

HBOI-364	
BROMOCHLOROOXAZOLE DERIVATIVE	
$C_{13}H_{14}BrClN_5O^+$	371.64
Axinellida	
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: 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=22.1 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

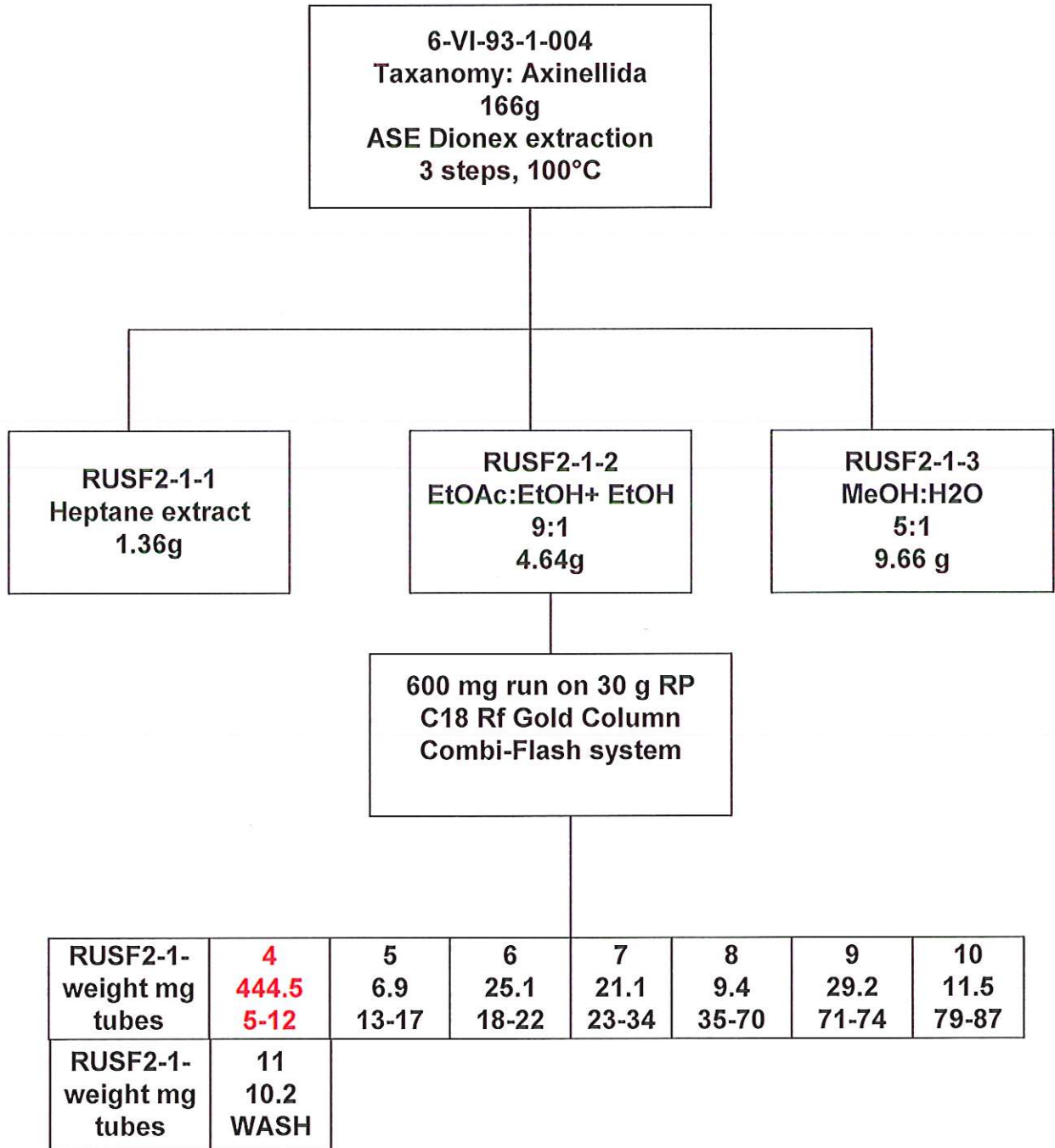
estimated purity >90%

sample weight 19.2 mg

For further information contact:

