

<b>Name:</b>	<b>21-Dehydro Flunisolide</b> (mixture of aldehyde and hydrate forms)
<b>Lot#:</b>	GR-17-161
<b>Test Date:</b>	06/20/2022 (re-test date: 06/20/2027)
<b>CAS No.:</b>	1188271-69-3
<b>MF:</b>	C <sub>24</sub> H <sub>29</sub> FO <sub>6</sub> , C <sub>24</sub> H <sub>31</sub> FO <sub>7</sub> (hydrate form)
<b>MW:</b>	432.48, 450.50 (hydrate form)
<b>Appearance:</b>	Yellow solid
<b>Purity:</b>	97.9% by HPLC (average of two sample preparations), (corresponds to the hydrate form)
<b><sup>1</sup>H-NMR:</b>	Conforms (shows a trace of ACN) (NMR spectra in DMSO-D <sub>6</sub> and CDCl <sub>3</sub> show a ~1:1 mixture of aldehyde and hydrate forms)
<b>MS-ESI (+)</b>	Conforms (shows peaks at m/z = 413.20 [M-HF+H] <sup>+</sup> , 433.2 [M+H] <sup>+</sup> , 451.2 [M+H <sub>2</sub> O+H] <sup>+</sup> and 473.2 [M+H <sub>2</sub> O+Na] <sup>+</sup> )
<b>Storage</b>	Store at -18°C in a dry place away from direct sunlight

Approved by:

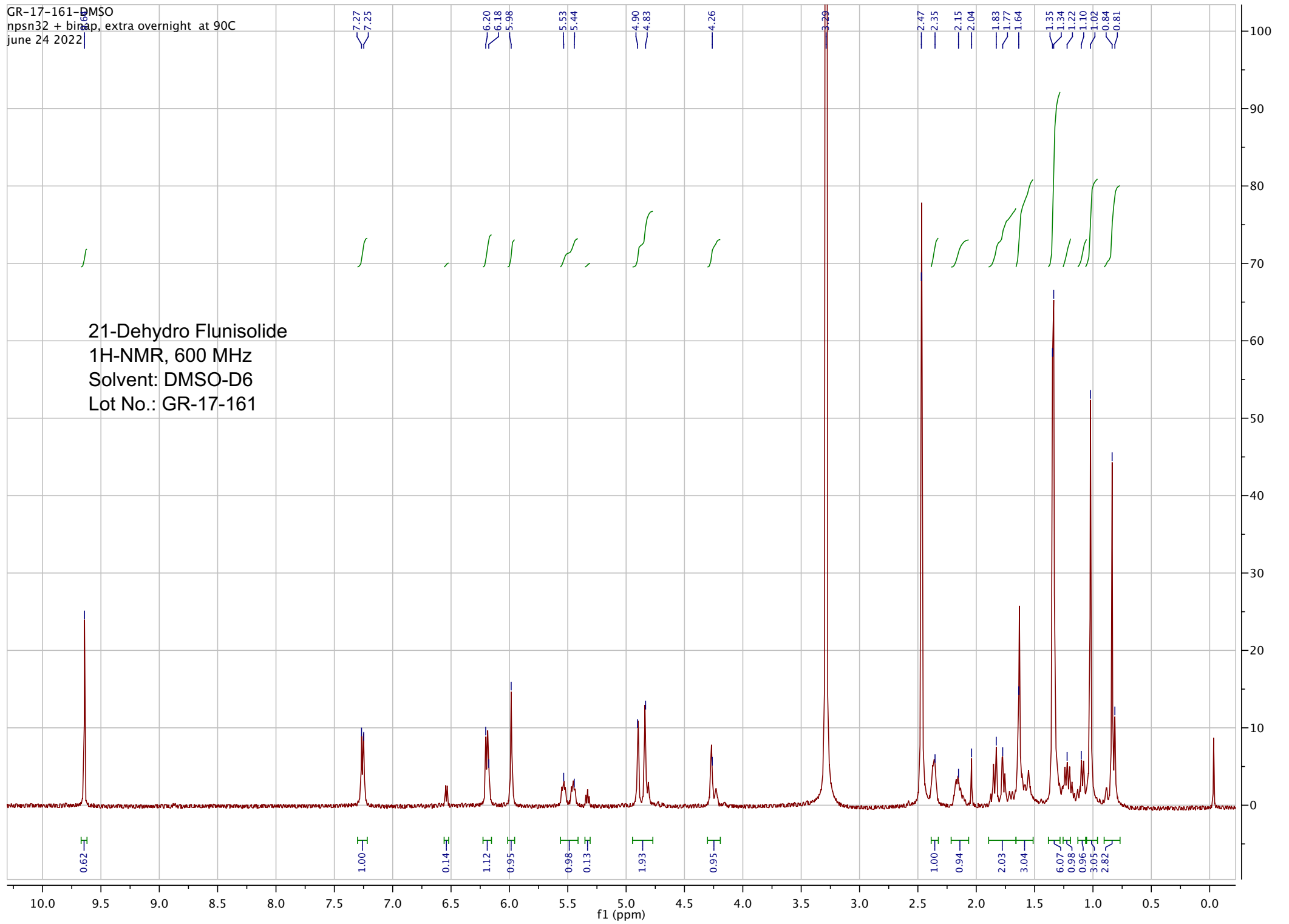
Date: 06/24/2022



Viorica Rusu, QC/QA Manager

