Electronic Supplementary Information

# A Solvent-Free Catalytic Protocol for Achmatowicz Rearrangement

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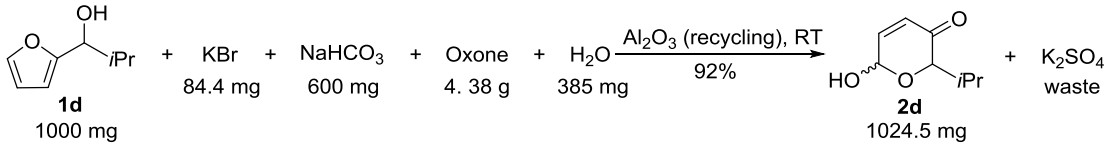
# General Information

Reactions were carried out in a glassware with vigorous stirring at RT, and no special operation was needed. $Al_2O_3$ (Merck KGaA, 0.063–0.200 mm, pH = 6.8–7.8) was equilibrated at 120 °C for 48 h before use. Oxone, KBr and $NaHCO_3$ were all ground to be fine powder before use. Dichloromethane was freshly distilled before use from calcium hydride ($CaH_2$). Solvents used in column chromatography were used as received from commercial suppliers without prior purification. Reactions were monitored by thin-layer chromatography (TLC, 0.25 mm) on pre-coated silica gel plates. Flash chromatography was performed with silica gel 60 (particle size 0.040−0.062 mm). $^1$H- and $^{13}$C-NMR spectra were recorded on a 400 MHz spectrometer (400 MHz for $^1$H, 100 MHz for $^{13}$C). Chemical shifts are reported in parts per million (ppm) as values relative to the internal chloroform (7.26 ppm for $^1$H and 77.16 ppm for $^{13}$C), methanol (3.31 ppm for $^1$H and 49.00 ppm for $^{13}$C), and DMSO (2.5 ppm for $^1$H and 39.52 ppm for $^{13}$C). Abbreviations for signal coupling are as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet.

# E-Factor Analysis

**Example A (this work):**



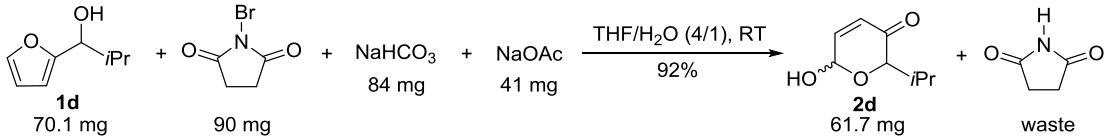

Total amount of reactants: 1000 mg + 84.4 mg + 600 mg + 4380 mg + 385 mg = 6449.4 mg

Amount of final product: 1024.5 mg

Amount of waste: 6449.4 mg – 1024.5 mg = 5424.9 mg

E-Factor = Amount of waste/Amount of product = 5424.9/1024.5 = 5.30

**Example B (**Z. Li and R. Tong, *J. Org. Chem.*., 2016, 81, 4847-4855**):**



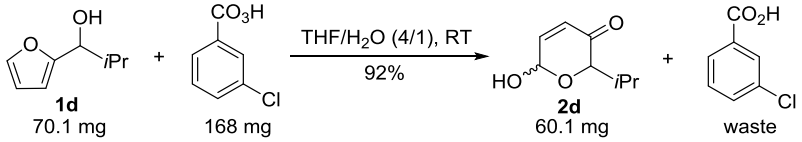

Total amount of reactants: 70.1 mg + 90 mg + 84 mg + 41 mg = 285.1 mg

Amount of final product: 61.7 mg

Amount of waste: 285.1 mg – 61.7 mg = 223.4 mg

E-Factor = Amount of waste/Amount of product = 223.4/61.7 = 3.62

**Example C (**Z. Li and R. Tong, *J. Org. Chem.*., 2016, 81, 4847-4855**):**




Total amount of reactants: 70.1 mg + 168 mg = 238.1 mg

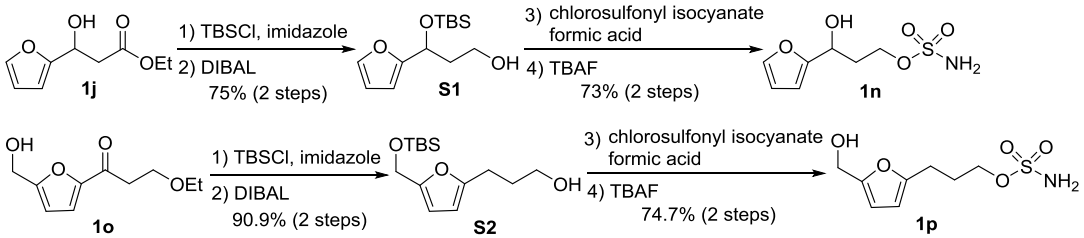
Amount of final product: 60.1 mg

Amount of waste: 238.1 mg – 60.1 mg = 178 mg

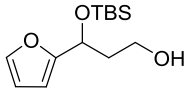
E-Factor = Amount of waste/Amount of product = 178/60.1 = 2.96

# Full Experimental Details and Spectroscopic Data

Substrates **1b**–**g**, **1j**–**m**, **1o, 1t, 1x**, **1z** and **1ab**, were prepared according to the published procedure[1-12]. The $^{1}$H- and $^{13}$C-NMR data of the above substrates were identical to the published literature, which were listed as follows. General Procedure A for the synthesis of substrates **1n** and **1p**; General Procedure B for the synthesis of substrates **1y**, **1aa** and **1ac**; General procedure C for the synthesis of substrates **1r**–**s**, **1u**–**w**; General procedure D for the synthesis of substrates **1q**, **1h**–**i**.



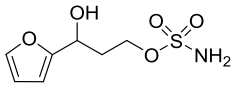

**General Procedure A**: To a stirred solution of the compound **1j**[3] (1.77 g, 9.61 mmol) in anhydrous DCM (40 mL) were added imidazole (1.3 g, 19.2 mmol) and TBSCl (2.17 g, 14.4 mmol) at 0 ºC. After completion of the addition, the reaction mixture was allowed to warm to room temperature and stirred for 12 h. The reaction was quenched by addition of water (100 mL). The organic fractions were collected, and the aqueous phase was extracted with DCM (3 × 30 mL). The combined organic fractions were washed with saturated aqueous $NH_4Cl$ solution and brine, dried over $Na_2SO_4$, filtered, and concentrated under reduced pressure. The crude protection product was used for the next step without further purification. To a stirred solution of the above residue in THF (80 mL) was added DIBAL (40 mL, 1M in hexane, 40 mmol) dropwise at 0 ºC. After completion of the addition, the reaction mixture was stirred for 0.5 h at the same temperature. The reaction was quenched by saturated aqueous $NH_4Cl$ solution (200 mL), and the residue was extracted with EtOAc (3 × 50 mL). The combined organic fractions were washed with brine, dried over $Na_2SO_4$, filtered and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography (EtOAc/hexane = 1:2) to give the alcohol **S1**.




**S1**: colorless oil, 1.33 g, 75% yield for 2 steps. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 7.34 (d, *J* = 1.0 Hz, 1H), 6.31 (dd, *J* = 3.2, 1.8 Hz, 1H), 6.23–6.16 (m, 1H), 4.97 (dd, *J* = 7.4, 4.6 Hz, 1H), 3.84–3.66 (m, 2H), 2.15–1.95 (m, 2H), 0.87 (s, 9H), 0.07 (s, 3H), -0.09 (s, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 156.4, 141.6, 110.2, 106.3, 67.8, 60.3, 38.8, 25.9, 18.2, -4.9, -5.2.

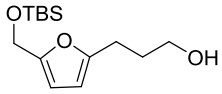
To a stirred solution of chlorosulfonyl isocyanate (0.54 mL, 6.2 mmol) in anhydrous DCM (2 mL) was added formic acid (0.23 mL, 6.2 mmol) at 0 °C. After completion of the addition,

the resulting mixture was allowed to stir for an additional 0.5 h. The above mixture was then added to a solution of **S1** (0.53 g, 2.07 mmol) and pyridine (0.75 mL, 9.3 mmol) in THF (10 mL), and the mixture was stirred at 0 °C for 0.5 h. The reaction was quenched by addition of water (100 mL), and the aqueous phase was extracted with EtOAc (3 × 30 mL). The combined organic fractions were washed with brine, dried over $Na_2SO_4$, filtered and concentrated under reduced pressure. The crude product was used for the next step without further purification. To a stirred solution of the above residue in THF (10 mL) was added TBAF (1.08 g, 4.14 mmol), and the reaction mixture was stirred for 0.5 h at room temperature. Then the mixture was concentrated and purified by flash column chromatography (EtOAc/hexane = 3:1) to give the desired compound **1n**.

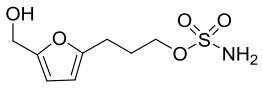



**1n**: colorless oil, 0.33 g, 73% yield for 2 steps. $^1$H-NMR (400 MHz, DMSO) δ: 7.59–7.58 (m, 1H), 7.45 (s, 2H), 6.40 (dd, *J* = 3.2, 1.8 Hz, 1H), 6.29 (d, *J* = 3.2 Hz, 1H), 5.49 (s, 1H), 4.74–4.60 (m, 1H), 4.18–4.07 (m, 2H), 2.09–2.04 (m, 2H). $^{13}$C-NMR (100 MHz, DMSO) δ: 157.6, 142.4, 110.7, 106.1, 66.5, 62.8, 35.2. IR (KBr) 3282.9, 2928.7, 1627.4, 1562.5, 1353.8, 1174.6, 1074.0, 999.7, 918.4, 742.8 cm$^{-1}$; HRMS (CI$^+$) (m/z) calcd. for $C_7H_{11}NO_5S$ $[M]^+$ 221.0352; found 221.0354.

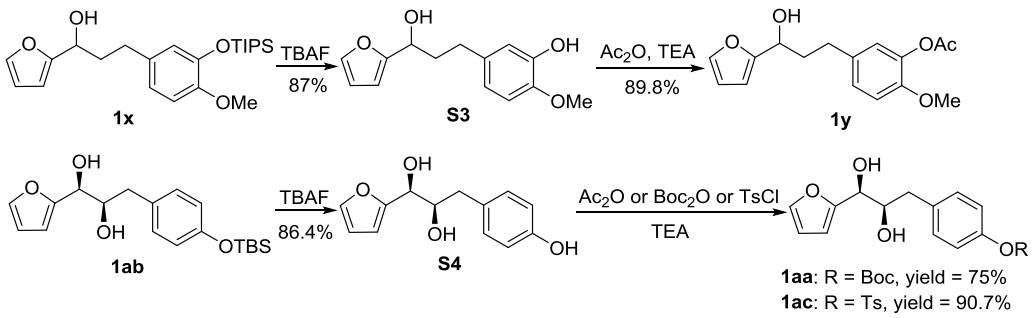
Substrate **1p** were synthesized from **1o**[11] (0.4 g, 2 mmol), and the detailed procedures were carried out as above.



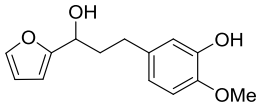

**S2**: colorless oil, 0.49 g, 90.9% for 2 steps. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 6.11 (d, *J* = 3.1 Hz, 1H), 5.93 (d, *J* = 3.1 Hz, 1H), 4.58 (s, 2H), 3.68 (t, *J* = 6.3 Hz, 2H), 2.71 (t, *J* = 7.5 Hz, 2H), 1.89 (ddd, *J* = 13.8, 7.5, 6.4 Hz, 2H), 0.89 (s, 9H), 0.07 (s, 6H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 155.3, 152.7, 108.1, 105.8, 62.2, 58.3, 31.1, 26.0, 24.5, 18.5, -5.1.



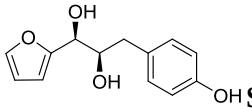

**1p**: colorless oil, 0.32 g, 74.7% for 2 steps. $^1$H-NMR (400 MHz, DMSO) δ: 7.46 (s, 2H), 6.16 (d, *J* = 3.0 Hz, 1H), 6.06 (d, *J* = 3.0 Hz, 1H), 5.12 (t, *J* = 5.7 Hz, 1H), 4.33 (d, *J* = 5.7 Hz, 2H), 4.07 (t, *J* = 6.4 Hz, 2H), 2.67 (t, *J* = 7.6 Hz, 2H), 2.07–1.82 (m, 2H). $^{13}$C-NMR (100 MHz, DMSO) δ: 154.4, 154.1, 108.0, 106.4, 68.6, 56.1, 27.5, 24.1. IR (KBr) 3282.6, 2939.1, 1562.4, 1446.7, 1356.8, 1174.9, 931.3, 795.3, 753.3 cm$^{-1}$; HRMS (CI$^+$) (m/z) calcd. for $C_8H_{13}NO_5S$ $[M]^+$ 235.0509; found 235.0508.




**General Procedure B**: To a stirred solution of **1x**[8] or **1ab**[9] (2.1 mmol) in THF (10 mL) was added TBAF (1.1 g, 4.2 mmol), and the reaction was stirred for 0.5 h at room temperature. Then the mixture was concentrated and purified by flash column chromatography (EtOAc/hexane = 3:2 to EA) to give the desired compound **S3** or **S4**.

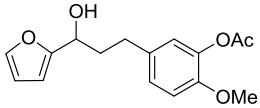



**S3**: colorless oil, 0.45 g, 87%. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 7.37 (dd, *J* = 1.9, 0.8 Hz, 1H), 6.84–6.72 (m, 2H), 6.67 (dd, *J* = 8.2, 2.1 Hz, 1H), 6.33 (dd, *J* = 3.3, 1.8 Hz, 1H), 6.23 (dd, *J* = 3.1, 0.7 Hz, 1H), 5.64 (s, 1H), 4.66 (t, *J* = 6.8 Hz, 1H), 3.86 (s, 3H), 2.73–2.55 (m, 2H), 2.19 – 2.08 (m, 2H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 156.6, 145.5, 144.9, 142.0, 134.8, 119.8, 114.8, 110.7, 110.2, 106.0, 66.9, 56.0, 37.0, 31.1.



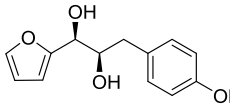

**S4**: colorless oil, 0.42 g, 86.4%. $^{1}$H-NMR (400 MHz, DMSO) δ: 9.11 (s, 1H), 7.56 (dd, *J* = 1.9, 0.8 Hz, 1H), 7.00–6.92 (m, 2H), 6.69–6.58 (m, 2H), 6.40 (dd, *J* = 3.2, 1.8 Hz, 1H), 6.30 (d, *J* = 3.2 Hz, 1H), 5.28 (d, *J* = 5.6 Hz, 1H), 4.62 (d, *J* = 5.9 Hz, 1H), 4.39–4.29 (m, 1H), 3.77 (dd, *J* = 8.9, 4.7 Hz, 1H), 2.57 (dd, *J* = 13.8, 4.3 Hz, 1H), 2.40 (dd, *J* = 13.8, 8.3 Hz, 1H). $^{13}$C-NMR (100 MHz, DMSO) δ: 156.7, 155.8, 142.0, 130.5, 130.0, 115.2, 110.6, 107.1, 74.5, 69.9, 38.6.

To a stirred solution of the compound **S3** or **S4** (0.8 mmol) in anhydrous THF (5 mL) were added TEA (0.45 mL, 3.2 mmol) and $Ac_2O$, $Boc_2O$ or Ts (1.3 mmol) at 0 ºC. After completion of the addition, the reaction mixture was allowed to stir at 0 ºC for 0.5 h. The reaction was quenched by addition of water (50 mL). The mixture was extracted with EtOAc (3 × 30 mL). The combined organic fractions were washed with brine, dried over $Na_2SO_4$, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography (EtOAc/hexane = 1:3 to 1:1) to give the product **1y**, **1aa** or **1ac**.

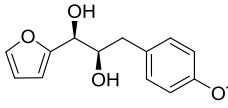



**1y**: colorless oil, 0.21 g, 89.8%. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 7.37 (dd, *J* = 1.9, 0.8 Hz, 1H), 7.02 (dd, *J* = 8.3, 2.2 Hz, 1H), 6.88 (dd, *J* = 5.3, 3.1 Hz, 2H), 6.33 (dd, *J* = 3.3, 1.8 Hz, 1H), 6.23 (d, *J* = 3.2 Hz, 1H), 4.66 (t, *J* = 6.8 Hz, 1H), 3.80 (s, 3H), 2.74–2.60 (m, 2H), 2.30 (s, 3H), 2.21–2.04 (m, 2H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 169.2, 156.5, 149.3, 142.0, 139.6, 134.1, 126.6, 122.8, 112.4, 110.2, 106.0, 66.8, 56.0, 36.9, 30.7, 20.7. IR (KBr) 3420.0, 2936.4, 2847.2, 1759.3, 1509.7, 1439.5, 1369.3, 1266.1, 1198.9, 1120.4,

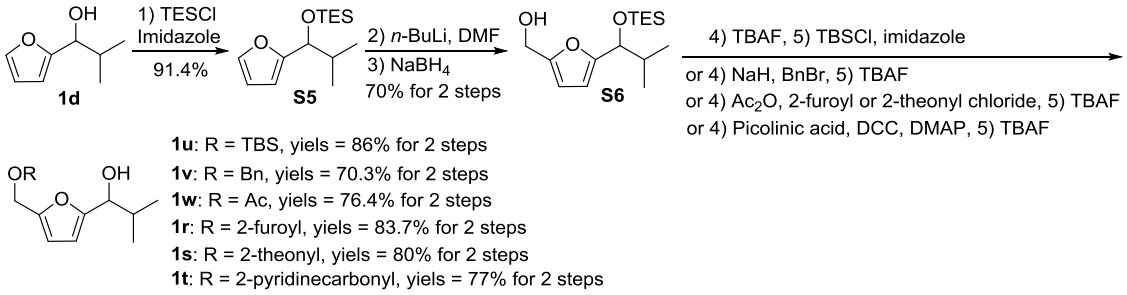
1012.0, 811.0, 739.7 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{16}H_{18}O_5$ $[M]^+$ 290.1149; found 290.1143.



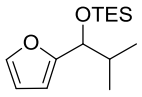

**1aa**: colorless oil, 0.2 g, 75%. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 7.41 (dd, *J* = 1.7, 0.9 Hz, 1H), 7.22 (d, *J* = 8.6 Hz, 2H), 7.08 (d, *J* = 8.6 Hz, 2H), 6.43–6.34 (m, 2H), 4.54 (d, *J* = 5.9 Hz, 1H), 4.13 (dt, *J* = 8.3, 5.0 Hz, 1H), 2.81–2.69 (m, 2H), 1.56 (s, 9H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 153.1, 151.4, 149.1, 141.8, 134.7, 129.8, 120.7, 109.8, 107.4, 82.9, 73.5, 69.5, 38.1, 27.1. IR (KBr) 3388.3, 2981.9, 2925.9, 1749.3, 1506.1, 1373.9, 1266.0, 1145.5, 1014.0, 828.1, 743.4 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{18}H_{22}O_6$ $[M]^+$ 334.1411; found 334.1425.




**1ac**: colorless oil, 0.28 g, 90.7%. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 7.71 (d, *J* = 8.3 Hz, 2H), 7.43–7.28 (m, 3H), 7.14 (d, *J* = 8.5 Hz, 2H), 6.90 (d, *J* = 8.5 Hz, 2H), 6.44–6.32 (m, 2H), 4.51 (d, *J* = 5.9 Hz, 1H), 4.12 (ddd, *J* = 7.9, 5.8, 4.7 Hz, 1H), 2.81–2.63 (m, 2H), 2.45 (s, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 153.0, 147.6, 144.7, 141.8, 136.4, 131.8, 129.9, 129.1, 127.9, 121.7, 109.8, 107.4, 73.4, 69.6, 38.0, 21.1. IR (KBr) 3388.7, 2922.7, 1656.1, 1598.1, 1500.9, 1365.7, 1159.6, 1090.4, 1014.5, 863.9, 739.8 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{20}H_{20}O_6S$ $[M]^+$ 388.0975; found 388.0979.



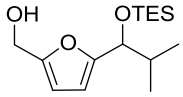

**General Procedure C**: To a stirred solution of the compound **1d**[2] (2.8 g, 20 mmol) in anhydrous DCM (100 mL) were added imidazole (2.04 g, 30 mmol) and TESCl (4.03 mL, 24 mmol) at 0 ºC. After completion of the addition, the reaction mixture was allowed to warm to room temperature and stirred for 0.5 h. The reaction was quenched by addition of water (100 mL). The organic fractions were collected, and the aqueous phase was extracted with DCM (3 × 50 mL). The combined organic fractions were washed with saturated aqueous $NH_4Cl$ solution and brine, dried over $Na_2SO_4$ filtered and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography (EtOAc/hexane = 1:10) to give the silyl ether **S5**.




**S5**: colorless oil, 4.65 g, 91.4%. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 7.33–7.31 (m, 1H), 6.29 (dd, *J* = 3.2, 1.8 Hz, 1H), 6.15 (d, *J* = 3.3 Hz, 1H), 4.29 (d, *J* = 7.3 Hz, 1H), 2.03 (dq, *J* = 13.7, 6.8 Hz, 1H), 0.97 (d, *J* = 6.6 Hz, 3H), 0.88 (t, *J* = 7.9 Hz, 9H), 0.77 (d, *J* = 6.7

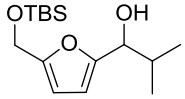
Hz, 3H), 0.52 (q, *J* = 7.9 Hz, 6H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 156.9, 141.2, 110.0, 106.5, 74.1, 34.5, 18.9, 18.8, 6.9, 4.8.

To a stirred solution of the compound **S5** (1.53 g, 6 mmol) in anhydrous THF (12 mL) was added *n*-BuLi (2.5 M, 2.64 mL, 6.6 mmol) dropwise at –78 °C. After completion of the addition, the reaction mixture was allowed to warm to room temperature and stirred for 0.5 h. The reaction was then cooled to –78 °C, followed by the addition of DMF (0.93 mL, 12 mmol). After completion of the addition, the reaction mixture was allowed to warm to room temperature and stirred for 2.5 h. The reaction was quenched by addition of water (100 mL). The aqueous phase was extracted with EtOAc (3 × 50 mL). The combined organic fractions were washed with saturated aqueous $NH_4Cl$ solution and brine, dried over $Na_2SO_4$, filtered and concentrated under reduced pressure. The crude protection product was used for the next step without further purification. To a stirred solution of the above residue in MeOH (30 mL) was added $NaBH_4$ (0.45 g, 12 mmol) dropwise at 0 °C. After completion of the addition, the reaction mixture was stirred for 0.5 h at the same temperature. The reaction was quenched by addition of water (150 mL), and the mixture was extracted with EtOAc (3 × 50 mL). The combined organic fractions were washed with brine, dried over $Na_2SO_4$, filtered and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography (EtOAc/hexane = 1:2) to give the alcohol **S6**.




**S6**: colorless oil, 1.2 g, 70% for 2 steps. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 6.22 (d, *J* = 3.1 Hz, 1H), 6.12 (d, *J* = 3.1 Hz, 1H), 4.59 (s, 2H), 4.30 (d, *J* = 7.2 Hz, 1H), 2.05 (dq, *J* = 13.7, 6.8 Hz, 1H), 1.00 (d, *J* = 6.7 Hz, 3H), 0.91 (t, *J* = 7.9 Hz, 9H), 0.81 (d, *J* = 6.8 Hz, 3H), 0.56 (q, *J* = 7.9 Hz, 6H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 157.1, 152.9, 108.3, 107.3, 74.2, 57.8, 34.4, 18.9, 18.7, 6.9, 4.8.

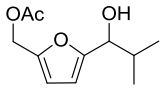
To a stirred solution of the compound **S6** (0.23 g, 0.8 mmol) in anhydrous THF (5 mL) was added TBAF (0.52 g, 2 mmol). After completion of the addition, the reaction mixture was allowed to warm to room temperature and stirred for 0.5 h. Then the mixture was concentrated and purified by flash column chromatography (EtOAc/hexane = 1:1) to give the crude residue. To a stirred solution of the above residue in anhydrous DCM (8 mL) were added imidazole (82 mg, 1.2 mmol) and TBSCl (0.14 g, 0.96 mmol) at 0 ºC. After completion of the addition, the reaction mixture was allowed to warm to room temperature and stirred for 0.5 h. The reaction was quenched by addition of water (50 mL). The organic fractions were collected, and the aqueous phase was extracted with DCM (3 × 10 mL). The combined organic fractions were washed with saturated aqueous $NH_4Cl$ solution and brine, dried over $Na_2SO_4$, filtered and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography (EtOAc/hexane = 1:5) to give the alcohol **1u**.



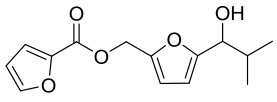

**1u**: colorless oil, 0.2 g, 86% for 2 steps. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 6.16 (q, *J* = 3.2 Hz, 2H), 4.61 (s, 2H), 4.34 (d, *J* = 7.0 Hz, 1H), 2.09 (dq, *J* = 13.6, 6.8 Hz, 1H),

1.01 (d, $J$ = 6.7 Hz, 3H), 0.90 (s, 9H), 0.87 (d, $J$ = 6.8 Hz, 3H), 0.08 (s, 6H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 155.9, 153.7, 107.9, 107.3, 73.8, 58.3, 33.4, 26.0, 18.9, 18.5, 18.3, -5.1. IR (KBr) 3390.2, 2944.3, 2864.8, 1464.0, 1374.7, 1252.3, 1072.6, 1014.3, 834.3, 776.8 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{15}H_{27}O_3Si$ $[M-H]^+$ 283.1724; found 283.1722.

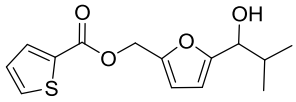
To a stirred solution of the compound **S6** (0.23 g, 0.8 mmol) in anhydrous DCM (5 mL) were added TEA (0.33 mL, 2.4 mmol), DMAP (9.8 mg, 0.08 mmol) and $Ac_2O$, 2-furoyl chloride or 2-theonyl chloride (1.2 mmol). After completion of the addition, the reaction mixture was stirred at room temperature for 0.5 h. The reaction was quenched by addition of water (50 mL). The organic fractions were collected, and the aqueous phase was extracted with DCM (3 × 10 mL). The combined organic fractions were washed with saturated aqueous $NH_4Cl$ solution and brine, dried over $Na_2SO_4$, filtered and concentrated under reduced pressure. The crude protection product was used for the next step without further purification. To a stirred solution of the above residue in THF (10 mL) was added TBAF (0.42 g, 1.6 mmol). After completion of the addition, the reaction mixture was stirred for 2 h at room temperature. The reaction was quenched by addition of water (50 mL). The aqueous phase was extracted with EtOAc (3 × 20 mL). The combined organic fractions were washed with brine, dried over $Na_2SO_4$, filtered and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography (EtOAc/hexane = 1:2) to give the alcohol **1w**, **1r** or **1s**.




**1w**: colorless oil, 0.13 g, 76.4% for 2 steps. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 6.34 (d, $J$ = 3.2 Hz, 1H), 6.19 (d, $J$ = 3.2 Hz, 1H), 5.01 (s, 2H), 4.36 (d, $J$ = 6.9 Hz, 1H), 2.21–2.01 (m, 4H), 1.00 (d, $J$ = 6.7 Hz, 3H), 0.87 (d, $J$ = 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 170.8, 157.2, 148.7, 111.4, 107.5, 73.6, 58.3, 33.4, 21.0, 18.9, 18.2. IR (KBr) 3432.4, 2962.4, 2877.2, 1733.6, 1444.8, 1373.7, 1230.0, 1015.3, 794.5 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{11}H_{16}O_4$ $[M]^+$ 212.1043; found 212.1051.



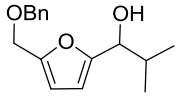

**1r**: colorless oil, 0.18 g, 83.7% for 2 steps. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 7.59 (s, 1H), 7.20 (d, $J$ = 3.5 Hz, 1H), 6.51 (dd, $J$ = 3.6, 1.7 Hz, 1H), 6.44 (d, $J$ = 3.2 Hz, 1H), 6.23 (d, $J$ = 3.2 Hz, 1H), 5.27 (s, 2H), 4.39 (d, $J$ = 6.9 Hz, 1H), 2.15–2.09 (m, 1H), 1.02 (d, $J$ = 6.7 Hz, 3H), 0.89 (d, $J$ = 6.7 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 158.3, 157.3, 148.2, 146.5, 144.3, 118.4, 111.9, 111.9, 107.5, 73.5, 58.4, 33.3, 18.7, 18.0. IR (KBr) 3433.2, 2963.6, 2877.5, 1717.4, 1575.6, 1472.0, 1392.0, 1294.5, 1175.8, 1112.4, 1017.9, 941.8, 763.0 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{14}H_{16}O_5$ $[M]^+$ 264.0992; found 264.0992.




**1s**: colorless oil, 0.18 g, 80% for 2 steps. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 7.82 (d, $J$ = 3.7 Hz, 1H), 7.57 (d, $J$ = 5.0 Hz, 1H), 7.12–7.09 (m, 1H), 6.44 (d, $J$ = 3.2 Hz, 1H), 6.24 (d, $J$ = 3.2 Hz, 1H), 5.26 (s, 2H), 4.40 (d, $J$ = 6.8 Hz, 1H), 2.17–2.07 (m,

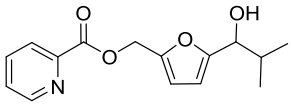
1H), 1.03 (d, *J* = 6.7 Hz, 3H), 0.89 (d, *J* = 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 161.9, 157.2, 148.4, 133.8, 133.4, 132.7, 127.8, 111.6, 107.5, 73.5, 58.7, 33.3, 18.7, 18.1. IR (KBr) 3473.4, 2964.3, 2876.4, 1705.5, 1523.9, 1417.9, 1369.0, 1266.0, 1083.2, 1022.2, 927.4, 753.3 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{14}H_{16}O_4S$ $[M]^+$ 280.0764; found 280.0772.

To a stirred solution of the compound **S6** (0.23 g, 0.8 mmol) in anhydrous THF (5 mL) were added 60% NaH (64 mg, 1.6 mmol). After completion of the addition, the reaction mixture was stirred at 0 ºC for 10 min. Then BnBr (0.19 mL, 1.6 mmol) was added, and the mixture was moved to 50 ºC and stirred for 2 h. The reaction was poured into ice water and extracted with EtOAc (3 × 20 mL). The combined organic fractions were washed with saturated aqueous $NH_4Cl$ solution and brine, dried over $Na_2SO_4$, filtered, and concentrated under reduced pressure. The crude protection product was used for the next step without further purification. To a stirred solution of the above residue in THF (10 mL) was added TBAF (0.42 g, 1.6 mmol). After completion of the addition, the reaction mixture was stirred for 2 h at room temperature. The reaction was quenched by addition of water (50 mL). The aqueous phase was extracted with EtOAc (3 × 20 mL). The combined organic fractions were washed with brine, dried over $Na_2SO_4$, filtered and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography (EtOAc/hexane = 1:2) to give the alcohol **1v** (yield = 70.3% for two steps) as a colorless oil.

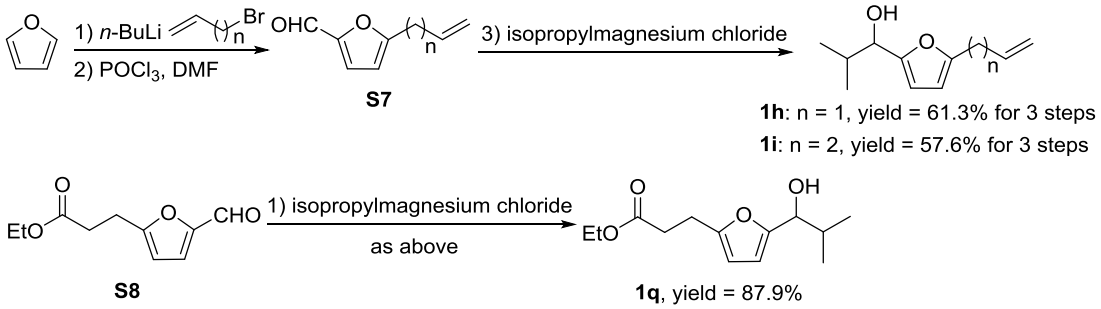



**1v**: colorless oil, 0.15 g, 70.3% for 2 steps. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 7.43–7.27 (m, 5H), 6.27 (d, *J* = 3.1 Hz, 1H), 6.19 (d, *J* = 3.1 Hz, 1H), 4.54 (s, 2H), 4.46 (s, 2H), 4.36 (d, *J* = 7.0 Hz, 1H), 2.12 (dq, *J* = 13.5, 6.8 Hz, 1H), 1.02 (d, *J* = 6.7 Hz, 3H), 0.88 (d, *J* = 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 156.0, 150.3, 137.3, 127.8, 127.3, 127.1, 109.5, 106.5, 72.9, 71.2, 63.3, 32.6, 18.2, 17.5. IR (KBr) 3393.1, 2961.1, 2866.9, 1633.6, 1556.0, 1458.0, 1359.4, 1200.3, 1059.4, 1014.0, 792.9, 739.4 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{16}H_{20}O_3$ $[M]^+$ 260.1407; found 260.1405.

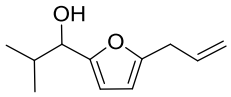
To a stirred solution of the compound **S6** (0.23 g, 0.8 mmol) in anhydrous DCM (5 mL) were added DCC (0.25 g, 1.2 mmol) and DMAP (0.15 g, 1.2 mmol). After completion of the addition, the reaction mixture was stirred at RT for 3 h. The reaction was quenched by addition of water (50 mL). The organic fractions were collected, and the aqueous phase was extracted with DCM (3 × 20 mL). The combined organic fractions were washed with saturated aqueous $NH_4Cl$ solution and brine, dried over $Na_2SO_4$, filtered and concentrated under reduced pressure. The crude protection product was used for the next step without further purification. To a stirred solution of the above residue in THF (10 mL) was added TBAF (0.42 g, 1.6 mmol). After completion of the addition, the reaction mixture was stirred for 2 h at room temperature. The reaction was quenched by addition of water (50 mL). The aqueous phase was extracted with EtOAc (3 × 20 mL). The combined organic fractions were washed with brine, dried over $Na_2SO_4$, filtered, and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography (EtOAc/hexane = 1:2) to give the alcohol **1t** (yield = 77% for two steps) as a colorless oil.



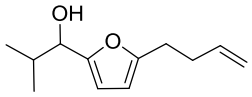
**1t**: colorless oil, 0.17 g, 77% for 2 steps. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 8.74 (s, 1H), 8.12 (d, *J* = 7.9Hz, 1H), 7.82 (t, *J* = 7.8 Hz, 1H), 7.49–7.45 (m, 1H), 6.46 (d, *J* = 2.8 Hz, 1H), 6.21 (t, *J* = 2.6 Hz, 1H), 5.36 (s, 2H), 4.36 (d, *J* = 6.8Hz, 1H), 2.14–2.05 (m, 1H), 1.00 (d, *J* = 6.7 Hz, 3H), 0.87 (d, *J* = 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 164.1, 156.7, 149.3, 147.5, 147.2, 136.4, 126.4, 124.7, 111.5, 106.8, 72.8, 58.7, 32.6, 18.1, 17.4. IR (KBr) 3373.8, 2963.2, 2874.3, 1728.3, 1581.8, 1439.8, 1373.6, 1291.5, 1125.6, 1018.8, 929.9, 749.6 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{15}H_{17}NO_4$ $[M]^+$ 275.1152; found 275.1146.




**General Procedure D**: To a stirred solution of the furan (2.18 mL, 30 mmol) in anhydrous THF (30 mL) was added *n*-BuLi (2.5 M, 13.2 mL, 33 mmol) dropwise at 0 °C. After completion of the addition, the reaction mixture was allowed to stir at 0 °C for 0.5 h. To the above mixture was added allyl bromide or 4-bromo-1-butene (39 mmol). After the addition, the reaction was moved to RT and stirred for 2.5 h (for allyl bromide) or 16 h (for 4-Bromo-1-butene). The mixture was quenched by addition of water (200 mL). The aqueous phase was extracted with EtOAc (3 × 50mL). The combined organic fractions were washed with saturated aqueous $NH_4Cl$ solution and brine, dried over $Na_2SO_4$, filtered and concentrated under reduced pressure. The residue was used for the next step without further purification. To a stirred solution of DMF (2.79 mL, 36 mmol) in dry DCM (20 mL) was added $POCl_3$ (3.4 mL, 36 mmol) dropwise at 0 °C. After completion of the addition, the mixture was stirred at 0 °C for 0.5 h. The above residue was added slowly to the mixture, and the reaction was stirred at 0 °C for 0.5 h. The reaction was quenched by addition of saturated $Na_2CO_3$ solution. The organic fractions were collected, and the aqueous phase was extracted with DCM (3 × 50mL). The combined organic fractions were washed with brine, dried over $Na_2SO_4$, filtered and concentrated under reduced pressure. To a stirred solution of the above residue in anhydrous THF (100 mL) was added isopropylmagnesium chloride (30 mL, 2 M in THF, 60 mmol) dropwise at 0 ºC. After completion of the addition, the reaction mixture was allowed to stir at 0 ºC for 0.5 h. The reaction was quenched by addition of water (500 mL). The aqueous phase was extracted with EtOAc (3 × 100 mL). The combined organic fractions were washed with saturated aqueous $NH_4Cl$ solution and brine, dried over $Na_2SO_4$, filtered and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography (EtOAc/hexane = 1:3) to give the alcohol **1h** (yield = 61.3% for 3 steps) or or **1i** (yield = 57.6% for 3 steps) as a colorless oil.

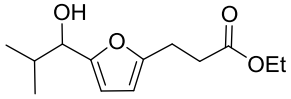


**1h**: colorless oil, 3.31 g, 61.3% for 3 steps. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 6.12 (d, *J* = 3.1 Hz, 1H), 5.97–5.87 (m, 2H), 5.15–5.09 (m, 2H), 4.30 (d, *J* = 7.2 Hz, 1H), 3.37 (dq, *J* = 6.5, 1.3 Hz, 2H), 2.09 (dq, *J* = 13.6, 6.8 Hz, 1H), 1.02 (d, *J* = 6.7 Hz, 3H), 0.86 (d, *J* = 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 154.9, 153.3, 134.0, 117.0, 107.4, 106.1, 73.7, 33.4, 32.7, 19.0, 18.4. IR (KBr) 3427.9, 2965.6, 2877.7, 1641.6, 1558.4, 1463.7, 1377.9, 1266.1, 1176.7, 1007.4, 919.0, 737.0 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{11}H_{16}O_2$ $[M]^+$ 180.1145; found 180.1152.

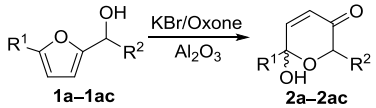


**1i**: colorless oil, 3.36 g, 57.6% for 3 steps. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 6.10 (d, *J* = 3.1 Hz, 1H), 5.93 (d, *J* = 3.2 Hz, 1H), 5.88–5.78 (m, 1H), 5.09–4.95 (m, 2H), 4.29 (d, *J* = 7.2 Hz, 1H), 2.67 (t, *J* = 8 Hz, 2H), 2.38 (dtt, *J* = 8.7, 7.3, 1.5 Hz, 2H), 2.09 (dq, *J* = 13.9, 6.9 Hz, 1H), 1.82 (brs, 1H), 1.02 (d, *J* = 6.7 Hz, 3H), 0.85 (d, *J* = 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 155.0, 154.4, 137.6, 115.4, 107.2, 105.5, 73.8, 33.4, 32.2, 27.7, 18.9, 18.5. IR (KBr) 3385.7, 2961.0, 2920.2, 2876.4, 1641.9, 1561.0, 1455.2, 1378.4, 1009.0, 913.9, 776.8 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{12}H_{18}O_2$ $[M]^+$ 194.1301; found 194.1304.

Substrate **1q** was synthesized from **S8**[11] (0.33 g, 1.68 mmol), and the detailed procedures were described as above.



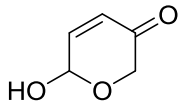
**1q**: colorless oil, 0.35 g, 87.9%. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 6.09 (d, *J* = 3.1 Hz, 1H), 5.94 (d, *J* = 3.1 Hz, 1H), 4.28 (dd, *J* = 7.2, 4.2 Hz, 1H), 4.13 (q, *J* = 7.1 Hz, 2H), 2.95–2.91 (m, 2H), 2.64–2.60 (m, 2H), 2.10–2.02 (m, 1H), 1.24 (t, *J* = 7.2 Hz, 3H), 1.00 (d, *J* = 6.7 Hz, 3H), 0.83 (d, *J* = 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 172.6, 154.9, 153.5, 107.3, 105.9, 73.7, 60.7, 33.4, 32.9, 23.7, 18.9, 18.5, 14.3. IR (KBr) 3464.4, 2969.7, 1728.3, 1376.5, 1265.1, 1186.0, 1019.2, 741.8 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{13}H_{20}O_4$ $[M]^+$ 240.1356; found 240.1365.



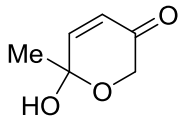

**1a–1ac** **2a–2ac**

**General Procedure E**: Aluminium oxide 90 active neutral (Merck, 0.063–0.200 mm) was equilibrated at 120 °C for 48 h, then it was cooled to RT under nitrogen. To a 5-mL round-bottomed flask was added a magnetic stirrer, a liquid furyl alcohol (0.2 mmol) and the above activated aluminium oxide (120 mg). The resulting mixture was stirred until uniformly free-flowing. To the above mixture was added $H_2O$ (7.2 mg, 0.4 mmol) and KBr (2.38 mg, 0.02 mmol), and the mixture was stirred again to be free-flowing. To the above mixture was added $NaHCO_3$ (16.8 mg, 0.2 mmol) and Oxone (123 mg, 0.2 mmol), the mixture was stirred vigorously for 5 min at RT. Then the solid was transferred into a glass column and eluted by EtOAc to get a crude product. A very pure product for NMR was got by silica gel column chromatography.

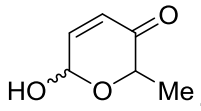
**Notes**: KBr, $NaHCO_3$ and Oxone were all ground to be fine powder before use. For substrate **1f**–**g**, **1h**–**I** and **1l**, another KBr (7.14 mg, 0.06 mmol), $H_2O$ (21.6 mg, 1.2 mmol), aluminium oxide (120 mg), Oxone (123 mg, 0.2 mmol) and $NaHCO_3$ (16.8 mg, 0.2 mmol) were needed.



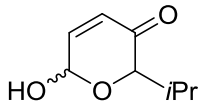
**2a** (EtOAc/hexane = 1:2): colorless oil, 20.8 mg, 91%. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 6.95 (dd, *J* = 10.4, 3.0 Hz, 1H), 6.16 (d, *J* = 10.4 Hz, 1H), 5.63 (dd, *J* = 5.6, 3.0 Hz, 1H), 4.57 (d, *J* = 16.9 Hz, 1H), 4.14 (d, *J* = 17.0 Hz, 1H), 3.41 (d, *J* = 5.4 Hz, 1H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 194.7, 145.8, 128.1, 88.4, 66.8.



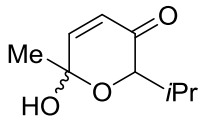
**2b** (EtOAc/hexane = 1:3): colorless oil, 16.9 mg, 66%. $^1$H-NMR (400 MHz, DMSO) δ: 7.03 (d, *J* = 10.3 Hz, 1H), 6.66 (s, 1H), 5.97 (d, *J* = 10.2 Hz, 1H), 4.39 (d, *J* = 16.9 Hz, 1H), 3.99 (dd, *J* = 17.0 Hz, 1H), 1.46 (s, 3H). $^{13}$C-NMR (100 MHz, DMSO) δ: 195.9, 152.1, 125.3, 92.4, 66.2, 27.6.



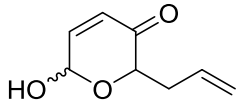
**2c** (EtOAc/hexane = 1:3): colorless oil (dr 7:3), 22.6 mg, 88%. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 7.09–6.74 (m, 1H), 6.14–6.18 (m, *J* = 20.2, 10.3 Hz, 1H), 5.83–5.58 (m, 1H), 4.73 (q, *J* = 6.8 Hz, 0.7 H), 4.25 (q, *J* = 6.7 Hz, 0.3 H), 1.44–1.48 (m, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 197.0, 196.5, 148.1, 144.5, 128.5, 127.23, 91.0, 87.7, 75.2, 70.4, 16.2, 15.3.



**2d** (EtOAc/hexane = 1:3): colorless oil (dr 7:3), 28.7 mg, 92%. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 7.01–6.84 (m, 1H), 6.15–6.08 (m, 1H), 5.68–5.63 (m, 1H), 4.40 (d, *J* = 3.1 Hz, 0.75H), 3.90 (dd, *J* = 3.4, 1.4 Hz, 0.25H), 3.81–3.62 (m, 0.25H), 3.41 (d, *J* = 4.7 Hz, 0.75H), 2.58–2.30 (m, 1H), 1.05–1.01 (m, 3H), 0.93–0.86 (m, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 196.9, 196.4, 148.3, 144.5, 129.6, 128.2, 91.4, 87.7, 83.2, 78.5, 28.9, 28.7, 19.2, 19.1, 16.6, 16.3.

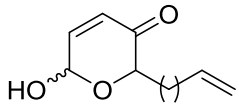


**2e** (EtOAc/hexane = 1:3): colorless oil (dr 9:1), 26.6 mg, 78%. $^1$H-NMR (400 MHz, DMSO) δ: 6.98 (d, *J* = 10.1 Hz, 1H), 6.53 (s, 1H), 5.91 (d, *J* = 10.1 Hz, 1H), 4.24 (d, *J* = 2.8 Hz, 0.9H), 3.97 (d, *J* = 4.1 Hz, 0.1H), 2.31–2.24 (m, 1H), 1.46 (s, 3H), 0.96 (d, *J* = 7.0 Hz, 3H), 0.77 (d, *J* = 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, DMSO) δ: 197.7, 151.4, 125.7, 92.2, 77.7, 28.7, 28.3, 19.4, 16.6.

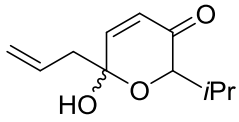


**2f** (EtOAc/hexane = 1:3): colorless oil (dr 7:3), 26.2 mg, 85%. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 7.00–6.85 (m, 1H), 6.18–6.13 (dd, *J* = 18.3, 10.3 Hz, 1H), 5.91–5.78 (m,

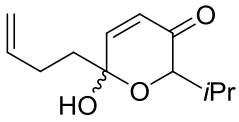
1H), 5.67–5.66 (m, 1H), 5.20–5.09 (m, 2H), 4.67 (dd, *J* = 8.0, 3.8 Hz, 0.7H), 4.17 (ddd, *J* = 8.3, 4.0, 1.2 Hz, 0.3H), 2.77–2.70 (m, 1H), 2.60–2.45 (m, 1H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 195.9, 195.5, 147.9, 144.5, 133.8, 133.7, 128.7, 127.7, 118.1, 117.9, 90.9, 87.9, 78.6, 73.8, 35.2, 34.2.



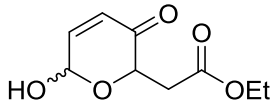
**2g** (EtOAc/hexane = 1:3): colorless oil (dr 7:3), 27.7 mg, 76%. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 6.94–6.87 (m, 1H), 6.16–6.09 (m, 1H), 5.85–5.75 (m, 1H), 5.65–5.64 (m, 1H), 5.04–4.94 (m, 2H), 4.57 (dd, *J* = 8.0, 3.9 Hz, 0.7H), 4.08 (ddd, *J* = 8.3, 4.0, 1.2 Hz, 0.3H), 2.16–2.01 (m, 2H), 1.99–1.90 (m, 1H), 1.80–1.67 (m, 1H), 1.58–1.49 (m, 2H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 196.6, 196.3, 147.8, 144.4, 138.5, 138.4, 128.9, 127.8, 114.99, 114.95, 91.0, 87.8, 78.9, 74.1, 33.6, 30.2, 29.2, 24.5, 24.4.



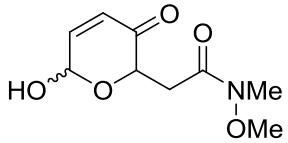
**2h** (EtOAc/hexane = 1:3): colorless oil (dr 9:1), 33 mg, 84%. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 6.77 (d, *J* = 10.1 Hz, 1H), 6.05 (d, *J* = 10.3 Hz, 1H), 5.96–5.87 (m, 1H), 5.31–5.23 (m, 2H), 4.35 (d, *J* = 2.8 Hz, 1H), 2.69 (ddt, *J* = 13.6, 6.2, 1.3 Hz, 1H), 2.53–2.41 (m, 2H), 1.03 (d, *J* = 6.9 Hz, 3H), 0.85 (d, *J* = 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 197.1, 147.3, 131.2, 128.1, 121.2, 93.0, 78.4, 46.0, 28.8, 19.2, 16.2.



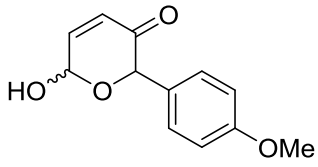
**2i** (EtOAc/hexane = 1:3): colorless oil (dr 9:1), 30.7 mg, 73%. $^1$H-NMR (400 MHz, DMSO) δ: 7.00 (d, *J* = 10.2 Hz, 1H), 5.97 (d, *J* = 10.2 Hz, 1H), 5.91–5.77 (m, 1H), 5.10–4.90 (m, 2H), 4.25 (d, *J* = 2.7 Hz, 1H), 2.34–2.25 (m, 1H), 2.22–1.99 (m, 2H), 1.84–1.77 (m, 2H), 0.97 (d, *J* = 7.0 Hz, 3H), 0.77 (d, *J* = 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, DMSO) δ: 197.64, 150.65, 138.83, 126.66, 115.08, 93.37, 77.63, 28.87, 28.17, 19.44, 16.63.



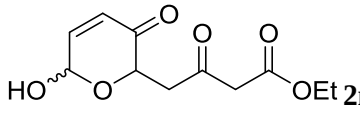
**2j** (EtOAc/hexane = 1:3): colorless oil (dr 7:3), 33.6 mg, 84%. $^1$H-NMR (400 MHz, $CDCl_3$) δ:7.02–6.88 (m, 1H), 6.20–6.13 (m, 1H), 5.72–5.62 (m, 1H), 5.02 (dd, *J* = 7.6, 3.8 Hz, 0.7H), 4.57 (dd, *J* = 7.9, 3.9 Hz, 0.3H), 4.19–4.13 (m, 2H), 3.03–2.97 (m, 1H), 2.83–2.71 (m, 1H), 1.28–1.24 (m, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 195.0, 194.6, 171.1, 148.4, 144.7, 128.5, 127.2, 91.0, 87.8, 75.4, 70.9, 61.3, 61.2, 36.2, 35.4, 14.2



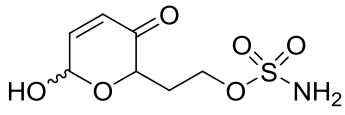
**2k** (EtOAc/hexane = 1:3): colorless oil (dr 7:3), 34.4 mg, 80%. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 6.98–6.90 (m, 1H), 6.20–6.14 (m, 1H), 5.71–5.61 (m, 1H), 5.12 (dd, *J* = 8.2, 3.4 Hz, 0.7H), 4.65 (ddd, *J* = 7.5, 3.8, 1.1 Hz, 0.3H), 3.704–3.700 (m, 3H), 3.19 (s, 3H), 3.14–2.86 (m, 2H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 196.0, 195.4, 171.3, 148.5, 144.9, 128.2, 127.2, 90.7, 87.9, 75.1, 70.6, 61.5, 34.2, 33.0, 32.3.



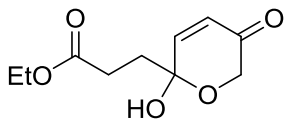
**2l** (EtOAc/hexane = 1:3): colorless oil (dr 7:3), 27.7 mg, 63%. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 7.33–7.27 (m, 2H), 7.05–7.00 (m, 1H), 6.95–6.92 (m, 2H), 6.32–6.23 (m, 1H), 5.83–5.75 (m, 1H), 5.57 (s, 0.75H), 5.09 (s, 0.25H), 3.83 (s, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 194.8, 159.8, 148.0, 144.6, 129.4, 129.3, 127.9, 127.4, 114.0, 91.5, 88.1, 80.8, 55.3.



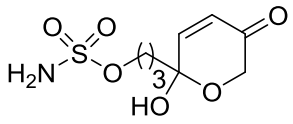
**2m** (EtOAc/hexane = 1:3): colorless oil (dr 7:3), 44.1 mg, 91%. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 7.00–6.91 (m, 1H), 6.21–6.13 (m, 1H), 5.73–5.61 (m, 1H), 5.10 (dd, *J* = 7.6, 3.8 Hz, 0.7H), 4.66 (dd, *J* = 7.8, 3.8 Hz, 0.3H), 4.21 (q, *J* = 7.1 Hz, 2H), 3.55–3.54 (m, 2H), 3.27–3.20 (m, 1H), 2.99–2.92 (m, 1H), 1.30 (t, *J* = 7.1 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 199.9, 199.8, 195.2, 194.7, 167.0, 148.5, 144.7, 128.3, 127.0, 91.0, 87.8, 74.5, 70.0, 65.6, 61.6, 49.8, 49.7, 43.7, 43.0, 14.1. IR (KBr) 3426.0, 2983.7, 2931.0, 1694.8, 1631.6, 1371.8, 1264.3, 1149.7, 1087.1, 1019.1, 936.2, 755.7 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{11}H_{15}O_6$ $[M+H]^+$ 243.0863; found 243.0866.



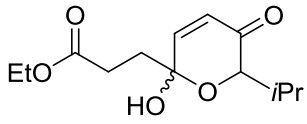
**2n** (EtOAc/hexane = 3:1):colorless oil (dr 7:3), 46 mg, 97%. $^{1}$H-NMR (400 MHz, DMSO) δ: 7.48–7.47 (m, 2H), 7.34–7.02 (m, 2H), 6.12–6.03 (m, 1H), 5.61–5.48 (m, 1H), 4.60 (dd, *J* = 8.3, 4.1 Hz, 0.7H), 4.28 (ddd, *J* = 8.8, 4.0, 1.3 Hz, 0.3H), 4.20–4.11 (m, 2H), 2.27–2.20 (m, 1H), 1.97–1.86 (m, 1H). $^{13}$C-NMR (100 MHz, DMSO) δ: 196.7, 196.5, 151.9, 148.3, 127.7, 126.0, 90.9, 87.2, 74.6, 70.1, 65.8, 65.8, 30.3, 29.6. IR (KBr) 3370.4, 2987.0, 1689.5, 1364.3, 1268.0, 1178.4, 1023.3, 928.3, 755.1 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_7H_{11}NO_6S$ $[M]^+$ 237.0302; found 237.0315.



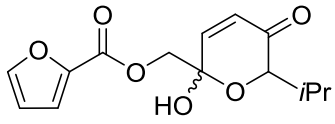
**2o** (EtOAc/hexane = 1:3): colorless oil, 33.8 mg, 79%. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 6.82 (d, *J* = 10.3 Hz, 1H), 6.05 (d, *J* = 10.3 Hz, 1H), 5.18 (s, 1H), 4.60 (d, *J* = 16.8 Hz, 1H), 4.17 (q, *J* = 7.1 Hz, 2H), 4.08 (d, *J* = 16.8 Hz, 1H), 2.82 (ddd, *J* = 17.6, 9.4, 5.3 Hz, 1H), 2.50 (ddd, *J* = 17.6, 6.5, 4.8 Hz, 1H), 2.23–2.04 (m, 2H), 1.27 (t, *J* = 7.1 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 195.3, 175.6, 148.7, 126.4, 92.6, 66.6, 61.6, 34.7, 28.4, 14.2. IR (KBr) 3425.5, 2982.3, 1698.4, 1378.0, 1272.0, 1184.7, 1082.3, 944.9, 867.2, 785.9 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{10}H_{13}O_5$ $[M-H]^+$ 213.0757; found 213.0768.



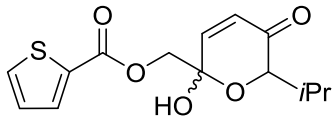
**2p** (EtOAc/hexane = 1:1): colorless oil, 46.7 mg, 93%. $^{1}$H-NMR (400 MHz, DMSO) δ: 7.42 (s, 2H), 7.05 (d, *J* = 10.3 Hz, 1H), 6.74 (s, 1H), 6.04 (d, *J* = 10.3 Hz, 1H), 4.43 (d, *J* = 17.0 Hz, 1H), 4.12–3.92 (m, 3H), 1.91–1.64 (m, 4H). $^{13}$C-NMR (100 MHz, DMSO) δ: 195.8, 151.1, 126.2, 93.6, 69.5, 66.3, 36.4, 23.5. IR (KBr) 3346.1, 2978.5, 1688.0, 1361.1, 1268.3, 1177.9, 971.1, 755.4 cm$^{-1}$; HRMS (CI$^{+}$) (m/z) calcd. for $C_8H_{13}NO_6S$ $[M]^{+}$ 251.0458; found 251.0469.



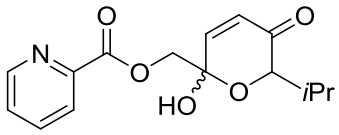
**2q** (EtOAc/hexane = 1:3): colorless oil (dr 9:1), 46.1 mg, 90%. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 6.78 (d, *J* = 10.1 Hz, 1H), 6.01 (d, *J* = 10.1 Hz, 1H), 4.40 (d, *J* = 2.8 Hz, 1H), 4.18 (q, *J* = 7.2 Hz, 2H), 2.90–2.80 (m, 1H), 2.57–2.40 (m, 2H), 2.29–2.08 (m, 2H), 1.28 (t, *J* = 7.1 Hz, 3H), 1.02 (d, *J* = 7.0 Hz, 3H), 0.84 (d, *J* = 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 197.1, 175.3, 147.8, 127.2, 92.5, 78.3, 61.4, 35.5, 28.7, 28.6, 19.2, 16.2, 14.2. IR (KBr) 3427.2, 2971.6, 2936.3, 2876.0, 1691.4, 1458.4, 1376.5, 1269.1, 1182.4, 1037.4, 755.6 cm$^{-1}$; HRMS (CI$^{+}$) (m/z) calcd. for $C_{13}H_{21}O_5$ $[M+H]^{+}$ 257.1384; found 257.1391.



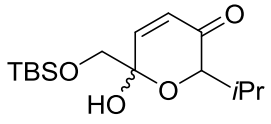
**2r** (EtOAc/hexane = 1:2): colorless oil (dr 9:1), 52.1 mg, 93%. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 7.64 (s, 1H), 7.28–7.23 (m, 1H), 7.03–6.87 (m, 1H), 6.56 (dd, *J* = 3.6, 1.7 Hz, 1H), 6.22–6.15 (m, 1H), 4.80–4.67 (m, 1H), 4.49–4.16 (m, 2H), 2.53–2.42 (m, 1H), 1.04 (d, *J* = 6.9 Hz, 3H), 0.85 (d, *J* = 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 196.3, 158.5, 147.1, 144.0, 143.8, 128.7, 119.1, 112.1, 92.2, 78.6, 68.1, 28.8, 18.9, 15.9. IR (KBr) 3405.2, 2967.7, 2876.2, 1692.0, 1576.3, 1470.5, 1393.9, 1297.4, 1177.8, 1114.8, 1068.9, 929.8, 760.7 cm$^{-1}$; HRMS (CI$^{+}$) (m/z) calcd. for $C_{14}H_{17}O_6$ $[M+H]^{+}$ 281.1020; found 281.1021.



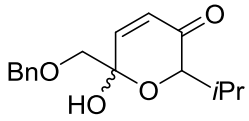
**2s** (EtOAc/hexane = 1:2): colorless oil (dr 9:1), 55.7 mg, 94%. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 7.88–7.84 (m, 1H), 7.64–7.62 (m, 1H), 7.17–7.14 (m, 1H), 6.99–6.89 (m, 1H), 6.23–6.16 (m, 1H), 4.77–4.67 (m, 1H), 4.45–4.21 (m, 2H), 2.52–2.41 (m, 1H), 1.05 (d, *J* = 7.0 Hz, 3H), 0.86 (d, *J* = 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 196.3, 162.3, 144.0, 134.4, 133.4, 132.5, 129.3, 128.7, 128.0, 93.5, 92.3, 82.8, 78.6, 68.4, 28.8, 18.9, 18.8, 16.9, 15.9. IR (KBr) 3401.3, 2966.6, 2876.1, 1687.9, 1524.0, 1415.3, 1367.4, 1265.2, 1078.5, 928.2, 748.0 cm$^{-1}$; HRMS (CI$^{+}$) (m/z) calcd. for $C_{14}H_{17}O_5S$ $[M+H]^{+}$ 297.0791; found 297.0796.



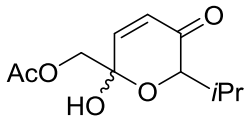
**2t** (EtOAc/hexane = 1:1): colorless oil (dr 9:1), 50.7 mg, 87%. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 8.80 (d, *J* = 4.8 Hz, 1H), 8.20 (d, *J* = 7.9 Hz, 1H), 7.93 (d, *J* = 7.7 Hz, 1H), 7.58 (dd, *J* = 7.7, 4.6 Hz, 1H), 7.01 (d, *J* = 10.3 Hz, 1H), 6.16 (d, *J* = 10.1 Hz, 1H), 4.78 (d, *J* = 11.4 Hz, 1H), 4.46–4.32 (m, 2H), 2.47–2.42 (m, 1H), 0.99 (d, *J* = 7.0 Hz, 3H), 0.83 (d, *J* = 6.7 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 196.4, 164.6, 149.6, 147.1, 144.3, 137.7, 128.6, 127.6, 125.7, 92.1, 78.5, 69.2, 28.7, 19.0, 15.9. IR (KBr) 3290.8, 2968.0, 2876.1, 1734.8, 1689.0, 1441.9, 1372.0, 1294.4, 1244.7, 1128.3, 1046.3, 753.3 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{15}H_{18}NO_5$ $[M+H]^+$ 292.1179; found 292.1190.



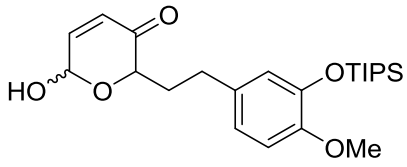
**2u** (EtOAc/hexane = 1:3): colorless oil (dr 9:1), 59.5 mg, 99%. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 6.72 (d, *J* = 10.2 Hz, 1H), 6.09 (d, *J* = 10.2 Hz, 1H), 4.39 (d, *J* = 2.6 Hz, 1H), 3.77 (d, *J* = 10.1 Hz, 1H), 3.67 (d, *J* = 10.1 Hz, 1H), 2.48–2.41 (m, 1H), 1.03 (d, *J* = 7.0 Hz, 3H), 0.93 (s, 9H), 0.84 (d, *J* = 6.8 Hz, 3H), 0.13 (d, *J* = 3.1 Hz, 6H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 197.4, 145.1, 129.3, 92.5, 78.6, 68.4, 28.7, 25.9, 19.1, 18.5, 16.1, -5.1, -5.3. IR (KBr) 3401.5, 2943.2, 2863.9, 1689.4, 1465.1, 1254.0, 1108.3, 1064.1, 841.9, 780.5 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{15}H_{29}O_4Si$ $[M+H]^+$ 301.1830; found 301.1842.



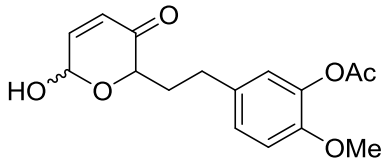
**2v** (EtOAc/hexane = 1:2): colorless oil (dr 9:1), 53 mg, 96%. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 7.43–7.29 (m, 5H), 6.79 (d, *J* = 10.2 Hz, 1H), 6.09 (d, *J* = 10.2 Hz, 1H), 4.78 (d, *J* = 11.9 Hz, 1H), 4.68 (d, *J* = 12.0 Hz, 1H), 4.43 (d, *J* = 2.7 Hz, 1H), 3.64 (q, *J* = 10.5 Hz, 2H), 2.51–2.43 (m, 1H), 1.07 (d, *J* = 7.0 Hz, 3H), 0.87 (d, *J* = 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 197.0, 145.0, 137.6, 129.0, 128.7, 128.2, 128.0, 93.0, 78.7, 74.4, 74.2, 28.8, 19.3, 16.3. IR (KBr) 3398.1, 2967.2, 2871.8, 1687.3, 1457.3, 1370.1, 1269.6, 1100.5, 752.7 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{16}H_{21}O_4$ $[M+H]^+$ 277.1434; found 277.1432.



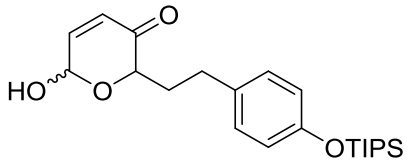
**2w** (EtOAc/hexane = 1:2): colorless oil (dr 9:1), 42 mg, 92%. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 6.86 (d, *J* = 10.3 Hz, 1H), 6.10 (d, *J* = 10.2 Hz, 1H), 4.43 (d, *J* = 11.6 Hz, 1H), 4.38 (d, *J* = 2.7 Hz, 1H), 4.07 (d, *J* = 11.7 Hz, 1H), 2.46–2.39 (m, 1H), 2.12 (s, 3H), 1.01 (d, *J* = 7.0 Hz, 3H), 0.82 (d, *J* = 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 196.6, 171.3, 144.2, 128.6, 93.4, 92.2, 82.8, 78.6, 68.0, 66.0, 30.3, 28.9, 20.9, 20.9, 19.0, 18.8, 17.1, 16.0.



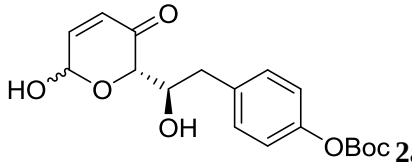

**2x** (EtOAc/hexane = 1:3): colorless oil (dr 7:3), 65.6 mg, 78%. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 6.92–6.86 (m, 1H), 6.76–6.71 (m, 3H), 6.15–6.07 (m, 1H), 5.65–5.61 (m, 1H), 4.52 (dd, *J* = 8.3, 3.7 Hz, 0.7H), 4.00 (ddd, *J* = 8.8, 3.9, 1.2 Hz, 0.3H), 3.76 (s, 3H), 3.46 (s, 0.3H), 3.16 (s, 0.7H), 2.79–2.60 (m, 2H), 2.28–2.10 (m, 1H), 1.97–1.93 (m, 1H), 1.30–1.17 (m, 3H), 1.15–1.01 (m, 18H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 196.5, 196.2, 149.2, 149.1, 147.6, 145.4, 144.2, 133.8, 133.6, 128.8, 127.6, 121.2, 121.1, 120.9, 120.9, 112.2, 112.2, 90.9, 87.7, 77.7, 73.2, 55.6, 32.2, 31.4, 30.3, 17.9, 12.9.




**2y** (EtOAc/hexane = 1:3): colorless oil (dr 7:3), 44.1 mg, 72%. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 7.10–7.02 (m, 1H), 6.96–6.80 (m, 3H), 6.14–6.00 (m, 1H), 5.54–5.53 (m, 1H), 4.43 (dd, *J* = 9.3, 3.6 Hz, 0.7H), 3.98 (ddd, *J* = 8.9, 3.7, 1.2 Hz, 0.3H), 3.79 (s, 3H), 2.78–2.65 (m, 2H), 2.31–2.20 (m, 4H), 2.11–1.85 (m, 1H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 196.9, 170.1, 169.7, 149.2, 149.1, 148.1, 144.7, 139.1, 133.8, 133.6, 128.7, 127.4, 126.7, 126.6, 123.7, 123.3, 112.5, 112.4, 90.9, 87.5, 77.8, 72.5, 56.0, 31.6, 30.3, 30.1, 20.9, 20.8. IR (KBr) 3437.6, 2931.0, 2847.0, 1758.9, 1687.1, 1510.8, 1437.0, 1369.6, 1264.9, 1201.0, 1121.5, 1087.9, 1018.4, 756.6 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{16}H_{18}O_6$ $[M]^+$ 306.1098; found 306.1115.

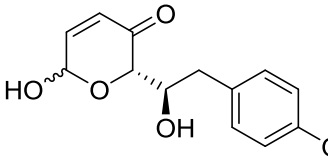



**2z** (EtOAc/hexane = 1:3): colorless oil (dr 7:3), 63.3 mg, 81%. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 7.04 (d, *J* = 8.4 Hz, 2H), 6.9–6.84 (m, 1H), 6.78 (d, *J* = 8.4 Hz, 2H), 6.17–6.05 (m, 1H), 5.67–5.58 (m, 1H), 4.52 (dd, *J* = 8.5, 3.7 Hz, 0.7H), 4.01 (dd, *J* = 9.0, 3.8 Hz, 0.3H), 2.81–2.57 (m, 2H), 2.31–2.13 (m, 1H), 2.11–1.90 (m, 1H), 1.33–1.18 (m, 3H), 1.15–1.00 (m, 18H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 196.5, 196.2, 154.4, 147.6, 144.2, 133.8, 133.5, 133.5, 129.6, 129.5, 129.0, 127.9, 119.9, 119.9, 91.0, 87.9, 77.9, 73.4, 32.5, 31.6, 30.3, 29.9, 18.1, 18.1, 13.1, 12.8.

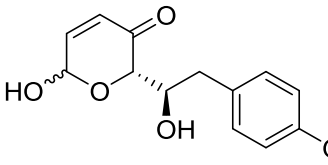



**2aa** (EtOAc/hexane = 1:1): colorless oil (dr 1:1), 61.7 mg, 88%. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 7.41–7.21 (m, 2H), 7.13–7.09 (m, 2H), 7.06–6.82 (m, 1H), 6.15 (dd, *J* = 19.5, 10.4 Hz, 1H), 5.85–5.53 (m, 1H), 4.72–4.34 (m, 1H), 4.34–4.17 (m, 1H), 3.20–2.76 (m, 2H), 1.57 (s, 9H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 195.6, 195.2, 152.2, 149.9, 149.7, 146.3, 144.9, 135.4, 134.9, 130.5, 130.4, 127.9, 127.5, 121.6, 121.3, 121.1, 87.8,

87.2, 83.8, 83.7, 80.3, 74.8, 73.2, 71.4, 38.7, 38.6, 27.7. IR (KBr) 3380.0, 2981.7, 2931.8, 1750.6, 1688.7, 1508.5, 1370.8, 1266.8, 1146.1, 1022.3, 893.1, 831.1, 754.3 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{18}H_{23}O_7$ $[M+H]^+$ 351.1438; found 351.1435.

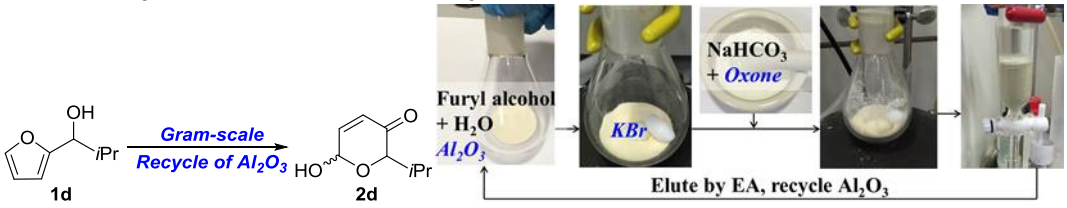



**2ab** (EtOAc/hexane = 1:2): colorless oil (dr 3:2), 62.7 mg, 86%. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 7.20–7.07 (m, 2H), 6.93–6.88 (m, 1H), 6.78–6.75 (m, 2H), 6.20–6.03 (m, 1H), 5.75–5.55 (m, 1H), 4.55–4.01 (m, 2H), 2.95–2.85 (m, 2H), 0.97 (s, 9H), 0.18–0.17 (m, 6H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 194.9, 153.9, 153.7, 145.8, 130.0, 129.8, 129.7, 129.6, 128.9, 127.4, 126.9, 119.7, 119.5, 87.2, 86.5, 79.5, 74.2, 72.9, 71.1, 37.8, 25.0, 17.6, -5.1. IR (KBr) 3363.3, 2936.6, 2858.6, 1686.0, 1609.6, 1508.4, 1463.7, 1255.3, 1018.7, 911.1, 834.3, 772.9 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{19}H_{28}O_5Si$ $[M]^+$ 364.1701; found 364.1699.




**2ac** (EtOAc/hexane = 1:2): colorless oil (dr 1:1), 68.7 mg, 85%. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 7.72 (dd, *J* = 8.2, 5.8 Hz, 2H), 7.33 (dd, *J* = 8.1, 4.6 Hz, 2H), 7.19 (dd, *J* = 8.6, 2.0 Hz, 2H), 7.01–6.87 (m, 3H), 6.16 (dd, *J* = 17.9, 10.4 Hz, 1H), 5.76–5.59 (m, 1H), 4.41–4.20 (m, 2H), 2.99–2.85 (m, 2H), 2.46 (d, *J* = 2.9 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 195.7, 195.2, 148.4, 148.3, 146.3, 145.5, 145.1, 137.0, 136.5, 132.4, 130.7, 130.6, 129.8, 129.8, 128.5, 128.5, 127.9, 127.6, 122.5, 122.4, 87.8, 87.2, 80.1, 74.7, 73.2, 71.4, 38.6, 38.6, 21.7. IR (KBr) 3344.0, 2927.9, 2867.2, 1686.3, 1598.0, 1500.6, 1364.3, 1270.2, 1150.1, 1095.3, 1020.9, 862.8, 753.7 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{20}H_{20}O_7S$ $[M]^+$ 404.0924; found 404.0912.

**Gram-scale synthesis of 2d with the recycle of $Al_2O_3$.**




**General Procedure F**:

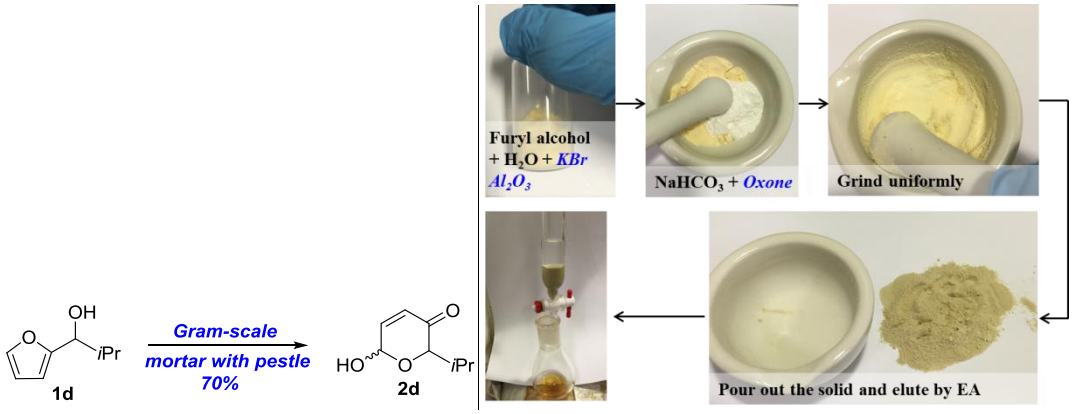
(Entry 1, Table 3, The main text) Aluminium oxide 90 active neutral (Merck, 0.063–0.200 mm) was equilibrated at 120 °C for 48 h, then it was cooled to RT under nitrogen. To a 100-mL round-bottomed flask was added two magnetic stirrers, furyl alcohol **1d** (1 g, 7.13 mmol) and the above activated aluminium oxide (4.28 g). The resulting mixture was stirred until uniformly free-flowing. To the above mixture was added $H_2O$ (385 mg, 21.4 mmol) and KBr (84.4 mg, 0.71 mmol), and the mixture was stirred again to be free-flowing. To the above mixture was added $NaHCO_3$ (600 mg, 7.13 mmol) and Oxone (4.38 g, 7.13 mmol), the mixture was stirred vigorously for 10 min at RT. Then the solid was transferred into a glass

column and eluted by EtOAc to get the crude product **2d**. The $Al_2O_3$ was reycled by air flowing to remove the absorbed EtOAc.

(Recycle of $Al_2O_3$ for the first time, Entry 2, Table 3, The main text) To the above recycled free-flowing solid was added $H_2O$ (770 mg, 42.8 mmol), and the mixture was stirred to be free-flowing. To the above mixture was added furyl alcohol **1d** (1 g, 7.13 mmol) and KBr (84.4 mg, 0.71 mmol), and the mixture was stirred to be free-flowing. $NaHCO_3$ (600 mg, 7.13 mmol) and Oxone (4.38 g, 7.13 mmol) were added in the end, and the mixture was stirred vigorously for 10 min at RT. Then the solid was transferred into a glass column and eluted by EtOAc to get the crude product **2d**. The $Al_2O_3$ was reactivated by air flowing to remove the absorbed EtOAc.

(Recycle of $Al_2O_3$ for the second to fifth time, Entry 3 to 6, Table 3, The main text) The detailed procedure was similar to the above, and the only difference was the $H_2O$ added and reaction time, which were listed in the Table 3 of the main text.

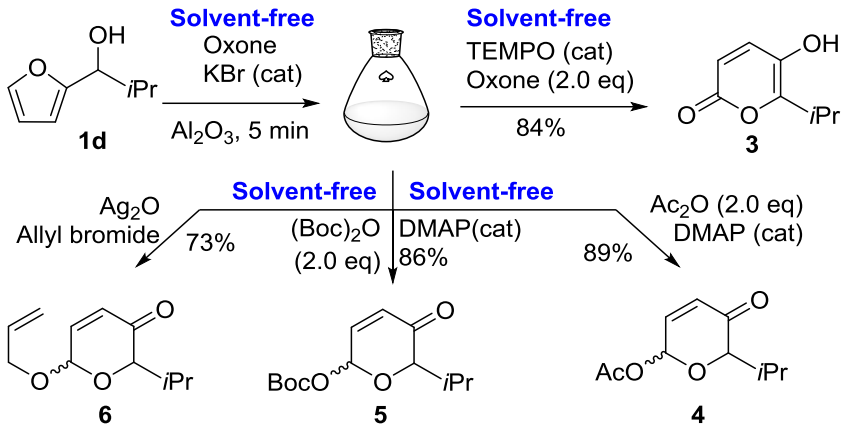
**Gram-scale synthesis of 2d using mortar with pestle.**




**General Procedure G**:

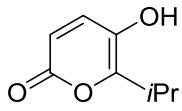
Add furyl alcohol **1d** (1 g, 7.13 mmol) to activated aluminium oxide (4.28 g), and the resulting mixture was shaked until uniformly free-flowing. To the above mixture was added $H_2O$ (385 mg, 21.4 mmol) and KBr (84.4 mg, 0.71 mmol), and the mixture was shaked again to be free-flowing. Pour the solid to mortar, add $NaHCO_3$ (600 mg, 7.13 mmol) and Oxone (4.38 g, 7.13 mmol), and the mixture was ground unformly for 10 min at RT by pestle. Then the solid was transferred into a glass column and eluted by EtOAc to get the crude product **2d** (yield: 70%).

