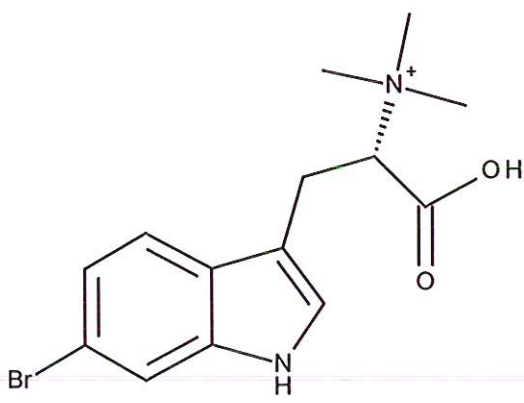


HBOI-355	
6-Br-HYPAPHORINE	
$C_{14}H_{18}BrN_2O_2$	326.21
Pachastrellidae	
Kondo, Nishi, Ishibashi and Kobayashi, 1994, <i>J. Nat. Prod.</i> , , 57: 1008-1011	

isolation scheme (Visio) attached

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 220 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 16.5 mg

For further information contact:

Amy E. Wright, PhD, HBOI@FAU, 5600 US 1, North, Fort Pierce, FL 34946
awrigh33@hboi.fau.edu, 772-242-2459

Date: 7/18/13

1-IX-93-1-005
 Pachastrellidae
 209g
 Extract 2 X EtOH, 2 X EtOAc/EtOH 9:1, 1 X MeOH
 ROBL23-50-1 5.7g Crude Extract

H₂O Partition

EtOAc Partition
 ROBL23-50-2
 140mg

H₂O Partition
 ROBL23-50-3
 4.97g

BuOH Partition
 ROBL23-50-4
 322.4mg

300g on 15.5g C18 Rf
 Gold Isco Column
 100% H₂O to 100%
 ACN, Washes

ROBL23-50- Weight (mg) Tubes	22 87.3 2-6	23 11.1 7-14	24/25 6.1 15-17	26 97.2 18-20 .	27 23.3 21-35	28 8.3 36-53
	29 6.3 54-56	30 26.2 57-65	31 6.7 66-69	32 9.5 ACN/H ₂ O/TFA 80:20:0.1		

