

BMS-986142

Exact Mass: 572.22

Elemental Analysis: C, 67.12; H, 5.28; F, 6.64; N, 9.78; O, 11.18

NSC-828251

Reaction Conditions:

Reaction Molarity	
Pressure	
Temperature	
Time	

Reactants:

	Reactant	MF	Limit?	MW	Eq	Moles	Sample Mass	Vol	Molarity	d	% Wt	FM	Reactant Mass

Solvents:

	Name	Ratio	Volume

Products:

	Product	Structure	MF	Actual Mass	Actual Mol	Yield	Purity	MW	Eq	Theo Mol	Theo Mass	FM

Preparation:

REF: *J. Med. Chem.* **2016**, 59, 9173–9200; *Org. Lett.* **2018**, 20, 13, 3736–3740

Samples from experiments NBook2-199 (both lots) and Nbook2-200 (both lots) were combined and the resulting solid (~2.6g) were recrystallized from THF, MeOH and acetone heating only to 35 C. Three different crops were obtained from this process with the respective analytical results:

201-001: 1.3g purity: 97% by chiral HPLC

201-002: 0.22g purity 93% by chiral HPLC

201-003: 0.44g purity 87% by chiral HPLC

Based on these results, crop 001 was further analyzed for submission to the repository as **NSC-828251**

LSC-ODL-NBook2-201-001:

Purity by HPLC: 97%

LCMS [M+H] for $C_{32}H_{31}F_2N_4O_4$; expected: 573.23; found: 573.40

Elemental Analysis for $C_{32}H_{30}F_2N_4O_4 \cdot 0.64 H_2O$: C, 65.80; H, 5.40; F, 6.50; N, 9.59; O, 12.71
Found: C, 65.83; H, 5.54; N, 9.57

mp: 224-227 C (lit: 222-225 C)

1H NMR (400 MHz, DMSO- d_6) δ 10.79 (s, 1H), 8.06 (s, 1H), 7.94 (d, J = 7.9 Hz, 1H), 7.72 (dd, J = 14.4, 8.1 Hz, 1H), 7.54 (d, J = 10.8 Hz, 1H), 7.45 (s, 1H), 7.43 – 7.25 (m, 4H), 4.16 (d, J = 1.4 Hz, 1H), 3.73 (d, J = 8.0 Hz, 3H), 2.90 (dd, J = 17.1, 4.8 Hz, 1H), 2.43 (dd, J = 16.9, 11.6 Hz, 1H), 1.92 (s, 1H), 1.86 (d, J = 16.1 Hz, 2H), 1.75 (s, 3H), 1.57 (dt, J = 11.6, 6.6 Hz, 1H), 1.20 – 1.11 (m, 1H), 1.09 (s, 6H).

^{19}F NMR (376 MHz, DMSO- d_6) δ -121.56 (ddt, J = 14.5, 9.6, 5.2 Hz), -129.64 (d, J = 11.1 Hz).

Single Injection Report



Agilent Technologies

Data file: LSC-ODL-NBook2-201-001.dx

Sequence Name: SingleSample

Project Name: Agilent

Sample name: LSC-ODL-NBook2-201-001

Operator: SYSTEM

Instrument: 1200 HPLC

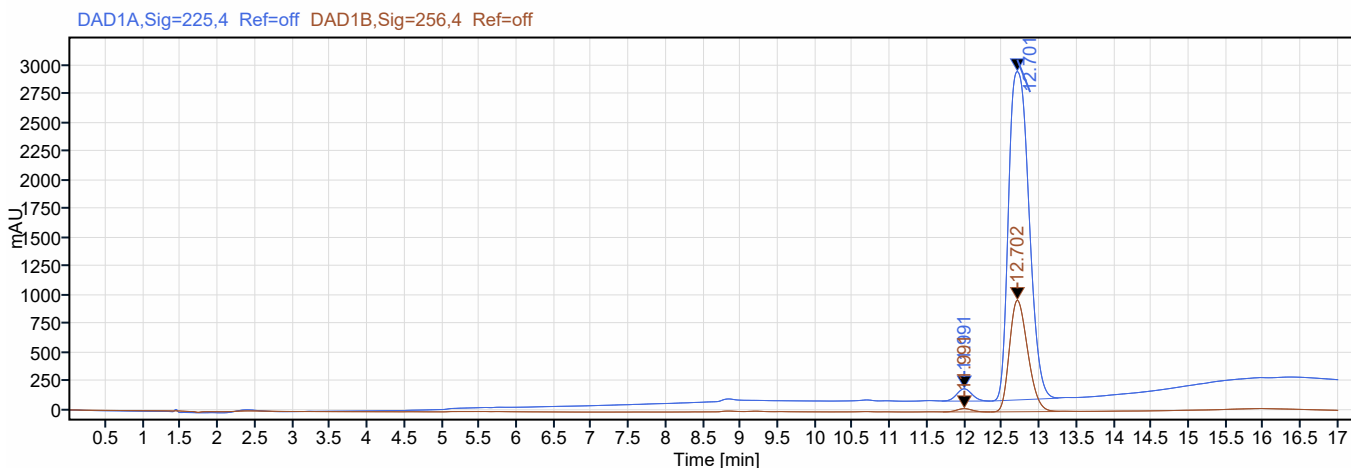
Injection date: 2020-12-03 09:10:05-05:00

