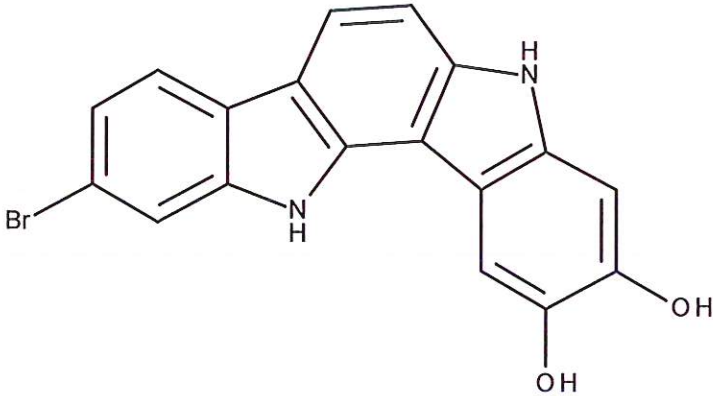


HBOI-361	
BIS-INDOLE ALKALOID	
$C_{18}H_{11}BrN_2O_2$	367.00
<i>Asteropus</i> sp.	
manuscript in preparation	

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, 0.1% TFA), B: CH₃CN (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: H₂O (0.1%formic acid), B: ACN (0.1%formic acid)
 t= 0 min A:B (90:10, v/v), t=15 min (100%B), t=21 min (100%B), t=22.1 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

