



BDG SYNTHESIS

Certificate of Analysis

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

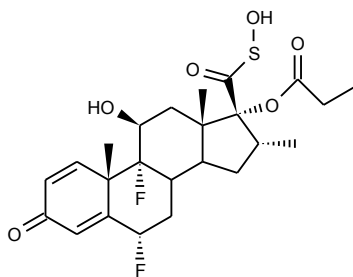
Neil Beare

Neil Beare, PhD, Director
8 August 2005

Name: Fluticasone Propionate RC A

CAS Number: 948566-12-9

Structure:



Molecular Weight: $C_{24}H_{30}F_2O_6S = 484.55$

Lot Number: BDG 2958.3

Appearance: White, crystalline solid

Corrected Purity: 98.7 % (HPLC) - 7.3 % (water) = 91.4 %

Re-test Date: 8 August 2010

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. Solutions in acetone quickly degrade.

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 (TOF MS ES+)

Found m/z 485.1810. $C_{24}H_{31}F_2O_6S$ $[M+H]^+$ requires m/z 485.1809. 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 (98.7 %). 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 55.95, H 6.29, F 7.47, S 6.10 %
$C_{24}H_{30}F_2O_6S \cdot 1.8H_2O$	Requires:	C 55.76, H 6.55, F 7.35, S 6.20 %
$C_{24}H_{30}F_2O_6S$	Requires:	C 59.49, H 6.24, F 7.84, S 6.62 %

The elemental analyses fall substantially 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 ₂ O 7.3 %
$C_{24}H_{30}F_2O_6S \cdot 1.8H_2O$	Requires:	H ₂ O 6.3 %

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.

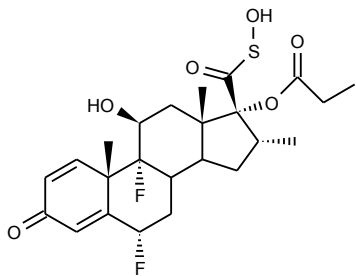
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

