

Supporting Information

Total Synthesis of the Lipid Anchor Attached Core Trisaccharides of Lipoteichoic Acids of *Streptococcus pneumoniae* and *Streptococcus oralis* Uo5

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Table of Contents

Experimental Procedures:

General Information	S6
Synthesis of compound 13	S6
Synthesis of compound 11	S7
Synthesis of compound 16	S7
Synthesis of compound 8	S8
Synthesis of compound 18	S9
Synthesis of compound 10	S10
Synthesis of compound 19	S10
Synthesis of compound 9	S11
Synthesis of compound 21	S12
Synthesis of compound 7	S13
Synthesis of compound 20a	S14
Synthesis of compound 22	S14
Synthesis of compound 5	S15
Synthesis of compound 23	S16
Synthesis of compound 25a	S16

Synthesis of compound 26	S17
Synthesis of compound 6	S18
Synthesis of compound 27	S18
Synthesis of compound 3	S19
Synthesis of compound 29	S20
Synthesis of compound 4	S21
Synthesis of compound 30	S22
Synthesis of compound 31	S23
Synthesis of compound 1	S23
Synthesis of compound 2	S24
II. Spectra:	
¹ H NMR spectrum of compound 13	S26
¹³ C NMR spectrum of compound 13	S27
DEPT spectrum of compound 13	S28
¹ H- ¹ H COSY spectrum of compound 13	S29
¹ H NMR spectrum of compound 11	S30
¹³ C NMR spectrum of compound 11	S31
DEPT spectrum of compound 11	S32
¹ H- ¹ H COSY spectrum of compound 11	S33
¹ H NMR spectrum of compound 16	S34
¹³ C NMR spectrum of compound 16	S35
¹ H- ¹ H COSY spectrum of compound 16	S36
¹ H NMR spectrum of compound 8	S37
¹³ C NMR spectrum of compound 8	S38
¹ H- ¹ H COSY spectrum of compound 8	S39

¹ H NMR spectrum of compound 18	S40
¹³ C NMR spectrum of compound 18	S41
¹ H- ¹ H COSY spectrum of compound 18	S42
¹ H NMR spectrum of compound 10	S43
¹³ C NMR spectrum of compound 10	S44
¹ H- ¹ H COSY spectrum of compound 10	S45
¹ H NMR spectrum of compound 19	S46
¹³ C NMR spectrum of compound 19	S47
¹ H- ¹ H COSY spectrum of compound 19	S48
¹ H- ¹³ C HSQC spectrum of compound 19	S49
¹ H NMR spectrum of compound 9	S50
¹³ C NMR spectrum of compound 9	S51
¹ H- ¹ H COSY spectrum of compound 9	S52
¹ H NMR spectrum of compound 21	S53
¹³ C NMR spectrum of compound 21	S54
¹ H- ¹ H COSY spectrum of compound 21	S55
¹ H- ¹³ C HSQC spectrum of compound 21	S56
¹ H NMR spectrum of compound 7	S57
¹³ C NMR spectrum of compound 7	S58
¹ H- ¹ H COSY spectrum of compound 7	S59
¹ H NMR spectrum of compound 20a	S60
¹³ C NMR spectrum of compound 20a	S61
¹ H- ¹ H COSY spectrum of compound 20a	S62
¹ H NMR spectrum of compound 22	S63
¹³ C NMR spectrum of compound 22	S64

DEPT spectrum of compound 22	S65
¹ H- ¹ H COSY spectrum of compound 22	S66
¹ H NMR spectrum of compound 5	S67
¹³ C NMR spectrum of compound 5	S68
¹ H- ¹ H COSY spectrum of compound 5	S69
¹ H NMR spectrum of compound 23	S70
¹³ C NMR spectrum of compound 23	S71
DEPT spectrum of compound 23	S72
¹ H- ¹ H COSY spectrum of compound 23	S73
¹ H NMR spectrum of compound 25a	S74
¹³ C NMR spectrum of compound 25a	S75
¹ H- ¹ H COSY spectrum of compound 25a	S76
¹ H NMR spectrum of compound 26	S77
¹³ C NMR spectrum of compound 26	S78
DEPT spectrum of compound 26	S79
¹ H- ¹ H COSY spectrum of compound 26	S80
¹ H NMR spectrum of compound 6	S81
¹³ C NMR spectrum of compound 6	S82
DEPT spectrum of compound 6	S83
¹ H- ¹ H COSY spectrum of compound 6	S84
¹ H NMR spectrum of compound 27	S85
¹³ C NMR spectrum of compound 27	S86
DEPT spectrum of compound 27	S87
¹ H- ¹ H COSY spectrum of compound 27	S88
¹ H NMR spectrum of compound 3	S89

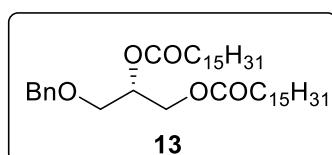
¹³ C NMR spectrum of compound 3	S90
¹ H- ¹ H COSY spectrum of compound 3	S91
¹ H- ¹³ C HSQC spectrum of compound 3	S92
¹ H NMR spectrum of compound 29	S93
¹³ C NMR spectrum of compound 29	S94
¹ H- ¹³ C HSQC spectrum of compound 29	S95
¹ H NMR spectrum of compound 4	S96
¹³ C NMR spectrum of compound 4	S97
¹ H- ¹ H COSY spectrum of compound 4	S98
¹ H- ¹³ C HSQC spectrum of compound 4	S99
¹ H NMR spectrum of compound 30	S100
¹³ C NMR spectrum of compound 30	S101
DEPT spectrum of compound 30	S102
¹ H- ¹ H COSY spectrum of compound 30	S103
¹ H- ¹³ C HSQC spectrum of compound 30	S104
¹ H NMR spectrum of compound 31	S105
¹³ C NMR spectrum of compound 31	S106
DEPT spectrum of compound 31	S107
¹ H- ¹³ C HSQC spectrum of compound 31	S108
¹ H NMR spectrum of compound 1	S109
¹³ C NMR spectrum of compound 1	S110
¹ H- ¹³ C HSQC spectrum of compound 1	S111
¹ H NMR spectrum of compound 2	S112
¹³ C NMR spectrum of compound 2	S113
¹ H- ¹³ C HSQC spectrum of compound 2	S114

I. Synthetic procedures and characterization data

Experimental Section

A. General Methods: All reactions were conducted under a dry nitrogen atmosphere. Solvents (CH_2Cl_2 >99%, THF 99.5%, Acetonitrile 99.8%, DMF 99.5%) were purchased in capped bottles and dried under sodium or CaH_2 . All other solvents and reagents were used without further purification. All glasswares used were oven dried before use. TLC was performed on pre-coated Aluminium plates of Silica Gel 60 F254 (0.25 mm, E. Merck). Developed TLC plates were visualized under a short-wave UV lamp and by heating plates that were dipped in ammonium molybdate/cerium (IV) sulfate solution. Silica gel column chromatography was performed using Silica Gel (100-200 mesh or 230-400 mesh) and employed a solvent polarity correlated with TLC mobility. We have used 3 Angstrom powdered molecular sieves in our study. The powdered MS were weighed in a dried pear shaped flask and activated by periodic heating of flask by using flame over 15 min period under high vacuum. Paraffin oil bath was used for heating wherever required. NMR experiments were conducted on 400 and 500 MHz instrument using CDCl_3 (D, 99.8%) or CD_3OD (D, 99.9%) as solvents. Chemical shifts are relative to the deuterated solvent peaks and are in parts per million (ppm). ^1H - ^1H COSY was used to confirm proton assignments. Mass spectra were acquired in the ESI mode. Specific rotation experiments were measured at 589 nm (Na) and 25 °C. IR spectra were recorded on an FT-IR spectrometer using CsCl plates.

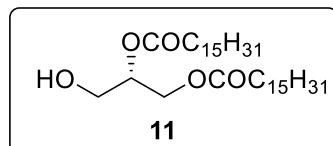
3-O-Benzyl-1,2-di-O-palmitoyl-sn-glycerol (13)



Compound **12** (0.85 g, 4.66 mmol) was dissolved in dry CH_2Cl_2 (12 mL). To this solution, DCC (2.4 g, 11.6 mmol), DMAP (0.28 g, 2.33 mmol) and palmitic acid (2.99 g, 11.6 mmol) were added. The reaction was stirred for 3 h at rt. The mixture was filtered over Celite, washed with saturated aqueous NaHCO_3 (3 × 20 mL), dried on anhyd. Na_2SO_4 and concentrated in *vacuo*. The residue was purified by silica gel column chromatography (100-200 mesh, 15% EtOAc-petroleum ether) to obtain compound **13** as white solid (2.52 g, 82%). $[\alpha]_D^{25} +5.89$ ($c = 1.0$, CHCl_3); **M.P.** 57-59 °C; **IR** (cm^{-1} , CHCl_3) ν 2916, 2849, 1731, 1470, 1173, 1111, 1027, 785, 745; **$^1\text{H NMR}$** (500 MHz, CDCl_3) δ 7.38-7.28 (m, 5H, ArH), 5.29-5.25 (m, 1H, CH), 4.56 (ABq, $J = 12.1, 8.1$ Hz, 2H, CH_2Ph), 4.38 (dd, $J = 3.7, 8.1$ Hz, 1H), 4.22 (dd, $J = 6.4, 5.4$ Hz,

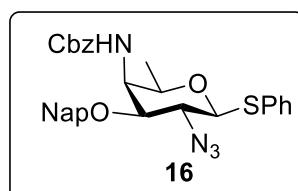
1H), 3.61 (dd, J = 3.8, 1.3 Hz, 2H), 2.36-2.28 (m, 4H, COCH₂), 1.65-1.61 (m, 4H), 1.28 (bs, 48H), 0.91 (t, 6H, CH₃); ¹³C NMR (125 MHz, CDCl₃) δ 173.4, 173.1, 137.7, 128.4, 127.7, 127.6, 73.3, 70.0, 68.2, 62.6, 34.3, 34.1, 31.9, 29.7, 29.6, 29.6, 29.5, 29.4, 29.3, 29.3, 29.1, 29.1, 24.9, 24.9, 22.7, 14.1; HR-ESI-MS (*m/z*): [M + Na]⁺ calcd. for C₄₂H₇₄O₅Na, 681.5428; found, 681.5459.

1,2 di-*O*-palmitoyl-*sn*-glycerol (11)



Compound **13** (0.8 g, 1.21 mmol) was dissolved in EtOH (25 mL). To this solution Pd(OH)₂/C (0.46 g) was added and the reaction was stirred under an atmosphere of hydrogen (1 atm) for 2 h. After complete consumption of starting material (as judged by TLC), reaction mixture was filtered through a Celite pad with CHCl₃/hexane as eluents. The crude product obtained after the removal of the solvent was purified by silica gel column chromatography (100-200 mesh, 20% EtOAc-petroleum ether) to afford **11** as a white solid (0.61 g, 88%). $[\alpha]_D^{25}$ -1.57 (*c* = 1.0, CHCl₃); M.P. 49-53 °C; IR (cm⁻¹, CHCl₃) ν 3458, 3015, 2936, 2861, 1729, 1449, 1366, 1334, 1282, 1216, 1163, 1101, 1042, 926, 667; ¹H NMR (400 MHz, CDCl₃) δ 5.09-5.04 (m, 1H), 4.30 (dd, J = 7.6, 4.2 Hz, 1H), 4.20 (dd, J = 6.0, 5.8 Hz, 1H), 3.70 (d, J = 5.0 Hz, 2H), 2.30 (m, 4H, COCH₂), 1.59 (m, 4H), 1.23 (bs, 48H), 0.85 (t, J = 6.4 Hz, 6H, CH₃); ¹³C NMR (100 MHz, CDCl₃) δ 173.7, 173.4, 72.0, 62.1, 61.3, 34.2, 34.0, 31.9, 29.7, 29.6, 29.6, 29.4, 29.3, 29.2, 29.1, 29.0, 24.9, 24.8, 24.6, 14.1; HR-ESI-MS (*m/z*): [M + Na]⁺ calcd. for C₃₅H₆₈O₅Na, 591.4959; found, 591.4975.

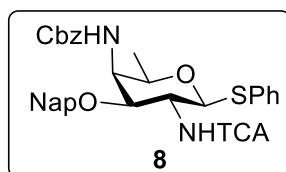
Phenyl-2-azido-3-*O*-(2-naphthylmethyl)-4-*N*-benzylcarbamate-2,4,6-trideoxy-1-thio- β -D galactopyranoside (16)



Tf₂O (0.34 mL, 2.01 mmol) was added dropwise at 0 °C to a stirred solution of compound **15** (0.2 g, 0.50 mmol), pyridine (0.48 mL, 6.03 mmol) in CH₂Cl₂ (10 mL) and the reaction mixture was gradually brought to 0 °C over 1 h. After complete conversion of the starting material the

reaction mixture was diluted with CH₂Cl₂ and washed successively with 1M HCl (2×5 mL), aq. NaHCO₃ (3×5 mL) and water. Separated organic layer was dried over Na₂SO₄, concentrated and the crude product was used for the next step without any purification. The crude product was dissolved in CH₃CN (13 mL), and to this, TBAN₃ (0.13 g, 0.47 mmol) was added at -30 °C and the reaction mixture was stirred at the same temperature for 16 h. After 16 h, the solvent was evaporated on a rotary evaporator under N₂ atmosphere and the residue was dissolved in DMF (2 mL). To this clear solution, CbzNH₂ (0.15 g, 1.00 mmol) was added. After 10 h, the reaction mixture was diluted with EtOAc and washed with brine (4×10 mL) and water. The separated aqueous layer was washed with EtOAc. The combined organic layers were dried over Na₂SO₄ and concentrated in *vacuo*. The crude product was purified by silica gel column chromatography (100-200 mesh, 20% EtOAc-petroleum ether) to give **16** as colourless sticky gum (0.17 g, 60%). $[\alpha]_D^{25} -5.07$ ($c = 0.25$, CHCl₃); **IR** (cm⁻¹, CHCl₃) ν 3402, 2924, 2114, 1717, 1700, 1217, 1034, 757; **¹H NMR** (400 MHz, CDCl₃) δ 7.85-7.82 (m, 4H, ArH), 7.58-7.53 (m, 3H, ArH), 7.50-7.47 (d, 2H, ArH), 7.36-7.31 (m, 8H, ArH), 5.14 (ABq, $J = 12.3$ Hz, 2H, CH₂Cbz), 5.02 (d, $J = 11.3$ Hz, 1H, CHHNAP), 4.83 (d, $J = 10.2$ Hz, 1H, NH), 4.71 (d, $J = 11.3$ Hz, 1H, CHHNAP), 4.34 (d, $J = 10.2$, 1H, H-1), 4.28 (dd, $J = 6.5, 3.7$ Hz, 1H, H-4), 3.64-3.59 (m, 1H, H-5), 3.55 (dd, $J = 5.6, 4.1$ Hz, 1H, H-3), 3.27 (t, $J = 9.9$ Hz, 1H, H-2), 1.29 (d, $J = 6.2$ Hz, 3H, CH₃); **¹³C NMR** (100 MHz, CDCl₃) δ 156.9, 136.4, 134.7, 133.8, 133.3, 131.3, 129.3, 128.8, 128.6, 127.8.4, 128.2, 128.1, 128.0, 127.8, 127.4, 126.3, 126.2, 126.1, 86.2, 79.9, 74.2, 71.3, 67.1, 67.4, 51.2, 17.3; **HR-ESI-MS** (m/z): [M + Na]⁺ calcd. for C₃₁H₃₀O₄N₄SNa, 577.1880; found, 577.1879.

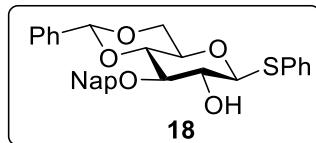
Phenyl-2-trichloroacetamido-3-O-(2-naphthylmethyl)-4-N-benzylcarbamate-2,4,6-trideoxy-1-thio- β -D-galactopyranoside (**8**)



Compound **16** (0.2 g, 0.36 mmol) was dissolved in Ethyl acetate : AcOH (1:2, 9 mL). To this stirred solution, Zn (50 mg) was added and then the reaction mixture was kept stirring for 2 h. After complete consumption of starting material (as judged by TLC), the reaction mixture was filtered through Celite, solvents were removed in *vacuo* and the crude product was dried under high vacuum for 1 h.

The crude product with 3 Å MS (0.4 g) was dissolved in THF (2.5 mL), and trichloro acetyl chloride (81 µL, 0.72 mmol) was added. After stirring the reaction mixture at rt for 3 h solvents were removed under reduced pressure and the crude product was purified by silica gel column chromatography (100-200 mesh, 30% EtOAc-petroleum ether) to obtain **8** as a foam (0.17 g, 71%). $[\alpha]_D^{25} +37.32$ ($c = 1.0$, CHCl₃); **IR** (cm⁻¹, CHCl₃) ν 3400, 3320, 3025, 2871, 1713, 1699, 1521, 1218, 1083, 1046, 819, 758, 697, 675; **¹H NMR** (400 MHz, CDCl₃) δ 7.82-7.77 (m, 4H, ArH), 7.52-7.43 (m, 5H, ArH), 7.33-7.29 (m, 8H, ArH), 6.87 (d, $J = 7.6$ Hz, 1H, NH), 5.15 (ABq, $J = 12.4$ Hz, 2H, -CH₂Cbz), 5.08-5.02 (m, 2H, NH, H-1), 4.97 (d, $J = 11.3$ Hz, 1H, CHHNAP), 4.60 (d, $J = 11.3$ Hz, 1H, CHHNap), 4.37 (dd, $J = 6.4, 3.5$ Hz, 1H, H-4), 4.06 (dd, $J = 6.5, 3.9$ Hz, 1H, H-3), 3.76-3.73 (m, 1H, H-5), 3.48 (m, 1H, H-2), 1.31 (d, $J = 6.4$ Hz, 3H); **¹³C NMR** (100 MHz, CDCl₃): δ 161.6, 157.0, 136.2, 134.5, 133.2, 133.2, 133.1, 131.9, 129.1, 128.5, 128.4, 128.1, 127.9, 127.8, 127.7, 126.2, 126.2, 126.1, 92.4, 85.2, 76.7, 75.9, 74.2, 71.1, 67.1, 53.7, 51.5, 17.2; **HR-ESI-MS** (m/z): [M + Na]⁺ calcd. for C₃₃H₃₁Cl₃N₂O₅NaS, 695.0911; found, 695.0912.

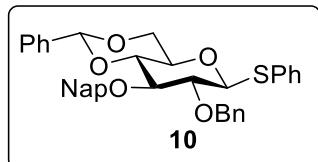
Phenyl-3-O-(2-naphthylmethyl)-4,6-O-benzylidene-1-thio-β-D-glucopyranoside (18)



Bu₂SnO (1.24 g, 4.99 mmol) was added to a stirred solution of phenyl 4,6-O-benzylidene-1-D-glucose **17** (1.5 g, 4.16 mmol) in toluene (30 mL) and the reaction mixture was refluxed at 110 °C for 8 h. After complete consumption of the starting material (as judged by TLC) solvent was removed under reduced pressure and the crude product was dried under high vacuum for 5 h. Above crude product was dissolved in dry toluene (30 mL) and TBAB (2.01 g, 6.24 mmol) followed by 2-naphthylmethyl bromide (1.38 g, 6.24 mmol) were added. Reaction mixture was kept stirring for 6 h at 60 °C temperature. After completion of the reaction (confirmed by TLC), reaction mixture was diluted with EtOAc, and washed with brine (2 × 25 mL) solution. Separated organic layer was dried over Na₂SO₄, concentrated and purified by silica gel column chromatography (100-200 mesh, 30% EtOAc-petroleum ether) to give **18** (1.46 g, 70%) as a white solid. $[\alpha]_D^{25} -13.74$ ($c = 1.0$, CHCl₃); **M.P.** 184-185 °C; **IR** (cm⁻¹, CHCl₃) ν 3542, 3018, 2881, 1733, 1374, 1215, 1086, 771, 756, 669; **¹H NMR** (400 MHz, CDCl₃) δ 7.82-7.78 (m, 3H, ArH), 7.73 (m, 1H, ArH), 7.55-7.44 (m, 7H, ArH), 7.39-7.34 (m, 3H, ArH), 7.32 (m, 3H, ArH), 5.59 (s, 1H, CHPh), 5.10, 4.97 (ABq, $J = 11.8$ Hz, 2H, CH₂NAP), 4.63 (d, $J = 9.7$ Hz, 1H, H-1), 4.39 (dd, $J = 5.5, 4.9$ Hz, 1H, H-6'), 3.80 (t, $J = 10.2$ Hz, 1H, H-6), 3.77-3.67 (m, 2H,

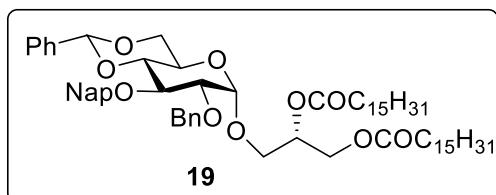
H-3, H-4), 3.58-3.49 (m, 2H, H-2, H-5), 2.57 (d, J = 2.1 Hz, 1H, OH); **¹³C NMR** (100 MHz, CDCl₃) δ 137.2, 135.6, 133.2, 133.2, 133.0, 132.2, 131.3, 129.0, 128.4, 128.3, 127.9, 127.7, 126.9, 126.2, 126.1, 126.0, 125.9, 101.3, 88.5, 81.4, 81.1, 74.8, 72.3, 70.7, 68.6; **HR-ESI-MS** (m/z): [M + Na]⁺ calcd. for C₃₀H₂₈O₅SNa, 523.1550; found, 523.1553.

Phenyl-4,6-O-benzylidene-3-O-(2-naphthylmethyl)-2-O-benzyl-1-thio- β -D-glucopyranoside (10)



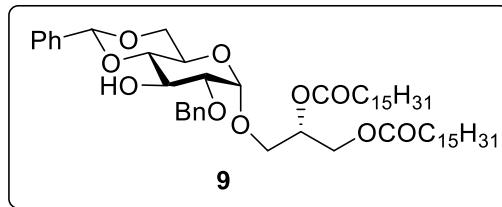
NaH (19 mg, 0.80 mmol) and BnBr (57 μ L, 0.48 mmol) were added sequentially to a stirred solution of compound **17** (0.16 g, 0.32 mmol) in DMF (0.7 mL) at 0 °C. After complete consumption of starting material (confirmed by TLC), reaction mixture was diluted with EtOAc and transferred into a separating funnel. Organic layer was washed with brine (4 \times 5 mL) and water, the collected organic layer was dried over Na₂SO₄, concentrated and purified by silica gel column chromatography (100-200 mesh, 15% EtOAc-petroleum ether) to afford **10** as a white solid (0.15g, 84%); $[\alpha]_D^{25}$ -9.96 (c = 1.0, CHCl₃); **M.P.** 139-142 °C; **IR** (cm⁻¹, CHCl₃) ν 3052, 2924, 2861, 1453, 1366, 1271, 1216, 1171, 1086, 1027, 815, 752, 696; **¹H NMR** (400 MHz, CDCl₃) δ 7.88-7.81 (m, 3H, ArH), 7.76 (m, 1H, ArH), 7.64-7.35 (m, 18 H, ArH), 5.68 (s, 1H, CHPh), 5.17, 5.03 (ABq, J = 11.5 Hz, 2H, CH₂NAP), 4.96 (ABq, J = 10.3 Hz, 2H, CH₂Ph), 4.86 (d, J = 9.7 Hz, 1H, H-1), 4.46 (dd, J = 5.4, 5 Hz, 1H, H-6'), 3.97 (dd, J = 8.6, 0.6 Hz, 1H, H-3), 3.88 (t, J = 10.3 Hz, 1H, H-6), 3.82 (t, J = 9.3 Hz, 1H, H-4), 3.6 (dd, J = 8.3, 1.3 Hz, 1H, H-2), 3.58-3.52 (m, 1H, H-5); **¹³C NMR** (100 MHz, CDCl₃) δ 138.1, 137.3, 135.8, 133.3, 133.2, 133.1, 132.4, 129.1, 128.5, 128.4, 128.3, 128.2, 128.0, 127.9, 127.7, 126.9, 126.3, 126.1, 126.1, 125.9, 101.3, 88.4, 83.0, 81.5, 80.6, 76.0, 75.4, 70.3, 68.80; **HR-ESI-MS** (m/z): [M + Na]⁺ calcd. for C₃₇H₃₄O₅SNa, 613.2019; found, 613.2022.

3-O-(4,6-O-Benzylidene-2-O-benzyl-3-O-(2-naphthylmethyl)-1- α -D-glucopyranosyl)-1,2-di-O-palmitoyl-sn-glycerol (19)



A suspension of donor **10** (0.5 g, 0.84 mmol), acceptor **11** (0.43 g, 0.76 mmol) and 3 Å MS (2 g) in CH₂Cl₂ : Et₂O (1:1, 8 mL) was stirred under nitrogen atmosphere at rt for about 0.5 h. The flask was cooled to 0 °C and NIS (0.38 g, 1.69 mmol) was added followed by addition of TMSOTf (15 µL, 0.08 mmol) at same temp. The reaction mixture was stirred at 0 °C under nitrogen atmosphere for 2 h. The reaction was quenched by adding Et₃N (0.1 mL). The reaction mixture was diluted with CH₂Cl₂, filtered through Celite and transferred to a separating funnel. The organic layer was washed with aq. Na₂S₂O₃ (2 × 15 mL), the separated organic layer was dried over Na₂SO₄ and concentrated. The crude product was purified by silica gel column chromatography (230-400 mesh, 15% EtOAc-petroleum ether) to afford **19** as a white solid (0.62 g, 78%). [α]_D²⁵ +5.74 (c = 1.0, CHCl₃); **M.P.** 65-66 °C; **IR** (cm⁻¹, CHCl₃) ν 3020, 2924, 2852, 1736, 1467, 1216, 1092, 770, 669; **¹H NMR** (400 MHz, CDCl₃) δ 7.82-7.68 (m, 4H, ArH), 7.50-7.29 (m, 13H, ArH), 5.57 (s, 1H, CHPh), 5.29-5.24 (m, 1H, CH), 5.07, 4.99 (ABq, J = 11.6 Hz, 2H, CH₂NAP), 4.84 (d, J = 12.0 Hz, 2H, CHHPh), 4.76 (d, J = 3.6 Hz, 1H, H-1), 4.69 (d, J = 12.0 Hz, 2H, CHHPh), 4.44 (dd, J = 8.3, 3.7 Hz, 1H), 4.27-4.17 (m, 2H, H-6), 4.08 (t, J = 9.2, 1H, H-3), 3.88-3.58 (m, 6H, H-5, H-6', H-2, H-4), 2.37-2.29 (m, 4H), 1.61 (m, 4H), 1.26 (bs, 48H), 0.89 (t, J = 6.6 Hz, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 173.3, 173.0, 138.2, 137.3, 136.2, 133.3, 132.9, 128.9, 128.4, 128.2, 127.9, 127.8, 127.8, 127.6, 126.5, 126.1, 125.8, 125.7, 101.4, 98.5, 82.0, 79.4, 78.4, 77.2, 75.3, 73.5, 69.7, 69.0, 66.4, 62.8, 62.4, 34.3, 34.1, 31.9, 29.7, 29.6, 29.5, 29.3, 29.3, 29.1, 29.1, 24.9, 22.7, 14.1; **HR-ESI-MS** (*m/z*): [M + Na]⁺ calcd. for C₆₆H₉₆O₁₀Na, 1071.6896; found, 1071.6891.

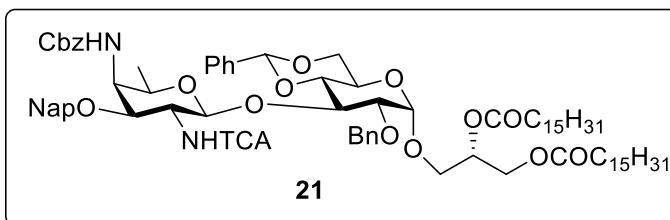
1,2-di-*O*-Palmitoyl-3-*O*-(4,6-*O*-benzylidene-2-*O*-benzyl-1- α -D-glucopyranosyl)-*sn*-glycerol (**9**)



To a solution of **19** (1.3 g, 1.23 mmol) in CH₂Cl₂ (104 mL) and water (5.2 mL), β-pinene (0.32 mL, 1.96 mmol), and DDQ (0.33 g, 1.48 mmol) were added at room temperature. After stirring for 2.5 h, the reaction mixture was diluted with CH₂Cl₂ (50 mL), and the organic layer was washed 3 times with 50 mL of 2 M NaOH. Organic layers were washed with 100 mL of sat. NaHCO₃, dried over Na₂SO₄, filtered, and concentrated in *vacuo*. Solvent was removed under reduced pressure and obtained crude product was purified by silica gel column chromatography

(100-200 mesh, 25% EtOAc-petroleum ether) to give **9** as sticky gum (0.91 g, 81%). $[\alpha]_D^{25} +18.93$ ($c = 1.0$, CHCl₃); **IR** (cm⁻¹, CHCl₃) ν 3544, 2920, 1652, 1260, 1048, 775; **¹H NMR** (500 MHz, CDCl₃) δ 7.49-7.47 (m, 2H, ArH), 7.37-7.30 (m, 8H, ArH), 5.51 (s, 1H, CHPh), 5.24-5.21 (m, 1H, CH), 4.75, 4.64 (ABq, $J = 12.0$ Hz, 2H, CH₂Ph), 4.73 (d, $J = 3.6$ Hz, 1H, H-1), 4.41 (dd, $J = 8.2, 3.7$ Hz, 1H), 4.23-4.18 (m, 2H, H-6'), 4.13 (t, $J = 9.2$ Hz, 1H, H-3), 3.84-3.73 (m, 2H, H-5), 3.68 (t, $J = 10.3$ Hz, 1H, H-6), 3.52-3.45 (m, 3H, H-2, H-4), 2.56 (bs, 1H, OH), 2.33-2.92 (m, 4H), 1.60 (m, 4H), 1.25 (bs, 48H), 0.87 (t, $J = 6.8$ Hz, 6H); **¹³C NMR** (125 MHz, CDCl₃) δ 173.3, 173.0, 138.0, 137.1, 129.1, 128.6, 128.2, 128.0, 127.9, 126.3, 101.9, 97.8, 81.1, 79.7, 73.1, 70.0, 69.7, 68.9, 66.3, 62.5, 62.4, 34.2, 34.1, 31.9, 29.7, 29.7, 29.6, 29.5, 29.4, 29.3, 29.3, 29.1, 29.1, 24.9, 22.7, 14.2; **HR-ESI-MS** (*m/z*): [M + Na]⁺ calcd. for C₅₅H₈₈O₁₀Na, 931.6270; found, 931.6271.

3-O-[(2-trichloroacetamido-3-O-(2-naphthylmethyl)-2,4,6-trideoxy-4-benzylcarbamate-1- β -galactopyranosyl)-(1 \rightarrow 3)-(4,6-O-benzylidene-2-benzyl-1- α -glucopyranosyl)]-1,2-di-*O*-palmitoyl-*sn*-glycerol (21)

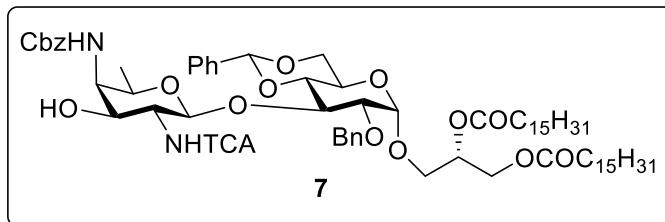


AAT thioglycoside **8** (0.1 g, 0.14 mmol) was dissolved in THF (2.52 mL) and H₂O (0.53 mL). NBS (79 mg, 0.44 mmol) was added to it at rt. after 1 h, after complete consumption of starting material (confirmed by TLC), reaction mixture was diluted with EtOAc and transferred into a separating funnel. Organic layer was washed with Na₂S₂O₃ (3 × 10 mL) and water, the collected organic layer was dried over Na₂SO₄, concentrated and the crude product was dried under high vacuum for 1 h. Crude product was dissolved in CH₂Cl₂ (12 mL). To this stirred solution CCl₃CN (0.3 mL, 2.81 mmol) and DBU (10 μ L, 0.07 mmol) was added at 0 °C. After 1 h, reaction mixture was directly concentrated and purified by silica gel column chromatography (100-200 mesh, 15% EtOAc-petroleum ether) to give **20** as colourless sticky gum.

Azeotropic mixture of donor **20** (0.15 g, 0.21 mmol) and acceptor **9** (0.11 g, 0.12 mmol) and 3 Å MS (0.36 g) was dissolved in dry CH₂Cl₂ (3 mL) and stirring was continued at rt for 0.5 h. TMSOTf (4 μ L, 0.02 mmol) was added at -30 °C. The reaction was stirred at same temperature for 1 h. After 1 h, reaction mixture was diluted with CH₂Cl₂ followed by filtration

through celite, concentrated and purified by silica gel column chromatography (230-400 mesh, 20% EtOAc-petroleum ether) to give compound **21** (0.3 g, 74%). $[\alpha]_D^{25} +7.35$ ($c = 1.0$, CHCl₃); **IR** (cm⁻¹, CHCl₃) ν 3404, 2925, 2857, 1733, 1245, 1217, 1055, 763, 670; **¹H NMR** (500 MHz, CDCl₃) δ 7.85-7.78 (m, 2H, ArH), 7.50-7.44 (m, 5H, ArH), 7.34-7.21 (m, 15H, ArH), 6.66 (d, $J = 7.7$ Hz, 1H, NH), 5.52 (s, 1H, CHPh), 5.31 (d, $J = 10.1$, 1H, NH), 5.20-5.13 (m, 3H), 4.99 (m, 2H, H-1'), 4.74, 4.54 (ABq, $J = 12.1$ Hz, 2H), 4.61 (m, 2H, H-1), 4.38 (dd, $J = 8.1$, 3.7 Hz, 1H), 4.32 (dd, $J = 6.3$, 3.7 Hz, 1H, H-4'), 4.22-4.14 (m, 3H, H-3, H-4, H-6''), 4.04 (dd, $J = 6.7$, 4.1 Hz, 1H, H-3'), 3.80-3.67 (m, 3H, H-5), 3.60-3.51 (m, 4H, H-2, H-2', H-5'), 3.44 (dd, $J = 5.6$, 5.1 Hz, 1H), 2.33-2.27 (m, 4H), 1.60 (m, 4H), 1.28 (bs, 48H), 1.18 (d, $J = 6.1$ Hz, 3H, H-6'), 0.91 (t, $J = 6.7$ Hz, 6H); **¹³C NMR** (125 MHz, CDCl₃) δ 173.5, 173.2, 162.1, 157.2, 138.4, 137.4, 136.6, 135.1, 133.4, 133.3, 129.2, 128.7, 128.7, 128.5, 128.3, 128.2, 128.1, 128.0, 127.9, 127.8, 127.5, 126.5, 126.3, 126.2, 101.6, 100.3, 98.3, 92.7, 80.7, 79.6, 75.3, 73.6, 71.2, 70.0, 69.8, 69.1, 67.1, 66.5, 62.7, 62.5, 56.6, 51.4, 34.4, 34.3, 32.1, 29.9, 29.9, 29.8, 29.7, 29.6, 29.5, 29.5, 29.3, 29.3, 25.1, 25.1, 22.9, 16.9, 14.3; **HR-ESI-MS** (*m/z*): [M + Na]⁺ calcd. for C₈₂H₁₁₃O₁₅Cl₃N₂Na, 1493.7099; found, 1493.7076.

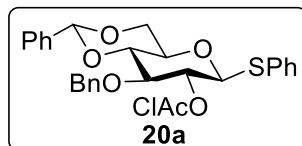
3-O-[(2-trichloroacetamido-2,4,6-trideoxy-4-benzylcarbamate-1- β -galactopyranosyl)-(1 \rightarrow 3)-(4,6-*O*-benzylidine-2-benzyl-1- α -glucopyranosyl)]-1,2-di-*O*-palmitoyl-*sn*-glycerol (7)



To a solution of **21** (0.17 g, 0.11 mmol) in CH₂Cl₂ (4.8 mL) and water (0.54 mL), DDQ (31 mg, 0.14 mmol) was added at room temperature. After stirring for 2 h, the reaction mixture was quenched with Et₃N (2-3 drops). Solvent was removed under reduced pressure and the obtained crude product was purified by silica gel column chromatography (100-200 mesh, 25% EtOAc-petroleum ether) to give **7** as sticky gum (0.11 g, 69%). $[\alpha]_D^{25} +46.42$ ($c = 0.1$, CHCl₃); **IR** (cm⁻¹, CHCl₃) ν 3511, 3404, 2929, 1723, 1650, 1454, 1244, 1217, 1086, 770, 575; **¹H NMR** (400 MHz, CDCl₃) δ 7.48-7.46 (m, 2H, ArH), 7.36-7.26 (m, 13H, ArH), 6.72 (d, $J = 7.6$ Hz, 1H, -NH), 5.49 (s, 1H, CHPh), 5.32 (d, $J = 9$ Hz, 1H, -NH), 5.18-5.05 (m, 3H), 4.84 (d, $J = 8.3$ Hz, 1H, H-1'), 4.73, 4.54 (ABq, $J = 12.1$ Hz, 2H, CH₂Ph), 4.62 (d, $J = 3.6$ Hz, 1H, H-1), 4.35 (dd, $J = 8.2$, 3.7 Hz, 1H), 4.20-4.12 (m, 3H, H-4), 3.97 (d, $J = 8.0$ Hz, 2H, H-4'), 3.79-3.67 (m,

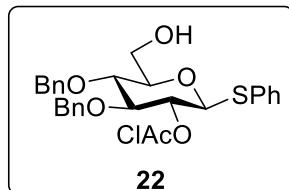
3H, H-3, H-5, H-6), 3.60-3.51 (m, 4H, H-2, H-2', H-3', H-5'), 3.41 (dd, $J = 5.4, 5.4$ Hz, 1H, H-6''), 3.10 (d, $J = 4.1$ Hz, 1H, -OH), 2.31-2.15 (m, 4H), 1.58 (m, 4H), 1.25 (bs, 48H), 1.12 (d, $J = 6.3$ Hz, 3H, H-6'), 0.88 (t, $J = 6.6$ Hz, 6H); **^{13}C NMR** (100 MHz, CDCl_3) δ 173.3, 173.0, 162.6, 157.9, 138.1, 137.2, 136.0, 129.0, 128.6, 128.5, 128.2, 128.1, 128.0, 127.4, 126.1, 101.3, 100.6, 97.9, 92.5, 80.1, 79.6, 77.2, 76.8, 73.0, 71.1, 69.6, 68.8, 67.3, 66.4, 62.6, 62.3, 57.4, 55.2, 34.2, 34.1, 31.9, 29.7, 29.6, 29.5, 29.3, 29.2, 29.1, 29.1, 24.8, 22.7, 16.6, 14.1; **HR-ESI-MS** (m/z): [M + Na]⁺ calcd. for $\text{C}_{71}\text{H}_{105}\text{O}_{15}\text{Cl}_3\text{N}_2\text{Na}$, 1353.6473; found, 1353.6472.

Phenyl-4,6-*O*-benzylidene-3-*O*-benzyl-2-*O*-chloroacetyl-1-thio- β -D-glucopyranoside (20a)



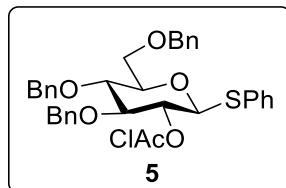
Compound **20** (0.14 g, 0.31 mmol) was dissolved in CH_2Cl_2 (2.5 mL). To this clear solution pyridine (75 μL , 0.94 mmol) and ClAcCl (75 μL , 0.94 mmol) were added. After 3 h, complete consumption of starting material (confirmed by TLC), reaction solvent was removed under reduced pressure and the obtained crude product was purified by silica gel column chromatography (100-200 mesh, 30% EtOAc-petroleum ether) to afford **20a** as white sticky solid (0.15 g, 92 %). $[\alpha]_D^{25} +2.21$ ($c = 1.0$, CHCl_3); **IR** (cm^{-1} , CHCl_3) ν 2916, 2849, 1731, 1470, 1173, 1111, 1027, 785, 745; **^1H NMR** (500 MHz, CDCl_3) δ 7.54-7.50 (m, 4H, ArH), 7.45-7.40 (m, 3H, ArH), 7.38-7.29 (m, 8H, ArH), 5.59 (s, 1H, CHPh), 5.08 (t, $J = 8.9$ Hz, 1H, H-2), 4.91, 4.68 (d, $J = 12.0$ Hz, 1H, CHHPh), 4.73 (d, $J = 10.0$ Hz, 1H, H-1), 4.68 (d, $J = 12.0$ Hz, 1H, CHHPh), 4.42 (dd, $J = 5.5, 5.0$ Hz, 1H), 3.98, 3.87 (ABq, $J = 14.8$ Hz, 2H, CH_2), 3.84-3.79 (m, 2H, H-3, H-6), 3.75 (t, $J = 9.2$ Hz, 1H, H-4), 3.55-3.51 (m, 1H, H-5); **^{13}C NMR** (125 MHz, CDCl_3) δ 165.8, 138.0, 137.1, 133.1, 131.6, 129.1, 129.1, 128.5, 128.3, 128.1, 127.9, 126.0, 101.3, 86.3, 81.2, 79.5, 77.4, 77.2, 76.9, 74.5, 72.9, 70.5, 68.5, 40.7; **HR-ESI-MS** (m/z): [M + K]⁺ calcd. for $\text{C}_{28}\text{H}_{27}\text{ClO}_6\text{SK}$, 565.0848; found, 565.0844.

Phenyl-2-*O*-chloroacetyl-3,4-di-*O*-benzyl-1-thio- β -D-glucopyranoside (22)



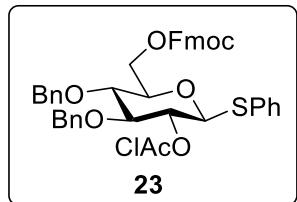
$\text{BH}_3\cdot\text{THF}$ (1 M, 4.1 mL, 3.46 mmol) was added to a solution of fully protected benzylidene compound (0.61 g, 1.15 mmol) in CH_2Cl_2 (2 mL) under nitrogen atmosphere. After 10 min. TMSOTf (41 μL , 0.23 mmol) was added at 0 °C. Reaction mixture was stirred for 2 h, and quenched with MeOH (3 mL), Et_3N , concentrated and purified by silica gel column chromatography (100-200 mesh, 20% ethyl acetate: pet ether) to give **22** as a viscous liquid (0.53 g, 88%). $[\alpha]_D^{25} +7.46$ ($c = 0.5$, CHCl_3); **IR** (cm^{-1} , CHCl_3) ν 3545, 3436, 2949, 2926, 1642, 1455, 1377, 1216, 1082, 916, 767; **1H NMR** (400 MHz, CDCl_3) δ 7.38-7.17 (m, 15H, ArH), 4.91 (t, $J = 9.1$ Hz, 1H, H-2), 4.92-4.74 (m, 2H, CH_2Ph), 4.59-4.56 (m, 3H, CH_2Ph , H-1), 3.80 (m, 2H, H-4), 3.67-3.54 (m, 5H, CH_2AcCl , H-4, H-6, 6'), 3.37-3.33 (m, 1H, H-5), 1.89 (bs, 1H, -OH). **13C NMR** (100 MHz, CDCl_3): δ 166.0, 137.9, 137.5, 132.5, 132.1, 129.1, 128.6, 128.5, 128.2, 128.1, 128.0, 127.9, 85.6, 83.8, 79.6, 77.5, 75.4, 75.26, 73.2, 61.8, 40.67; **HR-ESI-MS** (m/z): $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{28}\text{H}_{29}\text{ClO}_6\text{SNa}$, 551.1266; found, 551.1249.

Phenyl-2-O-chloroacetyl-3,4,6-tri-O-benzyl-1-thio- β -D-glucopyranoside (5)



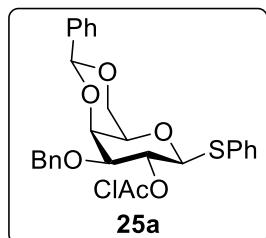
Benzyl trichloroimide (45 μL , 0.37 mmol) was added to a stirred solution of **22** (0.14 g, 0.26 mmol) in CH_2Cl_2 (0.53 mL). TfOH (7.5 mg, 0.31 mmol) was added to it at 0 °C and reaction mixture was kept stirring at same temp for 2 h. After 2 h, reaction mixture was diluted with CH_2Cl_2 and separated organic layer was washed with NaHCO_3 (2×5 mL) and water. Separated organic layer was dried over Na_2SO_4 and purified by silica gel column chromatography (100-200 mesh, 15% EtOAc-petroleum ether) to give **5** as a white sticky solid (0.114 g, 71%). $[\alpha]_D^{25} +5.30$ ($c = 0.25$, CHCl_3); **IR** (cm^{-1} , CHCl_3) ν 2923, 2861, 1749, 1498, 1357, 1169, 1054, 917, 696; **1H NMR** (400 MHz, CDCl_3) δ 7.52-7.49 (m, 2H, ArH), 7.36-7.21 (m, 18H, ArH), 5.03-4.99 (m, 1H, H-2), 4.85-4.79 (m, 2H, CH_2Ph), 4.66-4.53 (m, 5H, H-1), 3.90, 3.75 (ABq, $J = 14.8$ Hz, 2H, CH_2), 3.85-3.66 (m, 4H, H-3, H-4, H-6, H-6'), 3.57-3.54 (m, 1H, H-5); **13C NMR** (100 MHz, CDCl_3) δ 165.9, 138.1, 138.0, 137.7, 132.6, 132.3, 128.9, 128.5, 128.5, 128.4, 128.0, 128.0, 127.9, 127.7, 127.6, 85.6, 84.0, 79.4, 77.9, 75.4, 75.1, 73.5, 73.2, 68.7, 40.7; **HR-ESI-MS** (m/z): $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{35}\text{H}_{35}\text{ClO}_6\text{SNa}$, 641.1735; found, 641.1733.

Phenyl-2-*O*-chloroacetyl-3,4-di-*O*-benzyl-6-*O*-fluorenylmethyloxycarbonyl-1-thio- β -D-glucopyranoside (23)



Compound **22** (90 mg, 0.17 mmol) was dissolved in CH₂Cl₂ (2 mL) and to this solution, pyridine (55 μ L, 0.68 mmol), and FmocCl (88 mg, 0.34 mmol) were added at 0 °C. After 3 h, the reaction mixture was concentrated in *vacuo* to remove pyridine (confirmed by TLC) reaction mixture was diluted with EtOAc and organic layer was washed with dil. HCl (3×15 mL) and NaHCO₃ (2 \times 10 mL). Separated organic layer was dried over Na₂SO₄ and purified by silica gel column chromatography (100-200 mesh, 20% ethyl acetate: pet ether) to give **23** as a white solid. (98 mg, 77%). $[\alpha]_D^{25} +31.12$ ($c = 0.5$, CHCl₃); **M.P.** 159-162 °C; **IR** (cm⁻¹, CHCl₃) ν 3571, 3486, 2926, 1746, 1377, 1296, 1192, 1042, 965, 767, 667, 580; **¹H NMR** (400 MHz, CDCl₃) δ 7.81 (d, 2H, ArH), 7.66 (t, $J = 9.2$ Hz, 2H, ArH), 7.53-7.27 (m, 18H, ArH), 5.05 (t, $J = 9.1$ Hz, 1H, H-2), 4.88 (dd, $J = 7.91, 2.92$ Hz, 2H), 4.70 (d, $J = 11.5$ Hz, 1H), 4.65 (dd, $J = 7.24, 2.88$ Hz, 2H, H-1), 4.51 (d, 1H), 4.49-4.42 (m, 2H, H-6, H-6'), 4.33-4.28 (m, 2H, H-5), 3.93 (d, $J = 11.4$ Hz, 1H), 3.81-3.75 (m, 2H, H-3), 3.66 (m, 2H, H-4). **¹³C NMR** (100 MHz, CDCl₃): δ 165.9, 154.9, 143.4, 143.3, 141.3, 137.8, 137.3, 132.7, 132.1, 128.9, 128.6, 128.6, 128.2, 128.2, 128.0, 127.9, 127.3, 125.2, 125.2, 120.1, 85.7, 84.1, 77.5, 75.5, 75.3, 73.1, 70.0, 66.4, 46.7, 40.67. **HR-ESI-MS** (*m/z*): [M + Na]⁺ calcd. for C₄₃H₄₀ClO₇SNa, 758.2081; found, 758.2084.

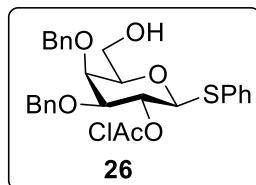
Phenyl-4,6-*O*-benzylidene-3-*O*-benzyl-2-*O*-chloroacetyl-1-thio- β -D-galactopyranoside



Compound **25** (0.35 g, 0.77 mmol) was dissolved in CH₂Cl₂ (6.2 mL). To this clear solution pyridine (0.18 mL, 2.32 mmol) and ClAcCl (0.18 mL, 2.32 mmol) were added. After 3 h, complete consumption of starting material (confirmed by TLC), reaction solvent was removed under reduced pressure and the obtained crude product was purified by silica gel column

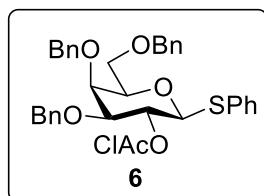
chromatography (100-200 mesh, 25% EtOAc-petroleum ether) to afford fully protected galactopyranoside as a white solid (0.35 g, 86%). $[\alpha]_D^{25} -0.30$ ($c = 1.0$, CHCl₃); **IR** (cm⁻¹, CHCl₃) ν 2921, 1757, 1737, 1364, 1217, 1185, 1169, 1095, 1073, 1027, 775, 749, 729, 697; **¹H NMR** (500 MHz, CDCl₃) δ 7.63-7.62 (m, 2H, ArH), 7.44-7.41 (m, 2H, ArH), 7.39-7.26 (m, 11H, ArH), 5.47 (s, 1H, CHPh), 5.37 (t, $J = 9.6$ Hz, 1H, H-2), 4.70, 4.59 (ABq, $J = 12.5$ Hz, 2H, CH₂Ph), 4.67 (d, $J = 9.8$, 1H, H-1), 4.37 (dd, $J = 10.9, 1.4$ Hz, 1H, H-6'), 4.22 (d, $J = 3.1$ Hz, 1H, H-4), 4.07-3.98 (m, 3H, H-6, CH₂), 3.68 (dd, $J = 6.2, 3.3$ Hz, 1H, H-3), 3.47 (s, 1H, H-5); **¹³C NMR** (125 MHz, CDCl₃) δ 165.6, 137.7, 137.5, 133.7, 131.1, 129.1, 128.8, 128.4, 128.1, 127.9, 127.7, 126.5, 101.2, 84.8, 78.3, 73.0, 71.3, 70.1, 70.0, 69.2, 40.82; **HR-ESI-MS** (m/z): [M + Na]⁺ calcd. for C₂₈H₂₇ClO₆SNa, 549.1109; found, 549.1113.

Phenyl-2-O-chloroacetyl-3,4-di-O-benzyl-1-thio- β -D-galactopyranoside (26)



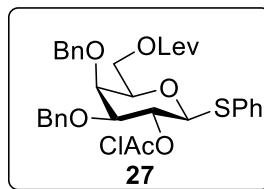
BH₃·THF (1 M, 2 mL, 1.47 mmol) and TMSOTf (18 μ L, 0.14 mmol) were sequentially added to a solution of the fully protected galactopyranoside (0.26 g, 0.50 mmol) in CH₂Cl₂ (3 mL) at 0 °C. After 2 h, reaction mixture quenched with MeOH (2 mL) and Et₃N, concentrated, and purified by silica gel column chromatography (100-200 mesh, 30% ethyl acetate: pet ether) to give **26** as a white solid (0.26 g, 91%). $[\alpha]_D^{25} +0.08$ ($c = 0.25$, CHCl₃); **M.P.** 144-145 °C; **IR** (cm⁻¹, CHCl₃) ν 3571, 3471, 2926, 1734, 1359, 1161, 1108, 1053, 754, 739, 698; **¹H NMR** (400 MHz, CDCl₃) δ 7.48-7.24 (m, 15H, ArH), 5.47 (t, $J = 9.7$ Hz, 1H, H-2), 4.95 (d, $J = 11.7$ Hz, 1H), 4.73-4.54 (m, 4H, H-1), 4.02-3.90 (m, 3H, H-4, H-6'), 3.84 (dd, $J = 10.49, 1.44$ Hz, 1H), 3.62 (dd, $J = 7.34, 4.70$ Hz, 1H), 3.63-3.60 (m, 2H, H-3), 3.57-3.48 (m, 2H, H-5, H-6), 1.80 (bs, 1H, -OH). **¹³C NMR** (100 MHz, CDCl₃): δ 165.9, 137.9, 137.5, 132.9, 132.0, 128.9, 128.6, 128.5, 128.3, 128.1, 127.9, 127.8, 127.7, 86.2, 81.4, 79.1, 74.2, 72.4, 72.3, 71.5, 62.0, 40.8. **HR-ESI-MS** (m/z): [M + Na]⁺ calcd. for C₂₈H₂₉ClO₆SNa, 551.1266; found, 551.1248.

Phenyl-2-*O*-chloroacetyl-3,4,6-tri-*O*-benzyl-1-thio- β -D-galactopyranoside (6)



Benzyl trichloroimide (45 μ L, 0.37 mmol) was added to a stirred solution of **26** (0.14 g, 0.26 mmol) in CH₂Cl₂ (0.53 mL). TfOH (7.5 mg, 0.31 mmol) was added at 0 °C and reaction mixture was kept stirring at same temp for 1 h. After 1 h, reaction mixture was diluted with CH₂Cl₂ and separated organic layer was washed with NaHCO₃ (2 \times 50 mL) and water. Separated organic layer was dried over Na₂SO₄ and purified by silica gel column chromatography (100-200 mesh, 15% EtOAc-petroleum ether) to give **6** as a white solid (0.15 mg, 76%). $[\alpha]_D^{25} +16.43$ ($c = 1.0$, CHCl₃); **M.P.** 159-162 °C **IR** (cm⁻¹, CHCl₃) ν 3035, 2881, 1758, 1454, 1409, 1364, 1216, 1191, 1150, 1114, 1063, 770, 734, 695; **¹H NMR** (500 MHz, CDCl₃) δ 7.55-7.53 (m, 2H, ArH), 7.41-7.26 (m, 18H, ArH), 5.52 (t, $J = 9.7$ Hz, 1H, H-2), 4.98 (d, $J = 11.5$ Hz, 1H, CH₂Ph), 4.74-4.69 (m, 3H, H-1), 4.56-4.47 (m, 3H), 4.06 (d, $J = 2.3$ Hz, 1H, H-4), 3.99 (ABq, $J = 14.7$ Hz, 2H), 3.74-3.68 (m, 3H, H-5, H-6, H-6'), 3.65 (dd, $J = 7.0$, 2.5 Hz, 1H, H-3); **¹³C NMR** (125 MHz, CDCl₃) δ 166.0, 138.4, 137.8, 137.7, 133.1, 132.1, 128.9, 128.6, 128.5, 128.3, 128.0, 128.0, 127.9, 127.7, 127.7, 127.6, 86.2, 81.3, 76.9, 74.5, 73.6, 72.8, 72.1, 71.5, 68.7, 40.93; **HR-ESI-MS** (*m/z*): [M + Na]⁺ calcd. for C₃₅H₃₅ClO₆SNa, 641.1735; found, 641.1720.

