

## BDG SYNTHESIS

### Certificate of Analysis

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

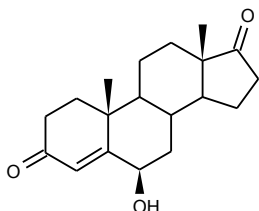
*Neil Beare*

Neil Beare, PhD, Director  
1 May 2016

**Name:** 4-Androsten-6 $\beta$ -ol-3,17-dione

**CAS Number:** 63-00-3

**Structure:**



**Molecular Weight:** C<sub>19</sub>H<sub>26</sub>O<sub>3</sub> = 302.41

**Lot Number:** BDG 16678.1

**Appearance:** White, crystalline solid

**Corrected Purity:** 99.4 % (HPLC) - 0.4 % (ethyl acetate) = 99.0 %

**Re-test Date:** 1 May 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: 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 (TOF MS ES+)

Found  $m/z$  325.1778.  $C_{19}H_{26}NaO_3$   $[M+Na]^+$  requires  $m/z$  325.1780. The deviation of 0.6 ppm is within normally accepted limits for the establishment of identity by HRMS.

### HPLC

A sharp, symmetrical peak is observed (99.4 %). 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 75.37, H 8.64 %
$C_{19}H_{26}O_3$	Requires:	C 75.46, H 8.67 %

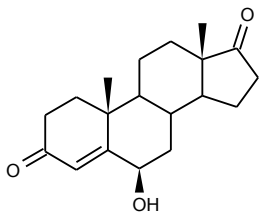
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

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.

