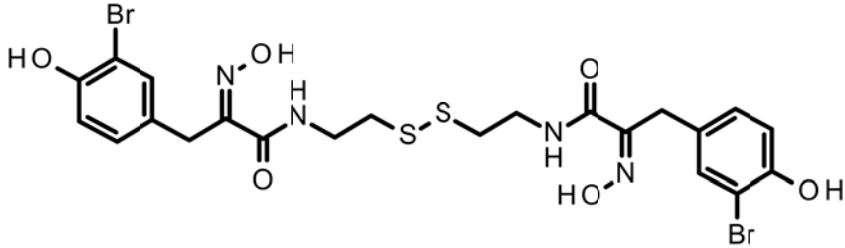


HBOI-391	
Formula Weight : 664.39(2) Exact Mass : 661.950401(4) Formula : C ₂₂ H ₂₄ Br ₂ N ₄ O ₆ S ₂	
Psammaphin A	
C ₂₂ H ₂₄ Br ₂ N ₄ O ₆ S ₂	664.39
<i>Jaspis</i> . Sp. 30-VII-10-4-005 FEDD1-53-4	
Rodriguez et. al. Tetrahedron Letters (1987), 28(42), 4989-92	

PO NUMBER: PO40657 Bar Code: 1447463

hplc (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:TFA (95:5:0.1, v/v), B: CH₃CN:TFA (100:0.1 v/v)

t=0 min A:B (90:10, v/v), t=20 min (100%B) , t=28 min (100%B)

LC-MS (spectrum attached)

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 __21.1_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: _____

30-VII-10-4-005
 540g
 Soaked in a 5:1 EtOAc and EtOH solution, then chopped and ground
 in a blender, slurry then vacuum
 filtered through a celite bed and dried.
 FedD01-52-1

Partitioned 3X w/200mL each H₂O
 and EtOAc

FedD01-52-2
 EtOAc part
 758 mg

FedD01-52-3
 H₂O partition
 XX g

FedD01-52-4
 Butanol partition
 1199.5 mg

CombiFlash
 758 mg run on a 15.5g C18 RF Gold isco column.
 80% H₂O to ACN grad., MeOH to DCM gradient

Psanmaplin

FedD01-53- weight tubes	1 38 mg 1-3,7-15	2 41.9 mg 4-6	3 49.9 mg 16-25	4 66.7 mg 26-29	5 7.2 mg 30-34	6 14 mg 35-49	7 85.2 mg 50-61	8 266.8 mg 62-64	9 5.1 mg 65-84	10 17.1 mg TFA Wash
-------------------------------	------------------------	---------------------	-----------------------	-----------------------	----------------------	---------------------	-----------------------	------------------------	----------------------	------------------------------

