Supporting information for:

Stereochemical effects of chiral monolayers on enhancing the resistance to mammalian cell adhesion

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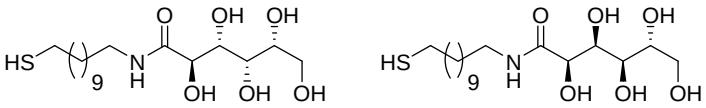
Materials and Methods

Chemicals. 1-Pentadecanethiol and chemicals used for synthesizing all other alkanethiols were purchased from Aldrich Chemicals (Milwaukee, WI) and used as received. Ethanol was used as a solvent for preparing all alkanethiol solutions and for washing the SAM modified gold substrates. A minimum amount of DMSO was mixed with ethanol for solubilizing polyol-terminated alkanethiols, which were poorly soluble in pure ethanol. Bovine serum albumin was purchased from Sigma Chemical Company (St. Louis, MO). Phosphate buffered saline, 1 × PBS (2.7 mM potassium chloride, 137 mM sodium chloride, 8 mM sodium phosphate dibasic, 1.48 mM potassium phosphate monobasic, pH 7.42) was prepared by dissolving 5 PBS tablets (SIGMA, Allentown, PA) in 1000 mL of deionized water with a resistivity of 18 MΩ cm (Millipore, Billerica, MA).

General Information for Synthesis of Alkanethiols. The processes involving reactants sensitive to moisture or air were executed under an atmosphere of argon using oven-dried glassware. Reagents and solvents were reagent grade and used as supplied unless otherwise mentioned. THF was distilled from sodium benzophenone ketyl. Solvents were removed under reduced pressure using rotary evaporator below 40 ºC. EMD Silica Gel 60 F_{254} precoated plates (0.25-mm thickness) were used for TLC and a solution of phosphomolybdic acid/ ceric sulfate/ sulfuric acid (10g : 1.25 g : 8% 250 mL), followed by charring at ~ 150 ºC, was used for visualization. Flash column chromatography was performed using SILICYCLE, Silica-P Flash Silica Gel with 40-63μ mesh size. NMR spectra (^{1}H, ^{13}C) were recorded on 300 MHz Bruker instrument. ^{1}H chemical shifts are reported in ppm relative to $CDCl_3$ δ 7.26 and DMSO-d_6 δ 2.50. ^{13}C chemical shifts are reported relative to $CDCl_3$ δ 77.23 and DMSO-d_6 δ 39.51. (High

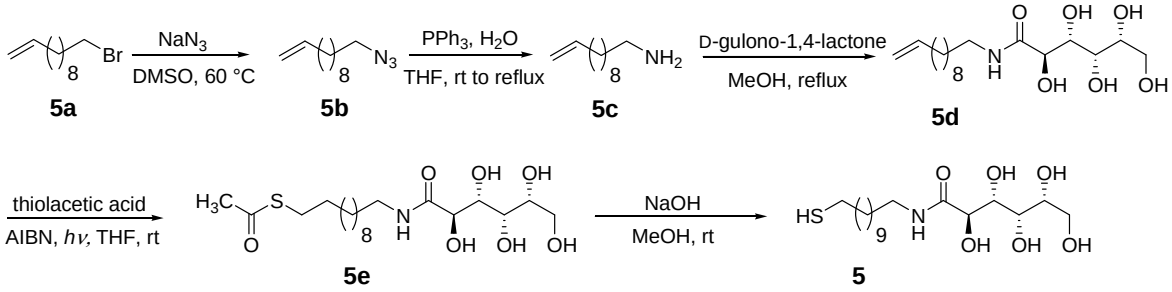
Resolution Mass Spectra) HRMS samples were analyzed by positive ion electrospray or electron impact.

Scheme S1



5 (D-gulonamide) **6 (D-gluconamide)**

Scheme S2



5a **5b** **5c** **5d** **5e** **5**

The synthesis for **5** is shown in Scheme S2. Briefly, the alkene, **5d** was obtained by aminolysis,[1] of D-gulono-1,4-lactone in refluxing methanol. Reaction of **5d** with thiolacetic acid using catalytic amount of AIBN (azobisisobutyronitrile) under UV light afforded the thioester **5e**. Base hydrolysis of the thioester **5e** with methanolic NaOH followed by acidification afforded alkanethiol **5**. Compound **6** was synthesized similarly.

Synthesis of Compound 5b. NaN_3 (1.115 g, 17.153 mmol) was added to a solution of 11-bromo-1-undecene, **5a** (2.000 g, 8.576 mmol) in DMSO (20.0 mL) and the mixture was stirred at rt for 10 min followed by heating at 60 ºC for 2 h. The mixture was cooled to rt followed by addition of 20 mL of brine. The organic layer was extracted with DCM and the combined organic extracts were dried over anhydrous Na_2SO_4. Evaporation of solvent yielded the crude product as yellow oil. The crude product was purified using silica gel column (Hexanes → 5%

EtOAc in Hexanes) to obtain pure compound, **5b** (1.028 g, 61%) as a colorless oil. R_f = 0.41, (Hexanes). ^{1}H NMR (300 MHz, $CDCl_3$): δ 5.89-5.75 (m, 1H), 5.04-4.92 (m, 2H), 3.26 (t, J_{H-H} = 6.9 Hz, 2H), 2.04 (q, J_{H-H} = 6.3 Hz, 2H), 1.65-1.56 (m, 2H), 1.39-1.36 (m, 2H), 1.30 (br s, 10H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 139.6, 114.5, 51.9, 34.2, 29.82, 29.8, 29.54, 29.5, 29.3, 29.2, 27.1. HRMS: found = 194.1649 [M - H] , calcd for [$C_{11}H_{21}N_3$ - H] 194.1652.

Synthesis of Compound 5c. PPh_3 (0.793 g, 3.023 mmol) was added to a solution of compound, **5b** (0.531 g, 2.748 mmol) in THF (15.0 mL) and the reaction mixture was stirred at rt for 2 h. 55 μL (~1 eq) of water was then added to the reaction mixture which was stirred at rt for another 30 min. The reaction mixture was then refluxed till no more starting material was seen on TLC. The reaction mixture was allowed to cool to rt, solvent was evaporated and the residue was directly loaded on to the silica gel column (EtOAc → DCM → 3:3:1 DCM : MeOH : Et_3N). Pure compound, **5c** (0.429 g, 92 %) was obtained as colorless viscous oil after evaporation of solvent. ^{1}H NMR (300 MHz, $CDCl_3$): δ 5.83-5.72 (m, 1H), 5.00-4.88 (m, 2H), 2.67 (t, J_{H-H} = 6.8 Hz, 2H), 2.05 (br s, 2H), 2.01 (q, J_{H-H} = 6.7 Hz, 2H), 1.45-1.32 (m, 4H), 1.26 (br s, 10H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 138.5, 113.7, 41.5, 33.4, 29.2, 29.09, 29.06, 28.7, 28.5, 26.5. HRMS: found = 170.1905 $[M + H]^+$, calcd for $[C_{11}H_{23}N + H]^+$ 170.1909.

