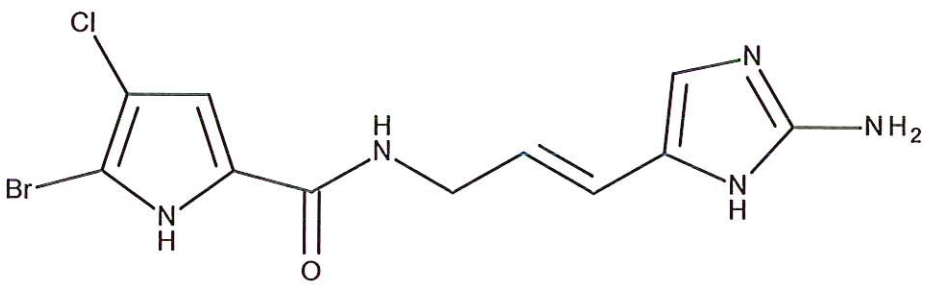
HARBOR BRANCH

FLORIDA ATLANTIC UNIVERSITY

HBOI-367	
BROMO-CHLORO-OROIDIN	
$C_{11}H_{11}BrClN_5O$	344.59
Axinellida	
Manuscript in prep	

☐ isolation scheme (Visio) attached

☐ hplc method (trace attached)

Method: Vydac C18 Protein and Peptide, 4.6 x 250mm, 10µ
flow 1 ml/min, detection: PDA: UV (extracted at 220 nm, black); ELSD (green dotted)
A: H_2O:CH_3CN (95:5, v/v, 0.1% TFA), B: CH_3CN (0.1% TFA)
t=0 min A:B (90:10, v/v), t=20 min (100%B) , t=28 min (100%B)

☐ LC-MS method

Method: Vydac C18 Protein and Peptide, 2.1x150 mm, flow 0.2 ml/min
A: H_2O (0.1% formic acid), B:CH_3CN (0.1% formic acid)
t= 0 min A:B (90:10, v/v), t=15 min (100%B), t=21 min (100%B), t=22 min A:B(90:10, v/v)
using a linear gradient

