



BDG SYNTHESIS

Certificate of Analysis

This material is a research-grade material prepared by custom synthesis. The quantity available is limited, 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 research-grade materials. Research 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.

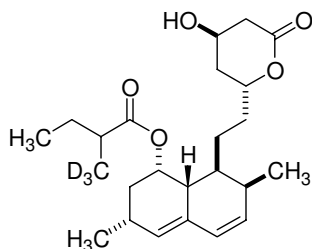
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated in this data sheet.

Barry Dent

Barry R. Dent, PhD, Director
18 January 2007

Name: Lovastatin-d₃
CAS Number: none (75330-75-5 unlabelled)

Structure:



Molecular Weight: C₂₄H₃₃D₃O₅ = 407.55
Lot Number: BDG 6633.1
Appearance: White, crystalline solid
Corrected Purity: 97.9 % (HPLC) – 0.3 % (chloroform) = 97.6 %
Isotopic Purity: Under 0.5 % d₀
Expiry Date: 18 January 2012

This expiry date is assigned from experience gained with the material in the laboratory and/or on storage. It is not possible to perform formal storage stability studies because of the small amount of material available.

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.

Identity and Purity:

Source of Material

The material was made by an unambiguous synthetic route, using literature procedures where possible; starting materials were purchased from reputable sources and all intermediates were checked for identity by NMR.

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 greatly diminished, 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. The relative intensities of these duplicated peaks are close to that seen for the two main peaks in the HPLC trace.

Isotopic labelling: the signal at the site of deuteration has collapsed to a small multiplet compared with what would be expected for unlabelled material, indicating clean deuteration.

High-resolution mass spectrum (FAB+): 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 %).

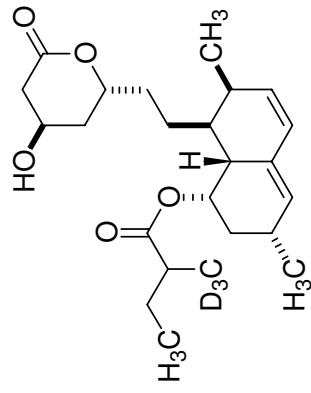
HPLC: Two sharp, overlapping peaks are observed (total integration = 97.9 area %). Note: in the absence of reference materials for preparing calibration curves, it is assumed that all peaks have the same detector response. Where possible, the conditions of analysis follow a pharmacopeial or literature method, or have been adapted from same.

Elemental Analysis: Found: C 70.61, H 8.18, N 1.49 %
 $C_{24}H_{33}D_3O_5$ requires: C 70.73, H 8.16, D 1.48 %

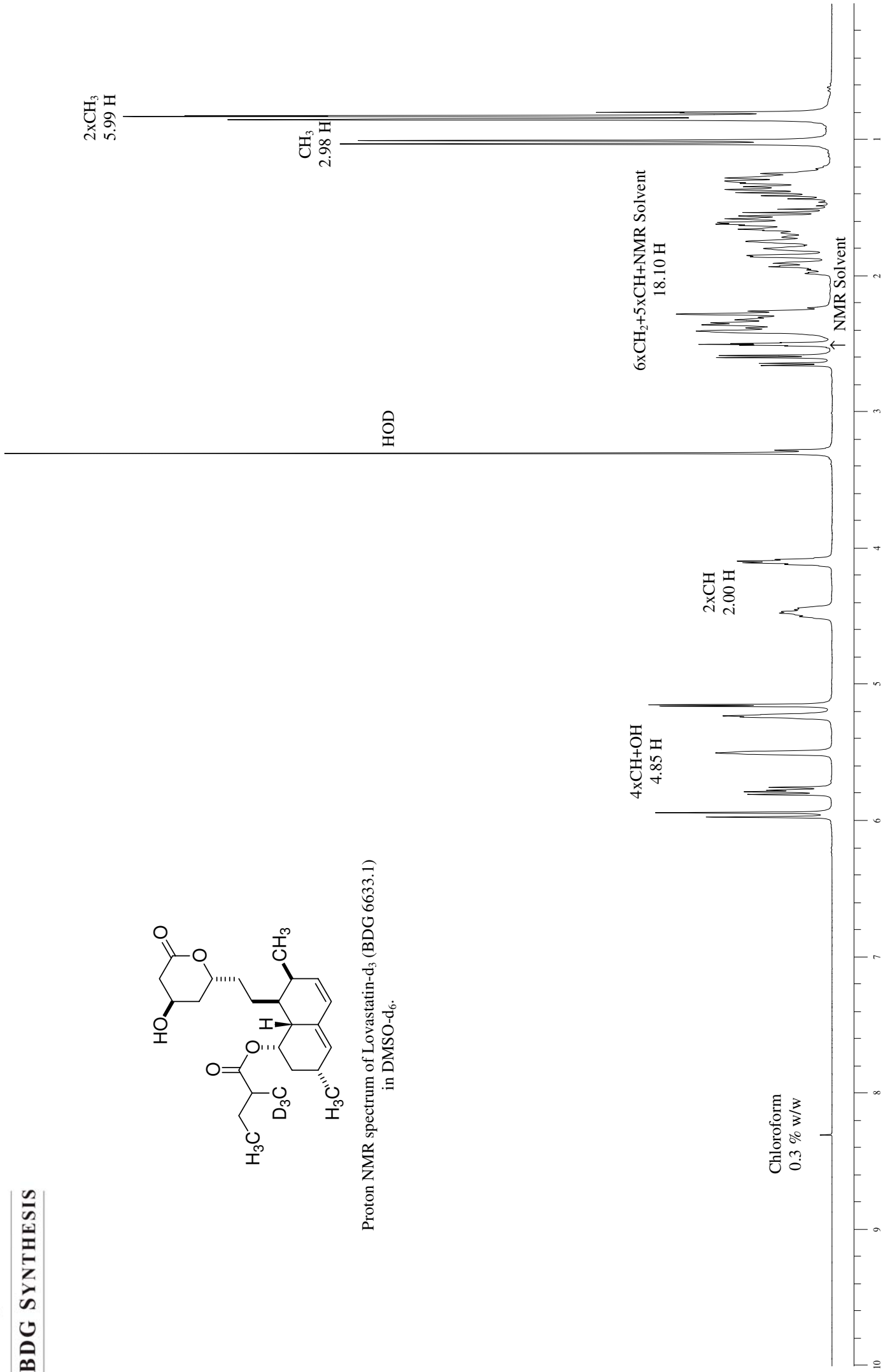
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).