**Solvent-free, one-pot, two-step reactions involving AchR.**



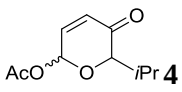

**General Procedure H**: Aluminium oxide 90 active neutral (Merck, 0.063–0.200 mm) was equilibrated with at 120 °C for 48 h, then it was cooled to RT under nitrogen. To a 5-mL round-bottomed flask was added a magnetic stirrer, furyl alcohol **1d** (28 mg, 0.2 mmol) and the above activated aluminium oxide (120 mg). The resulting mixture was stirred until uniformly free-flowing. To the above mixture was added $H_2O$ (7.2 mg, 0.4 mmol) and KBr (7.1 mg, 0.06 mmol), and the mixture was stirred again to be free-flowing. To the above mixture was added $NaHCO_3$ (16.8 mg, 0.2 mmol) and Oxone (123 mg, 0.2 mmol), the mixture was stirred vigorously for 5 min at RT to get the crude product **2d** suspended on $Al_2O_3$.

To the above solid containing **2d** was added TEMPO (6.25 mg, 0.04 mmol) and Oxone (246 mg, 0.4 mmol). After completion of the addition, the above solid was stirred vigorously for 10 min at RT. The resulting solid was transferred onto silica gel and purified by flash column chromatography (EtOAc/hexane = 1:1 to EtOAc) to give the product **3** (isomerize to the conjugated structure in silica gel for a long time).



**3** (EtOAc/hexane = 1:3): colorless oil, 25.9 mg, 84% for 2 steps. $^1$H-NMR (400 MHz, MeOD) δ: 7.40 (d, *J* = 9.7 Hz, 1H), 6.12 (d, *J* = 9.7 Hz, 1H), 3.24 (p, *J* = 7.0 Hz, 1H), 1.20 (d, *J* = 6.9 Hz, 6H). $^{13}$C-NMR (100 MHz, MeOD) δ: 164.8, 155.4, 143.9, 136.0, 113.4, 28.1, 19.9. IR (KBr) 3376.7, 2975.7, 2936.2, 2881.3, 1710.4, 1620.0, 1548.2, 1461.5, 1328.5, 1137.9, 754.8 cm$^{-1}$; HRMS (CI$^+$) (m/z) calcd. for $C_8H_{10}O_3$ $[M]^+$ 154.0624; found 154.0636.

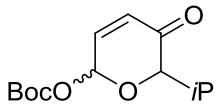
To the above solid containing **2d** was added $NaHCO_3$ (67.2 mg, 0.8 mmol), DMAP (12.2 mg, 0.1 mmol) and $Ac_2O$ (37.8 μL, 0.4 mmol). After completion of the addition, the above solid was stirred vigorously for 10 min at RT. The resulting mixture was transferred onto silica gel and purified by flash column chromatography (EtOAc/hexane = 1:5) to give the product **4**.



**4** (EtOAc/hexane = 1:5): colorless oil (dr 7:3), 35.3 mg, 89% for 2 steps. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 6.92–6.74 (m, 1H), 6.51–6.49 (m, 1H), 6.17 (d, *J* = 10.4 Hz, 1H), 4.28 (d, *J* = 2.8 Hz, 0.78H), 3.87 (d, *J* = 6.6 Hz, 0.22H), 2.48–2.22 (m, 1H), 2.13–2.09 (m, 3H), 1.04 (d, *J* = 6.9 Hz, 3H), 0.85 (d, *J* = 6.9 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ:

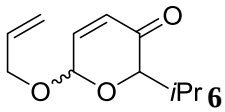
195.7, 195.3, 169.6, 169.4, 143.2, 141.6, 129.2, 129.2, 87.9, 87.3, 84.3, 80.1, 30.5, 28.9, 21.1, 21.0, 18.8, 18.8, 17.9, 16.0.

To the above solid containing **2d** was added $NaHCO_3$ (67.2 mg, 0.8 mmol), DMAP (31.8 mg, 0.26 mmol) and $Boc_2O$ (91.9 μL, 0.4 mmol). After completion of the addition, the above solid was stirred vigorously for 10 min at RT. The resulting mixture was transferred onto silica gel and purified by flash column chromatography (EtOAc/hexane = 1:5) to give the product **5**.



**5** (EtOAc/hexane = 1:5): colorless oil (dr 9:1), 44.1 mg, 86% for 2 steps. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 6.89 (dd, *J* = 10.2, 3.7 Hz, 1H), 6.39 (d, *J* = 3.7 Hz, 1H), 6.21 (d, *J* = 10.2 Hz, 1H), 4.38 (d, *J* = 2.8 Hz, 1H), 2.49–2.44 (m, 1H), 1.55–1.53 (s, 9H), 1.05 (d, *J* = 7.0 Hz, 3H), 0.90 (d, *J* = 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 195.5, 151.8, 140.8, 129.4, 89.4, 83.5, 79.7, 28.6, 27.7, 18.7, 15.9.

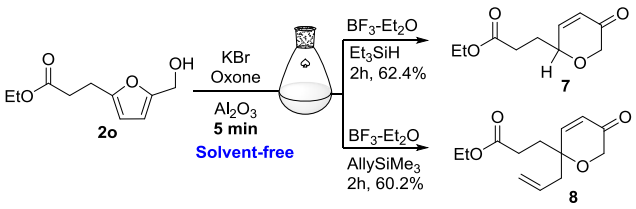
To the above solid containing **2d** (in the preparation of **2d**, only 3.6 mg $H_2O$ was added) was added $Ag_2O$ (92.7 mg, 0.4 mmol) and allyl bromide (69 μL, 0.8 mmol). After completion of the addition, the above solid was stirred vigorously for 15 min at RT. The resulting solid was transferred onto silica gel and purified by flash column chromatography (EtOAc/hexane = 1:5) to give the product **6**.



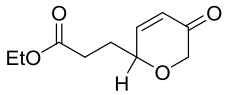
**6** (EtOAc/hexane = 1:5): colorless oil (dr 5:4), 28.6 mg, 73%. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 6.94–6.80 (m, 1H), 6.16–6.07 (m, 1H), 6.02–5.90 (m, 1H), 5.39–5.21 (m, 3H), 4.41 (ddt, *J* = 12.7, 5.2, 1.5 Hz, 0.5H), 4.33–4.11 (m, 2H), 3.84 (dd, *J* = 3.9, 1.2 Hz, 0.5H), 2.53–2.38 (m, 1H), 1.06 (dd, *J* = 8.0, 7.0 Hz, 3H), 0.92 (dd, *J* = 30.3, 6.8 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 196.1, 195.7, 146.4, 142.6, 133.0, 128.9, 127.7, 117.4, 94.6, 91.5, 82.5, 77.5, 76.7, 68.8, 68.8, 28.6, 27.7, 18.5, 18.5, 16.2, 15.4. IR (KBr) 2965.8, 2895.4, 2875.5, 1693.7, 1463.4, 1394.4, 1367.5, 1260.8, 1138.5, 1029.2, 927.4, 750.1 $cm^{-1}$; HRMS ($CI^+$) (m/z) calcd. for $C_{11}H_{17}O_3$ $[M+H]^+$ 197.1172; found 197.1182.

**One-Pot sequential AchR/Kishi reduction and AchR-Ferrier allylation.**

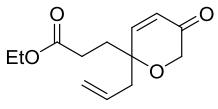
Nevertheless, we recognized that the one-pot, solvent-free condition could not be extended to AchR-Kishi reduction or AchR-Ferrier allylation. Since Kishi reduction and Ferrier-type allylation have been widely used for synthesis of tetrahydropyrans from AchR products, we aimed to achieve the one-pot reaction goal. Fortunately, we found that excess of $BF_3$-$Et_2O$ (8.0 eq) could effect Kishi reduction and Ferrier-type allylation of the AchR product in the same reaction vessel by adding solvent dichloromethane, $Et_3SiH$ (12.0 eq) and allyltrimethylsilane (12.0 eq), respectively. The presence of neutral alumina might reduce the $BF_3$-$Et_2O$ acidity and thus excess of $BF_3$-$Et_2O$ was required for both Kishi reduction and Ferrier allylation. It was also noted that without addition of dichloromethane, the AchR product was rapidly decomposed by $BF_3$-$Et_2O$. Nevertheless, the one-pot process of two reactions still presented sufficient greenness, cost, and time advantages over the two-pot operation.




**General Procedure I**: Aluminium oxide 90 active neutral (Merck, 0.063–0.200 mm) was equilibrated at 120 °C for 48 h, then it was cooled to RT under nitrogen. To a 5-mL round-bottomed flask was added a magnetic stirrer, furyl alcohol **1o** (29.7 mg, 0.15 mmol) and the above activated aluminium oxide (90 mg, 600 eq). The resulting mixture was stirred until uniformly free-flowing. To the above mixture was added $H_2O$ (2.7 mg, 0.15 mmol) and KBr (1.8 mg, 0.015 mmol), and the mixture was stirred again to be free-flowing. To the above mixture was added $NaHCO_3$ (12.6 mg, 0.15 mmol) and Oxone (92.2 mg, 0.15 mmol), the mixture was stirred vigorously for 5 min at RT to get the crude product **2o** suspended on $Al_2O_3$. To the above solid were added $Et_3SiH$ (287 μL, 1.8 mmol) or allyltrimethylsilane (287 μL, 1.8 mmol) and DCM (1 mL). The above mixture was cooled to –40 ºC, and $BF_3$•$OEt_2$ (148 μL, 1.2 mmol) was added dropwise under nitrogen. After completion of the addition, the mixture was warmed to –20 ºC and stirred for 2 h. The reaction was quenched by saturated aqueous $NaHCO_3$ solution (30 mL), and the residue was extracted with DCM (3 × 10 mL). The combined organic fractions were washed with brine, dried over $Na_2SO_4$, filtered and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography (EtOAc/hexane = 1:5) to give the product **7** or **8**.




**7** (EtOAc/hexane = 1:5): colorless oil, 18.6 mg, 62.4% for 2 steps. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 6.95 (dd, *J* = 10.5, 1.9 Hz, 1H), 6.15 (dd, *J* = 10.5, 2.3 Hz, 1H), 4.39 (ddd, *J* = 8.5, 4.2, 2.1 Hz, 1H), 4.26 (d, *J* = 16.3 Hz, 1H), 4.15 (q, *J* = 7.1 Hz, 2H), 4.08 (dd, *J* = 16.3, 1.8 Hz, 1H), 2.53–2.43 (m, 2H), 2.12–1.91 (m, 2H), 1.26 (t, *J* = 7.1 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 194.8, 173.2, 151.2, 127.4, 72.6, 71.3, 60.7, 29.8, 29.2, 14.4. IR (KBr) 2981.3, 2935.5, 2820.4, 1728.4, 1695.0, 1382.4, 1325.7, 1264.7, 1176.3, 1100.9, 1031.5, 754.4 cm$^{-1}$; HRMS (CI$^+$) (m/z) calcd. for $C_{10}H_{13}O_4$ $[M-H]^+$ 197.0808; found 197.0819.




**8** (EtOAc/hexane = 1:5): colorless oil, 21.5 mg, 60.2 for 2 steps. $^1$H-NMR (400 MHz, $CDCl_3$) δ: 6.87 (d, *J* = 10.6 Hz, 1H), 6.10 (d, *J* = 10.6 Hz, 1H), 5.85–5.75 (m, 1H), 5.19–5.14 (m, 2H), 4.31–4.16 (m, 2H), 4.12 (q, *J* = 7.1 Hz, 2H), 2.52–2.31 (m, 4H), 2.09–1.98 (m, 2H), 1.24 (t, *J* = 7.2 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 194.3, 173.4, 154.0, 132.0, 126.5, 119.6, 75.8, 67.3, 60.7, 40.8, 31.8, 28.8, 14.3. IR (KBr) 2980.1, 2936.1, 1729.9, 1693.5, 1438.7, 1391.9, 1266.1, 1181.3, 1094.4, 756.0 cm$^{-1}$; HRMS (CI$^+$) (m/z) calcd. for $C_{13}H_{19}O_4$ $[M+H]^+$ 239.1278; found 239.1280.

# Reference: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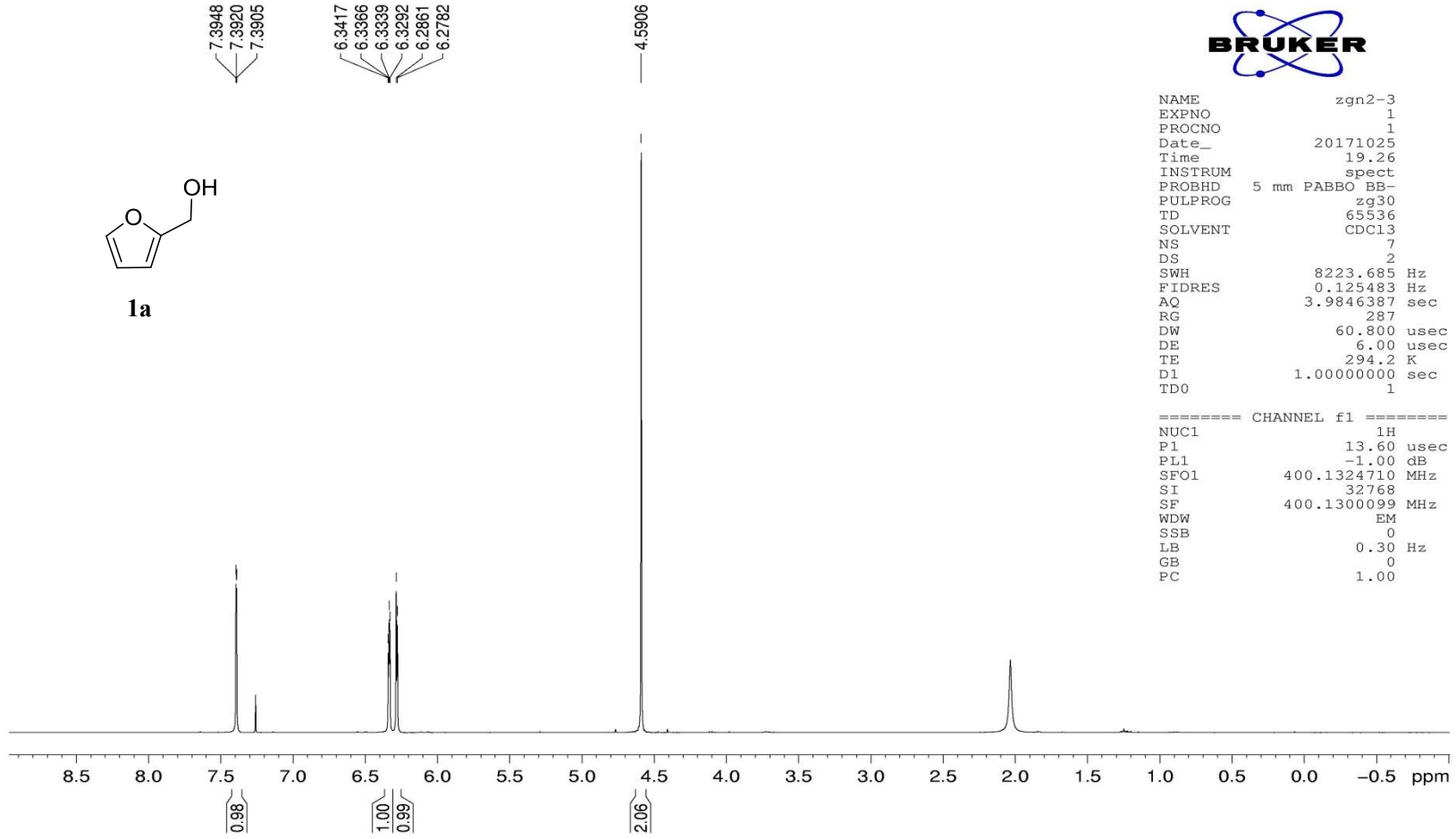
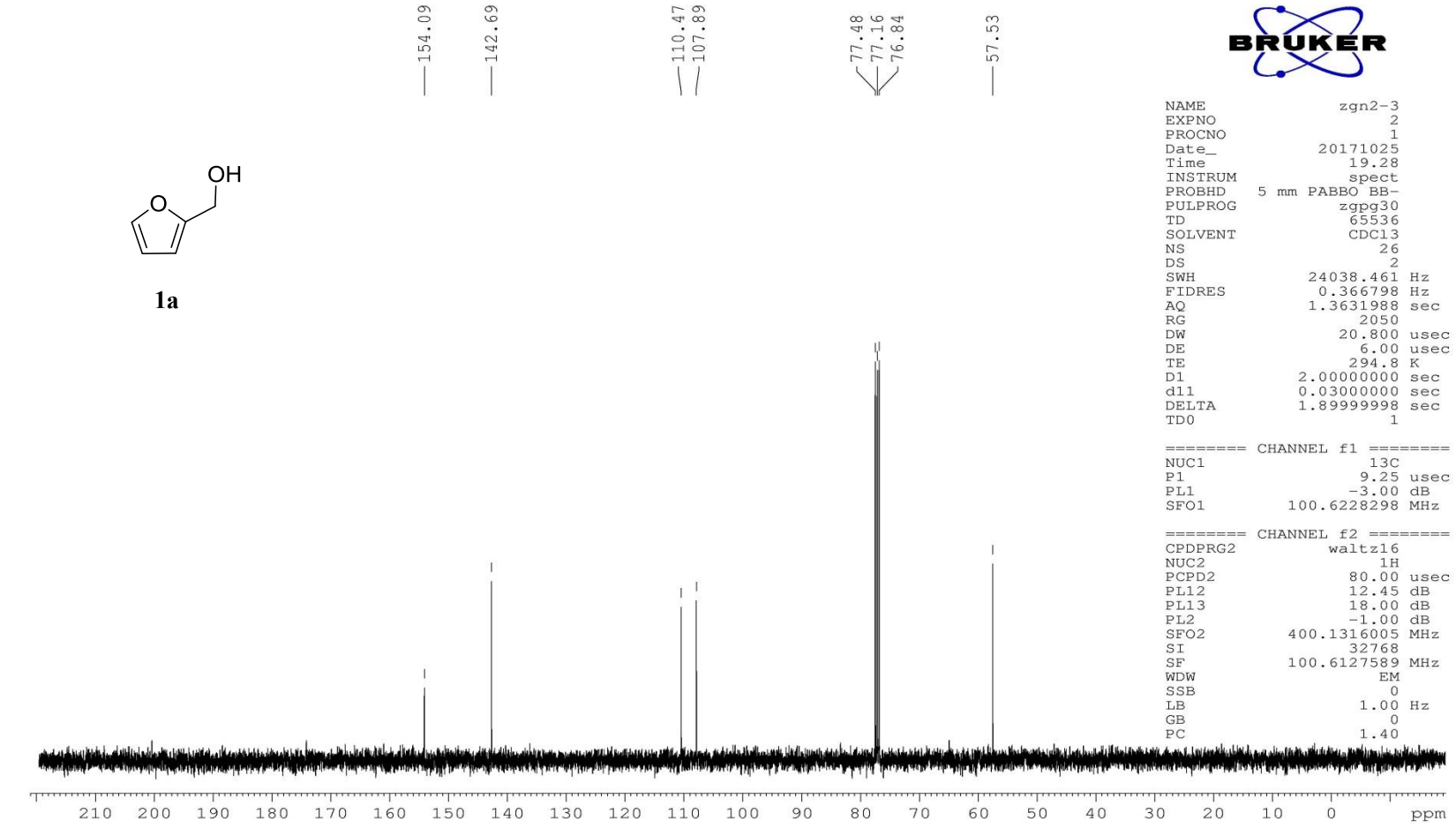
**Copies of $^{1}$H- and $^{13}$C-NMR Spectra**

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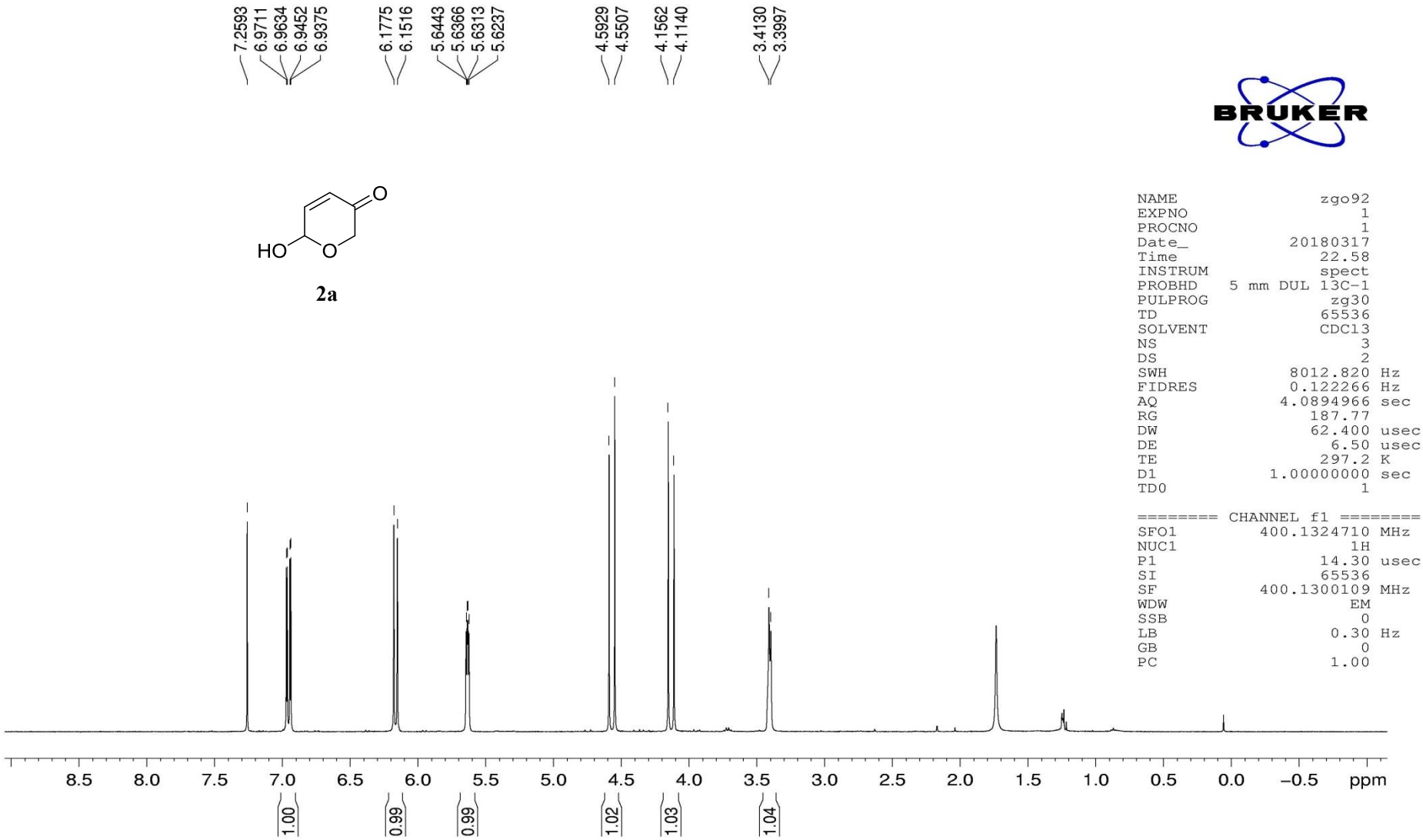
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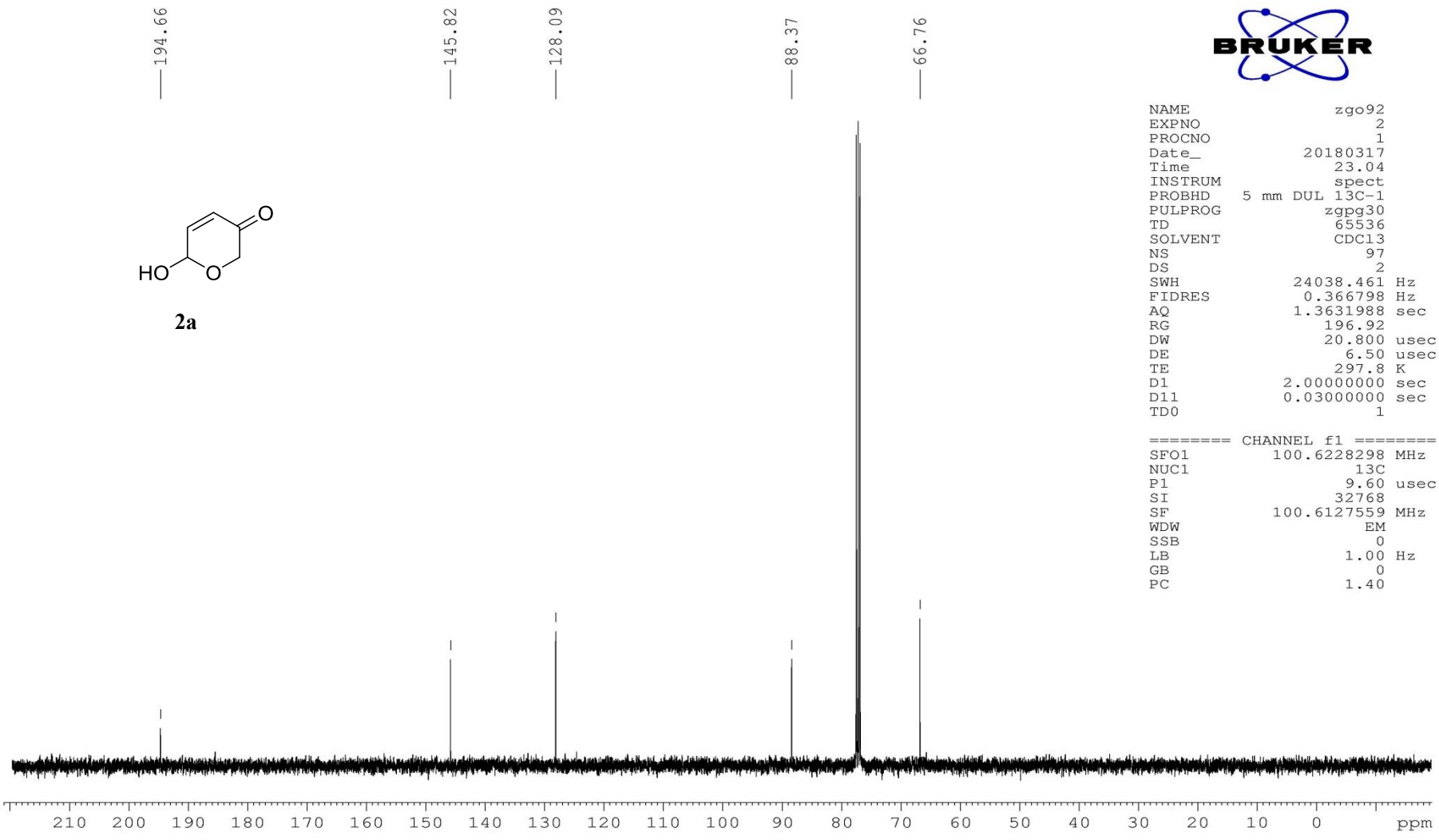
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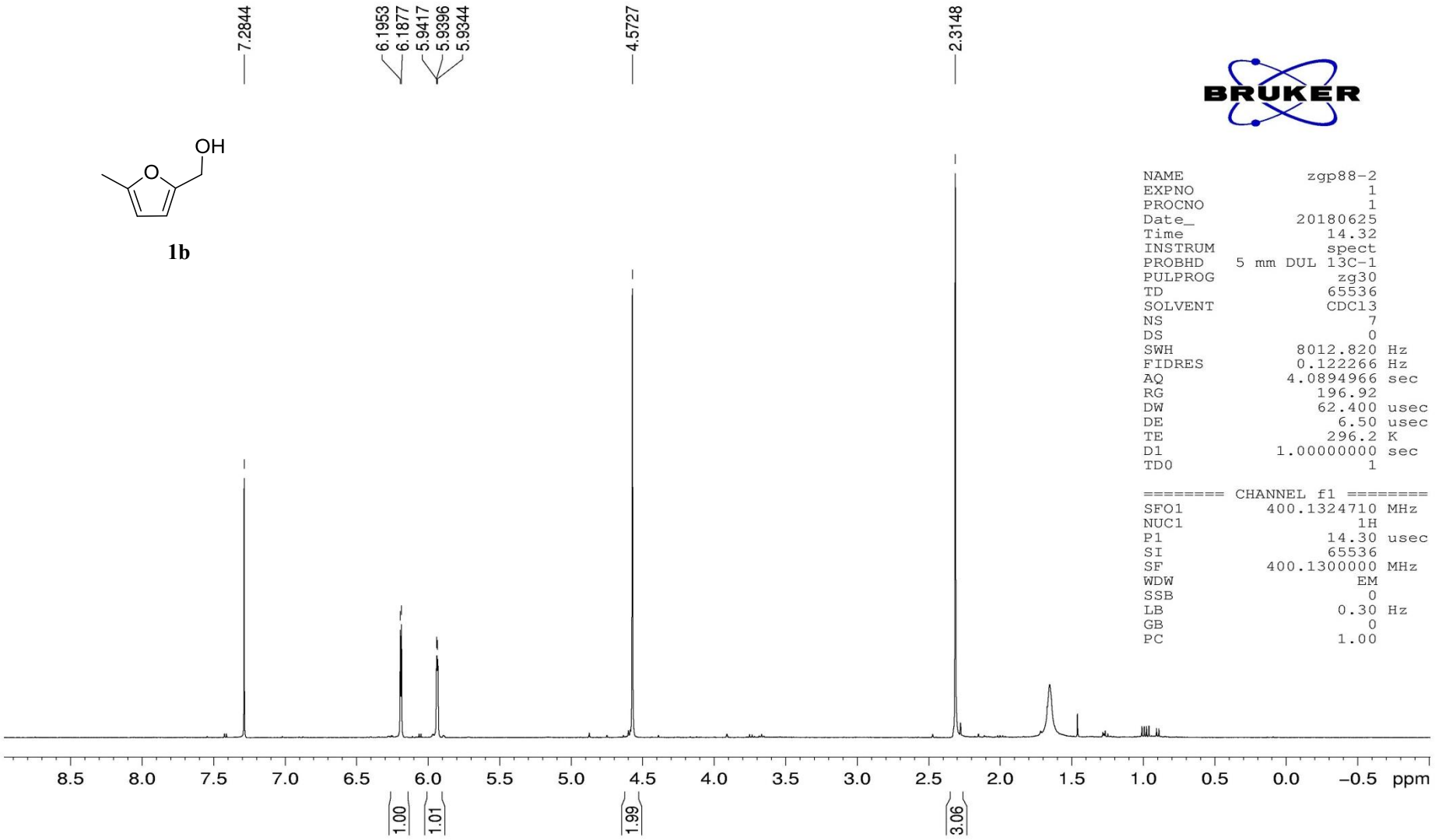
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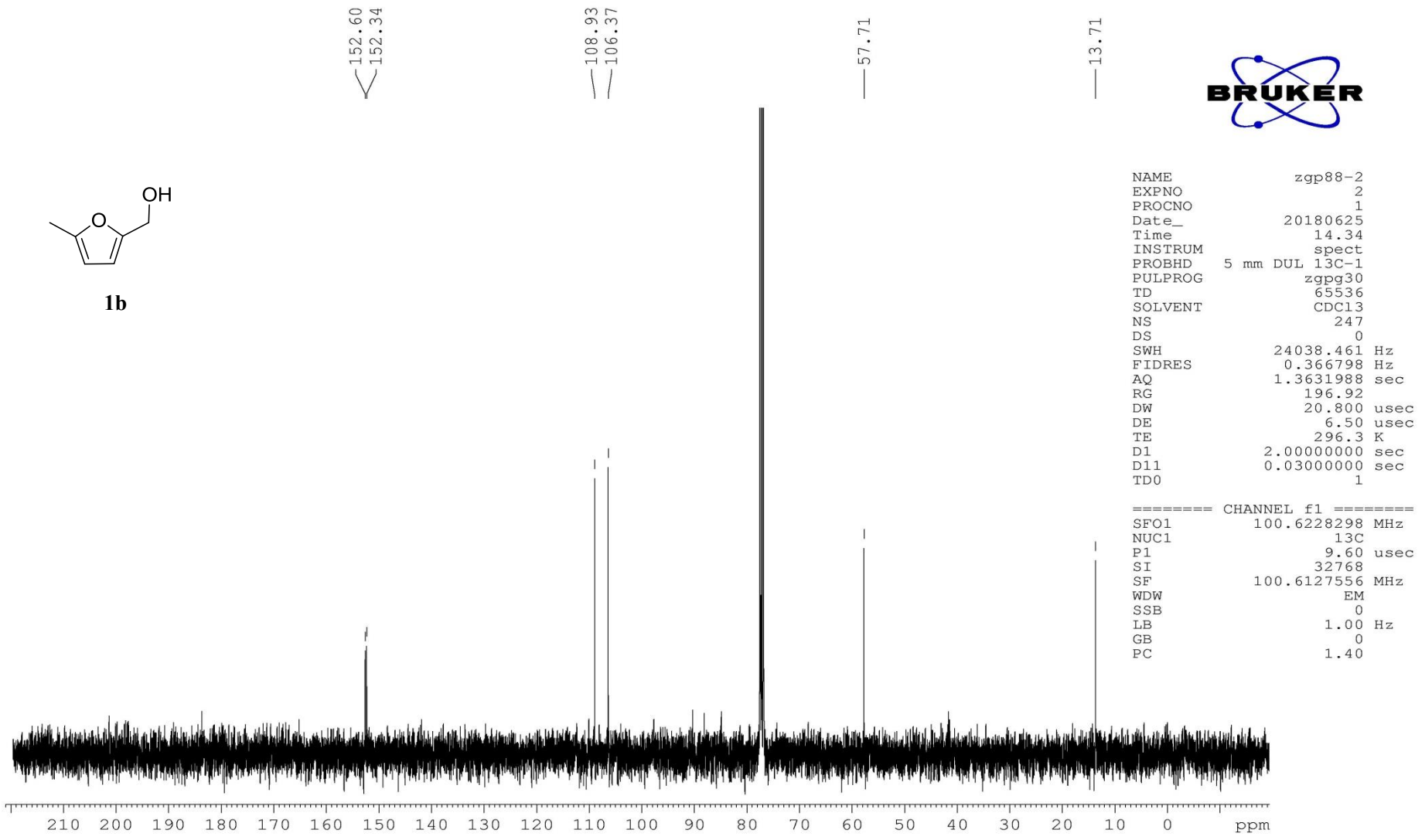
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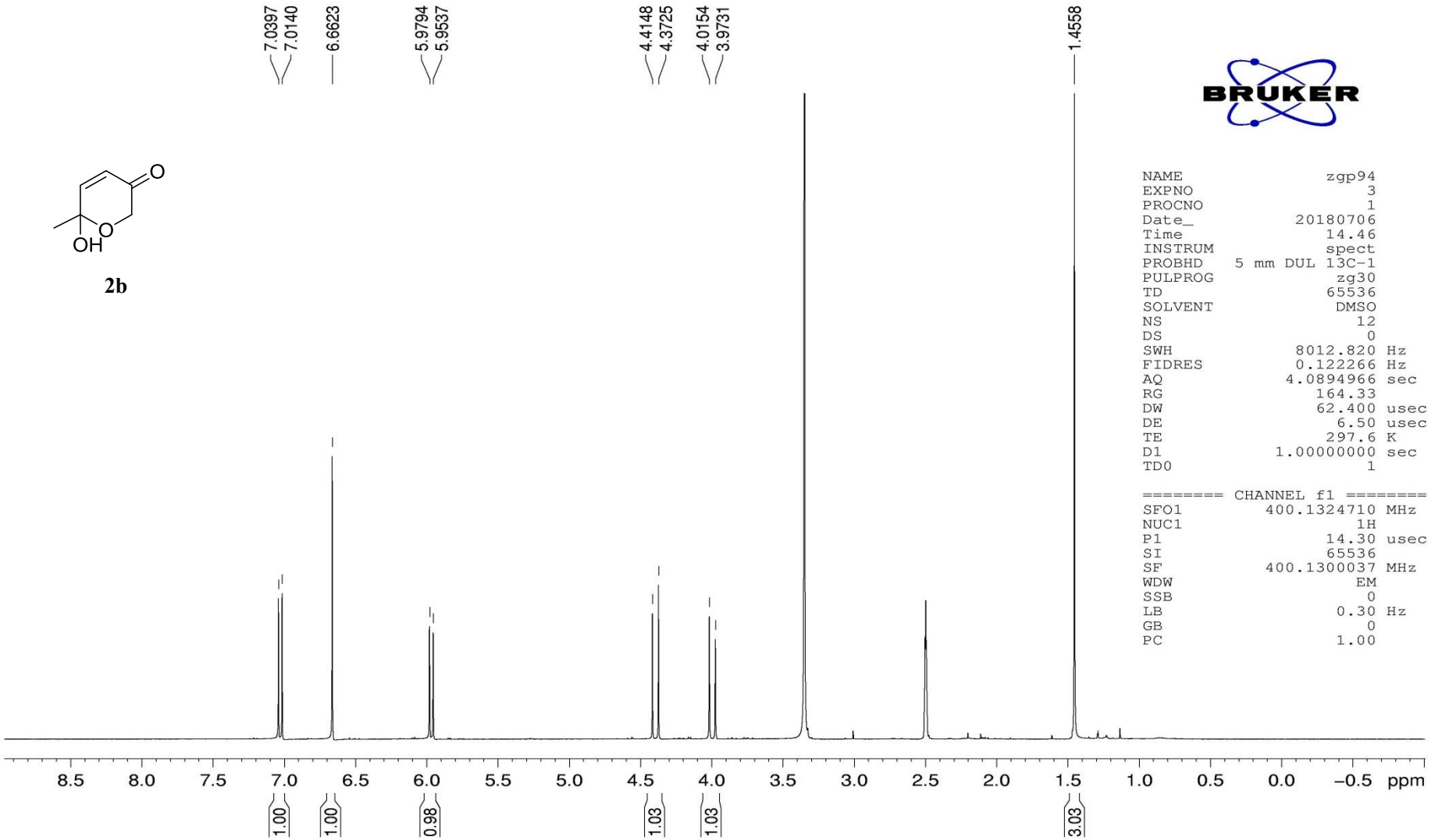
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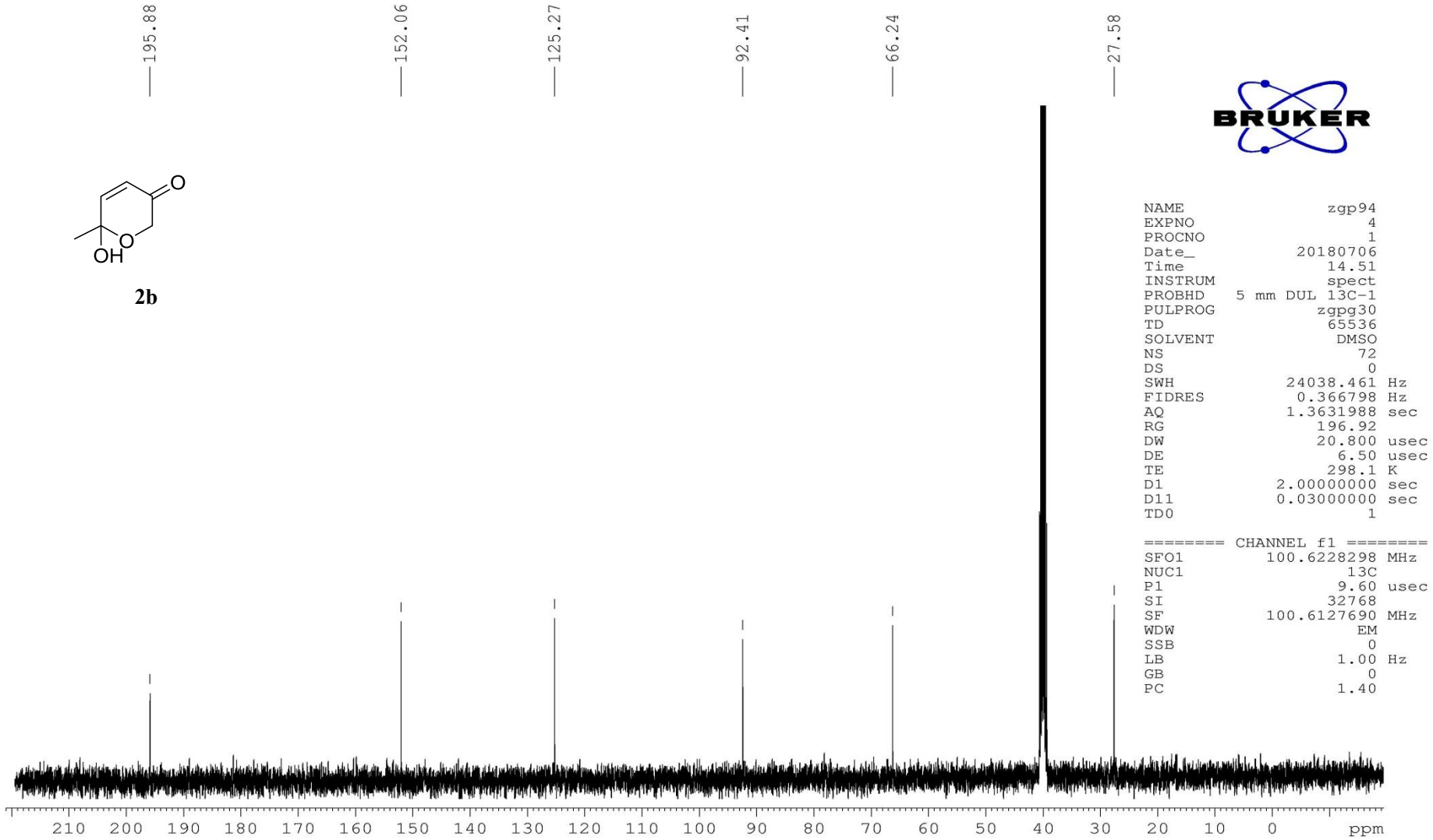
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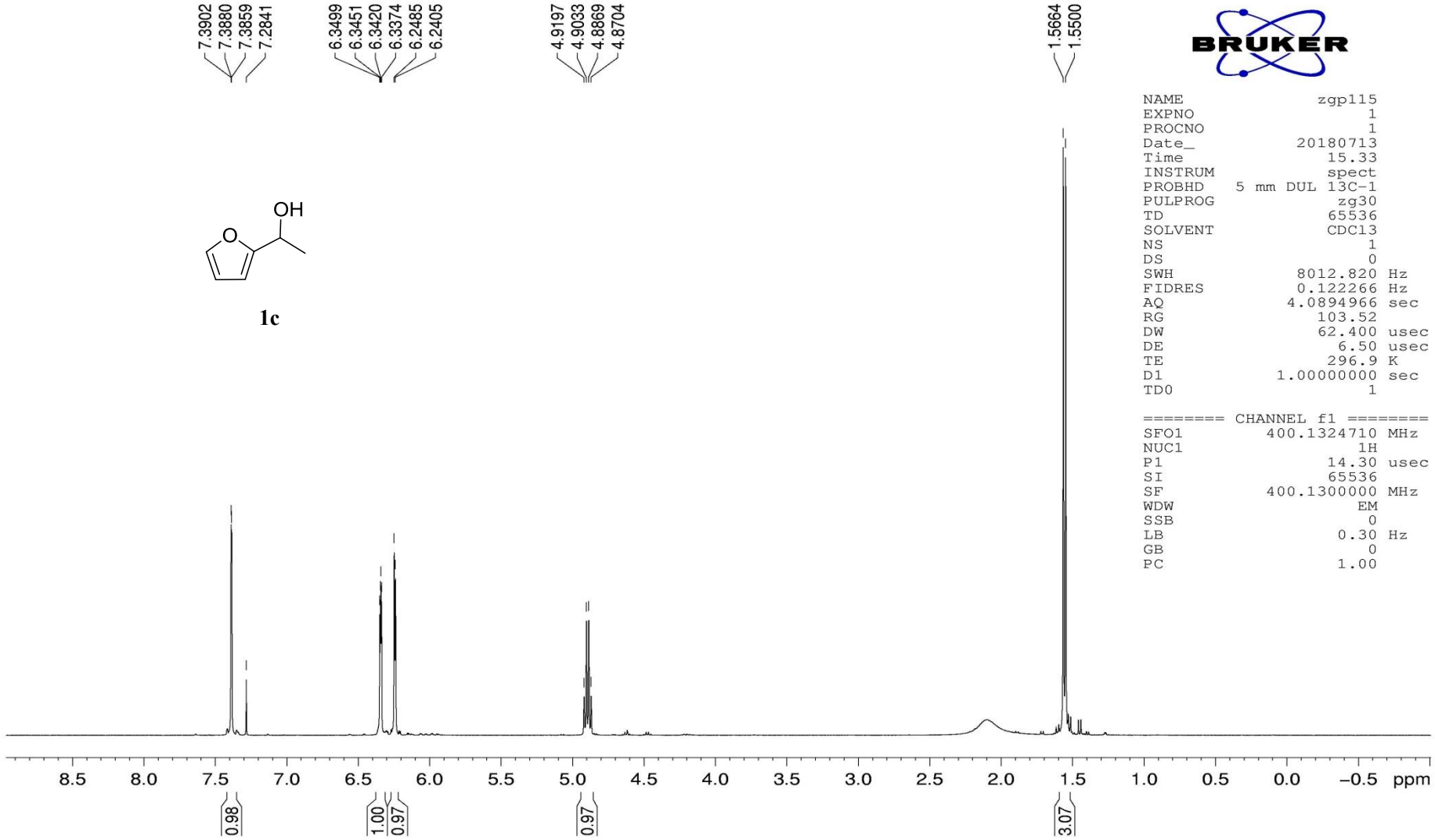
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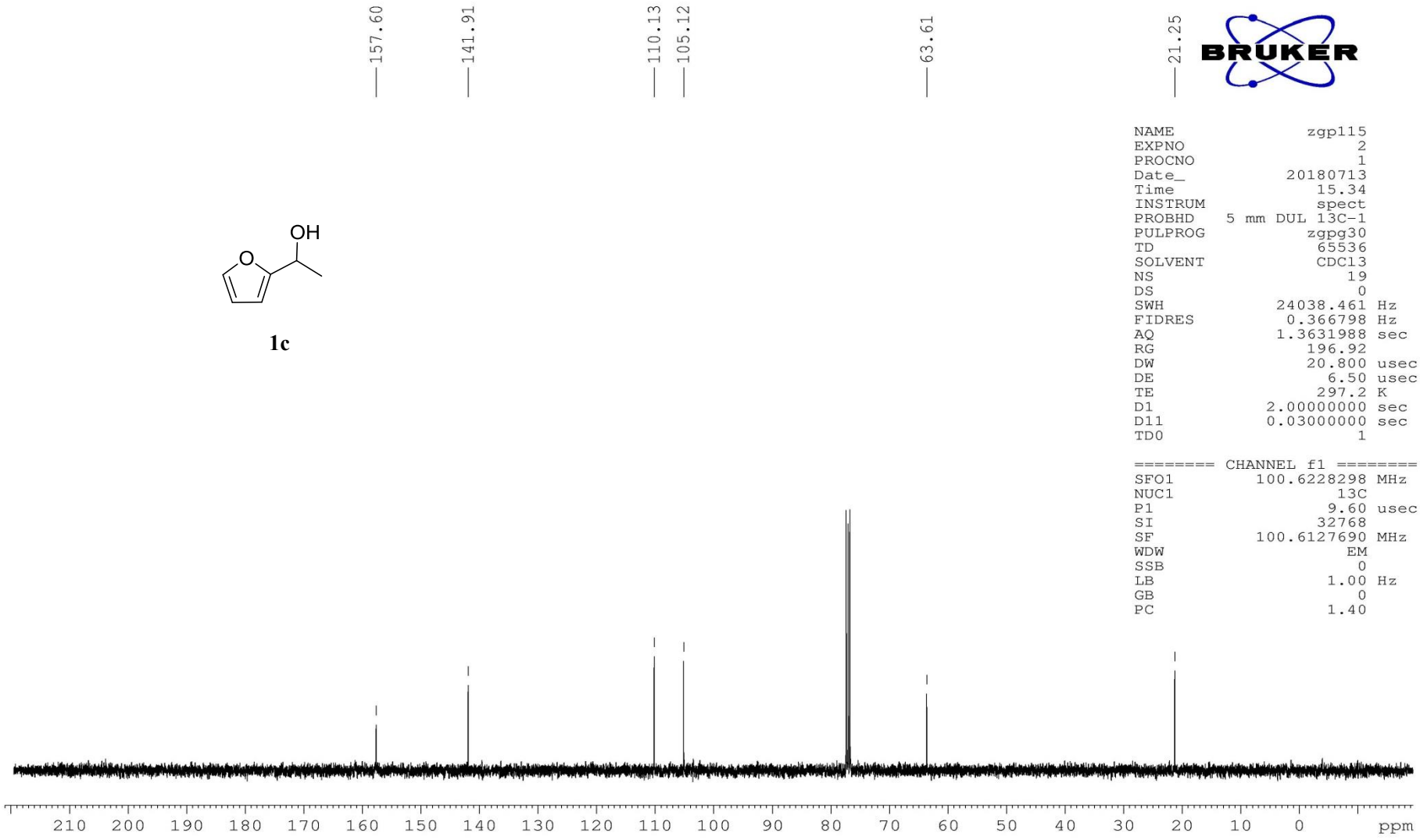
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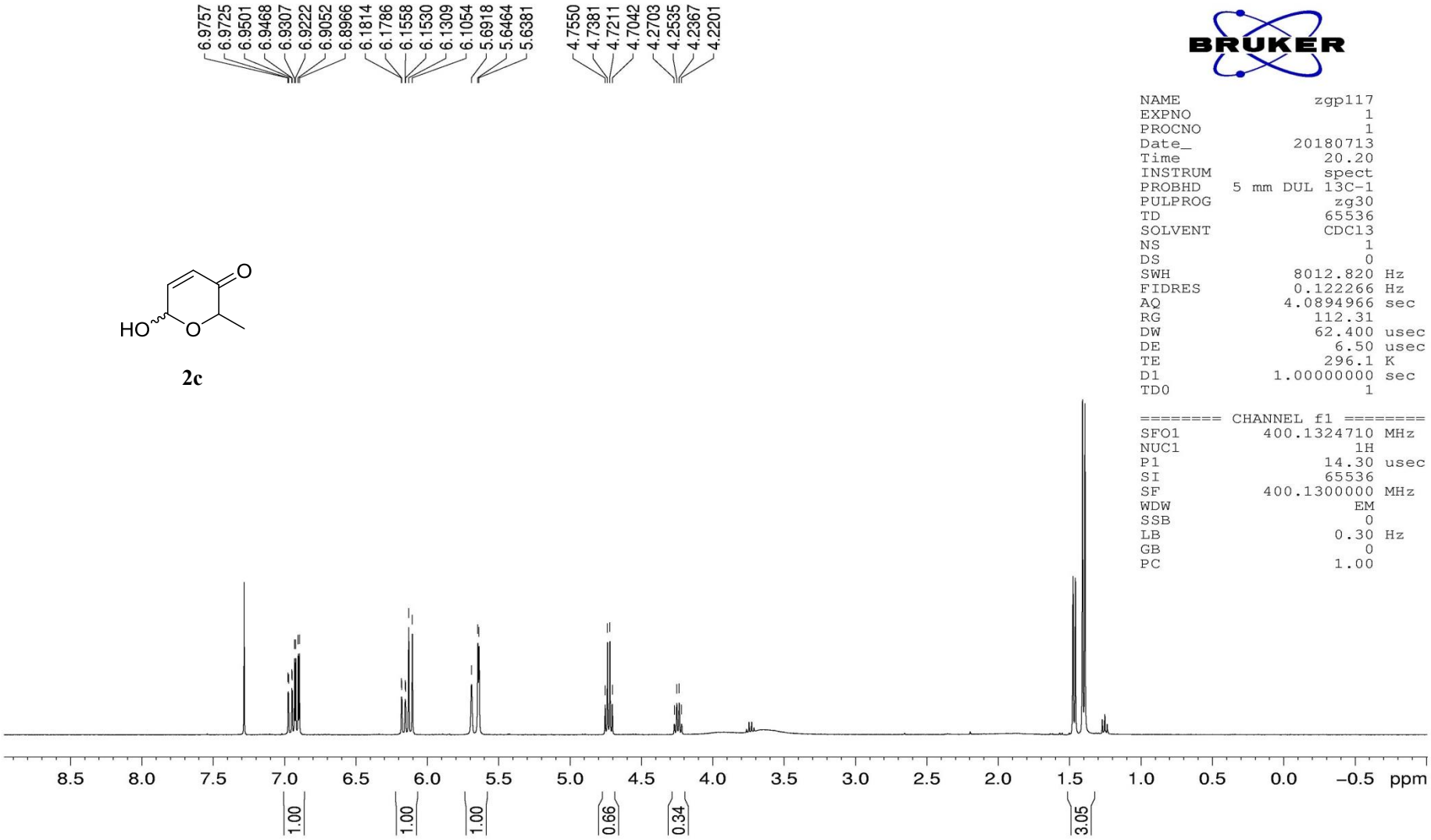
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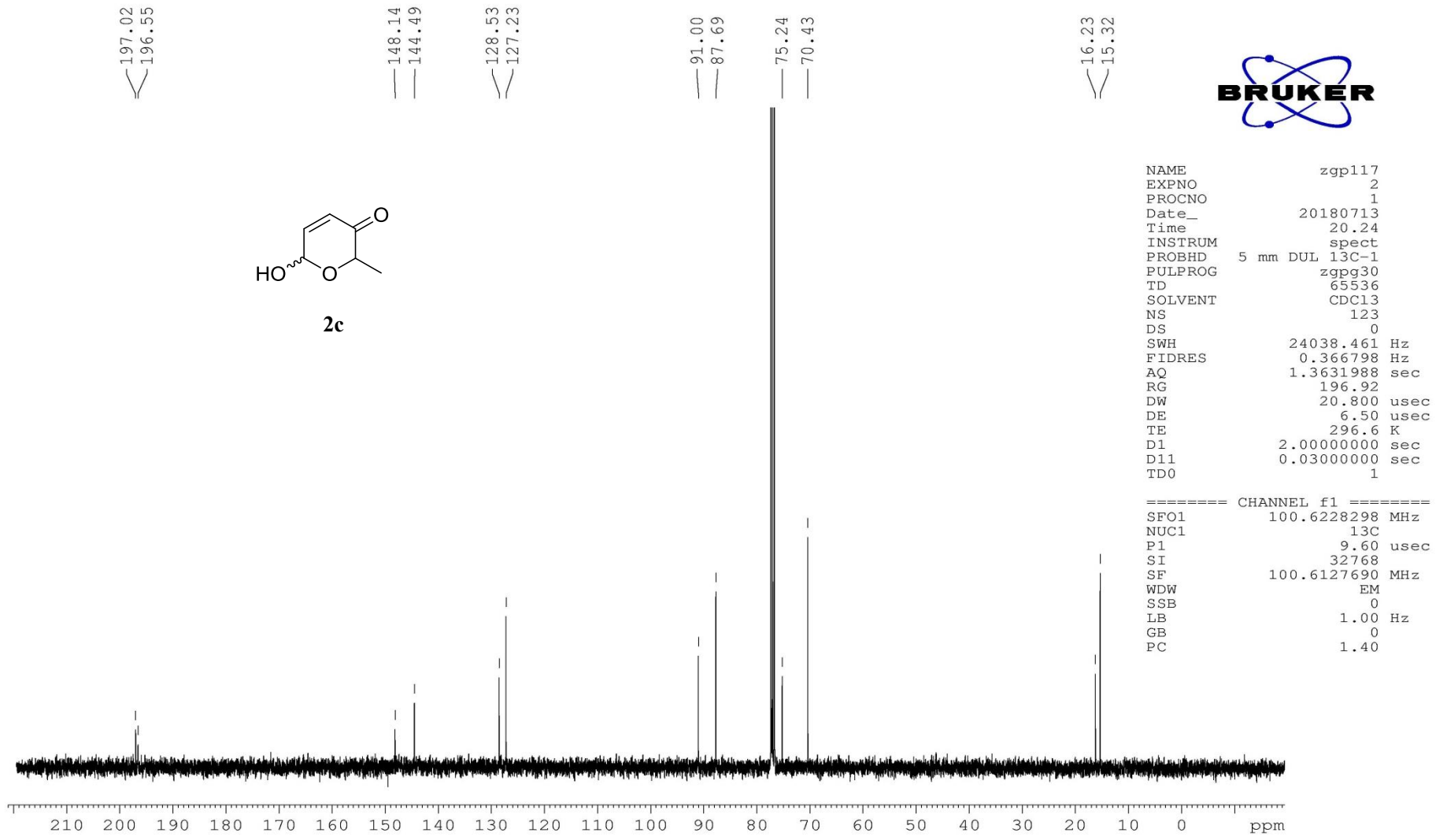
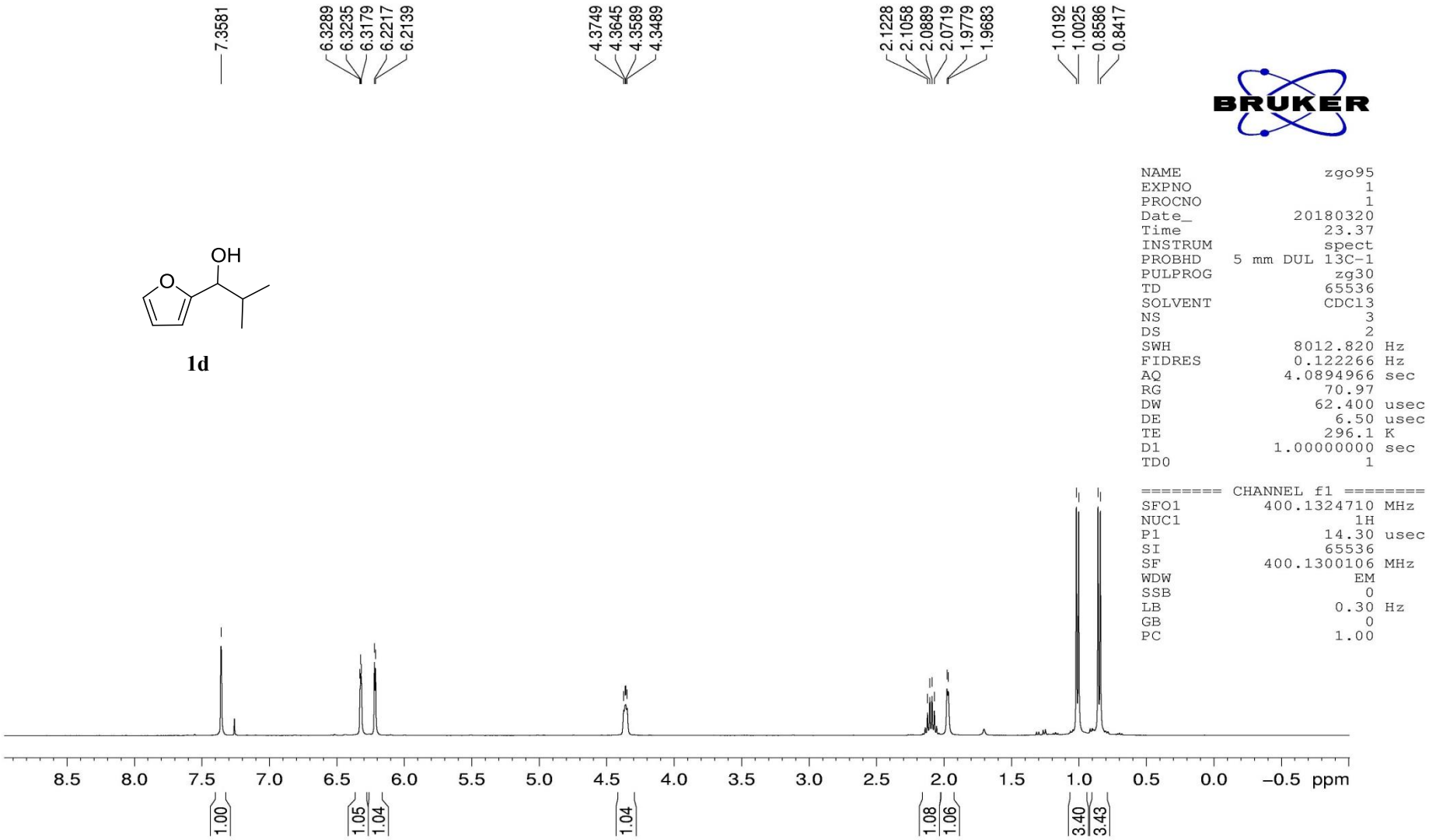
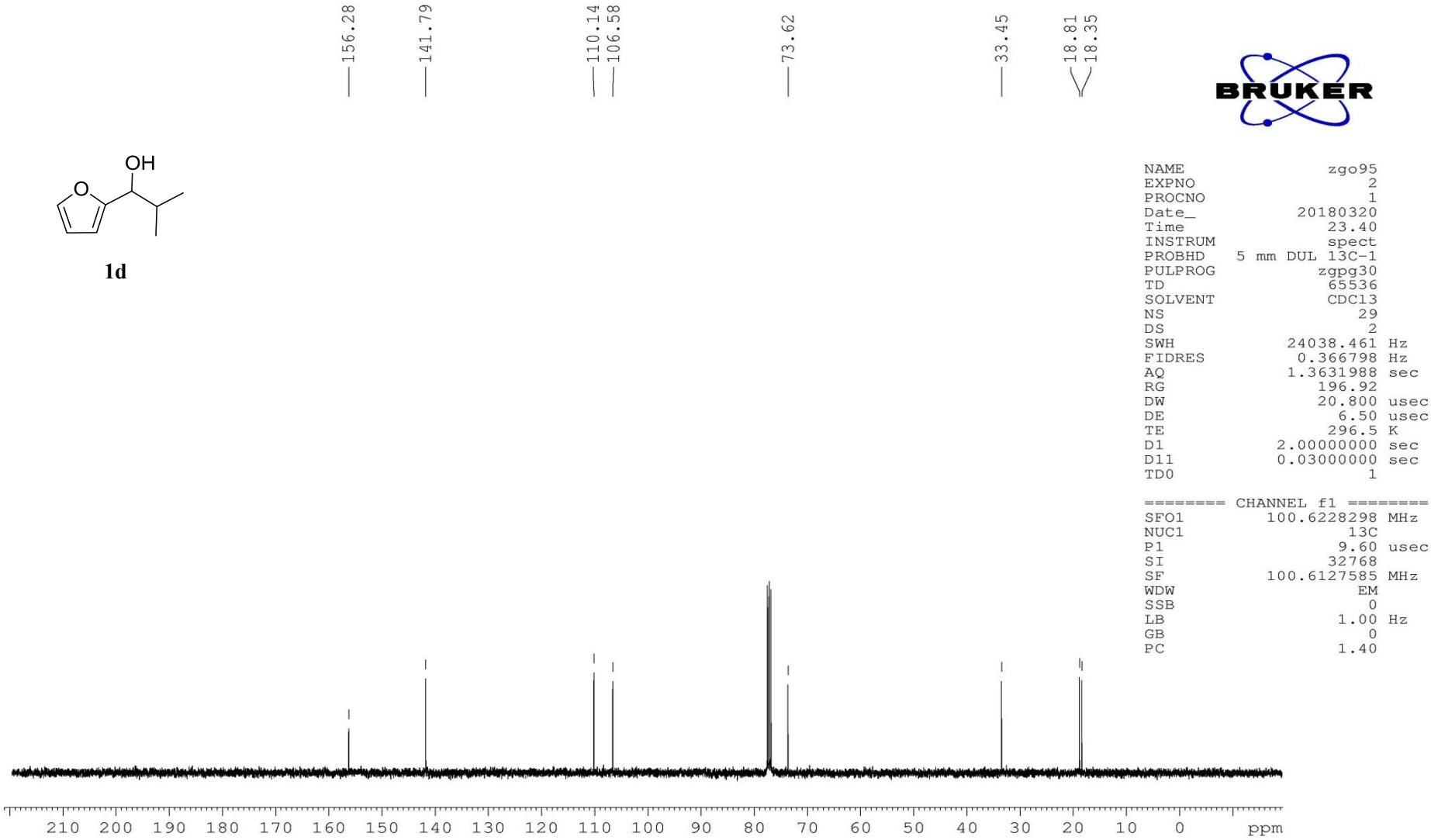
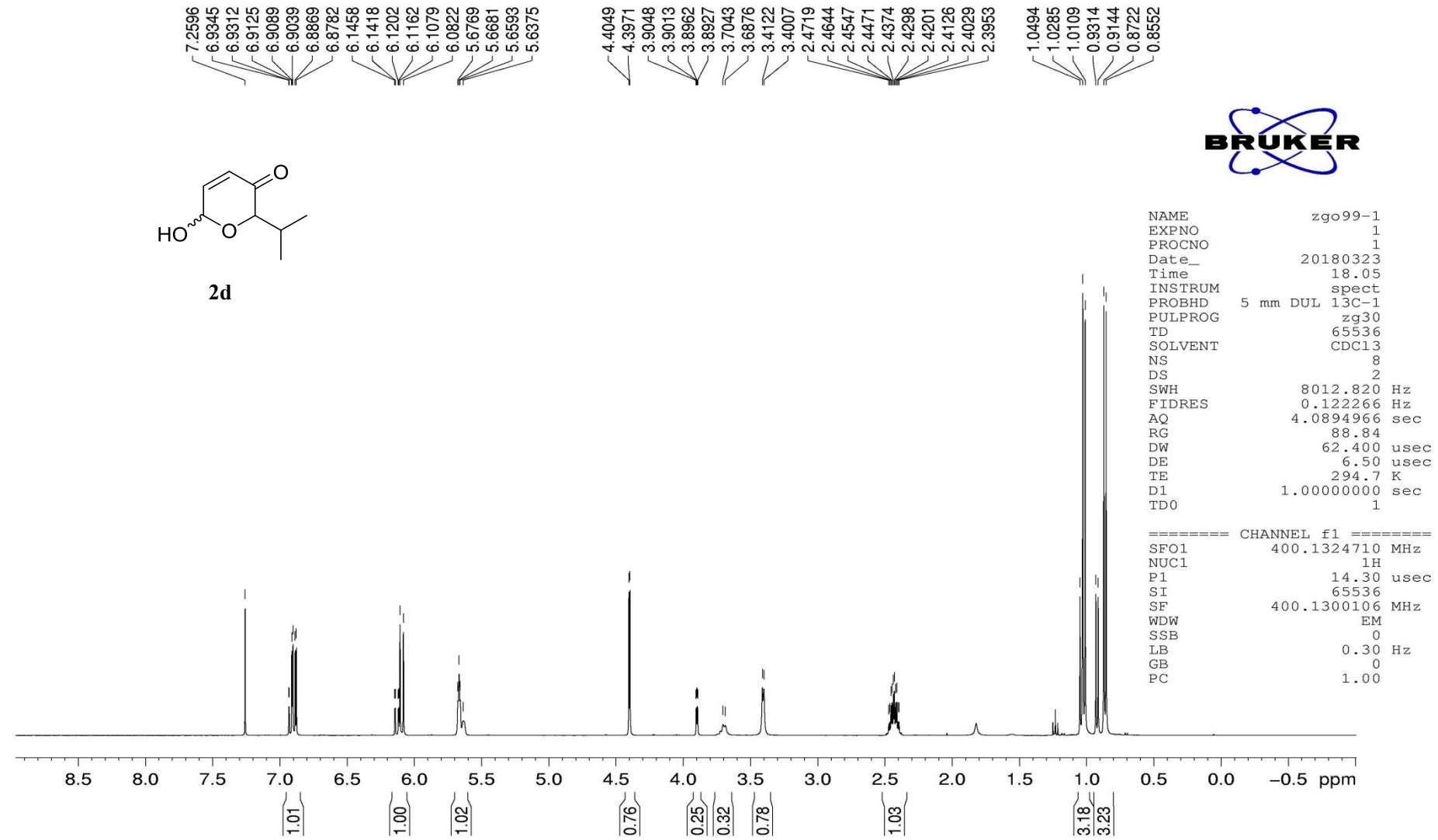
AQ 4.0894966 sec
RG 112.31
DW 62.400 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HO
O
O
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
ppm
1.00
1.00
1.00
0.66
0.34
3.05

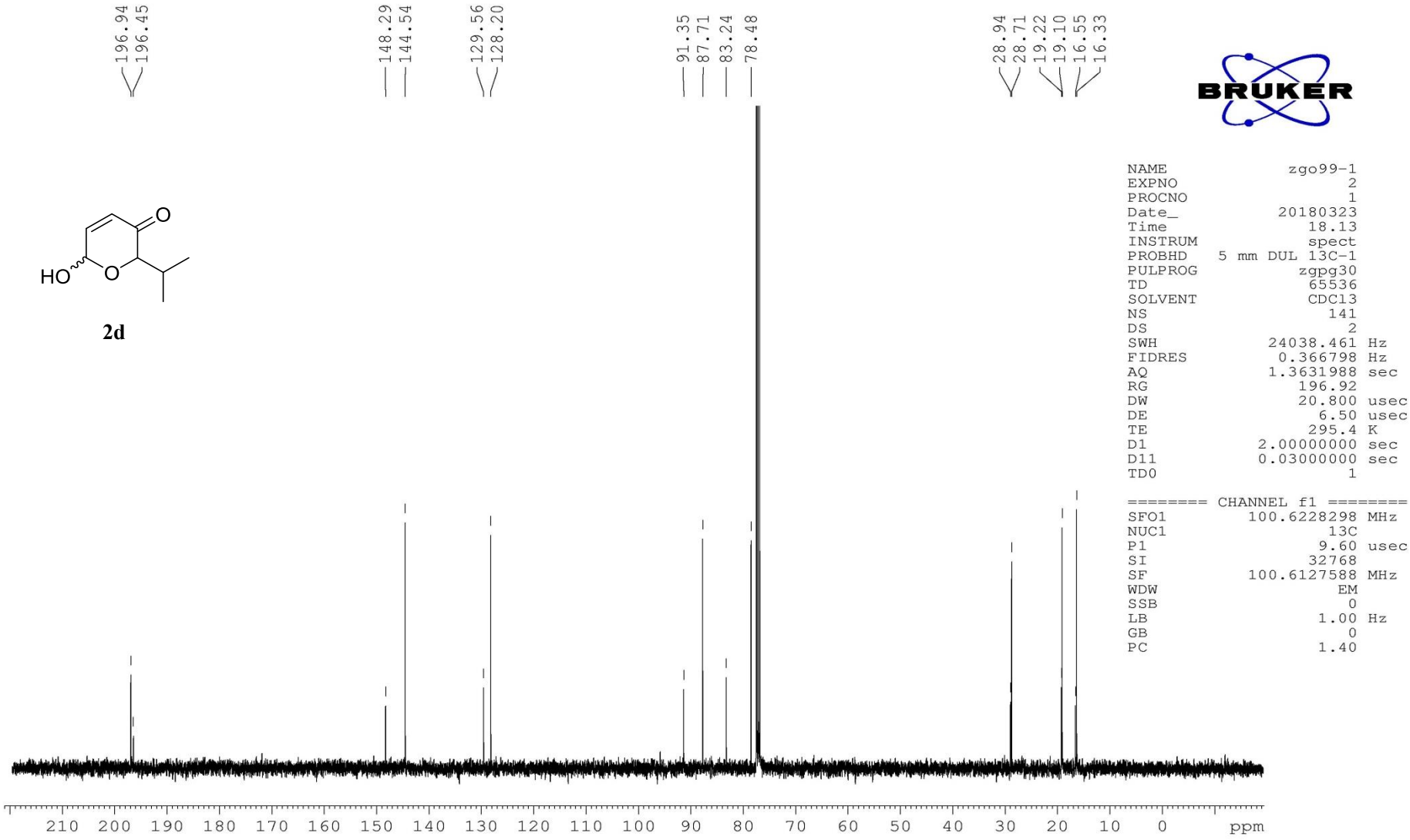
**2c**

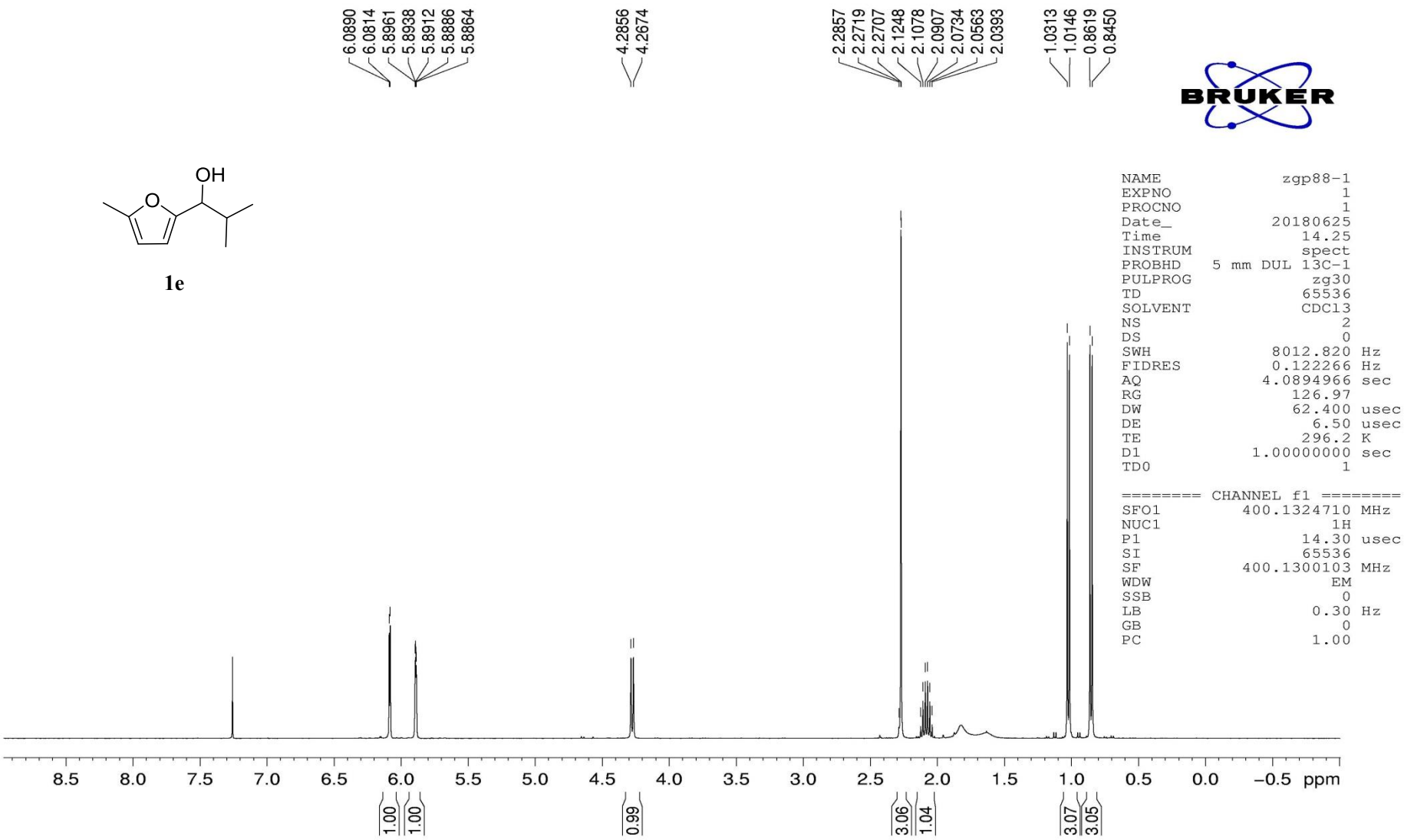

197.02
196.55
148.14
144.49
128.53
127.23
91.00
87.69
75.24
70.43
16.23
15.32
HO
O
O
2c
BRUKER
NAME zgp117
EXPNO 2
PROCNO 1
Date_ 20180713
Time 20.24
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 123
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

