# Supporting Information

# for

# The total synthesis of D-chalcose and its C-3 epimer

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## Experimental details, characterization data of all products, and copies of MS and NMR spectra

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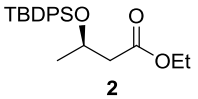
**Experimental details**

**General methods:** Commercial reagents were used without further purification unless specialized. Solvents were dried and redistilled prior to use in the usual way. Boiling range of petroleum ether was 60–90 °C. Thin-layer chromatography was performed on GF254 silica gel plates to monitor the reaction and the plates were examined under UV light or detected with a solution of aniline: diphenylamine: phosphoric acid = 1 mL: 1 g: 5 mL in acetone (50 mL). The purification of the products was performed using column chromatography (60 Å, 200–300 mesh, Qingdao Ocean Chemicals) with the designated solvents. Preparative column chromatography was performed with silica gel H. Melting points were detected on a hot-stage microscope (X-4, Beijing Taike Ltd). Optical rotations were measured at the sodium D-line at room temperature with a Jasco P-2000 polarimeter. $^{1}H$ and $^{13}C$ NMR spectra were taken in $CDCl_3$ solution on Bruker ARX-300, Bruker AV-400, and Bruker AV-600 spectrometers with TMS as the internal reference. Chemical shifts were reported in ppm downfield from tetramethylsilane and proton–proton coupling constants (J) in Hz. ESI-MS and EI-MS were obtained on an Agilent 1100 and an Agilent 6890N mass spectrometer, respectively. High-resolution mass spectra were detected on Bruker Daltonics micrOTOF-Q mass spectrometer.

**Ethyl (*R*)-3-(*tert*-butyldiphenylsilyloxy)butyrate (2) [1]**

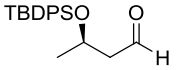



**2**

To a solution of ethyl (*R*)-3-hydroxybutyrate (**1**, 1.0 g, 7.6 mmol), imidazole (1.0 g, 14.7 mmol) and DMAP (0.09 g, 0.76 mmol) in 50 mL of dry $CH_2Cl_2$ was added dropwise *tert*-butyldiphenylsilyl chloride (TBDPSCl, 2.5 g, 9.1 mmol) at 0 °C. After being stirred overnight at room temperature, the mixture was diluted with $CH_2Cl_2$, washed with brine, and dried over anhydrous $Na_2SO_4$. The solvents were then removed under reduced pressure, and the residue was purified by flash silica gel column chromatography (hexanes/ethyl acetate, 8:1) to provide **2** as a colorless oil (2.78 g, 99%). $[\alpha]^{25}_{D} = -7.0°$ (*c* 1.0, $CHCl_3$); $^{1}H$-NMR (300 MHz, $CDCl_3$) $\delta$ 7.70 (m,

4 H), 7.41 (m, 6 H), 4.33 (m, 1 H), 4.07 (m, 2 H), 2.57 (dd, *J* = 14.7, 6.9 Hz, 1 H), 2.41 (dd, *J* = 14.7, 5.7 Hz, 1 H), 1.22 (t, *J* = 7.2 Hz, 3 H), 1.13 (d, *J* = 6.1 Hz, 3 H), 1.05 (s, 9 H); $^{13}$C-NMR (300 MHz, $CDCl_3$) $\delta$ 171.7, 136.1, 134.6, 134.2, 129.9, 129.8, 127.7, 127.6, 67.2, 60.6, 44.9, 27.1, 23.9, 19.5, 14.4; MS (ESI, *m*/*z*): 393.2 [M + $Na^+$]

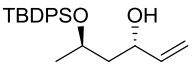
**(3*R*)-3-(*tert*-Butyldiphenylsilyloxy)butyraldehyde (3) [2]**




**3**

To a solution of **2** (2.0 g, 5.4 mmol) in 50 mL of anhydrous $CH_2Cl_2$ was added dropwise DIBAL-H (6.5 mL, 1.0 M solution in toluene) at −78 °C. After 2 h, the mixture was raised to −30 °C, methanol (5 mL) was then added, the cooling bath removed, and the mixture warmed to 0 °C. A saturated solution of potassium sodium tartrate was added and the mixture was extracted with $Et_2O$ (3 x 15 mL). The combined organic layers were washed with brine, dried (anhydrous $Na_2SO_4$), filtered and concentrated. The residue was purified by flash silica gel column chromatography (hexanes/ethyl acetate, 5:1) to afford aldehyde **3** as a colorless oil (1.64 g, 93%). $[\alpha]^{25}_D$ = +8.3° (*c* 1.0, $CHCl_3$); $^1$H-NMR (300 MHz, $CDCl_3$) $\delta$ 9.75(s, 1 H), 7.67 (m, 4 H), 7.40 (m, 6 H), 4.35 (m, 1 H), 2.51 (m, 2 H), 1.19 (d, *J* = 6.2 Hz, 3 H), 1.06 (s, 9 H); $^{13}$C-NMR (300 MHz, $CDCl_3$) $\delta$ 201.9, 135.8, 134.0, 133.5, 129.8, 129.7, 127.7, 127.5, 65.6, 52.7, 26.9, 23.8, 19.1; MS (EI, *m*/*z*): 325.2[M − $H^+$].

**(3*S*,5*R*)-5-(*tert*-Butyldiphenylsilyloxy)hex-1-en-3-ol (4) [3]**

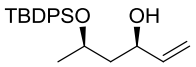



**4**

To freshly dried CuI (11.6 mg, 0.06 mmol) under $N_2$ was added 10 mL dry diethyl ether and 1.22 mL of 1.0 M vinylmagnesium bromide in THF (1.22 mmol). The mixture was stirred at −78 °C for 30 min. At this point, a solution of **3** (200 mg, 0.61 mmol) in dry diethyl ether (10 mL) was introduced dropwise, then the mixture was

stirred at −78 °C for 3 h and allowed to warm to 0 °C quenching with water (1.2 mL) and extraction with diethyl ether (3 x 15 mL). The combined organic layers were washed with brine, and dried over anhydrous $Na_2SO_4$. The solvents were then removed under reduced pressure, and the residue was purified by flash silica gel column chromatography (hexanes/ethyl acetate, 7:1) to provide **4** as a colorless oil (184 mg, 85%). $[\alpha]^{25}_D$ = +4.8°(*c* 1.4, $CHCl_3$); $^1$H-NMR (300 MHz, $CDCl_3$) $\delta$ 7.70 (m, 4 H), 7.41 (m, 6 H), 5.82 (dd, *J* = 17.2, 10.5 Hz, 1 H), 5.24 (d, *J* = 17.2 Hz, 1 H), 5.06 (d, *J* = 10.5 Hz, 1 H), 4.46 (m, 1 H), 4.17 (m, 1 H), 3.06 (s, 1 H), 1.66 (m, 2 H), 1.11 (d, *J* = 6.2 Hz, 3 H) , 1.06 (s, 9 H); $^{13}$C-NMR (300 MHz, $CDCl_3$) $\delta$ 141.3, 136.2, 134.2, 130.1, 130.0, 128.0, 127.9, 114.3, 69.9, 68.7, 45.0, 27.3, 23.0, 19.4; MS (ESI, *m*/*z*): 377.1 [M + $Na^+$].

**(3*R*,5*R*)-5-(*tert*-Butyldiphenylsilyloxy)hex-1-en-3-ol (4') [3]**




**4'**

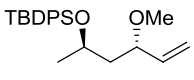
The same reaction condition was followed to obtain the required product **4'** in 85% as a colourless oil. $[\alpha]^{25}_D$ = -7.0° (*c* 1.3, $CHCl_3$); $^1$H-NMR (300 MHz, $CDCl_3$) $\delta$ 7.70 (m, 4 H), 7.41 (m, 6 H), 5.80 (dd, *J* = 17.2, 10.5 Hz, 1 H), 5.20 (d, *J* = 17.2 Hz, 1 H), 5.05 (d, *J* = 10.5 Hz, 1 H), 4.34 (m, 1 H), 4.12 (m, 1 H), 2.85 (s, 1 H), 1.68 (m, 2 H), 1.06 (s, 9 H), 1.01 (d, *J* = 6.2 Hz, 3 H); $^{13}$C-NMR (300 MHz, $CDCl_3$) $\delta$ 141.3, 136.2, 134.6, 133.8, 130.1, 129.9, 128.0, 127.8, 114.5, 71.9, 70.2, 46.5, 27.2, 24.3, 19.5; MS (ESI, *m*/z): 377.1 [M + $Na^+$].

Diisopropyl azodicarboxylate (DIAD, 430 mg, 2.13 mmol) was added dropwise to a solution of compound **4'** (200 mg, 0.56 mmol), triphenylphosphine (440 mg, 1.68 mmol) and benzoic acid (239 mg, 1.96 mmol) in dry THF (2 mL) at room temperature. The resulting mixture was stirred for 8 h. Then 1 N HCl (0.1 mL) was added and the mixture was extracted with $Et_2O$ (3 x 20 mL). The combined organic layers were washed with brine, dried (anhydrous $Na_2SO_4$), filtered and concentrated. The residue was purified by flash silica gel column chromatography (hexanes/ethyl acetate, 15:1) to afford ester as a colorless oil, which was dissolved in THF (0.5 mL)

and cooled to 0 °C. To it was added 10% aqueous NaOH (1 mL), and the mixture was stirred at room temperature for 16 h, diluted with $CHCl_3$ (3 x 20 mL), washed with brine, and dried (anhydrous $Na_2SO_4$). Concentration in vacuo and purification of the residue by flash silica gel column chromatography (hexanes/ethyl acetate, 7:1) to afford **4** as a colourless oil (180 mg, 90%).

Diethyl diazenedicarboxylate (DEAD, 371 mg, 2.13 mmol) was added dropwise to a solution of compound **4** (200 mg, 0.56 mmol), triphenylphosphine (440 mg, 1.68 mmol) and benzoic acid (239 mg, 1.96 mmol) in dry THF (2 mL) at room temperature. The resulting mixture was stirred for 1 h and then the mixture was heated at 40 °C for 10 h. Then 1 N HCl (0.1 mL) was added and the mixture was extracted with $Et_2O$ (3 x 20 mL). The combined organic layers were washed with brine, dried (anhydrous $Na_2SO_4$), filtered and concentrated. The residue was purified by flash silica gel column chromatography (hexanes/ethyl acetate, 15:1) to afford the ester as a colorless oil, which was dissolved in THF (0.5 mL) and cooled to 0 °C. To it was added 10% aqueous NaOH (0.6 mL), and the mixture was stirred at room temperature for 20 h, diluted with $CHCl_3$ (3 x 20 mL), washed with brine, and dried (anhydrous $Na_2SO_4$). Concentration in vacuo and purification of the residue by flash silica gel column chromatography (hexanes/ethyl acetate, 7:1) to afford **4'** as a colorless oil (182 mg, 91%).

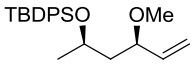
**_tert_-butyl((2_R_,4_S_)-4-methoxyhex-5-en-2-yloxy)diphenylsilane (5) and _tert_-butyl((2_R_,4_R_)-4-methoxyhex-5-en-2-yloxy)diphenylsilane (5')**




**5**

To a solution of **4** (200 mg, 0.56 mmol) in THF (2 mL) cooled to 0 °C under $N_2$ was add *t*-BuOK (1.13 mL, 1.13 mmol). The resulting mixture was stirred for 30 min, treated with freshly distilled methyl iodide (0.06 mL, 0.95 mmol) at 0 °C, allowed to warm slowly to room temperature overnight, quenched with water (5 mL), and extracted with EtOAc (3 x 10 mL). The combined organic layers were washed with brine, and dried over anhydrous $Na_2SO_4$. The solvents were then removed under

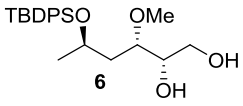
reduced pressure, and the residue was purified by flash silica gel column chromatography (hexanes/ethyl acetate, 15:1) to provide **5** as a colorless oil (198 mg, 96%). $[\alpha]^{25}_{D}$ = -4.9° (*c* 0.69, $CHCl_3$); $^1$H-NMR (300 MHz, $CDCl_3$) $\delta$ 7.69 (m, 4 H), 7.38 (m, 6 H), 5.51(m, 1 H), 5.12 (m, 1 H), 5.07 (m, 1 H), 4.08 (m, 1 H), 3.67 (m, 1 H), 3.08 (s, 3 H), 1.63 (m, 2 H), 1.06 (s, 9 H), 1.05 (d, *J* = 6.1 Hz, 3 H); $^{13}$C-NMR (300 MHz, $CDCl_3$) $\delta$ 138.8, 135.9, 134.9, 134.4, 129.5, 129.4, 127.5, 127.4, 116.7, 79.6, 66.6, 55.8, 44.6, 27.1, 24.2, 19.3; HRMS m/z: calcd for $[C_{23}H_{33}O_2Si]^+$ ($[M + H]^+$) 369.2244, found: 369.2241.




**5'**

The same reaction conditions was followed to obtain the required product **5'** in 96% as a colorless oil. $[\alpha]^{25}_{D}$ = +10.0° (*c* 0.75, $CHCl_3$); $^1$H-NMR (300 MHz, $CDCl_3$) $\delta$ 7.68 (m, 4 H), 7.37 (m, 6 H), 5.10 (m, 1 H), 5.03 (m, 1 H), 4.68 (m, 1 H), 3.97 (m, 1 H), 3.19 (s, 3 H), 1.93 (m,1 H), 1.52 (m,1 H), 1.08 (d, *J* = 6.1 Hz, 3 H), 1.05 (s, 9 H). $^{13}$C-NMR (300 MHz, $CDCl_3$) $\delta$ 138.6, 135.9, 135.8, 134.8, 134.4, 129.5, 129.4, 127.5, 127.4, 117.1, 80.1, 67.0, 55.9, 45.1, 27.0, 23.7, 19.2; HRMS m/z: calcd for $[C_{23}H_{32}NaO_2Si]^+$ ($[M + Na]^+$) 391.2064, found: 391.2060.

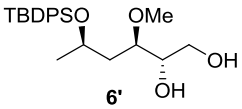
**(2*S*,3*S*,5*R*)-5-(*tert*-butyldiphenylsilyloxy)-3-methoxyhexane-1,2-diol (6) and (2*S*,3*R*,5*R*)-5-(*tert*-butyldiphenylsilyloxy)-3-methoxyhexane-1,2-diol (6')**




**6**

To a mixture of water (7.5 mL) and *tert*-butyl alcohol (7.5 mL) were added AD-mix-β (2.03 g) and **5**, and the mixture was stirred at 0 °C for 72 h. The reaction was diluted with EtOAc, filtered, and extracted with EtOAc (3 x 10 mL). The combined organic layers were washed with brine, and dried over anhydrous $Na_2SO_4$. The solvents were then removed under reduced pressure, and the residue was purified by flash silica gel column chromatography (hexanes/ethyl acetate, 1:2) to provide **6** as a colorless oil (440 mg, 80%). $[\alpha]^{25}_{D}$ = +1.5° (*c* 1.12, $CHCl_3$); $^1$H-NMR (300 MHz, $CDCl_3$) $\delta$ 7.69 (m, 4 H), 7.39 (m, 6 H), 4.02 (m, 1 H), 3.58 (m, 3 H),

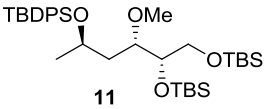
3.40 (m, 1H), 3.22 (s, 3 H), 1.76 (m, 1 H), 1.60 (m, 1 H), 1.10 (d, $J$ = 6.1 Hz, 3 H), 1.05 (s, 9 H); $^{13}$C-NMR (300 MHz, $CDCl_3$) $\delta$ 135.9, 134.7, 134.6, 134.1, 129.7, 129.5, 127.6, 127.4, 80.2, 72.3, 67.3, 63.3, 58.4, 40.6, 27.0, 24.3, 19.2; HRMS m/z : calcd for $[C_{23}H_{35}O_4Si]^+$ ($[M+H]^+$) 403.2299, found: 403.2286.



**6'**

The same reaction conditions was followed to obtain the required product **6'** in 82% as a colorless oil. $[\alpha]^{25}_D$ = -6.9° (*c* 1.17, $CHCl_3$);$^1$H-NMR (300 MHz, $CDCl_3$) $\delta$ 7.68 (m, 4 H), 7.40 (m, 6 H), 4.02 (m, 1 H), 3.62 (m, 2 H), 3.43 (m, 2 H), 3.29 (s, 3 H), 1.81 (m,1 H), 1.61 (m, 1 H), 1.10 (d, $J$ = 6.10 Hz, 3 H), 1.05 (s, 9 H); $^{13}$C-NMR (300 MHz, $CDCl_3$) $\delta$ 135.9, 135.8, 134.3, 133.7, 129.8, 129.6, 127.7, 127.5, 80.2, 72.7, 67.3, 63.4, 57.7, 39.9, 27.0, 23.2, 19.1; HRMS m/z : calcd for $[C_{23}H_{34}NaO_4Si]^+$ ($[M + Na]^+$) 425.2119, found: 425.2121.

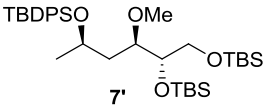
**(5*R*,7*S*,8*S*)-8-(*tert*-butyldimethylsilyloxy)-7-methoxy-2,2,5,11,11,12,12-hepta-methyl-3,3-diphenyl-4,10-dioxa-3,11-disilatridecane (11) and (5*R*,7*R*,8*S*)-8-(*tert*-butyldimethylsilyloxy)-7-methoxy-2,2,5,11,11,12,12-hepta-methyl-3,3-diphenyl-4,10-dioxa-3,11-disilatridecane (7')**



**11**

To a solution of **6** (200 mg, 0.5 mmol) in 2 mL of anhydrous $CH_2Cl_2$ was added 2,6-lutidine (0.287 mL, 1.25 mmol) under $N_2$. The resulting solution was stirred at 0 °C for 10 min. 2,6-Lutidine (0.175 mL , 1.5 mmol) was then added dropwise, the cooling bath removed, and the mixture warmed to room temperature for 24 h. Saturated aqueous ammonium chloride (5 mL) was added and the mixture was extracted with $CH_2Cl_2$. The combined organic layers were washed with brine, dried (anhydrous $Na_2SO_4$), filtered and concentrated. The residue was purified by flash silica gel column chromatography (hexanes/ethyl acetate, 10:1) to afford compound **11** as a colorless oil (284 mg, 90%). $[\alpha]^{25}_D$ = -10.1° (*c* 0.74, $CHCl_3$); $^1$H-NMR (300 MHz, $CDCl_3$) $\delta$ 7.71 (m, 4 H), 7.39 (m, 6 H), 4.11 (m, 1 H), 3.77 (m, 2 H), 3.54 (m,

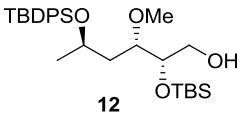
2 H), 3.29 (s, 3 H), 1.68 (m, 2 H), 1.05 (s, 9 H), 1.02 (d, *J* = 6.02 Hz, 3 H), 0.96 (s, 18 H), 0.07 (s, 12 H); $^{13}$C-NMR (300 MHz, $CDCl_3$) $\delta$ 135.9, 135.3, 134.3, 129.5, 129.3, 127.5, 127.3, 78.8, 74.8, 67.1, 64.3, 58.1, 41.2, 27.1, 26.0, 25.9, 19.3, 18.2, -4.2, -4.5, -4.8, -5.4; HRMS m/z : calcd for $[C_{35}H_{62}NaO_4Si_3]^+$ ($[M + Na]^+$) 653.3848, found: 653.3852.



**7'**

The same reaction conditions was followed to obtain the required product **7'** in 91% as a colorless oil. $[\alpha]^{25}_D$ = +15.2° (*c* 0.64, $CHCl_3$); $^1$H-NMR (300 MHz, $CDCl_3$) $\delta$ 7.70 (m, 4 H), 7.39 (m, 6 H), 4.06(m, 1 H), 3.74(m, 2 H), 3.52 (m, 1 H), 3.34 (m, 1 H), 3.26 (s, 3 H), 1.72 (m, 2 H), 1.07 (s, 9 H), 0.91 (s, 18 H), 0.85 (d, *J* = 7.01 Hz, 3 H), 0.07 (s, 12 H); $^{13}$C-NMR (300 MHz, $CDCl_3$) $\delta$ 135.9, 135.0, 134.6, 129.3, 127.4, 127.3, 79.7, 74.6, 67.6, 64.5, 57.4, 39.7, 27.1, 25.9, 22.9, 19.2, 18.3, 18.1, -4.5, -4.7, -5.3, -5.4; HRMS m/z : calcd for $[C_{35}H_{62}NaO_4Si_3]^+$ ($[M + Na]^+$) 653.3848, found: 653.3847.

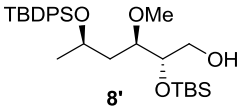
**(2*S*,3*S*,5*R*)-2-(*tert*-butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxy)-3-methoxyhexan-1-ol (12) and (2*S*,3*R*,5*R*)-2-(*tert*-butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxy)-3-methoxyhexan-1-ol (8')**



**12**

Compound **11** was dissolved in 5 mL methanol at 0 °C，added 23 mg CSA and stirred 1.5 h. Saturated aqueous sodium bicarbonate (2 mL) was added and the layers were separated. The aqueous layer was extracted with acetic ester (3 x 5 mL). The combined organic layers were washed with brine, dried (anhydrous $Na_2SO_4$), filtered and concentrated. The residue was purified by flash silica gel column chromatography (hexanes/ethyl acetate, 5:1) to afford compound **12** as a colorless oil (146 mg, 59%). $[\alpha]^{25}_D$ = -15.8° (*c* 0.66, $CHCl_3$); $^1$H-NMR (300 MHz, $CDCl_3$) $\delta$ 7.69 (m, 4 H), 7.38 (m, 6 H), 4.09 (m, 1 H), 3.63 (m, 2 H), 3.54 (m, 2 H), 3.28 (s, 3

H), 1.85 (m, 1 H), 1.44 (m, 1 H), 1.05 (s, 9 H), 1.02 (d, *J* = 6.1 Hz, 3 H), 0.91 (s, 9 H), 0.11 (s, 6 H); $^{13}$C-NMR (300 MHz, $CDCl_3$) $\delta$ 135.9, 135.0, 134.2, 129.6, 129.4, 127.5, 127.3, 80.1, 70.5, 66.7, 58.1, 39.4, 27.1, 25.8, 24.7, 19.3, 18.1,-4.6, -4.7; HRMS m/z : calcd for $[C_{29}H_{48}NaO_4Si_2]^+$ ($[M + Na]^+$) 539.2983, found: 539.2983.

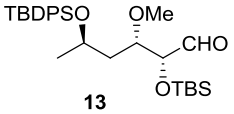



**8'**

The same reaction conditions was followed to obtain the required product **8'** in 60% as a colorless oil. $[\alpha]^{25}_D$ = -2.6° (*c* 0.46, $CHCl_3$); $^1$H-NMR (300 MHz, $CDCl_3$) $\delta$ 7.68 (m, 4 H), 7.39 (m, 6H), 4.06 (m, 1 H), 3.66 (m, 2 H), 3.50 (m, 2 H), 3.23 (s, 3 H), 1.67 (m, 1 H), 1.47 (m, 1 H), 1.06 (d, *J* = 6.2 Hz, 3 H), 1.04 (s, 9 H), 0.90 (s, 9 H), 0.08 (s, 6 H); $^{13}$C-NMR (300 MHz, $CDCl_3$) $\delta$ 135.9, 135.8, 134.9, 134.2, 129.6, 129.4, 127.5, 127.4, 80.2, 74.5, 67.1, 63.9, 58.8, 42.3, 27.1, 25.8, 24.7, 19.3, 18.1, -4.5, -4.6; HRMS m/z : calcd for $[C_{29}H_{48}NaO_4Si_2]^+$ ($[M + Na]^+$) 539.2983, found: 539.2982.

**(2*R*,3*S*,5*R*)-2-(*tert*-butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxy)-3-methoxyhexanal (13) and**

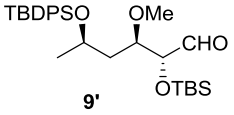
**(2*R*,3*R*,5*R*)-2-(*tert*-butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxy)-3-methoxyhexanal (9')**




**13**

To a solution of **12** (100 mg, 0.19 mmol) in 7 mL of anhydrous $CH_2Cl_2$ was added Dess–Martin periodinane (97 mg, 0.23 mmol) under $N_2$ and the resulting solution was stirred at room temperature for 1 h. Saturated aqueous sodium bicarbonate (5 mL) was added followed by the addition of 7 mL of saturated aqueous $Na_2S_2O_3$. This solution was stirred at room temperature for 5 min, $CH_2Cl_2$ was added and the layers were separated. The water layer was extracted with $CH_2Cl_2$. The combined organic layers were washed with brine, dried (anhydrous $Na_2SO_4$), filtered and concentrated. The residue was purified by flash silica gel column chromatography (hexanes/ethyl acetate, 8:1) to afford compound **13** as a colorless oil (84 mg, 86%).

$[\alpha]^{25}_{D}$ = -2.4° (*c* 0.54, $CHCl_3$); $^1$H-NMR (300 MHz, $CDCl_3$) $\delta$ 9.67 (d, *J* = 1.42 Hz, 1 H), 7.68 (m, 4 H), 7.38 (m, 6 H), 4.10 (m, 2 H), 3.77 (m, 1 H), 3.26 (s, 3 H), 1.83 (m, 1 H), 1.46 (m, 1 H), 1.06 (s, 9 H), 1.03 (d, *J* = 6.24 Hz, 3 H), 0.94 (s, 9 H), 0.09 (s, 6 H); $^{13}$C-NMR (300 MHz, $CDCl_3$) $\delta$ 203.8, 136.3, 136.2， 135.3, 134.5, 130.0, 129.9, 128.0, 127.8, 79.8, 78.5, 67.0, 58.7, 41.4, 35.3, 27.4, 26.2, 19.7, 18.7, -4.3, -4.7; HRMS m/z : calcd for $[C_{29}H_{46}NaO_4Si_2]^+$ ($[M + Na]^+$) 537.2827, found: 537.2823.

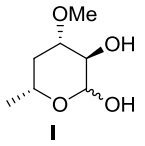



**9'**

The same reaction condition was followed to obtain the required product **9'** in 86% as a colorless oil. $[\alpha]^{25}_{D}$ = -4.1° (*c* 1.07, $CHCl_3$); $^1$H-NMR (300 MHz, $CDCl_3$) $\delta$ 9.61 (d, *J* = 1.46Hz, 1 H), 7.68 (m, 4 H), 7.38 (m, 6 H), 4.06 (m, 2 H), 3.70 (m, 1 H), 3.22 (s, 3 H), 1.67 (m, 1 H), 1.52 (m, 1 H), 1.05 (s, 9 H), 1.03 (d, *J* = 6.24 Hz, 3 H), 0.92 (s, 9 H), 0.07 (s, 6 H); $^{13}$C- MR (300 MHz, $CDCl_3$) $\delta$ 203.8, 135.9, 134.9, 134.1, 129.6, 129.4, 127.6, 127.4, 80.2, 79.3, 66.6, 58.0, 41.6, 29.7, 27.0, 25.7, 24.5, 19.3, 18.2, -4.9, -5.0; HRMS m/z : calcd for $[C_{29}H_{47}O_4Si_2]^+$ ($[M + H]^+$) 515.3007, found: 515.3005.

**4,6-dideoxy-3-*O*-methyl-D-*xylo*-hexose (I) [4] and**

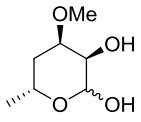
**4,6-dideoxy-3-*O*-methyl-D-*ribo*-hexose (I′)**




**I**

A solution of **13** (100 mg, 0.19 mmol) and TBAF (0.57 mL, 0.57 mmol, 1.0 M in THF) in THF (1 mL) was stirred at room temperature for 1 h. Saturated aqueous ammonium chloride (5 mL) was added and the mixture was extracted with EtOAc. The combined organic layers were washed with brine, dried (anhydrous $Na_2SO_4$), filtered and concentrated. The residue was purified by flash silica gel column chromatography (dichloromethane: acetone, 15: 1) to afford compound **I** as a white solid (26 mg, 84%). Mp 91–93 °C; $[\alpha]^{25}_{D}$ = +130.0º (*c* 0.49, $H_2O$, 5 min); $[\alpha]^{25}_{D}$ = +75.8° (*c* 0.49, $H_2O$, 4 h); $^1$H-NMR (300 MHz, $CDCl_3$) $\delta$ 5.57 (br. s, 0.49 H), 5.24 (d,

*J* = 2.01 Hz, 0.45 H-α), 4.80 (br. s, 0.51 H), 4.50 (d, *J* = 7.04 Hz, 0.55 H-β), 4.13 (m, 1 H), 3.56 (m, 1 H), 3.41 (s, 3 H), 3.27(m, 1 H), 2.07 (m, 1 H), 1.31 (m, 1 H), 1.24 (d, *J* = 6.29 Hz, 1.63 H), 1.18 (d, *J* = 6.29 Hz, 1.37 H); $^{13}$C-NMR (300 MHz, $CDCl_3$) $\delta$ 96.7 (β-D), 92.8 (α-D), 80.0, 72.7, 68.1, 56.9, 37.1, 21.0; HRMS m/z : calcd for $[C_7H_{15}O_4]^+$ ($[M + H]^+$) 163.0965, found: 163.0944.

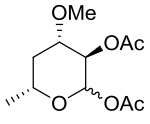



**I'**

The same reaction condition was followed to obtain the required product **I'** in 83% as a white solid. Mp 96–98 °C; $[\alpha]^{25}_D$ = -24.4° (*c* 0.54, $H_2O$, 5 min); $[\alpha]^{25}_D$ = -12.6º (*c* 0.54, $H_2O$, 4 h); $^1$H-NMR (300 MHz, $CDCl_3$) $\delta$ 5.01 (d, *J* = 2.07 Hz, 0.55 H-α), 4.81 (d, *J* = 8.10 Hz, 0.50 H-β), 4.08 (m, 1 H), 3.88 (m, 1 H), 3.43 (s, 3 H), 3.28 (m, 1 H), 2.03 (m, 1 H), 1.74 (m, 1 H), 1.18 (d, *J* = 6.29 Hz, 3 H); $^{13}$C-NMR (300 MHz, $CDCl_3$) $\delta$ 94.8 (β-D), 94.6 (α-D), 78.9, 72.7, 66.3, 57.9, 34.5, 20.6; HRMS m/z : calcd for $[C_7H_{14}NaO_4]^+$ ($[M + Na]^+$) 185.0784, found: 185.0799.