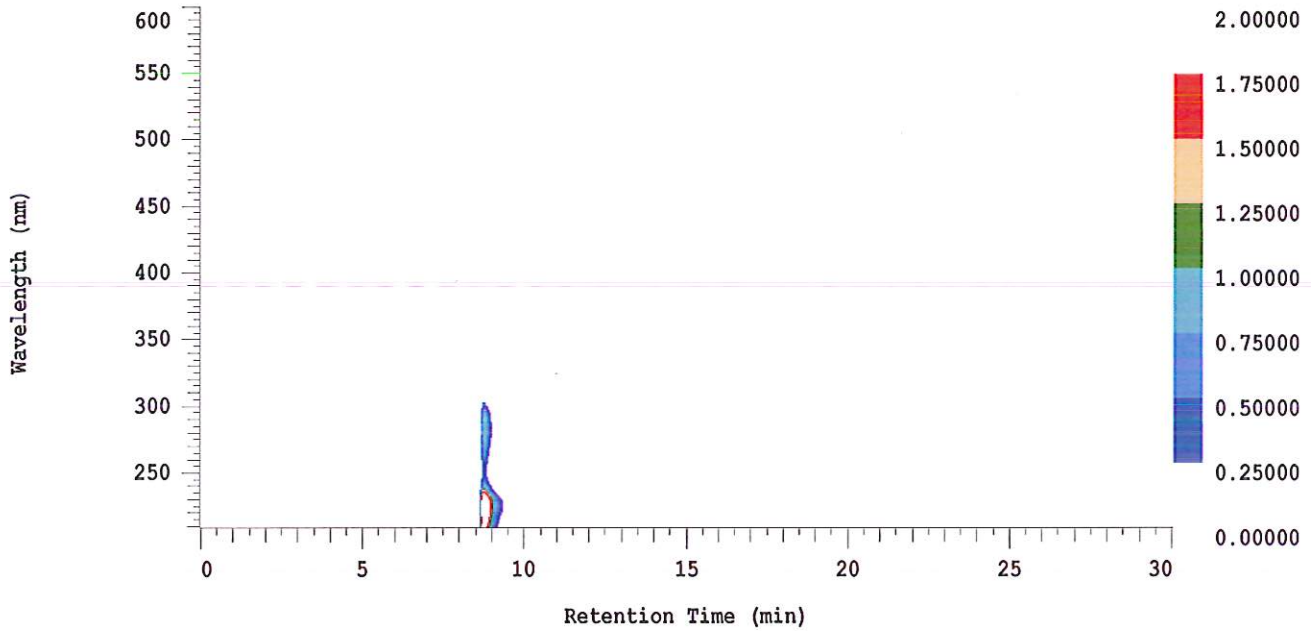
D-2000 Elite HPLC System Manager Report

Analyzed: 07/11/2013 11:25 AM

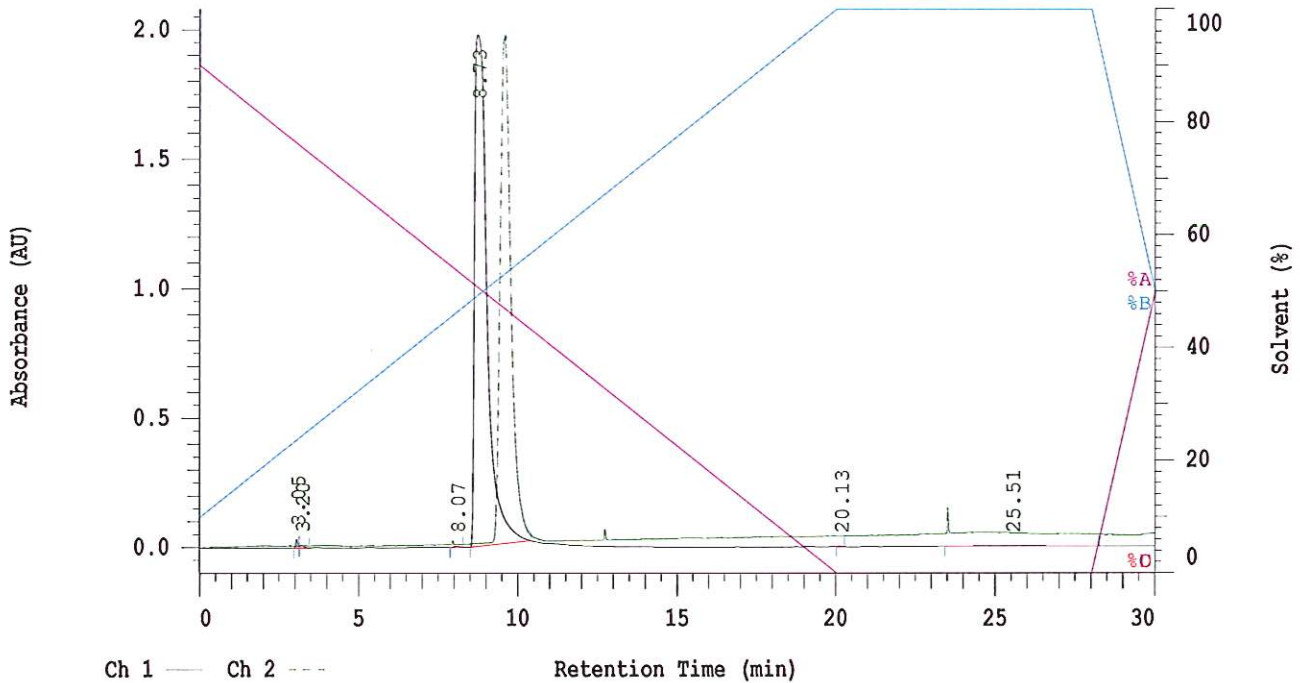
Reported: 07/11/2013 12:29 PM

Sample Name: HBOI-355_1

Sample Description: HBOI-355 hypaporphine 1mg-ml



Channel 1 Chrom Type: Fixed WL Chromatogram, 230 nm



