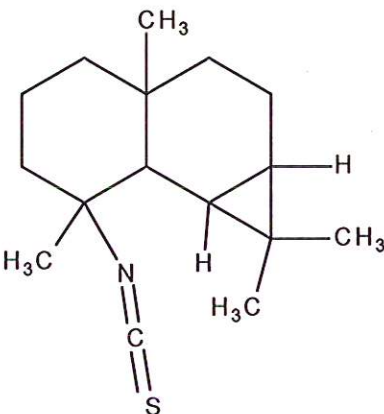


HBOI-385	
EPIPOLASIN A	
$C_{16}H_{25}NS$	263.17
Axinellida	
Capon and Macleod, 1988, <i>Aust. J. Chem.</i> , 41:979-983	

isolation scheme (Visio)

hplc method (trace attached)

Method: Vydac C18 Protein and Peptide, 4.6 x 250mm, 10 μ
 flow 1 ml/min, detection: PDA, UV (extracted at 230 nm, black); ELSD (green dotted)
 A: H₂O:CH₃CN (95:5, v/v), B: CH₃CN
 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:H₂O (0.1% formic acid), B:CH₃CN (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)

CDCl₃

CD₃OD (Methanol-d₄)

CDCl₃/CD₃OD

¹³C (150 MHz)

CDCl₃

CD₃OD (Methanol-d₄)

CDCl₃/CD₃OD

solubility

CHCl₃/MeOH (9:1)

MeOH

DMSO

estimated purity >90%

sample weight 12.5 mg

For further information contact:

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awrigh33@hboi.fau.edu, 772-242-2459

Date: 7/19/13

24-XI-92-4-002
AXINELLIDA
 ASE Dionex Extraction
 3 steps 100 degrees
 125 g of sample extracted / 75 g of sample remaining

GARJ 2-18-1
 Heptane extract
 1.1220 g

1.0959 g run on 12 g NP Rf Gold column,
 CV 16.8 ml, 30 ml/min
 CombiFlash Rf4 200 psi system
 Heptane to EtOAc gradient
 EtOAc to MeOH 70% gradient

GARJ 2-18- Weight Tubes Notes	4 49 mg Tube 2	5 623.5 mg Tubes 3-7	6 120.8 mg Tubes 8-11
GARJ 2-18- Weight Tubes Notes	7 267.8 mg Tubes 12-16	8 27.3 mg Tubes 17-19	9 23.7 mg Tubes 20-END

GARJ 2-18-2
 EtOAc: EtOH extract
 0.6241 g

0.6018 g run on 15.5 g C18 RP Rf Gold column,
 CV 13.5 ml, 30 ml/min
 Combi-Flash Rf4 200 psi system,
 H2O to ACN gradient
 MeOH to DCM gradient
 ACN:H2O:TFA 80:20 0.1% Wash

GARJ 2-18- Weight Tubes Notes	10 296.4 mg Tubes 2-4	11 14.8 mg Tubes 5-9	12 44.5 mg Tubes 10-14
GARJ 2-18- Weight Tubes Notes	13 148.8 mg Tubes 15-17	14 35.9 mg Tubes 18-20	15 5.5 mg Tubes 21-END
GARJ 2-18- Weight Tubes Notes	16 11.6 mg TFA Wash		

GARJ 2-18-3
 MeOH: H2O extract
 5.6669 g

HPLC tests were performed. Due to the
 lack of peaks, it was decided that a
 Combi-Flash run should not be performed.

