



## BDG SYNTHESIS

### Certificate of Analysis

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

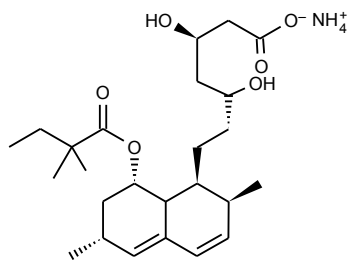
*Barry Dent*

Barry R. Dent, PhD, Director  
27 January 2010

**Name:** Simvastatin Acid Ammonium Salt

**CAS Number:** 139893-43-9

**Structure:**



**Molecular Weight:**  $C_{25}H_{39}O_6 \cdot NH_4 = 453.61$

**Lot Number:** BDG 2949

**Appearance:** White, crystalline solid

**Purity By HPLC:** 99.3 %

**Re-test Date:** 27 January 2011

**Storage and Handling:**

Temperature:	ambient laboratory temperature; may be refrigerated.
Humidity:	not believed to be hygroscopic; may be handled in normal laboratory atmosphere.
Light:	store in an amber vial and protect from bright light.
Caution:	only experienced laboratory personnel should handle the material. Avoid warming solutions - the material may revert to the lactone form.

## 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  $m/z$  435.2747.  $C_{25}H_{39}O_6$   $[M-NH_4]^+$  requires  $m/z$  435.2748. The deviation of 0.3 ppm is within normally accepted limits for the establishment of identity by HRMS.

### HPLC

A sharp, symmetrical peak is observed (99.3 %). 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

$C_{25}H_{39}O_6 \cdot NH_4$	Found:	C 66.23, H 9.69, N 3.06 %
	Requires:	C 66.19, H 9.55, N 3.09 %

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).

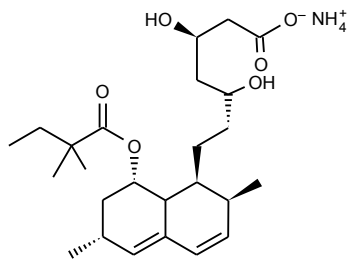
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