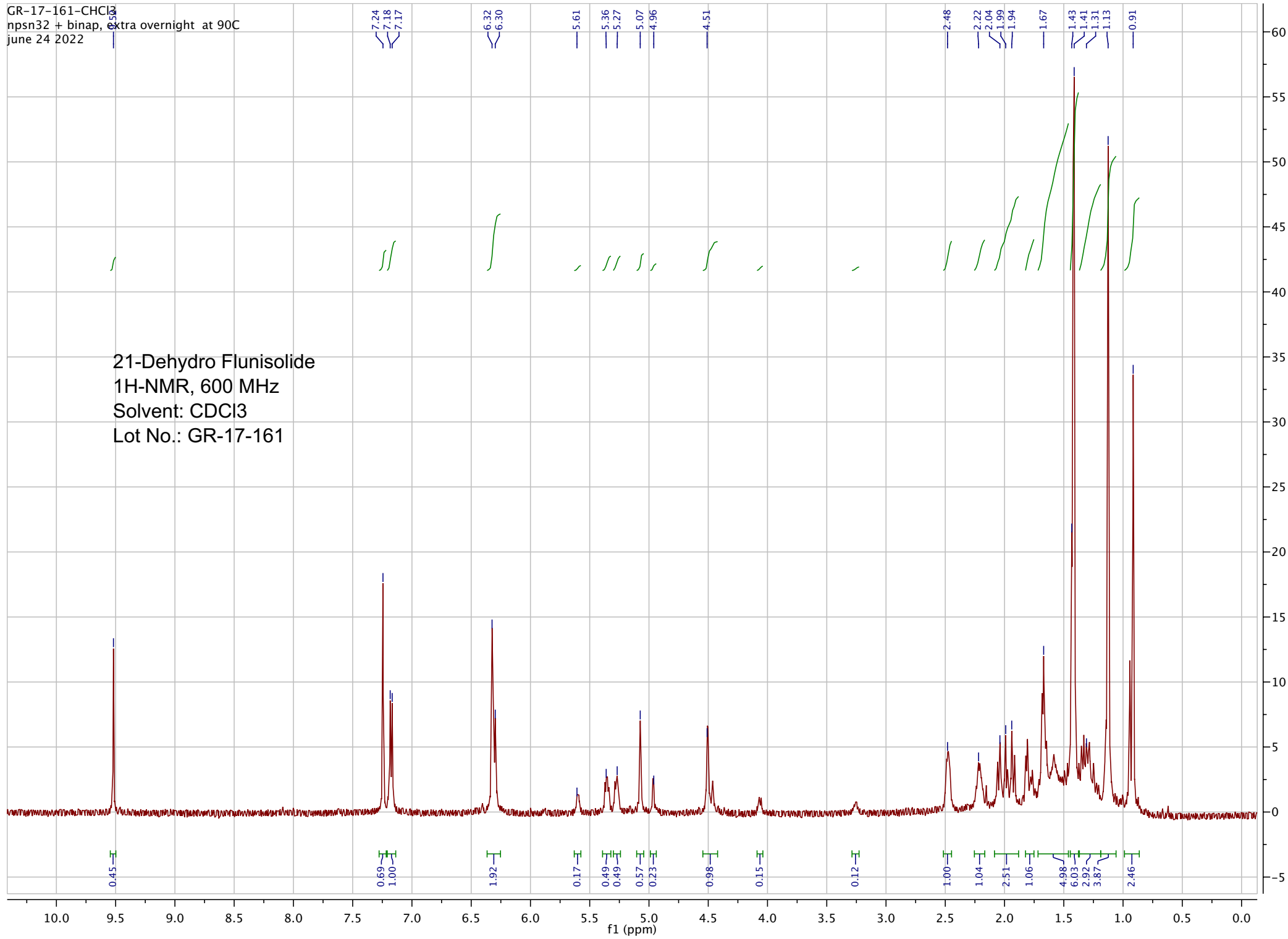
GR-17-161-DMSO  
npsn32 + binap, extra overnight at 90C  
june 24 2022

