


Name:	Siponimod-D11
Lot#:	GR-18-029
Test Date:	01/25/2023 (re-test date: 01/25/2028)
CAS No.:	1230487-00-9 (unlabelled)
MF:	C ₂₉ H ₂₄ D ₁₁ F ₃ N ₂ O ₃
MW:	527.70
Appearance:	Off-White Solid
Purity:	97.7% by HPLC (average of two sample preparations); 99% atom D
¹H-NMR:	Conforms (contains a trace of AcOH and DCM)
MS-ESI (-)	Conforms (shows peak at m/z = 526.32 [M-H] ⁻)
Storage	Store at 0-5°C in a dry place away from direct sunlight

Approved by:

Date: 01/28/2023



Viorica Rusu, QC/QA Manager

GR-18-029
STANDARD PHOSPHORUS PARAMETERS

7.66
7.62
7.60
7.41
7.38
7.37
7.25
7.24

5.73

5.20

3.53

3.36

3.17

2.63

2.61

2.48

2.18

1.89

1.14
1.12
1.11

Siponimod-D11
1H-NMR, 600 MHz
Solvent: DMSO-D6
Lot No.: GR-18-029

0.99
1.01
1.01
0.99
1.00
1.00

0.15

2.02

2.20

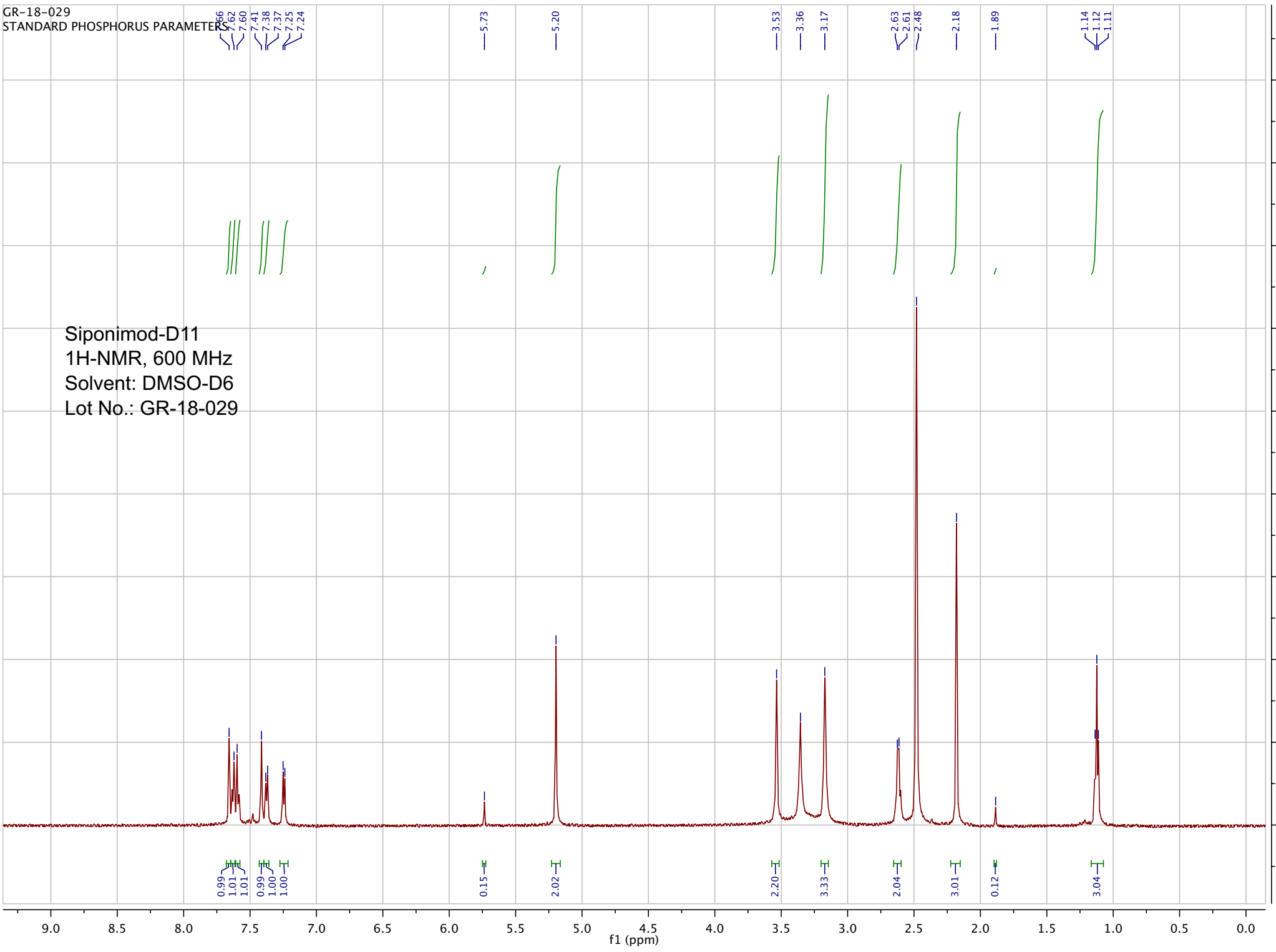
3.33

2.04

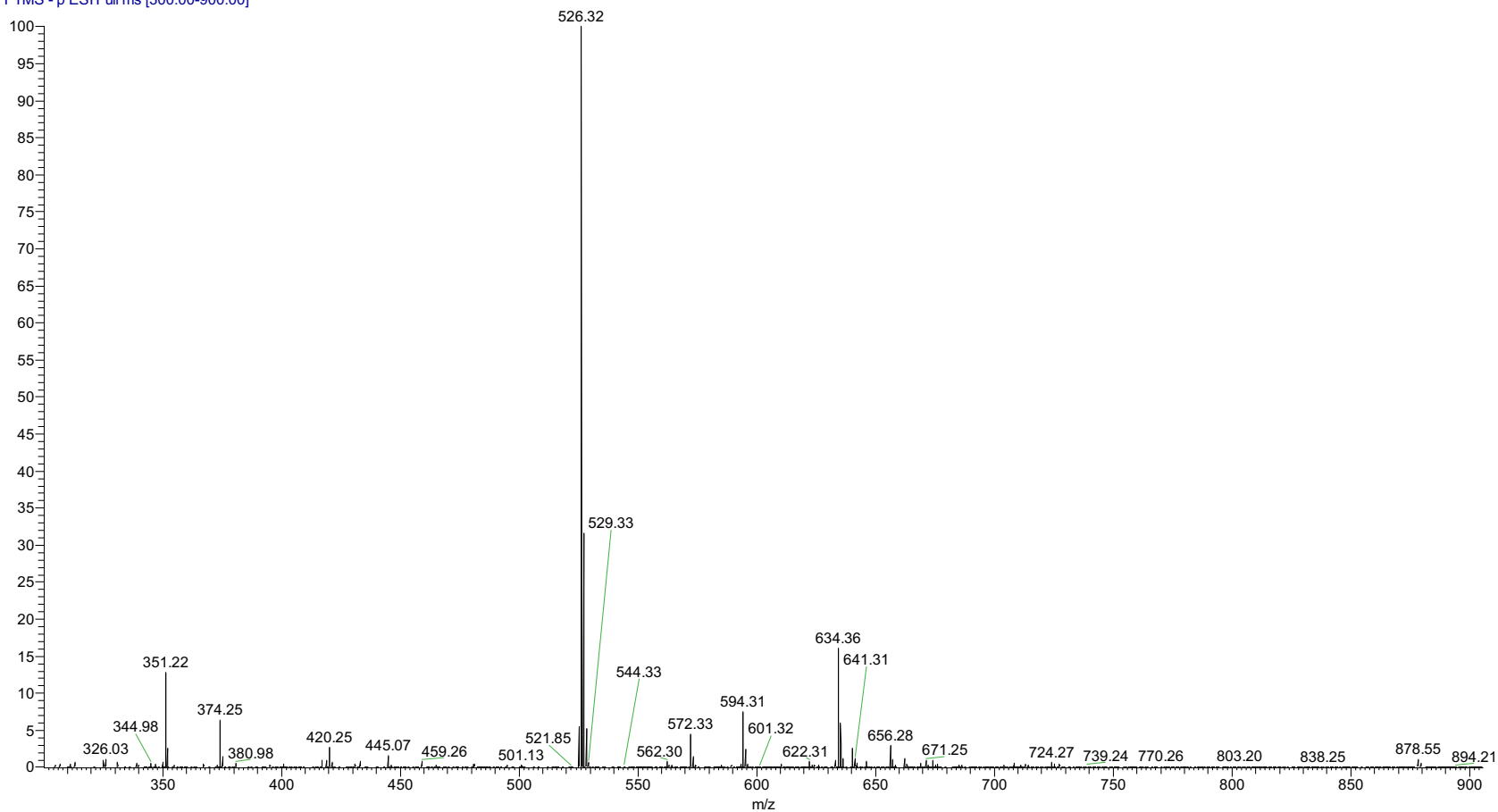
3.01

0.12

3.04

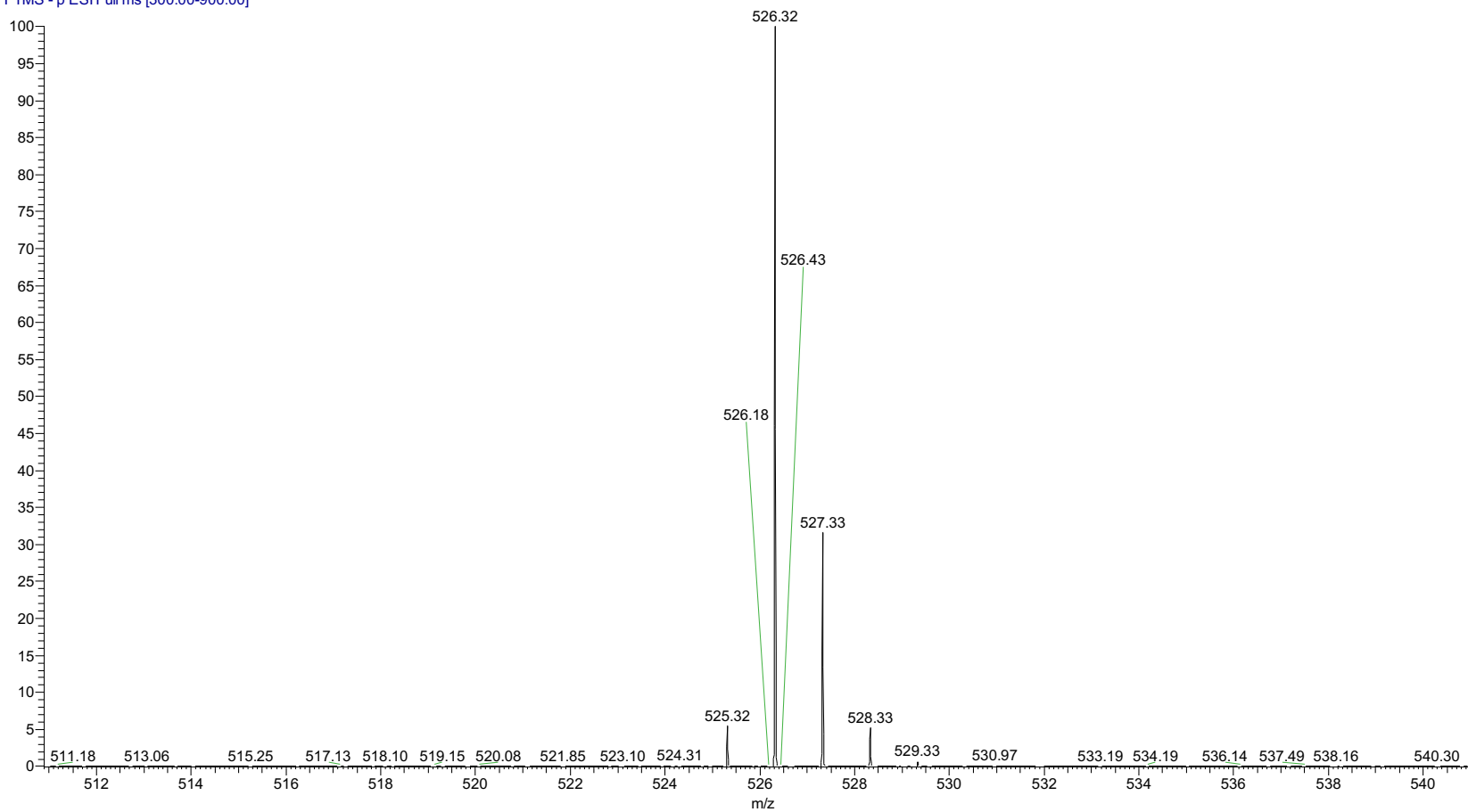


pp_2023_007_GR_18_029_2 #515-562 RT: 1.46-1.58 AV: 48 NL: 3.
T: FTMS - p ESI Full ms [300.00-900.00]



