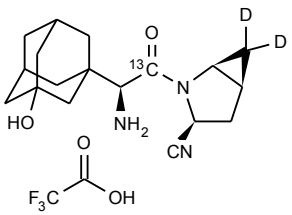
BDG SYNTHESIS

Certificate of Analysis

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

Neil Beare, PhD, Director
30 April 2016

Name:	**Saxagliptin-^{13}C,d$_2$ TFA Salt**
CAS Number:	361442-04-8 (unlabelled free base)
Structure:	
Molecular Weight:	$C_{17}{}^{13}CH_{23}D_2N_3O_2 \cdot C_2HF_3O_2$ = 432.44
Lot Number:	BDG 5992.7
Appearance:	White, crystalline solid
Purity By HPLC:	99.0 %
Isotopic Purity:	Under 0.5% M-3
Re-test Date:	30 April 2021

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.
Isotopic Labelling: signals at the site of deuteration are absent, compared with the spectrum of unlabelled material, indicating clean deuteration.
Residual Solvents: no residual solvents are observed.
Impurities: a trace of an unidentified impurity is seen in the baseline.

Carbon-13 NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available. Signals for the trifluoroacetate moiety are not observed.
Isotopic Labelling: the signal at the site of deuteration has collapsed to a small multiplet compared with the spectrum of unlabelled material, indicating clean deuteration and the signal at the ^{13}C labelled site is massively enhanced as expected.

High-resolution Mass Spectrum (TOF MS ES+)

Found *m/z* 319.2186. $C_{17}{}^{13}CH_{24}D_2N_3O_2$ $[M+H]^+$ requires *m/z* 319.2184. The deviation of 0.6 ppm is within normally accepted limits for the establishment of identity by HRMS. No signal for M-3 material was seen (detection limit about 0.5 %).

HPLC

A somewhat broadened, symmetrical peak is observed (99.0 %). 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 55.59, H 5.52, D 0.96, N 9.56 %
$C_{17}{}^{13}CH_{23}D_2N_3O_2 \cdot C_2HF_3O_2$	Requires:	C 55.78, H 5.59, D 0.93, N 9.72 %

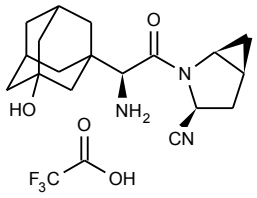
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

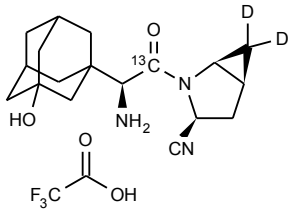
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials 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 Saxagliptin TFA Salt (top) and Saxagliptin-^{13}C,d_2 TFA Salt (bottom) in Methanol-d_4

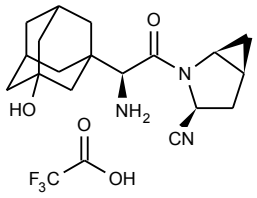


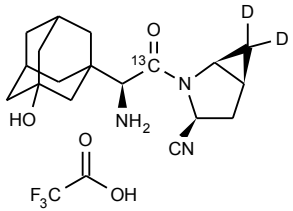


Lot Number: BDG 5992.7

BDG SYNTHESIS

Carbon-13 NMR Spectrum of Saxagliptin TFA Salt (top) and Saxagliptin-$^{13}C,d_2$ TFA Salt (bottom) in Methanol-d_4