Synthesis of Compound 5d. D-gulono-1,4-lactone (0.103 g, 0.579 mmol) was added to a solution of compound, **5c** (0.098 g, 0.579 mmol) in MeOH (5.0 mL) and the mixture was refluxed for 20 h. The mixture was allowed to cool to to rt and then in an ice bath, which led to precipitation of the crude product as a white solid. The solid was washed with ice cold methanol and any residual solvent was evaporated to yield the pure compound, **5d** (0.106 g, 53%) as a white solid. R_f = 0.61, (20% MeOH in DCM). ^{1}H NMR (300 MHz, DMSO-d_6): δ 7.83-7.79 (m, 1H), 5.83-5.70 (m, 1H), 5.44 (br s, 1H), 4.99-4.89 (m, 2H), 4.66 (br s, 1H), 4.46-4.37 (m, 3H),

3.88 (br s, 1H), 3.60-3.29 (m, 5H), 3.07-3.01 (m, 2H), 1.98 (q, J_{H-H} = 6.7 Hz, 2H), 1.38-1.31 (m, 4H), 1.22 (br s, 10 H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 173.2, 138.9, 114.7, 73.3, 72.9, 71.9, 69.5, 62.5, 38.3, 33.2, 29.1, 29.0, 28.9, 28.8, 28.6, 28.3, 26.4. HRMS: found = 370.2197 [M + Na]$^+$, calcd for [$C_{17}H_{33}NO_6$ + Na]$^+$ 370.2206.

Synthesis of Compound 5e. Recrystallized azobis(isobutyronitrile) (AIBN) (5 mg) and thiolacetic acid (0.026 g, 0.344 mmol) were added to a solution of compound, **5d** (0.030 g ,0.086 mmol) in MeOH (6.0 mL). The reaction mixture was stirred under UV source for 18 h. The solvent was removed under vacuum to yield the crude product as white solid. The solid was washed with ice cold methanol and any residual solvent evaporated to give pure compound, **5e** (0.030 g, 82%) as a white solid. R_f = 0.48, (20% MeOH in DCM). ^{1}H NMR (300 MHz, DMSO-d_6): δ 7.84-7.80 (m, 1H), 5.47 (br s, 1H), 4.67 (br s, 1H), 4.48-4.41 (m, 3H), 3.88-3.83 (m, 1H), 3.60-3.33 (m, 5H), 3.18 (br s, 1H), 3.08-3.02 (m, 2H), 2.82-2.77 (m, 1H), 2.29 (s, 3H), 1.47-1.38 (m, 4H), 1.22 (br s, 14 H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 195.7, 173.2, 73.3, 72.9, 71.8, 69.5, 68.9, 62.5, 52.5, 38.3, 35.9, 35.8, 30.6, 29.0, 28.8, 28.3, 28.1, 26.4, 25.1. HRMS: found = 446.2168 [M + Na]$^+$, calcd for [$C_{19}H_{37}NO_7S$ + Na]$^+$ 446.2188.

Synthesis of Compound 5. Argon gas was bubbled through a solution of compound, **5e** (0.031 g, 0.072 mmol) in MeOH (2.0 mL) for 10 min followed by addition of 80.5 µL solution of 1N NaOH in MeOH. The reaction mixture was stirred at rt till TLC (20% MeOH in DCM) indicated complete consumption of the starting material. The reaction mixture was cooled to 0 ºC in an ice bath and to it was added 80.5 µL solution of 1N HCl in MeOH. The mixture was stirred at 0 ºC for 20 min and then kept undisturbed at 0 ºC to allow precipitation of solid product. The supernatant was carefully decanted and compound, **5** (0.029 g, quantitative) was obtained as a white solid after evaporation of any residual solvent. ^{1}H NMR (300 MHz, DMSO-d_6): δ 7.81 (br

s, 1H), 5.46 (br s, 1H), 4.66 (br s, 1H), 4.47-4.38 (m, 3H), 3.88 (br s, 1H), 3.60-3.32 (m, 5H), 3.04 (m, 2H), 2.68-2.64 (m, 1H), 1.58-1.38 (m, 4H), 1.22 (br s, 14H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 173.2, 73.3, 72.8, 69.5, 62.5, 38.4, 29.1, 28.8, 28.6, 28.5, 27.7, 26.4. HRMS: found = 404.2068 $[M + Na]^+$, calcd for $[C_{17}H_{35}NO_6S + Na]^+$ 404.2083.

Cleaning of Glass Substrates. Substrates for the gold films were Fisher's finest premium microscope slides (Fisher Scientific, Pittsburgh, PA). Prior to gold deposition, the slides were cleaned with piranha solution. The slides were soaked in piranha solution (3 parts of 35% H_2O_2 in water and 7 parts of concentrated H_2SO_4) at 70 °C for 45 min. Warning! *Piranha solution is extremely corrosive and also has the potential for detonation if contaminated with a significant amount of oxidizable material.* After cooling, the piranha solution was poured off and the slides were rinsed 20 times with water having a resistivity of 18 MΩ cm (Millipore, Billerica, MA), followed by 10 rinses of ethanol and 10 rinses of methanol. The slides were then dried individually with a stream of nitrogen gas and stored in an 80 °C oven overnight.

Gold Deposition on Glass Substrates. Semitransparent gold films of approximately 280 Å thickness were deposited onto the piranha cleaned glass substrates with an electron beam evaporation system from Thermionics (Port Townsend, WA). A layer of titanium (approximately 70 Å thick) was applied first for adhesion of the gold film. Films were deposited at an oblique angle of 45° to the normal of the substrate. The rates of deposition were set at 0.2 Å/s for both gold and titanium. Pressure was maintained at or below 2×10^{-6} Torr throughout the deposition.

Microcontact Printing. Microcontact printing was done using polydimethylsiloxane (PDMS) stamps using slight modifications of literature reported procedures.[2] Briefly, gold slides were cut into approximately 1.0 cm × 1.0 cm pieces (gold substrates), rinsed with ethanol and then dried with a stream of nitrogen gas. PDMS stamps were dabbed with 2.0 mM solution of 1-

pentadecanethiol, dried with a stream of nitrogen gas and placed on the gold substrates to allow conformal contact for 20 seconds. The substrates were then rinsed with ethanol, dried with a stream of nitrogen gas and placed in 1.0 mM solutions of alkanethiols **3**, **4**, **5**, **6**; 0.2 mM solutions of alkanethiols **1**, **1′**and 0.2 mM solution containing 1:1 mixture of the enantiomers **1** and **1′**; 1.0 mM solutions of alkanethiols **2**, **2′** and 1.0 mM solution containing 1:1 mixture of the enantiomers **2** and **2′** for 15 h. The substrates were then taken out of the solution, rinsed with ethanol and dried with a stream of nitrogen gas before using them in mammalian cell or bacterial cell culture.

Mammalian Cell Culture. Swiss 3T3 Albino cells were cultured using general culture procedures.[3] Briefly, Swiss 3T3 Albino cells purchased from ATCC (Rockville, MD) were cultured in Dulbecco's modified Eagle's medium (DMEM; pH 7.4) supplemented with 10% fetal bovine serum (FBS), 10 μL/mL Penicillin-Streptomycin and 24 μL/mL Nystatin in a 25 mL Falcon tissue culture flask (Becton Dickson, Franklin Lakes, NJ). All cultures were kept in an incubator at 37 °C supplemented with 5% CO_2. The media was changed every 3 d. Confluent layer of 3T3 fibroblast cells were detached from culture flask by incubating with 2 mL 0.25% trypsin/0.5 mM EDTA solution for 5-10 min followed by addition of 8 mL of serum containing media.

Mammalian Cell Culture on SAM Modified Gold Substrates. SAM modified gold substrates were placed in a fresh 25 mL Falcon tissue culture flask and incubated in 7.5 mL of serum containing media supplemented with 1.0 μL Penicillin-Streptomycin and 2.4 μL Nystatin for 20 min. 2.5 mL of the cell suspension was introduced in the flask and incubated for 24 h. Media was changed after 24 h of incubation. Pictures of cell attachment and growth on the plate

were taken daily using Cannon C-5060 Wide Zoom camera and viewed from AE31 trinocular inverted microscope. Culture media was changed every 3 d.

