

BDG SYNTHESIS

Certificate of Analysis

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

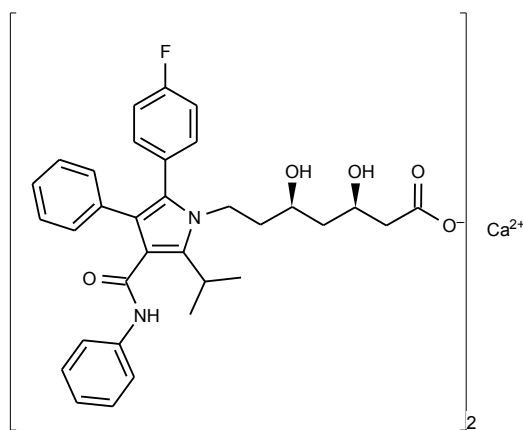
Barry Dent

Barry R. Dent, PhD, Director
12 February 2010

Name: Atorvastatin Calcium

CAS Number: 344423-98-9

Structure:



Molecular Weight: $2C_{33}H_{34}FN_2O_5 \cdot Ca = 1155.34$

Lot Number: BDG 4182

Appearance: Off-white, crystalline solid

Corrected Purity: 97.3 % (HPLC) - 4.6 % (water) = 92.7 %

Re-test Date: 12 February 2011

Storage and Handling:

| | |
|--------------|--|
| Temperature: | refrigerate for prolonged storage; may be handled and shipped at ambient temperature. |
| Humidity: | may be hygroscopic; store desiccated; recommended to determine water content periodically. |
| 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 (FAB-)

Found m/z 1153.4453. $C_{66}H_{67}CaF_2N_4O_{10}$ $[M-H]^-$ requires m/z 1153.4451. The deviation of 0.2 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A sharp, symmetrical peak is observed (97.3 %). 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

| | | |
|---|-----------|---------------------------|
| | Found: | C 66.58, H 6.11, N 4.85 % |
| $2C_{33}H_{34}FN_2O_5 \cdot Ca \cdot 2.0H_2O$ | Requires: | C 66.54, H 6.09, N 4.70 % |
| $2C_{33}H_{34}FN_2O_5 \cdot Ca$ | Requires: | C 68.61, H 5.93, N 4.85 % |

The elemental analyses fall substantially outside those expected for anhydrous material; the presence of water is reasonably expected from the method of purification and/or the type of material, and the "best-fit" hydrated molecular formula is given.

Karl-Fischer Analysis

| | | |
|---|-----------|------------------------|
| | Found: | H ₂ O 4.6 % |
| $2C_{33}H_{34}FN_2O_5 \cdot Ca \cdot 2.0H_2O$ | Requires: | H ₂ O 3.0 % |

Of necessity, only a small sample could be used and only a single or duplicate analysis performed. We are unable to state what the errors in the reported water content are, but recommend that the result be used, as the best available, when determining corrected purity.

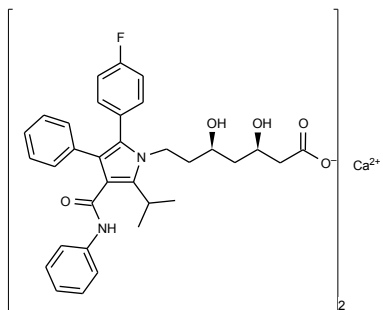
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

