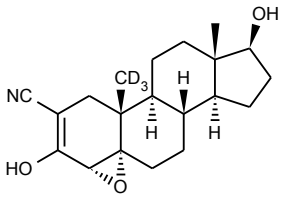
BDG SYNTHESIS

Certificate of Analysis

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

Neil Beare, PhD, Director
12 August 2015

Name:	**Trilostane-d_3**
CAS Number:	13647-35-3 (unlabelled)
Structure:	
Molecular Weight:	$C_{20}H_{24}D_3NO_3$ = 332.45
Lot Number:	BDG 8961
Appearance:	White, crystalline solid
Purity By HPLC:	99.6 %
Isotopic Purity:	Under 0.5 % d_0
Re-test Date:	12 August 2020

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: the signal at the site of deuteration is absent, compared with the spectrum of unlabelled material, indicating clean deuteration.
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.
Isotopic Labelling: the signal at the site of deuteration has collapsed to a small multiplet, compared with the spectrum of unlabelled material, indicating clean deuteration.

High-resolution Mass Spectrum (ESI+)

Found *m/z* 355.2079. $C_{20}H_{24}D_3NNaO_3$ $[M+Na]^+$ requires *m/z* 355.2077. The deviation of 0.6 ppm is within normally accepted limits for the establishment of identity by HRMS. No signal for d_0 material was seen (detection limit about 0.5 %).

HPLC

A broad, slightly tailing peak is observed (99.6 %). 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 72.30, H 7.52, D 1.88, N 4.13 %
$C_{20}H_{24}D_3NO_3$	Requires:	C 72.26, H 7.28, D 1.82, N 4.21 %

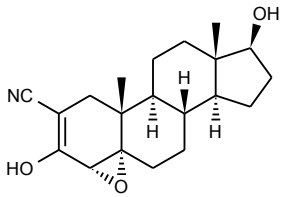
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

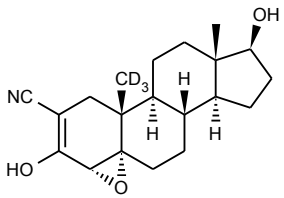
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials 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

Proton NMR Spectrum of Trilostane (top) and Trilostane-d_3 (bottom) in DMSO-d_6

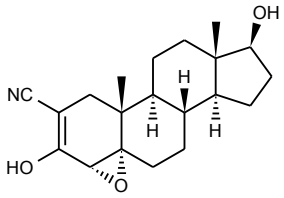


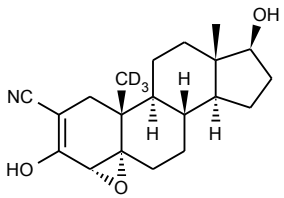


Lot Number: BDG 8961

BDG SYNTHESIS

Carbon-13 NMR Spectrum of Trilostane (top) and Trilostane-d_3 (bottom) in DMSO-d_6



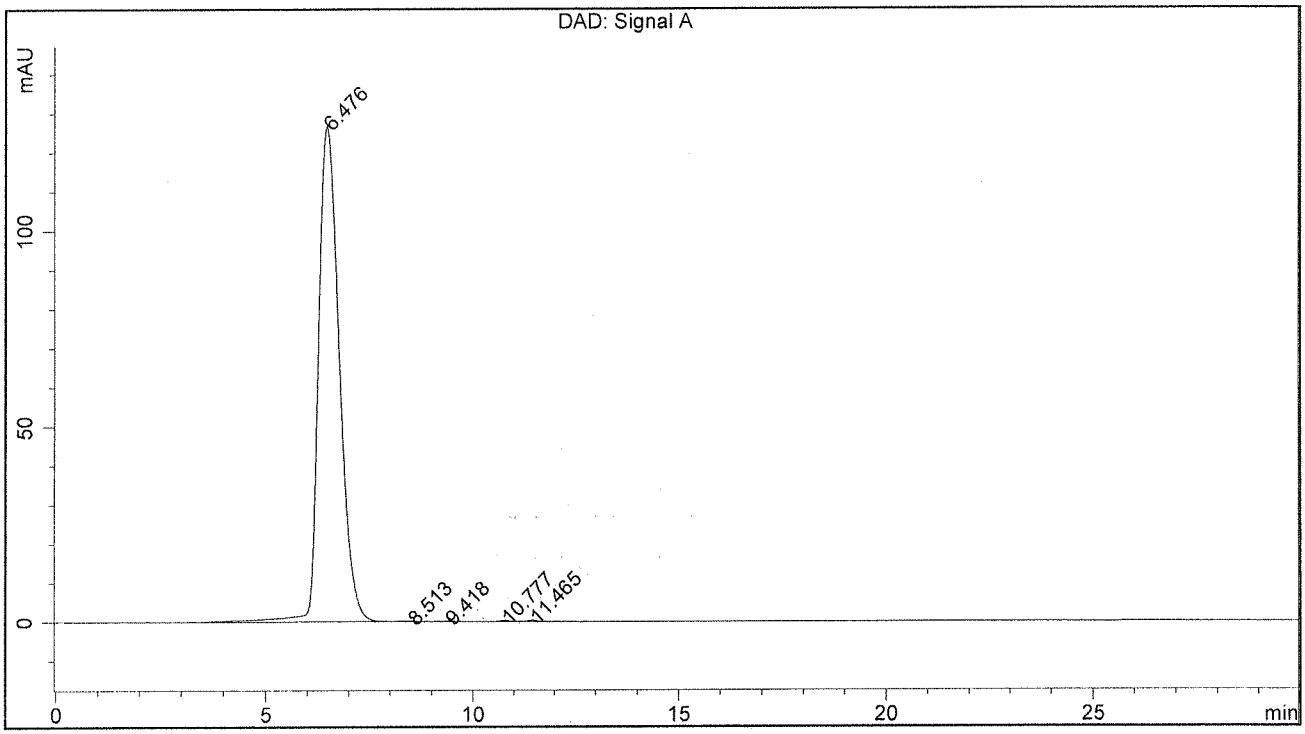


Lot Number: BDG 8961

BDG - Analysis of Trilostane-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 20 mM Potassium diHydrogen Phosphate pH=3.0 : Acetonitrile
Flow Rate : 1.0 mL/min
Sample Solvent : Mobile Phase
Column Temperature : 30C
Injection Volume : 10 uL
Detection : UV at 252 nm

Sample Name	BDG 8961	Instrument	AnalyticalLC01
Acquisition	12/08/2015, 18:06:53	Method (rev.)	LC10365c (8)
Sequence	BDG_12Aug2015c - Reprocessed	Vial Position	45
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	6.48 min	126.6018	4437.0515	0.5469 min	99.603 %
2	8.51 min	0.1400	3.8177	0.3302 min	0.086 %
3	9.42 min	0.1558	2.6990	0.2181 min	0.061 %
4	10.78 min	0.3268	4.9117	0.1994 min	0.110 %
5	11.46 min	0.4034	6.2554	0.2049 min	0.140 %