



BDG SYNTHESIS

Certificate of Analysis

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

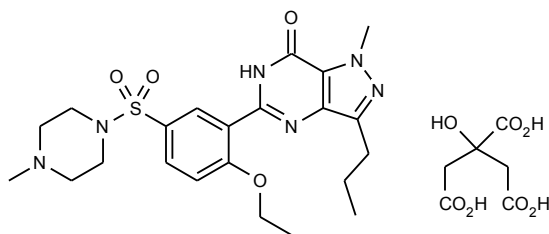
Barry Dent

Barry R. Dent, PhD, Director
26 July 2012

Name: Sildenafil Citrate

CAS Number: 171599-83-0

Structure:



Molecular Weight: $C_{22}H_{30}N_6O_4S \cdot C_6H_8O_7 = 666.70$

Lot Number: BDG 10421.1

Appearance: White, crystalline solid

Purity By HPLC: 99.7 %

Re-test Date: 26 July 2017

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 475.2119. $C_{22}H_{31}N_6O_4S$ $[M+H]^+$ requires m/z 475.2128. The deviation of 1.9 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A sharp, symmetrical peak is observed (99.7 %). 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

$C_{22}H_{30}N_6O_4S \cdot C_6H_8O_7$	Found:	C 50.26, H 5.75, N 12.57 %
	Requires:	C 50.44, H 5.74, N 12.61 %

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).

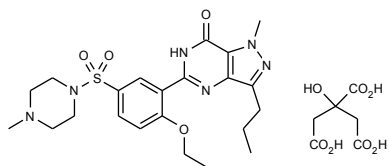
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

