (10 mL), NEt_3 (1.3 mL, 0.728 g/mL, 0.9464 g, 9.3 mmol), and alcohol **S2**[4] (1725.7 mg, 4.8 mmol) was stirred for 48 h and afforded **3aed** (3119.0 mg, 93%, purity 93%) [eluent: petroleum ether (60-90 °C, 200 mL) to petroleum ether/ethyl acetate = 20:1 (310 mL)] as a white solid: **m.p.** 75.6-76.6 °C (ether); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.68-7.59 (m, 10 H, ArH), 7.44-7.33 (m, 15 H, ArH), 3.77 (t, *J* = 6.4 Hz, 2 H, OCH_2), 3.61 (t, *J* = 6.4 Hz, 2 H, OCH_2), 1.61-1.47 (m, 4 H, 2 × CH_2), 1.34-1.26 (m, 4 H, 2 × CH_2), 1.06-1.00 (s, 9 H, 3 × CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 135.5, 135.4, 134.4, 134.1, 129.9, 129.5, 127.8, 127.5, 63.87, 63.85, 32.5, 26.8, 25.5, 19.2; **MS** (DART) *m/z* 632 $(M+NH_4)^+$; **IR** (neat): ν = 3067, 3046, 2934, 2855, 1464, 1427, 1358, 1305, 1113, 1079, 1023 cm^{-1}; **Anal.** calcd. for $C_{40}H_{46}O_2Si_2$: C 78.12, H 7.54, Found: C 77.81, H 7.47.

1.5 1-(Triisopropylsilyloxy)-6-(dimethylphenylsilyloxy)hexane **3afc** (lzz-2-016)



Following **Typical Procedure I**, the reaction of DMPSCl (967.6 mg, 5.5 mmol, 97% purity), DMAP (6.1 mg, 0.05 mmol), anhydrous DCM (10 mL), NEt_3 (1.3 mL, 0.728 g/mL, 0.9464 g, 9.3 mmol), and alcohol **S3**[4] (1374.1 mg, 5.0 mmol) was stirred for 22 h and afforded **3afc** (1708.6 mg, 83%) [eluent: petroleum ether (60-90 °C, 100 mL) to petroleum ether/ethyl acetate = 20:1 (300 mL)] as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.61-7.54 (m, 2 H, ArH), 7.42-7.34 (m, 3 H, ArH), 3.68-3.53 (m, 4 H, 2 × OCH_2), 1.58-1.47 (m, 4 H, 2 × CH_2), 1.36-1.28 (m, 4 H, 2 × CH_2), 1.10-1.00 (m, 21 H, 3 × SiCH and 6 × CH_3), 0.37 (s, 6 H, 2 × $SiCH_3$); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 138.0, 133.4, 129.5, 127.8, 63.4, 63.1, 33.0, 32.6, 25.6, 18.0, 12.0, -1.8; **MS** (DART) *m/z* 426 $(M+NH_4)^+$; **IR** (neat): ν = 2938, 2864, 1463, 1428, 1385, 1251, 1091, 1013 cm^{-1}; **HRMS** (DART) calcd. for $C_{23}H_{45}O_2Si_2$ $(M+H)^+$: 409.2953, Found: 409.2951.

1.6 1-(Triisopropylsilyloxy)-5-(dimethylphenylsilyloxy)pentane **3bfc** (lzz-2-019)



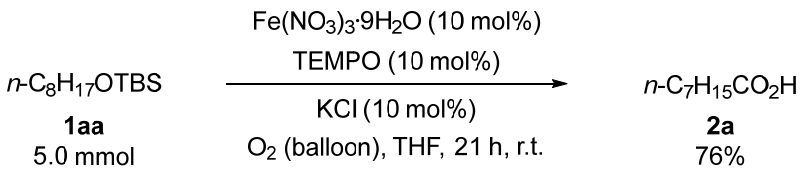
Following **Typical Procedure I**, the reaction of DMPSCl (1030.2 mg, 5.9 mmol, 97% purity), DMAP (5.7 mg, 0.05 mmol), anhydrous DCM (10 mL), NEt_3 (1.3 mL, 0.728 g/mL, 0.9464 g, 9.3 mmol), and alcohol **S4**[6] (1306.2 mg, 5.0 mmol) was stirred for 28 h and afforded **3bfc** (1582.3 mg, 80%) [eluent: petroleum ether (60-90 °C, 200 mL) to petroleum ether/ethyl acetate = 40:1 (200 mL) to 20:1 (210 mL)] as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.60-7.54 (m, 2 H, ArH), 7.42-7.34 (m, 3 H, ArH), 3.65 (t, *J* = 6.4 Hz, 2 H, OCH_2), 3.59 (t, *J* = 6.6 Hz, 2 H, OCH_2), 1.60-1.48 (m, 4 H, 2 × CH_2), 1.42-1.32 (m, 2 H, CH_2), 1.13-1.00 (m, 21 H, 3 × SiCH and 6 × CH_3), 0.37 (s, 6 H, 2 × $SiCH_3$); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 138.0, 133.4, 129.5, 127.8, 63.3, 63.1,

32.7, 32.4, 22.1, 18.0, 12.0, -1.8; **MS** (DART) *m/z* 395 $(M+H)^+$; **IR** (neat): ν = 3048, 2940, 2863, 1463, 1428, 1386, 1251, 1091 cm^{-1}; **HRMS** (DART) calcd. for $C_{22}H_{43}O_2Si_2$ $(M+H)^+$: 395.2796, Found: 395.2796.

2. Deprotective oxidation of silyl ethers to carboxylic acids with pure oxygen

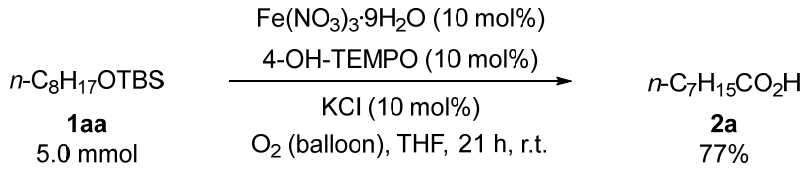
2.1 Preparation of octanoic acid **2a**

2.1.1 From **1aa** with TEMPO as co-catalyst (lzz-2-113)



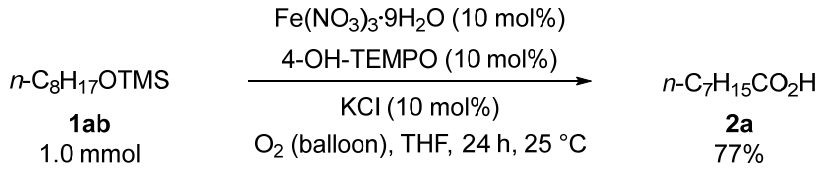
Typical Procedure II: To a Schlenk tube were added KCl (37.9 mg, 0.5 mmol), $Fe(NO_3)_3$•$9H_2O$ (205.8 mg, 0.5 mmol), TEMPO (79.2 mg, 0.5 mmol), **1aa** (1215.0 mg, 5.0 mmol), and THF (1085.6 mg, 14.9 mmol) sequentially. The atmosphere was replaced with using a water pump (until bubbles appeared in the mixture) for three times. The resulting mixture was stirred at room temperature for 21 h as monitored by TLC. After filtration through a short column of silica gel eluted with DCM/methanol = 10:1 (150 mL) and concentration, the residue was purified by column chromatography on silica gel [eluent: petroleum ether (60-90 °C)/ethyl acetate = 10:1 (220 mL) to 5:1 (480 mL)] to afford a crude product. To this crude product were added DCM (10 mL) and a saturated solution of $NaHCO_3$ (aq., 5 mL). After separation, the aqueous layer was treated with an aqueous solution of HCl (3 mol/L) for adjusting its *p*H to 1-2 and extracted with DCM (10 mL × 5). The combined organic layer was dried over anhydrous Na_2SO_4. After removing the solid by filtration, the filtrate was evaporated to afford **2a**[16] (545.3 mg, 76%) as a pale yellow liquid: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 10.71 (br, 1 H, CO_2H), 2.34 (t, *J* = 7.4 Hz, 2 H, CH_2), 1.68-1.58 (m, 2 H, CH_2), 1.39-1.23 (m, 8 H, 4 × CH_2), 0.92-0.84 (m, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 180.4, 34.1, 31.6, 29.0, 28.9, 24.7, 22.6, 14.0.

2.1.2 From **1aa** with 4-OH-TEMPO as co-catalyst (lzz-2-115)



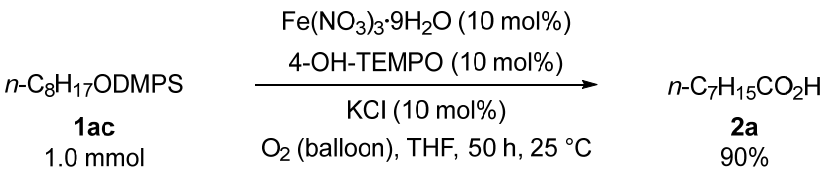
Following **Typical Procedure II**, the reaction of **1aa** (1226.0 mg, 5.0 mmol), KCl (37.3 mg, 0.5 mmol), $Fe(NO_3)_3$•$9H_2O$ (207.1 mg, 0.5 mmol), and 4-OH-TEMPO (87.8 mg, 0.1 mmol) in THF (1086.2 mg, 14.9 mmol) was stirred at room temperature for 21 h to afford **2a** (557.4 mg, 77%) as a pale yellow liquid.

2.1.3 From **1ab** with 4-OH-TEMPO as co-catalyst (lzz-1-47)



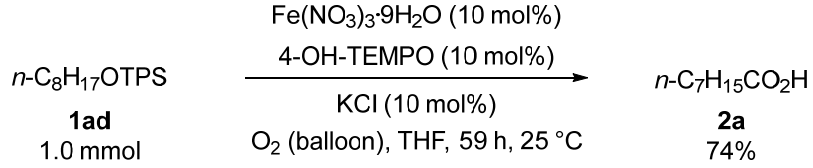
Typical Procedure III: To a Schlenk tube were added KCl (7.8 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (42.0 mg, 0.1 mmol), 4-OH-TEMPO (16.6 mg, 0.1 mmol), **1ab** (218.4 mg, 1.0 mmol), and THF (1.0 mL) sequentially. The atmosphere was reduced using a water pump (until bubbles appeared in the mixture) and refilled with pure oxygen for three times. The resulting mixture was stirred at 25 ºC for 24 h as monitored by TLC. After removing the solid by filtration through a short column of silica gel eluted with DCM/methanol = 10:1 (150 mL) and concentration, the residue was diluted with DCM (10 mL) and basified with a saturated solution of $NaHCO_3$ (aq., 10 mL). The aqueous layer was acidified with an aqueous solution of HCl (3 mol/L) and extracted with DCM (10 mL × 5). The combined organic layer was dried over anhydrous Na_2SO_4. After removing the solid by filtration, the filtrate was evaporated to afford **2a** (117.6 mg, 77%) as a pale yellow liquid.

2.1.4 From **1ac** with 4-OH-TEMPO as co-catalyst (lzz-1-41)



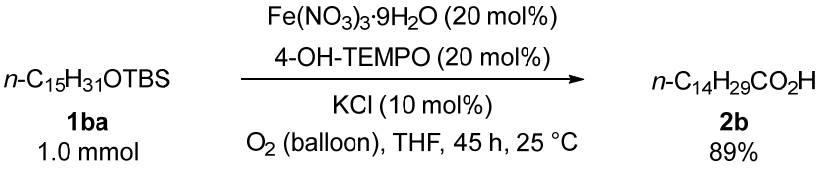
Following **Typical Procedure III**, the reaction of **1ac** (259.9 mg, 1.0 mmol), KCl (7.3 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (40.8 mg, 0.1 mmol), and 4-OH-TEMPO (18.1 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 50 h to afford **2a** (127.0 mg, 90%) as a pale yellow liquid.

2.1.5 From **1ad** with 4-OH-TEMPO as co-catalyst (lzz-1-49)



Following **Typical Procedure III**, the reaction of **1ad** (374.8 mg, 1.0 mmol), KCl (7.7 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (41.2 mg, 0.1 mmol), and 4-OH-TEMPO (17.1 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 59 h to afford **2a** (102.9 mg, 74%) as a pale yellow liquid.

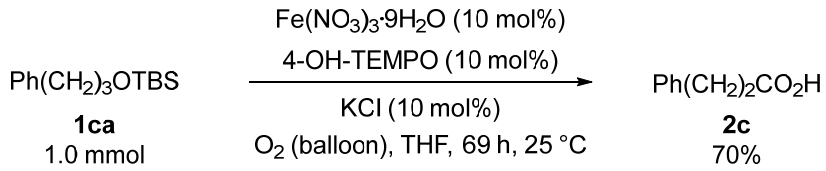
2.2 Preparation of pentadecanoic acid **2b** (lzz-2-084)



Typical Procedure IV: To a Schlenk tube were added KCl (8.0 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (81.8 mg, 0.2 mmol), 4-OH-TEMPO (35.4 mg, 0.2 mmol), **1ba** (340.6 mg, 1.0 mmol), and THF (1.0 mL) sequentially. The atmosphere was replaced with pure oxygen for three times using a water pump (until bubbles appeared in the mixture). The resulting mixture was stirred at 25 °C for 45 h as monitored by TLC. After removing the solid by filtration through a short column of silica gel eluted with DCM/methanol = 10:1 (150 mL) and concentration, the residue was purified by column chromatography on silica gel [eluent: petroleum ether (60-90 °C)/ethyl acetate = 50:1 (200 mL) to 30:1 (180 mL) to 10:1 (220 mL) to 5:1 (180 mL)] to afford **2b**[17] (213.4 mg, 89%) as a pale yellow solid: **m.p.** 52.2-52.9 °C (*n*-hexane) (reported:[8] 51-53 °C); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.35 (t, *J* = 7.4 Hz, 2 H, CH_2), 1.69-1.58 (m, 2 H, CH_2), 1.38-1.18 (m, 22 H, 11 × CH_2), 0.93-0.83 (m, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$)

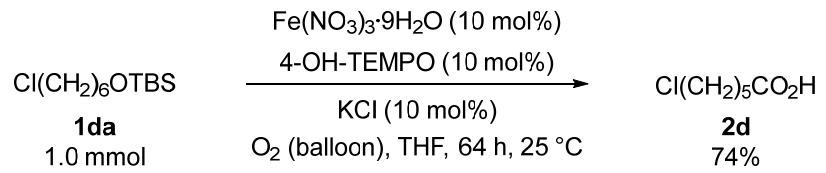
δ 180.6, 34.1, 31.9, 29.70, 29.68, 29.66, 29.60, 29.44, 29.37, 29.2, 29.1, 24.7, 22.7, 14.1; **MS** (ESI, Negative) *m/z* 241 $(M-H)^-$; **IR** (neat): ν = 3400-2600, 1693, 1469, 1429, 1411, 1316, 1297, 1276, 1253, 1229, 1206, 1186 cm^{-1}.

2.3 Preparation of 3-phenylpropanoic acid **2c** (lzz-4-011)



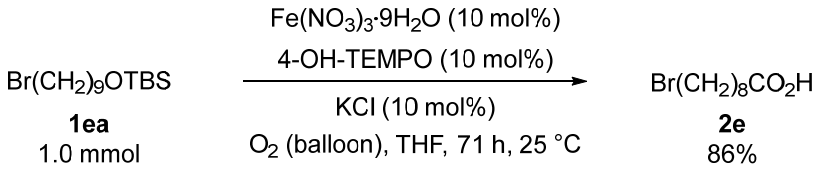
Following **Typical Procedure II**, the reaction of **1ca** (249.0 mg, 1.0 mmol), KCl (7.9 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (41.3 mg, 0.1 mmol), and 4-OH-TEMPO (17.4 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 69 h to afford **2c**[16] (106.1 mg, 70%) [eluent: petroleum ether (60-90 °C)/ethyl acetate = 5:1 (360 mL) to 2:1 (300 mL)] as a white solid: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33-7.26 (m, 2 H, ArH), 7.24-7.18 (m, 3 H, ArH), 2.96 (t, *J* = 7.8 Hz, 2 H, CH_2), 2.69 (t, *J* = 7.8 Hz, 2 H, CH_2); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 179.6, 140.1, 128.5, 128.2, 126.3, 35.6, 30.5.

2.4 Preparation of 6-chlorohexanoic acid **2d** (lzz-1-68)



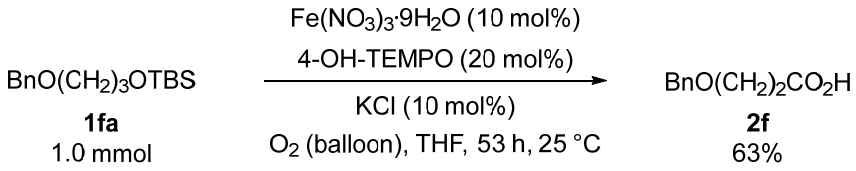
Following **Typical Procedure III**, the reaction of **1da** (265.2 mg, 1.0 mmol), KCl (7.0 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (41.5 mg, 0.1 mmol), and 4-OH-TEMPO (17.8 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 64 h to afford **2d**[18] (118.4 mg, 74%) as a pale yellow liquid: **^{1}H NMR** (300 MHz, $CDCl_3$) δ 10.48 (br, 1 H, CO_2H), 3.54 (t, *J* = 6.6 Hz, 2 H, CH_2), 2.39 (t, *J* = 7.4 Hz, 2 H, CH_2), 1.86-1.74 (m, 2 H, CH_2), 1.74-1.61 (m, 2 H, CH_2), 1.57-1.42 (m, 2 H, CH_2); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 179.9, 44.7, 33.8, 32.2, 26.3, 23.9; **MS** (ESI, Negative) *m/z* 151 $[M(^{37}Cl)-H]^-$, 149 $[M(^{35}Cl)-H]^-$; **IR** (neat): ν = 3400-2500, 1704, 1413, 1277, 1218, 1130 cm^{-1}.

2.5 Preparation of 9-bromononanoic acid **2e** (lzz-6-061)



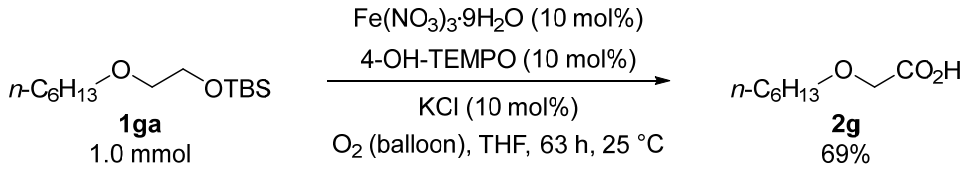
Following **Typical Procedure IV**, the reaction of **1ea** (333.3 mg, 1.0 mmol), KCl (8.0 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (41.5 mg, 0.1 mmol), and 4-OH-TEMPO (17.5 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 71 h to afford **2e**[16] (202.6 mg, 86%) [eluent: petroleum ether (60-90 °C)/ethyl acetate = 5:1 (240 mL) to 2:1 (600 mL)] as a yellow solid: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.41 (t, *J* = 6.8 Hz, 2 H, CH_2), 2.36 (t, *J* = 7.6 Hz, 2 H, CH_2), 1.90-1.80 (m, 2 H, CH_2), 1.69-1.58 (m, 2 H, CH_2), 1.48-1.28 (m, 8 H, 4 × CH_2); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 180.5, 33.9, 33.8, 32.6, 28.9, 28.7, 28.4, 27.9, 24.4.

2.6 Preparation of 3-benzyloxypropionic acid **2f** (lzz-2-061)



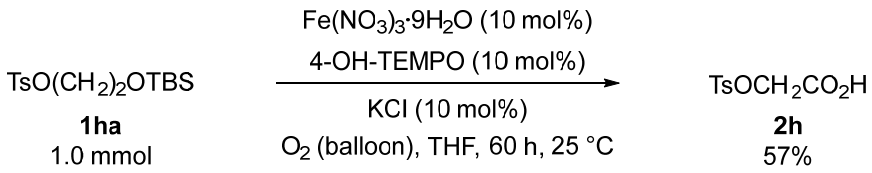
Following **Typical Procedure II**, the reaction of **1fa** (279.0 mg, 1.0 mmol), KCl (7.5 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (40.9 mg, 0.1 mmol), and 4-OH-TEMPO (35.6 mg, 0.2 mmol) in THF (1.0 mL) was stirred at 25 °C for 53 h to afford **2f**[19] (113.6 mg, 63%) [eluent: petroleum ether (60-90 °C)/ethyl acetate = 1:1 (220 mL) to 5:1 (240 mL) to 2:1 (210 mL) to 1:1 (200 mL)] as a pale yellow oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 10.75 (br, 1 H, CO_2H), 7.36-7.23 (m, 5 H, ArH), 4.53 (s, 2 H, CH_2), 3.73 (t, *J* = 6.2 Hz, 2 H, CH_2), 2.64 (t, *J* = 6.2 Hz, 2 H, CH_2); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 177.6, 137.7, 128.3, 127.65, 127.63, 73.0, 65.1, 34.8; **MS** (EI) *m/z* (%) 180 (M^+, 6.52), 107 (100); **IR** (neat): ν = 3400-2400, 1709, 1495, 1452, 1423, 1363, 1236, 1188, 1100, 1067, 1028 cm^{-1}.

2.7 Preparation of 2-(hexyloxy)acetic acid **2g** (lzz-6-055)



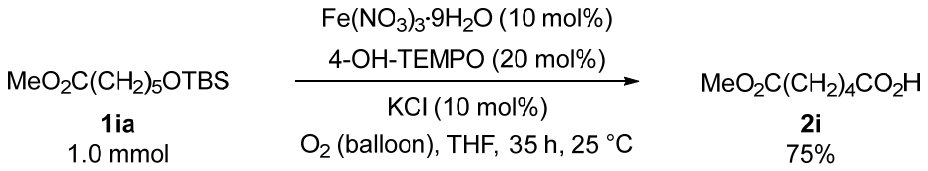
Following **Typical Procedure II**, the reaction of **1ga** (262.0 mg, 1.0 mmol), KCl (8.0 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (41.7 mg, 0.1 mmol), and 4-OH-TEMPO (17.7 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 63 h to afford **2g**[16] (109.9 mg, 69%) as a pale yellow oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 10.88 (br, 1 H, CO_2H), 4.12 (s, 2 H, CH_2), 3.55 (t, *J* = 6.8 Hz, 2 H, CH_2), 1.69-1.55 (m, 2 H, CH_2), 1.41-1.22 (m, 6 H, 3 × CH_2), 0.95-0.82 (m, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 175.7, 72.1, 67.7, 31.5, 29.3, 25.5, 22.5, 13.9.

2.8 Preparation of 2-(tosyloxy)acetic acid **2h** (lzz-6-056)



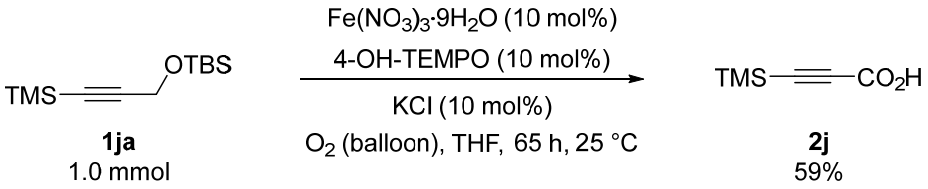
Following **Typical Procedure II**, the reaction of **1ha** (330.5 mg, 1.0 mmol), KCl (7.4 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (40.5 mg, 0.1 mmol), and 4-OH-TEMPO (17.4 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 60 h to afford **2h**[20] (135.3 mg, 57%, purity = 97%) [eluent: dichloromethane (200 mL) to dichloromethane/methanol = 50:1 (400 mL)] as a white solid: **m.p.** 133.4-134.4 °C (EA/*n*-hexane) (reported:[11] 136-138 °C); **^{1}H NMR** (400 MHz, CD_3OD) δ 7.81 (d, *J* = 8.4 Hz, 2 H, ArH), 7.43 (d, *J* = 8.0 Hz, 2 H, ArH), 4.59 (s, 2 H, CH_2), 2.44 (s, 3 H, CH_3); **^{13}C NMR** (100 MHz, CD_3OD) δ 169.4, 146.8, 133.9, 131.0, 129.0, 65.9, 21.6; **MS** (ESI) *m/z* 253 $(M+Na)^+$; **IR** (neat): ν = 3300-2500, 1710, 1596, 1495, 1442, 1413, 1357, 1293, 1253, 1170, 1096, 1053, 1034 cm^{-1}.

2.9 Preparation of 1,6-hexanedioic acid monomethyl ester **2i** (lzz-6-060)



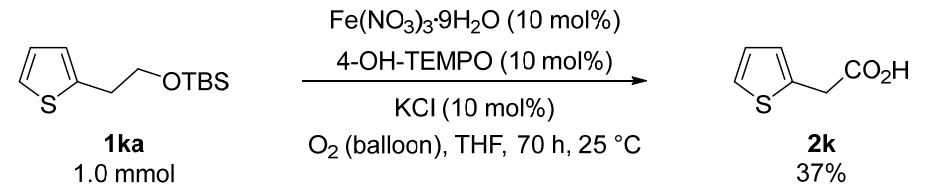
Following **Typical Procedure II**, the reaction of **1ia** (260.9 mg, 1.0 mmol), KCl (7.9 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (41.6 mg, 0.1 mmol), and 4-OH-TEMPO (35.5 mg, 0.2 mmol) in THF (1.0 mL) was stirred at 25 °C for 35 h to afford **2i**[16] (119.6 mg, 75%) [eluent: petroleum ether (60-90 °C)/ethyl acetate = 5:1 (240 mL) to 2:1 (600 mL)] as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.68 (s, 3 H, CH_3), 2.43-2.30 (m, 4 H, 2 × CH_2), 1.74-1.62 (m, 4 H, 2 × CH_2); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 179.4, 173.8, 51.5, 33.5, 24.1, 23.9.

2.10 Preparation of 3-(trimethylsilyl)propiolic acid **2j** (lzz-2-064)



Following **Typical Procedure IV**, the reaction of **1ja** (235.6 mg, 1.0 mmol), KCl (7.9 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (41.9 mg, 0.1 mmol), and 4-OH-TEMPO (17.7 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 65 h to afford **2j**[16] (81.5 mg, 59%) [eluent: petroleum ether (60-90 °C)/ethyl acetate = 5:1 (240 mL) to dichloromethane/methanol = 10:1 (110 mL)] as a pale yellow oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.92 (br, 1 H, CO_2H), 0.26 (s, 9 H, 3 × $SiCH_3$); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 157.0, 96.7, 94.0, -1.0.

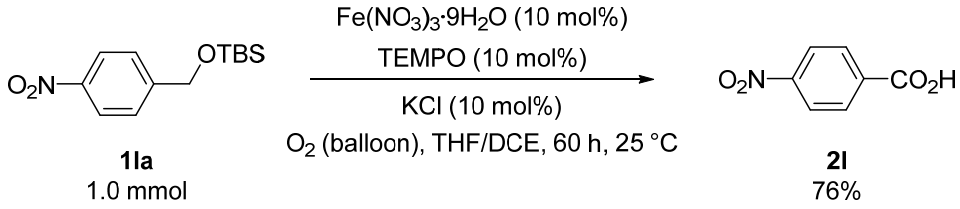
2.11 Preparation of 2-thienylacetic acid **2k** (lzz-2-075)



Following **Typical Procedure II**, the reaction of **1ka** (237.1 mg, 1.0 mmol), KCl

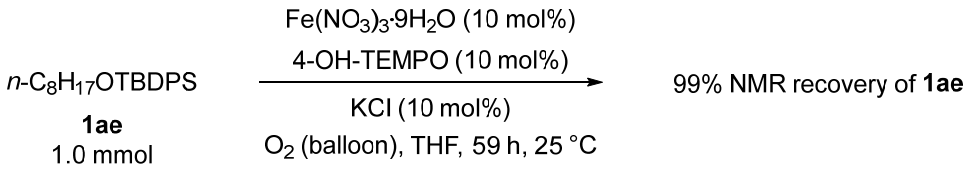
(7.8 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (41.2 mg, 0.1 mmol), and 4-OH-TEMPO (17.5 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 70 h to afford **2k**[16] (51.0 mg, 37%) [eluent: petroleum ether (60-90 °C)/ethyl acetate = 5:1 (240 mL) to 2:1 (210 mL) to 1:1 (200 mL)] as a yellow solid: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 11.19 (br, 1 H, CO_2H), 7.26-7.21 (m, 1 H, ArH), 7.00-6.93 (m, 2 H, ArH), 3.88 (s, 2 H, CH_2); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 177.0, 134.0, 127.2, 126.9, 125.3, 35.0.

2.12 Preparation of 4-nitrobenzoic acid **2l** (lzz-2-103)



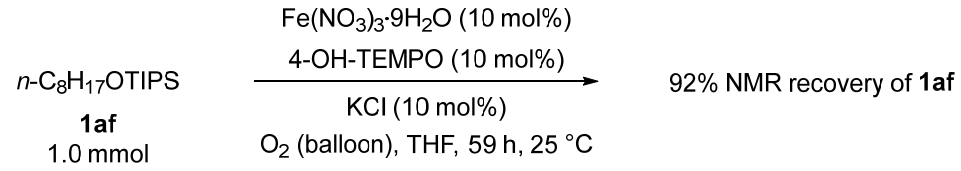
Following **Typical Procedure III**, the reaction of **1la** (262.2 mg, 1.0 mmol), KCl (8.0 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (40.7 mg, 0.1 mmol), TEMPO (15.8 mg, 0.1 mmol), THF (0.5 mL), and DCE (0.5 mL) was stirred at 25 °C for 60 h to afford **2l**[16] (127.8 mg, 76%, purity = 97%) as a white solid: **^{1}H NMR** (400 MHz, DMSO-d_6) δ 13.63 (br, 1 H, CO_2H), 8.32 (d, J = 9.2 Hz, 2 H, ArH), 8.17 (d, J = 9.2 Hz, 2 H, ArH); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 165.8, 150.0, 136.4, 130.7, 123.7.

2.13 The reaction of **1ae** (lzz-1-48)



Following **Typical Procedure IV**, the reaction of **1ae** (362.5 mg, 1.0 mmol), KCl (7.7 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (40.5 mg, 0.1 mmol), and 4-OH-TEMPO (17.5 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 59 h and failed to afford **2a** with 99% of **1ae** being recovered as determined by the ^{1}H NMR analysis with CH_2Br_2 as the internal standard.

2.14 The reaction of **1af** (lzz-3-128)

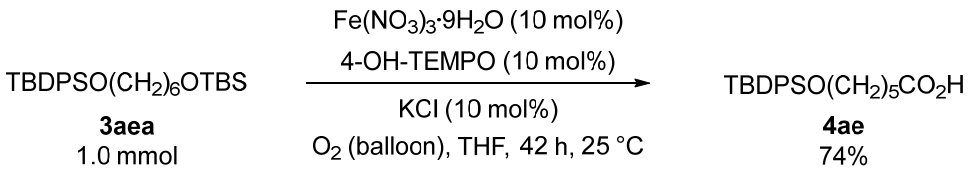


Following **Typical Procedure IV**, the reaction of **1af** (287.9 mg, 1.0 mmol), KCl (7.7 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (41.2 mg, 0.1 mmol), and 4-OH-TEMPO (17.8 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 59 h and failed to afford **2a** with 92% of **1af** being recovered as determined by the ^{1}H NMR analysis with CH_2Br_2 as the internal standard.

3. Selective deprotective oxidation of silyl ethers to carboxylic acids

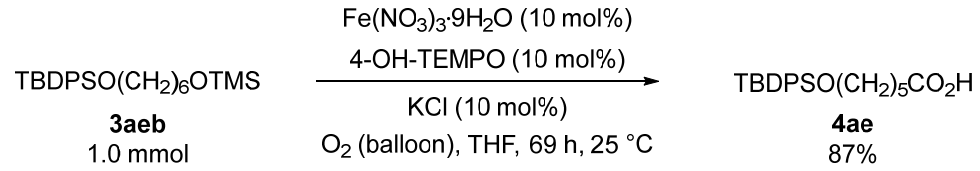
3.1 Preparation of 6-((1,1-dimethyethyl)diphenylsilyloxy)hexanoic acid **4ae**

3.1.1 From **3aea** (lzz-1-64)



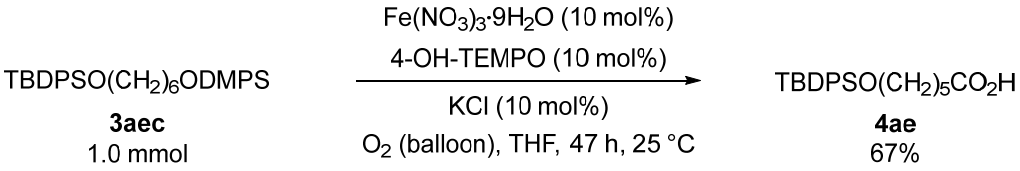
Following **Typical Procedure IV**, the reaction of **3aea** (441.4 mg, 1.0 mmol), KCl (7.3 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (42.8 mg, 0.1 mmol), and 4-OH-TEMPO (17.1 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 42 h to afford **4ae**[21] (255.5 mg, 74%) [eluent: petroleum ether (60-90 °C)/ethyl acetate = 10:1 (900 mL)] as a pale yellow oil: **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.70-7.61 (m, 4 H, ArH), 7.45-7.32 (m, 6 H, ArH), 3.70-3.61 (m, 2 H, CH_2), 2.33 (t, J = 7.5 Hz, 2 H, CH_2), 1.68-1.50 (m, 4 H, 2 × CH_2), 1.48-1.34 (m, 2 H, CH_2), 1.04 (s, 9 H, 3 × CH_3); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 179.6, 135.6, 134.1, 129.5, 127.6, 63.6, 33.9, 32.1, 26.8, 25.3, 24.4, 19.2; **MS** (ESI) *m/z* 393 $(M+Na)^+$; **IR** (neat): ν = 3500-2400, 1709, 1588, 1472, 1462, 1428, 1390, 1284, 1237, 1111 cm^{-1}.

3.1.2 From **3aeb** (lzz-2-055)



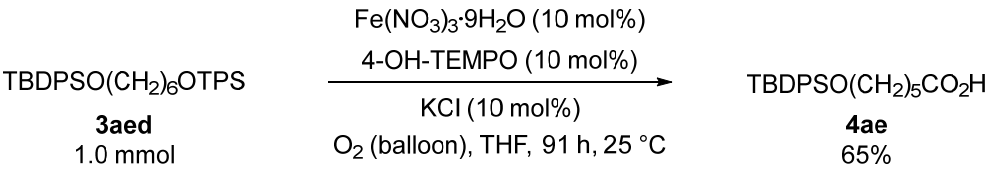
Following **Typical Procedure IV**, the reaction of **3aeb** (421.5 mg, 1.0 mmol), KCl (7.7 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (41.3 mg, 0.1 mmol), and 4-OH-TEMPO (17.9 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 69 h to afford **4ae** (316.3 mg, 87%) [eluent: petroleum ether (60-90 °C)/ethyl acetate = 10:1 (210 mL) to 5:1 (240 mL) to 2:1 (150 mL)] as a pale yellow oil.

3.1.3 From **3aec** (lzz-2-031)



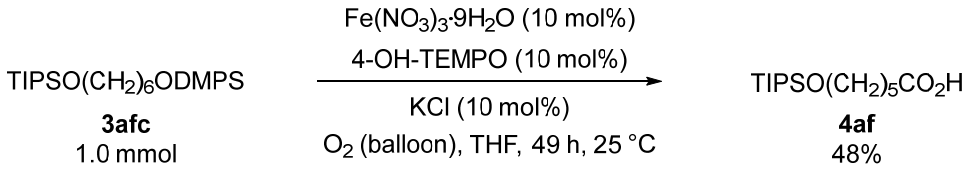
Following **Typical Procedure IV**, the reaction of **3aec** (484.0 mg, 1.0 mmol), KCl (8.0 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (42.2 mg, 0.1 mmol), and 4-OH-TEMPO (17.5 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 47 h to afford **4ae** (243.9 mg, 67%) [eluent: petroleum ether (60-90 °C, 200 mL) to petroleum ether/ethyl acetate = 10:1 (220 mL) to 5:1 (240 mL) to 2:1 (150 mL)] as a pale yellow oil.

3.1.4 From **3aed** (lzz-2-033)



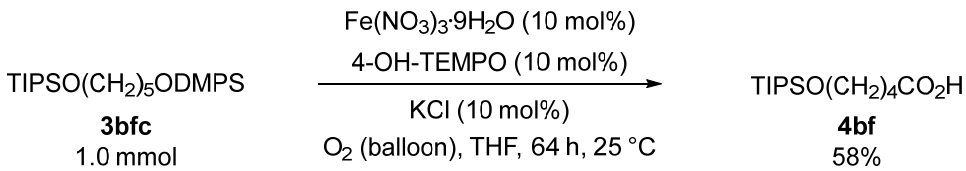
Following **Typical Procedure IV**, the reaction of **3aed** (606.2 mg, 1.0 mmol), KCl (7.9 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (41.9 mg, 0.1 mmol), and 4-OH-TEMPO (17.5 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 91 h to afford **4ae** (222.4 mg, 65%) [eluent: petroleum ether (60-90 °C, 200 mL) to petroleum ether/ethyl acetate = 10:1 (220 mL) to 5:1 (240 mL) to 2:1 (150 mL)] as a pale yellow oil.

3.2 Preparation of 6-(triisopropylsilyloxy)hexanoic acid **4af** (lzz-2-022)



Following **Typical Procedure IV**, the reaction of **3afc** (394.5 mg, 1.0 mmol), KCl (7.3 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (40.1 mg, 0.1 mmol), and 4-OH-TEMPO (17.7 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 49 h to afford **4af** (134.9 mg, 48%) [eluent: petroleum ether (60-90 °C, 200 mL) to petroleum ether/ethyl acetate = 10:1 (210 mL) to 5:1 (360 mL)] as a pale yellow oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 11.07 (br, 1 H, CO_2H), 3.68 (t, *J* = 6.4 Hz, 2 H, OCH_2), 2.37 (t, *J* = 7.4 Hz, 2 H, CH_2), 1.71-1.62 (m, 2 H, CH_2), 1.60-1.53 (m, 2 H, CH_2), 1.46-1.37 (m, 2 H, CH_2), 1.11-1.01 (m, 21 H, 3 × SiCH and 6 × CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 180.1, 63.1, 34.1, 32.5, 25.3, 24.5, 18.0, 11.9; **MS** (ESI) *m/z* 289 $(M+H)^+$; **IR** (neat): ν = 3718, 3029, 2941, 2864, 1709, 1462, 1386, 1365, 1285, 1239, 1103, 1012 cm^{-1}; **HRMS** (ESI) calcd. for $C_{15}H_{33}O_3Si$ $(M+H)^+$: 289.21935, Found: 289.21912.

3.3 Preparation of 5-(triisopropylsilyloxy)pentanoic acid **4bf** (lzz-2-027)



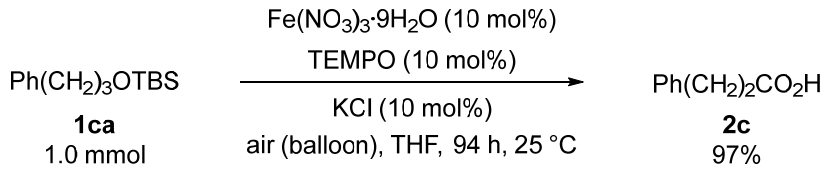
Following **Typical Procedure IV**, the reaction of **3bfc** (388.9 mg, 1.0 mmol), KCl (7.6 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (40.7 mg, 0.1 mmol), and 4-OH-TEMPO (17.6 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 64 h to afford **4bf** (155.8 mg, 58%) [eluent: petroleum ether (60-90 °C, 100 mL) to petroleum ether/ethyl acetate = 10:1 (220 mL) to 5:1 (240 mL)] as a pale yellow oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.71 (t, *J* = 6.0 Hz, 2 H, OCH_2), 2.40 (t, *J* = 7.2 Hz, 2 H, CH_2), 1.78-1.68 (m, 2 H, CH_2), 1.65-1.55 (m, 2 H, CH_2), 1.14-0.99 (m, 21 H, 3 × SiCH and 6 × CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 180.3, 62.8, 33.8, 32.1, 21.2, 18.0, 11.9; **MS** (ESI) *m/z* 275 $(M+H)^+$; **IR** (neat): ν = 2941, 2893, 2865, 1708, 1462, 1412, 1386, 1291, 1246, 1106, 1069, 1012

cm^{-1}; **HRMS** (ESI) calcd. for $C_{14}H_{31}O_3Si$ $(M+H)^+$: 275.20370, Found: 275.20401.

4. Deprotective oxidation of silyl ethers to carboxylic acids with air

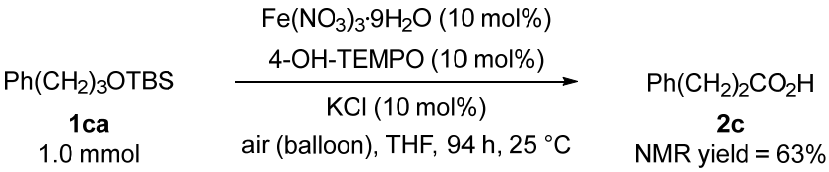
4.1 Preparation of 3-phenylpropanoic acid **2c**

4.1.1 With TEMPO as co-catalyst (lzz-3-192)



Typical Procedure V: To a Schlenk tube were added KCl (7.6 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (40.8 mg, 0.1 mmol), TEMPO (15.9 mg, 0.1 mmol), **1ca** (249.1 mg, 1.0 mmol), and THF (1.0 mL) sequentially. The resulting mixture was stirred at 25 °C for 94 h as monitored by TLC. After removing the solid by filtration through a short column of silica gel eluted with DCM/methanol = 10:1 (150 mL) and concentration, the residue was purified by column chromatography on silica gel [eluent: petroleum ether (60-90 °C)/ethyl acetate = 5:1 (360 mL) to 2:1 (300 mL)] to afford **2c**[16] (145.8 mg, 97%) as a white solid: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34-7.22 (m, 2 H, ArH), 7.25-7.18 (m, 3 H, ArH), 2.96 (t, *J* = 7.6 Hz, 2 H, CH_2), 2.69 (t, *J* = 7.8 Hz, 2 H, CH_2); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 179.6, 140.1, 128.5, 128.2, 126.3, 35.6, 30.5.

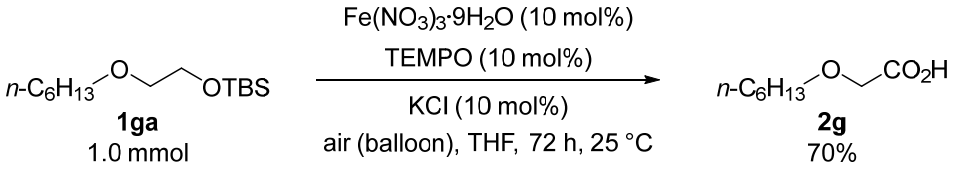
4.1.2 With 4-OH-TEMPO as co-catalyst (lzz-3-186)



Following **Typical Procedure V**, the reaction of **1ca** (245.4 mg, 1.0 mmol), KCl (7.6 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (41.5 mg, 0.1 mmol), and 4-OH-TEMPO (17.8 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 94 h to afford **2c** in 63% NMR yield as determined by the ^{1}H NMR analysis with CH_2Br_2 as the internal standard.

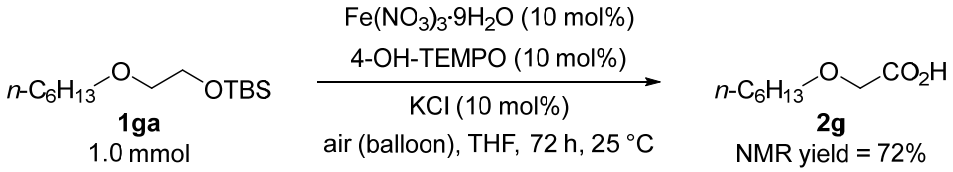
4.2 Preparation of 2-(hexyloxy)acetic acid **2g**

4.2.1 With TEMPO as co-catalyst (lzz-5-181)



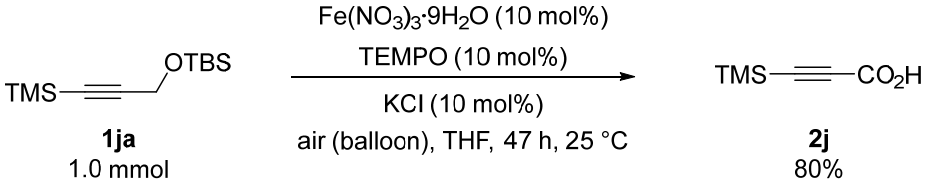
Following **Typical Procedure V**, the reaction of **1ga** (269.3 mg, 1.0 mmol), KCl (7.5 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (41.2 mg, 0.1 mmol), and TEMPO (16.3 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 72 h to afford **2g**[16] (119.8 mg, 70%, purity = 95%) [eluent: petroleum ether (60-90 °C)/ethyl acetate = 5:1 (240 mL) to 2:1 (300 mL)] as a yellow oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 10.51 (br, 1 H, CO_2H), 4.13 (s, 2 H, CH_2), 3.55 (t, *J* = 6.8 Hz, 2 H, CH_2), 1.70-1.55 (m, 2 H, CH_2), 1.42-1.21 (m, 6 H, 3 × CH_2), 0.95-0.81 (m, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 175.6, 72.0, 67.6, 31.4, 29.2, 25.4, 22.4, 13.8.

4.2.2 With 4-OH-TEMPO as co-catalyst (lzz-5-175)



Following **Typical Procedure V**, the reaction of **1ga** (263.1 mg, 1.0 mmol), KCl (7.3 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (42.0 mg, 0.1 mmol), and 4-OH-TEMPO (17.5 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 72 h to afford **2g** in 72% NMR yield as determined by the ^{1}H NMR analysis with CH_2Br_2 as the internal standard.

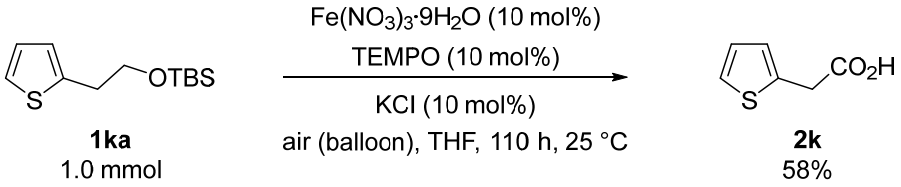
4.3 Preparation of 3-(trimethylsilyl)propiolic acid **2j** (lzz-4-010)



Following **Typical Procedure V**, the reaction of **1ja** (243.1 mg, 1.0 mmol), KCl (7.5 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (41.0 mg, 0.1 mmol), and TEMPO (15.9 mg, 0.1

mmol) in THF (1.0 mL) was stirred at 25 °C for 47 h to afford **2j**[16] (113.5 mg, 80%) [eluent: petroleum ether (60-90 °C)/ethyl acetate = 5:1 (240 mL) to 1:1 (200 mL)] as a pale yellow oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 11.45 (br, 1 H, CO_2H), 0.26 (s, 9 H, 3 × $SiCH_3$); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 157.8, 97.4, 93.7, -1.0.