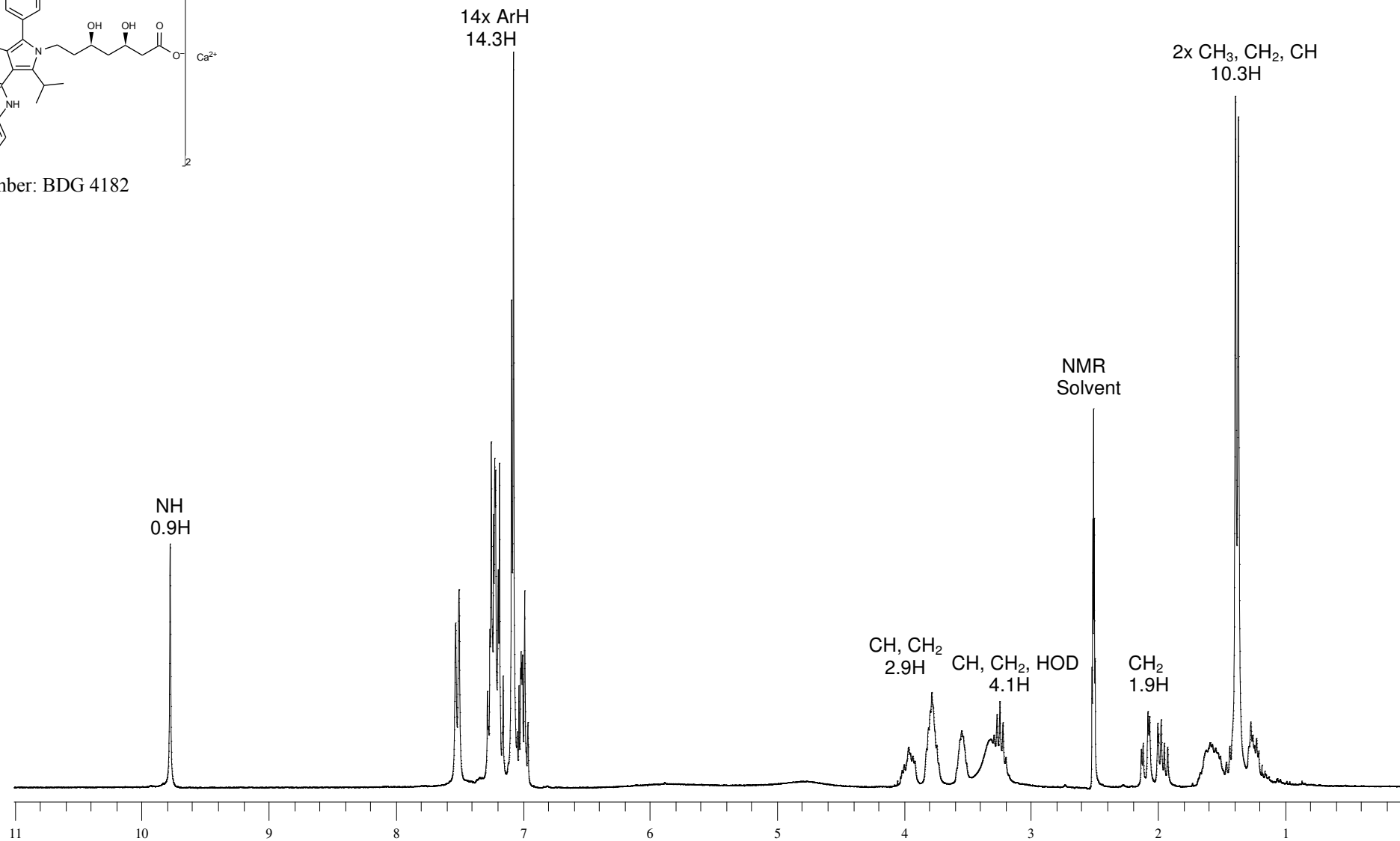


Proton NMR Spectrum of Atorvastatin Calcium in DMSO-d₆

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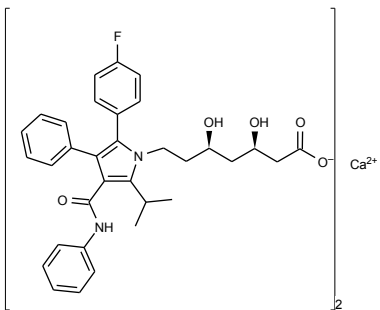
Lot Number: BDG 4182





