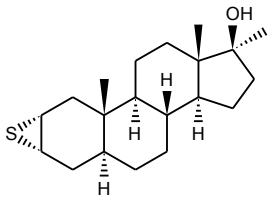
BDG SYNTHESIS

Certificate of Analysis

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

Barry Dent

Barry R. Dent, PhD, Director
15 February 2012

Name:	**2α,3α-Epithio-17α-methyl-5α-androstan-17β-ol**
CAS Number:	4267-80-5
Structure:	
Molecular Weight:	$C_{20}H_{32}OS$ = 320.53
Lot Number:	BDG 10838.8
Appearance:	White, crystalline solid
Corrected Purity:	94.7 % (HPLC) - 0.5 % (water) = 94.2 %
Re-test Date:	15 February 2014

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 trace (under 0.1 % *w/w*) of acetone is observed.
Impurities: approximately 6% *w/w* of the 2β,3β isomer of the product is observed.

Carbon-13 NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available.
Impurities: Signals for the 2β,3β isomer of the product are observed.

High-resolution Mass Spectrum (ESI+)

Found *m/z* 343.2072. $C_{20}H_{32}NaOS$ $[M+Na]^+$ requires *m/z* 343.2066. The deviation of 1.8 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A sharp, symmetrical peak is observed (94.7 %). The peak at 12.8 minutes is assigned to the 2β,3β isomer of the product. This was confirmed by spiking experiments. 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 74.76, H 10.21, S 9.89 %
$C_{20}H_{32}OS{\cdot}0.1H_2O$	Requires:	C 74.52, H 10.07, S 9.95 %
$C_{20}H_{32}OS$	Requires:	C 74.94, H 10.06, S 10.00 %

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.

Karl-Fischer Analysis

	Found:	H_2O 0.5 %
$C_{20}H_{32}OS{\cdot}0.1H_2O$	Requires:	H_2O 0.6 %

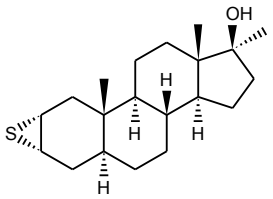
Of necessity, only a small sample could be used and only a single or duplicate analysis performed. We are unable to state what the errors in the reported water content are, but recommend that the result be used, as the best available, when determining corrected purity.

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 2α,3α-Epithio-17α-methyl-5α-androstan-17β-ol in $CDCl_3$



Lot Number: BDG 10838.8

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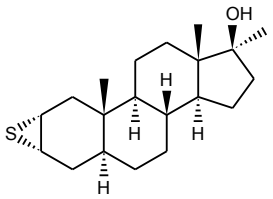
Carbon-13 NMR Spectrum of 2α,3α-Epithio-17α-methyl-5α-androstan-17β-ol in $CDCl_3$



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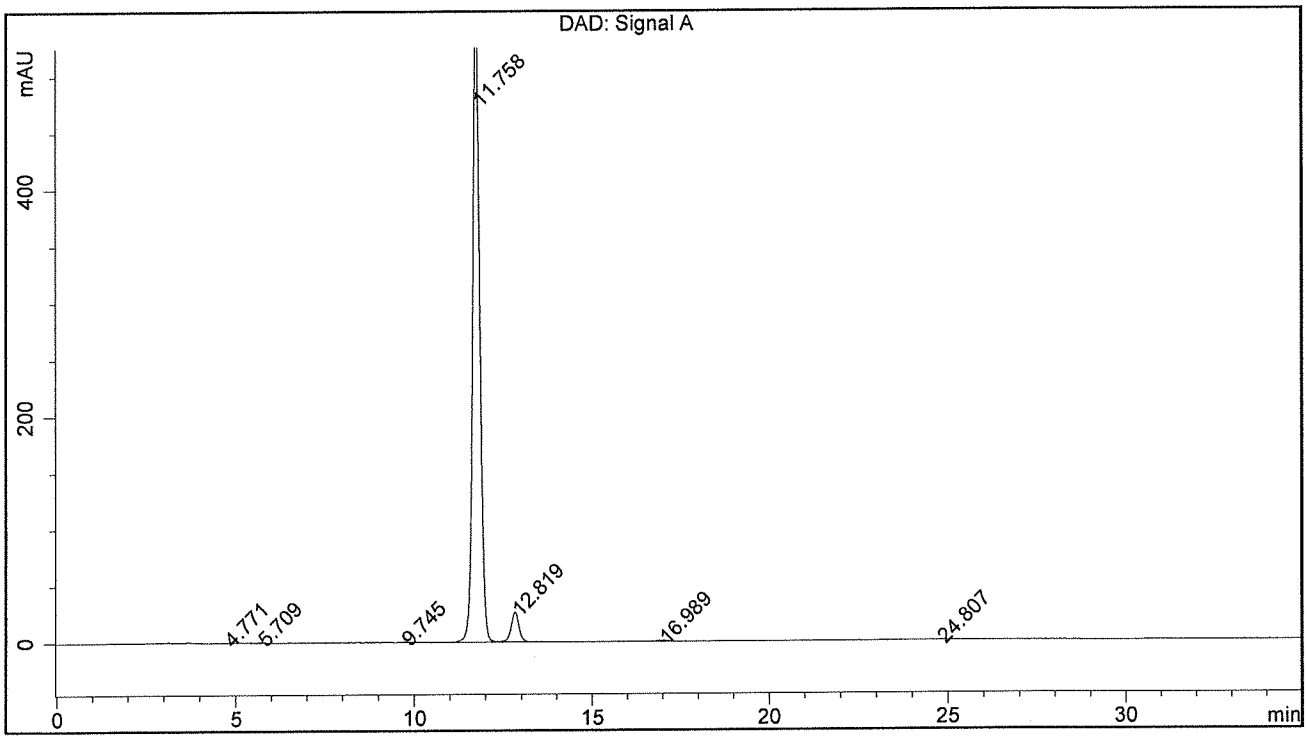
BDG - Analysis of 2a,3a-Epithio-17a-methyl-5a-androstan-17B-ol

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase : 10:90 Water : Acetonitrile
Flow Rate : 1.0 mL/min
Sample Solvent : Mobile Phase
Column Temperature : 20C
Injection Volume : 10 uL
Detection : UV at 205 nm

Sample Name	BDG 10838.8	Instrument	AnalyticalLC01
Acquisition	15/02/2012, 14:58:01	Method (rev.)	LC10389b (4)
Sequence	BDG_15Feb2012a	Vial Position	51
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	4.77 min	0.2651	2.1383	0.1141 min	0.026 %
2	5.71 min	0.5187	6.0579	0.1622 min	0.074 %
3	9.75 min	0.3677	6.6855	0.2401 min	0.081 %
4	11.76 min	562.8160	7784.5653	0.2114 min	94.692 %
5	12.82 min	26.2709	398.9357	0.2332 min	4.853 %
6	16.99 min	0.9340	18.4208	0.2461 min	0.224 %
7	24.81 min	0.1604	4.1310	0.3125 min	0.050 %