



BDG SYNTHESIS

Certificate of Analysis

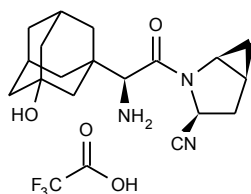
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

Neil Beare

Neil Beare, PhD, Director
24 June 2015

Name: Saxagliptin TFA Salt
CAS Number: 361442-04-8 (free base)

Structure:



Molecular Weight: $C_{18}H_{25}N_3O_2 \cdot C_2HF_3O_2 = 429.43$

Lot Number: BDG 14015.3

Appearance: White, crystalline solid

Corrected Purity: 99.2 % (HPLC) - 1.8 % (water) = 97.4 %

Re-test Date: 24 June 2020

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.

Identity and Purity

Proton NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available.

Residual Solvents: no residual solvents are observed.

Impurities: traces of unidentified impurities are seen in the baseline.

Carbon-13 NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available.

High-resolution Mass Spectrum (TOF MS ES+)

Found m/z 316.2023. $C_{18}H_{26}N_3O_2$ $[M+H]^+$ requires m/z 316.2025. The deviation of 0.6 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A broad, slightly tailing peak is observed (99.2 %). 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 55.34, H 6.18, F 13.00, N 9.41 %
$C_{18}H_{25}N_3O_2 \cdot C_2HF_3O_2 \cdot 0.4H_2O$	Requires:	C 55.01, H 6.19, F 13.05, N 9.62 %
$C_{18}H_{25}N_3O_2 \cdot C_2HF_3O_2$	Requires:	C 55.94, H 6.10, F 13.27, N 9.79 %

The elemental analyses fall slightly outside those expected for anhydrous material; the presence of water is reasonably expected from the method of purification and/or the type of material, and the "best-fit" hydrated molecular formula is given.

Karl-Fischer Analysis

	Found:	H ₂ O 1.8 %
$C_{18}H_{25}N_3O_2 \cdot C_2HF_3O_2 \cdot 0.4H_2O$	Requires:	H ₂ O 1.7 %

Of necessity, only a small sample could be used and only a single or duplicate analysis performed. We are unable to state what the errors in the reported water content are, but recommend that the result be used, as the best available, when determining corrected purity.

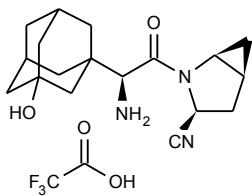
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials 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.

