

BDG SYNTHESIS

Certificate of Analysis

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

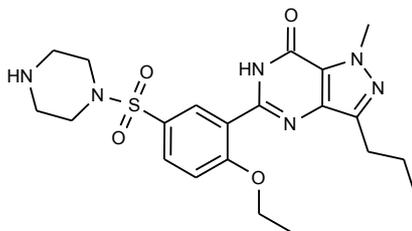
Neil Beare

Neil Beare, PhD, Director
1 November 2014

Name: *N*-Desmethylsildenafil

CAS Number: 139755-82-1

Structure:



Molecular Weight: $C_{21}H_{28}N_6O_4S = 460.55$

Lot Number: BDG 4434

Appearance: White, crystalline solid

Corrected Purity: 99.2 % (HPLC) - 1.9 % (water) = 97.3 %

Re-test Date: 1 November 2019

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: 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 461.1977. $C_{21}H_{29}N_6O_4S$ $[M+H]^+$ requires m/z 461.1971. The deviation of 1.3 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A sharp, 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 53.41, H 6.43, N 17.86 %
$C_{21}H_{28}N_6O_4S \cdot 0.5H_2O$	Requires:	C 53.72, H 6.23, N 17.90 %
$C_{21}H_{28}N_6O_4S$	Requires:	C 54.77, H 6.13, N 18.25 %

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 ₂ O 1.9 %
$C_{21}H_{28}N_6O_4S \cdot 0.5H_2O$	Requires:	H ₂ O 1.9 %

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.

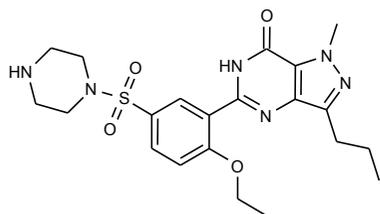
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

