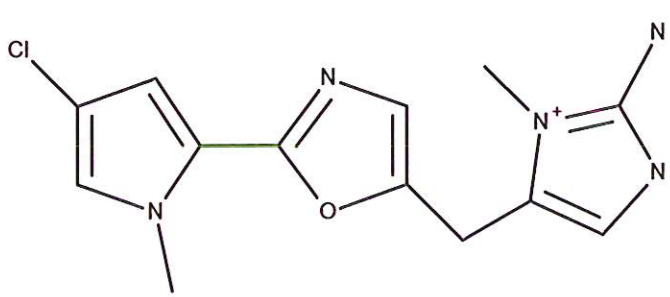


HBOI-366			
CHLORO-OXAZOLE			
$C_{13}H_{15}ClN_5O^+$		292.10	
Axinellida			
Manuscript in prep			

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 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 16.3 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

6-VI-93-1-004
 Taxonomy: Axinellida
 166g
 ASE Dionex extraction
 3 steps, 100°C

RUSF2-1-1
 Heptane extract
 1.36g

RUSF2-1-2
 EtOAc:EtOH+ EtOH
 9:1
 4.64g

RUSF2-1-3
 MeOH:H2O
 5:1
 9.66 g

600 mg run on 30 g RP C18 Rf Gold
 Column
 Combi-Flash system

RUSF2-1- weight mg tubes	4 444.5 5-12	5 6.9 13-17	6 25.1 18-22	7 21.1 23-34	8 9.4 35-70	9 29.2 71-74	10 11.5 79-87	11 10.2 WASH
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RUSF2-1-4
 70 mg
 LCMS Purification using a C-18
 Vydac prepcolumn with flow rate 15 mL/min
 (A)H2O/ACN(95/5)0.1%TFA:(B)ACN(0.1%TFA)
 t=0, A:B (90:10, t=20, A:B (70:30), t=21(100% B),0,t=25(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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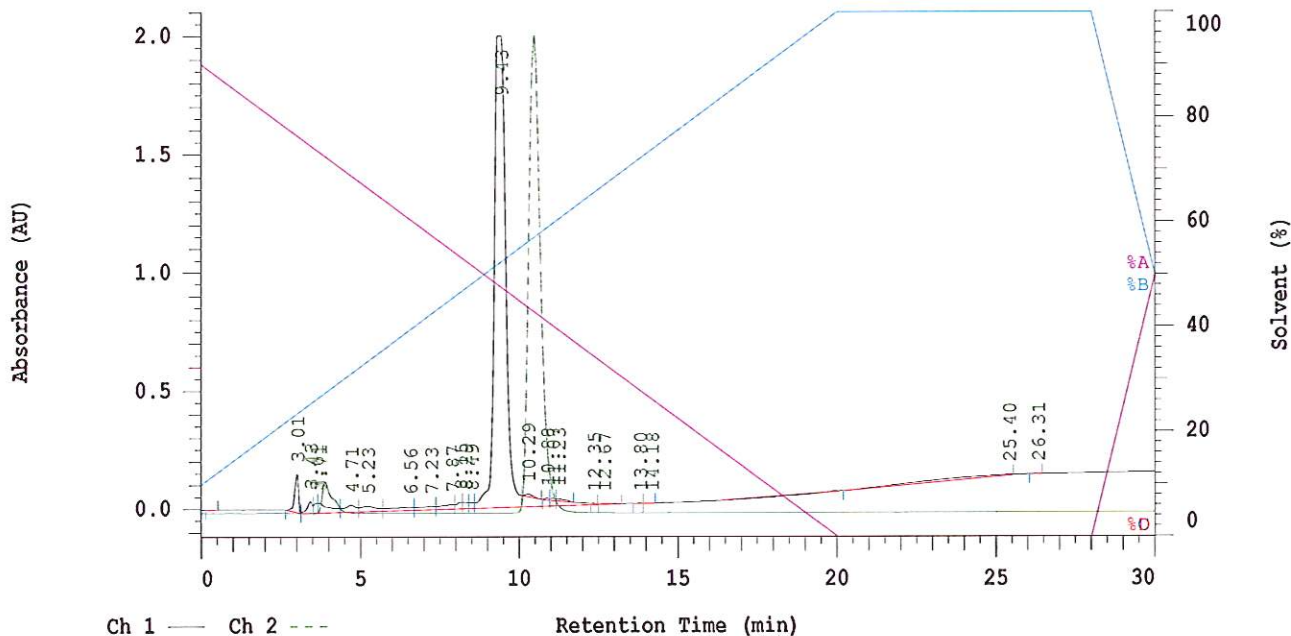
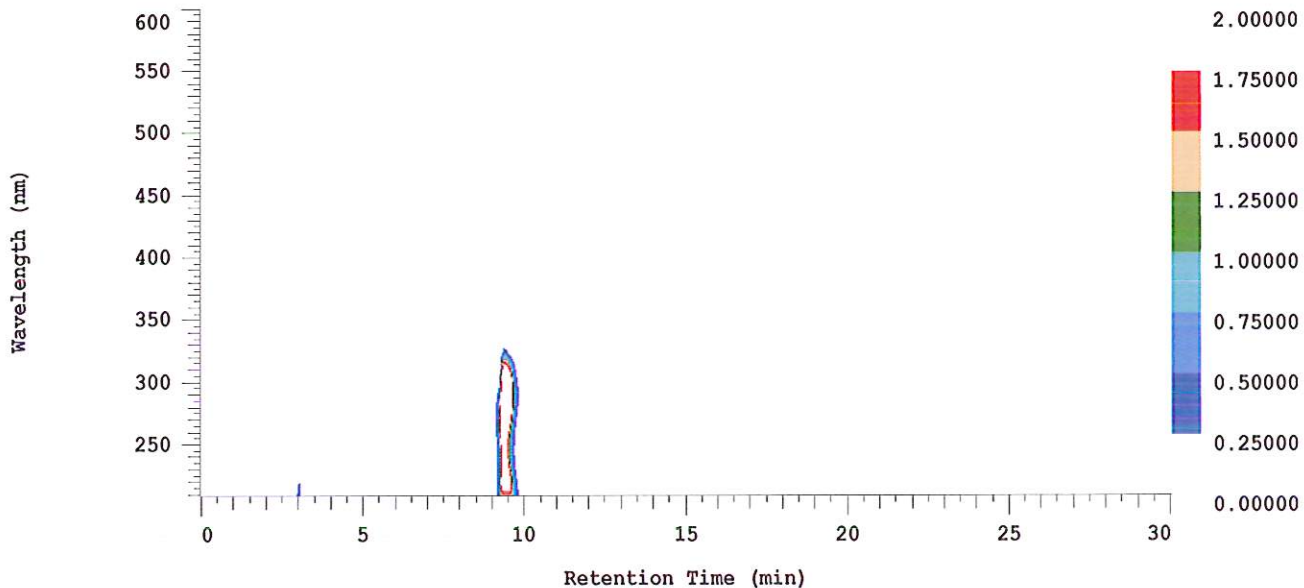
D-2000 Elite HPLC System Manager Report

