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Supporting Information

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Palladium(II)/Brønsted Acid-Catalyzed Enantioselective Oxidative Carbocyclization–Borylation of Enallenes**

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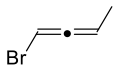
General remarks

Unless otherwise noted, all the reactions were run under argon with anhydrous conditions. All reagents were used as received from commercial suppliers. 1,4-Benzoquinone (BQ) was recrystallized from *n*-hexane before use. Phosphorus(V) oxychloride ($POCl_3$) was distilled over Na under argon and stored in a desiccator in the fridge. Arene-type solvents were dried over activated 4Å molecular sieves under argon. Room temperature is 23 ± 1 ^oC. Reactions were monitored using E. Merck silica gel 60 F254 plates (TLC analysis). TLC plates were visualized with UV light (254 nm), iodine, $KMnO_4$ stain or CAM stain (Cerium Ammonium Molybdate). Flash chromatography was carried out with 60Å (particle size 35-70 μm) normal flash silica gel. NMR spectra were recorded at 400 MHz (H) and at 100 MHz (C), respectively. Chemical shifts (δ) are reported in ppm, using the residual solvent peak in $CDCl_3$ (H: δ = 7.26 ppm and C: δ = 77.16 ppm) as internal standard, and coupling constants (J) are given in Hz. HRMS were recorded using ESI-TOF techniques. The enantiomeric excess of compounds was determined by chiral HPLC using the corresponding racemic compounds as the reference.

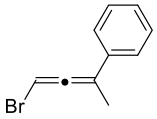
Experimental section

Starting materials and chiral catalysts

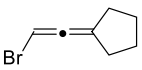
Allenyl bromides were prepared based on the reported protocols.[1] Due to the instability, all allenyl bromides were used immediately after the preparation.



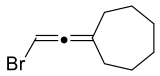
^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.91 (dq, J = 5.7, 2.4 Hz, 1H), 5.34 (dq, J = 7.3, 5.7 Hz, 1H), 1.82 (dd, J = 7.3, 2.4 Hz, 3H).



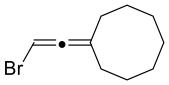
^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.46 – 7.44 (m, 2H), 7.39 – 7.35 (m, 2H), 7.31 – 7.27 (m, 1H), 6.25 (q, J = 2.1 Hz, 1H), 2.25 (d, J = 2.1 Hz, 3H).



^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.93 (quintet, J = 3.0 Hz, 1H), 2.58 – 2.45 (m, 4H), 1.75 – 1.69 (m, 4H).

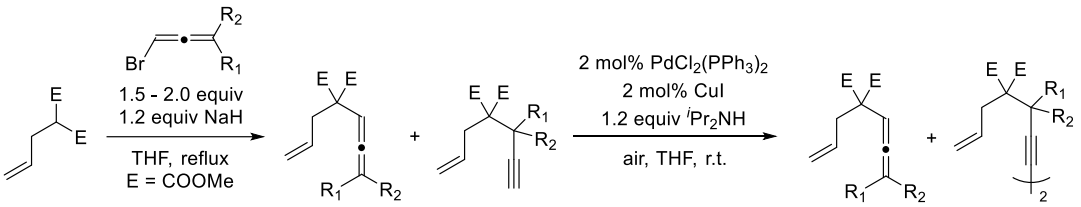


^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.81 (quintet, J = 1.8 Hz, 1H), 2.41 – 2.37 (m, 4H), 1.68 – 1.64 (m, 4H), 1.59 – 1.53 (m, 4H).

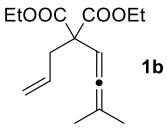


^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.85 (quintet, J = 1.6 Hz, 1H), 2.31 – 2.26 (m, 4H), 1.71 – 1.66 (m, 4H), 1.56 – 1.50 (m, 6H).

Enallenes were prepared according to our previous reports.[2,3] A general procedure is shown below.



New enallene substrates are listed below.

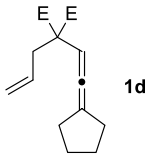


From diethyl allylmalonate (15 mmol), enallene **1b** was obtained as a light yellow oil (1.40 g, 35% for two steps).

^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.71 (ddt, J = 17.1, 10.0, 7.1 Hz, 1H), 5.51 (quintet, J = 2.9 Hz, 1H), 5.10 – 5.03 (m, 2H), 4.22 – 4.14 (m, 4H), 2.73 (dt, J = 7.1, 1.1 Hz, 2H), 1.69 (d, J = 2.9 Hz, 6H), 1.25 (t, J = 7.1 Hz, 6H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ 201.9, 170.4 (2C), 133.1, 118.6, 99.9, 88.5, 61.6 (2C), 58.1, 38.9, 20.2 (2C), 14.2 (2C).

HRMS (ESI) m/z for $C_{15}H_{22}O_4Na$ $[M+Na]^+$ calcd 289.1410, found 289.1399.

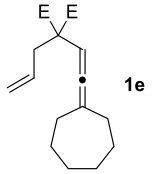


From dimethyl allylmalonate (10 mmol), enallene **1d** was obtained as a light yellow oil (1.27 g, 48% for two steps).

^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.71 (ddt, J = 17.1, 10.1, 7.2 Hz, 1H), 5.57 (quintet, J = 4.0 Hz, 1H), 5.10 – 5.04 (m, 2H), 3.72 (s, 6H), 2.74 (dt, J = 7.2, 1.2 Hz, 2H), 2.39 – 2.34 (m, 4H), 1.69 – 1.65 (m, 4H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ 197.5, 170.8 (2C), 133.2, 118.6, 108.7, 90.7, 58.7, 52.7 (2C), 38.9, 31.1 (2C), 27.1 (2C).

HRMS (ESI) m/z for $C_{15}H_{20}O_4Na$ $[M+Na]^+$ calcd 287.1254, found 287.1256.

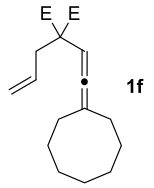


From dimethyl allylmalonate (8.8 mmol), enallene **1e** was obtained as a light yellow oil (1.35 g, 53% for two steps).

^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.73 (ddt, J = 17.1, 10.1, 7.2 Hz, 1H), 5.50 (quintet, J = 5.50 Hz, 1H), 5.11 – 5.04 (m, 2H), 3.72 (s, 6H), 2.75 (dt, J = 7.2, 1.2 Hz, 2H), 2.27 – 2.23 (m, 4H), 1.64 – 1.60 (m, 4H), 1.55 – 1.52 (m, 4H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ 201.8, 170.9 (2C), 133.2, 118.6, 109.4, 88.1, 58.5, 52.7 (2C), 39.2, 32.2 (2C), 29.5 (2C), 28.5 (2C).

HRMS (ESI) m/z for $C_{17}H_{24}O_4Na$ $[M+Na]^+$ calcd 315.1567, found 315.1571.

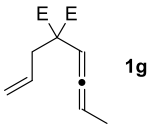


From dimethyl allylmalonate (2.6 mmol), enallene **1f** was obtained as a light yellow oil (455 mg, 57% after two steps).

^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.74 (ddt, J = 17.1, 10.1, 7.2 Hz, 1H), 5.55 (quintet, J = 2.2 Hz, 1H), 5.11 – 5.04 (m, 2H), 3.72 (s, 6H), 2.75 (dt, J = 7.2, 1.1 Hz, 2H), 2.20 – 2.15 (m, 4H), 1.65 – 1.51 (m, 10 H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ 202.0, 170.9 (2C), 133.2, 118.6, 109.6, 88.6, 58.4, 52.7 (2C), 39.4, 31.3 (2C), 27.0 (2C), 26.8 (2C), 26.0.

HRMS (ESI) m/z for $C_{18}H_{26}O_4Na$ $[M+Na]^+$ calcd 329.1723, found 329.1740.

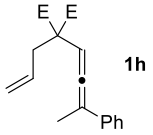


From dimethyl allylmalonate (8 mmol), enallene **1g** was obtained as a light yellow oil (415 mg, 23% for two steps). **Note:** After the homo-coupling reaction, enallene **1g** was further purified by Kugelrohr distillation.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.69 (ddt, J = 17.1, 10.0, 7.2 Hz, 1H), 5.59 (dq, J = 6.4, 3.2 Hz, 1H), 5.31 (dq, J = 7.1, 6.4 Hz, 1H), 5.10 – 5.05 (m, 2H), 3.72 (s, 3H), 3.71 (s, 3H), 2.74 (dt, J = 7.2, 1.1 Hz, 2H), 1.66 (dd, J = 7.2, 3.2 Hz, 3H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ 204.5, 170.6, 170.5, 132.9, 118.9, 90.3, 89.8, 58.0, 52.8, 52.8, 38.7, 13.9.

HRMS (ESI) m/z for $C_{12}H_{16}O_4Na$ $[M+Na]^+$ calcd 247.0941, found 247.0953.



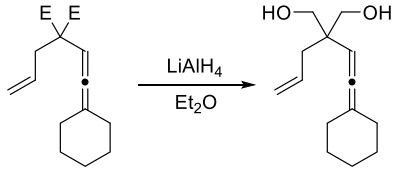
1h

From dimethyl allylmalonate (10 mmol), enallene **1h** was obtained as a light orange oil (1.49 g, 50% for two steps).

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.39 – 7.37 (m, 2H), 7.33 – 7.30 (m, 2H), 7.22 (m, 1H), 5.97 (q, J = 2.9 Hz, 1H), 5.74 (ddt, J = 17.0, 10.1, 7.2 Hz, 1H), 5.11 – 5.05 (m, 2H), 3.75 (s, 3H), 3.70 (s, 3H), 2.80 (dt, J = 7.2, 1.1 Hz, 1H), 2.11 (d, J = 2.9 Hz, 3H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ 204.4, 170.5, 170.5, 136.1, 132.8, 128.5 (2C), 127.3, 126.0 (2C), 119.1, 105.2, 92.6, 58.8, 52.9, 52.8, 39.3, 16.8.

HRMS (ESI) m/z for $C_{18}H_{20}O_4Na$ $[M+Na]^+$ calcd 323.1254, found 323.1237.



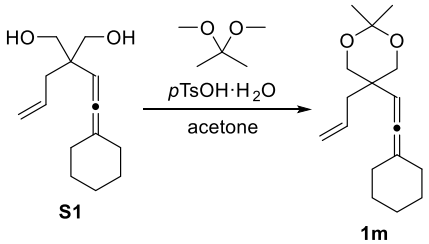
1c **S1**

To a suspension of $LiAlH_4$ (314 mg, 8.4 mmol) in Et_2O (10 mL) was added a solution of enallene **1a** (769 mg, 2.8 mmol) in Et_2O (10 mL) dropwise followed by vigorous stirring for 2 h at room temperature. The reaction mixture was diluted with Et_2O and quenched carefully with $Na_2SO_4 \cdot 10H_2O$/celite (1:1 mixture). Filtration and concentration gave a residue, which was purified by flash column chromatography (petroleum ether/EtOAc 4/1) to give **S1** as a colorless oil (454 mg, 73%).

^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.85 (ddt, J = 17.1, 10.0, 7.4 Hz, 1H), 5.14 – 5.07 (m, 2H), 4.85 (quintet, J = 2.1 Hz, 1H), 3.64 (dd, J = 11.1, 5.7 Hz, 2H), 3.59 (dd, J = 11.1, 6.8 Hz, 2H), 2.20 (dt, J = 7.4, 1.1 Hz, 2H), 2.14 – 2.10 (m, 4H), 2.02 (d, J = 5.7 Hz, 1H), 2.00 (d, J = 6.8 Hz, 1H), 1.68 – 1.60 (m, 2H), 1.56 – 1.47 (m, 4H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ 198.3, 134.4, 117.9, 105.2, 90.7, 68.1 (2C), 44.9, 38.0, 31.9 (2C), 27.5 (2C), 26.1.

HRMS (ESI) m/z for $C_{14}H_{22}O_2Na$ $[M+Na]^+$ calcd 245.1512, found 245.1508.



S1 **1m**

To a solution of **S1** (505 mg, 2.27 mmol) in acetone (11 mL) was added 2,2-dimethoxypropane (0.7 mL, 5.7 mmol) and pTsOH·H_2O (9 mg, 0.05 mmol). The reaction mixture was stirred at room temperature for 3 h and

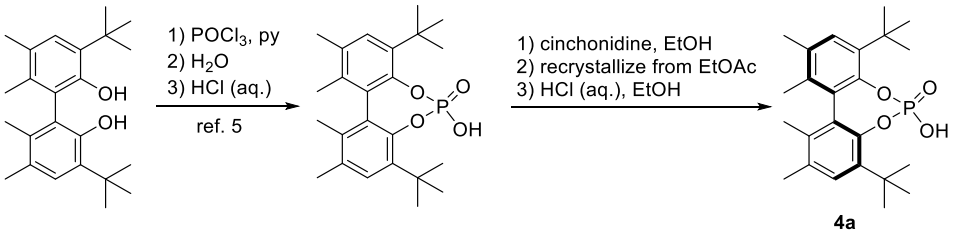
quenched with sat. $NaHCO_3$ (aq.). Acetone was evaporated and the residue was extracted with Et_2O, washed with brine, dried over $MgSO_4$. Concentration gave pure **1m** as a colorless oil (588 mg, 99%).

^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.81 (ddt, J = 17.0, 10.0, 7.4 Hz, 1H), 5.14 – 5.08 (m, 2H), 4.83 (quintet, J = 2.0 Hz, 1H), 3.69 (d, J = 11.8 Hz, 2H), 3.57 (d, J = 11.8 Hz, 2H), 2.36 (dt, J = 7.4, 1.1 Hz, 2H), 2.18 – 2.05 (m, 4H), 1.68 – 1.61 (m, 2H), 1.59 – 1.46 (m, 4H), 1.42 (s, 3H), 1.42 (s, 3H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ 198.3, 134.3, 118.1, 105.2, 97.9, 90.8, 67.8 (2C), 38.3, 37.1, 31.8 (2C), 27.6 (2C), 26.8, 26.2, 21.1.

HRMS (ESI) m/z for $C_{17}H_{26}O_2Na$ $[M+Na]^+$ calcd 285.1825, found 285.1814.

C_2-symmetric chiral phosphoric acids were prepared according to the literature.[4] Biphenol-type chiral phosphoric acids were prepared based on the reported protocol, with further modification.[5,6]



At 80 ^{o}C, racemic biphenol phosphoric acid (6.08 g, 14.6 mmol) was dissolved in absolute EtOH (150 mL). (–)-cinchonidine (4.48 g, 96%, 14.6 mmol) was added in one-portion and the mixture was stirred at room temperature for 2 h. Concentration of the reaction mixture gave the crude diastereomers (1:1 ratio), which was used directly for the next step.

The crude diastereomers was dissolved in minimum amount of boiling EtOAc and heated at 80 ^{o}C until white solid precipitated. The precipitate was filtered off when hot to give the pure complex of (*S*)-phosphoric acid **4a** & (–)-cinchonidine. The complex was dissolved in absolute EtOH followed by the addition of 50 mL 6N HCl (aq.). The biphasic mixture was stirred vigorously at room temperature for 1 h and white precipitation was filtered off and washed with deionized water. The solid was collected and dried under vacuum to give enantiopure (*S*)-phosphoric acid **4a** as a white powder (2.05 g, 34% based on the racemic biphenol phosphoric acid, theoretical yield = 50%).

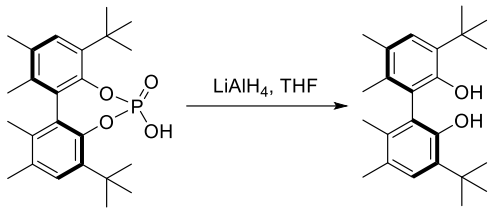
^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.20 (s, 2H), 2.25 (s, 6H), 1.83 (s, 6H), 1.46 (s, 18H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ 144.7 (d, J = 7.0 Hz, 2C), 137.9 (d, J = 4.2 Hz, 2C), 135.2 (d, J = 1.4 Hz, 2C), 133.7 (d, J = 1.8 Hz, 2C), 129.2 (2C), 128.7 (d, J = 1.8 Hz, 2C), 35.0 (2C), 31.6 (6C), 20.5 (2C), 16.8 (2C).

^{31}P NMR ($CDCl_3$, 162 MHz): δ -0.56.

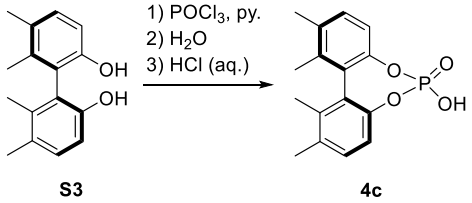
$[\alpha]_D^{23}$ = + 122.9 (c = 1.0, $CHCl_3$).

HRMS (ESI) m/z for $C_{24}H_{32}O_4P$ $[M-H]^-$ calcd 415.2044, found 415.2059.



At 0 °C, to a solution of (*S*)-phosphoric acid **4a** (2.05 g, 4.9 mmol) in THF (49 mL) was added $LiAlH_4$ (651 mg, 17.2 mmol) portionwise followed by reflux for 18 h. The reaction mixture was diluted with THF and quenched carefully with $Na_2SO_4 \cdot 10H_2O$/celite (1:1 mixture). Filtration and concentration gave pure (*S*)-biphenol **S2** as a white solid (1.26 g, 73%). Analytical data was in accordance with the literature.[5]

The debutylated biphenol **S3** was prepared from **S2** according to the literature.[7]



To a solution of (*S*)-biphenol **S3** (469 mg, 1.94 mmol) in pyridine (20 mL) was added $POCl_3$ (0.36 mL, 3.88 mmol) dropwise. The reaction mixture was heated at reflux for 3 h and cooled to room temperature. Deionized water (5 mL) was added dropwise to the reaction mixture followed by reflux for additional 4 h. Pyridine was removed by rotary evaporation and the residue was dissolved in DCM and acidified with 4N HCl (aq.). The aqueous phase was extracted thoroughly with DCM and the combined organic phase was washed with 4N HCl (aq.), dried over Na_2SO_4, and concentrated. The residue was purified by flash column chromatography (DCM/MeOH 5/1 to 2/1) to give a white solid. The white solid was dissolved in DCM, acidified with 4N HCl (aq.) and dried over Na_2SO_4. Removal of the solvent gave pure (*S*)-phosphoric acid **4c** as a white powder (412 mg, 70%).

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.20 (d, J = 8.1 Hz, 2H), 7.09 (d, J = 8.1 Hz, 2H), 2.31 (s, 6H), 2.04 (s, 6H).

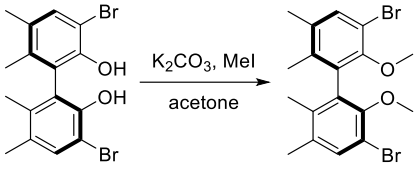
^{13}C NMR ($CDCl_3$, 100 MHz): δ 146.7 (d, J = 8.6 Hz, 2C), 137.6 (2C), 135.1 (d, J = 2.0 Hz, 2C), 130.6 (2C), 127.1 (2C), 118.3 (d, J = 3.7 Hz, 2C), 20.4 (2C), 17.7 (2C).

^{31}P NMR ($CDCl_3$, 162 MHz): δ -3.34.

$[\alpha]_D^{23}$ = + 157.0 (c = 0.1, $CHCl_3$).

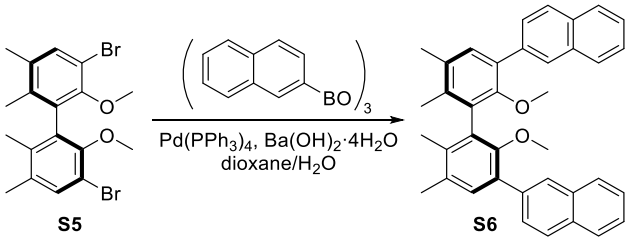
HRMS (ESI) m/z for $C_{16}H_{16}O_4P$ $[M-H]^-$ calcd 303.0792, found 303.0795.

The brominated (*S*)-biphenol **S4** was prepared according to the literature.[7]



S4 **S5**

To a solution of **S4** (166 mg, 0.41 mmol) in acetone (5 mL) was added K_2CO_3 (198 mg, 1.44 mmol) and MeI (0.15 mL, 2.46 mmol) and the mixture was heated at reflux for 18 h. Acetone was removed by rotary evaporation and the residue was dissolved with DCM and washed with deionized water. The aqueous phase was extracted thoroughly with DCM and the combined organic phase was washed with brine, dried over Na_2SO_4, and concentrated. The residue was obtained as a white solid (177 mg, 99%) and used directly for the next step. Analytical data of **S5** was in accordance with the literature.



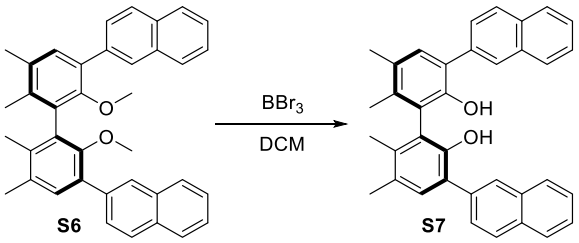
S5 **S6**

To a mixture of **S5** (177 mg, 0.41 mmol), 2-naphthylboroxine (663 mg, 1.44 mmol), $Pd(PPh_3)_4$ (47 mg, 0.04 mmol) and $Ba(OH)_2 \cdot 4H_2O$ (453 mg, 1.44 mmol) was added freshly degassed dioxane/H_2O (4 mL, 3/1). The reaction mixture was heated at 90 °C for 18 h. Dioxane was removed by rotary evaporation and the residue was dissolved in DCM and washed with 2 N HCl (aq.). The aqueous phase was extracted thoroughly with DCM and the combined organic phase was washed brine, dried over Na_2SO_4, and concentrated. The residue was purified by flash column chromatography (pentane/DCM 5/1) to give **S6** as a white solid (209 mg, 98 %).

^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.18 (br. s, 2H), 7.97 – 7.89 (m, 8H), 7.56 – 7.53 (m, 4H), 7.41 (s, 2H), 3.32 (s, 6H), 2.46 (s, 6H), 2.13 (s, 6H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ 153.7, 137.1, 135.9, 133.7, 132.6, 132.5, 132.3, 131.7, 131.5, 128.2, 127.9, 127.7, 127.7, 127.6, 126.0, 125.8, 60.4, 20.3, 17.0.

$[\alpha]_D^{23}$ = + 220.0 (c = 2.0, $CHCl_3$).



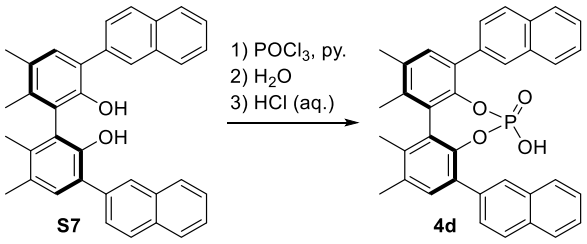
S6 S7

At 0 °C, to a solution of **S6** (209 mg, 0.40 mmol) in DCM (4 mL) was added BBr_3 (0.84 mL, 1.0 M in hexane, 0.84 mmol) dropwise. The reaction was maintained at 0 °C for 3 h and quenched by slow addition of deionized water. The aqueous phase was extracted thoroughly with DCM and the combined organic phase was washed brine, dried over Na_2SO_4 and passed through a short silica column. Removal of the solvent gave pure **S7** as a white solid (193 mg, 98%).

^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.07 (br. s, 2H), 7.91 (d, J = 8.6 Hz, 2H), 7.89 – 7.85 (m, 4H), 7.76 (dd, J = 8.6, 1.8 Hz, 2H), 7.50 – 7.48 (m, 4H), 7.36 (s, 2H), 5.00 (s, 2H), 2.37 (s, 6H), 2.05 (s, 6H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.8, 136.7, 135.6, 133.7, 132.6, 132.5, 129.6, 128.2, 128.0, 128.0, 127.8 (2C), 126.2, 126.0, 125.7, 121.8, 20.0, 16.7.

$[\alpha]_D^{23}$ = + 252.6 (c = 0.8, $CHCl_3$).



S7 4d

To a solution of **S7** (347 mg, 0.7 mmol) in pyridine (7 mL) was added $POCl_3$ (0.13 mL, 0.14 mmol) dropwise. The reaction mixture was heated at 90 °C for 18 h and cooled to room temperature. Deionized water (5 mL) was added dropwise to the reaction mixture followed by heating at 90 °C for additional 8 h. Pyridine was removed by rotary evaporation and the residue was dissolved in DCM and acidified with 4N HCl (aq.). The aqueous phase was extracted thoroughly with DCM and the combined organic phase was washed with deionized water, dried over Na_2SO_4, and concentrated. The residue was purified by flash column chromatography (DCM/MeOH 20/1) to give a white solid. The white solid was dissolved in DCM, acidified with 4N HCl (aq.) and dried over Na_2SO_4. Removal of the solvent gave pure (*S*)-phosphoric acid **4d** as a white powder (367 mg, 94%).

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.86 (br. s, 2H), 7.70 – 7.67 (m, 2H), 7.57 – 7.54 (m, 2H), 7.53 – 7.51 (m, 2H), 7.50 – 7.46 (m, 2H), 7.31 (br. s, 2H), 7.26 – 7.24 (m, 4H), 2.40 (s, 6H), 2.14 (s, 6H).

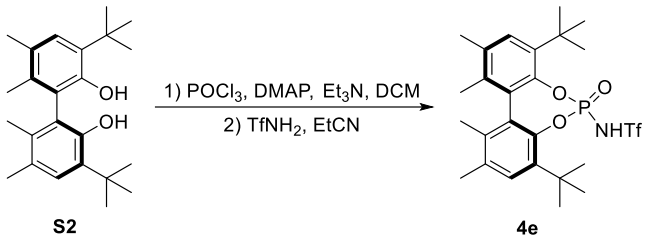
^{13}C NMR ($CDCl_3$, 100 MHz): δ 143.5 (d, J = 9.2 Hz, 2C), 136.9 (2C), 134.7 (2C), 134.5 (2C), 133.3 (2C), 132.6 (2C), 132.2 (2C), 131.6 (d, J = 9.2 Hz, 2C), 128.4 (2C), 128.4 (2C), 128.1 (2C), 127.6 (4C), 127.5 (2C), 125.7 (2C), 125.7 (2C), 20.5 (2C), 17.7 (2C).

^{31}P NMR ($CDCl_3$, 162 MHz): δ 1.17.

$[\alpha]_D^{23}$ = + 319.7 (c = 1.77, $CHCl_3$).

HRMS (ESI) m/z for $C_{36}H_{28}O_4P$ $[M-H]^-$ calcd 555.1731, found 555.1705.

(*S*)-Phosphoramide **4e** was prepared based on the literature protocol.[8]



S2 **4e**

At 0 °C, to a solution of **S2** (213 mg, 0.6 mmol) in DCM (3 mL) were added Et_3N (0.59 mL, 4.2 mmol), $POCl_3$ (67 μL, 0.72 mmol) and DMAP (147 mg, 1.2 mmol) at 0 °C. After stirring at room temperature for 1 h, EtCN (3 mL) was added followed by the addition of $TfNH_2$ (179 mg, 1.2 mmol). Then the reaction was heated 100 °C for 16 h. The reaction was quenched with deionized water, and extracted with Et_2O. The combined organic phase was washed with sat. $NaHCO_3$ (aq.), 4N HCl (aq.), dried over Na_2SO_4 and concentrated. The residue was purified by flash column chromatography (100% Et_2O) to give a pale white solid. The solid was dissolved in DCM, acidified with 4N HCl (aq.) and dried over Na_2SO_4. Removal of the solvent gave (*S*)-phosphoramide **4e** as a white powder (276 mg, 84%).

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.23 (s, 1H), 7.22 (s, 1H), 2.26 (s, 6H), 1.84 (s, 3H), 1.79 (s, 3H), 1.47 (s, 9H), 1.44 (s, 9H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ 144.0 (d, J = 10.5 Hz, 1C), 143.6 (d, J = 9.1 Hz, 1C), 138.3 (d, J = 4.3 Hz, 1C), 137.3 (d, J = 4.9 Hz, 1C), 135.8, 135.7, 134.6 (d, J = 1.5 Hz, 1C), 134.4 (d, J = 1.5 Hz, 1C), 129.4 (2C), 128.5, 128.3, 119.3 (q, J = 321.2 Hz, 1C), 119.3 (q, J = 321.2 Hz, 1C), 35.1, 34.8, 31.8 (3C), 31.3 (3C), 20.6, 20.5, 16.8, 16.7.

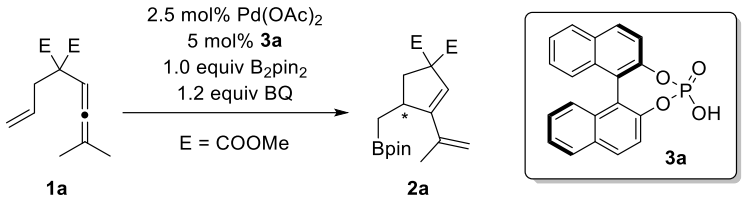
^{31}P NMR ($CDCl_3$, 162 MHz): δ -9.05.

$[\alpha]_D^{23}$ = + 122.9 (c = 1.0, $CHCl_3$).

HRMS (ESI) m/z for $C_{25}H_{32}O_5F_3NPS$ $[M-H]^-$ calcd 546.1696, found 546.1721.

Optimization of the reaction parameters.

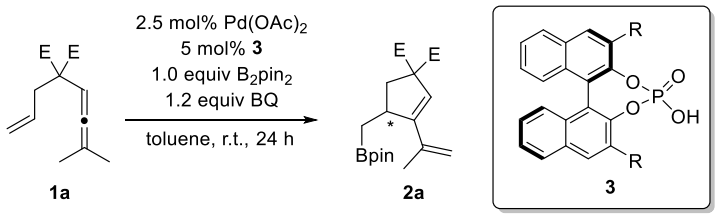
Initial screening of solvents.[a]



Entry	Solvent[b]	Time (h)	Yield (%)[c]	*ee* (%)[d]
1	toluene	24	> 95	30
2	acetone	70	75	8
3	DCE	70	76	8
4	DCM	70	70	8
5	EtOAc	70	85	10
6	THF	70	34	6
7	MeCN	70	0	–[e]
8	DMF	70	6	–[e]

(a) Reaction scale: Enallene **1a** (0.1 mmol) in the indicated solvent (1.0 mL) at room temperature. (b) Regular solvent. (c) Determined by ^{1}H NMR analysis of the crude mixture with anisole as the internal standard. (d) Determined by chiral HPLC. (e) Not determined.

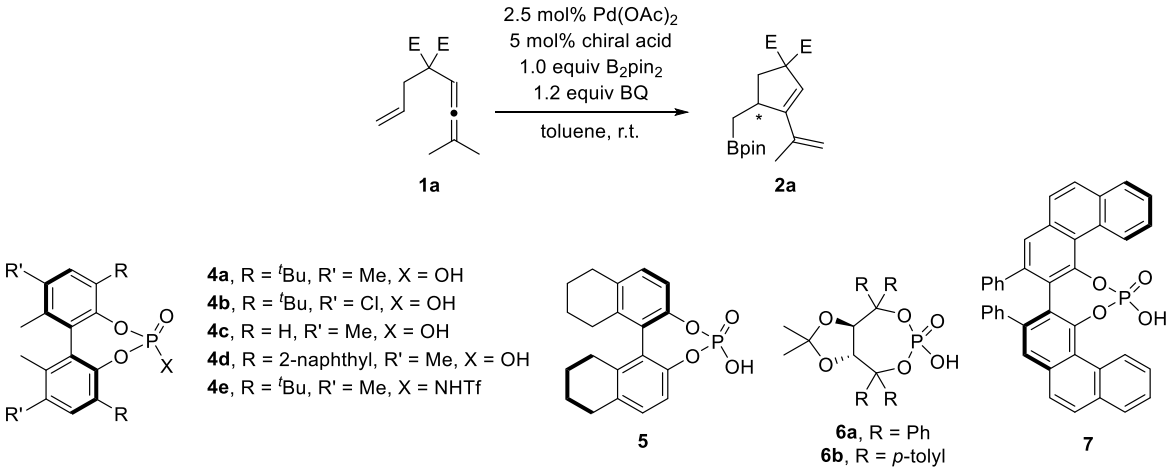
Screening of different (*R*)-BINOL phosphoric acids.[a]



Entry	R	Yield (%)[b]	*ee* (%)[c]
1	H, **3a**	> 95	30
2	Ph, **3b**	> 95	8
3	2-naphthyl, **3c**	> 95	45
4	9-anthracenyl, **3d**	67	6
5	TMS, **3e**	> 95	42
6	$SiPh_3$, **3f**	> 95	10
7	Si^tBuPh_2, **3g**	> 95	26
8	mesityl, **3h**	> 95	20
9[d]	2,4,6-tri-^{i}Pr-C_6H_2, **3i**	67	24
10	Me, **3j**	> 95	18
11	allyl, **3k**	> 95	45
12	CO^tBu, **3l**	> 95	24
13	3,5-di-CF_3-C_6H_3, **3m**	> 95	2
14	3,5-di-^{t}Bu-C_6H_3, **3n**	> 95	8

(a) Reaction scale: Enallene **1a** (0.1 mmol) in regular toluene (1.0 mL) at room temperature. (b) Determined by ^{1}H NMR analysis of the crude mixture with anisole as the internal standard. (c) Determined by chiral HPLC. (d) Reaction time is 16 h.

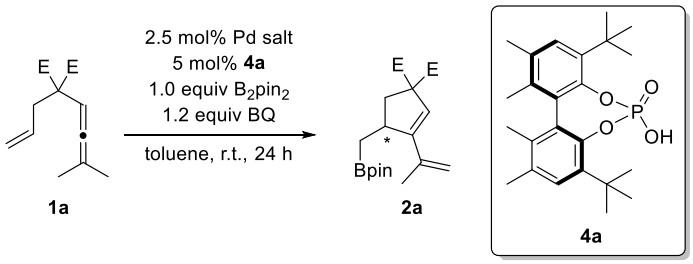
Screening of phosphoric acids with different backbones.[a]



Entry	Chiral phosphoric acid	Time (h)	Yield (%)[b]	*ee* (%)[c]
1	**4a**	24	> 95	77
2	**4b**	16	67	76
3	**4c**	16	> 95	40
4	**4d**	16	> 95	48
5	**4e**	48	< 5	–[d]
6	**5**	16	> 95	30
7	**6a**	40	68	20
8	**6b**	94	22	19
9[d]	**7**	60	70	40

(a) Reaction scale: Enallene **1a** (0.1 mmol) in regular toluene (1.0 mL) at room temperature. (b) Determined by ^{1}H NMR analysis of the crude mixture with anisole as the internal standard. (c) Determined by chiral HPLC. (d) Not determined.

Screening of different palladium salts.[a]

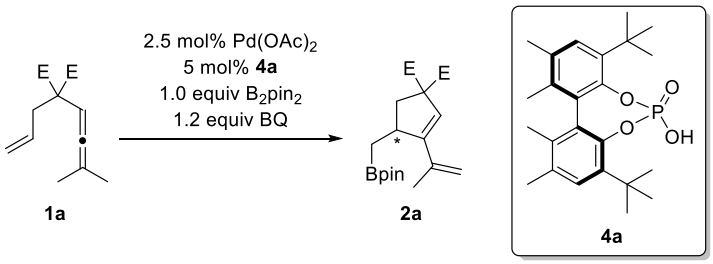


Entry	Pd salt	Yield (%)[b]	*ee* (%)[c]
1[d]	Pd_2dba_3	14	70
2[d]	$Pd_2dba_3 \cdot CHCl_3$	23	70
3	$Pd(PPh_3)_4$	48	47

4	Pd/C	0	–[e]
5	$PdCl_2$	0	–[e]
6	$PdCl_2(PPh_3)_2$	0	–[e]
7	$PdCl_2(PhCN)_2$	0	–[e]
8	$PdCl_2(DMSO)_2$	0	–[e]
9	$PdBr_2$	0	–[e]
10	$Pd(OAc)_2$	> 95	77
11[f]	$Pd(OAc)_2$	> 95	79
12[f]	$Pd(OOCCF_3)_2$	> 95	44

(a) Reaction scale: Enallene **1a** (0.1 mmol) in regular toluene (1.0 mL) at room temperature. (b) Determined by ^{1}H NMR analysis of the crude mixture with anisole as the internal standard. (c) Determined by chiral HPLC. (d) With 1.25 mol% of the Pd salt. (e) Not determined. (f) In anhydrous toluene under argon for 48 h.