☐ ^1H (600 MHz) ☐ $CDCL_3$ ☑ CD_3OD (Methanol-d_4) ☐ $CDCl_3/CD_3OD$

☐ ^{13}C (150 MHz) ☐ $CDCL_3$ ☑ CD_3OD (Methanol-d_4) ☐ $CDCl_3/CD_3OD$

☑ solubility ☐ $CHCl_3$/MeOH (9:1) ☑ MeOH ☐ DMSO

☐ estimated purity _>90_%

☐ sample weight 8.7 mg

For further information contact:
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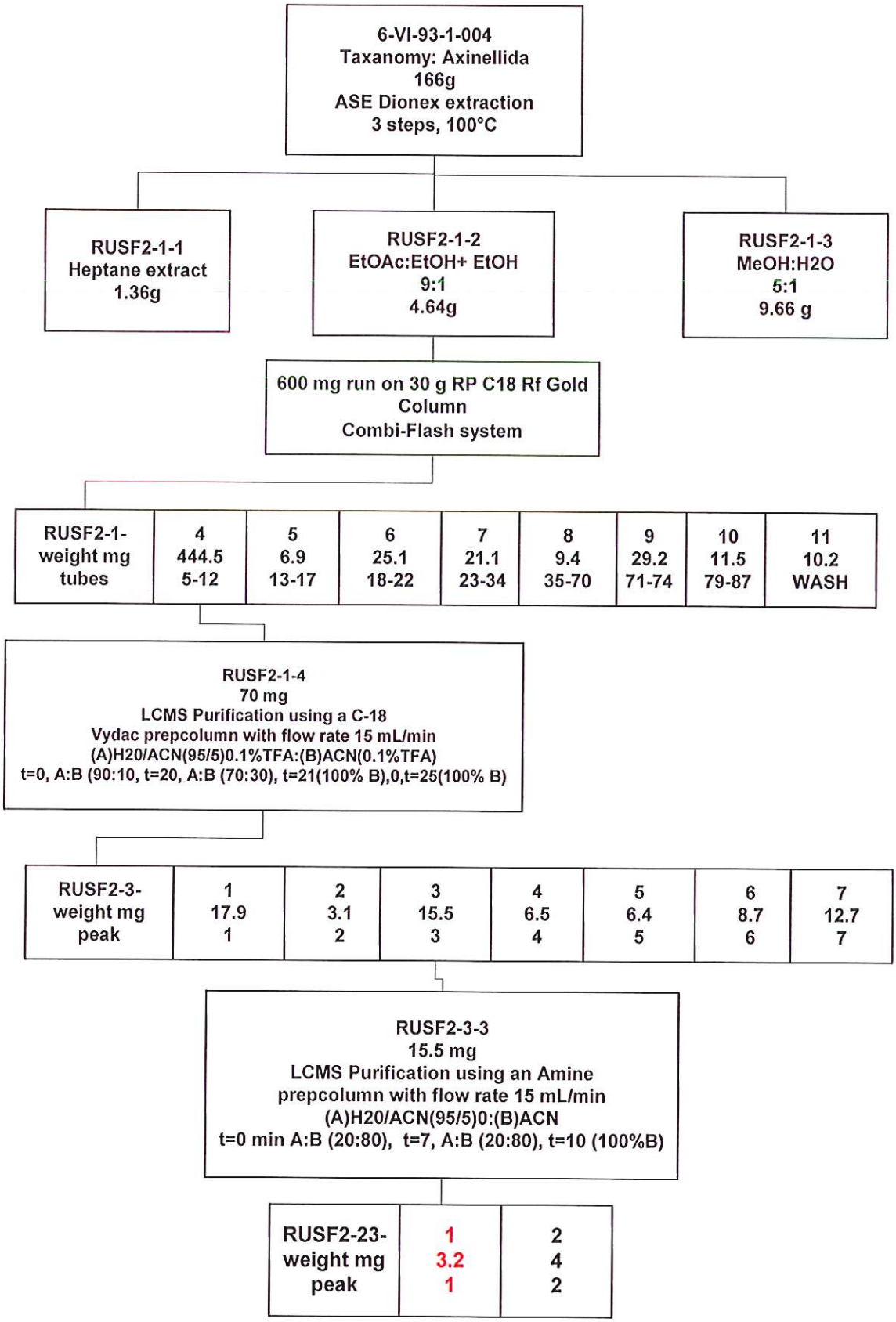
awrigh33@hboi.fau.edu, 772-242-2459

