

Name: 21-Dehydro Budesonide (aldehyde and hydrate forms; mixture of isomers)

Lot#: GR-15-029

Test Date: 07/17/2020 (re-test date: 07/17/2025)

CAS No.: 8523463-5

MF: C₂₅H₃₂O₆, C₂₄H₃₄O₇ (hydrate form)

MW: 428.52, 446.54 (hydrate form)

Appearance: Yellow Solid

Purity: 96.7% by HPLC (sum of two isomers; corresponds to the hydrate form)

¹H-NMR: Conforms (NMR in DMSO-D₆ and CDCl₃ shows mostly the hydrate form)

MS-ESI (+) Conforms (shows peaks at m/z = 429.22 [M+H]⁺ and 447.23 [M+H₂O+H]⁺ shows ~1:3 mixture of aldehyde and hydrate forms)

Note: In solid state and in solutions of aprotic solvents 21-Dehydro Budesonide exists as a mixture of aldehyde and hydrate forms; in the solvents containing water, it exists almost exclusively in the hydrate form)

Approved by:

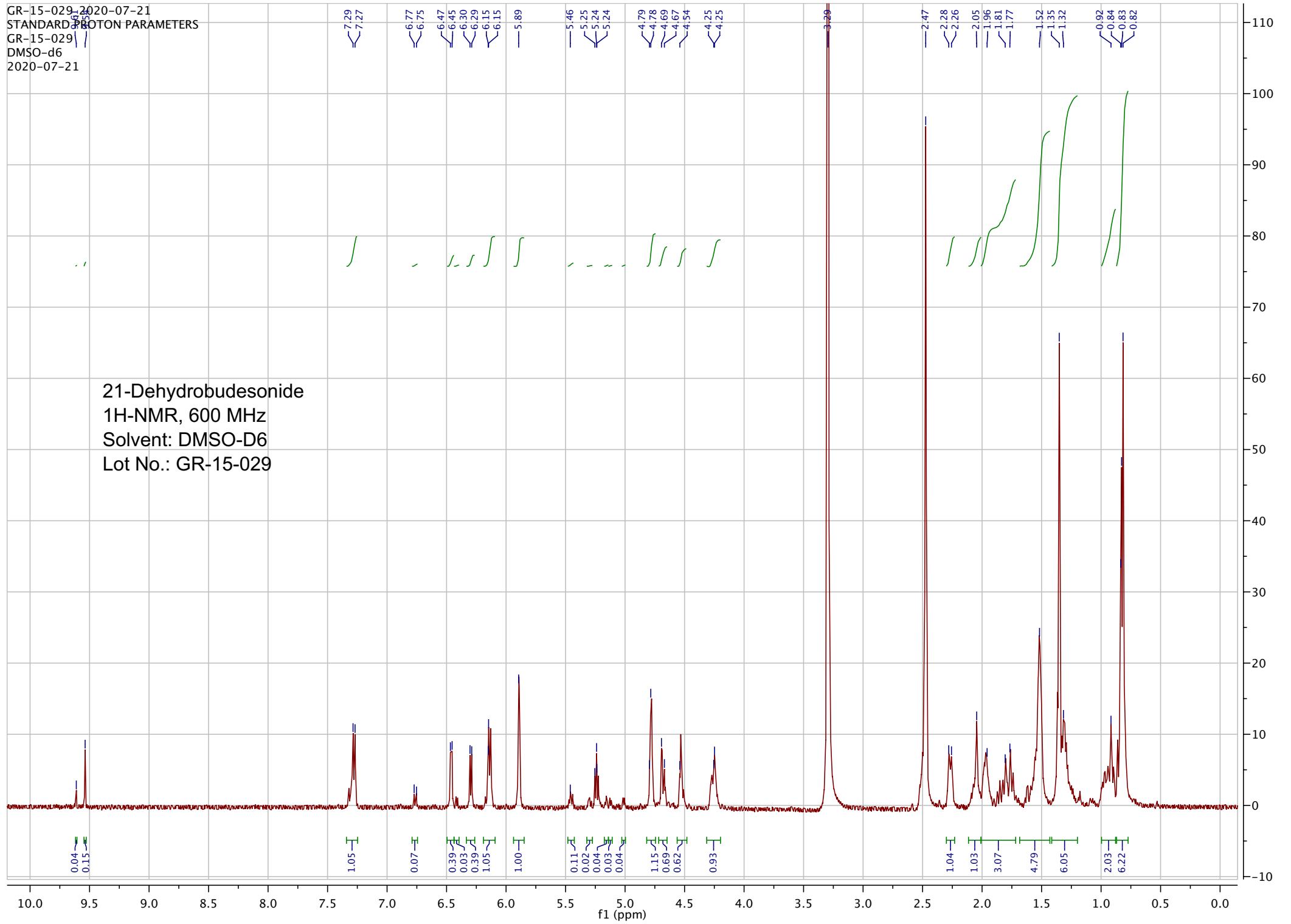
Date: 07/22/2020



Viorica Rusu, QC/QA Manager

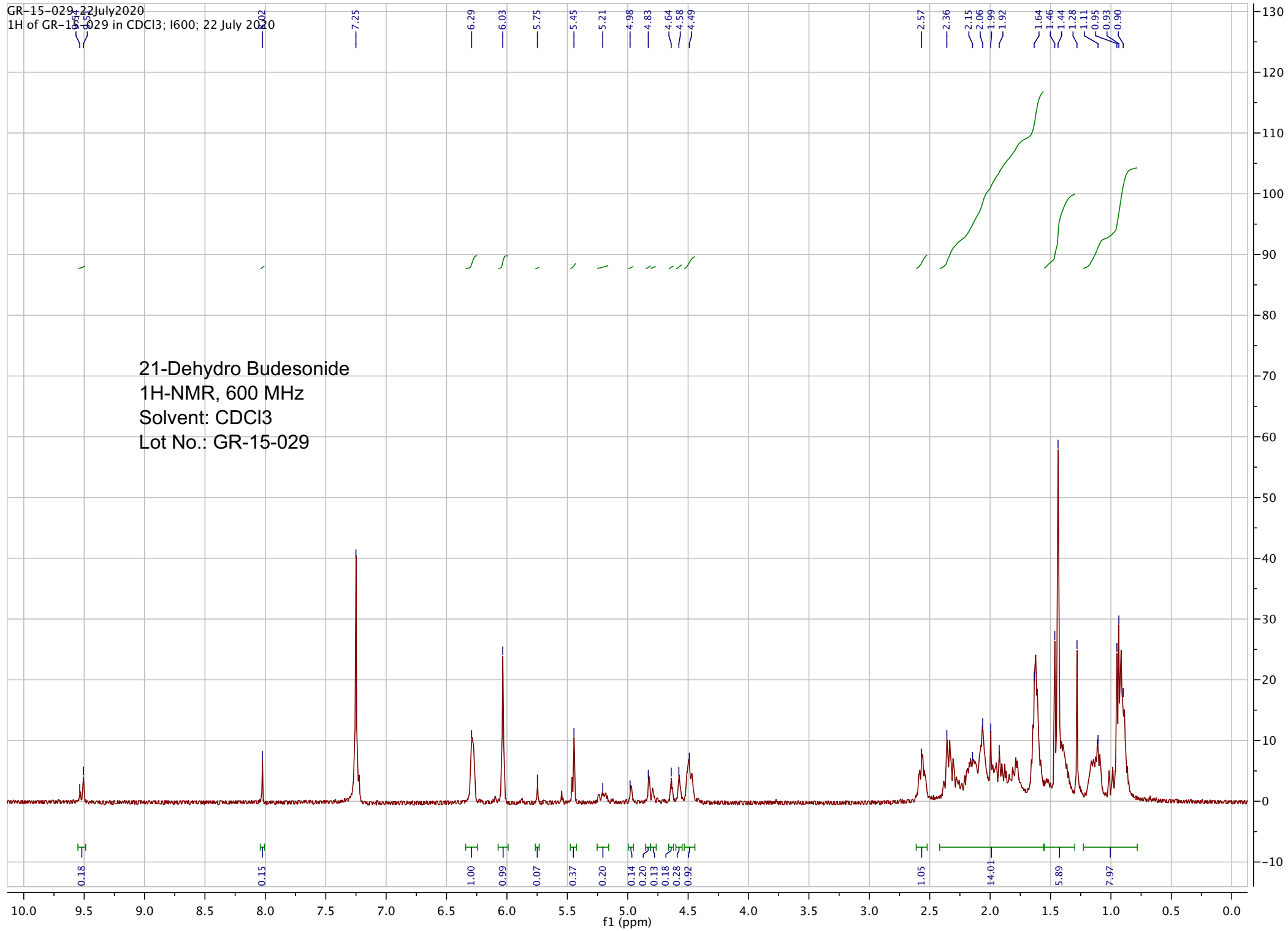
GR-15-029, 2020-07-21
STANDARD PROTON PARAMETERS
GR-15-029
DMSO-d6
2020-07-21