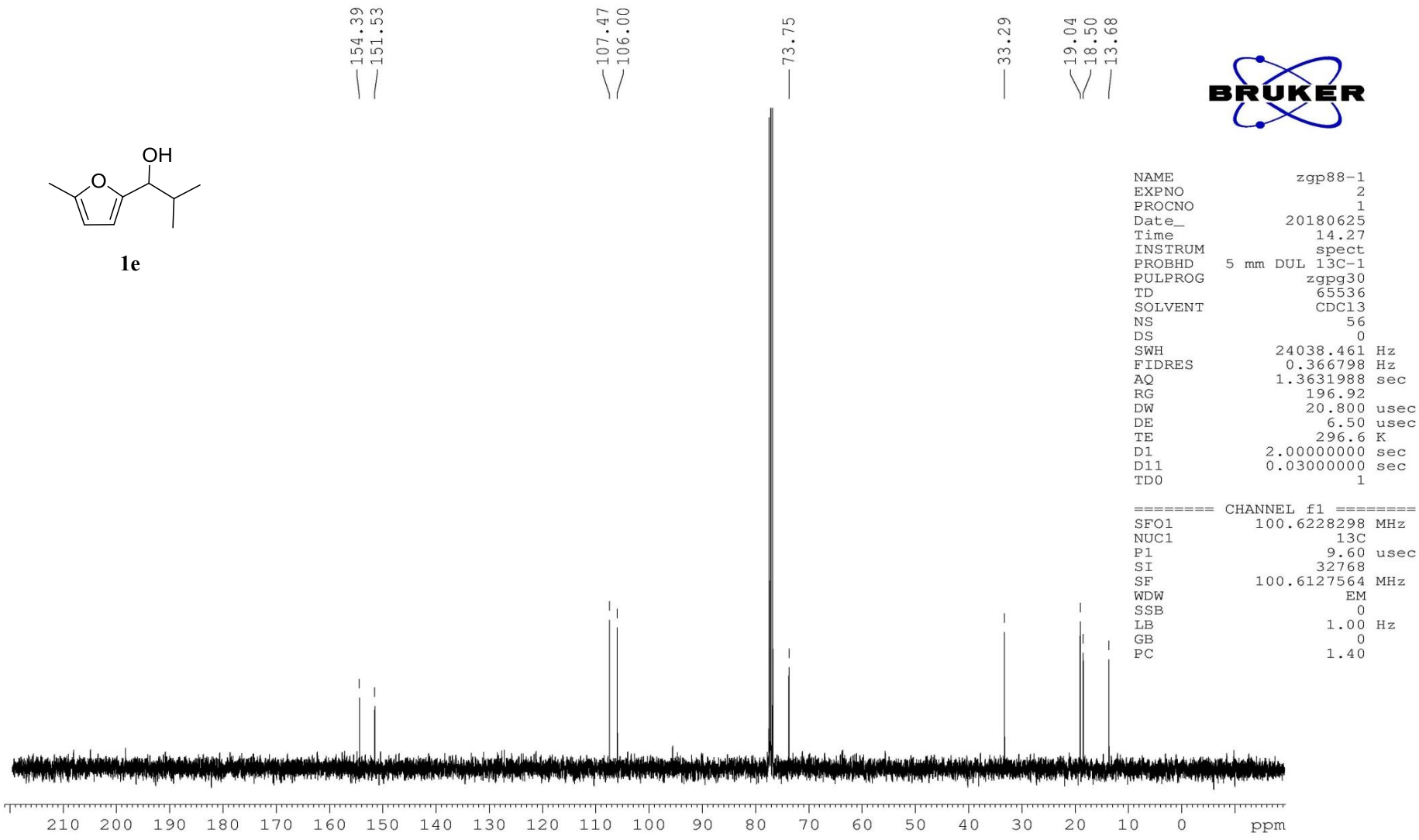

7.3581
6.3289
6.3235
6.3179
6.2217
6.2139
4.3749
4.3645
4.3589
4.3489
2.1228
2.1058
2.0889
2.0719
1.9779
1.9683
1.0192
1.0025
0.8586
0.8417
BRUKER
OH
O
1d
NAME zgo95
EXPNO 1
PROCNO 1
Date_ 20180320
Time 23.37
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 70.97
DW 62.400 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.00
1.05
1.04
1.04
1.08
1.06
3.40
3.43

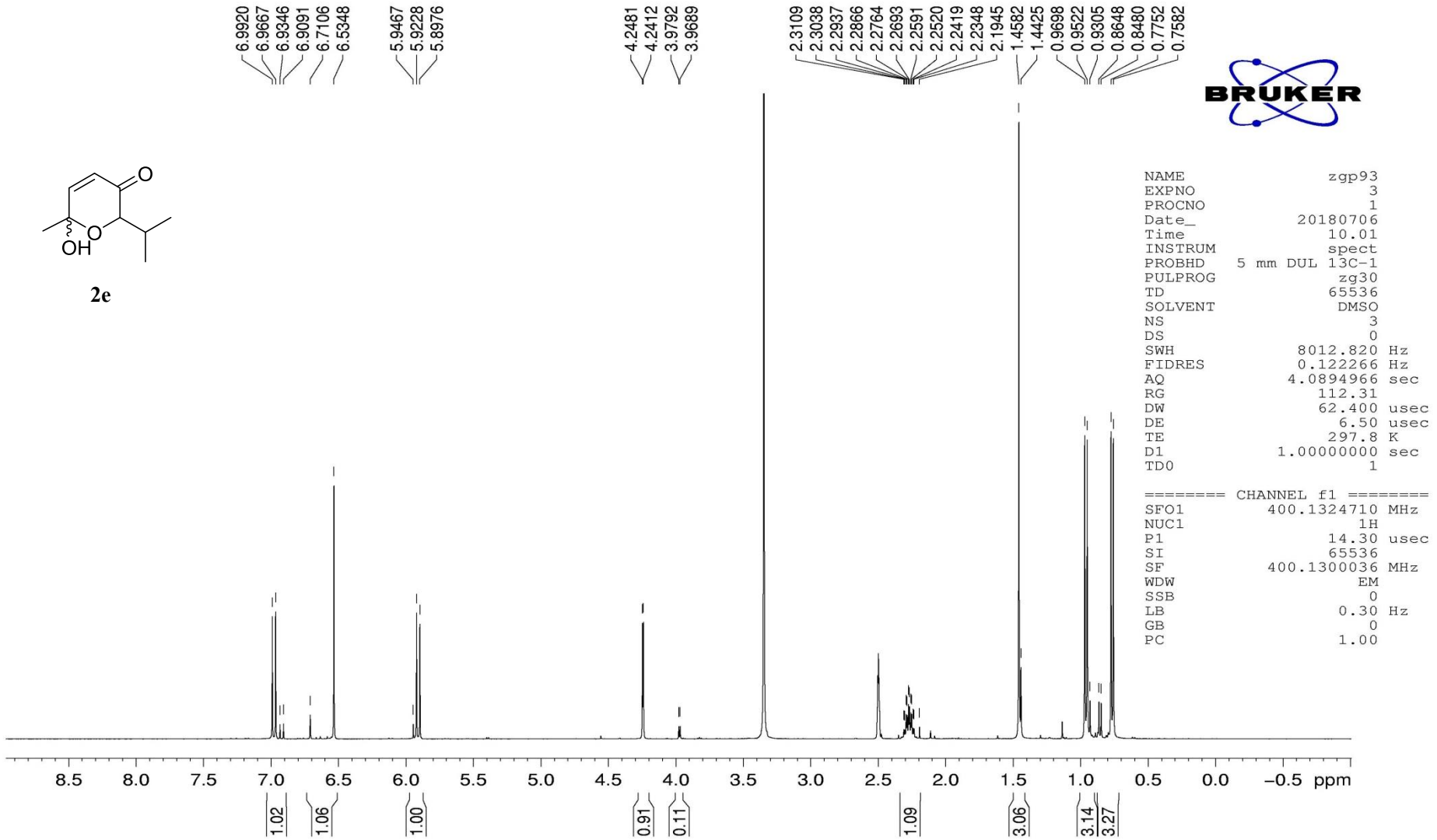

156.28
141.79
110.14
106.58
73.62
33.45
18.81
18.35
OH
O
1d
NAME zgo95
EXPNO 2
PROCNO 1
Date_ 20180320
Time 23.40
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 29
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127585 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.2596
6.9345
6.9312
6.9125
6.9089
6.9039
6.8869
6.8782
6.1458
6.1418
6.1202
6.1162
6.1079
6.0822
5.6769
5.6681
5.6593
5.6375
4.4049
4.3971
3.9048
3.9013
3.8962
3.8927
3.7043
3.6876
3.4122
3.4007
2.4719
2.4644
2.4547
2.4471
2.4374
2.4298
2.4201
2.4126
2.4029
2.3953
1.0494
1.0285
1.0109
0.9314
0.9144
0.8722
0.8552
HO
O
O
2d
BRUKER
NAME zgo99-1
EXPNO 1
PROCNO 1
Date_ 20180323
Time 18.05
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 88.84
DW 62.400 usec
DE 6.50 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.01
1.00
1.02
0.76
0.25
0.32
0.78
1.03
3.18
3.23

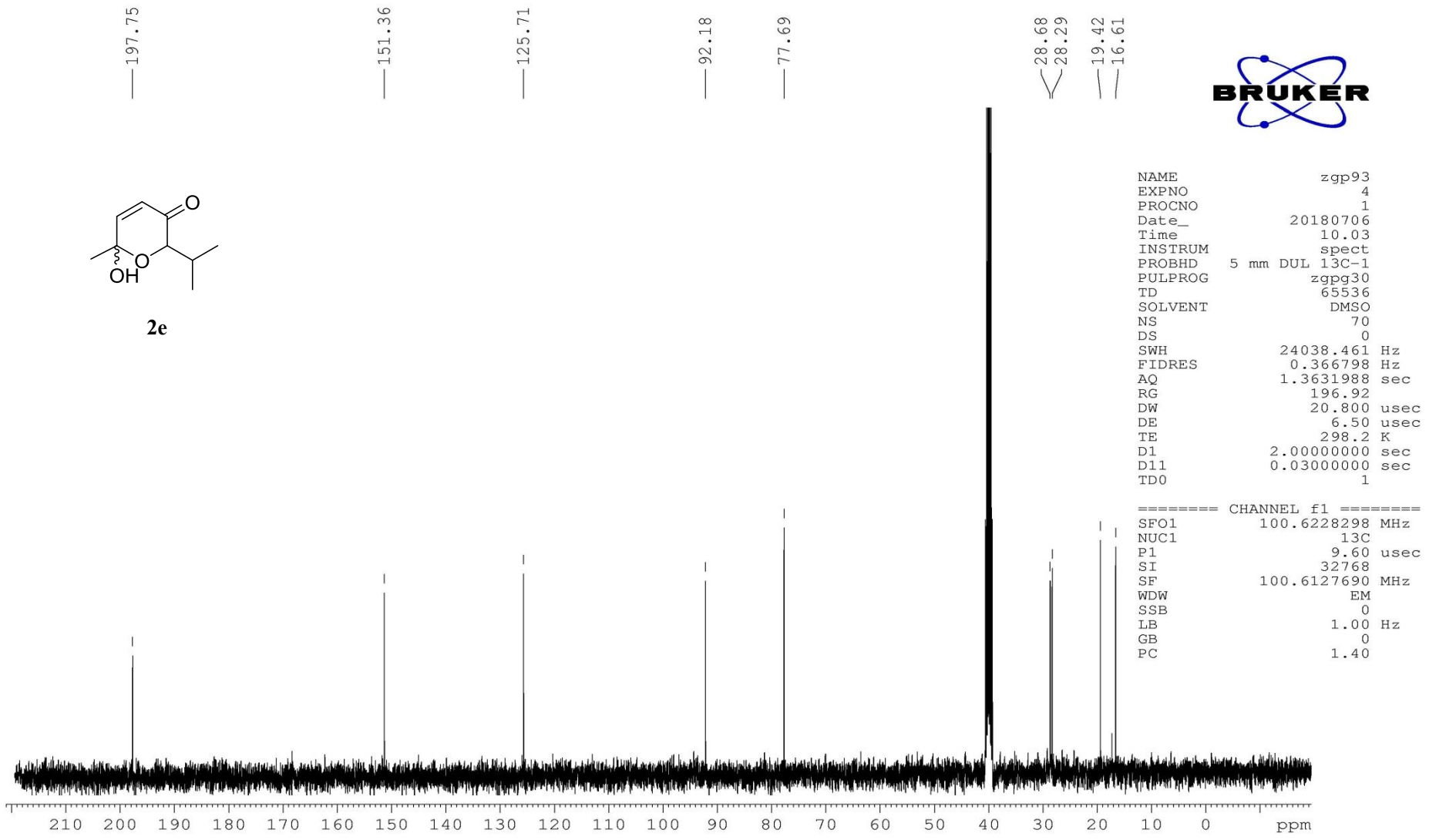

196.94
196.45
148.29
144.54
129.56
128.20
91.35
87.71
83.24
78.48
28.94
28.71
19.22
19.10
16.55
16.33
BRUKER
NAME zgo99-1
EXPNO 2
PROCNO 1
Date_ 20180323
Time 18.13
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 141
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 295.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127588 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
HO
O
O
2d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

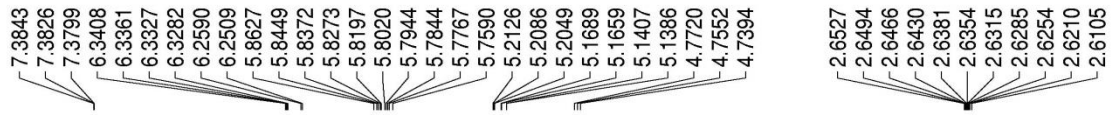

6.0890
6.0814
5.8961
5.8938
5.8912
5.8886
5.8864
4.2856
4.2674
2.2857
2.2719
2.2707
2.1248
2.1078
2.0907
2.0734
2.0563
2.0393
1.0313
1.0146
0.8619
0.8450
OH
O
1e
BRUKER
NAME zgp88-1
EXPNO 1
PROCNO 1
Date_ 20180625
Time 14.25
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 2
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 126.97
DW 62.400 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.00
1.00
0.99
3.06
1.04
3.07
3.05

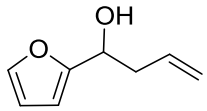

154.39
151.53
107.47
106.00
73.75
33.29
19.04
18.50
13.68
OH
O
1e
BRUKER
NAME zgp88-1
EXPNO 2
PROCNO 1
Date_ 20180625
Time 14.27
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 56
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127564 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


6.9920
6.9667
6.9346
6.9091
6.7106
6.5348
5.9467
5.9228
5.8976
4.2481
4.2412
3.9792
3.9689
2.3109
2.3038
2.2937
2.2866
2.2764
2.2693
2.2591
2.2520
2.2419
2.2348
2.1945
1.4582
1.4425
0.9698
0.9522
0.9305
0.8648
0.8480
0.7752
0.7582
O
O
OH
BRUKER
NAME zgp93
EXPNO 3
PROCNO 1
Date_ 20180706
Time 10.01
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 3
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 112.31
DW 62.400 usec
DE 6.50 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300036 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.02
1.06
1.00
0.91
0.11
1.09
3.06
3.14
3.27

**2e**

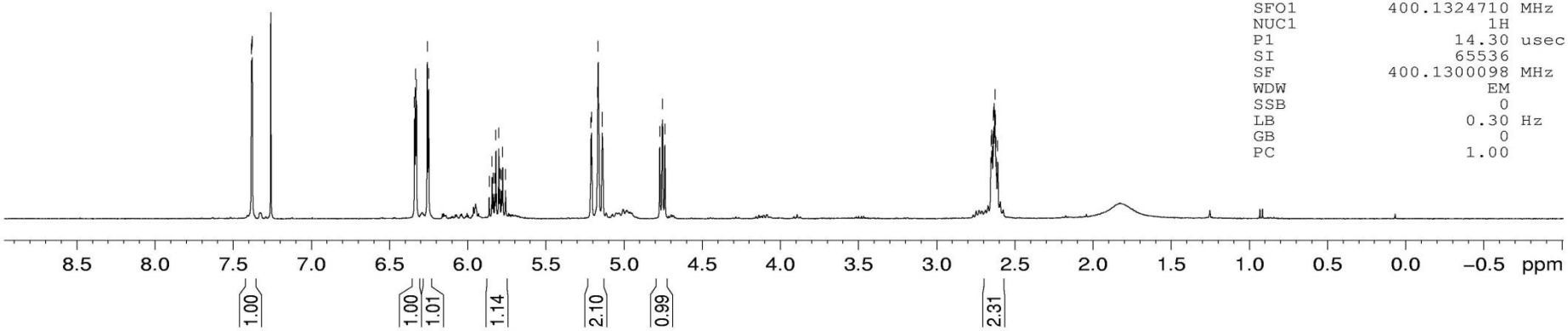

197.75
151.36
125.71
92.18
77.69
28.68
28.29
19.42
16.61
O
O
OH
2e
BRUKER
NAME zgp93
EXPNO 4
PROCNO 1
Date_ 20180706
Time 10.03
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 70
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

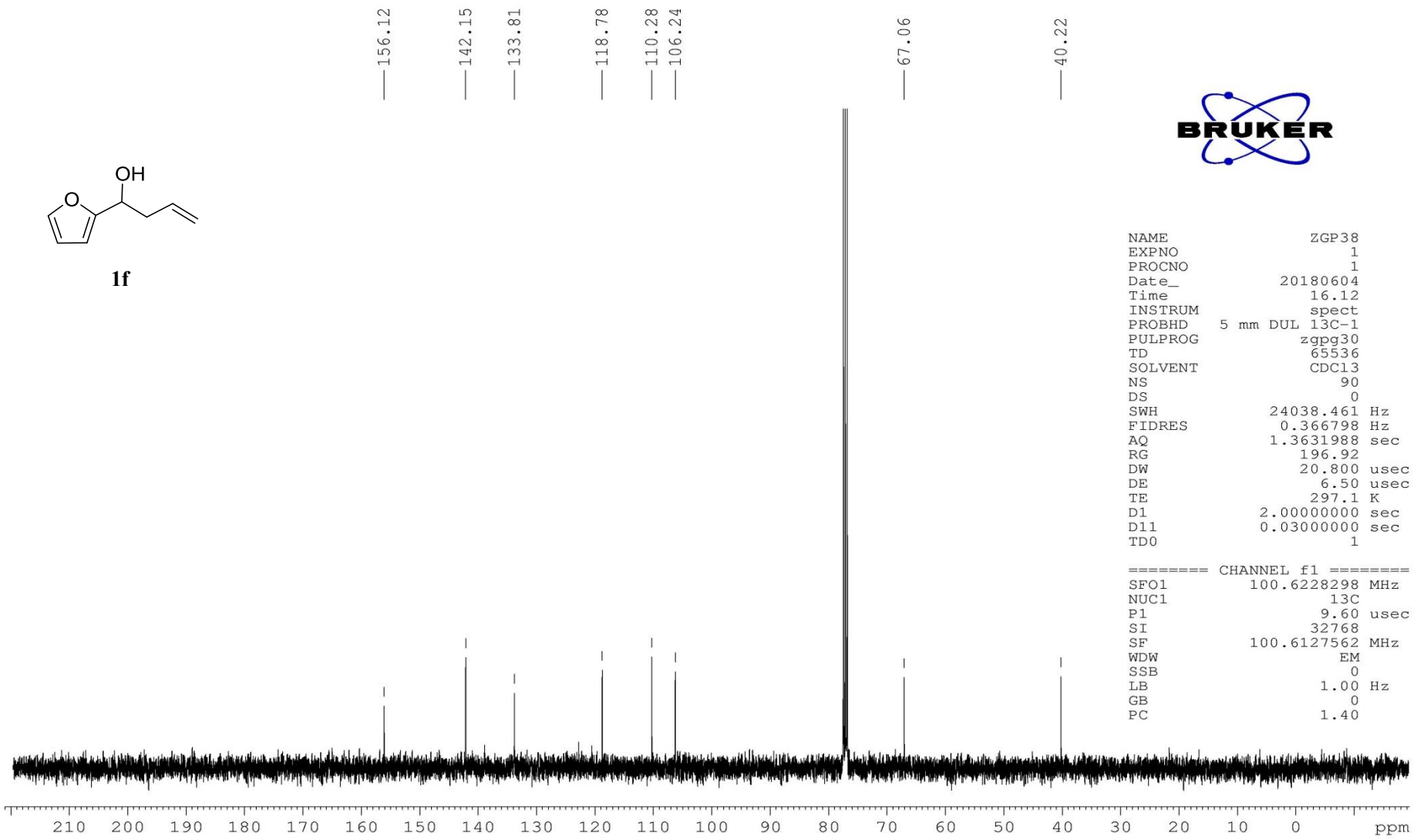


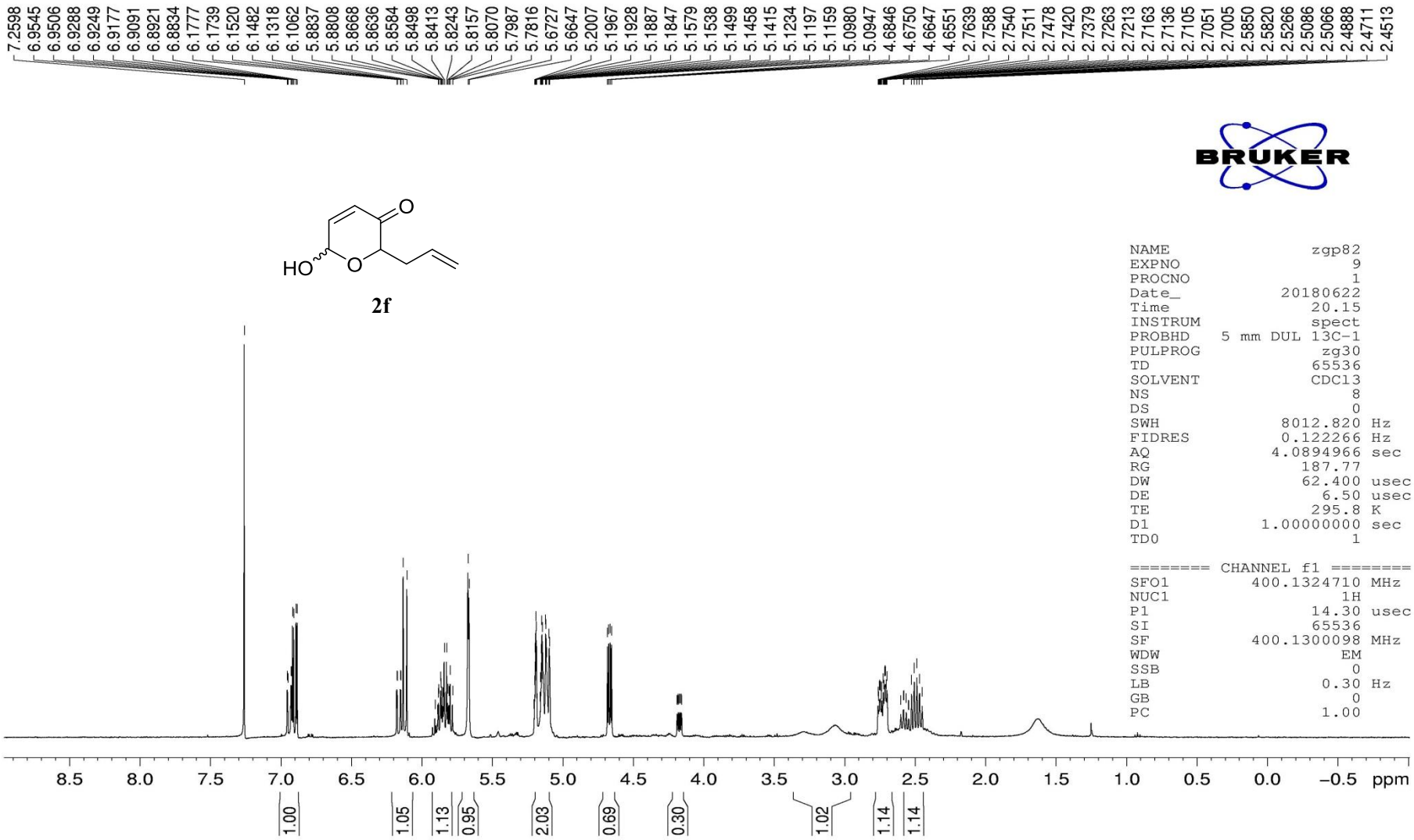



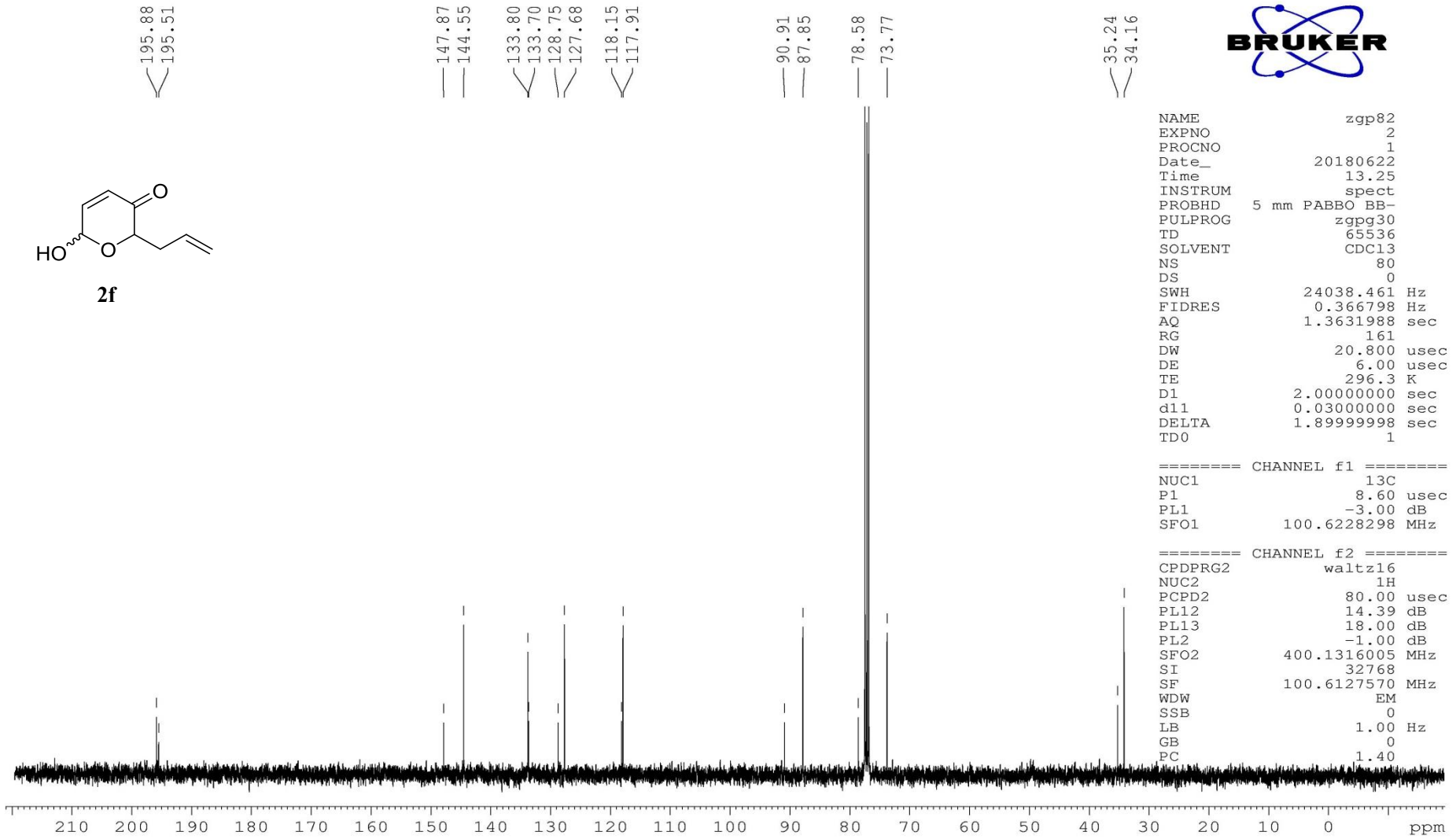

**1f**

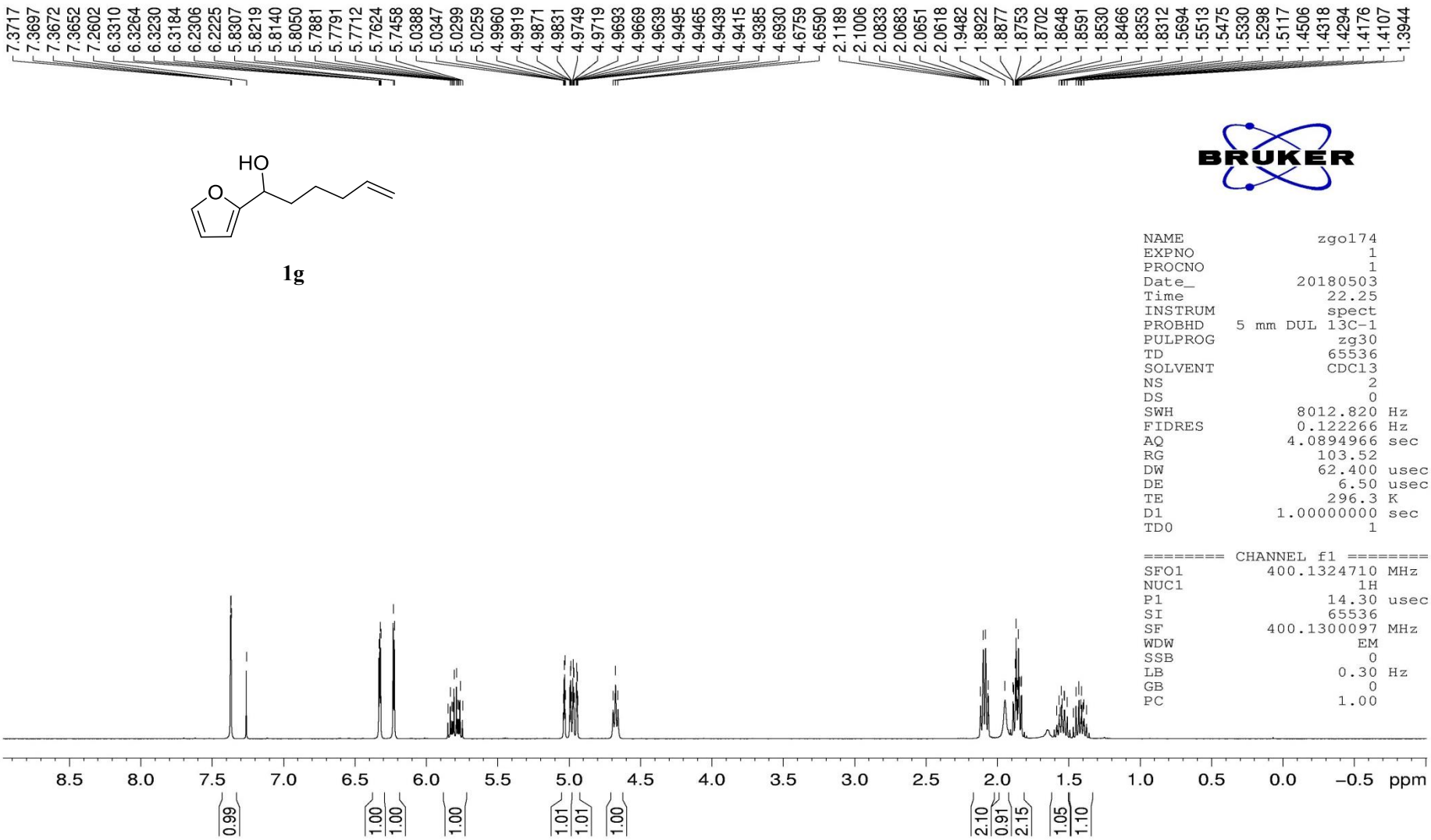
```
NAME                ZGP38
EXPNO                   2
PROCNO                  1
Date_            20180604
Time                16.07
INSTRUM             spect
PROBHD    5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      4
DS                      0
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894966 sec
RG                 196.92
DW                 62.400 usec
DE                  6.50 usec
TE                 296.7 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 14.30 usec
SI                  65536
SF           400.1300098 MHz
WDW                    EM
SSB                     0
LB                  0.30 Hz
GB                      0
PC                   1.00
```



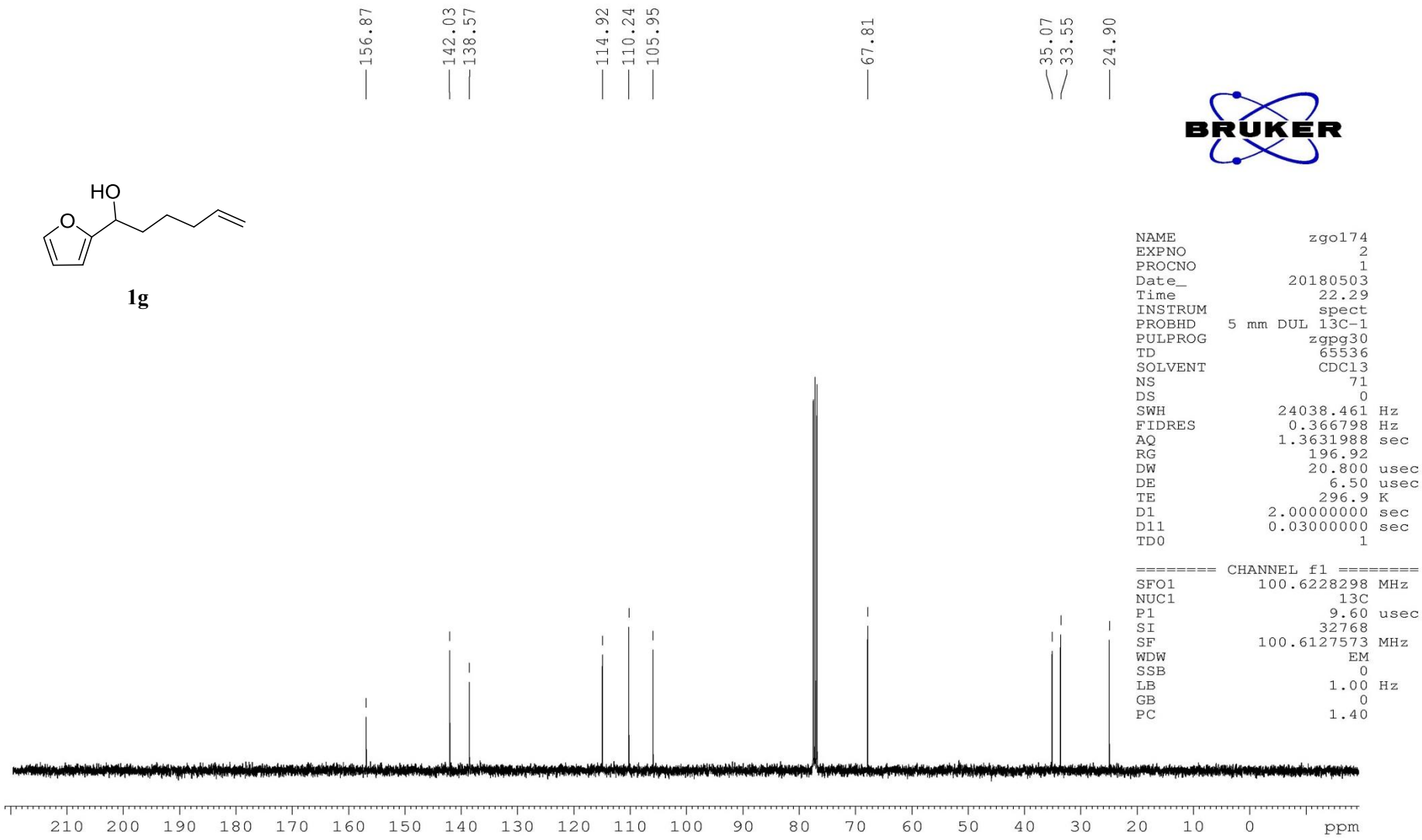

156.12
142.15
133.81
118.78
110.28
106.24
67.06
40.22
OH
O
1f
BRUKER
NAME ZGP38
EXPNO 1
PROCNO 1
Date_ 20180604
Time 16.12
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 90
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127562 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

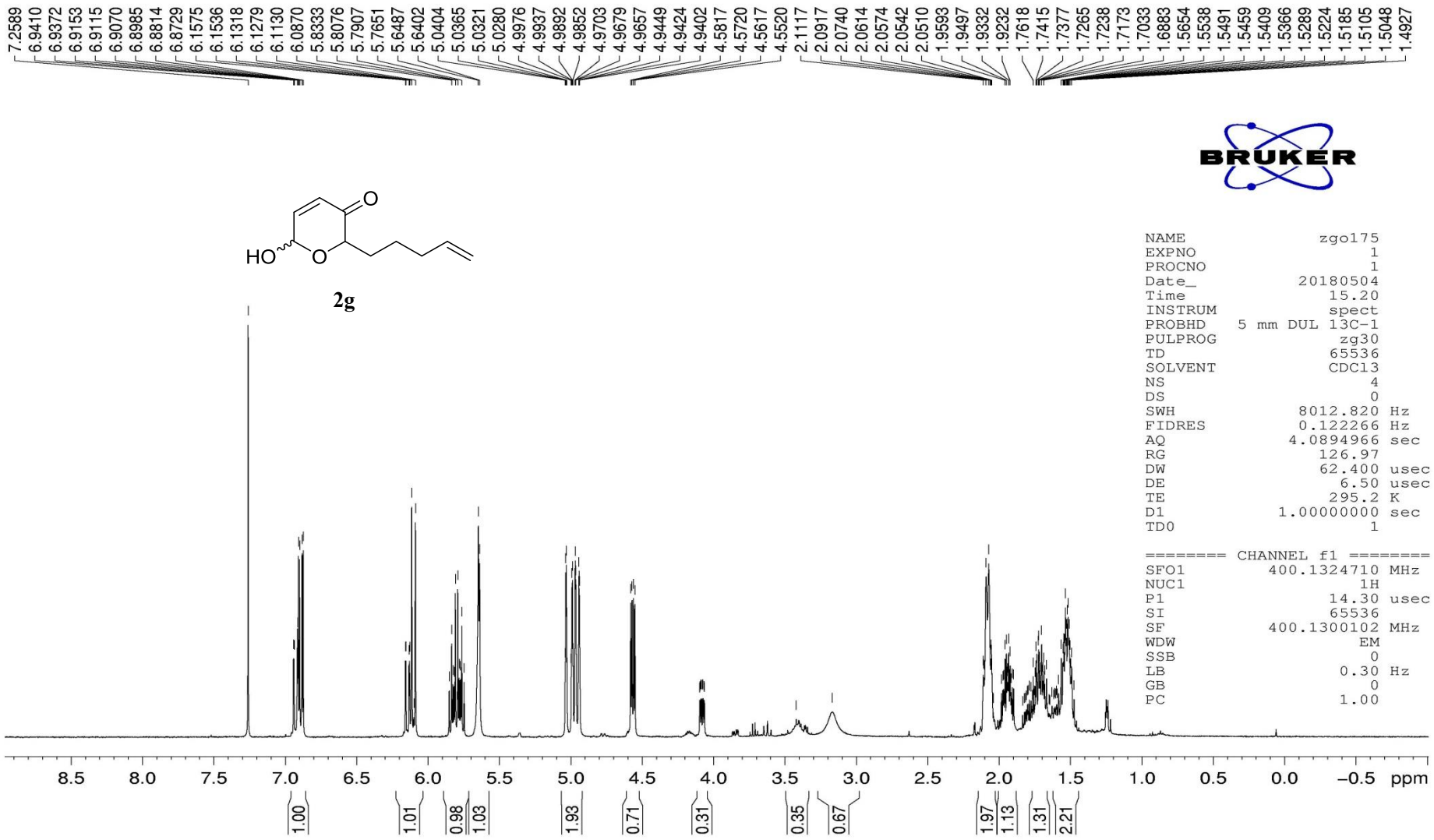

7.2598
6.9545
6.9506
6.9288
6.9249
6.9177
6.9091
6.8921
6.8834
6.1777
6.1739
6.1520
6.1482
6.1318
6.1062
5.8837
5.8808
5.8668
5.8636
5.8584
5.8498
5.8413
5.8243
5.8157
5.8070
5.7987
5.7816
5.6727
5.6647
5.2007
5.1967
5.1928
5.1887
5.1847
5.1579
5.1538
5.1499
5.1458
5.1415
5.1234
5.1197
5.1159
5.0980
5.0947
4.6846
4.6750
4.6647
4.6551
2.7639
2.7588
2.7540
2.7511
2.7478
2.7420
2.7379
2.7263
2.7213
2.7163
2.7136
2.7105
2.7051
2.7005
2.5850
2.5820
2.5266
2.5086
2.5066
2.4888
2.4711
2.4513
BRUKER
HO
O
O
2f
NAME zgp82
EXPNO 9
PROCNO 1
Date_ 20180622
Time 20.15
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 187.77
DW 62.400 usec
DE 6.50 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.00
1.05
1.13
0.95
2.03
0.69
0.30
1.02
1.14
1.14

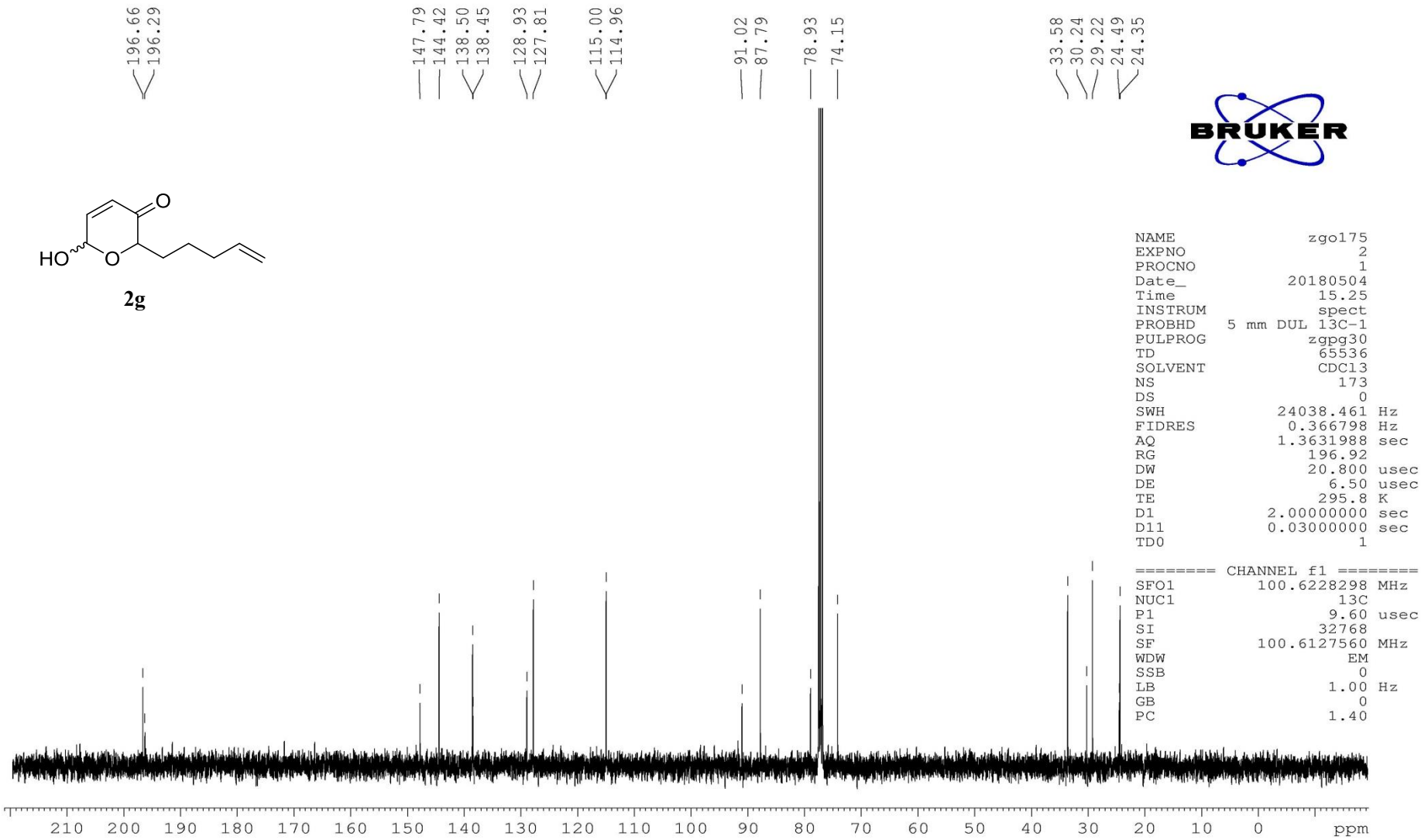

195.88
195.51
147.87
144.55
133.80
133.70
128.75
127.68
118.15
117.91
90.91
87.85
78.58
73.77
35.24
34.16
BRUKER
HO
O
O
2f
NAME zgp82
EXPNO 2
PROCNO 1
Date_ 20180622
Time 13.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 80
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 161
DW 20.800 usec
DE 6.00 usec
TE 296.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 14.39 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127570 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

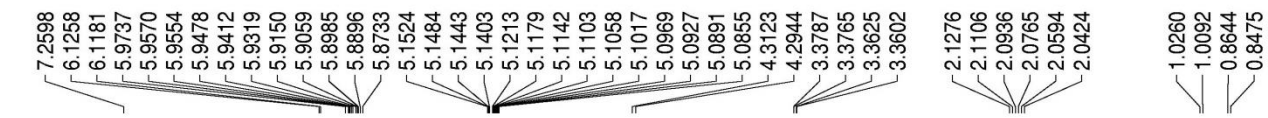

7.3717
7.3697
7.3672
7.3652
7.2602
6.3310
6.3264
6.3230
6.3184
6.2306
6.2225
5.8307
5.8219
5.8140
5.8050
5.7881
5.7791
5.7712
5.7624
5.7458
5.0388
5.0347
5.0299
5.0259
4.9960
4.9919
4.9871
4.9831
4.9749
4.9719
4.9693
4.9669
4.9639
4.9495
4.9465
4.9439
4.9415
4.9385
4.6930
4.6759
4.6590
2.1189
2.1006
2.0833
2.0683
2.0651
2.0618
1.9482
1.8922
1.8877
1.8753
1.8702
1.8648
1.8591
1.8530
1.8466
1.8353
1.8312
1.5694
1.5513
1.5475
1.5330
1.5298
1.5117
1.4506
1.4318
1.4294
1.4176
1.4107
1.3944
HO
O
BRUKER
NAME zgo174
EXPNO 1
PROCNO 1
Date_ 20180503
Time 22.25
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 2
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 103.52
DW 62.400 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
0.99
1.00
1.00
1.00
1.01
1.01
1.00
2.10
0.91
2.15
1.05
1.10

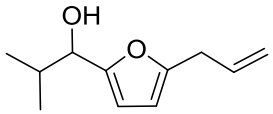
**1g**


156.87
142.03
138.57
114.92
110.24
105.95
67.81
35.07
33.55
24.90
HO
O
1g
BRUKER
NAME zgo174
EXPNO 2
PROCNO 1
Date_ 20180503
Time 22.29
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 71
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127573 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

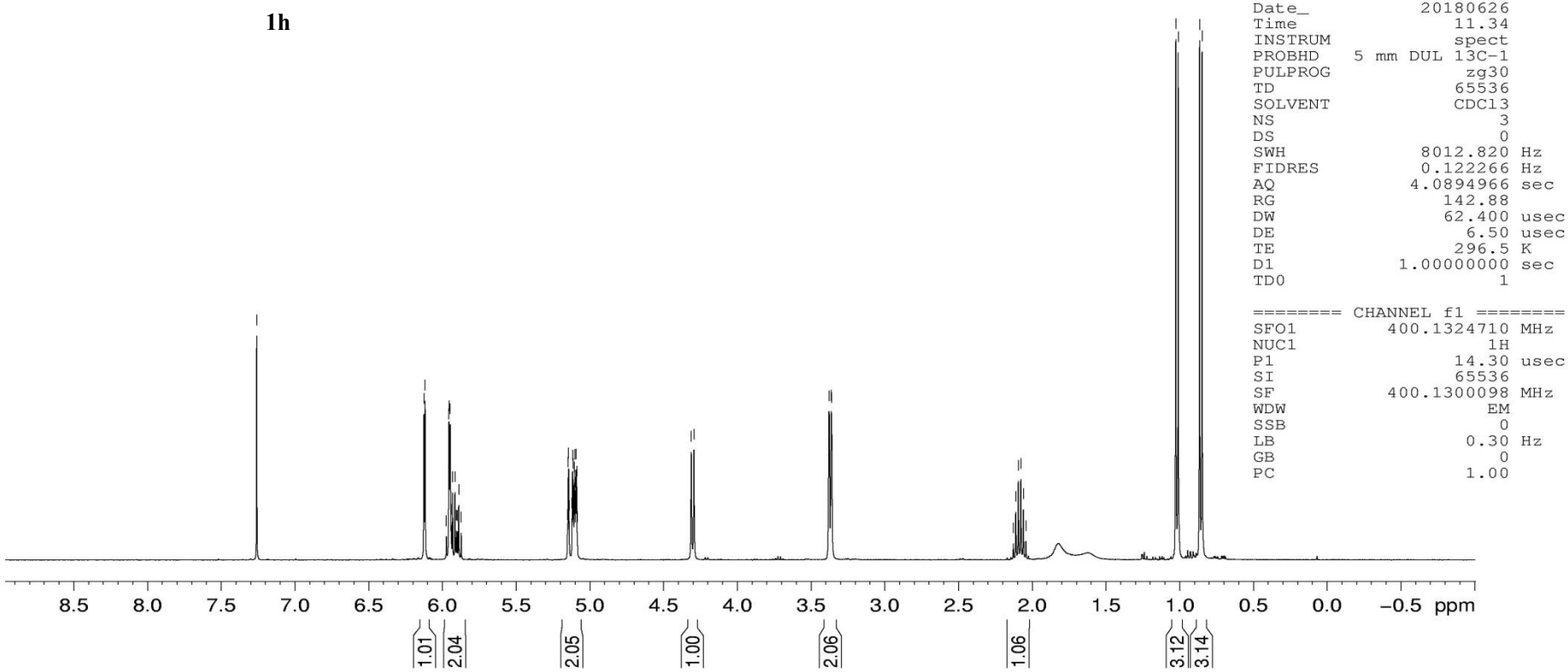

7.2589 6.9410 6.9372 6.9153 6.9115 6.9070 6.8985 6.8814 6.8729 6.1575 6.1536 6.1318 6.1279 6.1130 6.0870 5.8333 5.8076 5.7907 5.7651 5.6487 5.6402 5.0404 5.0365 5.0321 5.0280 4.9976 4.9937 4.9892 4.9852 4.9703 4.9679 4.9657 4.9449 4.9424 4.9402 4.5817 4.5720 4.5617 4.5520 2.1117 2.0917 2.0740 2.0614 2.0574 2.0542 2.0510 1.9593 1.9497 1.9332 1.9232 1.7618 1.7415 1.7377 1.7265 1.7238 1.7173 1.7033 1.6883 1.5654 1.5538 1.5491 1.5459 1.5409 1.5366 1.5289 1.5224 1.5185 1.5105 1.5048 1.4927
BRUKER
HO
O
O
2g
NAME zgo175
EXPNO 1
PROCNO 1
Date_ 20180504
Time 15.20
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 126.97
DW 62.400 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.00 1.01 0.98 1.03 1.93 0.71 0.31 0.35 0.67 1.97 1.13 1.31 2.21

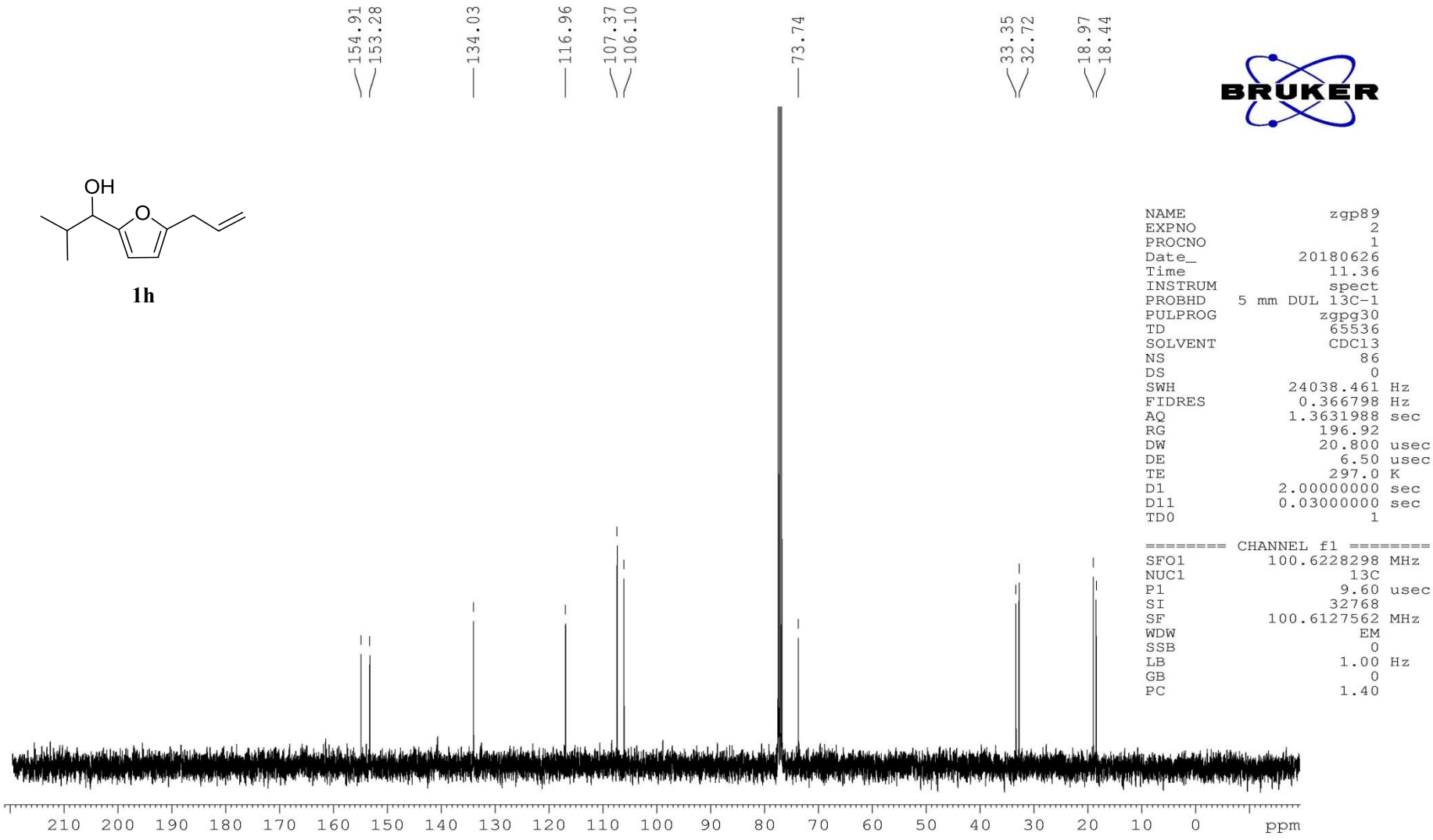

196.66
196.29
147.79
144.42
138.50
138.45
128.93
127.81
115.00
114.96
91.02
87.79
78.93
74.15
33.58
30.24
29.22
24.49
24.35
HO
O
O
2g
BRUKER
NAME zgo175
EXPNO 2
PROCNO 1
Date_ 20180504
Time 15.25
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 173
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 295.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127560 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

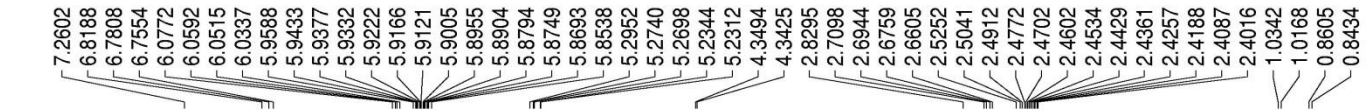

7.2598
6.1258
6.1181
5.9737
5.9570
5.9554
5.9478
5.9412
5.9319
5.9150
5.9059
5.8985
5.8896
5.8733
5.1524
5.1484
5.1443
5.1403
5.1213
5.1179
5.1142
5.1103
5.1058
5.1017
5.0969
5.0927
5.0891
5.0855
4.3123
4.2944
3.3787
3.3765
3.3625
3.3602
2.1276
2.1106
2.0936
2.0765
2.0594
2.0424
1.0260
1.0092
0.8644
0.8475

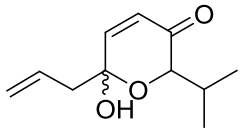

OH
O

**1h**


NAME zgp89
EXPNO 1
PROCNO 1
Date_ 20180626
Time 11.34
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 142.88
DW 62.400 usec
DE 6.50 usec
TE 296.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.01 2.04 2.05 1.00 2.06 1.06 3.12 3.14

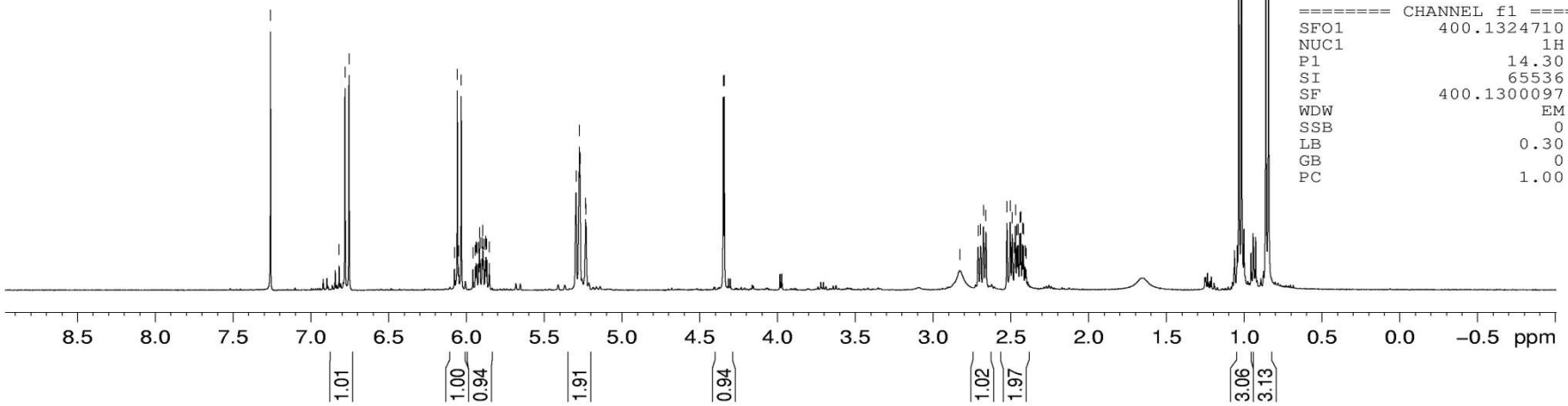

154.91
153.28
134.03
116.96
107.37
106.10
73.74
33.35
32.72
18.97
18.44
OH
O
1h
NAME zgp89
EXPNO 2
PROCNO 1
Date_ 20180626
Time 11.36
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 86
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127562 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