Date: 7/19/13

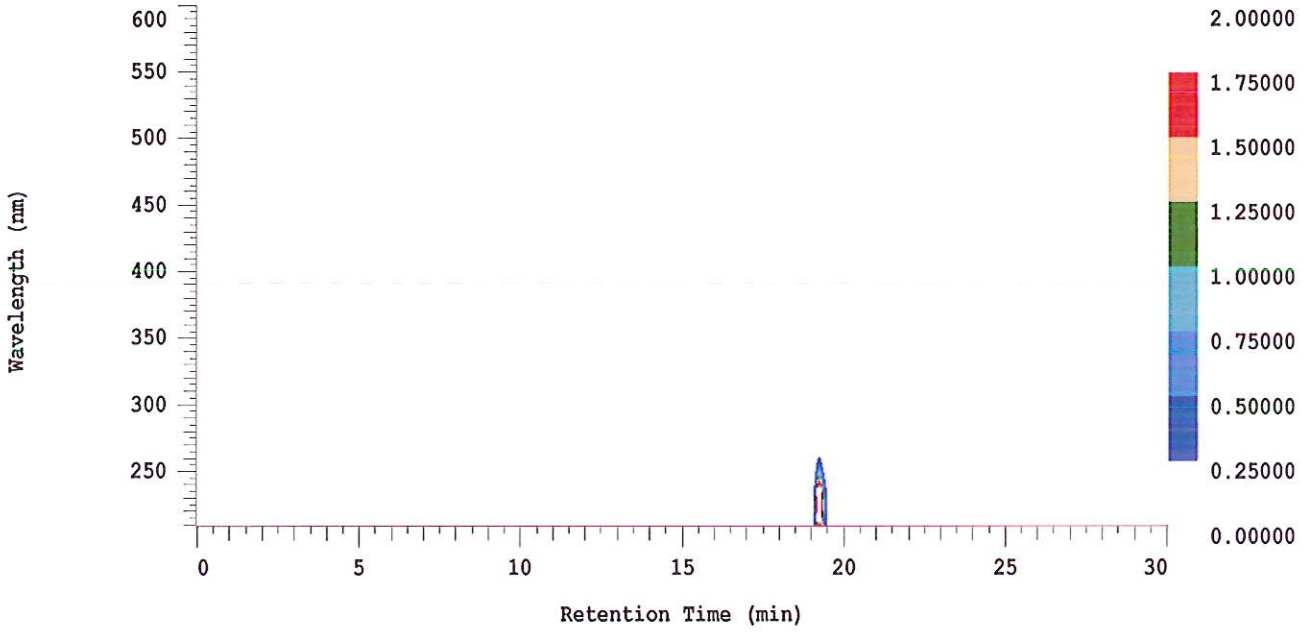

6-VI-93-1-004
Taxanomy: Axinellida
166g
ASE Dionex extraction
3 steps, 100°C
RUSF2-1-1
Heptane extract
1.36g
RUSF2-1-2
EtOAc:EtOH+ EtOH
9:1
4.64g
RUSF2-1-3
MeOH:H2O
5:1
9.66 g
600 mg run on 30 g RP C18 Rf Gold Column
Combi-Flash system
RUSF2-1-
weight mg
tubes
4
444.5
5-12
5
6.9
13-17
6
25.1
18-22
7
21.1
23-34
8
9.4
35-70
9
29.2
71-74
10
11.5
79-87
11
10.2
WASH
RUSF2-1-4
70 mg
LCMS Purification using a C-18
Vydac prepcolumn with flow rate 15 mL/min
(A)H20/ACN(95/5)0.1%TFA:(B)ACN(0.1%TFA)
t=0, A:B (90:10, t=20, A:B (70:30), t=21(100% B),0,t=25(100% B)
RUSF2-3-
weight mg
peak
1
17.9
1
2
3.1
2
3
15.5
3
4
6.5
4
5
6.4
5
6
8.7
6
7
12.7
7
RUSF2-3-3
15.5 mg
LCMS Purification using an Amine
prepcolumn with flow rate 15 mL/min
(A)H20/ACN(95/5)0:(B)ACN
t=0 min A:B (20:80), t=7, A:B (20:80), t=10 (100%B)
RUSF2-23-
weight mg
peak
1
3.2
1
2
4
2