Screening of arene-type solvents under inert & anhydrous conditions.[a,b]

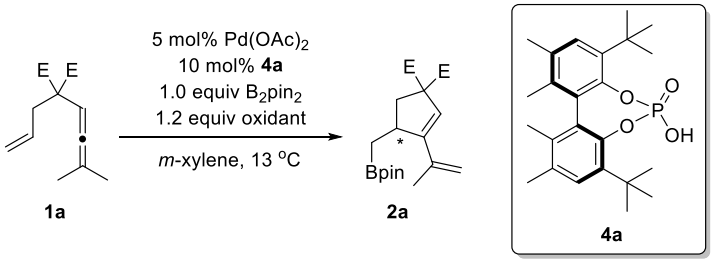


1a **2a** **4a**

Entry	Solvent	Temp (^{o}C)	Time (h)	Yield (%)[c,d]	*ee* (%)[e]
1	toluene	r.t.	48	> 95	79
2	toluene	13	84	71	80
3	benzene	13	84	85	73
4	$PhCF_3$	13	84	> 95	80
5	PhCl	13	60	> 95	73
6	*o*-xylene	13	84	> 95	79
7	*m*-xylene	13	84	85	80
8	*p*-xylene	13	84	85	77
9[f]	toluene	13	84	> 95	80
10[f]	$PhCF_3$	13	60	> 95	81
11[f]	*m*-xylene	13	84	> 95	83
12[g]	*m*-xylene	13	60	> 95	83
13[f,h]	*m*-xylene	13	36	> 95	83
14[f,h]	*m*-xylene	3	66	> 95	83
15[g,h]	*m*-xylene	3	48	> 95	83

(a) Reaction scale: Enallene **1a** (0.1 mmol) in the indicated solvent (1.0 mL). (b) In anhydrous solvent under argon. (c) Determined by ^{1}H NMR analysis of the crude mixture with anisole as the internal standard. (d) Isolated yield in parenthesis. (e) Determined by chiral HPLC. (f) Using $Pd(OAc)_2$ (5 mol%) and **4a** (10 mol%). (g) Using $Pd(OAc)_2$ (10 mol%) and **4a** (20 mol%). (h) In 0.5 mL of *m*-xylene.

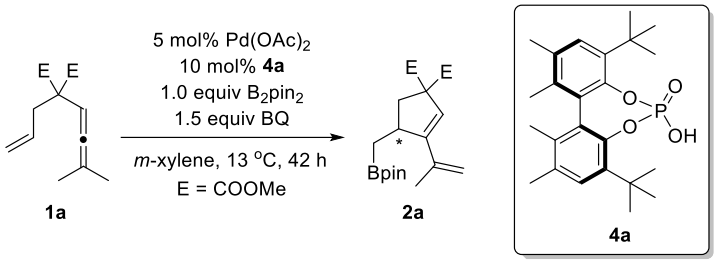
Screening of benzoquinone-type oxidants.[a]



Entry	Oxidant	Time (h)	Yield (%)[b,c]	ee (%)[d]
1	BQ	36	> 95	83
2	tetra-F-BQ	60	40	58
3	2,6-di-Me-BQ	36	> 95	76
4	2,6-di-OMe-BQ	60	> 95	78
5	2-Cl-BQ	60	80	34
6	2,6-di-^{t}Bu-BQ	60	47	80
7[e]	BQ	36	> 95	81
8[f]	BQ	36	> 95 (93)	83
8[f,g]	BQ	42	97 (93)	84
9[f,h]	BQ	48	94 (88)	84

(a) Reaction scale: Enallene **1a** (0.1 mmol) in anhydrous *m*-xylene solvent (0.5 mL) under argon. (b) Determined by ^{1}H NMR analysis of the crude mixture with anisole as the internal standard. (c) Isolated yield in parenthesis. (d) Determined by chiral HPLC. (e) Using BQ (1.0 equiv.). (f) With enallene **1a** (0.2 mmol) in anhydrous *m*-xylene solvent (1.0 mL) under argon. (g) Using BQ (1.5 equiv.). (h) Using BQ (1.8 equiv.).

General procedure for the Pd(II)/Brønsted acid-catalyzed enantioselective oxidative carbocyclization-borylation of enallenes

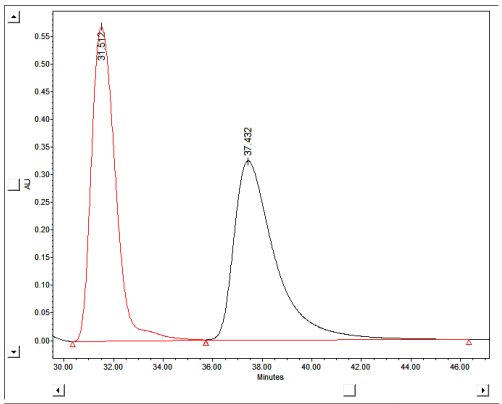


In a microwave vial was charged enallene **1a** (47.6 mg, 0.2 mmol), $Pd(OAc)_2$ (2.2 mg, 5 mol%), (*S*)-phosphoric acid **4a** (8.3 mg, 10 mol%), B_2pin_2 (51.8 mg, 98%, 0.2 mmol), BQ (32.4 mg, 0.3 mmol) and *m*-xylene (1 mL). The vial was sealed with a rubber cap and maintained at 13 °C for 42 h. The reaction was quenched with sat. $Na_2S_2O_3$ (aq.) and extracted with Et_2O. The combined organic phase was washed with brine, dried over Na_2SO_4 and concentrated. The residue was purified by flash column chromatography (pentane/Et_2O, 10/1) to afford **2a** (68 mg, 93%). *ee* = 84%.

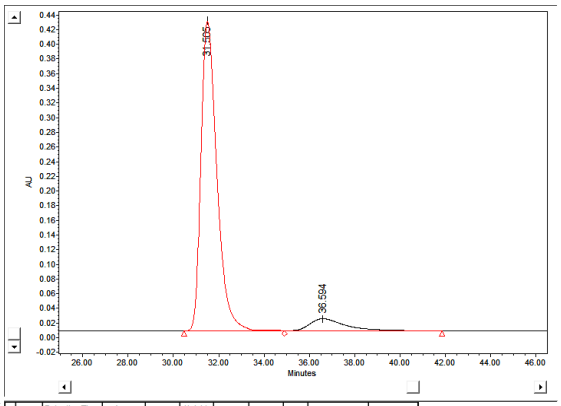
Spectral data was in accordance with the literature.[9]

NOTE: The carbon adjacent to boron may not have a visible peak in ^{13}C spectrum. However, HSQC experiments can clearly indicate the signals of those carbons.

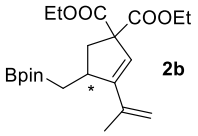
Retention time: 31.5 & 37.4 min, CHIRALCEL®-ID, *iso*-hexane/*iso*-propanol = 99/1, 25 °C, 0.3 mL/min.



	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		31.512	38024406	50.60	568109	BB			Unknown	
2		37.432	37128740	49.40	323350	Bb			Unknown	



	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		31.505	20507784	91.88	422411	bv			Unknown	
2		36.594	1811500	8.12	16619	vb			Unknown	



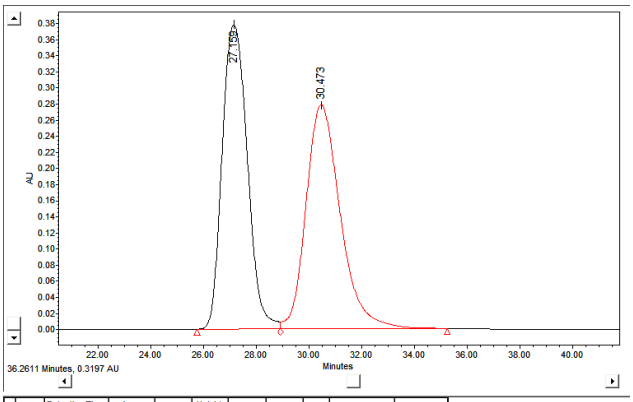
Reaction time: 42 h. Isolated yield: 69 mg (88%). *ee* = 84%.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.66 (s, 1H), 5.02 (br. s, 1H), 5.00 (br. s, 1H), 4.22 – 4.12 (m, 4H), 3.17 (m, 1H), 2.70 (dd, *J* = 13.8, 8.7 Hz, 1H), 2.25 (dd, *J* = 13.8, 3.0 Hz, 1H), 1.90 (br. s, 3H), 1.24 – 1.21 (m, 1H), 1.23 (t, *J* = 7.1 Hz, 6H), 1.23 (br. s, 6H), 1.22 (br. s, 6H), 0.75 (dd, *J* = 16.1, 11.6 Hz, 1H).

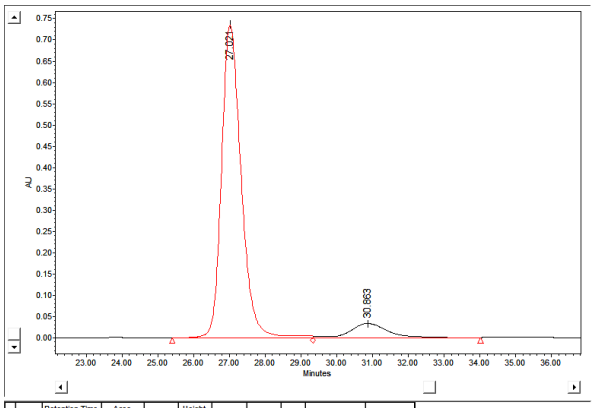
^{13}C NMR ($CDCl_3$, 100 MHz): δ 171.9, 171.6, 153.7, 138.0, 123.2, 115.3, 83.2 (2C), 65.7, 61.6, 61.5, 40.0, 39.6, 25.0 (2C), 24.8 (2C), 21.4, 18.1 (C–B), 14.1 (2C).

HRMS (ESI) m/z for $C_{21}H_{33}O_6BNa$ $[M+Na]^+$ calcd 415.2266, found 415.2246.

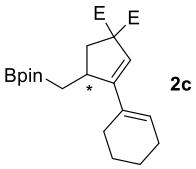
Retention time: 27.2 & 31.5 min, CHIRALCEL®-ID, *iso*-hexane/*iso*-propanol = 99/1, 25 °C, 0.3 mL/min.



	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		27.159	25111228	49.92	377802	BV			Unknown	
2		30.473	25195766	50.08	278013	Vb			Unknown	



	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		27.021	27841039	92.18	735264	bv			Unknown	
2		30.863	2363420	7.82	33107	vb			Unknown	



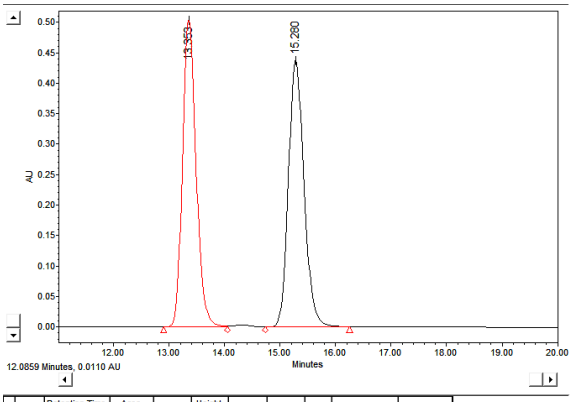
2c

Reaction time: 84 h. Isolated yield: 64 mg (79%). *ee* = 93%**.**

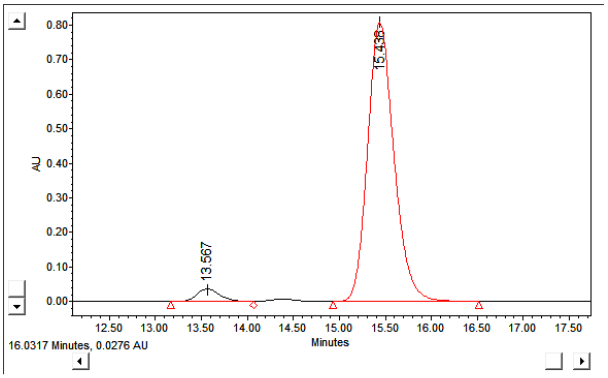
^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.82 (m, 1H), 5.55 (s, 1H), 3.71 (s, 3H), 3.70 (s, 3H), 3.17 (m, 1H), 2.68 (dd, J = 13.8, 6.9 Hz, 1H), 2.27 (dd, J = 13.8, 2.9 Hz, 2H), 2.12 (m, 3H), 1.66 (m, 3H), 1.57 (m, 2H), 1.24 (s, 6H), 1.24 (s, 6H), 0.72 (dd, J = 16.1, 11.8 Hz, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ 172.5, 172.3, 154.4, 131.6, 127.7, 119.4, 83.2 (2C), 65.4, 52.8 (2C), 39.7, 39.6, 26.6, 25.9, 25.1 (2C), 24.8 (2C), 22.8, 22.3.

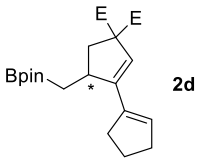
Retention time: 13.4 & 15.3 min, CHIRALCEL®-IC, *iso*-hexane/*iso*-propanol = 93/7, 30 °C, 0.5 mL/min.



	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		13.353	8542489	49.87	503269	BV			Unknown	
2		15.280	8588064	50.13	438445	VB			Unknown	



	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		13.567	629701	3.74	36391	BV			Unknown	
2		15.438	16193850	96.26	807764	BB			Unknown	



2d

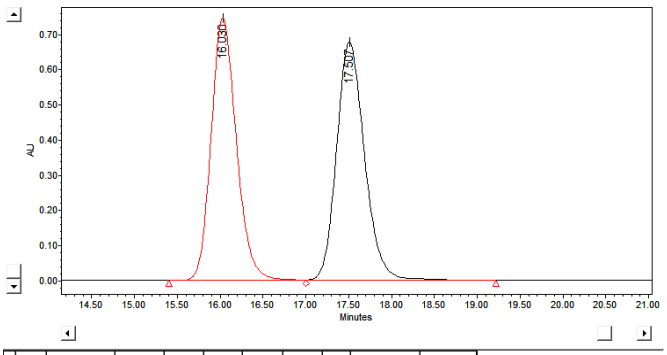
Reaction time: 72 h. Isolated yield: 65 mg (83%). *ee* = 92%.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.80 (m, 1H), 5.52 (s, 1H), 3.72 (s, 3H), 3.70 (s, 3H), 3.16 (m, 1H), 2.73 (dd, J = 13.8, 8.7 Hz, 1H), 2.55 – 2.35 (m, 4H), 2.26 (dd, J = 13.8, 3.5 Hz, 1H), 1.95 – 1.80 (m, 2H), 1.29 (dd, J = 16.0, 3.2 Hz, 1H), 1.24 (s, 6H), 1.24 (s, 6H), 0.77 (dd, J = 16.0, 11.6 Hz, 1H).

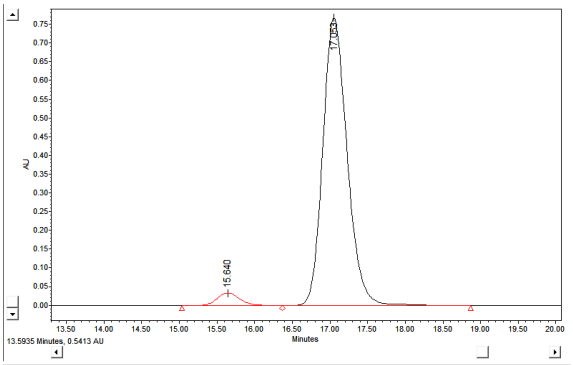
^{13}C NMR ($CDCl_3$, 100 MHz): δ 172.3, 172.2, 149.8, 138.0, 130.1, 122.1, 83.2 (2C), 65.5, 52.8 (2C), 40.8, 40.0, 33.5, 33.5, 25.1 (2C), 24.8 (2C), 23.1, 18.3 (C–B).

HRMS (ESI) m/z for $C_{21}H_{31}O_6BNa$ $[M+Na]^+$ calcd 413.2110, found 413.2130.

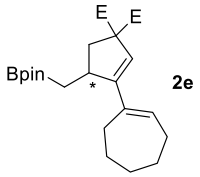
Retention time: 16.0 & 17.5 min, CHIRALCEL®-IC, *iso*-hexane/*iso*-propanol = 96/4, 30 °C, 0.5 mL/min.



	Name	Retention Time (min)	Area (µV*sec)	% Area	Height (µV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		16.030	15249445	49.82	745704	bv			Unknown	
2		17.507	15361733	50.18	677266	vb			Unknown	



	Name	Retention Time (min)	Area (µV*sec)	% Area	Height (µV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		15.640	683501	3.83	33594	bv			Unknown	
2		17.053	17169766	96.17	768123	vb			Unknown	



2e

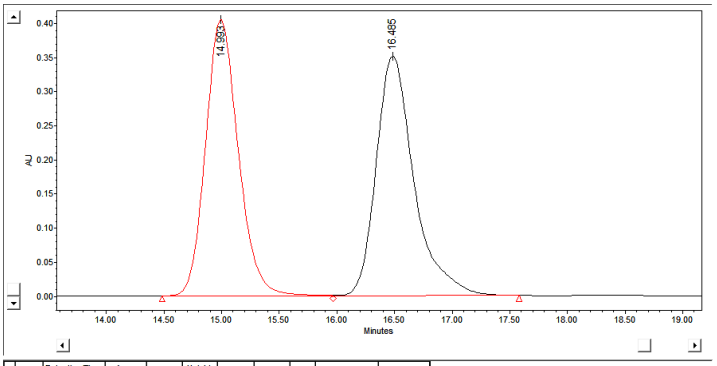
Reaction time: 72 h. Isolated yield: 57 mg (68%). *ee* = 92%.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.92 (t, J = 6.8 Hz, 1H), 5.56 (br. s, 1H), 3.72 (s, 3H), 3.70 (s, 3H), 3.17 – 3.11 (m, 1H), 2.74 (dd, J = 13.7, 6.8 Hz, 1H), 2.37 – 2.32 (m, 2H), 2.21 (dd, J = 13.7, 3.9 Hz, 1H), 2.21 – 2.17 (m, 2H), 1.75 – 1.72 (m, 2H), 1.50 – 1.46 (m, 4H), 1.24 (s, 6H), 1.24 (s, 6H), 1.22 (m, 1H), 0.69 (dd, J = 16.1, 11.6 Hz, 1H).

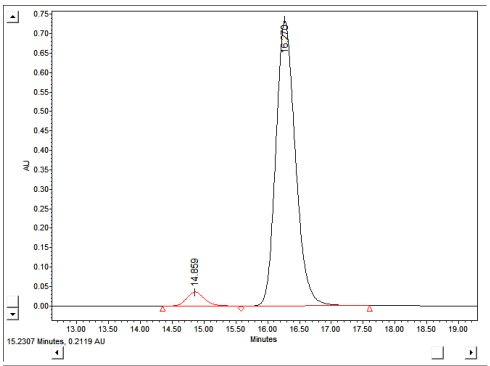
^{13}C NMR ($CDCl_3$, 100 MHz): δ 172.4, 172.4, 155.5, 139.2, 132.0, 119.8, 83.2 (2C), 65.3, 52.8, 52.8, 40.0, 40.0, 32.5, 31.0, 28.6, 26.8, 26.5, 25.1 (2C), 24.8 (2C).

HRMS (ESI) m/z for $C_{23}H_{35}O_6BNa$ $[M+Na]^+$ calcd 441.2423, found 441.2401.

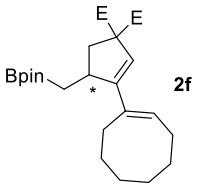
Retention time: 15.0 & 16.5 min, CHIRALCEL®-IC, *iso*-hexane/*iso*-propanol = 95/5, 30 °C, 0.5 mL/min.



	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		14.993	7983524	49.97	405083	BV			Unknown	
2		16.485	7991740	50.03	351353	VB			Unknown	



	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		14.859	685604	4.21	35933	bv			Unknown	
2		16.270	15584910	95.79	735820	vb			Unknown	



2f

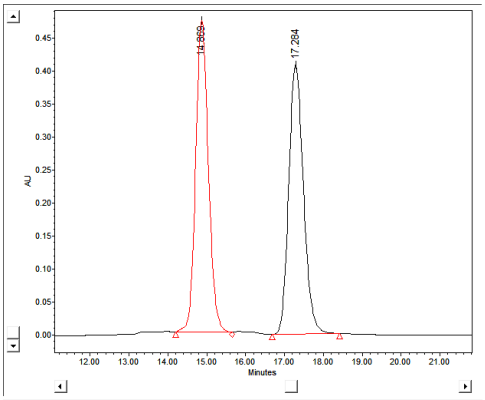
Reaction time: 96 h. Isolated yield: 74 mg (86%). *ee* = 88%.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.75 (t, J = 8.2 Hz, 1H), 5.61 (s, 1H), 3.71 (s, 3H), 3.70 (s, 3H), 3.20 – 3.14 (m, 1H), 2.69 (dd, J = 13.6, 8.4 Hz, 1H), 2.40 – 2.38 (m, 2H), 2.27 (dd, J = 13.6, 3.1 Hz, 1H), 2.22 – 2.16 (m, 2 H), 1.54 – 1.40 (m, 8H), 1.27 (dd, J = 16.2, 3.2 Hz, 1H), 1.25 (s, 6H), 1.24 (s, 6H), 0.69 (dd, J = 16.2, 11.9 Hz, 1H).

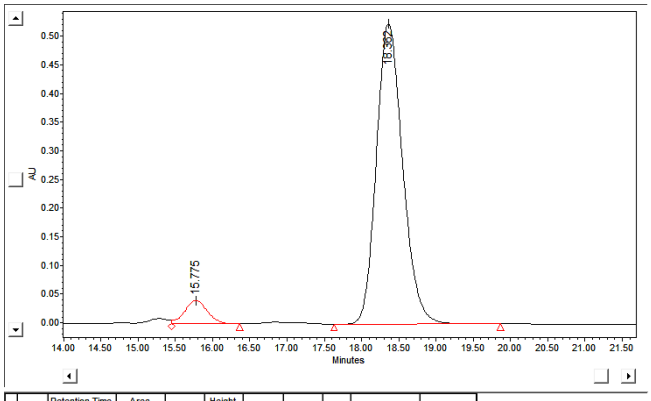
^{13}C NMR ($CDCl_3$, 100 MHz): δ 172.4, 172.3, 153.6, 135.0, 130.4, 120.3, 83.2 (2C), 65.3, 52.8 (2C), 39.7, 39.5, 30.3, 29.0, 27.2, 27.0, 26.9, 26.2, 25.1 (2C), 24.8 (2C), 18.2 (C–B).

HRMS (ESI) m/z for $C_{24}H_{37}O_6BNa$ $[M+Na]^+$ calcd 455.2580 , found 455.2601.

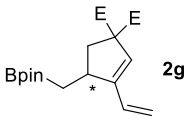
Retention time: 14.9 & 17.3 min, CHIRALCEL®-IC, *iso*-hexane/*iso*-propanol = 95/5, 30 °C, 0.5 mL/min.



	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		14.869	10725020	50.00	471966	bV			Unknown	
2		17.284	10723292	50.00	408477	BB			Unknown	



	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		15.775	834806	6.10	40232	vb			Unknown	
2		18.362	12851874	93.90	524056	bb			Unknown	



2g

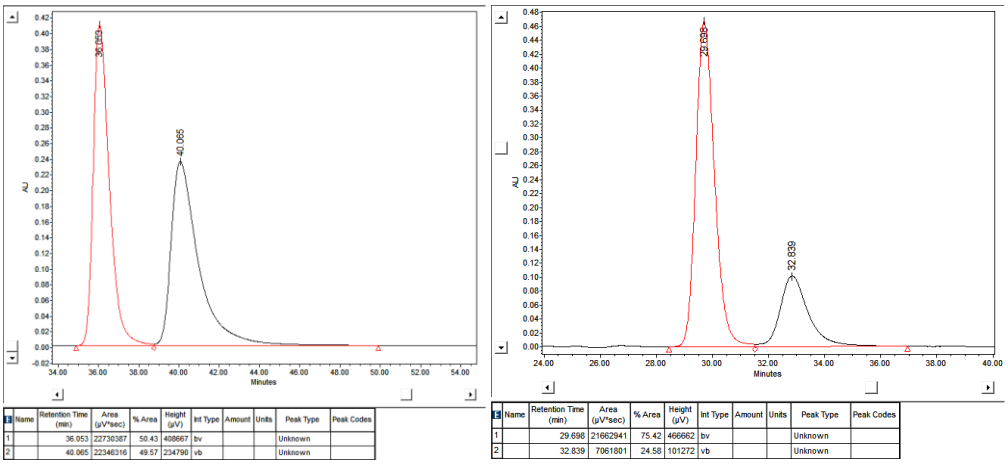
Reaction time: 96 h. Isolated yield: 24 mg (34%). *ee* = 51%.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.37 (dd, J = 17.8, 11.0 Hz, 1H), 5.68 (br. s, 1H), 5.31 (dd, J = 17.8, 0.9 Hz, 1H), 5.20 (dd, J = 11.0, 1.2 Hz, 1H), 3.73 (s, 3H), 3.71 (s, 3H), 3.15 (m, 1H), 2.77 (dd, J = 13.8, 8.5 Hz, 1H), 2.23 (dd, J = 13.8, 3.8 Hz, 1H), 1.27 (dd, J = 16.0, 3.6 Hz, 1H), 1.25 (s, 6H), 1.24 (s, 6H), 0.75 (dd, J = 16.0, 11.4 Hz, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ 172.0, 171.9, 151.7, 131.4, 125.3, 117.5, 83.3 (2C), 65.3, 52.9 (2C), 40.0, 39.4, 25.1 (2C), 24.8 (2C).

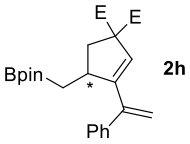
HRMS (ESI) m/z for $C_{18}H_{27}O_6BNa$ $[M+Na]^+$ calcd 373.1796, found 373.1812.

Retention time: 36.1 & 40.1 min, CHIRALCEL®-ID, *iso*-hexane/*iso*-propanol = 99/1, 30 °C, 0.3 mL/min.



#	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		36.053	22730387	50.43	408667	bv			Unknown	
2		40.065	22346316	49.57	234790	vb			Unknown	

#	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		29.698	21662941	75.42	466662	bv			Unknown	
2		32.839	7061801	24.58	101272	vb			Unknown	



2h

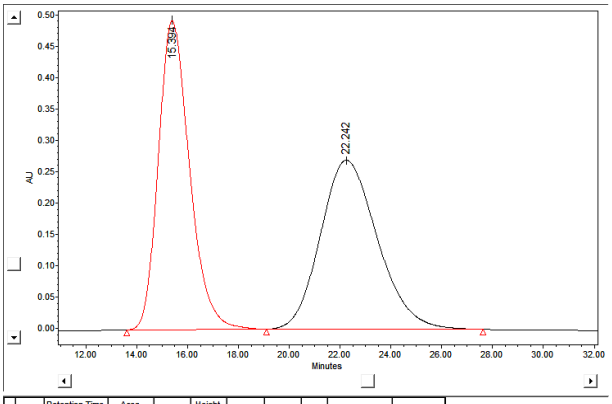
Reaction time: 48 h. Isolated yield: 64 mg (75%). *ee* = 71%.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.36 – 7.27 (m, 5H), 5.60 (d, J = 1.4 Hz, 1H), 5.33 (d, J = 1.2 Hz, 1H), 5.26 (d, J = 1.2 Hz, 1H), 3.74 (s, 3H), 3.69 (s, 3H), 3.27 (m, 1H), 2.88 (dd, J = 13.8, 8.4 Hz, 1H), 2.22 (dd, J = 13.8, 4.9 Hz, 1H), 1.24 (dd, J = 16.1, 3.7 Hz, 1H), 1.23 (s, 12H), 0.78 (dd, J = 16.1, 11.2 Hz, 1H).

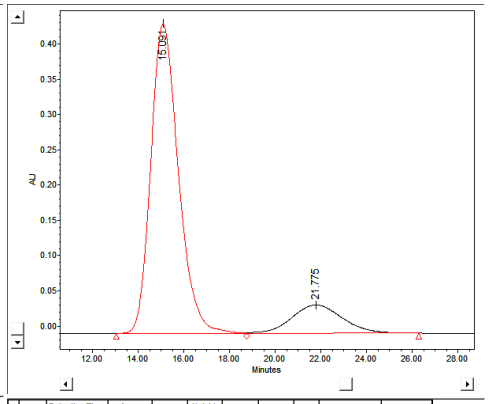
^{13}C NMR ($CDCl_3$, 100 MHz): δ 172.0, 171.8, 153.4, 144.3, 140.9, 128.2 (2C), 128.0 (2C), 127.6, 126.2, 116.0, 83.2 (2C), 65.5, 52.8, 52.8, 40.8, 39.8, 25.0 (2C), 24.8 (2C), 17.1 (C–B).

Retention time: 15.4 & 22.2 min, CHIRALCEL®-OJ, *iso*-hexane/*iso*-propanol = 98/2, 30 °C, 0.5 mL/min.

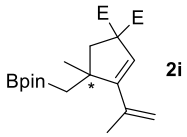
HRMS (ESI) m/z for $C_{24}H_{31}O_6BNa$ $[M+Na]^+$ calcd 449.2110, found 449.2093.



	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		15.394	42034918	49.98	492712	BB			Unknown	
2		22.242	42071530	50.02	269491	Bb			Unknown	



	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		15.091	36855021	85.66	438724	bv			Unknown	
2		21.775	6168403	14.34	40088	vb			Unknown	



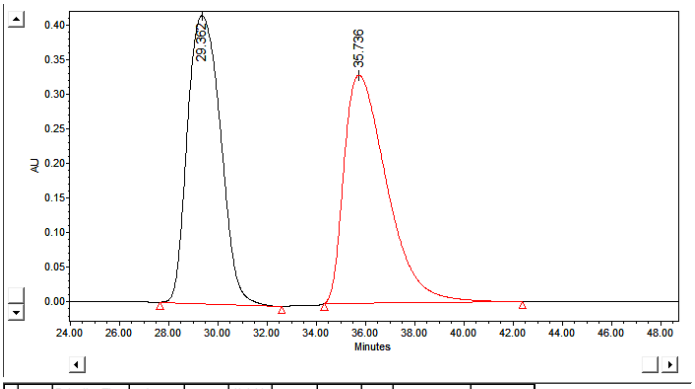
2i

Reaction time: 96 h. Isolated yield: 64 mg (79%). *ee* = 75%.

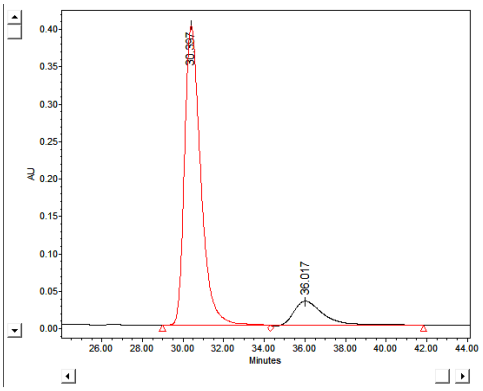
^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.64 (s, 1H), 5.14 (br. s, 1H), 5.02 (m, 1H), 3.71 (s, 3H), 3.71 (s, 3H), 2.62 (d, J = 13.8 Hz, 1H), 2.46 (d, J = 13.8 Hz, 1H), 1.89 (br. s, 3H), 1.31 (s, 3H), 1.28 (d, J = 15.4 Hz, 1H), 1.20 (s, 12H), 1.07 (d, J = 15.4 Hz, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ 172.1, 172.0, 156.0, 138.1, 123.8, 115.2, 83.0 (2C), 63.5, 52.9, 52.7, 47.9, 47.8, 29.2, 24.9 (2C), 24.9 (2C), 23.7.

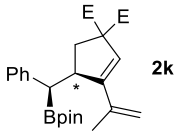
Retention time: 29.4 & 35.7 min, CHIRALCEL®-OJ, *iso*-hexane/*iso*-propanol = 99/1, 25 °C, 0.3 mL/min.



	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		29.362	38894854	49.45	415826	bb			Unknown	
2		35.736	39766716	50.55	329293	bb			Unknown	



	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		30.397	22093431	87.53	400089	bv			Unknown	
2		36.017	3148422	12.47	31760	vb			Unknown	



2k

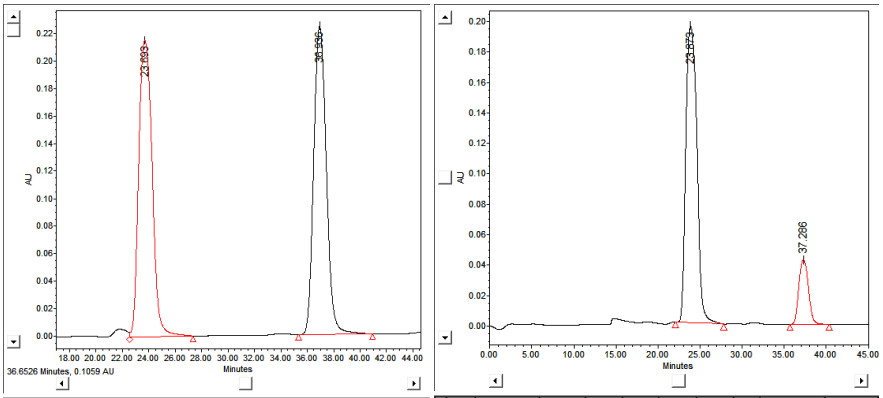
Reaction time: 86 h. Isolated yield: 9 mg (10%). *ee* = 68%.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.25 – 7.21 (m, 4H), 7.14 (m, 1H), 5.77 (d, J = 1.3 Hz, 1H), 4.94 (m, 1H), 4.91 (br. s, 1H), 3.77 (s, 3H), 3.67 (s, 3H), 3.48 (m, 1H), 2.92 (d, J = 6.0 Hz, 1H), 2.70 (dd, J = 14.1, 8.3 Hz, 1H), 2.62 (dd, J = 14.1, 5.5 Hz, 1H), 1.86 (br. s, 3H), 1.21 (s, 6H), 1.18 (s, 6H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ 171.9, 171.8, 152.2, 141.9, 138.6, 129.5 (2C), 128.4 (2C), 125.7, 124.2, 115.2, 83.4 (2C), 65.3, 52.9, 52.6, 48.1, 36.5, 24.9 (2C), 25.7 (2C), 21.6.

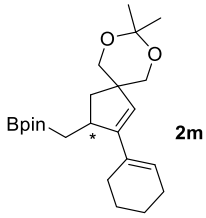
HRMS (ESI) m/z for $C_{25}H_{33}O_6BNa$ $[M+Na]^+$ calcd 463.2267, found 463.2273.

Retention time: 23.7 & 36.9 min, CHIRALCEL®-IA, *iso*-hexane/*iso*-propanol = 99/1, 25 °C, 0.3 mL/min.



	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		23.693	15141269	50.51	214562	vb			Unknown	
2		36.936	14833796	49.49	223469	bb			Unknown	

	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		23.873	17815711	84.11	194587	bb			Unknown	
2		37.286	3365871	15.89	42601	bb			Unknown	



2m

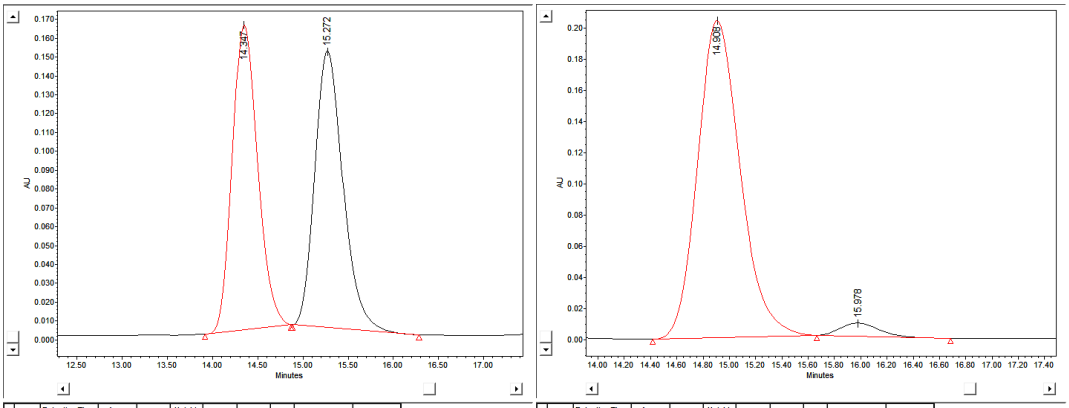
Reaction time: 108 h. Isolated yield: 59 mg (76%). *ee* = 93%.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.75 (t, J = 3.8 Hz, 1H), 5.52 (br. s, 1H), 3.76 (d, J = 11.2 Hz, 1H), 3.68 (d, J = 11.2 Hz, 1H), 3.63 (d, J = 11.2 Hz, 1H), 3.54 (d, J = 11.2 Hz, 1H), 3.14 – 3.09 (m, 1H), 2.28 – 2.21 (m, 1H), 2.15 – 2.06 (m, 3H), 2.04 (dd, J = 13.8, 9.1 Hz, 1H), 1.67 – 1.55 (m, 4H), 1.52 (dd, J = 13.8, 3.5 Hz, 1H), 1.43 (s, 3H), 1.42 (s, 3H), 1.32 (dd, J = 15.9, 3.0 Hz, 1H), 1.25 (s, 6H), 1.23 (s, 6H), 0.64 (dd, J = 15.9, 11.5 Hz, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ 151.6, 132.2, 125.9, 124.7, 97.5, 83.1 (2C), 69.8, 69.6, 48.0, 40.0, 38.9, 26.7, 25.8, 25.1 (2C), 24.8 (2C), 24.4, 23.6, 22.9, 22.4, 20.1 (C–B).