Inj. volume: 10.000

Location: 21

Acq. method: LC_20201027_BMS Analysis.amx

Processing method: LC Area Percent.pmx



Signal: DAD1A,Sig=225,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
11.991	VB	0.65	1536.95	106.98	2.75	
12.701	BV	0.89	54419.09	2862.84	97.25	
		Sum	55956.04			

Signal: DAD1B,Sig=256,4 Ref=off

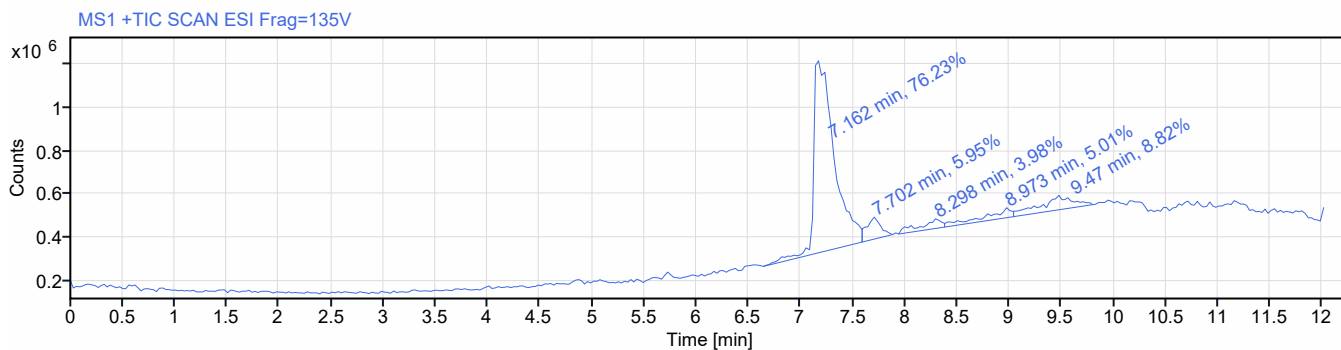
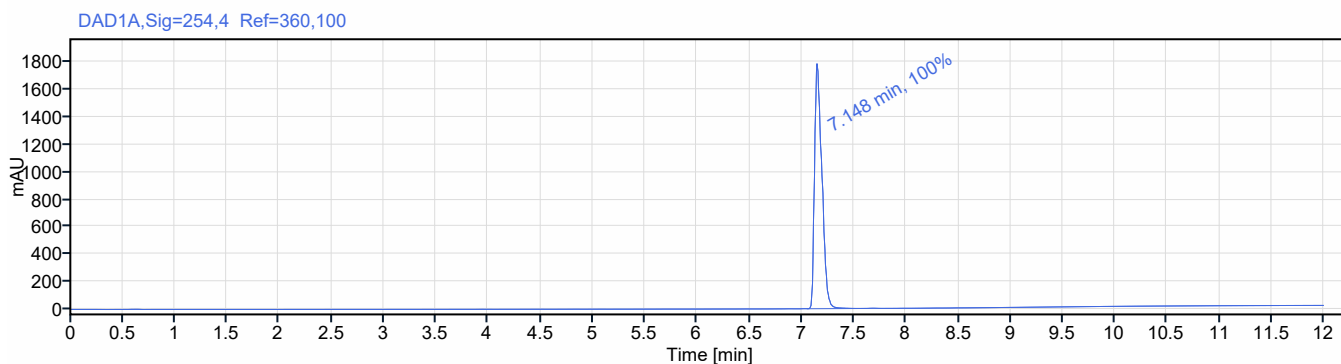
RT [min]	Type	Width [min]	Area	Height	Area%	Name
11.991	VB	0.63	412.37	28.91	2.43	
12.702	BV	0.92	16547.78	971.61	97.57	
		Sum	16960.14			