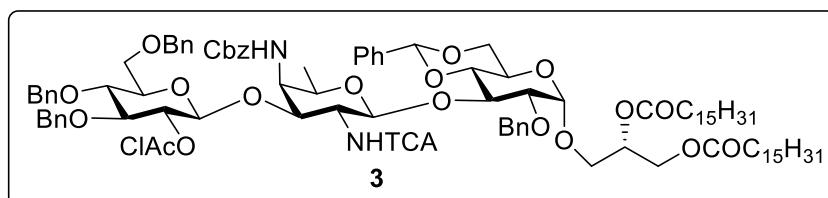
Phenyl-2-*O*-chloroacetyl-3,4-di-*O*-benzyl-6-*O*-Levulinoyl-1-thio- β -D-galactopyranoside (27)



Compound **26** (60 mg, 0.11 mmol) was dissolved in CH₂Cl₂ (1 mL). Sequentially LevOH (17.3 μ L, 0.17 mmol), EDC·HCl (51 mg, 0.33 mmol) and DMAP (6.3 mg, 0.05 mmol) were added at rt. The resulting mixture was stirred at room temperature for 2 h. Reaction mixture was diluted with EtOAc and organic layer was washed with dil. HCl (3×15 mL) and NaHCO₃ (2 \times 10 mL). Separated organic layer was dried over Na₂SO₄ and purified by silica gel column chromatography (100-200 mesh, 30% ethyl acetate: pet ether) to give **27** as a yellowish liquid.

(66 mg, 93%). $[\alpha]_D^{25} +7.46$ ($c = 0.5$, CHCl_3); **IR** (cm^{-1} , CHCl_3) ν 3572, 3349, 2926, 1757, 1734, 1453, 1411, 1291, 1214, 1192, 1175, 1047, 1030, 770, 746, 735, 693, 666; **$^1\text{H NMR}$** (500 MHz, CDCl_3) δ 7.48-7.28 (m, 15H, ArH), 4.99 (t, $J = 9.20$ Hz, 1H, H-2), 4.83 (d, $J = 11.1$ Hz, CH_2Ph), 4.67-4.59 (m, 3H, CH_2Ph , H-1), 4.43-4.40 (dd, $J = 10.9, 0.8$ Hz, 1H, H-6), 4.23-4.19 (m, 1H, H-6'), 3.90 (d, $J = 7.3, 4.6$ Hz, 1H, CH_2Lev), 3.77-3.70 (m, 2H, CH_2Lev , H-3), 3.58 (m, 1H, H-4), 2.074 (t, $J = 6.7$ Hz, 2H, CH_2Lev), 2.58 (m, 2H, CH_2Lev), 2.18 (s, 3H, CH_2Lev). **$^{13}\text{C NMR}$** (125 MHz, CDCl_3): δ 206.3, 172.3, 165.9, 137.8, 137.3, 132.7, 132.1, 128.9, 128.5, 128.2, 128.1, 128.0, 85.5, 84.0, 77.5, 77.1, 75.5, 75.2, 73.1, 62.9, 40.6, 37.8, 29.8, 27.8. **HR-ESI-MS** (m/z): [M + Na]⁺ calcd. for $\text{C}_{33}\text{H}_{36}\text{ClO}_8\text{SNa}$, 650.1717; found, 650.1718.

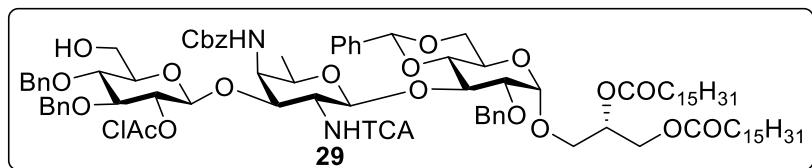
3-O-[(2-O-chloroacetyl-3,4,6-tri-O-benzyl-1- β -glucopyranosyl)-(1 \rightarrow 3)-(2-trichloroacetamido-2,4,6-trideoxy-4-N-benzylcarbamate-1- β -galactopyranosyl)-(1 \rightarrow 3)-(4,6-O-benzylidine-2-benzyl-1- α -glucopyranosyl)]-1,2-di-O-Palmitoyl -sn-glycerol (3)



To the donor **5** (58 mg, 0.09 mmol), disaccharide acceptor **7** (59 mg, 0.04 mmol), 3 Å MS (0.24 g) was added and stirred in anhydrous CH_2Cl_2 (2.2 mL) for 45 min. NIS (43 mg, 0.19 mmol) and TMSOTf (1.8 μL , 0.01 mmol) was added at 0 °C. The reaction mixture was stirred at same temp under nitrogen atmosphere for 2 h. The reaction was quenched by adding NEt_3 (1-2 drops). The reaction mixture was diluted with CH_2Cl_2 , filtered through Celite and transferred to a separating funnel. The organic layer was washed with aq. $\text{Na}_2\text{S}_2\text{O}_3$ (2×3 mL), the separated organic layer was dried over Na_2SO_4 and concentrated. The crude product was purified by silica gel column chromatography (230-400 mesh, 40% EtOAc-petroleum ether) to afford **3** as a white foam (63 mg, 77%). $[\alpha]_D^{25} +5.96$ ($c = 0.1$, CHCl_3); **IR** (cm^{-1} , CHCl_3) ν 3403, 2924, 2871, 1733, 1654, 1245, 1047, 770; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 7.46-7.18 (30H, ArH), 6.63 (d, $J = 8.4$ Hz, 1H, -NH), 5.48 (s, 1H, CHPh), 5.19-5.03 (m, 4H), 4.96 (t, $J = 8.2$ Hz, 1H), 4.80-4.68 (m, 4H, H-1'), 4.63-4.46 (m, 8H, H-1, H-1''), 4.36 (dd, $J = 8.2, 3.6$ Hz, 1H), 4.21-4.15 (m, 2H), 4.10-3.99 (m, 3H), 3.77-3.62 (m, 6H), 3.59-3.40 (m, 8H), 2.32-2.35 (m, 4H), 1.59 (m, 4H), 1.26 (bs, 48H), 1.09 (d, $J = 6.3$ Hz, 3H, H-6'), 0.88 (t, 6H); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3) δ 173.3, 173.0, 166.0, 161.6, 156.6, 138.3, 138.2, 137.9, 137.6, 137.1, 129.0, 128.6, 128.5, 128.4, 128.1, 128.1, 128.0, 127.9, 127.8, 127.7, 127.6, 126.0, 101.3, 100.2, 100.1,

100.1, 98.1, 92.5, 82.2, 80.5, 79.2, 78.2, 77.2, 75.4, 75.1, 75.0, 74.4, 74.1, 73.4, 73.3, 70.1, 69.6, 69.0, 68.8, 66.9, 66.3, 62.5, 62.3, 56.2, 54.7, 40.9, 34.2, 34.1, 31.9, 30.3, 29.9, 29.7, 29.6, 29.6, 29.5, 29.3, 29.3, 29.29, 29.1, 29.1, 27.3, 24.9, 24.8, 22.7, 16.5, 14.1; **HR-ESI-MS** (*m/z*): [M + Na]⁺ calcd. for C₁₀₀H₁₃₄O₂₁Cl₄N₂Na, 1861.8125; found, 1861.8107.

3-O-[(2-O-chloroacetyl-3,4-di-O-benzyl-1- β -glucopyranosyl)-(1 \rightarrow 3)-(2-trichloroacetamido-2,4,6-trideoxy-4-N-benzylcarbamate-1- β -galactopyranosyl)-(1 \rightarrow 3)-(4,6-O-benzylidine-2-benzyl-1- α -glucopyranosyl)]-1,2-di-O-Palmitoyl -sn-glycerol (29)

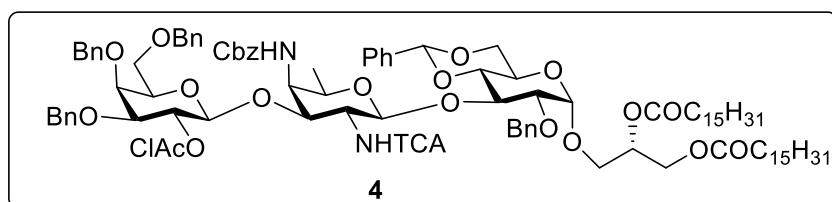


To a suspension of donor **23** (41 mg, 0.05 mmol), acceptor **7** (44 mg, 0.03 mmol) and 3 Å MS (0.1 g) in CH₂Cl₂ (2.5 mL) was stirred under nitrogen atmosphere at rt for about 0.5 h. The flask was cooled to 0 °C and NIS (25 mg, 0.11 mmol) was added followed by addition of TMSOTf (2 μ L, 0.005 mmol) at same temp. The reaction mixture was stirred at 0 °C under nitrogen atmosphere for 2 h. After 1 h, Et₃N (0.2 mL) was added to the reaction mixture for removal of Fmoc group as well as for quenching of TMSOTf in the reaction mixture. After completion of the reaction (confirmed by TLC) reaction mixture was filtered through Celite and transferred to a separating funnel. The organic layer was washed with aq. Na₂S₂O₃ (2 \times 5 mL), the separated organic layer was dried over Na₂SO₄ and concentrated. The crude product was purified by silica gel column chromatography (230-400 mesh, 40% ethyl acetate: pet ether) to afford **29** as a white foam (37mg, 66% over 2 steps). $[\alpha]_D^{25} +31.12$ (*c* = 0.5, CHCl₃); **IR** (cm⁻¹, CHCl₃) ν 3571, 3486, 2926, 1746, 1377, 1296, 1192, 1042, 965, 767, 667, 580; **¹H NMR** (500 MHz, CDCl₃) δ 7.48-7.26 (m, 25H, ArH), 6.88 (m, 1H, NHTCA), 5.51 (s, 1H, CHPh), 5.23-5.12 (m, 4H, CH₂Ph), 5.01 (d, 1H), 4.90-4.78 (m, 3H), 4.69-4.61 (m, 3H), 4.57-4.48 (m, 3H), 4.39 (dd, *J* = 8.0, 3.5 Hz, 1H), 4.26 (dd, *J* = 5.5, 5.0 Hz, 1H), 4.22-4.19 (m, 2H), 4.11 (t, *J* = 9.5 Hz, 1H), 3.92-3.84 (m, 2H), 3.77-3.46 (m, 10H), 3.29 (d, 1H), 3.09 (m, 1H), 2.35-2.30 (m, 4H, CH₂Linker), 1.64 (bs, 1H, CH₂Linker), 1.27 (bs, 48H), 1.05 (d, *J* = 6 Hz, 3H), 0.90 (t, *J* = 6.5 Hz, 6H, CH₂Linker). **¹³C NMR** (125 MHz, CDCl₃): δ 173.1, 173.0, 166.1, 161.9, 157.2, 138.1, 138.1, 137.8, 137.1, 136.6, 129.6, 129.1, 128.7, 128.6, 128.5, 128.4, 128.3, 128.1, 128.1, 128.1, 127.9, 127.9, 127.7, 127.6, 127.6, 126.0, 101.5, 100.8, 99.3, 98.1, 82.0, 81.0, 79.1, 77.4, 77.1, 75.4, 75.1, 75.1, 75.0, 74.6, 73.8, 70.0, 69.6, 68.8, 67.0, 66.4, 62.4, 62.2, 60.6, 57.6, 55.2,

40.8, 34.2, 34.1, 31.9, 29.7, 29.6, 29.5, 29.3, 29.2, 29.2, 29.1, 29.1, 24.9, 24.9, 22.6, 16.4, 14.1.

HR-ESI-MS (*m/z*): [M + K]⁺ calcd. for C₉₃H₁₂₈Cl₄N₂KO₂₁, 1787.7395; found, 1787.7398.

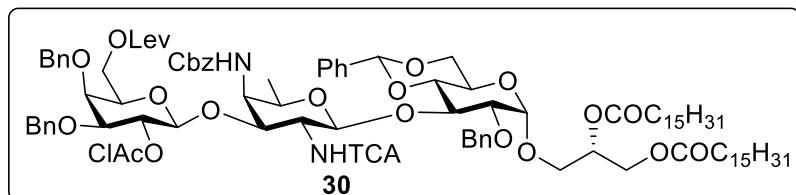
3-*O*-[(2-*O*-chloroacetyl-3,4,6-tri-*O*-benzyl-1- β -galactopyranosyl)-(1 \rightarrow 3)-(2-trichloroacetamido-3-hydroxy-2,4,6-trideoxy-4-benzylcarbamate-1- β -galactopyranosyl)-(1 \rightarrow 3)-(4,6-*O*-benzylidine-2-benzyl-1- α -glucopyranosyl)]-1,2-di-*O*-Palmatoyl-*sn*-glycerol (4)



To the donor **6** (60 mg, 0.1 mmol) and acceptor **7** (63 mg, 0.05 mmol) 3 Å MS (0.2 g) was added and stirred in anhydrous CH₂Cl₂ (2.2 mL) for 45 min. The flask was cooled to 0 °C and NIS (45 mg, 0.2 mmol) was added followed by addition of TMSOTf (2 μ L, 0.01 mmol) at same temp. The reaction mixture was stirred at 0 °C under nitrogen atmosphere for 2 h. The reaction was quenched by adding NEt₃ (1-2 drops). The reaction mixture was diluted with CH₂Cl₂, filtered through Celite and transferred to a separating funnel. The organic layer was washed with aq. Na₂S₂O₃ (2 \times 8 mL), the separated organic layer was dried over Na₂SO₄ and concentrated. The crude product was purified by silica gel column chromatography (230-400 mesh, 40% EtOAc-petroleum ether) to afford **4** as a white foam (63 mg, 73%). $[\alpha]_D^{25} +14.78$ (*c* = 1.0, CHCl₃); **IR** (cm⁻¹, CHCl₃) ν 3630, 2925, 2854, 1731, 1457, 1259, 1095, 824, 699, 607, 485; **1H NMR** (500 MHz, CDCl₃) δ 7.45-7.24 (m, 30H, ArH), 6.70 (d, *J* = 7.7 Hz, 1H, -NH), 5.47 (s, 1H, CHPh), 5.32 (t, *J* = 8.3 Hz, 1H, H-2''), 5.15 (m, 1H), 5.13, 4.99 (ABq, *J* = 12.2 Hz, 2H, CH₂Ph), 5.05 (d, *J* = 9.8 Hz, 1H), 4.90 (d, *J* = 11.6 Hz, 1H), 4.83 (d, *J* = 8.1 Hz, 1H, H-1'), 4.71-4.46 (m, 6H, H-1, H-1''), 4.43-4.35 (m, 4H), 4.23-4.15 (m, 3H), 4.09-4.02 (m, 3H), 3.93-3.83 (m, 2H), 3.74-3.62 (m, 5H), 3.55-3.38 (m, 7H), 2.31-2.26(m, 4H), 1.59 (m, 4H), 1.25 (bs, 48H), 1.05 (d, *J* = 6.1 Hz, 3H, H-6'), 0.88 (t, *J* = 6.8 Hz, 6H); **13C NMR** (125 MHz, CDCl₃) δ 173.3, 173.0, 166.2, 161.7, 156.5, 138.4, 138.3, 137.8, 137.5, 137.1, 136.6, 129.0, 128.6, 128.5, 128.4, 128.2, 128.1, 127.9, 127.8, 127.6, 127.5, 126.0, 101.3, 100.2, 100.0, 98.1, 92.4, 80.6, 79.9, 79.2, 74.4, 73.8, 73.6, 73.5, 72.4, 72.2, 71.8, 70.2, 69.6, 68.8, 68.2, 66.7, 66.3, 62.4, 62.3, 56.5, 54.2, 41.1, 37.1, 34.2, 34.1, 32.7, 31.9, 30.0, 29.7, 29.6, 29.5, 29.3, 29.3,

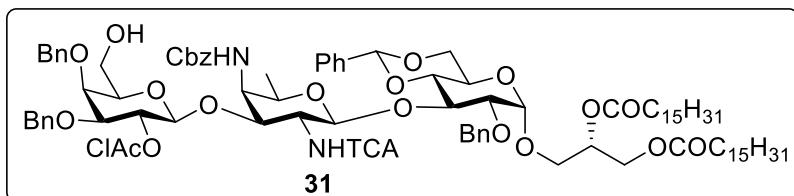
29.3, 29.1, 29.1, 27.1.24.9, 24.8, 22.7, 16.5, 14.1; **HR-ESI-MS** (m/z): [M + Na]⁺ calcd. for C₁₀₀H₁₃₄O₂₁Cl₄N₂Na, 1861.8125; found, 1861.8183.

3-O-[(2-O-chloroacetyl-3,4-di-O-benzyl-6-O-levulinoyl-1- β -galactopyranosyl)-(1 \rightarrow 3)-(2-trichloroacetamido-2,4,6-trideoxy-4-N-benzylcarbamate-1- β -galactopyranosyl)-(1 \rightarrow 3)-(4,6-O-benzylidine-2-benzyl-1- α -glucopyranosyl)]-1,2-di-O-Palmitoyl-sn-glycerol (30)



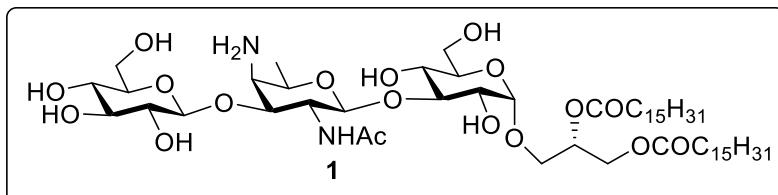
Freshly vacuum dried MS (0.16 g) were added to a clear solution of **27** (30 mg, 0.047 mmol) and **7** (50 mg, 0.042 mmol) in CH₂Cl₂ (2 mL), and the resultant turbid solution was stirred at room temperature for 30 min. Then NIS (21 mg, 0.095 mmol) and TMSOTf (0.85 μ L, 0.005 mmol) were added at 0 °C. After stirring the reaction mixture at the same temperature for 1 h, Et₃N (0.2 mL) was added. Reaction mixture was diluted with CH₂Cl₂, filtered through Celite and transferred to a separating funnel. The organic layer was washed with aq. Na₂S₂O₃ (2 \times 3 mL), the separated organic layer was dried over Na₂SO₄ and concentrated. The crude product was purified by silica gel column chromatography (230-400 mesh, 40% ethyl acetate: pet ether) to afford **30** as a yellowish liquid (62 mg, 91%). $[\alpha]_D^{25} +5.98$ ($c = 1$, CHCl₃); **IR** (cm⁻¹, CHCl₃) ν 3571, 3471, 2926, 2854, 1735, 1530, 1496, 1455, 1402, 1369, 1215, 1183, 1161, 1107, 1083, 1030, 822, 758, 699, 667; **¹H NMR** (500 MHz, CDCl₃) δ 7.66-7.88 (m, 25H, ArH), 5.47 (s, 1H, CHPh), 5.31 (t, $J = 9.20$ Hz, 1H, H-2''), 5.23-5.14 (m, 3H, CH₂Ph), 4.98 (d, $J = 11.5$ Hz, 1H), 4.82 (d, $J = 10.9$ Hz, 1H), 4.68-4.61 (m, 3H), 4.68-4.31 (m, 8H), 4.19-4.11 (m, 5H), 4.08-4.01 (m, 2H), 3.88 (m, 1H), 3.77 (bs, 1H), 3.66-3.55 (m, 4H), 3.44-3.34 (m, 4H), 2.82-2.77 (m, 1), 2.34-2.24 (m, 6H, CH₂Lev), 2.13 (s, 3H, CH₃Lev), 1.62 (bs, 4H, CH₂Linker), 1.27 (bs, 49H), 1.17 (d, $J = 6.1$ Hz, H-6'), 0.90 (t, $J = 6.6$ Hz, CH₃Linker). **¹³C NMR** (125 MHz, CDCl₃): δ 205.1, 173.4, 172.9, 172.4, 161.6, 138.7, 137.8, 137.4, 136.6, 129.1, 128.5, 128.4, 128.3, 128.1, 128.0, 127.9, 127.9, 127.8, 125.8, 101.1, 100.6, 100.5, 98.6, 93.0, 81.2, 79.7, 78.2, 78.0, 74.2, 74.1, 73.3, 72.4, 72.3, 71.8, 71.6, 70.6, 69.6, 68.9, 67.1, 66.3, 63.6, 62.3, 62.3, 54.5, 41.1, 37.8, 34.2, 34.1, 31.9, 29.9, 29.7, 29.6, 29.5, 29.4, 29.3, 29.2, 29.1, 29.1, 27.6, 24.9, 24.9, 22.7, 22.6, 16.7, 14.1. **HR-ESI-MS** (m/z): [M + Na]⁺ calcd. for C₉₈H₁₃₄C₁₄N₂NaO₂₃, 1869.8024; found, 1869.8028.

3-O-[(2-O-chloroacetyl-3,4-di-O-benzyl-1- β -galactopyranosyl)-(1→3)-(2-trichloroacetamido-2,4,6-trideoxy-4-N-benzylcarbamate-1- β -galactopyranosyl)-(1→3)-(4,6-O-benzylidine-2-benzyl-1- α -glucopyranosyl)]-1,2-di-O-Palmitoyl -sn-glycerol (31)



Compound **30** (19 mg, 0.01 mmol) was dissolved in CH₂Cl₂ (0.7 mL) and Py:AcOH (4:1, 1 mL) sequentially NH₂NH₂·H₂O (0.3 mg, 0.01 mmol) were added at rt. The resulting mixture was stirred at room temperature for 1.5 h. Reaction mixture was diluted with EtOAc concentrated and purified by silica gel column chromatography (100-200 mesh, 10% ethyl acetate: pet ether) to give **31** as a yellowish liquid. (16 mg, 88%). [α]_D²⁵ +11.24 (c = 0.5, CHCl₃); **IR** (cm⁻¹, CHCl₃) ν 3571, 3470, 2926, 1716, 1457, 1376, 1290, 1216, 1072, 772, 702, 569, 544; **1H NMR** (500 MHz, CDCl₃) δ 7.46-7.29 (m, 25), 5.50 (s, 1H, CHPh), 5.40-5.32 (m, 2H), 5.16-5.13 (m, 2H, CH₂Ph), 5.02-5.98 (m, 2H, CH₂Ph), 4.87 (d, *J* = 12.6 Hz, 1H), 4.70-4.63 (m, 2H), 4.63-4.55 (m, 4H), 4.48 (m, 1H), 4.40 (dd, 1H), 4.22-4.12 (m, 4H), 4.02-4.97 (m, 1H), 3.88-3.67 (m, 6H), 3.58 (m, 2H), 3.51-3.40 (m, 5H), 3.19 (m, 1H), 2.33 (m, 4H, CH₂Linker), 1.63 (bs, 4H, CH₂Linker), 1.28 (bs, 49H), 1.03 (d, *J* = 6.1 Hz, 3H), 0.90 (t, *J* = 6.5 Hz, 6H, CH₃Linker). **13C NMR** (125 MHz, CDCl₃): δ 173.3, 173.0, 166.3, 161.9, 157.7, 138.1, 137.9, 137.4, 137.0, 136.2, 129.1, 128.8, 128.6, 128.5, 128.3, 128.2, 128.2, 128.1, 128.0, 127.9, 127.8, 127.6, 126.0, 101.9, 101.5, 99.4, 98.1, 80.9, 80.7, 79.3, 77.5, 76.4, 75.8, 74.3, 73.8, 72.9, 72.8, 72.3, 70.2, 69.6, 68.8, 67.1, 66.3, 62.4, 62.3, 62.2, 57.4, 54.6, 41.1, 34.3, 34.1, 31.9, 29.7, 29.6, 29.5, 29.3, 29.2, 29.1, 24.9, 24.9, 24.8, 22.7, 16.5, 14.1. **HR-ESI-MS** (*m/z*): [M + K]⁺ calcd. for C₉₃H₁₂₈Cl₄N₂KO₂₁, 1787.7395; found, 1787.7384.

3-O-[(β -D-Glucopyranosyl)-(1→3)-(2-acetylamino-4-amino-2,4,6-trideoxy- β -D-galactopyranosyl)-(1→3)- α -D-glucopyranosyl)]-1,2-di-O-Palmitoyl-sn-glycerol (1)

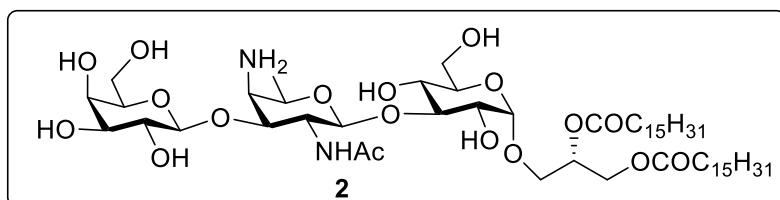


Thiourea (16 mg, 0.2 mmol) was added to a clear solution of **3** (55 mg, 0.03 mmol) in pyridine (0.38 mL) and EtOH (0.38 mL) and the reaction mixture was kept for reflux at 80 °C.

After 45 min, solvent was removed under reduced pressure and the crude product was dried under high vacuum for 3 h.

The crude compound was dissolved EtOH (4 mL). To this solution Pd(OH)₂/C (0.11 g, 20% Pd content) was added. The reaction was stirred under an atmosphere of hydrogen for 8 h. The reaction mixture was filtered through a celite pad with methanol as eluents. Filtrate was concentrated under reduced pressure and the crude product was purified by silica gel column chromatography using methanol:chloroform (1:9) mixture as eluent to give **1** as white foam (25 mg, 79%). $[\alpha]_D^{25} +58.30$ ($c = 0.1$, CHCl₃); **IR** (cm⁻¹, CHCl₃) ν 3421, 2920, 2850, 2509, 1645, 1467, 1116, 1037, 973, 771; **¹H NMR** (500 MHz, CDCl₃) δ 5.28 (bs, 1H), 4.83-4.81 (m, 2H), 4.50-4.44 (m, 2H), 4.23 (m, 1H), 4.13 (bs, 1H), 3.97-3.75 (m, 6H), 3.71-3.51 (m, 5H), 3.40-3.28 (m, 7H), 2.35 (m, 4H), 2.01 (s, 3H, COCH₃), 1.62 (bs, 4H), 1.31 (bs, 51H), 0.92 (t, $J = 6.2$ Hz, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 172.0, 171.8, 171.7, 103.3, 99.9, 97.8, 80.2, 75.7, 75.4, 74.7, 71.6, 70.7, 70.1, 68.6, 68.2, 66.9, 65.4, 64.3, 60.8, 59.5, 53.3, 49.8, 32.2, 32.0, 30.1, 27.9, 27.8, 27.7, 27.5, 27.3, 23.1, 20.8, 20.2, 13.8, 11.5; **HR-ESI-MS** (m/z): [M + H]⁺ calcd. for C₅₅H₁₀₃O₁₈N₂, 1079.7200; found, 1079.7200.

3-O-[$(\beta$ -D-Galactopyranosyl)-(1 \rightarrow 3)-(2-acetylamino-4-amino-2,4,6-trideoxy- β -D-galactopyranosyl)-(1 \rightarrow 3)- α -D-glucopyranosyl]-1,2-di-O-Palmitoyl-sn-glycerol (2)



Thiourea (14 mg, 0.19 mmol) was added to a clear solution of **4** (50 mg, 0.027 mmol) in pyridine (0.35 mL) and EtOH (0.35 mL) and the reaction mixture was kept for reflux at 80 °C. After 45 min, solvent was removed under reduced pressure and the crude product was dried under high vacuum for 3 h.

The crude compound was dissolved in EtOH (3.5 mL). To this solution Pd(OH)₂/C (0.1 g, 20% Pd content) was added. The reaction was stirred under an atmosphere of hydrogen for 8 h. The reaction mixture was filtered through a celite pad with methanol as eluents. Filtrate was concentrated under reduced pressure and the crude product was purified by silica gel column chromatography using methanol:chloroform (1:9) mixture as eluent to give **2** as white foam (24 mg, 81 %). $[\alpha]_D^{25} = 52.41$ ($c = 0.1$, CHCl₃); **IR** (cm⁻¹, CHCl₃) ν 3454, 2527, 1660, 1218, 973, 771; **¹H NMR** (400 MHz, CDCl₃) δ 5.26 (bs, 1H), 4.78 (m, 2H), 4.47 (d, $J = 9.8$

Hz, 1H, H-1), 4.39 (d, $J = 7.4$ Hz, 1H), 4.22-4.11 (m, 2H), 3.94-3.64 (m, 10H), 3.55-3.44 (m, 4H), 3.36 (m, 2H) 3.21 (m, 1H), 2.35-2.30 (m, 4H), 1.99 (s, 3H, COCH₃), 4.60 (bs, 4H), 1.28 (bs, 51H), 0.89 (t, $J = 6.0$ Hz, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 173.6, 173.4, 173.2, 105.5, 101.5, 99.3, 81.9, 77.2, 75.6, 73.0, 72.1, 71.6, 70.8, 70.1, 68.8, 68.4, 67.0, 65.8, 62.3, 61.3, 60.9, 54.9, 51.4, 33.7, 33.5, 31.6, 29.4, 29.3, 29.2, 29.0, 28.8, 24.6, 24.6, 22.3, 21.8, 15.4, 13.0; **HR-ESI-MS** (*m/z*): [M + H]⁺ calcd. for C₅₅H₁₀₃O₁₈N₂, 1079.7200; found, 1079.7210.

II. Spectra:

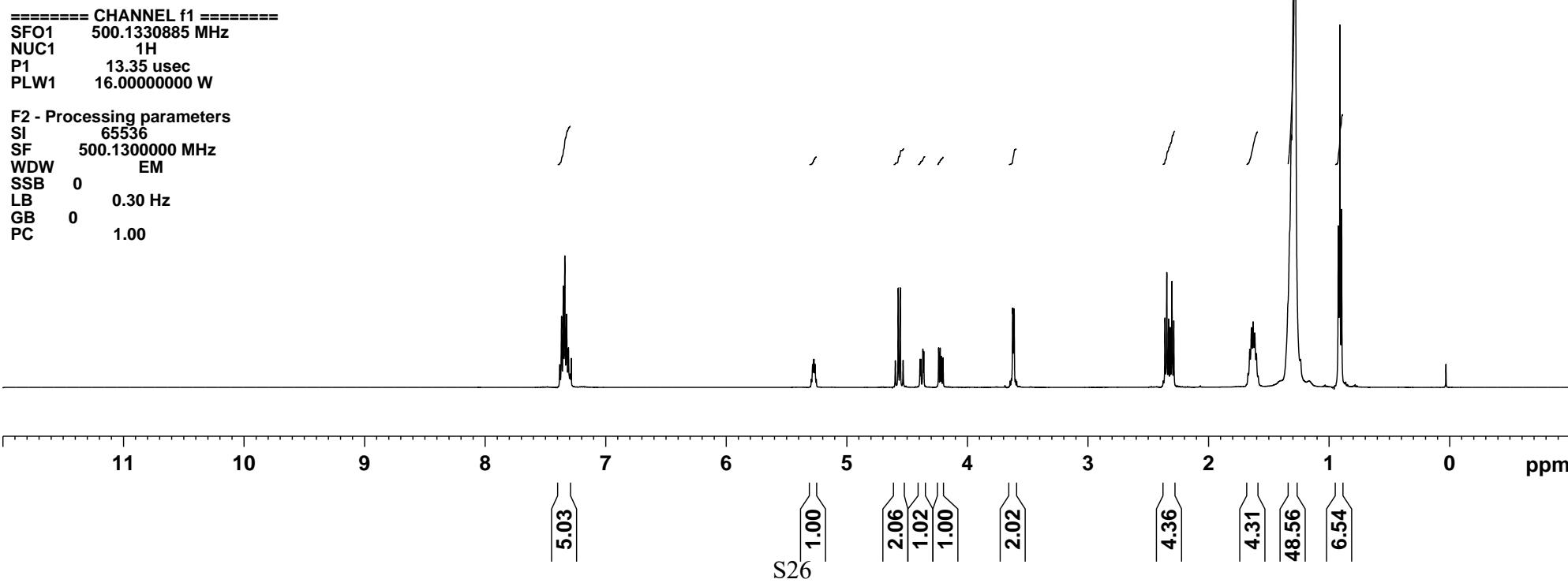
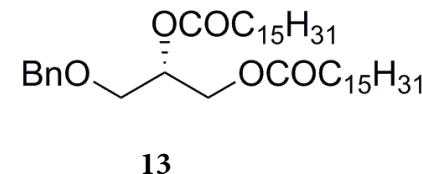
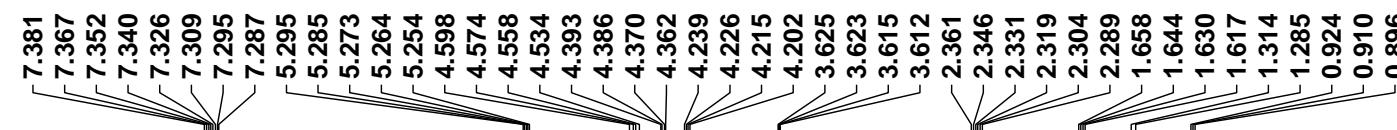
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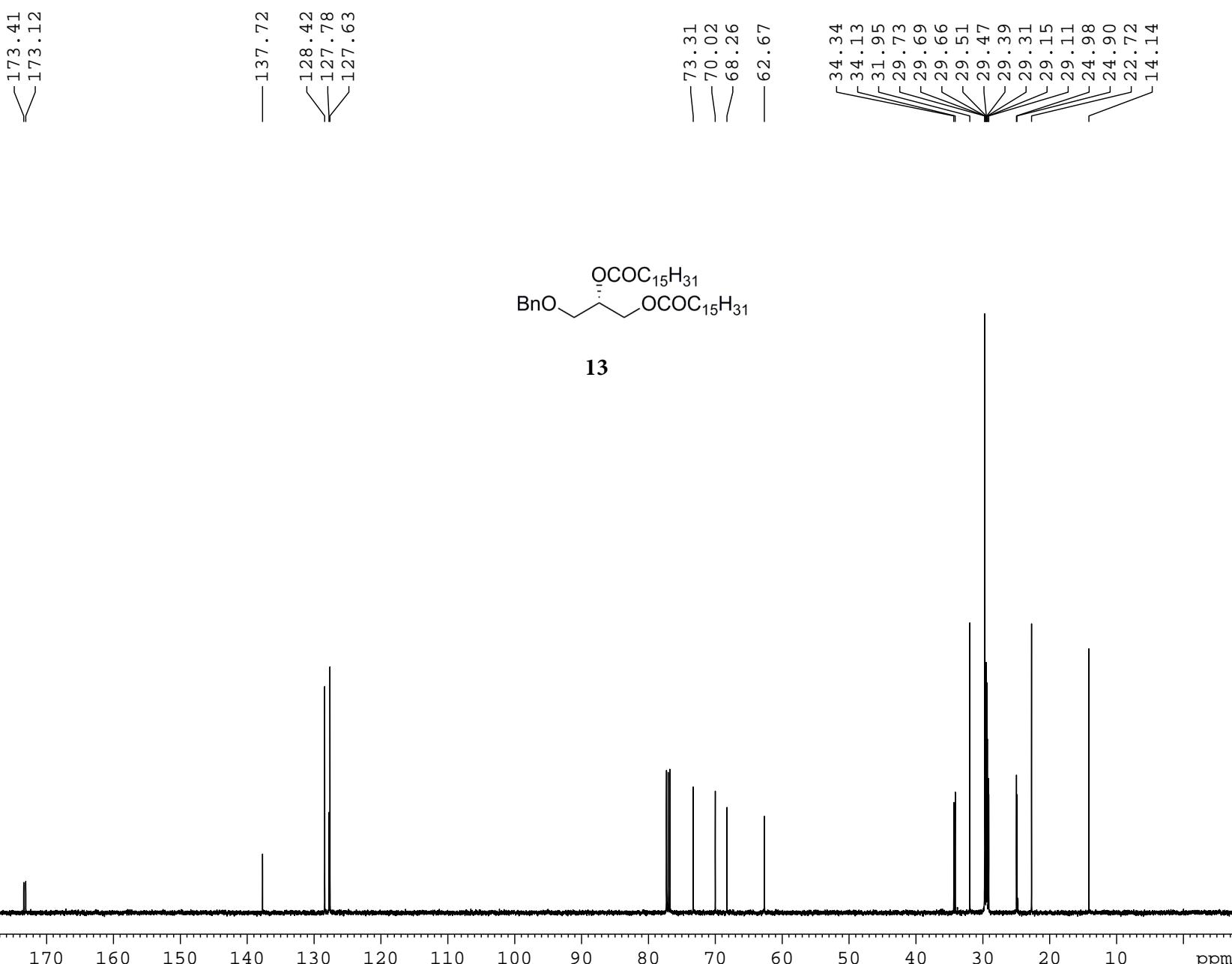
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 NUC2 ¹H
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F2 - Processing parameters
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Current Data Parameters
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PROCNO 1

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DS 0
SWH 20161.291 Hz
FIDRES 0.307637 Hz
AQ 1.625292 sec
RG 197.27
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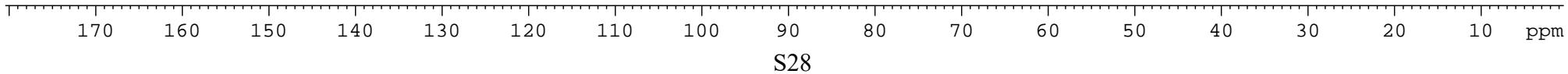
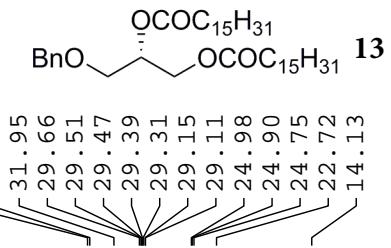
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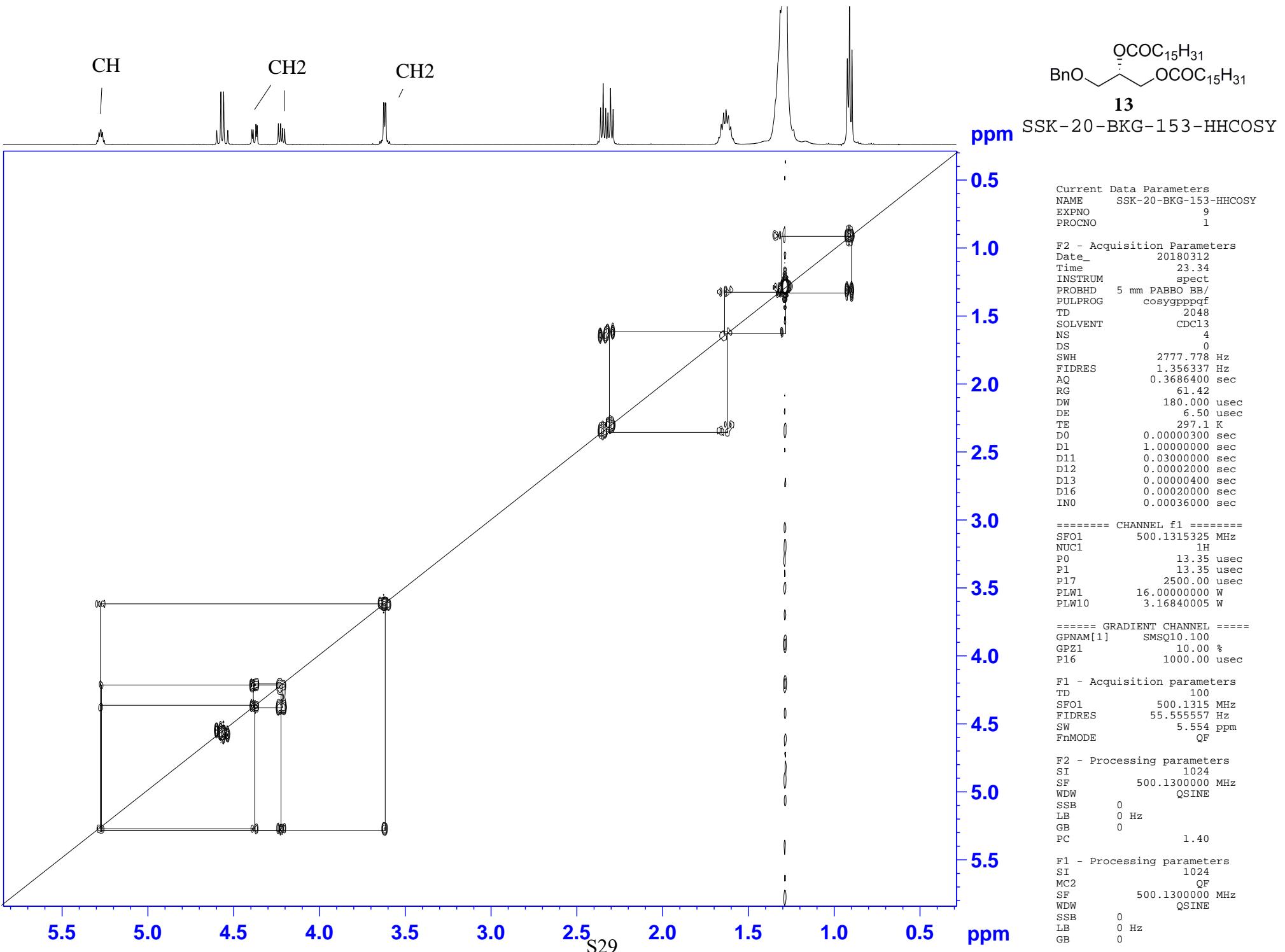
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PLW2 16.0000000 W
PLW12 0.44556001 W

F2 - Processing parameters
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WDW EM
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GB 0
PC 1.40

SSK-20-BKG-153-DEPT

— 73.31
— 70.02
— 68.26
— 62.67





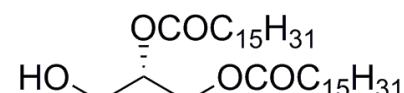
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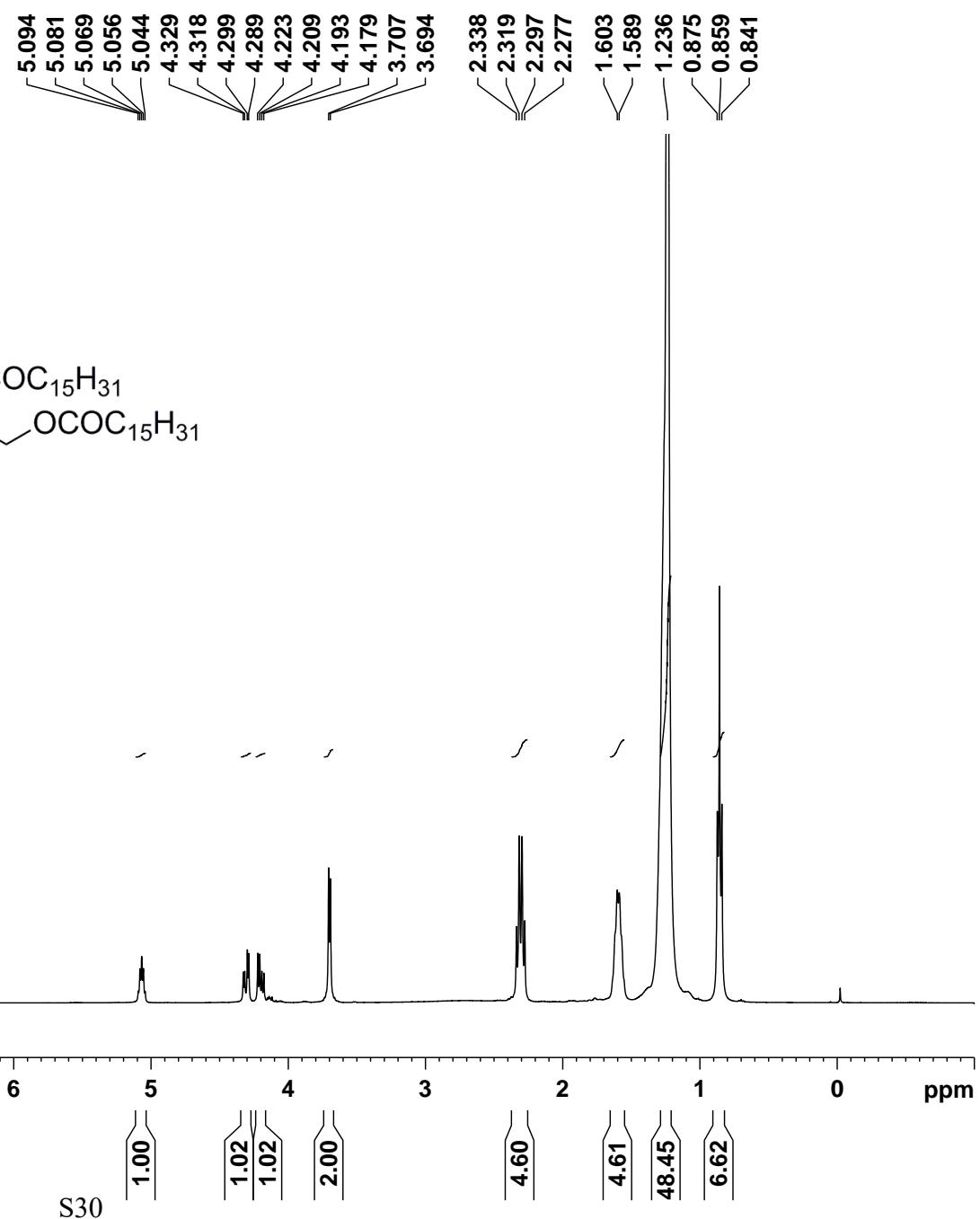
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TE 296.0 K
D1 1.0000000 sec
TD0 1

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F2 - Processing parameters
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PC 1.00



11



Current Data Parameters
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EXPNO 2
PROCNO 1

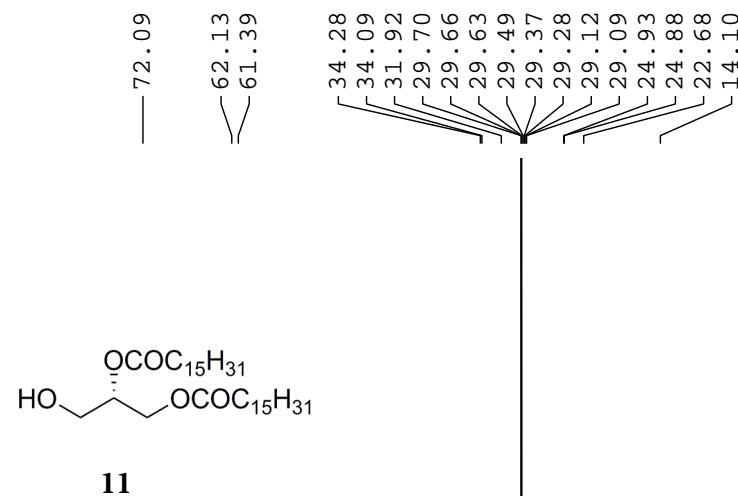
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 59
DS 0
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2582912 sec
RG 2050
DW 19.200 usec
DE 6.50 usec
TE 296.5 K
D1 1.0000000 sec
D11 0.03000000 sec
TD0 1

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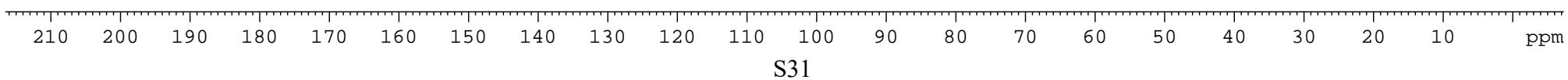
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PL12 13.69 dB
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PL2W 10.56200695 W
PL12W 0.35871249 W
PL13W 0.29767781 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

ssk-20-bkg-170-13c



11



Current Data Parameters
NAME ssk-20-bkg-170-dept
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
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PULPROG dept135
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RG 2050
DW 20.800 usec
DE 6.50 usec
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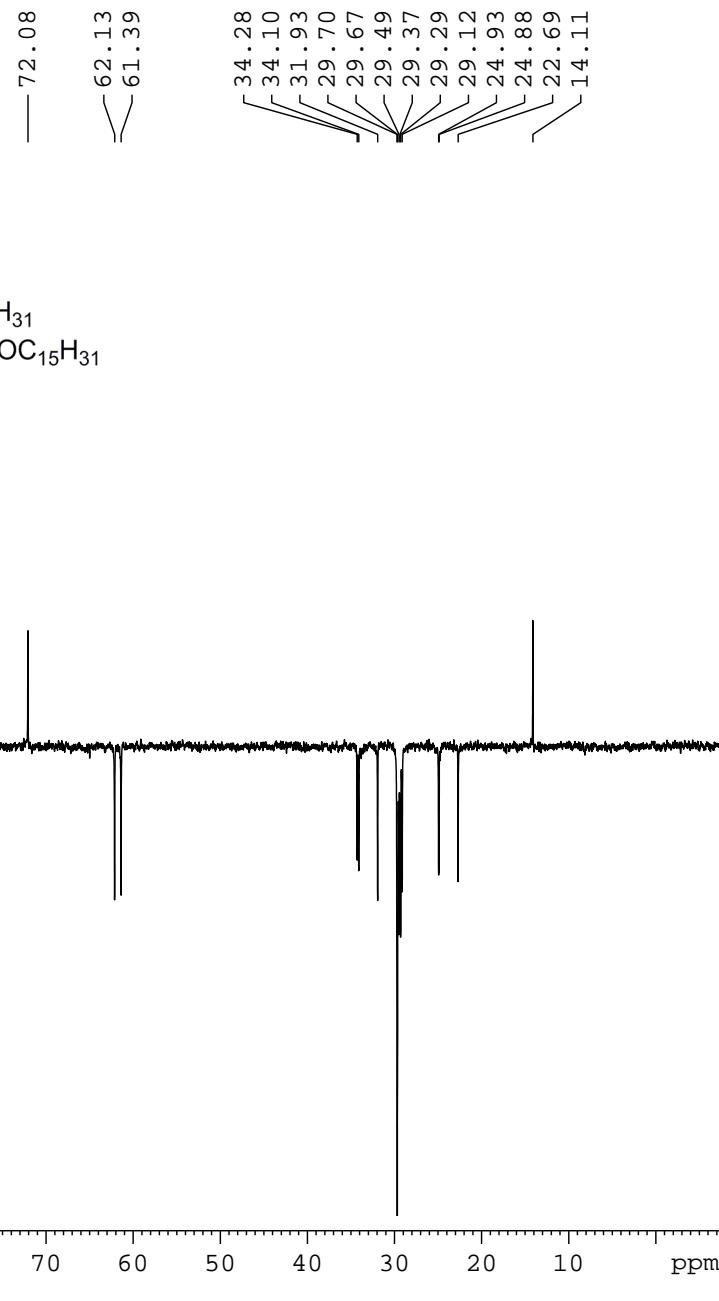
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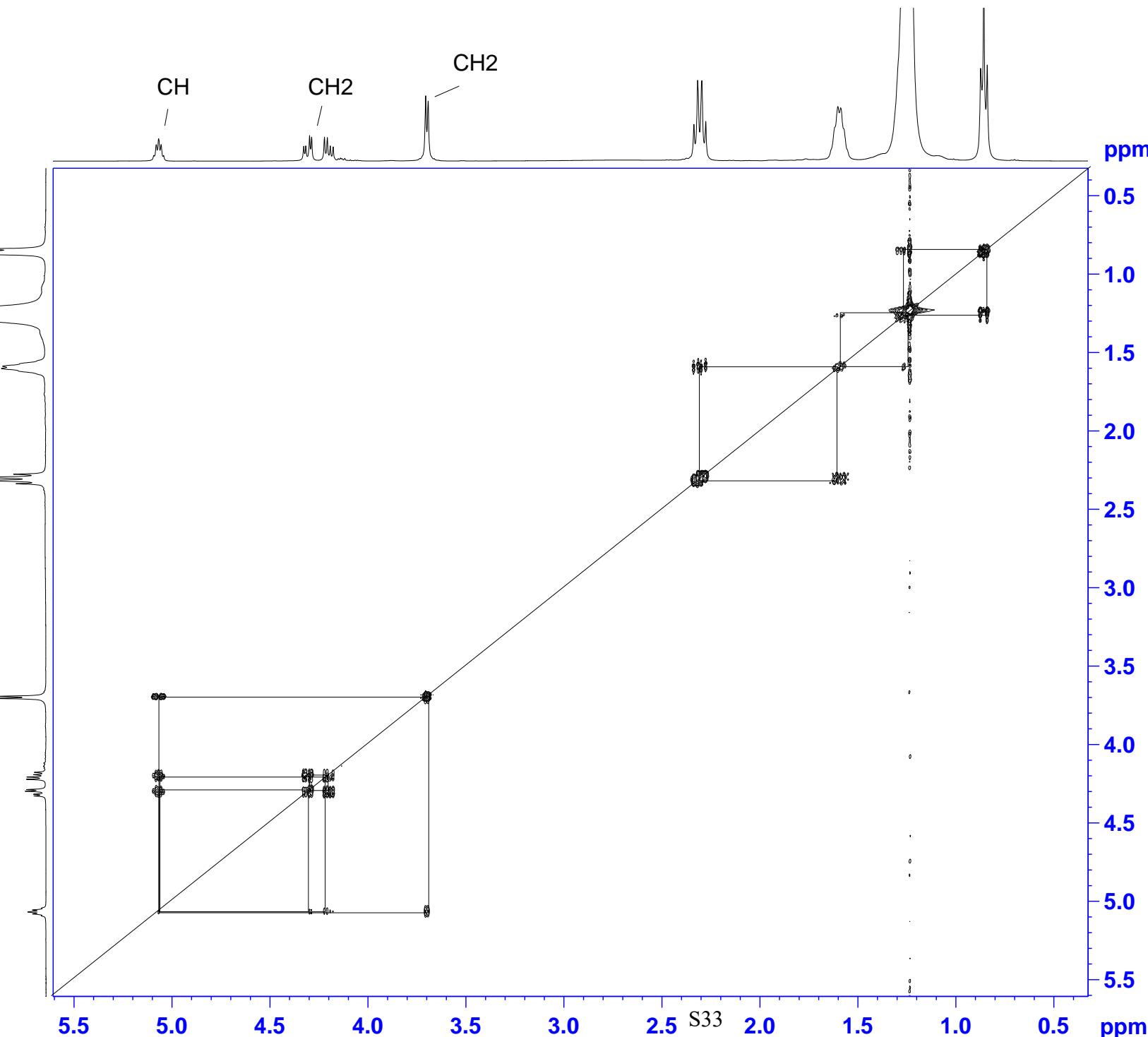
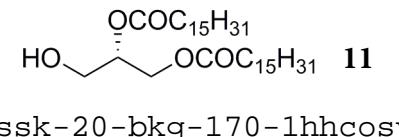
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P4 29.50 usec
PCPD2 80.00 usec
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PL12 13.69 dB
PL2W 10.56200000 W
PL12W 0.35871249 W
SFO2 400.1316005 MHz