**1,2-di-*O*-acetyl-D-chalcose (14) [5] and**

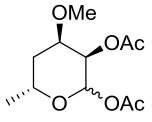
**4,6-dideoxy-3-*O*-methyl-D-*ribo*-hexopyranose diacetate (10')**




**14**

Acetic anhydride (0.40 mL, 1.08 mmol) and pyridine (0.11 mL, 1.34 mmol) were added to compound **I** (100 mg, 0.54 mmol) in dry $CH_2Cl_2$ (1 mL) and maintained at room temperature for 24 h. The mixture was then neutralized with diluted HCl and the mixture was extracted with EtOAc. The combined organic layers were washed with brine, dried (anhydrous $Na_2SO_4$), filtered and concentrated. The residue was purified by flash silica gel column chromatography ($CH_2Cl_2$: $CH_3OH$, 25: 1) to afford compound **14** as a white solid. (116 mg, 80%). Mp 77–79 °C; $[\alpha]^{25}_D$ = +34.2º (*c* 0.88, $CHCl_3$); $^1$H-NMR (300 MHz, $CDCl_3$) $\delta$ 6.24 (d, *J* = 3.54 Hz, 0.38 H-α), 5.56 (d, *J* = 8.25 Hz, 1 H-β), 4.88 (m, 1 H), 3.87 (m, 1 H), 3.65 (m, 1 H), 3.34 (s, 3 H),

2.07 (s, 3 H), 2.06 (s, 3 H), 1.70 (m, 1 H), 1.61 (m, 1 H), 1.28 (d, *J* = 6.32 Hz, 3 H); $^{13}$C-NMR (300 MHz, $CDCl_3$) $\delta$169.8, 169.6, 99.9 (β-D), 92.5 (α-D), 78.2, 77.6, 69.1, 55.2, 37.1, 29.8, 21.0, 20.8; HRMS m/z : calcd for $[C_{11}H_{18}NaO_6]^+$ ($[M + Na]^+$) 269.0996, found: 269.0998.




**10'**

The same reaction condition was followed to obtain the required product **10'** in 80% as a white solid. Mp 73–75 °C; $[\alpha]^{25}_D$ = -47.5° (*c* 1.08, $CHCl_3$); $^1$H-NMR (300 MHz, $CDCl_3$) $\delta$ 6.01 (d, *J* = 8.44 Hz, 1 H-β), 4.72 (dd, *J* = 8.44 Hz, *J* = 3.14 Hz, 1 H), 4.08 (m, 1 H), 3.84 (m, 1 H), 3.37 (s, 3 H ), 2.08 (s, 3 H), 2.07 (s, 3 H), 1.96 (m, 1 H), 1.49 (m, 1 H), 1.21 (d, *J* = 6.32 Hz, 3 H); $^{13}$C-NMR (300 MHz, $CDCl_3$) $\delta$ 170.2, 169.3, 90.5, 75.2, 72.1, 67.6, 58.0, 35.8, 21.0, 20.9, 20.6; HRMS m/z : calcd for $[C_{11}H_{18}NaO_6]^+$ ($[M + Na]^+$) 269.0996, found: 269.0991.

## References

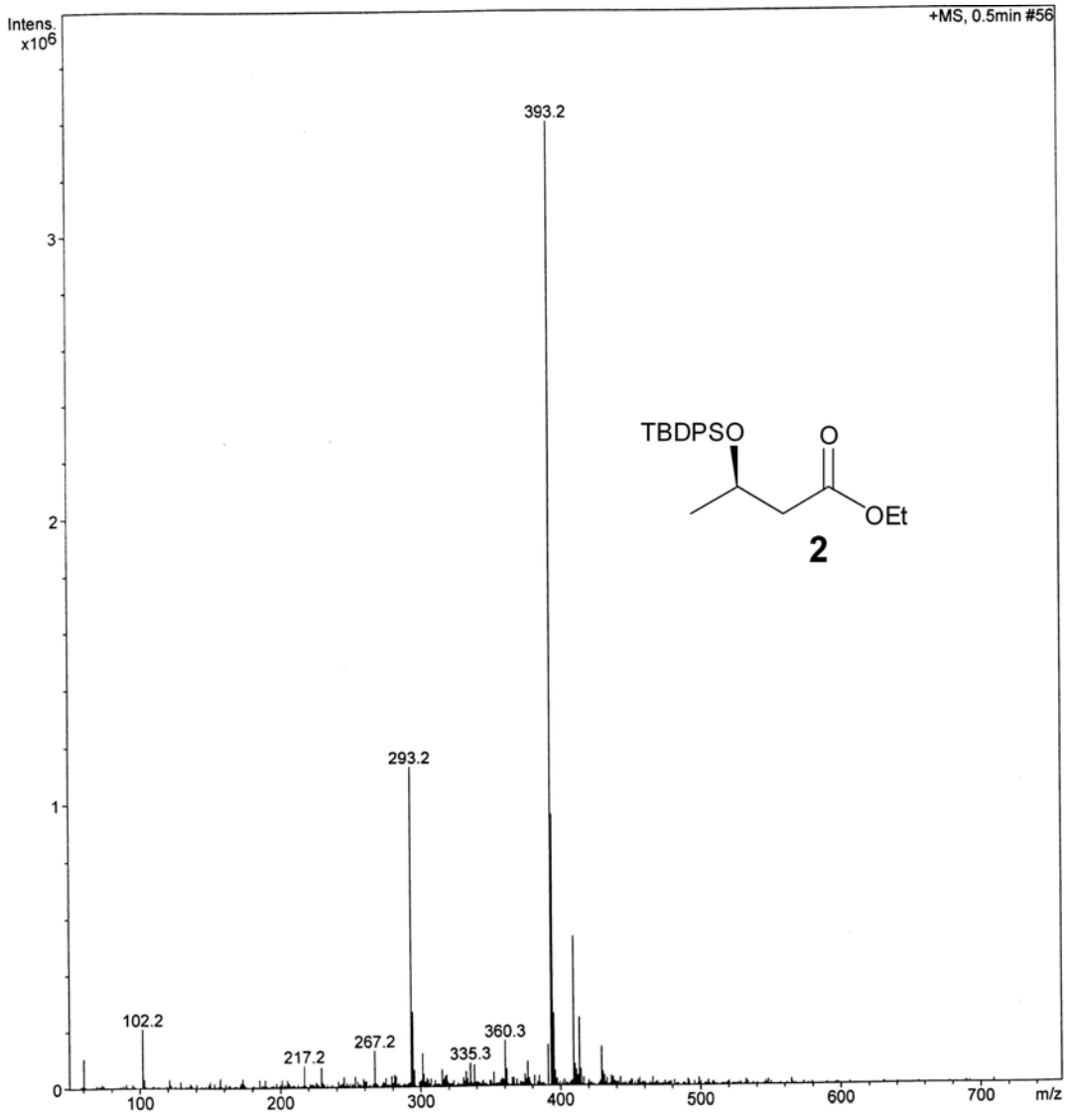

1. Snider, B. B.; Shi, Z. P. *J. Am. Chem. Soc*. **1994**, *116*, 549-557.
2. Claffey, M. M.; Heathcock, C. H. *J. Org. Chem*. **1996**, *61*, 7646-7647.
3. Jana, N.; Mahapatra, T.; Nanda, S. *Tetrahedron: Asymmetry*. **2009**, *20*, 2622-2628.
4. Woo, P. W. K.; Dion, H. W.; Johnson, L. F. *J. Amer. Chem. Soc*. **1962**, *84*, 1066-1067.
5. Redlich, H.; Roy, W. *Carbohydr. Chem*. **1979**, *68*, 275-285.

# Direct Mass Spectrometry Analysis

**Analysis Name:** 10030742.d
**Sample Name:** SJ20100305

**Instrument:** LC-MSD-Trap-SL
**Operator:** SL

**Print Date:** 2010-3-8 21:07:18
**Acq. Date:** 2010-3-8 21:05:37



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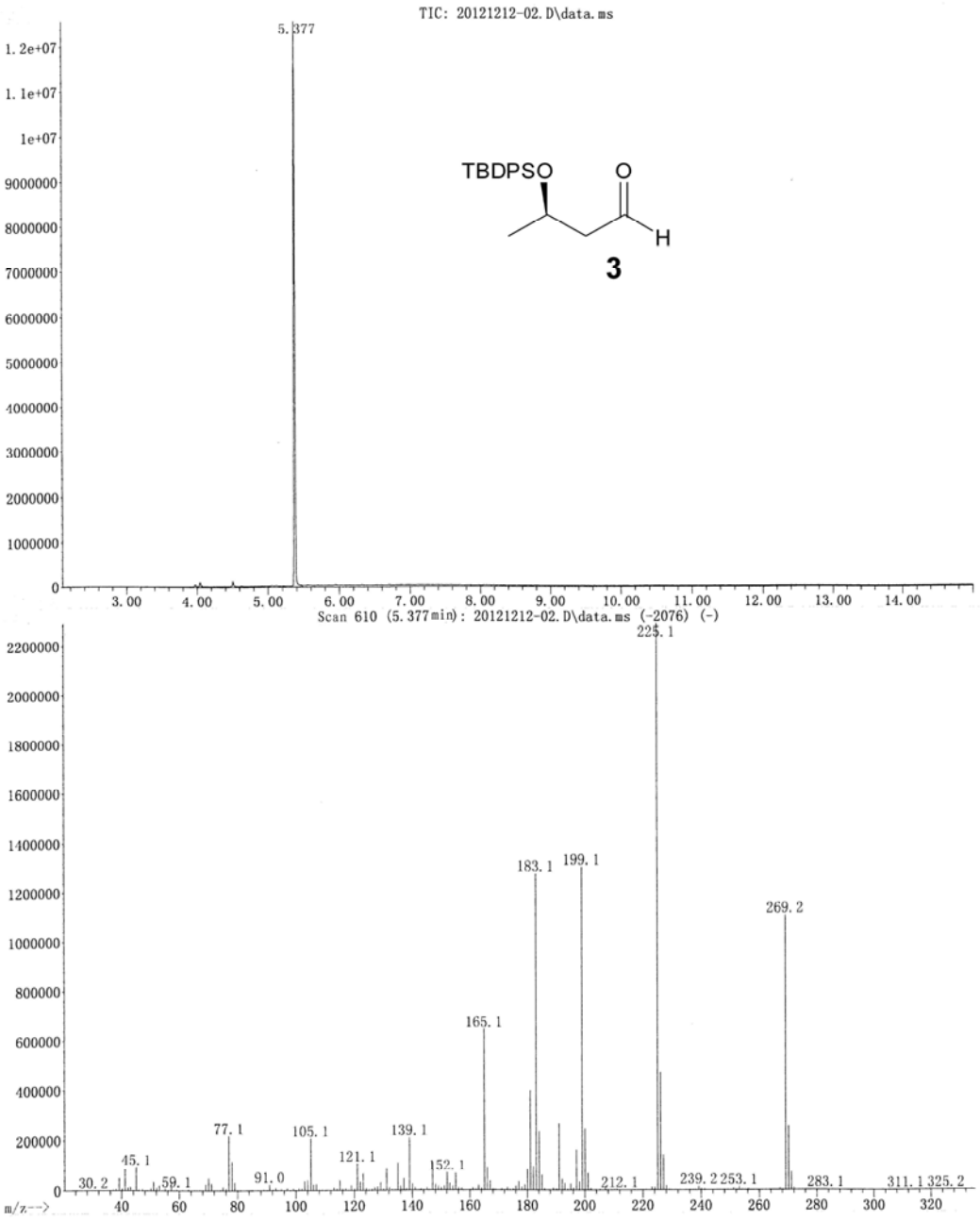
Operator : STJ
Collection :12 Dec 2012 17:58, Collection method METHOD-8.M
Instrument : Instrument 1
Sample Name : SJ20121211
Other information :
Sample number : 12

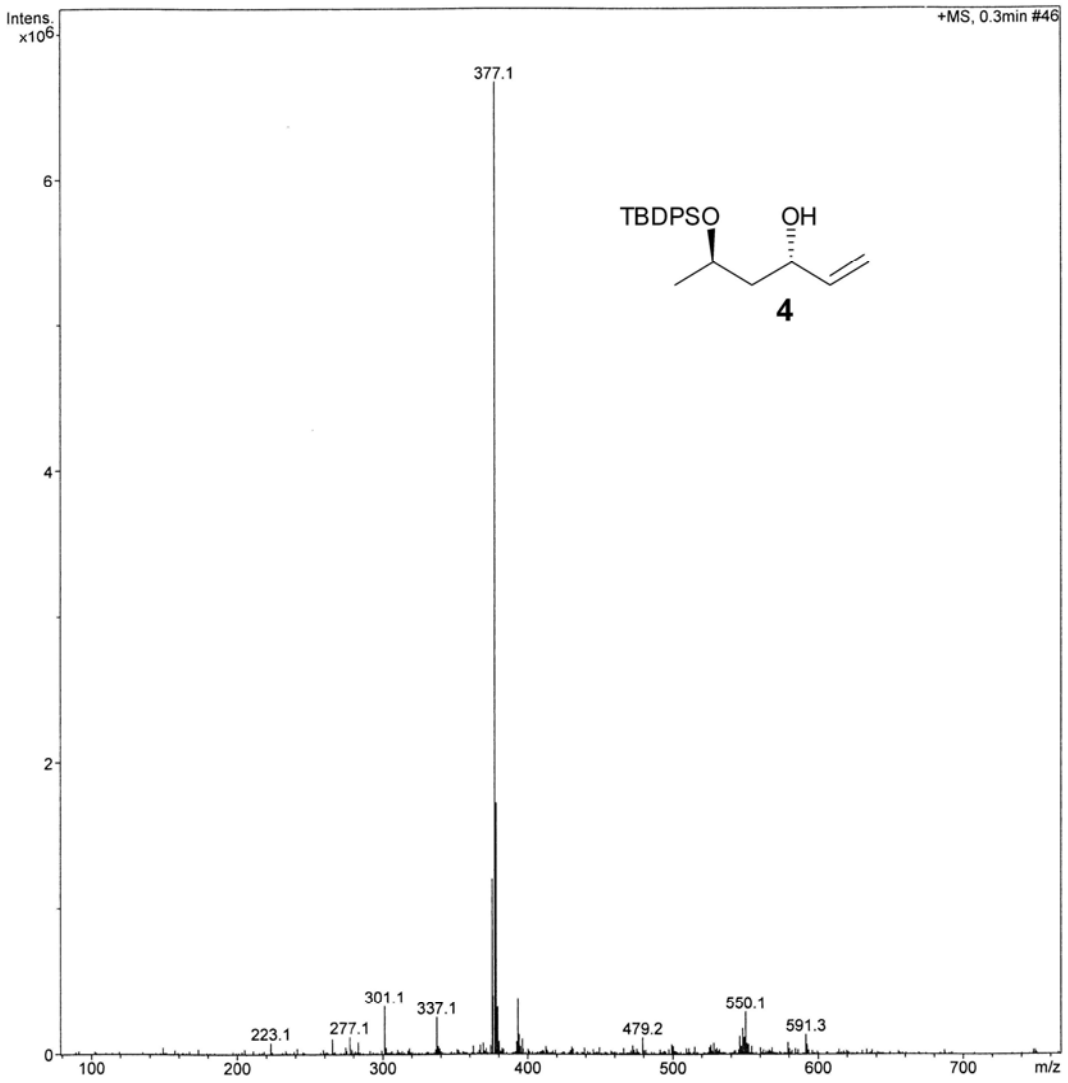


# Direct Mass Spectrometry Analysis

**Analysis Name:** 12022517.d
**Sample Name:** SJ201112141

**Instrument:** LC-MSD-Trap-SL
**Operator:** admin

**Print Date:** 2/25/2012 8:24:31 PM
**Acq. Date:** 2/25/2012 8:23:00 PM



# Direct Mass Spectrometry Analysis

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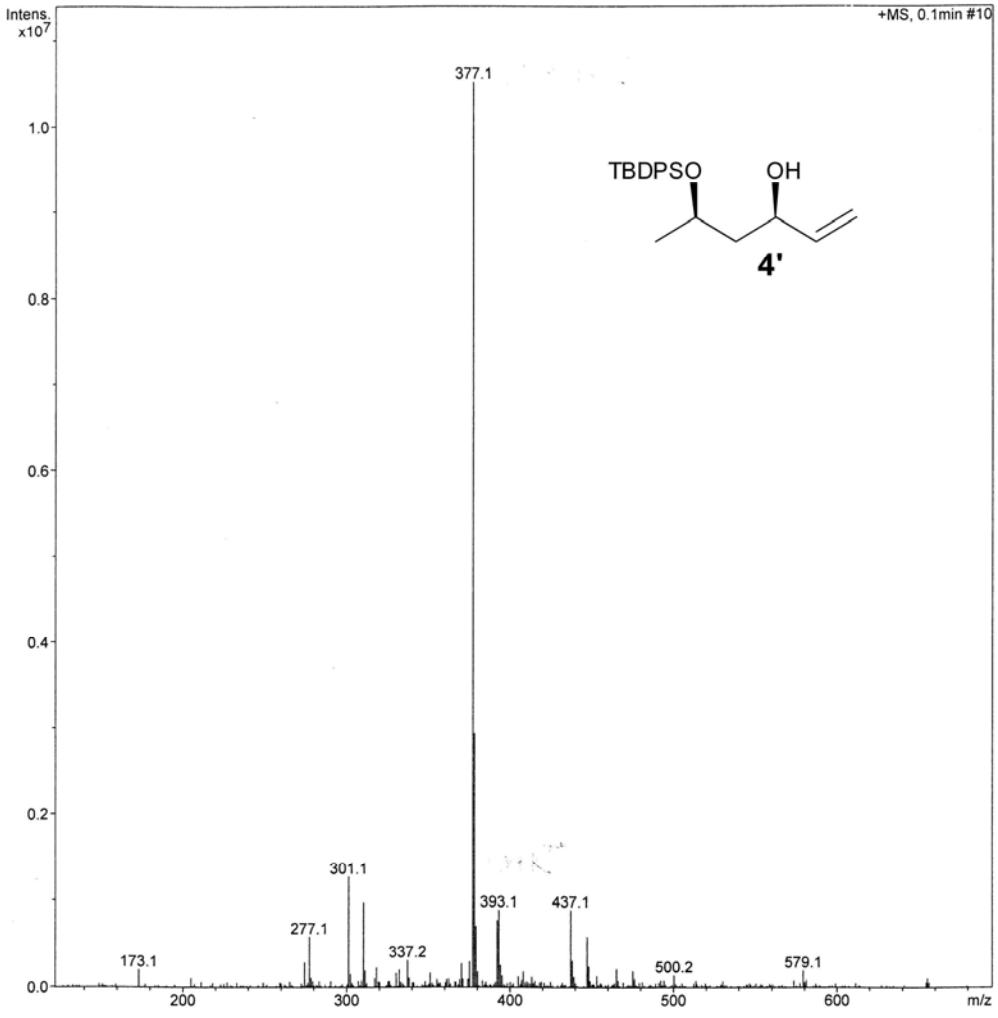
**Sample Name:** SJ201112142

**Instrument:** LC-MSD-Trap-SL
**Operator:** admin

**Print Date:** 2/24/2012 9:46:50 PM
**Acq. Date:** 2/24/2012 9:46:03 PM



# Mass Spectrum Molecular Formula Report

**Analysis Info**

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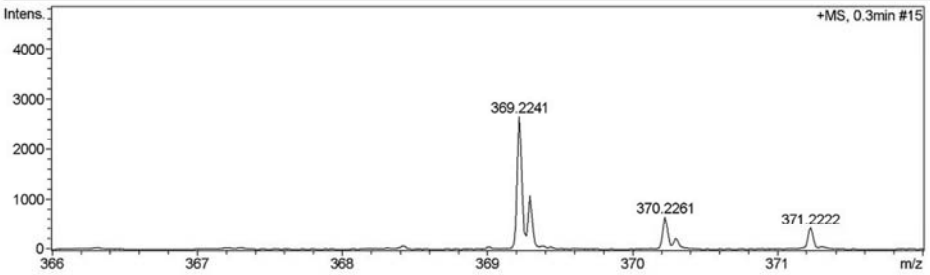
| Method | yujia-shuijie.m | Operator | Bruker Customer |
| Sample Name | SJ20120228-- | Instrument / Ser# | micrOTOF-Q 125 |
| Comment | | | |

**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.3 Bar |
| Focus | Not active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 1.2 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 450.0 Vpp | Set Divert Valve | Source |

**Generate Molecular Formula Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Formula, min. | C23H32O2SiH | | | | |
| Formula, max. | | | | | |
| Measured m/z | 369.224 | Tolerance | 5 ppm | Charge | 1 |
| Check Valence | no | Minimum | 0 | Maximum | 0 |
| Nirogen Rule | yes | Electron Configuration | both | | |
| Filter H/C Ratio | yes | Minimum | 0 | Maximum | 3 |
| Estimate Carbon | yes | | | | |




| Sum Formula | Sigma | m/z | Err [ppm] | Mean Err [ppm] | Err [mDa] | rdb | N Rule | e⁻ |
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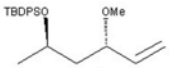
| C 23 H 33 O 2 Si 1 | 0.050 | 369.2244 | 0.86 | 2.38 | 0.32 | 8.50 | ok | even |




**5**

# Mass Spectrum Molecular Formula Report

**Analysis Info**

| | | | |
|---|---|---|---|
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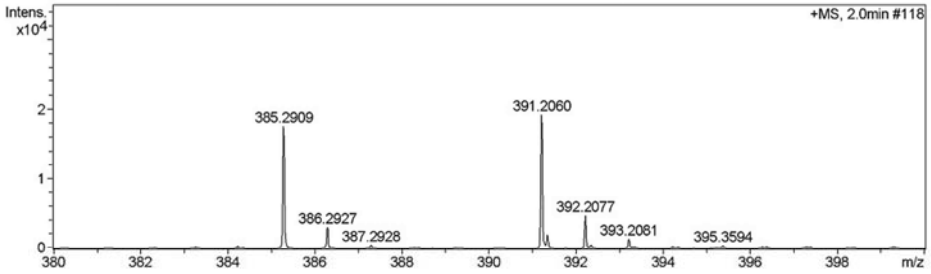
| Method | yujia-shuijie.m | Operator | Bruker Customer |
| Sample Name | SJ20120225 | Instrument / Ser# | micrOTOF-Q 125 |
| Comment | | | |

**Acquisition Parameter**

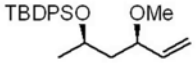
| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.3 Bar |
| Focus | Not active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 1.2 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 700.0 Vpp | Set Divert Valve | Source |

**Generate Molecular Formula Parameter**

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Formula, min. | C23H32O2SiNa | | | | | | |
| Formula, max. | | | | | | | |
| Measured m/z | 391.206 | Tolerance | 5 | ppm | Charge | 1 | |
| Check Valence | no | Minimum | 0 | | Maximum | 0 | |
| Nirogen Rule | yes | Electron Configuration | both | | | | |
| Filter H/C Ratio | yes | Minimum | 0 | | Maximum | 3 | |
| Estimate Carbon | yes | | | | | | |




| Sum Formula | Sigma | m/z | Err [ppm] | Mean Err [ppm] | Err [mDa] | rdb | N Rule | e⁻ |
|---|---|---|---|---|---|---|---|---|
| C 23 H 32 Na 1 O 2 Si 1 | 0.030 | 391.2064 | 0.97 | 1.13 | 0.38 | 8.50 | ok | even |




**5'**

# Mass Spectrum Molecular Formula Report

**Analysis Info**

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|---|---|---|---|
| Analysis Name | D:\Data\20130622\SJ20120510.d | Acquisition Date | 6/22/2013 4:32:31 PM |
| Method | yujia-shuijie.m | Operator | Bruker Customer |
| Sample Name | SJ20120510 | Instrument / Ser# | micrOTOF-Q 125 |
| Comment | | | |

**Acquisition Parameter**

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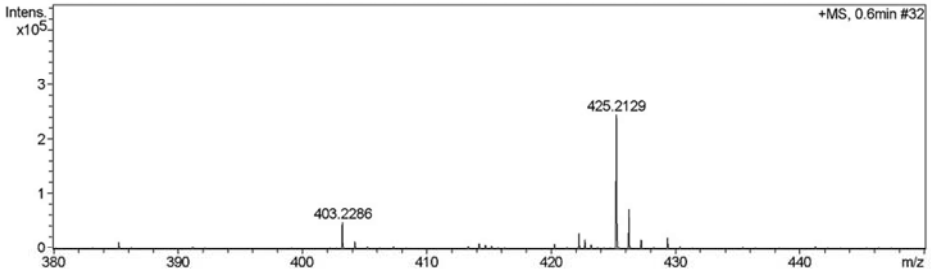
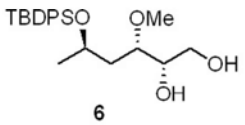
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.3 Bar |
| Focus | Not active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 1.2 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 450.0 Vpp | Set Divert Valve | Source |

**Generate Molecular Formula Parameter**

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Formula, min. | C23H34O4SiH | | | | | | |
| Formula, max. | | | | | | | |
| Measured m/z | 403.229 | Tolerance | 5 | ppm | Charge | 1 | |
| Check Valence | no | Minimum | 0 | | Maximum | 0 | |
| Nirogen Rule | yes | Electron Configuration | both | | | | |
| Filter H/C Ratio | yes | Minimum | 0 | | Maximum | 3 | |
| Estimate Carbon | yes | | | | | | |




| Sum Formula | Sigma | m/z | Err [ppm] | Mean Err [ppm] | Err [mDa] | rdb | N Rule | e⁻ |
|---|---|---|---|---|---|---|---|---|
| C 23 H 35 O 4 Si 1 | 0.033 | 403.2299 | 3.29 | 4.11 | 1.33 | 7.50 | ok | even |




**6**

# Mass Spectrum Molecular Formula Report

**Analysis Info**

| | | | |
|---|---|---|---|
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| Sample Name | SJ20120511 | Instrument / Ser# | micrOTOF-Q 125 |
| Comment | | | |

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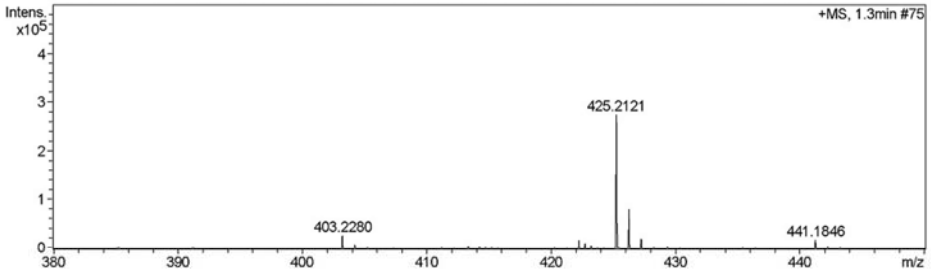
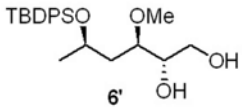
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 1.2 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 450.0 Vpp | Set Divert Valve | Source |

**Generate Molecular Formula Parameter**

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Formula, min. | C23H34O4SiNa | | | | | | |
| Formula, max. | | | | | | | |
| Measured m/z | 425.212 | Tolerance | 5 | ppm | Charge | 1 | |
| Check Valence | no | Minimum | 0 | | Maximum | 0 | |
| Nirogen Rule | yes | Electron Configuration | both | | | | |
| Filter H/C Ratio | yes | Minimum | 0 | | Maximum | 3 | |
| Estimate Carbon | yes | | | | | | |




| Sum Formula | Sigma | m/z | Err [ppm] | Mean Err [ppm] | Err [mDa] | rdb | N Rule | e⁻ |
|---|---|---|---|---|---|---|---|---|
| C 23 H 34 Na 1 O 4 Si 1 | 0.011 | 425.2119 | -0.55 | 0.32 | -0.24 | 7.50 | ok | even |




**6'**

# Mass Spectrum Molecular Formula Report

**Analysis Info**

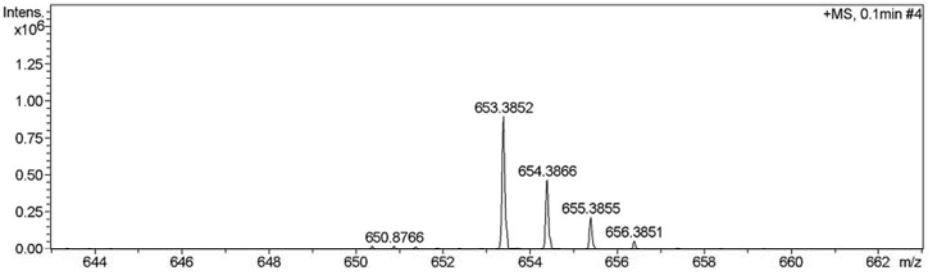
| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\20130626\SJ20121123-1.d | Acquisition Date | 6/26/2013 10:34:30 AM |
| Method | MA250-550POS.m | Operator | Bruker Customer |
| Sample Name | SJ20121123-1 | Instrument / Ser# | micrOTOF-Q 125 |
| Comment | | | |

**Acquisition Parameter**

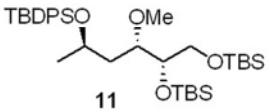
| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.3 Bar |
| Focus | Not active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 4.0 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 900.0 Vpp | Set Divert Valve | Source |

