



BDG SYNTHESIS

Certificate of Analysis

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

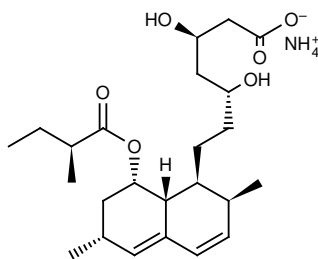
Neil Beare

Neil Beare, PhD, Director
1 August 2017

Name: Lovastatin Acid Ammonium Salt

CAS Number: 77550-67-5

Structure:



Molecular Weight: $C_{24}H_{37}O_6 \cdot NH_4 = 439.59$

Lot Number: BDG 5748

Appearance: White, crystalline powder

Purity By HPLC: 96.8 %

Re-test Date: 1 August 2018

Storage and Handling: Temperature: Freeze (-20°C) 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: 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: a trace of an unidentified impurity is seen in the baseline.

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 421.2590. $C_{24}H_{37}O_6 [M+NH_4]^+$ requires m/z 421.2596. The deviation of 1.2 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A sharp, symmetrical peak is observed (96.8 %). 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 65.52, H 9.44, N 3.17 %
$C_{24}H_{37}O_6 \cdot NH_4$	Requires:	C 65.57, H 9.40, N 3.19 %

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).

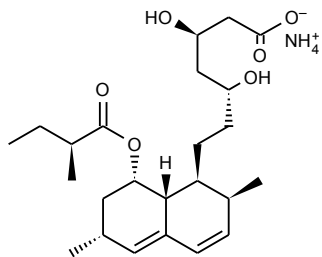
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

