A new artificial β-sheet that dimerizes through parallel β-sheet interactions

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General Procedures. Commercial solvents and reagents were used without further purification. THF was dried by filtration through alumina according to the procedure of Grubbs.[1] Solution-phase reactions were monitored by thin-layer. Column chromatography was performed on 40–63 μm silica. Solvents were removed by rotary evaporation. Residual solvents were removed under vacuum (< 0.01 mmHg). Products were dried under vacuum (< 0.01 mmHg) or by air suction through a filter funnel. High resolution mass spectra were obtained by electrospray ionization (ESI) on a Waters Micromass LCT Premier (instrument variation $\sigma < 5$ ppm). NMR spectra were recorded using 500 MHz Bruker AVANCE™, 600 MHz Bruker AVANCE™, and 800 MHz Varian UnityInova™ spectrometers. 125 MHz ^{13}C NMR spectra were obtained using a cryoprobe. ^{1}H spectra NMR were referenced with TMS (δ = 0.00 ppm) and ^{13}C NMR NMR spectra were referenced either with TMS (δ = 0.00 ppm) or DMSO-d_6 (δ = 39.53 ppm).

[1] Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Trimmers, F. J. *Organometallics* **1996**, *15*, 1518-1520.

Synthesis of artificial β-sheet 2

Artificial β-sheet **2** was synthesized according to the following schemes and procedures.

- **Typical procedure for EDCI-mediated coupling**

To a solution of amine (10.0 mmol) in dichloromethane (50 mL) was added a solution of carboxylic acid (10.0 mmol), *N*-ethyl-*N'*-(3-dimethylaminopropyl) carbodiimide hydrochloride (EDCI, 11.0 mmol), and 1-hydroxybenzotriazole (HOBt, 10.5 mmol) in dichloromethane (50 mL).[2] The solution was stirred for 2 hours, transferred to separatory funnel and washed with 1 M HCl (ca. 100 mL), saturated aqueous $NaHCO_3$ (ca. 100 mL), H_2O (ca. 100 mL), dried over $MgSO_4$, filtered, and concentrated to produce the desired amide.[3]

- **Typical procedure for HCTU-mediated coupling**

A solution of carboxylic acid (5.0 mmol) and *O*-(6-chlorobenzotriazol-1-yl)-*N*,*N*,*N'*,*N'*-tetramethyluronium hexafluorophosphate (HCTU, 5.5 mmol) in DMF (25 mL) was added to a solution of amine or amine trifluoroacetate (5.0 mmol) and either 2,4,6-collidine or diisopropylethylamine (DIPEA) (25 mmol) in DMF (25 mL). The solution was stirred for 2 hours and concentrated under vacuum. The residue was dissolved in dichloromethane[4] (200 mL), and the solution was washed 1 M HCl (3 × ca. 100 mL), saturated aqueous $NaHCO_3$ (3 × ca. 100 mL), water (3 × ca. 100 mL), dried over $MgSO_4$, filtered, and concentrated.[3]

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[2] If either the amine or the carboxylic acid component does not dissolve, *N,N*-dimethylformamide (DMF) is added as a co-solvent or used instead of dichloromethane. Upon completion of the reaction, DMF is removed under vacuum and the residue is dissolved in dichloromethane. Alternatively, DMF may be removed by pouring the solution into water to precipitate the product.

[3] The product is purified either by trituration with acetonitrile or by column chromatography.

[4] If the residue is not soluble in dichloromethane, it is purified by trituration with acetonitrile or by column chromatography.

- **Typical procedure for Boc removal with TFA**

Boc-protected amine (10.0 mmol) was dissolved in a mixture of dichloromethane (50 mL), trifluoroacetic acid (TFA, 50 mL) and trisopropylsilane (TiPS, 0.25 mL). After 0.5 h, the solution was concentrated and the residue was triturated with diethyl ether to yield the amine trifluoroacetate salt as a white powder.

- **Typical procedure for Fmoc removal with piperidine**

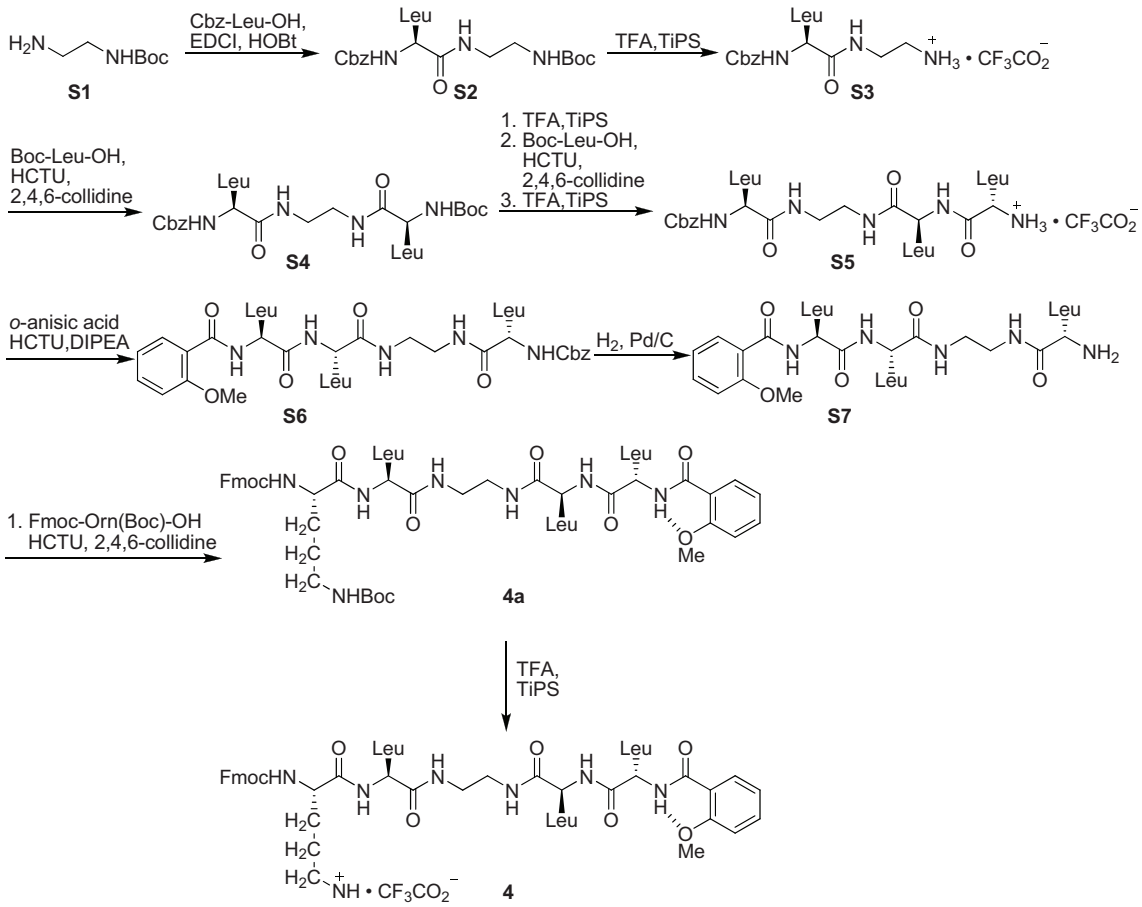
Fmoc-protected amine (1.0 mmol) was dissolved in a mixture of DMF and piperidine (50 mL, 4:1). After 30 min, the solution was concentrated under vacuum and the residue was triturated with hexanes (3 × ca. 50 mL).[5]

- **Typical procedure for Cbz removal with H_2-Pd/C**

A 500-mL, three-necked, round-bottomed flask equipped with a nitrogen inlet adapter, a glass stopper, a gas inlet adapter fitted with a hydrogen balloon, and a magnetic stirring bar was charged with 250 mL of tetrahydrofuran, 100 mL of methanol, and Cbz-protected amine (10.0 mmol). 10% Pd/C (0.5 g) was added to the reaction mixture under a nitrogen flow. The flask was evacuated and filled with hydrogen and then maintained under the hydrogen atmosphere for 2 h. The reaction mixture was filtered through celite, the celite was washed with ethyl acetate (100 mL), and the combined solution was concentrated under vacuum to yield the free amine.

[5] Free amines are of sufficient purity for further use after trituration with hexanes. Further purification can be achieved by preparative RP HPLC or by column chromatography.

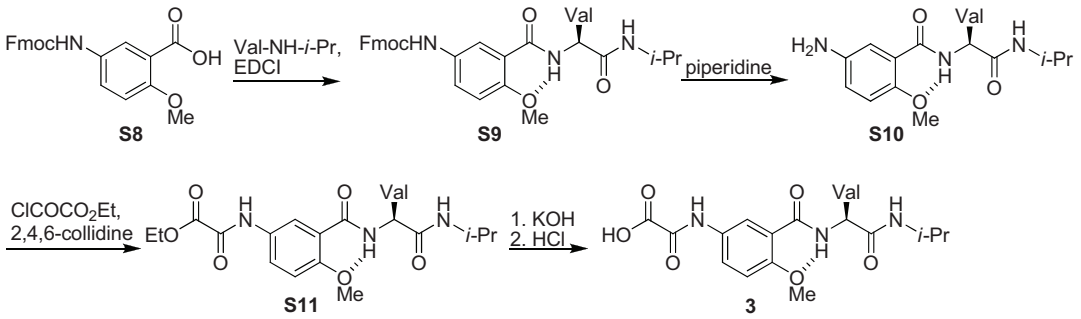
- **Synthesis of peptide 4a**



Peptide **4a**: ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.36 (d, J = 7.8 Hz, 1 H), 7.99 (d, J = 8.2 Hz, 1 H), 7.97-7.91 (m, 2 H), 7.89 (d, J = 7.5 Hz, 2 H), 7.82 (d, J = 8.1 Hz, 1 H), 7.76 (d, J = 7.6 Hz, 1 H), 7.74-7.68 (m, 2 H), 7.51-7.47 (m, 2 H), 7.41 (t, J = 8.1 Hz, 2 H), 7.32 (t, J = 7.5 Hz, 2 H), 7.16 (d, J = 8.7 Hz, 1 H), 7.05 (t, J = 7.7 Hz, 1 H), 6.79 (appar t, J = 5.5 Hz, 1 H), 4.53 (q, J = 7.1 Hz, 1 H), 4.30-4.20 (m, 5 H), 3.98 (td, J = 9.0, 5.0 Hz, 1 H), 3.87 (s, 3 H), 3.14-3.08 (m, 2 H), 3.07-3.01 (m, 2 H), 2.94-2.87 (m, 2 H), 1.67-1.52 (m, 6 H), 1.50-1.52 (m, 7 H), 1.37 (s, 9 H), 0.91-0.79 (m, 18 H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 172.0, 171.9, 171.6, 164.6, 157.1, 156.0, 155.6, 143.9, 143.7, 140.7, 132.5, 130.5, 127.6, 127.1, 125.3, 122.5, 120.7, 120.1, 112.2, 77.4,

65.5, 56.1, 54.4, 51.8, 51.0, 46.7, 41.2, 41.0, 38.2, 29.2, 28.3, 26.2, 24.4, 24.2, 24.1, 23.2, 23.0, 21.8, 21.72, 21.67; MS (ESI) *m/z* for $C_{53}H_{75}N_7O_{10}Na$ $[M + Na]^+$ calcd 992.5, found 992.2.

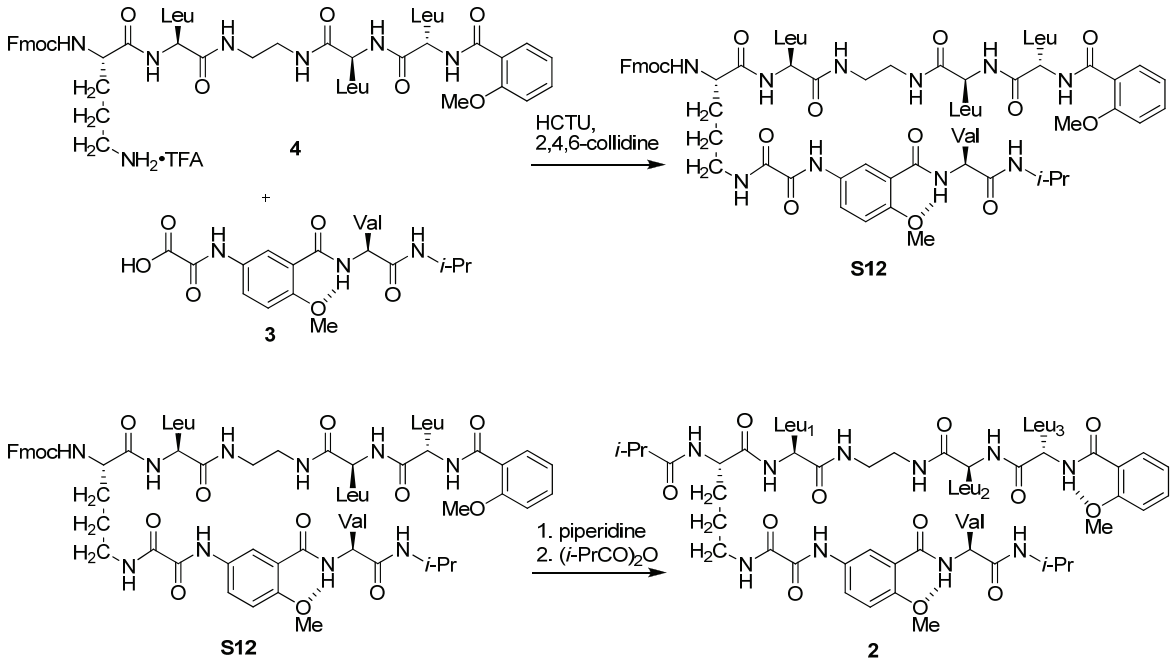
- **Synthesis of molecular template 3**



A solution of aniline S10 (0.80 g, 2.61 mmol) and 2,4,6-collidine (1.0 mL, 6.5 mmol) in dichloromethane (50 mL) was cooled to 0° C with an ice-bath. Ethyl chlorooxoacetate (0.32 mL, 2.87 mmol) was added in one portion. The solution was stirred for 40 minutes and washed with 1 M HCl (3 × ca. 50 mL). The solution was dried over $MgSO_4$, filtered, and concentrated to give 0.95 g (90%) of ethyl ester S11 as a white solid, which was used without further purification. To a solution of S11 (0.90 g, 2.2 mmol) in ethanol (40 mL) was added solution of KOH (0.25 g, 4.46 mmol) in H_2O (10 mL). After 15 min, the solution was acidified with 1 M HCl (100 mL) and concentrated under vacuum. The residue was washed with H_2O and dried under vacuum to afford 0.65 g (70%) of **3** as white solid, which was used in the next step without further purification. An analytical sample was purified by RP HPLC. ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.74 (s, 1 H), 8.37 (d, J = 8.3 Hz, 1 H), 8.28 (d, J = 2.8 Hz, 1 H), 7.97 (d, J = 7.6 Hz, 1 H), 7.85 (dd, J = 8.9, 2.7 Hz 1 H), 7.19 (d, J = 9.3 Hz, 1 H), 4.39 (dd, J = 8.7, 6.0 Hz, 1 H), 3.92 (s, 3 H), 3.86 (octet, J = 7.0 Hz, 1 H), 2.01 (octet, J = 6.7 Hz, 1 H), 1.07 (d, J = 6.4 Hz, 3 H), 1.06 (d,

J = 6.4 Hz, 3 H), 0.90 (d, J = 6.9 Hz, 3 H), 0.88 (d, J = 6.9 Hz, 3 H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 169.4, 163.6, 162.1, 156.6, 154.0, 131.0, 124.7, 123.2, 121.9, 112.7, 57.6, 56.5, 40.5, 31.5, 22.4, 22.2, 19.2, 18.0; HRMS (ESI) m/z for $C_{18}H_{25}N_3O_6Na$ [M + Na]$^+$ calcd 402.1641, found 402.1631.

- **Synthesis of artificial β-sheet 2 from building blocks 3 and 4.**



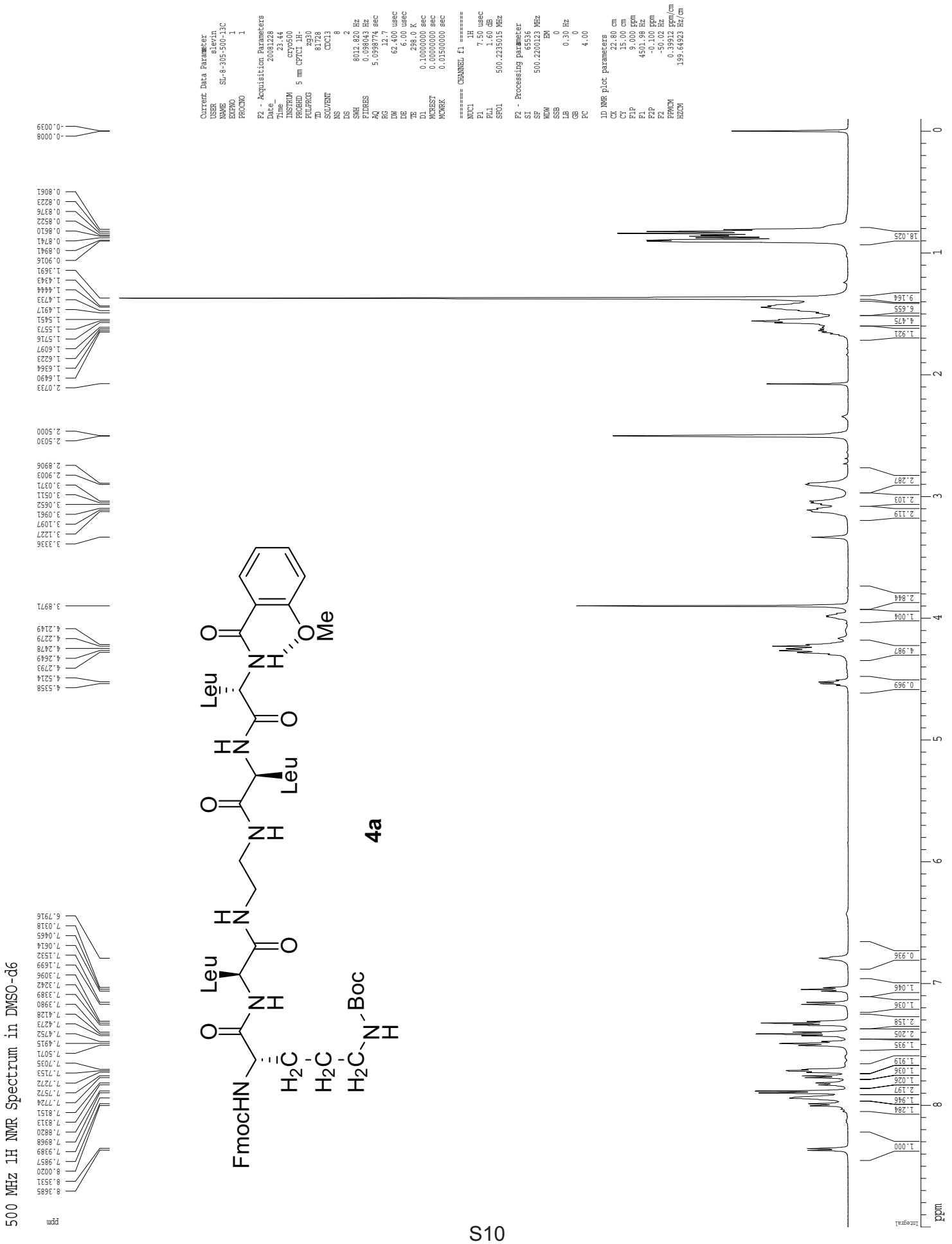
Fmoc-protected amine S12 (0.20 g, 0.16 mmol) was dissolved in a mixture of DMF and piperidine (10 mL, 4:1). After 30 min, the solution was concentrated under vacuum and the residue was triturated with hexanes (3 × ca. 25 mL). The deprotected amine was dissolved in a mixture of DMF and DIPEA (26 mL, 25:1). Isobutyric anhydride (0.03 mL, 18 mmol) was added to the solution. After 1 h, the solvent was removed under vacuum and the residue was triturated with acetronitrile to yield 0.08g (45%) of artificial β-sheet **2** as a white powder: ^{1}H NMR (600

MHz, DMSO-d_6) δ 10.63 (s, 1 H), 8.95 (t, J = 6.0 Hz, 1 H), 8.37-8.34 (m, 3 H), 7.98 (d, J = 8.2 Hz, 1 H), 7.95 (d, J = 7.8 Hz, 1 H), 7.93-7.87 (m, 4 H), 7.76 (dd, J = 7.8, 1.6 Hz, 1 H), 7.72 (d, J = 8.2 Hz, 1 H), 7.47 (td, J = 8.0, 1.5 Hz, 1 H), 7.19 (d, J = 9.0 Hz, 1 H), 7.16 (d, J = 8.4 Hz, 1 H), 7.05 (t, J = 7.5 Hz, 1 H), 4.53 (q, J = 7.7 Hz, 1 H), 4.39 (dd, J = 8.5, 6.3 Hz, 1 H), 4.27 (td, J = 8.8, 6.6 Hz, 1 H), 4.24-4.19 (m, 2 H), 3.92 (s, 3 H), 3.90 (s, 3 H), 3.84 (appar octet, J = 6.8 Hz, 1 H), 3.18 (appar q, 2 H), 3.13-3.08 (m, 2 H), 3.07-3.01 (m, 2 H), 2.45 (septet, J = 6.6 Hz, 1 H), 2.01 (appar octet, J = 6.6 Hz, 1 H), 1.69-1.61 (m, 2 H), 1.58-1.40 (m, 11 H), 1.07 (d, J = 5.3 Hz, 3 H), 1.05 (d, J = 5.3 Hz, 3 H), 1.00 (d, J = 6.6 Hz, 3 H), 0.98 (d, J = 6.9 Hz, 3 H), 0.91-0.88 (m, 12 H), 0.88-0.85 (m, 6 H), 0.84-0.81 (m, 6 H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 176.3, 171.84, 171.78, 171.5, 171.4, 169.3, 164.5, 163.5, 159.8, 158.4, 156.9, 153.8, 132.4, 130.9, 130.4, 124.7, 123.1, 122.4, 121.8, 120.5, 112.6, 112.1, 57.5, 56.4, 55.9, 52.1, 51.6, 50.8, 41.07, 41.06, 40.8, 40.3, 38.7, 38.0, 33.6, 31.3, 28.9, 25.3, 24.3, 24.1, 24.0, 23.0, 22.90, 22.86, 22.3, 22.1, 21.7, 21.55, 21.53, 19.6, 19.2, 19.1, 17.9; MS (ESI) *m/z* for $C_{55}H_{86}N_{10}O_{12}Na$ $[M + Na]^+$ calcd 1101.6, found 1101.3.

