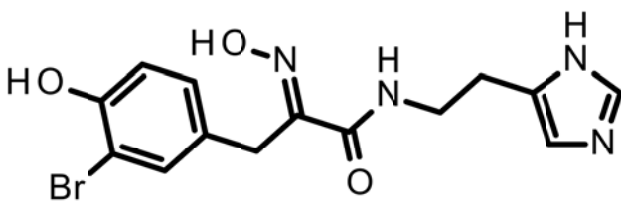


HBOI-390	 <p style="margin-top: 10px;"> Formula Weight : 367.20(1) Exact Mass : 366.032753(2) Formula : C₁₄H₁₅BrN₄O₃ </p>
Benzenepropanamide, 3-bromo-4-hydroxy-α-(hydroxyimino)-N-[2-(1H-imidazol-4-yl)ethyl]-, (E)-	
C ₁₄ H ₁₅ BrN ₄ O ₃	367.20
<i>Jaspis</i> . Sp. FEDD01-65-01	
Ciminiello <i>Journal of Natural Products</i> (1995), 58(5), 689-96	

PO NUMBER: PO40657 Bar Code: 1447453

hplc (trace attached)

Method: Vydac C18 Protein and Peptide, 4.6 x 250mm, 10μ
 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 5.8 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

Psammapin

FedD01-53-weight tubes	1 36 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
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Walter
Inject volume: 150 uL (4 injections)
Flow Rate: 15 mL/min
Column: Vydac, C18 prep
Gradient: (H₂O:ACN) w TFA
Time 0 - 95:5
Time 20 - 0:100
Time 23 - 0:100
Time 23:01 - 95:5
Time 25 - 95:5

Walter
Inject volume: 150 uL (5 injections)
Flow Rate: 15 mL/min
Column: Vydac, C18 prep
Gradient: (H₂O:ACN) w TFA
Time 0- 70:30
Time 14-31:69
Time 17-10:90

FedD01-61-weight tubes run 1	1 4.0 mg 13,14	2 7.8 mg 23,24	3 11.8 mg remainder	
FedD01-62-weight tubes run 2,3,4	1 3.5 mg 13,59,13	2 3.1 mg 14,60,14	3 17.0 mg 23,24,69, 70,23,24	4 16.0 mg remainder

FedD01-57-weight tubes	1 6.8 mg	2 9.1 mg	3 4.9 mg	4 3.1 mg	5 27.8 mg
Run 1	8,9	18	20	23	remainder
Run 2	55,56	65	67	70	remainder
Run 3	81,82	91	93	96	remainder
Run 4	6	16,17	18	21	remainder
Run 7	32	42,43	44	47	remainder

Combined FedD01-64-1 with FedD01-61-1 and FedD01-62-1 to make FedD01-65-1

Combined FedD01-57-1 with FedD01-62-2 then 2 injections on water (same gradient as FedD01-53-3) to make 64-1 (tube 13)

