



## BDG SYNTHESIS

### Certificate of Analysis

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

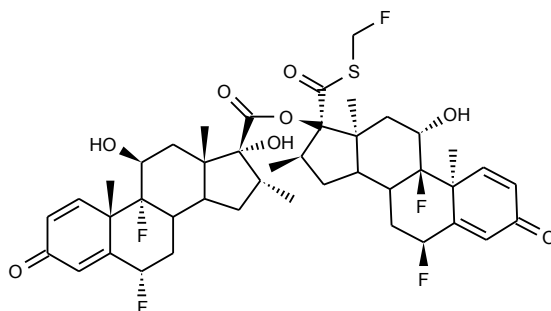
*Neil Beare*

Neil Beare, PhD, Director  
19 January 2017

**Name:** Fluticasone Propionate RC E

**CAS Number:** 220589-37-7

**Structure:**



**Molecular Weight:**  $C_{43}H_{51}F_5O_8S = 822.92$

**Lot Number:** BDG 17201.11

**Appearance:** White, crystalline solid

**Corrected Purity:** 98.8 % (HPLC) - 5.2 % (water) = 93.6 %

**Re-test Date:** 19 January 2022

**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. Susceptible to static.

## 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: traces of unidentified impurities are 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  $m/z$  845.3119.  $C_{43}H_{51}F_5NaO_8S$   $[M+Na]^+$  requires  $m/z$  845.3123. The deviation of 0.5 ppm is within normally accepted limits for the establishment of identity by HRMS.

### HPLC

A somewhat broadened, symmetrical peak is observed (98.8 %). 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 59.26, H 6.25 %
$C_{43}H_{51}F_5O_8S \cdot 2.5H_2O$	Requires:	C 59.50, H 6.50 %, $H_2O$ 5.19 %
$C_{43}H_{51}F_5O_8S$	Requires:	C 62.76, H 6.25 %

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. In the absence of a Karl-Fischer water analysis, we recommend that the "best-fit" water content be used when determining corrected purity.

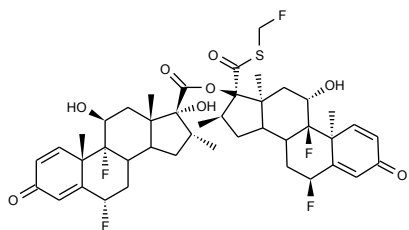
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

