



## BDG SYNTHESIS

### Certificate of Analysis

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

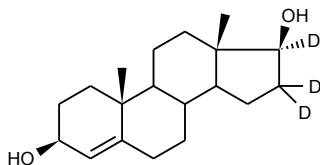
*Barry Dent*

Barry R. Dent, PhD, Director  
10 July 2014

**Name:** 4-Androstene-3 $\beta$ ,17 $\beta$ -diol-d<sub>3</sub>

**CAS Number:** 1156-92-9 (unlabelled)

**Structure:**



**Molecular Weight:** C<sub>19</sub>H<sub>27</sub>D<sub>3</sub>O<sub>2</sub> = 293.46

**Lot Number:** BDG 15046.2

**Appearance:** White, crystalline solid

**Corrected Purity:** 99.3 % (HPLC) - 0.2 % (methanol) - 0.6 % (water) = 98.5 %

**Isotopic Purity:** Under 1.5% d<sub>0</sub>

**Re-test Date:** 10 July 2019

**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.

Isotopic Labelling: signals at the sites of deuteration are absent, compared with the spectrum of unlabelled material, indicating clean deuteration.

Residual Solvents: a small amount of methanol (0.2 % 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.

Isotopic Labelling: signals at the sites of deuteration have collapsed to small multiplets compared with the spectrum of unlabelled material, indicating clean deuteration.

### High-resolution Mass Spectrum (ESI+)

Found  $m/z$  316.2330.  $C_{19}H_{27}D_3NaO_2$   $[M+Na]^+$  requires  $m/z$  316.2332. The deviation of 0.6 ppm is within normally accepted limits for the establishment of identity by HRMS. The sample is actually believed to contain significantly less  $d_0$  material than indicated above, due to an unusually weak signal for the molecular ion, resulting in a poor signal to noise ratio.

### HPLC

A sharp, slightly tailing peak is observed (99.3 %). 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 77.19, H 9.36, D 2.07 %
$C_{19}H_{27}D_3O_2 \cdot 0.1H_2O$	Requires:	C 77.29, H 9.29, D 2.05 %, $H_2O$ 0.61 %
$C_{19}H_{27}D_3O_2$	Requires:	C 77.76, H 9.27, D 2.06 %

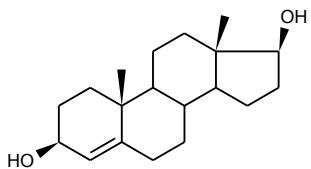
The elemental analyses fall slightly outside those expected for anhydrous material; the presence of water is reasonably expected from the method of purification and/or the type of material, and the "best-fit" hydrated molecular formula is given. In the absence of a Karl-Fischer water analysis, we recommend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