D-2000 Elite HPLC System Manager Report

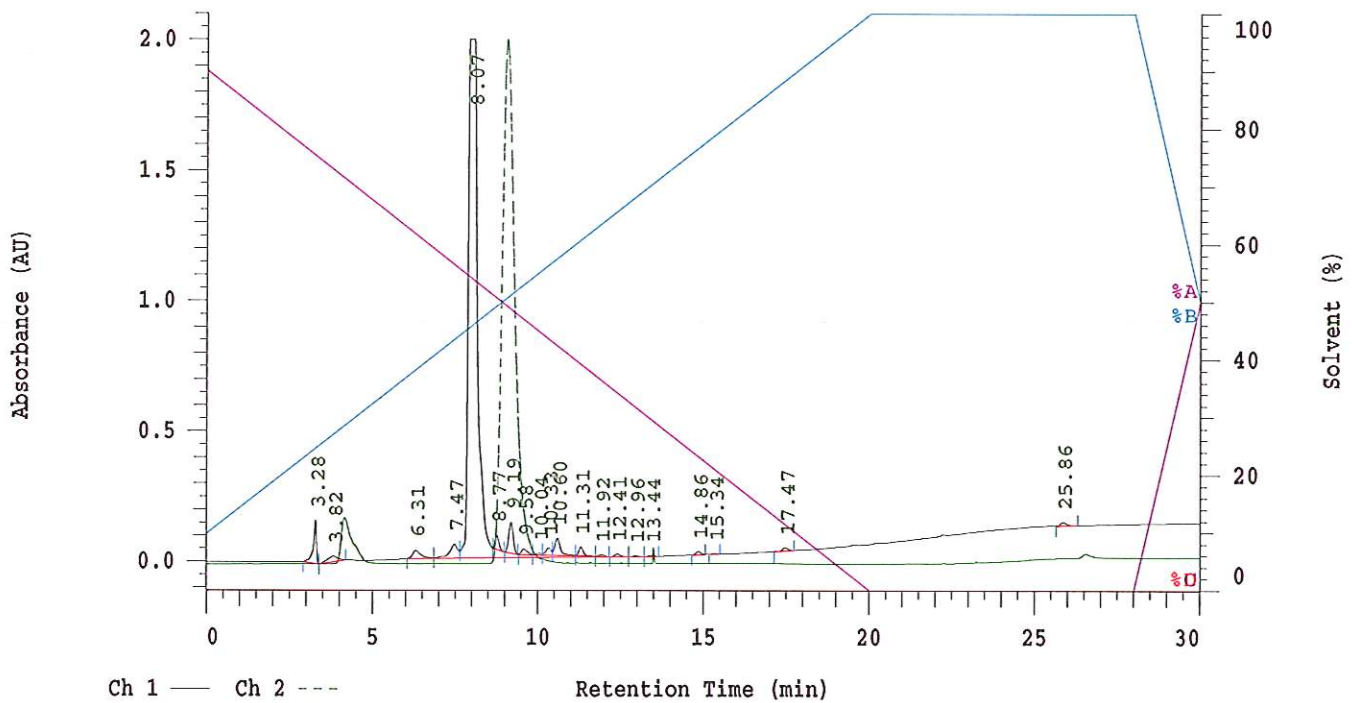
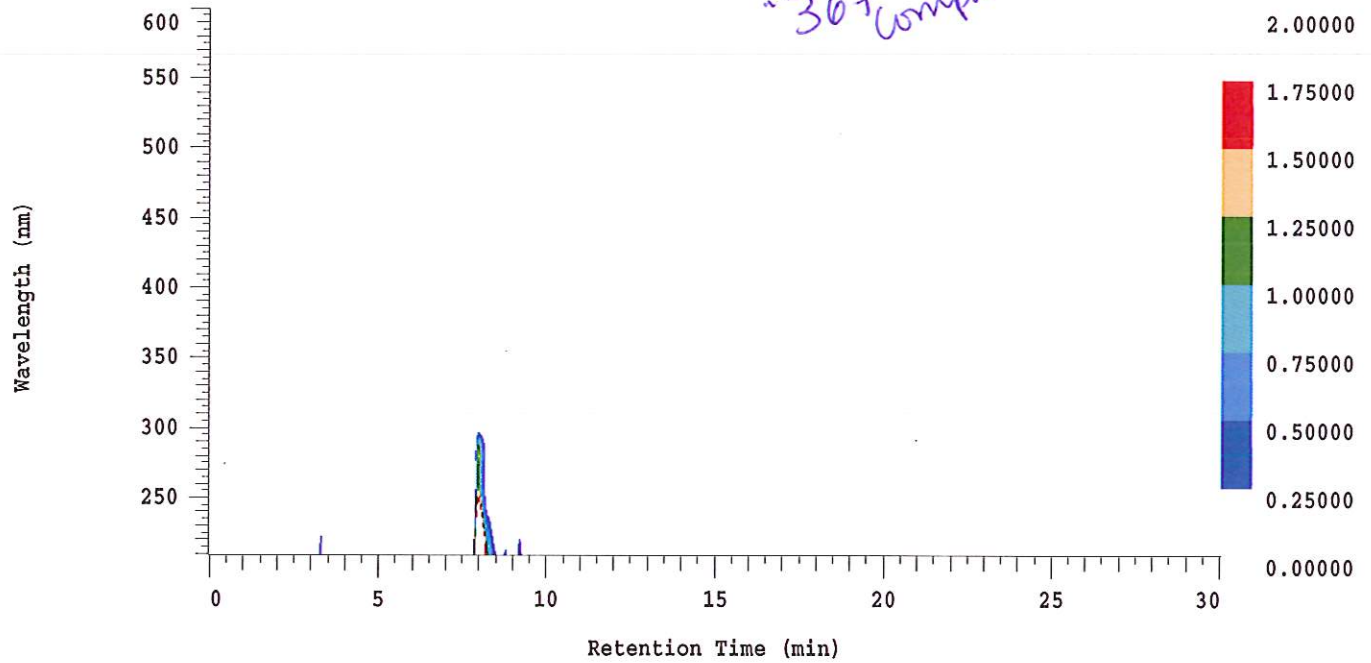
Analyzed: 03/21/2014 04:18 PM

