

Asymmetric Synthesis of Telcagepant, a CGRP Receptor Antagonist for the Treatment of Migraine

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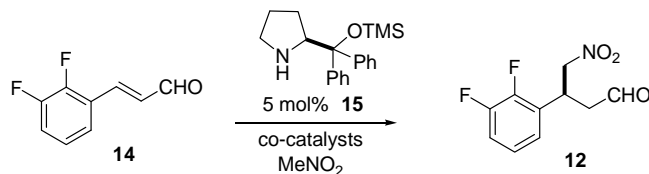
Supporting Information

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1. Enantioselective 1,4-addition of MeNO₂: Optimization, mechanistic consideration, and plausible pathways for the formation of byproducts

Table S-1. Selected Results of Asymmetric 1,4-Addition of MeNO₂^a



Entry	Co-catalysts	MeNO ₂ (equiv)	Solvents	Conditions	Conversion ^b (%)	Yield ^b (%)	ee ^b (%)	Byproducts (%)
1	20 mol% PhCO ₂ H	6	MeOH	10 °C, 17 h	99	~50-75	95	16a (varied from 2-20%), 18 (<0.5%), 19 (1%), 20 (2%)
2	10 mol% PhCO ₂ H	6	<i>i</i> -PrOH	20 °C, 25 h	98	~55-70	94	16b (varied from 1-15%), 18 (<1%), 19 (1%), 20 (7.9%)
3	10 mol% PhCO ₂ H	6	MeCN	20 °C, 14 h	99	~20	N/A	18 (<1%), 19 (3.9%), 20 (72%)
4	–	3	10% H ₂ O-THF	20 °C, 63 h	96	58	95	18 (<1%), 20 (2.9%)
5	10 mol% PhCO ₂ H	6	25% H ₂ O-THF	20 °C, 14 h	31	N/A	N/A	
6	15 mol% <i>t</i> -BuCO ₂ H	6	25% H ₂ O-THF	20 °C, 14 h	93.7	65	96	18 (<1%), 19 (1.0%), 20 (7.2%)
7	100 mol% B(OH) ₃	5	25% H ₂ O-THF	20 °C, 67 h	85	61	95	18 (<1%), 19 (1%), 20 (0.7%)
8	5 mol% <i>t</i> -BuCO ₂ H, 50 mol% B(OH) ₃	6	17% H ₂ O-THF	20 °C, 29 h	94	73	95	18 (<0.1%), 19 (1.0%), 20 (2.8%)
9	5 mol% <i>t</i> -BuCO ₂ H, 25 mol% B(OH) ₃	6	25% H ₂ O-THF	20 °C, 23 h	88	72	95	18 (<0.1%), 19 (0.8%), 20 (4.4%)
10	5 mol% <i>t</i> -BuCO ₂ H, 100 mol% B(OH) ₃	6	25% H ₂ O-THF	20 °C, 23 h	92	71	95	18 (<0.1%), 19 (0.9%), 20 (2.9%)
11	5 mol% <i>t</i> -BuCO ₂ H, 50 mol% B(OH) ₃	4	25% H ₂ O-THF	20 °C, 22 h	97	66	95	18 (<0.1%), 19 (0.9%), 20 (3.5%)
12	5 mol% <i>t</i> -BuCO ₂ H, 50 mol% B(OH) ₃	8	25% H ₂ O-THF	20 °C, 39 h	96	74	95	18 (<0.1%), 19 (1.6%), 20 (2.9%)
13	5 mol% LiOAc,	3	10% H ₂ O-THF	20 °C, 24 h	72	16	N/A	18 (17%), 20 (5.7%)

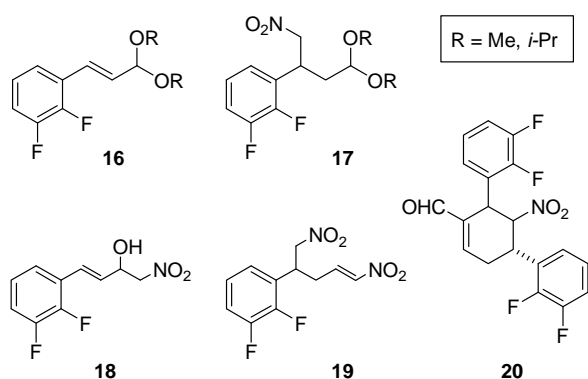
^a All the reactions were carried out in the presence of 5 mol% of **15**. ^b The enantiomeric excess (ee) was determined by high performance liquid chromatography (HPLC) analysis: ChiralPak AD-RH column, 4.6 x 150 mm, 5 μm particle size, 45 °C, isocratic 0.1% aq H₃PO₄/MeCN/ MeOH (30:45:25); flow rate: 0.5 mL/min. The yield and Z:E ratio were determined by high performance liquid chromatography (HPLC) analysis: Ascentis Express C18 column, 4.6 x 100 mm, 2.7 μm particle size, 60 °C, mobile phase: 0.1% aq HClO₄/MeCN; flow rate: 2.5 mL/min. HPLC sample preparation: **12** was converted to the corresponding hydrazone by treatment with 2,4-dinitrophenylhydrazine in aq H₃PO₄-MeCN at 40 °C for 5 min.

The use of *t*-BuCO₂H led to a higher reaction rate and conversion; however, overcharging *t*-BuCO₂H had an adverse effect on the stability of product **12**, giving rise to a higher level of impurities including **20**. In contrast, the use of B(OH)₃ alone resulted in a much slower reaction

rate. Interestingly, the use of a combination of $B(OH)_3$ and $t\text{-BuCO}_2H$ allowed for reducing the $t\text{-BuCO}_2H$ charge from 15 mol% to 5 mol% without sacrificing the reaction rate or conversion, while the formation of impurities, especially **20**, was successfully suppressed to improve the yield significantly.

In addition, with a reduced amount of nitromethane (4 equiv), the reaction yield was decreased to 66%. Increasing the nitromethane charge to 8 equiv improved the yield to 74%; however, the reaction became sluggish. As a result, the use of a combination of proper weak acids $t\text{-BuCO}_2H$ (5 mol%)/ $B(OH)_3$ (50 mol%) allowed for the balance of the reaction efficiency and product stability. The formation of the byproducts, such as **19** and **20**, was thus suppressed significantly.

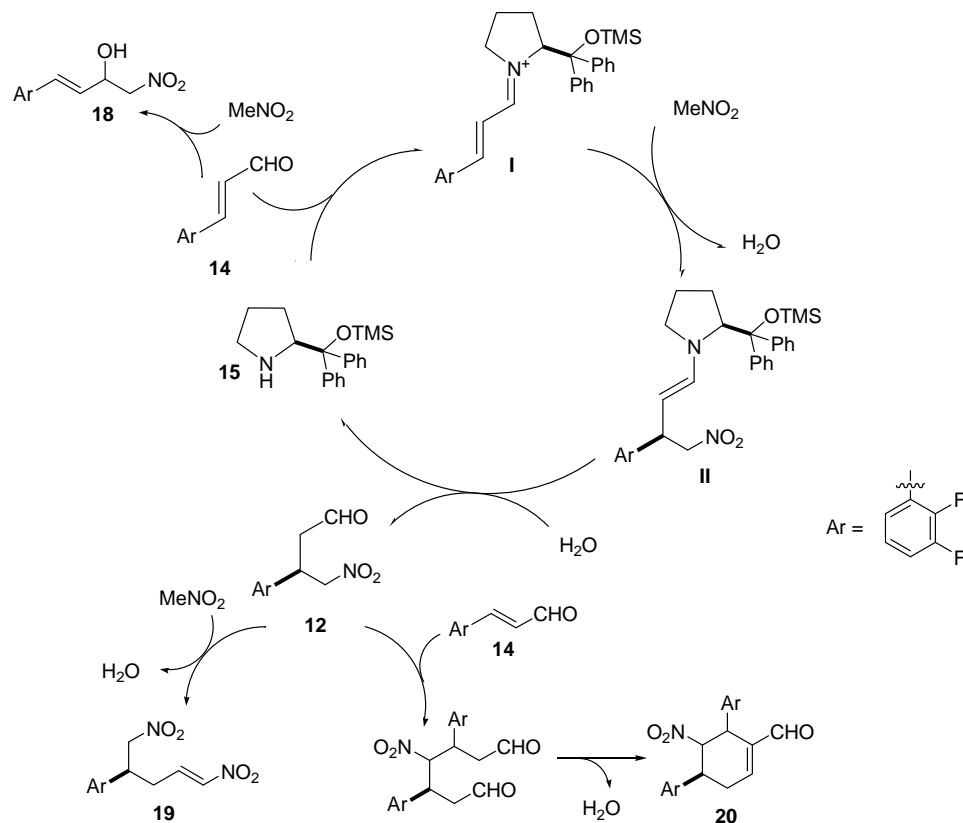
Figure S-1. Identified Byproducts Formed During the Asymmetric Addition of MeNO_2 to **14**.



Many low level impurities were formed during the reaction. The impurity profile varied as the reactions were changed. Several significant impurities were isolated and characterized (Figure S-1). The plausible pathways for the formation of the desired aldehyde product **12** as well as byproducts **16-20** are depicted in Scheme S-1.

The formation of alcohol **18** could be via 1.2 addition of MeNO_2 to **14**. **19** could be a product of further condensation of the desired product **12** with MeNO_2 . Similarly, the desired aldehyde product **12** could react with starting aldehyde **14** or the intermediate **II** followed by a intramolecular condensation to give **20**, which was the major byproduct. The use of the combination of $t\text{-BuCO}_2H/B(OH)_3$ significantly suppressed the formation of these byproducts and improved the reaction rate. The function of the co-catalysts $t\text{-BuCO}_2H/B(OH)_3$ is also believed to accelerate both of the formation of iminium ion intermediate **I** and hydrolysis of the enamine intermediate **II**.

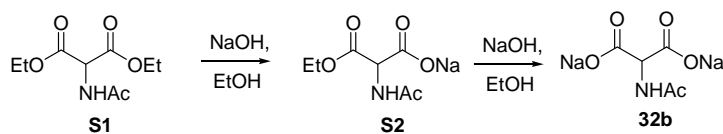
Scheme S-1. Plausible Pathways to Form Aldehyde **12** as well as Byproducts **16-20**



Various catalysts were screened to identify an optimal catalyst for this transformation. For example, modification on the structure on **15**, including replace the TMS group with TBS or TES and studying the electronic effects as well as the steric effects by replacing the phenyl group with various aromatic rings, was explored. Other types of organocatalysts outside the pyrrolidine skeleton were also screened. To date, the use of TMS-prolinol **15** was optimal.

2. Preparation of sodium 2-acetamidomalonate (**32b**)

Scheme S-2. Preparation of Sodium 2-Acetamidomalonate (**32b**)



The literature procedure for the preparation of acid **32a** by hydrolysis of ethyl 2-acetamidomalonate (**S1**) in EtOH is not ideal.^{S1} The isolation of **32a** was laborious and the purity of the isolated solid was poor. Treatment of **S1** with 2 equiv of NaOH (50 wt% aqueous solution) in EtOH under literature conditions^{S1} gave the corresponding sodium salt as an

^{S1} For example, see: (a) Hellmann, H.; Teichmann, K.; Lingens, F. *Chem. Ber.* **1958**, *91*, 2427. (b) Hellmann, H.; Olson, W. H., Jr.; Piechota, H. *J. Liebigs Annalen der Chemie* **1960**, *631*, 76.

amorphous slurry, which was then treated with 2 equiv of 37% HCl. As such, the di-acid **32a** could be dissolved in EtOH as NaCl precipitated in this "slurry-to-slurry" operation. The NaCl was removed through filtration and the resulting EtOH solution of **32a** was concentrated to remove EtOH and H₂O, and solvent-switched to *i*-PrOAc in order to crystallize acid **32a**. Unfortunately, **32a** was not suitable for large scale preparation due to safety concerns of its decomposition. In contrast, the corresponding di-sodium salt **32b** is quite stable.

However, the amorphous di-Na salt **32b** obtained under literature conditions^{S1} was hygroscopic and contaminated with both NaOH residue and the sodium salt of the mono acid **S2**. Fortunately, we found that the dihydrate of the di-sodium salt **32b** is a highly crystalline, non-hygroscopic solid. Thus, saponification of **S1** was carried out by using 5N aq NaOH. The resulting dihydrate **32b** was directly crystallized with high quality control from aq MeOH during the reaction. The process was scaled up to 100 kg scale and gave 98.6% isolated yield.

