BDG SYNTHESIS

Certificate of Analysis

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

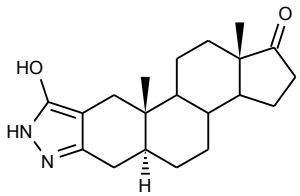
Barry Dent

Barry R. Dent, PhD, Director
14 December 2011

Name: **3'-Hydroxy-2'H-5α-androst-2-eno[3,2-c]pyrazol-17-one**

CAS Number: 1173998-80-5

Structure:



Molecular Weight: $C_{20}H_{28}N_2O_2$ = 328.45

Lot Number: BDG 13331.1

Appearance: White, crystalline solid

Corrected Purity: 99.8 % (HPLC) - 17.4 % (water) = 82.4 %

Re-test Date: 14 December 2012

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: 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 (ESI+)

Found *m/z* 329.2226. $C_{20}H_{29}N_2O_2$ $[M+H]^+$ requires *m/z* 329.2229. The deviation of 0.9 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A somewhat broadened, tailing peak is observed (99.8 %). Non-integrated peaks are present in the solvent blank. 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 60.73, H 9.00, N 7.04 %
$C_{20}H_{28}N_2O_2 \cdot 3.8H_2O$	Requires:	C 60.52, H 9.04, N 7.06 %
$C_{20}H_{28}N_2O_2$	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).

Karl-Fischer Analysis

	Found:	H_2O 17.4 %
$C_{20}H_{28}N_2O_2 \cdot 3.8H_2O$	Requires:	H_2O 17.3 %

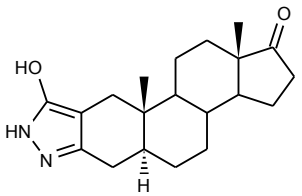
Of necessity, only a small sample could be used and only a single or duplicate analysis performed. We are unable to state what the errors in the reported water content are, but recommend that the result be used, as the best available, when determining corrected purity.

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.

BDG SYNTHESIS

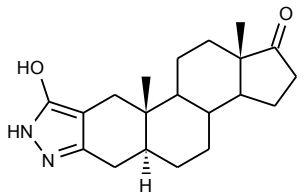
Proton NMR Spectrum of 3'-Hydroxy-2'H-5α-androst-2-eno[3,2-c]pyrazol-17-one in DMSO-d_6



Lot Number: BDG 13331.1

BDG SYNTHESIS

Carbon-13 NMR Spectrum of 3'-Hydroxy-2'H-5α-androst-2-eno[3,2-c]pyrazol-17-one in DMSO-d_6

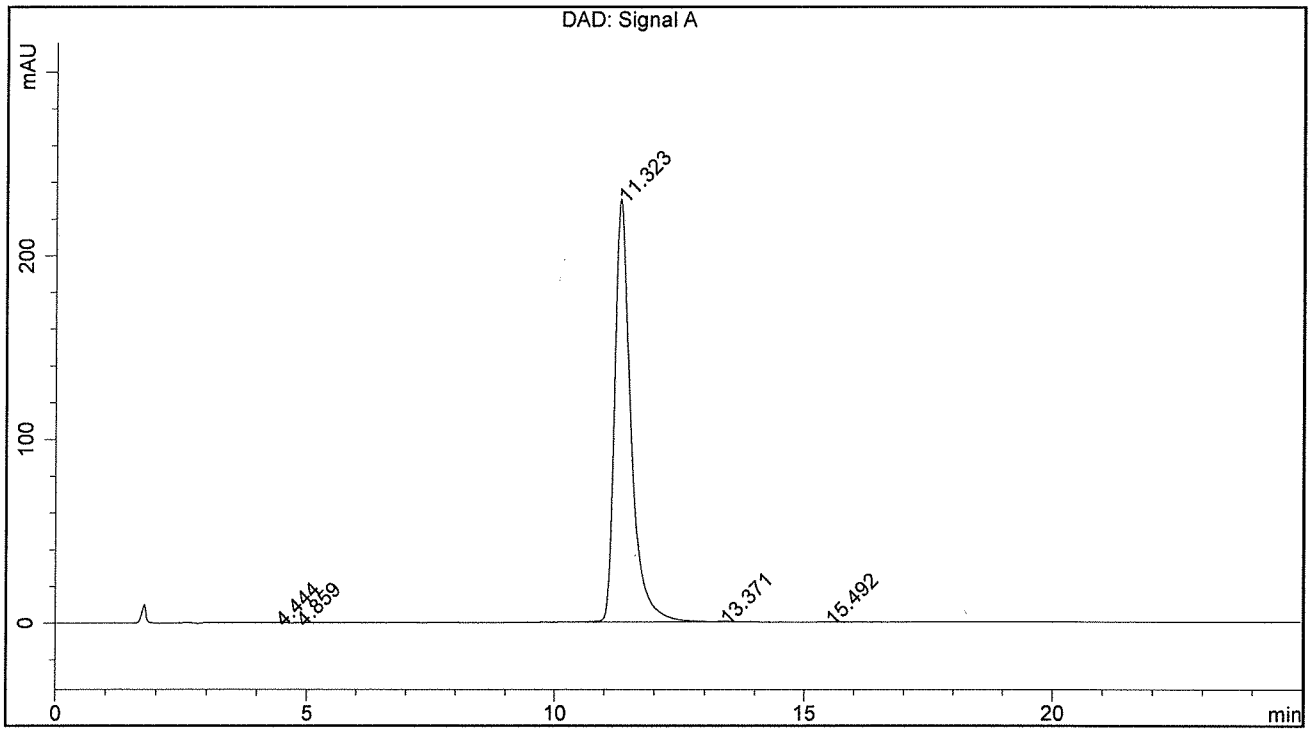


Lot Number: BDG 13331.1

BDG - Analysis of 3'-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 : 60:40 Water : Acetonitrile
Column Temperature : 20C
Injection Volume : 10 uL
Detection : UV at 250 nm

Sample Name	BDG 13331.1	**Instrument**	AnalyticalLC01
Acquisition	14/12/2011, 13:00:31	**Method (rev.)**	LC10475b (8)
Sequence	BDG_14Dec2011e - Reprocessed	**Vial Position**	3
Operator	solvation010\cerityadmin	**Injection**	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	4.44 min	0.2099	2.3006	0.1539 min	0.044 %
2	4.86 min	0.1103	1.1056	0.1249 min	0.021 %
3	11.32 min	230.1938	5194.9691	0.3333 min	99.795 %
4	13.37 min	0.3137	4.9100	0.2026 min	0.094 %
5	15.49 min	0.1426	2.3656	0.2015 min	0.045 %