4.4 Preparation of 2-thienylacetic acid **2k** (lzz-4-027)

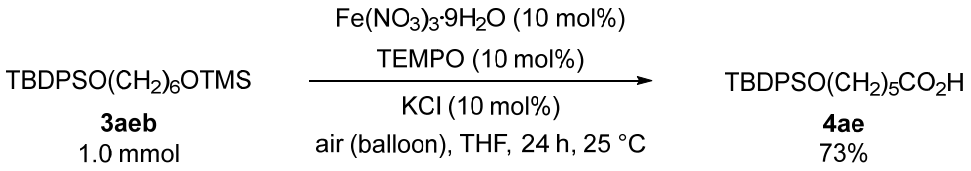


1ka
1.0 mmol

2k
58%

Following **Typical Procedure V**, the reaction of **1ka** (243.1 mg, 1.0 mmol), KCl (7.9 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (41.3 mg, 0.1 mmol), and TEMPO (15.9 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 110 h to afford **2k**[16] (82.5 mg, 58%) {eluent: petroleum ether [60-90 °C]/ethyl acetate = 5:1 [240 mL] to petroleum ether/ethyl acetate = 2:1 [300 mL, containing formic acid (1%, v/v)]} as a yellow solid: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.27-7.20 (m, 1 H, ArH), 7.00-6.93 (m, 2 H, ArH), 3.89 (s, 2 H, CH_2); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 177.1, 133.9, 127.2, 126.9, 125.3, 35.0.

4.5 Preparation of 6-((1,1-dimethyethyl)diphenylsilyloxy)hexanoic acid **4ae** (lzz-4-002)



3aeb
1.0 mmol

4ae
73%

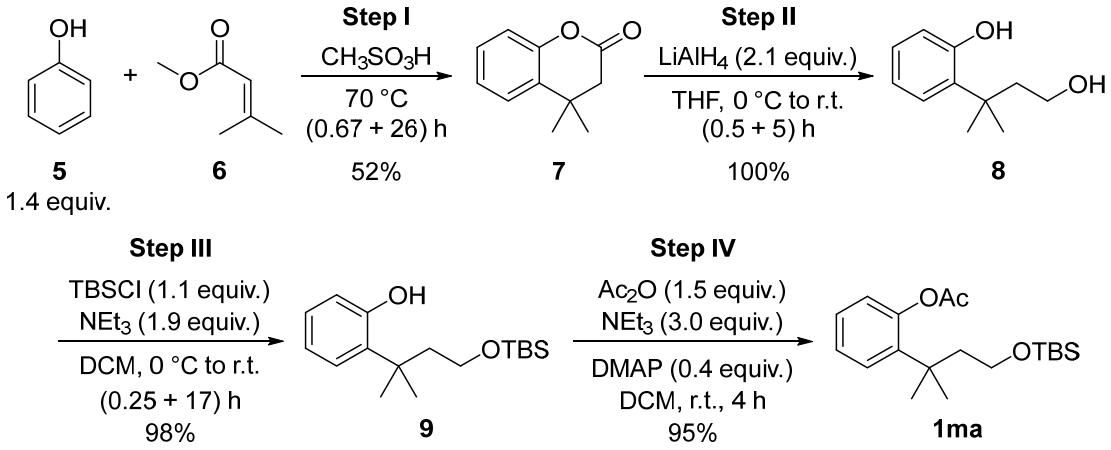
Following **Typical Procedure V**, the reaction of **3aeb** (425.3 mg, 1.0 mmol), KCl (7.9 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (41.4 mg, 0.1 mmol), and TEMPO (15.9 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 24 h to afford **4ae**[21] (275.5 mg, 73%, purity = 98%) [eluent: petroleum ether (60-90 °C)/ethyl acetate = 10:1 (220 mL) to 5:1 (240 mL) to 2:1 (150 mL)] as a pale yellow oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.71-7.62 (m, 4 H, ArH), 7.46-7.32 (m, 6 H, ArH), 3.66 (t, *J* = 6.4 Hz, 2 H, CH_2), 2.33 (t, *J* = 7.6 Hz, 2 H, CH_2), 1.68-1.51 (m, 4 H, 2 × CH_2), 1.47-1.36 (m, 2 H, CH_2), 1.04 (s, 9

H, 3 × CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 180.2, 135.4, 133.9, 129.4, 127.6, 63.5, 34.0, 32.0, 26.7, 25.2, 24.3, 19.1.

5. Formal syntheses of bioactive molecules 10 (from 2m) and 11 (from 2n)

5.1 Synthesis of 3-(2-acetoxyphenyl)-3-methylbutanoic acid **2m**

5.1.1 Synthesis of 2-(4-(*tert*-butyldimethylsilyloxy)-2-methylbutan-2-yl)phenyl acetate **1ma**[22,23] (lzz-1-75, lzz-1-77, lzz-2-110, lzz-2-117)



Step I: To a flask were added phenol **5** (2604.5 mg, 27.7 mmol). The atmosphere was reduced using a water pump and refilled with nitrogen for three times. Methanesulfonic acid (25 mL) was then added to the flask. The resulting solution was stirred at 70 °C and **6** (2371.4 mg, 20.8 mmol) was added dropwise over 40 min. The resulting solution was stirred at 70 °C for another 26 h, cooled to room temperature, poured into ice water, and extracted with ethyl acetate (60 mL × 3). The combined organic layer was washed with a saturated solution of $NaHCO_3$ (aq., 60 mL × 3) and a saturated solution of NaCl (aq., 60 mL) sequentially. The organic layer was dried over anhydrous Na_2SO_4. After filtration, the filtrate was concentrated and the residue was purified by column chromatography on silica gel [eluent: petroleum ether (60-90 °C, 200 mL) to petroleum ether/ethyl acetate = 50:1 (1500 mL) to 5:1 (300 mL)] to afford **7** (1894.8 mg, 52%) as a pale yellow oil: **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.36-7.21 (m, 2 H, ArH), 7.20-7.11 (m, 1 H, ArH), 7.09-7.03 (m, 1 H, ArH), 2.63 (s, 2 H, CH_2), 1.36 (s, 6 H, 2 × CH_3); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 168.2, 150.6, 131.7, 128.2, 124.7, 124.3, 117.0, 43.5, 33.2, 27.6.

Step II: A dried flask was degassed and refilled with nitrogen for three times. To this flask were added $LiAlH_4$ (857.2 mg, 22.6 mmol) and anhydrous THF (30 mL). A solution of **7** (1862.0 mg, 10.6 mmol) in THF (15 mL) was added dropwise to the flask in an ice-water bath over 30 min. The resulting mixture was stirred at room temperature for another 5 h and quenched with a saturated solution of NH_4Cl (aq., 20 mL). After filtration through a short column of silica gel eluted with ethyl acetate (50 mL), the filtrate was washed with a saturated solution of NaCl (aq., 50 mL × 2) and dried over anhydrous Na_2SO_4. After removing the solid by filtration, the filtrate was evaporated to afford crude **8** (1912.0 mg, 100%) as a pale yellow solid: **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.20 (d, *J* = 7.8 Hz, 1 H, ArH), 7.10-7.01 (m, 1 H, ArH), 6.89-6.80 (m, 1 H, ArH), 6.62 (d, *J* = 7.5 Hz, 1 H, ArH), 6.14-5.89 (m, 1 H, ArOH), 3.53 (t, *J* = 7.2 Hz, 2 H, CH_2), 2.22 (t, *J* = 7.0 Hz, 2 H, CH_2), 1.76-1.66 (m, 1 H, OH), 1.41 (s, 6 H, 2 × CH_3); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 154.8, 133.7, 127.5, 127.4, 120.1, 116.6, 61.0, 42.6, 36.6, 28.6.

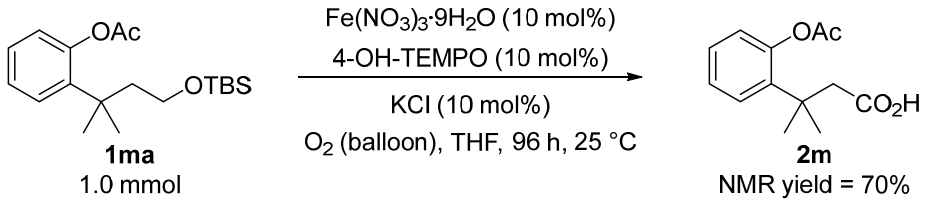
Step III: A dried flask was degassed and refilled with argon for three times. To this flask were added **8** (2052.5 mg, 11.4 mmol), anhydrous DCM (13 mL), and NEt_3 (3.0 mL, 0.728 g/mL, 2.1840 g, 21.6 mmol). The solution was stirred in an ice-water bath and a solution of TBSCl (1935.2 mg, 12.6 mmol) in anhydrous DCM (10 mL) was added dropwise over 15 min. The resulting mixture was stirred at room temperature for another 17 h. After filtration, the filtrate was washed with H_2O (20 mL × 2) and dried over anhydrous Na_2SO_4. After removing the solid by filtration, the filtrate was concentrated and the residue was purified by column chromatography on silica gel [eluent: petroleum ether (60-90 °C)/ethyl acetate = 10:1 (220 mL)] to afford **9** (3280.7 mg, 98%) as a pale yellow oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.19 (d, *J* = 7.6 Hz, 1 H, ArH), 7.06 (t, *J* = 7.6 Hz, 1 H, ArH), 6.85 (t, *J* = 7.6 Hz, 1 H, ArH), 6.68 (d, *J* = 7.6 Hz, 1 H, ArH), 5.60-5.52 (m, 1 H, ArOH), 3.49 (t, *J* = 7.2 Hz, 2 H, CH_2), 2.11 (t, *J* = 7.0 Hz, 2 H, CH_2), 1.40 (s, 6 H, 2 × CH_3), 0.85 (s, 9 H, 3 × CH_3), -0.02 (s, 6 H, 2 × $SiCH_3$); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 154.5, 134.3, 127.6, 127.2, 120.4, 117.0, 61.3, 43.4, 36.6, 28.9, 25.9, 18.3, -5.4.

Step IV: A dried Schlenk tube was degassed and refilled with argon for three times.

To this Schlenk tube were added **9** (1471.7 mg, 5.0 mmol)/anhydrous DCM (10 mL), Ac_2O (800.0 mg, 7.7 mmol), NEt_3 (2.1 mL, 0.728 g/mL, 1.5288 g, 15.1 mmol), and DMAP (240.8 mg, 2.0 mmol) sequentially. The solution was stirred at room temperature for 4 h, quenched with H_2O (10 mL), and extracted with ethyl acetate (50 mL × 2). The combined organic layer was dried over anhydrous Na_2SO_4. After removing the solid by filtration, the filtrate was concentrated and the residue was purified by column chromatography on silica gel [eluent: petroleum ether (60-90 °C, 100 mL)/ethyl acetate = 20:1 (210 mL)] to afford **1ma** (1602.6 mg, 95%) as a colorless liquid: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33 (d, *J* = 8.0 Hz, 1 H, ArH), 7.25-7.18 (m, 1 H, ArH), 7.18-7.11 (m, 1 H, ArH), 6.99 (d, *J* = 8.0 Hz, 1 H, ArH), 3.40 (t, *J* = 7.4 Hz, 2 H, CH_2), 2.32 (s, 3 H, CH_3), 2.00 (t, *J* = 7.2 Hz, 2 H, CH_2), 1.36 (s, 6 H, 2 × CH_3), 0.84 (s, 9 H, 3 × CH_3), -0.05 (s, 6 H, 2 × $SiCH_3$); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.3, 149.1, 138.9, 128.0, 127.0, 125.7, 124.0, 60.5, 44.4, 36.7, 29.1, 25.9, 21.7, 18.2, -5.4; **MS** (EI) *m/z* (%) 336 (M^+, 2.38), 150 (100); **IR** (neat): ν = 2955, 2930, 2857, 1766, 1469, 1442, 1367, 1253, 1185, 1087, 1048, 1006 cm^{-1}.

5.1.2 Synthesis of the carboxylic acid **2m**

5.1.2.1 With 4-OH-TEMPO as co-catalyst (lzz-2-118)

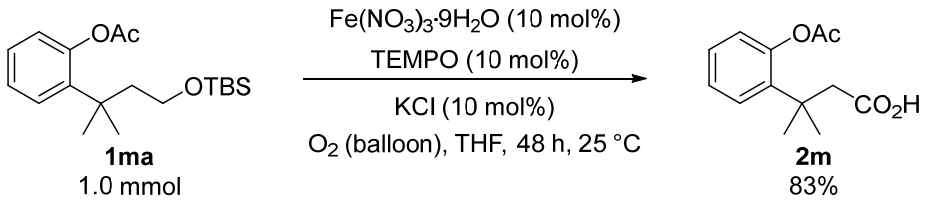


1ma
1.0 mmol

2m
NMR yield = 70%

Following **Typical Procedure III**, the reaction of **1ma** (332.4 mg, 1.0 mmol), KCl (7.9 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (40.9 mg, 0.1 mmol), and 4-OH-TEMPO (17.5 mg, 0.1 mmol) in THF (1.0 mL) was stirred at room temperature for 96 h to afford **2m** in 70% NMR yield as determined by the ^{1}H NMR analysis with CH_2Br_2 as the internal standard.

5.1.2.2 With TEMPO as co-catalyst (lzz-2-119)



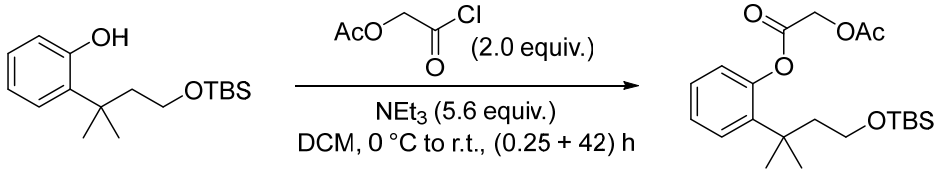
1ma
1.0 mmol

2m
83%

Following **Typical Procedure IV**, the reaction of **1ma** (335.0 mg, 1.0 mmol), KCl (7.5 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (40.6 mg, 0.1 mmol), and TEMPO (16.1 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 48 h to afford **2m**[22] (196.1 mg, 83%) [eluent: petroleum ether (60-90 °C)/ethyl acetate = 5:1 (480 mL) to 2:1 (360 mL)] as a pale yellow solid: **m.p.** 97.2-98.1 °C (*n*-hexane/ethyl acetate); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.41-7.35 (m, 1 H, ArH), 7.28-7.22 (m, 1 H, ArH), 7.20-7.14 (m, 1 H, ArH), 7.06-7.00 (m, 1 H, ArH), 2.79 (s, 2 H, CH_2), 2.35 (s, 3 H, CH_3), 1.47 (s, 6 H, 3 × CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 176.4, 169.3, 148.8, 137.9, 127.8, 127.5, 125.8, 124.0, 45.6, 36.5, 28.4, 21.7; **MS** (ESI) *m/z* 259 $(M+Na)^+$; **IR** (neat): ν = 3500-2400, 1764, 1716, 1483, 1442, 1420, 1407, 1368, 1242, 1192, 1176, 1164, 1082, 1008 cm^{-1}.

5.2 Synthesis of 3-(2-(2-(acetyloxy)acetyl)oxy)phenyl-3-methylbutanoic acid **2n**

5.2.1 Synthesis of 2-(4-(*tert*-butyldimethylsilyloxy)-2-methylbutan-2-yl)phenyl 2-(acetyloxy)acetate **1na** (lzz-2-125)



9
5.0 mmol

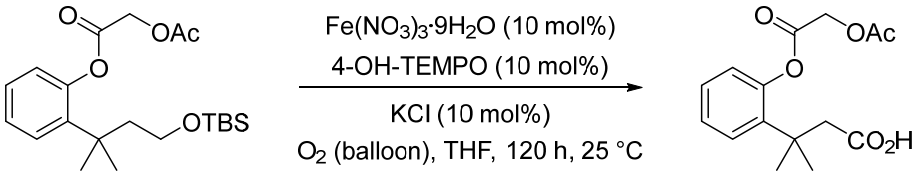
1na
94%

A dried flask was degassed and refilled with argon for three times. To this flask were added phenol **9** (1433.1 mg, 5.0 mmol), anhydrous DCM (100 mL), and NEt_3 (3.9 mL, 0.728 g/mL, 2.8392 g, 27.8 mmol) subsequently. The solution was stirred in an ice-water bath and 2-(acetyloxy)acetyl chloride (1.1 mL, 1.27 g/mL, 1.3970 g, 10.0 mmol) was added dropwise over 15 min. The resulting solution was stirred at room temperature for 42 h, quenched with H_2O (50 mL) in an ice-water bath, and extracted with DCM (25 mL × 3). The combined organic layer was dried over anhydrous Na_2SO_4. After removing the solid by filtration, the filtrate was concentrated and the residue was

purified by column chromatography on silica gel [eluent: petroleum ether (60-90 °C)/ethyl acetate = 20:1 (210 mL)] to afford **1na**[24] as a pale yellow oil (1805.4 mg, 94%): **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.37-7.32 (m, 1 H, ArH), 7.26-7.15 (m, 2 H, ArH), 7.05-7.01 (m, 1 H, ArH), 4.87 (s, 2 H, CH_2), 3.40 (t, *J* = 7.4 Hz, 2 H, OCH_2), 2.20 (s, 3 H, CH_3), 1.98 (t, *J* = 7.4 Hz, 2 H, CH_2), 1.36 (s, 6 H, 2 × CH_3), 0.83 (s, 9 H, 3 × CH_3), -0.06 (s, 6 H, 2 × $SiCH_3$); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.2, 166.4, 148.5, 138.9, 128.2, 127.1, 126.1, 123.6, 61.0, 60.4, 44.4, 36.8, 29.1, 25.9, 20.4, 18.2, -5.5; **MS** (ESI) *m/z* 395 $(M+H)^+$; **IR** (neat): ν = 2954, 2930, 2884, 2857, 1784, 1756, 1471, 1442, 1421, 1382, 1253, 1233, 1183, 1161, 1083, 1050 cm^{-1}.

5.2.2 Synthesis of the carboxylic acid **2n**

5.2.2.1 With 4-OH-TEMPO as co-catalyst (lzz-2-127)

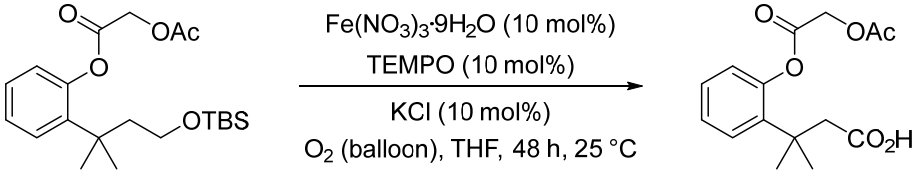


1na
1.0 mmol

2n
NMR yield = 60%

Following **Typical Procedure IV**, the reaction of **1na** (392.2 mg, 1.0 mmol), KCl (7.9 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (40.6 mg, 0.1 mmol), and 4-OH-TEMPO (17.8 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 120 h to afford **2n** in 60% NMR yield as determined by the ^{1}H NMR analysis with CH_2Br_2 as the internal standard.

5.2.2.2 With TEMPO as co-catalyst (lzz-2-128)



1na
1.0 mmol

2n
75%

Following **Typical Procedure IV**, the reaction of **1na** (394.3 mg, 1.0 mmol), KCl (7.8 mg, 0.1 mmol), $Fe(NO_3)_3$•$9H_2O$ (40.6 mg, 0.1 mmol), and TEMPO (15.9 mg, 0.1 mmol) in THF (1.0 mL) was stirred at 25 °C for 48 h to afford **2n**[24] (222.8 mg, 75%) [eluent: petroleum ether (60-90 °C)/ethyl acetate = 5:1 (480 mL) to 2:1 (600 mL)] as a

pale yellow solid: **m.p.** 99.5-100.5 °C (DCM); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 7.6 Hz, 1 H, ArH), 7.27-7.14 (m, 2 H, ArH), 7.05 (d, *J* = 7.6 Hz, 1 H, ArH), 4.86 (s, 2 H, CH_2), 2.77 (s, 2 H, CH_2), 2.18 (s, 3 H, CH_3), 1.45 (s, 6 H, 2 × CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 177.3, 170.3, 166.3, 148.2, 137.9, 127.9, 127.5, 126.1, 123.5, 61.0, 45.6, 36.4, 28.4, 20.3; **MS** (ESI) *m/z* 317 $(M+Na)^+$; **IR** (neat): ν = 3400-2700, 1779, 1750, 1705, 1487, 1442, 1419, 1382, 1234, 1186, 1160, 1080, 1048 cm^{-1}.

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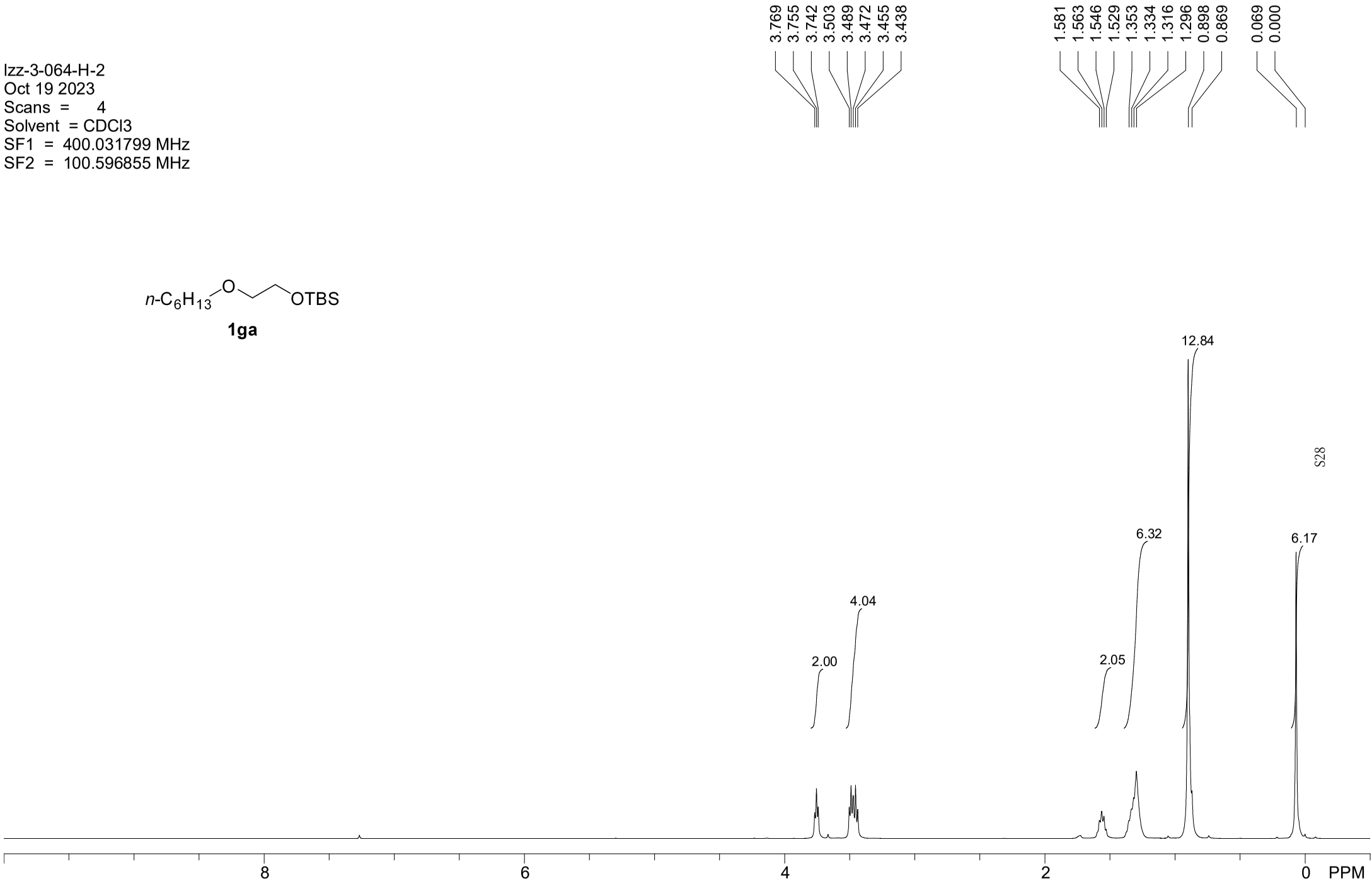
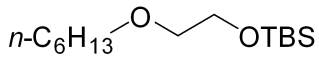
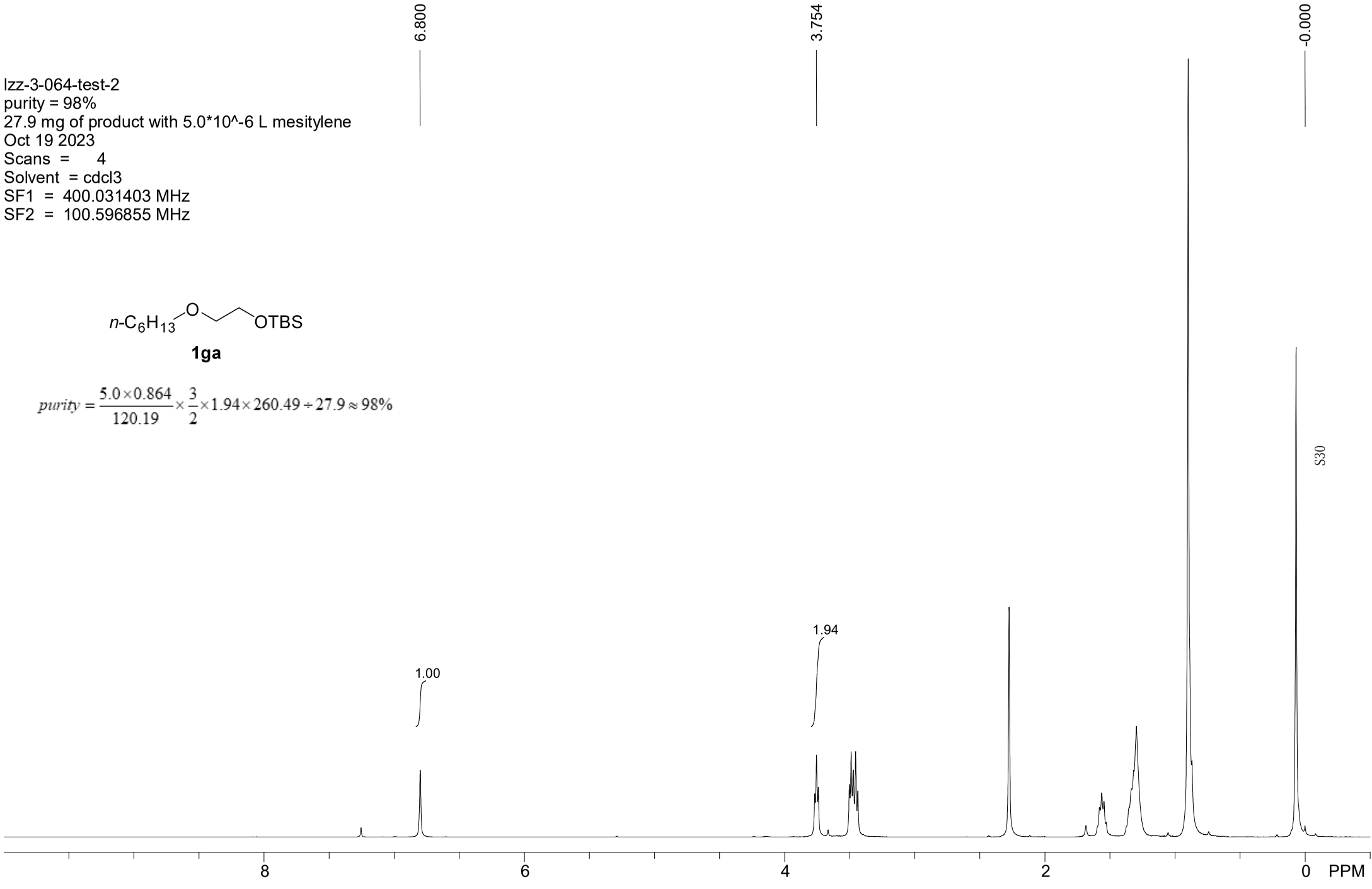
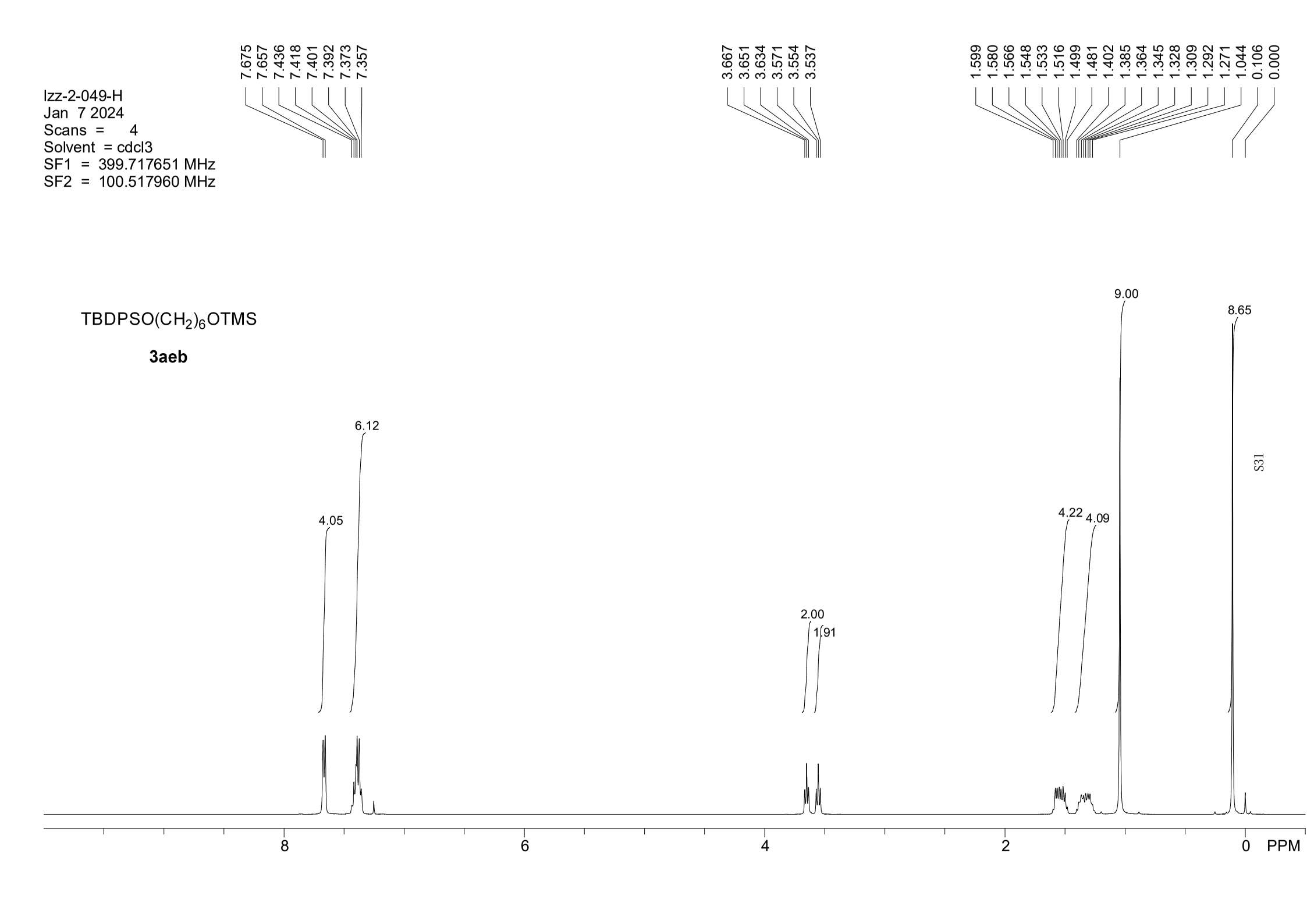

3.769
3.755
3.742
3.503
3.489
3.472
3.455
3.438
1.581
1.563
1.546
1.529
1.353
1.334
1.316
1.296
0.898
0.869
0.069
0.000
lzz-3-064-H-2
Oct 19 2023
Scans = 4
Solvent = CDCl3
SF1 = 400.031799 MHz
SF2 = 100.596855 MHz
n-C6H13
O
OTBS
1ga
12.84
6.32
6.17
4.04
2.00
2.05
S28
8
6
4
2
0
PPM

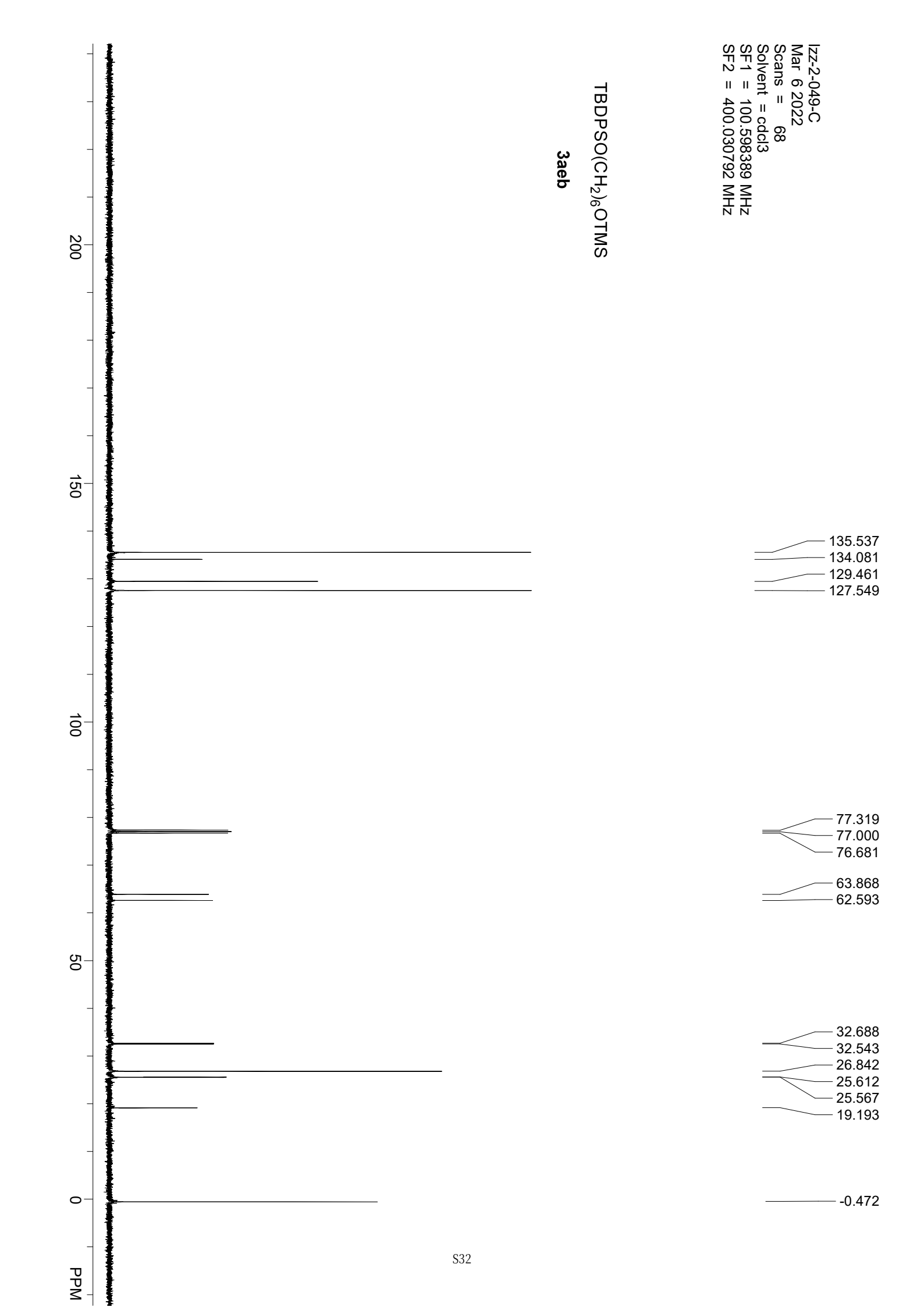
lzz-3-064
CDCl3
Sun Oct 22 01:16:54 2023
Scans = 120
SF1 = 100.617798 MHz
SF2 = 1.000000 MHz

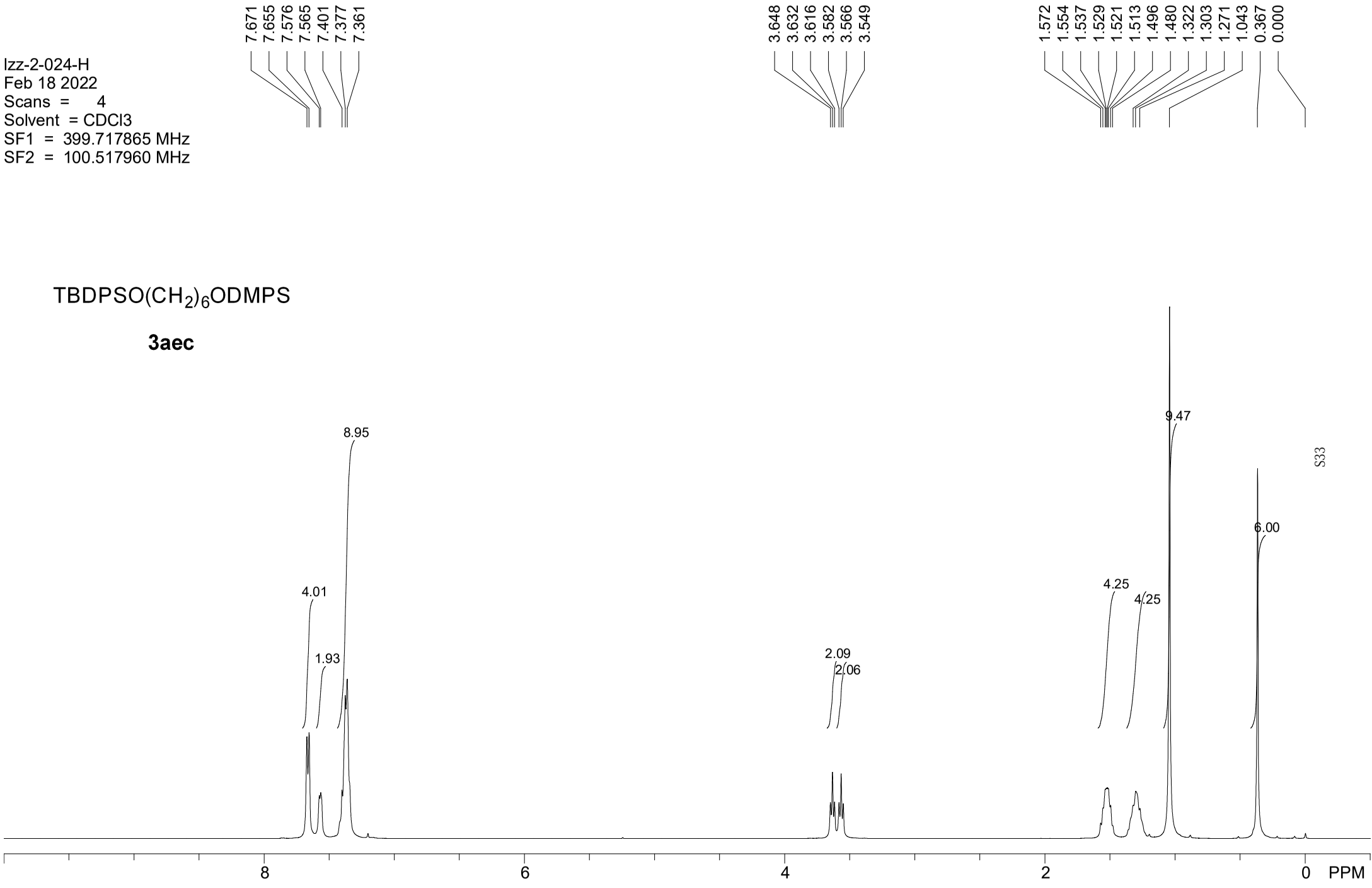


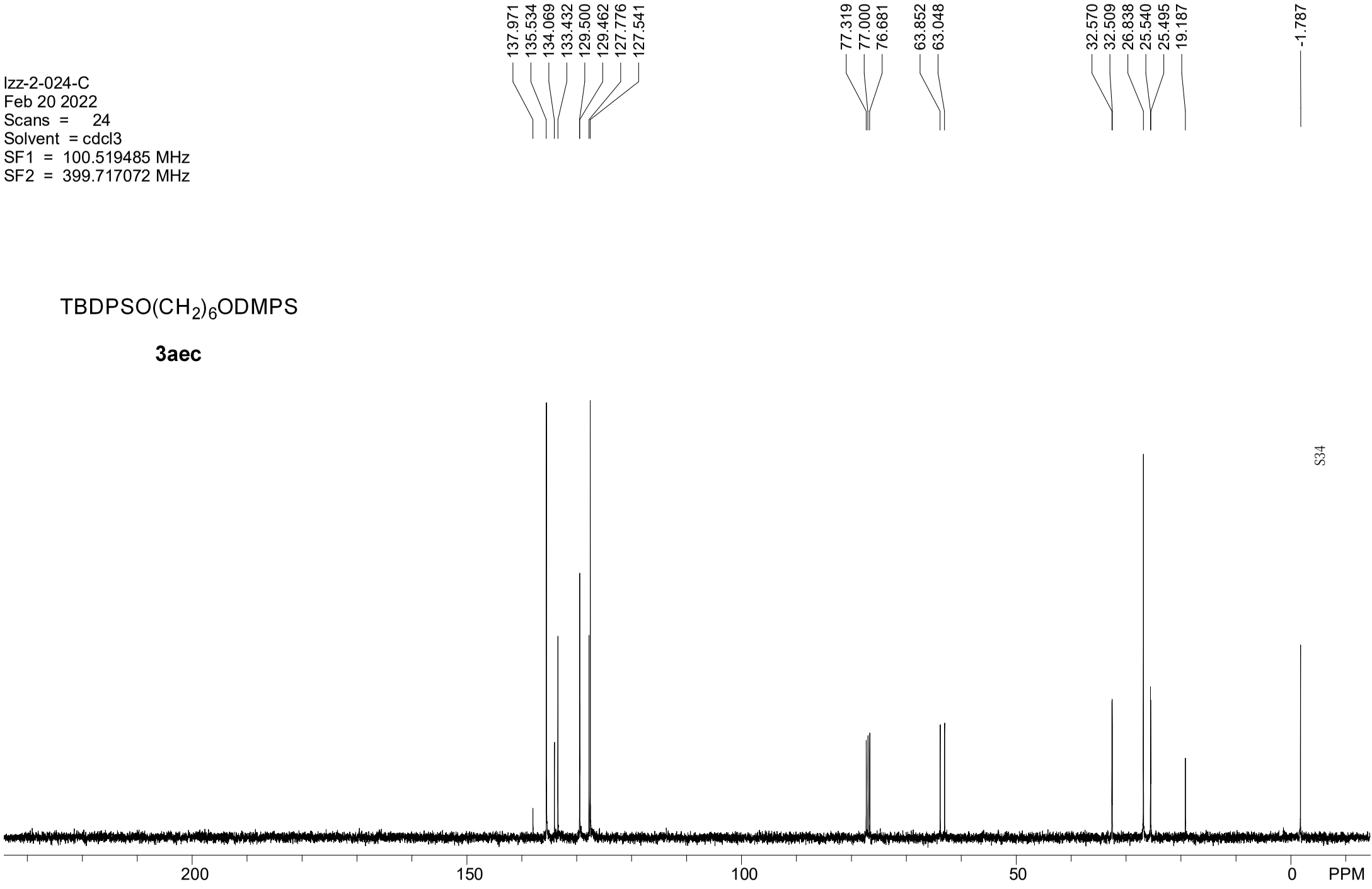
1ga

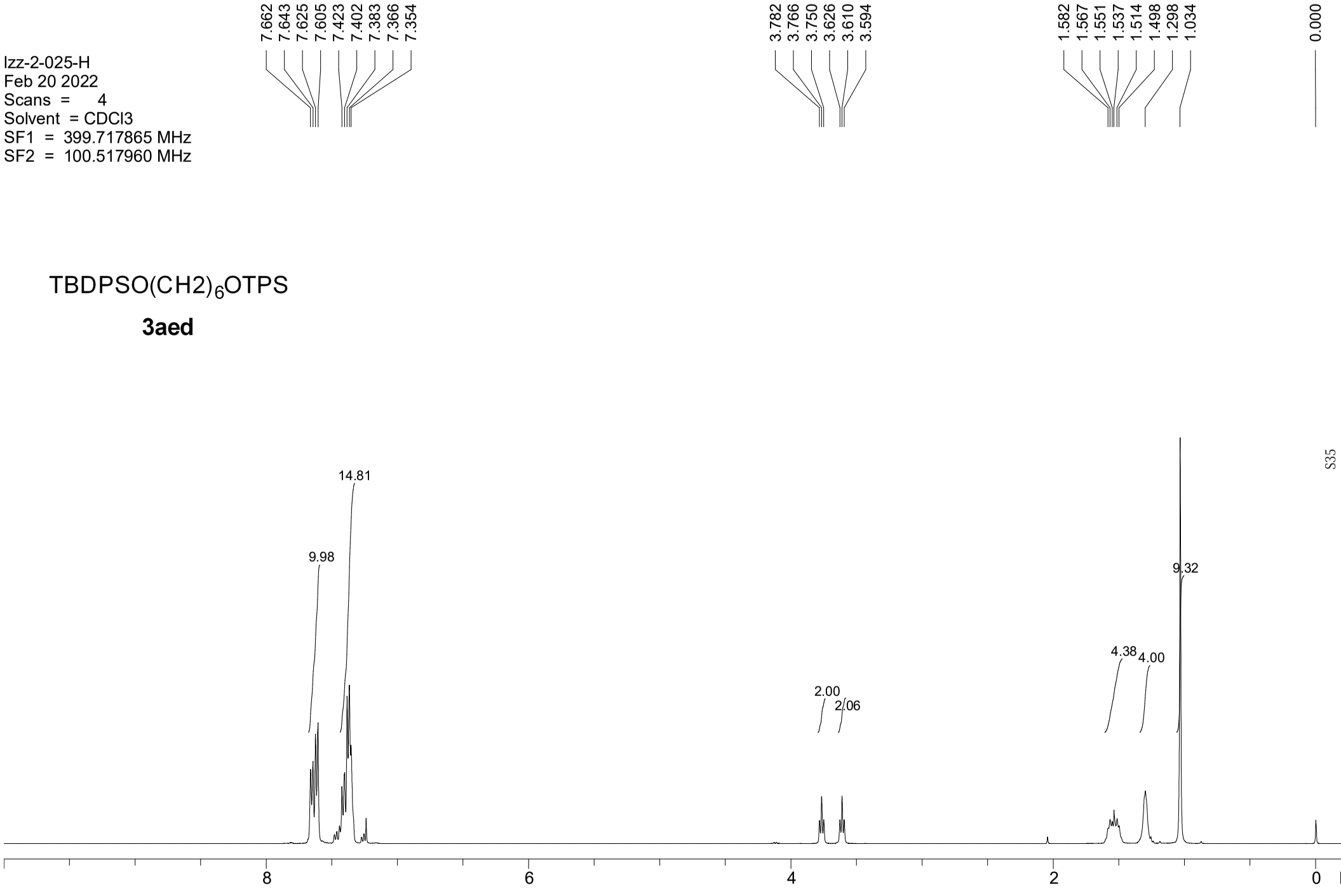

6.800
3.754
-0.000
lzz-3-064-test-2
purity = 98%
27.9 mg of product with 5.0*10^-6 L mesitylene
Oct 19 2023
Scans = 4
Solvent = cdcl3
SF1 = 400.031403 MHz
SF2 = 100.596855 MHz
n-C6H13
O
OTBS
1ga
purity = 5.0×0.864/120.19 × 3/2 × 1.94 × 260.49 ÷ 27.9 ≈ 98%
S30
1.94
1.00
8
6
4
2
0
PPM