The di-Na salt **32b** is a stable solid, which has no safety concerns regarding its preparation or storage. In order to dehydrate **32b** dihydrate efficiently, it is necessary and crucial to dry the dihydrate in a 70-80 °C vacuum oven *with dry N₂ sweeping*.

Sodium 2-acetamidomalonate dihydrate (32b). To a solution of NaOH (40.5 g, 1.013 mol) in water (155 mL) and MeOH (5 vol, 500 mL) at 40-45 °C was added a solution of malonate (100 g, 0.46 mol) in MeOH (3 vol, 300 mL) dropwise. The batch was seeded (~100 mg seeds) right after ~15% of the SM MeOH solution (~75 mL) was added over 30 min dropwise. The seeded batch was aged 1 h at 40-45 °C. Then, the rest of the methanol solution was added over 2 h at 40-45 °C. After 30 min age, by LC no SM was left. The batch was gradually cooled to ambient temperature and aged 2 h before filtration. The wet cake was displacement washed with 5% water in MeOH (200 mL) followed by a slurry wash with 5% water in MeOH (200 mL, 2 vol). Suction dry gave 109 g of di Na salt dihydrate **32b**. 98% yield. ¹H-NMR (400 MHz, D₂O): δ 4.64 (s, 1 H), 2.12 (s, 3 H). ¹³C-NMR (100 MHz, D₂O): δ 174.2, 173.0, 62.0, 22.0. Anal calc'd for C₅H₉NNa₂O₇: C, 24.91; H, 3.76; N, 5.81. Found: C, 24.97; H, 3.47; N, 5.82.

3. Hydrogenation of acid 34a

As the reaction proceeds, the corresponding hydrogenation intermediates, shown in Scheme S-3, could be observed by HPLC (Ascentis Express C18, 4.6x100 mm, 2.7 µm particle size, 40 °C, mobile phase: 0.1% HClO₄/MeCN; flow rate: 2.0 mL/min.).

Scheme S-3. Hydrogenation of **34b** *n*-Bu₃N Salt in the Presence of 10% Pd(OH)₂-C and H₂SO₄ in *i*-PrOH

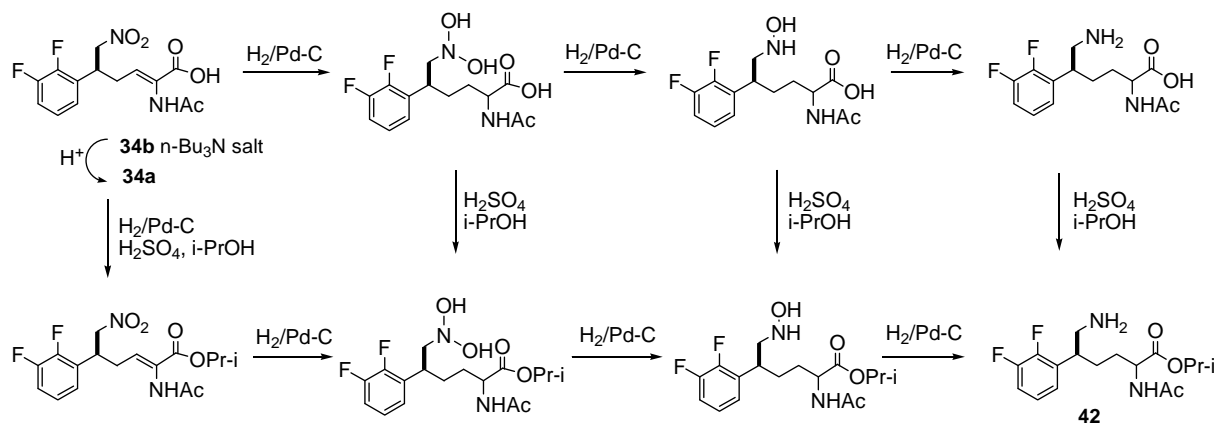
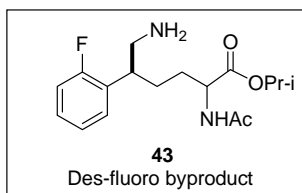


Table S-2. Selected Results for Optimization of Hydrogenation in Attempts to Minimize Defluorination.^a

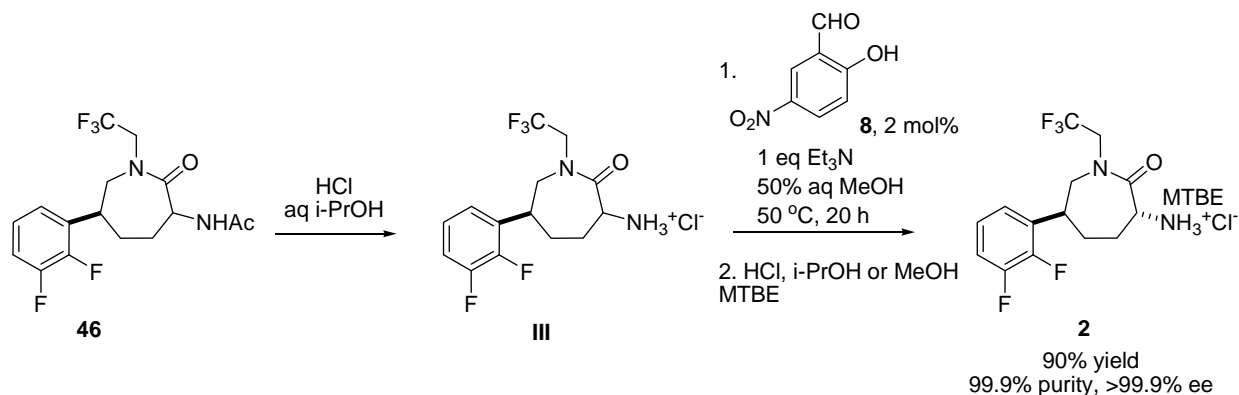
Entry	Catalysts (10 wt% loading)	Additives	H ₂ (psig)	Temp (°C)	Isomers of 43 (%)	Conversion (%)
1	10% Pd(OH) ₂ -C with 50% water	—	50	50	1.06-2.5%	>99
2		—	100	35	1.06-2.5	>98
3		LiCl, 0.25 equiv	100	50	0.05, 0.13	98
4		<i>LiCl</i> , 0.3 equiv	75	35	0.03, 0.09	98 ^d
5		<i>LiCl</i> , 0.3 equiv	100	35	0.03, 0.09	98
6		35% HCl, 0.3 equiv	100	35	0.06, 0.11	97
7		<i>n</i> -Bu ₃ NH ⁺ Cl ⁻ , 0.3 equiv	100	35	0.04, 0.08	94
8		Et ₃ NH ⁺ Cl ⁻ , 0.3 equiv	100	35	0.07, 0.08	92
9		ZnCl ₂ , 0.5 equiv	100	50	0.28, 0.33	95
10		NaCl, 0.5 equiv	100	50	0.26, 0.52	99
11		CuCl, 0.5 equiv	100	50	Complicated reaction profile	
12		CsF, 0.5 equiv	100	50	0.8, 1.2	90
13	5% Pd/C shell	LiCl, 0.5 equiv	100	50	<0.02, <0.02	65
14	Pd/BaSO ₄	LiCl, 1.0 equiv	100	50	<0.02, <0.02	20

^a Unless otherwise mentioned, hydrogenation of **34b** *n*-Bu₃N salt was carried out in the presences of H₂SO₄ (2.5 equiv) and 10wt % of 10% Pd(OH)₂-C in *i*-PrOH. Full conversion was typically obtained after 20 h.



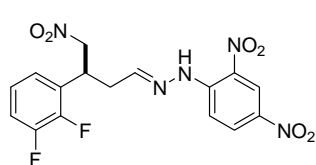
4. Alternative preparation of caprolactam **2** via an epimerization catalyzed by aldehyde **8**

Scheme S-4. Alternative Preparation of Caprolactam via Epimerization of **III**



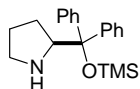
An alternative preparation of caprolactam **2** via epimerization of **S3** is depicted in Scheme S-4. A mixture of cis/trans isomers **46** was hydrolyzed to give a mixture of diastereomers **III**, which were then isolated as the corresponding HCl salt. Epimerization of the deacylated caprolactam epimers **III** in the presence of 2 mol% ArCHO in aq MeOH was very efficient and gave a ratio of 97:3 (trans/cis) in the crude reaction solution, although this epimerization was previously carried out in dry MeOH in the presence of 10 mol% aldehyde **8**.⁹ The final isolated HCl salt MTBE solvate **2** from 20% MTBE in *i*-PrOH (preferred) or 70% MTBE in MeOH gave 99.9 A% purity of HCl salt **2** MTBE solvate with no impurity >0.1A% and 100% ee.

5. Experimental



(S,E)-1-(3-(2,3-difluorophenyl)-4-nitrobutylidene)-2-(2,4-dinitrophenyl)hydrazine. Aldehyde **12** (1 equiv) could be treated with 2,4-dinitrophenylhydrazine (1.4 equiv) in the presence of 15 mol% of HOAc in MeCN at ambient temperature overnight to give the corresponding hydrazone derivative, which could be isolated as a

stable crystalline solid from heptane/EtOAc (1:4) for further characterization. ¹H-NMR (400 MHz, *d*₆-DMSO): δ 11.33 (s 1H), 8.80 (d, *J* = 2.6 Hz, 1 H), 8.29 (dd, *J* = 9.6, 2.7 Hz, 1 H), 7.93 (t, *J* = 9.9 Hz, 1 H), 7.69 (d, *J* = 9.6 Hz, 1 H), 7.38 (m, 1 H), 7.31 (m, 1 H), 7.23 (m, 1 H), 5.09 (dd, *J* = 13.6, 5.9 Hz, 1 H), 5.01 (dd, *J* = 13.6, 9.4 Hz, 1 H), 4.20 (m, 1 H), 2.83 (m, 2 H). ¹³C-NMR (100 MHz, *d*₆-DMSO): δ 150.9, 149.7 (dd, *J* = 245.6, 13.2 Hz), 148.1 (dd, *J* = 246.4, 12.9 Hz), 144.5, 136.7, 129.6, 129.0 (d, *J* = 10.4 Hz), 128.9, 125.1 (dd, *J* = 7.2, 4.8 Hz), 124.1 (t, *J* = 3.0 Hz), 122.9, 116.4 (d, *J* = 16.8 Hz), 116.0, 78.1, 34.7, 33.8. ¹⁹F NMR (376 MHz, *d*₆-DMSO): δ -138.6 (d, *J* = 22.6 Hz), -143.2 (d, *J* = 22.6 Hz). Anal calc'd for C₁₆H₁₃F₂N₅O₆: C, 46.95; H, 3.20; N, 17.11. Found: C, 46.81; H, 3.01; N, 16.95.



(S)-2-(Diphenyl(trimethylsilyloxy)methyl)pyrrolidine (15). To a slurry of prolinol **24** (10 kg, 39.5 mol) and imidazole (4.57 kg, 67.1 mol) in THF (100 L) was added chlorotrimethylsilane (5.57 kg, 51.3 mol) dropwise over 1 h while the

internal temperature was maintained below 30 °C with external cooling. The resulting slurry was then agitated at 50 °C for additional 5 h. The batch was cooled to ambient temperature and quenched by addition of MTBE (50 L) and 15% NaCl (100 L). The organic phase was washed with 15% NaCl (50 L). HPLC assay: 12.47 kg of **15**. 98% yield.