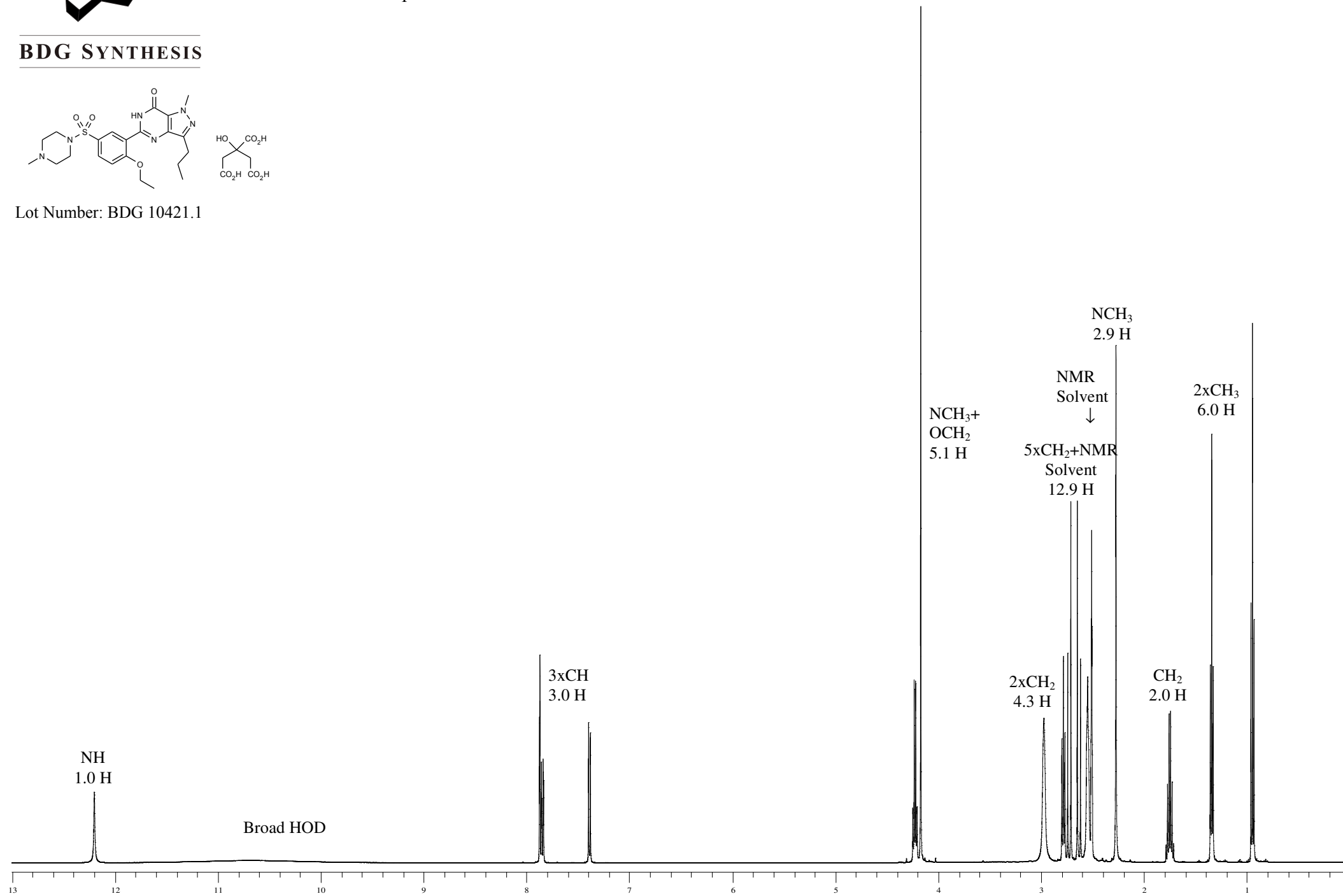


Proton NMR Spectrum of Sildenafil Citrate in DMSO-d₆

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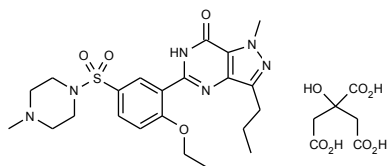
Lot Number: BDG 10421.1



