

# Total Synthesis of the Antifungal Depsipeptide, Petriellin A

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## Supporting Information

### Contents

General Experimental S2

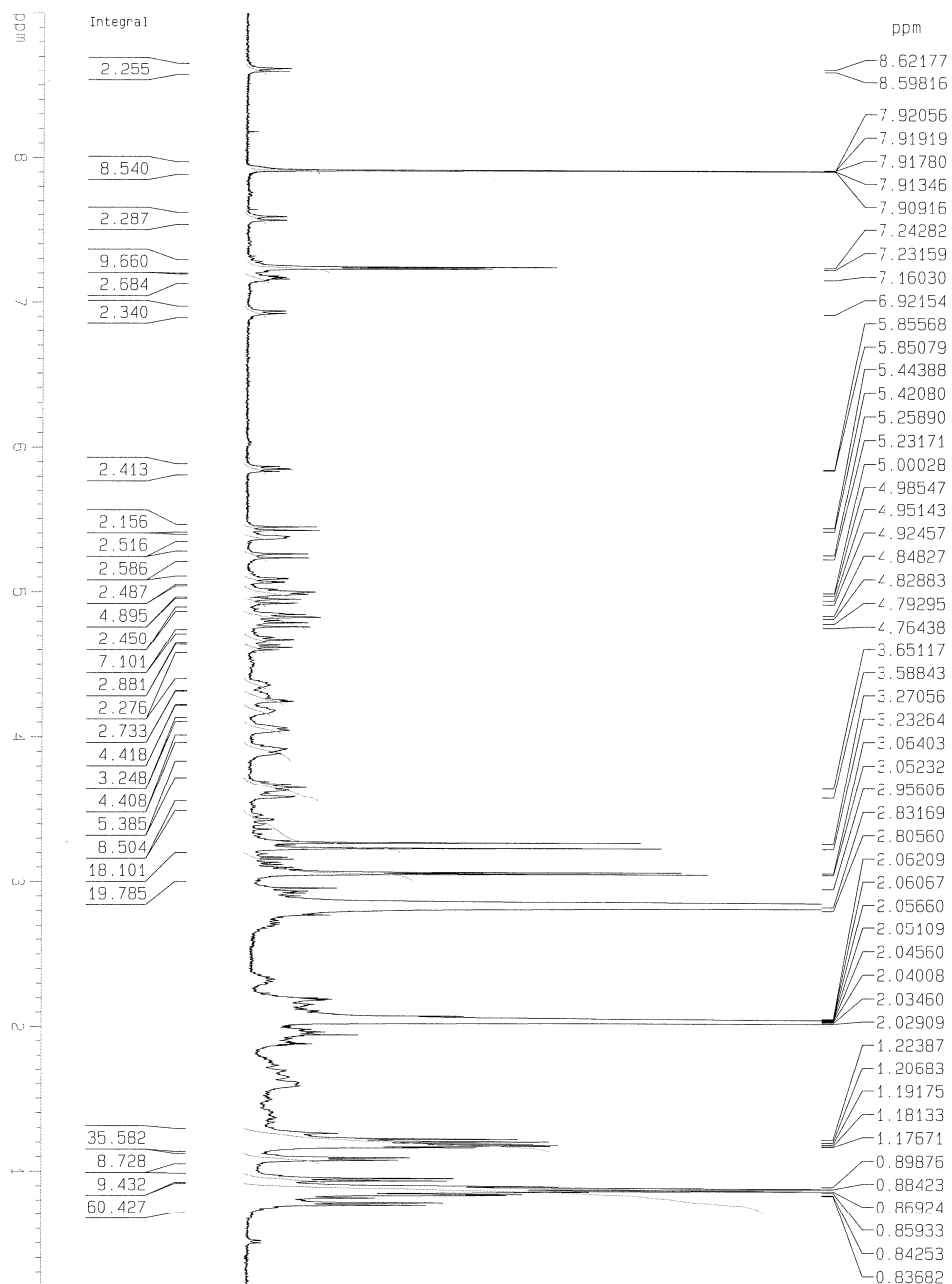
Compound Number	Page	<sup>1</sup> H NMR Spectrum	<sup>13</sup> C NMR Spectrum	ESMS	HPLC
<b>1</b>	S3	√			
<b>1</b>	S4	√			
<b>1</b>	S5				√
<b>1</b>	S6				√
<b>1</b>	S7			√	
<b>9</b>	S8	√			
<b>9</b>	S9		√		
<b>9</b>	S10		√		
<b>10</b>	S11	√			
<b>10</b>	S12		√		
<b>10</b>	S13		√		

## General Methods

NMR was performed in (D)-chloroform at 300 K unless otherwise stated on a 300 MHz spectrometer. Low and high resolution mass spectra (LSIMS) were also measured at 70 eV; all spectra were acquired using water/methanol/acetic acid (0:99:1 or 50:50:1) mixtures as the mobile phase. Flash column chromatography was carried out using silica gel 60 particle size 0.040-0.063  $\mu\text{m}$  (230-400 mesh ASTM). HPLC analysis was carried out on an instrument using a photodiode array detector. The HPLC samples were eluted with pH controlled buffers. Buffer A consisted of 0.1% TFA:water (pH 2). Buffer B consisted of 90% acetonitrile:water containing 0.1% TFA (pH 2). Ethyl acetate and hexane used for chromatography were distilled prior to use. TLC was performed on kieselgel 60 F<sub>254</sub> plates and visualized with a UV lamp. For dry solvents, procedures from Perrin and Armarego<sup>\*</sup> were followed. All other reagents and solvents were purified or dried as described by Perrin and Armarego.<sup>\*</sup>

<sup>\*</sup>Perrin, D. D.; Armarego, W. L. F., Purification Of Laboratory Chemicals; 3rd Ed.; Pergamon: Oxford, 1988.