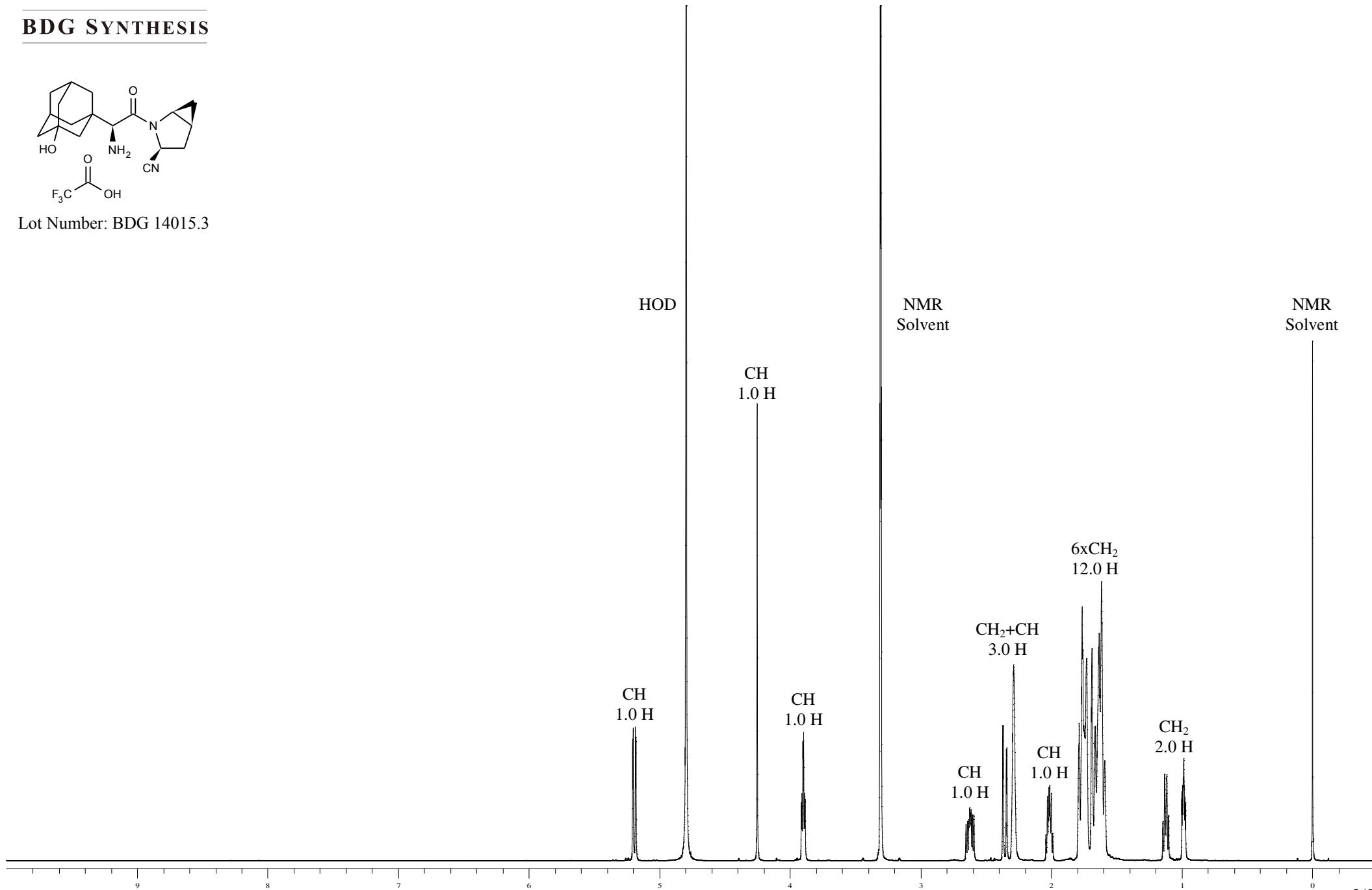


Proton NMR Spectrum of Saxagliptin TFA Salt in Methanol-d₄

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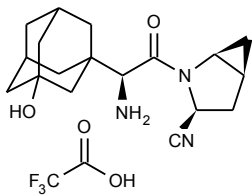
Lot Number: BDG 14015.3