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Date: 1/28/13



CombiFlash

A: H₂O:CH₃CN (95:5)-0.1 %TFA, B:CH₃CN - 0.1% TFA, 30 ml/min

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

RUSF2-1-4
70 mg
LCMS Purification using a C-18
Vydac prepcolumn with flow rate 15 mL/min
(A)H₂O:CH₃CN(95:5) -0.1%TFA:(B)CH₃CN - 0.1%TFA
t=0 min A:B (90:10), t=20 min A:B (70:30) t=21min (100% B), t=25 min (100%B)

RUSF2-3- weight mg peak	1 17.9 1	2 3.1 2	3 15.5 3	4 6.5 4	5 6.4 5	6 8.7 6	7 12.7 7
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Purification of RUSF2-1-4 was repeated several times and similar fractions combined. RUSF2-11-7 was the combination product of peak 7 in each run.

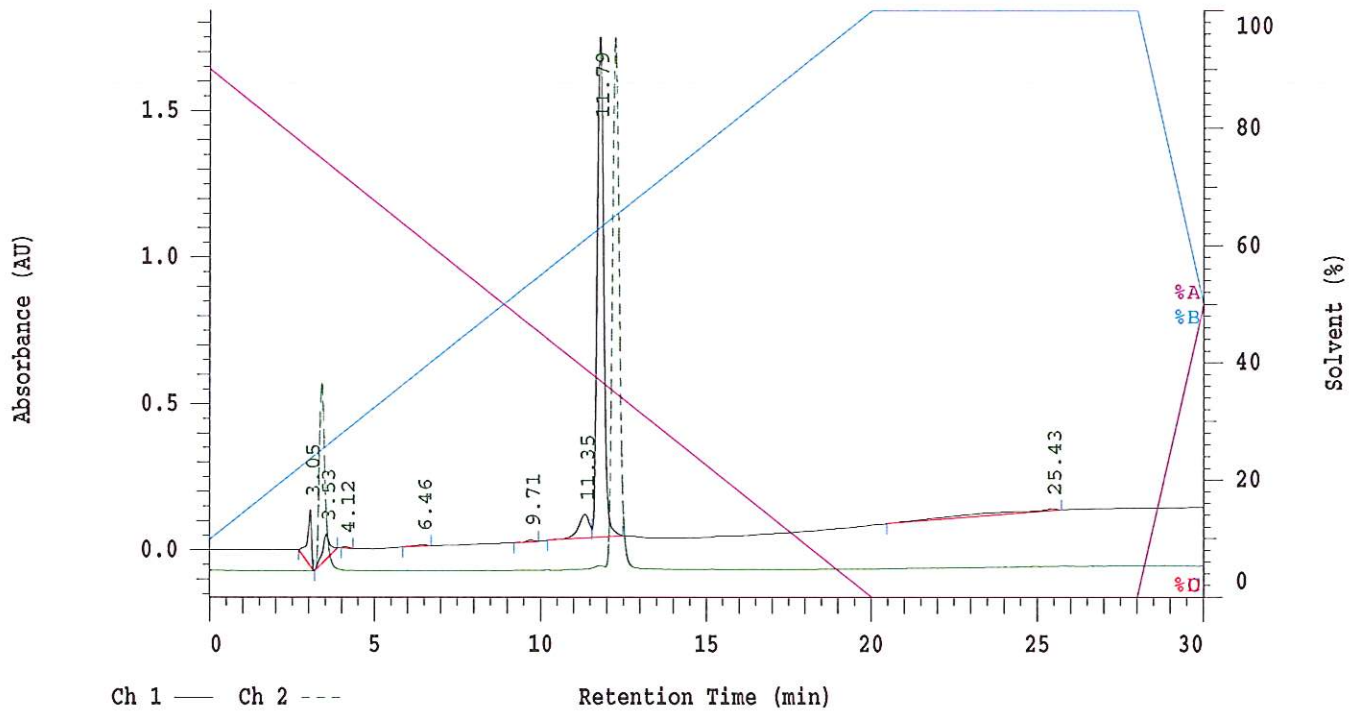
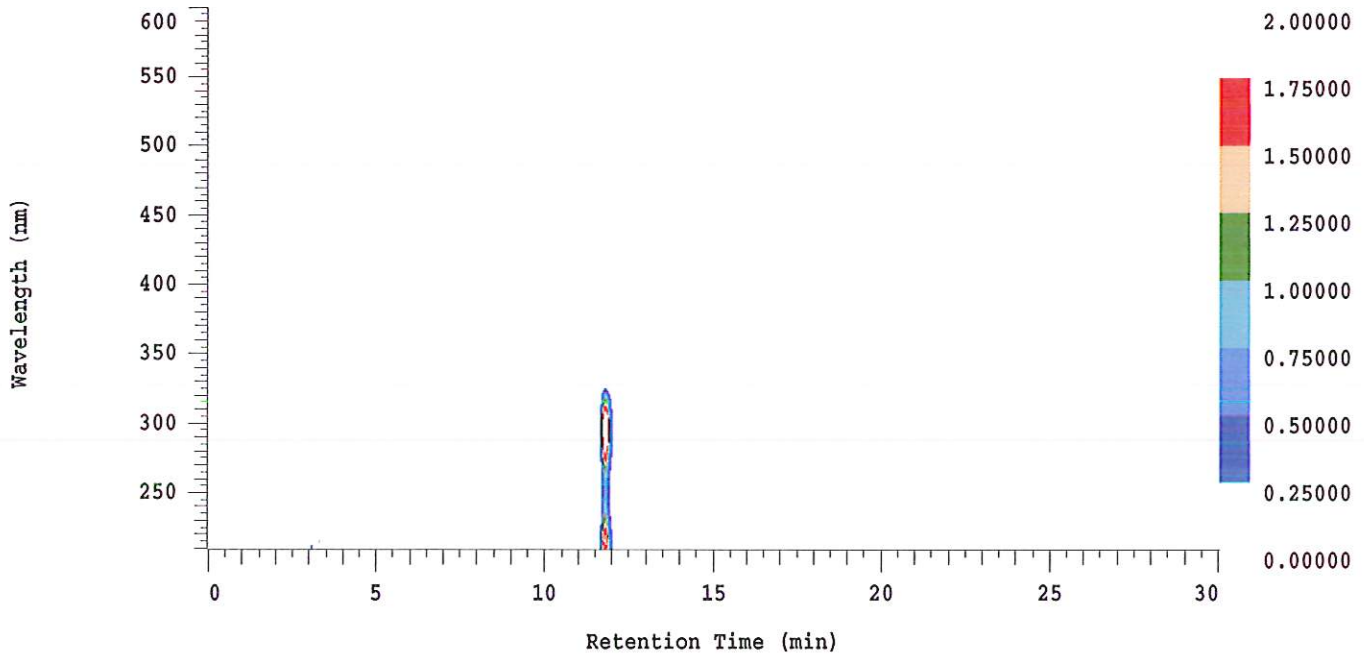
D-2000 Elite HPLC System Manager Report

