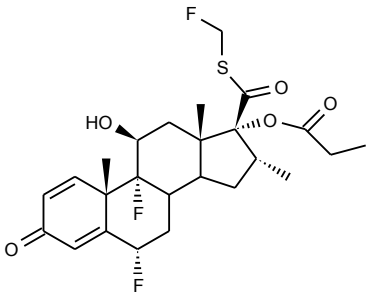
BDG SYNTHESIS

Certificate of Analysis

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

Barry Dent

Barry R. Dent, PhD, Director
5 December 2013

Name:	**Fluticasone Propionate**
CAS Number:	80474-14-2
Structure:	
Molecular Weight:	$C_{25}H_{31}F_3O_5S$ = 500.57
Lot Number:	BDG 7979.3
Appearance:	White powder
Corrected Purity:	98.2 % (HPLC) - 0.4 % (ethyl acetate) = 97.8 %
Re-test Date:	5 December 2018

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.

Mailing:
BDG Synthesis Limited
PO Box 38627, Wellington Mail Centre,
Wellington 5045, New Zealand.

Shipping:
BDG Synthesis Limited
Gracefield Research Centre, Building F,
69 Gracefield Rd, Lower Hutt 5010, New Zealand.

Phone: + 64 4 569 0520
Fax: + 64 4 569 0521

info@bdg.co.nz
www.bdg.co.nz

Identity and Purity

Proton NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available.
Residual Solvents: a small amount of ethyl acetate (0.4 % *w/w*) is 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* 523.1743. $C_{25}H_{31}F_3NaO_5S$ $[M+Na]^+$ requires *m/z* 523.1737. 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 (98.2 %). 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 60.23, H 6.29 %
$C_{25}H_{31}F_3O_5S$	Requires:	C 59.98, H 6.24 %

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).

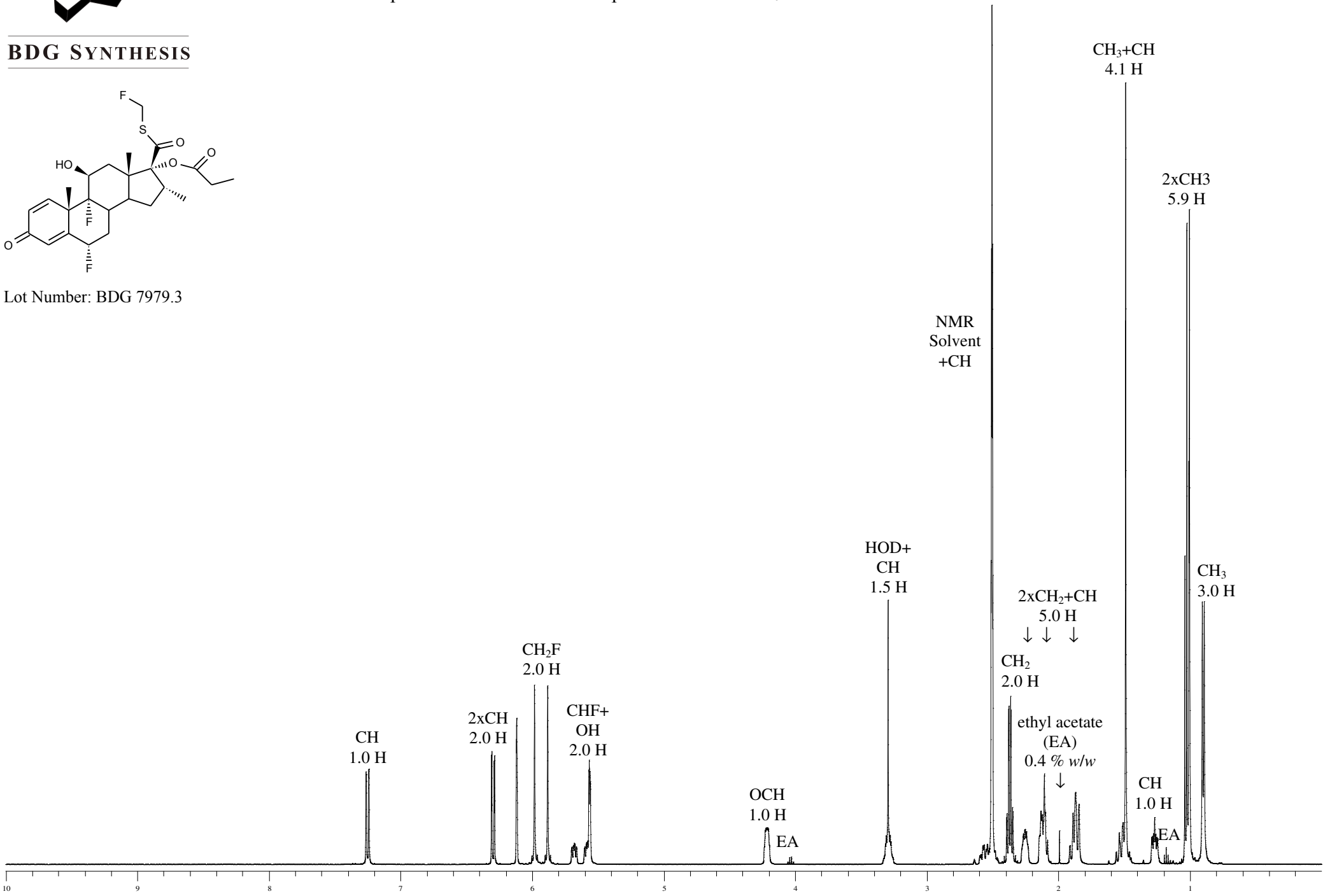
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