^{1}H NMR (800 MHz, $CDCl_3$, 4 mM, 268 K)

Amino Acid	H_α Shift, ppm	NH Shift, ppm
Orn	5.05	7.79
Leu$_1$	4.74	8.15
Leu$_2$	5.07	8.64
Leu$_3$	5.34	8.62
Val	4.63	8.77

Proton	Shift, ppm
Ar-NH	10.91
***i*-Pr-NH**	8.10
NH$_{en-a}$	8.59
NH$_{en-b}$	8.53
Orn$_{\delta NH}$	7.67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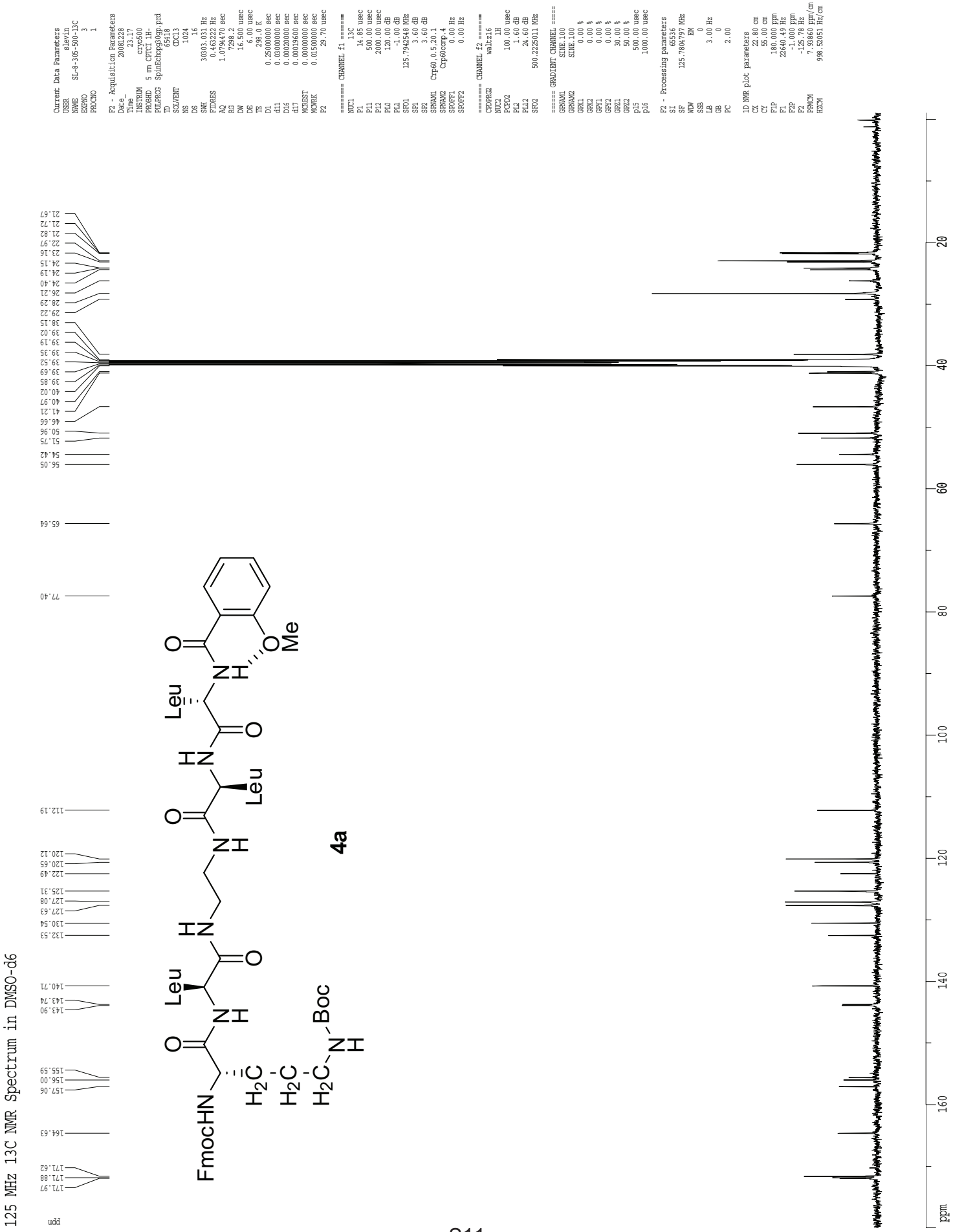
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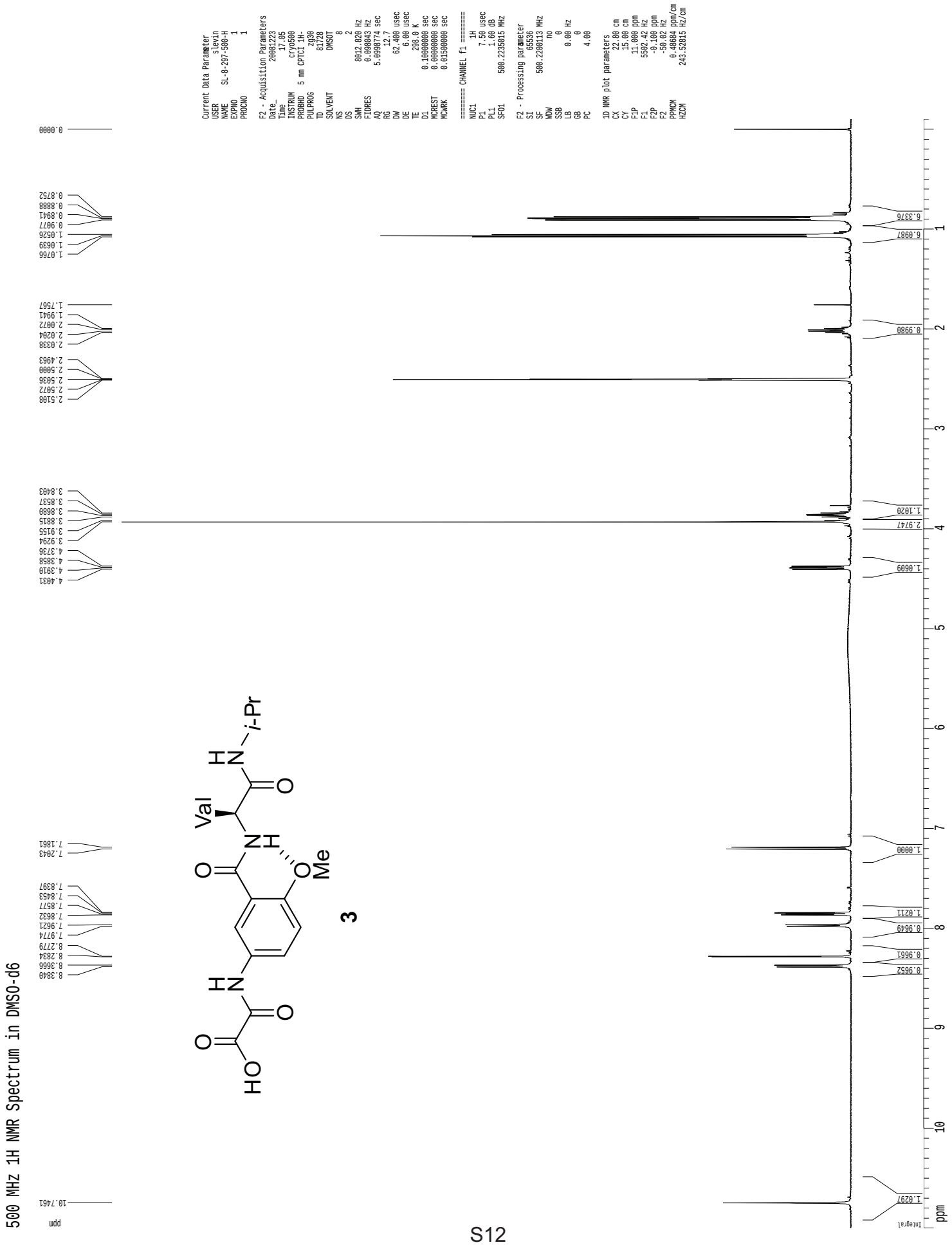
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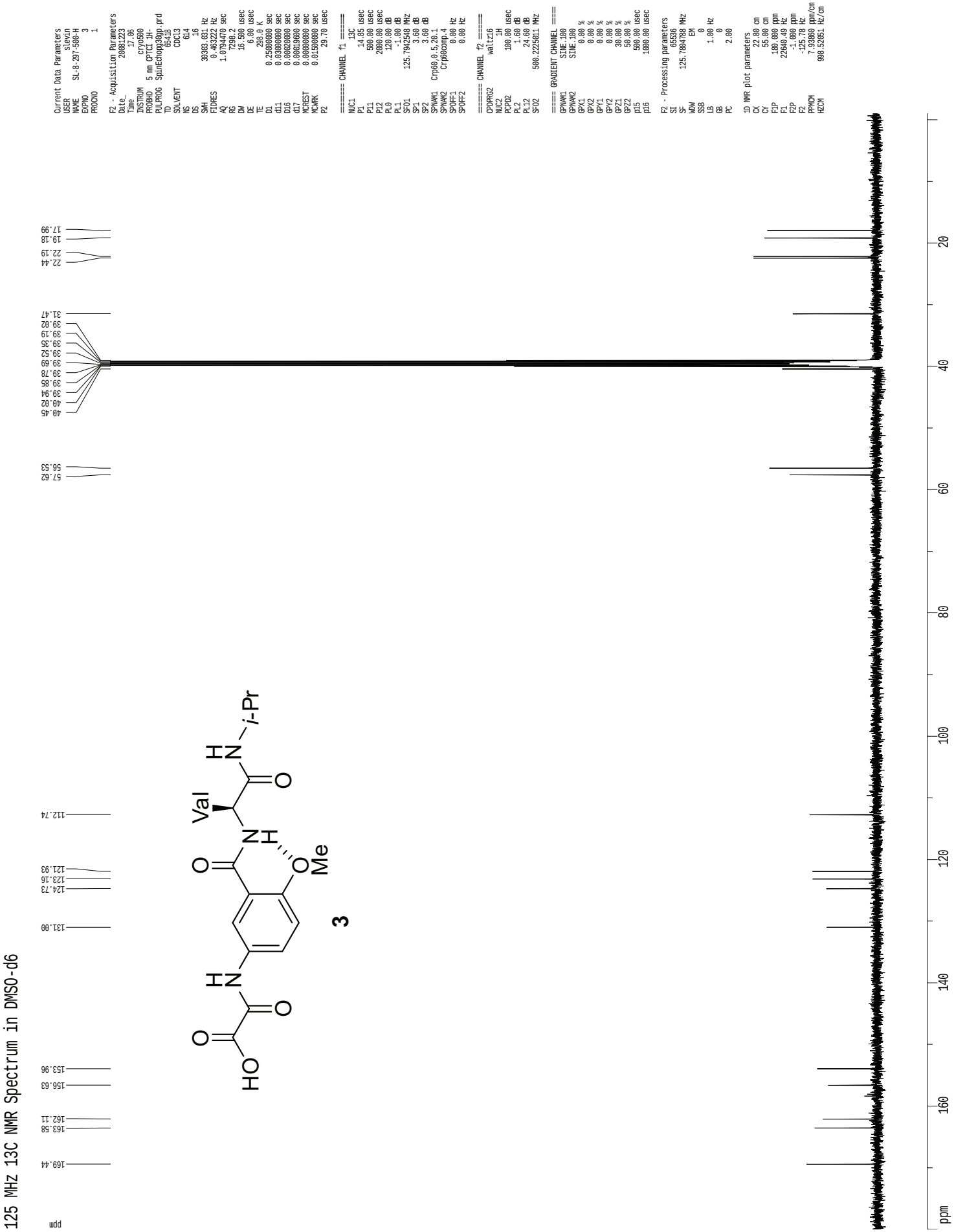
Me
3
ppm
Integral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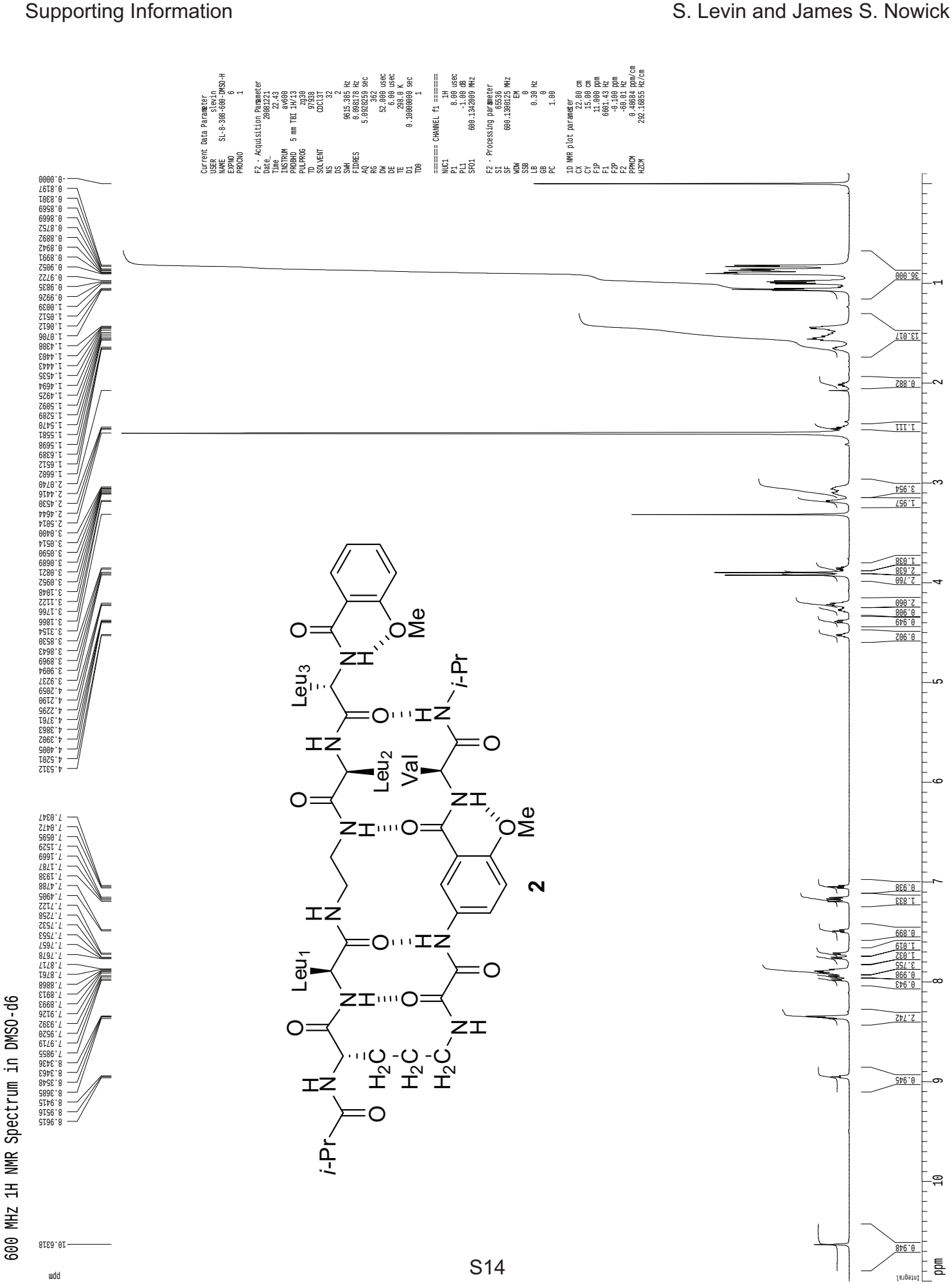
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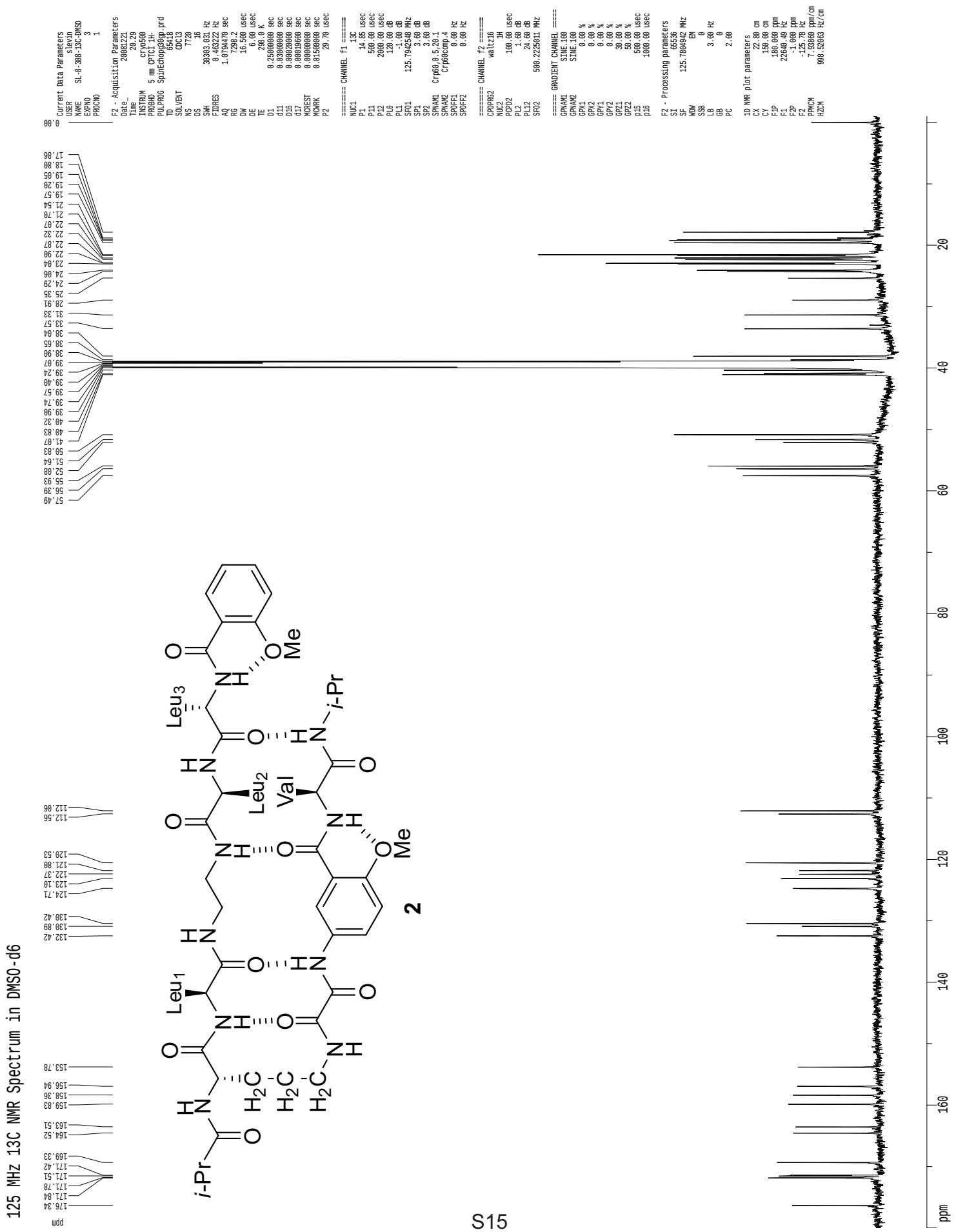
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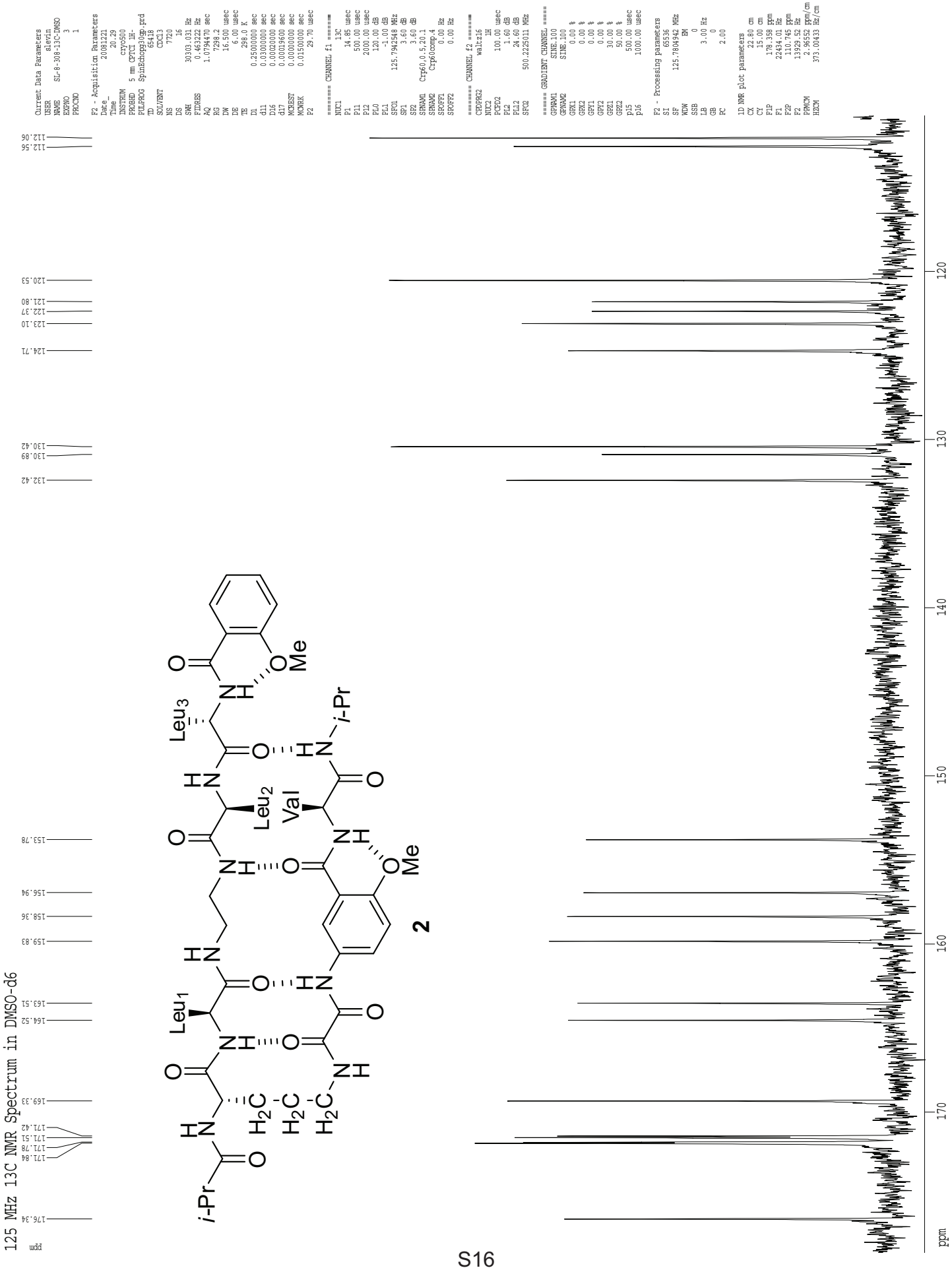
