



## BDG SYNTHESIS

### Certificate of Analysis

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

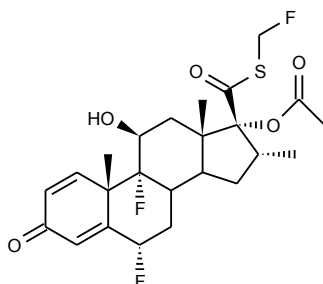
*Neil Beare*

Neil Beare, PhD, Director  
21 December 2015

**Name:** Fluticasone Propionate RC C

**CAS Number:** 80474-24-4

**Structure:**



**Molecular Weight:**  $C_{24}H_{29}F_3O_5S = 486.54$

**Lot Number:** BDG 16598.5

**Appearance:** White, crystalline solid

**Corrected Purity:** 98.0 % (HPLC) - 0.2 % (ethyl acetate) - 0.2 % (ethanol) = 97.6 %

**Re-test Date:** 21 December 2016

**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.  
Residual Solvents: small amounts of ethyl acetate (0.2 % w/w) and ethanol (0.2 % w/w) 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 (ESI+)

Found  $m/z$  509.1587.  $C_{24}H_{29}F_3NaO_5S$   $[M+Na]^+$  requires  $m/z$  509.1586. 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 (98.0 %). 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 59.40, H 6.08 %
$C_{24}H_{29}F_3O_5S$	Requires:	C 59.25, H 6.01 %

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).

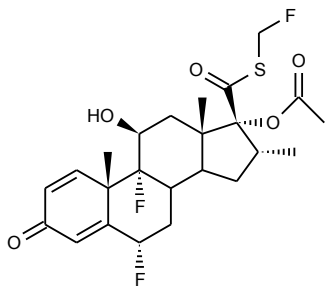
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