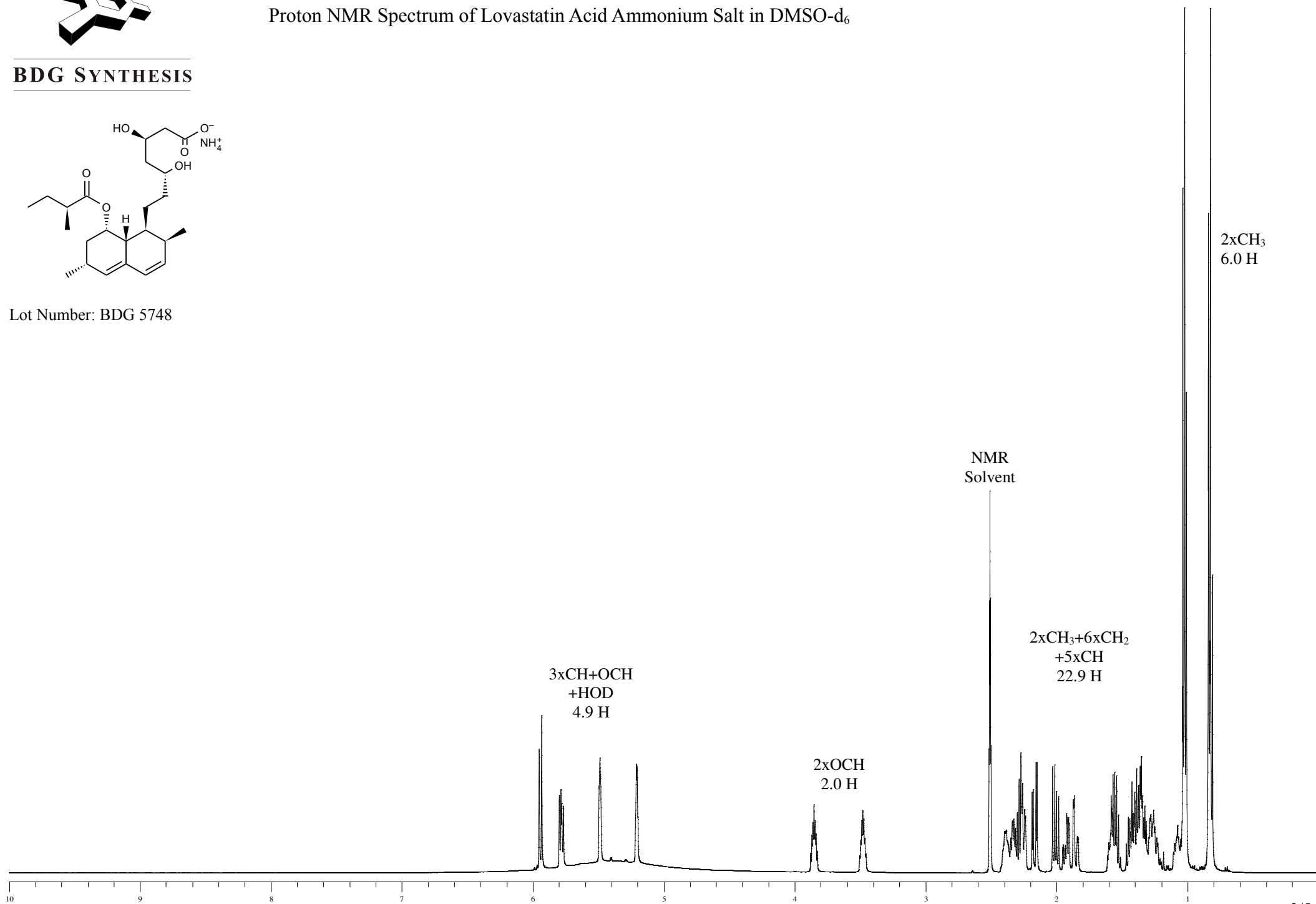


Proton NMR Spectrum of Lovastatin Acid Ammonium Salt in DMSO-d₆

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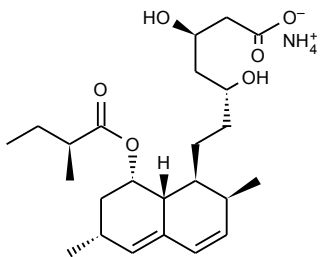
Lot Number: BDG 5748