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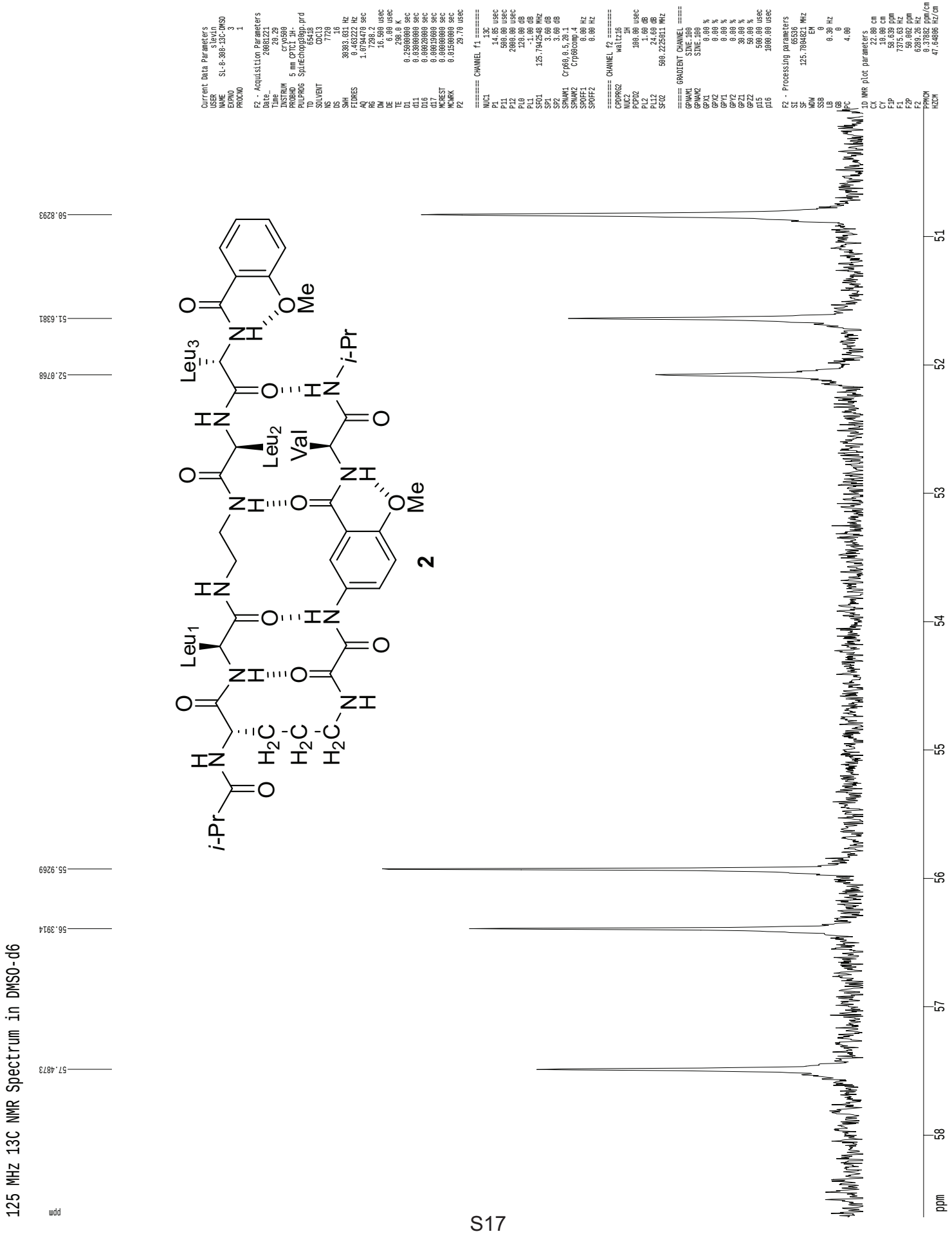
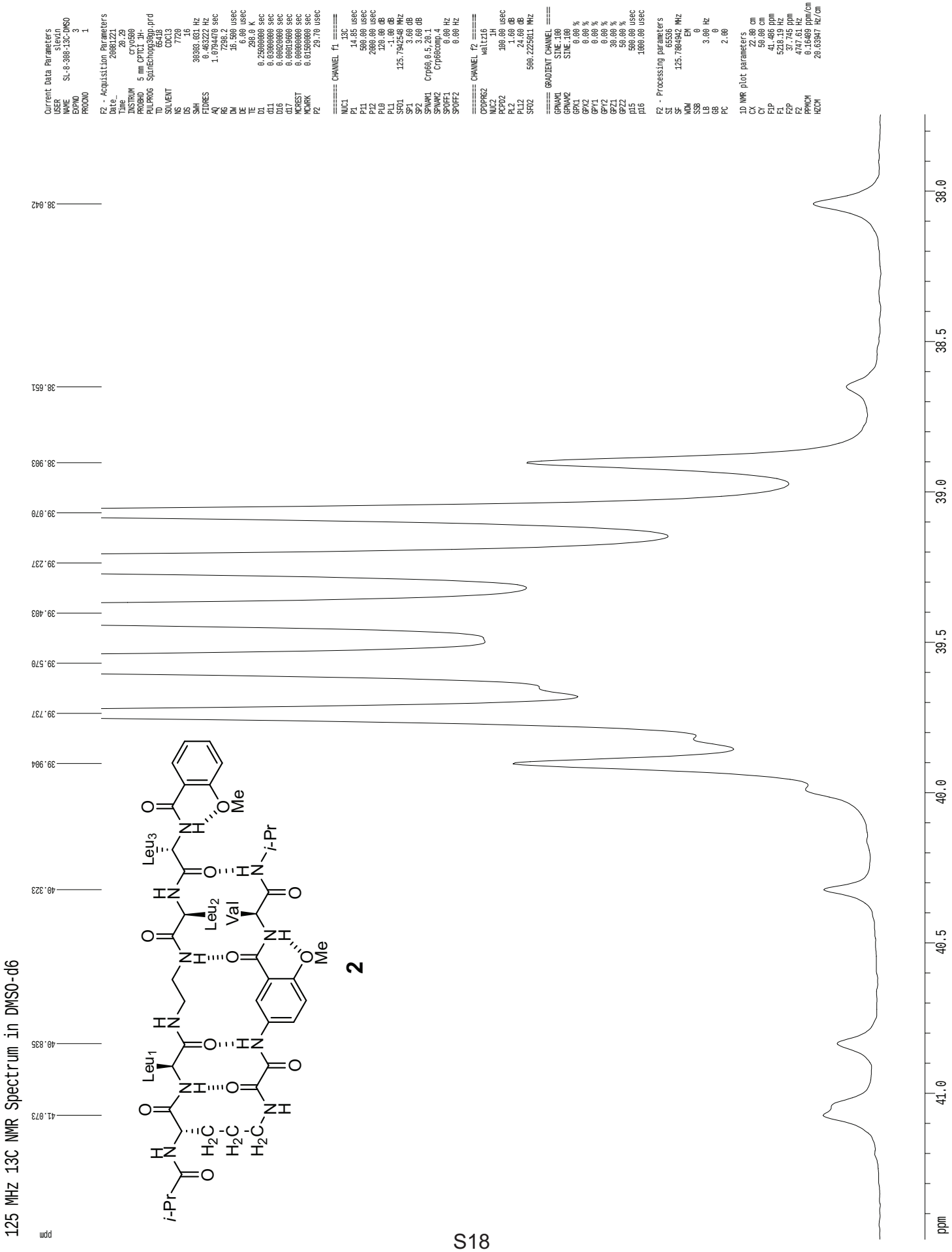
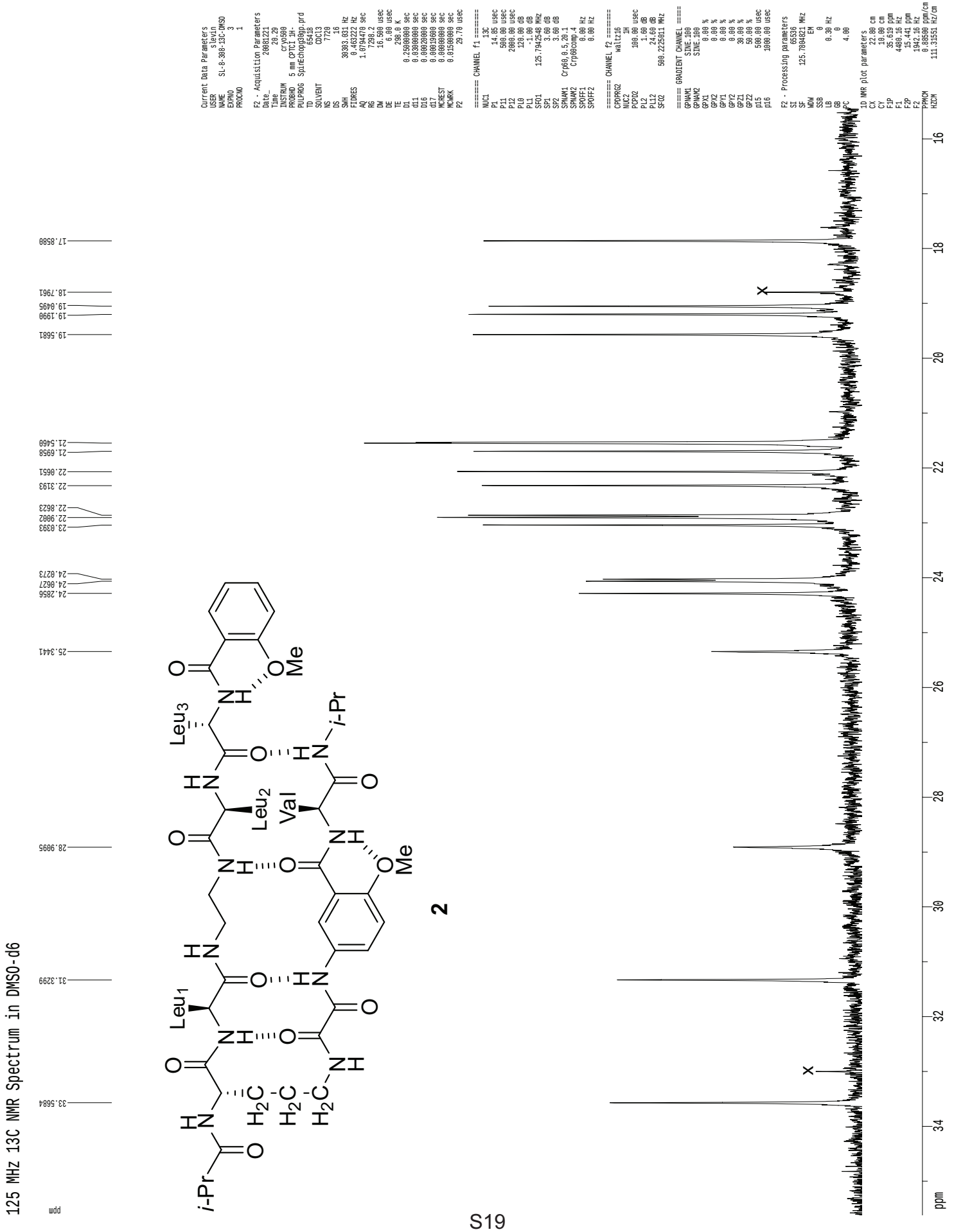
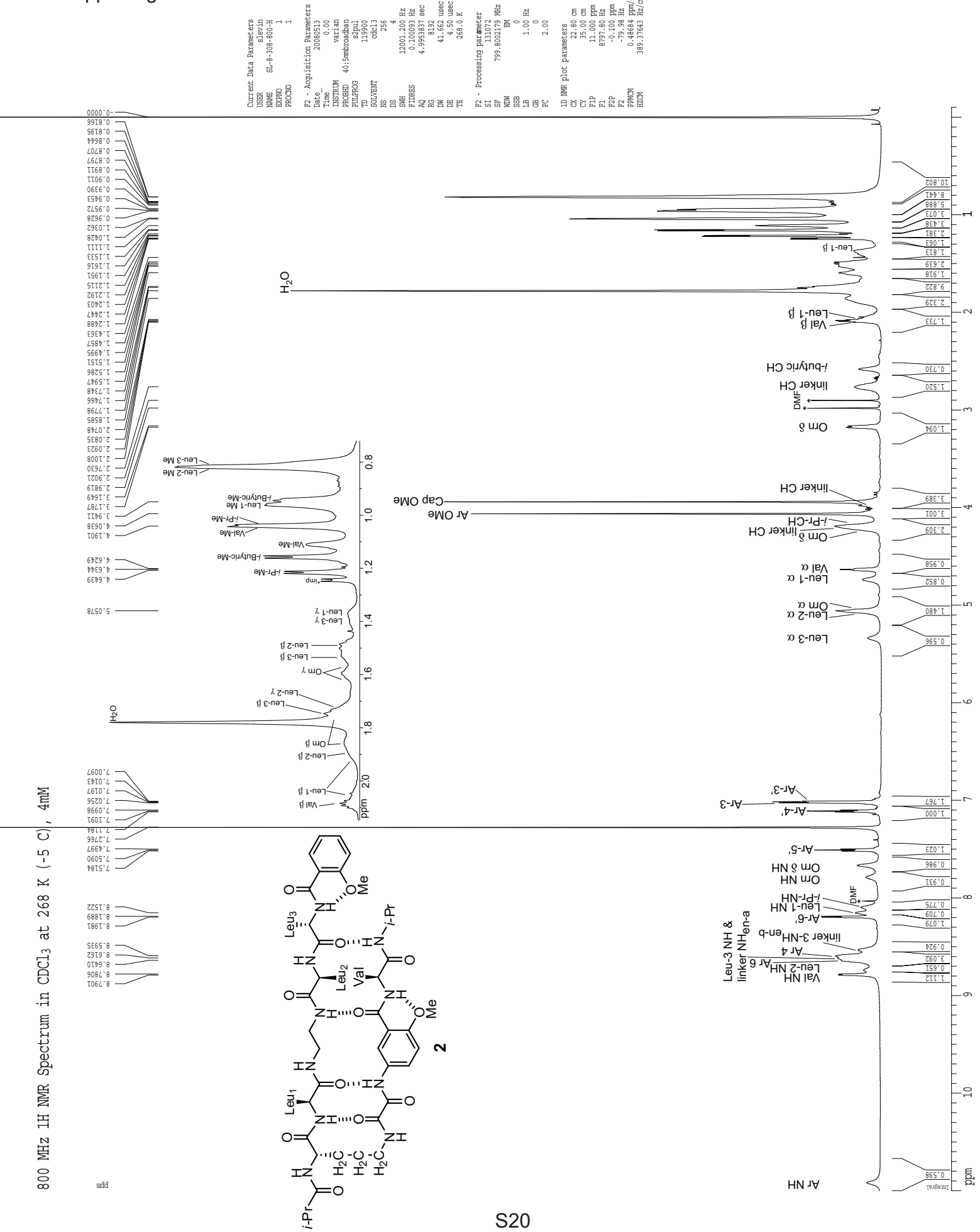
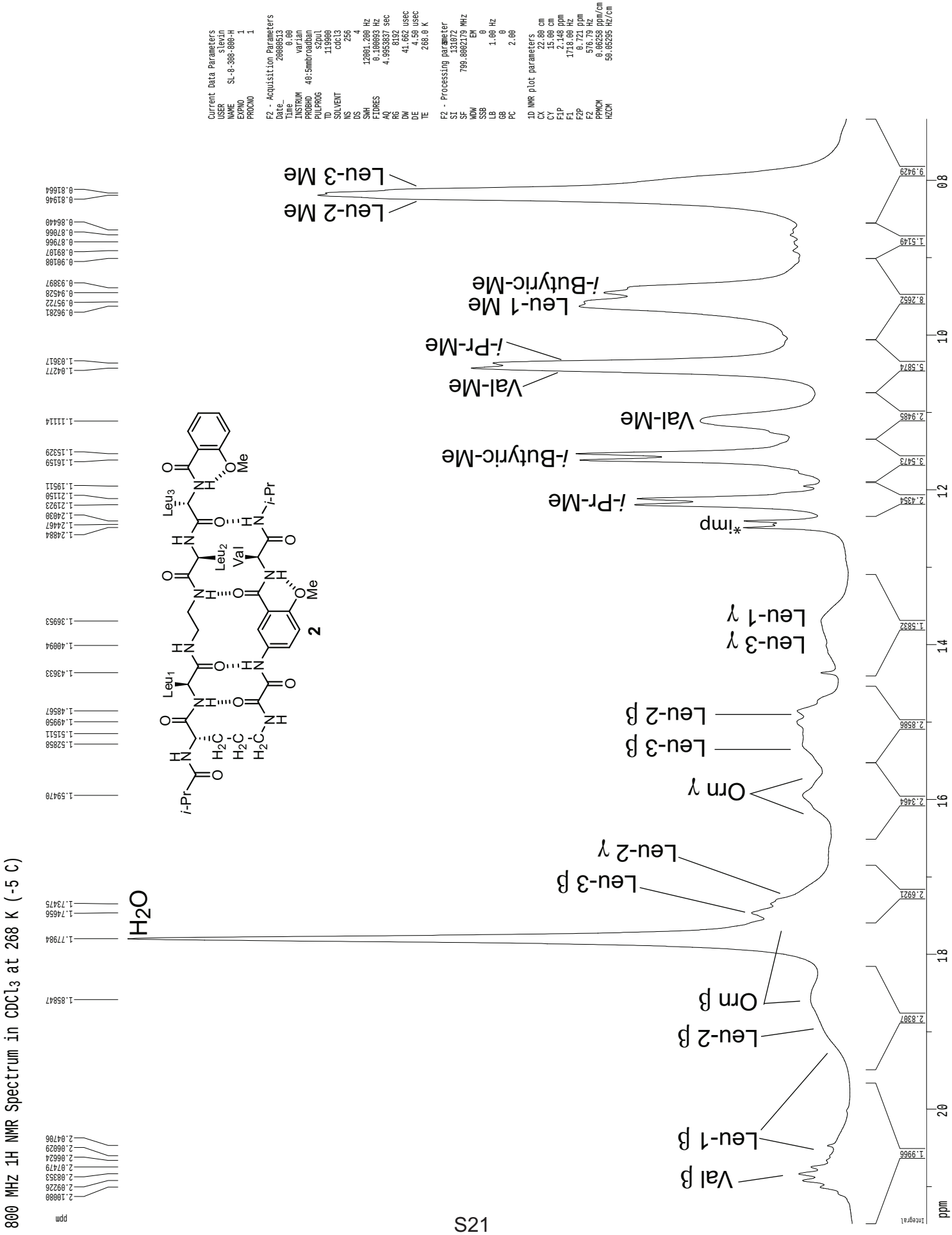
GPZ3 0.00 %
GPZ4 30.00 %
GPZ5 50.00 %
GPZ6 500.00 usec
p15 1000.00 usec
p16
F2 - Processing parameters
SI 65536
SF 125.7804942 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 178.358 ppm
F1 224314.01 Hz
F2P 110.745 ppm
F2 13929.52 Hz
PPMCM 2.96552 ppm/cm
HZCM 373.00433 Hz/cm
ppm
176.34
171.84
171.78
171.51
171.42
169.33
164.52
163.51
159.83
158.36
156.94
153.78
132.42
130.89
130.42
124.71
123.10
122.37
121.80
120.53
112.56
112.06
120
130
140
150
160
170
i-Pr
Leu1
Leu2
Leu3
Val
i-Pr
Me
Me
2