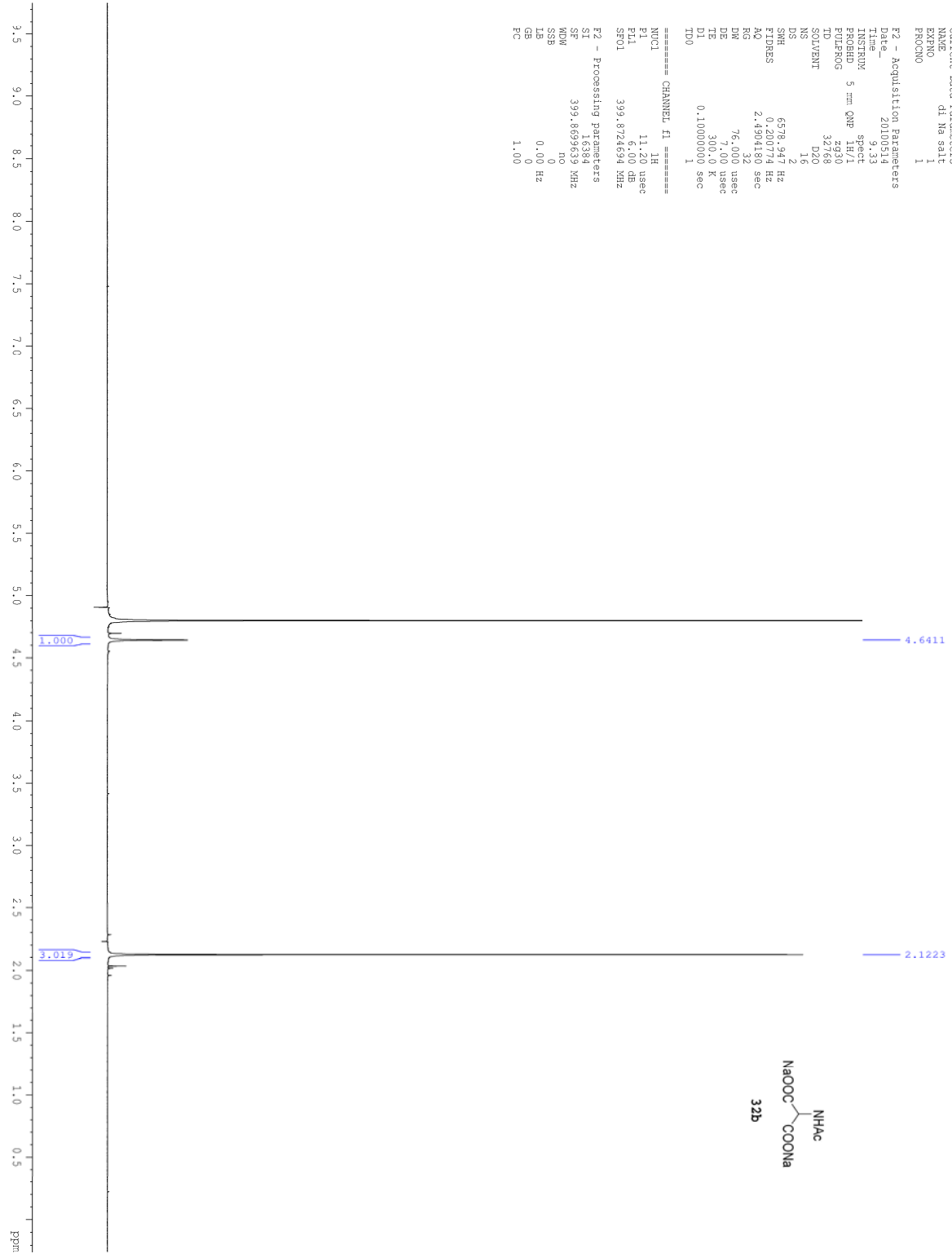
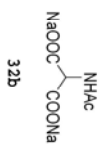
The solution was directly used for the nitromethane addition and could be stored at 5 °C for several months. In addition, the crude reaction mixture without any aqueous workup could also be directly used for the nitromethane addition; however, the chemical yield could drop by 2-3%.

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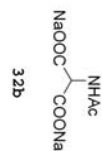
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6. ¹H and ¹³C NMR spectra

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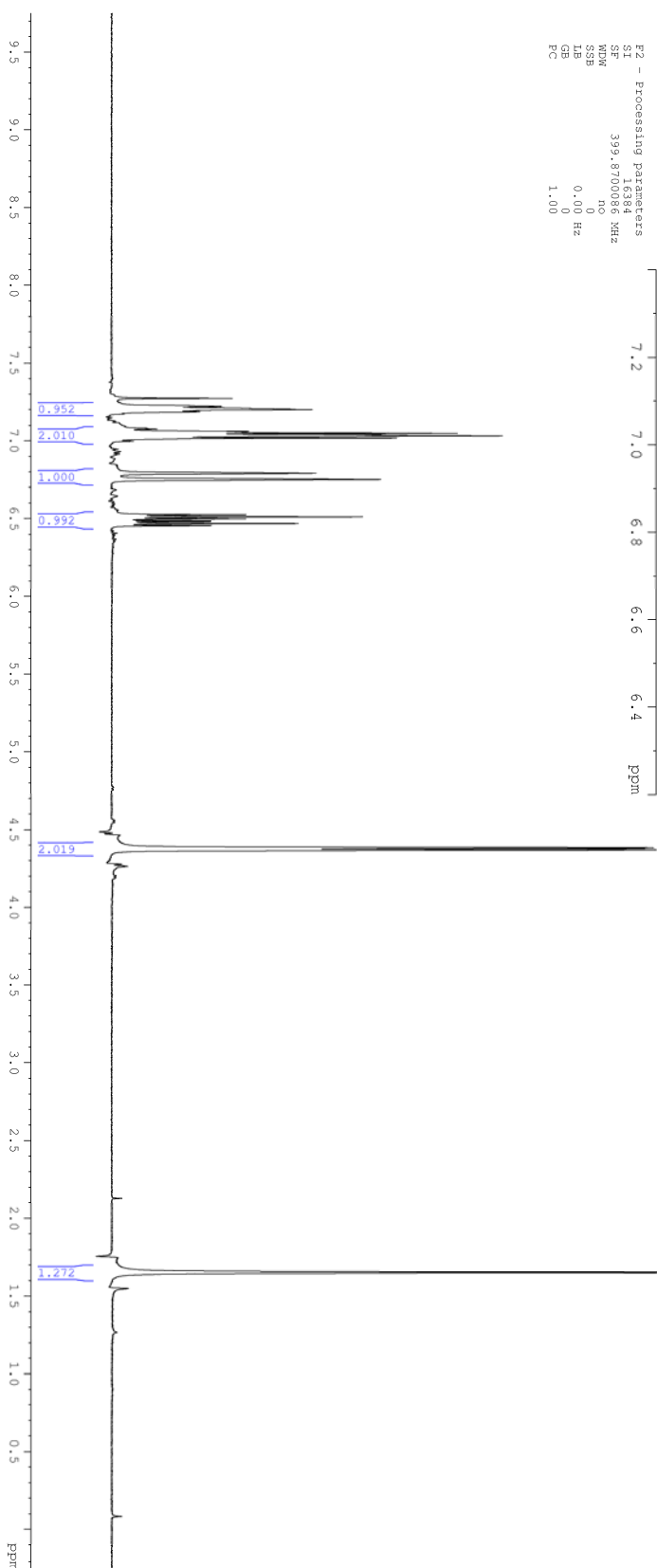
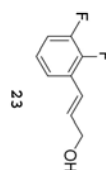
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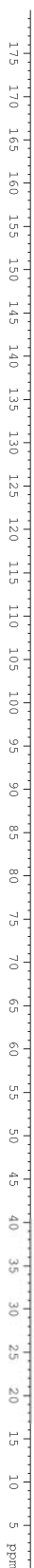
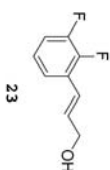
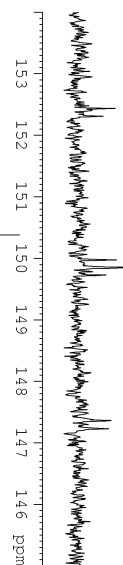
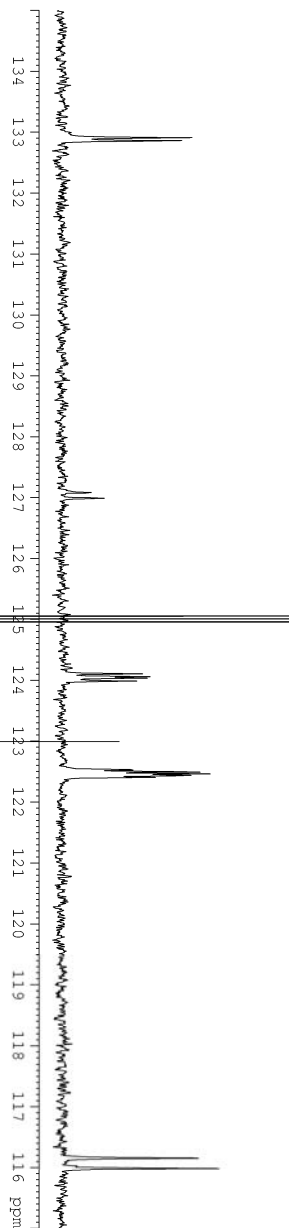
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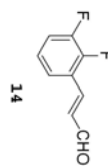
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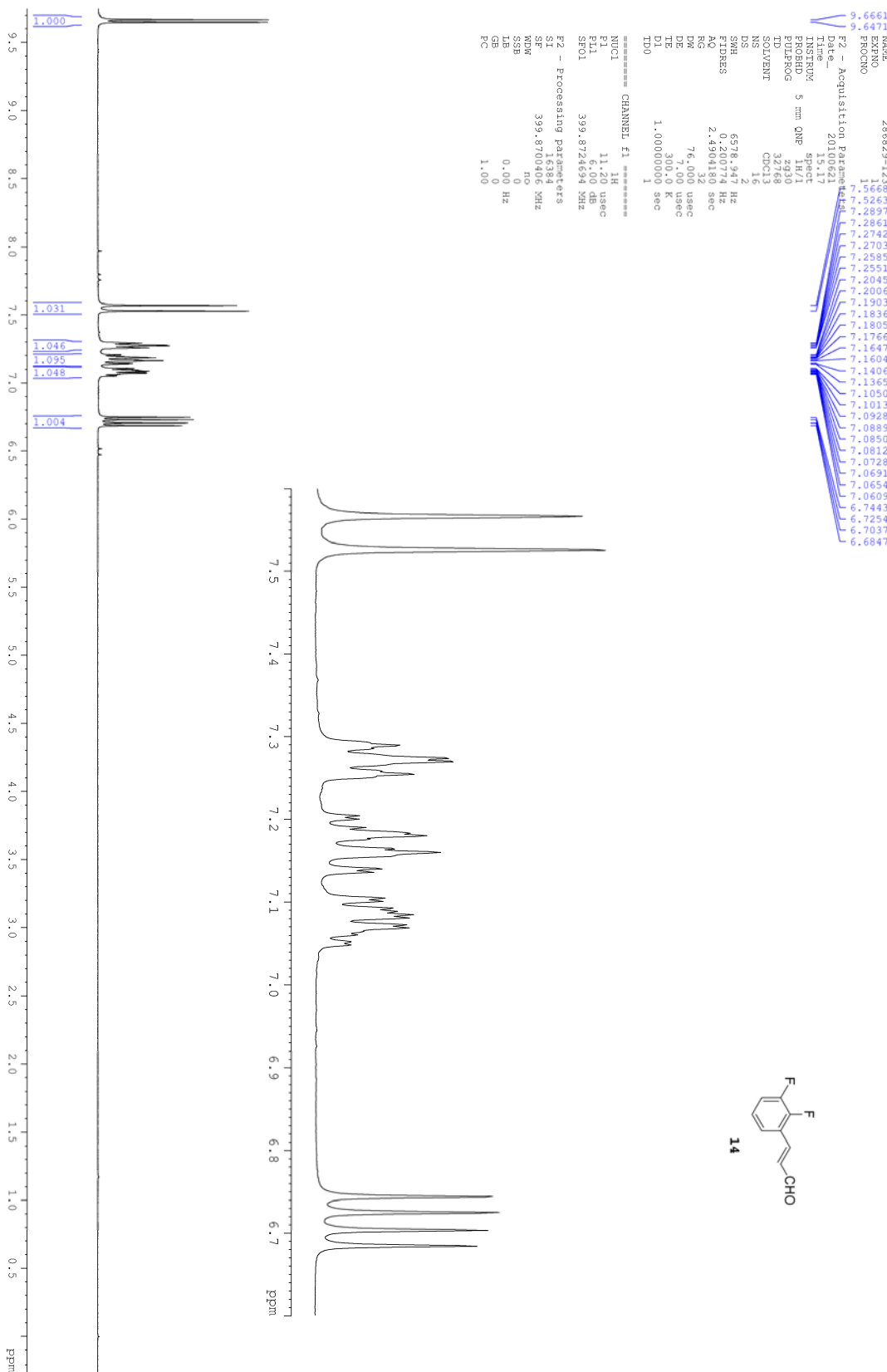
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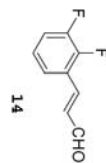
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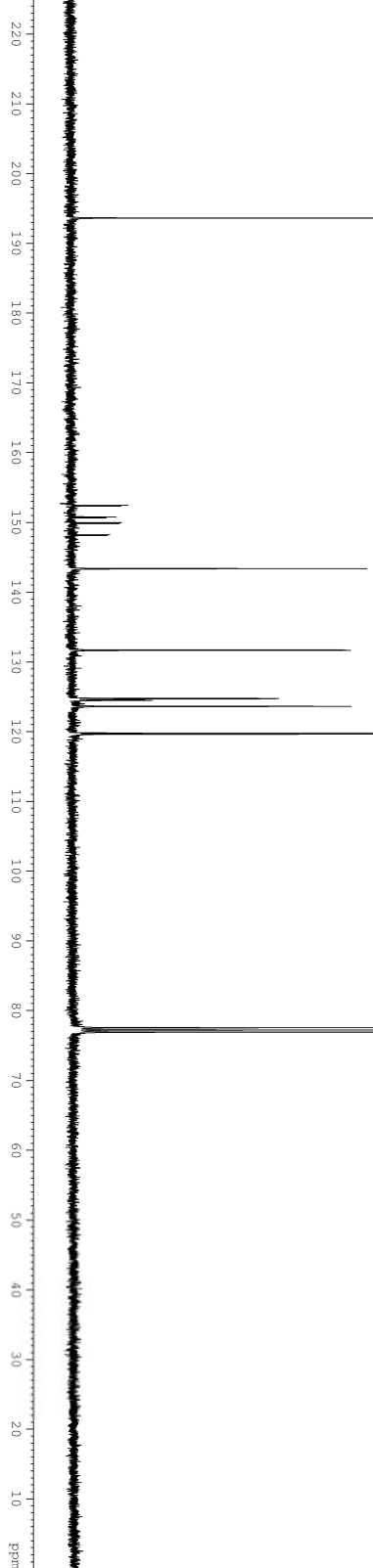
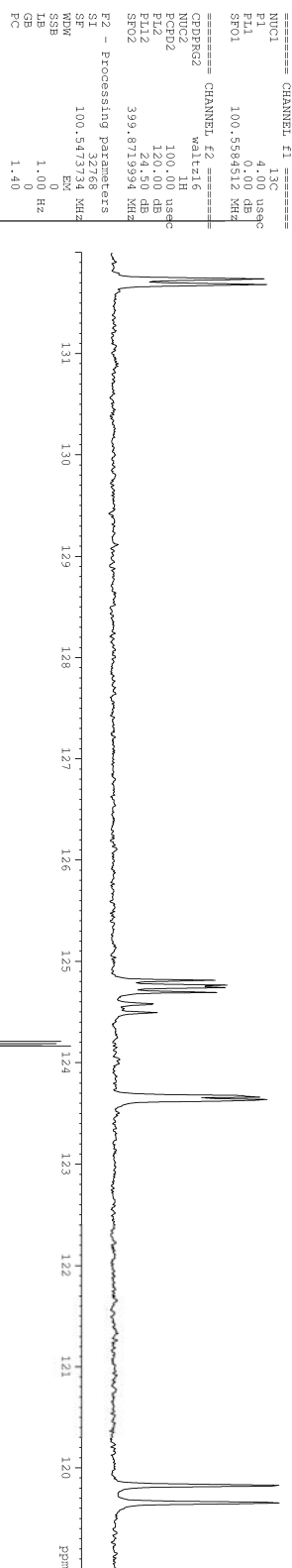
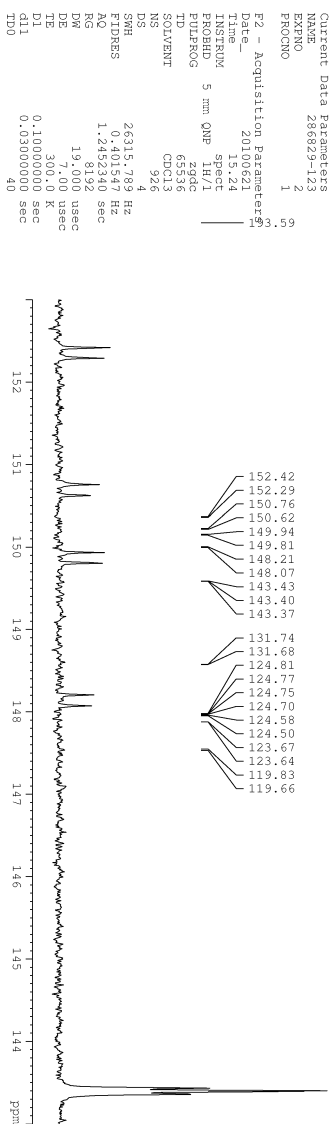
Current Data Parameters
 NAME 28682-133
 EXNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20100621
 Time 15.17
 INSTRUM spect
 PROBD 5 mm QNP 1H/1
 NUC1 13C
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6578.944 Hz
 FIDRES 0.220774 Hz
 AQ 2.4904180 sec
 RG 32
 DW 76.000 usec
 DE 3.00 usec
 TE 300.2 K
 D1 1.00000000 sec
 TDO 1
 ===== CHANNEL f1 =====
 NUC1 13C
 P1 11.20 usec
 PL1 6.00 dB
 SF01 399.8724694 MHz
 F2 - Processing parameters
 SI 32768
 SF 399.8700406 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