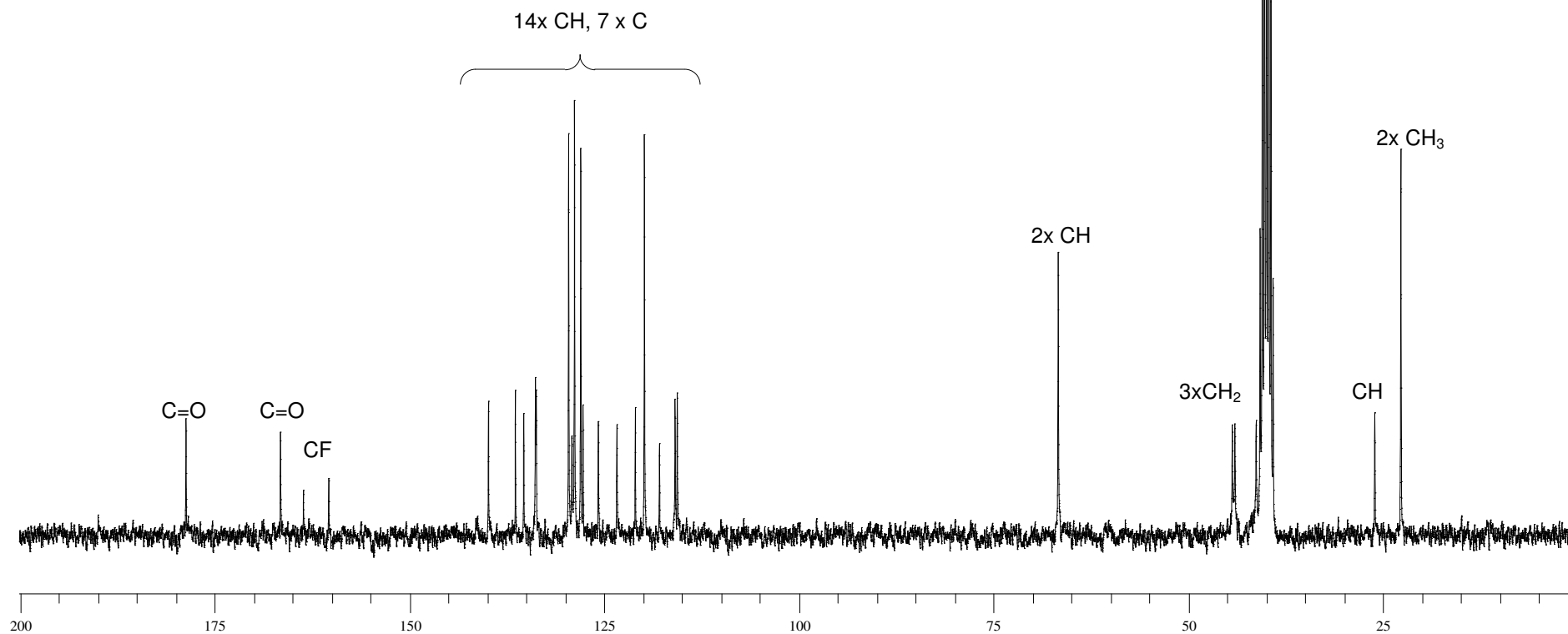
Carbon-13 NMR Spectrum of Atorvastatin Calcium in DMSO-d₆