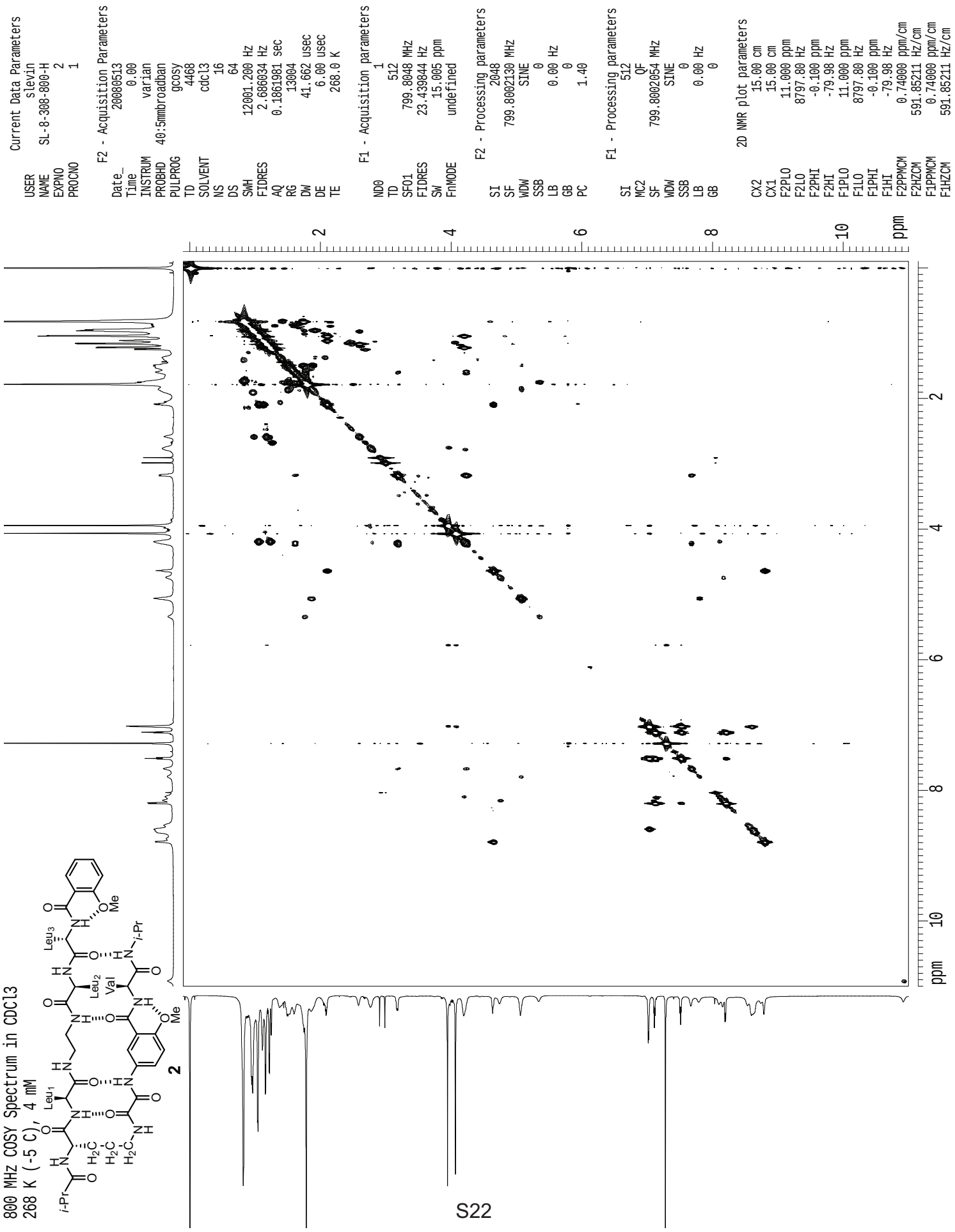

125 MHz 13C NMR Spectrum in DMSO-d6
2
Leu1
Leu2
Leu3
Val
i-Pr
Me
ppm

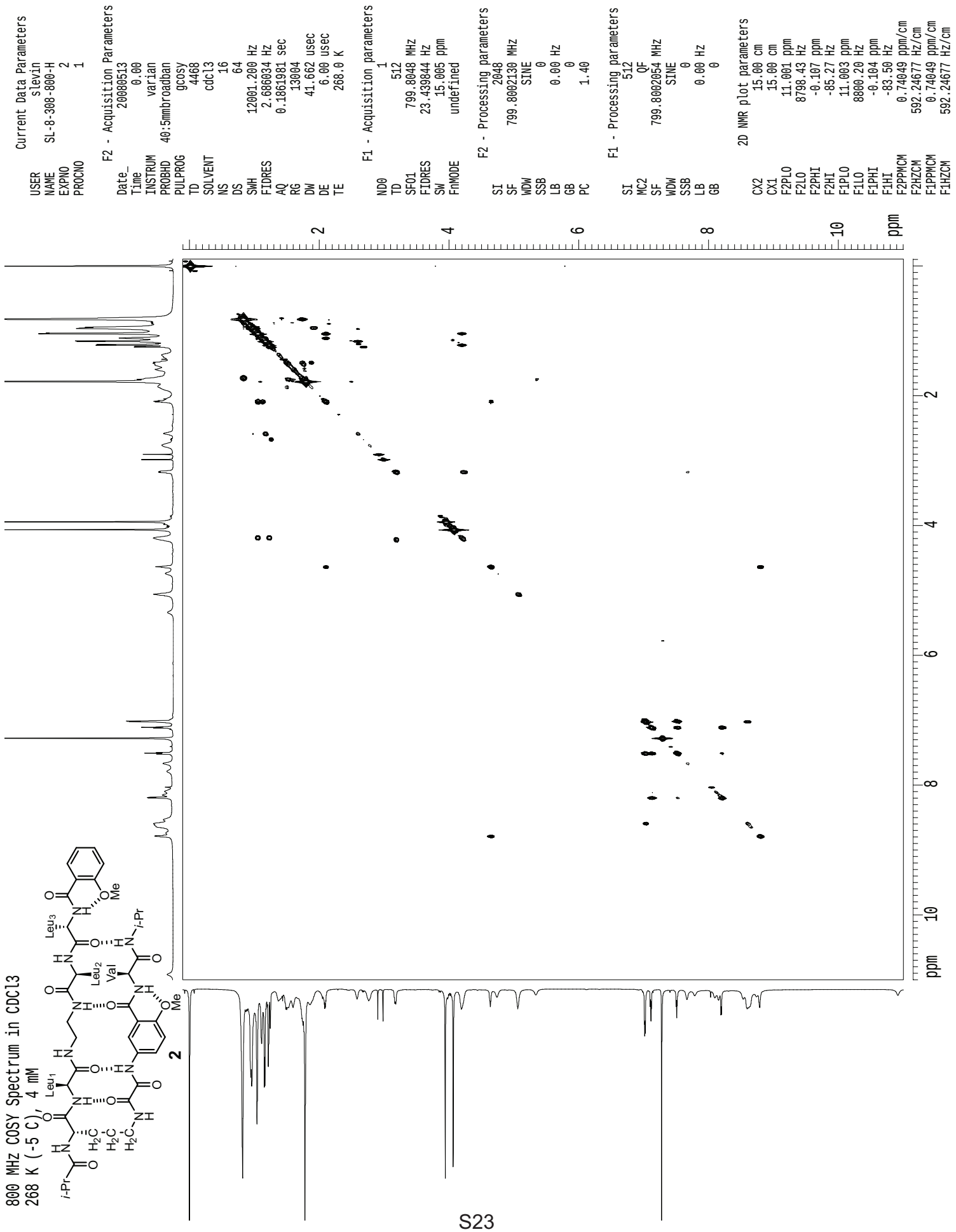

125 MHz 13C NMR Spectrum in DMSO-d6
Current Data Parameters
USER slevin
NAME SL-8-308-13C-DMSO
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061221
Time 20.29
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG Spinechoqg30gp.prd
TD 65418
SOLVENT CDCl3
NS 7720
DS 16
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 7298.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 29.70 usec
======== CHANNEL f1 ========
NUC1 13C
P1 14.85 usec
P11 500.00 usec
P12 2000.00 usec
PL9 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.60 dB
SP2 3.60 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
d15 500.00 usec
d16 1000.00 usec
F2 - Processing parameters
SI 65536
SF 125.7894942 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.00 cm
CY 59.00 cm
F1P 41.406 ppm
F1 5219.19 Hz
F2P 37.745 ppm
F2 4747.61 Hz
PPMCM 0.16409 ppm/cm
HZCM 20.63947 Hz/cm
38.042
38.651
38.903
39.070
39.237
39.403
39.570
39.737
39.904
40.323
40.835
41.073
ppm
38.0
38.5
39.0
39.5
40.0
40.5
41.0
ppm
i-Pr
H2C
H2C
H2C
Leu1
Leu2
Val
Leu3
i-Pr
Me
Me
2

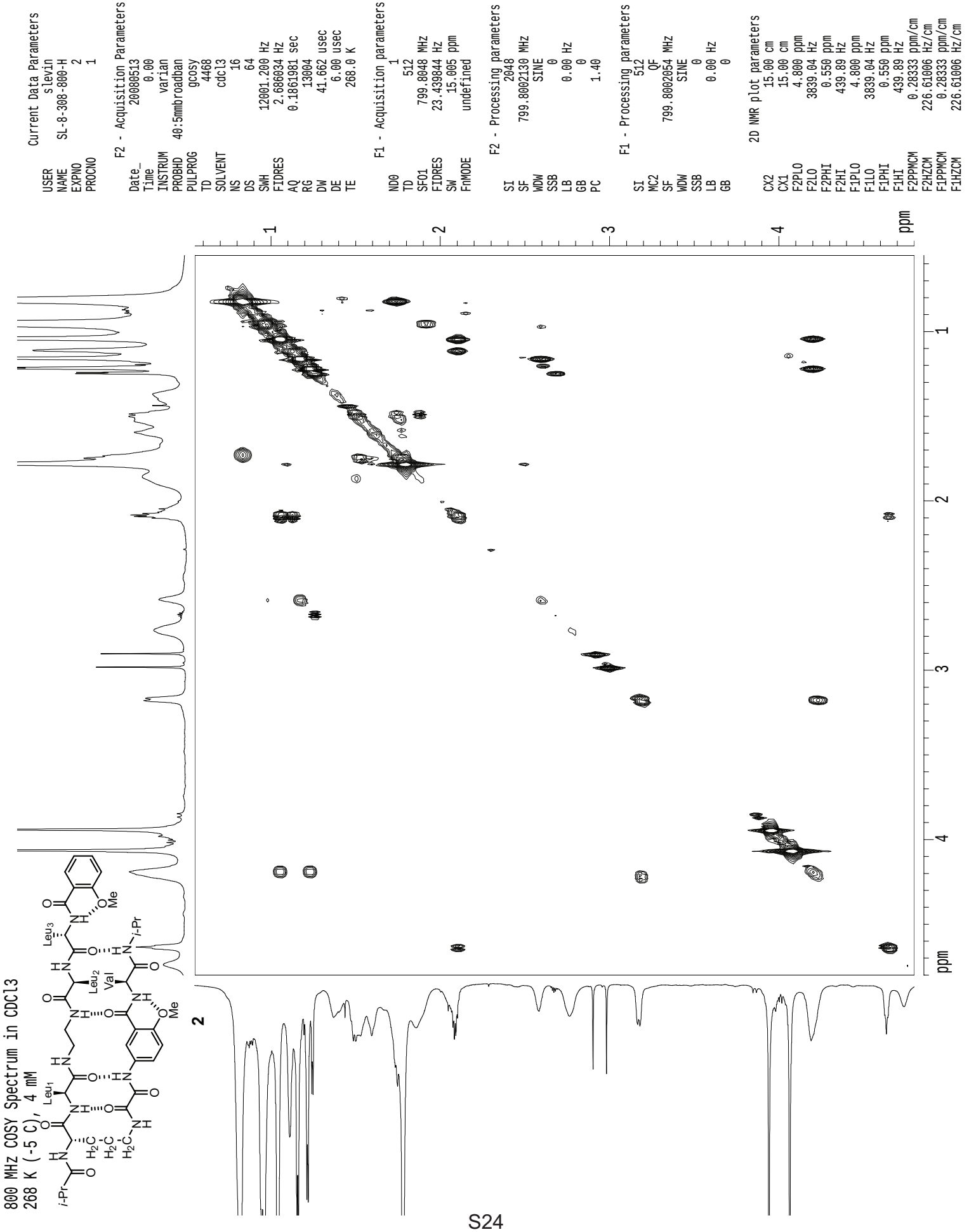

125 MHz 13C NMR Spectrum in DMSO-d6
2
Leu1
Leu2
Val
Leu3
i-Pr
Me
ppm

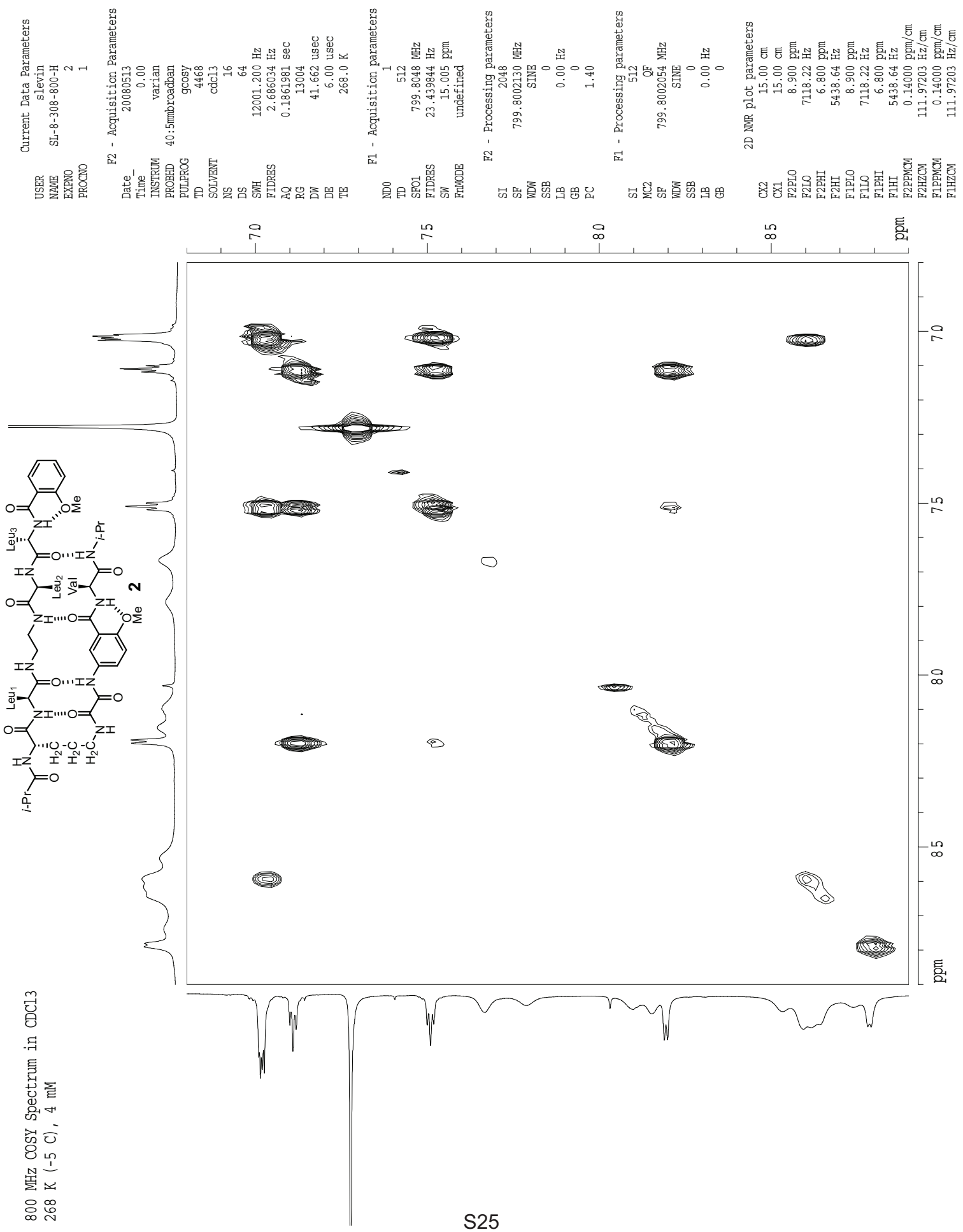

800 MHz 1H NMR Spectrum in CDCl3 at 268 K (-5 C), 4mM
Current Data Parameters
USER slevin
NAME SL-8-308-800-H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080513
Time 0.00
INSTRUM varian
PROBHD 40:5mmbroadban
PULPROG s2pul
TD 119900
SOLVENT cdcl3
NS 256
DS 4
SWH 12001.200 Hz
FIDRES 0.100093 Hz
AQ 4.9953837 sec
RG 8192
DW 41.662 usec
DE 4.50 usec
TE 268.0 K
F2 - Processing parameter
SI 1310720
SF 799.8002179 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 35.00 cm
F1P 11.000 ppm
F1 8797.80 Hz
F2P -0.100 ppm
F2 -79.98 Hz
PPMCM 0.48684 ppm/cm
HZCM 389.37643 Hz/cm
ppm
Integral
H2O
Leu-1 β
Val β
Leu-1 β
linker CH
i-butyric CH
linker CH
DMF
Orn δ
linker CH
Cap OMe
Ar OMe
Orn δ
linker CH
i-Pr-CH
Leu-1 α
Val α
Leu-2 α
Orn α
Leu-3 α
Ar-3'
Ar-4'
Ar-3
Ar-5'
Orn δ NH
Orn NH
DMF
i-Pr-NH
Leu-1 NH
Ar-6'
linker 3-NH
Ar 4
Leu-3 NH &
linker NH
Ar 6
Leu-2 NH
Val NH
Ar NH
Leu-3 β
Leu-2 β
Orn β
Leu-3 γ
Leu-1 γ
i-Pr-Me
i-Butyric-Me
Val-Me
Val-Me
i-Pr-Me
Leu-1 Me
i-Butyric-Me
Leu-2 Me
Leu-3 Me
Leu-3 γ
Leu-2 γ
Orn γ
Leu-3 β
Leu-2 β
Orn β
Leu-1 β
Val β
i-Pr
Leu1
Leu3
Leu2
Val
Me
Me
2


800 MHz 1H NMR Spectrum in CDCl3 at 268 K (-5 C)
Current Data Parameters
USER slevin
NAME SL-8-388-888-H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080513
Time 0.00
INSTRUM varian
PROBHD 40:5mmbroadban
PULPROG s2pul
TD 119900
SOLVENT cdcl3
NS 256
DS 4
SWH 12001.200 Hz
FIDRES 0.100093 Hz
AQ 4.99533837 sec
RG 8192
DW 41.662 usec
DE 4.50 usec
TE 268.0 K
F2 - Processing parameter
SI 131072
SF 799.8002179 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.00 cm
CY 15.00 cm
F1P 2.148 ppm
F1 1718.00 Hz
F2P 0.721 ppm
F2 576.79 Hz
PPMCM 0.06258 ppm/cm
HZCM 50.05295 Hz/cm
ppm
Integral
H2O
Leu-3 Me
Leu-2 Me
Leu-1 Me
i-Butyric-Me
Val-Me
i-Pr-Me
*imp
Leu-3 γ
Leu-1 γ
Leu-2 β
Leu-3 β
Orn γ
Leu-3 β
Leu-2 γ
Leu-2 β
Orn β
Leu-1 β
Val β
i-Pr
Leu1
Leu2
Val
Leu3
i-Pr
Me
2

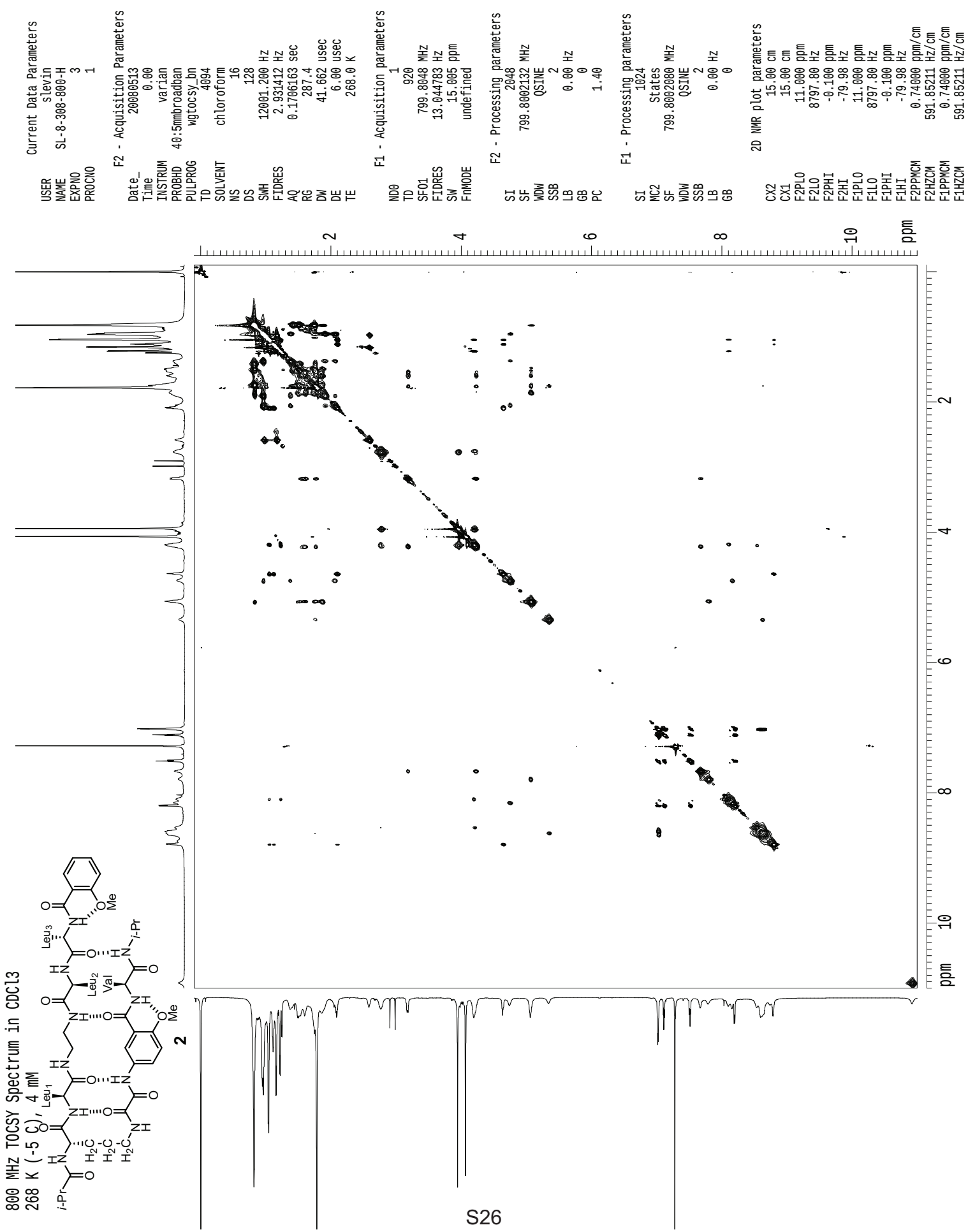

800 MHz COSY Spectrum in CDCl3
268 K (-5 C), 4 mM
2
Current Data Parameters
USER slevin
NAME SL-8-308-800-H
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080513
Time 0.00
INSTRUM varian
PROBHD 40:5mmbroadban
PULPROG gcosy
TD 4468
SOLVENT cdcl3
NS 16
DS 64
SWH 12001.200 Hz
FIDRES 2.686034 Hz
AQ 0.1861981 sec
RG 13004
DW 41.662 usec
DE 6.00 usec
TE 268.0 K
F1 - Acquisition parameters
ND0 1
TD 512
SFO1 799.8048 MHz
FIDRES 23.439844 Hz
SW 15.005 ppm
FnMODE undefined
F2 - Processing parameters
SI 2048
SF 799.8002130 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 512
MC2 QF
SF 799.8002054 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 11.000 ppm
F2LO 8797.80 Hz
F2PHI -0.100 ppm
F2HI -79.98 Hz
F1PLO 11.000 ppm
F1LO 8797.80 Hz
F1PHI -0.100 ppm
F1HI -79.98 Hz
F2PPMCM 0.74000 ppm/cm
F2HZCM 591.85211 Hz/cm
F1PPMCM 0.74000 ppm/cm
F1HZCM 591.85211 Hz/cm
ppm