**Generate Molecular Formula Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Formula, min. | C35H62O4Si3Na | | | | |
| Formula, max. | | | | | |
| Measured m/z | 653.385 | Tolerance | 5 ppm | Charge | 1 |
| Check Valence | no | Minimum | 0 | Maximum | 0 |
| Nirogen Rule | no | Electron Configuration | both | | |
| Filter H/C Ratio | no | Minimum | 0 | Maximum | 3 |
| Estimate Carbon | yes | | | | |




| Sum Formula | Sigma | m/z | Err [ppm] | Mean Err [ppm] | Err [mDa] | rdb | N Rule | e⁻ |
|---|---|---|---|---|---|---|---|---|
| C 35 H 62 Na 1 O 4 Si 3 | 0.010 | 653.3848 | -0.66 | 0.24 | -0.43 | 7.50 | ok | even |




**11**

# Mass Spectrum Molecular Formula Report

**Analysis Info**

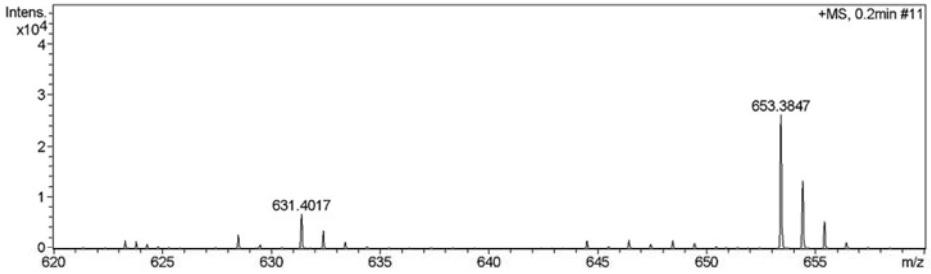
| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\20130622\SJ201205218.d | Acquisition Date | 6/22/2013 4:43:04 PM |
| Method | yujia-shuijie.m | Operator | Bruker Customer |
| Sample Name | SJ201205218 | Instrument / Ser# | micrOTOF-Q 125 |
| Comment | | | |

**Acquisition Parameter**

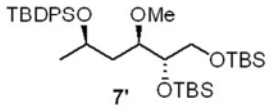
| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.3 Bar |
| Focus | Not active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 1.2 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 700.0 Vpp | Set Divert Valve | Source |

**Generate Molecular Formula Parameter**

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Formula, min. | C35H62O4Si3Na | | | | | | |
| Formula, max. | | | | | | | |
| Measured m/z | 653.385 | Tolerance | 5 | ppm | Charge | 1 | |
| Check Valence | no | Minimum | 0 | | Maximum | 0 | |
| Nirogen Rule | yes | Electron Configuration | both | | | | |
| Filter H/C Ratio | yes | Minimum | 0 | | Maximum | 3 | |
| Estimate Carbon | yes | | | | | | |




| Sum Formula | Sigma | m/z | Err [ppm] | Mean Err [ppm] | Err [mDa] | rdb | N Rule | e⁻ |
|---|---|---|---|---|---|---|---|---|
| C 35 H 62 Na 1 O 4 Si 3 | 0.031 | 653.3848 | 0.16 | 0.74 | 0.10 | 7.50 | ok | even |




7'

# Mass Spectrum Molecular Formula Report

**Analysis Info**

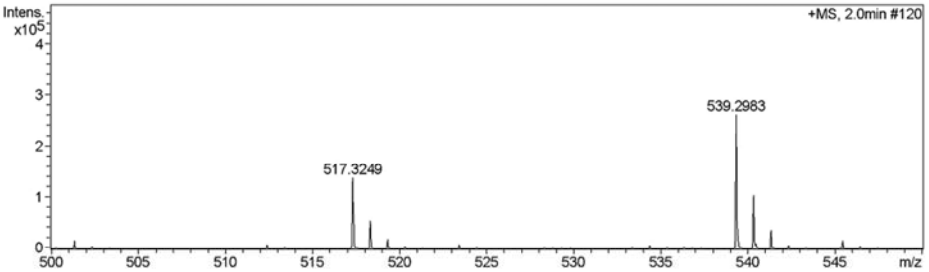
| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\20130622\SJ201205242--.d | Acquisition Date | 6/22/2013 4:24:24 PM |
| Method | yujia-shuijie.m | Operator | Bruker Customer |
| Sample Name | SJ201205242-- | Instrument / Ser# | micrOTOF-Q 125 |
| Comment | | | |

**Acquisition Parameter**

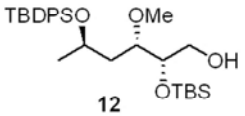
| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.3 Bar |
| Focus | Not active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 1.2 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 500.0 Vpp | Set Divert Valve | Source |

**Generate Molecular Formula Parameter**

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Formula, min. | C29H48O4Si2Na | | | | | | |
| Formula, max. | | | | | | | |
| Measured m/z | 539.298 | Tolerance | 5 | ppm | Charge | 1 | |
| Check Valence | no | Minimum | 0 | | Maximum | 0 | |
| Nirogen Rule | yes | Electron Configuration | both | | | | |
| Filter H/C Ratio | yes | Minimum | 0 | | Maximum | 3 | |
| Estimate Carbon | yes | | | | | | |




| Sum Formula | Sigma | m/z | Err [ppm] | Mean Err [ppm] | Err [mDa] | rdb | N Rule | e⁻ |
|---|---|---|---|---|---|---|---|---|
| C 29 H 48 Na 1 O 4 Si 2 | 0.227 | 539.2983 | 0.14 | 0.14 | 0.07 | 7.50 | ok | even |




**12**

# Mass Spectrum Molecular Formula Report

**Analysis Info**

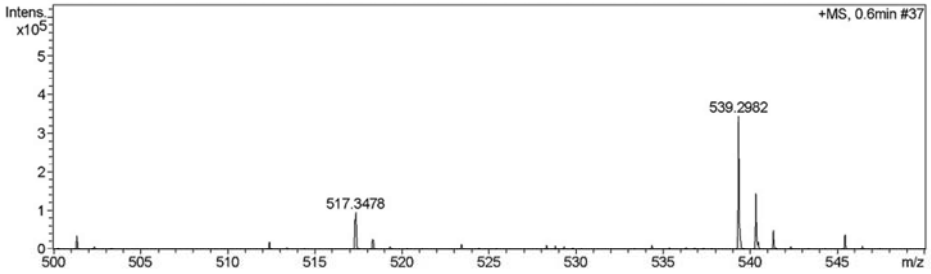
| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\20130622\SJ201205241.d | Acquisition Date | 6/22/2013 4:45:45 PM |
| Method | yujia-shuijie.m | Operator | Bruker Customer |
| Sample Name | SJ201205241 | Instrument / Ser# | micrOTOF-Q 125 |
| Comment | | | |

**Acquisition Parameter**

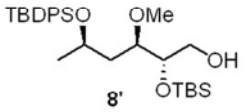
| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.3 Bar |
| Focus | Not active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 1.2 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 700.0 Vpp | Set Divert Valve | Source |

**Generate Molecular Formula Parameter**

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Formula, min. | C29H48O4Si2Na | | | | | | |
| Formula, max. | | | | | | | |
| Measured m/z | 539.298 | Tolerance | 5 | ppm | Charge | 1 | |
| Check Valence | no | Minimum | 0 | | Maximum | 0 | |
| Nirogen Rule | yes | Electron Configuration | both | | | | |
| Filter H/C Ratio | yes | Minimum | 0 | | Maximum | 3 | |
| Estimate Carbon | yes | | | | | | |




| Sum Formula | Sigma | m/z | Err [ppm] | Mean Err [ppm] | Err [mDa] | rdb | N Rule | e⁻ |
|---|---|---|---|---|---|---|---|---|
| C 29 H 48 Na 1 O 4 Si 2 | 0.011 | 539.2983 | 0.22 | 0.60 | 0.12 | 7.50 | ok | even |




**8'**

# Mass Spectrum Molecular Formula Report

**Analysis Info**

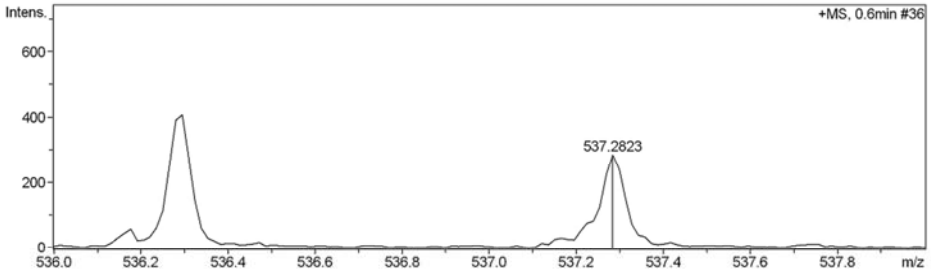
| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\20130622\SJ20121129A.d | Acquisition Date | 6/22/2013 4:51:26 PM |
| Method | yujia-shuijie.m | Operator | Bruker Customer |
| Sample Name | SJ20121129A | Instrument / Ser# | micrOTOF-Q 125 |
| Comment | | | |

**Acquisition Parameter**

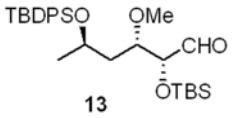
| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.3 Bar |
| Focus | Not active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 1.2 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 700.0 Vpp | Set Divert Valve | Source |

**Generate Molecular Formula Parameter**

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Formula, min. | C29H46O4Si2Na | | | | | | |
| Formula, max. | | | | | | | |
| Measured m/z | 537.282 | Tolerance | 5 | ppm | Charge | 1 | |
| Check Valence | no | Minimum | 0 | | Maximum | 0 | |
| Nirogen Rule | yes | Electron Configuration | both | | | | |
| Filter H/C Ratio | yes | Minimum | 0 | | Maximum | 3 | |
| Estimate Carbon | yes | | | | | | |




| Sum Formula | Sigma | m/z | Err [ppm] | Mean Err [ppm] | Err [mDa] | rdb | N Rule | e⁻ |
|---|---|---|---|---|---|---|---|---|
| C 29 H 46 Na 1 O 4 Si 2 | 0.227 | 537.2827 | 0.64 | 0.63 | 0.34 | 8.50 | ok | even |




**13**

# Mass Spectrum Molecular Formula Report

**Analysis Info**

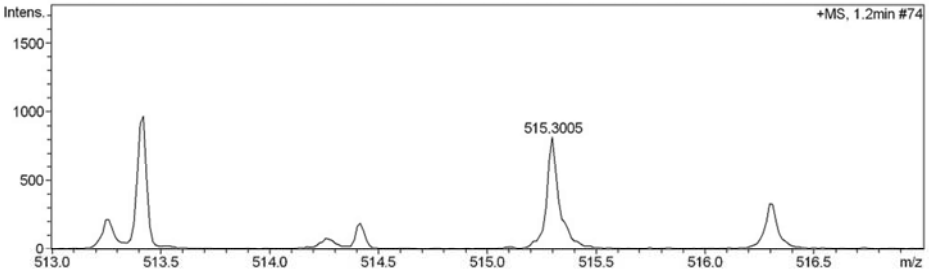
| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\20130622\SJ20120904.d | Acquisition Date | 6/22/2013 4:48:20 PM |
| Method | yujia-shuijie.m | Operator | Bruker Customer |
| Sample Name | SJ20120904 | Instrument / Ser# | micrOTOF-Q 125 |
| Comment | | | |

**Acquisition Parameter**

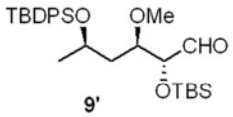
| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.3 Bar |
| Focus | Not active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 1.2 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 600.0 Vpp | Set Divert Valve | Source |

**Generate Molecular Formula Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Formula, min. | C29H46O4Si2H | | | | |
| Formula, max. | | | | | |
| Measured m/z | 515.301 | Tolerance | 10 ppm | Charge | 1 |
| Check Valence | no | Minimum | 0 | Maximum | 0 |
| Nirogen Rule | yes | Electron Configuration | both | | |
| Filter H/C Ratio | yes | Minimum | 0 | Maximum | 3 |
| Estimate Carbon | yes | | | | |




| Sum Formula | Sigma | m/z | Err [ppm] | Mean Err [ppm] | Err [mDa] | rdb | N Rule | e⁻ |
|---|---|---|---|---|---|---|---|---|
| C 29 H 47 O 4 Si 2 | 0.227 | 515.3007 | 0.48 | 0.48 | 0.25 | 8.50 | ok | even |




**9'**

# Mass Spectrum Molecular Formula Report

**Analysis Info**

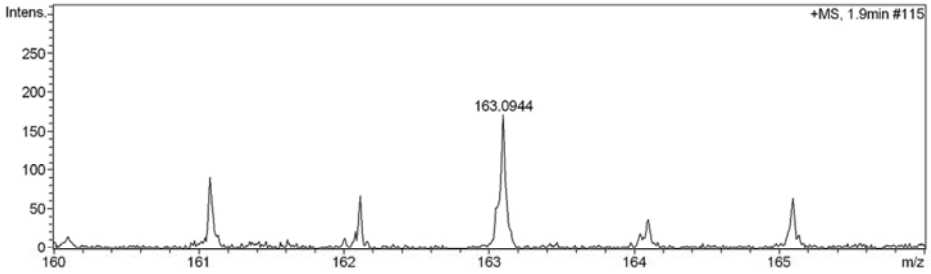
| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\20130622\SJ20121130A.d | Acquisition Date | 6/22/2013 4:55:08 PM |
| Method | yujia-shuijie.m | Operator | Bruker Customer |
| Sample Name | SJ20121130A | Instrument / Ser# | micrOTOF-Q 125 |
| Comment | | | |

**Acquisition Parameter**

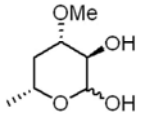
| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.3 Bar |
| Focus | Not active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 1.2 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 200.0 Vpp | Set Divert Valve | Source |

**Generate Molecular Formula Parameter**

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Formula, min. | C7H14O4H | | | | | | |
| Formula, max. | | | | | | | |
| Measured m/z | 163.094 | Tolerance | 5 | mDa | Charge | 1 | |
| Check Valence | no | Minimum | 0 | | Maximum | 0 | |
| Nirogen Rule | yes | Electron Configuration | both | | | | |
| Filter H/C Ratio | yes | Minimum | 0 | | Maximum | 3 | |
| Estimate Carbon | yes | | | | | | |




| Sum Formula | Sigma | m/z | Err [ppm] | Mean Err [ppm] | Err [mDa] | rdb | N Rule | e⁻ |
|---|---|---|---|---|---|---|---|---|
| C 7 H 15 O 4 | 0.046 | 163.0965 | 12.84 | 12.69 | 2.09 | 0.50 | ok | even |




I

# Mass Spectrum Molecular Formula Report

**Analysis Info**

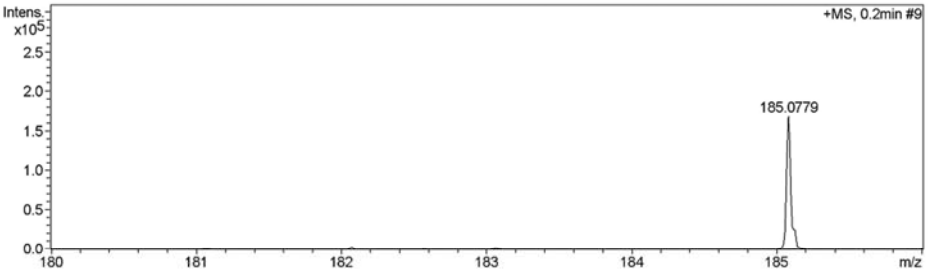
| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\20130626\SJ20121128A.d | Acquisition Date | 6/26/2013 10:29:15 AM |
| Method | MA250-550POS.m | Operator | Bruker Customer |
| Sample Name | SJ20121128A | Instrument / Ser# | micrOTOF-Q 125 |
| Comment | | | |

**Acquisition Parameter**

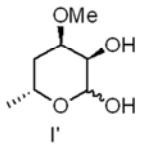
| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.3 Bar |
| Focus | Not active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 4.0 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 300.0 Vpp | Set Divert Valve | Source |

**Generate Molecular Formula Parameter**

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Formula, min. | C7H14O4Na | | | | | | |
| Formula, max. | | | | | | | |
| Measured m/z | 185.078 | Tolerance | 5 | ppm | Charge | 1 | |
| Check Valence | no | Minimum | 0 | | Maximum | 0 | |
| Nirogen Rule | no | Electron Configuration | both | | | | |
| Filter H/C Ratio | no | Minimum | 0 | | Maximum | 3 | |
| Estimate Carbon | yes | | | | | | |




| Sum Formula | Sigma | m/z | Err [ppm] | Mean Err [ppm] | Err [mDa] | rdb | N Rule | e⁻ |
|---|---|---|---|---|---|---|---|---|
| C 7 H 14 Na 1 O 4 | 0.046 | 185.0784 | 3.01 | 3.01 | 0.56 | 0.50 | ok | even |




I'

# Mass Spectrum Molecular Formula Report

**Analysis Info**

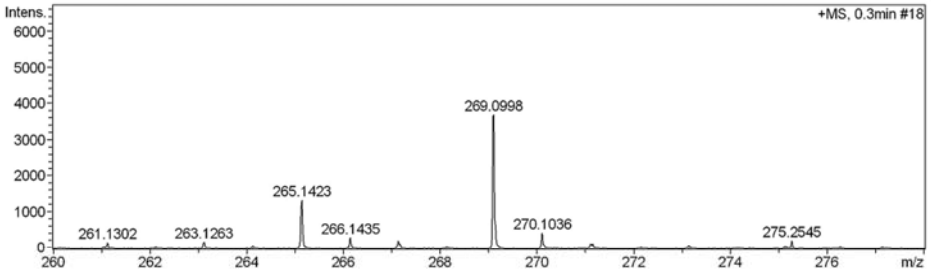
| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\20130622\SJ20130424B.d | Acquisition Date | 6/22/2013 5:07:07 PM |
| Method | yujia-shuijie.m | Operator | Bruker Customer |
| Sample Name | SJ20130424B | Instrument / Ser# | micrOTOF-Q 125 |
| Comment | | | |

**Acquisition Parameter**

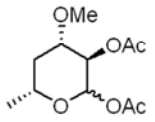
| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.3 Bar |
| Focus | Not active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 1.2 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 450.0 Vpp | Set Divert Valve | Source |

**Generate Molecular Formula Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Formula, min. | C11H18O6Na | | | | |
| Formula, max. | | | | | |
| Measured m/z | 269.1 | Tolerance | 5 ppm | Charge | 1 |
| Check Valence | no | Minimum | 0 | Maximum | 0 |
| Nirogen Rule | yes | Electron Configuration | both | | |
| Filter H/C Ratio | yes | Minimum | 0 | Maximum | 3 |
| Estimate Carbon | yes | | | | |




| Sum Formula | Sigma | m/z | Err [ppm] | Mean Err [ppm] | Err [mDa] | rdb | N Rule | e⁻ |
|---|---|---|---|---|---|---|---|---|
| C 11 H 18 Na 1 O 6 | 0.013 | 269.0996 | -0.89 | -1.02 | -0.24 | 2.50 | ok | even |




**14**

# Mass Spectrum Molecular Formula Report

**Analysis Info**

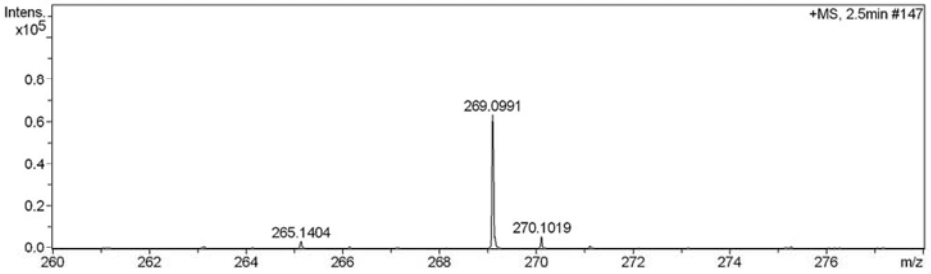
| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\20130622\SJ20130417.d | Acquisition Date | 6/22/2013 5:02:42 PM |
| Method | yujia-shuijie.m | Operator | Bruker Customer |
| Sample Name | SJ20130417 | Instrument / Ser# | micrOTOF-Q 125 |
| Comment | | | |

**Acquisition Parameter**

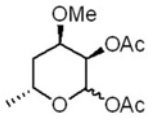
| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.3 Bar |
| Focus | Not active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 1.2 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 450.0 Vpp | Set Divert Valve | Source |

**Generate Molecular Formula Parameter**

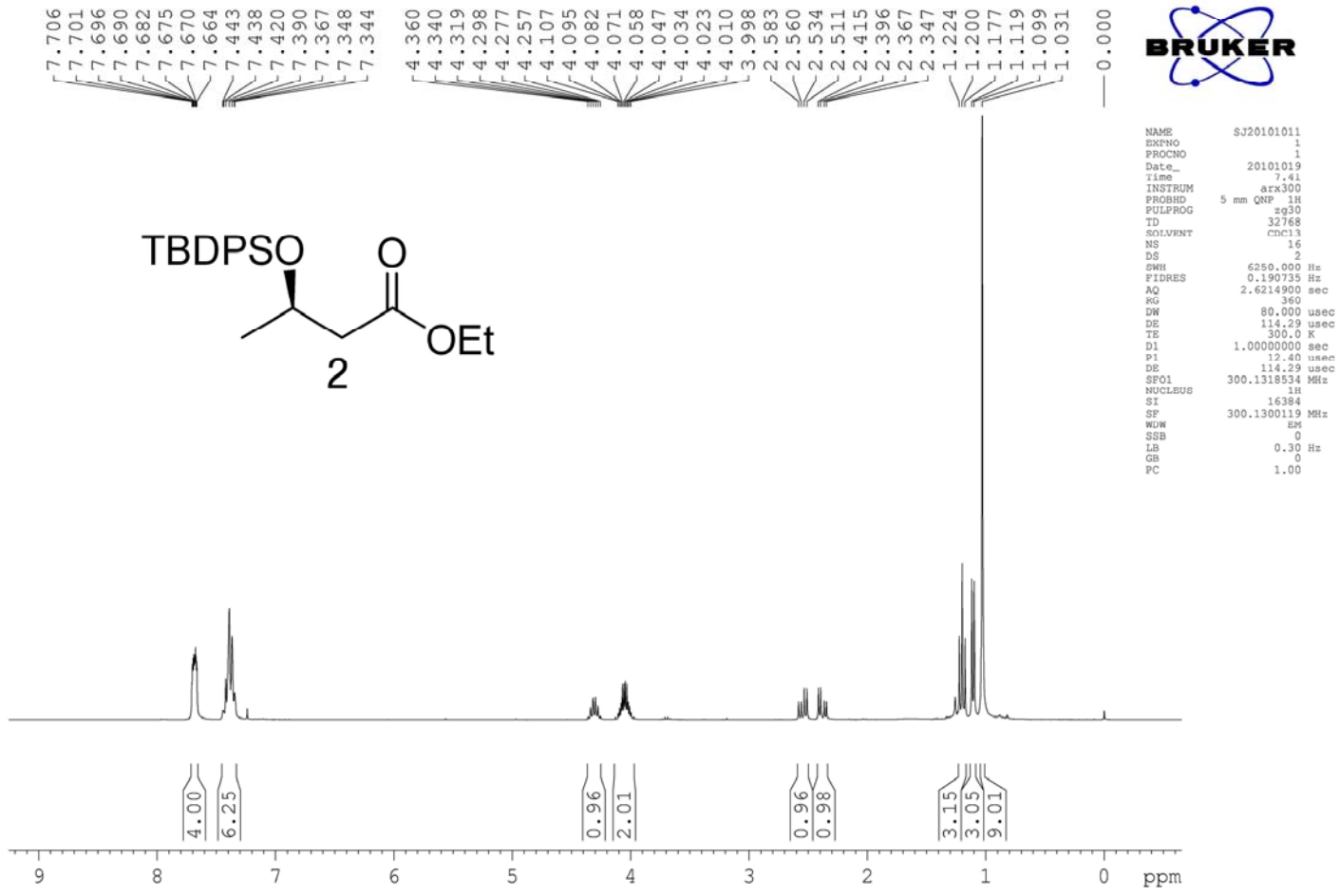
| | | | | | |
|---|---|---|---|---|---|
| Formula, min. | C11H18O6Na | | | | |
| Formula, max. | | | | | |
| Measured m/z | 269.099 | Tolerance | 5 ppm | Charge | 1 |
| Check Valence | no | Minimum | 0 | Maximum | 0 |
| Nirogen Rule | yes | Electron Configuration | both | | |
| Filter H/C Ratio | yes | Minimum | 0 | Maximum | 3 |
| Estimate Carbon | yes | | | | |




| Sum Formula | Sigma | m/z | Err [ppm] | Mean Err [ppm] | Err [mDa] | rdb | N Rule | e⁻ |
|---|---|---|---|---|---|---|---|---|
| C 11 H 18 Na 1 O 6 | 0.019 | 269.0996 | 1.81 | 1.85 | 0.49 | 2.50 | ok | even |




**10'**


7.706
7.701
7.696
7.690
7.682
7.675
7.670
7.664
7.443
7.438
7.420
7.390
7.367
7.348
7.344
4.360
4.340
4.319
4.298
4.277
4.257
4.107
4.095
4.082
4.071
4.058
4.047
4.034
4.023
4.010
3.998
2.583
2.560
2.534
2.511
2.415
2.396
2.367
2.347
1.224
1.200
1.177
1.119
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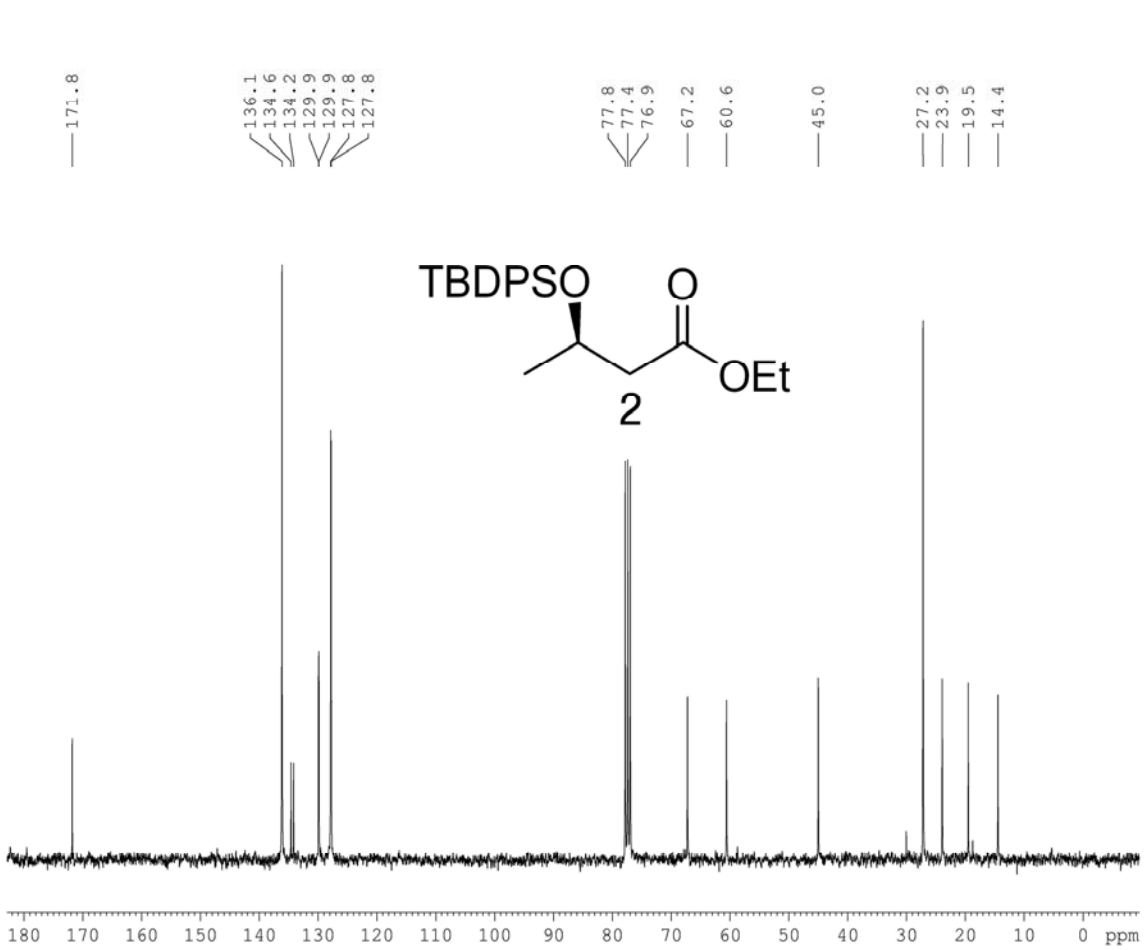
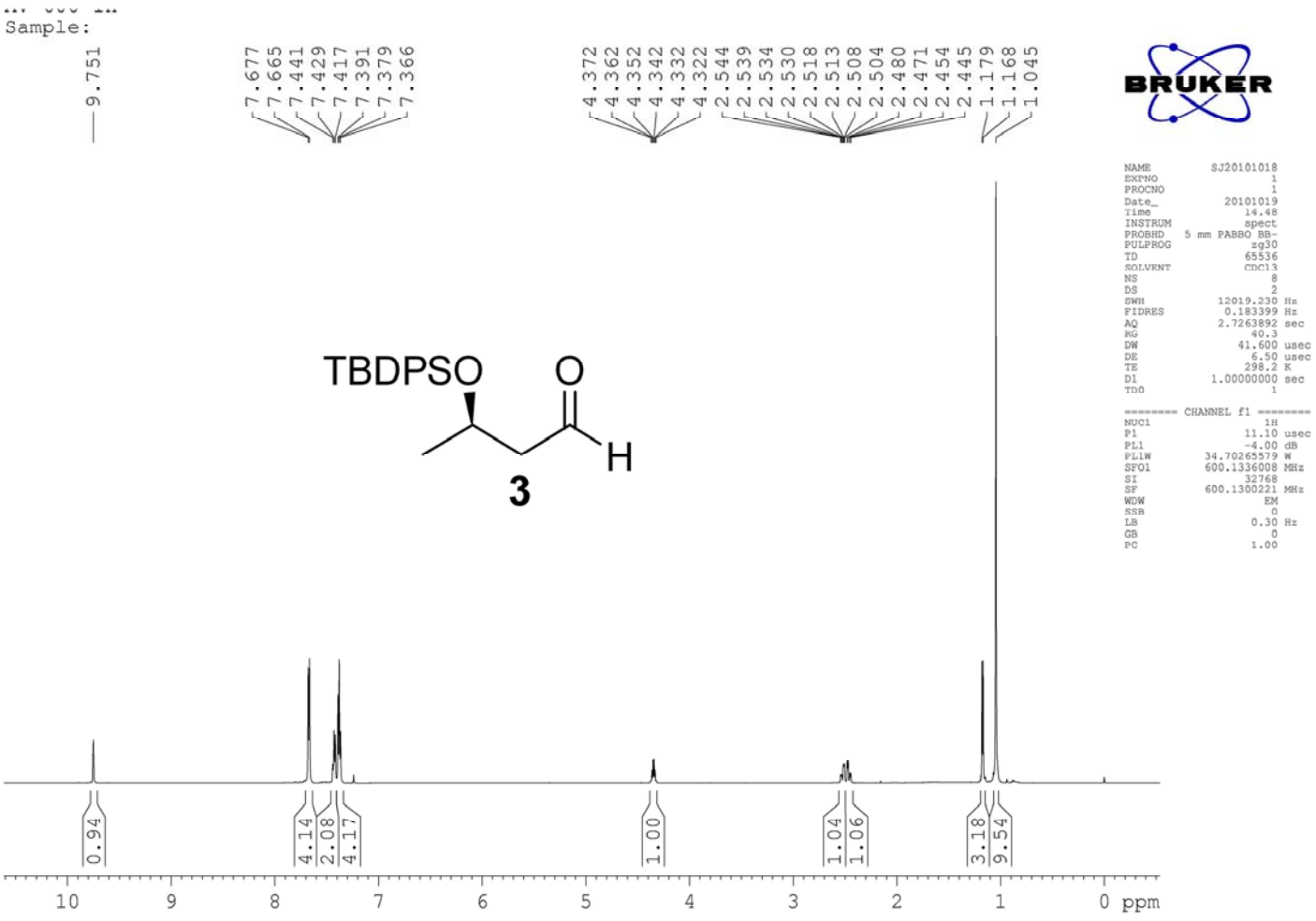
PROCNO 1
Date_ 20101019
Time 7.41
INSTRUM arx300
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6250.000 Hz
FIDRES 0.190735 Hz
AQ 2.6214900 sec
RG 360
DW 80.000 usec
DE 114.29 usec
TE 300.0 K
D1 1.00000000 sec
P1 12.40 usec
DE 114.29 usec
SFO1 300.1318534 MHz
NUCLEUS 1H
SI 16384
SF 300.1300119 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
4.00
6.25
0.96
2.01
0.96
0.98
3.15
3.05
9.01
9
8
7
6
5
4
3
2
1
0
ppm