21-Dehydro Flunisolide  
1H-NMR, 600 MHz  
Solvent: DMSO-D6  
Lot No.: GR-17-161

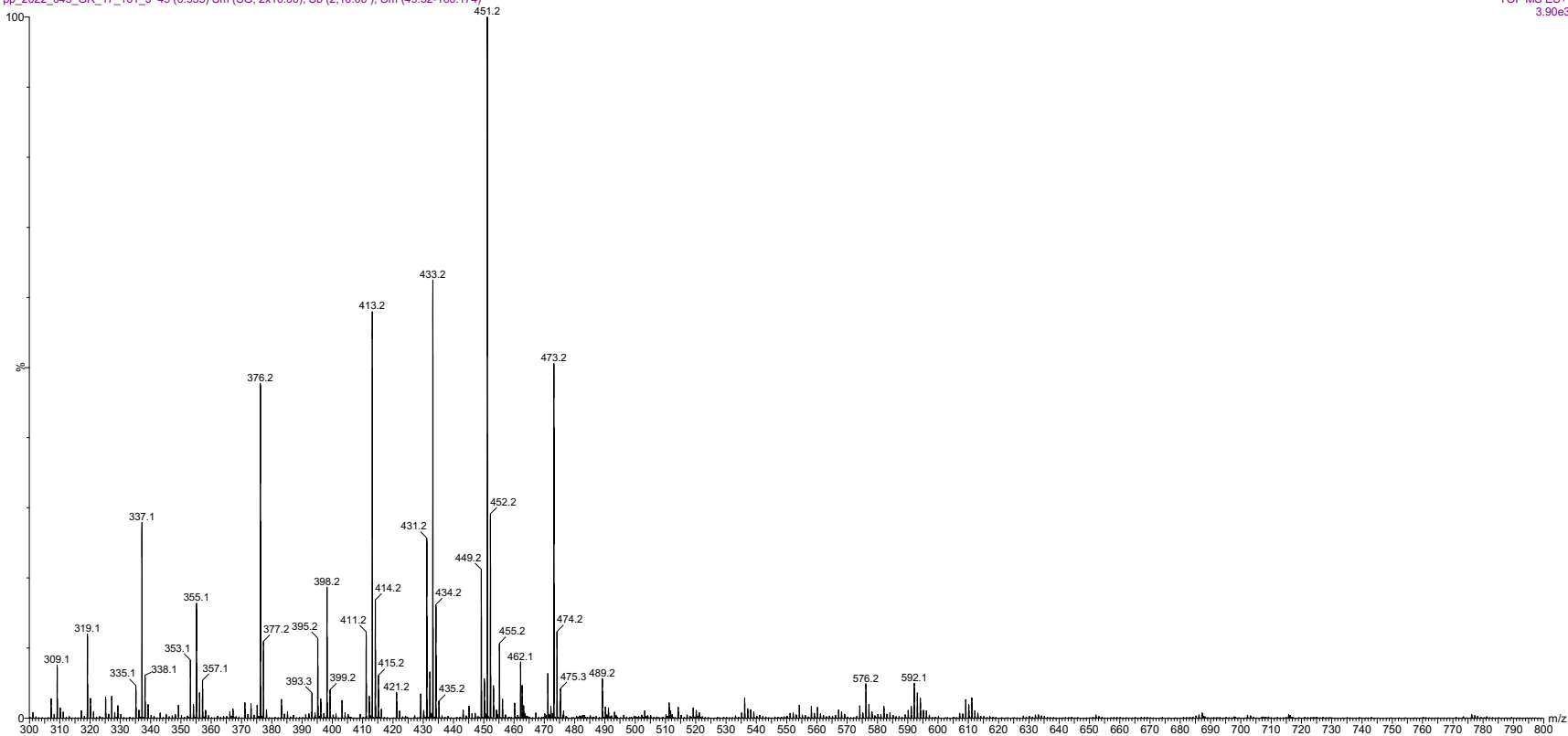


GR-17-161-CHCl3  
npsn32 + binap, extra overnight at 90C  
june 24 2022