Surface Plasmon Resonance. All surface plasmon resonance (SPR) experiments were performed on a SPRimagerII Array System (GWC Technologies, Madison, WI). 1 μL solutions of the alkanethiols **2**, **2′** and 1 μL solutions containing 1:1 mixture of the enantiomers **2** and **2′** were spotted with a micropipette on SpotReady 16 chips (GWC Technologies, Madison, WI). The chips were placed in an environment saturated with vapors of ethanol to prevent evaporation of ethanol from the spotted solutions (chips were placed in a tightly covered Petri dish, containing swabs of Kimwipes (Kimberly-Clark Corporation, Roswell, GA) saturated with ethanol and placed on the bench top at room temperature). After 15 h the chips were rinsed with ethanol and blow dried with a stream of nitrogen gas. The chips were then given a final rinse with water and gently blow dried with a stream of nitrogen gas before mounting them in a standard flow cell (GWC Technologies, Madison, WI) for the experiment. Phosphate buffered saline, 1× PBS (2.7 mM potassium chloride, 137 mM sodium chloride, 8 mM sodium phosphate dibasic, 1.48 mM potassium phosphate monobasic, pH 7.42) was used as buffer and for preparing the protein solution. The protein solution was filtered through a 0.25-μm filter before use.

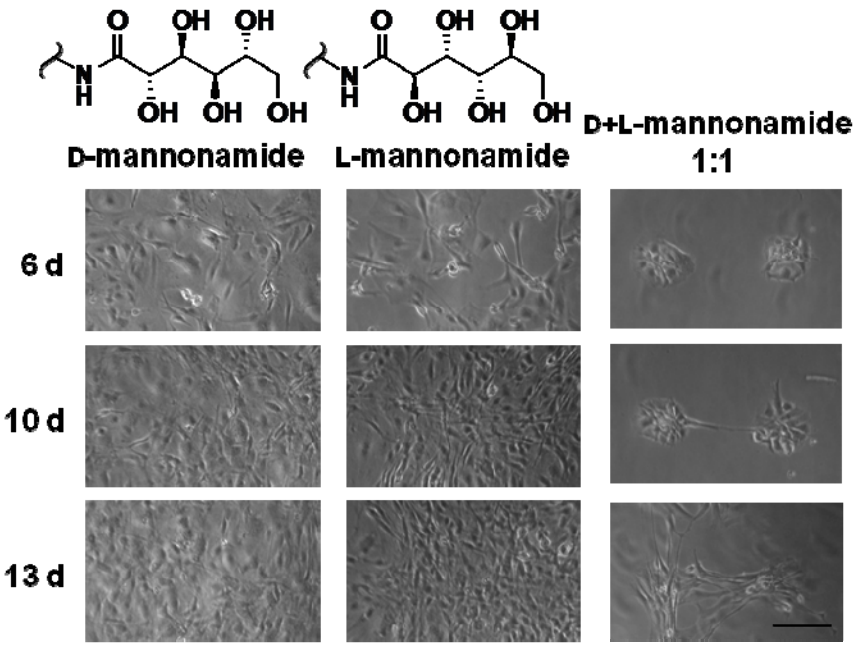
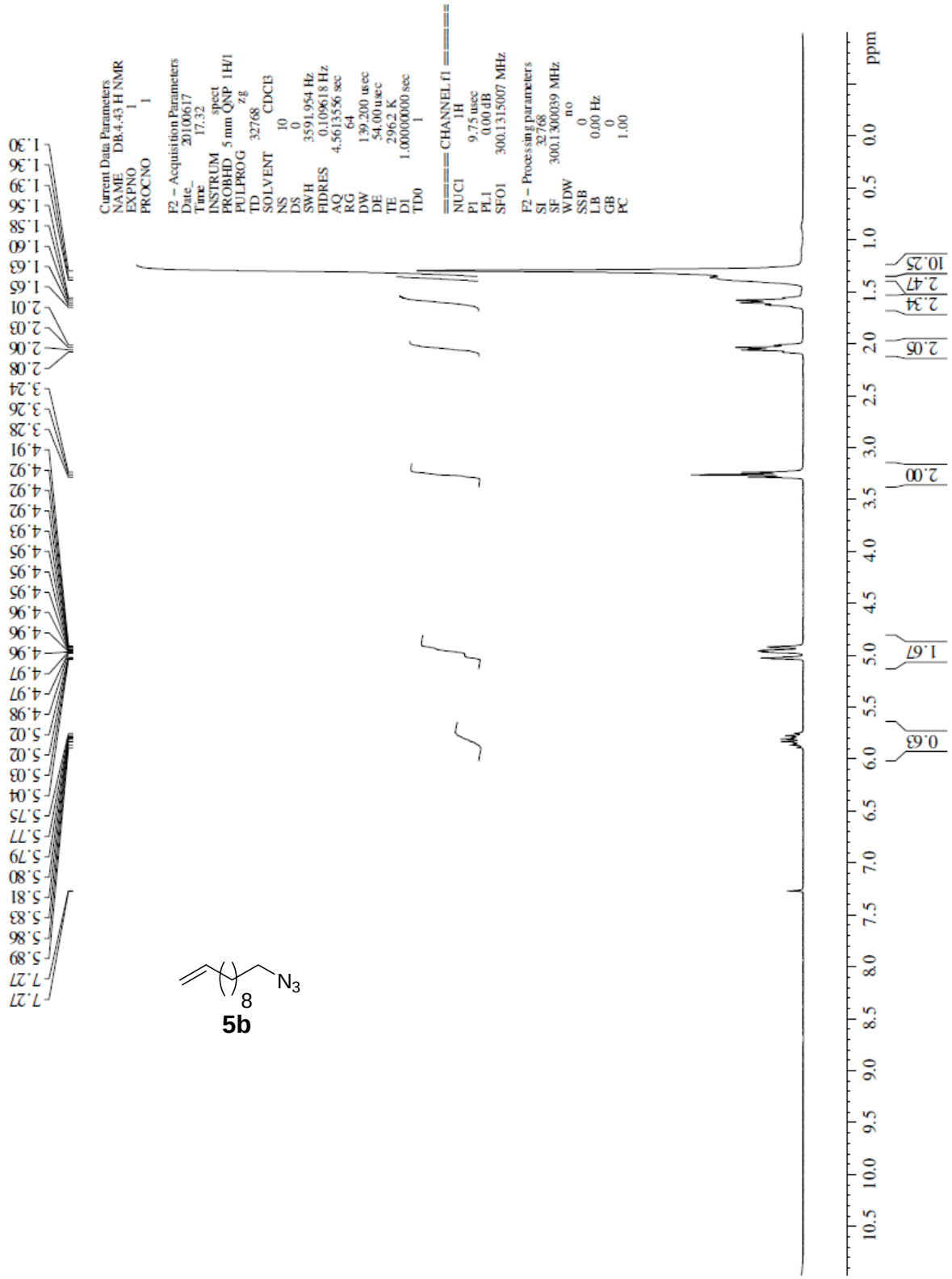


