



## BDG SYNTHESIS

### Certificate of Analysis

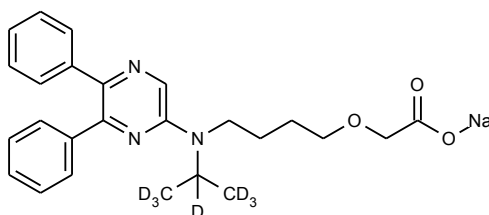
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

*Neil Beare*

Neil Beare, PhD, Director  
30 March 2017

**Name:** MRE-269-d<sub>7</sub> Sodium Salt  
**CAS Number:** 475085-57-5 (unlabelled acid)

**Structure:**



**Molecular Weight:** C<sub>25</sub>H<sub>21</sub>D<sub>7</sub>N<sub>3</sub>NaO<sub>3</sub> = 448.54  
**Lot Number:** BDG 16866.5  
**Appearance:** Off-white, crystalline solid  
**Corrected Purity:** 99.8 % (HPLC) - 2.0 % (water) = 97.8 %  
**Isotopic Purity:** Under 0.5 % d<sub>0</sub>  
**Re-test Date:** 30 March 2022  
**Storage and Handling:** Temperature: refrigerate for prolonged storage; may be handled and shipped at ambient temperature.  
Humidity: hygroscopic; open under dry conditions; store desiccated; recommended to flush container with dry nitrogen before re-sealing; 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.

Isotopic Labelling: signals at the sites of deuteration are absent, compared with what would be expected for unlabelled material, indicating clean deuteration.

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.

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 (TOF MS ES+)

Found  $m/z$  427.2729.  $C_{25}H_{23}D_7N_3O_3$   $[M-Na+2H]^+$  requires  $m/z$  427.2721. The deviation of 1.8 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

A sharp, slightly tailing peak is observed (99.8 %). 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 65.65, H 4.95, D 3.15, N 9.29 %
$C_{25}H_{21}D_7N_3NaO_3 \cdot 0.5H_2O$	Requires:	C 65.63, H 4.85, D 3.08, N 9.18 %
$C_{25}H_{21}D_7N_3NaO_3$	Requires:	C 66.94, H 4.72, D 3.14, N 9.37 %

The elemental analyses fall somewhat 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 <sub>2</sub> O 2.0 %
$C_{25}H_{21}D_7N_3NaO_3 \cdot 0.5H_2O$	Requires:	H <sub>2</sub> O 2.0 %

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.

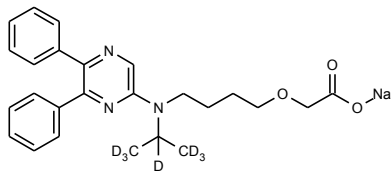
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