Analyzed: 07/08/2013 11:57 AM

Reported: 07/09/2013 08:53 AM

Sample Name: HBOI.366

Sample Description: HBOI.366



Acquisition Method: autosampler_30min_wELSD

Column Type: Vydac C18

Pump A Solvent A: H2O/5% ACN

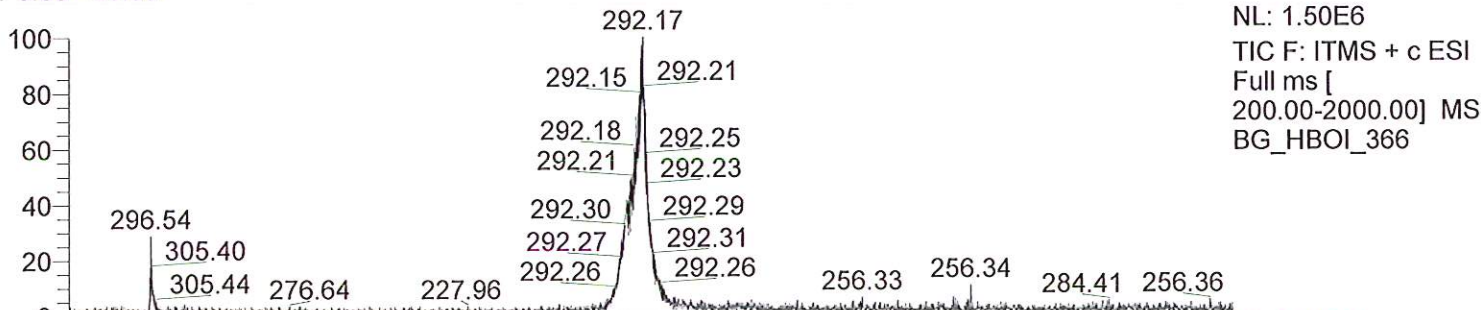
Pump A Solvent B: ACN

Pump A Solvent C:

