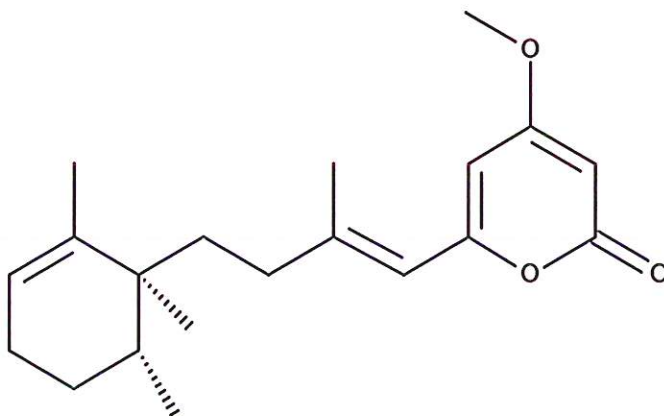


HBOI-384



SOLASSIN D

 $C_{20}H_{28}O_3$

316.20

Pachastrellidae

Killday, Longley, McCarthy, Pomponi, Wright, Neale, and Sills, 1993, *J. Nat. Prod.*, 56(4) 500-507

☒ isolation scheme (Visio) attached

☒ 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 (95:5, v/v), B: CH₃CN
 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 20.4 mg

For further information contact:

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awrigh33@hboi.fau.edu, 772-242-2459

Date: 1/28/13

22-V-93-3-004
Pachestrellidae
Heptane Dionex Extraction
ROBL22-99-2
500mg

12g Redi-Sep Gold
Combi-Flash Non-
Polar Method
100% HEP to 100%
EtOAc plus wash
gradient
30 ml/min

ROBL25-52- Weight Tubes	1 67.1mg 5-11	2 20.3mg 12-14	3 91.6mg 15-18	4 32.1mg 19-23	5 12.9mg 24-40	6 15.5mg 41-43
			7 31.3mg 44-53	8 1.3mg 54-57	9 96.5mg 58-71	10 31.2mg 72-79