Acquisition Method: autosampler_30min_wELSD

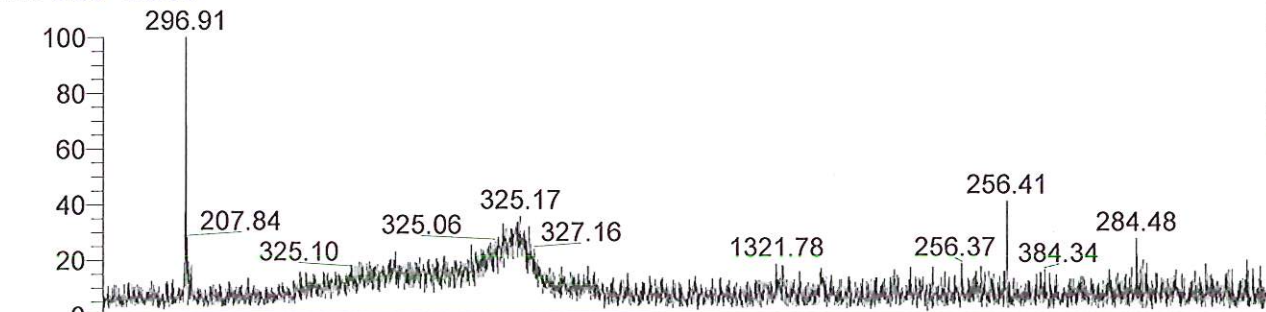
Column Type: Vydac C18

Pump A Solvent A: H2O/5% ACN

Pump A Solvent B: ACN

Method Description:

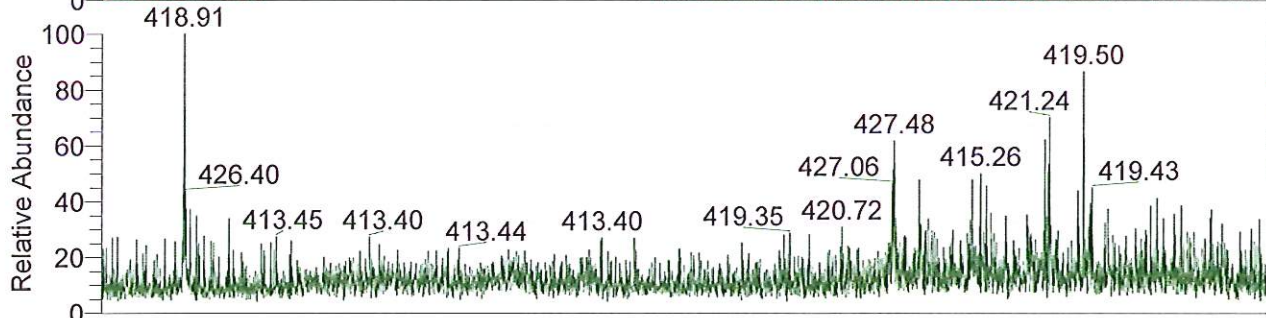
RT: 0.00 - 20.99



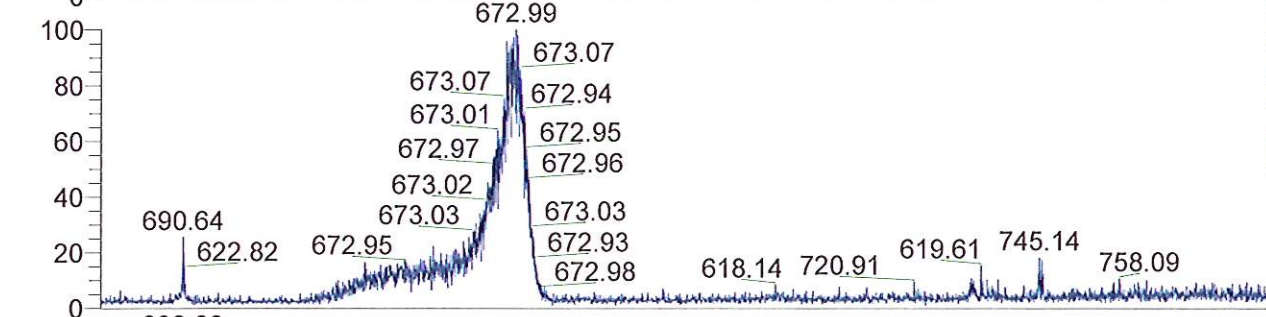
NL: 4.22E5
TIC F: ITMS + c ESI
Full ms [
200.00-2000.00] MS
BG_HBOI_355