7.675
7.657
7.436
7.418
7.401
7.392
7.373
7.357
3.667
3.651
3.634
3.571
3.554
3.537
1.599
1.580
1.566
1.548
1.533
1.516
1.499
1.481
1.402
1.385
1.364
1.345
1.328
1.309
1.292
1.271
1.044
0.106
0.000
lzz-2-049-H
Jan 7 2024
Scans = 4
Solvent = cdcl3
SF1 = 399.717651 MHz
SF2 = 100.517960 MHz
TBDPSO(CH2)6OTMS
3aeb
9.00
8.65
6.12
4.05
4.22
4.09
2.00
1.91
S31
8
6
4
2
0
PPM


lzz-2-049-C
Mar 6 2022
Scans = 68
Solvent = cdcl3
SF1 = 100.598389 MHz
SF2 = 400.030792 MHz
TBDPSO(CH2)6OTMS
3aeb
135.537
134.081
129.461
127.549
77.319
77.000
76.681
63.868
62.593
32.688
32.543
26.842
25.612
25.567
19.193
-0.472
200
150
100
50
0
PPM


7.671
7.655
7.576
7.565
7.401
7.377
7.361
3.648
3.632
3.616
3.582
3.566
3.549
1.572
1.554
1.537
1.529
1.521
1.513
1.496
1.480
1.322
1.303
1.271
1.043
0.367
0.000
lzz-2-024-H
Feb 18 2022
Scans = 4
Solvent = CDCl3
SF1 = 399.717865 MHz
SF2 = 100.517960 MHz
TBDPSO(CH2)6ODMPS
3aec
8.95
4.01
1.93
2.09
2.06
9.47
4.25
4.25
6.00
S33
8
6
4
2
0
PPM


137.971
135.534
134.069
133.432
129.500
129.462
127.776
127.541
77.319
77.000
76.681
63.852
63.048
32.570
32.509
26.838
25.540
25.495
19.187
-1.787
lzz-2-024-C
Feb 20 2022
Scans = 24
Solvent = cdcl3
SF1 = 100.519485 MHz
SF2 = 399.717072 MHz
TBDPSO(CH2)6ODMPS
3aec
S34
200
150
100
50
0
PPM

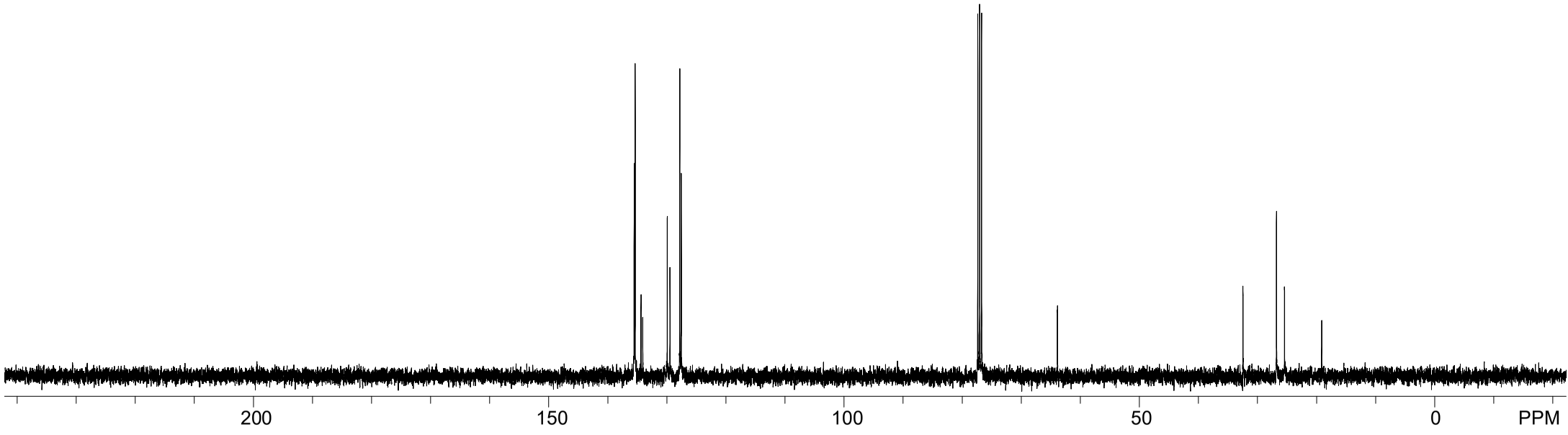

7.662
7.643
7.625
7.605
7.423
7.402
7.383
7.366
7.354
3.782
3.766
3.750
3.626
3.610
3.594
1.582
1.567
1.551
1.537
1.514
1.498
1.298
1.034
0.000
lzz-2-025-H
Feb 20 2022
Scans = 4
Solvent = CDCl3
SF1 = 399.717865 MHz
SF2 = 100.517960 MHz
TBDPSO(CH2)6OTPS
3aed
S35
14.81
9.98
2.00
2.06
4.38
4.00
9.32
8
6
4
2
0
PPM

lzz-2-025-C
Feb 21 2022
Scans = 92
Solvent = cdcl3
SF1 = 100.598389 MHz
SF2 = 400.030792 MHz

135.533
135.356
134.371
134.077
129.921
129.461
127.803
127.545
77.319
77.000
76.685
63.872
63.851
32.495
26.834
25.479
19.185

TBDPSO(CH2)$_6$OTPS

3aed



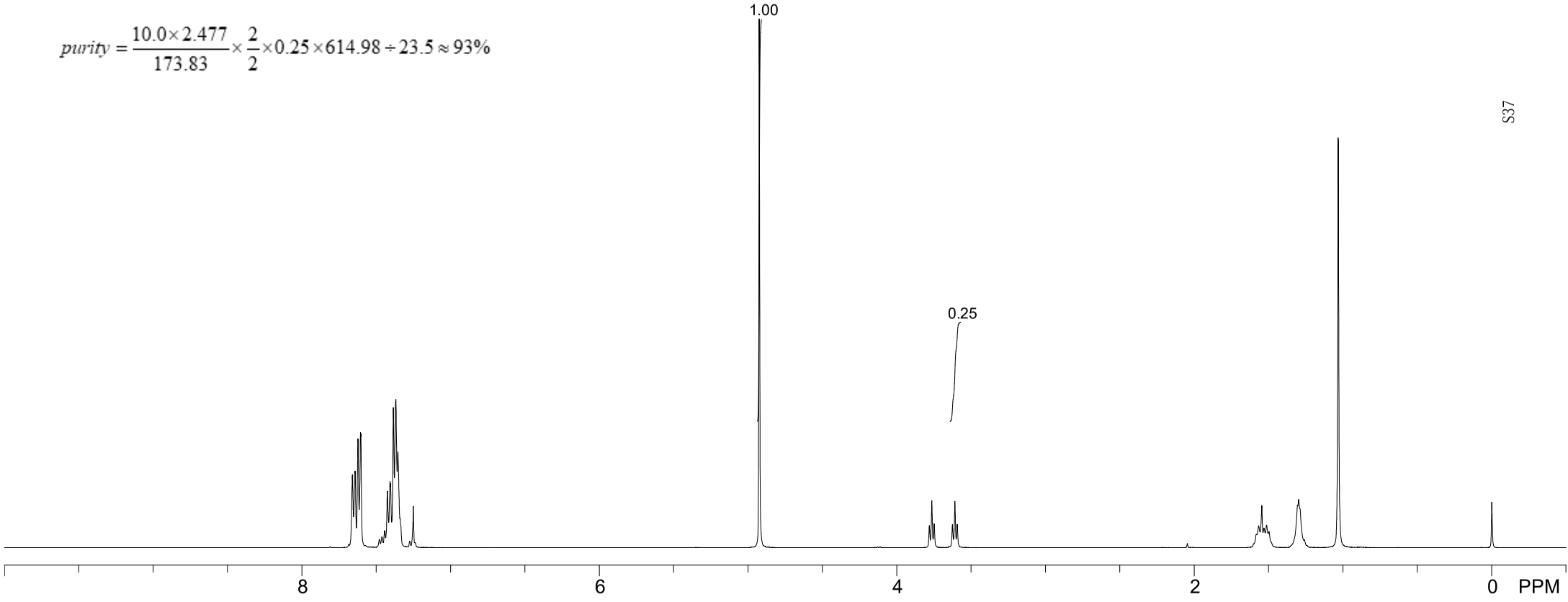
lzz-2-025-test
purity = 93%
23.5 mg of poduct with 10.0*10^-6 L dibromomethane
Feb 20 2022
Scans = 4
Solvent = cdcl3
SF1 = 399.717865 MHz
SF2 = 100.517960 MHz

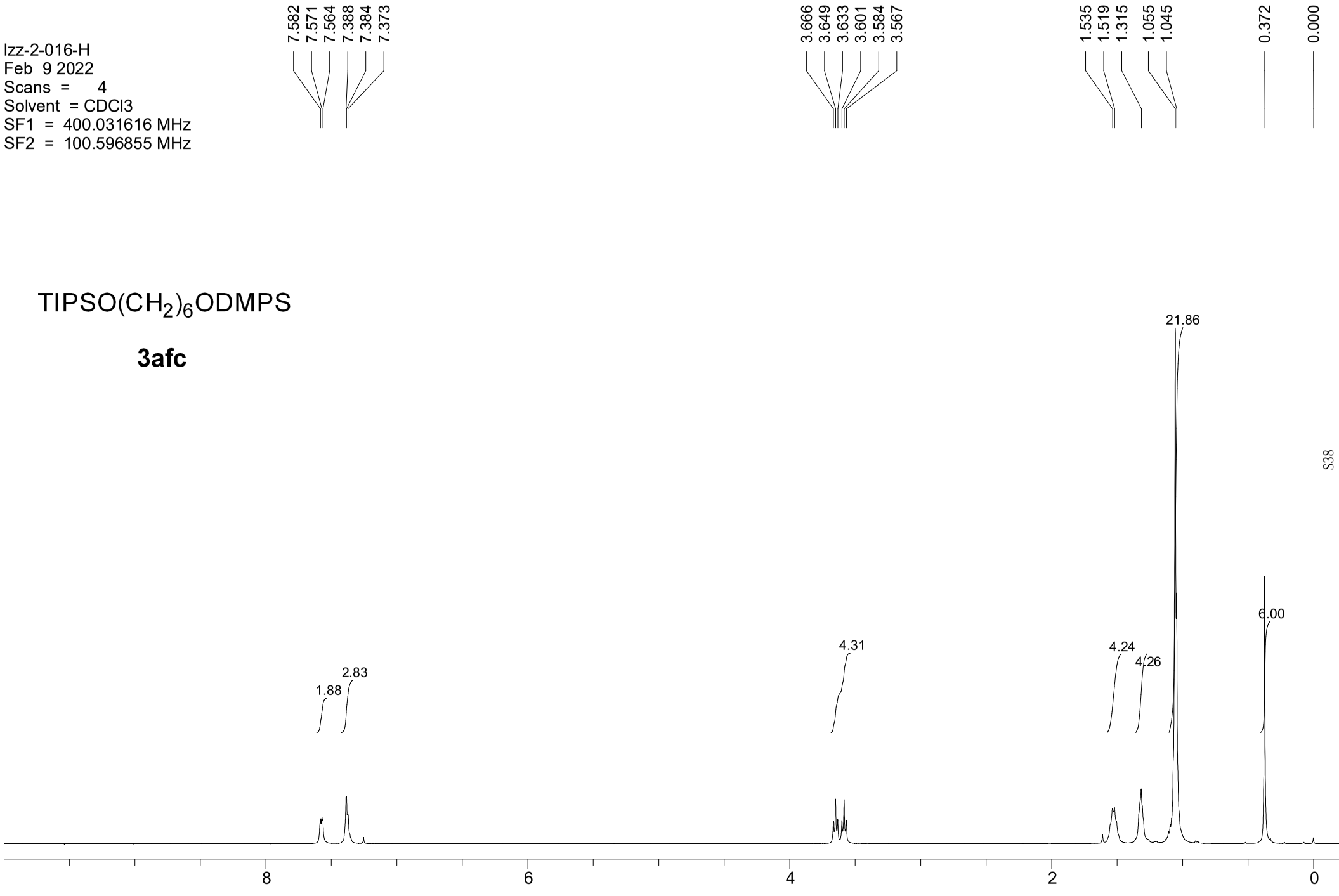
4.925
3.625
3.609
3.592
-0.000

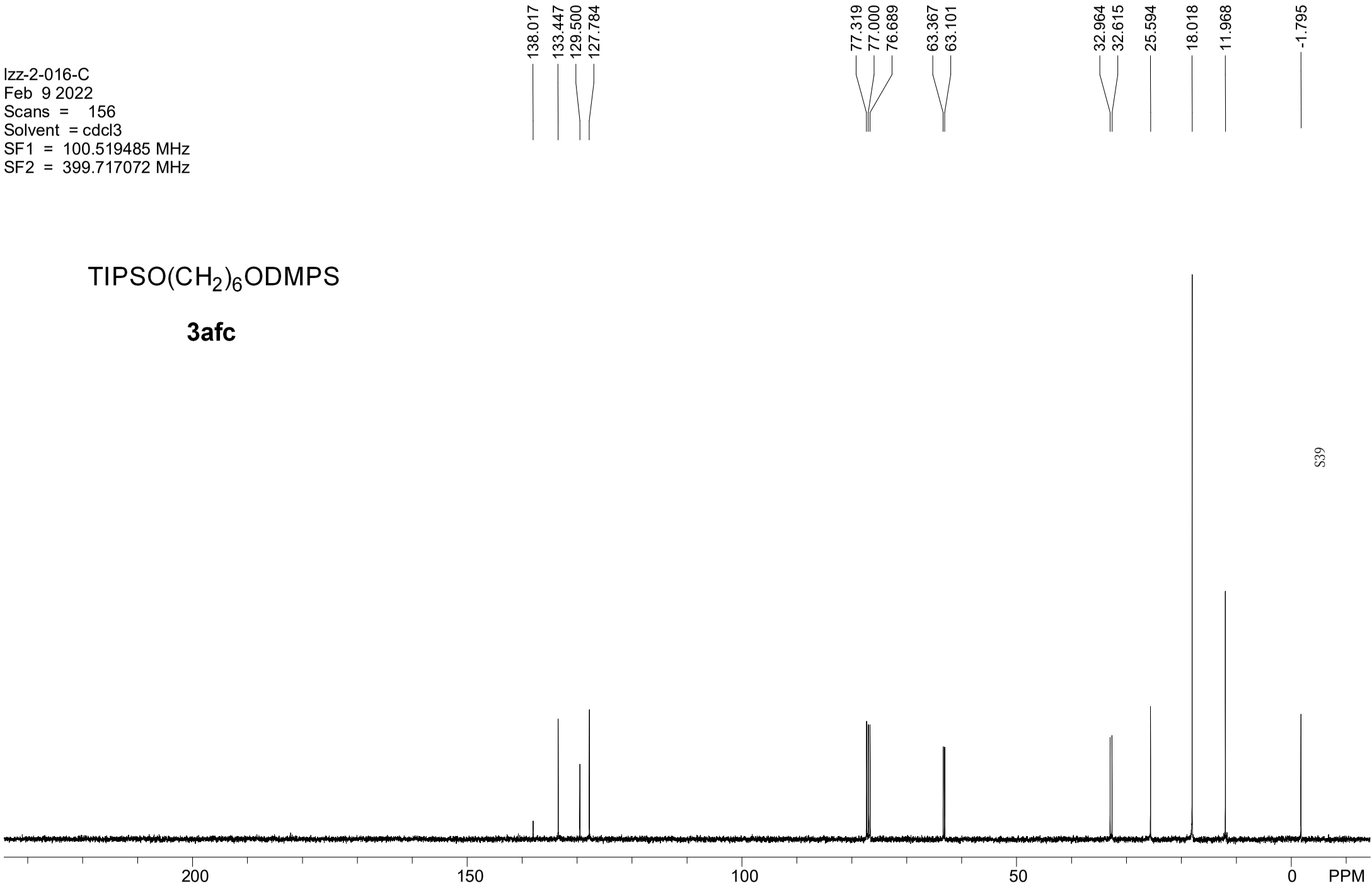
TBDPSO(CH2)$_6$OTPS

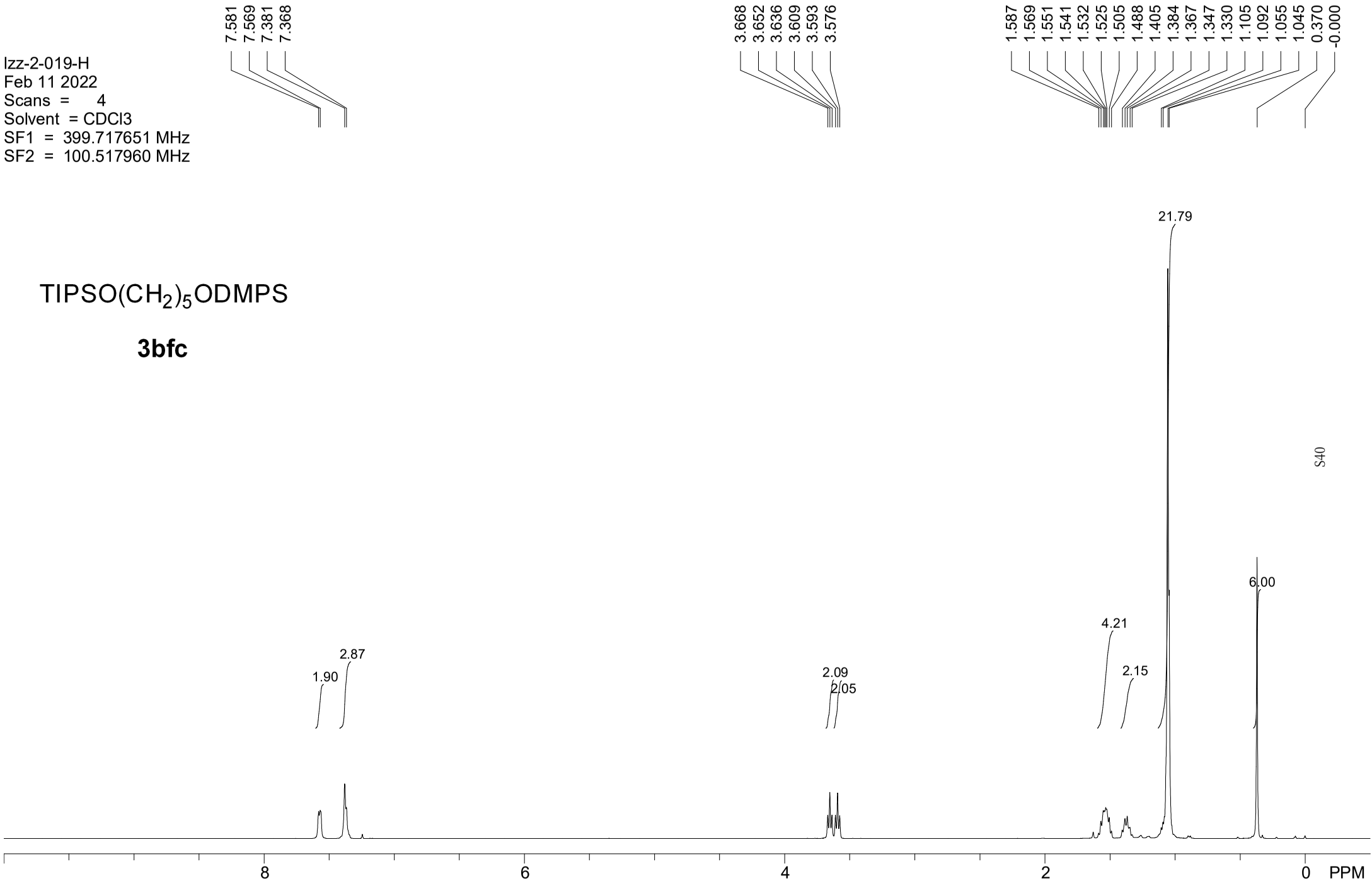
3aed

$$purity = \frac{10.0 \times 2.477}{173.83} \times \frac{2}{2} \times 0.25 \times 614.98 \div 23.5 \approx 93\%$$




lzz-2-016-H
Feb 9 2022
Scans = 4
Solvent = CDCl3
SF1 = 400.031616 MHz
SF2 = 100.596855 MHz
7.582
7.571
7.564
7.388
7.384
7.373
3.666
3.649
3.633
3.601
3.584
3.567
1.535
1.519
1.315
1.055
1.045
0.372
0.000
TIPSO(CH2)6ODMPS
3afc
21.86
6.00
4.31
4.24
4.26
2.83
1.88
S38
8
6
4
2
0
PPM


138.017
133.447
129.500
127.784
77.319
77.000
76.689
63.367
63.101
32.964
32.615
25.594
18.018
11.968
-1.795
lzz-2-016-C
Feb 9 2022
Scans = 156
Solvent = cdcl3
SF1 = 100.519485 MHz
SF2 = 399.717072 MHz
TIPSO(CH2)6ODMPS
3afc
S39
200
150
100
50
0
PPM

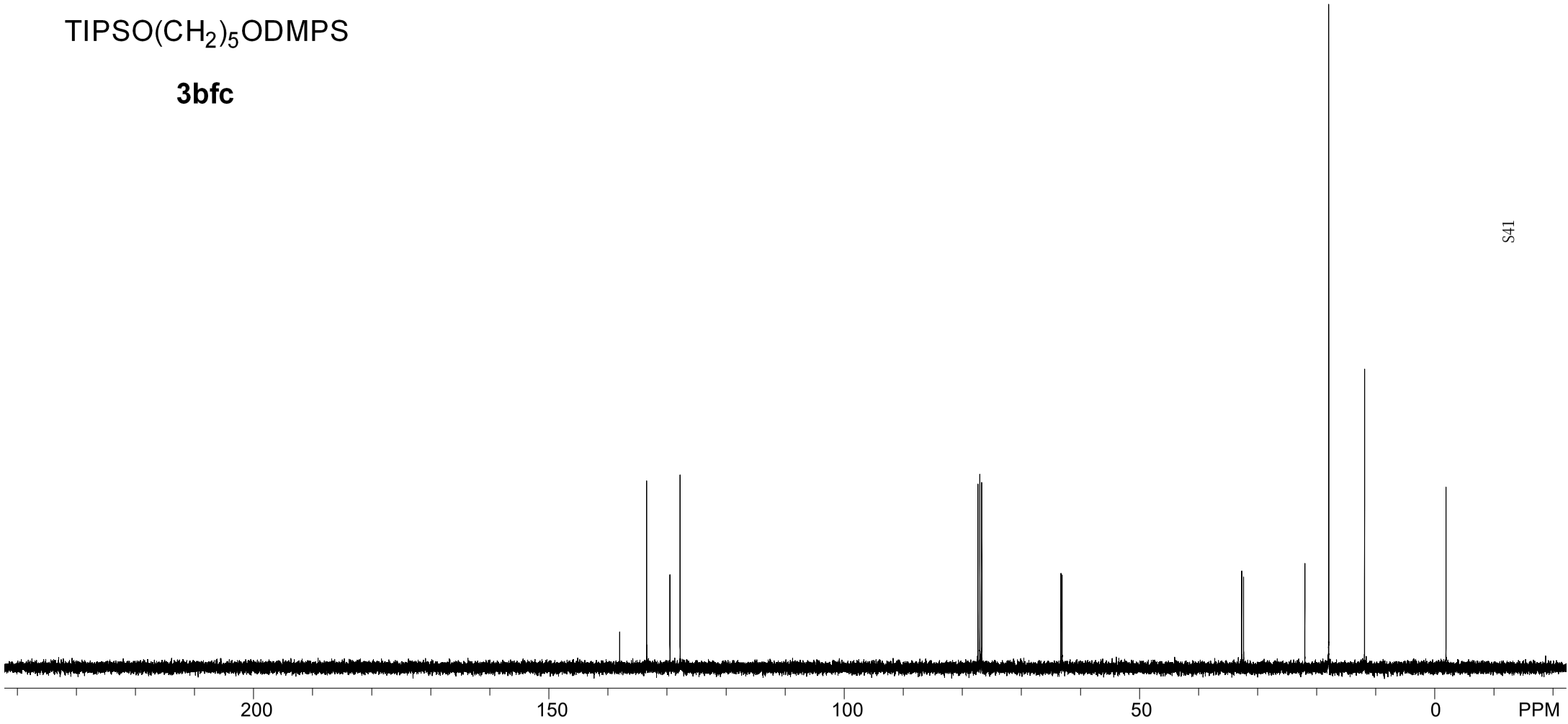

7.581
7.569
7.381
7.368
3.668
3.652
3.636
3.609
3.593
3.576
1.587
1.569
1.551
1.541
1.532
1.525
1.505
1.488
1.405
1.384
1.367
1.347
1.330
1.105
1.092
1.055
1.045
0.370
-0.000
lzz-2-019-H
Feb 11 2022
Scans = 4
Solvent = CDCl3
SF1 = 399.717651 MHz
SF2 = 100.517960 MHz
TIPSO(CH2)5ODMPS
3bfc
21.79
6.00
4.21
2.87
1.90
2.09
2.05
2.15
S40
8
6
4
2
0
PPM

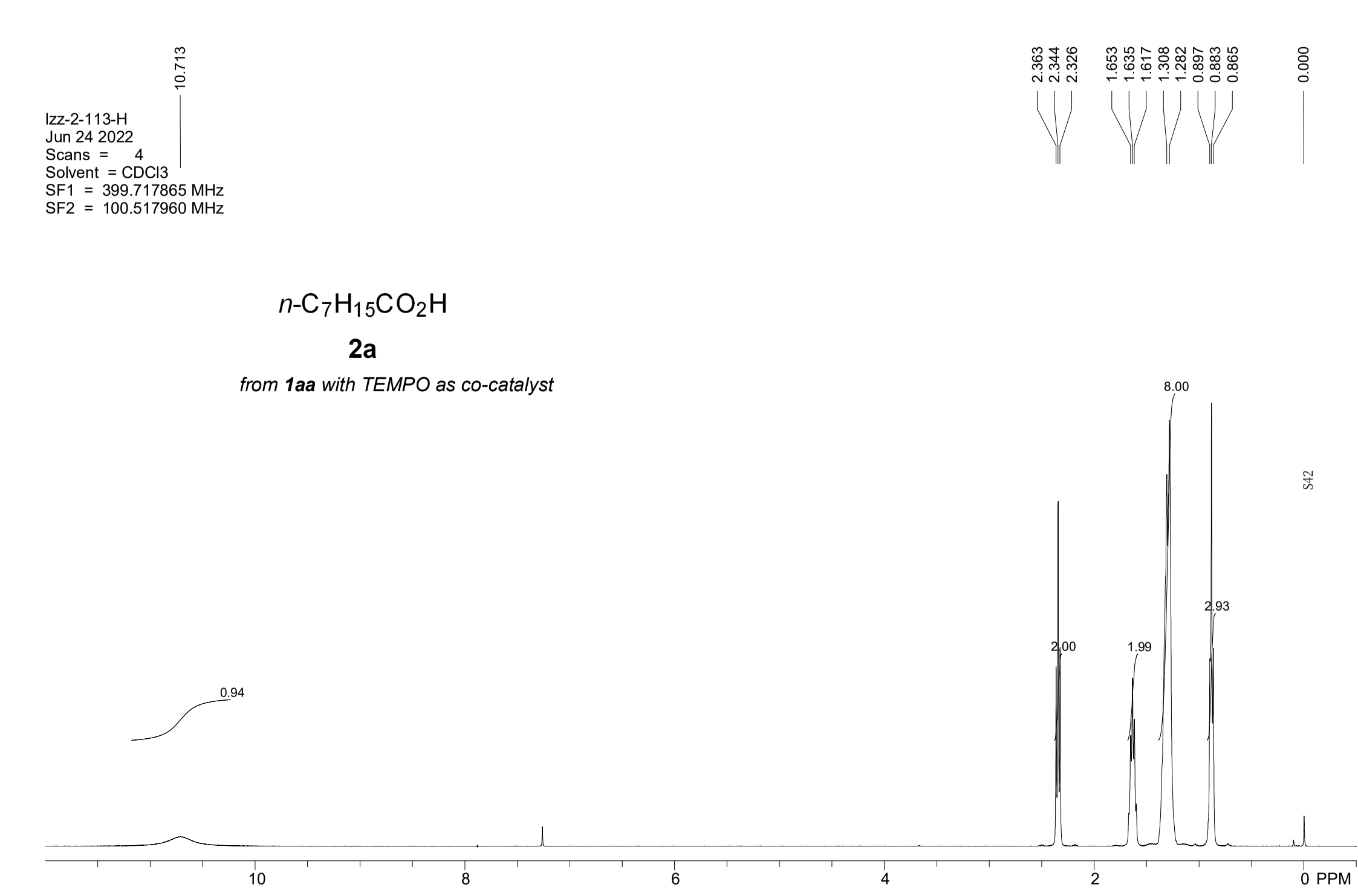
lzz-2-019-C
Feb 12 2022
Scans = 88
Solvent = cdcl3
SF1 = 100.598389 MHz
SF2 = 400.030792 MHz

138.019
133.439
129.490
127.779
77.319
77.000
76.681
63.299
63.089
32.741
32.410
22.061
18.015
11.967
-1.803

TIPSO$(CH_2)_5$ODMPS

3bfc




10.713
lzz-2-113-H
Jun 24 2022
Scans = 4
Solvent = CDCl3
SF1 = 399.717865 MHz
SF2 = 100.517960 MHz
2.363
2.344
2.326
1.653
1.635
1.617
1.308
1.282
0.897
0.883
0.865
0.000
n-C7H15CO2H
2a
from 1aa with TEMPO as co-catalyst
8.00
2.93
2.00
1.99
0.94
S42
10
8
6
4
2
0 PPM

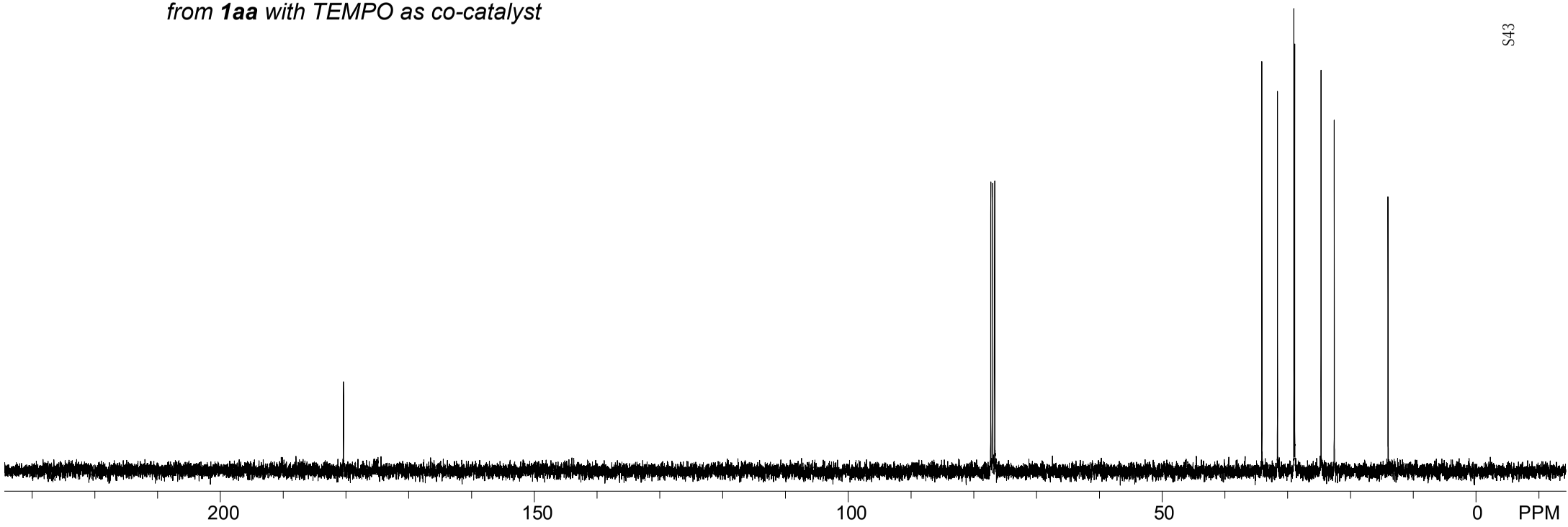
lzz-2-113-C
Jun 24 2022
Scans = 92
Solvent = cdcl3
SF1 = 100.519485 MHz
SF2 = 399.717072 MHz

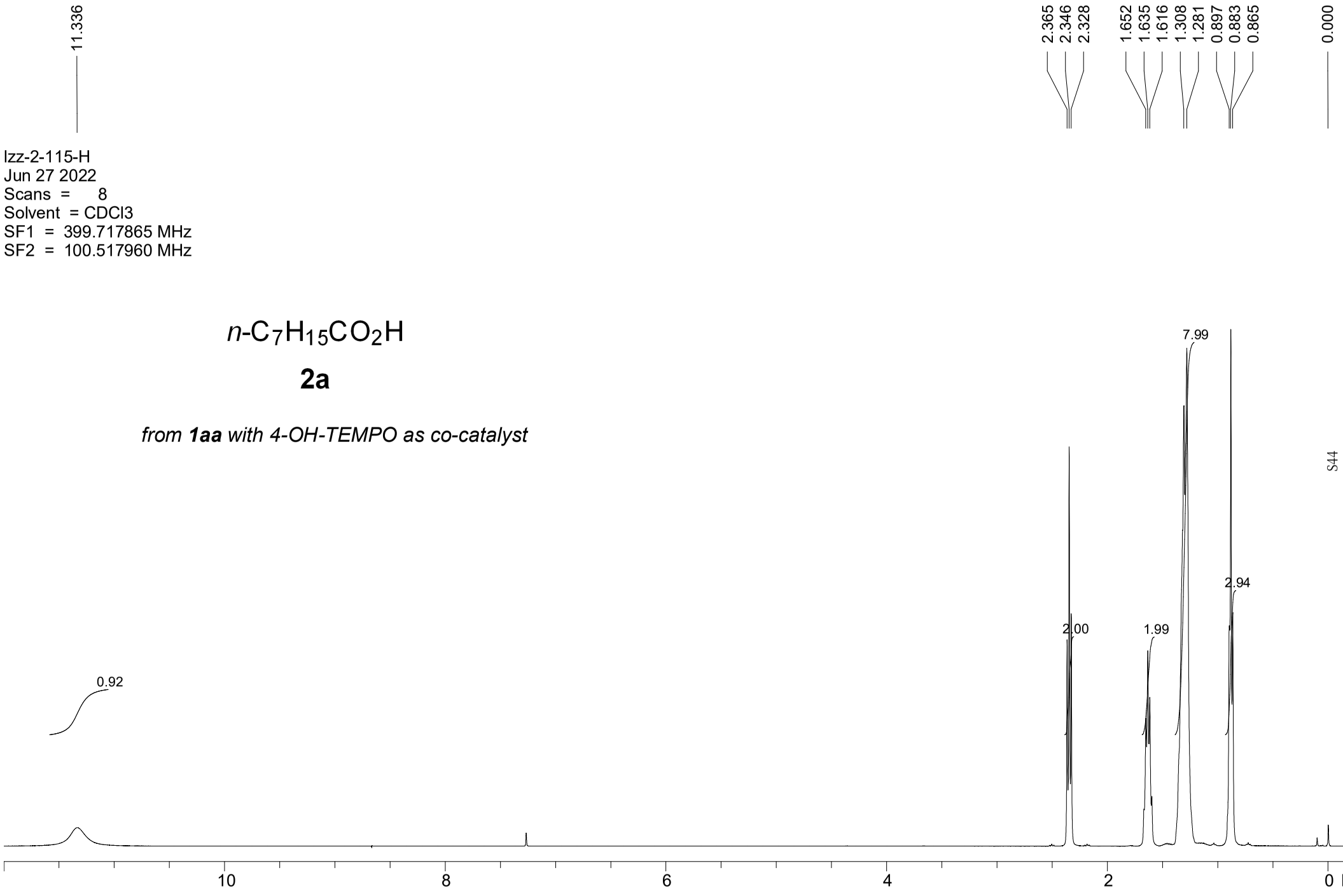
180.390
77.319
77.000
76.689
34.111
31.621
29.009
28.880
24.675
22.572
14.002

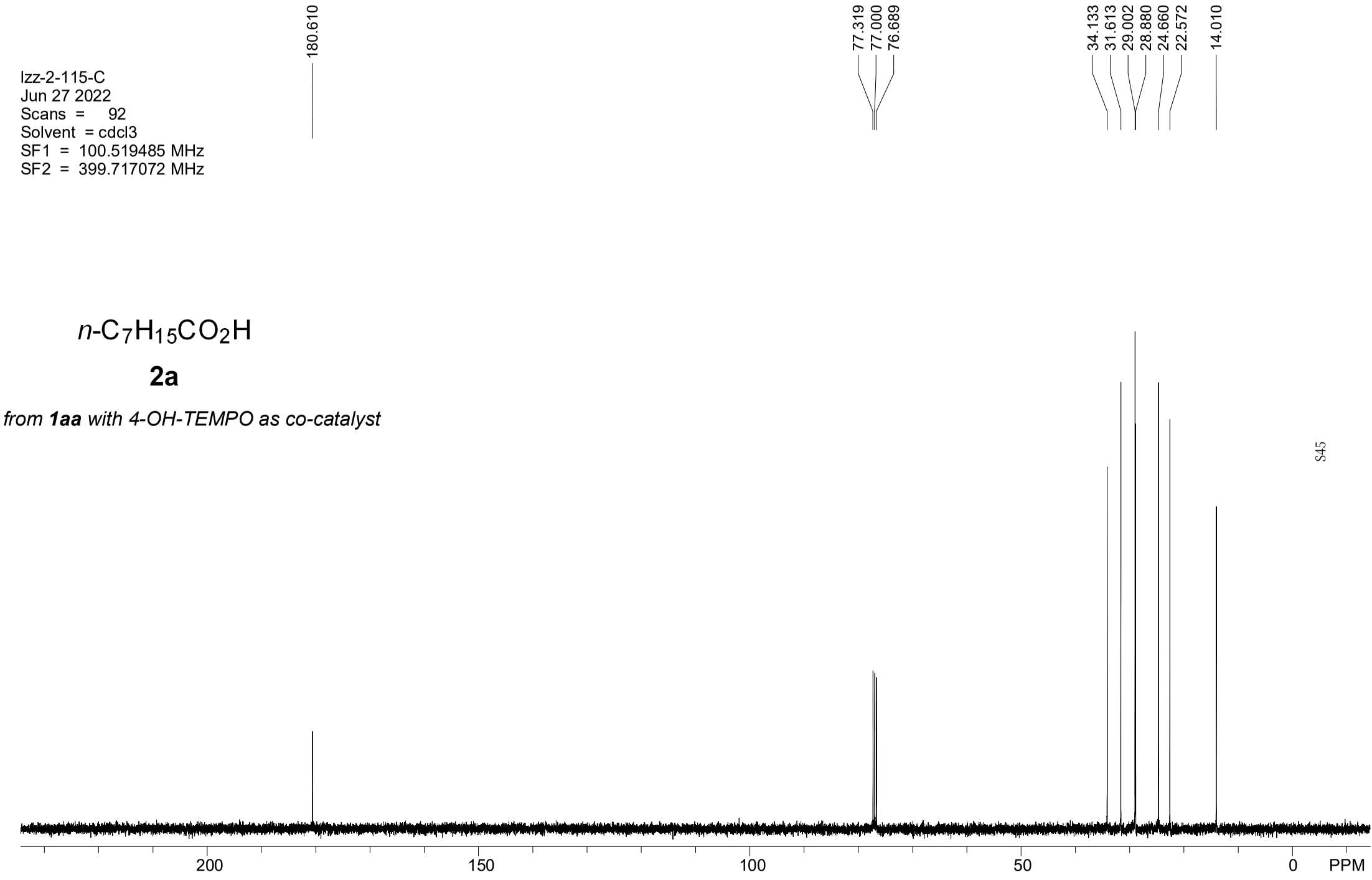
n-$C_7H_{15}CO_2H$

2a

*from **1aa** with TEMPO as co-catalyst*




11.336
2.365
2.346
2.328
1.652
1.635
1.616
1.308
1.281
0.897
0.883
0.865
0.000
lzz-2-115-H
Jun 27 2022
Scans = 8
Solvent = CDCl3
SF1 = 399.717865 MHz
SF2 = 100.517960 MHz
n-C7H15CO2H
2a
from 1aa with 4-OH-TEMPO as co-catalyst
S44
7.99
2.94
2.00
1.99
0.92
10
8
6
4
2
0 PPM

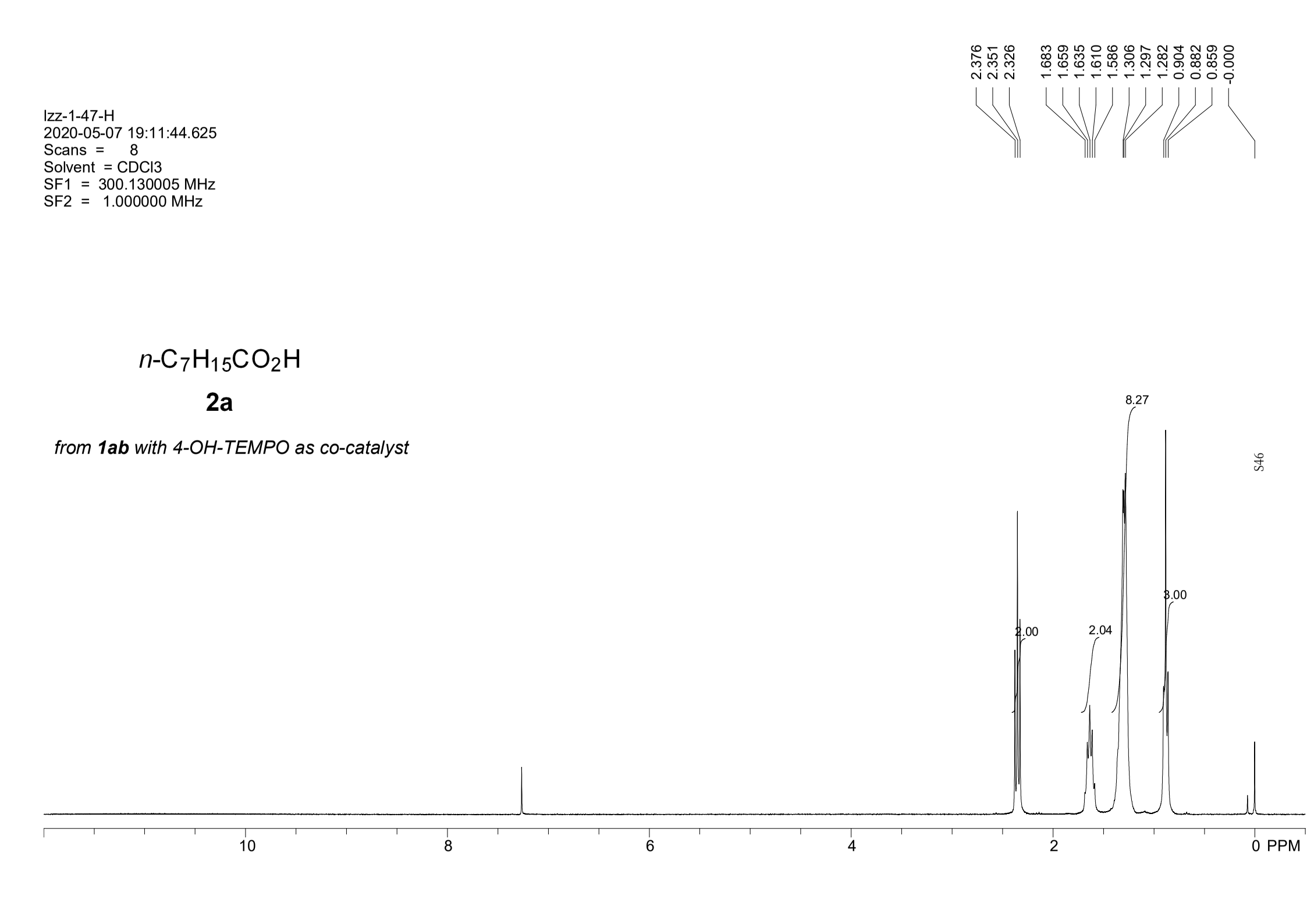


n-$C_7H_{15}CO_2H$

2a

*from **1aa** with 4-OH-TEMPO as co-catalyst*

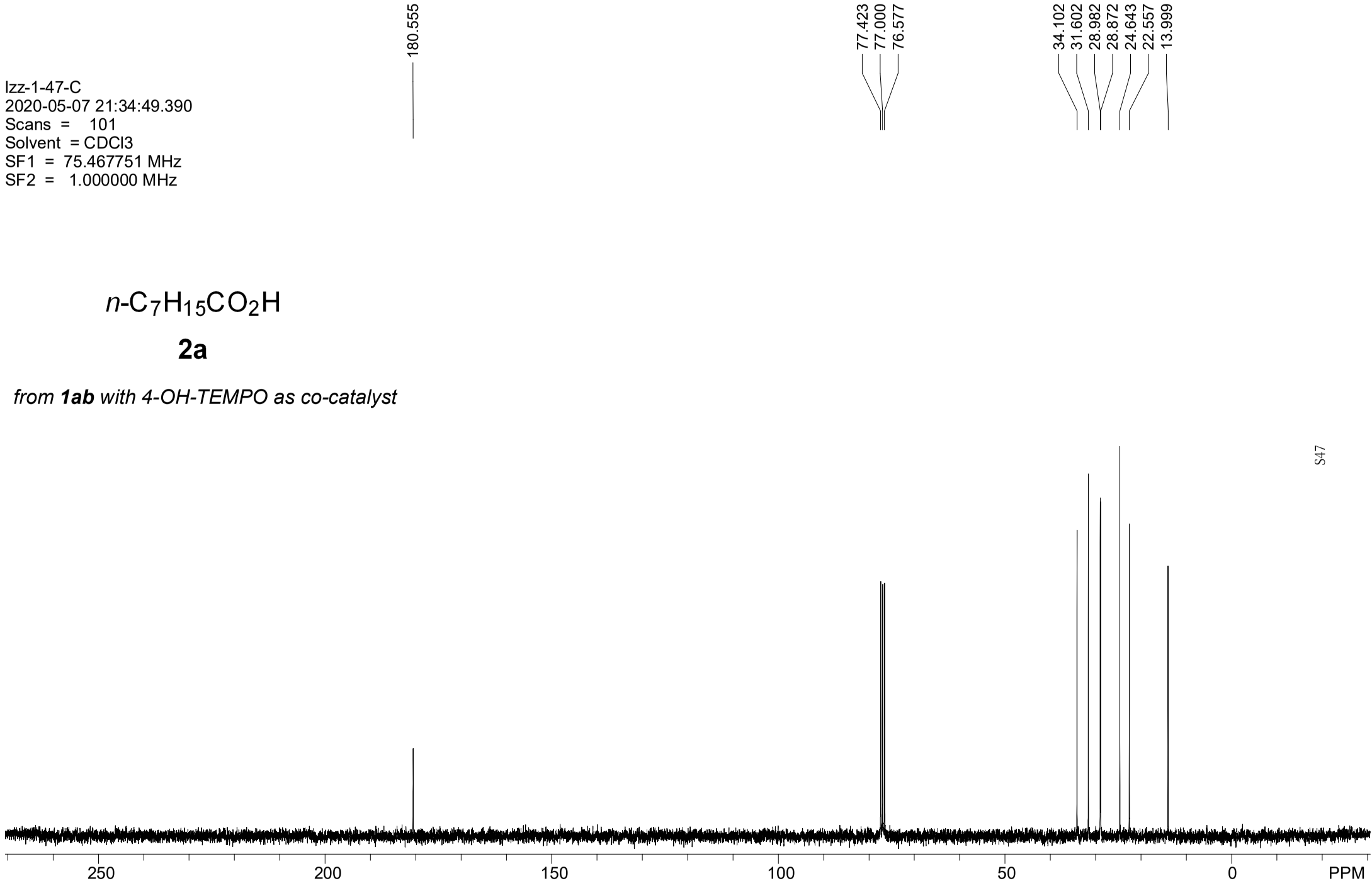
lzz-1-47-H
2020-05-07 19:11:44.625
Scans = 8
Solvent = CDCl3
SF1 = 300.130005 MHz
SF2 = 1.000000 MHz



n-$C_7H_{15}CO_2H$

2a

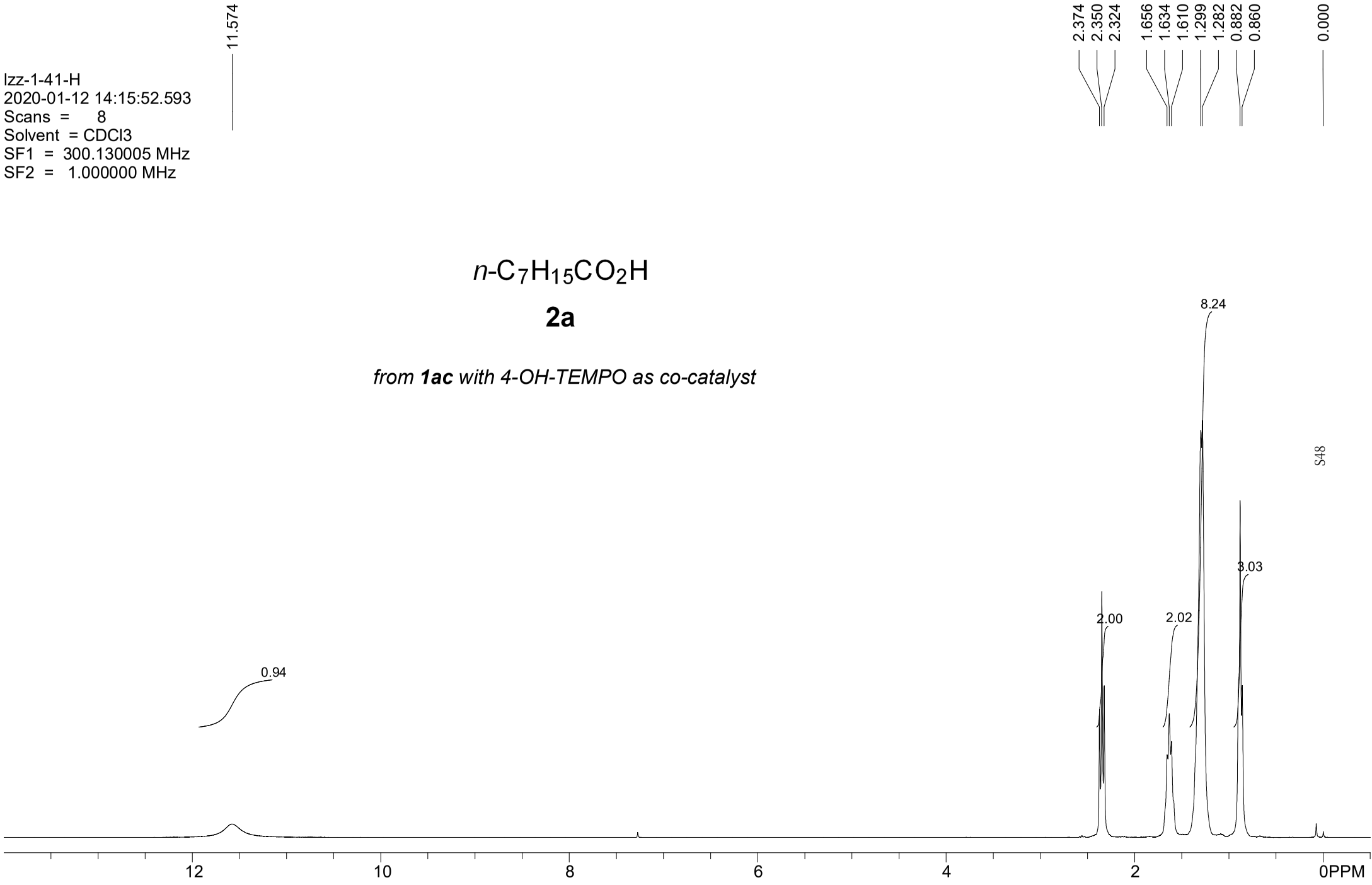
*from **1ab** with 4-OH-TEMPO as co-catalyst*



n-$C_7H_{15}CO_2H$

2a

*from **1ab** with 4-OH-TEMPO as co-catalyst*


11.574
2.374
2.350
2.324
1.656
1.634
1.610
1.299
1.282
0.882
0.860
0.000
lzz-1-41-H
2020-01-12 14:15:52.593
Scans = 8
Solvent = CDCl3
SF1 = 300.130005 MHz
SF2 = 1.000000 MHz
n-C7H15CO2H
2a
from 1ac with 4-OH-TEMPO as co-catalyst
8.24
S48
3.03
2.00
2.02
0.94
12
10
8
6
4
2
0PPM