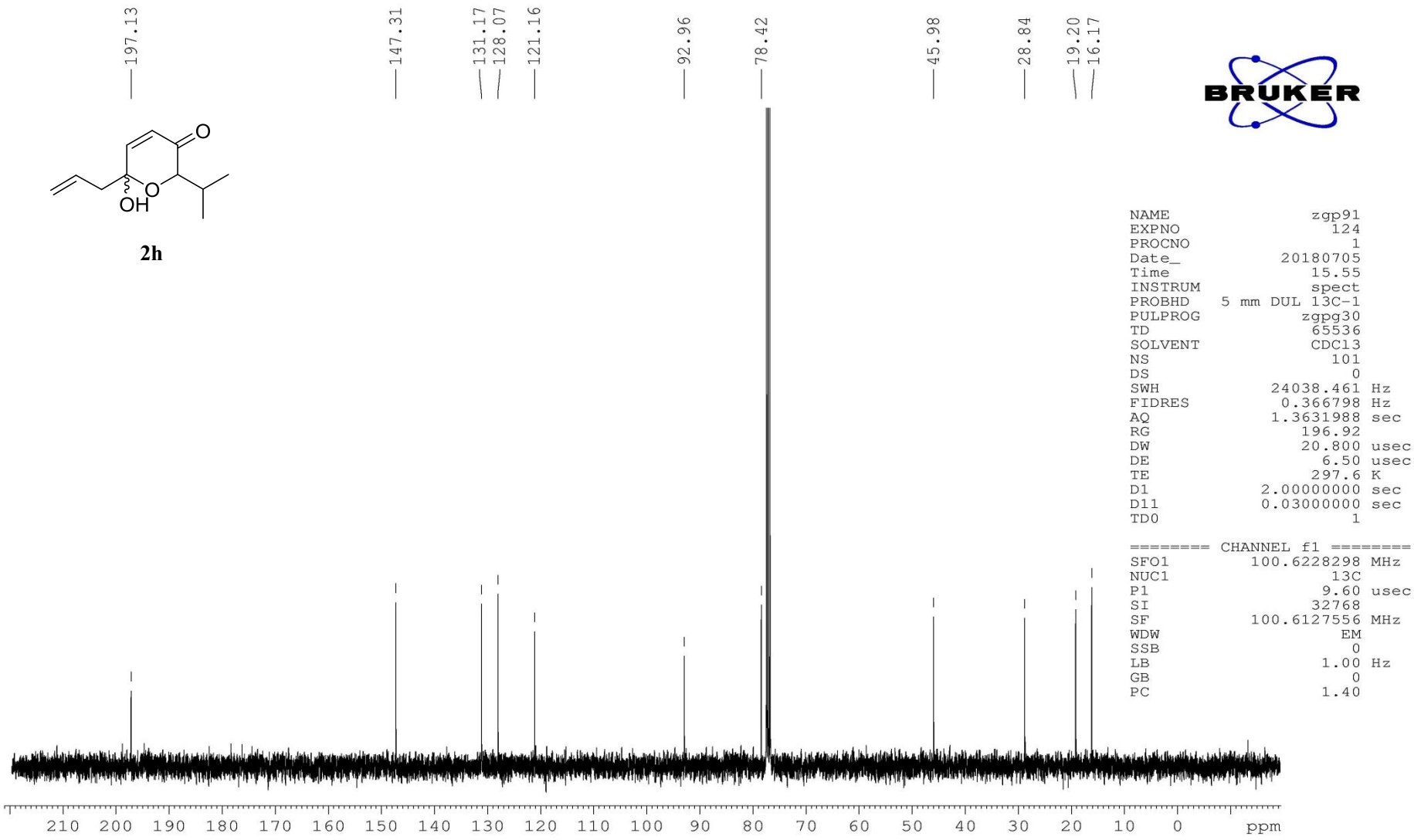


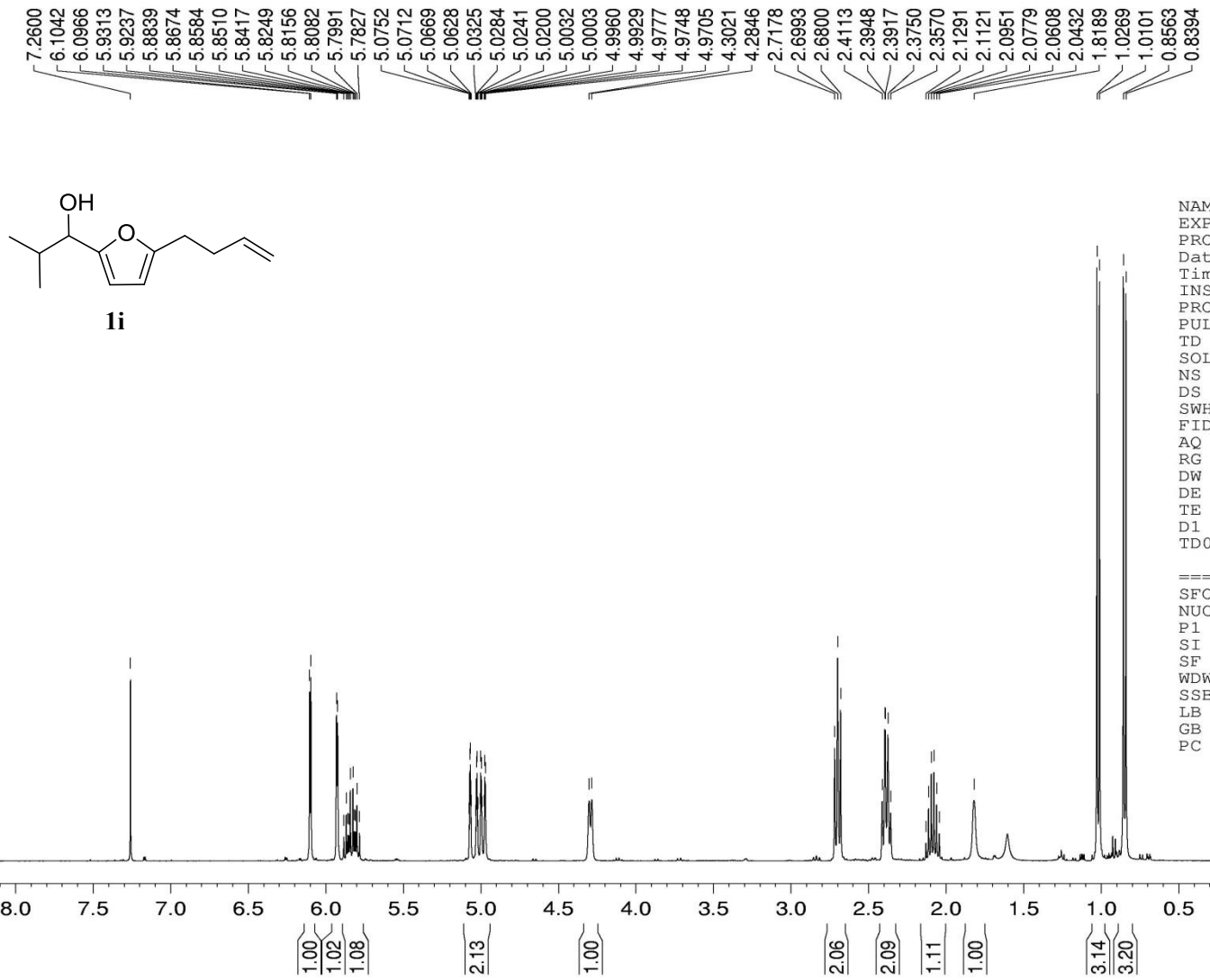

**2h**

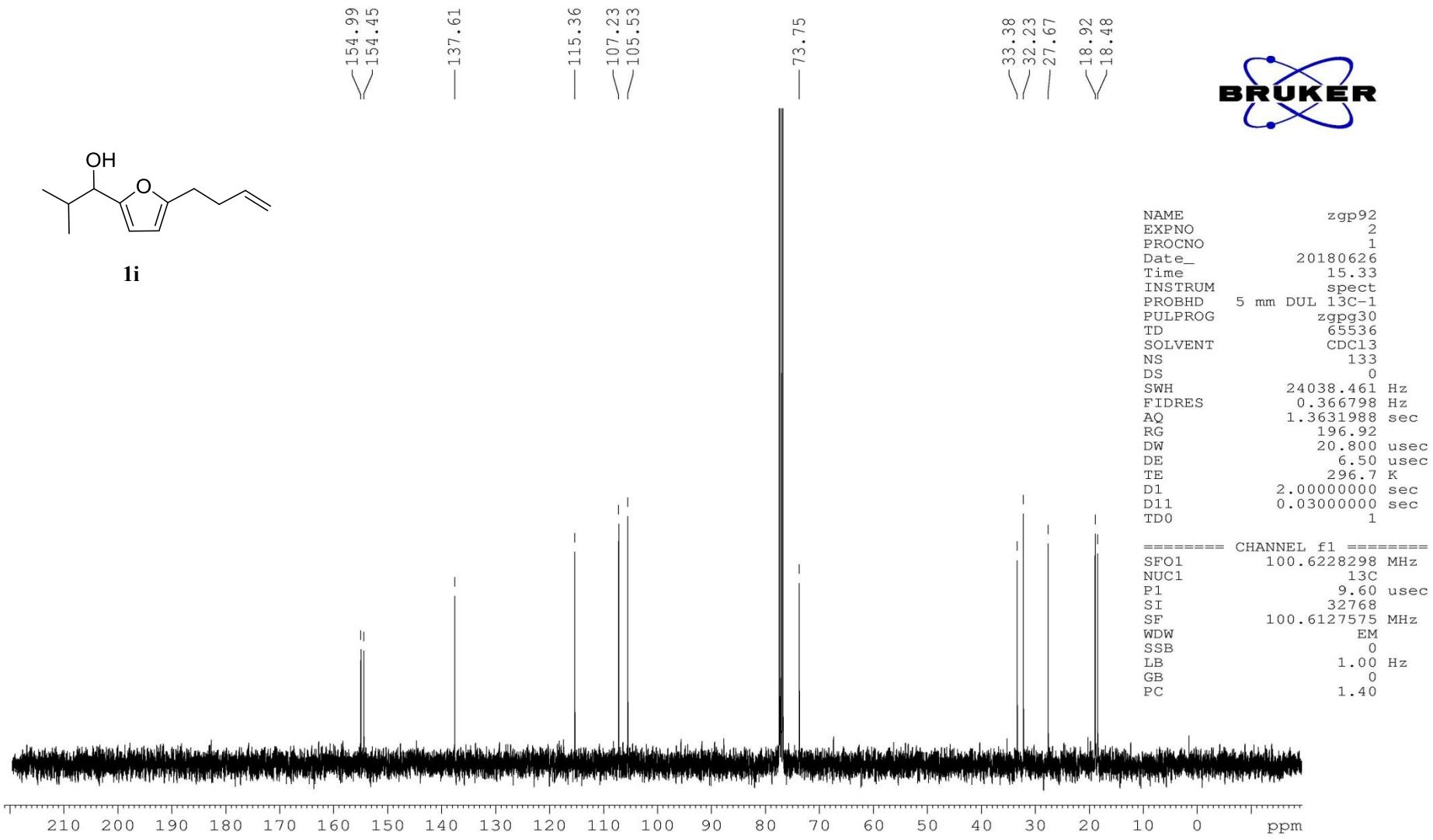
```
NAME                zgp91
EXPNO                 123
PROCNO                  1
Date_            20180705
Time                15.51
INSTRUM             spect
PROBHD    5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      2
DS                      0
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894966 sec
RG                  126.97
DW                  62.400 usec
DE                   6.50 usec
TE                  297.2 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                   1H
P1                  14.30 usec
SI                  65536
SF            400.1300097 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

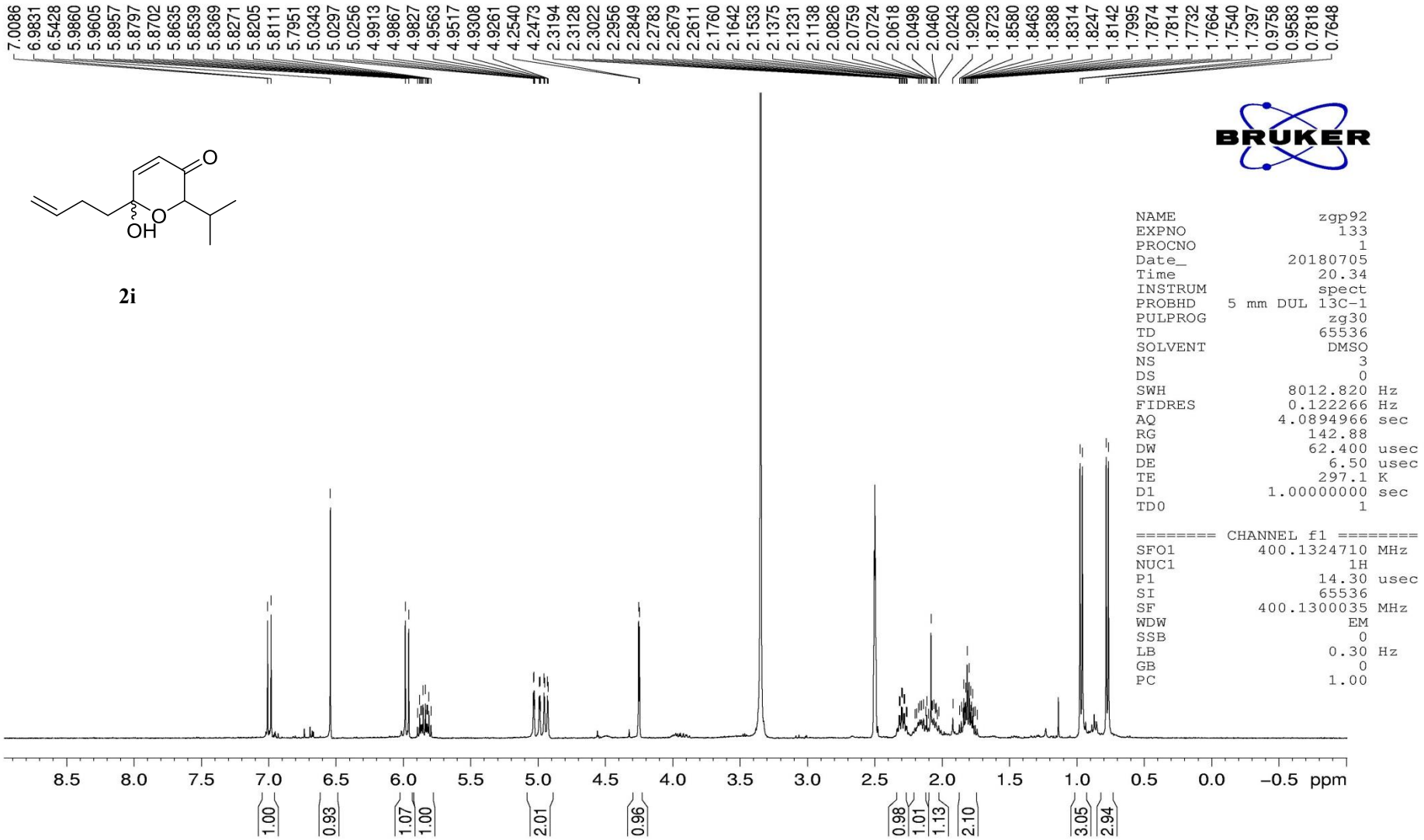


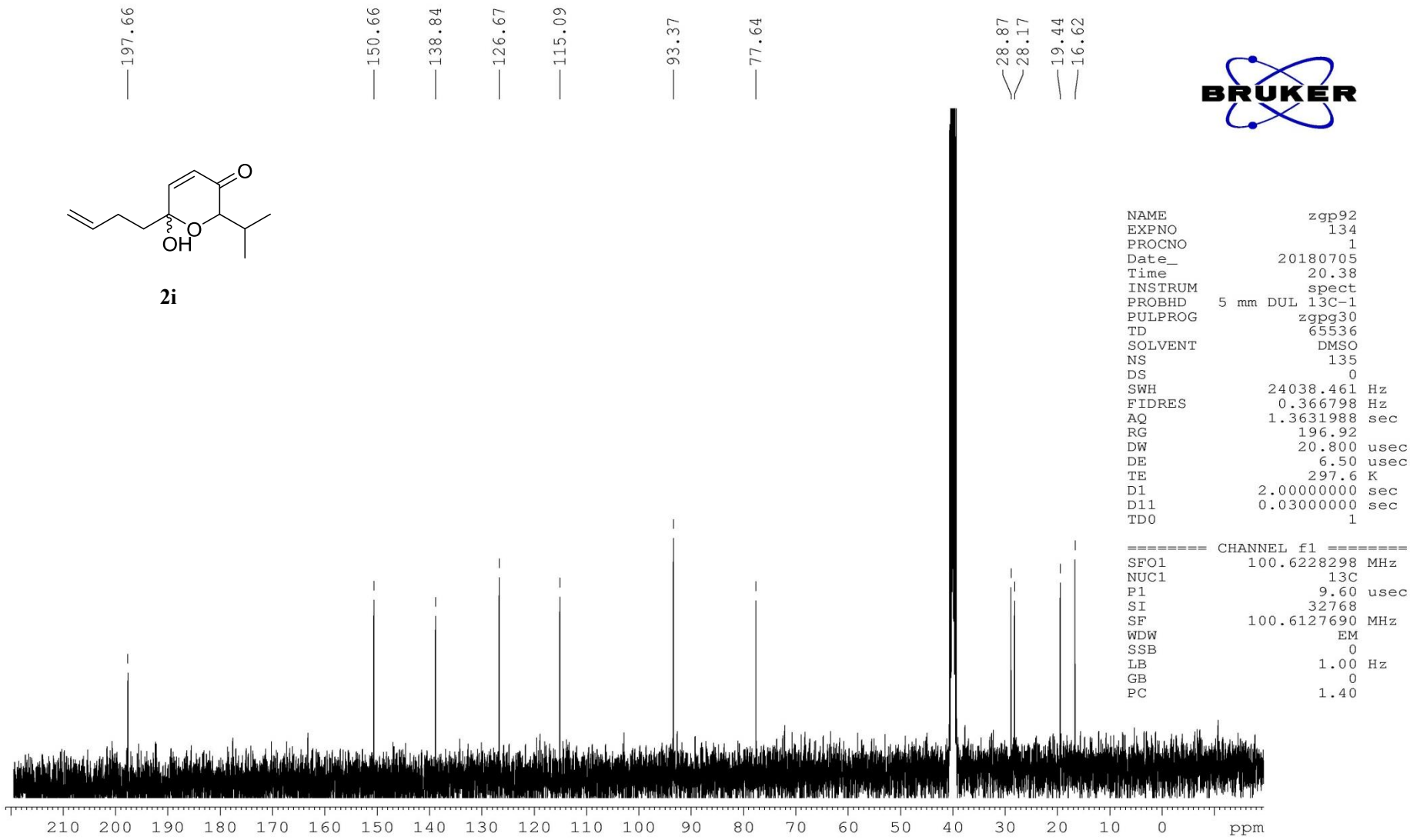

197.13
147.31
131.17
128.07
121.16
92.96
78.42
45.98
28.84
19.20
16.17
O
O
OH
BRUKER
NAME zgp91
EXPNO 124
PROCNO 1
Date_ 20180705
Time 15.55
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 101
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127556 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

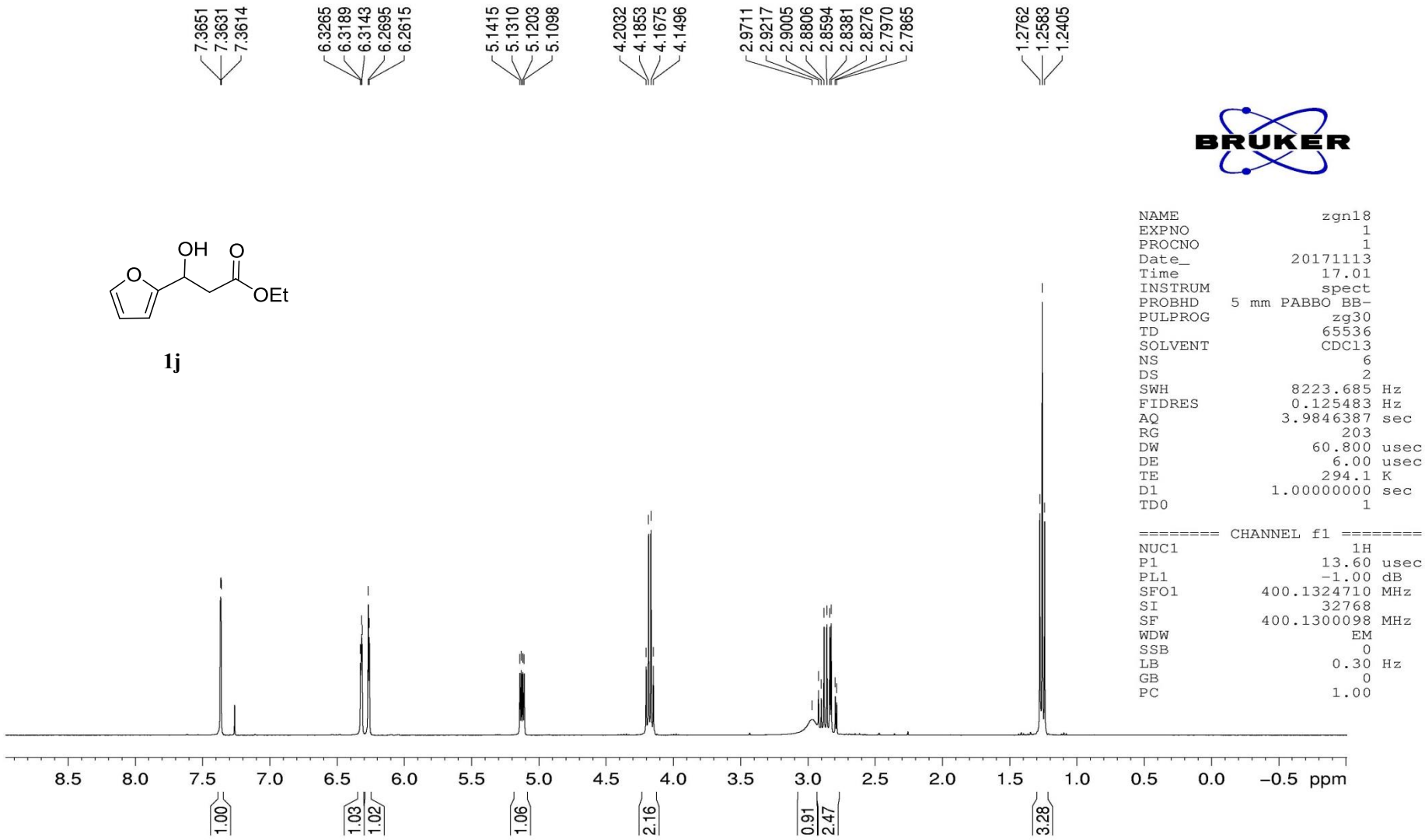
**2h**

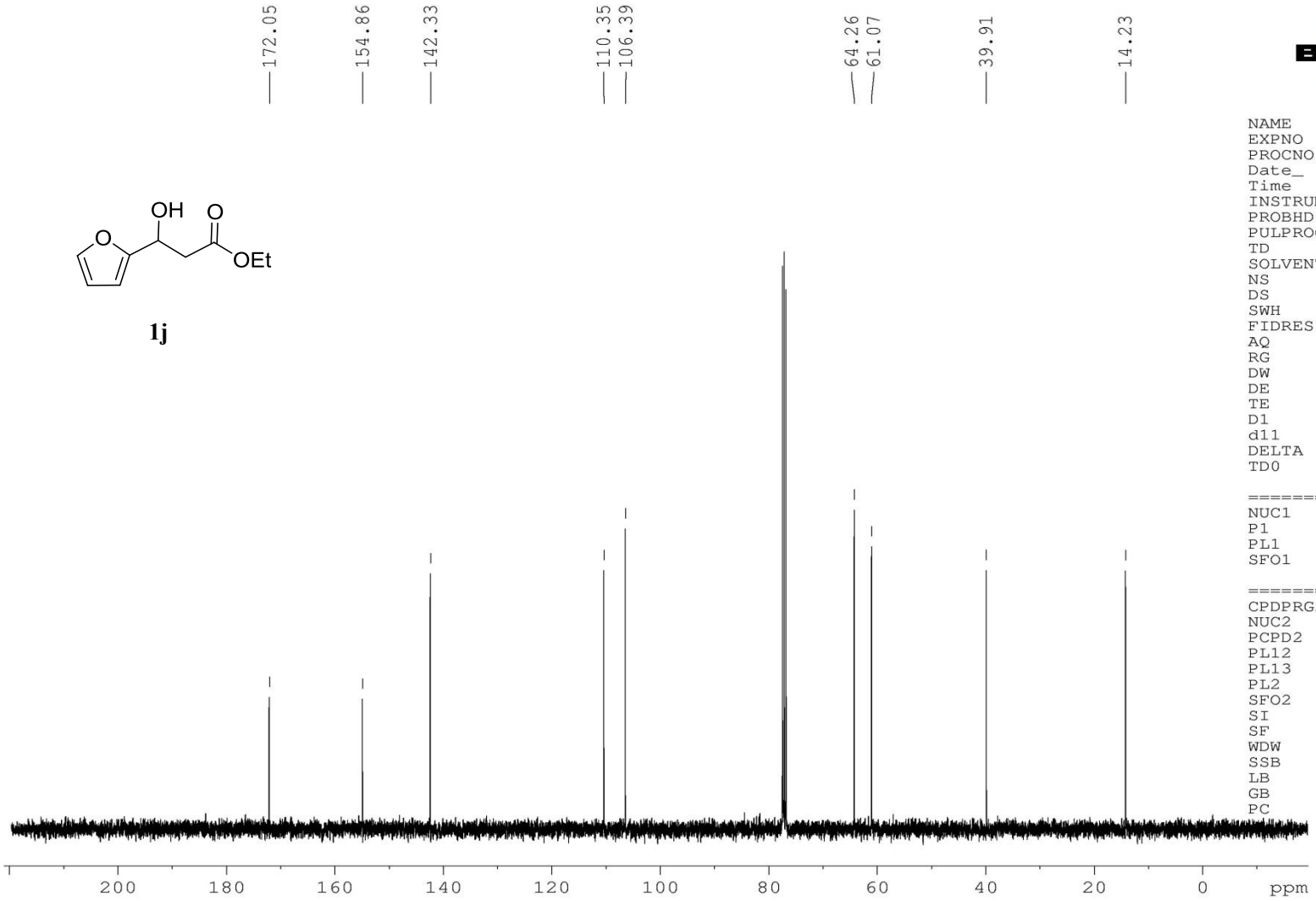

7.2600
6.1042
6.0966
5.9313
5.9237
5.8839
5.8674
5.8584
5.8510
5.8417
5.8249
5.8156
5.8082
5.7991
5.7827
5.0752
5.0712
5.0669
5.0628
5.0325
5.0284
5.0241
5.0200
5.0032
5.0003
4.9960
4.9929
4.9777
4.9748
4.9705
4.3021
4.2846
2.7178
2.6993
2.6800
2.4113
2.3948
2.3917
2.3750
2.3570
2.1291
2.1121
2.0951
2.0779
2.0608
2.0432
1.8189
1.0269
1.0101
0.8563
0.8394
OH
O
1i
NAME zgp92
EXPNO 1
PROCNO 1
Date_ 20180626
Time 15.29
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 7
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 126.97
DW 62.400 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.00 1.02 1.08 2.13 1.00 2.06 2.09 1.11 1.00 3.14 3.20


154.99
154.45
137.61
115.36
107.23
105.53
73.75
33.38
32.23
27.67
18.92
18.48
OH
O
1i
BRUKER
NAME zgp92
EXPNO 2
PROCNO 1
Date_ 20180626
Time 15.33
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 133
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127575 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.0086
6.9831
6.5428
5.9860
5.9605
5.8957
5.8797
5.8702
5.8635
5.8539
5.8369
5.8271
5.8205
5.8111
5.7951
5.0343
5.0297
5.0256
4.9913
4.9867
4.9827
4.9563
4.9517
4.9308
4.9261
4.2540
4.2473
2.3194
2.3128
2.3022
2.2956
2.2849
2.2783
2.2679
2.2611
2.1760
2.1642
2.1533
2.1375
2.1231
2.1138
2.1123
2.0826
2.0759
2.0724
2.0618
2.0498
2.0460
2.0243
1.9208
1.8723
1.8580
1.8463
1.8388
1.8314
1.8247
1.8142
1.7995
1.7874
1.7814
1.7732
1.7664
1.7540
1.7397
0.9758
0.9583
0.7818
0.7648
O
O
OH
2i
BRUKER
NAME zgp92
EXPNO 133
PROCNO 1
Date_ 20180705
Time 20.34
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 3
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 142.88
DW 62.400 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300035 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5 ppm
1.00
0.93
1.07
1.00
2.01
0.96
0.98
1.01
1.13
2.10
3.05
2.94

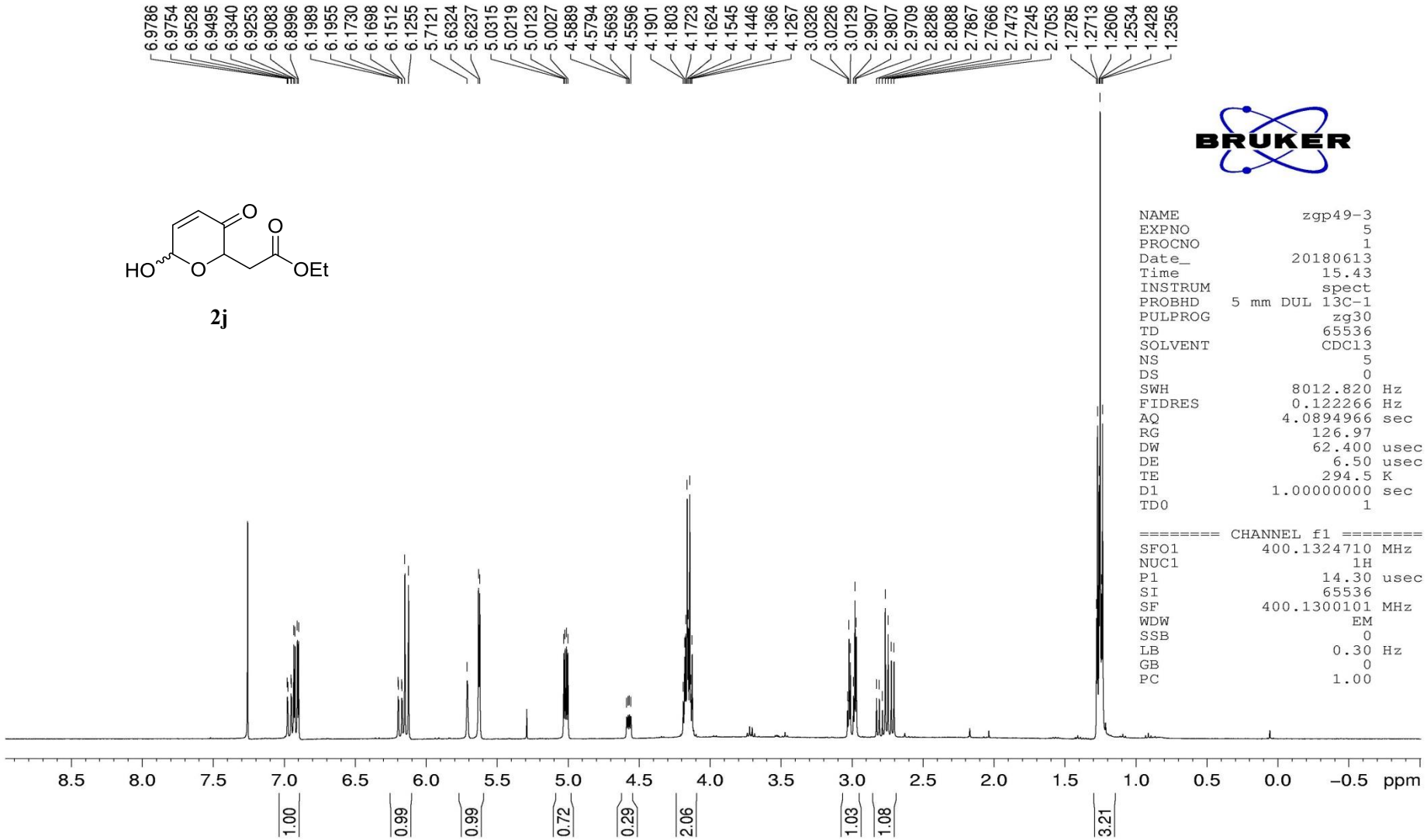

197.66
150.66
138.84
126.67
115.09
93.37
77.64
28.87
28.17
19.44
16.62
O
O
OH
2i
BRUKER
NAME zgp92
EXPNO 134
PROCNO 1
Date_ 20180705
Time 20.38
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 135
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

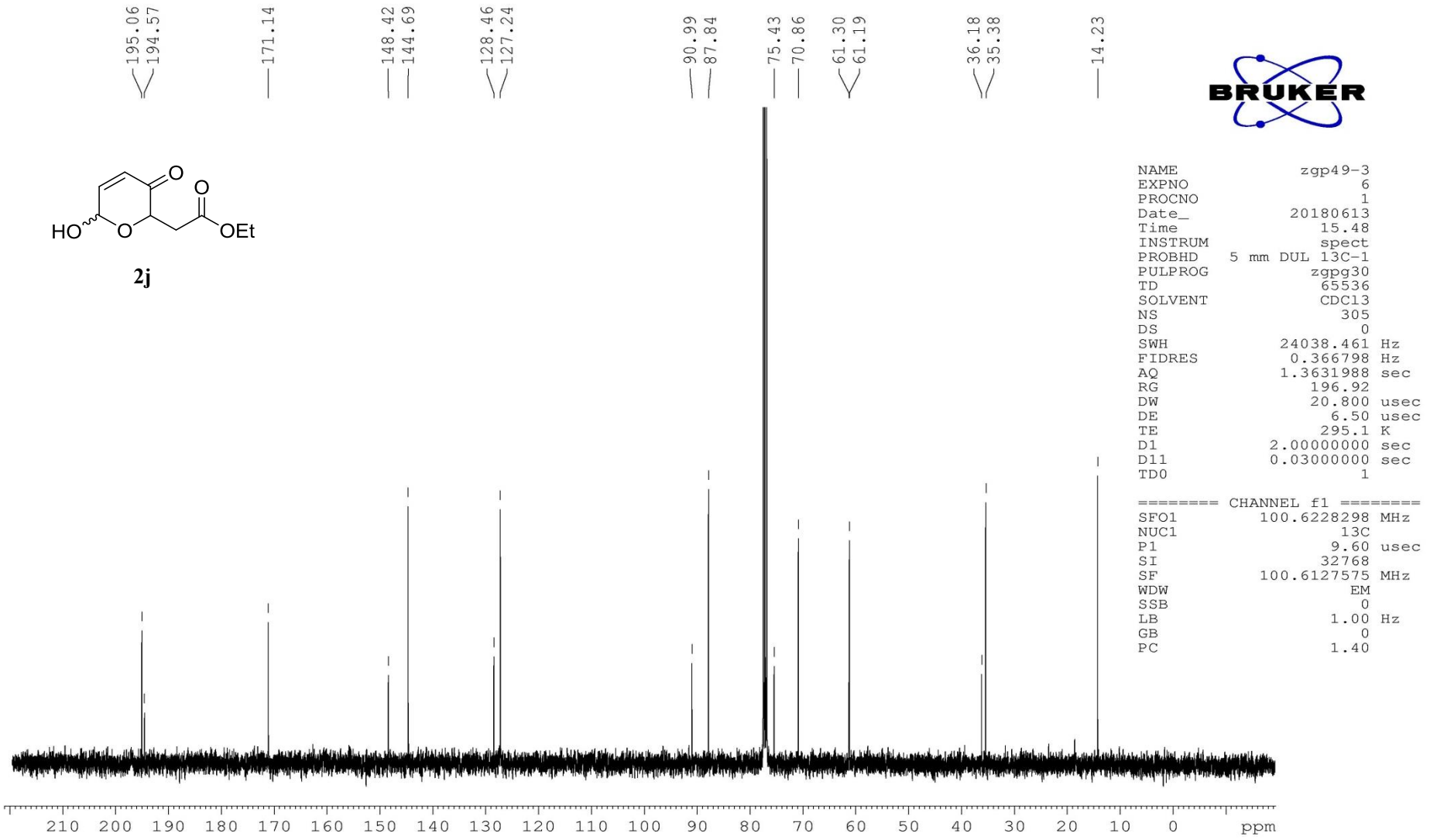

7.3651
7.3631
7.3614
6.3265
6.3189
6.3143
6.2695
6.2615
5.1415
5.1310
5.1203
5.1098
4.2032
4.1853
4.1675
4.1496
2.9711
2.9217
2.9005
2.8806
2.8594
2.8381
2.8276
2.7970
2.7865
1.2762
1.2583
1.2405
OH
O
O
OEt
1j
BRUKER
NAME zgn18
EXPNO 1
PROCNO 1
Date_ 20171113
Time 17.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.00 usec
TE 294.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.00
1.03
1.02
1.06
2.16
0.91
2.47
3.28

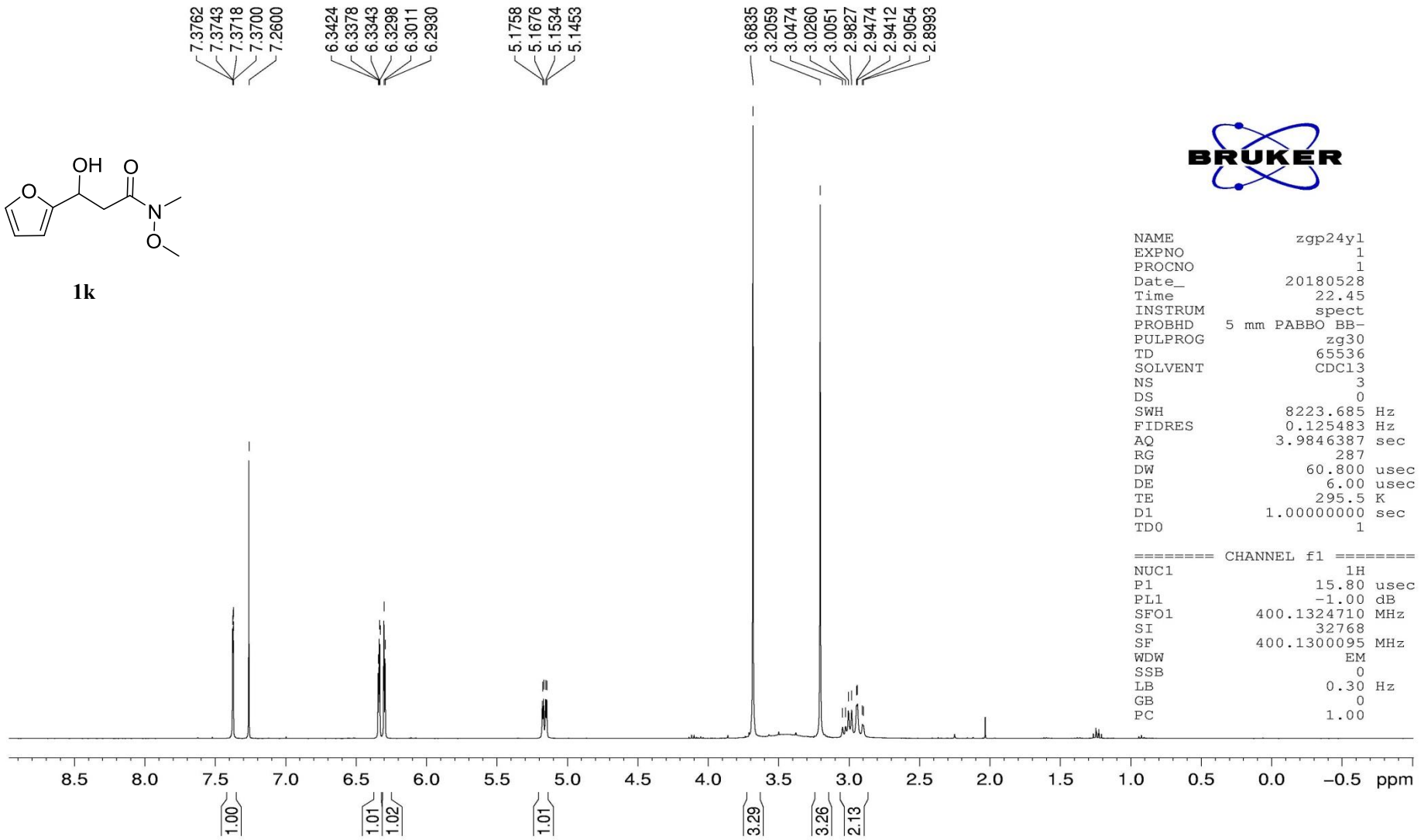


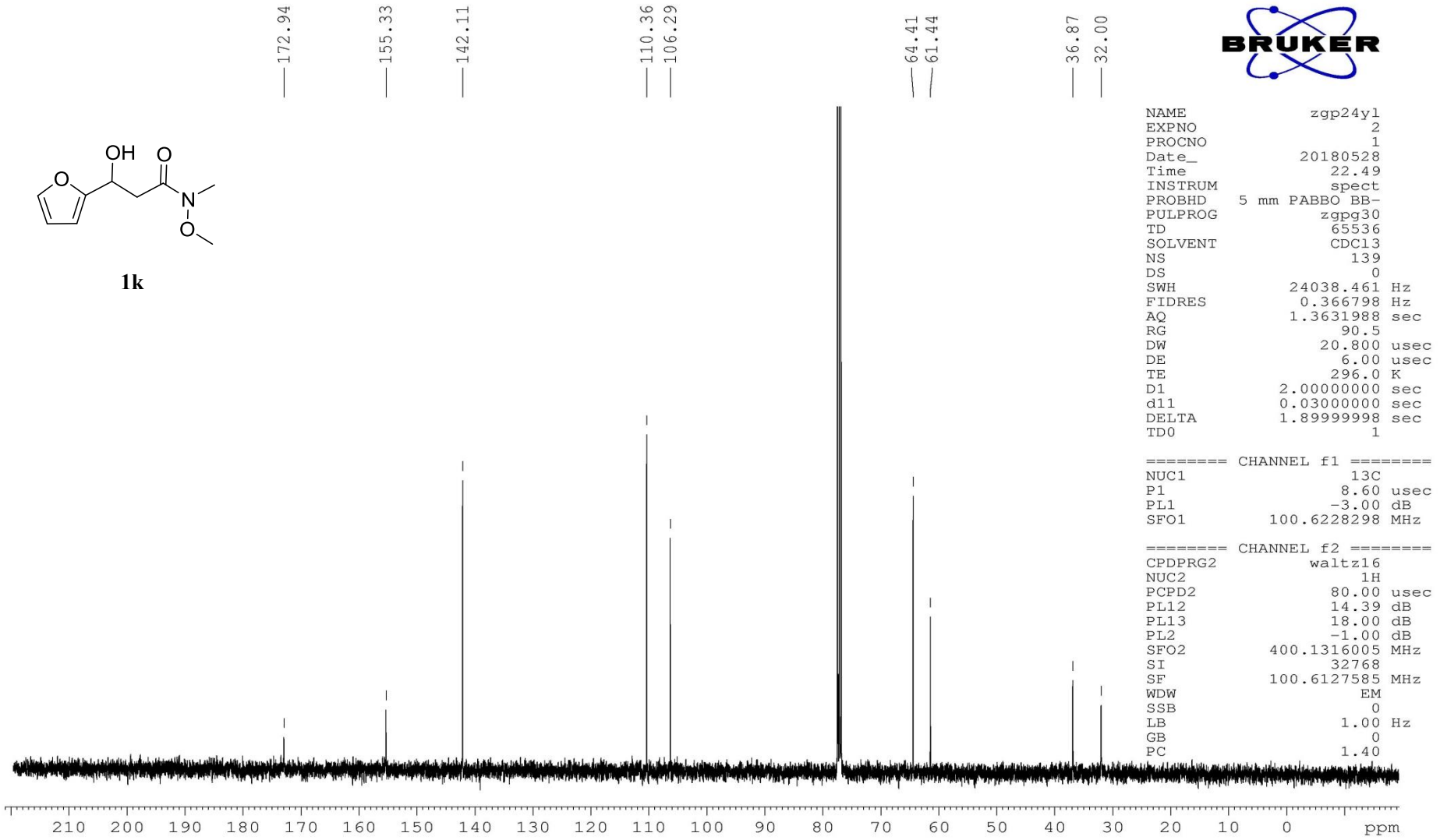

**1j**

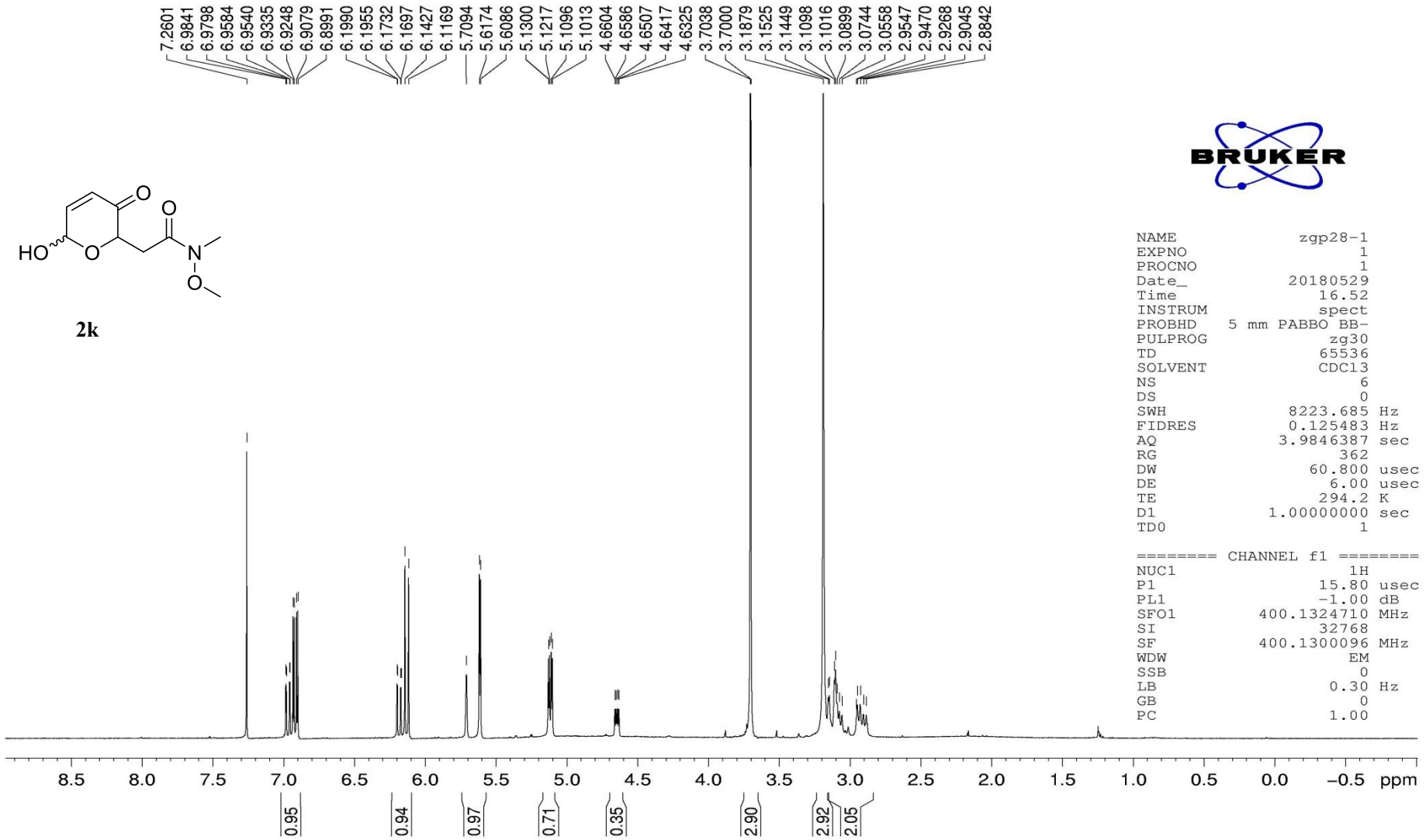
```
NAME                 zgn18
EXPNO                    2
PROCNO                   1
Date_             20171113
Time                 17.06
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      65
DS                       2
SWH              24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                    2050
DW                  20.800 usec
DE                   6.00 usec
TE                  294.8 K
D1               2.00000000 sec
d11              0.03000000 sec
DELTA            1.89999998 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.25 usec
PL1                  -3.00 dB
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2                80.00 usec
PL12                 12.45 dB
PL13                 18.00 dB
PL2                  -1.00 dB
SFO2          400.1316005 MHz
SI                   32768
SF            100.6127589 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

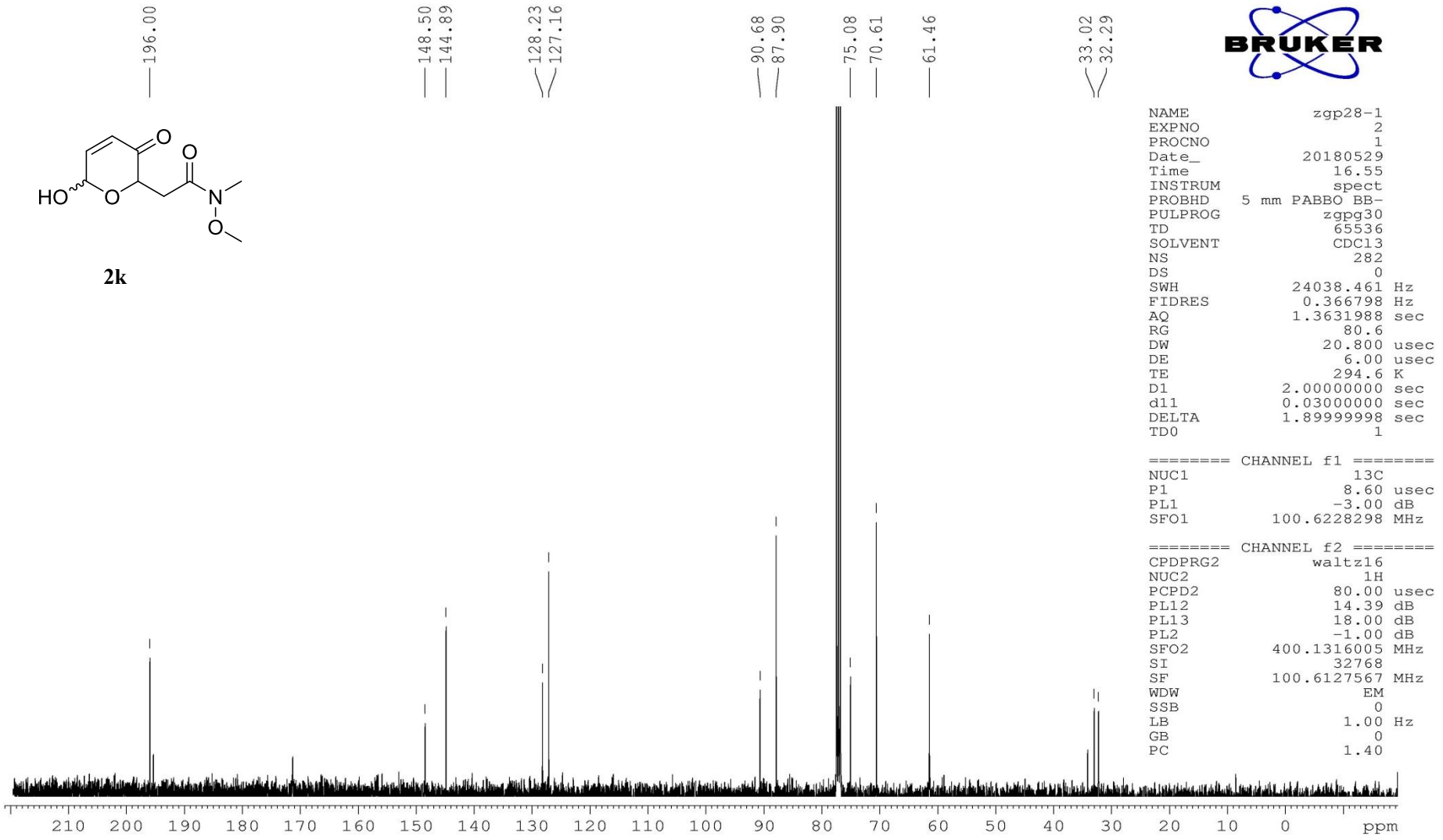

6.9786
6.9754
6.9528
6.9495
6.9340
6.9253
6.9083
6.8996
6.1989
6.1955
6.1730
6.1698
6.1512
6.1255
5.7121
5.6324
5.6237
5.0315
5.0219
5.0123
5.0027
4.5889
4.5794
4.5693
4.5596
4.1901
4.1803
4.1723
4.1624
4.1545
4.1446
4.1366
4.1267
3.0326
3.0226
3.0129
2.9907
2.9807
2.9709
2.8286
2.8088
2.7867
2.7666
2.7473
2.7245
2.7053
1.2785
1.2713
1.2606
1.2534
1.2428
1.2356
BRUKER
HO
O
O
O
OEt
2j
NAME zgp49-3
EXPNO 5
PROCNO 1
Date_ 20180613
Time 15.43
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 5
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 126.97
DW 62.400 usec
DE 6.50 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
ppm
1.00
0.99
0.99
0.72
0.29
2.06
1.03
1.08
3.21

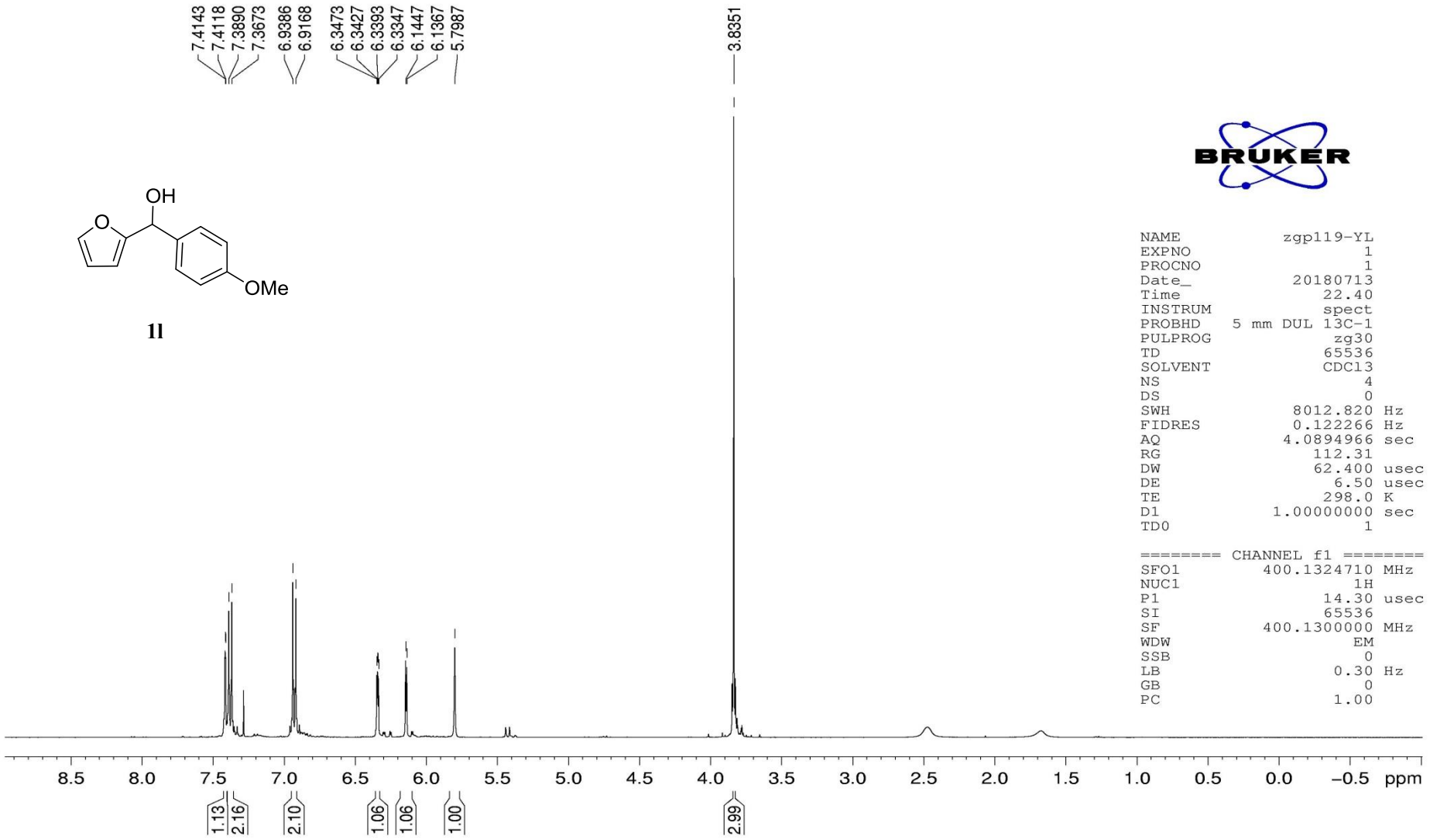

195.06
194.57
171.14
148.42
144.69
128.46
127.24
90.99
87.84
75.43
70.86
61.30
61.19
36.18
35.38
14.23
HO
O
O
O
OEt
2j
BRUKER
NAME zgp49-3
EXPNO 6
PROCNO 1
Date_ 20180613
Time 15.48
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 305
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127575 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

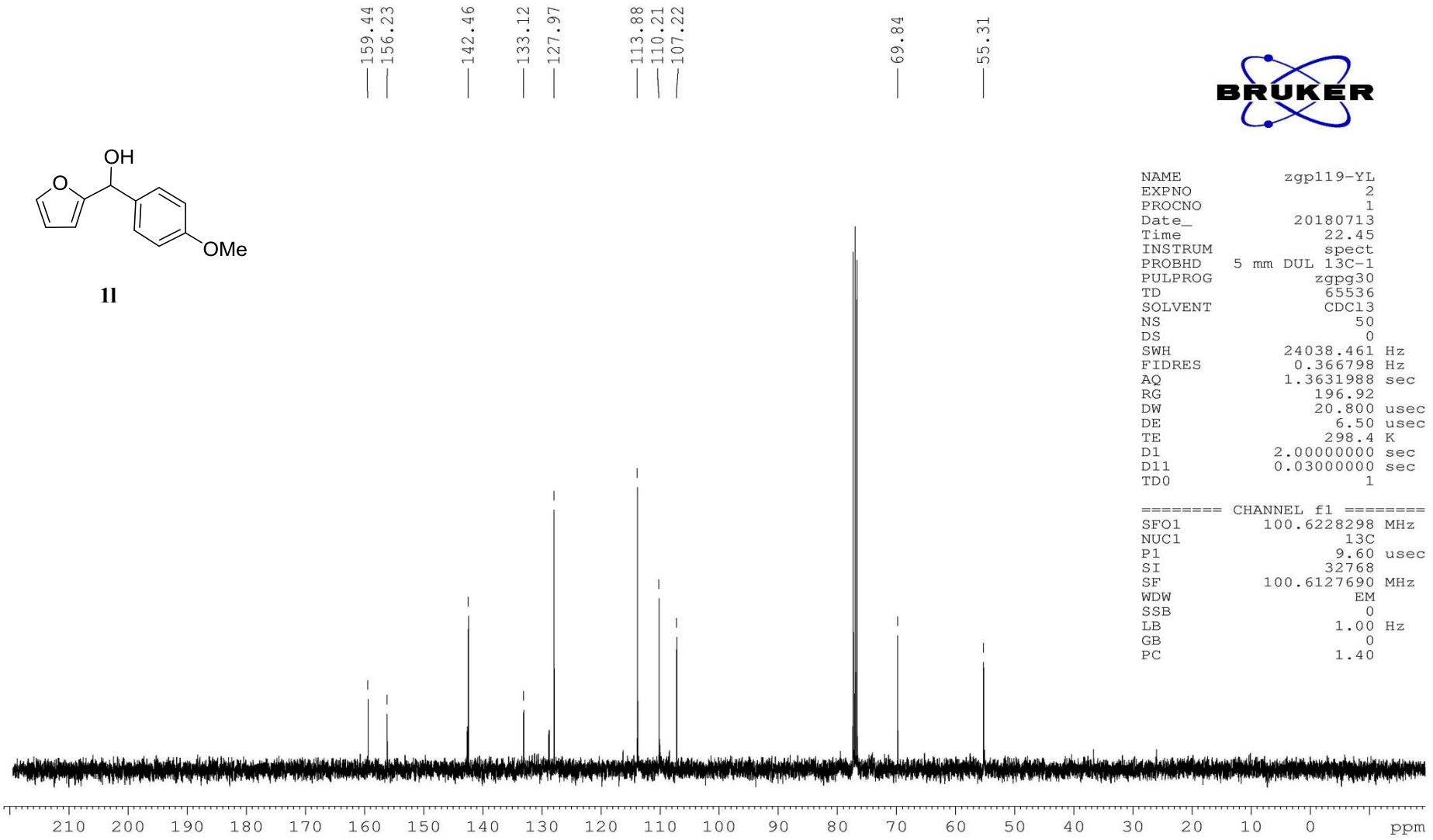

7.3762
7.3743
7.3718
7.3700
7.2600
6.3424
6.3378
6.3343
6.3298
6.3011
6.2930
5.1758
5.1676
5.1534
5.1453
3.6835
3.2059
3.0474
3.0260
3.0051
2.9827
2.9474
2.9412
2.9054
2.8993
OH
O
O
N
O
1k
BRUKER
NAME zgp24yl
EXPNO 1
PROCNO 1
Date_ 20180528
Time 22.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 287
DW 60.800 usec
DE 6.00 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
ppm
1.00
1.01
1.02
1.01
3.29
3.26
2.13

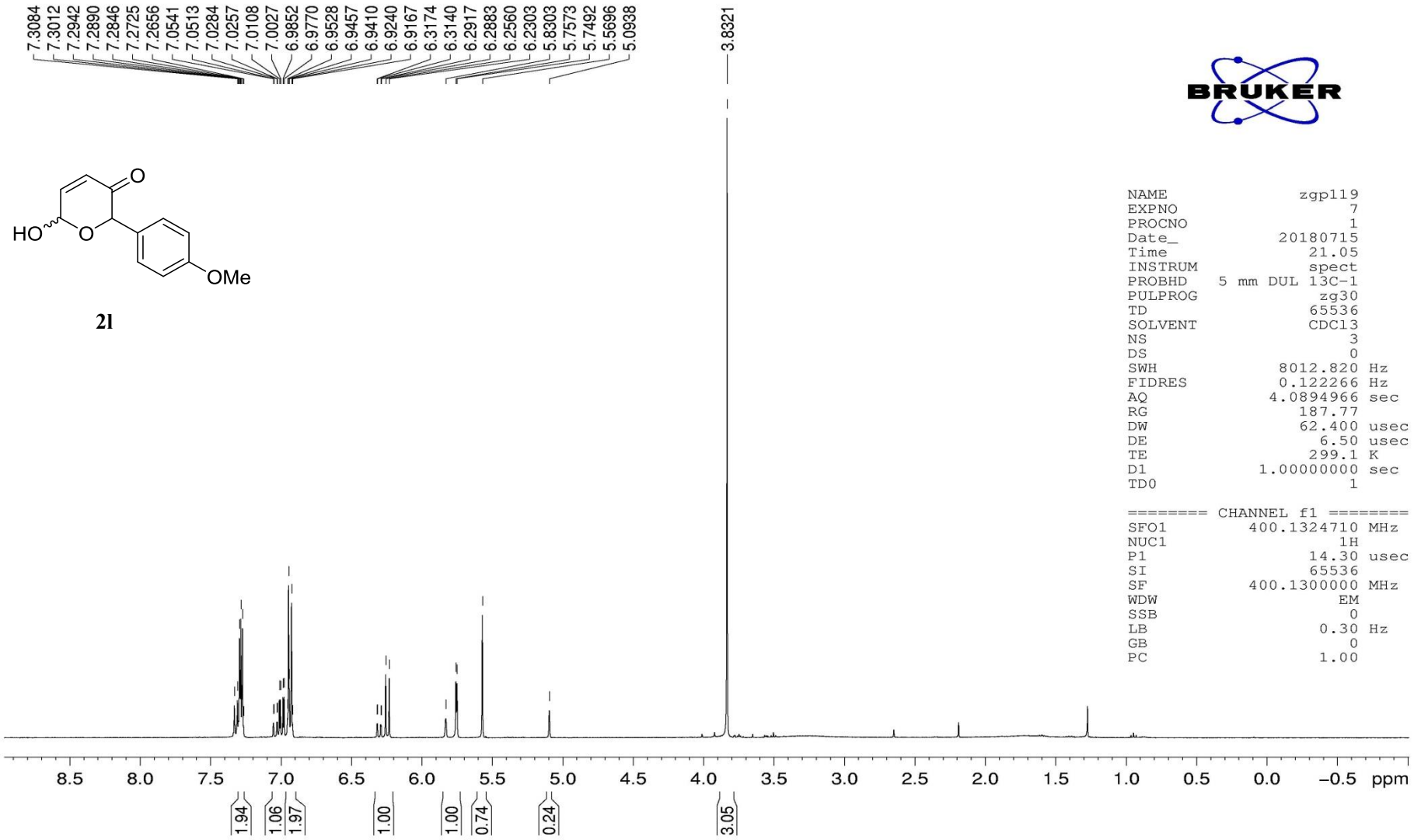

172.94
155.33
142.11
110.36
106.29
64.41
61.44
36.87
32.00
OH
O
O
N
O
1k
NAME zgp24yl
EXPNO 2
PROCNO 1
Date_ 20180528
Time 22.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 139
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 90.5
DW 20.800 usec
DE 6.00 usec
TE 296.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 14.39 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127585 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.2601
6.9841
6.9798
6.9584
6.9540
6.9335
6.9248
6.9079
6.8991
6.1990
6.1955
6.1732
6.1697
6.1427
6.1169
5.7094
5.6174
5.6086
5.1300
5.1217
5.1096
5.1013
4.6604
4.6586
4.6507
4.6417
4.6325
3.7038
3.7000
3.1879
3.1525
3.1449
3.1098
3.1016
3.0899
3.0744
3.0558
2.9547
2.9470
2.9268
2.9045
2.8842
HO
O
O
O
N
O
2k
BRUKER
NAME zgp28-1
EXPNO 1
PROCNO 1
Date_ 20180529
Time 16.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 362
DW 60.800 usec
DE 6.00 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
0.95
0.94
0.97
0.71
0.35
2.90
2.92
2.05

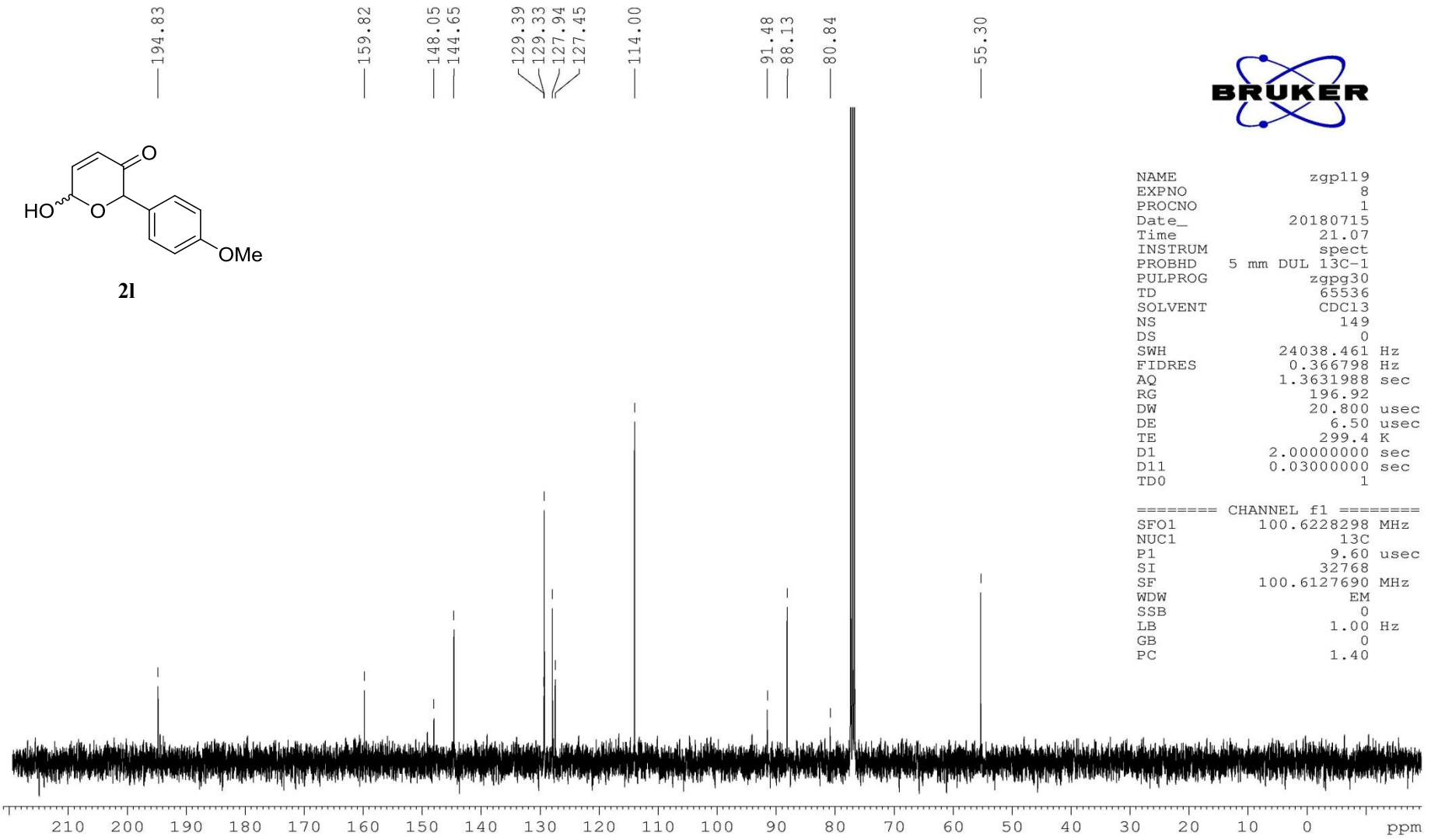

196.00
148.50
144.89
128.23
127.16
90.68
87.90
75.08
70.61
61.46
33.02
32.29
HO
O
O
O
N
O
2k
BRUKER
NAME zgp28-1
EXPNO 2
PROCNO 1
Date_ 20180529
Time 16.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 282
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 80.6
DW 20.800 usec
DE 6.00 usec
TE 294.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 14.39 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127567 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

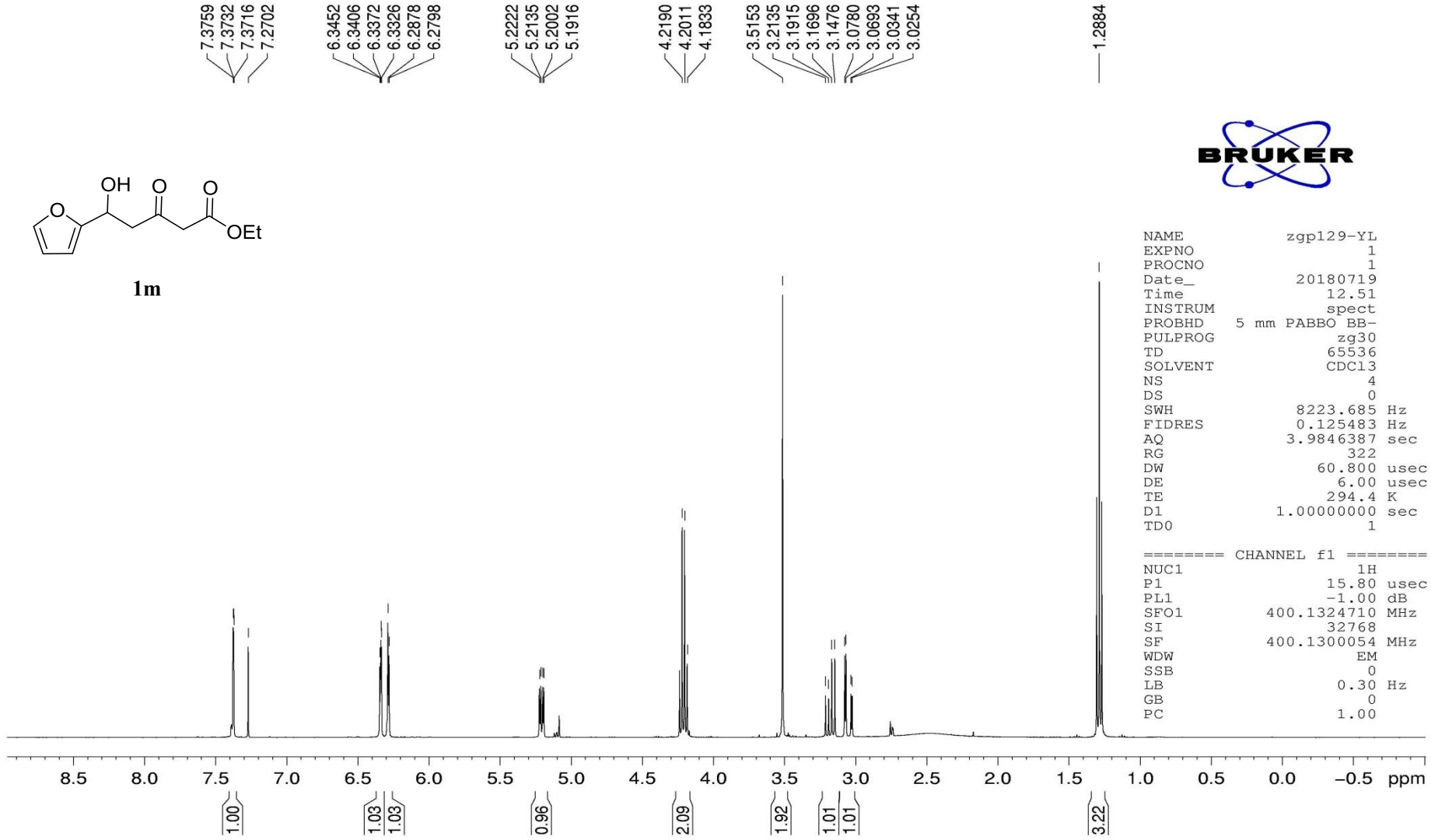

7.4143
7.4118
7.3890
7.3673
6.9386
6.9168
6.3473
6.3427
6.3393
6.3347
6.1447
6.1367
5.7987
3.8351
OH
O
OMe
1l
BRUKER
NAME zgp119-YL
EXPNO 1
PROCNO 1
Date_ 20180713
Time 22.40
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 112.31
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.13
2.16
2.10
1.06
1.06
1.00
2.99

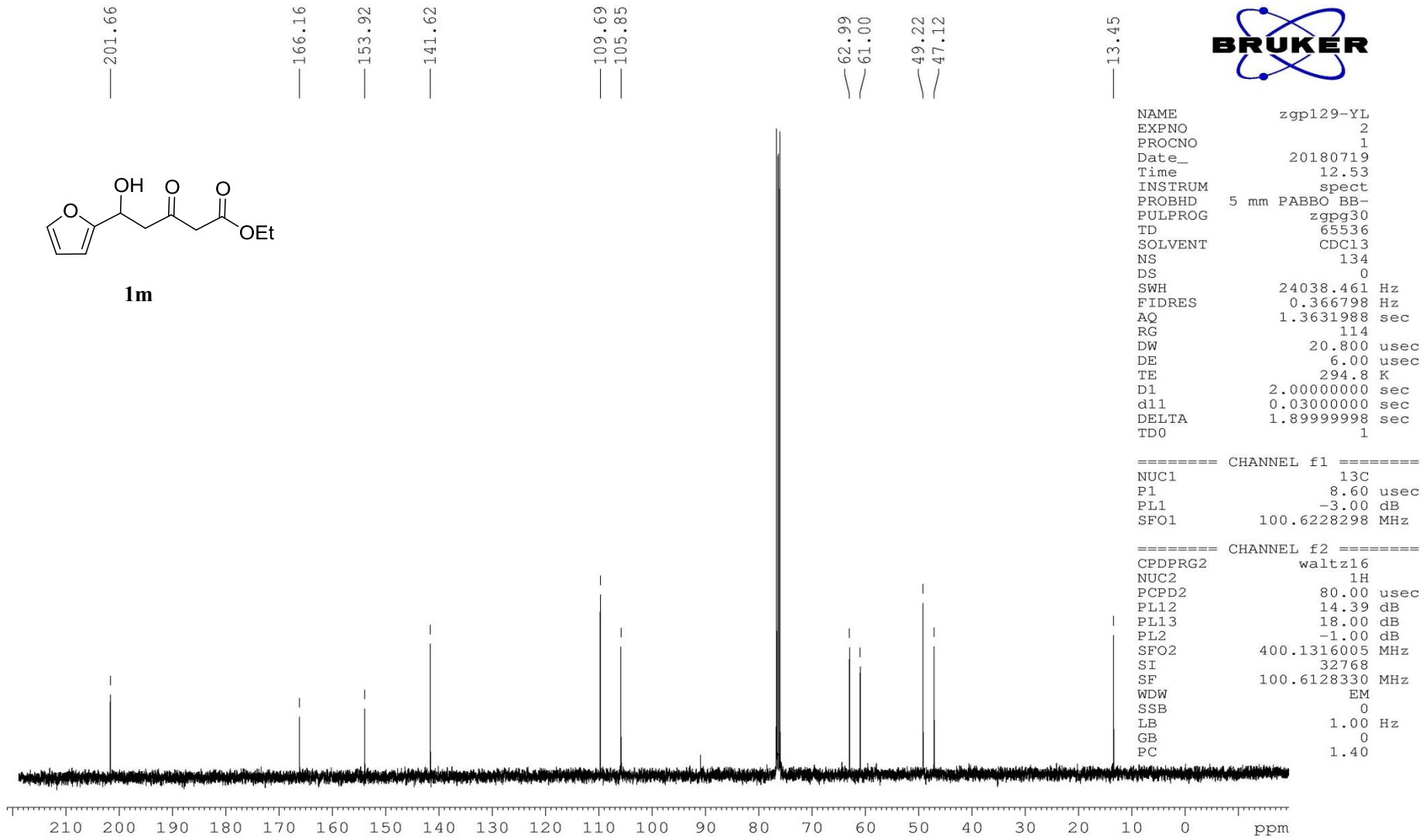

159.44
156.23
142.46
133.12
127.97
113.88
110.21
107.22
69.84
55.31
OH
O
OMe
1l
NAME zgp119-YL
EXPNO 2
PROCNO 1
Date_ 20180713
Time 22.45
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 50
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

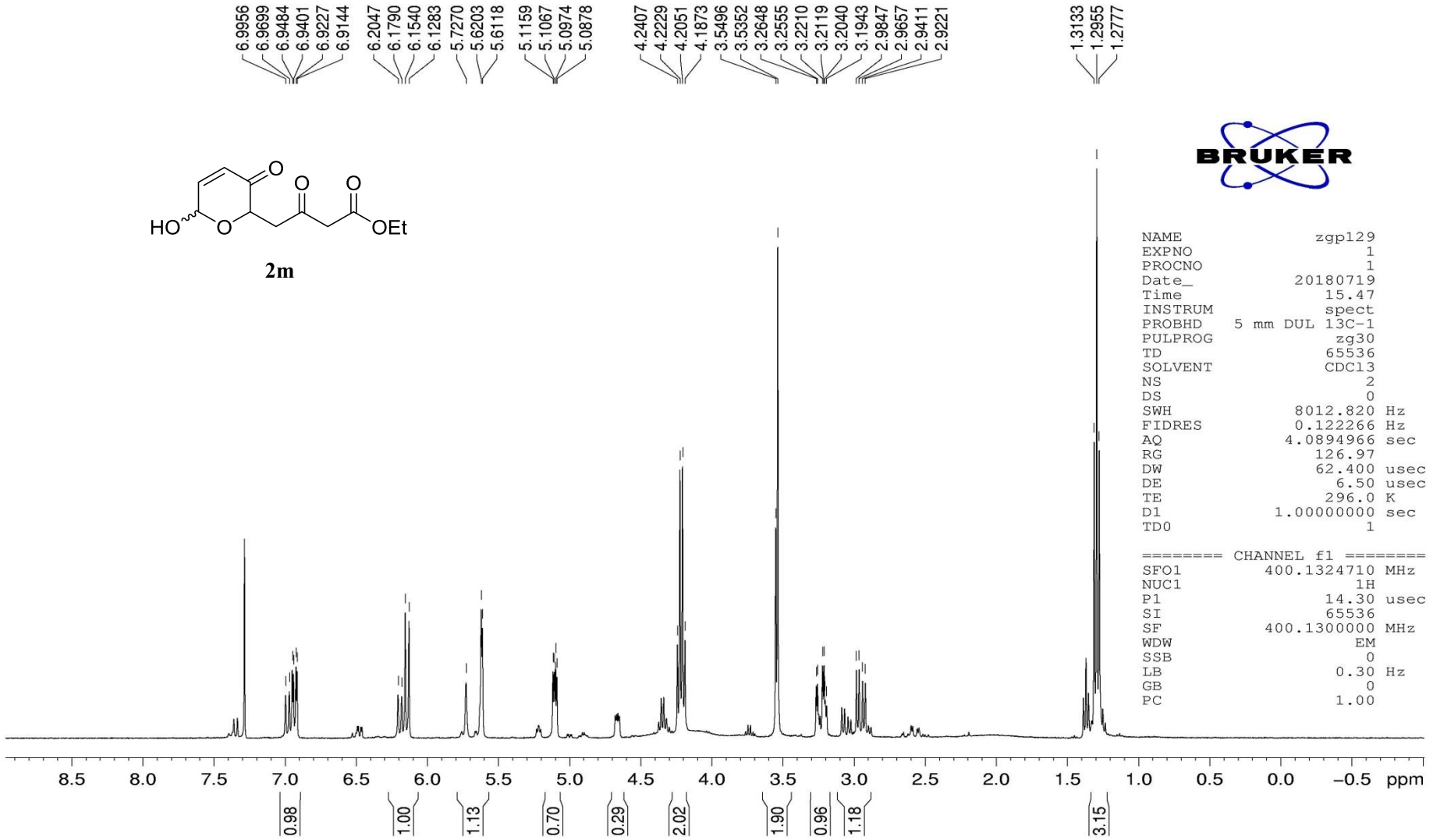

7.3084
7.3012
7.2942
7.2890
7.2846
7.2725
7.2656
7.0541
7.0513
7.0284
7.0257
7.0108
7.0027
6.9852
6.9770
6.9528
6.9457
6.9410
6.9240
6.9167
6.3174
6.3140
6.2917
6.2883
6.2560
6.2303
5.8303
5.7573
5.7492
5.5696
5.0938
3.8321
HO
O
O
OMe
NAME zgp119
EXPNO 7
PROCNO 1
Date_ 20180715
Time 21.05
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 187.77
DW 62.400 usec
DE 6.50 usec
TE 299.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.94
1.06
1.97
1.00
1.00
0.74
0.24
3.05

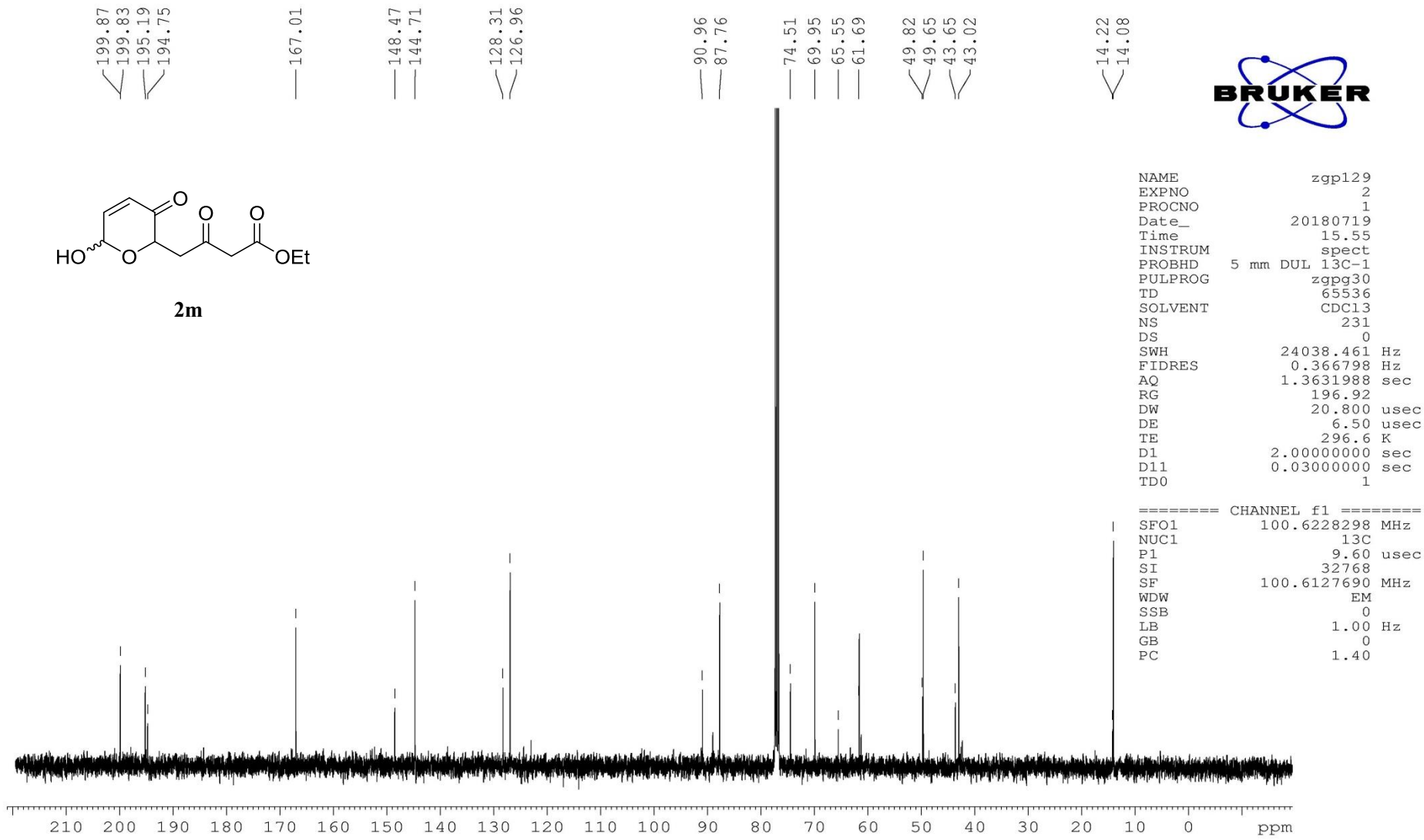
**2l**

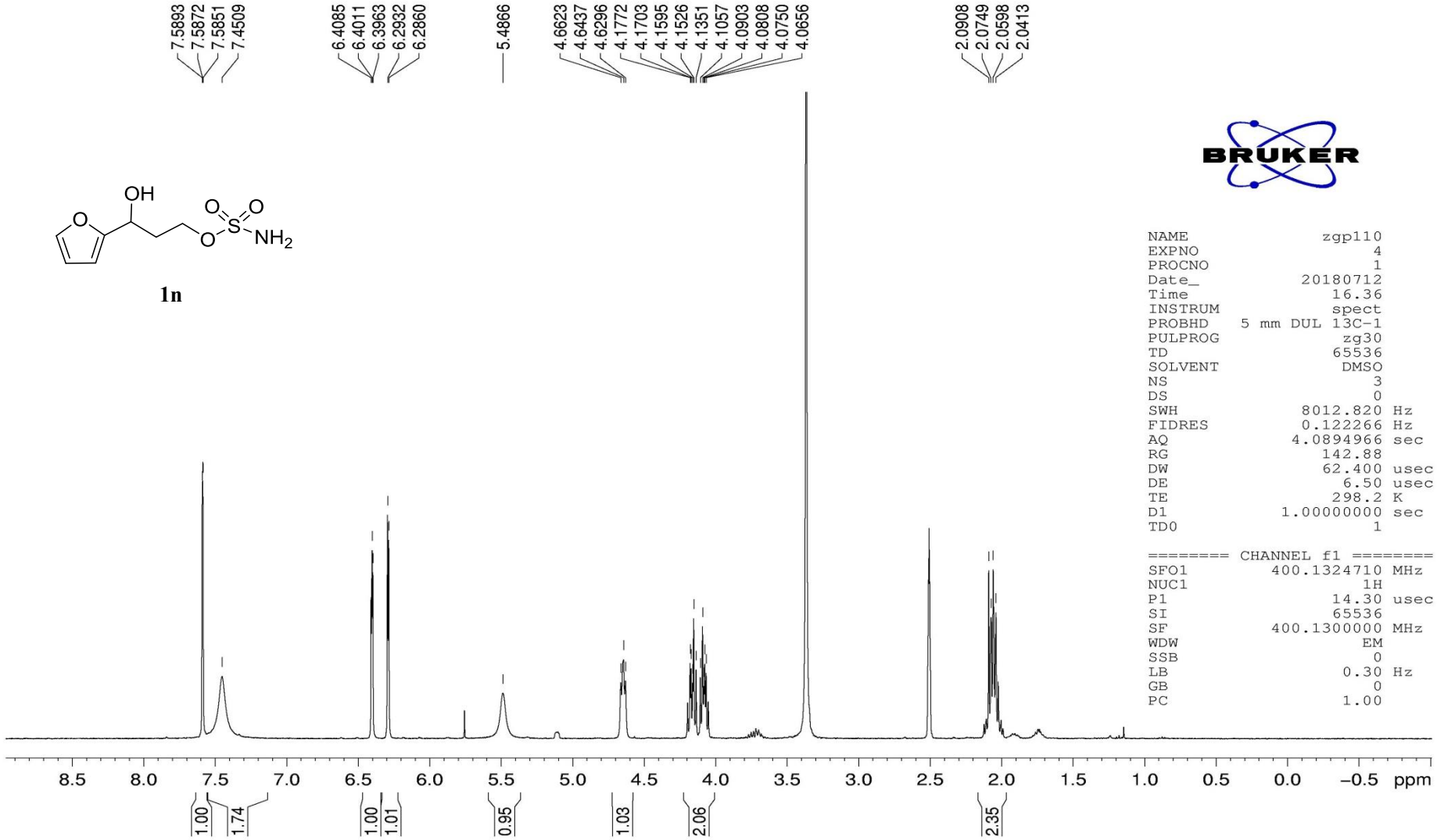

194.83
159.82
148.05
144.65
129.39
129.33
127.94
127.45
114.00
91.48
88.13
80.84
55.30
HO
O
O
OMe
2l
NAME zgp119
EXPNO 8
PROCNO 1
Date_ 20180715
Time 21.07
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 149
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 299.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

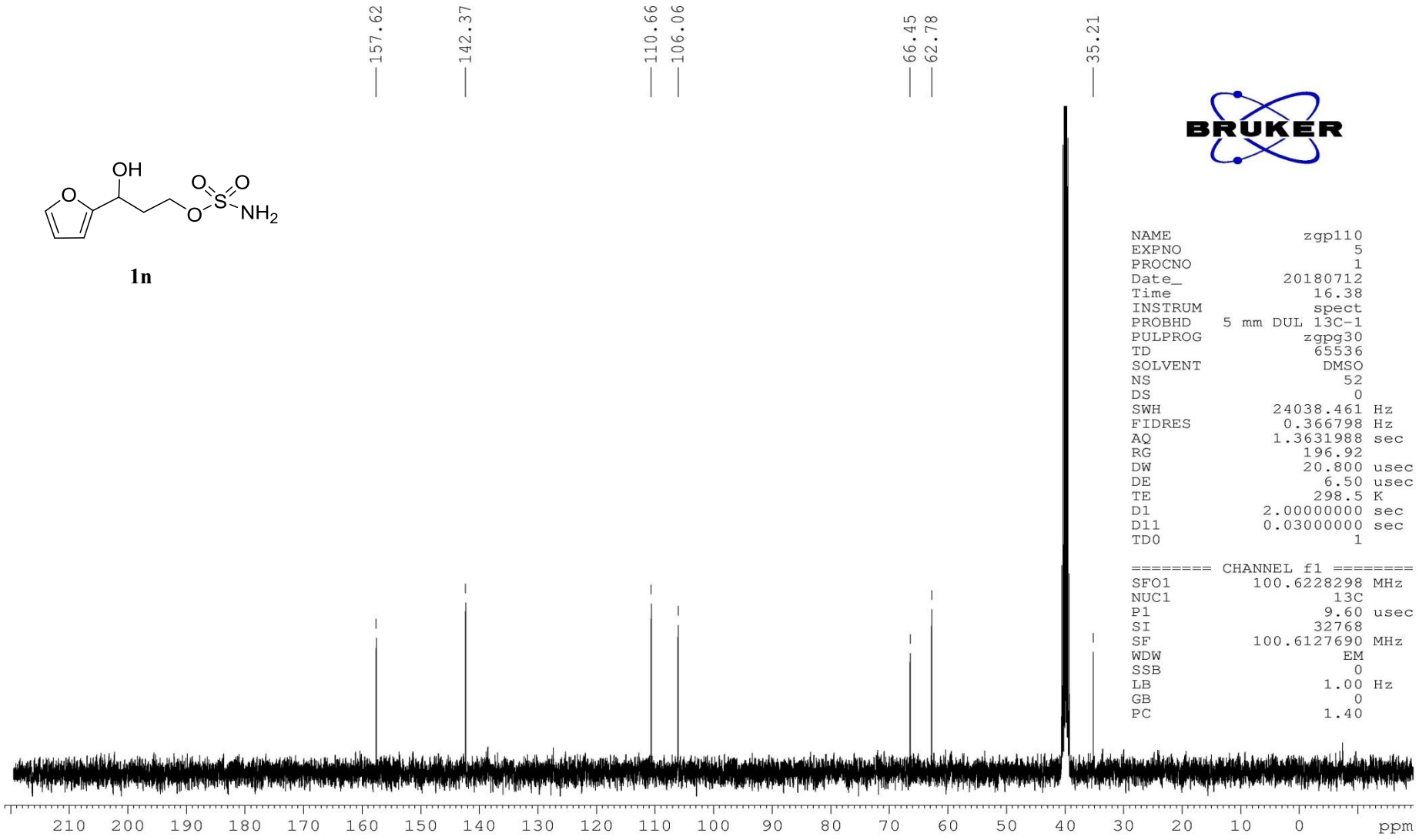

7.3759
7.3732
7.3716
7.2702
6.3452
6.3406
6.3372
6.3326
6.2878
6.2798
5.2222
5.2135
5.2002
5.1916
4.2190
4.2011
4.1833
3.5153
3.2135
3.1915
3.1696
3.1476
3.0780
3.0693
3.0341
3.0254
1.2884
OH
O
O
O
OEt
1m
BRUKER
NAME zgp129-YL
EXPNO 1
PROCNO 1
Date_ 20180719
Time 12.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 4
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 322
DW 60.800 usec
DE 6.00 usec
TE 294.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.00
1.03
1.03
0.96
2.09
1.92
1.01
1.01
3.22

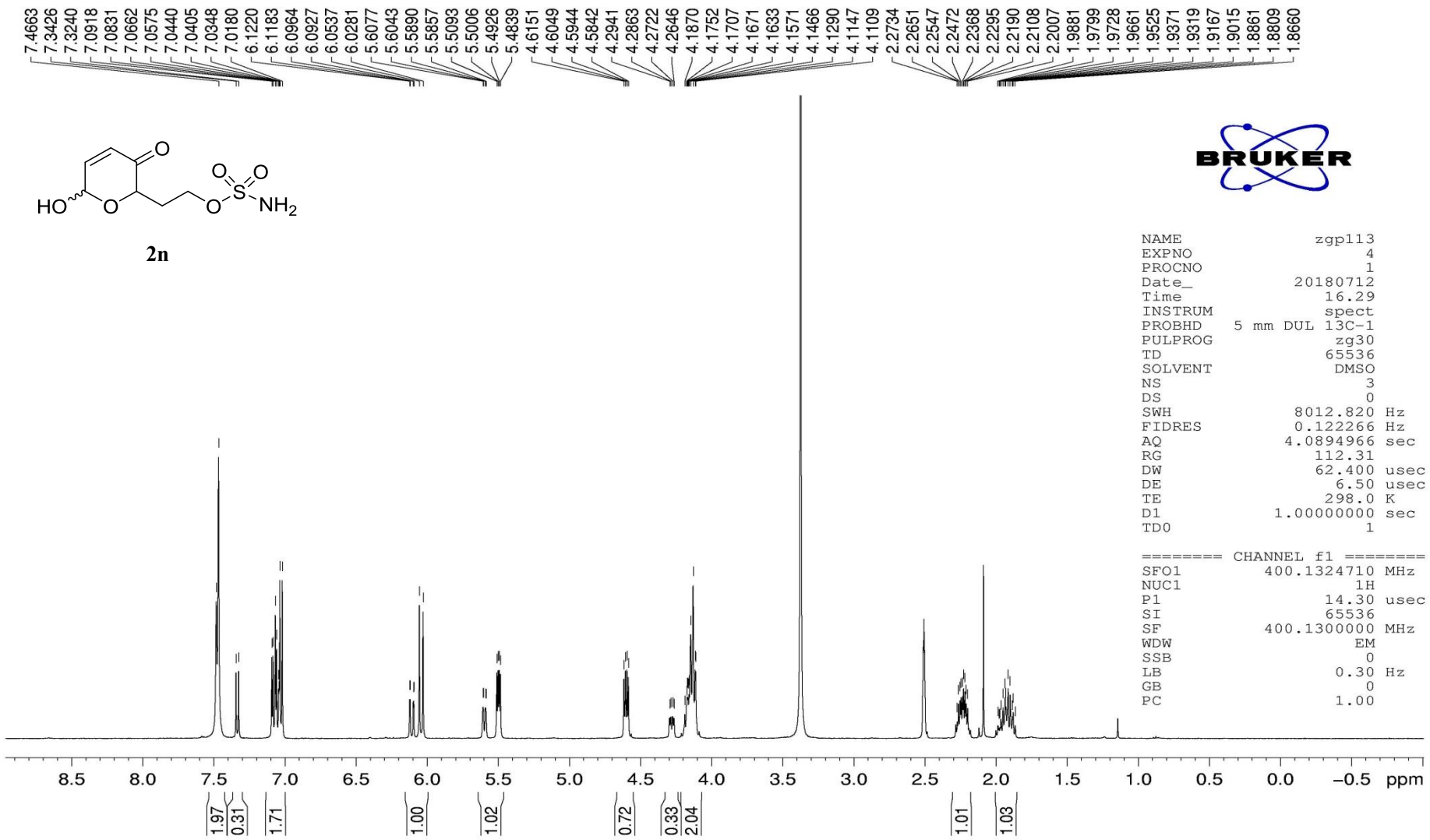

201.66
166.16
153.92
141.62
109.69
105.85
62.99
61.00
49.22
47.12
13.45
OH
O
O
O
OEt
1m
BRUKER
NAME zgp129-YL
EXPNO 2
PROCNO 1
Date_ 20180719
Time 12.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 134
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 114
DW 20.800 usec
DE 6.00 usec
TE 294.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 14.39 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6128330 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