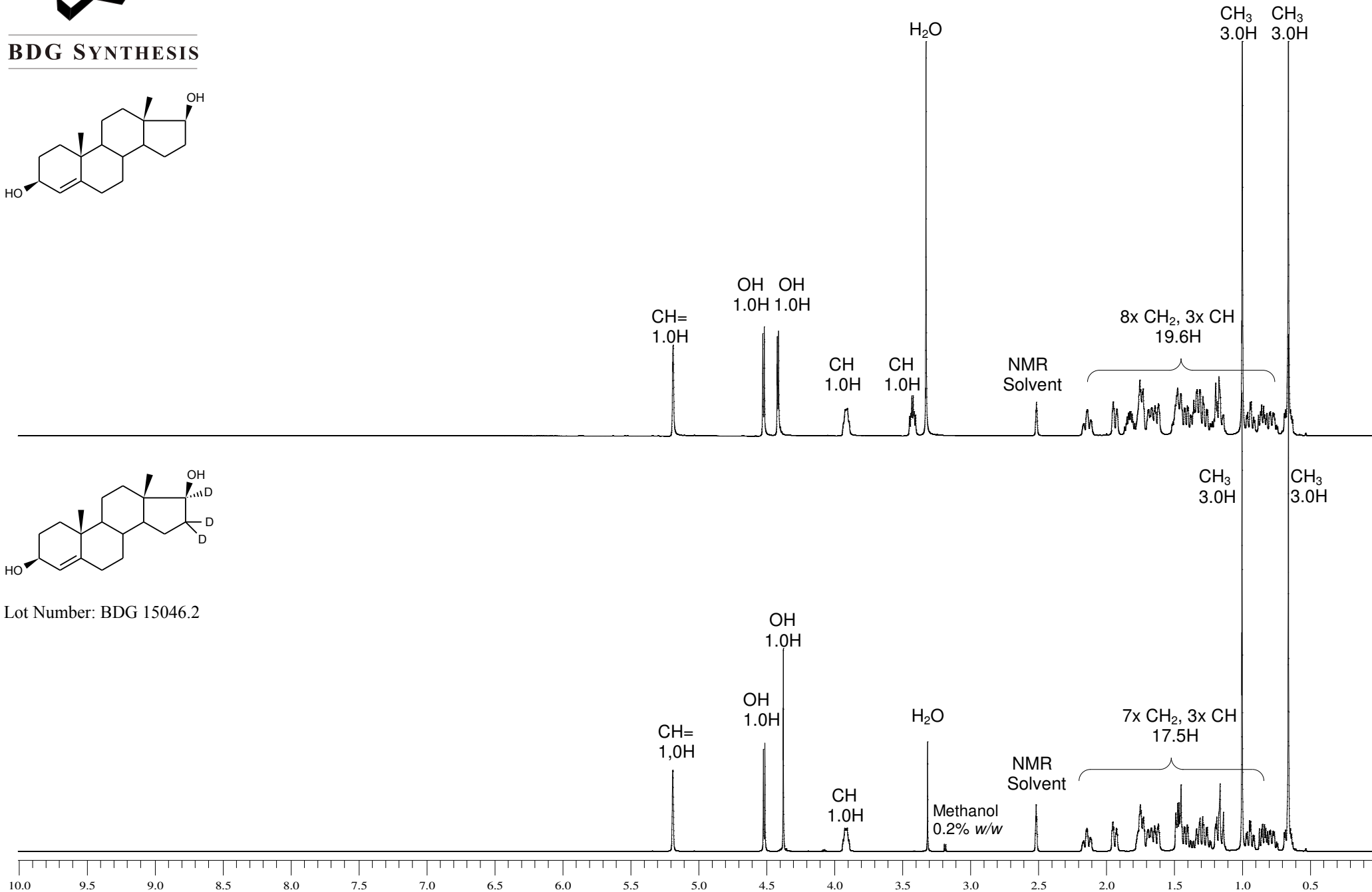
The re-test date is assigned from experience gained with the material in the laboratory and/or on storage. It is not possible to perform formal storage studies because of the small amount of material available.



**BDG SYNTHESIS**



Proton NMR Spectrum of 4-Androstene-3 $\beta$ ,17 $\beta$ -diol (top) and 4-Androstene-3 $\beta$ ,17 $\beta$ -diol-d<sub>3</sub> (bottom) in DMSO-d<sub>6</sub>

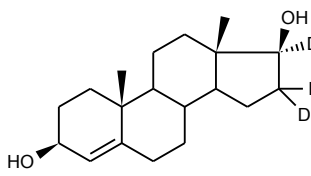
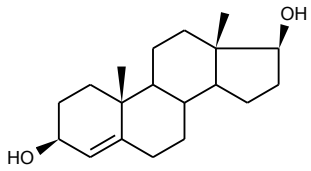


Lot Number: BDG 15046.2

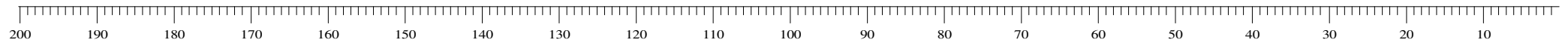
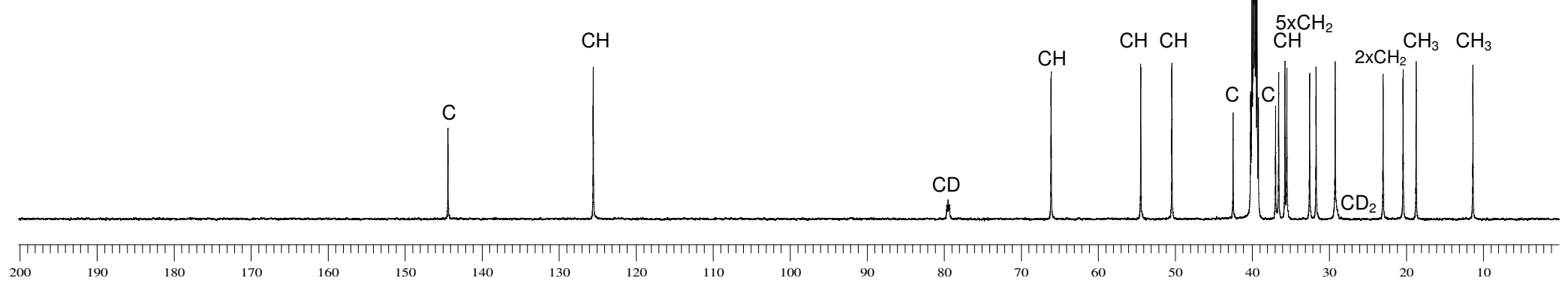