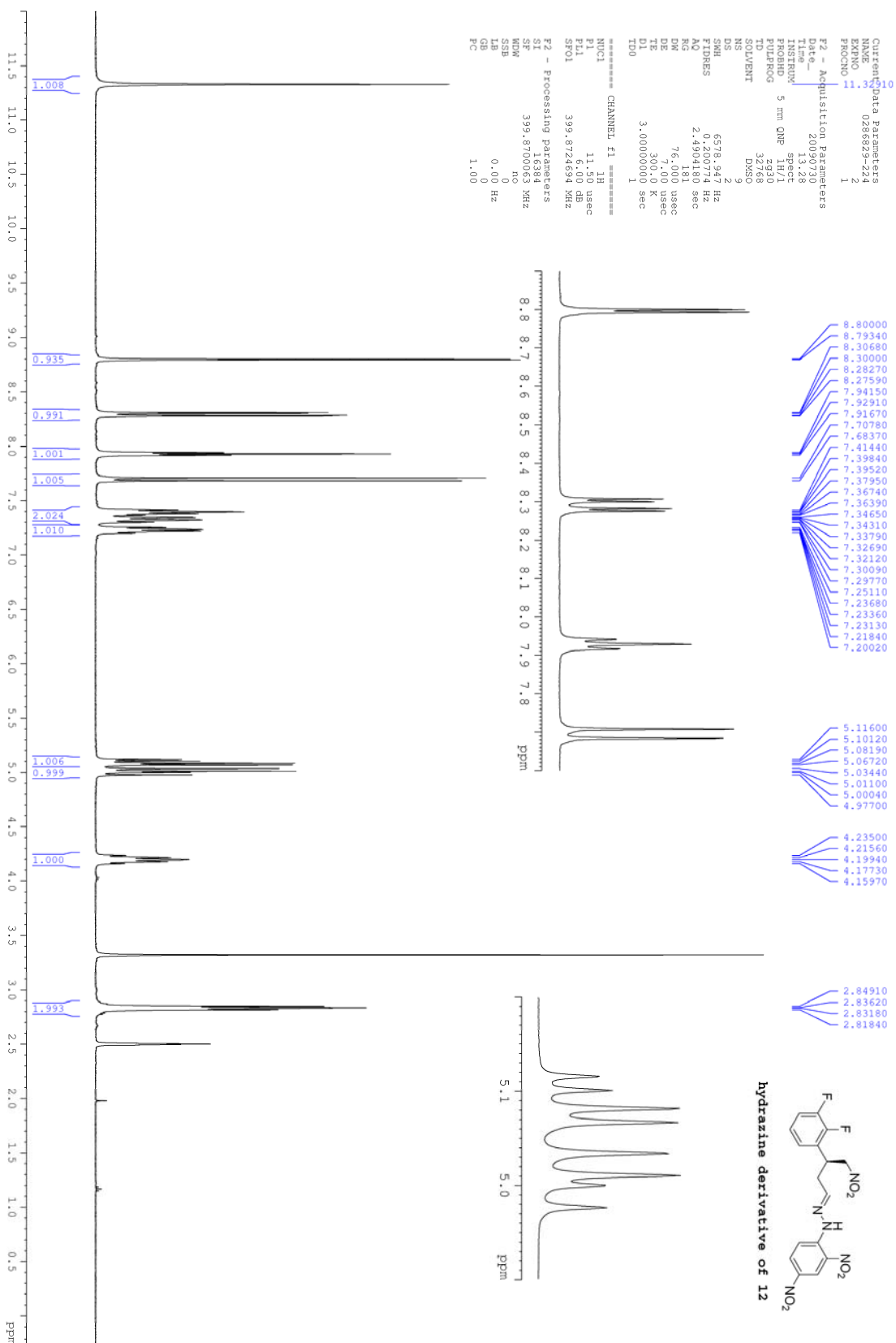


nmr400b c-13



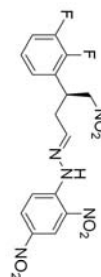
152.42
152.29
150.76
150.62
149.94
149.81
148.21
148.07
143.43
143.40
143.37
131.74
131.68
124.81
124.77
124.75
124.74
124.58
124.50
123.67
123.64
119.83
119.66
119.65





Hydrazone of nitroaldehyde

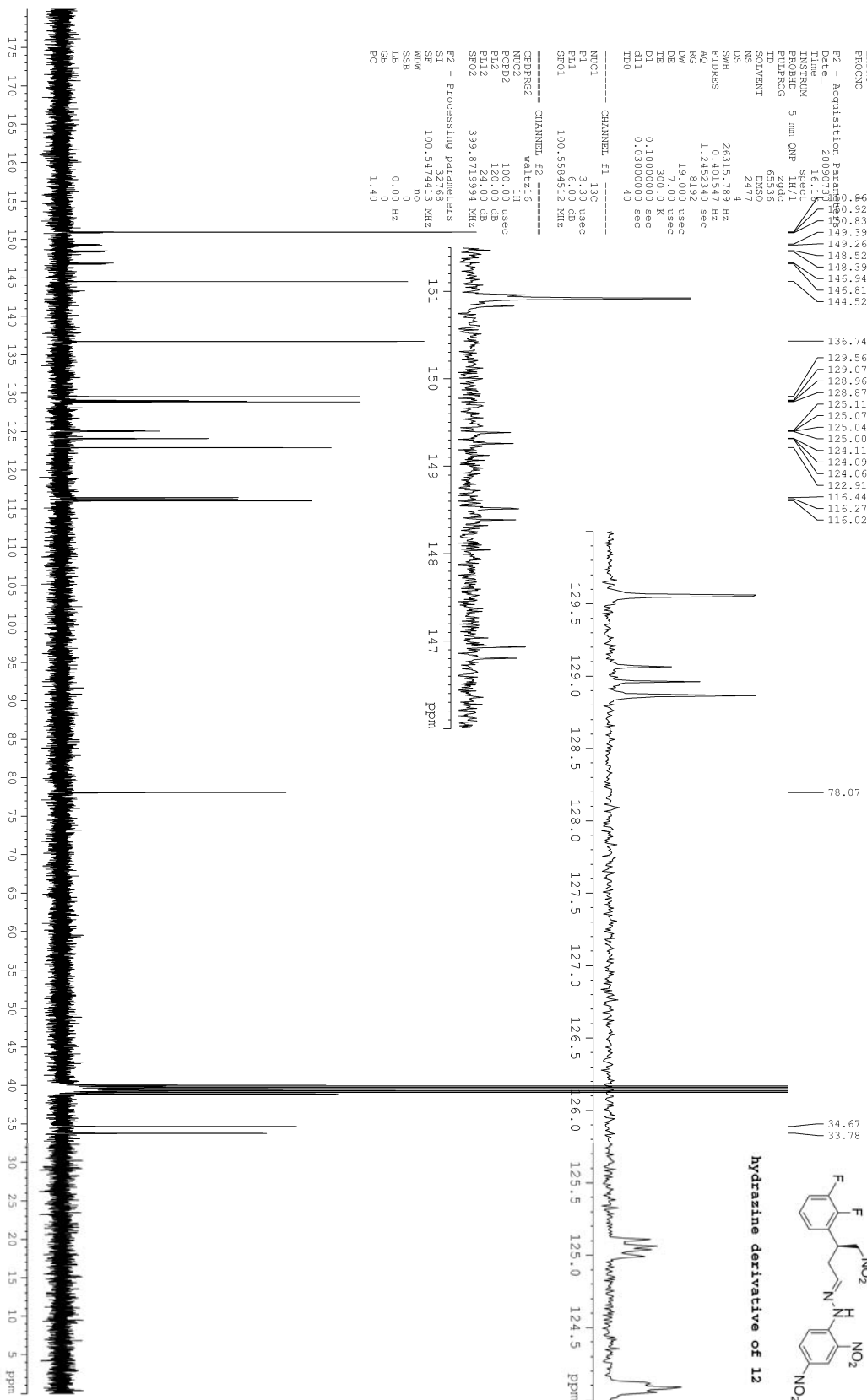
34.67
33.78



hydrazone derivative of 12

Current Data Parameters
NAME 0286829-224
EXPNO 3
PROCNO 2
F2 - Acquisition Parameters
Date_ 20090730
Time 16.18
INSTRUM spect
PROBHD 5 mm QNP
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2477
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.000 usec
DE 7.000 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
TD0 40