Figure S1. Optical micrographs for the adhesion of Swiss albino 3T3 fibroblast on circular patterns (135 μm in diameter) of pentadecanethiolate, surrounded by aldonamide-terminated SAMs on gold films. The aldonamide-terminated SAMs (terminal groups shown above the cell pictures) consist of D- or L-mannonamide-terminated alkanethiols, or a racemic mixture of D- and L-mannonamide-terminated alkanethiols (prepared from a solution containing 1:1 mixture of the enantiomers). The number of days the substrates were in the culture is indicated to the left. Scale bar = 152 μm.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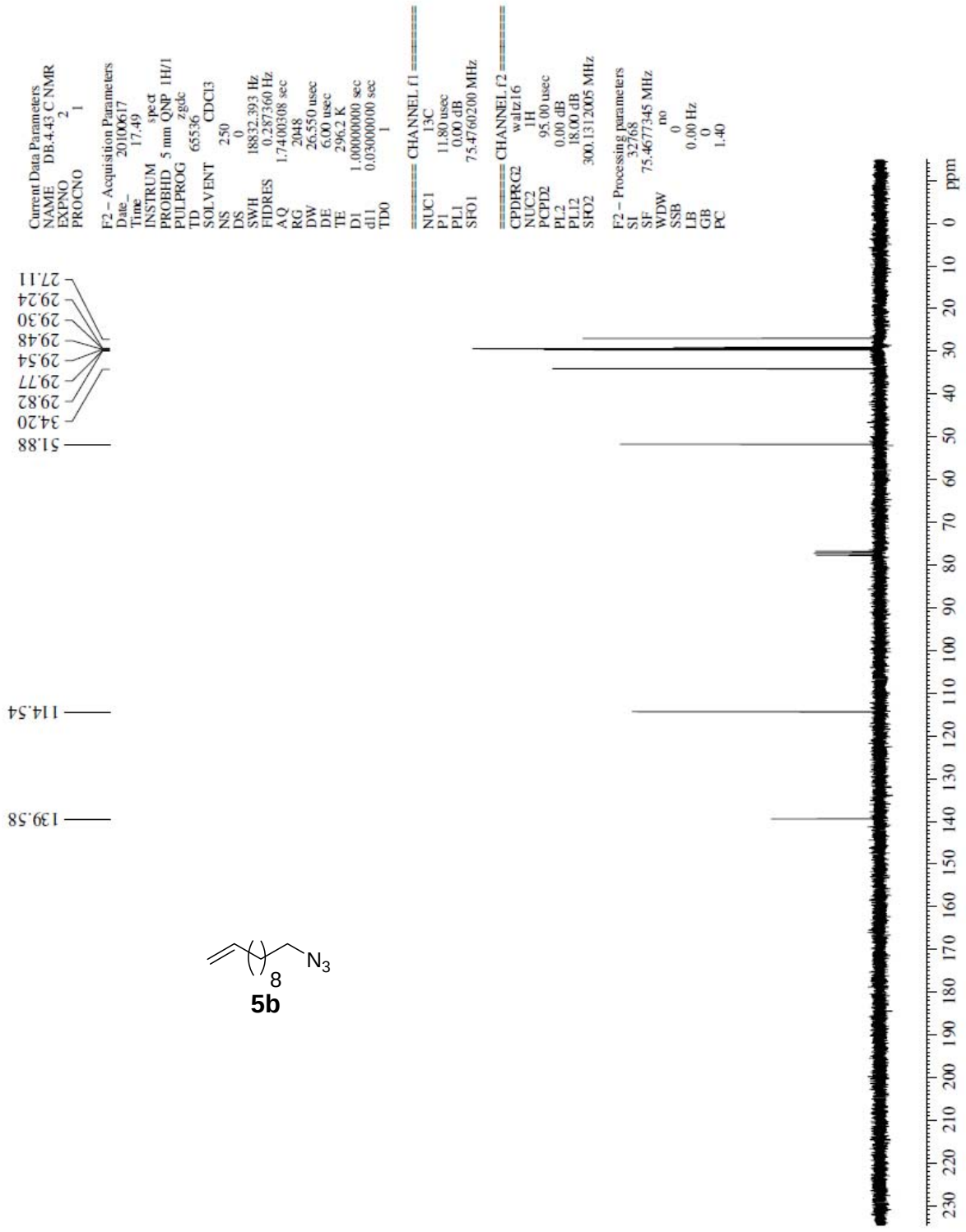