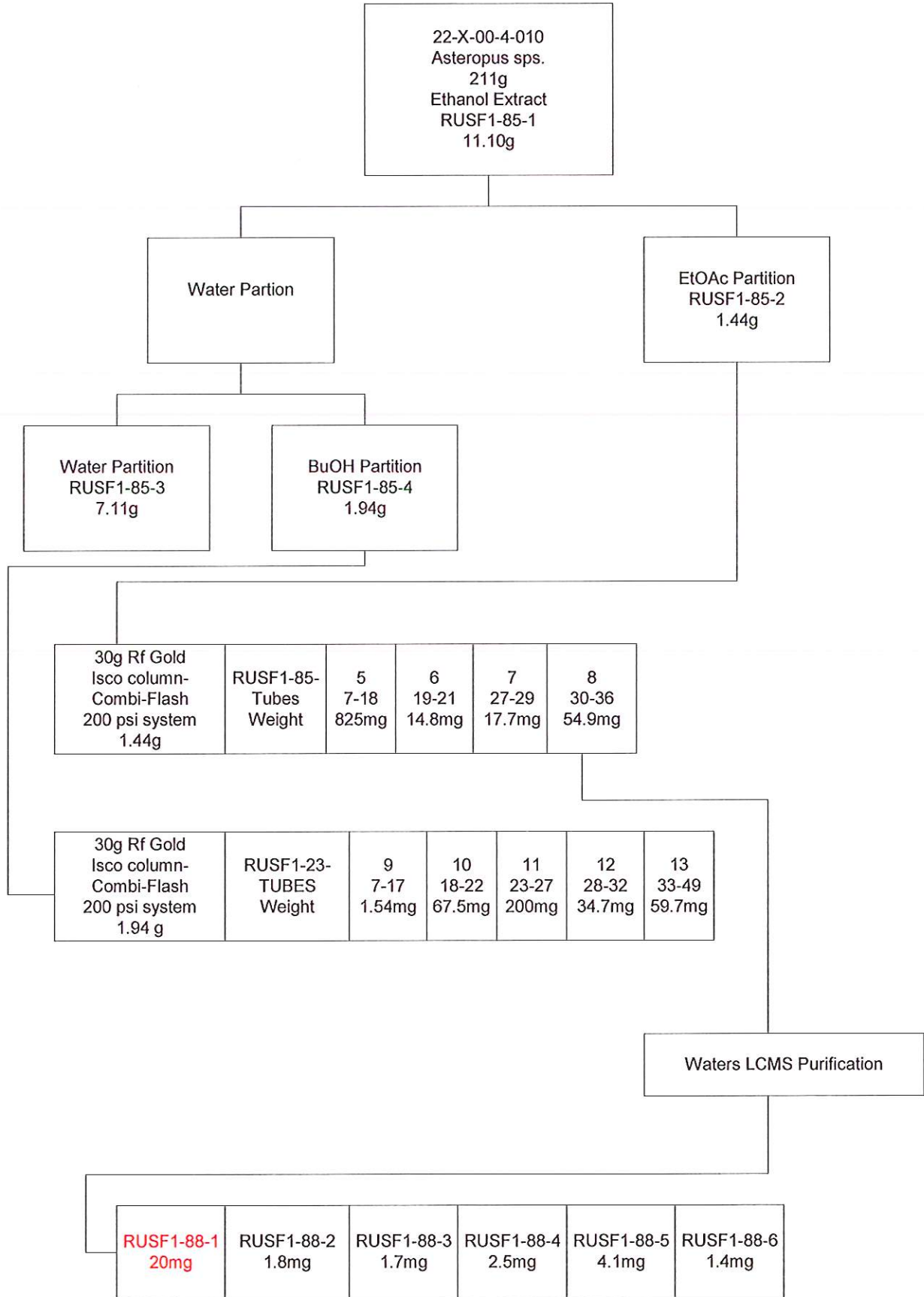
estimated purity >90%

sample weight 20.4 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: 1/28/13



22-X-00-4-010
Asteropus spp.
21g
Ethanol Extract
RUSF1-85-1
11.10g

Water Partion

EtOAc Partion
RUSF1-85-2
1.44g

Water Partion
RUSF1-85-3
7.11g

BuOH Partion
RUSF1-85-4
1.94g

30g Rf Gold Isco column- Combi-Flash 200 psi system 1.44g	RUSF1-85- Tubes Weight	5	6	7	8
		7-18	19-21	27-29	30-36
		825mg	14.8mg	17.7mg	54.9mg

30g Rf Gold Isco column- Combi-Flash 200 psi system 1.94 g	RUSF1-23- TUBES Weight	9	10	11	12	13
		7-17	18-22	23-27	28-32	33-49
		1.54mg	67.5mg	200mg	34.7mg	59.7mg

Waters LCMS Purification

RUSF1-88-1 20mg	RUSF1-88-2 1.8mg	RUSF1-88-3 1.7mg	RUSF1-88-4 2.5mg	RUSF1-88-5 4.1mg	RUSF1-88-6 1.4mg
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For Combi-Flash run:

30g Rf Gold Isco column

Flow rate 30mL/min

A:H₂O/ACN(95:5, 0.1% TFA), B:ACN (0.1%TFA)

t=0-2.3 A:B (80:20), t= 5.8 A:B (65:45), t=30 (5:95)

For Waters LCMS Purification

Grace C-18, 150×22mm

Flow rate 15mL/min

A:H₂O/ACN(95:5, 0.1% TFA), B:ACN (0.1%TFA)

t=0 A:B (95:5), t=20-23 A:B (0:100), t=23.01-25 A:B (5:95)

