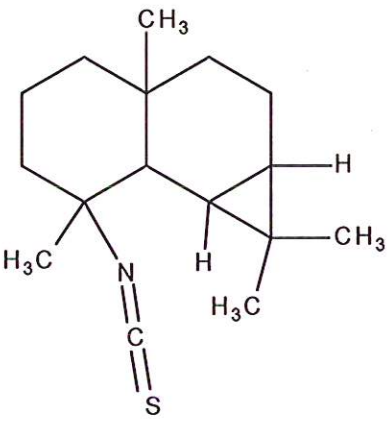


HBOI-385	
EIPOLASIN A	
$C_{16}H_{25}NS$	263.17
Axinellida	
Capon and Macleod, 1988, <i>Aust. J. Chem.</i> , 41:979-983	

isolation scheme (Visio)

hplc method (trace attached)

Method: Vydac C18 Protein and Peptide, 4.6 x 250mm, 10 $\mu$   
 flow 1 ml/min, detection: PDA, UV (extracted at 230 nm, black); ELSD (green dotted)  
 A: H<sub>2</sub>O:CH<sub>3</sub>CN (95:5, v/v), B: CH<sub>3</sub>CN  
 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<sub>2</sub>O (0.1% formic acid), B:CH<sub>3</sub>CN (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

<sup>1</sup>H (600 MHz)

CDCl<sub>3</sub>

CD<sub>3</sub>OD (Methanol-d<sub>4</sub>)

CDCl<sub>3</sub>/CD<sub>3</sub>OD

<sup>13</sup>C (150 MHz)

CDCl<sub>3</sub>

CD<sub>3</sub>OD (Methanol-d<sub>4</sub>)

CDCl<sub>3</sub>/CD<sub>3</sub>OD

solubility

CHCl<sub>3</sub>/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](mailto: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	<b>4</b> 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.

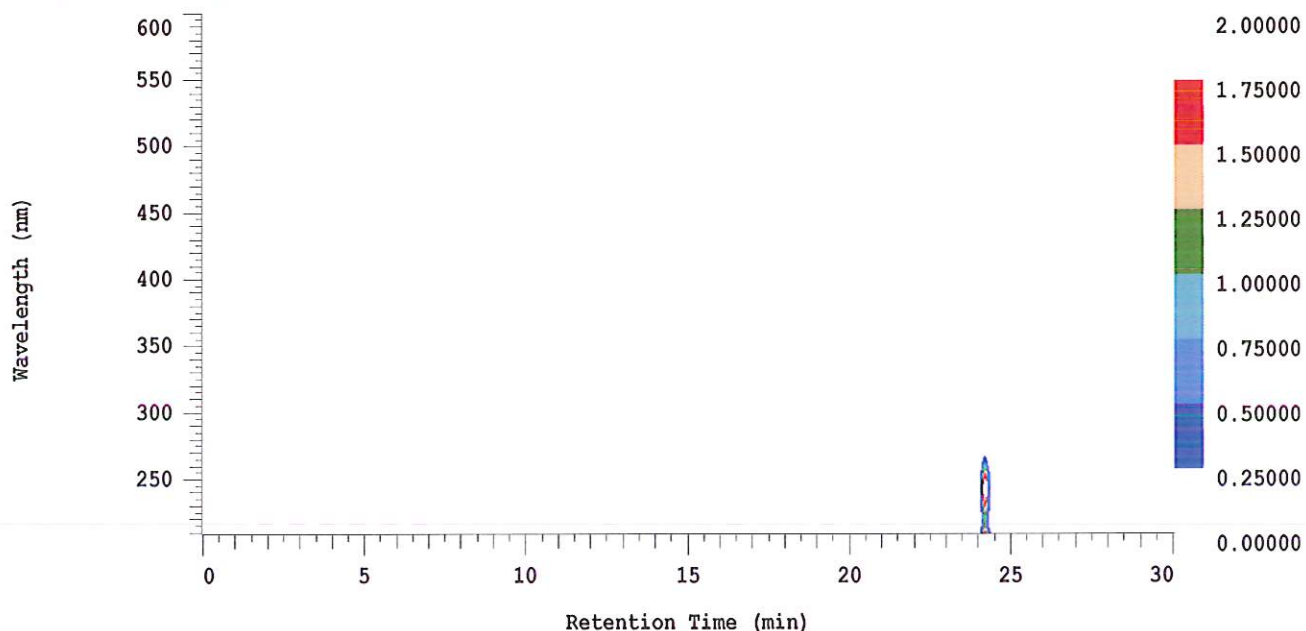
### D-2000 Elite HPLC System Manager Report

