BDG SYNTHESIS

Certificate of Analysis

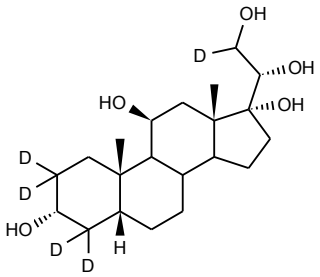
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

Neil Beare, PhD, Director
4 December 2015

Name: **α-Cortol-d_5**

CAS Number: 516-38-1 (unlabelled)

Structure:



Molecular Weight: $C_{21}H_{31}D_5O_5$ = 373.54

Lot Number: BDG 17240.3

Appearance: White, crystalline solid

Corrected Purity: 99.7 % (HPLC) - 13.4 % (water) = 86.3 %

Isotopic Purity: Under 0.5 % d_0

Re-test Date: 4 December 2020

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.

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Identity and Purity

Proton NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available.
Isotopic Labelling: signals at the sites of deuteration are greatly diminished, compared with what would be expected for unlabelled material.
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.
Isotopic Labelling: most signals at the sites of deuteration have collapsed to small multiplets compared with what would be expected for unlabelled material, however the signal assigned to C-21 has sharp spike indicating some H/D exchange has occurred at this position.

High-resolution Mass Spectrum (ESI+)

Found *m/z* 396.2769. $C_{21}H_{31}D_5NaO_5$ $[M+Na]^+$ requires *m/z* 396.2774. The deviation of 1.3 ppm is within normally accepted limits for the establishment of identity by HRMS. Minor signals are observed for d_4 and d_6 material. No signal for d_0 material was seen (detection limit about 0.5 %).

HPLC

A sharp, symmetrical peak is observed (99.7 %). 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 58.41, H 8.52, D 2.27 %
$C_{21}H_{31}D_5O_5 \cdot 3.2H_2O$	Requires:	C 58.50, H 8.74, D 2.34 %, H_2O 13.37 %
$C_{21}H_{31}D_5O_5$	Requires:	C 67.52, H 8.36, D 2.70 %

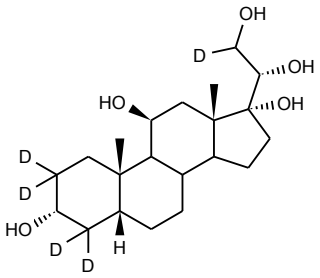
The elemental analyses fall substantially outside those expected for anhydrous material; the presence of water is reasonably expected from the method of purification and/or the type of material, and the “best-fit” hydrated molecular formula is given. In the absence of a Karl-Fischer water analysis, we 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.

BDG SYNTHESIS

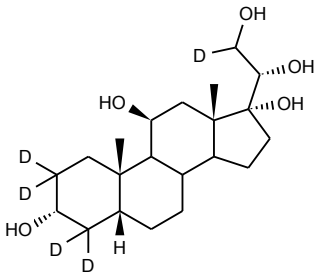
Proton NMR Spectrum of α-Cortol-d_5 in DMSO-d_6