D-2000 Elite HPLC System Manager Report

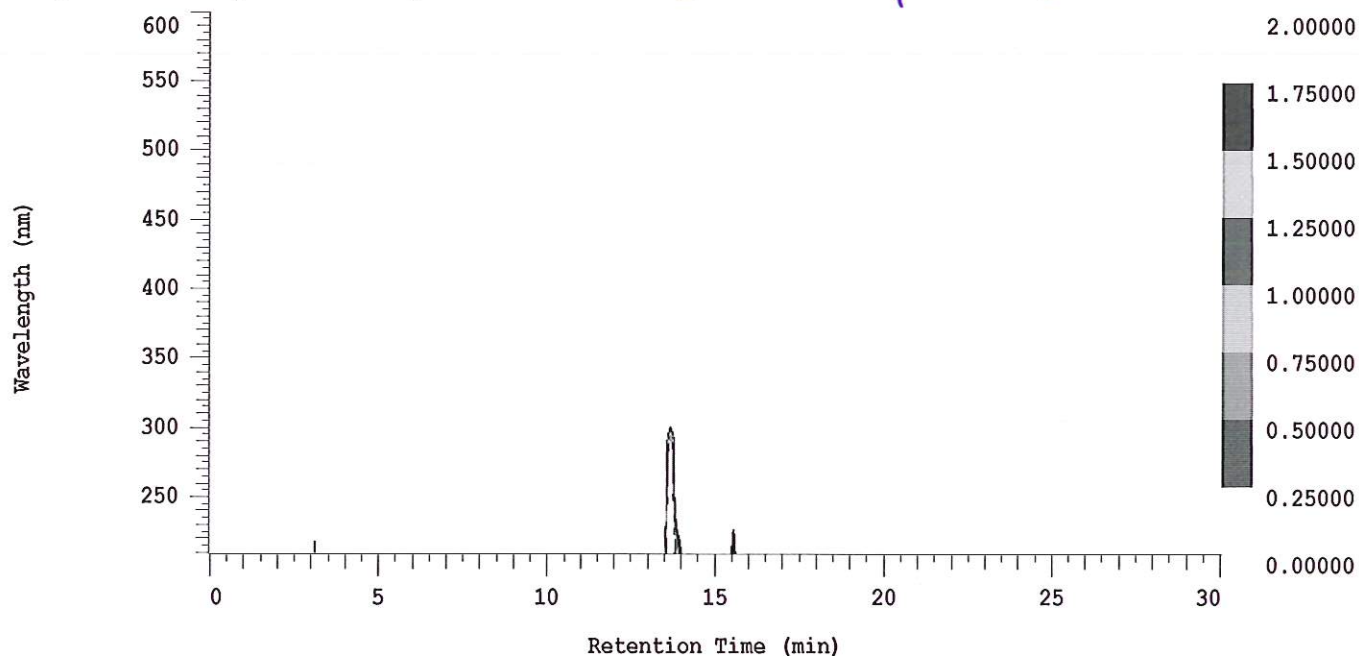
Analyzed: 04/30/2014 10:58 AM

Reported: 04/30/2014 11:29 AM

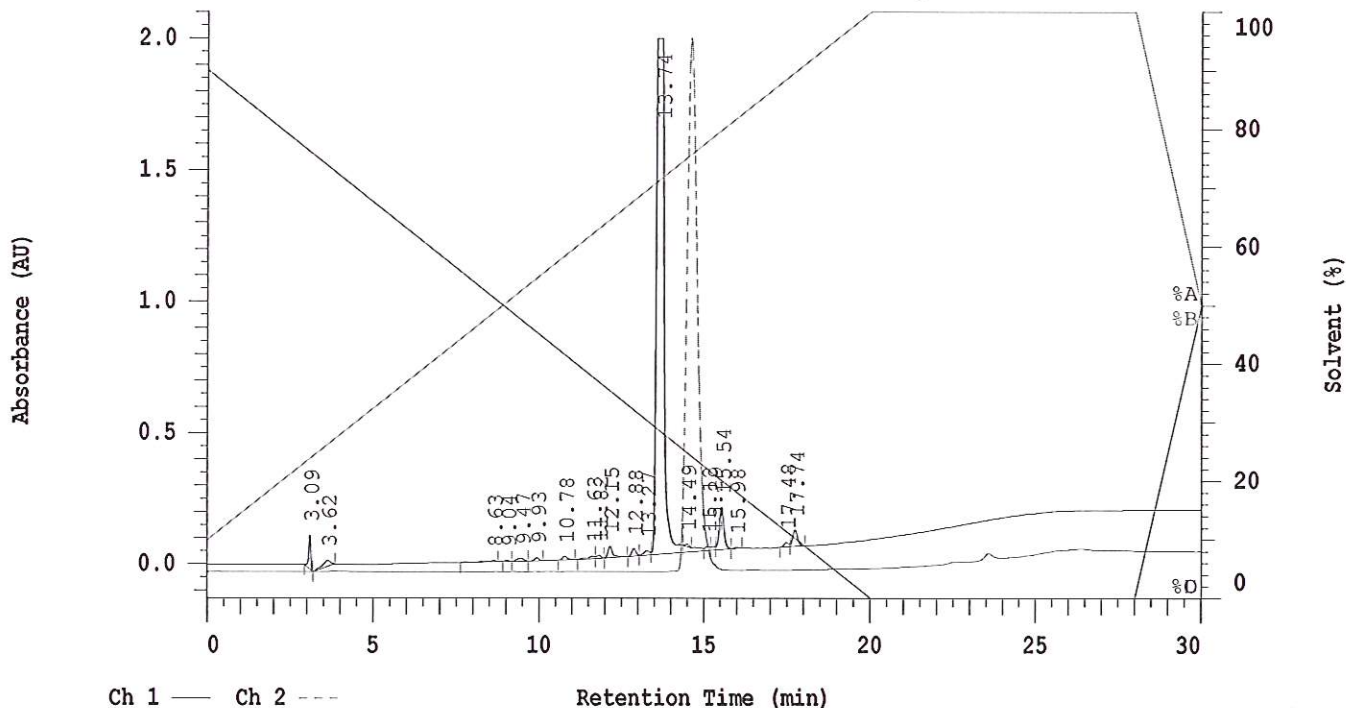
Sample Name: FedD01-53-4

Sample Description: 5 mg/mL