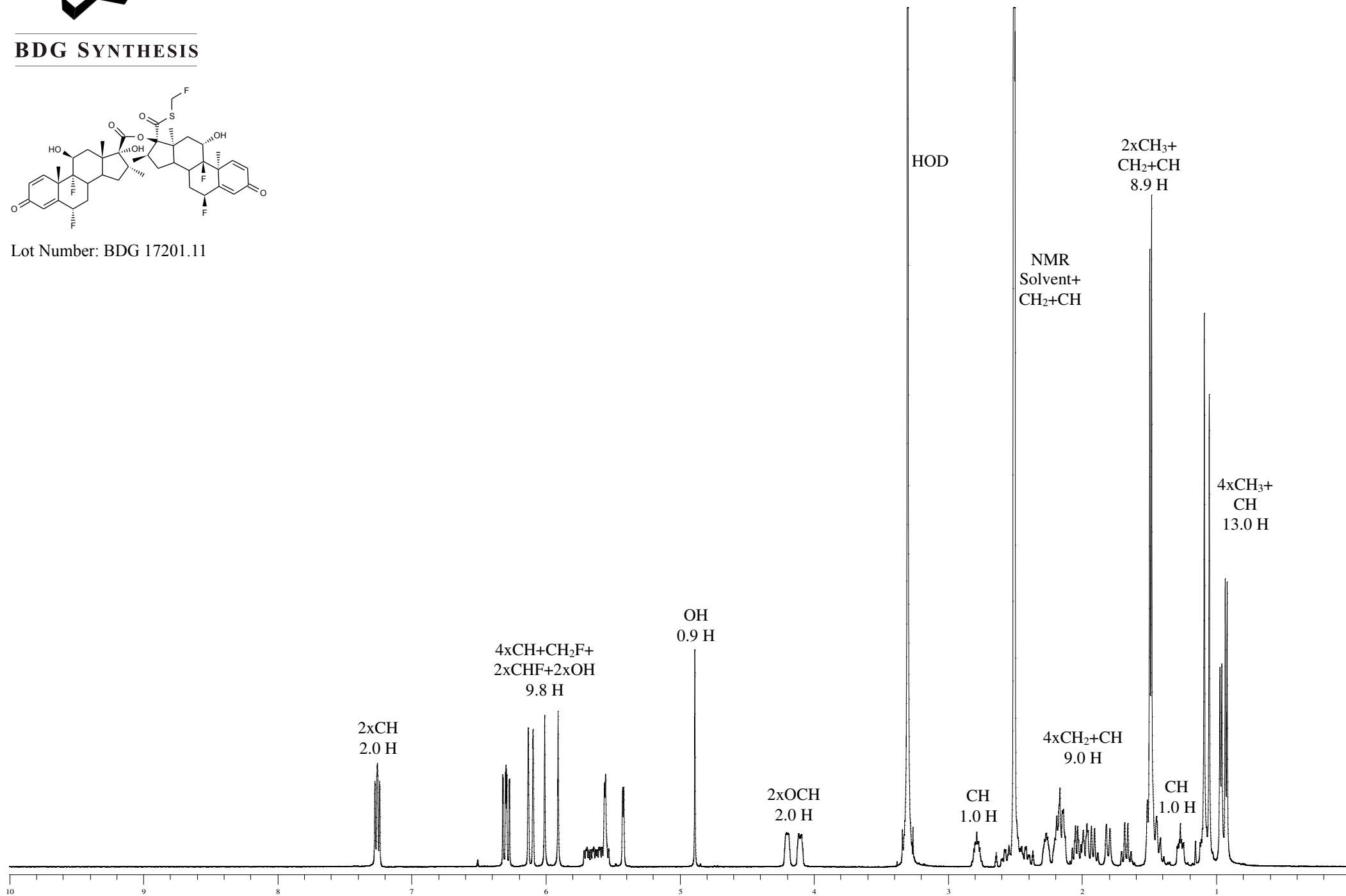


Proton NMR Spectrum of Fluticasone Propionate RC E in DMSO-d<sub>6</sub>

**BDG SYNTHESIS**



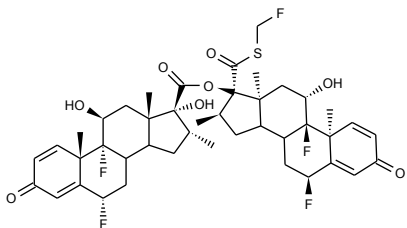
Lot Number: BDG 17201.11



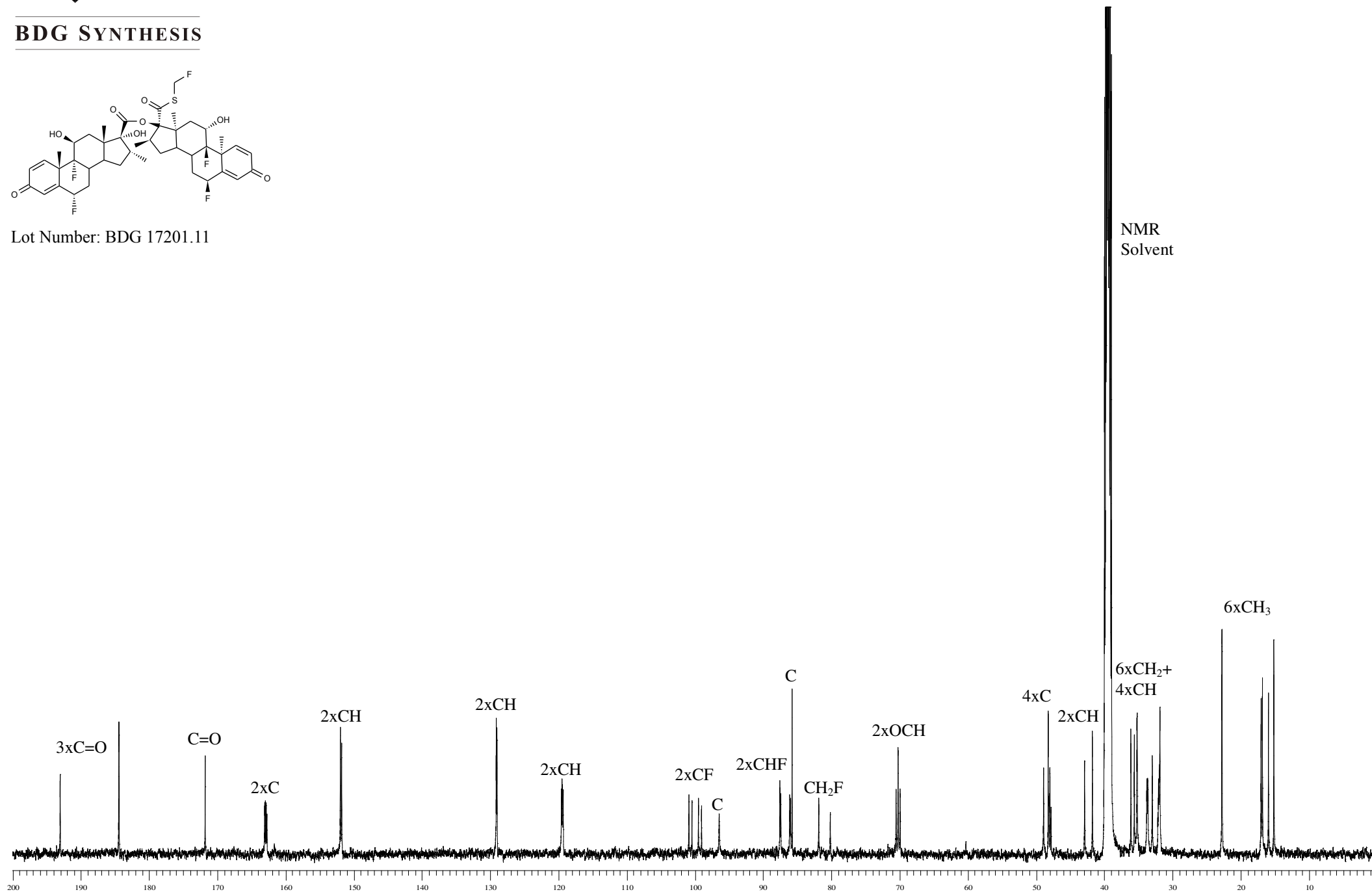


# Carbon-13 NMR Spectrum of Fluticasone Propionate RC E in DMSO-d<sub>6</sub>

## BDG SYNTHESIS



Lot Number: BDG 17201.11



BDG - Analysis of Fluticasone Propionate RC E

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm

Guard : Phenomenex Security Guard C18 RP 4 x 3 mm

Mobile Phase A : 45:55 Water : Acetonitrile

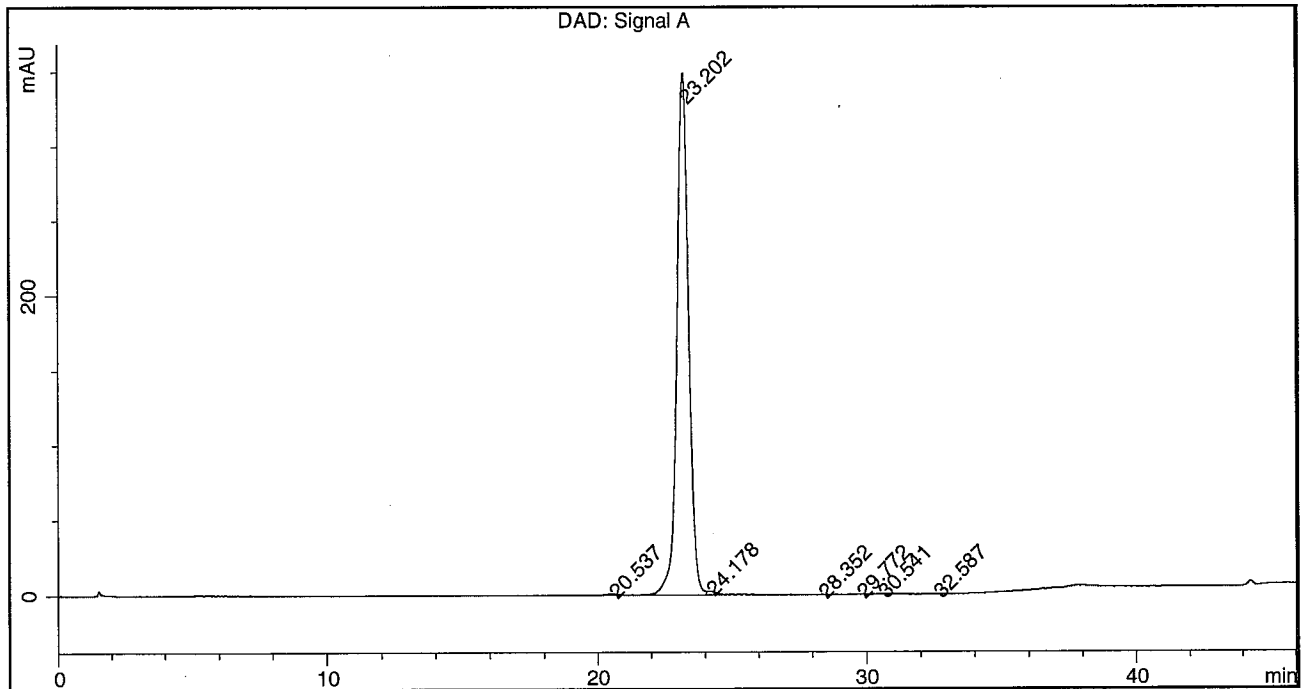
Mobile Phase B : Acetonitrile

Gradient (A:B) : T0=100:0, T30=100:0, T35=0:100, T45=0:100, T46=100:0, T50=100:0

Column Temperature : 30 C . . . . Flow Rate : 1.0 mL/min

Sample Solvent : Mobile Phase A . . . . Injection Volume : 10 uL . . . . Detection : UV 239 nm

<b>Sample Name</b>	BDG 17201.11	<b>Instrument</b>	AnalyticalLC01
<b>Acquisition</b>	19/01/2017, 17:02:08	<b>Method (rev.)</b>	LC10632y ( 23)
<b>Sequence</b>	BDG_19Jan2017b - 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	20.54 min	0.8364	23.9237	0.3537 min	0.220 %
2	23.20 min	349.3800	10727.9510	0.4702 min	98.816 %
3	24.18 min	2.4737	63.9874	0.3578 min	0.589 %
4	28.35 min	0.1789	5.3223	0.3711 min	0.049 %
5	29.77 min	0.2817	7.5108	0.3271 min	0.069 %
6	30.54 min	0.3755	19.8934	0.6540 min	0.183 %
7	32.59 min	0.1910	7.8714	0.5088 min	0.073 %