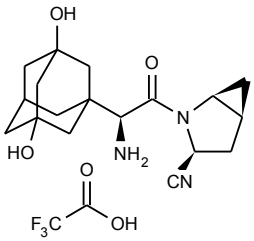
BDG SYNTHESIS

Certificate of Analysis

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

Neil Beare, PhD, Director
25 June 2015

Name:	**5-Hydroxysaxagliptin TFA Salt**
CAS Number:	841302-24-7 (free base)
Structure:	
Molecular Weight:	$C_{18}H_{25}N_3O_3 \cdot C_2HF_3O_2$ = 445.43
Lot Number:	BDG 14021.3
Appearance:	White, crystalline solid
Corrected Purity:	98.9 % (HPLC) - 1.2 % (acetone) - 0.4 % (diethyl ether) - 2.0 % (water) = 95.3 %
Re-test Date:	25 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.

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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.
Residual Solvents: small amounts of diethyl ether (0.4 % *w/w*) and acetone (1.2 % *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.

High-resolution Mass Spectrum (ESI+)

Found *m/z* 332.1972. $C_{18}H_{26}N_3O_3$ $[M+H]^+$ requires *m/z* 332.1974. The deviation of 0.6 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A sharp, symmetrical peak is observed (98.9 %). 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 52.15, H 6.12, F 12.72, N 8.78 %
$C_{18}H_{25}N_3O_3{\cdot}C_2HF_3O_2{\cdot}0.8H_2O$	Requires:	C 52.24, H 6.05, F 12.39, N 9.14 %
$C_{18}H_{25}N_3O_3{\cdot}C_2HF_3O_2$	Requires:	C 53.93, H 5.88, F 12.80, N 9.43 %

The elemental analyses fall somewhat 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_2O 2.0 %
$C_{18}H_{25}N_3O_3{\cdot}C_2HF_3O_2{\cdot}0.8H_2O$	Requires:	H_2O 2.0 %

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.

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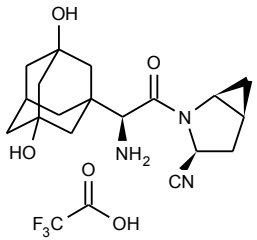

Lot Number: BDG 14021.3

Proton NMR Spectrum of 5-Hydroxysaxagliptin TFA Salt in Methanol-d_4

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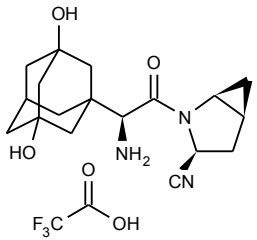
Carbon-13 NMR Spectrum of 5-Hydroxysaxagliptin TFA Salt in Methanol-d_4

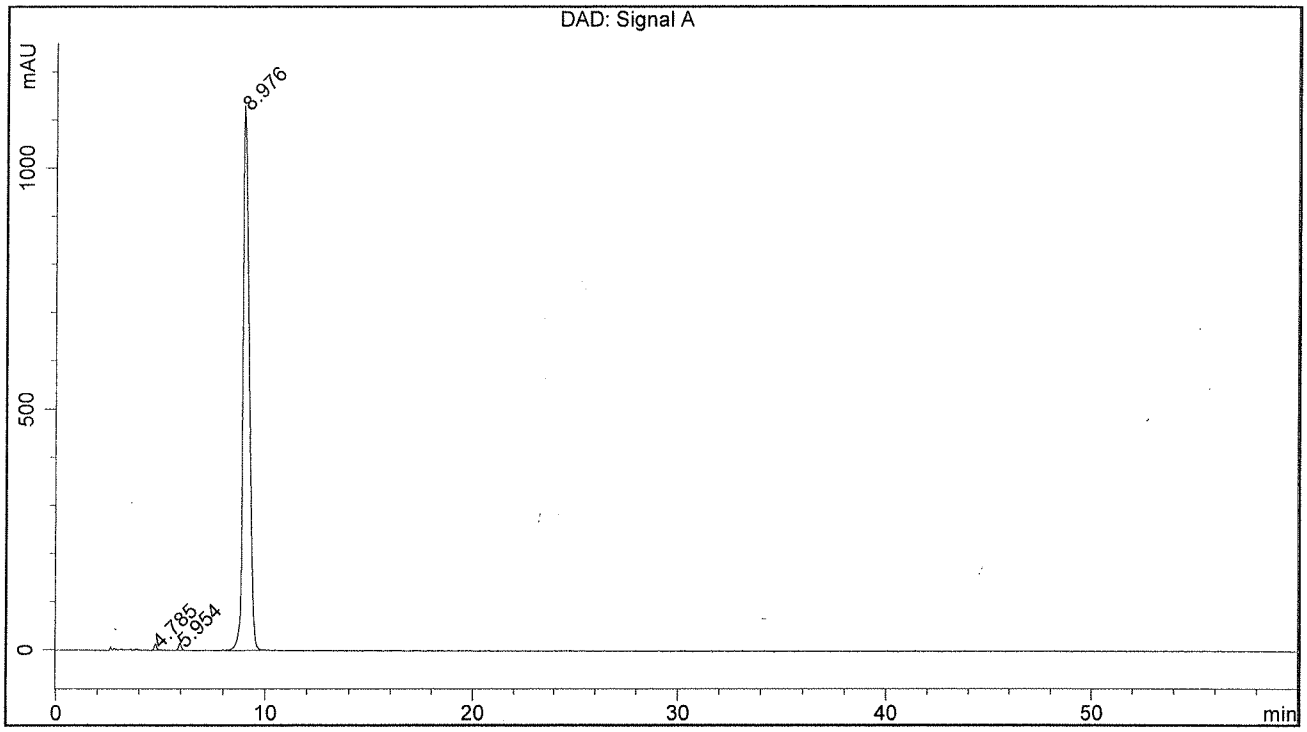


Lot Number: BDG 14021.3

BDG - Analysis of 5-Hydroxysaxagliptin 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 : 90:10:0.10 Water : Acetonitrile : Trifluoroacetic Acid
Flow Rate : 1.0 mL/min
Sample Solvent : Mobile Phase
Column Temperature : 20C
Injection Volume : 10 uL
Detection : UV at 212 nm

Sample Name	BDG 14021.3	**Instrument**	AnalyticalLC01
Acquisition	25/06/2015, 08:42:47	**Method (rev.)**	LC10431k (9)
Sequence	BDG_24Jun2015c - Reprocessed	**Vial Position**	25
Operator	solvation010\cerityadmin	**Injection**	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	4.79 min	14.0335	115.8573	0.1243 min	0.484 %
2	5.95 min	14.5427	140.9621	0.1451 min	0.589 %
3	8.98 min	1129.2753	23678.7065	0.3226 min	98.927 %