

BDG SYNTHESIS

Certificate of Analysis

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

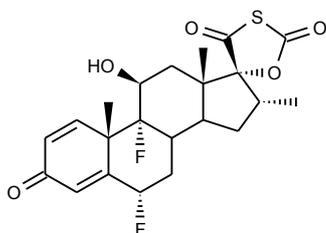
Neil Beare

Neil Beare, PhD, Director
29 June 2016

Name: Fluticasone Propionate RC B

CAS Number: 219719-95-6

Structure:



Molecular Weight: $C_{22}H_{24}F_2O_5S = 438.49$

Lot Number: BDG 17219.2

Appearance: White, crystalline solid

Purity By HPLC: 99.2 %

Re-test Date: 29 June 2017

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: 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 (ESI+)

Found m/z 439.1392. $C_{22}H_{25}F_2O_5S$ $[M+H]^+$ requires m/z 439.1391. 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 (99.2 %). 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 60.02, H 5.58 %
$C_{22}H_{24}F_2O_5S$	Requires:	C 60.26, H 5.52 %

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).

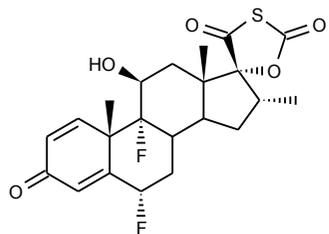
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