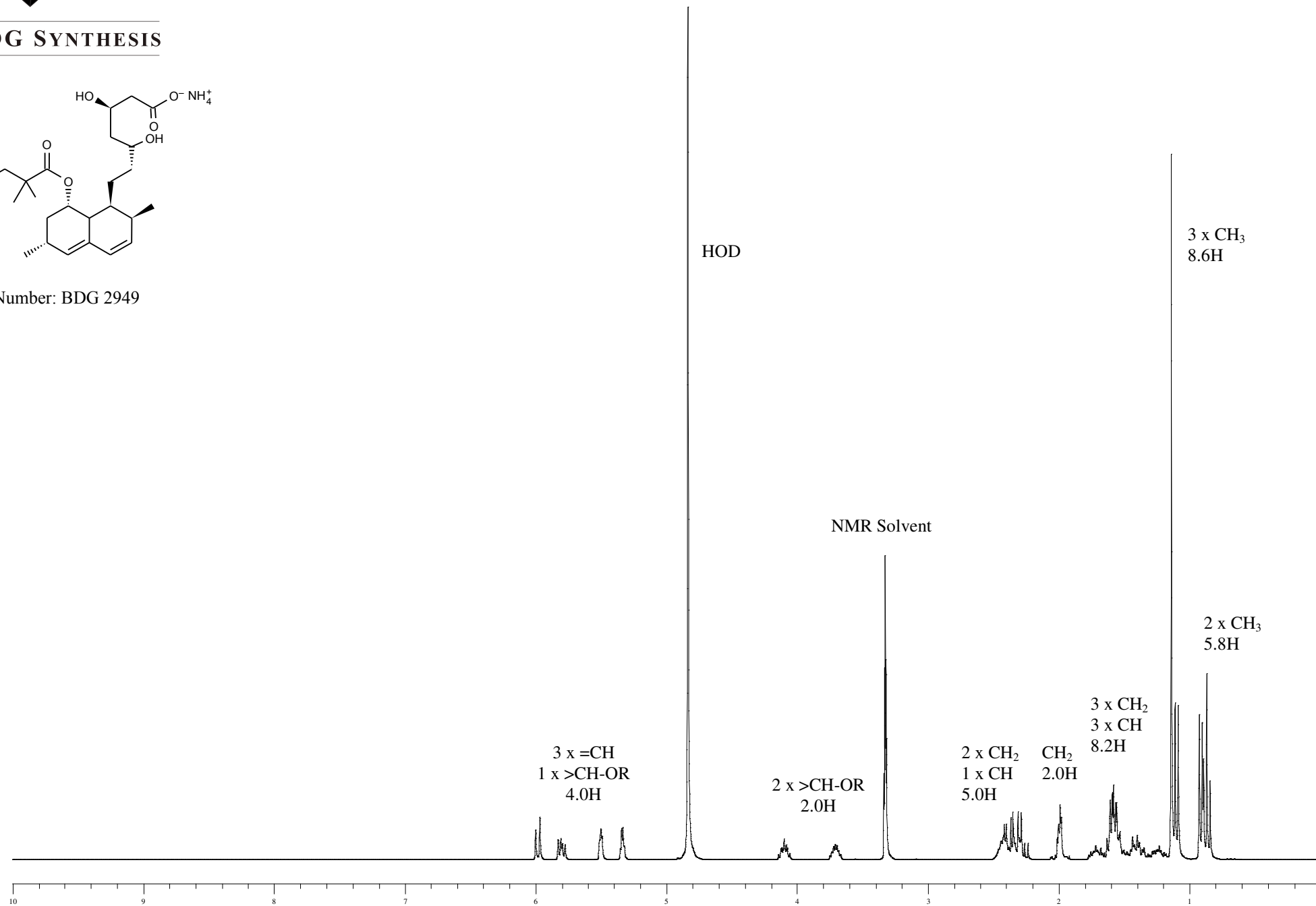


# Proton NMR Spectrum of Simvastatin Acid Ammonium Salt in Methanol-d<sub>4</sub>

## BDG SYNTHESIS



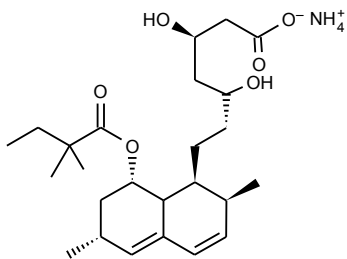
Lot Number: BDG 2949



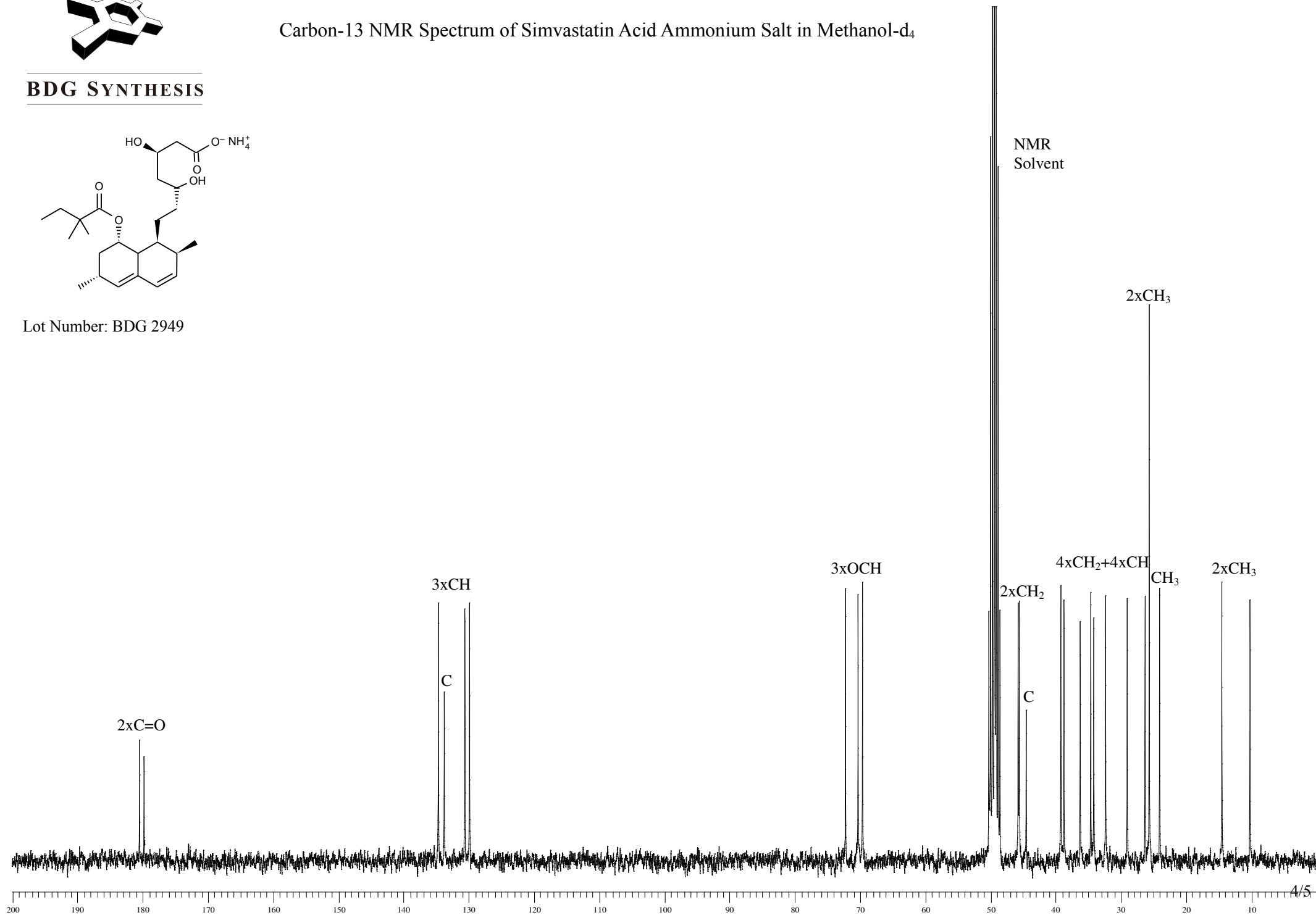


# Carbon-13 NMR Spectrum of Simvastatin Acid Ammonium Salt in Methanol-d<sub>4</sub>

**BDG SYNTHESIS**



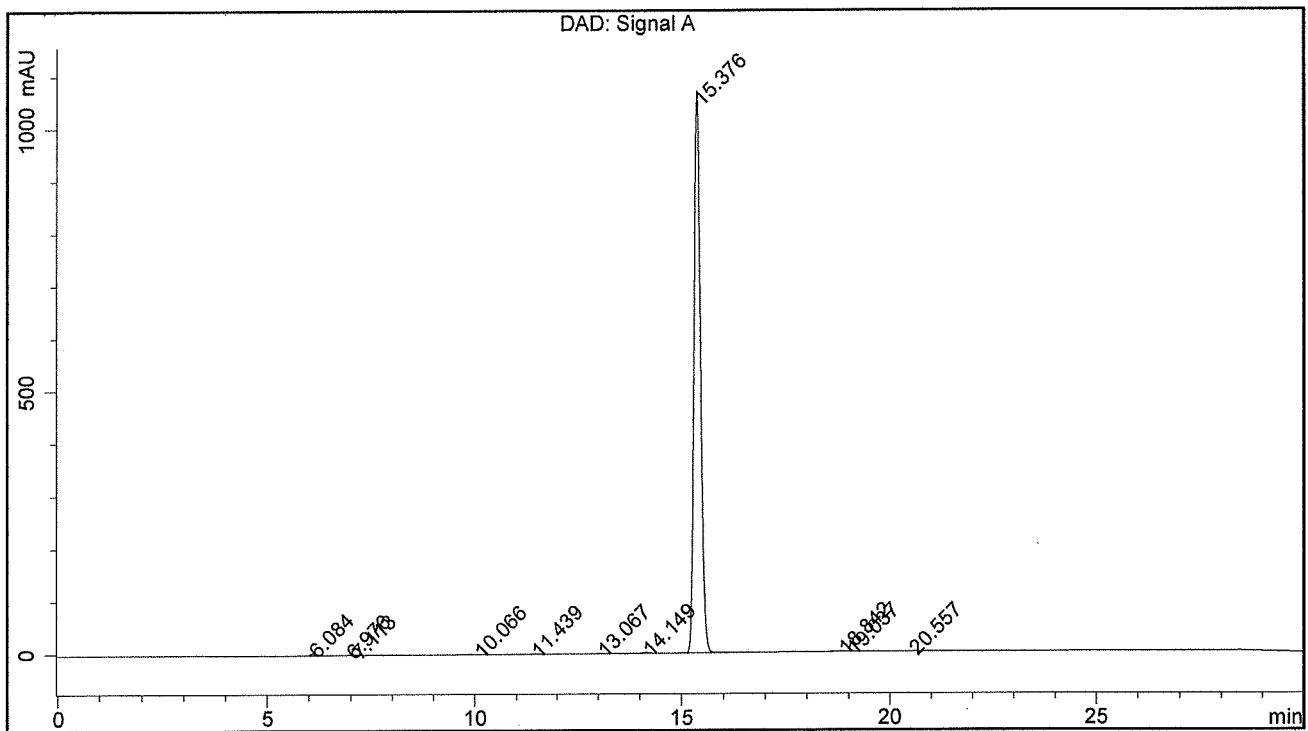
Lot Number: BDG 2949



BDG - Analysis of Simvastatin acid ammonium salt

Column : Phenomenex Luna C18 5um 250 x 4.6 mm  
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm  