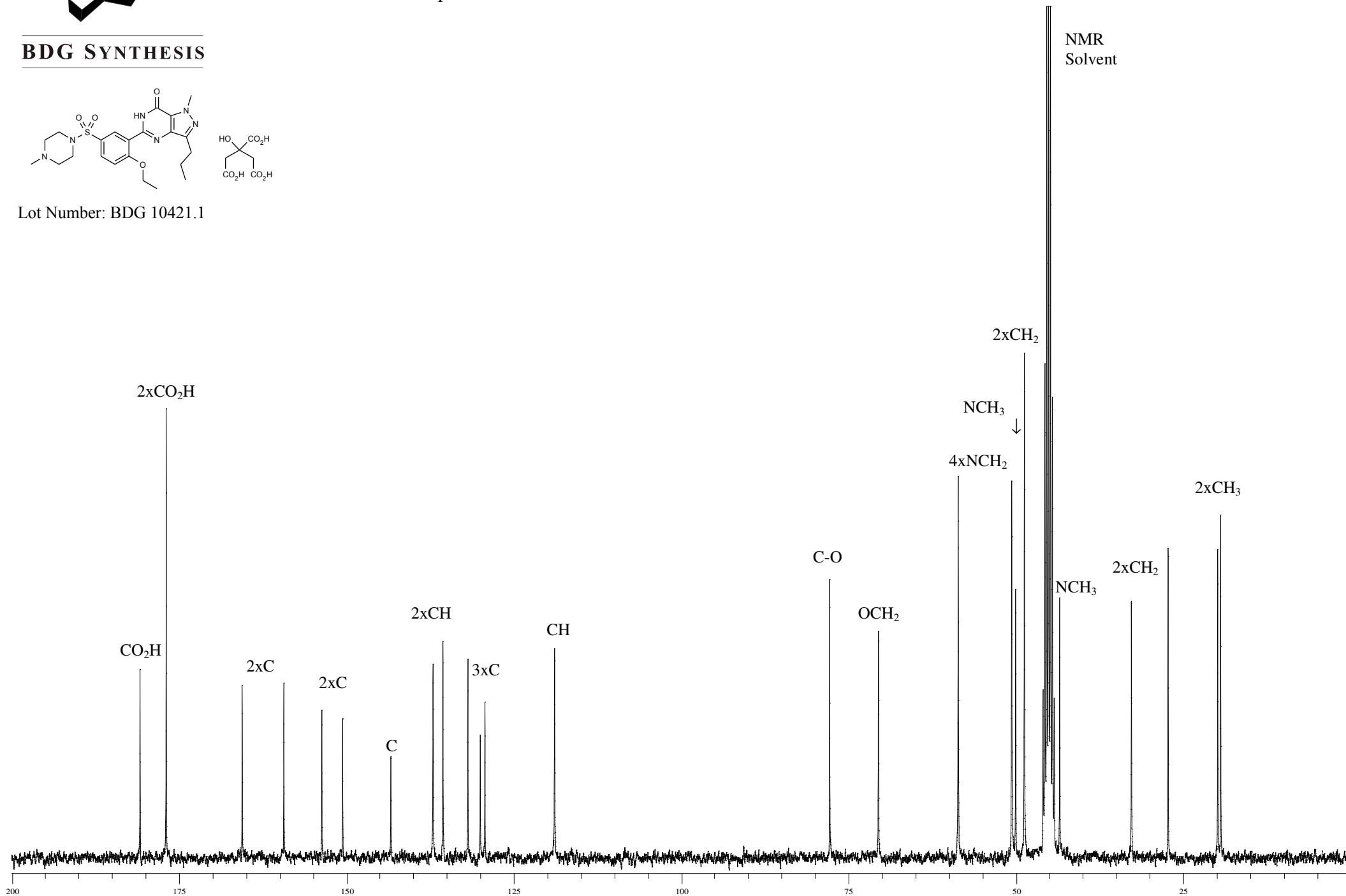


Carbon-13 NMR Spectrum of Sildenafil Citrate in DMSO-d₆

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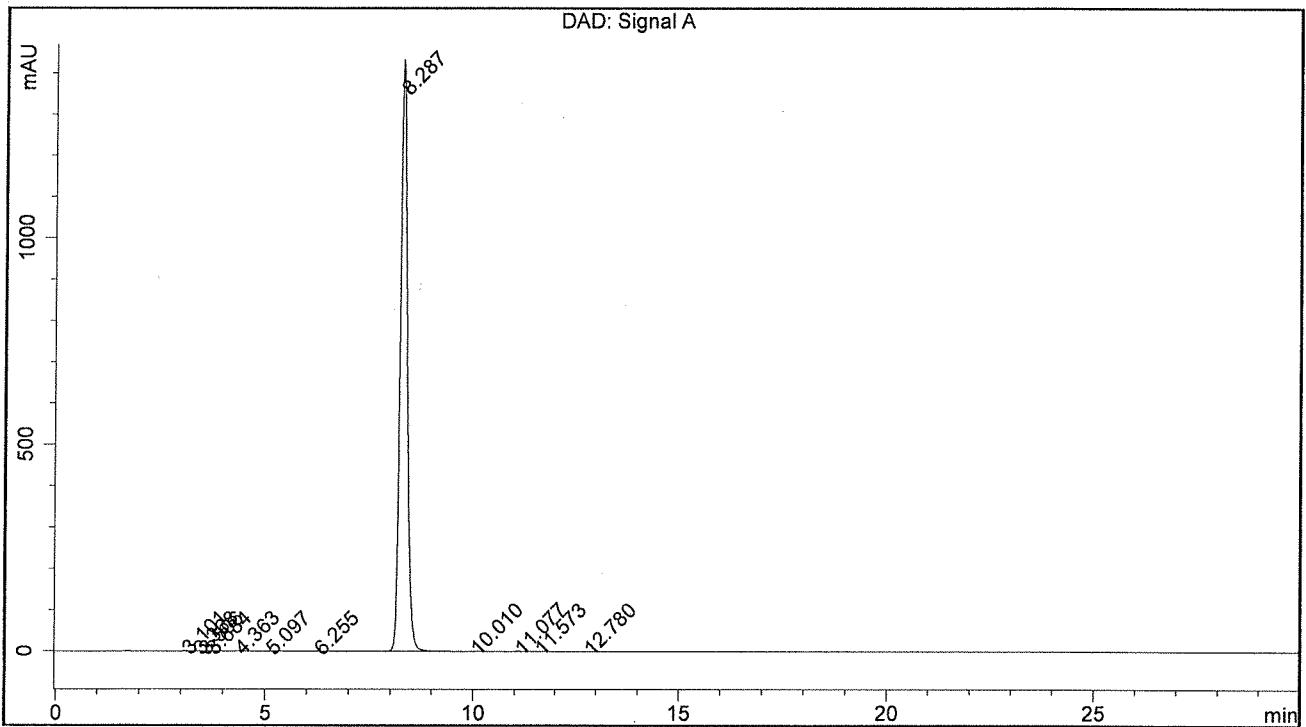
Lot Number: BDG 10421.1



BDG - Analysis of Sildenafil Citrate

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 4 x 3 mm
 Mobile Phase : 1:1 200 mM Ammonium Acetate (pH=7.0) : Acetonitrile
 Flow Rate : 1.0 mL/min
 Sample Solvent : Mobile Phase
 Column Temperature : 20C
 Injection Volume : 10 uL
 Detection : UV at 240 nm

Sample Name	BDG 10421.1	Instrument	AnalyticalLC01
Acquisition	26/07/2012, 15:05:20	Method (rev.)	LC10527a (4)
Sequence	BDG_26Jul2012b - Reprocessed	Vial Position	71
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	3.10 min	1.3184	7.0434	0.0823 min	0.041 %
2	3.37 min	0.2446	1.7749	0.1066 min	0.010 %
3	3.51 min	0.1002	0.5075	0.0771 min	0.003 %
4	3.68 min	0.1318	0.8123	0.0959 min	0.005 %
5	4.36 min	0.9910	8.0495	0.1208 min	0.047 %
6	5.10 min	0.1142	1.0360	0.1259 min	0.006 %
7	6.25 min	0.4922	5.9663	0.1714 min	0.035 %
8	8.29 min	1433.2053	17090.1717	0.1831 min	99.695 %
9	10.01 min	0.1249	2.0492	0.2134 min	0.012 %
10	11.08 min	0.5656	8.8665	0.2029 min	0.052 %
11	11.57 min	0.5011	8.7308	0.2233 min	0.051 %
12	12.78 min	0.4887	7.3767	0.2421 min	0.043 %