



# Frontier BioPharm

LLC

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## CERTIFICATE OF ANALYSIS

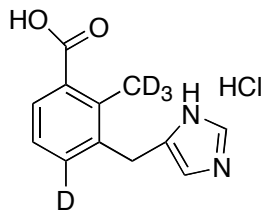
### 3-Carboxydetomidine-*d*<sub>4</sub> HCl

0.100 mg/mL in Methanol (Free base concentration)

Sample Identity: C-5001  
Lot No.: 10280138

Chemical Identity: Chemical Name: 3-Carboxydetomidine-*d*<sub>4</sub> HCl  
Residual Solvents: <0.15% (TGA, <sup>1</sup>H-NMR)  
Solvent: Methanol  
Volume per Ampule: 1 mL  
Storage Condition: Protect from light, freeze  
Chemical Purity: 99.6% by HPLC-UV (220 nm)  
Isotopic Purity: >95%

Analysis Date: January 30, 2014  
Re-analysis Date: January 30, 2017




<u>Purity</u>	<u>Prepared Concentration*</u>	<u>Analyzed Concentration**</u>
99.1%	0.100 mg/mL(as the free base) (±0.003 mg/mL)	0.0.101 mg/mL (±0.003 mg/mL)

Concentrations are reported as the free base. (Hydrochloride salt prepared concentration.0.116 mg/mL)

\* Masses are determined using balances calibrated with NIST traceable weights. The prepared concentration is corrected for residual solvents, chromatographic purity, and the salt counterion mass, and is expressed as the free base concentration.

\*\* Concentration values are determined by independent calibration curves. It is suggested that the prepared concentration value be used.

Approved:   
Rodney L. Eisenberg, Ph.D.

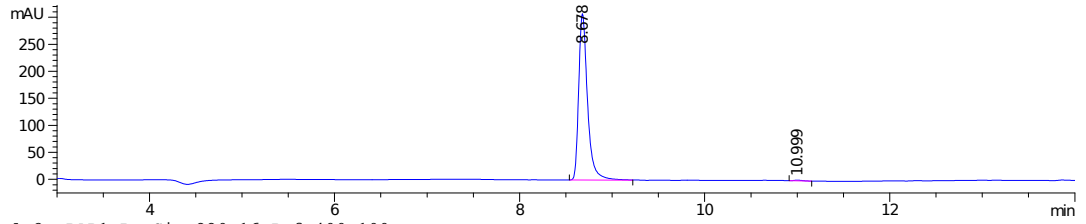
01-30-2014  
Date

Spectral and Physical Data

LCMS Data Column:

HPLC-UV (220 nm)

\*DAD1 B, Sig=220,16 Ref=400,100 (10280424.D)

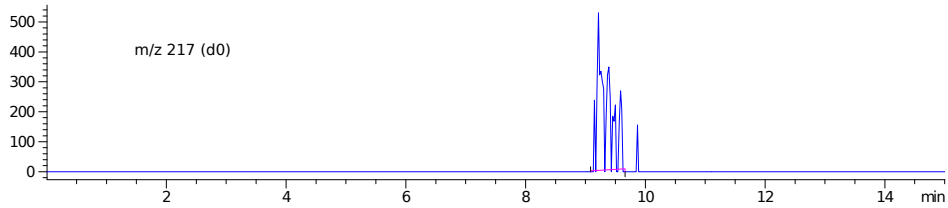
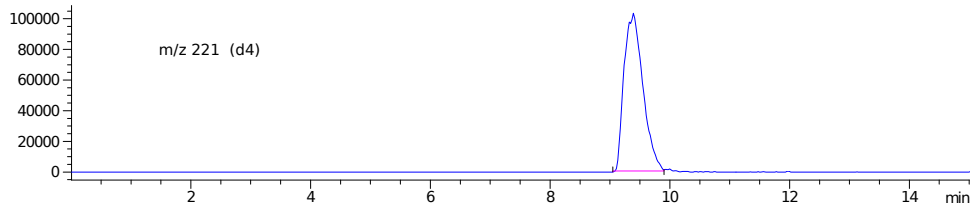
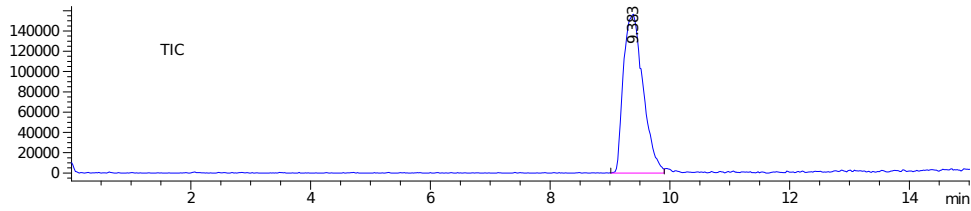