===== CHANNEL f1 =====
NUC1 13C
P1 3.30 usec
PL1 6.00 dB
SFO1 100.5584512 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 12.00 dB
PL12 24.00 dB
SFO2 399.8719994 MHz
===== Processing parameters =====
SI 32768
WDW 100.544481 MHz
SSB 0
LB 0.00 Hz
GB 0
FC 1.40



nmr400b f-19

Current Data Parameters
NAME 0286829-224
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters

Date_ 20090730

Time 17.16

INSTRUM spect

PULPROG zgpg30

TD 131072

SOLVENT CDCl3

NS 101

DS 4

SWH 11294.352 Hz

FIDRES 0.862078 Hz

AQ 0.5800436 sec

RG 2048

DM 4.465 usec

DE 1700 usec

TE 300.0 K

D1 1.00000000 sec

d11 0.03000000 sec

d12 0.00002000 sec

ID0 1

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

NUC1 19F

P1 3.00 usec

PL1 0.00 dB

SFO1 376.1372640 MHz

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

CEPPRG2 waltz16

NUC2 1H

P2 100.00 usec

PL2 6.00 dB

SFO2 399.8719990 MHz

F2 - Processing parameters

SI 32768

SF 376.2537220 MHz

WDW no

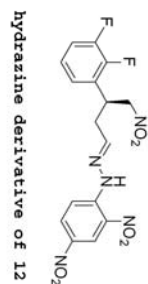
SSB 0

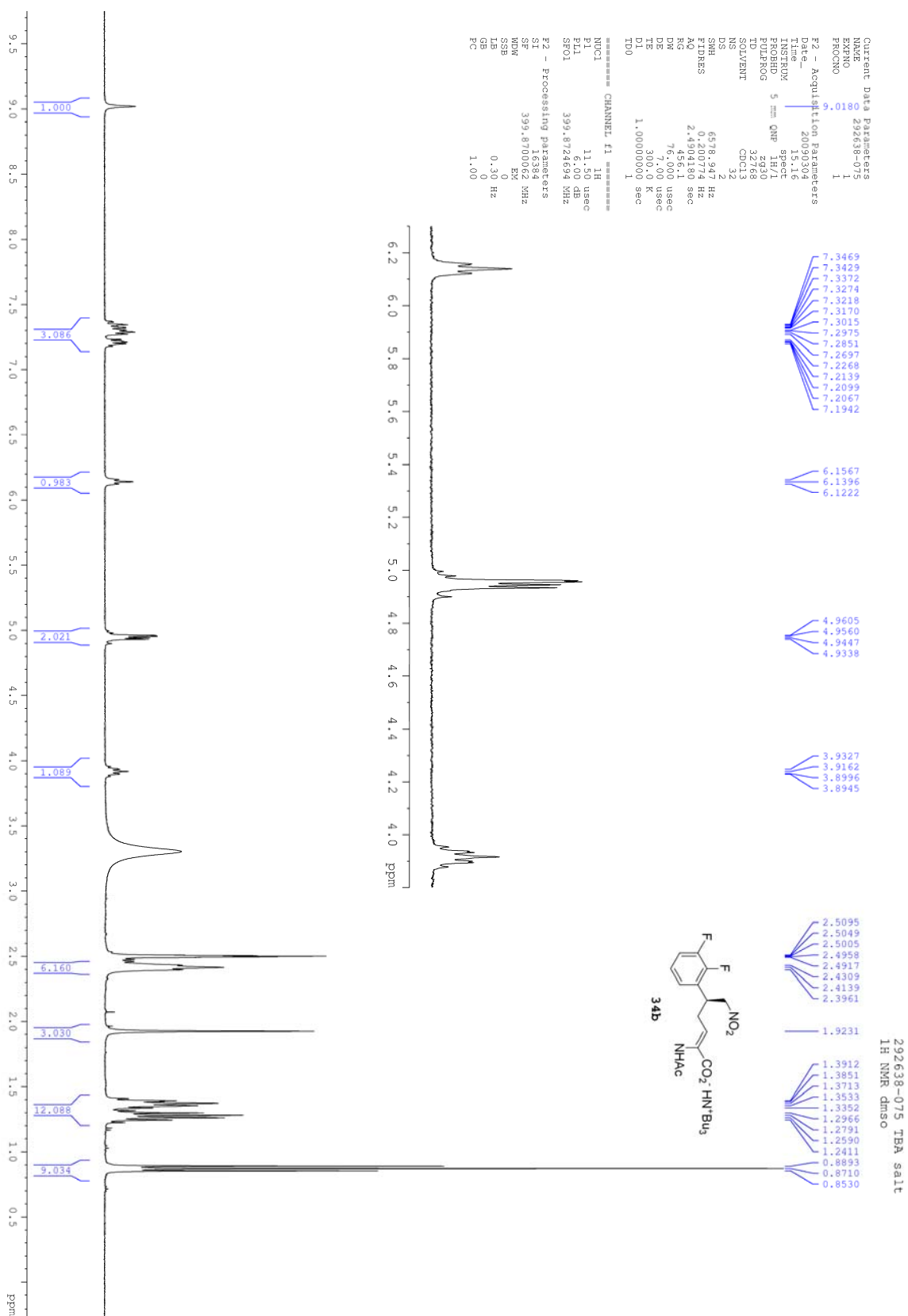
LB 0.00 Hz

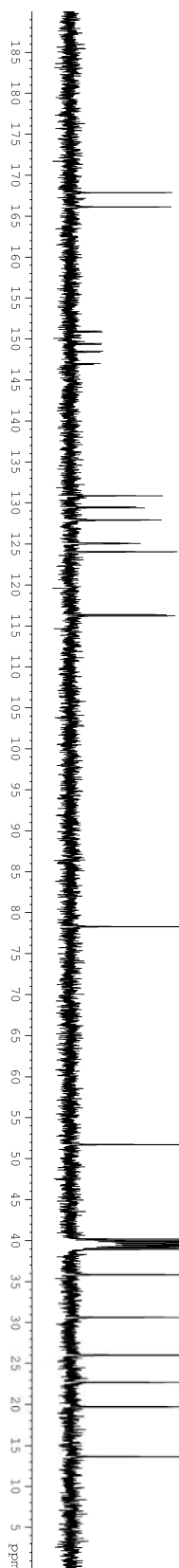
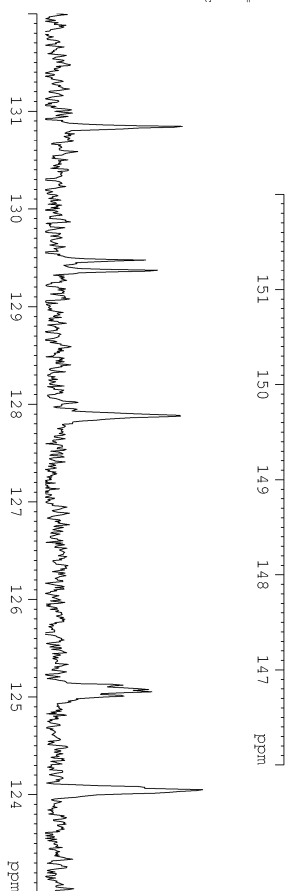
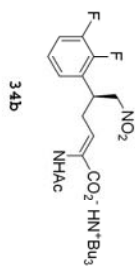
GB 0

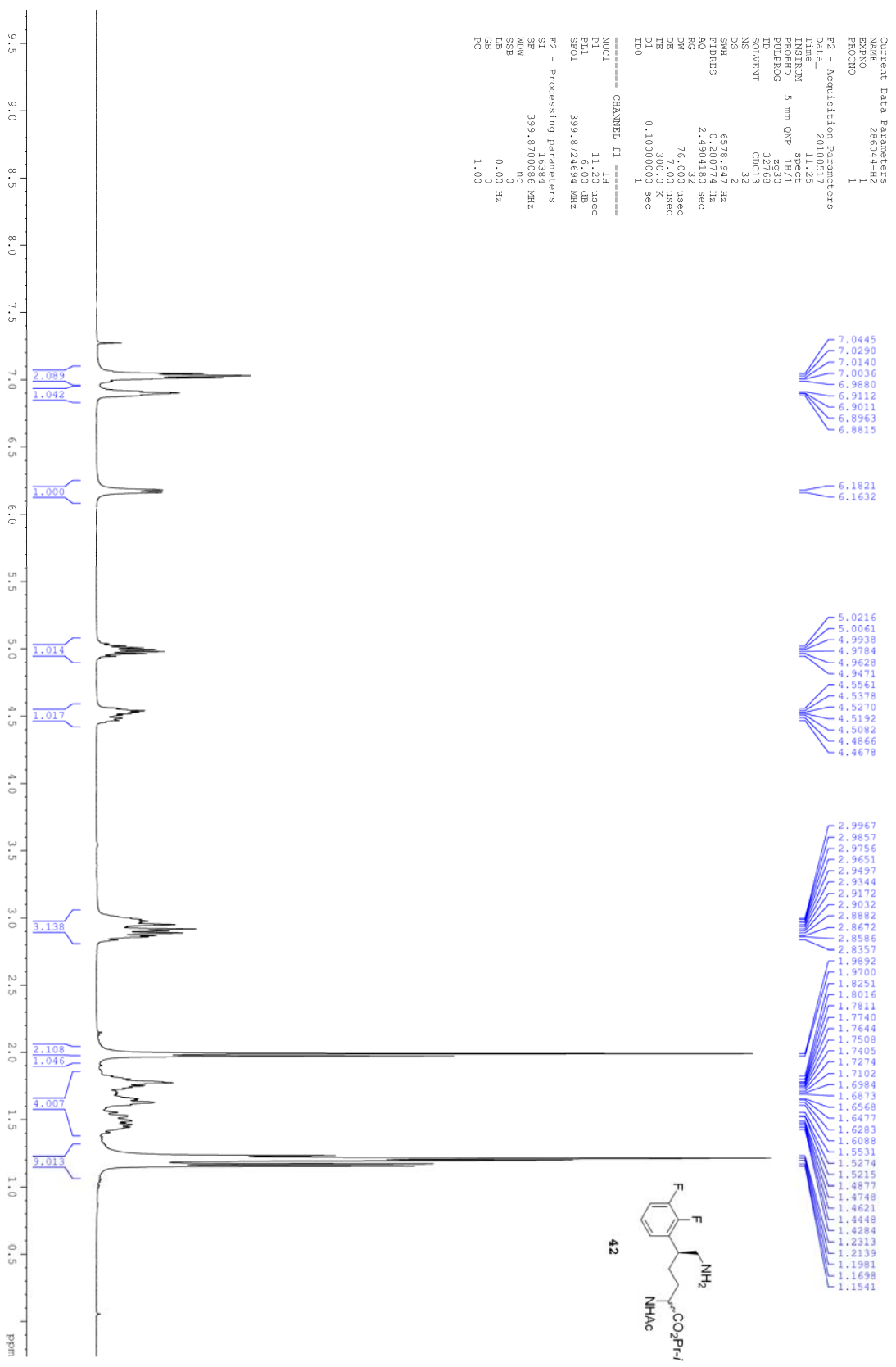
PC 1.00

138.57
138.63
143.22
143.28









nmr400b f-19

Current Data Parameters
NAME 286044-H2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100517

