



BDG SYNTHESIS

Certificate of Analysis

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

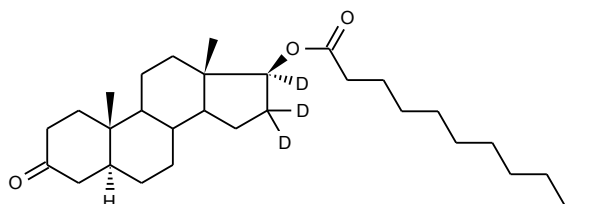
Neil Beare

Neil Beare, PhD, Director
14 October 2014

Name: 5 α -Dihydrotestosterone-16,16,17-d₃ Undecanoate

CAS Number: 6804-12-2 (unlabelled)

Structure:



Molecular Weight: C₃₀H₄₇D₃O₃ = 461.73

Lot Number: BDG 5858.1

Appearance: Off-white, crystalline solid

Purity By HPLC: 99.2 %

Isotopic Purity: 0.5 % d₀

Re-test Date: 14 October 2019

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: signals at the sites of deuteration are absent, compared with the spectrum of unlabelled material, indicating clean deuteration.

Residual Solvents: a trace (under 0.1 % w/w) of ethanol 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.

Isotopic Labelling: signals at the sites of deuteration have collapsed to small multiplets compared with the spectrum of unlabelled material, indicating clean deuteration.

High-resolution Mass Spectrum (ESI+)

Found m/z 484.3821. $C_{30}H_{47}D_3NaO_3$ $[M+Na]^+$ requires m/z 484.3840. The deviation of 4.0 ppm is within normally accepted limits for the establishment of identity by HRMS. A small signal of approximately 0.5 % intensity for d_0 material was observed.

HPLC

A somewhat broadened, symmetrical peak is observed (99.2 %). 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 78.64, H 10.56, D 1.35 %
$C_{30}H_{47}D_3O_3$	Requires:	C 78.04, H 10.26, D 1.31 %

The elemental analyses fall within generally accepted limits (± 0.4 %) for establishing the molecular formula given, except the result for carbon. 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).

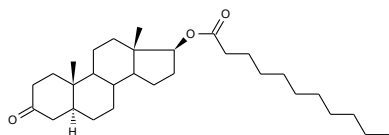
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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



Proton NMR Spectrum of 5 α -Dihydrotestosterone Undecanoate (top) and 5 α -Dihydrotestosterone-16,16,17-d₃ Undecanoate (bottom) in C₆D₆

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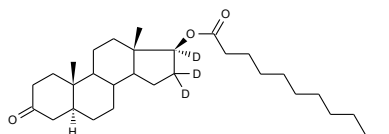


NMR solvent

OCH
1.0 H

2xCH₂
4.0 H

4xCH₂
8.3 H



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ethanol

CH₂+CH
2.9 H

3xCH₂+
CH
7.2 H

CH₃

2xCH₃

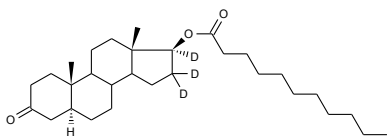
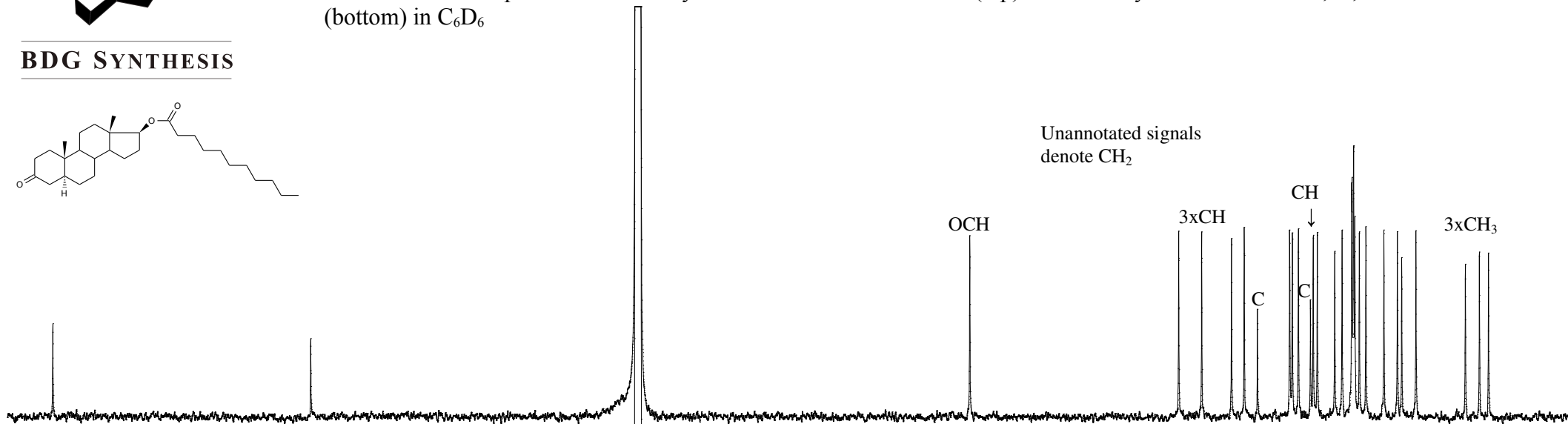
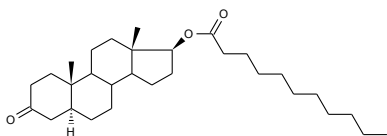
HOD