HRMS (ESI) m/z for $C_{23}H_{37}BO_4Na$ $[M+Na]^+$ calcd 411.2677, found 411.2691.

Retention time: 14.3 & 15.3 min, CHIRALCEL®-IA, *iso*-hexane/*iso*-propanol = 98/2, 25 °C, 0.3 mL/min.



	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		14.347	3154860	49.68	161661	bb			Unknown	
2		15.272	3195590	50.32	146785	bb			Unknown	

	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		14.908	4575933	96.41	203150	bb			Unknown	
2		15.978	170156	3.59	8400	bb			Unknown	

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(4) (a) Hatano, M.; Ikeno, T.; Matsumura, T.; Torii, S.; Ishihara, K. *Adv. Synth. Catal.* **2008**, *350*, 1776–1780. (b) Storer, R. I.; Carrera, D. E.; Ni, Y.; Macmillan, D. W. C. *J. Am. Chem. Soc.* **2006**, *128*, 84–86. (c) Holloway, C. A.; Muratore, M. E.; Storer, R.; Dixon, D. J. *Org. Lett.* **2010**, *12*, 4720–4723. (d) Itoh, J.; Fuchibe, K.; Akiyama, T. *Angew. Chem. Int. Ed.* **2006**, *45*, 4796–4798. (e) Yamanaka, M.; Itoh, J.; Fuchibe, K.; Akiyama, T. *J. Am. Chem. Soc.* **2007**, *129*, 6756–6764.(f) Seayad, J.; Seayad, A. M.; List, B. *J. Am. Chem. Soc.* **2006**, *128*, 1086–1087. (g) Huang, Y.-Y.; Chakrabarti, A.; Morita, N.; Schneider, U.; Kobayashi, S. *Angew. Chem. Int. Ed.* **2011**, *50*, 11121–11124.

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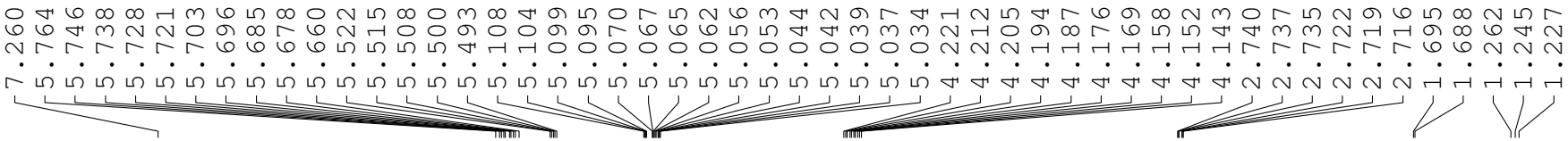
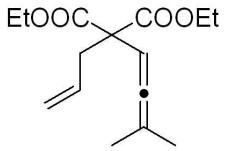
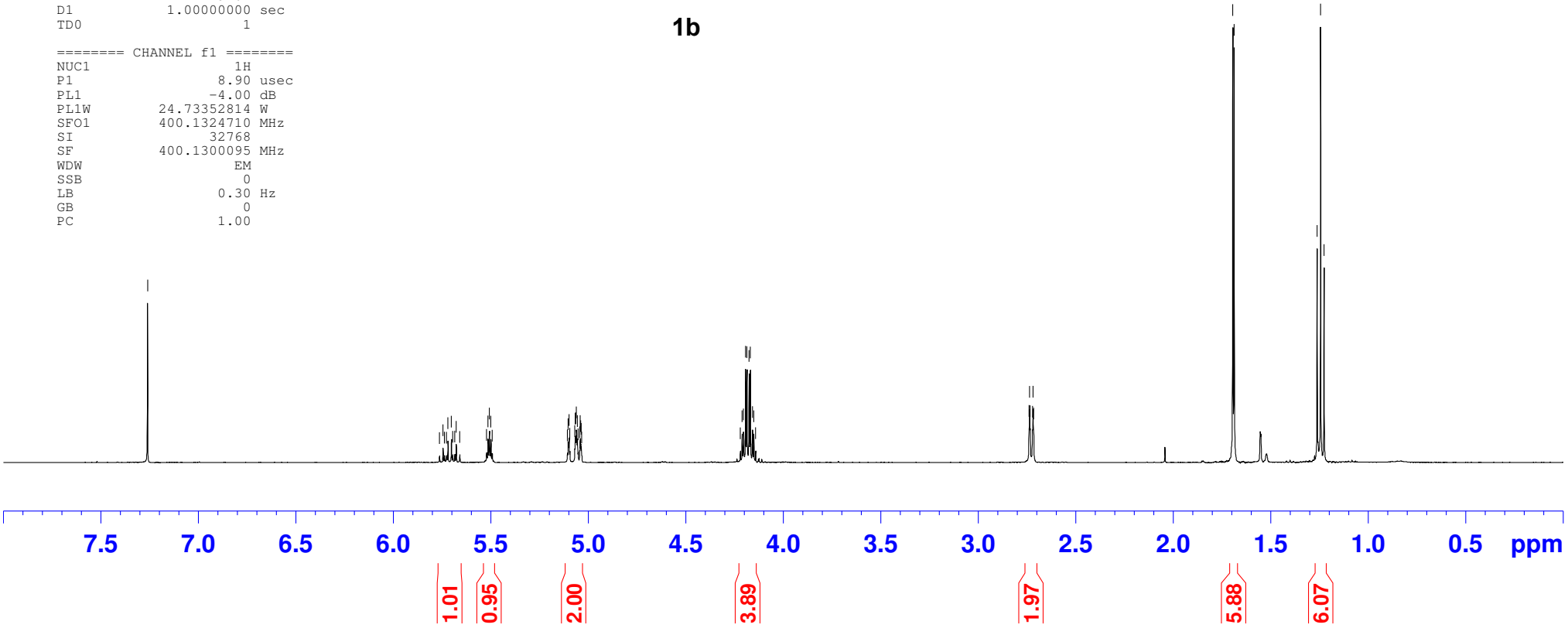


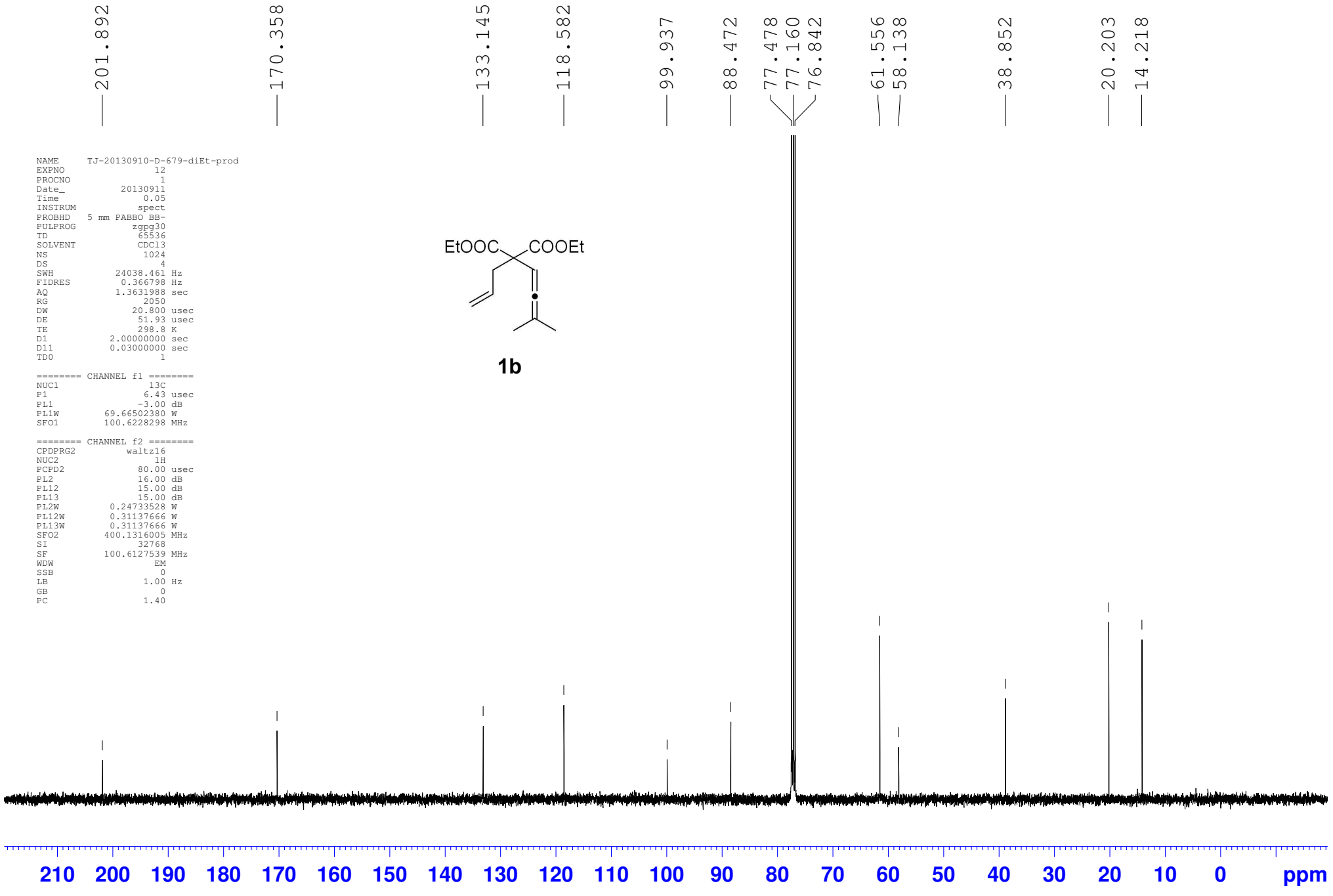

```
NAME      TJ-20130910-D-679-diEt-prod
EXPNO                10
PROCNO                1
Date_          20130910
Time              16.51
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ            3.9846387 sec
RG                  724
DW                60.800 usec
DE                 6.50 usec
TE                297.7 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.90 usec
PL1               -4.00 dB
PL1W        24.73352814 W
SFO1        400.1324710 MHz
SI                32768
SF          400.1300095 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



1b

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133.145
118.582
99.937
88.472
77.478
77.160
76.842
61.556
58.138
38.852
20.203
14.218
NAME TJ-20130910-D-679-diEt-prod
EXPNO 12
PROCNO 1
Date_ 20130911
Time 0.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 51.93 usec
TE 298.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.43 usec
PL1 -3.00 dB
PL1W 69.66502380 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 16.00 dB
PL12 15.00 dB
PL13 15.00 dB
PL2W 0.24733528 W
PL12W 0.31137666 W
PL13W 0.31137666 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127539 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
EtOOC
COOEt
1b
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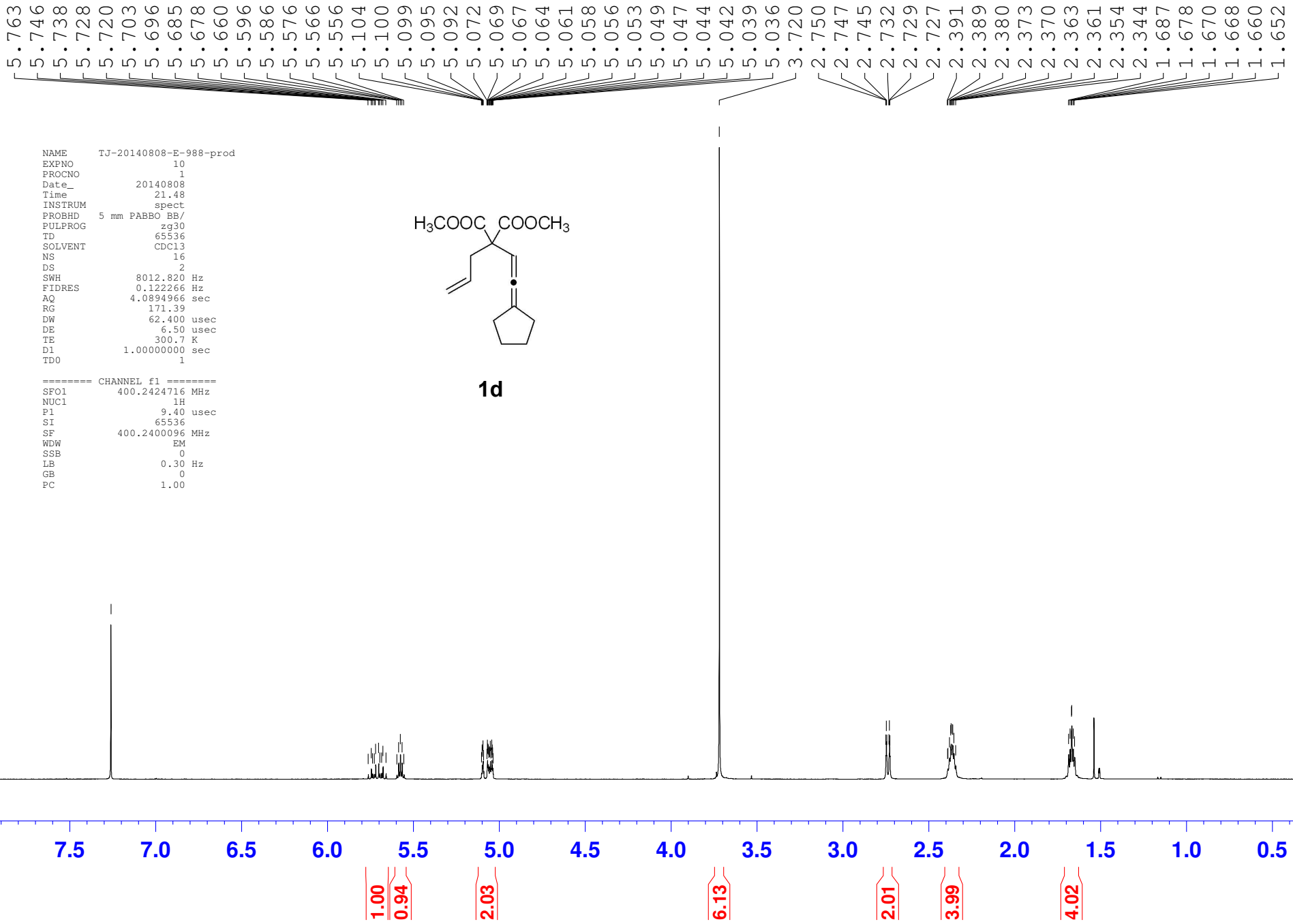
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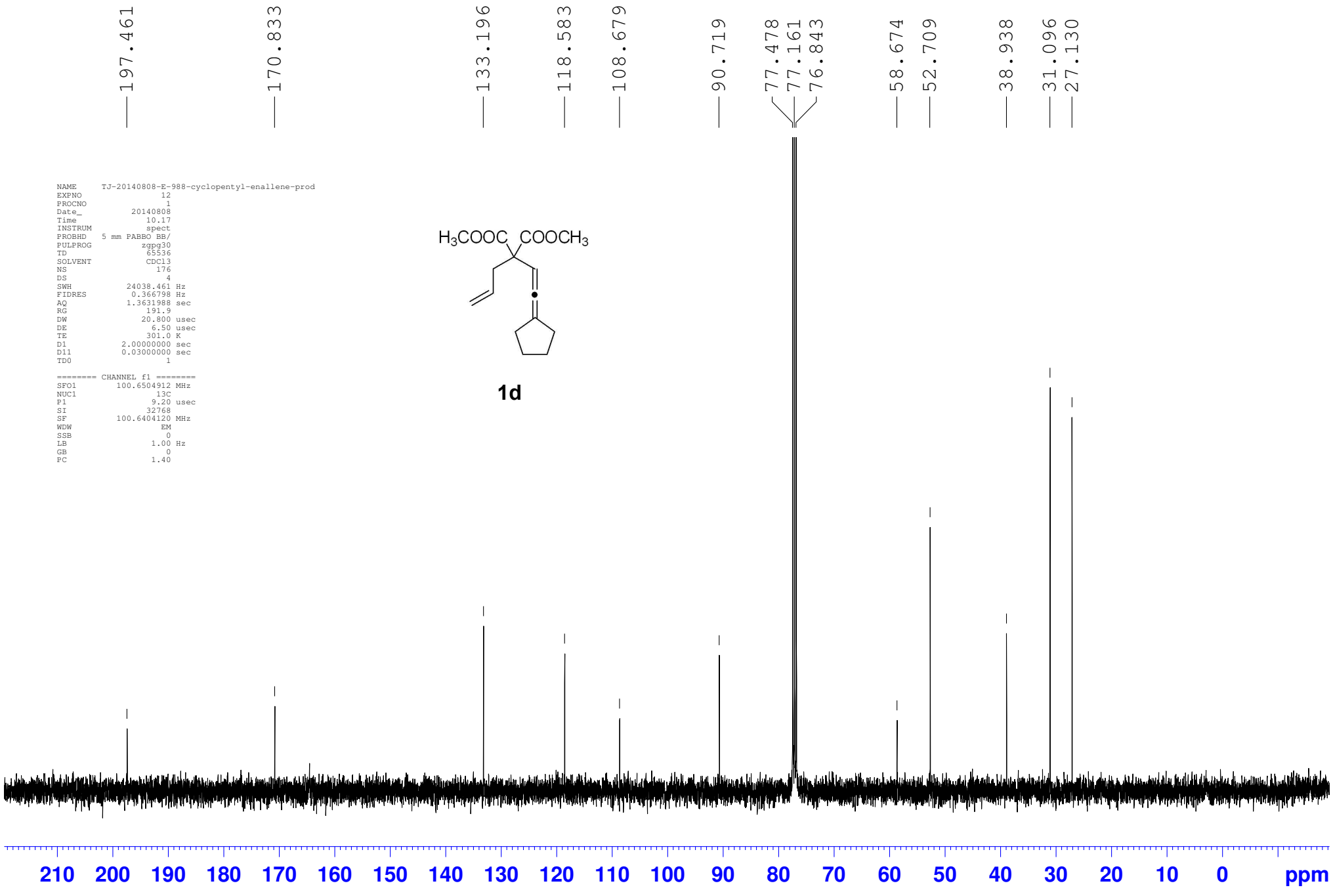
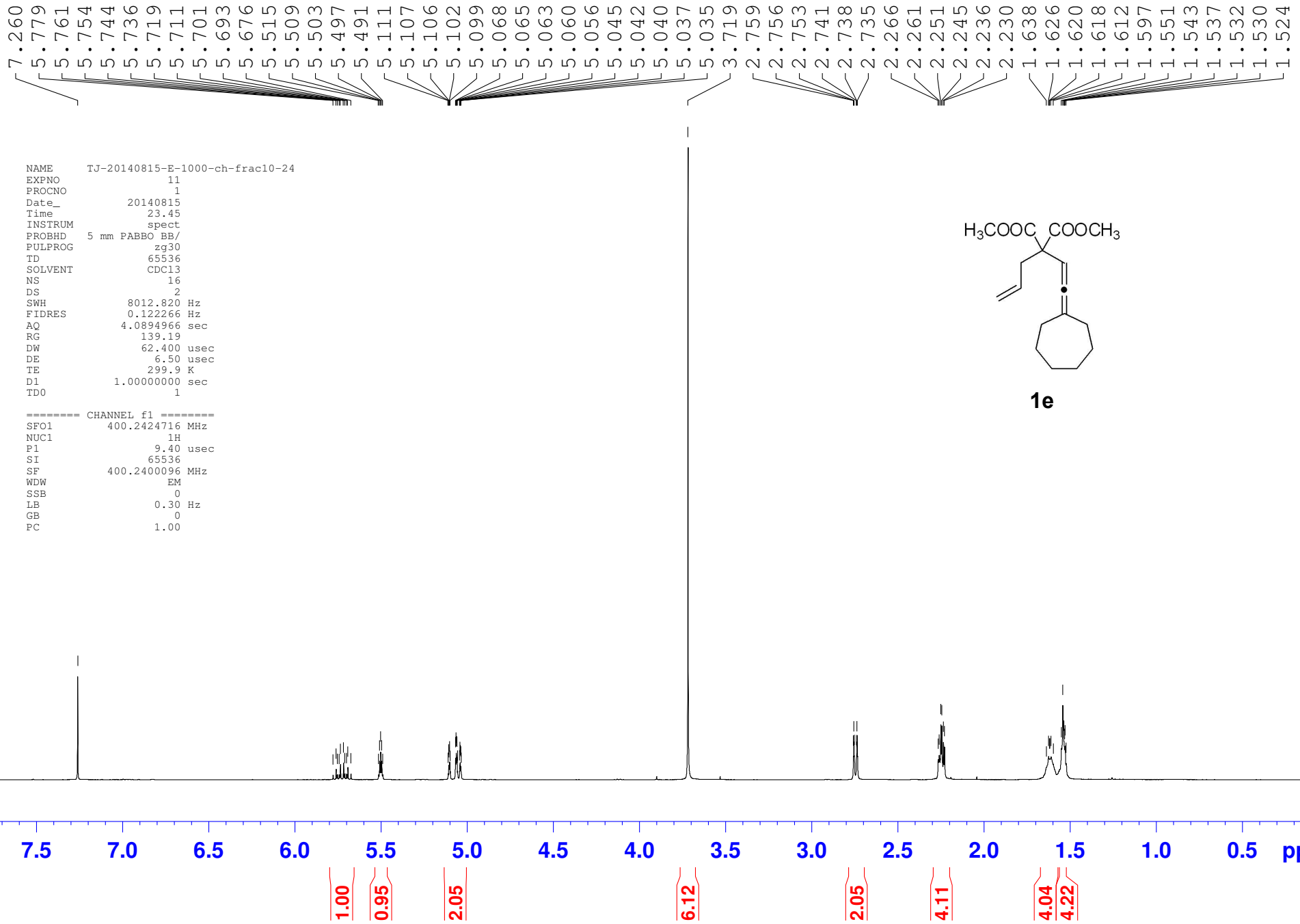
5.746
5.738
5.728
5.720
5.703
5.696
5.685
5.678
5.660
5.596
5.586
5.576
5.566
5.556
5.104
5.100
5.099
5.095
5.092
5.072
5.069
5.067
5.064
5.061
5.058
5.056
5.053
5.049
5.047
5.044
5.042
5.039
5.036
3.720
2.750
2.747
2.745
2.732
2.729
2.727
2.391
2.389
2.380
2.373
2.370
2.363
2.361
2.354
2.344
1.687
1.678
1.670
1.668
1.660
1.652
NAME TJ-20140808-E-988-prod
EXPNO 10
PROCNO 1
Date_ 20140808
Time 21.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 171.39
DW 62.400 usec
DE 6.50 usec
TE 300.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.2424716 MHz
NUC1 1H
P1 9.40 usec
SI 65536
SF 400.2400096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H3COOC COOCH3
1d
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
ppm
1.00
0.94
2.03
6.13
2.01
3.99
4.02


197.461
170.833
133.196
118.583
108.679
90.719
77.478
77.161
76.843
58.674
52.709
38.938
31.096
27.130
NAME TJ-20140808-E-988-cyclopentyl-enallene-prod
EXPNO 12
PROCNO 1
Date_ 20140808
Time 10.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 176
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 191.9
DW 20.800 usec
DE 6.50 usec
TE 301.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6504912 MHz
NUC1 13C
P1 9.20 usec
SI 32768
SF 100.6404120 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
H3COOC COOCH3
1d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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5.779
5.761
5.754
5.744
5.736
5.719
5.711
5.701
5.693
5.676
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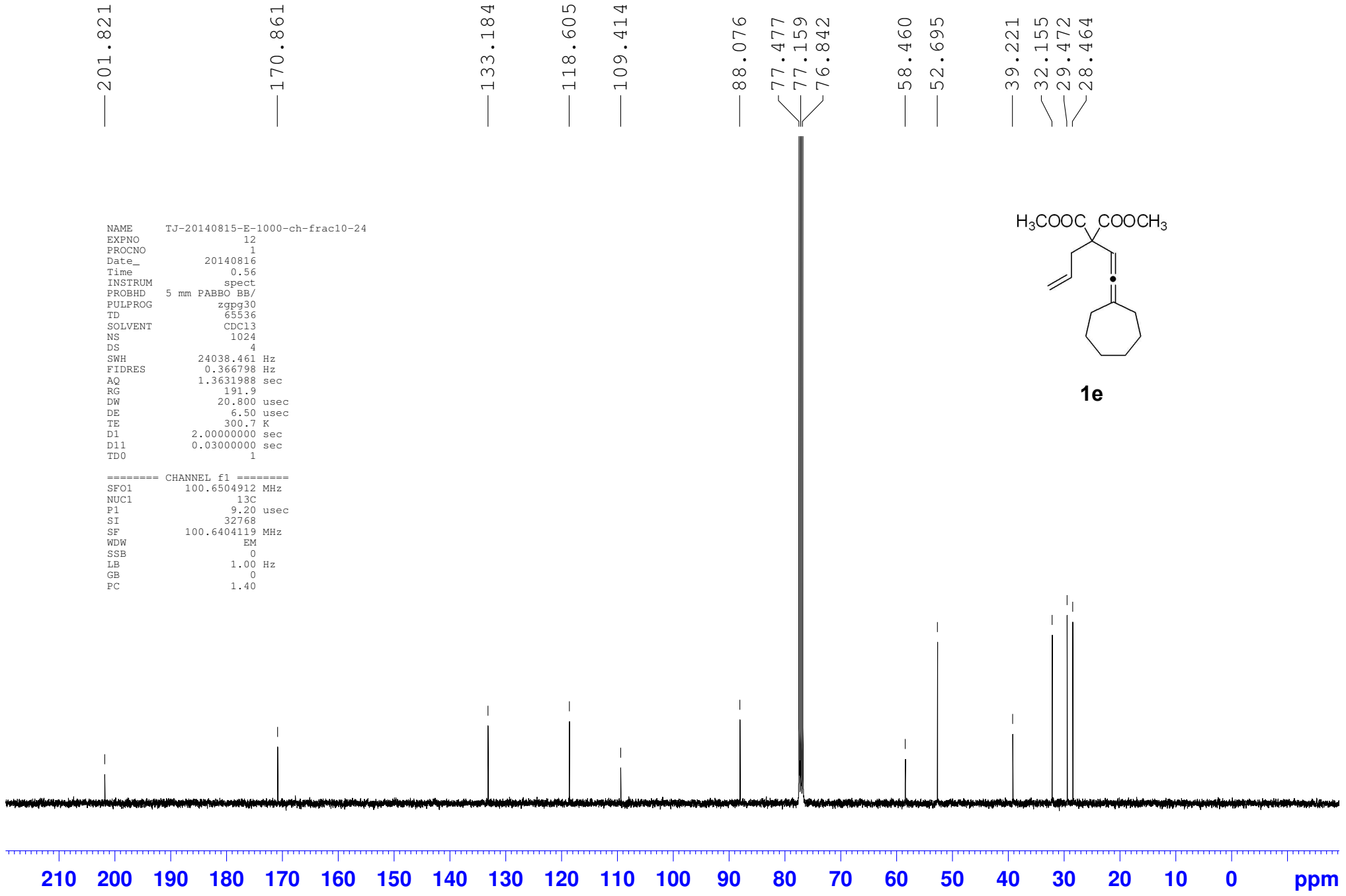
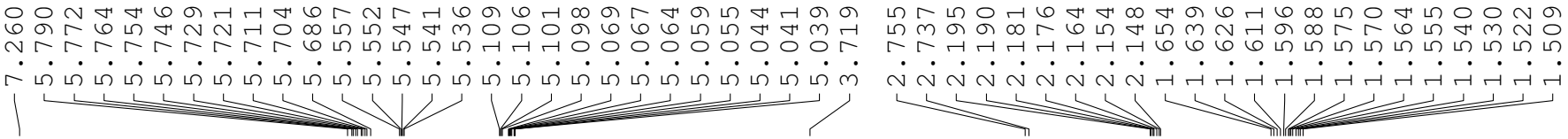
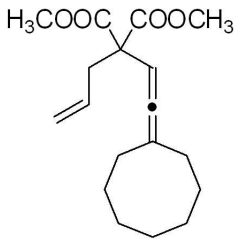
5.509
5.503
5.497
5.491
5.111
5.107
5.106
5.102
5.099
5.068
5.065
5.063
5.060
5.056
5.045
5.042
5.040
5.037
5.035
3.719
2.759
2.756
2.753
2.741
2.738
2.735
2.266
2.261
2.251
2.245
2.236
2.230
1.638
1.626
1.620
1.618
1.612
1.597
1.551
1.543
1.537
1.532
1.530
1.524
NAME TJ-20140815-E-1000-ch-frac10-24
EXPNO 11
PROCNO 1
Date_ 20140815
Time 23.45
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 139.19
DW 62.400 usec
DE 6.50 usec
TE 299.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.2424716 MHz
NUC1 1H
P1 9.40 usec
SI 65536
SF 400.2400096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H3COOC COOCH3
1e
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
ppm
1.00
0.95
2.05
6.12
2.05
4.11
4.04
4.22

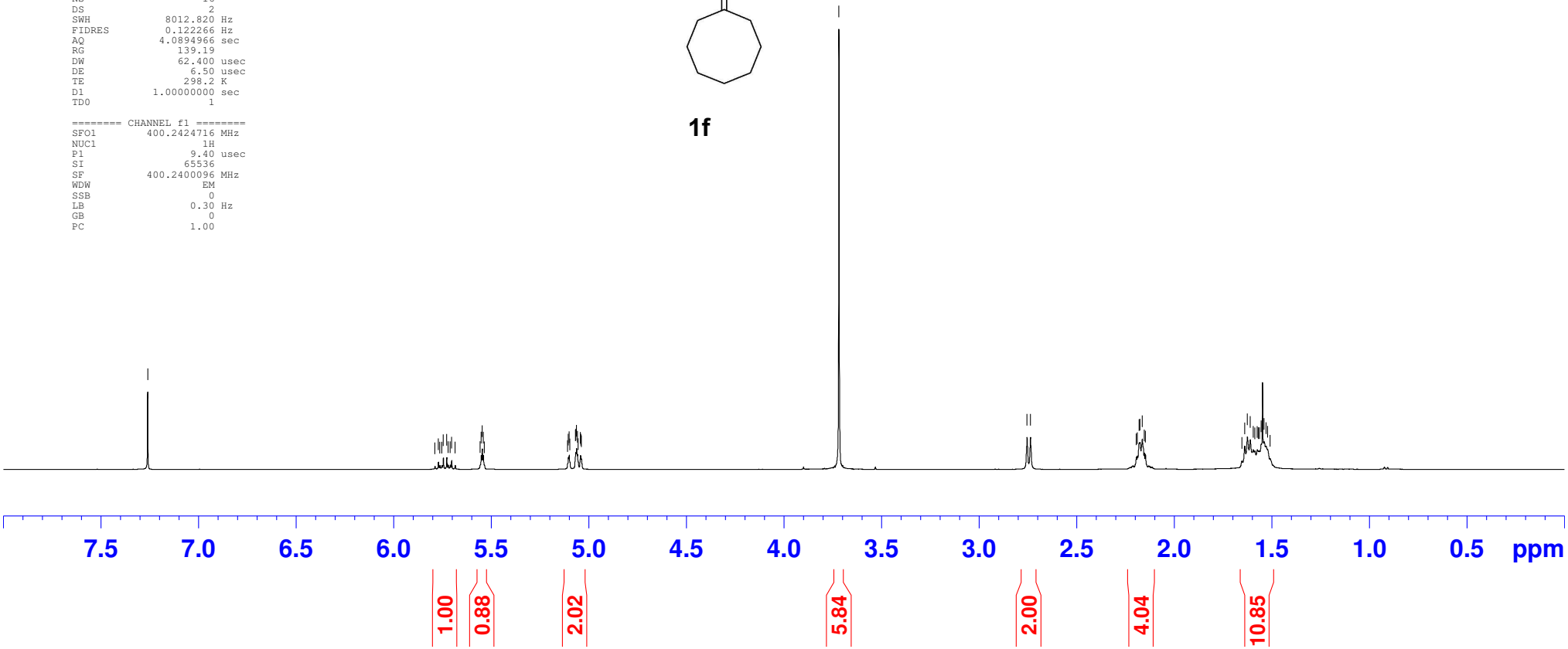

201.821
170.861
133.184
118.605
109.414
88.076
77.477
77.159
76.842
58.460
52.695
39.221
32.155
29.472
28.464
NAME TJ-20140815-E-1000-ch-frac10-24
EXPNO 12
PROCNO 1
Date_ 20140816
Time 0.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 191.9
DW 20.800 usec
DE 6.50 usec
TE 300.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6504912 MHz
NUC1 13C
P1 9.20 usec
SI 32768
SF 100.6404119 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
H3COOC COOCH3
1e
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

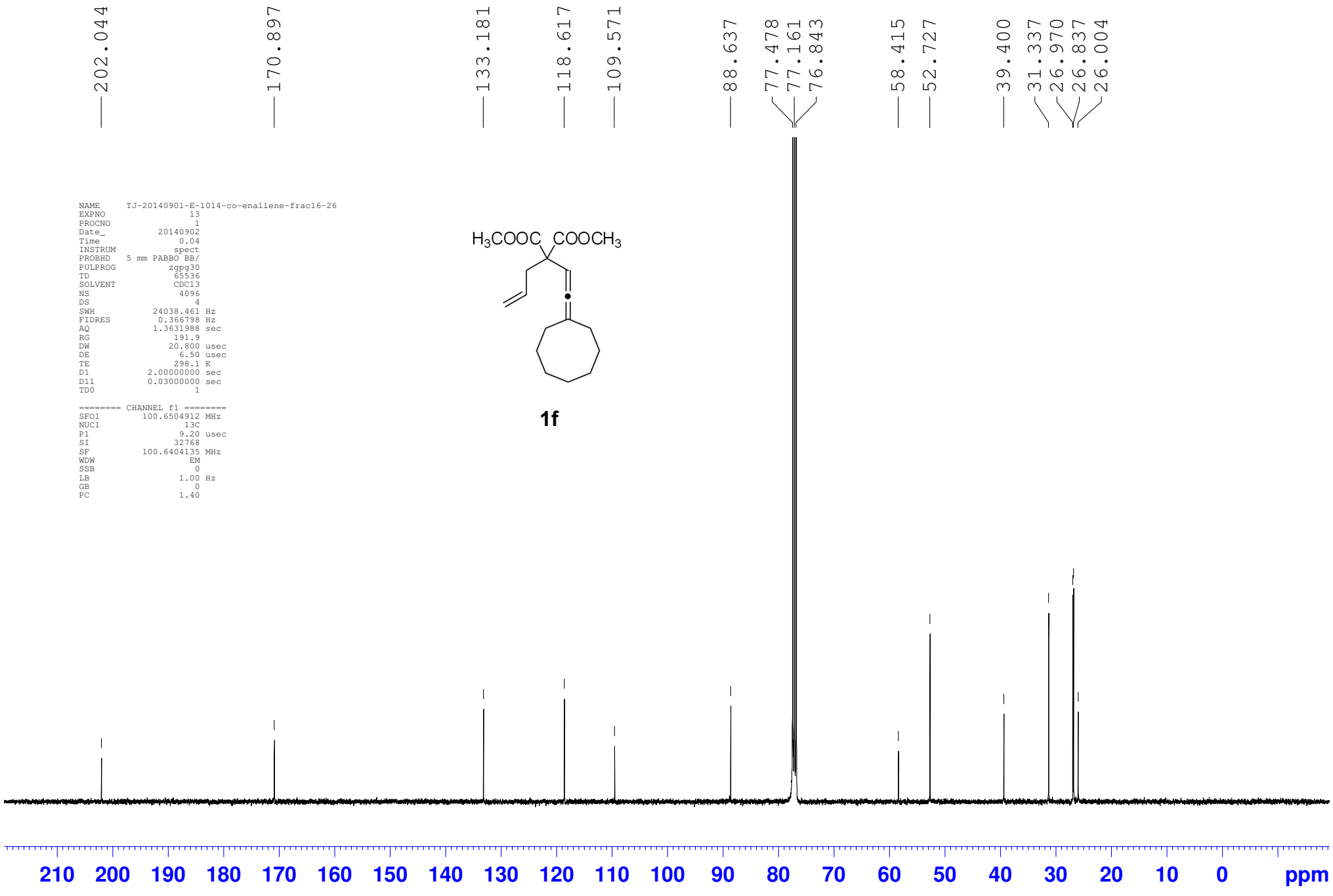


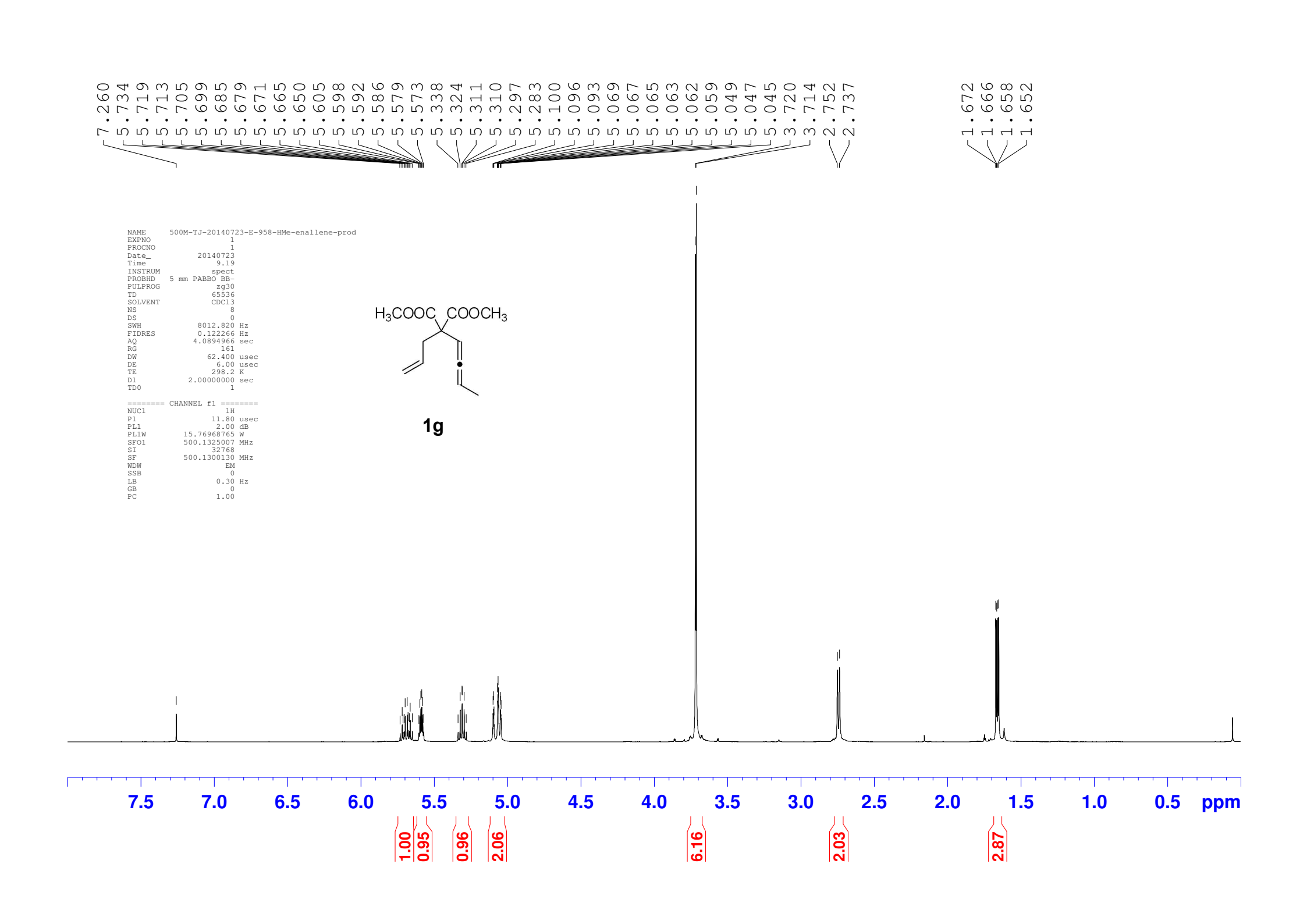