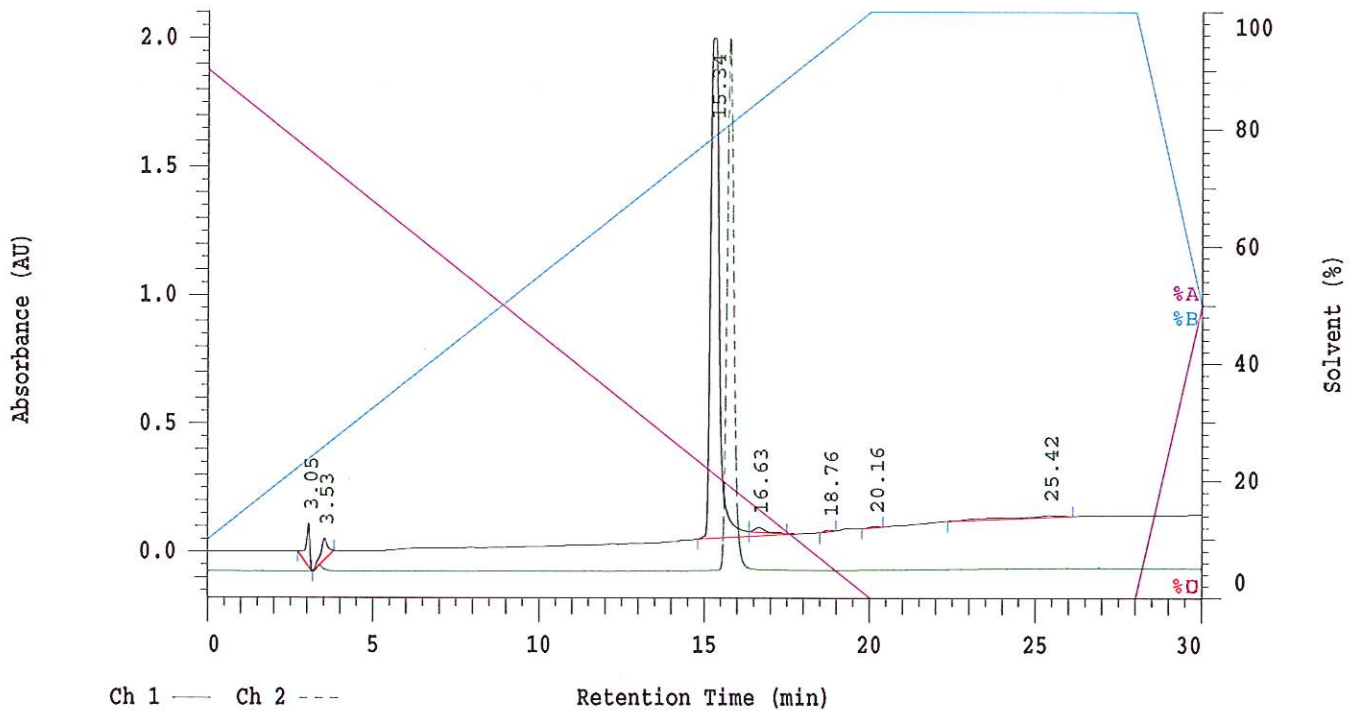
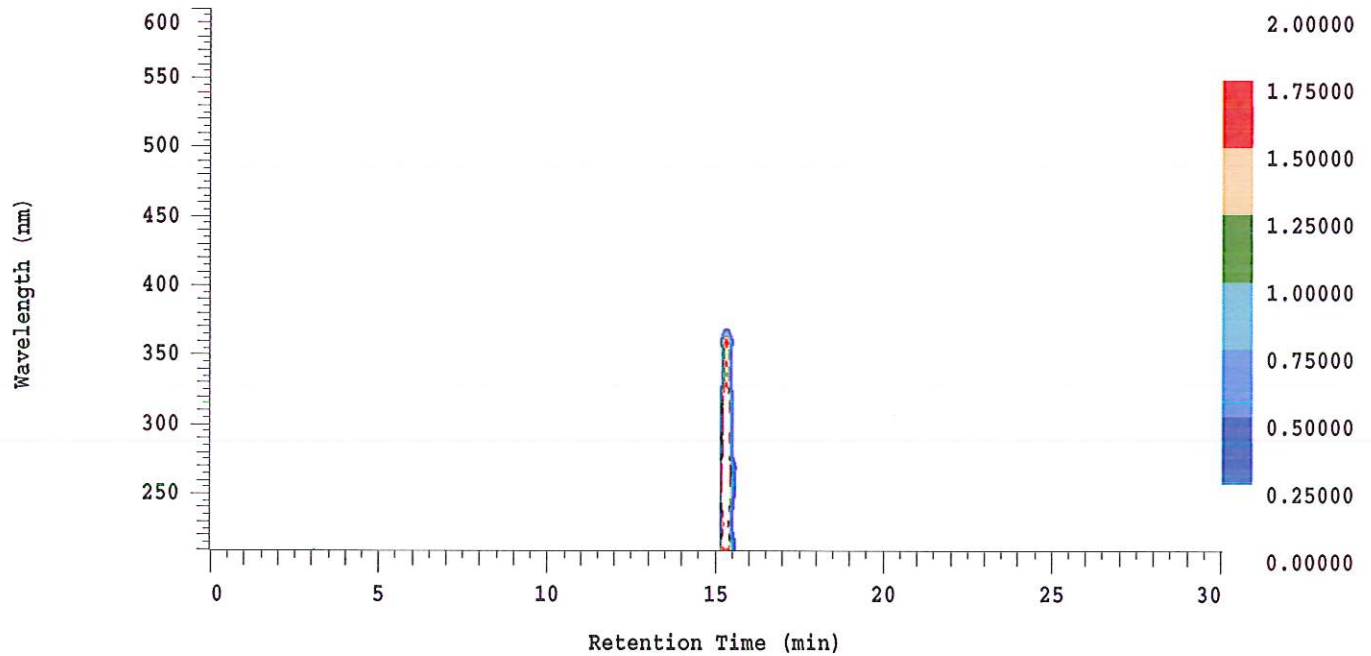
D-2000 Elite HPLC System Manager Report