Reported: 04/25/2014 09:38 AM

Sample Name: FedD01-65-1

Sample Description: 5 mg/mL

"367" compound



Acquisition Method: gradient30minTFA_noauto_wELSD

Column Type: Vydac C18

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

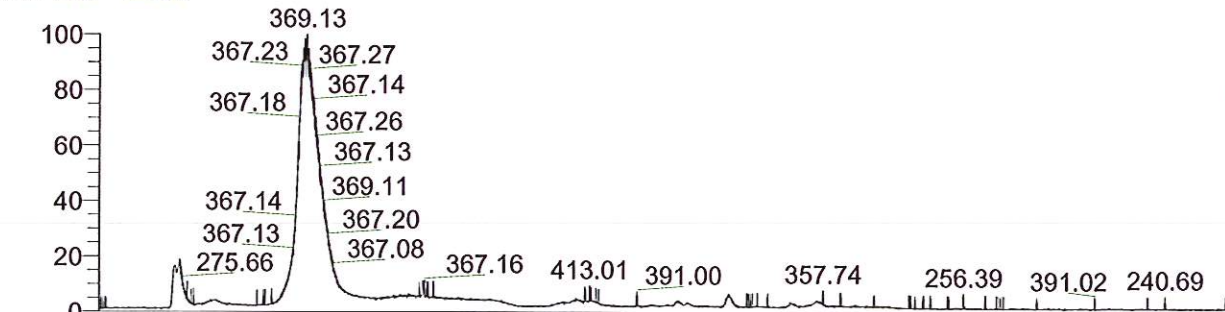
Pump A Solvent B: ACN:0.1%TFA

Pump A Solvent C:

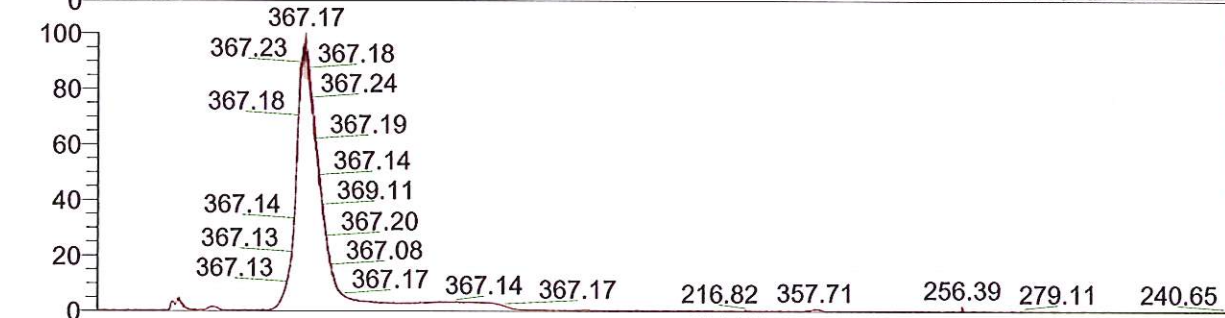
Pump A Solvent D:

Method Description:

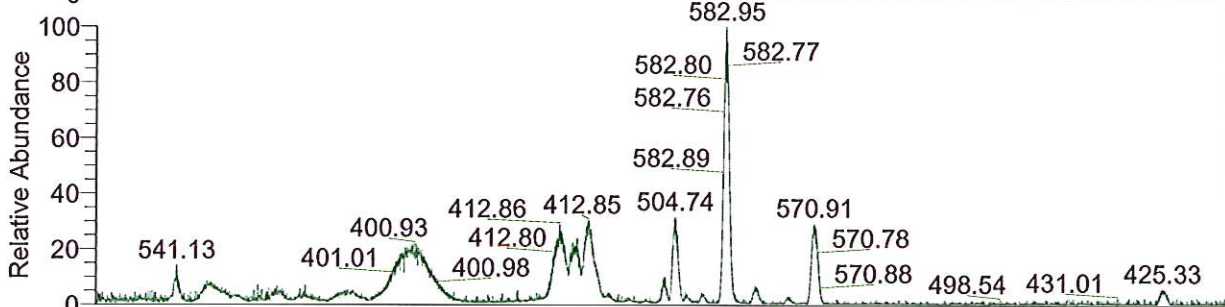
RT: 0.00 - 20.99



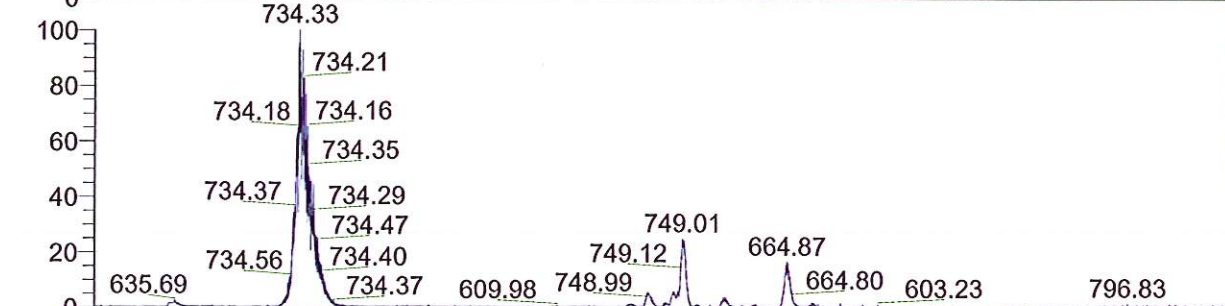
NL: 1.04E7
TIC F: ITMS + c ESI Full ms [200.00-2000.00] MS
BG_Fedd01-65-1_140430111045



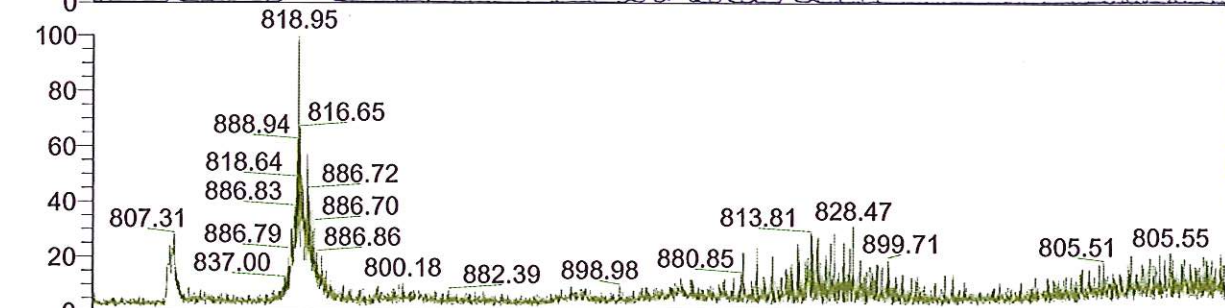
NL: 4.18E6
Base Peak m/z= 200.00-400.00 F: ITMS + c ESI Full ms [200.00-2000.00] MS
BG_Fedd01-65-1_140430111045



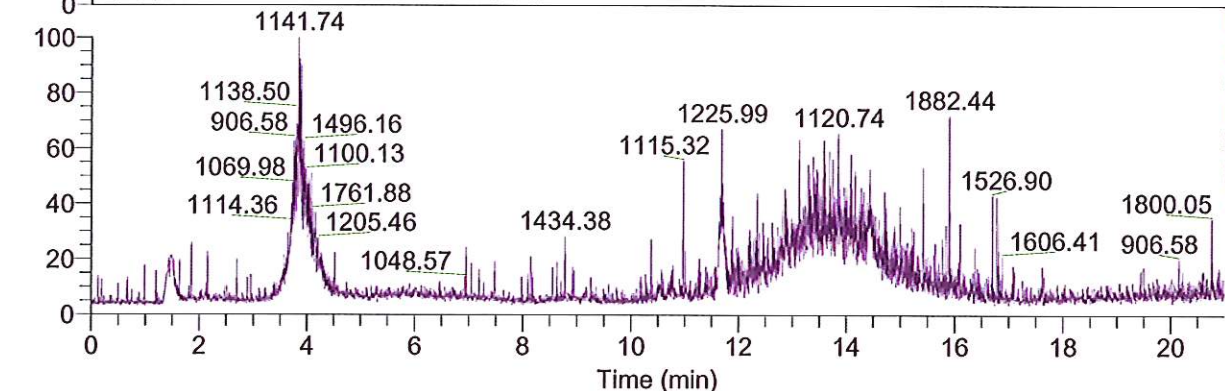
NL: 1.31E5
Base Peak m/z= 400.00-600.00 F: ITMS + c ESI Full ms [200.00-2000.00] MS
BG_Fedd01-65-1_140430111045



NL: 1.28E5
Base Peak m/z= 600.00-800.00 F: ITMS + c ESI Full ms [200.00-2000.00] MS
BG_Fedd01-65-1_140430111045