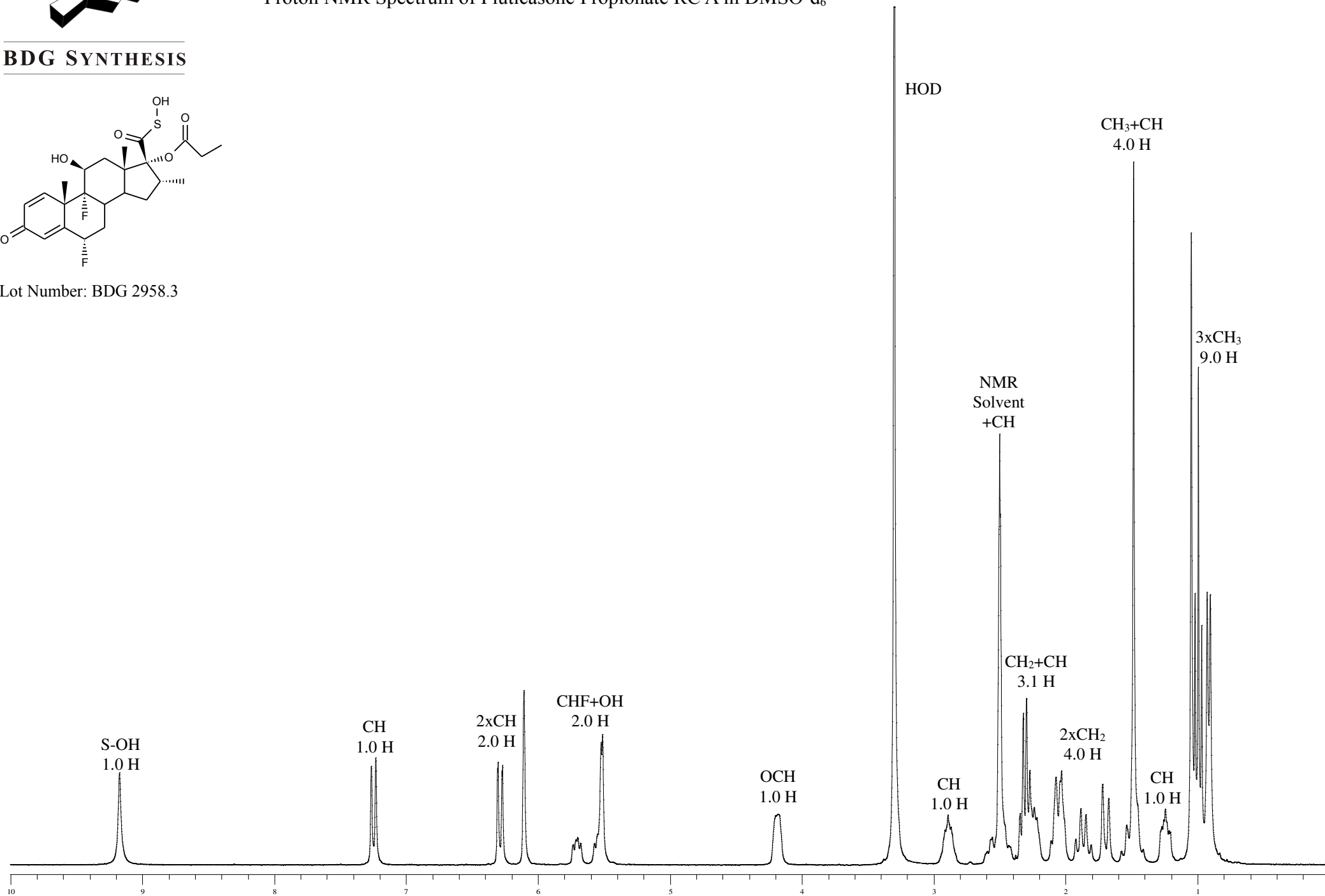


Proton NMR Spectrum of Fluticasone Propionate RC A in DMSO-d₆

BDG SYNTHESIS



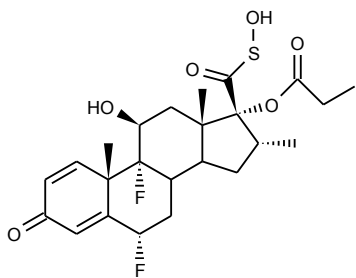
Lot Number: BDG 2958.3



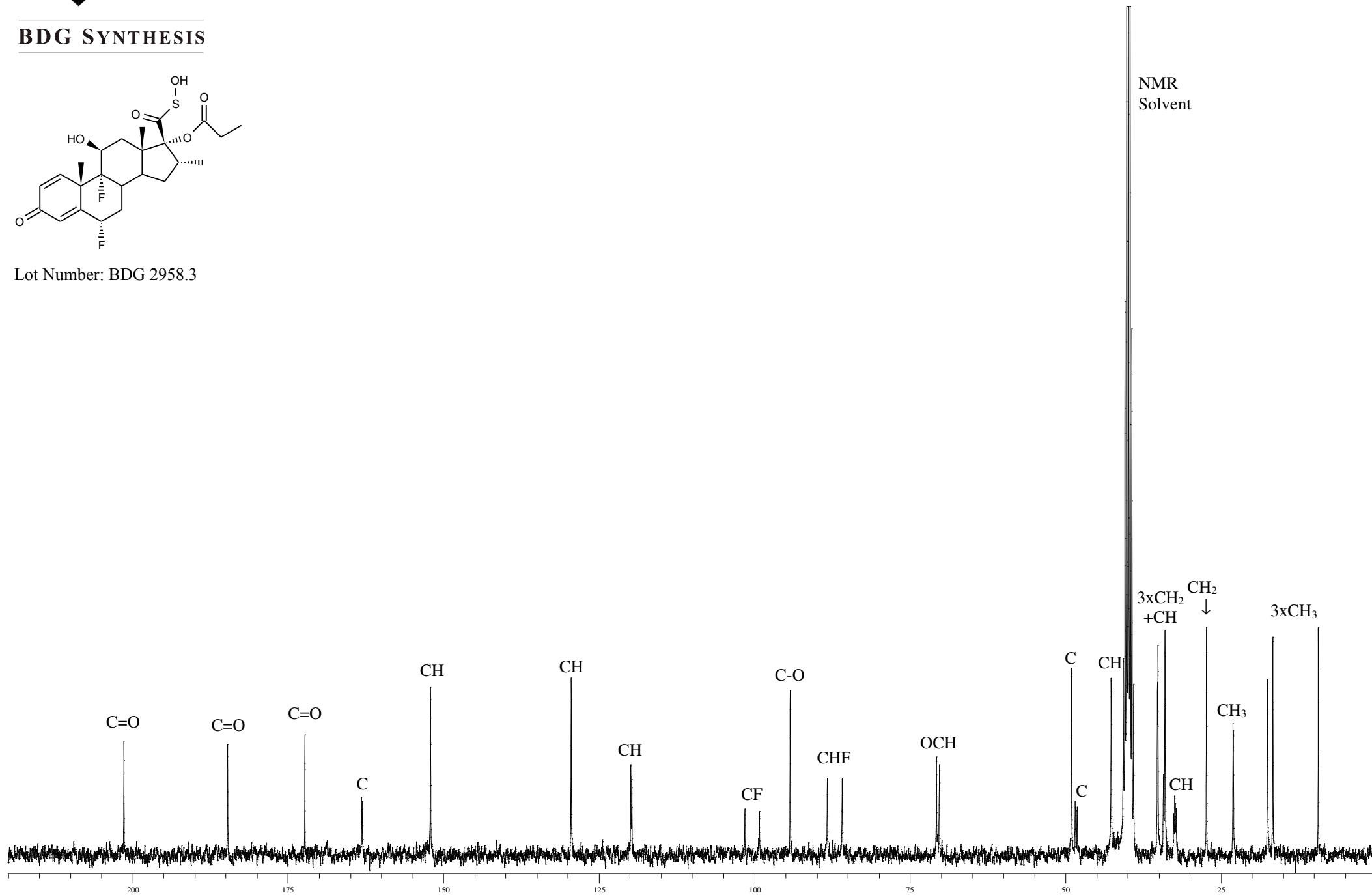


Carbon-13 NMR Spectrum of Fluticasone Propionate RC A in DMSO-d₆

BDG SYNTHESIS



Lot Number: BDG 2958.3



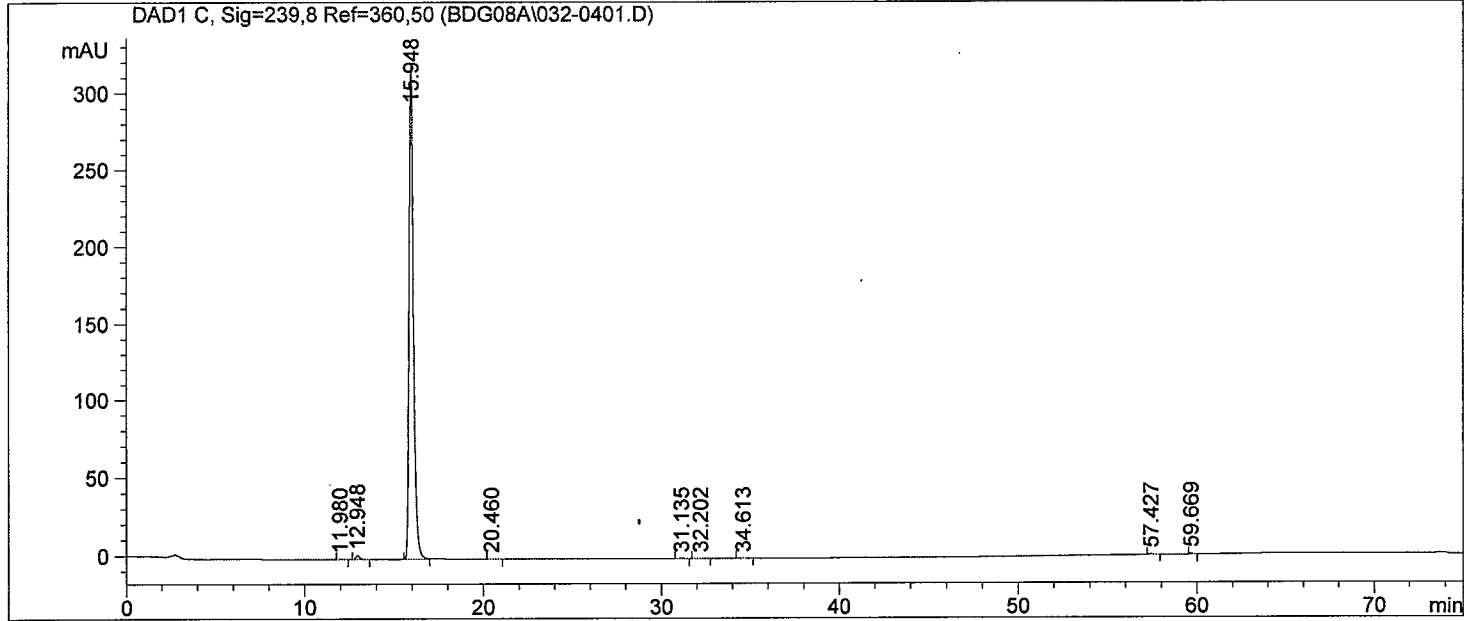
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Injection Date : 8/8/05 5:16:21 PM          Seq. Line : 4
Sample Name    : BDG 2958.3                  Location  : Vial 32
Acq. Operator  : YRLman                      Inj      : 1
                                           Inj Volume : 10 µl

Acq. Method   : N:\LC1100_3\1\METHODS\LC50231A.M
Last changed  : 8/8/05 1:19:19 PM by YRLman
Analysis Method : N:\LC1100_3\1\METHODS\LC50231A.M
Last changed  : 8/9/05 8:54:28 AM by YRLman
                (modified after loading)

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BDG - Gradient analysis of fluticasone metabolite on Luna C18, 5µm, 250 x 4.6mm, #LC50231



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Area Percent Report
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Sorted By      : Signal
Multiplier    : 1.0000
Dilution      : 1.0000

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Signal 1: DAD1 C, Sig=239,8 Ref=360,50

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.980	MM	0.2840	5.06762	2.97423e-1	0.0916
2	12.948	MM	0.2474	39.41850	2.65544	0.7125
3	15.948	MM	0.2826	5462.77832	322.21445	98.7465
4	20.460	MM	0.2926	2.29308	1.30634e-1	0.0415
5	31.135	MM	0.3871	3.99740	1.72091e-1	0.0723
6	32.202	MM	0.4396	3.13807	1.18969e-1	0.0567
7	34.613	MM	0.5366	4.62374	1.43612e-1	0.0836
8	57.427	MM	0.1698	4.80332	4.71590e-1	0.0868
9	59.669	MM	0.1477	6.00592	6.77666e-1	0.1086

Totals : 5532.12597 326.88187

Results obtained with enhanced integrator!

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*** End of Report ***