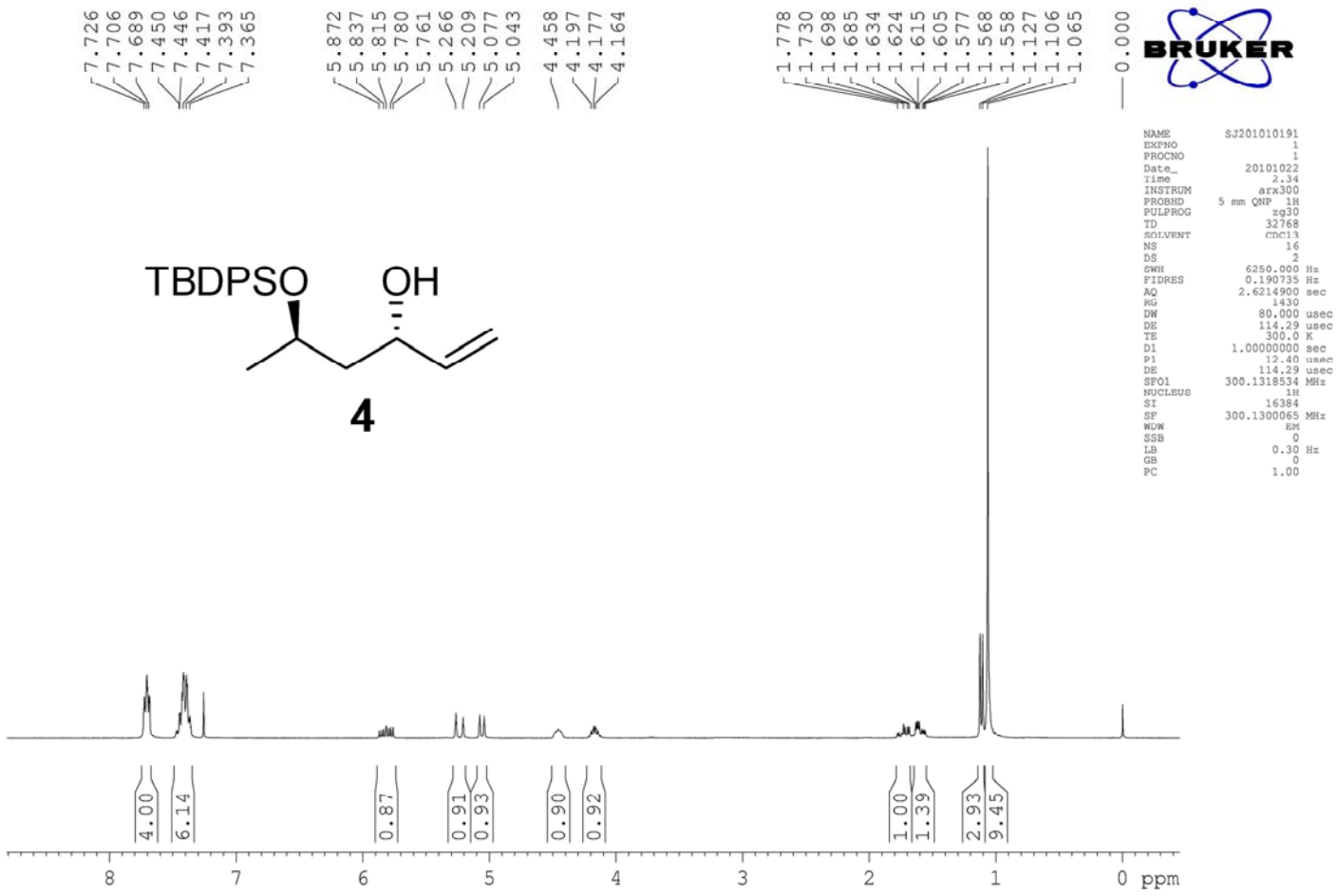

BRUKER

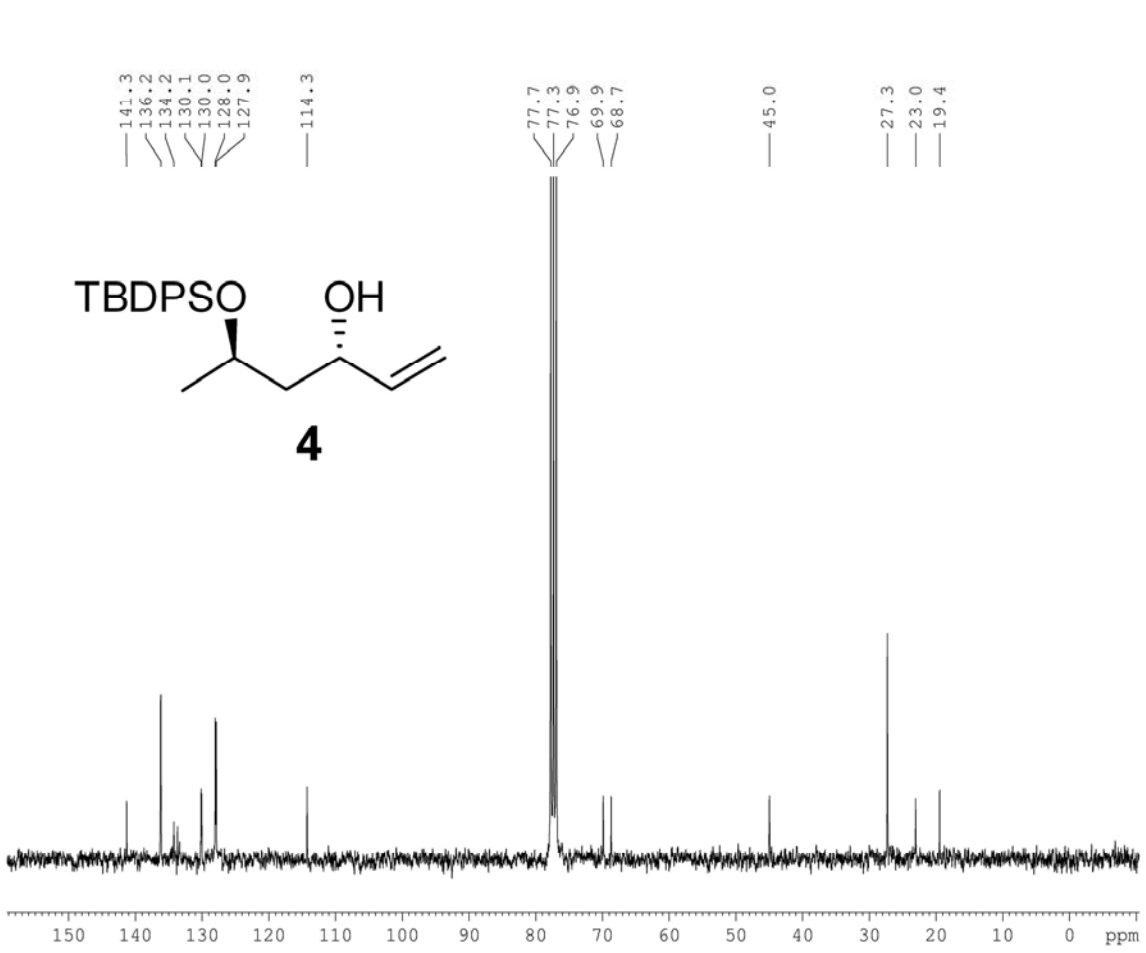
| | |
|---|---|
| NAME | SJ20101011 |
| EXPNO | 2 |
| PROCNO | 1 |
| Date_ | 20101019 |
| Time | 23.52 |
| INSTRUM | arx300 |
| PROBHD | 5 mm QNP 1H |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 261 |
| DS | 2 |
| SWH | 25000.000 Hz |
| FIDRES | 0.381470 Hz |
| AQ | 1.3107700 sec |
| RG | 22800 |
| DW | 20.000 usec |
| DE | 28.57 usec |
| TE | 300.0 K |
| D12 | 0.00002000 sec |
| DL6 | 20.00 dB |
| D1 | 2.00000000 sec |
| CPDPRG | waltz16 |
| P31 | 80.00 usec |
| D11 | 0.03000000 sec |
| DL5 | 20.00 dB |
| P1 | 13.70 usec |
| DE | 28.57 usec |
| SFO1 | 75.4772501 MHz |
| NUCLEUS | 13C |
| SI | 32768 |
| SF | 75.4677274 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 3.00 Hz |
| GB | 0 |
| PC | 1.40 |


Sample:
9.751
7.677
7.665
7.441
7.429
7.417
7.391
7.379
7.366
4.372
4.362
4.352
4.342
4.332
4.322
2.544
2.539
2.534
2.530
2.518
2.513
2.508
2.504
2.480
2.471
2.454
2.445
1.179
1.168
1.045
BRUKER
NAME SJ20101018
EXPNO 1
PROCNO 1
Date_ 20101019
Time 14.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263892 sec
RG 40.3
DW 41.600 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.10 usec
PL1 -4.00 dB
PL1W 34.70265579 W
SFO1 600.1336008 MHz
SI 32768
SF 600.1300221 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TBDPSO
O
H
3
0.94
4.14
2.08
4.17
1.00
1.04
1.06
3.18
9.54
10
9
8
7
6
5
4
3
2
1
0 ppm


BRUKER

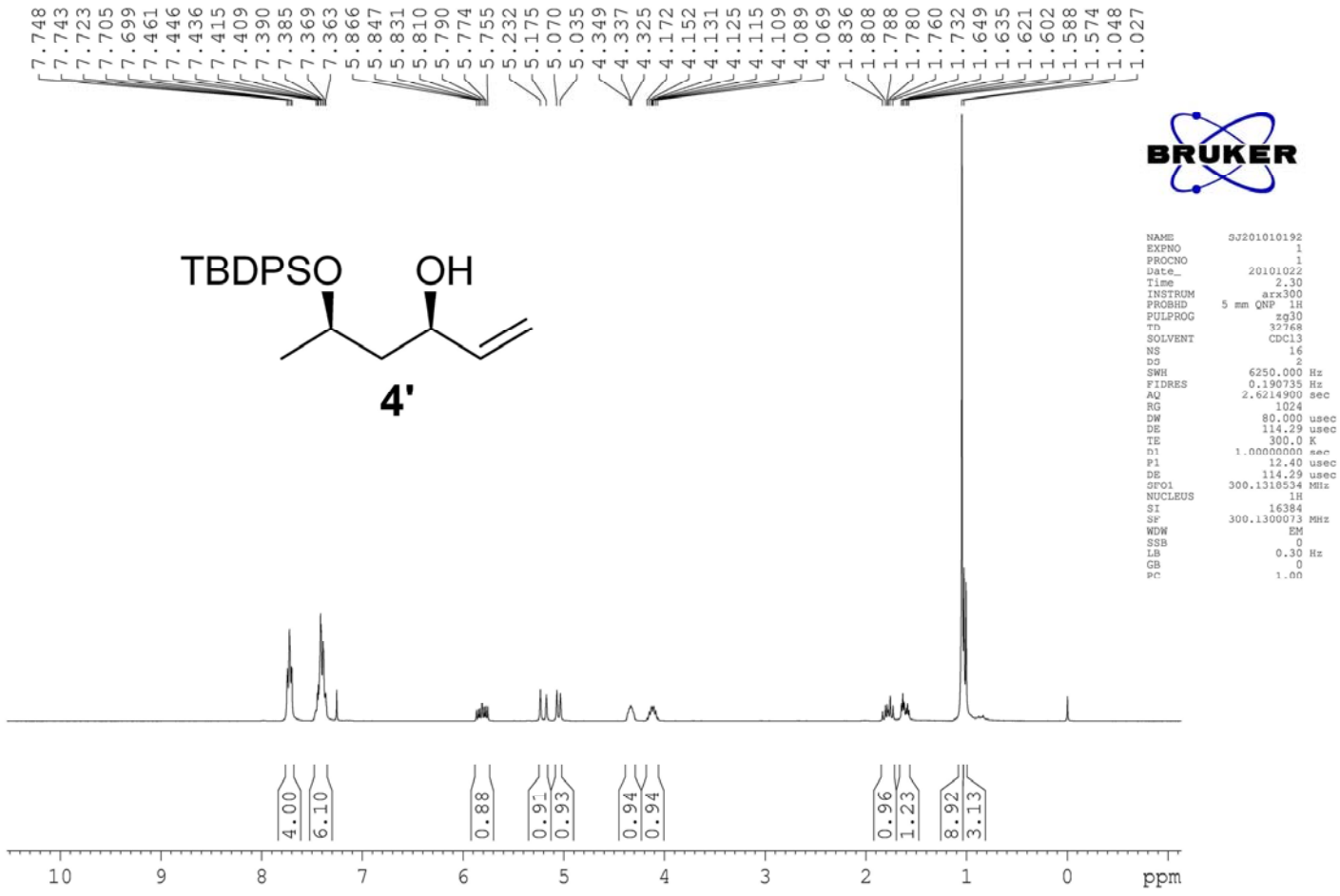
| | | |
|---|---|---|
| NAME | SJ20101018 | |
| EXPNO | 2 | |
| PROCNO | 1 | |
| Date_ | 20101019 | |
| Time | 14.56 | |
| INSTRUM | spect | |
| PROBHD | 5 mm PABBO BB- | |
| PULPROG | zgpg30 | |
| TD | 65536 | |
| SOLVENT | CDCl3 | |
| NS | 59 | |
| DS | 2 | |
| SWH | 45454.547 | Hz |
| FIDRES | 0.693581 | Hz |
| AQ | 0.7209570 | sec |
| RG | 20600 | |
| DW | 11.000 | usec |
| DE | 6.50 | usec |
| TE | 298.2 | K |
| D1 | 2.00000000 | sec |
| D11 | 0.03000000 | sec |
| TD0 | 1 | |
| ======== CHANNEL f1 ======== | | |
| NUC1 | 13C | |
| P1 | 6.00 | usec |
| PL1 | 1.00 | dB |
| PL1W | 83.20243835 | W |
| SFO1 | 150.9178993 | MHz |
| ======== CHANNEL f2 ======== | | |
| CPDPRG2 | waltz16 | |
| NUC2 | 1H | |
| PCPD2 | 80.00 | usec |
| PL2 | -4.00 | dB |
| PL12 | 13.16 | dB |
| PL13 | 16.00 | dB |
| PL2W | 34.70265579 | W |
| PL12W | 0.66736388 | W |
| PL13W | 0.34702653 | W |
| SFO2 | 600.1324005 | MHz |
| SI | 32768 | |
| SF | 150.9028165 | MHz |
| WDW | EM | |
| SSB | 0 | |
| LB | 3.00 | Hz |
| GB | 0 | |
| PC | 1.40 | |

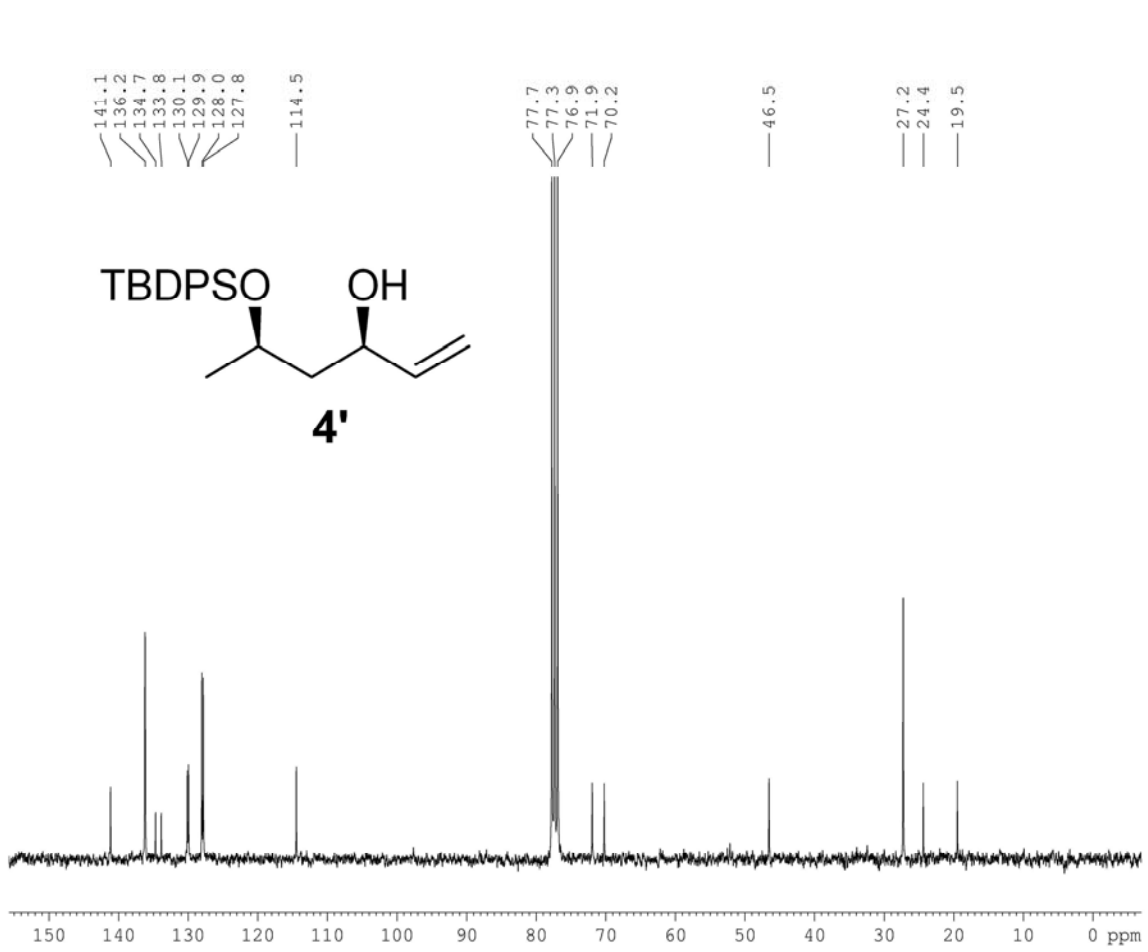

7.726
7.706
7.689
7.450
7.446
7.417
7.393
7.365
5.872
5.837
5.815
5.780
5.761
5.266
5.209
5.077
5.043
4.458
4.197
4.177
4.164
1.778
1.730
1.698
1.685
1.634
1.624
1.615
1.605
1.577
1.568
1.558
1.127
1.106
1.065
0.000
BRUKER
TBDPSO
OH
4
NAME SJ201010191
EXPNO 1
PROCNO 1
Date_ 20101022
Time 2.34
INSTRUM arx300
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6250.000 Hz
FIDRES 0.190735 Hz
AQ 2.6214900 sec
RG 1430
DW 80.000 usec
DE 114.29 usec
TE 300.0 K
D1 1.00000000 sec
P1 12.40 usec
DE 114.29 usec
SFO1 300.1318534 MHz
NUCLEUS 1H
SI 16384
SF 300.1300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
4.00
6.14
0.87
0.91
0.93
0.90
0.92
1.00
1.39
2.93
9.45
8
7
6
5
4
3
2
1
0 ppm




BRUKER

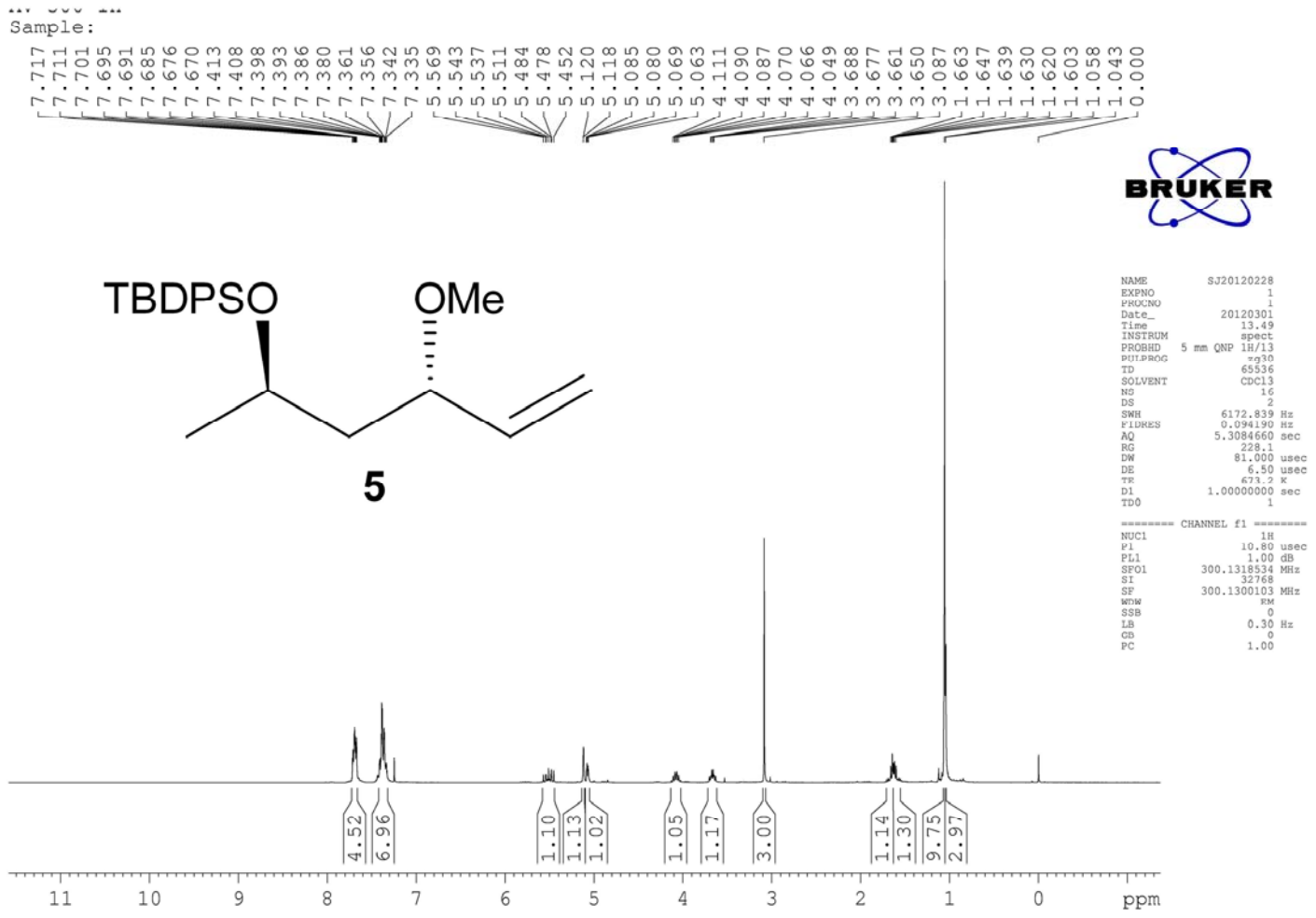
| | |
|---|---|
| NAME | SJ201010191 |
| EXPNO | 2 |
| PROCNO | 1 |
| Date_ | 20101023 |
| Time | 8.26 |
| INSTRUM | arx300 |
| PROBHD | 5 mm QNP 1H |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 467 |
| DS | 2 |
| SWH | 25000.000 Hz |
| FIDRES | 0.381470 Hz |
| AQ | 1.3107700 sec |
| RG | 22800 |
| DW | 20.000 usec |
| DE | 28.57 usec |
| TE | 300.0 K |
| D12 | 0.00002000 sec |
| DL6 | 20.00 dB |
| D1 | 2.00000000 sec |
| CPDPRG | waltz16 |
| P31 | 80.00 usec |
| D11 | 0.03000000 sec |
| DL5 | 20.00 dB |
| P1 | 13.70 usec |
| DE | 28.57 usec |
| SFO1 | 75.4772501 MHz |
| NUCLEUS | 13C |
| SI | 32768 |
| SF | 75.4677274 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 3.00 Hz |
| GB | 0 |
| PC | 1.40 |

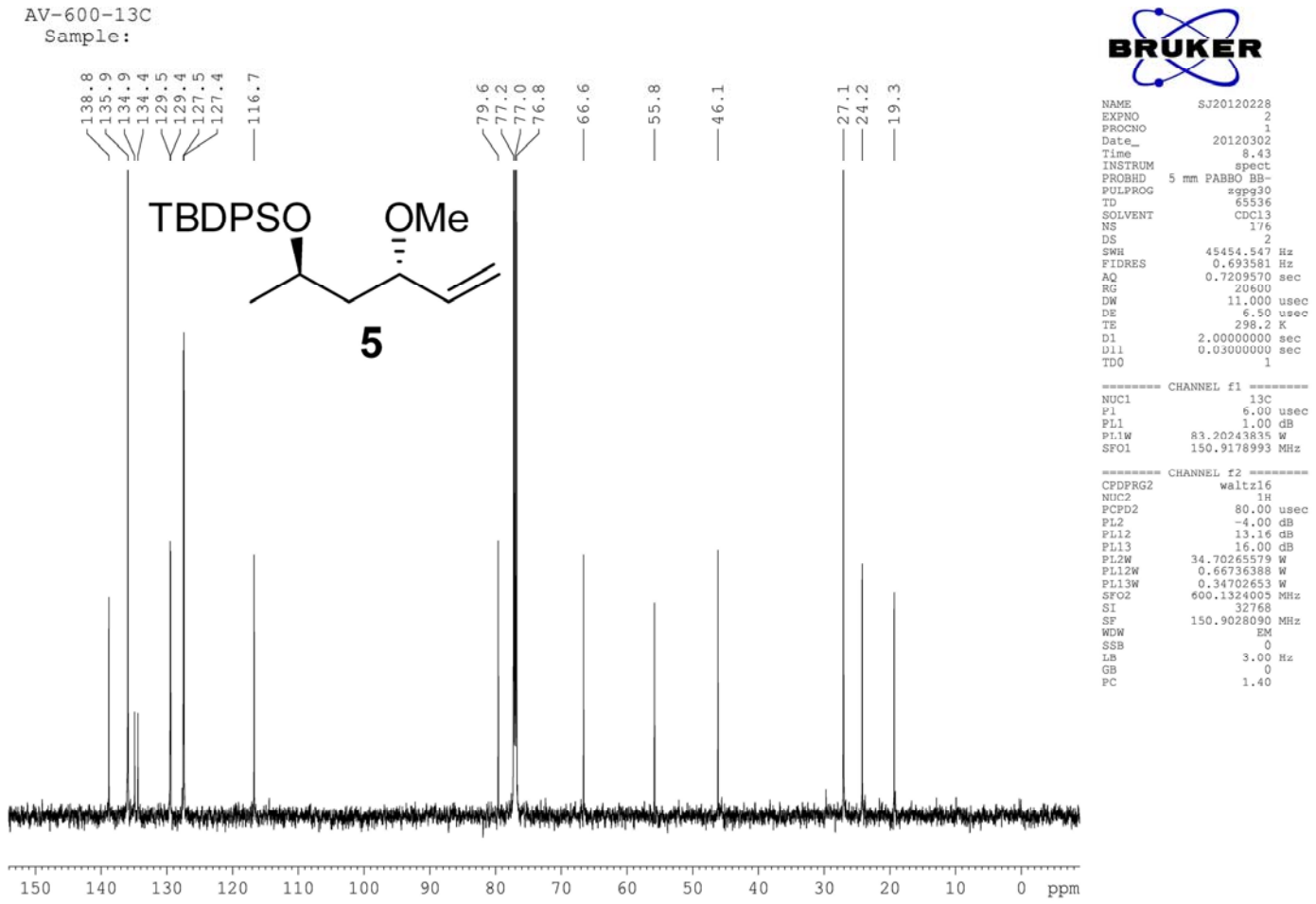

7.748
7.743
7.723
7.705
7.699
7.461
7.446
7.436
7.415
7.409
7.390
7.385
7.369
7.363
5.866
5.847
5.831
5.810
5.790
5.774
5.755
5.232
5.175
5.070
5.035
4.349
4.337
4.325
4.172
4.152
4.131
4.125
4.115
4.109
4.089
4.069
1.836
1.808
1.788
1.780
1.760
1.732
1.649
1.635
1.621
1.602
1.588
1.574
1.048
1.027
TBDPSO
OH
4'
BRUKER
NAME 5J201010192
EXPNO 1
PROCNO 1
Date_ 20101022
Time 2.30
INSTRUM arx300
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6250.000 Hz
FIDRES 0.190735 Hz
AQ 2.6214900 sec
RG 1024
DW 80.000 usec
DE 114.29 usec
TE 300.0 K
D1 1.00000000 sec
P1 12.40 usec
DE 114.29 usec
SFO1 300.1310534 MHz
NUCLEUS 1H
SI 16384
SF 300.1300073 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
4.00
6.10
0.88
0.91
0.93
0.94
0.94
0.96
1.23
8.92
3.13
10
9
8
7
6
5
4
3
2
1
0
ppm