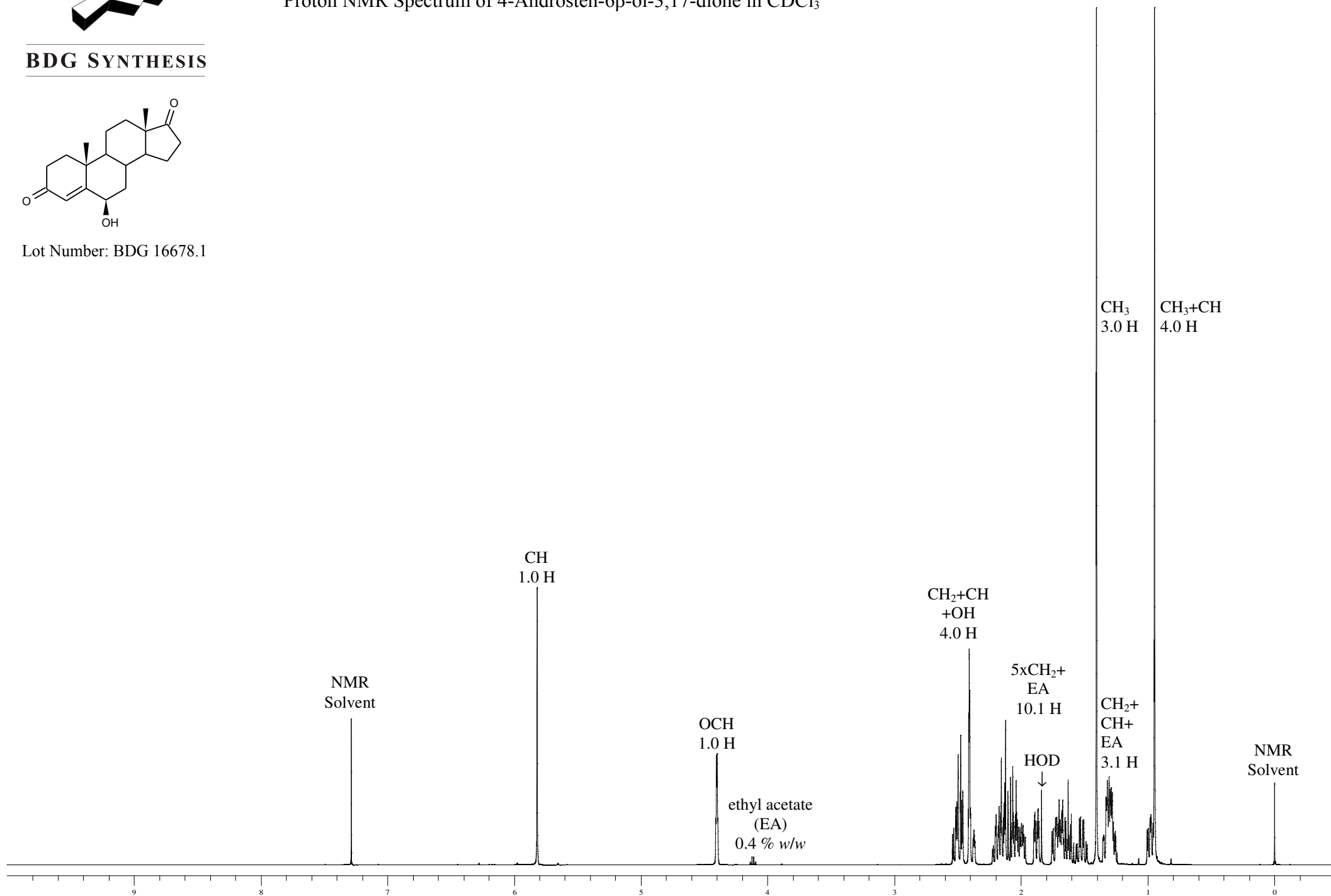


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Lot Number: BDG 16678.1

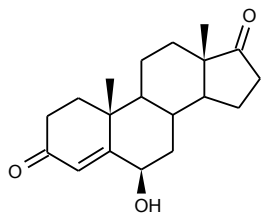
Proton NMR Spectrum of 4-Androsten-6 $\beta$ -ol-3,17-dione in CDCl<sub>3</sub>



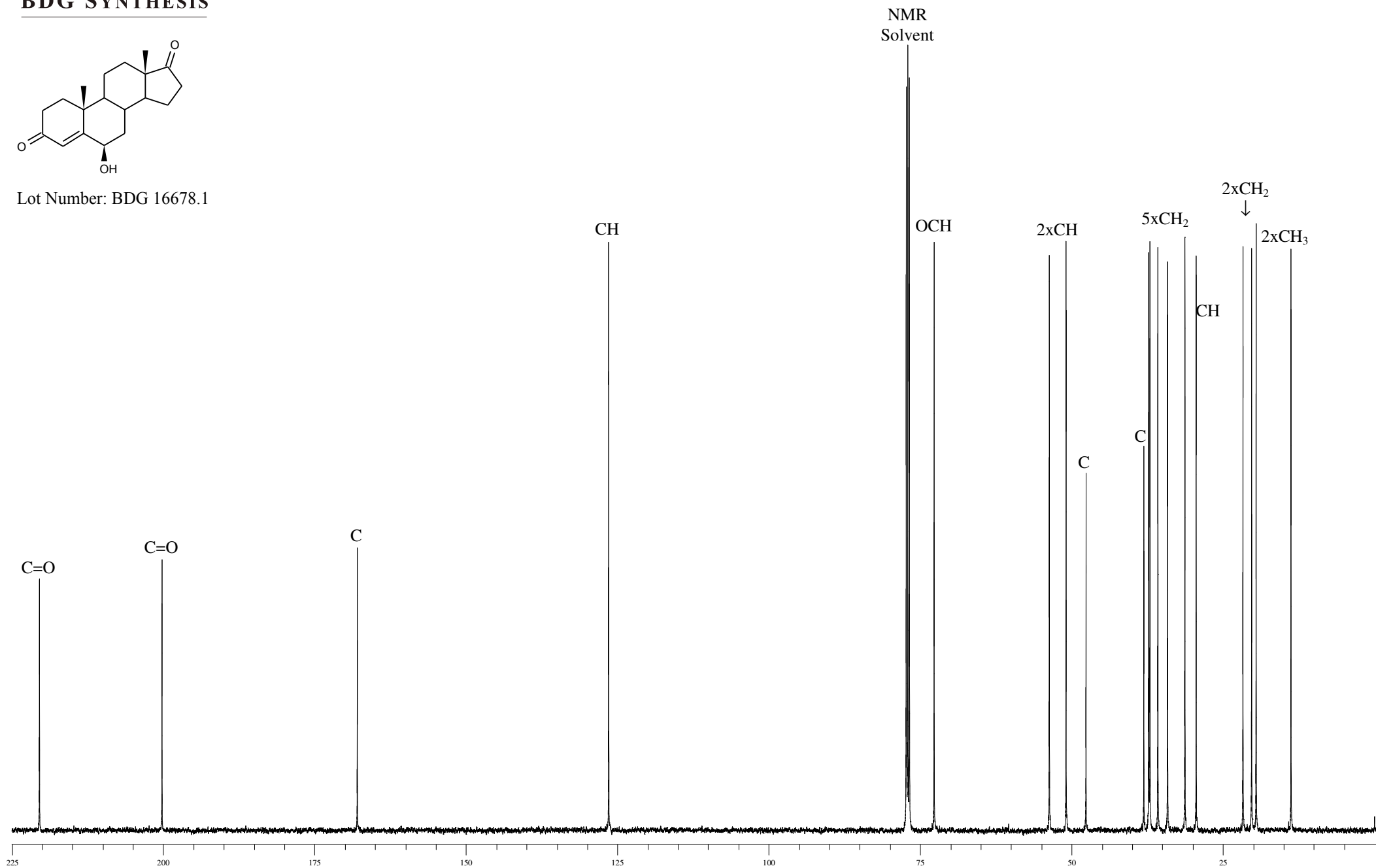


Carbon-13 NMR Spectrum of 4-Androsten-6 $\beta$ -ol-3,17-dione in CDCl<sub>3</sub>

