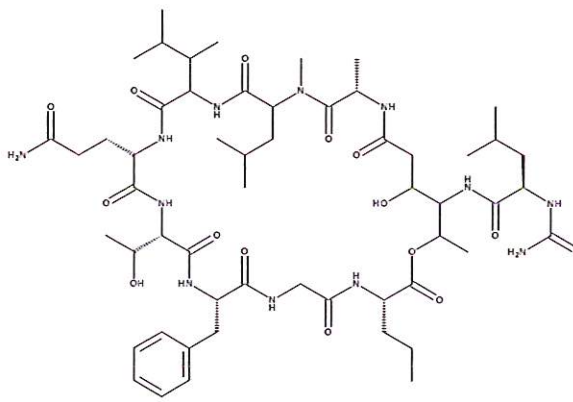


HBOI-343	AND Enantiomer
	
CYCLOLITHISTIDE A	
$C_{54}H_{86}ClN_{11}O_{15}$	1164.78
<i>Discodermia transcripta</i>	

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 17.7 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: 7/15/13

8-IV-05-3-2
Theonellidae
494 g
EtOAc:EtOH (9:1)
& EtOH extraction
17.08 g

EtOAc Partition
WIN14-71-1
5.24 g

H2O Partition

H2O	80	60	40	20	0	IPA
ACN	20	40	60	80	100	MeOH
wash	0	0	0	0	0	CH2Cl2
WIN14-72	1	2	3	4	5	6
wt (mg)	1199.9	279.9	1098.4	47.8	71.7	510.0

RP-18 Combiflash
1/2 of WIN14-72-4

tubes	1-4	5-6	7-8	9-10	11	12-20	21-40	41-wash
WIN14-77	1	2	3	4	5	6	7	8
wt (mg)	0.3	27.8	141.7	82.1	11.0	4.5	4.3	4.9

RP-18
Combiflash

tubes	1-2	3	4-10	11-17	18-20	21-22	23	24	25	26	27-30	31-40	41-50	51-wash
WIN14-80	1	2	3	4	5	6	7	8	9	10	11	12	13	14
wt (mg)	0.5	1.5	2.1	3.2	9.7	46.1	55.3	57.5	30.7	6.0	1.4	1.0	1.1	0.9