ESI-MS spectrum for sample GR_18_029. Shows monoisotopic [M-H]- mass.

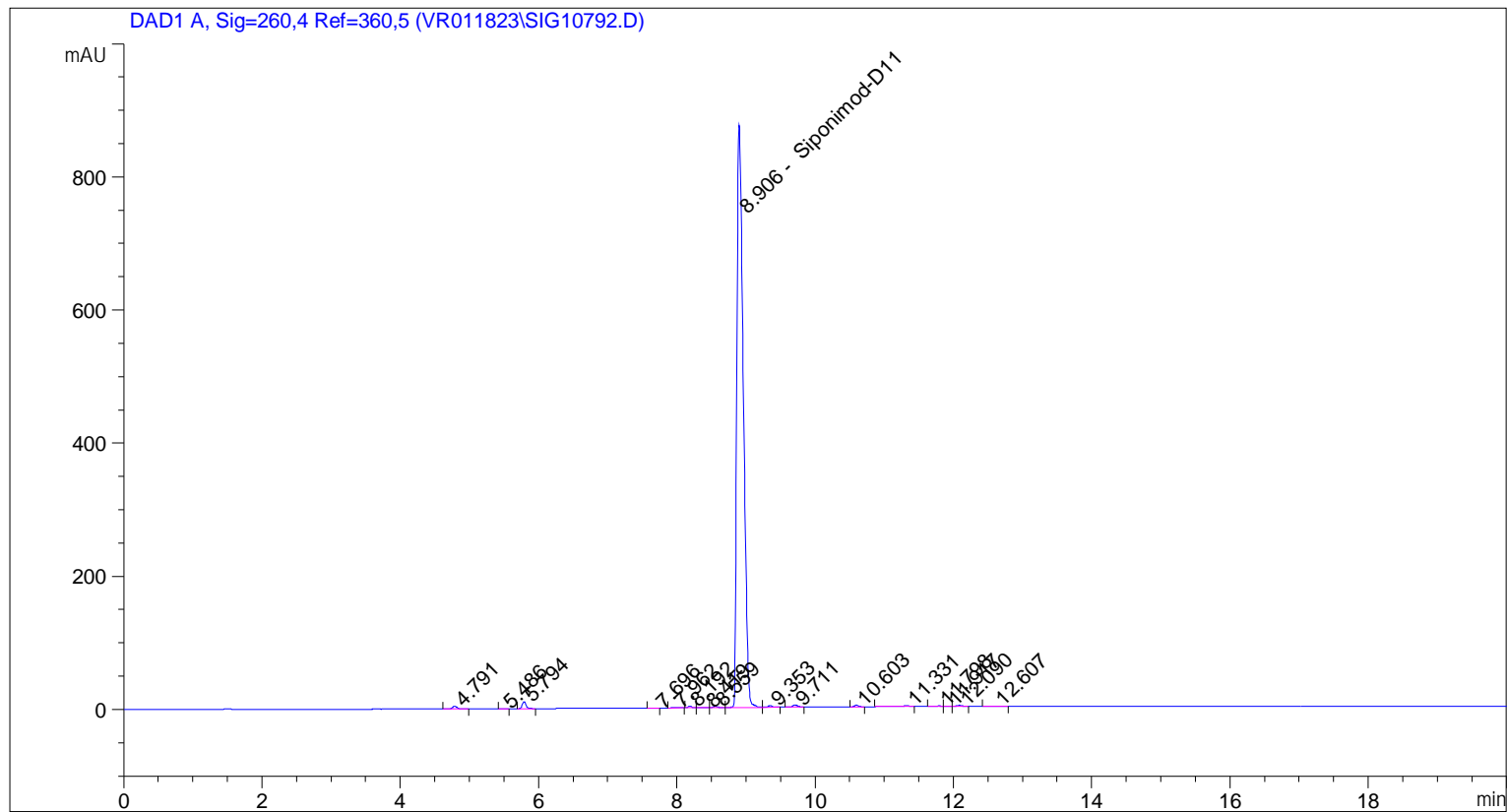
pp_2023_007_GR_18_029_2 #515-562 RT: 1.46-1.58 AV: 48 NL: 3.
T: FTMS - p ESI Full ms [300.00-900.00]