Analyzed: 01/24/2013 11:45 PM

Reported: 01/25/2013 08:03 AM

Sample Name: HBOI-361

Sample Description: HBOI-361



Acquisition Method: NIH_30min_UV220_wELSD_TFA

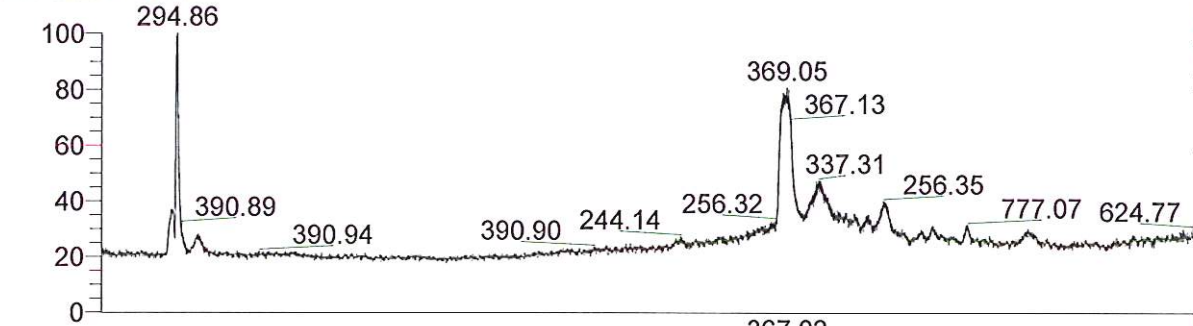
Column Type: Vydac C18

Pump A Solvent A: H2O/5% ACN TFA 0.1%

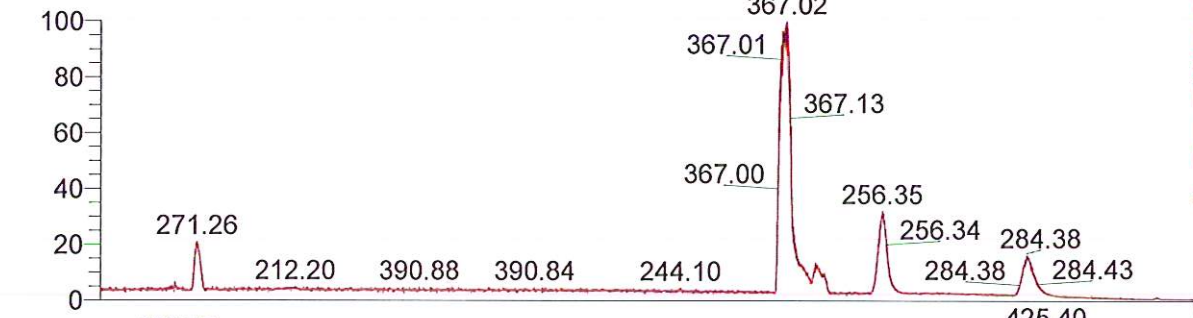
Pump A Solvent B: ACN TFA 0.1%

Method Description:

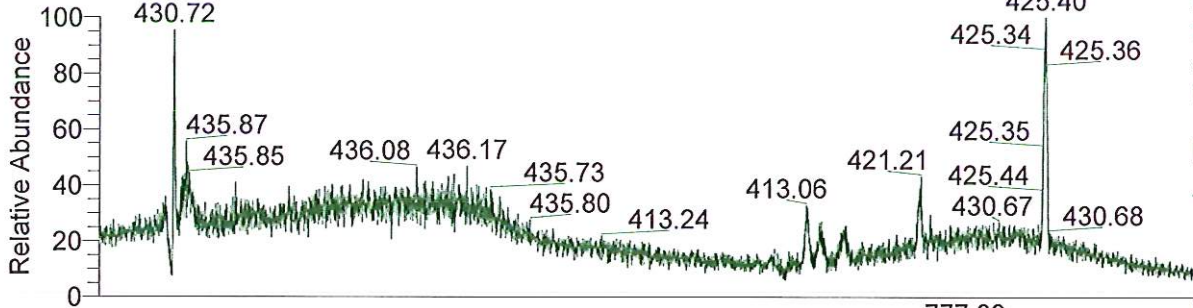
RT: 0.00 - 24.99



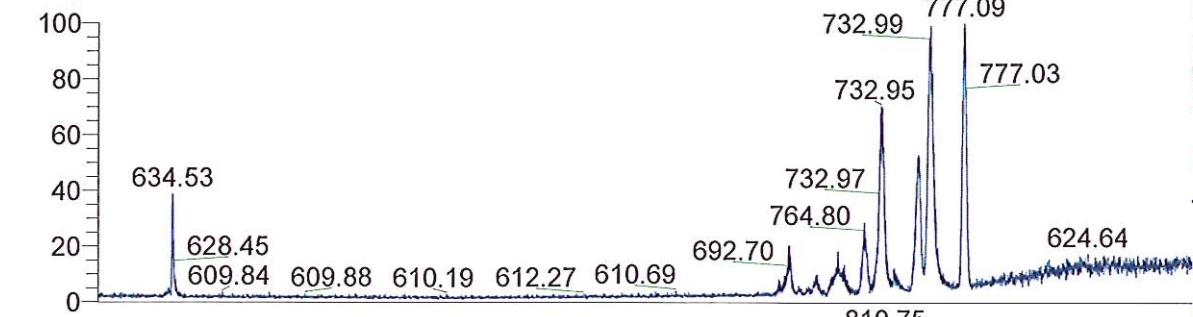
NL: 1.06E6
 TIC F: ITMS + c ESI Full ms [200.00-2000.00] MS
 BG_HB_361_New_Bis_indole_alkaloid



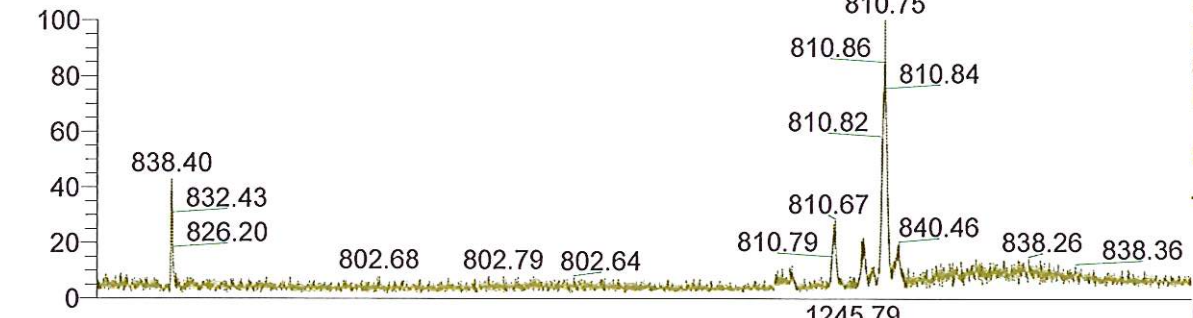
NL: 2.43E5
 Base Peak m/z=
 200.00-400.00 F: ITMS + c
 ESI Full ms [200.00-2000.00] MS
 BG_HB_361_New_Bis_indole_alkaloid