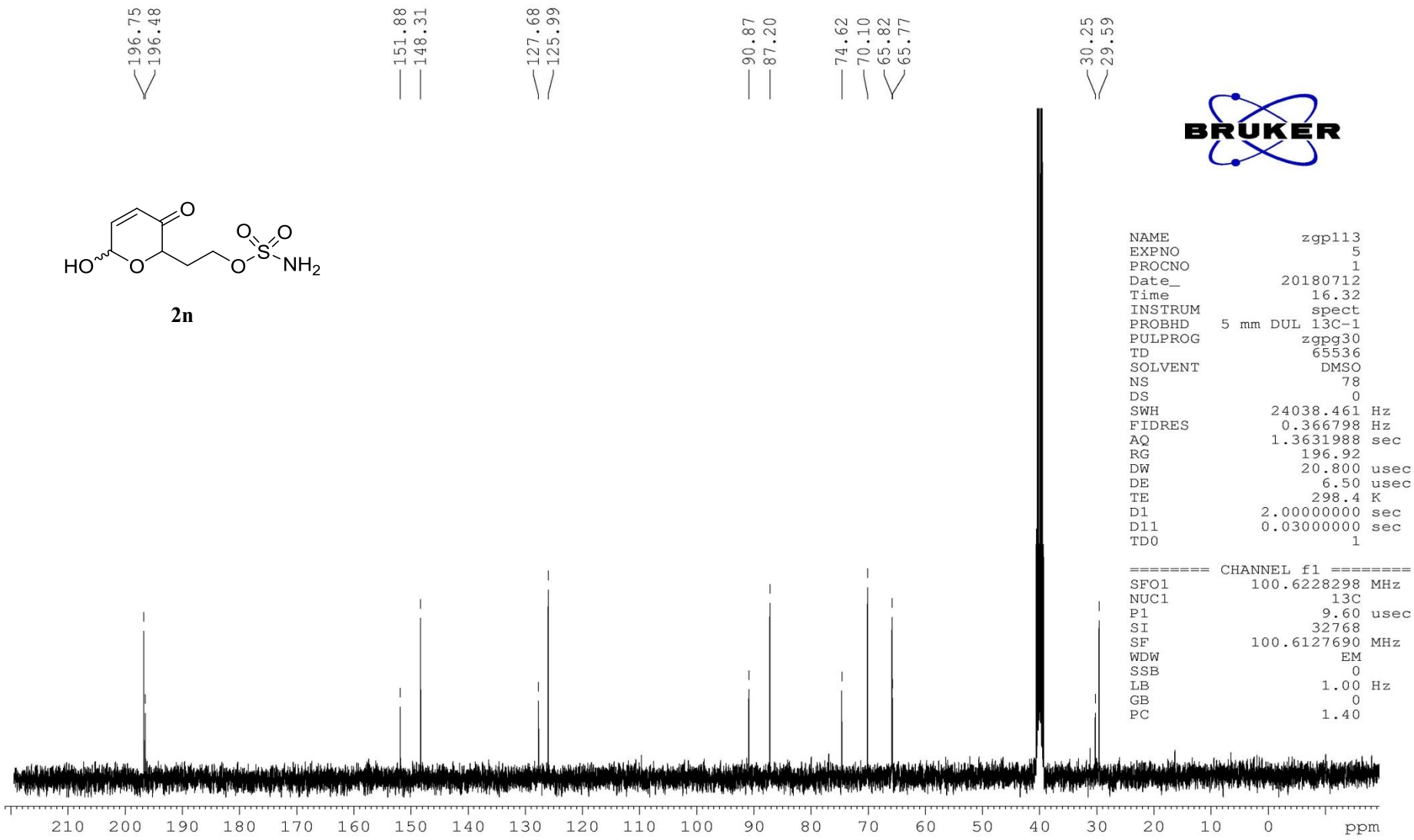

6.9956
6.9699
6.9484
6.9401
6.9227
6.9144
6.2047
6.1790
6.1540
6.1283
5.7270
5.6203
5.6118
5.1159
5.1067
5.0974
5.0878
4.2407
4.2229
4.2051
4.1873
3.5496
3.5352
3.2648
3.2555
3.2210
3.2119
3.2040
3.1943
2.9847
2.9657
2.9411
2.9221
1.3133
1.2955
1.2777
HO
O
O
O
OEt
2m
BRUKER
NAME zgp129
EXPNO 1
PROCNO 1
Date_ 20180719
Time 15.47
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 2
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 126.97
DW 62.400 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
ppm
0.98
1.00
1.13
0.70
0.29
2.02
1.90
0.96
1.18
3.15

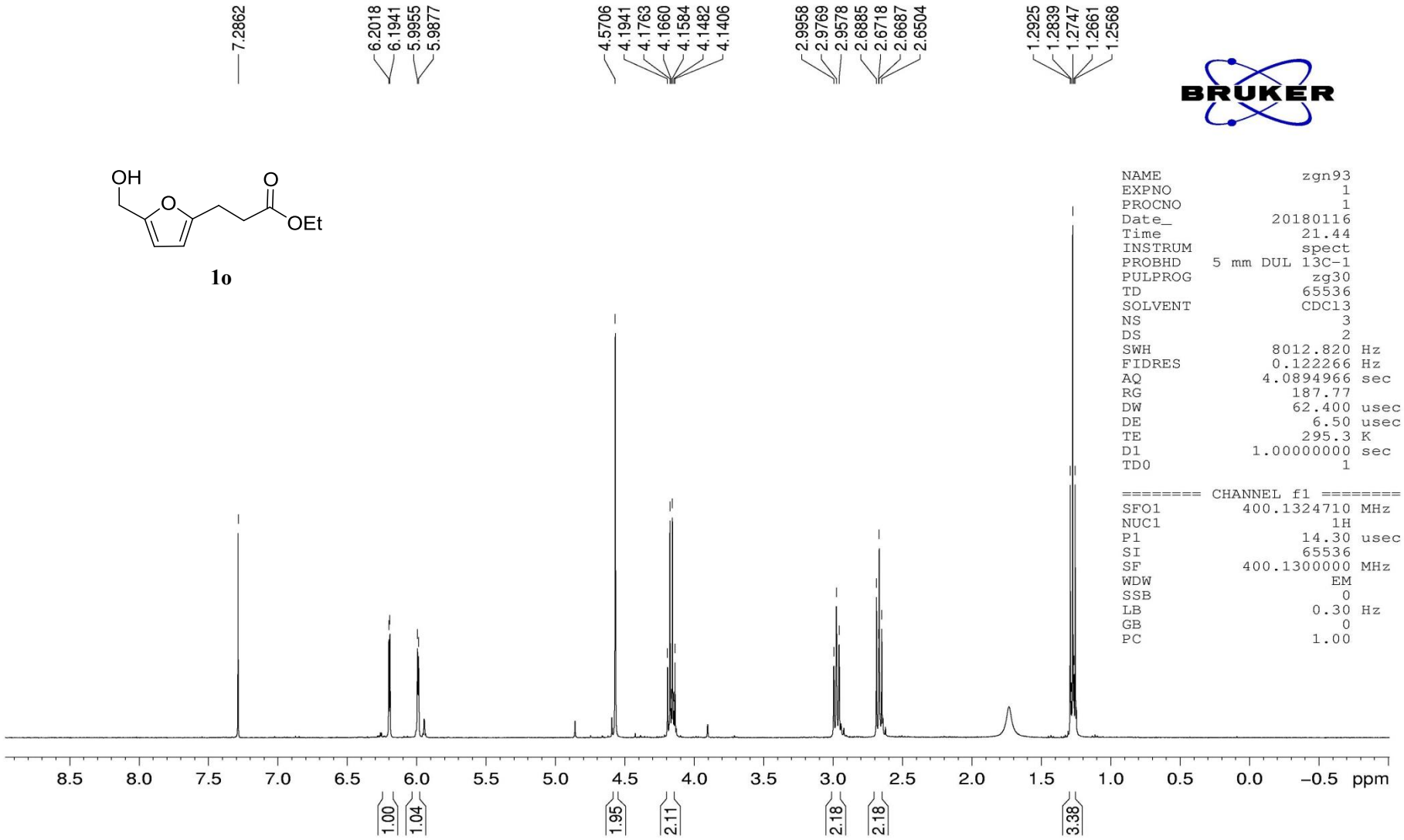

199.87
199.83
195.19
194.75
167.01
148.47
144.71
128.31
126.96
90.96
87.76
74.51
69.95
65.55
61.69
49.82
49.65
43.65
43.02
14.22
14.08
BRUKER
HO
O
O
O
O
OEt
2m
NAME zgp129
EXPNO 2
PROCNO 1
Date_ 20180719
Time 15.55
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 231
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

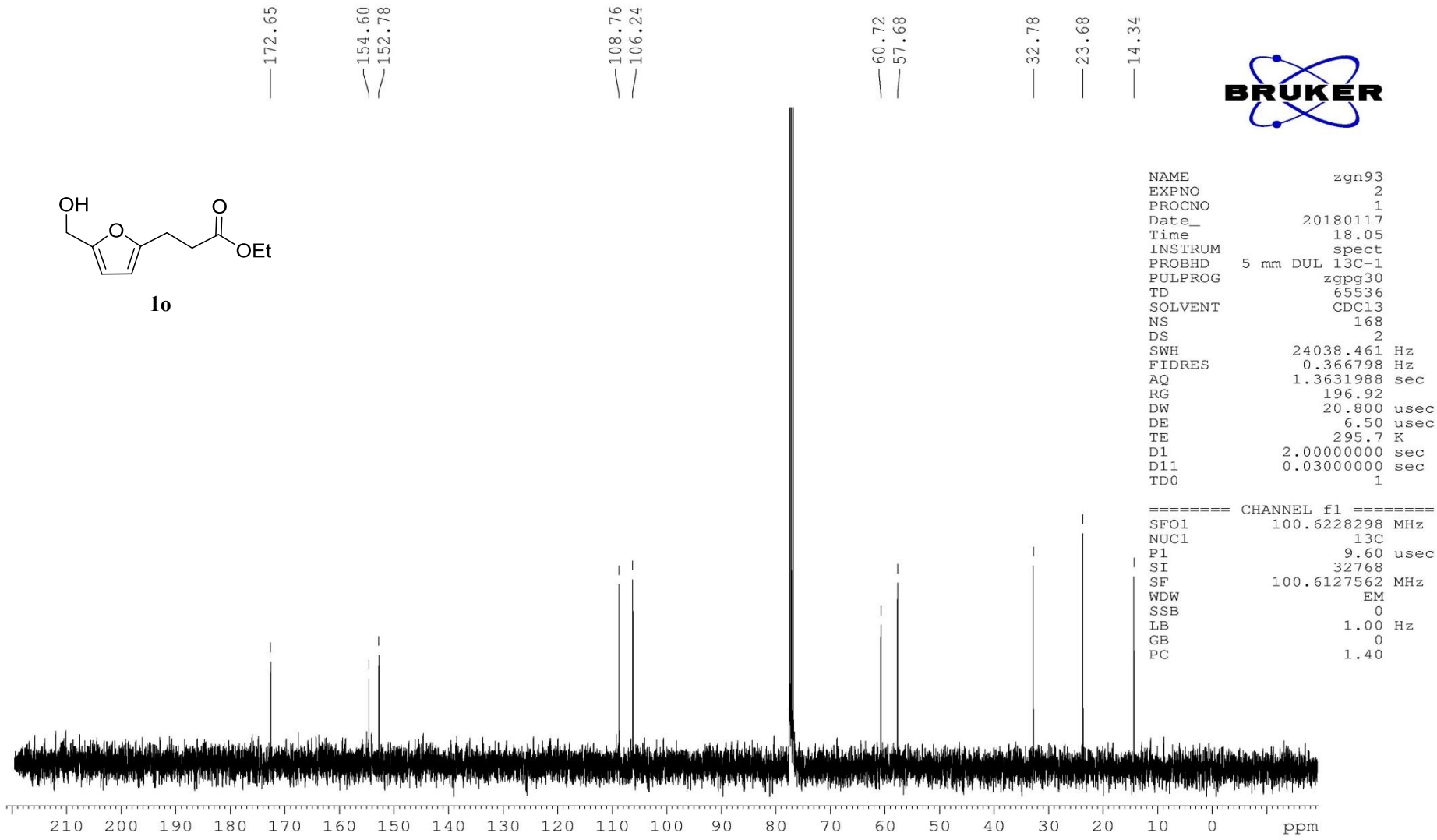

7.5893
7.5872
7.5851
7.4509
6.4085
6.4011
6.3963
6.2932
6.2860
5.4866
4.6623
4.6437
4.6296
4.1772
4.1703
4.1595
4.1526
4.1351
4.1057
4.0903
4.0808
4.0750
4.0656
2.0908
2.0749
2.0598
2.0413
OH
O
O
S
O
O
NH2
1n
BRUKER
NAME zgp110
EXPNO 4
PROCNO 1
Date_ 20180712
Time 16.36
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 3
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 142.88
DW 62.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.00
1.74
1.00
1.01
0.95
1.03
2.06
2.35

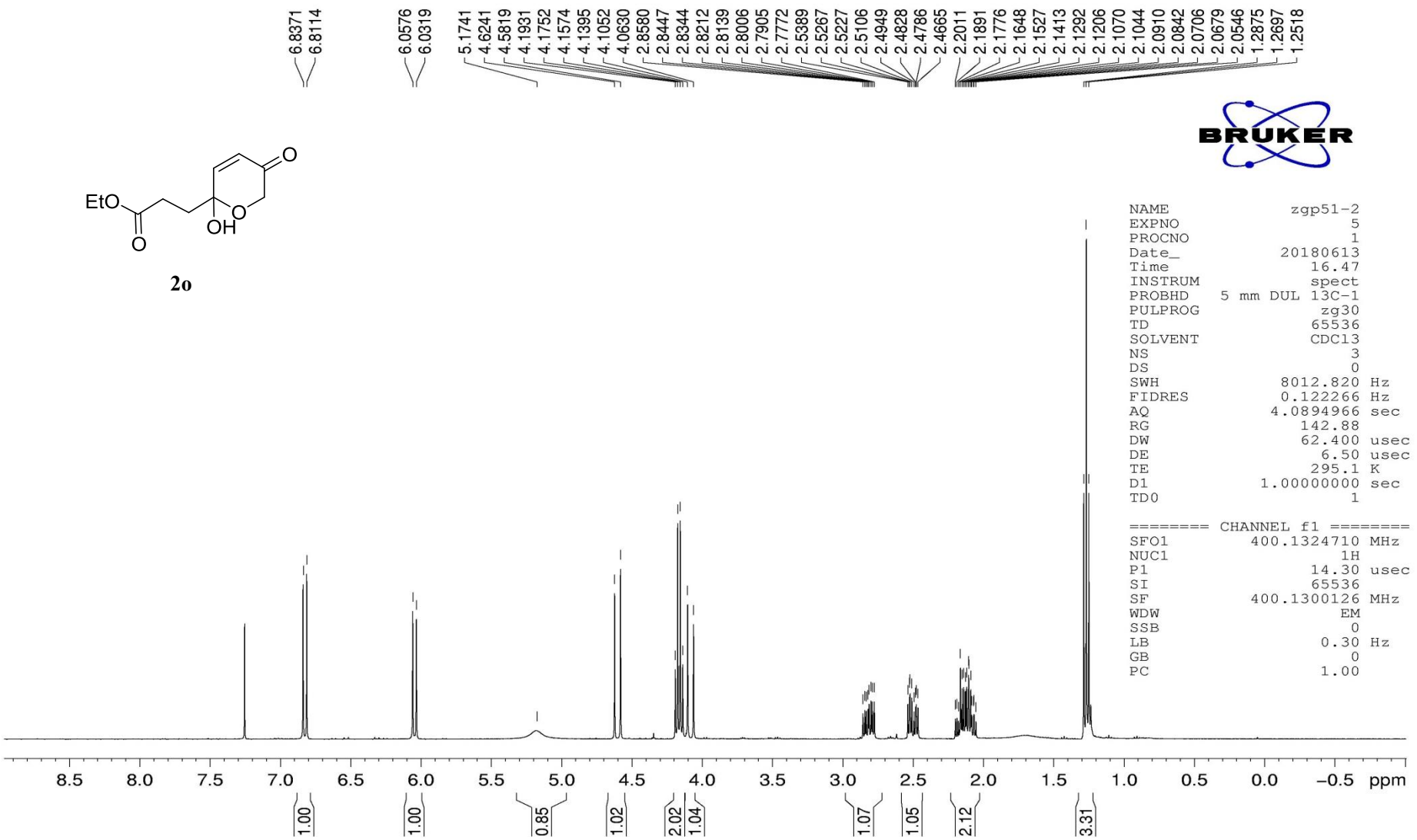

157.62
142.37
110.66
106.06
66.45
62.78
35.21
OH
O
O
O
S
O
NH2
1n
BRUKER
NAME zgp110
EXPNO 5
PROCNO 1
Date_ 20180712
Time 16.38
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 52
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.4663
7.3426
7.3240
7.0918
7.0831
7.0662
7.0575
7.0440
7.0405
7.0348
7.0180
6.1220
6.1183
6.0964
6.0927
6.0537
6.0281
5.6077
5.6043
5.5890
5.5857
5.5093
5.5006
5.4926
5.4839
4.6151
4.6049
4.5944
4.5842
4.2941
4.2863
4.2722
4.2646
4.1870
4.1752
4.1707
4.1671
4.1633
4.1571
4.1466
4.1290
4.1147
4.1109
2.2734
2.2651
2.2547
2.2472
2.2368
2.2295
2.2190
2.2108
2.2007
1.9881
1.9799
1.9728
1.9661
1.9525
1.9371
1.9319
1.9167
1.9015
1.8861
1.8809
1.8660
HO
O
O
O
O
S
NH2
2n
BRUKER
NAME zgp113
EXPNO 4
PROCNO 1
Date_ 20180712
Time 16.29
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 3
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 112.31
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
ppm
1.97
0.31
1.71
1.00
1.02
0.72
0.33
2.04
1.01
1.03

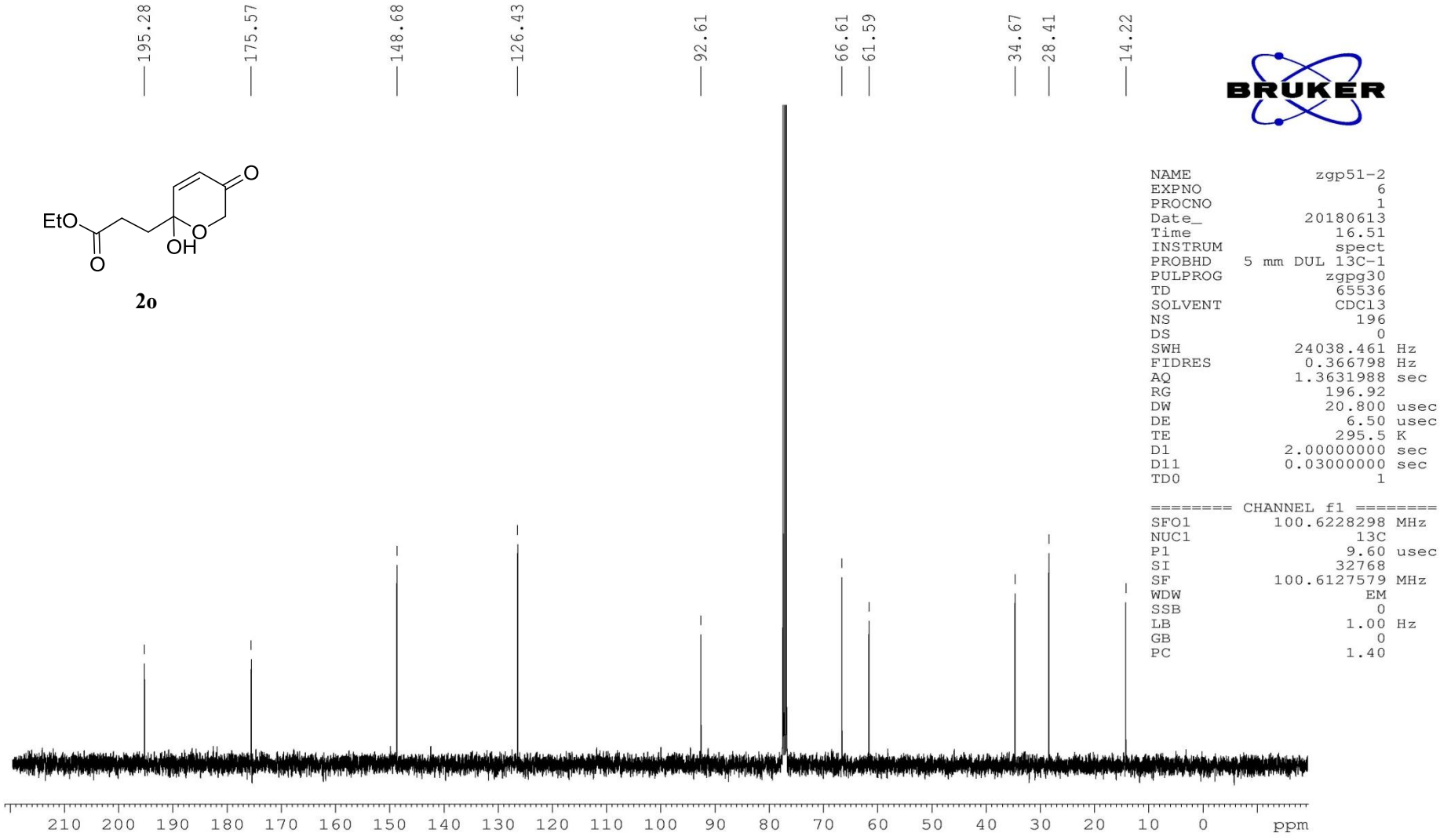

196.75
196.48
151.88
148.31
127.68
125.99
90.87
87.20
74.62
70.10
65.82
65.77
30.25
29.59
BRUKER
HO
O
O
O
S
O
NH2
2n
NAME zgp113
EXPNO 5
PROCNO 1
Date_ 20180712
Time 16.32
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 78
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

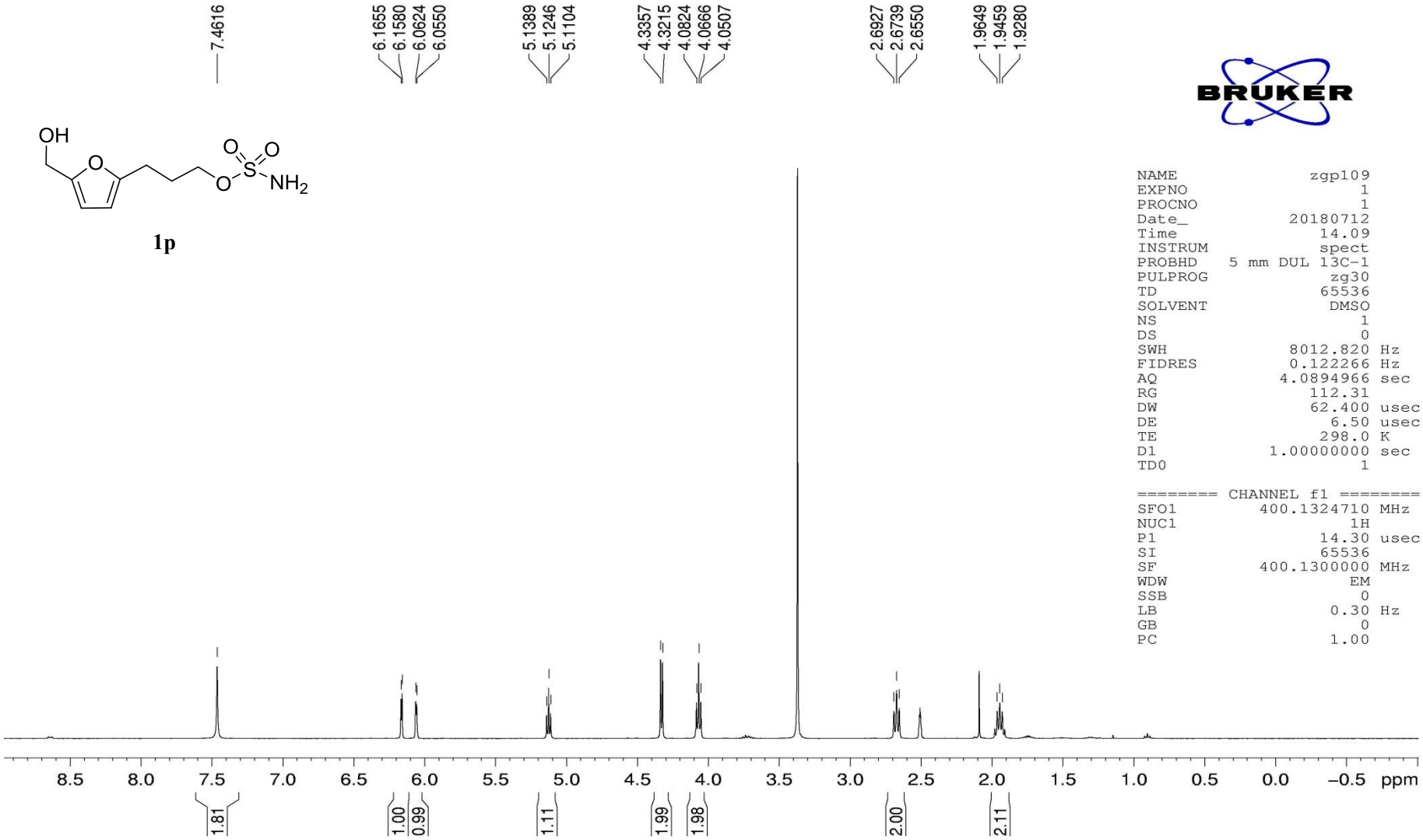

7.2862
6.2018
6.1941
5.9955
5.9877
4.5706
4.1941
4.1763
4.1660
4.1584
4.1482
4.1406
2.9958
2.9769
2.9578
2.6885
2.6718
2.6687
2.6504
1.2925
1.2839
1.2747
1.2661
1.2568
OH
O
O
OEt
1o
NAME zgn93
EXPNO 1
PROCNO 1
Date_ 20180116
Time 21.44
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 187.77
DW 62.400 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.00
1.04
1.95
2.11
2.18
2.18
3.38

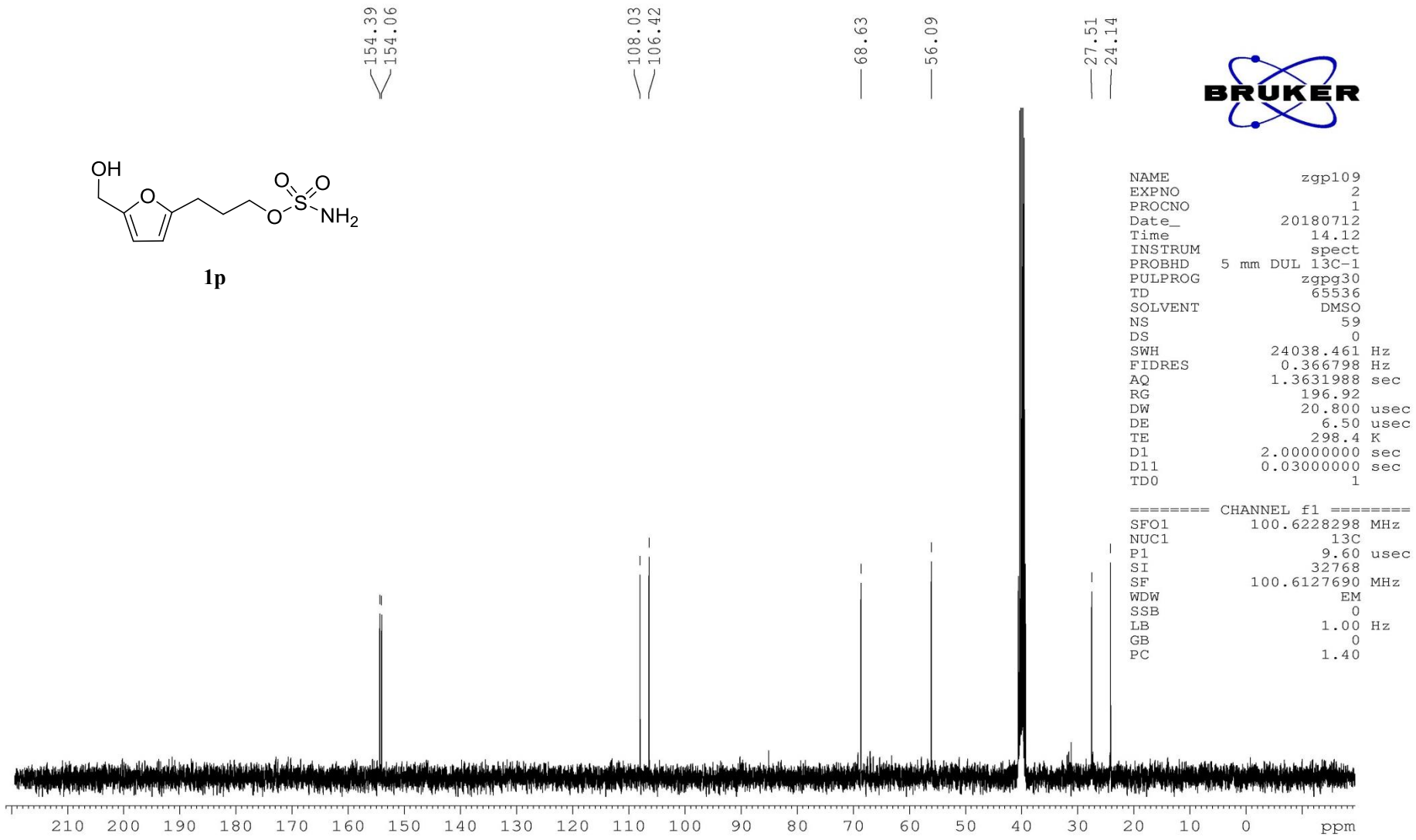

172.65
154.60
152.78
108.76
106.24
60.72
57.68
32.78
23.68
14.34
OH
O
O
OEt
1o
NAME zgn93
EXPNO 2
PROCNO 1
Date_ 20180117
Time 18.05
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 168
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 295.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127562 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

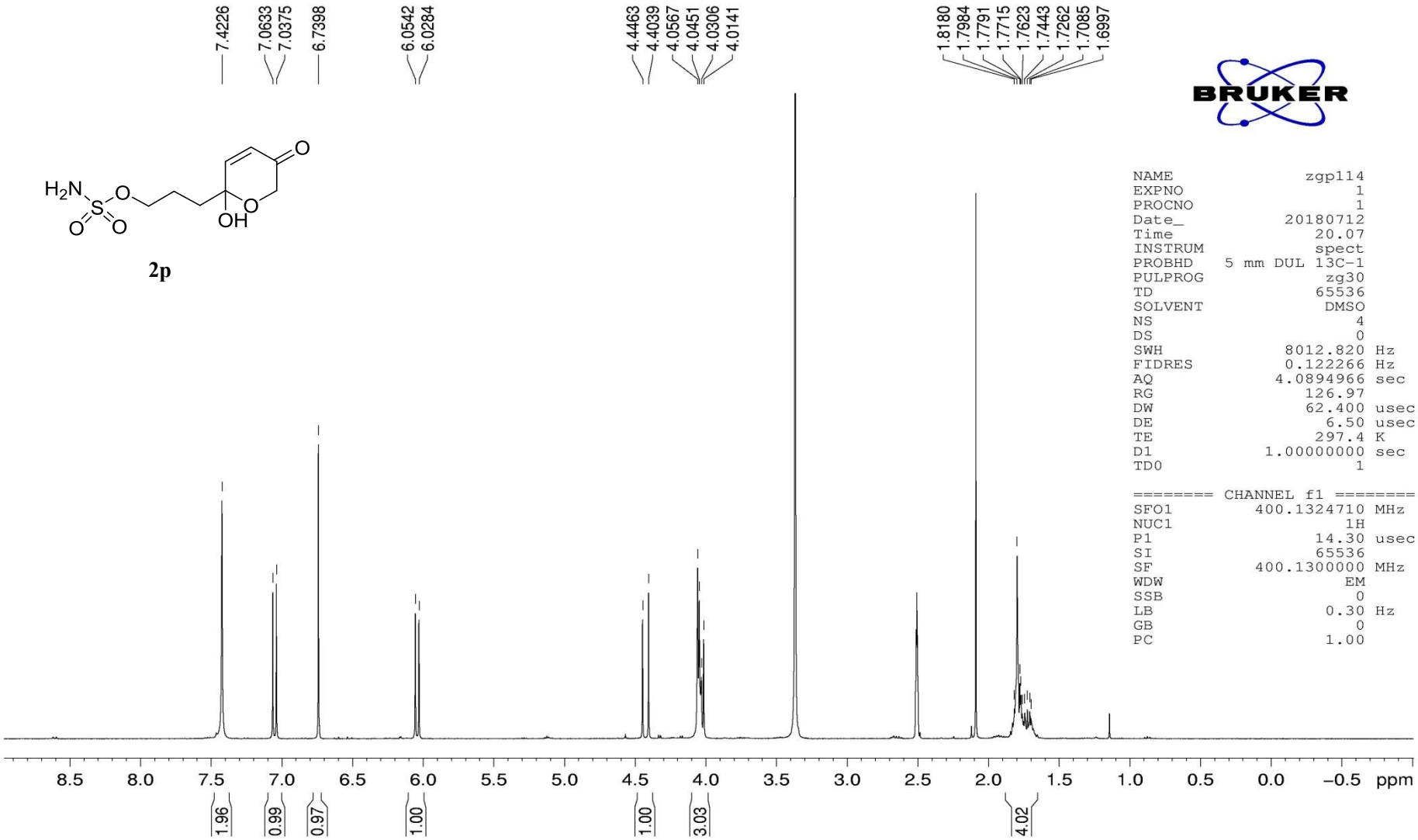

6.8371
6.8114
6.0576
6.0319
5.1741
4.6241
4.5819
4.1931
4.1752
4.1574
4.1395
4.1052
4.0630
2.8580
2.8447
2.8344
2.8212
2.8139
2.8006
2.7905
2.7772
2.5389
2.5267
2.5227
2.5106
2.4949
2.4828
2.4786
2.4665
2.2011
2.1891
2.1776
2.1648
2.1527
2.1413
2.1292
2.1206
2.1070
2.1044
2.0910
2.0842
2.0706
2.0679
2.0546
1.2875
1.2697
1.2518
EtO
O
O
OH
O
BRUKER
NAME zgp51-2
EXPNO 5
PROCNO 1
Date_ 20180613
Time 16.47
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 142.88
DW 62.400 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300126 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.00
1.00
0.85
1.02
2.02
1.04
1.07
1.05
2.12
3.31

**2o**

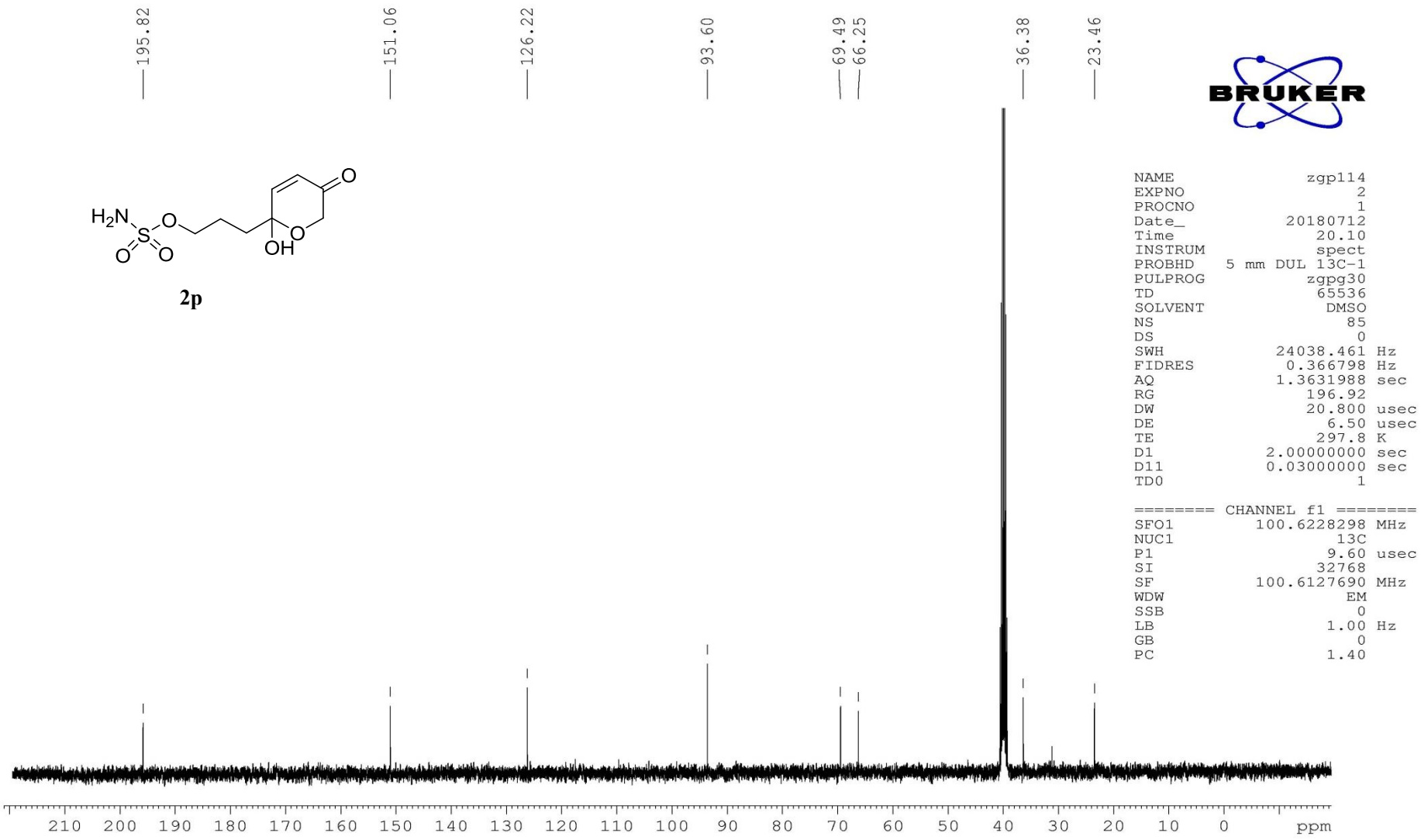

195.28
175.57
148.68
126.43
92.61
66.61
61.59
34.67
28.41
14.22
EtO
O
O
OH
O
2o
BRUKER
NAME zgp51-2
EXPNO 6
PROCNO 1
Date_ 20180613
Time 16.51
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 196
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 295.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127579 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.4616
6.1655
6.1580
6.0624
6.0550
5.1389
5.1246
5.1104
4.3357
4.3215
4.0824
4.0666
4.0507
2.6927
2.6739
2.6550
1.9649
1.9459
1.9280
OH
O
O
O
S
O
NH2
1p
BRUKER
NAME zgp109
EXPNO 1
PROCNO 1
Date_ 20180712
Time 14.09
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 112.31
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
ppm
1.81
1.00
0.99
1.11
1.99
1.98
2.00
2.11

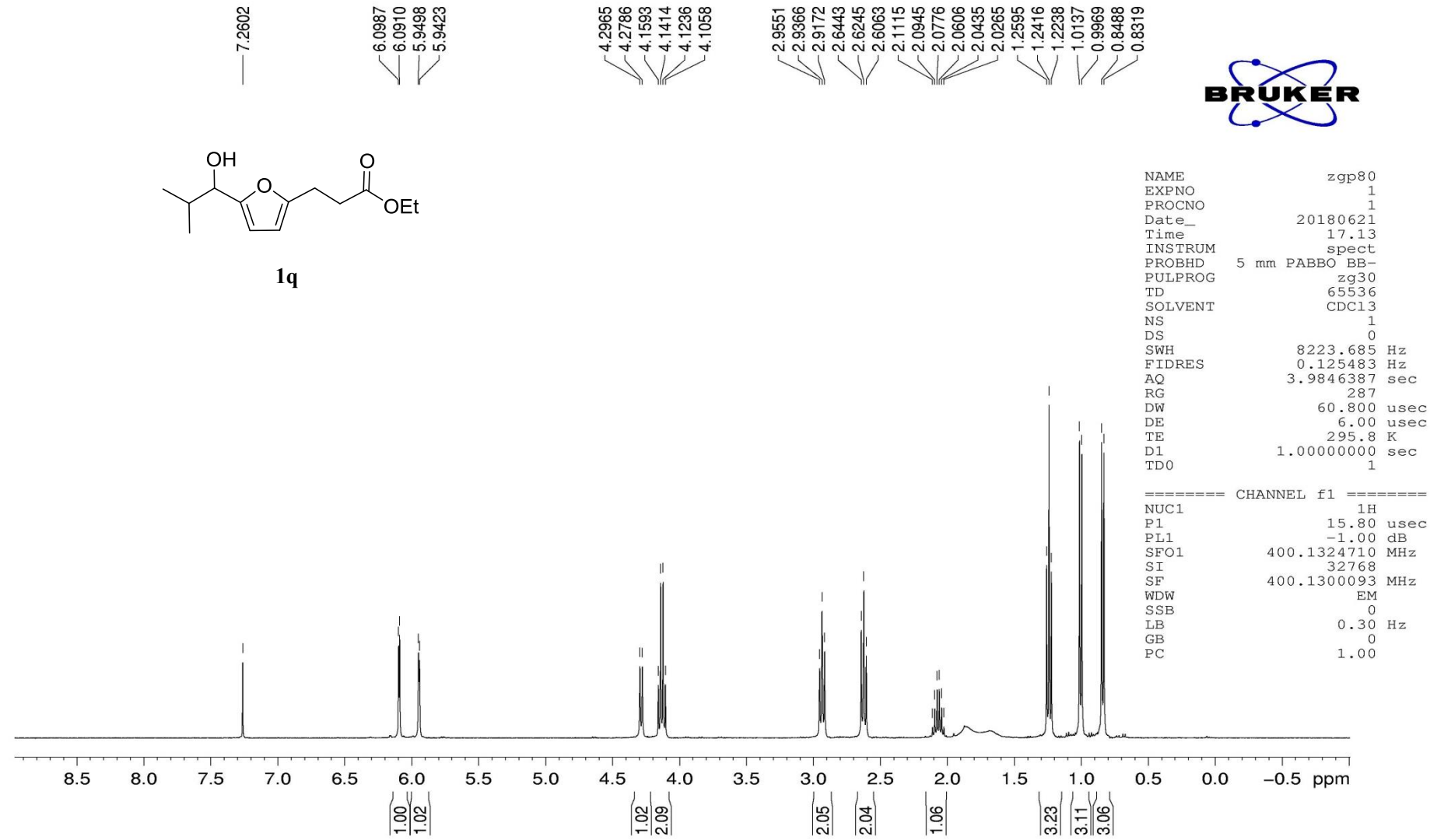

154.39
154.06
108.03
106.42
68.63
56.09
27.51
24.14
OH
O
O
S
O
O
NH2
1p
NAME zgp109
EXPNO 2
PROCNO 1
Date_ 20180712
Time 14.12
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 59
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

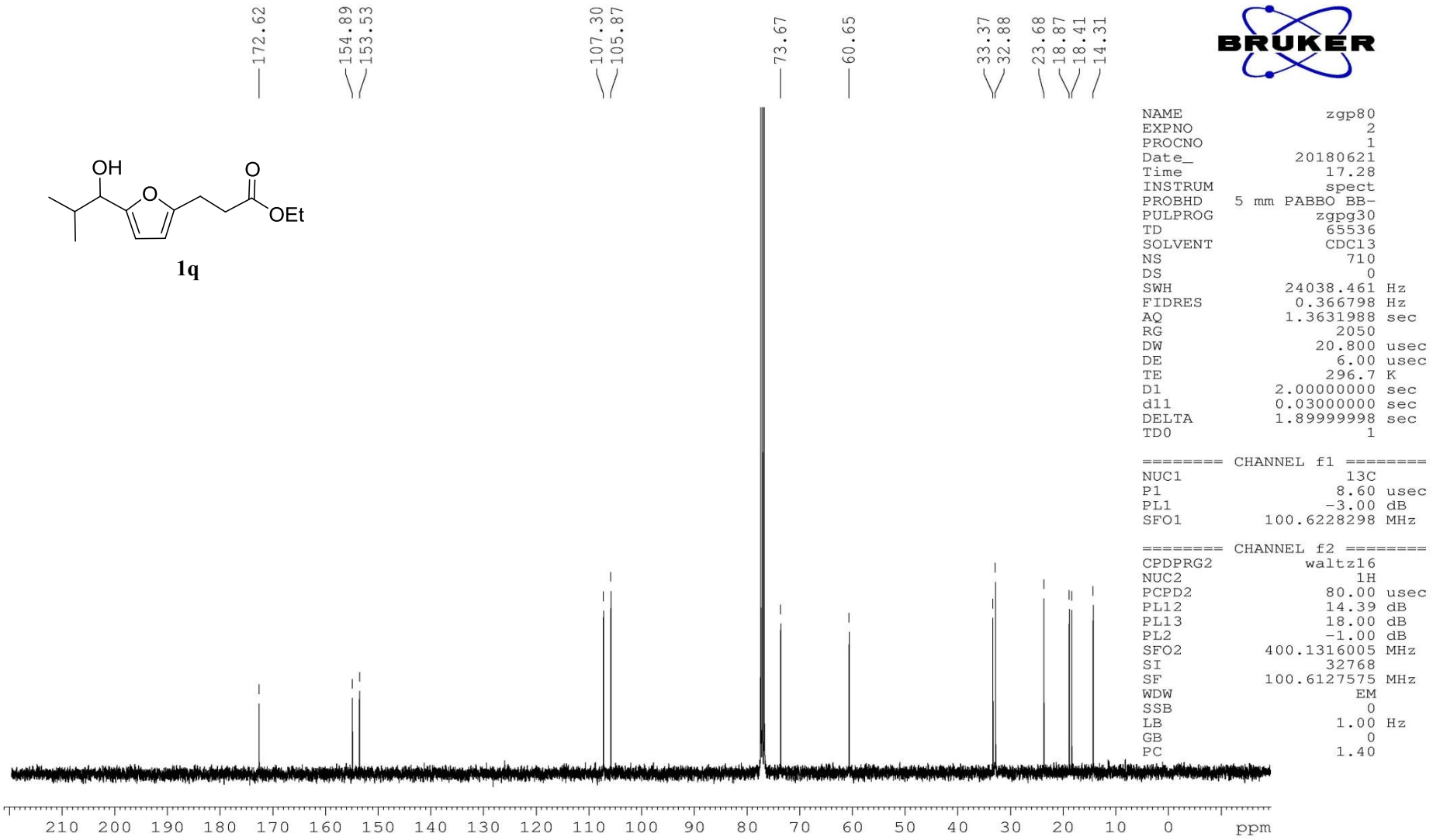

7.4226
7.0633
7.0375
6.7398
6.0542
6.0284
4.4463
4.4039
4.0567
4.0451
4.0306
4.0141
1.8180
1.7984
1.7791
1.7715
1.7623
1.7443
1.7262
1.7085
1.6997
BRUKER
NAME zgp114
EXPNO 1
PROCNO 1
Date_ 20180712
Time 20.07
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 4
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 126.97
DW 62.400 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.96
0.99
0.97
1.00
1.00
3.03
4.02

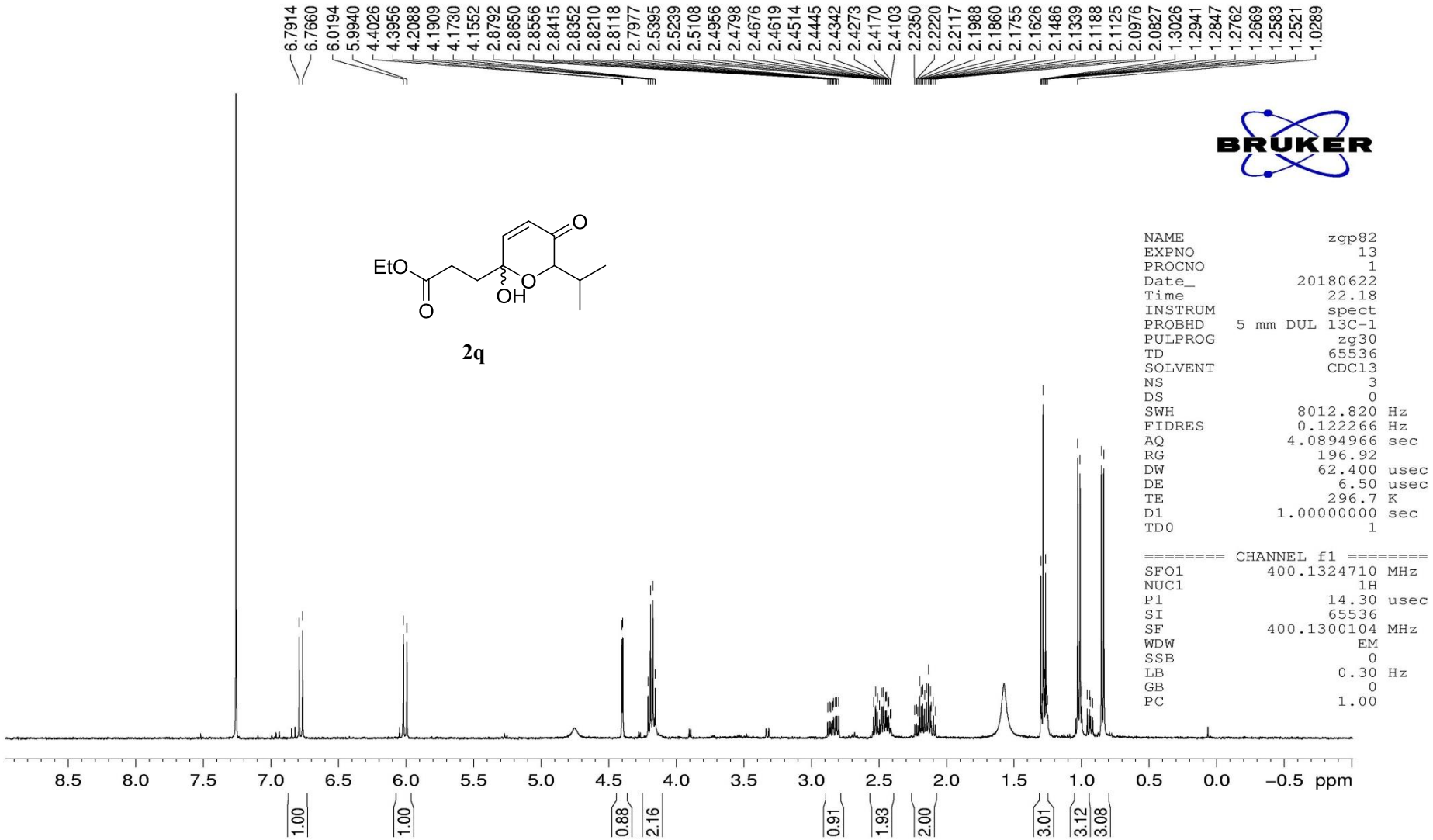
**2p**

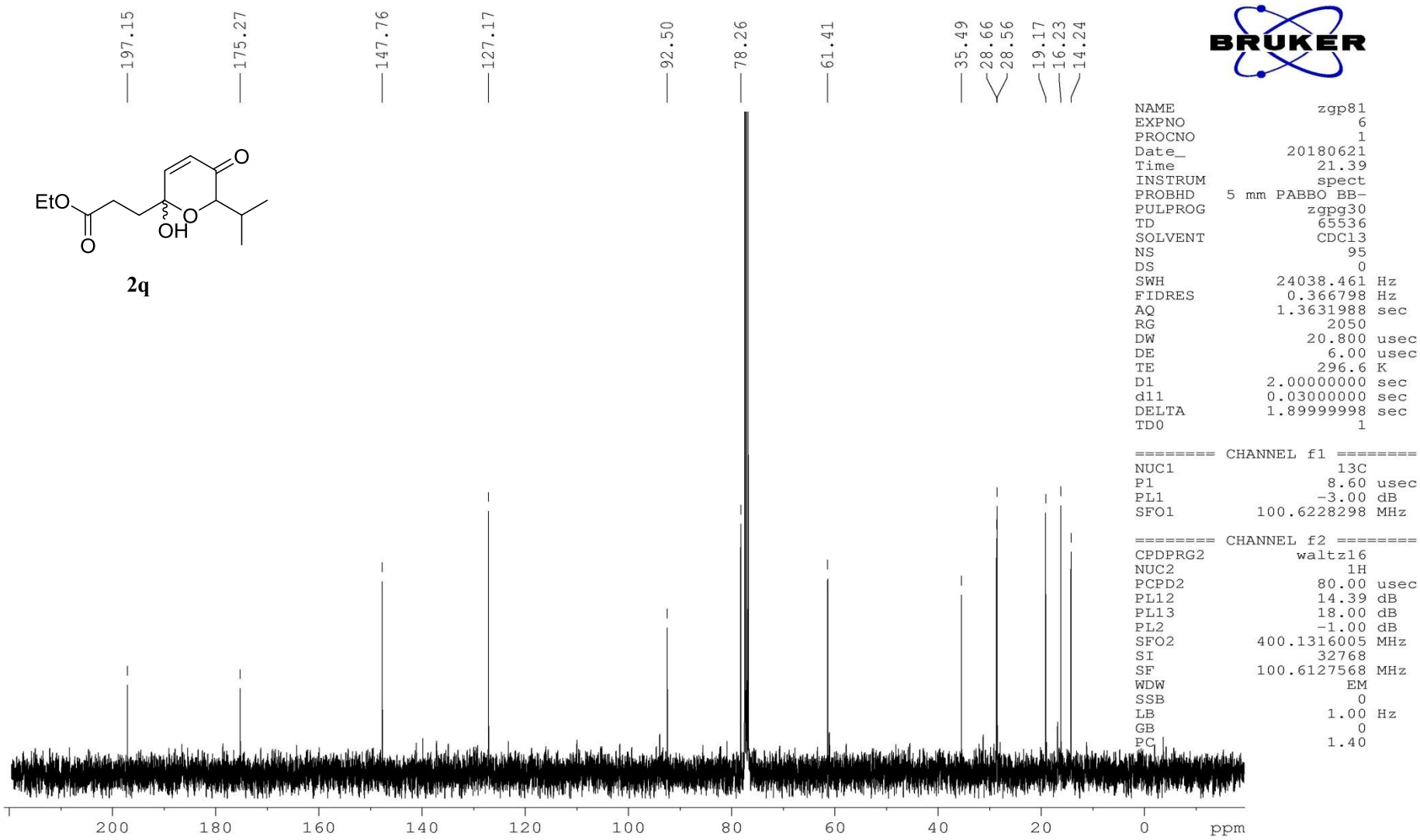

195.82
151.06
126.22
93.60
69.49
66.25
36.38
23.46
BRUKER
NAME zgp114
EXPNO 2
PROCNO 1
Date_ 20180712
Time 20.10
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 85
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

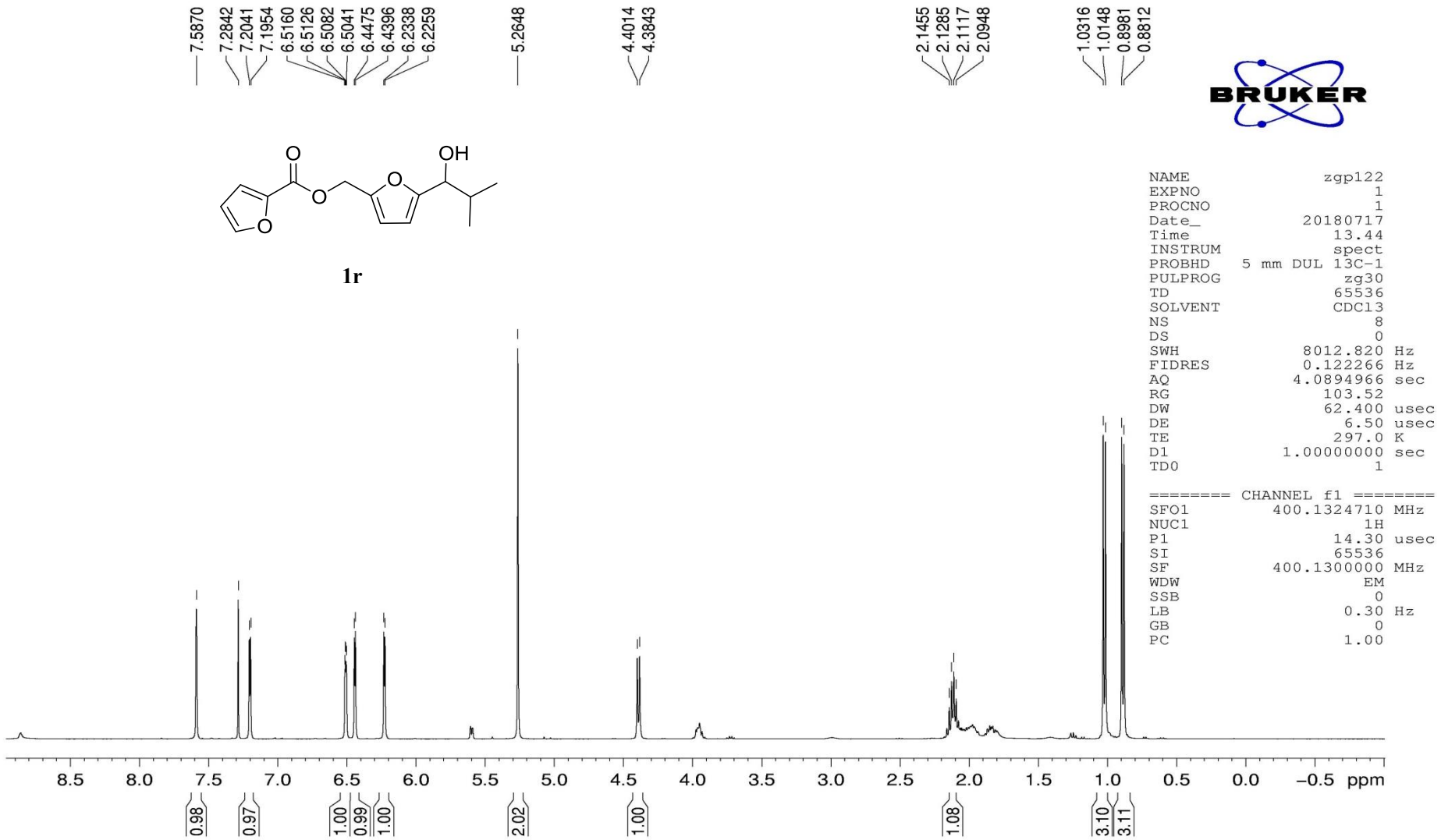
**2p**

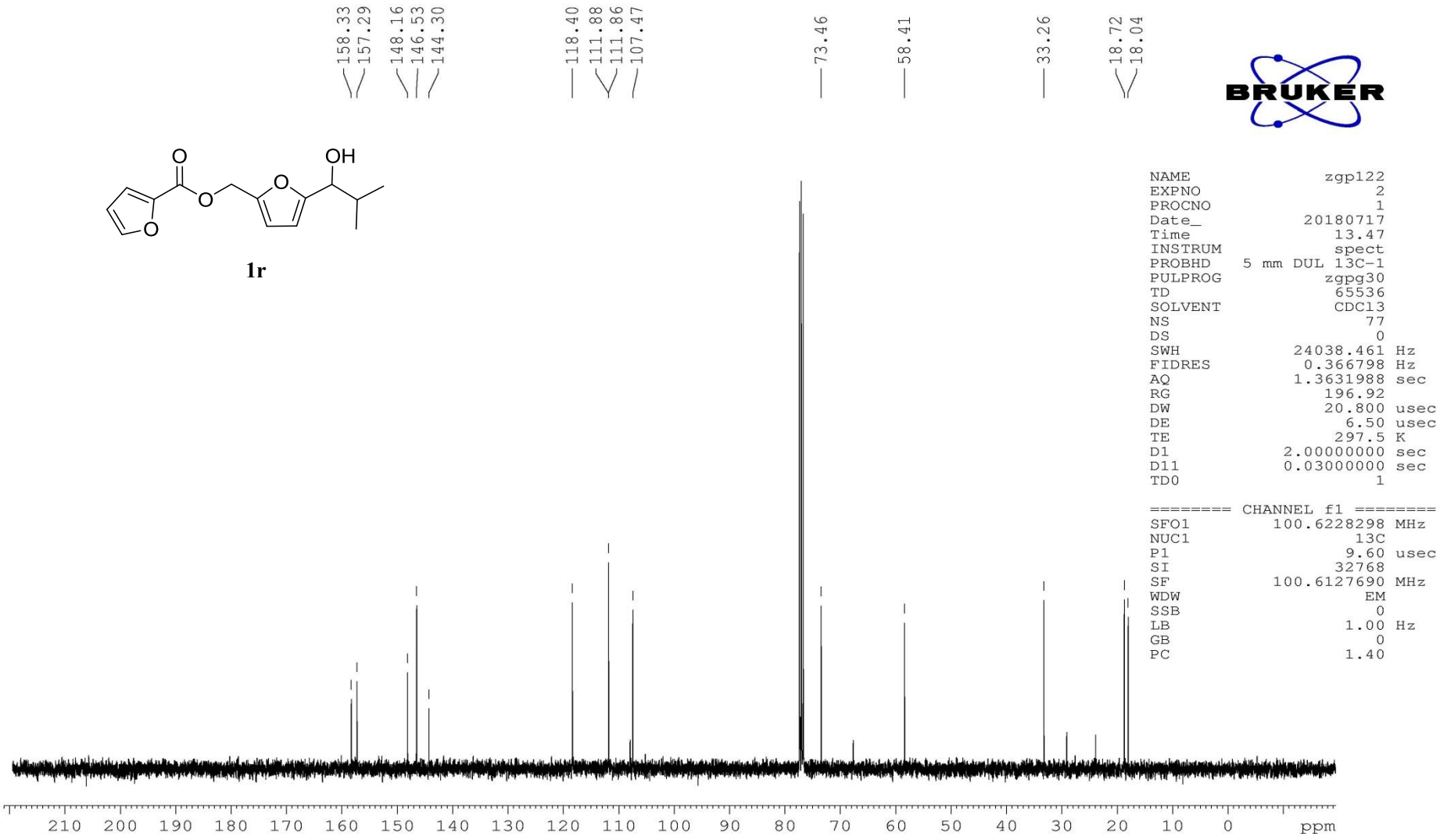

7.2602
6.0987
6.0910
5.9498
5.9423
4.2965
4.2786
4.1593
4.1414
4.1236
4.1058
2.9551
2.9366
2.9172
2.6443
2.6245
2.6063
2.1115
2.0945
2.0776
2.0606
2.0435
2.0265
1.2595
1.2416
1.2238
1.0137
0.9969
0.8488
0.8319
OH
O
O
OEt
1q
BRUKER
NAME zgp80
EXPNO 1
PROCNO 1
Date_ 20180621
Time 17.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 287
DW 60.800 usec
DE 6.00 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.00 1.02 1.02 2.09 2.05 2.04 1.06 3.23 3.11 3.06

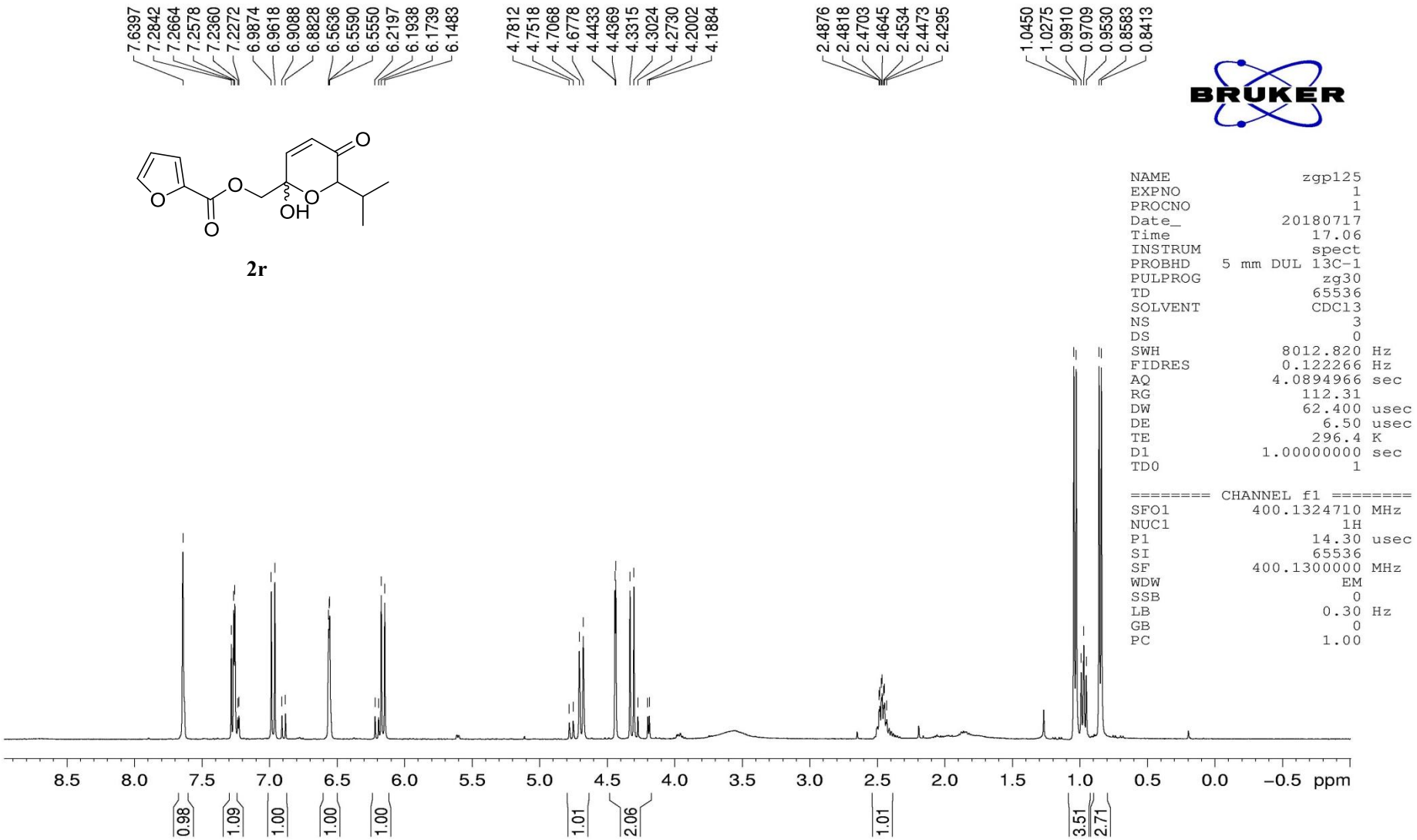

172.62
154.89
153.53
107.30
105.87
73.67
60.65
33.37
32.88
23.68
18.87
18.41
14.31
OH
O
O
OEt
1q
BRUKER
NAME zgp80
EXPNO 2
PROCNO 1
Date_ 20180621
Time 17.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 710
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 296.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 14.39 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127575 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

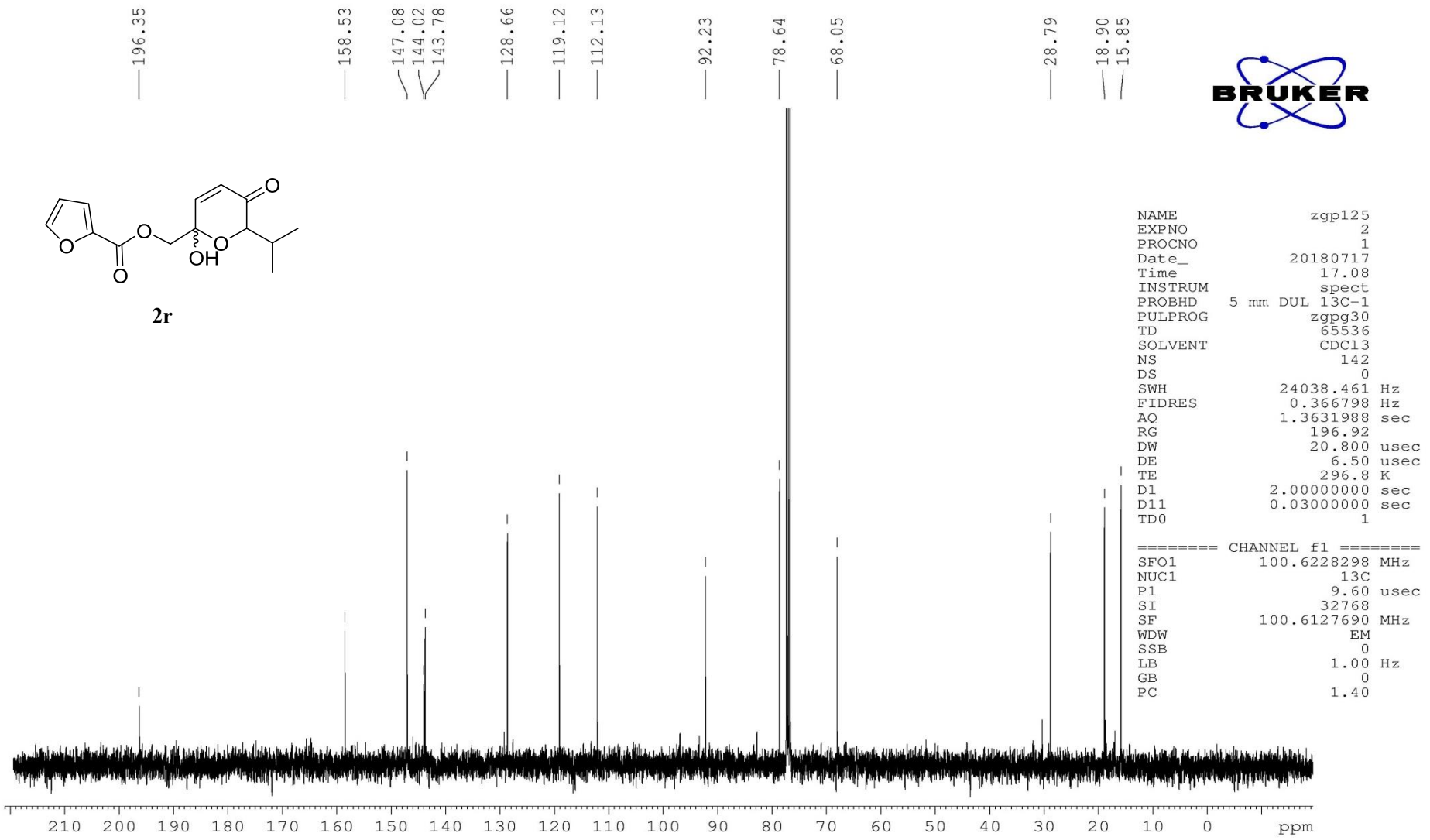

6.7914
6.7660
6.0194
5.9940
4.4026
4.3956
4.2088
4.1909
4.1730
4.1552
2.8792
2.8650
2.8556
2.8415
2.8352
2.8210
2.8118
2.7977
2.5395
2.5239
2.5108
2.4956
2.4798
2.4676
2.4619
2.4514
2.4445
2.4342
2.4273
2.4170
2.4103
2.2350
2.2220
2.2117
2.1988
2.1860
2.1755
2.1626
2.1486
2.1339
2.1188
2.1125
2.0976
2.0827
1.3026
1.2941
1.2847
1.2762
1.2669
1.2583
1.2521
1.0289
BRUKER
EtO
O
O
OH
O
2q
NAME zgp82
EXPNO 13
PROCNO 1
Date_ 20180622
Time 22.18
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 196.92
DW 62.400 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300104 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.00
1.00
0.88
2.16
0.91
1.93
2.00
3.01
3.12
3.08

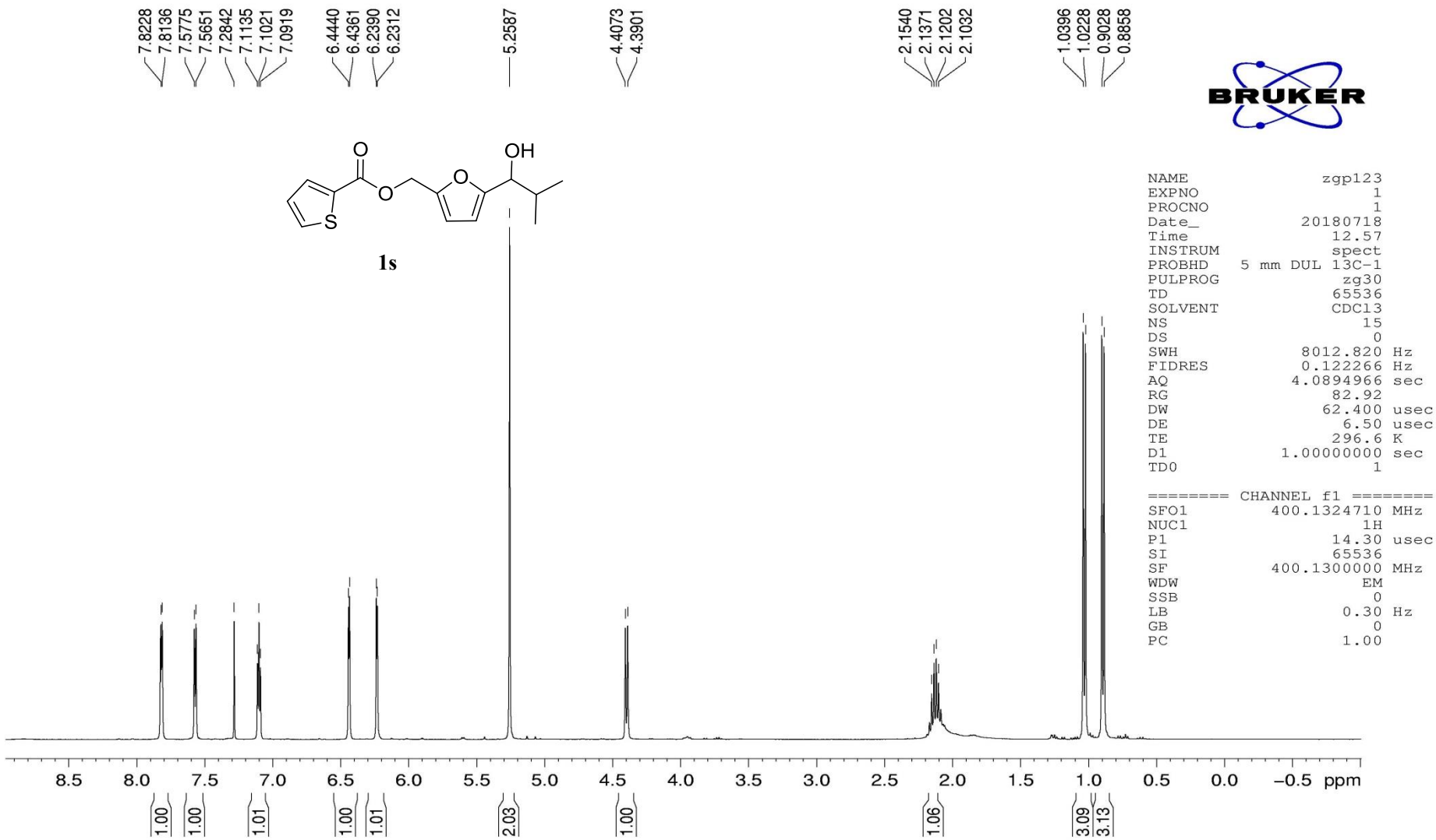

197.15
175.27
147.76
127.17
92.50
78.26
61.41
35.49
28.66
28.56
19.17
16.23
14.24
EtO
O
O
OH
O
2q
NAME zgp81
EXPNO 6
PROCNO 1
Date_ 20180621
Time 21.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 95
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 296.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 14.39 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127568 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