BRUKER

| | |
|---|---|
| NAME | SJ201010192 |
| EXPNO | 2 |
| PROCNO | 1 |
| Date_ | 20101023 |
| Time | 7.42 |
| INSTRUM | arx300 |
| PROBHD | 5 mm QNP 1H |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 738 |
| DS | 2 |
| SWH | 25000.000 Hz |
| FIDRES | 0.381470 Hz |
| AQ | 1.3107700 sec |
| RG | 22800 |
| DW | 20.000 usec |
| DE | 28.57 usec |
| TE | 300.0 K |
| D12 | 0.00002000 sec |
| DL6 | 20.00 dB |
| D1 | 2.00000000 sec |
| CPDPRG | waltz16 |
| P31 | 80.00 usec |
| D11 | 0.03000000 sec |
| DL5 | 20.00 dB |
| P1 | 13.70 usec |
| DE | 28.57 usec |
| SFO1 | 75.4772501 MHz |
| NUCLEUS | 13C |
| SI | 32768 |
| SF | 75.4677274 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 3.00 Hz |
| GB | 0 |
| PC | 1.40 |

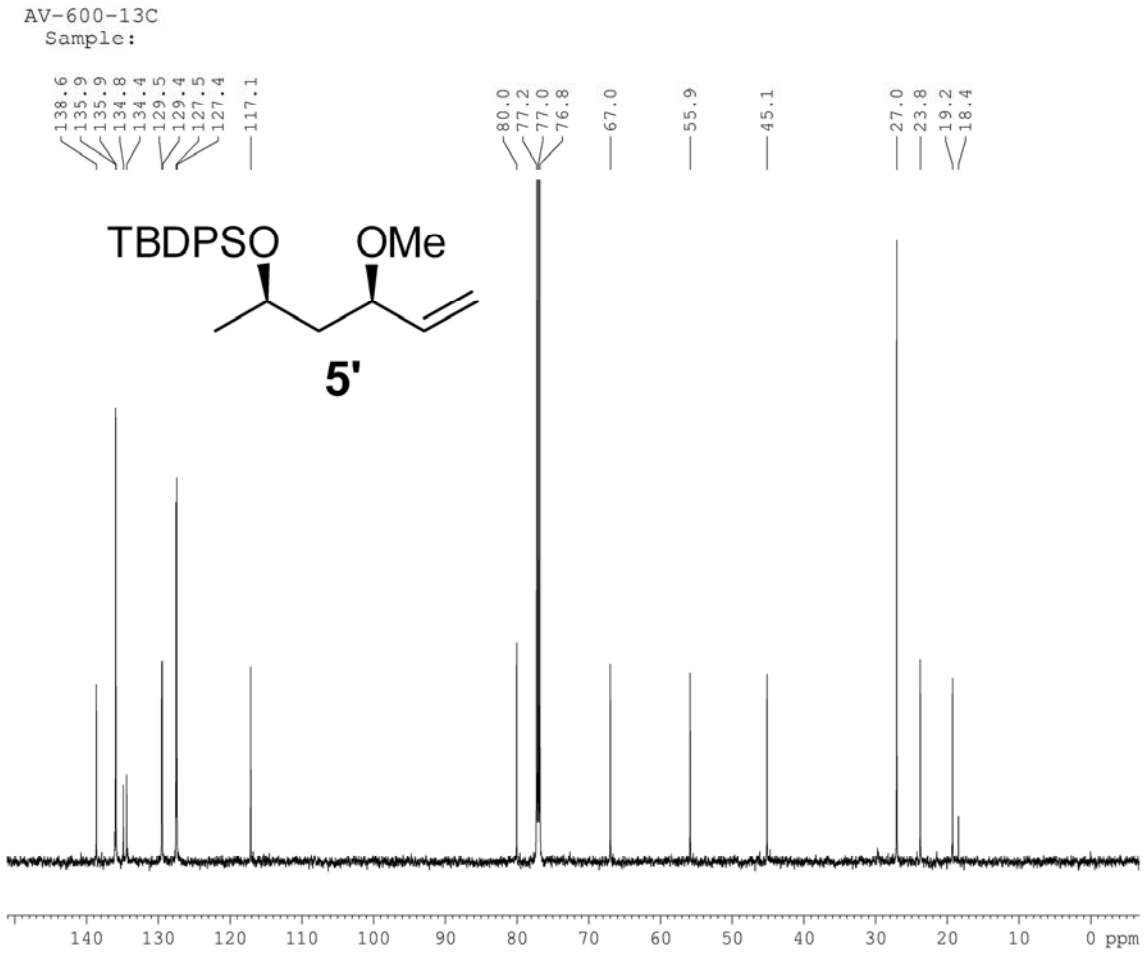

Sample:
7.717
7.711
7.701
7.695
7.691
7.685
7.676
7.670
7.413
7.408
7.398
7.393
7.386
7.380
7.361
7.356
7.342
7.335
5.569
5.543
5.537
5.511
5.484
5.478
5.452
5.120
5.118
5.085
5.080
5.069
5.063
4.111
4.090
4.087
4.070
4.066
4.049
3.688
3.677
3.661
3.650
3.087
1.663
1.647
1.639
1.630
1.620
1.603
1.058
1.043
0.000
BRUKER
TBDPSO
OMe
5
NAME SJ20120228
EXPNO 1
PROCNO 1
Date_ 20120301
Time 13.49
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 228.1
DW 81.000 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PL1 1.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
4.52
6.96
1.10
1.13
1.02
1.05
1.17
3.00
1.14
1.30
9.75
2.97
11
10
9
8
7
6
5
4
3
2
1
0
ppm




BRUKER

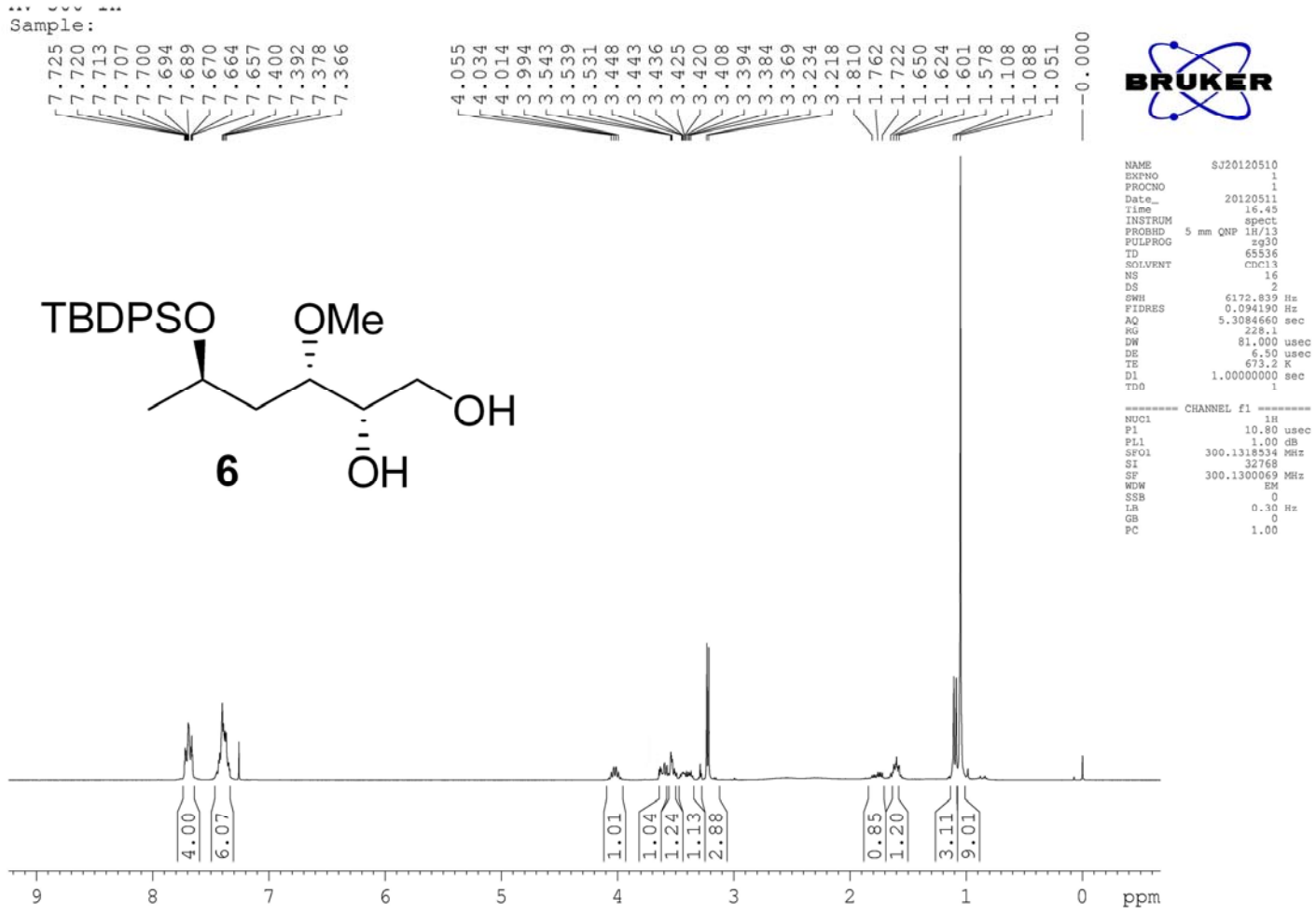
| | |
|---|---|
| NAME | SJ20120228 |
| EXPNO | 2 |
| PROCNO | 1 |
| Date_ | 20120302 |
| Time | 8.43 |
| INSTRUM | spect |
| PROBHD | 5 mm PABBO BB- |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 176 |
| DS | 2 |
| SWH | 45454.547 Hz |
| FIDRES | 0.693581 Hz |
| AQ | 0.7209570 sec |
| RG | 20600 |
| DW | 11.000 usec |
| DE | 6.50 usec |
| TE | 298.2 K |
| D1 | 2.00000000 sec |
| D11 | 0.03000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 6.00 usec |
| PL1 | 1.00 dB |
| PL1W | 83.20243835 W |
| SFO1 | 150.9178993 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | -4.00 dB |
| PL12 | 13.16 dB |
| PL13 | 16.00 dB |
| PL2W | 34.70265579 W |
| PL12W | 0.66736388 W |
| PL13W | 0.34702653 W |
| SFO2 | 600.1324005 MHz |
| SI | 32768 |
| SF | 150.9028090 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 3.00 Hz |
| GB | 0 |
| PC | 1.40 |

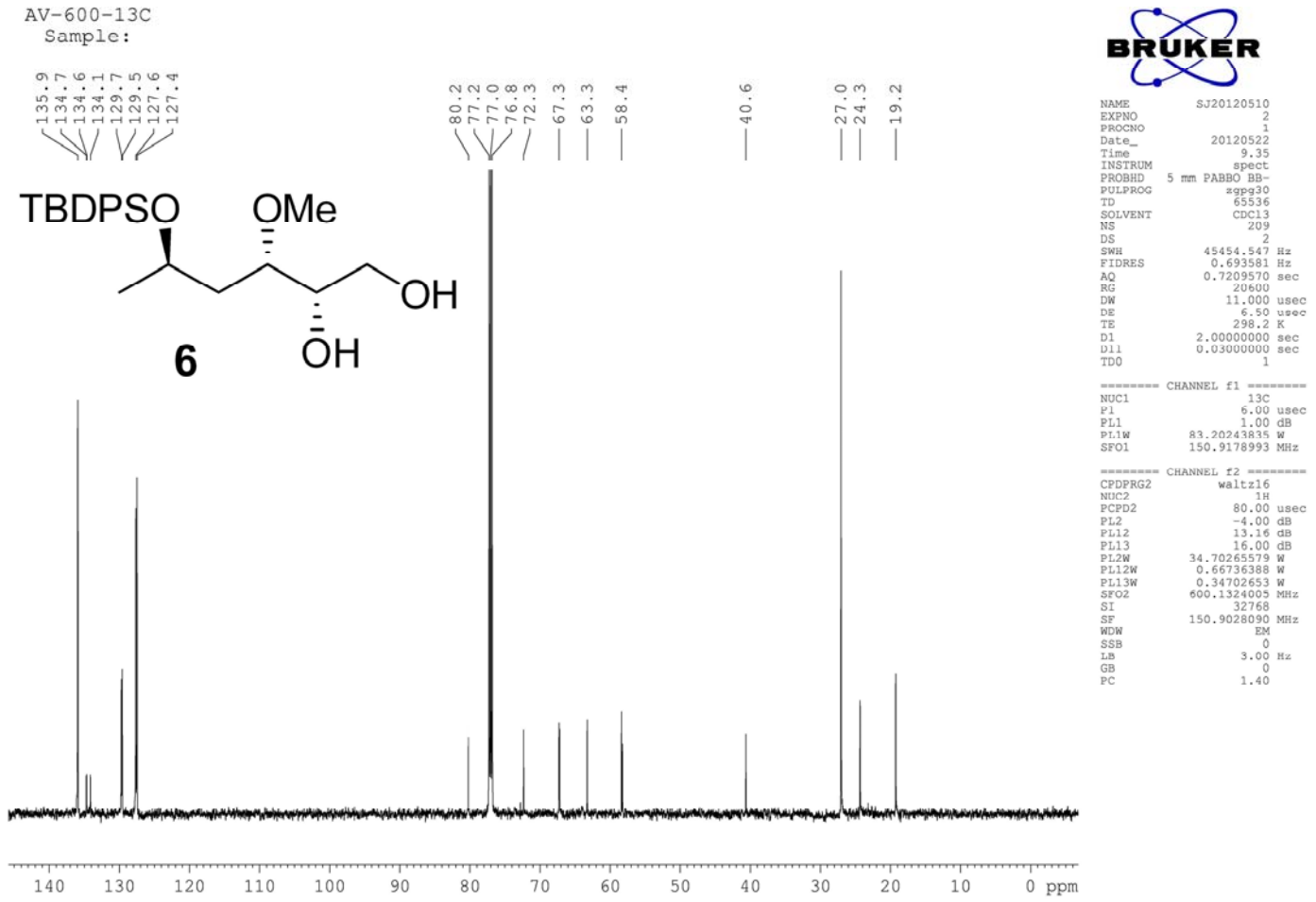

Sample:
7.697
7.691
7.671
7.665
7.408
7.378
7.353
5.594
5.537
5.535
5.477
5.110
5.080
5.076
5.061
5.059
5.002
3.975
3.955
3.715
3.695
3.670
3.648
3.186
1.979
1.957
1.934
1.911
1.889
1.563
1.523
1.477
1.090
1.070
1.049
0.000
BRUKER
TBDPSO
OMe
5'
NAME SJ20120225
EXPNO 1
PROCNO 1
Date_ 20120227
Time 10.49
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 114
DW 81.000 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PL1 1.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
4.43
6.96
1.10
1.13
1.12
1.02
1.31
3.00
1.11
1.17
2.84
8.95
8
7
6
5
4
3
2
1
ppm




BRUKER

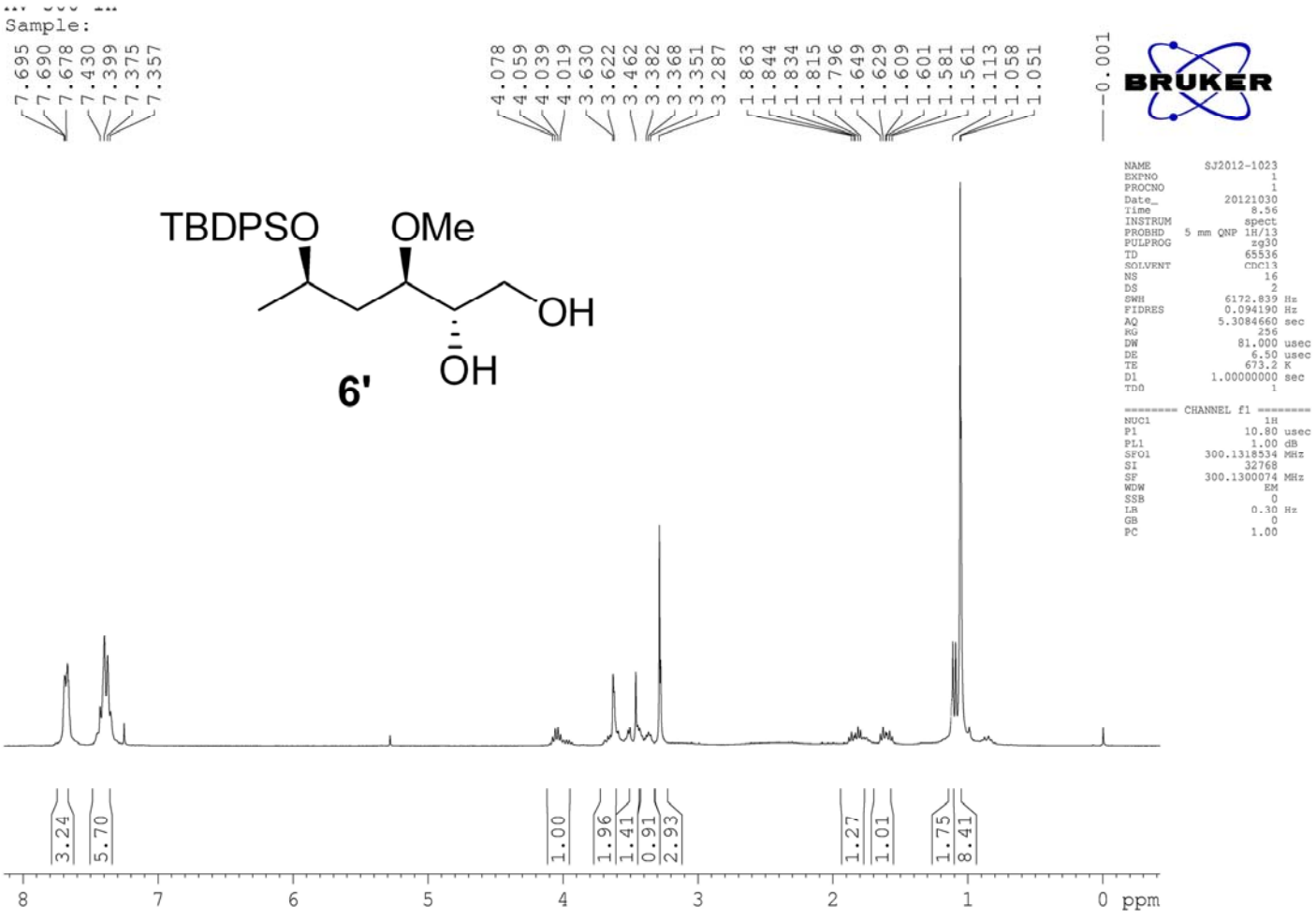
| | | |
|---|---|---|
| NAME | SJ20120225 | |
| EXPNO | 2 | |
| PROCNO | 1 | |
| Date_ | 20120228 | |
| Time | 8.28 | |
| INSTRUM | spect | |
| PROBHD | 5 mm PABBO BB- | |
| PULPROG | zgpg30 | |
| TD | 65536 | |
| SOLVENT | CDCl3 | |
| NS | 133 | |
| DS | 2 | |
| SWH | 45454.547 | Hz |
| FIDRES | 0.693581 | Hz |
| AQ | 0.7209570 | sec |
| RG | 20600 | |
| DW | 11.000 | usec |
| DE | 6.50 | usec |
| TE | 298.2 | K |
| D1 | 2.00000000 | sec |
| D11 | 0.03000000 | sec |
| TD0 | 1 | |
| ======== CHANNEL f1 ======== | | |
| NUC1 | 13C | |
| P1 | 6.00 | usec |
| PL1 | 1.00 | dB |
| PL1W | 83.20243835 | W |
| SFO1 | 150.9178993 | MHz |
| ======== CHANNEL f2 ======== | | |
| CPDPRG2 | waltz16 | |
| NUC2 | 1H | |
| PCPD2 | 80.00 | usec |
| PL2 | -4.00 | dB |
| PL12 | 13.16 | dB |
| PL13 | 16.00 | dB |
| PL2W | 34.70265579 | W |
| PL12W | 0.66736388 | W |
| PL13W | 0.34702653 | W |
| SFO2 | 600.1324005 | MHz |
| SI | 32768 | |
| SF | 150.9028090 | MHz |
| WDW | EM | |
| SSB | 0 | |
| LB | 3.00 | Hz |
| GB | 0 | |
| PC | 1.40 | |

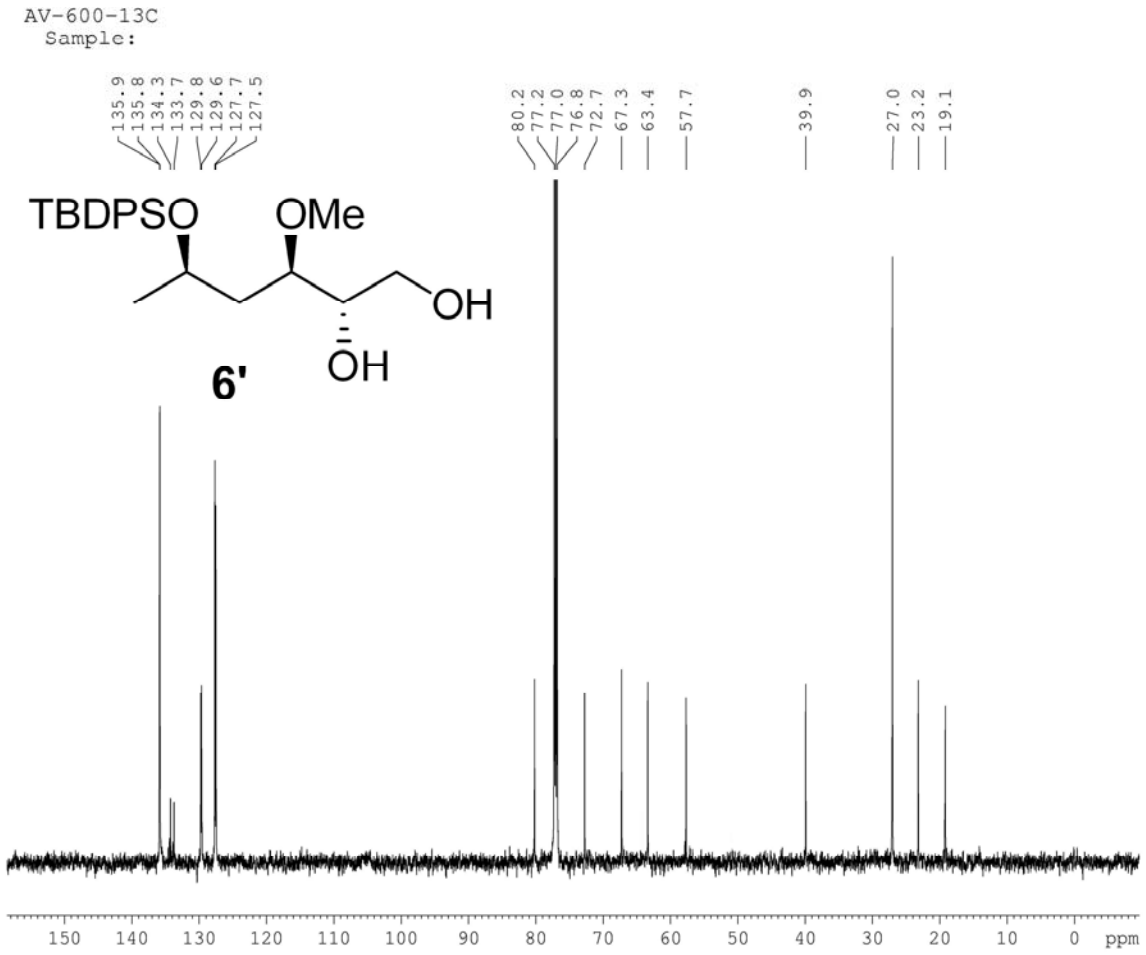

Sample:
7.725
7.720
7.713
7.707
7.700
7.694
7.689
7.670
7.664
7.657
7.400
7.392
7.378
7.366
4.055
4.034
4.014
3.994
3.543
3.539
3.531
3.448
3.443
3.436
3.425
3.420
3.408
3.394
3.384
3.369
3.234
3.218
1.810
1.762
1.722
1.650
1.624
1.601
1.578
1.108
1.088
1.051
0.000
BRUKER
TBDPSO
OMe
OH
OH
6
NAME SJ20120510
EXPNO 1
PROCNO 1
Date_ 20120511
Time 16.45
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 228.1
DW 81.000 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PL1 1.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
4.00
6.07
1.01
1.04
1.24
1.13
2.88
0.85
1.20
3.11
9.01
9
8
7
6
5
4
3
2
1
0
ppm