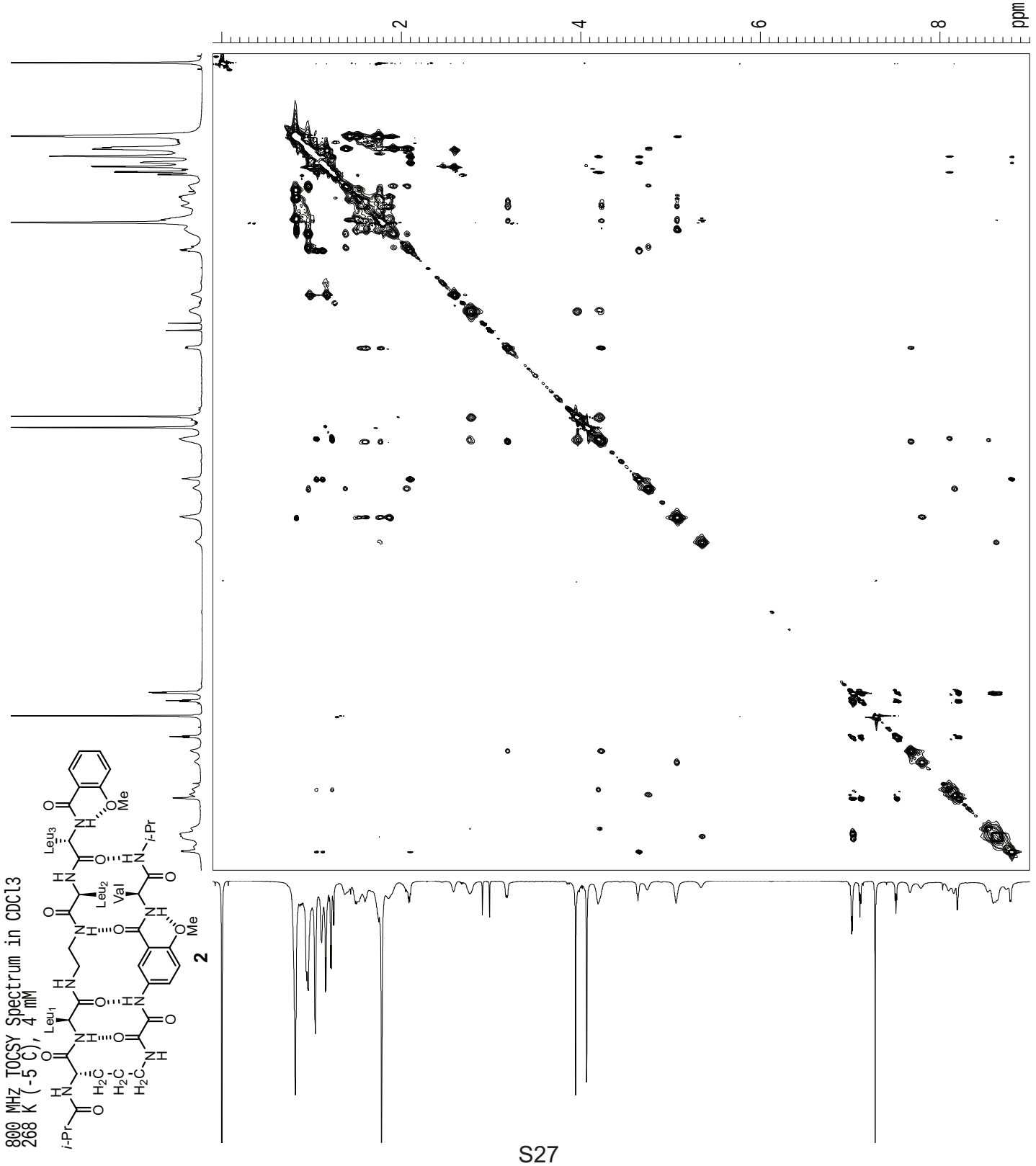

800 MHz COSY Spectrum in CDCl3
268 K (-5 C), 4 mM
2
Current Data Parameters
USER slevin
NAME SL-8-308-800-H
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080513
Time 0.00
INSTRUM varian
PROBHD 40:5mmbroadban
PULPROG gcosy
TD 4468
SOLVENT cdcl3
NS 16
DS 64
SWH 12001.200 Hz
FIDRES 2.686034 Hz
AQ 0.1861981 sec
RG 13004
DW 41.662 usec
DE 6.00 usec
TE 268.0 K
F1 - Acquisition parameters
ND0 1
TD 512
SFO1 799.8048 MHz
FIDRES 23.439844 Hz
SW 15.005 ppm
FnMODE undefined
F2 - Processing parameters
SI 2048
SF 799.8002130 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 512
MC2 QF
SF 799.8002054 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 11.001 ppm
F2LO 8798.43 Hz
F2PHI -0.107 ppm
F2HI -85.27 Hz
F1PLO 11.003 ppm
F1LO 8800.20 Hz
F1PHI -0.104 ppm
F1HI -83.50 Hz
F2PPMCM 0.74049 ppm/cm
F2HZCM 592.24677 Hz/cm
F1PPMCM 0.74049 ppm/cm
F1HZCM 592.24677 Hz/cm
ppm


800 MHz COSY Spectrum in CDCl3
268 K (-5 C), 4 mM
2
Current Data Parameters
USER slevin
NAME SL-8-308-800-H
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080513
Time 0.00
INSTRUM varian
PROBHD 40:5mmbroadban
PULPROG gcosy
TD 4468
SOLVENT cdcl3
NS 16
DS 64
SWH 12001.200 Hz
FIDRES 2.686034 Hz
AQ 0.1861981 sec
RG 13004
DW 41.662 usec
DE 6.00 usec
TE 268.0 K
F1 - Acquisition parameters
ND0 1
TD 512
SFO1 799.8048 MHz
FIDRES 23.439844 Hz
SW 15.005 ppm
FnMODE undefined
F2 - Processing parameters
SI 2048
SF 799.8002130 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 512
MC2 QF
SF 799.8002054 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 4.800 ppm
F2LO 3839.04 Hz
F2PHI 0.550 ppm
F2HI 439.89 Hz
F1PLO 4.800 ppm
F1LO 3839.04 Hz
F1PHI 0.550 ppm
F1HI 439.89 Hz
F2PPMCM 0.28333 ppm/cm
F2HZCM 226.61006 Hz/cm
F1PPMCM 0.28333 ppm/cm
F1HZCM 226.61006 Hz/cm
ppm
1
2
3
4



800 MHz COSY Spectrum in CDCl3
268 K (-5 C), 4 mM


800 MHz TOCSY Spectrum in CDCl3
268 K (-5 C), 4 mM
2
Current Data Parameters
USER slevin
NAME SL-8-308-800-H
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080513
Time 0.00
INSTRUM varian
PROBHD 40:5mmbroadban
PULPROG wgtocsy_bn
TD 4094
SOLVENT chloroform
NS 16
DS 128
SWH 12001.200 Hz
FIDRES 2.931412 Hz
AQ 0.1706163 sec
RG 287.4
DW 41.662 usec
DE 6.00 usec
TE 268.0 K
F1 - Acquisition parameters
ND0 1
TD 920
SFO1 799.8048 MHz
FIDRES 13.044783 Hz
SW 15.005 ppm
FnMODE undefined
F2 - Processing parameters
SI 2048
SF 799.8002132 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 1024
MC2 States
SF 799.8002080 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 11.000 ppm
F2LO 8797.80 Hz
F2PHI -0.100 ppm
F2HI -79.98 Hz
F1PLO 11.000 ppm
F1LO 8797.80 Hz
F1PHI -0.100 ppm
F1HI -79.98 Hz
F2PPMCM 0.74000 ppm/cm
F2HZCM 591.85211 Hz/cm
F1PPMCM 0.74000 ppm/cm
F1HZCM 591.85211 Hz/cm
ppm

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Current Data Parameters
     USER      slevin
     NAME   SL-8-308-800-H
    EXPNO            3
    PROCNO           1

F2 - Acquisition Parameters
    Date_     20080513
    Time          0.00
INSTRUM          varian
PROBHD   40:5mmbroadban
PULPROG     wgtocsy_bn
     TD           4094
  SOLVENT     chloroform
     NS             16
     DS            128
    SWH      12001.200 Hz
  FIDRES      2.931412 Hz
      AQ      0.1706163 sec
      RG          287.4
      DW         41.662 usec
      DE          6.00 usec
      TE         268.0 K

F1 - Acquisition parameters
     ND0            1
      TD          920
    SFO1    799.8048 MHz
  FIDRES    13.044783 Hz
      SW       15.005 ppm
  FnMODE     undefined

F2 - Processing parameters
      SI         2048
      SF   799.8002132 MHz
     WDW         QSINE
     SSB             2
      LB      0.00 Hz
      GB             0
      PC          1.40

F1 - Processing parameters
      SI          1024
     MC2        States
      SF   799.8002080 MHz
     WDW         QSINE
     SSB             2
      LB      0.00 Hz
      GB             0

2D NMR plot parameters
    CX2        15.00 cm
    CX1        15.00 cm
  F2PLO        9.000 ppm
  F2LO       7198.20 Hz
  F2PHI       -0.100 ppm
    F2HI       -79.98 Hz
  F1PLO        9.000 ppm
  F1LO       7198.20 Hz
  F1PHI       -0.100 ppm
    F1HI       -79.98 Hz
F2PPMCM     0.60667 ppm/cm
  F2HZCM    485.21210 Hz/cm
F1PPMCM     0.60667 ppm/cm
  F1HZCM    485.21210 Hz/cm
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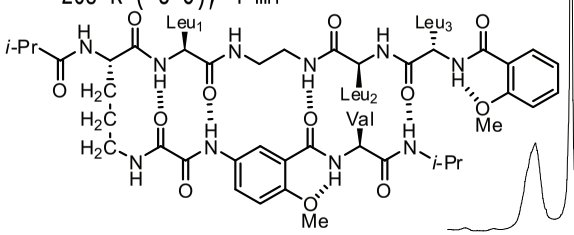
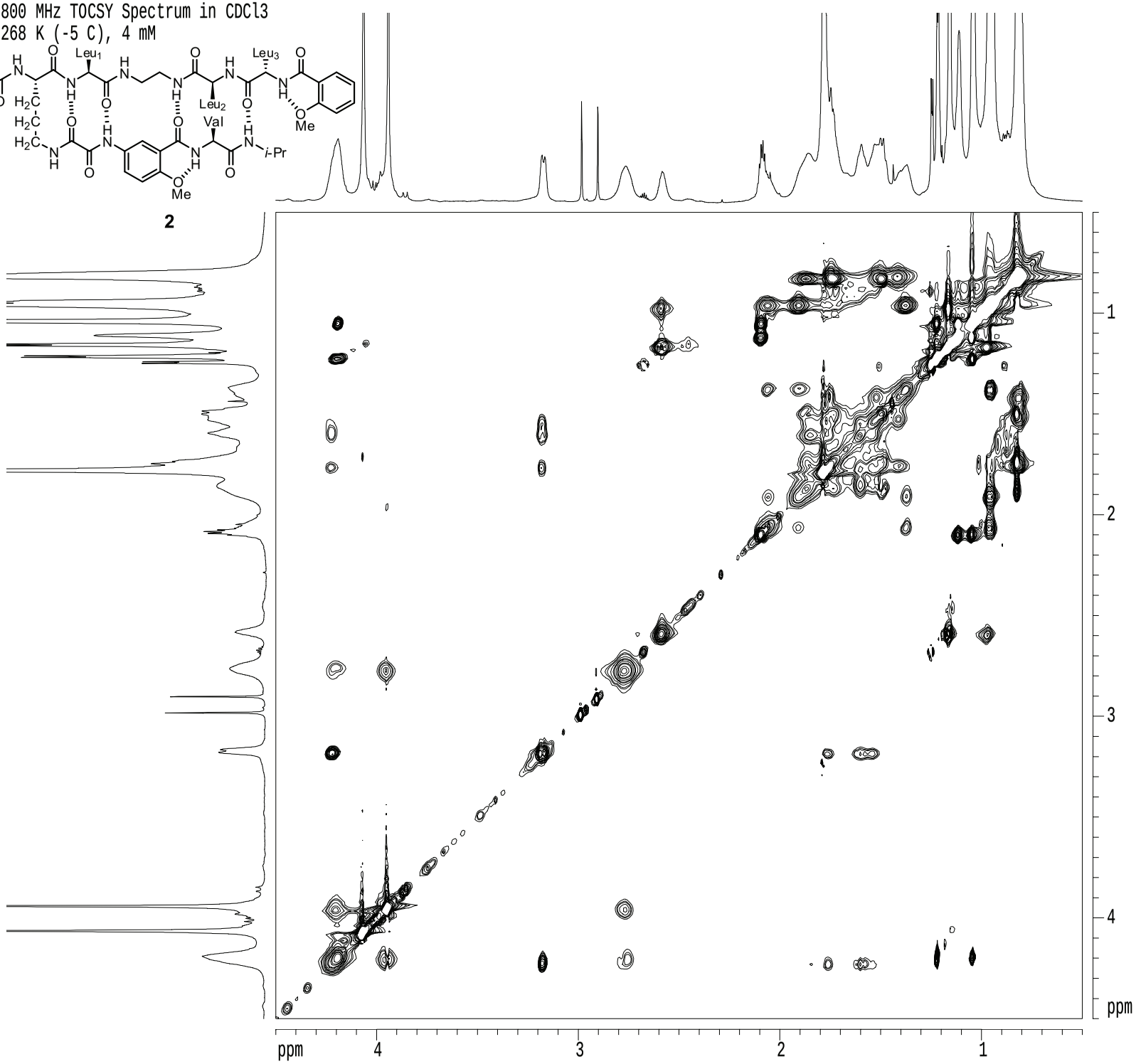
800 MHz TOCSY Spectrum in CDCl3
268 K (-5 C), 4 mM



2




```
        Current Data Parameters
USER              slevin
NAME      SL-8-308-800-H
EXPNO                  3
PROCNO                 1

      F2 - Acquisition Parameters
Date_           20080513
Time               0.00
INSTRUM           varian
PROBHD   40:5mmbroadban
PULPROG      wgtocsy_bn
TD                 4094
SOLVENT       chloroform
NS                   16
DS                  128
SWH           12001.200 Hz
FIDRES         2.931412 Hz
AQ            0.1706163 sec
RG                287.4
DW               41.662 usec
DE                 6.00 usec
TE                268.0 K

      F1 - Acquisition parameters
ND0                    1
TD                   920
SFO1           799.8048 MHz
FIDRES        13.044783 Hz
SW               15.005 ppm
FnMODE        undefined

      F2 - Processing parameters
SI                  2048
SF          799.8002132 MHz
WDW               QSINE
SSB                    2
LB                 0.00 Hz
GB                    0
PC                 1.40

      F1 - Processing parameters
SI                  1024
MC2               States
SF          799.8002080 MHz
WDW               QSINE
SSB                    2
LB                 0.00 Hz
GB                    0

        2D NMR plot parameters
CX2               15.00 cm
CX1               15.00 cm
F2PLO             4.500 ppm
F2LO            3599.10 Hz
F2PHI             0.500 ppm
F2HI             399.90 Hz
F1PLO             4.500 ppm
F1LO            3599.10 Hz
F1PHI             0.500 ppm
F1HI             399.90 Hz
F2PPMCM         0.26667 ppm/cm
F2HZCM        213.28006 Hz/cm
F1PPMCM         0.26667 ppm/cm
F1HZCM        213.28006 Hz/cm
```

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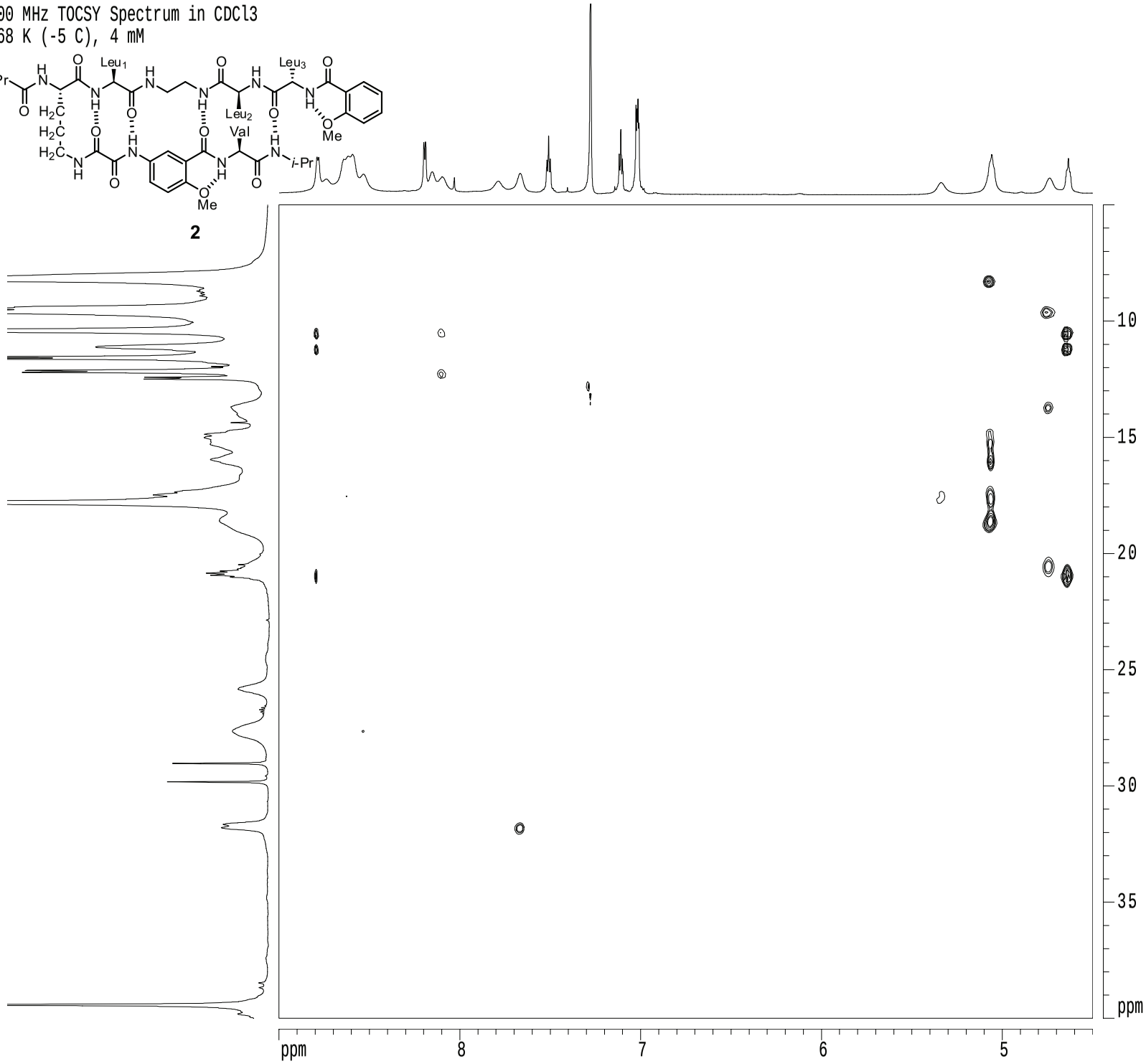
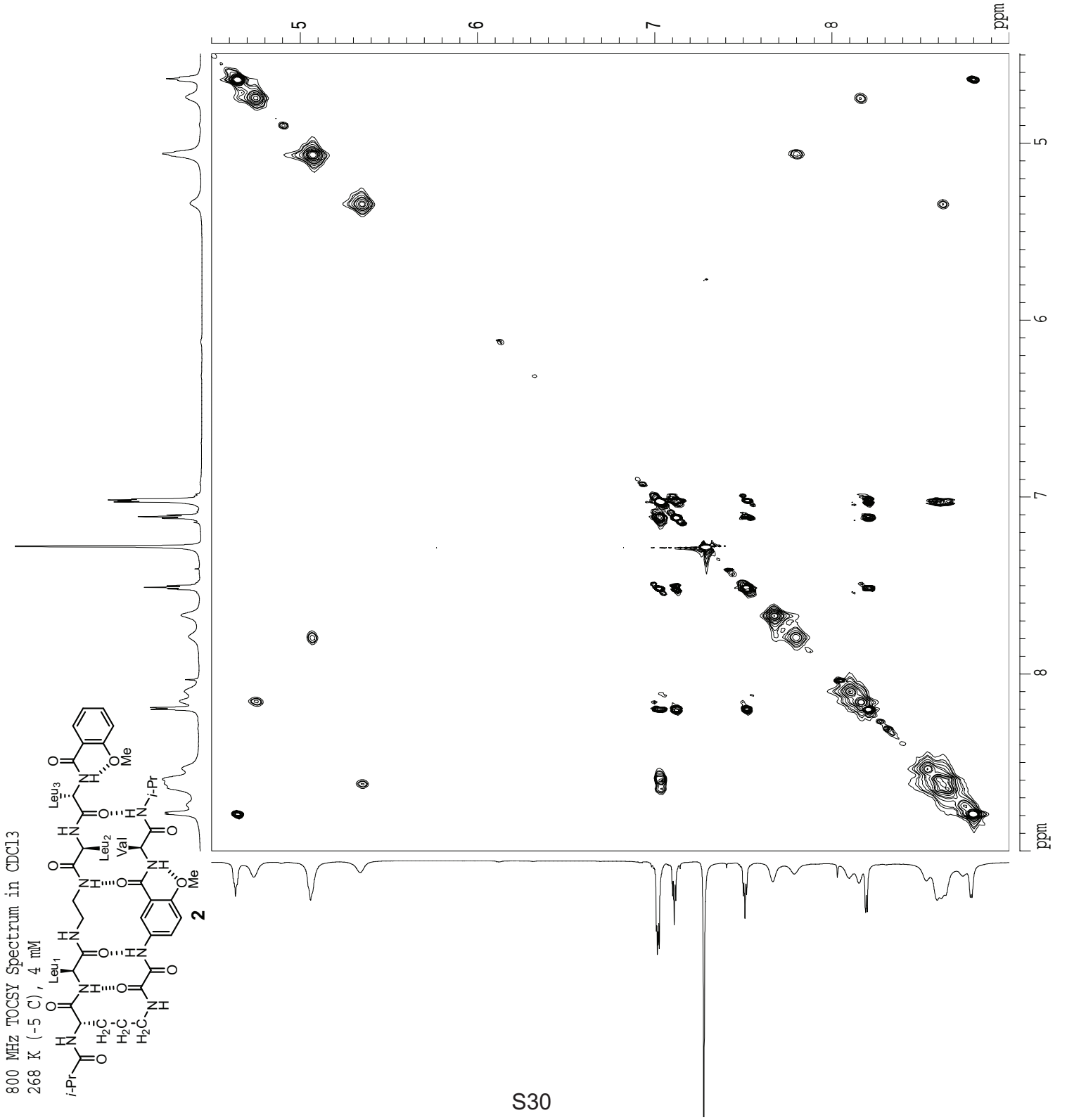
800 MHz TOCSY Spectrum in CDCl3
268 K (-5 C), 4 mM