F2 - Processing parameters
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SF 100.6127690 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

ssk-20-bkg-170-dept





Current Data Parameters
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 PROCNO 1

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 NS 4
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 AQ 0.4845568 sec
 RG 18
 DW 236.600 usec
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 TE 296.6 K
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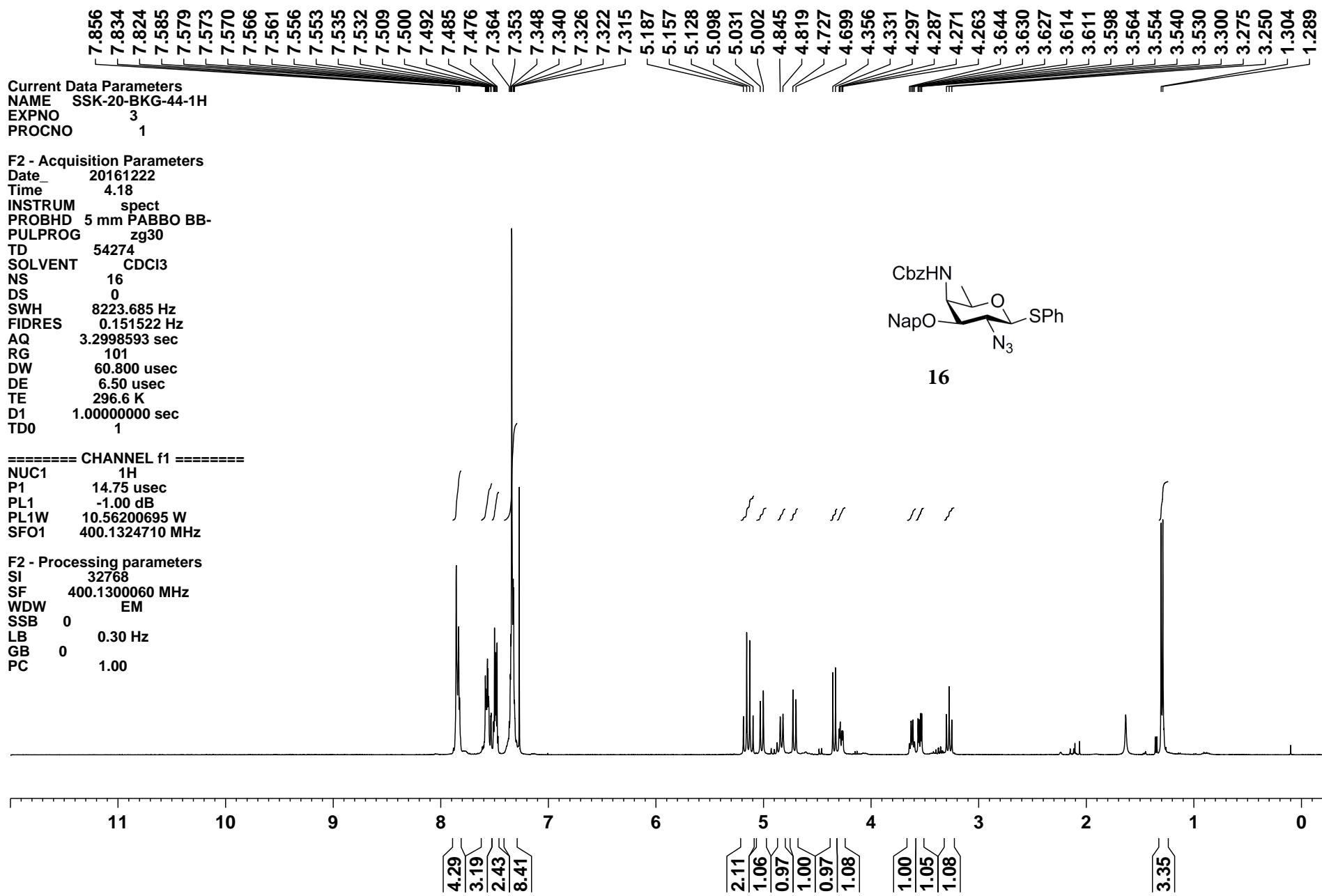
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 SW 5.281 ppm
 FnMODE QF

F2 - Processing parameters
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 LB 0 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
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SSK-20-BKG-44-1H



SSK-20-BKG-44-13C

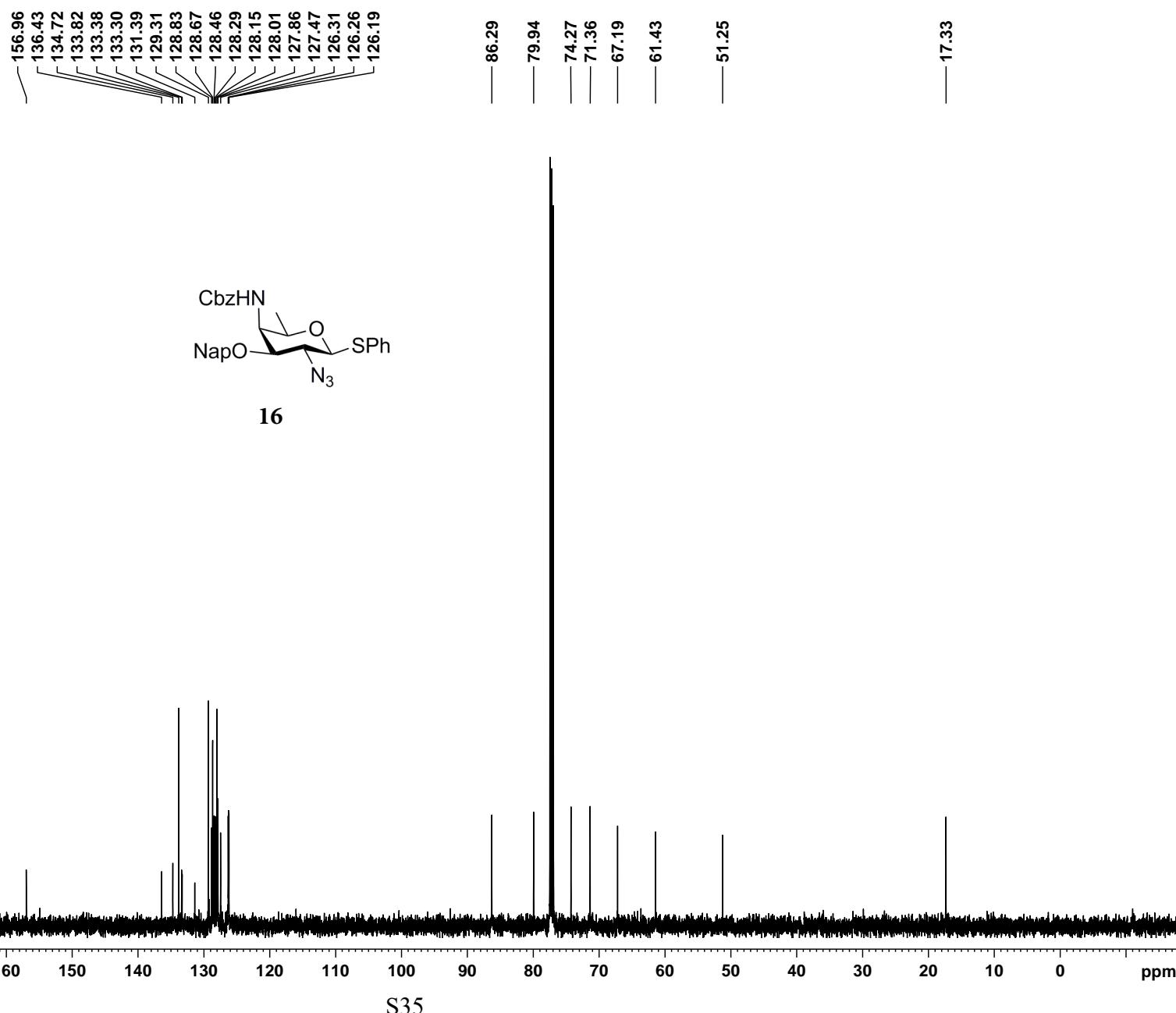
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 SOLVENT CDCl3
 NS 51
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 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010048 sec
 RG 197.27
 DW 16.800 usec
 DE 6.50 usec
 TE 295.7 K
 D1 2.0000000 sec
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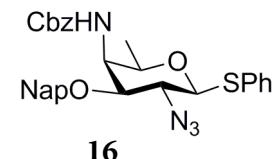
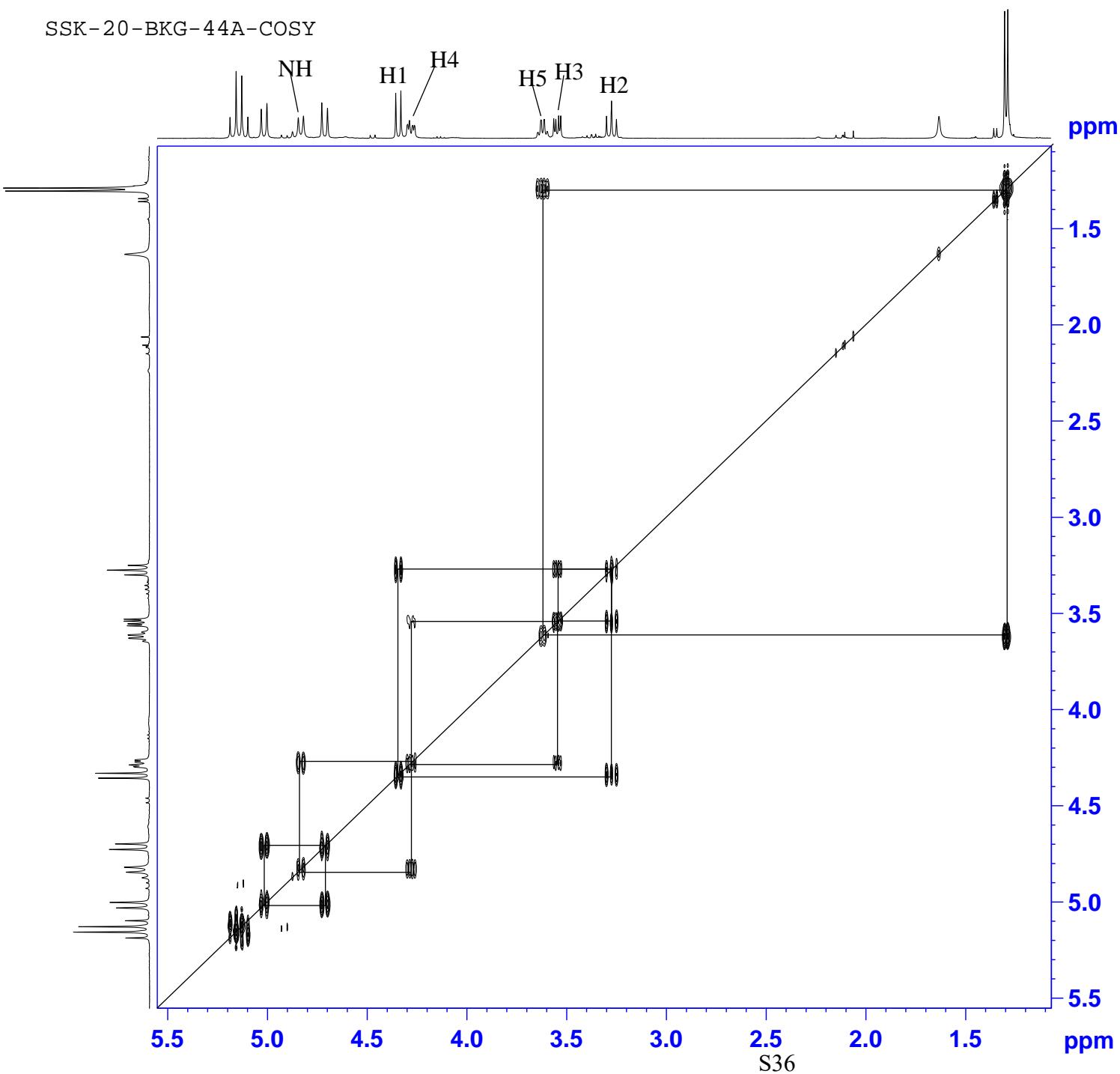
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 PLW13 0.17267001 W

F2 - Processing parameters
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 LB 1.00 Hz
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H6

SSK-20-BKG-44A-COSY



Current Data Parameters
 NAME SSK-20-BKG-44A-COSY
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161222
 Time 4.34
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG cosygpaf
 TD 2048
 SOLVENT CDCl3
 NS 8
 DS 16
 SWH 1793.400 Hz
 FIDRES 0.875684 Hz
 AQ 0.5709824 sec
 RG 203
 DW 278.800 usec
 DE 6.50 usec
 TE 296.5 K
 D0 0.00000300 sec
 D1 1.5000000 sec
 D13 0.00000400 sec
 D16 0.00020000 sec
 IN0 0.00055760 sec

===== CHANNEL f1 =====
 NUC1 1H
 P0 14.75 usec
 P1 14.75 usec
 PL1 -1.00 dB
 PLLW 10.56200695 W
 SF01 400.1313310 MHz

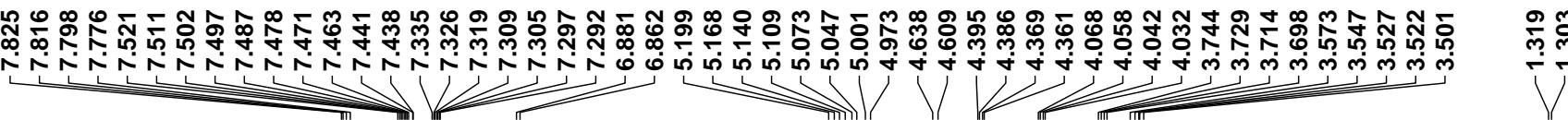
===== GRADIENT CHANNEL =====
 GPNAME[1] SINE.100
 GPZ1 10.00 %
 P16 1000.00 usec

F1 - Acquisition parameters
 TD 56
 SF01 400.1313 MHz
 FIDRES 32.024796 Hz
 SW 4.482 ppm
 FnMODE QF

F2 - Processing parameters
 SI 2048
 SF 400.1300060 MHz
 WDW SINE
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
 SI 512
 MC2 QF
 SF 400.1300060 MHz
 WDW SINE
 SSB 0
 LB 0 Hz
 GB 0

SSK-20-BKG-AAT-1H

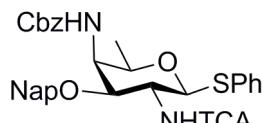


Current Data Parameters
NAME SSK-20-BKG-AAT-1H
EXPNO 1
PROCNO 1

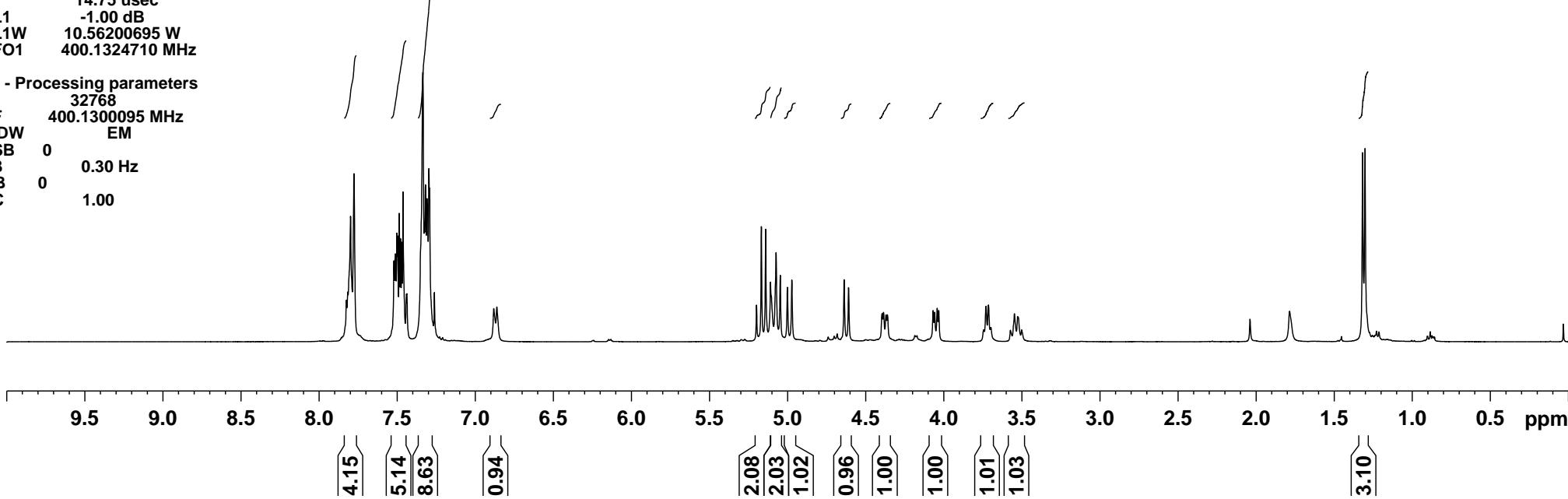
F2 - Acquisition Parameters
Date 20180424
Time 3.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 54274
SOLVENT CDCl₃
NS 23
DS 0
SWH 8223.685 Hz
FIDRES 0.151522 Hz
AQ 3.2998593 sec
RG 50.8
DW 60.800 usec
DE 6.50 usec
TE 295.8 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 ¹H
P1 14.75 usec
PL1 -1.00 dB
PL1W 10.56200695 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



8



SSK-20-BKG-AAT-13C

Current Data Parameters
 NAME SSK-20-BKG-AAT-13C
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date 20180424
 Time 4.00
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 161
 DS 0
 SWH 26041.666 Hz
 FIDRES 0.397364 Hz
 AQ 1.2582912 sec
 RG 1820
 DW 19.200 usec
 DE 6.50 usec
 TE 296.1 K
 D1 1.0000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====

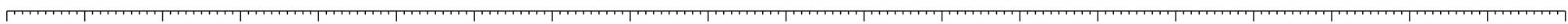
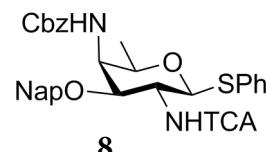
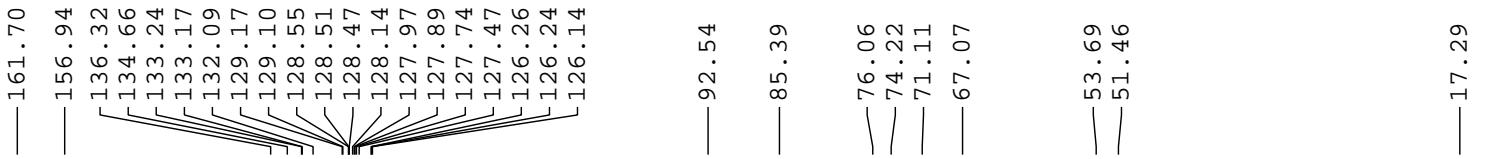
NUC1 13C
 P1 8.50 usec
 PL1 -2.00 dB
 PL1W 56.53121948 W
 SFO1 100.6238364 MHz

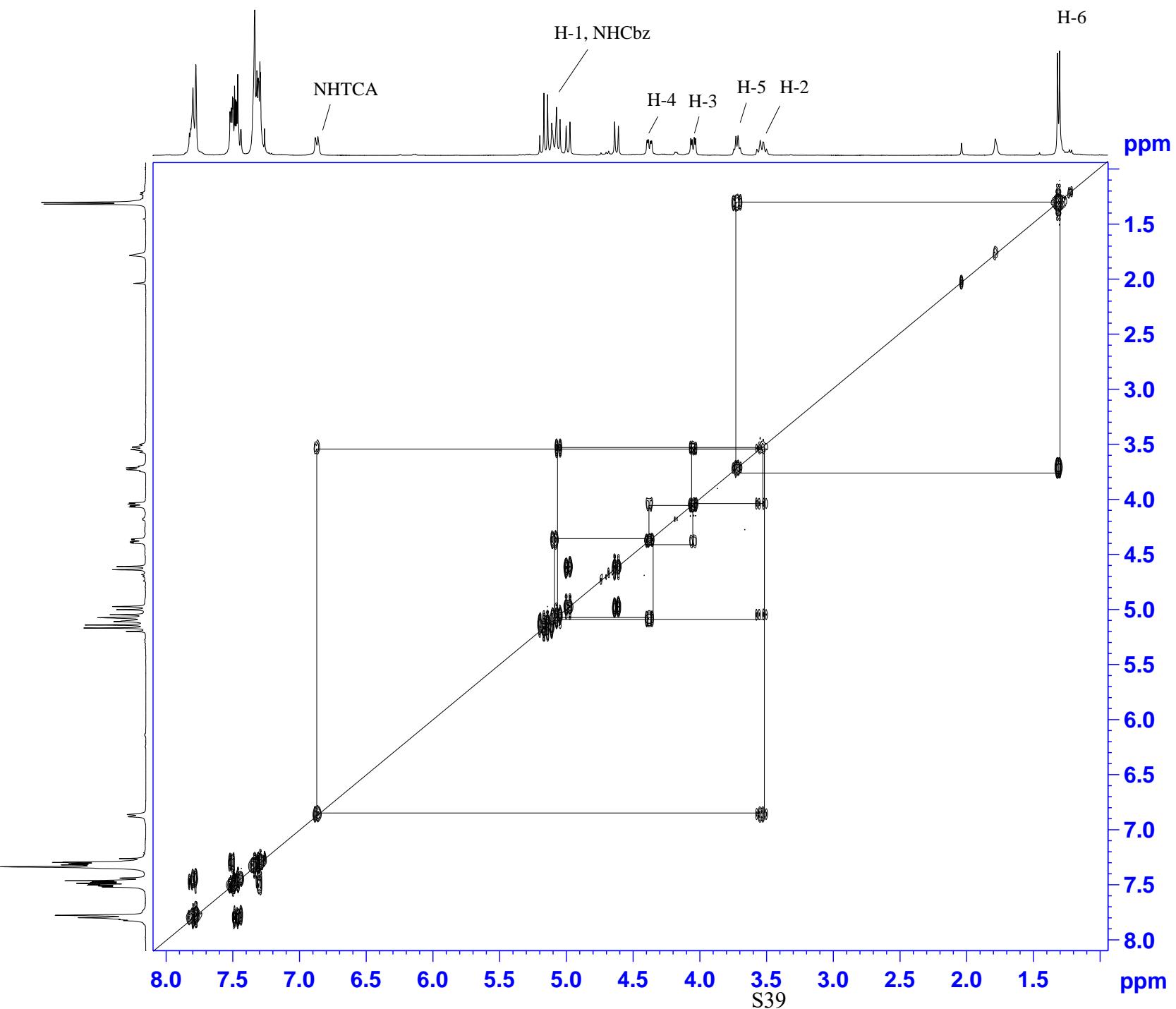
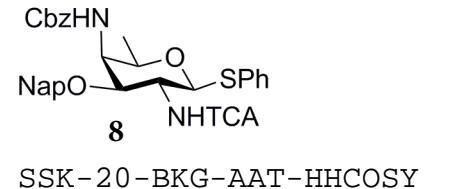
===== CHANNEL f2 =====

CPDPRG[2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 13.69 dB
 PL13 14.50 dB
 PL2W 10.56200695 W
 PL12W 0.35871249 W
 PL13W 0.29767781 W
 SFO2 400.1316005 MHz

F2 - Processing parameters

SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





Current Data Parameters
NAME SSK-20-BKG-AAT-HHCO_{SY}
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180424
Time 4.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG cosygpgrf
TD 2048
SOLVENT CDCl₃
NS 4
DS 0
SWH 2863.688 Hz
FIDRES 1.398285 Hz
AQ 0.3575808 sec
RG 101
DW 174.600 usec
DE 6.50 usec
TE 296.5 K
D0 0.00000300 sec
D1 1.00000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00034920 sec

===== CHANNEL f1 =====
NUC1 1H
P0 14.75 usec
P1 14.75 usec
PL1 -1.00 dB
PL1W 10.56200695 W
SFO1 400.1318179 MHz

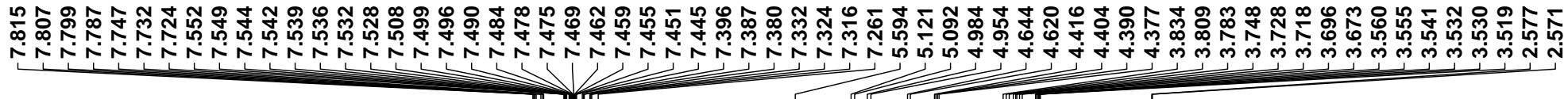
===== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPZ1 10.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 71
SFO1 400.1318 MHz
FIDRES 80.667702 Hz
SW 7.157 ppm
FnMODE QF

F2 - Processing parameters
SI 2048
SF 400.1300095 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 512
MC2 QF
SF 400.1300095 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0

SSK-20-BKG-30B-1H

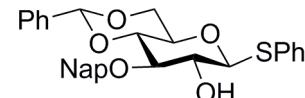


Current Data Parameters
 NAME SSK-20-BKG-30B-1H
 EXPNO 1
 PROCNO 1

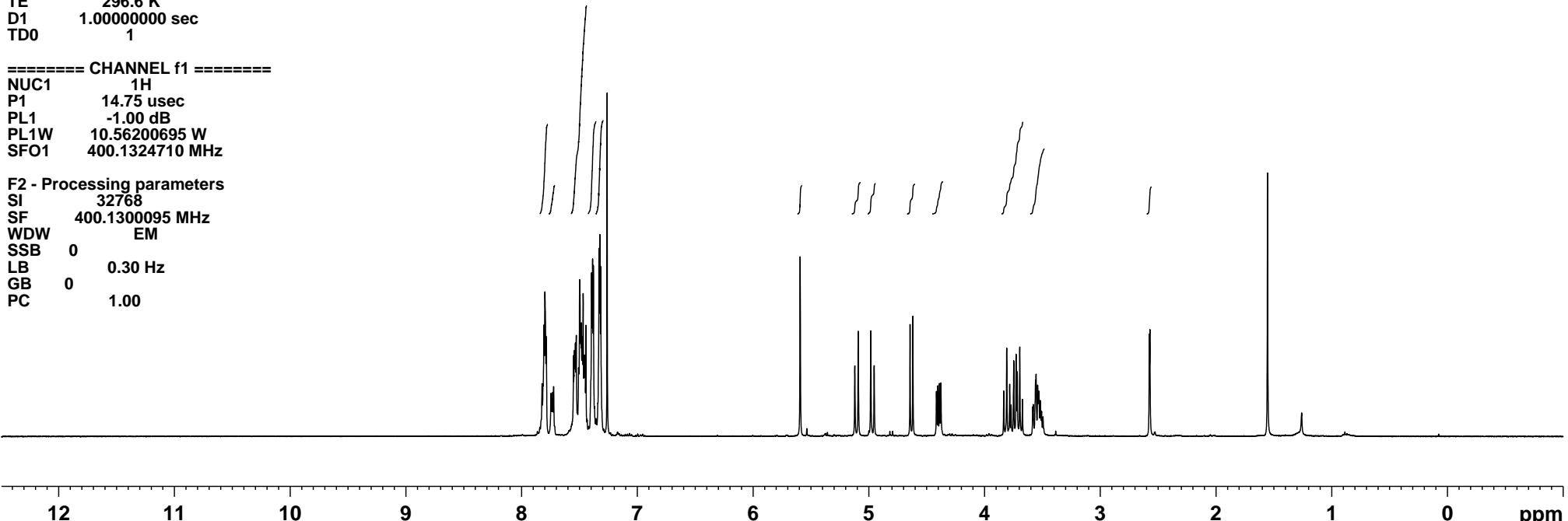
F2 - Acquisition Parameters
 Date 20161231
 Time 5.32
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 54274
 SOLVENT CDCl3
 NS 14
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.151522 Hz
 AQ 3.2998593 sec
 RG 32
 DW 60.800 usec
 DE 6.50 usec
 TE 296.6 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.75 usec
 PL1 -1.00 dB
 PL1W 10.56200695 W
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



18



SSK-20-BKG-205-13C

Current Data Parameters
 NAME SSK-20-BKG-205-13C
 EXPNO 9
 PROCNO 1

F2 - Acquisition Parameters
 Date 20180301
 Time 10.53
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 112
 DS 0
 SWH 26041.666 Hz
 FIDRES 0.397364 Hz
 AQ 1.2582912 sec
 RG 912
 DW 19.200 usec
 DE 6.50 usec
 TE 303.3 K
 D1 1.0000000 sec
 D11 0.03000000 sec
 TDO 1

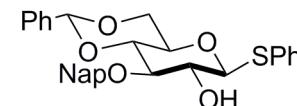
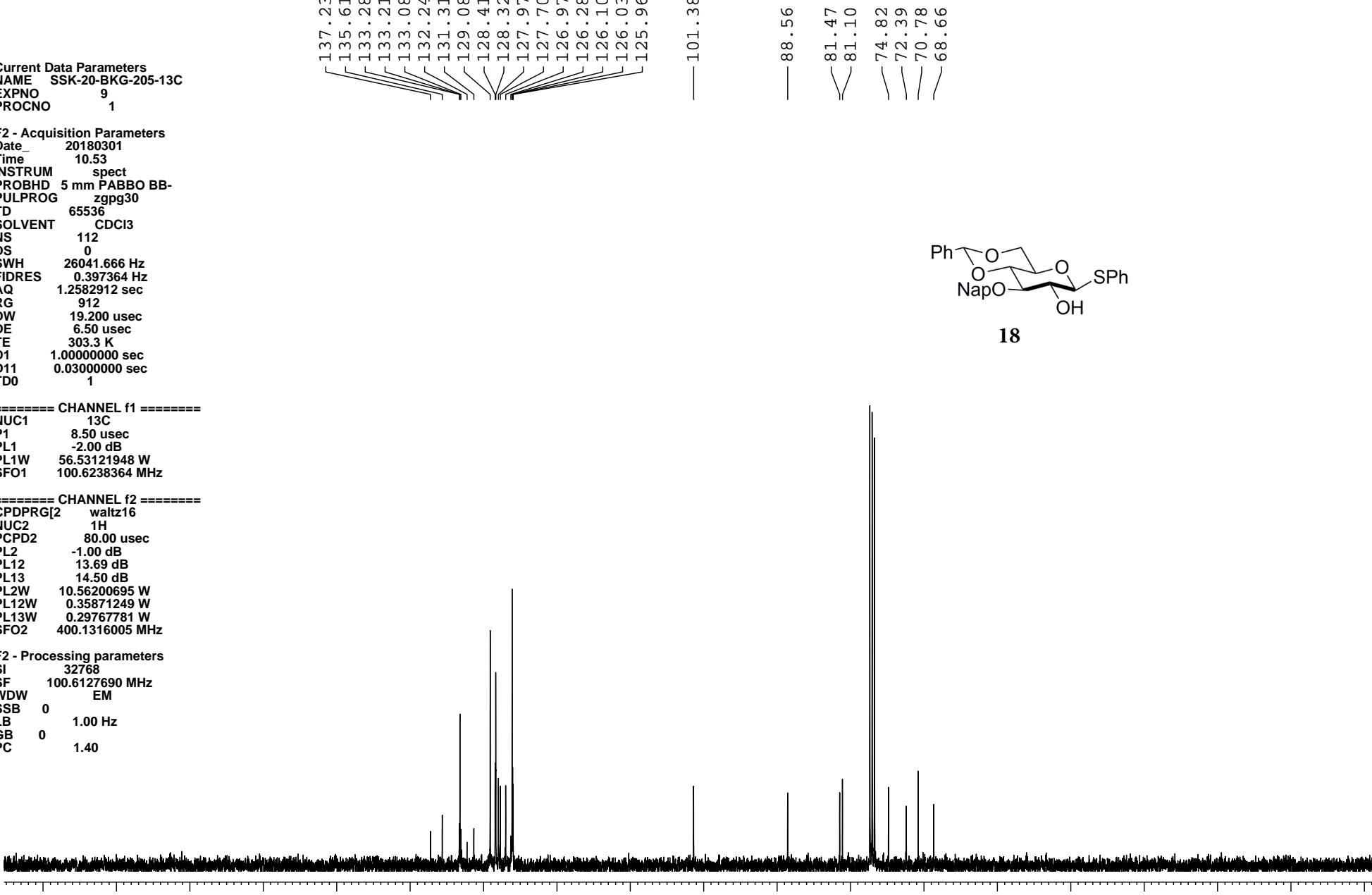
===== CHANNEL f1 =====

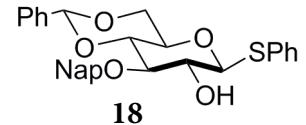
NUC1 13C
 P1 8.50 usec
 PL1 -2.00 dB
 PL1W 56.53121948 W
 SFO1 100.6238364 MHz

===== CHANNEL f2 =====

CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 13.69 dB
 PL13 14.50 dB
 PL2W 10.56200695 W
 PL12W 0.35871249 W
 PL13W 0.29767781 W
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





SSK-20-BKG-30B-COSY

Current Data Parameters
NAME SSK-20-BKG-30B-COSY
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161231
Time 5.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG cosygpqf
TD 2048
SOLVENT CDCl3
NS 4
DS 0
SWH 1559.576 Hz
FIDRES 0.761512 Hz
AQ 0.6565888 sec
RG 2.56
DW 320.600 usec
DE 6.50 usec
TE 297.4 K
D0 0.00000300 sec
D1 1.5000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
INO 0.00064120 sec

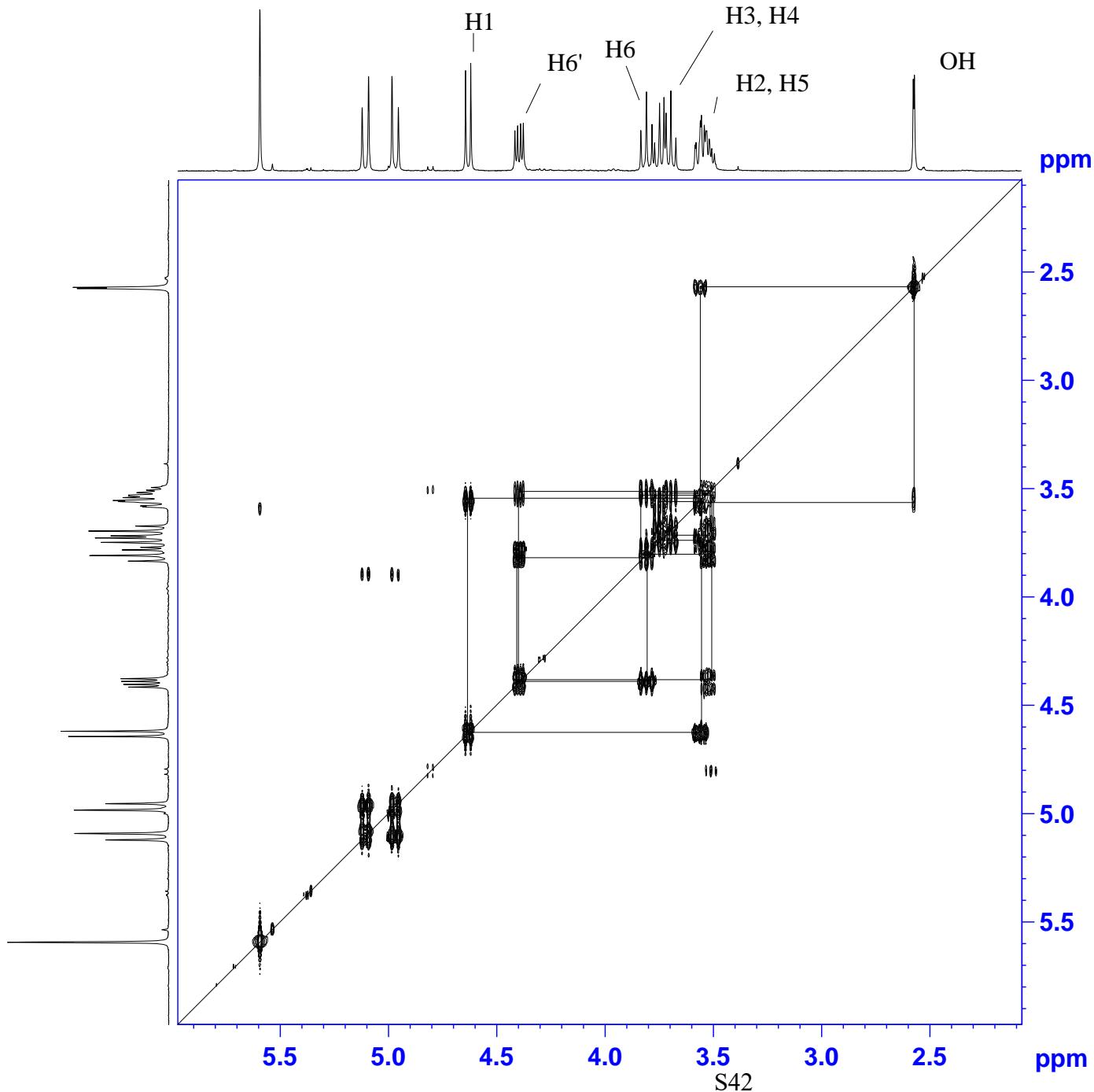
===== CHANNEL f1 ======
NUC1 1H
P0 14.75 usec
P1 14.75 usec
PL1 -1.00 dB
PL1W 10.5620095 W
SFO1 400.1316197 MHz

===== GRADIENT CHANNEL =====
GPNAME[1] SINE.100
GPZ1 10.00 %
P16 1000.00 usec

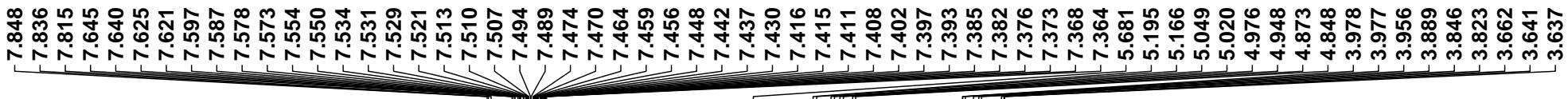
F1 - Acquisition parameters
TD 89
SFO1 400.1316 MHz
FIDRES 17.523518 Hz
SW 3.898 ppm
FnMODE QF

F2 - Processing parameters
SI 2048
SF 400.1300095 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 512
MC2 QF
SF 400.1300095 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0



SSK-20-BKG-273-1H

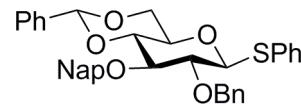


Current Data Parameters
 NAME SSK-20-BKG-273-1H
 EXPNO 14
 PROCNO 1

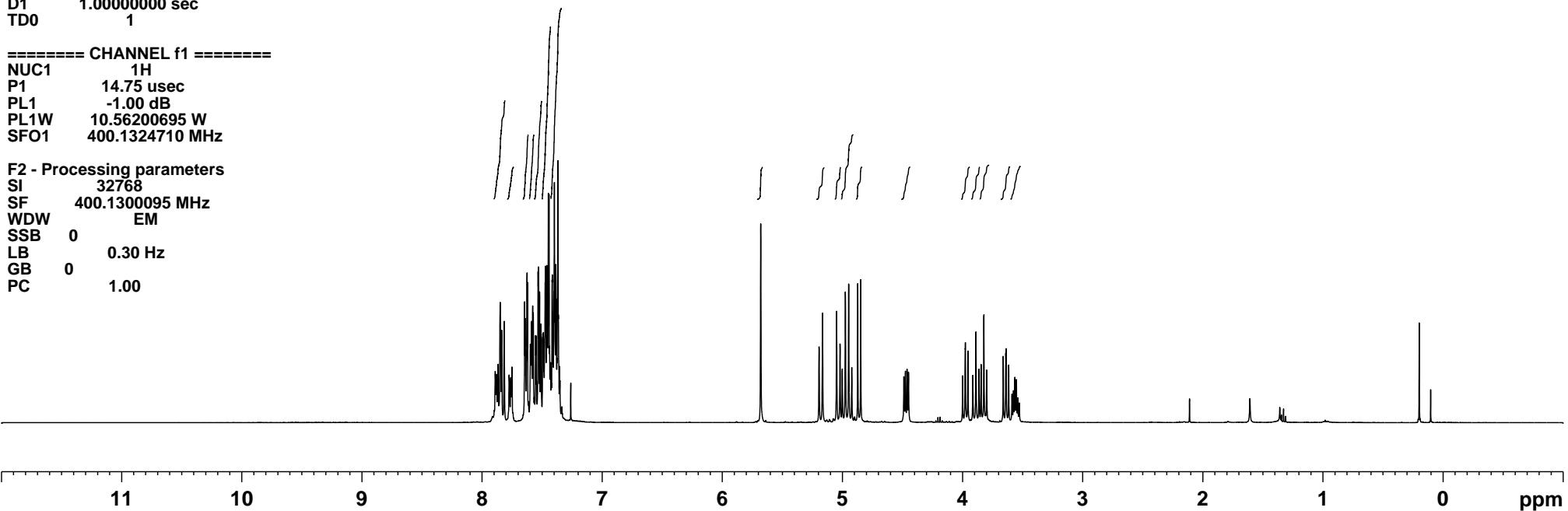
F2 - Acquisition Parameters
 Date 20180308
 Time 15.14
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 54274
 SOLVENT CDCl3
 NS 20
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.151522 Hz
 AQ 3.2998593 sec
 RG 32
 DW 60.800 usec
 DE 6.50 usec
 TE 296.7 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.75 usec
 PL1 -1.00 dB
 PL1W 10.56200695 W
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



10



3.07
1.00
2.03
2.02
3.07
5.35
5.93

1.00
0.99
1.01
2.01
1.01
1.01
1.00
1.01
1.07
1.02
1.03

SSK-20-BKG-273-13C(2)

Current Data Parameters
 NAME SSK-20-BKG-273-13C(2)
 EXPNO 16
 PROCNO 1

F2 - Acquisition Parameters

Date 20180308
 Time 15.18
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 50
 DS 0
 SWH 26041.666 Hz
 FIDRES 0.397364 Hz
 AQ 1.2582912 sec
 RG 1030
 DW 19.200 usec
 DE 6.50 usec
 TE 296.9 K
 D1 1.0000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====

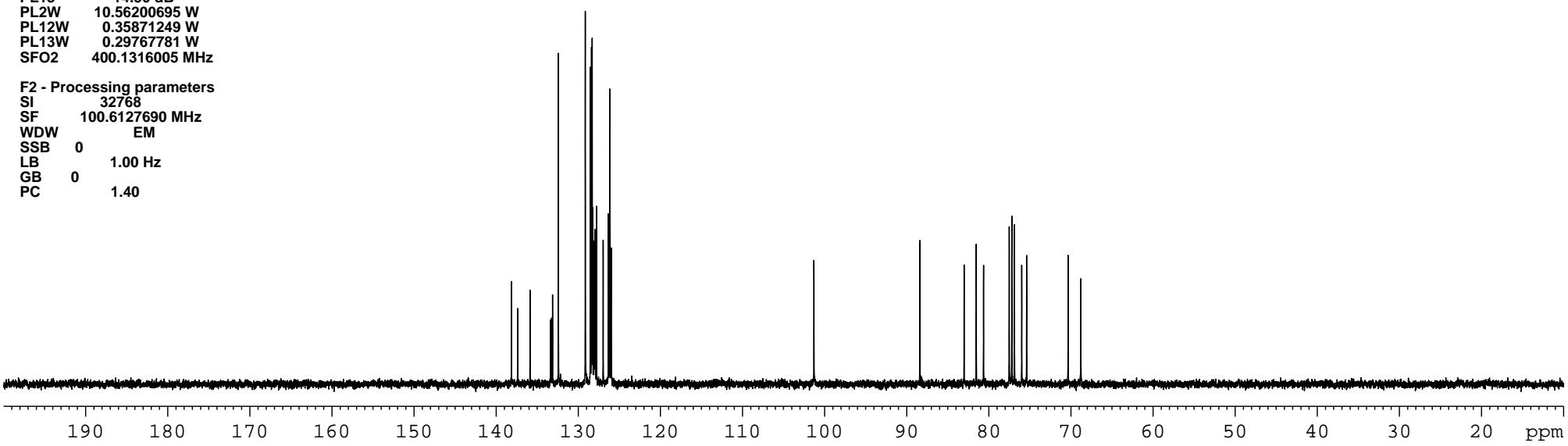
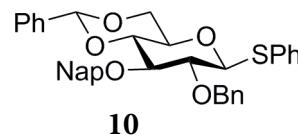
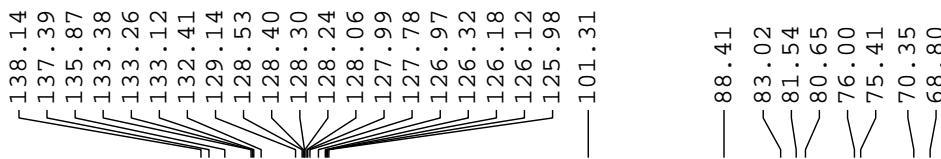
NUC1 13C
 P1 8.50 usec
 PL1 -2.00 dB
 PL1W 56.53121948 W
 SFO1 100.6238364 MHz

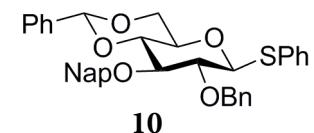
===== CHANNEL f2 =====

CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 13.69 dB
 PL13 14.50 dB
 PL2W 10.56200695 W
 PL12W 0.35871249 W
 PL13W 0.29767781 W
 SFO2 400.1316005 MHz

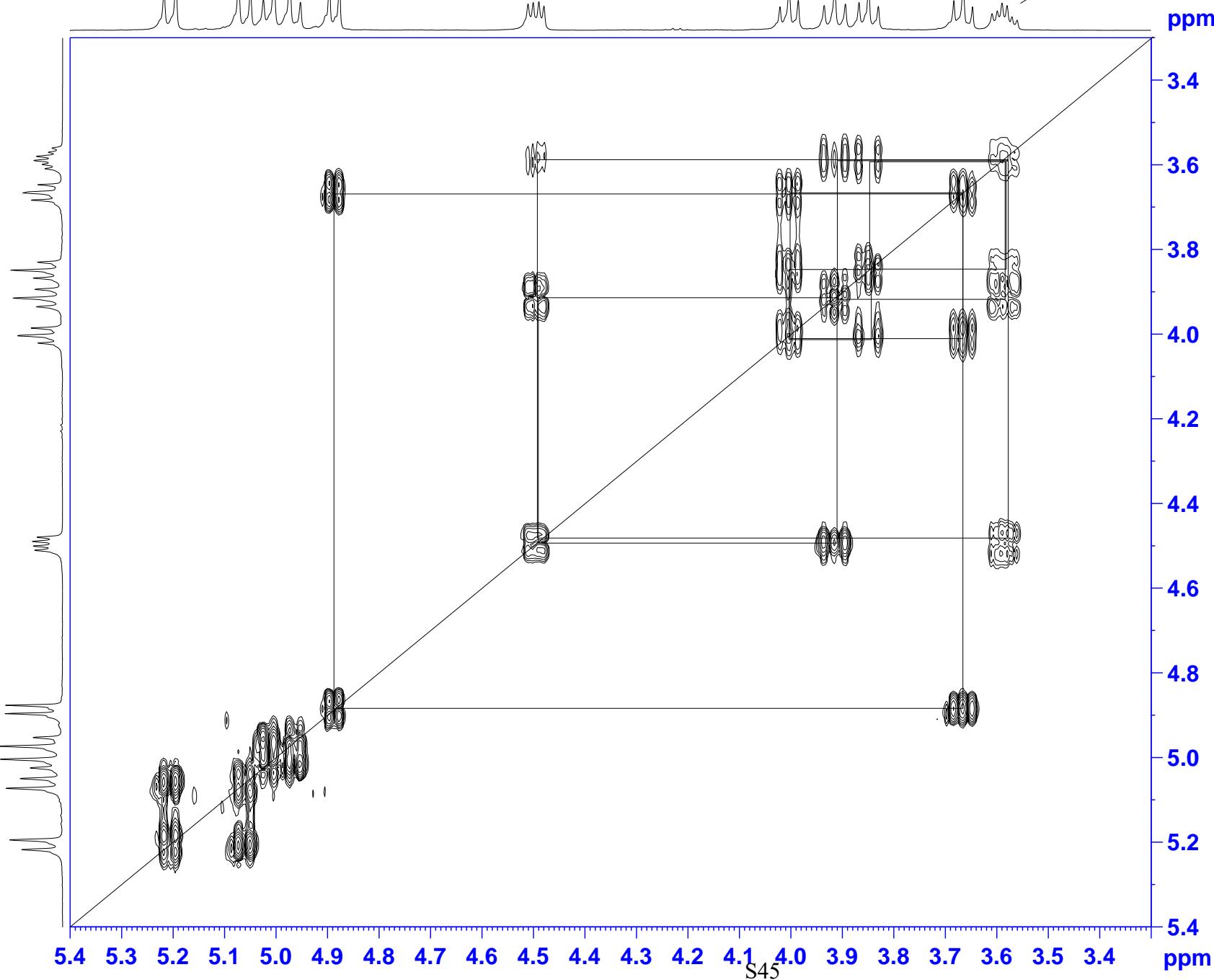
F2 - Processing parameters

SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





SSK-20-BKG-273-HHCOSY



Current Data Parameters
 NAME SSK-20-BKG-273-HHCOSY
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20180308
 Time 22.29
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG cosygpppqf
 TD 2048
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 2325.581 Hz
 FIDRES 1.135538 Hz
 AQ 0.4403200 sec
 RG 61.42
 DW 215.000 usec
 DE 6.50 usec
 TE 296.4 K
 D0 0.00000300 sec
 D1 1.0000000 sec
 D11 0.03000000 sec
 D12 0.00002000 sec
 D13 0.00000400 sec
 D16 0.00020000 sec
 IN0 0.00043000 sec

===== CHANNEL f1 =====
 SFO1 500.1317571 MHz
 NUC1 1H
 P0 13.35 usec
 P1 13.35 usec
 P17 2500.00 usec
 PLW1 16.0000000 W
 PLW10 3.16840005 W

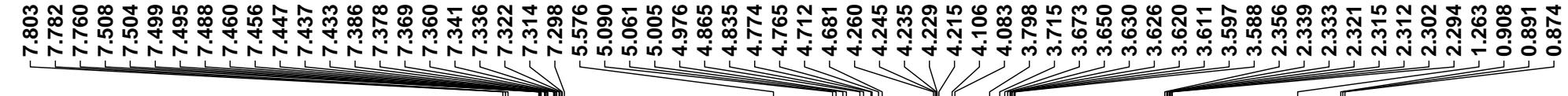
===== GRADIENT CHANNEL =====
 GPNAM[1] SMSQ10.100
 GPZ1 10.00 %
 P16 1000.00 usec

F1 - Acquisition parameters
 TD 128
 SFO1 500.1318 MHz
 FIDRES 36.337208 Hz
 SW 4.650 ppm
 FnMODE QF

F2 - Processing parameters
 SI 1024
 SF 500.1300000 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
 SI 1024
 MC2 QF
 SF 500.1300000 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0

SSK-20-BKG-173-1H



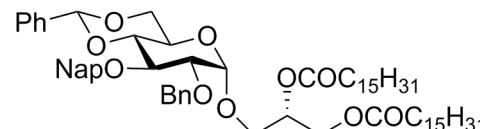
Current Data Parameters
 NAME SSK-20-BKG-173-1H
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date 20170714
 Time 11.04
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 54274
 SOLVENT CDCl3
 NS 6
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.151522 Hz
 AQ 3.2998593 sec
 RG 50.8
 DW 60.800 usec
 DE 6.50 usec
 TE 297.1 K
 D1 1.0000000 sec
 TD0 1

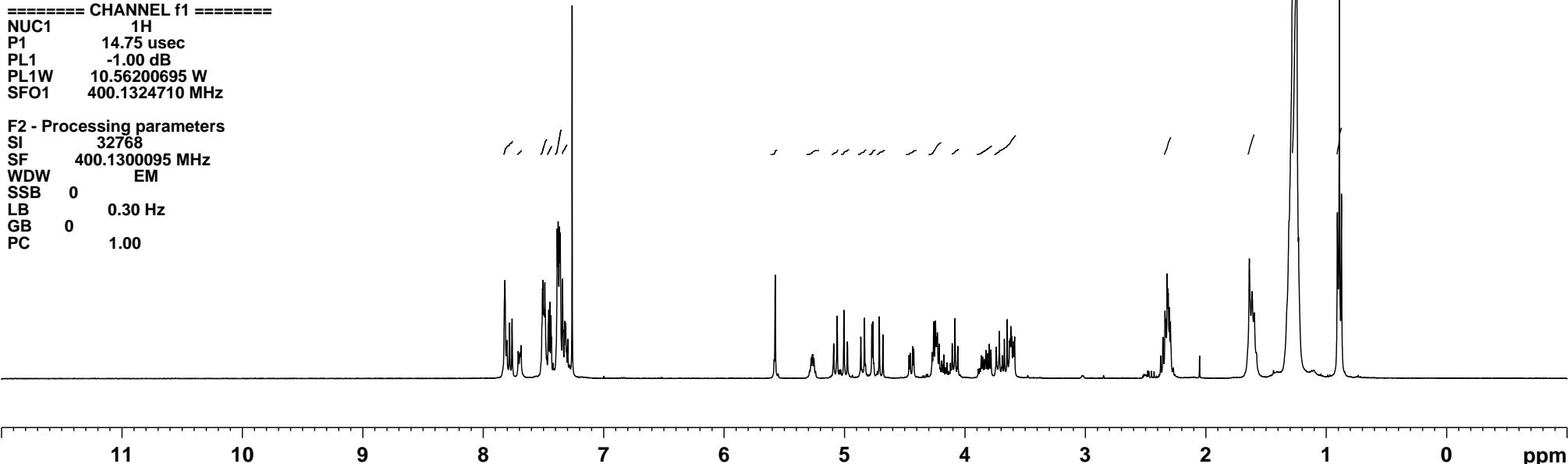
===== CHANNEL f1 ======

NUC1 1H
 P1 14.75 usec
 PL1 -1.00 dB
 PL1W 10.56200695 W
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