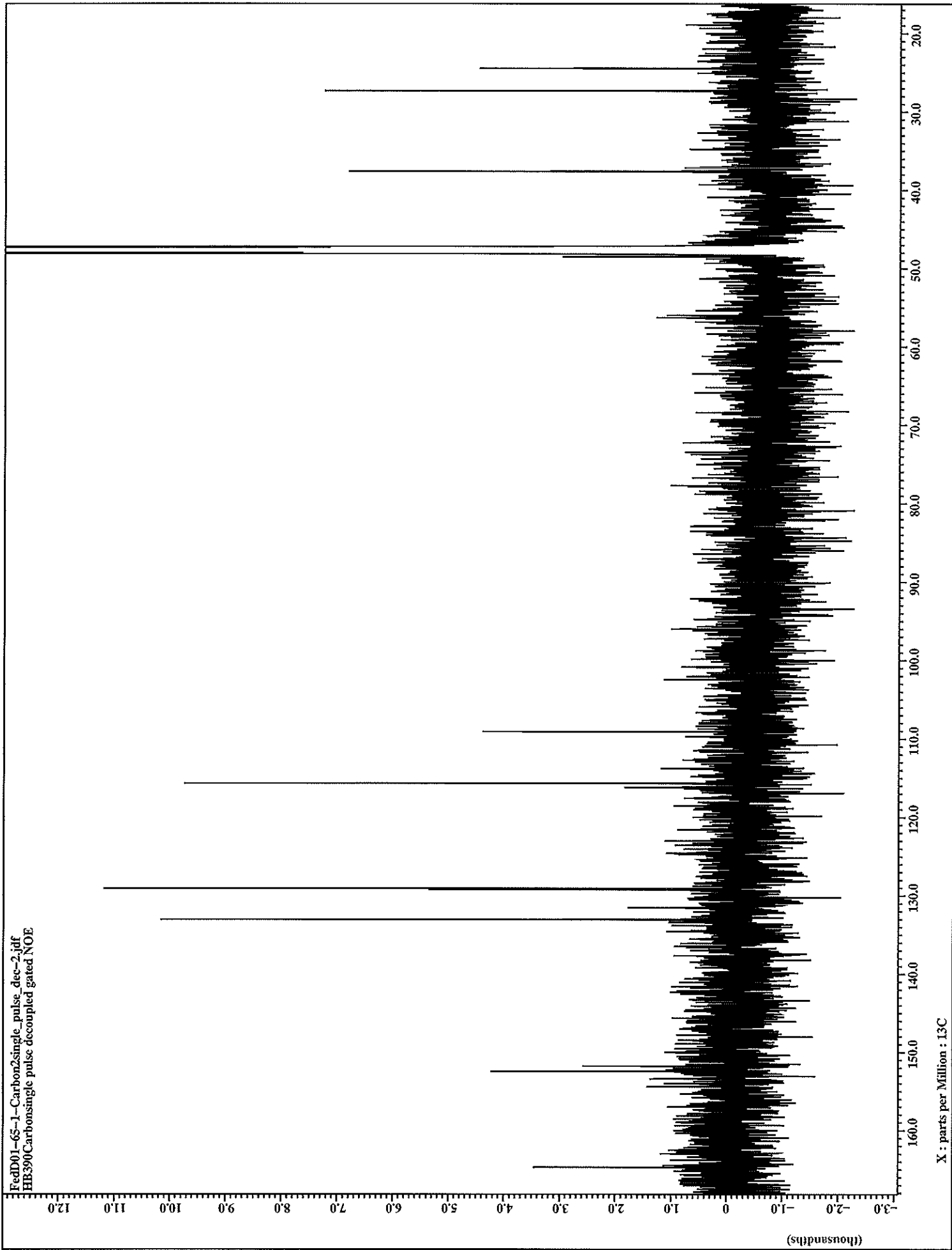
NL: 7.56E3
Base Peak m/z= 800.00-900.00 F: ITMS + c ESI Full ms [200.00-2000.00] MS
BG_Fedd01-65-1_140430111045



NL: 6.65E3
Base Peak m/z= 900.00-2000.00 F: ITMS + c ESI Full ms [200.00-2000.00] MS
BG_Fedd01-65-1_140430111045

Time (min)

FedD01-6S-1-Carbon2single_pulse_dec-2.jdf
EB390Carbon2single_pulse decoupled gated NOE



FcdD01-65-1-psinglc_pulse-4.jdt
HB390single_pulse