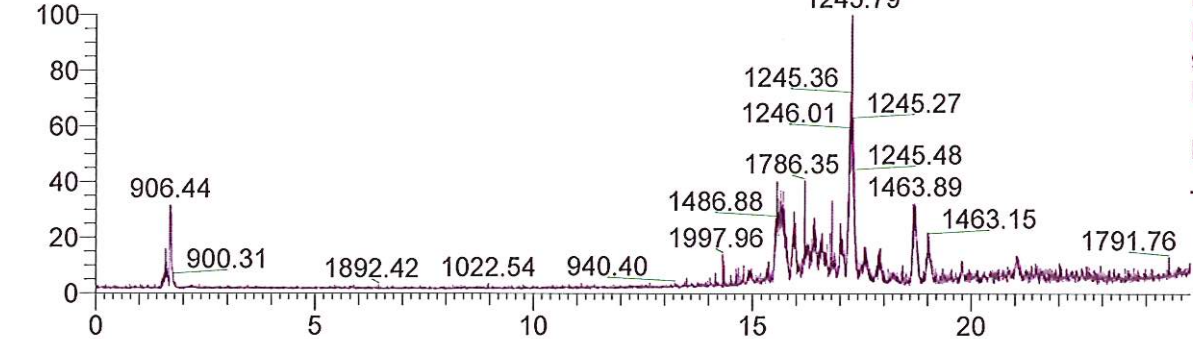
NL: 1.06E4
 Base Peak m/z=
 400.00-600.00 F: ITMS + c
 ESI Full ms [200.00-2000.00] MS
 BG_HB_361_New_Bis_indole_alkaloid



NL: 1.60E4
 Base Peak m/z=
 600.00-800.00 F: ITMS + c
 ESI Full ms [200.00-2000.00] MS
 BG_HB_361_New_Bis_indole_alkaloid



NL: 7.49E3
 Base Peak m/z=
 800.00-900.00 F: ITMS + c
 ESI Full ms [200.00-2000.00] MS
 BG_HB_361_New_Bis_indole_alkaloid

