



Frontier BioPharm

LLC

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CERTIFICATE OF ANALYSIS

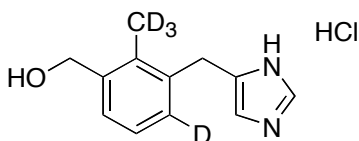
D₄-3-Hydroxydetomidine Hydrochloride

0.100 mg/mL in methanol (free base concentration)

Sample Identity: HD-2001
Lot No.: 1004100905

Chemical Identity: Chemical Name: D₄-3-Hydroxydetomidine HCl
Storage Condition: Protect from light, refrigerate or freeze
Chemical Purity: 98.8%
Residual solvents (TGA): <0.1%
Elemental Analysis: Calculated (%) C: 59.38, H, 7.89, Cl: 14.61, N: 11.54
Found: C: 59.14, H, 7.93, Cl: 14.45, N: 11.41
Melting Point: 181-182 °C

Date of Solution preparation: 7 September, 2010
Analysis Date: 21 August, 2012
Re-analysis Date: 21 August, 2014




Purity	Prepared Concentration*	Analyzed Concentration**
98.8% (As the free base)	0.100 mg/mL (±0.003 mg/mL)	0.099 mg/mL (± 0.003 mg/mL)

Concentrations are reported as the free base. (Hydrochloride salt prepared concentration.0.118 mg/mL)

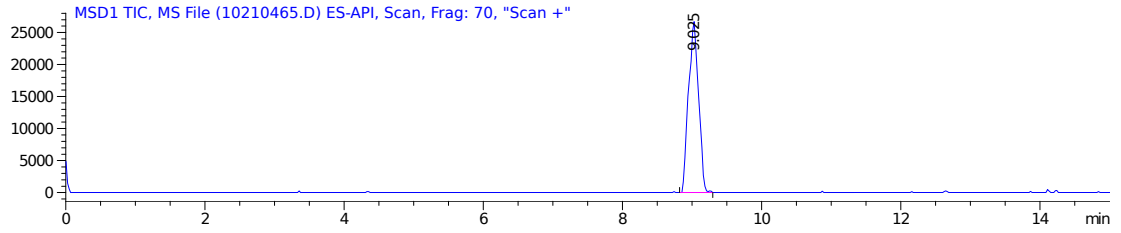
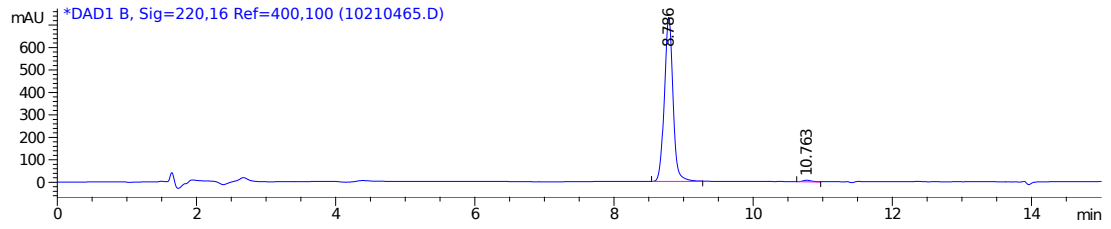
* Masses are determined using balances calibrated with NIST traceable weights. The prepared concentration is corrected for residual solvents, chromatographic purity, and the salt counterion mass, and is expressed as the free base concentration.