19



3.26
0.99
3.70
2.05
6.01
2.33

1.06
S46
1.00
1.11
1.17
1.15
1.07
0.99
0.99
2.88
2.08
4.58

4.25
4.96
48.34
6.72

Current Data Parameters
 NAME SSK-20-BKG-173-13C
 EXPNO 2
 PROCNO 1

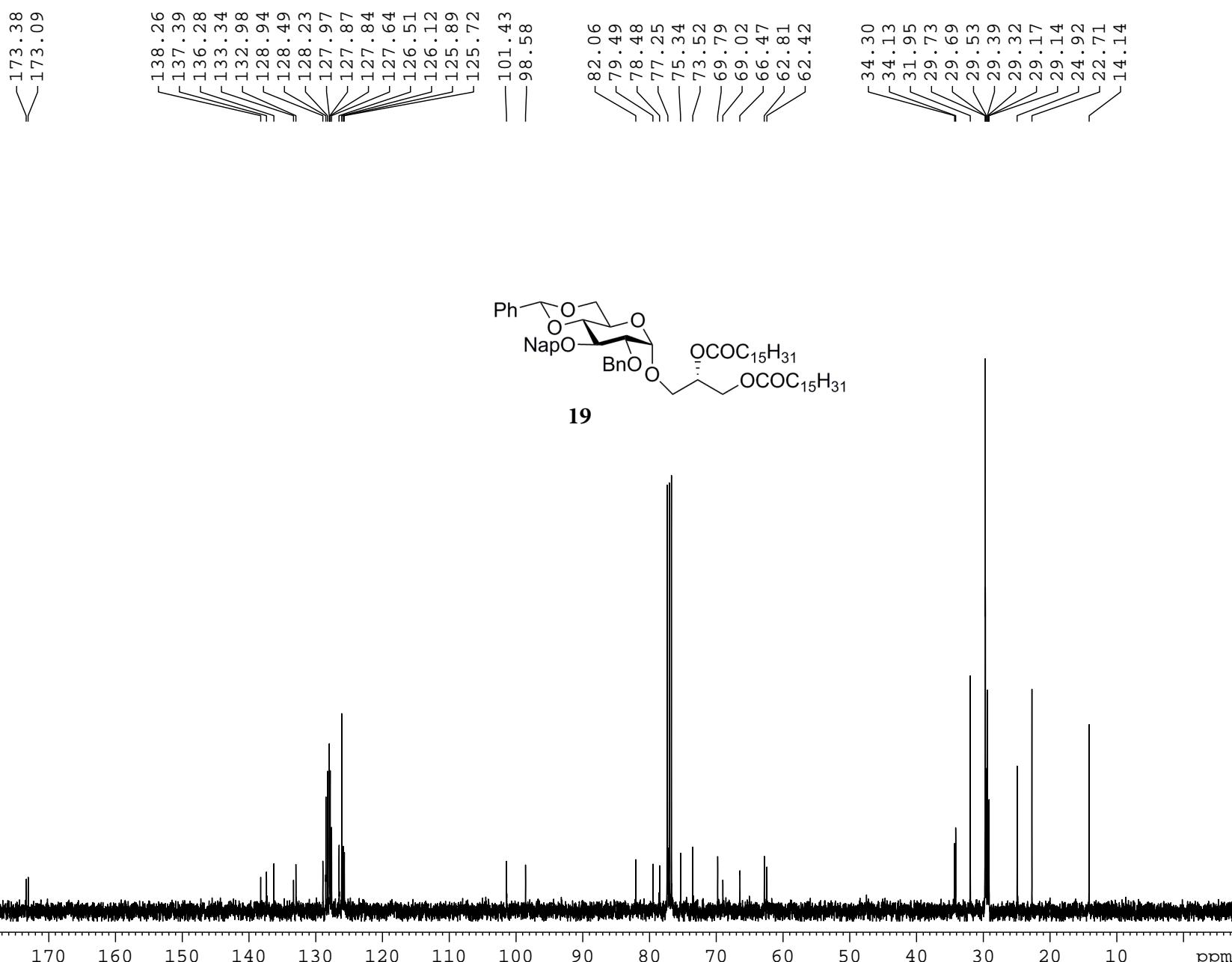
F2 - Acquisition Parameters
 Date 20170714
 Time 11.04
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 85
 DS 0
 SWH 26041.666 Hz
 FIDRES 0.397364 Hz
 AQ 1.2582912 sec
 RG 2050
 DW 19.200 usec
 DE 6.50 usec
 TE 297.1 K
 D1 1.0000000 sec
 D11 0.03000000 sec
 TDO 1

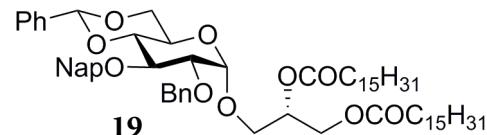
===== CHANNEL f1 =====
 NUC1 13C
 P1 8.50 usec
 PL1 -2.00 dB
 PL1W 56.53121948 W
 SFO1 100.6238364 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 13.69 dB
 PL13 14.50 dB
 PL2W 10.56200695 W
 PL12W 0.35871249 W
 PL13W 0.29767781 W
 SFO2 400.1316005 MHz

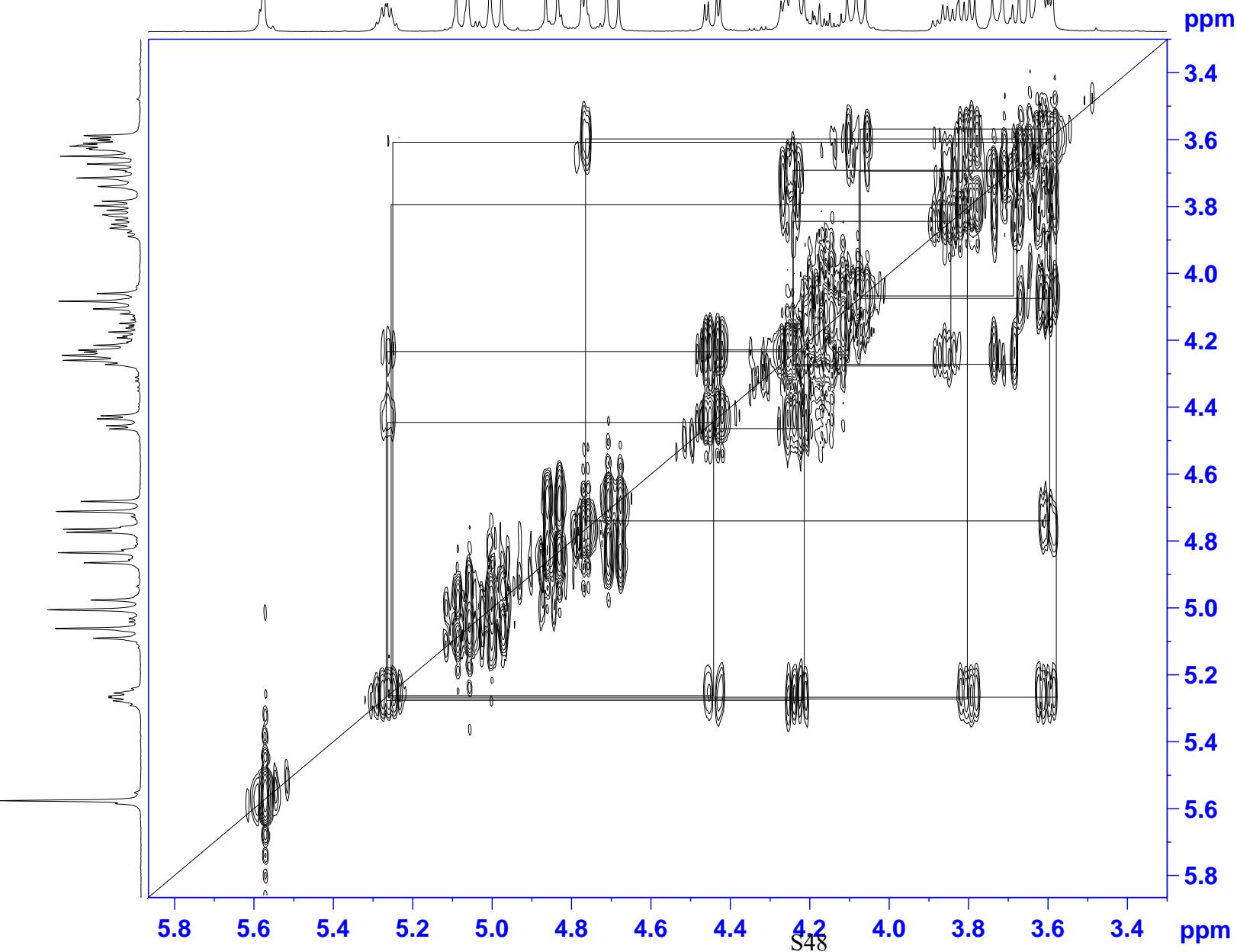
F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

SSK-20-BKG-173-13C





SSK-20-BKG-173-HHCOSY



Current Data Parameters
 NAME SSK-20-BKG-173-HHCOSY
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180322
 Time 5.25
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG cosygpqf
 TD 2048
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 2253.606 Hz
 FIDRES 1.100393 Hz
 AQ 0.4543829 sec
 RG 57
 DW 221.867 usec
 DE 6.50 usec
 TE 296.2 K
 D0 0.00000300 sec
 D1 1.0000000 sec
 D13 0.00000400 sec
 D16 0.00020000 sec
 IN0 0.00044375 sec

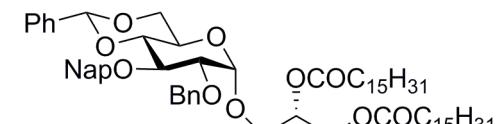
===== CHANNEL f1 ======
 NUC1 1H
 P0 14.75 usec
 P1 14.75 usec
 PLL -1.00 dB
 PL1W 10.56200695 W
 SF01 400.1312297 MHz

===== GRADIENT CHANNEL =====
 GPNAME[1] SINE.100
 GPZ1 10.00 %
 P16 1000.00 usec

F1 - Acquisition parameters
 TD 47
 SF01 400.1312 MHz
 FIDRES 95.898689 Hz
 SW 5.632 ppm
 FnMODE QF

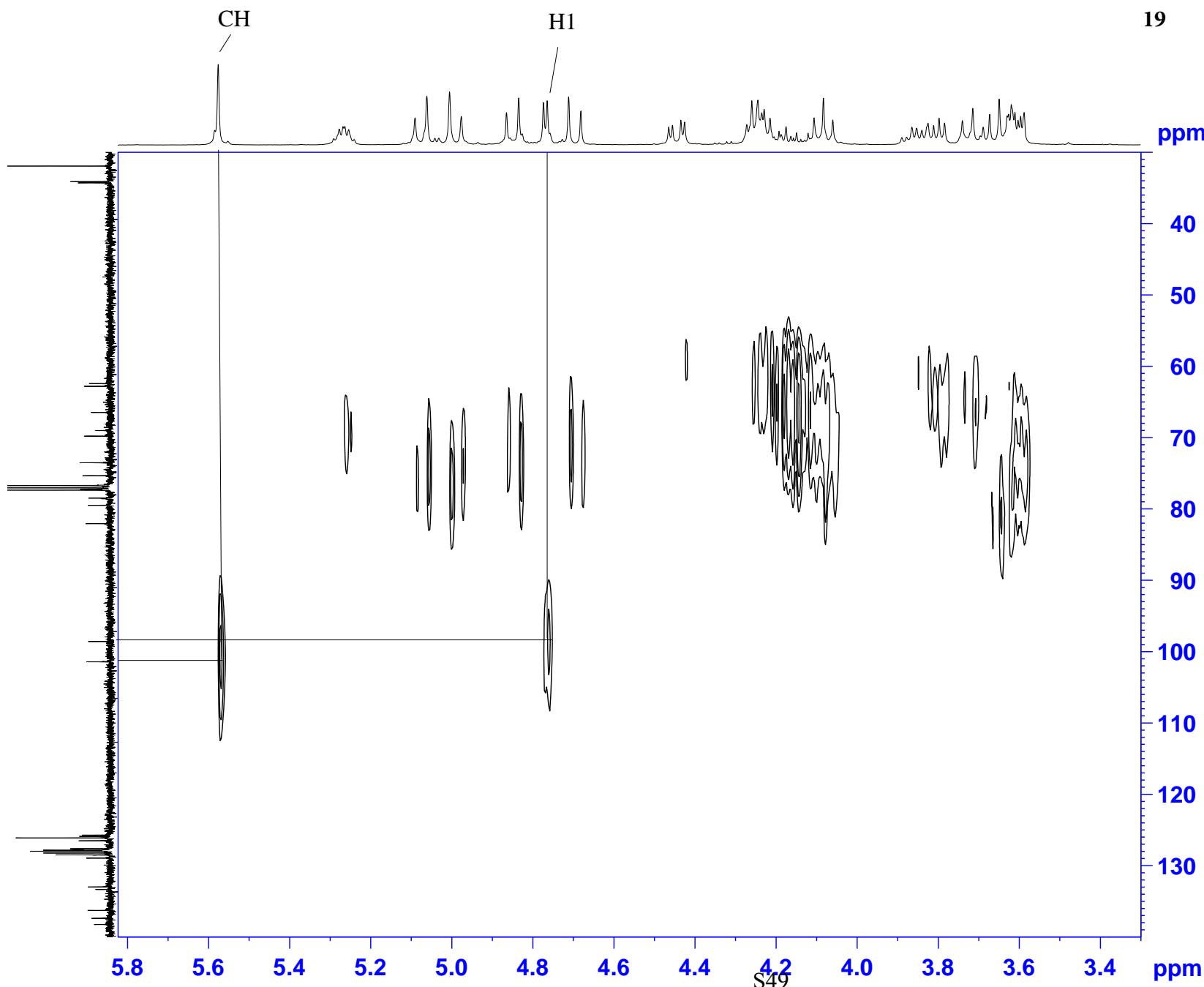
F2 - Processing parameters
 SI 2048
 SF 400.1300095 MHz
 WDW SINE
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
 SI 512
 MC2 QF
 SF 400.1300095 MHz
 WDW SINE
 SSB 0
 LB 0 Hz
 GB 0



19

SSK-20-BKG-173-HSQC



Current Data Parameters
 NAME SSK-20-BKG-173-HSQC
 EXPNO 6
 PROCNO 1

F2 - Acquisition Parameters
 Date 20180322
 Time 8.08
 INSTRUM spect
 PROBHD 5 mm FABBO BB-PULPROG
 PULPROG hsqcetgp
 TD 2048
 SOLVENT CDCl₃
 NS 4
 DS 0
 SWH 2185.315 Hz
 FIDRES 1.067048 Hz
 AQ 0.4685824 sec
 RG 1820
 DW 228.800 usec
 DE 6.50 usec
 TE 296.8 K
 CNST2 145.000000
 D0 0.0000300 sec
 DI 1.0000000 sec
 D4 0.00172414 sec
 D11 0.0300000 sec
 D13 0.00000400 sec
 D16 0.00020000 sec
 IN0 0.00002070 sec
 ZGOPTNS

===== CHANNEL f1 ======
 NUC1 1H
 P1 14.75 usec
 P2 29.50 usec
 P28 0.10 usec
 PL1 -1.00 dB
 PL1W 10.56200695 W
 SFO1 400.1312468 MHz

===== CHANNEL f2 ======
 CDPFG[2] garp
 NUC2 13C
 P3 8.50 usec
 P4 17.00 usec
 PCPD2 70.00 usec
 PL2 -2.00 dB
 PL12 16.31 dB
 PL2W 56.53121948 W
 PL12W 0.83423501 W
 SFO2 100.6248425 MHz

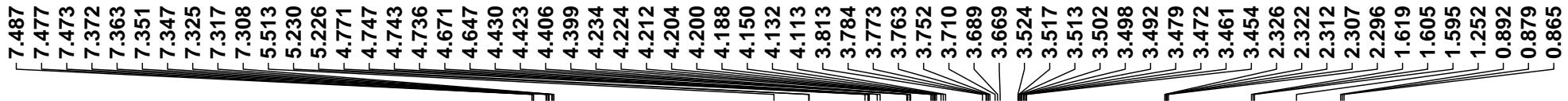
===== GRADIENT CHANNEL =====
 GPNAM[1] SINE 100
 GPNAM[2] SINE 100
 GPZ1 80.00 %
 GPZ2 20.10 %
 PL6 1000.00 usec

F1 - Acquisition parameters
 TD 16
 SFO1 100.6248 MHz
 FIDRES 3018.745361 Hz
 SW 240.000 ppm
 FRMODE Echo-Antiecho

F2 - Processing parameters
 SI 2048
 SF 400.1300095 MHz
 WDW QSINE
 SSB 2
 LB 0 Hz
 GB 0 1.40
 PC

F1 - Processing parameters
 SI 1024
 MC2 echo-antiecho
 SF 100.6127690 MHz
 WDW QSINE
 SSB 2
 LB 0 Hz
 GB 0

SSK-20-BKG-274-1H

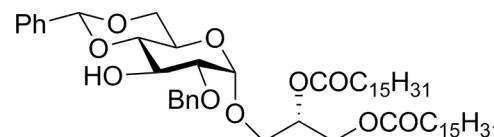


Current Data Parameters
 NAME SSK-20-BKG-274-1H
 EXPNO 7
 PROCNO 1

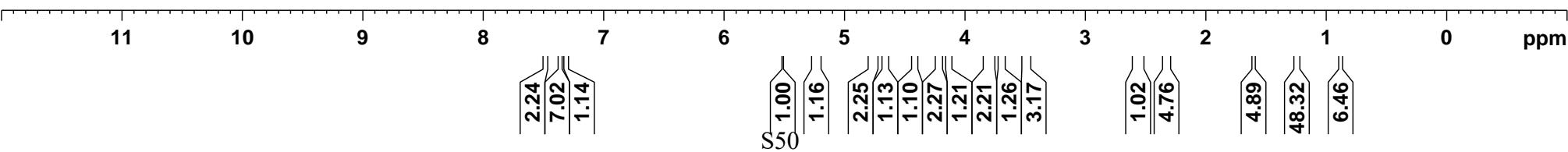
F2 - Acquisition Parameters
 Date 20180308
 Time 22.11
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 10
 DS 0
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2767999 sec
 RG 30.72
 DW 50.000 usec
 DE 6.50 usec
 TE 296.5 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 500.1330885 MHz
 NUC1 1H
 P1 13.35 usec
 PLW1 16.00000000 W

F2 - Processing parameters
 SI 65536
 SF 500.1300131 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



9



SSK-20-BKG-MONOA-

Current Data Parameters
 NAME SSK-20-BKG-MONOA-13C
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date 20180106
 Time 5.45
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 28
 DS 0
 SWH 26041.666 Hz
 FIDRES 0.397364 Hz
 AQ 1.2582912 sec
 RG 1030
 DW 19.200 usec
 DE 6.50 usec
 TE 297.2 K
 D1 1.0000000 sec
 D11 0.03000000 sec
 TD0 1

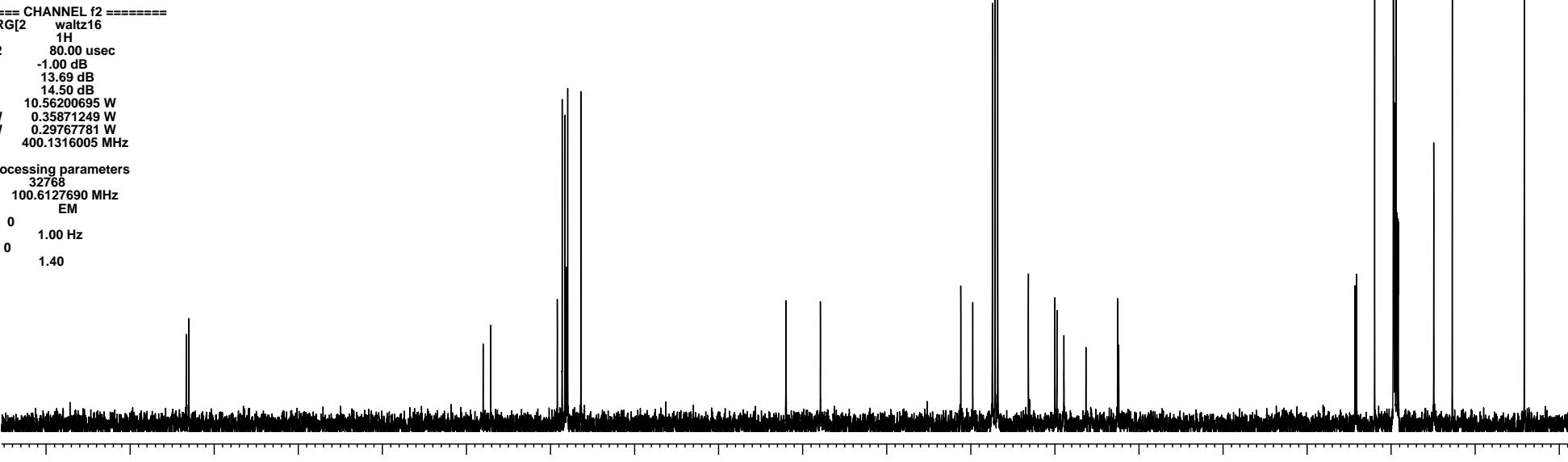
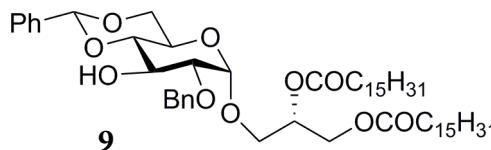
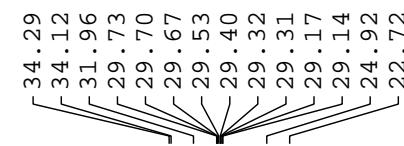
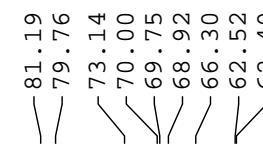
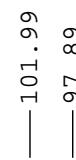
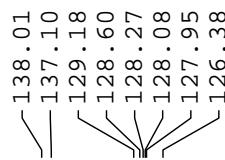
===== CHANNEL f1 ======

NUC1 13C
 P1 8.50 usec
 PL1 -2.00 dB
 PL1W 56.53121948 W
 SF01 100.6238364 MHz

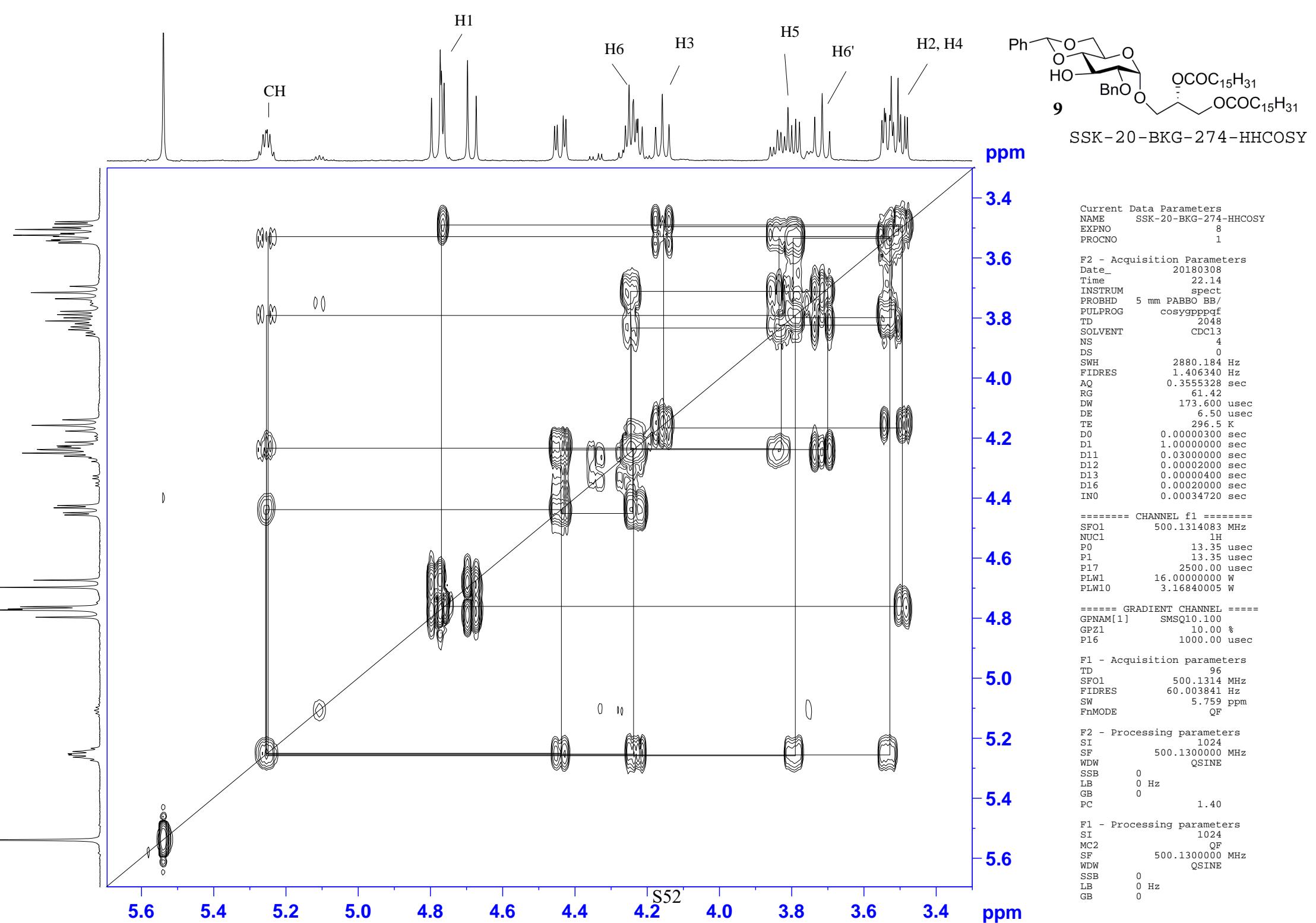
===== CHANNEL f2 ======

CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 13.69 dB
 PL13 14.50 dB
 PL2W 10.56200695 W
 PL12W 0.35871249 W
 PL13W 0.29767781 W
 SF02 400.1316005 MHz

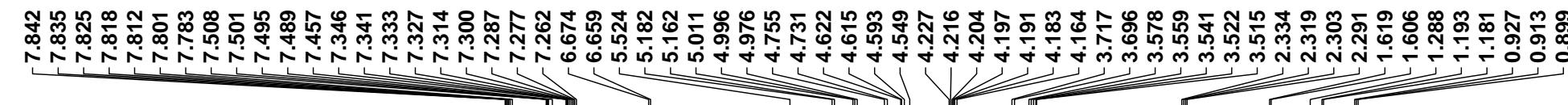
F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



S51



SSK-20-BKG-103-1H



Current Data Parameters
NAME SSK-20-BKG-103-1H
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters

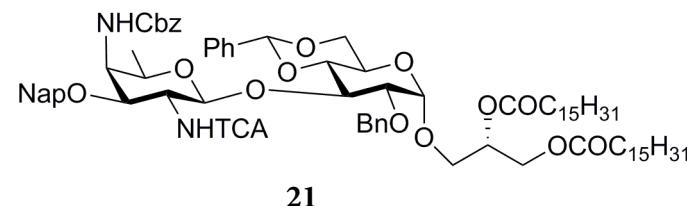
Date 20170325
Time 0.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 30.72
DW 50.000 usec
DE 6.50 usec
TE 298.8 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====

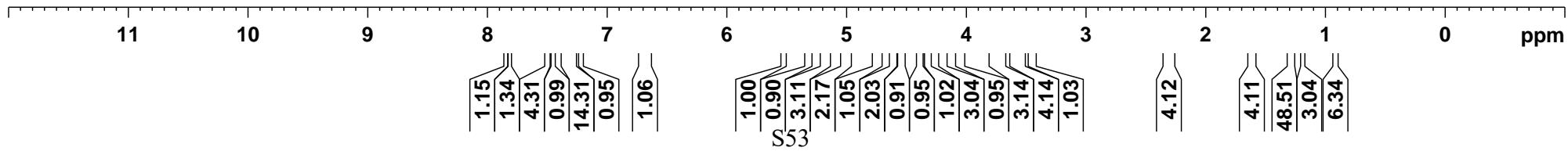
SFO1 500.1330885 MHz
NUC1 1H
P1 13.00 usec
PLW1 13.0000000 W

F2 - Processing parameters

SI 65536
SF 500.1299999 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



21



SSK-20-BKG-103-13C

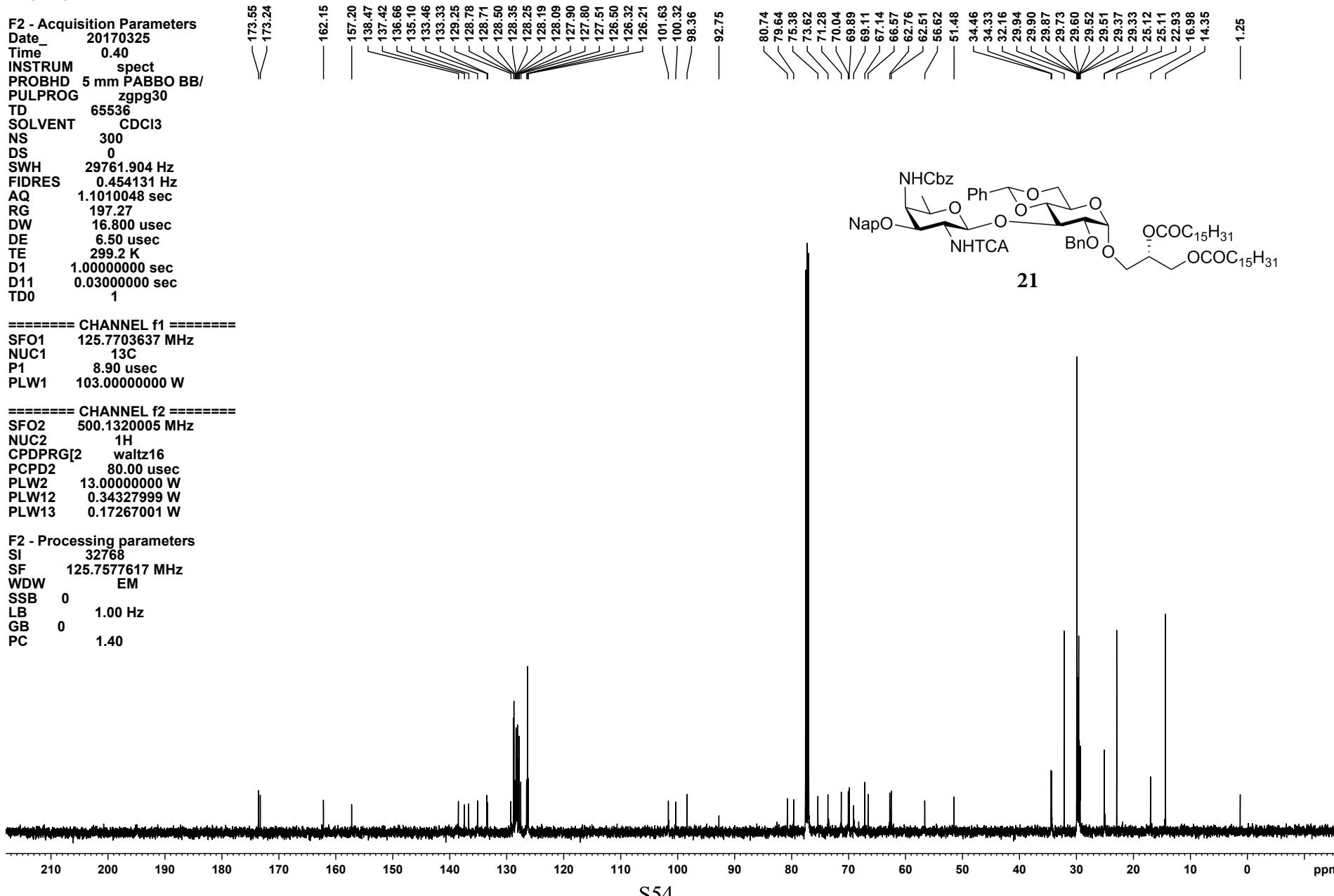
Current Data Parameters
 NAME SSK-20-BKG-103-13C
 EXPNO 21
 PROCNO 1

F2 - Acquisition Parameters
 Date 20170325
 Time 0.40
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl₃
 NS 300
 DS 0
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010048 sec
 RG 197.27
 DW 16.800 usec
 DE 6.50 usec
 TE 299.2 K
 D1 1.0000000 sec
 D11 0.03000000 sec
 TD0 1

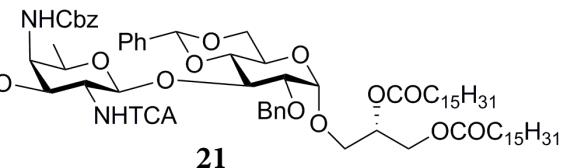
===== CHANNEL f1 ======
 SFO1 125.7703637 MHz
 NUC1 ¹³C
 P1 8.90 usec
 PLW1 103.00000000 W

===== CHANNEL f2 ======
 SFO2 500.1320005 MHz
 NUC2 ¹H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 13.00000000 W
 PLW12 0.34327999 W
 PLW13 0.17267001 W

F2 - Processing parameters
 SI 32768
 SF 125.7577617 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

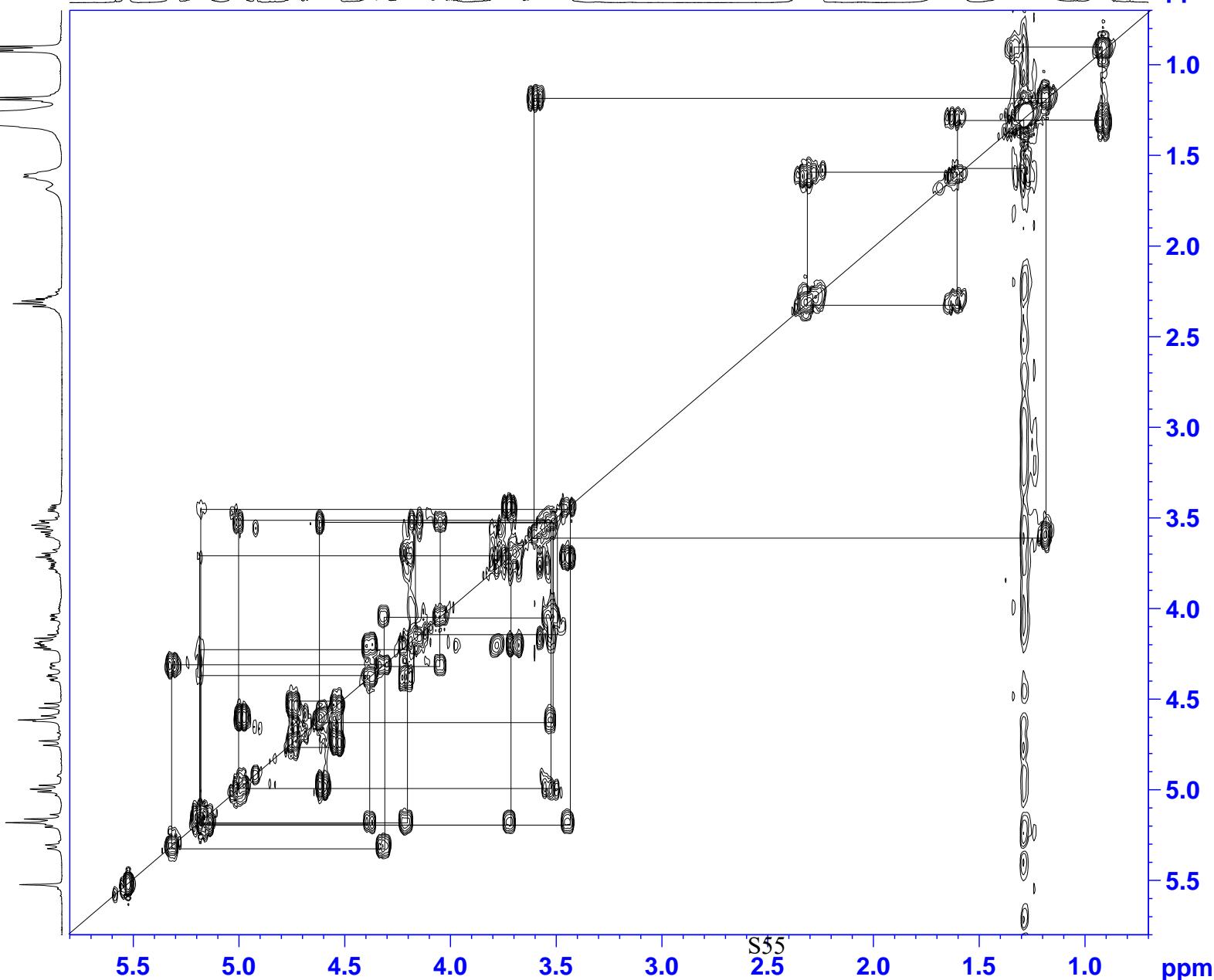


NH H1' H1 H4' H3, H4, H6'' H5', H2', H2,



SSK-20-BKG-103-HHCOSY

ppm



Current Data Parameters
NAME SSK-20-BKG-103-HHCOSY
EXPNO 22
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170325
Time 0.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG cosygpppqr
TD 2048
SOLVENT CDCl3
NS 5
DS 0
SWH 4273.504 Hz
FIDRES 2.086672 Hz
AQ 0.2396160 sec
RG 61.42
DW 117.000 usec
DE 6.50 usec
TE 298.9 K
D0 0.00000300 sec
D1 1.0000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00023400 sec

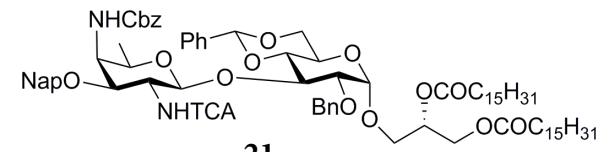
===== CHANNEL f1 =====
SFO1 500.1319787 MHz
NUC1 1H
P0 13.00 usec
P1 13.00 usec
P17 2500.00 usec
PLW1 13.0000000 W
PLW10 2.44109988 W

===== GRADIENT CHANNEL =====
GPNAME[1] SMSQ10.100
GPZ1 10.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 128
SFO1 500.132 MHz
FIDRES 66.773506 Hz
SW 8.545 ppm
FnMODE QF

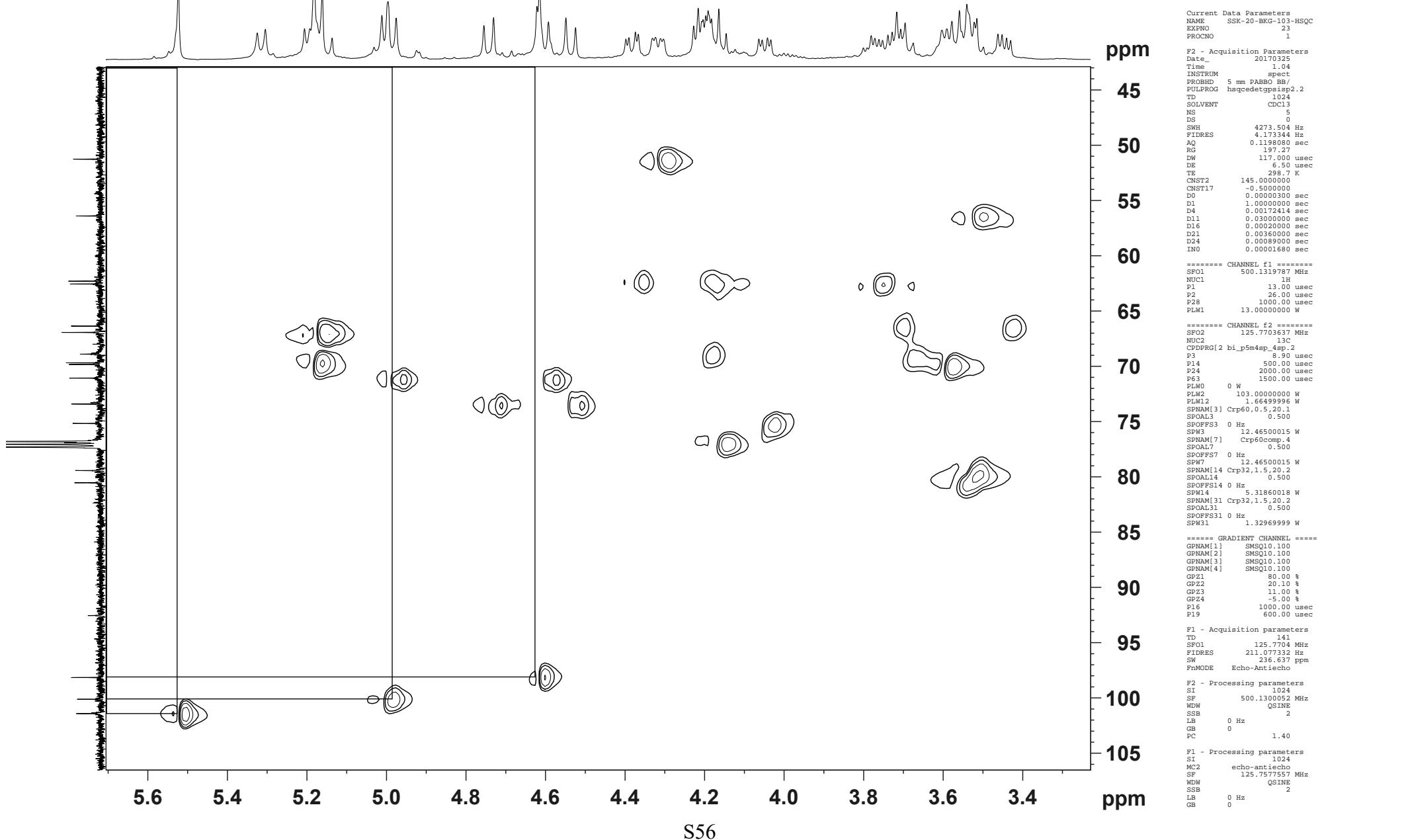
F2 - Processing parameters
SI 1024
SF 500.1300000 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

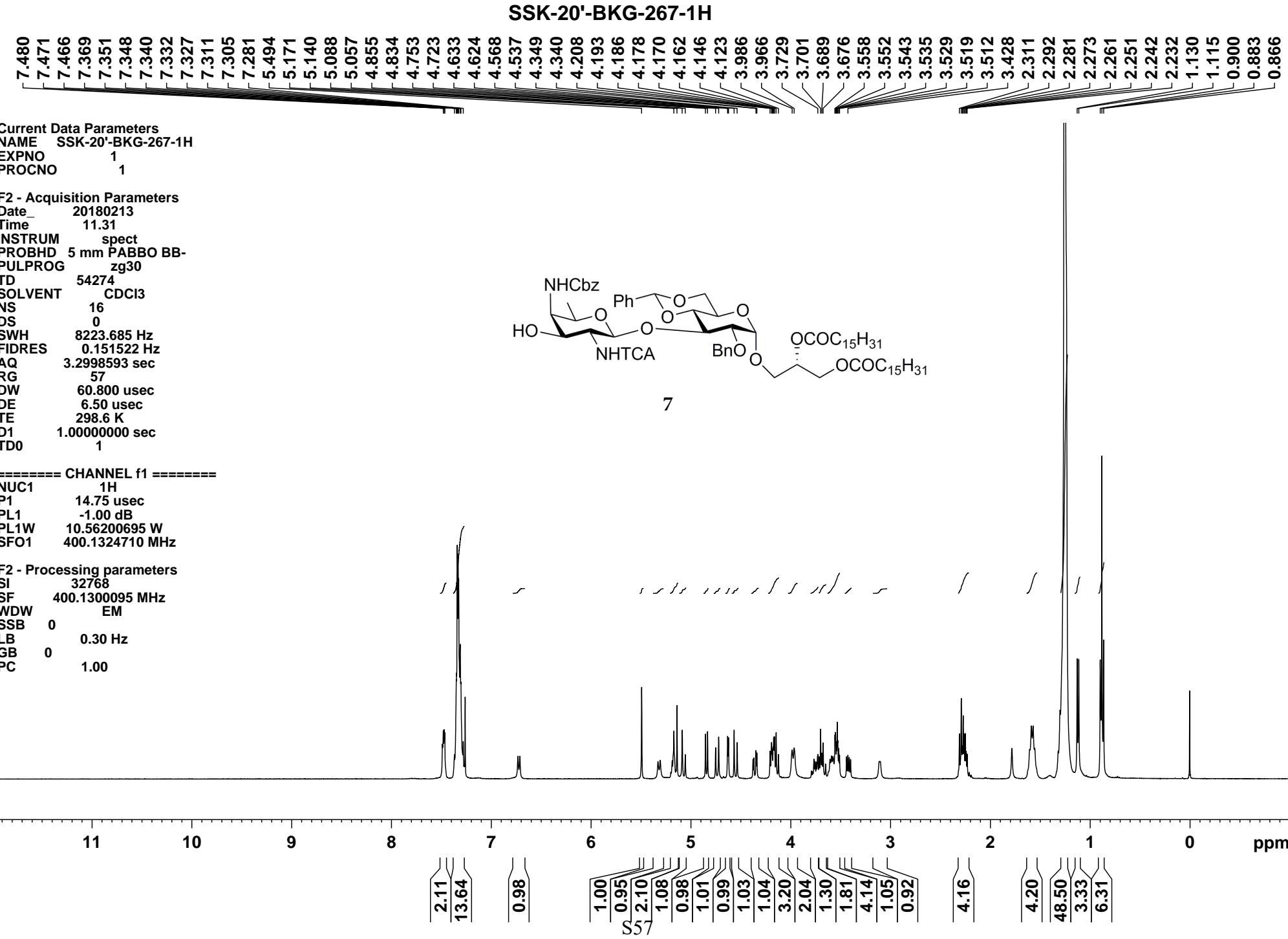
F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0



21

SSK-20-BKG-103-HSQC





SSK-20-BKG-267-1H

Current Data Parameters
 NAME SSK-20-BKG-267-1H
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date 20180213
 Time 15.30
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 269
 DS 0
 SWH 26041.666 Hz
 FIDRES 0.397364 Hz
 AQ 1.2582912 sec
 RG 28.5
 DW 19.200 usec
 DE 6.50 usec
 TE 297.6 K
 D1 1.0000000 sec
 D11 0.03000000 sec
 TDO 1

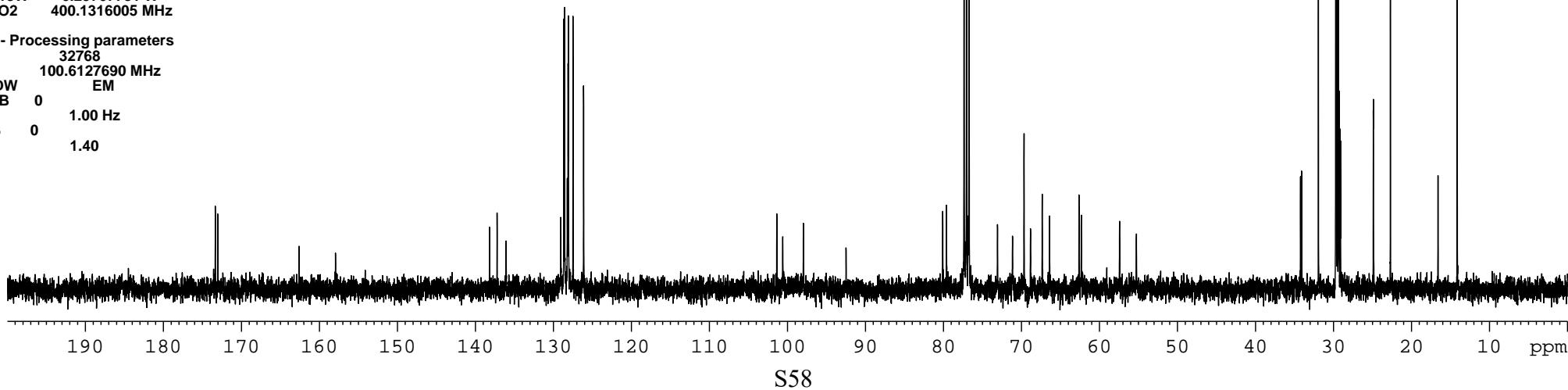
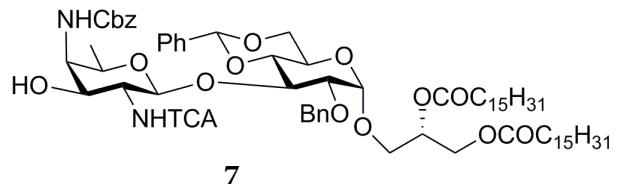
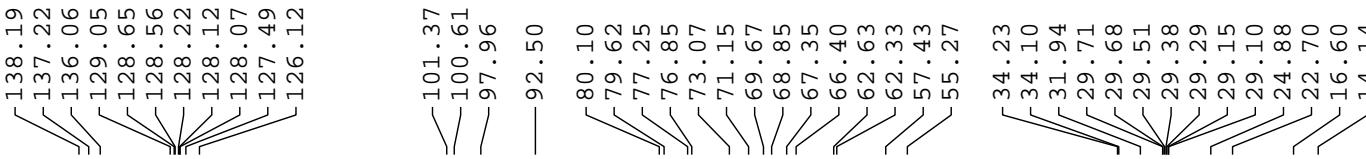
===== CHANNEL f1 =====

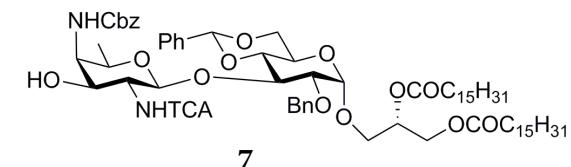
NUC1 13C
 P1 8.50 usec
 PL1 -2.00 dB
 PL1W 56.53121948 W
 SFO1 100.6238364 MHz

===== CHANNEL f2 =====

CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 13.69 dB
 PL13 14.50 dB
 PL2W 10.56200695 W
 PL12W 0.35871249 W
 PL13W 0.29767781 W
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





ppm

SSK-20-BKG-267-HHCOSY

Current Data Parameters
NAME SSK-20-BKG-267-HHCOSY
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180213
Time 15.42
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG cosyggpf
TD 2048
SOLVENT CDCl₃
NS 16
DS 16
SWH 2076.412 Hz
FIDRES 1.013873 Hz
AQ 0.4931584 sec
RG 80.6
DW 240.800 usec
DE 6.50 usec
TE 297.9 K
D0 0.00000300 sec
D1 1.00000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
INO 0.00048160 sec

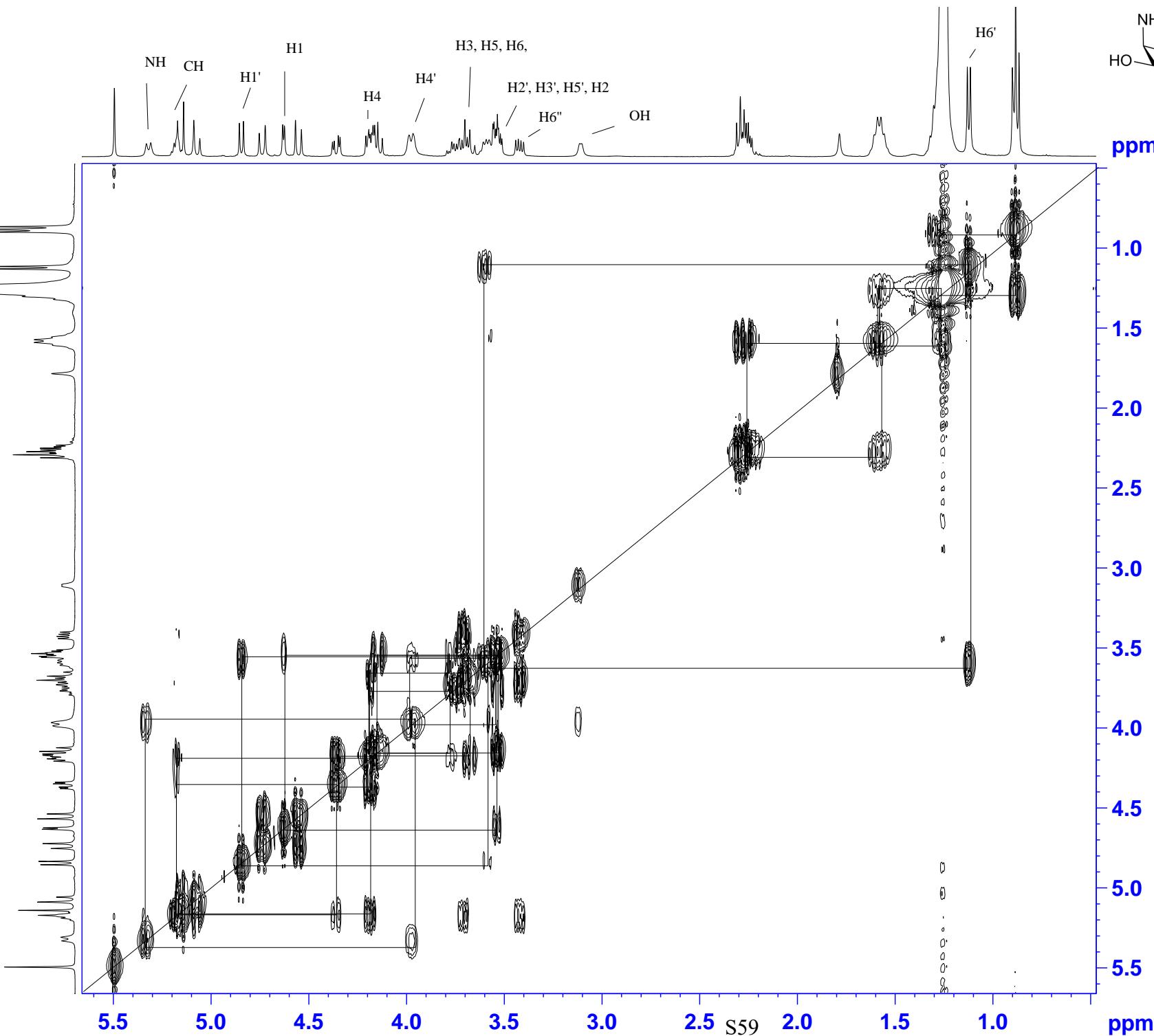
===== CHANNEL f1 =====
NUC1 1H
P0 14.75 usec
P1 14.75 usec
PL1 -1.00 dB
PL1W 10.56200695 W
SF01 400.1312368 MHz