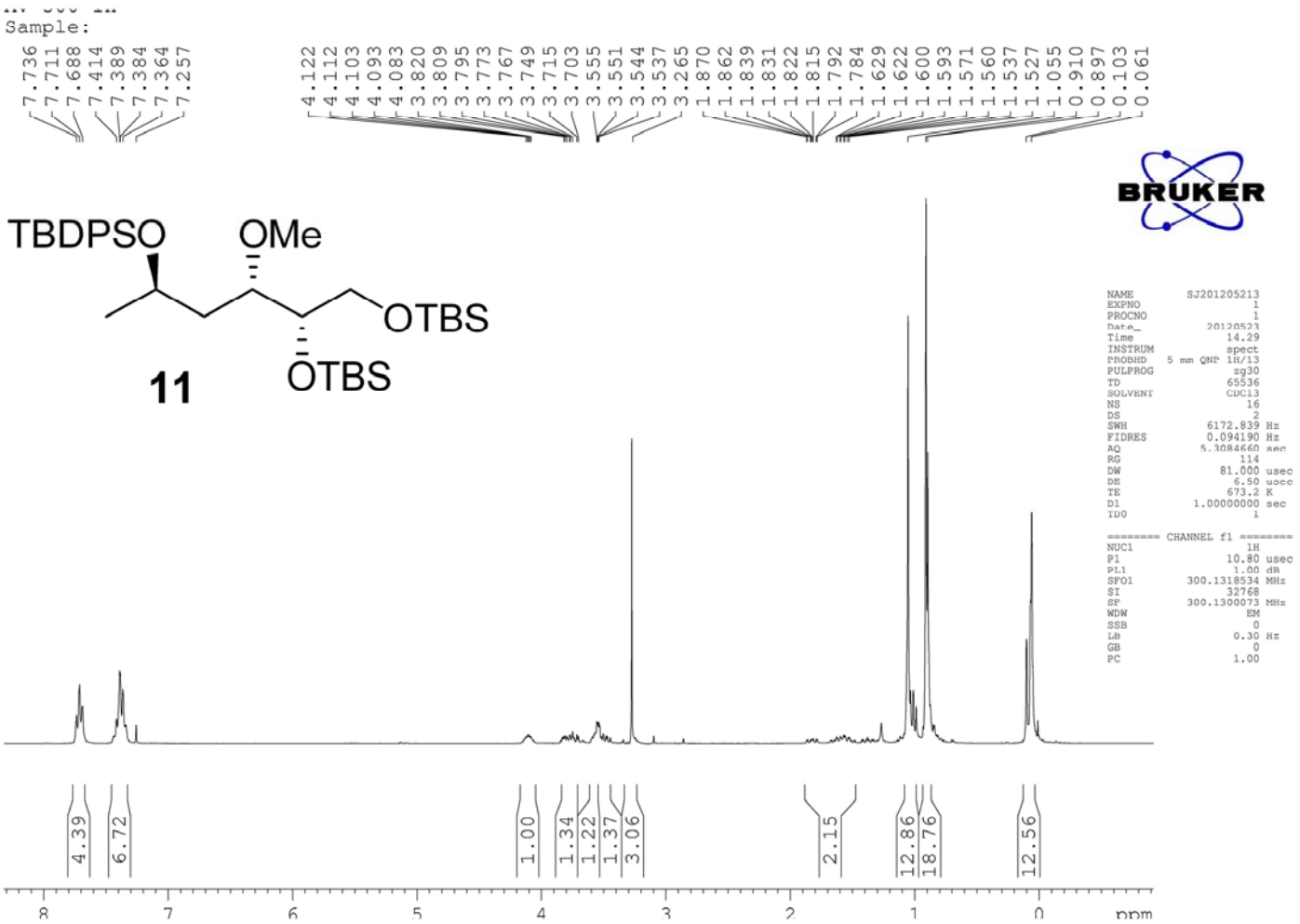

BRUKER

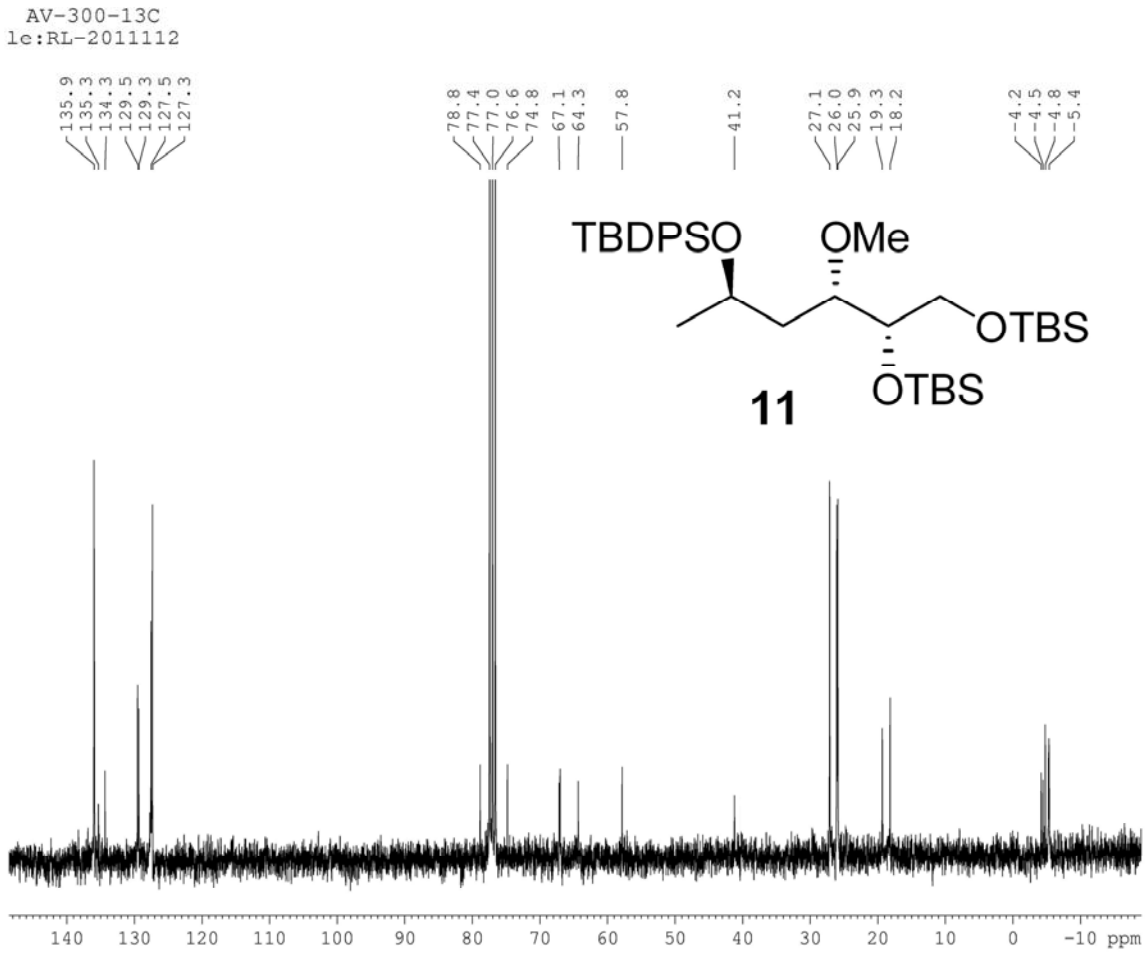
| | |
|---|---|
| NAME | SJ20120510 |
| EXPNO | 2 |
| PROCNO | 1 |
| Date_ | 20120522 |
| Time | 9.35 |
| INSTRUM | spect |
| PROBHD | 5 mm PABBO BB- |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 209 |
| DS | 2 |
| SWH | 45454.547 Hz |
| FIDRES | 0.693581 Hz |
| AQ | 0.7209570 sec |
| RG | 20600 |
| DW | 11.000 usec |
| DE | 6.50 usec |
| TE | 298.2 K |
| D1 | 2.00000000 sec |
| D11 | 0.03000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 6.00 usec |
| PL1 | 1.00 dB |
| PL1W | 83.20243835 W |
| SFO1 | 150.9178993 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | -4.00 dB |
| PL12 | 13.16 dB |
| PL13 | 16.00 dB |
| PL2W | 34.70265579 W |
| PL12W | 0.66736388 W |
| PL13W | 0.34702653 W |
| SFO2 | 600.1324005 MHz |
| SI | 32768 |
| SF | 150.9028090 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 3.00 Hz |
| GB | 0 |
| PC | 1.40 |


Sample:
7.695
7.690
7.678
7.430
7.399
7.375
7.357
4.078
4.059
4.039
4.019
3.630
3.622
3.462
3.382
3.368
3.351
3.287
1.863
1.844
1.834
1.815
1.796
1.649
1.629
1.609
1.601
1.581
1.561
1.113
1.058
1.051
0.001
BRUKER
TBDPSO
OMe
OH
OH
6'
NAME SJ2012-1023
EXPNO 1
PROCNO 1
Date_ 20121030
Time 8.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 256
DW 81.000 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PL1 1.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300074 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.24
5.70
1.00
1.96
1.41
0.91
2.93
1.27
1.01
1.75
8.41
8
7
6
5
4
3
2
1
0 ppm



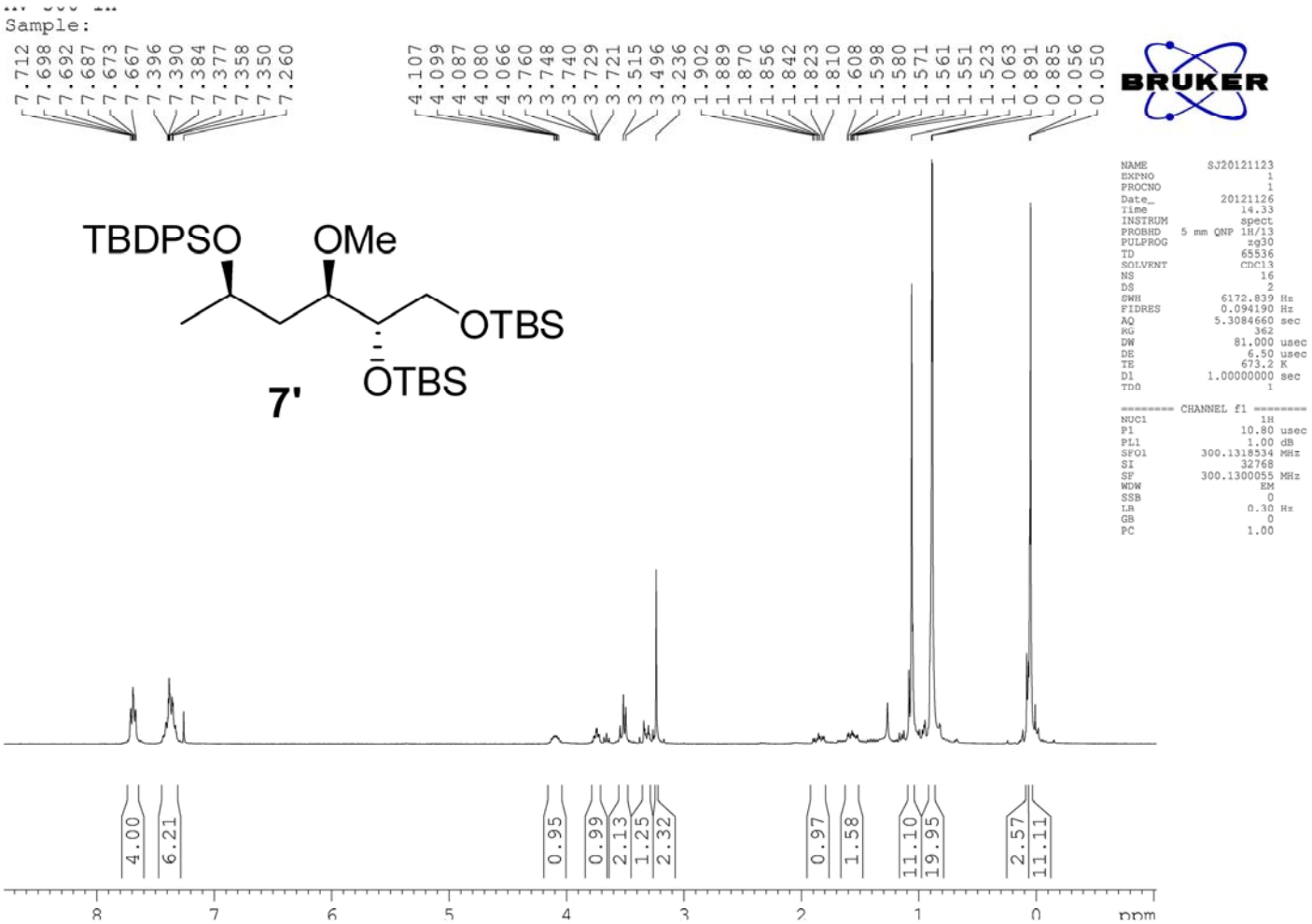

| | |
|---|---|
| NAME | SJ2012-1023 |
| EXPNO | 2 |
| PROCNO | 1 |
| Date_ | 20121030 |
| Time | 16.11 |
| INSTRUM | spect |
| PROBHD | 5 mm PABBO BB- |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 137 |
| DS | 2 |
| SWH | 45454.547 Hz |
| FIDRES | 0.693581 Hz |
| AQ | 0.7209570 sec |
| RG | 20600 |
| DW | 11.000 usec |
| DE | 6.50 usec |
| TE | 298.2 K |
| D1 | 2.00000000 sec |
| D11 | 0.03000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 6.00 usec |
| PL1 | 1.00 dB |
| PL1W | 83.20243835 W |
| SFO1 | 150.9178993 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | -4.00 dB |
| PL12 | 13.16 dB |
| PL13 | 16.00 dB |
| PL2W | 34.70265579 W |
| PL12W | 0.66736388 W |
| PL13W | 0.34702653 W |
| SFO2 | 600.1324005 MHz |
| SI | 32768 |
| SF | 150.9028090 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 3.00 Hz |
| GB | 0 |
| PC | 1.40 |

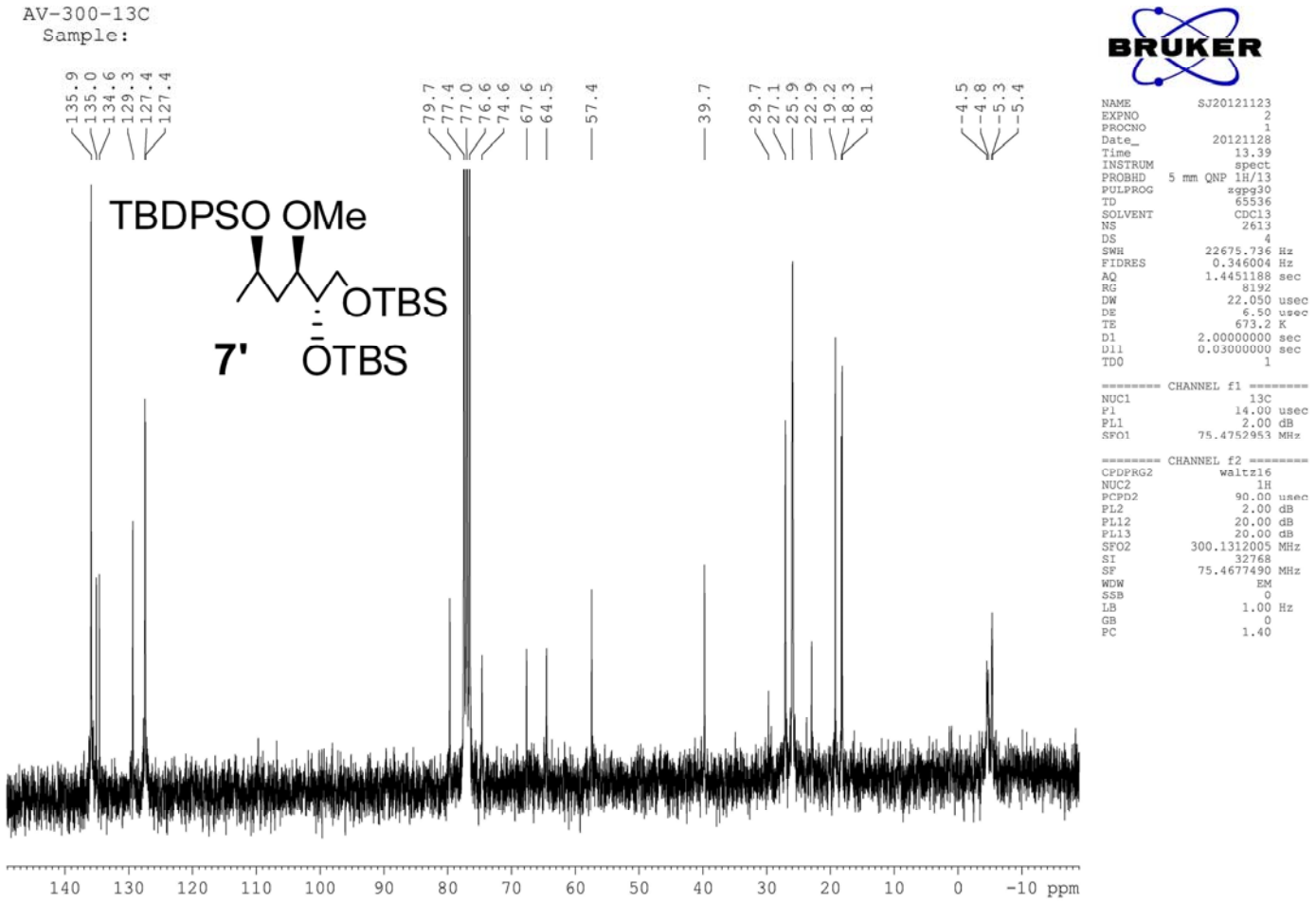

Sample:
7.736
7.711
7.688
7.414
7.389
7.384
7.364
7.257
4.122
4.112
4.103
4.093
4.083
3.820
3.809
3.795
3.773
3.767
3.749
3.715
3.703
3.555
3.551
3.544
3.537
3.265
1.870
1.862
1.839
1.831
1.822
1.815
1.792
1.784
1.629
1.622
1.600
1.593
1.571
1.560
1.537
1.527
1.055
0.910
0.897
0.103
0.061
TBDPSO
OMe
OTBS
OTBS
11
BRUKER
NAME SJ201205213
EXPNO 1
PROCNO 1
Date_ 20120523
Time 14.29
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 114
DW 81.000 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PL1 1.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300073 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
4.39
6.72
1.00
1.34
1.22
1.37
3.06
2.15
12.86
18.76
12.56
8
7
6
5
4
3
2
1
0
ppm




BRUKER

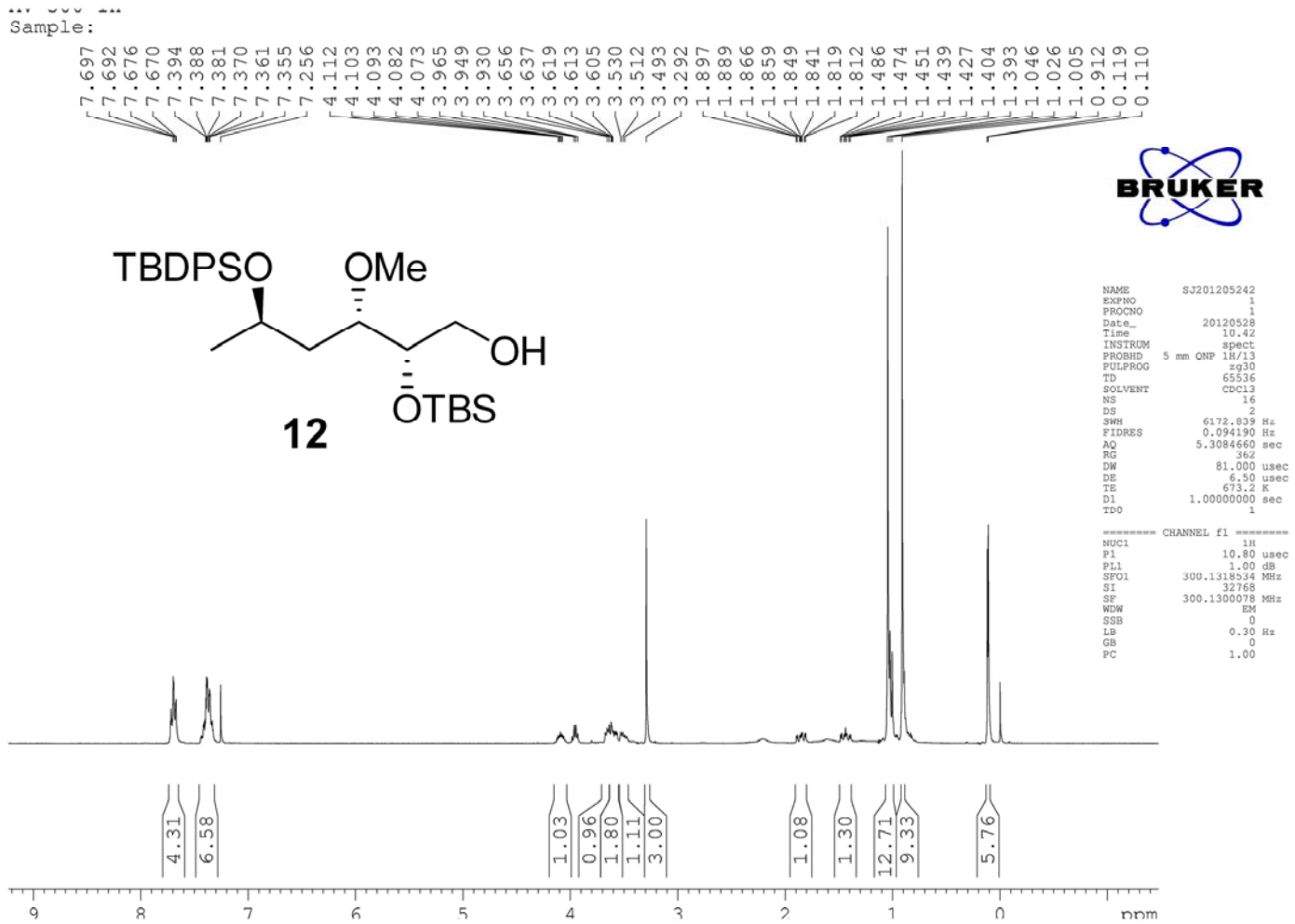
| | |
|---|---|
| NAME | SJ201205213 |
| EXPNO | 2 |
| PROCNO | 1 |
| Date_ | 20120529 |
| Time | 15.51 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 183 |
| DS | 4 |
| SWH | 22675.736 Hz |
| FIDRES | 0.346004 Hz |
| AQ | 1.4451188 sec |
| RG | 3649.1 |
| DW | 22.050 usec |
| DE | 6.50 usec |
| TE | 673.2 K |
| D1 | 2.00000000 sec |
| D11 | 0.03000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 14.00 usec |
| PL1 | 2.00 dB |
| SFO1 | 75.4752953 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 90.00 usec |
| PL2 | 2.00 dB |
| PL12 | 20.00 dB |
| PL13 | 20.00 dB |
| SFO2 | 300.1312005 MHz |
| SI | 32768 |
| SF | 75.4677490 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 1.40 |

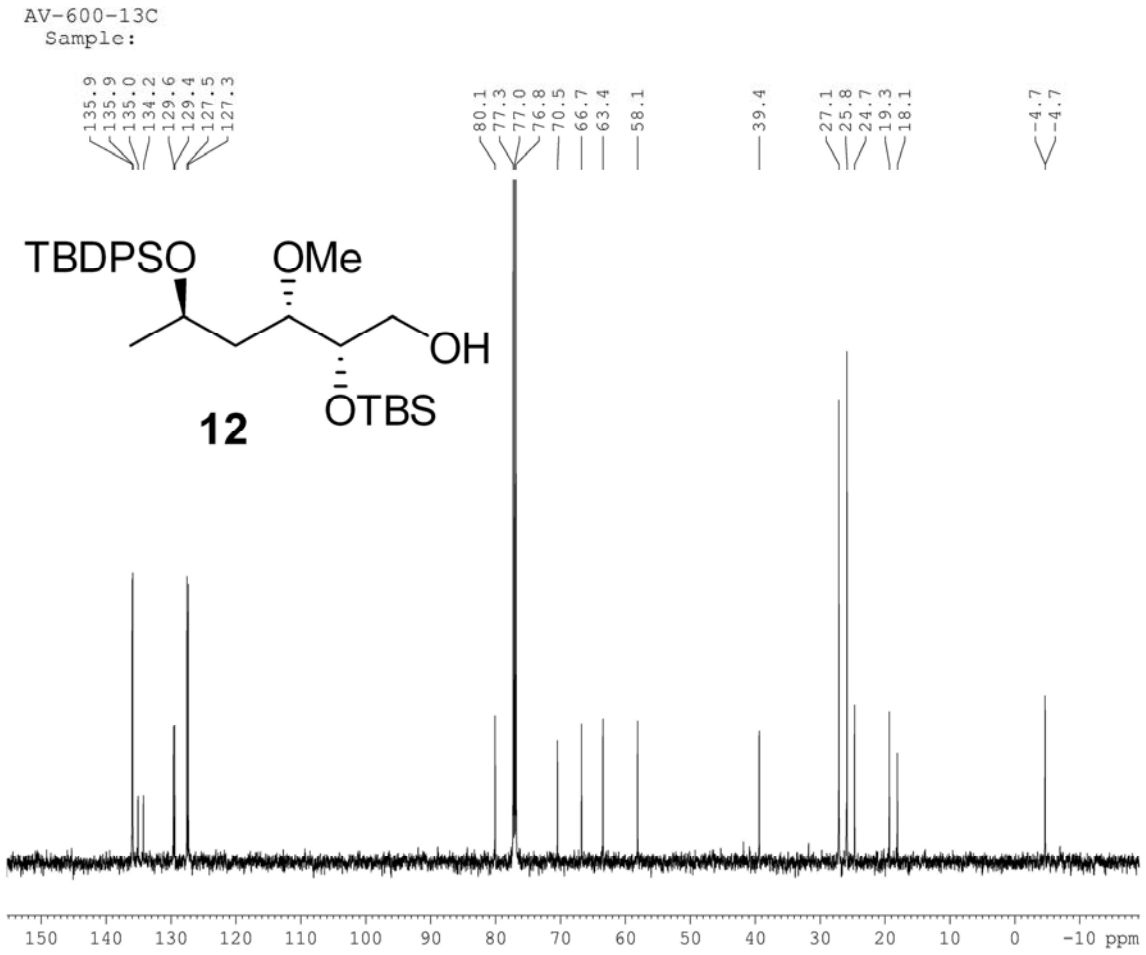

Sample:
7.712
7.698
7.692
7.687
7.673
7.667
7.396
7.390
7.384
7.377
7.358
7.350
7.260
4.107
4.099
4.087
4.080
4.066
3.760
3.748
3.740
3.729
3.721
3.515
3.496
3.236
1.902
1.889
1.870
1.856
1.842
1.823
1.810
1.608
1.598
1.580
1.571
1.561
1.551
1.523
1.063
0.891
0.885
0.056
0.050
BRUKER
TBDPSO
OMe
OTBS
OTBS
7'
NAME SJ20121123
EXPNO 1
PROCNO 1
Date_ 20121126
Time 14.33
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 362
DW 81.000 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PL1 1.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300055 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
4.00
6.21
0.95
0.99
2.13
1.25
2.32
0.97
1.58
11.10
19.95
2.57
11.11
8
7
6
5
4
3
2
1
0
ppm




BRUKER

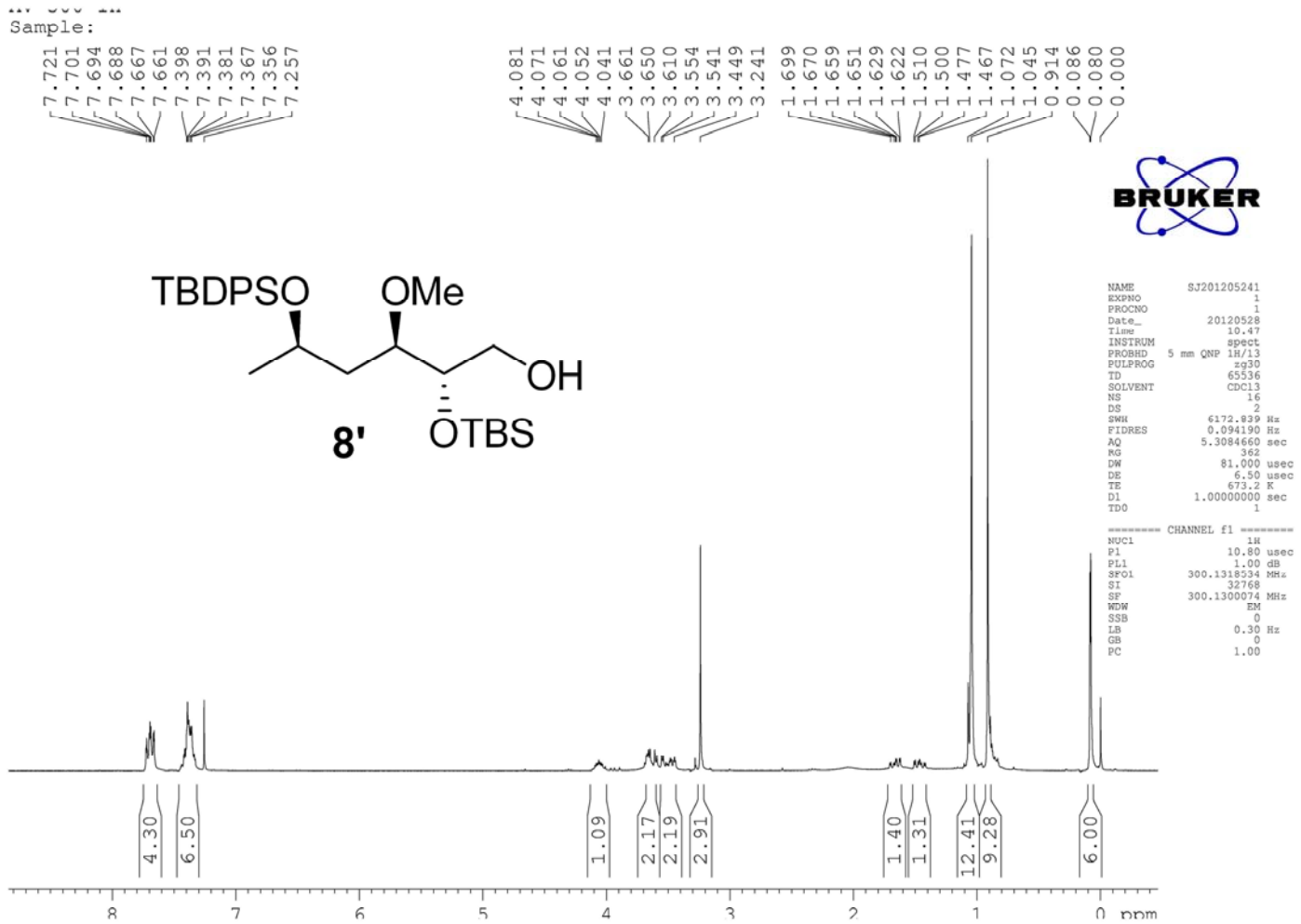
| | |
|---|---|
| NAME | SJ20121123 |
| EXPNO | 2 |
| PROCNO | 1 |
| Date_ | 20121128 |
| Time | 13.39 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 2613 |
| DS | 4 |
| SWH | 22675.736 Hz |
| FIDRES | 0.346004 Hz |
| AQ | 1.4451188 sec |
| RG | 8192 |
| DW | 22.050 usec |
| DE | 6.50 usec |
| TE | 673.2 K |
| D1 | 2.00000000 sec |
| D11 | 0.03000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 14.00 usec |
| PL1 | 2.00 dB |
| SFO1 | 75.4752953 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 90.00 usec |
| PL2 | 2.00 dB |
| PL12 | 20.00 dB |
| PL13 | 20.00 dB |
| SFO2 | 300.1312005 MHz |
| SI | 32768 |
| SF | 75.4677490 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 1.40 |