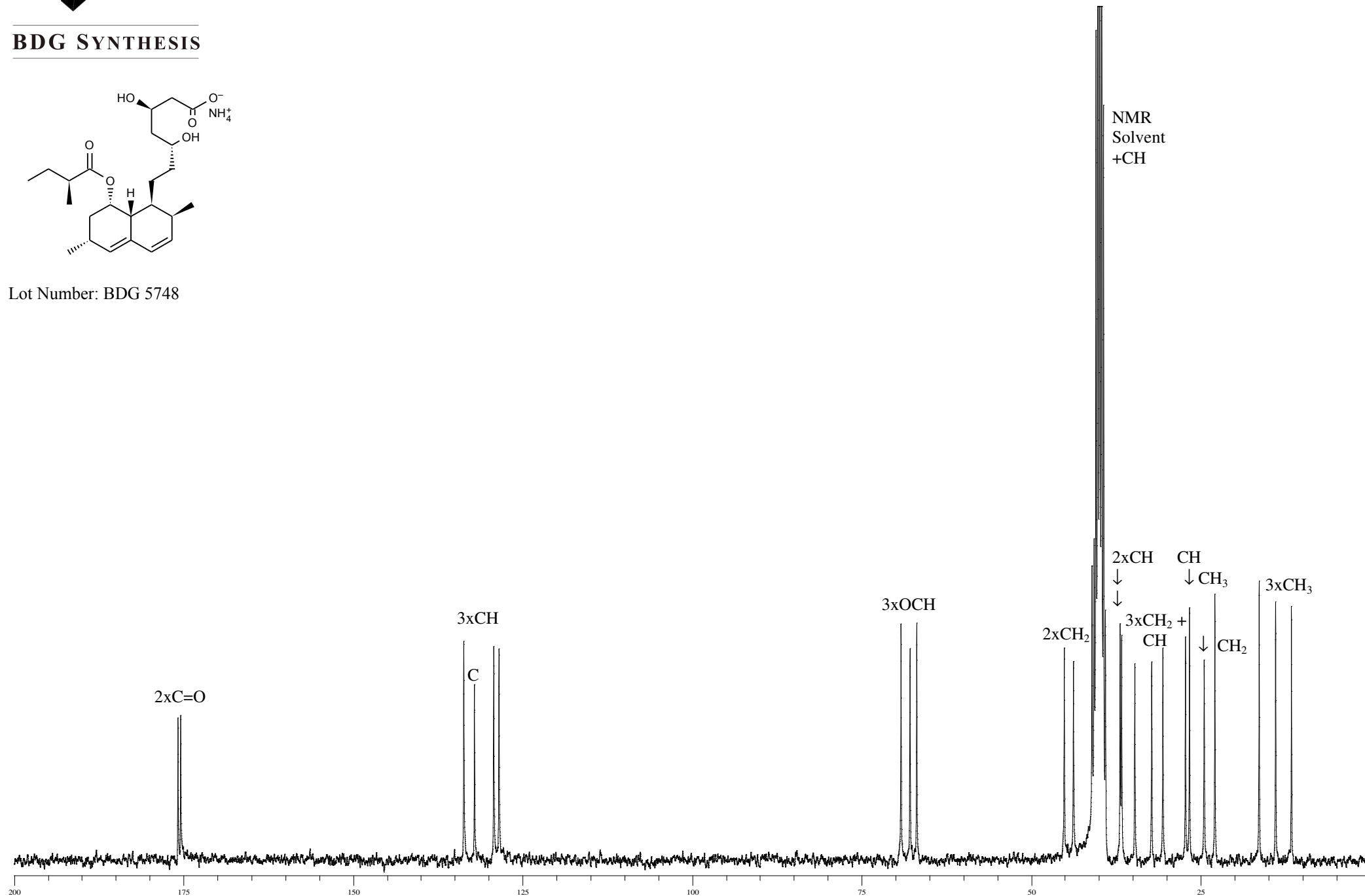


Carbon-13 NMR Spectrum of Lovastatin Acid Ammonium Salt in DMSO-d₆

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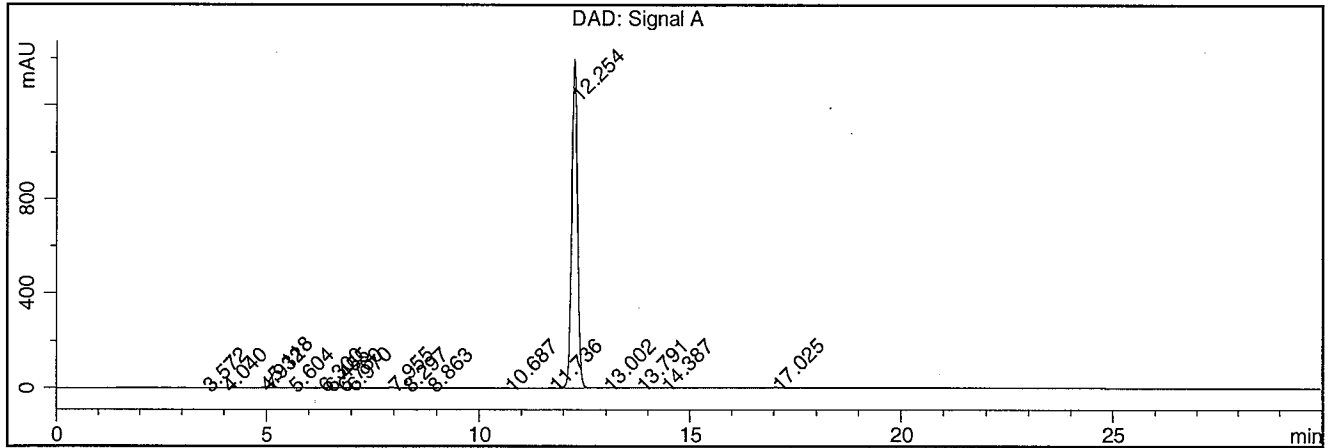
Lot Number: BDG 5748



BDG - Analysis of Lovastatin Acid Ammonium Salt

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18(2) RP 4 x 3 mm
 Mobile Phase A : 50:50:0.1 Water : Acetonitrile : Phosphoric Acid
 Mobile Phase B : 25:75:0.1 Water : Acetonitrile : Phosphoric Acid
 Gradient : T0=100:0, T15=0:100, T20=0:100, T25=100:0, T30=100:0
 Sample Solvent : 50:50 10 mM KH2PO4 pH=3.0 : Acetonitrile
 Flow Rate : 1.0 mL/min Column Temperature : 20 C Detection UV 238 nm

Sample Name	BDG 5748	Instrument	AnalyticalLC01
Acquisition	01/08/2017, 10:47:33	Method (rev.)	LC10120d (16)
Sequence	BDG_01Aug2017a - Reprocessed	Vial Position	1
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	3.57 min	0.5518	3.7647	0.1076 min	0.027 %
2	4.04 min	0.8346	4.6536	0.0870 min	0.034 %
3	4.93 min	1.7179	7.8493	0.0733 min	0.057 %
4	5.12 min	46.0702	300.8573	0.1022 min	2.177 %
5	5.60 min	0.3245	2.3641	0.0990 min	0.017 %
6	6.30 min	0.6955	5.6476	0.1167 min	0.041 %
7	6.46 min	0.3194	2.0188	0.0997 min	0.015 %
8	6.76 min	0.3564	3.4760	0.1438 min	0.025 %
9	6.97 min	1.2144	8.9162	0.1137 min	0.065 %
10	7.95 min	1.2708	17.9459	0.1908 min	0.130 %
11	8.30 min	0.3781	3.1416	0.1229 min	0.023 %
12	8.86 min	0.5544	5.8696	0.1655 min	0.042 %
13	10.69 min	0.4472	4.0773	0.1284 min	0.030 %
14	11.74 min	0.5083	4.0791	0.1316 min	0.030 %
15	12.25 min	1396.6195	13376.8704	0.1497 min	96.808 %
16	13.00 min	0.6039	17.6908	0.3558 min	0.128 %
17	13.79 min	1.0272	9.6346	0.1474 min	0.070 %
18	14.39 min	0.6434	9.0580	0.1903 min	0.066 %
19	17.02 min	2.7560	30.0388	0.1672 min	0.217 %