```
NAME      TJ-20140901-E-1014-co-enallene-frac16-26
EXPNO                 12
PROCNO                 1
Date_           20140901
Time                20.08
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894966 sec
RG                 139.19
DW                62.400 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.2424716 MHz
NUC1                 1H
P1                  9.40 usec
SI                 65536
SF           400.2400096 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



1f




202.044
170.897
133.181
118.617
109.571
88.637
77.478
77.161
76.843
58.415
52.727
39.400
31.337
26.970
26.837
26.004
NAME TJ-20140901-E-1014-co-enallene-frac16-26
EXPNO 13
PROCNO 1
Date_ 20140902
Time 0.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 4096
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 191.9
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 100.6504912 MHz
NUC1 13C
P1 9.20 usec
SI 32768
SF 100.6404135 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
H3COOC COOCH3
1f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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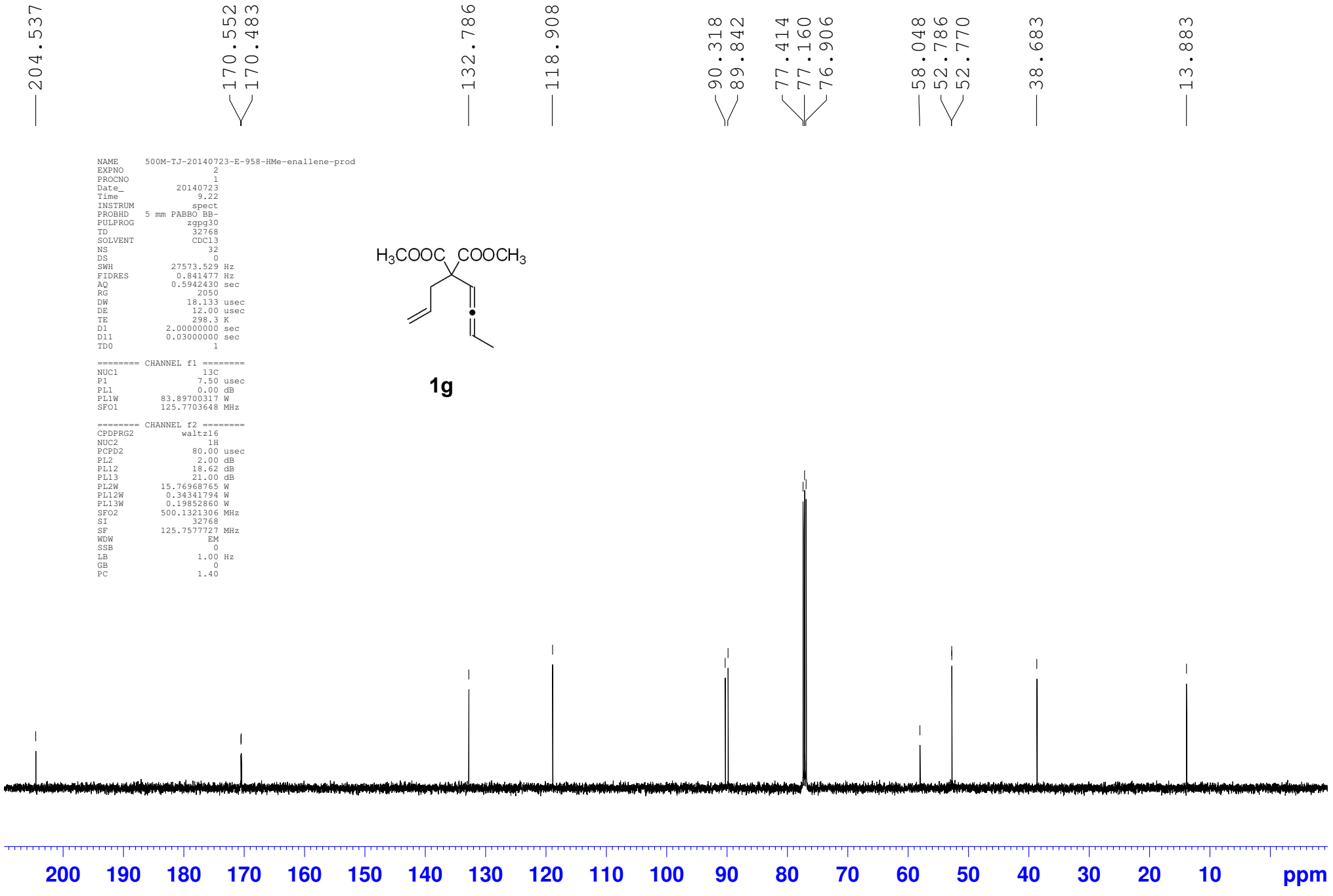
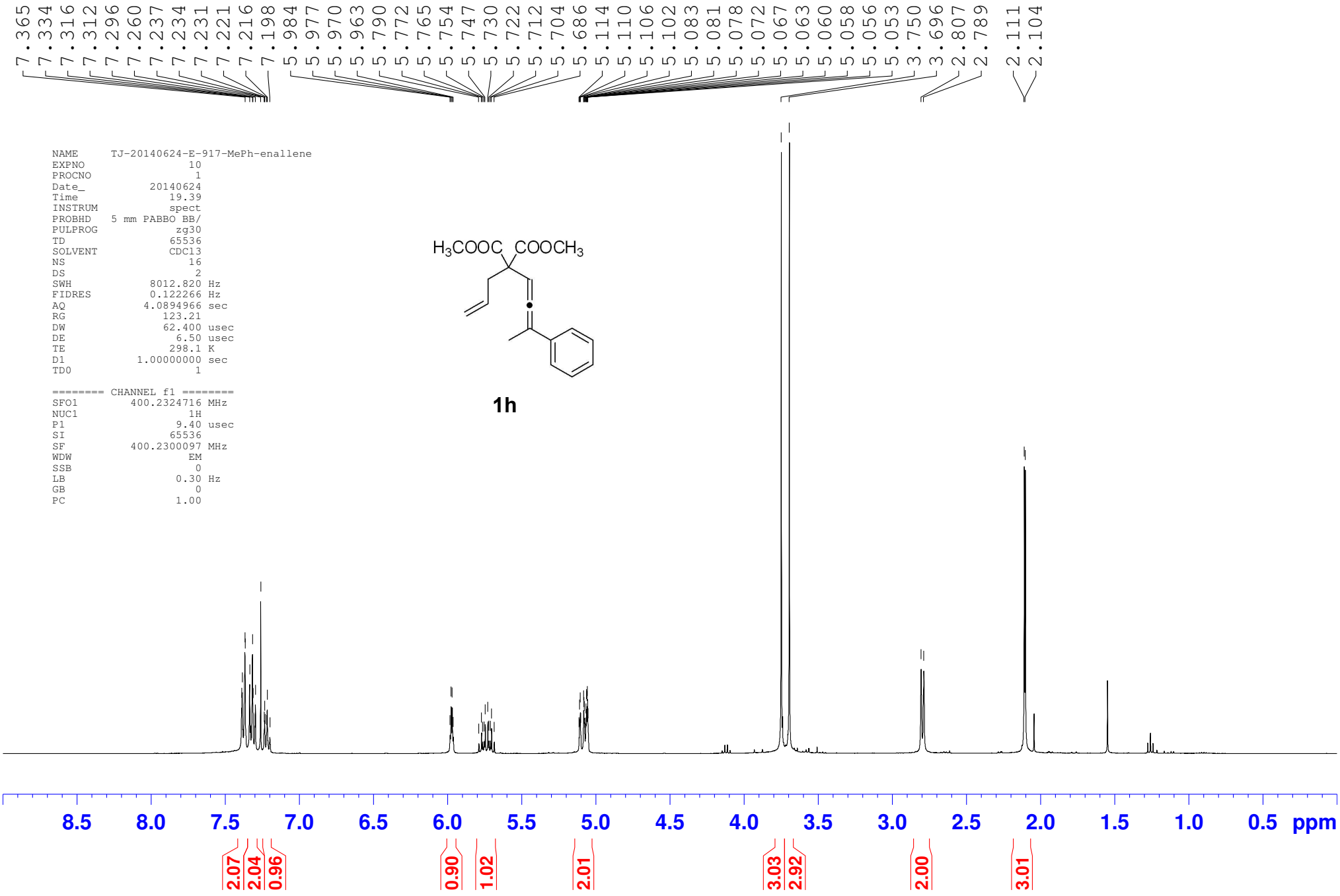
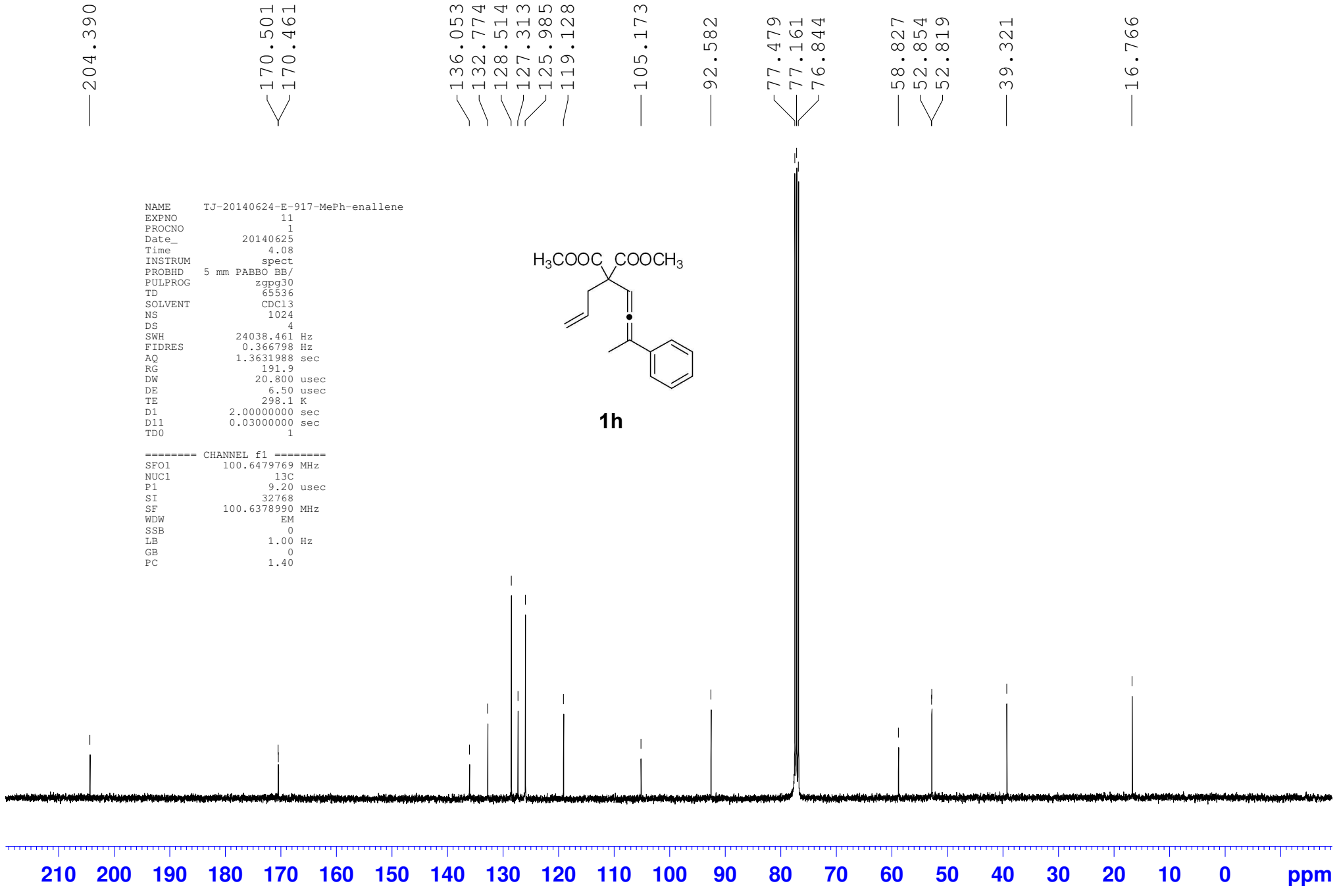
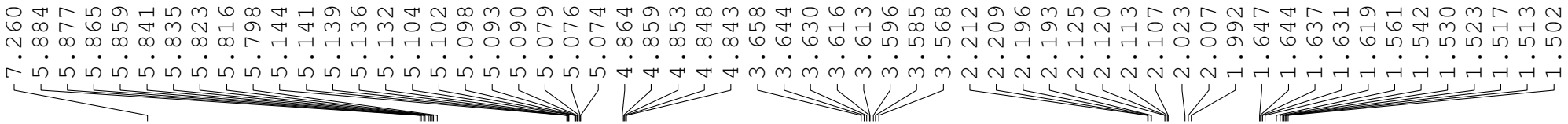
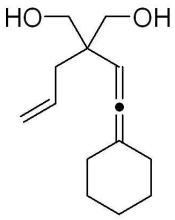
5.719
5.713
5.705
5.699
5.685
5.679
5.671
5.665
5.650
5.605
5.598
5.592
5.586
5.579
5.573
5.338
5.324
5.311
5.310
5.297
5.283
5.100
5.096
5.093
5.069
5.067
5.065
5.063
5.062
5.059
5.049
5.047
5.045
3.720
3.714
2.752
2.737
1.672
1.666
1.658
1.652
NAME 500M-TJ-20140723-E-958-HMe-enallene-prod
EXPNO 1
PROCNO 1
Date_ 20140723
Time 9.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 161
DW 62.400 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 2.00 dB
PL1W 15.76968765 W
SFO1 500.1325007 MHz
SI 32768
SF 500.1300130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H3COOC COOCH3
1g
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
ppm
1.00
0.95
0.96
2.06
6.16
2.03
2.87


204.537
170.552
170.483
132.786
118.908
90.318
89.842
77.414
77.160
76.906
58.048
52.786
52.770
38.683
13.883
NAME 500M-TJ-20140723-E-958-HMe-enallene-prod
EXPNO 2
PROCNO 1
Date_ 20140723
Time 9.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 32
DS 0
SWH 27573.529 Hz
FIDRES 0.841477 Hz
AQ 0.5942430 sec
RG 2050
DW 18.133 usec
DE 12.00 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PL1 0.00 dB
PL1W 83.89700317 W
SFO1 125.7703648 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 18.62 dB
PL13 21.00 dB
PL2W 15.76968765 W
PL12W 0.34341794 W
PL13W 0.19852860 W
SFO2 500.1321306 MHz
SI 32768
SF 125.7577727 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
H3COOC COOCH3
1g
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.365
7.334
7.316
7.312
7.296
7.260
7.237
7.234
7.231
7.221
7.216
7.198
5.984
5.977
5.970
5.963
5.790
5.772
5.765
5.754
5.747
5.730
5.722
5.712
5.704
5.686
5.114
5.110
5.106
5.102
5.083
5.081
5.078
5.072
5.067
5.063
5.060
5.058
5.056
5.053
3.750
3.696
2.807
2.789
2.111
2.104
NAME TJ-20140624-E-917-MePh-enallene
EXPNO 10
PROCNO 1
Date_ 20140624
Time 19.39
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 123.21
DW 62.400 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.2324716 MHz
NUC1 1H
P1 9.40 usec
SI 65536
SF 400.2300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H3COOC COOCH3
1h
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
ppm
2.07
2.04
0.96
0.90
1.02
2.01
3.03
2.92
2.00
3.01

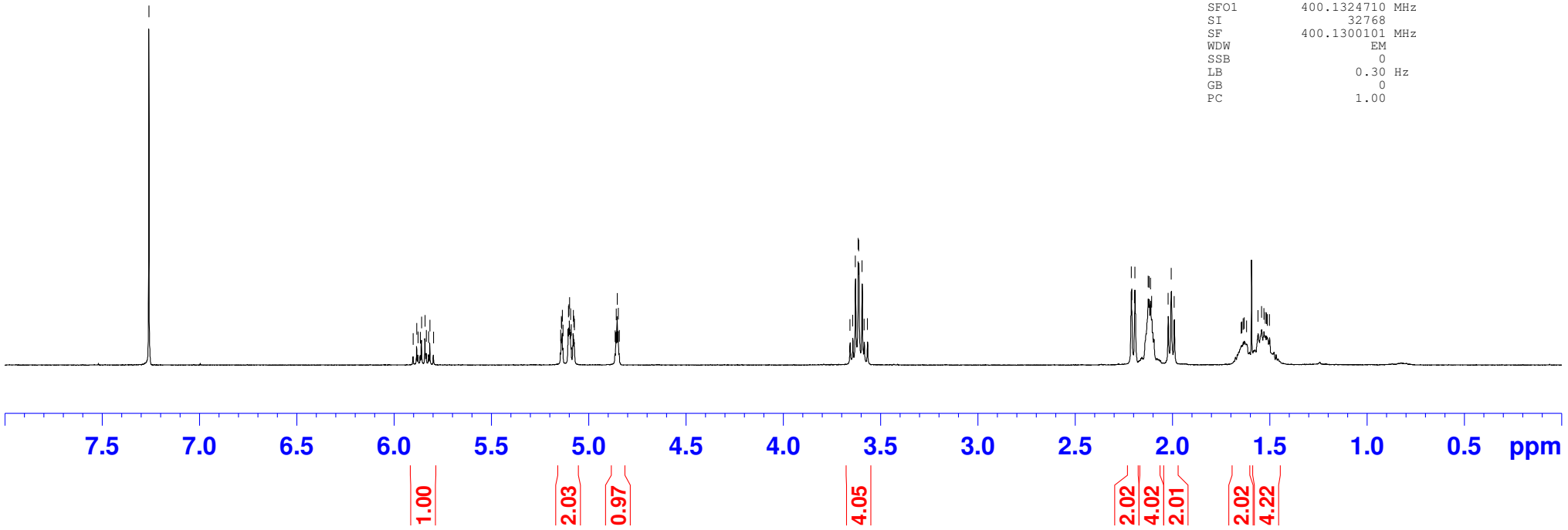

204.390
170.501
170.461
136.053
132.774
128.514
127.313
125.985
119.128
105.173
92.582
77.479
77.161
76.844
58.827
52.854
52.819
39.321
16.766
NAME TJ-20140624-E-917-MePh-enallene
EXPNO 11
PROCNO 1
Date_ 20140625
Time 4.08
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 191.9
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 100.6479769 MHz
NUC1 13C
P1 9.20 usec
SI 32768
SF 100.6378990 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
H3COOC COOCH3
1h
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

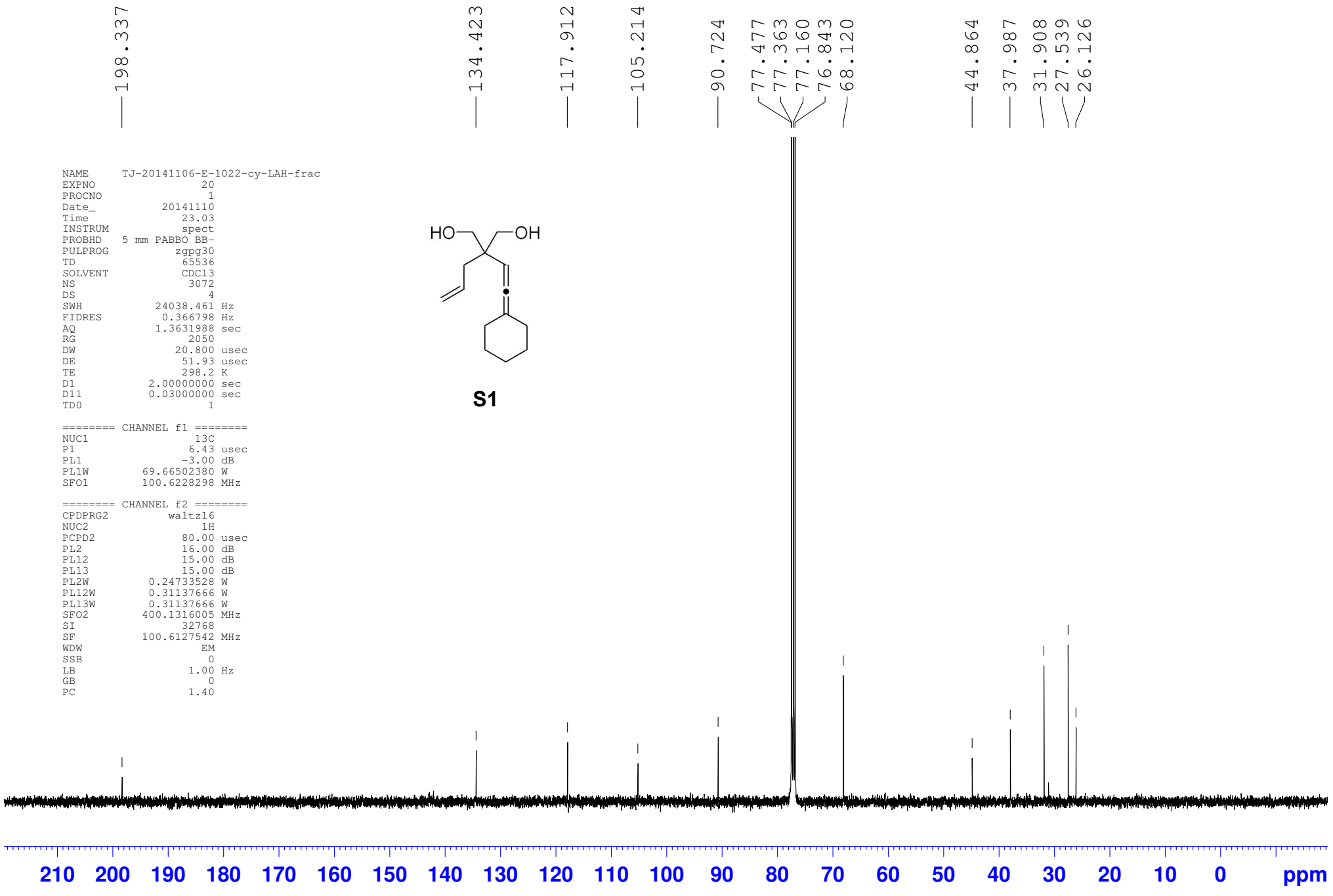


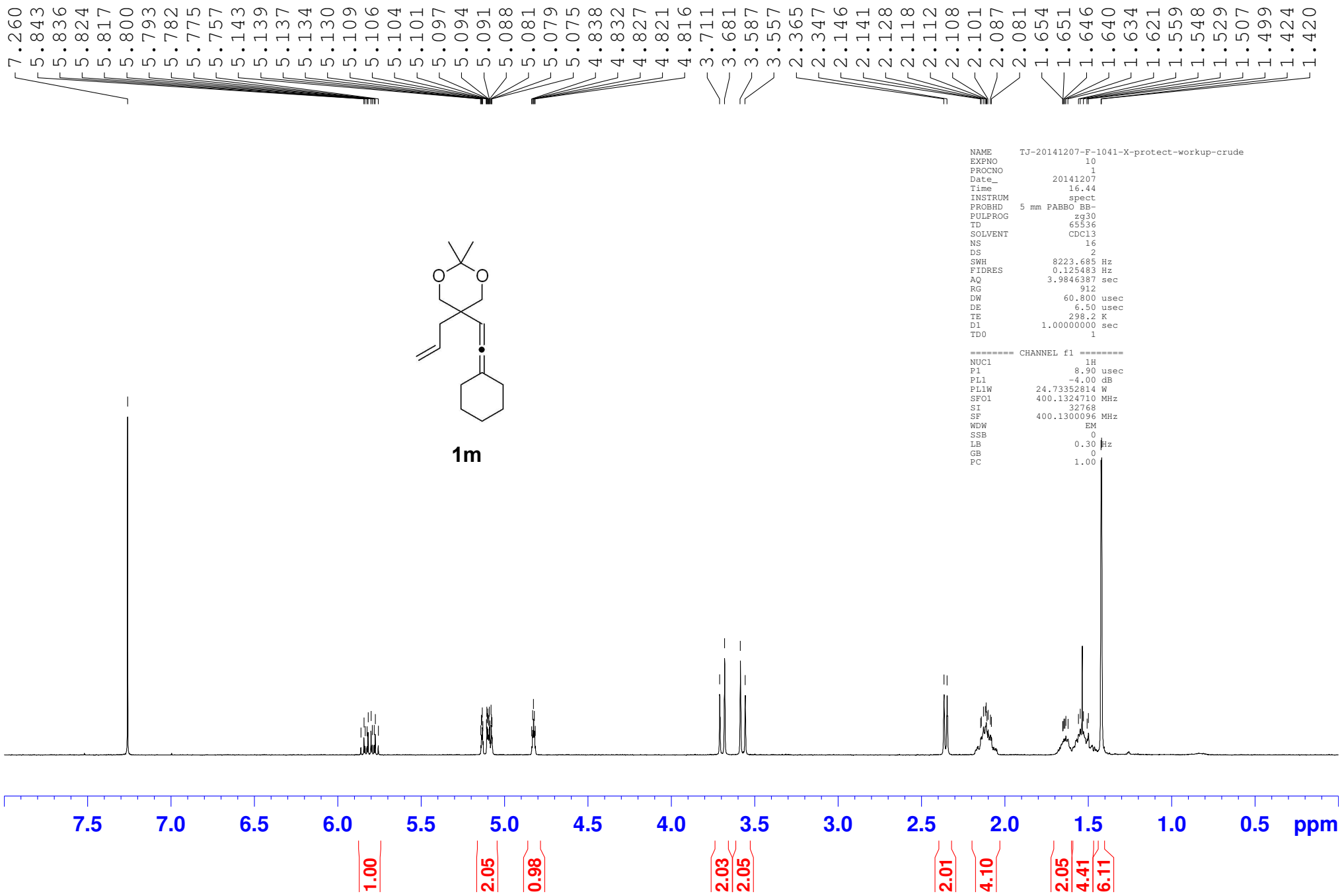


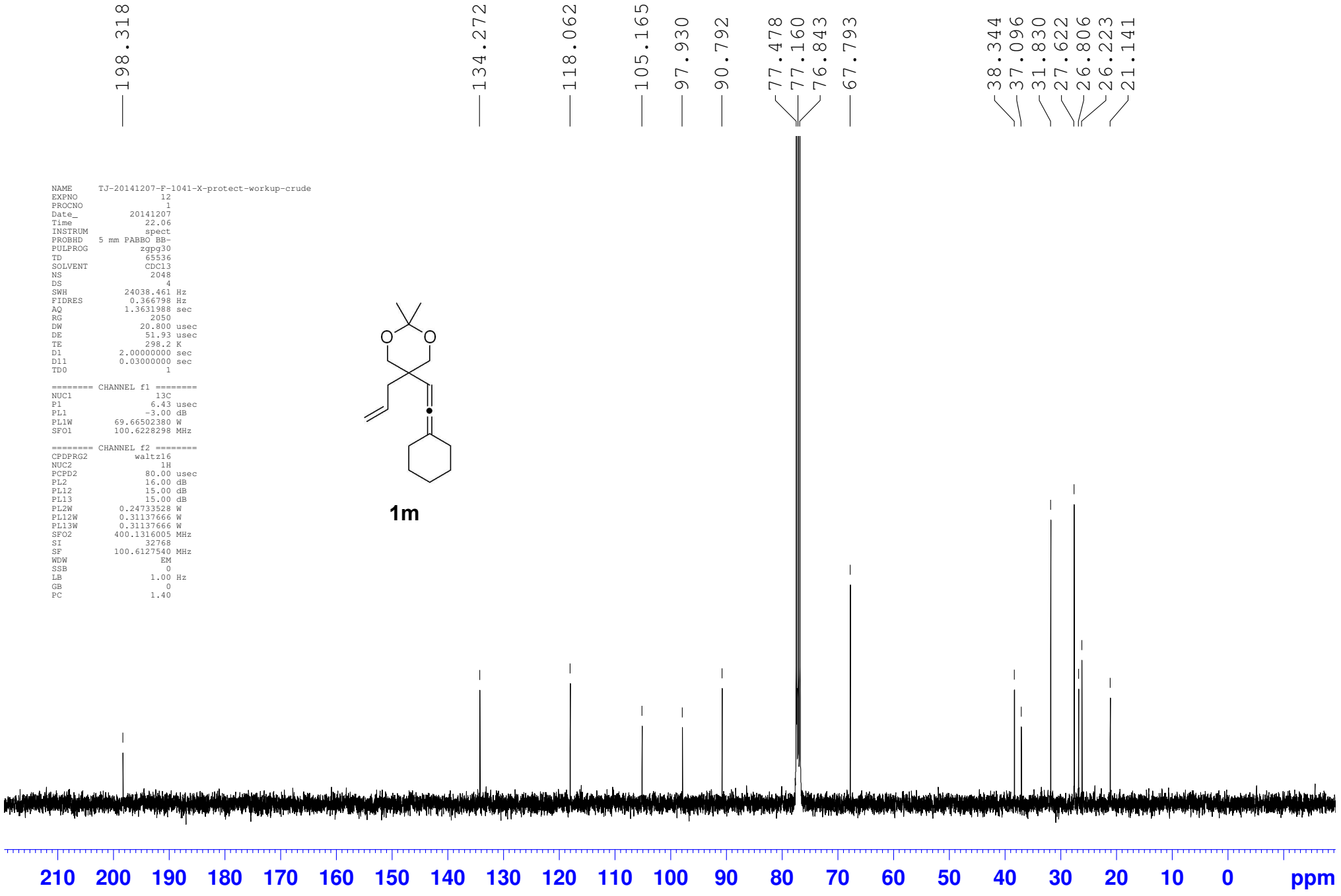
S1

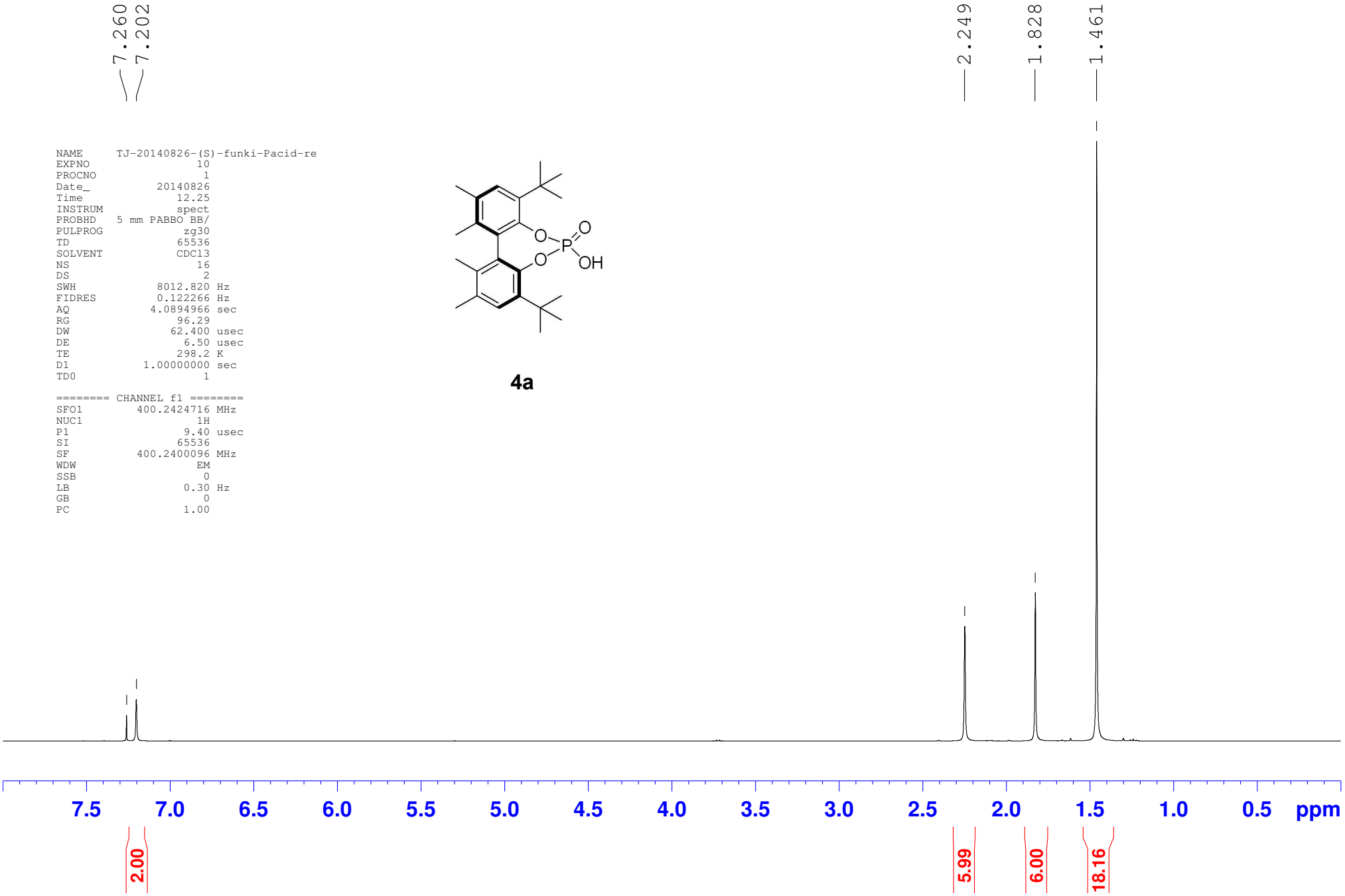
NAME	TJ-20141106-E-1022-cy-LAH-frac
EXPNO	10
PROCNO	1
Date_	20141106
Time	10.27
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	912
DW	60.800 usec
DE	6.50 usec
TE	673.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.90 usec
PL1	-4.00 dB
PL1W	24.73352814 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300101 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