Qualitative Report



Agilent Technologies

Sequence Name: SingleSample
Data file: LSC-ODL-NBook2-201-001.dx
Sample name: LSC-ODL-NBook2-201-001
Instrument: LCMS
Inj. volume: 2.000
Acq. method: LCMS_20190424NucleosidediF.amx
Processing method: LC_MS Sample Purity_DefaultMethod_report.pmx
Project Name: LSC
Operator : SYSTEM
Acquired on: 2020-12-03 10:14:55-05:00
Location: P1-A4



Signal: DAD1A,Sig=254,4 Ref=360,100

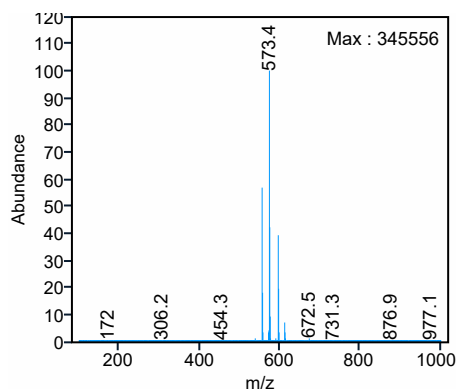
RT [min]	Area	Area%
7.148	8767.4745	100.0000
Sum	8767.4745	

Signal: MS1 +TIC SCAN ESI Frag=135V

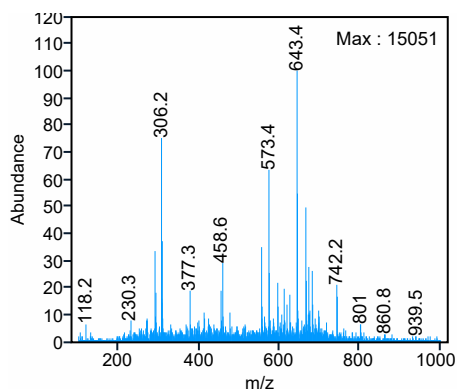
RT [min]	Area	Area%
7.162	12227734.7987	76.2335
7.702	953894.9243	5.9470
8.298	638542.9318	3.9810
8.973	804219.2542	5.0139
9.470	1415442.7156	8.8245
Sum	16039834.6245	