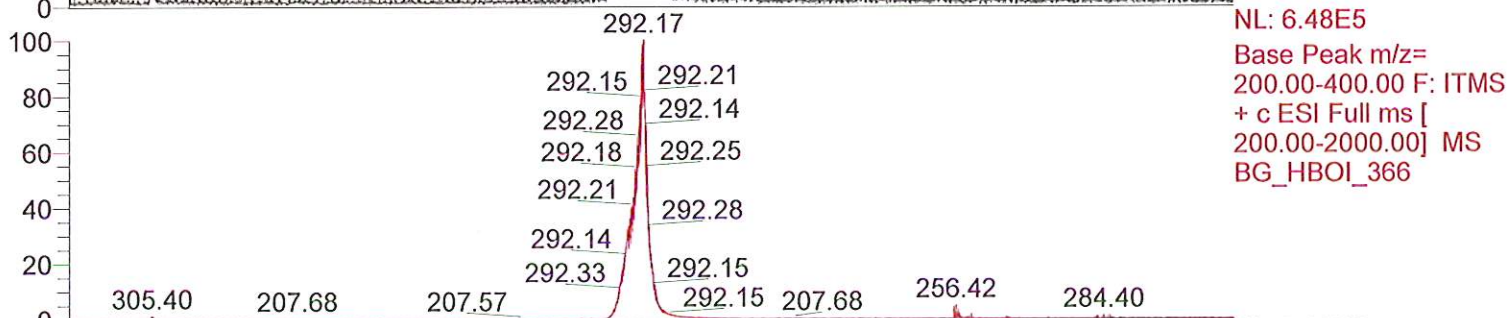
Pump A Solvent D:

Method Description:

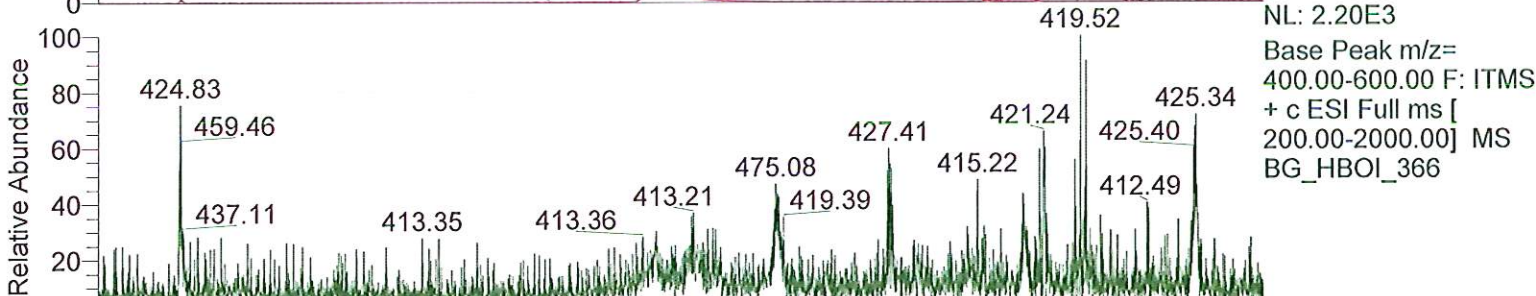
RT: 0.00 - 20.99



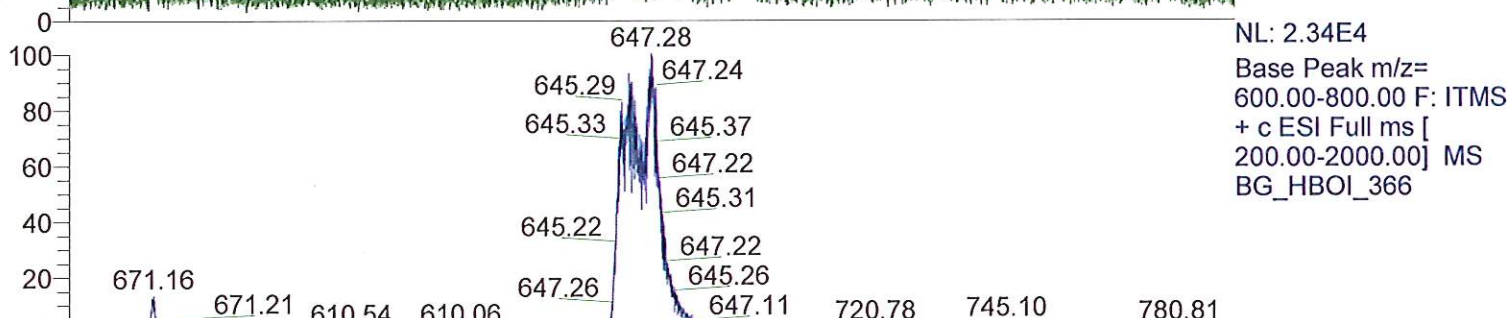
NL: 1.50E6
TIC F: ITMS + c ESI
Full ms [200.00-2000.00] MS
BG_HBOI_366