Analyzed: 07/19/2013 10:48 AM

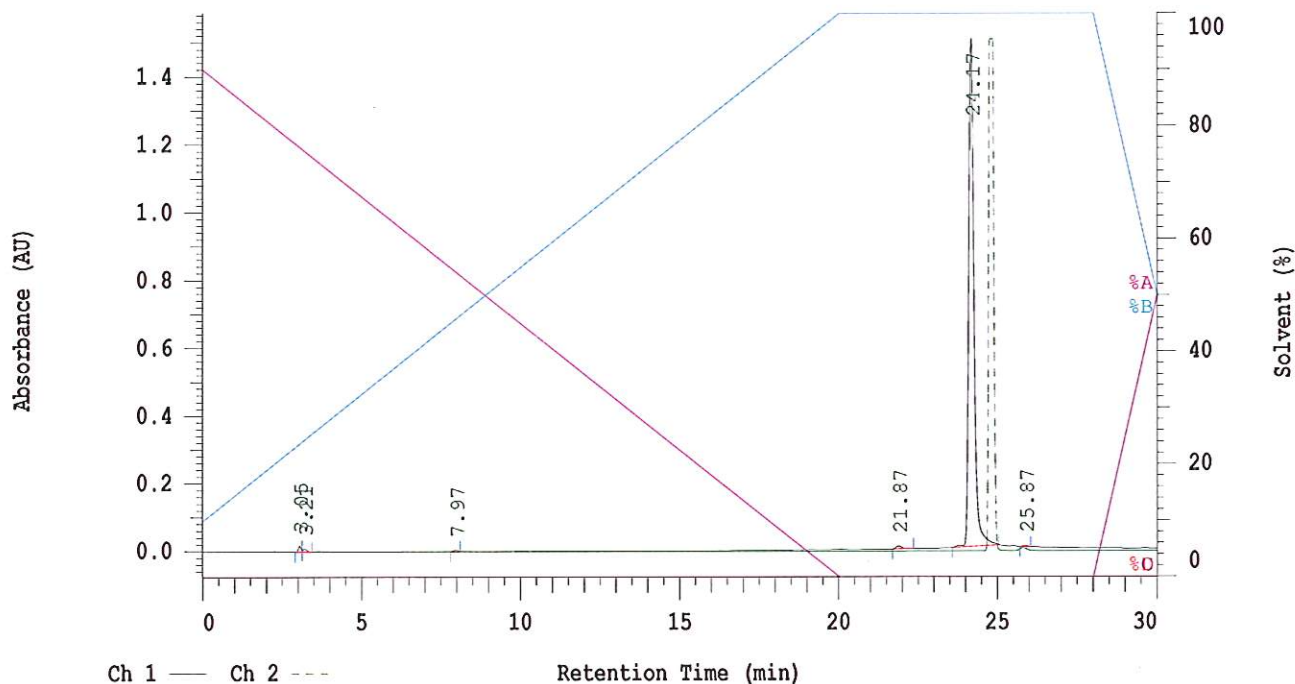
Reported: 07/19/2013 11:20 AM

Sample Name: HBOI-385

Sample Description: HBOI-385



Channel 1 Chrom Type: Fixed WL Chromatogram, 230 nm



Acquisition Method: autosampler\_30min\_wELSD

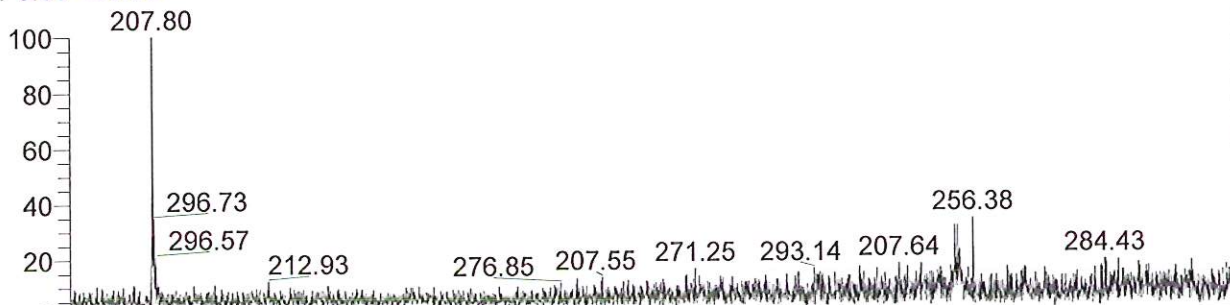
Column Type: Vydac C18

Pump A Solvent A: H2O/5% ACN

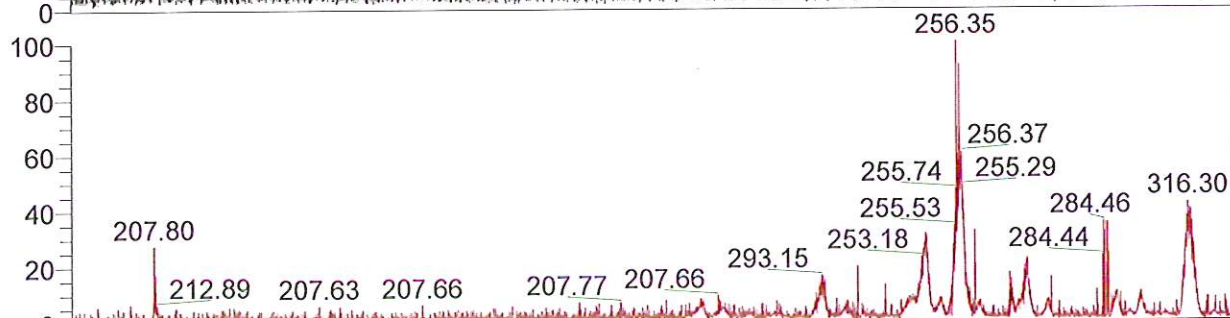
Pump A Solvent B: ACN

Method Description:

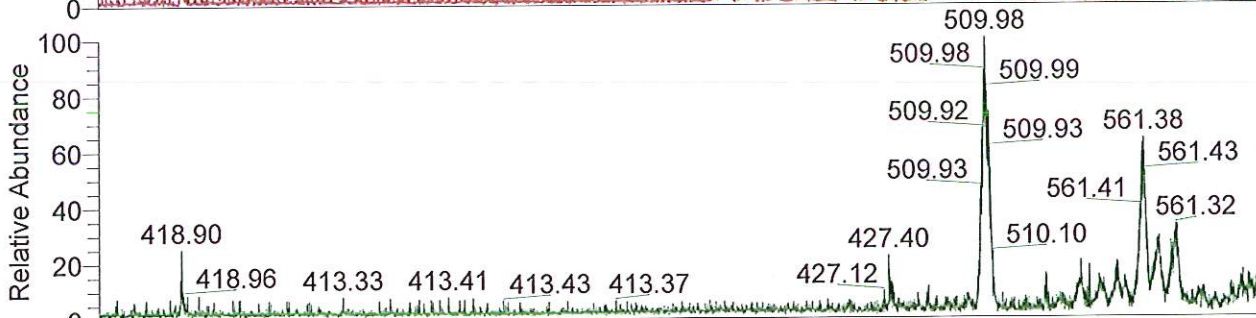
RT: 0.00 - 20.99



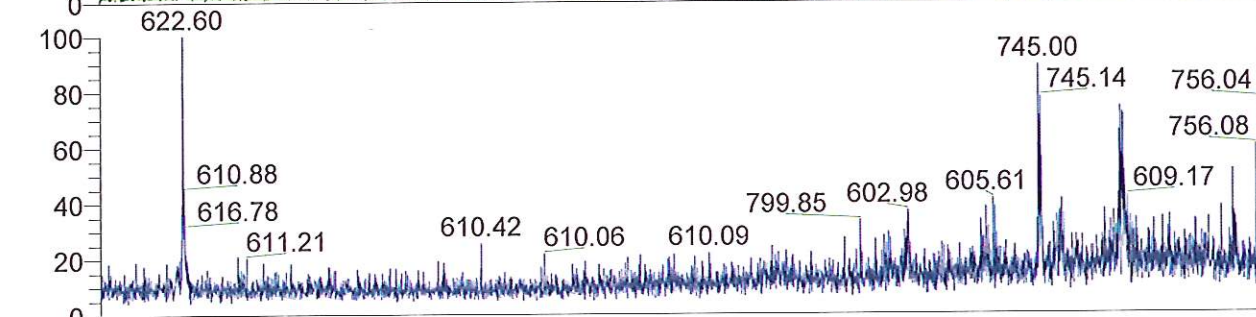
NL: 4.90E5  
TIC F: ITMS + c ESI  
Full ms [  
200.00-2000.00] MS  
BG\_HBOI\_385



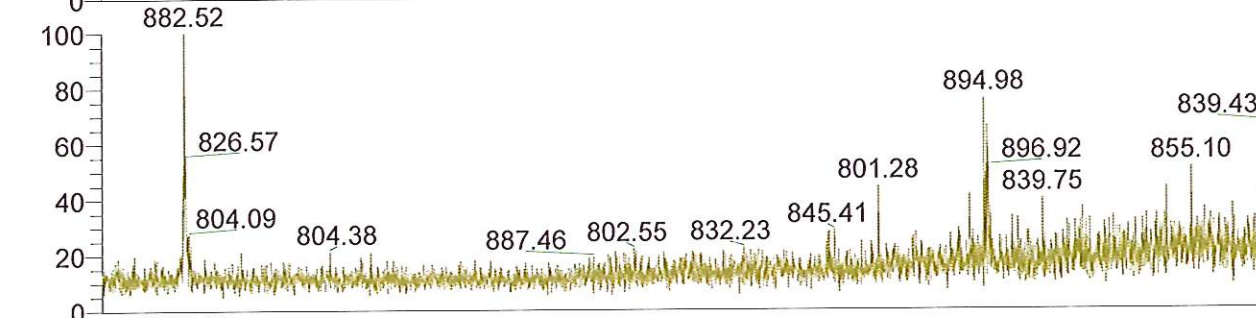
NL: 3.34E4  
Base Peak m/z=  
200.00-400.00 F: ITMS  