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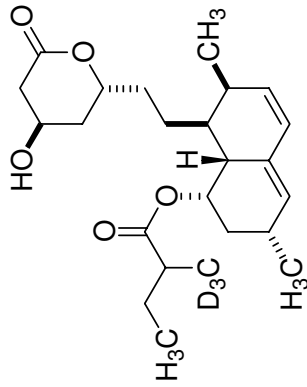


Proton NMR spectrum of Lovastatin-d₃ (BDG 6633.1) in DMSO-d₆.

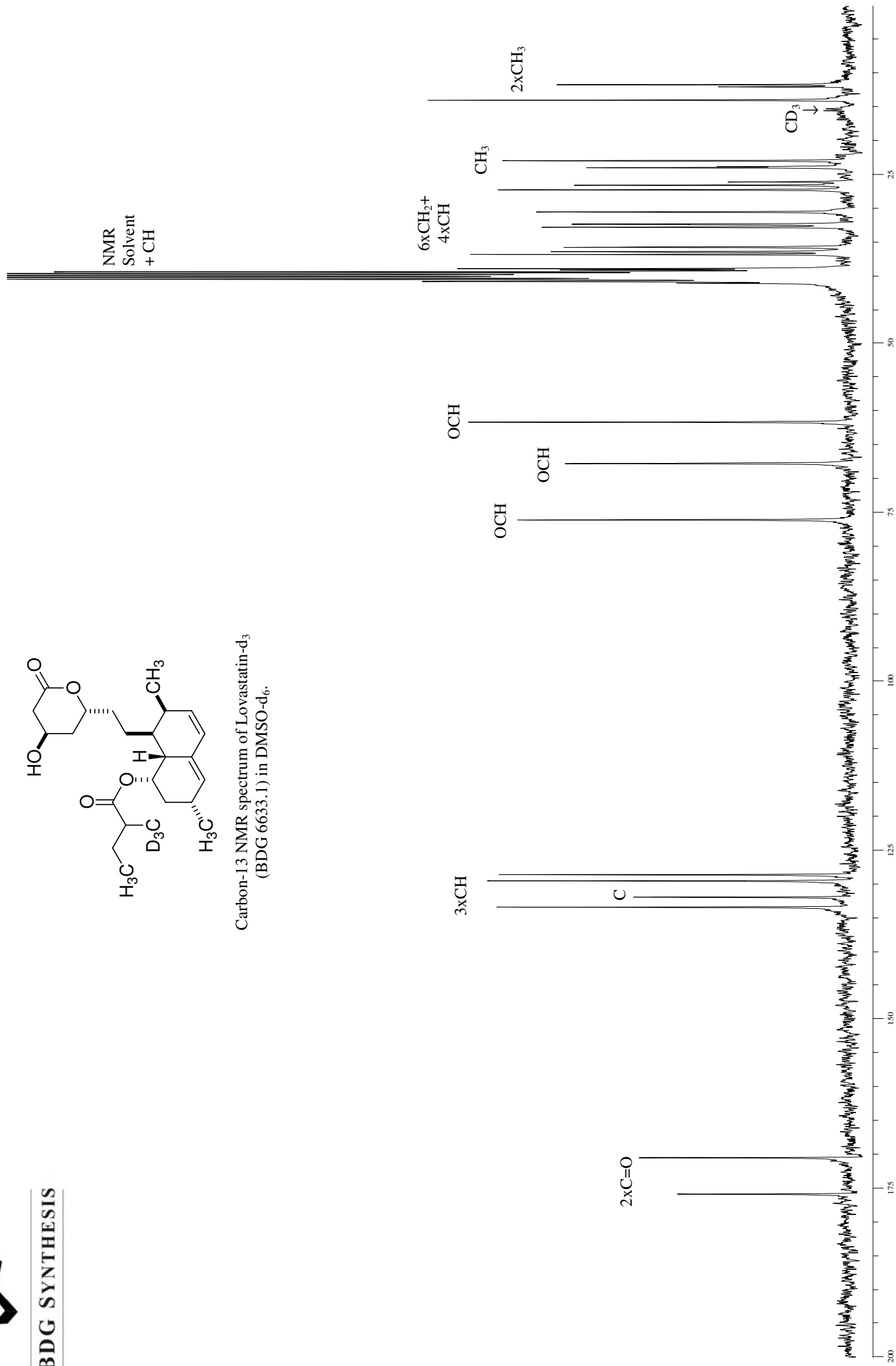




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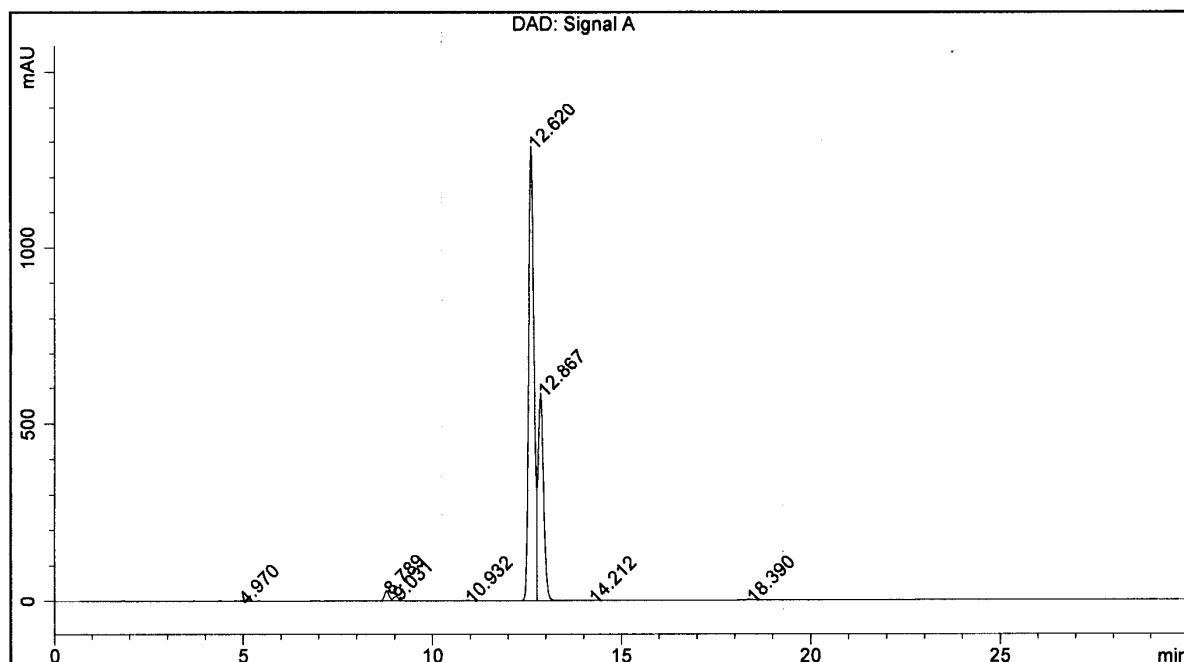
Carbon-13 NMR spectrum of Lovastatin-d₃
(BDG 6633.1) in DMSO-d₆.



BDG - Analysis of Lovastatin-d3

Column : Phenomenex Luna C18 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase A : Water/ACN 50/50 & 0.1% H3P04
 Mobile Phase B : Acetonitrile & 0.1% H3P04
 Gradient : T0=80:20, T15=30:70, T30=30:70, T35=80:20, T40=80:20
 Flow Rate : 1.0 mL/min
 Column Temperature : 20C
 Sample Solvent : Solvent A
 Run time: 40 mins
 Detection: UV 238nm

Sample Name	BDG 6633.1	Instrument	AnalyticalLC01
Acquisition	13/12/2006, 17:42:01	Method (rev.)	LC10121a
Sequence	BDG_13Dec2006d	Vial Position	77
Operator	LC10121a	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	4.97 min	0.5584	4.5235	0.1285 min	0.024 %
2	8.79 min	28.2788	244.5496	0.1349 min	1.297 %
3	9.03 min	12.7494	117.5941	0.1376 min	0.624 %
4	10.93 min	1.3008	14.8005	0.1747 min	0.078 %
5	12.62 min	1286.4059	12564.3494	0.1499 min	66.633 %
6	12.87 min	585.1209	5890.1891	0.1514 min	31.238 %
7	14.21 min	0.5883	6.9886	0.1846 min	0.037 %
8	18.39 min	1.2551	13.0886	0.1617 min	0.069 %