BDG SYNTHESIS



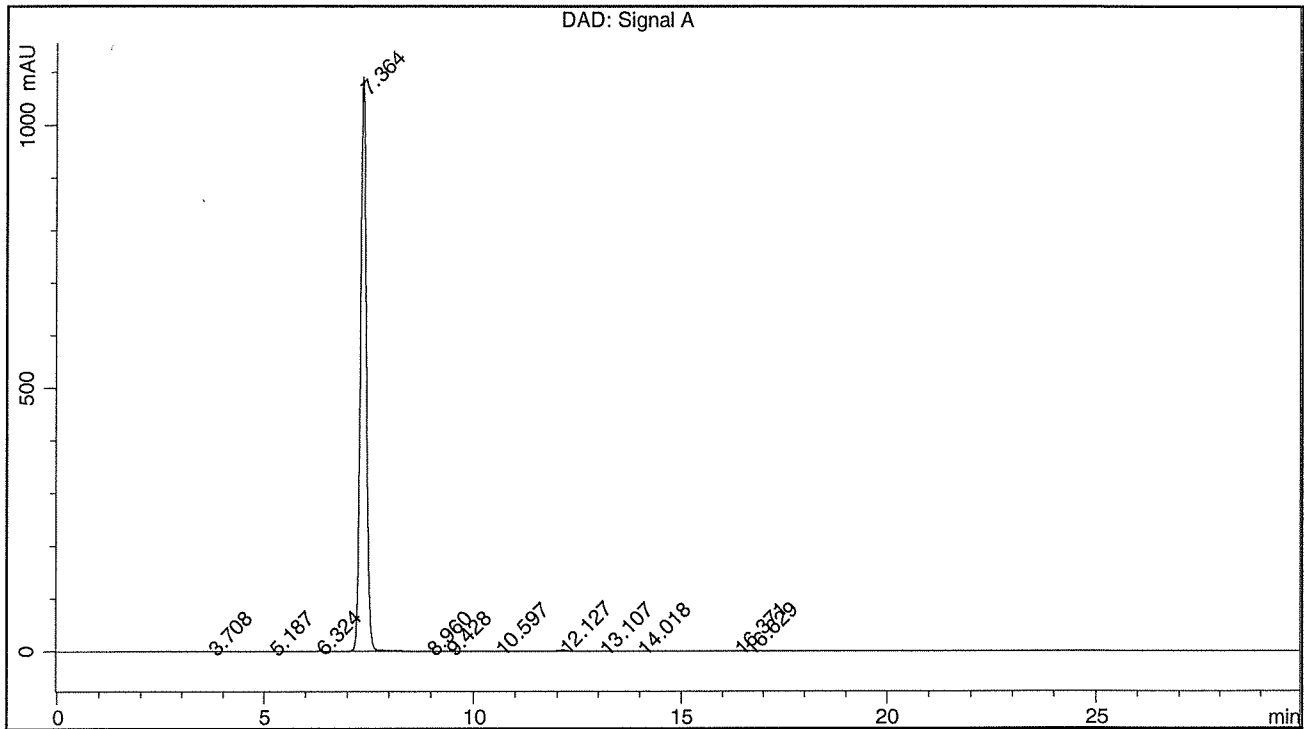
Lot Number: BDG 16678.1



BDG - Analysis of 4-Androsten-6B-ol-3,17-dione

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm  
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm  
 Mobile Phase : 60:40 Water : Acetonitrile . . . . . Flow Rate : 1.0 mL/min . . . . . Column Temperature : 20 C  
 Sample Solvent : Mobile Phase . . . . . Injection Volume : 10 uL . . . . . Detection : UV at 238 nm

<b>Sample Name</b>	BDG 16678.1	<b>Instrument</b>	AnalyticalLC01
<b>Acquisition</b>	01/05/2016, 14:44:14	<b>Method (rev.)</b>	LC10339b ( 6)
<b>Sequence</b>	BDG_01May2016c - Reprocessed	<b>Vial Position</b>	1
<b>Operator</b>	solvation010\cerityadmin	<b>Injection</b>	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	3.71 min	0.2093	2.1200	0.1382 min	0.020 %
2	5.19 min	0.1474	1.2554	0.1314 min	0.012 %
3	6.32 min	0.8252	7.5123	0.1422 min	0.069 %
4	7.36 min	1090.9297	10752.4443	0.1550 min	99.400 %
5	8.96 min	0.1683	2.7376	0.2338 min	0.025 %
6	9.43 min	0.1067	1.8184	0.2349 min	0.017 %
7	10.60 min	0.3113	5.0032	0.2395 min	0.046 %
8	12.13 min	2.1273	33.7224	0.2409 min	0.312 %
9	13.11 min	0.1144	2.1556	0.2478 min	0.020 %
10	14.02 min	0.2140	5.0811	0.3130 min	0.047 %
11	16.37 min	0.0965	2.0495	0.2797 min	0.019 %
12	16.63 min	0.0887	1.5001	0.2134 min	0.014 %