Expansion of above in requested mass range. Chart below shows relative ion intensities.

```

=====
Acq. Operator   : vrusu
Acq. Instrument : Instrument 1                Location : Vial 58
Injection Date  : 1/25/2023 9:13:41 AM
                                           Inj Volume : 5.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\VR012523_029.M
Last changed    : 1/25/2023 9:12:37 AM by vrusu
Analysis Method : C:\CHEM32\1\METHODS\VR012523_029PM.M
Last changed    : 1/25/2023 10:28:21 AM by vrusu
  
```



=====
 Area Percent Report
 =====

```

Sorted By           :      Signal
Calib. Data Modified :      1/25/2023 10:27:44 AM
Multiplier:         :      1.0000
Dilution:           :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 A, Sig=260,4 Ref=360,5

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	4.791	BB	0.0630	17.90421	0.3086	?
2	5.486	BB	0.0682	8.14536e-1	0.0140	?
3	5.794	BB	0.0597	39.51351	0.6811	?
4	7.696	BB	0.0702	6.52091e-1	0.0112	?
5	7.962	BB	0.0675	1.95919	0.0338	?
6	8.192	BB	0.0582	8.66429	0.1493	?
7	8.419	BV	0.0785	1.58106	0.0273	?
8	8.559	VB	0.0667	8.77481	0.1512	?

Sample Name: GR-18-029

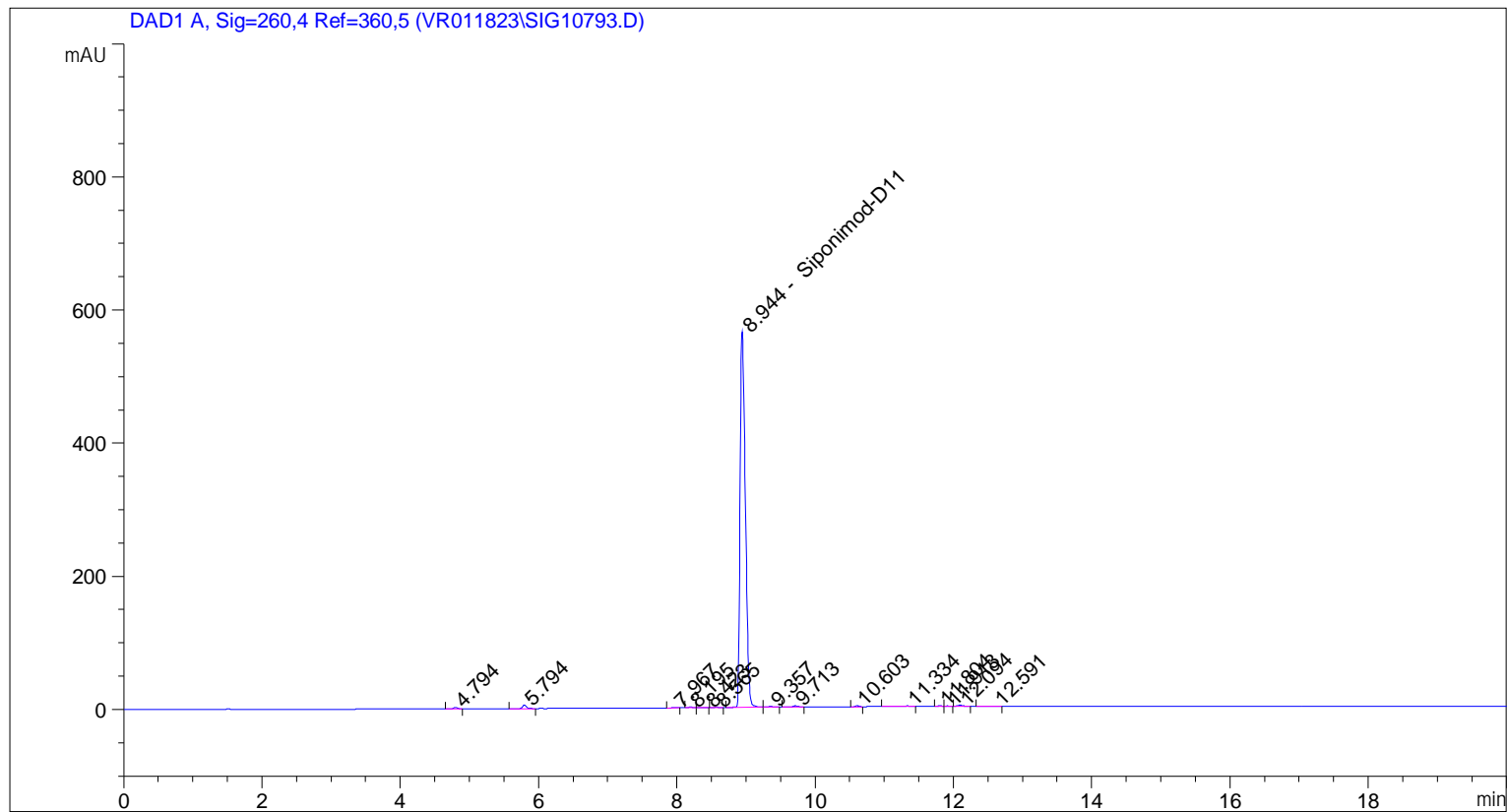
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
9	8.906	BV	0.1015	5667.78906	97.6923	Siponimod-D11
10	9.353	VB	0.0647	12.03735	0.2075	?
11	9.711	BB	0.0795	16.17199	0.2787	?
12	10.603	BB	0.0564	8.84596	0.1525	?
13	11.331	BB	0.0949	5.30857	0.0915	?
14	11.798	BV	0.0751	1.85067	0.0319	?
15	11.917	VV	0.0667	1.22022	0.0210	?
16	12.090	VB	0.0701	5.93800	0.1023	?
17	12.607	BB	0.1129	2.64730	0.0456	?

Totals : 5801.67281

=====
*** End of Report ***