Signal 3: DAD1 B, Sig=220,16 Ref=400,100

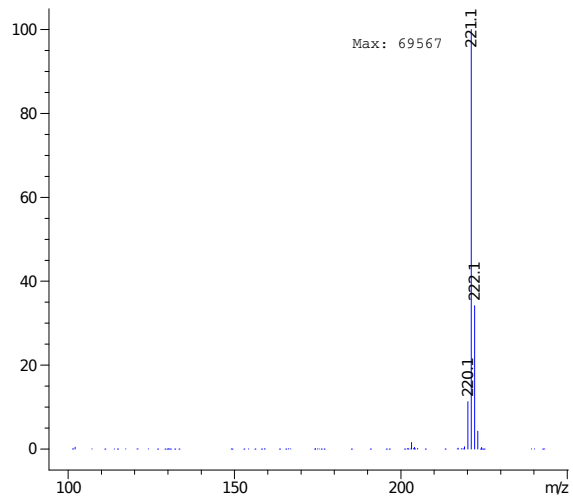
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.678	BB	0.0995	2045.74927	307.79620	99.6545
2	10.999	BB	0.0884	7.09222	1.24525	0.3455
Totals :				2052.84148	309.04145	

Isotope labels

ESI MS positive ion mode.

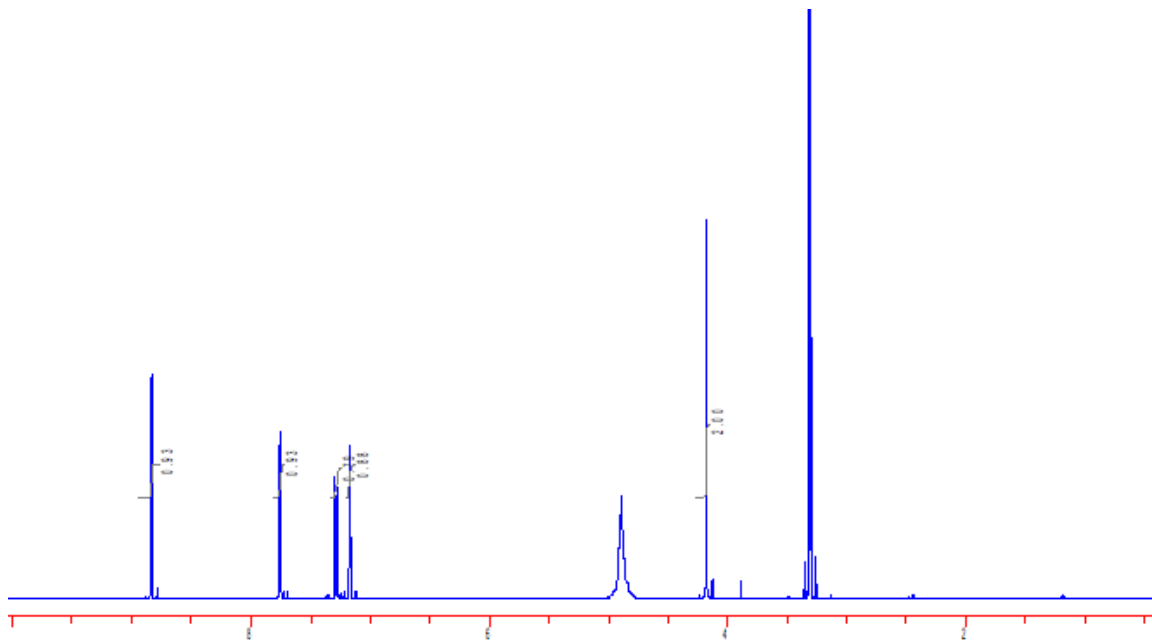


Mass	Abundance
217.0	0.249
218.0	0.249
219.2	0.578
220.1	11.064
221.1	100.000
222.1	32.444
223.0	3.957
224.0	0.405
243.0	0.654



<sup>1</sup>H-NMR (CD<sub>3</sub>OD, 400 MHz)

NUMBER	FROM	TO	VALUE
1	8.95 PPM	8.75 PPM	0.93
2	7.81 PPM	7.72 PPM	0.93
3	7.33 PPM	7.25 PPM	0.79
4	7.20 PPM	7.13 PPM	0.88
5	4.26 PPM	4.14 PPM	2.00



COA History

Version 1.0 Original COA lot 10280138 Analyzed 01-30-2014