

## BDG SYNTHESIS

### Certificate of Analysis

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

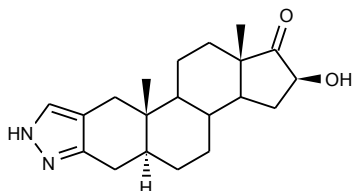
*Barry Dent*

Barry R. Dent, PhD, Director  
30 April 2012

**Name:** 16 $\beta$ -Hydroxy-2'H-5 $\alpha$ -androst-2-eno[3,2-c]pyrazol-17-one

**CAS Number:** none

**Structure:**



**Molecular Weight:** C<sub>20</sub>H<sub>28</sub>N<sub>2</sub>O<sub>2</sub> = 328.45

**Lot Number:** BDG 13439.2

**Appearance:** White, crystalline solid

**Corrected Purity:** 97.6 % (HPLC) - 6.1 % (chloroform) = 91.5 %

**Re-test Date:** 30 April 2013

**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. Avoid exposure to heating and alkali.

## 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 chloroform (6.1 % w/w) and a trace (under 0.1 % w/w) of acetone 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 (ESI+)

Found  $m/z$  329.2225.  $C_{20}H_{29}N_2O_2$   $[M+H]^+$  requires  $m/z$  329.2229. 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 (97.6 %). 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 73.28, H 8.81, N 8.56 %
$C_{20}H_{28}N_2O_2$	Requires:	C 73.14, H 8.59, N 8.53 %

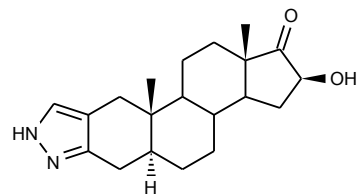
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

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.