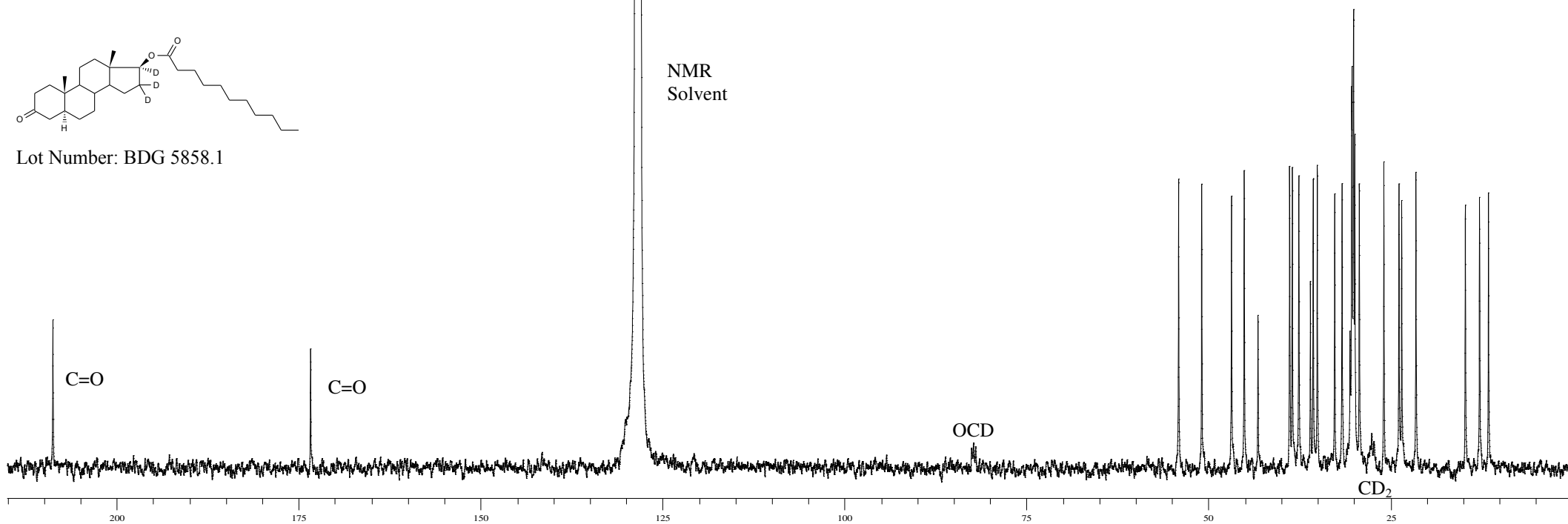


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Carbon-13 NMR Spectrum of 5 α -Dihydrotestosterone Undecanoate (top) and 5 α -Dihydrotestosterone-16,16,17-d₃ Undecanoate (bottom) in C₆D₆