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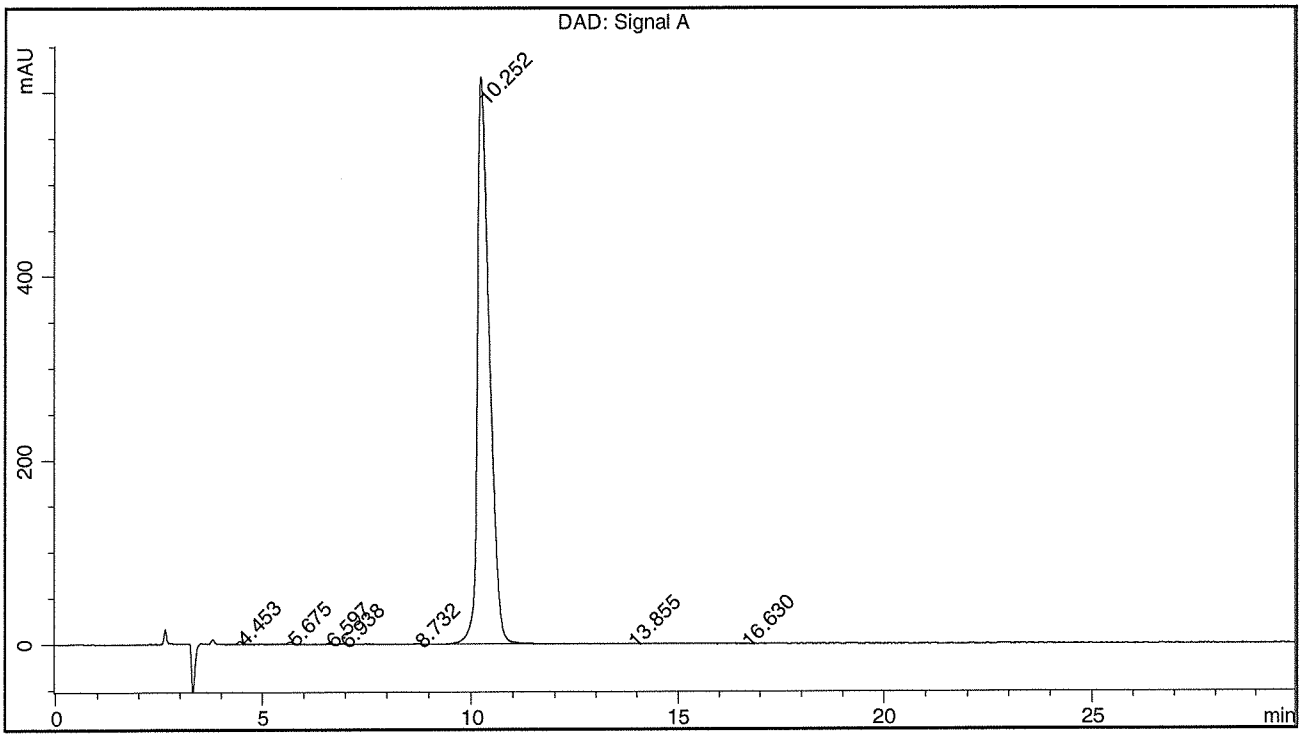
BDG - Analysis of Saxagliptin-13C,d2 TFA Salt

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase : 82:18:0.1 Water : Acetonitrile : Trifluoroacetic Acid
Flow Rate : 1.0 mL/min
Sample Solvent : 80:20 Water : Acetonitrile
Column Temperature : 20 C
Injection Volume : 10 uL
Detection : UV at 212 nm

Sample Name	BDG 5992.7	**Instrument**	AnalyticalLC01
Acquisition	30/04/2016, 17:19:00	**Method (rev.)**	LC10431f (14)
Sequence	BDG_30Apr2016a - Reprocessed	**Vial Position**	2
Operator	solvation010\cerityadmin	**Injection**	1 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	4.45 min	3.3766	27.1152	0.1217 min	0.218 %
2	5.68 min	2.8220	36.7559	0.1880 min	0.296 %
3	6.60 min	1.2010	10.4703	0.1297 min	0.084 %
4	6.94 min	0.9289	10.6729	0.1660 min	0.086 %
5	8.73 min	0.6105	8.5656	0.2197 min	0.069 %
6	10.25 min	614.9090	12312.1692	0.3056 min	99.030 %
7	13.85 min	0.4413	24.0347	0.6514 min	0.193 %
8	16.63 min	0.4268	2.9978	0.1059 min	0.024 %