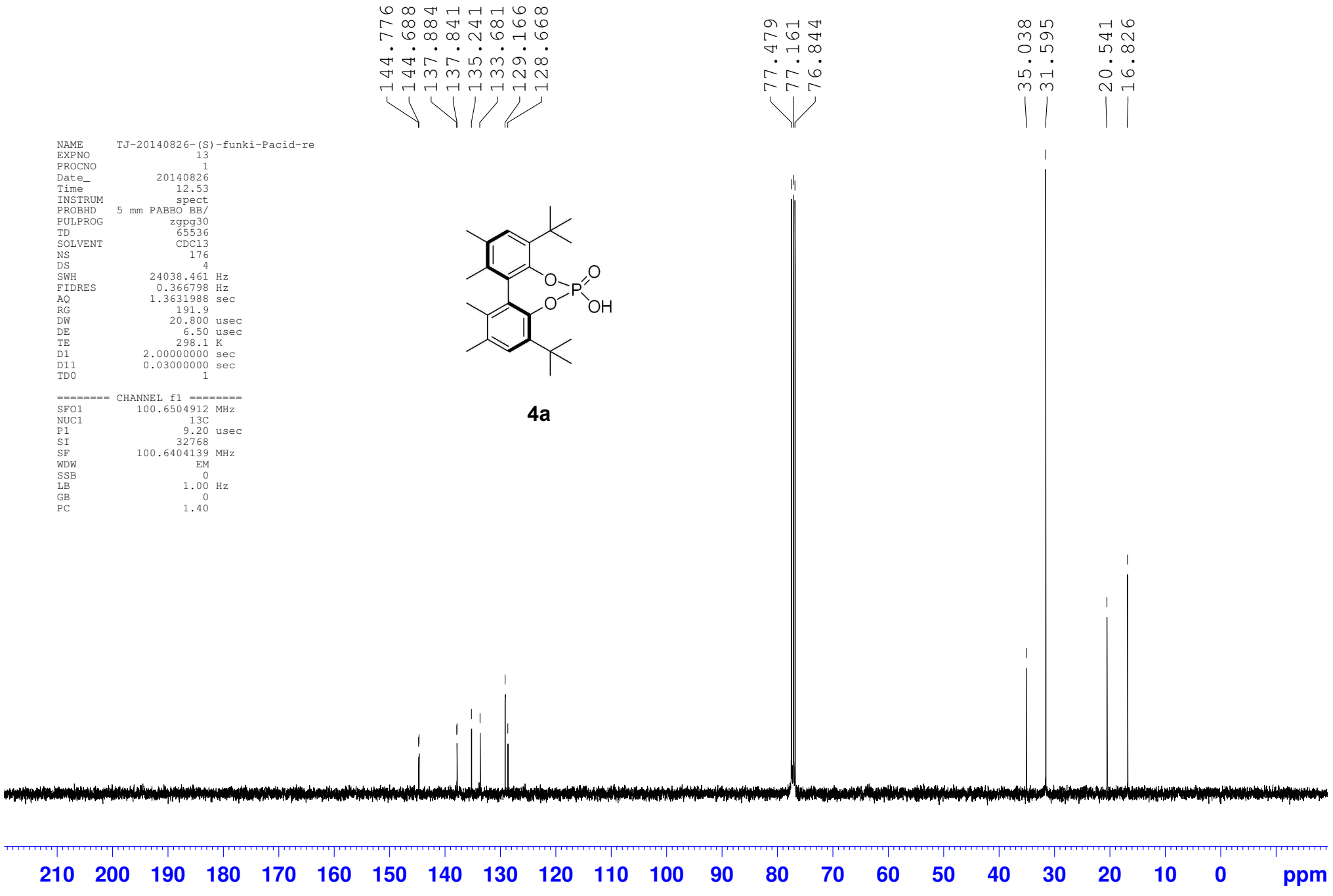


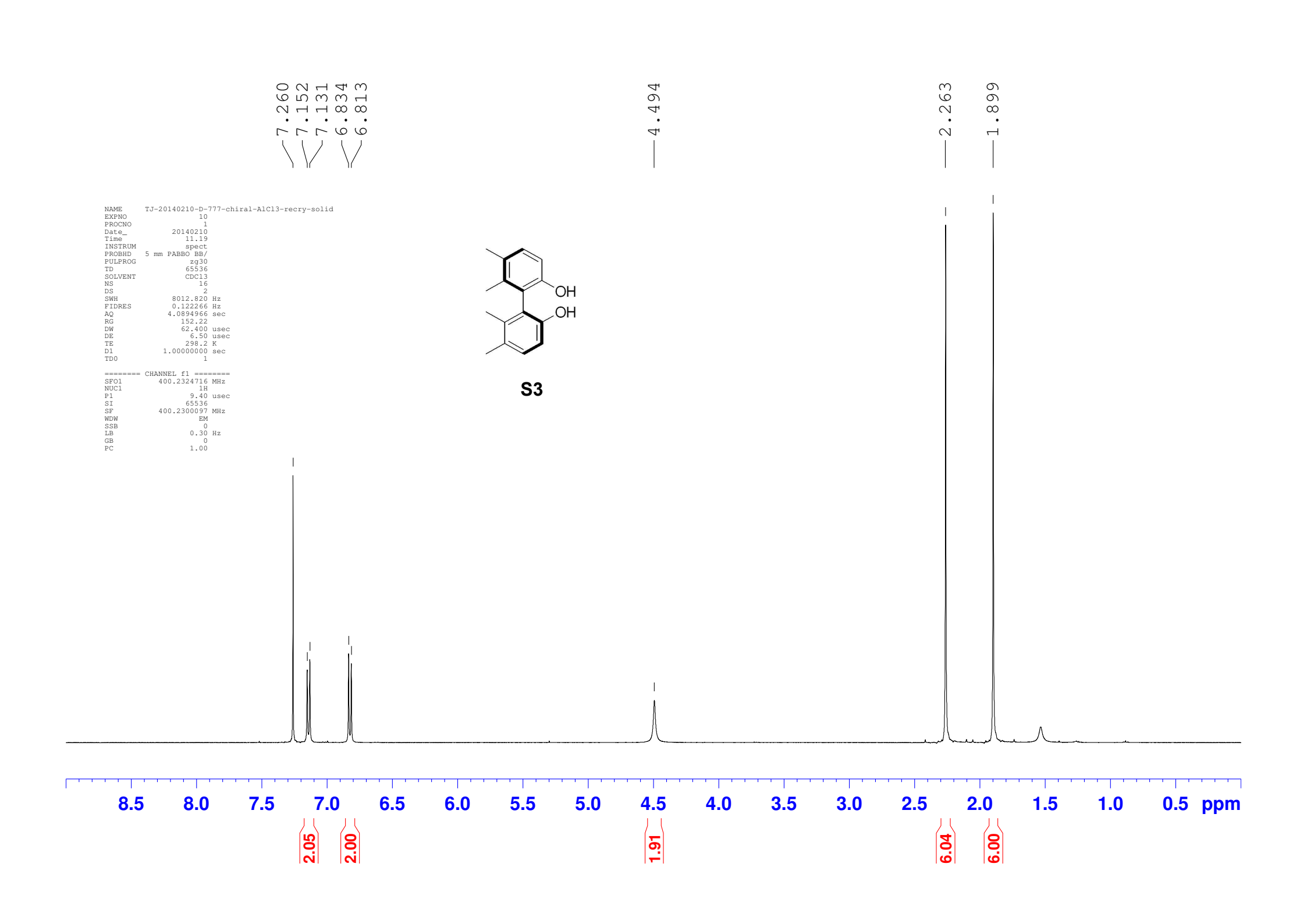

198.337
134.423
117.912
105.214
90.724
77.477
77.363
77.160
76.843
68.120
44.864
37.987
31.908
27.539
26.126
NAME TJ-20141106-E-1022-cy-LAH-frac
EXPNO 20
PROCNO 1
Date_ 20141110
Time 23.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3072
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 51.93 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.43 usec
PL1 -3.00 dB
PL1W 69.66502380 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 16.00 dB
PL12 15.00 dB
PL13 15.00 dB
PL2W 0.24733528 W
PL12W 0.31137666 W
PL13W 0.31137666 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
HO
OH
S1
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

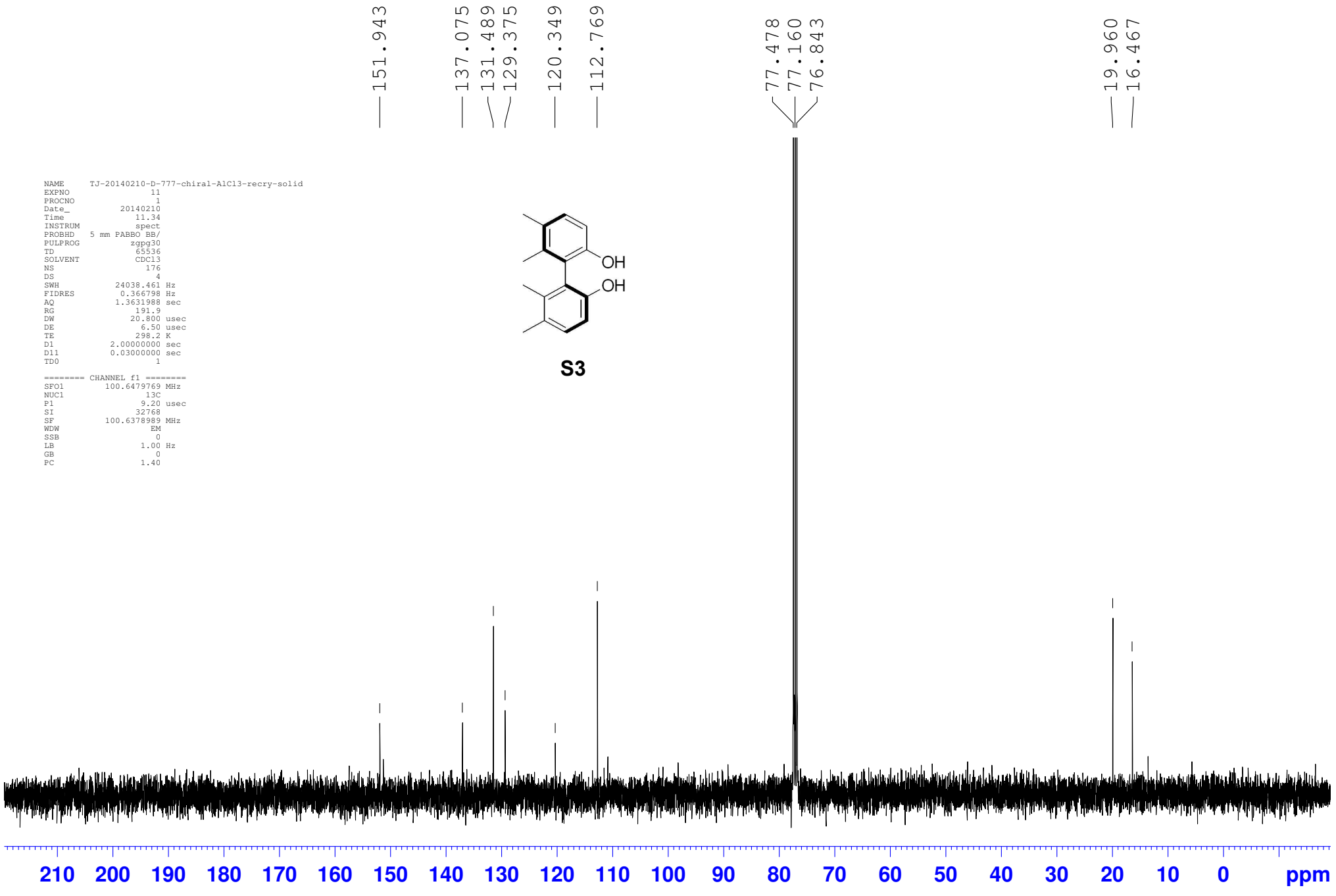

7.260
5.843
5.836
5.824
5.817
5.800
5.793
5.782
5.775
5.757
5.143
5.139
5.137
5.134
5.130
5.109
5.106
5.104
5.101
5.097
5.094
5.091
5.088
5.081
5.079
5.075
4.838
4.832
4.827
4.821
4.816
3.711
3.681
3.587
3.557
2.365
2.347
2.146
2.141
2.128
2.118
2.112
2.108
2.101
2.087
2.081
1.654
1.651
1.646
1.640
1.634
1.621
1.559
1.548
1.529
1.507
1.499
1.424
1.420
1m
NAME TJ-20141207-F-1041-X-protect-workup-crude
EXPNO 10
PROCNO 1
Date_ 20141207
Time 16.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 912
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.90 usec
PL1 -4.00 dB
PL1W 24.73352814 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
1.00
2.05
0.98
2.03
2.05
2.01
4.10
2.05
4.41
6.11

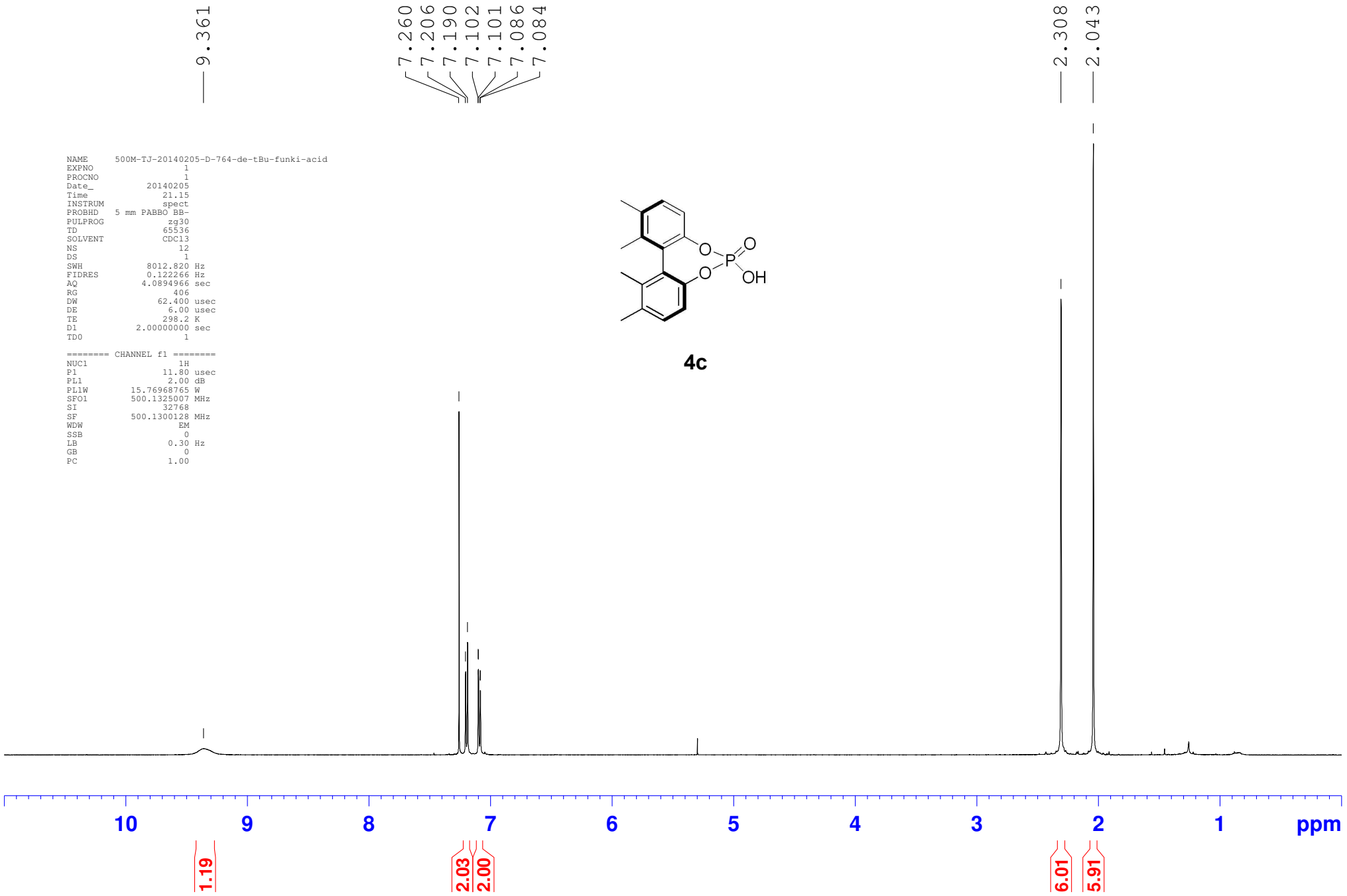

198.318
134.272
118.062
105.165
97.930
90.792
77.478
77.160
76.843
67.793
38.344
37.096
31.830
27.622
26.806
26.223
21.141
NAME TJ-20141207-F-1041-X-protect-workup-crude
EXPNO 12
PROCNO 1
Date_ 20141207
Time 22.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 51.93 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.43 usec
PL1 -3.00 dB
PL1W 69.66502380 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 16.00 dB
PL12 15.00 dB
PL13 15.00 dB
PL2W 0.24733528 W
PL12W 0.31137666 W
PL13W 0.31137666 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127540 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1m
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

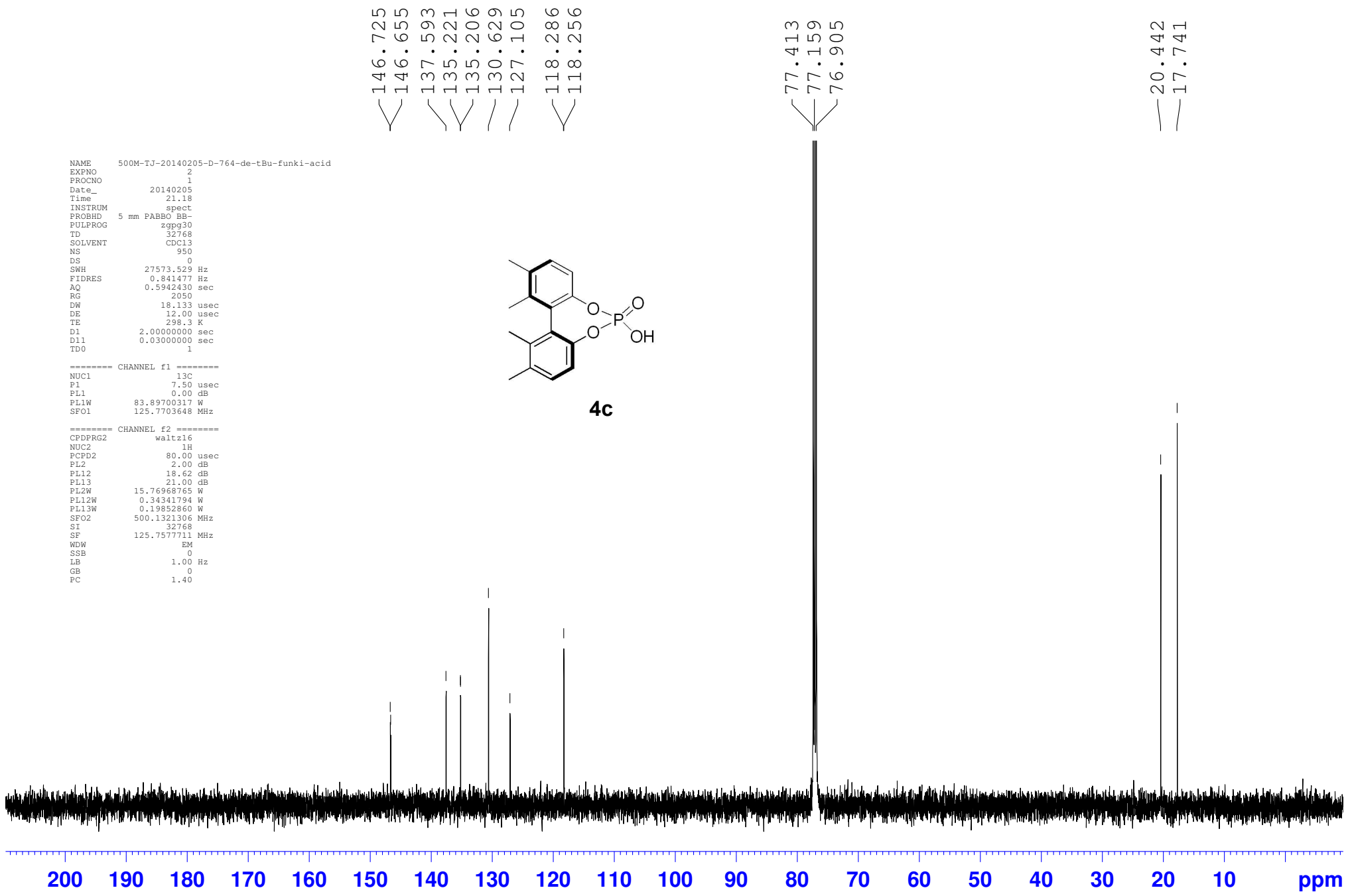

7.260
7.202
2.249
1.828
1.461
NAME TJ-20140826-(S)-funki-Pacid-re
EXPNO 10
PROCNO 1
Date_ 20140826
Time 12.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 96.29
DW 62.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.2424716 MHz
NUC1 1H
P1 9.40 usec
SI 65536
SF 400.2400096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
P
O
O
OH
4a
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
ppm
2.00
5.99
6.00
18.16

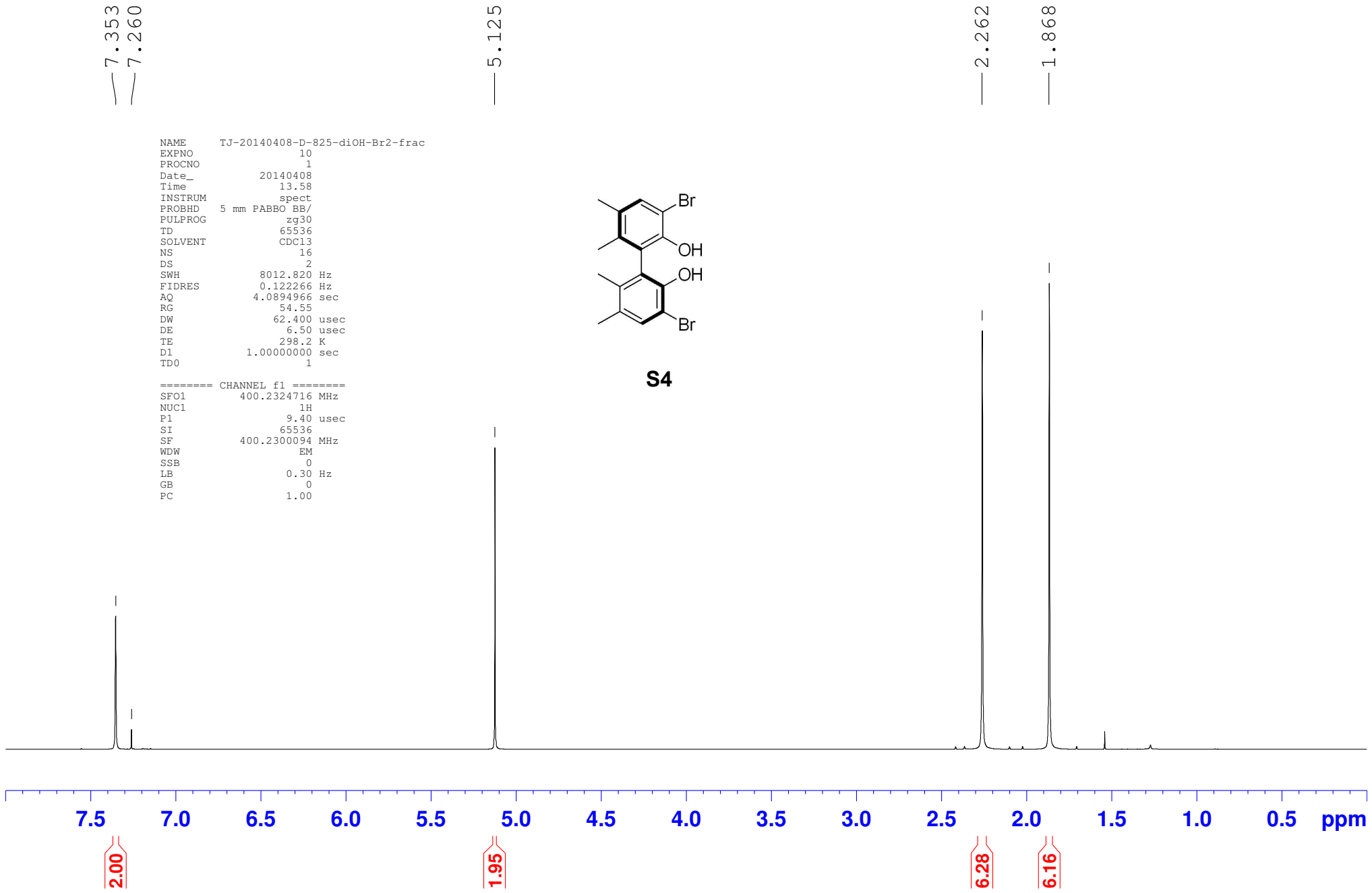

144.776
144.688
137.884
137.841
135.241
133.681
129.166
128.668
77.479
77.161
76.844
35.038
31.595
20.541
16.826
NAME TJ-20140826-(S)-funki-Pacid-re
EXPNO 13
PROCNO 1
Date_ 20140826
Time 12.53
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 176
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 191.9
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 100.6504912 MHz
NUC1 13C
P1 9.20 usec
SI 32768
SF 100.6404139 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
P
O
O
OH
4a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

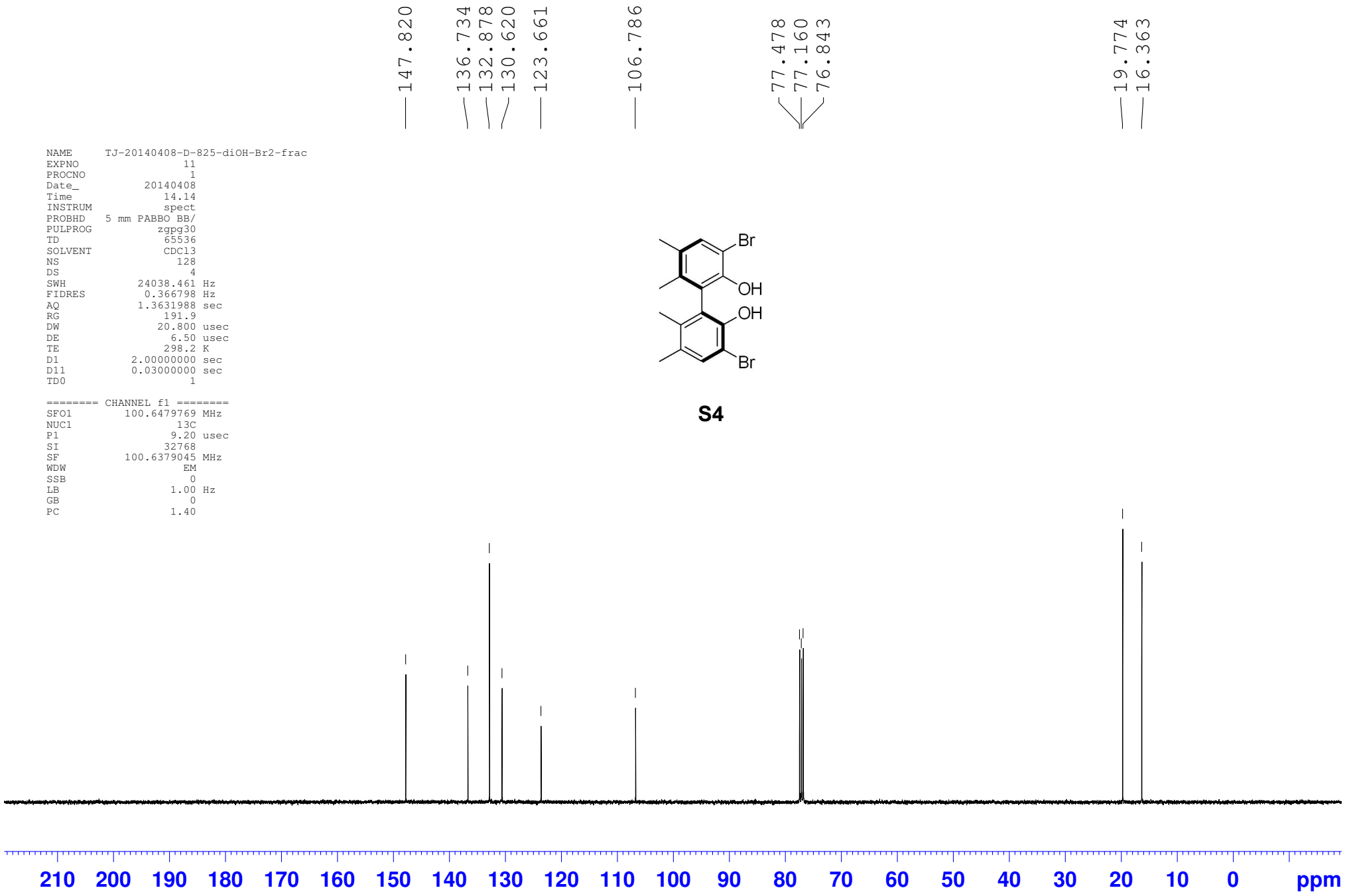

7.260
7.152
7.131
6.834
6.813
4.494
2.263
1.899
NAME TJ-20140210-D-777-chiral-AlCl3-recry-solid
EXPNO 10
PROCNO 1
Date_ 20140210
Time 11.19
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 152.22
DW 62.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.2324716 MHz
NUC1 1H
P1 9.40 usec
SI 65536
SF 400.2300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
OH
S3
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
ppm
2.05
2.00
1.91
6.04
6.00

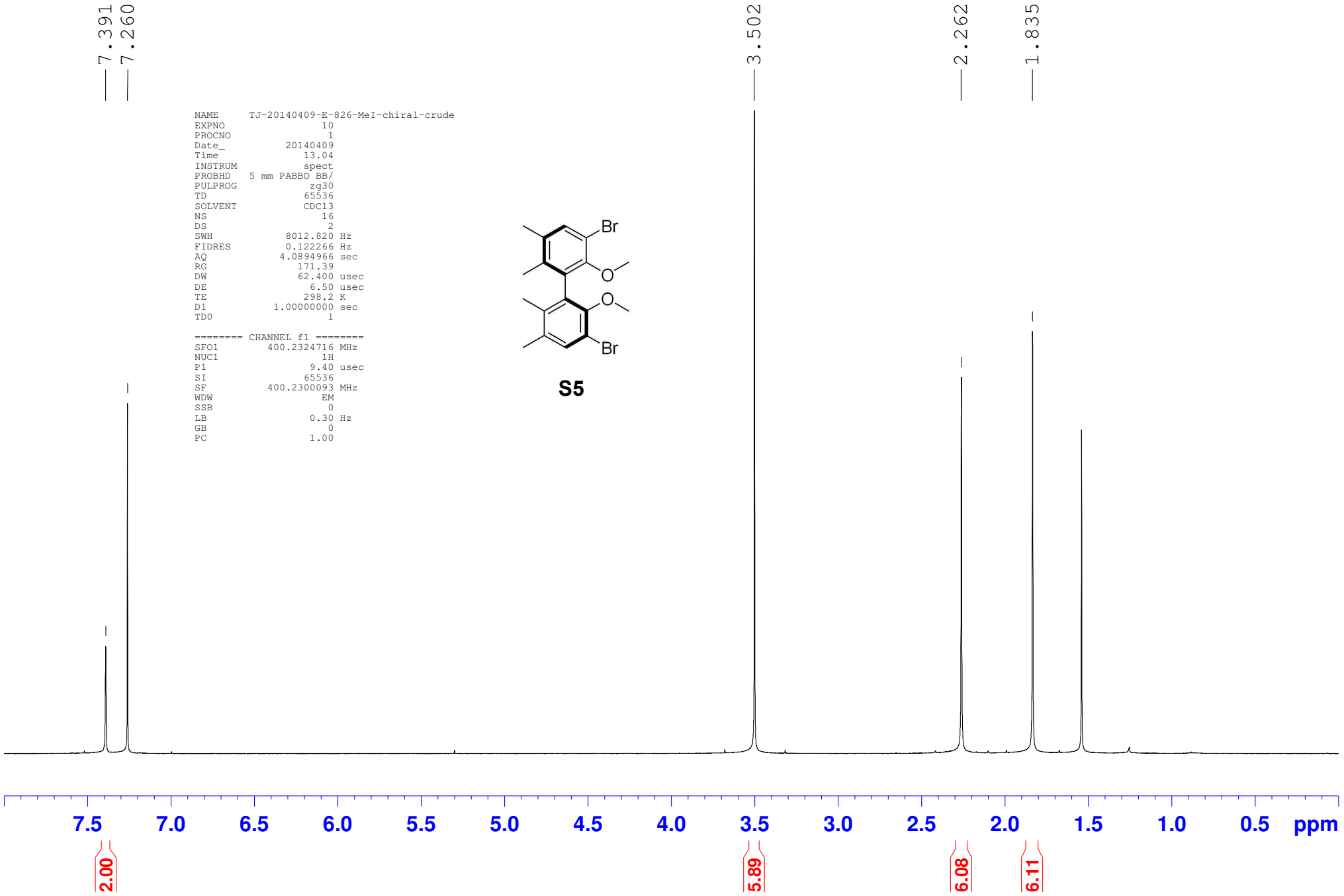

151.943
137.075
131.489
129.375
120.349
112.769
77.478
77.160
76.843
19.960
16.467
NAME TJ-20140210-D-777-chiral-AlCl3-recry-solid
EXPNO 11
PROCNO 1
Date_ 20140210
Time 11.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 176
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 191.9
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 100.6479769 MHz
NUC1 13C
P1 9.20 usec
SI 32768
SF 100.6378989 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OH
OH
S3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

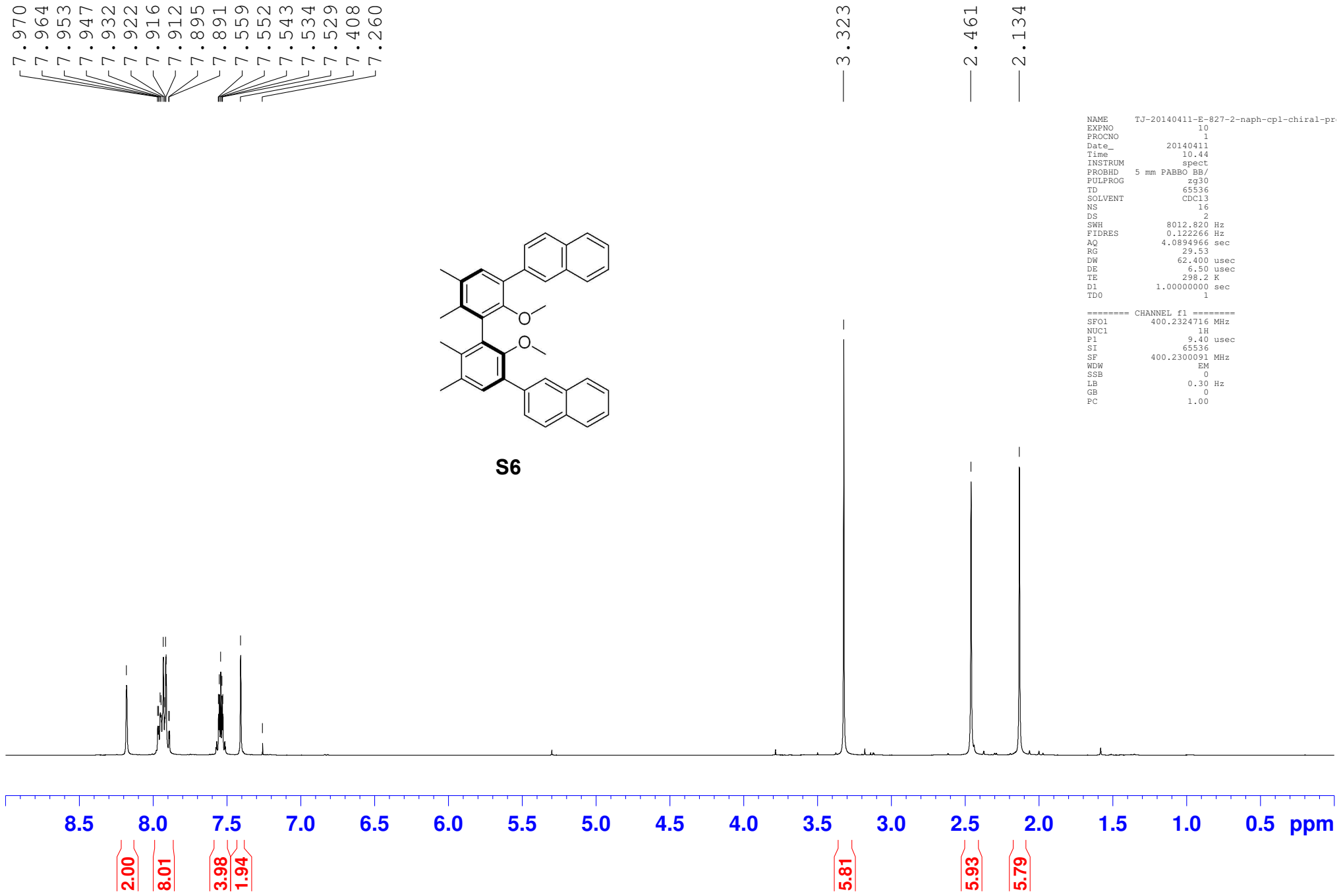

9.361
7.260
7.206
7.190
7.102
7.101
7.086
7.084
2.308
2.043
NAME 500M-TJ-20140205-D-764-de-tBu-funki-acid
EXPNO 1
PROCNO 1
Date_ 20140205
Time 21.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 12
DS 1
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 406
DW 62.400 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 2.00 dB
PL1W 15.76968765 W
SFO1 500.1325007 MHz
SI 32768
SF 500.1300128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
P
O
OH
4c
10
9
8
7
6
5
4
3
2
1
ppm
1.19
2.03
2.00
6.01
5.91