21-Dehydro Flunisolid  
1H-NMR, 600 MHz  
Solvent: CDCl3  
Lot No.: GR-17-161



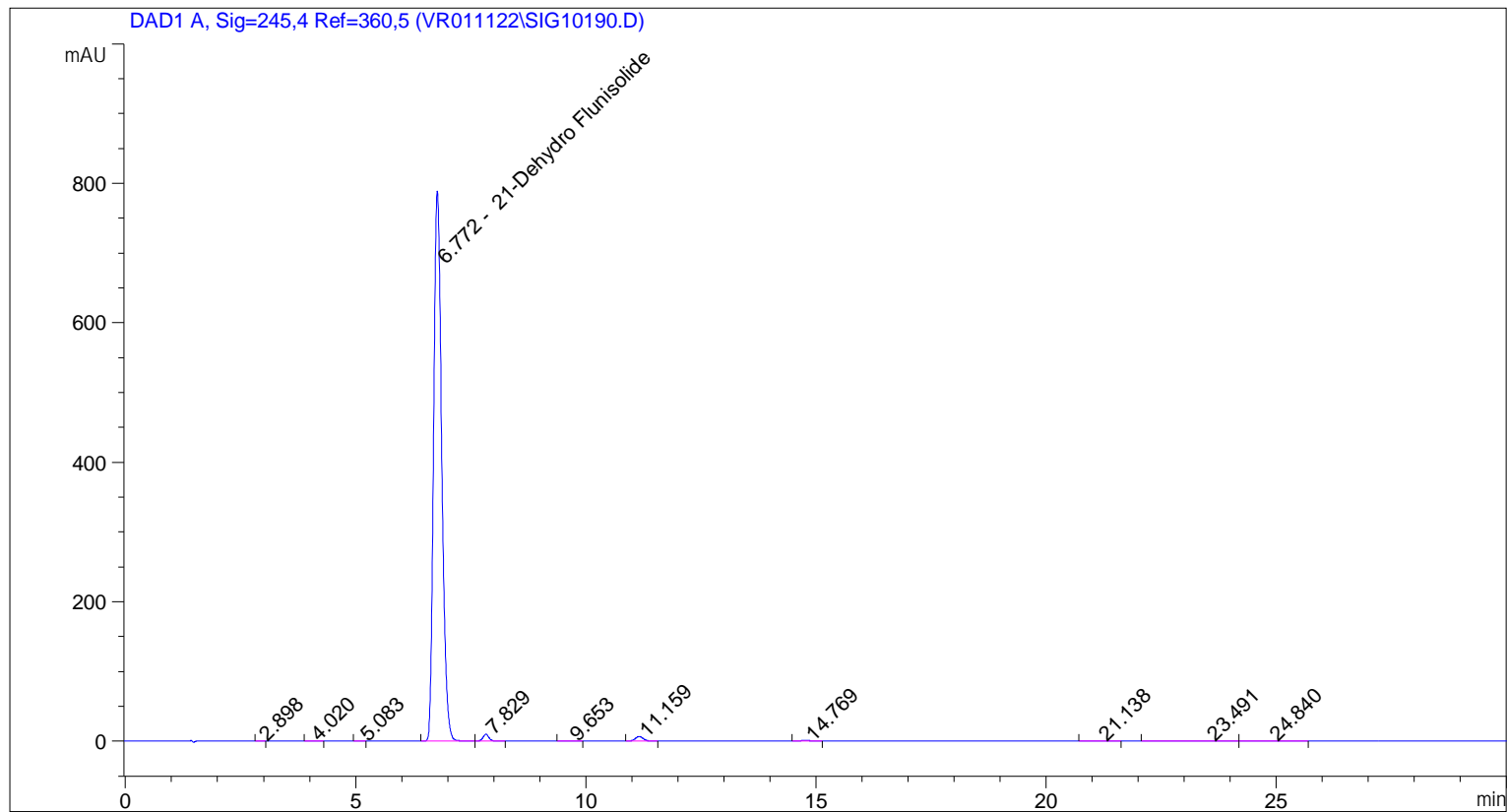
pp\_2022\_045\_GR\_17\_161\_3 49 (0.935) Sm (SG, 2x10.00); Sb (2,10.00); Cm (49:52-166:174)



