BDG SYNTHESIS

Certificate of Analysis

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

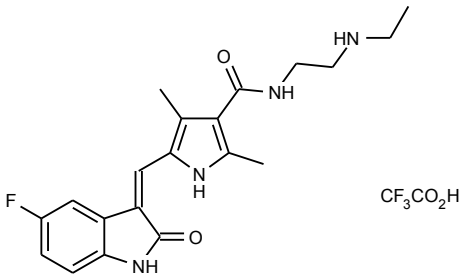
Barry Dent

Barry R. Dent, PhD, Director
22 August 2010

Name: ***N*-Desethylsunitinib TFA Salt**

CAS Number: 356068-97-8 (free base)

Structure:



Molecular Weight: $C_{20}H_{23}FN_4O_2 \cdot C_2HF_3O_2$ = 484.44

Lot Number: BDG 8426.6

Appearance: Orange, crystalline solid

Corrected Purity: 99.5 % (HPLC) - 3.6 % (water) = 95.9 %

Re-test Date: 22 August 2011

Storage and Handling:

Temperature:	refrigerate for prolonged storage; may be handled and shipped at ambient temperature.
Humidity:	may be hygroscopic; store desiccated; recommended to determine water content periodically.
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: 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 (ESI+)

Found *m/z* 371.1875. $C_{20}H_{24}FN_4O_2$ $[M+H]^+$ requires *m/z* 371.1878. The deviation of 2.2 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A sharp, symmetrical peak is observed (99.5 %). 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 52.26, H 4.93, N 11.21 %
$C_{20}H_{23}FN_4O_2 \cdot C_2HF_3O_2 \cdot 1.0H_2O$	Requires:	C 52.59, H 5.22, N 11.15 %
$C_{20}H_{23}FN_4O_2 \cdot C_2HF_3O_2$	Requires:	C 54.54, H 4.99, N 11.57 %

The elemental analyses fall substantially 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 3.6 %
$C_{20}H_{23}FN_4O_2 \cdot C_2HF_3O_2 \cdot 1.0H_2O$	Requires:	H_2O 3.6 %

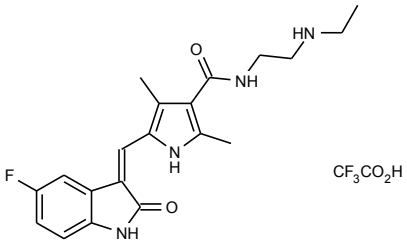
Of necessity, only a small sample could be used and only a single or duplicate analysis performed. We are unable to state what the errors in the reported water content are, but recommend that the result be used, as the best available, when determining corrected purity.

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.

BDG SYNTHESIS

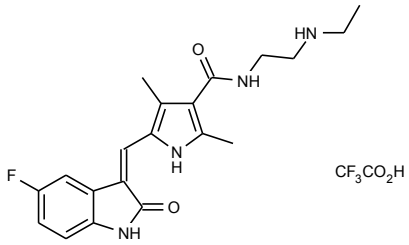
Proton NMR Spectrum of *N*-Desethylsunitinib TFA Salt in DMSO-d_6