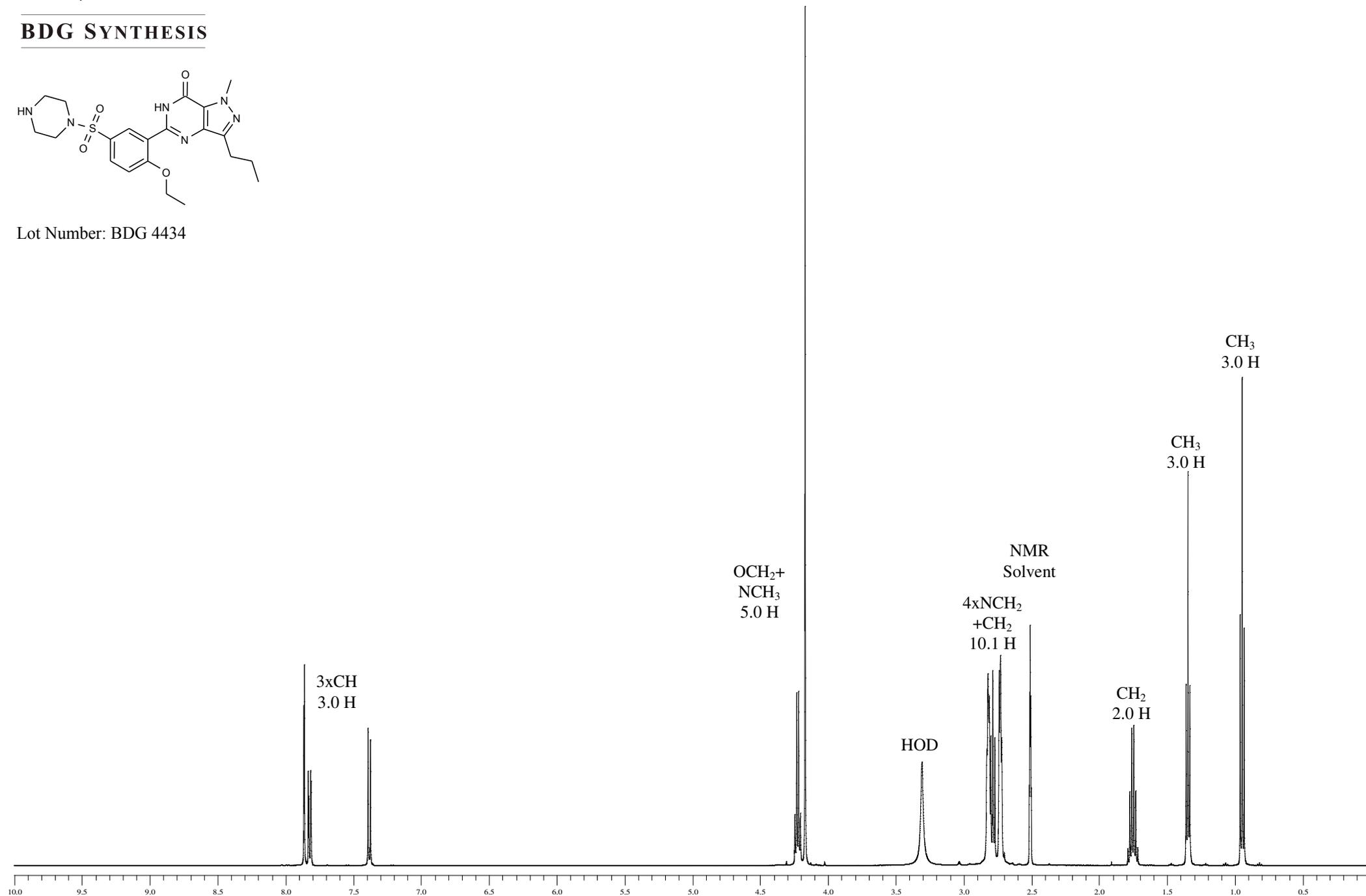


Proton NMR Spectrum of *N*-Desmethylsildenafil in DMSO- d_6

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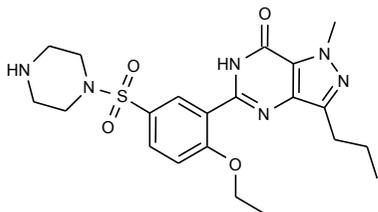
Lot Number: BDG 4434