BDG SYNTHESIS



Lot Number: BDG 4182

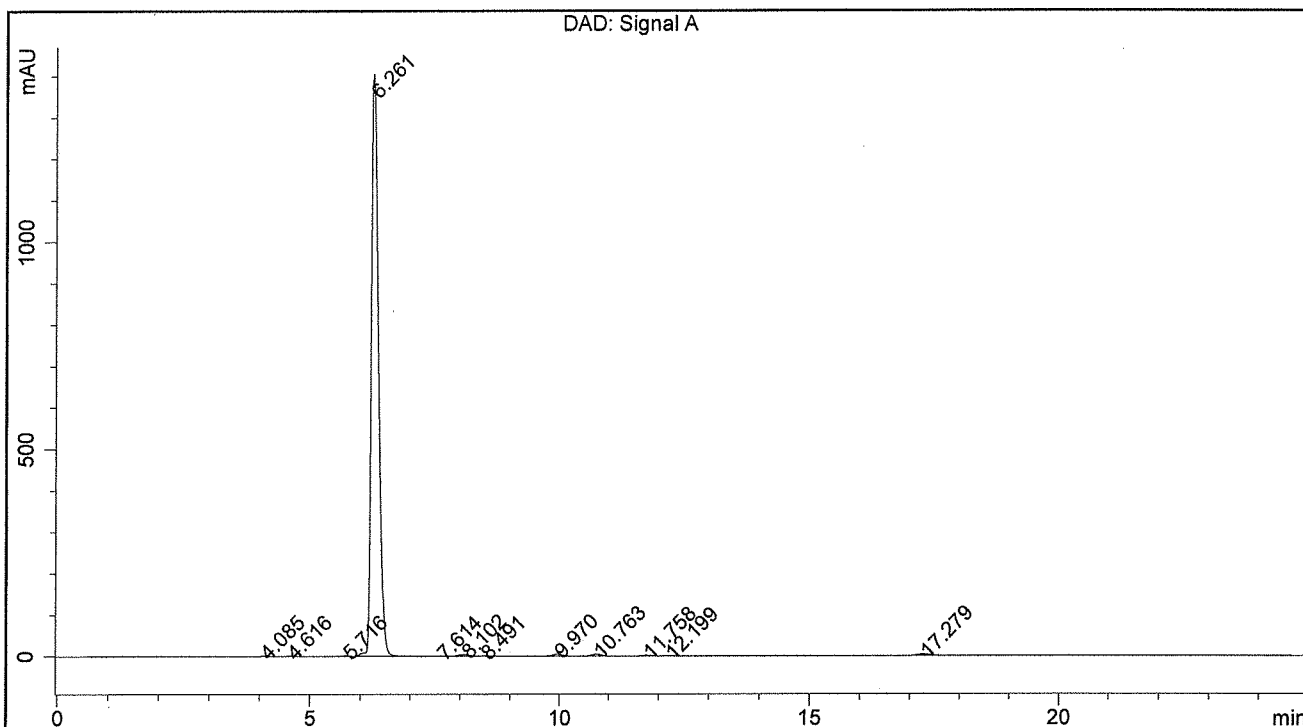
NMR Solvent
+ CH₂



BDG - Analysis of Atorvastatin calcium salt

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase : 35:65 0.1% Acetic Acid : Acetonitrile
 Flow Rate : 1.0 mL/min
 Sample Solvent : Mobile Phase
 Column Temperature : 20C
 Injection Volume : 10 uL
 Detection : UV at 250 nm

| | | | |
|--------------------|--------------------------|----------------------|----------------|
| Sample Name | BDG 4182 | Instrument | AnalyticalLC01 |
| Acquisition | 12/02/2010, 08:47:12 | Method (rev.) | LC10123f (2) |
| Sequence | BDG_12Feb2010a | Vial Position | 1 |
| Operator | solvation010\cerityadmin | Injection | 1 of 1 |



Area Percent Report

| Peak# | RT | Peak Height | Peak Area | Width | Area % |
|-------|-----------|-------------|------------|------------|----------|
| 1 | 4.08 min | 1.2940 | 12.5965 | 0.1376 min | 0.087 % |
| 2 | 4.62 min | 1.9794 | 14.1616 | 0.1095 min | 0.098 % |
| 3 | 5.72 min | 1.4596 | 14.7367 | 0.1518 min | 0.102 % |
| 4 | 6.26 min | 1405.7819 | 14017.1895 | 0.1523 min | 97.286 % |
| 5 | 7.61 min | 0.5856 | 5.9126 | 0.1558 min | 0.041 % |
| 6 | 8.10 min | 3.7737 | 58.3178 | 0.2523 min | 0.405 % |
| 7 | 8.49 min | 1.0611 | 13.7905 | 0.1956 min | 0.096 % |
| 8 | 9.97 min | 5.1570 | 74.7898 | 0.2192 min | 0.519 % |
| 9 | 10.76 min | 4.7734 | 67.6945 | 0.2195 min | 0.470 % |
| 10 | 11.76 min | 2.1728 | 31.2914 | 0.2220 min | 0.217 % |
| 11 | 12.20 min | 1.0585 | 18.2060 | 0.2587 min | 0.126 % |
| 12 | 17.28 min | 3.7818 | 79.4740 | 0.3232 min | 0.552 % |