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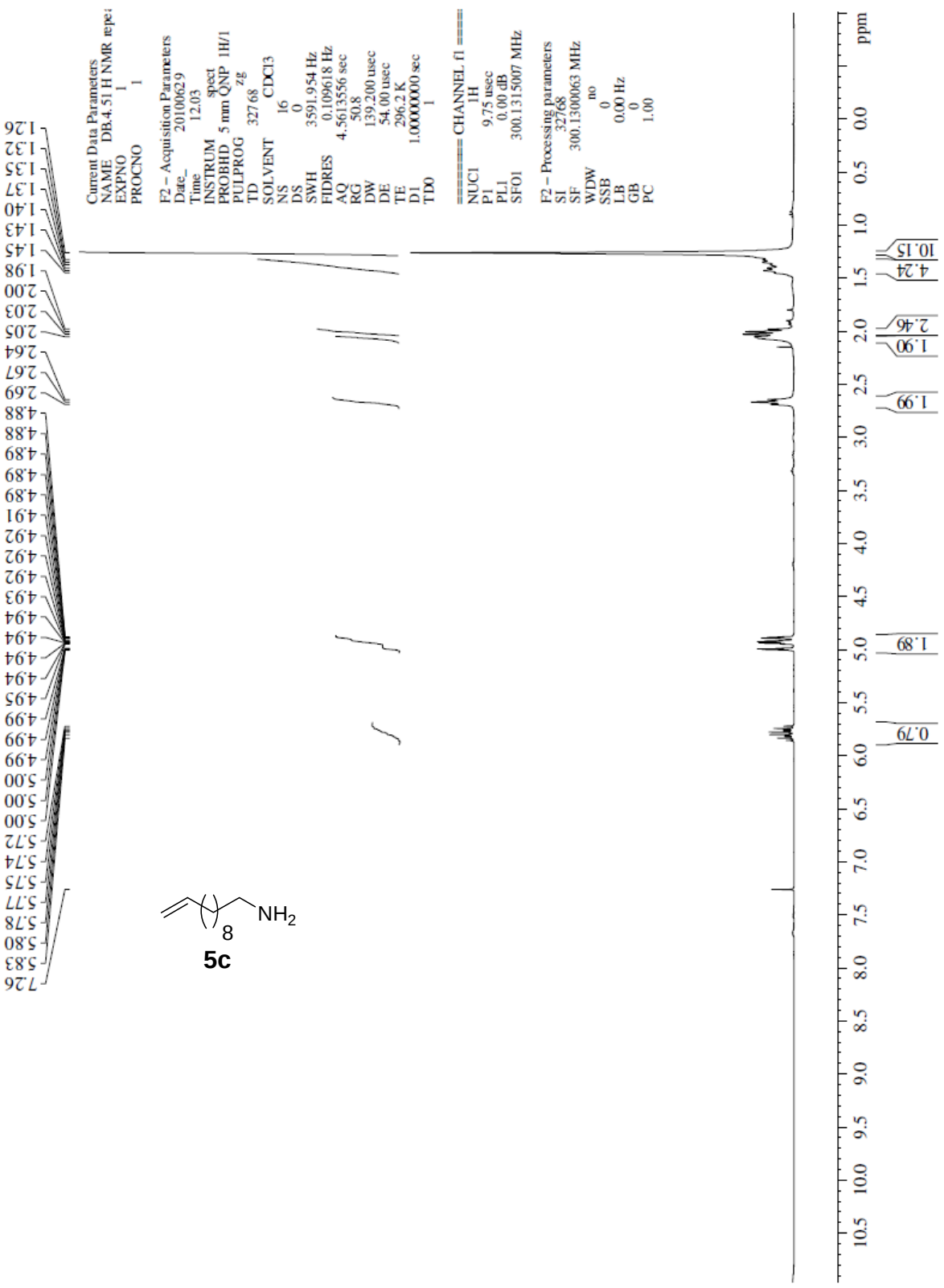
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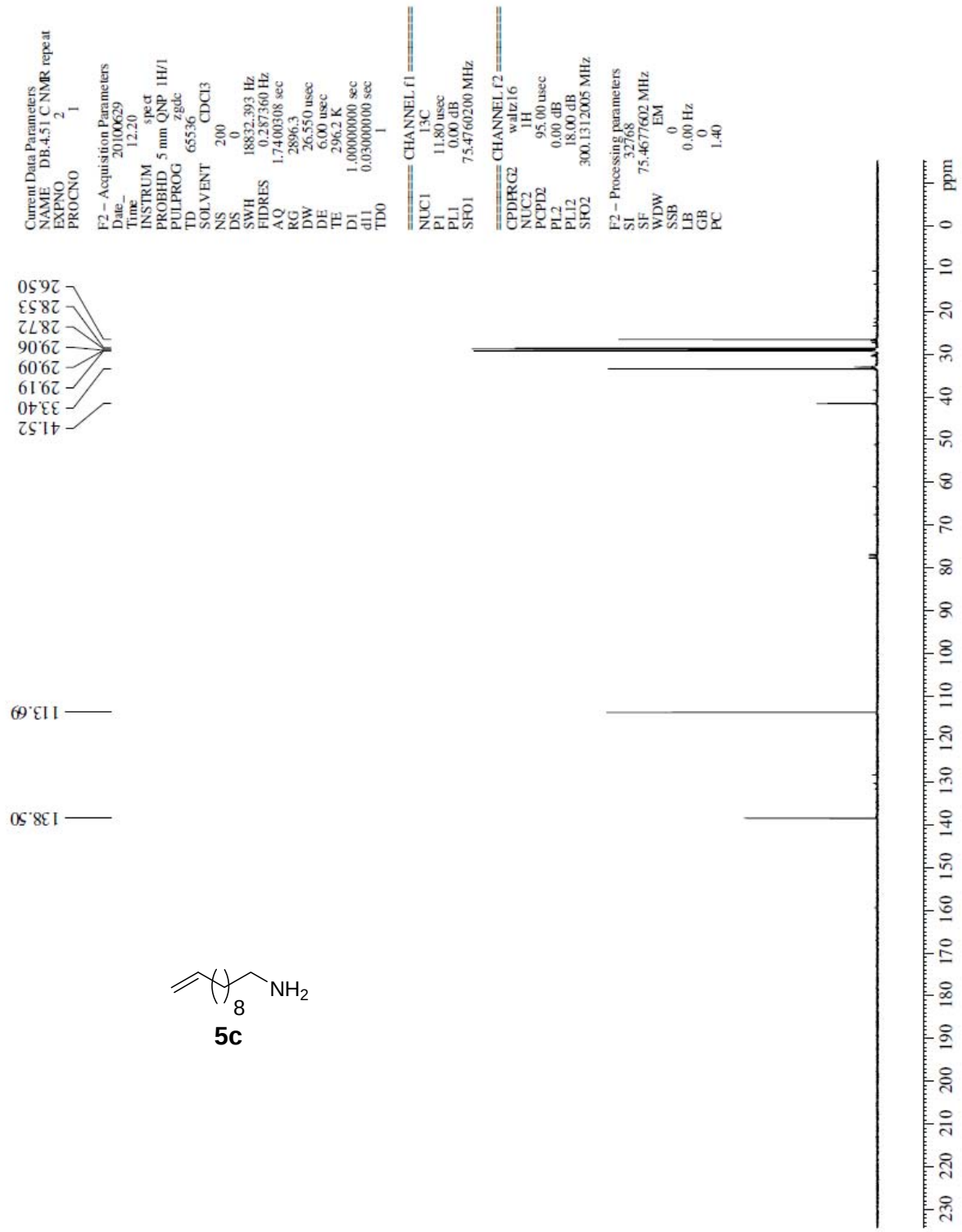
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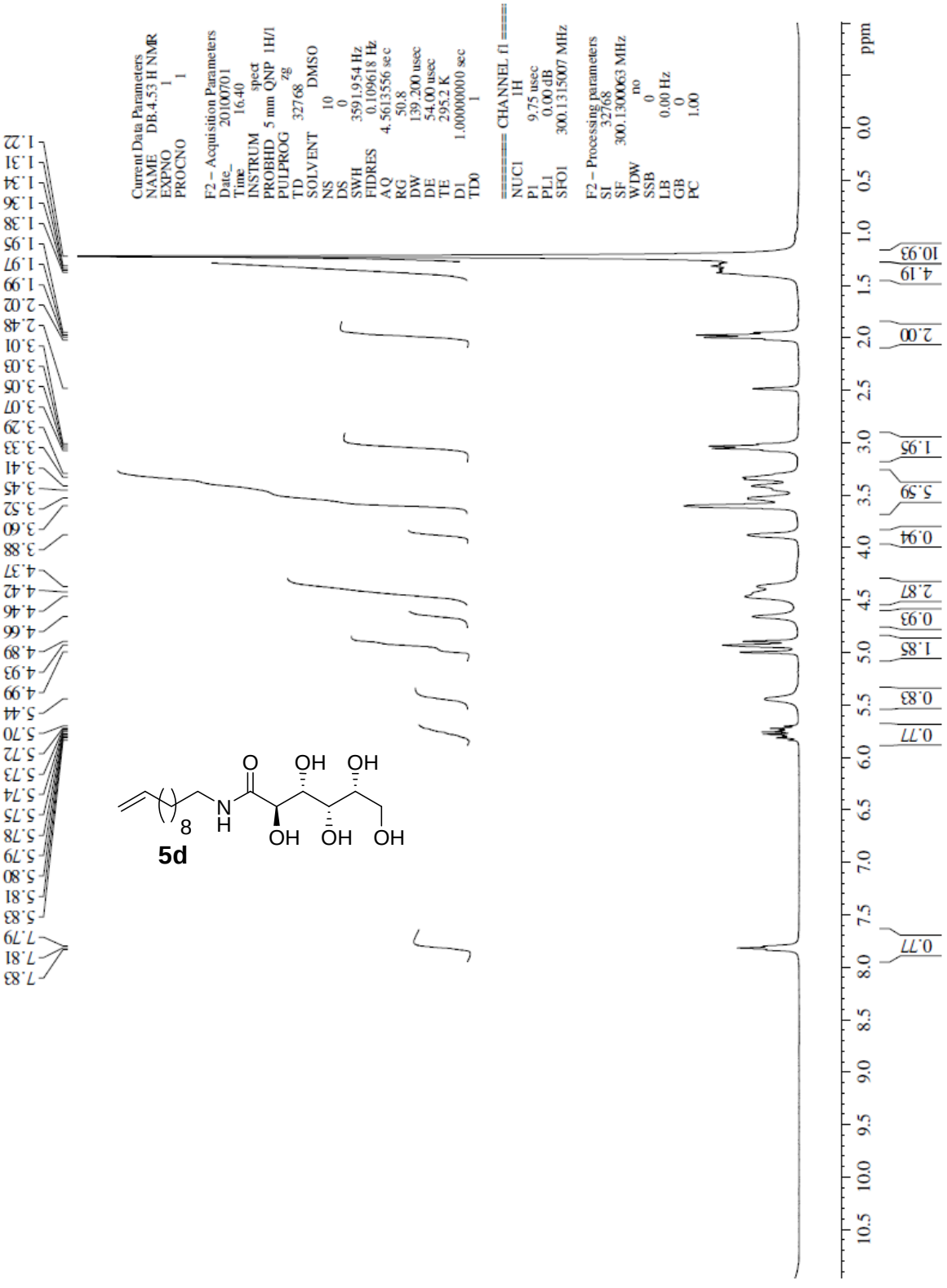
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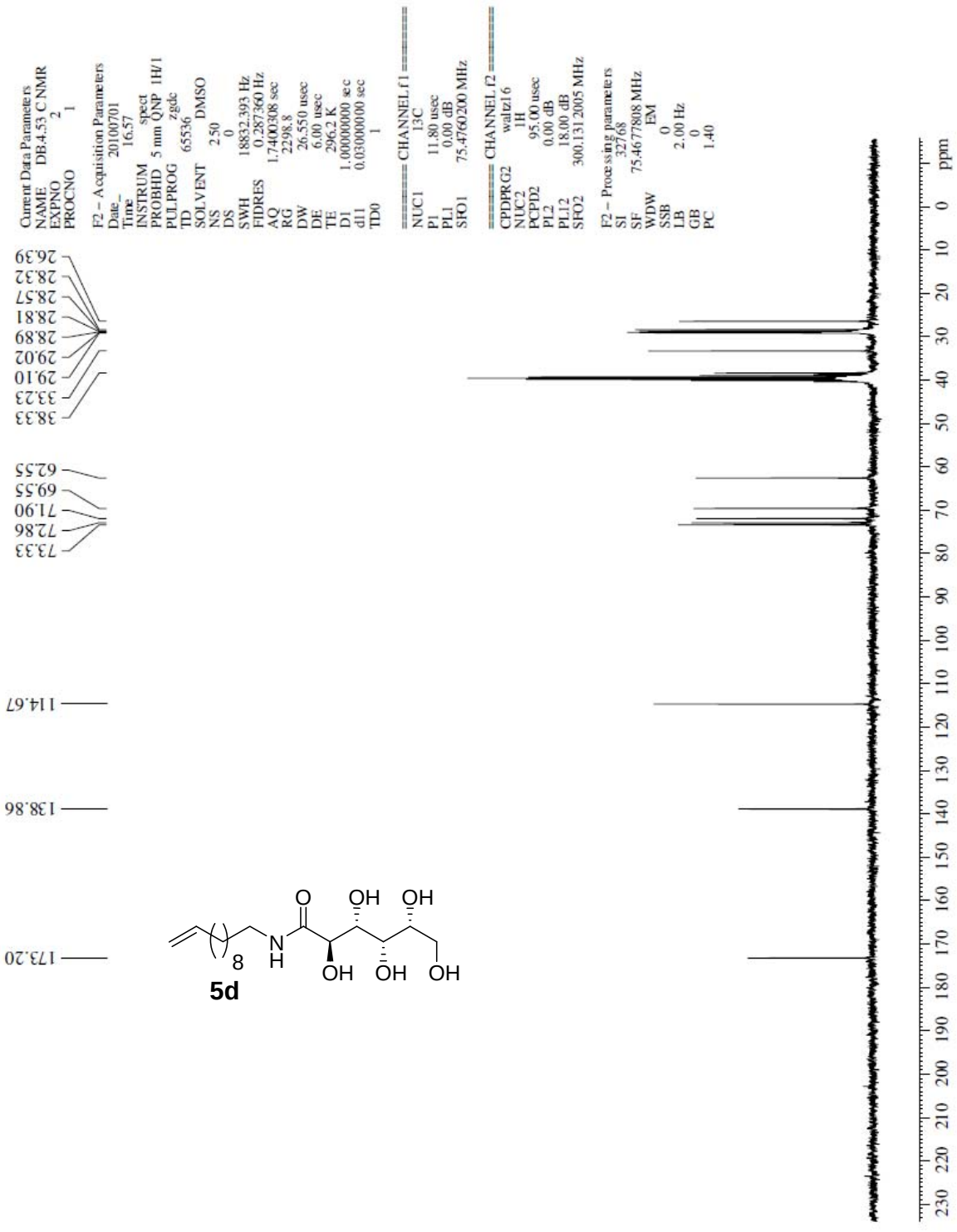
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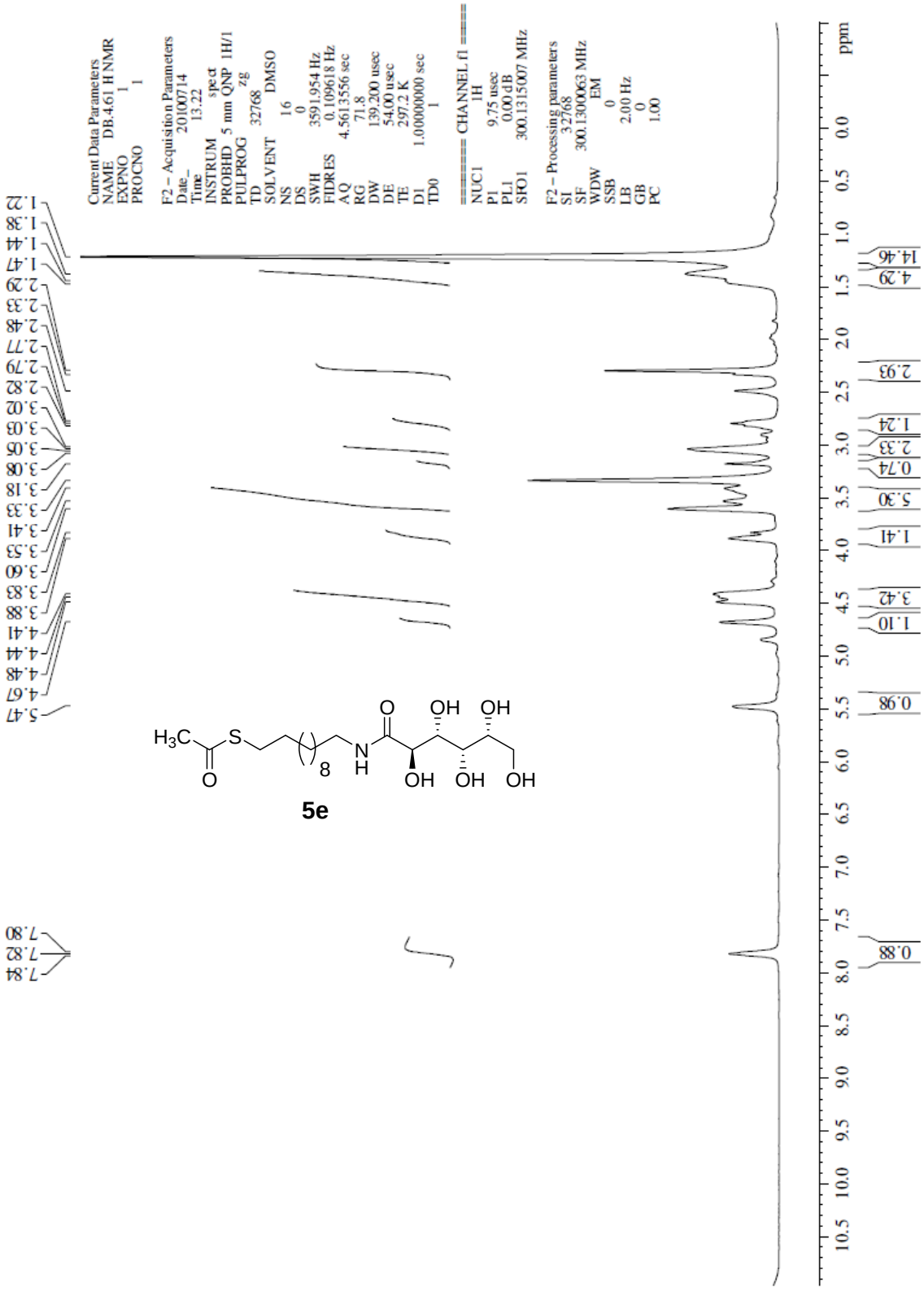
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NAME DB.4.61 H NMR
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100714