D-2000 Elite HPLC System Manager Report

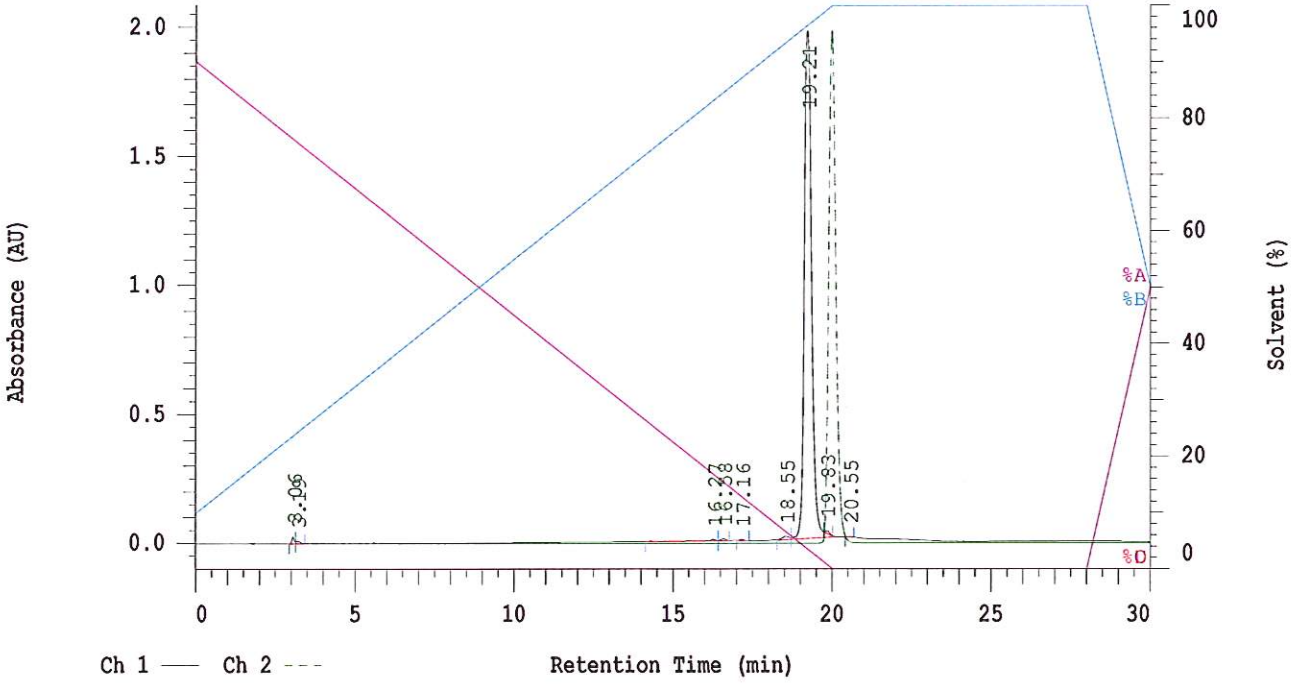
Analyzed: 06/28/2013 02:28 PM Reported: 06/28/2013 04:02 PM