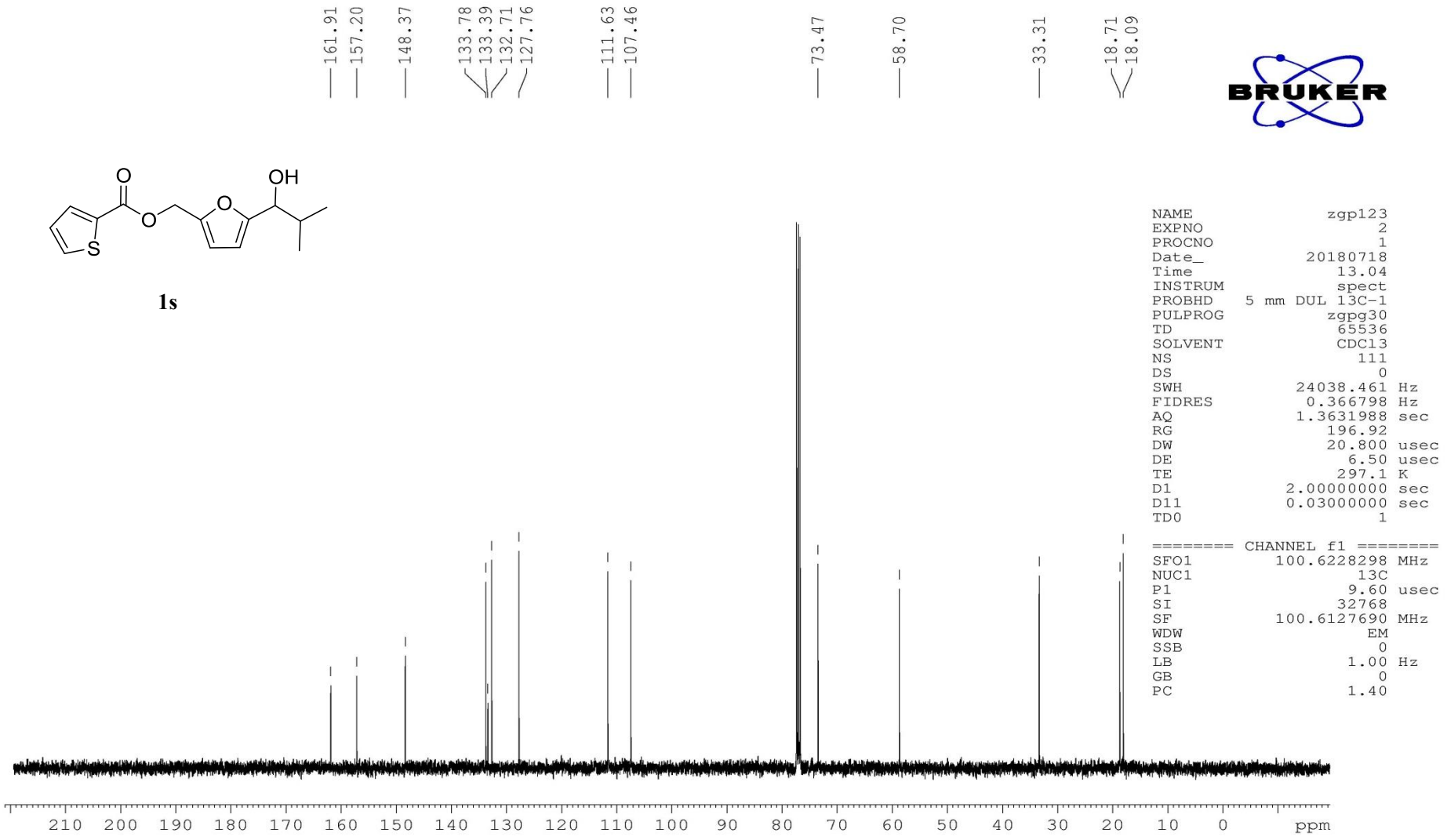

7.5870
7.2842
7.2041
7.1954
6.5160
6.5126
6.5082
6.5041
6.4475
6.4396
6.2338
6.2259
5.2648
4.4014
4.3843
2.1455
2.1285
2.1117
2.0948
1.0316
1.0148
0.8981
0.8812
O
O
O
OH
1r
BRUKER
NAME zgp122
EXPNO 1
PROCNO 1
Date_ 20180717
Time 13.44
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 103.52
DW 62.400 usec
DE 6.50 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
0.98
0.97
1.00
0.99
1.00
2.02
1.00
1.08
3.10
3.11

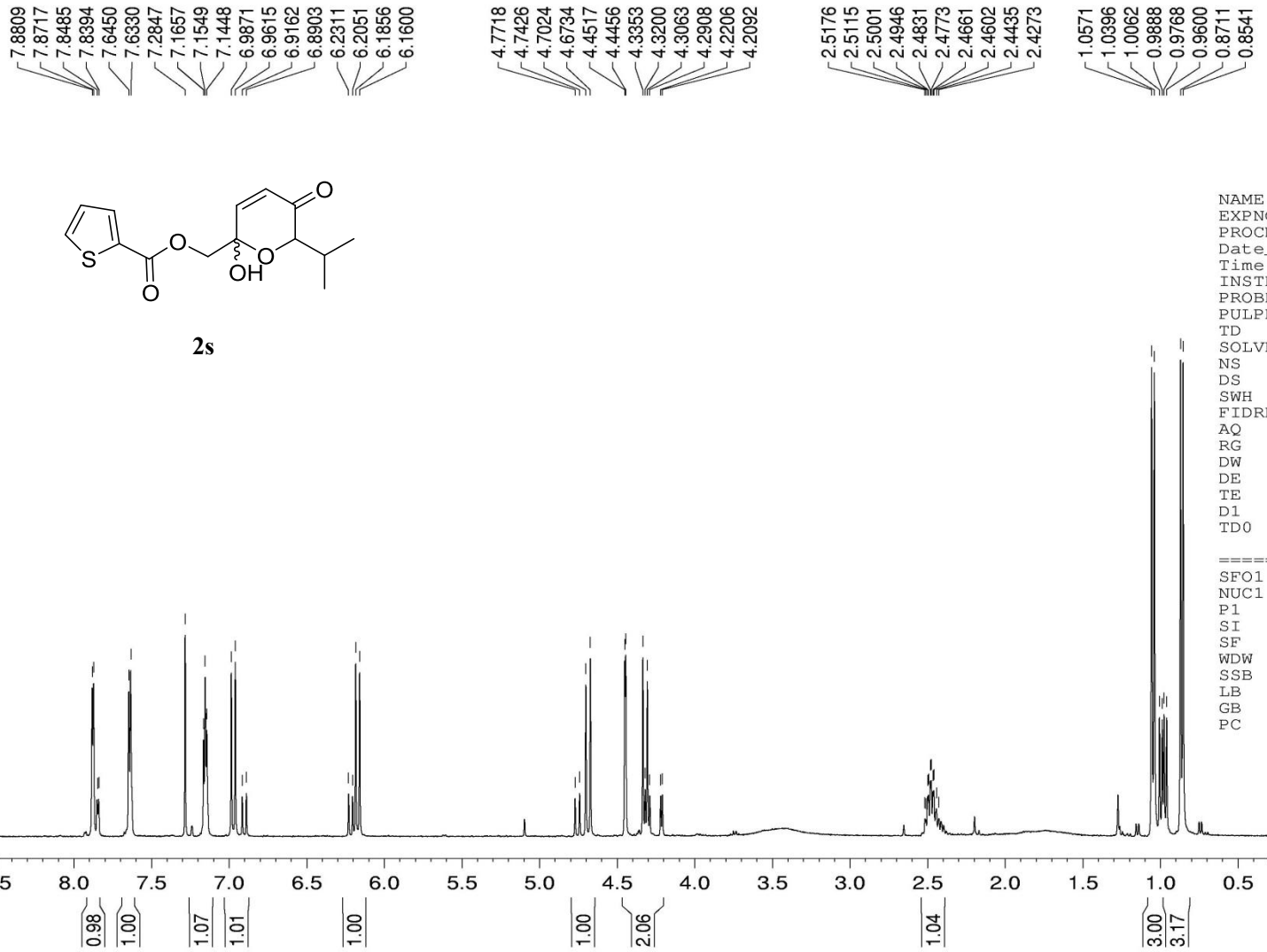

158.33
157.29
148.16
146.53
144.30
118.40
111.88
111.86
107.47
73.46
58.41
33.26
18.72
18.04
BRUKER
O
O
O
OH
1r
NAME zgp122
EXPNO 2
PROCNO 1
Date_ 20180717
Time 13.47
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 77
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.6397
7.2842
7.2664
7.2578
7.2360
7.2272
6.9874
6.9618
6.9088
6.8828
6.5636
6.5590
6.5550
6.2197
6.1938
6.1739
6.1483
4.7812
4.7518
4.7068
4.6778
4.4433
4.4369
4.3315
4.3024
4.2730
4.2002
4.1884
2.4876
2.4818
2.4703
2.4645
2.4534
2.4473
2.4295
1.0450
1.0275
0.9910
0.9709
0.9530
0.8583
0.8413
BRUKER
O
O
O
O
OH
O
2r
NAME zgp125
EXPNO 1
PROCNO 1
Date_ 20180717
Time 17.06
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 112.31
DW 62.400 usec
DE 6.50 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
0.98
1.09
1.00
1.00
1.00
1.01
2.06
1.01
3.51
2.71

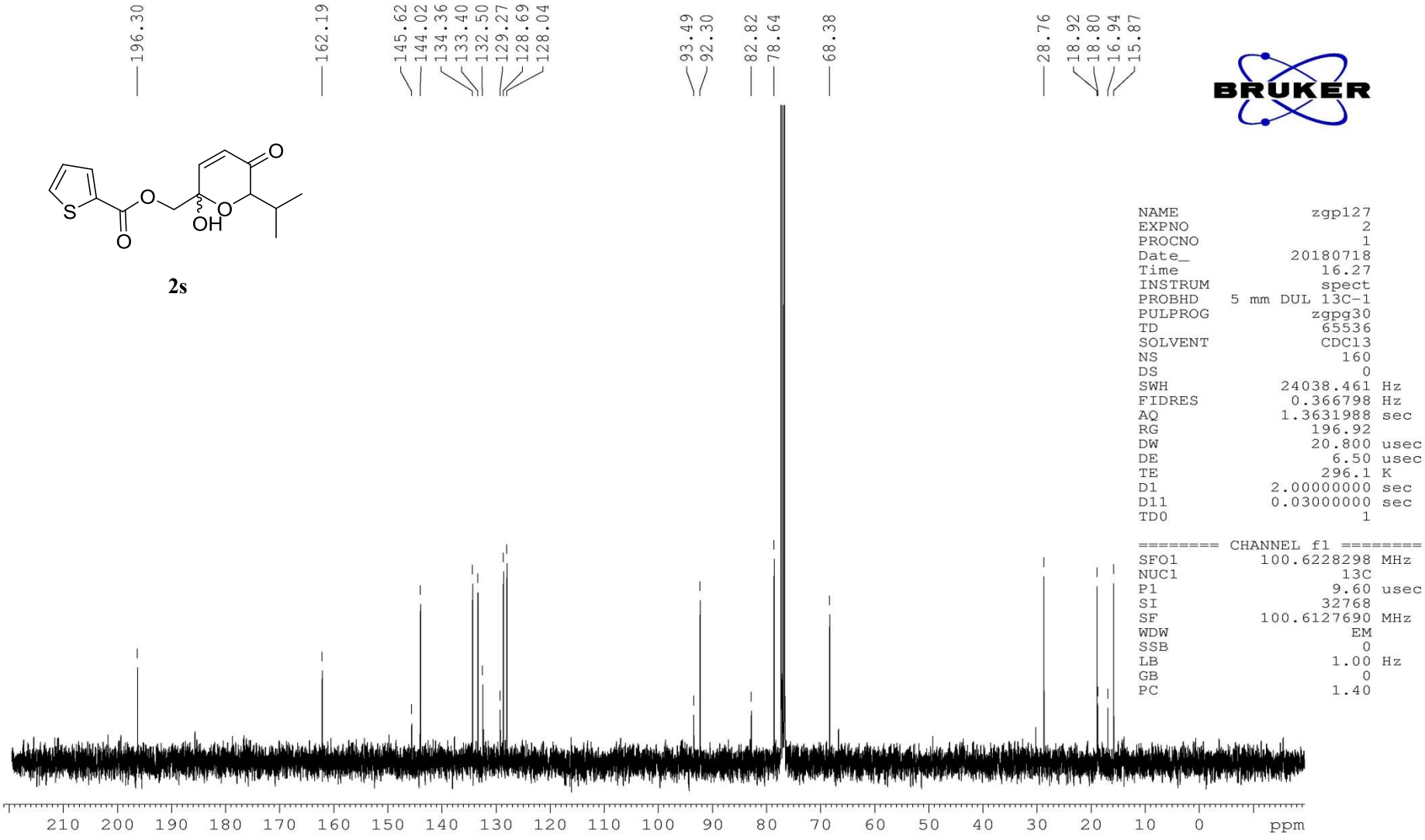

196.35
158.53
147.08
144.02
143.78
128.66
119.12
112.13
92.23
78.64
68.05
28.79
18.90
15.85
BRUKER
O
O
O
O
OH
2r
NAME zgp125
EXPNO 2
PROCNO 1
Date_ 20180717
Time 17.08
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 142
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

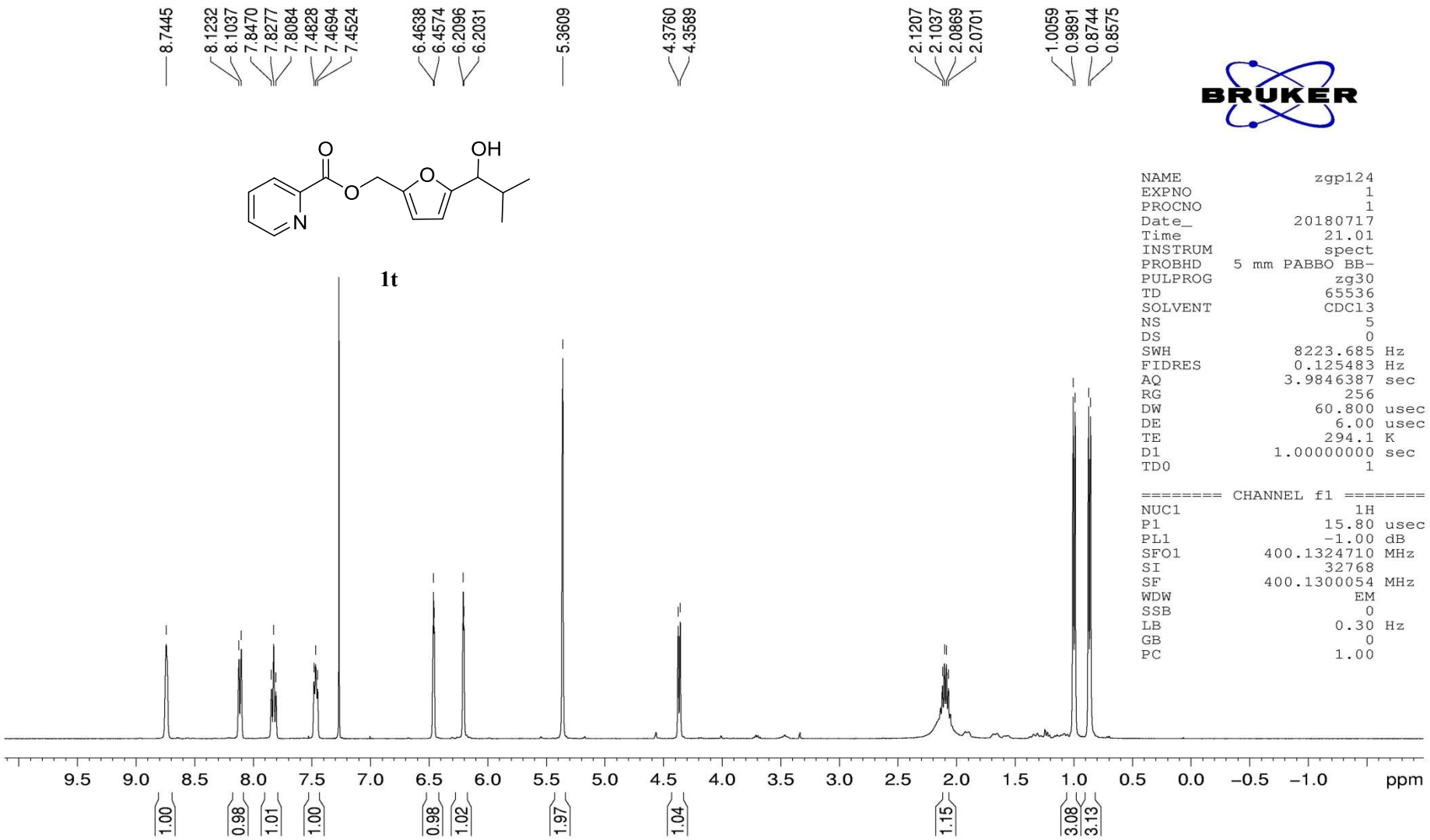

7.8228
7.8136
7.5775
7.5651
7.2842
7.1135
7.1021
7.0919
6.4440
6.4361
6.2390
6.2312
5.2587
4.4073
4.3901
2.1540
2.1371
2.1202
2.1032
1.0396
1.0228
0.9028
0.8858
O
OH
O
O
S
1s
BRUKER
NAME zgp123
EXPNO 1
PROCNO 1
Date_ 20180718
Time 12.57
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 15
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 82.92
DW 62.400 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.00
1.00
1.01
1.00
1.01
2.03
1.00
1.06
3.09
3.13

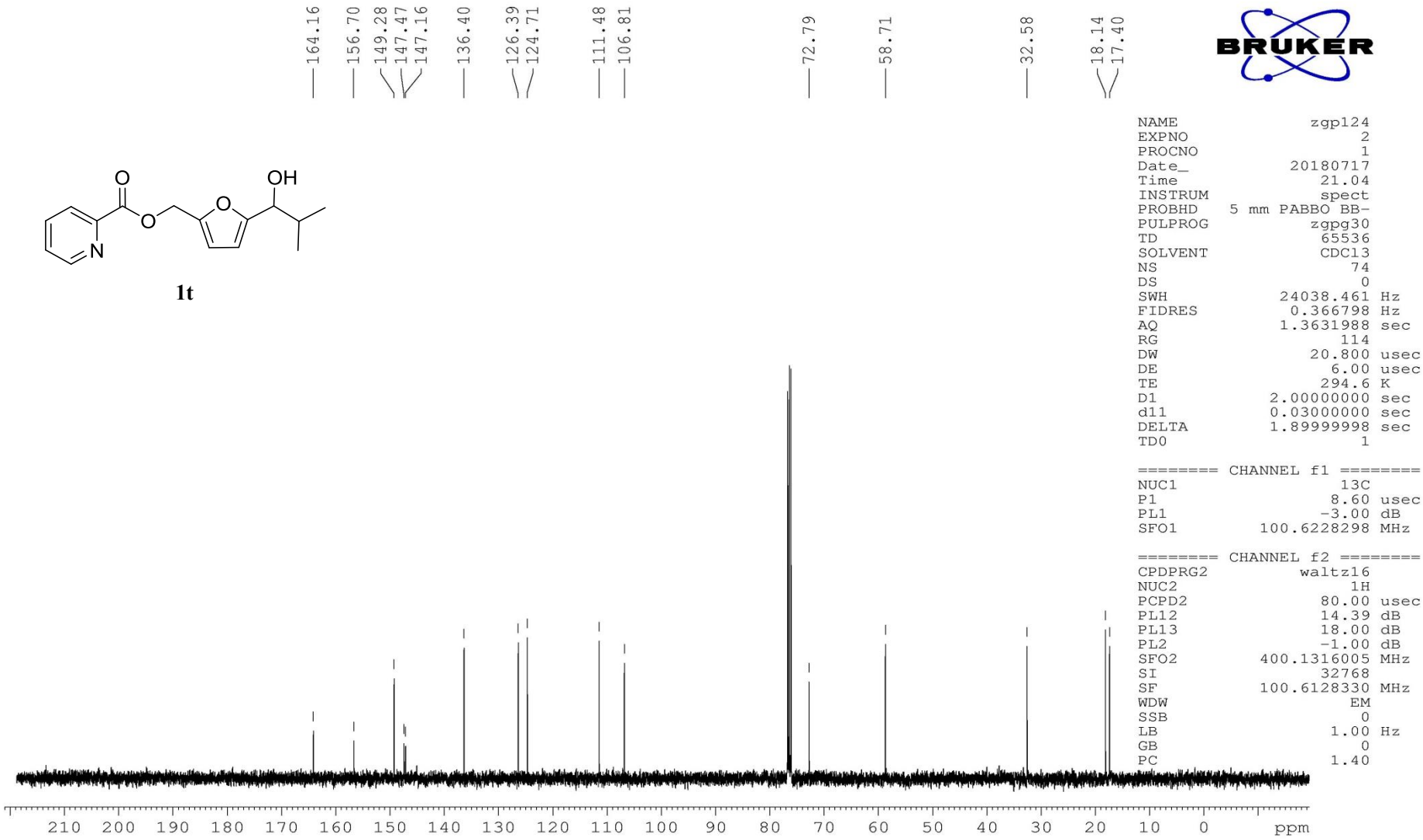

161.91
157.20
148.37
133.78
133.39
132.71
127.76
111.63
107.46
73.47
58.70
33.31
18.71
18.09
BRUKER
O
O
O
OH
S
1s
NAME zgp123
EXPNO 2
PROCNO 1
Date_ 20180718
Time 13.04
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 111
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

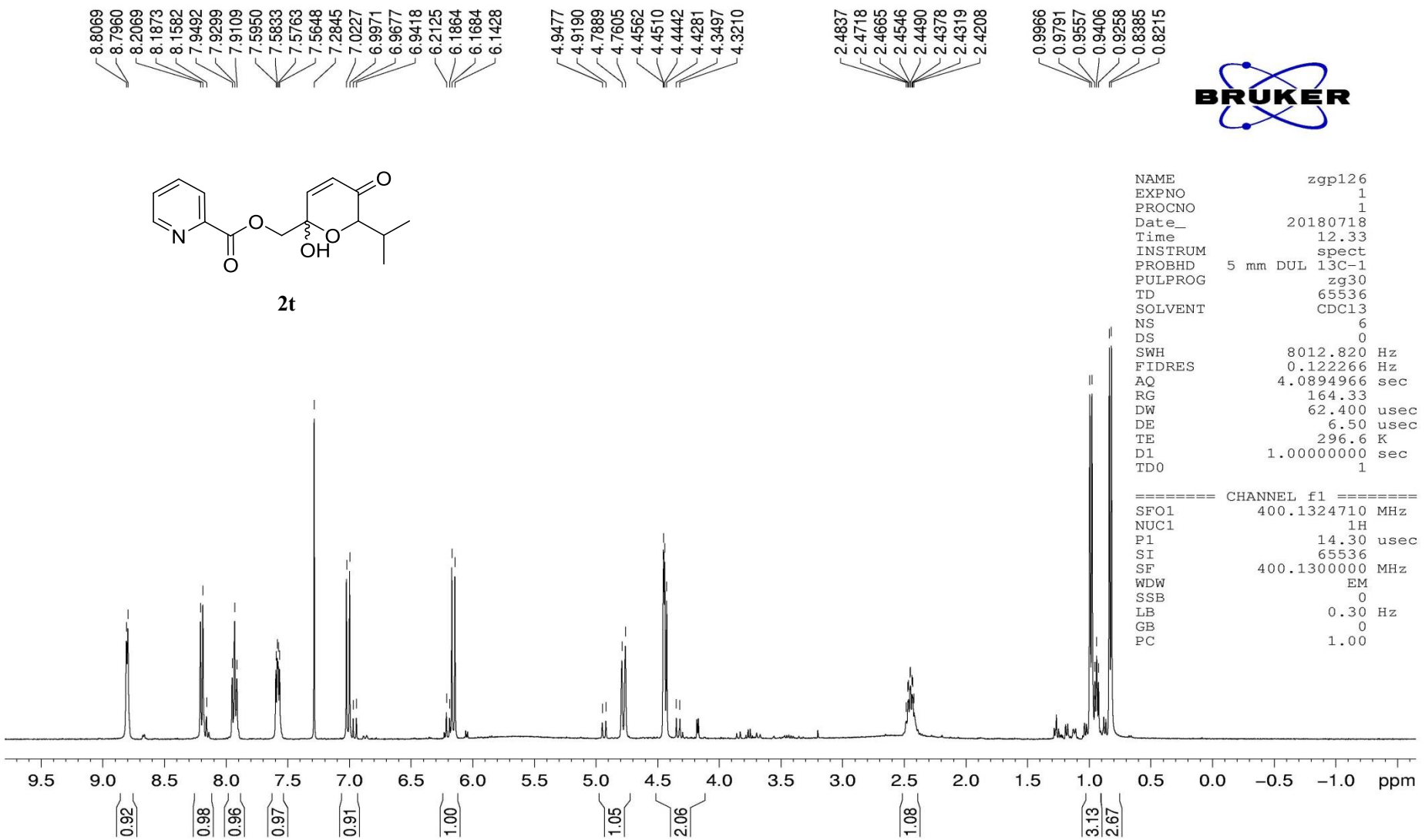

7.8809
7.8717
7.8485
7.8394
7.6450
7.6330
7.2847
7.1657
7.1549
7.1448
6.9871
6.9615
6.9162
6.8903
6.2311
6.2051
6.1856
6.1600
4.7718
4.7426
4.7024
4.6734
4.4517
4.4456
4.3353
4.3200
4.3063
4.2908
4.2206
4.2092
2.5176
2.5115
2.5001
2.4946
2.4831
2.4773
2.4661
2.4602
2.4435
2.4273
1.0571
1.0396
1.0062
0.9888
0.9768
0.9600
0.8711
0.8541
BRUKER
O
O
S
O
O
OH
NAME zgp127
EXPNO 1
PROCNO 1
Date_ 20180718
Time 16.25
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 3
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 142.88
DW 62.400 usec
DE 6.50 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
0.98
1.00
1.07
1.01
1.00
1.00
2.06
1.04
3.00
3.17

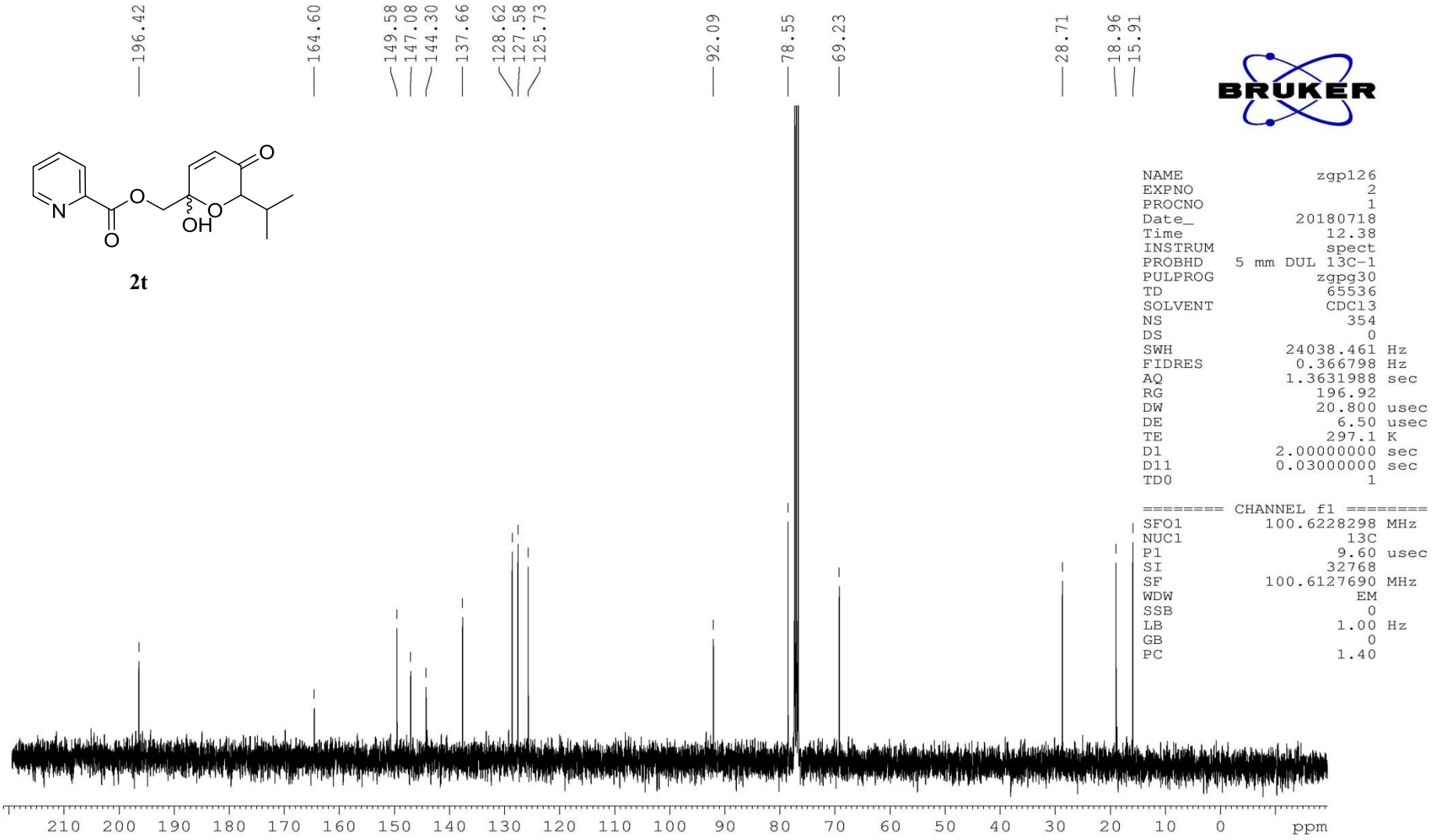
**2s**

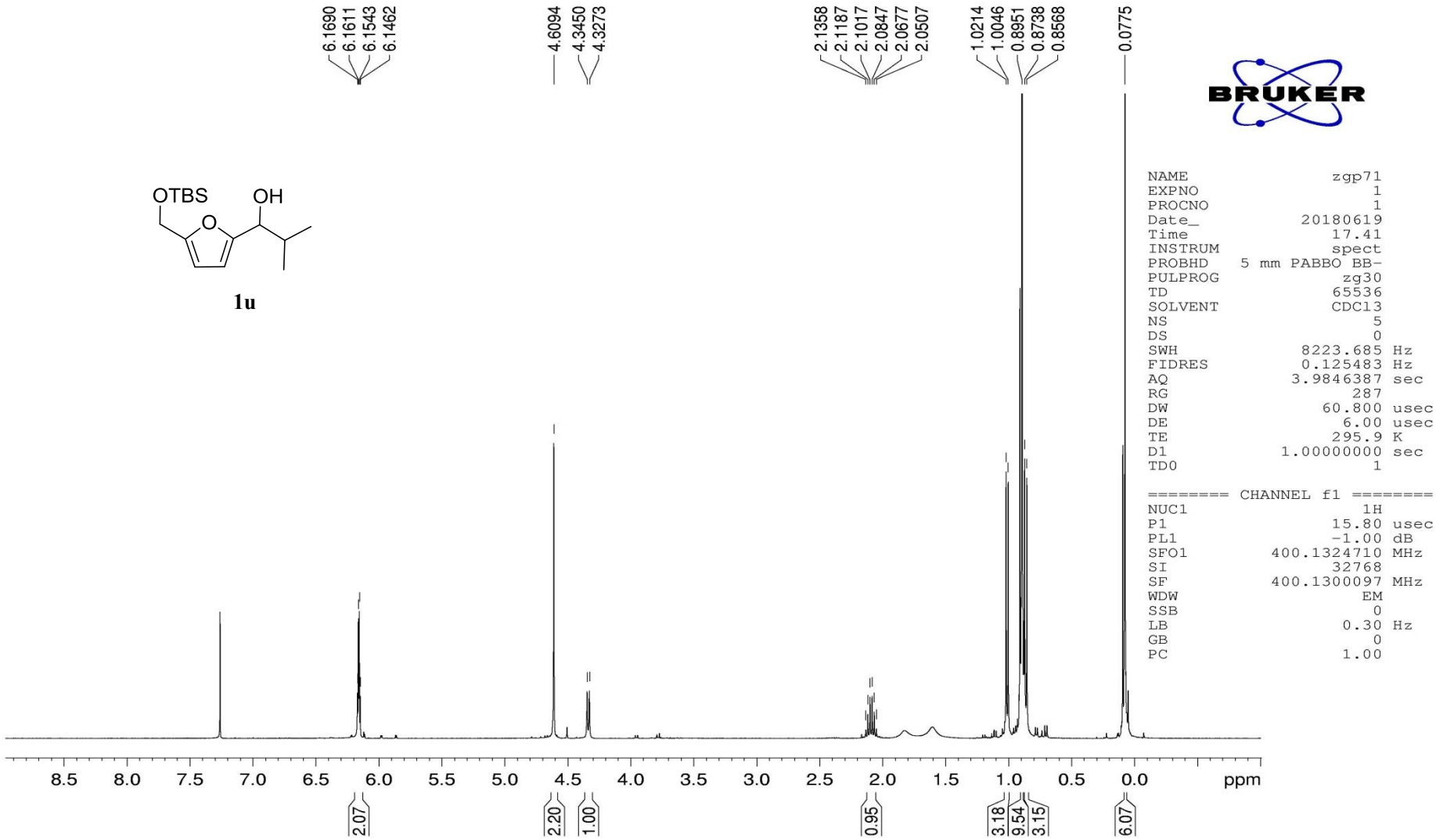

196.30
162.19
145.62
144.02
134.36
133.40
132.50
129.27
128.69
128.04
93.49
92.30
82.82
78.64
68.38
28.76
18.92
18.80
16.94
15.87
BRUKER
S
O
O
O
OH
O
2s
NAME zgp127
EXPNO 2
PROCNO 1
Date_ 20180718
Time 16.27
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 160
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

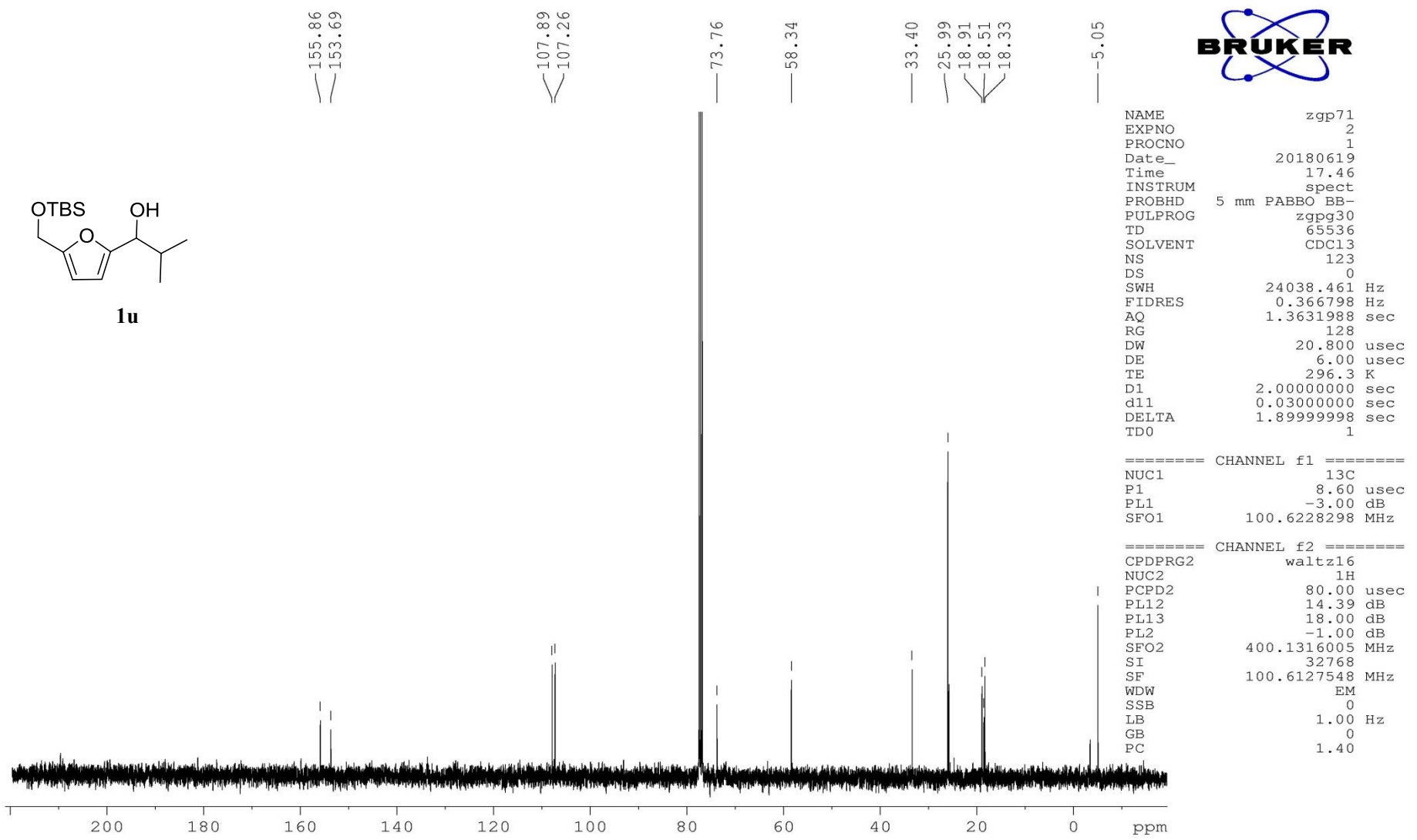

8.7445
8.1232
8.1037
7.8470
7.8277
7.8084
7.4828
7.4694
7.4524
6.4638
6.4574
6.2096
6.2031
5.3609
4.3760
4.3589
2.1207
2.1037
2.0869
2.0701
1.0059
0.9891
0.8744
0.8575
O
O
OH
N
1t
BRUKER
NAME zgp124
EXPNO 1
PROCNO 1
Date_ 20180717
Time 21.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 5
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 256
DW 60.800 usec
DE 6.00 usec
TE 294.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0 ppm
1.00
0.98
1.01
1.00
0.98
1.02
1.97
1.04
1.15
3.08
3.13

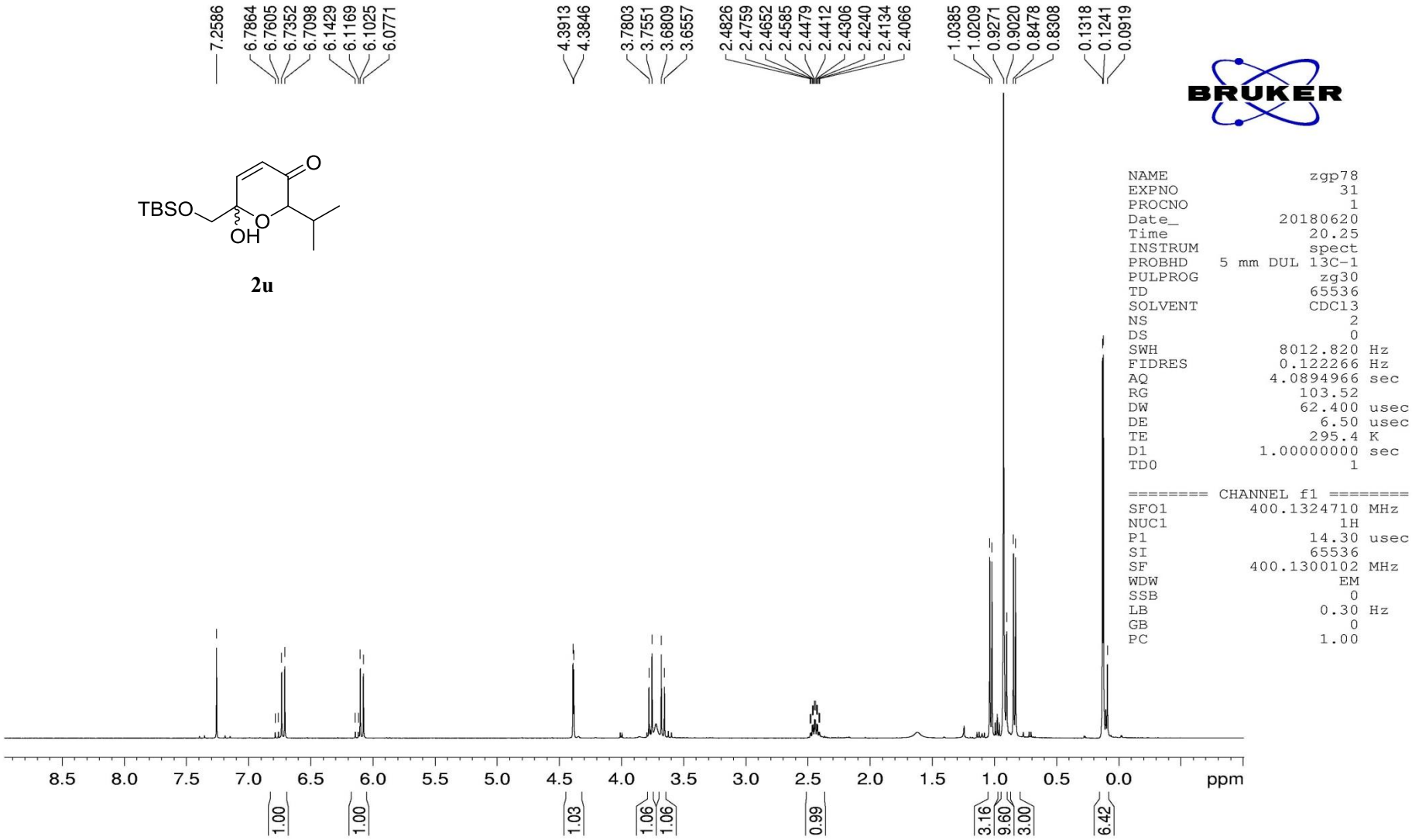

164.16
156.70
149.28
147.47
147.16
136.40
126.39
124.71
111.48
106.81
72.79
58.71
32.58
18.14
17.40
BRUKER
O
O
O
OH
N
1t
NAME zgp124
EXPNO 2
PROCNO 1
Date_ 20180717
Time 21.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 74
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 114
DW 20.800 usec
DE 6.00 usec
TE 294.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 14.39 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6128330 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

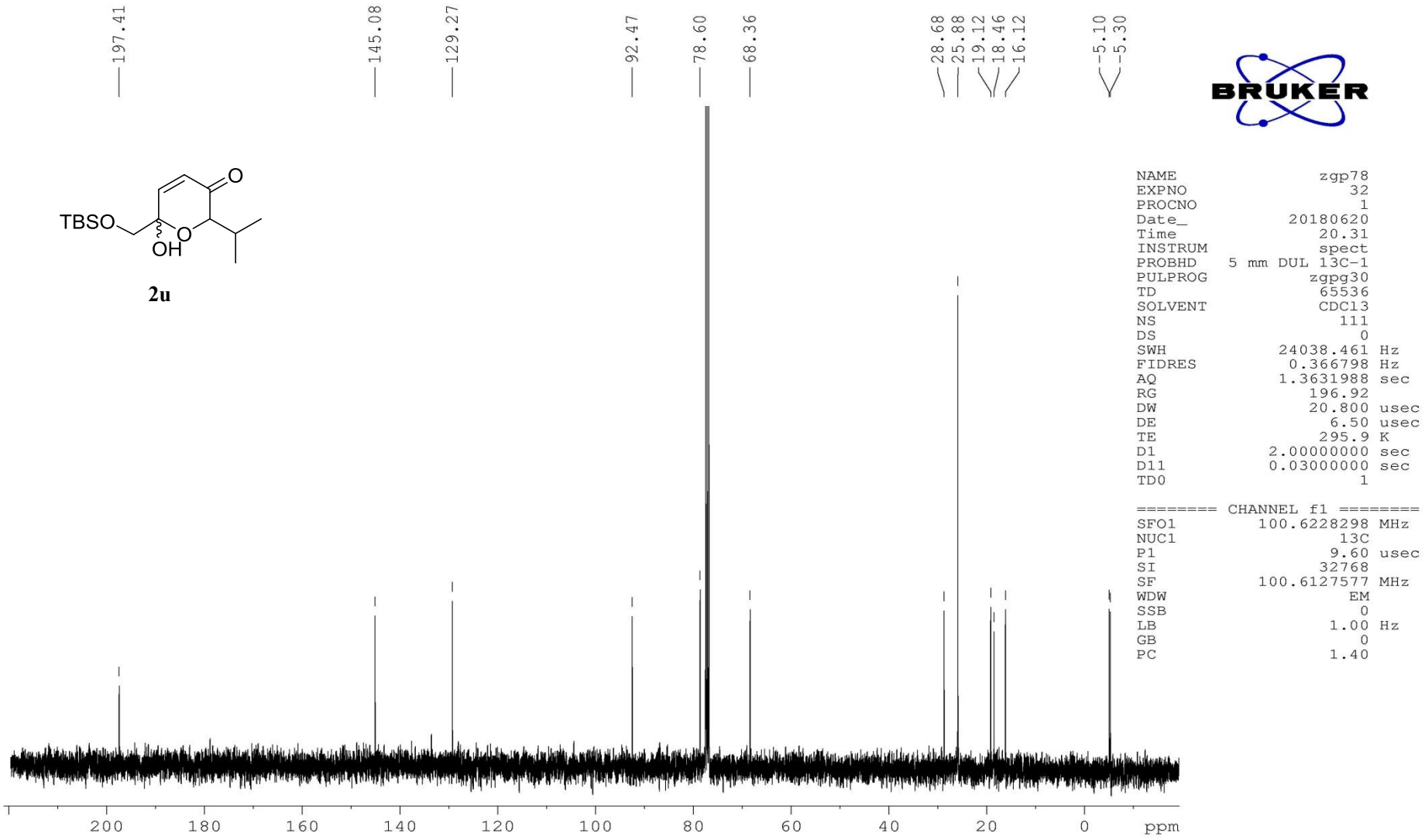

8.8069
8.7960
8.2069
8.1873
8.1582
7.9492
7.9299
7.9109
7.5950
7.5833
7.5763
7.5648
7.2845
7.0227
6.9971
6.9677
6.9418
6.2125
6.1864
6.1684
6.1428
4.9477
4.9190
4.7889
4.7605
4.4562
4.4510
4.4442
4.4281
4.3497
4.3210
2.4837
2.4718
2.4665
2.4546
2.4490
2.4378
2.4319
2.4208
0.9966
0.9791
0.9557
0.9406
0.9258
0.8385
0.8215
BRUKER
O
N
O
O
OH
O
2t
NAME zgp126
EXPNO 1
PROCNO 1
Date_ 20180718
Time 12.33
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 164.33
DW 62.400 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0 ppm
0.92
0.98
0.96
0.97
0.91
1.00
1.05
2.06
1.08
3.13
2.67

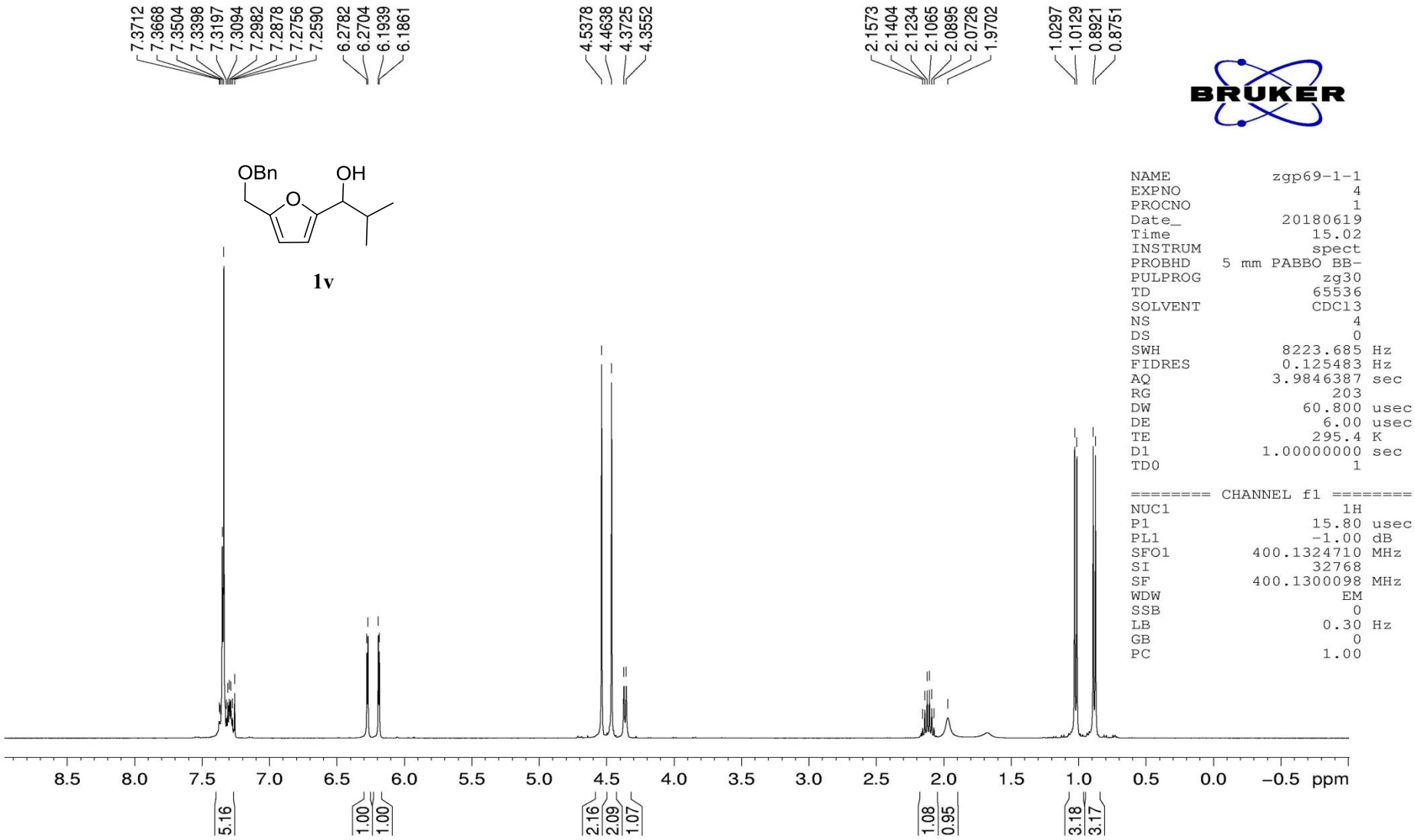

196.42
164.60
149.58
147.08
144.30
137.66
128.62
127.58
125.73
92.09
78.55
69.23
28.71
18.96
15.91
BRUKER
O
N
O
O
OH
O
2t
NAME zgp126
EXPNO 2
PROCNO 1
Date_ 20180718
Time 12.38
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 354
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

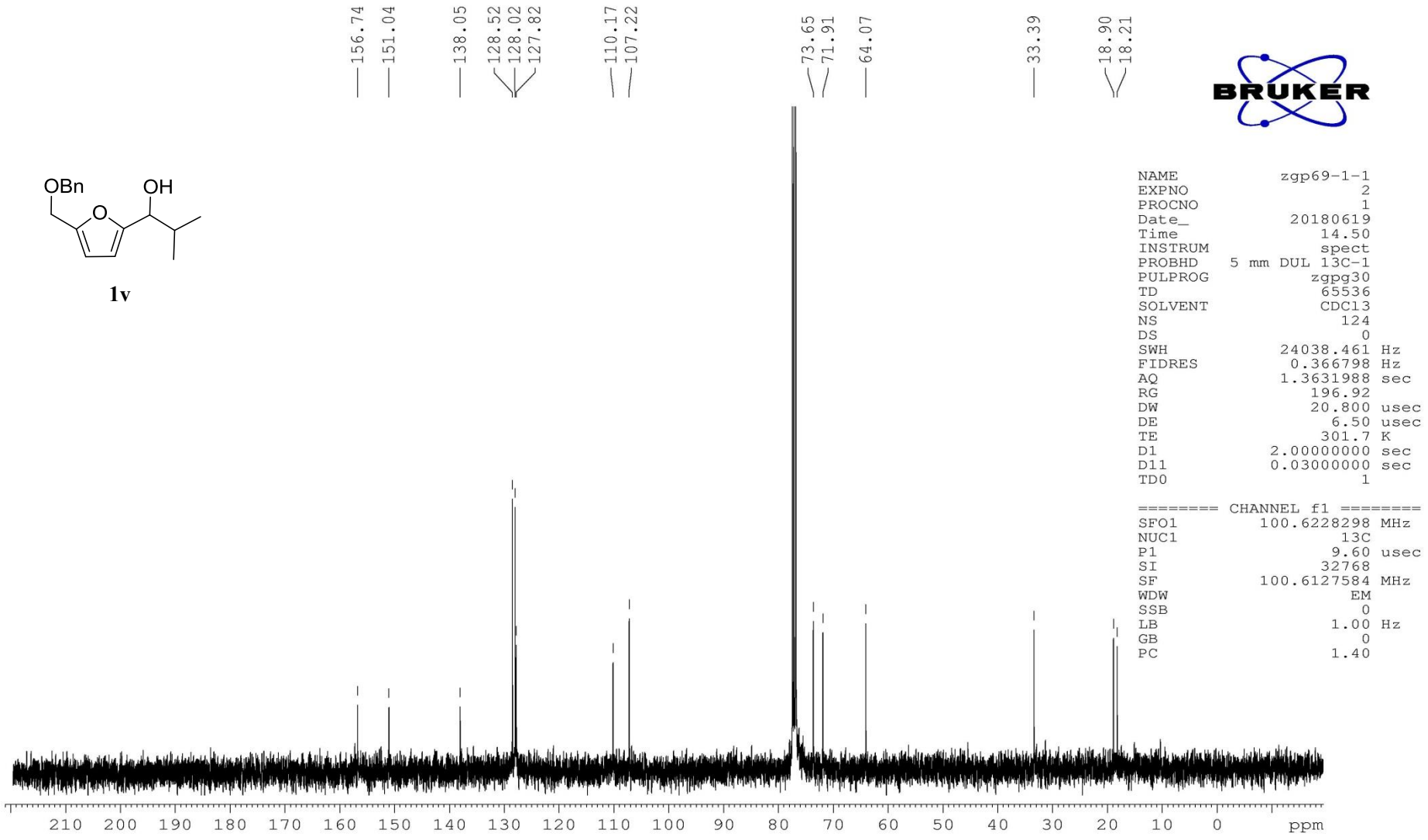

6.1690
6.1611
6.1543
6.1462
4.6094
4.3450
4.3273
2.1358
2.1187
2.1017
2.0847
2.0677
2.0507
1.0214
1.0046
0.8951
0.8738
0.8568
0.0775
OTBS
OH
O
1u
BRUKER
NAME zgp71
EXPNO 1
PROCNO 1
Date_ 20180619
Time 17.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 5
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 287
DW 60.800 usec
DE 6.00 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.07
2.20
1.00
0.95
3.18
9.54
3.15
6.07

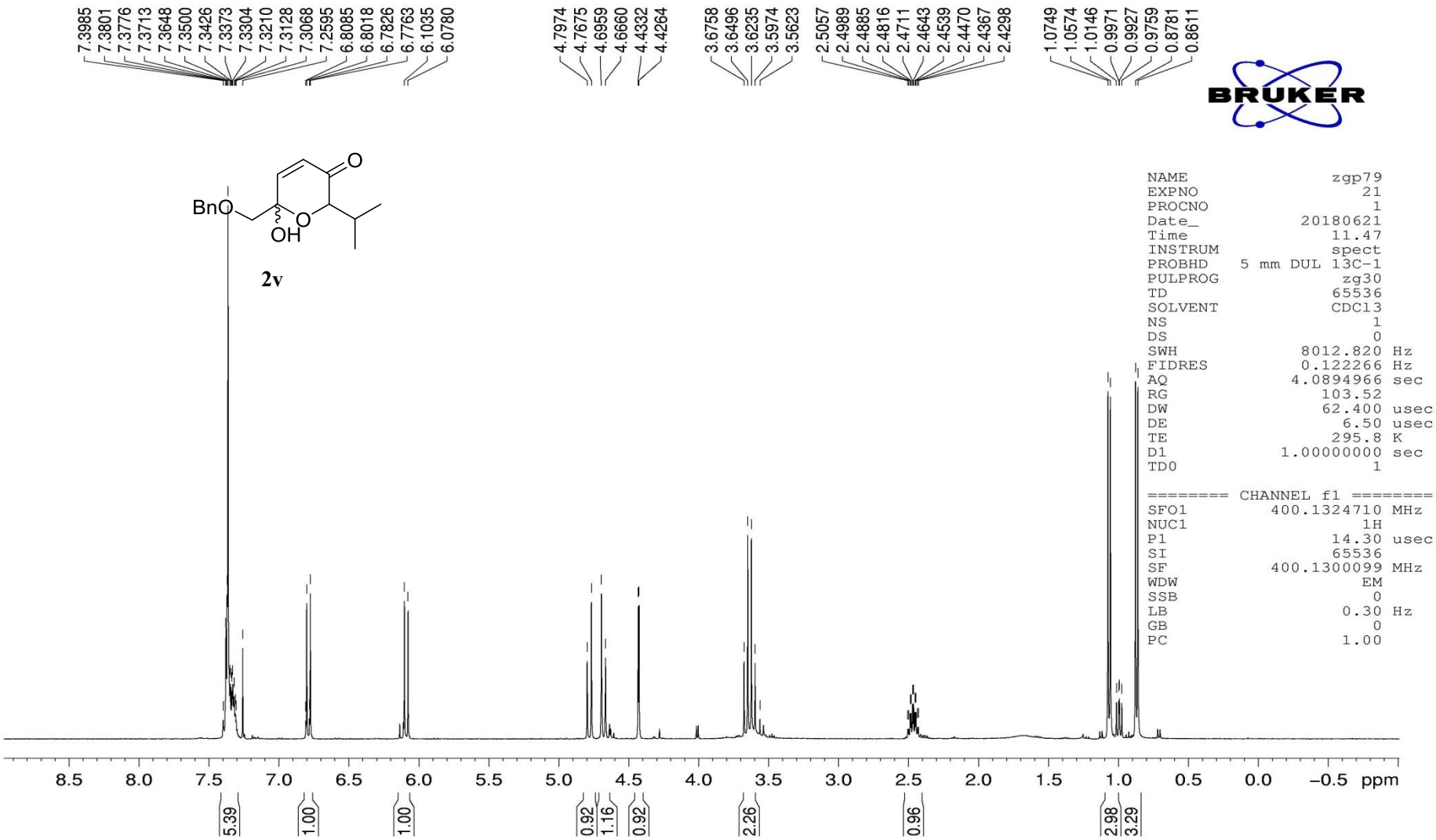

155.86
153.69
107.89
107.26
73.76
58.34
33.40
25.99
18.91
18.51
18.33
-5.05
OTBS
OH
O
1u
NAME zgp71
EXPNO 2
PROCNO 1
Date_ 20180619
Time 17.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 123
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 128
DW 20.800 usec
DE 6.00 usec
TE 296.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 14.39 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127548 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

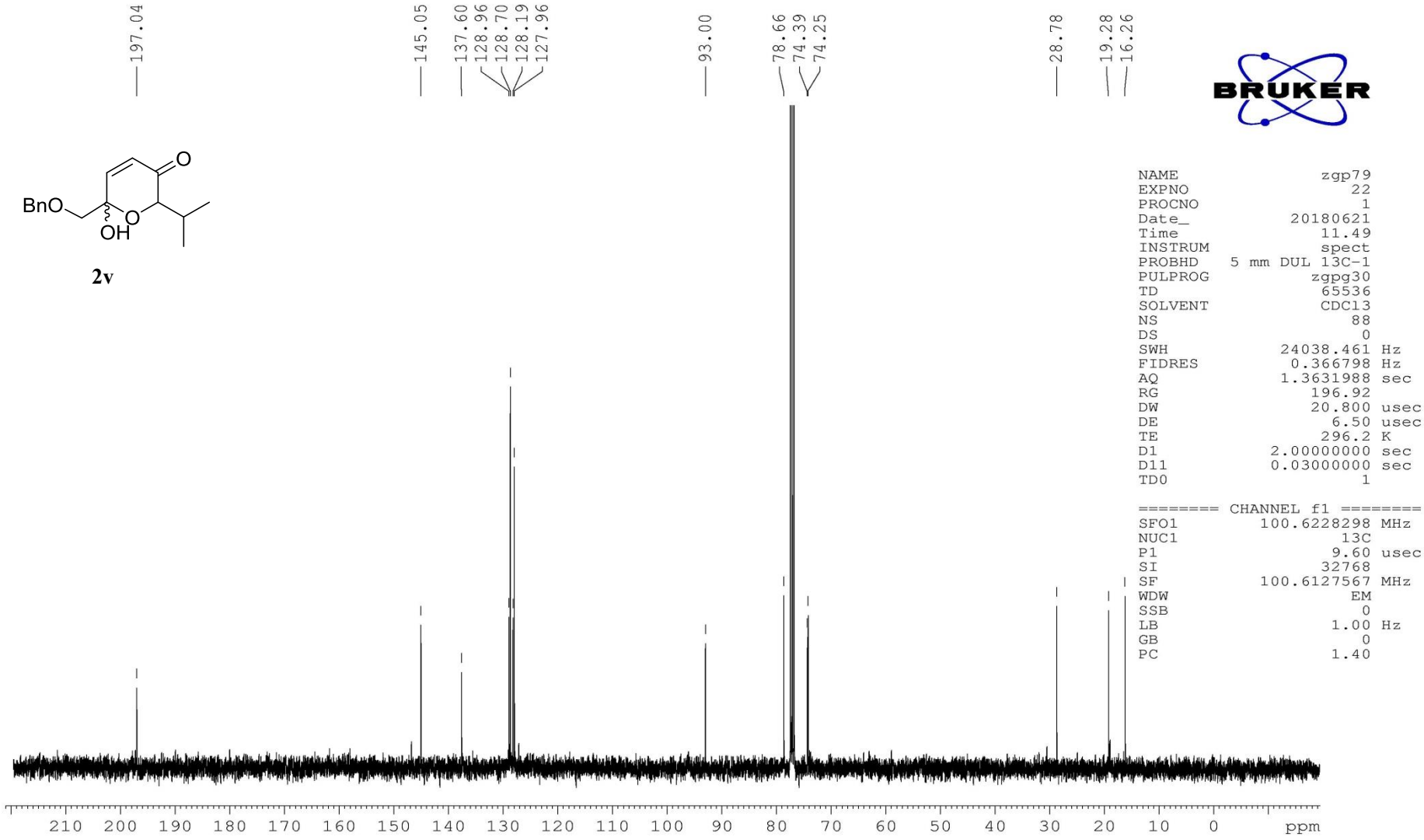

7.2586
6.7864
6.7605
6.7352
6.7098
6.1429
6.1169
6.1025
6.0771
4.3913
4.3846
3.7803
3.7551
3.6809
3.6557
2.4826
2.4759
2.4652
2.4585
2.4479
2.4412
2.4306
2.4240
2.4134
2.4066
1.0385
1.0209
0.9271
0.9020
0.8478
0.8308
0.1318
0.1241
0.0919
TBSO
O
O
OH
2u
BRUKER
NAME zgp78
EXPNO 31
PROCNO 1
Date_ 20180620
Time 20.25
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 2
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 103.52
DW 62.400 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
1.00
1.03
1.06
1.06
0.99
3.16
9.60
3.00
6.42

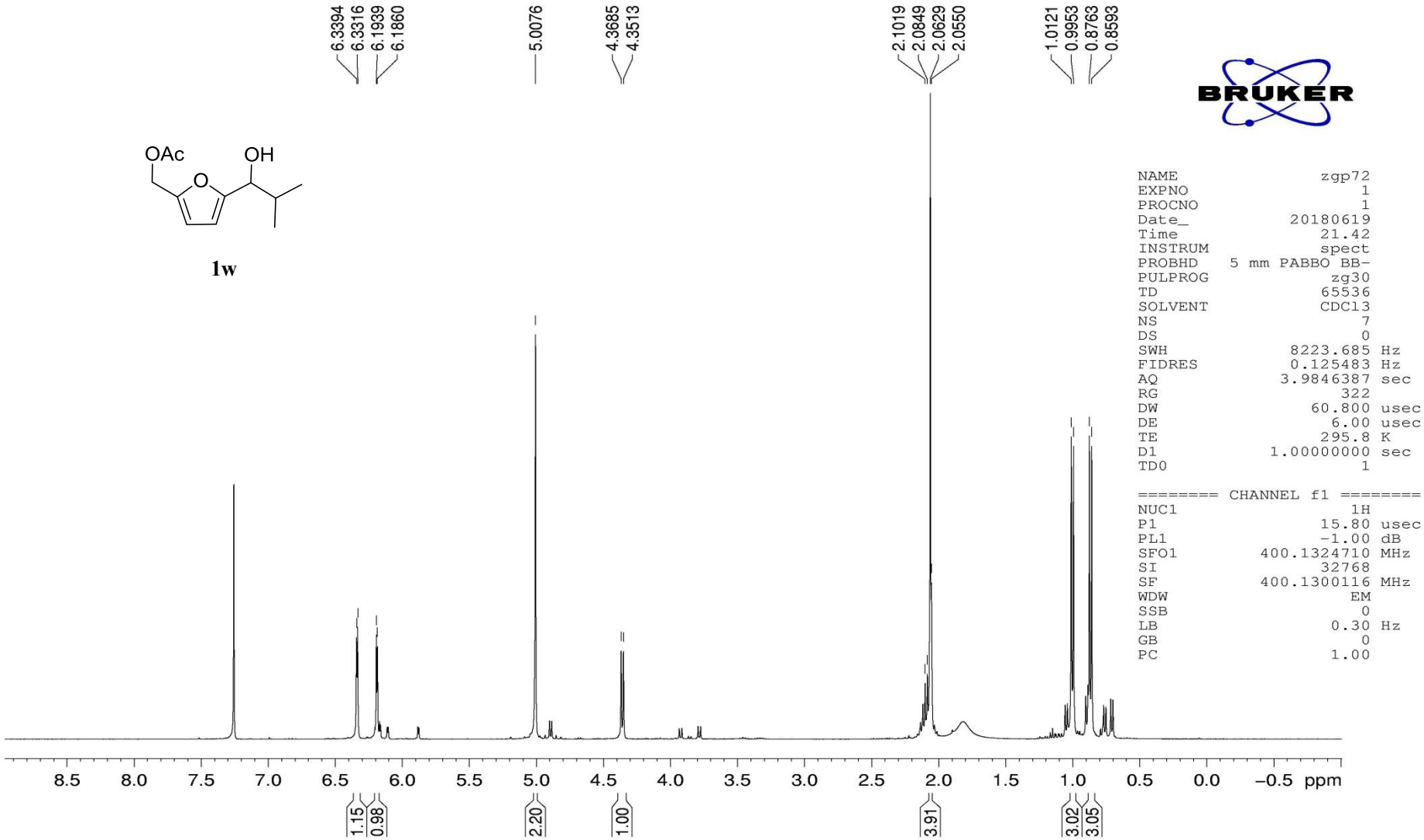

197.41
145.08
129.27
92.47
78.60
68.36
28.68
25.88
19.12
18.46
16.12
5.10
5.30
TBSO
O
OH
O
2u
BRUKER
NAME zgp78
EXPNO 32
PROCNO 1
Date_ 20180620
Time 20.31
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 111
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127577 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

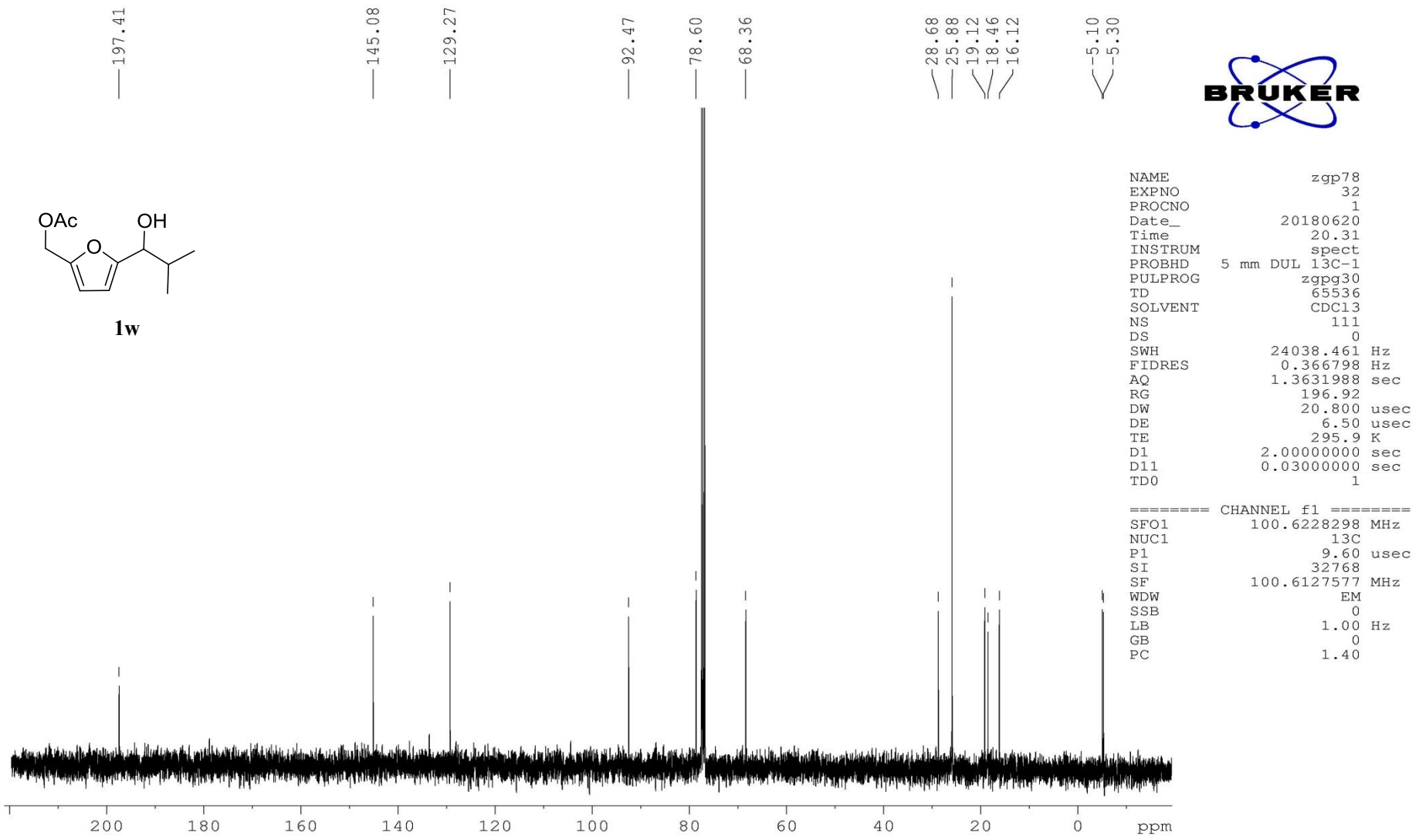

7.3712
7.3668
7.3504
7.3398
7.3197
7.3094
7.2982
7.2878
7.2756
7.2590
6.2782
6.2704
6.1939
6.1861
4.5378
4.4638
4.3725
4.3552
2.1573
2.1404
2.1234
2.1065
2.0895
2.0726
1.9702
1.0297
1.0129
0.8921
0.8751
BRUKER
OBn
OH
O
1v
NAME zgp69-1-1
EXPNO 4
PROCNO 1
Date_ 20180619
Time 15.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.00 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
5.16
1.00
1.00
2.16
2.09
1.07
1.08
0.95
3.18
3.17

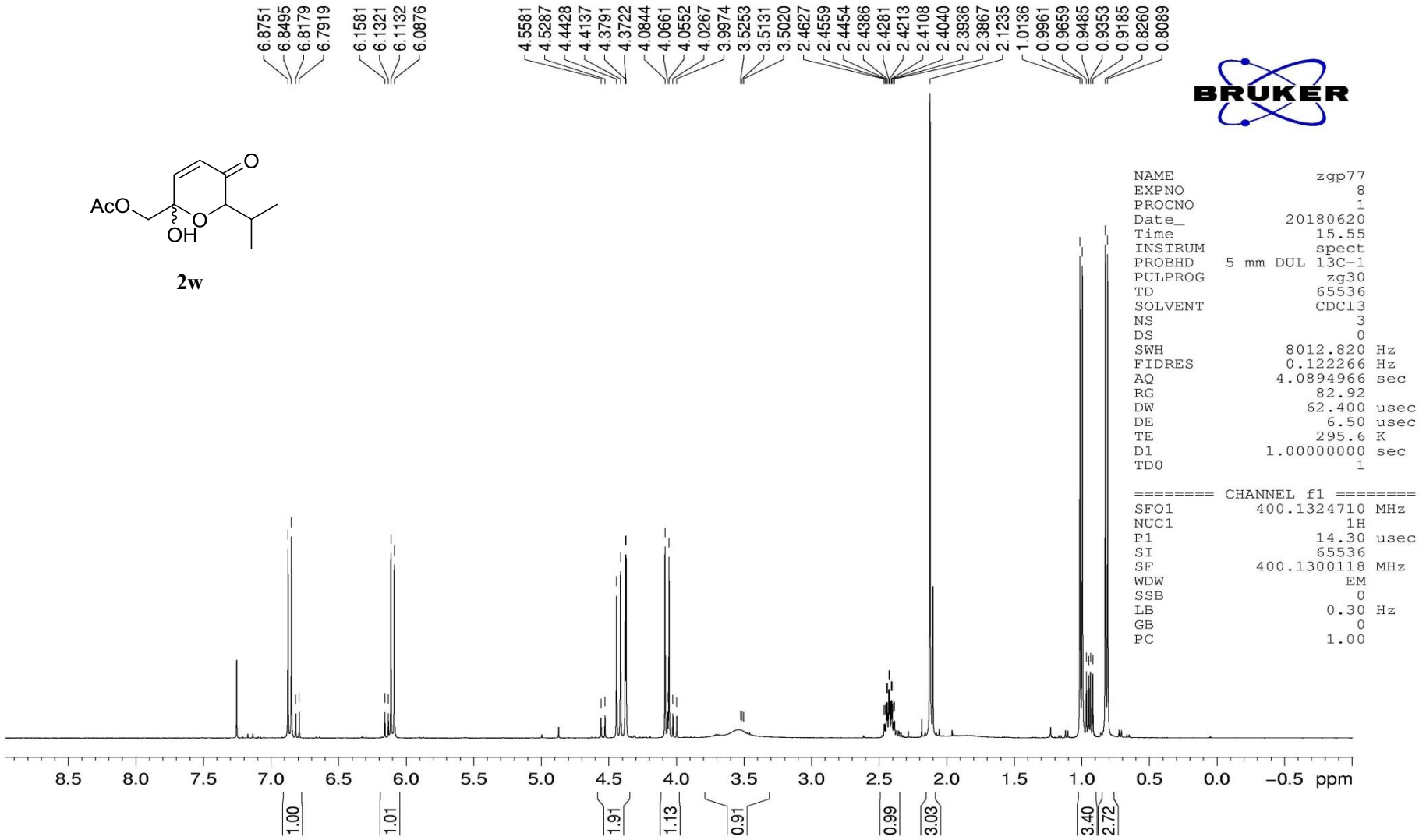

156.74
151.04
138.05
128.52
128.02
127.82
110.17
107.22
73.65
71.91
64.07
33.39
18.90
18.21
OBn
OH
O
1v
NAME zgp69-1-1
EXPNO 2
PROCNO 1
Date_ 20180619
Time 14.50
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 124
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 301.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127584 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

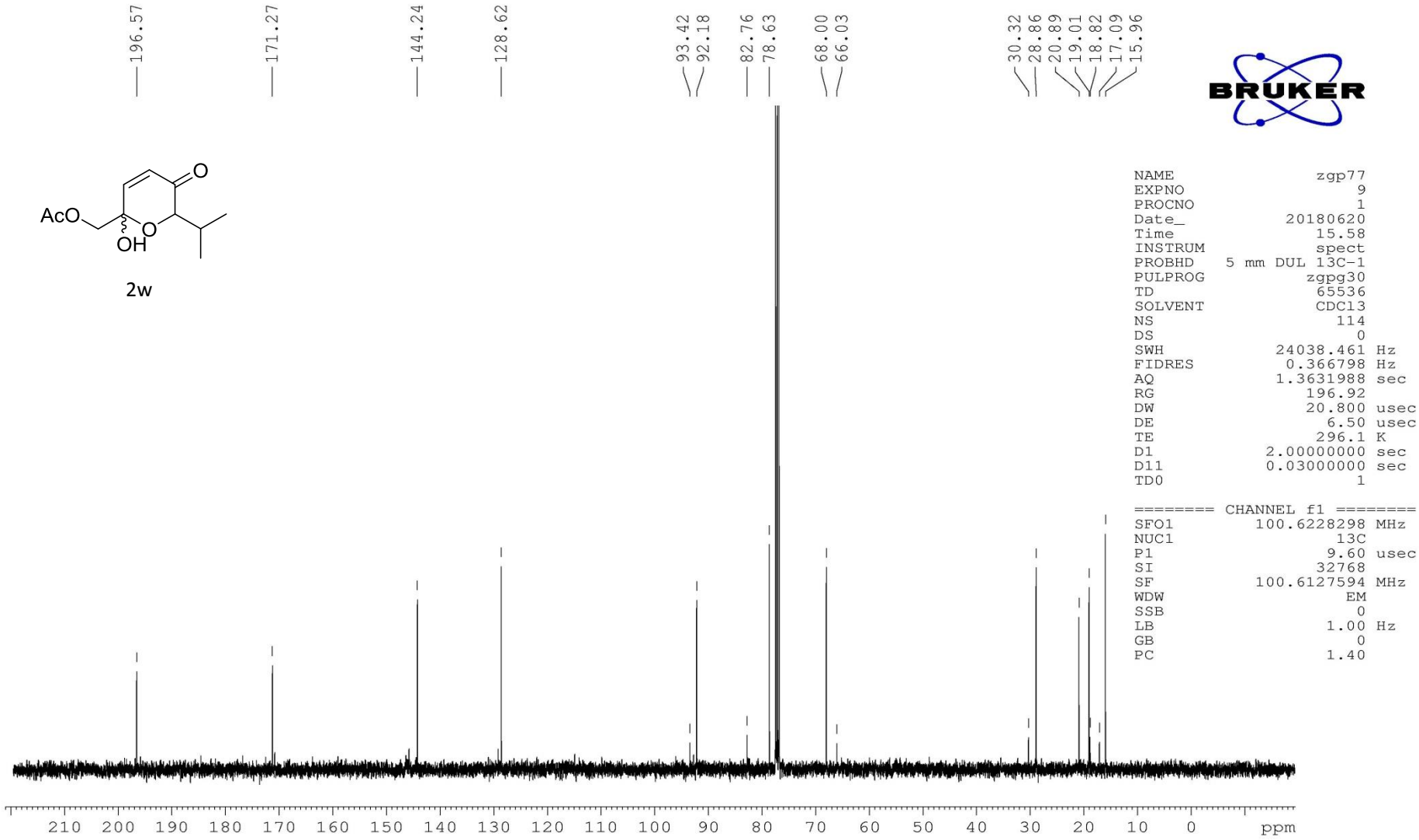

7.3985
7.3801
7.3776
7.3713
7.3648
7.3500
7.3426
7.3373
7.3304
7.3210
7.3128
7.3068
7.2595
6.8085
6.8018
6.7826
6.7763
6.1035
6.0780
4.7974
4.7675
4.6959
4.6660
4.4332
4.4264
3.6758
3.6496
3.6235
3.5974
3.5623
2.5057
2.4989
2.4885
2.4816
2.4711
2.4643
2.4539
2.4470
2.4367
2.4298
1.0749
1.0574
1.0146
0.9971
0.9927
0.9759
0.8781
0.8611
BRUKER
BnO
O
OH
O
2v
NAME zgp79
EXPNO 21
PROCNO 1
Date_ 20180621
Time 11.47
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 103.52
DW 62.400 usec
DE 6.50 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
5.39
1.00
1.00
0.92
1.16
0.92
2.26
0.96
2.98
3.29

