



## BDG SYNTHESIS

### Certificate of Analysis

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

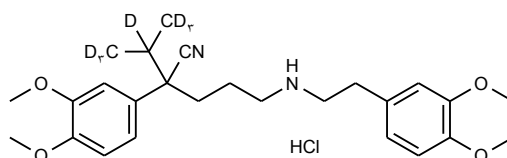
*Barry Dent*

Barry R. Dent, PhD, Director  
6 March 2012

**Name:** *N*-Desmethylverapamil-*d*<sub>7</sub> HCl

**CAS Number:** 67018-85-3 (unlabelled free base)

**Structure:**



**Molecular Weight:** C<sub>26</sub>H<sub>29</sub>D<sub>7</sub>N<sub>2</sub>O<sub>4</sub>·HCl = 484.08

**Lot Number:** BDG 13408.4

**Appearance:** White, crystalline solid

**Corrected Purity:** 100.0 % (HPLC) - 0.3 % (diethyl ether) = 99.7 %

**Isotopic Purity:** Under 0.5 % d<sub>0</sub>

**Re-test Date:** 6 March 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.

Isotopic Labelling: signals at the sites of deuteration are absent, compared with what would be expected for unlabelled material, indicating clean deuteration.

Residual Solvents: a small amount of diethyl ether (0.3 % w/w) and a trace (under 0.1 % w/w) of ethyl acetate 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.

Isotopic Labelling: signals at the sites of deuteration have collapsed to small multiplets compared with what would be expected for unlabelled material, indicating clean deuteration.

### High-resolution Mass Spectrum (ESI+)

Found  $m/z$  448.3186.  $C_{26}H_{30}D_7N_2O_4$   $[M+H]^+$  requires  $m/z$  448.3193. The deviation of 1.6 ppm is within normally accepted limits for the establishment of identity by HRMS. No signal for  $d_0$  material was seen (detection limit about 0.5 %).

### HPLC

A sharp, slightly tailing peak is observed (100.0 %). 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 64.21, H 6.32, D 2.95, N 5.97 %
$C_{26}H_{29}D_7N_2O_4 \cdot HCl$	Requires:	C 64.51, H 6.25, D 2.91, N 5.79 %

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).

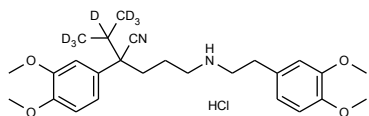
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