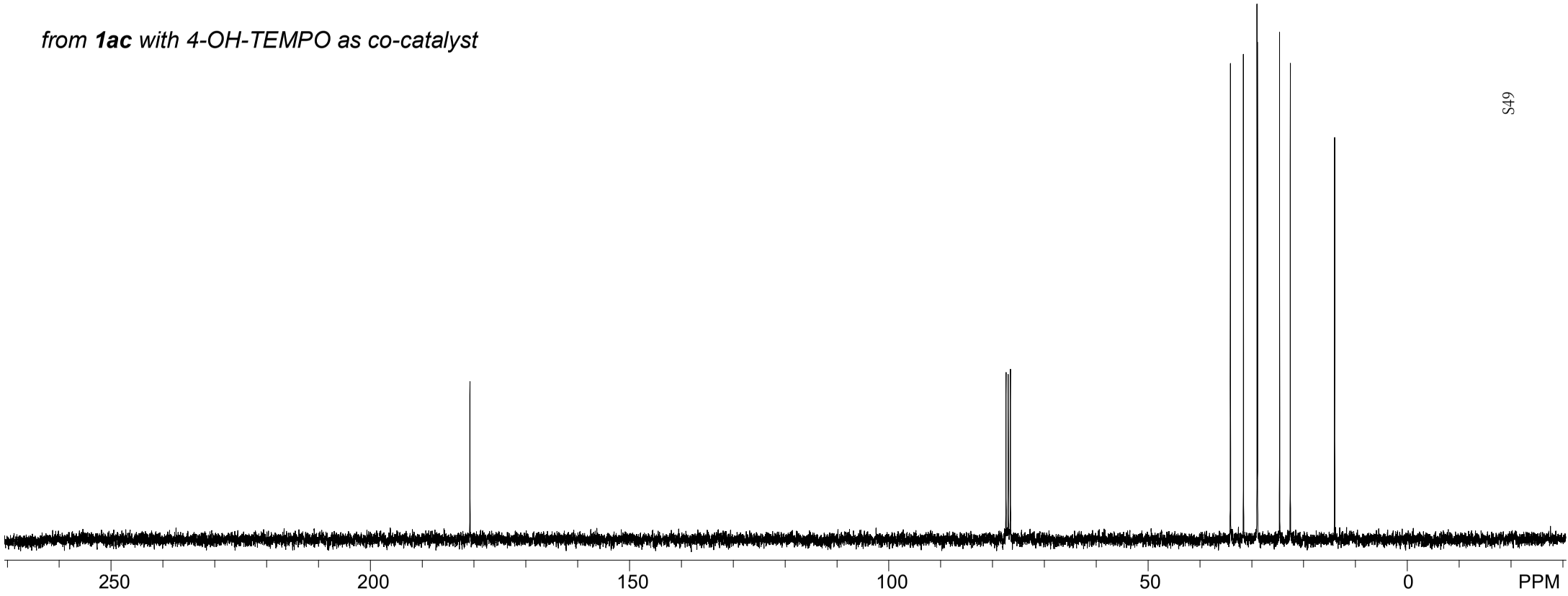
lzz-1-41-C
2020-01-13 10:12:08.312
Scans = 36
Solvent = CDCl3
SF1 = 75.467751 MHz
SF2 = 1.000000 MHz

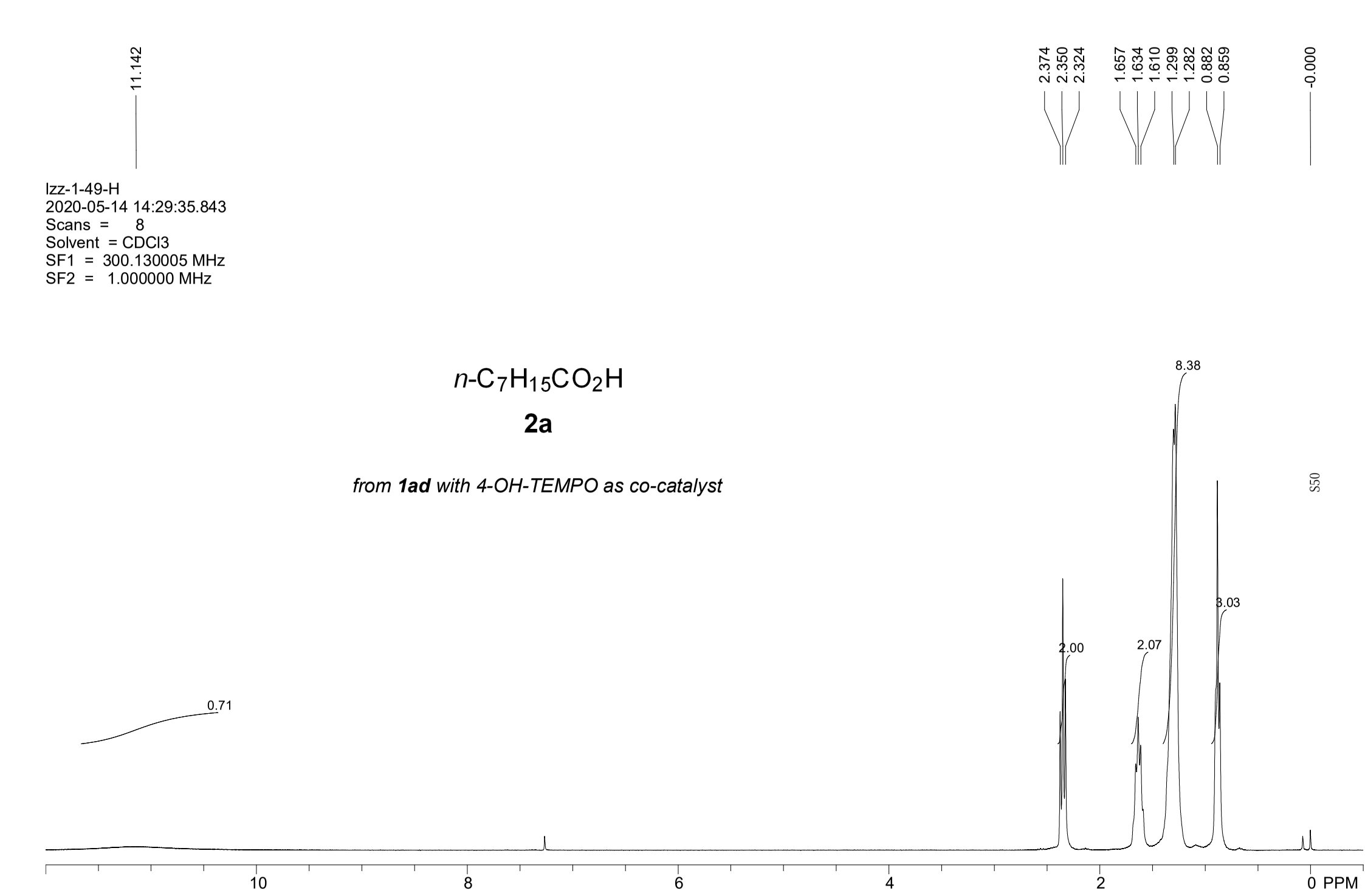
180.785
77.423
77.000
76.577
34.111
31.602
28.973
28.881
24.616
22.557
13.999

n-$C_7H_{15}CO_2H$

2a

*from **1ac** with 4-OH-TEMPO as co-catalyst*




11.142
2.374
2.350
2.324
1.657
1.634
1.610
1.299
1.282
0.882
0.859
-0.000
lzz-1-49-H
2020-05-14 14:29:35.843
Scans = 8
Solvent = CDCl3
SF1 = 300.130005 MHz
SF2 = 1.000000 MHz
n-C7H15CO2H
2a
from 1ad with 4-OH-TEMPO as co-catalyst
S50
8.38
3.03
2.00
2.07
0.71
10
8
6
4
2
0 PPM

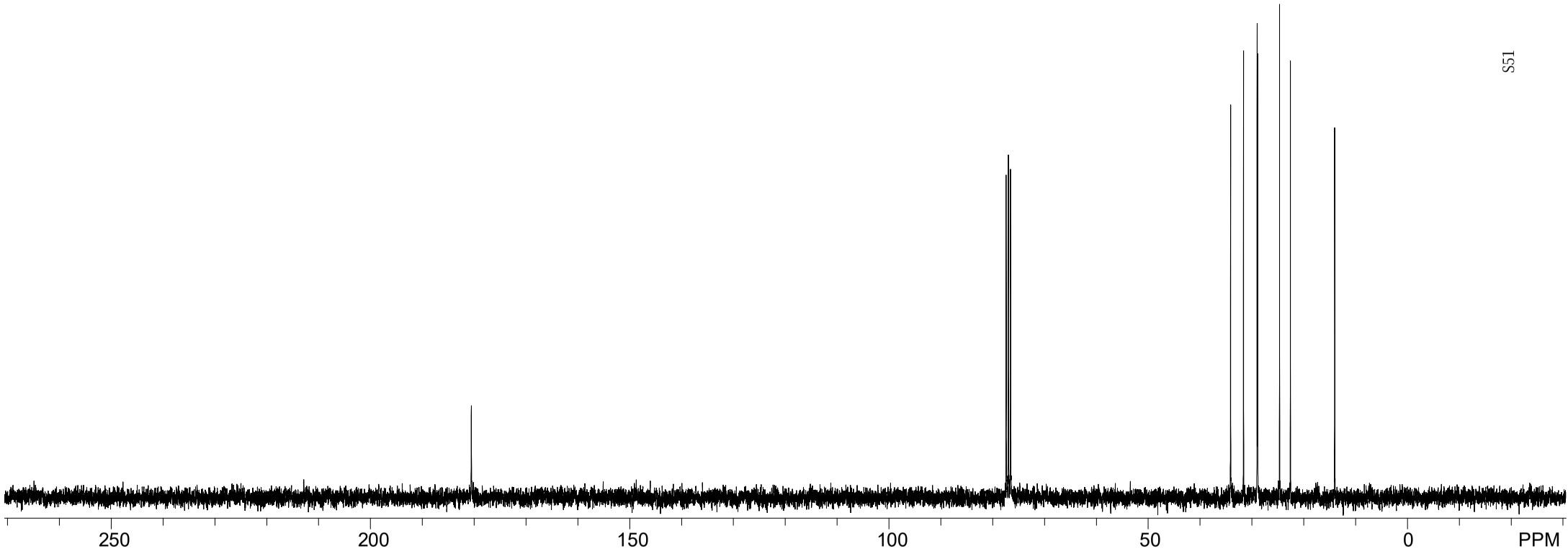
lzz-1-49-C
2020-05-14 15:37:01.109
Scans = 108
Solvent = CDCl3
SF1 = 75.467751 MHz
SF2 = 1.000000 MHz

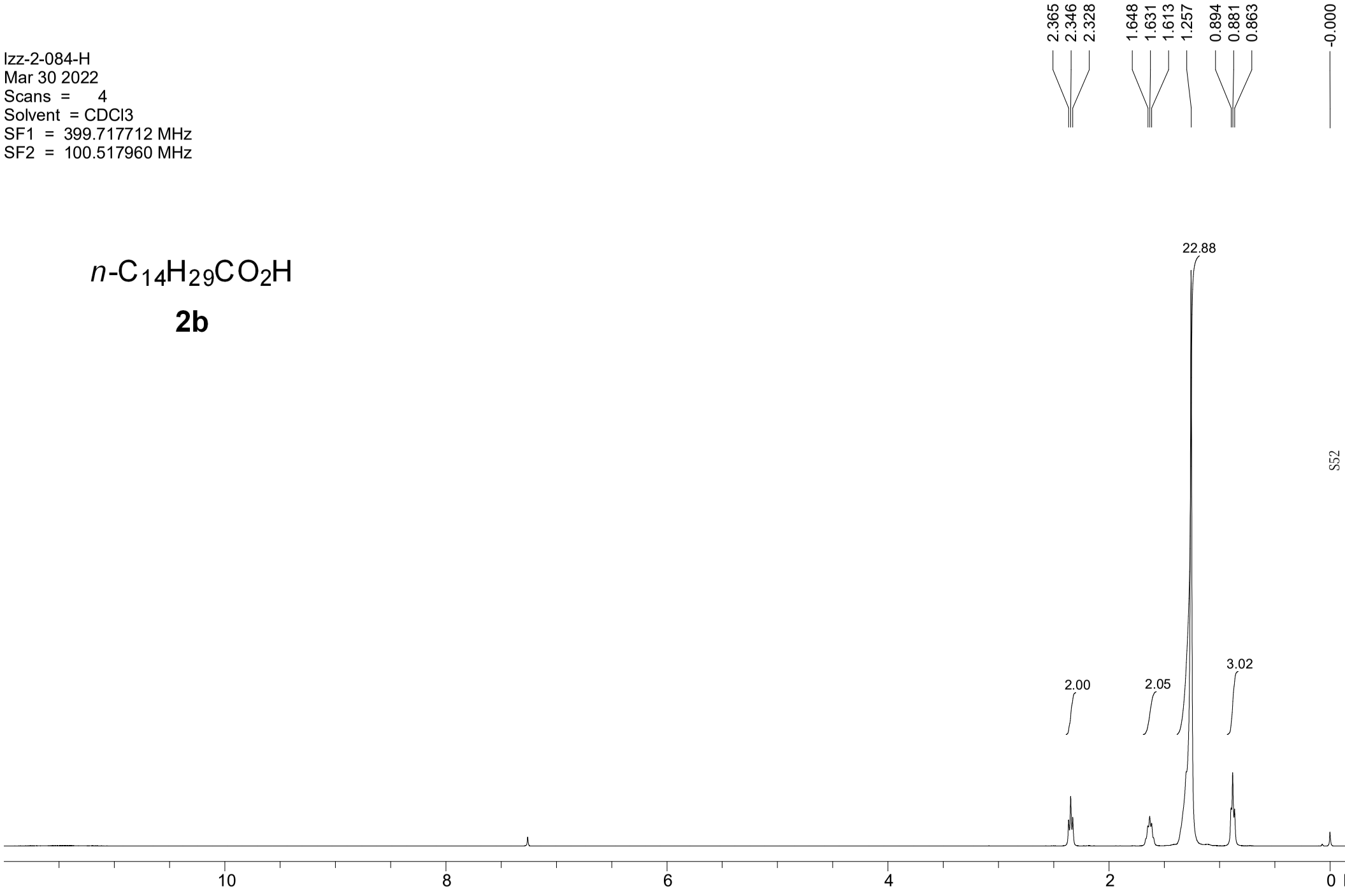
180.546
77.423
77.000
76.577
34.102
31.611
28.991
28.872
24.653
22.566
14.008

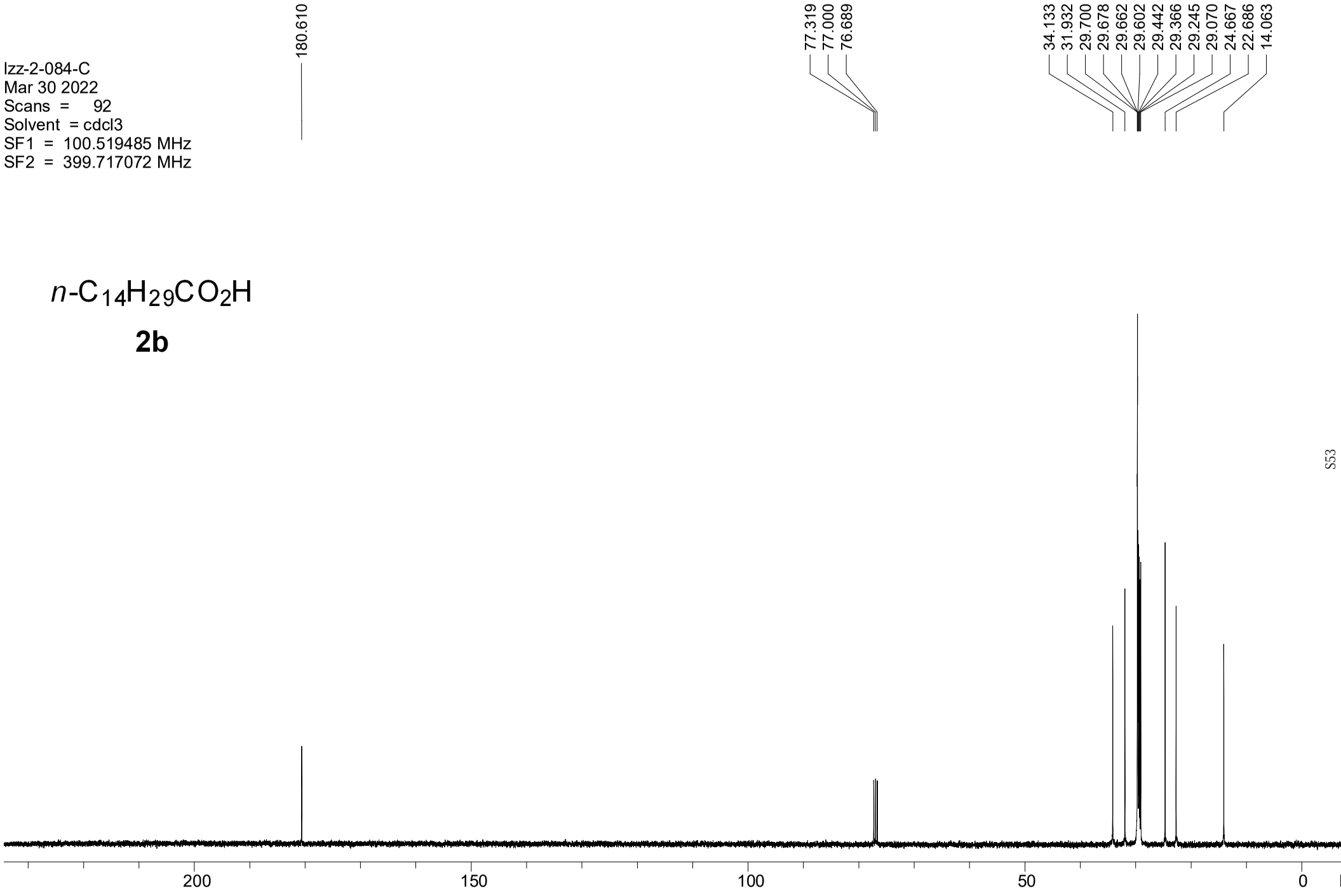
n-$C_7H_{15}CO_2H$

2a

*from **1ad** with 4-OH-TEMPO as co-catalyst*




lzz-2-084-H
Mar 30 2022
Scans = 4
Solvent = CDCl3
SF1 = 399.717712 MHz
SF2 = 100.517960 MHz
2.365
2.346
2.328
1.648
1.631
1.613
1.257
0.894
0.881
0.863
-0.000
n-C14H29CO2H
2b
22.88
3.02
2.00
2.05
S52
10
8
6
4
2
0 PPM


180.610
77.319
77.000
76.689
34.133
31.932
29.700
29.678
29.662
29.602
29.442
29.366
29.245
29.070
24.667
22.686
14.063
lzz-2-084-C
Mar 30 2022
Scans = 92
Solvent = cdcl3
SF1 = 100.519485 MHz
SF2 = 399.717072 MHz
n-C14H29CO2H
2b
S53
200
150
100
50
0
PPM

lzz-4-011-H
Feb 11 2023
Scans = 8
Solvent = CDCl3
SF1 = 399.717651 MHz
SF2 = 100.517960 MHz

7.318 7.298 7.281 7.231 7.220 7.201

2.980 2.961 2.941 2.708 2.688 2.669

-0.000

$Ph(CH_2)_2CO_2H$

2c

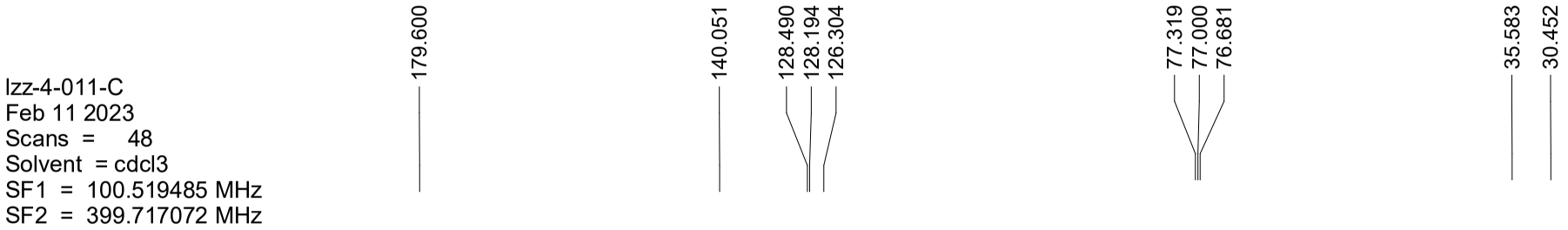
2.90

2.00

2.10 2.12

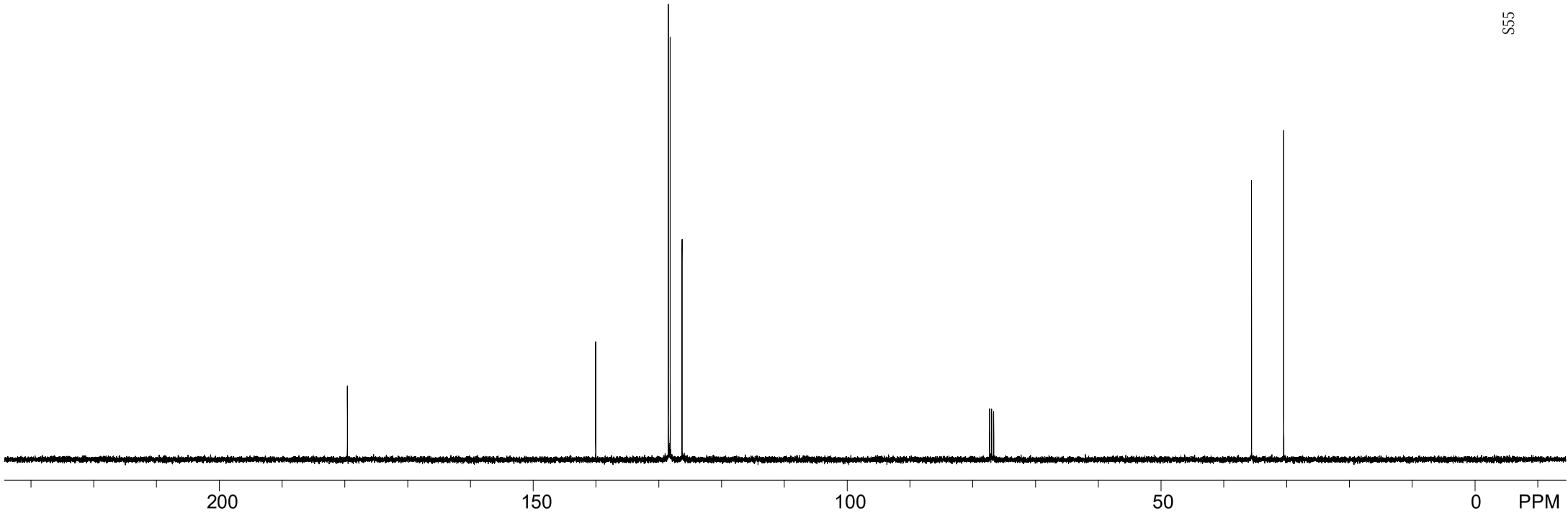
S54

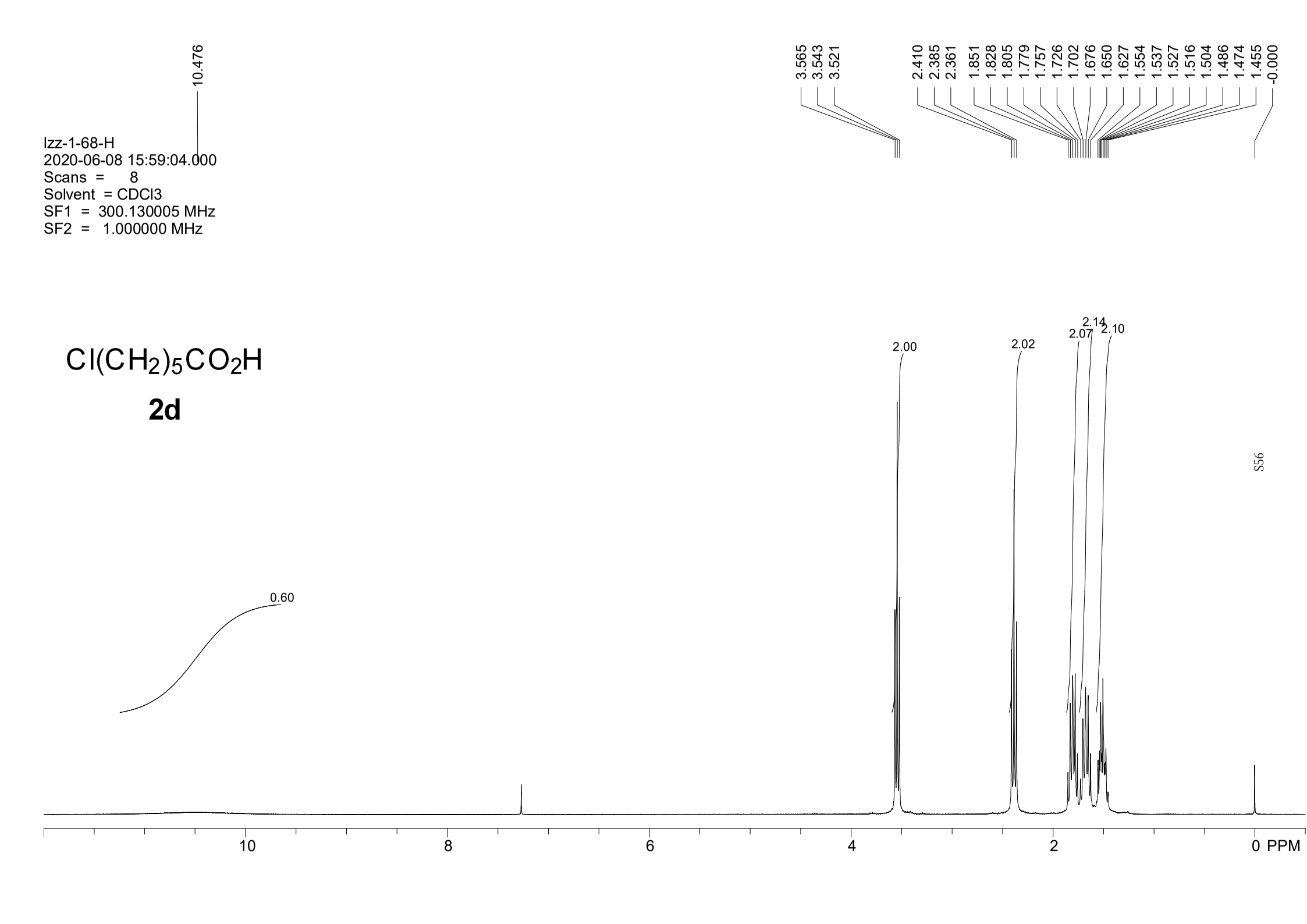
10 8 6 4 2 0 PPM

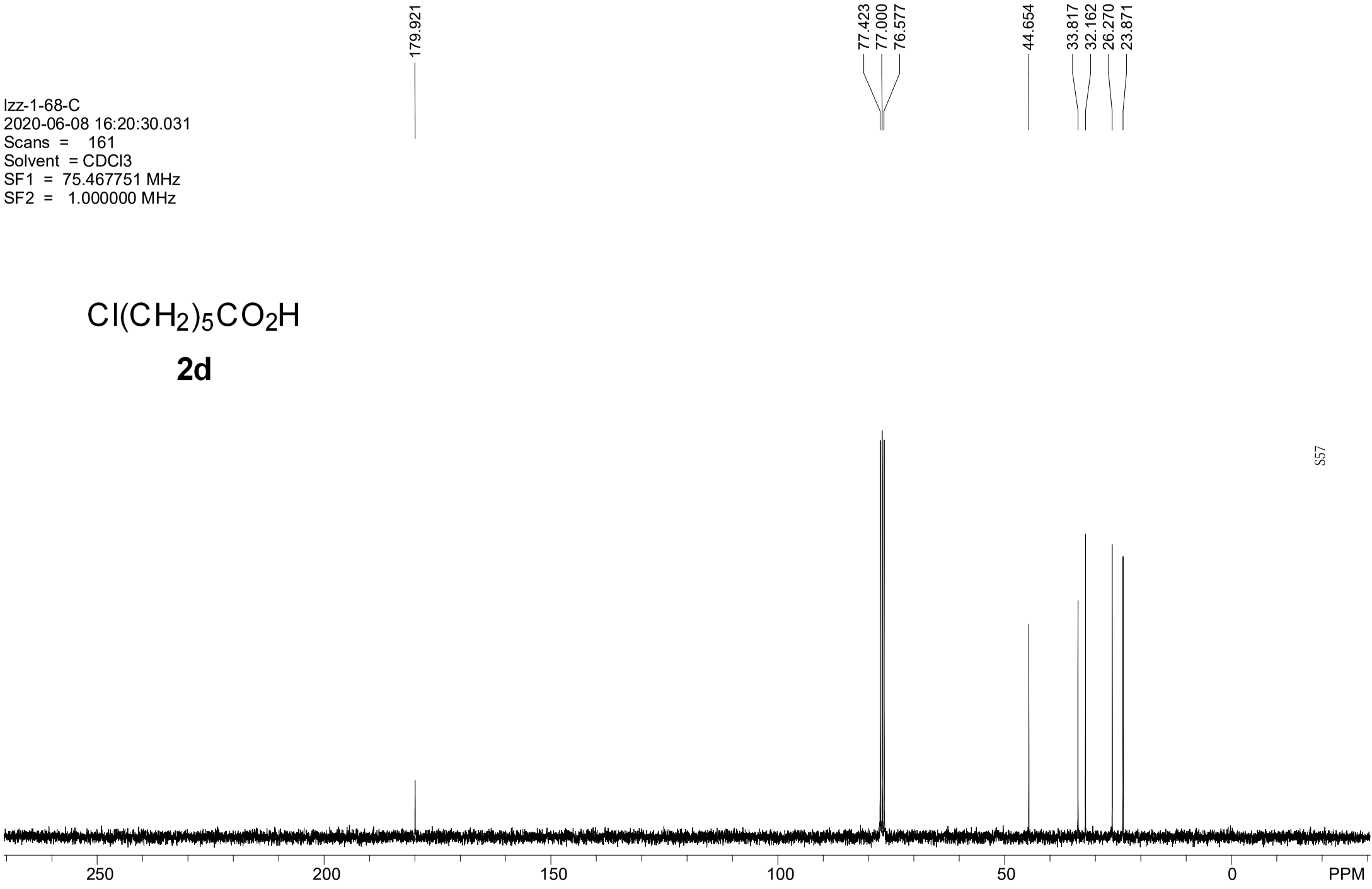


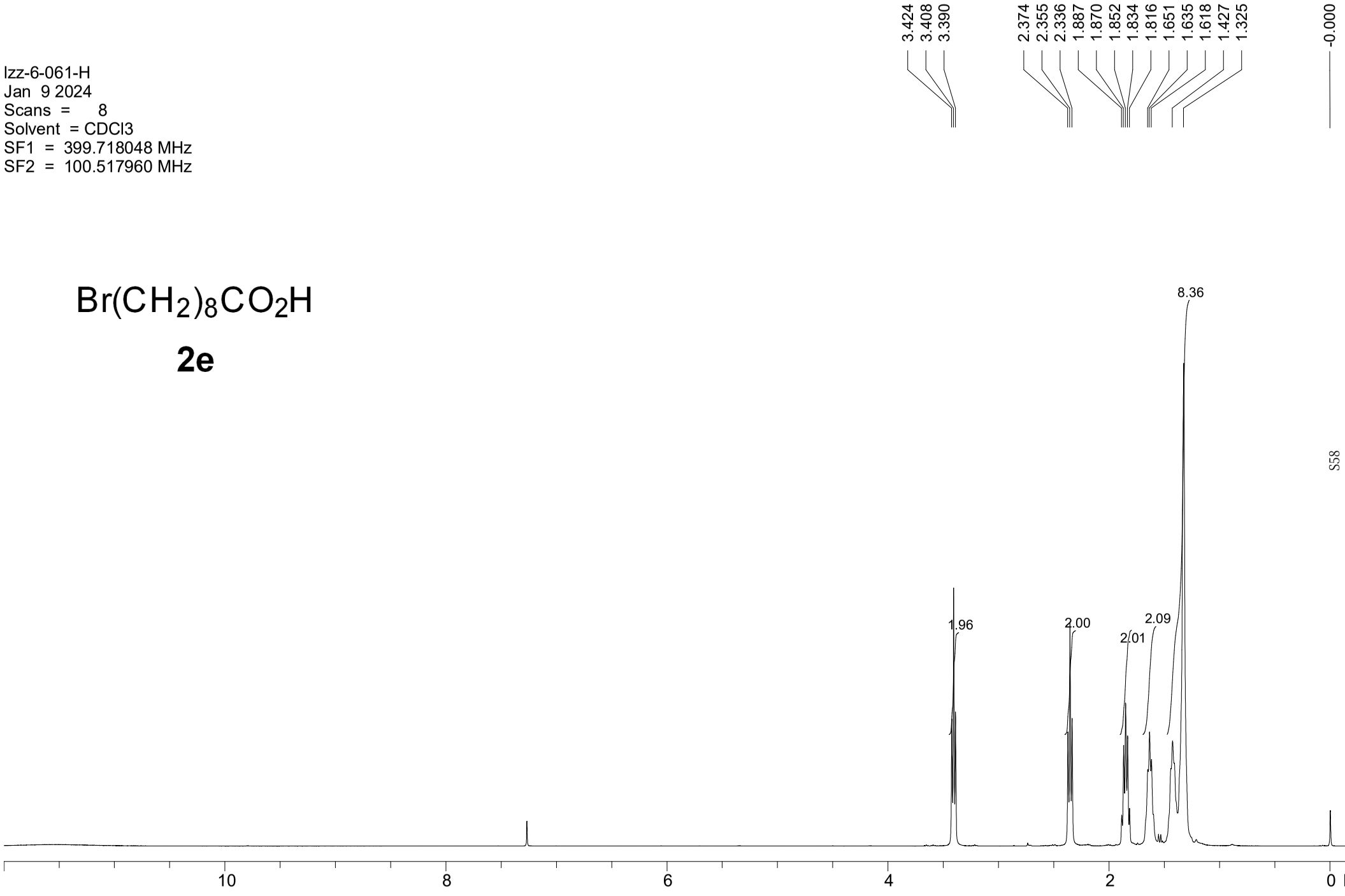
$Ph(CH_2)_2CO_2H$

2c




10.476
lzz-1-68-H
2020-06-08 15:59:04.000
Scans = 8
Solvent = CDCl3
SF1 = 300.130005 MHz
SF2 = 1.000000 MHz
3.565
3.543
3.521
2.410
2.385
2.361
1.851
1.828
1.805
1.779
1.757
1.726
1.702
1.676
1.650
1.627
1.554
1.537
1.527
1.516
1.504
1.486
1.474
1.455
-0.000
Cl(CH2)5CO2H
2d
2.00
2.02
2.07
2.14
2.10
0.60
S56
10
8
6
4
2
0 PPM


179.921
77.423
77.000
76.577
44.654
33.817
32.162
26.270
23.871
lzz-1-68-C
2020-06-08 16:20:30.031
Scans = 161
Solvent = CDCl3
SF1 = 75.467751 MHz
SF2 = 1.000000 MHz
Cl(CH2)5CO2H
2d
S57
250
200
150
100
50
0
PPM

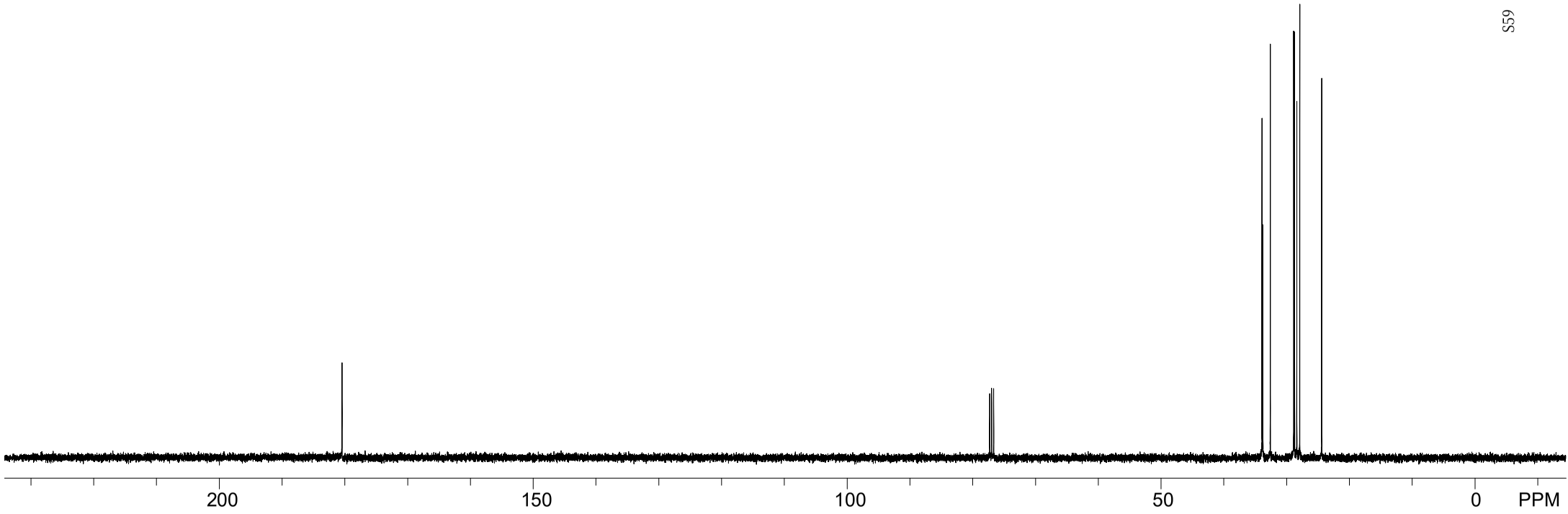

lzz-6-061-H
Jan 9 2024
Scans = 8
Solvent = CDCl3
SF1 = 399.718048 MHz
SF2 = 100.517960 MHz
3.424
3.408
3.390
2.374
2.355
2.336
1.887
1.870
1.852
1.834
1.816
1.651
1.635
1.618
1.427
1.325
-0.000
Br(CH2)8CO2H
2e
S58
8.36
1.96
2.00
2.01
2.09
10
8
6
4
2
0 PPM

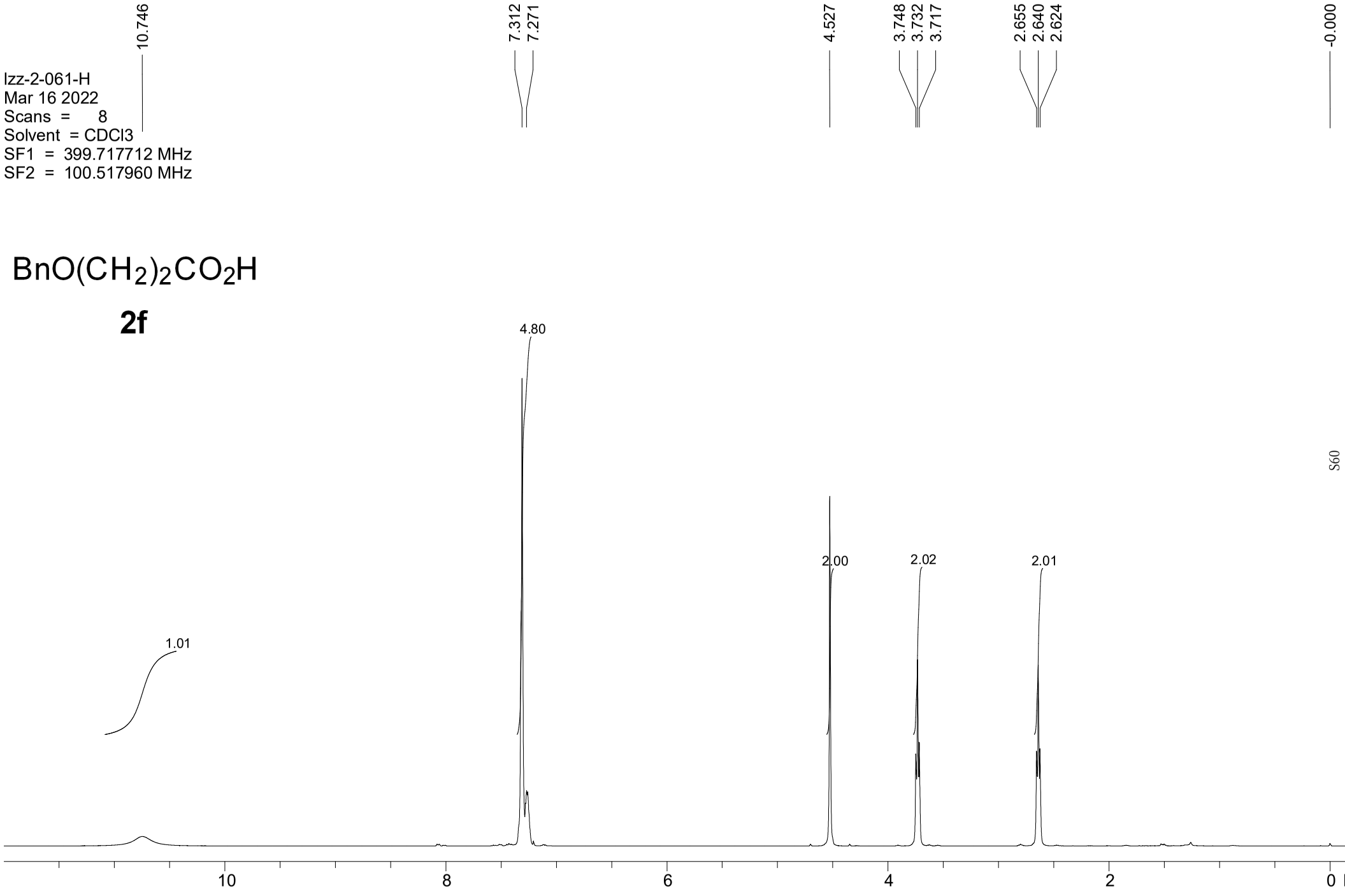
lzz-6-061-C
Jan 9 2024
Scans = 40
Solvent = cdcl3
SF1 = 100.519485 MHz
SF2 = 399.717072 MHz