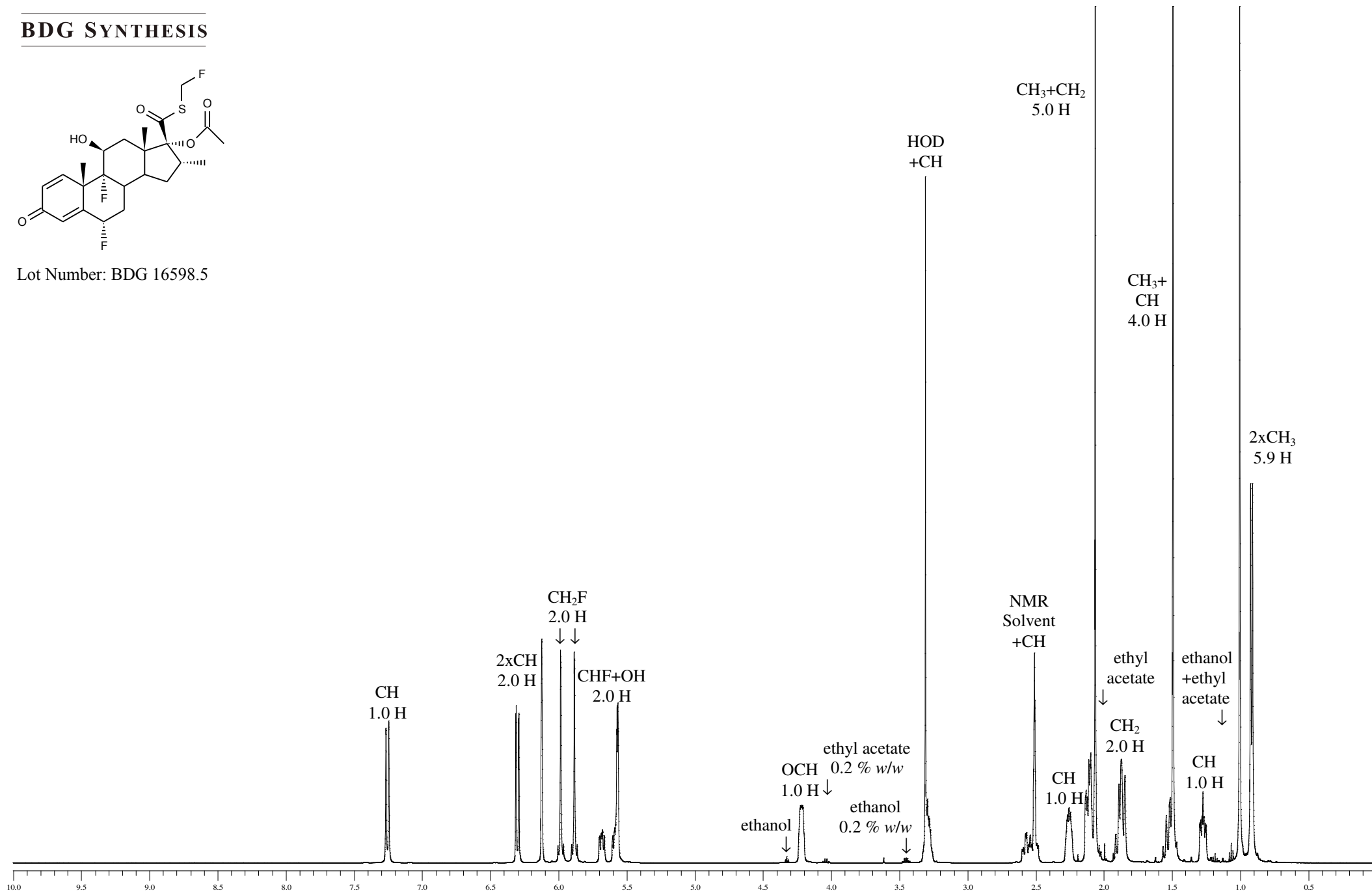


Proton NMR Spectrum of Fluticasone Propionate RC C in DMSO-d<sub>6</sub>

**BDG SYNTHESIS**



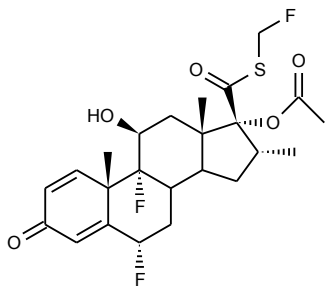
Lot Number: BDG 16598.5



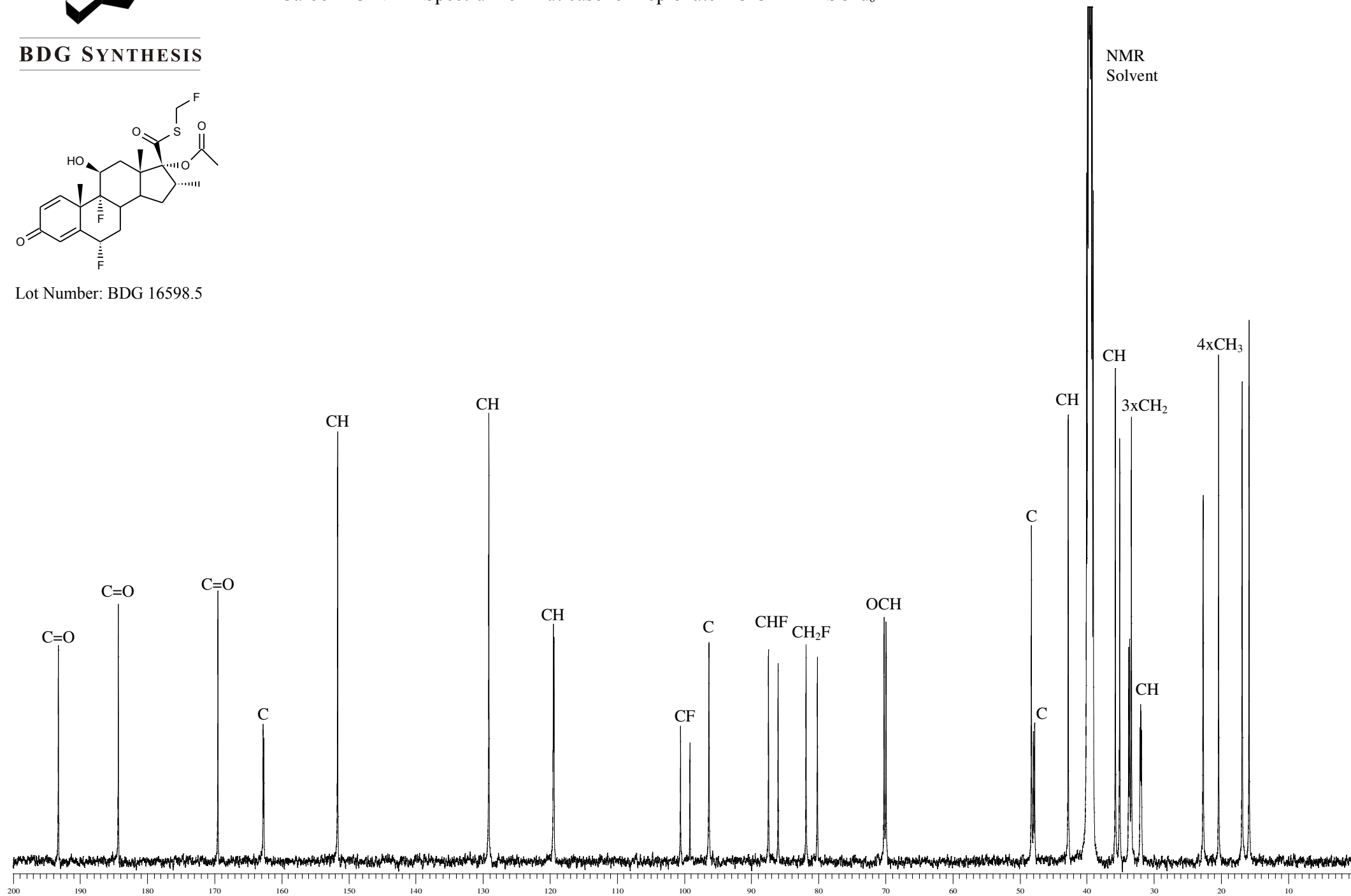


# Carbon-13 NMR Spectrum of Fluticasone Propionate RC C in DMSO-d<sub>6</sub>

**BDG SYNTHESIS**



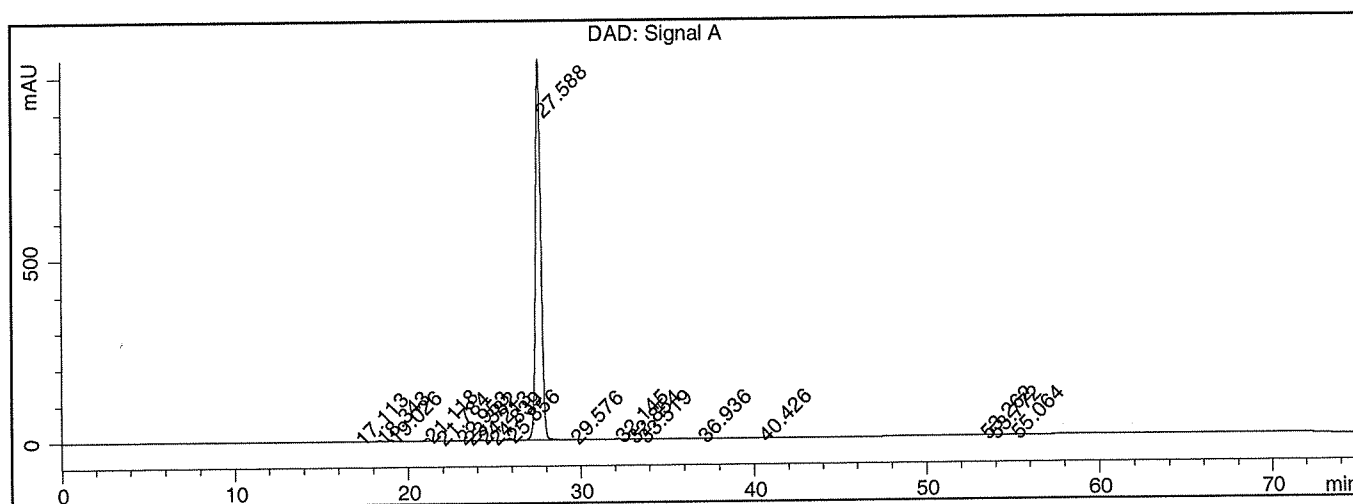
Lot Number: BDG 16598.5



BDG - Analysis of Fluticasone Propionate RC C

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm  
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm  
 Mobile Phase A : 57:43:3:0.05 Water : Acetonitrile : Methanol : Phosphoric Acid  
 Mobile Phase B : 10:90:3:0.05 Water : Acetonitrile : Methanol : Phosphoric Acid  
 Gradient (A:B) : T0=100:0, T40=74:26, T60=0:100, T68=0:100, T70=100:0, T75=100:0  
 Column Temperature : 40 C . . . . . Flow Rate : 1.0 mL/min . . . . . Injection Volume : 10 uL  
 Sample Solvent : 1:1 Mobile Phase A : Mobile Phase B . . . . . Detection : UV 239 nm

<b>Sample Name</b>	BDG 16598.5	<b>Instrument</b>	AnalyticalLC01
<b>Acquisition</b>	21/12/2015, 13:23:48	<b>Method (rev.)</b>	LC10632a ( 12)