===== GRADIENT CHANNEL =====
GPNAME[1] SINE.100
GPZ1 10.00 %
P16 1000.00 usec

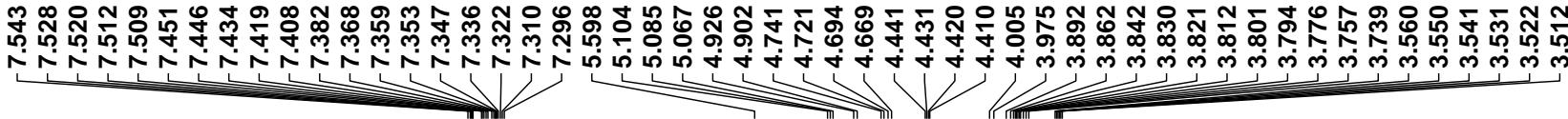
F1 - Acquisition parameters
TD 35
SF01 400.1312368 MHz
FIDRES 118.651489 Hz
SW 5.189 ppm
FnMODE QF

F2 - Processing parameters
SI 2048
SF 400.1300101 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 512
MC2 QF
SF 400.1300101 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0



SSK-20-BKG-261-1H



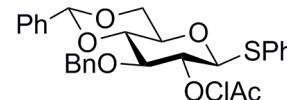
Current Data Parameters
 NAME SSK-20-BKG-261-1H
 EXPNO 14
 PROCNO 1

F2 - Acquisition Parameters

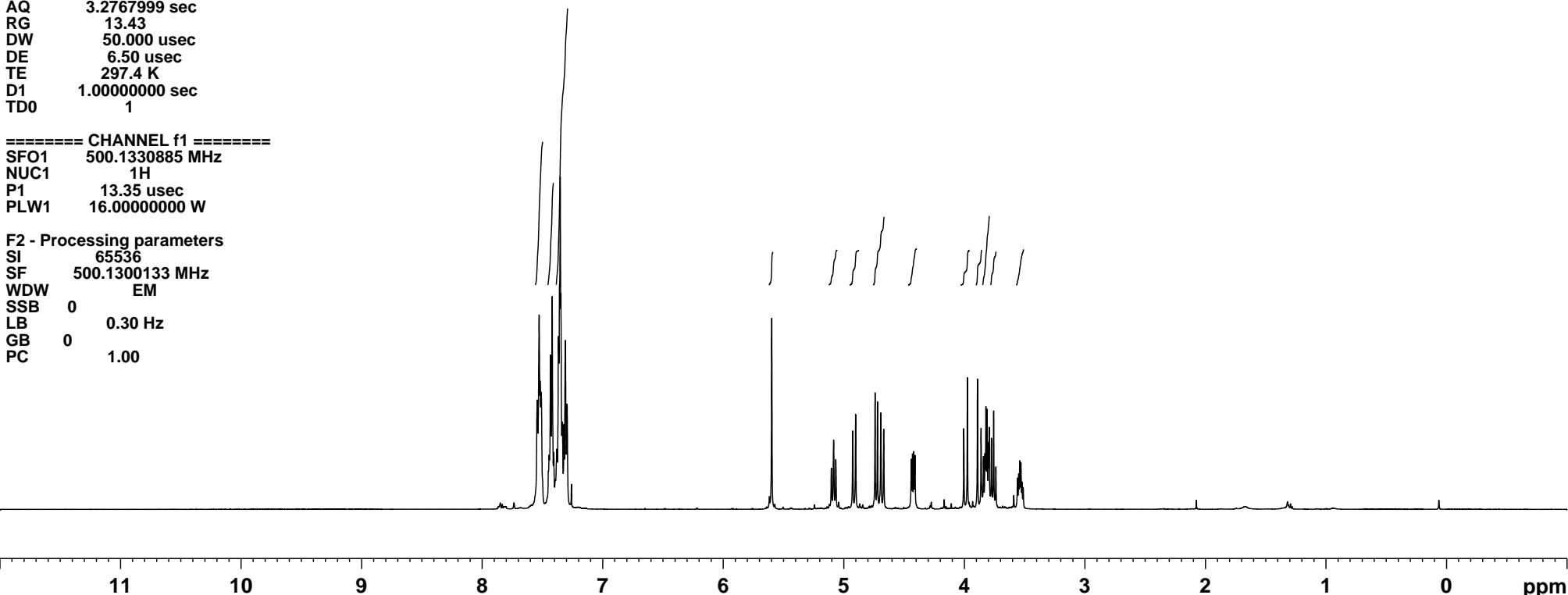
Date_ 20180206
 Time 22.15
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 11
 DS 0
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2767999 sec
 RG 13.43
 DW 50.000 usec
 DE 6.50 usec
 TE 297.4 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 500.1330885 MHz
 NUC1 1H
 P1 13.35 usec
 PLW1 16.00000000 W

F2 - Processing parameters
 SI 65536
 SF 500.1300133 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



20a



4.35
3.12
8.39

1.00
1.06
1.05
2.07
1.11
1.06
1.08
2.12
1.03
1.07

Current Data Parameters
NAME SSK-20-BKG-261-13C
EXPNO 15
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180206
Time 22.18
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 51
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 197.27
DW 16.800 usec
DE 6.50 usec
TE 297.9 K
D1 1.0000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 125.7703637 MHz
NUC1 13C
P1 8.90 usec
PLW1 103.00000000 W

===== CHANNEL f2 =====
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 16.00000000 W
PLW12 0.44556001 W
PLW13 0.22411001 W

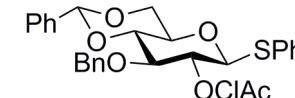
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

SSK-20-BKG-261-13C

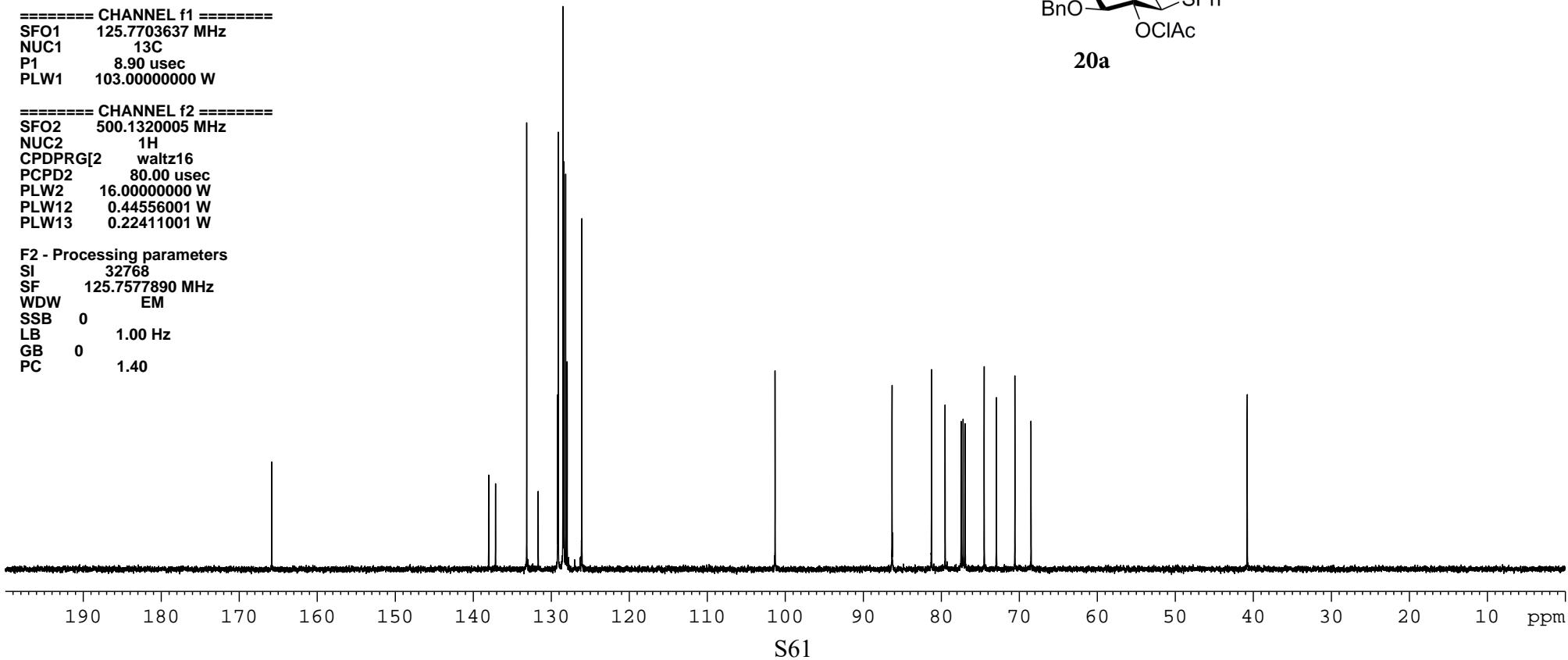
— 101.31

— 165.84
138.03
137.14
133.15
131.67
129.18
129.10
128.50
128.39
128.15
127.96
126.09

— 40.79



20a



H2

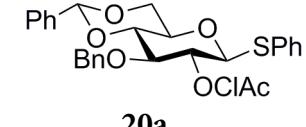
H1

H6'

H3, H6

H5

H4

**20a****ppm**

SSK-20-BKG-261-HHCOSY

Current Data Parameters
 NAME SSK-20-BKG-261-HHCOSY
 EXPNO 17
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180206
 Time 22.19
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG cosygpppof
 TD 2048
 SOLVENT CDCl3
 NS 4
 DS 8
 SWH 1650.165 Hz
 FIDRES 0.805745 Hz
 AQ 0.6205440 sec
 RG 42.12
 DW 303.000 usec
 DE 6.50 usec
 TE 297.6 K
 D0 0.0000000 sec
 D1 1.0000000 sec
 D11 0.0300000 sec
 D12 0.0000200 sec
 D13 0.0000040 sec
 D16 0.0000600 sec
 IN0 0.0006060 sec

===== CHANNEL f1 =====
 SFO1 500.1322341 MHz
 NUC1 1H
 P0 13.35 usec
 P1 13.35 usec
 P17 2500.00 usec
 PLW1 16.0000000 W
 PLW10 3.16840005 W

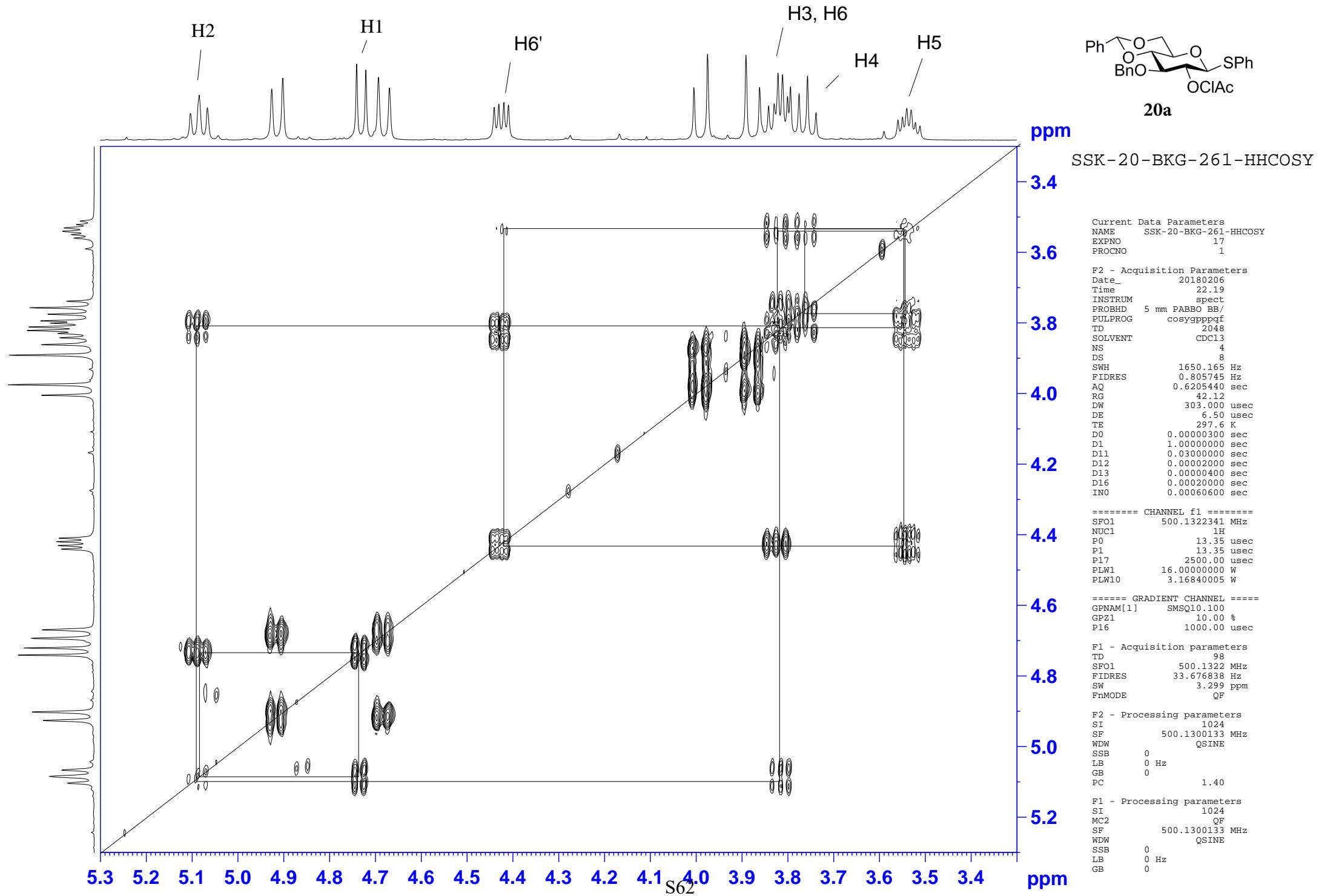
===== GRADIENT CHANNEL =====
 GPNAME[1] SMSQ10.100
 GPZ1 10.00 %
 P16 1000.00 usec

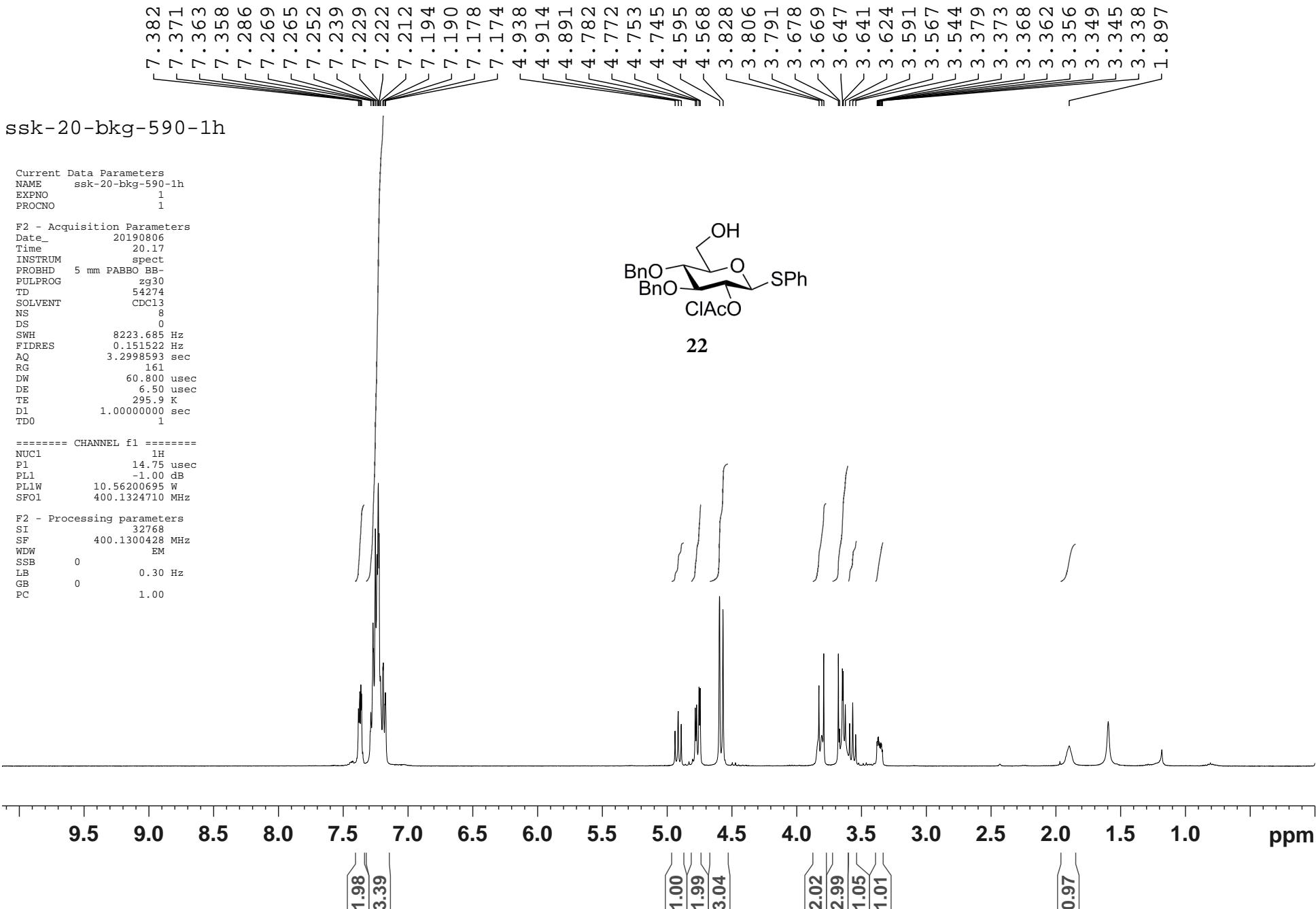
F1 - Acquisition parameters
 TD 98
 SFO1 500.1322 MHz
 FIDRES 33.676838 Hz
 SW 3.299 ppm
 FnMODE QF

F2 - Processing parameters
 SI 1024
 SF 500.1300133 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
 SI 1024
 MC2 QF
 SF 500.1300133 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0

5.3 5.2 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6 3.5 3.4 S62 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 ppm





Current Data Parameters
NAME ssk-20-bkg-590-13c
EXPNO 2
PROCNO 1

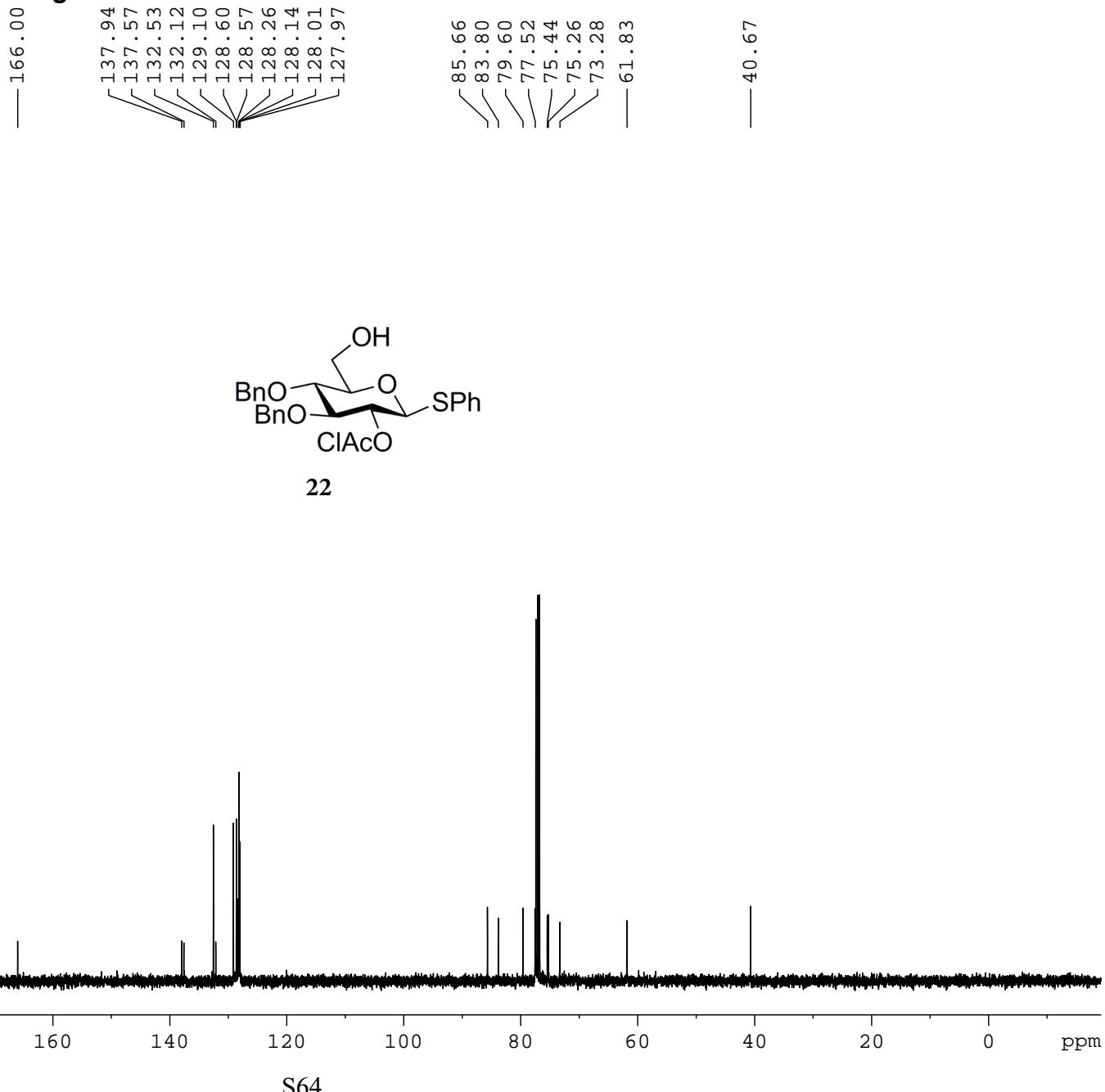
F2 - Acquisition Parameters
Date 20190806
Time 20.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2582912 sec
RG 1030
DW 19.200 usec
DE 6.50 usec
TE 296.5 K
D1 1.0000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 56.53121948 W
SFO1 100.6238364 MHz

===== CHANNEL f2 =====
CPDPGRG[2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 13.69 dB
PL13 14.50 dB
PL2W 10.56200695 W
PL12W 0.35871249 W
PL13W 0.29767781 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

ssk-20-bkg-590-13c



SSK-20-BKG-590-1dept

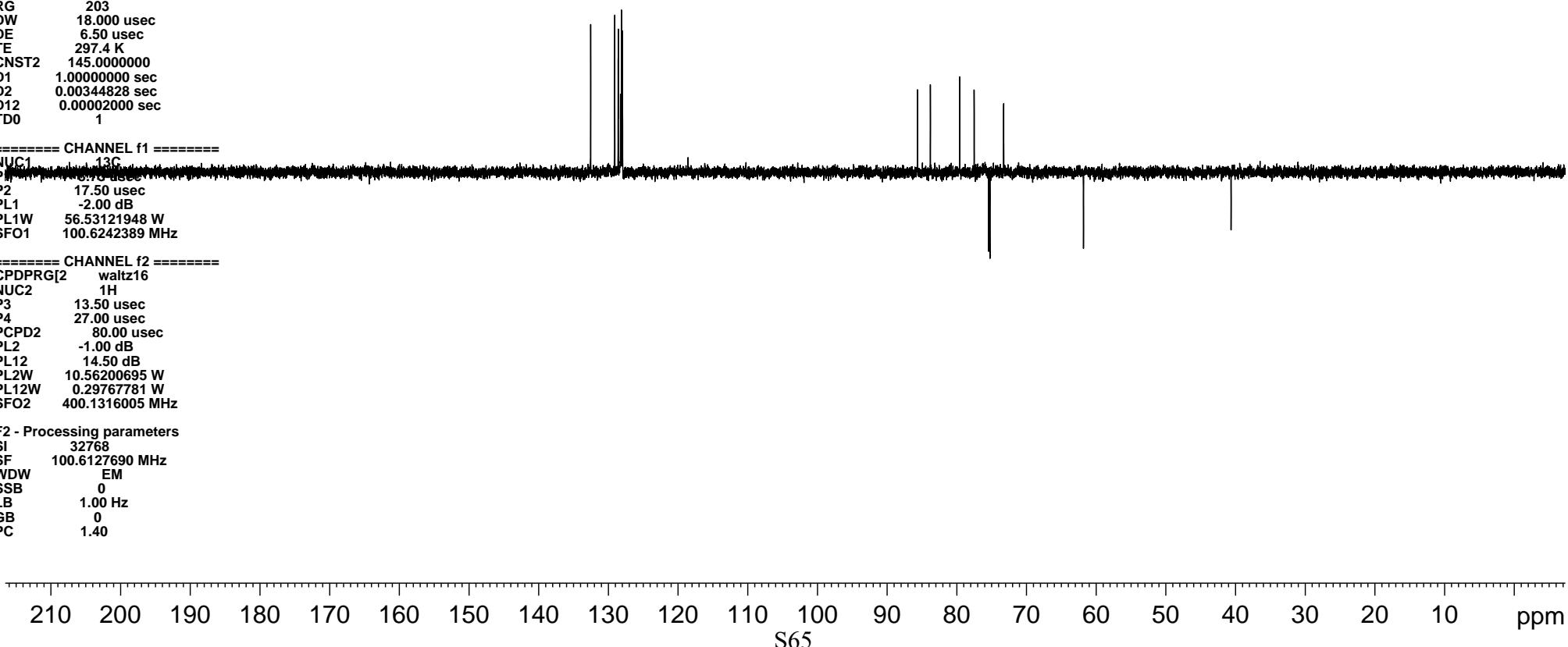
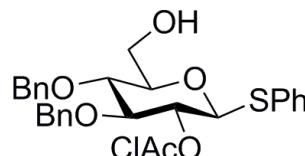
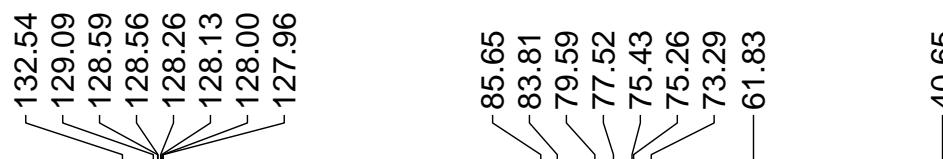
Current Data Parameters
 NAME SSK-20-BKG-590-1dept
 EXPNO 9
 PROCNO 1

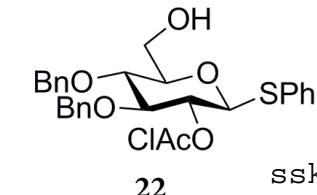
F2 - Acquisition Parameters
 Date 20190808
 Time 0.02
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG dept135
 TD 65536
 SOLVENT CDCl3
 NS 153
 DS 0
 SWH 27777.777 Hz
 FIDRES 0.423855 Hz
 AQ 1.1796480 sec
 RG 203
 DW 18.000 usec
 DE 6.50 usec
 TE 297.4 K
 CNST2 145.000000
 D1 1.0000000 sec
 D2 0.00344828 sec
 D12 0.00002000 sec
 TD0 1

===== CHANNEL f1 ======
 NUC1 13C
 P1 6.00 usec
 P2 17.50 usec
 PL1 -2.00 dB
 PL1W 56.53121948 W
 SFO1 100.6242389 MHz

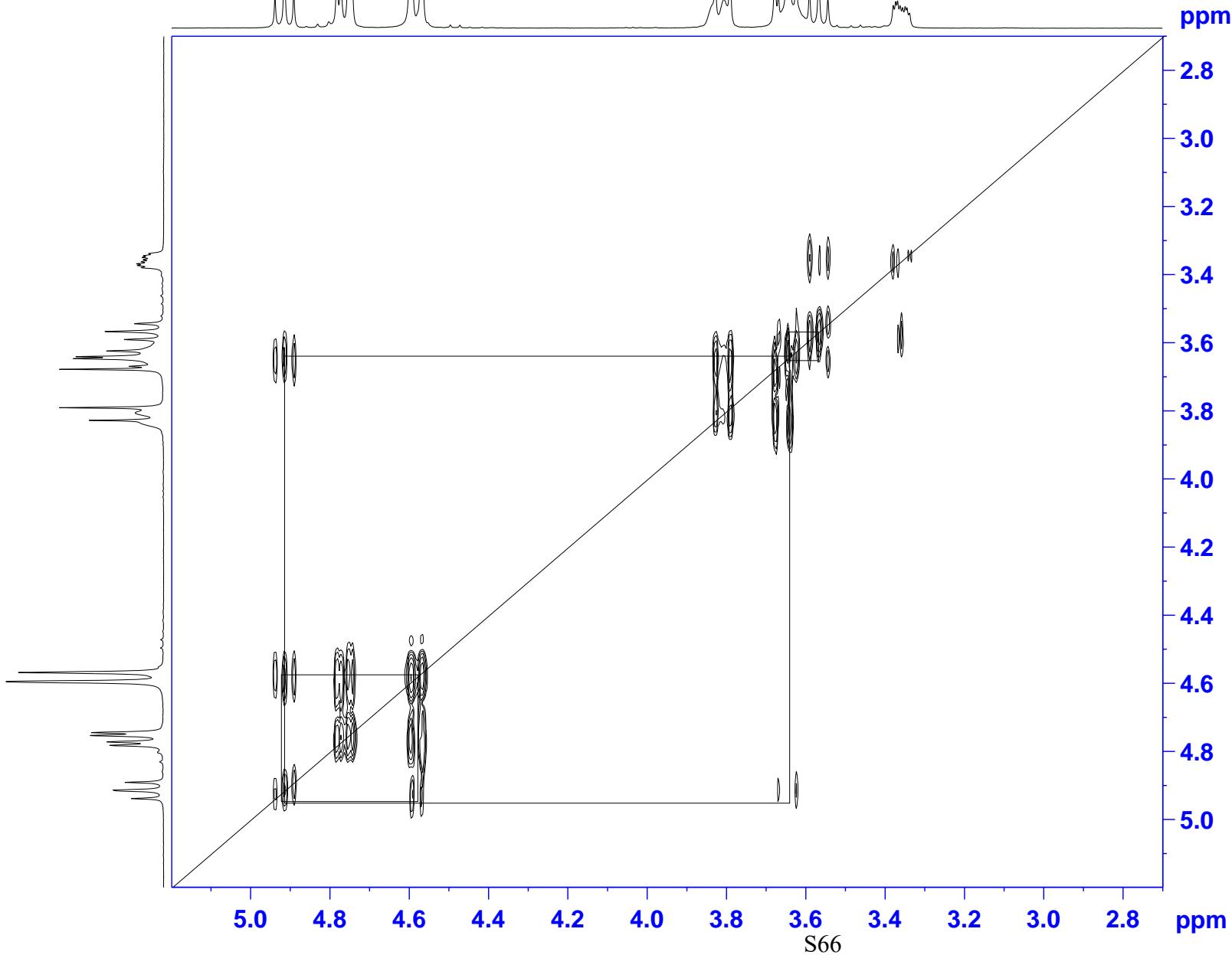
===== CHANNEL f2 ======
 CPDPRG[2 waltz16
 NUC2 1H
 P3 13.50 usec
 P4 27.00 usec
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 14.50 dB
 PL2W 10.56200695 W
 PL12W 0.29767781 W
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





ssk-20-bkg-590-cosy



Current Data Parameters
NAME ssk-20-bkg-590-cosy
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190806
Time 20.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG cosygpqf
TD 2048
SOLVENT CDCl3
NS 4
DS 0
SWH 1723.346 Hz
FIDRES 0.841477 Hz
AQ 0.5941930 sec
RG 64
DW 290.133 usec
DE 6.50 usec
TE 296.5 K
D0 0.00000300 sec
D1 1.0000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00058025 sec

===== CHANNEL f1 ======
NUC1 1H
P0 13.50 usec
P1 13.50 usec
PL1 -1.00 dB
PL1W 10.56200695 W
SF01 400.1312613 MHz

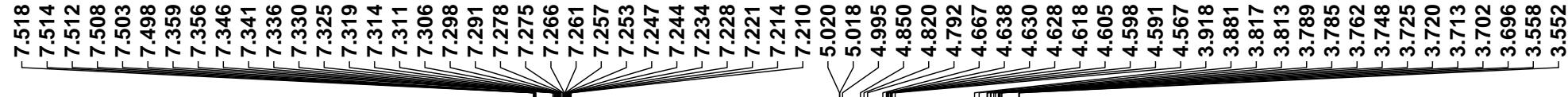
===== GRADIENT CHANNEL =====
GPNAME[1] SINE.100
GPZ1 10.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 47
SF01 400.1313 MHz
FIDRES 73.334694 Hz
SW 4.307 ppm
FnMODE QF

F2 - Processing parameters
SI 1024
SF 400.1300428 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 400.1300428 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0

SSK-20-BKG-266B-1H

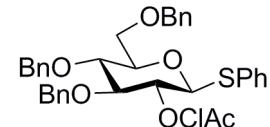


Current Data Parameters
 NAME SSK-20-BKG-266B-1H
 EXPNO 7
 PROCNO 1

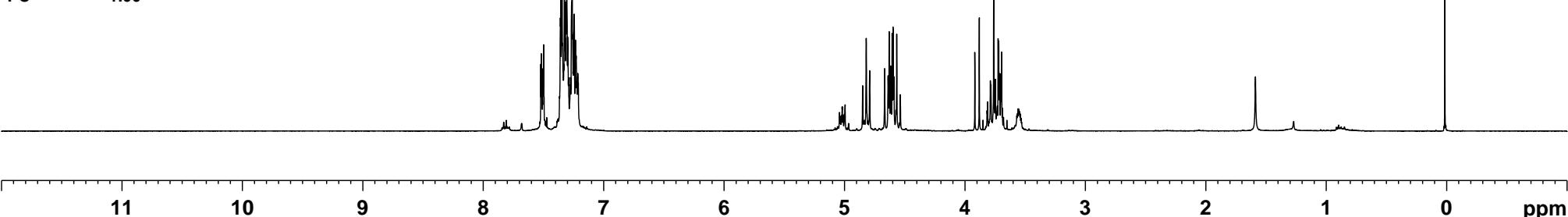
F2 - Acquisition Parameters
 Date_ 20180213
 Time 15.09
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 54274
 SOLVENT CDCl3
 NS 11
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.151522 Hz
 AQ 3.2998593 sec
 RG 101
 DW 60.800 usec
 DE 6.50 usec
 TE 297.3 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.75 usec
 PL1 -1.00 dB
 PL1W 10.56200695 W
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



5



SSK-20-BKG-266B-13C

Current Data Parameters
 NAME SSK-20-BKG-266B-13C
 EXPNO 8
 PROCNO 1

F2 - Acquisition Parameters
 Date 20180213
 Time 15.11
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 90
 DS 0
 SWH 26041.666 Hz
 FIDRES 0.397364 Hz
 AQ 1.2582912 sec
 RG 1030
 DW 19.200 usec
 DE 6.50 usec
 TE 297.8 K
 D1 1.0000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.50 usec
 PL1 -2.00 dB
 PL1W 56.53121948 W
 SFO1 100.6238364 MHz

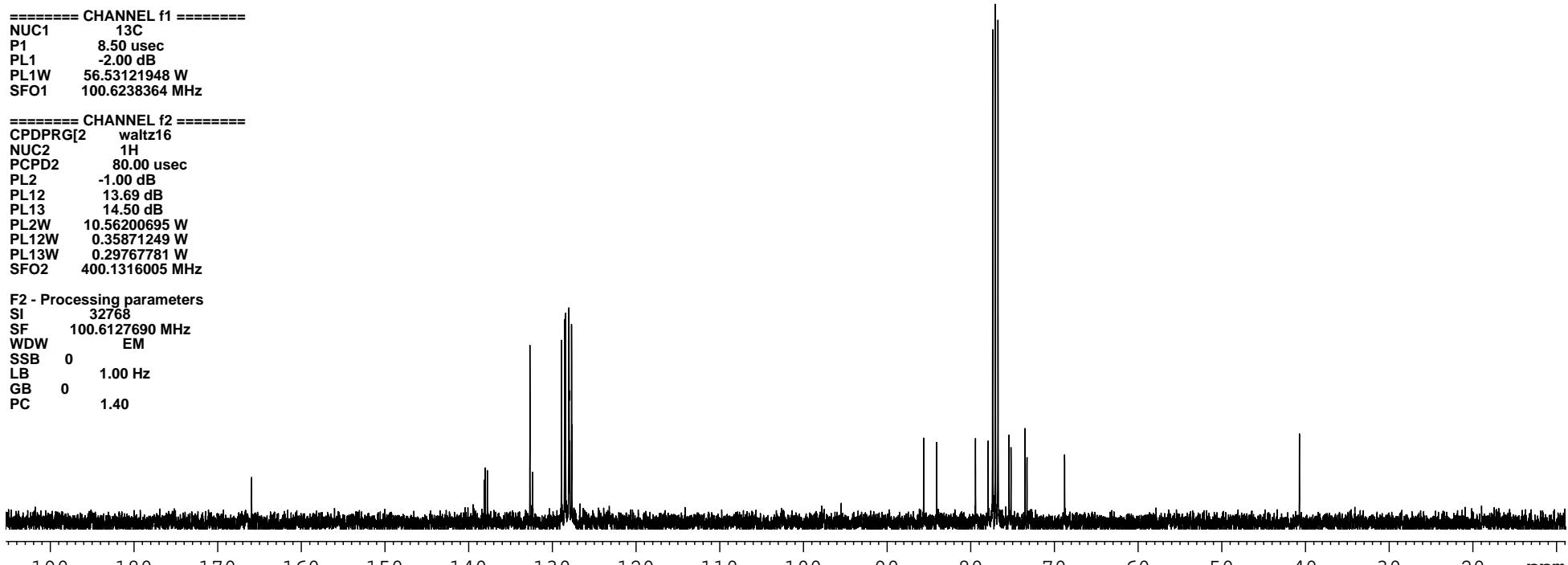
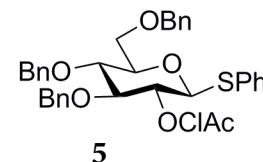
===== CHANNEL f2 =====
 CDPGR[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 13.69 dB
 PL13 14.50 dB
 PL2W 10.56200695 W
 PL12W 0.35871249 W
 PL13W 0.29767781 W
 SFO2 400.1316005 MHz

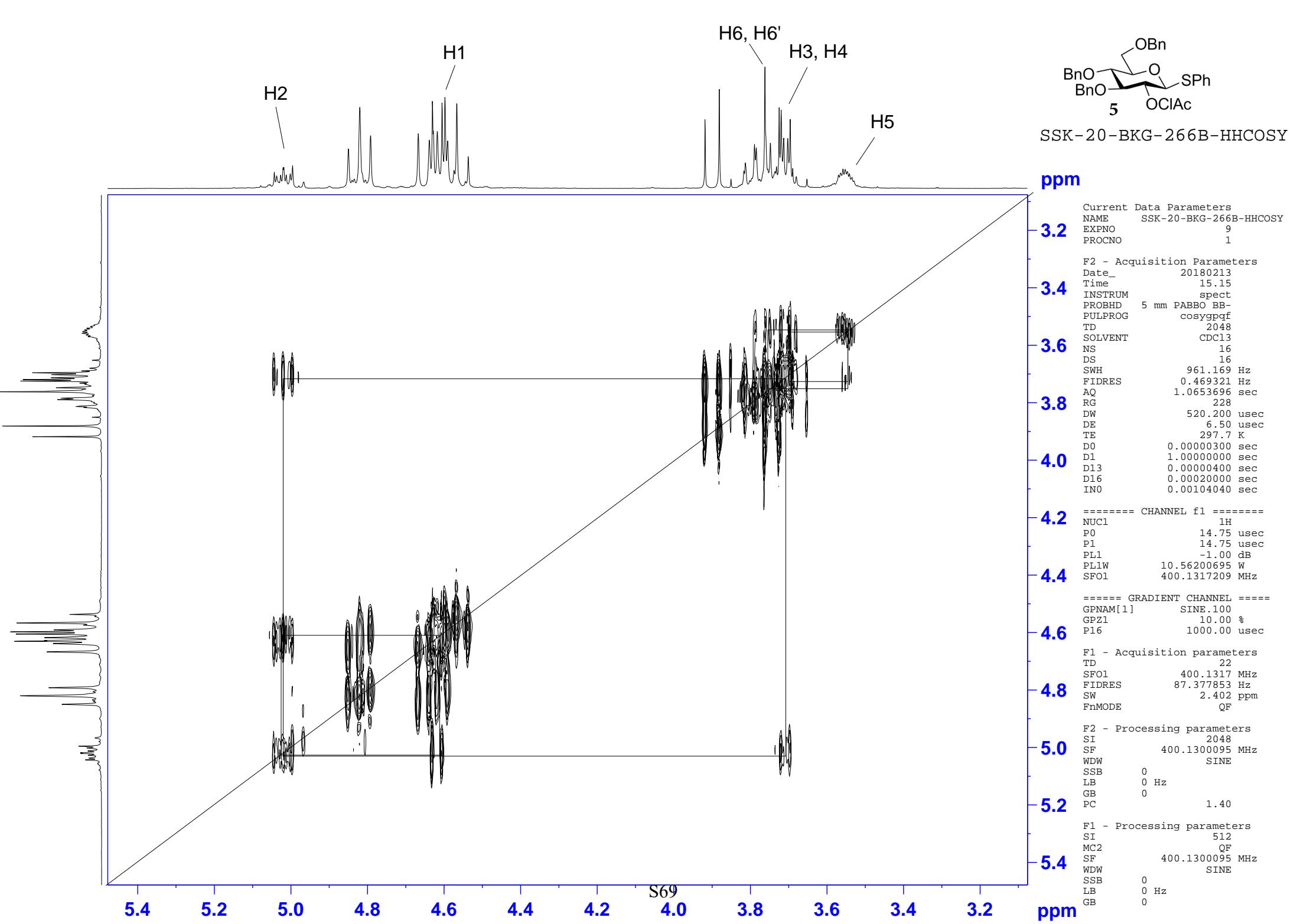
F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

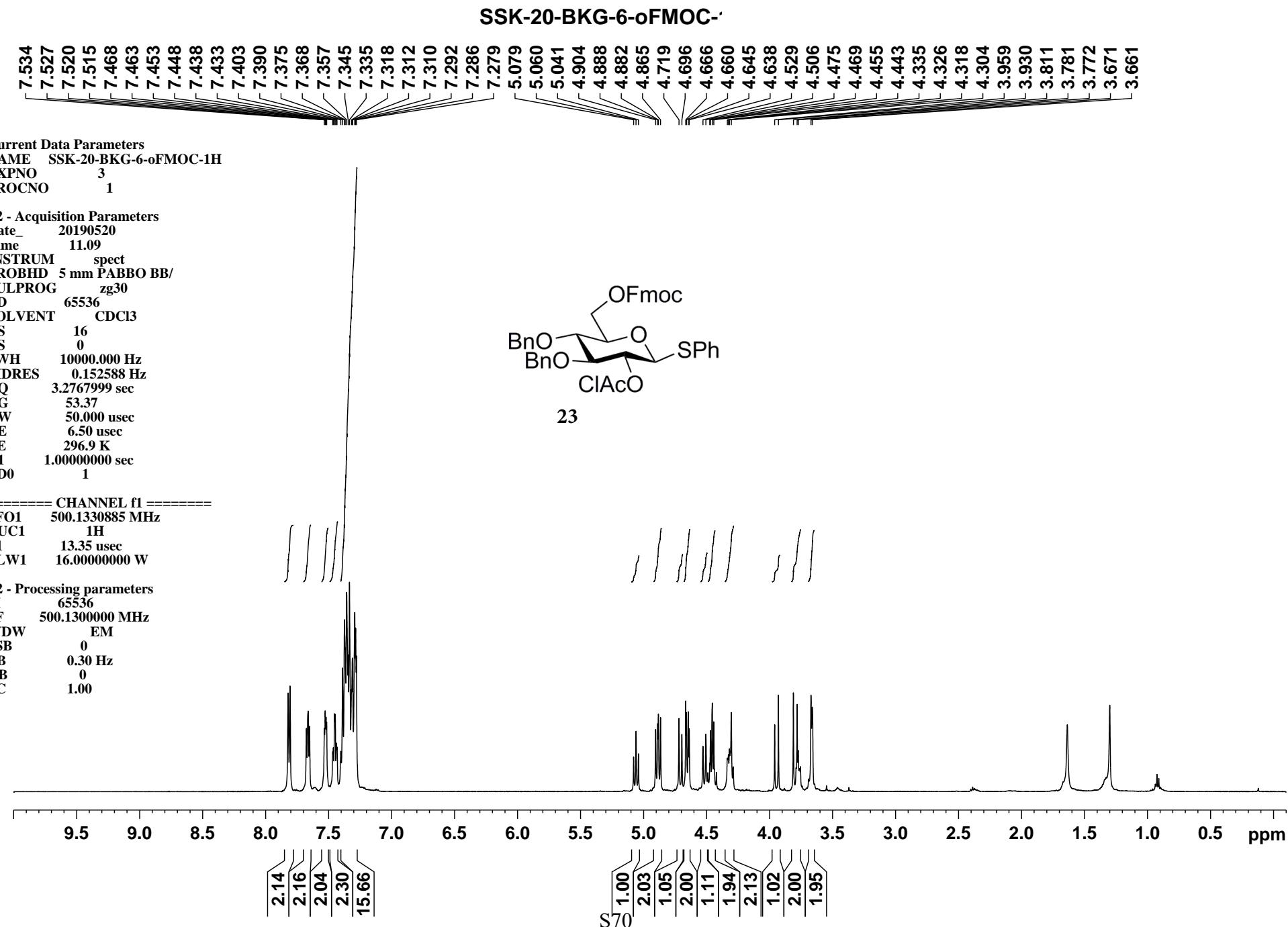
138.14
 138.02
 137.77
 132.67
 132.34
 128.91
 128.54
 128.51
 128.40
 128.04
 128.00
 127.91
 127.70
 127.65

85.62
 84.06
 79.43
 77.91
 75.41
 75.18
 73.51
 73.27
 68.77

— 40.71







SSK-20-BKG-6-oFMOC-13c

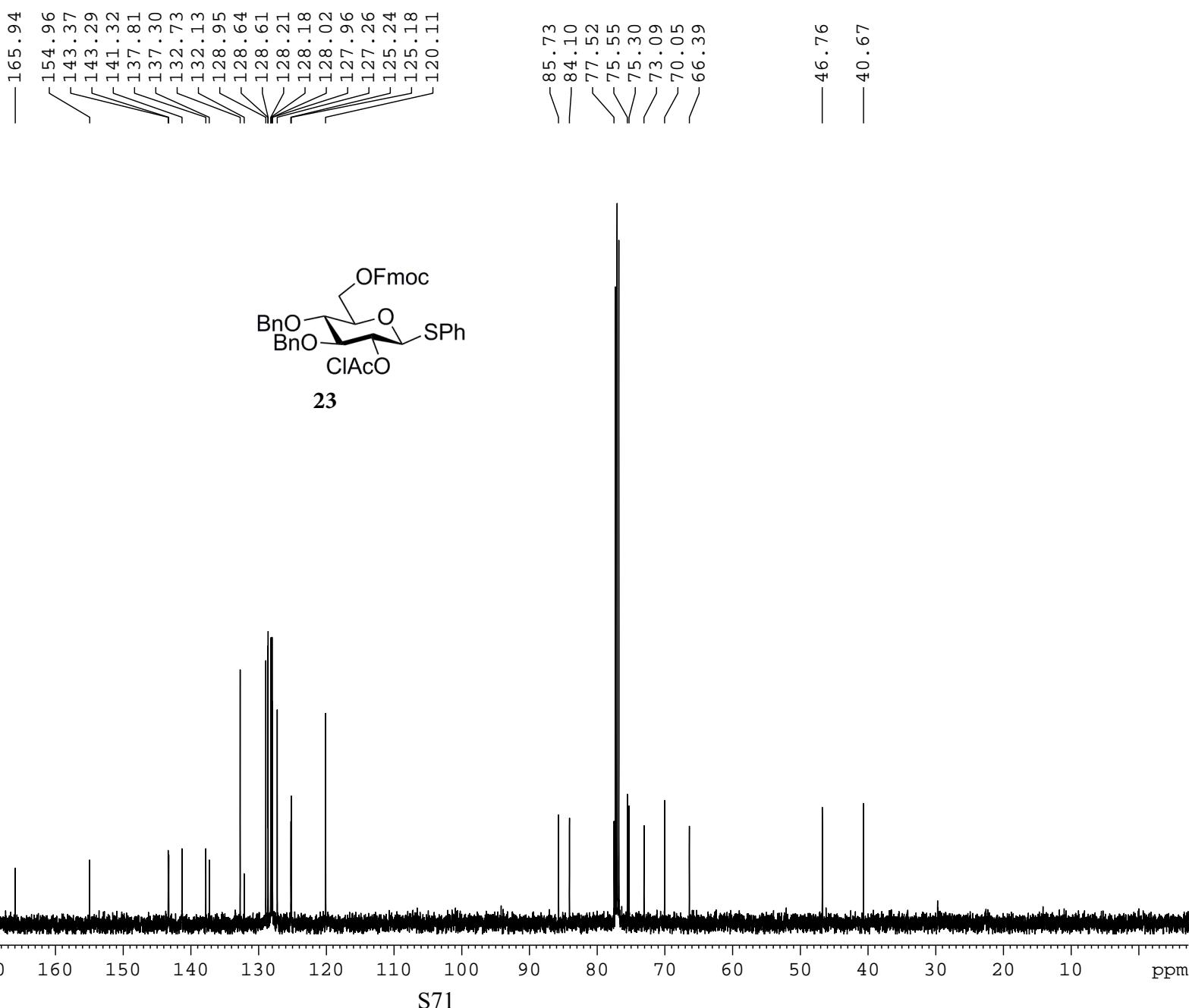
Current Data Parameters
 NAME SSK-20-BKG-6-oFMOC-13c
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date 20190520
 Time 11.12
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl₃
 NS 65
 DS 0
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010048 sec
 RG 197.27
 DW 16.800 usec
 DE 6.50 usec
 TE 297.7 K
 D1 1.0000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 125.7703637 MHz
 NUC1 13C
 P1 8.90 usec
 PLW1 103.0000000 W

===== CHANNEL f2 =====
 SFO2 500.1320005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 16.00000000 W
 PLW12 0.44556001 W
 PLW13 0.22411001 W

F2 - Processing parameters
 SI 32768
 SF 125.7577890 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



SSK-20-BKG-6-oFMOC-dept

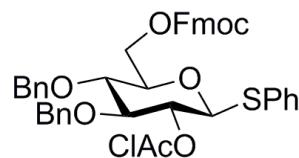
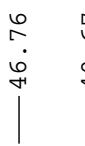
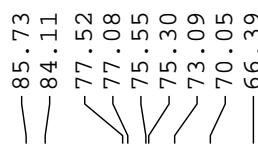
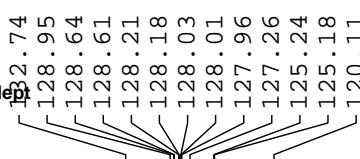
Current Data Parameters

NAME SSK-20-BKG-6-oFMOC-dept
 EXPNO 5
 PROCNO 1
 F2 - Acquisition Parameters
 Date 20190520
 Time 11.13
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG deptsp135
 TD 65536
 SOLVENT CDCl3
 NS 62
 DS 0
 SWH 20161.291 Hz
 FIDRES 0.307637 Hz
 AQ 1.6252928 sec
 RG 197.27
 DW 24.800 usec
 DE 6.50 usec
 TE 297.7 K
 CNST2 145.000000
 D1 1.0000000 sec
 D2 0.00344828 sec
 D12 0.00002000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 125.7678486 MHz
 NUC1 13C
 P1 8.90 usec
 P13 2000.00 usec
 PLW0 0 W
 PLW1 103.0000000 W
 SPNAM[5] Crp60comp.4
 SPOAL5 0.500
 SPOFFS5 0 Hz
 SPW5 12.46500015 W

===== CHANNEL f2 =====
 SFO2 500.1315995 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 P3 13.35 usec
 P4 26.70 usec
 PCPD2 80.00 usec
 PLW2 16.0000000 W
 PLW12 0.44556001 W

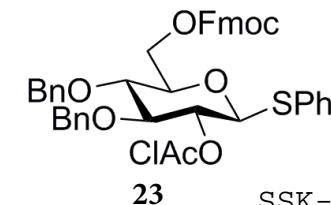
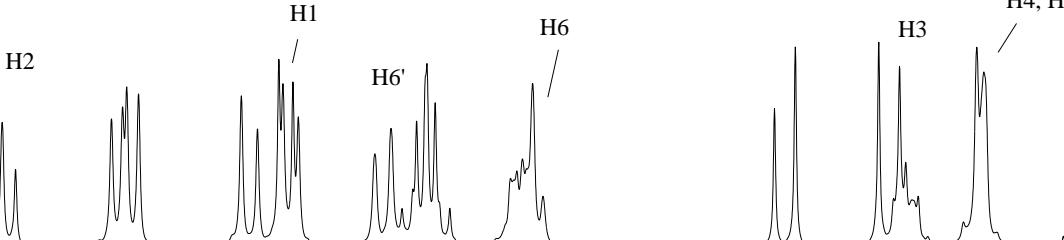
F2 - Processing parameters
 SI 32768
 SF 125.7577890 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