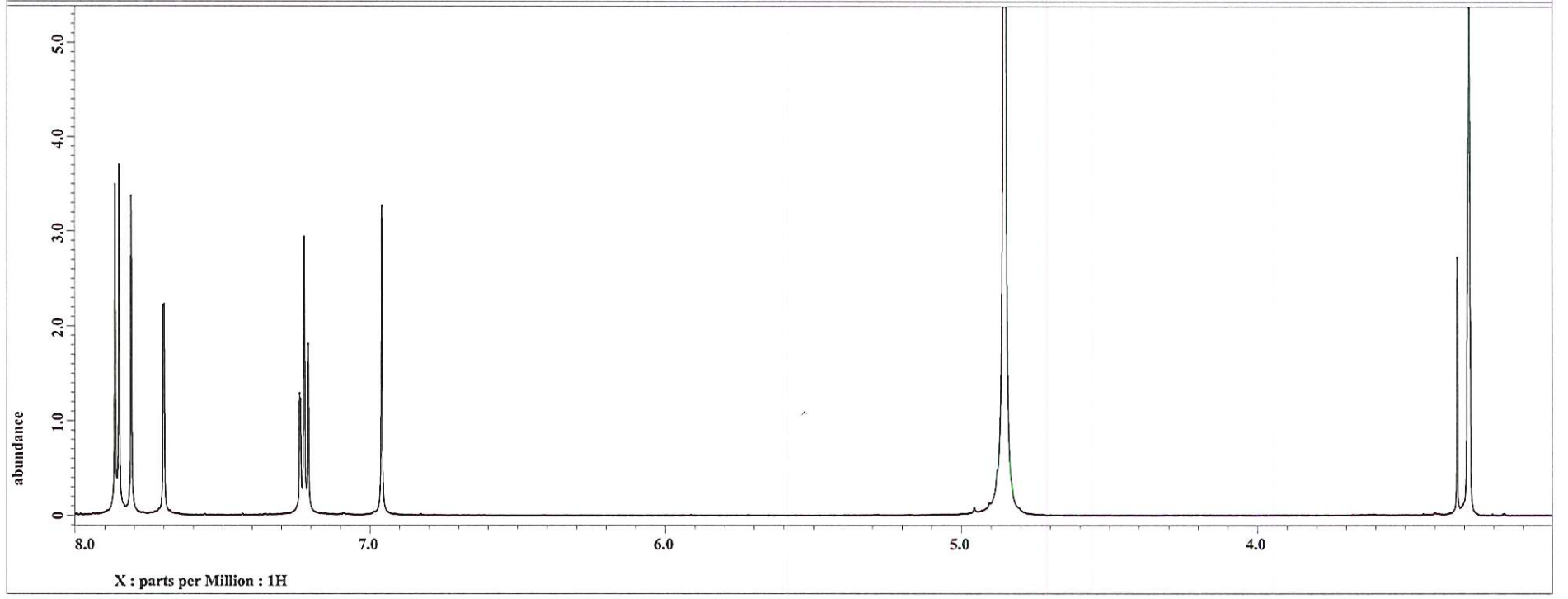
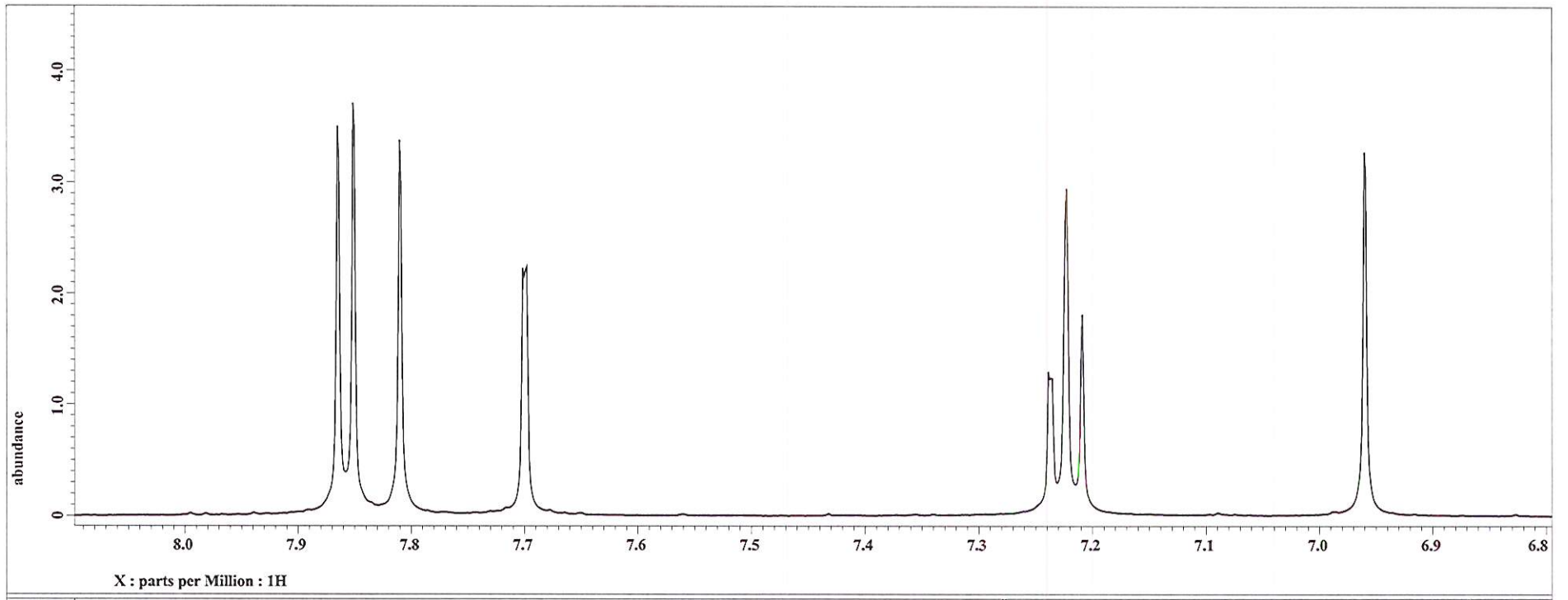


NL: 7.99E3
 Base Peak m/z=
 900.00-2000.00 F: ITMS + c
 ESI Full ms [200.00-2000.00] MS
 BG_HB_361_New_Bis_indole_alkaloid

Time (min)

RUSF1-88-8

H3361



HB361

