BDG SYNTHESIS

Certificate of Analysis

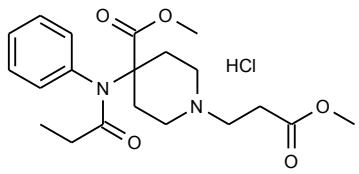
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

Neil Beare, PhD, Director
6 April 2017

Name: **Remifentanil HCl**

CAS Number: 132539-07-2

Structure:



Molecular Weight: $C_{20}H_{28}N_2O_5 \cdot HCl = 412.91$

Lot Number: BDG 13122.4

Appearance: Off white, crystalline solid

Corrected Purity: 98.3 % (HPLC) - 0.6 % (acetone) - 0.2 % (diethyl ether) = 97.5 %

Re-test Date: 6 April 2022

Storage and Handling:

Temperature:	refrigerate for prolonged storage; may be handled and shipped at ambient temperature.
Humidity:	not believed to be hygroscopic; may be handled in normal laboratory atmosphere.
Light:	protect from strong sunlight.
Caution:	only experienced laboratory personnel should handle the material.

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Identity and Purity

Proton NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available. Most signals are duplicated indicating two conformers of the product exist in solution.
Residual Solvents: small amounts of diethyl ether (0.2 % *w/w*) and acetone (0.6 % *w/w*) are observed.
Impurities: no significant impurities are evident in the spectrum.

Carbon-13 NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available. Most signals are duplicated indicating two conformers of the product exist in solution.

High-resolution Mass Spectrum (ESI+)

Found *m/z* 377.2077. $C_{20}H_{29}N_2O_5$ $[M+H]^+$ (free base) requires *m/z* 377.2076. The deviation of 0.3 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A somewhat broadened, slightly tailing peak is observed (98.3 %). Note: in the absence of reference materials for preparing calibration curves, it is assumed that all peaks have the same detector response. Where possible, the conditions of analysis follow a pharmacopeial or literature method, or have been adapted from same.

Elemental Analysis

	Found:	C 58.22, H 7.13, N 6.70 %
$C_{20}H_{28}N_2O_5 \cdot HCl$	Requires:	C 58.18, H 7.08, N 6.78 %

The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

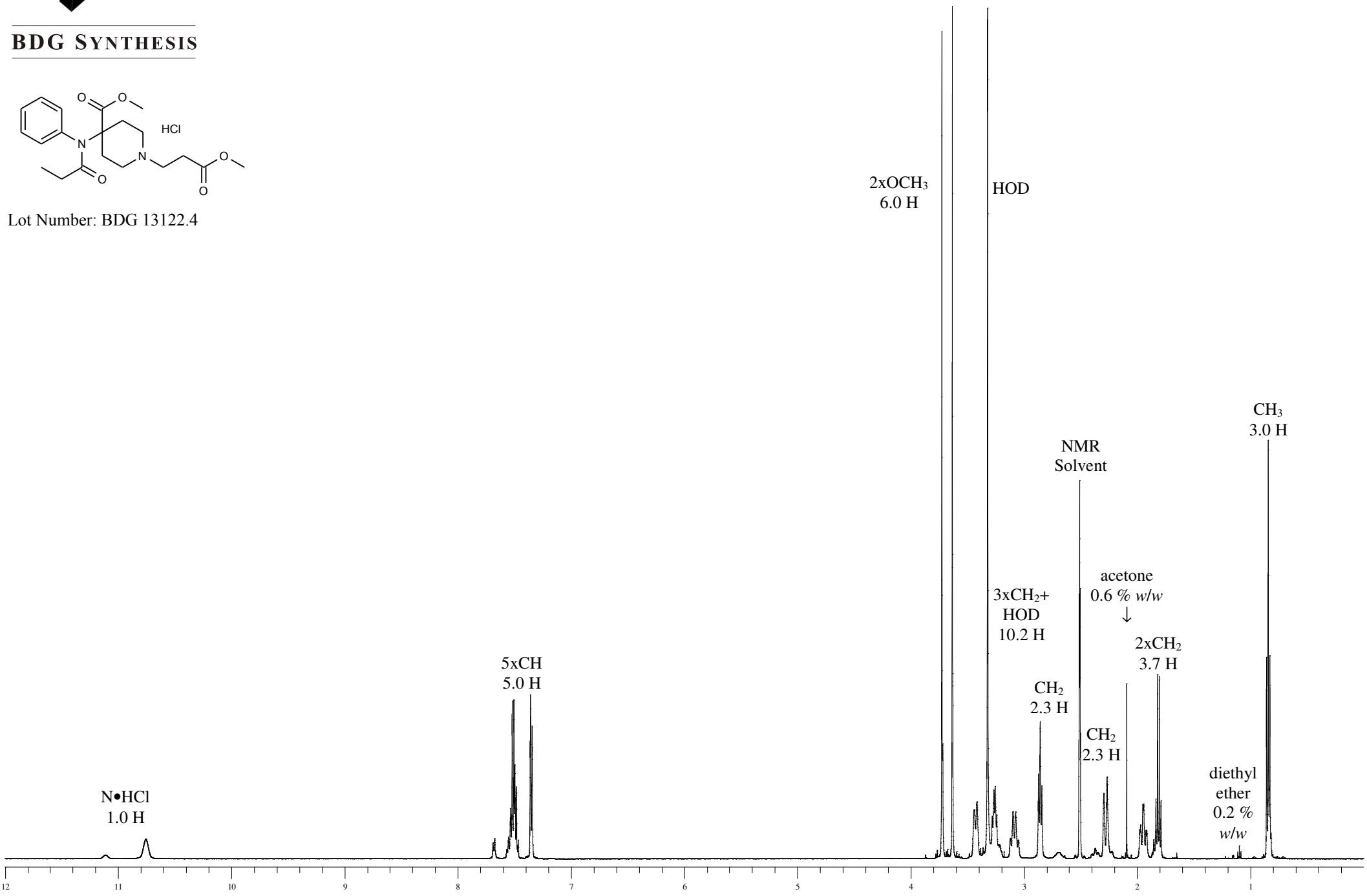
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials often contain higher levels of residual solvents and/or water, and we urge you to use the corrected purity where needed rather than the raw HPLC purity. This compound is intended for use as an analytical reference material and it is not for human administration. Structures are shown with relative stereochemistry unless otherwise specified.