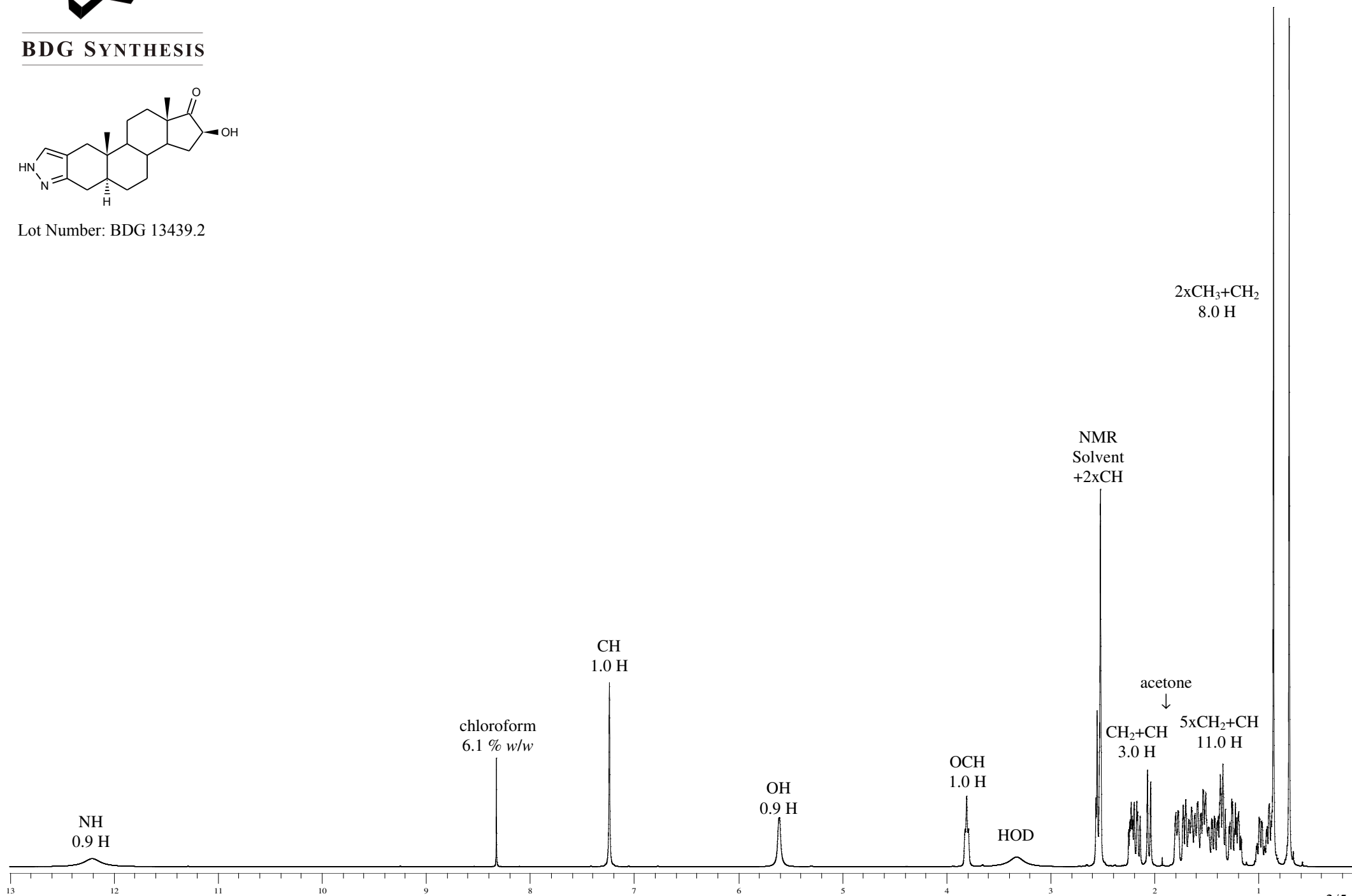


**BDG SYNTHESIS**



Lot Number: BDG 13439.2

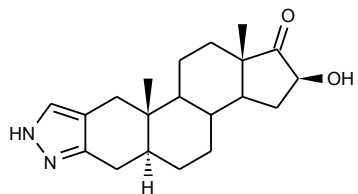
Proton NMR Spectrum of 16 $\beta$ -Hydroxy-2'H-5 $\alpha$ -androst-2-eno[3,2-c]pyrazol-17-one in DMSO-d<sub>6</sub>



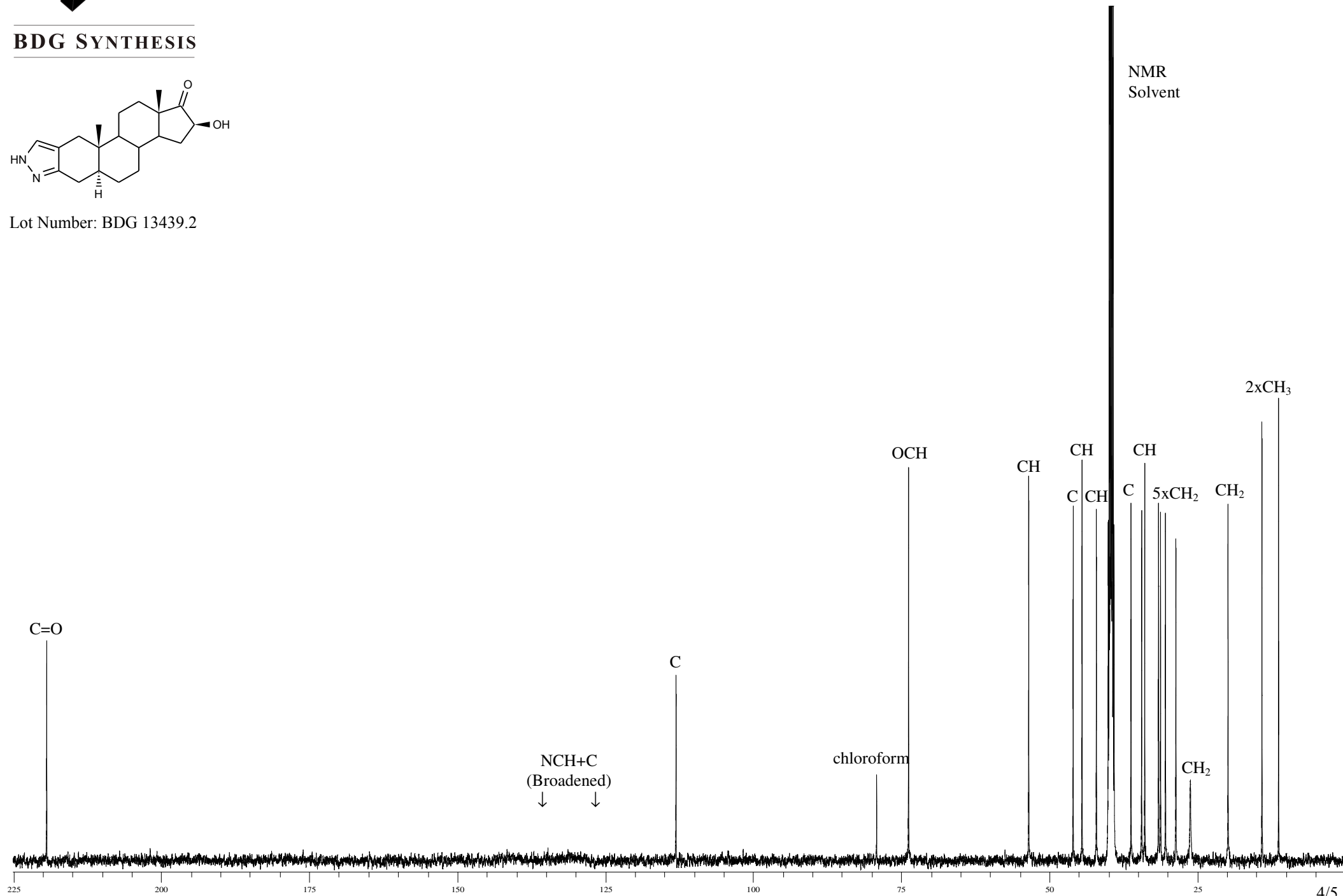


Carbon-13 NMR Spectrum of 16 $\beta$ -Hydroxy-2'H-5 $\alpha$ -androst-2-eno[3,2-c]pyrazol-17-one in DMSO-d<sub>6</sub>