+ c ESI Full ms [  
200.00-2000.00] MS  
BG\_HBOI\_385



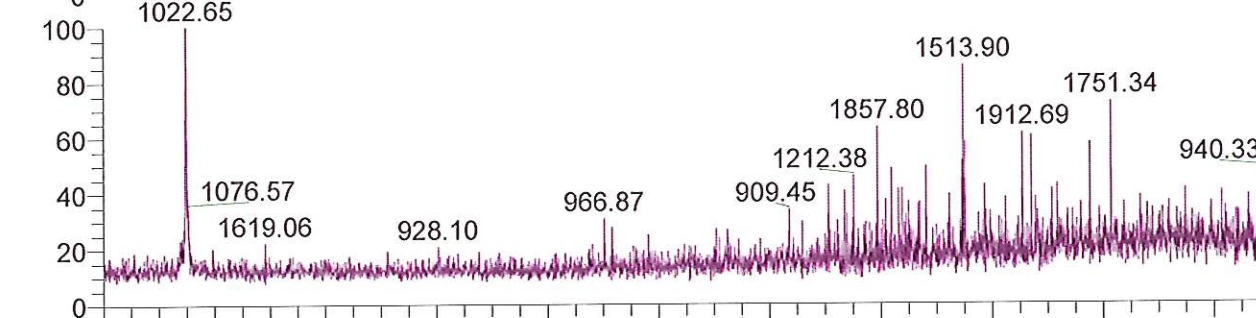
NL: 7.80E3  
Base Peak m/z=  
400.00-600.00 F: ITMS  
+ c ESI Full ms [  
200.00-2000.00] MS  
BG\_HBOI\_385



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)