ROBL25-52-3
HPLC 20:80 H₂O/ACN
50mg

ROBL25-62- mg	1 3.1	2 1.2	3 33.0	4 1.2

ROBL25-52-4
HPLC 20:80 H₂O/ACN
32mg

ROBL25-62- mg	5 5.3	6 21.0

ROBL25-52-7 HB383
ROBL25-52-9 HB382
ROBL25-62-6 HB384

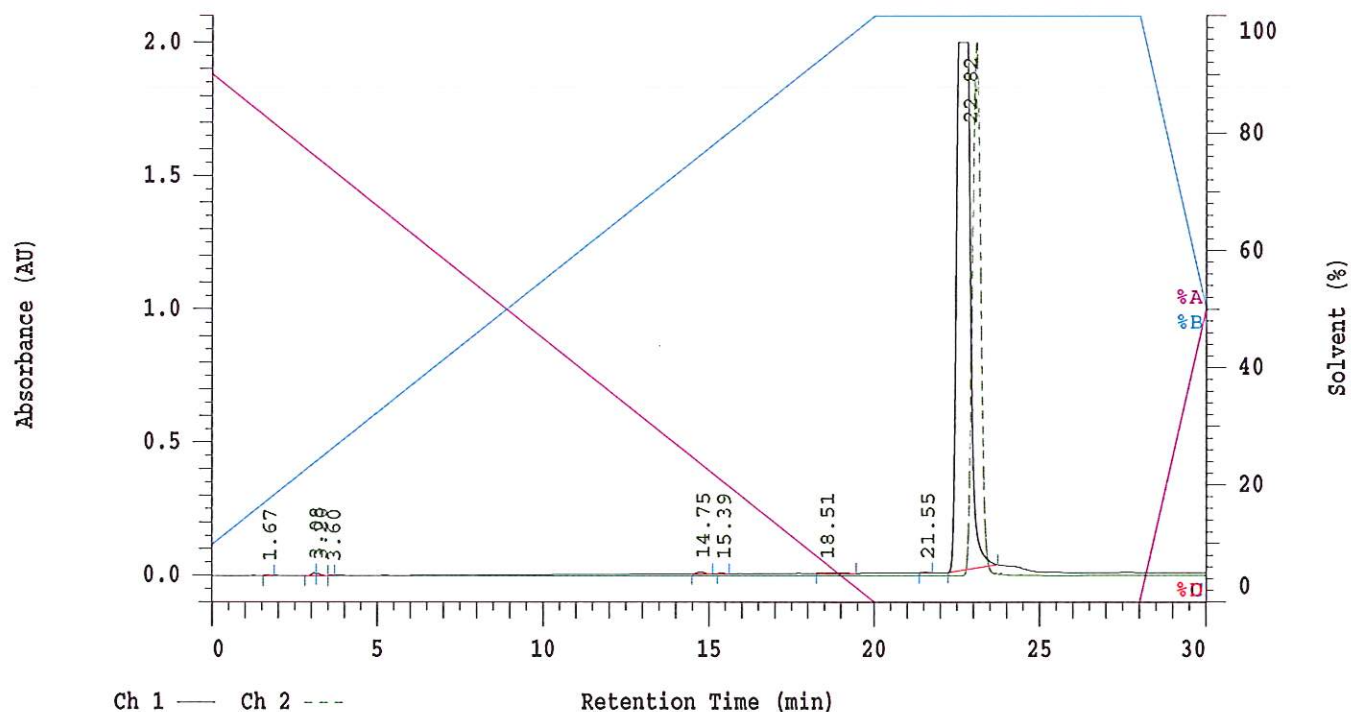
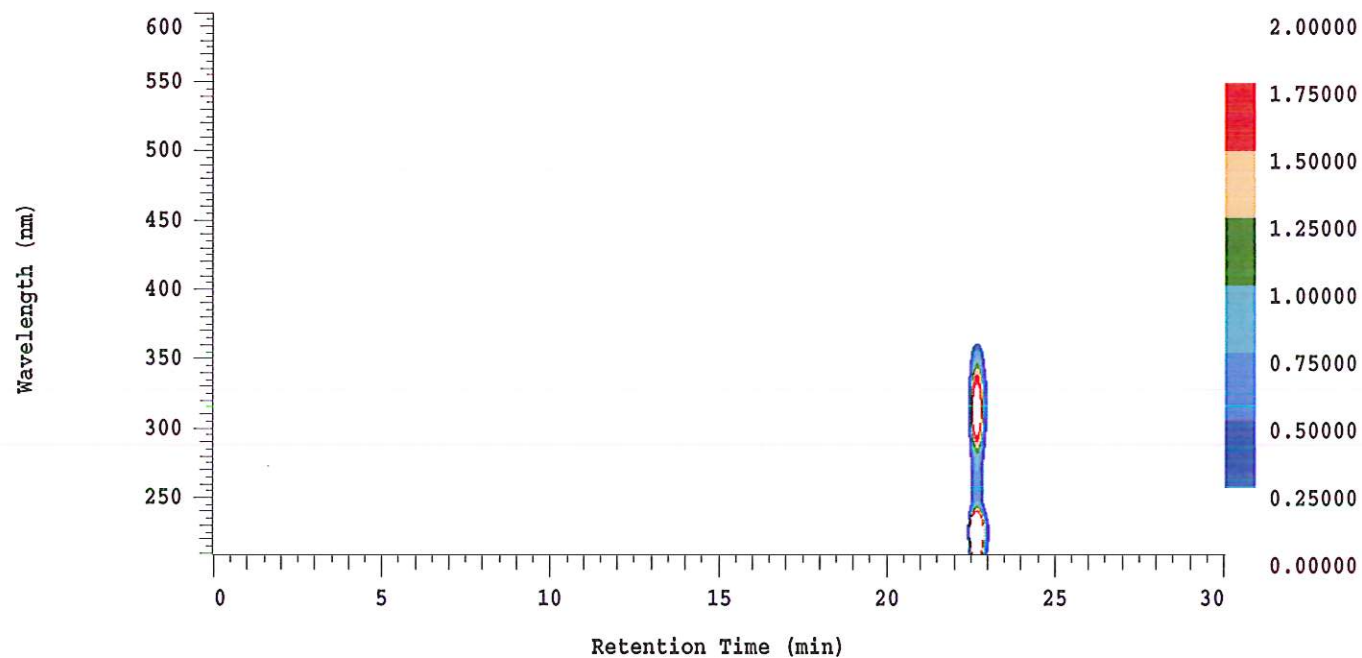
D-2000 Elite HPLC System Manager Report

