



## BDG SYNTHESIS

### Certificate of Analysis

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

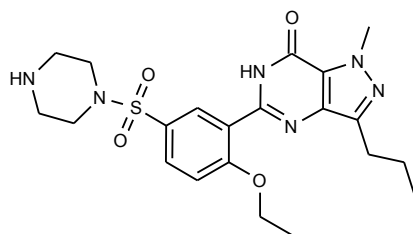
*Barry Dent*

Barry R. Dent, PhD, Director  
11 February 2010

**Name:** *N*-Desmethyilsildenafil

**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:** 99.1 % (HPLC) - 3.3 % (water) = 95.8 %

**Re-test Date:** 11 February 2011

**Storage and Handling:**

Temperature:	ambient laboratory temperature; may be refrigerated.
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.

## 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 (99.1 %). 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 <sub>2</sub> O 3.3 %
$C_{21}H_{28}N_6O_4S \cdot 0.5H_2O$	Requires:	H <sub>2</sub> 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.

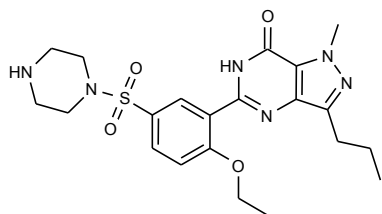
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

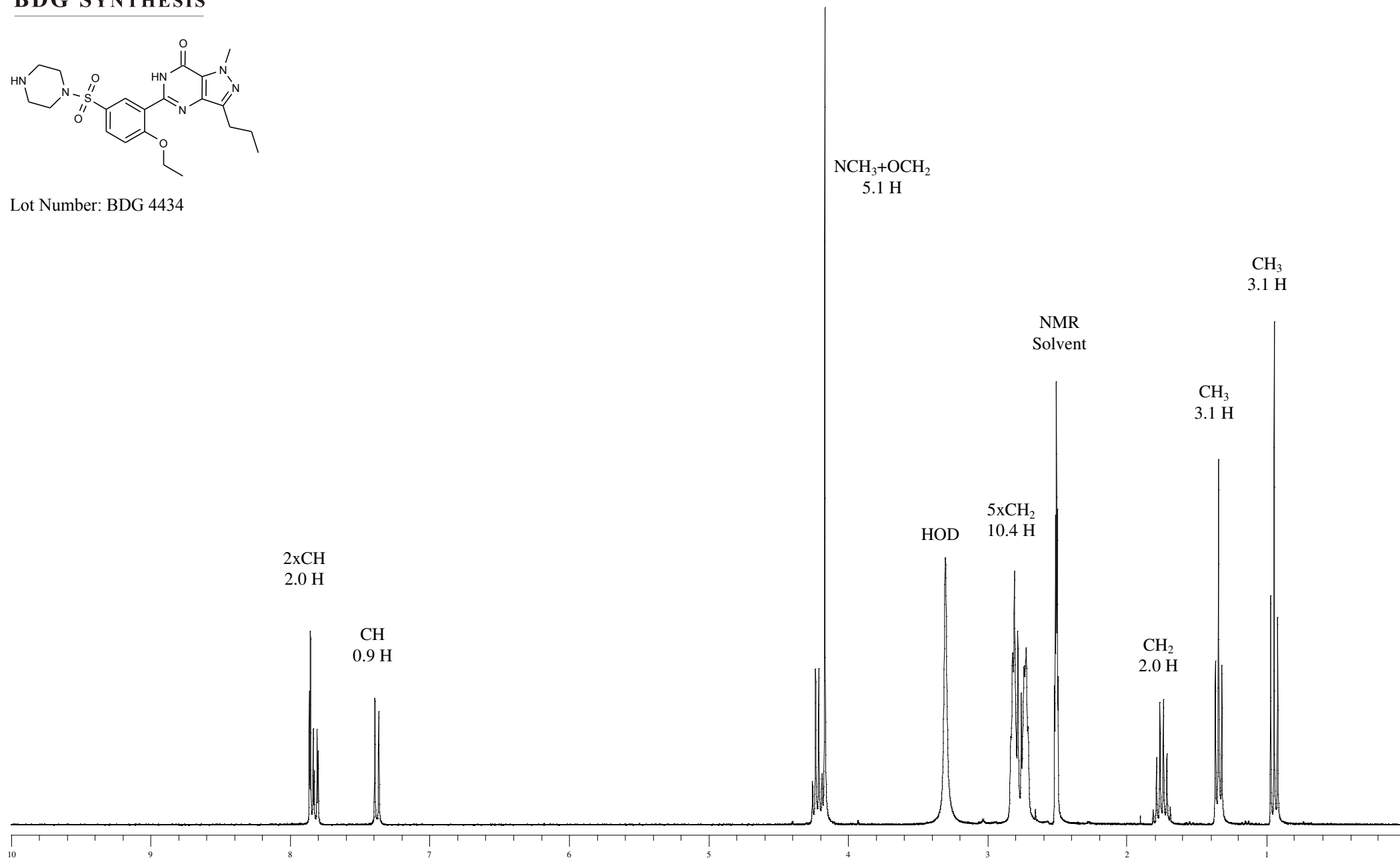


Proton NMR Spectrum of *N*-Desmethylsildenafil in DMSO-d<sub>6</sub>

**BDG SYNTHESIS**



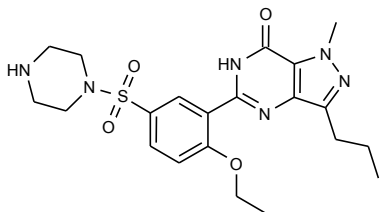
Lot Number: BDG 4434



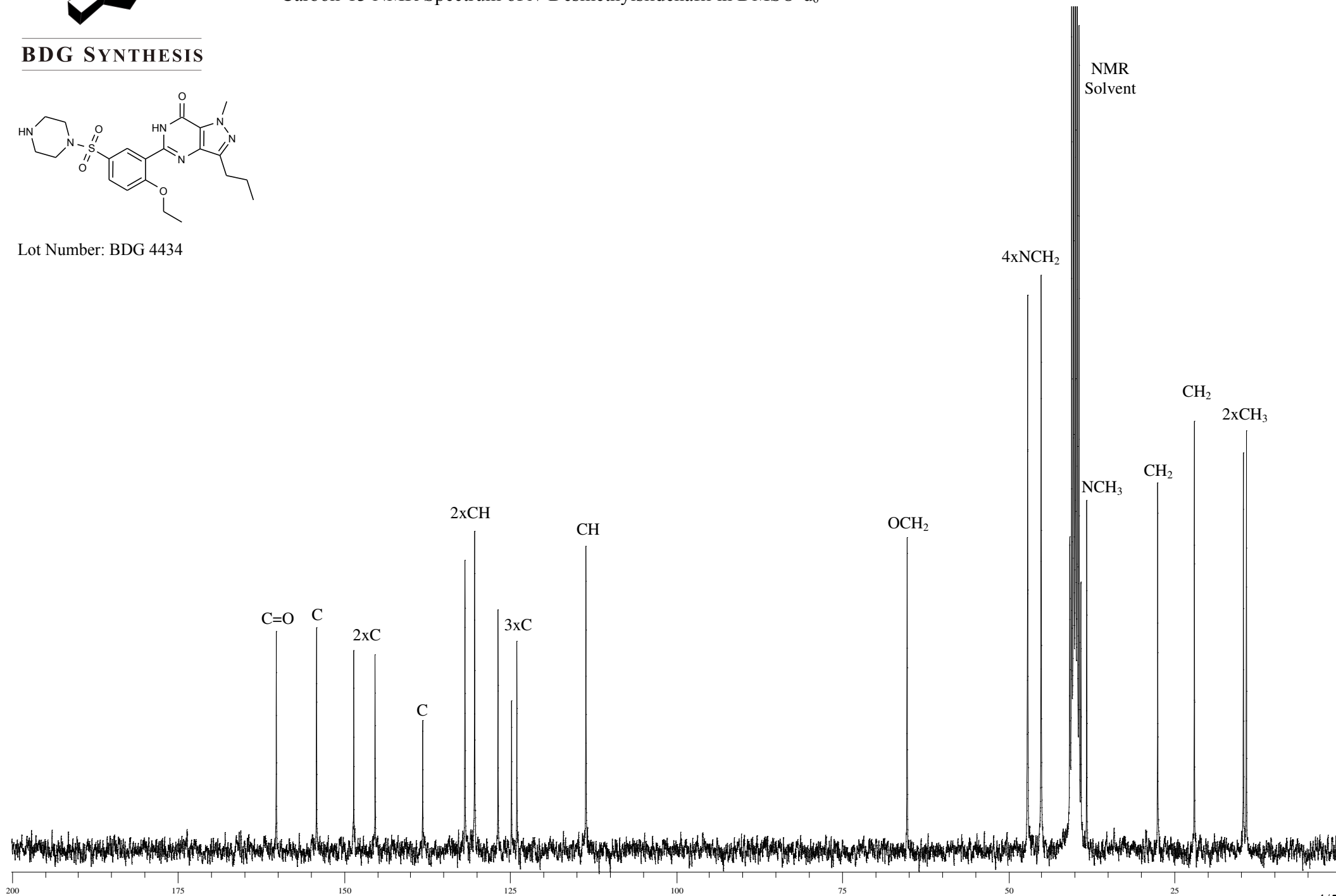


Carbon-13 NMR Spectrum of *N*-Desmethylsildenafil in DMSO- $d_6$

**BDG SYNTHESIS**



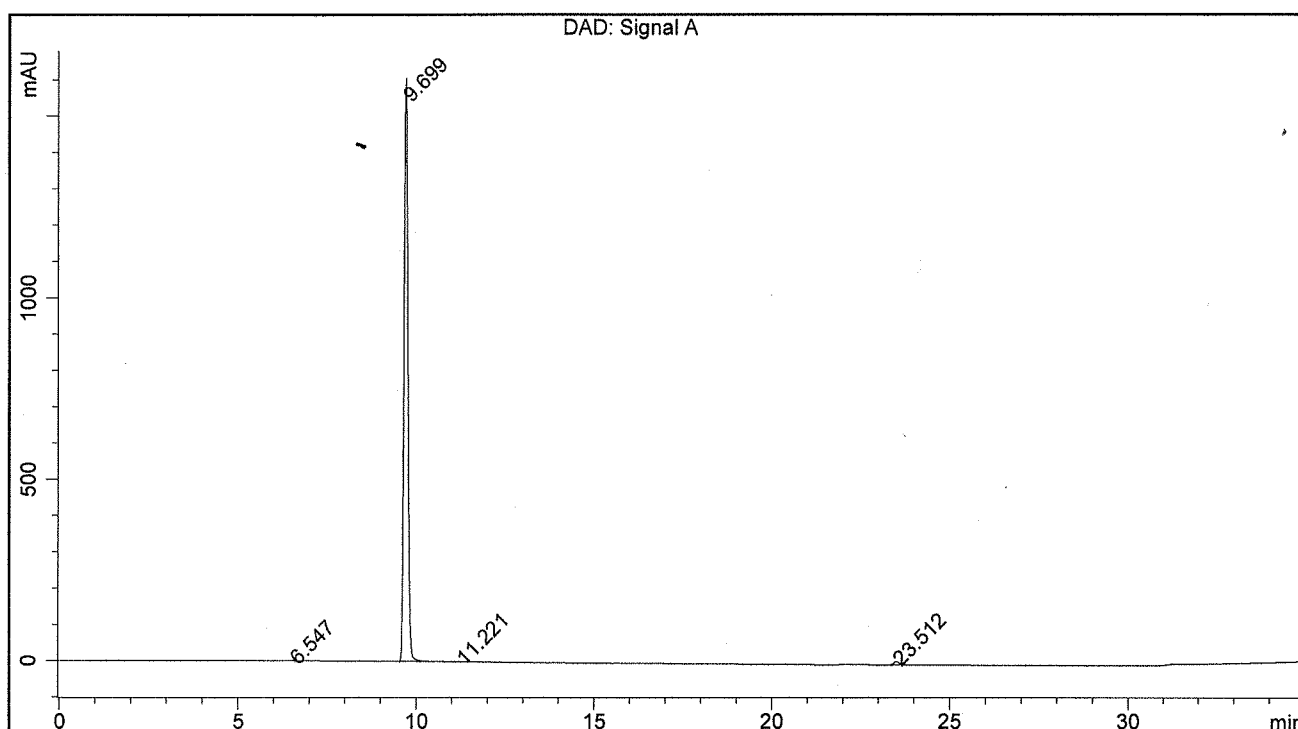
Lot Number: BDG 4434



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 : 0.20 M Ammonium Acetate  
 Mobile Phase B : Acetonitrile  
 Gradient ( A:B ) : T0=70:30, T20=30:70, T28=30:70. T31=70:30, T35=70:30  
 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	11/02/2010, 15:46:40	Method (rev.)	LC10271c ( 4)
Sequence	BDG_11Feb2010b - Reprocessed	Vial Position	2
Operator	solvation010\cerityadmin	Injection	2 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	6.55 min	0.6072	4.0142	0.1011 min	0.033 %
2	9.70 min	1607.6085	12087.8464	0.1177 min	99.106 %
3	11.22 min	0.7531	5.2470	0.1093 min	0.043 %
4	23.51 min	8.9471	99.7981	0.1681 min	0.818 %