Time 13.22
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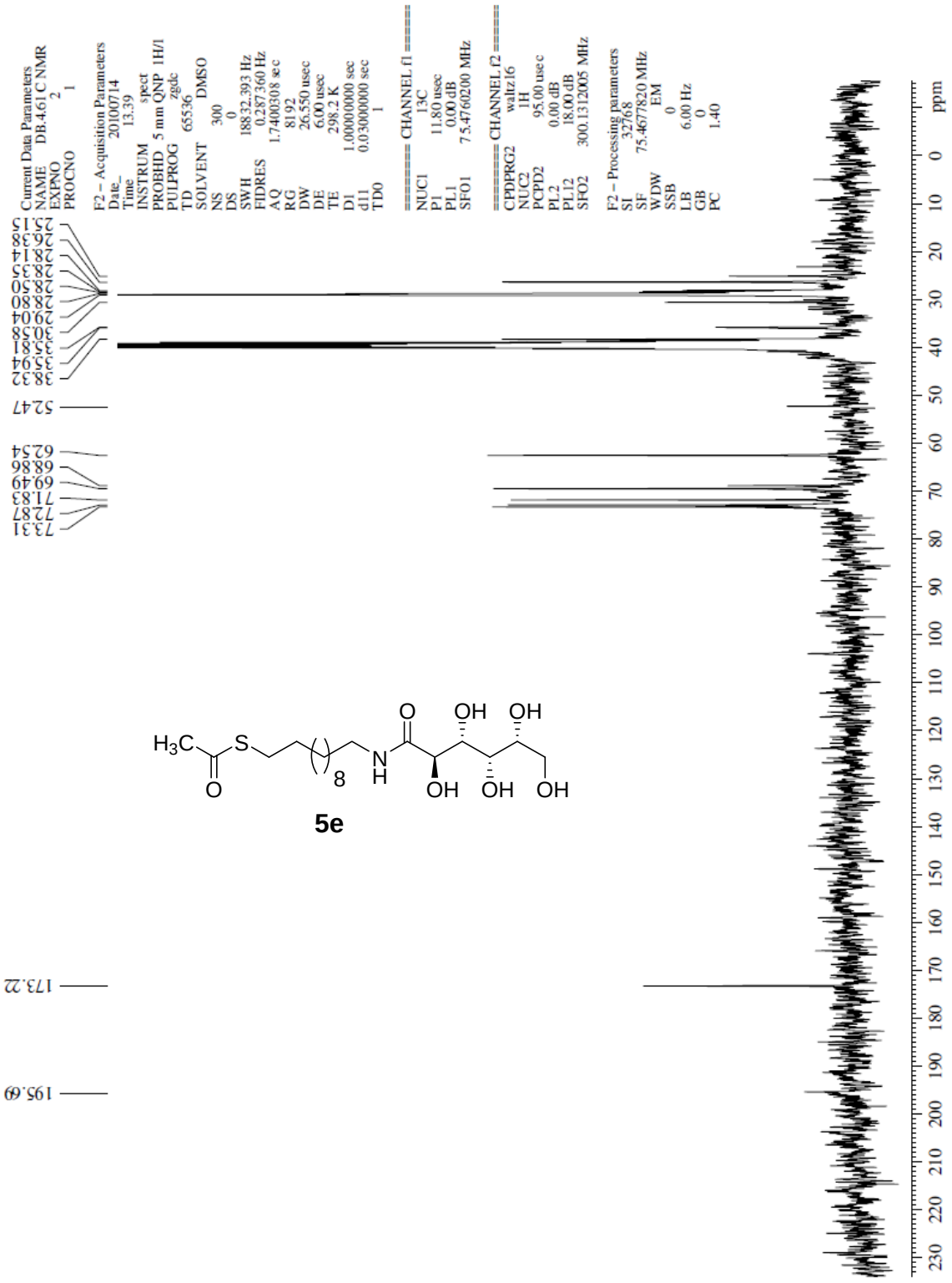
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 3591.954 Hz
FIDRES 0.109618 Hz
AQ 4.5613556 sec
RG 71.8
DW 139.200 usec
DE 54.00 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.1315007 MHz
F2 – Processing parameters
SI 32768
SF 300.1300063 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
ppm
H3C
S
O
8
N
H
O
OH
OH
OH
OH
OH
5e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EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100714
Time 13.39
INSTRUM spect
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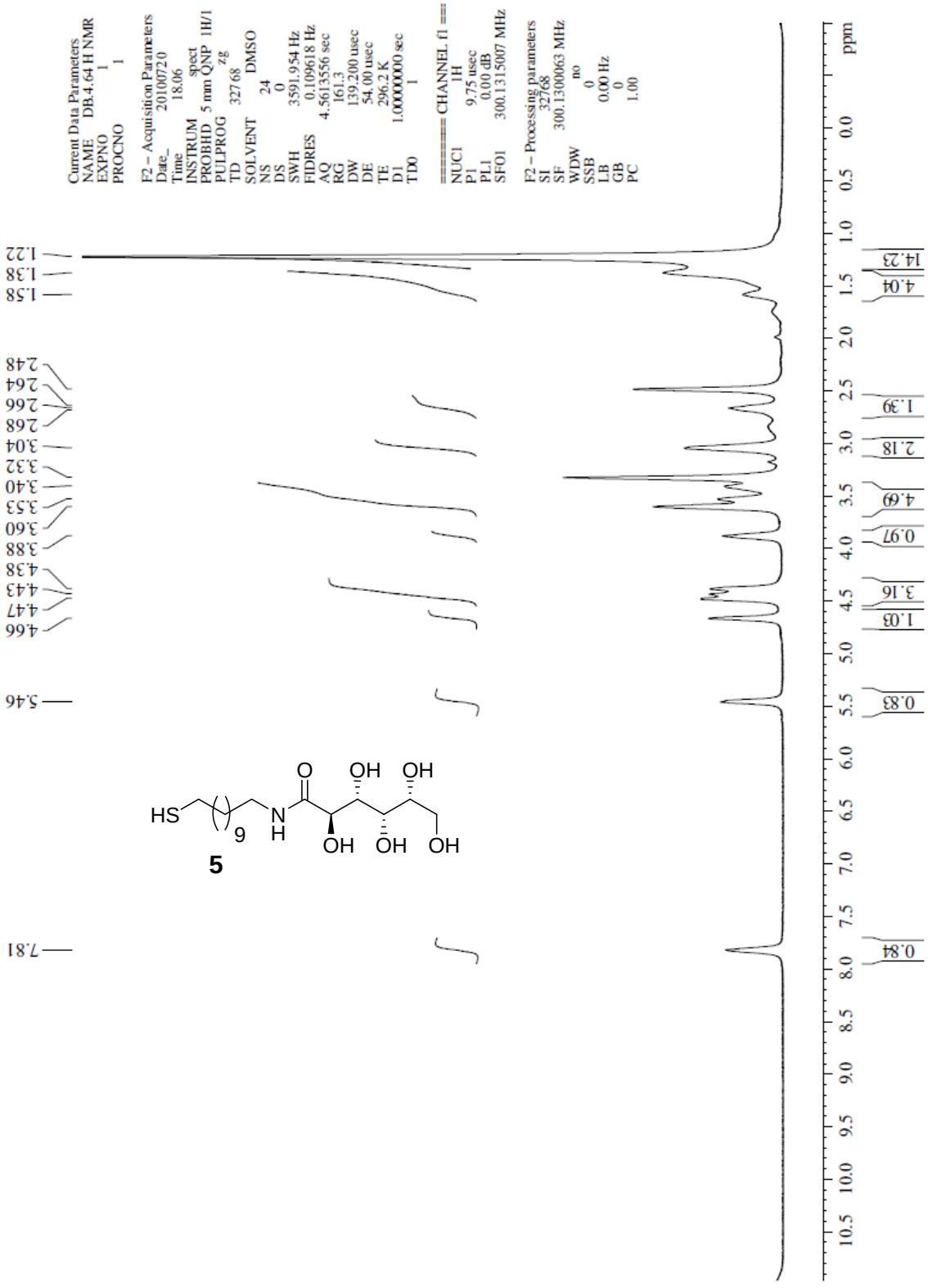
PULPROG zgdc
TD 65536
SOLVENT DMSO
NS 300
DS 0
SWH 188.32.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 8192
DW 26.550 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.80 usec
PL1 0.00 dB
SFO1 75.47760200 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 0.00 dB
PL12 18.00 dB
SFO2 300.1312005 MHz
F2 – Processing parameters
SI 32768
SF 75.4677820 MHz
WDW EM
SSB 0
LB 6.00 Hz
GB 0
PC 1.40
25.15
26.38
28.14
28.35
28.50
28.80
29.04
30.58
35.81
35.94
38.32
52.47
62.54
68.86
69.49
71.83
72.87
73.31
173.22
195.69
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
220
230