Lot Number: BDG 8426.6

BDG SYNTHESIS

Carbon-13 NMR Spectrum of *N*-Desethylsunitinib TFA Salt in DMSO-d_6

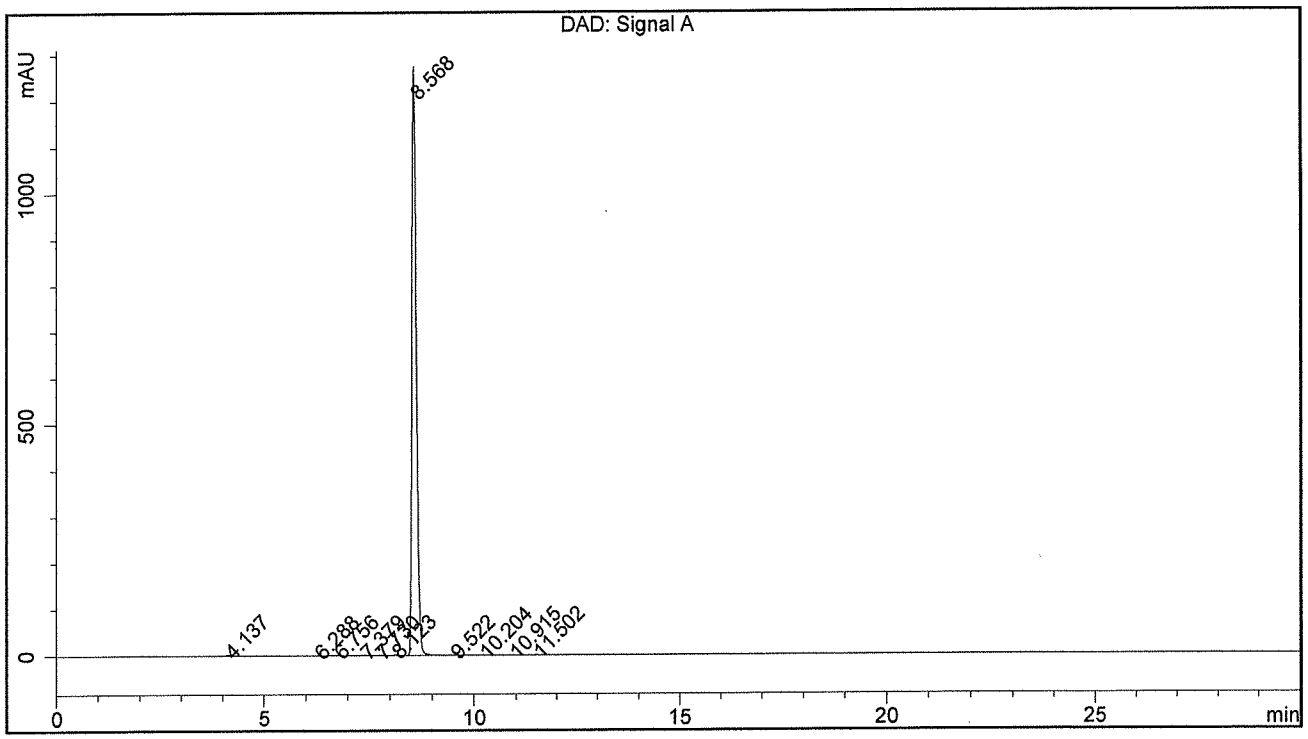


Lot Number: BDG 8426.6

BDG - Analysis of N-Desethylsunitinib TFA salt

Column : Phenomenex Luna C18 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 4 x 3 mm
Mobile Phase A : Water + 0.10% Trifluoroacetic Acid
Mobile Phase B : Acetonitrile + 0.10% Trifluoroacetic Acid
Gradient (A:B) : T0 = 70:30, T25 = 30:70, T30 = 30:70, T35 = 70:30, T40 = 70:30
Flow Rate : 1.0 mL/min
Sample Solvent : 70:30 Water : Acetonitrile
Column Temperature : 20C
Injection Volume : 10 uL
Detection : UV-Vis at 435 nm

Sample Name	BDG 8426.6	Instrument	AnalyticalLC01
Acquisition	22/08/2010, 11:13:41	Method (rev.)	LC10211a (13)
Sequence	BDG_22Aug2010a	Vial Position	11
Operator	solvation010\cerityadmin	Injection	2 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	4.14 min	2.2222	17.6185	0.1225 min	0.189 %
2	6.29 min	0.1112	0.6965	0.0891 min	0.007 %
3	6.76 min	0.5624	3.8486	0.1018 min	0.041 %
4	7.38 min	1.0453	7.2778	0.1052 min	0.078 %
5	7.73 min	0.1254	0.8343	0.0936 min	0.009 %
6	8.12 min	1.2542	8.7415	0.1073 min	0.094 %
7	8.57 min	1276.6611	9271.5191	0.1147 min	99.499 %
8	9.52 min	0.1231	0.7353	0.0837 min	0.008 %
9	10.20 min	0.1139	0.8483	0.0989 min	0.009 %
10	10.91 min	0.2520	1.7165	0.0995 min	0.018 %
11	11.50 min	0.6356	4.3474	0.1018 min	0.047 %