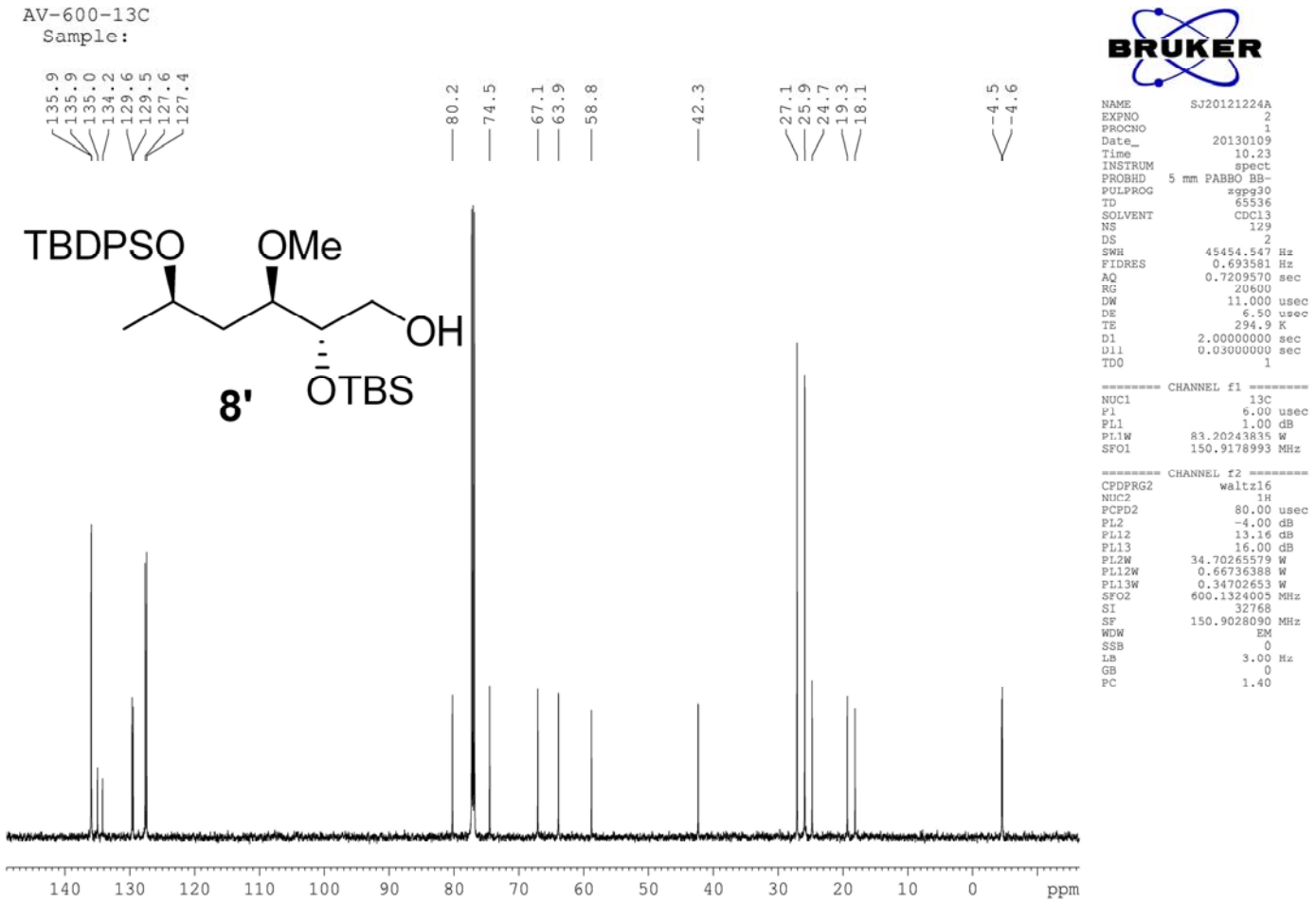

Sample:
7.697
7.692
7.676
7.670
7.394
7.388
7.381
7.370
7.361
7.355
7.256
4.112
4.103
4.093
4.082
4.073
3.965
3.949
3.930
3.656
3.637
3.619
3.613
3.605
3.530
3.512
3.493
3.292
1.897
1.889
1.866
1.859
1.849
1.841
1.819
1.812
1.486
1.474
1.451
1.439
1.427
1.404
1.393
1.046
1.026
1.005
0.912
0.119
0.110
BRUKER
TBDPSO
OMe
OH
OTBS
12
NAME SJ201205242
EXPNO 1
PROCNO 1
Date_ 20120528
Time 10.42
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 362
DW 81.000 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PL1 1.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300078 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
4.31
6.58
1.03
0.96
1.80
1.11
3.00
1.08
1.30
12.71
9.33
5.76
9
8
7
6
5
4
3
2
1
0
ppm




BRUKER

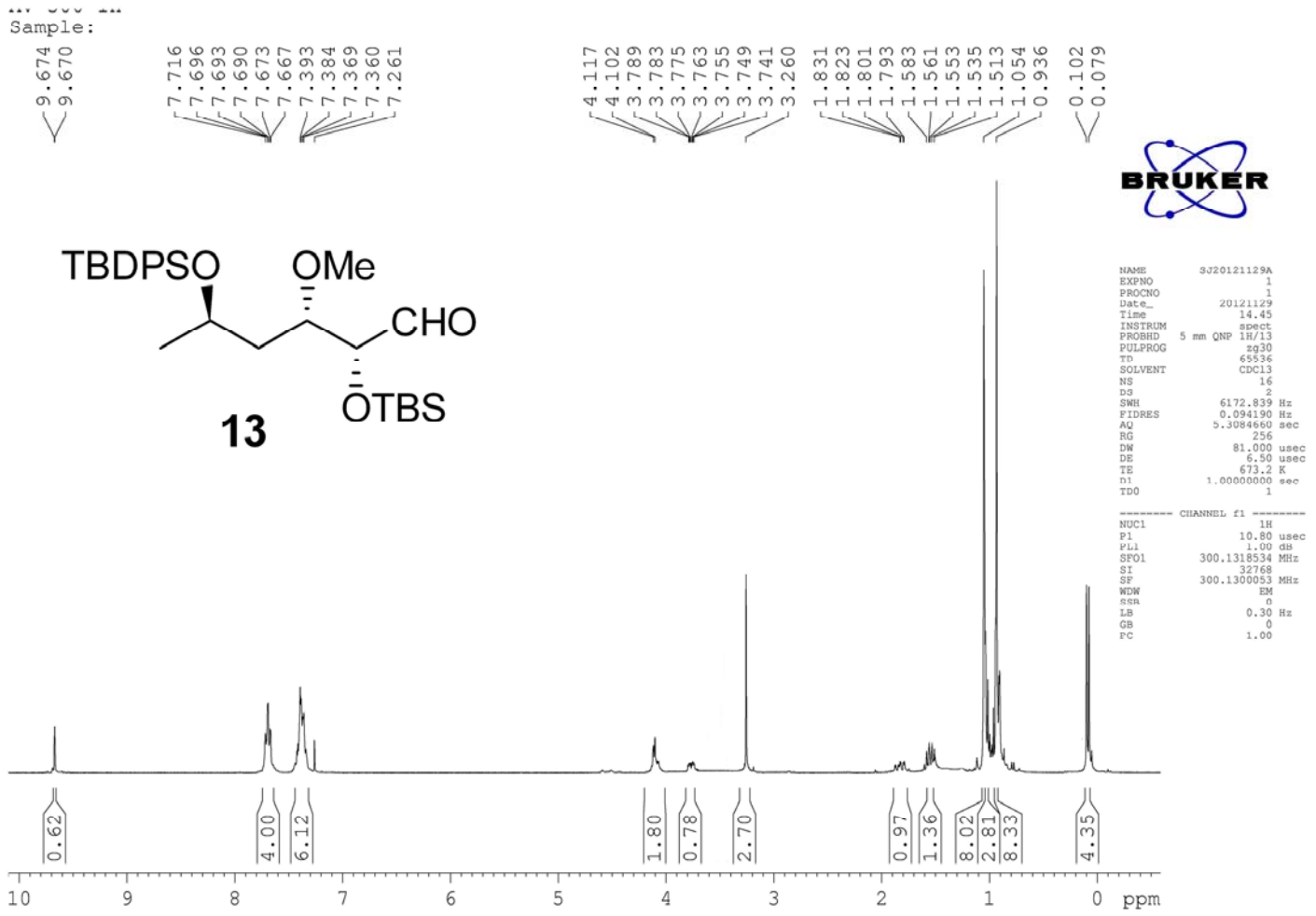
| | | |
|---|---|---|
| NAME | SJ20121224B | |
| EXPNO | 2 | |
| PROCNO | 1 | |
| Date_ | 20130109 | |
| Time | 10.27 | |
| INSTRUM | spect | |
| PROBHD | 5 mm PABBO BB- | |
| PULPROG | zgpg30 | |
| TD | 65536 | |
| SOLVENT | CDCl3 | |
| NS | 40 | |
| DS | 2 | |
| SWH | 45454.547 | Hz |
| FIDRES | 0.693581 | Hz |
| AQ | 0.7209570 | sec |
| RG | 20600 | |
| DW | 11.000 | usec |
| DE | 6.50 | usec |
| TE | 294.3 | K |
| D1 | 2.00000000 | sec |
| D11 | 0.03000000 | sec |
| TD0 | 1 | |
| ======== CHANNEL f1 ======== | | |
| NUC1 | 13C | |
| P1 | 6.00 | usec |
| PL1 | 1.00 | dB |
| PL1W | 83.20243835 | W |
| SFO1 | 150.9178993 | MHz |
| ======== CHANNEL f2 ======== | | |
| CPDPRG2 | waltz16 | |
| NUC2 | 1H | |
| PCPD2 | 80.00 | usec |
| PL2 | -4.00 | dB |
| PL12 | 13.16 | dB |
| PL13 | 16.00 | dB |
| PL2W | 34.70265579 | W |
| PL12W | 0.66736388 | W |
| PL13W | 0.34702653 | W |
| SFO2 | 600.1324005 | MHz |
| SI | 32768 | |
| SF | 150.9028090 | MHz |
| WDW | EM | |
| SSB | 0 | |
| LB | 3.00 | Hz |
| GB | 0 | |
| PC | 1.40 | |

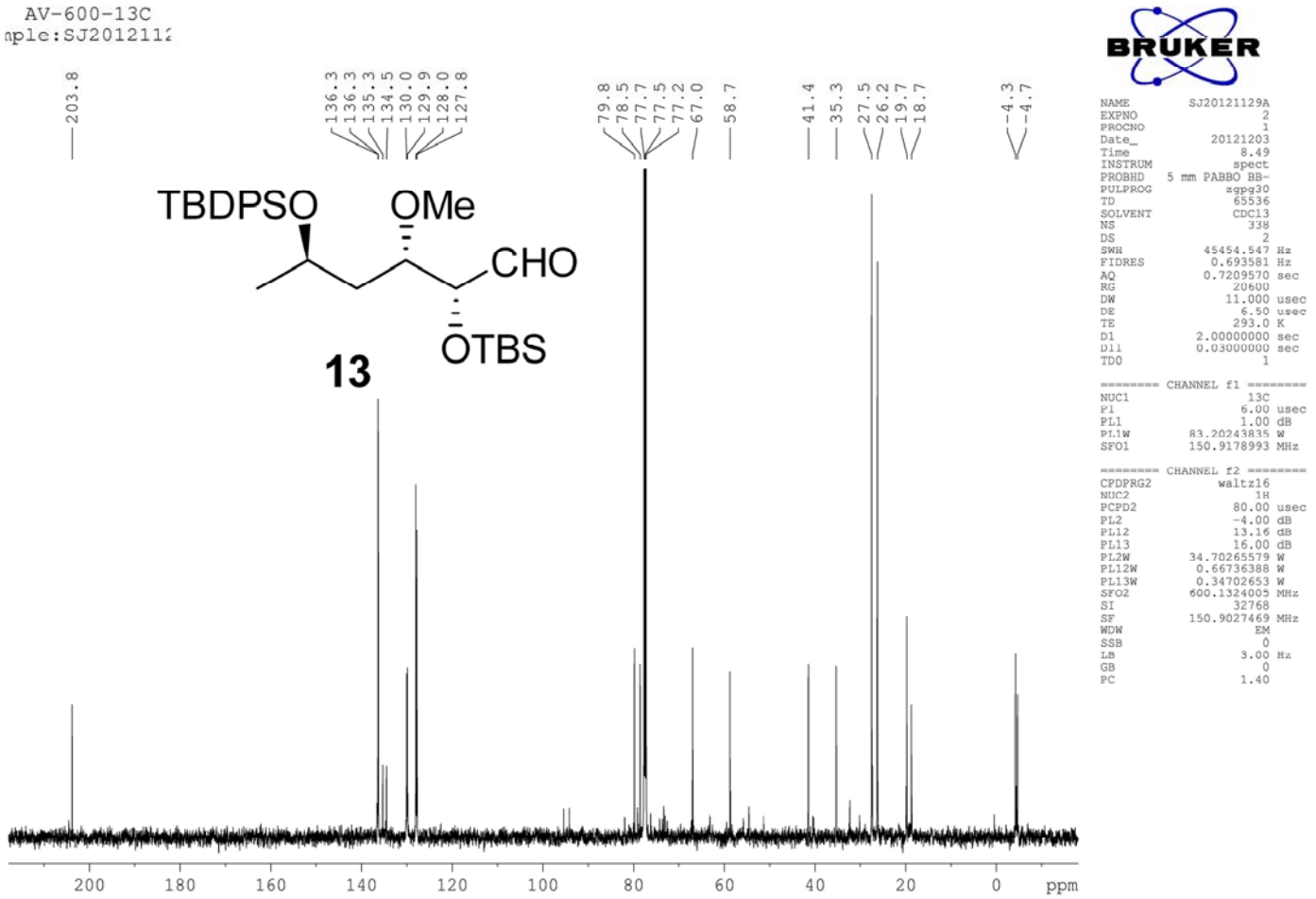

Sample:
7.721
7.701
7.694
7.688
7.667
7.661
7.398
7.391
7.381
7.367
7.356
7.257
4.081
4.071
4.061
4.052
4.041
3.661
3.650
3.610
3.554
3.541
3.449
3.241
1.699
1.670
1.659
1.651
1.629
1.622
1.510
1.500
1.477
1.467
1.072
1.045
0.914
0.086
0.080
0.000
TBDPSO
OMe
OH
OTBS
8'
BRUKER
NAME SJ201205241
EXPNO 1
PROCNO 1
Date_ 20120528
Time 10.47
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 362
DW 81.000 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PL1 1.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300074 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
4.30
6.50
1.09
2.17
2.19
2.91
1.40
1.31
12.41
9.28
6.00
8
7
6
5
4
3
2
1
0 ppm




BRUKER

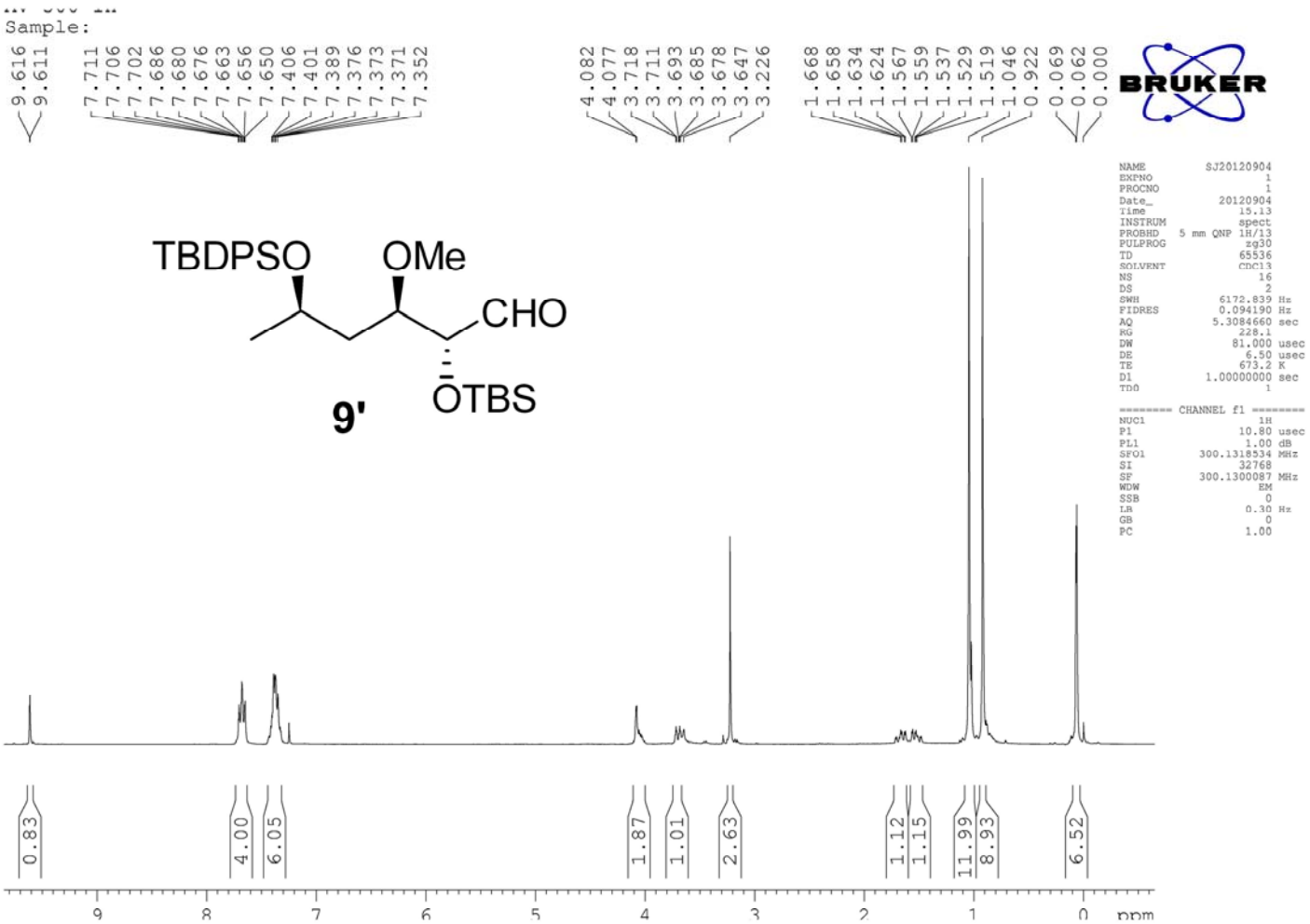
| | |
|---|---|
| NAME | SJ20121224A |
| EXPNO | 2 |
| PROCNO | 1 |
| Date_ | 20130109 |
| Time | 10.23 |
| INSTRUM | spect |
| PROBHD | 5 mm PABBO BB- |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 129 |
| DS | 2 |
| SWH | 45454.547 Hz |
| FIDRES | 0.693581 Hz |
| AQ | 0.7209570 sec |
| RG | 20600 |
| DW | 11.000 usec |
| DE | 6.50 usec |
| TE | 294.9 K |
| D1 | 2.00000000 sec |
| D11 | 0.03000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 6.00 usec |
| PL1 | 1.00 dB |
| PL1W | 83.20243835 W |
| SFO1 | 150.9178993 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | -4.00 dB |
| PL12 | 13.16 dB |
| PL13 | 16.00 dB |
| PL2W | 34.70265579 W |
| PL12W | 0.66736388 W |
| PL13W | 0.34702653 W |
| SFO2 | 600.1324005 MHz |
| SI | 32768 |
| SF | 150.9028090 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 3.00 Hz |
| GB | 0 |
| PC | 1.40 |

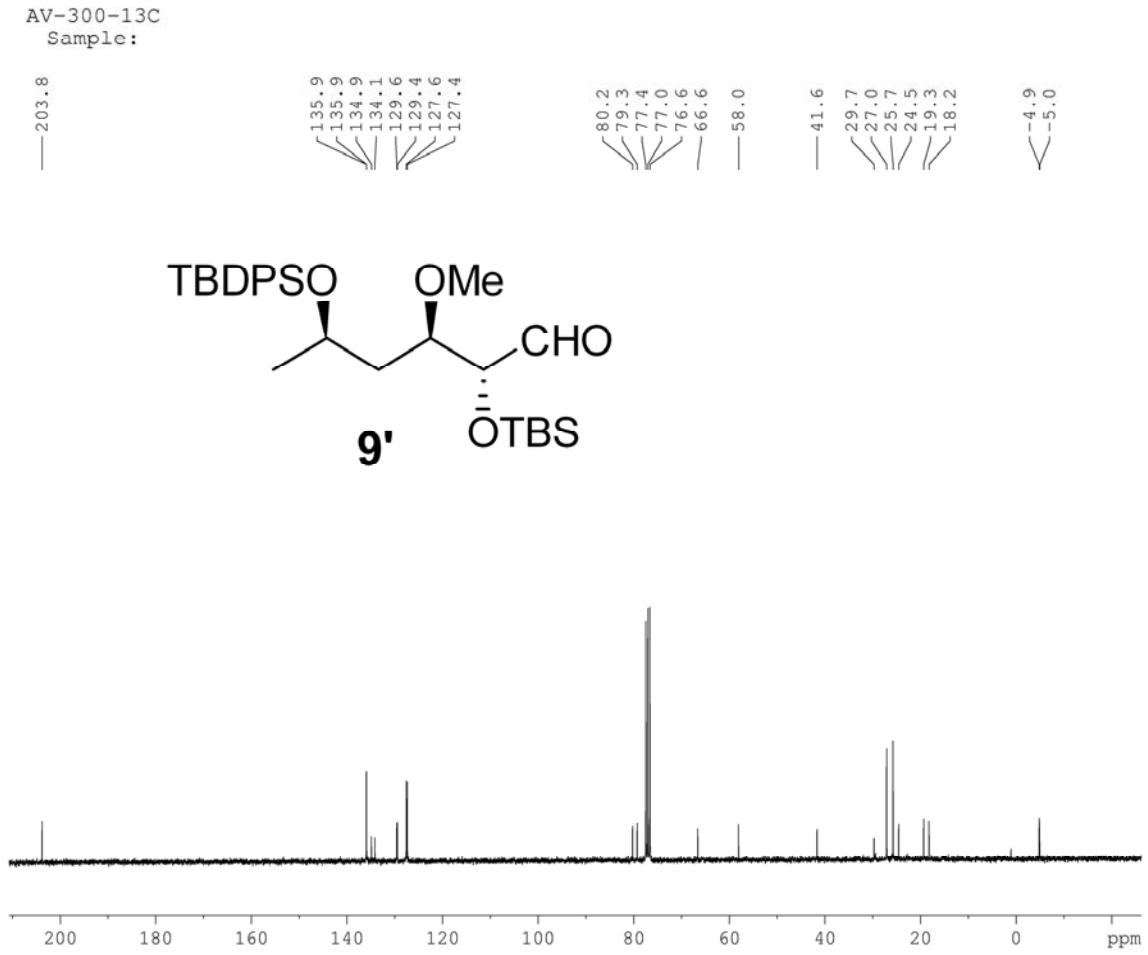

Sample:
9.674
9.670
7.716
7.696
7.693
7.690
7.673
7.667
7.393
7.384
7.369
7.360
7.261
4.117
4.102
3.789
3.783
3.775
3.763
3.755
3.749
3.741
3.260
1.831
1.823
1.801
1.793
1.583
1.561
1.553
1.535
1.513
1.054
0.936
0.102
0.079
TBDPSO
OMe
CHO
OTBS
13
BRUKER
NAME 3J20121129A
EXPNO 1
PROCNO 1
Date_ 20121129
Time 14.45
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 256
DW 81.000 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PL1 1.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300053 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.62
4.00
6.12
1.80
0.78
2.70
0.97
1.36
8.02
2.81
8.33
4.35
10
9
8
7
6
5
4
3
2
1
0
ppm




BRUKER

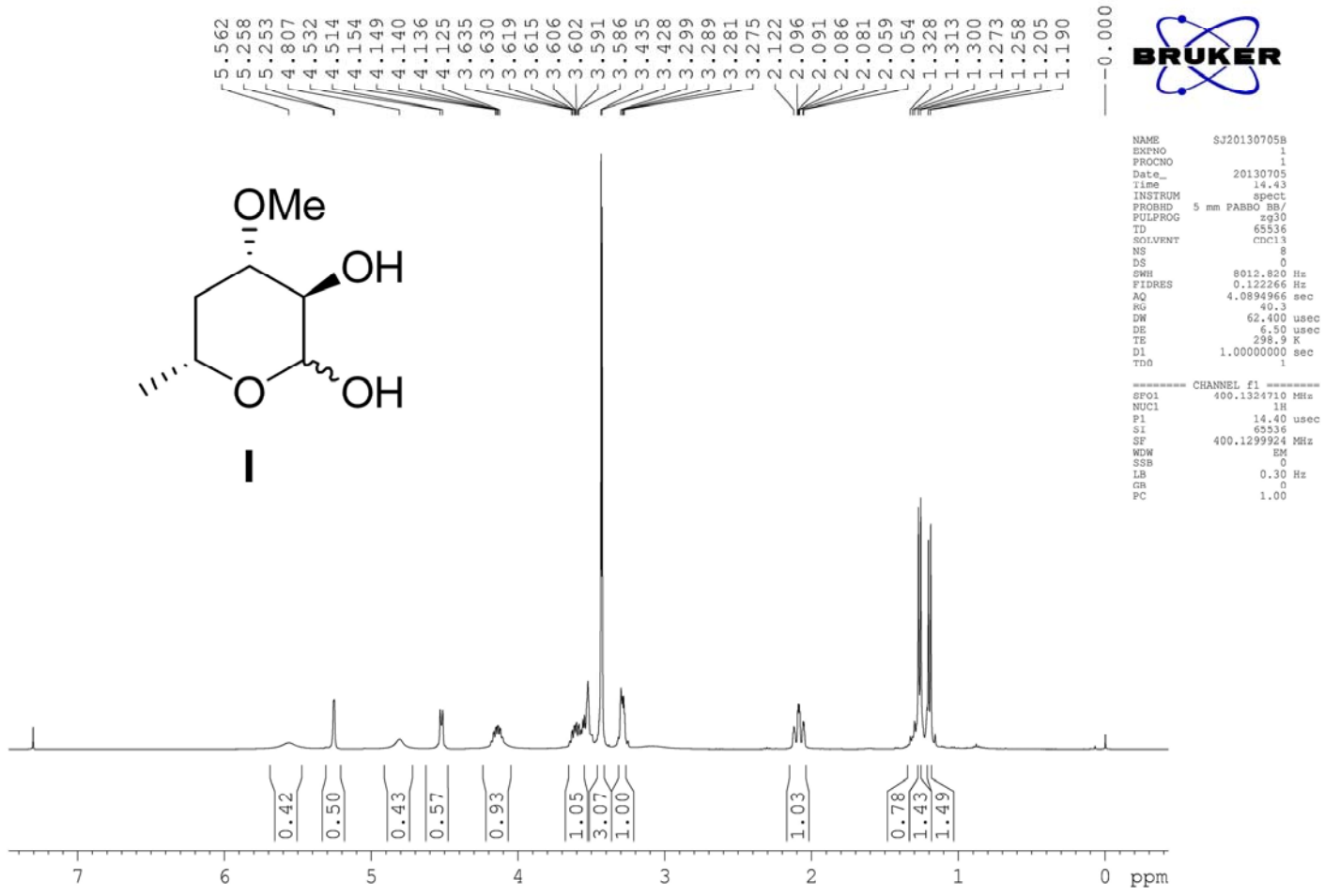
| | |
|---|---|
| NAME | SJ20121129A |
| EXPNO | 2 |
| PROCNO | 1 |
| Date_ | 20121203 |
| Time | 8.49 |
| INSTRUM | spect |
| PROBHD | 5 mm PABBO BB- |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 338 |
| DS | 2 |
| SWH | 45454.547 Hz |
| FIDRES | 0.693581 Hz |
| AQ | 0.7209570 sec |
| RG | 20600 |
| DW | 11.000 usec |
| DE | 6.50 usec |
| TE | 293.0 K |
| D1 | 2.00000000 sec |
| D11 | 0.03000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 6.00 usec |
| PL1 | 1.00 dB |
| PL1W | 83.20243835 W |
| SFO1 | 150.9178993 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | -4.00 dB |
| PL12 | 13.16 dB |
| PL13 | 16.00 dB |
| PL2W | 34.70265579 W |
| PL12W | 0.66736388 W |
| PL13W | 0.34702653 W |
| SFO2 | 600.1324005 MHz |
| SI | 32768 |
| SF | 150.9027469 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 3.00 Hz |
| GB | 0 |
| PC | 1.40 |


Sample:
9.616
9.611
7.711
7.706
7.702
7.686
7.680
7.676
7.663
7.656
7.650
7.406
7.401
7.389
7.376
7.373
7.371
7.352
4.082
4.077
3.718
3.711
3.693
3.685
3.678
3.647
3.226
1.668
1.658
1.634
1.624
1.567
1.559
1.537
1.529
1.519
1.046
0.922
0.069
0.062
0.000
BRUKER
TBDPSO
OMe
CHO
OTBS
9'
NAME SJ20120904
EXPNO 1
PROCNO 1
Date_ 20120904
Time 15.13
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 228.1
DW 81.000 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PL1 1.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.83
4.00
6.05
1.87
1.01
2.63
1.12
1.15
11.99
8.93
6.52
9
8
7
6
5
4
3
2
1
0
ppm




BRUKER

| | |
|---|---|
| NAME | SJ20120904 |
| EXPNO | 2 |
| PROCNO | 1 |
| Date_ | 20120904 |
| Time | 17.42 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 720 |
| DS | 4 |
| SWH | 22675.736 Hz |
| FIDRES | 0.346004 Hz |
| AQ | 1.4451188 sec |
| RG | 2896.3 |
| DW | 22.050 usec |
| DE | 6.50 usec |
| TE | 673.2 K |
| D1 | 2.00000000 sec |
| D11 | 0.03000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 14.00 usec |
| PL1 | 2.00 dB |
| SFO1 | 75.4752953 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 90.00 usec |
| PL2 | 2.00 dB |
| PL12 | 20.00 dB |
| PL13 | 20.00 dB |
| SFO2 | 300.1312005 MHz |
| SI | 32768 |
| SF | 75.4677490 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 1.40 |