Time 11.31

INSTRUM 5 mm QNP spect

PULPROG zgpg30m

TD 131072

SOLVENT CDCl3

NS 199

DS 4

SWH 11294.352 Hz

FIDRES 0.862078 Hz

AQ 0.5800436 sec

RG 4096

DM 4.425 usec

DE 1700.0 usec

TE 300.0 K

D1 1.00000000 sec

d11 0.03000000 sec

d12 0.00002000 sec

TD 1

NUC1 CHANNEL f1

P1 197

P2 3.00 usec

PL1 6.00 dB

PL2 376.1372440 MHz

NUC2 CHANNEL f2

waitz16

CPDPRG2

NUC2 1H

PL1 100.00 usec

PL2 6.00 dB

PL12 24.00 dB

SFO2 399.8719990 MHz

F2 - Processing parameters

SF 376.2537220 MHz

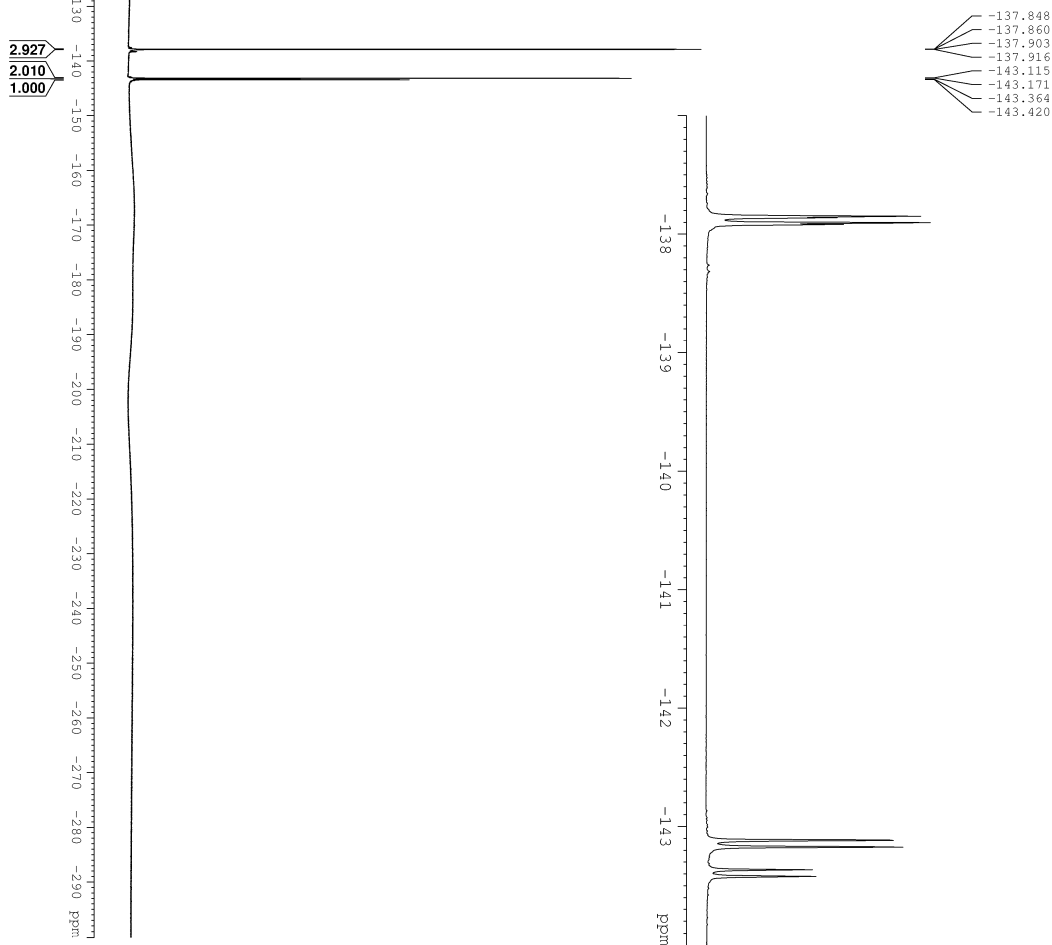
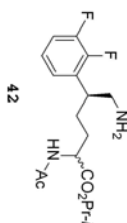
WDW EX