Analyzed: 01/24/2013 05:12 PM

Reported: 01/24/2013 05:47 PM

Sample Name: HBOI-384

Sample Description: HBOI-384



Acquisition Method: autosamp_30mn_UV220_wELSD

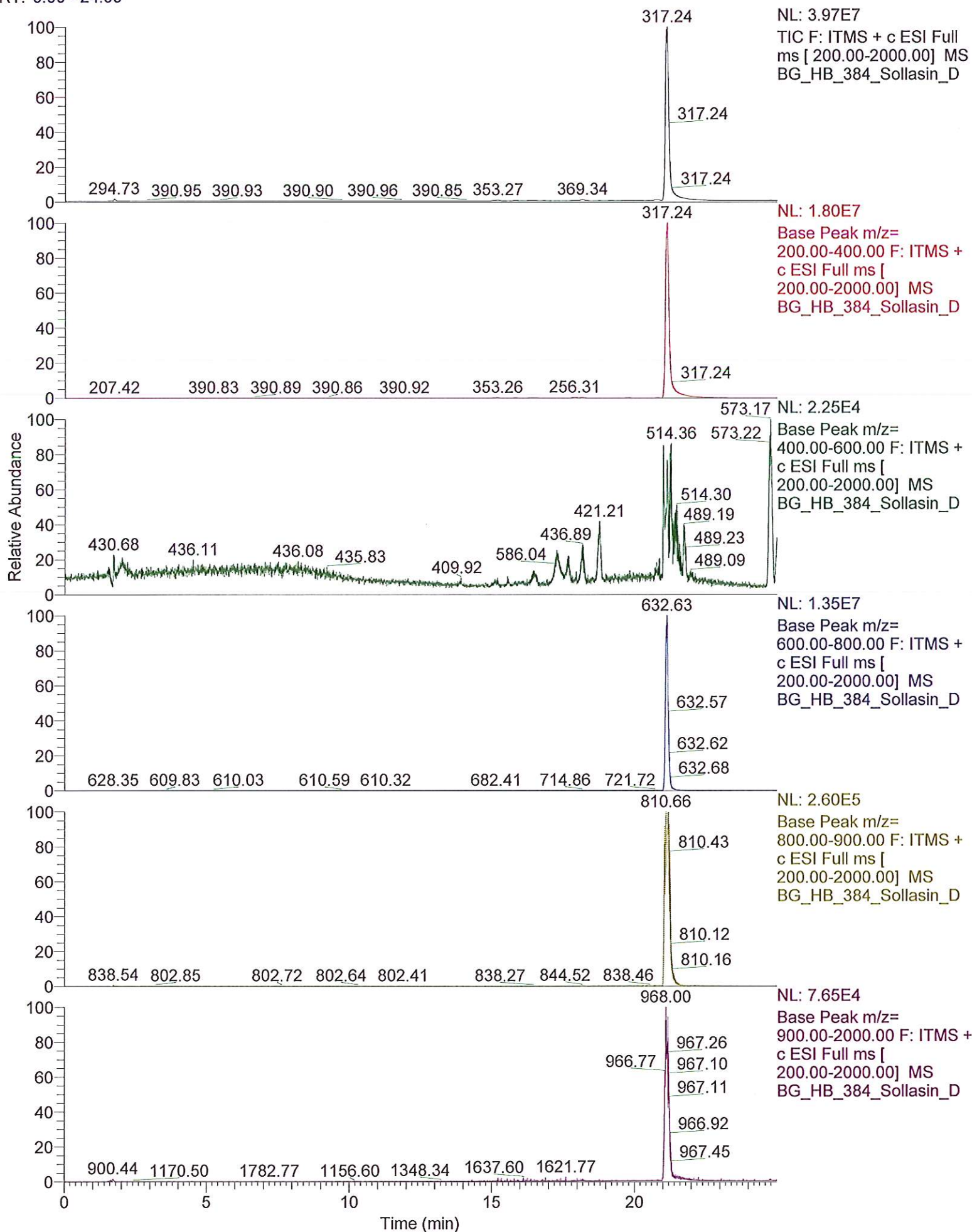
Column Type: Vydac C18

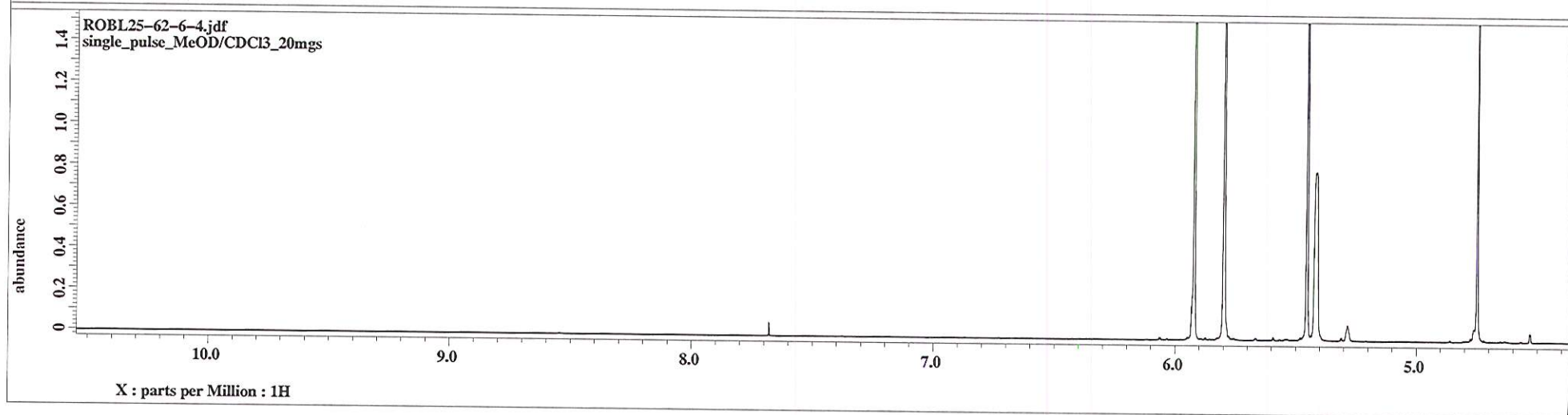
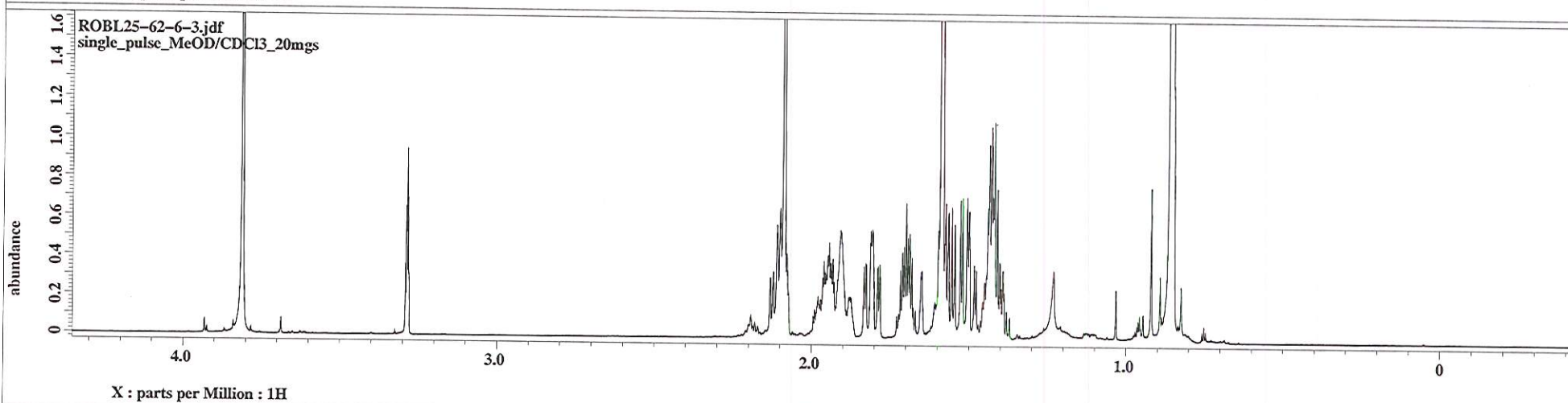
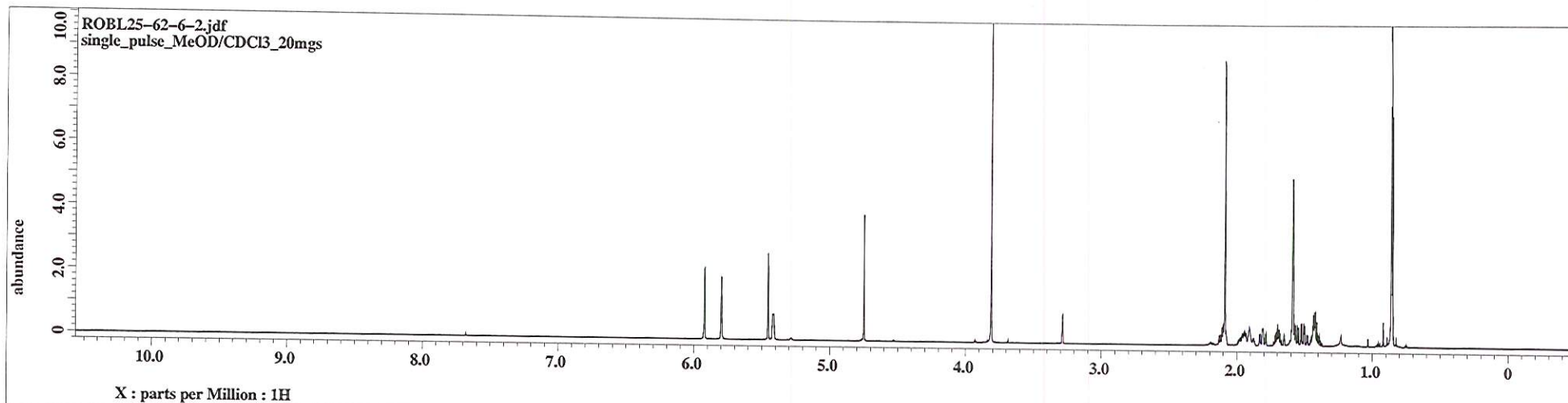
Pump A Solvent A: H2O/5% ACN

Pump A Solvent B: ACN

Method Description:

RT: 0.00 - 24.99





single_pulse_decROBL25-62-6-2.jdf
single pulse decoupled gated NOE_MeOD/CDCl3