**BDG SYNTHESIS**



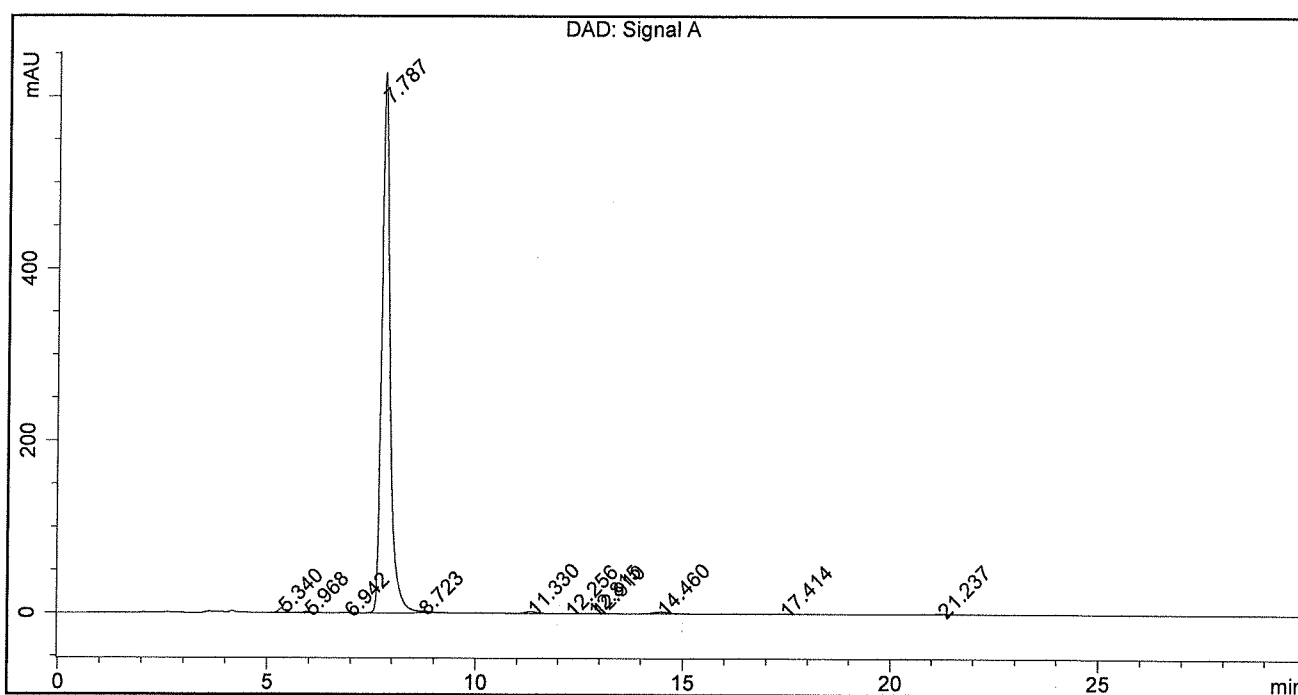
Lot Number: BDG 13439.2



BDG - Analysis of 16beta-Hydroxy-2'H-5alpha-androst-2-eno(3,2-c)pyrazol-17-one

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  
 Sample Solvent : 50:50 Water : Acetonitrile  
 Column Temperature : 20C  
 Injection Volume : 10 uL  
 Detection : UV at 224 nm

Sample Name	BDG 13439.2	Instrument	AnalyticalLC01
Acquisition	30/04/2012, 02:55:59	Method (rev.)	LC10504a ( 7)
Sequence	BDG_29Apr2012f - Reprocessed	Vial Position	1
Operator	solvation010\cerityadmin	Injection	2 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	5.34 min	5.0539	49.2201	0.1456 min	0.540 %
2	5.97 min	1.4043	16.0752	0.1736 min	0.176 %
3	6.94 min	0.1131	1.3852	0.1510 min	0.015 %
4	7.79 min	627.7155	8901.7175	0.2134 min	97.625 %
5	8.72 min	1.0645	22.6557	0.2943 min	0.248 %
6	11.33 min	2.0421	46.5022	0.3157 min	0.510 %
7	12.26 min	0.1204	1.4878	0.1562 min	0.016 %
8	12.82 min	0.1133	1.1738	0.1268 min	0.013 %
9	12.91 min	0.1136	1.2115	0.1384 min	0.013 %
10	14.46 min	1.9395	56.2598	0.4204 min	0.617 %
11	17.41 min	0.1583	3.1634	0.2472 min	0.035 %
12	21.24 min	0.5701	17.3887	0.3978 min	0.191 %