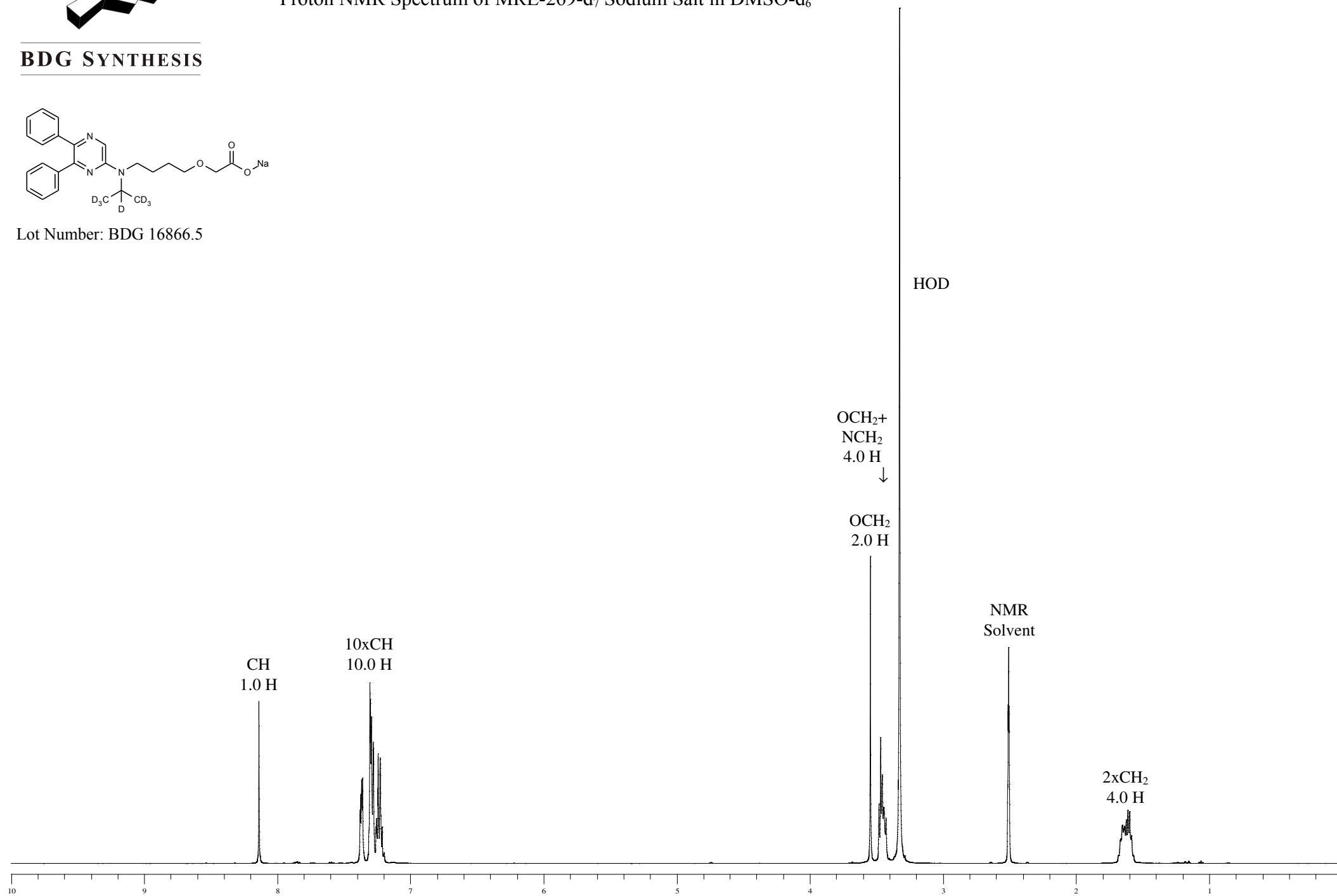


Proton NMR Spectrum of MRE-269-d<sub>7</sub> Sodium Salt in DMSO-d<sub>6</sub>

**BDG SYNTHESIS**



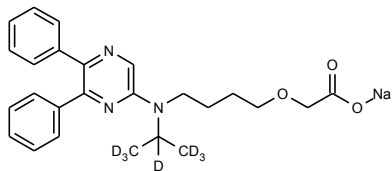
Lot Number: BDG 16866.5



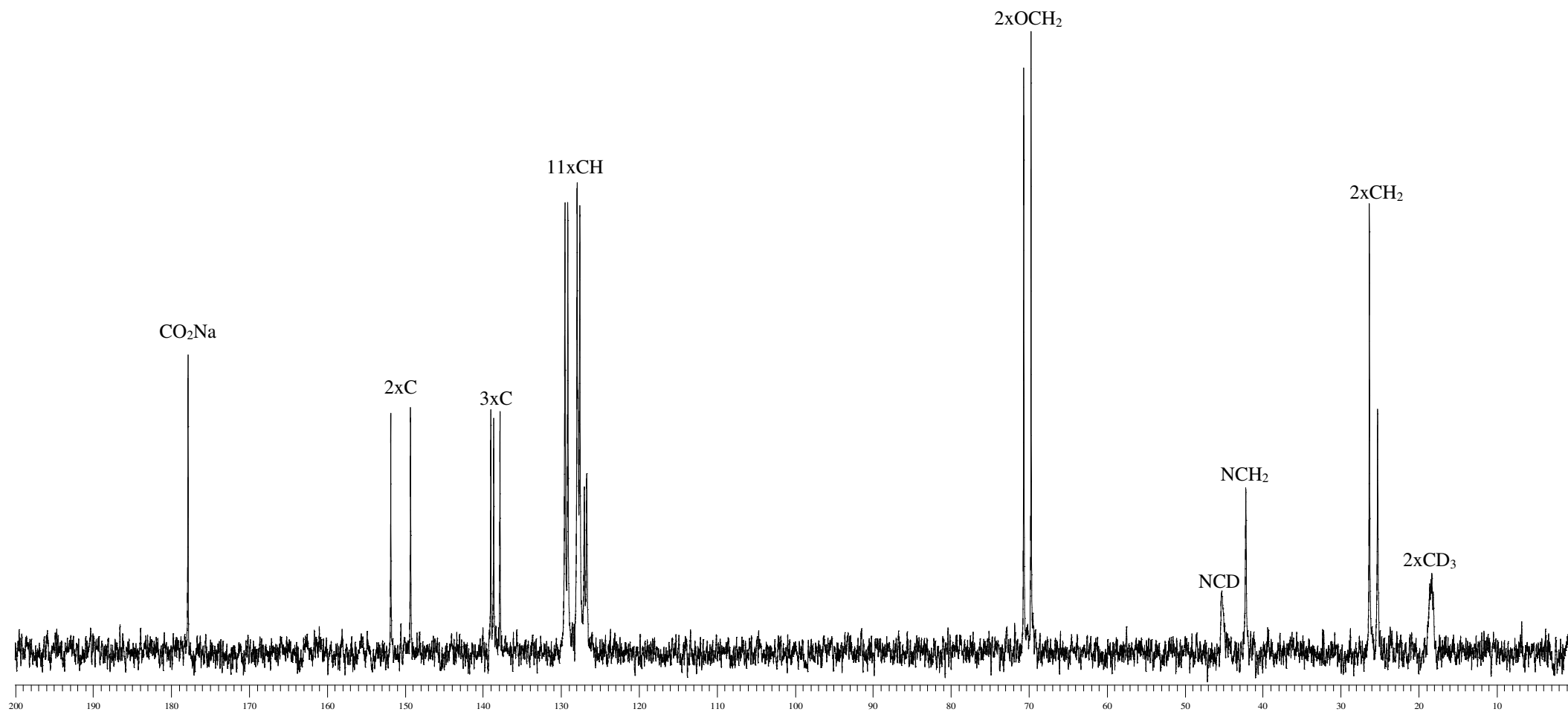


# Carbon-13 NMR Spectrum of MRE-269-d<sub>7</sub> Sodium Salt in D<sub>2</sub>O

**BDG SYNTHESIS**



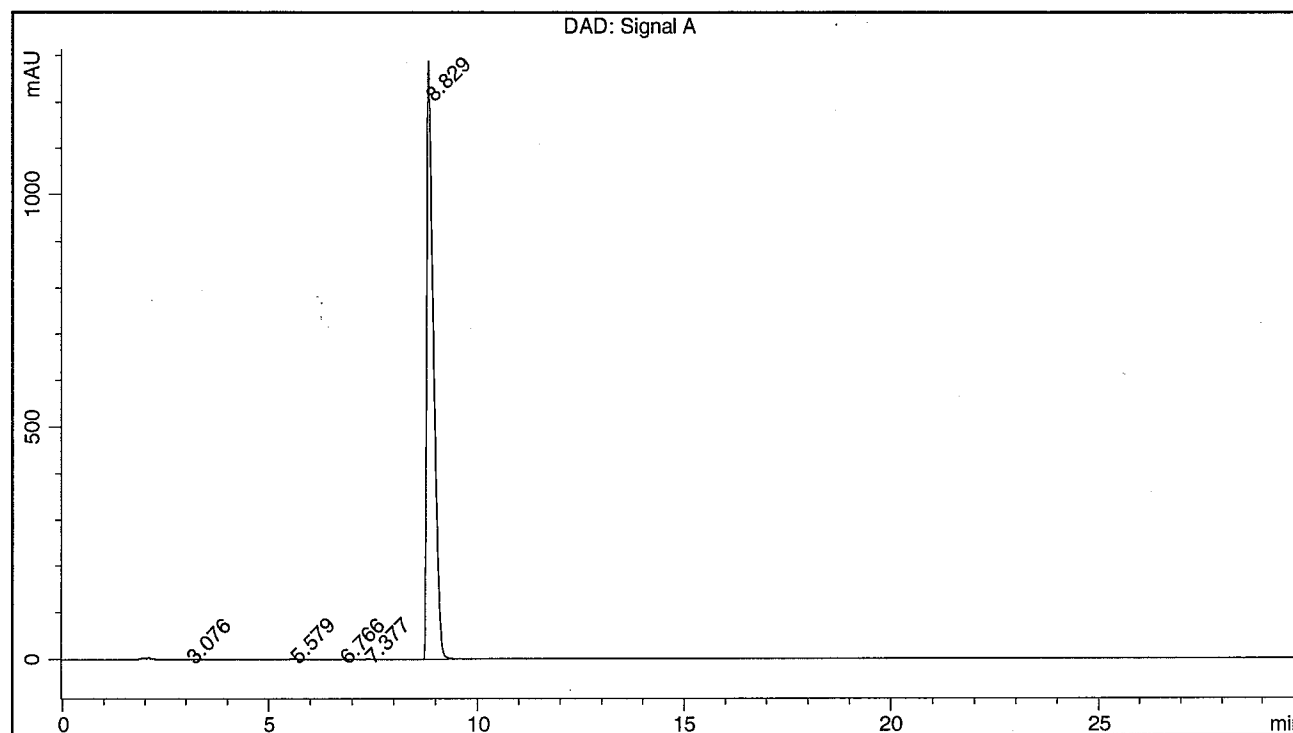
Lot Number: BDG 16866.5



BDG - Analysis of MRE-269-d7 Sodium Salt

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm  