Sample Name: HBOI-379

Sample Description: HBOI-379 Bebryc Cembrane



Channel 1 Chrom Type: Fixed WL Chromatogram, 230 nm

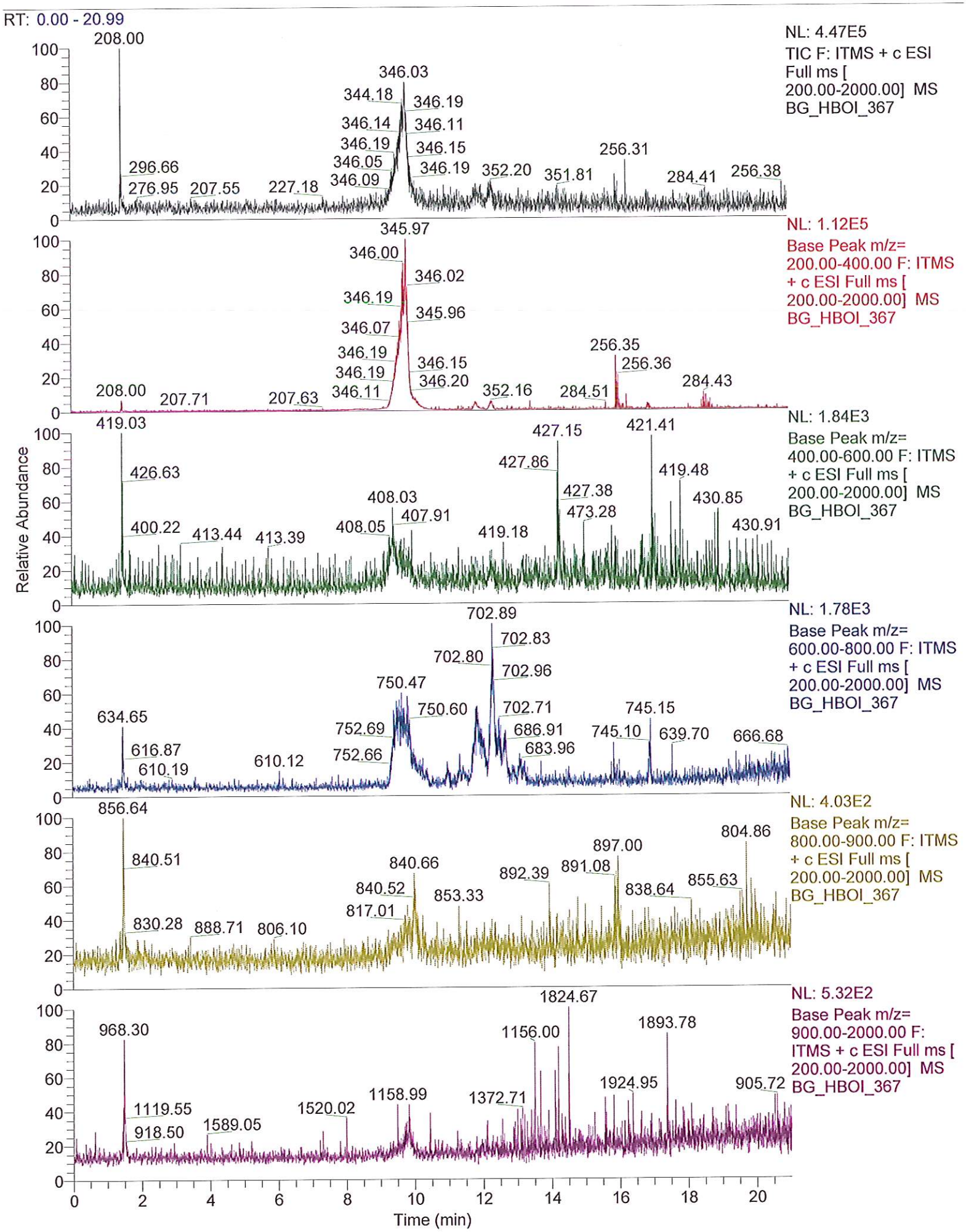


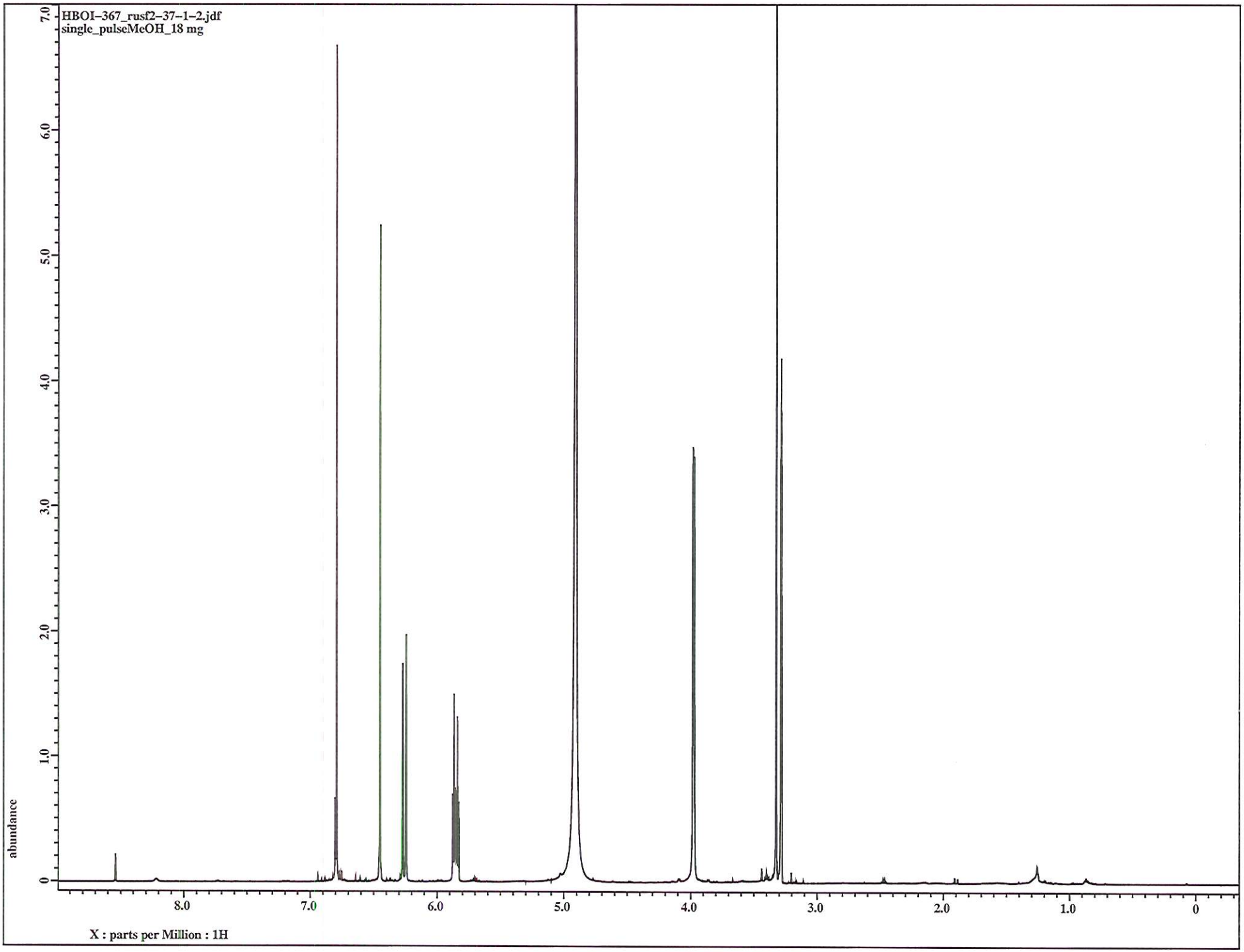
Acquisition Method: autosampler_30min_wELSD