**BDG SYNTHESIS**

Carbon-13 NMR Spectrum of 4-Androstene-3 $\beta$ ,17 $\beta$ -diol (top) and 4-Androstene-3 $\beta$ ,17 $\beta$ -diol-d<sub>3</sub> (bottom) in DMSO-d<sub>6</sub>



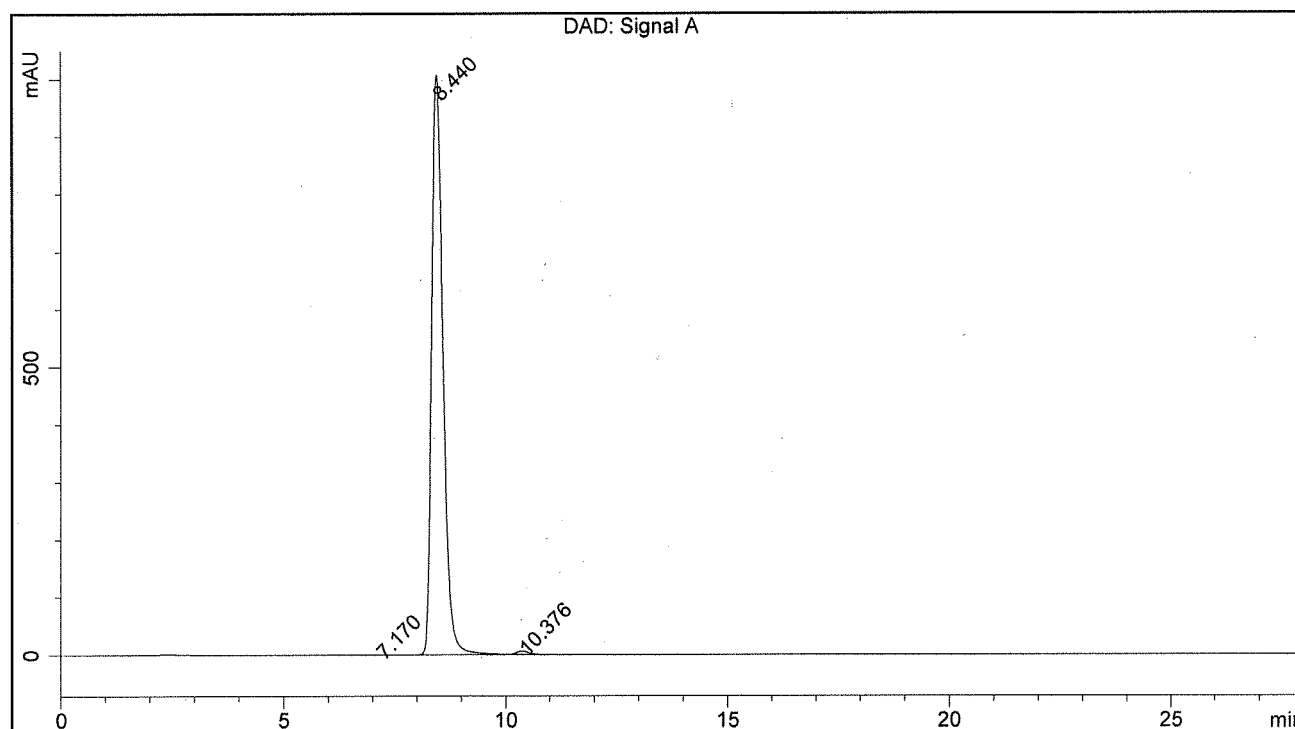
Lot Number: BDG 15046.2



BDG - Analysis of 4-Androstene-3b,17b-diol-d3

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm  
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm  
 Mobile Phase : 50:50 Water : Acetonitrile  
 Flow Rate : 1.0 mL/min  
 Sample Solvent : Mobile Phase  
 Column Temperature : 20C  
 Injection Volume : 10 uL  
 Detection : UV 205 nm

<b>Sample Name</b>	BDG 15046.2	<b>Instrument</b>	AnalyticalLC01
<b>Acquisition</b>	10/07/2014, 15:06:27	<b>Method (rev.)</b>	LC10620a ( 5)
<b>Sequence</b>	BDG_10Jul2014e	<b>Vial Position</b>	49
<b>Operator</b>	solvation010\cerityadmin	<b>Injection</b>	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	7.17 min	0.4444	5.8910	0.1647 min	0.033 %
2	8.44 min	1007.2491	17894.9608	0.2753 min	99.319 %
3	10.38 min	5.8183	116.7377	0.2981 min	0.648 %