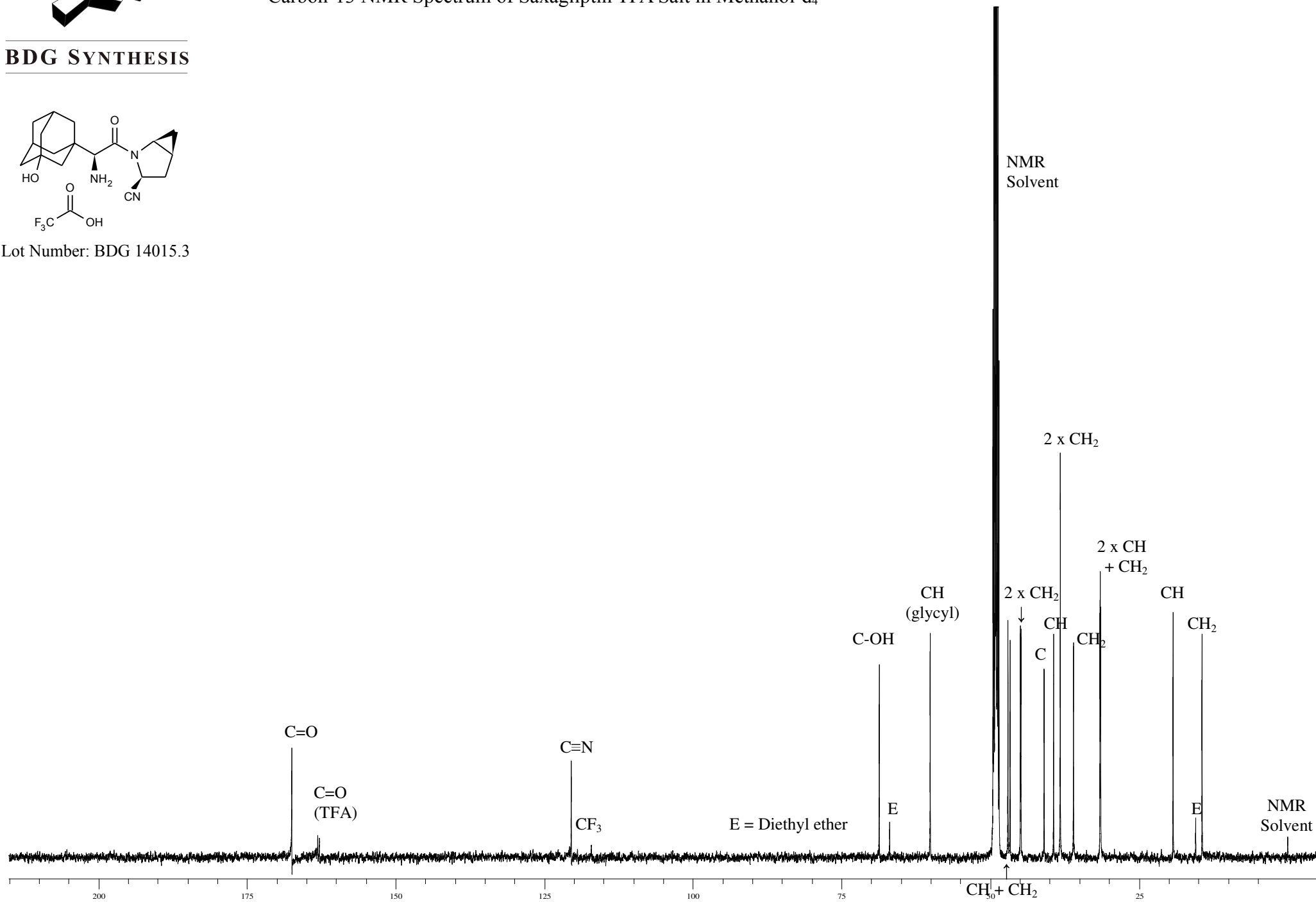


Carbon-13 NMR Spectrum of Saxagliptin TFA Salt in Methanol-d₄

BDG SYNTHESIS



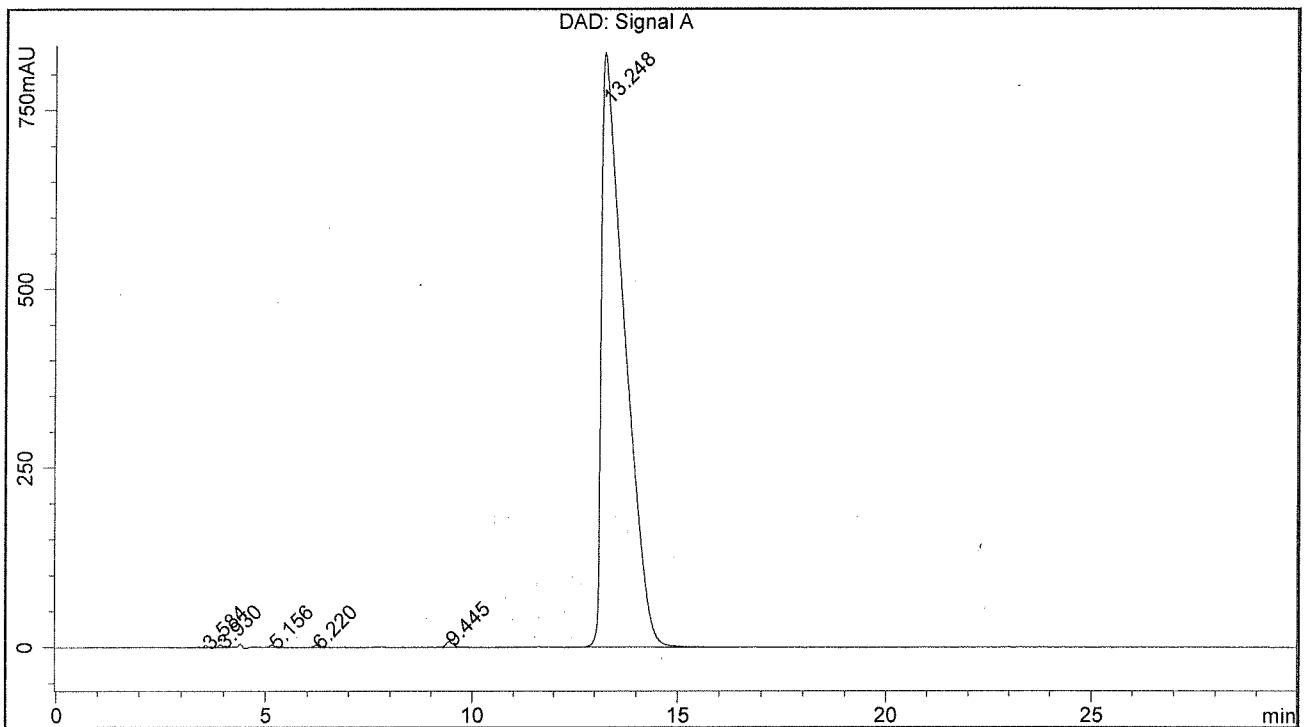
Lot Number: BDG 14015.3



BDG - Analysis of Saxagliptin TFA Salt

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase : 84:16:0.04 Water : Acetonitrile : Trifluoroacetic Acid
 Flow Rate : 1.0 mL/min
 Sample Solvent : Initial Mobile Phase
 Column Temperature : 30C
 Injection Volume : 10 uL
 Detection : UV at 212 nm

Sample Name	BDG 14015.3	Instrument	AnalyticalLC01
Acquisition	24/06/2015, 14:42:07	Method (rev.)	LC10431m (4)
Sequence	BDG_24Jun2015b - Reprocessed	Vial Position	24
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	3.58 min	3.0424	31.7321	0.1457 min	0.098 %
2	3.93 min	3.6212	26.3432	0.1129 min	0.081 %
3	5.16 min	3.1576	34.4061	0.1591 min	0.106 %
4	6.22 min	3.4237	37.6483	0.1603 min	0.116 %
5	9.44 min	8.2467	115.9986	0.2041 min	0.356 %
6	13.25 min	830.6453	32295.6230	0.5376 min	99.244 %