2

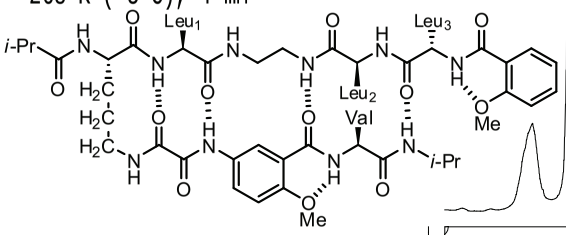



```
          Current Data Parameters
USER                slevin
NAME       SL-8-308-800-H
EXPNO                   3
PROCNO                  1

        F2 - Acquisition Parameters
Date_            20080513
Time                 0.00
INSTRUM            varian
PROBHD   40:5mmbroadban
PULPROG       wgtocsy_bn
TD                   4094
SOLVENT       chloroform
NS                     16
DS                    128
SWH             12001.200 Hz
FIDRES           2.931412 Hz
AQ              0.1706163 sec
RG                  287.4
DW                 41.662 usec
DE                   6.00 usec
TE                  268.0 K

        F1 - Acquisition parameters
ND0                     1
TD                    920
SFO1            799.8048 MHz
FIDRES         13.044783 Hz
SW                15.005 ppm
FnMODE         undefined

        F2 - Processing parameters
SI                   2048
SF           799.8002132 MHz
WDW                 QSINE
SSB                     2
LB                   0.00 Hz
GB                      0
PC                   1.40

        F1 - Processing parameters
SI                   1024
MC2                States
SF           799.8002080 MHz
WDW                 QSINE
SSB                     2
LB                   0.00 Hz
GB                      0

          2D NMR plot parameters
CX2                 15.00 cm
CX1                 15.00 cm
F2PLO               9.000 ppm
F2LO              7198.20 Hz
F2PHI               4.500 ppm
F2HI              3599.10 Hz
F1PLO               4.000 ppm
F1LO              3199.20 Hz
F1PHI               0.500 ppm
F1HI                399.90 Hz
F2PPMCM           0.30000 ppm/cm
F2HZCM          239.94006 Hz/cm
F1PPMCM           0.23333 ppm/cm
F1HZCM          186.62006 Hz/cm
```

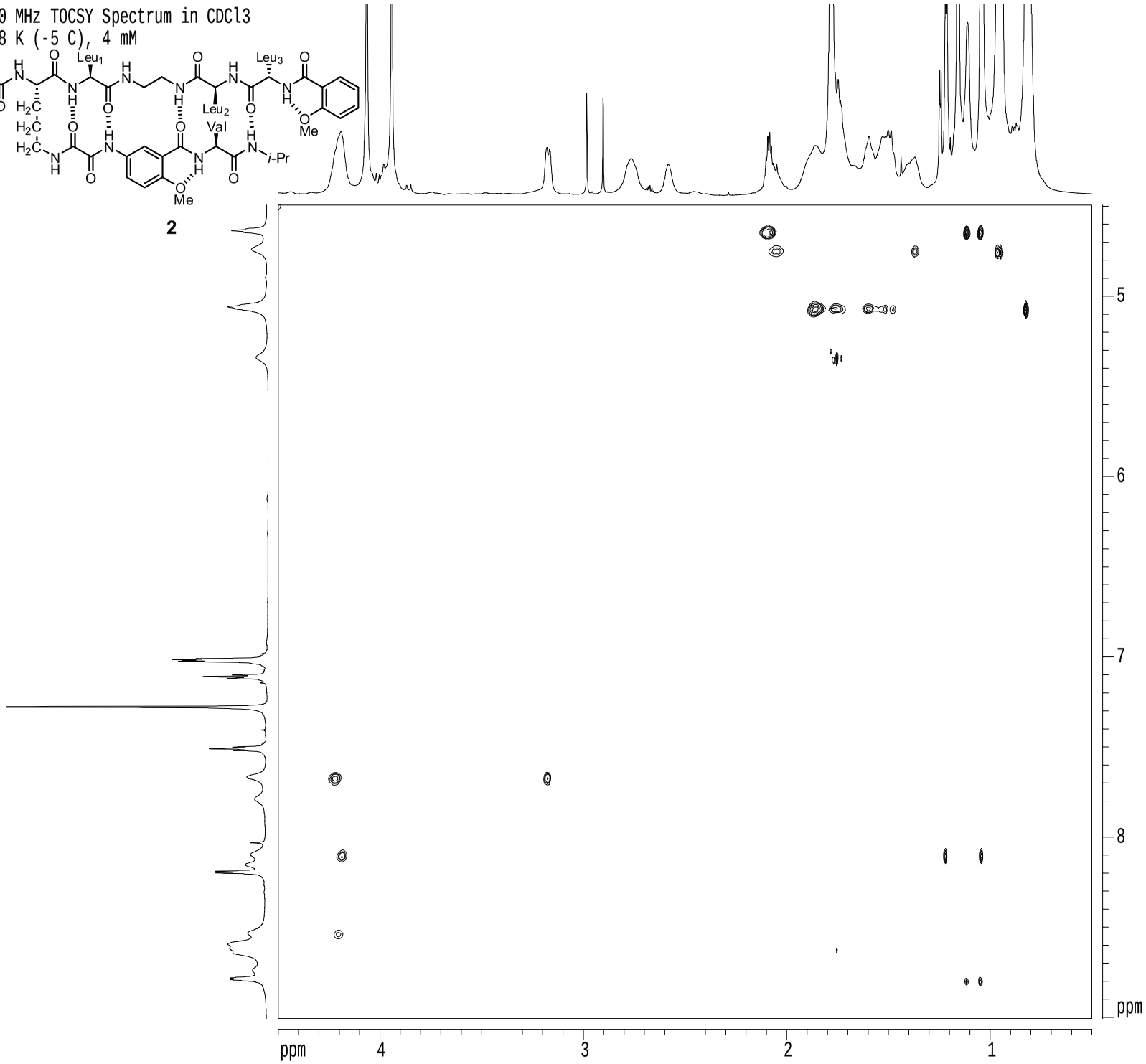
S. Levin and James S. Nowick


800 MHz TOCSY Spectrum in CDCl3
268 K (-5 C), 4 mM
2
Leu1
Leu2
Val
Leu3
i-Pr
Me
Current Data Parameters
USER slevin
NAME SL-8-308-800-H
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080513
Time 0.00
INSTRUM varian
PROBHD 40:5mmbroadban
PULPROG wgtocsy_bn
TD 4094
SOLVENT chloroform
NS 16
DS 128
SWH 12001.200 Hz
FIDRES 2.931412 Hz
AQ 0.1706163 sec
RG 287.4
DW 41.662 usec
DE 6.00 usec
TE 268.0 K
F1 - Acquisition parameters
ND0 1
TD 920
SFO1 799.8048 MHz
FIDRES 13.044783 Hz
SW 15.005 ppm
FnMODE undefined
F2 - Processing parameters
SI 2048
SF 799.8002132 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 1024
MC2 States
SF 799.8002080 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 9.000 ppm
F2LO 7198.47 Hz
F2PHI 4.494 ppm
F2HI 3594.59 Hz
F1PLO 9.000 ppm
F1LO 7198.20 Hz
F1PHI 4.500 ppm
F1HI 3599.10 Hz
F2PPMCM 0.30040 ppm/cm
F2HZCM 240.25842 Hz/cm
F1PPMCM 0.30000 ppm/cm
F1HZCM 239.94006 Hz/cm
ppm
5
6
7
8

800 MHz TOCSY Spectrum in CDCl3
268 K (-5 C), 4 mM



2

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        Current Data Parameters
USER              slevin
NAME      SL-8-308-800-H
EXPNO                  3
PROCNO                 1

      F2 - Acquisition Parameters
Date_           20080513
Time               0.00
INSTRUM           varian
PROBHD   40:5mmbroadban
PULPROG      wgtocsy_bn
TD                 4094
SOLVENT       chloroform
NS                   16
DS                  128
SWH           12001.200 Hz
FIDRES         2.931412 Hz
AQ            0.1706163 sec
RG                287.4
DW               41.662 usec
DE                 6.00 usec
TE                268.0 K

      F1 - Acquisition parameters
ND0                    1
TD                   920
SFO1           799.8048 MHz
FIDRES        13.044783 Hz
SW               15.005 ppm
FnMODE        undefined

      F2 - Processing parameters
SI                  2048
SF          799.8002132 MHz
WDW                QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
PC                  1.40

      F1 - Processing parameters
SI                  1024
MC2               States
SF          799.8002080 MHz
WDW                QSINE
SSB                    2
LB                  0.00 Hz
GB                     0

        2D NMR plot parameters
CX2                15.00 cm
CX1                15.00 cm
F2PLO              4.500 ppm
F2LO            3599.10 Hz
F2PHI              0.500 ppm
F2HI              399.90 Hz
F1PLO              9.007 ppm
F1LO            7203.68 Hz
F1PHI              4.494 ppm
F1HI            3593.94 Hz
F2PPMCM          0.26667 ppm/cm
F2HZCM         213.28006 Hz/cm
F1PPMCM          0.30089 ppm/cm
F1HZCM         240.64906 Hz/cm
```

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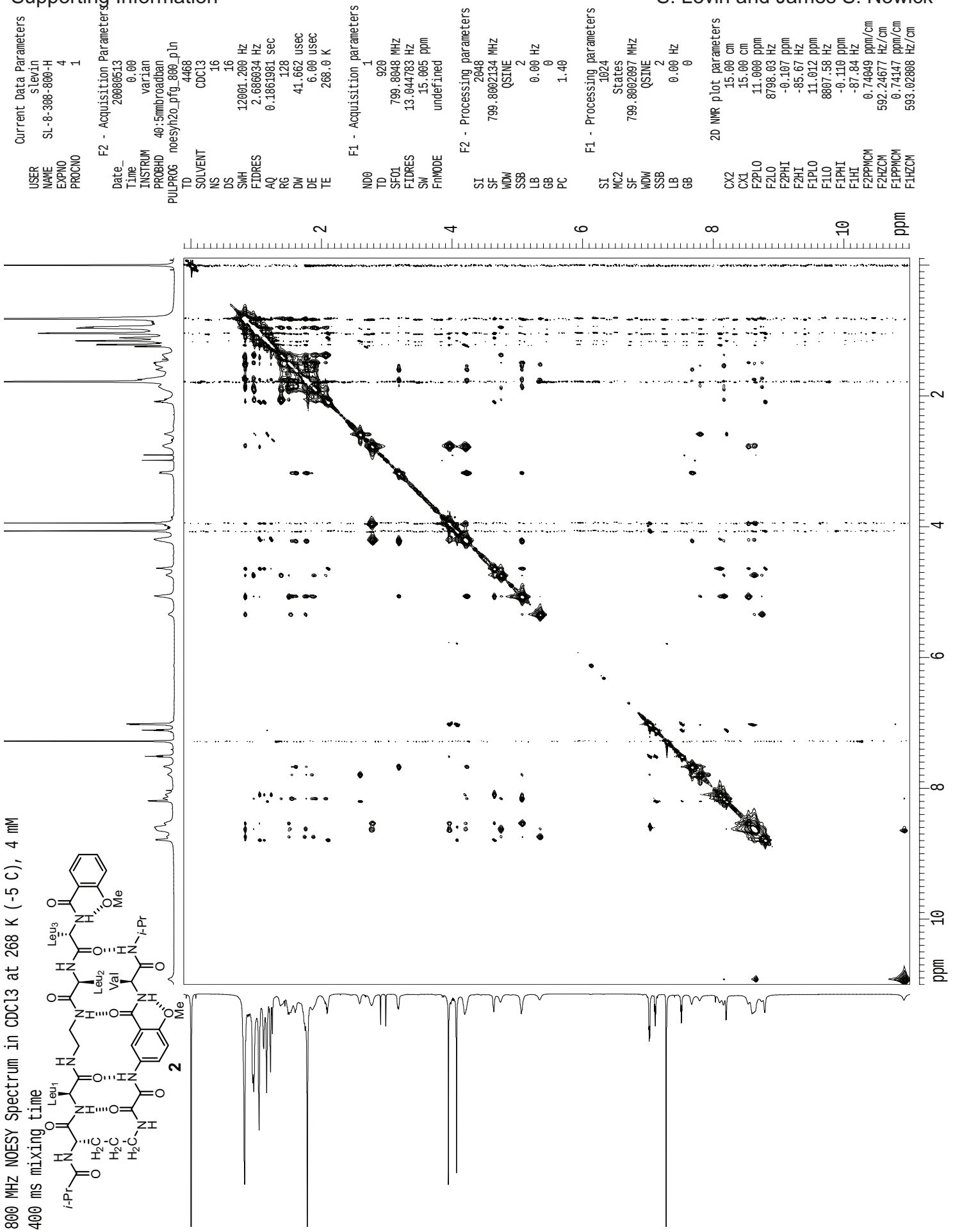
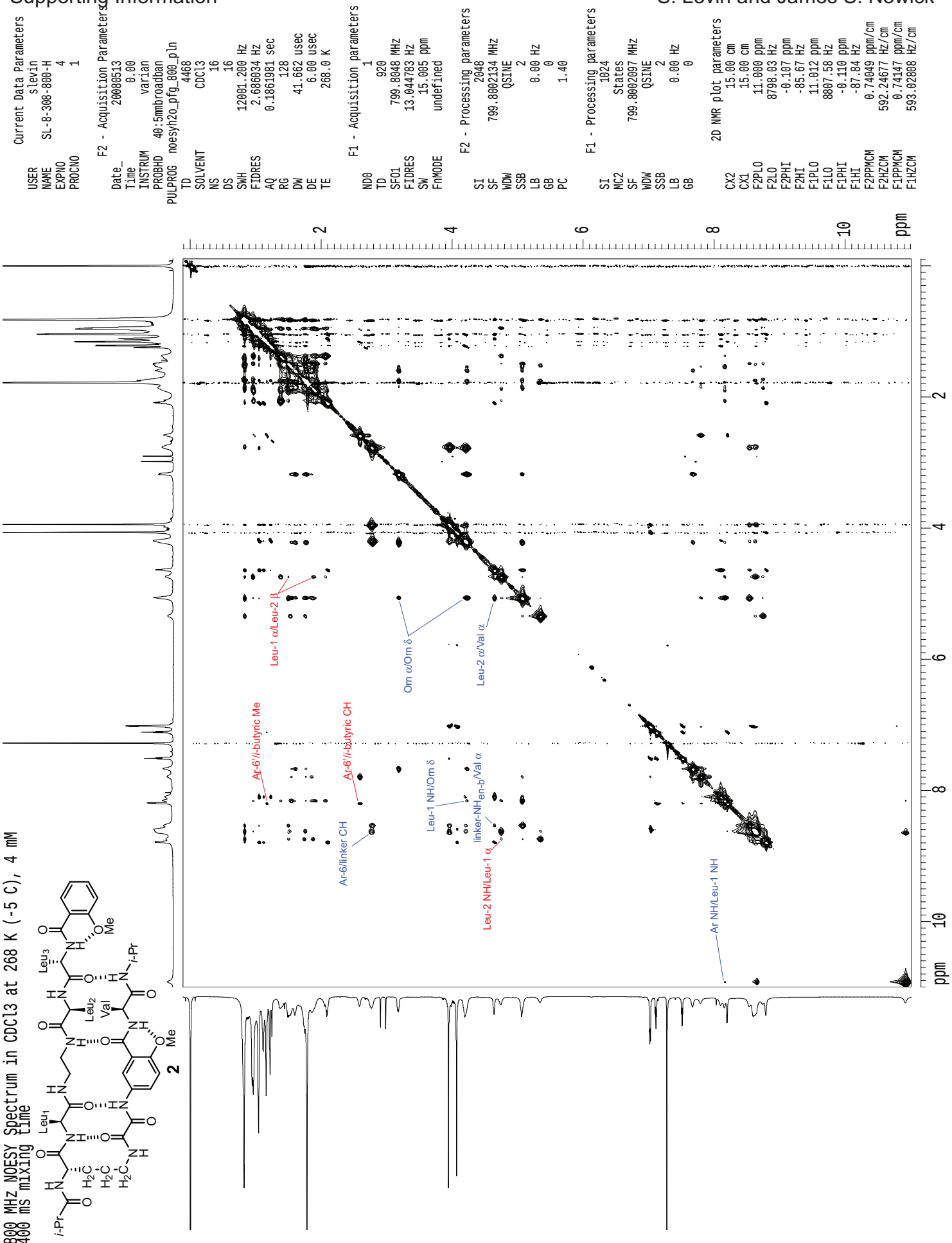
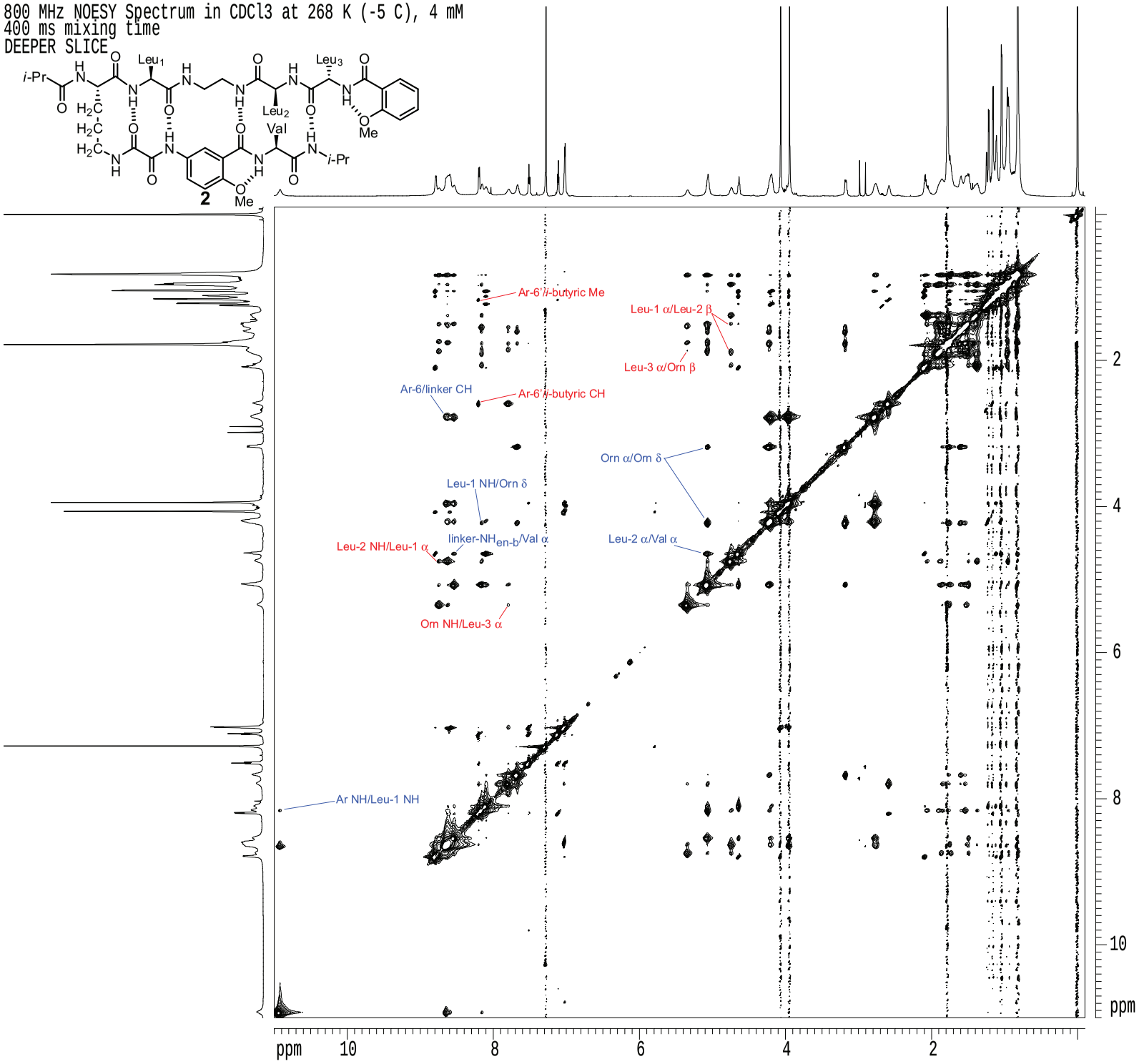

800 MHz NOESY Spectrum in CDCl3 at 268 K (-5 C), 4 mM
400 ms mixing time
Current Data Parameters
USER slevin
NAME SL-8-308-800-H
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080513
Time 0.00
INSTRUM varian
PROBHD 40:5mmbroadban
PULPROG noesyh2o_pfg_800_pln
TD 4468
SOLVENT CDCl3
NS 16
DS 16
SWH 12001.200 Hz
FIDRES 2.686034 Hz
AQ 0.1861981 sec
RG 128
DW 41.662 usec
DE 6.00 usec
TE 268.0 K
F1 - Acquisition parameters
ND0 1
TD 920
SFO1 799.8048 MHz
FIDRES 13.044783 Hz
SW 15.005 ppm
FnMODE undefined
F2 - Processing parameters
SI 2048
SF 799.8002134 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 1024
MC2 States
SF 799.8002097 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 11.000 ppm
F2LO 8798.03 Hz
F2PHI -0.107 ppm
F2HI -85.67 Hz
F1PLO 11.012 ppm
F1LO 8807.58 Hz
F1PHI -0.110 ppm
F1HI -87.84 Hz
F2PPMCM 0.74049 ppm/cm
F2HZCM 592.24677 Hz/cm
F1PPMCM 0.74147 ppm/cm
F1HZCM 593.02808 Hz/cm
ppm
2
4
6
8
10
Leu1
Leu2
Leu3
Val
i-Pr
Me
2


800 MHz NOESY Spectrum in CDCl3 at 268 K (-5 C), 4 mM
400 ms mixing time
Current Data Parameters
USER slevin
NAME SL-8-308-800-H
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080513
Time 0.00
INSTRUM varian
PROBHD 40.5mmbroadban
PULPROG noesyh2o_pfg_800_pln
TD 4468
SOLVENT CDCl3
NS 16
DS 16
SWH 12001.200 Hz
FIDRES 2.686034 Hz
AQ 0.1861981 sec
RG 128
DW 41.662 usec
DE 6.00 usec
TE 268.0 K
F1 - Acquisition parameters
ND0 1
TD 920
SFO1 799.8048 MHz
FIDRES 13.044783 Hz
SW 15.005 ppm
FnMODE undefined
F2 - Processing parameters
SI 2048
SF 799.8002134 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 1024
MC2 States
SF 799.8002097 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 11.000 ppm
F2LO 8798.03 Hz
F2PHI -0.107 ppm
F2HI -85.67 Hz
F1PLO 11.012 ppm
F1LO 8807.58 Hz
F1PHI -0.110 ppm
F1HI -87.84 Hz
F2PPMCM 0.74049 ppm/cm
F2HZCM 592.24677 Hz/cm
F1PPMCM 0.74147 ppm/cm
F1HZCM 593.02808 Hz/cm
Leu-1 α/Leu-2 β
Om α/Om δ
Leu-2 α/Val α
Ar-6/i-butyric Me
Ar-6/i-butyric CH
Ar-6/linker CH
Leu-1 NH/Om δ
linker-NHen-b/Val α
Leu-2 NH/Leu-1 α
Ar NH/Leu-1 NH
ppm
2
4
6
8
10

800 MHz NOESY Spectrum in CDCl3 at 268 K (-5 C), 4 mM
400 ms mixing time
DEEPER SLICE

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                Current Data Parameters
USER                     slevin
NAME            SL-8-308-800-H
EXPNO                         4
PROCNO                        1

            F2 - Acquisition Parameters
Date_                  20080513
Time                       0.00
INSTRUM                   varian
PROBHD          40:5mmbroadban
PULPROG         noesyh2o_pfg_800_pln
TD                         4468
SOLVENT                    CDCl3
NS                            16
DS                            16
SWH                   12001.200 Hz
FIDRES                 2.686034 Hz
AQ                    0.1861981 sec
RG                           128
DW                        41.662 usec
DE                          6.00 usec
TE                         268.0 K

            F1 - Acquisition parameters
ND0                           1
TD                          920
SFO1                   799.8048 MHz
FIDRES                13.044783 Hz
SW                       15.005 ppm
FnMODE                 undefined

            F2 - Processing parameters
SI                         2048
SF                 799.8002134 MHz
WDW                       QSINE
SSB                           2
LB                         0.00 Hz
GB                            0
PC                         1.40

            F1 - Processing parameters
SI                         1024
MC2                      States
SF                 799.8002097 MHz
WDW                       QSINE
SSB                           2
LB                         0.00 Hz
GB                            0

              2D NMR plot parameters
CX2                       15.00 cm
CX1                       15.00 cm
F2PLO                    11.000 ppm
F2LO                    8797.80 Hz
F2PHI                    -0.100 ppm
F2HI                     -79.98 Hz
F1PLO                    11.000 ppm
F1LO                    8797.80 Hz
F1PHI                    -0.100 ppm
F1HI                     -79.98 Hz
F2PPMCM                 0.74000 ppm/cm
F2HZCM                591.85211 Hz/cm
F1PPMCM                 0.74000 ppm/cm
F1HZCM                591.85211 Hz/cm
```