prep LC
gradient
70:30
H2O:ACN

ROBL26-71- wt(mg)	1	2	3	4	5	6
	0.3	0.8	7.9	11.9	0.6	0.6

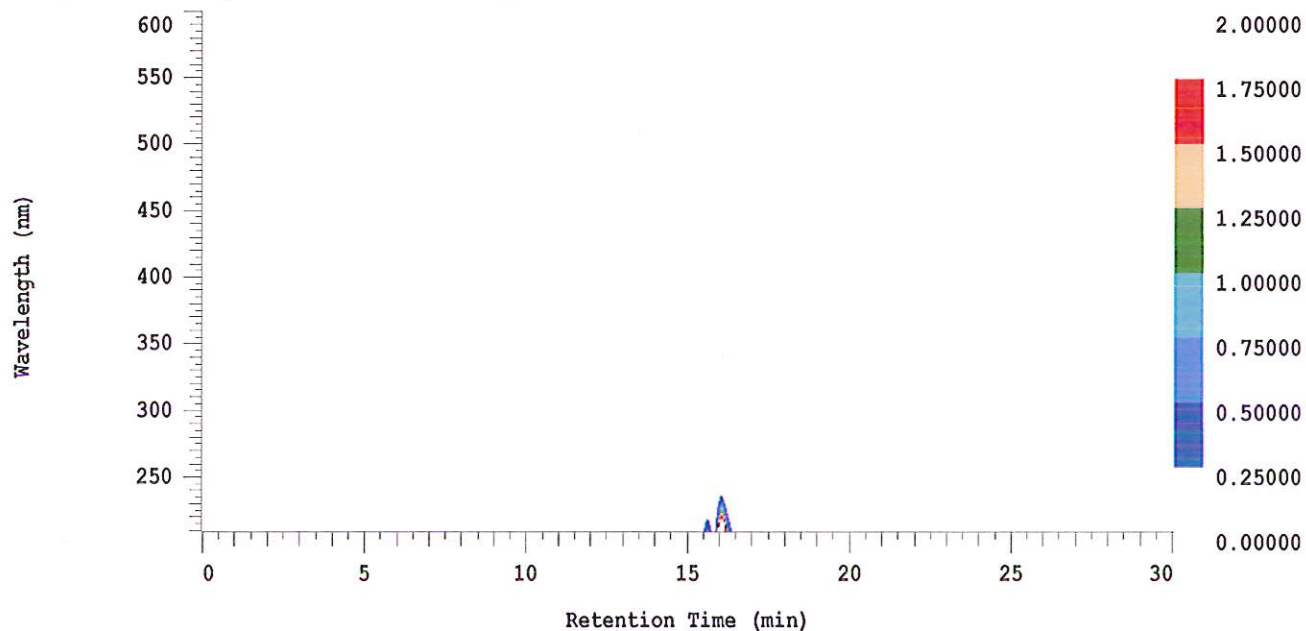
D-2000 Elite HPLC System Manager Report

Analyzed: 07/03/2013 12:47 PM

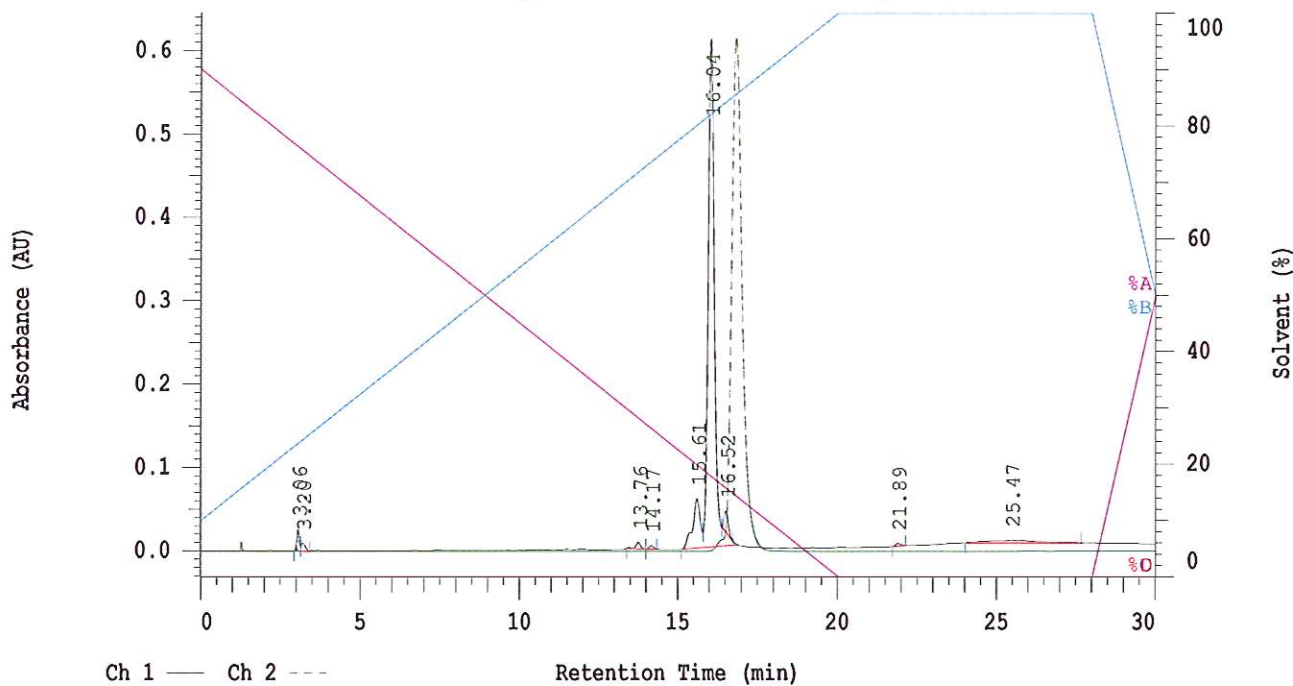
Reported: 07/03/2013 03:25 PM

Sample Name: HBOI-343

Sample Description: HBOI-343 Cyclololithistide



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