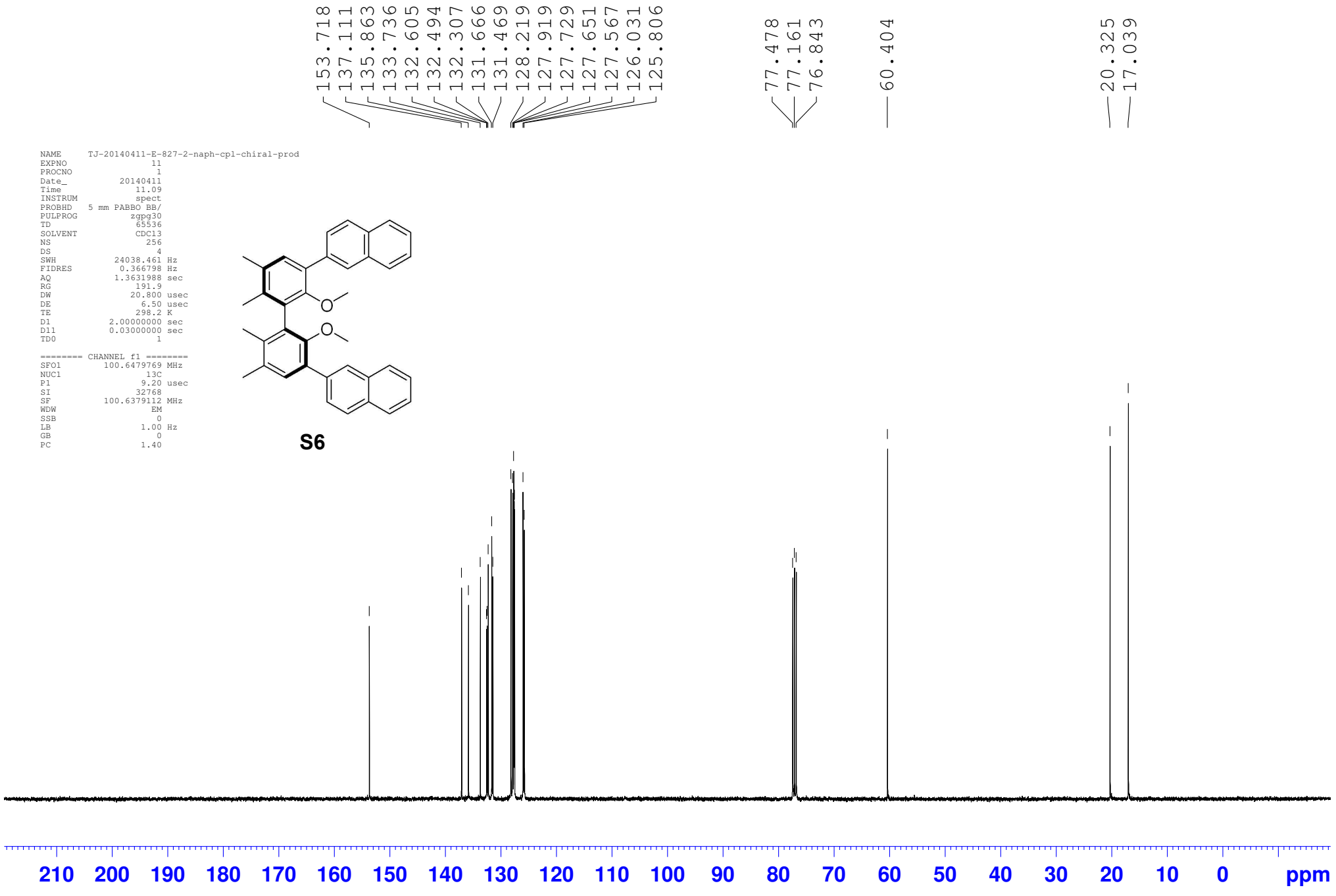

146.725
146.655
137.593
135.221
135.206
130.629
127.105
118.286
118.256
77.413
77.159
76.905
20.442
17.741
NAME 500M-TJ-20140205-D-764-de-tBu-funki-acid
EXPNO 2
PROCNO 1
Date_ 20140205
Time 21.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 950
DS 0
SWH 27573.529 Hz
FIDRES 0.841477 Hz
AQ 0.5942430 sec
RG 2050
DW 18.133 usec
DE 12.00 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PL1 0.00 dB
PL1W 83.89700317 W
SFO1 125.7703648 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 18.62 dB
PL13 21.00 dB
PL2W 15.76968765 W
PL12W 0.34341794 W
PL13W 0.19852860 W
SFO2 500.1321306 MHz
SI 32768
SF 125.7577711 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
O
P
O
OH
4c
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

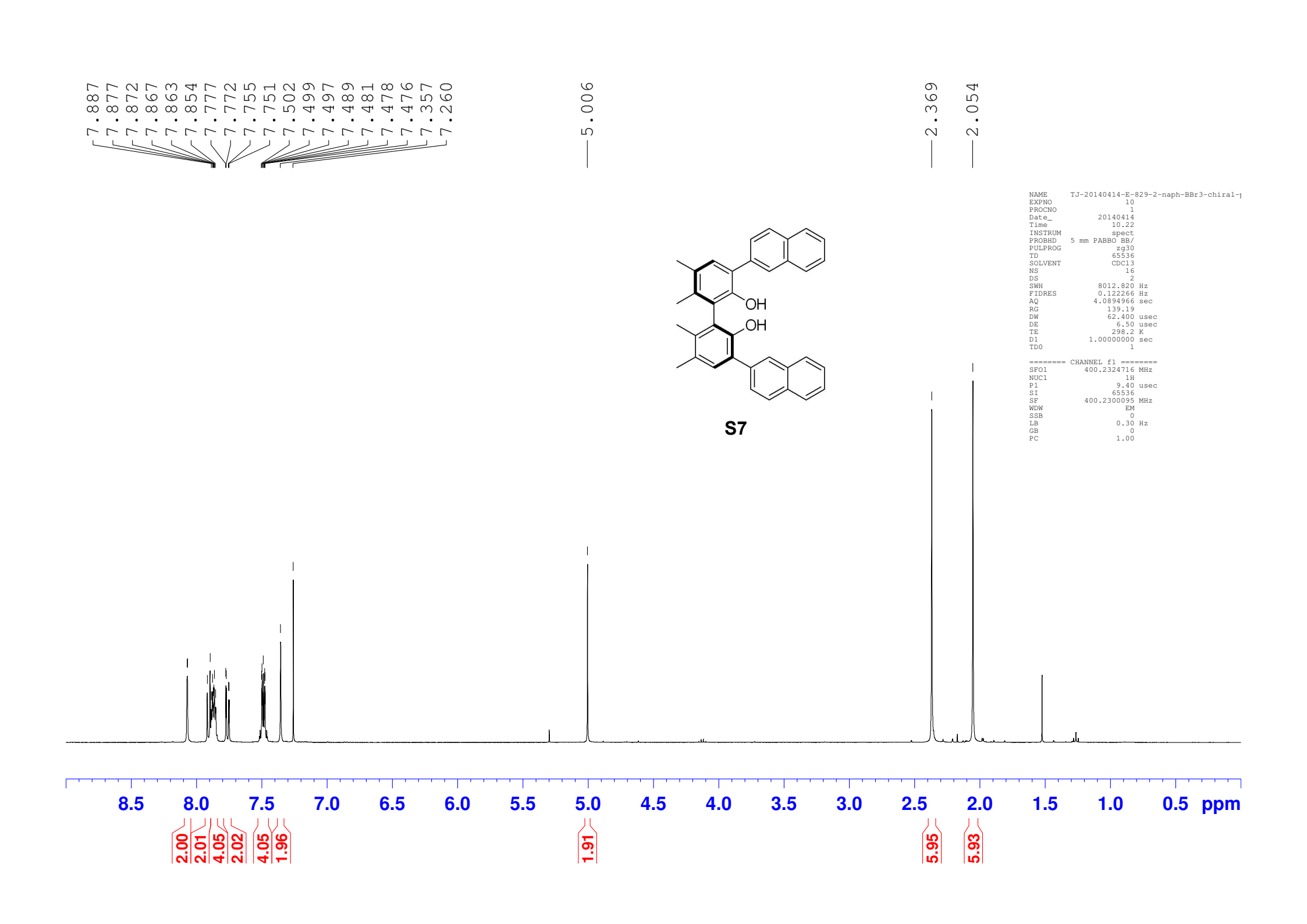

7.353
7.260
5.125
2.262
1.868
NAME TJ-20140408-D-825-diOH-Br2-frac
EXPNO 10
PROCNO 1
Date_ 20140408
Time 13.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 54.55
DW 62.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.2324716 MHz
NUC1 1H
P1 9.40 usec
SI 65536
SF 400.2300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Br
OH
OH
Br
S4
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
ppm
2.00
1.95
6.28
6.16

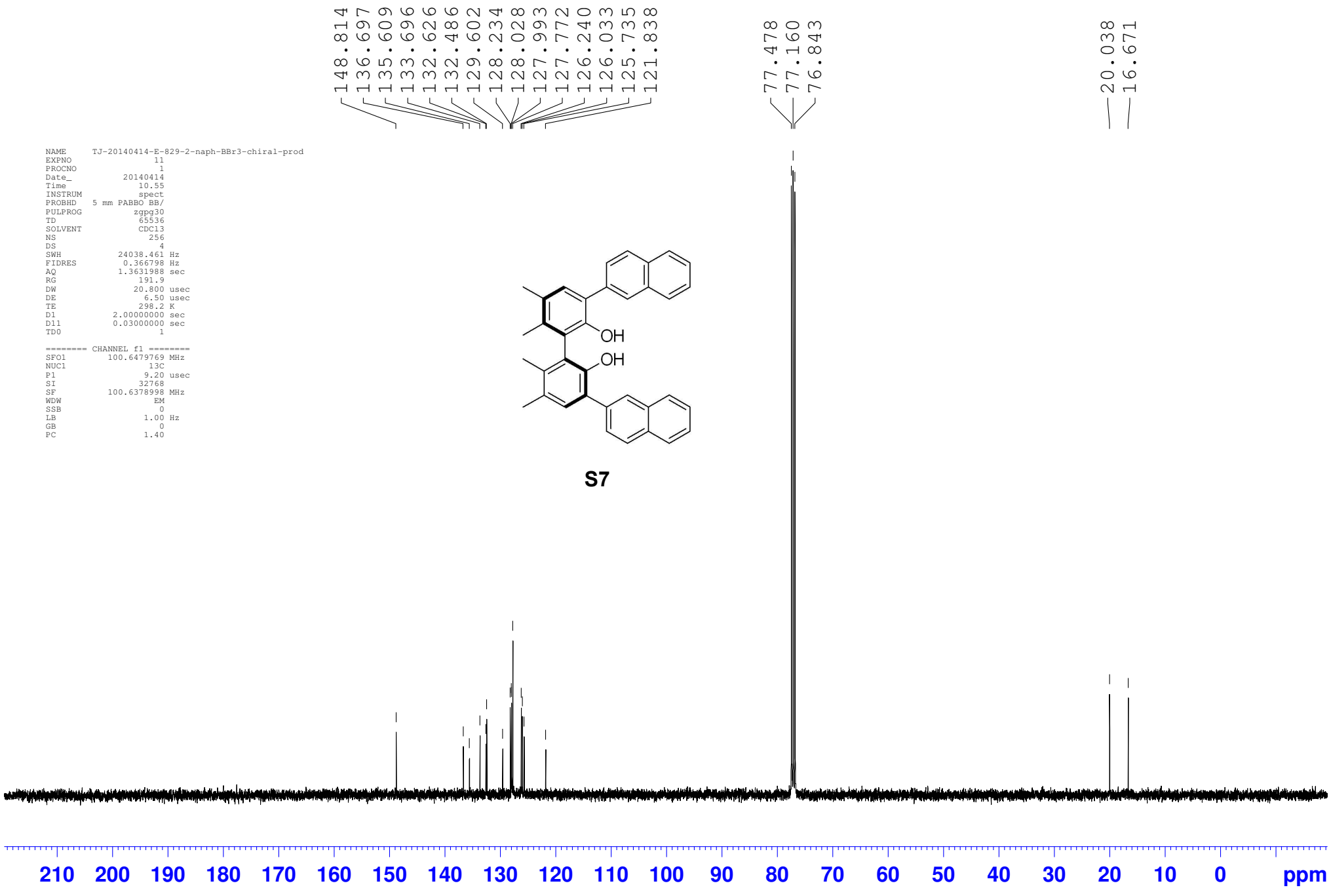

147.820
136.734
132.878
130.620
123.661
106.786
77.478
77.160
76.843
19.774
16.363
NAME TJ-20140408-D-825-diOH-Br2-frac
EXPNO 11
PROCNO 1
Date_ 20140408
Time 14.14
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 191.9
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 100.6479769 MHz
NUC1 13C
P1 9.20 usec
SI 32768
SF 100.6379045 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Br
OH
OH
Br
S4
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

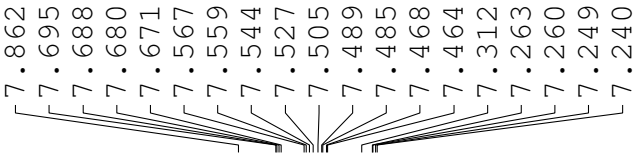

7.391
7.260
3.502
2.262
1.835
NAME TJ-20140409-E-826-MeI-chiral-crude
EXPNO 10
PROCNO 1
Date_ 20140409
Time 13.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 171.39
DW 62.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.2324716 MHz
NUC1 1H
P1 9.40 usec
SI 65536
SF 400.2300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Br
O
O
Br
S5
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
ppm
2.00
5.89
6.08
6.11

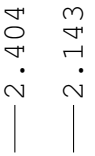

7.970
7.964
7.953
7.947
7.932
7.922
7.916
7.912
7.895
7.891
7.559
7.552
7.543
7.534
7.529
7.408
7.260
3.323
2.461
2.134
NAME TJ-20140411-E-827-2-naph-cpl-chiral-pr
EXPNO 10
PROCNO 1
Date_ 20140411
Time 10.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 29.53
DW 62.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.2324716 MHz
NUC1 1H
P1 9.40 usec
SI 65536
SF 400.2300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
S6
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
ppm
2.00
8.01
3.98
1.94
5.81
5.93
5.79

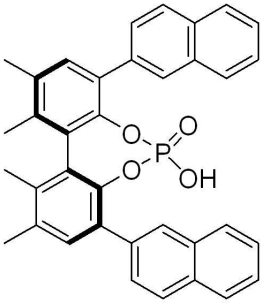

153.718
137.111
135.863
133.736
132.605
132.494
132.307
131.666
131.469
128.219
127.919
127.729
127.651
127.567
126.031
125.806
77.478
77.161
76.843
60.404
20.325
17.039
NAME TJ-20140411-E-827-2-naph-cpl-chiral-prod
EXPNO 11
PROCNO 1
Date_ 20140411
Time 11.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 191.9
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 100.6479769 MHz
NUC1 13C
P1 9.20 usec
SI 32768
SF 100.6379112 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
S6
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.887
7.877
7.872
7.867
7.863
7.854
7.777
7.772
7.755
7.751
7.502
7.499
7.497
7.489
7.481
7.478
7.476
7.357
7.260
5.006
2.369
2.054
OH
OH
S7
NAME TJ-20140414-E-829-2-naph-BBr3-chiral-
EXPNO 10
PROCNO 1
Date_ 20140414
Time 10.22
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 139.19
DW 62.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.2324716 MHz
NUC1 1H
P1 9.40 usec
SI 65536
SF 400.2300095 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
ppm
2.00
2.01
4.05
2.02
4.05
1.96
1.91
5.95
5.93


148.814
136.697
135.609
133.696
132.626
132.486
129.602
128.234
128.028
127.993
127.772
126.240
126.033
125.735
121.838
77.478
77.160
76.843
20.038
16.671
NAME TJ-20140414-E-829-2-naph-BBr3-chiral-prod
EXPNO 11
PROCNO 1
Date_ 20140414
Time 10.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 191.9
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 100.6479769 MHz
NUC1 13C
P1 9.20 usec
SI 32768
SF 100.6378998 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OH
OH
S7
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