 Guard : Phenomenex Security Guard C18 4 x 3 mm  
 Mobile Phase A : 60:40 20 mM diPotassium Hydrogen Phosphate pH = 7.0 : Acetonitrile  
 Mobile Phase B : 30:70 20 mM diPotassium Hydrogen Phosphate pH = 7.0 : Acetonitrile  
 Gradient (A:B) : T0=100:0, T15=0:100, T25=0:100, T26=100:0, T30=100:0  
 Flow Rate : 1.0 mL/min  
 Sample Solvent : 1:1 Water : Acetonitrile  
 Column Temperature : 20 C  
 Injection Volume : 10 uL  
 Detection : UV at 300 nm

<b>Sample Name</b>	BDG 16866.5	<b>Instrument</b>	AnalyticalLC01
<b>Acquisition</b>	30/03/2017, 10:13:46	<b>Method (rev.)</b>	LC10706a ( 4)
<b>Sequence</b>	BDG_30Mar2017b - Reprocessed	<b>Vial Position</b>	1
<b>Operator</b>	solvation010\cerityadmin	<b>Injection</b>	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	3.08 min	0.4629	3.3706	0.1130 min	0.023 %
2	5.58 min	1.8113	18.2562	0.1576 min	0.126 %
3	6.77 min	0.4339	4.6840	0.1579 min	0.032 %
4	7.38 min	1.0163	7.0759	0.1052 min	0.049 %
5	8.83 min	1287.2401	14474.7913	0.1632 min	99.770 %