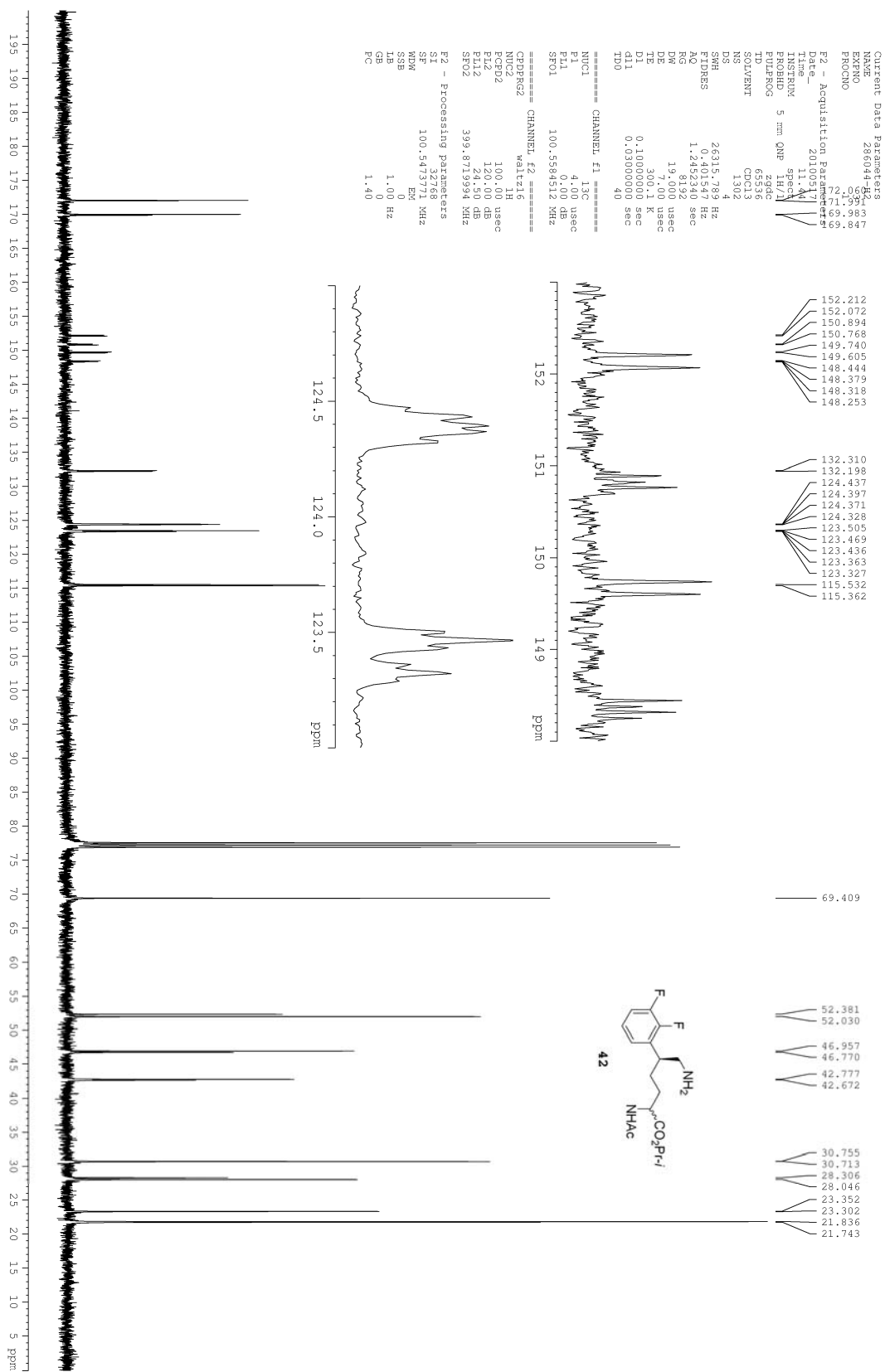
SSB 0

LB 0.20 Hz

GB 0

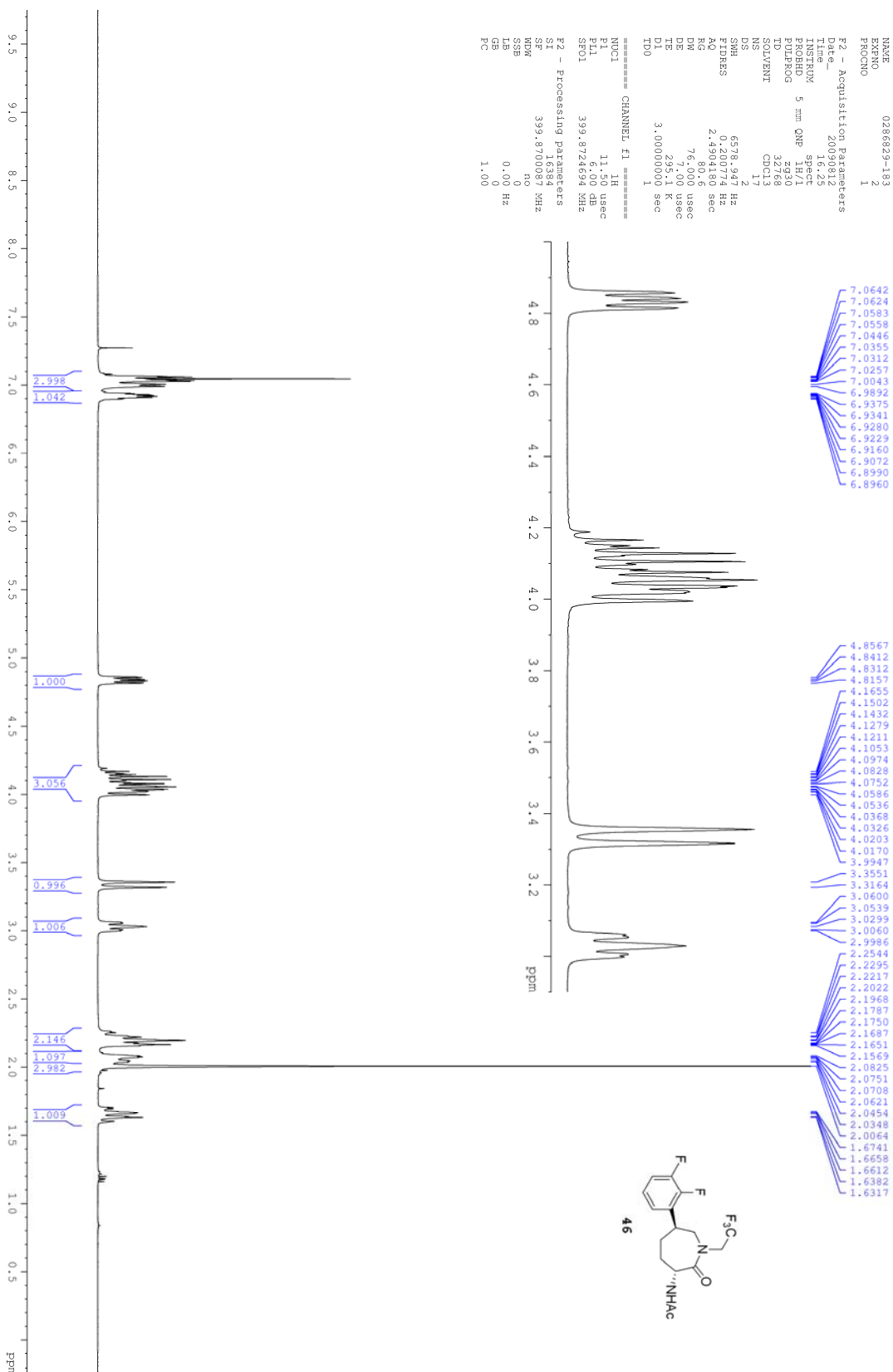
PC 1.00



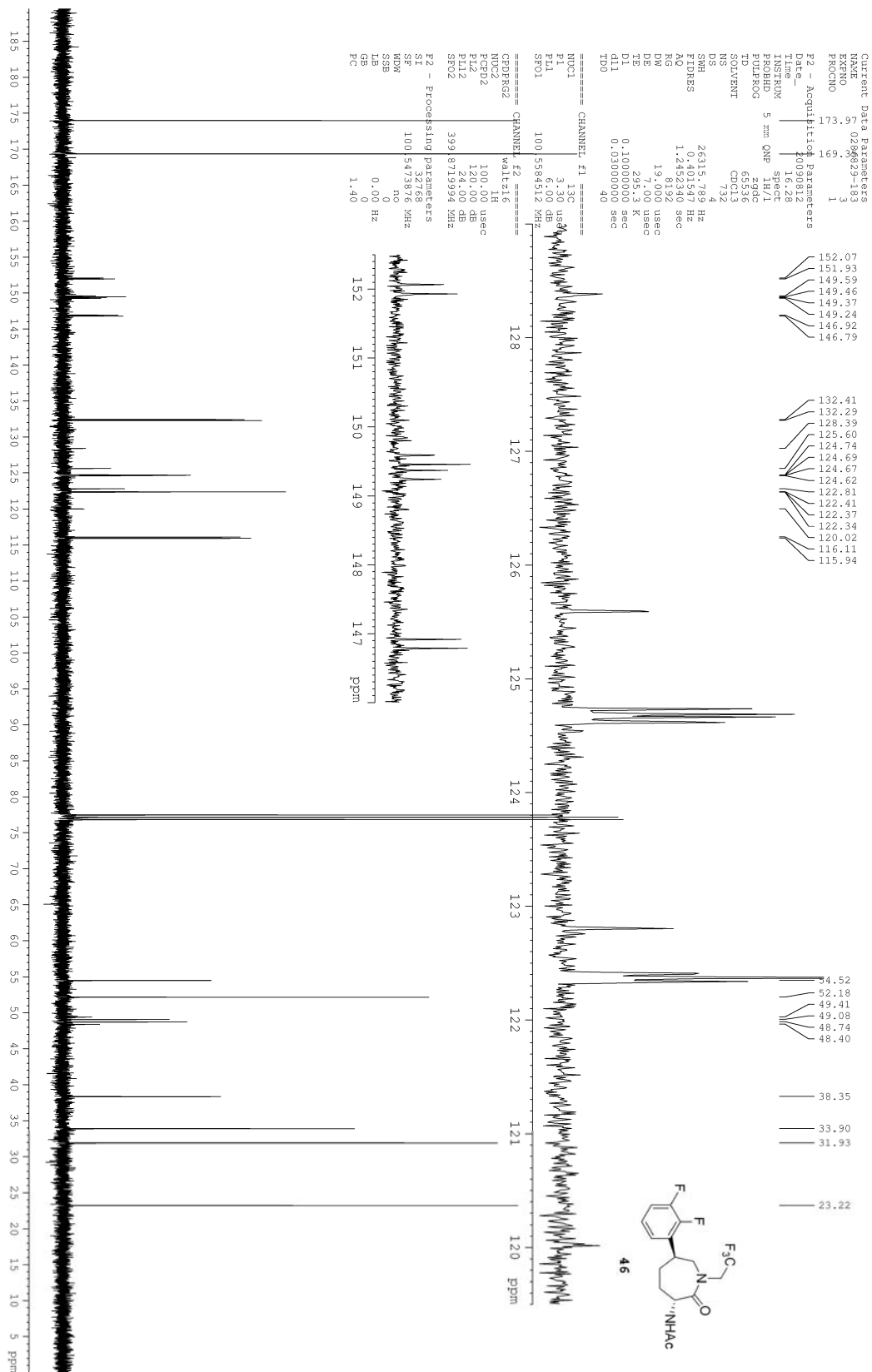


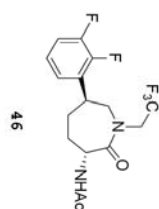
Caprolactam acetamide

Current Data Parameters
 NAME 028682-133
 EXPNO 2
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20090812
 Time 16:25
 INSTRUM spect
 PROBHD 5 mm QNP
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 17
 DS 2
 SWH 6578.944 Hz
 FIDRES 0.220774 Hz
 AQ 2.4904180 sec
 RG 80.6
 DW 76.000 usec
 DE 23.00 usec
 DI 3.00000000 sec
 TDO 1
 ===== CHANNEL f1 =====
 NUC1 1
 P1 11.50 usec
 PL1 6.00 dB
 SFO1 399.8724694 MHz
 F2 - Processing parameters
 SF 399.8700087 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

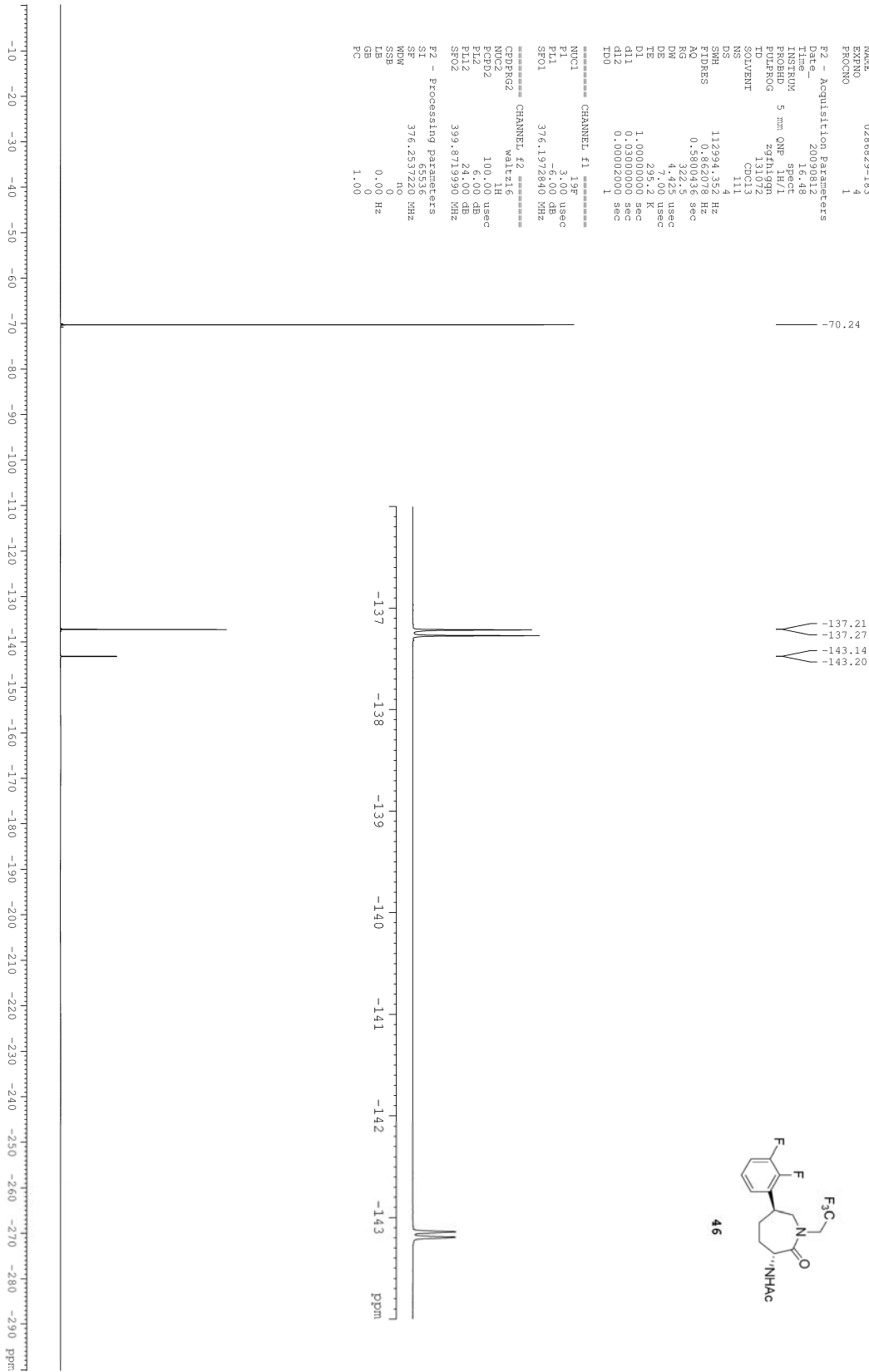


caprolactam acetamide

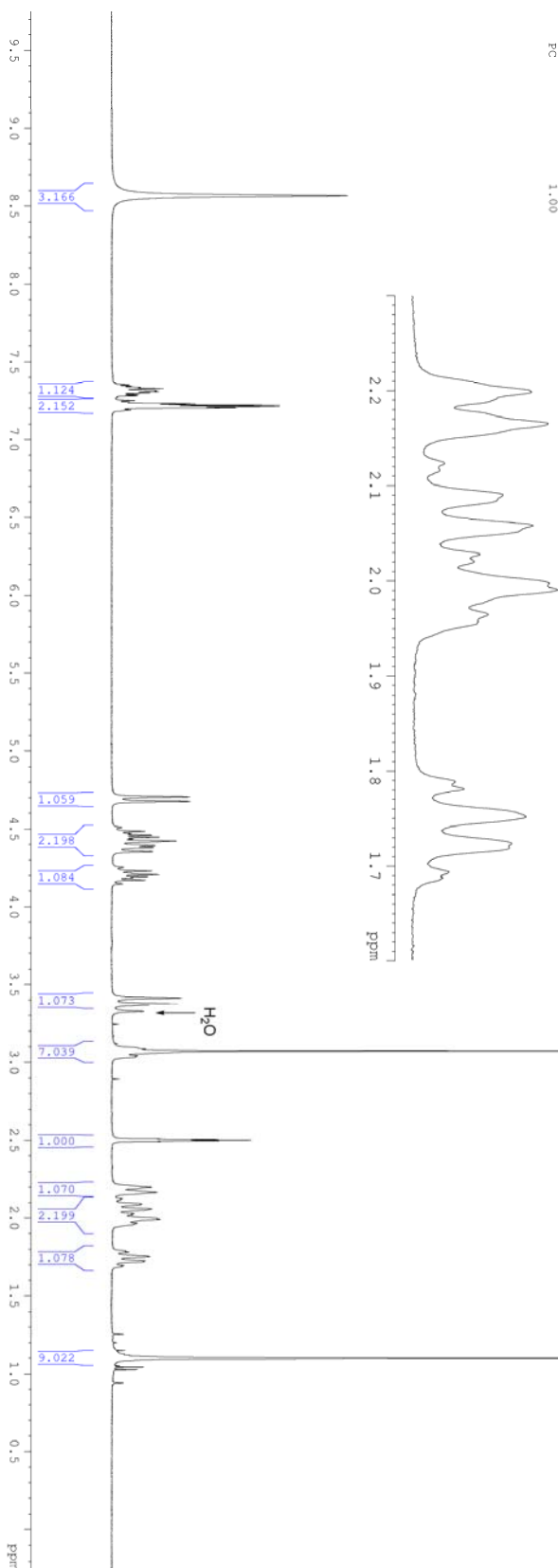
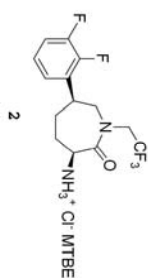
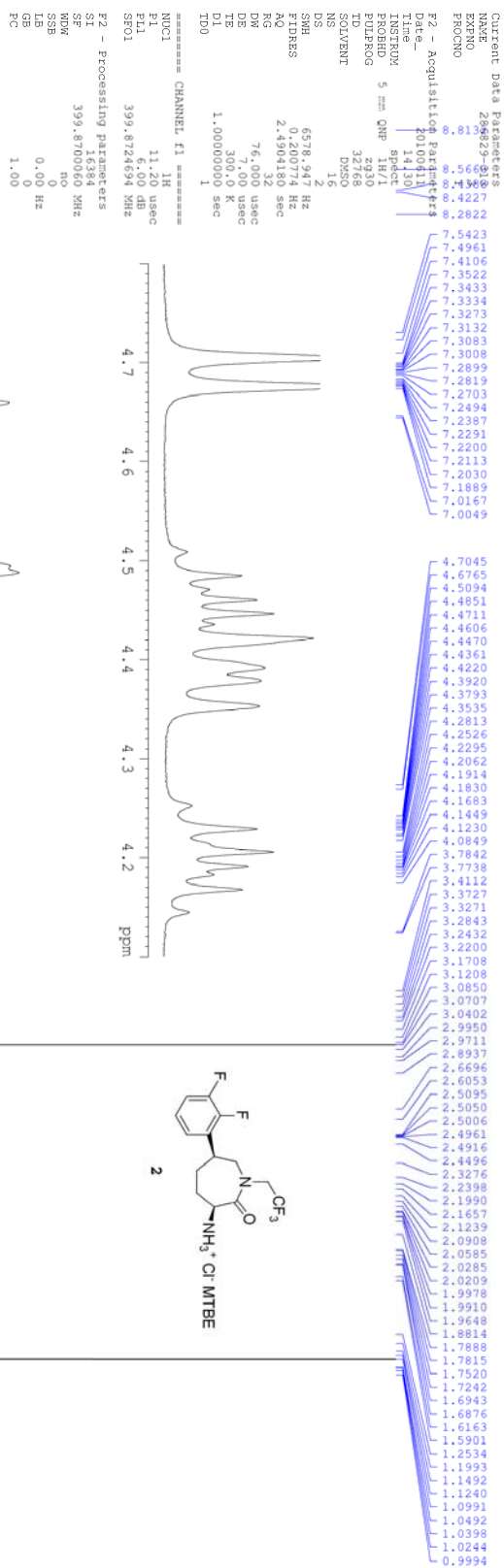