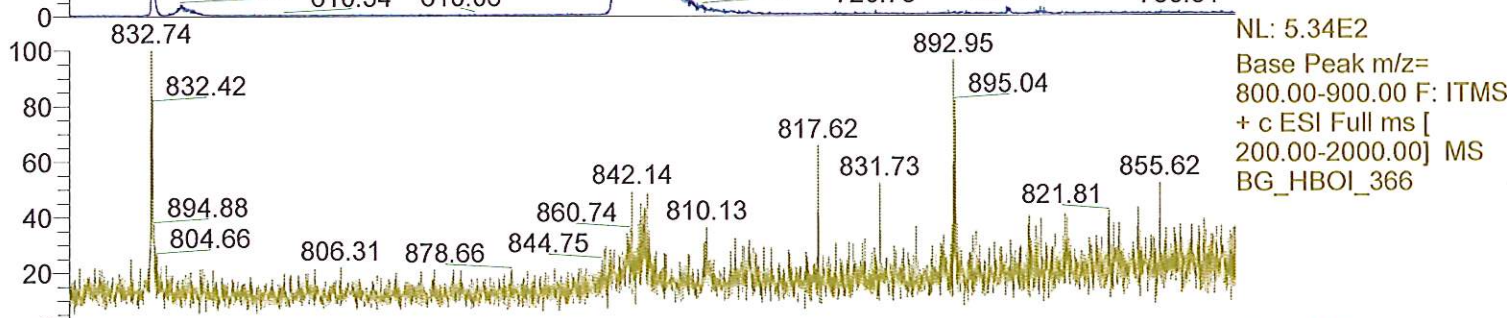
NL: 6.48E5
Base Peak m/z= 200.00-400.00 F: ITMS + c ESI Full ms [200.00-2000.00] MS
BG_HBOI_366



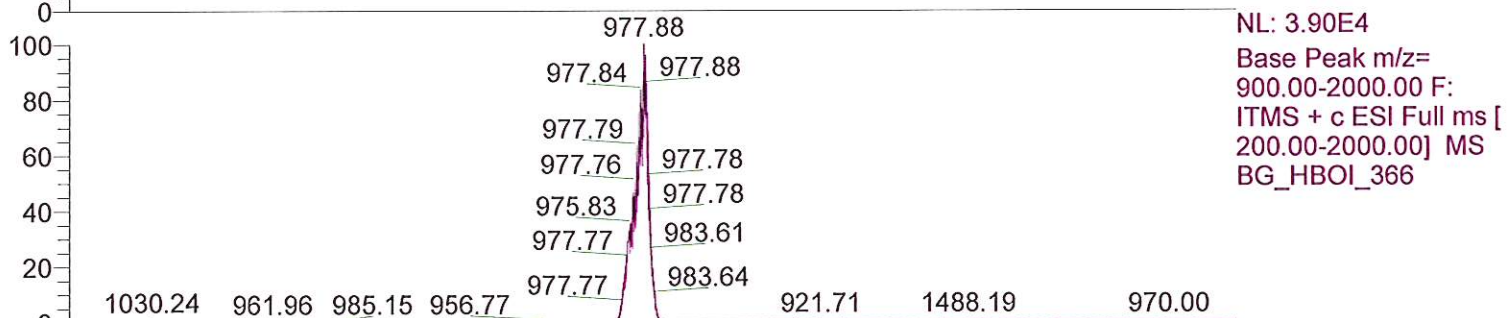
NL: 2.20E3
Base Peak m/z= 400.00-600.00 F: ITMS + c ESI Full ms [200.00-2000.00] MS
BG_HBOI_366



NL: 2.34E4
Base Peak m/z= 600.00-800.00 F: ITMS + c ESI Full ms [200.00-2000.00] MS
BG_HBOI_366