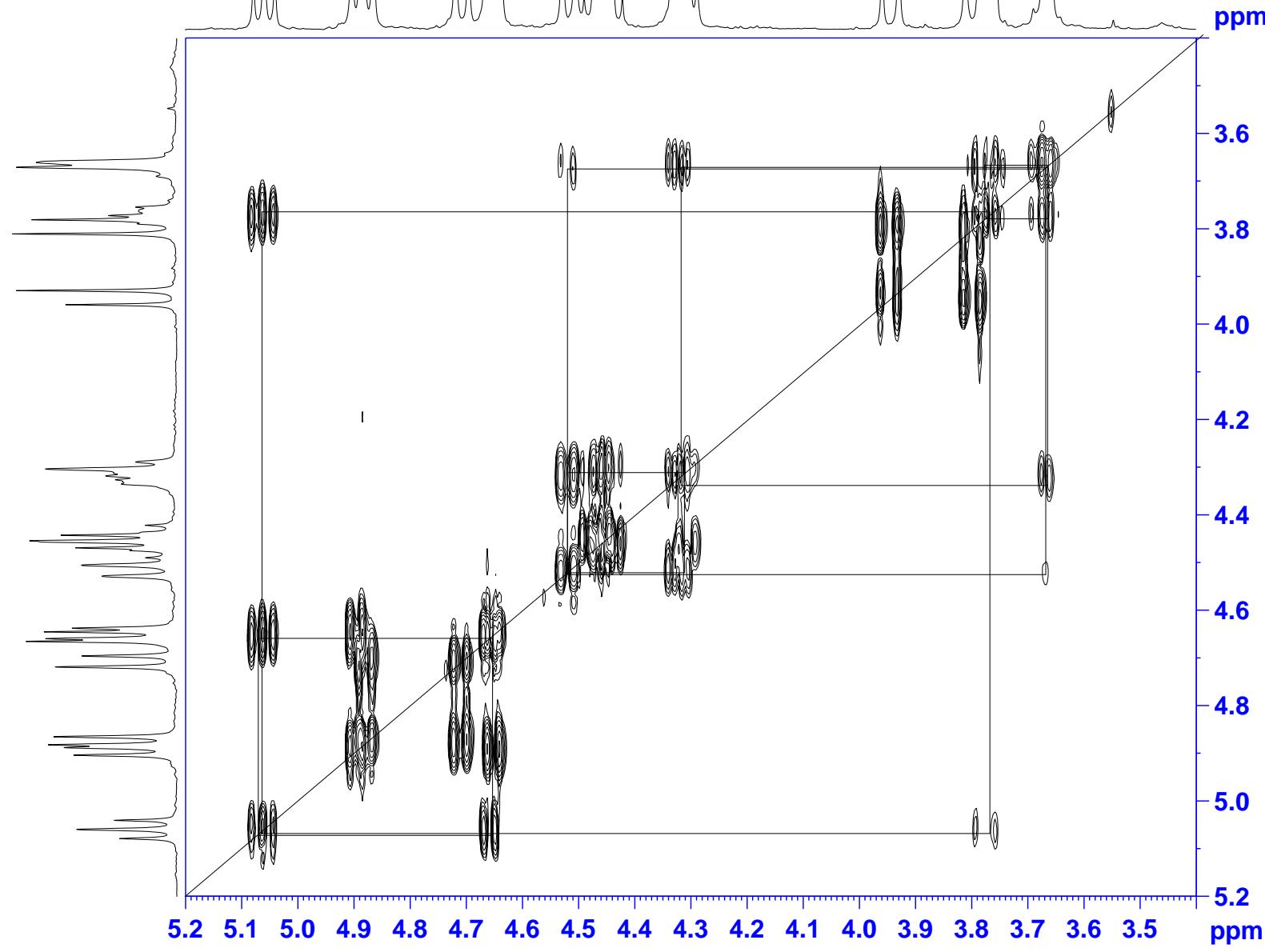
23



S72



SSK-20-BKG-6-oFMOC-cosy



S73

Current Data Parameters
NAME SSK-20-BKG-6-oFMOC-cosy
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190520
Time 11.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG cosygpppqr
TD 2048
SOLVENT CDCl3
NS 4
DS 0
SWH 1559.576 Hz
FIDRES 0.761512 Hz
AQ 0.6565888 sec
RG 61.42
DW 320.600 usec
DE 6.50 usec
TE 297.6 K
D0 0.00000300 sec
D1 1.0000000 sec
D11 0.0300000 sec
D12 0.00002000 sec
D13 0.00000400 sec
D16 0.00020000 sec
INO 0.00064120 sec

===== CHANNEL f1 ======
SF01 500.1321102 MHz
NUC1 1H
P0 13.35 usec
P1 13.35 usec
P17 5000.00 usec
PLW1 16.0000000 W
PLW10 3.16840005 W

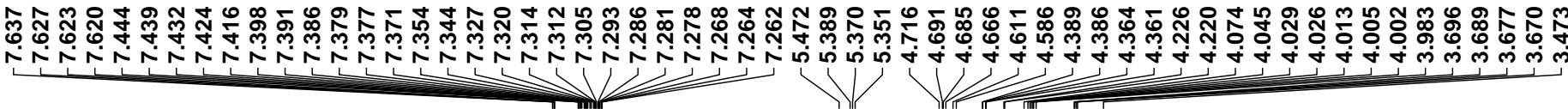
===== GRADIENT CHANNEL =====
GPNAME[1] SMSQ10.100
GPZ1 10.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 53
SF01 500.1321 MHz
FIDRES 58.851917 Hz
SW 3.118 ppm
FnMODE QF

F2 - Processing parameters
SI 1024
SF 500.1300000 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0

SSK-20-BKG-276-1H

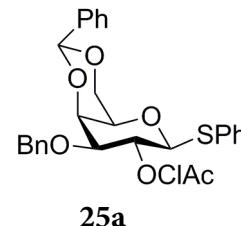


Current Data Parameters
 NAME SSK-20-BKG-276-1H
 EXPNO 1
 PROCNO 1

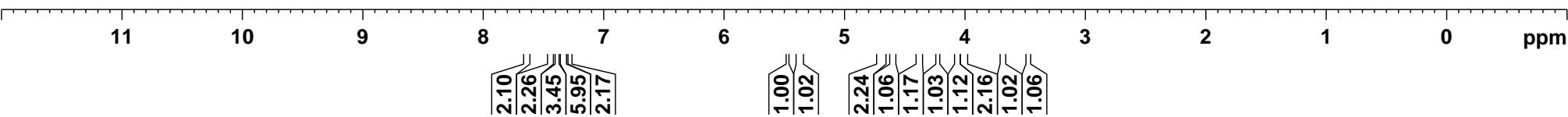
F2 - Acquisition Parameters
 Date_ 20180312
 Time 23.06
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 30
 DS 0
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2767999 sec
 RG 53.37
 DW 50.000 usec
 DE 6.50 usec
 TE 296.6 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 500.1330885 MHz
 NUC1 1H
 P1 13.35 usec
 PLW1 16.00000000 W

F2 - Processing parameters
 SI 65536
 SF 500.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



25a



SSK-20-BKG-276-13C

Current Data Parameters
 NAME SSK-20-BKG-276-13C
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date 20180312
 Time 23.08
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 104
 DS 0
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010048 sec
 RG 197.27
 DW 16.800 usec
 DE 6.50 usec
 TE 297.2 K
 D1 1.0000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 125.7703637 MHz
 NUC1 13C
 P1 8.90 usec
 PLW1 103.0000000 W

===== CHANNEL f2 =====
 SFO2 500.1320005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 16.0000000 W
 PLW12 0.44556001 W
 PLW13 0.22411001 W

F2 - Processing parameters
 SI 32768
 SF 125.7577890 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

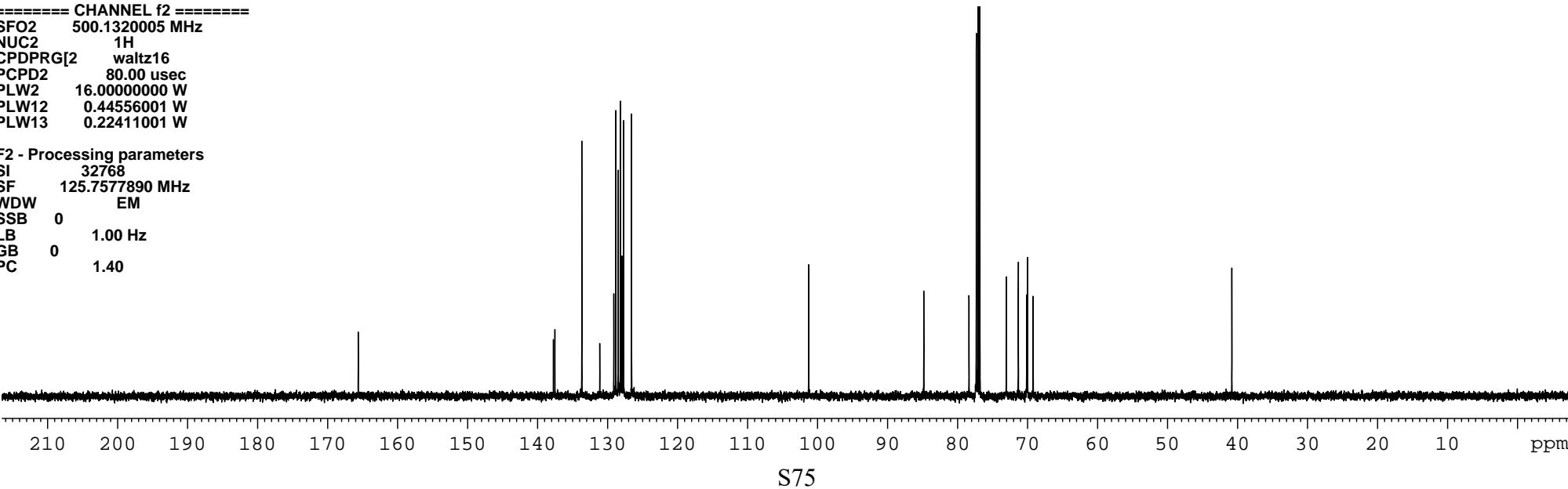
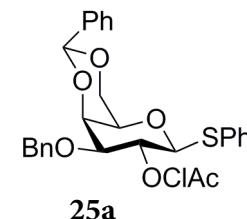
— 165.62

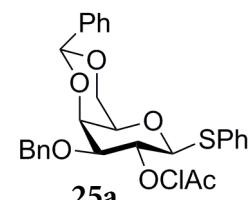
137.76
 137.54
 133.70
 131.14
 129.13
 128.84
 128.48
 128.16
 127.97
 127.74
 126.59

— 101.29

84.84
 78.38
 73.06
 71.32
 70.14
 70.03
 69.22

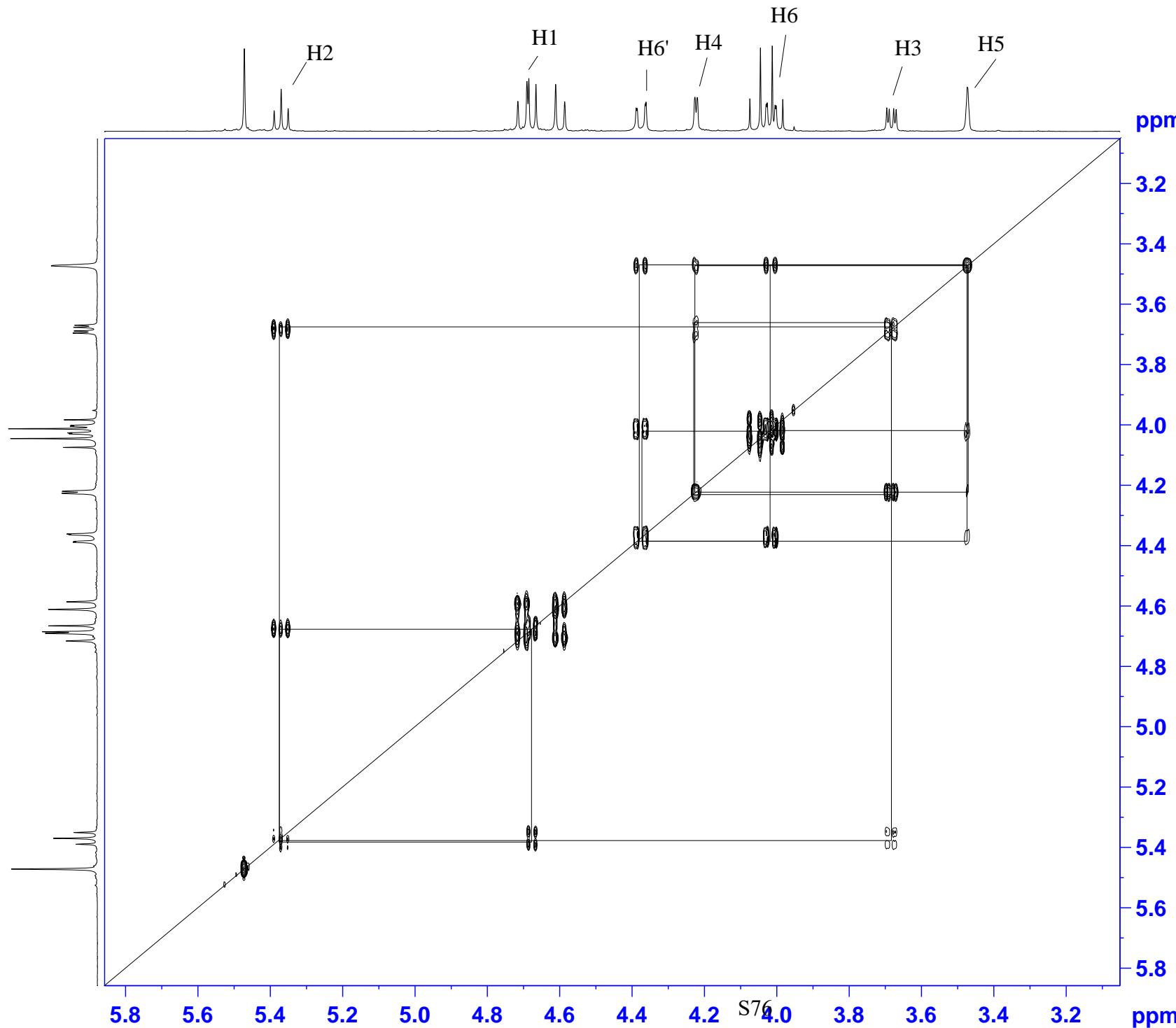
— 40.82





ppm

SSK-20-BKG-276-HHCOSY



Current Data Parameters
 NAME SSK-20-BKG-276-HHCOSY
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180312
 Time 23.11
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG cosygpppgf
 TD 2048
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 1403.706 Hz
 FIDRES 0.685403 Hz
 AQ 0.7294976 sec
 RG 134.65
 DW 356.200 usec
 DE 6.50 usec
 TE 297.1 K
 D0 0.00000300 sec
 D1 1.0000000 sec
 D11 0.03000000 sec
 D12 0.00002000 sec
 D13 0.00000400 sec
 D16 0.000020000 sec
 IN0 0.00071240 sec

===== CHANNEL f1 =====
 SP01 500.1322279 MHz
 NUC1 1H
 P0 13.35 usec
 P1 13.35 usec
 P17 2500.00 usec
 PLW1 16.0000000 W
 PLW10 3.16840005 W

===== GRADIENT CHANNEL =====
 GPNAME[1] SMSQ10.100
 GPZ1 10.00 %
 P16 1000.00 usec

F1 - Acquisition parameters
 TD 94
 SP01 500.1322 MHz
 FIDRES 29.866081 Hz
 SW 2.807 ppm
 FnMODE QF

F2 - Processing parameters
 SI 1024
 SF 500.1300000 MHz
 WDW QSINE
 SSB 0 Hz
 LB 0 Hz
 GB 0
 PC 1.40

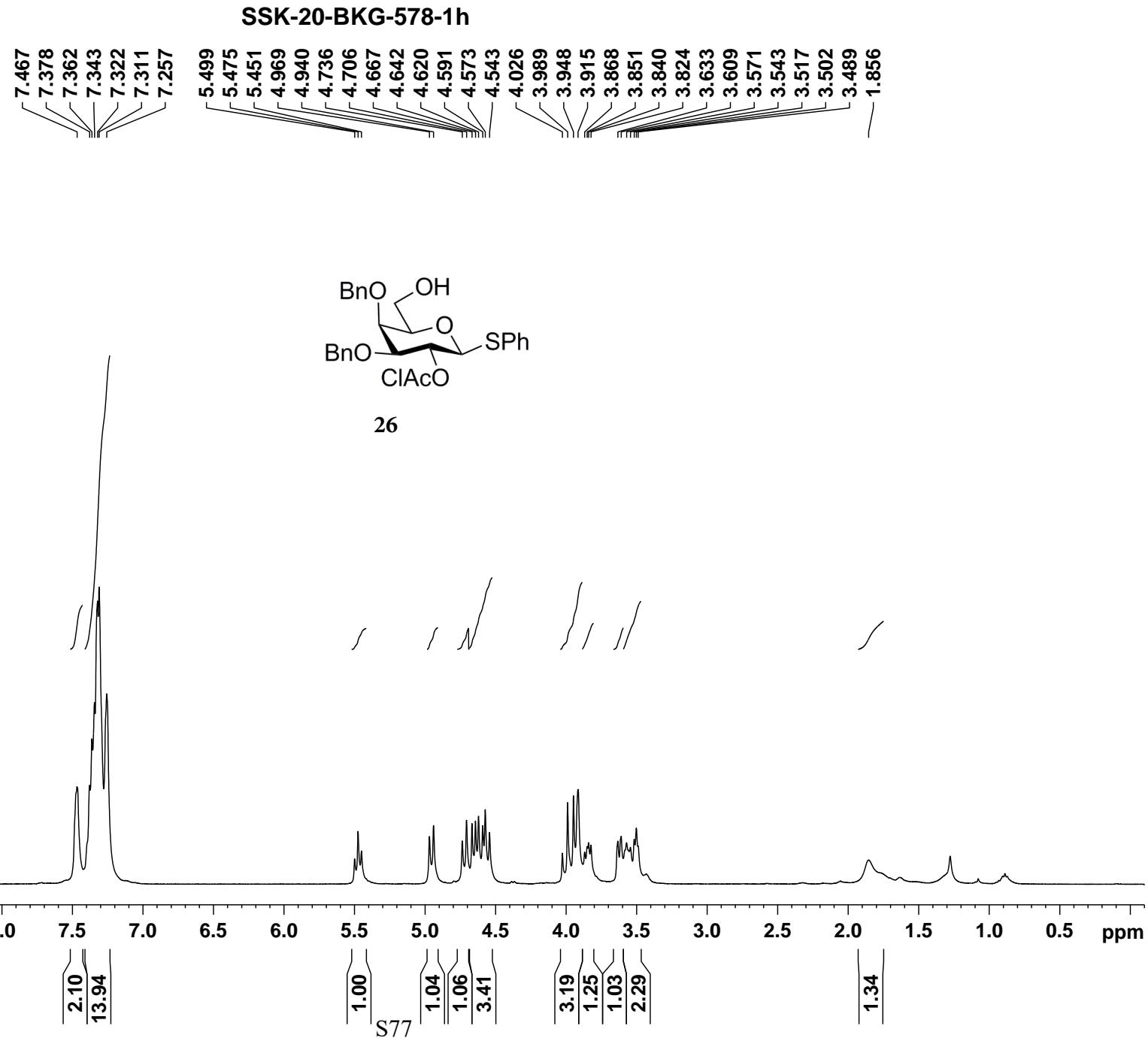
F1 - Processing parameters
 SI 1024
 MC2 QF
 SF 500.1300000 MHz
 WDW QSINE
 SSB 0 Hz
 LB 0 Hz
 GB 0

Current Data Parameters
 NAME SSK-20-BKG-578-1h
 EXPNO 6
 PROCNO 1

F2 - Acquisition Parameters
 Date 20191018
 Time 13.27
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 54274
 SOLVENT CDCl3
 NS 10
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.151522 Hz
 AQ 3.2998593 sec
 RG 64
 DW 60.800 usec
 DE 6.50 usec
 TE 296.8 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.75 usec
 PL1 -1.00 dB
 PL1W 10.56200695 W
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.13000095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



SSK-20-BKG-585-13C

Current Data Parameters
 NAME SSK-20-BKG-585-13C
 EXPNO 8
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190817
 Time 17.21
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl₃
 NS 69
 DS 0
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010048 sec
 RG 197.27
 DW 16.800 usec
 DE 6.50 usec
 TE 297.0 K
 D1 1.0000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====

SFO1 125.7703637 MHz
 NUC1 ¹³C
 P1 8.90 usec
 PLW1 103.00000000 W

===== CHANNEL f2 =====

SFO2 500.1320005 MHz
 NUC2 ¹H
 CPDPRG[2 waltz16
 PCPD2 80.00 usec
 PLW2 16.00000000 W
 PLW12 0.44556001 W
 PLW13 0.22411001 W

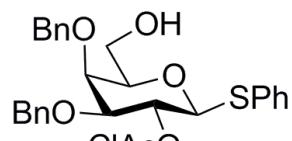
F2 - Processing parameters

SI 32768
 SF 125.7577890 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

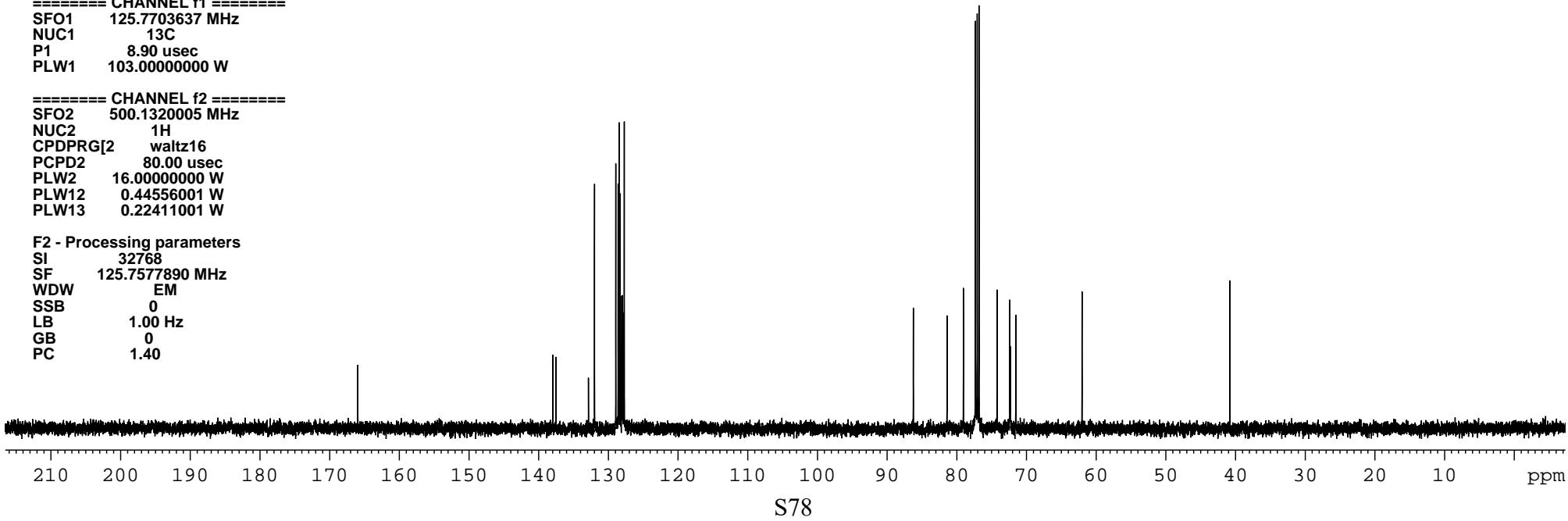
165.96

62.03

40.81



26



SSK-20-BKG-585-DEPT135

Current Data Parameters
 NAME SSK-20-BKG-585-DEPT135
 EXPNO 9
 PROCNO 1

F2 - Acquisition Parameters

Date 20190817
 Time 17.25
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG deptsp135
 TD 65536
 SOLVENT CDCl₃
 NS 35
 DS 0
 SWH 20161.291 Hz
 FIDRES 0.307637 Hz
 AQ 1.6252928 sec
 RG 197.27
 DW 24.800 usec
 DE 6.50 usec
 TE 297.4 K
 CNST2 145.0000000
 D1 1.00000000 sec
 D2 0.00344828 sec
 D12 0.00002000 sec
 TD0 1

===== CHANNEL f1 =====

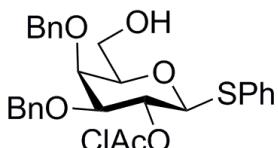
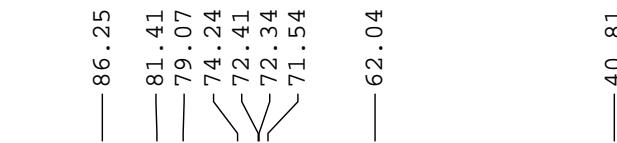
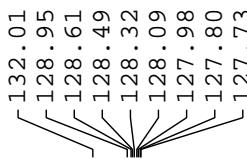
SFO1 125.7678486 MHz
 NUC1 ¹³C
 P1 8.90 usec
 P13 2000.00 usec
 PLW0 0 W
 PLW1 103.00000000 W
 SPNAM[5] Crp60comp.4
 SPOAL5 0.00000000
 SPOFFS5 0 Hz
 SPW5 12.46500015 W

===== CHANNEL f2 =====

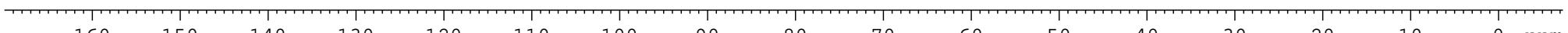
SFO2 500.1315995 MHz
 NUC2 ¹H
 CPDPRG[2] waltz16
 P3 13.35 usec
 P4 26.70 usec
 PCPD2 80.00 usec
 PLW2 16.00000000 W
 PLW12 0.44556001 W

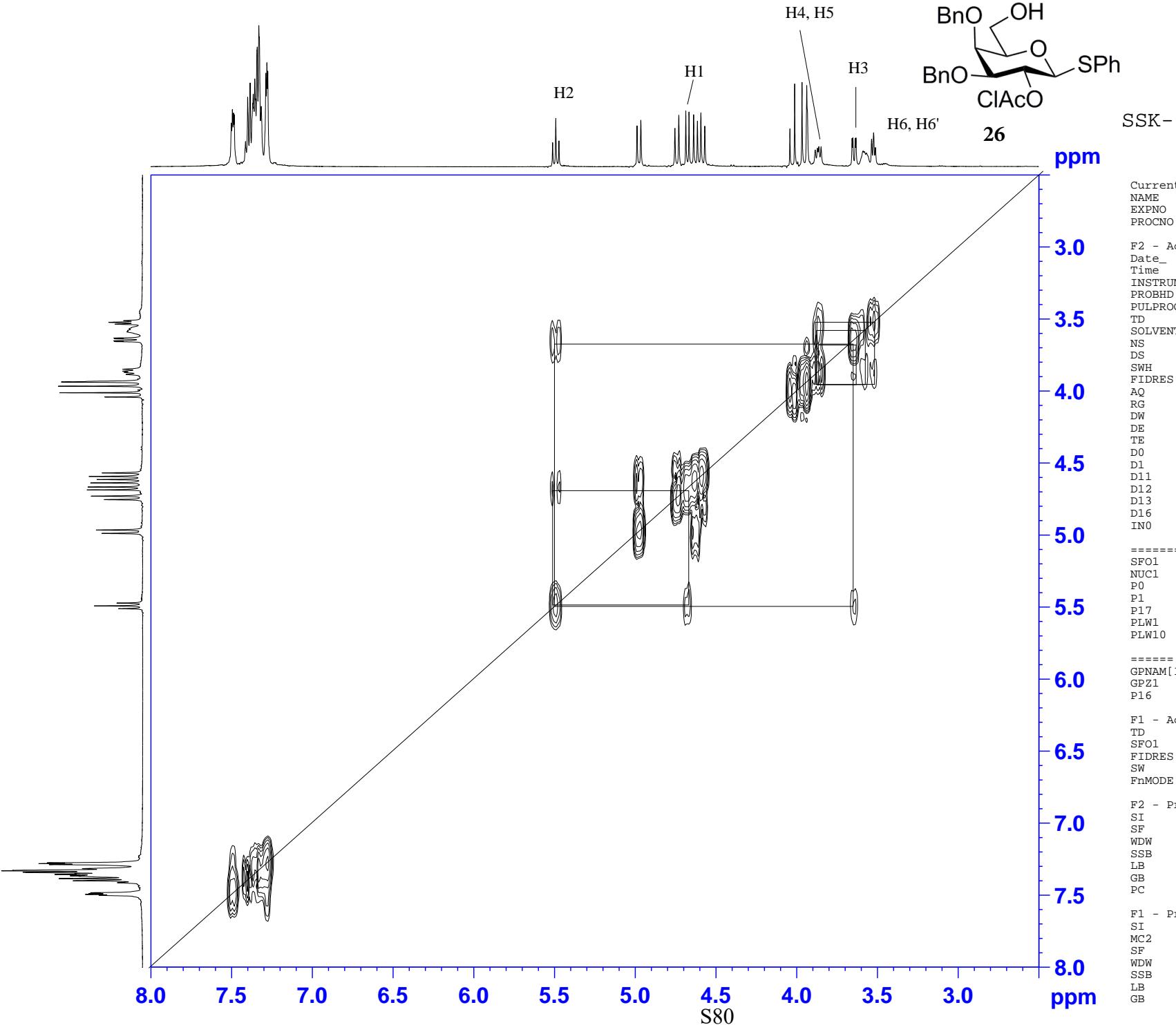
F2 - Processing parameters

SI 32768
 SF 125.7577890 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



26





SSK-20-BKG-585-COSY

Current Data Parameters
 NAME SSK-20-BKG-585-COSY
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190817
 Time 17.25
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG cosygpppqf
 TD 2048
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 5933.544 Hz
 FIDRES 2.897238 Hz
 AQ 0.1725781 sec
 RG 61.42
 DW 84.267 usec
 DE 6.50 usec
 TE 297.4 K
 D0 0.00000300 sec
 D1 1.0000000 sec
 D11 0.0300000 sec
 D12 0.00002000 sec
 D13 0.00000400 sec
 D16 0.0002000 sec
 IN0 0.00016860 sec

===== CHANNEL f1 =====
 SFO1 500.1326904 MHz
 NUC1 1H
 P0 13.35 usec
 P1 13.35 usec
 P17 5000.00 usec
 PLW1 16.00000000 W
 PLW10 3.16840005 W

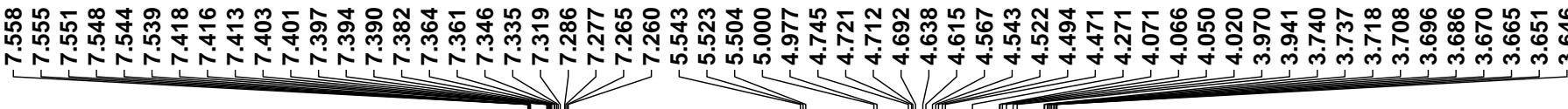
===== GRADIENT CHANNEL =====
 GPNAM[1] SMSQ10.100
 GPZ1 10.00 %
 P16 1000.00 usec

F1 - Acquisition parameters
 TD 58
 SFO1 500.1327 MHz
 FIDRES 204.524078 Hz
 SW 11.859 ppm
 FnMODE QF

F2 - Processing parameters
 SI 1024
 SF 500.1300000 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
 SI 1024
 MC2 QF
 SF 500.1300000 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0

SSK-20-BKG-251-1H

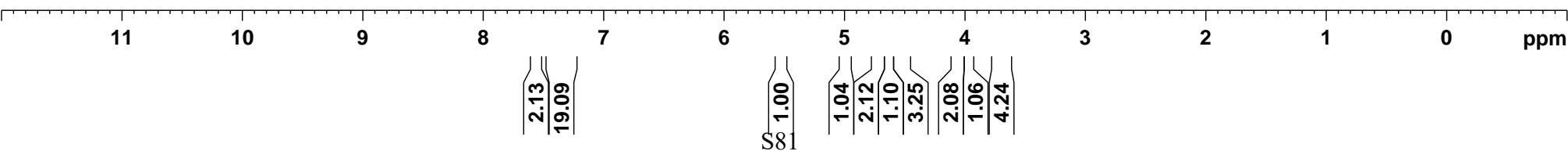
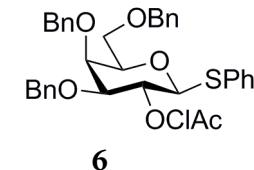


Current Data Parameters
 NAME SSK-20-BKG-251-1H
 EXPNO 1
 PROCNO 1

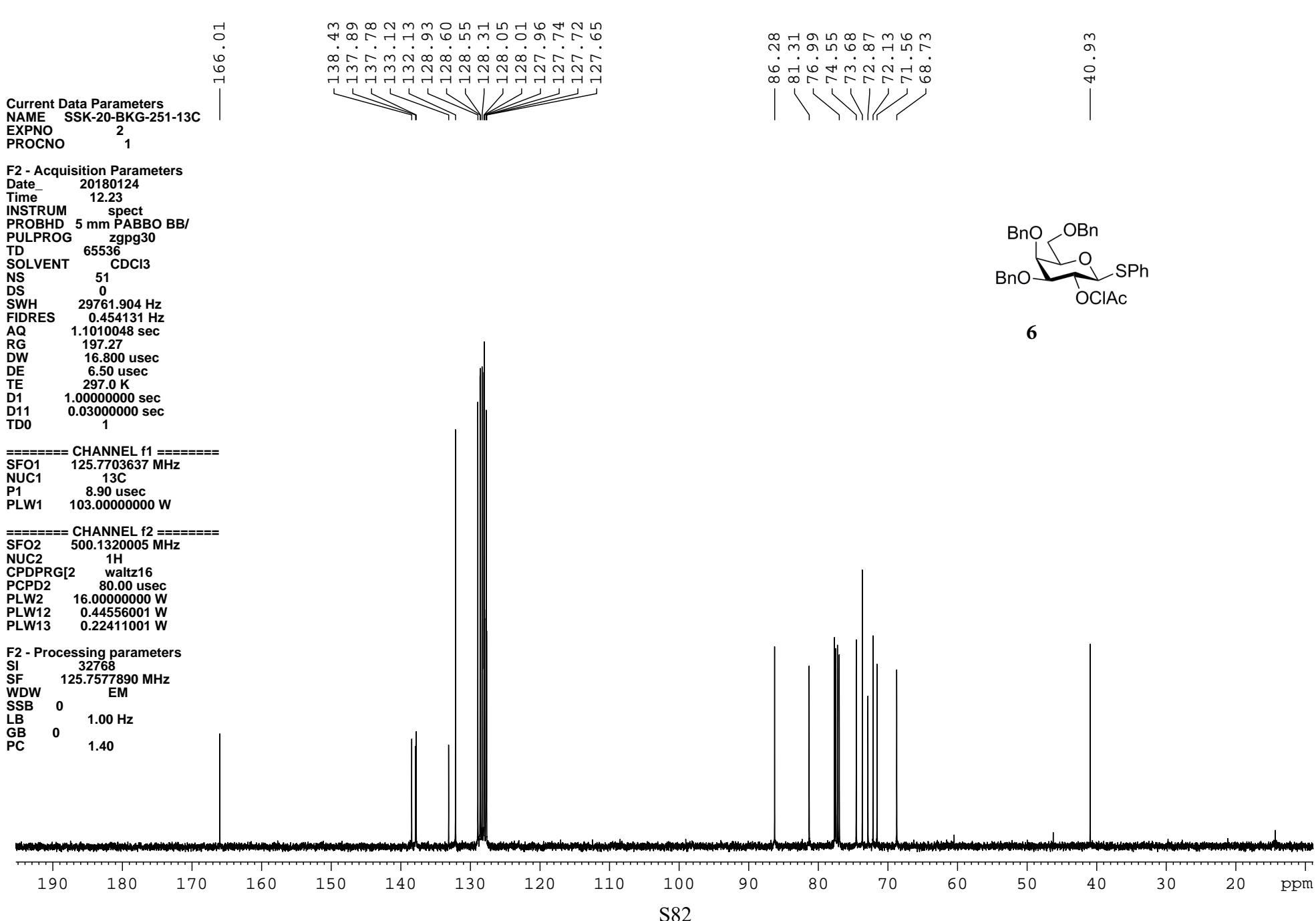
F2 - Acquisition Parameters
 Date 20180124
 Time 12.20
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 16
 DS 0
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2767999 sec
 RG 12.52
 DW 50.000 usec
 DE 6.50 usec
 TE 296.4 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 500.1330885 MHz
 NUC1 1H
 P1 13.35 usec
 PLW1 16.00000000 W

F2 - Processing parameters
 SI 65536
 SF 500.1300132 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



SSK-20-BKG-251-13C



SSK-20-BKG-251-DEPT

Current Data Parameters
 NAME SSK-20-BKG-251-DEPT135
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters

Date 20180124
 Time 12.25
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG deptsp135
 TD 65536
 SOLVENT CDCl₃
 NS 37
 DS 0
 SWH 20161.291 Hz
 FIDRES 0.307637 Hz
 AQ 1.6252928 sec
 RG 197.27
 DW 24.800 usec
 DE 6.50 usec
 TE 297.0 K
 CNST2 145.000000
 D1 1.0000000 sec
 D2 0.00344828 sec
 D12 0.00002000 sec
 TD0 1

===== CHANNEL f1 =====

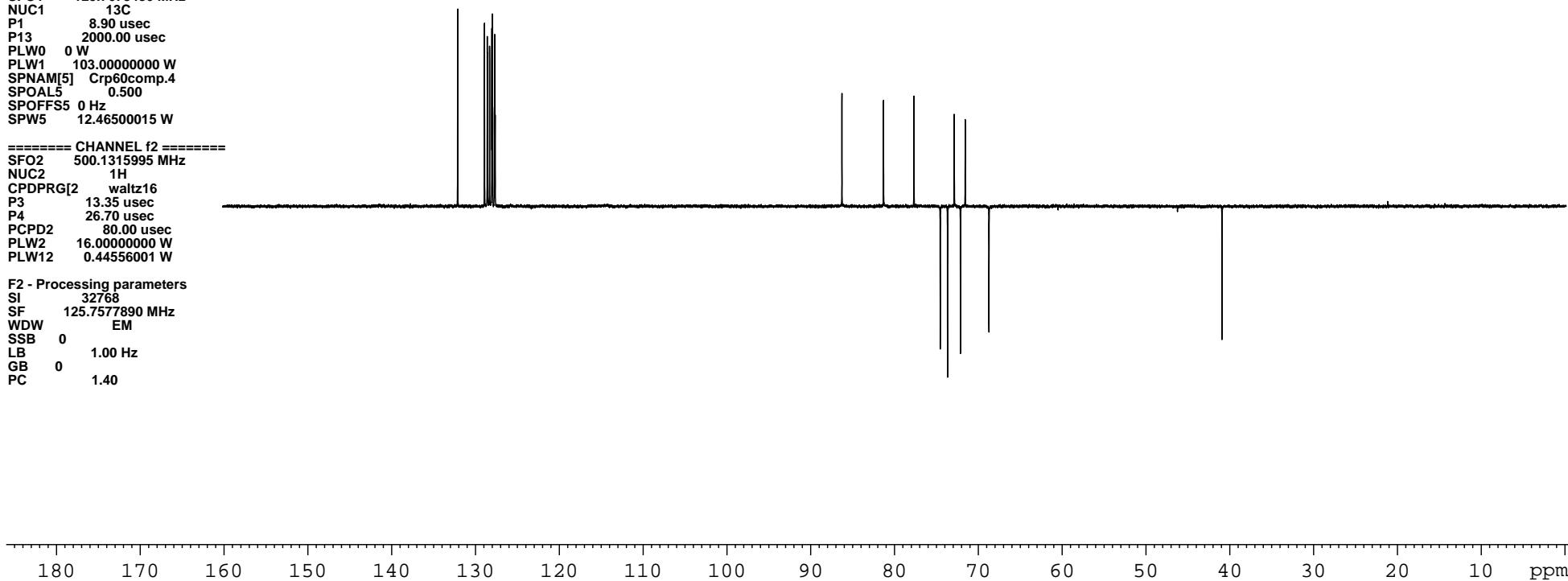
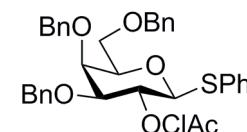
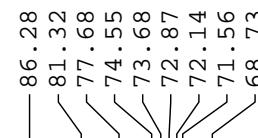
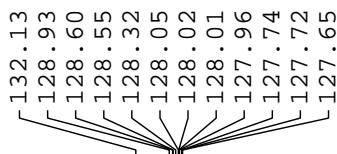
SFO1 125.7678486 MHz
 NUC1 13C
 P1 8.90 usec
 P13 2000.00 usec
 PLW0 0 W
 PLW1 103.00000000 W
 SPNAM[5] Crp60comp.4
 SPOAL5 0.500
 SPOFFS5 0 Hz
 SPW5 12.46500015 W

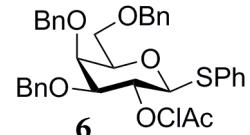
===== CHANNEL f2 =====

SFO2 500.1315995 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 P3 13.35 usec
 P4 26.70 usec
 PCPD2 80.00 usec
 PLW2 16.00000000 W
 PLW12 0.44556001 W

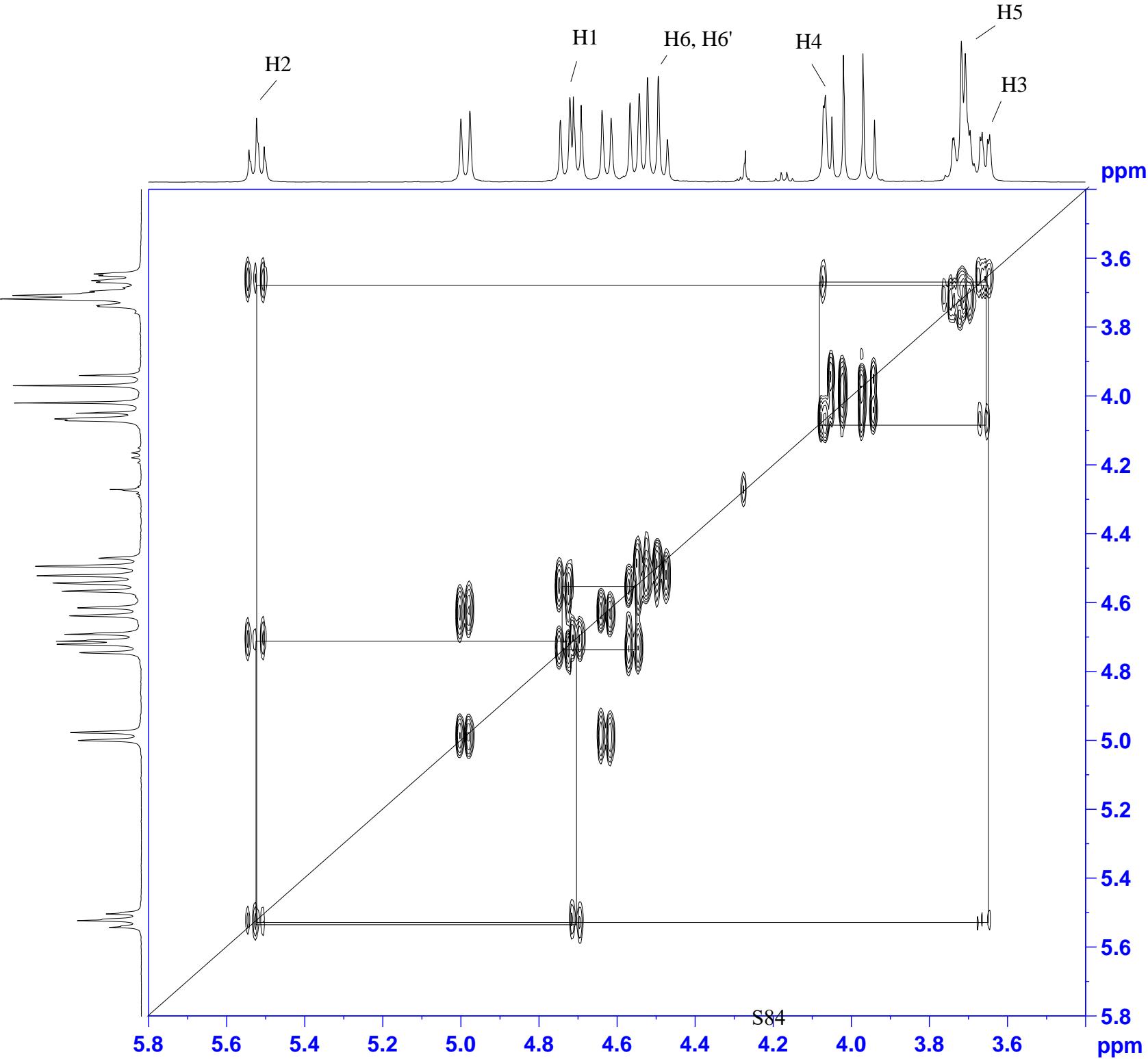
F2 - Processing parameters

SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40





SSK-20-BKG-251-HHCOSY



Current Data Parameters

NAME SSK-20-BKG-251-HHCOSY
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters

Date_ 20180124
Time 12.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG cosygppqf
TD 2048
SOLVENT CDCl3
NS 4
DS 8
SWH 2032.520 Hz
FIDRES 0.992442 Hz
AQ 0.5038080 sec
RG 38.51
DW 246.000 usec
DE 6.50 usec
TE 296.7 K
D0 0.00000300 sec
D1 1.0000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D13 0.00000400 sec
D16 0.00002000 sec
INO 0.00049200 sec

===== CHANNEL f1 =====

SFO1 500.1318905 MHz
NUC1 1H
P0 13.35 usec
P1 13.35 usec
P17 2500.00 usec
PLW1 16.0000000 W
PLW10 3.16840005 W

===== GRADIENT CHANNEL =====

GPNAM[1] SMSQ10.100

GPZ1 10.00 %

P16 1000.00 usec

F1 - Acquisition parameters

TD 54

SFO1 500.1319 MHz

FIDRES 75.278534 Hz

SW 4.064 ppm

FnMODE QF

F2 - Processing parameters

SI 1024

SF 500.1300132 MHz

WDW QSINE

SSB 0

LB 0 Hz

GB 0

PC 1.40

F1 - Processing parameters

SI 1024

MC2 QF

SF 500.1300132 MHz

WDW QSINE

SSB 0

LB 0 Hz

GB 0

S84

5.8
ppm

5.6
ppm

5.4
ppm

5.2
ppm

5.0
ppm

4.8
ppm

4.6
ppm

4.4
ppm

4.2
ppm

4.0
ppm

3.8
ppm

3.6
ppm

SSK-20-BKG-566LEV-1H



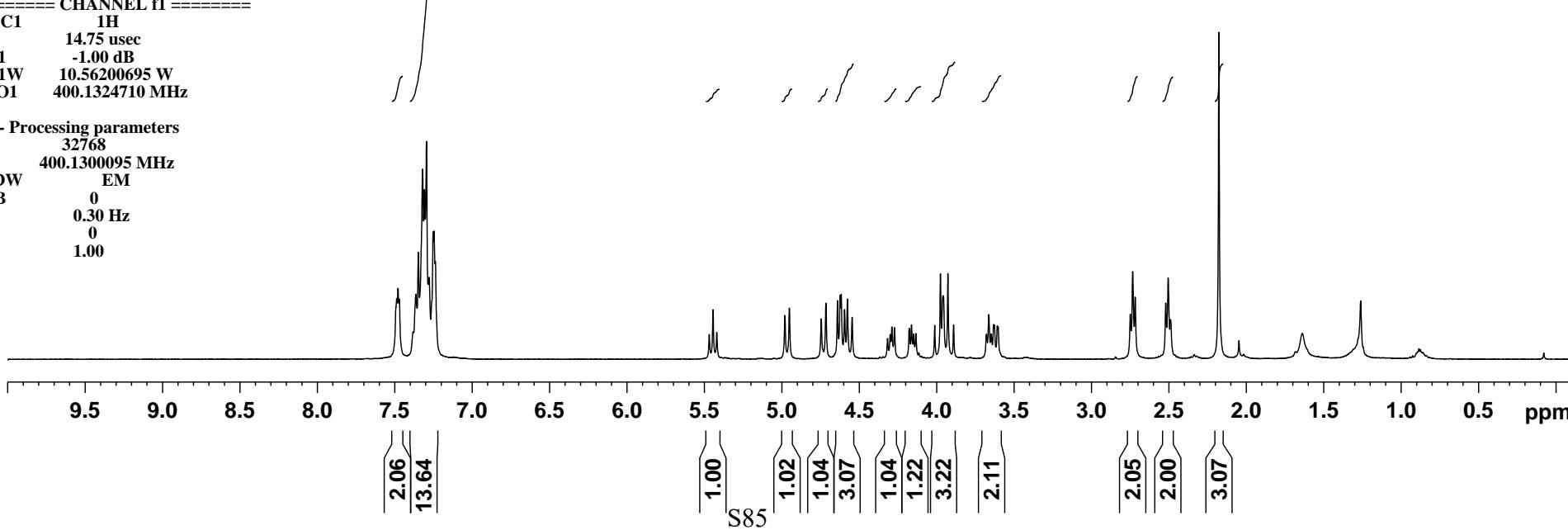
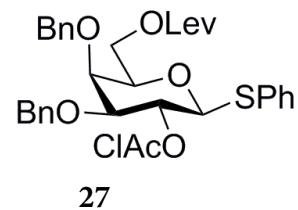
Current Data Parameters
 NAME SSK-20-BKG-566LEV-1H
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20191018
 Time 13.24
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 54274
 SOLVENT CDCl₃
 NS 8
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.151522 Hz
 AQ 3.2998593 sec
 RG 90.5
 DW 60.800 usec
 DE 6.50 usec
 TE 296.8 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.75 usec
 PL1 -1.00 dB
 PL1W 10.56200695 W
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



SSK-21-BKG-566LEV-13C

— 206.56

— 172.45

— 165.90

138.01
137.55
132.91
132.12
128.82
128.56
128.32
128.15
128.04
127.76

86.12
81.18
76.07
74.34
72.35
72.24
71.32
63.16

40.80
37.93
29.83
27.82

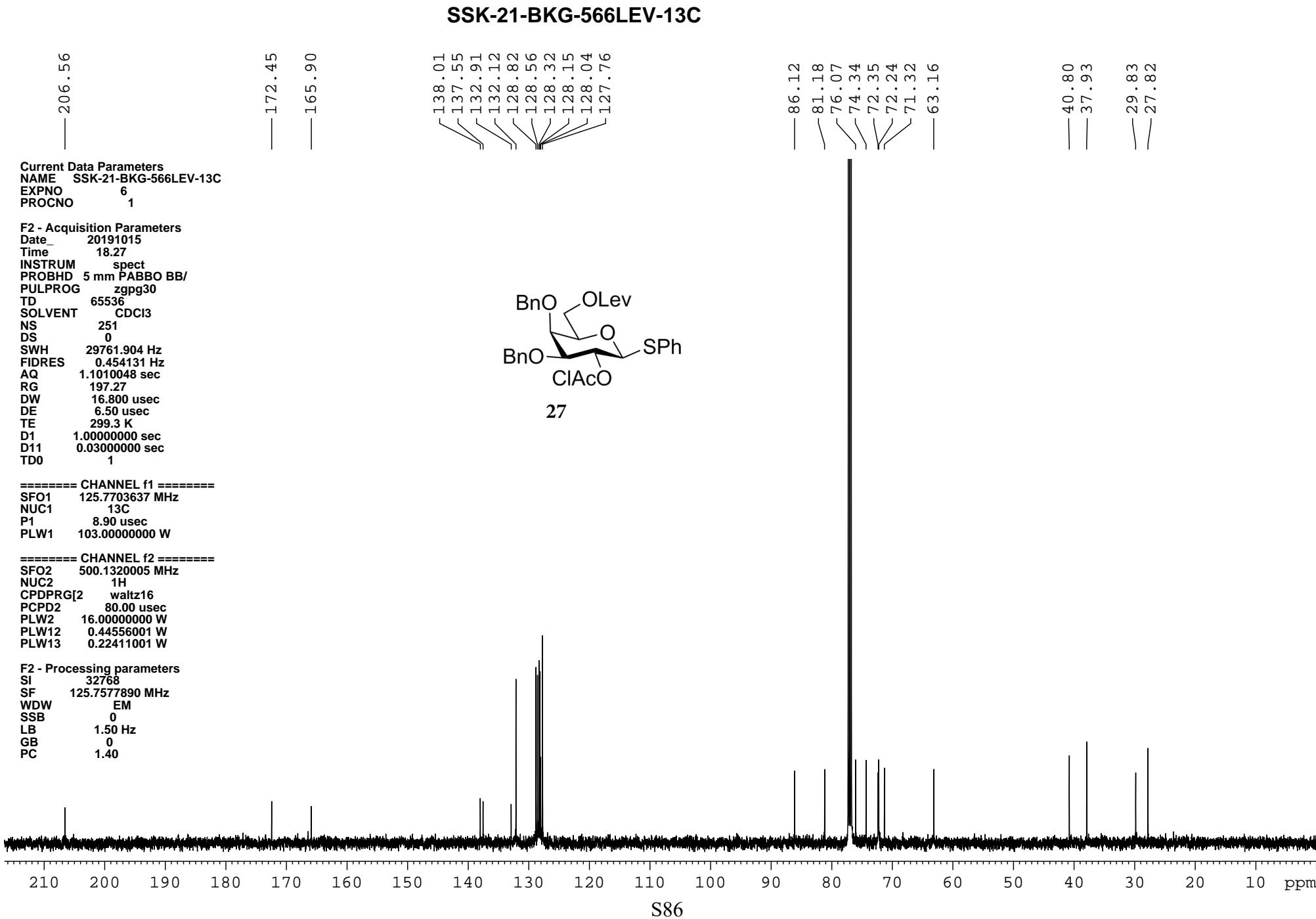
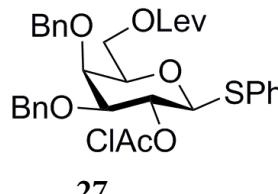
Current Data Parameters
NAME SSK-21-BKG-566LEV-13C
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date 20191015
Time 18.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 251
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 197.27
DW 16.800 usec
DE 6.50 usec
TE 299.3 K
D1 1.0000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 125.7703637 MHz
NUC1 13C
P1 8.90 usec
PLW1 103.0000000 W