21-Dehydrobudesonide
1H-NMR, 600 MHz
Solvent: DMSO-D6
Lot No.: GR-15-029

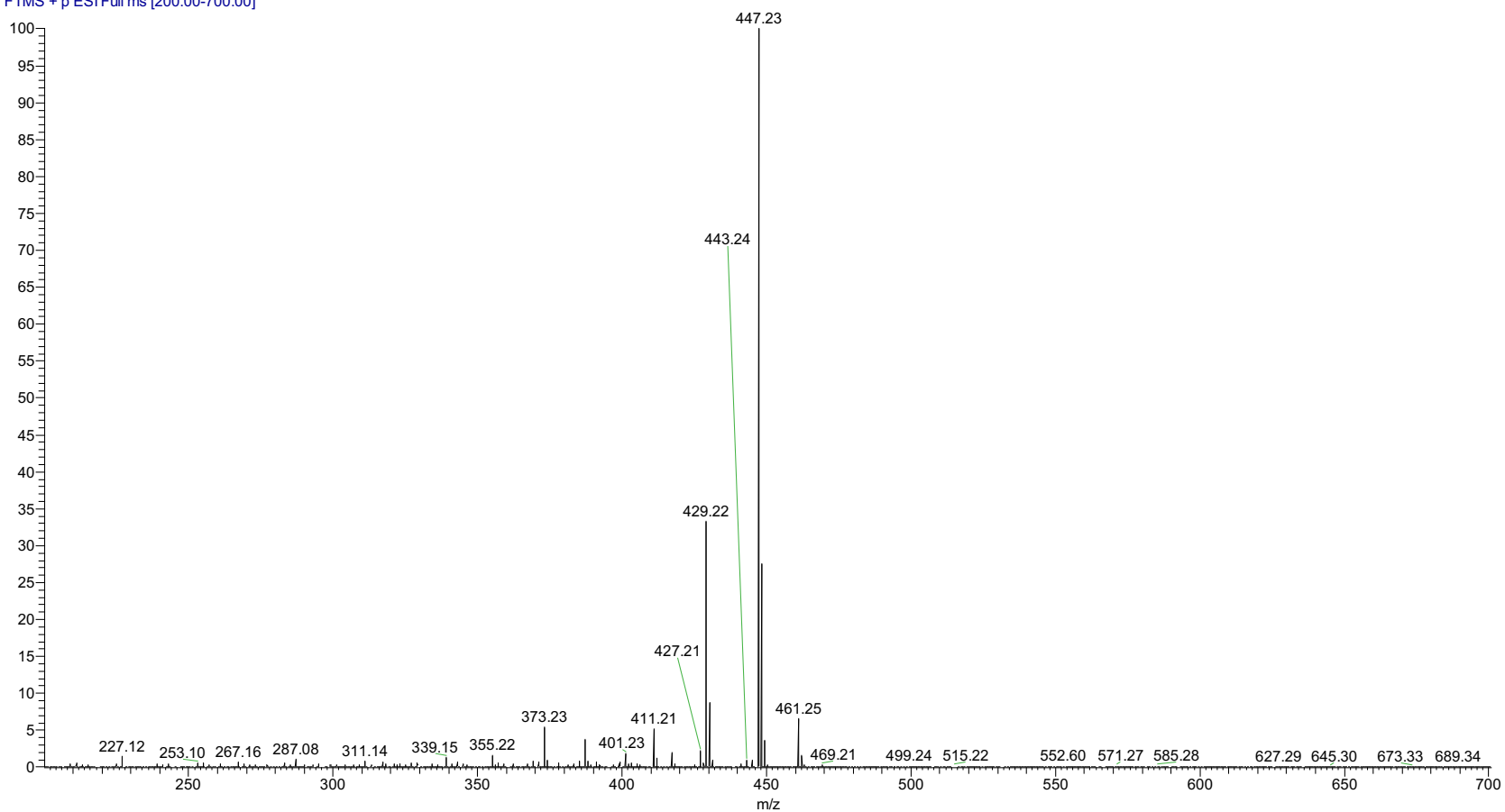


GR-15-029, 22 July 2020
1H of GR-15-029 in CDCl3; 1600; 22 July 2020

21-Dehydro Budesonide
1H-NMR, 600 MHz
Solvent: CDCl3