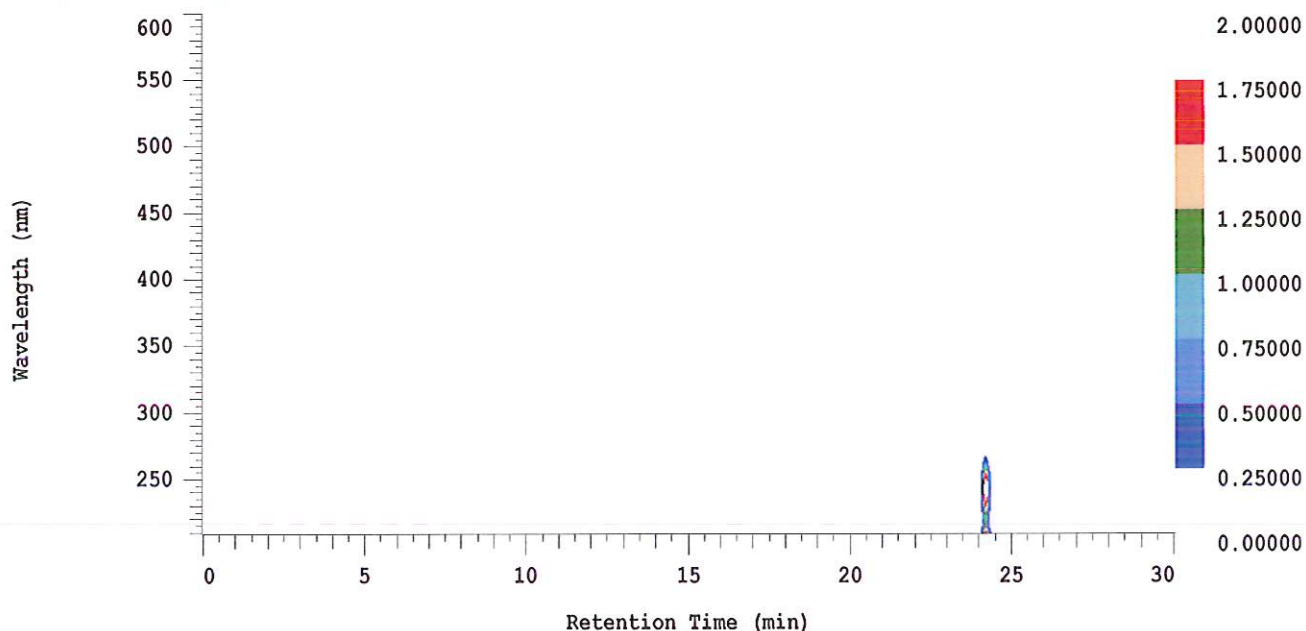
D-2000 Elite HPLC System Manager Report

Analyzed: 07/19/2013 10:48 AM

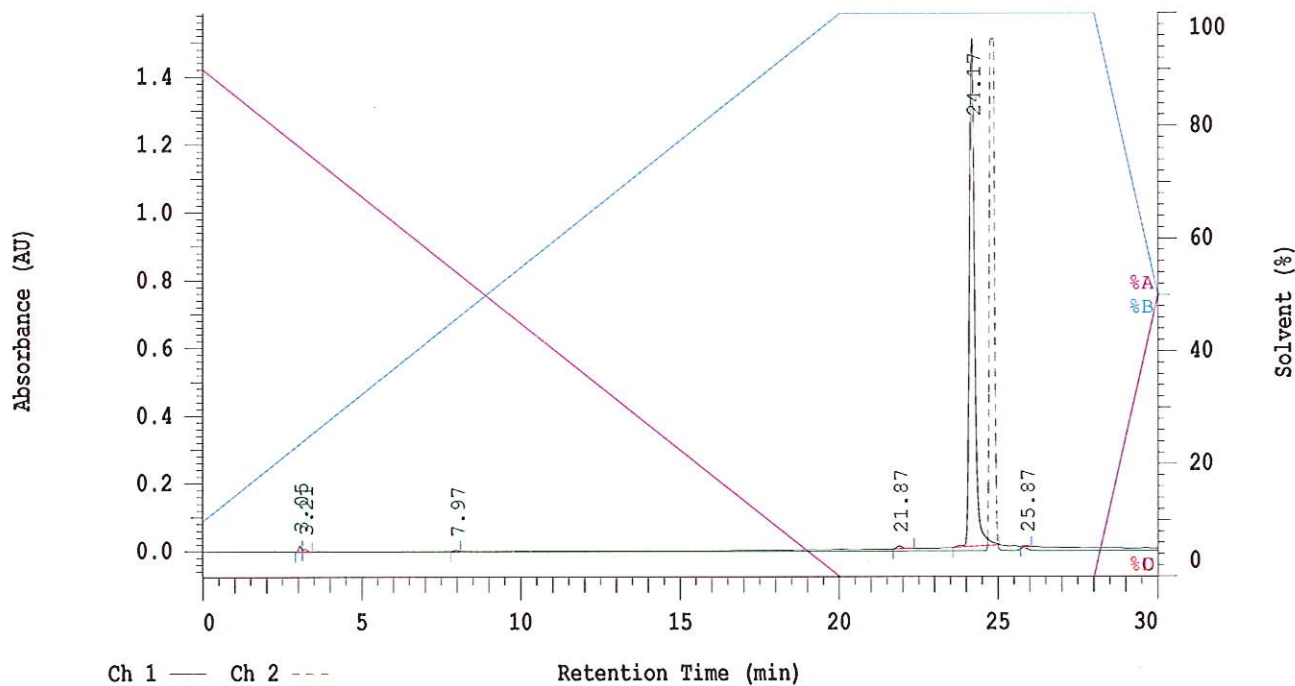
Reported: 07/19/2013 11:20 AM

Sample Name: HBOI-385

Sample Description: HBOI-385