H3C
S
O
8
N
H
O
OH
OH
OH
OH
OH

5e


Current Data Parameters
NAME DB.4.64 H NMR
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100720
Time 18.06
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT DMSO
NS 24
DS 0
SWH 3591.954 Hz
FIDRES 0.109618 Hz
AQ 4.56133556 sec
RG 161.3
DW 139.200 usec
DE 54.00 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.1315007 MHz
F2 – Processing parameters
SI 32768
SF 300.1300063 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1.22
1.38
1.58
2.48
2.64
2.66
2.68
3.04
3.32
3.40
3.53
3.60
3.88
4.38
4.43
4.47
4.66
5.46
7.81
14.23
4.04
1.39
2.18
4.69
0.97
3.16
1.03
0.83
0.84
ppm
HS
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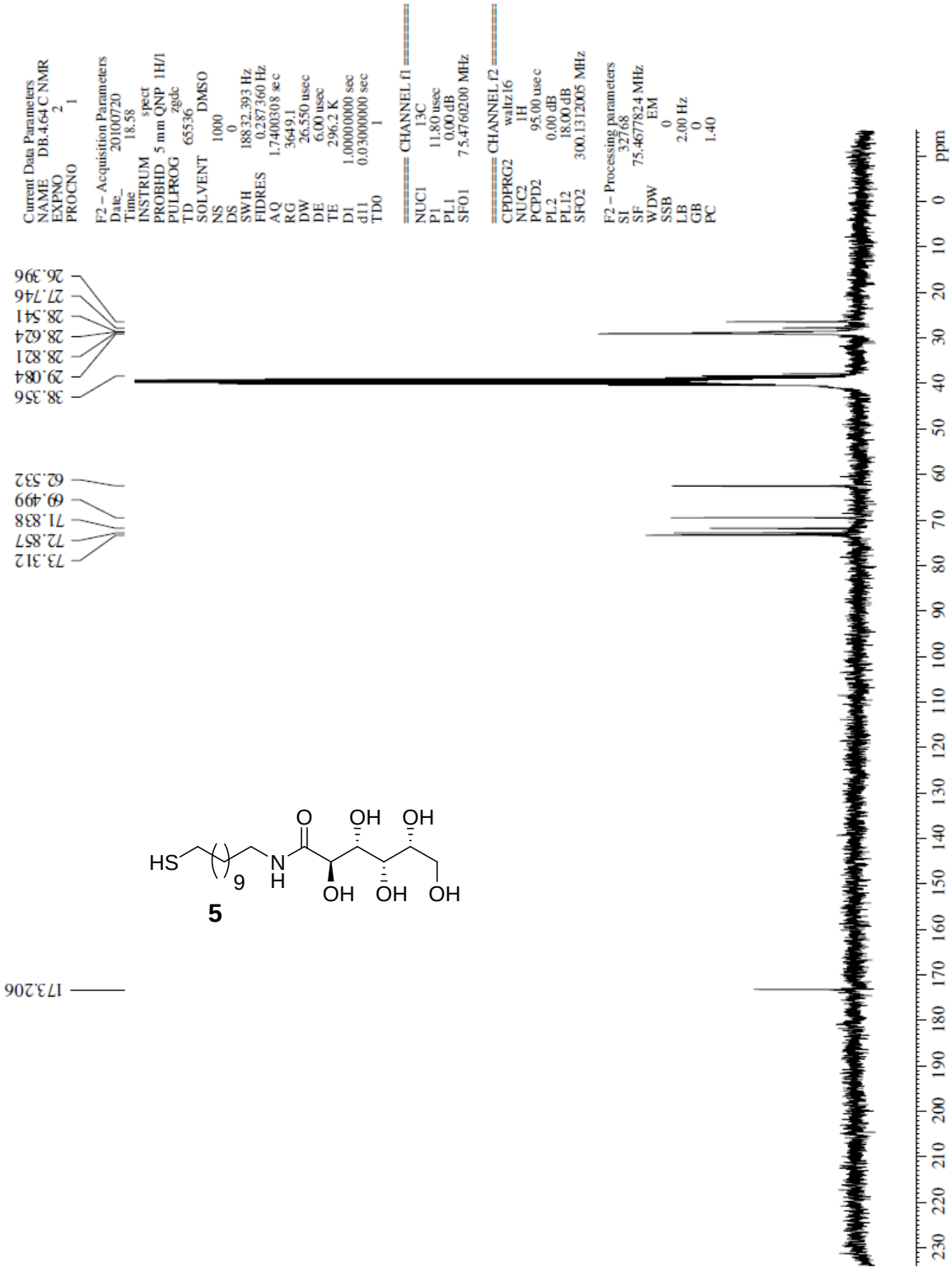
N
H
O
OH
OH
OH
OH
OH

5


Current Data Parameters
NAME DB.4.64 C NMR
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100720
Time 18.58
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgdc
TD 65536
SOLVENT DMSO
NS 1000
DS 0
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.74400308 sec
RG 3649.1
DW 26.550 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.80 usec
PL1 0.00 dB
SFO1 75.4760200 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 0.00 dB
PL12 18.00 dB
SFO2 300.1312005 MHz
F2 – Processing parameters
SI 32768
SF 75.4677824 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
38.356
29.084
28.821
28.624
28.541
27.746
26.396
73.312
72.857
71.838
69.499
62.532
173.206
ppm
HS
9
N
H
O
OH
OH
OH
OH
OH

5

References

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