ESI-MS spectrum for sample GR-17-161. Shows monoisotopic  $[M+H]^+$  mass.

```

=====
Acq. Operator   : vrusu
Acq. Instrument : Instrument 1                Location : Vial 68
Injection Date  : 6/20/2022 2:08:38 PM
                                           Inj Volume : 5.0 µl
Acq. Method     : C:\CHEM32\1\METHODS\VR062022_161.M
Last changed    : 6/20/2022 2:07:29 PM by vrusu
Analysis Method : C:\CHEM32\1\METHODS\VR062022_161PM.M
Last changed    : 6/20/2022 3:32:52 PM by vrusu
  
```



Area Percent Report

```

Sorted By           : Signal
Calib. Data Modified : 6/20/2022 3:31:00 PM
Multiplier:         : 1.0000
Dilution:           : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 A, Sig=245,4 Ref=360,5

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	2.898	BB	0.0702	2.08588	0.0213	?
2	4.020	BB	0.1278	1.58997	0.0162	?
3	5.083	BB	0.0973	2.18325	0.0223	?
4	6.772	BB	0.1880	9597.09766	97.8212	21-Dehydro Flunisolide
5	7.829	BB	0.1367	86.02991	0.8769	?
6	9.653	BB	0.2142	5.18765	0.0529	?
7	11.159	BB	0.1850	81.26106	0.8283	?
8	14.769	BB	0.2111	15.17496	0.1547	?

Sample Name: GR-17-161

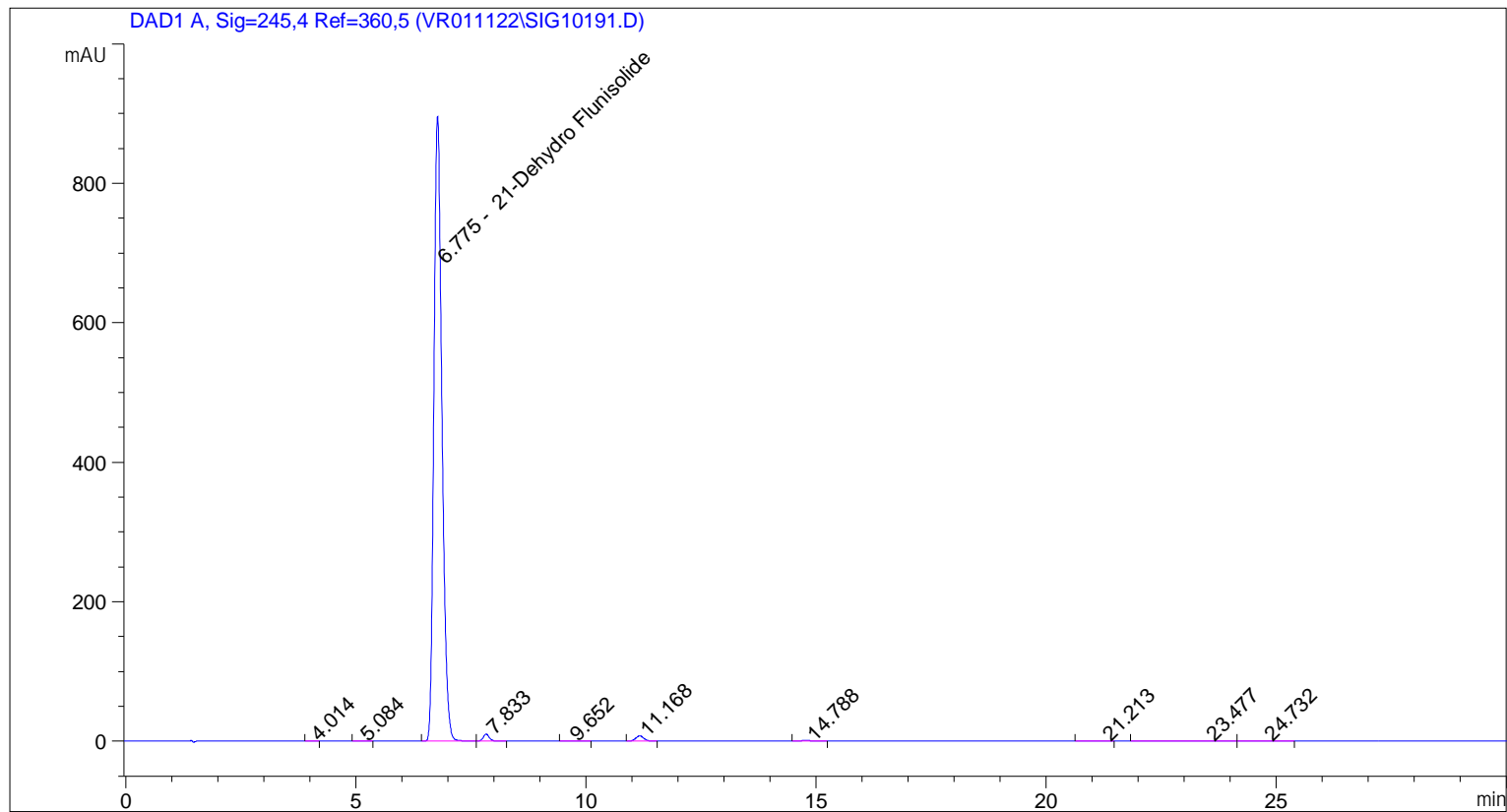
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
9	21.138	BB	0.3849	5.04990	0.0515	?
10	23.491	BV	0.9647	9.67255	0.0986	?
11	24.840	VB	0.4181	5.52778	0.0563	?

Totals : 9810.86056

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

```

=====
Acq. Operator   : vrusu
Acq. Instrument : Instrument 1                Location : Vial 69
Injection Date  : 6/20/2022 2:41:22 PM
                                           Inj Volume : 5.0 µl
Acq. Method    : C:\CHEM32\1\METHODS\VR062022_161.M
Last changed   : 6/20/2022 2:40:18 PM by vrusu
Analysis Method: C:\CHEM32\1\METHODS\VR062022_161PM.M
Last changed   : 6/20/2022 3:32:52 PM by vrusu
  
```



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

```

Sorted By      : Signal
Calib. Data Modified : 6/20/2022 3:31:00 PM
Multiplier:    : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 A, Sig=245,4 Ref=360,5

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	4.014	BB	0.1226	1.79784	0.0161	?
2	5.084	BB	0.1111	3.19252	0.0286	?
3	6.775	BV	0.1905	1.09424e4	97.9058	21-Dehydro Flunisolide
4	7.833	VB	0.1397	93.33167	0.8351	?
5	9.652	BB	0.2415	5.73273	0.0513	?
6	11.168	BB	0.1873	93.52608	0.8368	?
7	14.788	BB	0.2028	14.36548	0.1285	?
8	21.213	BB	0.4647	5.33361	0.0477	?

Sample Name: GR-17-161 spl-2

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
9	23.477	BV	0.8594	10.36413	0.0927	?
10	24.732	VB	0.4712	6.41082	0.0574	?

Totals : 1.11764e4

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