===== CHANNEL f2 =====
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 16.00000000 W
PLW12 0.44556001 W
PLW13 0.22411001 W

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40



SSK-21-BKG-566LEV-DEPT

Current Data Parameters
 NAME SSK-21-BKG-566LEV-DEPT
 EXPNO 7
 PROCNO 1

F2 - Acquisition Parameters
 Date 20191015
 Time 18.37
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG deptsp135
 TD 65536
 SOLVENT CDCl₃
 NS 115
 DS 0
 SWH 20161.291 Hz
 FIDRES 0.307637 Hz
 AQ 1.6252928 sec
 RG 197.27
 DW 24.800 usec
 DE 6.50 usec
 TE 299.3 K
 CNST2 145.0000000
 D1 1.00000000 sec
 D2 0.00344828 sec
 D12 0.000002000 sec
 TD0 1

===== CHANNEL f1 ======
 SFO1 125.7678486 MHz
 NUC1 ¹³C
 P1 8.90 usec
 P13 2000.00 usec
 PLW0 0 W
 PLW1 103.00000000 W
 SPNAM[5] Crp60comp.4
 SPOALS 0.500
 SPOFFS5 0 Hz
 SPW5 12.46500015 W

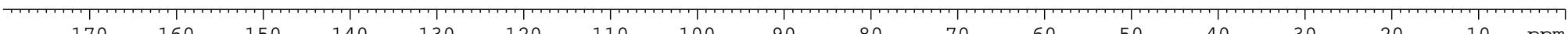
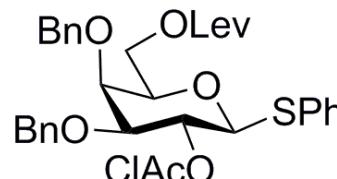
===== CHANNEL f2 ======
 SFO2 500.1315995 MHz
 NUC2 ¹H
 CPDPRG[2] waltz16
 P3 13.35 usec
 P4 26.70 usec
 PCPD2 80.00 usec
 PLW2 16.00000000 W
 PLW12 0.44556001 W

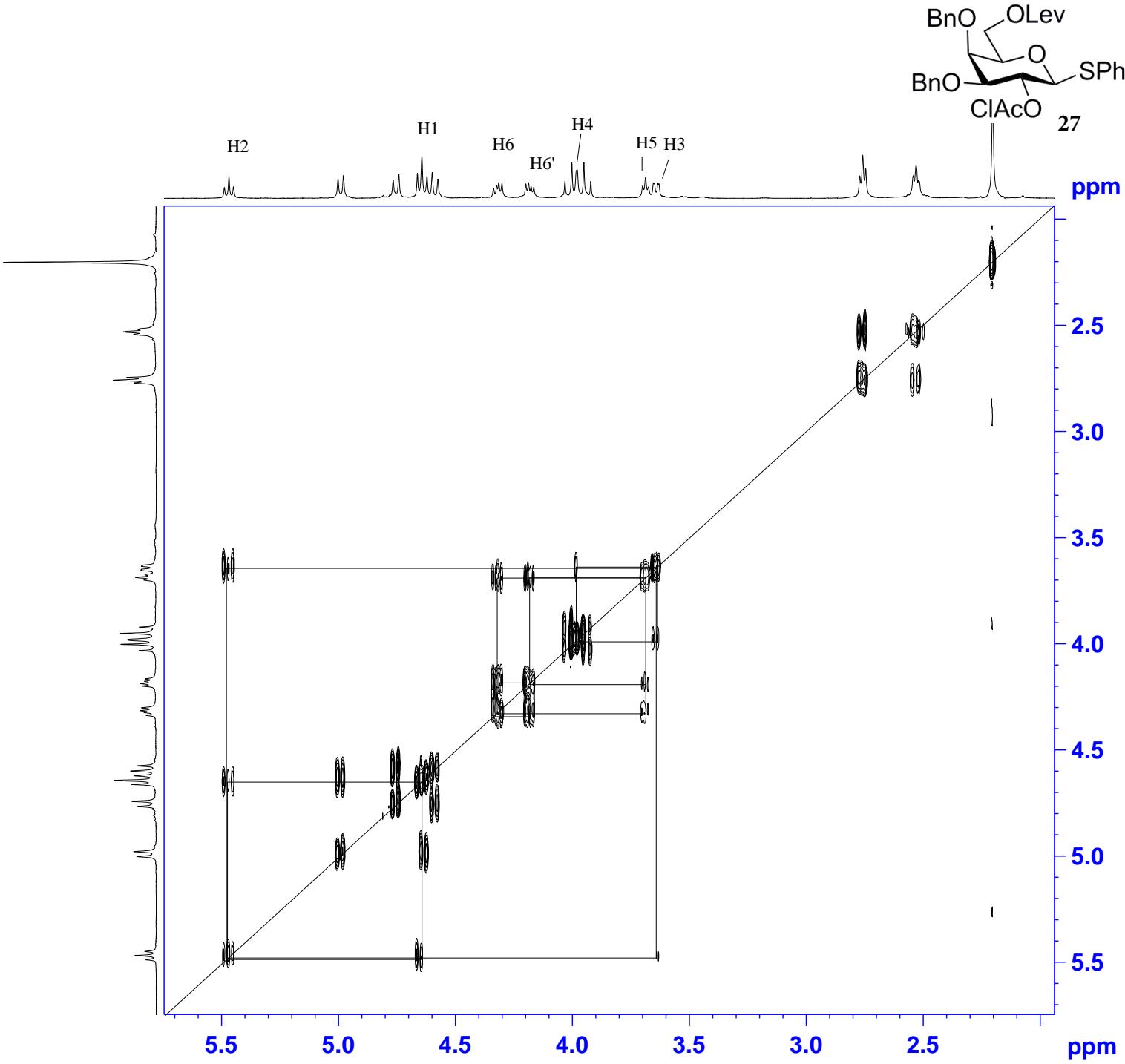
F2 - Processing parameters
 SI 32768
 SF 125.7577890 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

132.12
 128.82
 128.56
 128.32
 128.15
 128.04
 127.76
 127.72

86.12
 81.18
 76.07
 74.34
 72.35
 72.24
 71.32
 63.16

40.80
 37.93
 29.84
 27.82





SSK-21-BKG-566LEV-COSY

Current Data Parameters
 NAME SSK-21-BKG-566LEV-COSY
 EXPNO 7
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20191015
 Time 18.41
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG cosygpppqf
 TD 2048
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 1904.036 Hz
 FIDRES 0.929705 Hz
 AQ 0.5378048 sec
 RG 61.42
 DW 262.600 usec
 DE 6.50 usec
 TE 299.2 K
 D0 0.00000300 sec
 D1 1.00000000 sec
 D11 0.03000000 sec
 D12 0.00002000 sec
 D13 0.00000400 sec
 D16 0.00020000 sec
 IN0 0.00052520 sec

===== CHANNEL f1 =====
 SFO1 500.1319215 MHz
 NUC1 1H
 P0 13.35 usec
 P1 13.35 usec
 P17 5000.00 usec
 PLW1 16.00000000 W
 PLW10 3.16840005 W

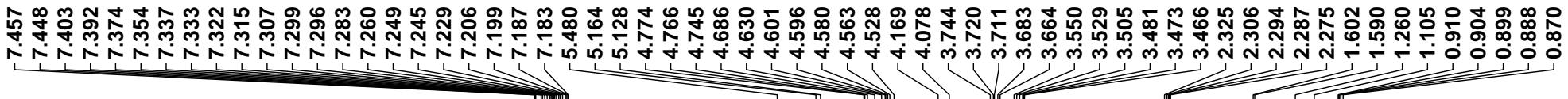
===== GRADIENT CHANNEL =====
 GPNAME[1] SMSQ10.100
 GPZ1 10.00 %
 P16 1000.00 usec

F1 - Acquisition parameters
 TD 42
 SFO1 500.1319 MHz
 FIDRES 90.668411 Hz
 SW 3.807 ppm
 FmMODE QF

F2 - Processing parameters
 SI 1024
 SF 500.1300000 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
 SI 1024
 MC2 QF
 SF 500.1300000 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0

SSK-20-BKG-268(II)-1H

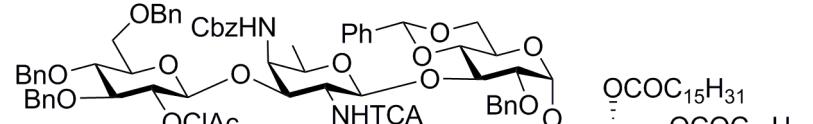


Current Data Parameters
NAME SSK-20-BKG-268(II)-1H
EXPNO 1
PROCNO 1

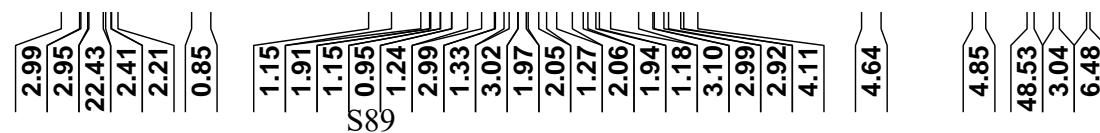
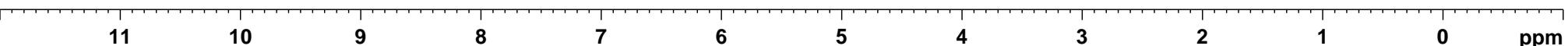
F2 - Acquisition Parameters
Date_ 20180223
Time 3.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 54274
SOLVENT CDCl3
NS 46
DS 0
SWH 8223.685 Hz
FIDRES 0.151522 Hz
AQ 3.2998593 sec
RG 57
DW 60.800 usec
DE 6.50 usec
TE 296.8 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 -1.00 dB
PL1W 10.56200695 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



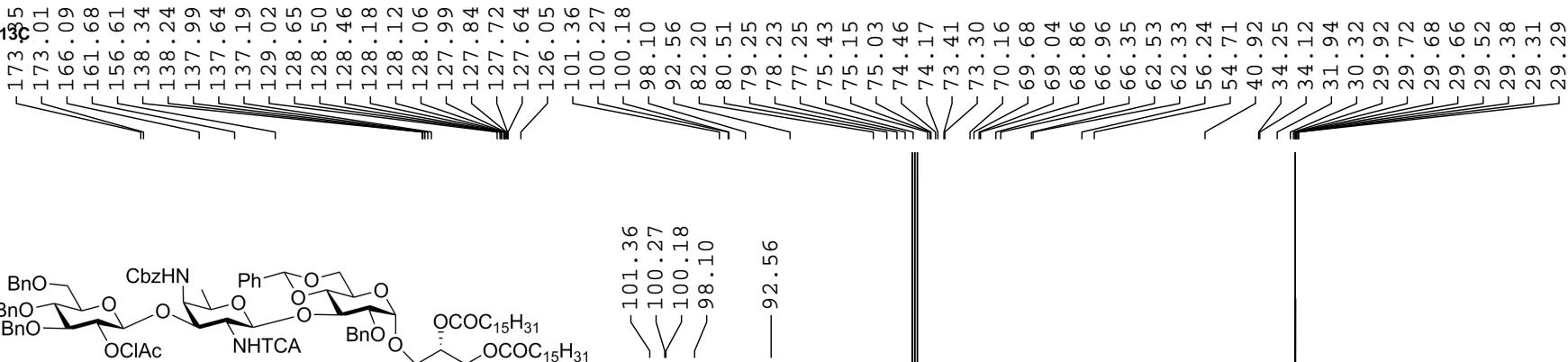
3



SSK-20-BKG-268(II)-13C

Current Data Parameters

NAME SSK-20-BKG-268(II)-13C
 EXPNO 12
 PROCNO 1
 F2 - Acquisition Parameters
 Date 20180223
 Time 3.43
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zpgpg30
 TD 65536
 SOLVENT CDCl3
 NS 646
 DS 0
 SWH 26041.666 Hz
 FIDRES 0.397364 Hz
 AQ 1.2582912 sec
 RG 1030
 DW 19.200 usec
 DE 6.50 usec
 TE 297.8 K
 D1 1.0000000 sec
 D11 0.03000000 sec
 TDO 1


===== CHANNEL f1 =====

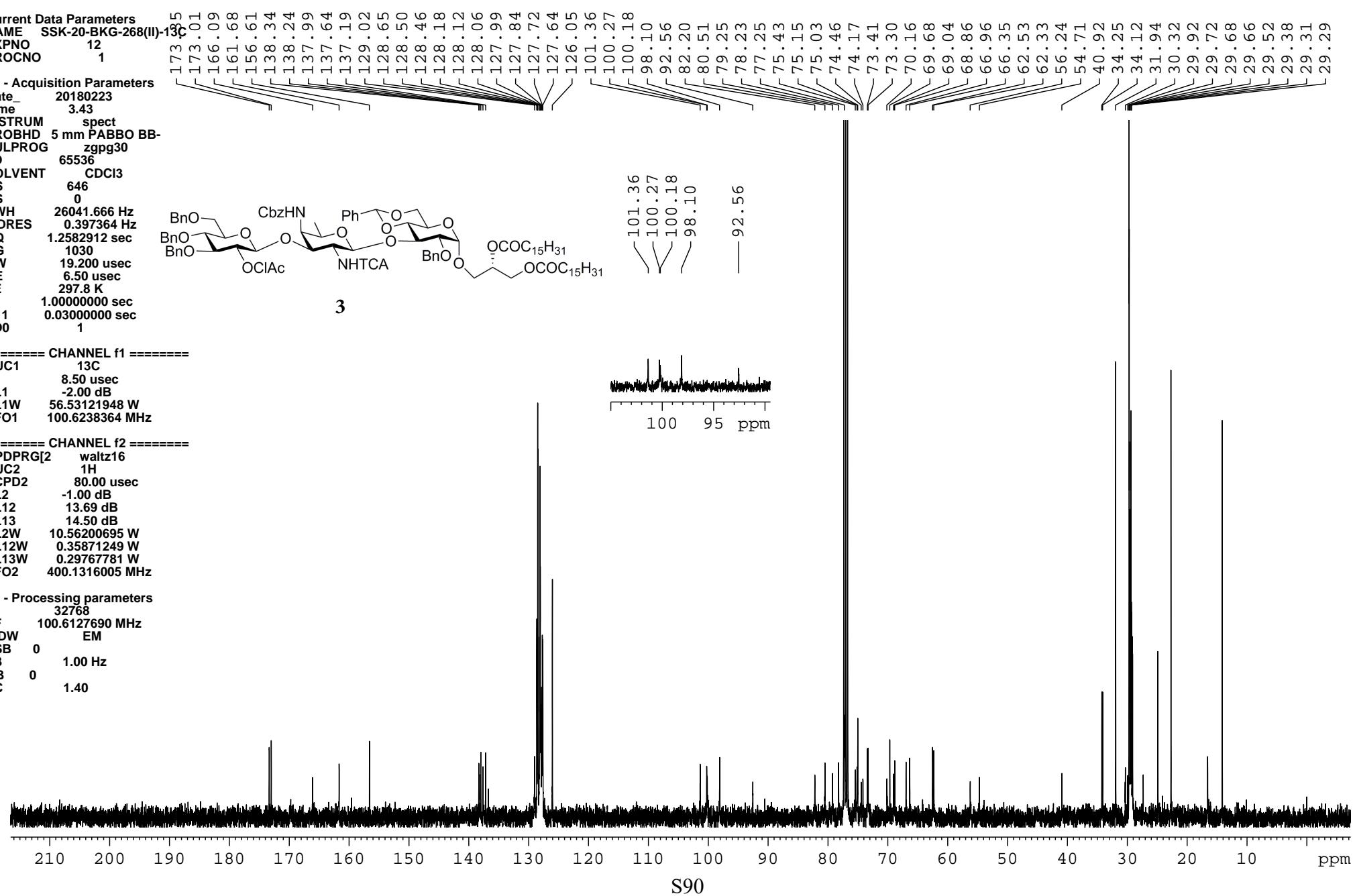
NUC1 13C
 P1 8.50 usec
 PL1 -2.00 dB
 PL1W 56.53121948 W
 SFO1 100.6238364 MHz

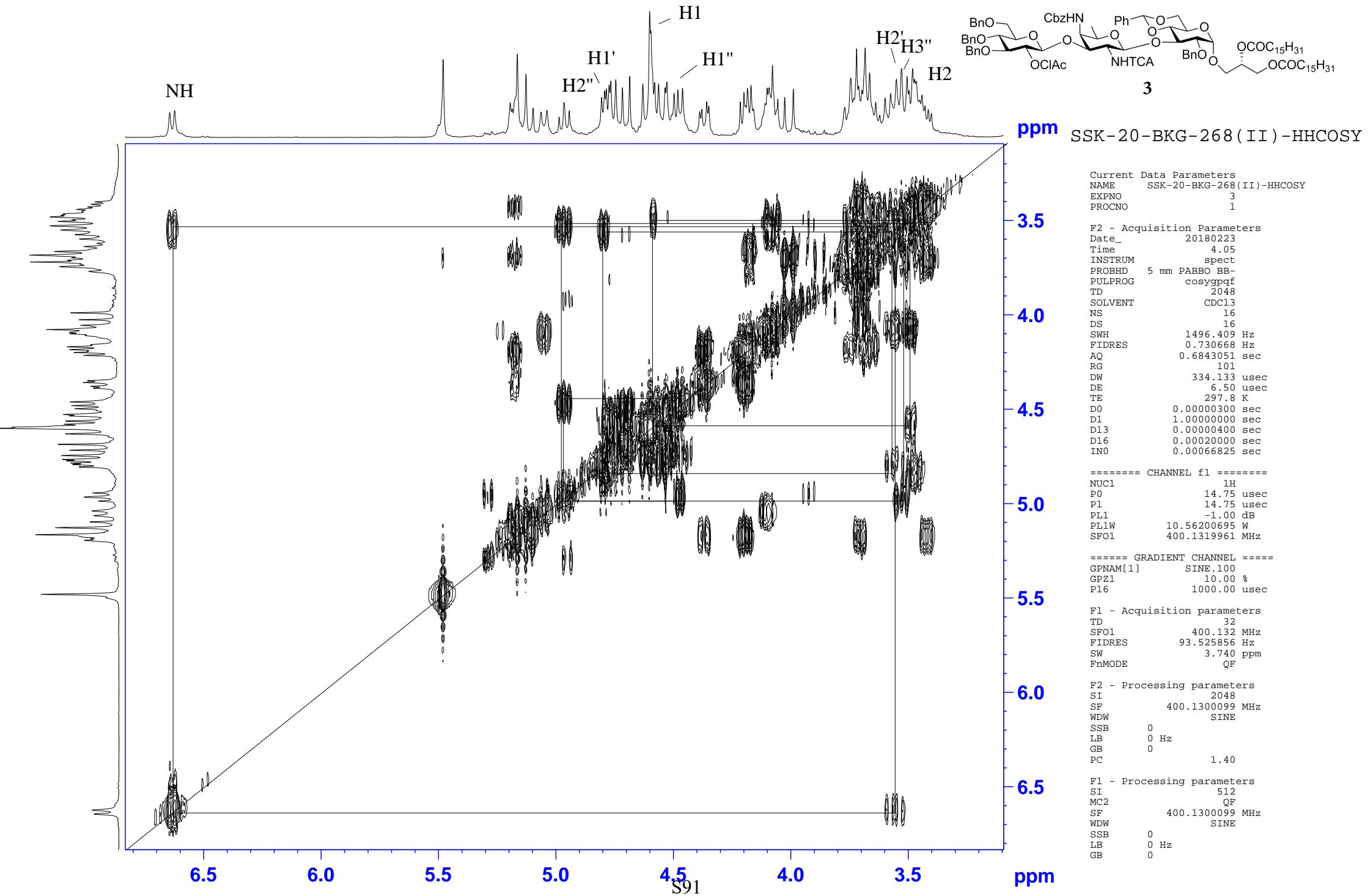
===== CHANNEL f2 =====

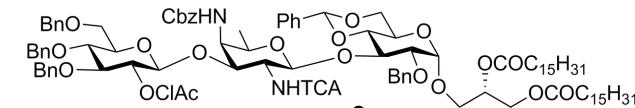
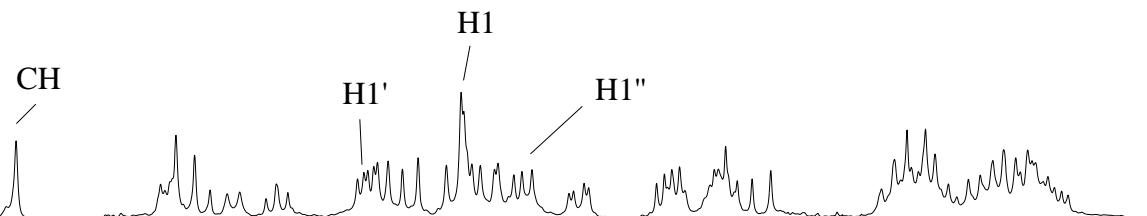
CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 13.69 dB
 PL13 14.50 dB
 PL2W 10.56200695 W
 PL12W 0.35871249 W
 PL13W 0.29767781 W
 SFO2 400.1316005 MHz

F2 - Processing parameters

SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40







SSK-20-BKG-268 (II)-HSQC

ppm

Current Data Parameters
NAME SSK-20-BKG-268(II)-HSQC
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180223
Time 4.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG hsqcetgp
TD 2048
SOLVENT CDCl3
NS 16
DS 16
SWH 1496.409 Hz
FIDRES 0.730668 Hz
AQ 0.6843051 sec
RG 2050
DW 334.133 usec
DE 6.50 usec
TE 297.50 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.0000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
INO 0.00002070 sec
ZGOPTNS

===== CHANNEL f1 ======
NUC1 1H
P1 14.75 usec
P2 29.50 usec
P28 0.10 usec
PLL -1.00 dB
PLLW 10.56200695 W
SF01 400.1319961 MHz

===== CHANNEL f2 ======
CPDPRG[2] garp
NUC2 13C
P3 8.50 usec
P4 17.00 usec
PCPD2 70.00 usec
PL2 -2.00 dB
PLL2 16.31 dB
PLL2W 56.53121948 W
PLL2W 0.83423501 W
SF02 100.6248425 MHz

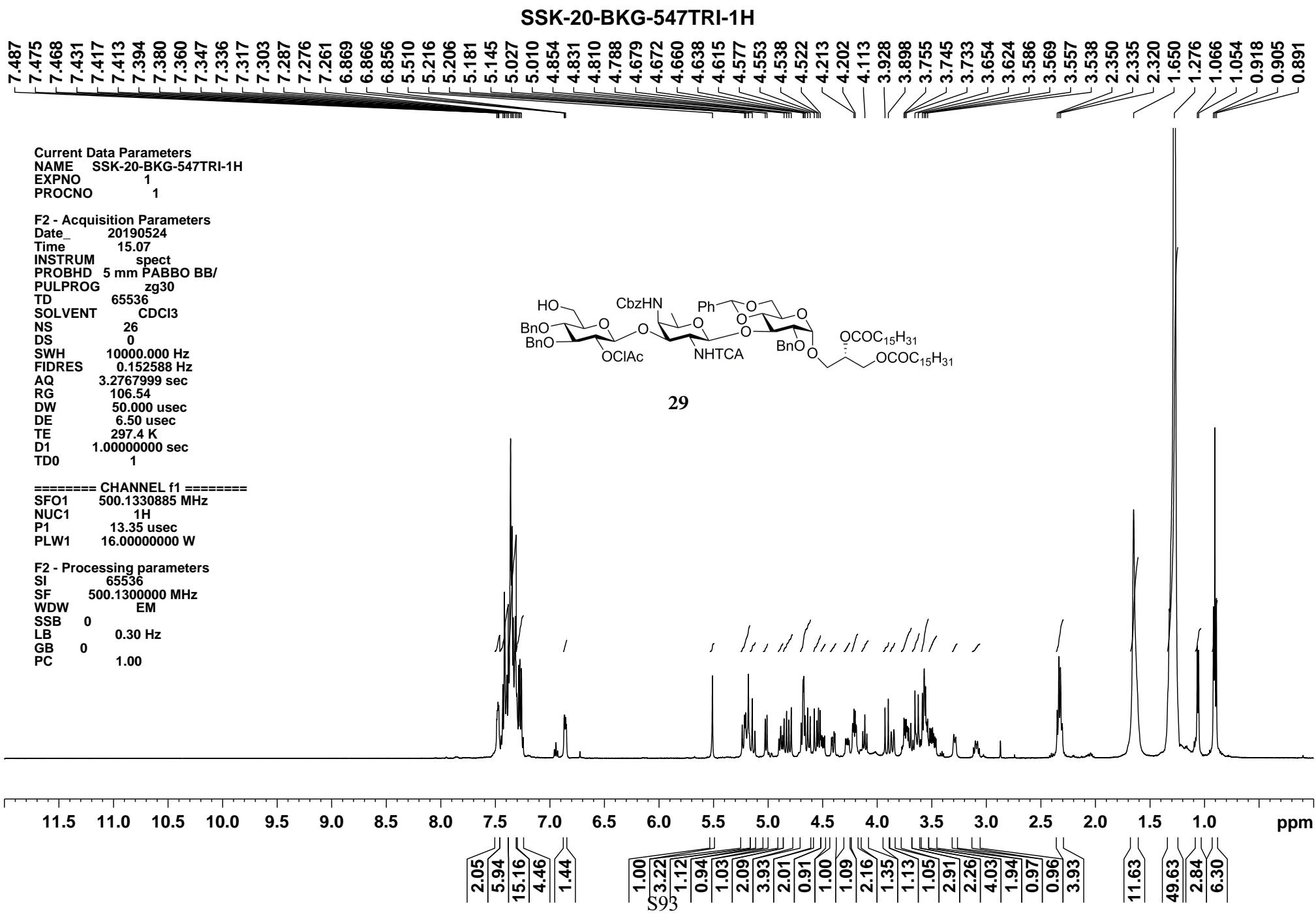
===== GRADIENT CHANNEL =====
GPNAME[1] SINE.100
GPNAME[2] SINE.100
GPZ1 80.00 %
GPZ2 20.10 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 25
SF01 100.6248 MHz
FIDRES 1857.689453 Hz
SW 240.000 ppm
FnMODE Echo-Antiecho

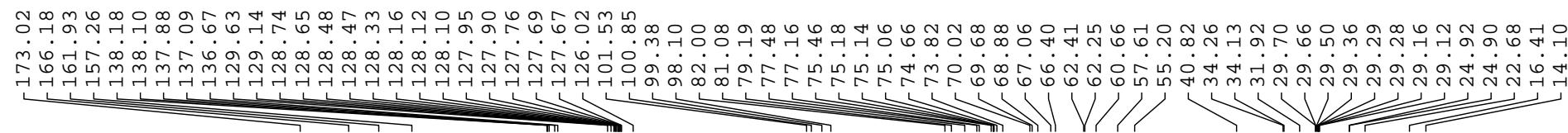
F2 - Processing parameters
SI 2048
SF 400.1300099 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 echo-antiecho
SF 100.6127690 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0

6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 ppm



SSK-20-BKG-546TRI-13



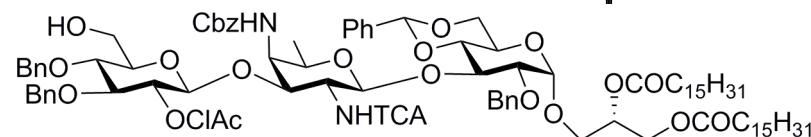
Current Data Parameters
NAME SSK-20-BKG-546TRI-13C
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date 20190527
Time 7.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 10704
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 197.27
DW 16.800 usec
DE 6.50 usec
TE 297.8 K
D1 1.0000000 sec
D11 0.03000000 sec
TD0 1

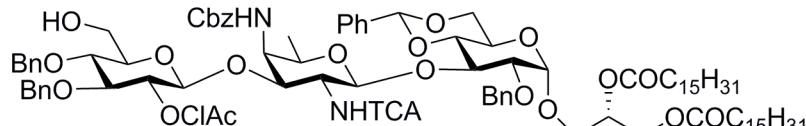
===== CHANNEL f1 =====
SFO1 125.7703637 MHz
NUC1 ¹³C
P1 8.90 usec
PLW1 103.0000000 W

===== CHANNEL f2 =====
SFO2 500.1320005 MHz
NUC2 ¹H
CPDPG[2] waltz16
PCPD2 80.00 usec
PLW2 16.0000000 W
PLW12 0.44556001 W
PLW13 0.22411001 W

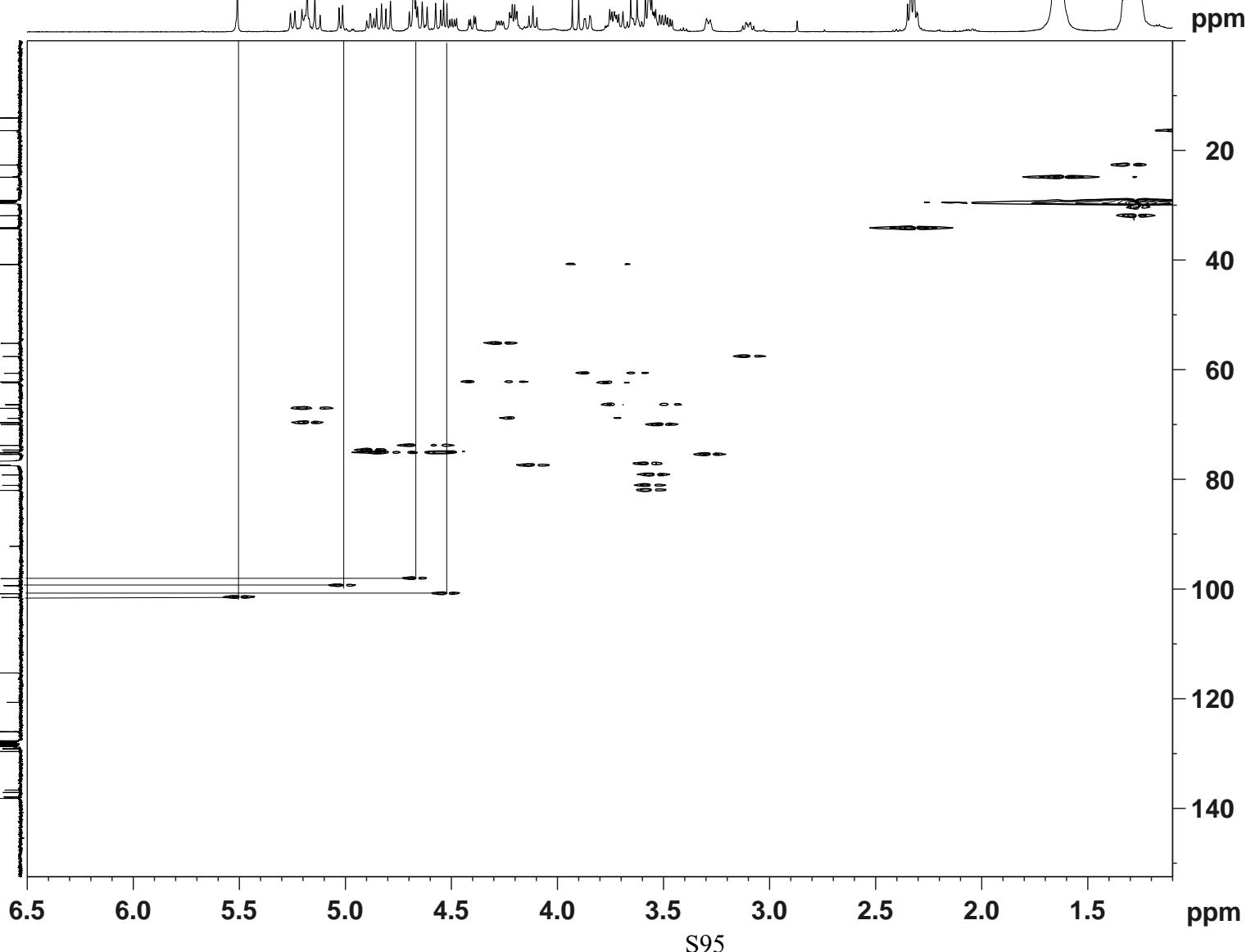
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



SSK-20-BKG-546TRI-HSQC

**29**

CHPh H1 H1''



Current Data Parameters
 NAME SSK-20-BKG-546TRI-HSQC
 EXPNO 6
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190527
 Time 10:34
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG hsqcetgpsisp2.2
 TD 1024
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 7.825020 Hz
 AQ 0.0638976 sec
 RG 197
 DW 62.400 usec
 DE 6.50 usec
 TE 297.6 K
 CNST12 145.000000
 CNST17 -0.5000000
 D1 0.0000000 sec
 D1 1.0000000 sec
 D4 0.00172414 sec
 D11 0.0300000 sec
 D16 0.00020000 sec
 D21 0.00360000 sec
 D24 0.00089000 sec
 IN0 0.00002410 sec

===== CHANNEL f1 ======
 SP01 500.1323506 MHz
 NUC1 13C
 D1 13.35 usec
 P2 26.70 usec
 P28 2000.00 usec
 PLW1 16.00000000 W

===== CHANNEL E2 ======
 SP02 125.7665016 MHz
 NUC2 13C
 CPDPGR[2 bi_p5m4sp_4sp.2 8.90 usec
 P3 500.00 usec
 P14 2000.00 usec
 D63 1500.00 usec
 PLW0 0 W
 PLW2 103.0000000 W
 PLW12 1.66499996 W

SPNAM[3] Crp60,0,5,20.1
 SPQFS3 0 Hz
 SPQFS3 0.500
 SPW3 12.46500015 W
 SPNAM[7] Crp60comp.4
 SPQAL7 0.500
 SPQFS7 0 Hz
 SPW1 12.46500015 W
 SPNAM[14] Crp32,1.5,20.2
 SPQAL14 0.500
 SPQFS14 0 Hz
 SPW14 5.31860018 W
 SPNAM[31] Crp32,1.5,20.2
 SPQAL31 0.500
 SPQFS31 0 Hz
 SPW31 1.32969999 W

===== GRADIENT CHANNEL =====
 GPNAME[1] SMS010.100
 GPNAME[2] SMS010.100
 GPNAME[3] SMS010.100
 GPNAME[4] SMS010.100
 GPZ1 80.00 %
 GPZ2 20.10 %
 GPZ3 11.10 %
 GPZ4 5.50 %
 P16 1000.00 usec
 P19 600.00 usec

F1 - Acquisition parameters
 TD 512
 SF01 125.7666 MHz
 FIDRES 81.042534 Hz
 SW 164.963 ppm
 FmMode Echo-Antiecho

F2 - Processing parameters
 SI 1024
 SF 500.1300000 MHz
 WDW QSINE
 SSB 2
 LB 0 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
 SI 1024
 MC2 echo-antiecho
 SF 125.7577890 MHz
 WDW QSINE
 SSB 2
 LB 0 Hz
 GB 0

SSK-20-BKG-TRI2-1H

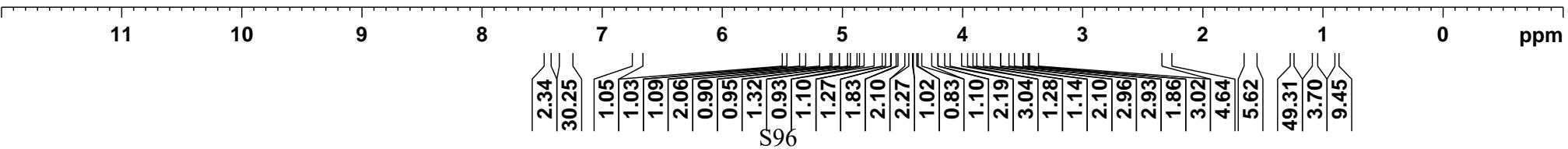
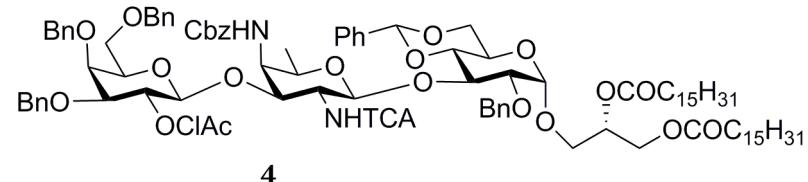


Current Data Parameters
 NAME SSK-20-BKG-TRI2-1H
 EXPNO 8
 PROCNO 1

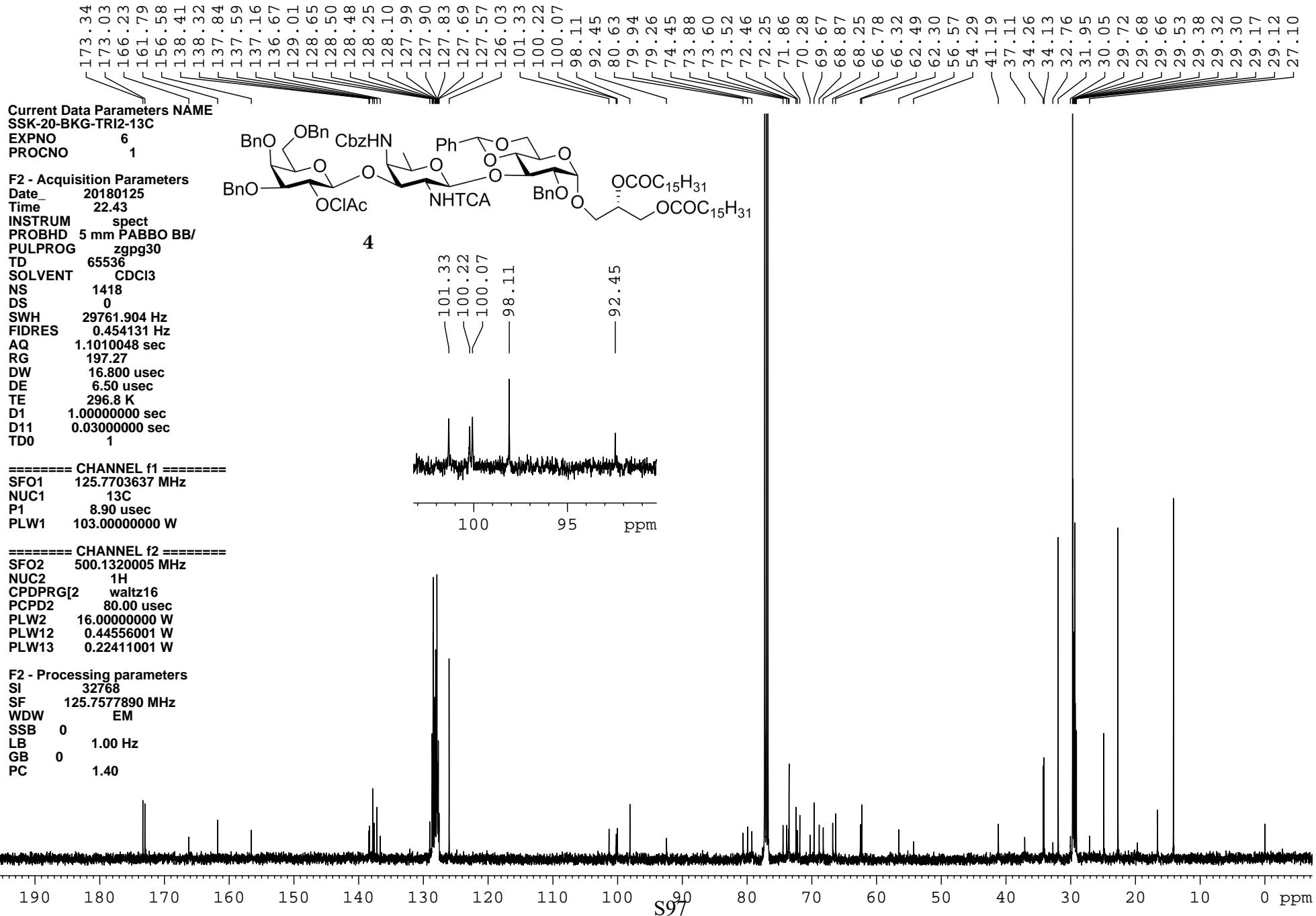
F2 - Acquisition Parameters
 Date_ 20180125
 Time 18.09
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 48
 DS 0
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2767999 sec
 RG 48.36
 DW 50.000 usec
 DE 6.50 usec
 TE 296.7 K
 D1 1.0000000 sec
 TD0 1

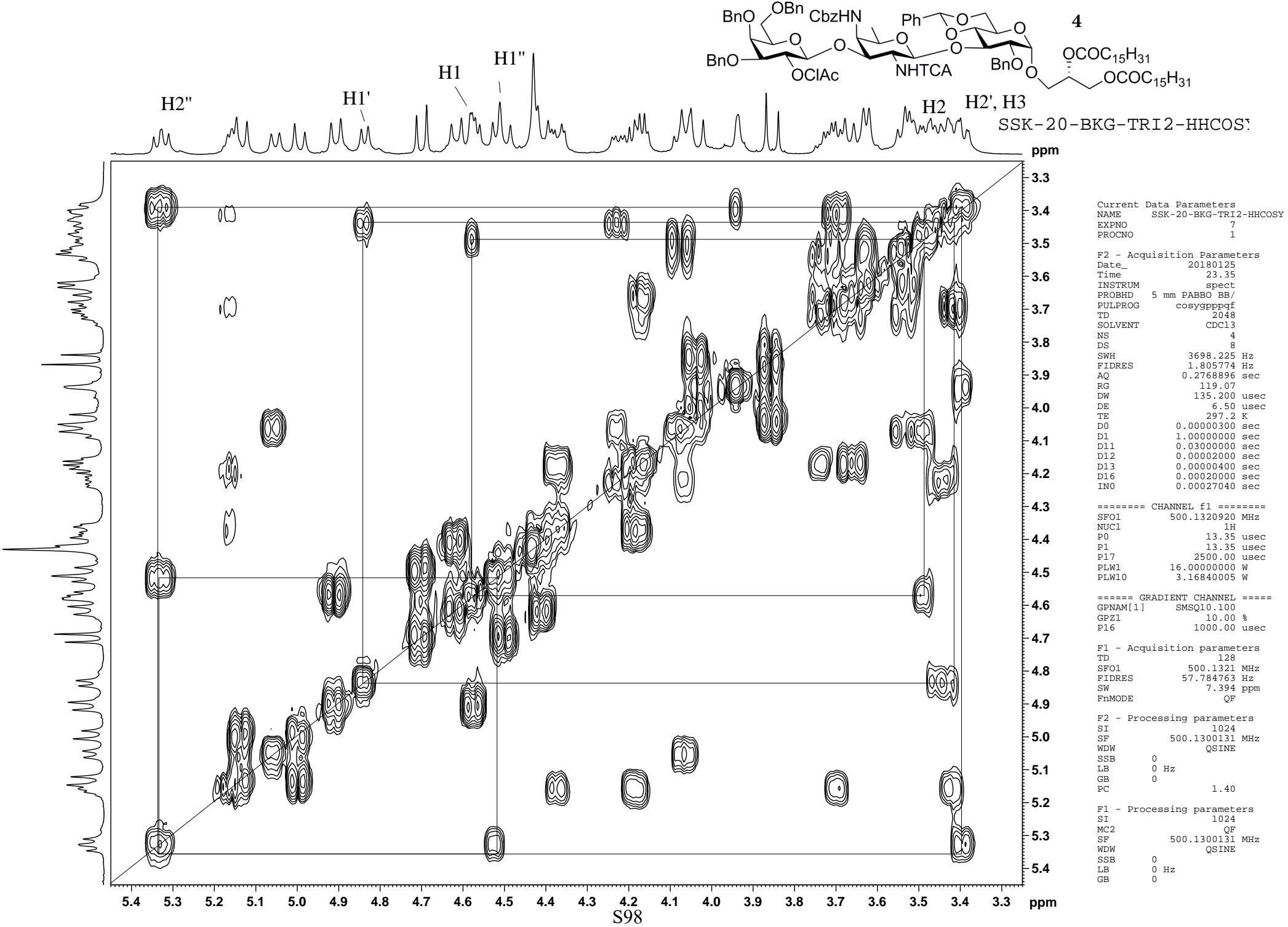
===== CHANNEL f1 =====
 SFO1 500.1330885 MHz
 NUC1 1H
 P1 13.35 usec
 PLW1 16.00000000 W

F2 - Processing parameters
 SI 65536
 SF 500.1300131 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



SSK-20-BKG-TRI2-13C

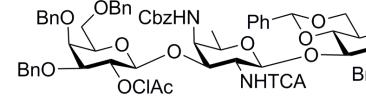




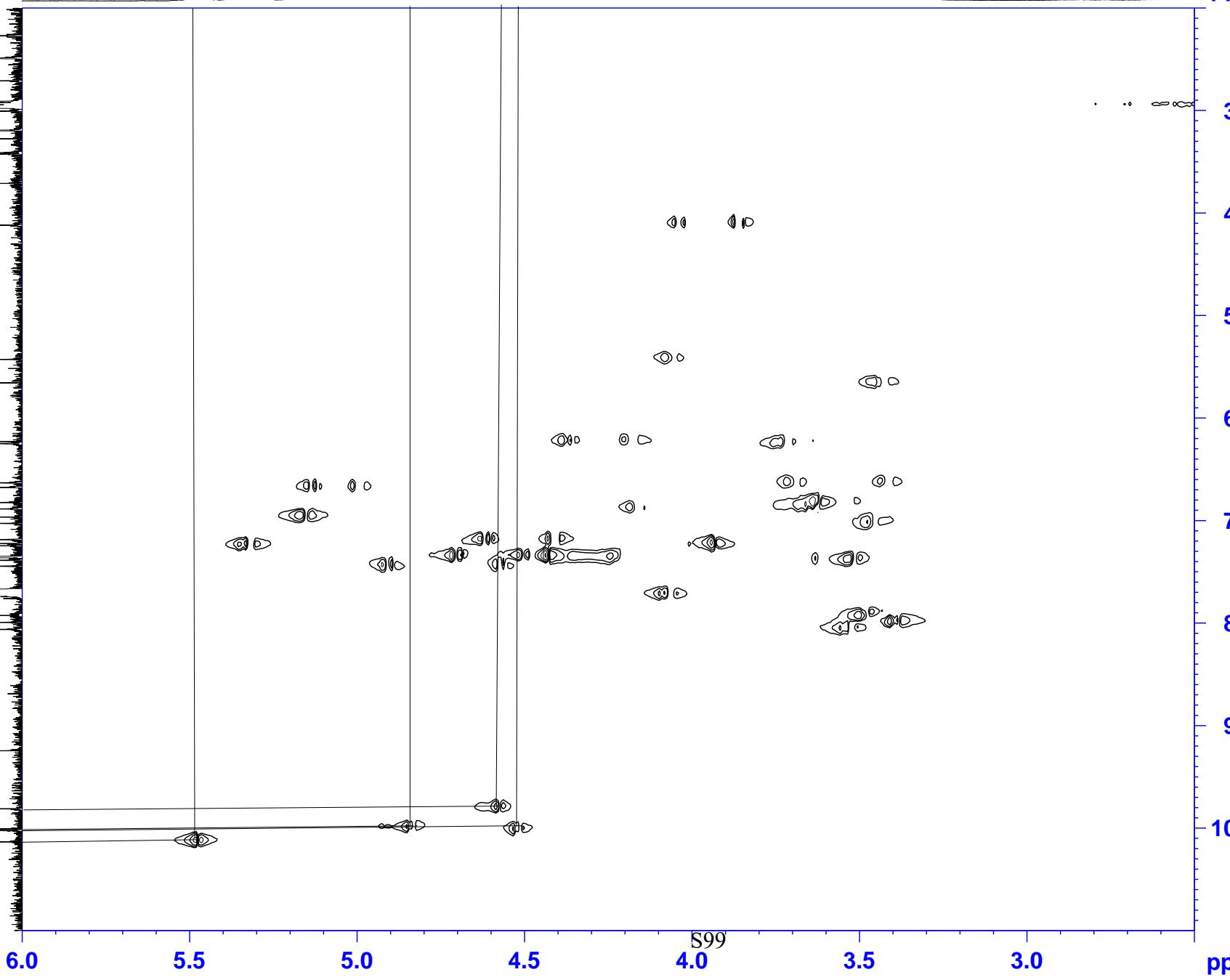
CH

H1'

H1''



ppm



Current Data Parameters
NAME SSK-20-BKG-TRIS-HSQC
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date 20180127
Time 14.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PROBFRG hsqcetgrdpr2.2
TD 3072
SOLVENT CDCl3
NS 8
DS 32
SWH 3787.50 Hz
FIDRES 1.223033 Hz
AQ 0.4055040 sec
RG 197.27
DW 132.000 usec
DE 6.50 usec
TE 27.9 K
CNST2 145.000000
CNST17 -0.500000
DO 0.00000300 sec
D1 1.000000 sec
P1 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D21 0.00360000 sec
D24 0.00089000 sec
IN0 0.00002410 sec

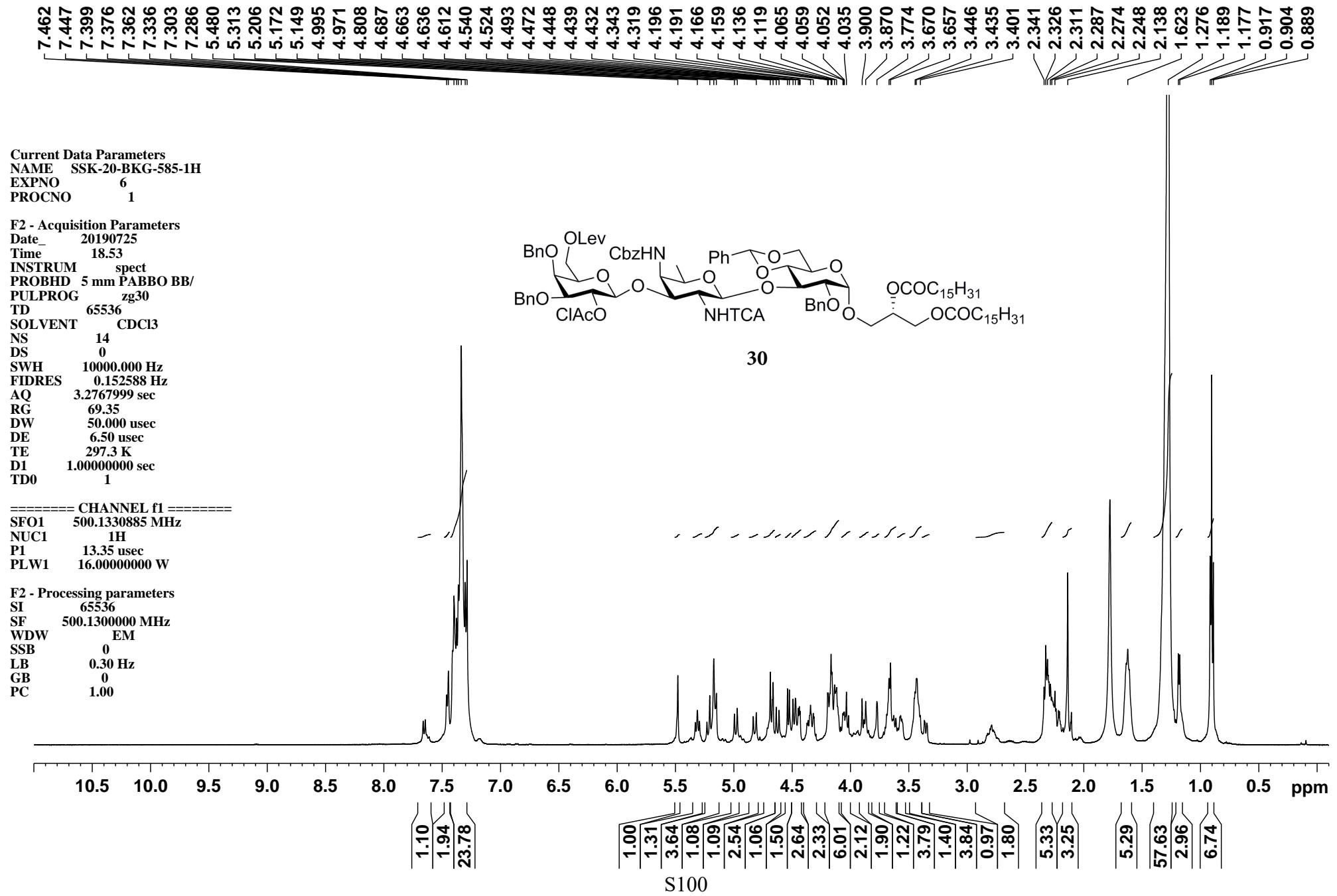
===== CHANNEL f1 =====
SP01 500.1320752 MHz
NUC1 1H
P1 13.00 usec
P2 26.70 usec
P28 1000.00 usec
PLW1 16.0000000 W
===== CHANNEL f2 =====
SP02 125.7665916 MHz
NUC2 13C
CPDPGRG[2 bi_p5m4sp_4sp_2
P3 8.90 usec
P14 50.00 usec
P24 2000.00 usec
P63 1500.00 usec
PLW0 0 W
PLW2 103.0000000 W
PLW2 1.6649996 W
SPNAM[3] Crp60,0,5,20,1
SPNAM[3] 0.500
SPOFFS3 0 Hz
SPW3 12.46500015 W
SPNAM[7] Crp60comp,4
SPW7 0.500
SPOFFS7 0 Hz
SPW7 12.46500015 W
SPNAM[14] Crp32,1,5,20,2
SPNAM[14] 0.500
SPW14 5,31860018 W
SPNAM[31] Crp32,1,5,20,2
SPNAM[31] 0.500
SPOFFS31 0 Hz
SPW31 1.32969999 W
===== GRADIENT CHANNEL =====
GPNAM[1] SMS010.100
GPNAM[2] SMS010.100
GPNAM[3] SMS010.100
GPNAM[4] SMS010.100
GPZ1 80.00 %
GPZ2 20.10 %
GPZ3 11.00 %
GPZ4 5.00 %
P16 1000.00 usec
P19 600.00 usec

F1 - Acquisition parameters
TD 177
SP01 125.7665916 MHz
FIDRES 23.428116 Hz
SW 164.963 ppm
FnMODE Echo-Antiecho