Lot No.: GR-15-029



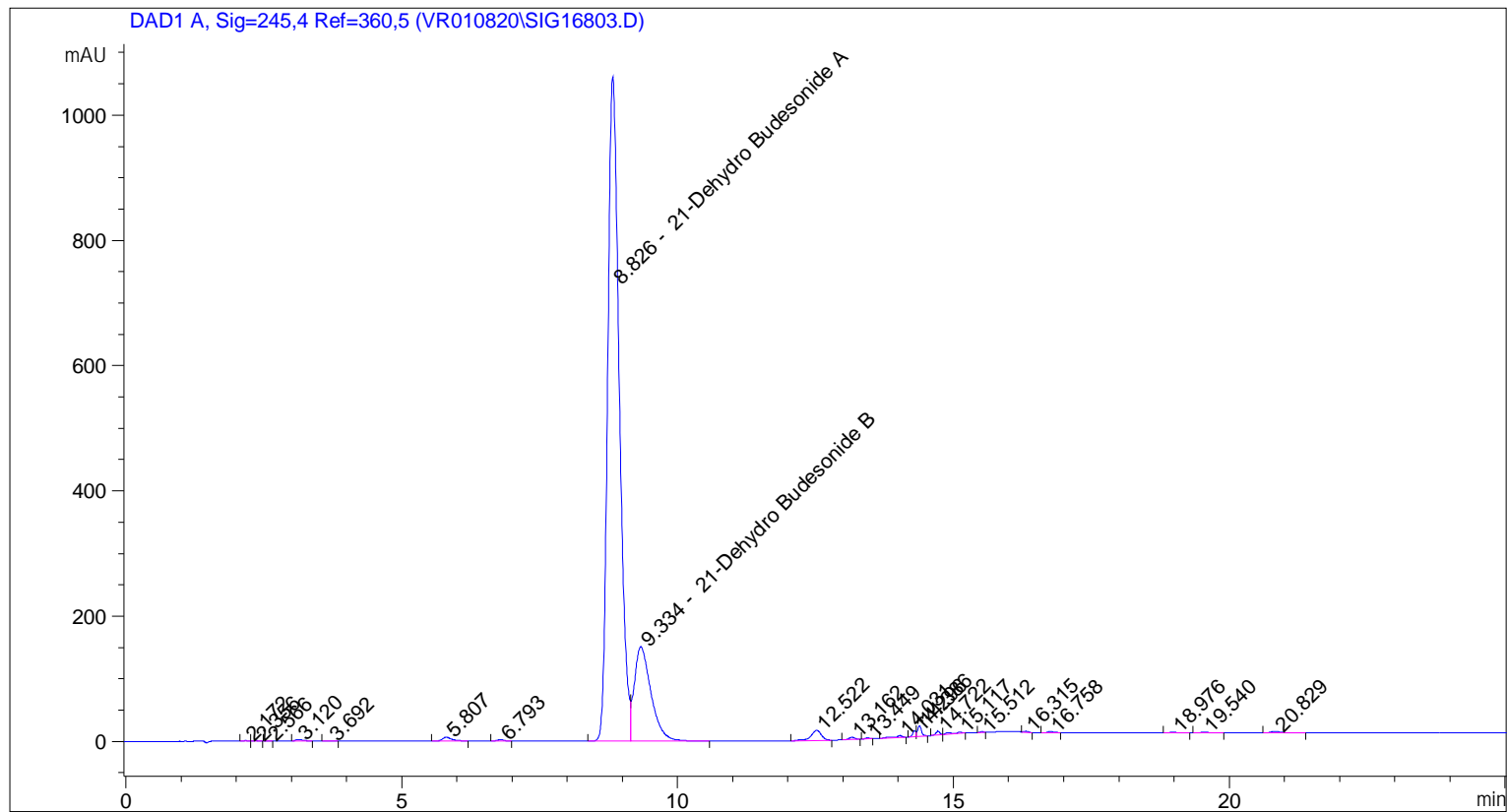
pp_2020_015_GR15029_003 #209-212 RT: 1.50-1.52 AV: 4 NL: 7:
T: FTMS + p ESIFull.ms [200.00-700.00]



ESI-MS spectrum for sample GR-15-029. Shows monoisotopic $[M+H]^+$ mass.