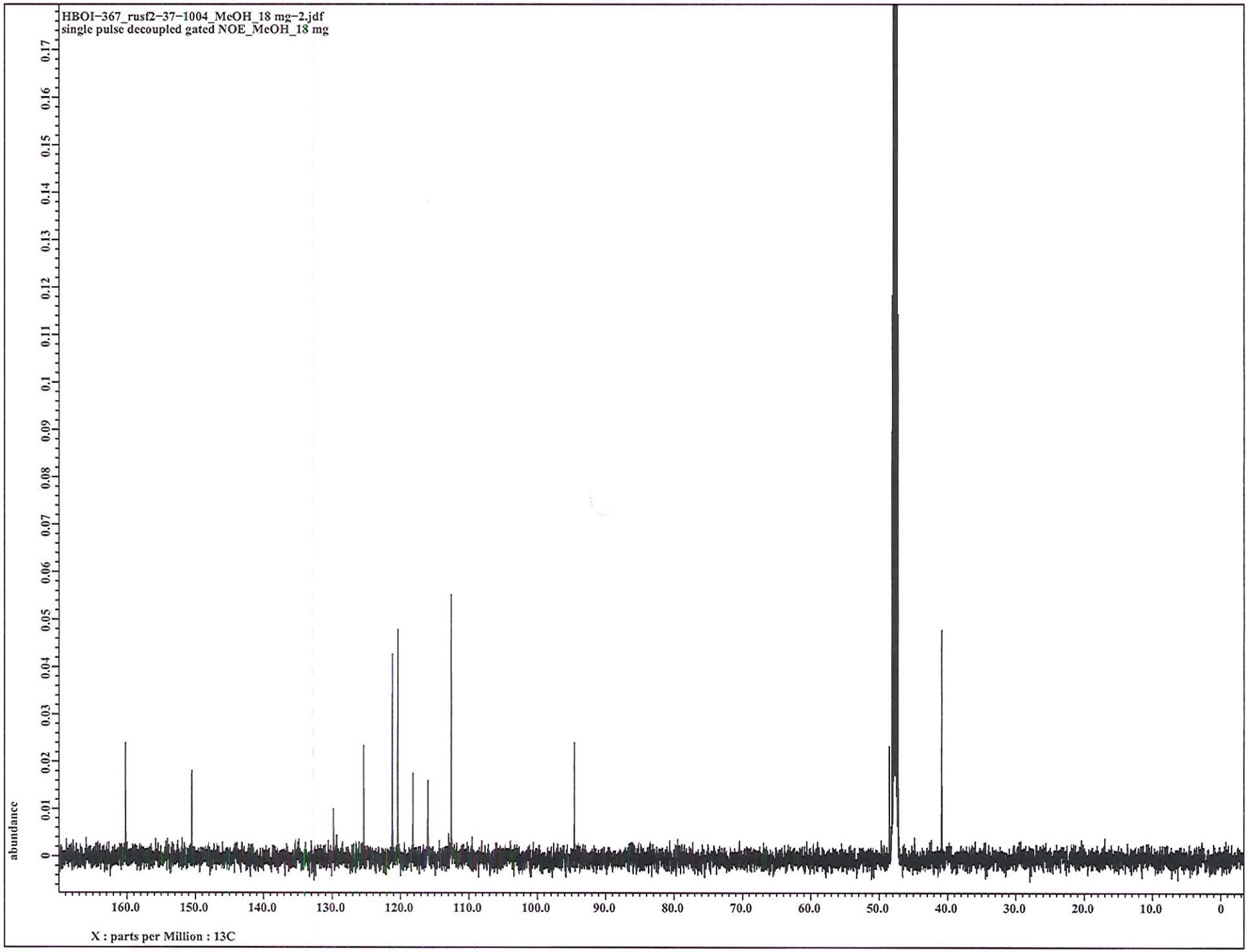
Column Type: Vydac C18

Pump A Solvent A: H2O/5% ACN Pump A Solvent B: ACN

Method Description:


RT: 0.00 - 20.99
NL: 4.47E5
TIC F: ITMS + c ESI Full ms [200.00-2000.00] MS BG_HBOI_367
NL: 1.12E5
Base Peak m/z= 200.00-400.00 F: ITMS + c ESI Full ms [200.00-2000.00] MS BG_HBOI_367
NL: 1.84E3
Base Peak m/z= 400.00-600.00 F: ITMS + c ESI Full ms [200.00-2000.00] MS BG_HBOI_367
NL: 1.78E3
Base Peak m/z= 600.00-800.00 F: ITMS + c ESI Full ms [200.00-2000.00] MS BG_HBOI_367
NL: 4.03E2
Base Peak m/z= 800.00-900.00 F: ITMS + c ESI Full ms [200.00-2000.00] MS BG_HBOI_367
NL: 5.32E2
Base Peak m/z= 900.00-2000.00 F: ITMS + c ESI Full ms [200.00-2000.00] MS BG_HBOI_367
Relative Abundance
Time (min)


HBOI-367_rusf2-37-1-2.jdf
single_pulseMeOH_18 mg
abundance
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : 1H


HBOI-367_rusf2-37-1004_MeOH_18 mg-2.jdf
single pulse decoupled gated NOE_MeOH_18 mg
abundance
0
0.01
0.02
0.03
0.04
0.05
0.06
0.07
0.08
0.09
0.1
0.11
0.12
0.13
0.14
0.15
0.16
0.17
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : 13C