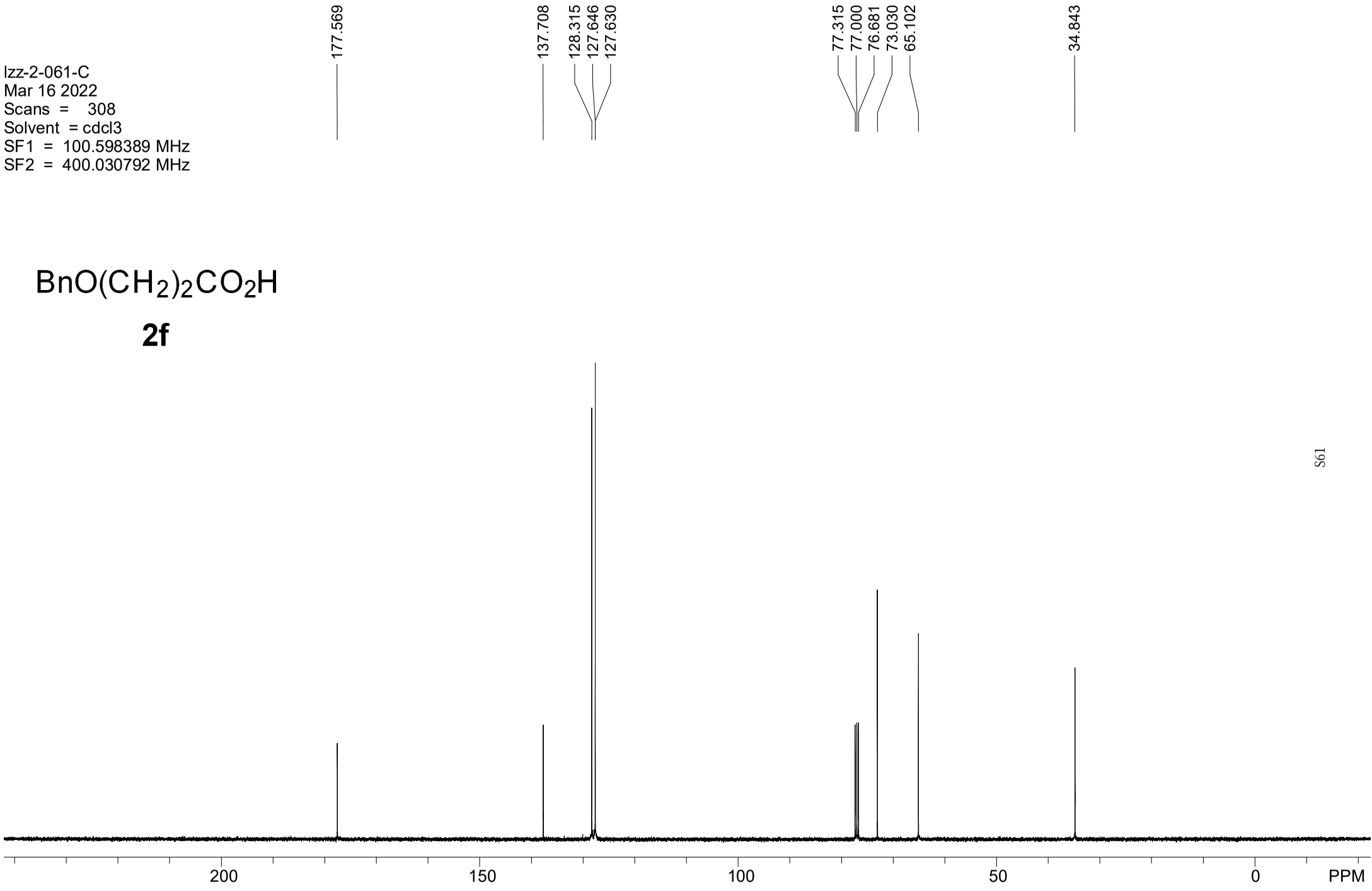
180.451
77.319
77.000
76.681
33.921
33.777
32.585
28.858
28.729
28.364
27.894
24.402

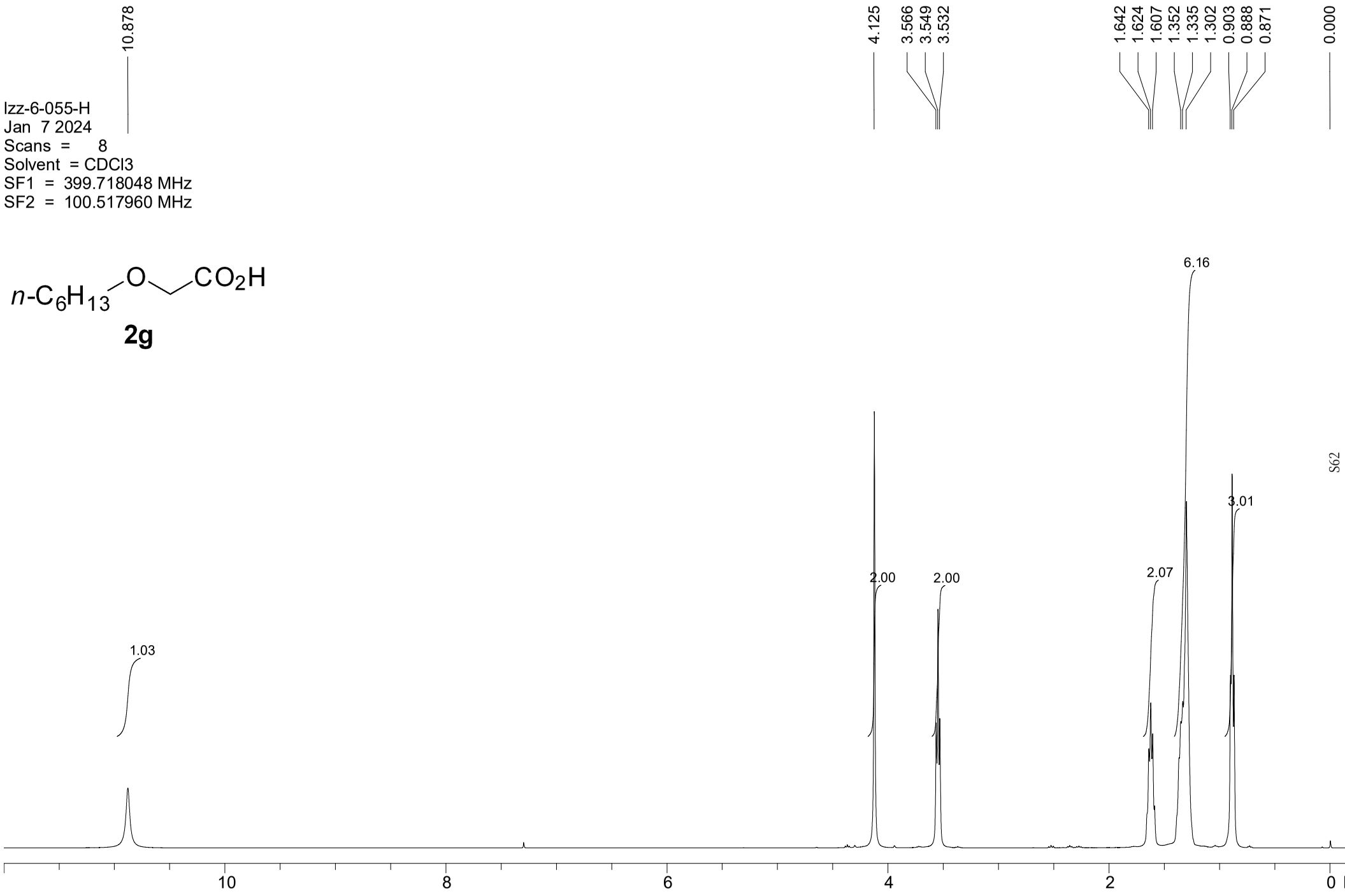
$Br(CH_2)_8CO_2H$

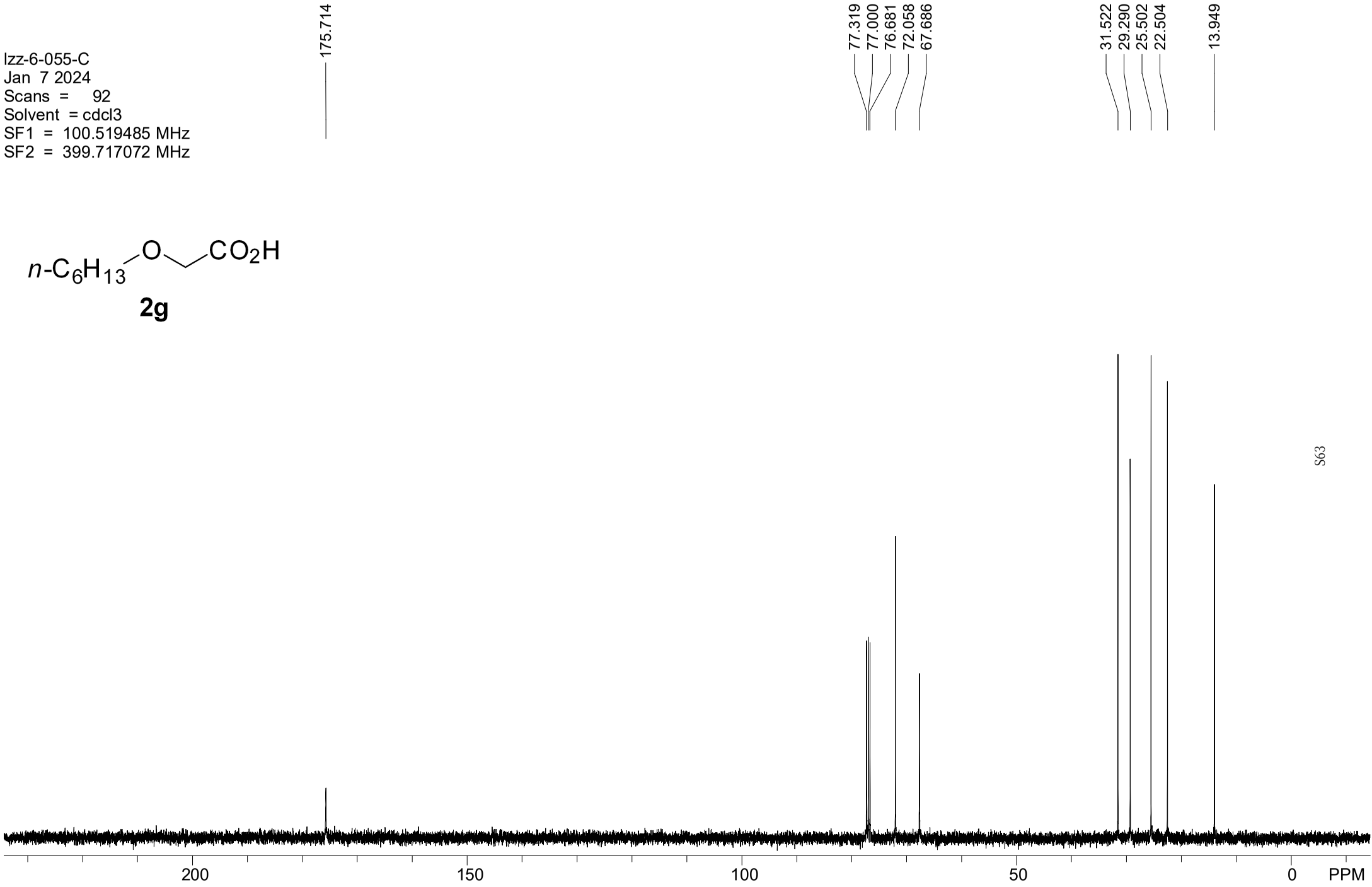
2e




10.746
7.312
7.271
4.527
3.748
3.732
3.717
2.655
2.640
2.624
-0.000
lzz-2-061-H
Mar 16 2022
Scans = 8
Solvent = CDCl3
SF1 = 399.717712 MHz
SF2 = 100.517960 MHz
BnO(CH2)2CO2H
2f
4.80
2.00
2.02
2.01
1.01
S60
10
8
6
4
2
0 PPM


177.569
137.708
128.315
127.646
127.630
77.315
77.000
76.681
73.030
65.102
34.843
lzz-2-061-C
Mar 16 2022
Scans = 308
Solvent = cdcl3
SF1 = 100.598389 MHz
SF2 = 400.030792 MHz
BnO(CH2)2CO2H
2f
200
150
100
50
0
PPM


10.878
4.125
3.566
3.549
3.532
1.642
1.624
1.607
1.352
1.335
1.302
0.903
0.888
0.871
0.000
Izz-6-055-H
Jan 7 2024
Scans = 8
Solvent = CDCl3
SF1 = 399.718048 MHz
SF2 = 100.517960 MHz
n-C6H13
O
CO2H
2g
6.16
3.01
2.00
2.00
2.07
1.03
S62
10
8
6
4
2
0 PPM

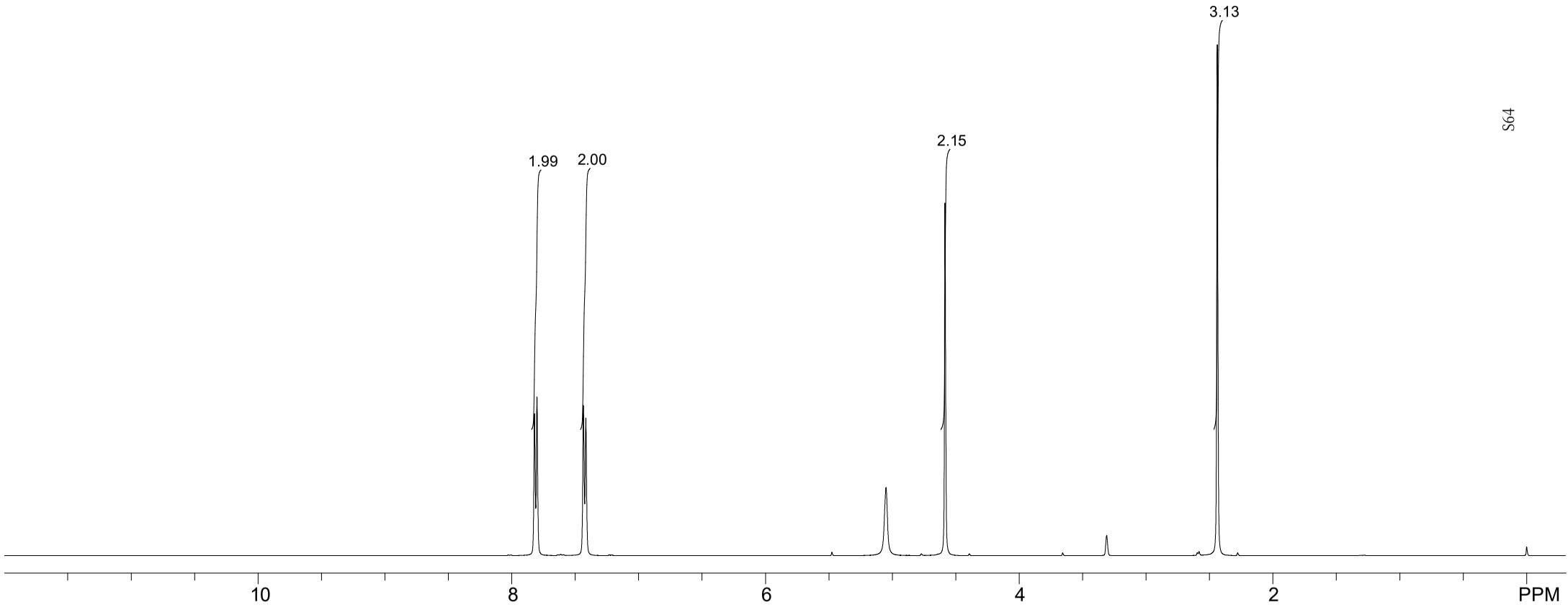

lzz-6-055-C
Jan 7 2024
Scans = 92
Solvent = cdcl3
SF1 = 100.519485 MHz
SF2 = 399.717072 MHz
175.714
77.319
77.000
76.681
72.058
67.686
31.522
29.290
25.502
22.504
13.949
n-C6H13
O
CO2H
2g
S63
200
150
100
50
0
PPM

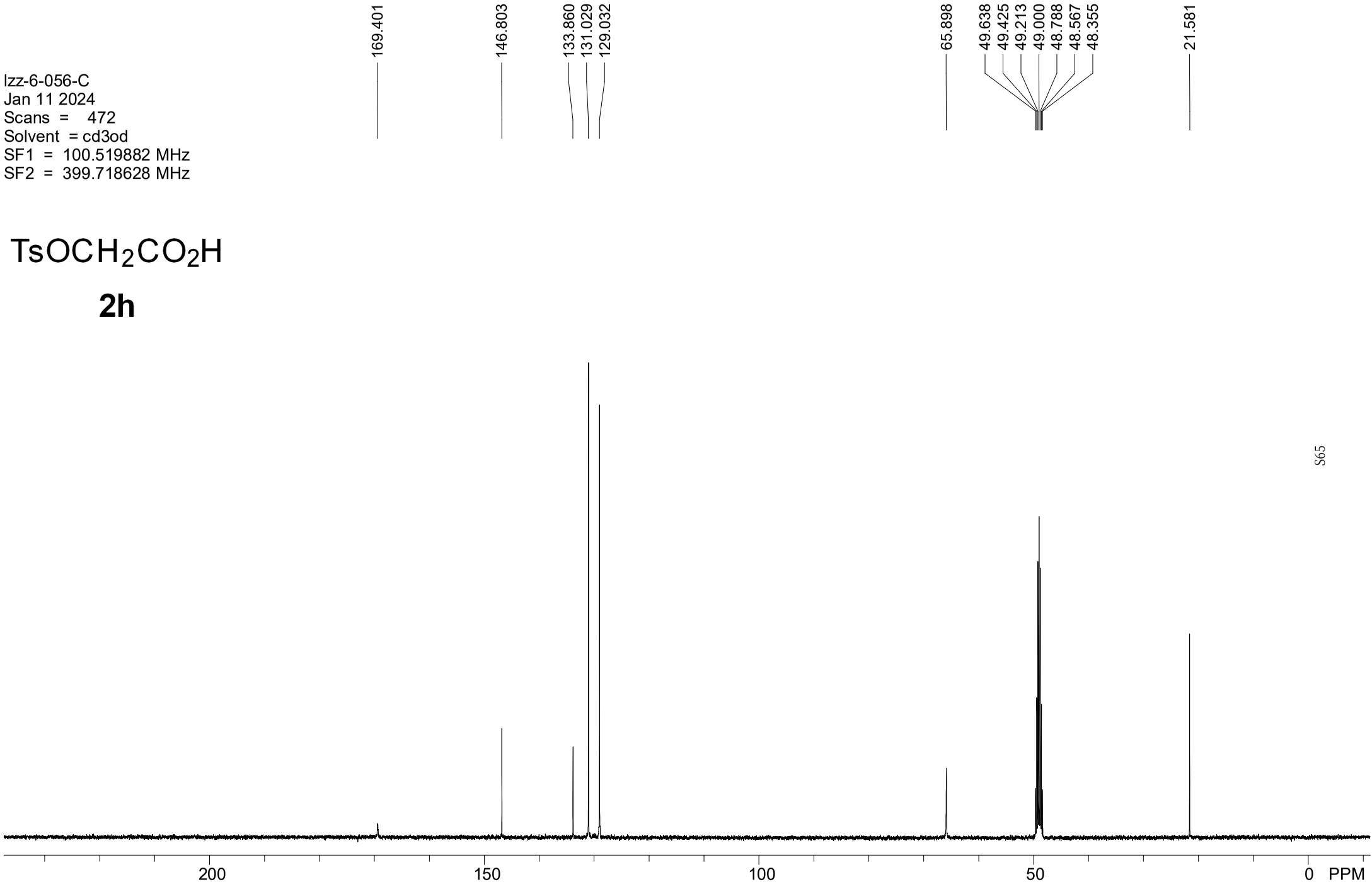
lzz-6-056-H
Jan 8 2024
Scans = 8
Solvent = cd3od
SF1 = 399.719025 MHz
SF2 = 100.518356 MHz

7.822
7.801
7.436
7.416
4.585
3.310
2.437

$TsOCH_2CO_2H$

2h



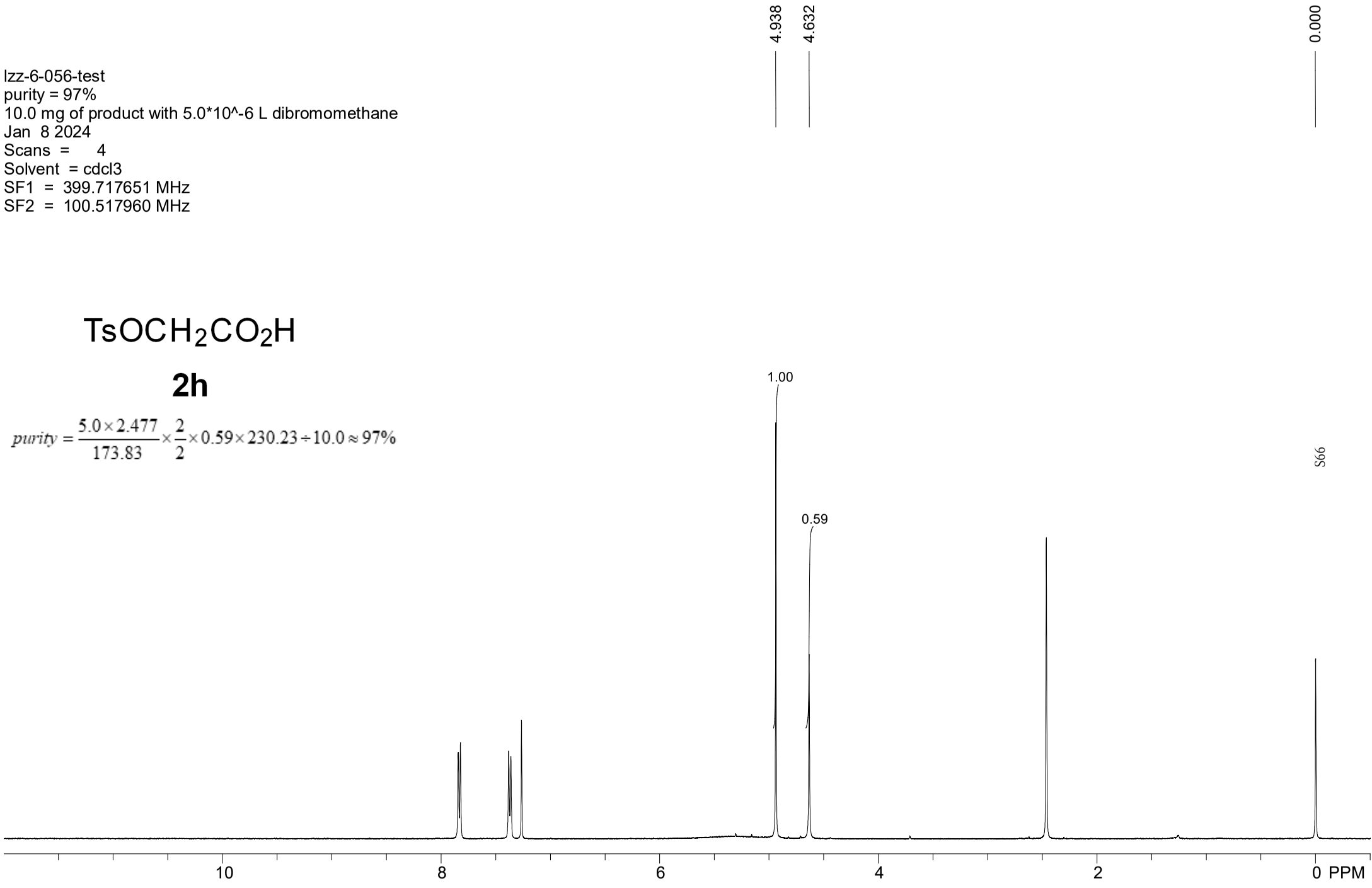

169.401
146.803
133.860
131.029
129.032
65.898
49.638
49.425
49.213
49.000
48.788
48.567
48.355
21.581
Izz-6-056-C
Jan 11 2024
Scans = 472
Solvent = cd3od
SF1 = 100.519882 MHz
SF2 = 399.718628 MHz
TsOCH2CO2H
2h
S65
200
150
100
50
0 PPM

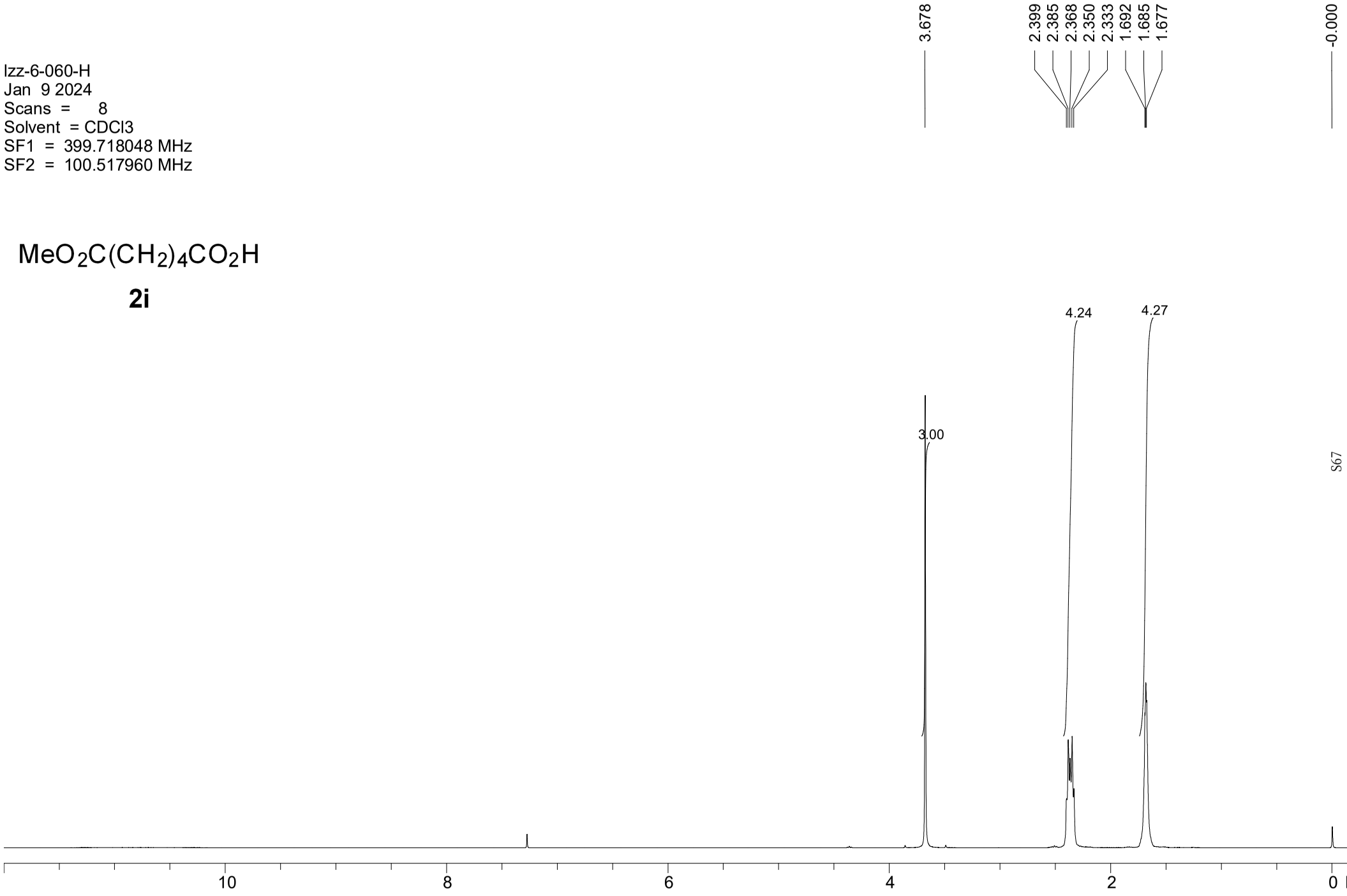
lzz-6-056-test
purity = 97%
10.0 mg of product with 5.0*10^-6 L dibromomethane
Jan 8 2024
Scans = 4
Solvent = cdcl3
SF1 = 399.717651 MHz
SF2 = 100.517960 MHz

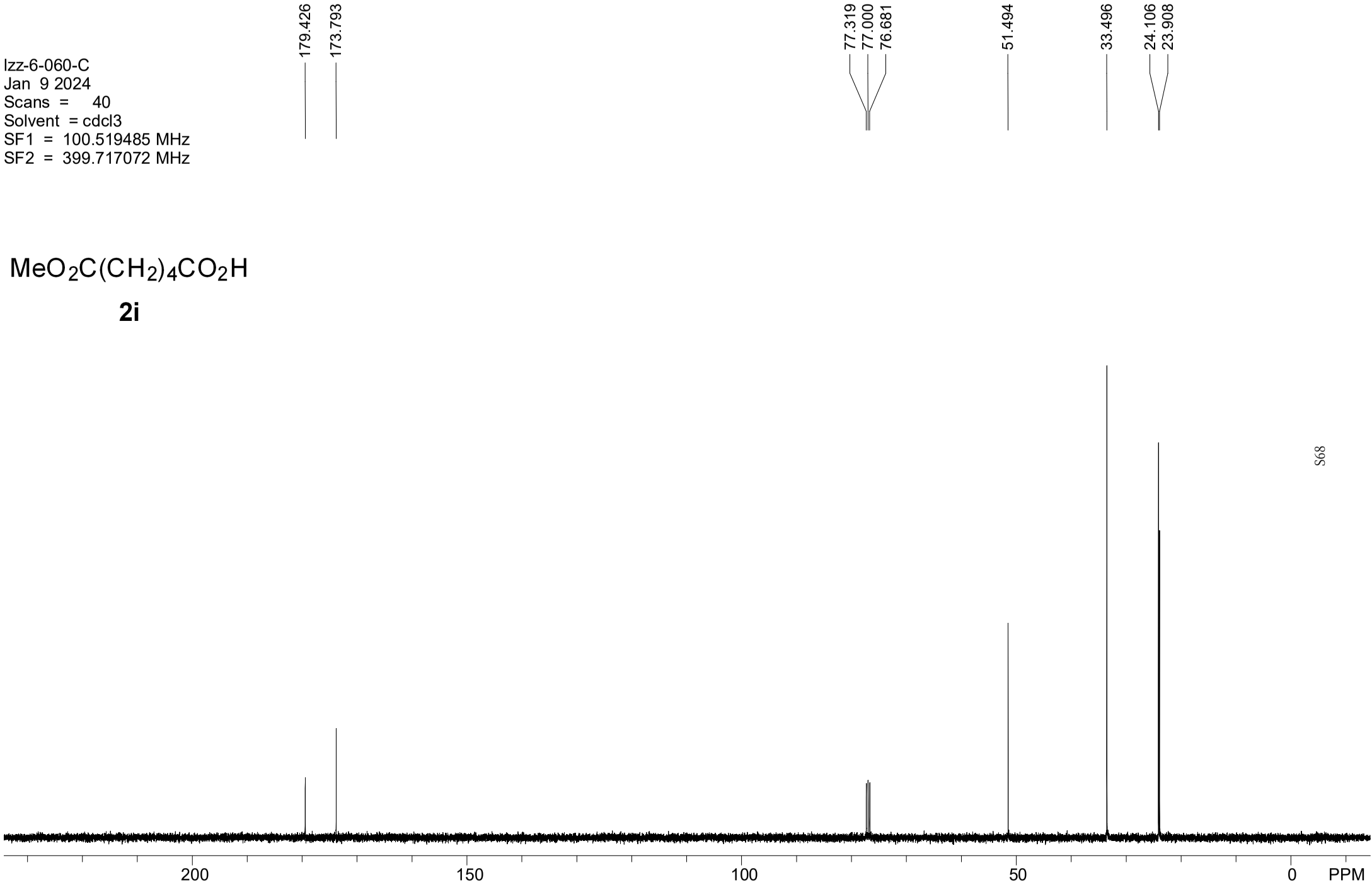
$TsOCH_2CO_2H$

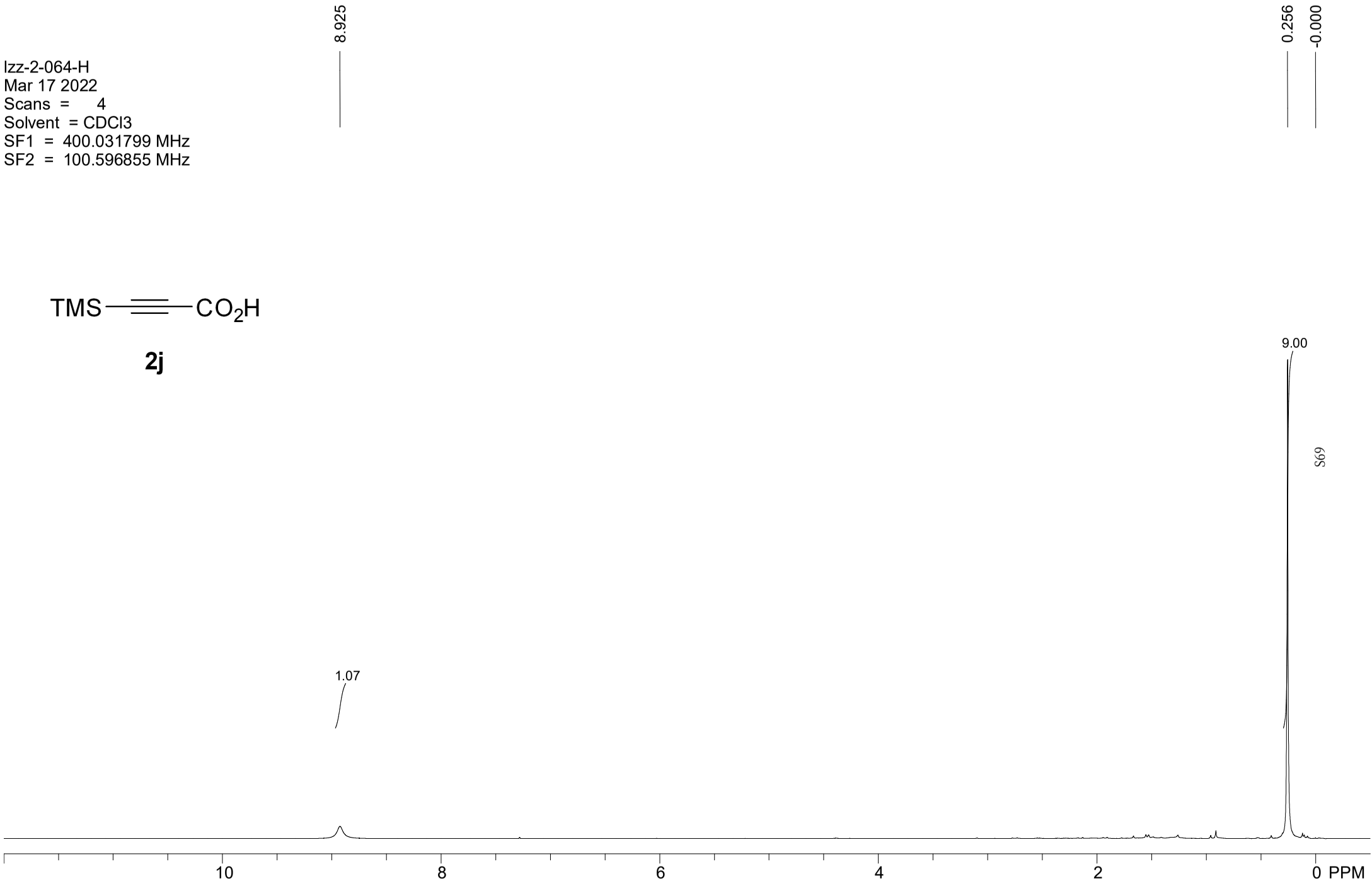
2h

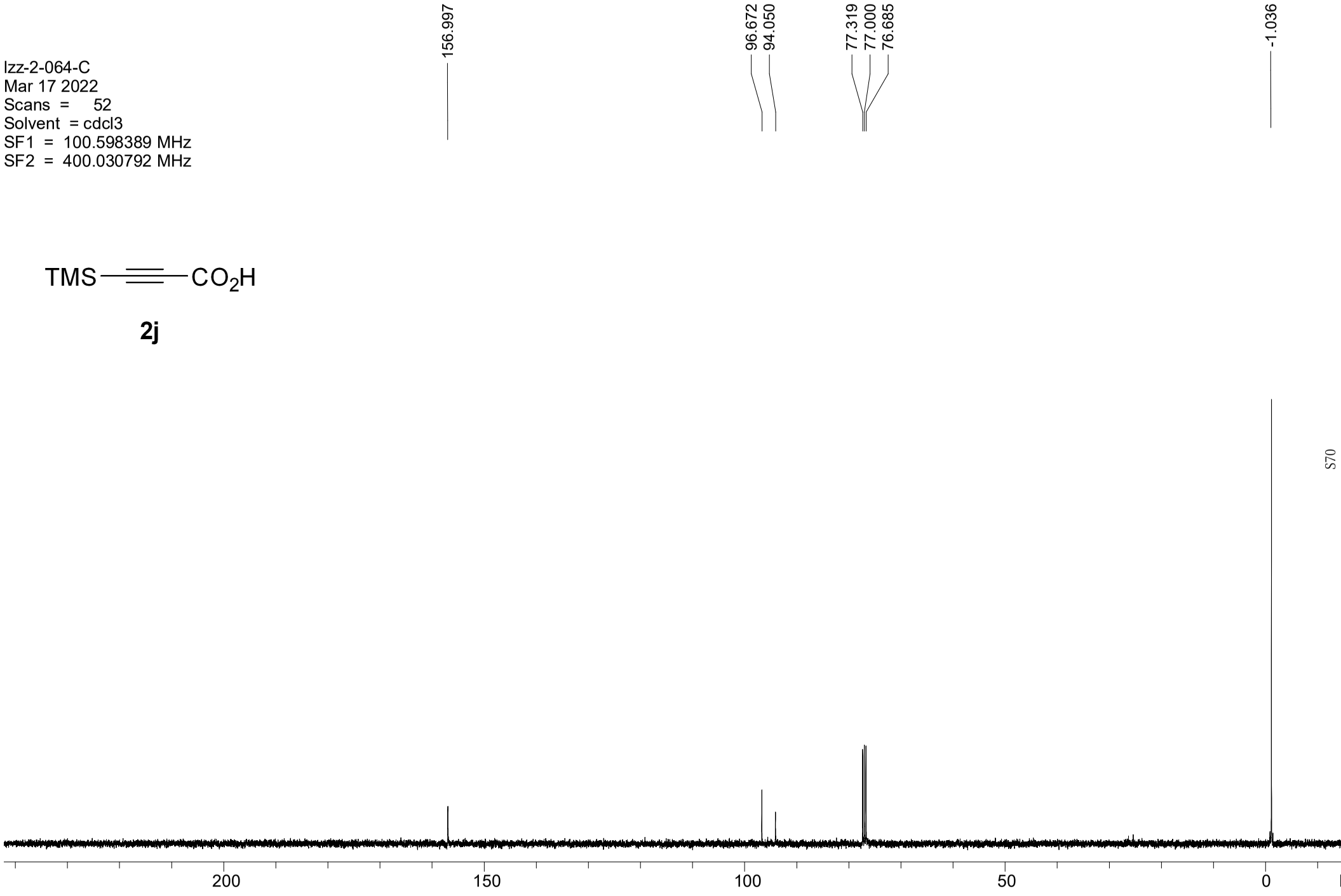
$$purity = \frac{5.0 \times 2.477}{173.83} \times \frac{2}{2} \times 0.59 \times 230.23 \div 10.0 \approx 97\%$$

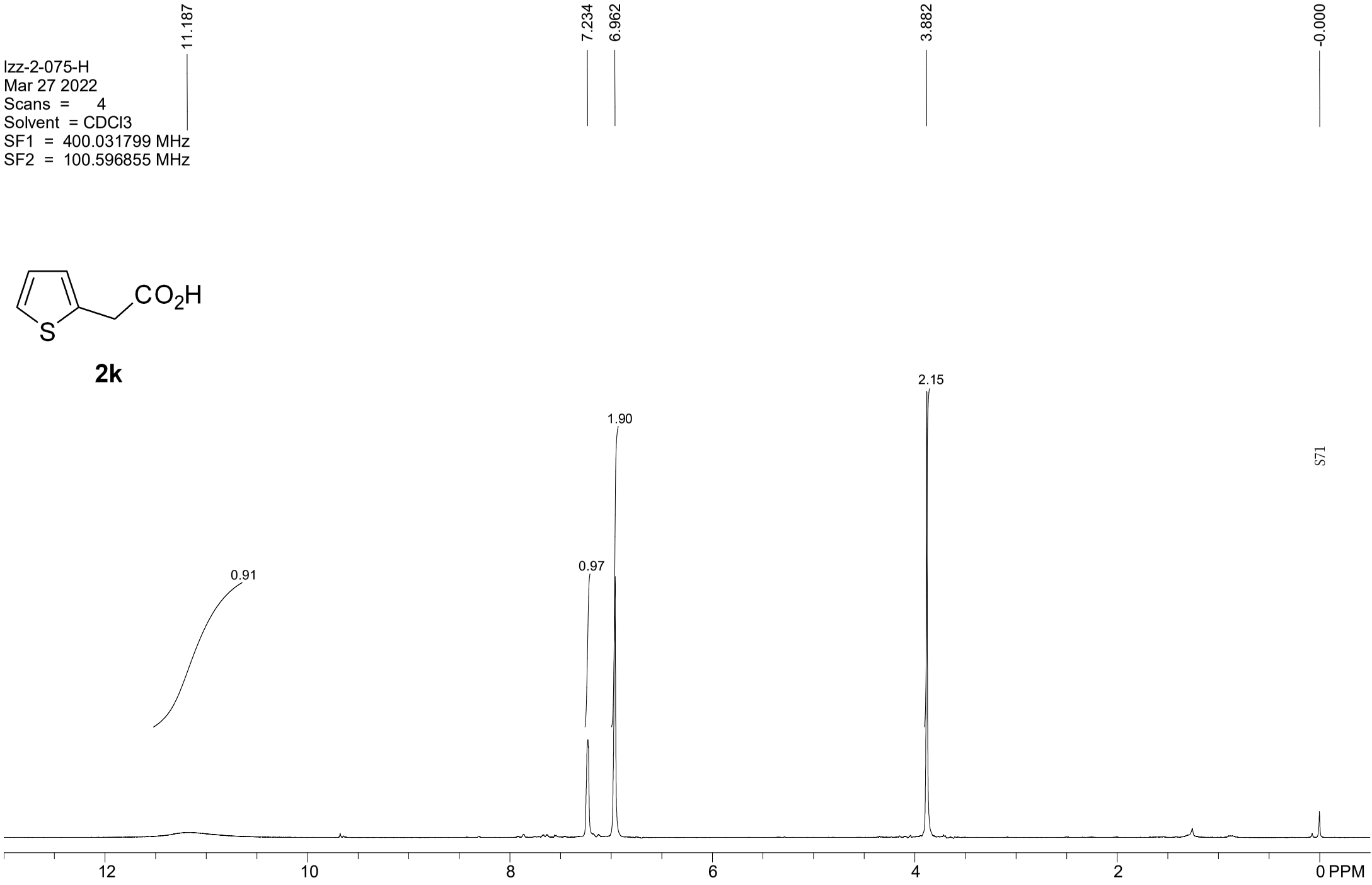



3.678
2.399
2.385
2.368
2.350
2.333
1.692
1.685
1.677
-0.000
lzz-6-060-H
Jan 9 2024
Scans = 8
Solvent = CDCl3
SF1 = 399.718048 MHz
SF2 = 100.517960 MHz
MeO2C(CH2)4CO2H
2i
4.24
4.27
3.00
S67
10
8
6
4
2
0 PPM


179.426
173.793
77.319
77.000
76.681
51.494
33.496
24.106
23.908
lzz-6-060-C
Jan 9 2024
Scans = 40
Solvent = cdcl3
SF1 = 100.519485 MHz
SF2 = 399.717072 MHz
MeO2C(CH2)4CO2H
2i
S68
200
150
100
50
0
PPM

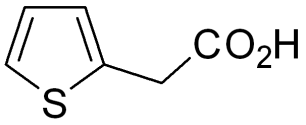

8.925
0.256
-0.000
lzz-2-064-H
Mar 17 2022
Scans = 4
Solvent = CDCl3
SF1 = 400.031799 MHz
SF2 = 100.596855 MHz
TMS
CO2H
2j
9.00
1.07
10
8
6
4
2
0 PPM


156.997
96.672
94.050
77.319
77.000
76.685
-1.036
lzz-2-064-C
Mar 17 2022
Scans = 52
Solvent = cdcl3
SF1 = 100.598389 MHz
SF2 = 400.030792 MHz
TMS
CO2H
2j
S70
200
150
100
50
0
PPM

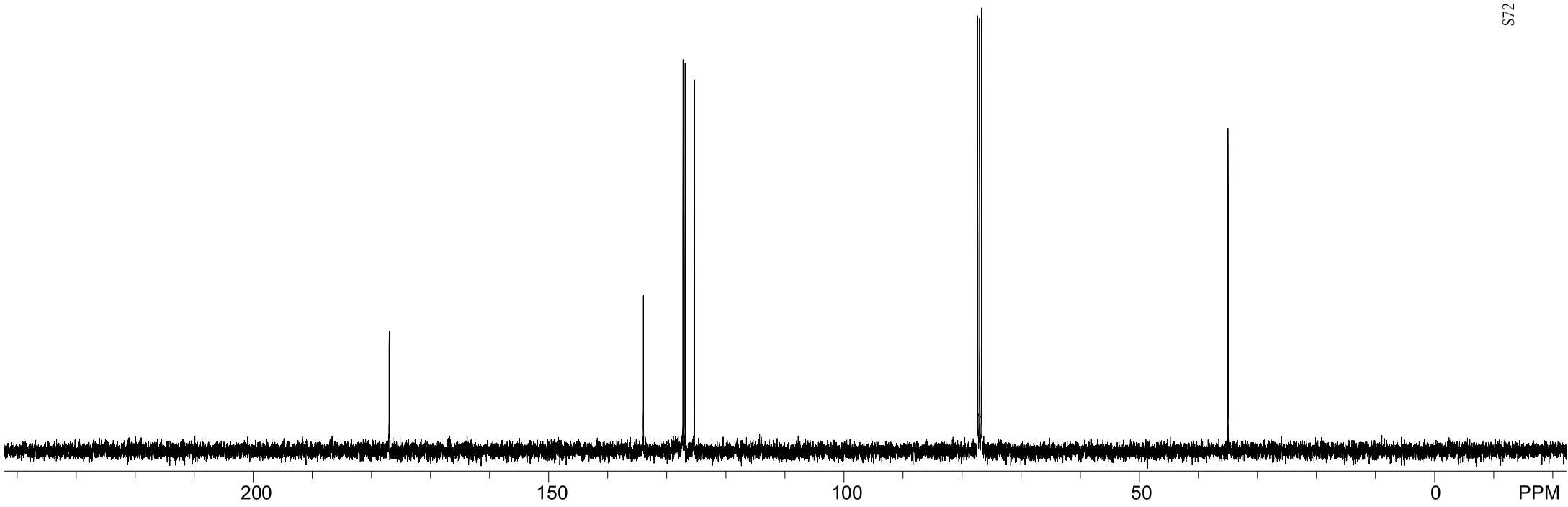

11.187
7.234
6.962
3.882
-0.000
lzz-2-075-H
Mar 27 2022
Scans = 4
Solvent = CDCl3
SF1 = 400.031799 MHz
SF2 = 100.596855 MHz
CO2H
S
2k
2.15
1.90
0.97
0.91
S71
12
10
8
6
4
2
0 PPM