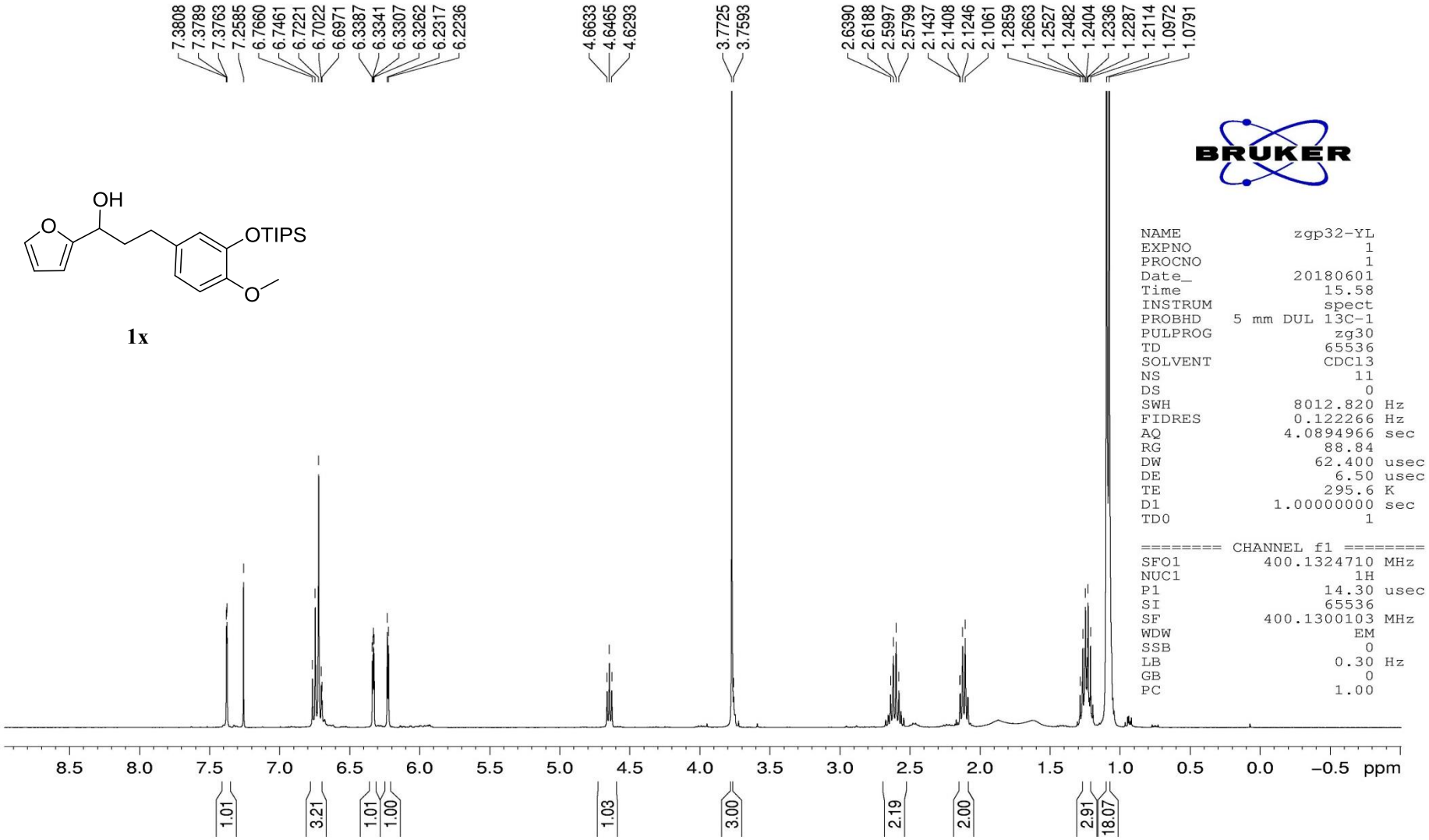

197.04
145.05
137.60
128.96
128.70
128.19
127.96
93.00
78.66
74.39
74.25
28.78
19.28
16.26
BRUKER
BnO
O
OH
O
2v
NAME zgp79
EXPNO 22
PROCNO 1
Date_ 20180621
Time 11.49
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 88
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127567 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

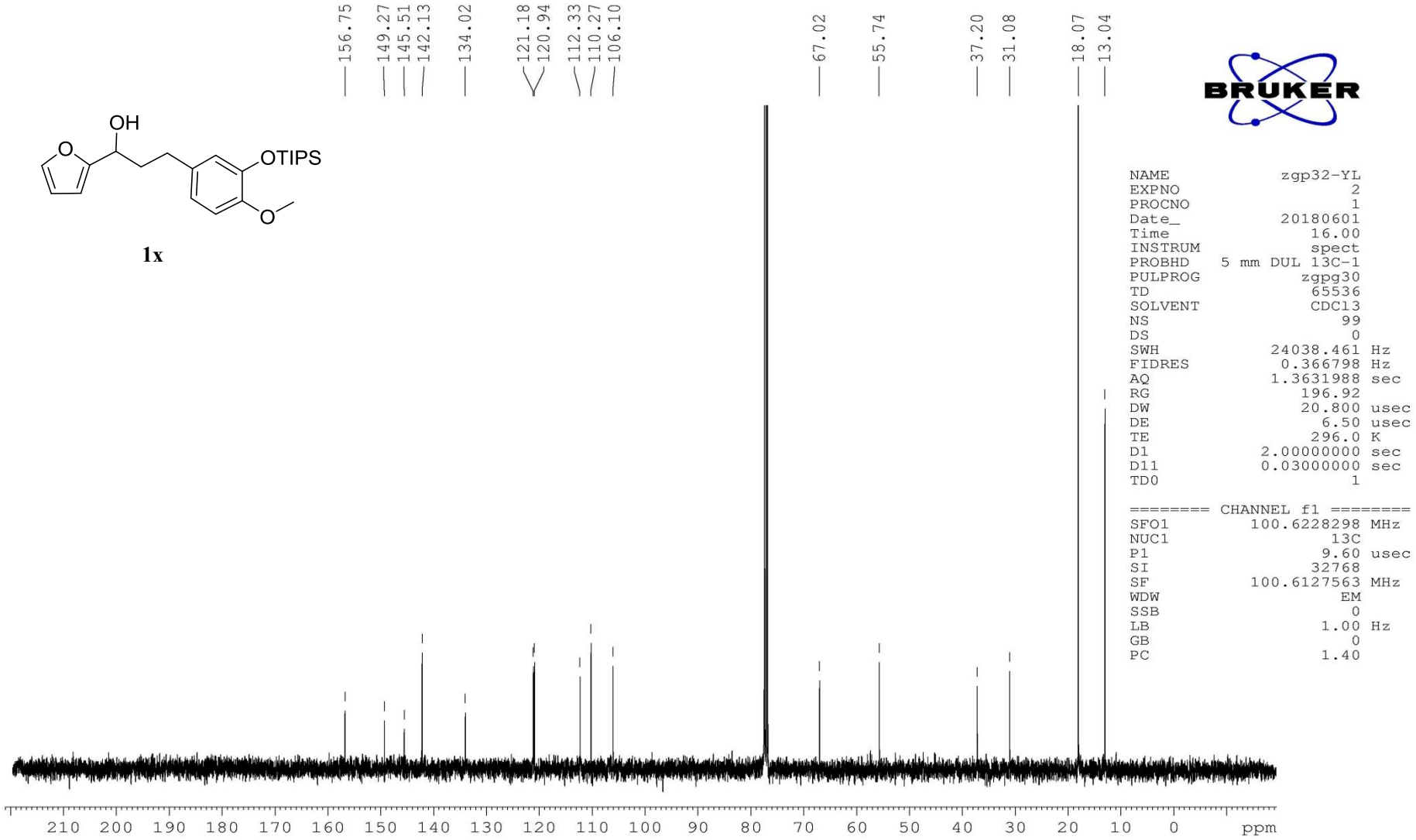

6.3394
6.3316
6.1939
6.1860
5.0076
4.3685
4.3513
2.1019
2.0849
2.0629
2.0550
1.0121
0.9953
0.8763
0.8593
OAc
OH
O
1w
BRUKER
NAME zgp72
EXPNO 1
PROCNO 1
Date_ 20180619
Time 21.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 7
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 322
DW 60.800 usec
DE 6.00 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300116 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.15
0.98
2.20
1.00
3.91
3.02
3.05

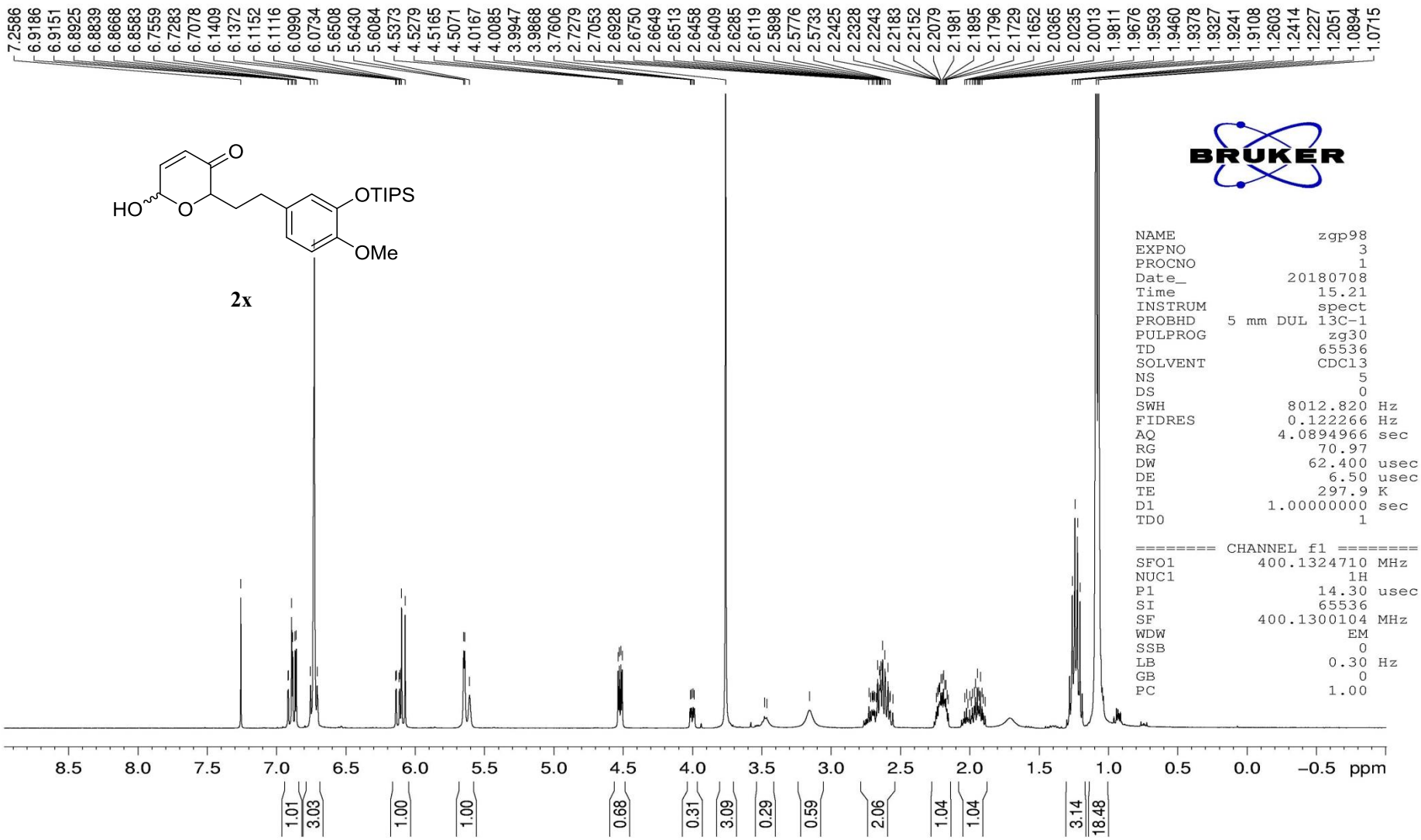

197.41
145.08
129.27
92.47
78.60
68.36
28.68
25.88
19.12
18.46
16.12
5.10
5.30
OAc
OH
O
1w
NAME zgp78
EXPNO 32
PROCNO 1
Date_ 20180620
Time 20.31
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 111
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127577 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

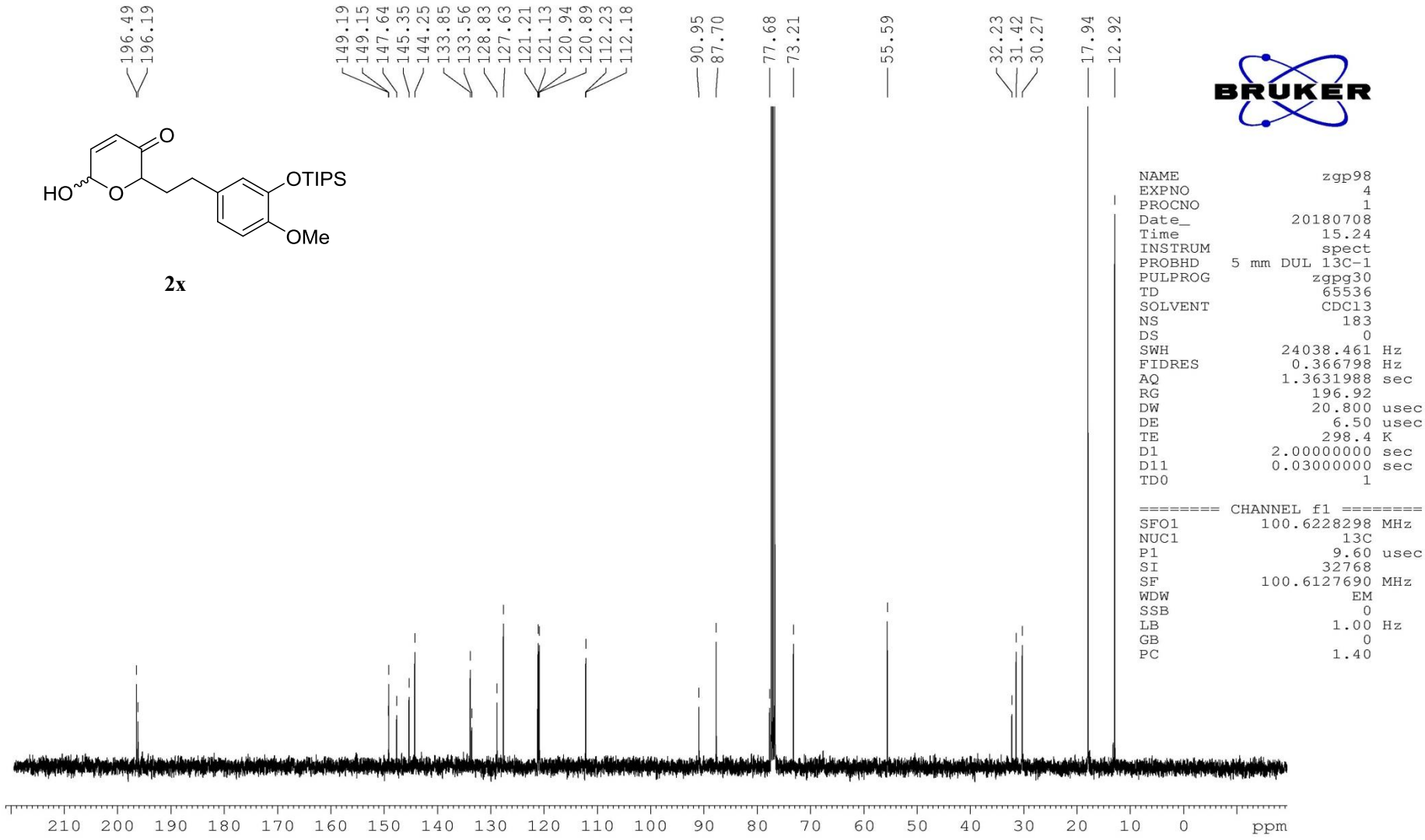

6.8751
6.8495
6.8179
6.7919
6.1581
6.1321
6.1132
6.0876
4.5581
4.5287
4.4428
4.4137
4.3791
4.3722
4.0844
4.0661
4.0552
4.0267
3.9974
3.5253
3.5131
3.5020
2.4627
2.4559
2.4454
2.4386
2.4281
2.4213
2.4108
2.4040
2.3936
2.3867
2.1235
1.0136
0.9961
0.9659
0.9485
0.9353
0.9185
0.8260
0.8089
AcO
O
OH
O
2w
BRUKER
NAME zgp77
EXPNO 8
PROCNO 1
Date_ 20180620
Time 15.55
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 82.92
DW 62.400 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5 ppm
1.00
1.01
1.91
1.13
0.91
0.99
3.03
3.40
2.72

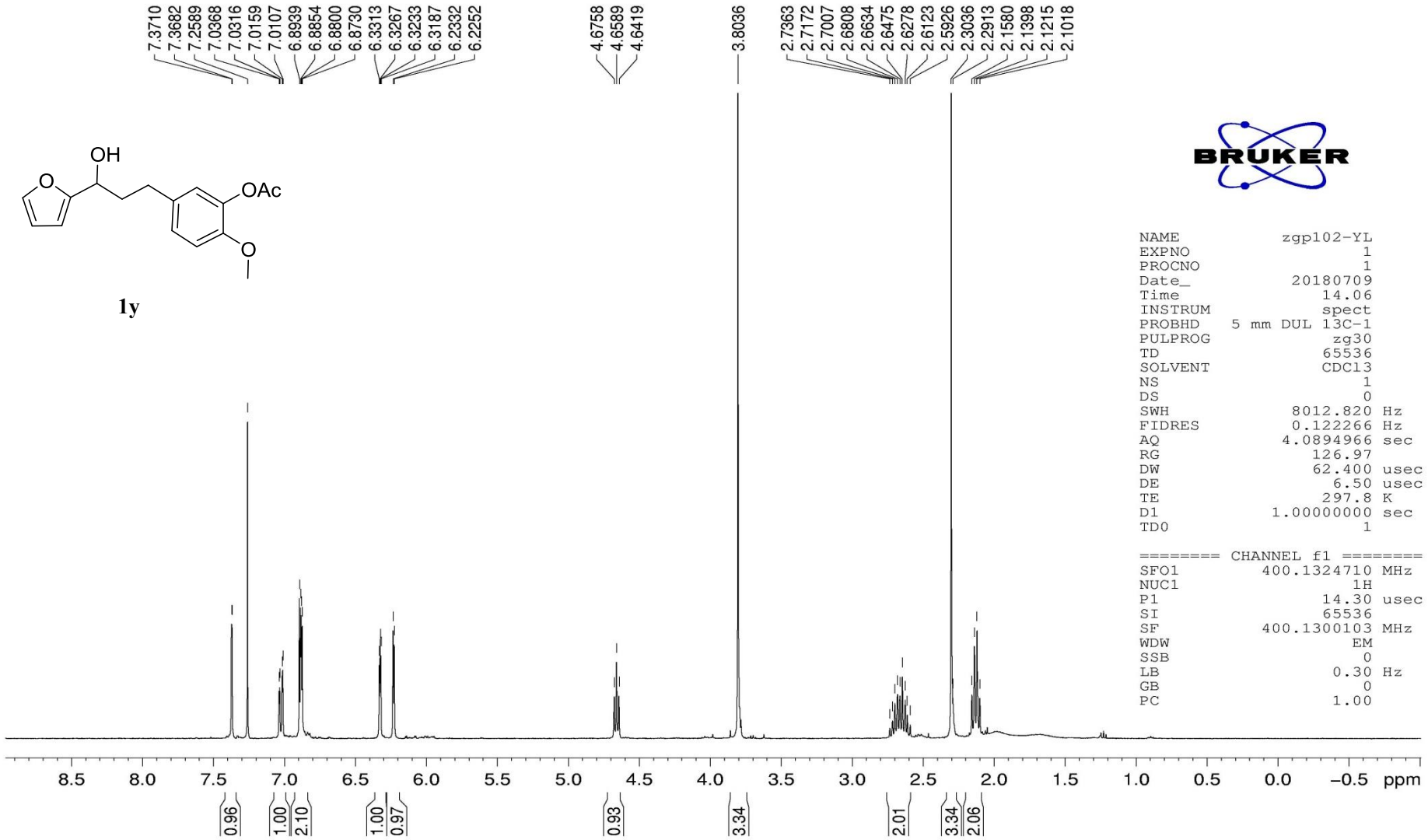

196.57
171.27
144.24
128.62
93.42
92.18
82.76
78.63
68.00
66.03
30.32
28.86
20.89
19.01
18.82
17.09
15.96
BRUKER
AcO
O
OH
O
2w
NAME zgp77
EXPNO 9
PROCNO 1
Date_ 20180620
Time 15.58
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 114
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127594 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

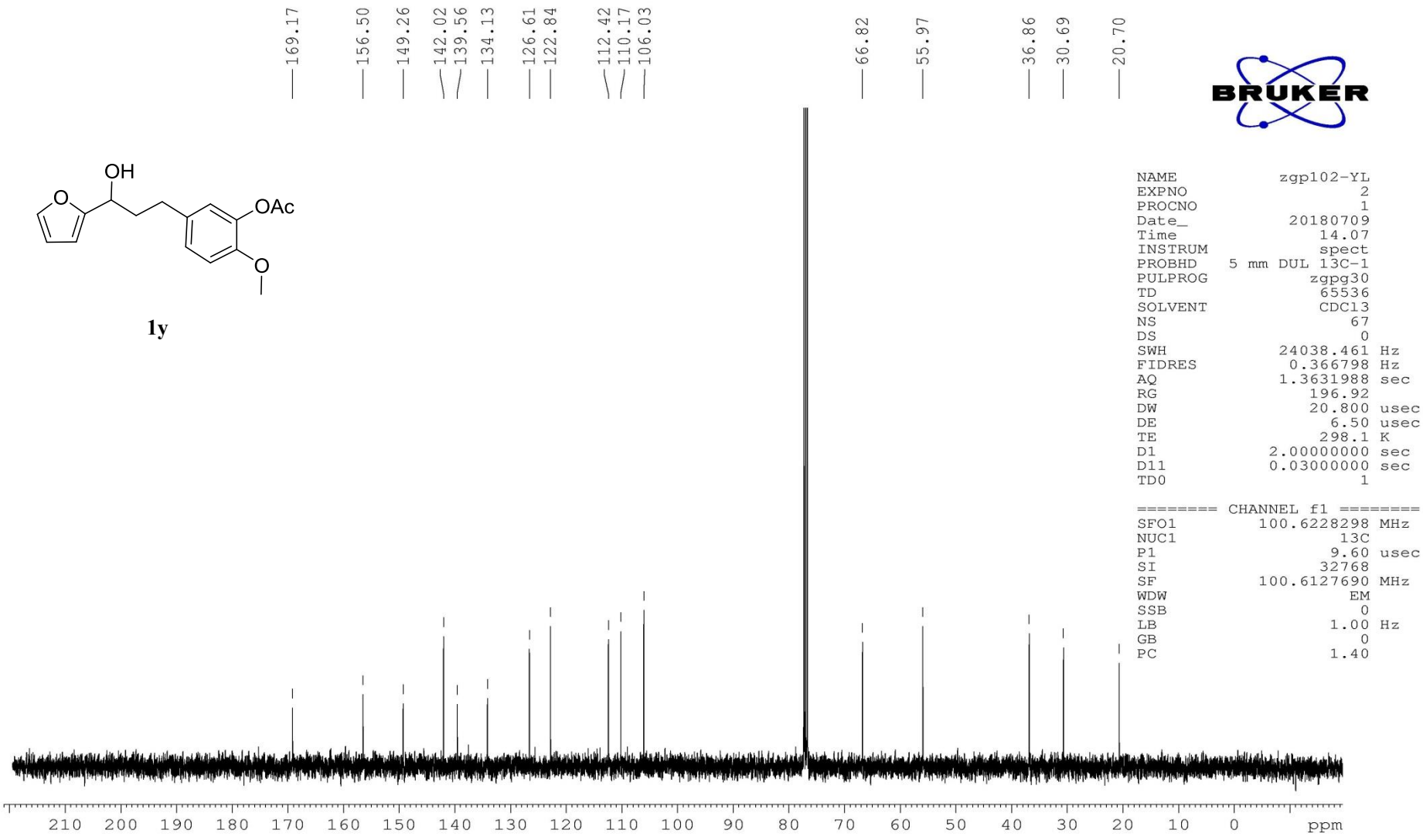

7.3808
7.3789
7.3763
7.2585
6.7660
6.7461
6.7221
6.7022
6.6971
6.3387
6.3341
6.3307
6.3262
6.2317
6.2236
4.6633
4.6465
4.6293
3.7725
3.7593
2.6390
2.6188
2.5997
2.5799
2.1437
2.1408
2.1246
2.1061
1.2859
1.2663
1.2527
1.2482
1.2404
1.2336
1.2287
1.2114
1.0972
1.0791
OH
O
OTIPS
O
1x
BRUKER
NAME zgp32-YL
EXPNO 1
PROCNO 1
Date_ 20180601
Time 15.58
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 88.84
DW 62.400 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.01
3.21
1.01
1.00
1.03
3.00
2.19
2.00
2.91
18.07

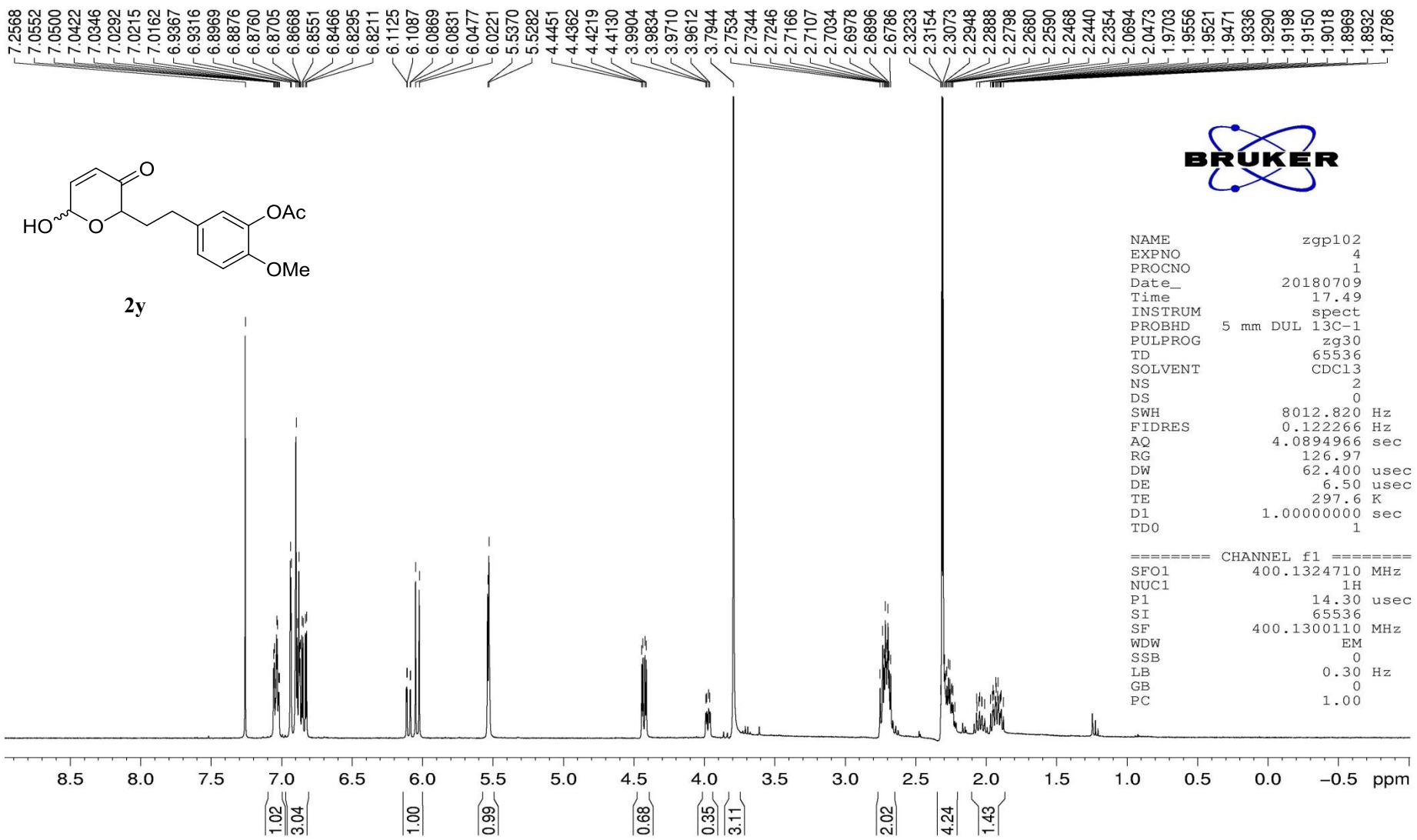

156.75
149.27
145.51
142.13
134.02
121.18
120.94
112.33
110.27
106.10
67.02
55.74
37.20
31.08
18.07
13.04
OH
O
OTIPS
O
1x
BRUKER
NAME zgp32-YL
EXPNO 2
PROCNO 1
Date_ 20180601
Time 16.00
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 99
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127563 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

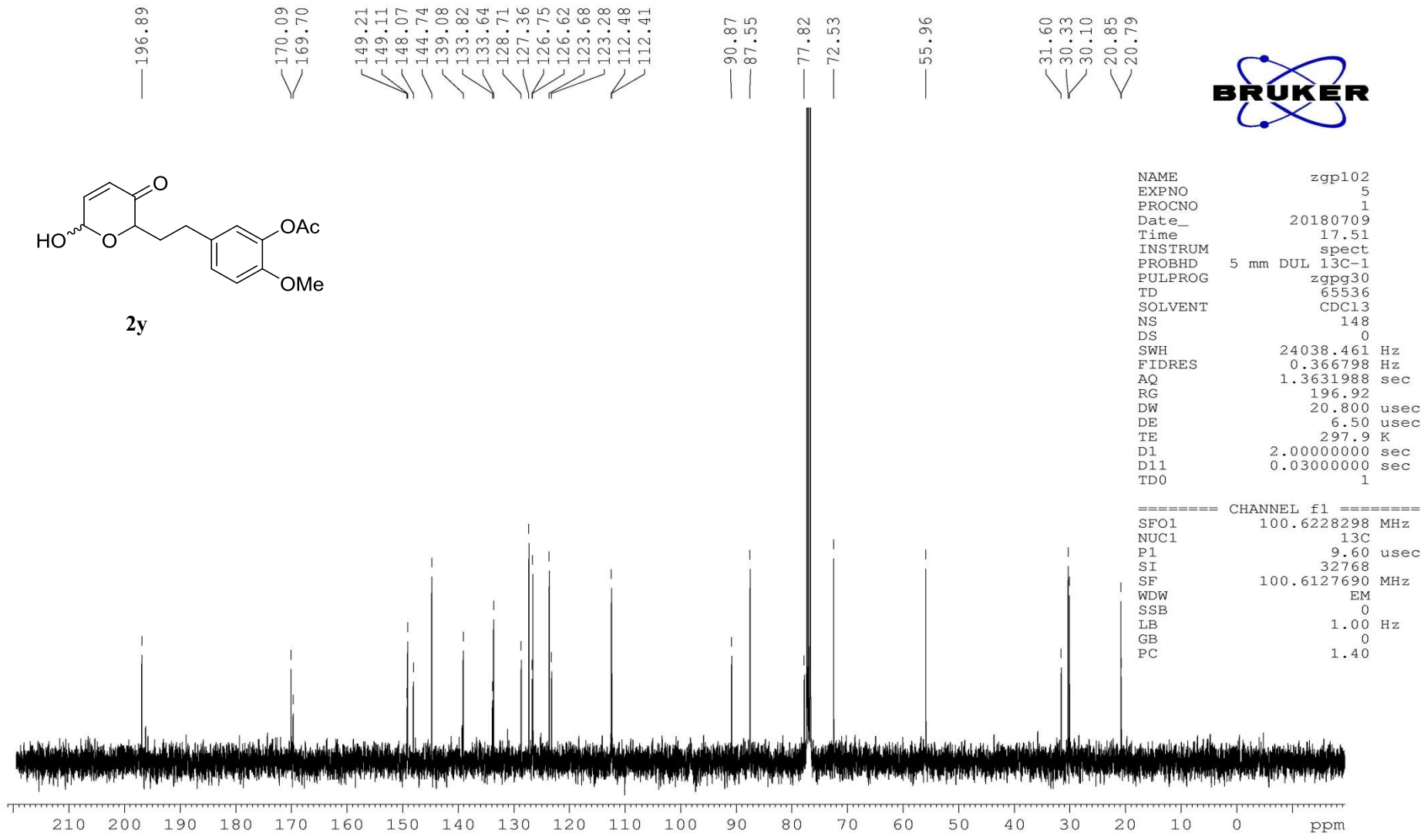

7.2586 6.9186 6.9151 6.8925 6.8839 6.8668 6.8583 6.7559 6.7283 6.7078 6.1409 6.1372 6.1152 6.1116 6.0990 6.0734 5.6508 5.6430 5.6084 4.5373 4.5279 4.5165 4.5071 4.0167 4.0085 3.9947 3.9868 3.7606 2.7279 2.7053 2.6928 2.6750 2.6649 2.6513 2.6458 2.6409 2.6285 2.6119 2.5898 2.5776 2.5733 2.2425 2.2328 2.2243 2.2183 2.2152 2.2079 2.1981 2.1895 2.1796 2.1729 2.1652 2.0365 2.0235 2.0013 1.9811 1.9676 1.9593 1.9460 1.9378 1.9327 1.9241 1.9108 1.2603 1.2414 1.2227 1.2051 1.0894 1.0715
HO
O
O
OTIPS
OMe
2x
BRUKER
NAME zgp98
EXPNO 3
PROCNO 1
Date_ 20180708
Time 15.21
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 5
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 70.97
DW 62.400 usec
DE 6.50 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300104 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.01 3.03 1.00 1.00 0.68 0.31 3.09 0.29 0.59 2.06 1.04 1.04 3.14 18.48

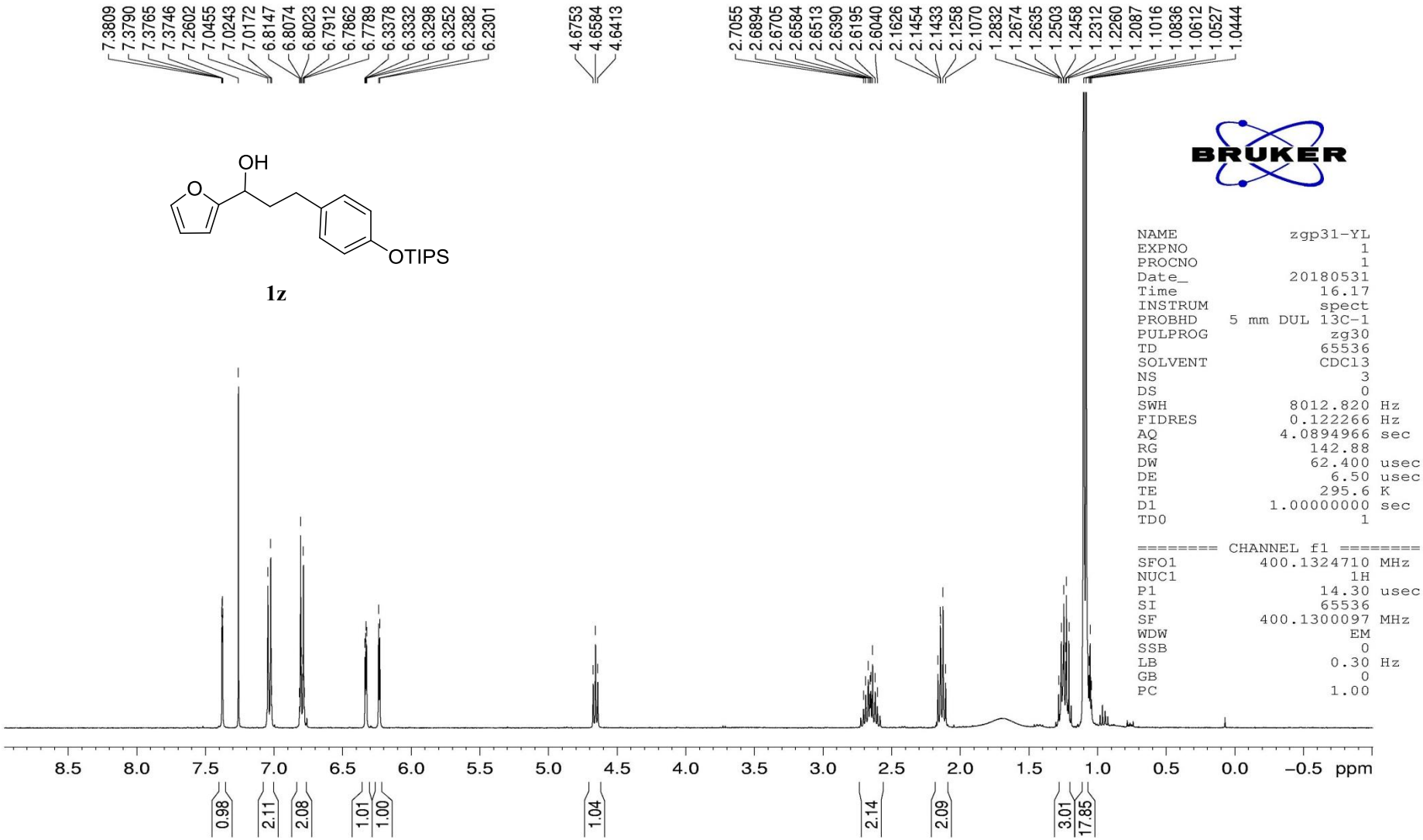

196.49
196.19
149.19
149.15
147.64
145.35
144.25
133.85
133.56
128.83
127.63
121.21
121.13
120.94
120.89
112.23
112.18
90.95
87.70
77.68
73.21
55.59
32.23
31.42
30.27
17.94
12.92
HO
O
O
OTIPS
OMe
2x
BRUKER
NAME zgp98
EXPNO 4
PROCNO 1
Date_ 20180708
Time 15.24
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 183
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.3710
7.3682
7.2589
7.0368
7.0316
7.0159
7.0107
6.8939
6.8854
6.8800
6.8730
6.3313
6.3267
6.3233
6.3187
6.2332
6.2252
4.6758
4.6589
4.6419
3.8036
2.7363
2.7172
2.7007
2.6808
2.6634
2.6475
2.6278
2.6123
2.5926
2.3036
2.2913
2.1580
2.1398
2.1215
2.1018
OH
O
OAc
O
1y
BRUKER
NAME zgp102-YL
EXPNO 1
PROCNO 1
Date_ 20180709
Time 14.06
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 126.97
DW 62.400 usec
DE 6.50 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
0.96
1.00
2.10
1.00
0.97
0.93
3.34
2.01
3.34
2.06

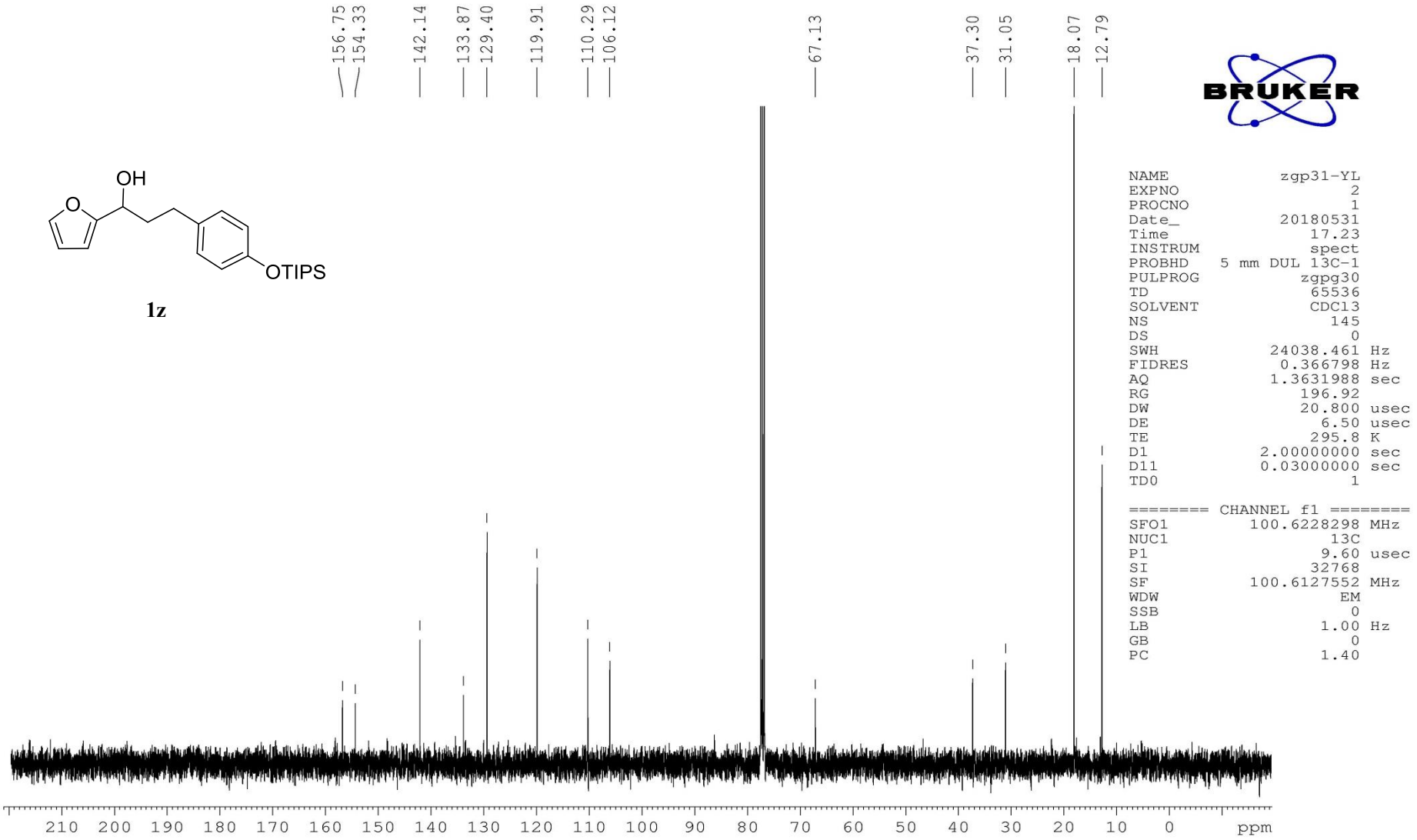

169.17
156.50
149.26
142.02
139.56
134.13
126.61
122.84
112.42
110.17
106.03
66.82
55.97
36.86
30.69
20.70
OH
O
OAc
O
1y
BRUKER
NAME zgp102-YL
EXPNO 2
PROCNO 1
Date_ 20180709
Time 14.07
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 67
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

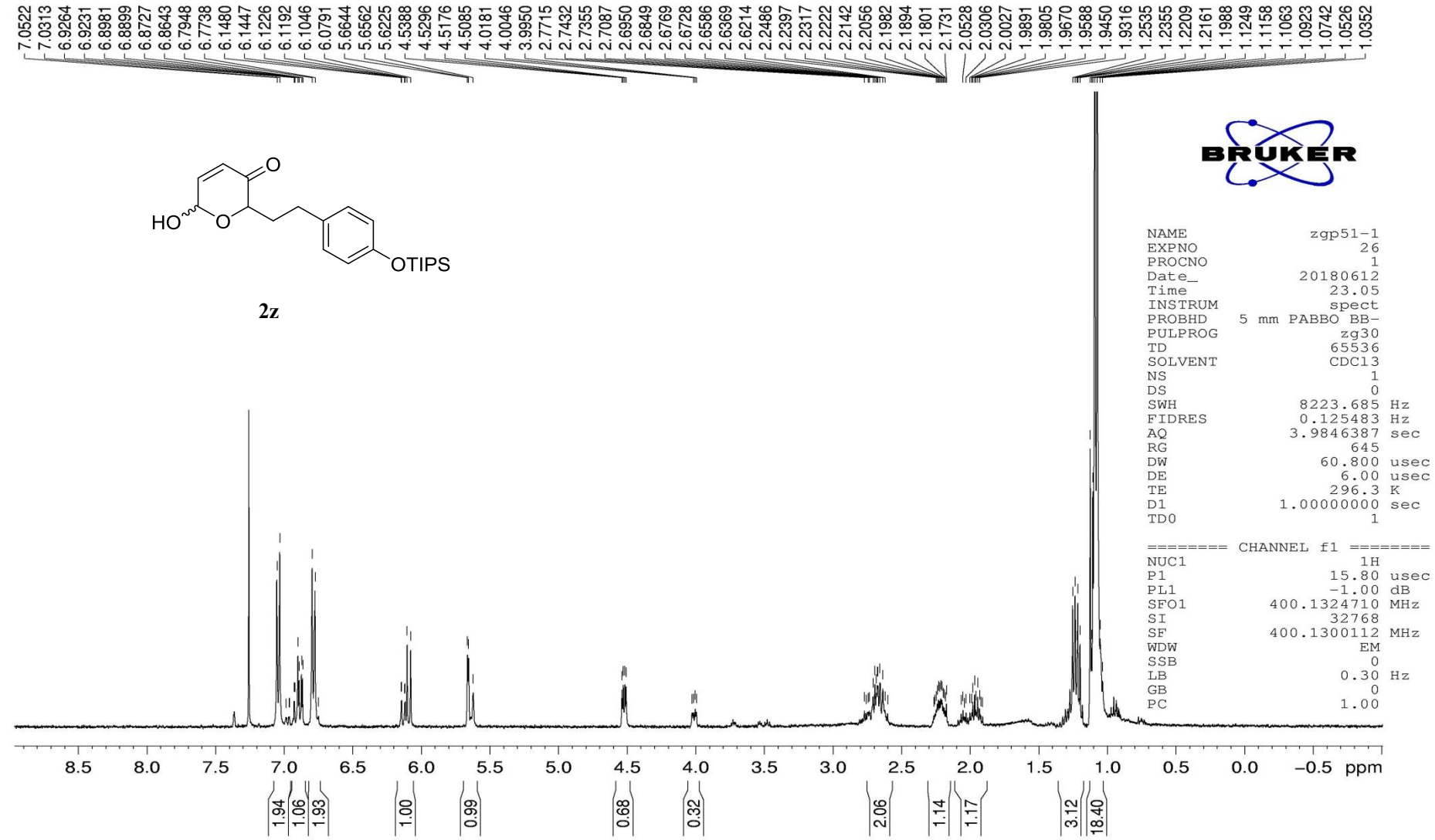

7.2568 7.0552 7.0500 7.0422 7.0346 7.0292 7.0215 7.0162 6.9367 6.9316 6.8969 6.8876 6.8760 6.8705 6.8668 6.8551 6.8466 6.8295 6.8211 6.1125 6.1087 6.0869 6.0831 6.0477 6.0221 5.5370 5.5282 4.4451 4.4362 4.4219 4.4130 3.9904 3.9834 3.9710 3.9612 3.7944 2.7534 2.7344 2.7246 2.7166 2.7107 2.7034 2.6978 2.6896 2.6786 2.3233 2.3154 2.3073 2.2948 2.2888 2.2798 2.2680 2.2590 2.2468 2.2440 2.2354 2.0694 2.0473 1.9703 1.9556 1.9521 1.9471 1.9336 1.9290 1.9198 1.9150 1.9018 1.8969 1.8932 1.8786
HO
O
O
OAc
OMe
2y
BRUKER
NAME zgp102
EXPNO 4
PROCNO 1
Date_ 20180709
Time 17.49
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 2
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 126.97
DW 62.400 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.02 3.04 1.00 0.99 0.68 0.35 3.11 2.02 4.24 1.43


196.89
170.09
169.70
149.21
149.11
148.07
144.74
139.08
133.82
133.64
128.71
127.36
126.75
126.62
123.68
123.28
112.48
112.41
90.87
87.55
77.82
72.53
55.96
31.60
30.33
30.10
20.85
20.79
BRUKER
HO
O
O
OAc
OMe
2y
NAME zgp102
EXPNO 5
PROCNO 1
Date_ 20180709
Time 17.51
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 148
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

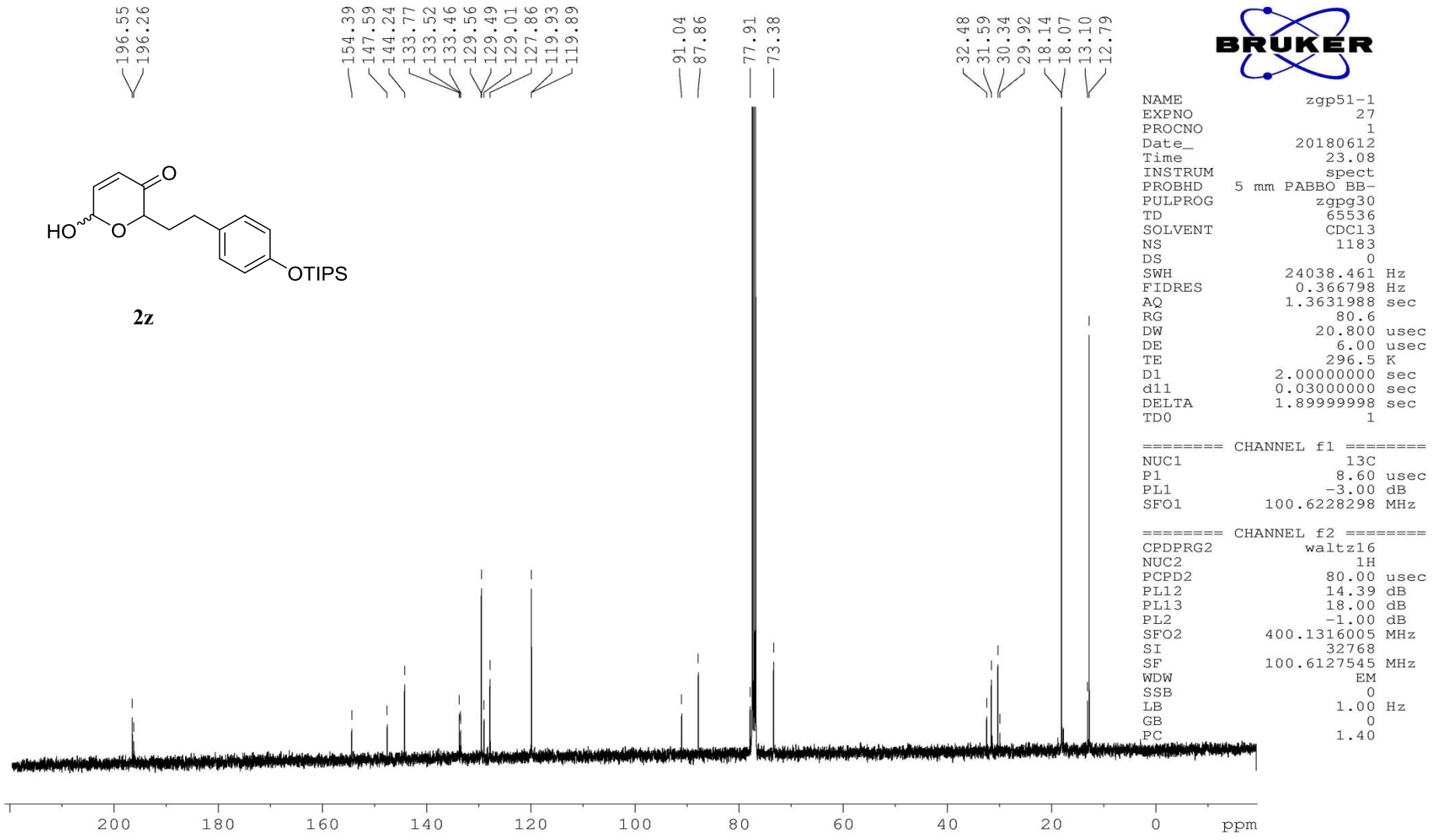

7.3809
7.3790
7.3765
7.3746
7.2602
7.0455
7.0243
7.0172
6.8147
6.8074
6.8023
6.7912
6.7862
6.7789
6.3378
6.3332
6.3298
6.3252
6.2382
6.2301
4.6753
4.6584
4.6413
2.7055
2.6894
2.6705
2.6584
2.6513
2.6390
2.6195
2.6040
2.1626
2.1454
2.1433
2.1258
2.1070
1.2832
1.2674
1.2635
1.2503
1.2458
1.2312
1.2260
1.2087
1.1016
1.0836
1.0612
1.0527
1.0444
OH
O
OTIPS
BRUKER
NAME zgp31-YL
EXPNO 1
PROCNO 1
Date_ 20180531
Time 16.17
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 142.88
DW 62.400 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
0.98
2.11
2.08
1.01
1.00
1.04
2.14
2.09
3.01
17.85

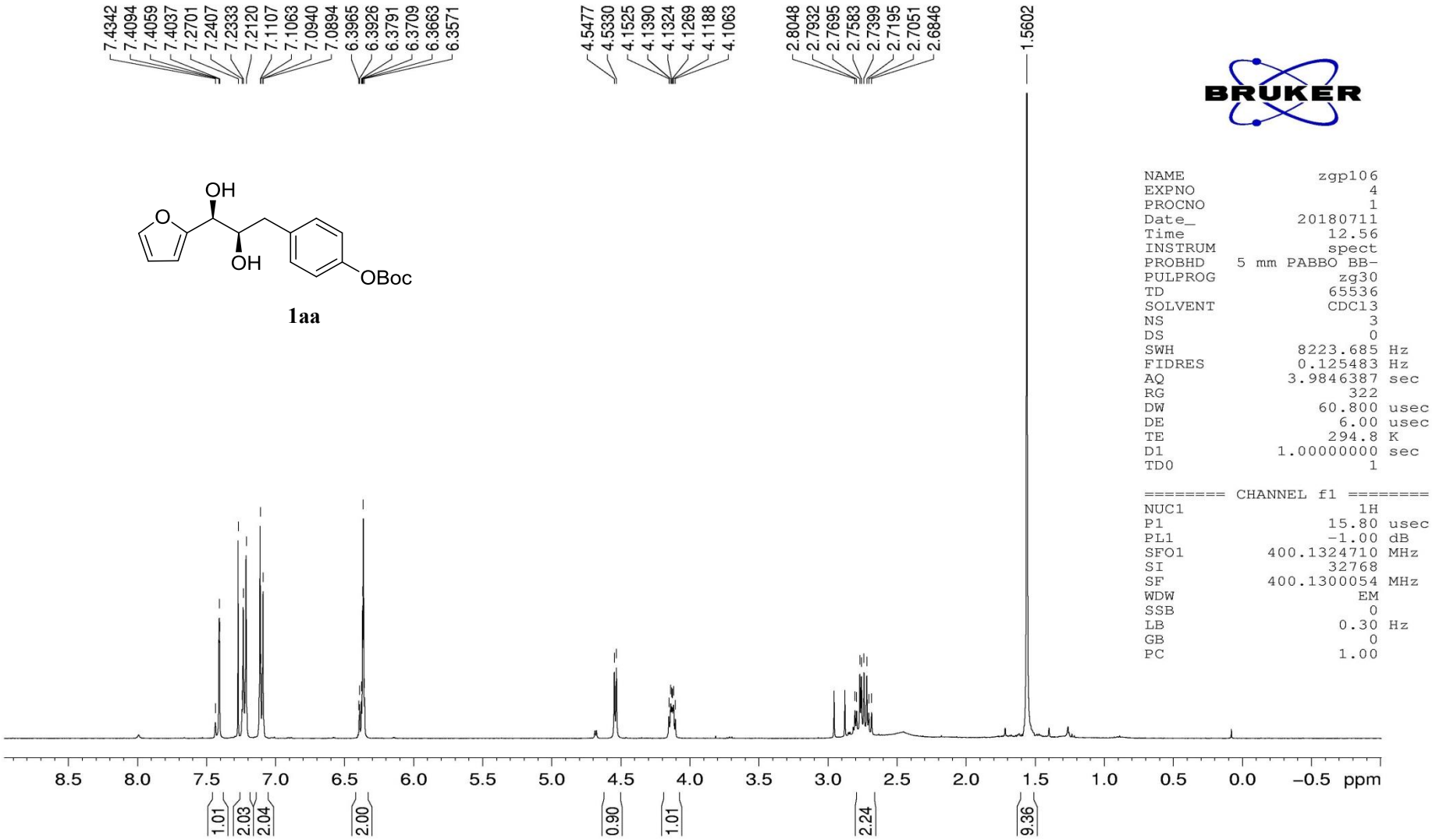
**1z**

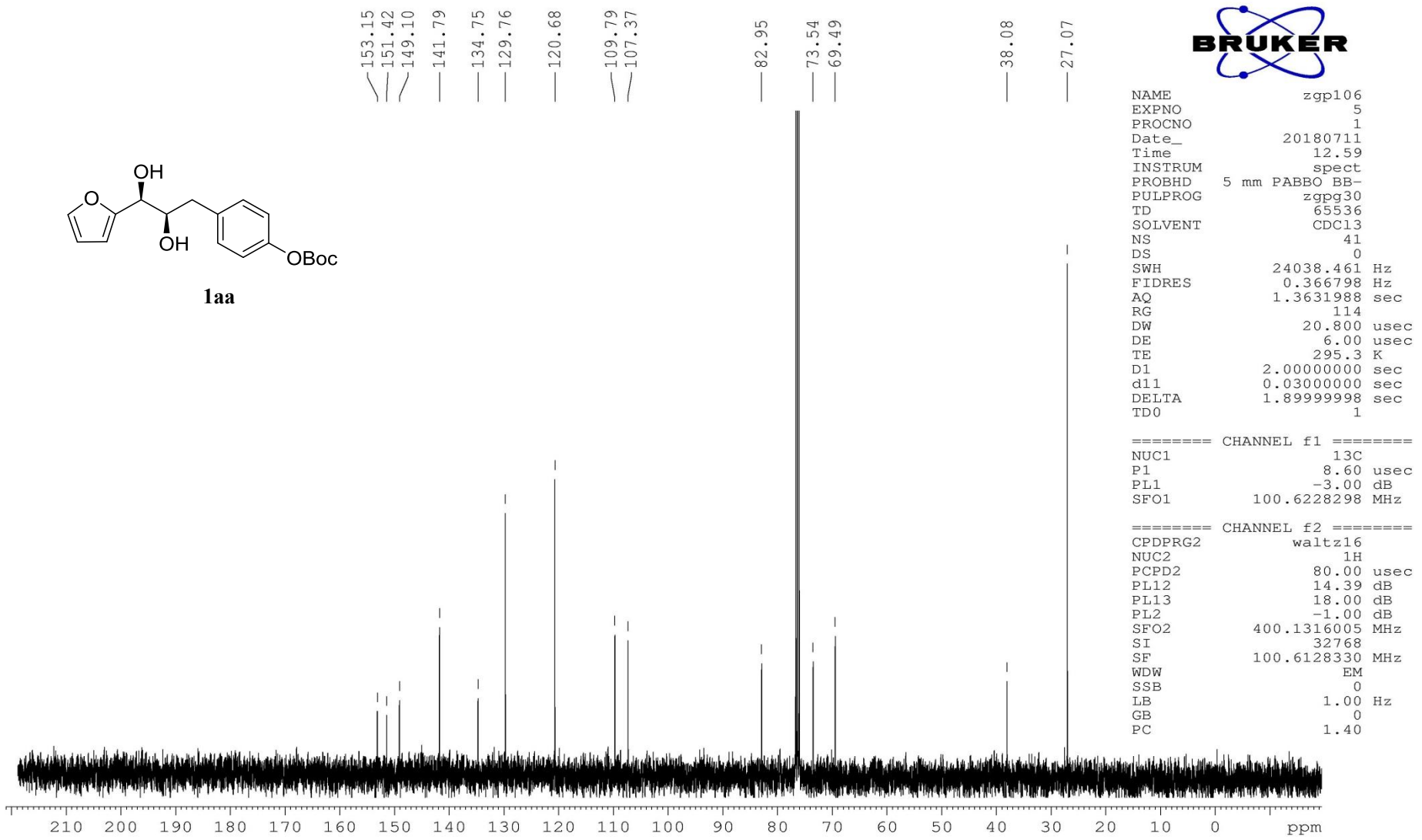

156.75
154.33
142.14
133.87
129.40
119.91
110.29
106.12
67.13
37.30
31.05
18.07
12.79
OH
O
OTIPS
1z
BRUKER
NAME zgp31-YL
EXPNO 2
PROCNO 1
Date_ 20180531
Time 17.23
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 145
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 295.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127552 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

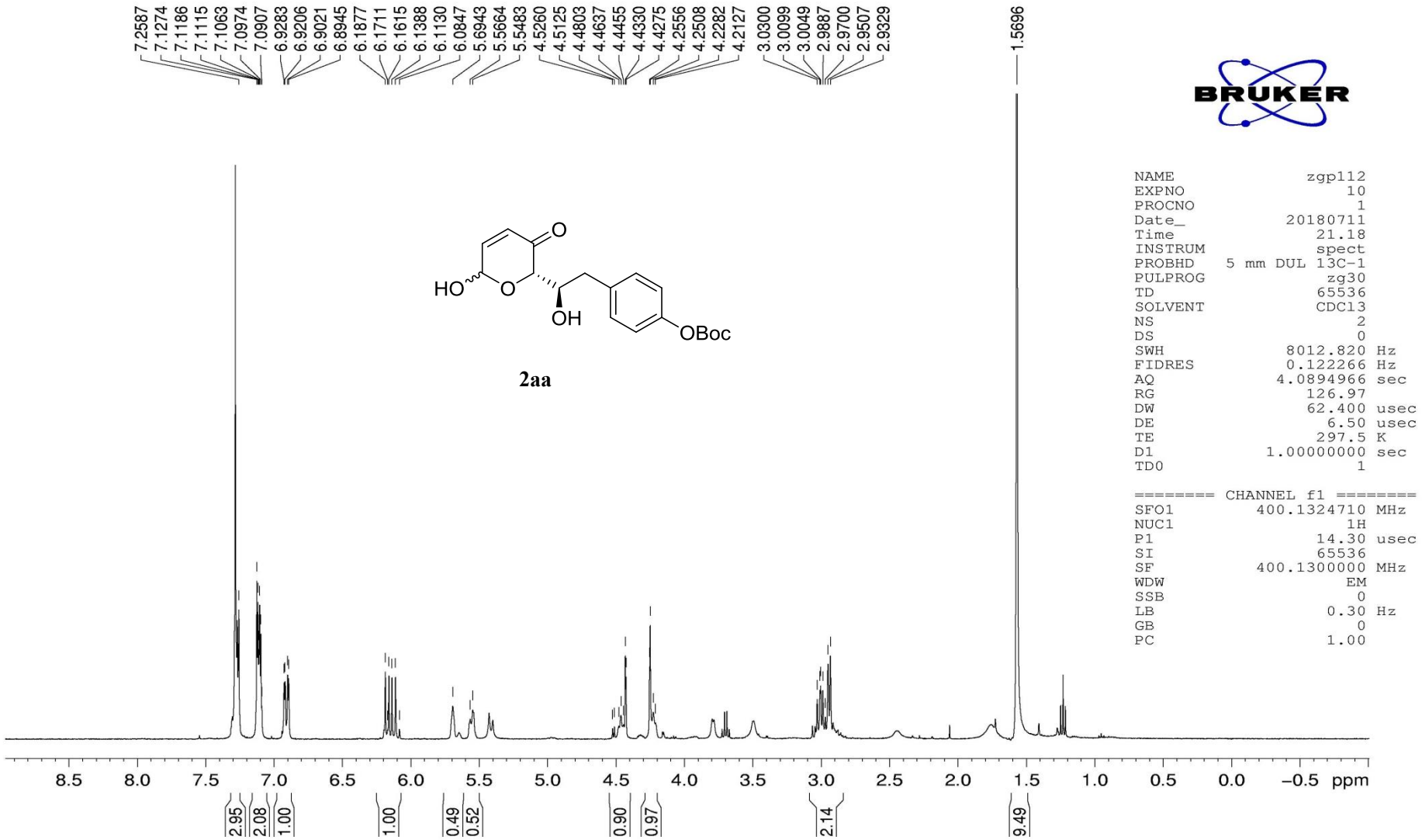

7.0522 7.0313 6.9264 6.9231 6.8981 6.8899 6.8727 6.8643 6.7948 6.7738 6.1480 6.1447 6.1226 6.1192 6.1046 6.0791 5.6644 5.6562 5.6225 4.5388 4.5296 4.5176 4.5085 4.0181 4.0046 3.9950 2.7715 2.7432 2.7355 2.7087 2.6950 2.6849 2.6769 2.6728 2.6586 2.6369 2.6214 2.2486 2.2397 2.2317 2.2222 2.2142 2.2056 2.1982 2.1894 2.1801 2.1731 2.0528 2.0306 2.0027 1.9891 1.9805 1.9670 1.9588 1.9450 1.9316 1.2535 1.2355 1.2209 1.2161 1.1988 1.1249 1.1158 1.1063 1.0923 1.0742 1.0526 1.0352
HO
O
O
OTIPS
BRUKER
NAME zgp51-1
EXPNO 26
PROCNO 1
Date_ 20180612
Time 23.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 645
DW 60.800 usec
DE 6.00 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.94 1.06 1.93 1.00 0.99 0.68 0.32 2.06 1.14 1.17 3.12 18.40

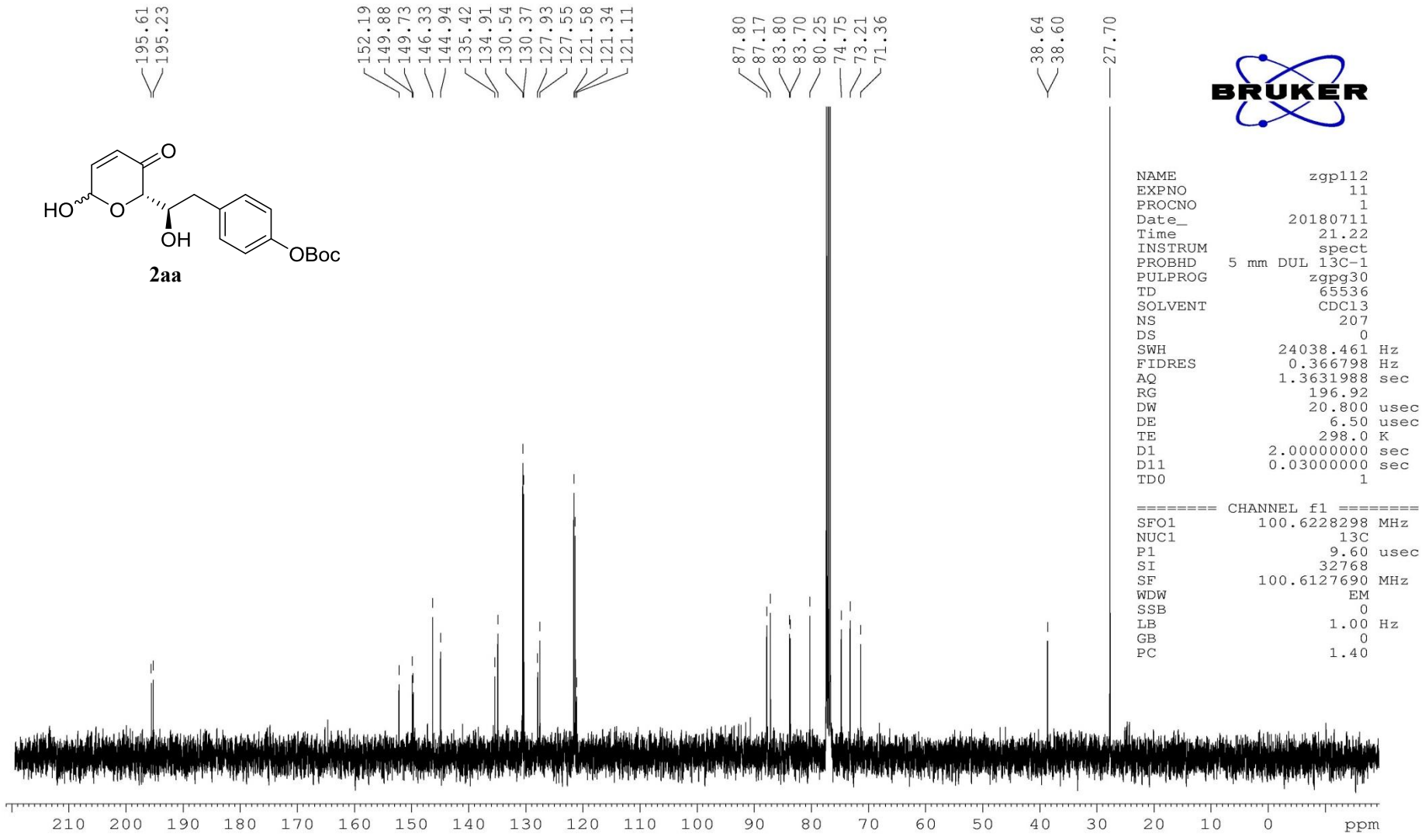
**2z**

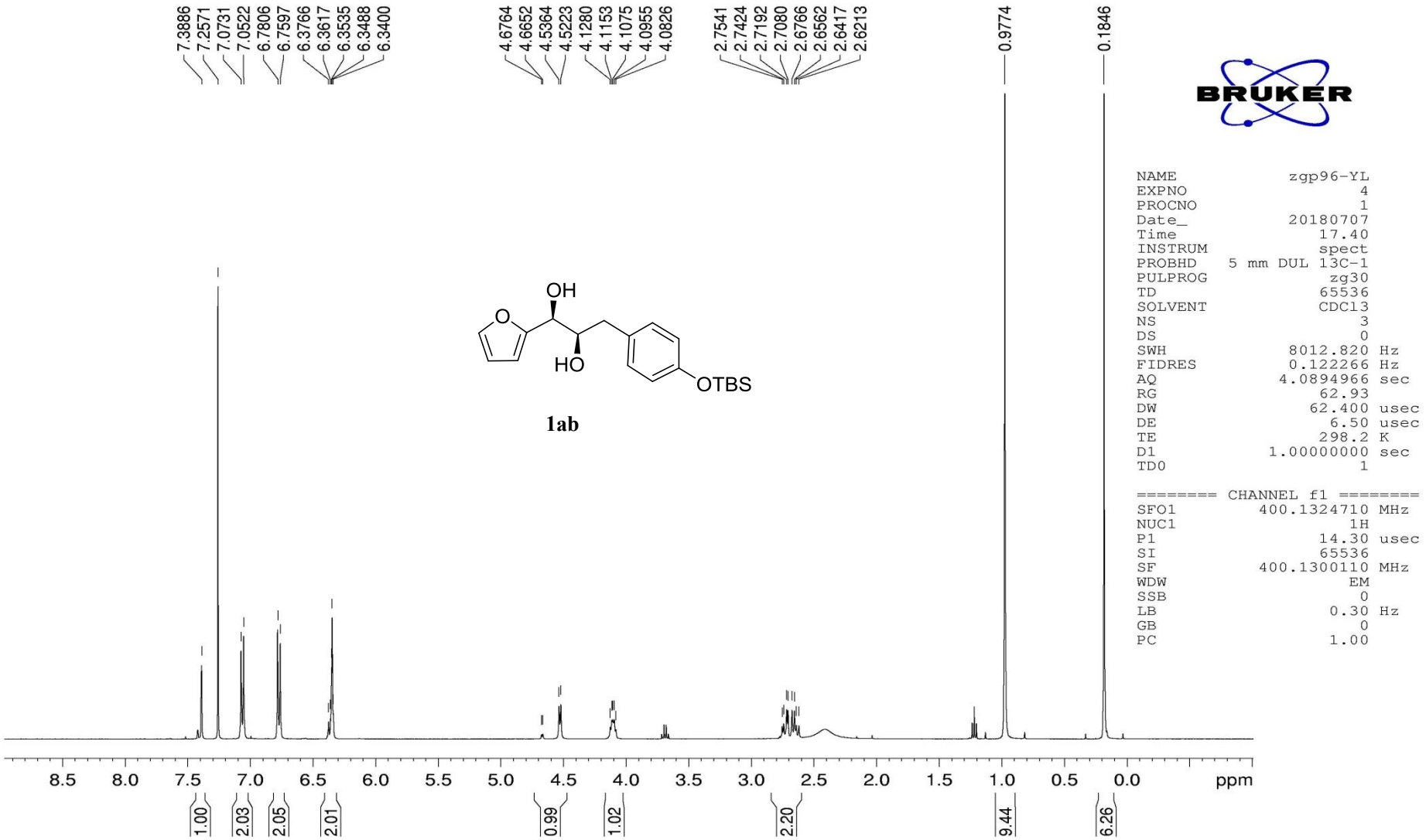

196.55
196.26
154.39
147.59
144.24
133.77
133.52
133.46
129.56
129.49
129.01
127.86
119.93
119.89
91.04
87.86
77.91
73.38
32.48
31.59
30.34
29.92
18.14
18.07
13.10
12.79
HO
O
O
OTIPS
2z
BRUKER
NAME zgp51-1
EXPNO 27
PROCNO 1
Date_ 20180612
Time 23.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1183
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 80.6
DW 20.800 usec
DE 6.00 usec
TE 296.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 14.39 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127545 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

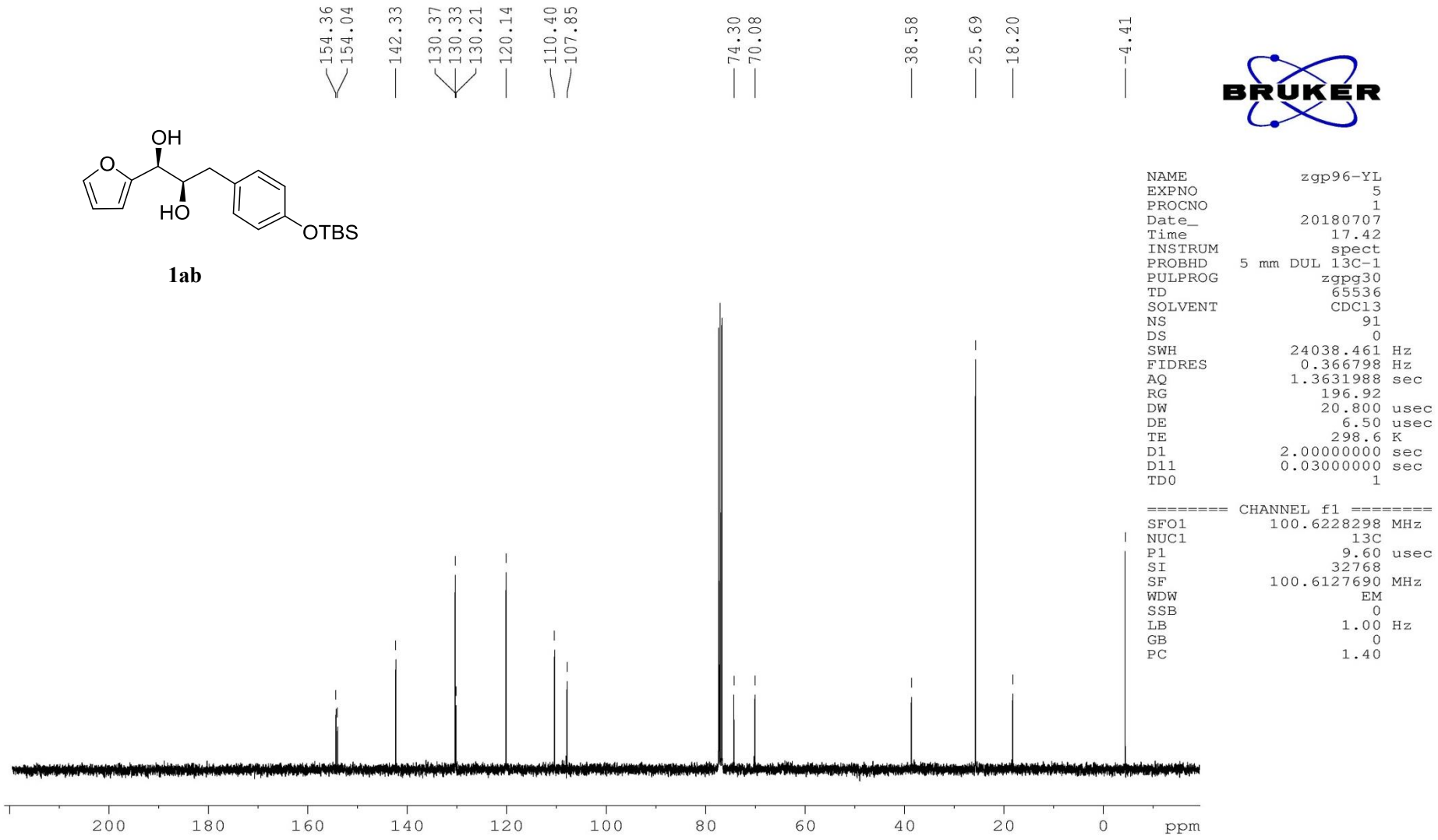

7.4342
7.4094
7.4059
7.4037
7.2701
7.2407
7.2333
7.2120
7.1107
7.1063
7.0940
7.0894
6.3965
6.3926
6.3791
6.3709
6.3663
6.3571
4.5477
4.5330
4.1525
4.1390
4.1324
4.1269
4.1188
4.1063
2.8048
2.7932
2.7695
2.7583
2.7399
2.7195
2.7051
2.6846
1.5602
OH
O
OH
OBoc
1aa
BRUKER
NAME zgp106
EXPNO 4
PROCNO 1
Date_ 20180711
Time 12.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 322
DW 60.800 usec
DE 6.00 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 ppm
1.01
2.03
2.04
2.00
0.90
1.01
2.24
9.36