The re-test date is assigned from experience gained with the material in the laboratory and/or on storage. It is not possible to perform formal storage studies because of the small amount of material available.

BDG SYNTHESIS

Proton NMR Spectrum of Remifentanil HCl in DMSO-d_6

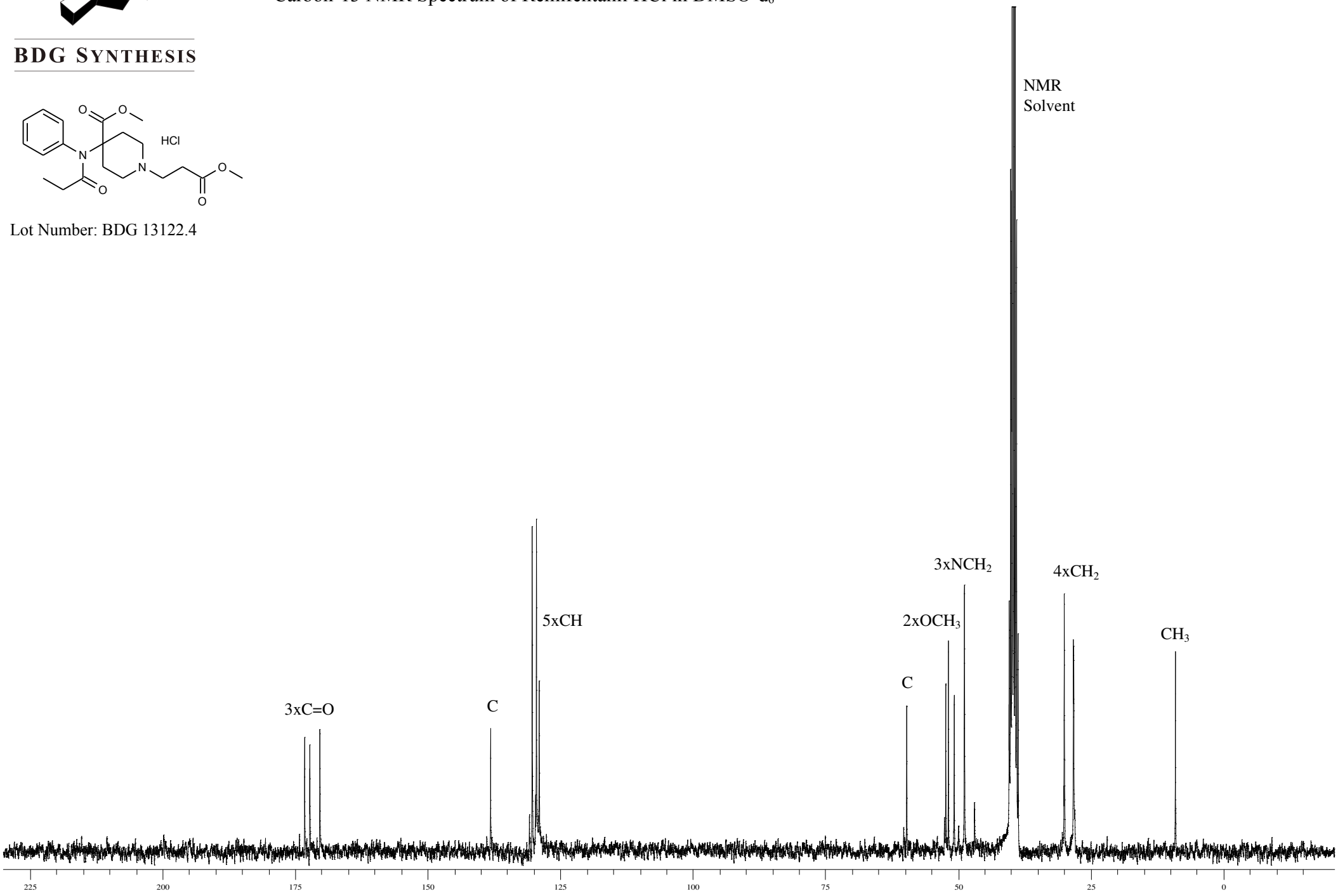
Lot Number: BDG 13122.4



BDG SYNTHESIS

Carbon-13 NMR Spectrum of Remifentanil HCl in DMSO-d_6

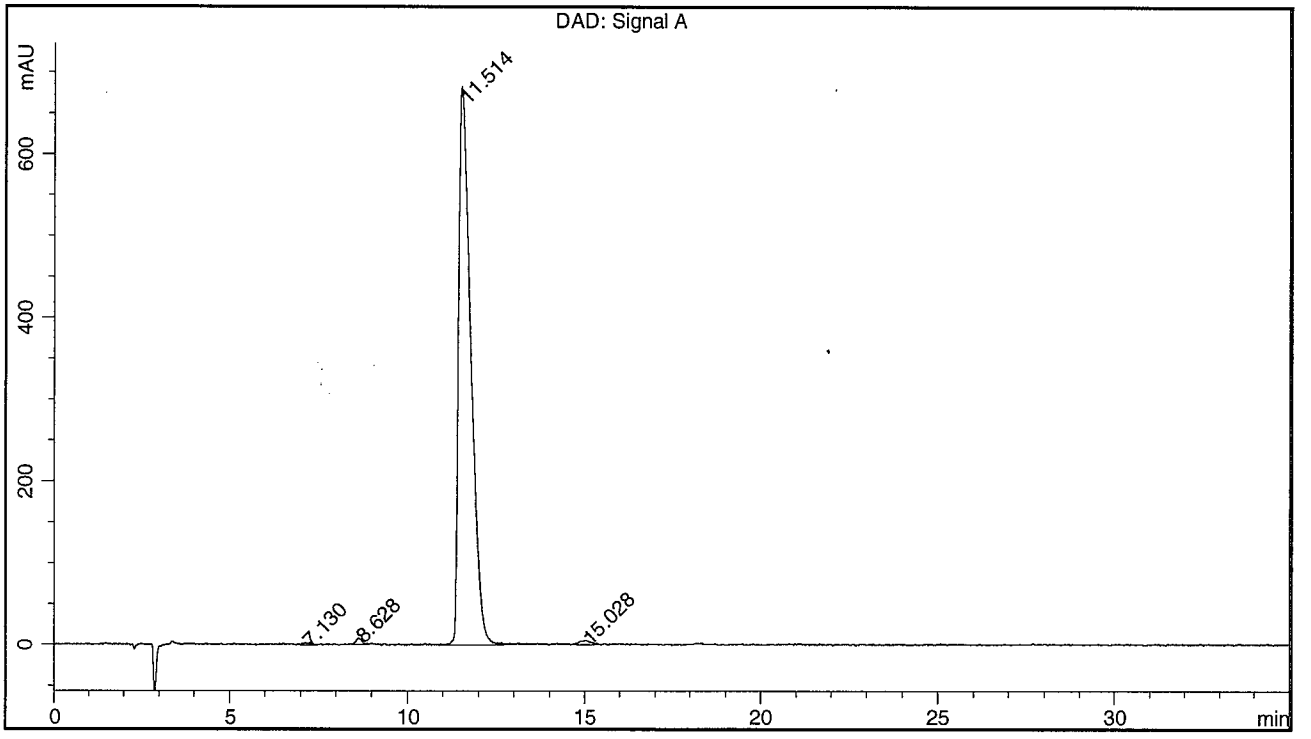
Lot Number: BDG 13122.4



BDG - Analysis of Remifentanil HCl

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase : 75:25:0.1 Water : Acetonitrile : Trifluoroacetic Acid
Flow Rate : 1.0 mL/min
Sample Solvent : 7:3 Water : Acetonitrile
Column Temperature : 20C
Injection Volume : 10 uL
Detection : UV at 210 nm

Sample Name	BDG 13122.4	Instrument	AnalyticalLC01
Acquisition	06/04/2017, 16:53:01	Method (rev.)	LC10373b (20)
Sequence	BDG_06Apr2017b - Reprocessed	Vial Position	2
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	7.13 min	2.8654	44.8172	0.1985 min	0.270 %
2	8.63 min	7.7600	90.8782	0.1586 min	0.548 %
3	11.51 min	683.2138	16293.0523	0.3462 min	98.283 %
4	15.03 min	5.8563	148.8910	0.3126 min	0.898 %