Current Data Parameters
NAME 028623-183
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090812
Time 16:48
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 111
DS 4
SWH 112394.352 Hz
FIDRES 0.862078 Hz
AQ 0.5800436 sec
RG 322.5
DM 4.425 usec
DE 1700.000 usec
TE 295.2 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 19F
P1 3.00 usec
PL1 6.00 dB
SFO1 376.137240 MHz
===== CHANNEL f2 =====
CEPPRG2 waltz16
NUC2 1H
P2 10.00 usec
PL2 6.00 dB
PL12 24.00 dB
SFO2 399.8719990 MHz
F2 - Processing parameters
SI 32768
SF 376.2537220 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



nmr400b h-1



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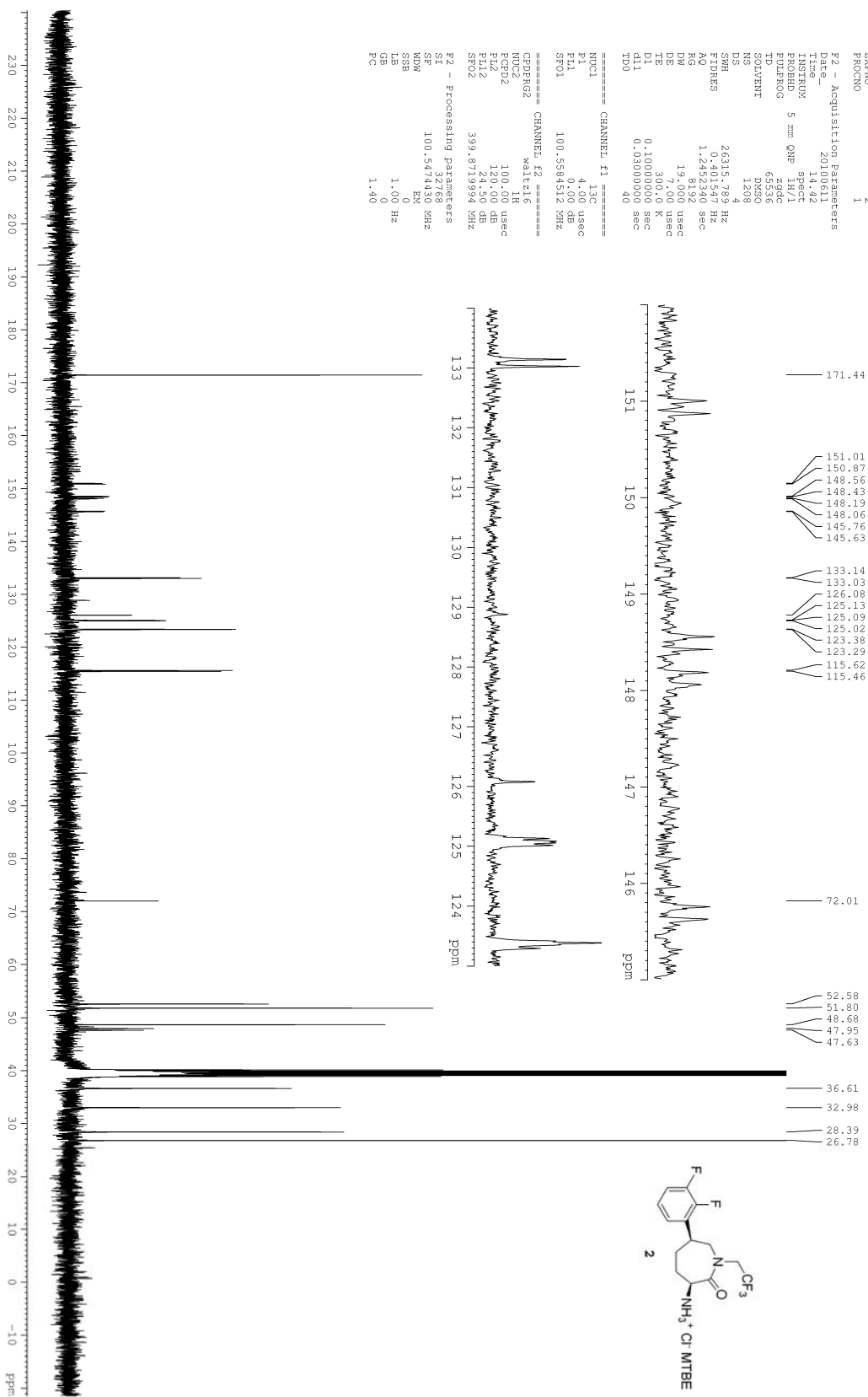
Current Data Parameters
NAME      286823-313
EXPNO     1
PROCNO    1
F2 - Acquisition Parameters
Date_     20100611
Time      14.42
INSTRUM   spect
PROBHD    5 mm QNP
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         1208
DS         4
SFO1       26315.789 Hz
FIDRES    0.401547 Hz
AQ         1.2452340 sec
RG         8192
DM         19.000 usec
DE         700.0 usec
TE         300.0 K
D1         0.10000000 sec
d11        0.03000000 sec
TD0        40

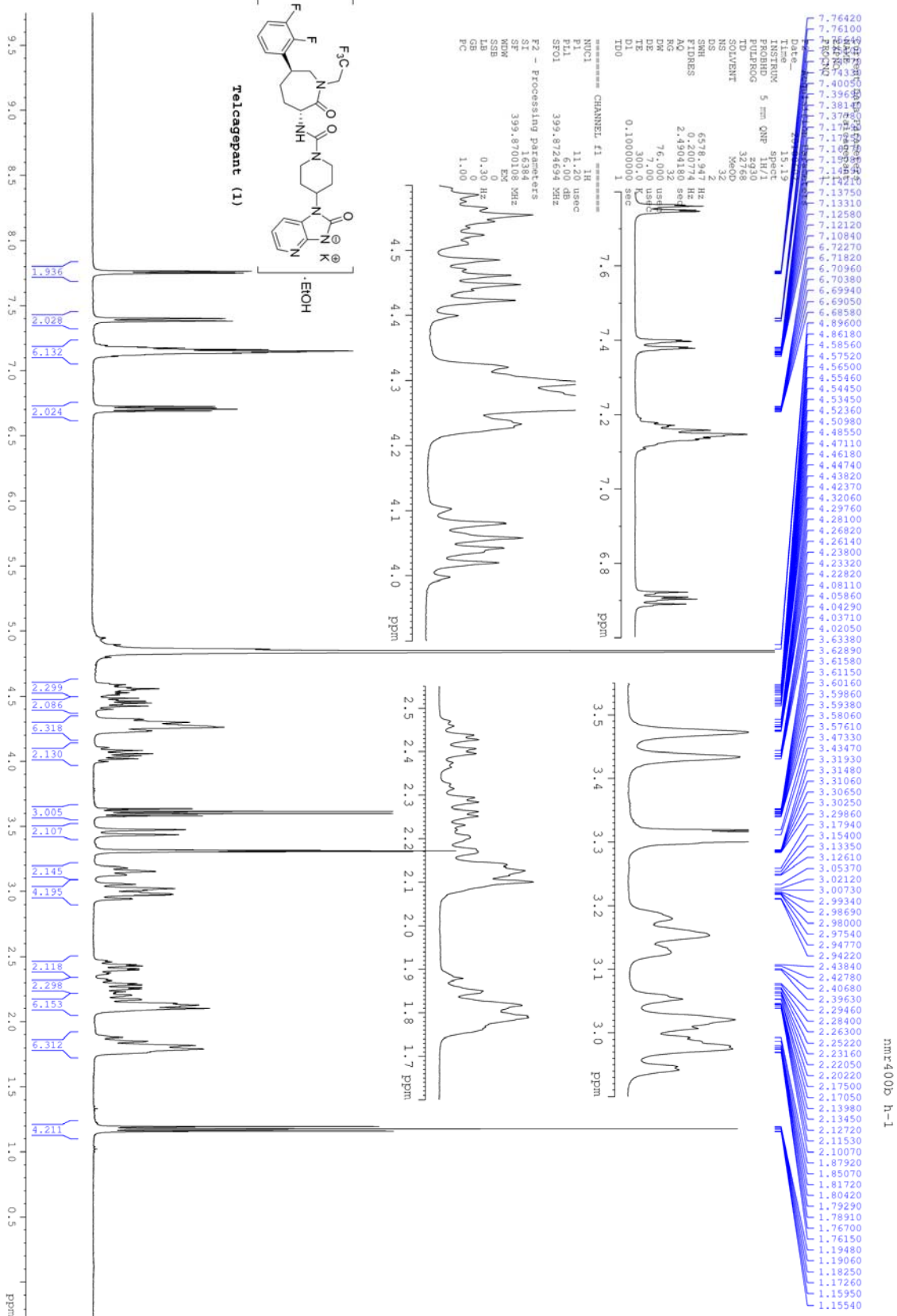
===== CHANNEL f1 =====
NUC1       13C
P1         4.00 usec
PL1        0.00 dB
SFO1       100.5584512 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      100.00 usec
PL2        120.00 dB
PL12       24.50 dB
SFO2       399.8719994 MHz

F2 - Processing parameters
SI         32768
WDW         100.544433 MHz
SSB         0
LB          1.00 Hz
GB          0
PC          1.40

```





nmr400b c-13

Current Data Parameters
NAME Telcagepant
EXPNO 2
PROCNO 1
Date_ 20100602
Time 15.56
F2 - Acquisition Parameters
Date_ 20100602
Time 15.56
F2INSTRUM spect
F2PROB 5 mm QNP 129dc
F2PULPROG zgpg30
F2TD 65536
F2SOLVENT CDCl3
F2NS 1133
F2DS 1024
F2SWH 26315.789 Hz
F2FIDRES 0.401547 Hz
F2AQ 1.2452340 sec
F2RG 8192
F2DM 19.000 usec
F2DE 7.000 usec
F2TE 300.0 K
F2D1 0.10000000 sec
F2d11 0.03000000 sec
F2TD0 40

===== CHANNEL f1 =====
NUC1 13C
P1 4.00 usec
PL1 0.00 dB
SFO1 100.5584512 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PCPD2 120.00 dB
PL12 24.50 dB
SFO2 399.8719994 MHz

F2 - Processing parameters
SI 32768
SF 100.54289 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
FC 1.40