Lot Number: BDG 17240.3

BDG SYNTHESIS

Carbon-13 NMR Spectrum of α-Cortol-d_5 in DMSO-d_6



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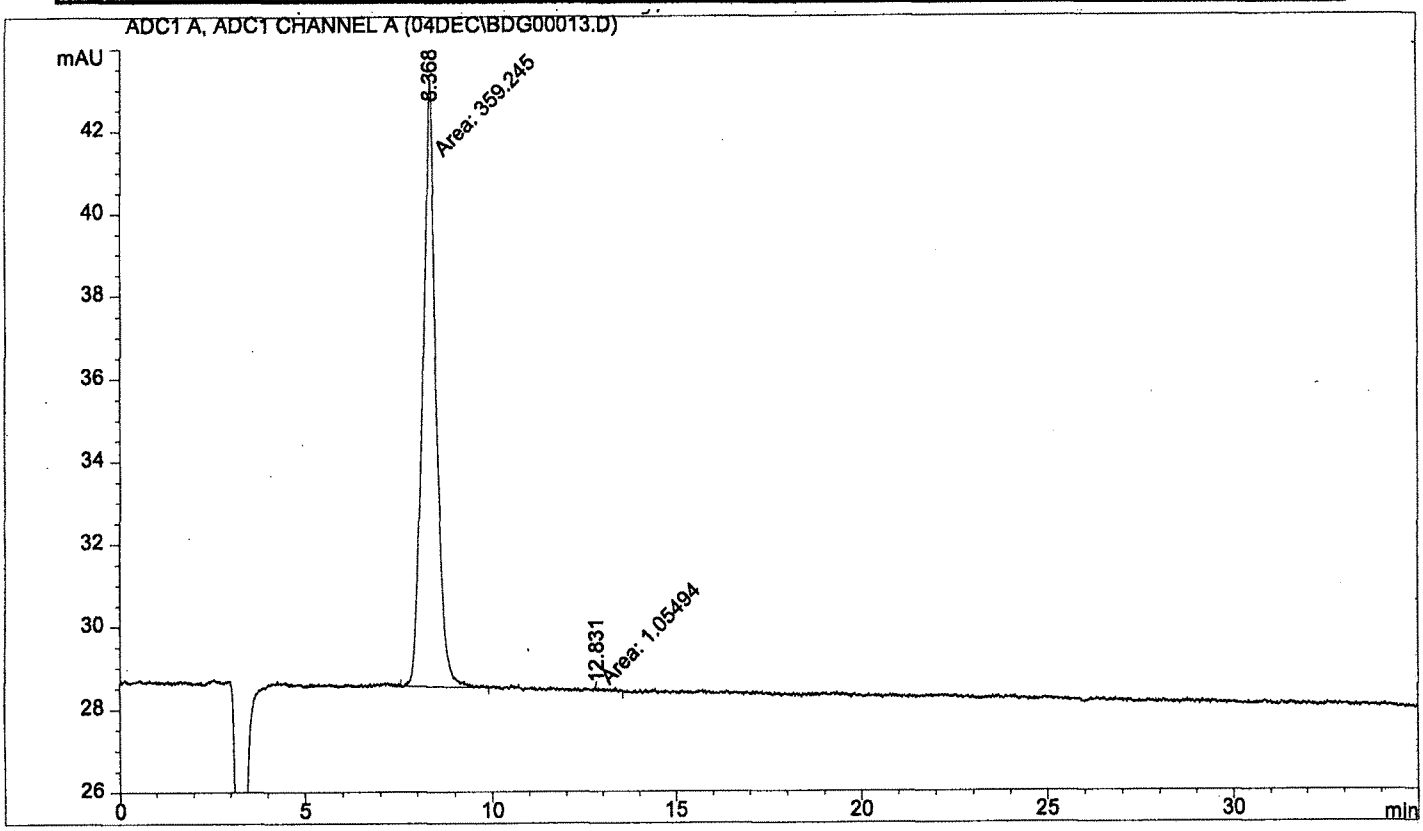
BDG - Analysis of Cortol-d5

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase : 70:30 Water : Acetonitrile
Flow Rate : 1.0 mL/min
Sample Solvent : Mobile Phase
Column Temperature : 20C
Injection Volume : 40 uL
Detection : RI

Sample Name	BDG 17240.3	Instrument	AnalyticalLC01
Acquisition	04/12/2015, 12:28:38	Method (rev.)	LC10218c (3)
Sequence	BDG_04Dec2015a	Vial Position	1
Operator	solvation010\cerityadmin	Injection	1 of 1

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================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: ADC1 A, ADC1 CHANNEL A

Peak RetTime Type  Width     Area      Height      Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.368 MM    0.4034  359.24521   14.84337  99.7072
   2  12.831 MM    0.2642    1.05494 5.13127e-2   0.2928

Totals :                   360.30015   14.89468

 Results obtained with enhanced integrator!
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                          *** End of Report ***
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