Channel 1 Chrom Type: Fixed WL Chromatogram, 230 nm



Acquisition Method: autosampler_30min_wELSD

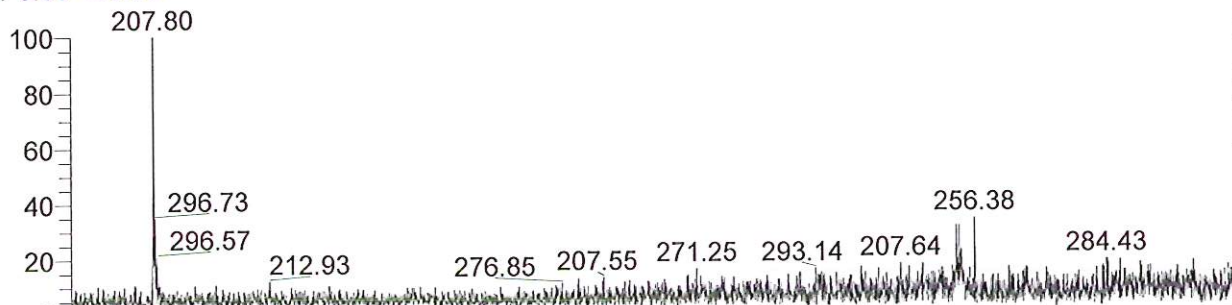
Column Type: Vydac C18

Pump A Solvent A: H2O/5% ACN

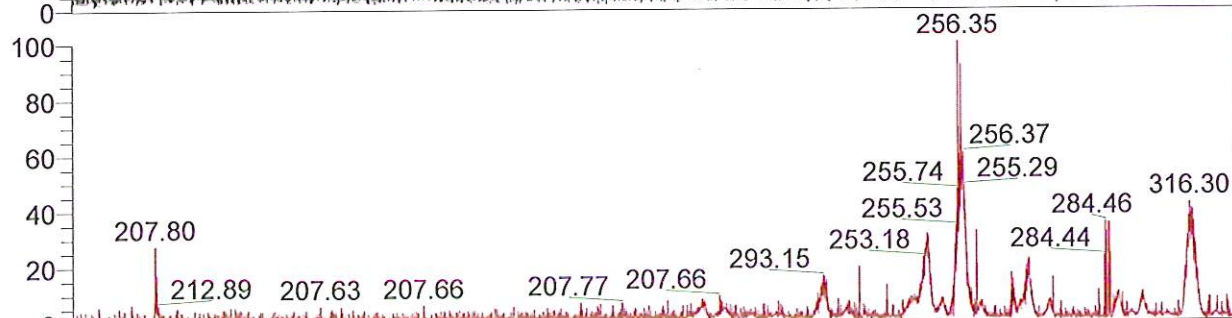
Pump A Solvent B: ACN

Method Description:

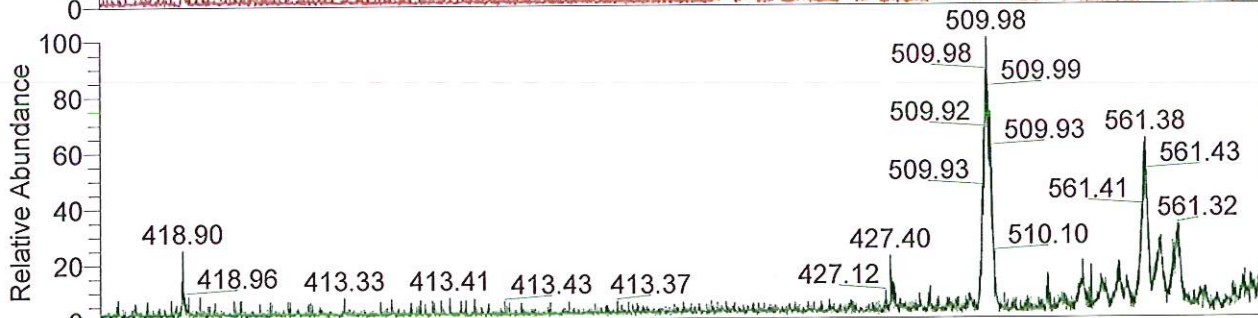
RT: 0.00 - 20.99



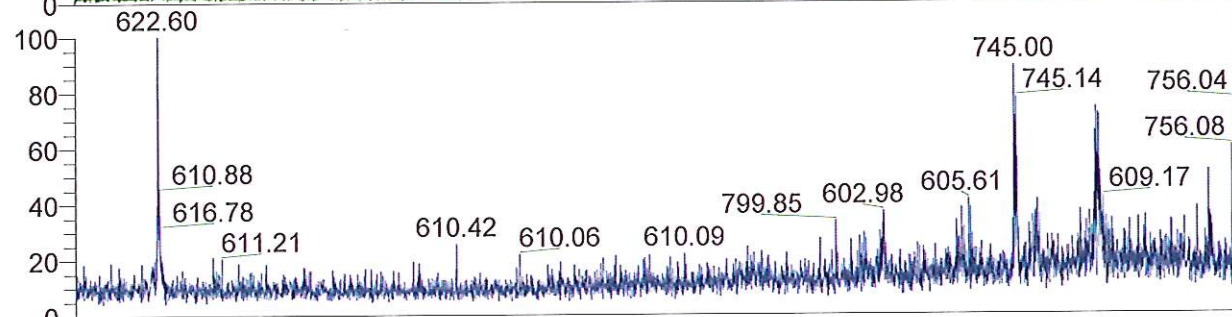
NL: 4.90E5
TIC F: ITMS + c ESI
Full ms [
200.00-2000.00] MS
BG_HBOI_385



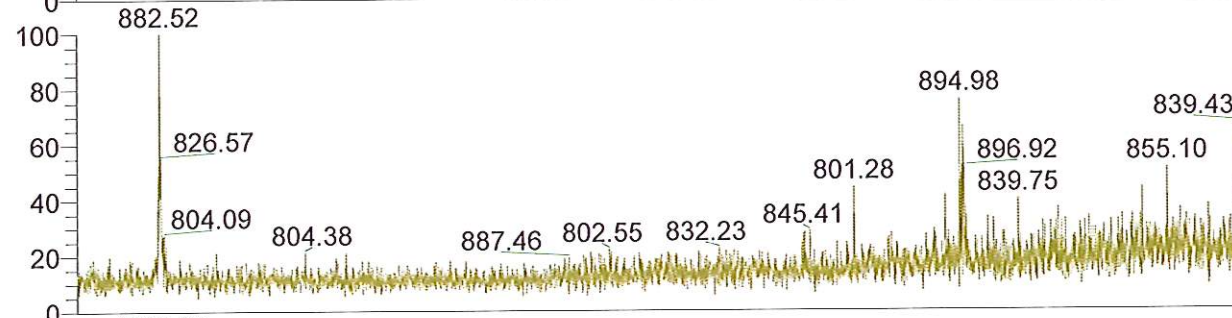
NL: 3.34E4
Base Peak m/z=
200.00-400.00 F: ITMS
+ c ESI Full ms [
200.00-2000.00] MS
BG_HBOI_385



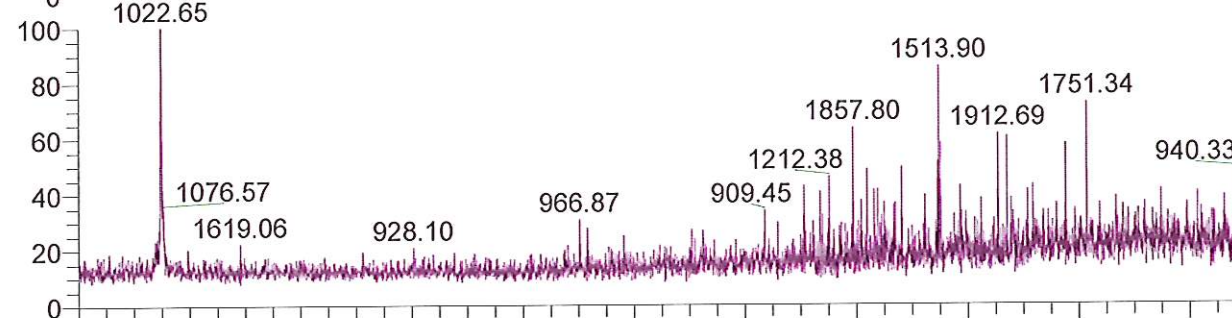
NL: 7.80E3
Base Peak m/z=
400.00-600.00 F: ITMS
+ c ESI Full ms [
200.00-2000.00] MS
BG_HBOI_385



NL: 1.00E3
Base Peak m/z=
600.00-800.00 F: ITMS
+ c ESI Full ms [
200.00-2000.00] MS
BG_HBOI_385



NL: 5.82E2
Base Peak m/z=
800.00-900.00 F: ITMS
+ c ESI Full ms [
200.00-2000.00] MS
BG_HBOI_385



NL: 5.50E2
Base Peak m/z=
900.00-2000.00 F:
ITMS + c ESI Full ms [
200.00-2000.00] MS
BG_HBOI_385

Time (min)