# <sup>1</sup>H NMR of Natural Petriellin A 1



Petriellin: 2mg in 1:4 Chloroform: Acetone 5mL mixture.

Current Data Parameters  
NAME Dec-99  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters

Date\_ 991202  
Time 7.44  
INSTRUM spect  
PROBHD 5 mm Multinu  
PULPROG zg  
TD 16384  
SOLVENT Acetone  
NS 64  
DS 4  
SWH 4401.409 Hz  
FIDRES 0.26641 Hz  
AQ 1.8612725 sec  
RG 50  
DM 113.600 usec  
DE 4.50 usec  
TE 273.0 K  
D1 2.0000000 sec

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

NUC1 1H  
P1 6.00 usec  
PL1 1.00 dB  
SFO1 400.1320200 MHz

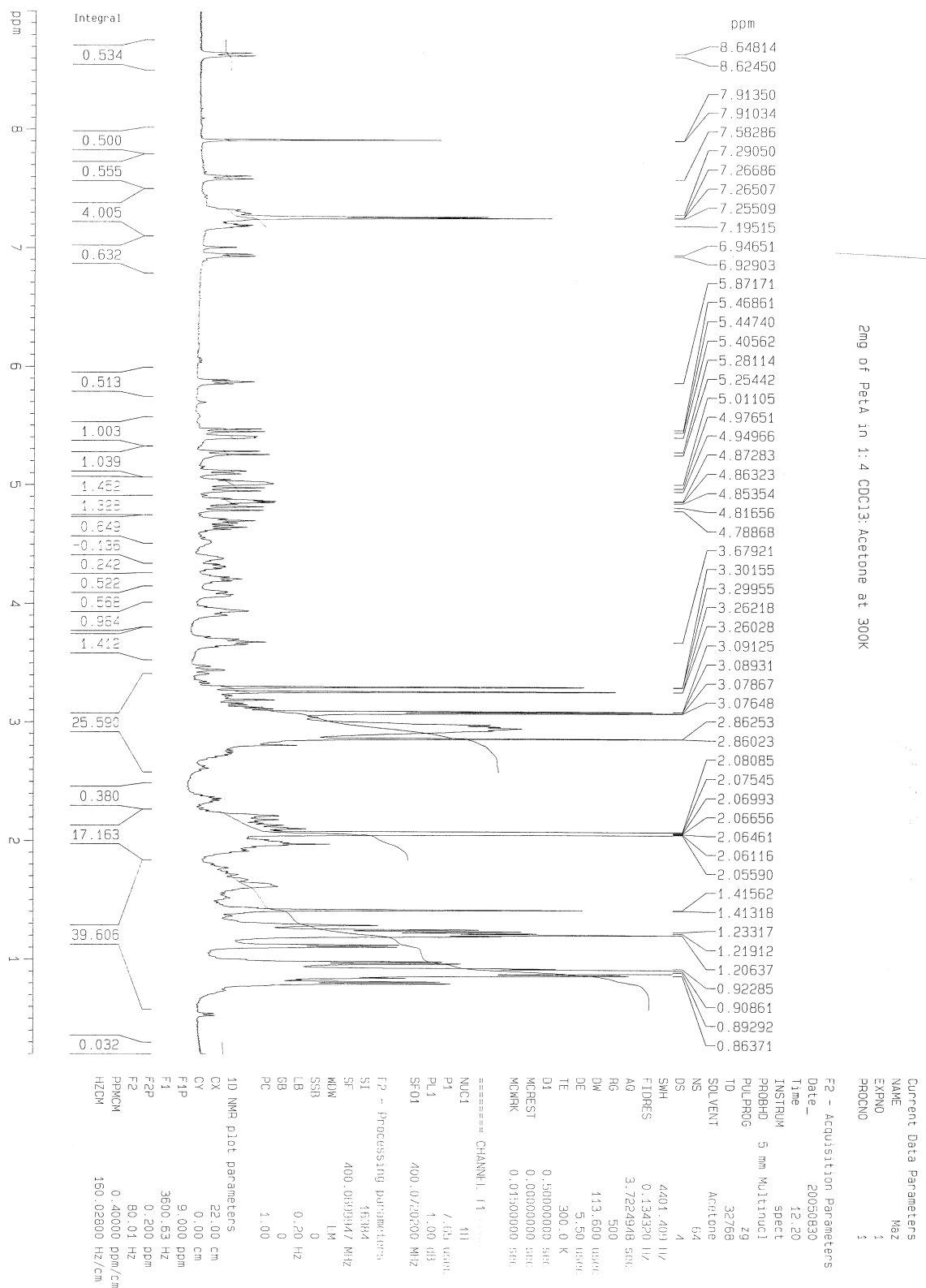
F2 - Processing parameters

SF 32768  
WDW EM  
SSB 0  
LB 0.25 Hz  
GB 0  
PC 1.00

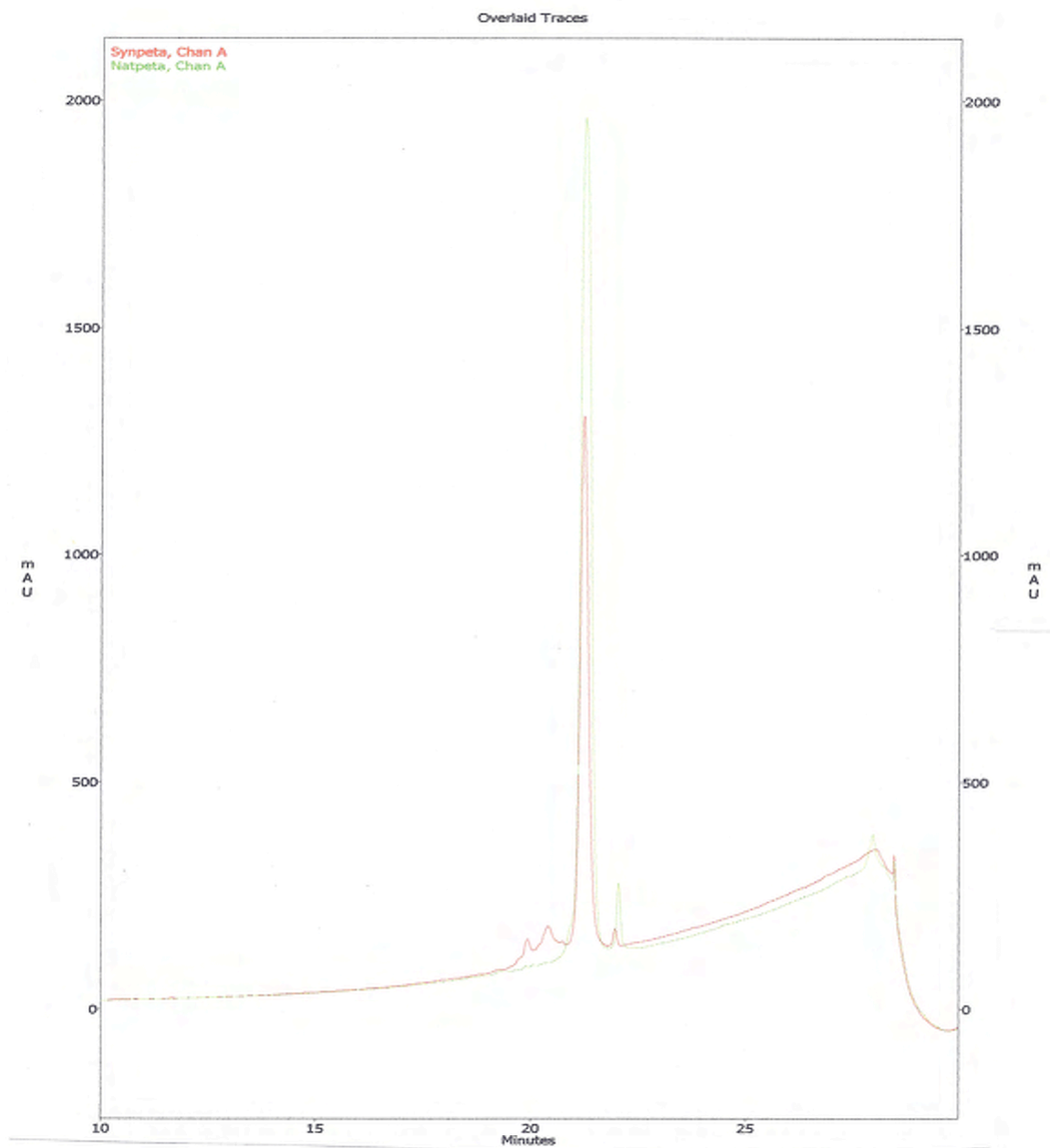
1D NMR plot parameters

CX 22.00 cm  
CY 0.00 cm  
F1P 9.000 ppm  
F1 3601.17 Hz  
F2P 0.200 ppm  
F2 80.03 Hz  
PPMCM 0.40000 ppm/cm  
HZCM 160.05202 Hz/cm

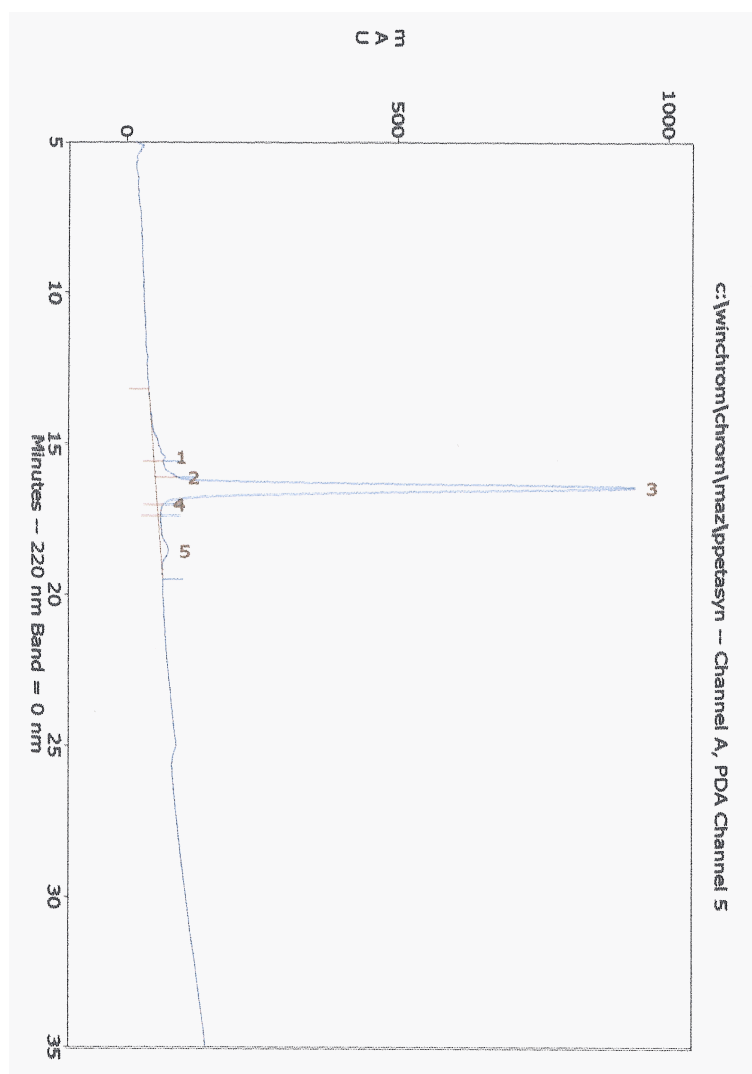
# <sup>1</sup>H NMR of Synthetic Petriellin A 1



## Overlaid HPLC Trace of Natural and Synthetic Petriellin A 1

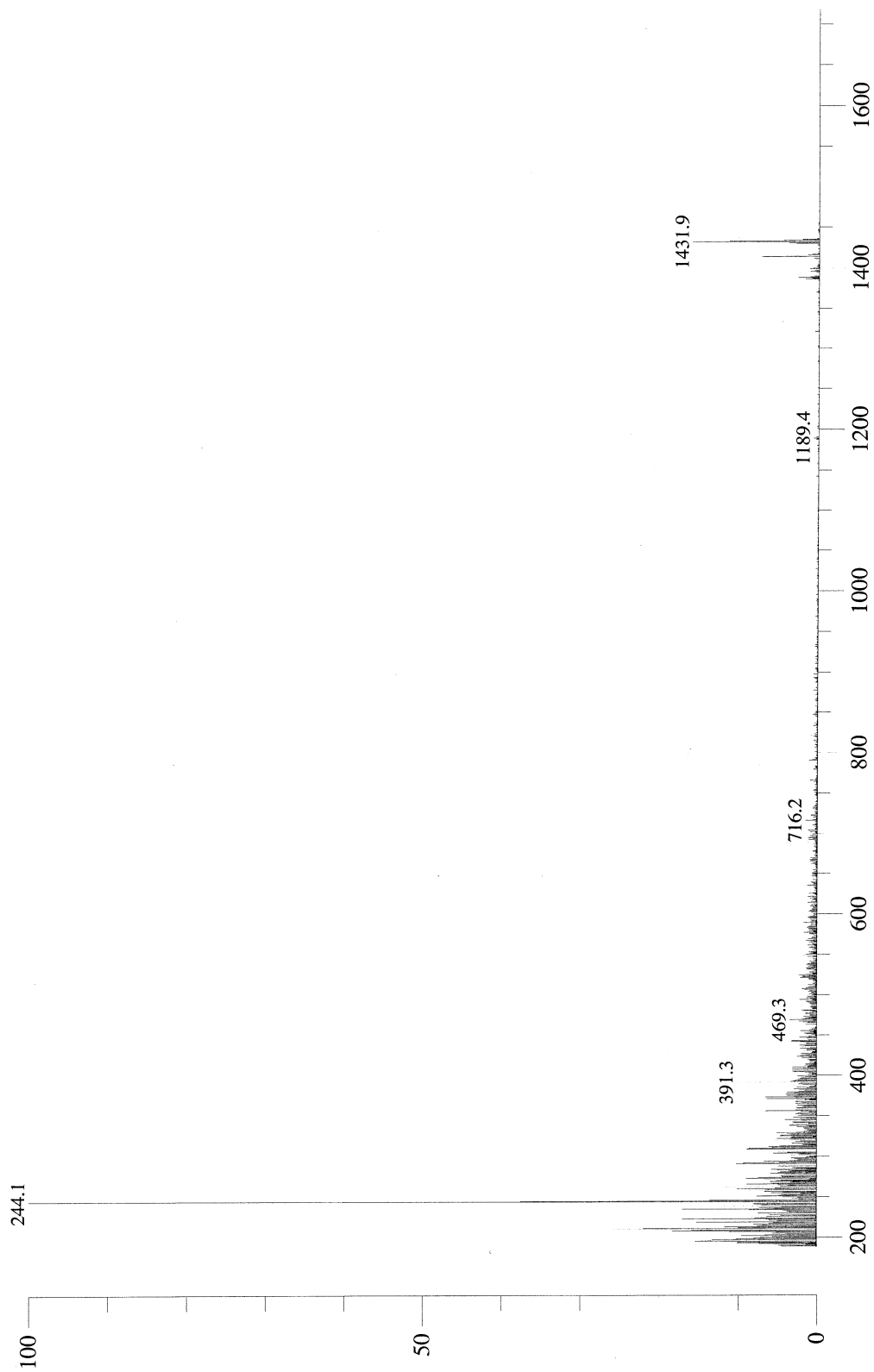


## HPLC Trace of Synthetic Petriellin A 1

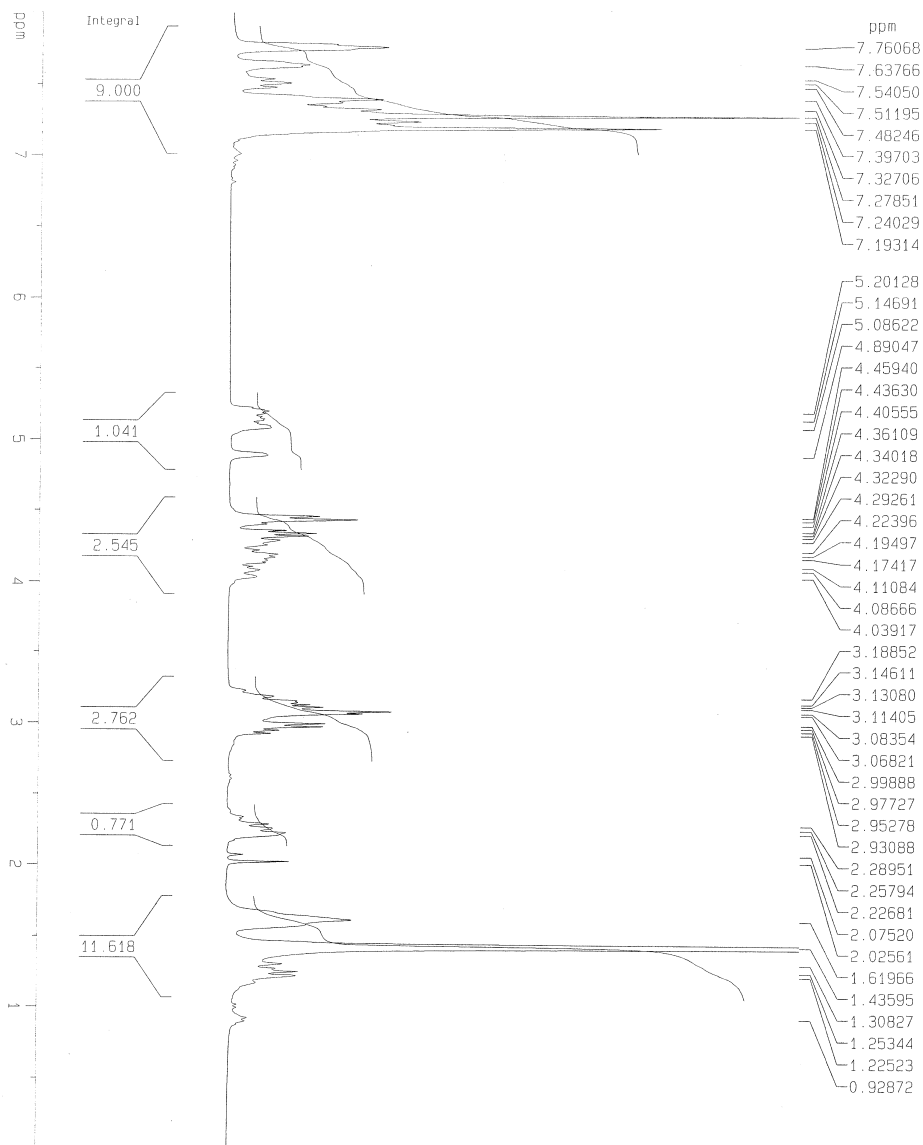


# ESMS spectrum of Synthetic Petriellin A 1

latrob0618 Scan 3 (Av 2-28 Acq) 100%=413248 mv 10 Oct 105 12:24  
LRP +LSIMS M. Sleebs MMS9 LSIMS in mnba



# <sup>1</sup>H NMR Spectrum of Compound 9



Fmoc-L-Pip-D-Phe1ac-0tBu

Current Data Parameters

NAME mas04

EXPNO 307

PROCNO 1

F2 - Acquisition Parameters

Date\_ 20060515

Time 16.01

INSTRUM spect

PROBHD 5 mm PROUL 13C

PULPROG zg30

TD 32768

SOLVENT CDCl3

DS 8

NS 2

SMH 3631.073 Hz

FIDRES 0.107760 Hz

AQ 4.639089 sec

RG 20.2

DM 141.600 usec

DE 6.00 usec

TE 300.0 K

D1 1.70000005 sec

MCRES1 0.00000000 sec

MCNRK 0.01500000 sec

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

NUC1 1H

P1 8.00 usec

PL1 -2.25 dB

SFO1 300.1317000 MHz

F2 - Processing parameters

SF 16384

SI 300.1300110 MHz

WDW EM

SSB 0

LB 0.30 Hz

GB 0

PC 1.00

1D NMR plot parameters

CX 21.00 cm

CY 0.00 cm

CP 8.000 ppm

F1 2401.04 Hz

F2 0.000 ppm

F2 0.00 Hz

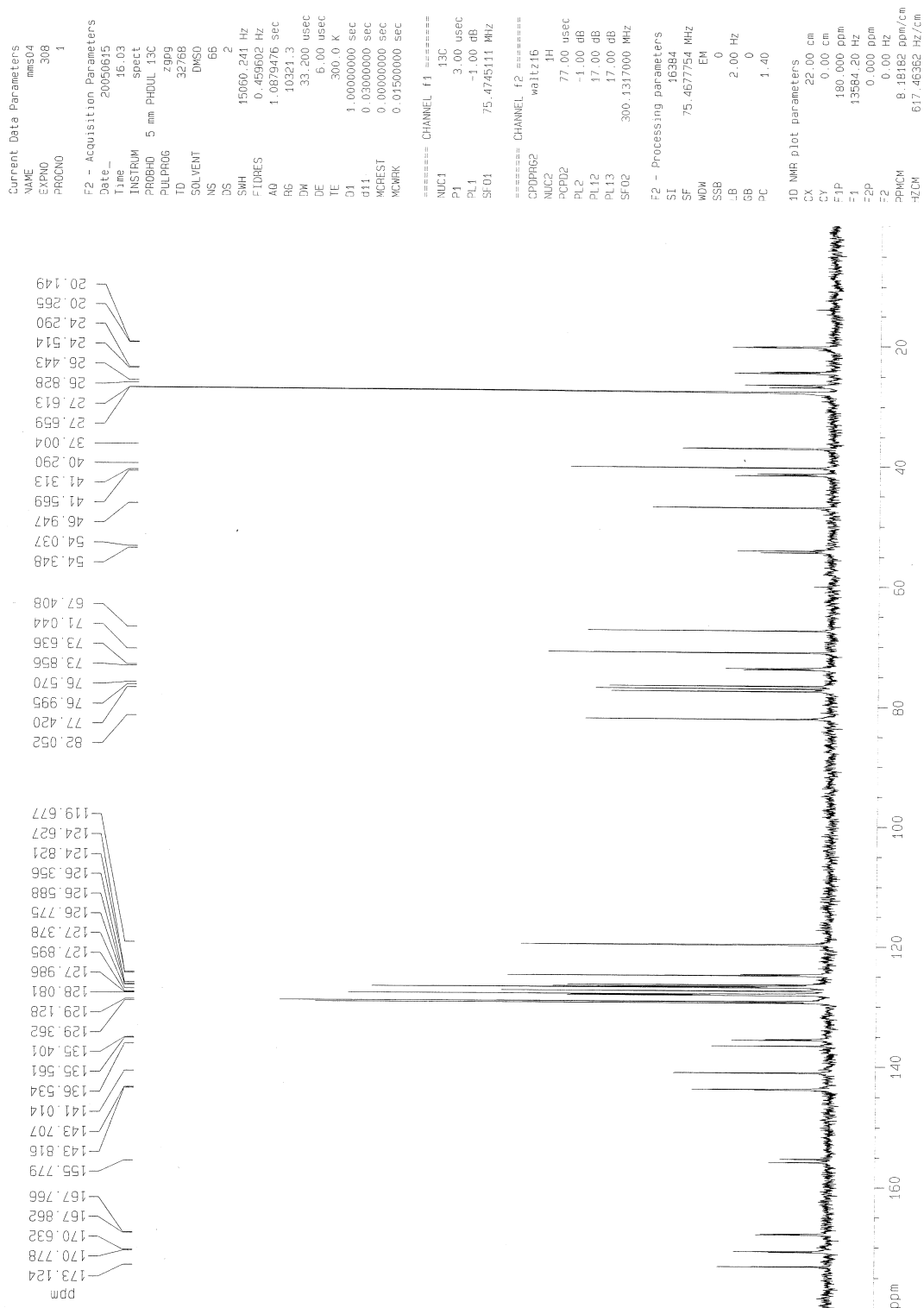
PPMCM 0.38095 ppm/cm

HZCM 114.33624 Hz/cm



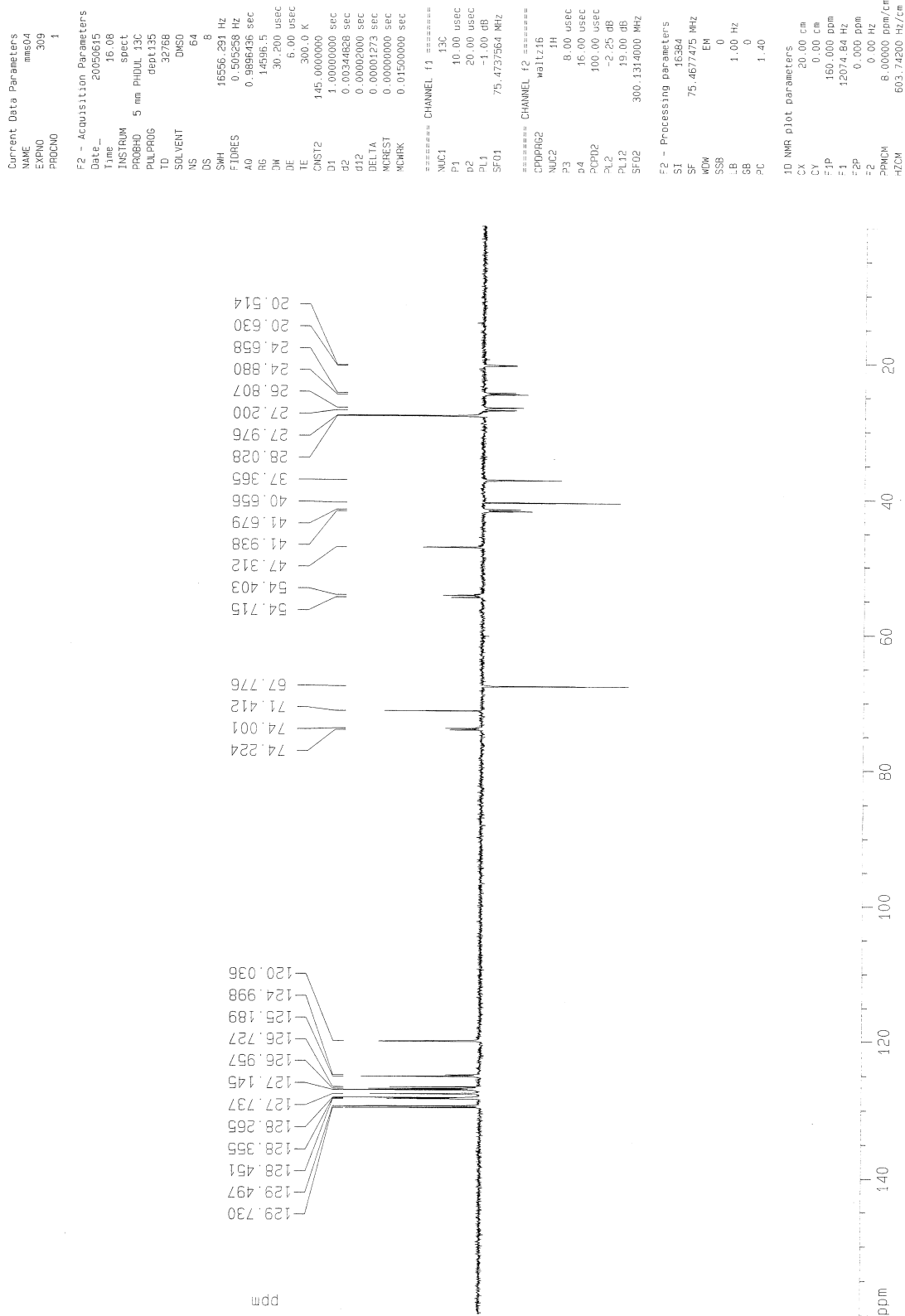
# <sup>13</sup>C NMR Spectrum of Compound 9

Fmoc-L-Pip-D-PheIac-OtBu

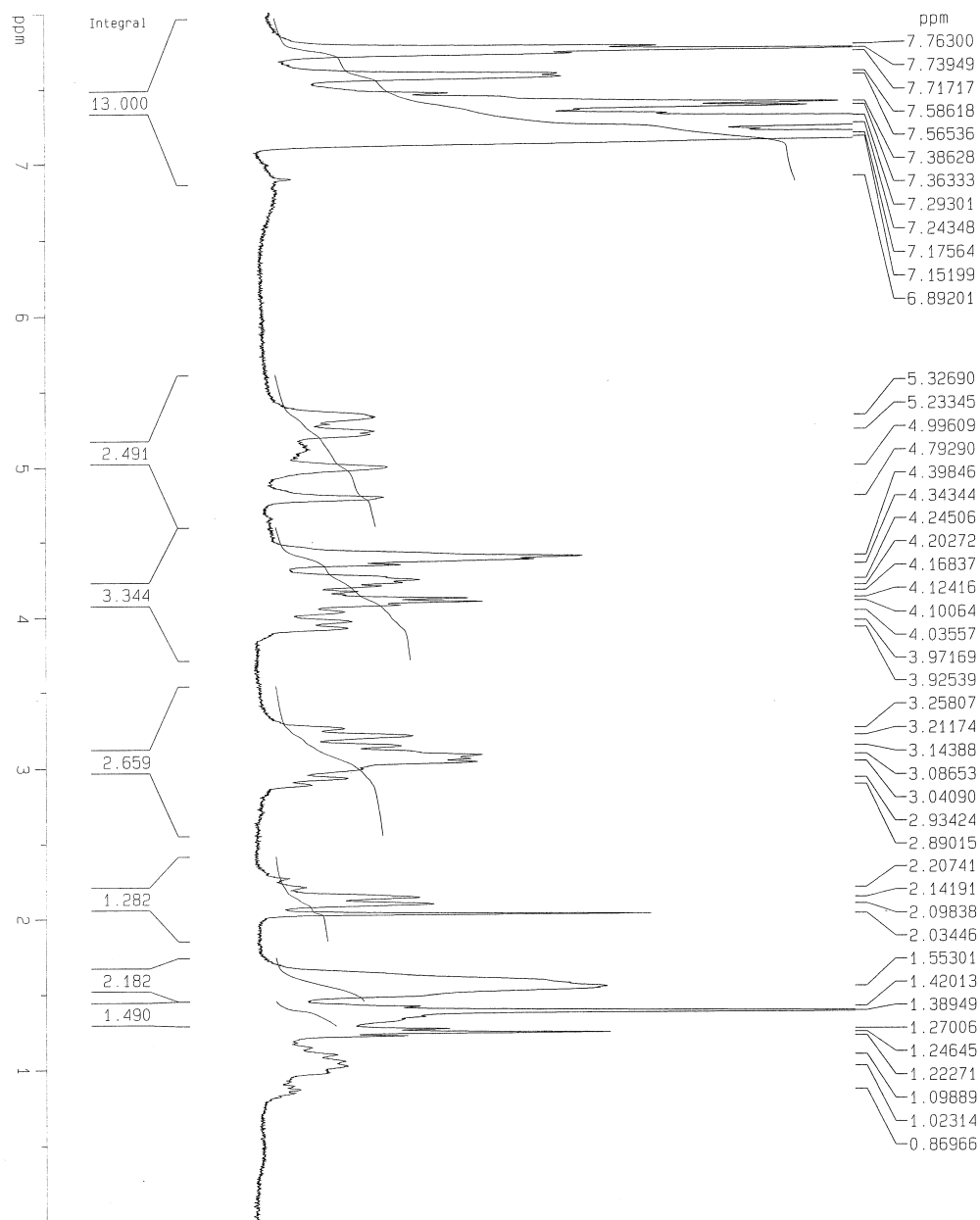


# <sup>13</sup>C DEPT NMR Spectrum of Compound 9

Fmoc-L-Pip-D-Phelac-OtBu



# <sup>1</sup>H NMR Spectrum of Compound 10



Fmoc-L-pip-D-phe1ac-OH

Current Data Parameters  
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EXPNO 285  
PROCNO 1

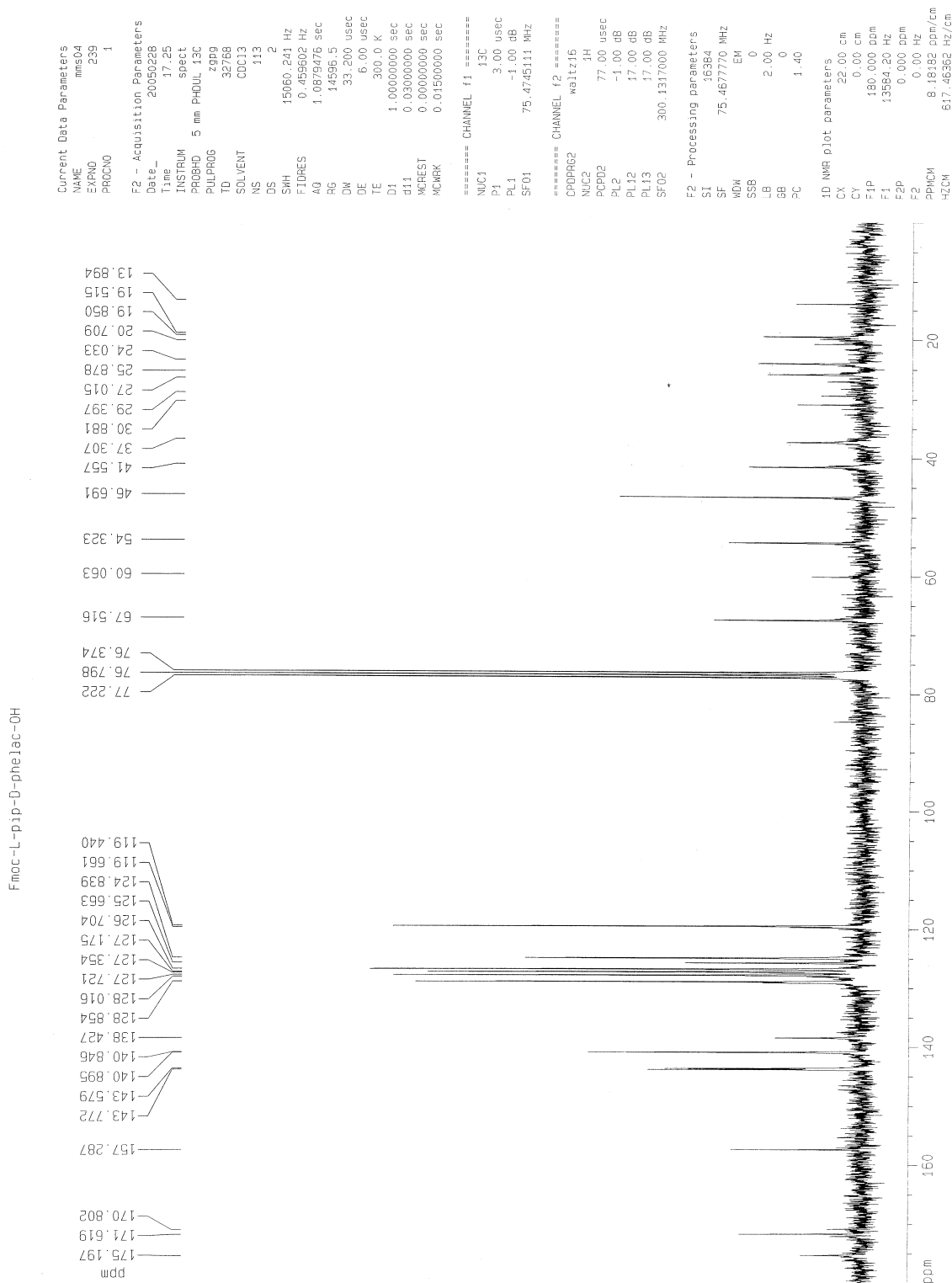
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TD 32768  
SOLVENT DMS  
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DS 2  
SWH 3531.073 Hz  
FIDRES 0.107760 Hz  
AQ 4.639989 sec  
RG 256  
DM 141.600 usec  
DE 5.00 usec  
TE 300.0 K  
D1 1.70000005 sec  
MCREST 0.00000000 sec  
MCWRK 0.01500000 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 8.00 usec  
PL1 -2.25 dB  
SFO1 300.1317000 MHz

F2 - Processing parameters  
SI 16384  
SF 300.1300110 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 21.00 cm  
CY 0.00 cm  
F1P 8.000 ppm  
F1 2401.04 Hz  
F2P 0.000 ppm  
F2 0.00 Hz  
PPMCM 0.38095 ppm/cm  
HZCM 114.33524 Hz/cm

# <sup>13</sup>C NMR Spectrum of Compound 10



# <sup>13</sup>C DEPT NMR Spectrum of Compound 10