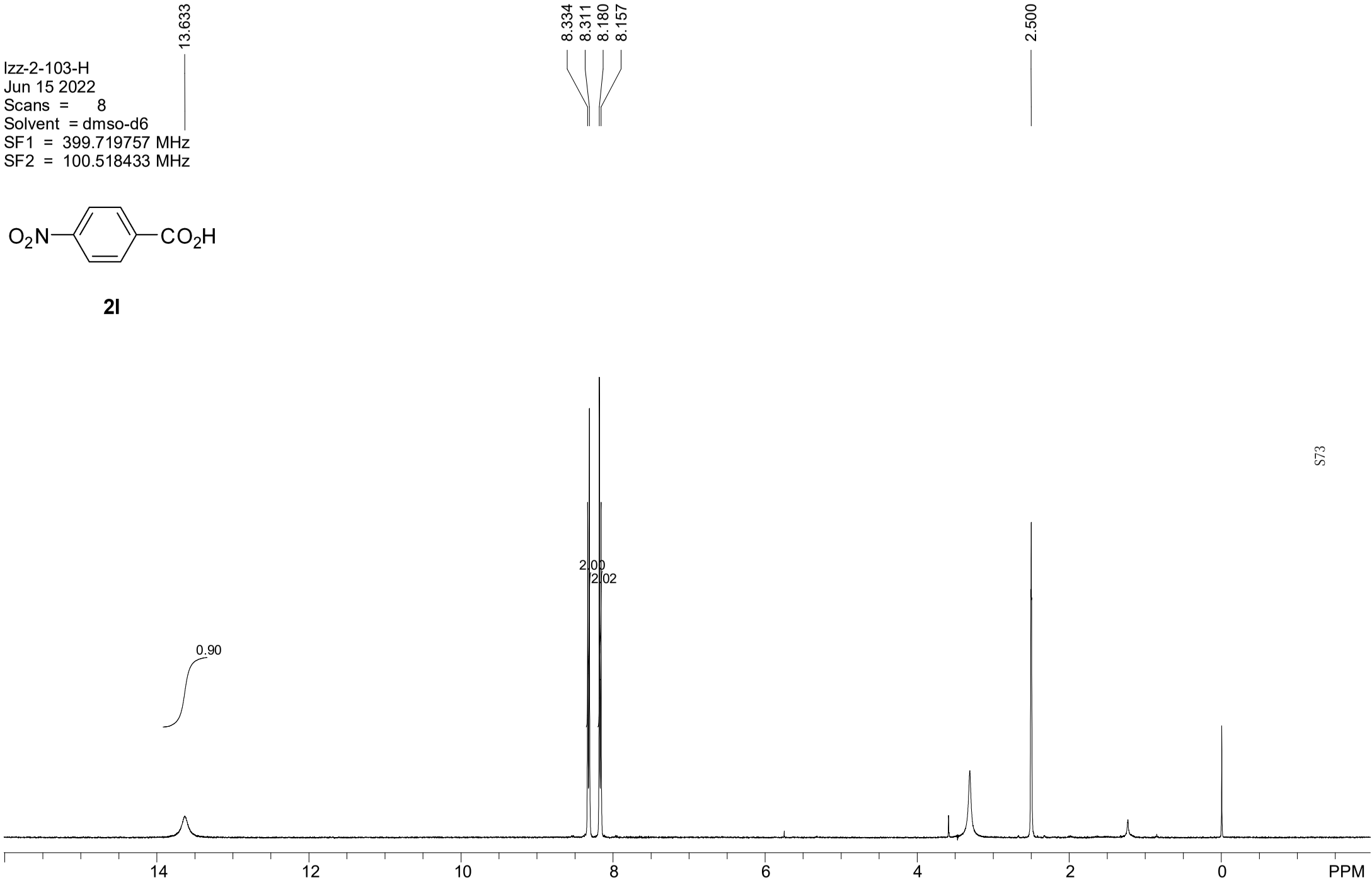
lzz-2-075-C
Mar 28 2022
Scans = 92
Solvent = cdcl3
SF1 = 100.598389 MHz
SF2 = 400.030792 MHz

177.005
133.952
127.246
126.891
125.306
77.315
77.000
76.681
35.012

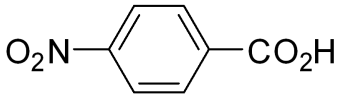


2k



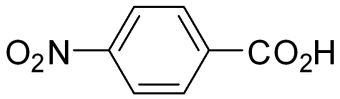

13.633
8.334
8.311
8.180
8.157
2.500
lzz-2-103-H
Jun 15 2022
Scans = 8
Solvent = dmso-d6
SF1 = 399.719757 MHz
SF2 = 100.518433 MHz
O2N
CO2H
2I
S73
2.00
2.02
0.90
14
12
10
8
6
4
2
0
PPM

lzz-2-103-C
d6-DMSO
Tue Jun 14 07:51:37 2022
Scans = 480
SF1 = 100.617798 MHz
SF2 = 1.000000 MHz



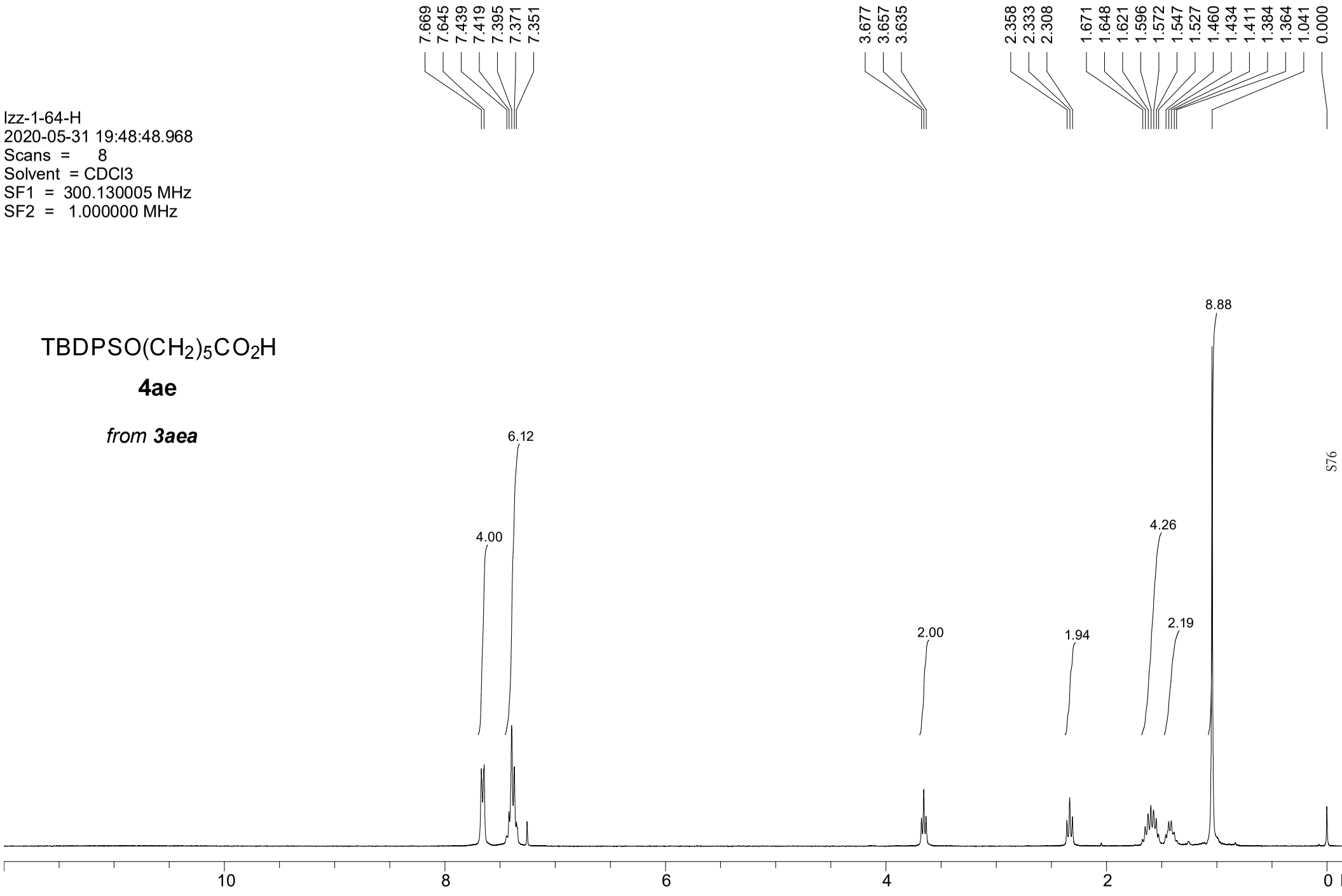
2l

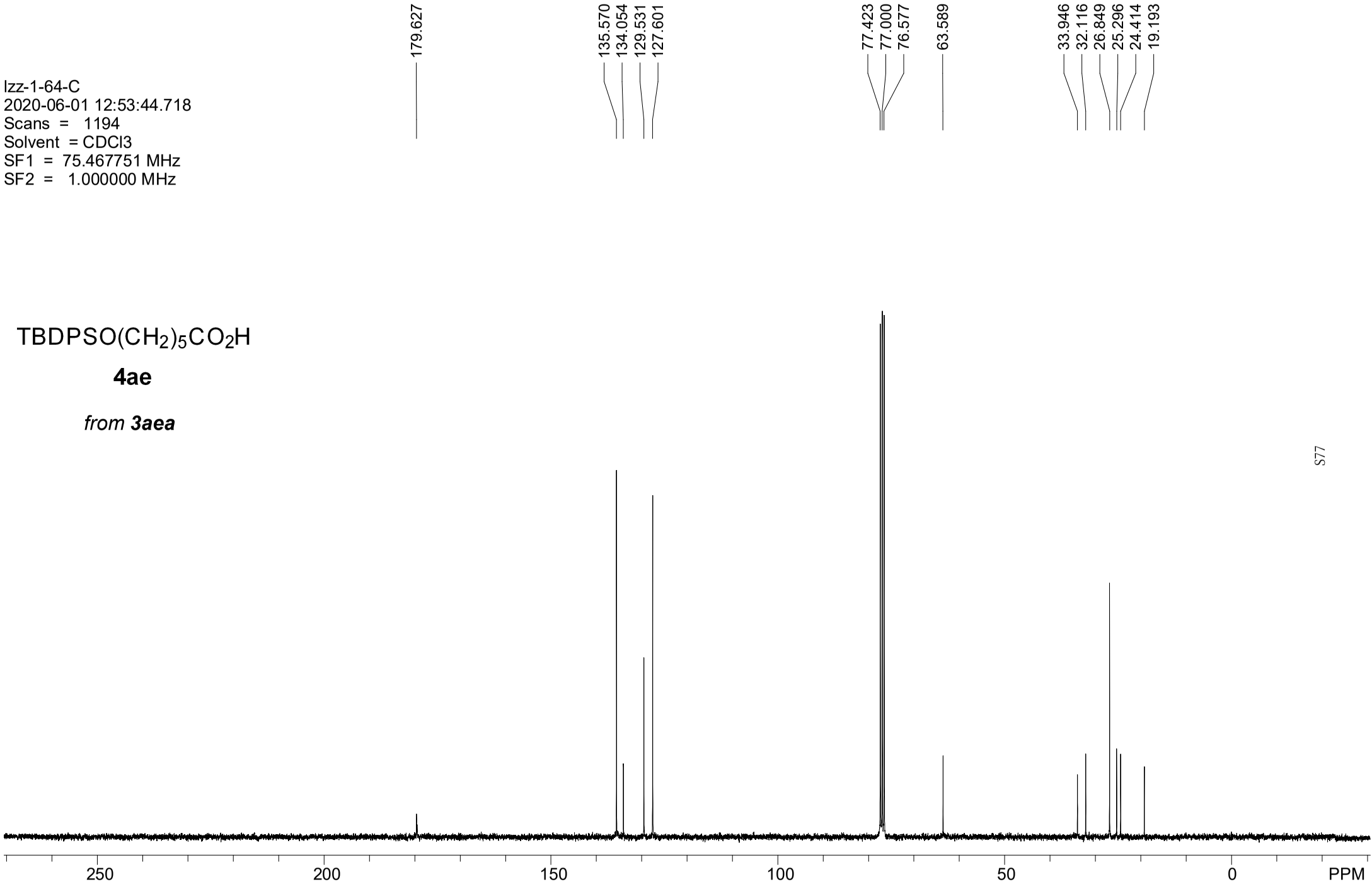
lzz-2-103-test
purity = 97%
12.6 mg of product with 5.0*10^-6 L dibromomethane
DMSO
Tue Jun 21 22:46:23 2022
Scans = 16
SF1 = 400.102600 MHz
SF2 = 1.000000 MHz

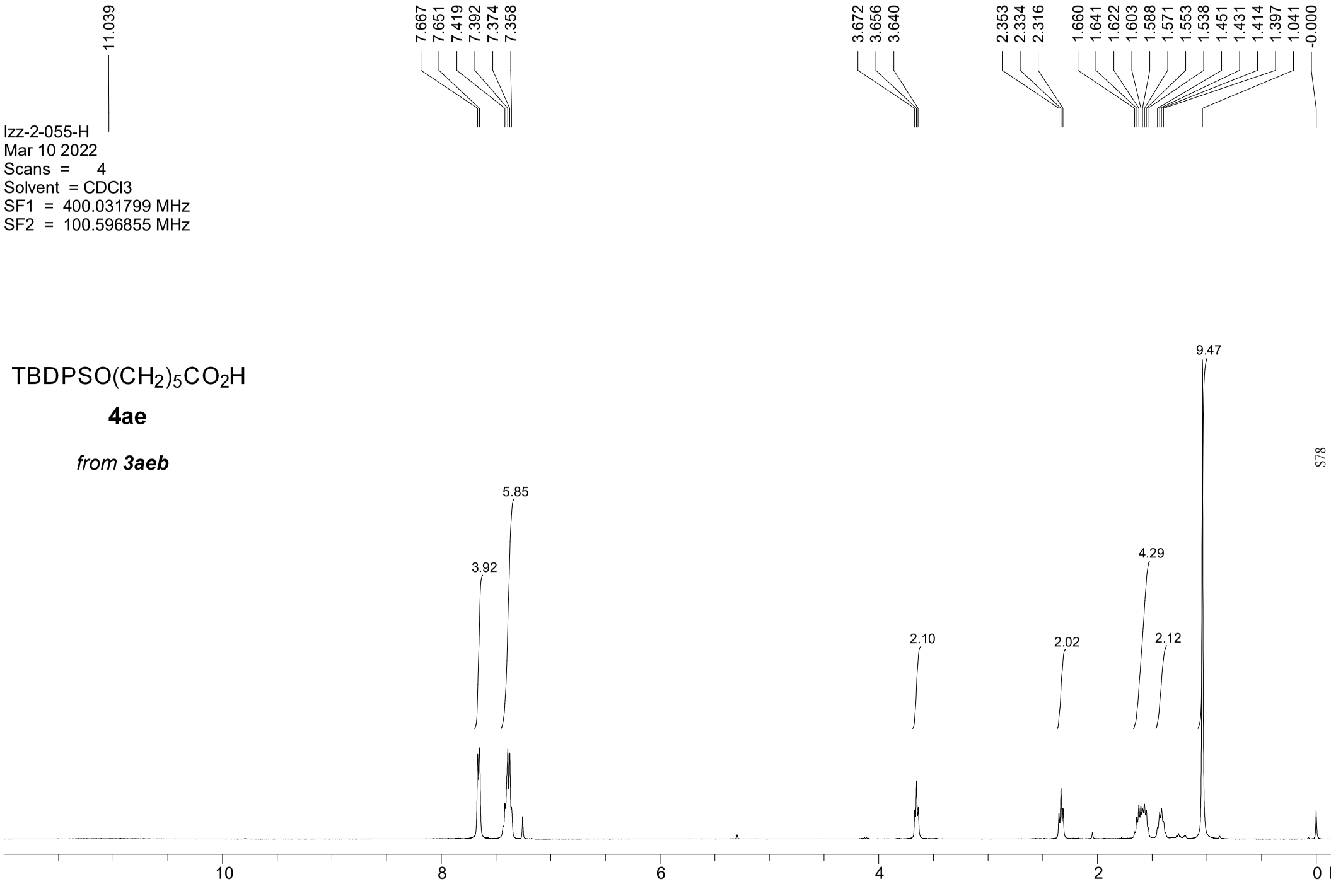


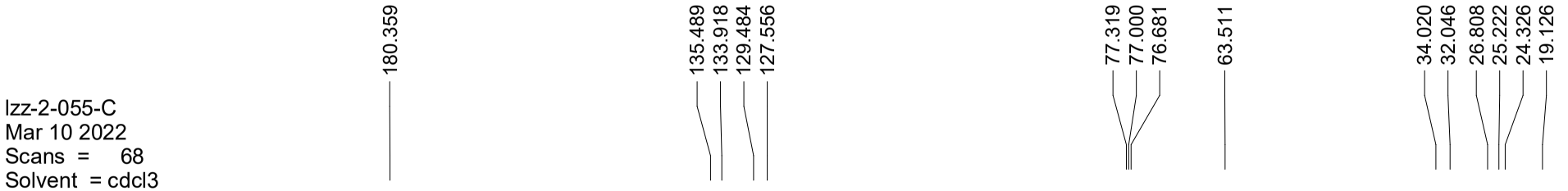
2l

$$purity = \frac{5.0 \times 2.477}{173.83} \times \frac{2}{2} \times 1.03 \times 167.1 \div 12.6 \approx 97\%$$


7.669
7.645
7.439
7.419
7.395
7.371
7.351
3.677
3.657
3.635
2.358
2.333
2.308
1.671
1.648
1.621
1.596
1.572
1.547
1.527
1.460
1.434
1.411
1.384
1.364
1.041
0.000
lzz-1-64-H
2020-05-31 19:48:48.968
Scans = 8
Solvent = CDCl3
SF1 = 300.130005 MHz
SF2 = 1.000000 MHz
TBDPSO(CH2)5CO2H
4ae
from 3aea
8.88
6.12
4.26
4.00
2.19
2.00
1.94
10
8
6
4
2
0 PPM


179.627
135.570
134.054
129.531
127.601
77.423
77.000
76.577
63.589
33.946
32.116
26.849
25.296
24.414
19.193
lzz-1-64-C
2020-06-01 12:53:44.718
Scans = 1194
Solvent = CDCl3
SF1 = 75.467751 MHz
SF2 = 1.000000 MHz
TBDPSO(CH2)5CO2H
4ae
from 3aea
S77
250
200
150
100
50
0
PPM


11.039
7.667
7.651
7.419
7.392
7.374
7.358
3.672
3.656
3.640
2.353
2.334
2.316
1.660
1.641
1.622
1.603
1.588
1.571
1.553
1.538
1.451
1.431
1.414
1.397
1.041
-0.000
lzz-2-055-H
Mar 10 2022
Scans = 4
Solvent = CDCl3
SF1 = 400.031799 MHz
SF2 = 100.596855 MHz
TBDPSO(CH2)5CO2H
4ae
from 3aeb
S78
9.47
5.85
3.92
4.29
2.10
2.02
2.12
10
8
6
4
2
0 PPM

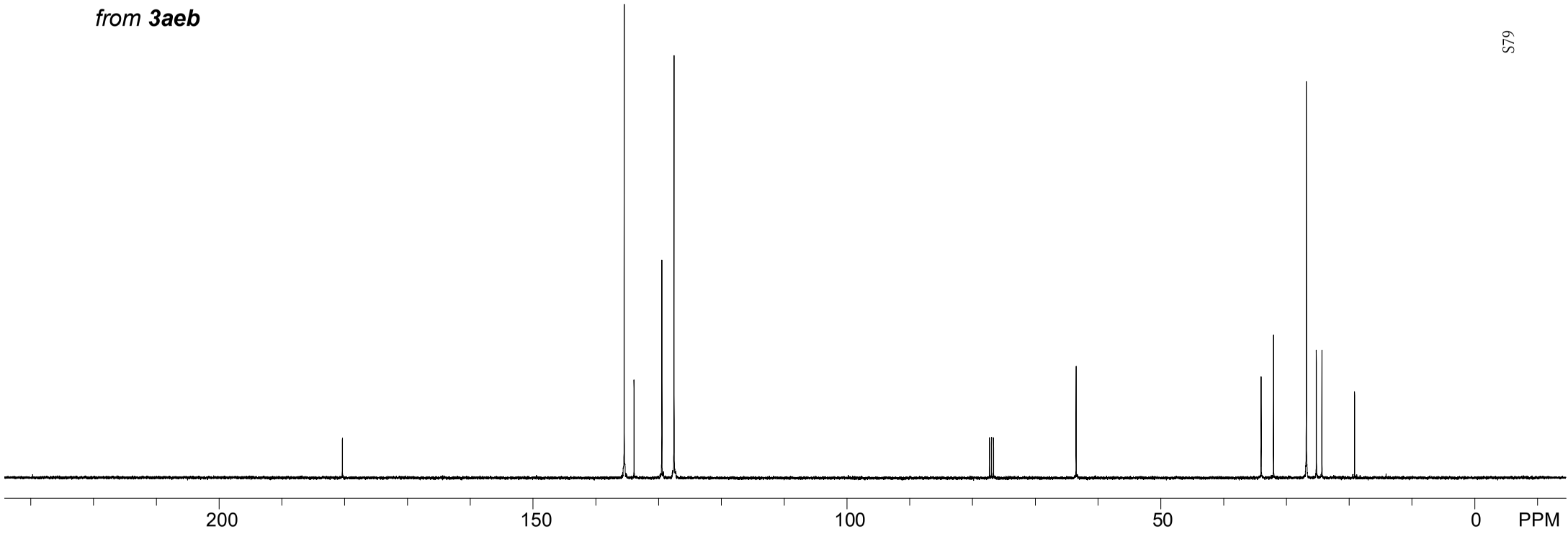


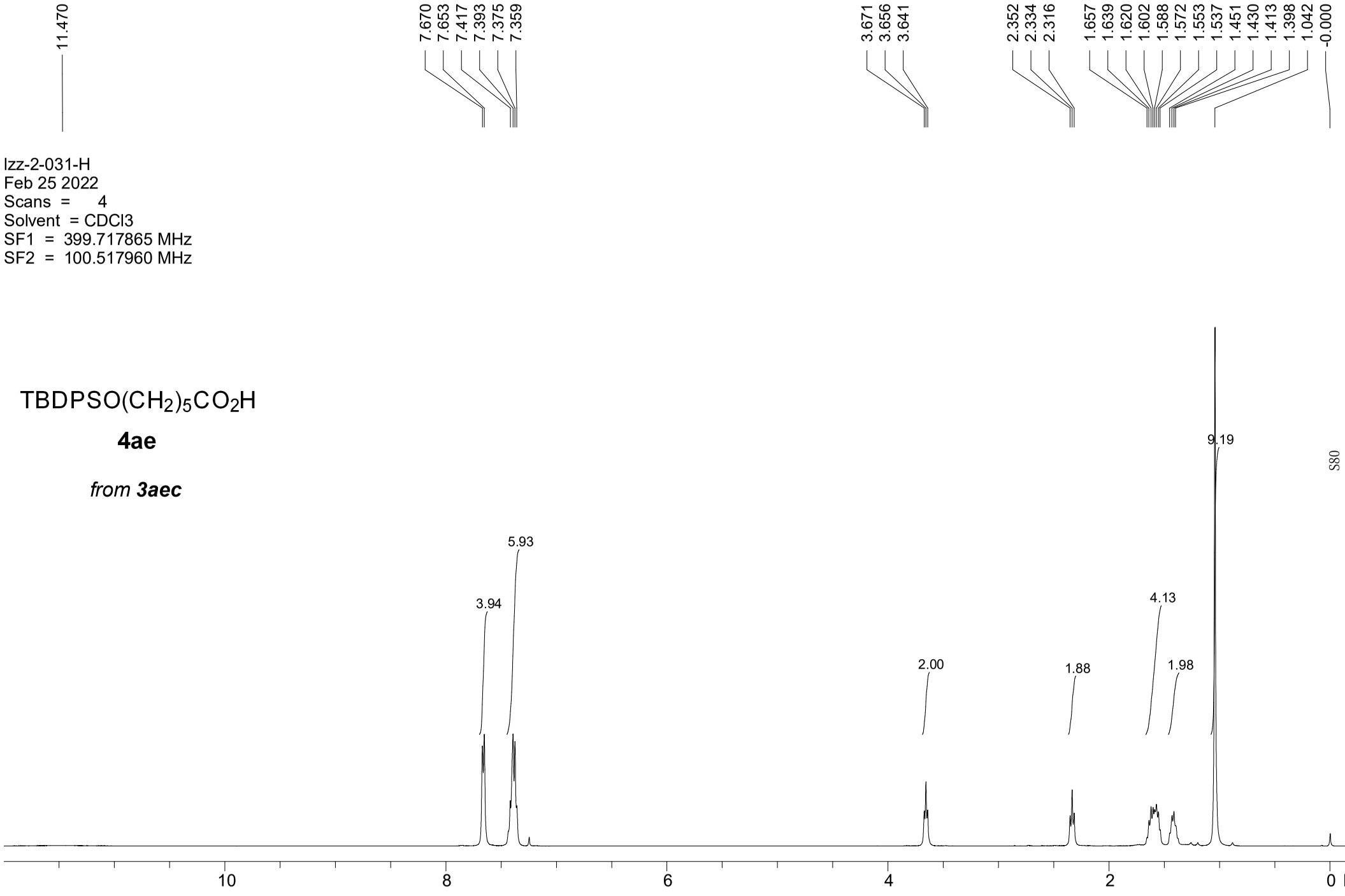
lzz-2-055-C
Mar 10 2022
Scans = 68
Solvent = cdcl3
SF1 = 100.519485 MHz
SF2 = 399.717072 MHz

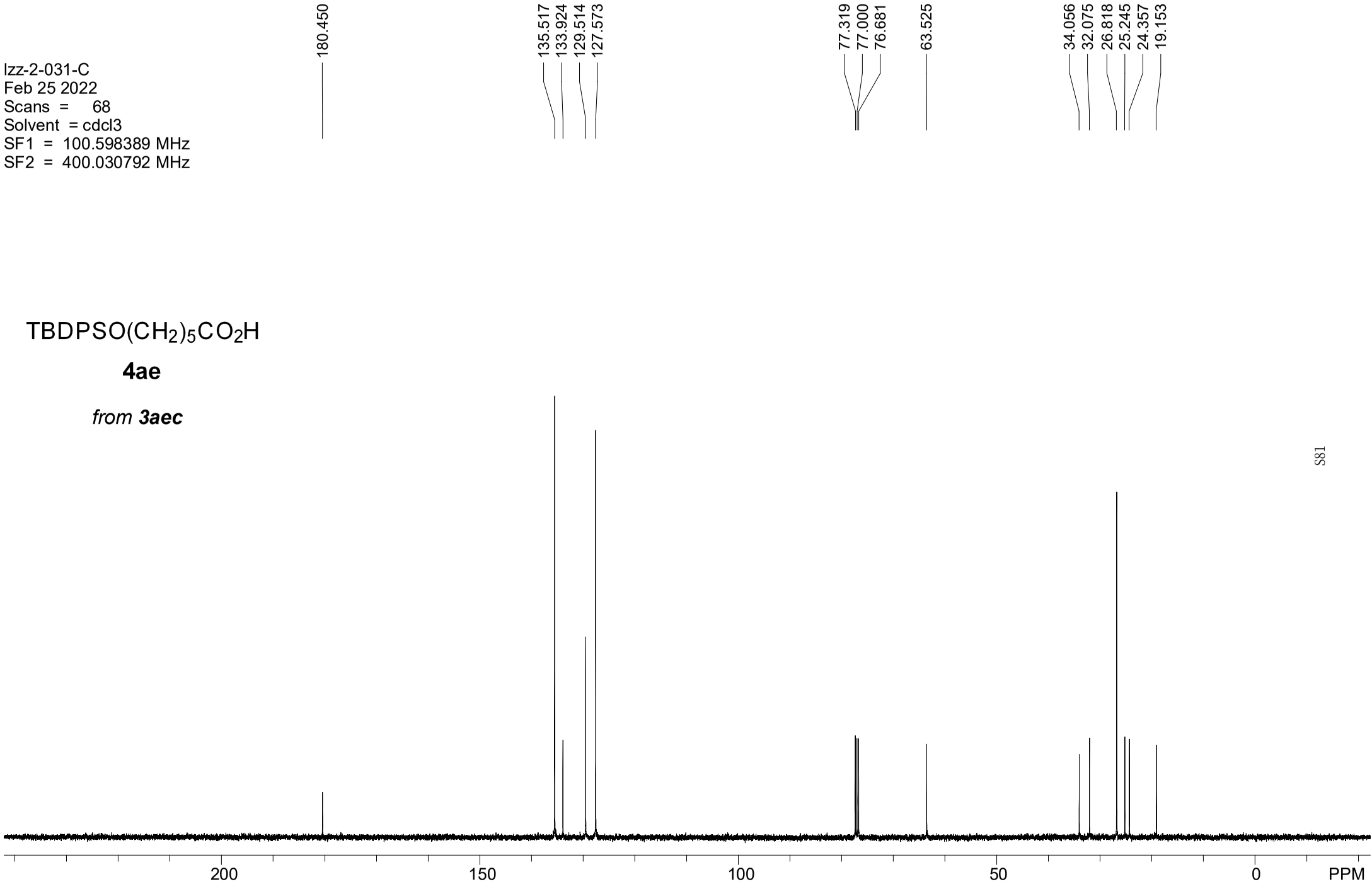
$TBDPSO(CH_2)_5CO_2H$

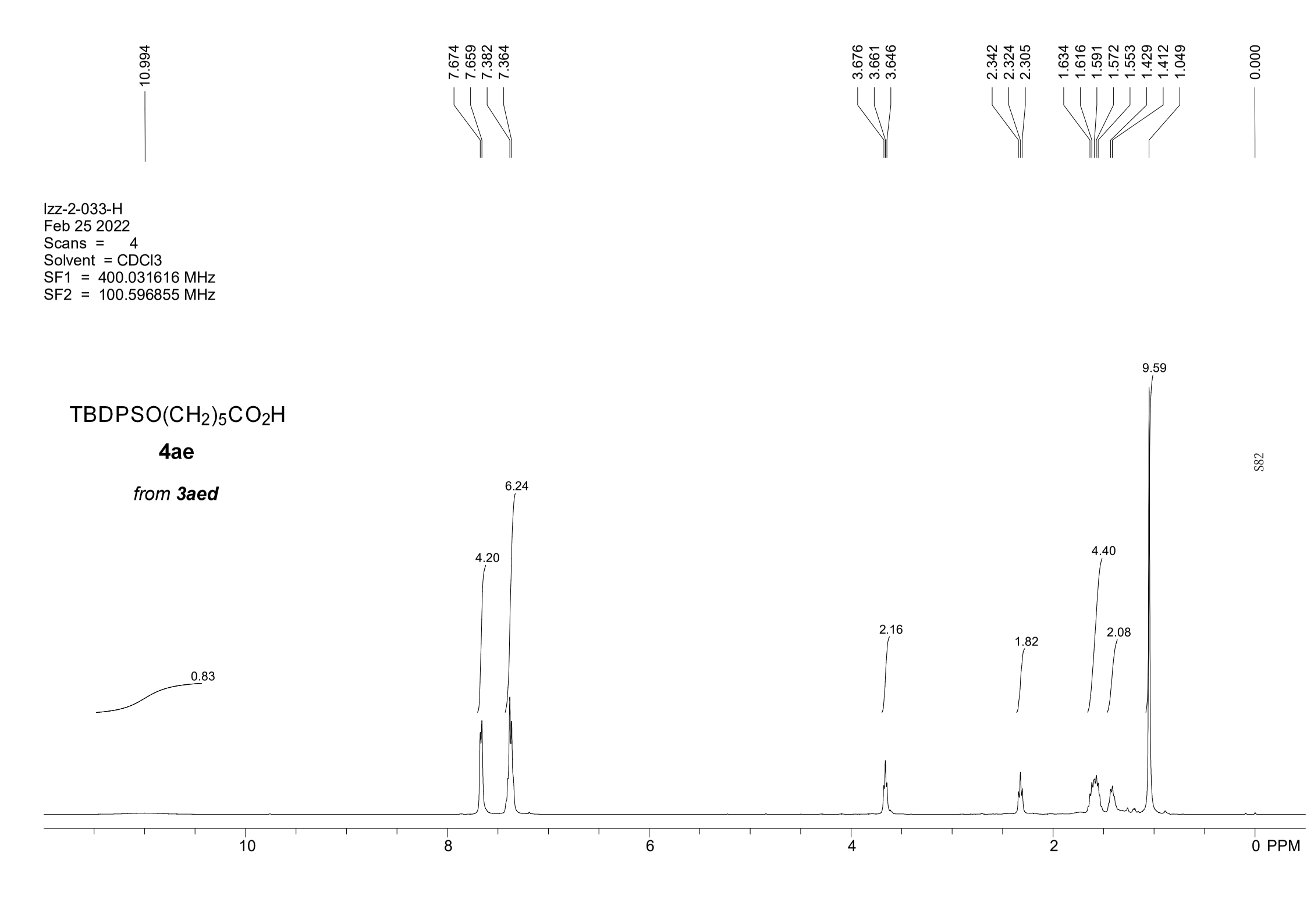
4ae

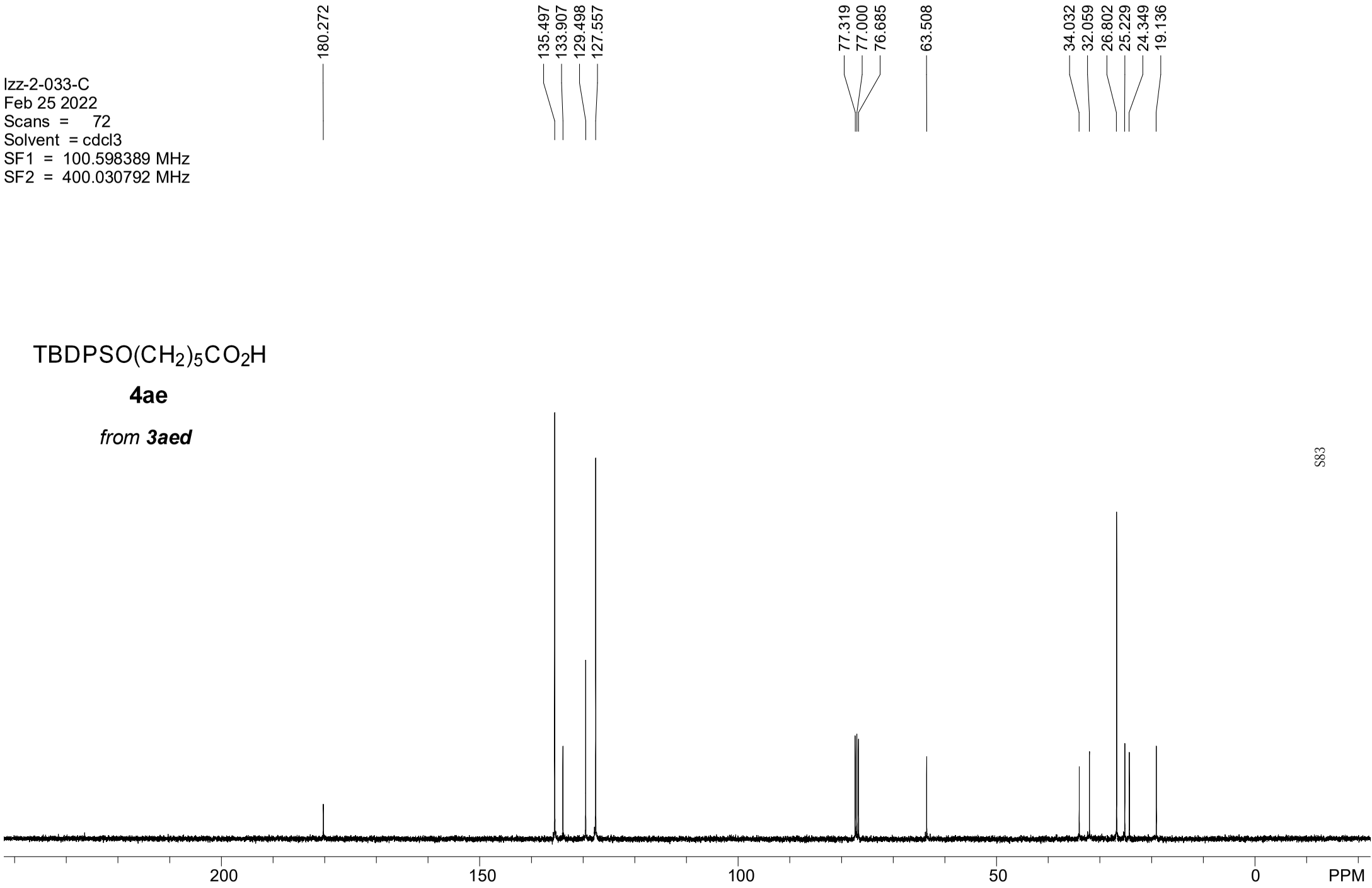
*from **3aeb***

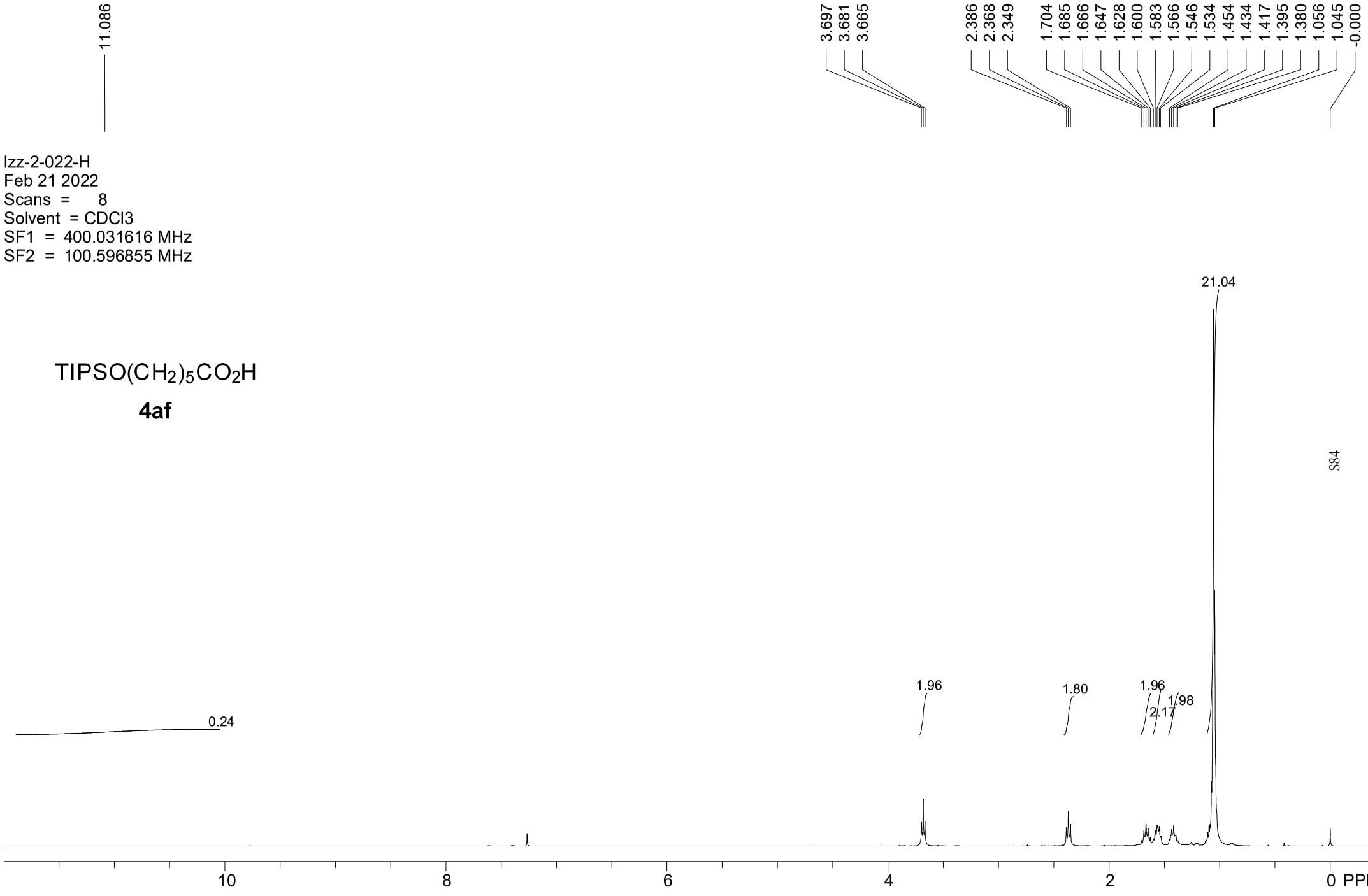


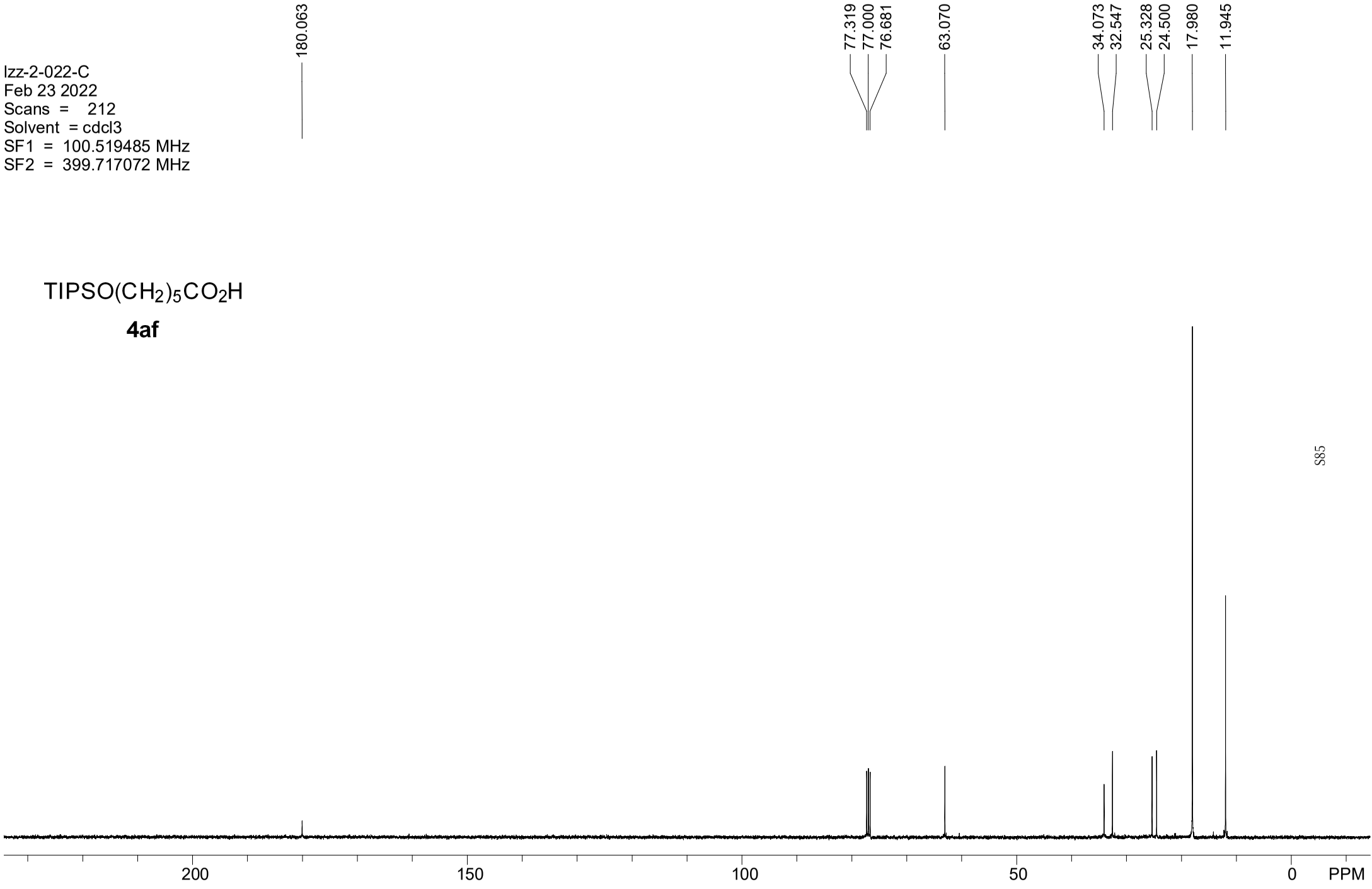

11.470
7.670
7.653
7.417
7.393
7.375
7.359
3.671
3.656
3.641
2.352
2.334
2.316
1.657
1.639
1.620
1.602
1.588
1.572
1.553
1.537
1.451
1.430
1.413
1.398
1.042
-0.000
lzz-2-031-H
Feb 25 2022
Scans = 4
Solvent = CDCl3
SF1 = 399.717865 MHz
SF2 = 100.517960 MHz
TBDPSO(CH2)5CO2H
4ae
from 3aec
S80
9.19
5.93
3.94
4.13
2.00
1.88
1.98
10
8
6
4
2
0 PPM