Psammaphin A



Channel 1 Chrom Type: Fixed WL Chromatogram, 230 nm



Acquisition Method: autosampler_30min_wELSD_TFA

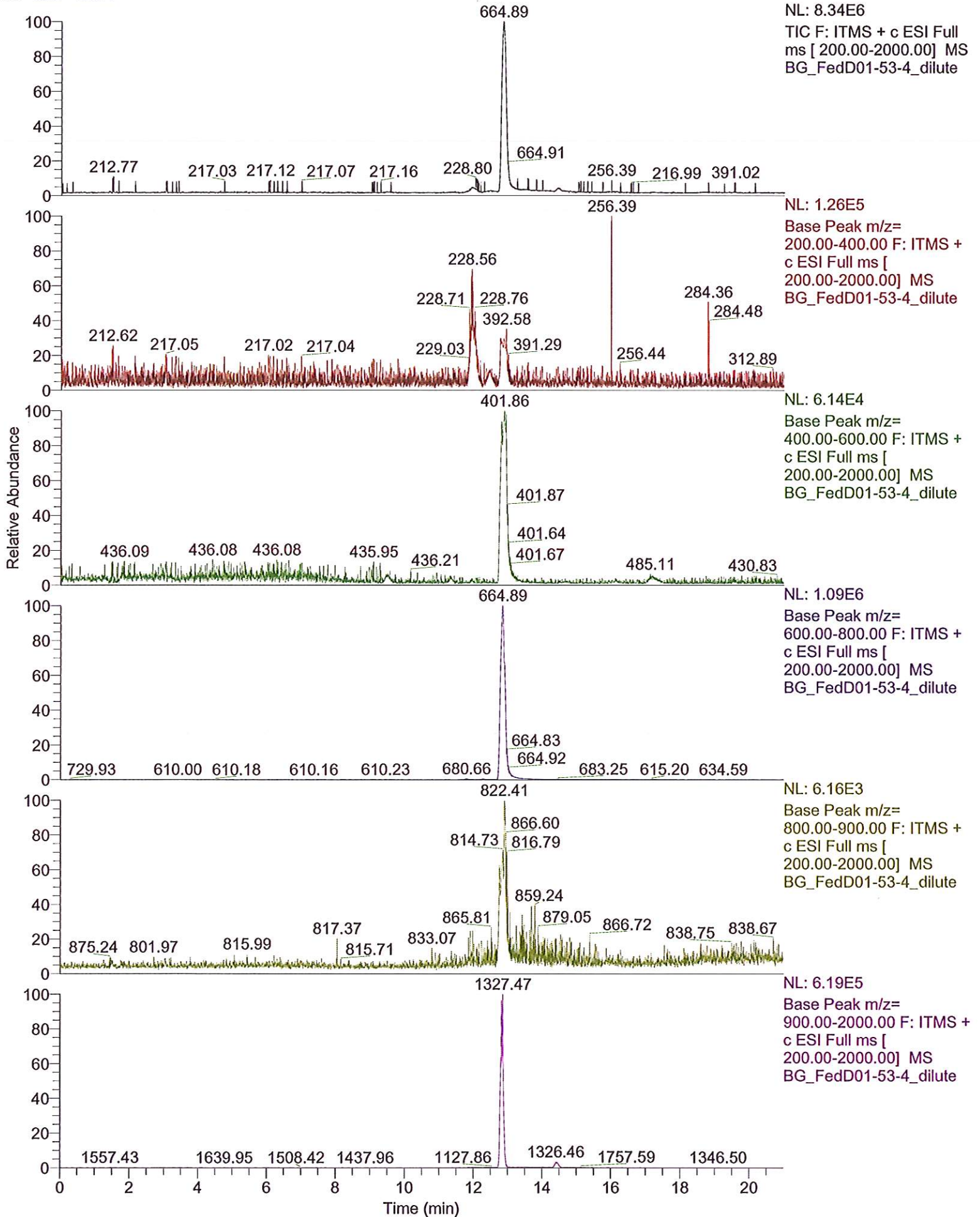
Column Type: Vydac C18

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