Analyzed: 01/25/2013 12:23 AM

Reported: 01/25/2013 08:03 AM

Sample Name: HBOI-364

Sample Description: HBOI-364



Acquisition Method: NIH_30min_UV220_wELSD_TFA

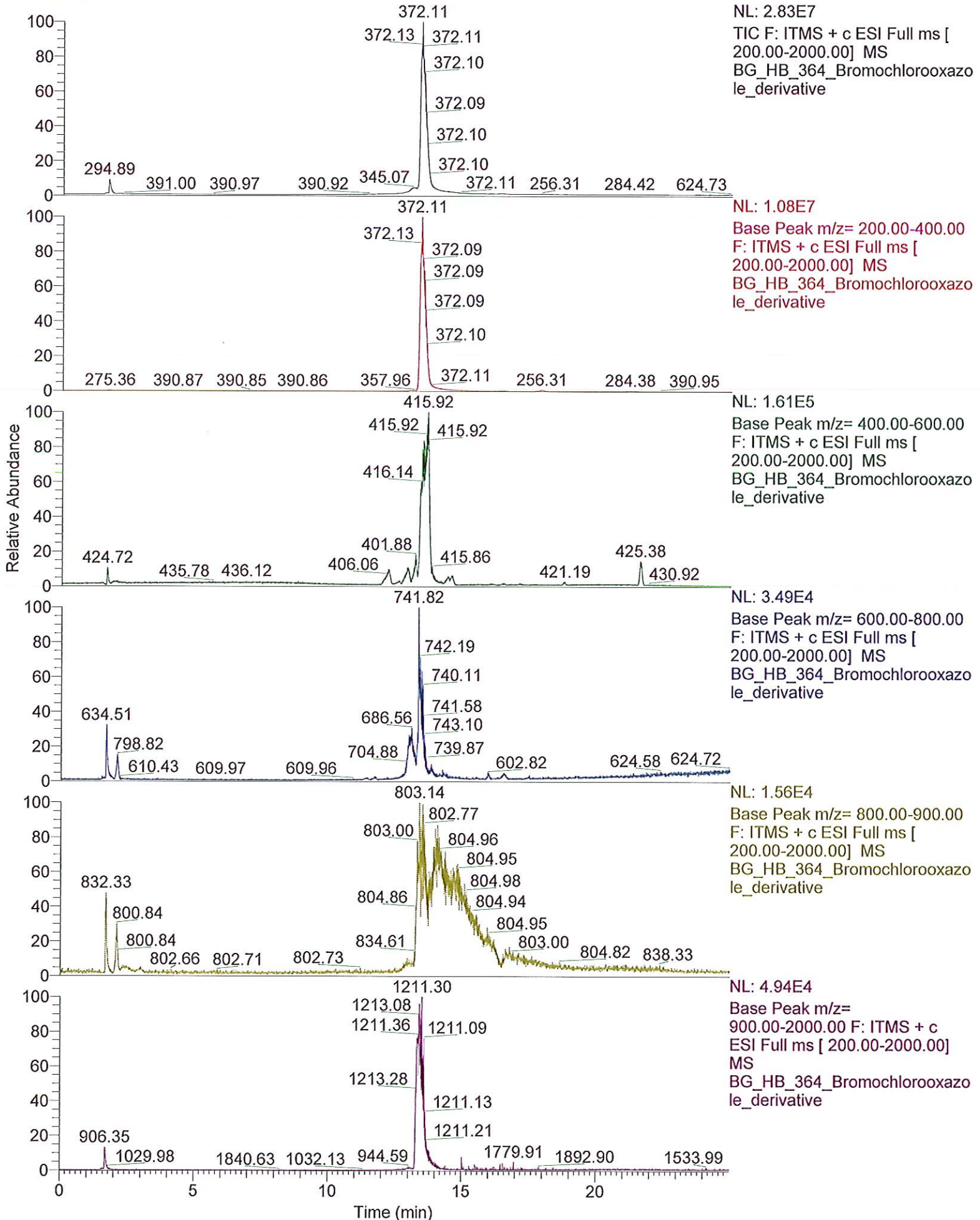
Column Type: Vydac C18

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



RUSF2-11-7