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Acq. Operator   : vrusu
Acq. Instrument : Instrument 1                Location : Vial 67
Injection Date  : 7/17/2020 8:46:04 AM
                                           Inj Volume : 10.0 µl
Acq. Method    : C:\CHEM32\1\METHODS\VR071520_029.M
Last changed   : 7/17/2020 8:41:59 AM by vrusu
Analysis Method: C:\CHEM32\1\METHODS\VR071720_029PM.M
Last changed   : 7/17/2020 10:43:41 AM by vrusu
  
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Area Percent Report

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Sorted By           : Signal
Calib. Data Modified : 7/15/2020 1:36:34 PM
Multiplier:         : 1.0000
Dilution:           : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
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Signal 1: DAD1 A, Sig=245,4 Ref=360,5

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	2.172	BV	0.0780	5.09180	0.0263	?
2	2.356	VB	0.0860	5.85702	0.0303	?
3	2.566	BB	0.0702	1.95718	0.0101	?
4	3.120	BB	0.1532	22.50309	0.1163	?
5	3.692	BB	0.1049	3.59402	0.0186	?
6	5.807	BB	0.1853	74.49847	0.3850	?
7	6.793	BB	0.1361	19.90395	0.1029	?
8	8.826	BV	0.2250	1.55402e4	80.3057	21-Dehydro Budesonide A

Sample Name: GR-15-029

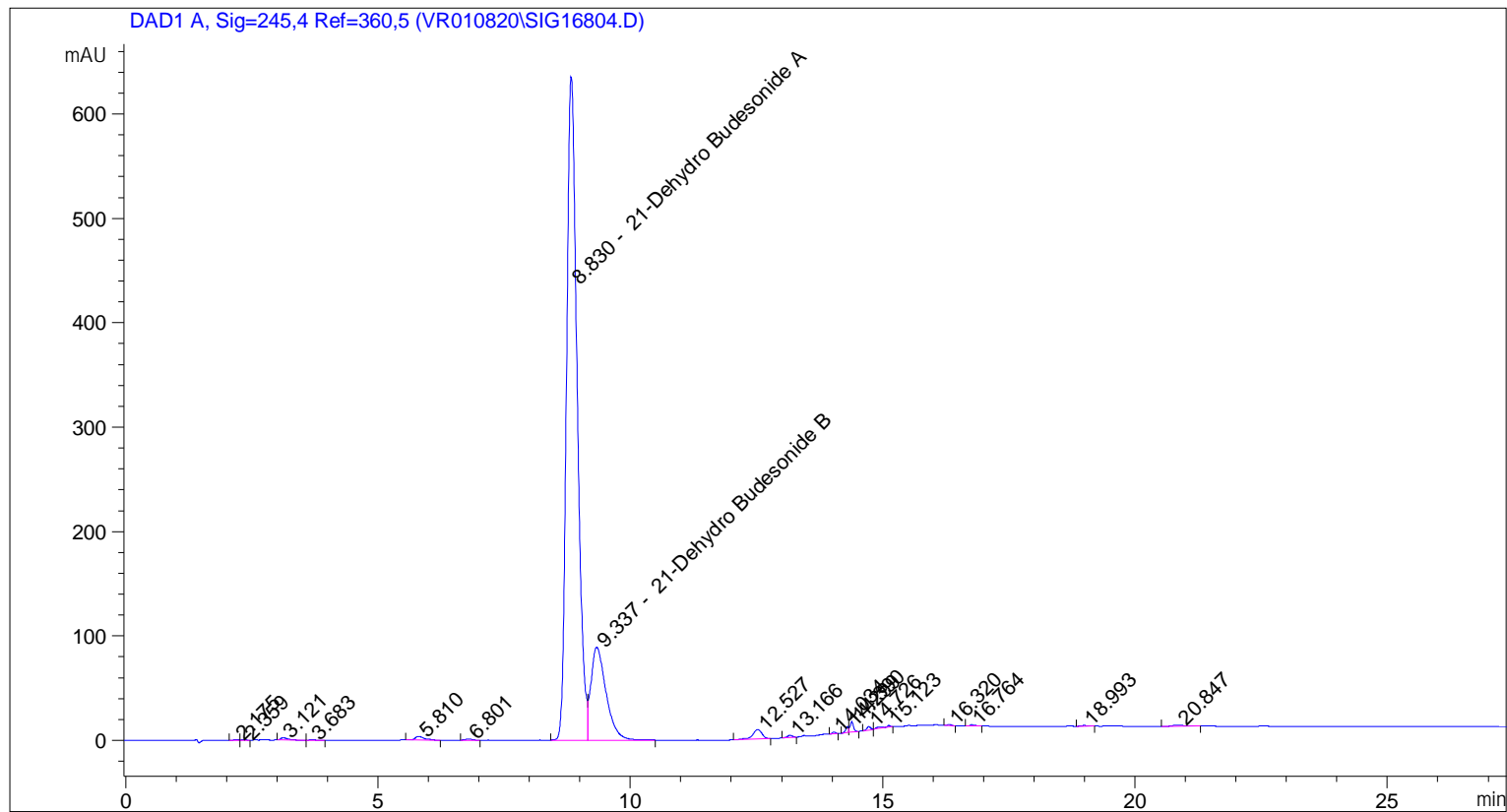
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
9	9.334	VB	0.3117	3144.51807	16.2497	21-Dehydro Budesonide B
10	12.522	BB	0.2012	213.43663	1.1030	?
11	13.162	BB	0.1170	25.34521	0.1310	?
12	13.449	BB	0.0900	7.99867	0.0413	?
13	14.031	BB	0.1153	24.01487	0.1241	?
14	14.296	BV	0.0637	38.47724	0.1988	?
15	14.386	VB	0.0714	80.17386	0.4143	?
16	14.722	BV	0.0764	31.04635	0.1604	?
17	15.117	VB	0.1661	31.27165	0.1616	?
18	15.512	BB	0.0648	4.09930	0.0212	?
19	16.315	BB	0.0809	8.33092	0.0431	?
20	16.758	BB	0.1072	16.47625	0.0851	?
21	18.976	BB	0.1271	8.95329	0.0463	?
22	19.540	BB	0.1829	10.69794	0.0553	?
23	20.829	BB	0.2082	32.85007	0.1698	?