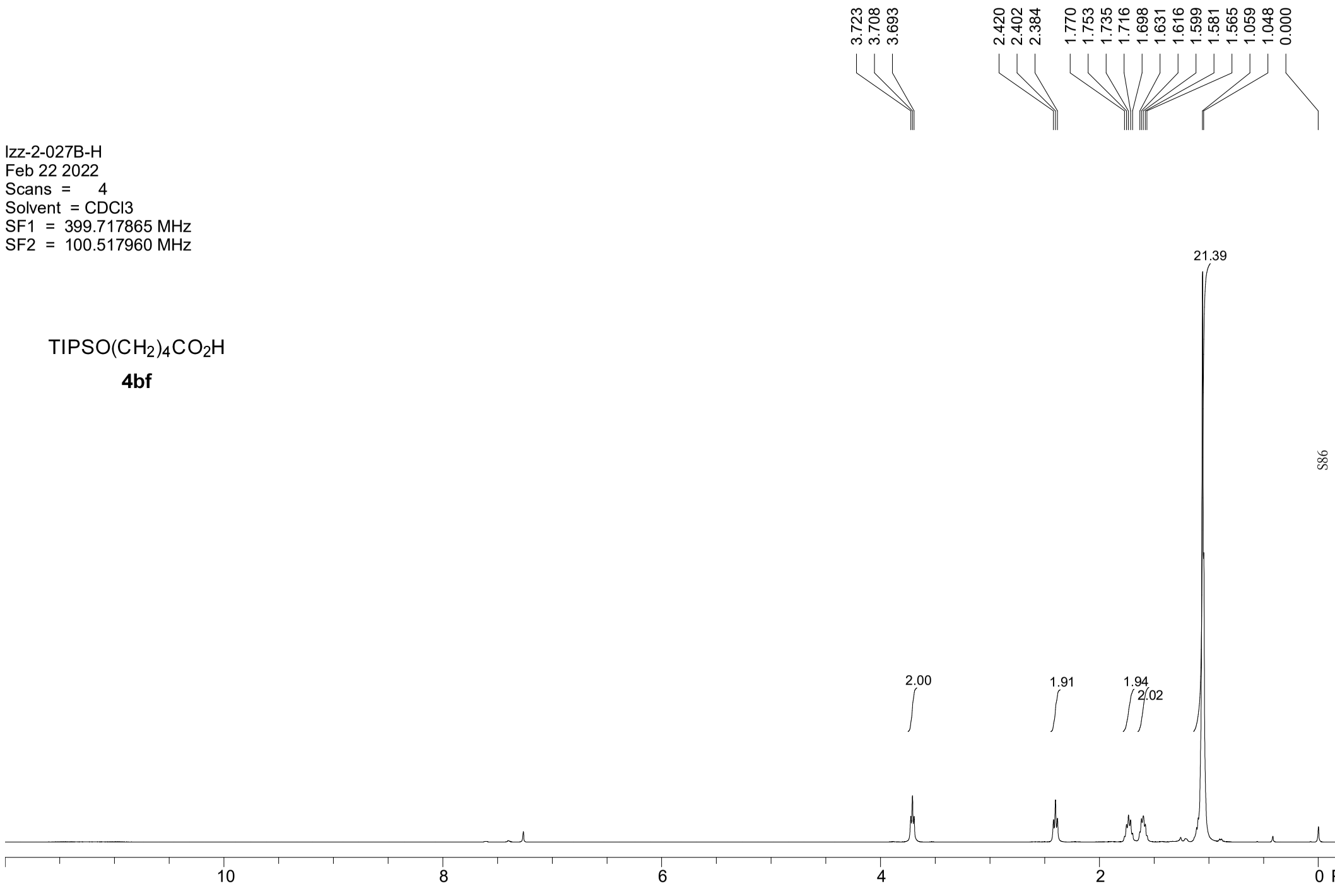

180.450
135.517
133.924
129.514
127.573
77.319
77.000
76.681
63.525
34.056
32.075
26.818
25.245
24.357
19.153
lzz-2-031-C
Feb 25 2022
Scans = 68
Solvent = cdcl3
SF1 = 100.598389 MHz
SF2 = 400.030792 MHz
TBDPSO(CH2)5CO2H
4ae
from 3aec
S81
200
150
100
50
0
PPM

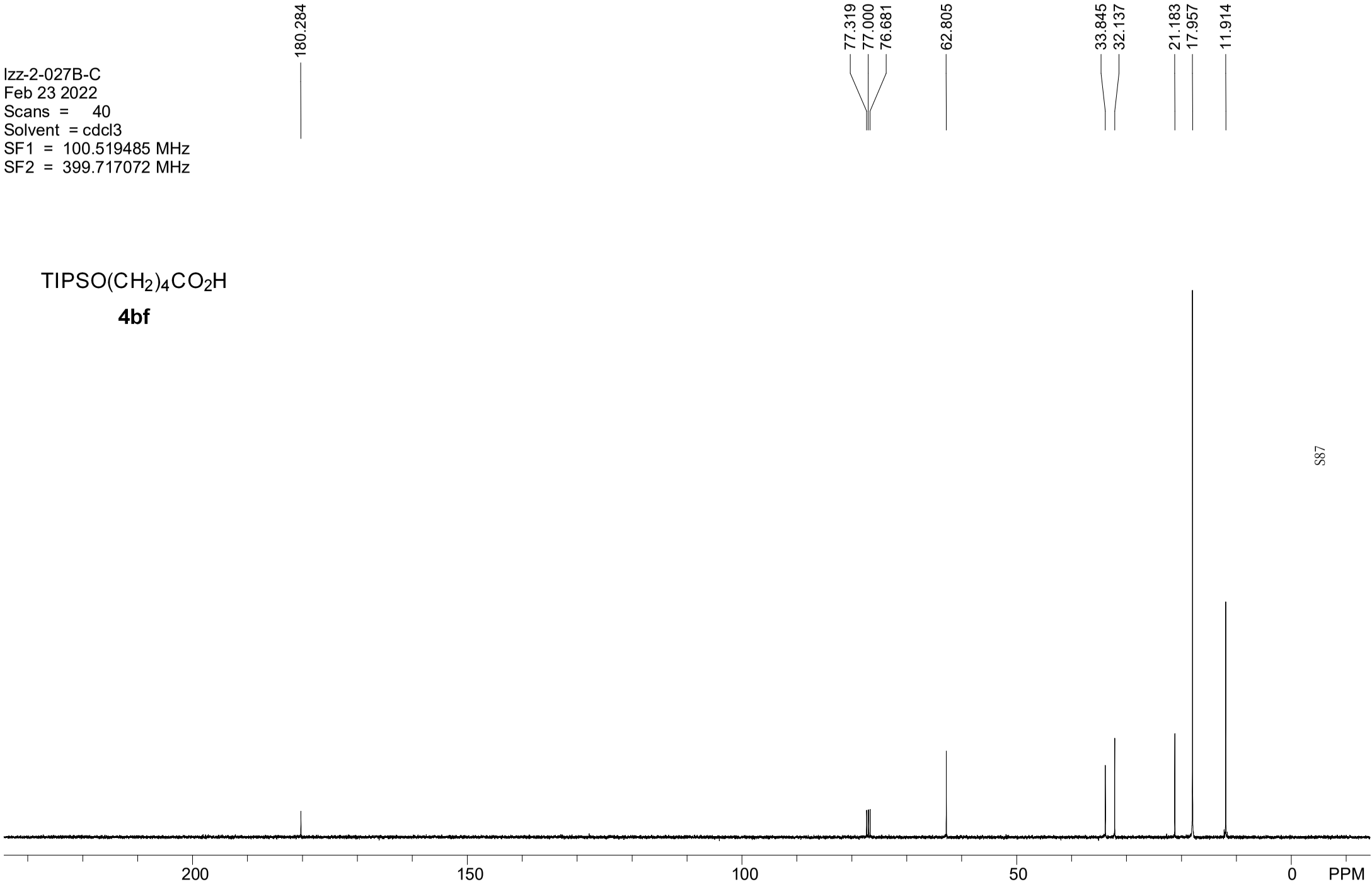

10.994
7.674
7.659
7.382
7.364
3.676
3.661
3.646
2.342
2.324
2.305
1.634
1.616
1.591
1.572
1.553
1.429
1.412
1.049
0.000
lzz-2-033-H
Feb 25 2022
Scans = 4
Solvent = CDCl3
SF1 = 400.031616 MHz
SF2 = 100.596855 MHz
TBDPSO(CH2)5CO2H
4ae
from 3aed
S82
9.59
6.24
4.20
4.40
2.16
2.08
1.82
0.83
10
8
6
4
2
0 PPM

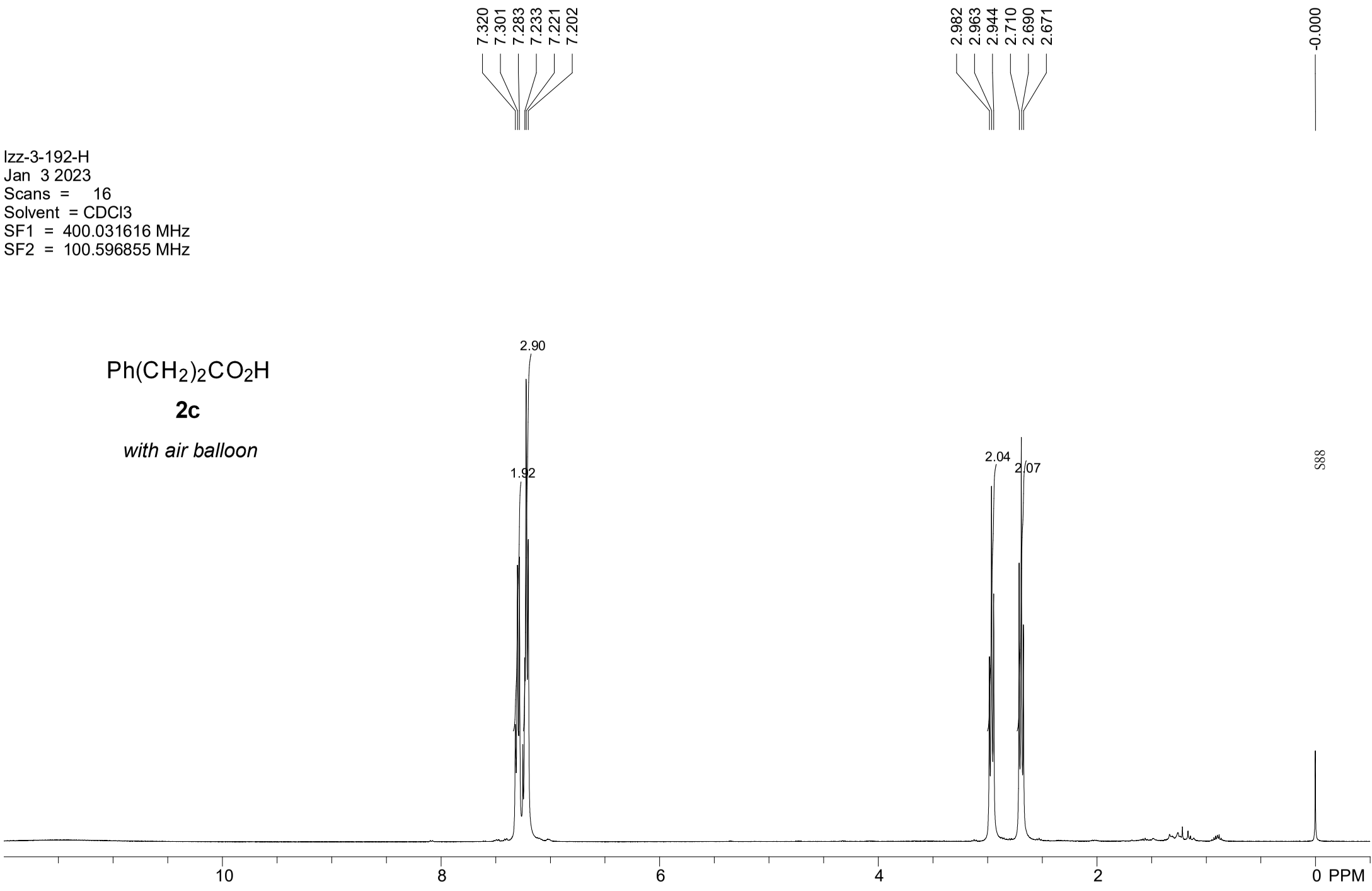

180.272
135.497
133.907
129.498
127.557
77.319
77.000
76.685
63.508
34.032
32.059
26.802
25.229
24.349
19.136
Izz-2-033-C
Feb 25 2022
Scans = 72
Solvent = cdcl3
SF1 = 100.598389 MHz
SF2 = 400.030792 MHz
TBDPSO(CH2)5CO2H
4ae
from 3aed
S83
200
150
100
50
0
PPM

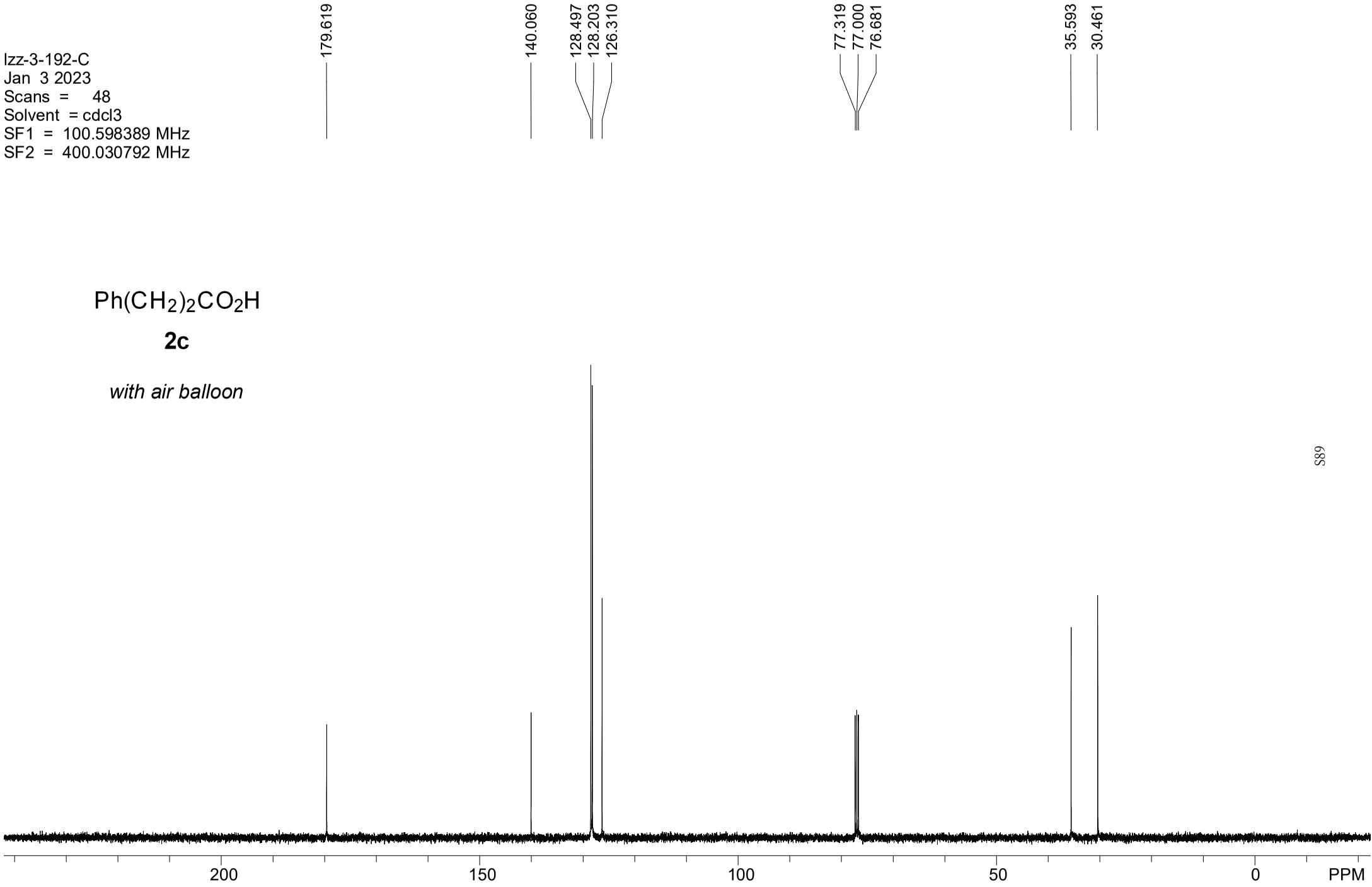

11.086
3.697
3.681
3.665
2.386
2.368
2.349
1.704
1.685
1.666
1.647
1.628
1.600
1.583
1.566
1.546
1.534
1.454
1.434
1.417
1.395
1.380
1.056
1.045
-0.000
lzz-2-022-H
Feb 21 2022
Scans = 8
Solvent = CDCl3
SF1 = 400.031616 MHz
SF2 = 100.596855 MHz
TIPSO(CH2)5CO2H
4af
S84
21.04
1.96
1.80
1.96
1.98
2.17
0.24
10
8
6
4
2
0 PPM

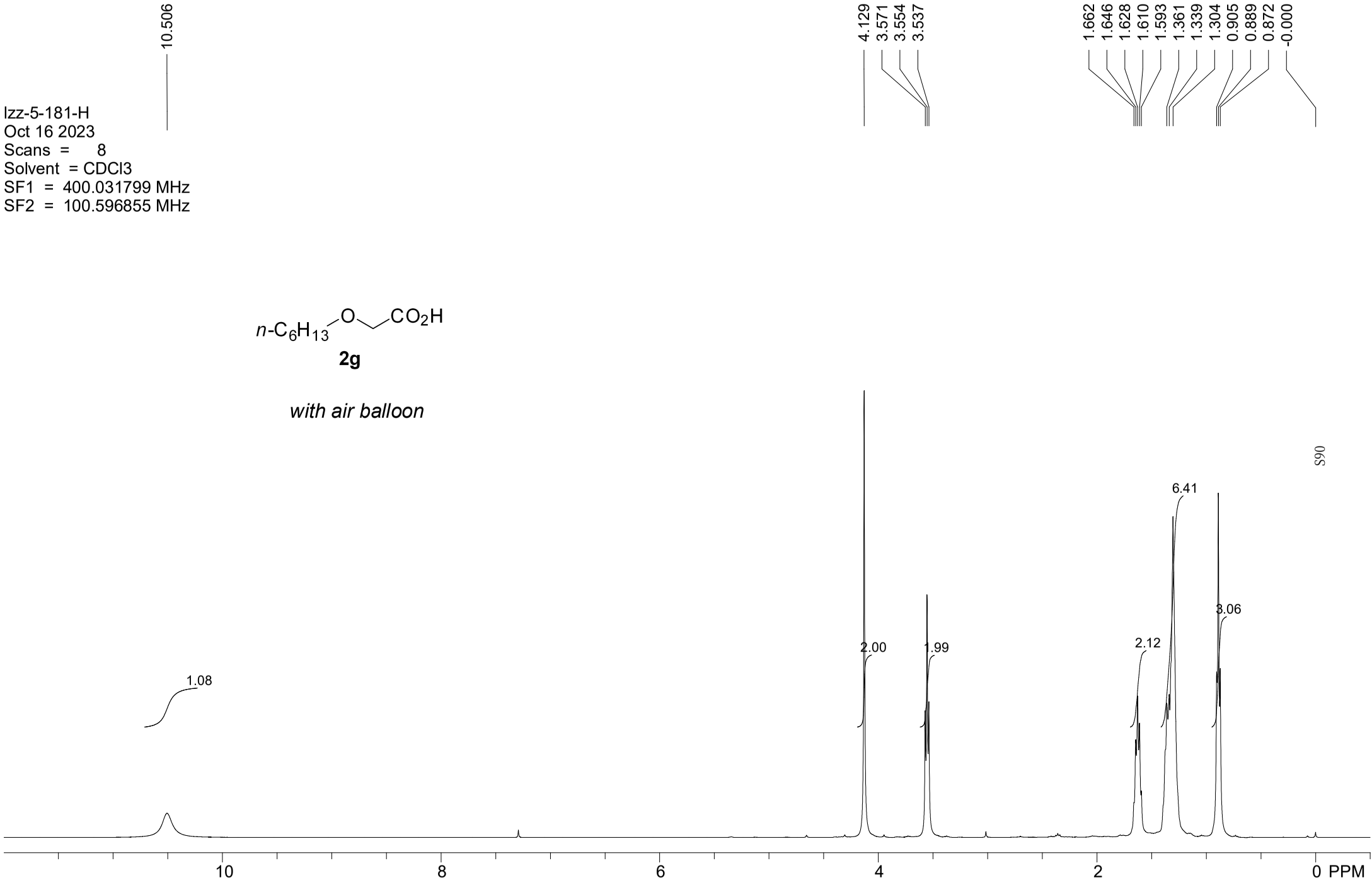

180.063
77.319
77.000
76.681
63.070
34.073
32.547
25.328
24.500
17.980
11.945
lzz-2-022-C
Feb 23 2022
Scans = 212
Solvent = cdcl3
SF1 = 100.519485 MHz
SF2 = 399.717072 MHz
TIPSO(CH2)5CO2H
4af
S85
200
150
100
50
0
PPM

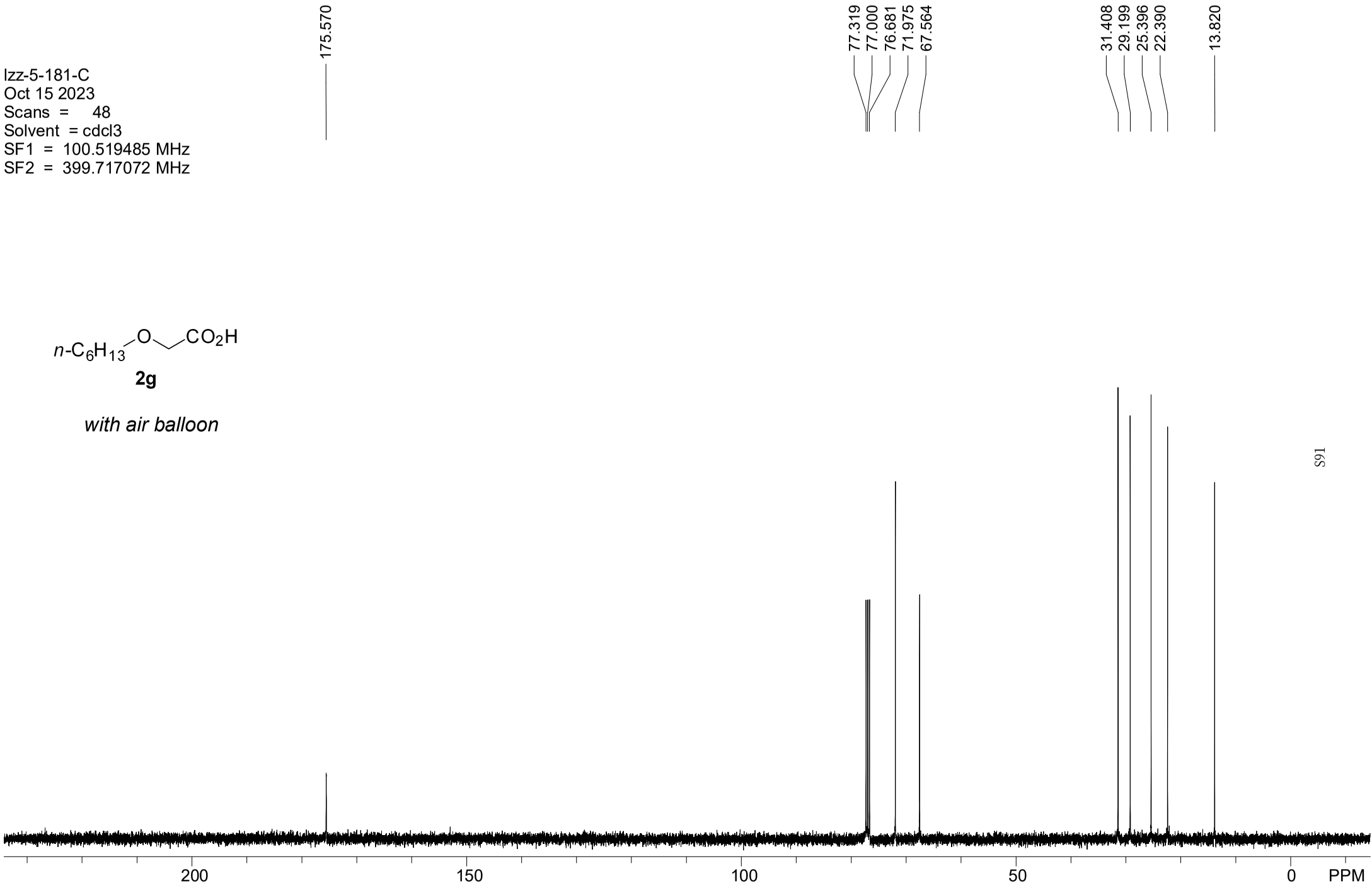

3.723
3.708
3.693
2.420
2.402
2.384
1.770
1.753
1.735
1.716
1.698
1.631
1.616
1.599
1.581
1.565
1.059
1.048
0.000
lzz-2-027B-H
Feb 22 2022
Scans = 4
Solvent = CDCl3
SF1 = 399.717865 MHz
SF2 = 100.517960 MHz
TIPSO(CH2)4CO2H
4bf
21.39
2.00
1.91
1.94
2.02
S86
10
8
6
4
2
0 PPM

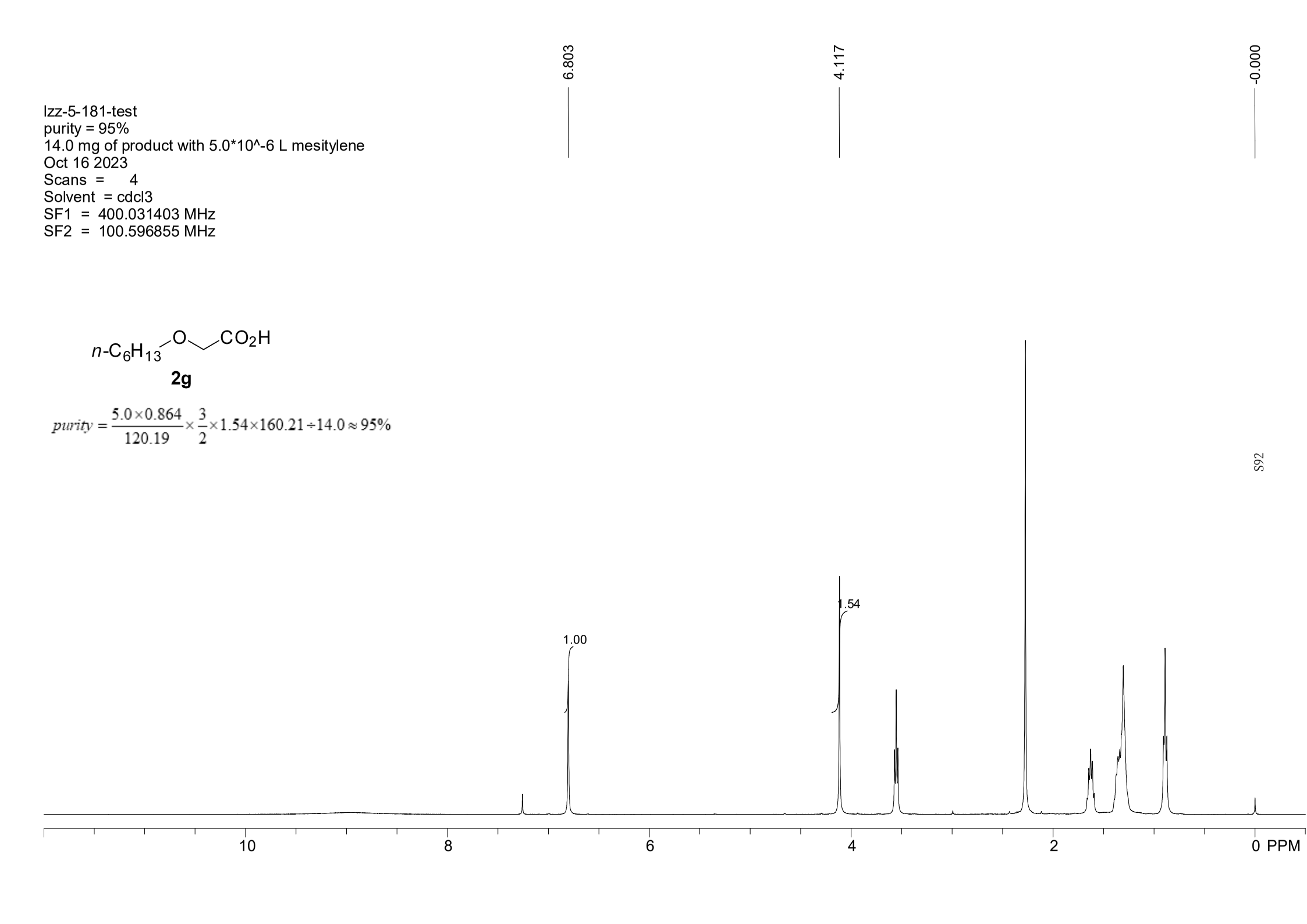

180.284
77.319
77.000
76.681
62.805
33.845
32.137
21.183
17.957
11.914
lzz-2-027B-C
Feb 23 2022
Scans = 40
Solvent = cdcl3
SF1 = 100.519485 MHz
SF2 = 399.717072 MHz
TIPSO(CH2)4CO2H
4bf
200
150
100
50
0
PPM

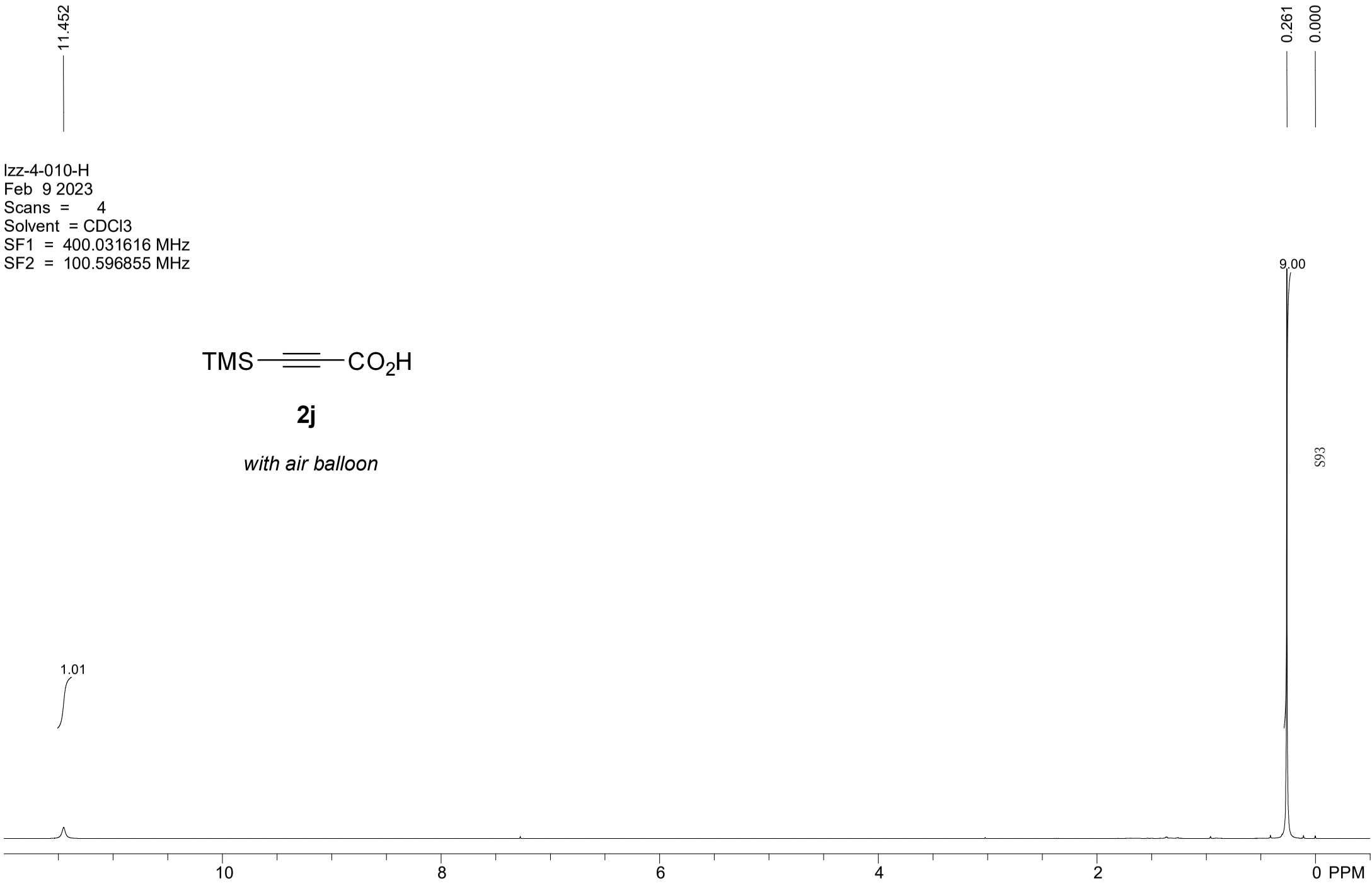

7.320
7.301
7.283
7.233
7.221
7.202
2.982
2.963
2.944
2.710
2.690
2.671
-0.000
Izz-3-192-H
Jan 3 2023
Scans = 16
Solvent = CDCl3
SF1 = 400.031616 MHz
SF2 = 100.596855 MHz
Ph(CH2)2CO2H
2c
with air balloon
2.90
1.92
2.04
2.07
S88
10
8
6
4
2
0 PPM

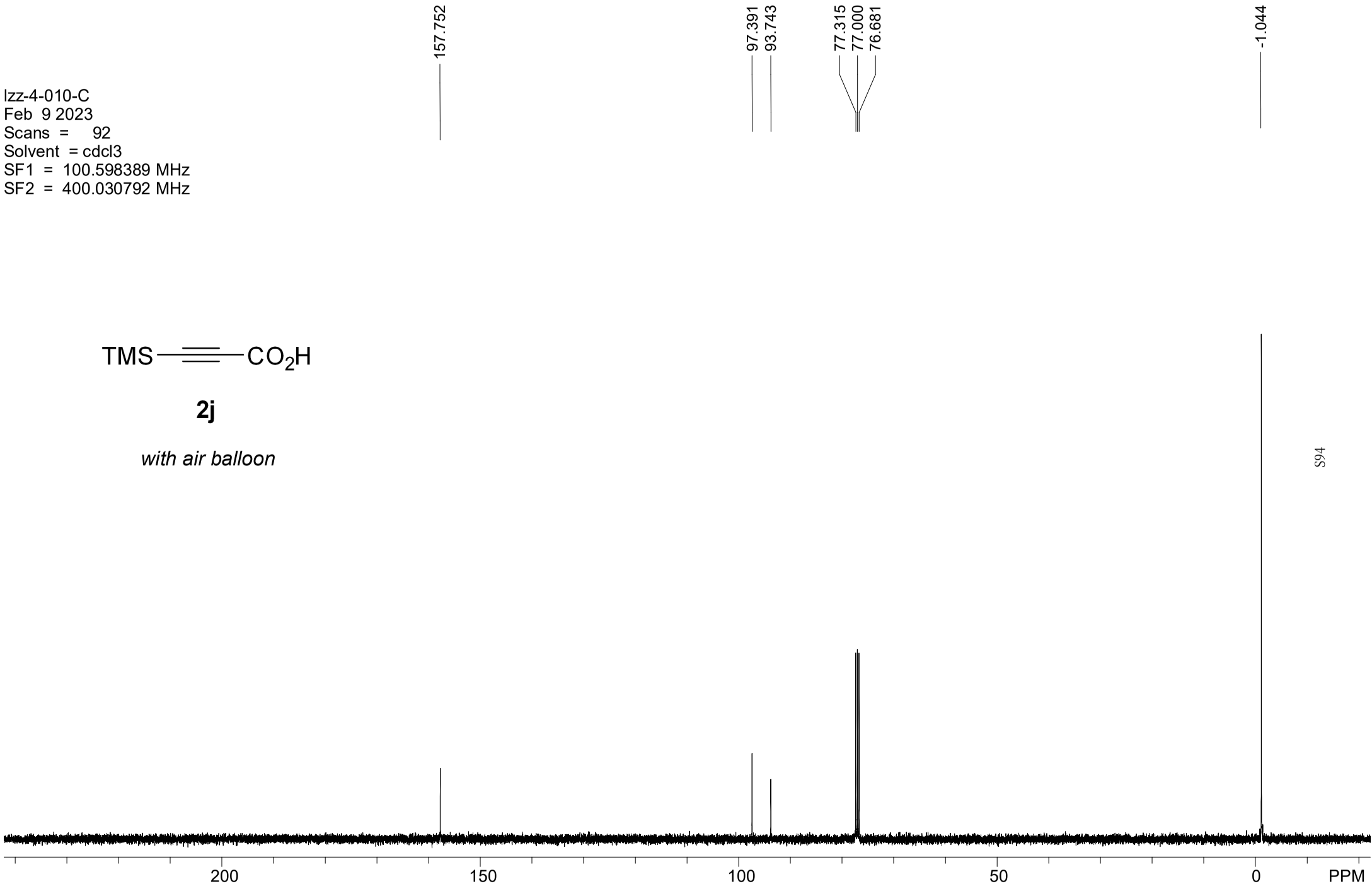

179.619
140.060
128.497
128.203
126.310
77.319
77.000
76.681
35.593
30.461
lzz-3-192-C
Jan 3 2023
Scans = 48
Solvent = cdcl3
SF1 = 100.598389 MHz
SF2 = 400.030792 MHz
Ph(CH2)2CO2H
2c
with air balloon
200
150
100
50
0
PPM

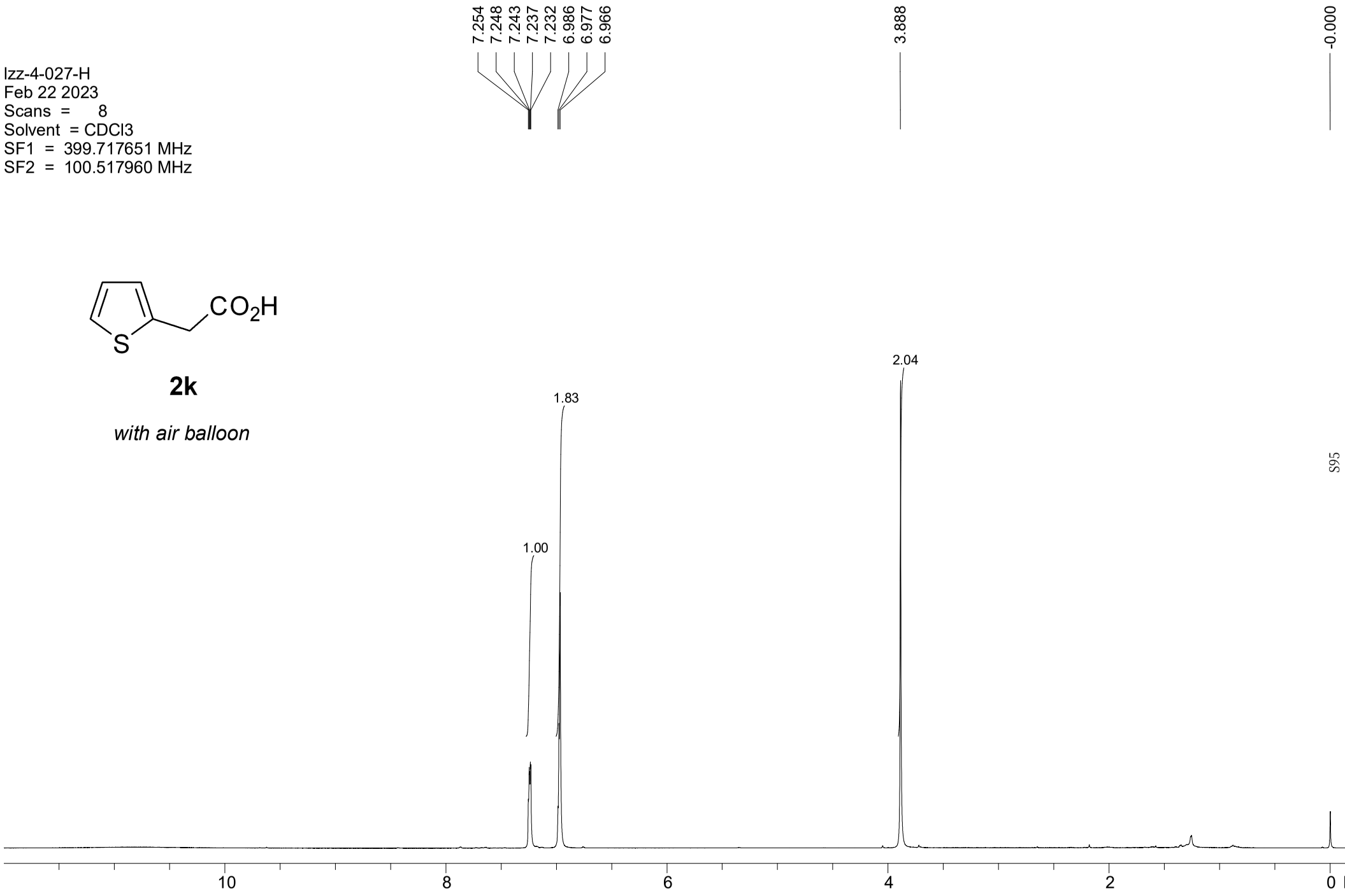

10.506
4.129
3.571
3.554
3.537
1.662
1.646
1.628
1.610
1.593
1.361
1.339
1.304
0.905
0.889
0.872
-0.000
lzz-5-181-H
Oct 16 2023
Scans = 8
Solvent = CDCl3
SF1 = 400.031799 MHz
SF2 = 100.596855 MHz
n-C6H13 O CO2H
2g
with air balloon
S90
1.08
2.00
1.99
2.12
6.41
3.06
10
8
6
4
2
0 PPM

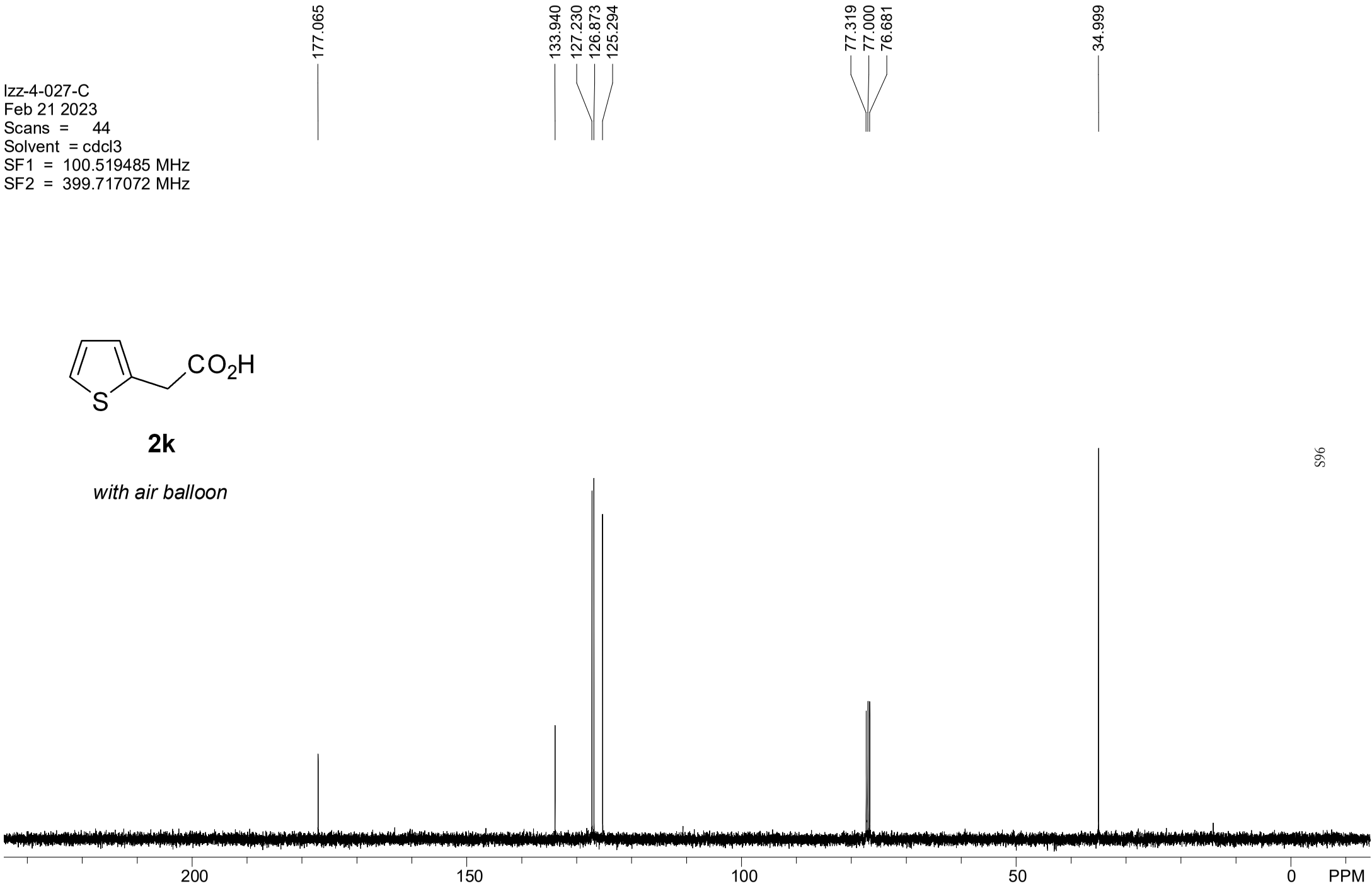

175.570
77.319
77.000
76.681
71.975
67.564
31.408
29.199
25.396
22.390
13.820
lzz-5-181-C
Oct 15 2023
Scans = 48
Solvent = cdcl3
SF1 = 100.519485 MHz
SF2 = 399.717072 MHz
n-C6H13
O
CO2H
2g
with air balloon
S91
200
150
100
50
0
PPM


6.803
4.117
-0.000
lzz-5-181-test
purity = 95%
14.0 mg of product with 5.0*10^-6 L mesitylene
Oct 16 2023
Scans = 4
Solvent = cdcl3
SF1 = 400.031403 MHz
SF2 = 100.596855 MHz
n-C6H13 O CO2H
2g
purity = 5.0×0.864/120.19 × 3/2 × 1.54 × 160.21 ÷ 14.0 ≈ 95%
S92
1.00
1.54
10
8
6
4
2
0 PPM


11.452
0.261
0.000
lzz-4-010-H
Feb 9 2023
Scans = 4
Solvent = CDCl3
SF1 = 400.031616 MHz
SF2 = 100.596855 MHz
TMS
CO2H
2j
with air balloon
9.00
S93
1.01
10
8
6
4
2
0 PPM


157.752
97.391
93.743
77.315
77.000
76.681
-1.044
lzz-4-010-C
Feb 9 2023
Scans = 92
Solvent = cdcl3
SF1 = 100.598389 MHz
SF2 = 400.030792 MHz
TMS
CO2H
2j
with air balloon
S94
200
150
100
50
0
PPM


7.254
7.248
7.243
7.237
7.232
6.986
6.977
6.966
3.888
-0.000
lzz-4-027-H
Feb 22 2023
Scans = 8
Solvent = CDCl3
SF1 = 399.717651 MHz
SF2 = 100.517960 MHz
CO2H
S
2k
with air balloon
2.04
1.83
1.00
S95
10
8
6
4
2
0 PPM


177.065
133.940
127.230
126.873
125.294
77.319
77.000
76.681
34.999
lzz-4-027-C
Feb 21 2023
Scans = 44
Solvent = cdcl3
SF1 = 100.519485 MHz
SF2 = 399.717072 MHz
CO2H
S
2k
with air balloon
S96
200
150
100
50
0
PPM