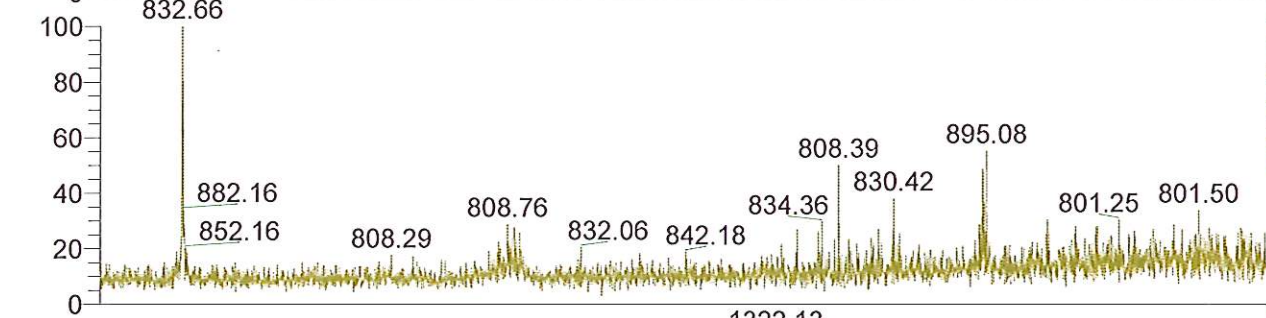
NL: 2.40E4
Base Peak m/z=
200.00-400.00 F: ITMS
+ c ESI Full ms [
200.00-2000.00] MS
BG_HBOI_355



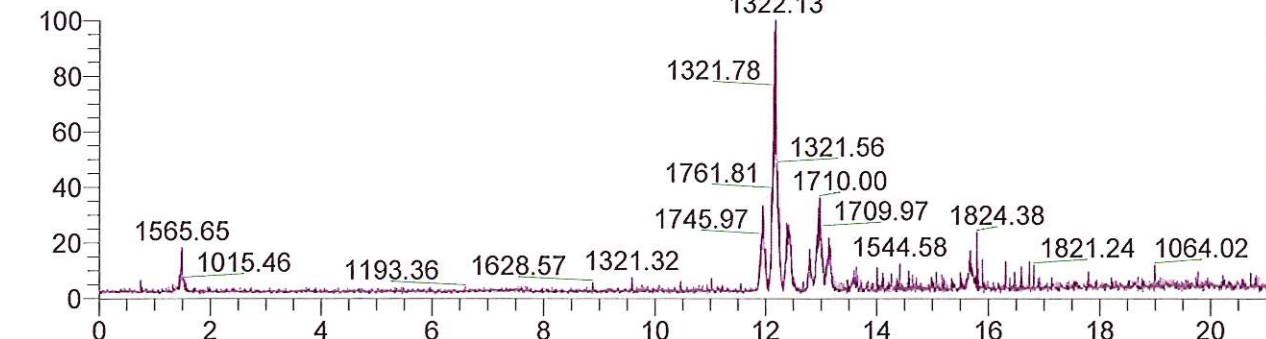
NL: 1.96E3
Base Peak m/z=
400.00-600.00 F: ITMS
+ c ESI Full ms [
200.00-2000.00] MS
BG_HBOI_355



NL: 3.65E3
Base Peak m/z=
600.00-800.00 F: ITMS
+ c ESI Full ms [
200.00-2000.00] MS
BG_HBOI_355



NL: 7.55E2
Base Peak m/z=
800.00-900.00 F: ITMS
+ c ESI Full ms [
200.00-2000.00] MS
BG_HBOI_355



NL: 2.69E3
Base Peak m/z=
900.00-2000.00 F:
ITMS + c ESI Full ms [
200.00-2000.00] MS
BG_HBOI_355

Time (min)