BDG SYNTHESIS

Proton NMR Spectrum of Fluticasone Propionate in DMSO-d_6

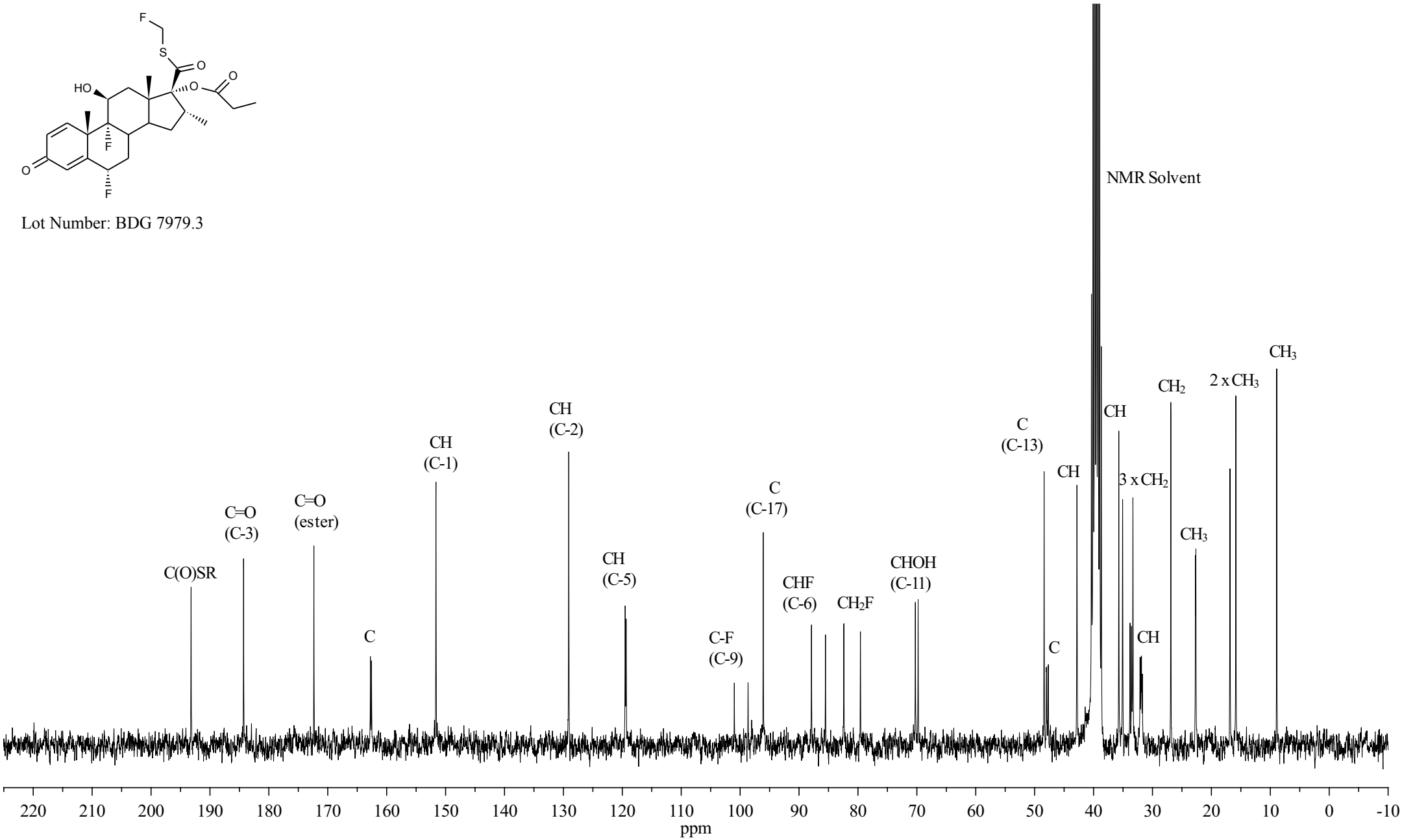
Lot Number: BDG 7979.3



BDG SYNTHESIS

Carbon-13 NMR Spectrum of Fluticasone Propionate in DMSO-d_6

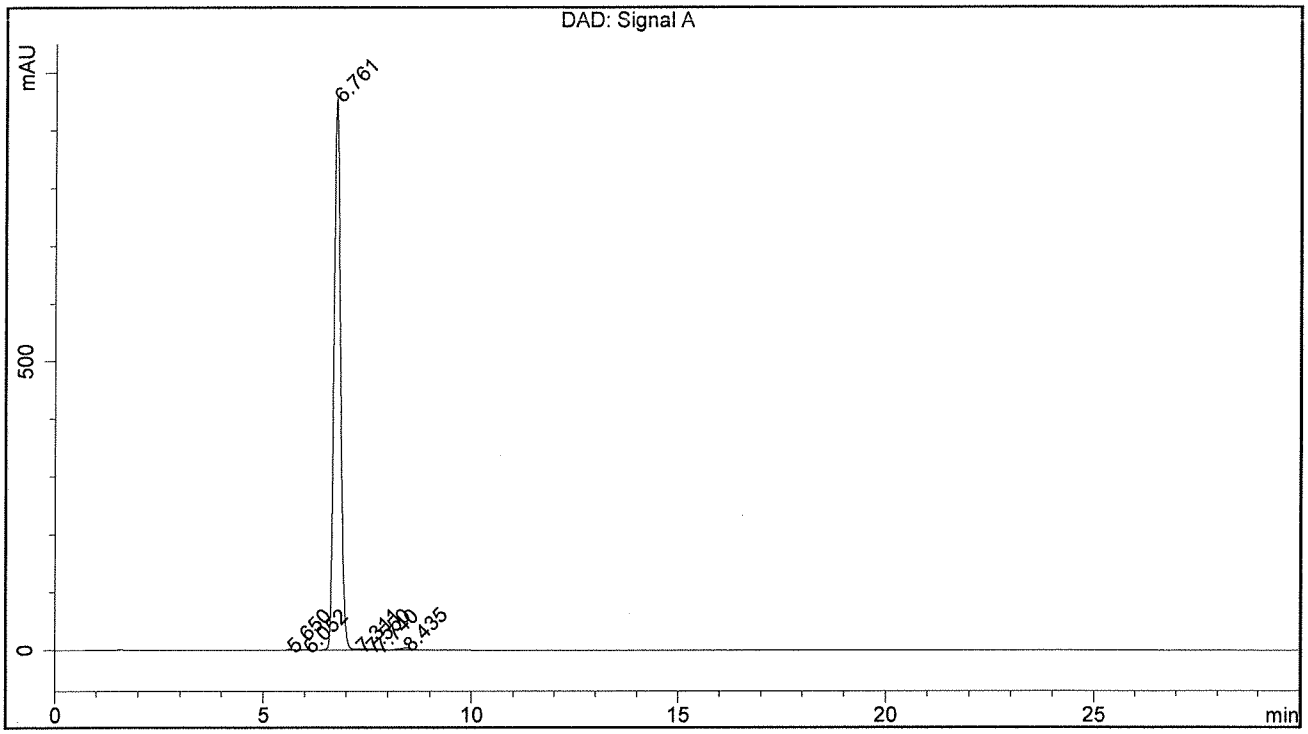
Lot Number: BDG 7979.3



BDG - Analysis of Fluticasone propionate

Column : Phenomenex Luna C18 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 4 x 3 mm
Mobile Phase : 30:70 Water : Acetonitrile . . . . . Flow Rate : 1.0 mL/min
Sample Solvent : 30:70 Water : Acetonitrile . . . . . Injection Volume : 10 uL
Column Temperature : 20C . . . . . Detection : UV at 239 nm

Sample Name	BDG 7979.3	Instrument	AnalyticalLC01
Acquisition	05/12/2013, 09:33:02	Method (rev.)	LC10240c (4)
Sequence	BDG_05Dec2013a	Vial Position	2
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	5.65 min	1.8745	25.4236	0.1902 min	0.233 %
2	6.05 min	1.0712	11.6247	0.1666 min	0.107 %
3	6.76 min	955.7267	10706.3450	0.1747 min	98.235 %
4	7.31 min	2.1720	28.5149	0.1912 min	0.262 %
5	7.55 min	2.4664	30.6282	0.1829 min	0.281 %
6	7.74 min	1.8422	20.9568	0.1767 min	0.192 %
7	8.43 min	4.0482	75.2282	0.2668 min	0.690 %