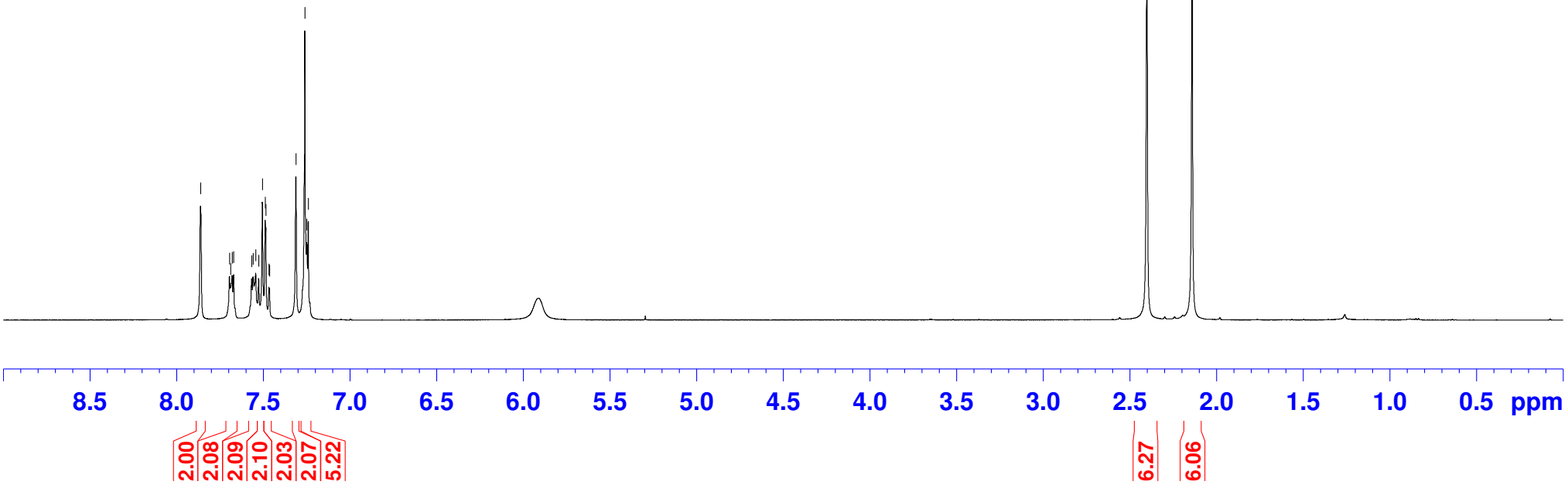


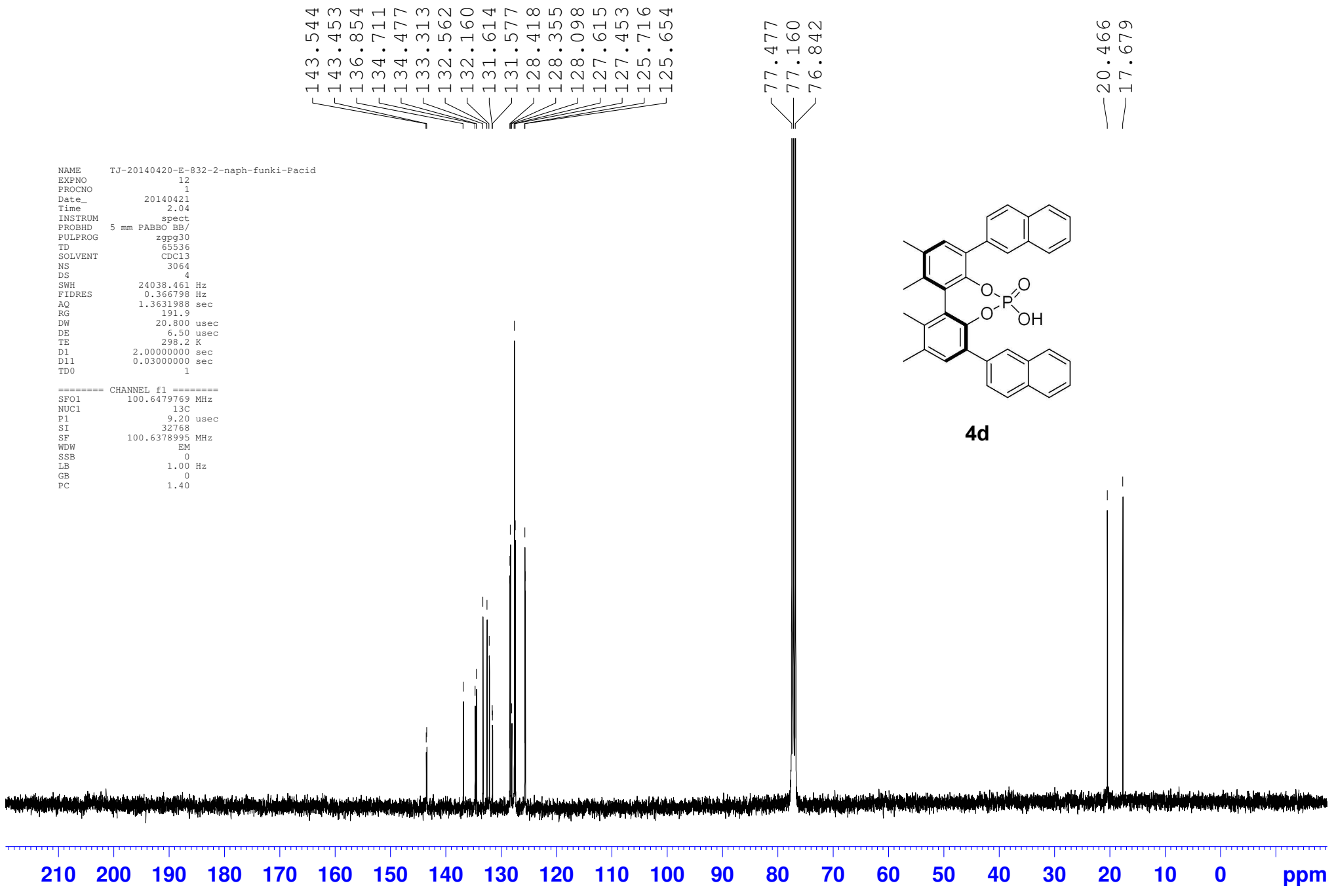


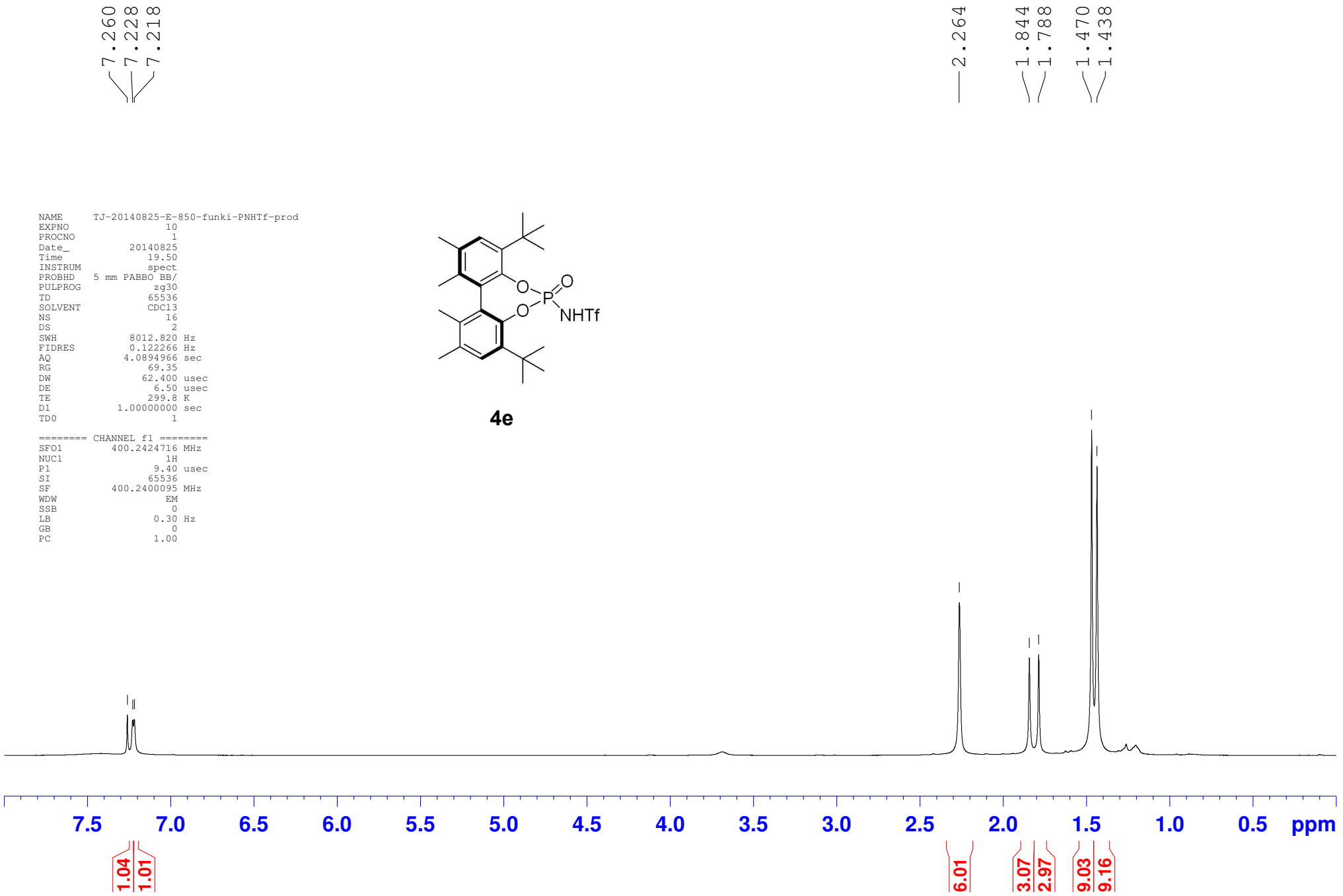


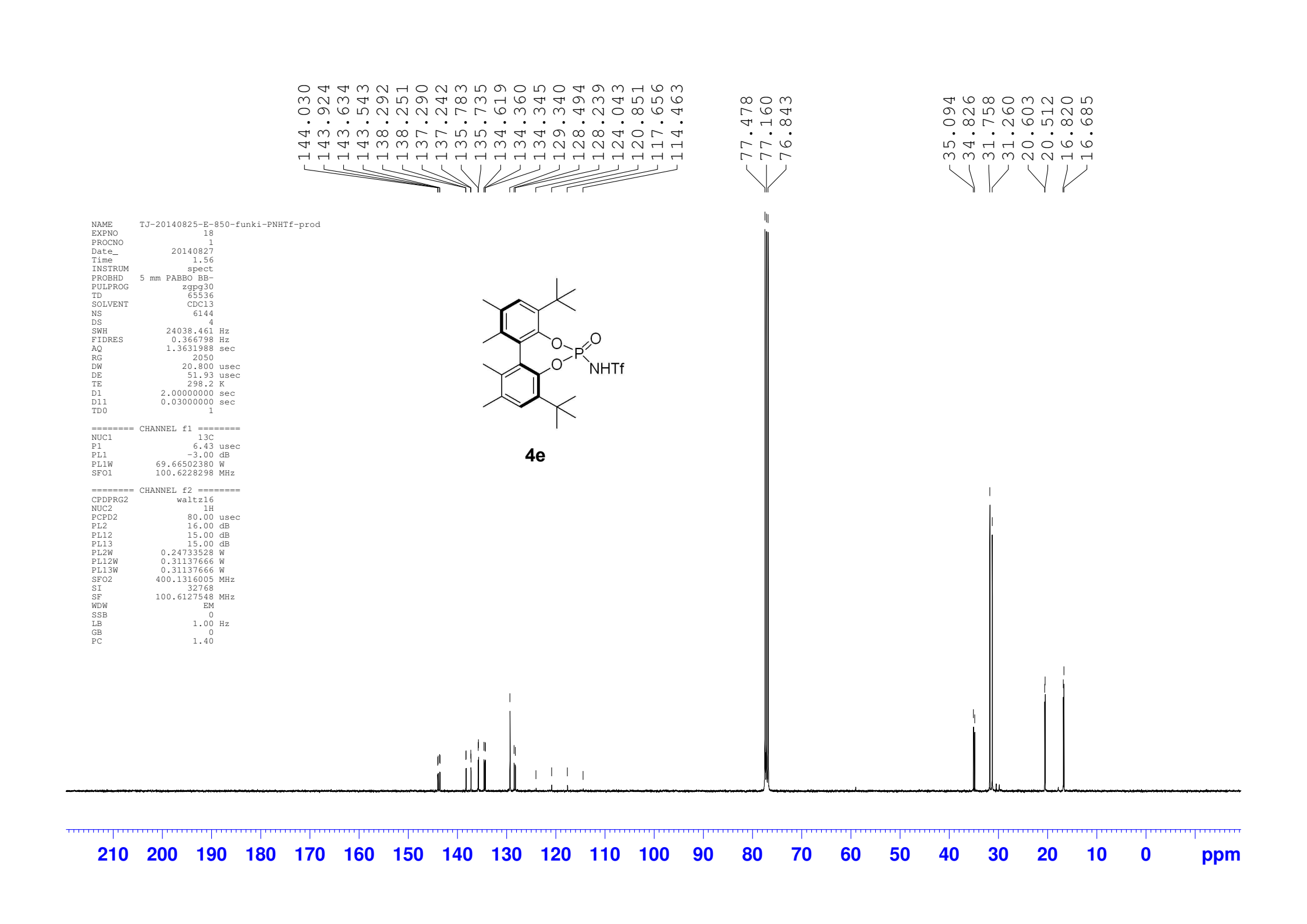
4d

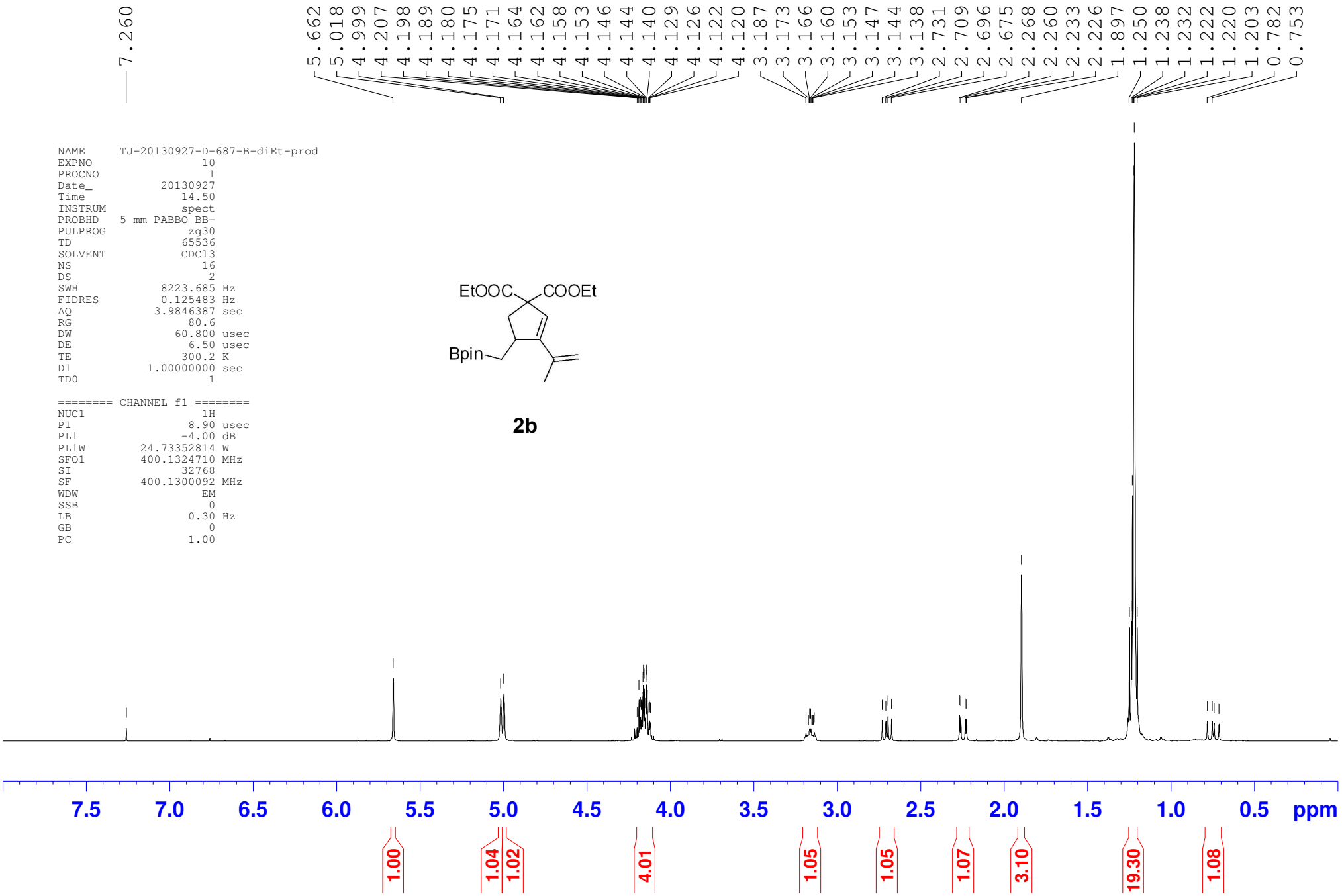
```
NAME      TJ-20140420-E-832-2-naph-funki-Pacid
EXPNO                 11
PROCNO                 1
Date_           20140420
Time               23.07
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES             0.122266 Hz
AQ              4.0894966 sec
RG                139.19
DW                62.400 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.2324716 MHz
NUC1                  1H
P1                 9.40 usec
SI                 65536
SF            400.2300094 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

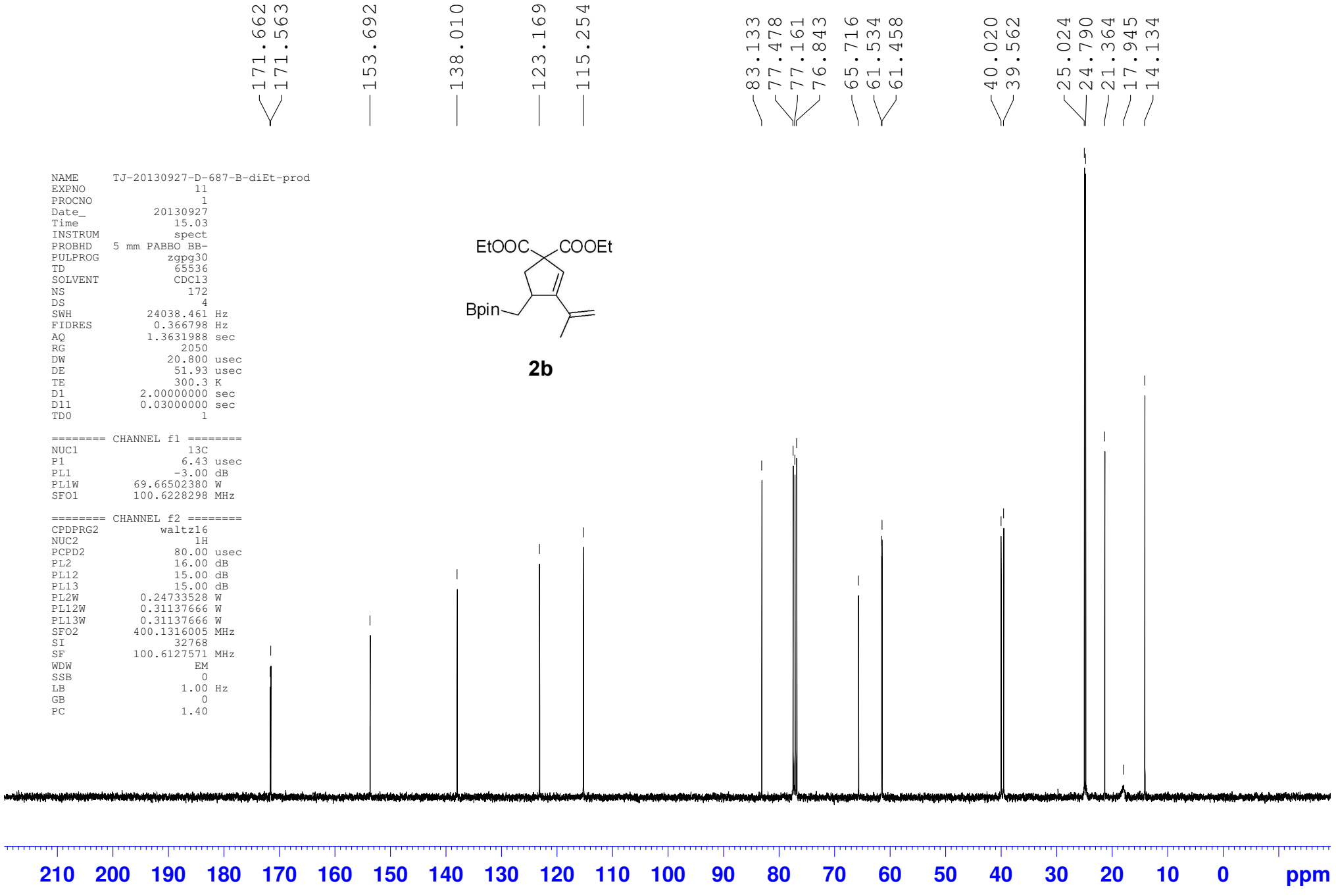


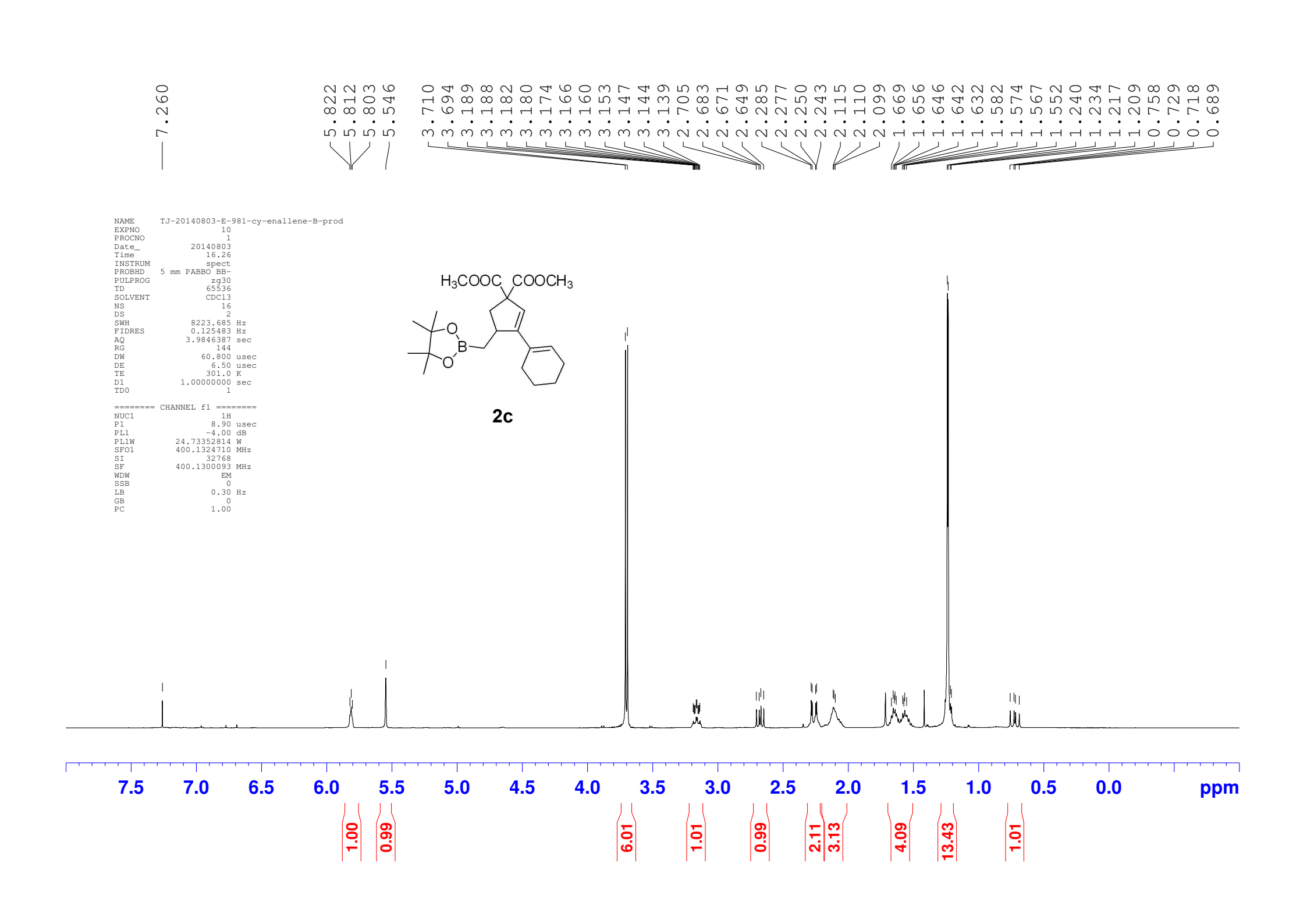

143.544
143.453
136.854
134.711
134.477
133.313
132.562
132.160
131.614
131.577
128.418
128.355
128.098
127.615
127.453
125.716
125.654
77.477
77.160
76.842
20.466
17.679
NAME TJ-20140420-E-832-2-naph-funki-Pacid
EXPNO 12
PROCNO 1
Date_ 20140421
Time 2.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3064
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 191.9
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 100.6479769 MHz
NUC1 13C
P1 9.20 usec
SI 32768
SF 100.6378995 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
O
P
O
OH
4d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

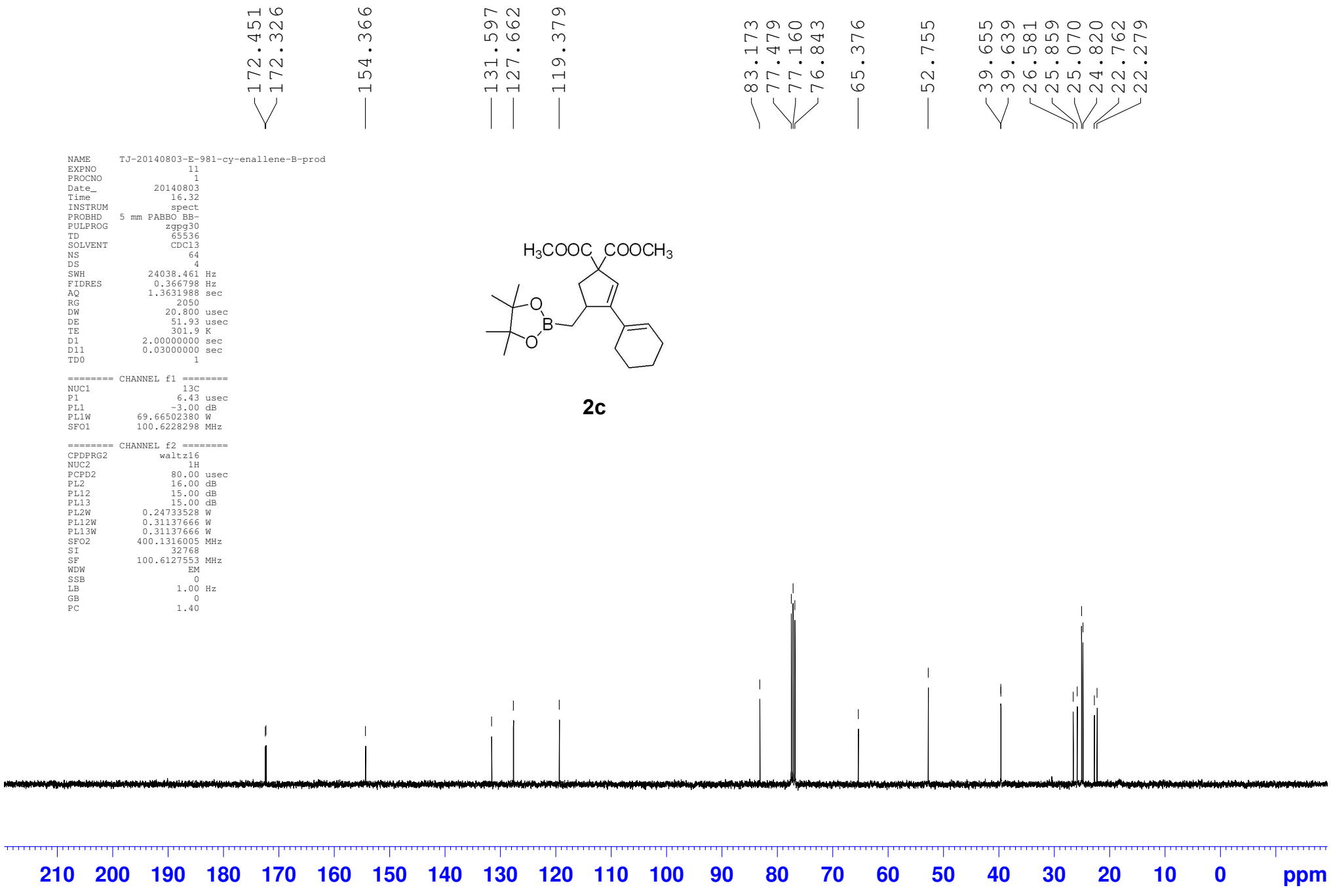

7.260
7.228
7.218
2.264
1.844
1.788
1.470
1.438
NAME TJ-20140825-E-850-funki-PNHTf-prod
EXPNO 10
PROCNO 1
Date_ 20140825
Time 19.50
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 69.35
DW 62.400 usec
DE 6.50 usec
TE 299.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.2424716 MHz
NUC1 1H
P1 9.40 usec
SI 65536
SF 400.2400095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
P
O
NHTf
4e
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
ppm
1.04
1.01
6.01
3.07
2.97
9.03
9.16

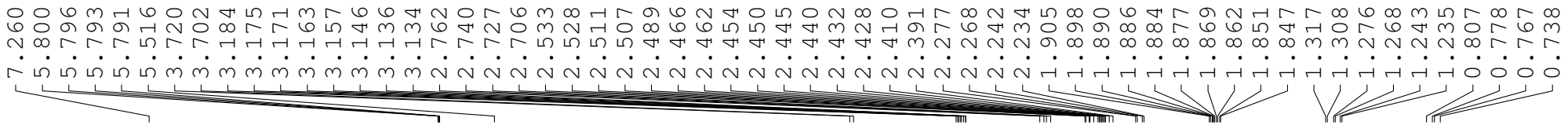

144.030
143.924
143.634
143.543
138.292
138.251
137.290
137.242
135.783
135.735
134.619
134.360
134.345
129.340
128.494
128.239
124.043
120.851
117.656
114.463
77.478
77.160
76.843
35.094
34.826
31.758
31.260
20.603
20.512
16.820
16.685
NAME TJ-20140825-E-850-funki-PNHTf-prod
EXPNO 18
PROCNO 1
Date_ 20140827
Time 1.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6144
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 51.93 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.43 usec
PL1 -3.00 dB
PL1W 69.66502380 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 16.00 dB
PL12 15.00 dB
PL13 15.00 dB
PL2W 0.24733528 W
PL12W 0.31137666 W
PL13W 0.31137666 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127548 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
O
P
O
NHTf
4e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.260
5.662
5.018
4.999
4.207
4.198
4.189
4.180
4.175
4.171
4.164
4.162
4.158
4.153
4.146
4.144
4.140
4.129
4.126
4.122
4.120
3.187
3.173
3.166
3.160
3.153
3.147
3.144
3.138
2.731
2.709
2.696
2.675
2.268
2.260
2.233
2.226
1.897
1.250
1.238
1.232
1.222
1.220
1.203
0.782
0.753
NAME TJ-20130927-D-687-B-diEt-prod
EXPNO 10
PROCNO 1
Date_ 20130927
Time 14.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 80.6
DW 60.800 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.90 usec
PL1 -4.00 dB
PL1W 24.73352814 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
EtOOC
COOEt
Bpin
2b
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
ppm
1.00
1.04
1.02
4.01
1.05
1.05
1.07
3.10
19.30
1.08

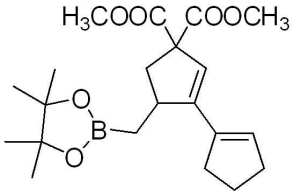

171.662
171.563
153.692
138.010
123.169
115.254
83.133
77.478
77.161
76.843
65.716
61.534
61.458
40.020
39.562
25.024
24.790
21.364
17.945
14.134
NAME TJ-20130927-D-687-B-diEt-prod
EXPNO 11
PROCNO 1
Date_ 20130927
Time 15.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 172
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 51.93 usec
TE 300.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.43 usec
PL1 -3.00 dB
PL1W 69.66502380 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 16.00 dB
PL12 15.00 dB
PL13 15.00 dB
PL2W 0.24733528 W
PL12W 0.31137666 W
PL13W 0.31137666 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127571 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
EtOOC
COOEt
Bpin
2b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.260
5.822
5.812
5.803
5.546
3.710
3.694
3.189
3.188
3.182
3.180
3.174
3.166
3.160
3.153
3.147
3.144
3.139
2.705
2.683
2.671
2.649
2.285
2.277
2.250
2.243
2.115
2.110
2.099
1.669
1.656
1.646
1.642
1.632
1.582
1.574
1.567
1.552
1.240
1.234
1.217
1.209
0.758
0.729
0.718
0.689
NAME TJ-20140803-E-981-cy-enallene-B-prod
EXPNO 10
PROCNO 1
Date_ 20140803
Time 16.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 144
DW 60.800 usec
DE 6.50 usec
TE 301.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.90 usec
PL1 -4.00 dB
PL1W 24.73352814 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H3COOC
COOCH3
O
B
O
2c
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
0.99
6.01
1.01
0.99
2.11
3.13
4.09
13.43
1.01


172.451
172.326
154.366
131.597
127.662
119.379
83.173
77.479
77.160
76.843
65.376
52.755
39.655
39.639
26.581
25.859
25.070
24.820
22.762
22.279
NAME TJ-20140803-E-981-cy-enallene-B-prod
EXPNO 11
PROCNO 1
Date_ 20140803
Time 16.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 51.93 usec
TE 301.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.43 usec
PL1 -3.00 dB
PL1W 69.66502380 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 16.00 dB
PL12 15.00 dB
PL13 15.00 dB
PL2W 0.24733528 W
PL12W 0.31137666 W
PL13W 0.31137666 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127553 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
H3COOC COOCH3
O
B
O
2c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



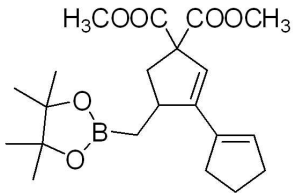

```
NAME      TJ-20140809-E-993-cyclopentyl-B-prod
EXPNO                 10
PROCNO                 1
Date_           20140809
Time               12.06
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   181
DW                60.800 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.90 usec
PL1               -4.00 dB
PL1W        24.73352814 W
SFO1        400.1324710 MHz
SI                 32768
SF          400.1300094 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```



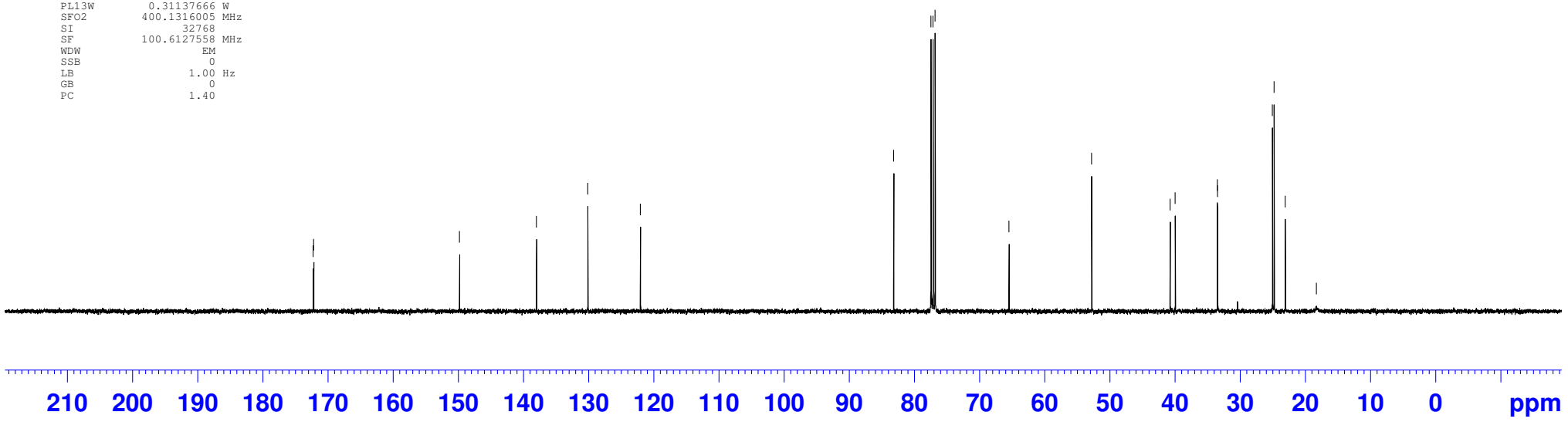
2d

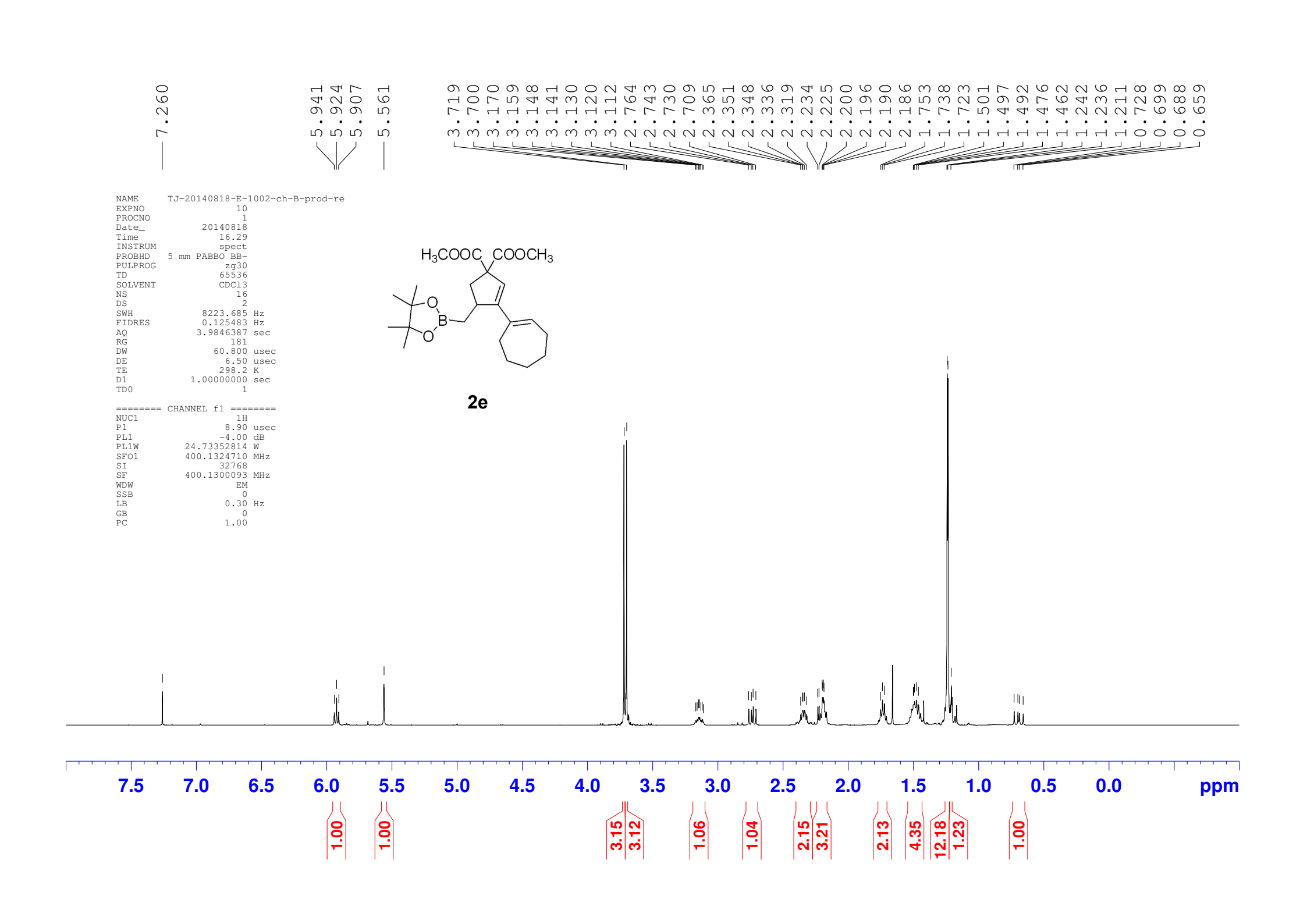
172.298
172.220
149.835
138.023
130.148
122.073
83.200
77.478
77.161
76.843
65.502
52.821
40.767
39.998
33.524
33.496
25.077
24.810
23.104
18.316

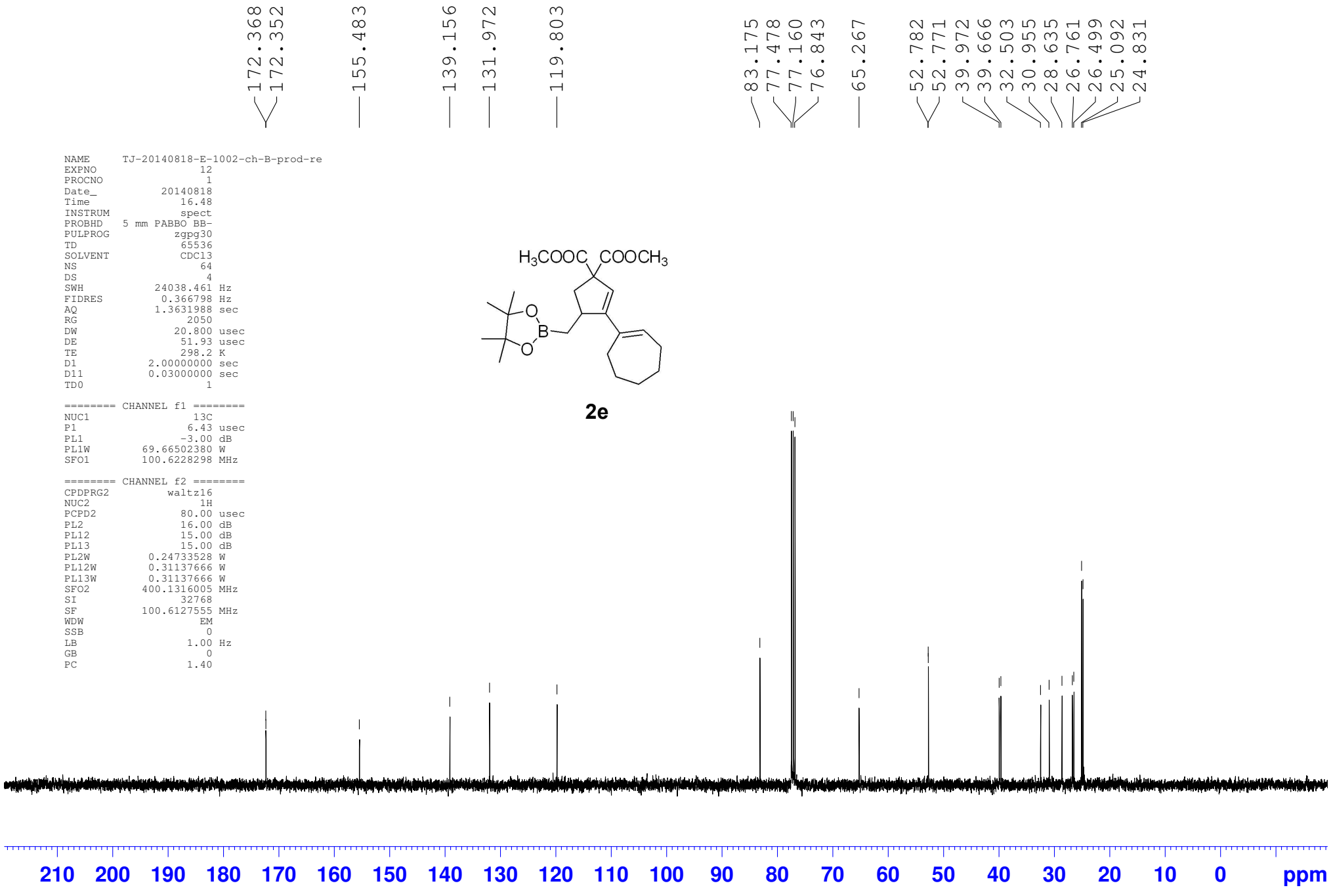
```
NAME      TJ-20140809-E-993-cyclopentyl-B-prod
EXPNO                  11
PROCNO                  1
Date_            20140809
Time                12.36
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     360
DS                       4
SWH              24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                    2050
DW                  20.800 usec
DE                  51.93 usec
TE                  298.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                    6.43 usec
PL1                  -3.00 dB
PL1W          69.66502380 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2                80.00 usec
PL2                  16.00 dB
PL12                 15.00 dB
PL13                 15.00 dB
PL2W           0.24733528 W
PL12W          0.31137666 W
PL13W          0.31137666 W
SFO2         400.1316005 MHz
SI                   32768
SF           100.6127558 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



2d




7.260
5.941
5.924
5.907
5.561
3.719
3.700
3.170
3.159
3.148
3.141
3.130
3.120
3.112
2.764
2.743
2.730
2.709
2.365
2.351
2.348
2.336
2.319
2.234
2.225
2.200
2.196
2.190
2.186
1.753
1.738
1.723
1.501
1.497
1.492
1.476
1.462
1.242
1.236
1.211
0.728
0.699
0.688
0.659
NAME TJ-20140818-E-1002-ch-B-prod-re
EXPNO 10
PROCNO 1
Date_ 20140818
Time 16.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 181
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.90 usec
PL1 -4.00 dB
PL1W 24.73352814 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H3COOC COOCH3
O
B
O
2e
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
3.15
3.12
1.06
1.04
2.15
3.21
2.13
4.35
12.18
1.23
1.00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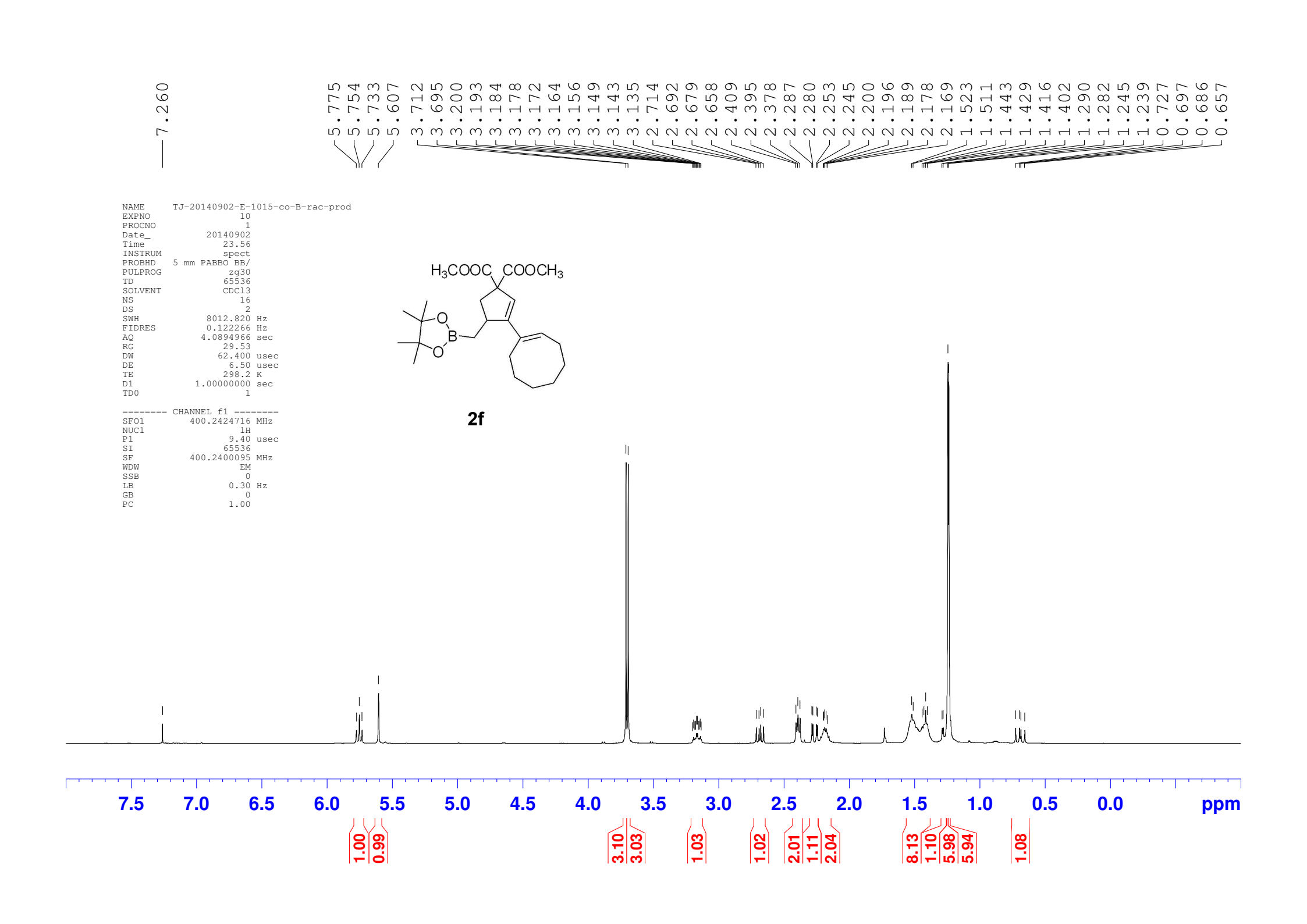
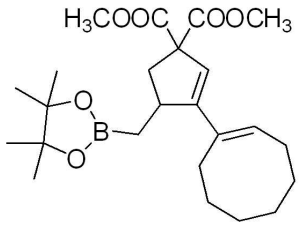
172.352
155.483
139.156
131.972
119.803
83.175
77.478
77.160
76.843
65.267
52.782
52.771
39.972
39.666
32.503
30.955
28.635
26.761
26.499
25.092
24.831
NAME TJ-20140818-E-1002-ch-B-prod-re
EXPNO 12
PROCNO 1
Date_ 20140818
Time 16.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 51.93 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.43 usec
PL1 -3.00 dB
PL1W 69.66502380 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 16.00 dB
PL12 15.00 dB
PL13 15.00 dB
PL2W 0.24733528 W
PL12W 0.31137666 W
PL13W 0.31137666 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127555 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
H3COOC COOCH3
O
B
O
2e
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.260
5.775
5.754
5.733
5.607
3.712
3.695
3.200
3.193
3.184
3.178
3.172
3.164
3.156
3.149
3.143
3.135
2.714
2.692
2.679
2.658
2.409
2.395
2.378
2.287
2.280
2.253
2.245
2.200
2.196
2.189
2.178
2.169
1.523
1.511
1.443
1.429
1.416
1.402
1.290
1.282
1.245
1.239
0.727
0.697
0.686
0.657
NAME TJ-20140902-E-1015-co-B-rac-prod
EXPNO 10
PROCNO 1
Date_ 20140902
Time 23.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 29.53
DW 62.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.2424716 MHz
NUC1 1H
P1 9.40 usec
SI 65536
SF 400.2400095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H3COOC COOCH3
O
B
O
2f
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
0.99
3.10
3.03
1.03
1.02
2.01
1.11
2.04
8.13
1.10
5.98
5.94
1.08

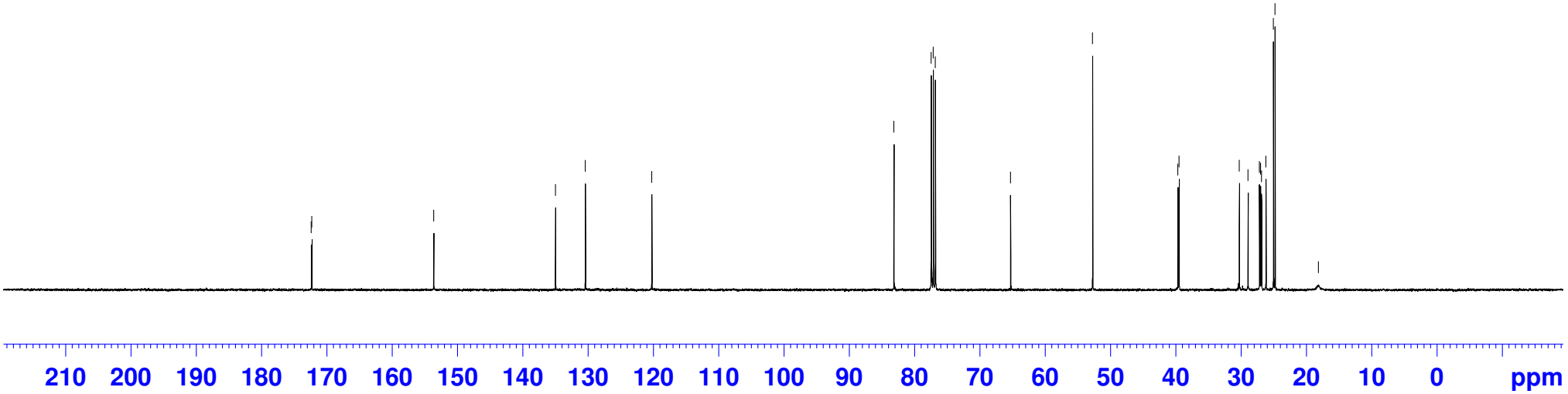
172.411
172.318
153.649
135.000
130.442
120.263
83.186
77.478
77.160
76.843
65.321
52.769
39.703
39.517
30.296
28.957
27.212
27.032
26.889
26.214
25.071
24.822
18.172

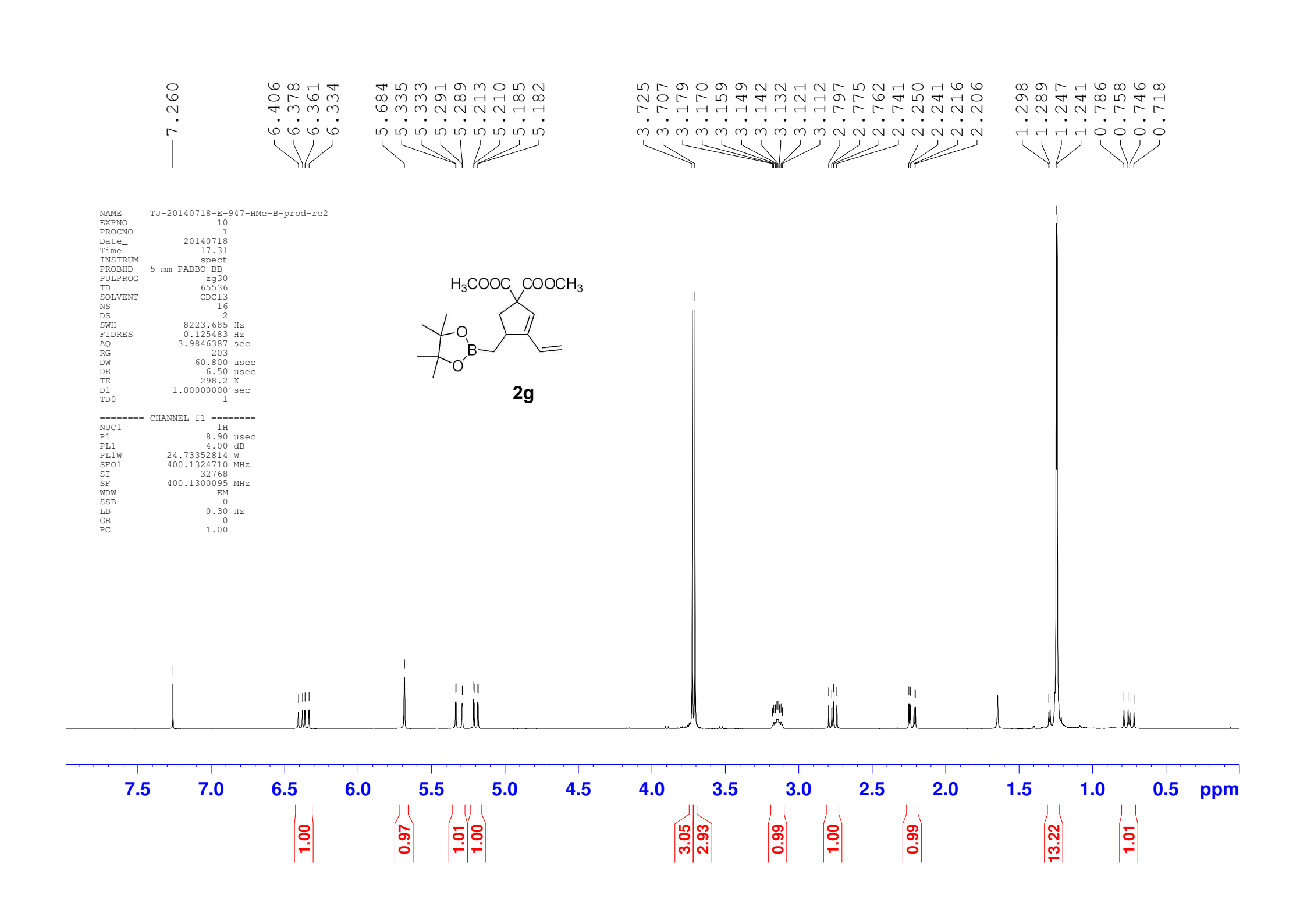
```
NAME      TJ-20140902-E-1015-co-B-rac-prod
EXPNO                12
PROCNO                1
Date_          20140903
Time               0.54
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  876
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                191.9
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         100.6504912 MHz
NUC1                13C
P1                 9.20 usec
SI                32768
SF           100.6404162 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