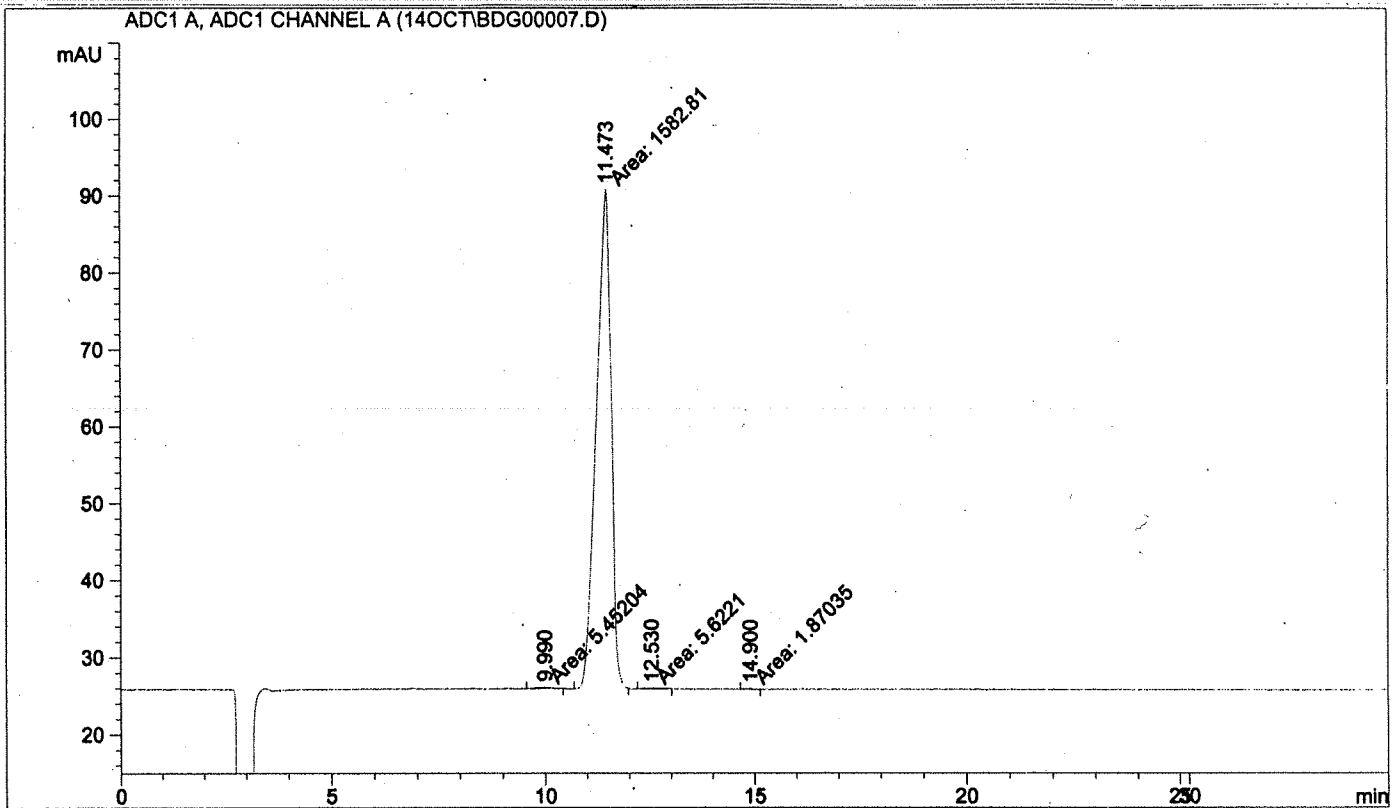
Lot Number: BDG 5858.1



BDG - Analysis of 5alpha-Dihydrotestosterone Undecanoate

Column : Phenomenex Luna C8(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C8 RP 4 x 3 mm
 Mobile Phase : 5:95 Water : Acetonitrile
 Flow Rate : 1.0 mL/min
 Sample Solvent : Acetonitrile
 Column Temperature : 40C
 Injection Volume : 100 uL
 Detection : RI

Sample Name	BDG 5858.1	Instrument	AnalyticalLC01
Acquisition	14/10/2014, 11:56:15	Method (rev.)	LC10341e (5)
Sequence	BDG_14Oct2014b - Reprocessed	Vial Position	1
Operator	solvation010\cerityadmin	Injection	1 of 1



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 Area Percent Report
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Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000

Signal 1: ADC1 A, ADC1 CHANNEL A

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.990	MM	0.4801	5.45204	1.89267e-1	0.3417
2	11.473	MM	0.4066	1582.80713	64.87444	99.1888
3	12.530	MM	0.5672	5.62210	1.65193e-1	0.3523
4	14.900	MM	0.2176	1.87035	1.43285e-1	0.1172

Totals : 1595.75163 65.37219

Results obtained with enhanced integrator!

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 *** End of Report ***