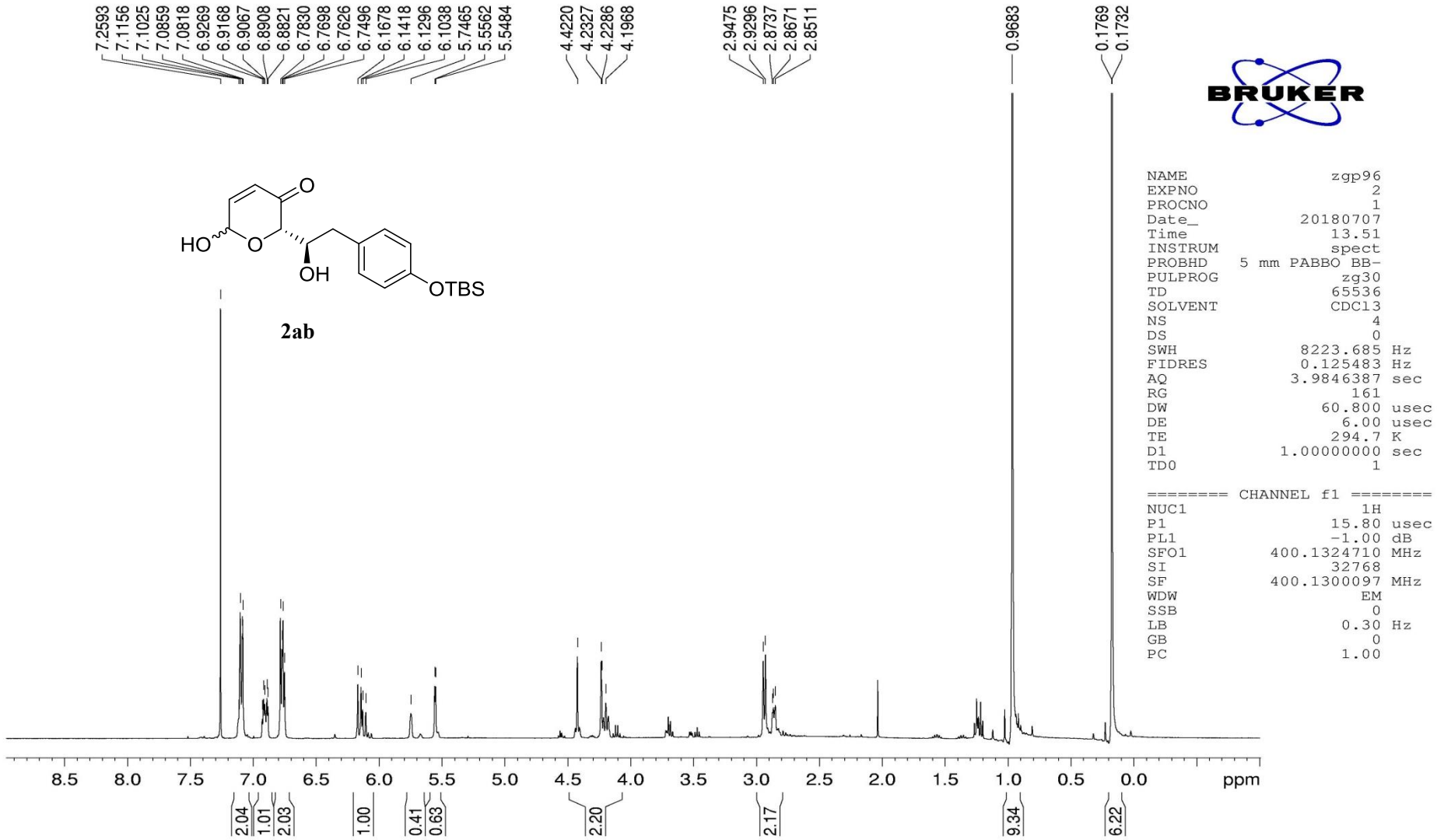

153.15
151.42
149.10
141.79
134.75
129.76
120.68
109.79
107.37
82.95
73.54
69.49
38.08
27.07
OH
O
OH
OBoc
1aa
BRUKER
NAME zgp106
EXPNO 5
PROCNO 1
Date_ 20180711
Time 12.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 41
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 114
DW 20.800 usec
DE 6.00 usec
TE 295.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 14.39 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6128330 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

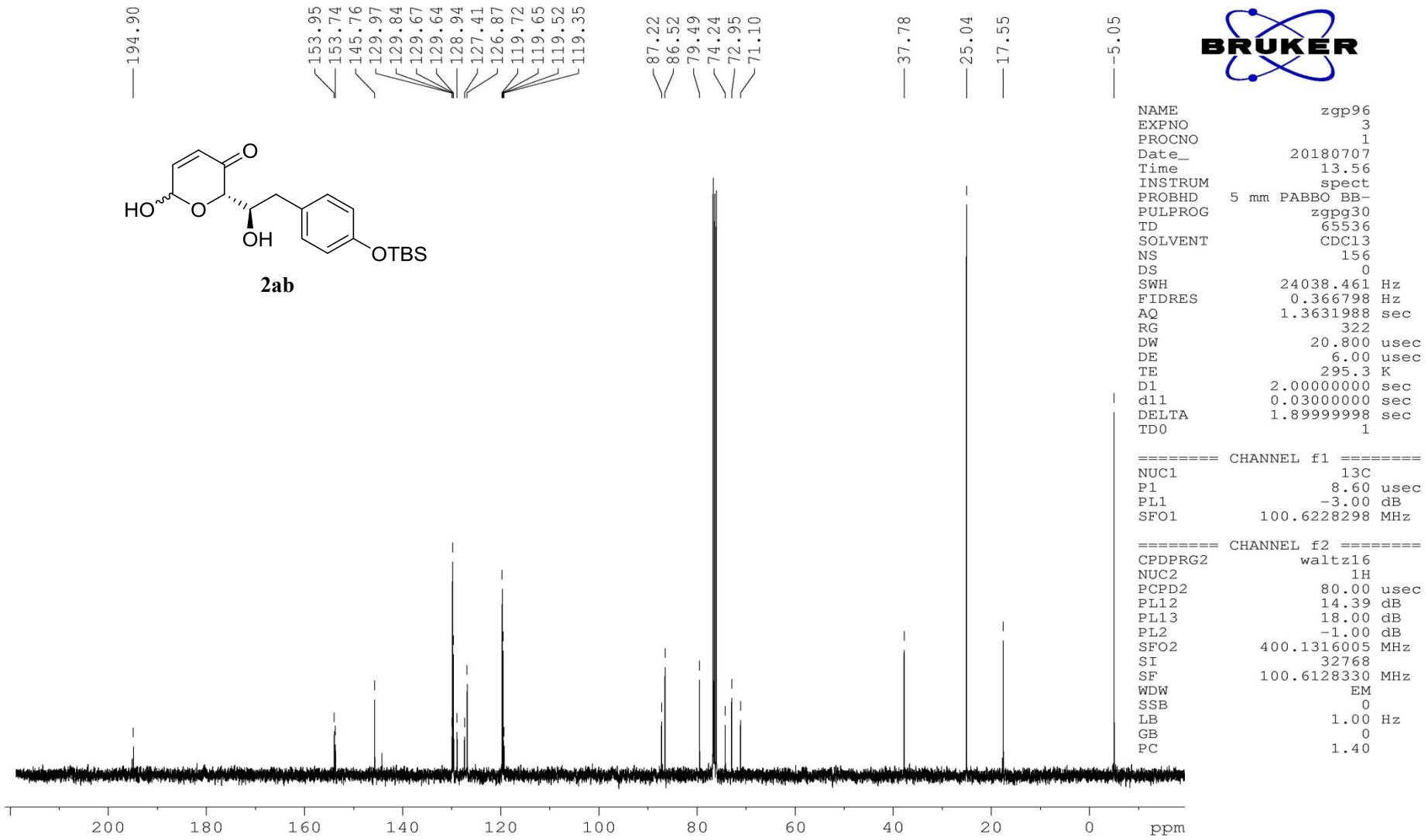

7.2587
7.1274
7.1186
7.1115
7.1063
7.0974
7.0907
6.9283
6.9206
6.9021
6.8945
6.1877
6.1711
6.1615
6.1388
6.1130
6.0847
5.6943
5.5664
5.5483
4.5260
4.5125
4.4803
4.4637
4.4455
4.4330
4.4275
4.2556
4.2508
4.2282
4.2127
3.0300
3.0099
3.0049
2.9887
2.9700
2.9507
2.9329
1.5696
HO
O
O
OH
OBoc
2aa
NAME zgp112
EXPNO 10
PROCNO 1
Date_ 20180711
Time 21.18
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 2
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 126.97
DW 62.400 usec
DE 6.50 usec
TE 297.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
2.95 2.08 1.00 1.00 0.49 0.52 0.90 0.97 2.14 9.49

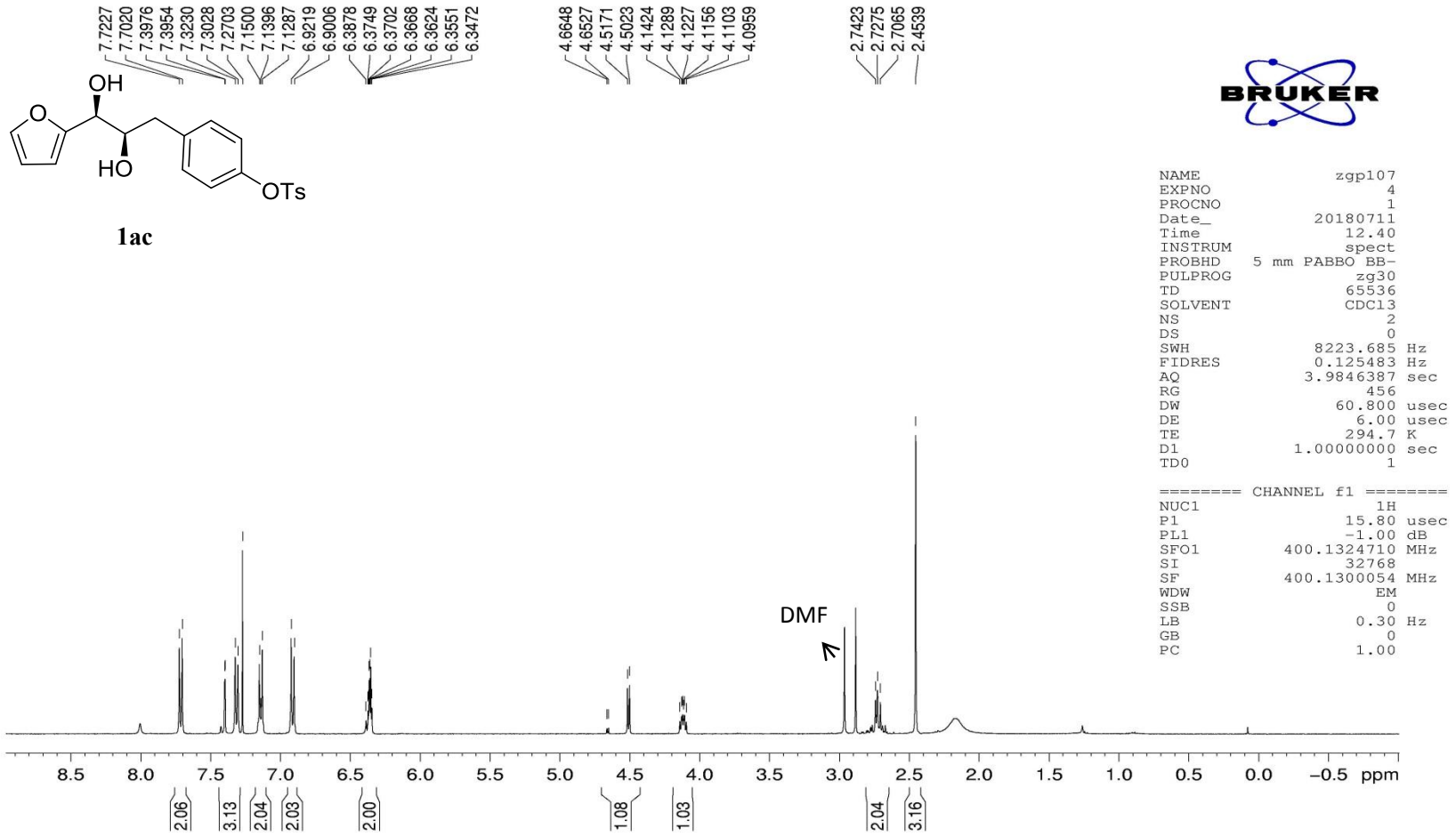

195.61
195.23
152.19
149.88
149.73
146.33
144.94
135.42
134.91
130.54
130.37
127.93
127.55
121.58
121.34
121.11
87.80
87.17
83.80
83.70
80.25
74.75
73.21
71.36
38.64
38.60
27.70
HO
O
O
OH
OBoc
2aa
BRUKER
NAME zgp112
EXPNO 11
PROCNO 1
Date_ 20180711
Time 21.22
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 207
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

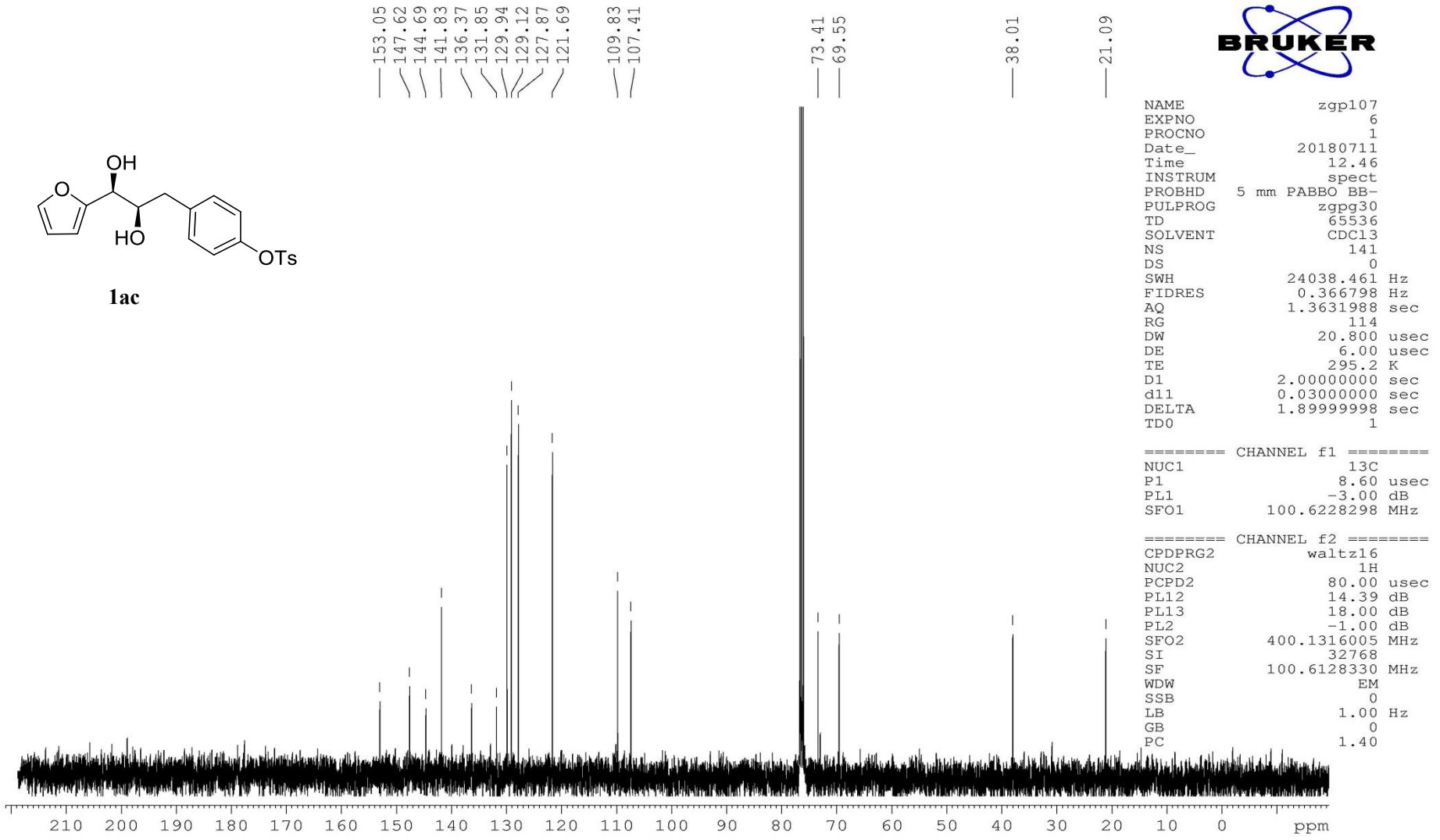

7.3886
7.2571
7.0731
7.0522
6.7806
6.7597
6.3766
6.3617
6.3535
6.3488
6.3400
4.6764
4.6652
4.5364
4.5223
4.1280
4.1153
4.1075
4.0955
4.0826
2.7541
2.7424
2.7192
2.7080
2.6766
2.6562
2.6417
2.6213
0.9774
0.1846
OH
O
HO
OTBS
1ab
BRUKER
NAME zgp96-YL
EXPNO 4
PROCNO 1
Date_ 20180707
Time 17.40
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 62.93
DW 62.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
2.03
2.05
2.01
0.99
1.02
2.20
9.44
6.26

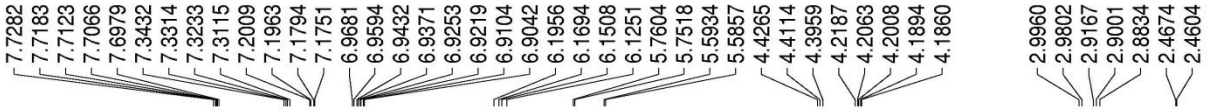

154.36
154.04
142.33
130.37
130.33
130.21
120.14
110.40
107.85
74.30
70.08
38.58
25.69
18.20
-4.41
OH
O
HO
OTBS
1ab
NAME zgp96-YL
EXPNO 5
PROCNO 1
Date_ 20180707
Time 17.42
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 91
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

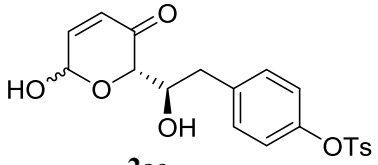

7.2593
7.1156
7.1025
7.0859
7.0818
6.9269
6.9168
6.9067
6.8908
6.8821
6.7830
6.7698
6.7626
6.7496
6.1678
6.1418
6.1296
6.1038
5.7465
5.5562
5.5484
4.4220
4.2327
4.2286
4.1968
2.9475
2.9296
2.8737
2.8671
2.8511
0.9683
0.1769
0.1732
HO
O
O
OH
OTBS
2ab
BRUKER
NAME zgp96
EXPNO 2
PROCNO 1
Date_ 20180707
Time 13.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.00 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.04
1.01
2.03
1.00
0.41
0.63
2.20
2.17
9.34
6.22


194.90
153.95
153.74
145.76
129.97
129.84
129.67
129.64
128.94
127.41
126.87
119.72
119.65
119.52
119.35
87.22
86.52
79.49
74.24
72.95
71.10
37.78
25.04
17.55
-5.05
HO
O
O
OH
OTBS
2ab
NAME zgp96
EXPNO 3
PROCNO 1
Date_ 20180707
Time 13.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 156
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 322
DW 20.800 usec
DE 6.00 usec
TE 295.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 14.39 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6128330 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


7.7227
7.7020
7.3976
7.3954
7.3230
7.3028
7.2703
7.1500
7.1396
7.1287
6.9219
6.9006
6.3878
6.3749
6.3702
6.3668
6.3624
6.3551
6.3472
4.6648
4.6527
4.5171
4.5023
4.1424
4.1289
4.1227
4.1156
4.1103
4.0959
2.7423
2.7275
2.7065
2.4539
OH
O
HO
OTs
1ac
BRUKER
NAME zgp107
EXPNO 4
PROCNO 1
Date_ 20180711
Time 12.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 2
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 456
DW 60.800 usec
DE 6.00 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
DMF
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
2.06
3.13
2.04
2.03
2.00
1.08
1.03
2.04
3.16

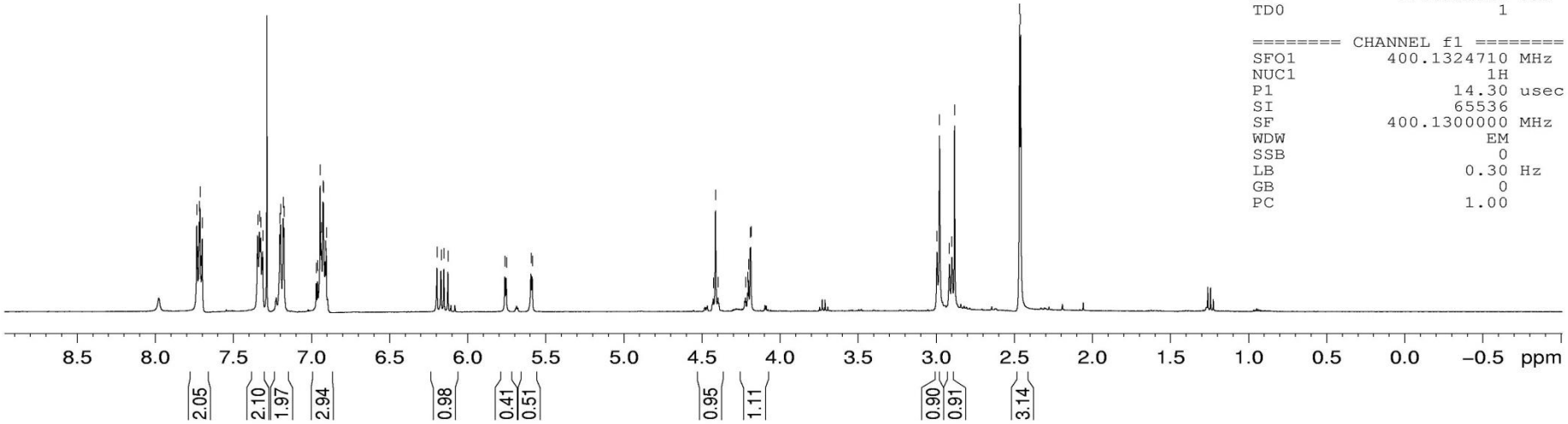

153.05
147.62
144.69
141.83
136.37
131.85
129.94
129.12
127.87
121.69
109.83
107.41
73.41
69.55
38.01
21.09
OH
O
HO
OTs
1ac
BRUKER
NAME zgp107
EXPNO 6
PROCNO 1
Date_ 20180711
Time 12.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 141
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 114
DW 20.800 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 14.39 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6128330 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

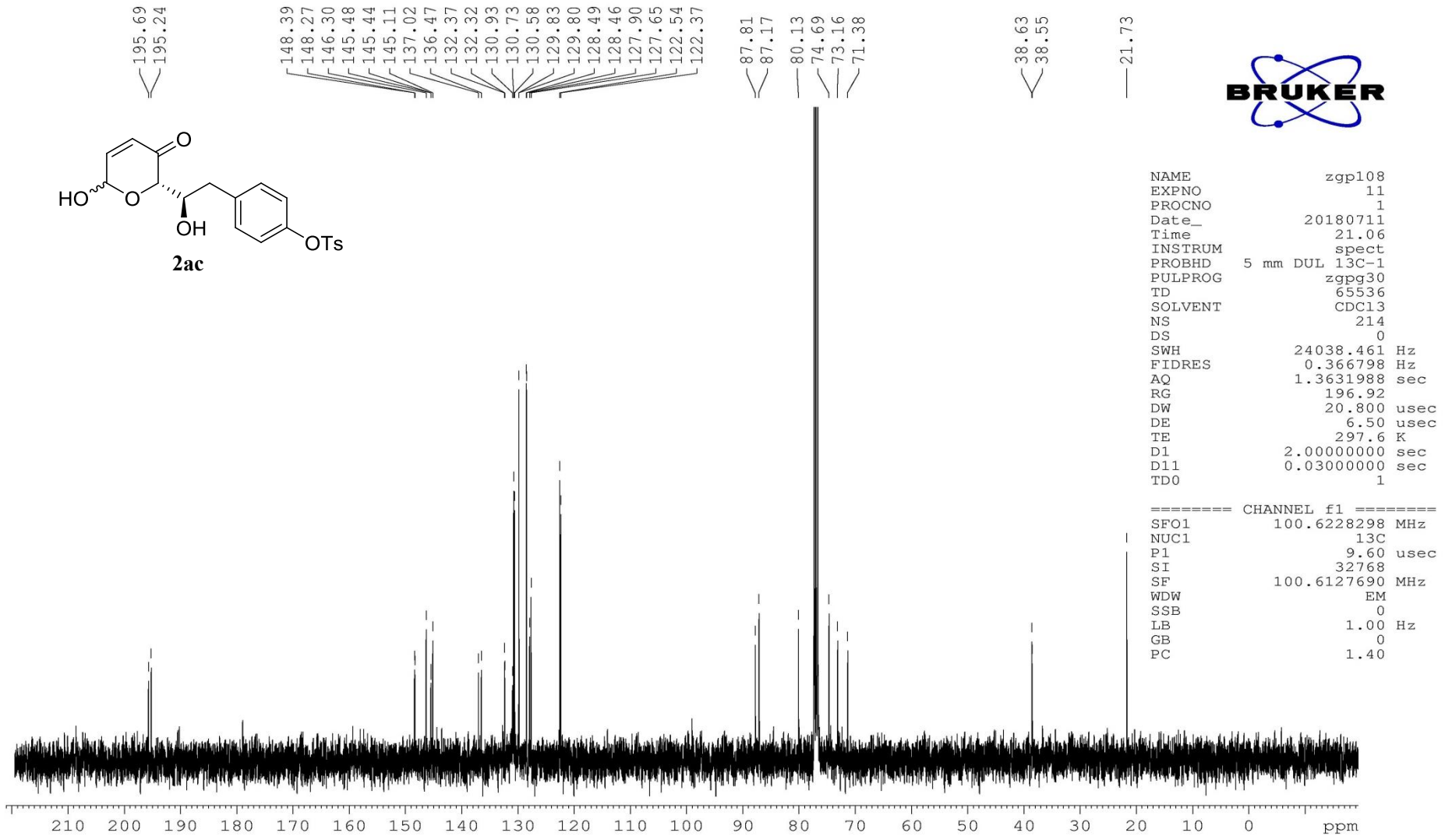


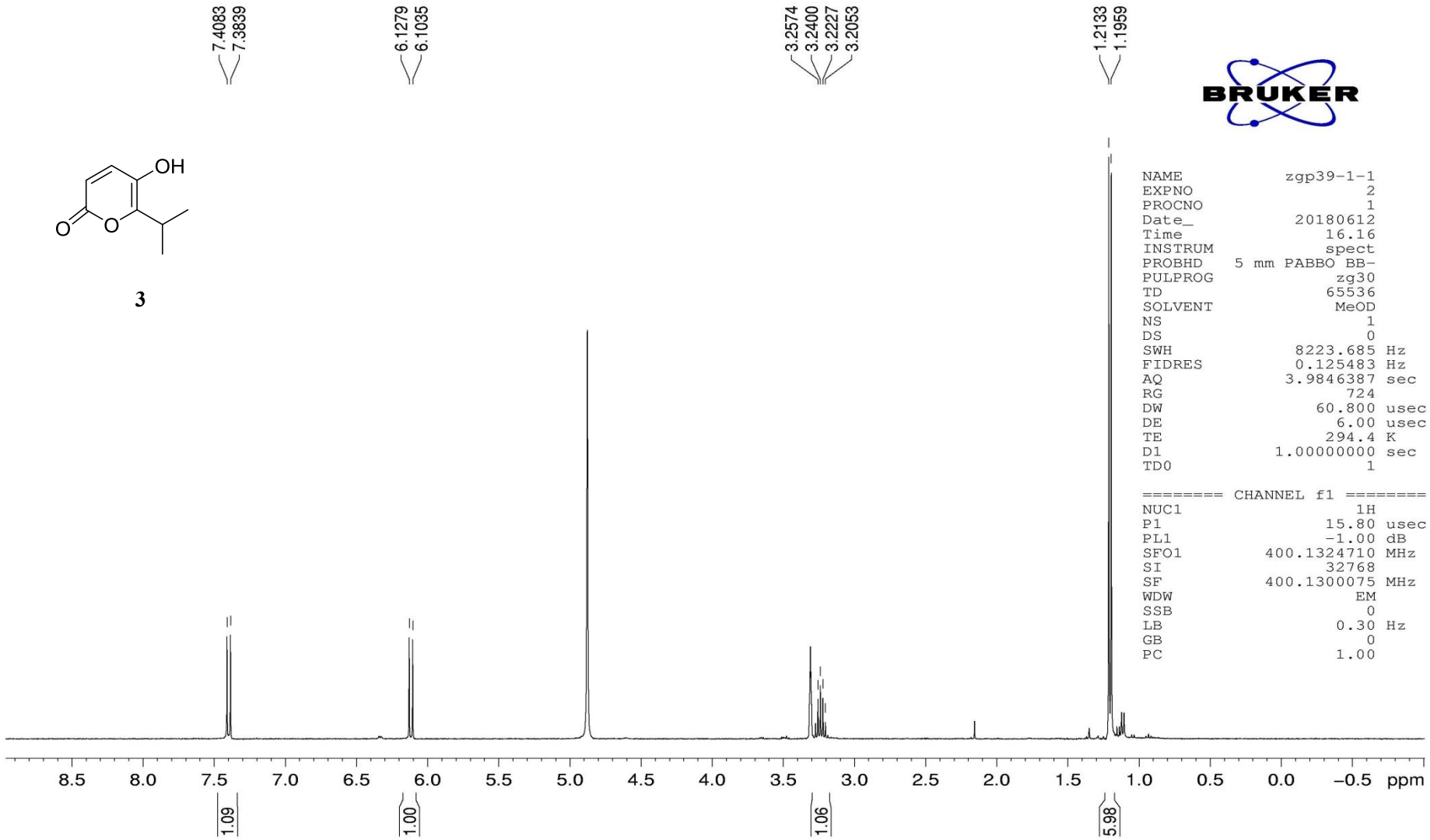



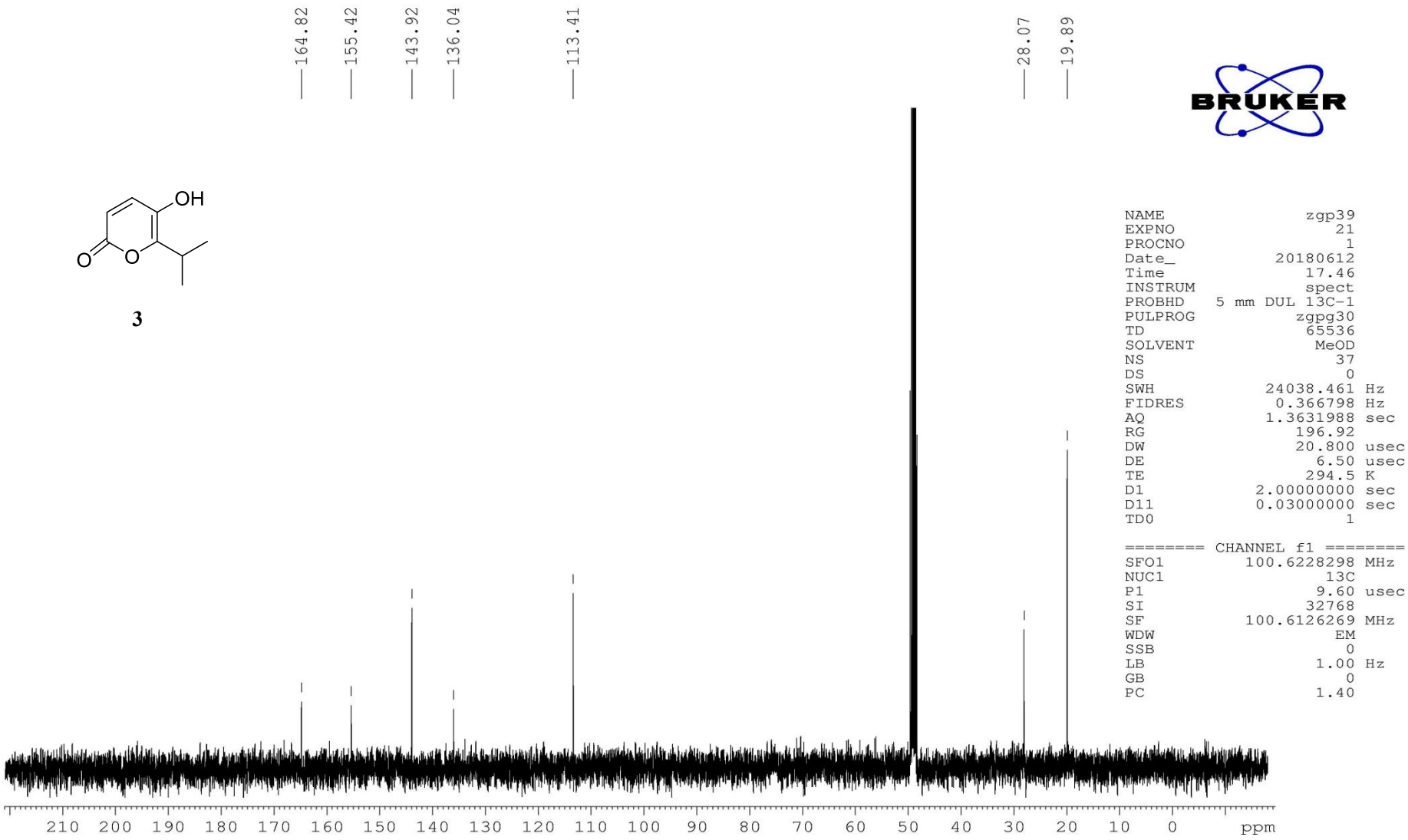

**2ac**

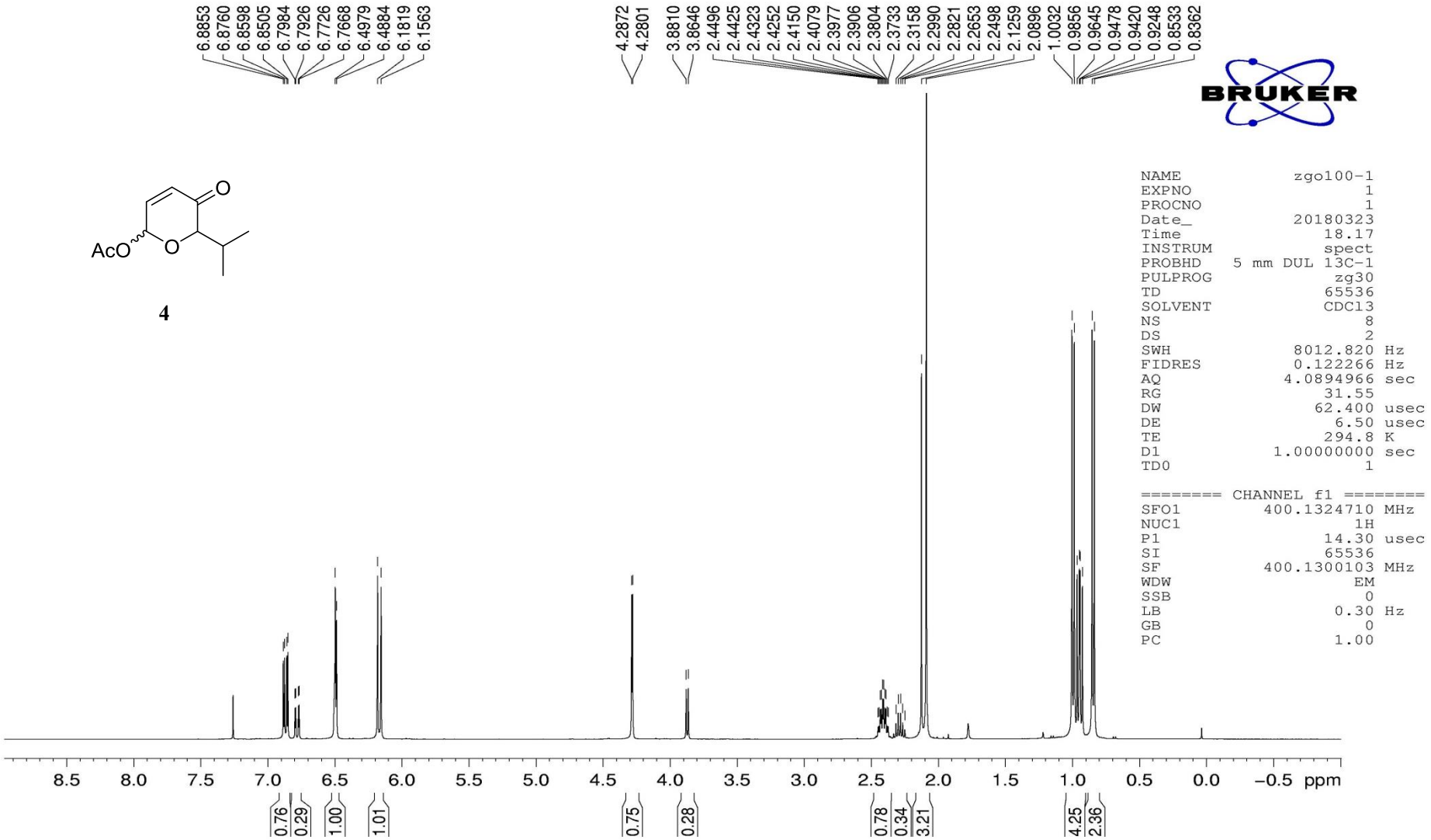
```
NAME                zgp108
EXPNO                   10
PROCNO                   1
Date_             20180711
Time                 21.03
INSTRUM              spect
PROBHD    5 mm DUL 13C-1
PULPROG               zg30
TD                   65536
SOLVENT              CDC13
NS                       3
DS                       0
SWH               8012.820 Hz
FIDRES               0.122266 Hz
AQ                4.0894966 sec
RG                   126.97
DW                   62.400 usec
DE                    6.50 usec
TE                   297.2 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           400.1324710 MHz
NUC1                    1H
P1                   14.30 usec
SI                   65536
SF             400.1300000 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

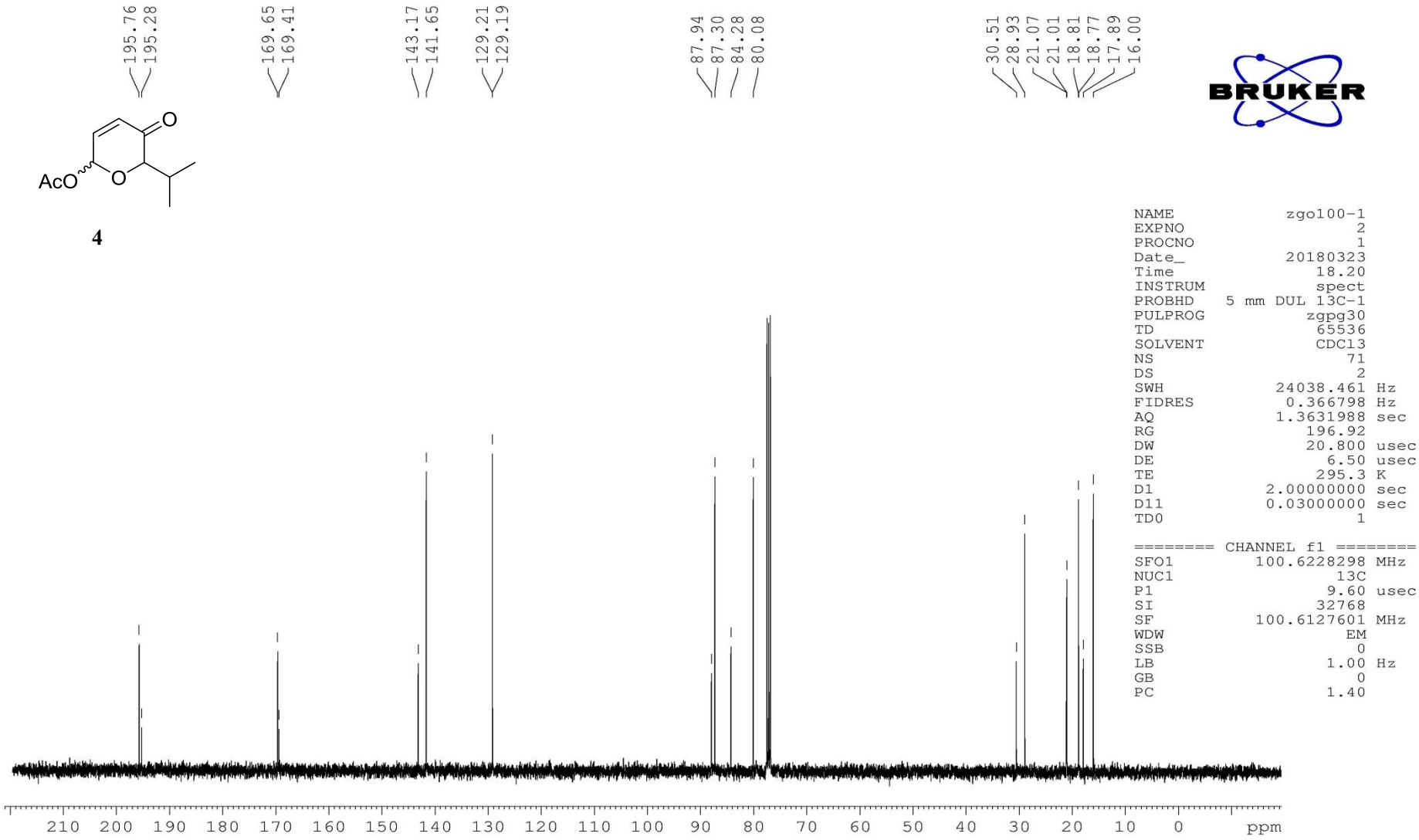


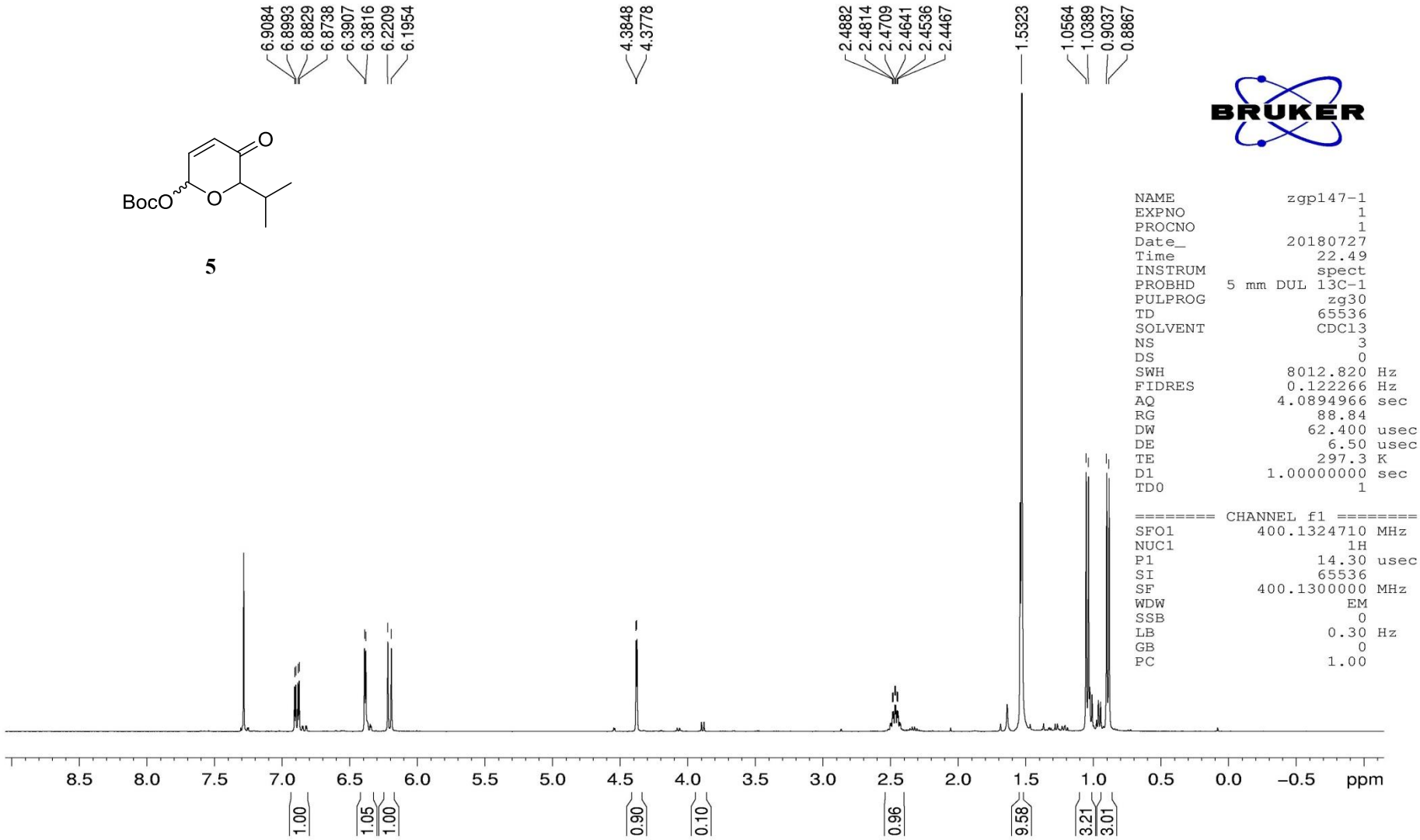

195.69
195.24
148.39
148.27
146.30
145.48
145.44
145.11
137.02
136.47
132.37
132.32
130.93
130.73
130.58
129.83
129.80
128.49
128.46
127.90
127.65
122.54
122.37
87.81
87.17
80.13
74.69
73.16
71.38
38.63
38.55
21.73
HO
O
O
OH
OTs
2ac
BRUKER
NAME zgp108
EXPNO 11
PROCNO 1
Date_ 20180711
Time 21.06
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 214
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

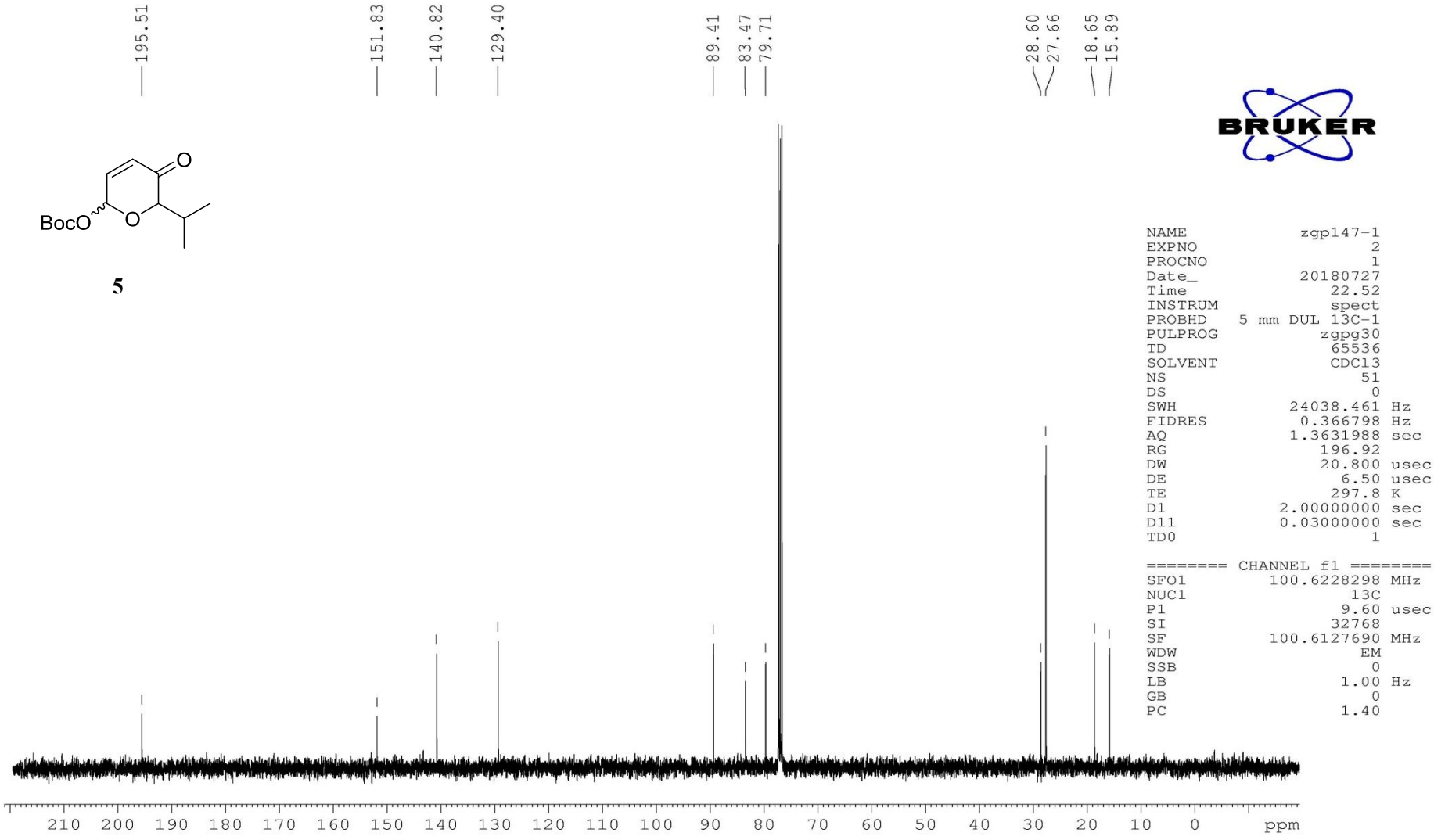

7.4083
7.3839
6.1279
6.1035
3.2574
3.2400
3.2227
3.2053
1.2133
1.1959
OH
O
O
3
BRUKER
NAME zgp39-1-1
EXPNO 2
PROCNO 1
Date_ 20180612
Time 16.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 1
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 724
DW 60.800 usec
DE 6.00 usec
TE 294.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300075 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 ppm
1.09
1.00
1.06
5.98

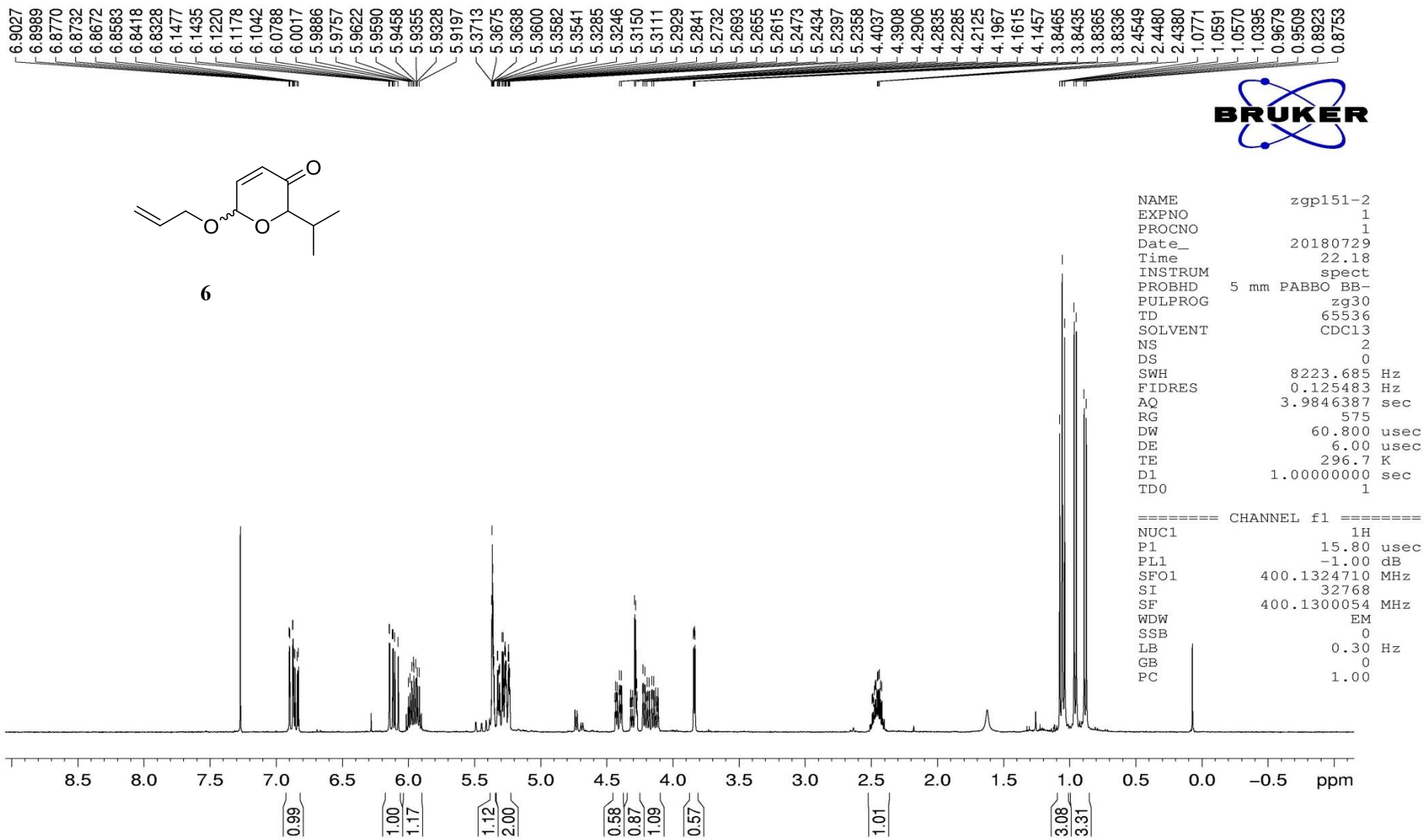

164.82
155.42
143.92
136.04
113.41
28.07
19.89
OH
O
O
3
NAME zgp39
EXPNO 21
PROCNO 1
Date_ 20180612
Time 17.46
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 37
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 294.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6126269 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

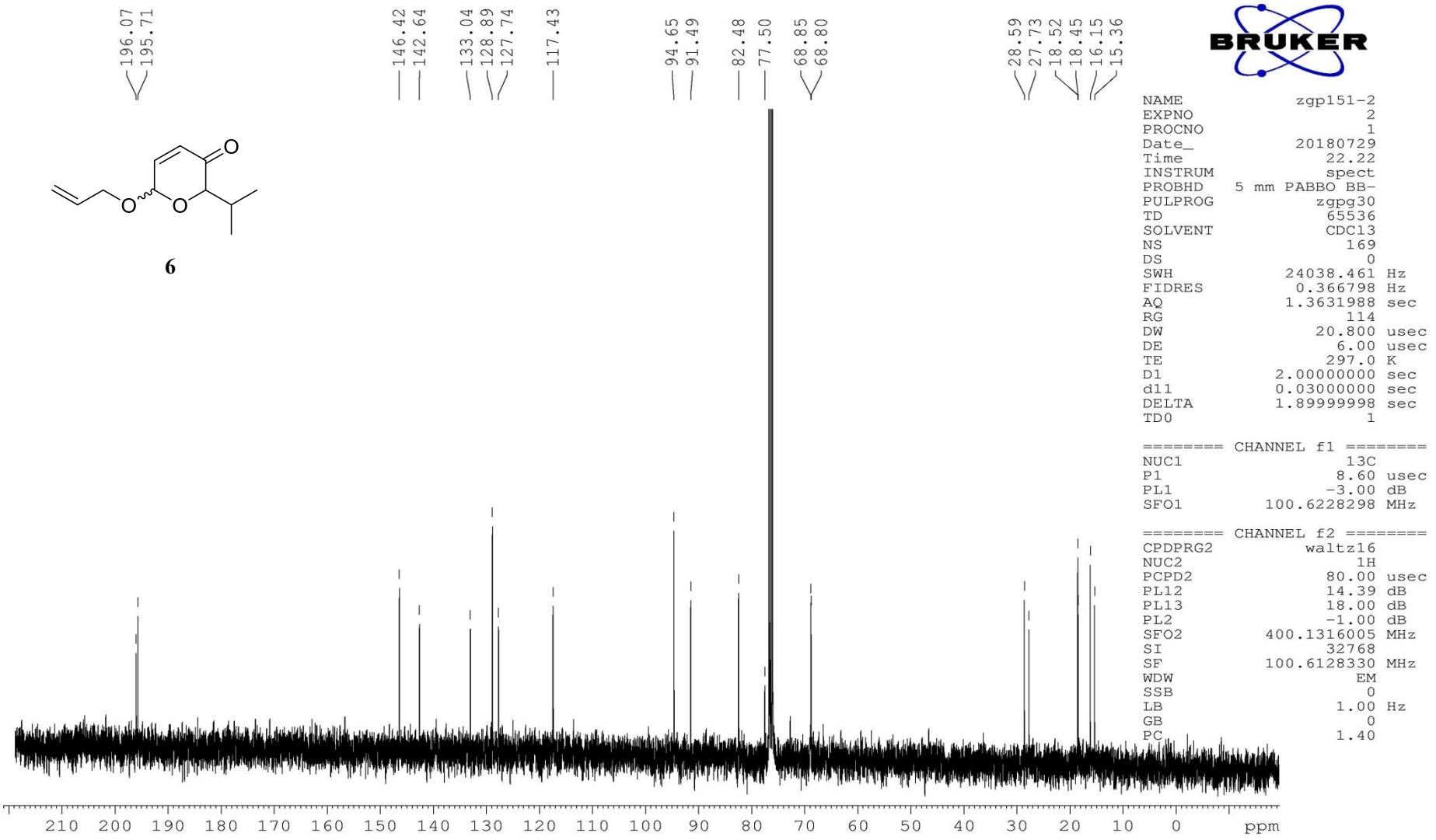

6.8853
6.8760
6.8598
6.8505
6.7984
6.7926
6.7726
6.7668
6.4979
6.4884
6.1819
6.1563
4.2872
4.2801
3.8810
3.8646
2.4496
2.4425
2.4323
2.4252
2.4150
2.4079
2.3977
2.3906
2.3804
2.3733
2.3158
2.2990
2.2821
2.2653
2.2498
2.1259
2.0896
1.0032
0.9856
0.9645
0.9478
0.9420
0.9248
0.8533
0.8362
BRUKER
AcO
O
O
4
NAME zgo100-1
EXPNO 1
PROCNO 1
Date_ 20180323
Time 18.17
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 31.55
DW 62.400 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
0.76
0.29
1.00
1.01
0.75
0.28
0.78
0.34
3.21
4.25
2.36

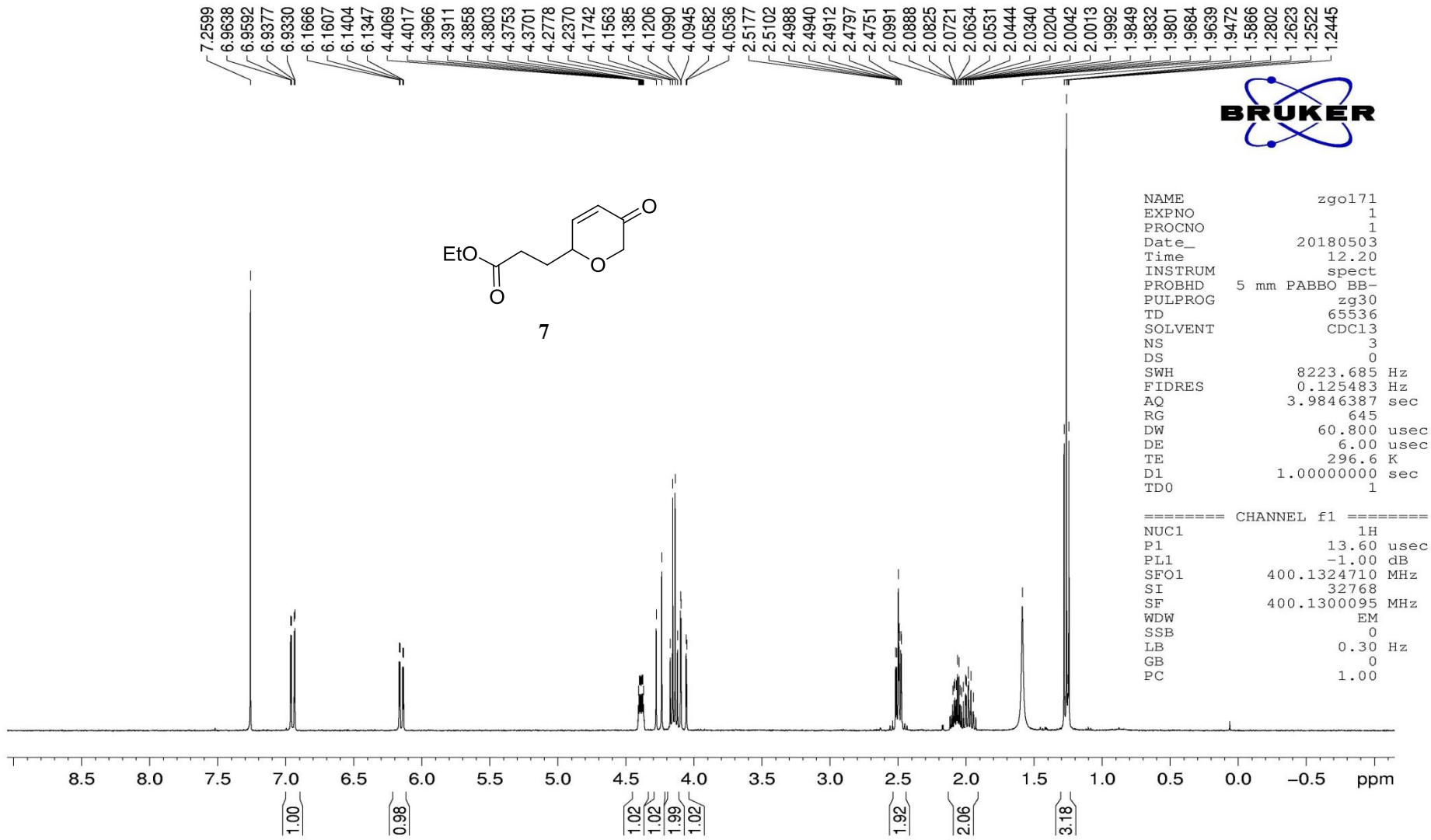

195.76
195.28
169.65
169.41
143.17
141.65
129.21
129.19
87.94
87.30
84.28
80.08
30.51
28.93
21.07
21.01
18.81
18.77
17.89
16.00
AcO
O
O
4
NAME zgo100-1
EXPNO 2
PROCNO 1
Date_ 20180323
Time 18.20
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 71
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127601 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

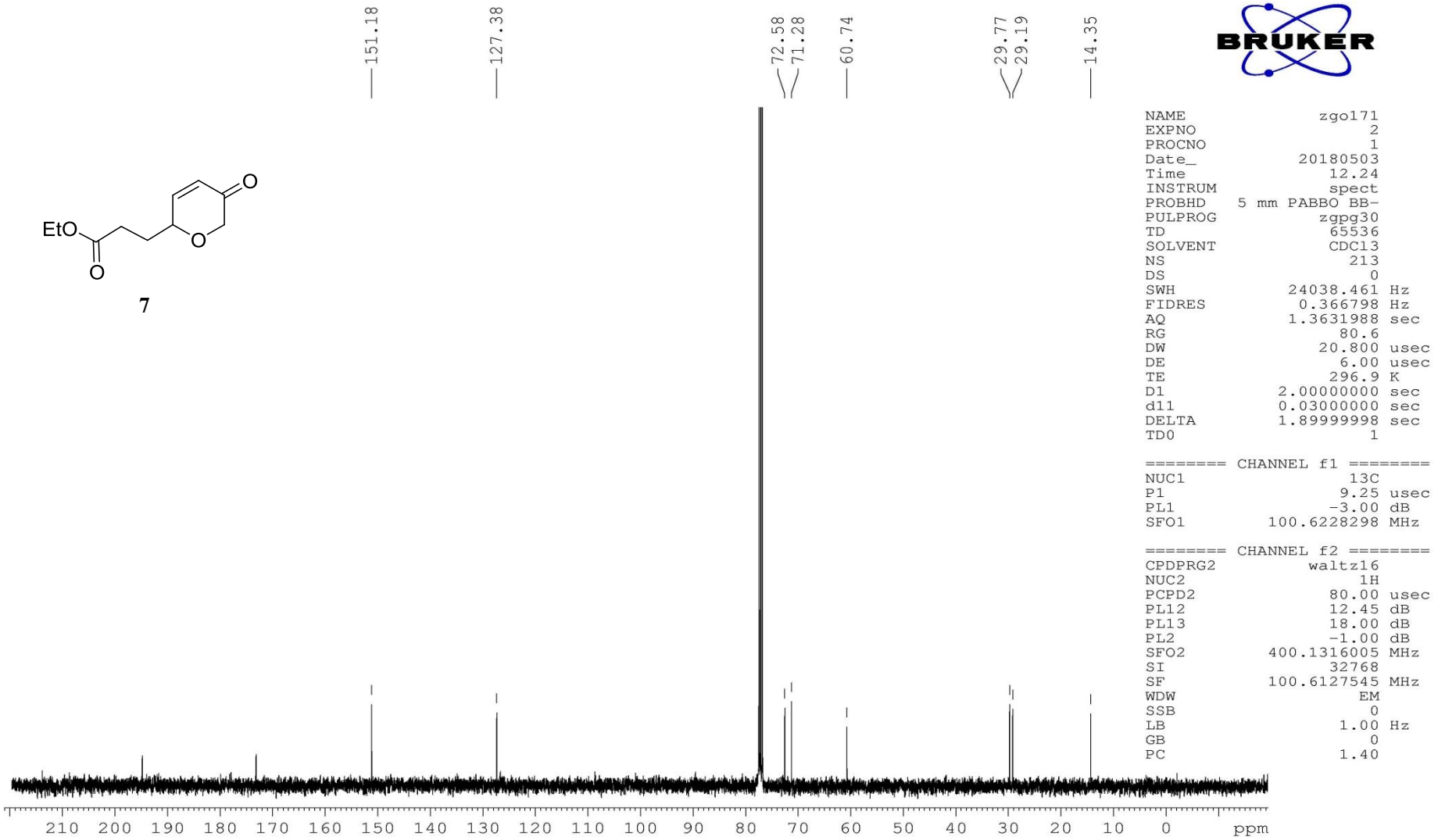

6.9084
6.8993
6.8829
6.8738
6.3907
6.3816
6.2209
6.1954
4.3848
4.3778
2.4882
2.4814
2.4709
2.4641
2.4536
2.4467
1.5323
1.0564
1.0389
0.9037
0.8867
BocO
O
O
5
BRUKER
NAME zgp147-1
EXPNO 1
PROCNO 1
Date_ 20180727
Time 22.49
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 88.84
DW 62.400 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.30 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.00
1.05
1.00
0.90
0.10
0.96
9.58
3.21
3.01

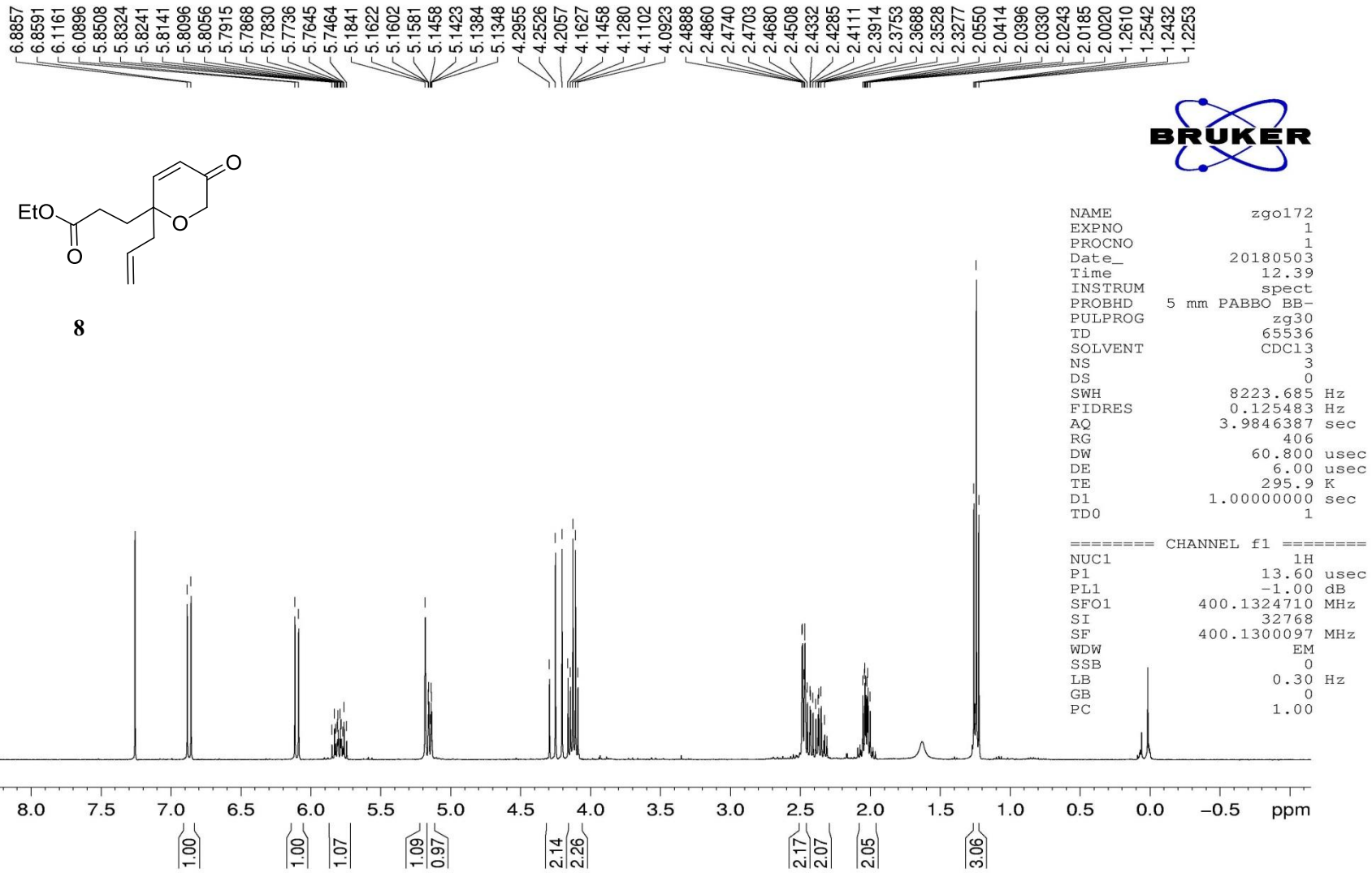

195.51
151.83
140.82
129.40
89.41
83.47
79.71
28.60
27.66
18.65
15.89
BocO
O
O
5
BRUKER
NAME zgp147-1
EXPNO 2
PROCNO 1
Date_ 20180727
Time 22.52
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 51
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

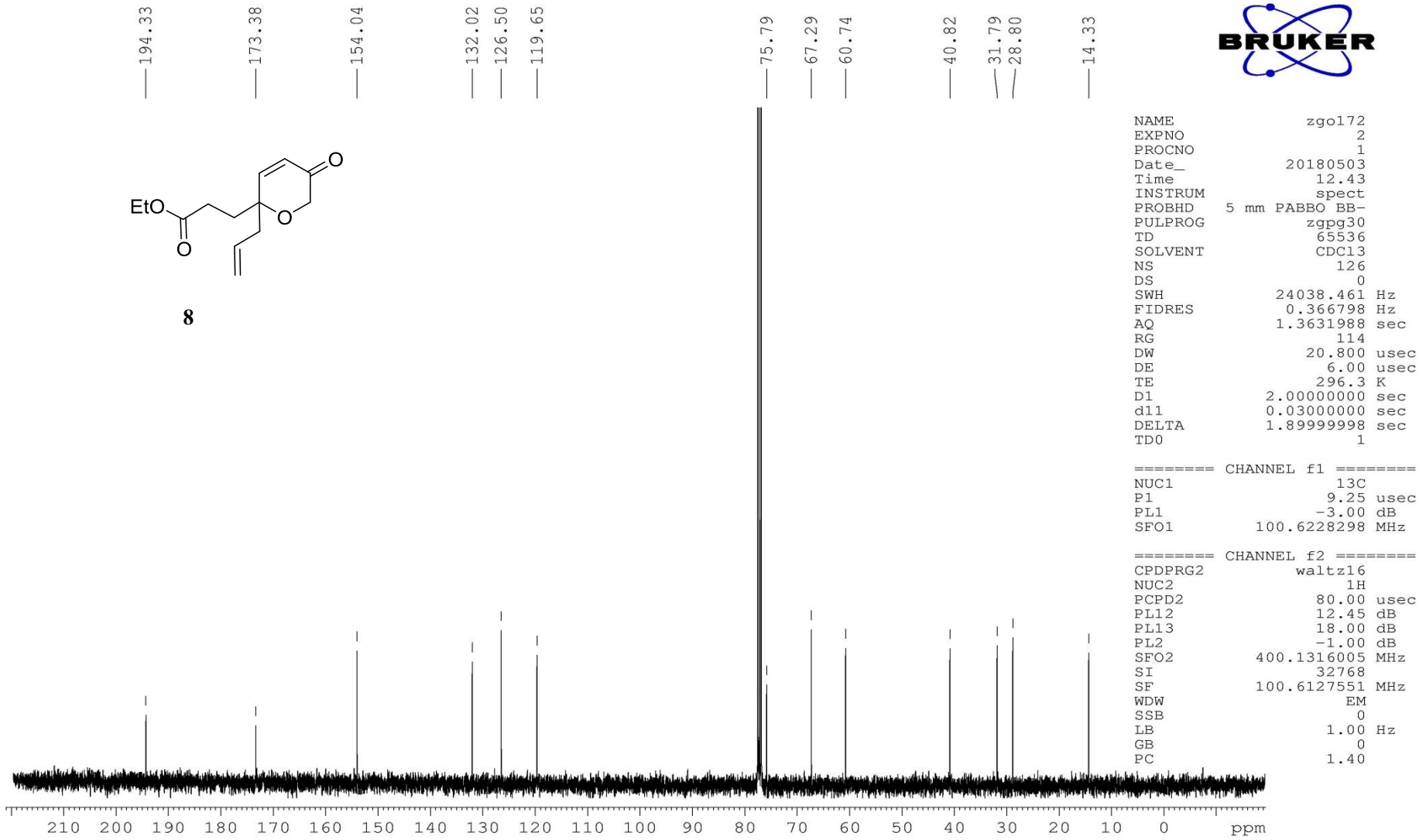

6.9027
6.8989
6.8770
6.8732
6.8672
6.8583
6.8418
6.8328
6.1477
6.1435
6.1220
6.1178
6.1042
6.0788
6.0017
5.9886
5.9757
5.9622
5.9590
5.9458
5.9355
5.9328
5.9197
5.3713
5.3675
5.3638
5.3600
5.3582
5.3541
5.3285
5.3246
5.3150
5.3111
5.2929
5.2841
5.2732
5.2693
5.2655
5.2615
5.2473
5.2434
5.2397
5.2358
4.4037
4.3908
4.2906
4.2835
4.2285
4.2125
4.1967
4.1615
4.1457
3.8465
3.8435
3.8365
3.8336
2.4549
2.4480
2.4380
1.0771
1.0591
1.0570
1.0395
0.9679
0.9509
0.8923
0.8753
BRUKER
O
O
O
6
NAME zgp151-2
EXPNO 1
PROCNO 1
Date_ 20180729
Time 22.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 2
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 575
DW 60.800 usec
DE 6.00 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.80 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
ppm
0.99
1.00
1.17
1.12
2.00
0.58
0.87
1.09
0.57
1.01
3.08
3.31


196.07
195.71
146.42
142.64
133.04
128.89
127.74
117.43
94.65
91.49
82.48
77.50
68.85
68.80
28.59
27.73
18.52
18.45
16.15
15.36
O
O
O
6
BRUKER
NAME zgp151-2
EXPNO 2
PROCNO 1
Date_ 20180729
Time 22.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 169
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 114
DW 20.800 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.60 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 14.39 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6128330 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.2599
6.9638
6.9592
6.9377
6.9330
6.1666
6.1607
6.1404
6.1347
4.4069
4.4017
4.3966
4.3911
4.3858
4.3803
4.3753
4.3701
4.2778
4.2370
4.1742
4.1563
4.1385
4.1206
4.0990
4.0945
4.0582
4.0536
2.5177
2.5102
2.4988
2.4940
2.4912
2.4797
2.4751
2.0991
2.0888
2.0825
2.0721
2.0634
2.0531
2.0444
2.0340
2.0204
2.0042
2.0013
1.9992
1.9849
1.9832
1.9801
1.9684
1.9639
1.9472
1.5866
1.2802
1.2623
1.2522
1.2445
BRUKER
EtO
O
O
O
7
NAME zgo171
EXPNO 1
PROCNO 1
Date_ 20180503
Time 12.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 645
DW 60.800 usec
DE 6.00 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
ppm
1.00
0.98
1.02
1.02
1.99
1.02
1.92
2.06
3.18


151.18
127.38
72.58
71.28
60.74
29.77
29.19
14.35
EtO
O
O
O
7
NAME zgo171
EXPNO 2
PROCNO 1
Date_ 20180503
Time 12.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 213
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 80.6
DW 20.800 usec
DE 6.00 usec
TE 296.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127545 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


6.8857
6.8591
6.1161
6.0896
5.8508
5.8324
5.8241
5.8141
5.8096
5.8056
5.7915
5.7868
5.7830
5.7736
5.7645
5.7464
5.1841
5.1622
5.1602
5.1581
5.1458
5.1423
5.1384
5.1348
4.2955
4.2526
4.2057
4.1627
4.1458
4.1280
4.1102
4.0923
2.4888
2.4860
2.4740
2.4703
2.4680
2.4508
2.4332
2.4285
2.4111
2.3914
2.3753
2.3688
2.3528
2.3277
2.0550
2.0414
2.0396
2.0330
2.0243
2.0185
2.0020
1.2610
1.2542
1.2432
1.2253
BRUKER
EtO
O
O
O
8
NAME zgo172
EXPNO 1
PROCNO 1
Date_ 20180503
Time 12.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 406
DW 60.800 usec
DE 6.00 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
ppm
1.00
1.00
1.07
1.09
0.97
2.14
2.26
2.17
2.07
2.05
3.06


194.33
173.38
154.04
132.02
126.50
119.65
75.79
67.29
60.74
40.82
31.79
28.80
14.33
EtO
O
O
O
8
BRUKER
NAME zgo172
EXPNO 2
PROCNO 1
Date_ 20180503
Time 12.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 126
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 114
DW 20.800 usec
DE 6.00 usec
TE 296.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127551 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm