

Supporting Information

Rec. Nat. Prod. 10:4 (2016) 397-406

The First Solid-phase Synthesis and Structural Studies on Phakellistatin 15

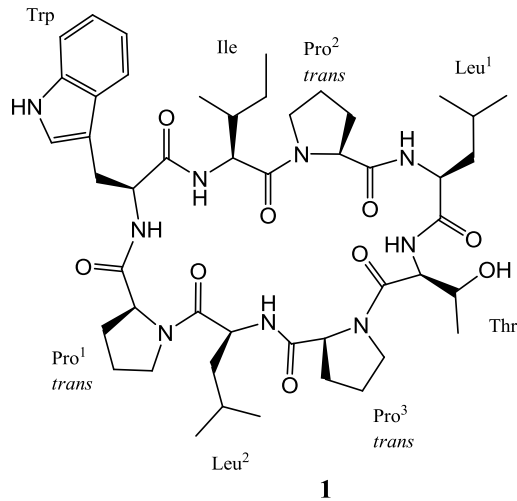
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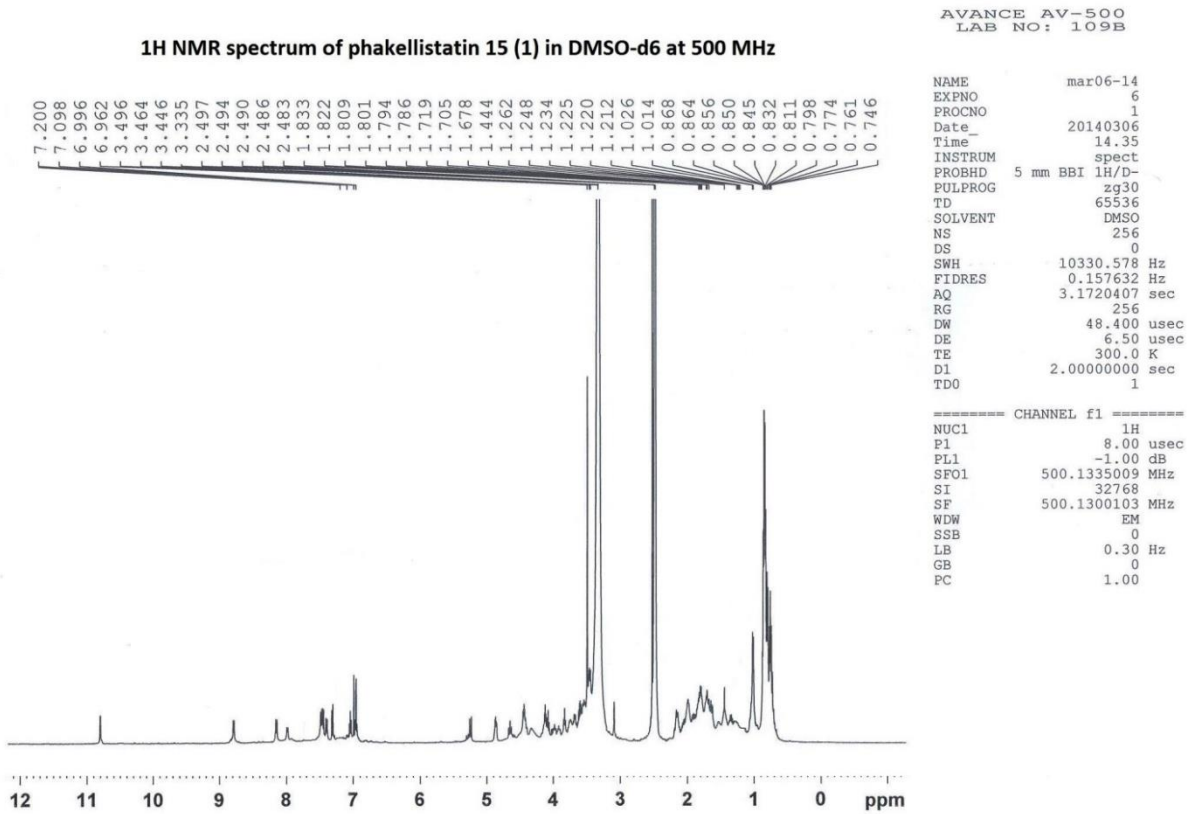
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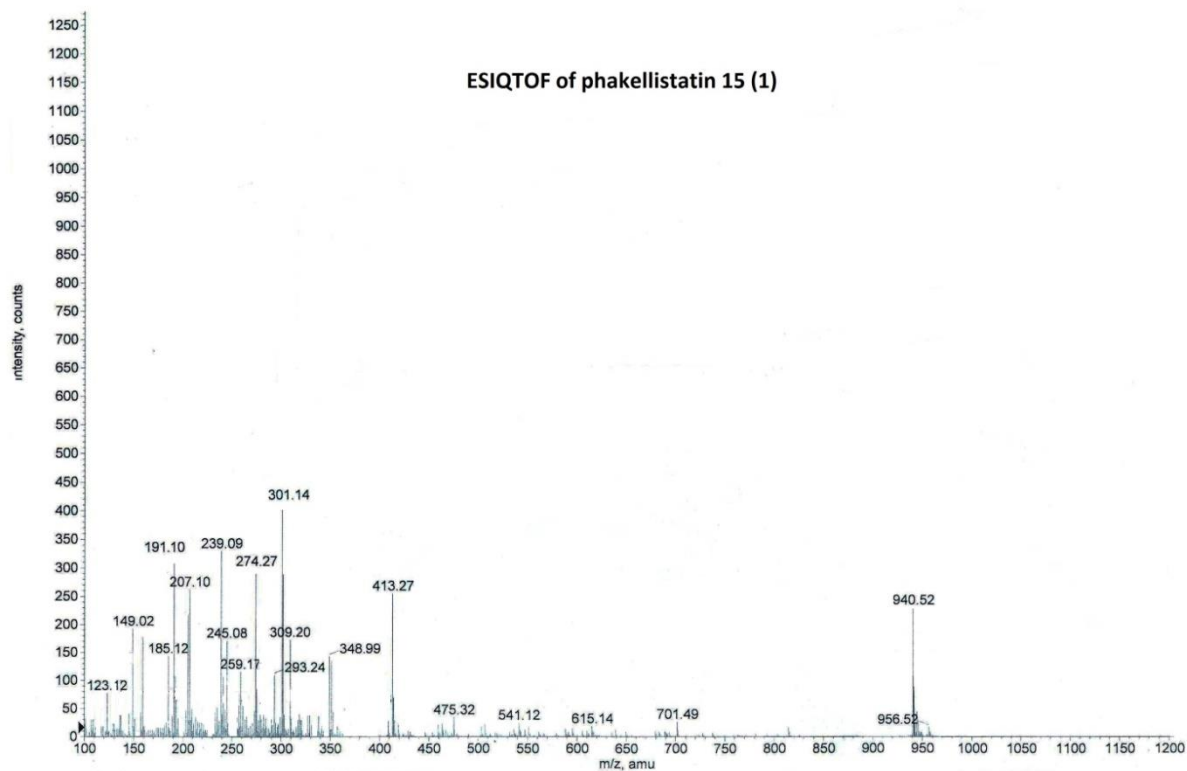
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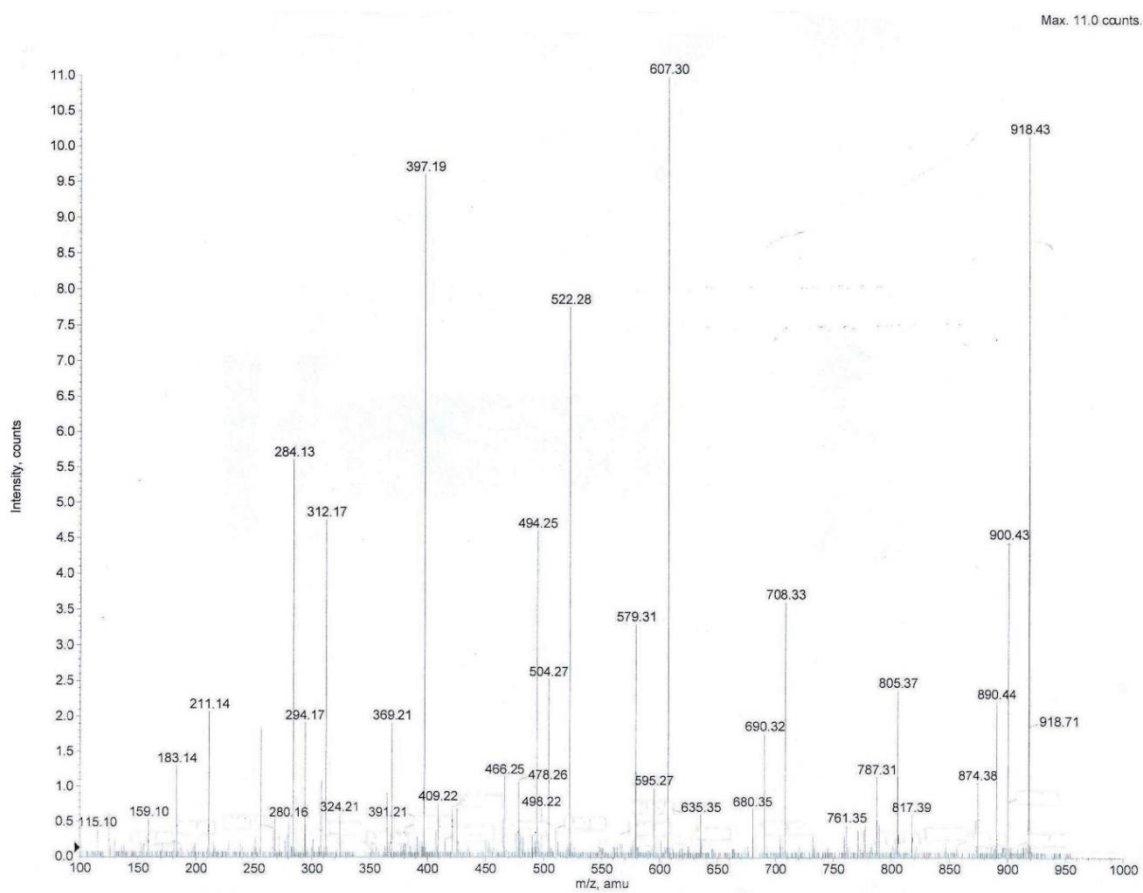
¹H NMR spectrum of phakellistatin 15 (1) in DMSO-d₆ at 500 MHz



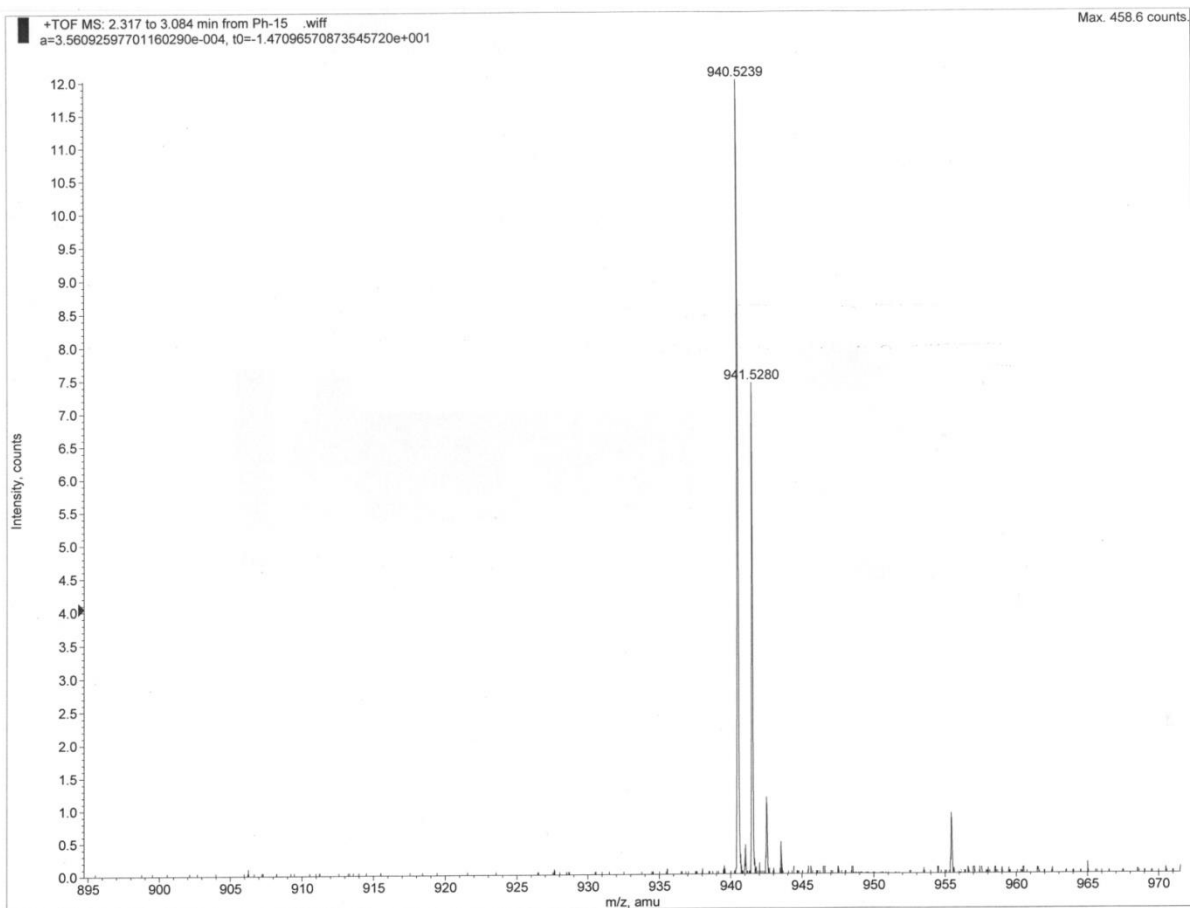
S1: ¹H NMR Spectra of synthetic phakellistatin 15 (1)



S2: ESIMS Spectra of synthetic phakellistatin 15 (1)



S3: ESIMSMS Spectra of peak 918 of synthetic phakellistatin 15 (1)



Elemental composition calculator

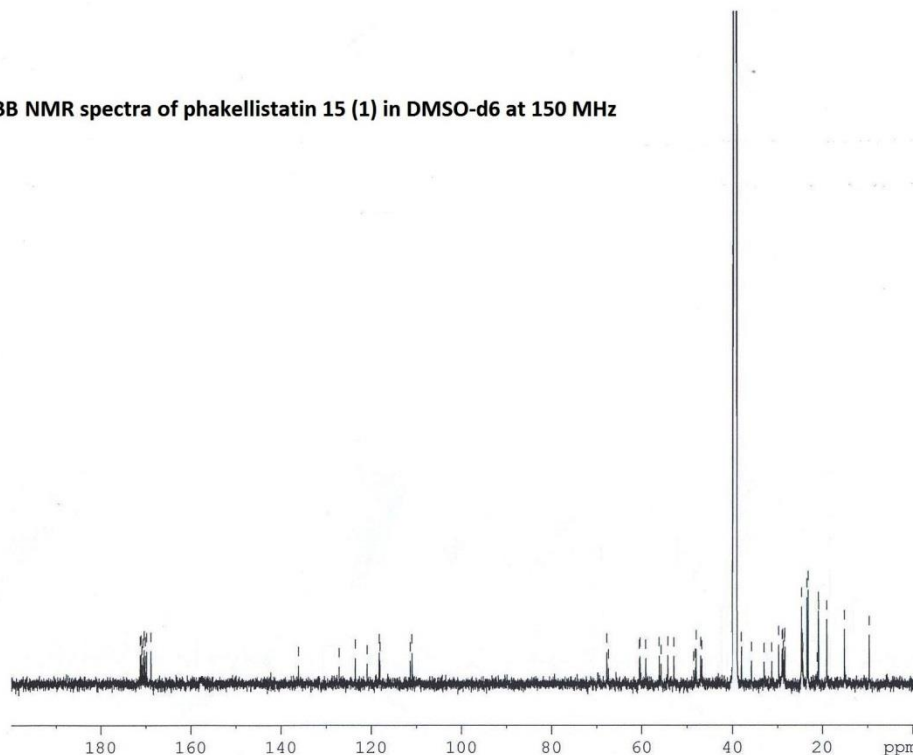
Target m/z: +940.5239 amu
 Tolerance: +10.0000 ppm
 Result type: Elemental
 Max num of results: 100
 Min DBE: -0.5000 Max DBE: +50.0000
 Electron state: OddAndEven
 Num of charges: 0
 Add water: N/A
 Add proton: N/A
 File Name: Ph-15 .wiff

	Elements	Min Number	Max Number
1	C	0	50
2	H	0	80
3	N	0	10
4	O	0	10
5	Na	0	1

	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C48 H71 N9 O9 Na	940.5272	-3.3453	-3.5569	17.5
2	C50 H73 N6 O10 Na	940.5285	-4.6880	-4.9845	17.0
3	C50 H70 N9 O9	940.5296	-5.7506	-6.1142	20.5
4	C49 H68 N10 O9	940.5170	6.8254	7.2570	21.0
5	C49 H71 N7 O10 Na	940.5160	7.8880	8.3868	17.5
6	C47 H69 N10 O9 Na	940.5146	9.2307	9.8144	18.0

S4:HR-ESIMS Spectra of synthetic phakellistatin 15 (1)

¹³C/ BB NMR spectra of phakellistatin 15 (1) in DMSO-d6 at 150 MHz



AVANCE AV-600
CRYO PROBE
LAB NO: 108

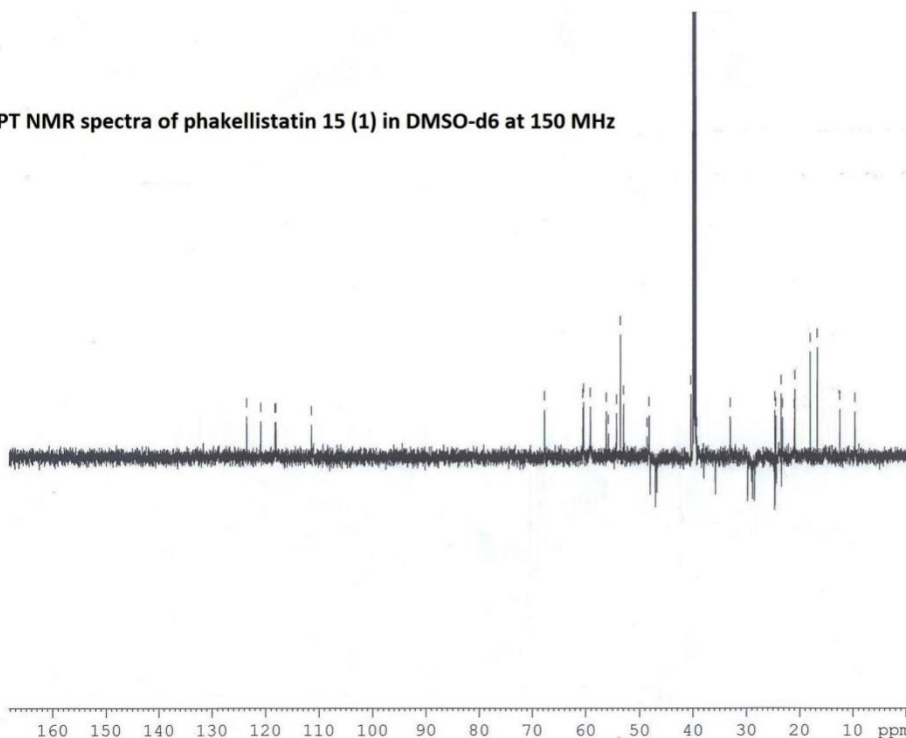
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NAME          mar08-14
EXPNO         6
PROCNO        1
Date_         20140309
Time          12.25
INSTRUM       spect
PROBHD        5 mm CPTCI 1H-
PULPROG       zgpg
TD            32768
SOLVENT       DMSO
NS            14336
DS            2
SWH           35971.223 Hz
FIDRES        1.097755 Hz
AQ            0.4555391 sec
RG            32768
DW            13.900 usec
DE            6.50 usec
TE            298.0 K
D1            1.50000000 sec
D11           0.03000000 sec
TD0           14

===== CHANNEL f1 =====
NUC1          13C
P1            15.40 usec
PL1           1.00 dB
PL1W          83.60149384 W
SFO1          150.9453107 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         65.00 usec
PL2           3.30 dB
PL12          22.06 dB
PL13          27.00 dB
PL2W          9.16420078 W
PL12W         0.12192553 W
PL13W         0.03909260 W
SFO2          600.2336014 MHz
SI            16384
SF            150.9280214 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.00
    
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¹³C/DEPT NMR spectra of phakellistatin 15 (1) in DMSO-d6 at 150 MHz



AVANCE AV-600
CRYO PROBE
LAB NO: 108

```

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PROCNO        1
Date_         20140309
Time          20.29
INSTRUM       spect
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PULPROG       deptsp135
TD            32768
SOLVENT       DMSO
NS            8192
DS            2
SWH           30303.031 Hz
FIDRES        0.924775 Hz
AQ            0.5407385 sec
RG            32768
DW            16.500 usec
DE            6.50 usec
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CNST2         145.0000000
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D2            0.00344828 sec
D12           0.00002000 sec
TD0           8

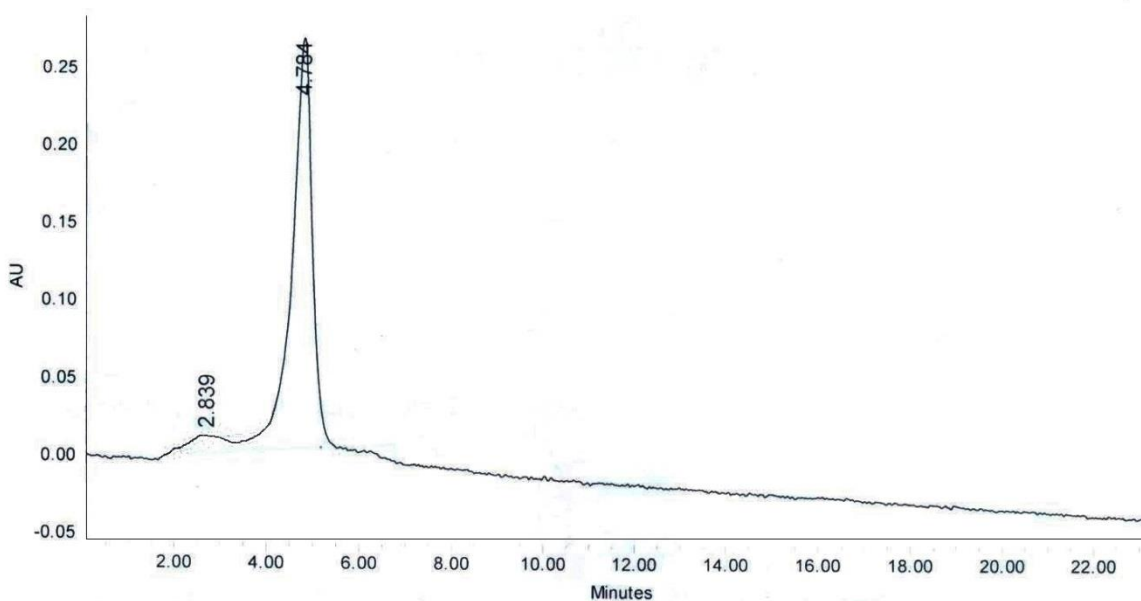
===== CHANNEL f1 =====
NUC1          13C
P1            15.40 usec
P12           2000.00 usec
PL0           120.00 dB
PL1           1.00 dB
PL0W          0.00000000 W
PL1W          83.60149384 W
SFO1          150.9430468 MHz
SP2           5.40 dB
SPNAM2        Crp60comp.4
SPOAL2        0.500
SPOFFS2       0.00 Hz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
P3            7.50 usec
P4            15.00 usec
PCPD2         65.00 usec
PL2           3.30 dB
PL12          22.06 dB
PL2W          9.16420078 W
PL12W         0.12192553 W
SFO2          600.2324009 MHz
SI            32768
SF            150.9280214 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.20
    
```

S5: ¹³C Spectra of synthetic phakellistatin 15 (1)

SAMPLE INFORMATION

Sample Name: Ph-15(iv)	Acquired By: Maria
Sample Type: Unknown	Date Acquired: 8/13/2014 11:26:10 AM
Vial: 1	Acq. Method Set: ASAD
Injection #: 1	Date Processed: 8/13/2014 12:24:17 PM
Injection Volume: 100.00 ul	Processing Method: Default
Run Time: 40.0 Minutes	Channel Name: WWin Ch1
Sample Set Name:	Proc. Chnl. Descr.: PDA 282.0 nm



Processed Channel: PDA 282.0 nm

Processed Channel	Retention Time (min)	Area	% Area	Height
1 PDA 282.0 nm	2.839	268667	3.66	13830
2 PDA 282.0 nm	4.784	7067404	96.34	256522

S6: HPLC Profile of synthetic phakellistatin 15 (1)