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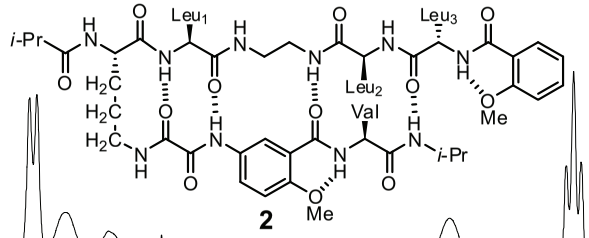
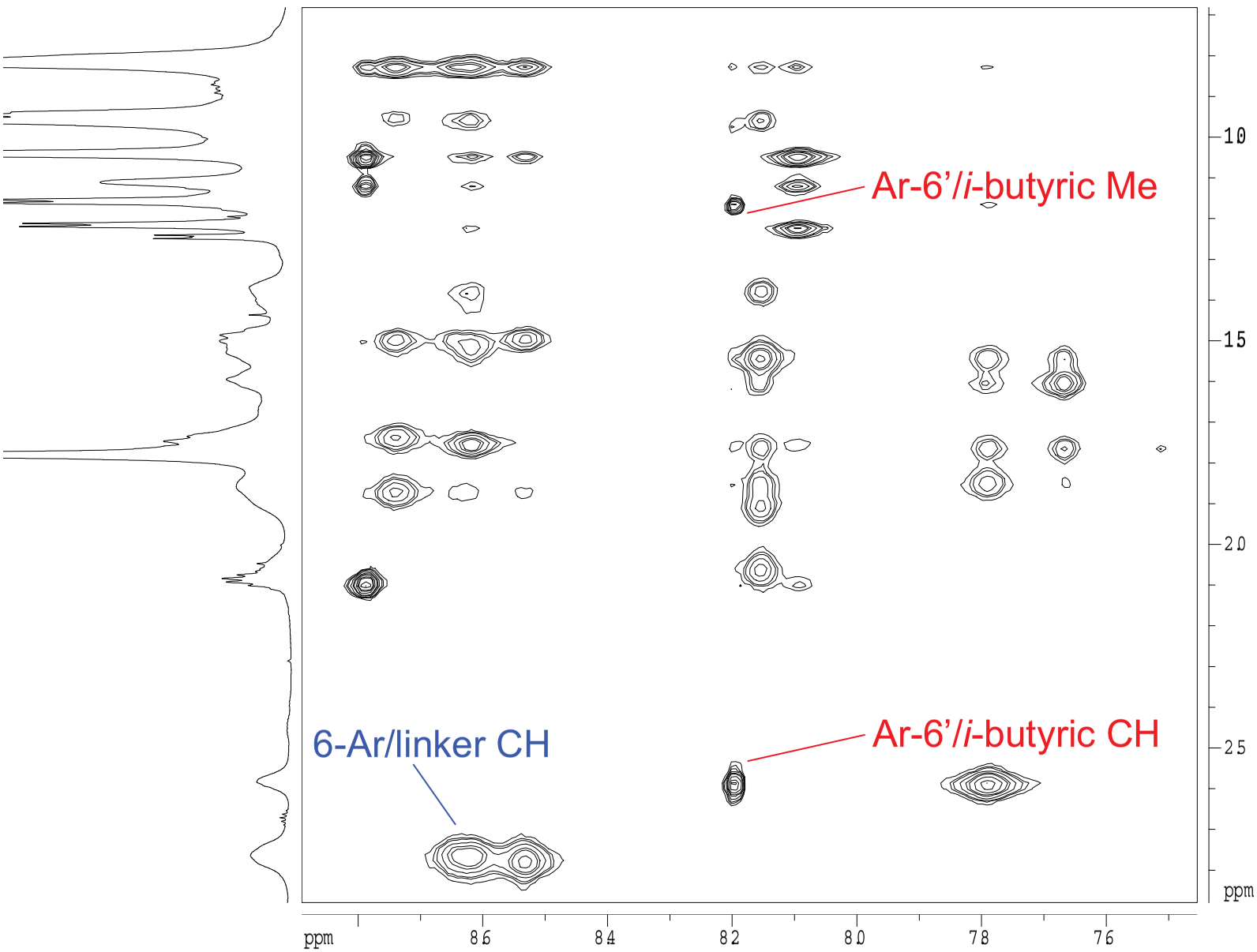
800 MHz NOESY Spectrum in CDCl3 at 268 K (-5 C), 4 mM
400 ms mixing time

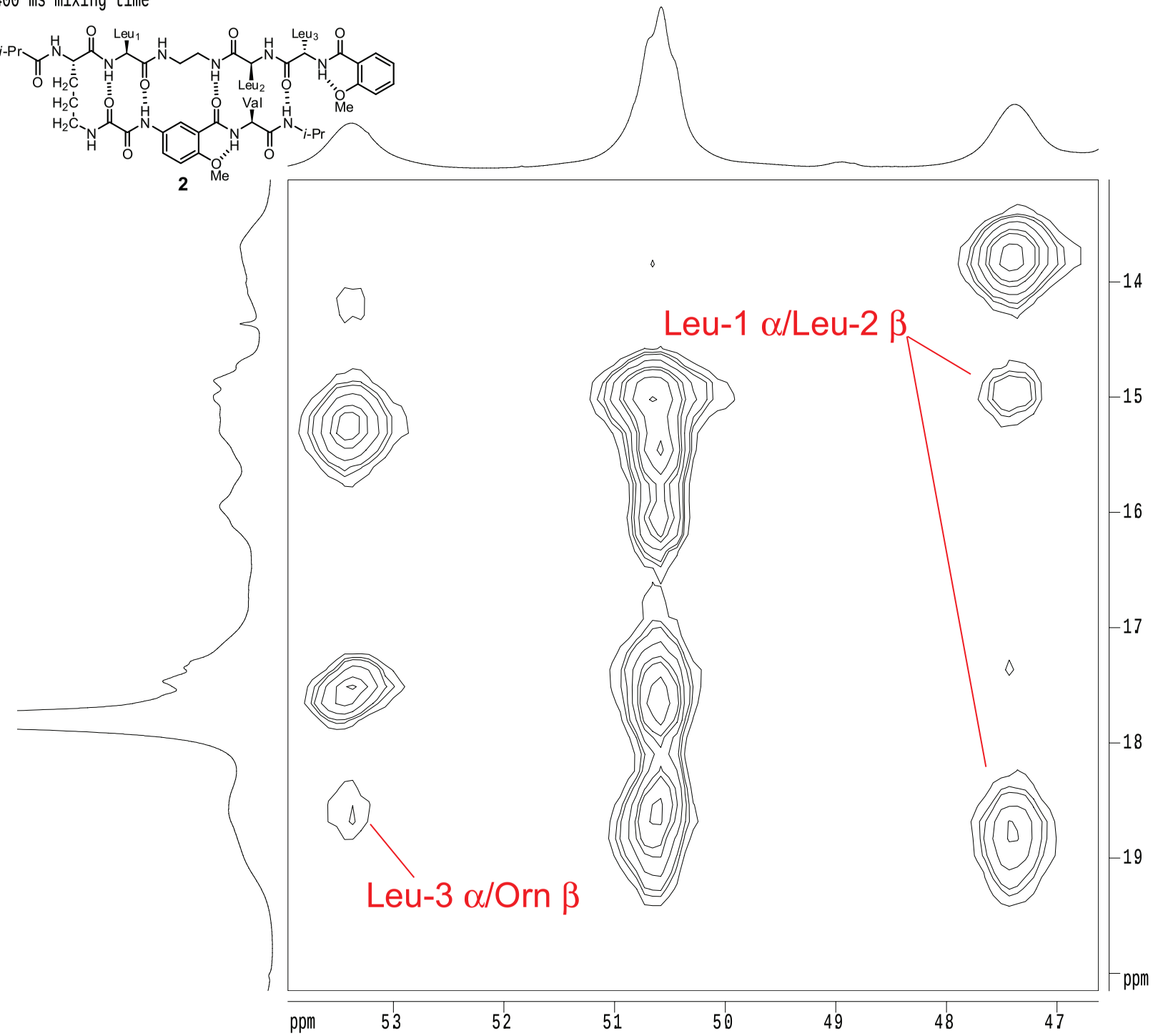





```
          Current Data Parameters
  USER                slevin
  NAME       SL-8-308-800-H
  EXPNO                    4
  PROCNO                   1

        F2 - Acquisition Parameters
  Date_             20080513
  Time                  0.00
  INSTRUM             varian
  PROBHD    40:5mmbroadban
PULPROG   noesyh2o_pfg_800_pln
  TD                    4468
  SOLVENT              CDCl3
  NS                      16
  DS                      16
  SWH             12001.200 Hz
  FIDRES           2.686034 Hz
  AQ              0.1861981 sec
  RG                     128
  DW                  41.662 usec
  DE                    6.00 usec
  TE                   268.0 K

        F1 - Acquisition parameters
  ND0                      1
  TD                     920
  SFO1             799.8048 MHz
  FIDRES          13.044783 Hz
  SW                 15.005 ppm
  FnMODE          undefined

        F2 - Processing parameters
  SI                    2048
  SF            799.8002134 MHz
  WDW                  QSINE
  SSB                      2
  LB                    0.00 Hz
  GB                       0
  PC                    1.40

        F1 - Processing parameters
  SI                    1024
  MC2                 States
  SF            799.8002097 MHz
  WDW                  QSINE
  SSB                      2
  LB                    0.00 Hz
  GB                       0

          2D NMR plot parameters
  CX2                  15.00 cm
  CX1                  15.00 cm
  F2PLO                8.890 ppm
  F2LO               7110.36 Hz
  F2PHI                7.454 ppm
  F2HI               5961.81 Hz
  F1PLO                2.880 ppm
  F1LO               2303.03 Hz
  F1PHI                0.681 ppm
  F1HI                545.04 Hz
  F2PPMCM           0.09574 ppm/cm
  F2HZCM           76.57015 Hz/cm
  F1PPMCM           0.14654 ppm/cm
  F1HZCM          117.19922 Hz/cm
```

S. Levin and James S. Nowick

800 MHz NOESY Spectrum in CDCl3 at 268 K (-5 C), 4 mM
400 ms mixing time




```
          Current Data Parameters
USER                     slevin
NAME            SL-8-308-800-H
EXPNO                         4
PROCNO                        1

        F2 - Acquisition Parameters
Date_                  20080513
Time                       0.00
INSTRUM                   varian
PROBHD          40:5mmbroadban
PULPROG         noesyh2o_pfg_800_pln
TD                         4468
SOLVENT                    CDCl3
NS                            16
DS                            16
SWH                   12001.200 Hz
FIDRES                 2.686034 Hz
AQ                    0.1861981 sec
RG                          128
DW                       41.662 usec
DE                        6.00 usec
TE                       268.0 K

        F1 - Acquisition parameters
ND0                            1
TD                           920
SFO1                   799.8048 MHz
FIDRES                13.044783 Hz
SW                       15.005 ppm
FnMODE                 undefined

        F2 - Processing parameters
SI                          2048
SF                 799.8002134 MHz
WDW                        QSINE
SSB                            2
LB                        0.00 Hz
GB                             0
PC                         1.40

        F1 - Processing parameters
SI                          1024
MC2                       States
SF                 799.8002097 MHz
WDW                        QSINE
SSB                            2
LB                        0.00 Hz
GB                             0

          2D NMR plot parameters
CX2                       15.00 cm
CX1                       15.00 cm
F2PLO                      5.395 ppm
F2LO                     4315.16 Hz
F2PHI                      4.663 ppm
F2HI                     3729.16 Hz
F1PLO                      2.015 ppm
F1LO                     1611.56 Hz
F1PHI                      1.312 ppm
F1HI                     1049.00 Hz
F2PPMCM                  0.04885 ppm/cm
F2HZCM                  39.06640 Hz/cm
F1PPMCM                  0.04689 ppm/cm
F1HZCM                  37.50375 Hz/cm
```

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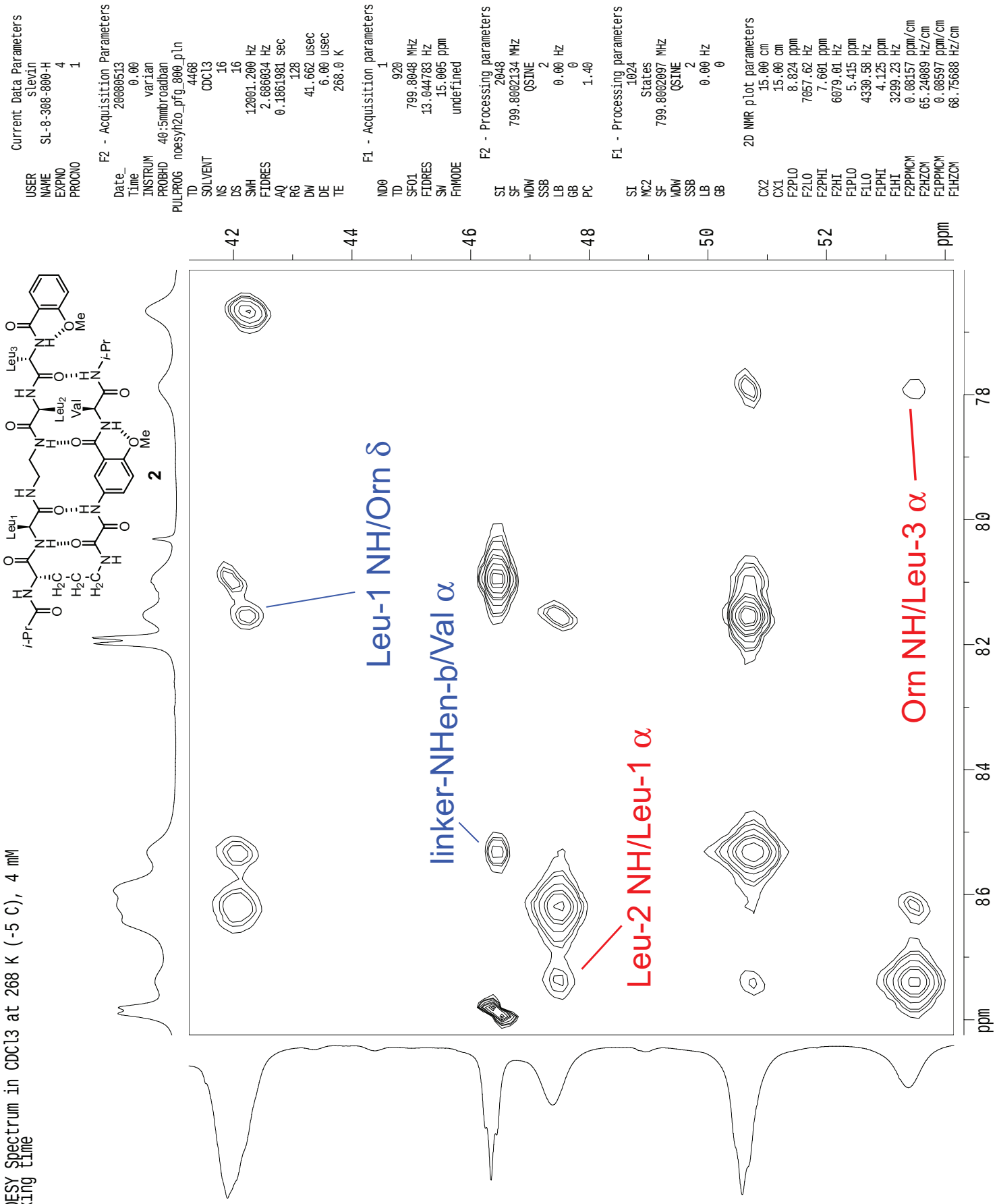
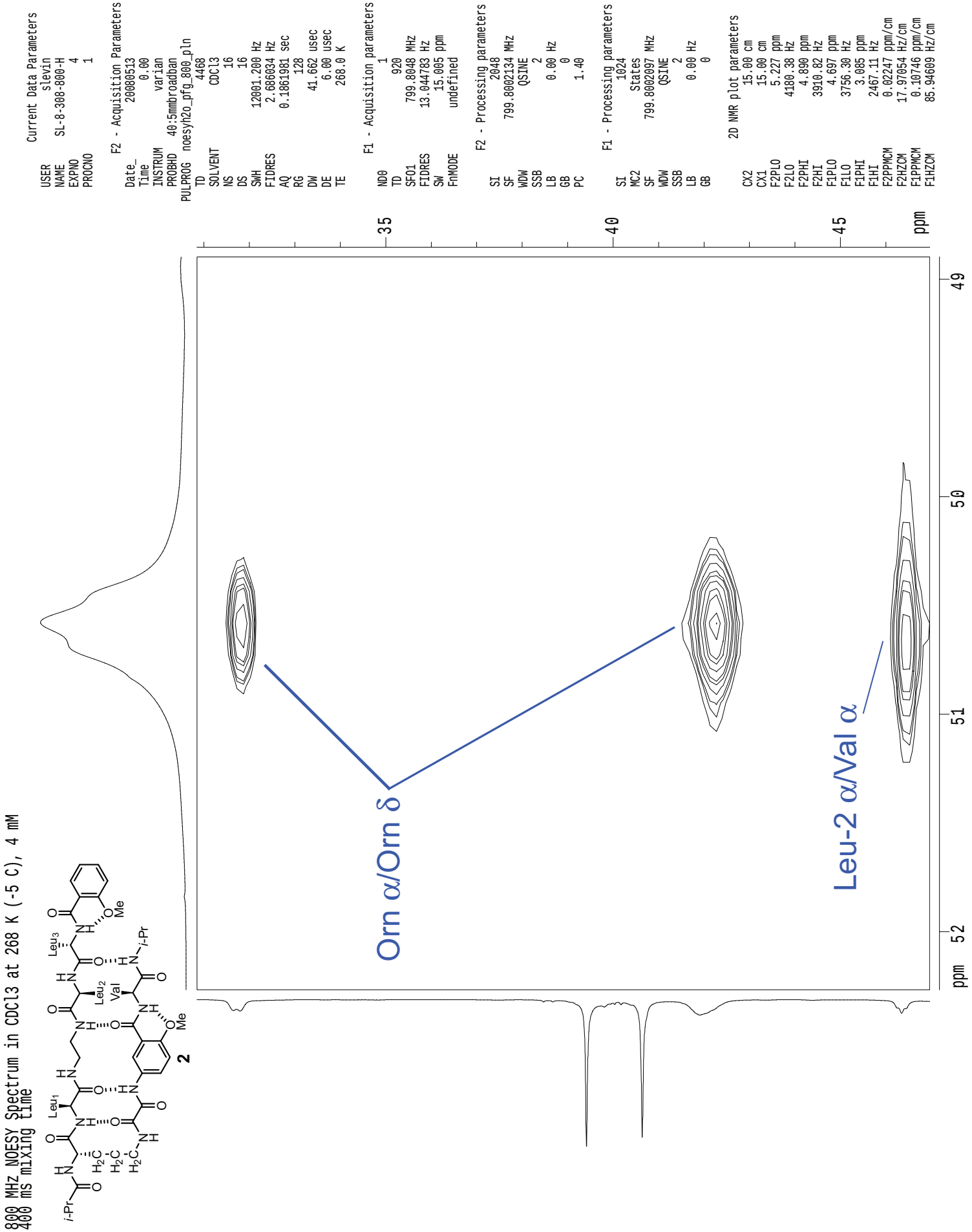
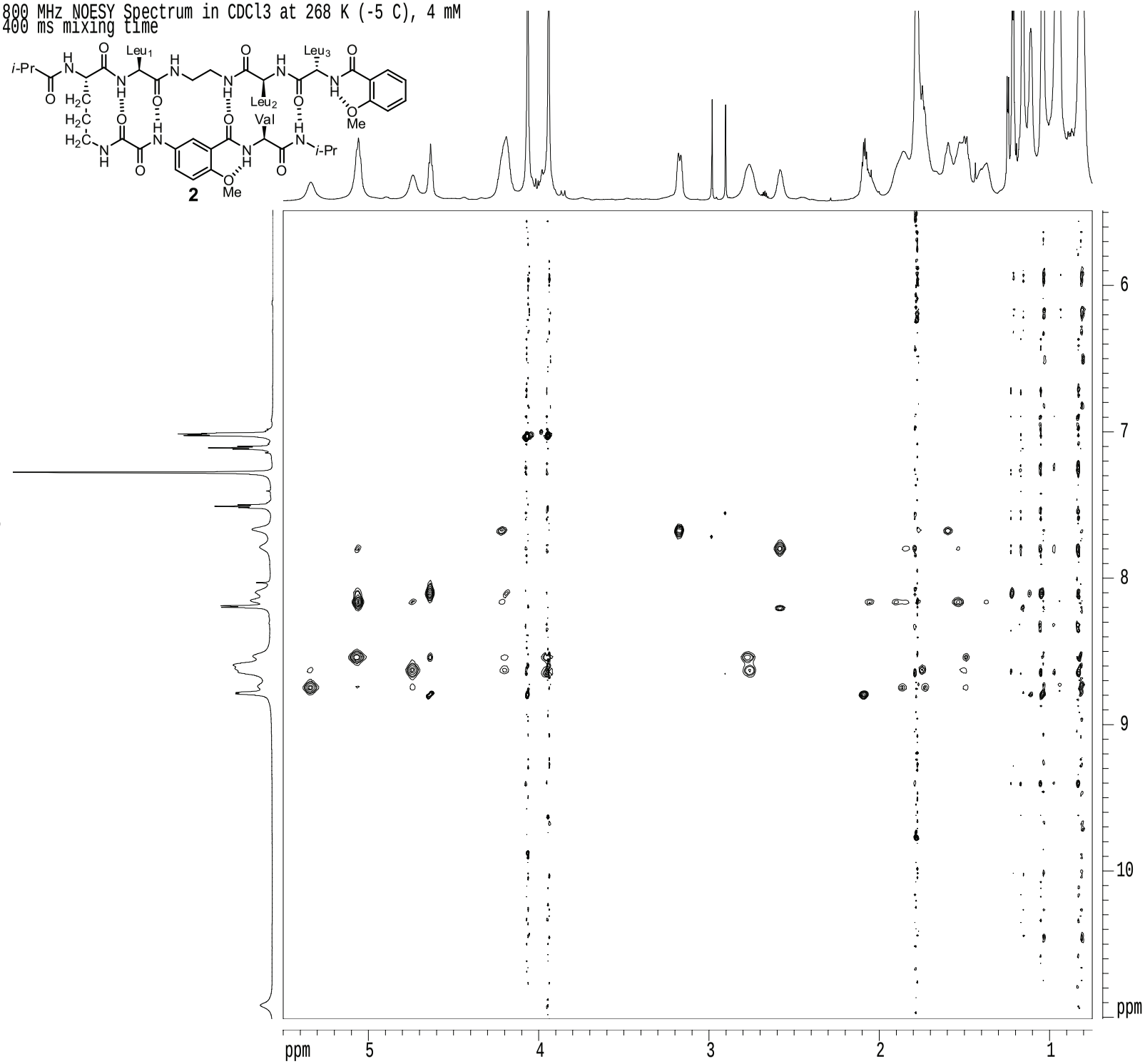