Totals : 1.93513e4

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*** End of Report ***

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Acq. Operator   : vrusu
Acq. Instrument : Instrument 1                Location : Vial 68
Injection Date  : 7/17/2020 9:19:19 AM
                                           Inj Volume : 10.0 µl
Acq. Method    : C:\CHEM32\1\METHODS\VR071520_029.M
Last changed   : 7/17/2020 9:11:15 AM by vrusu
Analysis Method : C:\CHEM32\1\METHODS\VR071720_029PM.M
Last changed   : 7/17/2020 10:43:41 AM by vrusu
  
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Area Percent Report

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Sorted By      : Signal
Calib. Data Modified : 7/15/2020 1:36:34 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	2.175	BV	0.0798	2.91611	0.0253	?
2	2.359	VB	0.0832	2.95008	0.0256	?
3	3.121	BB	0.1544	21.62209	0.1877	?
4	3.683	BB	0.0995	3.43003	0.0298	?
5	5.810	BB	0.1879	44.95354	0.3903	?
6	6.801	BB	0.1338	10.76698	0.0935	?
7	8.830	BV	0.2247	9293.67871	80.6887	21-Dehydro Budesonide A
8	9.337	VB	0.3133	1854.00977	16.0967	21-Dehydro Budesonide B

Sample Name: GR-15-029

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
9	12.527	BB	0.1949	119.09447	1.0340	?
10	13.166	BB	0.1122	13.80108	0.1198	?
11	14.034	BB	0.0742	7.79252	0.0677	?
12	14.299	BV	0.0641	22.97429	0.1995	?
13	14.390	VB	0.0707	46.63761	0.4049	?
14	14.726	BB	0.0756	16.56851	0.1438	?
15	15.123	BB	0.1449	18.06680	0.1569	?
16	16.320	BB	0.0848	5.73034	0.0498	?
17	16.764	BB	0.1068	9.84316	0.0855	?
18	18.993	BB	0.1189	5.09930	0.0443	?
19	20.847	BB	0.2136	18.00850	0.1564	?

Totals : 1.15179e4

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*** End of Report ***