<b>Sequence</b>	BDG_21Dec2015a - Reprocessed	<b>Vial Position</b>	5
<b>Operator</b>	solvation010\cerityadmin	<b>Injection</b>	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	17.11 min	0.2157	3.2542	0.1880 min	0.014 %
2	18.34 min	3.0076	51.2502	0.2548 min	0.217 %
3	19.03 min	3.5867	63.2562	0.2758 min	0.268 %
4	21.12 min	7.2642	136.9093	0.2919 min	0.580 %
5	21.78 min	0.3090	6.1616	0.2886 min	0.026 %
6	22.95 min	0.9652	21.5740	0.3248 min	0.091 %
7	23.38 min	0.1450	1.5394	0.1479 min	0.007 %
8	24.21 min	1.3136	30.6828	0.3245 min	0.130 %
9	24.84 min	0.2333	4.3463	0.2294 min	0.018 %
10	25.86 min	4.9951	110.4002	0.3339 min	0.468 %
11	27.59 min	1045.3422	23112.6308	0.3420 min	97.975 %
12	29.58 min	0.1236	1.0053	0.1069 min	0.004 %
13	32.15 min	1.0640	23.8776	0.3418 min	0.101 %
14	32.85 min	0.1171	1.4081	0.1643 min	0.006 %
15	33.52 min	0.1568	2.6495	0.2091 min	0.011 %
16	36.94 min	0.3286	10.1903	0.3818 min	0.043 %
17	40.43 min	0.1808	3.2158	0.2196 min	0.014 %
18	53.26 min	0.1019	1.4570	0.1948 min	0.006 %
19	53.77 min	0.1492	1.9788	0.1707 min	0.008 %
20	55.06 min	0.1798	2.6202	0.1940 min	0.011 %