2f

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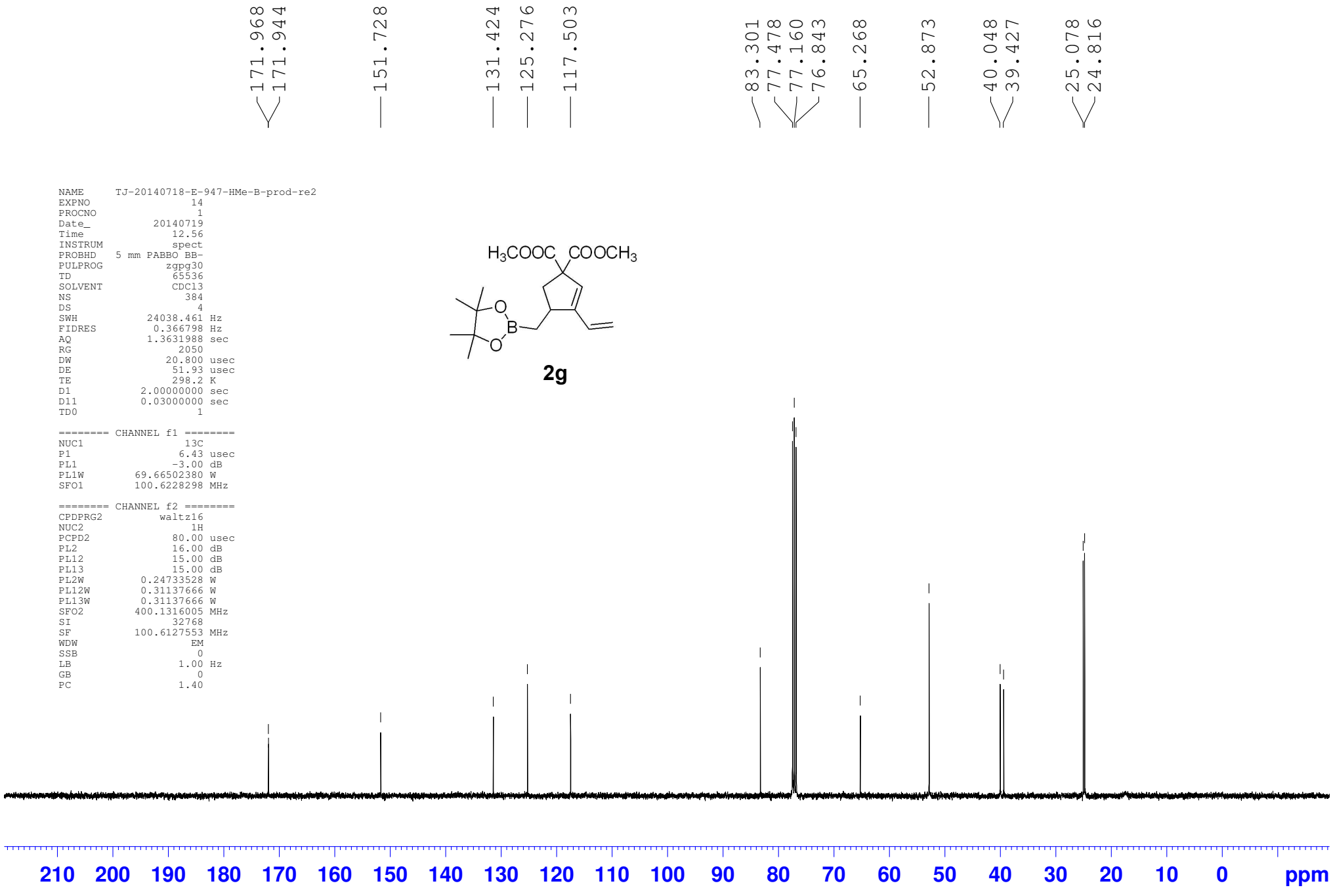
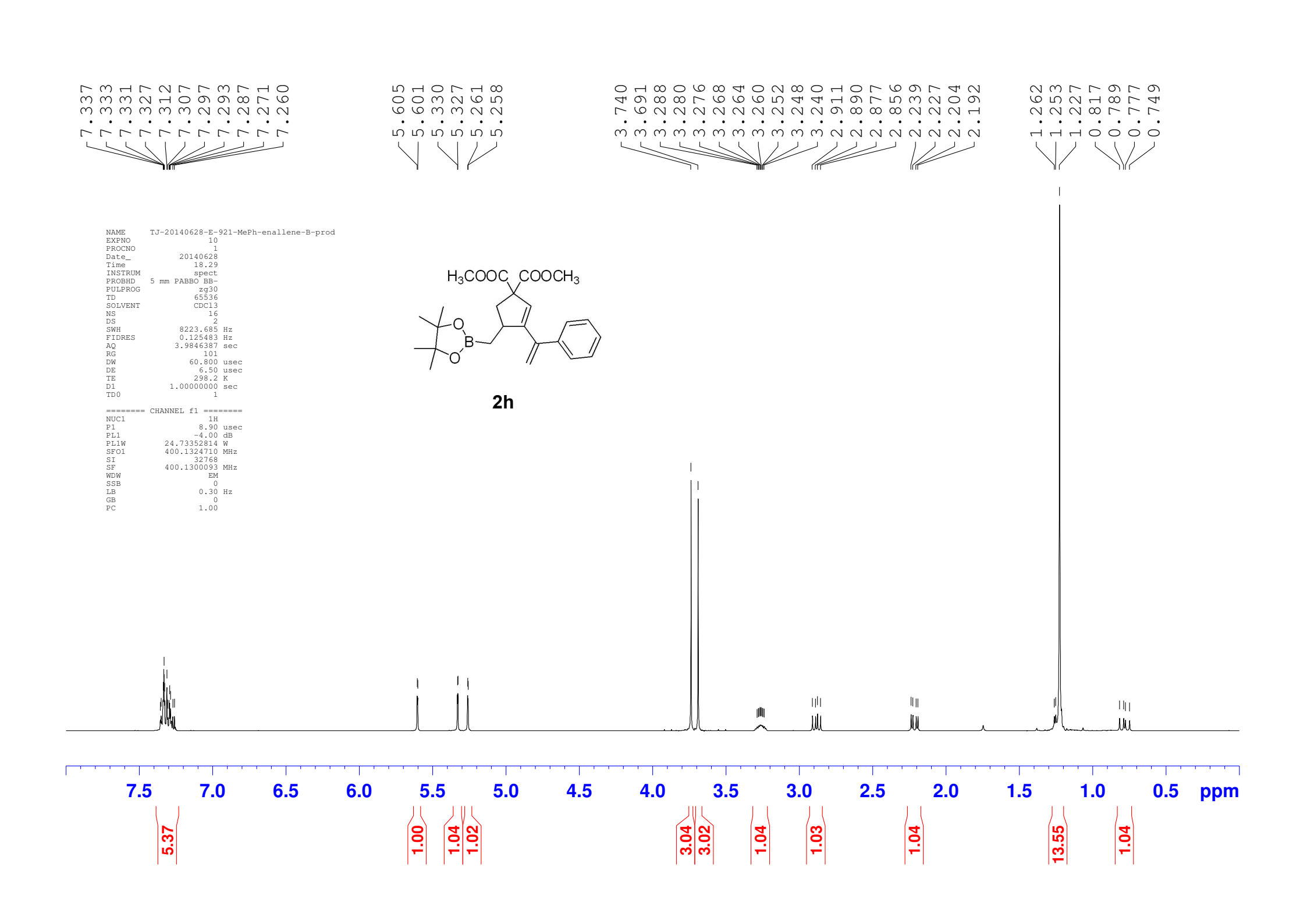
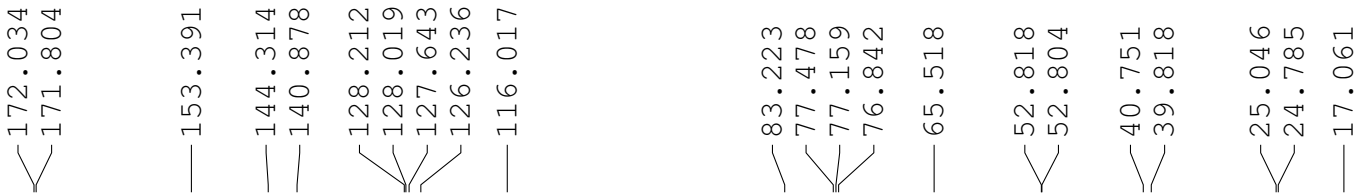
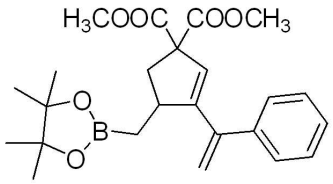
6.406
6.378
6.361
6.334
5.684
5.335
5.333
5.291
5.289
5.213
5.210
5.185
5.182
3.725
3.707
3.179
3.170
3.159
3.149
3.142
3.132
3.121
3.112
2.797
2.775
2.762
2.741
2.250
2.241
2.216
2.206
1.298
1.289
1.247
1.241
0.786
0.758
0.746
0.718
NAME TJ-20140718-E-947-HMe-B-prod-re2
EXPNO 10
PROCNO 1
Date_ 20140718
Time 17.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.90 usec
PL1 -4.00 dB
PL1W 24.73352814 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H3COOC COOCH3
O
B
O
2g
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
ppm
1.00
0.97
1.01
1.00
3.05
2.93
0.99
1.00
0.99
13.22
1.01


171.968
171.944
151.728
131.424
125.276
117.503
83.301
77.478
77.160
76.843
65.268
52.873
40.048
39.427
25.078
24.816
NAME TJ-20140718-E-947-HMe-B-prod-re2
EXPNO 14
PROCNO 1
Date_ 20140719
Time 12.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 384
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 51.93 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.43 usec
PL1 -3.00 dB
PL1W 69.66502380 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 16.00 dB
PL12 15.00 dB
PL13 15.00 dB
PL2W 0.24733528 W
PL12W 0.31137666 W
PL13W 0.31137666 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127553 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
H3COOC COOCH3
O
B
O
2g
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

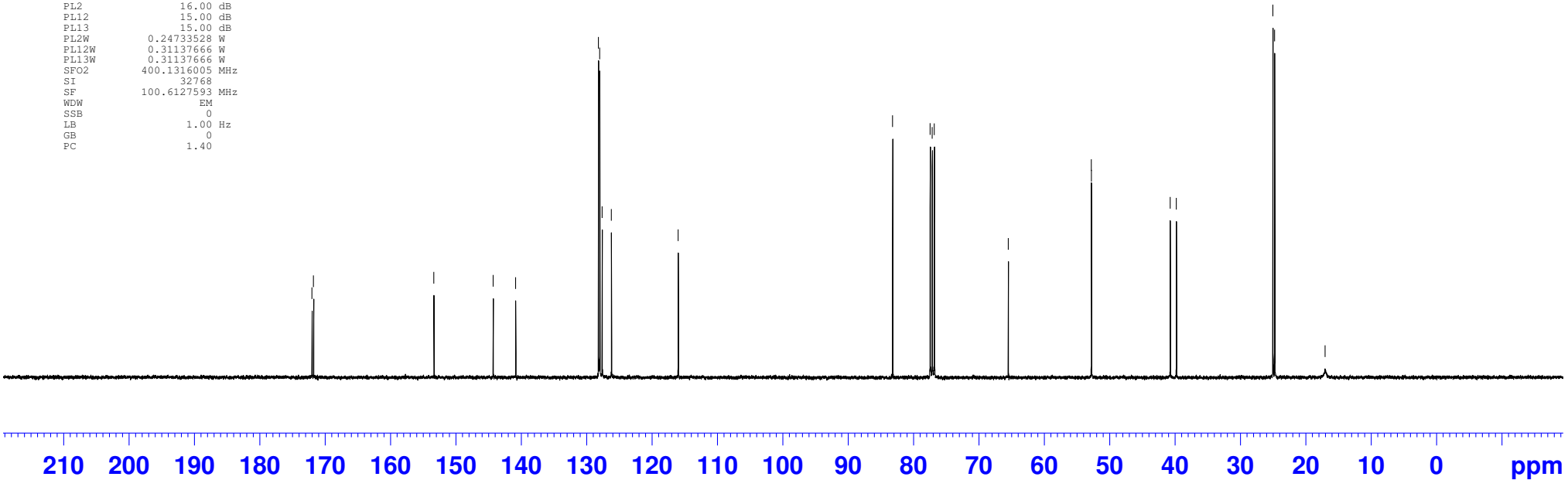

7.337
7.333
7.331
7.327
7.312
7.307
7.297
7.293
7.287
7.271
7.260
5.605
5.601
5.330
5.327
5.261
5.258
3.740
3.691
3.288
3.280
3.276
3.268
3.264
3.260
3.252
3.248
3.240
2.911
2.890
2.877
2.856
2.239
2.227
2.204
2.192
1.262
1.253
1.227
0.817
0.789
0.777
0.749
NAME TJ-20140628-E-921-MePh-enallene-B-prod
EXPNO 10
PROCNO 1
Date_ 20140628
Time 18.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 101
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.90 usec
PL1 -4.00 dB
PL1W 24.73352814 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H3COOC COOCH3
O
B
O
2h
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
ppm
5.37
1.00
1.04
1.02
3.04
3.02
1.04
1.03
1.04
13.55
1.04

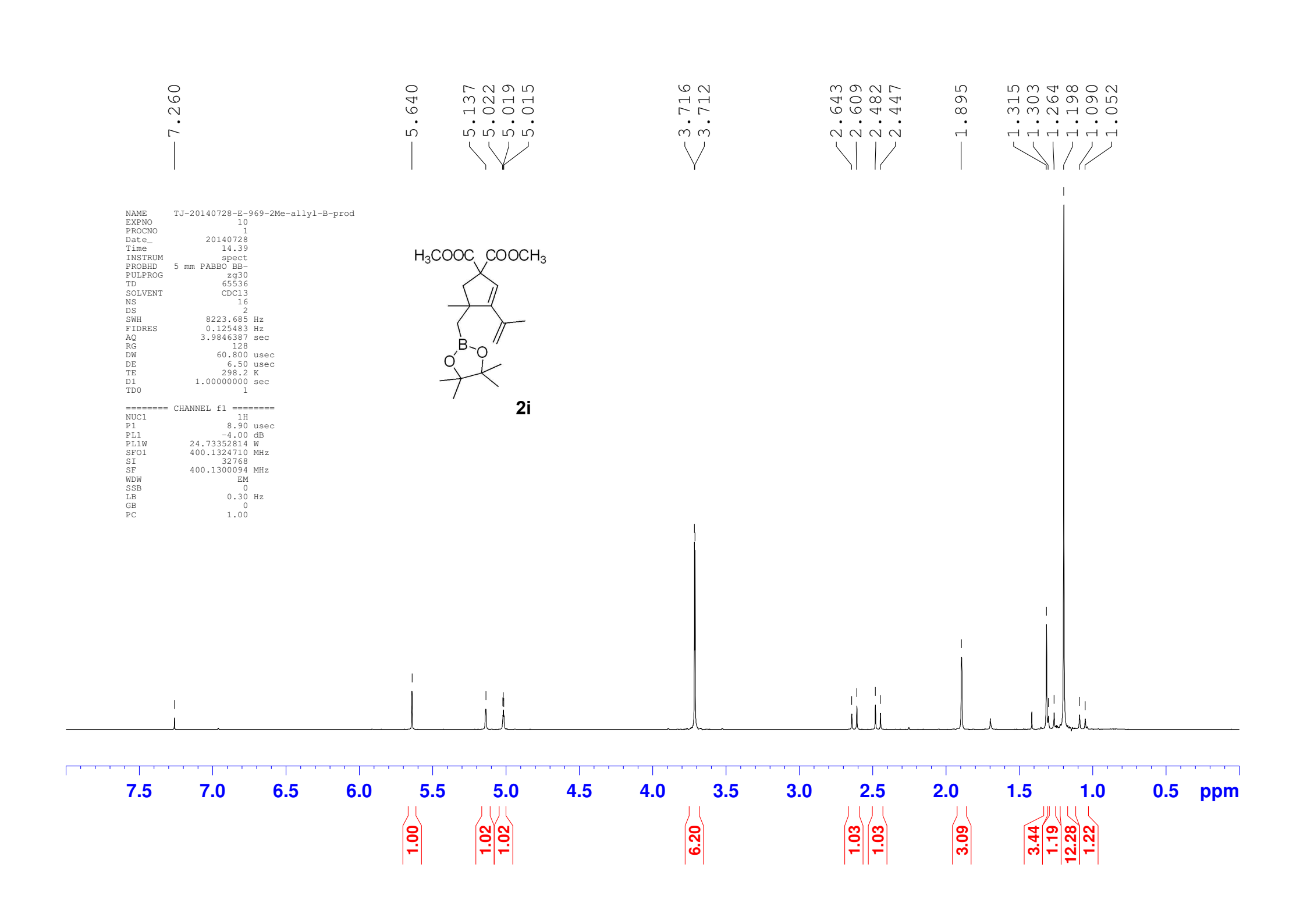



```
NAME      TJ-20140628-E-921-MePh-enallene-B-prod
EXPNO                12
PROCNO                1
Date_          20140629
Time               2.30
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  384
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                 2050
DW               20.800 usec
DE               51.93 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  6.43 usec
PL1                -3.00 dB
PL1W        69.66502380 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                16.00 dB
PL12               15.00 dB
PL13               15.00 dB
PL2W         0.24733528 W
PL12W        0.31137666 W
PL13W        0.31137666 W
SFO2       400.1316005 MHz
SI                32768
SF         100.6127593 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



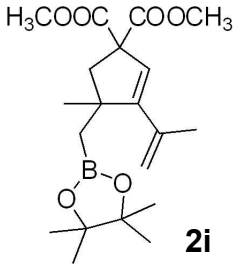
2h



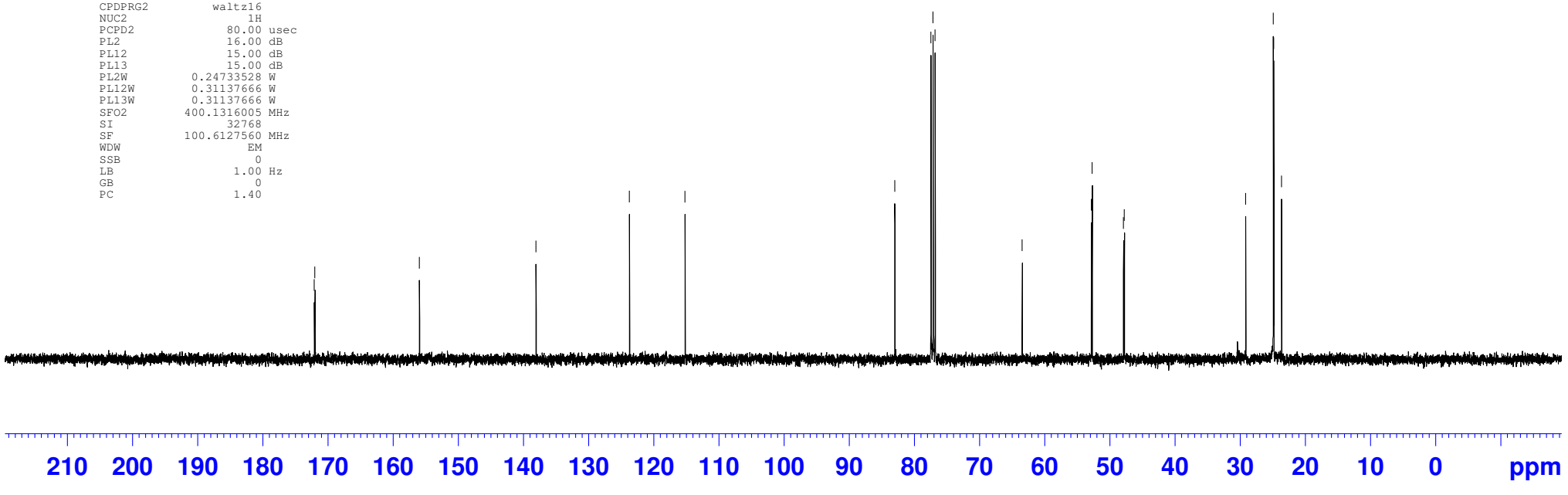

7.260
5.640
5.137
5.022
5.019
5.015
3.716
3.712
2.643
2.609
2.482
2.447
1.895
1.315
1.303
1.264
1.198
1.090
1.052
NAME TJ-20140728-E-969-2Me-allyl-B-prod
EXPNO 10
PROCNO 1
Date_ 20140728
Time 14.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.90 usec
PL1 -4.00 dB
PL1W 24.73352814 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H3COOC COOCH3
B
O
O
2i
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
ppm
1.00
1.02
1.02
6.20
1.03
1.03
3.09
3.44
1.19
12.28
1.22

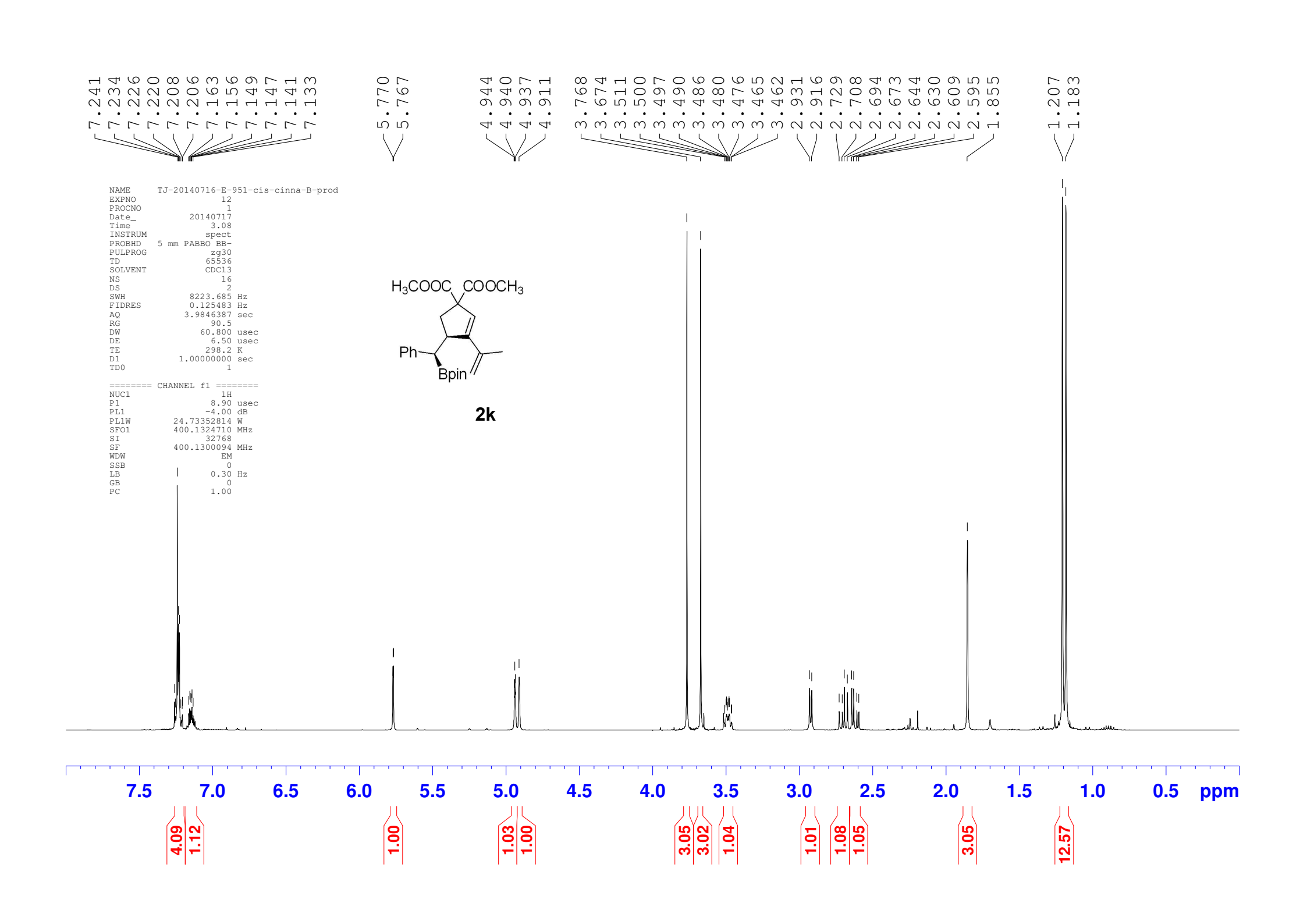
172.126
172.035
155.986
138.093
123.757
115.226
83.020
77.478
77.160
76.842
63.480
52.854
52.735
47.931
47.802
29.174
24.932
24.866
23.666

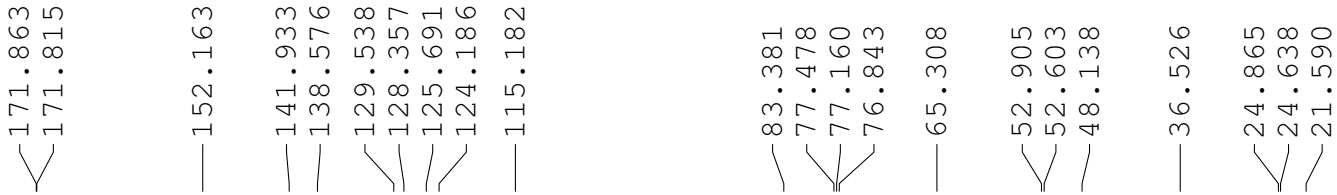
```
NAME      TJ-20140728-E-969-2Me-allyl-B-prod
EXPNO                 11
PROCNO                 1
Date_           20140728
Time                14.56
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     64
DS                      4
SWH             24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                   2050
DW                 20.800 usec
DE                 51.93 usec
TE                 298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   6.43 usec
PL1                 -3.00 dB
PL1W         69.66502380 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2                80.00 usec
PL2                  16.00 dB
PL12                 15.00 dB
PL13                 15.00 dB
PL2W          0.24733528 W
PL12W         0.31137666 W
PL13W         0.31137666 W
SFO2         400.1316005 MHz
SI                  32768
SF           100.6127560 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



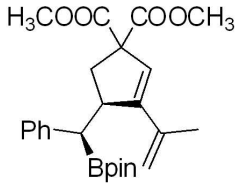
2i



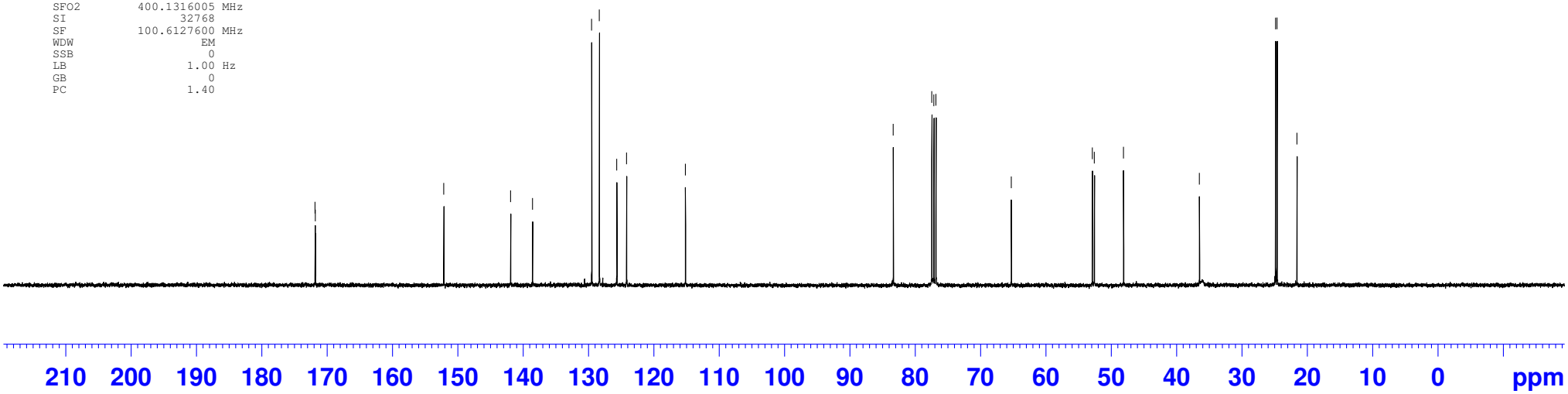

7.241
7.234
7.226
7.220
7.208
7.206
7.163
7.156
7.149
7.147
7.141
7.133
5.770
5.767
4.944
4.940
4.937
4.911
3.768
3.674
3.511
3.500
3.497
3.490
3.486
3.480
3.476
3.465
3.462
2.931
2.916
2.729
2.708
2.694
2.673
2.644
2.630
2.609
2.595
1.855
1.207
1.183
NAME TJ-20140716-E-951-cis-cinna-B-prod
EXPNO 12
PROCNO 1
Date_ 20140717
Time 3.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.90 usec
PL1 -4.00 dB
PL1W 24.73352814 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H3COOC
COOCH3
Ph
Bpin
2k
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
ppm
4.09
1.12
1.00
1.03
1.00
3.05
3.02
1.04
1.01
1.08
1.05
3.05
12.57

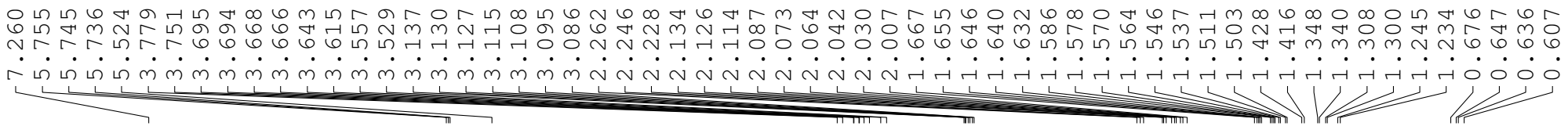



```
NAME      TJ-20140716-E-951-cis-cinna-B-prod
EXPNO                 11
PROCNO                 1
Date_           20140716
Time                19.57
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT             CDCl3
NS                    172
DS                      4
SWH             24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                   2050
DW                 20.800 usec
DE                 51.93 usec
TE                 298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   6.43 usec
PL1                 -3.00 dB
PL1W         69.66502380 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               80.00 usec
PL2                 16.00 dB
PL12                15.00 dB
PL13                15.00 dB
PL2W          0.24733528 W
PL12W         0.31137666 W
PL13W         0.31137666 W
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127600 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

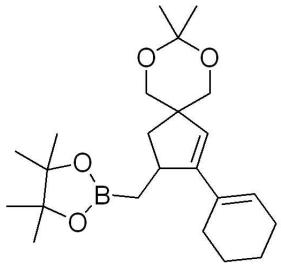


2k

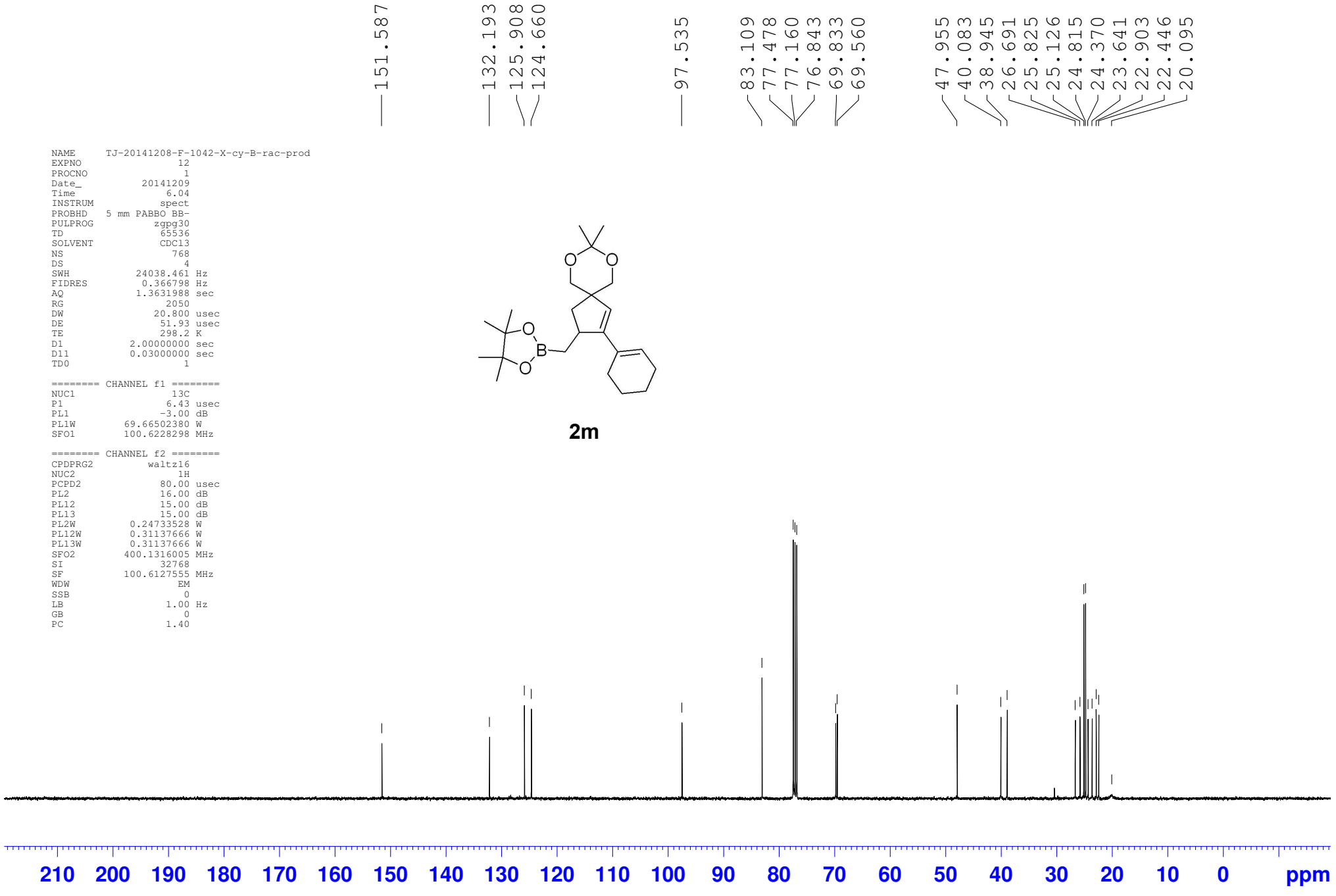





```
NAME      TJ-20141208-F-1042-X-cy-B-rac-prod
EXPNO                 10
PROCNO                 1
Date_           20141209
Time                5.14
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   144
DW                60.800 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.90 usec
PL1               -4.00 dB
PL1W        24.73352814 W
SFO1        400.1324710 MHz
SI                 32768
SF          400.1300094 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



2m




```
NAME      TJ-20141208-F-1042-X-cy-B-rac-prod
EXPNO                 12
PROCNO                 1
Date_           20141209
Time                6.04
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   768
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                  2050
DW                20.800 usec
DE                51.93 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  6.43 usec
PL1                -3.00 dB
PL1W        69.66502380 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2               80.00 usec
PL2                 16.00 dB
PL12                15.00 dB
PL13                15.00 dB
PL2W         0.24733528 W
PL12W        0.31137666 W
PL13W        0.31137666 W
SFO2       400.1316005 MHz
SI                 32768
SF          100.6127555 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```