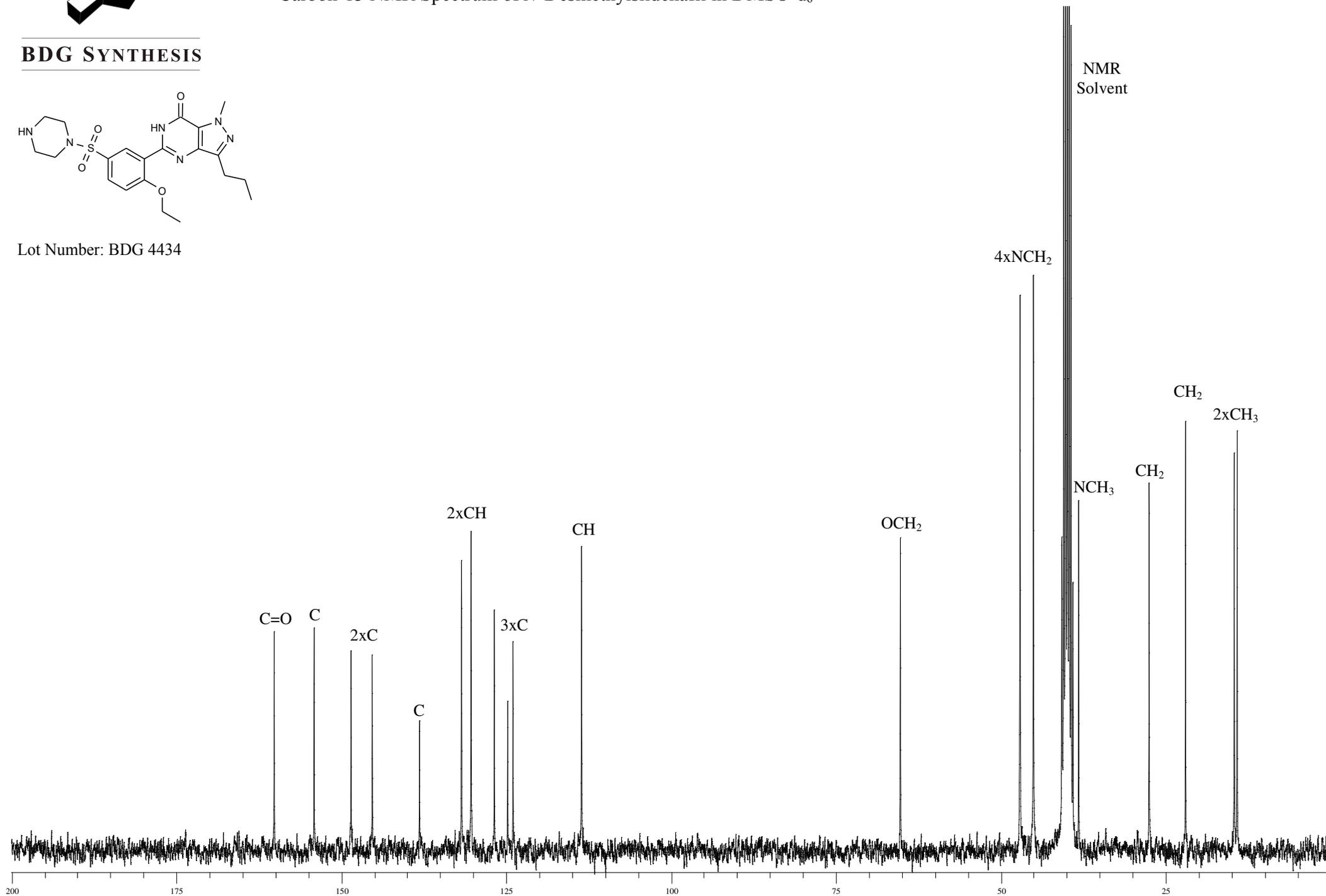


Carbon-13 NMR Spectrum of *N*-Desmethylsildenafil in DMSO- d_6

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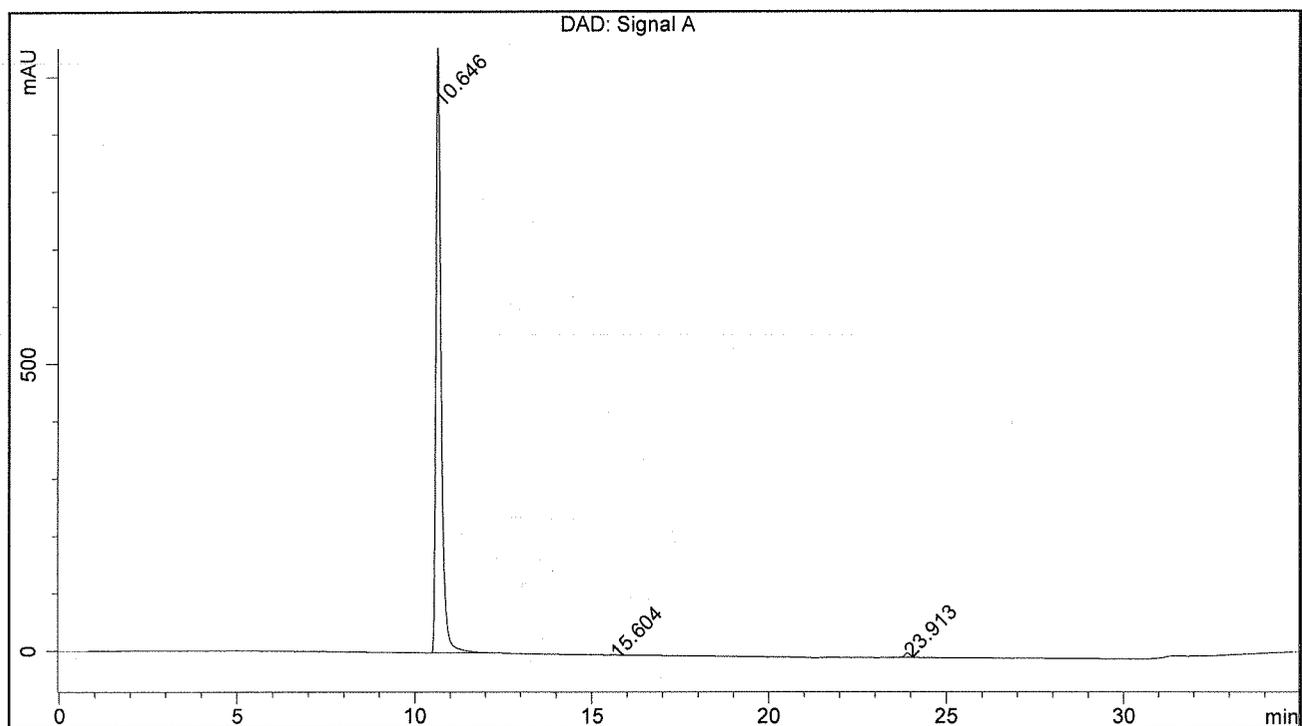
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BDG - Analysis of N-Desmethylsildenafil

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase A : 70:30 200 mM Ammonium Acetate : Acetonitrile
 Mobile Phase B : 30:70 200 mM Ammonium Acetate : Acetonitrile
 Gradient (A:B) : T0=100:0, T20=0:100, T28=0:100. T31=100:0, T35=100:0
 Flow Rate : 1.0 mL/min
 Sample Solvent : Initial Mobile Phase
 Column Temperature : 20C
 Injection Volume : 10 uL
 Detection : UV at 240 nm

Sample Name	BDG 4434	Instrument	AnalyticalLC01
Acquisition	01/11/2014, 15:09:42	Method (rev.)	LC10271c (6)
Sequence	BDG_01Nov2014a	Vial Position	1
Operator	solvation010\cerityadmin	Injection	1 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	10.65 min	1063.1008	10838.3822	0.1529 min	99.174 %
2	15.60 min	0.5272	5.2128	0.1474 min	0.048 %
3	23.91 min	7.8705	85.0264	0.1680 min	0.778 %