 Mobile Phase A : 50:50 Water : Acetonitrile + 0.1% Phosphoric Acid  
 Mobile Phase B : 25:75 Water : Acetonitrile + 0.1% Phosphoric Acid  
 Gradient : T0=100:0, T15=0:100, T25=0:100, T27=100:0, T30=100:0  
 Flow Rate : 1.0 mL/min  
 Column Temperature : 20C  
 Sample Solvent : 40:60 10 mM KH2PO4 pH=4.0 : Acetonitrile  
 Run time : 30 mins  
 Detection : UV 238nm

Sample Name	BDG 2949	Instrument	AnalyticalLC01
Acquisition	27/01/2010, 21:16:10	Method (rev.)	LC10170c ( 3)
Sequence	BDG_27Jan2010a - Reprocessed	Vial Position	2
Operator	solvation010\cerityadmin	Injection	2 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	6.08 min	3.3224	22.1819	0.1019 min	0.191 %
2	6.98 min	0.3298	2.7803	0.1164 min	0.024 %
3	7.11 min	0.5016	3.6285	0.1064 min	0.031 %
4	10.07 min	0.3056	6.6612	0.2863 min	0.057 %
5	11.44 min	0.3505	4.0504	0.1688 min	0.035 %
6	13.07 min	0.3265	3.3185	0.1566 min	0.029 %
7	14.15 min	1.6388	17.9771	0.1660 min	0.155 %
8	15.38 min	1070.4664	11519.8176	0.1656 min	99.277 %
9	18.84 min	0.7697	12.4770	0.2251 min	0.108 %
10	19.04 min	0.4484	4.3787	0.1419 min	0.038 %
11	20.56 min	0.3328	6.3974	0.2723 min	0.055 %