BDG SYNTHESIS

Certificate of Analysis

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

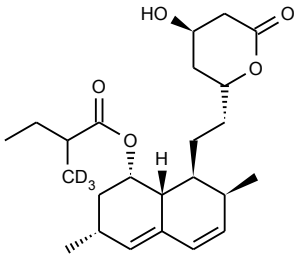
Barry Dent

Barry R. Dent, PhD, Director
1 October 2013

Name: **Lovastatin-d_3**

CAS Number: 75330-75-5 (unlabelled)

Structure:



Molecular Weight: $C_{24}H_{33}D_3O_5$ = 407.56

Lot Number: BDG 6633.1

Appearance: White, crystalline solid

Corrected Purity: 98.8 % (HPLC) - 0.3 % (chloroform) = 98.5 %

Isotopic Purity: Under 0.5 % d_0

Re-test Date: 1 October 2018

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 site of deuteration are absent, compared with what would be expected for unlabelled material, indicating clean deuteration.
Residual Solvents: a small amount of chloroform (0.3 % *w/w*) is 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. Some of the peaks are duplicated indicating that the product is a mixture of two diastereoisomers which is expected and is a consequence of the synthetic route used to generate the product.
Isotopic Labelling: signals at the site 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* 408.2817. $C_{24}H_{34}D_3O_5$ $[M+H]^+$ requires *m/z* 408.2829. The deviation of 3.1 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

Two overlapping peaks are observed (total integration = 98.8 area %). 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 70.61, H 8.18, D 1.49 %
$C_{24}H_{33}D_3O_5$	Requires:	C 70.73, H 8.16, D 1.48 %

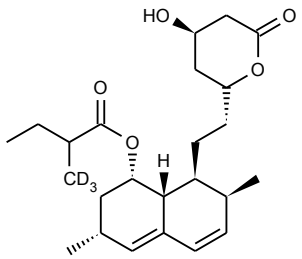
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

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.

BDG SYNTHESIS

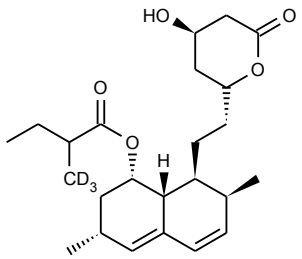
Proton NMR Spectrum of Lovastatin-d_3 in DMSO-d_6



Lot Number: BDG 6633.1

BDG SYNTHESIS

Carbon-13 NMR Spectrum of Lovastatin-d_3 in DMSO-d_6



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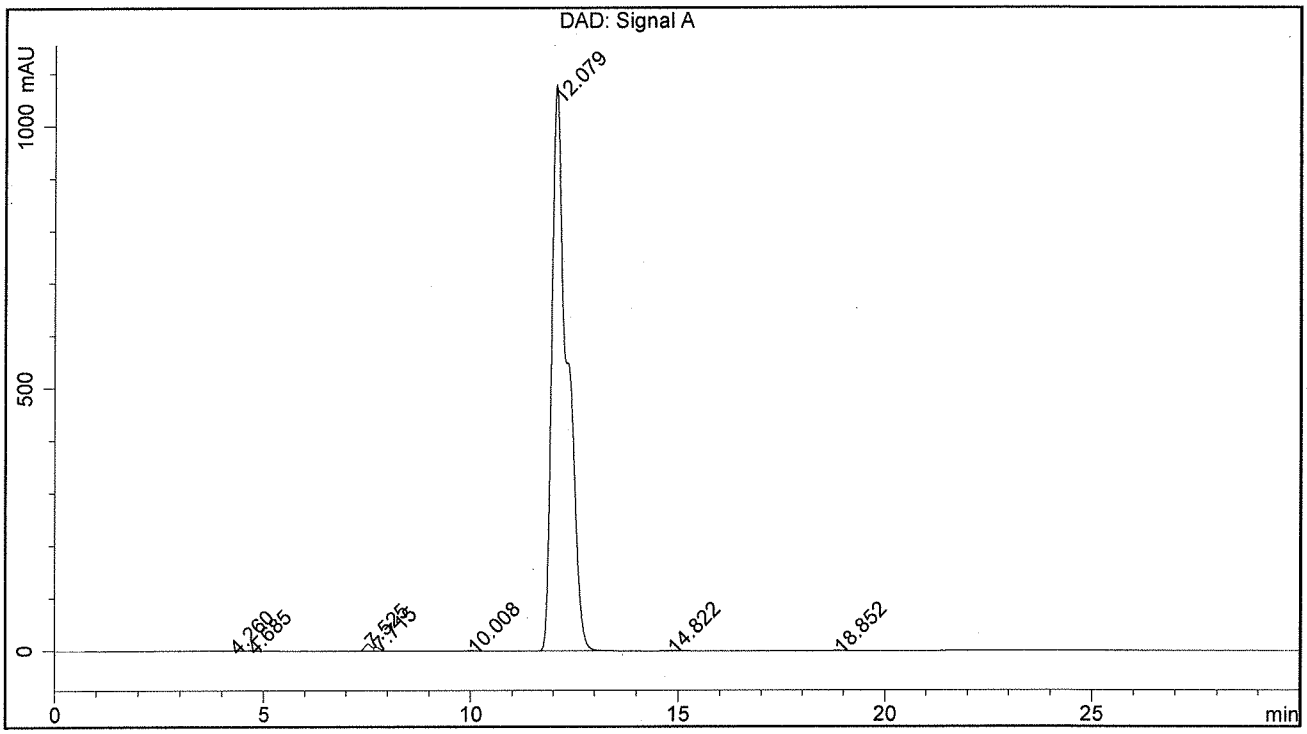
BDG - Analysis of Lovastatin-d3

Column : Phenomenex Luna C8 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase A : 40:60:0.1 Water : Acetonitrile : Phosphoric Acid
Mobile Phase B : 10:90 Water : Acetonitrile
Gradient : T0=100:0, T10=100:0, T20=0:100, T30=0:100, T31=100:0, T35=100:0
Flow Rate : 1.0 mL/min
Column Temperature : 20C
Sample Solvent : 40:60 Water : Acetonitrile
Run time: 30 minutes
Detection: UV 238 nm

Sample Name	BDG 6633.1	**Instrument**	AnalyticalLC01
Acquisition	01/10/2013, 17:36:35	**Method (rev.)**	LC10121b (4)
Sequence	BDG_01Oct2013b	**Vial Position**	1
Operator	solvation010\cerityadmin	**Injection**	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	4.26 min	0.8086	6.8257	0.1265 min	0.024 %
2	4.68 min	1.4093	17.8629	0.1739 min	0.063 %
3	7.53 min	13.6483	158.1201	0.1732 min	0.556 %
4	7.71 min	7.2317	77.5560	0.1591 min	0.273 %
5	10.01 min	1.1671	19.9657	0.2496 min	0.070 %
6	12.08 min	1079.4098	28103.8003	0.3658 min	98.811 %
7	14.82 min	1.2134	34.5560	0.3943 min	0.121 %
8	18.85 min	1.4742	23.3963	0.2312 min	0.082 %