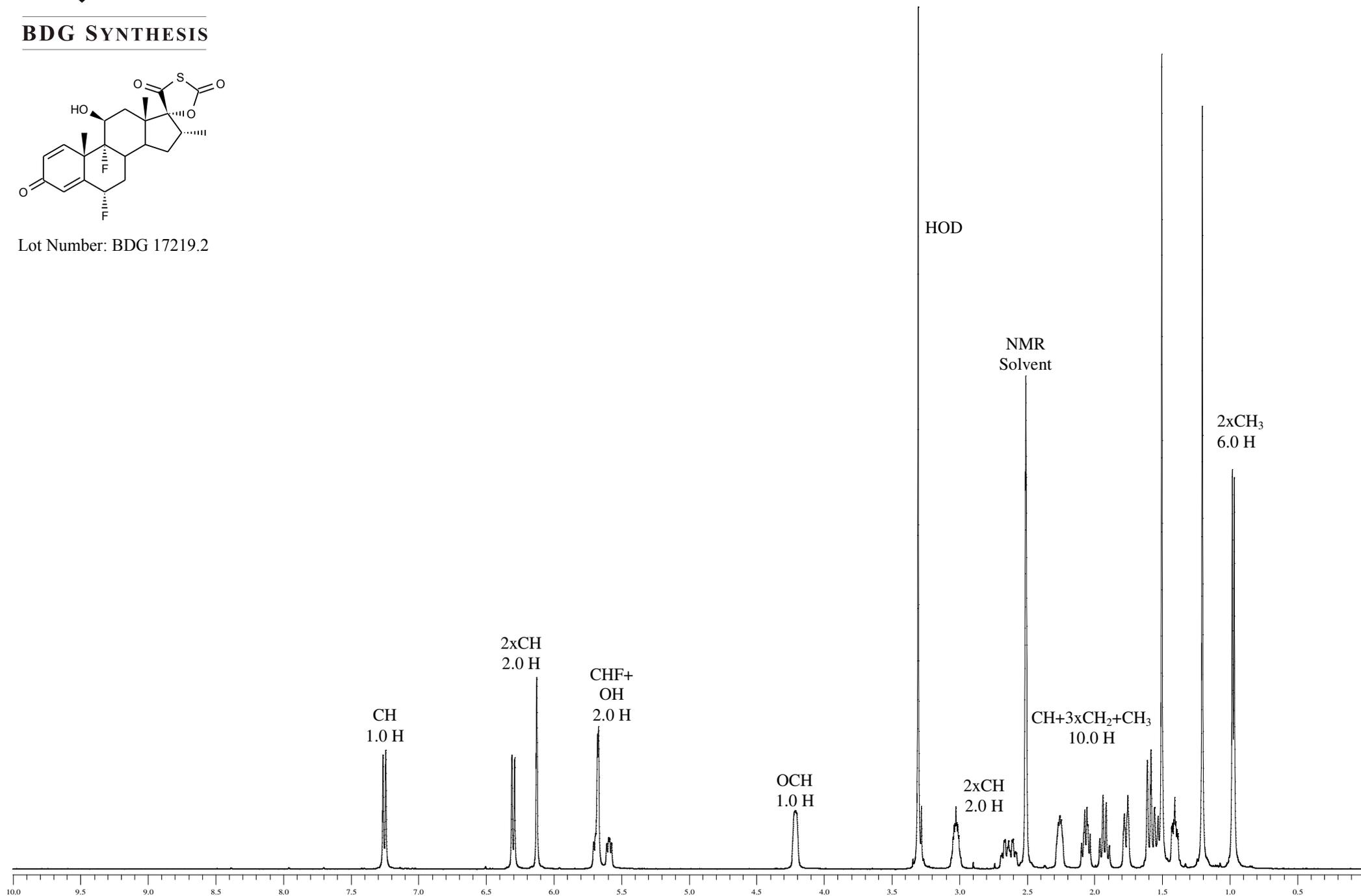


Proton NMR Spectrum of Fluticasone Propionate RC B in DMSO-d₆

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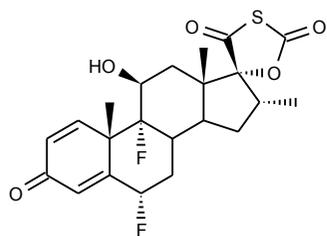
Lot Number: BDG 17219.2