5.562
5.258
5.253
4.807
4.532
4.514
4.154
4.149
4.140
4.136
4.125
3.635
3.630
3.619
3.615
3.606
3.602
3.591
3.586
3.435
3.428
3.299
3.289
3.281
3.275
2.122
2.096
2.091
2.086
2.081
2.059
2.054
1.328
1.313
1.300
1.273
1.258
1.205
1.190
0.000
BRUKER
OMe
OH
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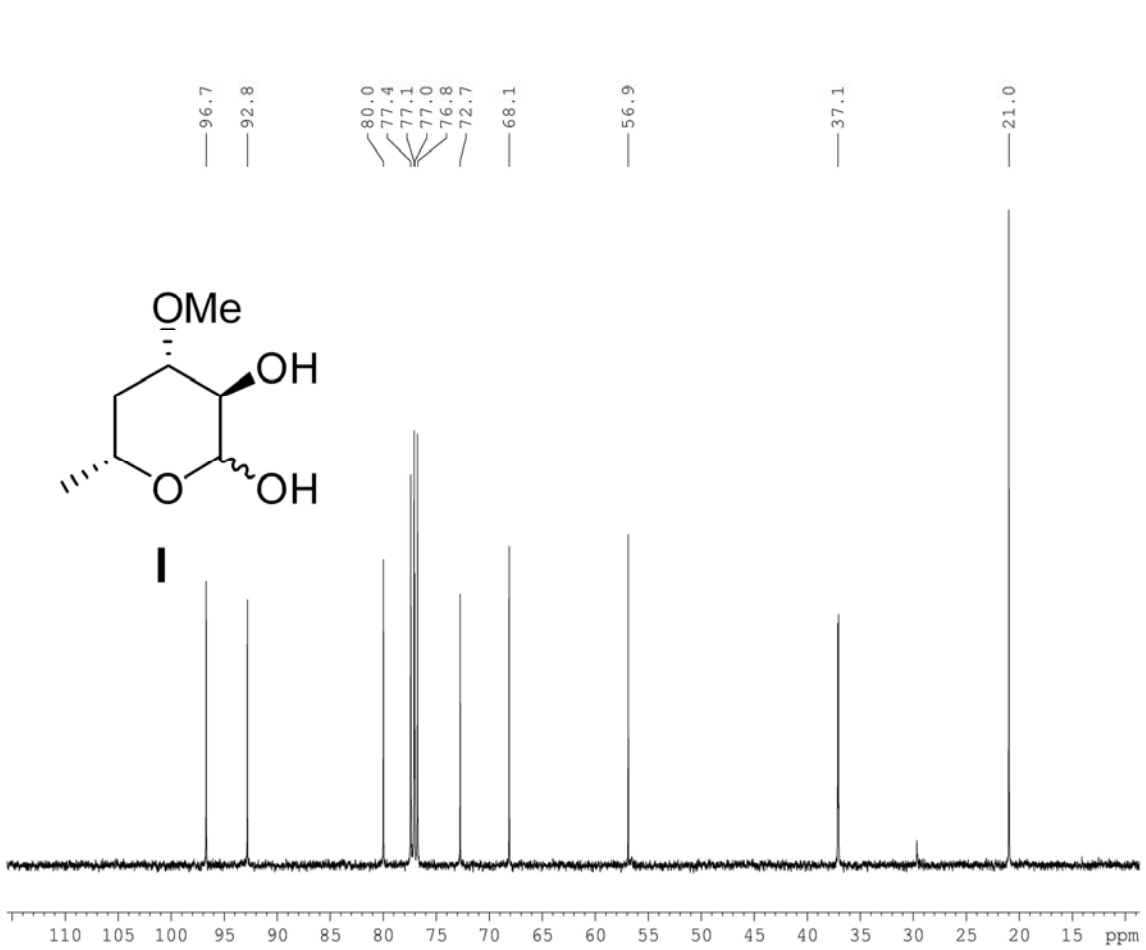
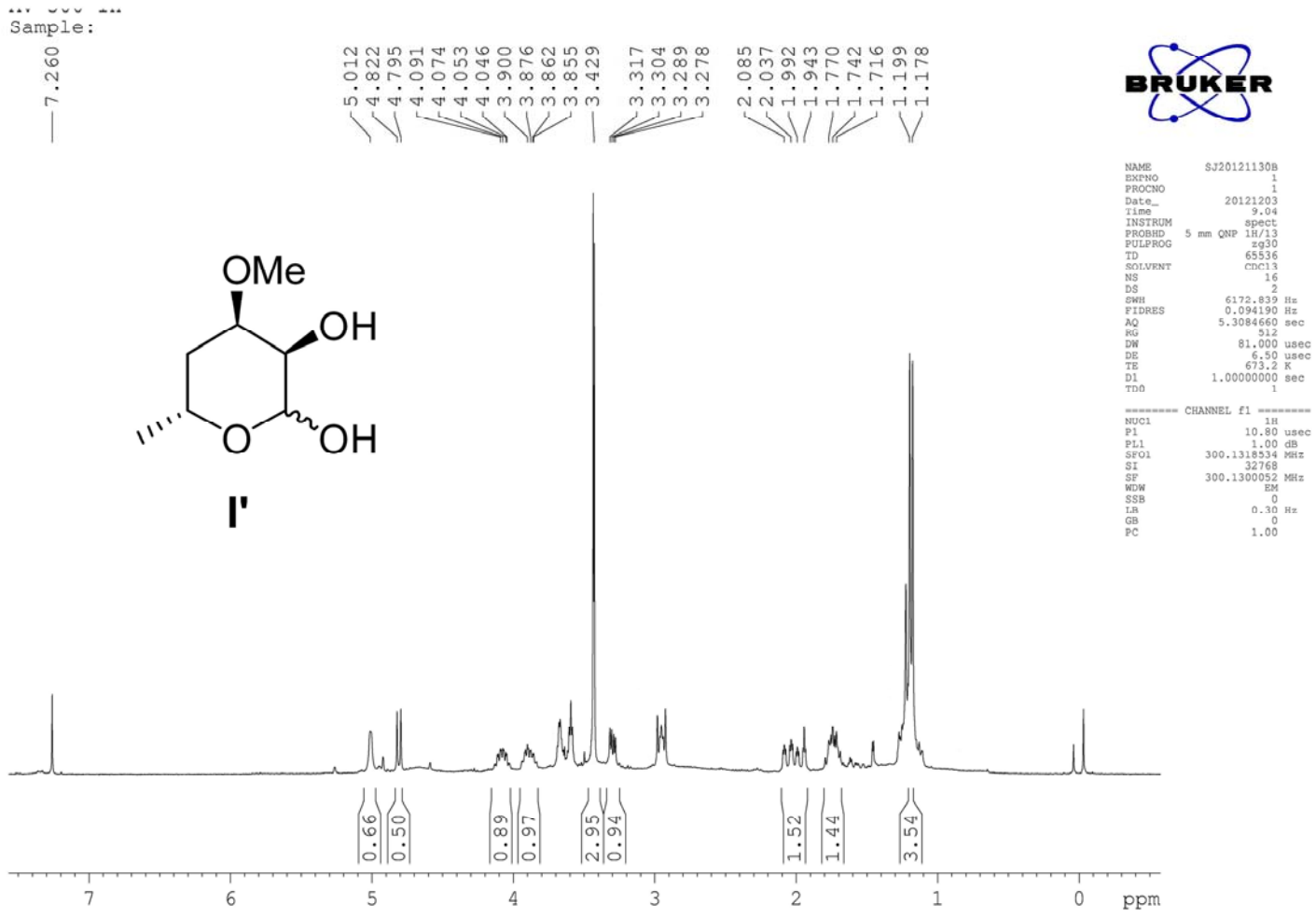
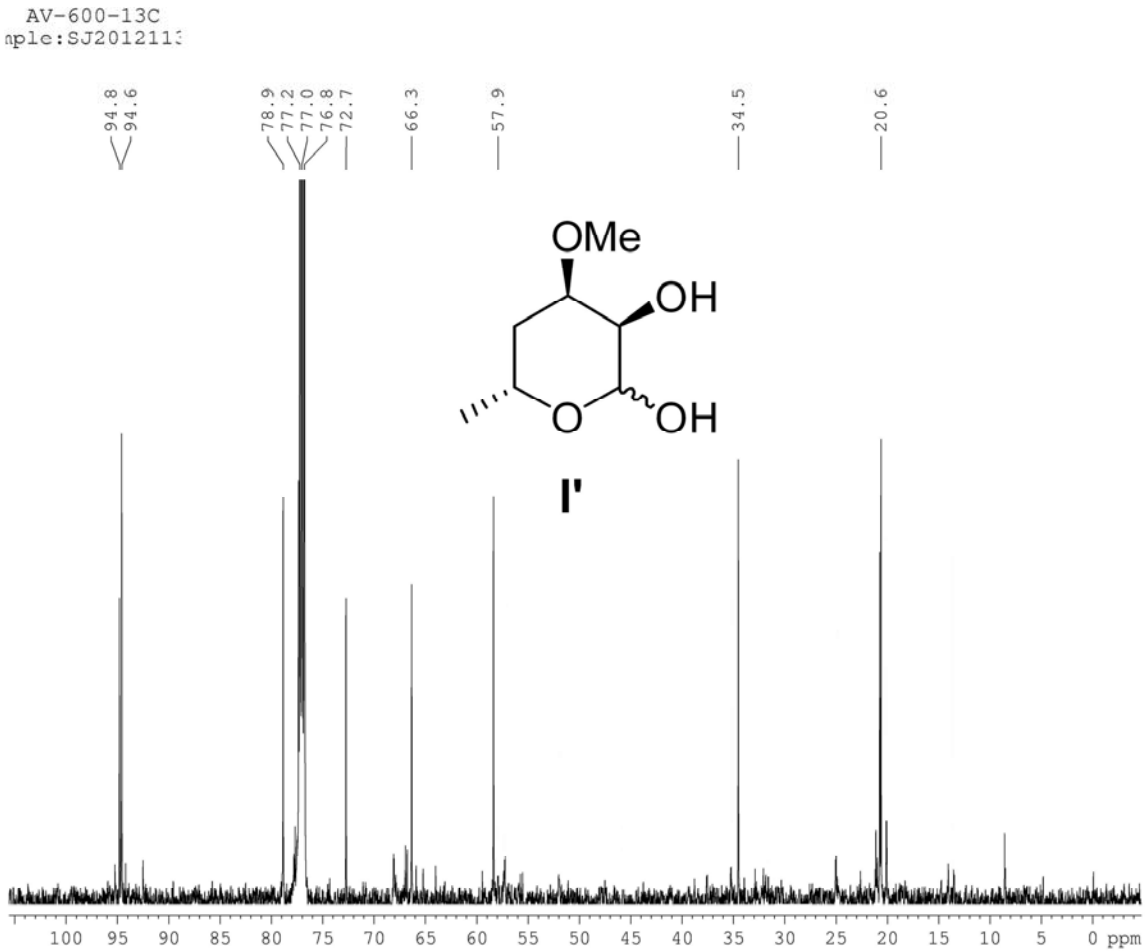
I
NAME SJ20130705B
EXPNO 1
PROCNO 1
Date_ 20130705
Time 14.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 40.3
DW 62.400 usec
DE 6.50 usec
TE 298.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.40 usec
SI 65536
SF 400.1299924 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.42
0.50
0.43
0.57
0.93
1.05
3.07
1.00
1.03
0.78
1.43
1.49
7
6
5
4
3
2
1
0
ppm



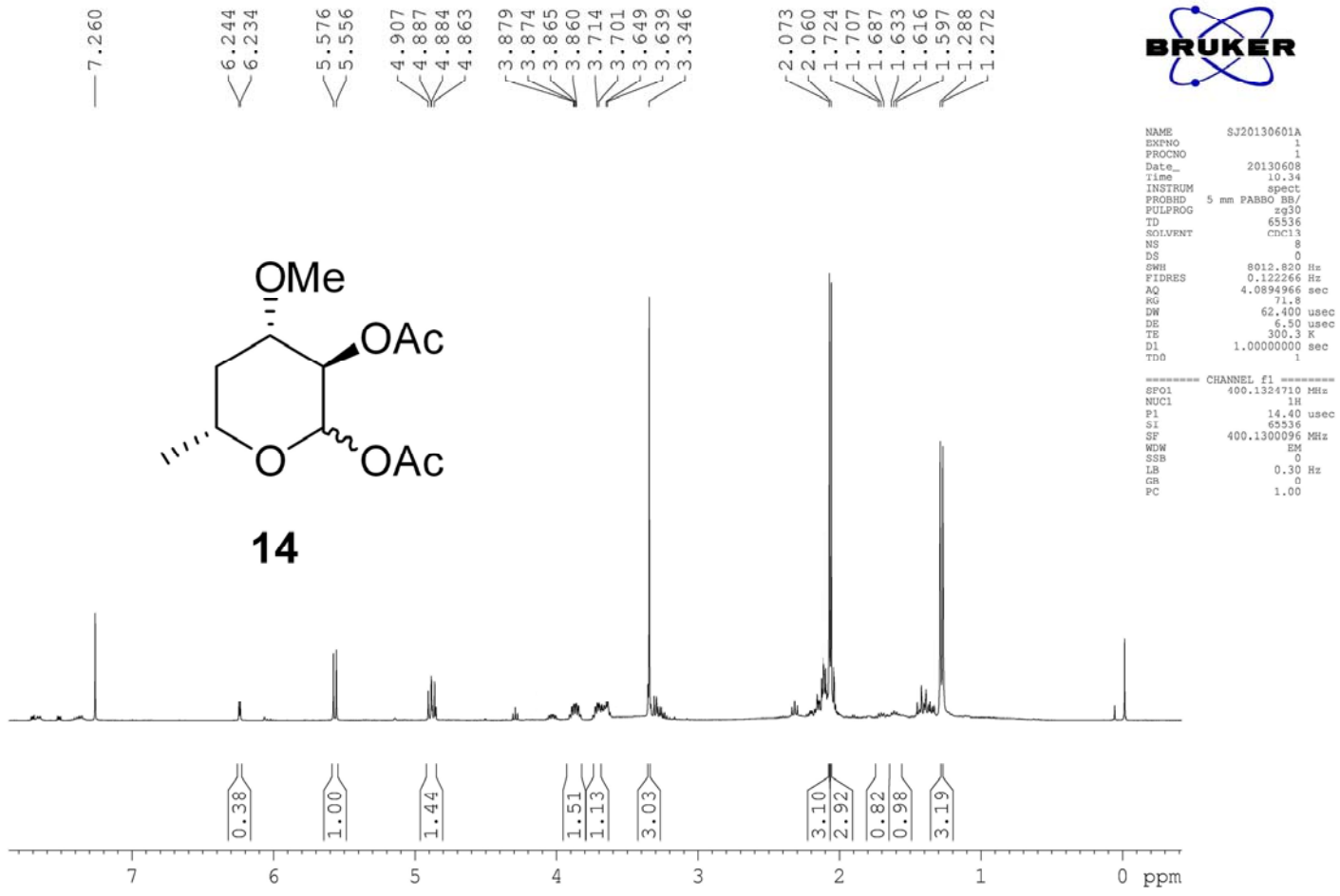

BRUKER

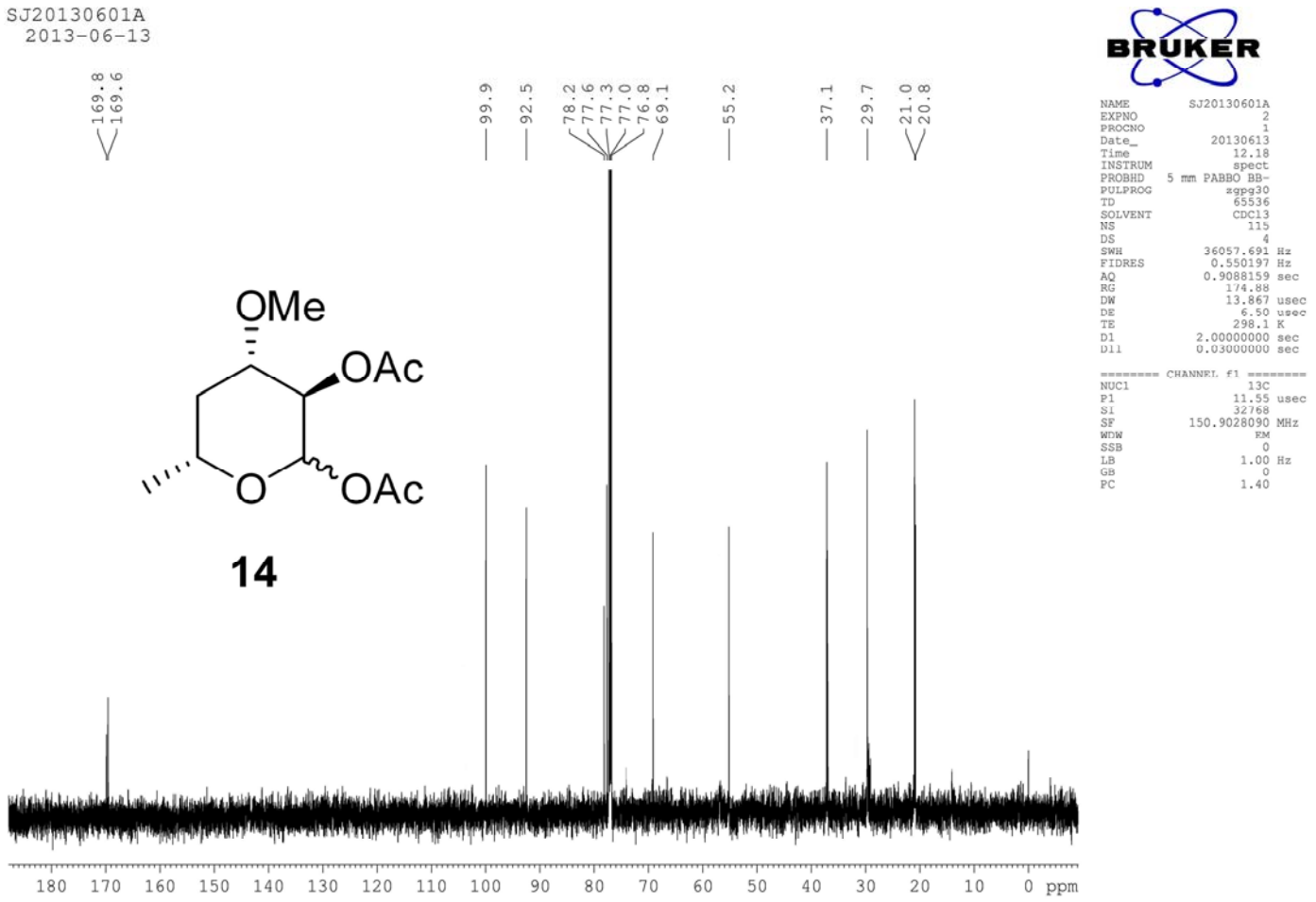
| | |
|---|---|
| NAME | SJ20130705B |
| EXPNO | 2 |
| PROCNO | 1 |
| Date_ | 20130705 |
| Time | 16.20 |
| INSTRUM | spect |
| PROBHD | 5 mm PABBO BB/ |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 118 |
| DS | 4 |
| SWH | 29761.904 Hz |
| FIDRES | 0.454131 Hz |
| AQ | 1.1010548 sec |
| RG | 203 |
| DW | 16.800 usec |
| DE | 6.50 usec |
| TE | 299.9 K |
| D1 | 2.00000000 sec |
| D11 | 0.03000000 sec |
| TD0 | 1000000 |
| ======== | CHANNEL f1 ======== |
| SFO1 | 100.6228293 MHz |
| NUC1 | 13C |
| P1 | 9.40 usec |
| ST | 32768 |
| SF | 100.6127690 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 1.40 |

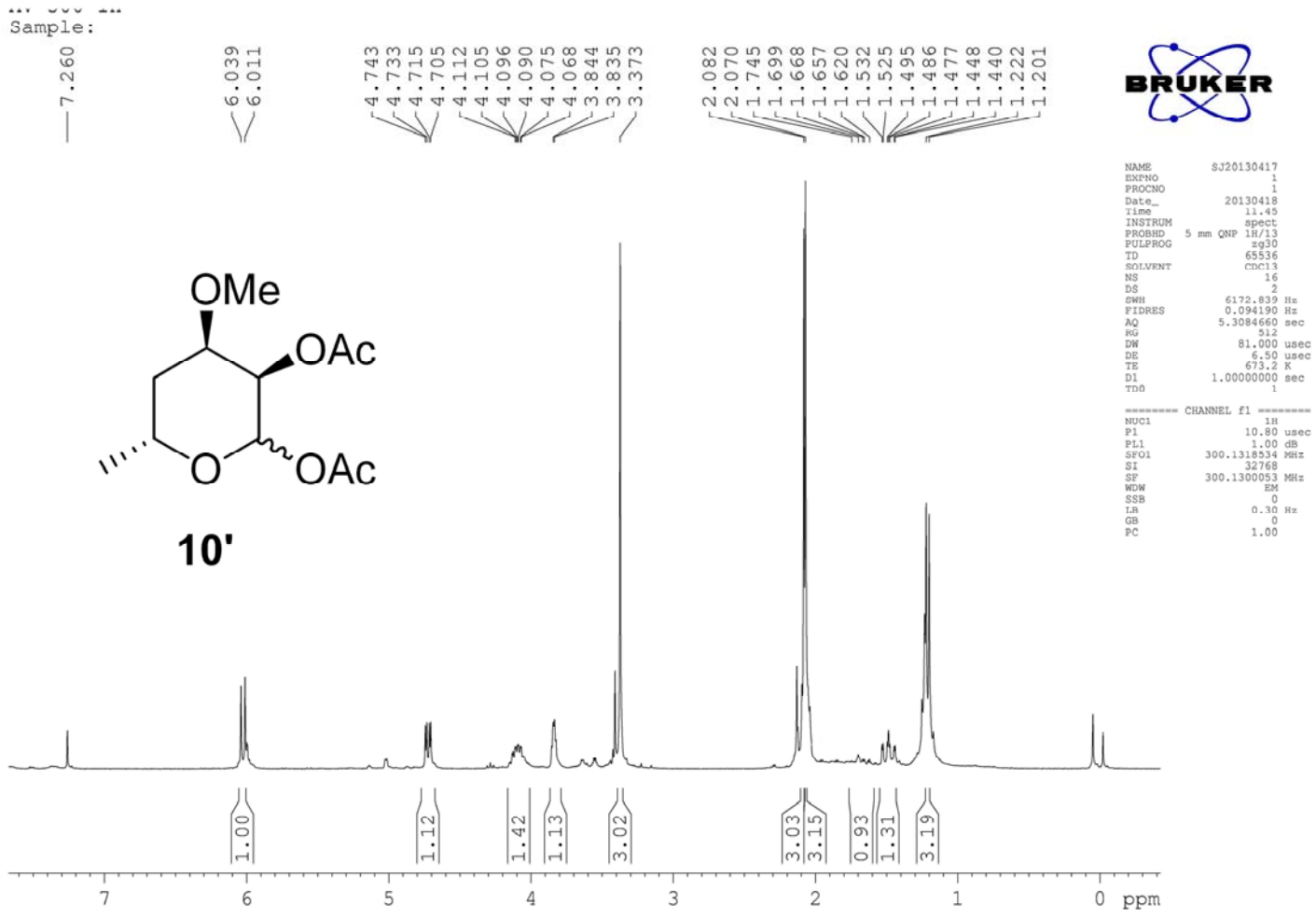

Sample:
7.260
5.012
4.822
4.795
4.091
4.074
4.053
4.046
3.900
3.876
3.862
3.855
3.429
3.317
3.304
3.289
3.278
2.085
2.037
1.992
1.943
1.770
1.742
1.716
1.199
1.178
BRUKER
OMe
OH
OH
O
I'
NAME SJ20121130B
EXPNO 1
PROCNO 1
Date_ 20121203
Time 9.04
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 512
DW 81.000 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PL1 1.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300052 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.66
0.50
0.89
0.97
2.95
0.94
1.52
1.44
3.54
7
6
5
4
3
2
1
0
ppm

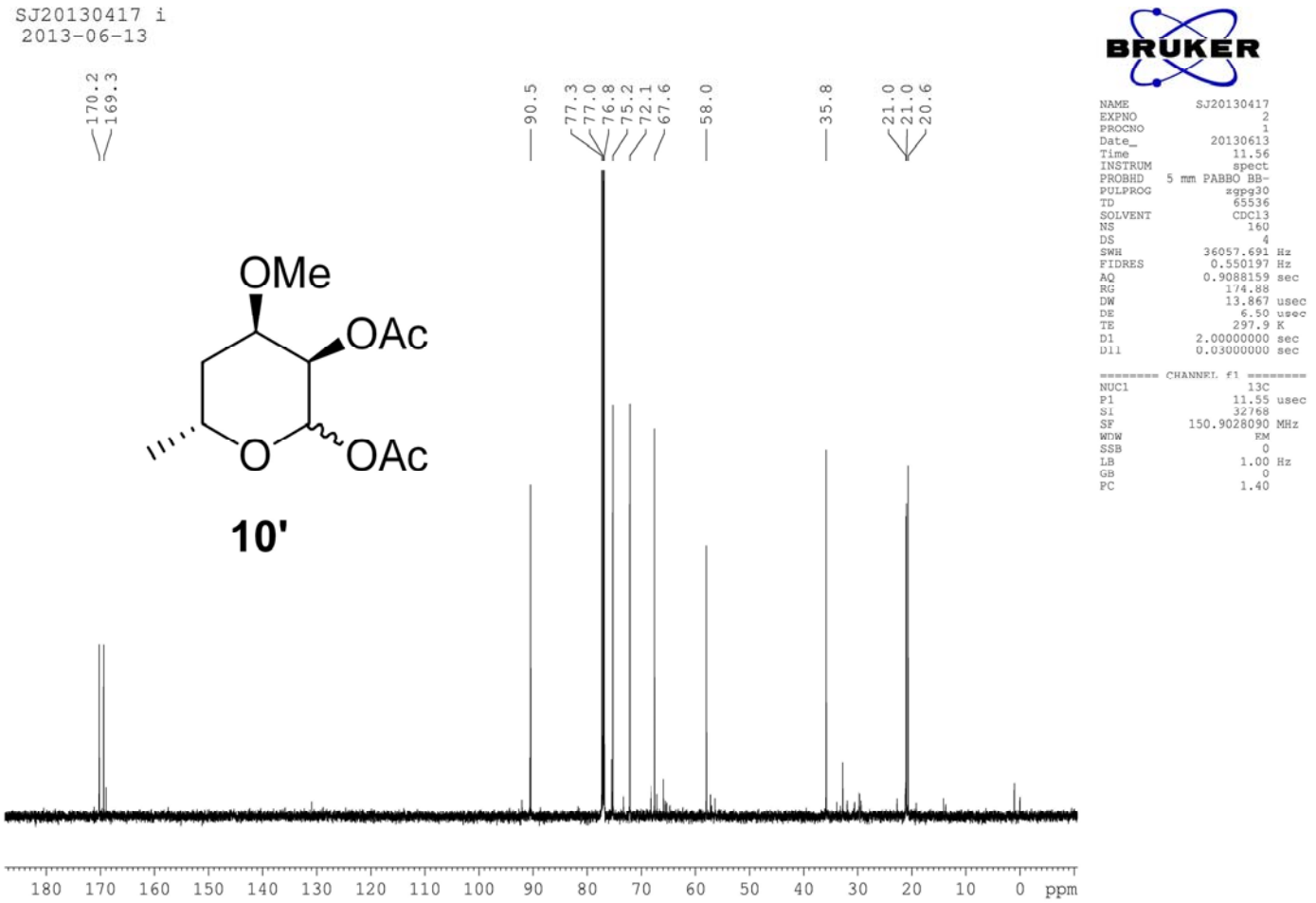



| | |
|---|---|
| NAME | SJ20121130B |
| EXPNO | 2 |
| PROCNO | 1 |
| Date_ | 20121204 |
| Time | 10.37 |
| INSTRUM | spect |
| PROBHD | 5 mm PABBO BB- |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 486 |
| DS | 2 |
| SWH | 45454.547 Hz |
| FIDRES | 0.693581 Hz |
| AQ | 0.7209570 sec |
| RG | 20600 |
| DW | 11.000 usec |
| DE | 6.50 usec |
| TE | 292.0 K |
| D1 | 2.00000000 sec |
| D11 | 0.03000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 6.00 usec |
| PL1 | 1.00 dB |
| PL1W | 83.20243835 W |
| SFO1 | 150.9178993 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | -4.00 dB |
| PL12 | 13.16 dB |
| PL13 | 16.00 dB |
| PL2W | 34.70265579 W |
| PL12W | 0.66736388 W |
| PL13W | 0.34702653 W |
| SFO2 | 600.1324005 MHz |
| SI | 32768 |
| SF | 150.9028204 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 3.00 Hz |
| GB | 0 |
| PC | 1.40 |


7.260
6.244
6.234
5.576
5.556
4.907
4.887
4.884
4.863
3.879
3.874
3.865
3.860
3.714
3.701
3.649
3.639
3.346
2.073
2.060
1.724
1.707
1.687
1.633
1.616
1.597
1.288
1.272
BRUKER
NAME SJ20130601A
EXPNO 1
PROCNO 1
Date_ 20130608
Time 10.34
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 71.8
DW 62.400 usec
DE 6.50 usec
TE 300.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.40 usec
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OMe
OAc
OAc
O
14
0.38
1.00
1.44
1.51
1.13
3.03
3.10
2.92
0.82
0.98
3.19
7
6
5
4
3
2
1
0
ppm


SJ20130601A
2013-06-13
169.8
169.6
99.9
92.5
78.2
77.6
77.3
77.0
76.8
69.1
55.2
37.1
29.7
21.0
20.8
OMe
OAc
O
OAc
14
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
BRUKER
NAME SJ20130601A
EXPNO 2
PROCNO 1
Date_ 20130613
Time 12.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 115
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 174.88
DW 13.867 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 11.55 usec
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


Sample:
7.260
6.039
6.011
4.743
4.733
4.715
4.705
4.112
4.105
4.096
4.090
4.075
4.068
3.844
3.835
3.373
2.082
2.070
1.745
1.699
1.668
1.657
1.620
1.532
1.525
1.495
1.486
1.477
1.448
1.440
1.222
1.201
BRUKER
OMe
OAc
OAc
O
10'
NAME SJ20130417
EXPNO 1
PROCNO 1
Date_ 20130418
Time 11.45
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 512
DW 81.000 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.80 usec
PL1 1.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300053 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.00
1.12
1.42
1.13
3.02
3.03
3.15
0.93
1.31
3.19
7
6
5
4
3
2
1
0
ppm


SJ20130417 i
2013-06-13
170.2
169.3
90.5
77.3
77.0
76.8
75.2
72.1
67.6
58.0
35.8
21.0
21.0
20.6
OMe
OAc
OAc
O
10'
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
BRUKER
NAME SJ20130417
EXPNO 2
PROCNO 1
Date_ 20130613
Time 11.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 160
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 174.88
DW 13.867 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 11.55 usec
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40