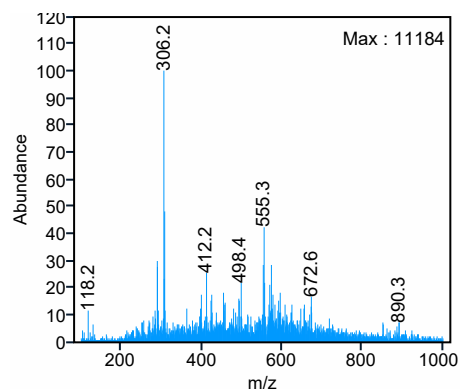
Peak RT 7.162



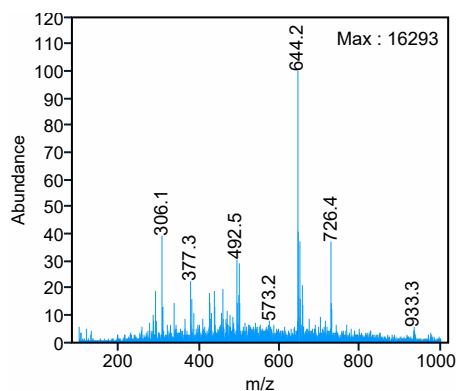
Peak RT 7.702



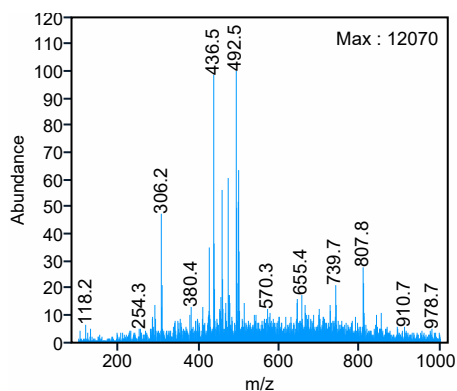
Peak RT 8.298

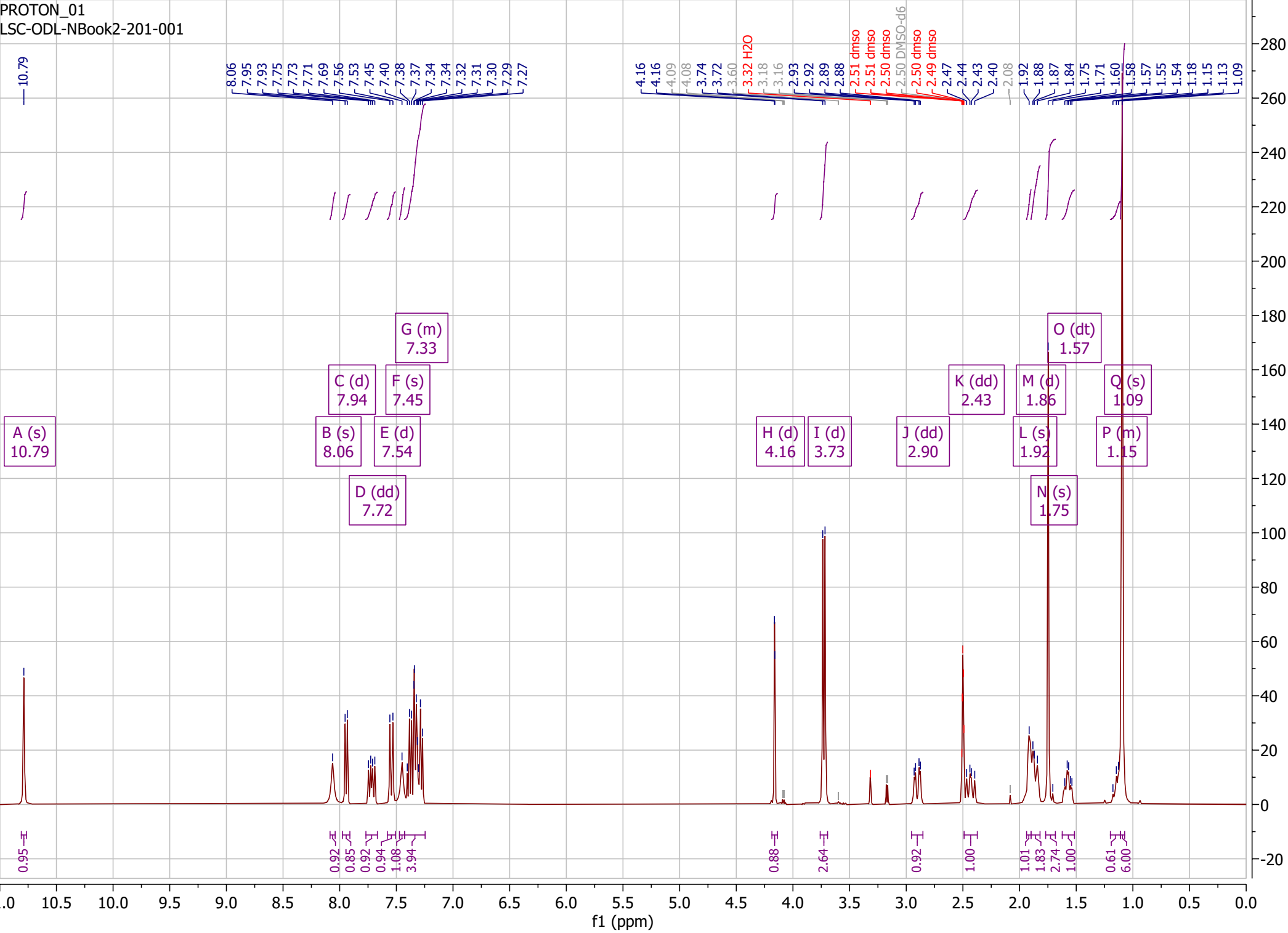


Peak RT 8.973

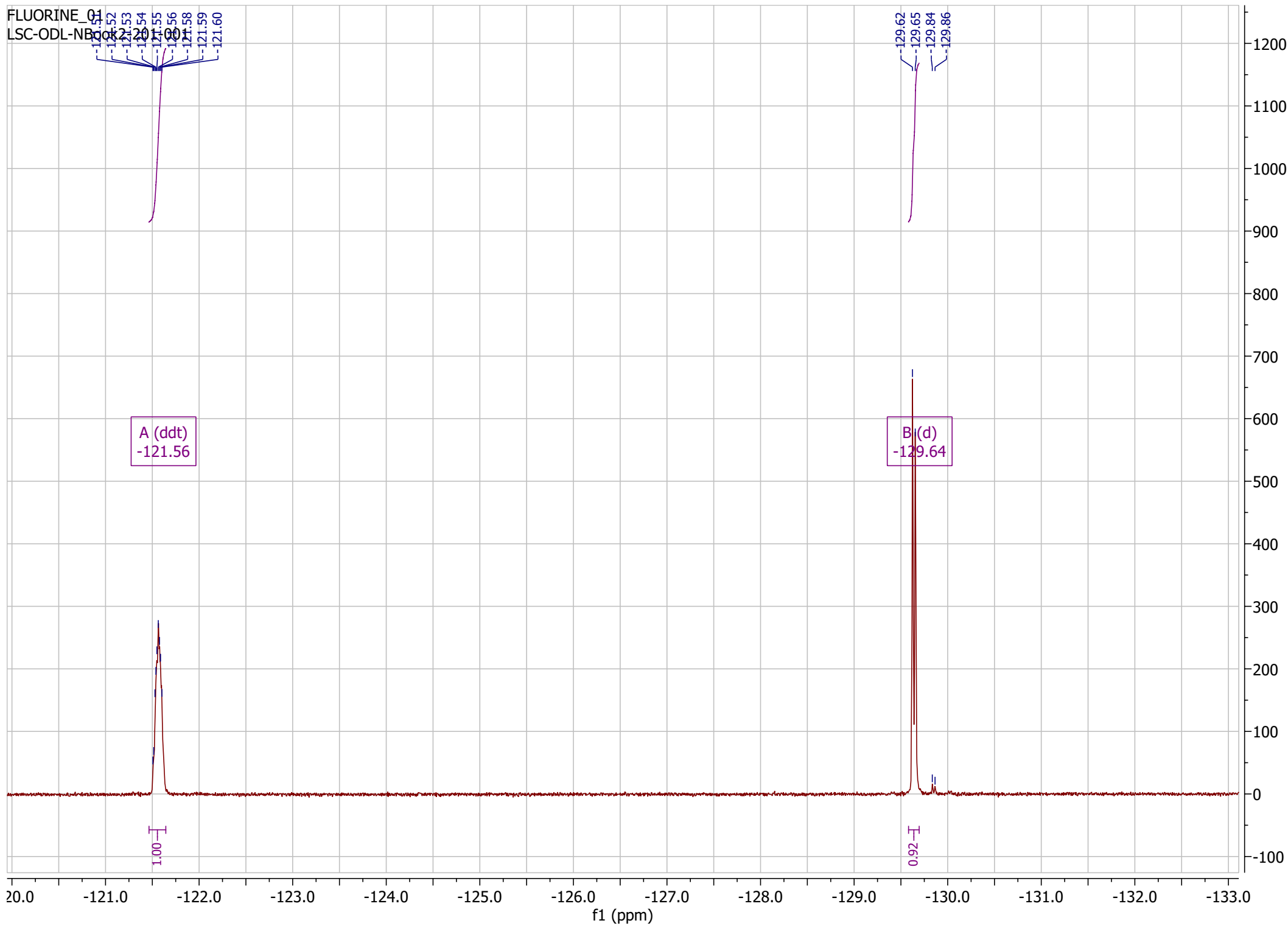


Peak RT 9.470