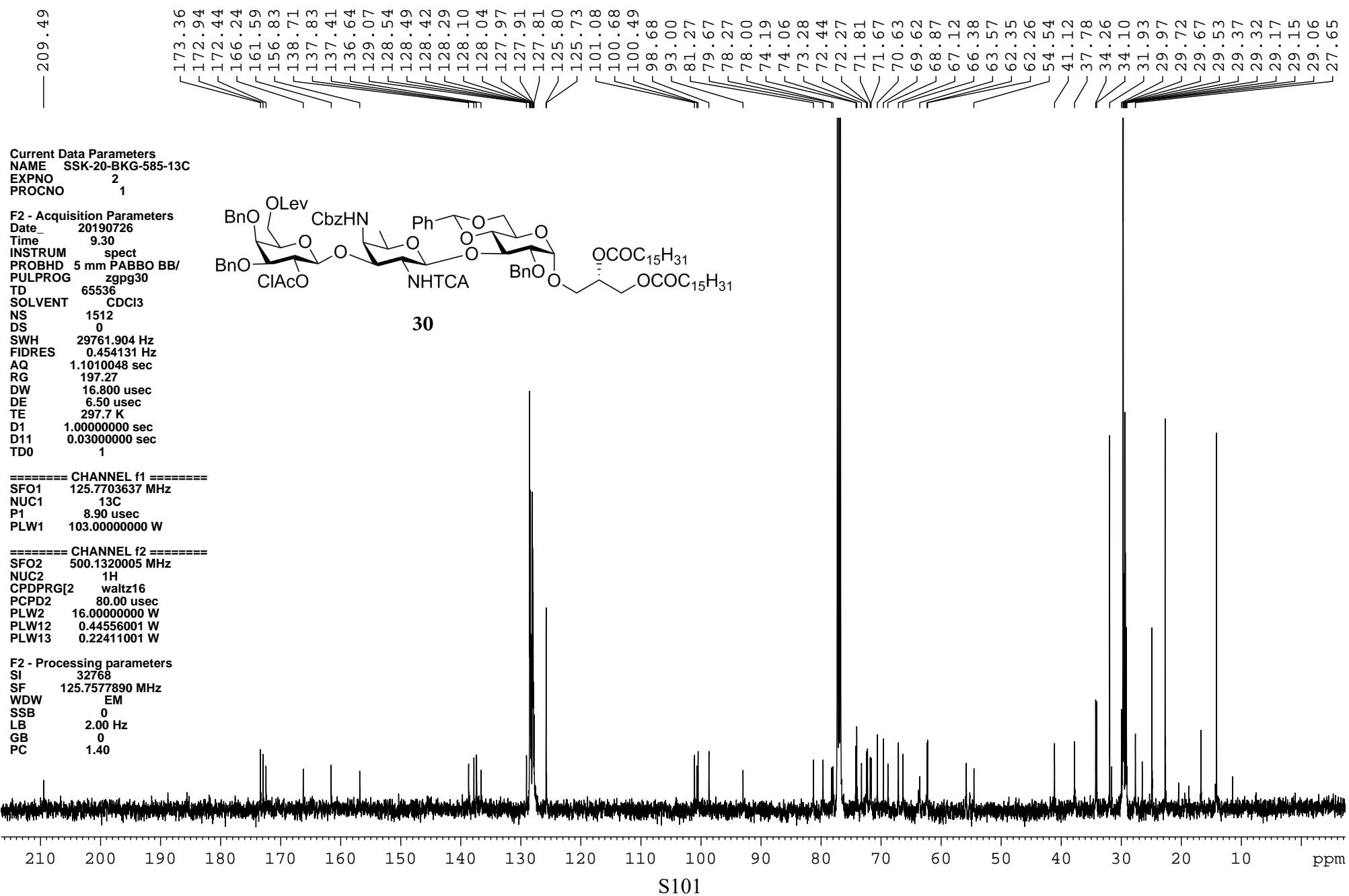
F2 - Processing parameters
SI 32768
SF 500.1300133 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 32768
MC2 echo-antiecho
SF 125.7578023 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0

SSK-20-BKG-585-1H



SSK-20-BKG-585-13C



SSK-20-BKG-585-dept135

Current Data Parameters
 NAME SSK-20-BKG-585-dept135
 EXPNO 6
 PROCNO 1

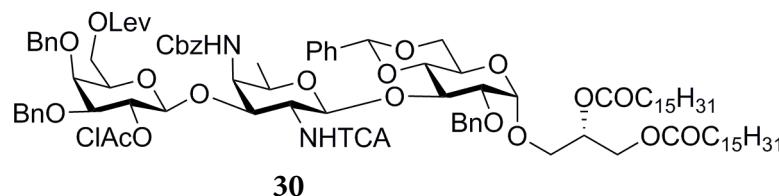
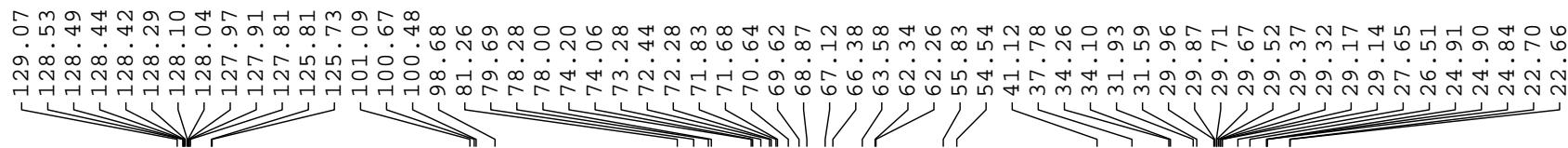
F2 - Acquisition Parameters

Date 20190726
 Time 17.27
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG deptsp135
 TD 65536
 SOLVENT CDCl₃
 NS 532
 DS 0
 SWH 20161.291 Hz
 FIDRES 0.307637 Hz
 AQ 1.6252928 sec
 RG 197.27
 DW 24.800 usec
 DE 6.50 usec
 TE 298.7 K
 CNST2 145.0000000
 D1 1.00000000 sec
 D2 0.00344828 sec
 D12 0.00002000 sec
 TD0 1

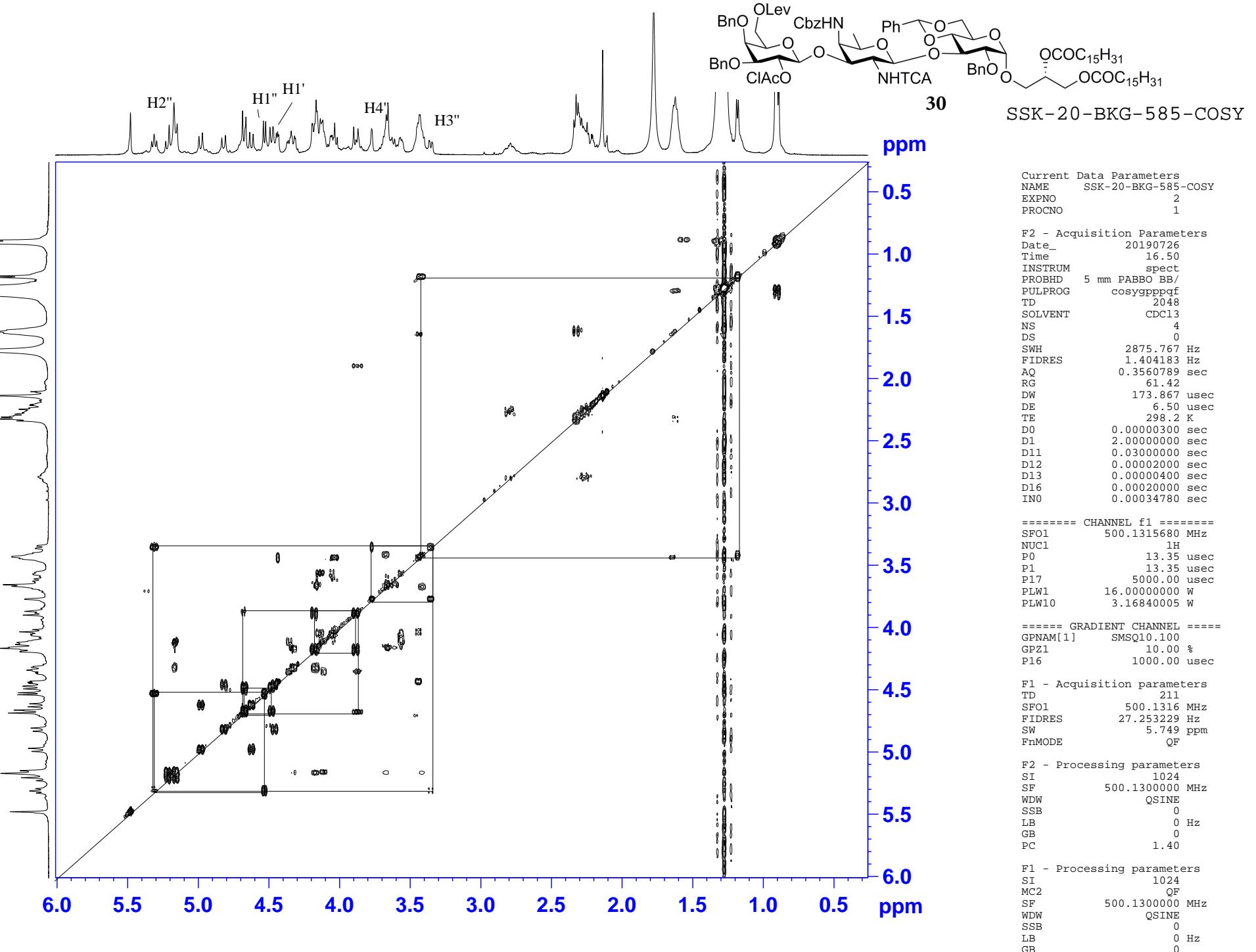
===== CHANNEL f1 ======
 SFO1 125.7678486 MHz
 NUC1 ¹³C
 P1 8.0000 usec
 P13 2000.00 usec
 PLW0 0 W
 PLW1 103.0000000 W
 SPNAM[5] Crp60comp.4
 SPOAL5 0.500
 SPOFFS5 0 Hz
 SPW5 12.46500015 W

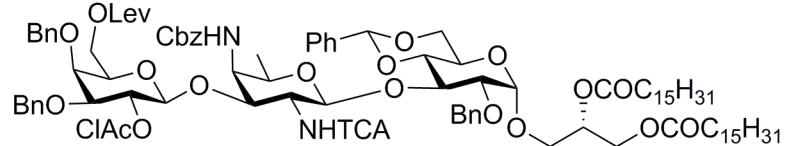
===== CHANNEL f2 ======
 SFO2 500.1315995 MHz
 NUC2 ¹H
 CPDPRG[2] waltz16
 P3 13.35 usec
 P4 26.70 usec
 PCPD2 80.00 usec
 PLW2 16.0000000 W
 PLW12 0.44556001 W

F2 - Processing parameters
 SI 32768
 SF 125.7577890 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



S102





SSK-20-BKG-585-HSQC

CHPh

H1'

H1''

H1

30

ppm

40

50

60

70

80

90

100

S104

Current Data Parameters
NAME SSK-20-BKG-585-HSQC
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date 2010/7/26
Time 16.27
INSTRUM spect

PROBHD 5 mm PABBO BB/
PULPROG hegedetgpsisp2.2

TD 1024

SOLVENT CDCl3

TE 298.2 K

CNST1 145.000000

CNST17 0.500000

D1 0.000000 sec

D4 1.500000 sec

D14 0.00172414 sec

D11 0.03000000 sec

D16 0.00020000 sec

D21 0.00360000 sec

D24 0.00089000 sec

IN0 0.00003800 sec

===== CHANNEL f1 =====

SP01 500.1315679 MHz

SPC1 18

P1 13.15 usec

P2 26.70 usec

P28 2000.00 usec

PLW1 16.0000000 W

===== CHANNEL f2 =====

SP02 125.7650012 MHz

NUC2 13C

CPDPGR[2 bi_p5m4sp_4sp_2

P3 8.90 usec

P14 500.00 usec

P14 2000.00 usec

P63 1500.00 usec

PLW0 0 W

PLW2 103.0000000 W

PLW12 1.66499996 W

SPNAM[3] Crp60,0,5,20.1

SPD1 0.500

SPOFFS3 0 Hz

SPW3 12.46500015 W

SPNAM[7] Crp60ccomp.4

SPQAL7 0.500

SPOFFS7 0 Hz

SPW7 12.46500015 W

SPNAM[14] Crp32,1,5,20.2

SPQAL4 0.500

SPOFFS14 0 Hz

SPW14 5.31860018 W

SPNAM[31] Crp32,1,5,20.2

SPQAL31 0.500

SPOFFS31 0 Hz

SPW31 1.32969999 W

===== GRADIENT CHANNEL =====

GPNAME[1] SMS010.100

GPNAME[2] SMS010.100

GPNAME[3] SMS010.100

GPNAME[4] SMS010.100

GPZ1 80.00 %

GPZ2 20.00 %

GPZ3 10.00 %

GPZ4 -5.00 %

P16 1000.00 usec

P19 600.00 usec

F1 - Acquisition parameters

TD 1024

SP01 125.765 MHz

FIDRES 202.429153 Hz

SW 104.623 ppm

FnMODE Echo-Antiecho

F2 - Processing parameters

SI 1024

SF 500.1300000 MHz

WDW QSIMINE

SSB 2

LB 0 Hz

GB 0

PC 1.40

F1 - Processing parameters

SI 1024

MC2 echo-antiecho

SF 125.7577890 MHz

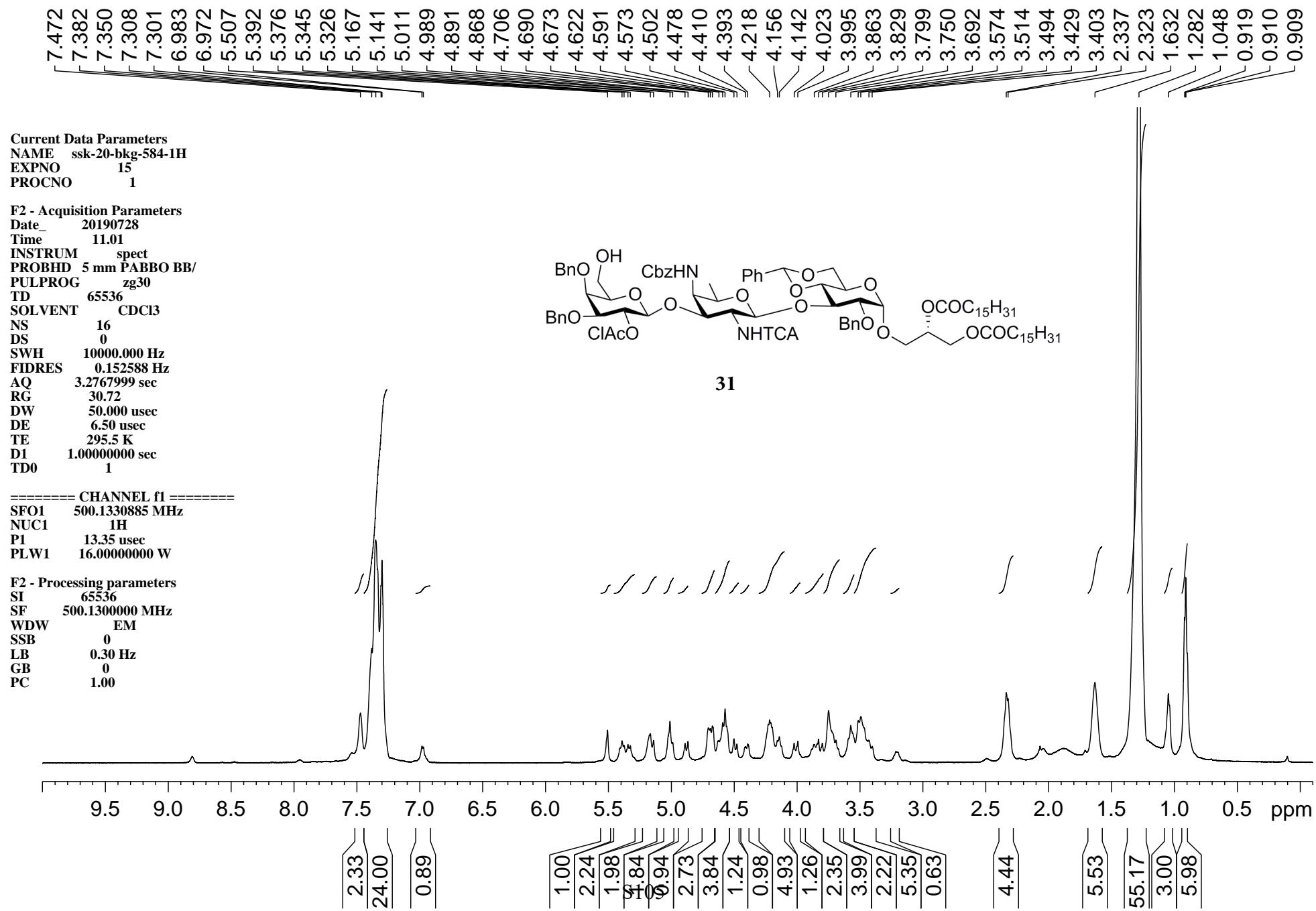
WDW QSIMINE

SSB 2

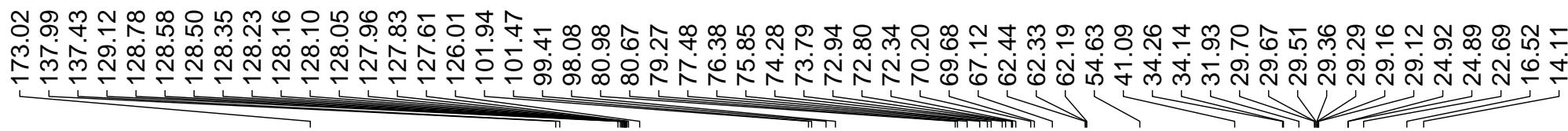
LB 0 Hz

GB 0

ssk-20-bkg-584-1h



ssk-20-bkg-584-13C



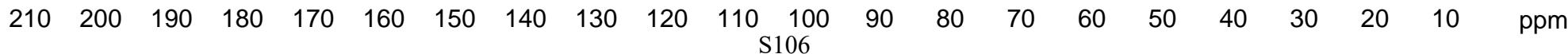
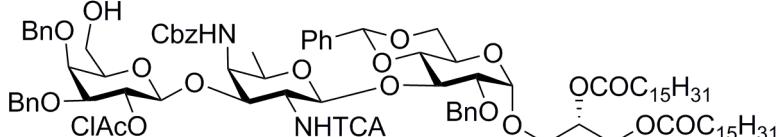
Current Data Parameters
 NAME ssk-20-bkg-584-13C
 EXPNO 14
 PROCNO 1

F2 - Acquisition Parameters
 Date 20190728
 Time 1.00
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl₃
 NS 7000
 DS 0
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010048 sec
 RG 197.27
 DW 16.800 usec
 DE 6.50 usec
 TE 296.3 K
 D1 1.0000000 sec
 D11 0.03000000 sec
 TD0 1

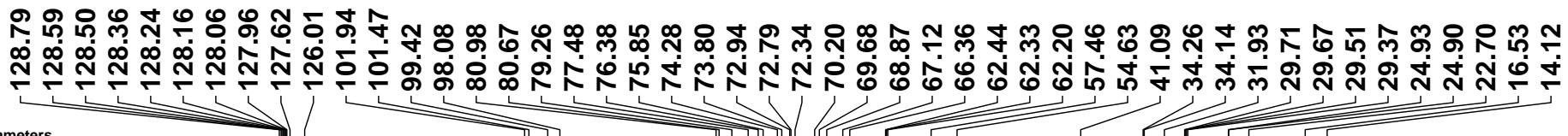
===== CHANNEL f1 ======
 SFO1 125.7703637 MHz
 NUC1 ¹³C
 P1 8.90 usec
 PLW1 103.00000000 W

===== CHANNEL f2 ======
 SFO2 500.1320005 MHz
 NUC2 ¹H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 16.00000000 W
 PLW12 0.44556001 W
 PLW13 0.22411001 W

F2 - Processing parameters
 SI 32768
 SF 125.7577890 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



ssk-20-bkg-584-DEPT135



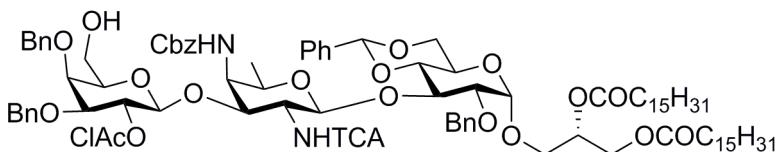
Current Data Parameters
 NAME ssk-20-bkg-584-DEPT135
 EXPNO 14
 PROCNO 1

F2 - Acquisition Parameters
 Date 20190728
 Time 8.55
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG deptsp135
 TD 65536
 SOLVENT CDCl3
 NS 5000
 DS 0
 SWH 20161.291 Hz
 FIDRES 0.307637 Hz
 AQ 1.6252928 sec
 RG 197.27
 DW 24.800 usec
 DE 6.50 usec
 TE 297.1 K
 CNST2 145.0000000
 D1 1.0000000 sec
 D2 0.00344828 sec
 D12 0.00002000 sec
 TD0 1

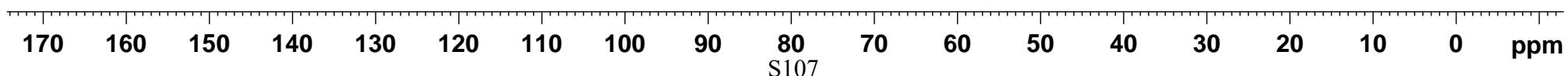
===== CHANNEL f1 =====
 SFO1 125.7678486 MHz
 NUC1 13C
 P1 8.90 usec
 P13 2000.00 usec
 PLW0 0 W
 PLW1 103.0000000 W
 SPNAM[5] Crpdcomp4
 SPOAL5 0.500
 SPOFFS5 0 Hz
 SPW5 12.46500015 W

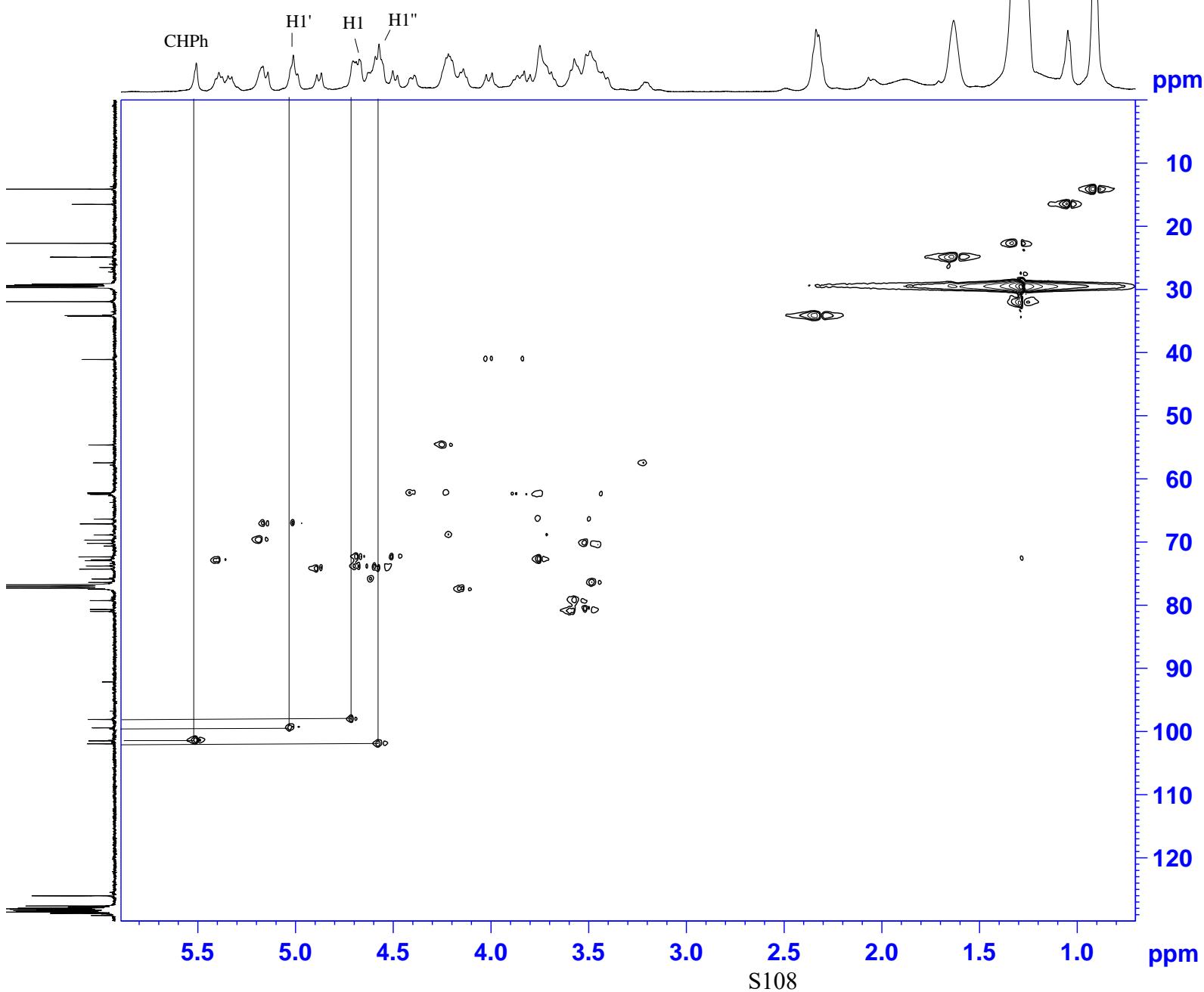
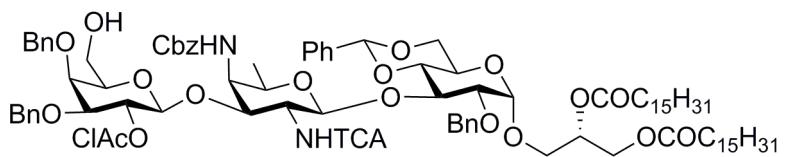
===== CHANNEL f2 =====
 SFO2 500.1315995 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 P3 13.35 usec
 P4 26.70 usec
 PCPD2 80.00 usec
 PLW2 16.00000000 W
 PLW12 0.44556001 W

F2 - Processing parameters
 SI 32768
 SF 125.7577890 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



31





ssk-20-bkg-584-HSQC

Current Data Parameters
NAME ssk-20-bkg-584-HSQC
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
TD 2048000 points
Time 0.27 sec
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG hsqcetgpsisp2.2
TE 1024
SOLVENT CDCl3
NS 4
DS 0
SWH 2596.054 Hz
FIDRES 2.535209 Hz
AQ 0.122424 sec
RG 19.27
DW 192.600 usec
DE 6.50 usec
TE 296.4 K
CNST2 145.000000
CNST17 -0.100000
D0 0.00000000 sec
D1 1.00000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D25 0.00020000 sec
D21 0.00000000 sec
D24 0.00089000 sec
INO 0.00002410 sec

===== CHANNEL f1 =====
SF01 500.131640 MHz
NUC1 1H
P1 13.35 usec
P2 26.70 usec
P28 2000.00 usec
PLW1 16.0000000 W

===== CHANNEL f2 =====
SF02 125.7665916 MHz
NUC2 13C
CPDPGR[2 bi_p5m4sp_4sp_2
P3 8.90 usec
P14 500.00 usec
P24 2000.00 usec
P63 1500.00 usec
PLW0 0 W
PLW2 103.0000000 W
PLW4[3] Crp60.0_5.1
SPQAL3 0.500
SPOFFS3 0 Hz
SPW3 12.46500015 W
SPNAM[7] Crp60ccmp_4
SPQAL7 0.500
SPOFFS7 0 Hz
SPW7 12.46500015 W
SPNAM[14] Crp32.1_5.20_2
SPQAL14 0.500
SPOFFS14 0 Hz
SPW15 5.3180000 W
SPNAM[31] Crp32.1_5.20_2
SPQAL31 0.500
SPOFFS31 0 Hz
SPW31 1.32969999 W

===== GRADIENT CHANNEL =====
GPNNM[1] SMS010.100
GPNNM[2] SMS010.100
GPNNM[3] SMS010.100
GPNNM[4] SMS010.100
GPZ1 80.00 \$
GPZ2 20.10 \$
GPZ3 11.00 \$
GPZ4 -5.00 \$
P16 1000.00 usec
P19 600.00 usec

F1 - Acquisition parameters
TD 175
SF01 125.7666 MHz
FIDRES 237.107285 Hz
SW 164.963 ppm
FmMode Echo-Antiecho

F2 - Processing Parameters
SI 1024
SF 500.1300000 MHz
WDW QSINE
SSB 0 Hz
LB 0 Hz
GB 0
PC 1.40

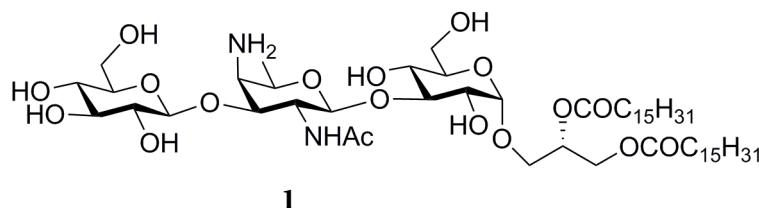
F1 - Processing parameters
SI 1024
MC2 echo-antiecho
SF 125.7577890 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0

SSK-20-BKG-269-1H

Current Data Parameters
 NAME SSK-20-BKG-269-1H
 EXPNO 8
 PROCNO 1

F2 - Acquisition Parameters

Date 20180223
 Time 20.02
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT MeOD
 NS 100
 DS 0
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2767999 sec
 RG 85.91
 DW 50.000 usec
 DE 6.50 usec
 TE 297.3 K
 D1 1.00000000 sec
 TD0 1

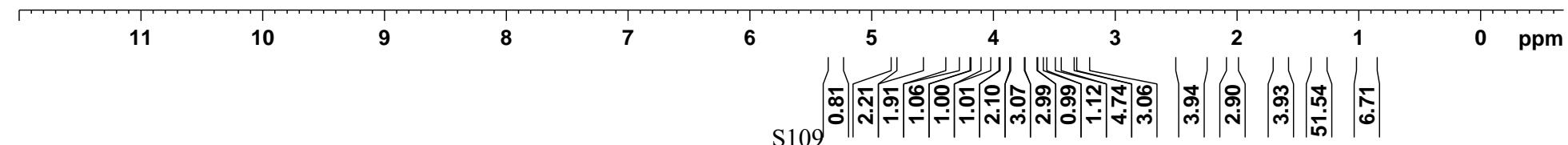


===== CHANNEL f1 =====

SFO1 500.130885 MHz
 NUC1 1H
 P1 13.35 usec
 PLW1 16.0000000 W

F2 - Processing parameters

SI 65536
 SF 500.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



SSK-20-BKG-269-13C

Current Data Parameters

NAME SSK-20-BKG-269-13C
 EXPNO 9
 PROCNO 1

F2 - Acquisition Parameters
 Date 20180312
 Time 17.01
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT MeOD
 NS 13736
 DS 0
 SWH 26041.666 Hz
 FIDRES 0.397364 Hz
 AQ 1.2582912 sec
 RG 1620
 DW 19.200 usec
 DE 6.50 usec
 TE 296.1 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====

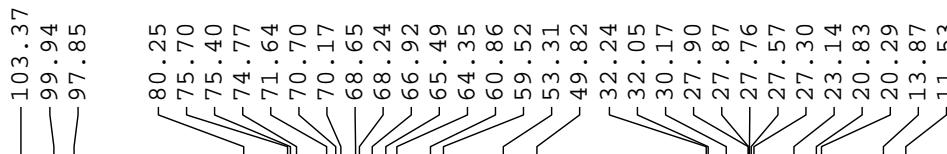
NUC1 13C
 P1 8.50 usec
 PL1 -2.00 dB
 PL1W 56.53121948 W
 SFO1 100.6238364 MHz

===== CHANNEL f2 =====

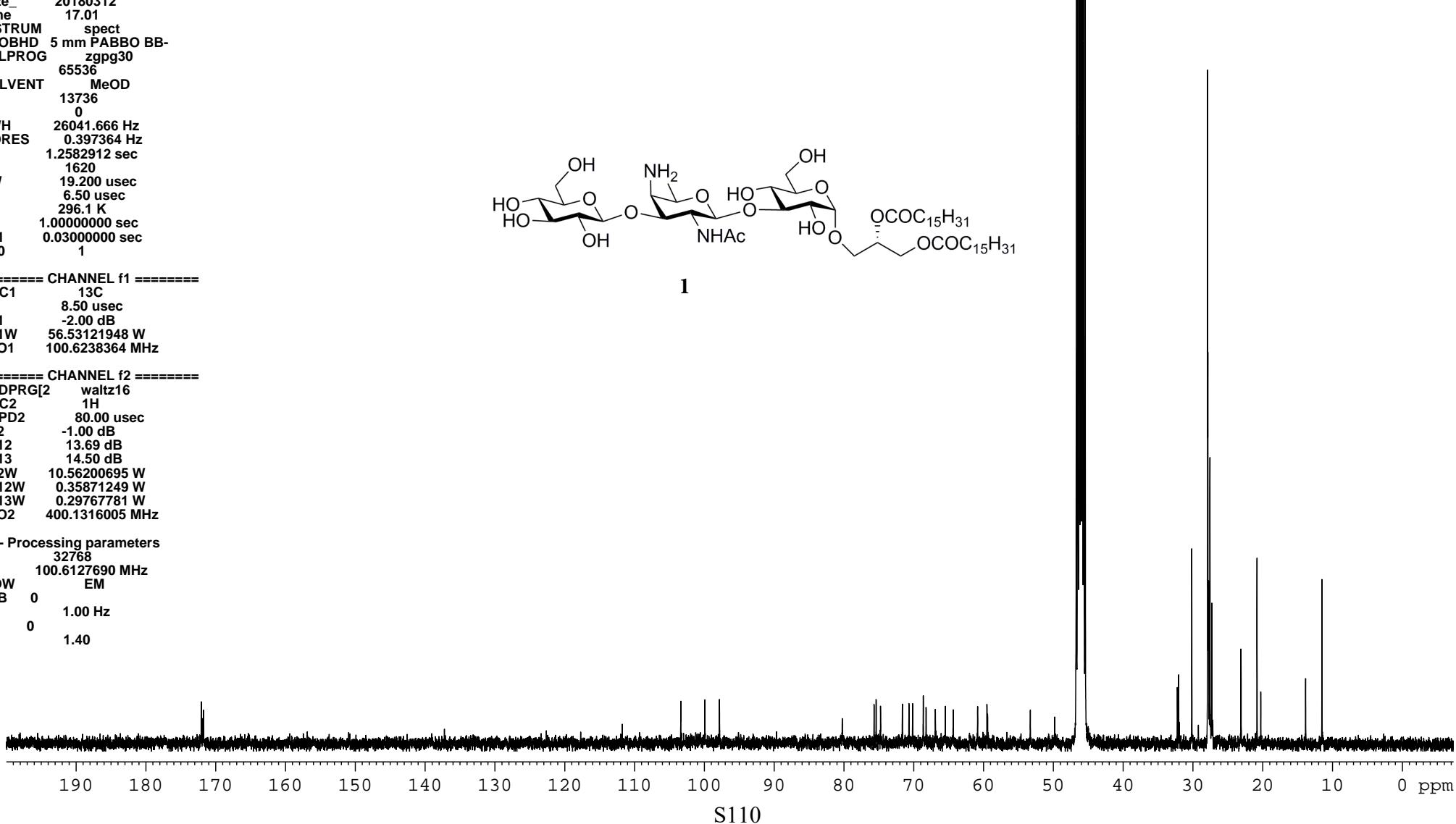
CPDPRG[2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 13.69 dB
 PL13 14.50 dB
 PL2W 10.56200695 W
 PL12W 0.35871249 W
 PL13W 0.29767781 W
 SFO2 400.1316005 MHz

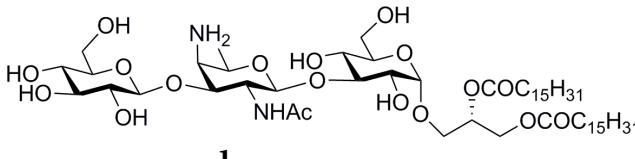
F2 - Processing parameters

SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

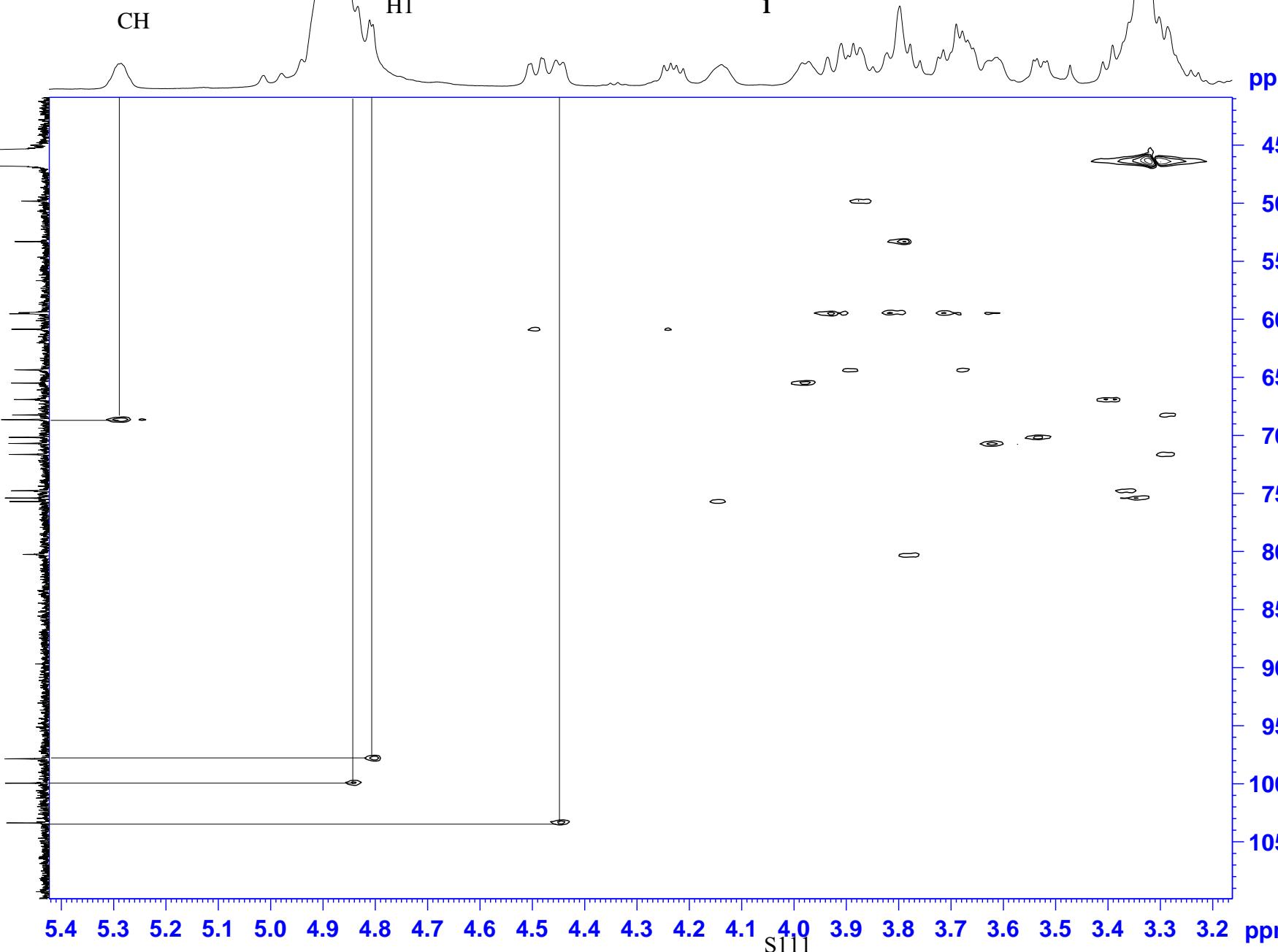


1





SSK-20-BKG-269-HSQC



Current Data Parameters
NAME SSK-20-BKG-269-HSQC
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180811
Time 23.50
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG hsqcetgpsisp2.2
TD 65536
T2 1024
SOLVENT MeOD
NS 8
DS 0
SWH 2551.021 Hz
FIDRES 2.493124 Hz
AQ 0.0007640 sec
RG 197.27
DW 196.000 usec
DE 6.50 usec
TE 298.1 K
CNS172 145.000000
CNS17 145.000000
D0 0.00000300 sec
D1 1.00000000 sec
D4 0.00072414 sec
D11 0.00000000 sec
D16 0.00020000 sec
D21 0.00360000 sec
D24 0.00089000 sec
IN0 0.00002410 sec

===== CHANNEL f1 =====
SP01 500.1315557 MHz
NUC1 1H
P1 13.35 usec
P2 26.70 usec
P28 1000.00 usec
PLW1 16.0000000 W
===== CHANNEL f2 =====
SP02 125.7665916 MHz
NUC2 13C
CPDPFG[2 bi_p5m4sp_4s_2 8.90 usec
P14 500.00 usec
P24 2000.00 usec
P63 1500.00 usec
PLW0 0 W
PLW2 103.0000000 W
PLW12 1.66499999 W
SPNAM[3] Crp60_0.5,20.1
SP0AL3 0.500
SP0FFS3 0 Hz
SPW3 12.46500015 W
SPNAM[7] Crp60comp.4
SP0AL7 0.500
SP0FFS7 0 Hz
SPW7 12.46500015 W
SPNAM[14] Crp32_1.5,20.2
SP0AL14 0.500
SP0FFS14 0 Hz
SPW14 5.31860018 W
SPNAM[31] Crp32_1.5,20.2
SP0AL31 0.500
SP0FFS31 0 Hz
SPW31 1.32969999 W

===== GRADIENT CHANNEL =====
GPNAME[1] SMSQ10.100
GPNAME[2] SMSQ10.100
GPNAME[3] SMSQ10.100
GPNAME[4] SMSQ10.100

GPZ1 80.00 \$
GPZ2 20.10 \$
GPZ3 11.00 \$
GPZ4 -5.00 \$
P16 1000.00 usec
P19 600.00 usec

F1 - Acquisition parameters
TD 513
SP01 125.7666 MHz
FIDRES 80.884552 Hz
SW 164.963 ppm
FmMode Echo-Antiecho

F2 - Processing parameters
SI 1024
SF 500.1300068 MHz
MW 1000 QSIIN
SSB 2
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 echo-antiecho
SF 125.7579708 MHz
MW 1000 QSIIN
SSB 2
LB 0 Hz
GB 0

SSK-20-BKG-260(3)-1F

Current Data Parameters
 NAME SSK-20-BKG-260(3)-1H
 EXPNO 6
 PROCNO 1

F2 - Acquisition Parameters

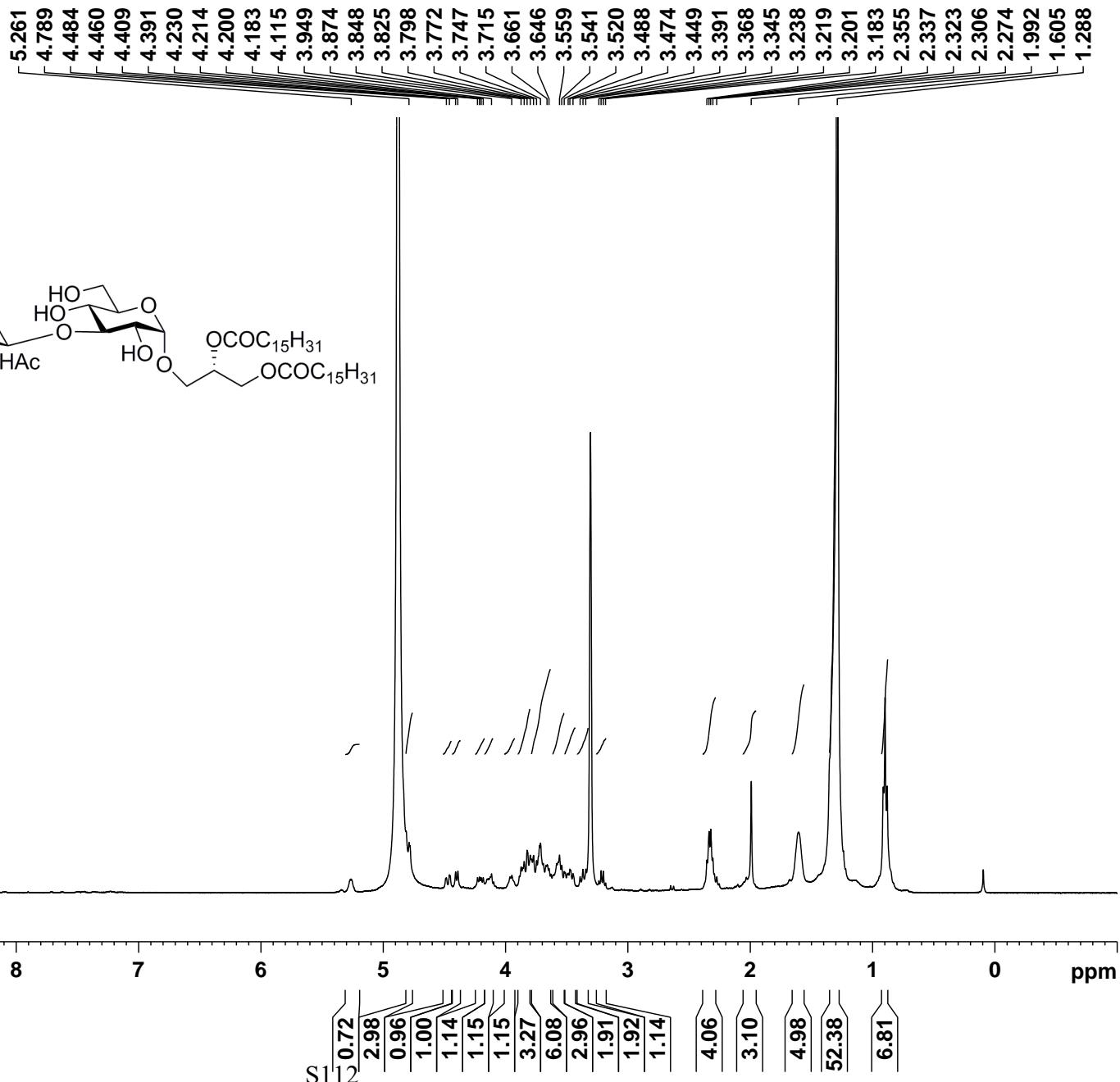
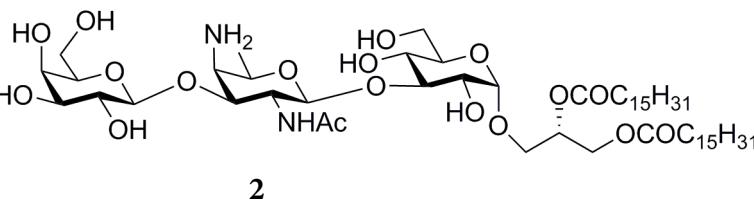
Date 20180210
 Time 17.56
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 54274
 SOLVENT MeOD
 NS 17
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.151522 Hz
 AQ 3.2998593 sec
 RG 114
 DW 60.800 usec
 DE 6.50 usec
 TE 297.6 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====

NUC1 1H
 P1 14.75 usec
 PL1 -1.00 dB
 PL1W 10.56200695 W
 SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
NAME SSK-20-BKG-260(3)-13C
EXPNO 9
PROCNO 1

F2 - Acquisition Parameters
Date 20180211
Time 1.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 10886
DS 0
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2582912 sec
RG 2050
DW 19.200 usec
DE 6.50 usec
TE 298.6 K
D1 1.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 56.53121948 W
SFO1 100.6238364 MHz

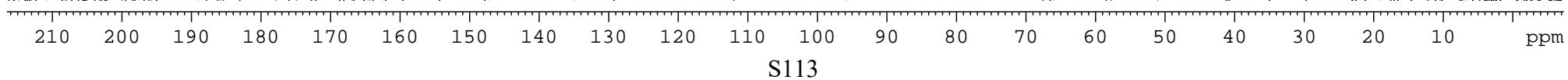
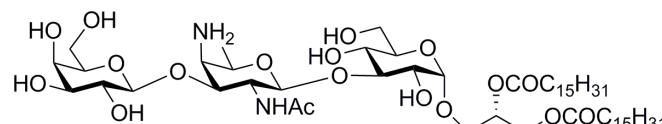
===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 13.69 dB
PL13 14.50 dB
PL2W 10.56200695 W
PL12W 0.35871249 W
PL13W 0.29767781 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

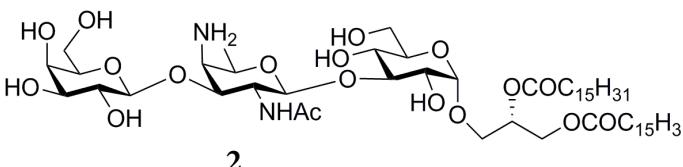
SSK-20-BKG-260(3)-13C

173.61
173.42
173.26

105.54
101.53
99.35
81.96
77.26
75.65
73.06
72.19
71.62
70.83
70.16
68.85
68.42
67.03
65.86
62.38
61.35
60.94
54.96
51.46
33.76
33.57
31.69
29.42
29.39
29.27
29.09
28.82
24.65
22.34
21.82
15.42
13.06
7.82



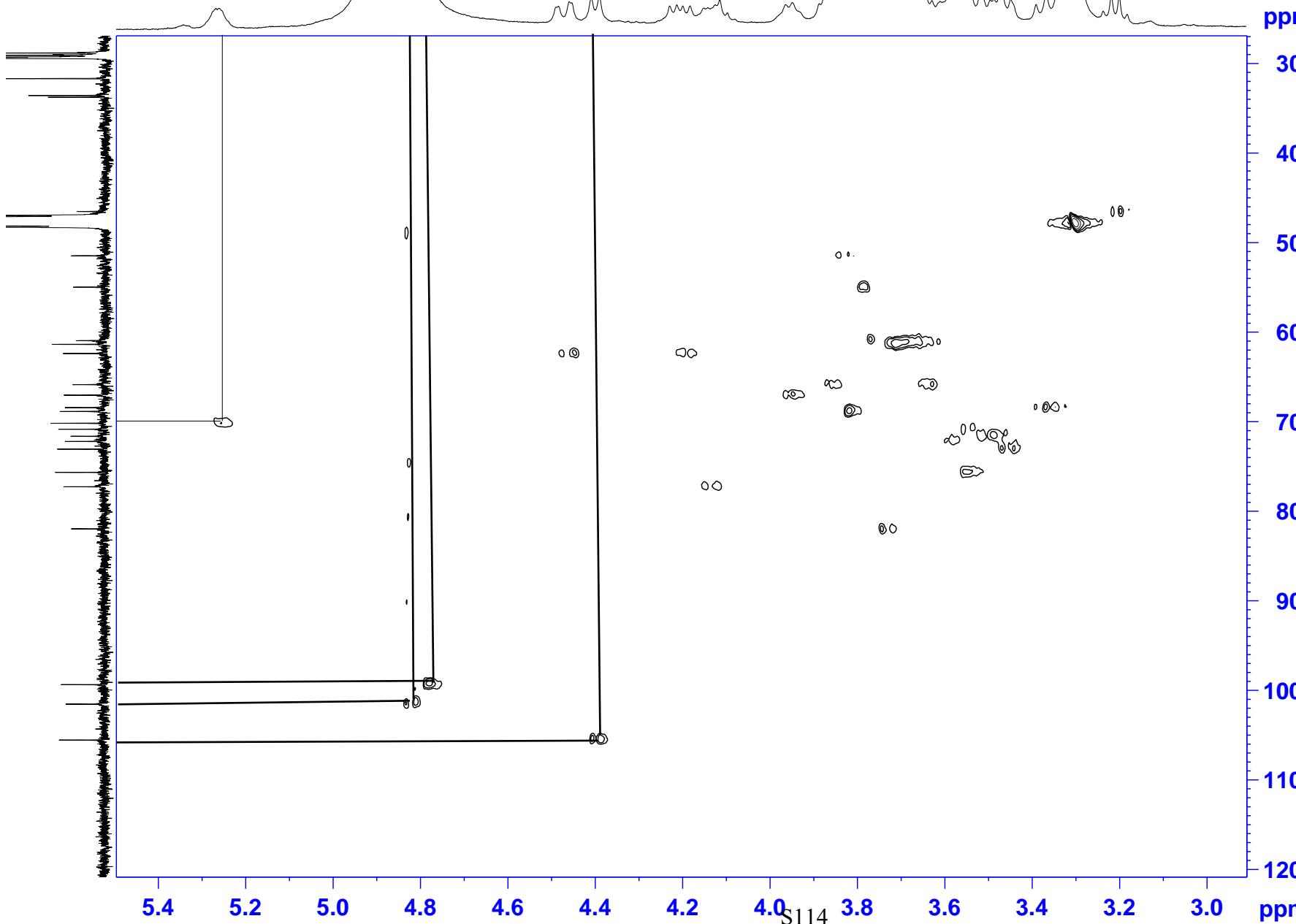
S113



SSK-20-BKG-260 (3)-HSQC

CH

H1



Current Data Parameters
NAME SSK-20-BKG-260(3)-HSQC
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180210
Time 18.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG hsqcetgp
TD 2048
SOLVENT MeOD
NS 8
DS 16
SWH 2556.237 Hz
FIDRES 1.248163 Hz
AQ 0.4005888 sec
RG 2050
DW 195.600 usec
DE 6.50 usec
TE 298.0 K
CNUST2 145.000000 sec
D0 0.00000300 sec
D1 1.0000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00002070 sec
ZGOPTNS

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
P2 29.50 usec
P28 0.10 usec
PL1 -1.00 dB
PL1W 10.56200695 W
SFO1 400.1310621 MHz

===== CHANNEL f2 =====
CPDPGR12 garp
NUC2 13C
P3 8.50 usec
P4 17.00 usec
PCPD2 70.00 usec
PL2 -2.00 dB
PL12 16.31 dB
PL2W 56.53121948 W
PL12W 0.83423501 W
SFO2 100.6248425 MHz

===== GRADIENT CHANNEL =====
GPNAME[1] SINE_100
GPNAME[2] SINE_100
GPZ1 80.00 °
GPZ2 20.10 °
P16 1000.00 usec

F1 - Acquisition parameters
TD 256
SFO1 100.6248 MHz
FIDRES 188.671585 Hz
SW 240.000 ppm
F1MODE Echo-Antiecho

F2 - Processing parameters
SI 2048
SF 400.1300095 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 echo-antiecho
SF 100.6127690 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0