800 MHz NOESY Spectrum in CDCl3 at 268 K (-5 C), 4 mM
400 ms mixing time
Current Data Parameters
slevin
USER SL-8-308-800-H
NAME 4
EXPNO 1
PROCNO
F2 - Acquisition Parameters
20080513
Date_ 0.00
Time varian
INSTRUM 40:5mmbroadban
PROBHD noesyh2o_pfg_800_pln
PULPROG 4468
TD CDCl3
SOLVENT 16
NS 16
DS 12001.200 Hz
SWH 2.686034 Hz
FIDRES 0.1861981 sec
AQ 128
RG 41.662 usec
DW 6.00 usec
DE 268.0 K
TE
F1 - Acquisition parameters
ND0 1
TD 920
SFO1 799.8048 MHz
FIDRES 13.044783 Hz
SW 15.005 ppm
FnMODE undefined
F2 - Processing parameters
SI 2048
SF 799.8002134 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 1024
MC2 States
SF 799.8002097 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 8.824 ppm
F2LO 7057.62 Hz
F2PHI 7.601 ppm
F2HI 6079.01 Hz
F1PLO 5.415 ppm
F1LO 4330.58 Hz
F1PHI 4.125 ppm
F1HI 3299.23 Hz
F2PPMCM 0.08157 ppm/cm
F2HZCM 65.24089 Hz/cm
F1PPMCM 0.08597 ppm/cm
F1HZCM 68.75688 Hz/cm
Leu-1 NH/Orn δ
linker-NHen-b/Val α
Leu-2 NH/Leu-1 α
Orn NH/Leu-3 α
2
ppm


800 MHz NOESY Spectrum in CDCl3 at 268 K (-5 C), 4 mM
400 ms mixing time
Current Data Parameters
USER slevin
NAME SL-8-308-800-H
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080513
Time 0.00
INSTRUM varian
PROBHD 40:5mmbroadban
PULPROG noesyh2o_pfg_800_pln
TD 4468
SOLVENT CDCl3
NS 16
DS 16
SWH 12001.200 Hz
FIDRES 2.686034 Hz
AQ 0.1861981 sec
RG 128
DW 41.662 usec
DE 6.00 usec
TE 268.0 K
F1 - Acquisition parameters
ND0 1
TD 920
SFO1 799.8048 MHz
FIDRES 13.044783 Hz
SW 15.005 ppm
FnMODE undefined
F2 - Processing parameters
SI 2048
SF 799.8002134 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 1024
MC2 States
SF 799.8002097 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 5.227 ppm
F2LO 4180.38 Hz
F2PHI 4.890 ppm
F2HI 3910.82 Hz
F1PLO 4.697 ppm
F1LO 3756.30 Hz
F1PHI 3.085 ppm
F1HI 2467.11 Hz
F2PPMCM 0.02247 ppm/cm
F2HZCM 17.97054 Hz/cm
F1PPMCM 0.10746 ppm/cm
F1HZCM 85.94609 Hz/cm
Orn α/Orn δ
Leu-2 α/Val α
ppm
35
40
45
52
51
50
49
2

800 MHz NOESY Spectrum in CDCl3 at 268 K (-5 C), 4 mM
400 ms mixing time




```
          Current Data Parameters
USER                    slevin
NAME            SL-8-308-800-H
EXPNO                        4
PROCNO                       1

        F2 - Acquisition Parameters
Date_                 20080513
Time                      0.00
INSTRUM                  varian
PROBHD          40:5mmbroadban
PULPROG       noesyh2o_pfg_800_pln
TD                        4468
SOLVENT                   CDCl3
NS                           16
DS                           16
SWH                  12001.200 Hz
FIDRES                2.686034 Hz
AQ                   0.1861981 sec
RG                         128
DW                      41.662 usec
DE                        6.00 usec
TE                       268.0 K

        F1 - Acquisition parameters
ND0                           1
TD                          920
SFO1                   799.8048 MHz
FIDRES                13.044783 Hz
SW                       15.005 ppm
FnMODE                 undefined

        F2 - Processing parameters
SI                         2048
SF                799.8002134 MHz
WDW                       QSINE
SSB                           2
LB                         0.00 Hz
GB                            0
PC                         1.40

        F1 - Processing parameters
SI                         1024
MC2                      States
SF                799.8002097 MHz
WDW                       QSINE
SSB                           2
LB                         0.00 Hz
GB                            0

          2D NMR plot parameters
CX2                       15.00 cm
CX1                       15.00 cm
F2PLO                     5.500 ppm
F2LO                    4398.90 Hz
F2PHI                     0.750 ppm
F2HI                     599.85 Hz
F1PLO                    11.012 ppm
F1LO                    8807.59 Hz
F1PHI                     5.488 ppm
F1HI                    4389.18 Hz
F2PPMCM                 0.31667 ppm/cm
F2HZCM                253.27008 Hz/cm
F1PPMCM                 0.36829 ppm/cm
F1HZCM                294.56073 Hz/cm
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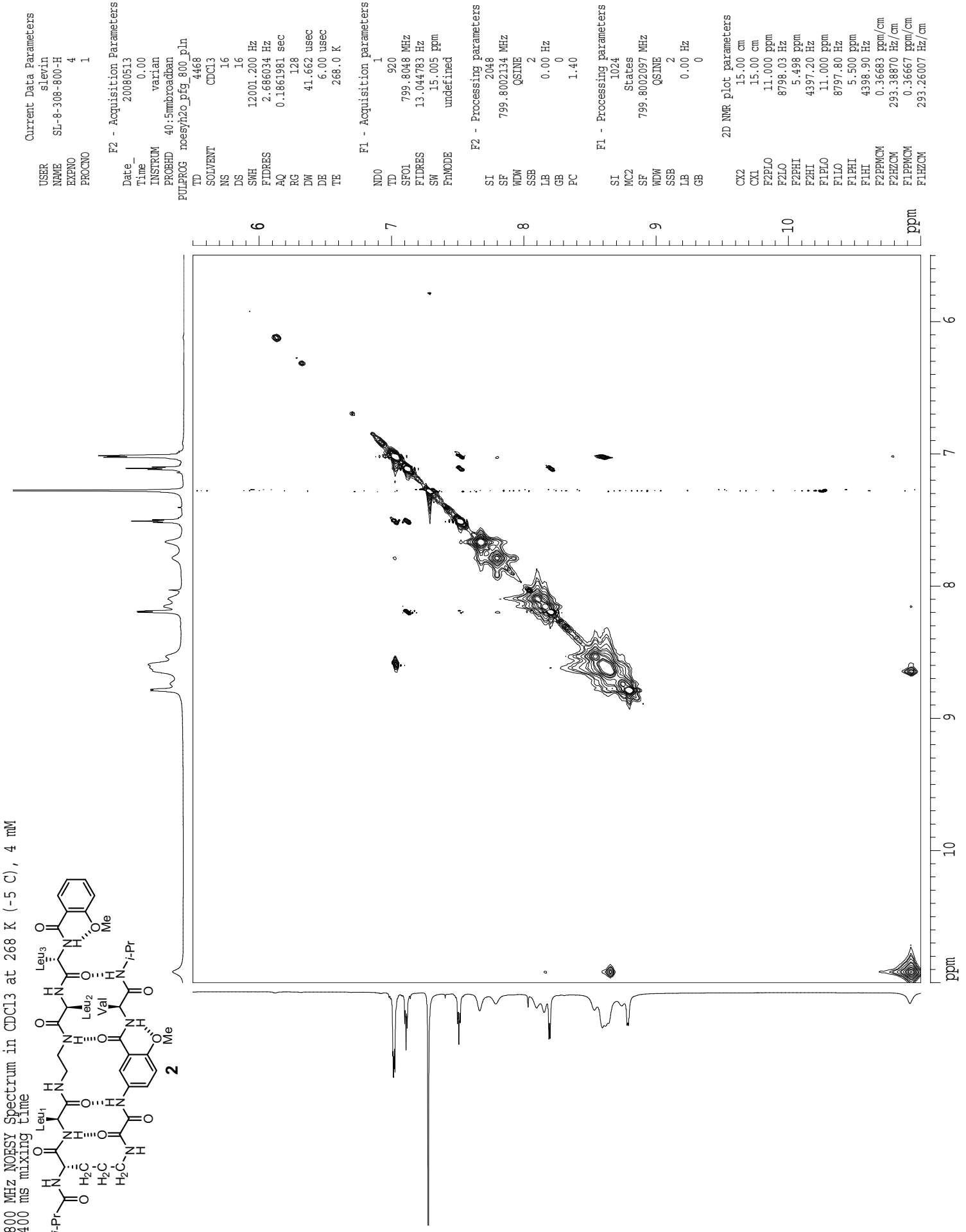
800 MHz NOESY Spectrum in CDCl3 at 268 K (-5 C), 4 mM
400 ms mixing time
2
Leu1
Leu2
Val
Leu3
i-Pr
Me
Current Data Parameters
USER slevin
NAME SL-8-308-800-H
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080513
Time 0.00
INSTRUM varian
PROBHD 40:5mmbroadban
PULPROG noesyh2o.pfg_800_pln
TD 4468
SOLVENT CDCl3
NS 16
DS 16
SWH 12001.200 Hz
FIDRES 2.686034 Hz
AQ 0.1861981 sec
RG 128
DW 41.662 usec
DE 6.00 usec
TE 268.0 K
F1 - Acquisition parameters
ND0 1
TD 920
SFO1 799.8048 MHz
FIDRES 13.044783 Hz
SW 15.005 ppm
FnMODE undefined
F2 - Processing parameters
SI 2048
SF 799.8002134 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 1024
MC2 States
SF 799.8002097 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 11.000 ppm
F2LO 8798.03 Hz
F2PHI 5.498 ppm
F2HI 4397.20 Hz
F1PLO 11.000 ppm
F1LO 8797.80 Hz
F1PHI 5.500 ppm
F1HI 4398.90 Hz
F2PPMCM 0.36683 ppm/cm
F2HZCM 293.38870 Hz/cm
F1PPMCM 0.36667 ppm/cm
F1HZCM 293.26007 Hz/cm
ppm
6
7
8
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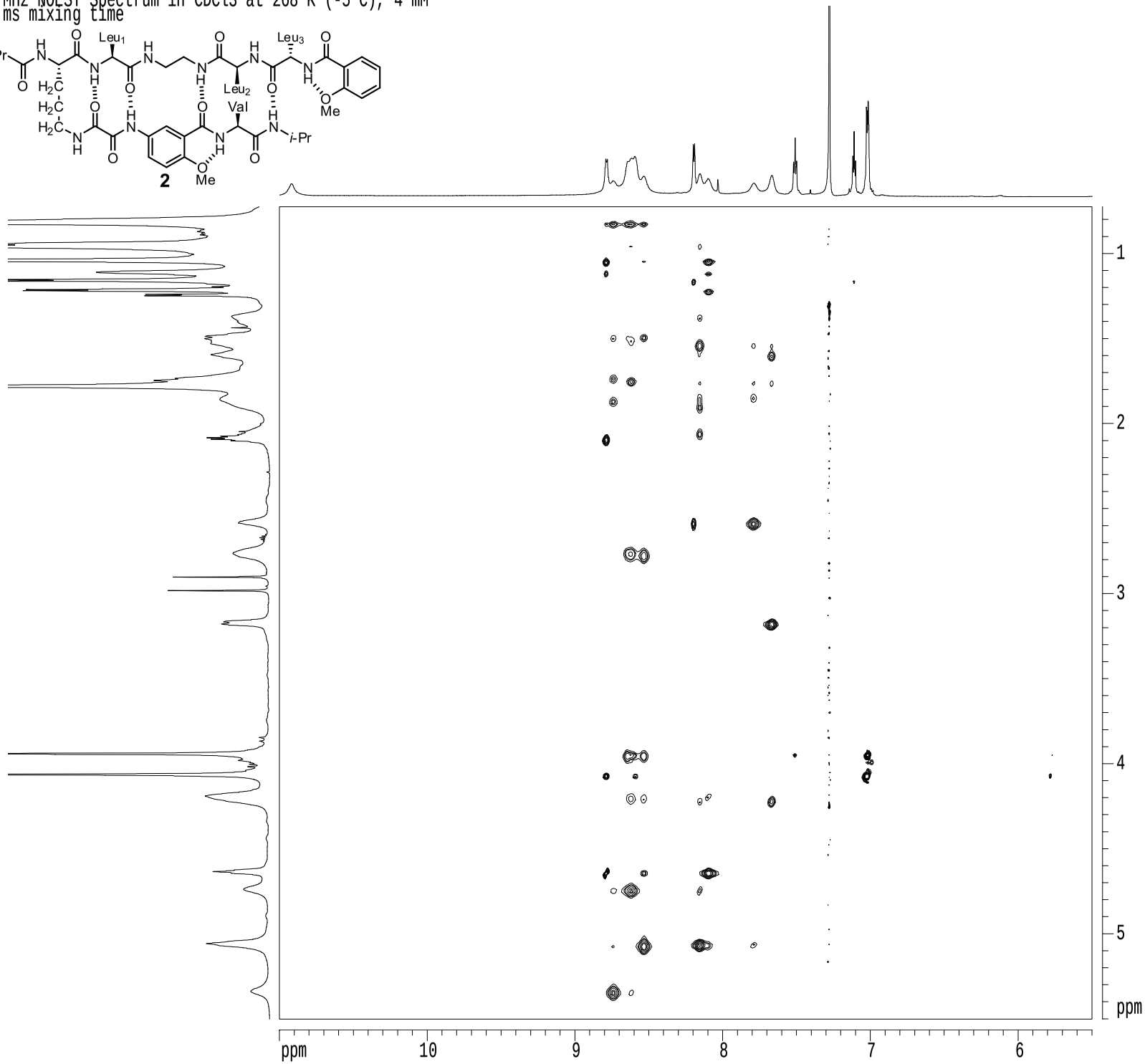
800 MHz NOESY Spectrum in CDCl3 at 268 K (-5 C), 4 mM
400 ms mixing time

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        Current Data Parameters
USER              slevin
NAME      SL-8-308-800-H
EXPNO                  4
PROCNO                 1

      F2 - Acquisition Parameters
Date_           20080513
Time               0.00
INSTRUM           varian
PROBHD   40:5mmbroadban
PULPROG  noesyh2o_pfg_800_pln
TD                  4468
SOLVENT            CDCl3
NS                    16
DS                    16
SWH            12001.200 Hz
FIDRES          2.686034 Hz
AQ             0.1861981 sec
RG                   128
DW                41.662 usec
DE                  6.00 usec
TE                 268.0 K

      F1 - Acquisition parameters
ND0                    1
TD                   920
SFO1            799.8048 MHz
FIDRES         13.044783 Hz
SW                15.005 ppm
FnMODE         undefined

      F2 - Processing parameters
SI                  2048
SF           799.8002134 MHz
WDW                 QSINE
SSB                     2
LB                   0.00 Hz
GB                      0
PC                   1.40

      F1 - Processing parameters
SI                  1024
MC2                States
SF           799.8002097 MHz
WDW                 QSINE
SSB                     2
LB                   0.00 Hz
GB                      0

        2D NMR plot parameters
CX2                 15.00 cm
CX1                 15.00 cm
F2PLO              11.000 ppm
F2LO              8797.80 Hz
F2PHI              5.500 ppm
F2HI              4398.90 Hz
F1PLO              5.502 ppm
F1LO              4400.90 Hz
F1PHI              0.725 ppm
F1HI               580.20 Hz
F2PPMCM          0.36667 ppm/cm
F2HZCM         293.26007 Hz/cm
F1PPMCM          0.31847 ppm/cm
F1HZCM         254.71297 Hz/cm
```

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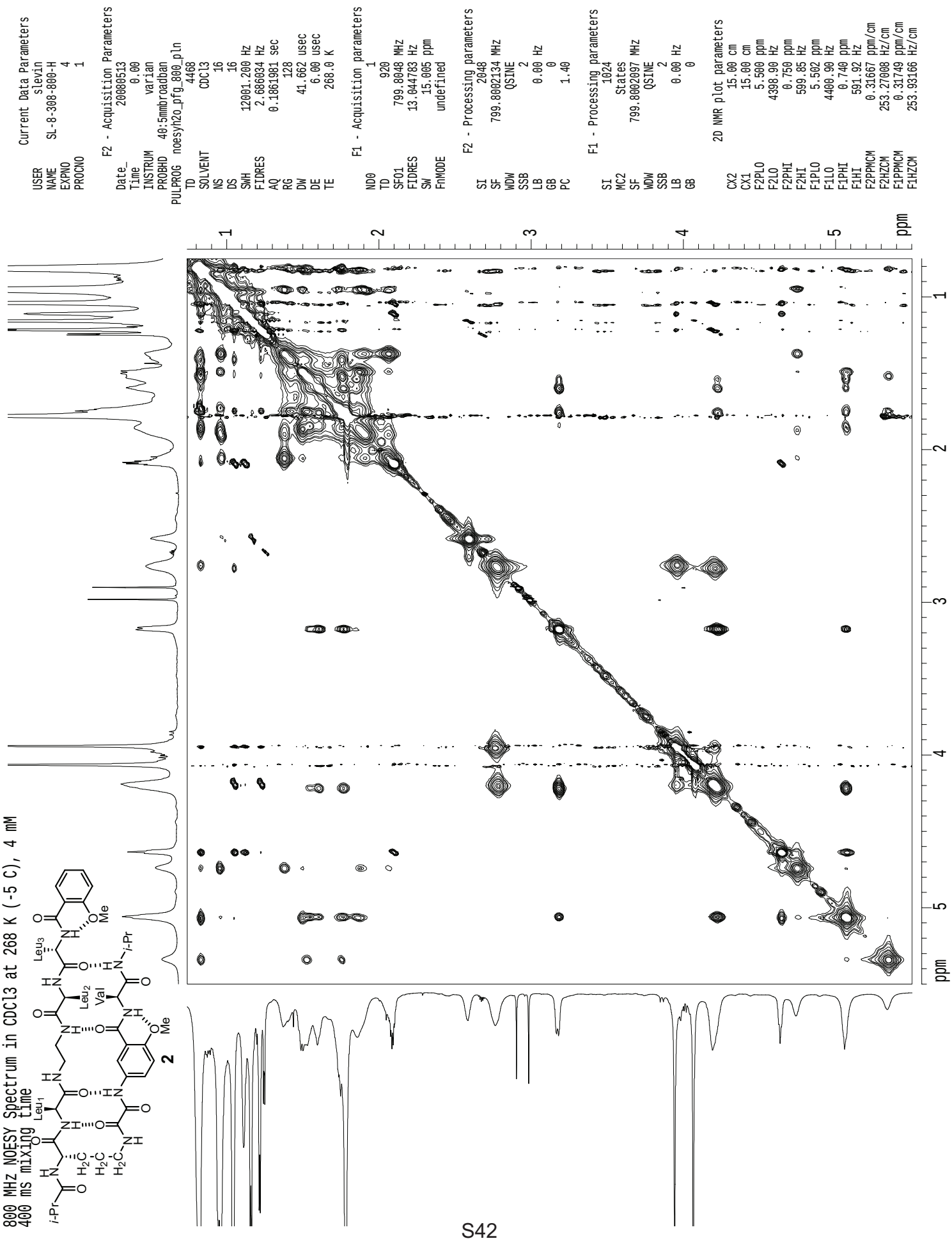
800 MHz NOESY Spectrum in CDCl3 at 268 K (-5 C), 4 mM
400 ms mixing time
Current Data Parameters
USER slevin
NAME SL-8-308-800-H
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080513
Time 0.00
INSTRUM varian
PROBHD 40:5mmbroadban
PULPROG noesyh2o_pfg_800_pln
TD 4468
SOLVENT CDCl3
NS 16
DS 16
SWH 12001.200 Hz
FIDRES 2.686634 Hz
AQ 0.1861981 sec
RG 128
DW 41.662 usec
DE 6.00 usec
TE 268.0 K
F1 - Acquisition parameters
ND0 1
TD 920
SFO1 799.8048 MHz
FIDRES 13.044783 Hz
SW 15.005 ppm
FnMODE undefined
F2 - Processing parameters
SI 2048
SF 799.8002134 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 1024
MC2 States
SF 799.8002097 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 5.500 ppm
F2LO 4398.90 Hz
F2PHI 0.750 ppm
F2HI 599.85 Hz
F1PLO 5.502 ppm
F1LO 4400.90 Hz
F1PHI 0.740 ppm
F1HI 591.92 Hz
F2PPMCM 0.31667 ppm/cm
F2HZCM 253.27008 Hz/cm
F1PPMCM 0.31749 ppm/cm
F1HZCM 253.93166 Hz/cm
ppm
1
2
3
4
5
Leu1
Leu2
Leu3
Val
i-Pr
Me
2