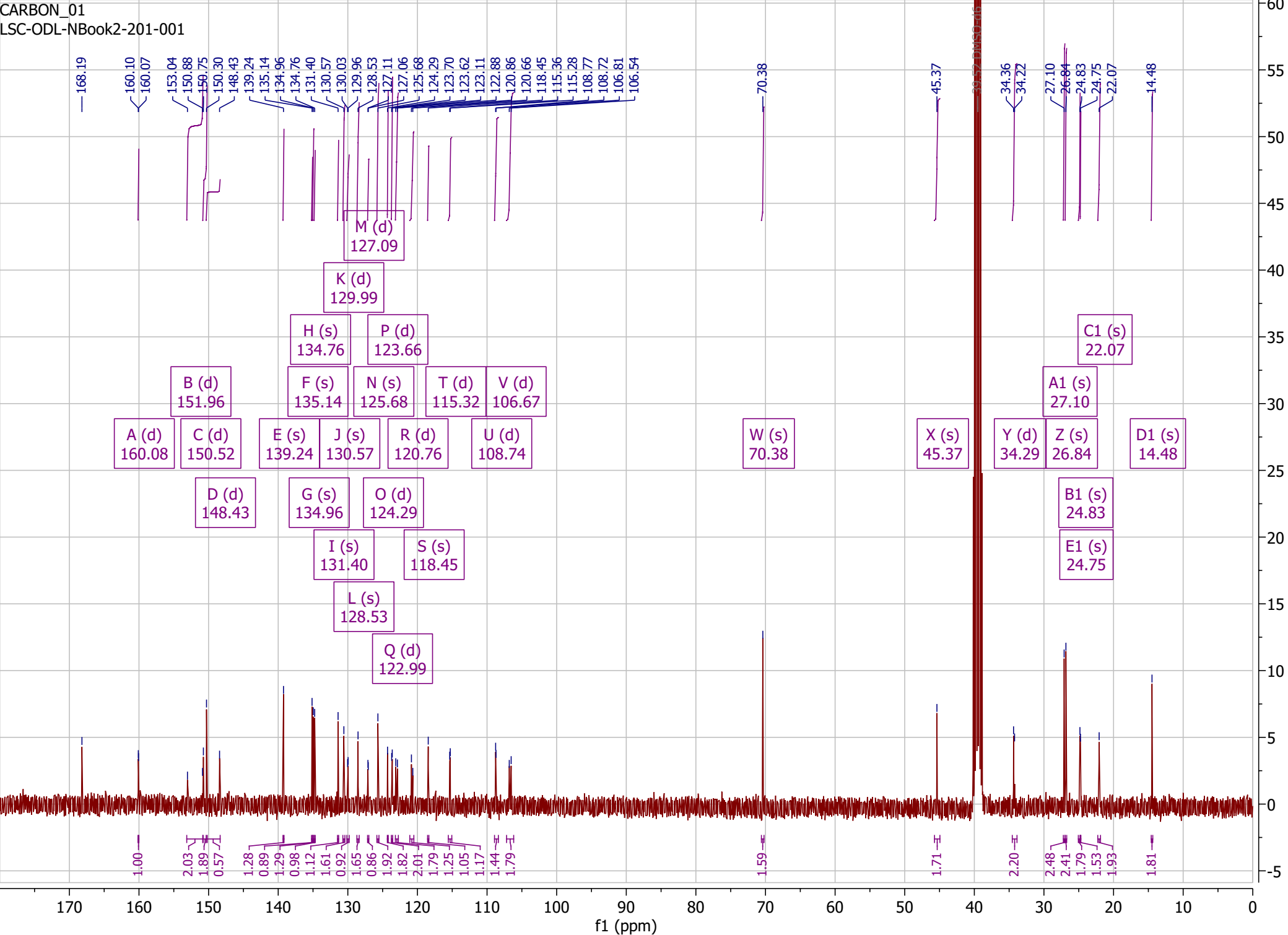




FLUORINE_Q1
LSC-ODL-NB



CARBON_01
LSC-ODL-NBook2-201-001



Omar Lopez

NAT003

National Cancer Institute at Frederick

1050 Boyles St Bld 322

Frederick, Maryland 21702

Sample #: LSC-OLD-NBOOK2-201-001		Test #: 1	Received: 12/09/2020	Completed: 12/10/2020
C : 65.81 %	H : 5.48 %	N : 9.59 %		
<i>Physicochemical Tests</i>	Karl Fischer : 1.98 %			
Sample #: LSC-OLD-NBOOK2-201-001		Test #: 2	Received: 12/09/2020	Completed: 12/10/2020
C : 65.85 %	H : 5.61 %	N : 9.55 %		
<i>Physicochemical Tests</i>	Karl Fischer : 2.07 %			