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

Approved: 
Rodney L. Eisenberg, Ph.D.
Director

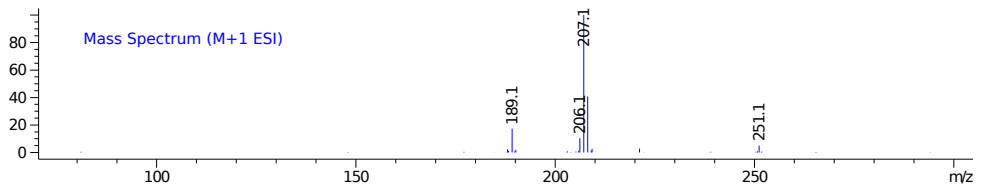
08-21-2012
Date

LCMS data

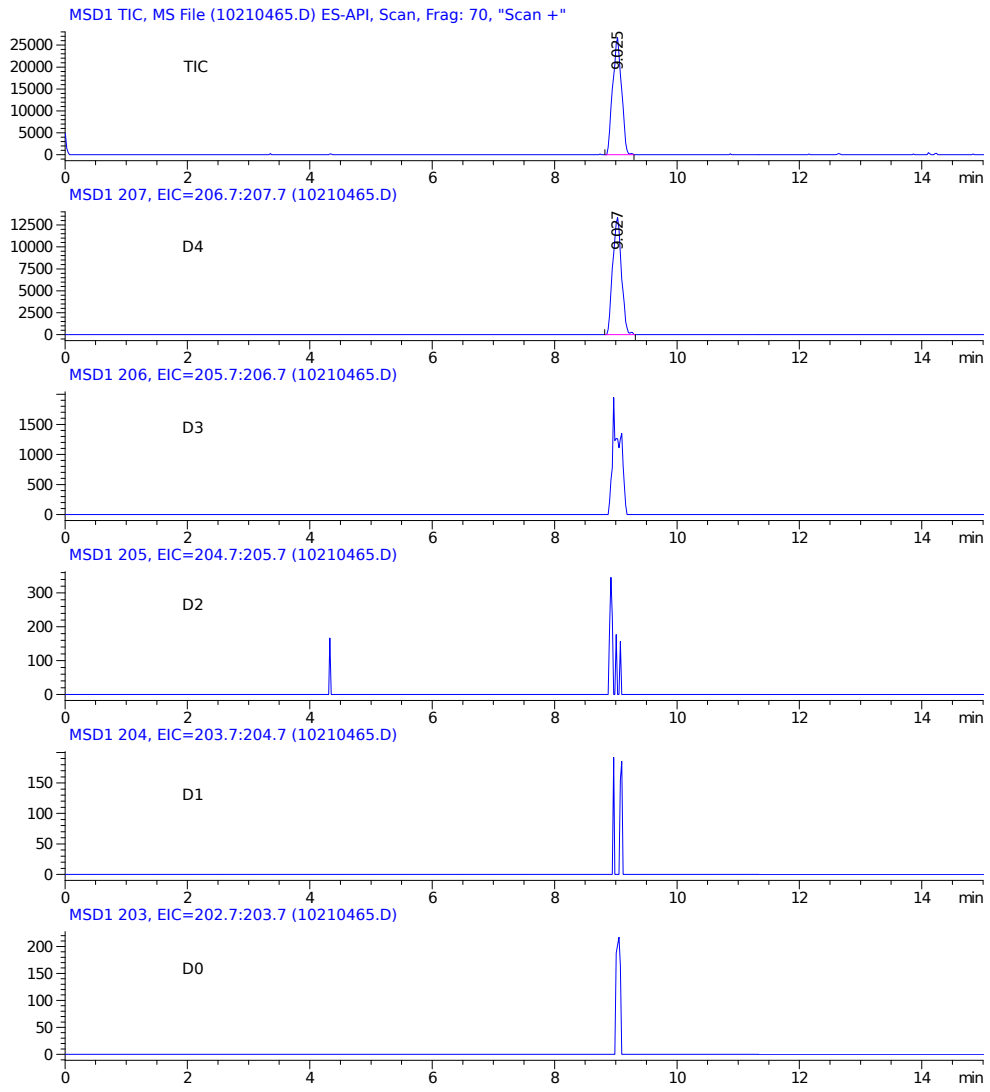


Signal 1: DAD1 B, Sig=220,16 Ref=400,100

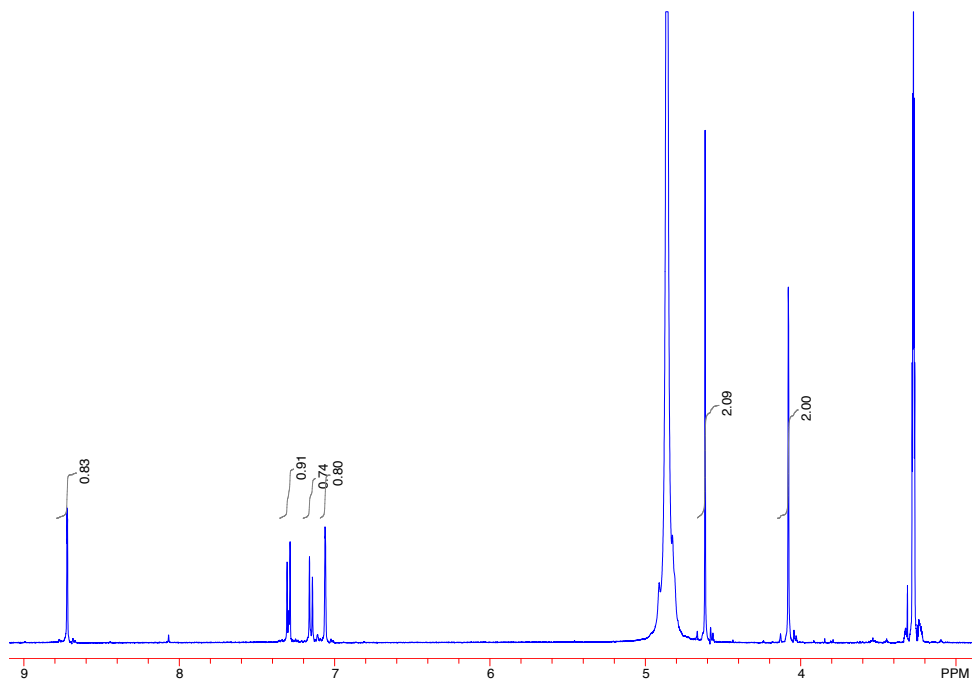
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.786	BB	0.1303	6286.90723	731.20233	98.8066
2	10.763	BY	0.1500	75.93140	7.37946	1.1934
Totals :				6362.83862	738.58179	



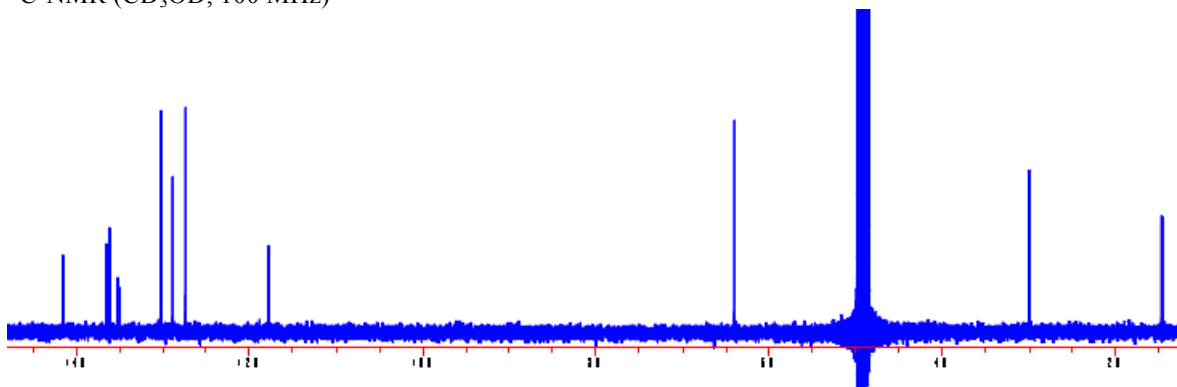
Mass Extracted ion spectra (ESI Positive Ion Mode)



$^1\text{H-NMR}$ (CD_3OD , 400 MHz)



$^{13}\text{C-NMR}$ (CD_3OD , 100 MHz)



LINE	HZ	PPM
1	14221.61	141.483
2	13713.76	136.430
3	13684.53	136.140
4	13591.18	135.211
5	13567.41	134.974
6	13089.31	130.218
7	12944.07	128.773
8	12800.84	127.348
9	11840.14	117.791
10	6428.79	63.956
11	3013.25	29.977
12	1467.99	14.604

COA History

Version 1.0 Original COA Lot No.: 1004100905

Version 1.1 Re-Analyzed solution for purity. August 21, 2012.