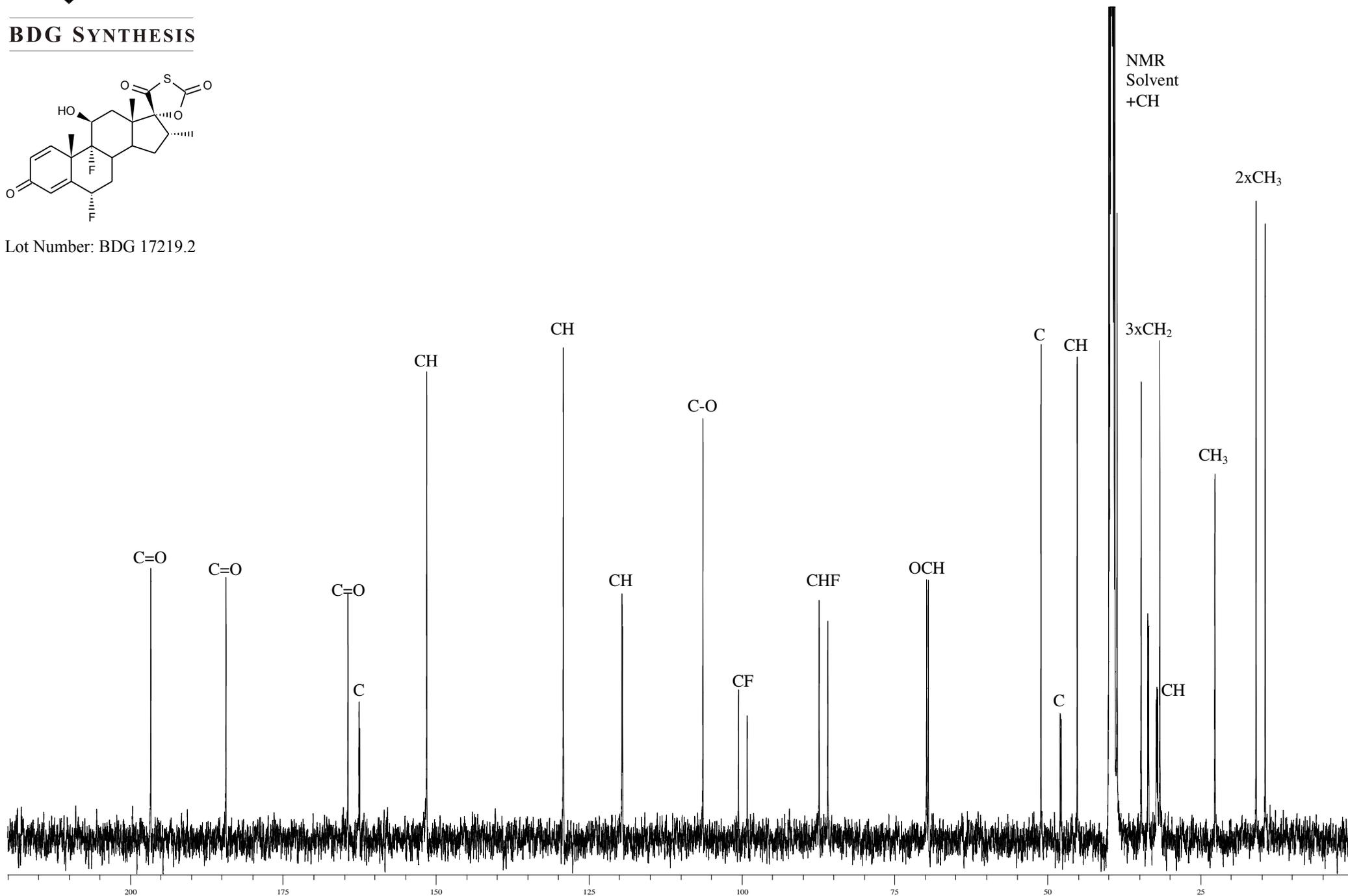


Carbon-13 NMR Spectrum of Fluticasone Propionate RC B in DMSO-d₆

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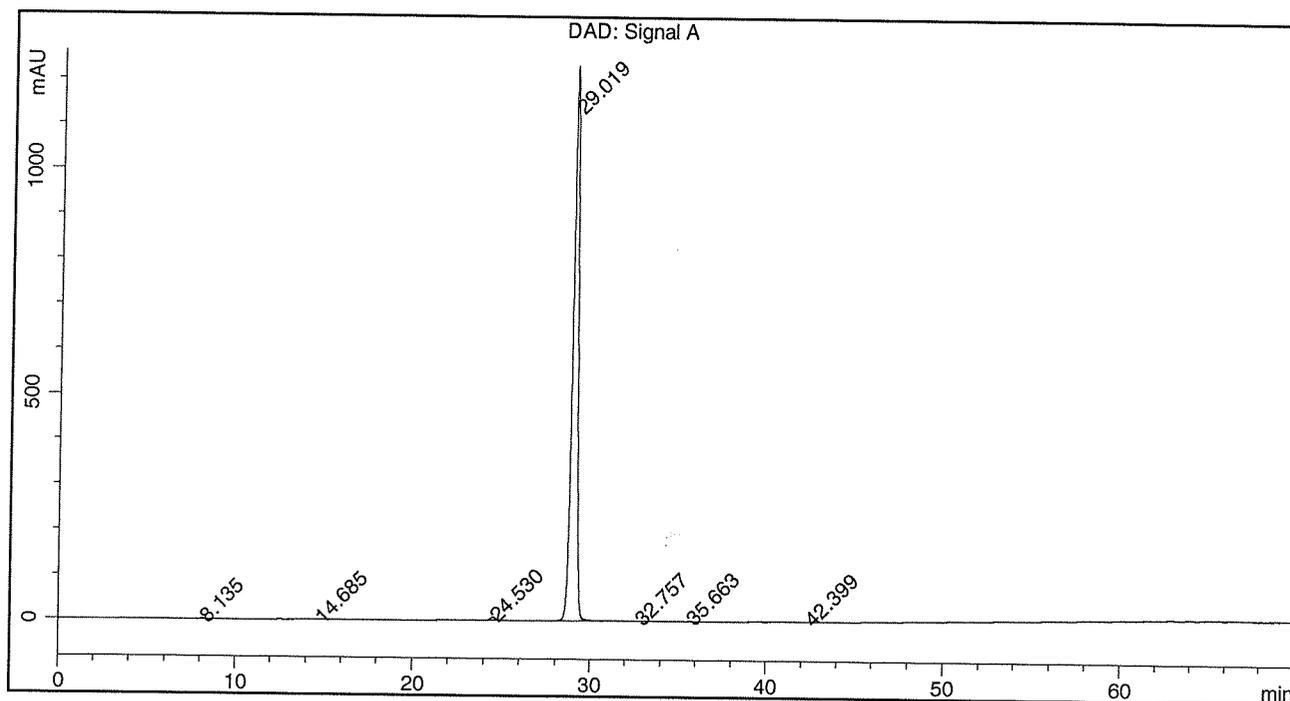
Lot Number: BDG 17219.2



BDG - Analysis of Fluticasone Propionate RC B

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

Sample Name	BDG 17219.2	Instrument	AnalyticalLC01
Acquisition	29/06/2016, 15:29:51	Method (rev.)	LC10632f (4)
Sequence	BDG_29Jun2016a - Reprocessed	Vial Position	4
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	8.14 min	0.8641	8.4409	0.1500 min	0.031 %
2	14.68 min	0.8143	11.5672	0.1937 min	0.042 %
3	24.53 min	7.0530	145.8692	0.3273 min	0.531 %
4	29.02 min	1231.3097	27266.0525	0.3423 min	99.231 %
5	32.76 min	0.7090	16.0265	0.2797 min	0.058 %
6	35.66 min	0.7316	17.9281	0.2979 min	0.065 %
7	42.40 min	0.4793	11.5504	0.2931 min	0.042 %