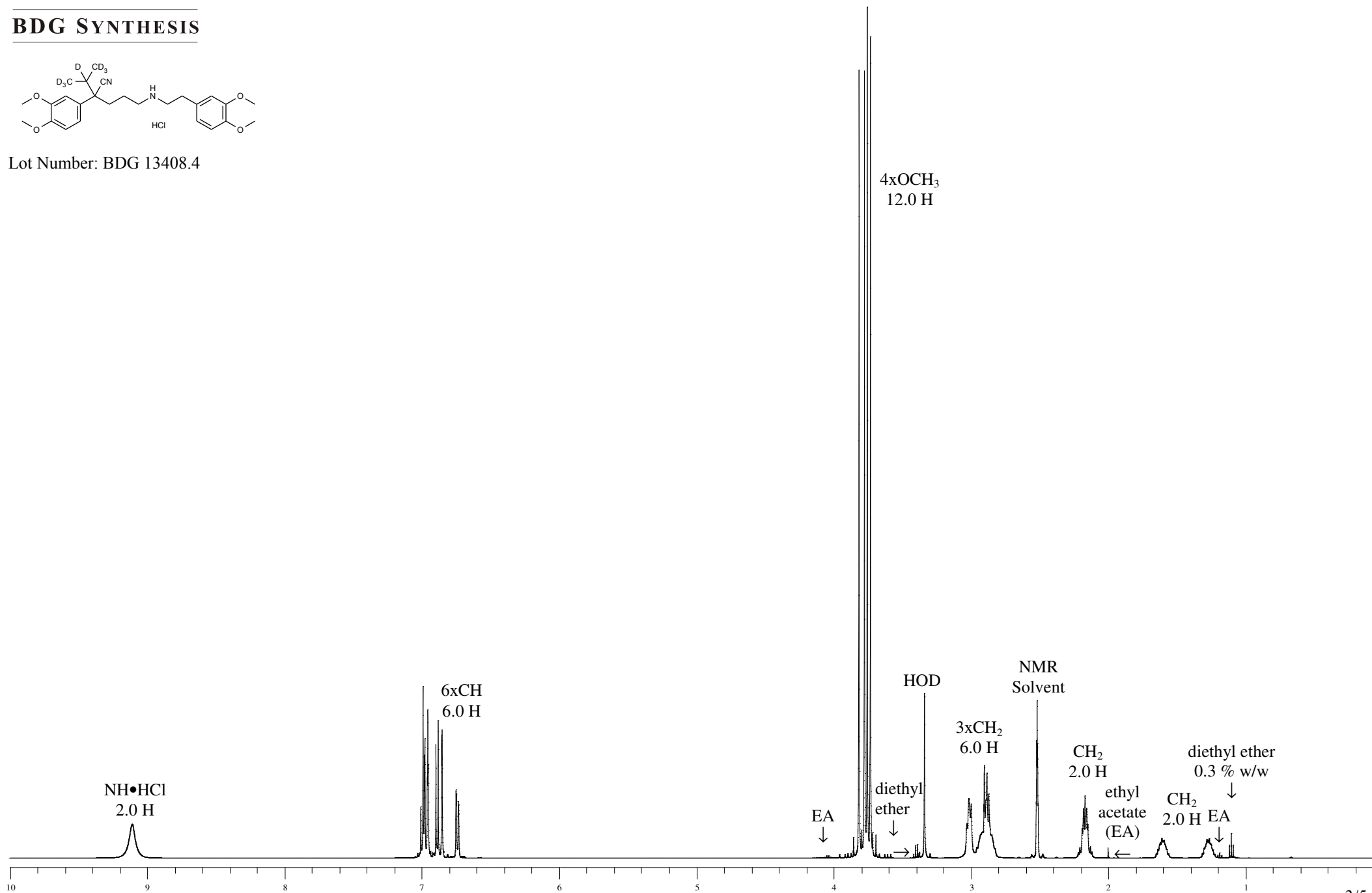


Proton NMR Spectrum of *N*-Desmethylverapamil- $d_7$  HCl in DMSO- $d_6$

**BDG SYNTHESIS**



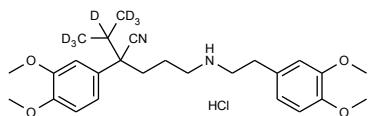
Lot Number: BDG 13408.4



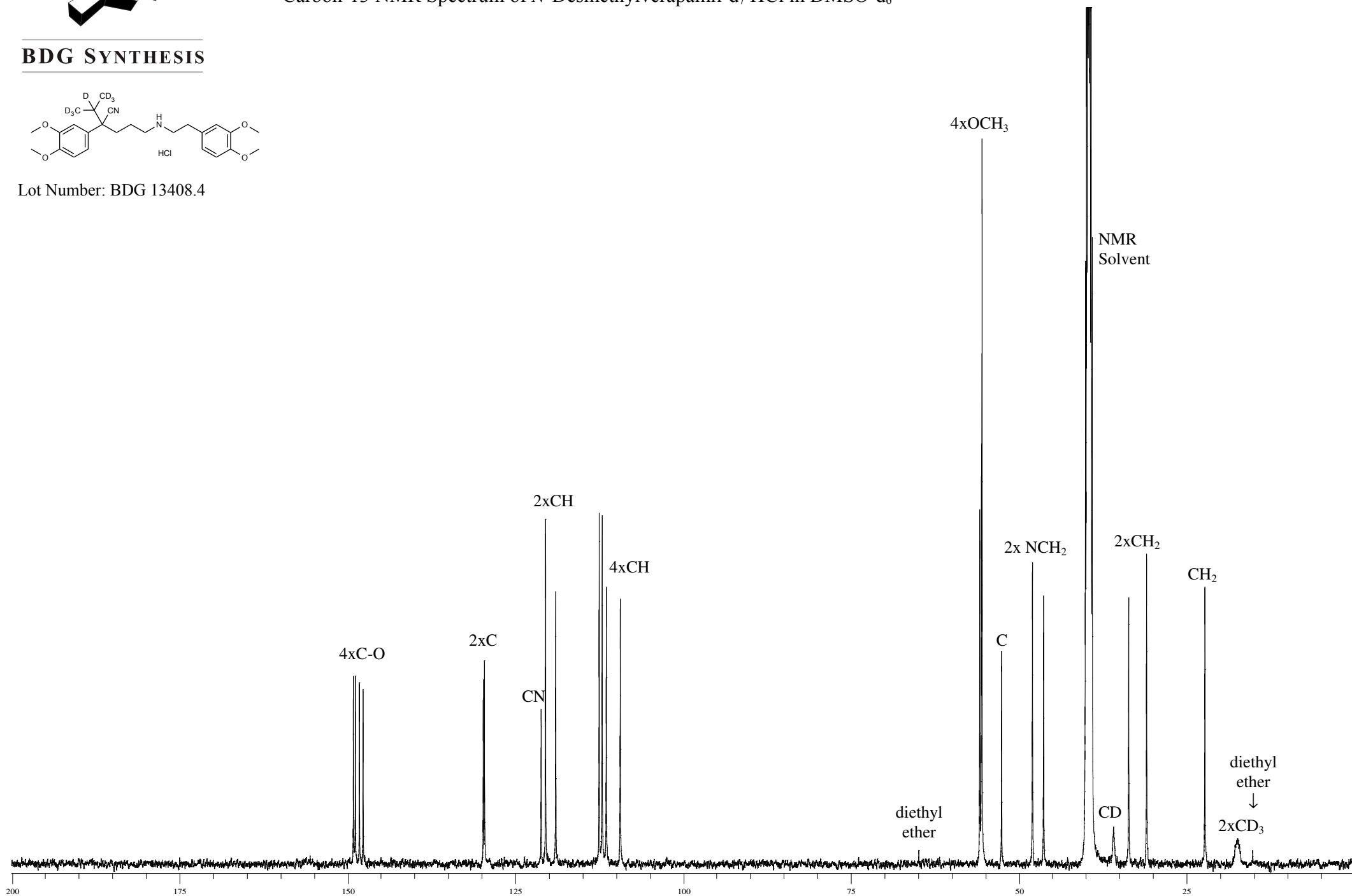


Carbon-13 NMR Spectrum of *N*-Desmethylverapamil- $d_7$  HCl in DMSO- $d_6$

**BDG SYNTHESIS**



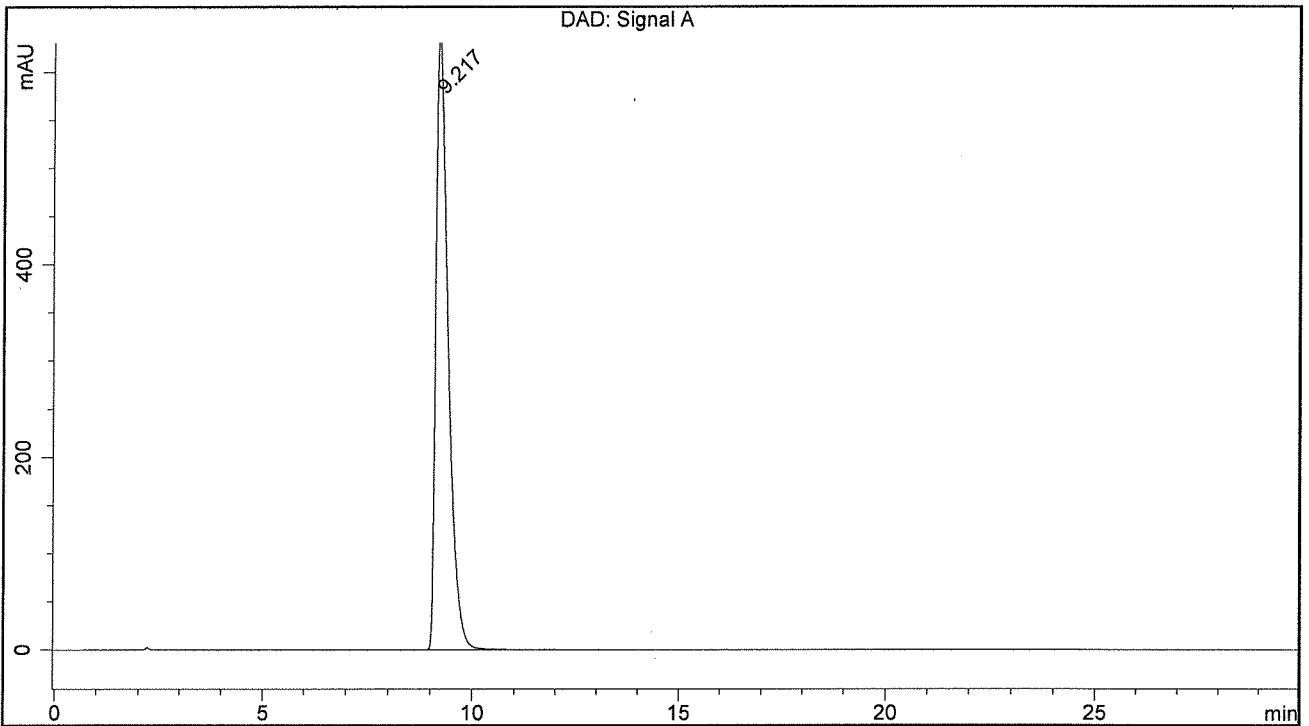
Lot Number: BDG 13408.4



BDG - Analysis of N-Desmethylverapamil-d7 HCl

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm  
 Guard : Phenomenex SecurityGuard C18 4 x 3mm  
 Mobile Phase : 40:40:20 20 mM Potassium diHydrogen Phosphate pH=7.0 : Acetonitrile : Methanol  
 Flow Rate : 1.0 mL/min  
 Sample Solvent : Mobile Phase  
 Column Temperature : 20C  
 Injection Volume : 10 uL  
 Detection : UV at 230 nm

<b>Sample Name</b>	BDG 13408.4	<b>Instrument</b>	AnalyticalLC01
<b>Acquisition</b>	06/03/2012, 20:19:12	<b>Method (rev.)</b>	LC10494a ( 5)
<b>Sequence</b>	BDG_06Mar2012i - Reprocessed	<b>Vial Position</b>	1
<b>Operator</b>	solvation010\cerityadmin	<b>Injection</b>	2 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	9.22 min	639.3261	13106.9616	0.3112 min	100.000 %