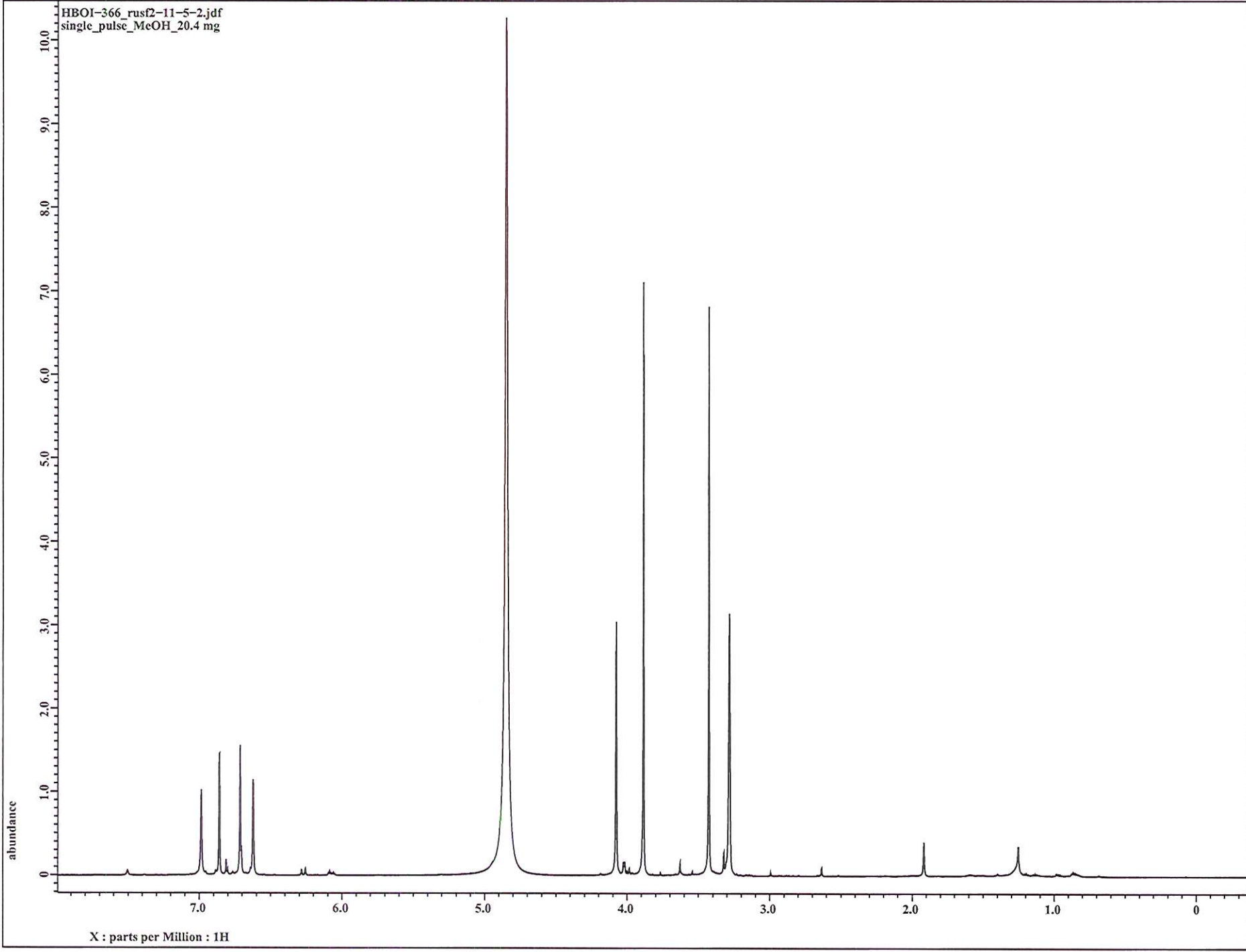
NL: 5.34E2
Base Peak m/z= 800.00-900.00 F: ITMS + c ESI Full ms [200.00-2000.00] MS
BG_HBOI_366



NL: 3.90E4
Base Peak m/z= 900.00-2000.00 F: ITMS + c ESI Full ms [200.00-2000.00] MS
BG_HBOI_366

Time (min)

HBOI-366_rusf2-11-5-2.jdf
single_pulse_MeOH_20.4 mg



HBOI-366_rusf2-11-5-3.jdf
single pulse decoupled gated NOE_MeOH_20.4mg