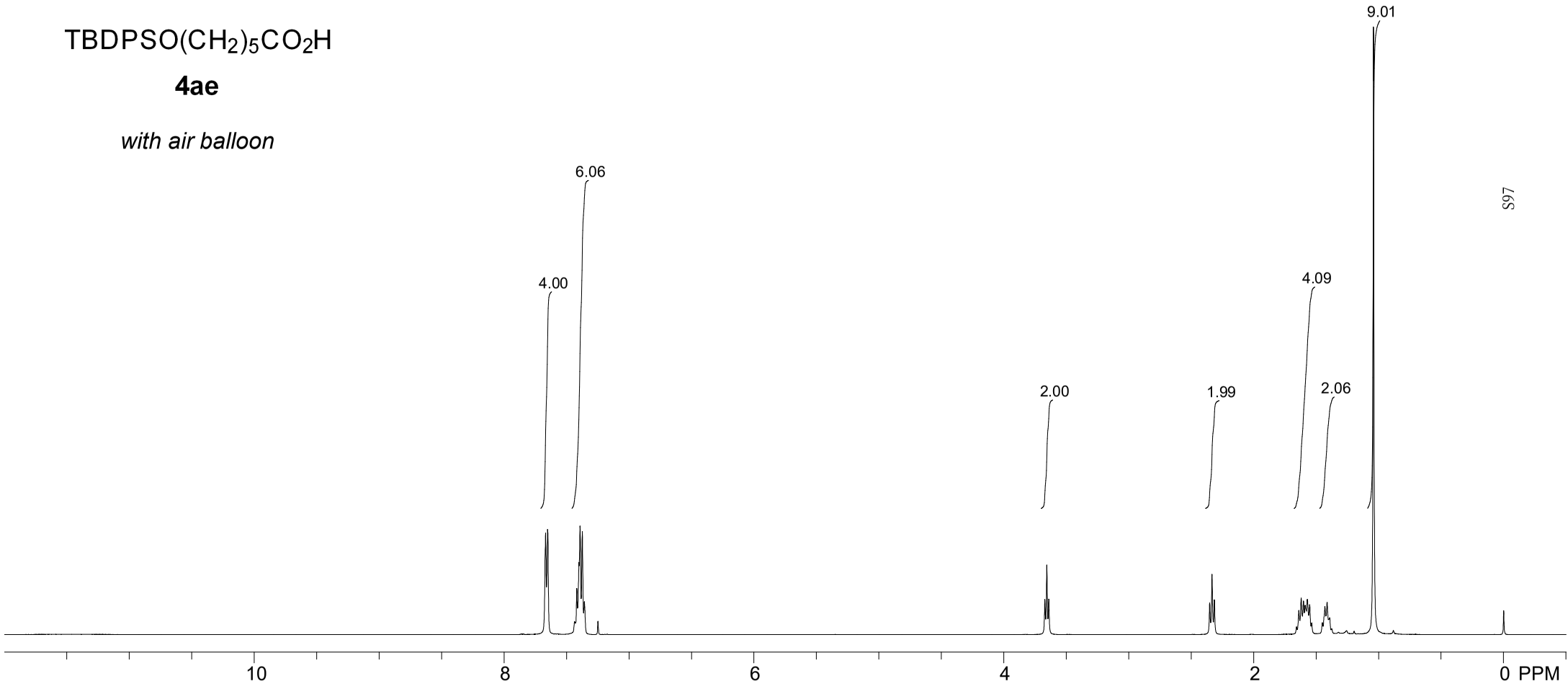
lzz-4-002-H-2
Jan 7 2024
Scans = 4
Solvent = cdcl3
SF1 = 399.717651 MHz
SF2 = 100.517960 MHz

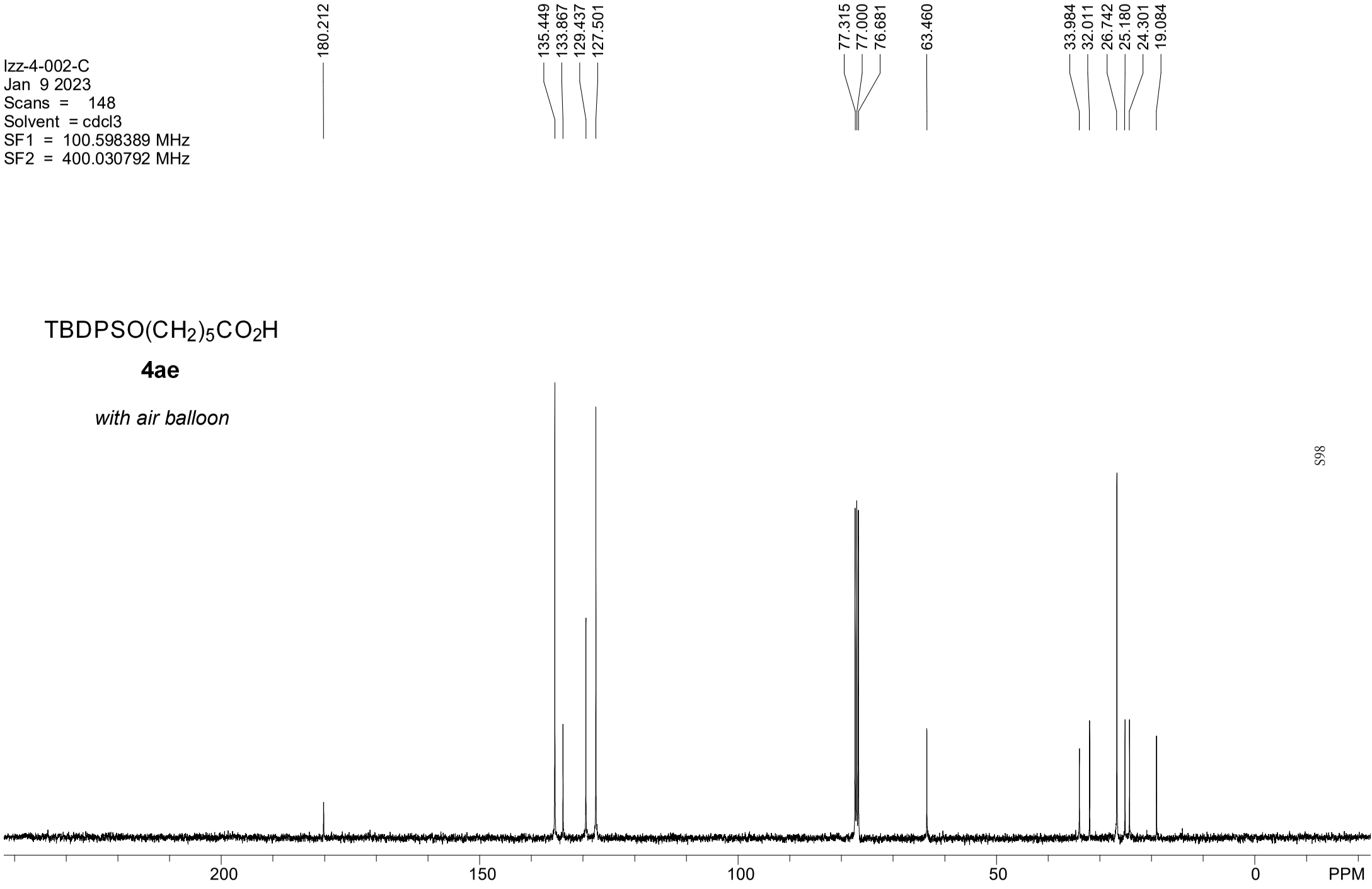
7.668 7.652 7.436 7.418 7.401 7.392 7.374 7.358
3.671 3.655 3.639
2.352 2.333 2.314
1.658 1.639 1.620 1.601 1.588 1.571 1.552 1.536 1.451 1.430 1.413 1.392 1.376 1.041

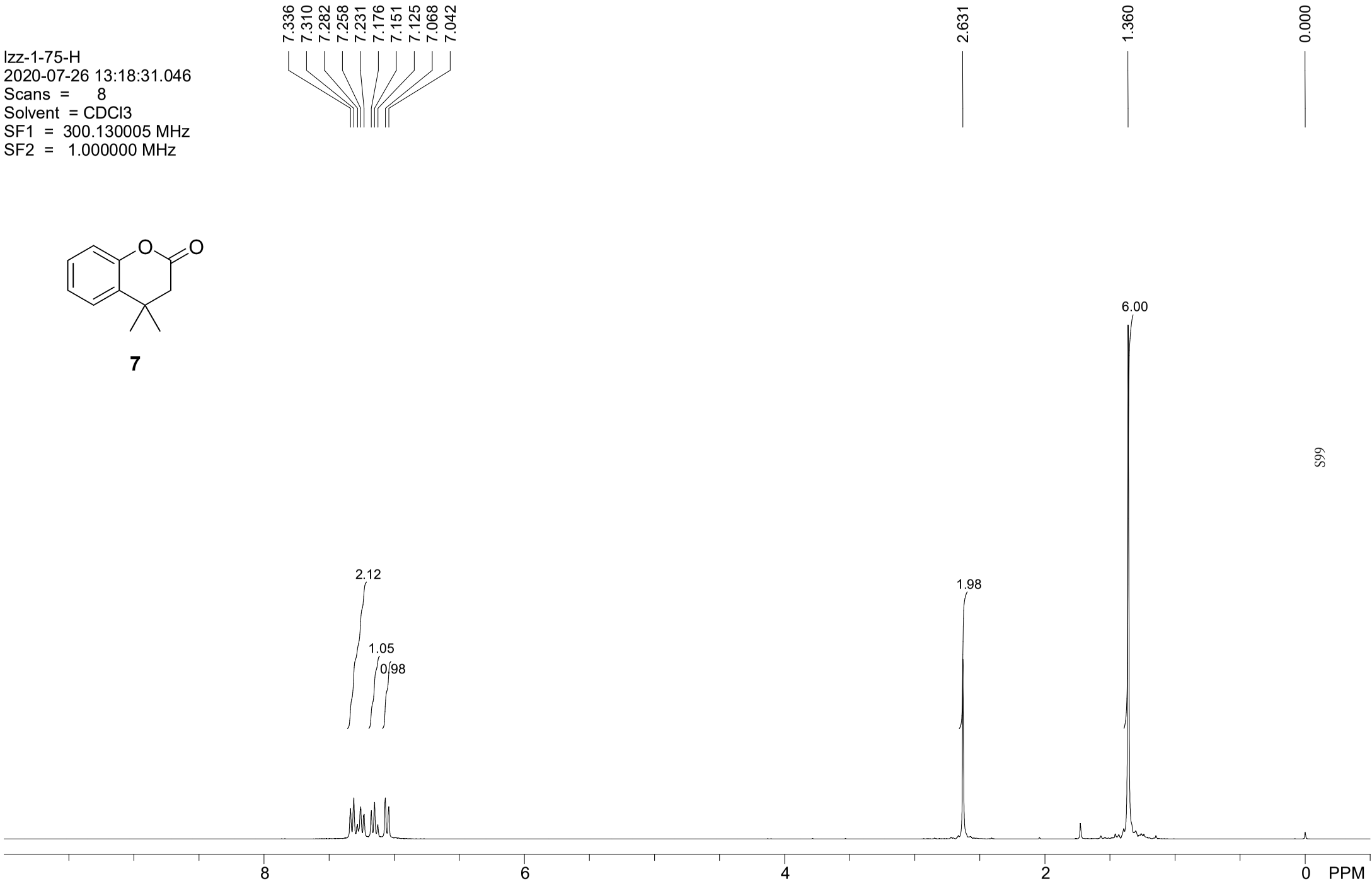
$TBDPSO(CH_2)_5CO_2H$

4ae

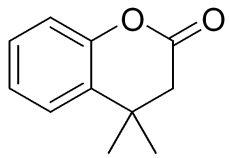
with air balloon



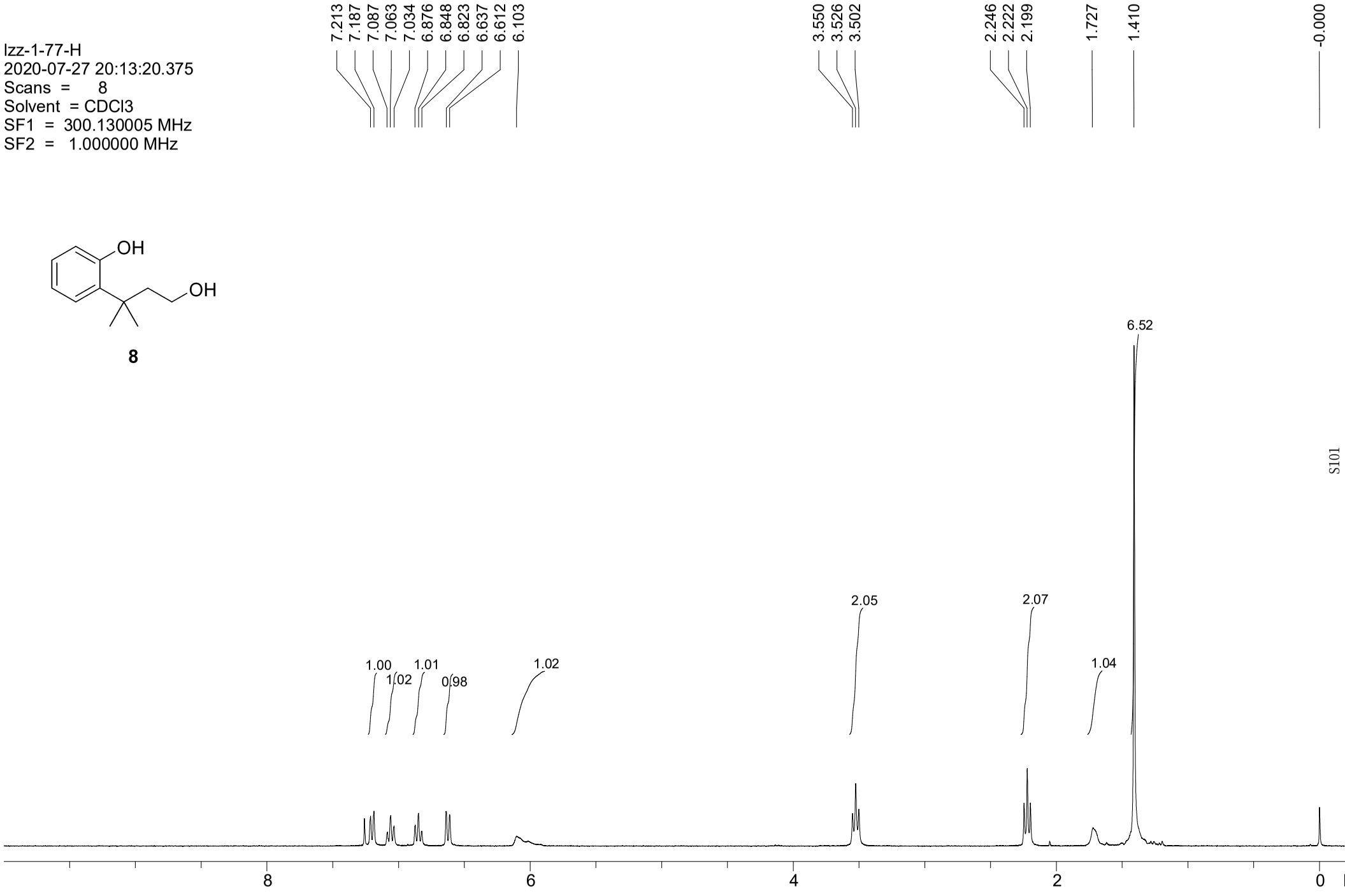

180.212
135.449
133.867
129.437
127.501
77.315
77.000
76.681
63.460
33.984
32.011
26.742
25.180
24.301
19.084
lzz-4-002-C
Jan 9 2023
Scans = 148
Solvent = cdcl3
SF1 = 100.598389 MHz
SF2 = 400.030792 MHz
TBDPSO(CH2)5CO2H
4ae
with air balloon
S98
200
150
100
50
0
PPM


7.336
7.310
7.282
7.258
7.231
7.176
7.151
7.125
7.068
7.042
2.631
1.360
0.000
lzz-1-75-H
2020-07-26 13:18:31.046
Scans = 8
Solvent = CDCl3
SF1 = 300.130005 MHz
SF2 = 1.000000 MHz
O
O
7
6.00
2.12
1.98
1.05
0.98
8
6
4
2
0
PPM

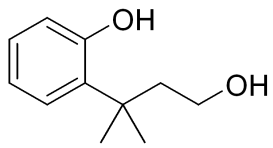
lzz-1-75-C
2020-07-26 13:24:42.031
Scans = 81
Solvent = CDCl3
SF1 = 75.467751 MHz
SF2 = 1.000000 MHz



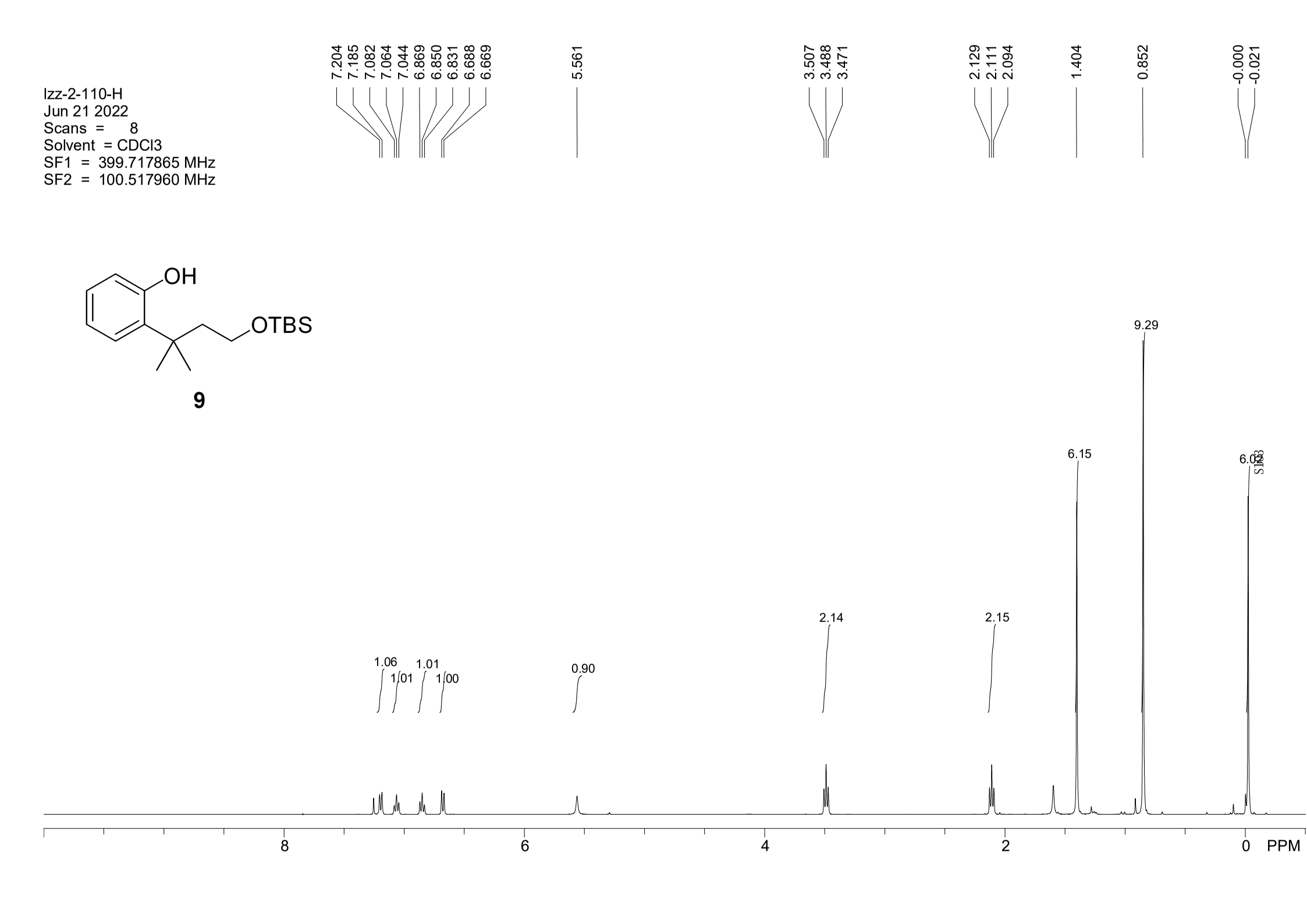
7


lzz-1-77-H
2020-07-27 20:13:20.375
Scans = 8
Solvent = CDCl3
SF1 = 300.130005 MHz
SF2 = 1.000000 MHz
7.213
7.187
7.087
7.063
7.034
6.876
6.848
6.823
6.637
6.612
6.103
3.550
3.526
3.502
2.246
2.222
2.199
1.727
1.410
-0.000
OH
OH
8
6.52
2.05
2.07
1.04
1.00
1.02
1.01
0.98
1.02
S101
8
6
4
2
0
PPM

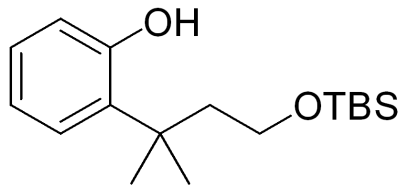
lzz-1-77-C
2020-07-28 15:59:34.796
Scans = 65
Solvent = CDCl3
SF1 = 75.467751 MHz
SF2 = 1.000000 MHz



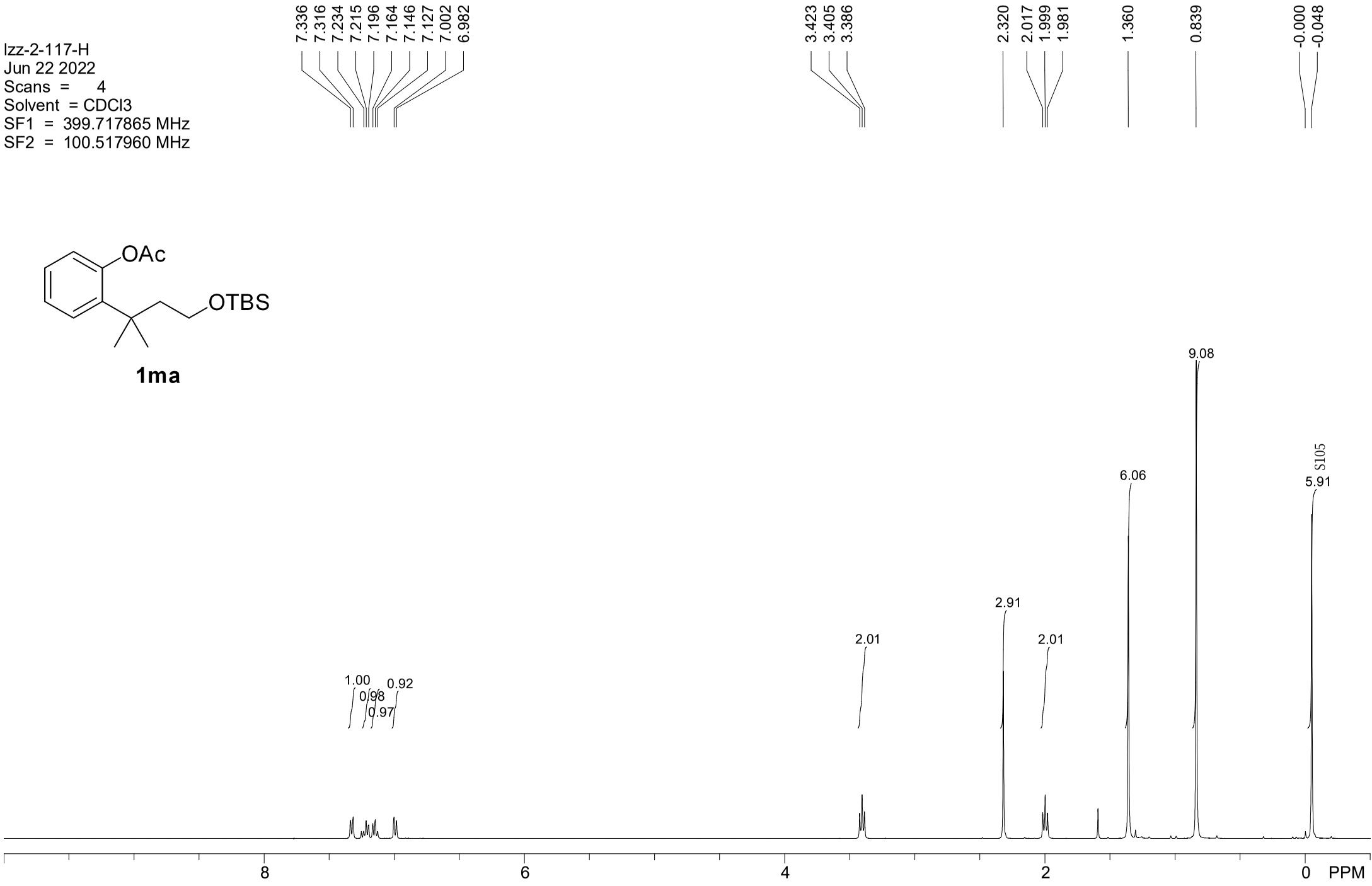
8


lzz-2-110-H
Jun 21 2022
Scans = 8
Solvent = CDCl3
SF1 = 399.717865 MHz
SF2 = 100.517960 MHz
7.204
7.185
7.082
7.064
7.044
6.869
6.850
6.831
6.688
6.669
5.561
3.507
3.488
3.471
2.129
2.111
2.094
1.404
0.852
-0.000
-0.021
OH
OTBS
9
9.29
6.15
6.02
2.14
2.15
1.06
1.01
1.01
1.00
0.90
8
6
4
2
0
PPM

lzz-2-110-C
CDCl3
Tue Jun 21 21:58:43 2022
Scans = 240
SF1 = 100.617798 MHz
SF2 = 1.000000 MHz

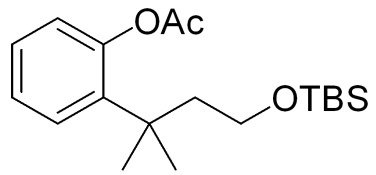


9


lzz-2-117-H
Jun 22 2022
Scans = 4
Solvent = CDCl3
SF1 = 399.717865 MHz
SF2 = 100.517960 MHz
7.336
7.316
7.234
7.215
7.196
7.164
7.146
7.127
7.002
6.982
3.423
3.405
3.386
2.320
2.017
1.999
1.981
1.360
0.839
-0.000
-0.048
OAc
OTBS
1ma
9.08
S105
6.06
5.91
2.91
2.01
2.01
1.00
0.98
0.97
0.92
8
6
4
2
0
PPM

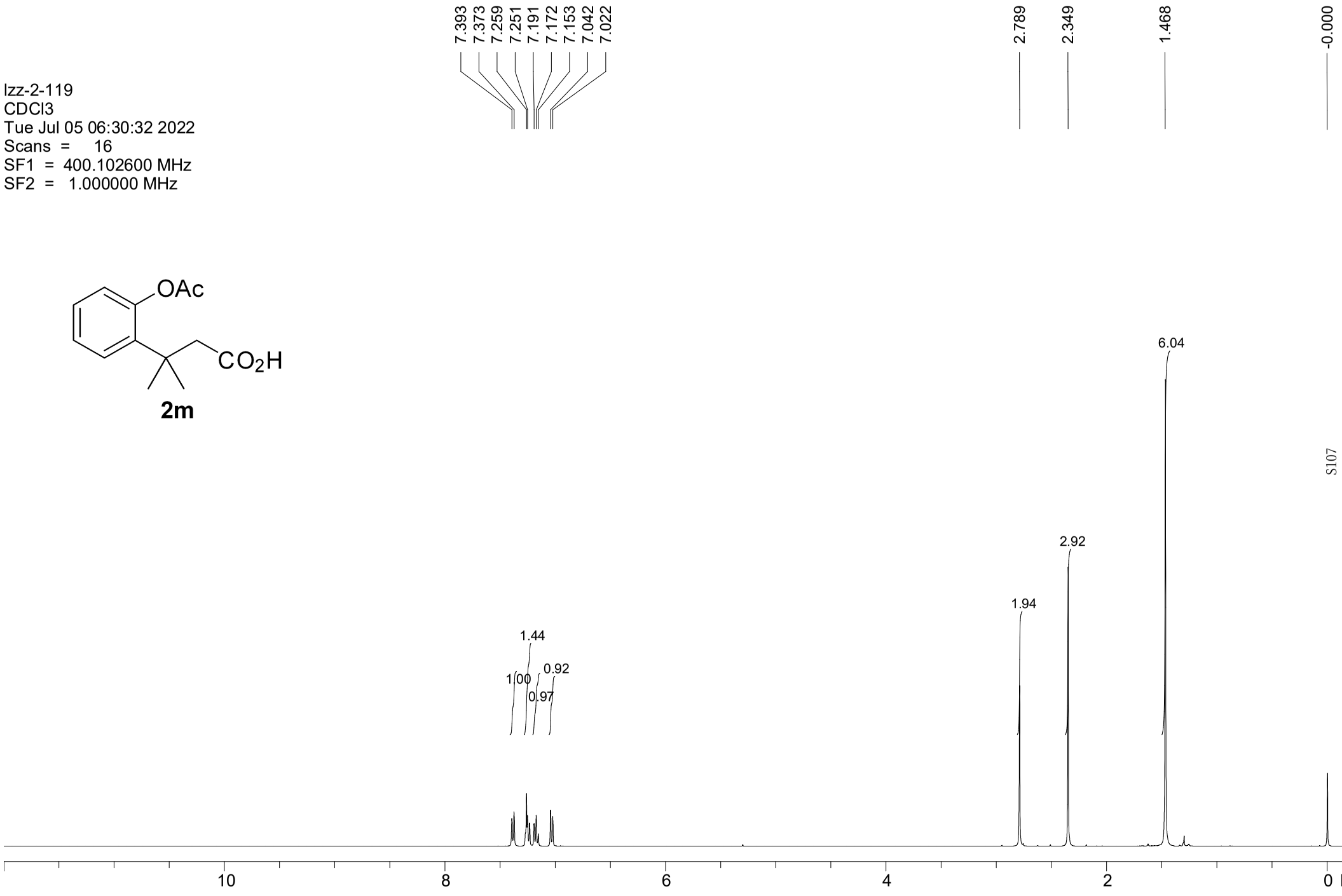
lzz-2-117-C
Jun 26 2022
Scans = 244
Solvent = cdcl3
SF1 = 100.519485 MHz
SF2 = 399.717072 MHz

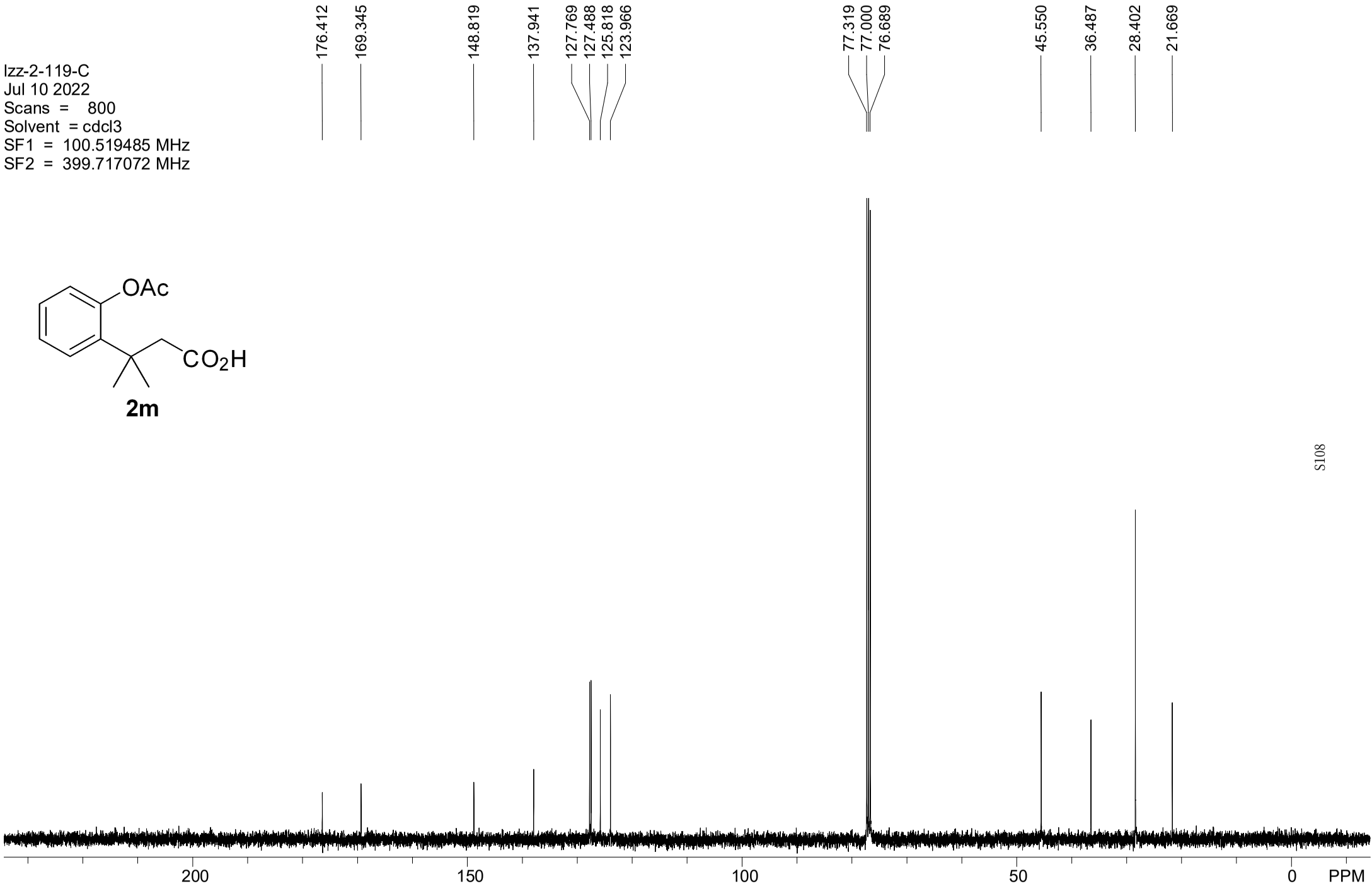
169.299
149.054
138.890
128.042
126.987
125.651
123.966
77.319
77.000
76.681
60.474
44.389
36.745
29.123
25.897
21.654
18.185
-5.416

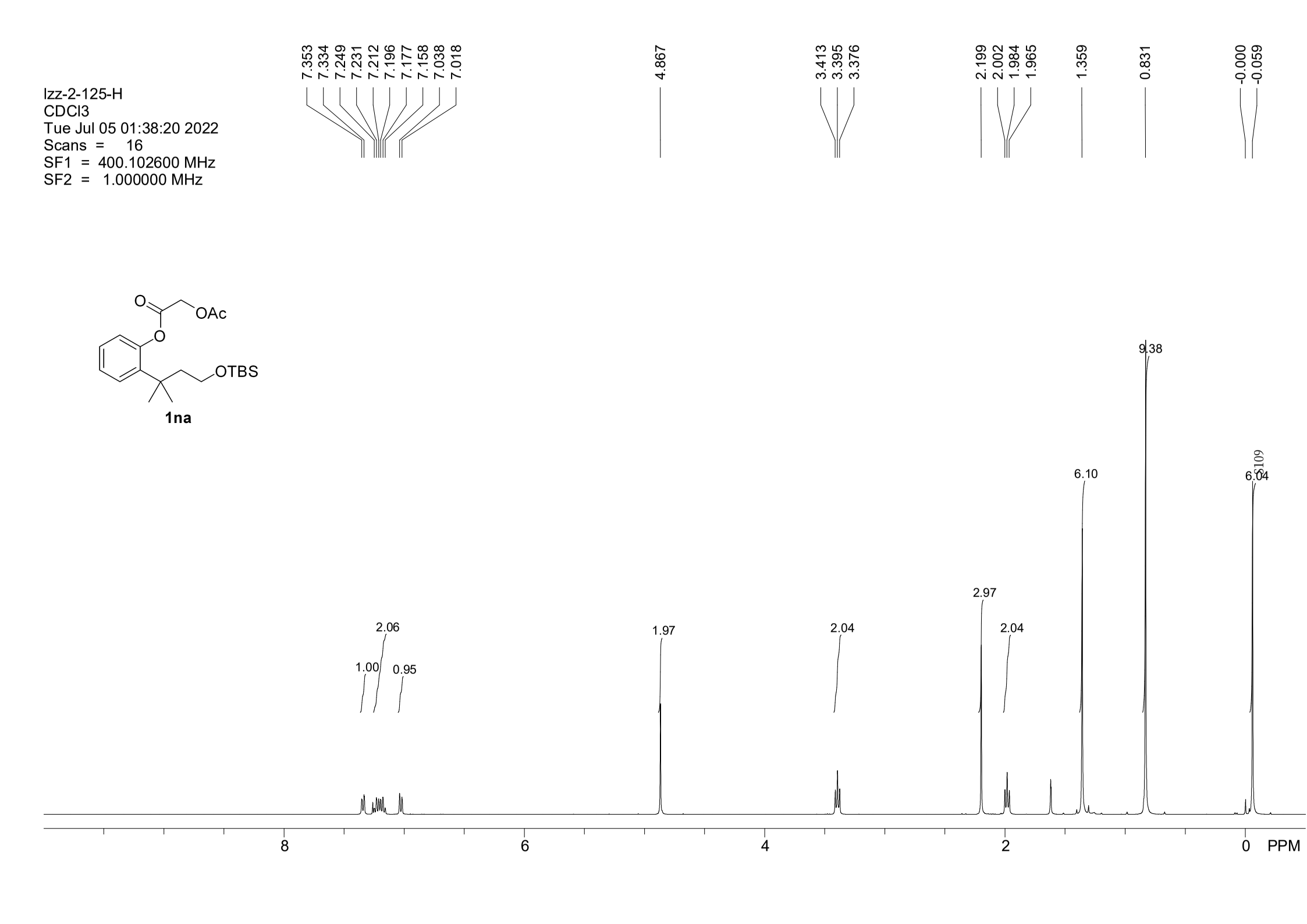


1ma

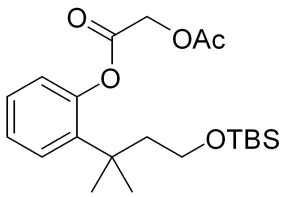
200 150 100 50 0 PPM


7.393
7.373
7.259
7.251
7.191
7.172
7.153
7.042
7.022
2.789
2.349
1.468
-0.000
lzz-2-119
CDCl3
Tue Jul 05 06:30:32 2022
Scans = 16
SF1 = 400.102600 MHz
SF2 = 1.000000 MHz
OAc
CO2H
2m
S107
6.04
2.92
1.94
1.44
0.92
1.00
0.97
10
8
6
4
2
0 PPM

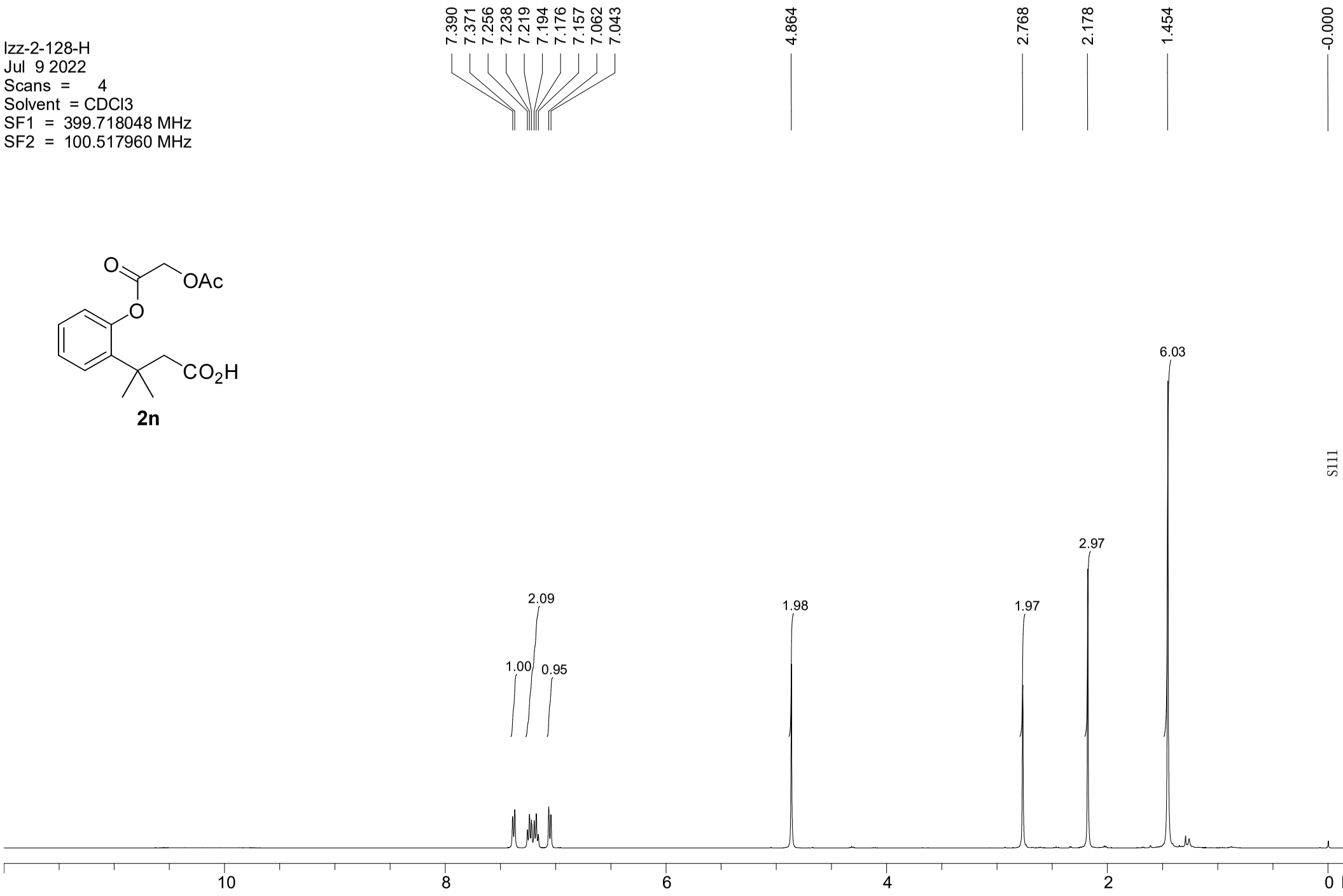

176.412
169.345
148.819
137.941
127.769
127.488
125.818
123.966
77.319
77.000
76.689
45.550
36.487
28.402
21.669
lzz-2-119-C
Jul 10 2022
Scans = 800
Solvent = cdcl3
SF1 = 100.519485 MHz
SF2 = 399.717072 MHz
OAc
CO2H
2m
S108
200
150
100
50
0
PPM

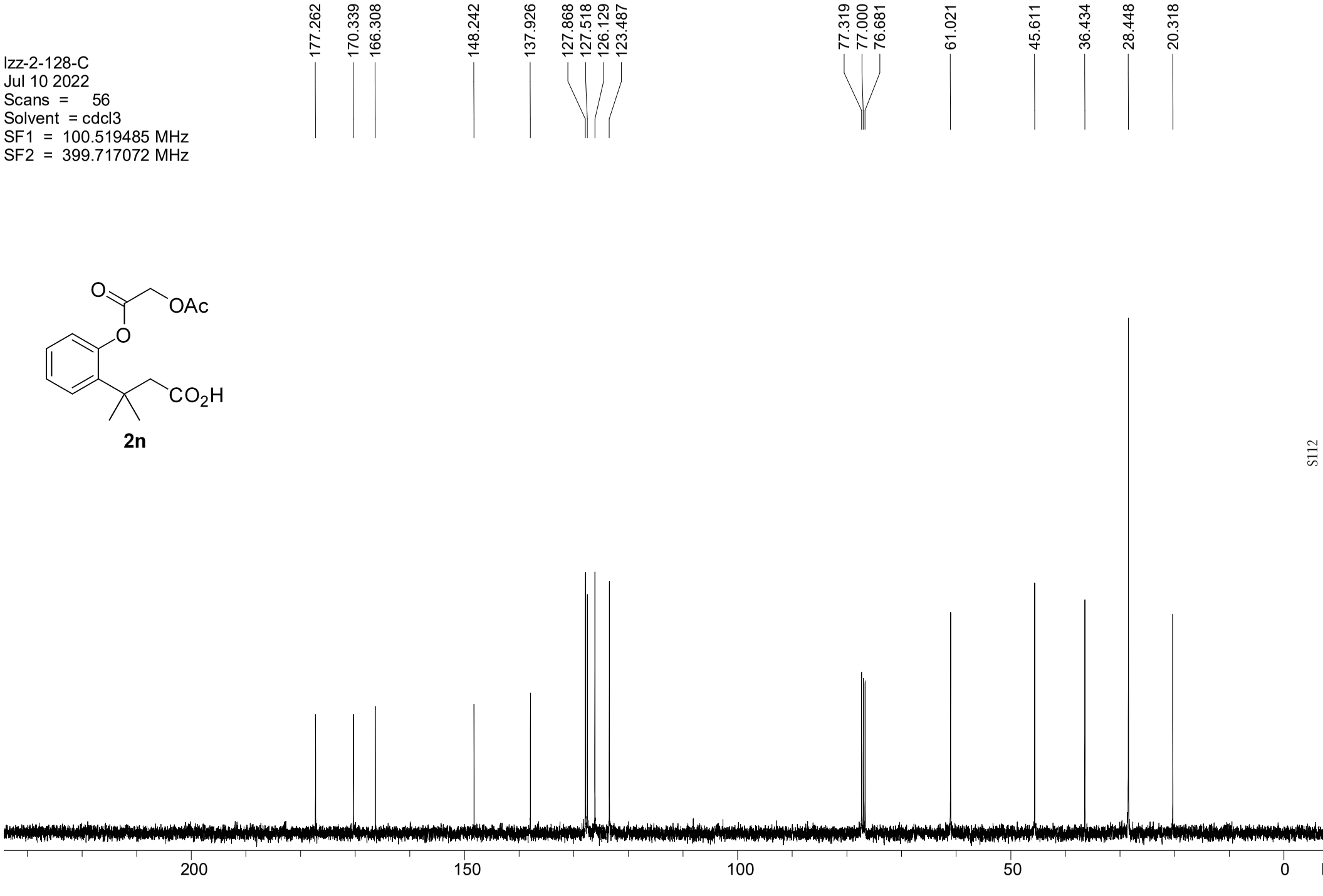

lzz-2-125-H
CDCl3
Tue Jul 05 01:38:20 2022
Scans = 16
SF1 = 400.102600 MHz
SF2 = 1.000000 MHz
7.353
7.334
7.249
7.231
7.212
7.196
7.177
7.158
7.038
7.018
4.867
3.413
3.395
3.376
2.199
2.002
1.984
1.965
1.359
0.831
-0.000
-0.059
O
OAc
O
OTBS
1na
9.38
6.10
6.09
6.04
2.97
2.06
1.97
2.04
2.04
1.00
0.95
8
6
4
2
0
PPM

lzz-2-125-C
CDCl3
Tue Jul 05 01:54:32 2022
Scans = 240
SF1 = 100.617798 MHz
SF2 = 1.000000 MHz



1na


lzz-2-128-H
Jul 9 2022
Scans = 4
Solvent = CDCl3
SF1 = 399.718048 MHz
SF2 = 100.517960 MHz
7.390
7.371
7.256
7.238
7.219
7.194
7.176
7.157
7.062
7.043
4.864
2.768
2.178
1.454
-0.000
O
OAc
O
CO2H
2n
6.03
2.97
2.09
1.98
1.97
1.00
0.95
S111
10
8
6
4
2
0 PPM


177.262
170.339
166.308
148.242
137.926
127.868
127.518
126.129
123.487
77.319
77.000
76.681
61.021
45.611
36.434
28.448
20.318
Izz-2-128-C
Jul 10 2022
Scans = 56
Solvent = cdcl3
SF1 = 100.519485 MHz
SF2 = 399.717072 MHz
O
OAc
O
CO2H
2n
S112
200
150
100
50
0
PPM