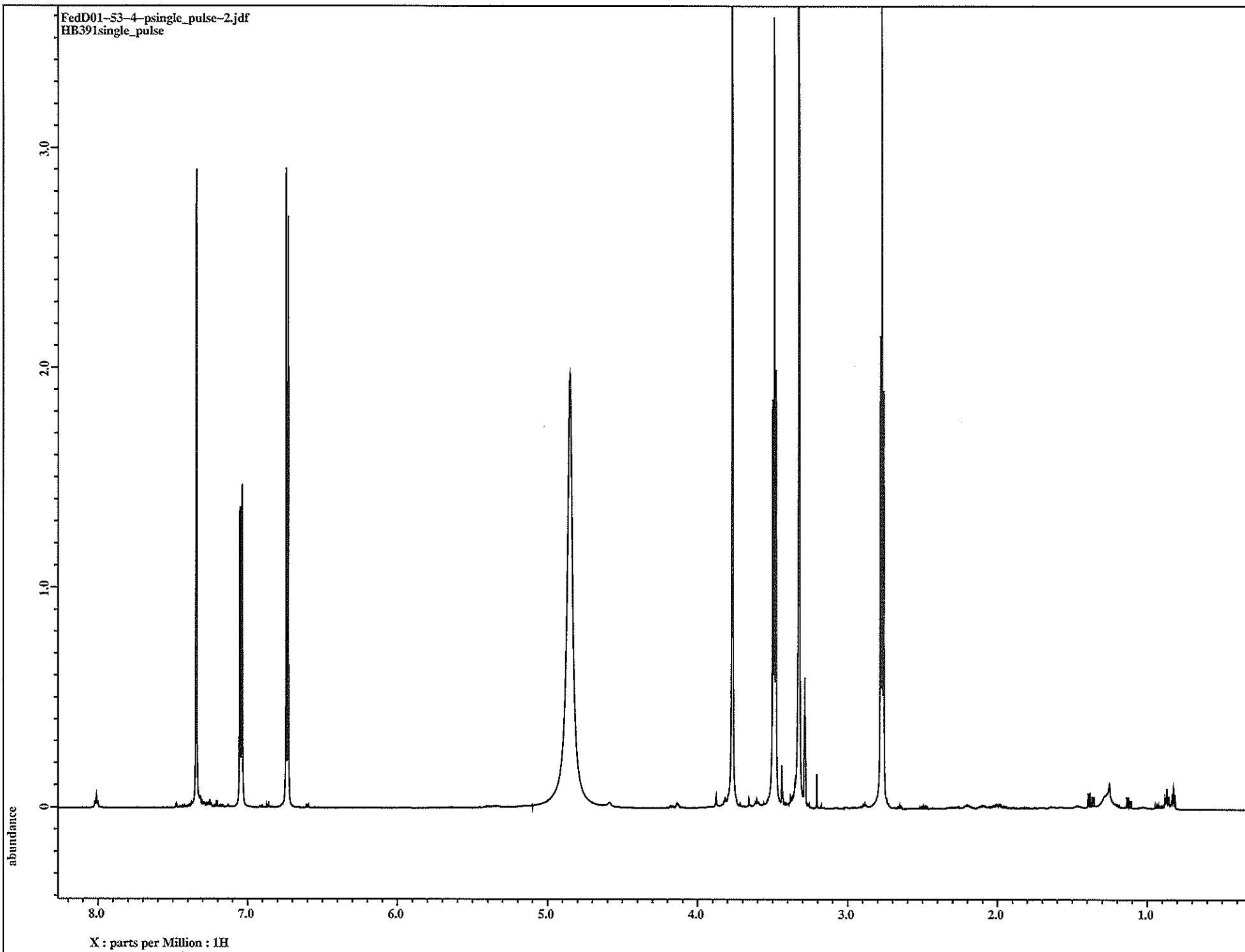
Pump A Solvent B: ACN TFA 0.1%

Method Description:

RT: 0.00 - 20.99



FedD01-53-4-psingle_pulse-2.jdf
HB391single_pulse



FcdD01-53-4-Carbon2single_pulse_dec-2.jdf
HB391-Carbonsingle pulse decoupled gated NOE