```

=====
Acq. Operator   : vrusu
Acq. Instrument : Instrument 1                Location : Vial 59
Injection Date  : 1/25/2023 9:38:44 AM
                                           Inj Volume : 5.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\VR012523_029.M
Last changed    : 1/25/2023 9:37:46 AM by vrusu
Analysis Method : C:\CHEM32\1\METHODS\VR012523_029PM.M
Last changed    : 1/25/2023 10:28:21 AM by vrusu
  
```



=====
 Area Percent Report
 =====

```

Sorted By           :      Signal
Calib. Data Modified :      1/25/2023 10:27:44 AM
Multiplier:         :      1.0000
Dilution:           :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 A, Sig=260,4 Ref=360,5

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	4.794	BB	0.0647	9.38975	0.3059	?
2	5.794	BB	0.0605	21.34108	0.6953	?
3	7.967	BB	0.0631	9.12476e-1	0.0297	?
4	8.195	BB	0.0587	4.66207	0.1519	?
5	8.423	BB	0.0794	6.20355e-1	0.0202	?
6	8.565	BB	0.0675	3.12037	0.1017	?
7	8.944	BB	0.0820	2995.66235	97.5994	Siponimod-D11
8	9.357	BB	0.0554	5.14741	0.1677	?

Sample Name: GR-18-029 spl-2

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
9	9.713	BB	0.0799	8.73112	0.2845	?
10	10.603	BB	0.0573	4.88025	0.1590	?
11	11.334	BB	0.1003	3.29219	0.1073	?
12	11.804	BV	0.0579	1.12059	0.0365	?
13	11.913	VB	0.0652	9.00203e-1	0.0293	?
14	12.094	BB	0.0680	6.23421	0.2031	?
15	12.591	BB	0.1712	3.33128	0.1085	?

Totals : 3069.34572

=====
*** End of Report ***